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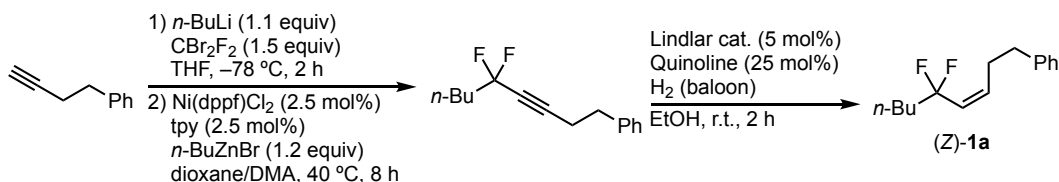
## 1. Instrumentation and Chemicals

Materials were obtained from commercial suppliers and purified by standard procedures unless otherwise noted. Solvents were also purchased from commercial suppliers, degassed via three freeze-pump-thaw cycles, and further dried over molecular sieves (MS 4Å). NMR spectra were recorded on JEOL JNM-ECX400P, JNM-ECS400 and Varian Unity INOVA 400, 500, or 600 spectrometers ( $^1\text{H}$ : 392, 396, 399, 400, 401, 500, and 600 MHz,  $^{13}\text{C}$ : 100, 125 and 150 MHz and  $^{19}\text{F}$ : 373, 376, 564 MHz). Tetramethylsilane ( $^1\text{H}$ ),  $\text{CDCl}_3$  ( $^1\text{H}$ ,  $^{13}\text{C}$ ) and Fluorobenzene ( $^{19}\text{F}$ ,  $\delta$  -113.60) were employed as the external standards, respectively. Fluorobenzene was used as an internal standard to determine NMR yield. Multiplicity was recorded as follows: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet.  $\text{CuCl}$  (ReagentPlus® grade,  $\geq 99\%$ ) was purchased from Sigma-Aldrich Co. and Strem. and used as received. GLC analyses were conducted with a Shimadzu GC-2014 or GC-2025 equipped with a ULBON HR-1 glass capillary column (Shinwa Chemical Industries) and a FID detector. Enantiomeric ratios were determined by HPLC analysis (high-performance liquid chromatography) with a Hitachi Chromaster HPLC [Chiral Technologies Chiralpak IA-3 (4.6 x 250 mm), Chiral Technologies Chiralpak IBN-3 (4.6 x 250 mm), Chiral Technologies Chiralpak IC-3 (4.6 x 250 mm), Chiral Technologies Chiralpak ID-3 (4.6 x 250 mm), Chiral Technologies Chiralpak IE-3 (4.6 x 250 mm), or Chiral Technologies Chiralpak IF-3 (4.6 x 250 mm)] and Shimadzu chromatograph [Chiral Technologies Chiralcel AZ-H (4.6 x 250 mm), Chiral Technologies Chiralcel OC-H (4.6 x 250 mm), Chiral Technologies Chiralcel OD-H (4.6 x 250 mm), Chiral Technologies Chiralcel OJ-H (4.6 x 250 mm), Chiral Technologies Chiralcel OZ-H (4.6 x 250 mm), or Chiral Technologies Chiralpak AD-H (4.6 x 250 mm)]. Specific optical rotations were measured with HORIBA SEPA-300 and a Rudolph Research Analytical Autopol IV Polarimeter. Medium-pressure column chromatography was carried out on a Biotage Flash Purification System Isolera, which is equipped with a UV detector. Recycle preparative gel permeation chromatography (GPC) was conducted with a JAI LC-9101 using  $\text{CHCl}_3$  as the eluent. High-resolution mass spectra were recorded at the Global Facility Center, Hokkaido University and at the Boston College Mass Spectrometry Facility.

## 2. Substrate Preparation Procedures

### 2.1. (Z)-Alkene substrates synthesis

#### Preparation of (Z)-(5,5-difluoronon-3-en-1-yl)benzene [(Z)-1a].<sup>1,2</sup>



To a solution of 4-phenyl-1-butyne (1.0 g, 7.7 mmol) in anhydrous THF (16 mL) at  $-78\text{ }^\circ\text{C}$  was added  $n\text{-BuLi}$  (2.5 M in hexanes, 3.4 mL, 8.5 mmol) in a dropwise manner under  $\text{N}_2$  atm. The mixture was allowed to stir for 60 min at  $-78\text{ }^\circ\text{C}$ , after which it was cooled to  $-196\text{ }^\circ\text{C}$  (liquid  $\text{N}_2$ ). Dibromodifluoromethane (2.413 g, 11.6 mmol) was added to the mixture by cannula. The mixture was then allowed to warm to  $22\text{ }^\circ\text{C}$  slowly and stir for 2 h at  $22\text{ }^\circ\text{C}$ . The reaction was quenched by the addition of a saturated solution of  $\text{NH}_4\text{Cl}$  (10 mL), and the volatiles were removed in vacuo. The brown oil residue was washed with  $n$ -pentane (3 x 50 mL) and the combined organic layers were washed with a saturated solution of  $\text{NaCl}$  (50 mL), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated in vacuo. The brown oil residue was purified by silica gel chromatography (hexanes) to give (5-bromo-5,5-difluoropent-3-yn-1-yl)benzene (1.9 g, 95% yield) as colorless oil.

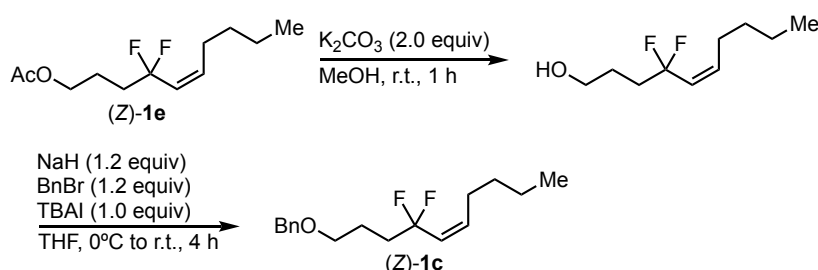
To a 100 mL Schlenk tube was added  $\text{Ni}(\text{dppf})\text{Cl}_2$  (20.5 mg, 0.03 mmol) and terpyridine (7.0 mg, 0.03 mmol). The tube was placed under vacuum and backfilled with  $\text{N}_2$  3 times, then (5-bromo-5,5-difluoropent-3-yn-1-yl)benzene (335.0 mg, 1.2 mmol) was added followed by 1,4-dioxane (8 mL). The mixture was allowed to stir for 10 min at  $22\text{ }^\circ\text{C}$ , at which time  $n$ -butylzinc(II) bromide (1.44 mmol, 0.41 M in dimethylacetamide) was added in a dropwise manner over a period of 15 min. The tube was capped and placed in a preheated oil bath ( $40\text{ }^\circ\text{C}$ ). The mixture was allowed to stir for 8 h at  $40\text{ }^\circ\text{C}$  for 8 h and to cool to r.t. The mixture was diluted with EtOAc (50 mL) and filtered through a pad of celite. The filtrate was washed with a saturated solution of  $\text{NaCl}$  (50 mL). The organic layer was dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. The brown oil residue was purified by silica gel chromatography (hexanes) to furnish (5,5-difluoronon-3-yn-1-yl)benzene (224 mg, 79% yield) as colorless oil.

In a vacuum dried 50 mL round bottomed flask, Lindlar's catalyst (744.9 mg, 0.35 mmol) was dissolved in EtOH (100 mL) and quinoline (207  $\mu\text{L}$ , 1.75 mmol) was added to this mixture. The corresponding difluoro compound (1.78 g, 7.0 mmol) was then added to this mixture and stirred for 2 h under  $\text{H}_2$  atmosphere ( $\text{H}_2$  balloon). The mixture was then filtered through a Celite pad and concentrated under reduced pressure. The oily residue was subjected

to silica gel chromatography (hexanes). (*Z*)-**1a** was obtained in 73% yield (1.56 g, 6.6 mmol) as colorless oil.

<sup>1</sup>H NMR (396 MHz, CDCl<sub>3</sub>): δ 0.90 (t, *J* = 7.1 Hz, 3H), 1.25–1.44 (m, 4H), 1.74–1.89 (m, 2H), 2.53–2.62 (m, 2H), 2.70 (t, *J* = 7.5 Hz, 2H), 5.40–5.53 (m, 1H), 5.70 (tdt, *J* = 1.9, 7.7, 11.7 Hz, 1H), 7.18–7.31 ppm (m, 5H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 13.8 (CH<sub>3</sub>), 22.4 (CH<sub>2</sub>), 24.4 (CH<sub>2</sub>), 30.1 (CH<sub>2</sub>), 35.6 (CH<sub>2</sub>), 38.1 (t, *J* = 26.8 Hz, CH<sub>2</sub>), 122.7 (t, *J* = 241.4 Hz, C), 125.4 (t, *J* = 27.8 Hz, CH), 126.0 (CH), 128.4 (d, *J* = 10.6 Hz, CH), 136.4 (t, *J* = 6.2 Hz, CH), 141.3 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ -91.3 ppm (d, *J* = 34.3 Hz, 2F). HRMS–EI (*m/z*): [M]<sup>+</sup> calcd for C<sub>15</sub>H<sub>20</sub>F<sub>2</sub>, 238.1533; found, 238.1532.

#### Preparation of (*Z*)-{[(4,4-difluorodec-5-en-1-yl)oxy]methyl}benzene [(*Z*)-**1c**].

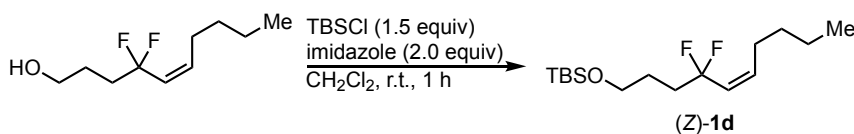


(*Z*)-**1e** was added to a solution of K<sub>2</sub>CO<sub>3</sub> (2.21 g, 16 mmol) in MeOH (100 mL). The mixture was allowed to stir for 1 h, afterwards the solution was filtered and volatiles removed in vacuo. The oily residue was subjected to silica gel chromatography (Hexanes) to afford the corresponding alcohol in 89% yield (1.37 g, 7.1 mmol) as colorless oil.

The alcohol (384.5 mg, 2.0 mmol) was added dropwise to a solution of NaH (95.2 mg, 2.4 mmol) in THF (5.0 mL) at 0 °C. Benzylbromide (282 μL, 2.38 mmol) was then added dropwise and the mixture was allowed to stir at 22 °C for 4 h. Water is added and the resulting biphasic solution extracted with ether. The residue was subjected to silica gel column chromatography (hexanes) to afford (*Z*)-**1c** in 75% yield (421.3 mg, 1.5 mmol) as colorless oil.

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 0.90 (t, *J* = 7.3 Hz, 3H), 1.24–1.44 (m, 4H), 1.74–1.86 (m, 2H), 1.93–2.11 (m, 2H), 2.20–2.29 (m, 2H), 3.52 (t, *J* = 6.3 Hz, 2H), 4.51 (s, 2H), 5.38–5.52 (m, 1H), 5.65–5.76 (m, 1H), 7.23–7.38 (m, 5H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 13.8 (CH<sub>3</sub>), 22.2 (CH<sub>2</sub>), 22.8 (CH<sub>2</sub>), 27.9 (CH<sub>2</sub>), 31.4 (CH<sub>2</sub>), 35.3 (t, *J* = 27.2 Hz, CH<sub>2</sub>), 69.3 (CH<sub>2</sub>), 72.7 (CH<sub>2</sub>), 122.5 (t, *J* = 238.4 Hz, C), 124.5 (t, *J* = 27.2 Hz, CH), 127.4 (CH), 128.2 (CH), 138.0 (t, *J* = 6.2 Hz, CH), 138.3 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ -91.4 (d, *J* = 34.3 Hz, 2F). HRMS–ESI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>17</sub>H<sub>24</sub>OF<sub>2</sub>Na, 305.1687; found, 305.1688.

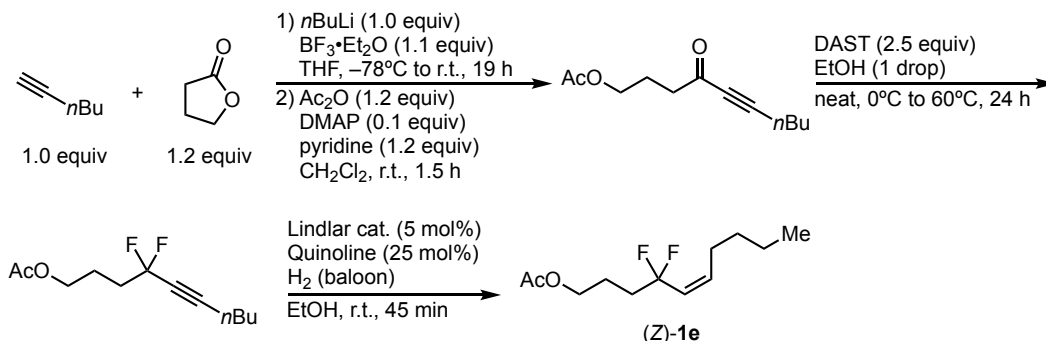
#### Preparation of (*Z*)-*tert*-butyl[(4,4-difluorodec-5-en-1-yl)oxy]dimethylsilane [(*Z*)-**1d**].



The corresponding alcohol (384.5 mg, 2.0 mmol) was added dropwise to a solution of imidazole (272.3 mg, 4.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (5.0 mL) at 22 °C. TBSCl (452.2 mg, 3.0 mmol) was then added to the solution and the mixture was allowed to stir at 22 °C for 1 h. Subsequently water was added and the mixture extracted with ether. The oily residue was subjected to silica gel chromatography (hexanes) to afford (Z)-1d in 58% yield (355.1 mg, 1.2 mmol) as colorless oil.

$^1\text{H}$  NMR (401 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.05 (s, 6H), 0.89–0.92 (m, 12H), 1.28–1.42 (m, 4H), 1.64–1.73 (m, 2H), 1.92–2.05 (m, 2H), 2.21–2.29 (m, 2H), 3.65 (t,  $J = 6.4$  Hz, 2H), 5.40–5.51 (m, 1H), 5.71 (tdt,  $J = 1.8, 7.8, 11.7$  Hz, 1H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.4 ( $\text{CH}_3$ ), 13.9 (CH), 18.2 (C), 22.3 ( $\text{CH}_2$ ), 25.8 ( $\text{CH}_2$ ), 25.9 ( $\text{CH}_3$ ), 28.1 ( $\text{CH}_2$ ), 31.6 ( $\text{CH}_2$ ), 35.1 (t,  $J = 27.3$  Hz, CH), 62.3 ( $\text{CH}_2$ ), 122.7 (t,  $J = 236.4$  Hz, C), 124.8 (t,  $J = 27.2$  Hz, CH), 138.0 (t,  $J = 5.8$  Hz, CH).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -91.1 (s, 2F). HRMS–EI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{32}\text{OF}_2\text{NaSi}$ , 329.2083; found, 329.2085.

### Preparation of (Z)-4,4-difluorodec-5-en-1-yl acetate [(Z)-1e].<sup>3</sup>



In a vacuum dried 500 mL round bottomed flask, *n*-BuLi (1.6 M in hexane, 37.5 mL) was added dropwise to the mixture of hex-1-yne (6.9 mL, 60 mmol) and dry THF (180 mL) at -78 °C under a nitrogen atmosphere. After 30 minutes,  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (66 mL) was added dropwise to the mixture. The mixture was allowed to stir for 30 minutes, then dihydrofuran-2(3*H*)-one was added dropwise. The mixture was allowed to stir and warm to 22 °C over the course of 19 h. Subsequently aqueous  $\text{NH}_4\text{Cl}$  was added at 0 °C and the solution extracted with  $\text{Et}_2\text{O}$  three times. The combined organic layer was dried over  $\text{MgSO}_4$ . After filtration, the volatiles were removed under reduced pressure. The oily residue was subjected to silica gel chromatography (EtOAc/Hexane 0:100–30:70) to give the corresponding  $\gamma$ -hydroxy ketone (5.47 g, 54% yield) as colorless oil.

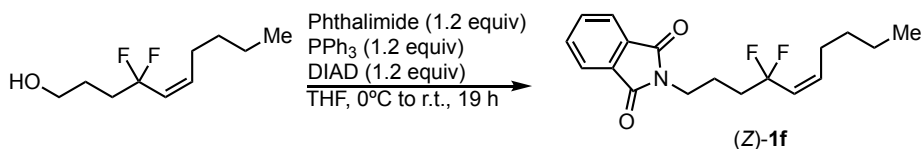
A vacuum dried 200 mL round bottomed flask was charged with  $\gamma$ -hydroxy ketone (30 mmol) and  $\text{CH}_2\text{Cl}_2$  (90 mL). DMAP (367.0 mg, 3.0 mmol), pyridine (2.91 mL, 36 mmol) and  $\text{Ac}_2\text{O}$  (3.4 mL, 36 mmol) were added and the solution was allowed to stir at 22°C for 17 hours. Subsequently water was added and the mixture extracted with  $\text{CH}_2\text{Cl}_2$ . After volatiles were removed in vacuo, the resulting oily residue was subjected to silica gel chromatography (EtOAc/Hexane 0:100–15:85) to give the corresponding ketone (6.97g, quant.) as colorless oil.

To a vacuum dried 100 mL round bottomed flask containing the corresponding ketone (6.97 g, 30 mmol) was added DAST (9.9 mL, 75.0 mmol) in one portion at 0 °C. EtOH (1 drop) was added and the mixture allowed to stir for 22 h at 60 °C.  $\text{CH}_2\text{Cl}_2$  (40 mL) was added and aqueous  $\text{NH}_4\text{Cl}$  at carefully added at 0 °C. The mixture is subsequently extracted with  $\text{CH}_2\text{Cl}_2$ . The oily residue was subjected to silica gel chromatography (EtOAc/Hexane 0:100–7:93) to give the corresponding difluoro compound (3.03 g, 44% yield) as brown oil.

A vacuum dried 300 mL round bottomed flask was charged with Lindlar's catalyst (1.19 g, 0.56 mmol), EtOH (200 mL) and quinoline (331  $\mu\text{L}$ , 2.8 mmol). The difluoro compound (2.73 g, 11.2 mmol) was added and the mixture allowed to stir for 45 minutes under a  $\text{H}_2$  atmosphere ( $\text{H}_2$  balloon). The suspension was then filtered through a Celite pad and volatiles were removed under reduced pressure. The oily residue was subjected to silica gel chromatography (hexanes) to give (*Z*)-**1e** in 86% yield (2.36 g, 9.63 mmol) as colorless oil.

$^1\text{H}$  NMR (392 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.91 (t,  $J$  = 7.1 Hz, 3H), 1.28–1.43 (m, 4H), 1.79–2.05 (m, 4H), 2.06 (s, 3H), 2.20–2.29 (m, 2H), 4.10 (t,  $J$  = 6.5 Hz, 2 H), 5.45 (tdt,  $J$  = 1.7, 12.2, 14.1 Hz, 1H), 5.73 (tdt,  $J$  = 1.9, 7.8, 11.7 Hz, 1H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  13.6 ( $\text{CH}_3$ ), 20.6 ( $\text{CH}_3$ ), 21.7 (t,  $J$  = 4.3 Hz,  $\text{CH}_2$ ), 22.1 ( $\text{CH}_2$ ), 27.8 ( $\text{CH}_2$ ), 31.4 ( $\text{CH}_2$ ), 34.9 (t,  $J$  = 27.3 Hz,  $\text{CH}_2$ ), 122.0 (t,  $J$  = 240.5 Hz, C), 124.2 (t,  $J$  = 27.3 Hz, CH), 138.3 (CH), 170.7 (C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -91.6 (d,  $J$  = 34.3 Hz, 2F). HRMS–ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{12}\text{H}_{20}\text{O}_2\text{F}_2\text{Na}$ , 257.1324; found, 257.1326.

#### Preparation of (*Z*)-2-(4,4-difluorodec-5-en-1-yl)isoindoline-1,3-dione [(*Z*)-**1f**].

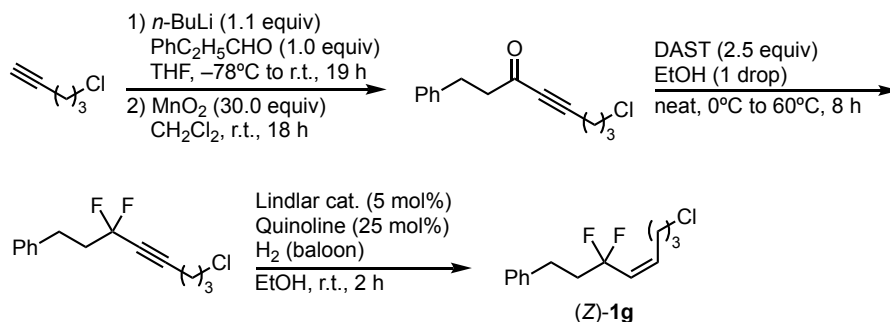


DIAD (467  $\mu\text{L}$ , 2.4 mmol) was added dropwise to a solution of phthalimide (353.1 mg, 2.4 mmol),  $\text{PPh}_3$  (629.5 mg, 2.4 mmol) and the corresponding alcohol (384.5 mg, 2.0 mmol) in THF (5.0 mL) at 0 °C. The mixture was allowed to stir at 22°C for 19 h. and then concentrated under reduced pressure. The oily residue was subjected to silica gel

chromatography (EtOAc/Hexane 4:96–10:90) to afford (*Z*)-**1f** in 70% yield (451.7 mg, 1.4 mmol) as colorless oil.

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 0.87 (t, *J* = 7.1 Hz, 3H), 1.19–1.40 (m, 4H), 1.83–2.06 (m, 4H), 2.16–2.27 (m, 2H), 3.74 (t, *J* = 7.1 Hz, 2H), 5.36–5.49 (m, 1H), 5.71 (tdt, *J* = 1.8, 7.8, 11.8 Hz, 1H), 7.70–7.76 (m, 2H), 7.82–7.89 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 13.6 (CH<sub>3</sub>), 21.6 (t, *J* = 3.8 Hz, CH<sub>2</sub>), 22.0 (CH<sub>2</sub>), 27.7 (CH<sub>2</sub>), 31.2 (CH<sub>2</sub>), 35.7 (t, *J* = 27.8 Hz, CH<sub>2</sub>), 37.1 (CH<sub>2</sub>), 121.9 (t, *J* = 240.8 Hz, C), 122.9 (CH), 124.0 (t, *J* = 26.9 Hz, CH), 131.7 (C), 133.7 (CH), 138.2 (t, *J* = 6.1 Hz, CH), 167.9 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –91.4 (d, *J* = 22.8 Hz, 2F). HRMS–ESI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>21</sub>O<sub>2</sub>NF<sub>2</sub>Na, 344.1433; found, 344.1434.

### Preparation of (*Z*)-(8-chloro-3,3-difluorooct-4-en-1-yl)benzene [(*Z*)-**1g**].<sup>3</sup>



In a vacuum dried 200 mL round bottomed flask, *n*-BuLi (1.6 M hexane solution, 13.8 mL) was added dropwise to the mixture of 5-chloropent-1-yne (2.09 mL, 20 mmol) and dry THF (30 mL) at –78 °C under a nitrogen atmosphere. After the 30 minutes, PhC<sub>2</sub>H<sub>5</sub>CHO (2.63 mL, 20 mL) was added dropwise to the mixture, then the mixture was allowed to warm to room temperature and stirred for 19 hours. The mixture was quenched by aqueous NH<sub>4</sub>Cl at 0 °C and extracted with Et<sub>2</sub>O three times. The combined organic layer was dried over MgSO<sub>4</sub>. After filtration, the solvent was removed by evaporation under reduced pressure. The product was carried forward without purification.

In a vacuum dried 200 mL round bottomed flask, MnO<sub>2</sub> (52.1 g, 600 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (50 mL). The alcohol was then added dropwise to the mixture. After the mixture was allowed to stir for 18 hours at room temperature, CH<sub>2</sub>Cl<sub>2</sub> (100 mL) was added and the mixture was filtered through a Celite pad. After the removal of the solvent, the oily residue was subjected to silica gel chromatography (EtOAc/Hexane 0:100–15:85) to give the corresponding ketone (2.72 g, 58% yield) as colorless oil.

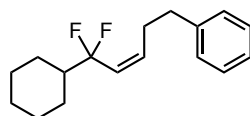
In a vacuum dried 50 mL round bottomed flask, DAST (3.82 mL, 29 mmol) was added by one portion to the corresponding ketone (2.72 g, 11.6 mmol) at 0 °C. EtOH (1 drop) was added and the mixture was allowed to stir for 8 h at 60 °C. CH<sub>2</sub>Cl<sub>2</sub> (20 mL) was then added

and the mixture was carefully quenched by aqueous  $\text{NH}_4\text{Cl}$  at  $0\text{ }^\circ\text{C}$  and extracted with  $\text{CH}_2\text{Cl}_2$ . The oily residue was subjected to silica gel chromatography (hexanes) to give the corresponding difluoro compound (1.76 g, 59% yield) as brown oil.

In a vacuum dried 50 mL round bottomed flask, Lindlar's catalyst (592.3 mg, 0.28 mmol) was dissolved in EtOH (100 mL) and quinoline (165.0  $\mu\text{L}$ , 1.39 mmol) was added to this mixture. The corresponding difluoro compound (1.76 g, 5.6 mmol) was then added to the mixture and stirred for 2 h under  $\text{H}_2$  atmosphere ( $\text{H}_2$  balloon). The mixture was then filtered through a Celite pad and volatiles removed under reduced pressure. The oily residue was subjected to silica gel chromatography (hexanes). (*Z*)-**1g** was obtained in 43% yield (0.62 g, 2.4 mmol) as colorless oil.

$^1\text{H}$  NMR (401 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.90 (dt,  $J = 7.1, 14.0$  Hz, 2H), 2.16–2.30 (m, 2H), 2.40–2.49 (m, 2H), 2.77–2.85 (m, 2H), 3.56 (t,  $J = 6.6$  Hz, 2H), 5.51–5.63 (m, 1H), 5.69–5.78 (m, 1H), 7.17–7.33 (m, 5H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  25.6 ( $\text{CH}_2$ ), 28.5 ( $\text{CH}_2$ ), 32.1 ( $\text{CH}_2$ ), 40.2 (t,  $J = 26.7$  Hz,  $\text{CH}_2$ ), 44.1 ( $\text{CH}_2$ ), 121.9 (t,  $J = 238.8$  Hz, C), 125.8 (t,  $J = 26.7$  Hz, CH), 126.1 (CH), 128.2 (CH), 128.4 (CH), 136.1 (t,  $J = 5.8$  Hz, CH), 140.4 (C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -92.3 (s, 2F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{14}\text{H}_{17}\text{ClF}_2$ , 258.0987; found, 258.0987.

#### (*Z*)-(5-Cyclohexyl-5,5-difluoropent-3-en-1-yl)benzene [(*Z*)-**1h**].



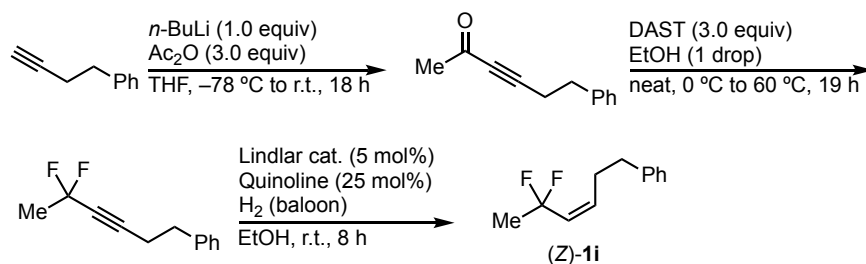
(*Z*)-**1h**

(*Z*)-**1h** was prepared from the corresponding alkyne and aldehyde according to the procedure described above.

$^1\text{H}$  NMR (396 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.03–1.26 (m, 5H), 1.60–1.85 (m, 6H), 2.53–2.62 (m, 2H), 2.71 (t,  $J = 7.5$  Hz, 2H), 5.34–5.48 (m, 1H), 5.71–5.80 (m, 1H), 7.14–7.23 (m, 3H), 7.24–7.33 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  25.6 ( $\text{CH}_2$ ), 25.9 ( $\text{CH}_2$ ), 30.2 ( $\text{CH}_2$ ), 35.7 ( $\text{CH}_2$ ), 45.7 (t,  $J = 25.4$  Hz, CH), 123.7 (t,  $J = 242.9$  Hz, C), 124.1 (t,  $J = 27.3$  Hz, CH), 125.9 (CH), 128.3 (CH), 128.4 (CH), 136.6 (t,  $J = 5.8$  Hz, CH), 141.3 (C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -99.3 (t,  $J = 15.5$  Hz, 2F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{17}\text{H}_{22}\text{F}_2$ , 264.1690; found, 264.1691.

#### Preparation of (*Z*)-(5,5-difluorohex-3-en-1-yl)benzene [(*Z*)-**1i**].<sup>3</sup>





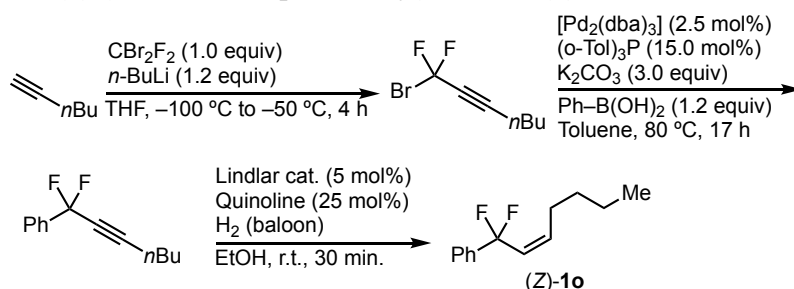
In a vacuum dried 200 mL round bottomed flask, *n*-BuLi (1.6 M hexane solution, 12.5 mL) was added dropwise to the mixture of 3-butyn-1-ylbenzene (2.77 mL, 20 mmol) and dry THF (30 mL) at  $-78\text{ }^{\circ}\text{C}$  under a nitrogen atmosphere. After 30 minutes,  $\text{Ac}_2\text{O}$  (5.62 mL, 60 mmol) was added dropwise to the mixture. The mixture was warmed up to room temperature over the course of 18 h. The mixture was then quenched by aqueous  $\text{NH}_4\text{Cl}$  at  $0\text{ }^{\circ}\text{C}$  and extracted with  $\text{Et}_2\text{O}$  three times. The combined organic layer was dried over  $\text{MgSO}_4$ . After filtration, the solvent was removed by evaporation under reduced pressure. The oily residue was subjected to silica gel chromatography ( $\text{EtOAc/Hexane}$  0:100–10:70) to give the corresponding ketone (3.11 g, 90% yield) as colorless oil.

In a vacuum dried 50 mL round bottomed flask, DAST (5.80 mL, 45.0 mmol) was added by one portion to the corresponding ketone (2.58 g, 15.0 mmol) at  $0\text{ }^{\circ}\text{C}$ . EtOH (1 drop) was added and the mixture was allowed to stir for 19 h at  $60\text{ }^{\circ}\text{C}$ . After that,  $\text{CH}_2\text{Cl}_2$  (40 mL) was added and the mixture was carefully quenched by aqueous  $\text{NH}_4\text{Cl}$  at  $0\text{ }^{\circ}\text{C}$  and extracted with  $\text{CH}_2\text{Cl}_2$ . The oily residue was subjected to silica gel chromatography ( $\text{EtOAc/Hexane}$  0:100–7:93) to give the corresponding difluoro-containing compound (1.28 g, 42% yield) as brown oil.

In a vacuum dried 200 mL round bottomed flask, Lindlar's catalyst (319.3 g, 0.15 mmol) was dissolved in EtOH (60 mL) and quinoline (88.9  $\mu\text{L}$ , 0.75 mmol) was added to this mixture. Then, the corresponding difluoro-containing compound (582.6 mg, 3.0 mmol) was added to this mixture and stirred for 8 h under  $\text{H}_2$  atmosphere ( $\text{H}_2$  balloon). The mixture was then filtered through a Celite pad and concentrated under reduced pressure. The oily residue was subjected to silica gel chromatography (hexanes). (*Z*)-**1i** was obtained in 58% yield (341.6 mg, 1.74 mmol) as colorless oil.

$^1\text{H}$  NMR (399 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.62 (t,  $J = 18.0$  Hz, 3H), 2.55–2.61 (m, 2H), 2.72 (t,  $J = 7.6$  Hz, 2H), 5.48–5.58 (m, 1H), 5.72 (tdt,  $J = 1.8, 7.6, 11.5$  Hz, 1H), 7.18–7.22 (m, 3H), 7.27–7.31 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  25.3 (t,  $J = 29.3$  Hz,  $\text{CH}_3$ ), 29.9 ( $\text{CH}_2$ ), 35.5 ( $\text{CH}_2$ ), 121.4 (t,  $J = 233.1$  Hz, C), 126.0 (CH), 126.2 (t,  $J = 26.9$  Hz, CH), 128.3 (CH), 128.4 (CH), 136.1 (t,  $J = 6.1$  Hz, CH), 141.2 (C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -84.1 (d,  $J = 22.8$  Hz, 2F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{11}\text{H}_{14}\text{F}_2$ , 196.1064; found, 196.1061.

**Preparation of (Z)-(1,1-difluorohept-2-en-1-yl)benzene [(Z)-1o].<sup>1,4</sup>**



To a solution of hex-1-yne (5.7 mL, 50 mmol) in THF (250 mL), a solution of *n*-BuLi (1.6 M in hexane, 31.3 mL) was added dropwise at  $-90\text{ }^{\circ}\text{C}$ . After the mixture had been stirred for 30 min at that temperature, the mixture was cooled to  $-110\text{ }^{\circ}\text{C}$ . Cold  $\text{CF}_2\text{Br}_2$  ( $-78\text{ }^{\circ}\text{C}$ ) (5.5 mL, 60 mmol) was added to the mixture. After the addition was complete, the mixture was allowed to warm to  $-50\text{ }^{\circ}\text{C}$  and stirring for 3 h before being quenched with aqueous  $\text{NH}_4\text{Cl}$  solution. The aqueous layer was extracted with  $\text{Et}_2\text{O}$  and the organic layer was washed with  $\text{H}_2\text{O}$ . The organic layer was dried over  $\text{Na}_2\text{SO}_4$ . After evaporation of the solvent, the oily residue was subjected to distillation to afford the corresponding difluoro compound in 73% yield (7.69 g, 36.5 mmol).

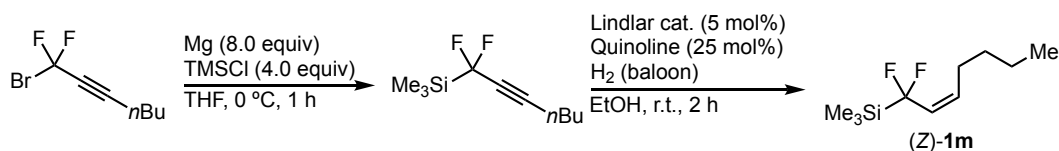
To a solution of  $[\text{Pd}_2(\text{dba})_3]$  (207.1 mg, 0.25 mmol),  $(o\text{-Tol})_3\text{P}$  (456.6 mg, 1.5 mmol),  $\text{K}_2\text{CO}_3$  (4.15 g, 30 mmol) and  $\text{PhB}(\text{OH})_2$  (1.46 g, 12 mmol) in toluene (66 mL), the corresponding difluoro compound (2.15 g, 10 mmol) was added dropwise. Then, the mixture was allowed to stir at  $80\text{ }^{\circ}\text{C}$  for 17 h. The mixture was then extracted with  $\text{Et}_2\text{O}$  and the organic layer was washed with  $\text{H}_2\text{O}$ . The organic layer was dried over  $\text{MgSO}_4$ . After evaporation of the solvent, the oily residue was subjected to silica gel chromatography (hexanes) to afford the coupling product in 53% yield (1.30 g, 6.2 mmol) as colorless oil.

In a vacuum dried 200 mL round bottomed flask, Lindlar's catalyst (564 mg, 0.27 mmol) was dissolved in  $\text{EtOH}$  (100 mL) and quinoline (158  $\mu\text{L}$ , 1.33 mmol) was added to this mixture. Then, the corresponding difluoro compound (1.11 g, 5.3 mmol) was added to this mixture and stirred for 30 minutes under  $\text{H}_2$  atmosphere ( $\text{H}_2$  balloon). The mixture was then filtered through a Celite pad and concentrated under reduced pressure. The oily residue was subjected to silica gel chromatography (hexanes) and gel-permeation chromatography. (Z)-1o was obtained in 53% yield (591.7 mg, 2.8 mmol) as colorless oil.

$^1\text{H}$  NMR (401 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.83 (t,  $J = 7.0$  Hz, 3H), 1.20–1.37 (m, 4H), 2.10–2.20 (m, 2H), 5.72–5.87 (m, 2H), 7.37–7.46 (m, 3H), 7.51–7.58 (m, 2H).  $^{13}\text{C}$  NMR (99 MHz,  $\text{CDCl}_3$ ):  $\delta$  13.8 ( $\text{CH}_3$ ), 22.2 (CH), 28.1 ( $\text{CH}_2$ ), 31.3 ( $\text{CH}_2$ ), 120.0 (t,  $J = 242.3$  Hz, C), 125.3 (t,  $J = 29.6$  Hz, CH), 125.3 (t,  $J = 5.3$  Hz, CH), 128.4 (CH), 129.8 (CH), 138.0 (t,  $J = 28.2$  Hz, C), 138.8 (t,  $J = 7.2$  Hz, CH).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$   $-85.2$  (s, 2F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$

calcd for C<sub>13</sub>H<sub>16</sub>F<sub>2</sub>, 210.1220; found, 210.1224.

#### Preparation of (Z)-(1,1-difluorohept-2-en-1-yl)trimethylsilane [(Z)-1m].<sup>5</sup>

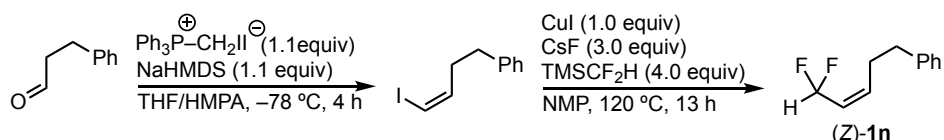


TMSCl (3.03 mL, 24 mmol) was added to a solution of activated Mg turnings (1.17 g, 48 mmol) in THF (60 mL) at 0 °C. After being allowed to stir for 1 h, the propargyl bromide (1.26 g, 6.0 mmol) was added dropwise. The mixture was allowed to stir at 0 °C for 1 h. Subsequently the mixture was filtered, extracted with Et<sub>2</sub>O and the organic layer was washed with H<sub>2</sub>O, then dried over MgSO<sub>4</sub>. After removal of volatiles in vacuo, the oily residue was subjected to silica gel chromatography (hexanes) to afford the silylated compound in 51% yield (627.5 mg, 3.1 mmol) as colorless oil.

In a vacuum dried 100 mL round bottomed flask, Lindlar's catalyst (266.1 mg, 0.125 mmol) was suspended in EtOH (50 mL) and quinoline (74.1 μL, 0.63 mmol) was added to the mixture. The corresponding silylated compound (520.6 mg, 2.5 mmol) was added to the mixture and allowed to stir for 30 minutes under a H<sub>2</sub> atmosphere (H<sub>2</sub> balloon). The mixture was then filtered through a Celite pad and solvent removed under reduced pressure. The oily residue was subjected to silica gel chromatography (hexanes) followed by gel-permeation chromatography. (Z)-1m was obtained in 44% yield (232.0 mg, 1.1 mmol) as colorless oil.

<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>): δ 0.16 (s, 9H), 0.90 (t, *J* = 6.8 Hz, 3H), 1.24–1.43 (m, 4H), 2.15–2.26 (m, 2H), 5.29–5.46 (m, 1H), 5.59–5.70 (m, 1H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ –4.9 (CH<sub>3</sub>), 13.9 (CH<sub>3</sub>), 22.4 (CH), 28.5 (CH<sub>2</sub>), 32.1 (CH<sub>2</sub>), 124.5 (t, *J* = 20.3 Hz, C), 128.9 (t, *J* = 260.6 Hz, CH), 136.2 (t, *J* = 8.5 Hz, CH). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –109.2 (d, *J* = 23.1, 2F). HRMS–FI (*m/z*): [M]<sup>+</sup> calcd for C<sub>10</sub>H<sub>20</sub>F<sub>2</sub>Si, 206.1302; found, 206.1309.

#### Preparation of (Z)-(5,5-difluoropent-3-en-1-yl)benzene [(Z)-1n].<sup>6</sup>



Sodium hexamethyldisilazide (NaHMDS) (3.03 mL, 24 mmol) was added to a solution of the ylide (15.8 g, 29.9 mmol) in THF (110 mL) at 22 °C. After the solution was allowed to cool to –78 °C, hexamethylphosphoric triamide (HMPA) (420 μL) was added. The aldehyde (3.50 mL, 23.9 mmol) was added dropwise and the mixture was allowed to stir at –78 °C for 1 h. Subsequently the solution was allowed to warm to 22 °C and allowed to stir for 1 h. The

mixture was quenched by cold brine, extracted with pentane three times, and the organic layer was washed with H<sub>2</sub>O. After the organic layer was dried over MgSO<sub>4</sub> and evaporation of the solvent, the oily residue was subjected to silica gel chromatography (hexanes) to afford the compound in 21% yield (1.35 g, 5.0 mmol) as colorless oil.

CuI (1.24 g, 6.5 mmol) and CsF (2.96 g, 19.5 mmol) were dissolved in *N*-methylpyrrolidone (NMP) (32.5 mL). The corresponding vinyl iodide (1.82 g, 6.5 mmol) and TMSCF<sub>2</sub>H (3.23 g, 4.0 mmol) were added to the mixture. The mixture was allowed to stir at 120 °C for 17 h and was quenched with aqueous NH<sub>4</sub>Cl solution. The mixture was extracted with Et<sub>2</sub>O and washed with H<sub>2</sub>O three times, then the organic layer was dried over MgSO<sub>4</sub>. After evaporation of the solvent, the oily residue was subjected to silica gel chromatography (hexanes) to afford (*Z*)-**1n** in 39% yield (465.2 mg, 2.5 mmol) as colorless oil.

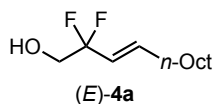
<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>): δ 2.46–2.55 (m, 2H), 2.74 (t, *J* = 7.6 Hz, 2H), 5.54–5.65 (m, 1H), 5.86–5.95 (m, 1H), 6.28 (td, *J* = 6.8, 55.7 Hz, 1H), 7.16–7.24 (m, 3H), 7.28–7.33 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 29.6 (CH<sub>2</sub>), 35.2 (CH<sub>2</sub>), 111.8 (t, *J* = 230.7 Hz, CH), 123.3 (t, *J* = 24.9 Hz, CH), 126.2 (CH), 128.3 (CH), 128.4 (CH), 138.5 (t, *J* = 12.0 Hz, CH), 140.6 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –111.4 (d, *J* = 57.1 Hz, 2F). HRMS–EI (*m/z*): [M]<sup>+</sup> calcd for C<sub>11</sub>H<sub>12</sub>F<sub>2</sub>, 182.0907; found, 182.0907.

## 2.2. (*E*)-Alkene substrates synthesis

### General Procedure for Substrates made through Olefin Metathesis

In a N<sub>2</sub>-filled glove box, an oven-dried 20 mL vial equipped with a magnetic stirring bar was charged with *gem*-difluoromethyl substituted olefin (1.0 equiv.), a terminal olefin cross partner (1.5–2.0 equiv.) and methylene chloride (0.5 M). Ruthenium complex **Ru-1** (5 mol%) was added subsequently, the vial tightly capped and transferred to an oil bath preheated to 50 °C. The mixture was allowed to stir for 12 h after which the solvent was evaporated under reduced pressure, the residue was subjected to silica gel chromatography (hexanes and Et<sub>2</sub>O) to afford the desired product.

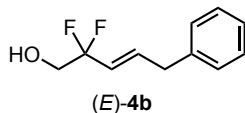
#### (*E*)-2,2-Difluorododec-3-en-1-ol [(*E*)-4a]



Prepared from 2,2-Difluorobut-3-en-1-ol (21.6 mg, 0.20 mmol, 1.0 equiv.)<sup>7</sup> and 1-decene (56 mg, 0.40 mmol, 2.0 equiv.), isolated as colorless oil in 77% yield (34 mg, 0.154 mmol).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 0.88 ppm (3H, t, *J* = 6.8 Hz), 1.28 (12H, td, *J* = 10.3, 8.1, 4.7 Hz), 1.41 (2H, dd, *J* = 9.1, 5.4 Hz), 2.22–2.01 (3H, m), 3.76 (2H, td, *J* = 12.9, 6.0 Hz), 5.75–5.39 (1H, m), 6.18 (1H, dddd, *J* = 16.1, 9.4, 6.8, 2.6 Hz). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 14.0 (CH<sub>3</sub>), 22.6 (CH<sub>2</sub>), 28.3 (CH<sub>2</sub>), 29.1 (CH<sub>2</sub>), 29.2 (CH<sub>2</sub>), 29.3 (CH<sub>2</sub>), 31.8 (CH<sub>2</sub>), 31.9 (CH<sub>2</sub>), 65.0 (t, *J* = 32.3 Hz, CH<sub>2</sub>), 119.3 (t, *J* = 239.2 Hz, CF<sub>2</sub>), 122.1 (t, *J* = 25.4 Hz, CH), 138.5 (t, *J* = 9.0 Hz, CH). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ –106.99 ppm (dt, *J* = 13.5, 2.7 Hz). HRMS (DART) Calcd for C<sub>12</sub>H<sub>26</sub>NOF<sub>2</sub> [M+NH<sub>4</sub>]<sup>+</sup>: 238.1977; Found 238.1975.

#### (*E*)-2,2-Difluoro-5-phenylpent-3-en-1-ol [(*E*)-4b]

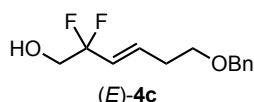


Prepared from 2,2-Difluorobut-3-en-1-ol (21.6 mg, 0.20 mmol, 1.0 equiv.) and allylbenzene (47 mg, 0.4 mmol, 2.0 equiv.), isolated as colorless oil in 55% yield (22 mg, 0.111 mmol).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 2.05 ppm (1H, t, *J* = 6.3 Hz), 3.52 – 3.41 (2H, m), 3.79 (2H, td, *J* = 13.0, 5.1 Hz), 5.63 (1H, dtt, *J* = 15.7, 11.1, 1.7 Hz), 6.37 (1H, dtt, *J* = 15.9, 6.7, 2.6 Hz), 7.20 (2H, d, *J* = 7.3 Hz), 7.26 (1H, t, *J* = 7.0 Hz), 7.33 (2H, t, *J* = 7.5 Hz). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz): δ 38.3 (CH<sub>2</sub>), 65.1 (t, *J* = 32.2 Hz, CH<sub>2</sub>), 119.4 (t, *J* = 239.5 Hz, CF<sub>2</sub>),

123.6 (t,  $J = 25.5$  Hz, CH), 126.7 ( $C_{Ar}$ ), 128.8 ( $C_{Ar}$ ), 128.8 ( $C_{Ar}$ ), 137.0 (t,  $J = 9.1$  Hz, CH), 138.4 ( $C_{Ar}$ ).  $^{19}F$  NMR ( $CDCl_3$ , 376 MHz):  $\delta -106.1$  ppm (m). HRMS (DART) Calcd for  $C_{11}H_{12}OF_2$   $[M]^+$ : 198.0851; Found 198.0841.

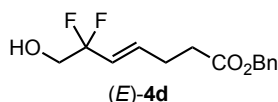
**(*E*)-6-(Benzyloxy)-2,2-difluorohex-3-en-1-ol [(*E*)-4c]**



Prepared from 2,2-Difluorobut-3-en-1-ol (ref for the preparation)<sup>7</sup> (21.6 mg, 0.20 mmol, 1.0 equiv.) and 4-benzyloxybut-1-ene (81 mg, 0.500 mmol, 2.5 equiv.), isolated as colorless oil in 50% yield (25 mg, 0.100 mmol).

$^1H$  NMR (500 MHz,  $CDCl_3$ ):  $\delta$  2.56–2.24 (3H, m), 3.57 (2H, t,  $J=6.5$  Hz), 3.74 (2H, t,  $J = 12.8$  Hz), 4.52 (2H, s), 5.68 (1H, dtt,  $J = 16.0, 11.1, 1.6$  Hz), 6.28–6.13 (1H, m), 7.40–7.24 (5H, m).  $^{13}C$  NMR (101 MHz,  $CDCl_3$ ):  $\delta$  32.5 ( $CH_2$ ), 65.0 (t,  $J = 32.4$  Hz,  $CH_2$ ), 68.7 (d,  $J = 1.8$  Hz,  $CH_2$ ), 73.1 ( $CH_2$ ), 119.3 (t,  $J = 239.8$  Hz,  $CF_2$ ), 124.3 (t,  $J = 25.6$  Hz, CH), 127.9 ( $C_{Ar}$ ), 128.6 ( $C_{Ar}$ ), 134.9 (t,  $J = 9.2$  Hz, CH), 138.1 ( $C_{Ar}$ ).  $^{19}F$  NMR (470 MHz,  $CDCl_3$ ):  $\delta -103.52$  to  $-110.52$  ppm (m). HRMS (DART) Calcd for  $C_{13}H_{20}NO_2F_2$   $[M+NH_4]^+$ : 260.1457; Found 260.1460.

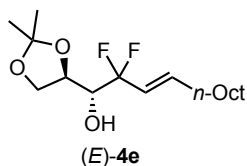
**Benzyl (*E*)-6,6-difluoro-7-hydroxyhept-4-enoate [(*E*)-4d]**



Prepared from 2,2-Difluorobut-3-en-1-ol (ref for the preparation)<sup>7</sup> (18.1 mg, 0.166 mmol, 1.0 equiv.) and 4-benzyloxybut-1-ene (47.3 mg, 0.249 mmol, 1.5 equiv.), isolated as colorless oil in 94% yield (42 mg, 0.155 mmol).

$^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  2.30 – 2.18 ppm (1H, m), 2.56 – 2.40 (4H, m), 3.72 (2H, ddd,  $J = 16.8, 9.4, 4.1$  Hz), 5.13 (2H, s), 5.62 (1H, dtt,  $J = 15.8, 11.0, 1.4$  Hz), 6.26 – 6.09 (1H, m), 7.43 – 7.30 (5H, m).  $^{13}C$  NMR (151 MHz,  $CDCl_3$ ):  $\delta$  27.3 ( $CH_2$ ), 33.1 ( $CH_2$ ), 65.0 (t,  $J = 32.5$  Hz,  $CH_2$ ), 66.6 ( $CH_2$ ), 119.3 (t,  $J = 239.9$  Hz,  $CF_2$ ), 123.8 (t,  $J = 25.7$  Hz, CH), 128.4 (d,  $J = 8.4$  Hz, CH), 128.7 ( $C_{Ar}$ ), 135.9 ( $C_{Ar}$ ), 136.0 ( $C_{Ar}$ ), 172.5 ( $CO_2$ ).  $^{19}F$  NMR (376 MHz,  $CDCl_3$ ):  $\delta -106.48$  ppm (tdd,  $J = 13.0, 10.5, 2.9$  Hz). HRMS (DART) Calcd for  $C_{14}H_{17}O_3F_2$   $[M]^+$ : 271.1140; Found 271.1148.

**Preparation of (*R,E*)-1-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-difluorododec-3-en-1-ol [(*E*)-4e]**



1-Decene (1.51 mL, 8.00 mmol, 4.0 equiv.) and **Ru-1** (63.0 mg, 0.10 mmol, 5 mol%) were added to a solution of (*R*)-1-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-difluorobut-3-en-1-ol **4<sup>8</sup>** (88:12 *d.r.*, 416 mg, 2.00 mmol, 1.0 equiv.) in CH<sub>2</sub>Cl<sub>2</sub> (2.0 mL, 1.0 M). The mixture was heated at 50 °C for 16 h, then was allowed to cool to r.t. and concentrated under reduced pressure. <sup>19</sup>F NMR spectra of the crude mixture showed 75% conversion to the desired product. Purification by silica gel chromatography (petroleum ether:Et<sub>2</sub>O = 9:1 to 5:1) afforded the alkene (*E*)-**4e** as colourless oil in 71% yield (455 mg, 1.42 mmol, 91:9 *d.r.*).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 0.87 ppm (3H, t, *J* = 6.7 Hz), 1.33–1.22 (m, 10H), 1.35 (3H, s), 1.43–1.35 (2H, m), 1.42 (3H, s), 2.18–2.05 (2H, m), 2.66 (1H, d, *J* = 3.8 Hz), 4.07–3.93 (3H, m), 4.26 (1H, td, *J* = 6.6, 3.8 Hz), 5.69–5.54 (1H, m), 6.16 (1H, dddd, *J* = 15.9, 11.8, 5.8, 2.4 Hz). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz): δ 14.2 (CH<sub>3</sub>), 22.8 (CH<sub>2</sub>), 25.4 (CH<sub>2</sub>), 26.4 (CH<sub>2</sub>), 28.5 (CH<sub>2</sub>), 29.2 (CH<sub>2</sub>), 29.3 (CH<sub>2</sub>), 29.5 (CH<sub>2</sub>), 32.0 (CH<sub>2</sub>), 32.1 (CH<sub>2</sub>), 64.8 (dd, *J*<sub>C,F</sub> = 3.8, 1.8 Hz, CH<sub>2</sub>), 73.2 (dd, *J*<sub>C,F</sub> = 29.7, 27.9 Hz), 74.4 (t, *J*<sub>C,F</sub> = 2.4 Hz), 109.0 (CH), 119.5 (dd, *J*<sub>C,F</sub> = 244.2, 241.5 Hz, CF<sub>2</sub>), 122.1 (t, *J*<sub>C,F</sub> = 24.4 Hz, CH), 138.2 (t, *J*<sub>C,F</sub> = 9.1 Hz, CH). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 377 MHz): δ -105.2 (dtd, *J* = 251.3, 10.9, 2.8 Hz), -107.4 ppm (dt, *J* = 251.3, 12.9 Hz). HRMS (MALDI): Calcd for C<sub>17</sub>H<sub>30</sub>F<sub>2</sub>KO<sub>3</sub> [M+K]<sup>+</sup>: 359.1795, Found: 359.1779. Specific rotation: [α]<sub>D</sub><sup>20</sup> = +70.1 (*c* 1.0, CHCl<sub>3</sub>).

### 3. General Borylation and Derivatization Procedures

#### Procedure for the copper(I)-catalyzed enantioselective boryl substitution of (Z)-1a (Scheme 2, Condition A).

An oven-dried reaction vial was charged with copper chloride (2.5 mg, 0.025 mmol), bis(pinacolato)diboron (190.0 mg, 0.75 mmol), (*R,R*)-BenzP\* (7.1 mg, 0.025 mmol) and transferred to an argon filled glove box. NaOMe (40.7 mg, 0.75 mmol) was added, the vial was capped with a rubber septum and removed from the glovebox. The solids were suspended in THF (1.0 mL) and the suspension was allowed to stir for 30 min at 30 °C. (Z)-1 (120.1 mg, 0.50 mmol) was added to the suspension and the mixture was allowed to stir until full consumption of starting material was observed. Subsequently, the mixture was passed through a short plug of silica gel ( $\Phi$ : 10 mm, height of the silica-gel column: 30 mm), eluted with Et<sub>2</sub>O and volatiles removed in vacuo. The resulting oily residue was subjected to silica gel chromatography (Et<sub>2</sub>O/hexane, typically 0:100–5:95) to give the corresponding borylation product **2** as colorless oil. Racemic products were synthesized with (9,9-dimethyl-9*H*-xanthene-4,5-diyl)bis(diphenylphosphane) replacing (*R,R*)-BenzP\*.

#### Procedure for the copper(I)-catalyzed enantioselective boryl substitution of (E)-4a (Scheme 2, Condition B).

In a N<sub>2</sub>-filled glove box, an oven-dried 8 mL vial, equipped with a magnetic stirring bar was charged with CuCl (5 mol%), (*S,S*)-phenyl-bpe (6 mol%), bis(pinacolato)diboron (1.1 equiv.), LiOt-Bu (1.5 equiv.) followed by (*E*)-4a (1.0 equiv.) in toluene solution (0.1 M). The vial was tightly capped and allowed to stir for 14 h at 22 °C. Subsequently the solvent was evaporated under reduced pressure and the oily residue subjected to silica gel chromatography (hexanes and Et<sub>2</sub>O) to afford the desired product. Racemic products were synthesized with 1,3-Bis-(2,6-diisopropylphenyl) imidazolium chloride replacing (*S,S*)-phenyl-bpe.

#### Procedure for the oxidation of the borylation products to allylic alcohols.

An 8 mL vial equipped with a magnetic stirring bar is charged with the borylation product (0.1 mmol) and THF/H<sub>2</sub>O (1:1, 1.0 mL). NaBO<sub>3</sub>•4H<sub>2</sub>O (76.9 mg, 0.5 mmol) was added, and the suspension allowed to stir for 2 hours at 22 °C. The mixture was extracted with Et<sub>2</sub>O and the organic layer was dried over MgSO<sub>4</sub>. After filtration, the residue is subjected to silica gel chromatography (ethyl acetate/hexanes, typically 5:95–15:85) to afford the corresponding alcohol.

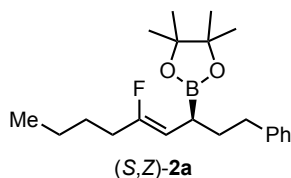
#### Esterification procedure of the borylation product



The alcohol was obtained through the oxidation of the borylation product according to the procedure described above. In a reaction vial, the obtained alcohol (0.1 mmol) and DMAP (0.05 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (2.0 mL). Et<sub>3</sub>N (28.0 μL, 0.2 mmol) and *p*-nitrobenzoyl chloride (27.8 mg, 0.15 mmol) were then added to the mixture at room temperature. After stirred for 3 hours, the mixture was passed through a short silica gel column (Φ: 10 mm, height of the silica-gel column: 30 mm) eluting with Et<sub>2</sub>O. The crude material was purified by silica gel chromatography (ethyl acetate/hexane, typically 0:100–4:96) to give the corresponding ester.

#### 4. Borylation Product Characterizations

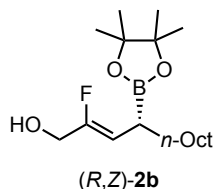
##### **(*S,Z*)-2-(5-Fluoro-1-phenylnon-4-en-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-2a].**



Prepared according to condition A. The reaction was conducted for 2 h with 120.1 mg (0.50 mmol) of (*Z*)-**1a**. The product (*S,Z*)-**2a** was obtained in 93% yield (160.2 mg) with 97/3 e.r. The stereoselectivity of (*S,Z*)-**2a** was determined by GC analysis (*Z*:*E* = 95:5).

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 0.91 (t, *J* = 7.3 Hz, 3H), 1.24 (s, 12H), 1.35 (dq, *J* = 7.2, 14.6 Hz, 2H), 1.43–1.52 (m, 2H), 1.58–1.70 (m, 1H), 1.77–1.89 (m, 1H), 2.11–2.22 (m, 3H), 2.49–2.71 (m, 2H), 4.50 (dd, *J* = 9.4, 38.4 Hz, 1H), 7.13–7.20 (m, 3H), 7.23–7.29 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 13.7 (CH<sub>3</sub>), 19.9 (br, B–CH), 21.8 (CH<sub>2</sub>), 24.56 (CH<sub>3</sub>), 24.63 (CH<sub>3</sub>), 28.4 (CH<sub>2</sub>), 31.7 (d, *J* = 28.3 Hz, CH<sub>2</sub>), 33.2 (CH<sub>2</sub>), 35.4 (CH<sub>2</sub>), 83.1 (C), 105.3 (d, *J* = 16.1 Hz, CH), 125.5 (CH), 128.1 (CH), 128.4 (CH), 142.6 (C), 159.4 (d, *J* = 253.9 Hz, C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –108.9 – –108.7 (m, 1F). HRMS–EI (*m/z*): [M]<sup>+</sup> calcd for C<sub>21</sub>H<sub>32</sub>BFO<sub>2</sub>, 346.2483; found, 346.2481. [α]<sub>D</sub><sup>18.8</sup> +0.35 (*c* 1.60, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IC-3, 2-PrOH/Hexane = 4/96, 0.5 mL/min, 40°C, retention time: 18.88 min [(*Z*)-alcohol major enantiomer], 16.37 min [(*Z*)-alcohol minor enantiomer], and 13.88 min [(*E*)-alcohol major enantiomer]. Minor enantiomer of (*E*)-alcohol was not detected by HPLC analysis.

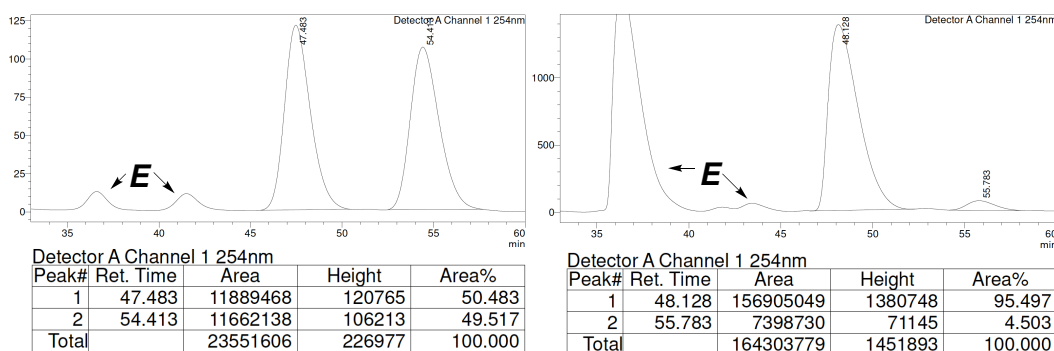
##### **(*R,Z*)-2-Fluoro-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)dodec-2-en-1-ol [(*R,Z*)-2b]**



Prepared according to condition B from (*E*)-**4a** (34 mg, 154 μmol) and isolated as colorless oil in 51% yield (26 mg, 79 μmol).

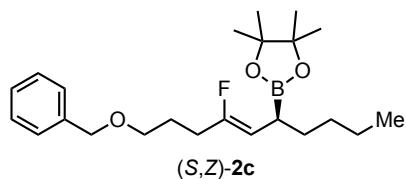
<sup>1</sup>H NMR (CDCl<sub>3</sub>, 600 MHz): δ 0.87 (3H, td, *J* = 7.0, 1.2 Hz), 1.42 – 1.17 (25H, m), 1.53 (1H, tt, *J* = 10.9, 5.1 Hz), 1.73 (1H, d, *J* = 7.2 Hz), 2.14 (1H, q, *J* = 8.7 Hz), 4.11 (2H,

dd,  $J = 16.2, 4.1$  Hz), 4.82 (1H, dd,  $J = 37.1, 9.9$  Hz).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 151 MHz):  $\delta$  14.3 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$ ), 24.8 ( $\text{CH}_3$ ), 24.8 ( $\text{CH}_3$ ), 29.2, 29.4 ( $\text{CH}_2$ ), 29.6 ( $\text{CH}_2$ ), 29.7 ( $\text{CH}_2$ ), 31.1 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 61.7 (d,  $J = 31.9$  Hz,  $\text{CH}_2\text{OH}$ ), 83.5 (CH), 109.6 (d,  $J = 14.5$  Hz CH), 157.1 (d,  $J = 252.5$  Hz, CF).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 376 MHz):  $\delta$  -120.8 ppm (dt,  $J = 37.1, 16.4$  Hz). HRMS (DART-TOF)  $m/z$ :  $\text{C}_{18}\text{H}_{38}\text{BNO}_3\text{F}$   $[\text{M}+\text{NH}_4]^+$ : 346.2923; Found: 346.2927.  $[\alpha]_{\text{D}}^{20} +11.1$  ( $c = 1.0$ ,  $\text{CHCl}_3$ ). Enantiomeric purity of (*R,Z*)-**2b** was determined by HPLC analysis after oxidation of **2b** to the corresponding allylic alcohol followed by benzylation, in comparison with authentic racemic material, during oxidation the double bond isomerized to a 1:1 *E:Z* mixture (ODH column, 97:3 hexanes:*i*PrOH, 0.5 mL/min, 254 nm). (*R*)-enantiomer  $t_{\text{r}} = 48.1$  min, (*S*)-enantiomer  $t_{\text{r}} = 55.8$  min.



| Peak # | Ret. Time  | Area     | Area % | Peak # | Ret. Time | Area      | Area % |
|--------|------------|----------|--------|--------|-----------|-----------|--------|
| 1      | 47.483 min | 11889468 | 50.483 | 1      | 48.128    | 156905049 | 95.497 |
| 2      | 54.413 min | 11662138 | 49.517 | 2      | 55.783    | 71145     | 4.503  |

(*S,Z*)-2-(10-(Benzyloxy)-7-fluorodec-6-en-5-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-**2c**].

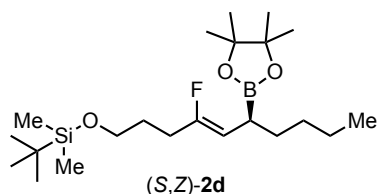


Prepared according to condition A. The reaction was conducted for 1.5 h with 141.1 mg (0.50 mmol) of (*Z*)-**1c**. The product (*S,Z*)-**2c** was obtained in 92% yield (180.0 mg) with 96:4 e.r. The stereoselectivity of (*S,Z*)-**2c** was determined by GC analysis (*Z:E* = 94:6).

$^1\text{H}$  NMR (392 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.87 (t,  $J = 6.9$  Hz, 3H), 1.18–1.55 (m, 18H), 1.80 (quin,  $J = 7.0$  Hz, 2H), 2.08 (q,  $J = 8.0$  Hz, 1H), 2.26 (dt,  $J = 7.4, 17.9$  Hz, 2H), 3.50 (t,  $J = 6.3$  Hz, 2H), 4.47 (dd,  $J = 9.8, 38.4$  Hz, 1H), 4.50 (s, 2H), 7.27–7.37 (m, 5H).  $^{13}\text{C}$  NMR (99 MHz,  $\text{CDCl}_3$ ):  $\delta$  14.0 ( $\text{CH}_3$ ), 19.9 (br, B–CH), 22.5 ( $\text{CH}_2$ ), 24.5 ( $\text{CH}_3$ ), 24.6 ( $\text{CH}_3$ ), 26.5 ( $\text{CH}_2$ ), 28.7 (d,  $J = 29.3$  Hz,  $\text{CH}_2$ ), 30.7 ( $\text{CH}_2$ ), 31.2 ( $\text{CH}_2$ ), 69.0 ( $\text{CH}_2$ ), 72.8 ( $\text{CH}_2$ ), 82.9 (C), 106.3 (d,  $J =$

17.0 Hz, CH), 127.4 (CH), 127.5 (CH), 128.2 (CH), 138.4 (C), 158.2 (d,  $J = 253.0$  Hz,  $\text{CF}_2$ ).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -110.0 to -110.2 (m, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{23}\text{H}_{37}\text{O}_3\text{BF}$ , 391.2819; found, 391.2818.  $[\alpha]_{\text{D}}^{21.9} +1.2$  ( $c$  1.02,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 2/98, 0.5 mL/min, 40°C, retention time: 34.69 min [(*Z*)-alcohol major enantiomer], 31.64 min [(*Z*)-alcohol minor enantiomer] and 27.26 min [(*E*)-alcohol major enantiomer]. Minor enantiomer of (*E*)-alcohol was not detected by HPLC analysis.

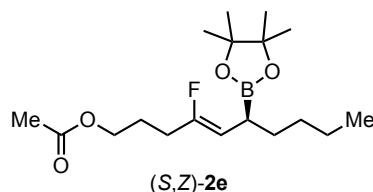
**(*S,Z*)-tert-Butyl{(4-fluoro-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)dec-4-en-1-yl)oxy}dimethylsilane [(*S,Z*)-2d].**



Prepared according to condition A. The reaction was conducted for 1.5 h with 157.4 mg (0.51 mmol) of (*Z*)-1d. The product (*S,Z*)-2d was obtained in 99% yield (200.1 mg) with 96:4 e.r. The stereoselectivity of (*S,Z*)-2d was determined by GC analysis (*Z*:*E* = 95:5).

$^1\text{H}$  NMR (392 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.04 (s, 6H), 0.84–0.93 (m, 12H), 1.18–1.55 (m, 18H), 1.69 (quin,  $J = 6.9$  Hz, 2H), 2.08 (q,  $J = 8.0$  Hz, 1H), 2.22 (dt,  $J = 7.4, 17.9$  Hz, 2H), 3.62 (t,  $J = 6.3$  Hz, 2H), 4.46 (dd,  $J = 9.8, 38.4$  Hz, 1H).  $^{13}\text{C}$  NMR (99 MHz,  $\text{CDCl}_3$ ):  $\delta$  -5.4 ( $\text{CH}_3$ ), 14.0 ( $\text{CH}_3$ ), 18.3 (C), 20.0 (br, B-CH), 22.6 ( $\text{CH}_2$ ), 24.59 ( $\text{CH}_3$ ), 24.62 ( $\text{CH}_3$ ), 25.9 ( $\text{CH}_3$ ), 28.4 (d,  $J = 28.8$  Hz,  $\text{CH}_2$ ), 29.5 ( $\text{CH}_2$ ), 30.8 ( $\text{CH}_2$ ), 31.3 ( $\text{CH}_2$ ), 61.9 ( $\text{CH}_2$ ), 83.0 (C), 106.1 (d,  $J = 16.3$  Hz, CH), 158.6 (d,  $J = 252.9$  Hz, C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -109.5 to -109.7 (m, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{44}\text{O}_3\text{BFNaSi}$ , 437.3033; found, 437.3030.  $[\alpha]_{\text{D}}^{21.7} +1.1$  ( $c$  1.55,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IC-3, 2-PrOH/Hexane = 1.5/98.5, 0.5 mL/min, 40°C, retention time: 15.97 min [(*Z*)-alcohol major enantiomer], 14.87 min [(*Z*)-alcohol minor enantiomer], 12.59 min [(*E*)-alcohol major enantiomer], and 13.46 min [(*E*)-alcohol minor enantiomer].

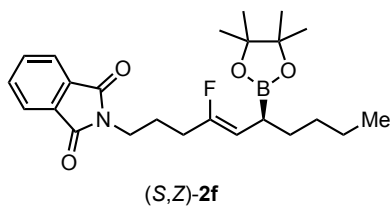
**(*S,Z*)-4-Fluoro-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)dec-4-en-1-yl acetate [(*S,Z*)-2e].**



Prepared according to condition A. The reaction was conducted for 2 h with 117.2 mg (0.50 mmol) of (*Z*)-1e. The product (*S,Z*)-2e was obtained in 90% yield (153.2 mg) with 96:4 e.r. The stereoselectivity of (*S,Z*)-2e was determined by GC analysis (*Z*:*E* = 90:10).

<sup>1</sup>H NMR (396 MHz, CDCl<sub>3</sub>): δ 0.87 (t, *J* = 7.0 Hz, 3H), 1.16–1.56 (m, 18H), 1.82 (quin, *J* = 7.0 Hz, 2H), 2.05–2.12 (m, 4H), 2.24 (dt, *J* = 7.5, 17.9 Hz, 2H), 4.09 (t, *J* = 6.6 Hz, 2H), 4.50 (dd, *J* = 7.5, 17.9 Hz, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 13.9 (CH<sub>3</sub>), 19.9 (br, B–CH), 20.8 (CH<sub>3</sub>), 22.5 (CH<sub>2</sub>), 24.5 (CH<sub>3</sub>), 25.4 (CH<sub>2</sub>), 28.5 (d, *J* = 28.7 Hz, CH<sub>2</sub>), 30.7 (CH<sub>2</sub>), 31.2 (CH<sub>2</sub>), 63.3 (CH<sub>2</sub>), 83.0 (C), 106.8 (d, *J* = 16.3 Hz, CH), 157.4 (d, *J* = 252.9 Hz, C), 170.9 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –110.6 (t, *J* = 22.8 Hz, 1F). HRMS–EI (*m/z*): [*M*]<sup>+</sup> calcd for C<sub>18</sub>H<sub>32</sub>O<sub>4</sub>BF, 342.2381; found, 342.2382. [α]<sub>D</sub><sup>21.8</sup> +1.2 (*c* 1.09, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 4/96, 0.5 mL/min, 40 °C, retention time: 36.06 min [(*Z*)-alcohol major enantiomer], 35.01 min [(*Z*)-alcohol minor enantiomer] and 53.45 min [(*E*)-alcohol major enantiomer]. Minor enantiomer of (*E*)-alcohol was not detected by HPLC analysis.

**(*S,Z*)-2-{4-Fluoro-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)dec-4-en-1-yl}isoindolin-1,3-dione [(*S,Z*)-2f].**

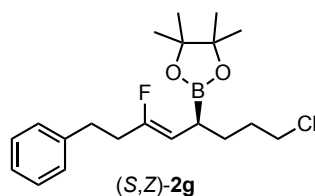


Prepared according to condition A. The reaction was conducted for 1.5 h with 160.0 mg (0.50 mmol) of (*Z*)-1f. The product (*S,Z*)-2f was obtained in 56% yield (119.4 mg) with 95:5 e.r. The stereoselectivity of (*S,Z*)-2f was determined by GC analysis (*Z*:*E* = 96:4).

<sup>1</sup>H NMR (396 MHz, CDCl<sub>3</sub>): δ 0.87 (t, *J* = 5.8 Hz, 3H), 1.01–1.54 (m, 18H), 1.87 (quin, *J* = 7.0 Hz, 2H), 2.07 (q, *J* = 8.0 Hz, 1H), 2.22 (dt, *J* = 8.0, 16.0 Hz, 2H), 3.72 (t, *J* = 7.2 Hz, 2H), 4.52 (dd, *J* = 9.6, 38.5 Hz, 1H), 7.65–7.73 (m, 2H), 7.78–7.86 (m, 2H). <sup>13</sup>C NMR (99

MHz, CDCl<sub>3</sub>):  $\delta$  14.0 (CH<sub>3</sub>), 19.8 (br, B–CH), 22.5 (CH<sub>2</sub>), 24.5 (CH<sub>3</sub>), 24.6 (CH<sub>3</sub>), 25.5 (CH<sub>2</sub>), 29.6 (d,  $J$  = 28.3 Hz, CH<sub>2</sub>), 30.7 (CH<sub>2</sub>), 31.2 (CH<sub>2</sub>), 37.2 (CH<sub>2</sub>), 83.0 (C), 106.7 (d,  $J$  = 17.0 Hz, CH), 123.1 (CH), 132.0 (C), 133.8 (CH), 157.4 (d,  $J$  = 255.0 Hz, C), 168.2 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>):  $\delta$  –109.9 to –110.1 (m, 1F). HRMS–EI ( $m/z$ ): [M]<sup>+</sup> calcd for C<sub>24</sub>H<sub>33</sub>O<sub>4</sub>BFN, 429.2491; found, 429.2475. [ $\alpha$ ]<sub>D</sub><sup>21.7</sup> +0.66 ( $c$  1.14, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the boryl group. Daicel CHIRALPAK® IE-3, 2-PrOH/Hexane = 1.5/98.5, 0.5 mL/min, 40 °C, retention time: 32.78 min [(*Z*)-**2f** major enantiomer], 29.56 min [(*Z*)-**2f** minor enantiomer], 25.84 min [(*E*)-**2f** major enantiomer] and 27.53 min [(*E*)-**2f** minor enantiomer].

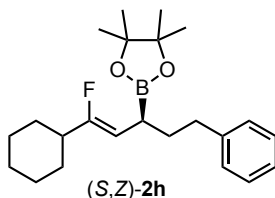
**(*S,Z*)-2-(1-Chloro-6-fluoro-8-phenyloct-5-en-4-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-**2g**].**



Prepared according to condition A. The reaction was conducted for 1 h with 129.4 mg (0.50 mmol) of (*Z*)-**1g**. The product (*S,Z*)-**2g** was obtained in 93% yield (170.0 mg) with 93.5:6.5 e.r. The stereoselectivity of (*S,Z*)-**2g** was determined by GC analysis (*Z*:*E* = 94:6).

<sup>1</sup>H NMR (396 MHz, CDCl<sub>3</sub>):  $\delta$  1.23 (s, 12H), 1.34–1.46 (m, 1H), 1.58–1.73 (m, 3H), 2.08 (td,  $J$  = 5.7, 9.6 Hz, 1H), 2.36–2.56 (m, 2H), 2.80 (t,  $J$  = 7.7 Hz, 2H), 3.47 (t,  $J$  = 6.5 Hz, 2H), 4.42 (dd,  $J$  = 10.1, 38.2 Hz, 1H), 7.16–7.22 (m, 3H), 7.24–7.31 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>):  $\delta$  19.2 (br, B–CH), 24.60 (CH<sub>3</sub>), 24.63 (CH<sub>3</sub>), 28.2 (CH<sub>2</sub>), 31.8 (CH<sub>2</sub>), 32.6 (CH<sub>2</sub>), 33.9 (d,  $J$  = 27.7 Hz, CH<sub>2</sub>), 45.0 (CH<sub>2</sub>), 83.0 (C), 105.9 (d,  $J$  = 15.3 Hz, CH), 126.0 (CH), 128.3 (CH), 128.4 (CH), 140.8 (C), 158.5 (d,  $J$  = 253.0 Hz, C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>):  $\delta$  –110.2 to –110.0 (m, 1F). HRMS–ESI ( $m/z$ ): [M+Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>29</sub>O<sub>2</sub>BClFNa, 389.1829; found, 389.1831. [ $\alpha$ ]<sub>D</sub><sup>17.7</sup> +1.3 ( $c$  0.94, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 5/95, 0.5 mL/min, 40°C, retention time: 21.83 min [(*Z*)-alcohol major enantiomer], 27.77 min [(*Z*)-alcohol minor enantiomer], 17.53 min [(*E*)-alcohol major enantiomer], and 23.13 min [(*E*)-alcohol minor enantiomer].

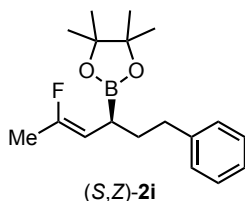
**(*S,Z*)-2-(1-cyclohexyl-1-fluoro-5-phenylpent-1-en-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-2h].**



Prepared according to condition A. The reaction was conducted for 1 h with 132.2 mg (0.50 mmol) of (*Z*)-1h. The product (*S,Z*)-2h was obtained in 95% yield (176.0 mg) with 97:5 e.r. The stereoselectivity of (*S,Z*)-2h was determined by GC analysis (*Z:E* = >98:2).

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 1.11–1.31 (m, 18H), 1.56–1.69 (m, 2H), 1.71–1.88 (m, 4H), 2.00–2.17 (m, 2H), 2.47–2.70 (m, 2H), 4.46 (dd, *J* = 9.6, 39.4 Hz, 1H), 7.12–7.19 (m, 3H), 7.22–7.28 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 19.7 (br, B–CH), 24.5 (CH<sub>3</sub>), 24.6 (CH<sub>3</sub>), 25.8 (CH<sub>2</sub>), 26.0 (CH<sub>2</sub>), 30.1 (CH<sub>2</sub>), 33.2 (CH<sub>2</sub>), 35.4 (CH<sub>2</sub>), 40.5 (d, *J* = 26.4 Hz, CH), 83.1 (C), 103.0 (d, *J* = 17.0 Hz, CH), 125.5 (CH), 128.1 (CH), 128.4 (CH), 142.6 (C), 163.6 (d, *J* = 255.0 Hz, C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –112.7 (d, *J* = 22.8 Hz, 1F). HRMS–EI (*m/z*): [M]<sup>+</sup> calcd for C<sub>23</sub>H<sub>34</sub>O<sub>2</sub>BF, 372.2640; found, 372.2639. [α]<sub>D</sub><sup>20.6</sup> +0.44 (*c* 0.90, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 5/95, 0.5 mL/min, 40°C, retention time: 17.27 min [(*Z*)-alcohol major enantiomer], 18.22 min [(*Z*)-alcohol minor enantiomer].

**(*S,Z*)-2-(5-Fluoro-1-phenylhex-4-en-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-2i].**

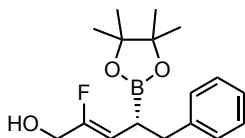


Prepared according to condition A. The reaction was conducted for 4 h with 99.6 mg (0.50 mmol) of (*Z*)-1i. The product (*S,Z*)-2i was obtained in 86% yield (132.2 mg) with 99:1 e.r. The stereoselectivity of (*S,Z*)-2i was determined by <sup>1</sup>H NMR analysis (*Z:E* = 83:17).

<sup>1</sup>H NMR (396 MHz, CDCl<sub>3</sub>): δ 1.24 (s, 12H), 1.59–1.70 (m, 1H), 1.77–1.95 (m, 4H), 2.15 (q, *J* = 8.3 Hz, 1H), 2.47–2.76 (m, 2H), 4.51 (dd, *J* = 9.5, 37.6 Hz, 1H), 7.13–7.21 (m, 3H), 7.23–7.31 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 17.9 (d, *J* = 30.6 Hz, CH<sub>3</sub>), 20.1 (br, B–CH), 24.6 (CH<sub>3</sub>), 24.7 (CH<sub>3</sub>), 33.2 (CH<sub>2</sub>), 35.4 (CH<sub>2</sub>), 83.1 (C), 105.8 (d, *J* = 16.3 Hz, CH),

125.5 (CH), 128.2 (CH), 128.4 (CH), 142.6 (C), 156.0 (d,  $J = 251.0$  Hz, C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta -102.4 - -102.1$  (m, 1F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{18}\text{H}_{26}\text{O}_2\text{BF}$ , 303.2046; found, 303.2047.  $[\alpha]_{\text{D}}^{21.6} +0.07$  ( $c$  1.00,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding ester after oxidation of the boryl group and esterification of the alcohol. Daicel CHIRALPAK® IE-3, 2-PrOH/Hexane = 3/97, 0.5 mL/min, 40°C, retention time: 26.46 min [(*Z*)-ester major enantiomer], 23.30 min [(*Z*)-ester minor enantiomer], 16.87 min [(*E*)-ester major enantiomer], and 15.43 min [(*E*)-ester minor enantiomer].

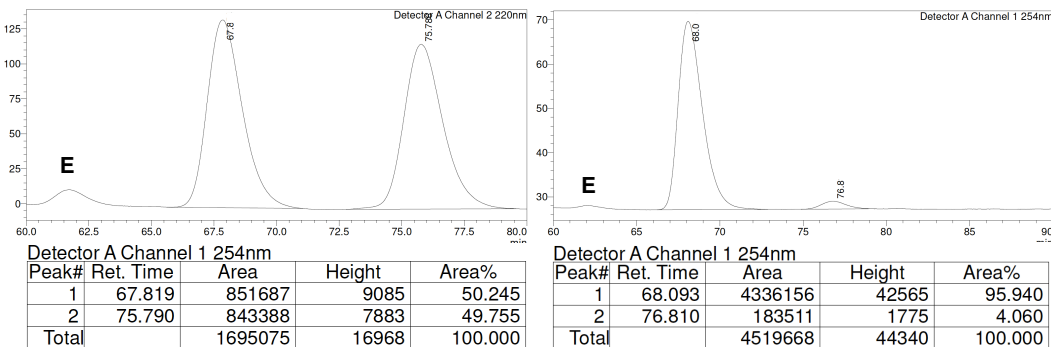
**(*R,Z*)-2-Fluoro-5-phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-2-en-1-ol**  
**[(*R,Z*)-2j].**



(*R,Z*)-2j

Prepared according to conditions B from (*E*)-4b (67 mg, 338  $\mu\text{mol}$ ) and isolated as colorless oil in 61% yield (63 mg, 206  $\mu\text{mol}$ ).

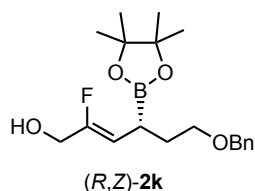
$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  1.17 (6H, s), 1.19 (6H, s), 1.25 (1H, br s), 2.53 (1H, q,  $J = 8.5$  Hz), 2.72 (1H, dd,  $J = 13.8, 8.5$  Hz), 2.88 (1H, dd,  $J = 13.8, 7.5$  Hz), 4.05 (2H, d,  $J = 15.2$  Hz), 4.84 (1H, dd,  $J = 36.9, 9.6$  Hz), 7.16 (1H, t,  $J = 7.1$  Hz), 7.20 (2H, d,  $J = 6.7$  Hz), 7.27–7.22 (2H, m).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150 MHz):  $\delta$  24.8 ( $\text{CH}_3$ ), 29.9 ( $\text{CH}_3$ ), 36.9 ( $\text{CH}_2$ ), 61.6 (d,  $J = 31.8$  Hz,  $\text{CH}_2$ ), 83.7 (C), 108.6 (d,  $J = 14.1$  Hz, CH), 126.1 (C), 128.2 (C), 129.0 (C), 141.2 (C), 157.4 (d,  $J = 254.0$  Hz, CF).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 376 MHz):  $\delta -119.01$  (dt,  $J = 35.5, 16.3$  Hz). HRMS (DART-TOF)  $m/z$ :  $[\text{M}+\text{NH}_4]^+$  Calcd for  $\text{C}_{17}\text{H}_{28}\text{BNO}_3\text{F}$   $[\text{M}+\text{NH}_4]^+$ : 324.2141; Found: 324.2153.  $[\alpha]_{\text{D}}^{20} +18.13$  ( $c = 0.5, \text{CH}_2\text{Cl}_2$ ). Enantiomeric purity of 2j was determined by HPLC analysis after oxidation of 2j to the corresponding allylic alcohol in comparison with authentic racemic material (ADH column, 95:5 hexanes:*i*PrOH, 0.5 mL/min, 254 nm). (*R*)-enantiomer  $t_{\text{r}} = 68.1$  min, (*S*)-enantiomer  $t_{\text{r}} = 76.8$  min.





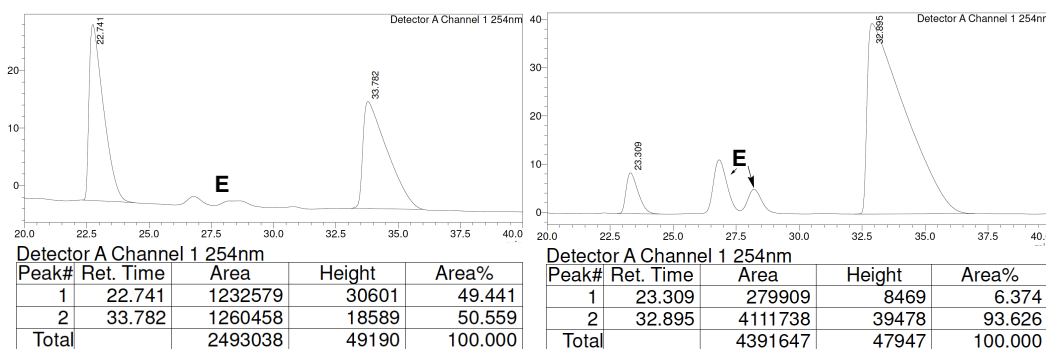
| Peak # | Ret. Time  | Area   | Area % | Peak # | Ret. Time | Area    | Area % |
|--------|------------|--------|--------|--------|-----------|---------|--------|
| 1      | 67.819 min | 851687 | 50.245 | 1      | 68.093    | 4336156 | 95.940 |
| 2      | 75.790 min | 843388 | 49.755 | 2      | 76.810    | 183511  | 4.060  |

**(*R,Z*)-6-(Benzyloxy)-2-fluoro-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hex-2-en-1-ol**  
**1 [(*R,Z*)-2k]**



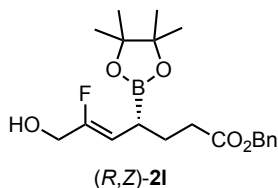
Prepared according to conditions B from (*E*)-**4c** (28 mg 122  $\mu$ mol) and isolated as colorless oil in 52% yield (22 mg, 63  $\mu$ mol).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  1.21 (12H, s), 1.22 (1H, m, overlapping), 1.69 (1H, ddt,  $J = 20.6, 13.2, 6.3$  Hz), 1.92 (1H, dq,  $J = 13.7, 6.9$  Hz), 2.32–2.22 (1H, m), 3.47 (2H, m), 4.09 (2H, d,  $J = 16.2$  Hz), 4.49 (2H, s), 4.84 (1H, dd,  $J = 36.9, 10.0$  Hz), 7.37–7.29 (5H, m).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 126 MHz):  $\delta$  24.77 ( $\text{CH}_3$ ), 24.81 ( $\text{CH}_3$ ), 30.9 ( $\text{CH}_2$ ), 34.8 (CH), 58.5 ( $\text{CH}_2$ ), 61.6 (d,  $J = 32.0$  Hz,  $\text{CH}_2$ ), 69.4 (C), 73.0 (C), 108.8 (d,  $J = 14.3$  Hz, CH), 127.6 (C), 127.9 (C), 128.4 (C), 138.7 (C), 157.5 (d,  $J = 253.6$  Hz, CF).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 376 MHz):  $\delta$  –119.93 (dt,  $J = 37.3, 16.2$  Hz). HRMS (DART-TOF)  $m/z$ :  $[\text{M}]^+$  Calcd for  $\text{C}_{19}\text{H}_{29}\text{BF}_1\text{O}_4^+$  = 351.2137, Found 351.2134.  $[\alpha]_{\text{D}}^{20} +9.5$  ( $c = 1.0, \text{CHCl}_3$ ). Enantiomeric purity of **2k** was determined by HPLC analysis after oxidation of **2k** to the corresponding allylic alcohol in comparison with authentic racemic material (OZ3 column, 92:8 hexanes:*i*PrOH, 0.5 mL/min, 254 nm). (*R*)-enantiomer  $t_{\text{r}} = 23.3$  min, (*S*)-enantiomer  $t_{\text{r}} = 32.9$  min.



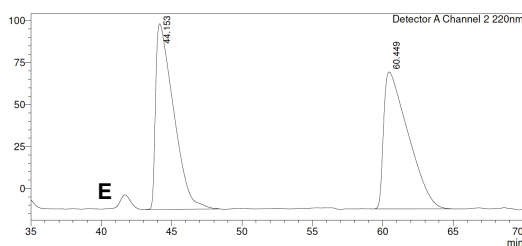
| Peak # | Ret. Time  | Area    | Area % | Peak # | Ret. Time | Area    | Area % |
|--------|------------|---------|--------|--------|-----------|---------|--------|
| 1      | 22.741 min | 1232579 | 49.441 | 1      | 23.309    | 279909  | 6.374  |
| 2      | 33.782 min | 1260458 | 50.559 | 2      | 32.895    | 4111738 | 93.626 |

**(R)-Benzyl-(Z)-6-fluoro-7-hydroxy-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hept-5-enoate [(R,Z)-2I]**

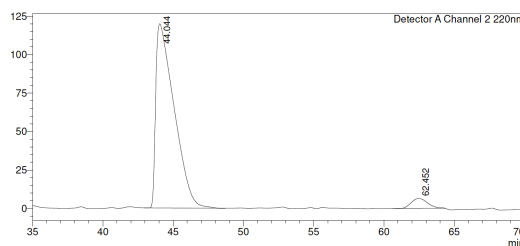


Prepared according to conditions B from (*E*)-**4d** (28 mg, 100  $\mu$ mol) and isolated as colorless oil in 63% yield (24 mg, 63  $\mu$ mol).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  1.23 ppm (12H, s), 1.27 (1H, s), 1.72 (1H, dtd,  $J=13.3, 9.0, 6.3$  Hz), 1.94 (1H, ddt,  $J=12.6, 9.3, 6.2$  Hz), 2.15 (1H, td,  $J=9.7, 5.8$  Hz), 2.49 – 2.28 (2H, m), 4.09 (2H, dd,  $J=15.9, 5.8$  Hz), 4.80 (1H, dd,  $J=36.5, 10.2$  Hz), 5.10 (2H, s), 7.40–7.28 (5H, m).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 151 MHz):  $\delta$  24.7 ( $\text{CH}_3$ ), 24.8 ( $\text{CH}_3$ ), 25.0 ( $\text{CH}_2$ ), 26.0 ( $\text{CH}_2$ ), 33.8 (CH), 61.5 (d,  $J=32.2$  Hz,  $\text{CH}_2$ ), 66.3 ( $\text{CH}_2$ ), 83.7 (C), 108.0 (d,  $J=14.3$  Hz, CH), 128.3 (C), 128.3 (C), 128.7 (C), 136.2 (C), 158.0 (d,  $J=254.2$  Hz, CF), 173.5 (C).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 376 MHz):  $\delta$  -119.51 (dt,  $J=36.4, 15.8$  Hz). HRMS (DART-TOF)  $m/z$ :  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{20}\text{H}_{29}\text{F}_1\text{O}_5\text{B}^+$  = 379.20866; Found 379.20804.  $[\alpha]_{\text{D}}^{20} +3.1$  ( $c = 0.6$ ,  $\text{CHCl}_3$ ). Enantiomeric purity of **2I** was determined by HPLC analysis after oxidation of **2I** to the corresponding allylic alcohol in comparison (OZ3 column, 92:8 hexanes:*i*PrOH, 0.5 mL/min, 220 nm). (*R*)-enantiomer  $t_{\text{r}} = 44.0$  min, (*S*)-enantiomer  $t_{\text{r}} = 62.4$  min.



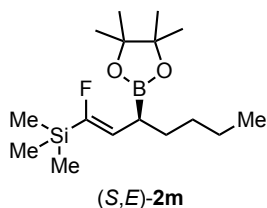
| Peak# | Ret. Time | Area     | Area%   | Height |
|-------|-----------|----------|---------|--------|
| 1     | 44.153    | 9818011  | 49.849  | 110558 |
| 2     | 60.449    | 9877358  | 50.151  | 81580  |
| Total |           | 19695369 | 100.000 | 192138 |



| Peak# | Ret. Time | Area     | Area%   | Height |
|-------|-----------|----------|---------|--------|
| 1     | 44.044    | 10869602 | 95.470  | 119627 |
| 2     | 62.452    | 515785   | 4.530   | 6371   |
| Total |           | 11385386 | 100.000 | 125998 |

| Peak # | Ret. Time  | Area    | Area % | Peak # | Ret. Time | Area    | Area % |
|--------|------------|---------|--------|--------|-----------|---------|--------|
| 1      | 44.153 min | 9818011 | 49.849 | 1      | 44.044    | 1086902 | 95.470 |
| 2      | 60.449 min | 9877358 | 50.151 | 2      | 62.452    | 515785  | 4.530  |

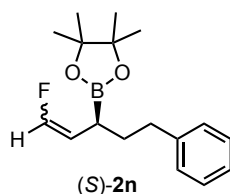
**(S,E)-{1-Fluoro-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hept-1-en-1-yl}trimethylsilane [(S,E)-2m].**



Prepared according to conditions A. The reaction was conducted for 24 h with 103.2 mg (0.50 mmol) of (Z)-1m. The product (S,E)-2m was obtained in 30% yield (46.8 mg) with 88.5:11.5 e.r. The stereoselectivity of (S,E)-2m was determined by GC analysis (*E*:*Z* = >98:2).

<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>): δ 0.13 (s, 9H), 0.87 (t, *J* = 6.8 Hz, 3H), 1.17–1.44 (m, 18H), 2.27 (q, *J* = 8.2 Hz, 1H), 5.00 (dd, *J* = 9.2, 50.1 Hz, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ –2.5 (CH<sub>3</sub>), 14.0 (CH<sub>3</sub>), 20.7 (br, B–CH), 22.6 (CH<sub>2</sub>), 24.5 (CH<sub>3</sub>), 30.6 (CH<sub>2</sub>), 31.4 (CH<sub>2</sub>), 83.0 (C), 122.9 (d, *J* = 4.7 Hz, CH), 167.1 (d, *J* = 274.0 Hz, C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –106.4 (s, 1F). HRMS–ESI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>16</sub>H<sub>32</sub>O<sub>2</sub>BFNaSi, 337.2144; found, 337.2142. [α]<sub>D</sub><sup>21.9</sup> +1.6 (*c* 1.27, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IE-3, 2-PrOH/Hexane = 3/97, 0.5 mL/min, 40°C, retention time: 9.23 min [(*E*)-alcohol major enantiomer], 9.62 min [(*E*)-alcohol minor enantiomer].

**(S)-2-(1-Fluoro-5-phenylpent-1-en-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(S)-2n].**

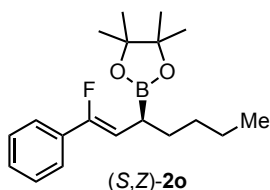


Prepared according to conditions A. The reaction was conducted for 1.5 h with 99.1 mg (0.50 mmol) of (Z)-1n. The product (S)-2n was obtained in 95% yield (137.5 mg) with 94:6 e.r. The stereoselectivity of (S)-2n was determined by GC analysis (*Z*:*E* = 50:50).

<sup>1</sup>H NMR (396 MHz, \* indicates signals of the isomer, CDCl<sub>3</sub>): δ 1.24 (s, 12H), 1.60–1.73 (m, 2H), 1.79–1.93\* (m, 2H), 2.27 (td, *J* = 6.2, 9.5 Hz, 1H), 2.50–2.73 (m, 2H), 4.77 (ddd, *J* = 5.0, 10.4, 43.7 Hz, 1H), 5.32–5.42\* (m, 1H), 6.38–6.44 (m, 1H), 6.60–6.66\* (m, 1H), 7.14–7.20 (m, 3H), 7.24–7.30 (m, 2H). <sup>13</sup>C NMR (100 MHz, \* indicates signals of the isomer, CDCl<sub>3</sub>): δ 19.5 (br, B–CH), 20.6\* (br, B–CH), 24.6 (CH<sub>3</sub>), 24.7\* (CH<sub>3</sub>), 32.78 (CH<sub>2</sub>), 32.84\* (CH<sub>2</sub>), 35.0 (CH<sub>2</sub>), 35.3\* (CH<sub>2</sub>), 83.3 (C), 83.4\* (C), 111.6 (d, *J* = 5.8 Hz, CH), 112.1\* (d, *J* = 9.6 Hz, CH), 125.6 (CH), 125.7\* (CH), 128.2 (CH), 128.3\* (CH), 128.4 (CH), 142.1 (C), 142.4\* (C), 147.4 (d, *J* = 256.7 Hz, CH), 148.4\* (d, *J* = 254.8 Hz, CH). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ –130.6 – –130.2 (m, 1F). HRMS–EI (*m/z*): [M]<sup>+</sup> calcd for C<sub>17</sub>H<sub>24</sub>O<sub>2</sub>BF, 290.1857;

found, 290.1866.  $[\alpha]_D^{21.6} +0.07$  ( $c$  1.16,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding ester after oxidation of the boryl group and esterification of the alcohol. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 3/97, 0.5 mL/min, 40°C, retention time: 25.33 min [major enantiomer], 32.98 min [minor enantiomer], 24.21 min [isomer major enantiomer], and 29.70 min [isomer minor enantiomer].

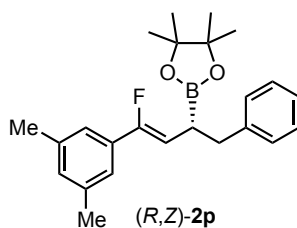
**(*S,Z*)-2-(1-Fluoro-1-phenylhept-1-en-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*S,Z*)-2o].**



Prepared according to conditions A. The reaction was conducted with for 1 h with 105.1 mg (0.50 mmol) of (*Z*)-1o. The product (*S,Z*)-2o was obtained in 88% yield (140.4 mg) with 97.5:2.5 e.r. The stereoselectivity of (*S,Z*)-2o was determined by GC analysis (*Z*:*E* = >98:2).

$^1\text{H}$  NMR (392 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.88 (t,  $J$  = 4.9 Hz, 3H), 1.26 (s, 12H), 1.23–1.39 (m, 4H), 1.41–1.66 (m, 2H), 2.28–2.38 (m, 1H), 5.40 (dd,  $J$  = 10.0, 37.4 Hz, 1H), 7.23–7.35 (m, 3H), 7.46–7.52 (m, 2H).  $^{13}\text{C}$  NMR (99 MHz,  $\text{CDCl}_3$ ):  $\delta$  14.0 ( $\text{CH}_3$ ), 20.8 (br, B–CH), 22.6 ( $\text{CH}_2$ ), 24.6 ( $\text{CH}_3$ ), 30.8 ( $\text{CH}_2$ ), 31.4 ( $\text{CH}_2$ ), 83.2 (C), 107.6 (d,  $J$  = 18.2 Hz, CH), 123.7 (d,  $J$  = 6.7 Hz, CH), 127.9 (CH), 128.2 (CH), 133.0 (d,  $J$  = 29.7 Hz, C), 156.0 (d,  $J$  = 245.2 Hz, C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  –121.4 (d,  $J$  = 45.9 Hz, 1F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{19}\text{H}_{28}\text{O}_2\text{BF}$ , 318.2170; found, 318.2167.  $[\alpha]_D^{20.8} -0.8$  ( $c$  1.21,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 5/95, 0.5 mL/min, 40°C, retention time: 29.51 min [(*Z*)-alcohol major enantiomer], 16.22 min [(*Z*)-alcohol minor enantiomer].

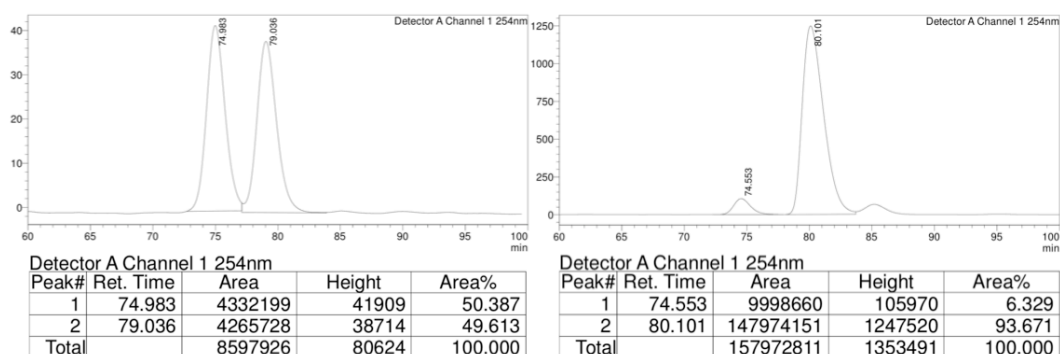
**(*R,Z*)-2-(4-(3,5-Dimethylphenyl)-4-fluoro-1-phenylbut-3-en-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane [(*R,Z*)-2p]**



Prepared according to conditions B from the corresponding crude difluoroallyl compound. In a  $\text{N}_2$ -filled glove box, an oven-dried 10 mL vial equipped with a magnetic stirring bar was

charged with 1-(1,1-difluoroallyl)-3,5-dimethylbenzene (18.2 mg, 100  $\mu\text{mol}$ , 1.0 equiv.) and allylbenzene (18 mg, 150  $\mu\text{mol}$ , 1.5 equiv.) then ruthenium complex **Ru-1** (3.6 mg, 5  $\mu\text{mol}$ , 5 mol%) in methylene chloride solution (0.2 mL, 0.5 M), was added, the vial tightly capped and transferred into an oil bath preheated to 50 °C. The mixture was allowed to stir for 12 h after which the solvent was evaporated under reduced pressure, the dark solid residue taken up in hexanes, passed through a short plug of silica gel (2 cm x 1 cm), concentrated under reduced pressure. The dark oily residue containing unpurified *gem*-allyldifluoride (70  $\mu\text{mol}$ ) was transferred back into the glove box. An oven-dried 8 mL vial, equipped with a magnetic stirring bar was charged with CuCl (0.4 mg, 4  $\mu\text{mol}$ , 5 mol%), (*S,S*)-phenyl-bpe (2.5 mg, 5  $\mu\text{mol}$ , 6 mol%), B<sub>2</sub>(pin)<sub>2</sub> (20 mg, 77  $\mu\text{mol}$ , 1.1 equiv.) and Li*O**t*-Bu (8.4 mg, 105  $\mu\text{mol}$ , 1.5 equiv.). The allylic fluoride residue was taken up in toluene (0.7 mL, 0.1 M) and introduced to the eight mL vial containing the other reagents. The vial was tightly capped and allowed to stir for 14 h at 22 °C. Subsequently the reaction was concentrated under reduced pressure and the residue subjected to silica gel chromatography (hexanes and Et<sub>2</sub>O) to afford the desired product as colorless oil in 49% yield (13 mg, 34  $\mu\text{mol}$ ).

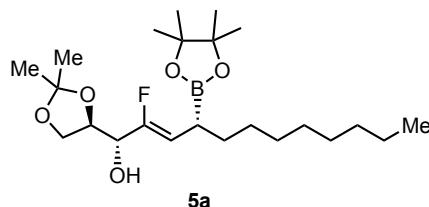
<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  1.17 (6H, s), 1.19 (6H, s), 2.30 (6H, s), 2.70 (1H, q, *J* = 8.4 Hz), 2.83 (1H, dd, *J* = 13.7, 8.1 Hz), 2.94 (1H, dd, *J* = 13.6, 8.0 Hz), 5.37 (1H, dd, *J* = 37.3, 9.6 Hz), 6.91 (1H, s), 7.08 (2H, s), 7.16 (1H, m), 7.26–7.22 (4H, m). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 126 MHz):  $\delta$  21.5 (CH<sub>3</sub>), 24.8 (CH<sub>3</sub>), 37.2 (CH<sub>2</sub>), 83.6 (C), 106.4 (d, *J* = 17.8 Hz, C), 121.8 (d, *J* = 6.7 Hz C), 126.0 (C), 128.3 (C), 129.0 (C), 130.0 (C), 132.9 (d, *J* = 28.6 Hz CH), 138.0 (d, *J* = 2.0 Hz, C), 141.5 (C), 156.7 (d, *J* = 245.7 Hz, CF). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 376 MHz):  $\delta$  -118.33 (d, *J* = 37.4 Hz). HRMS (DART-TOF) *m/z*: [M+NH<sub>4</sub>]<sup>+</sup> Calcd for C<sub>24</sub>H<sub>34</sub>B<sub>1</sub>F<sub>1</sub>O<sub>2</sub>N<sub>1</sub><sup>+</sup> = 398.2661, Found 398.2650. Specific rotation: [ $\alpha$ ]<sub>D</sub><sup>20</sup> +12.2 (*c* = 0.6, CHCl<sub>3</sub>) after oxidation. Enantiomeric purity of **2p** was determined by HPLC analysis after oxidation of **2p** to the corresponding allylic alcohol in comparison with authentic racemic material (ADH column, 99:1 hexanes:*i*PrOH, 0.5 mL/min, 254 nm). (*R*)-enantiomer *t*<sub>r</sub> = 80.1 min, (*S*)-enantiomer *t*<sub>r</sub> = 74.5 min.



| Peak # | Ret. Time | Area | Area % | Peak # | Ret. Time | Area | Area % |
|--------|-----------|------|--------|--------|-----------|------|--------|
|--------|-----------|------|--------|--------|-----------|------|--------|

|   |            |         |        |   |        |           |        |
|---|------------|---------|--------|---|--------|-----------|--------|
| 1 | 74.983 min | 4332199 | 50.387 | 1 | 74.553 | 9998660   | 6.329  |
| 2 | 79.036 min | 4265728 | 49.613 | 2 | 80.101 | 147974151 | 93.671 |

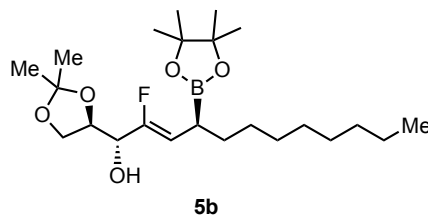
**(1*R*,4*R*,*Z*)-1-{(*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2-fluoro-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)}dodec-2-en-1-ol [5a]**



In a N<sub>2</sub>-filled glove box, an oven-dried vial (7 mL) containing a magnetic stir bar was charged with (*S,S*)-phenyl-bpe (11.1 mg, 2.20 μmol, 5.5 mol%), CuCl (2.00 mg, 2.00 μmol, 5.0 mol%), Li*Ot*-Bu (48.0 mg, 0.60 mmol, 1.5 equiv.) and toluene (2 mL). The resulting mixture was allowed to stir at 22 °C, then a solution of B<sub>2</sub>(pin)<sub>2</sub> (112 mg, 0.44 mmol, 1.5 equiv.) and alkene (*E*)-**4e** (112 mg, 0.40 mmol, 1.0 equiv.) in toluene (2 mL) was added. The reaction was allowed to stir at 22 °C for 14 h then was concentrated under reduced pressure. Purification by silica gel chromatography (petroleum ether:Et<sub>2</sub>O = 4:1 to 3:1) afforded the trisubstituted alkenyl fluoride **5a** as colourless oil in 77% yield (132 mg, 0.31 mmol).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 0.85 (3H, t, *J* = 6.8 Hz), 1.30–1.14 (m, 25H), 1.34 (3H, s), 1.41 (3H, s), 1.57–1.45 (1H, m), 2.14 (1H, q, *J* = 8.5 Hz), 2.53 (1H, bs), 4.05–3.92 (2H, m), 4.32–4.17 (2H, m), 4.87 (1H, dd, *J* = 38.1, 10.1 Hz). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz): δ 14.2 (CH<sub>3</sub>), 20.2 (CH<sub>2</sub>), 24.8 (CH<sub>3</sub>), 24.8 (CH<sub>3</sub>), 25.2 (CH<sub>2</sub>), 26.6 (CH<sub>2</sub>), 29.1 (CH<sub>2</sub>), 29.4 (CH<sub>2</sub>), 29.6 (CH<sub>2</sub>), 30.9 (CH<sub>2</sub>), 32.0 (CH<sub>2</sub>), 65.0 (d, *J*<sub>C,F</sub> = 2.2 Hz, CH), 70.0 (C), 70.3 (C), 76.2 (CH), 83.4 (CH), 109.6 (CH), 109.9 (d, *J*<sub>C,F</sub> = 13.1 Hz, CH), 155.6 (d, *J*<sub>C,F</sub> = 252.3 Hz, CF). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 377 MHz): δ –126.0 (dd, *J* = 38.1, 15.9 Hz). HRMS (MALDI): Calcd for C<sub>23</sub>H<sub>42</sub>BFNaO<sub>5</sub> [M+Na]<sup>+</sup>: 451.3002, Found: 451.2949. Specific rotation: [α]<sub>D</sub><sup>20</sup> +75.0 (*c* 1.0, CHCl<sub>3</sub>).

**(1*R*,4*S*,*Z*)-1-{(*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2-fluoro-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)}dodec-2-en-1-ol [5b]**



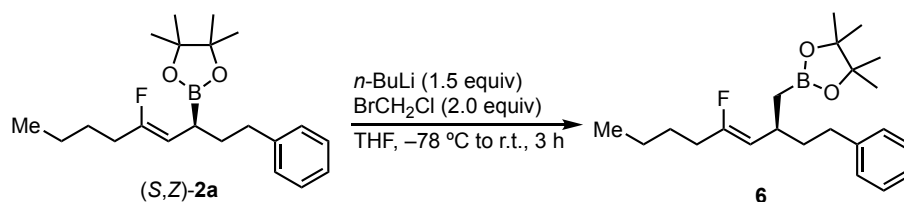
In a N<sub>2</sub>-filled glove box, an oven-dried vial (7 mL) containing a magnetic stir bar was charged with (*R,R*)-phenyl-bpe (11.1 mg, 2.20 μmol, 5.5 mol%), CuCl (2.00 mg, 2.00 μmol,

5.0 mol%), LiOt-Bu (48.0 mg, 0.60 mmol, 1.5 equiv.) and toluene (2 mL). The resulting mixture was allowed to stir at 22 °C, then a solution of B<sub>2</sub>(pin)<sub>2</sub> (112 mg, 0.44 mmol, 1.5 equiv.) and alkene (*E*)-**4e** (112 mg, 0.40 mmol, 1.0 equiv.) in toluene (2 mL) was added. The reaction was allowed to stir at 22 °C for 14 h then was concentrated under reduced pressure. Purification by silica gel chromatography (petroleum ether:Et<sub>2</sub>O = 4:1 to 3:1) afforded the trisubstituted alkenyl fluoride **5b** as colourless oil in 92% yield (158 mg, 0.37 mmol).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 0.85 (3H, t, *J* = 6.8 Hz), 1.29–1.18 (m, 25H), 1.33 (3H, s), 1.41 (3H, s), 1.59–1.44 (1H, m), 2.12 (1H, q, *J* = 8.8, 8.2 Hz), 2.50 (1H, s), 3.97 (2H, d, *J* = 5.9 Hz), 4.31–4.15 (2H, m), 4.88 (1H, dd, *J* = 38.4, 10.1 Hz). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz): δ 14.2 (CH<sub>3</sub>), 20.2 (CH<sub>3</sub>), 22.8 (CH<sub>3</sub>), 24.7 (CH<sub>3</sub>), 25.3 (CH<sub>2</sub>), 26.6 (CH<sub>2</sub>), 29.2 (CH<sub>2</sub>), 29.4 (CH<sub>2</sub>), 29.6 (CH<sub>2</sub>), 30.9 (CH<sub>2</sub>), 32.0 (CH<sub>2</sub>), 65.0 (d, *J*<sub>C,F</sub> = 2.2 Hz, CH), 70.0 (C), 70.3 (C), 76.3 (C), 83.4 (C), 109.5 (CH), 110.0 (d, *J*<sub>C,F</sub> = 13.0 Hz, CH), 155.7 (d, *J*<sub>C,F</sub> = 252.8 Hz, CF). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 377 MHz): δ –125.4 ppm (dd, *J* = 38.4, 16.5 Hz). HRMS (MALDI): Calcd for C<sub>23</sub>H<sub>42</sub>BFNaO<sub>5</sub> [M+Na]<sup>+</sup>: 451.3002, Found: 451.2978. Specific rotation: [α]<sub>D</sub><sup>20</sup> +74.1 (*c* 1.0, CHCl<sub>3</sub>).

## 5. Derivatization of $\gamma$ -Monofluoroallylboronates

### Experimental Procedure of Homologation of (*S,Z*)-2a.

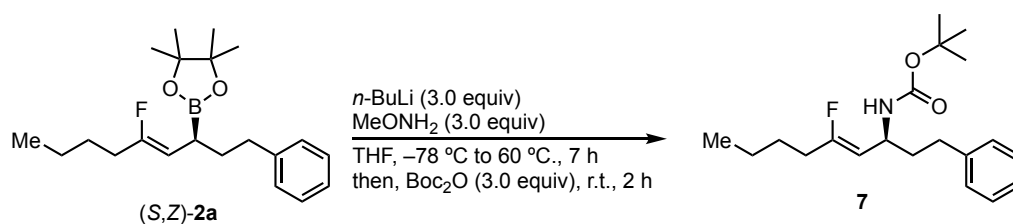


The homologation was performed according to the literature procedure.<sup>9</sup> In an oven-dried reaction vial, (*S,Z*)-**2a** (34.6 mg, 0.10 mmol) and bromochloromethane (13.4  $\mu\text{L}$ , 0.20 mmol) were dissolved in dry THF (600  $\mu\text{L}$ ) in nitrogen atmosphere. After the mixture was cooled to  $-78\text{ }^\circ\text{C}$ , a solution of *n*-BuLi in hexane (1.6 M, 94  $\mu\text{L}$ , 0.15 mmol) was added dropwise. The mixture was allowed to stir at  $22\text{ }^\circ\text{C}$  for 3 h. The mixture was then quenched by addition of saturated aqueous  $\text{NH}_4\text{Cl}$  solution and extracted three times with  $\text{Et}_2\text{O}$ . The combined organic layer was washed with brine, dried over  $\text{MgSO}_4$  followed by filtration. The crude material was purified by silica gel chromatography ( $\text{Et}_2\text{O}/\text{hexane}$ , 0:100–10:90) to give the corresponding **6** (32.5 mg, 0.090 mmol, 90%) as colorless oil with 97:3 e.r. The stereoselectivity of **6** was determined by GC analysis (*Z*:*E* = 95:5).

$^1\text{H NMR}$  (392 MHz,  $\text{CDCl}_3$ ):  $\delta$  0.75–0.96 (m, 5H), 1.22 (s, 12H), 1.29–1.40 (m, 2H), 1.41–1.53 (m, 3H), 1.64–1.76 (m, 1H), 2.08–2.20 (m, 2H), 2.46–2.66 (m, 2H), 2.72–2.84 (m, 1H), 4.36 (dd,  $J = 10.1, 38.2$  Hz, 1H), 7.11–7.18 (m, 3H), 7.21–7.28 (m, 2H).  $^{13}\text{C NMR}$  (99 MHz,  $\text{CDCl}_3$ ):  $\delta$  13.8 ( $\text{CH}_3$ ), 18.7 (br, B– $\text{CH}_2$ ), 22.0 ( $\text{CH}_2$ ), 24.7 ( $\text{CH}_3$ ), 24.8 ( $\text{CH}_3$ ), 28.5 ( $\text{CH}_2$ ), 30.4 (d,  $J = 3.8$  Hz, CH), 31.7 (d,  $J = 28.3$  Hz,  $\text{CH}_2$ ), 34.0 ( $\text{CH}_2$ ), 40.1 ( $\text{CH}_2$ ), 82.9 (C), 110.6 (d,  $J = 15.6$  Hz, CH), 125.5 (CH), 128.2 (CH), 128.4 (CH), 142.9 (C), 158.9 (d,  $J = 254.5$  Hz, C).  $^{19}\text{F NMR}$  (373 MHz,  $\text{CDCl}_3$ ):  $\delta$   $-109.2$  ppm (dt,  $J = 20.8$  Hz, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{34}\text{BFO}_2\text{Na}$ , 383.2532; found, 383.2530.  $[\alpha]_{\text{D}}^{18.9} +0.66$  ( $c$  1.00,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IC-3, 2-PrOH/Hexane = 2/98, 0.5 mL/min,  $40\text{ }^\circ\text{C}$ , retention time: 42.91 min [*Z*-alcohol major enantiomer], 30.85 min [*Z*-alcohol minor enantiomer], 26.29 min [*E*-alcohol major enantiomer], and 33.97 min [*E*-alcohol minor enantiomer].

### Experimental Procedure of Amination of (*S,Z*)-2a.



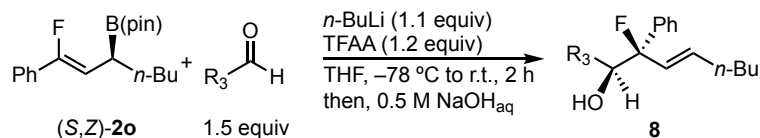


The amination was performed according to the literature procedure.<sup>10</sup> In an oven-dried reaction vial, MeONH<sub>2</sub> (0.5 M in THF, 600 μL, 0.30 mmol) was dissolved in THF (200 μL). After the mixture was cooled to  $-78\text{ }^\circ\text{C}$ , a solution of *n*-BuLi in hexane (1.6 M, 188 μL, 0.30 mmol) was added dropwise. Then the boronate (34.6 mg, 0.10 mmol) in THF (240 μL) was added dropwise to the solution and stirred at  $60\text{ }^\circ\text{C}$ . After 7 h, (Boc)<sub>2</sub>O was added to the mixture and stirred for 2 h at room temperature. The mixture was then quenched by addition of H<sub>2</sub>O and extracted three times with Et<sub>2</sub>O. The combined organic layer was dried over MgSO<sub>4</sub> followed by filtration. The crude material was purified by silica gel chromatography (EtOAc/hexane, 0:100–10:90) to give the corresponding **7** (20.9 mg, 0.060 mmol, 62%) as colorless oil with 97.5:2.5 e.r. The stereoselectivity of **7** was determined by GC analysis (*Z*:*E* = >98:2).  $\pm$ H and <sup>13</sup>C NMR spectra contain conformational isomers, which is caused by the restricted C–N bond rotation around the carbamate group.

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 0.91 (t, *J* = 7.1 Hz, 3H), 1.29–1.41 (m, 2H), 1.44 (s, 9H), 1.45–1.52 (m, 2H), 1.71–1.83 (m, 1H), 1.86–1.97 (m, 1H), 2.15 (dt, *J* = 7.3, 12.5 Hz, 2H), 2.63 (ddd, *J* = 2.8, 6.7, 9.5 Hz, 2H), 4.36–4.61 (m, 3H), 7.13–7.19 (m, 3H), 7.23–7.28 (m, 2H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 13.8 (CH<sub>3</sub>), 21.9 (CH<sub>2</sub>), 28.1 (CH<sub>2</sub>), 28.4 (CH<sub>3</sub>), 29.7 (C), 31.5 (d, *J* = 3.8 Hz, CH), 31.7 (d, *J* = 26.9 Hz, CH<sub>2</sub>), 32.3 (CH<sub>2</sub>), 37.7 (CH<sub>2</sub>), 46.3 (CH), 83.7 (C), 106.2 (CH), 125.8 (CH), 128.3 (CH), 141.7 (C), 155.1 (C). <sup>19</sup>F NMR (373 MHz, CDCl<sub>3</sub>): δ  $-104.2$  (s, 1F). HRMS-ESI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>30</sub>NFO<sub>2</sub>Na, 358.2153; found, 358.2153. [α]<sub>D</sub><sup>18.1</sup> +0.14 (*c* 1.58, CHCl<sub>3</sub>). Enantiomeric purity was determined by HPLC analysis of the corresponding alcohol after oxidation of the boryl group. Daicel CHIRALPAK® IC-3, 2-PrOH/Hexane = 4/96, 0.5 mL/min, 40°C, retention time: 24.21 min [major enantiomer], 13.81 min [minor enantiomer].

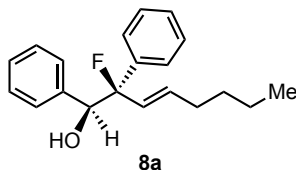
## 6. Alkylation of Aldehyde with $\gamma$ -Monofluoroallylboronates

### Experimental procedures of allylation reaction between (*S,Z*)-**2o** and aldehyde.<sup>11</sup>



In an oven-dried reaction vial, a solution of  $\gamma$ -monofluoroallylboronate (*S,Z*)-**2o** (31.8 mg, 0.10 mmol) in THF (1.0 mL) was treated with *n*-BuLi in hexane (1.6 M, 70.6  $\mu$ L, 0.113 mmol) at  $-78$   $^\circ\text{C}$  and the solution was allowed to stir for 15 min. Trifluoroacetic anhydride (17  $\mu$ L, 0.12 mmol) was added dropwise to the mixture and the reaction was allowed to stir for another 30 min at  $-78$   $^\circ\text{C}$ . Aldehyde (0.15 mmol) was then added at  $-78$   $^\circ\text{C}$  and the mixture was allowed to stir for 2 h at  $-78$   $^\circ\text{C}$ , then allowed to slowly warm up to room temperature. After 2 h, the reaction was quenched by addition of 0.5 M aqueous NaOH solution and extracted three times with Et<sub>2</sub>O. The combined organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> followed by filtration. The crude material was purified by silica gel chromatography (Et<sub>2</sub>O/hexane, 0:100–5:95) or pentane wash of the crude solid material to give the corresponding monofluoro compounds. The products were unstable in CHCl<sub>3</sub>.

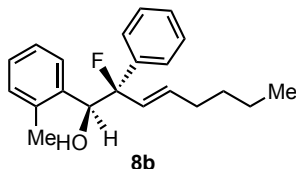
**(1*S*,2*R*,*E*)-2-Fluoro-1,2-diphenyloct-3-en-1-ol (8a).**



The product **8a** was obtained by pentane wash in 54% yield (16.2 mg) with 99:1 e.r. The stereoselectivity of **8a** was determined by  $^1\text{H}$  NMR analysis ( $E/Z = >98:2$ , d.r. =  $>98:2$ ).

$^1\text{H}$  NMR (396 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  0.87 (t,  $J = 7.1$  Hz, 3H), 1.17–1.39 (m, 4H), 1.98–2.12 (m, 2H), 4.72 (dd,  $J = 2.2, 5.0$  Hz, 1H), 5.02 (dd,  $J = 5.0, 14.5$  Hz, 1H), 5.63 (dt,  $J = 7.4, 15.0$  Hz, 1H), 6.12–6.21 (m, 1H), 7.12–7.43 (m, 10H).  $^{13}\text{C}$  NMR (100 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  14.3 ( $\text{CH}_3$ ), 22.9 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 32.8 ( $\text{CH}_2$ ), 79.5 (d,  $J = 27.8$  Hz, CH), 99.2 (d,  $J = 183.0$  Hz, C), 127.1 (d,  $J = 8.6$  Hz, CH), 127.9 (CH), 128.1 (CH), 128.2 (CH), 128.5 (CH), 129.3 (CH), 129.5 (d,  $J = 19.1$  Hz, CH), 133.1 (d,  $J = 10.5$  Hz, CH), 141.2 (C), 142.6 (d,  $J = 22.0$  Hz, C).  $^{19}\text{F}$  NMR (373 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  -160.1 (s, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{23}\text{OFNa}$ , 321.1625; found, 321.1627.  $[\alpha]_{\text{D}}^{21.9} -0.17$  ( $c$  1.47, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 1/99, 0.5 mL/min, 40°C, retention time: 44.26 min [major enantiomer], 39.73 min [minor enantiomer].

**(1*S*,2*R*,*E*)-2-Fluoro-2-phenyl-1-(*o*-tolyl)oct-3-en-1-ol (8b).**

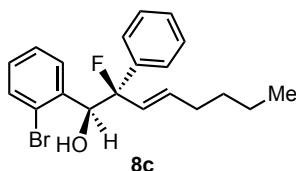


The product **8b** was obtained by flash column chromatography in 40% yield (12.4 mg) with 96.5:3.5 e.r. The stereoselectivity of **8b** was determined by  $^1\text{H}$  NMR analysis ( $E:Z = >98:2$ , d.r. =  $>98:2$ ).

$^1\text{H}$  NMR (396 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  0.89 (t,  $J = 7.2$  Hz, 3H), 1.20–1.38 (m, 4H), 2.05–2.12 (m, 2H), 2.17 (s, 3H), 4.57 (dd,  $J = 0.8, 4.8$  Hz, 1H), 5.28 (dd,  $J = 5.2, 11.2$  Hz, 1H), 5.61 (dt,  $J = 3.2, 11.2$  Hz, 1H), 6.26 (ddt,  $J = 1.5, 15.6, 21.1$  Hz, 1H), 6.99–7.05 (m, 1H), 7.08–7.1 (m, 2H), 7.24–7.34 (m, 3H), 7.42–7.46 (m, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  14.2 ( $\text{CH}_3$ ), 20.0 (d,  $J = 3.8$  Hz), 22.9 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 32.9 ( $\text{CH}_2$ ), 74.9 (d,  $J = 29.2$  Hz, CH), 99.6 (d,  $J = 178.2$  Hz, C), 125.6 (CH), 127.1 (d,  $J = 8.4$  Hz, CH), 128.0 (CH), 128.3 (CH), 128.5 (CH), 129.0 (d,  $J = 22.7$  Hz, CH), 129.8 (CH), 130.2 (CH), 132.7 (d,  $J = 11.4$  Hz, CH), 137.1 (C), 139.6 (C), 143.3 (d,  $J = 22.0$  Hz, C).  $^{19}\text{F}$  NMR (373 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  -159.9 (s, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{21}\text{H}_{25}\text{OFNa}$ , 335.1782; found, 335.1791.  $[\alpha]_{\text{D}}^{21.4} +2.1$

(*c* 1.24, THF). The ee value was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 1/99, 0.5 mL/min, 40°C, retention time: 28.38 min [major enantiomer], 33.68 min [minor enantiomer].

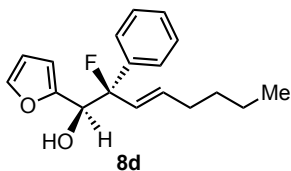
**(1*S*,2*R*,*E*)-1-(2-Bromophenyl)-2-fluoro-2-phenyloct-3-en-1-ol (8c).**



The product **8c** was obtained by flash column chromatography in 58% yield (21.7 mg) with 97.5:2.5 e.r. The stereoselectivity of **8c** was determined by <sup>1</sup>H NMR analysis (*E:Z* = >98:2, d.r. = >98:2).

<sup>1</sup>H NMR (399 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ 0.89 (t, *J* = 7.2 Hz, 3H), 1.24–1.42 (m, 4H), 2.09–2.17 (m, 2H), 5.01 (d, *J* = 5.2 Hz, 1H), 5.52 (dd, *J* = 5.2, 9.2 Hz, 1H), 5.59 (dt, *J* = 7.0, 15.7 Hz, 1H), 6.30 (ddt, *J* = 1.5, 15.7, 20.9 Hz, 1H), 7.17 (td, *J* = 1.6, 7.6 Hz, 1H), 7.25–7.37 (m, 4H), 7.46–7.59 (m, 4H). <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ 14.3 (CH<sub>3</sub>), 23.0 (CH<sub>2</sub>), 32.0 (CH<sub>2</sub>), 32.9 (CH<sub>2</sub>), 77.3 (d, *J* = 28.8 Hz, CH), 99.1 (d, *J* = 183.0 Hz, C), 125.1 (C), 127.2 (d, *J* = 8.6 Hz, CH), 127.5 (CH), 128.3 (d, *J* = 18.2 Hz, CH), 128.5 (CH), 128.6 (CH), 130.1 (CH), 131.9 (CH), 132.8 (CH), 133.2 (d, *J* = 11.5 Hz, CH), 140.7 (C), 143.31 (d, *J* = 23.0 Hz, C). <sup>19</sup>F NMR (373 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ -158.9 (s, 1F). HRMS–ESI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>22</sub>OBrFNa, 399.0730; found, 399.0734. [α]<sub>D</sub><sup>21.8</sup> +3.2 (*c* 1.58, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 1/99, 0.5 mL/min, 40°C, retention time: 26.81 min [major enantiomer], 31.59 min [minor enantiomer].

**(1*S*,2*R*,*E*)-2-Fluoro-1-(furan-2-yl)-2-phenyloct-3-en-1-ol (8d).**

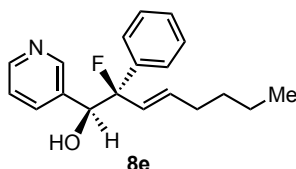


The product **8d** was obtained by flash column chromatography in 60% yield (17.4 mg) with 97.5:2.5 e.r. The stereoselectivity of **8d** was determined by <sup>1</sup>H NMR analysis (*E:Z* = >98:2, d.r. = >98:2).

<sup>1</sup>H NMR (396 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ 0.88 (t, *J* = 7.3 Hz, 3H), 1.24–1.41 (m, 4H), 2.08–2.15 (m, 2H), 4.80 (d, *J* = 6.3 Hz, 1H), 5.03 (dd, *J* = 6.3, 15.4 Hz, 1H), 5.71 (dtd, *J* = 1.6, 6.9, 15.6 Hz, 1H), 6.13–6.22 (m, 2H), 6.29 (dd, *J* = 1.8, 3.2 Hz, 1H), 7.03–7.35 (m, 3H), 7.36–7.43 (m,

3H).  $^{13}\text{C}$  NMR (100 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  14.2 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$ ), 31.9 ( $\text{CH}_2$ ), 32.8 ( $\text{CH}_2$ ), 73.9 (d,  $J = 28.7$  Hz, CH), 98.9 (d,  $J = 182.0$  Hz, C), 108.9 (CH), 110.9 (CH), 126.7 (d,  $J = 9.6$  Hz, CH), 128.3 (CH), 128.5 (CH), 129.7 (d,  $J = 18.2$  Hz, CH), 133.5 (d,  $J = 10.5$  Hz, CH), 142.1 (d,  $J = 23.0$  Hz, C), 142.3 (CH), 154.8 (C).  $^{19}\text{F}$  NMR (373 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  -160.0 (s, 1F). HRMS–ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{18}\text{H}_{21}\text{O}_2\text{FNa}$ , 311.1418; found, 311.1421.  $[\alpha]_{\text{D}}^{21.8} +1.3$  ( $c$  1.76, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 2/98, 0.5 mL/min, 40°C, retention time: 44.69 min [major enantiomer], 38.80 min [minor enantiomer].

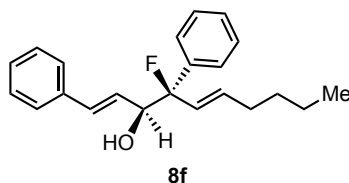
**(1*S*,2*R*,*E*)-2-Fluoro-2-phenyl-1-(pyridin-3-yl)oct-3-en-1-ol (8e).**



The product **8e** was obtained by flash column chromatography in 56% yield (16.6 mg) with 97:3 e.r. The stereoselectivity of **8e** was determined by  $^1\text{H}$  NMR analysis ( $E:Z = >98:2$ , d.r. = 93:7).

$^1\text{H}$  NMR (401 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  0.87 (t,  $J = 7.2$  Hz, 3H), 1.20–1.43 (m, 4H), 2.07–2.13 (m, 2H), 4.96–5.07 (m, 1H), 5.08–5.14 (m, 1H), 5.64 (dt,  $J = 7.4, 15.1$  Hz, 1H), 6.14–6.26 (m, 1H), 7.18–7.24 (m, 1H), 7.25–7.36 (m, 3H), 7.37–7.42 (m, 2H), 7.63 (d,  $J = 8.4$  Hz, 1H), 8.37–8.42 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  14.2 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$ ), 31.9 ( $\text{CH}_2$ ), 32.8 ( $\text{CH}_2$ ), 77.4 (d,  $J = 27.8$  Hz, CH), 99.1 (d,  $J = 183.0$  Hz, C), 123.2 (CH), 126.9 (d,  $J = 8.8$  Hz, CH), 128.5 (CH), 128.7 (CH), 129.0 (CH), 133.7 (d,  $J = 11.5$  Hz, CH), 136.4 (CH), 136.6 (C), 142.3 (d,  $J = 22.1$  Hz, C), 149.4 (CH), 150.5 (CH).  $^{19}\text{F}$  NMR (373 MHz,  $(\text{CD}_3)_2\text{CO}$ ):  $\delta$  -161.3 (s, 1F). HRMS–EI ( $m/z$ ):  $[\text{M}]^+$  calcd for  $\text{C}_{19}\text{H}_{23}\text{ONF}$ , 300.1758; found, 300.1767.  $[\alpha]_{\text{D}}^{21.3} +0.14$  ( $c$  1.66, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 1/99, 0.5 mL/min, 40°C, retention time: 43.49 min [major enantiomer], 36.49 min [minor enantiomer].

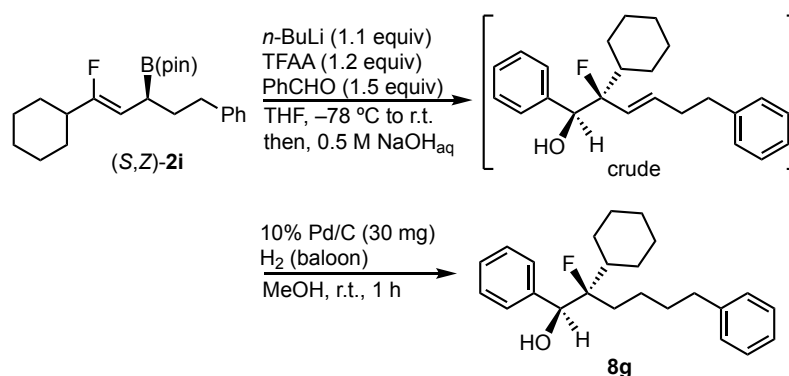
**(1*E*,3*S*,4*R*,5*E*)-4-Fluoro-1,4-diphenyldeca-1,5-dien-3-ol (8f).**



The product **8f** was obtained by pentane wash in 33% yield (10.7 mg) with >99:1 e.r. The stereoselectivity of **8f** was determined by  $^1\text{H}$  NMR analysis ( $E:Z = >98:2$ , d.r. = >98:2).

<sup>1</sup>H NMR (396 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ 0.86 (t, *J* = 7.1 Hz, 3H), 1.21–1.40 (m, 4H), 2.06–2.16 (m, 2H), 4.50–4.49 (m, 1H), 4.61 (dt, *J* = 6.3, 13.0 Hz, 1H), 5.79 (dt, *J* = 7.4, 15.0 Hz, 1H), 6.13–6.22 (m, 1H), 6.28 (dd, *J* = 6.0, 16.0 Hz, 1H), 6.62 (d, *J* = 15.8 Hz, 1H), 7.20–7.38 (m, 8H), 7.48–7.50 (m, 2H). <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ 14.2 (CH<sub>3</sub>), 22.9 (CH<sub>2</sub>), 32.1 (CH<sub>2</sub>), 32.9 (CH<sub>2</sub>), 78.3 (d, *J* = 28.8 Hz, CH), 99.3 (d, *J* = 180.1 Hz, C), 126.9 (d, *J* = 9.6 Hz, CH), 127.3 (CH), 128.3 (d, *J* = 4.8 Hz, CH), 128.4 (CH), 128.7 (CH), 128.9 (d, *J* = 3.8 Hz, CH), 129.5 (CH), 129.9 (d, *J* = 18.2 Hz, CH), 132.7 (CH), 133.5 (d, *J* = 10.5 Hz, CH), 138.1 (C), 142.4 (d, *J* = 22.0 Hz, C). <sup>19</sup>F NMR (373 MHz, (CD<sub>3</sub>)<sub>2</sub>CO): δ –158.8 (s, 1F). HRMS–EI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>22</sub>H<sub>25</sub>OFNa, 347.1782; found, 347.1787. [α]<sub>D</sub><sup>20.8</sup> –11.2 (*c* 1.0, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 2/98, 0.5 mL/min, 40°C, retention time: 35.08 min [major enantiomer], 45.09 min [minor enantiomer].

**Allylation reaction between (*S,Z*)-2i and benzaldehyde followed by H<sub>2</sub> hydrogenation with Pd/C.**



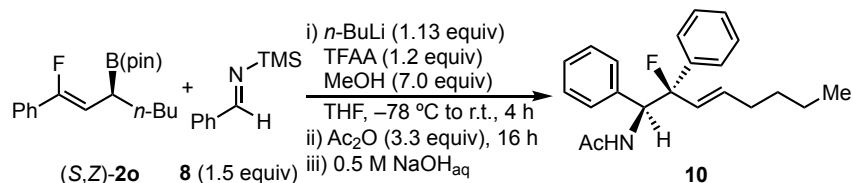
The reaction was conducted with 37.2 mg (0.10 mmol) of (*S,Z*)-**2i**. The title compound was submitted to the subsequent hydrogenation without further purification. In a reaction vial, 10% Pd/C (30 mg) was dissolved in MeOH (1.0 mL). The crude material of the allylation reaction in MeOH (0.5 mL) was then added to this solution. After stirred for 1 h at room temperature under H<sub>2</sub> atmosphere (H<sub>2</sub> balloon), the mixture was filtered through a Celite pad and concentrated under reduced pressure. The crude material was purified by silica gel chromatography (Et<sub>2</sub>O/hexane, 0:100–6:94). The product **8g** was obtained in 53% yield (18.9 mg) with 97:3 e.r. The stereoselectivity of **8g** was determined by <sup>1</sup>H NMR analysis (d.r. = 92:8).

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>): δ 1.02–1.88 (m, 17H), 2.02 (d, *J* = 3.9 Hz, 1H), 2.60 (t, *J* = 7.4 Hz, 2H), 4.93 (dd, *J* = 4.3, 13.3 Hz, 1H), 7.14–7.41 (m, 10H). <sup>13</sup>C NMR (99 MHz, CDCl<sub>3</sub>): δ 22.9 (d, *J* = 8.0 Hz, CH<sub>2</sub>), 26.4 (CH<sub>2</sub>), 26.5 (CH<sub>2</sub>), 26.7 (CH<sub>2</sub>), 26.5 (d, *J* = 8.5 Hz, CH<sub>2</sub>), 27.4 (d, *J* = 5.2 Hz, CH<sub>2</sub>), 31.3 (d, *J* = 23.2 Hz, CH<sub>2</sub>), 32.1 (CH<sub>2</sub>), 35.7 (CH<sub>2</sub>), 42.1 (d, *J*

= 21.3 Hz, CH), 100.6 (d,  $J = 175.5$  Hz, C), 125.6 (CH), 127.4 (d,  $J = 1.9$  Hz, CH), 127.7 (CH), 128.0 (CH), 128.2 (CH), 128.3 (CH), 140.4 (C), 142.5 (C).  $^{19}\text{F}$  NMR (373 MHz,  $\text{CDCl}_3$ ):  $\delta$  -164.5 (d,  $J = 33.9$  Hz, 1F). HRMS-ESI ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{24}\text{H}_{31}\text{OFNa}$ , 377.2251; found, 377.2255.  $[\alpha]_{\text{D}}^{19.7} +1.64$  ( $c$  1.55,  $\text{CHCl}_3$ ). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IBN-3, 2-PrOH/Hexane = 1.5/98.5, 0.5 mL/min, 40°C, retention time: 46.95 min [*syn*-**8g** major enantiomer], 29.58 min [*syn*-**8g** minor enantiomer], 29.58 min [*anti*-**8g** major enantiomer], and 27.17 min [*anti*-**8g** minor enantiomer].

## 7. Alkylation of Aldimine with $\gamma$ -Monofluoroallylboronates

### Experimental procedures of alkylation reaction between (*S,Z*)-**2o** and aldimine.<sup>10</sup>



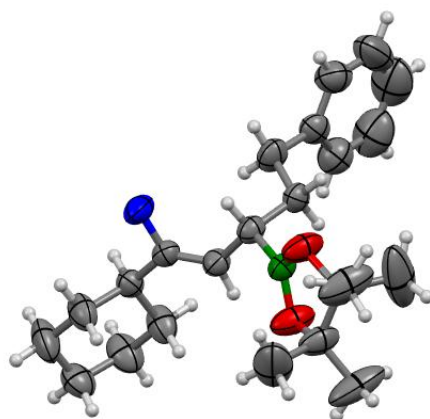
In an oven-dried reaction vial, a solution of  $\gamma$ -monofluoroallylboronate (*S,Z*)-**2o** (31.8 mg, 0.10 mmol) in THF (1.0 mL) was treated with *n*-BuLi in hexane (1.6 M, 70.6  $\mu$ L, 0.113 mmol) at  $-78$  °C and the solution was allowed to stir for 15 min. Trifluoroacetic anhydride (16.9  $\mu$ L, 0.12 mmol) was added dropwise to the mixture and the reaction was allowed to stir for a further 30 min at  $-78$  °C. *N*-Trimethylsilyl benzaldimine (18.4  $\mu$ L, 0.15 mmol) and MeOH (30  $\mu$ L, 0.74 mmol) was then added at  $-78$  °C and the mixture was allowed to stir for 1 h at  $-78$  °C, then allowed to slowly warm up to room temperature. After 3 h, Ac<sub>2</sub>O (34  $\mu$ L, 0.33 mmol) was added to the mixture and stirred at room temperature. After 16 h, the reaction was quenched by addition of 0.5 M aqueous NaOH solution and extracted three times with Et<sub>2</sub>O. The combined organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> followed by filtration. The solid crude material was purified by hexane wash. The product **10** was obtained in 59% yield (20.0 mg) with 98:2 e.r. The stereoselectivity of **10** was determined by <sup>1</sup>H NMR analysis (*E*:*Z* = >98:2, d.r. = >98:2).

<sup>1</sup>H NMR (396 MHz, (CD<sub>3</sub>)<sub>2</sub>CO):  $\delta$  0.89 (t, *J* = 6.9 Hz, 3H), 1.25–1.43 (m, 4H), 1.93 (s, 3H), 2.07–2.13 (m, 2H), 5.53 (dd, *J* = 9.5, 30.1 Hz, 1H), 5.77–5.84 (m, 1H), 5.95–6.03 (m, 1H), 7.06–7.24 (m, 10H), 7.83–7.94 (m, 1H). <sup>13</sup>C NMR (100 MHz, (CD<sub>3</sub>)<sub>2</sub>CO):  $\delta$  14.2 (CH<sub>3</sub>), 22.8 (CH<sub>2</sub>), 22.9 (CH<sub>3</sub>), 32.1 (CH<sub>2</sub>), 32.7 (CH<sub>2</sub>), 60.2 (d, *J* = 20.2 Hz, CH), 100.8 (d, *J* = 185.9 Hz, C), 125.8 (d, *J* = 9.5 Hz, CH), 127.8 (CH), 128.0 (CH), 128.3 (CH), 128.7 (CH), 129.9 (CH), 131.1 (d, *J* = 19.1 Hz, CH), 132.3 (d, *J* = 10.5 Hz, CH), 139.1 (C), 142.1 (d, *J* = 22.0 Hz, C), 169.3 (C). <sup>19</sup>F NMR (373 MHz, (CD<sub>3</sub>)<sub>2</sub>CO):  $\delta$  -169.6 (t, *J* = 22.8 Hz, 1F). HRMS–EI (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>22</sub>H<sub>26</sub>ONFNa, 362.1891; found, 362.1900. [ $\alpha$ ]<sub>D</sub><sup>21.0</sup> +0.12 (*c* 1.68, THF). Enantiomeric purity was determined by HPLC analysis. Daicel CHIRALPAK® IF-3, 2-PrOH/Hexane = 15/85, 0.5 mL/min, 40°C, retention time: 13.15 min [major enantiomer], 16.64 min [minor enantiomer].

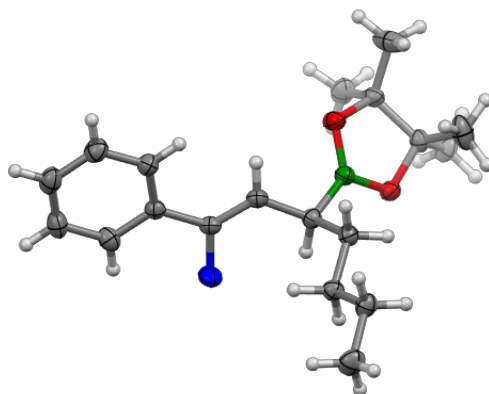


## 8. Single Crystal X-ray Structural Analysis

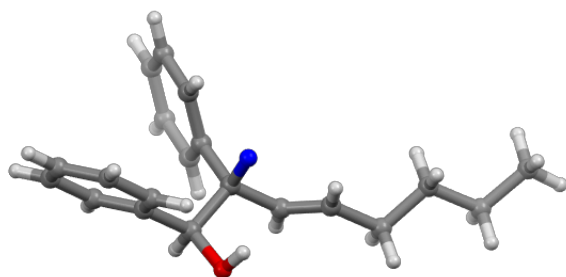
The absolute configuration of the products were determined based on X-ray crystallographic analysis of the products **2h**, **2o**, **8a** and **8g**. The absolute configurations of other products were deduced by these products. The details were summarized in Figure S1–S4 and Table S1–S4.



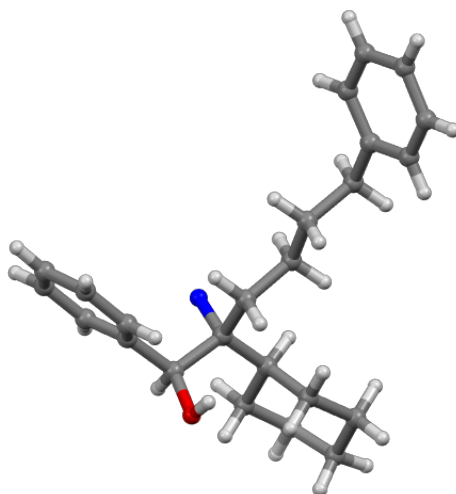
**Figure S1.** Molecular structure of (*S,Z*)-**2h**. Thermal ellipsoids set at 50% probability.



**Figure S2.** Molecular structure of (*S,Z*)-**2o**. Thermal ellipsoids set at 50% probability.



**Figure S3.** Molecular structure of (1*S*,2*R*,*E*)-**8a**.



**Figure S4.** Molecular structure of (1*S*,2*R*)-**8g**.

**Table S1.** Summary of X-ray crystallographic data for (*S,Z*)-**2h**.

|  |  |
|--|--|
| CCDC   | 1905700  |
| Empirical formula  | C <sub>23</sub> H <sub>34</sub> BFO <sub>2</sub>                             |
| Formula weight   | 372.31   |
| Temperature/K  | 243  |
| Crystal system   | monoclinic   |
| Space group  | <i>P</i> 2 <sub>1</sub>  |
| <i>a</i> / Å   | 6.51580(10)  |
| <i>b</i> / Å   | 20.1138(3)   |
| <i>c</i> / Å   | 8.74810(10)  |
| $\alpha$ /°  | 90   |
| $\beta$ /°   | 99.239(2)  |
| $\gamma$ /°  | 90   |
| Volume/Å <sup>3</sup>  | 1131.63(3)   |
| <i>Z</i>   | 2  |
| $\rho_{\text{calc}}$ g/cm <sup>3</sup>                       | 1.093  |
| $\mu$ /mm <sup>-1</sup>                                      | 0.572  |
| F(000)   | 404.0  |
| Crystal size/mm <sup>3</sup>                                 | 0.38×0.26×0.21   |
| Radiation  | CuK $\alpha$ ( $\lambda$ = 1.54184)  |
| 2 $\theta$ range for data collection/°                       | 8.792 to 147.57  |
| Index ranges   | -7 ≤ <i>h</i> ≤ 7, -24 ≤ <i>k</i> ≤ 24, -10 ≤ <i>l</i> ≤ 10                  |
| Reflections collected  | 11342  |
| Independent reflections                                      | 4405 [ <i>R</i> <sub>int</sub> = 0.0233, <i>R</i> <sub>sigma</sub> = 0.0251] |
| Data/restraints/parameters                                   | 4405/1/248   |
| Goodness-of-fit on F <sup>2</sup>                            | 1.072  |
| Final <i>R</i> indexes [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )] | <i>R</i> <sub>1</sub> = 0.0581, <i>wR</i> <sub>2</sub> = 0.1715              |
| Final <i>R</i> indexes [all data]                            | <i>R</i> <sub>1</sub> = 0.0611, <i>wR</i> <sub>2</sub> = 0.1735              |
| Largest diff. peak/hole / e Å <sup>-3</sup>                  | 0.33/-0.18   |
| Flack parameter  | 0.06(8)  |

**Table S2.** Summary of X-ray crystallographic data for (*S,Z*)-**2o**.

|  |  |
|--|--|
| CCDC   | 1905696  |
| Empirical formula  | C <sub>19</sub> H <sub>28</sub> BFO <sub>2</sub>                             |
| Formula weight   | 318.22   |
| Temperature/K  | 123  |
| Crystal system   | orthorhombic   |
| Space group  | <i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>                        |
| <i>a</i> / Å   | 9.29320(10)  |
| <i>b</i> / Å   | 11.89520(10)   |
| <i>c</i> / Å   | 16.6460(10)  |
| $\alpha$ /°  | 90   |
| $\beta$ /°   | 90   |
| $\gamma$ /°  | 90   |
| Volume/Å <sup>3</sup>  | 1840.12(3)   |
| <i>Z</i>   | 4  |
| $\rho_{\text{calc}}$ g/cm <sup>3</sup>                       | 1.149  |
| $\mu$ /mm <sup>-1</sup>                                      | 0.625  |
| F(000)   | 688.0  |
| Crystal size/mm <sup>3</sup>                                 | 0.274×0.147×0.1  |
| Radiation  | CuK $\alpha$ ( $\lambda$ = 1.54184)  |
| 2 $\theta$ range for data collection/°                       | 9.138 to 148.066   |
| Index ranges   | -11 ≤ <i>h</i> ≤ 11, -14 ≤ <i>k</i> ≤ 14, -20 ≤ <i>l</i> ≤ 20                |
| Reflections collected  | 53118  |
| Independent reflections                                      | 3701 [ <i>R</i> <sub>int</sub> = 0.1171, <i>R</i> <sub>sigma</sub> = 0.0316] |
| Data/restraints/parameters                                   | 3701/0/213   |
| Goodness-of-fit on F <sup>2</sup>                            | 1.099  |
| Final <i>R</i> indexes [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )] | <i>R</i> <sub>1</sub> = 0.0460, <i>wR</i> <sub>2</sub> = 0.1165              |
| Final <i>R</i> indexes [all data]                            | <i>R</i> <sub>1</sub> = 0.0486, <i>wR</i> <sub>2</sub> = 0.1200              |
| Largest diff. peak/hole / e Å <sup>-3</sup>                  | 0.14/-0.28   |
| Flack parameter  | -0.05(7)   |

**Table S3.** Summary of X-ray crystallographic data for **8a**.

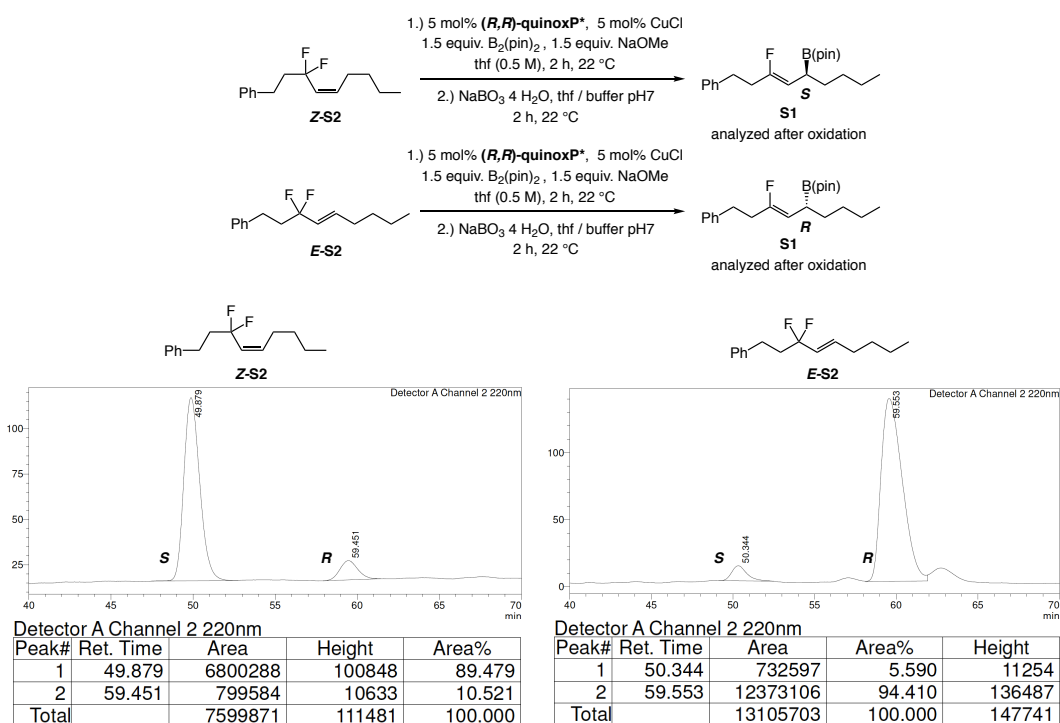
|  |   |
|--|---|
| CCDC   | 1905693   |
| Empirical formula  | C <sub>20</sub> H <sub>23</sub> FO  |
| Formula weight   | 298.38  |
| Temperature/K  | 123   |
| Crystal system   | monoclinic  |
| Space group  | <i>P</i> 2 <sub>1</sub>   |
| <i>a</i> / Å   | 16.0231(2)  |
| <i>b</i> / Å   | 5.46870(10)   |
| <i>c</i> / Å   | 18.5413(3)  |
| $\alpha$ /°  | 90  |
| $\beta$ /°   | 92.4760(10)   |
| $\gamma$ /°  | 90  |
| Volume/Å <sup>3</sup>  | 1623.17(4)  |
| <i>Z</i>   | 4   |
| $\rho_{\text{calc}}$ g/cm <sup>3</sup>                       | 1.221   |
| $\mu$ /mm <sup>-1</sup>                                      | 0.644   |
| F(000)   | 640.0   |
| Crystal size/mm <sup>3</sup>                                 | 0.342×0.04×0.04   |
| Radiation  | CuK $\alpha$ ( $\lambda$ = 1.54184)   |
| 2 $\theta$ range for data collection/°                       | 4.77 to 140.628   |
| Index ranges   | -17 ≤ <i>h</i> ≤ 19, -5 ≤ <i>k</i> ≤ 6, -22 ≤ <i>l</i> ≤ 22                   |
| Reflections collected  | 55714   |
| Independent reflections                                      | 5650 [ <i>R</i> <sub>int</sub> = 0.01826, <i>R</i> <sub>sigma</sub> = 0.0531] |
| Data/restraints/parameters                                   | 5650/1/353  |
| Goodness-of-fit on F <sup>2</sup>                            | 1.036   |
| Final <i>R</i> indexes [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )] | <i>R</i> <sub>1</sub> = 0.0598, <i>wR</i> <sub>2</sub> = 0.1584               |
| Final <i>R</i> indexes [all data]                            | <i>R</i> <sub>1</sub> = 0.0627, <i>wR</i> <sub>2</sub> = 0.1619               |
| Largest diff. peak/hole / e Å <sup>-3</sup>                  | 0.50/-0.41  |
| Flack parameter  | 0.03(15)  |

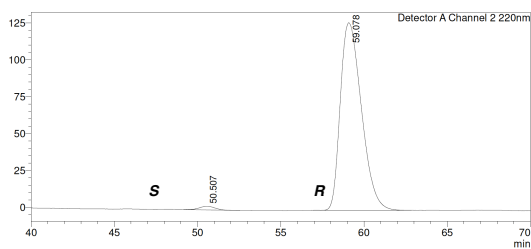
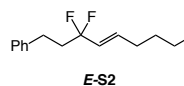
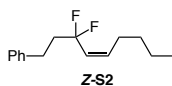
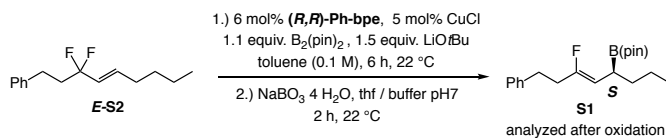
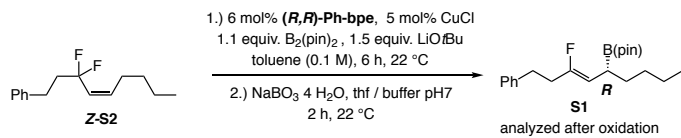
**Table S4.** Summary of X-ray crystallographic data for **8g**

|  |  |
|--|--|
| CCDC   | 1905684  |
| Empirical formula  | C <sub>24</sub> H <sub>31</sub> FO   |
| Formula weight   | 354.49   |
| Temperature/K  | 123  |
| Crystal system   | monoclinic   |
| Space group  | <i>P</i> 2 <sub>1</sub>  |
| <i>a</i> / Å   | 12.0730(10)  |
| <i>b</i> / Å   | 5.54330(10)  |
| <i>c</i> / Å   | 14.6277(2)   |
| $\alpha$ /°  | 90   |
| $\beta$ /°   | 96.7300(10)  |
| $\gamma$ /°  | 90   |
| Volume/Å <sup>3</sup>  | 972.26(2)  |
| <i>Z</i>   | 2  |
| $\rho_{\text{calc}}$ g/cm <sup>3</sup>                       | 1.211  |
| $\mu$ /mm <sup>-1</sup>                                      | 0.613  |
| F(000)   | 384.0  |
| Crystal size/mm <sup>3</sup>                                 | 0.295×0.11×0.06  |
| Radiation  | CuK $\alpha$ ( $\lambda$ = 1.54184)  |
| 2 $\theta$ range for data collection/°                       | 6.084 to 148.214   |
| Index ranges   | -14 ≤ <i>h</i> ≤ 15, -6 ≤ <i>k</i> ≤ 6, -18 ≤ <i>l</i> ≤ 18                  |
| Reflections collected  | 37395  |
| Independent reflections                                      | 3847 [ <i>R</i> <sub>int</sub> = 0.1160, <i>R</i> <sub>sigma</sub> = 0.0378] |
| Data/restraints/parameters                                   | 3847/1/236   |
| Goodness-of-fit on F <sup>2</sup>                            | 1.065  |
| Final <i>R</i> indexes [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )] | <i>R</i> <sub>1</sub> = 0.0473, <i>wR</i> <sub>2</sub> = 0.1284              |
| Final <i>R</i> indexes [all data]                            | <i>R</i> <sub>1</sub> = 0.0501, <i>wR</i> <sub>2</sub> = 0.1312              |
| Largest diff. peak/hole / e Å <sup>-3</sup>                  | 0.23/-0.22   |
| Flack parameter  | 0.03(11)   |

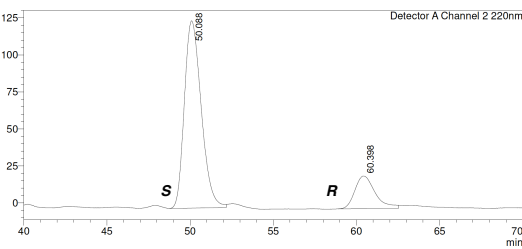
## 9. Stereoproof of the borylation products form (*E*)-4.

As the stereochemistry was proven for Cu–B(pin) addition, fluoride elimination of *Z*-gem-difluoro substituted olefins, comparison of stereochemical outcome of a *Z*-substrate and its *E*- counterpart with (*R,R*)-quinoxP\* and (*R,R*)-phenyl-bpe reveals, *Z*- and *E*- olefins lead to opposing stereochemical outcomes with the same ligand enantiomer, further (*R,R*)-phenyl-bpe forms the *R*-enantiomer of the product from *Z*-substrates and the *S*-enantiomer from *E*-substrates.





| Peak# | Ret. Time | Area     | Area%   | Height |
|-------|-----------|----------|---------|--------|
| 1     | 50.507    | 157600   | 1.385   | 2495   |
| 2     | 59.078    | 11218257 | 98.615  | 127224 |
| Total |           | 11375856 | 100.000 | 129719 |



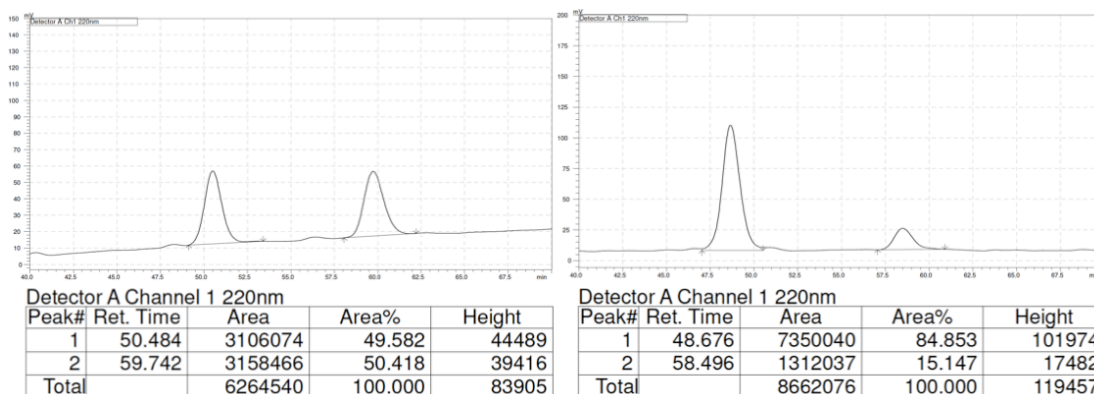
| Peak# | Ret. Time | Area     | Area%   | Height |
|-------|-----------|----------|---------|--------|
| 1     | 50.088    | 8925583  | 82.552  | 126483 |
| 2     | 60.398    | 1886500  | 17.448  | 22085  |
| Total |           | 10812083 | 100.000 | 148568 |



**(S)-(Z)-2-(3-Fluoro-1-phenylnon-3-en-5-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (S1).**

Prepared according to conditions B with (*R,R*)-phenyl-bpe and isolated as colorless oil in 59% yield (59:41 *Z/E*).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 600 MHz): δ 0.87 (3H, t, *J* = 7.0 Hz), 1.23 (16H, s), 1.48 (1H, ddd, *J* = 12.9, 6.5, 3.5 Hz), 2.08 (1H, td, *J* = 9.0, 6.7 Hz), 2.56–2.37 (2H, m), 2.80 (2H, t, *J* = 7.7 Hz), 4.45 (1H, dd, *J* = 38.4, 9.7 Hz), 7.19 (3H, m, *J* = 7.3 Hz), 7.30–7.24 (2H, m). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 150 MHz): δ 14.2 (CH<sub>3</sub>), 22.78 (CH<sub>3</sub>), 24.80 (CH<sub>3</sub>), 24.82 (CH<sub>2</sub>), 31.0 (d, *J* = 1.6 Hz, CH<sub>2</sub>), 31.4 (CH), 33.0 (CH<sub>2</sub>), 34.1 (CH<sub>2</sub>), 34.3 (CH<sub>2</sub>), 83.2 (C), 106.9 (d, *J* = 16.1 Hz, CH), 126.1 (C), 128.4 (C), 128.6 (C), 141.2 (C), 158.1 (d, *J* = 251.7 Hz, CF). <sup>19</sup>F NMR (CDCl<sub>3</sub>, 376 MHz): δ -110.18 (dt, *J* = 37.4, 17.8 Hz). HRMS (DART-TOF) *m/z*: [M]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>33</sub>BF<sub>1</sub>O<sub>2</sub><sup>+</sup> = 347.2552, Found 347.2569. Specific rotation: [α]<sub>D</sub><sup>20</sup> = +5.5 (*c* 1.0, CHCl<sub>3</sub>). Enantiomeric purity of **S1** was determined by HPLC analysis after oxidation of **S1** to the corresponding allylic alcohol in comparison with authentic racemic material (ADH column, 99:1 hexanes:*i*PrOH, 0.5 mL/min, 254 nm). (*S*)-enantiomer *t*<sub>r</sub> = 48.7 min, (*R*)-enantiomer *t*<sub>r</sub> = 58.5 min.



| Peak # | Ret. Time  | Area    | Area % | Peak # | Ret. Time  | Area    | Area % |
|--------|------------|---------|--------|--------|------------|---------|--------|
| 1      | 50.484 min | 3106074 | 49.582 | 1      | 48.676 min | 7350040 | 84.853 |
| 2      | 59.742 min | 3158466 | 50.418 | 2      | 58.496 min | 1312037 | 15.147 |

**(E)-(3,3-Difluoronon-4-en-1-yl)benzene (S2).**

Prepared through olefin metathesis from (3,3-Difluoropent-4-en-1-yl)benzene (1.0 equiv.) and *cis*-5-decene (2.0 equiv.), isolated as colorless oil in 79% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 0.92 ppm (3H, t, *J* = 7.1 Hz), 1.37 (2H, dtdd, *J* = 11.5, 8.0, 4.7, 2.0 Hz), 2.36–2.11 (2H, m), 2.88–2.73 (1H, m), 5.56–5.44 (1H, m), 5.75 (1H, dtt, *J* = 11.4,

7.7, 1.8 Hz), 7.24–7.17 (2H, m), 7.34–7.27 (1H, m). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 14.0 (CH<sub>3</sub>), 22.5 (CH<sub>2</sub>), 28.2 (t, *J* = 1.7 Hz, CH<sub>2</sub>), 28.8 (t, *J* = 4.4 Hz, CH<sub>2</sub>), 31.7 (t, *J* = 1.3 Hz, CH<sub>2</sub>), 40.6 (t, *J* = 27.0 Hz, CH<sub>2</sub>), 122.0 (C), 125.1–123.8 (m, CH), 126.3 (C), 128.4 (C), 128.7 (C), 138.6 (t, *J* = 6.2 Hz, CH), 140.8 (C). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ –88.88 to –93.35 (m). HRMS (DART) Calcd for C<sub>15</sub>H<sub>20</sub>F<sub>2</sub> [M]<sup>+</sup>: 238.1528; Found 238.1518.

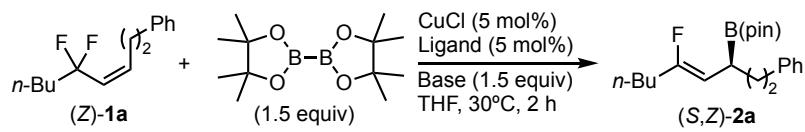
### **(3,3-Difluoropent-4-en-1-yl)benzene (S3).**

A 20 mL vial equipped with a magnetic stirring bar was charged with 5-phenylpent-1-en-3-one (1.02 g, 6.4 mmol, 1.0 equiv.), then DAST (3.08 g, 19.1 mmol, 3.0 equiv.) was added dropwise. Ethanol (40 μL) was carefully added, the vial tightly capped and allowed to stir for 12 h at 65 °C. The mixture was then diluted in dichloromethane (20 mL) and slowly introduced to a sat. aq. NaHCO<sub>3</sub> solution. The organic layer was separated, dried over MgSO<sub>4</sub> and concentrated. The oily reddish residue is subjected to silica gel chromatography affording the product as colorless liquid in 35% yield (405 mg, 2.2 mmol).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 2.36–2.06 (2H, m), 2.90–2.67 (2H, m), 5.45 (1H, dq, *J* = 11.0, 0.7 Hz), 5.66 (1H, dtd, *J* = 17.4, 2.6, 0.8 Hz), 5.95 (1H, dtd, *J* = 17.3, 11.1, 10.6 Hz), 7.24–7.17 (3H, m), 7.30 (2H, ddt, *J* = 8.0, 6.6, 0.9 Hz). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 28.7 (t, *J* = 4.6 Hz), 39.1 (t, *J* = 26.5 Hz, CH<sub>2</sub>), 119.5 (t, *J* = 9.5 Hz, CH<sub>2</sub>), 121.0 (t, *J* = 238.8 Hz, CF<sub>2</sub>), 126.4 (C), 128.4 (C), 128.7 (C), 133.1 (C), 140.7 (C), (t, *J* = 27.4 Hz, CH). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ –98.48 to –98.70 (m).

## 10. Optimization Conditions for the borylation products

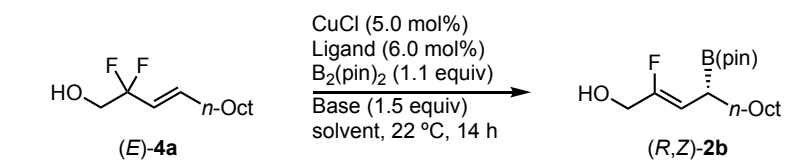
**Table S5.** Optimization of the Reaction Conditions for the synthesis of (*S,Z*)-**2a**<sup>a</sup>



| Entry           | Ligand                  | Base                | Conv. (%) <sup>b</sup> | Z:E <sup>b</sup> | e.r. <sup>c</sup> |
|-----------------|-------------------------|---------------------|------------------------|------------------|-------------------|
| 1 <sup>d</sup>  | ( <i>R,R</i> )-BenzP*   | NaOMe               | 99 (93)                | 95:5             | 97:3              |
| 2               | ( <i>R,R</i> )-QuinoxP* | NaOMe               | 93                     | 95:5             | 96:4              |
| 3               | ( <i>R,S</i> )-Josiphos | NaOMe               | 66                     | 93:8             | 83.5:6.5          |
| 4               | ( <i>R</i> )-BINAP      | NaOMe               | trace                  | –                | –                 |
| 5               | ( <i>R</i> )-Segphos    | NaOMe               | 36                     | 91:9             | 50:50             |
| 6               | ( <i>R,R</i> )-BenzP*   | KOMe                | 85                     | 96:4             | 95:5              |
| 7               | ( <i>R,R</i> )-BenzP*   | LiOMe               | trace                  | –                | –                 |
| 8               | ( <i>R,R</i> )-BenzP*   | Na(O- <i>t</i> -Bu) | 16                     | –                | –                 |
| 9 <sup>e</sup>  | ( <i>R,R</i> )-BenzP*   | NaOMe               | 97                     | 97:3             | 96.5:3.5          |
| 10 <sup>f</sup> | ( <i>R,R</i> )-BenzP*   | NaOMe               | 70                     | 94:6             | 96:4              |

<sup>a</sup>Conditions: **1a** (0.20 mmol), CuCl (0.0100 mmol), ligand (0.0100 mmol), base (0.350 mmol), diboron (0.350 mmol) in THF (400  $\mu$ L). <sup>b</sup>Determined by <sup>19</sup>F NMR analysis of crude mixture with an internal standard. <sup>c</sup>The e.r. values of the products were determined by HPLC analysis of the saturated alcohols derived from the corresponding boronates. <sup>d</sup>0.50 mmol scale, Isolated yield. <sup>e</sup>The reaction was conducted at 0°C. <sup>f</sup>Toluene was used as solvent.

**Table S6.** Optimization of the Reaction Conditions for the synthesis of (*R,Z*)-**2b**<sup>a</sup>



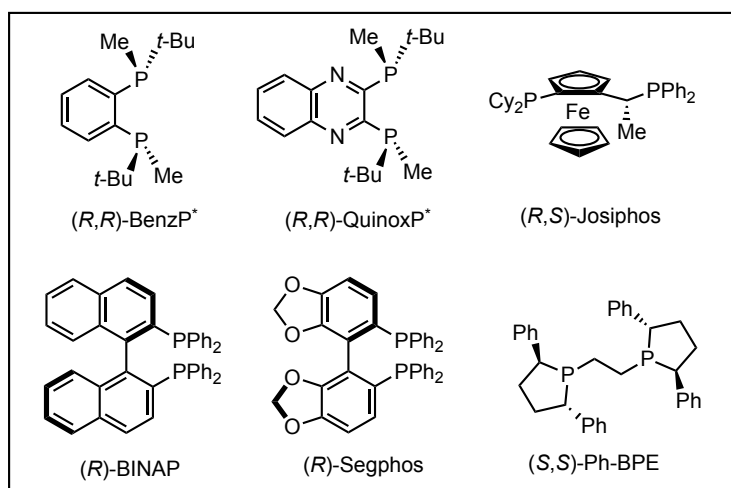
| Entry | Ligand                  | Base             | Solvent | Conv. (%) <sup>b</sup> | Z:E <sup>b</sup> | e.r. <sup>c</sup> |
|-------|-------------------------|------------------|---------|------------------------|------------------|-------------------|
| 1     | ( <i>S,S</i> )-Ph-BPE   | LiO <i>t</i> -Bu | toluene | 98                     | >98:2            | 95:5              |
| 2     | ( <i>S,S</i> )-Ph-BPE   | NaOMe            | THF     | 30                     | 98:2             | 90:10             |
| 3     | ( <i>R,R</i> )-QuinoxP* | NaOMe            | THF     | 45                     | 98:2             | 93:7              |

<sup>a</sup>Conditions: **4a** (0.10 mmol), CuCl (0.005 mmol), ligand (0.006 mmol), base (0.11 mmol), diboron (0.15 mmol) in solvent (1 mL). <sup>b</sup>Determined by <sup>19</sup>F NMR analysis of crude mixture with an internal standard. <sup>c</sup>The e.r. values of the products were determined by HPLC analysis of the saturated alcohols derived from the corresponding boronates.

**Table S7.** Comparison Data of the Reaction Conditions for the synthesis of (*S,Z*)-**2b**<sup>a</sup>

| Entry          | Base    | Solvent | Conv. (%) <sup>b</sup> | Z:E <sup>b</sup> | e.r. <sup>c</sup> |
|----------------|---------|---------|------------------------|------------------|-------------------|
| 1 <sup>d</sup> | NaOMe   | THF     | 80                     | 96:4             | 92.5:7.5          |
| 2              | LiOt-Bu | THF     | 52                     | 89:11            | 97.5:2.5          |
| 3              | NaOMe   | toluene | 92                     | 93:7             | 99:1              |
| 4              | LiOt-Bu | toluene | 35                     | 90:10            | 97.5:2.5          |

<sup>a</sup>Conditions: **1a** (0.10 mmol), CuCl (0.005 mmol), ligand (0.006 mmol), base (0.11 mmol), diboron (0.15 mmol) in solvent (1 mL). <sup>b</sup>Determined by <sup>19</sup>F NMR analysis of crude mixture with an internal standard. <sup>c</sup>The e.r. values of the products were determined by HPLC analysis of the saturated alcohols derived from the corresponding boronates. <sup>d</sup>0.2 mmol scale. The reaction time was 16 h.



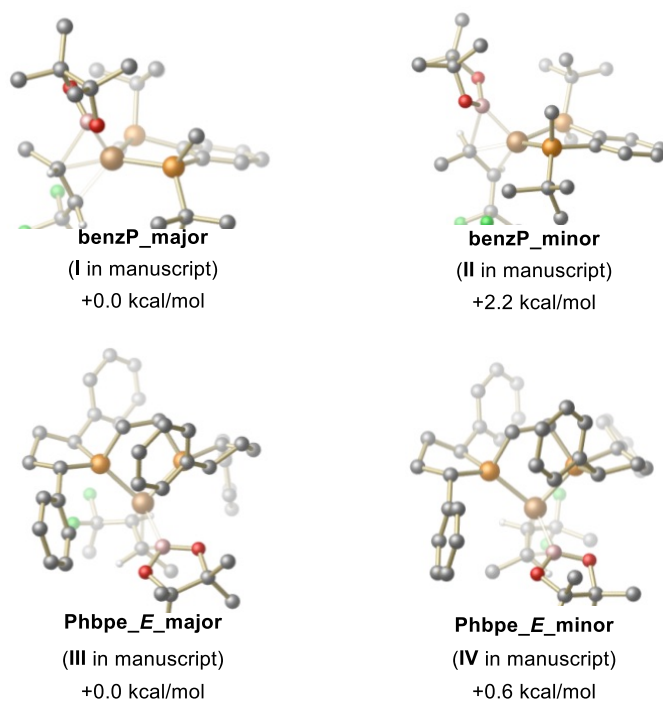
## 11. Density Functional Theory (DFT) Calculations

DFT computations<sup>12</sup> were performed with the Gaussian 09/Gaussian 16 suite of programs.<sup>13</sup> Geometries were optimized with the M06-L<sup>14</sup> functional and the Def2SVP basis set<sup>15</sup> in conjunction with the corresponding Coulomb fitting basis set to speed up calculations.<sup>16</sup> The effect of a polar reaction medium (tetrahydrofuran, THF) was approximated by means of the SMD solvation model.<sup>17</sup> Stationary points were probed through vibrational analysis and Gibbs free energy corrections were performed under standard conditions (298.15 K, 1.0 atm). Transition states have been verified through Intrinsic Reaction Coordinate calculations (IRC) employing the L(ocal) Q(uadratic) A(pproximation) method,<sup>18</sup> followed by subsequent optimization of the end points with the above mentioned optimization method. We furthermore probed the performance of various density functionals through single point energy calculations at the geometries optimized with the level described above by means of the SMD solvation model with THF as solvent and the larger def2-TZVPP<sup>14</sup> basis set. The results with M06L/DF-Def2SVP<sub>THF(SMD)</sub> and M06L/Def2TZVPP<sub>THF(SMD)</sub>/M06L/DF-Def2SVP<sub>THF(SMD)</sub> energies are reported (Table. S6–S11). A file for convenient viewing of computed geometries with the program Mercury 3.3 is appended as separate “coordinates.xyz” file in Section 2.<sup>19</sup>

### 11.1 Energies of transition states in Fig. 2

For the stereochemistry determining Cu–B addition step, the methyl group attached to the fluorine containing carbon can adopt various different conformations, which are analyzed and the lowest energy transition state are selected. The driving force for the high enantioselectivity is the steric interaction between the olefin substituent and *t*Bu group of the ligand in the minor transition states.

**Scheme S1.** 3D models for the transition states.



**Table S8.** BenzP-Cu promoted reaction transition state energies and Gibbs free energies calculated with M06-L/Def2SVP, calculations were carried out in thf with the SMD solvation model.

| Transition State   | E [Hartree]  | $\Delta E$ [kcal/mol] | G [Hartree]  | $\Delta G$ [kcal/mol] | $\Delta G_{\text{corr}}$ [kcal/mol] | Freq [ $\text{cm}^{-1}$ ] |
|--------------------|--------------|-----------------------|--------------|-----------------------|-------------------------------------|---------------------------|
| <b>benzP_major</b> | -3754.449082 | 0.0                   | -3753.815863 | 0.0                   | 0.0                                 | -111.0521                 |
| <b>benzP_minor</b> | -3754.444127 | 3.1                   | -3753.811742 | 2.6                   | -0.5                                | -118.70                   |

**Table S9.** BenzP-Cu promoted reaction transition state single point energies calculated with M06-L/Def2TZVPP, calculations were carried out in thf with the SMD solvation model.

| Transition State   | E [Hartree]  | $\Delta E$ [kcal/mol] | $\Delta G$ [kcal/mol] |
|--------------------|--------------|-----------------------|-----------------------|
| <b>benzP_major</b> | -3756.491896 | 0.0                   | 0.0                   |
| <b>benzP_minor</b> | -3756.487545 | 2.7                   | 2.2                   |

**Table S10.** Phenyl-bpe-Cu promoted *E* olefin reaction transition state energies and Gibbs free energies calculated with M06-L/Def2SVP, calculations were carried out in thf with the SMD solvation model.

| Transition State | E            | $\Delta E$ | G            | $\Delta G$ | $\Delta G_{\text{corr}}$ | Freq                |
|------------------|--------------|------------|--------------|------------|--------------------------|---------------------|
|                  | [Hartree]    | [kcal/mol] | [Hartree]    | [kcal/mol] | [kcal/mol]               | [cm <sup>-1</sup> ] |
| Phbpe_E_major    | -4444.648098 | 0.0        | -4443.822364 | 0.0        | 0.0                      | -44.73              |
| Phbpe_E_minor    | -4444.647524 | 0.4        | -4443.817626 | 3.0        | 2.6                      | -140.55             |

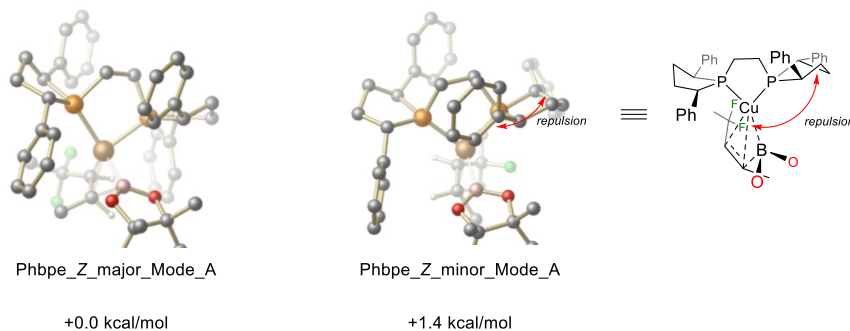
**Table S11.** Phenyl-bpe-Cu promoted *E* olefin reaction transition state single point energies calculated with M06-L /Def2TZVPP, calculations were carried out in thf with the SMD solvation model.

| Transition State | E            | $\Delta E$ | $\Delta G$ |
|------------------|--------------|------------|------------|
|                  | [Hartree]    | [kcal/mol] | [kcal/mol] |
| Phbpe_E_major    | -4447.395047 | 0.0        | 0.0        |
| Phbpe_E_minor    | -4447.398245 | -2.0       | 0.6        |

## 11.2. Energies of transition states of the reaction with *Z* olefin promoted by phenyl-bpe

Phenyl-bpe-Cu is shown to be suitable catalyst for the reaction with *Z* olefin. DFT studies are carried out to investigate the origin of enantioselectivity. Similar to the reactions with *E* olefins, there is significant steric repulsion between the phenyl ring in the ligand and the CF<sub>2</sub>Me substituent of the olefin in the minor transition state. A series of conformations are analyzed and the lowest energy conformation is selected.

**Scheme S2.** 3D models for the transition states involving *Z* olefin and phenyl-bpe-Cu complex.



**Table S12.** Phenyl-bpe-Cu promoted Z olefin reaction transition state energies and Gibbs free energies calculated with M06-L/Def2SVP, calculations were carried out in thf with the SMD solvation model.

| <b>Transition</b>    | <b>E</b>     | <b><math>\Delta E</math></b> | <b>G</b>     | <b><math>\Delta G</math></b> | <b><math>\Delta G_{\text{corr}}</math></b> | <b>Freq</b>         |
|----------------------|--------------|------------------------------|--------------|------------------------------|--|---------------------|
| <b>State</b>         | [Hartree]    | [kcal/mol]                   | [Hartree]    | [kcal/mol]                   | [kcal/mol]                                 | [cm <sup>-1</sup> ] |
| <b>Phbpe_Z_major</b> | -4444.643311 | 2.2                          | -4443.815522 | 0.9                          | -1.4                                       | -85.19              |
| <b>Phbpe_Z_minor</b> | -4444.643477 | 2.1                          | -4443.813744 | 2.0                          | -0.2                                       | -147.29             |

**Table S13.** Phenyl-bpe-Cu promoted Z olefin reaction transition state single point energies calculated with M06-L/Def2TZVPP, calculations were carried out in thf with the SMD solvation model.

| <b>Transition</b>    | <b>E</b>     | <b><math>\Delta E</math></b> | <b><math>\Delta G</math></b> |
|----------------------|--------------|------------------------------|------------------------------|
| <b>State</b>         | [Hartree]    | [kcal/mol]                   | [kcal/mol]                   |
| <b>Phbpe_Z_major</b> | -4447.393365 | 0.0                          | 0.0                          |
| <b>Phbpe_Z_minor</b> | -4447.393022 | 0.2                          | 1.4                          |



### 11.3. Coordinates after optimization with M06L/DF-Def2SVP<sub>THF(SMD)</sub>

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benzP\_Major / electronic energy: -3754.449082 a.u. / lowest freq: -111.0521 cm<sup>-1</sup>

|    |           |           |           |
|----|-----------|-----------|-----------|
| C  | 4.677919  | -2.402873 | 0.282091  |
| C  | 3.513870  | -2.297138 | -0.475714 |
| C  | 4.863299  | -1.567334 | 1.382402  |
| C  | 3.858560  | -0.674962 | 1.750311  |
| C  | 2.670269  | -0.571543 | 1.011147  |
| C  | 2.521263  | -1.358891 | -0.155359 |
| H  | 5.443054  | -3.133492 | 0.008485  |
| H  | 3.377949  | -2.962235 | -1.331507 |
| H  | 5.781950  | -1.625976 | 1.971341  |
| H  | 4.002145  | -0.058057 | 2.641091  |
| P  | 1.250870  | 0.455179  | 1.591764  |
| P  | 1.057286  | -1.088712 | -1.243241 |
| C  | 0.550605  | -0.586147 | 3.022707  |
| Cu | -0.136755 | 0.603509  | -0.266438 |
| C  | 2.065504  | 1.850712  | 2.445220  |
| C  | 0.373348  | -2.774176 | -1.408123 |
| C  | 0.042768  | -1.894688 | 2.426683  |
| C  | -0.626994 | 0.210565  | 3.582009  |
| C  | 1.558043  | -0.877343 | 4.125732  |
| H  | 2.760391  | 1.547889  | 3.241022  |
| H  | 1.300085  | 2.503262  | 2.885578  |
| H  | 2.616640  | 2.450712  | 1.708698  |
| H  | -0.672220 | -1.706015 | 1.609941  |
| H  | -0.477431 | -2.487883 | 3.196700  |
| H  | 0.862167  | -2.517821 | 2.033044  |
| H  | 2.397115  | -1.497219 | 3.777292  |
| H  | 1.066190  | -1.438551 | 4.937372  |
| H  | 1.971050  | 0.035674  | 4.579939  |
| H  | -1.387853 | 0.402936  | 2.811003  |
| H  | -0.314229 | 1.174146  | 4.013024  |
| H  | -1.114664 | -0.359174 | 4.390095  |
| C  | 1.733358  | -0.712366 | -2.976023 |

C 2.616825 0.523537 -2.855914  
C 0.492083 -0.383739 -3.808196  
C 2.502334 -1.843025 -3.646831  
H -0.114386 0.407904 -3.342687  
H -0.159540 -1.259642 -3.949889  
H 0.788930 -0.035077 -4.810202  
H -0.063094 -3.079313 -0.447263  
H 1.129248 -3.519638 -1.695380  
H -0.431034 -2.776438 -2.156197  
H 3.477238 -2.030253 -3.176868  
H 2.710051 -1.571480 -4.695038  
H 1.940835 -2.788812 -3.671324  
H 2.081202 1.345749 -2.358663  
H 2.926066 0.874128 -3.853675  
H 3.533387 0.321872 -2.279378  
B -2.050511 -0.092286 -0.142459  
O -2.860261 -0.186405 0.974557  
O -2.579988 -0.834731 -1.193012  
C -4.095165 -0.846808 0.597823  
C -3.663259 -1.645201 -0.673656  
C -5.113993 0.237751 0.290179  
C -4.561633 -1.697202 1.758772  
C -4.733300 -1.770380 -1.736361  
C -3.097400 -3.015063 -0.335894  
H -3.776072 -2.377469 2.112555  
H -5.441051 -2.298051 1.483462  
H -4.850311 -1.057747 2.604917  
H -2.319631 -2.952097 0.439217  
H -2.645410 -3.462774 -1.231561  
H -3.878766 -3.701476 0.020400  
H -5.059109 -0.792720 -2.112739  
H -5.615209 -2.301611 -1.348646  
H -4.352131 -2.344353 -2.592688  
H -4.793388 0.868895 -0.551600  
H -5.242850 0.885950 1.168320  
H -6.098264 -0.184695 0.042102

|   |           |          |           |
|---|-----------|----------|-----------|
| C | -1.614451 | 1.939518 | -0.663327 |
| C | -0.326092 | 2.413437 | -1.197918 |
| C | -2.269647 | 2.689206 | 0.477377  |
| H | -1.674789 | 2.658374 | 1.399120  |
| H | -3.258167 | 2.281273 | 0.718648  |
| H | -2.395869 | 3.750732 | 0.212720  |
| H | -2.332894 | 1.702707 | -1.463297 |
| C | 0.331839  | 3.642325 | -0.711402 |
| C | 1.708065  | 3.913029 | -1.251694 |
| F | 0.400457  | 3.698744 | 0.674129  |
| F | -0.445141 | 4.795133 | -1.014920 |
| H | 2.412445  | 3.143389 | -0.911769 |
| H | 2.059159  | 4.892213 | -0.900525 |
| H | 1.699604  | 3.918480 | -2.348843 |
| H | -0.216380 | 2.333443 | -2.286669 |

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benzP\_Minor / electronic enery: -3754.444127 a.u. / lowest freq: -118.7019 cm<sup>-1</sup>

|    |           |           |           |
|----|-----------|-----------|-----------|
| C  | -4.868574 | 1.532579  | -1.379345 |
| C  | -3.736210 | 0.840454  | -1.802571 |
| C  | -4.891767 | 2.114220  | -0.112779 |
| C  | -3.764128 | 2.036375  | 0.700835  |
| C  | -2.609863 | 1.357600  | 0.281925  |
| C  | -2.610266 | 0.707328  | -0.975683 |
| H  | -5.737026 | 1.609288  | -2.038209 |
| H  | -3.737976 | 0.390117  | -2.796902 |
| H  | -5.782183 | 2.642736  | 0.236489  |
| H  | -3.782542 | 2.522046  | 1.680036  |
| P  | -1.074668 | 1.355902  | 1.299611  |
| P  | -1.167301 | -0.353003 | -1.431906 |
| C  | -0.423380 | 3.130415  | 1.127760  |
| Cu | 0.118000  | -0.456017 | 0.467484  |
| C  | -1.710269 | 1.293815  | 3.012357  |
| C  | -0.474266 | 0.515819  | -2.885433 |
| C  | 0.107984  | 3.266492  | -0.294609 |
| C  | 0.727496  | 3.252540  | 2.125728  |
| C  | -1.463058 | 4.208909  | 1.398021  |

|   |           |           |           |
|---|-----------|-----------|-----------|
| H | -2.323786 | 2.159639  | 3.302141  |
| H | -0.865886 | 1.223196  | 3.712207  |
| H | -2.312243 | 0.381876  | 3.132470  |
| H | 0.867011  | 2.497817  | -0.507544 |
| H | 0.577947  | 4.253624  | -0.436753 |
| H | -0.695704 | 3.177943  | -1.043754 |
| H | -2.259303 | 4.224847  | 0.639923  |
| H | -0.981799 | 5.200806  | 1.374746  |
| H | -1.934391 | 4.111043  | 2.387810  |
| H | 1.473040  | 2.455007  | 1.988319  |
| H | 0.375784  | 3.228704  | 3.168378  |
| H | 1.244930  | 4.215694  | 1.983860  |
| C | -1.828218 | -1.963911 | -2.184612 |
| C | -2.852764 | -2.546400 | -1.218519 |
| C | -0.591696 | -2.864733 | -2.234469 |
| C | -2.409571 | -1.861930 | -3.589923 |
| H | -0.134577 | -2.968012 | -1.241709 |
| H | 0.177475  | -2.474335 | -2.919685 |
| H | -0.866676 | -3.870584 | -2.590484 |
| H | 0.032483  | 1.432700  | -2.556212 |
| H | -1.244344 | 0.788145  | -3.622424 |
| H | 0.274875  | -0.122489 | -3.374602 |
| H | -3.344210 | -1.287964 | -3.640348 |
| H | -2.649035 | -2.873680 | -3.956683 |
| H | -1.701705 | -1.427033 | -4.310225 |
| H | -2.451606 | -2.609724 | -0.195941 |
| H | -3.129723 | -3.567697 | -1.525630 |
| H | -3.777641 | -1.950396 | -1.185110 |
| B | 2.035325  | -0.027165 | -0.042400 |
| O | 2.759177  | 0.994997  | 0.553493  |
| O | 2.660290  | -0.462727 | -1.197116 |
| C | 4.043727  | 1.092517  | -0.108632 |
| C | 3.743824  | 0.452511  | -1.503231 |
| C | 5.029280  | 0.279736  | 0.714248  |
| C | 4.459626  | 2.546761  | -0.150699 |
| C | 4.892453  | -0.333127 | -2.097484 |

|   |           |           |           |
|---|-----------|-----------|-----------|
| C | 3.216432  | 1.460622  | -2.510737 |
| H | 3.682739  | 3.185768  | -0.590615 |
| H | 5.384806  | 2.679051  | -0.731051 |
| H | 4.653011  | 2.915137  | 0.866941  |
| H | 2.387315  | 2.051374  | -2.094855 |
| H | 2.841389  | 0.934409  | -3.399499 |
| H | 4.000077  | 2.157247  | -2.841275 |
| H | 5.196826  | -1.169196 | -1.455281 |
| H | 5.768679  | 0.311744  | -2.261095 |
| H | 4.603182  | -0.751253 | -3.071852 |
| H | 4.742328  | -0.780927 | 0.756775  |
| H | 5.048345  | 0.660101  | 1.745249  |
| H | 6.051625  | 0.341254  | 0.314810  |
| C | 1.574816  | -1.578260 | 1.351634  |
| C | 0.265275  | -1.790308 | 1.998457  |
| C | 2.255569  | -2.744981 | 0.668049  |
| H | 2.382186  | -3.575543 | 1.379830  |
| H | 3.249095  | -2.475434 | 0.286025  |
| H | 1.684655  | -3.138671 | -0.182155 |
| H | 2.276088  | -1.030200 | 1.998690  |
| C | -0.373167 | -3.112599 | 2.127915  |
| C | -1.732094 | -3.111701 | 2.771174  |
| F | 0.425514  | -4.005210 | 2.894122  |
| F | -0.465109 | -3.788097 | 0.915365  |
| H | -1.671628 | -2.697690 | 3.785588  |
| H | -2.116808 | -4.137699 | 2.836299  |
| H | -2.436298 | -2.503157 | 2.190133  |
| H | 0.129605  | -1.225659 | 2.929902  |

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Phenyl\_bpe\_E\_Major / electronic energy: -4444.648098 a.u. / lowest freq: -44.7263 cm<sup>-1</sup>

|   |           |           |           |
|---|-----------|-----------|-----------|
| P | -0.263335 | -1.982130 | -0.107530 |
| C | 0.960702  | -3.405460 | -0.283944 |
| C | 0.281723  | -4.367793 | -1.266928 |
| C | 2.378926  | -3.043604 | -0.625075 |
| H | 0.967045  | -3.904793 | 0.701155  |
| H | 0.370694  | -3.976981 | -2.295449 |

|   |           |           |           |
|---|-----------|-----------|-----------|
| H | 0.778664  | -5.350647 | -1.257457 |
| C | -1.176176 | -4.441915 | -0.854242 |
| C | -1.695318 | -3.007773 | -0.809595 |
| H | -1.780685 | -5.062592 | -1.535050 |
| H | -1.237626 | -4.914473 | 0.140589  |
| H | -1.784351 | -2.645171 | -1.843036 |
| C | -2.996768 | -2.743364 | -0.108876 |
| C | -0.586086 | -2.054951 | 1.715868  |
| C | -1.418706 | -0.889009 | 2.229914  |
| H | -1.057544 | -3.019329 | 1.973052  |
| H | 0.407310  | -2.067958 | 2.192275  |
| H | -2.482999 | -1.030323 | 1.980959  |
| H | -1.365314 | -0.820714 | 3.330919  |
| P | -0.926355 | 0.726224  | 1.468467  |
| C | -2.264393 | 1.888484  | 2.131458  |
| C | 0.361500  | 1.342213  | 2.710482  |
| C | -0.458807 | 2.018200  | 3.802142  |
| C | 1.331004  | 0.270645  | 3.111901  |
| H | 0.904709  | 2.115070  | 2.143036  |
| H | -0.940952 | 1.265764  | 4.449837  |
| H | 0.174958  | 2.636726  | 4.458623  |
| C | -1.530308 | 2.828355  | 3.098341  |
| C | -3.515809 | -3.603156 | 0.870575  |
| C | -3.713064 | -1.567646 | -0.397087 |
| C | -4.901500 | -1.268773 | 0.264831  |
| C | -4.701653 | -3.298679 | 1.542052  |
| C | -5.401543 | -2.130702 | 1.242922  |
| H | -3.319390 | -0.876569 | -1.146564 |
| H | -2.991649 | -4.529416 | 1.119239  |
| H | -5.083369 | -3.987330 | 2.300618  |
| H | -5.444646 | -0.355151 | 0.009347  |
| H | -6.334901 | -1.897912 | 1.761931  |
| C | 3.431918  | -3.777101 | -0.056353 |
| C | 2.695766  | -2.017414 | -1.524972 |
| C | 4.759533  | -3.498010 | -0.376240 |
| C | 4.023289  | -1.730883 | -1.840959 |

|   |           |           |           |
|---|-----------|-----------|-----------|
| C | 5.060211  | -2.469223 | -1.270839 |
| H | 6.100480  | -2.240610 | -1.517655 |
| H | 3.200253  | -4.576527 | 0.655460  |
| H | 5.563506  | -4.080987 | 0.081090  |
| H | 4.242768  | -0.911149 | -2.529215 |
| H | 1.893700  | -1.414506 | -1.961809 |
| u | -0.165559 | 0.325613  | -0.731617 |
| B | 1.536822  | 1.472471  | -0.713360 |
| O | 1.521452  | 2.686502  | -0.047395 |
| O | 2.787393  | 1.212014  | -1.225231 |
| H | -2.237617 | 3.299224  | 3.798343  |
| H | -1.059134 | 3.649050  | 2.530491  |
| C | 2.341915  | -0.123271 | 2.217112  |
| C | 1.235241  | -0.415844 | 4.331206  |
| C | 3.209102  | -1.170338 | 2.517155  |
| C | 2.100528  | -1.469833 | 4.632989  |
| C | 3.086836  | -1.857680 | 3.726475  |
| C | -3.036603 | 2.567810  | 1.038966  |
| H | -2.953316 | 1.252054  | 2.712190  |
| C | -2.389848 | 3.417890  | 0.127993  |
| C | -4.412808 | 2.359747  | 0.889257  |
| C | -5.126264 | 2.978833  | -0.138530 |
| C | -3.100881 | 4.046226  | -0.891987 |
| C | -4.473100 | 3.826760  | -1.031784 |
| H | -1.308426 | 3.576117  | 0.216071  |
| H | -4.930803 | 1.699113  | 1.591329  |
| H | -6.199944 | 2.798340  | -0.238906 |
| H | -2.579495 | 4.706680  | -1.590309 |
| H | -5.029543 | 4.315037  | -1.835624 |
| H | 2.001081  | -1.990572 | 5.589114  |
| H | 3.762596  | -2.683269 | 3.964275  |
| H | 2.433844  | 0.383761  | 1.253394  |
| H | 3.981586  | -1.451741 | 1.795689  |
| H | 0.471652  | -0.132257 | 5.060002  |
| C | 2.842530  | 3.283530  | -0.037690 |
| C | 3.699234  | 2.300556  | -0.939306 |

|   |           |           |           |
|---|-----------|-----------|-----------|
| C | 2.722118  | 4.694553  | -0.585111 |
| C | 3.298881  | 3.358486  | 1.410580  |
| C | 4.915326  | 1.717806  | -0.242233 |
| C | 4.118878  | 2.890214  | -2.275220 |
| H | 3.330239  | 2.373511  | 1.894420  |
| H | 4.297439  | 3.809325  | 1.498465  |
| H | 2.602693  | 3.988507  | 1.983266  |
| H | 4.648429  | 1.157885  | 0.663899  |
| H | 5.436896  | 1.024338  | -0.916485 |
| H | 5.630132  | 2.504124  | 0.039852  |
| H | 2.318208  | 4.722249  | -1.604383 |
| H | 2.046684  | 5.281309  | 0.053551  |
| H | 3.696119  | 5.204689  | -0.592235 |
| H | 3.259905  | 3.239095  | -2.863367 |
| H | 4.812401  | 3.733854  | -2.150703 |
| H | 4.633158  | 2.122675  | -2.870644 |
| H | -0.271363 | 3.055162  | -3.690890 |
| H | 0.839152  | 0.800903  | -3.077502 |
| H | 1.092793  | 3.235921  | -2.574134 |
| C | 0.087806  | 2.806201  | -2.676929 |
| C | 0.056378  | 1.312255  | -2.496889 |
| H | -2.111073 | 1.281818  | -2.433620 |
| C | -1.221889 | 0.639119  | -2.502496 |
| H | -0.567205 | 3.323173  | -1.963577 |
| H | -2.157472 | 0.733675  | -5.147592 |
| H | -0.398273 | 0.466741  | -5.198467 |
| C | -1.362766 | 0.000180  | -4.961899 |
| C | -1.391340 | -0.435355 | -3.520235 |
| F | -2.592978 | -1.107476 | -3.330378 |
| H | -1.513420 | -0.860316 | -5.628066 |
| F | -0.408044 | -1.413745 | -3.370721 |

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Phenyl\_bpe\_E\_Minor / electronic enery: -4444.647524 a.u. / lowest freq: -140.5469 cm<sup>-1</sup>

|   |           |          |           |
|---|-----------|----------|-----------|
| P | -0.179029 | 2.012768 | -0.737605 |
| C | -1.619279 | 3.214077 | -1.065010 |
| C | -1.149069 | 4.086921 | -2.227311 |



|   |           |           |           |
|---|-----------|-----------|-----------|
| C | -2.965844 | 2.564443  | -1.198858 |
| H | -1.639744 | 3.843522  | -0.158717 |
| H | -1.262706 | 3.552207  | -3.185092 |
| H | -1.761399 | 4.998993  | -2.303931 |
| C | 0.318837  | 4.383079  | -1.991616 |
| C | 1.006218  | 3.035739  | -1.804602 |
| H | 0.775019  | 4.954126  | -2.815918 |
| H | 0.421797  | 5.002027  | -1.084283 |
| H | 0.987307  | 2.503319  | -2.772207 |
| C | 2.415698  | 3.022449  | -1.297601 |
| C | 0.274815  | 2.483260  | 0.999094  |
| C | 1.264230  | 1.523629  | 1.646543  |
| H | 0.655254  | 3.518911  | 1.029496  |
| H | -0.676106 | 2.489823  | 1.559407  |
| H | 2.260666  | 1.632723  | 1.183338  |
| H | 1.389534  | 1.749762  | 2.718157  |
| P | 0.818713  | -0.250867 | 1.387885  |
| C | 2.155542  | -1.193127 | 2.327586  |
| C | -0.496132 | -0.712373 | 2.690785  |
| C | 0.179546  | -1.707829 | 3.646092  |
| C | -1.177957 | 0.452449  | 3.345657  |
| H | -1.238594 | -1.255799 | 2.086270  |
| H | 0.545676  | -1.185182 | 4.543679  |
| H | -0.548048 | -2.453264 | 4.001441  |
| C | 1.358947  | -2.342322 | 2.935624  |
| C | 2.935632  | 4.039098  | -0.482573 |
| C | 3.247936  | 1.935472  | -1.610647 |
| C | 4.554927  | 1.869986  | -1.136406 |
| C | 4.243306  | 3.969884  | 0.001464  |
| C | 5.059754  | 2.886668  | -0.323407 |
| H | 2.860490  | 1.128148  | -2.236366 |
| H | 2.319581  | 4.904283  | -0.224234 |
| H | 4.628420  | 4.776397  | 0.631119  |
| H | 5.180773  | 1.016146  | -1.407825 |
| H | 6.086548  | 2.839074  | 0.049304  |
| C | -3.756014 | 2.379656  | -0.054701 |

|    |           |           |           |
|----|-----------|-----------|-----------|
| C  | -3.480283 | 2.126990  | -2.427853 |
| C  | -5.008391 | 1.773794  | -0.127351 |
| C  | -4.735749 | 1.524038  | -2.506163 |
| C  | -5.505773 | 1.342218  | -1.357771 |
| H  | -6.489936 | 0.870945  | -1.421778 |
| H  | -3.375084 | 2.729929  | 0.909389  |
| H  | -5.602748 | 1.643882  | 0.781821  |
| H  | -5.114585 | 1.192916  | -3.476852 |
| H  | -2.896712 | 2.256585  | -3.342802 |
| Cu | 0.044533  | -0.318847 | -0.751775 |
| B  | -1.537876 | -1.622781 | -0.676061 |
| O  | -1.269434 | -2.818102 | -0.015021 |
| O  | -2.886070 | -1.502680 | -0.941374 |
| H  | 1.981340  | -2.945980 | 3.614715  |
| H  | 1.002501  | -3.022059 | 2.141131  |
| C  | -2.449502 | 0.864312  | 2.918999  |
| C  | -0.564273 | 1.182563  | 4.377090  |
| C  | -3.091989 | 1.951485  | 3.511850  |
| C  | -1.198018 | 2.277105  | 4.961970  |
| C  | -2.469022 | 2.664934  | 4.535267  |
| C  | 3.416492  | -1.546717 | 1.596902  |
| H  | 2.426313  | -0.500564 | 3.146853  |
| C  | 3.695536  | -2.857800 | 1.186125  |
| C  | 4.386743  | -0.561429 | 1.356264  |
| C  | 5.589237  | -0.873305 | 0.726596  |
| C  | 4.896979  | -3.171357 | 0.550703  |
| C  | 5.849876  | -2.181483 | 0.316896  |
| H  | 2.966846  | -3.651714 | 1.366605  |
| H  | 4.201512  | 0.464931  | 1.689305  |
| H  | 6.333289  | -0.089481 | 0.561631  |
| H  | 5.089317  | -4.201562 | 0.239047  |
| H  | 6.793663  | -2.428132 | -0.175475 |
| H  | -0.696652 | 2.828829  | 5.761377  |
| H  | -2.971533 | 3.516501  | 5.000333  |
| H  | -2.935561 | 0.325786  | 2.099514  |
| H  | -4.089352 | 2.243395  | 3.171278  |

|   |           |           |           |
|---|-----------|-----------|-----------|
| H | 0.430844  | 0.894337  | 4.729027  |
| C | -2.449889 | -3.656544 | -0.113834 |
| C | -3.586877 | -2.600890 | -0.301307 |
| C | -2.269045 | -4.545662 | -1.332960 |
| C | -2.557549 | -4.496282 | 1.139409  |
| C | -4.136359 | -2.092483 | 1.019820  |
| C | -4.719742 | -3.038433 | -1.204737 |
| H | -2.539844 | -3.884036 | 2.050264  |
| H | -3.485304 | -5.087475 | 1.142555  |
| H | -1.716187 | -5.201511 | 1.196539  |
| H | -3.330534 | -1.810285 | 1.711828  |
| H | -4.757052 | -1.202810 | 0.844630  |
| H | -4.761282 | -2.847164 | 1.518412  |
| H | -2.203029 | -3.959188 | -2.261360 |
| H | -1.340583 | -5.125353 | -1.233200 |
| H | -3.097241 | -5.259384 | -1.447017 |
| H | -4.374207 | -3.265925 | -2.221119 |
| H | -5.223242 | -3.930789 | -0.804144 |
| H | -5.471862 | -2.240382 | -1.281641 |
| H | -1.103150 | 0.514687  | -3.372075 |
| H | -0.625925 | -2.405165 | -2.464955 |
| H | -2.397824 | -0.680310 | -3.150467 |
| C | -1.332612 | -0.562318 | -3.387982 |
| C | -0.433989 | -1.320604 | -2.443400 |
| H | 1.246804  | -0.152345 | -3.183588 |
| C | 0.981371  | -0.948409 | -2.475054 |
| H | -1.172000 | -0.909024 | -4.423098 |
| H | 1.406123  | -2.906615 | -0.534023 |
| H | 0.932574  | -3.782336 | -2.006023 |
| C | 1.732865  | -3.192616 | -1.541861 |
| C | 2.033855  | -1.975358 | -2.366164 |
| F | 3.222275  | -1.405399 | -1.907110 |
| H | 2.630086  | -3.820462 | -1.474761 |
| F | 2.413560  | -2.433614 | -3.649133 |

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Phenyl\_boe\_Z\_Major / electronic energy: -4444.643311 a.u. / lowest freq: -85.1912 cm<sup>-1</sup>

|   |           |           |           |
|---|-----------|-----------|-----------|
| P | 0.396102  | 2.069080  | -0.293021 |
| C | -0.872556 | 3.445139  | -0.616354 |
| C | -0.090703 | 4.582940  | -1.291939 |
| C | -2.085626 | 3.002188  | -1.380912 |
| H | -1.190830 | 3.778686  | 0.385635  |
| H | -0.061636 | 4.410561  | -2.381276 |
| H | -0.603099 | 5.546141  | -1.145480 |
| C | 1.322324  | 4.580968  | -0.745202 |
| C | 1.835811  | 3.155142  | -0.886712 |
| H | 1.975825  | 5.298994  | -1.266771 |
| H | 1.309075  | 4.885968  | 0.315017  |
| H | 1.931002  | 2.933742  | -1.964027 |
| C | 3.122391  | 2.789608  | -0.211107 |
| C | 0.615121  | 2.252658  | 1.546620  |
| C | 1.388337  | 1.109680  | 2.191409  |
| H | 1.095250  | 3.221478  | 1.767246  |
| H | -0.400713 | 2.315899  | 1.967054  |
| H | 2.452463  | 1.143760  | 1.906040  |
| H | 1.357040  | 1.191556  | 3.291964  |
| P | 0.738972  | -0.526336 | 1.630259  |
| C | 1.965392  | -1.781515 | 2.334370  |
| C | -0.546867 | -0.894720 | 2.960464  |
| C | 0.274834  | -1.449284 | 4.118067  |
| C | -1.466182 | 0.253129  | 3.257152  |
| H | -1.137883 | -1.714006 | 2.515079  |
| H | 0.832776  | -0.638476 | 4.616810  |
| H | -0.368529 | -1.903772 | 4.889176  |
| C | 1.252056  | -2.443196 | 3.524840  |
| C | 3.724762  | 3.594603  | 0.764980  |
| C | 3.727140  | 1.561512  | -0.523146 |
| C | 4.891707  | 1.147609  | 0.116363  |
| C | 4.890846  | 3.178799  | 1.412093  |
| C | 5.478913  | 1.954981  | 1.093610  |
| H | 3.259307  | 0.913211  | -1.265380 |
| H | 3.286033  | 4.560831  | 1.027343  |
| H | 5.345452  | 3.822857  | 2.169535  |

|    |           |           |           |
|----|-----------|-----------|-----------|
| H  | 5.339884  | 0.187593  | -0.155322 |
| H  | 6.393868  | 1.635422  | 1.598783  |
| C  | -3.367693 | 3.106833  | -0.827725 |
| C  | -1.965989 | 2.493633  | -2.682514 |
| C  | -4.495931 | 2.709732  | -1.546606 |
| C  | -3.089039 | 2.099023  | -3.405773 |
| C  | -4.360722 | 2.203481  | -2.838937 |
| H  | -5.242706 | 1.891106  | -3.403926 |
| H  | -3.478952 | 3.514704  | 0.181186  |
| H  | -5.487115 | 2.799214  | -1.093430 |
| H  | -2.970336 | 1.699298  | -4.416626 |
| H  | -0.970770 | 2.405052  | -3.132945 |
| Cu | 0.179457  | -0.283381 | -0.592361 |
| B  | -1.619392 | -1.272085 | -0.533347 |
| O  | -1.661511 | -2.467862 | 0.174996  |
| O  | -2.876360 | -0.921254 | -0.970058 |
| H  | 1.982503  | -2.808979 | 4.262312  |
| H  | 0.698376  | -3.329130 | 3.173397  |
| C  | -2.469893 | 0.609208  | 2.338613  |
| C  | -1.349043 | 1.018275  | 4.426966  |
| C  | -3.328733 | 1.677185  | 2.587071  |
| C  | -2.198600 | 2.099323  | 4.669352  |
| C  | -3.194795 | 2.433211  | 3.753381  |
| C  | 2.451814  | -2.740271 | 1.284399  |
| H  | 2.817875  | -1.183927 | 2.698501  |
| C  | 1.578140  | -3.683150 | 0.719595  |
| C  | 3.769865  | -2.693117 | 0.816303  |
| C  | 4.209314  | -3.565583 | -0.180746 |
| C  | 2.016073  | -4.560817 | -0.268869 |
| C  | 3.335410  | -4.505996 | -0.724278 |
| H  | 0.529219  | -3.703151 | 1.032330  |
| H  | 4.460802  | -1.958994 | 1.241832  |
| H  | 5.241584  | -3.507380 | -0.535935 |
| H  | 1.316203  | -5.281484 | -0.701420 |
| H  | 3.677300  | -5.188408 | -1.506321 |
| H  | -2.084268 | 2.678221  | 5.589591  |

|   |           |           |           |
|---|-----------|-----------|-----------|
| H | -3.868148 | 3.271226  | 3.949909  |
| H | -2.565870 | 0.051618  | 1.402377  |
| H | -4.111484 | 1.916733  | 1.862492  |
| H | -0.586657 | 0.770451  | 5.169324  |
| C | -3.016117 | -2.973008 | 0.228341  |
| C | -3.789602 | -2.031015 | -0.775419 |
| C | -2.984017 | -4.433466 | -0.182936 |
| C | -3.496297 | -2.872114 | 1.666733  |
| C | -5.096926 | -1.483456 | -0.241091 |
| C | -4.007794 | -2.653946 | -2.144177 |
| H | -3.502384 | -1.838687 | 2.037192  |
| H | -4.510945 | -3.277933 | 1.785539  |
| H | -2.829796 | -3.457140 | 2.317482  |
| H | -4.955141 | -0.893937 | 0.674489  |
| H | -5.564506 | -0.827974 | -0.989040 |
| H | -5.807616 | -2.293403 | -0.020913 |
| H | -2.513763 | -4.580495 | -1.163545 |
| H | -2.406588 | -5.014917 | 0.550073  |
| H | -3.996328 | -4.861839 | -0.219920 |
| H | -3.068392 | -3.019172 | -2.582318 |
| H | -4.714443 | -3.495036 | -2.107632 |
| H | -4.421266 | -1.898119 | -2.827506 |
| C | -0.855327 | -0.906994 | -3.385001 |
| H | -0.531051 | -2.521487 | -1.971793 |
| C | -0.164030 | -1.507703 | -2.190301 |
| C | 1.271201  | -1.374099 | -1.989321 |
| H | 1.234152  | 0.373397  | -4.482724 |
| H | 1.730178  | 1.202982  | -2.989346 |
| C | 2.012327  | 0.430554  | -3.714526 |
| C | 2.202163  | -0.899228 | -3.037860 |
| F | 2.289890  | -1.860173 | -4.072474 |
| H | 2.949571  | 0.723501  | -4.205442 |
| F | 3.491659  | -0.931888 | -2.505717 |
| H | -0.515100 | -1.397653 | -4.313676 |
| H | -0.662708 | 0.167530  | -3.490281 |
| H | -1.944268 | -1.026592 | -3.322292 |

H 1.732429 -2.231262 -1.487211

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Phenyl\_boe\_Z\_Minor / electronic enery: -4444.643477 a.u. / lowest freq: -147.2905 cm<sup>-1</sup>

P -0.135321 2.068595 -0.454236

C -1.636643 3.209985 -0.654727

C -1.156150 4.395943 -1.510840

C -2.852782 2.539109 -1.227922

H -1.871028 3.570655 0.360995

H -1.298026 4.150447 -2.576920

H -1.768708 5.289530 -1.315953

C 0.316503 4.621587 -1.241984

C 0.992552 3.265682 -1.393660

H 0.757407 5.370508 -1.919924

H 0.452034 5.009603 -0.218286

H 0.898035 2.944015 -2.446800

C 2.430259 3.150719 -0.991801

C 0.361923 2.418102 1.298076

C 1.371762 1.417960 1.840773

H 0.749665 3.447422 1.387369

H -0.564712 2.392162 1.892106

H 2.372840 1.601747 1.410867

H 1.479892 1.525638 2.934276

P 0.998278 -0.347079 1.420639

C 2.483719 -1.194981 2.244243

C -0.172621 -0.932703 2.792249

C 0.702463 -1.592611 3.852989

C -1.132126 0.132341 3.229116

H -0.744047 -1.720462 2.275849

H 1.116458 -0.842569 4.546542

H 0.114848 -2.295359 4.465283

C 1.847712 -2.268425 3.124150

C 3.039862 4.039757 -0.094760

C 3.200164 2.091953 -1.498278

C 4.531011 1.925010 -1.126418

C 4.373016 3.870520 0.285067

C 5.124754 2.813996 -0.227566

|    |           |           |           |
|----|-----------|-----------|-----------|
| H  | 2.734365  | 1.391479  | -2.196334 |
| H  | 2.474682  | 4.882250  | 0.311957  |
| H  | 4.828234  | 4.578048  | 0.983092  |
| H  | 5.109148  | 1.095325  | -1.543208 |
| H  | 6.169963  | 2.686184  | 0.066596  |
| C  | -4.103205 | 2.649651  | -0.608523 |
| C  | -2.770341 | 1.831158  | -2.435787 |
| C  | -5.238645 | 2.069384  | -1.177300 |
| C  | -3.898290 | 1.247563  | -3.004306 |
| C  | -5.139769 | 1.366716  | -2.377340 |
| H  | -6.027393 | 0.909425  | -2.822480 |
| H  | -4.187446 | 3.210347  | 0.327466  |
| H  | -6.206741 | 2.170272  | -0.678866 |
| H  | -3.807902 | 0.689697  | -3.940574 |
| H  | -1.798785 | 1.736226  | -2.935384 |
| Cu | 0.062237  | -0.300387 | -0.670566 |
| B  | -1.649199 | -1.451829 | -0.543887 |
| O  | -1.526859 | -2.637206 | 0.154521  |
| O  | -2.969992 | -1.151778 | -0.789907 |
| H  | 2.590609  | -2.704548 | 3.810271  |
| H  | 1.460202  | -3.096717 | 2.506443  |
| C  | -2.264486 | 0.398037  | 2.440863  |
| C  | -0.923113 | 0.929779  | 4.364039  |
| C  | -3.159936 | 1.410714  | 2.776588  |
| C  | -1.812013 | 1.953799  | 4.695783  |
| C  | -2.935397 | 2.199507  | 3.906160  |
| C  | 3.613775  | -1.624690 | 1.358773  |
| H  | 2.871535  | -0.409609 | 2.916198  |
| C  | 3.641610  | -2.882658 | 0.740608  |
| C  | 4.708102  | -0.768372 | 1.162261  |
| C  | 5.798387  | -1.156859 | 0.384898  |
| C  | 4.731565  | -3.275469 | -0.033322 |
| C  | 5.817040  | -2.417291 | -0.212554 |
| H  | 2.795783  | -3.564152 | 0.858745  |
| H  | 4.707164  | 0.214770  | 1.644495  |
| H  | 6.642113  | -0.473390 | 0.253501  |



|   |           |           |           |
|---|-----------|-----------|-----------|
| H | 4.728679  | -4.260675 | -0.507364 |
| H | 6.672392  | -2.728741 | -0.817549 |
| H | -1.625685 | 2.560687  | 5.585773  |
| H | -3.635168 | 2.994979  | 4.174286  |
| H | -2.429473 | -0.189533 | 1.533706  |
| H | -4.040044 | 1.580020  | 2.150233  |
| H | -0.053951 | 0.756960  | 5.003748  |
| C | -2.834155 | -3.206552 | 0.421009  |
| C | -3.836061 | -2.200074 | -0.287866 |
| C | -2.847283 | -4.610698 | -0.155900 |
| C | -2.998955 | -3.285764 | 1.929283  |
| C | -4.855538 | -1.568734 | 0.645184  |
| C | -4.582404 | -2.790846 | -1.473826 |
| H | -2.935538 | -2.301239 | 2.411511  |
| H | -3.963199 | -3.734638 | 2.207031  |
| H | -2.204993 | -3.916998 | 2.354325  |
| H | -4.397248 | -1.048914 | 1.494582  |
| H | -5.456318 | -0.833537 | 0.091482  |
| H | -5.545256 | -2.324836 | 1.046909  |
| H | -2.629099 | -4.622107 | -1.230657 |
| H | -2.081793 | -5.221956 | 0.342291  |
| H | -3.818564 | -5.100944 | 0.001796  |
| H | -3.915687 | -3.214944 | -2.234073 |
| H | -5.274887 | -3.583133 | -1.156202 |
| H | -5.178111 | -2.005554 | -1.959906 |
| C | -0.944259 | -2.655914 | -2.769634 |
| H | -1.268523 | -0.526010 | -2.821481 |
| C | -0.562578 | -1.242659 | -2.379600 |
| H | 1.001115  | 0.085956  | -3.136250 |
| C | 0.829111  | -0.797919 | -2.509302 |
| H | 3.466755  | -0.489100 | -3.345194 |
| H | 3.532920  | -0.816899 | -1.589251 |
| C | 3.324178  | -1.256790 | -2.573312 |
| C | 1.920991  | -1.787360 | -2.617923 |
| F | 1.820328  | -2.537291 | -3.819681 |
| H | 4.035292  | -2.071717 | -2.760430 |

F 1.796677 -2.775131 -1.646479

H -0.758039 -2.785432 -3.847307

H -0.368701 -3.425557 -2.244796

H -2.010790 -2.853418 -2.602177

## 12. References

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Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. *Gaussian 09, Revision D.01*, Gaussian, Inc., Wallingford CT, **2009**.

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19. Lichtenberger, D. L. & Gladysz, J. A. *Organometallics* **2014**, *33*, 835. The “coordinates.xyz” file can be generated by copying all the coordinates in Section 11.3 into a text file without empty lines and changing the extension to “.xyz”.

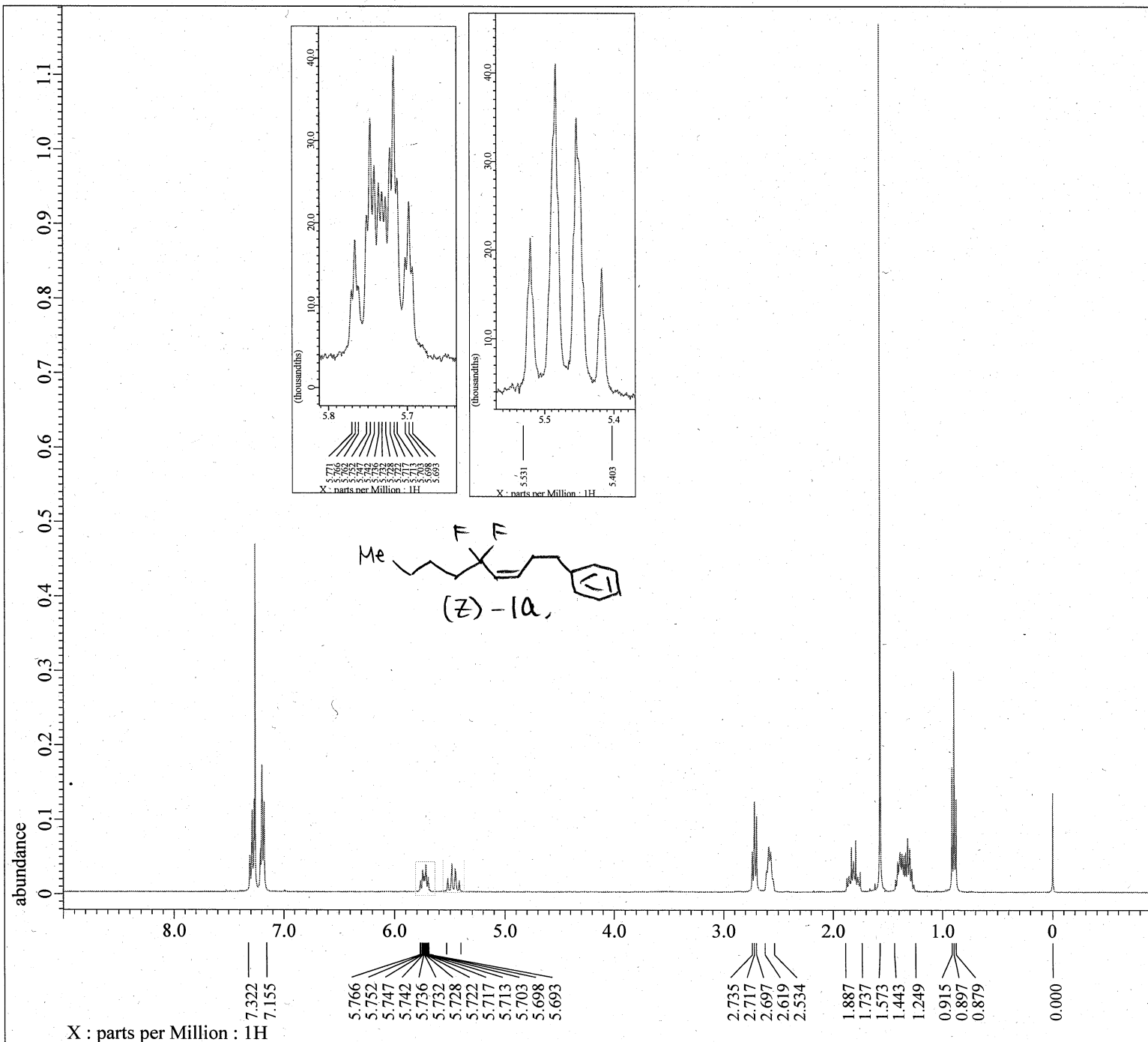


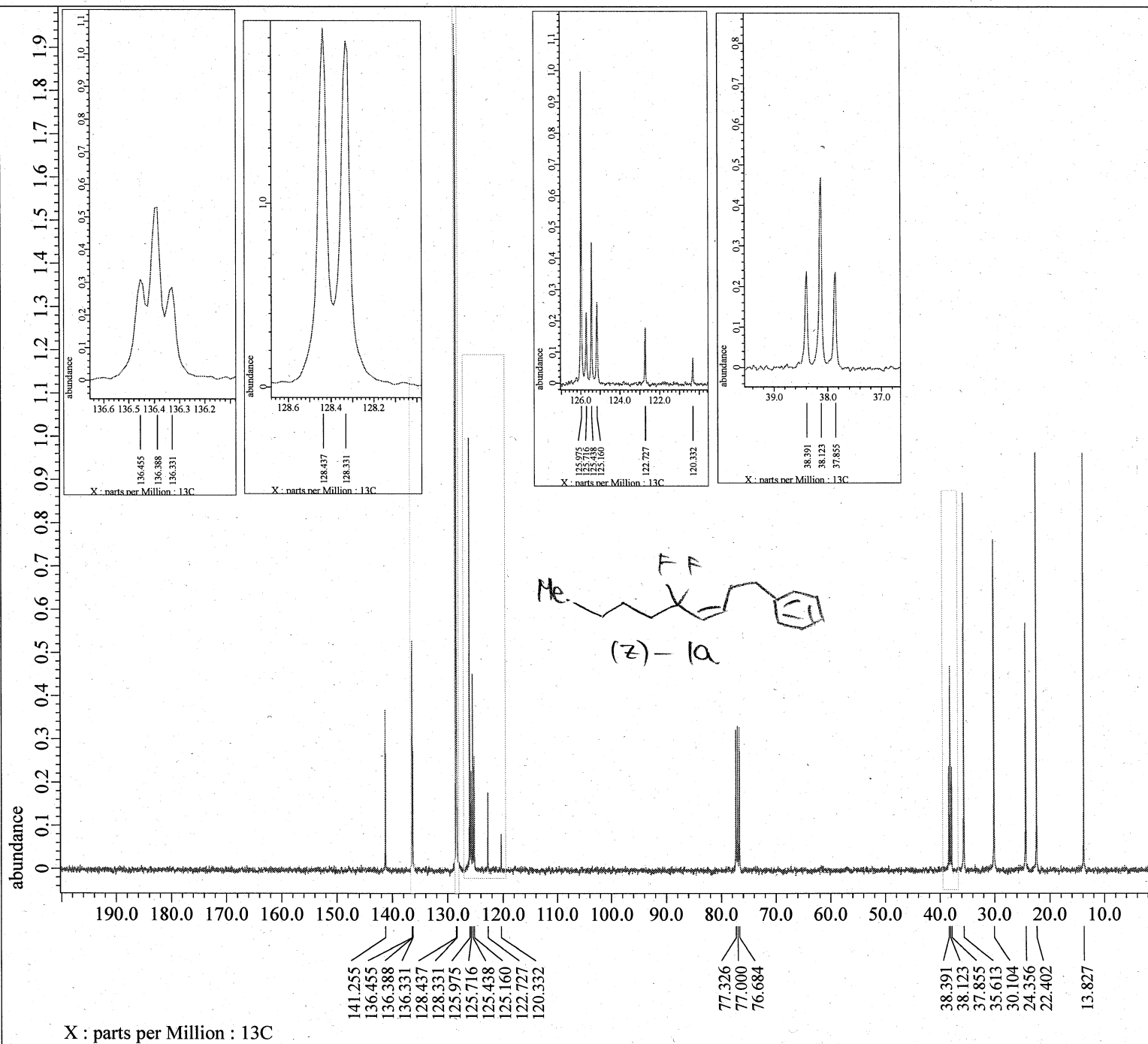
Filename = AKY610-data-again-10.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#384934  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 10-JUL-2018 19:16:30  
Revision\_Time = 29-MAR-2019 18:28:28

Comment = single\_pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

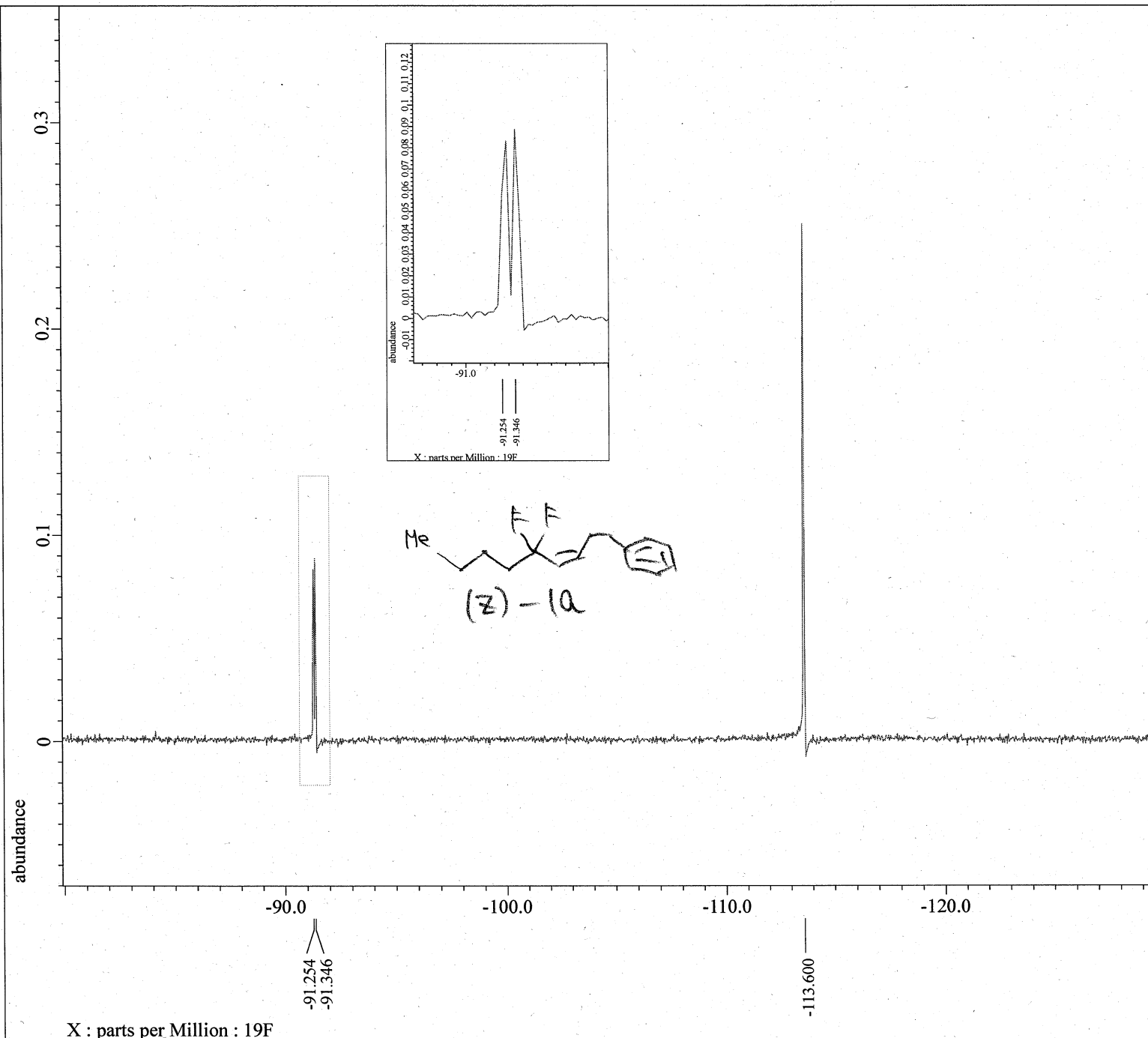
Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq\_Duration = 2.20725248 [s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Tri\_Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr Gain = 38  
Temp Get = 20.6 [dC]  
X 90\_Width = 13.2 [us]  
X Acq Time = 2.20725248 [s]  
X Angle = 45 [deg]  
X Atn = 3.5 [dB]  
X Pulse = 6.6 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1 [s]  
Repetition\_Time = 7.20725248 [s]





Filename = AKY610-carbon-data-4.jdf  
 Author = element  
 Experiment = single\_pulse\_dec  
 Sample Id = 2  
 Solvent = CHLOROFORM-D  
 Creation Time = 10-JUL-2018 09:17:30  
 Revision Time = 10-JUL-2018 15:06:02  
 Current Time = 10-JUL-2018 15:07:53  
  
 Comment = single pulse decoupled gat  
 Data Format = 1D COMPLEX  
 Dim Size = 26214  
 Dim Title = 13C  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECX 400P  
 Spectrometer = DELTA2\_NMR  
  
 Field Strength = 9.2982153[T] (400[MHz])  
 X Acq Duration = 1.048576[s]  
 X Domain = 13C  
 X Freq = 99.54517646[MHz]  
 X Offset = 100[ppm]  
 X Points = 32768  
 X Prescans = 4  
 X Resolution = 0.95367432[Hz]  
 X Sweep = 31.25[kHz]  
 Irr Domain = 1H  
 Irr Freq = 395.88430144[MHz]  
 Irr Offset = 5[ppm]  
 Clipped = FALSE  
 Scans = 128  
 Total Scans = 128  
  
 Relaxation Delay = 2[s]  
 Recvr Gain = 54  
 Temp Get = 20.6[dC]  
 X 90 Width = 10.1[us]  
 X Acq Time = 1.048576[s]  
 X Angle = 30[deg]  
 X Atn = 3.4[dB]  
 X Pulse = 3.36666667[us]  
 Irr Atn Dec = 22.3[dB]  
 Irr Atn Noe = 22.3[dB]  
 Irr Noise = WALTZ  
 Decoupling = TRUE  
 Initial Wait = 1[s]  
 Noe = TRUE  
 Noe Time = 2[s]  
 Repetition Time = 3.048576[s]

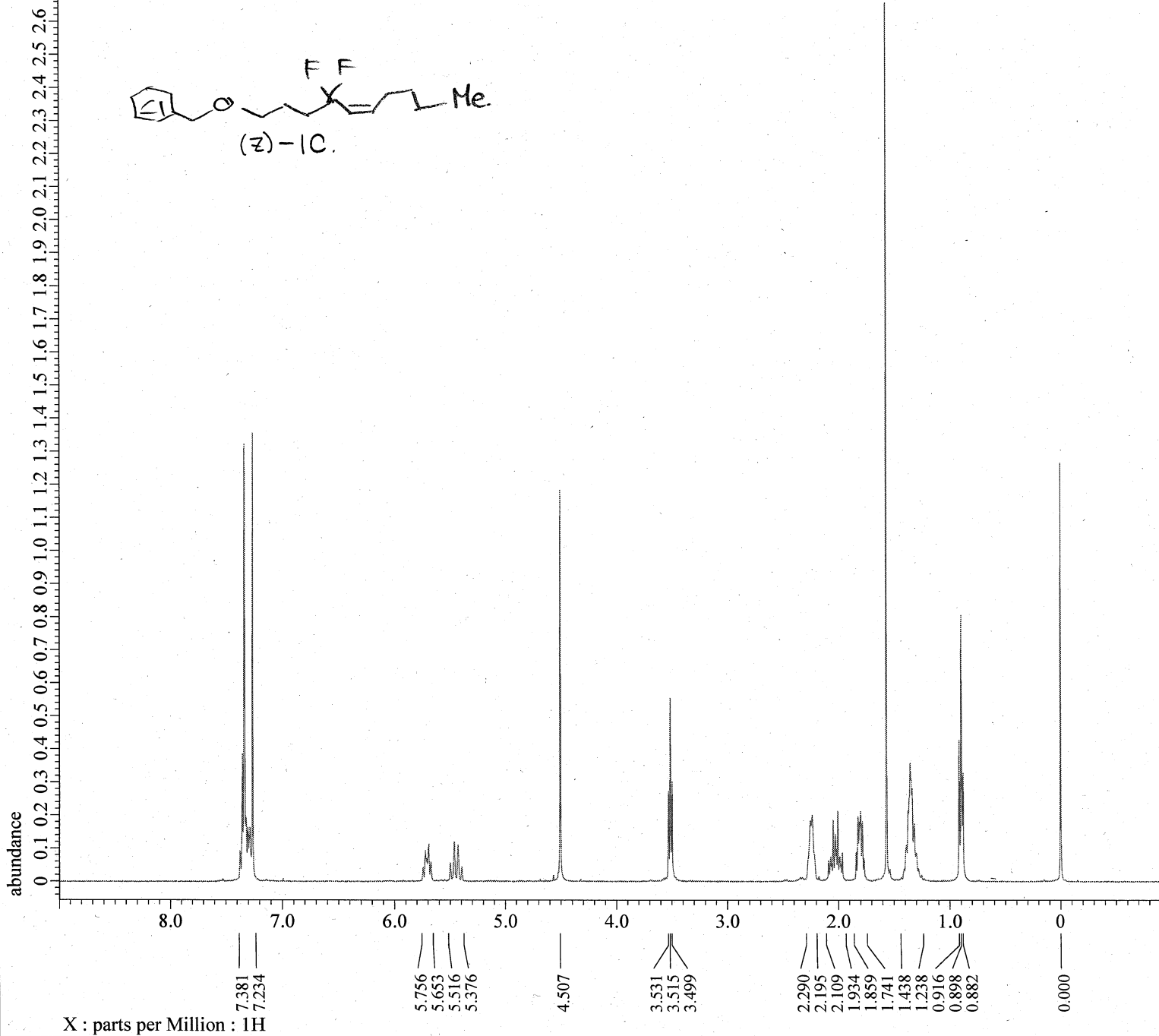
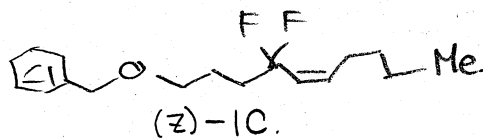


Filename = AKY610-fnmr-data-9.jdf  
 Author = element  
 Experiment = single pulse.ex2  
 Sample Id = S#380692  
 Solvent = CHLOROFORM-D  
 Creation Time = 10-JUL-2018 10:10:09  
 Revision Time = 10-JUL-2018 15:25:27  
 Current Time = 10-JUL-2018 15:25:31

Comment = single pulse  
 Data Format = 1D COMPLEX  
 Dim Size = 13107  
 Dim Title = 19F  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECX 400P  
 Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
 X Acq Duration = 87.81824[ms]  
 X Domain = 19F  
 X Freq = 372.50336686[MHz]  
 X Offset = 0[ppm]  
 X Points = 16384  
 X Prescans = 1  
 X Resolution = 11.38715602[Hz]  
 X Sweep = 186.56716418[kHz]  
 Irr Domain = 19F  
 Irr Freq = 372.50336686[MHz]  
 Irr Offset = 5[ppm]  
 Tri Domain = 19F  
 Tri Freq = 372.50336686[MHz]  
 Tri Offset = 5[ppm]  
 Clipped = FALSE  
 Scans = 8  
 Total Scans = 8

Relaxation Delay = 5[s]  
 Recvr Gain = 24  
 Temp Get = 20.4[dC]  
 X 90 Width = 13.9[us]  
 X Acq Time = 87.81824[ms]  
 X Angle = 45[deg]  
 X Atn = 4[dB]  
 X Pulse = 6.95[us]  
 Irr Mode = Off  
 Tri Mode = Off  
 Dante Presat = FALSE  
 Initial Wait = 1[s]  
 Repetition Time = 5.08781824[s]



Filename = AKY684-pure-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#773695  
Solvent = CHLOROFORM-D  
Actual Start Time = 10-OCT-2018 05:01:06  
Revision Time = 29-MAR-2019 18:48:42

Comment = single\_pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 48  
Temp Get = 19.6[dC]  
X 90 Width = 11.04[us]  
X Acq Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 7.228224[s]



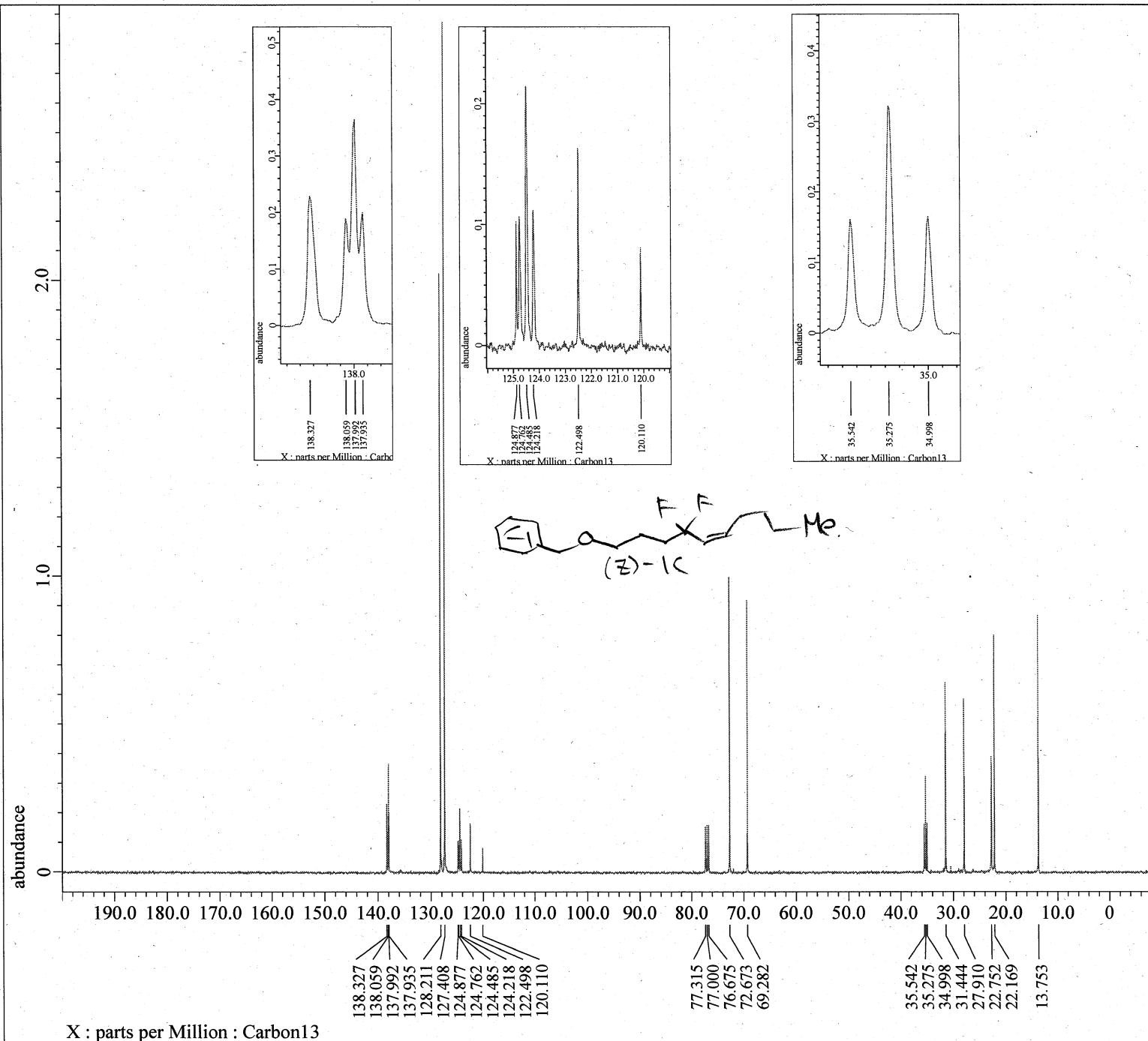


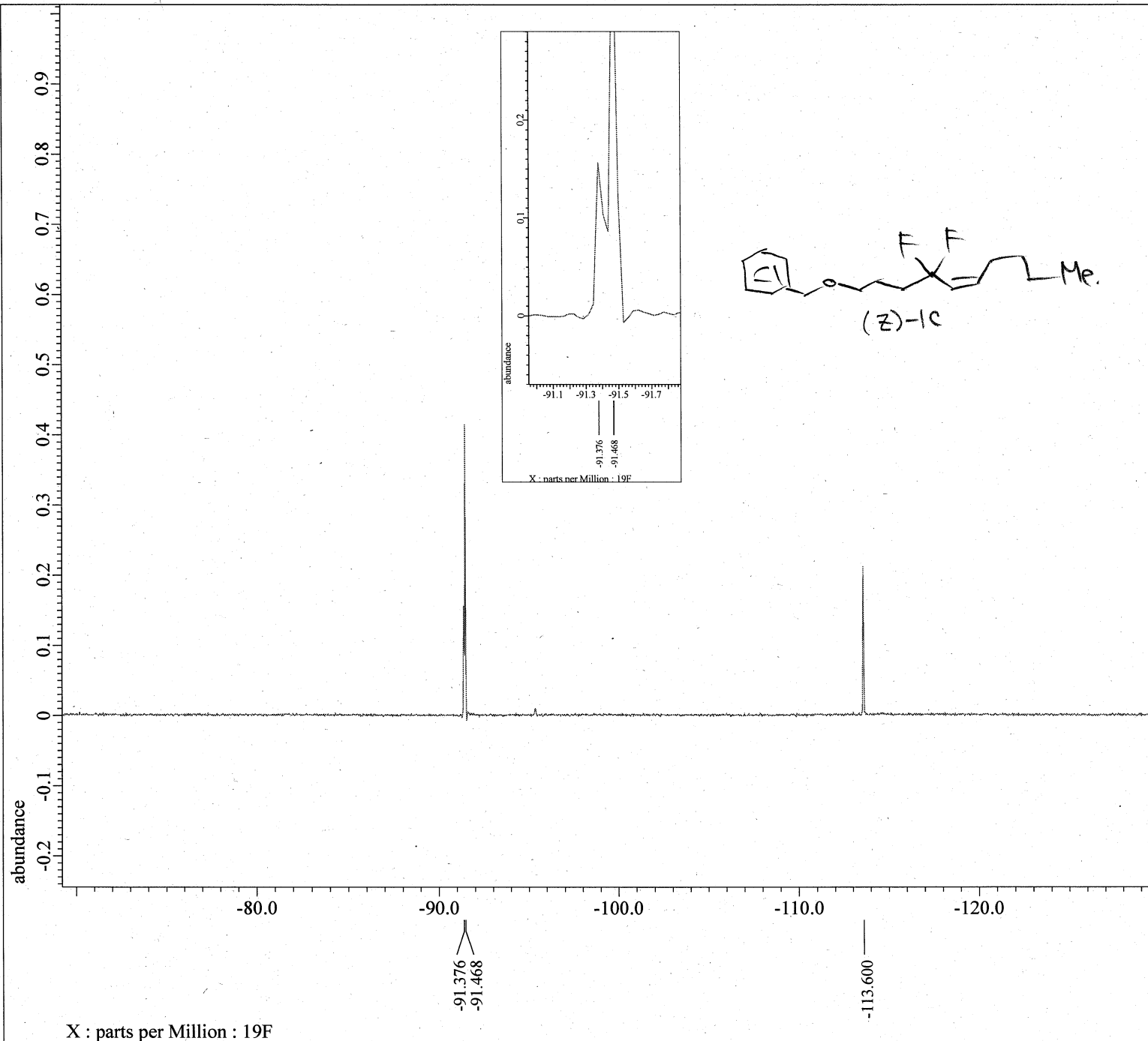
Filename = AKY684-carbon\_Carbon-1-2.  
Author = element  
Experiment = carbon\_jxp  
Sample Id = AKY684-carbon  
Solvent = CHLOROFORM-D  
Actual Start Time = 9-OCT-2018 22:33:41  
Revision Time = 13-NOV-2018 12:54:34

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Carbon  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X Acq Duration = 1.0433312[s]  
X Domain = 13C  
X Freq = 100.33735165[MHz]  
X Offset = 100.0[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95846665[Hz]  
X Sweep = 31.40703518[kHz]  
X Sweep Clipped = 25.12562814[kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754[MHz]  
Irr Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 128  
Total Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 50  
Temp Get = 19.1[dC]  
X 90 Width = 10.9[us]  
X Acq Time = 1.0433312[s]  
X Angle = 30[deg]  
X Atn = 5.4[dB]  
X Pulse = 3.63333333[us]  
Irr Atn Dec = 25.823[dB]  
Irr Atn Noe = 25.823[dB]  
Irr Noise = WALTZ  
Irr Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.0433312[s]





Filename = AKY684-fnmr-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#825610  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 10-OCT-2018 07:25:41  
Revision\_Time = 13-NOV-2018 12:59:12

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 23.3[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
DanTe Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

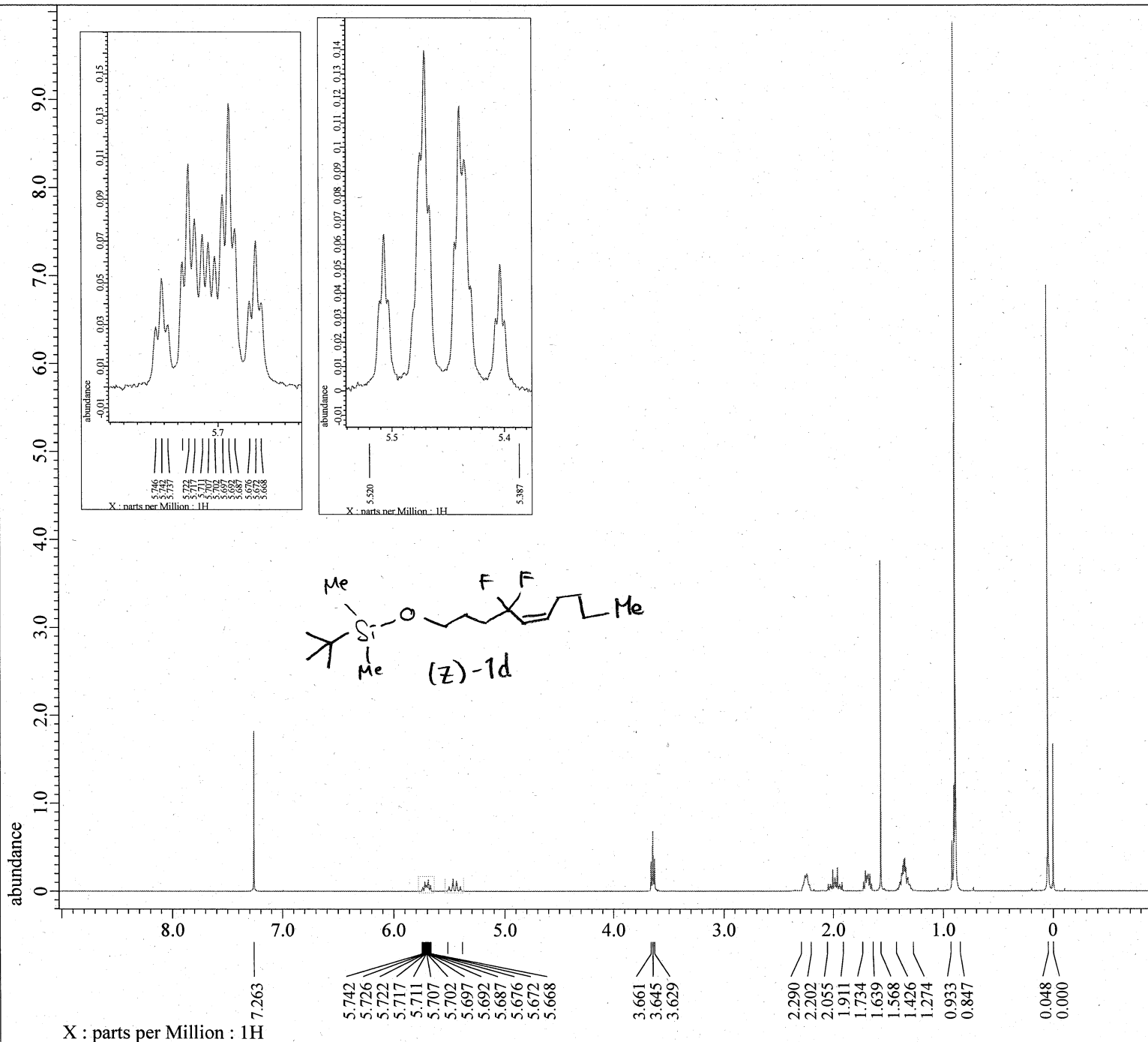


Filename = AKY685-pure-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#777806  
Solvent = CHLOROFORM-D  
Actual Start Time = 10-OCT-2018 05:07:57  
Revision Time = 29-MAR-2019 19:00:07

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 46  
Temp Get = 19.5[dC]  
X 90 Width = 11.04[us]  
X Acq Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.228224[s]



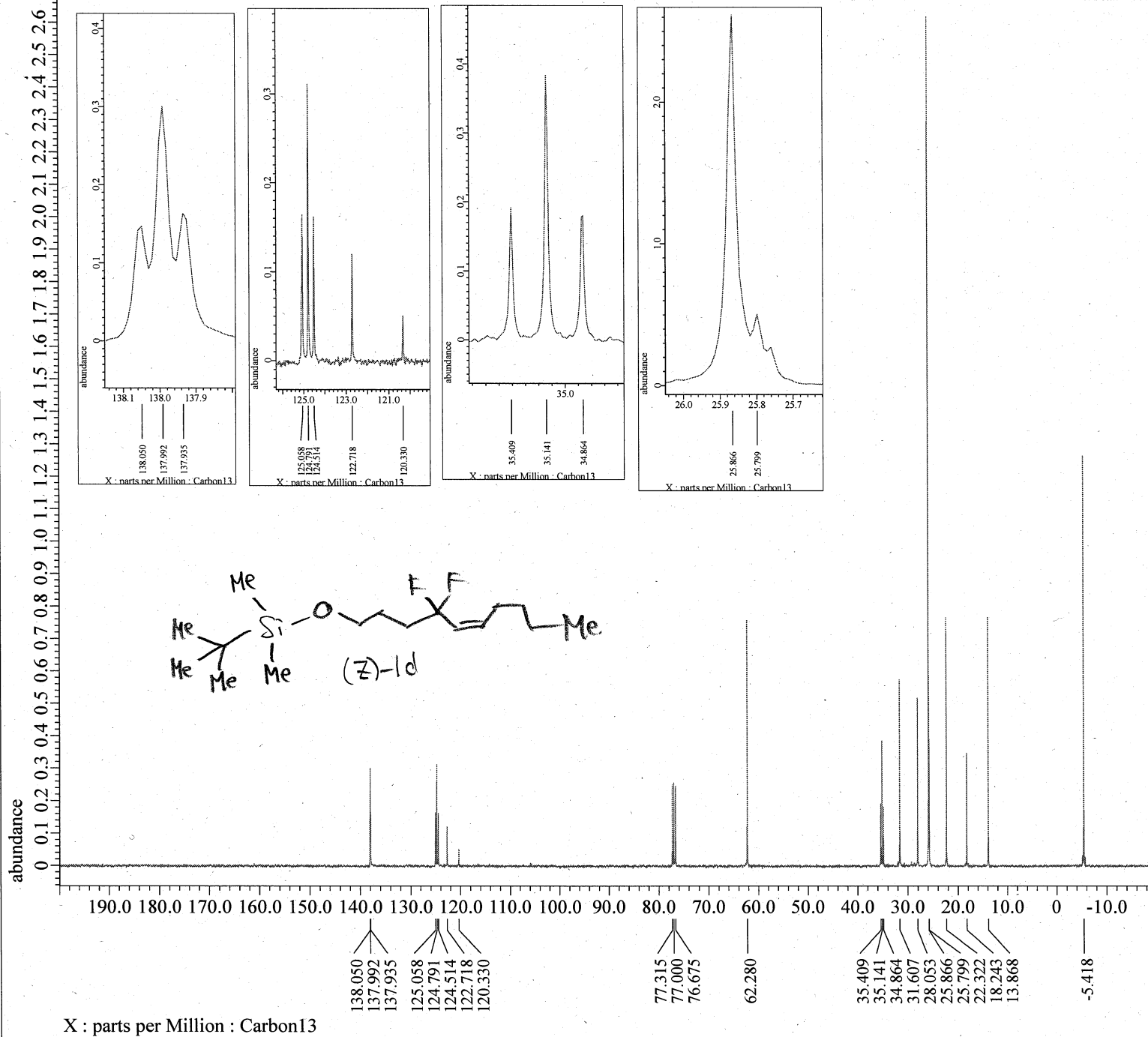


Filename = AKY685-carbon\_Carbon-1-2.  
Author = element  
Experiment = carbon.jxp  
Sample Id = AKY685-carbon  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 9-OCT-2018 22:54:14  
Revision\_Time = 22-MAR-2019 10:42:16

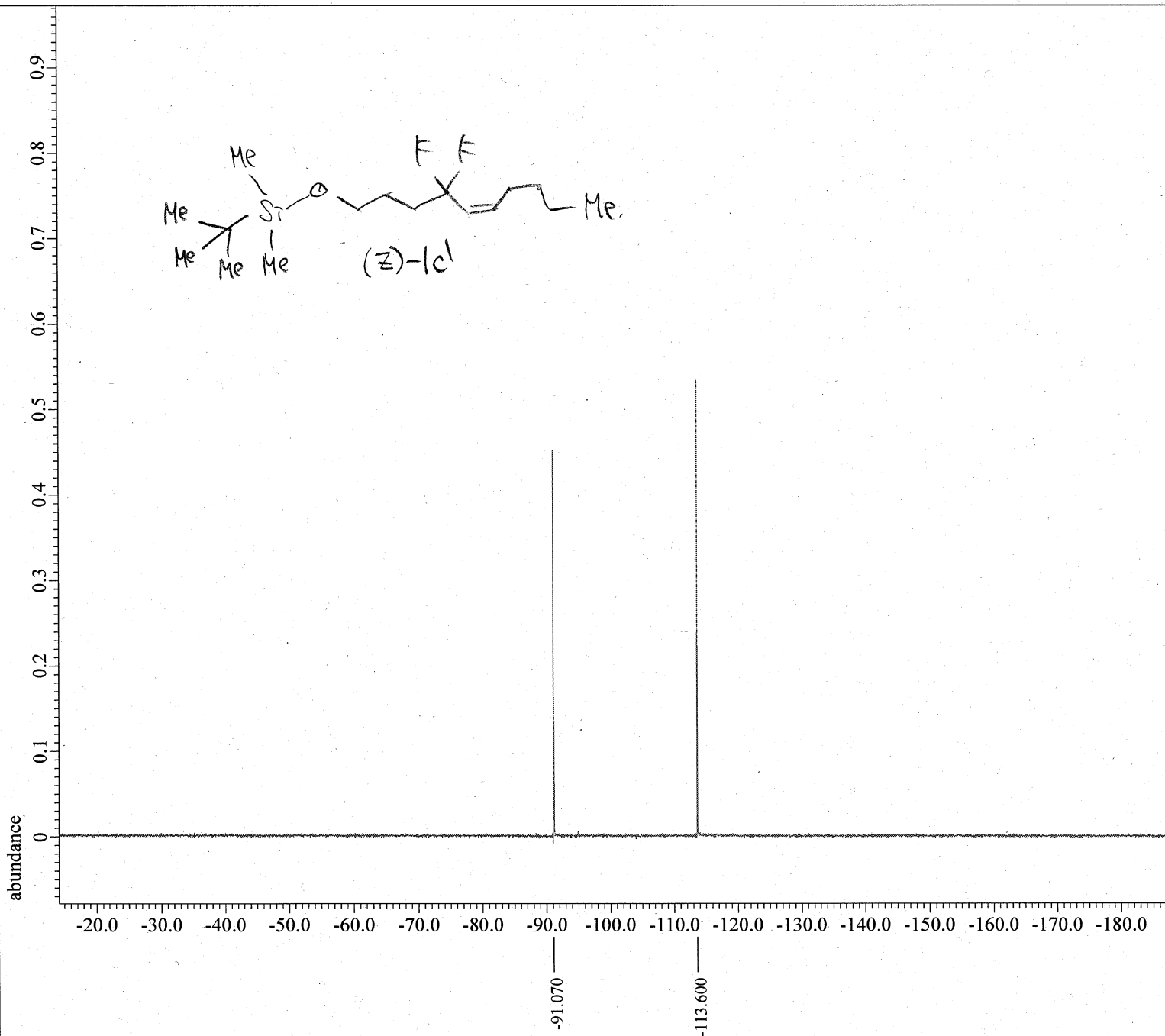
Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Carbon  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X Acq\_Duration = 1.0433312[s]  
X Domain = 13C  
X Freq = 100.33735165[MHz]  
X Offset = 100.0[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95846665[Hz]  
X Sweep = 31.40703518[kHz]  
X Sweep\_Clippped = 25.12562814[kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754[MHz]  
Irr Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2[s]  
Recvr Gain = 50  
Temp Get = 20.5[dc]  
X 90\_Width = 10.9[us]  
X Acq\_Time = 1.04333312[s]  
X Angle = 30[deg]  
X Atn = 5.4[dB]  
X Pulse = 3.63333333[us]  
Irr Atn\_Dec = 25.823[dB]  
Irr Atn\_Noise = 25.823[dB]  
Irr Noise = WALTZ  
Irr Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition\_Time = 3.04333312[s]



X : parts per Million : Carbon13



Filename = AKY685-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#829270  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 10-OCT-2018 07:31:38  
Revision\_Time = 22-MAR-2019 10:54:29

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp\_Get = 23.5[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

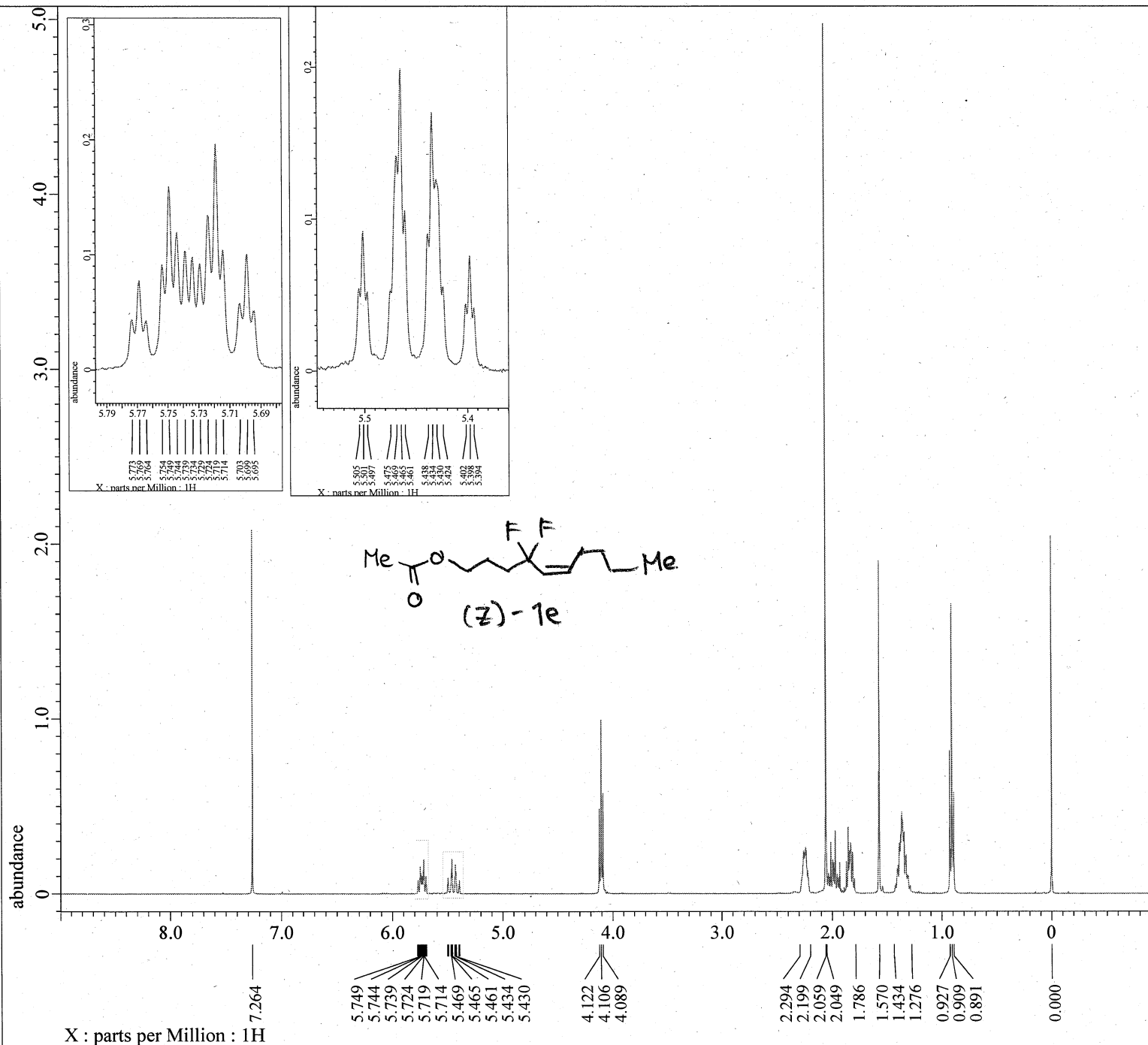


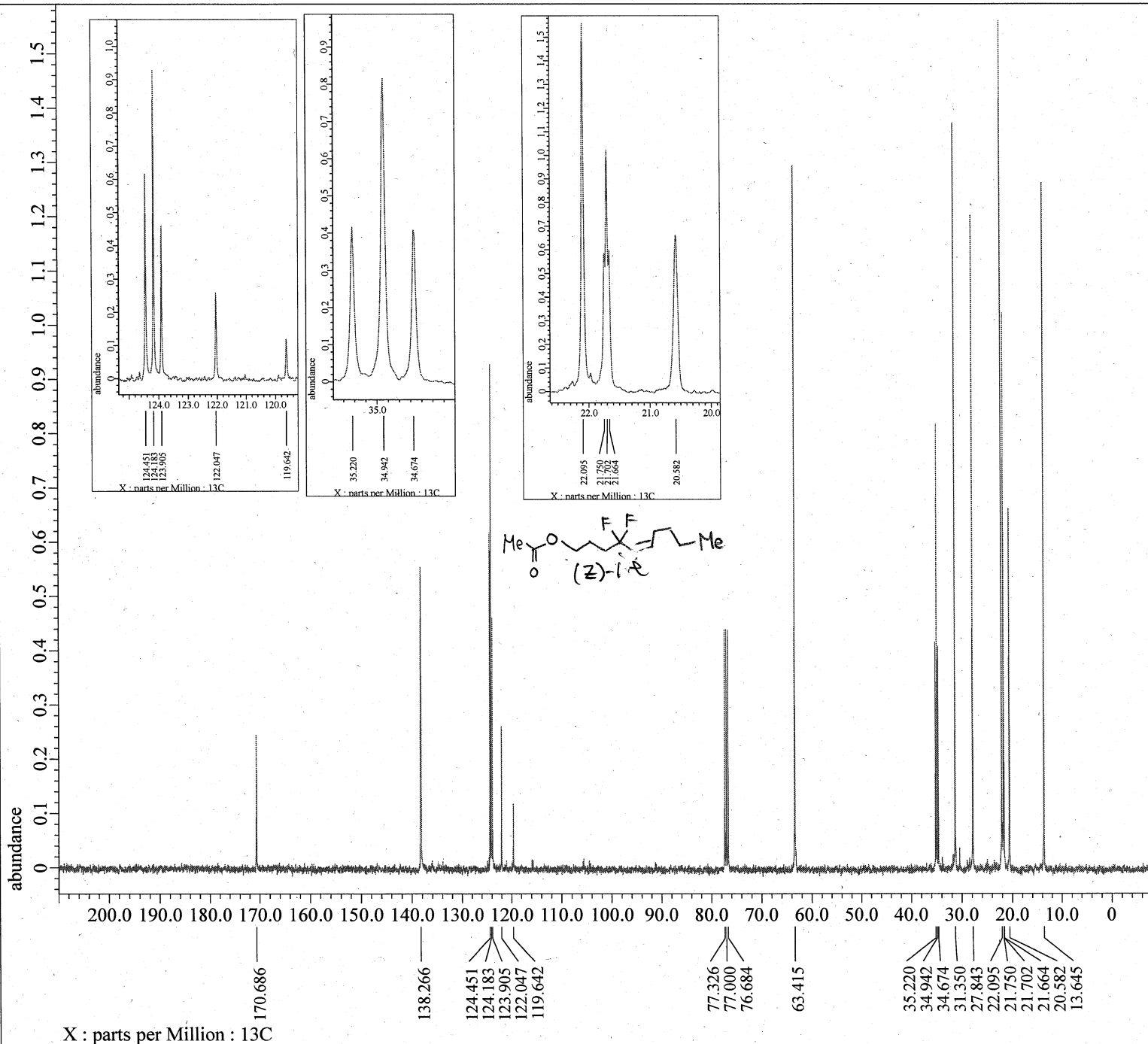
Filename = AKY662-pureag-3.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample\_Id = S#635534  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 13-SEP-2018 01:14:39  
Revision\_Time = 29-MAR-2019 19:10:28

Comment = single pulse  
Data\_Format = 1D COMPLEX  
Dim\_Size = 26214  
X\_Domain = 1H  
Dim\_Title = 1H  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field\_Strength = 9.20197068[T] (390[MHz])  
X\_Acq\_Duration = 2.228224[s]  
X\_Domain = 1H  
X\_Freq = 391.78655441[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.44878791[Hz]  
X\_Sweep = 7.35294118[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 1H  
Tri\_Freq = 391.78655441[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 46  
Temp\_Get = 20.4[dC]  
X\_90\_Width = 11.04[us]  
X\_Acq\_Time = 2.228224[s]  
X\_Angle = 45[deg]  
X\_Atn = 1.9[dB]  
X\_Pulse = 5.52[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.228224[s]



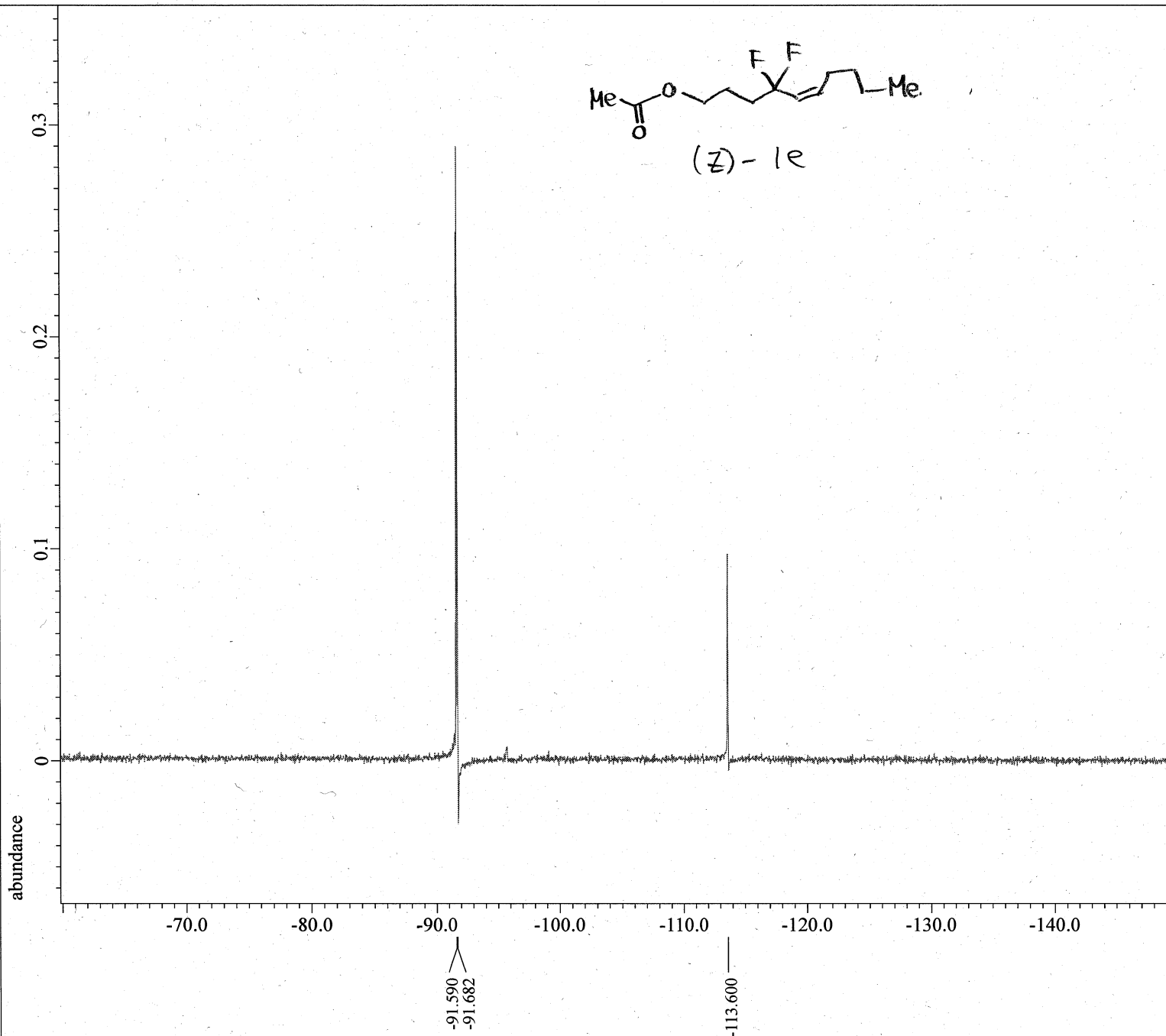
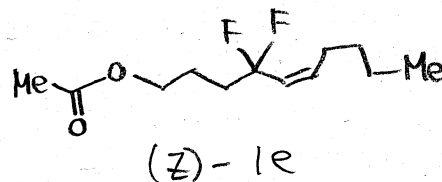


Filename = AKY662-carbon-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#471018  
Solvent = CHLOROFORM-D  
Actual Start Time = 25-SEP-2018 21:33:54  
Revision Time = 20-MAR-2019 19:43:19

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 21.3[dc]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]



X : parts per Million : 19F

```

Filename      = AKY662-pure-fnmrag-13.jdf
Author       = element
Experiment   = single_pulse.ex2
Sample Id    = S#705644
Solvent      = CHLOROFORM-D
Creation Time = 12-SEP-2018 19:06:17
Revision Time = 5-OCT-2018 13:26:14
Current Time  = 5-OCT-2018 13:26:23

Comment      = single_pulse
Data Format   = 1D_COMPLEX
Dim Size     = 13107
Dim Title    = 19F
Dim Units    = [ppm]
Dimensions   = X
Site         = ECX 400P
Spectrometer = DELTA2_NMR

Field Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 87.81824[ms]
X_Domain       = 19F
X_Freq        = 372.50336686[MHz]
X_Offset      = 0[ppm]
X_Points      = 16384
X_Prescans    = 1
X_Resolution  = 11.38715602[Hz]
X_Sweep       = 186.56716418[kHz]
Irr_Domain    = 19F
Irr_Freq      = 372.50336686[MHz]
Irr_Offset    = 5[ppm]
Tri_Domain    = 19F
Tri_Freq      = 372.50336686[MHz]
Tri_Offset    = 5[ppm]
Clipped       = FALSE
Scans         = 8
Total Scans   = 8

Relaxation Delay = 5[s]
Recvr Gain       = 24
Temp_Get        = 21.2[dc]
X_90_Width      = 13.9[us]
X_Acq_Time      = 87.81824[ms]
X_Angle         = 45[deg]
X_Atn           = 4[dB]
X_Pulse         = 6.95[us]
Irr_Mode        = Off
Tri_Mode        = Off
DanTe Presat    = FALSE
Initial Wait    = 1[s]
Repetition Time = 5.08781824[s]
    
```



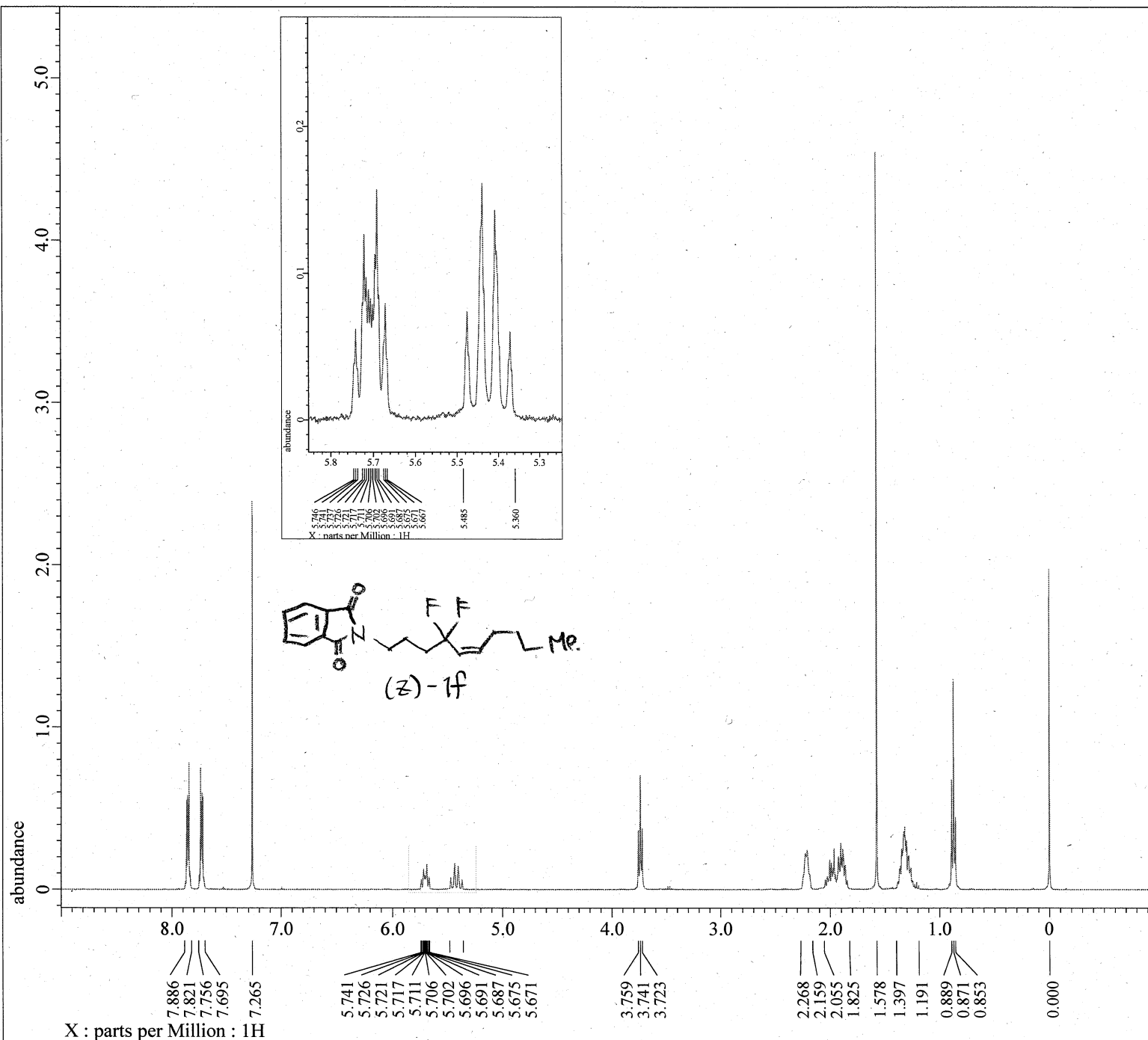


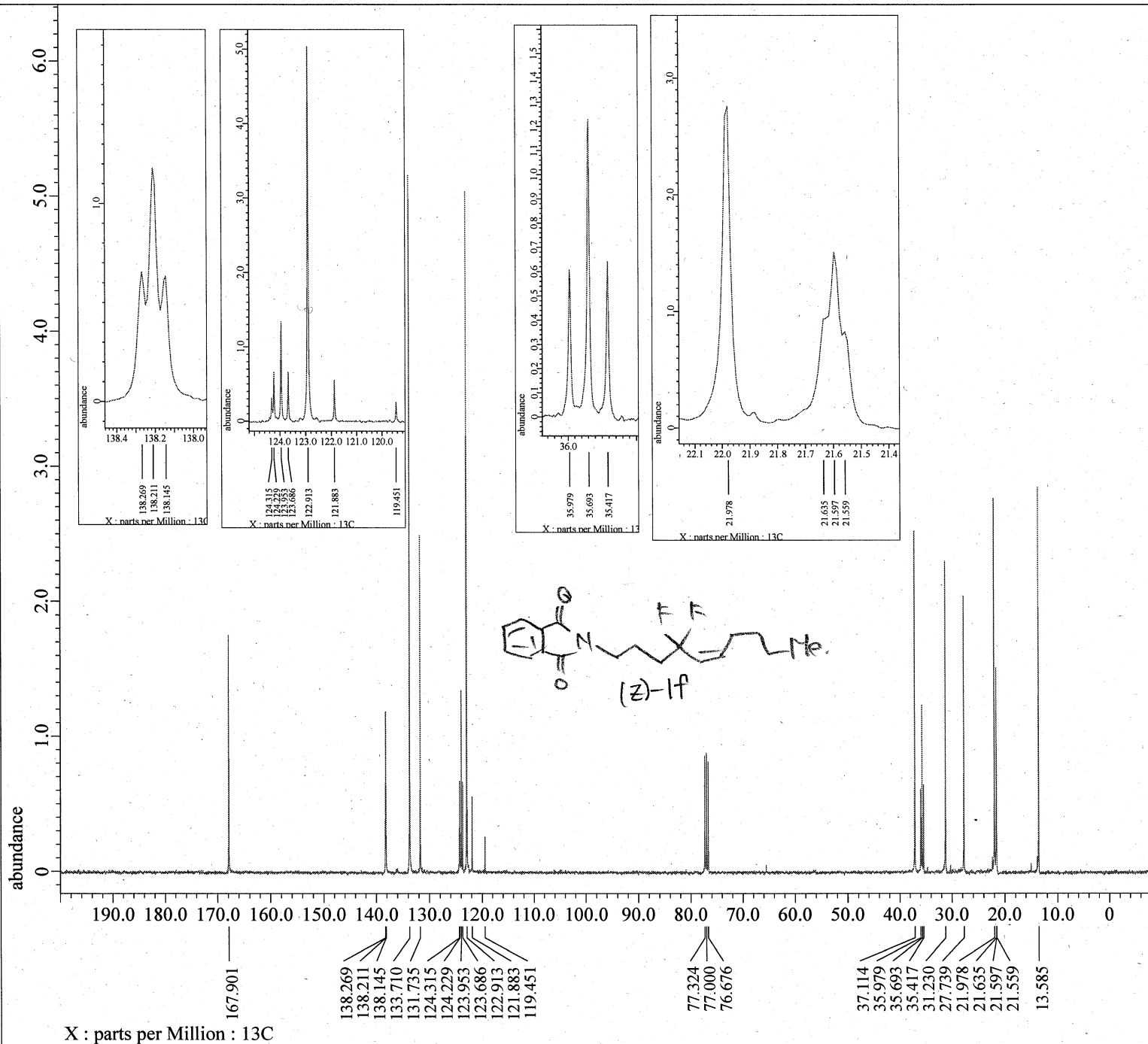
Filename = AKY694-pure-ag-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#510561  
Solvent = CHLOROFORM-D  
Actual Start Time = 18-OCT-2018 21:41:06  
Revision Time = 29-MAR-2019 19:07:17

Comment = single\_pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

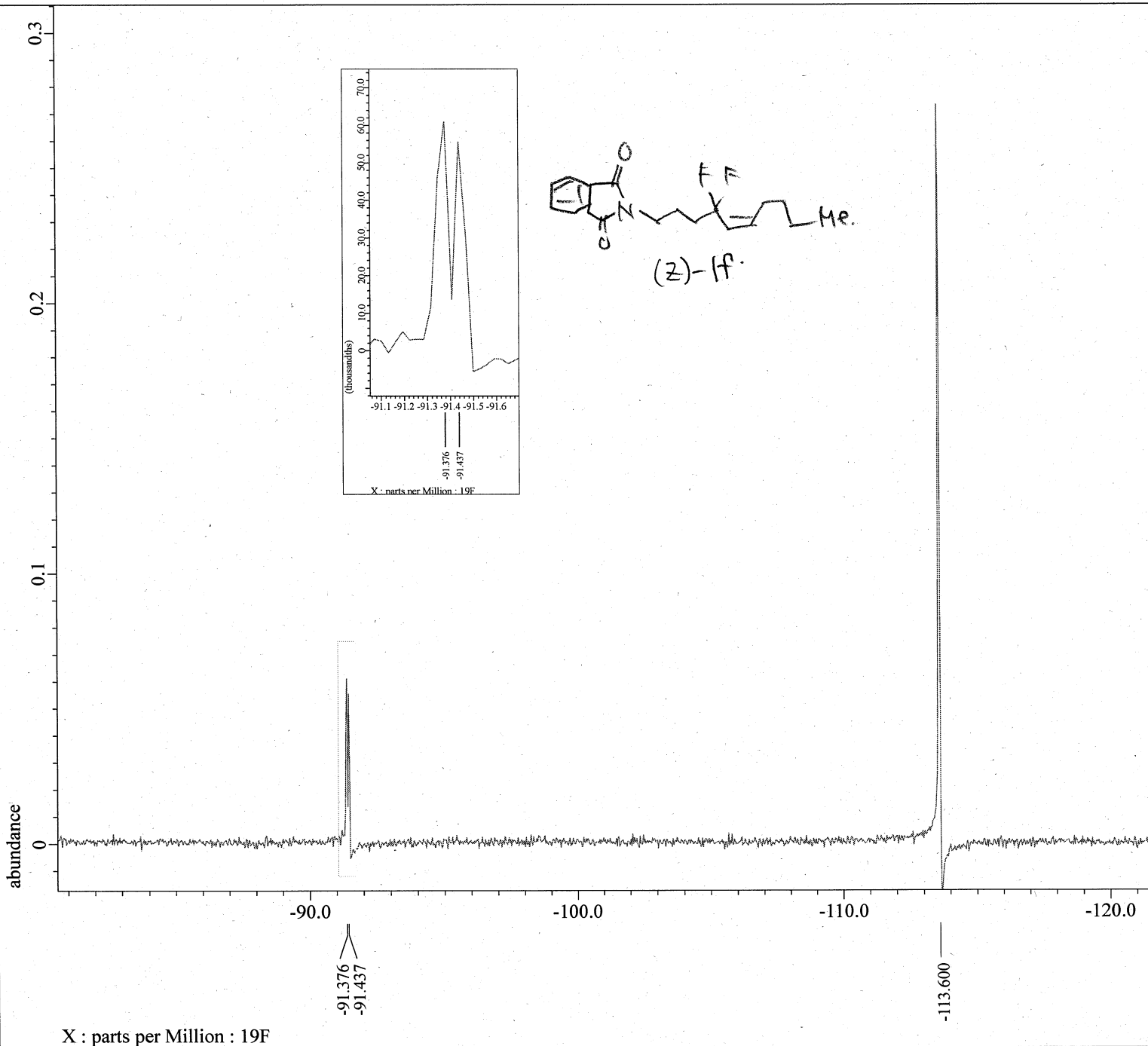
Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 50  
Temp Get = 18.9[dC]  
X 90 Width = 11.04[us]  
X Acq Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.228224[s]





Filename = AKY694-carbon-4.jdf  
 Author = element  
 Experiment = single\_pulse\_dec  
 Sample Id = S#514858  
 Solvent = CHLOROFORM-D  
 Creation Time = 18-OCT-2018 12:54:06  
 Revision Time = 18-OCT-2018 15:38:19  
 Current Time = 18-OCT-2018 15:38:29  
  
 Comment = single pulse decoupled gat  
 Data Format = 1D COMPLEX  
 Dim Size = 26214  
 Dim Title = 13C  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECS 400  
 Spectrometer = JNM-ECS400  
  
 Field Strength = 9.20197068 [T] (390 [MHz])  
 X Acq Duration = 1.06430464 [s]  
 X Domain = 13C  
 X Freq = 98.51479726 [MHz]  
 X Offset = 100 [ppm]  
 X Points = 32768  
 X Prescans = 4  
 X Resolution = 0.93958061 [Hz]  
 X Sweep = 30.78817734 [kHz]  
 ITr Domain = 1H  
 Irr Freq = 391.78655441 [MHz]  
 Irr Offset = 5 [ppm]  
 Clipped = FALSE  
 Scans = 128  
 Total Scans = 128  
  
 Relaxation Delay = 2 [s]  
 Recvr Gain = 60  
 Temp Get = 19.1 [dC]  
 X 90 Width = 9.11 [us]  
 X Acq Time = 1.06430464 [s]  
 X Angle = 30 [deg]  
 X Atn = 4.9 [dB]  
 X Pulse = 3.03666667 [us]  
 ITr Atn Dec = 22.255 [dB]  
 Irr Atn Noe = 22.255 [dB]  
 Irr Noise = WALTZ  
 Decoupling = TRUE  
 Initial Wait = 1 [s]  
 Noe = TRUE  
 Noe Time = 2 [s]  
 Repetition Time = 3.06430464 [s]



Filename = AKY694-fnmr-10.jdf  
 Author = element  
 Experiment = single\_pulse.ex2  
 Sample Id = S#526358  
 Solvent = CHLOROFORM-D  
 Creation Time = 18-OCT-2018 14:07:39  
 Revision Time = 18-OCT-2018 15:29:59  
 Current Time = 18-OCT-2018 15:30:04  
 Comment = single\_pulse  
 Data Format = 1D\_COMPLEX  
 Dim Size = 13107  
 Dim Title = 19F  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECX 400P  
 Spectrometer = DELTA2 NMR  
 Field Strength = 9.2982153[T] (400[MHz])  
 X Acq\_Duration = 87.81824[ms]  
 X Domain = 19F  
 X Freq = 372.50336686[MHz]  
 X\_Offset = 0[ppm]  
 X Points = 16384  
 X\_Prescans = 1  
 X Resolution = 11.38715602[Hz]  
 X\_Sweep = 186.56716418[kHz]  
 Irr\_Domain = 19F  
 Irr\_Freq = 372.50336686[MHz]  
 Irr\_Offset = 5[ppm]  
 Tri\_Domain = 19F  
 Tri\_Freq = 372.50336686[MHz]  
 Tri\_Offset = 5[ppm]  
 Clipped = FALSE  
 Scans = 8  
 Total\_Scans = 8  
 Relaxation\_Delay = 5[s]  
 Recvr\_Gain = 24  
 Temp\_Get = 19.7[dc]  
 X\_90\_Width = 13.9[us]  
 X\_Acq\_Time = 87.81824[ms]  
 X\_Angle = 45[deg]  
 X\_Atn = 4[dB]  
 X\_Pulse = 6.95[us]  
 Irr\_Mode = Off  
 Tri\_Mode = Off  
 Dante\_Presat = FALSE  
 Initial\_Wait = 1[s]  
 Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

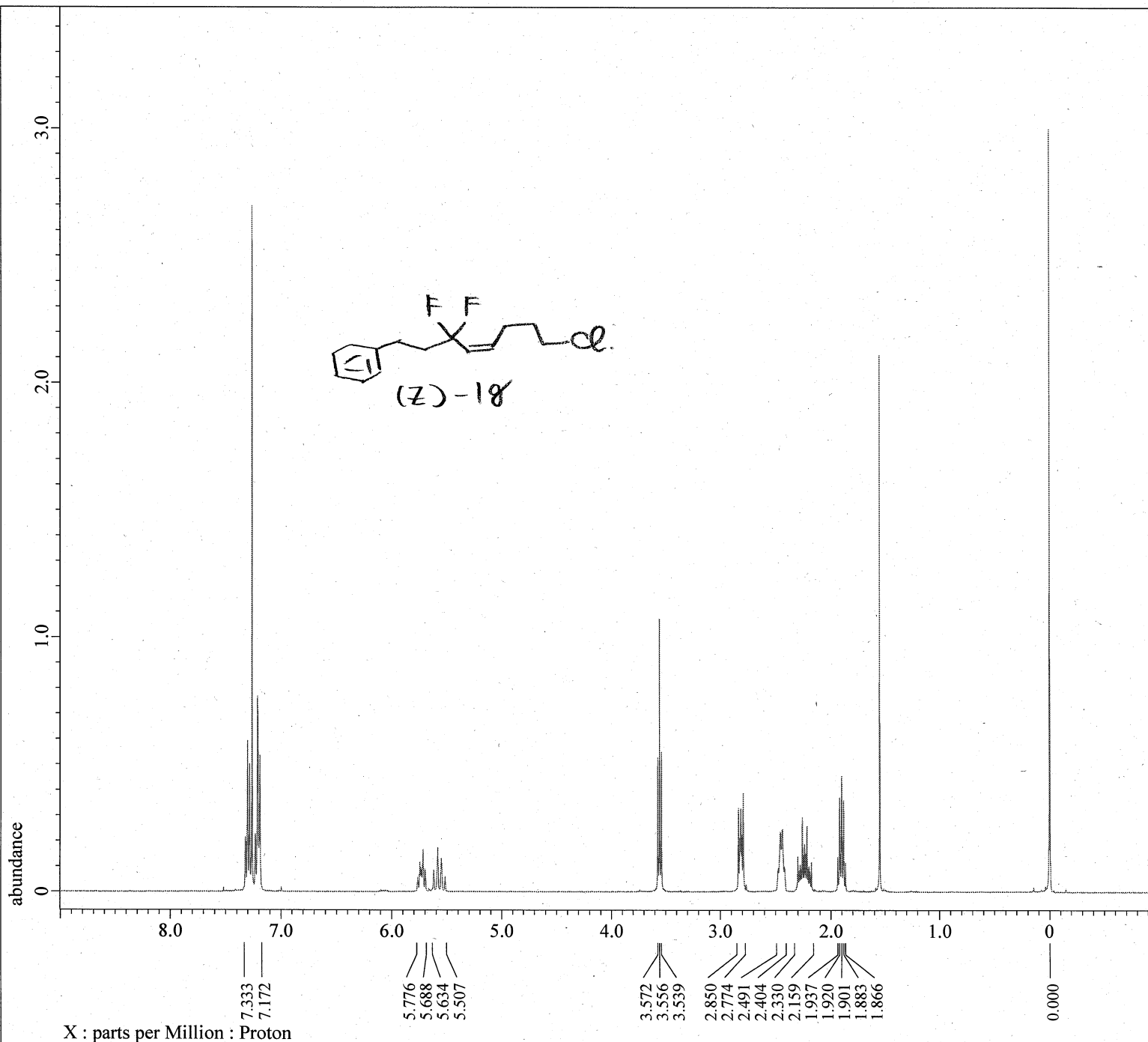


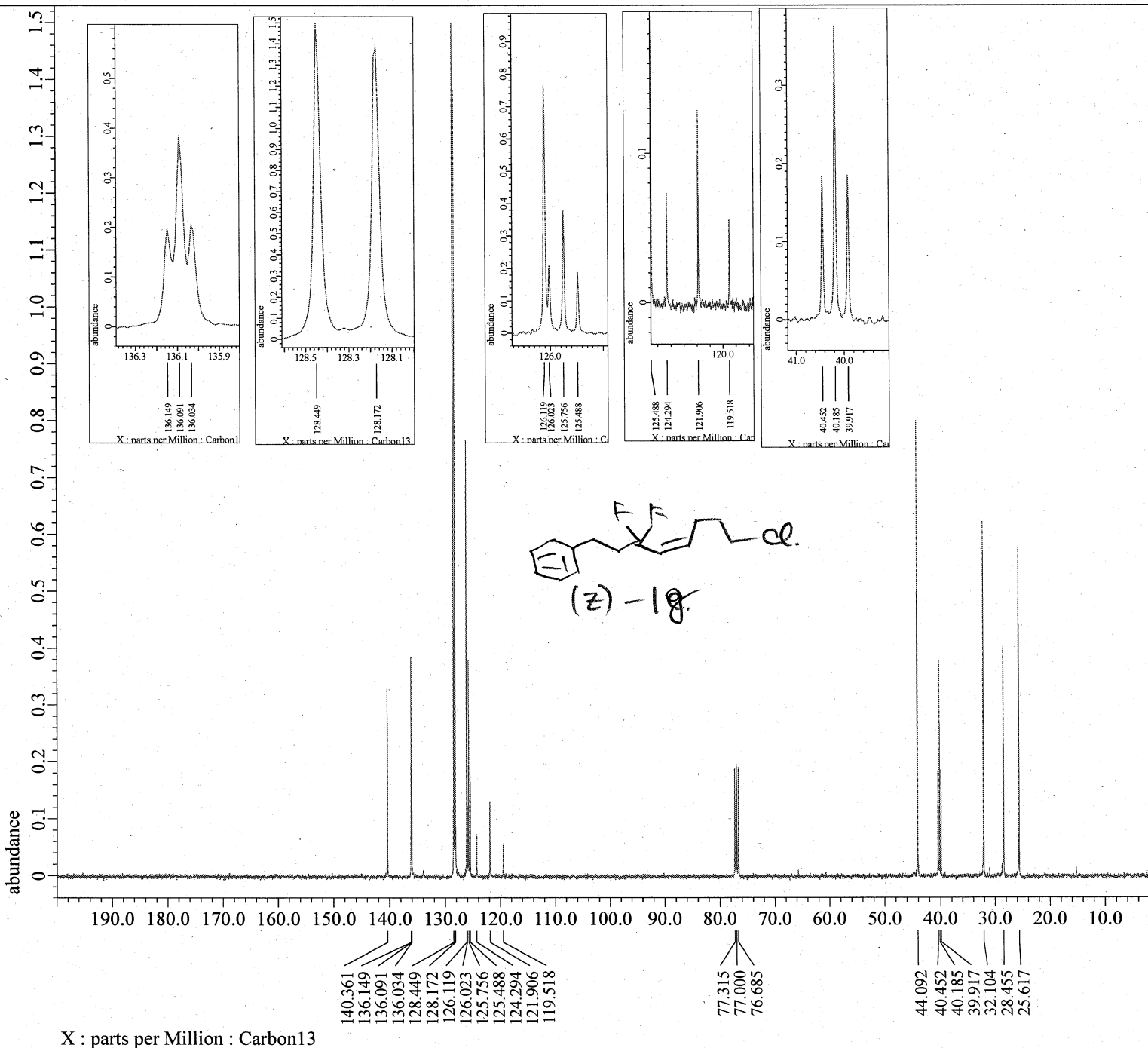
Filename = AKY626-gpc Proton-1-4 .jdf  
Author = element  
Experiment = proton .jxp  
Sample Id = AKY626-gpc  
Solvent = CHLOROFORM-D  
Actual Start Time = 21-NOV-2018 09:55:46  
Revision Time = 29-MAR-2019 19:15:22

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814 [T] (400 [MHz])  
X Acq Duration = 2.18103808 [s]  
X Domain = 1H  
X Freq = 400.53219825 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45849727 [Hz]  
X Sweep = 7.51201923 [kHz]  
X Sweep Clipped = 6.00961538 [kHz]  
Irr Domain = Proton  
Irr Freq = 400.53219825 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = Proton  
Tri Freq = 400.53219825 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5 [s]  
Recvr Gain = 48  
Temp Get = 19.4 [dC]  
X 90 Width = 6.22 [us]  
X Acq Time = 2.18103808 [s]  
X Angle = 45 [deg]  
X Atn = 0.8 [dB]  
X Pulse = 3.11 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.18103808 [s]



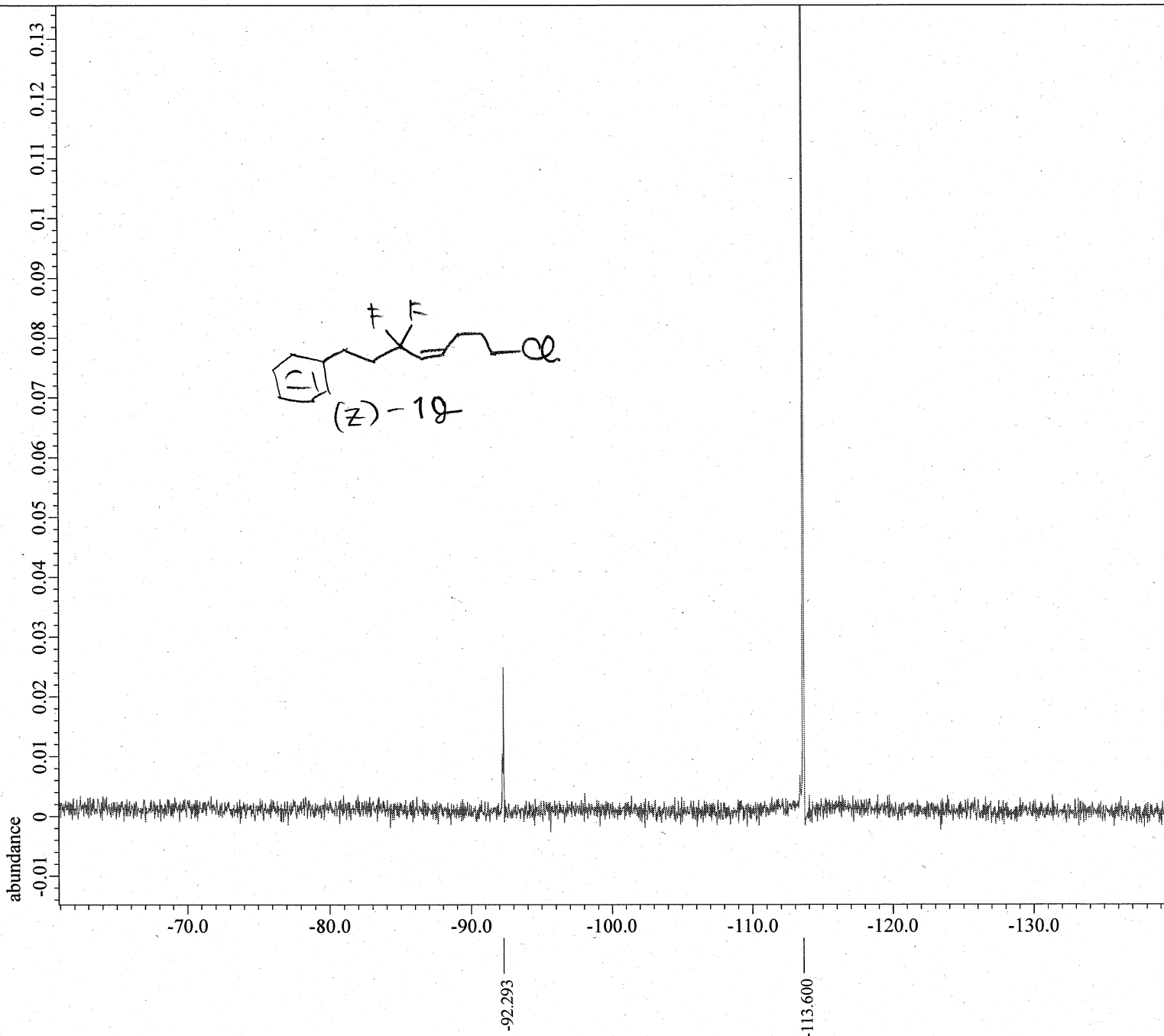


Filename = AKY626-carbon128\_Carbon-1  
Author = element  
Experiment = carbon.jxp  
Sample Id = AKY627-carbon128  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 7-MAR-2019 17:48:48  
Revision\_Time = 20-MAR-2019 15:49:34

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim\_Size = 26214  
X\_Domain = Carbon  
Dim\_Title = Carbon13  
Dim\_Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X\_Acq\_Duration = 1.0433312[s]  
X\_Domain = 13C  
X\_Freq = 100.33735165[MHz]  
X\_Offset = 100.0[ppm]  
X\_Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.95846665[Hz]  
X\_Sweep = 31.40703518[kHz]  
X\_Sweep\_Clipped = 25.12562814[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 399.03472754[MHz]  
Irr\_Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 50  
Temp\_Get = 17.4[dC]  
X\_90\_Width = 10.9[us]  
X\_Acq\_Time = 1.04333312[s]  
X\_Angle = 30[deg]  
X\_Atn = 5.4[dB]  
X\_Pulse = 3.63333333[us]  
Irr\_Atn\_Dec = 25.823[dB]  
Irr\_Atn\_No = 25.823[dB]  
Irr\_Noise = WALTZ  
Irr\_Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.04333312[s]



Filename = AKY626-FNMRag-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#632685  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 8-MAR-2019 02:05:08  
Revision\_Time = 20-MAR-2019 16:54:37

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 19[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

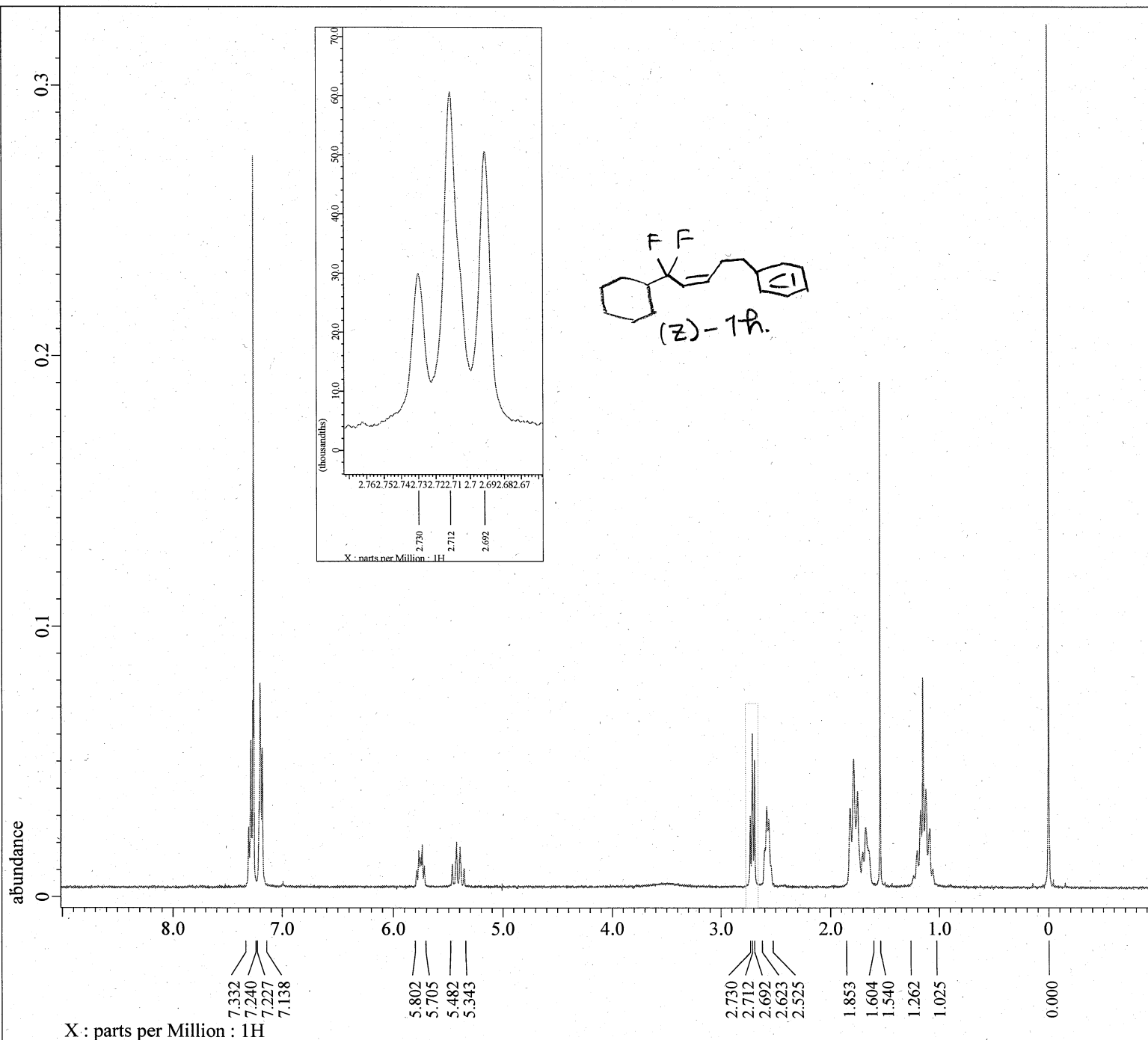
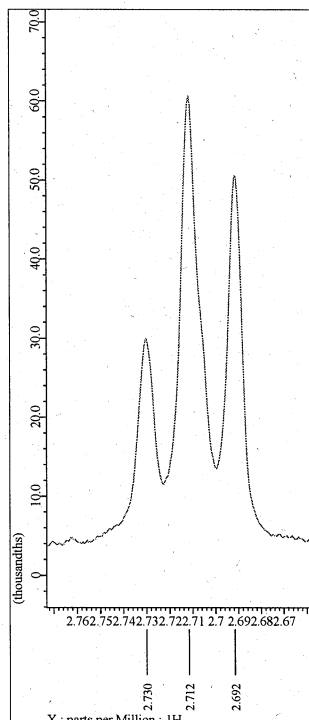
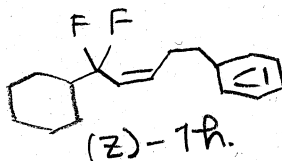


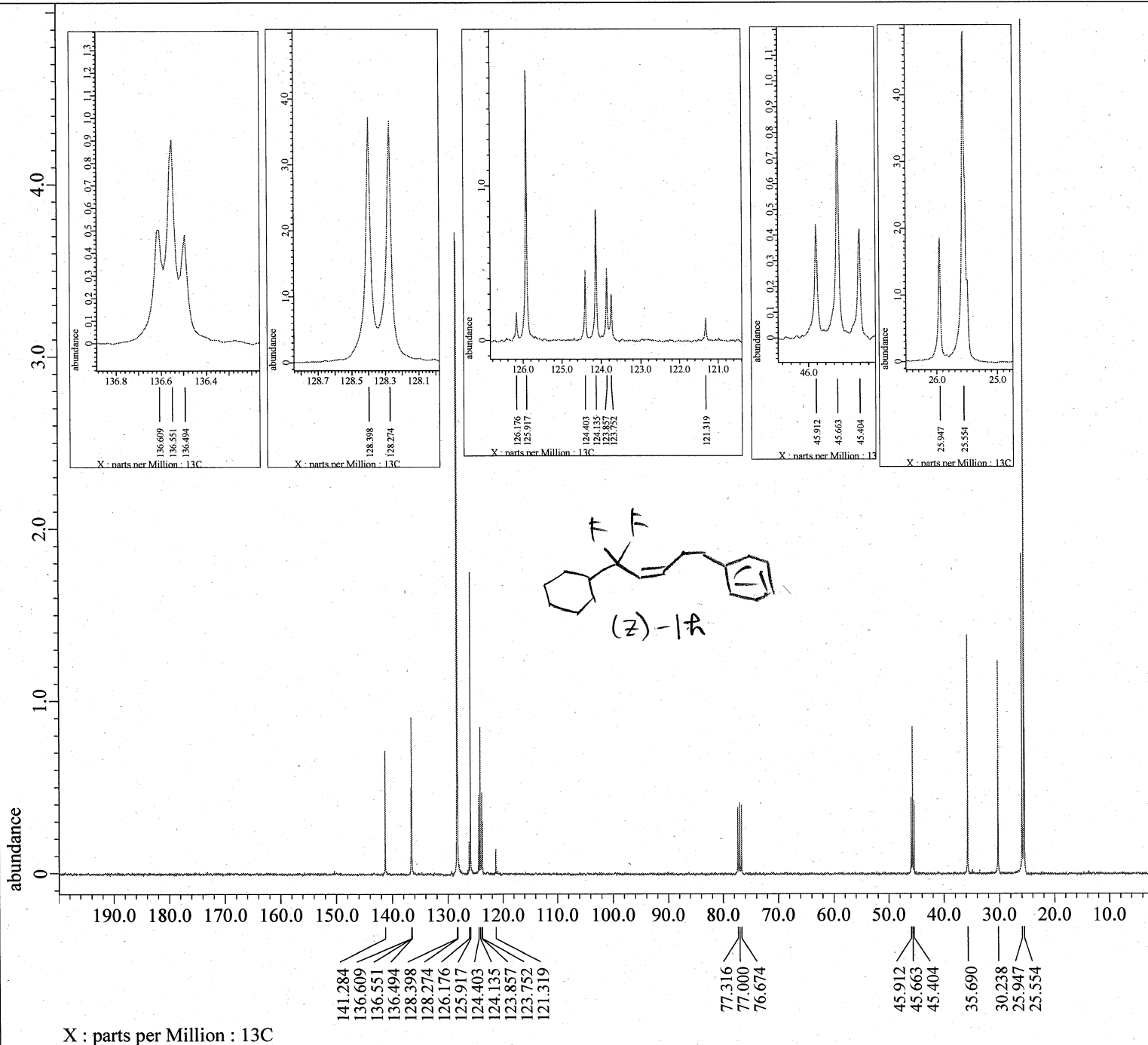
Filename = AKY627-pure-again-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = 1  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 12-NOV-2018 23:15:49  
Revision\_Time = 29-MAR-2019 19:20:35

Comment = single\_pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 26214  
X\_Domain = 1H  
Dim\_Title = 1H  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 2.20725248[s]  
X\_Domain = 1H  
X\_Freq = 395.88430144[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45305193[Hz]  
X\_Sweep = 7.42280285[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 395.88430144[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 1H  
Tri\_Freq = 395.88430144[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 38  
Temp\_Get = 28.2[dC]  
X\_90\_Width = 13.2[us]  
X\_Acq\_Time = 2.20725248[s]  
X\_Angle = 45[deg]  
X\_Atn = 3.5[dB]  
X\_Pulse = 6.6[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.20725248[s]





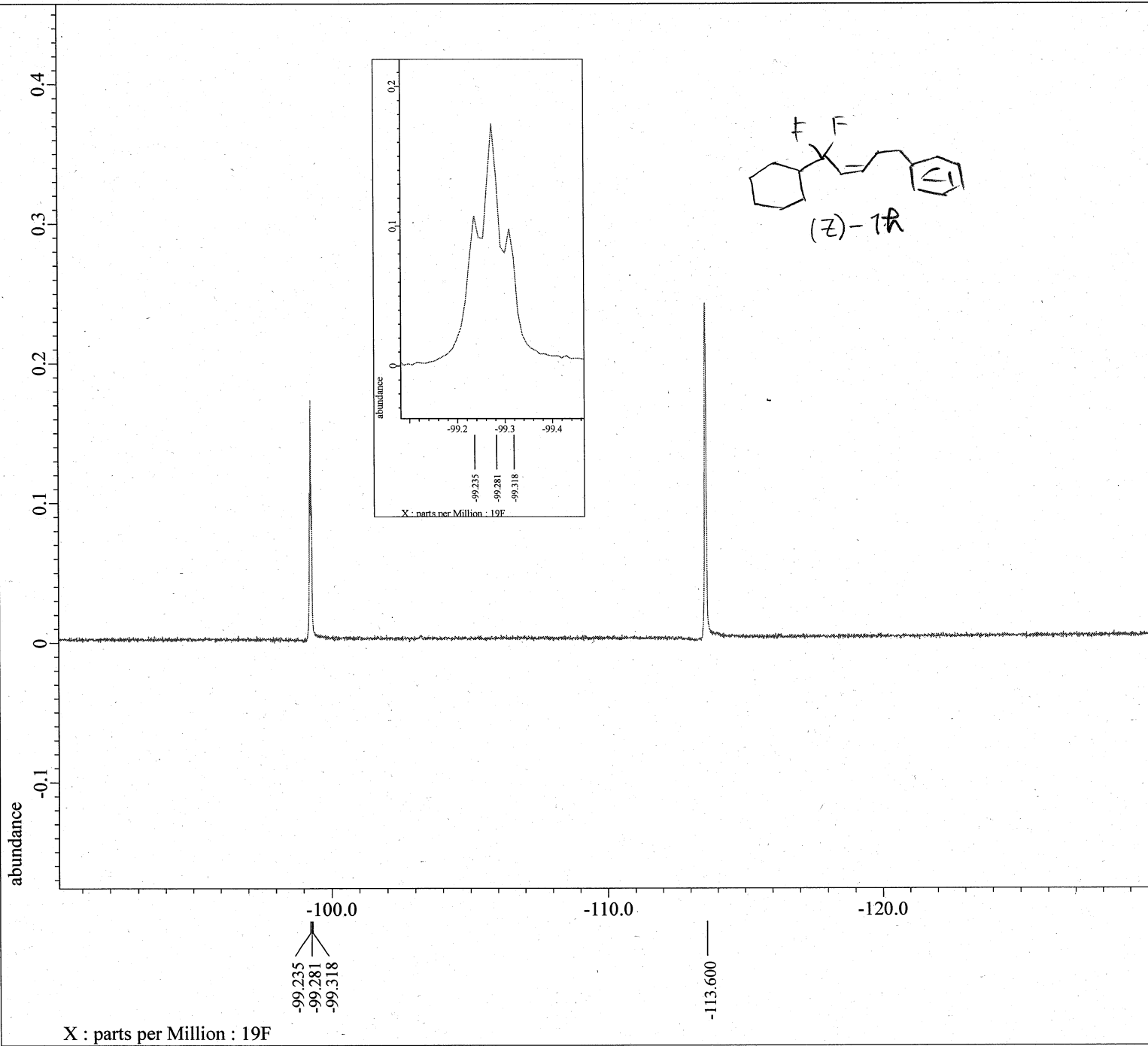
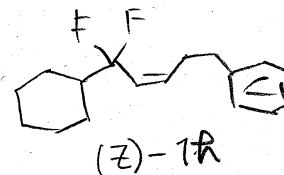
Filename = AKY627-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = 2  
Solvent = CHLOROFORM-D  
Actual Start Time = 12-NOV-2018 23:24:03  
Revision Time = 12-NOV-2018 20:54:25

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2 NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 30.7[dC]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]



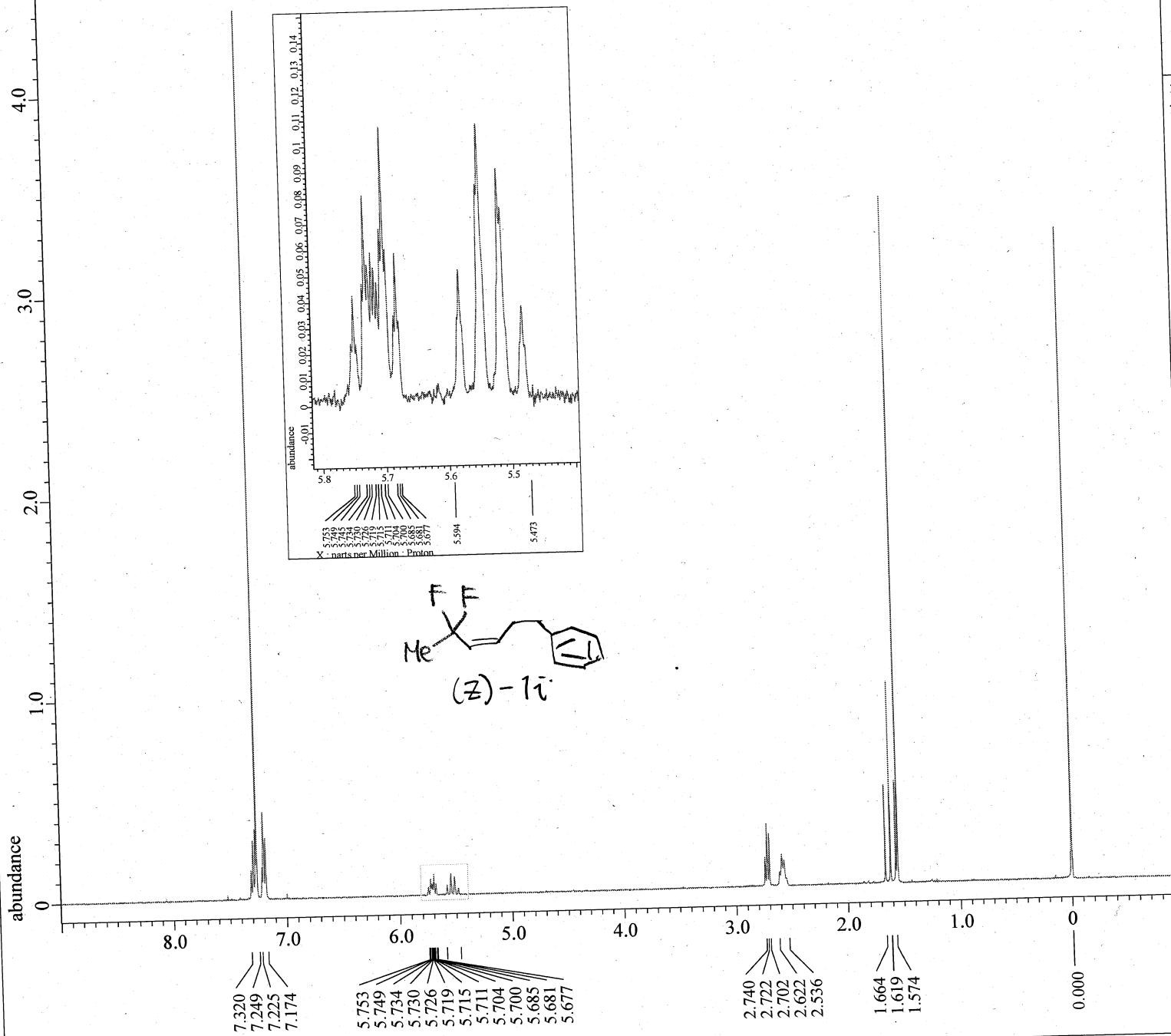


Filename = AKY627-fnmr-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#574099  
Solvent = CHLOROFORM-D  
Actual Start Time = 13-NOV-2018 00:26:14  
Revision Time = 12-NOV-2018 20:59:15

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq Duration = 0.29097984 [s]  
X Domain = 19F  
X Freq = 372.50336686 [MHz]  
X Offset = -70 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 3.4366642 [Hz]  
X Sweep = 56.30630631 [kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5 [s]  
Recvr Gain = 30  
Temp Get = 63.7 [dC]  
X 90 Width = 13.9 [us]  
X Acq Time = 0.29097984 [s]  
X Angle = 45 [deg]  
X Atn = 4 [dB]  
X Pulse = 6.95 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 5.29097984 [s]



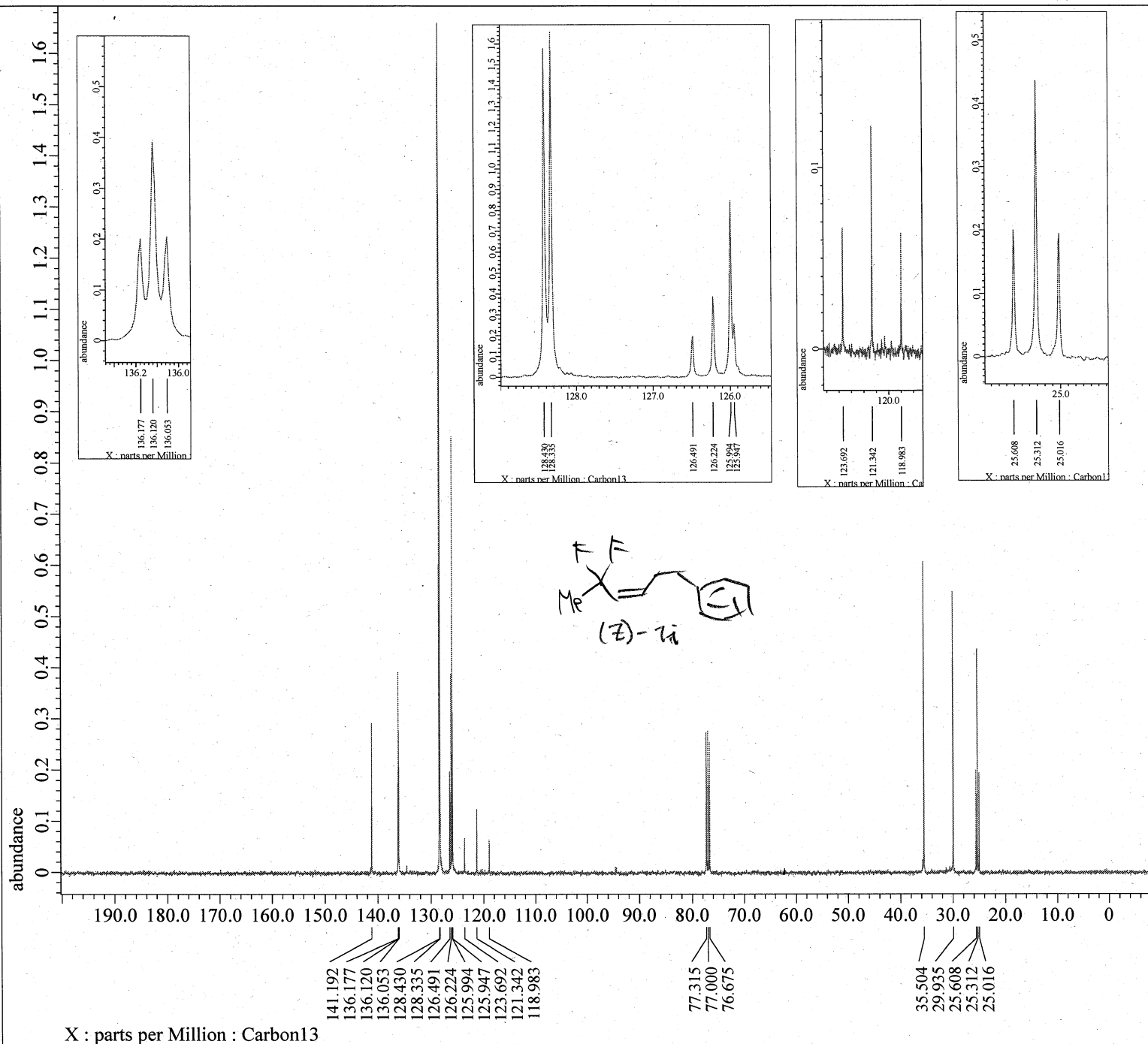
Filename = AKY837-pure-again\_Proton-  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY837-pure-again  
Solvent = CHLOROFORM-D  
Actual Start Time = 7-MAR-2019 14:12:53  
Revision Time = 29-MAR-2019 20:01:43

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221 [T] (400[MHz])  
X Acq Duration = 2.1889024 [s]  
X Domain = 1H  
X Freq = 399.03472754 [MHz]  
X Offset = 5.0 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45684997 [Hz]  
X Sweep = 7.48502994 [kHz]  
X Sweep Clipped = 5.98802395 [kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754 [MHz]  
Irr Offset = 5.0 [ppm]  
Tri Domain = Proton  
Tri Freq = 399.03472754 [MHz]  
Tri Offset = 5.0 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5 [s]  
Recvr Gain = 54  
Temp Get = 17.6 [dC]  
X 90 Width = 6.6 [us]  
X Acq Time = 2.1889024 [s]  
X Angle = 45 [deg]  
X Atn = 1 [dB]  
X Pulse = 3.3 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.1889024 [s]

X : parts per Million : Proton



Filename = AKY837-carbon-128\_Carbon-  
Author = element  
Experiment = carbon.jxp  
Sample Id = AKY837-carbon-128  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 7-MAR-2019 14:20:32  
Revision\_Time = 22-MAR-2019 13:26:03

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Carbon  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X Acq Duration = 1.04333312[s]  
X Domain = 13C  
X Freq = 100.33735165[MHz]  
X Offset = 100.0[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95846665[Hz]  
X Sweep = 31.40703518[kHz]  
X Sweep Clipped = 25.12562814[kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754[MHz]  
Irr Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 128  
Total Scans = 128

Relaxation\_Delay = 2[s]  
Recvr Gain = 50  
Temp Get = 17.6[dc]  
X 90\_Width = 10.9[us]  
X Acq Time = 1.04333312[s]  
X Angle = 30[deg]  
X Atn = 5.4[dB]  
X Pulse = 3.63333333[us]  
Irr Atn\_Dec = 25.823[dB]  
Irr Atn\_Noise = 25.823[dB]  
Irr Noise = WALTZ  
Irr Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition\_Time = 3.04333312[s]

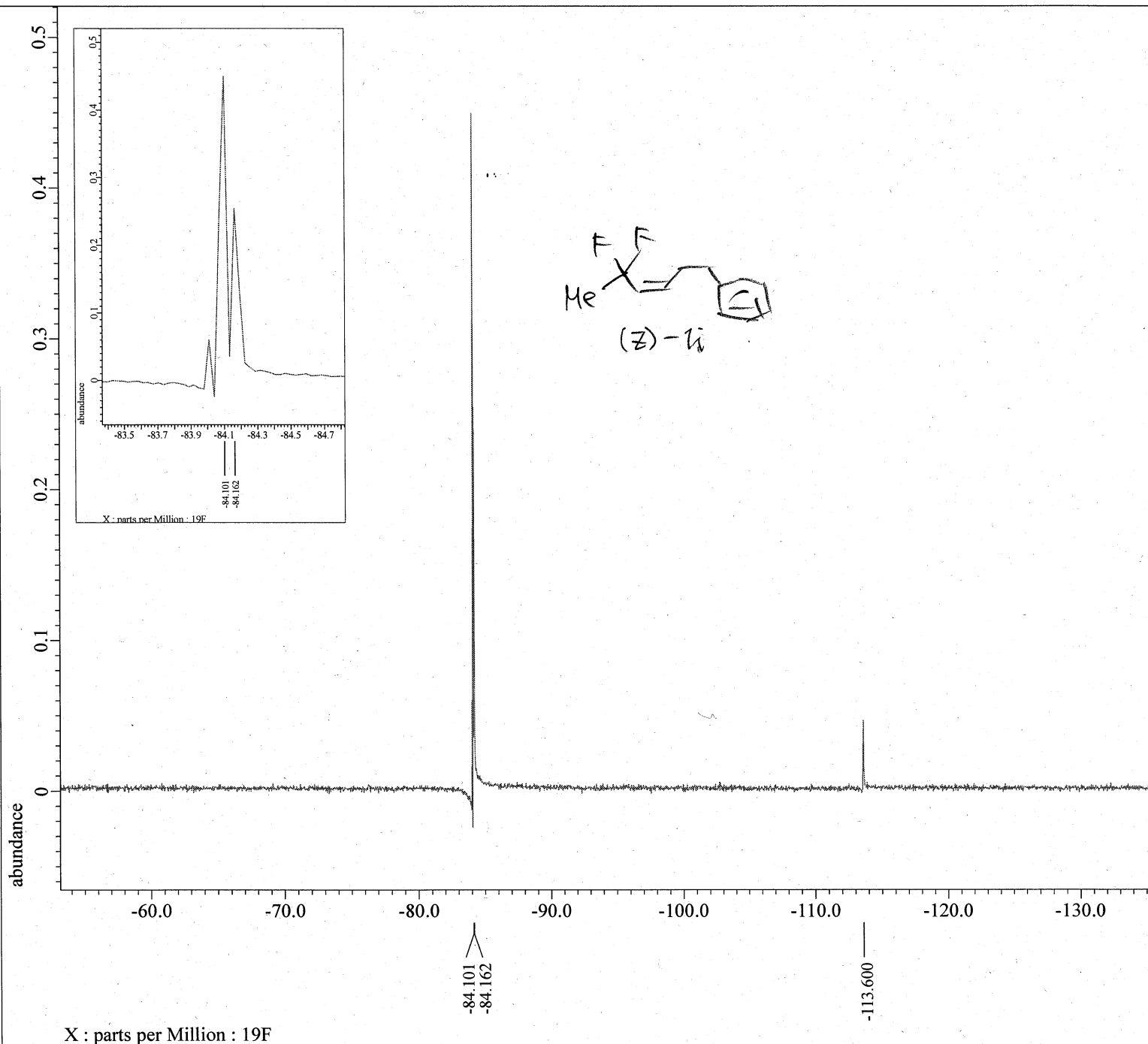
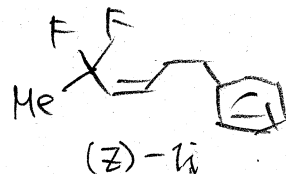


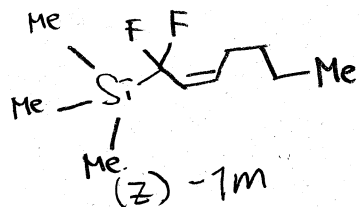
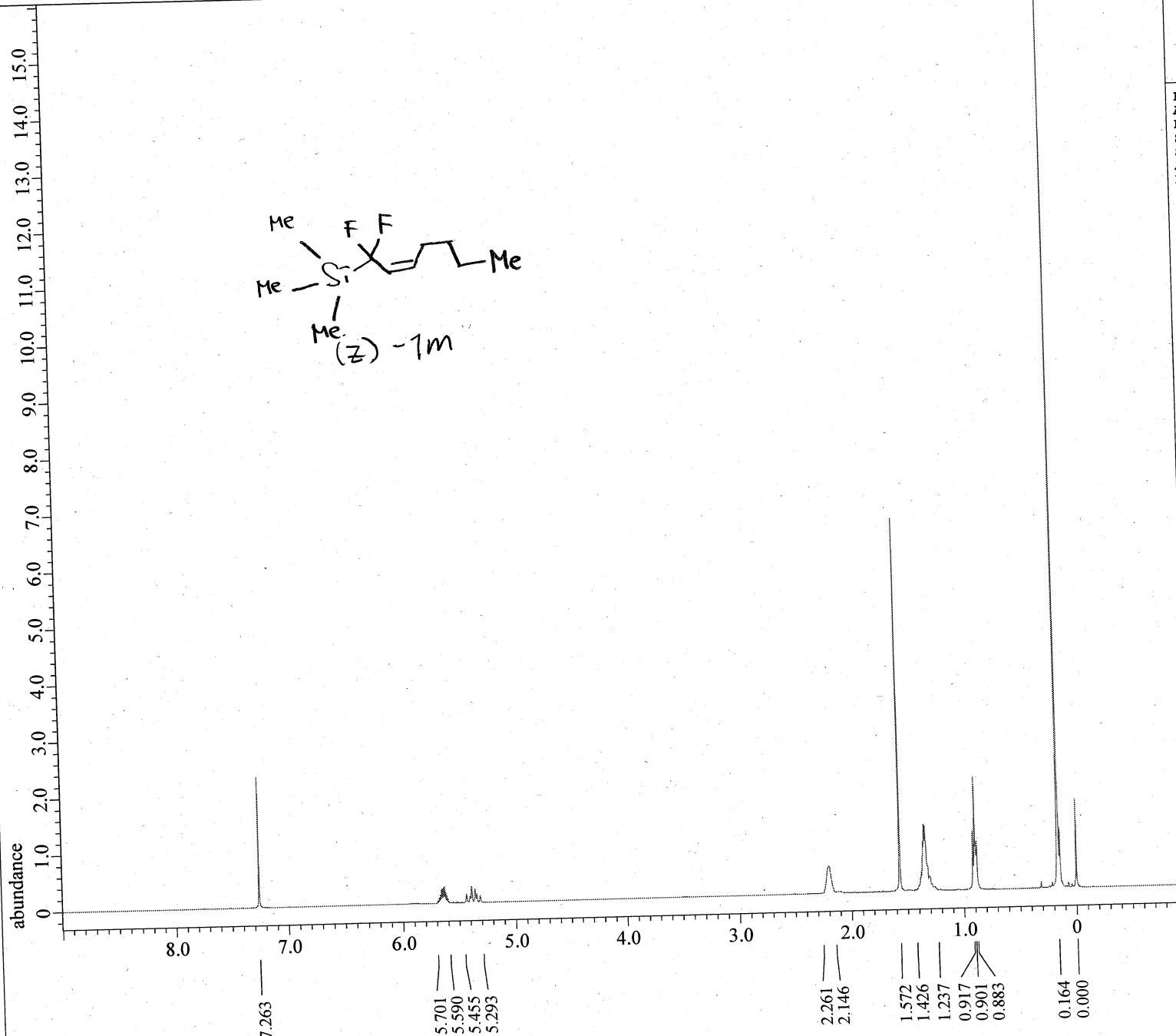
Filename = AKY837-pure-FNMR-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#508373  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 7-MAR-2019 22:38:00  
Revision\_Time = 22-MAR-2019 13:28:47

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 26  
Temp\_Get = 19.5[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
DanTe Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]





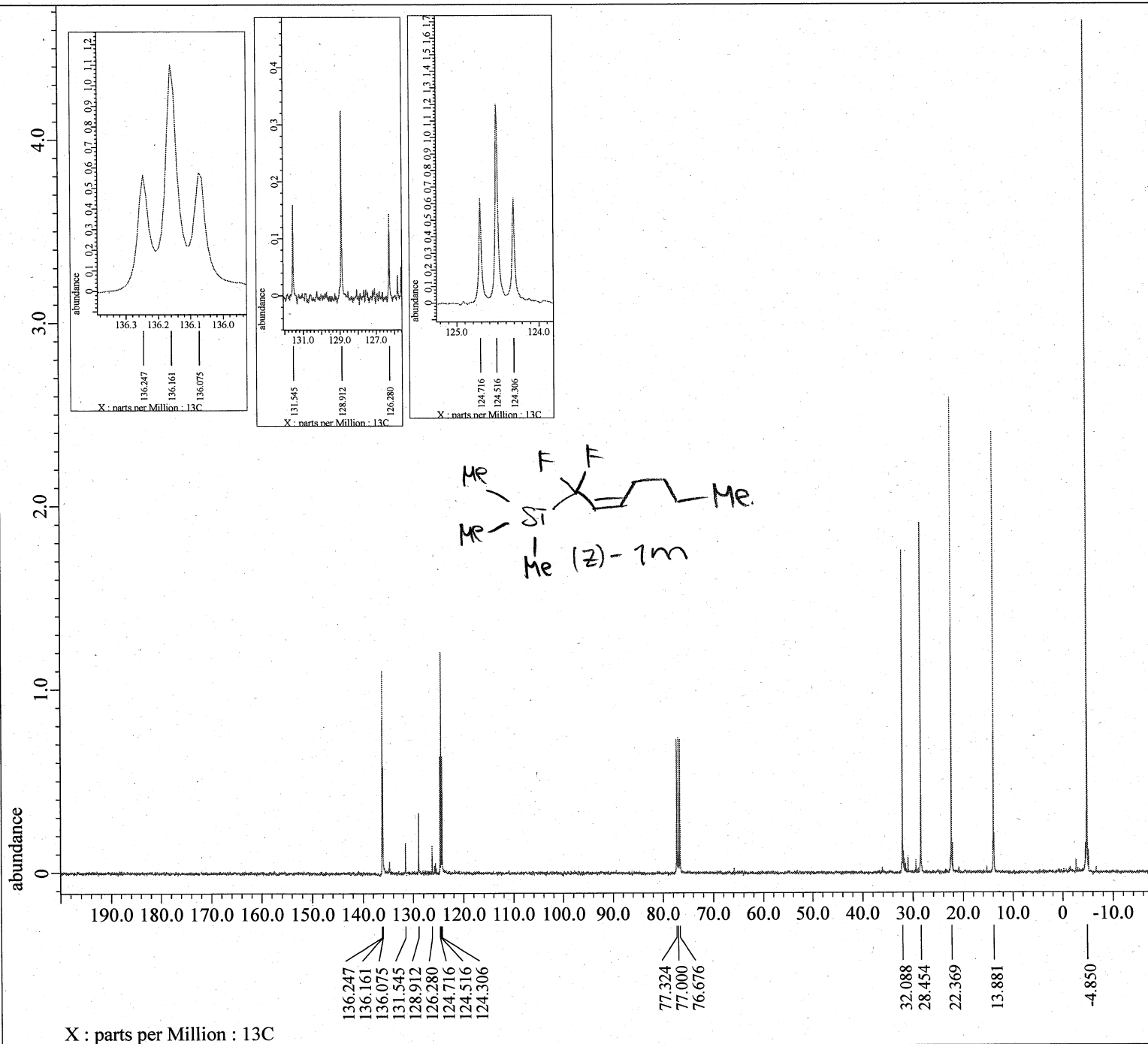
Filename = AKY639-pure\_Proton-1-2.jd  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY639-pure  
Solvent = CHLOROFORM-D  
Actual Start Time = 2-AUG-2018 15:39:47  
Revision Time = 29-MAR-2019 20:07:17

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814[T] (400[MHz])  
X Acq Duration = 2.18103808[s]  
X Domain = 1H  
X Freq = 400.53219825[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45849727[Hz]  
X Sweep = 7.51201923[kHz]  
X Sweep Clipped = 6.00961538[kHz]  
Irr Domain = Proton  
Irr Freq = 400.53219825[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = Proton  
Tri Freq = 400.53219825[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 42  
Temp Get = 20.3[dC]  
X 90 Width = 6.22[us]  
X Acq Time = 2.18103808[s]  
X Angle = 45[deg]  
X Atn = 0.8[dB]  
X Pulse = 3.11[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.18103808[s]

X : parts per Million : Proton



Filename = AKY639-carbonag-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = S#463997  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 7-AUG-2018 20:27:36  
Revision Time = 13-NOV-2018 14:07:50

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 1.06430464[s]  
X Domain = 13C  
X Freq = 98.51479726[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061[Hz]  
X Sweep = 30.78817734[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total\_Scans = 256

Relaxation\_Delay = 2[s]  
Recvr Gain = 60  
Temp Get = 21.9[dC]  
X 90 Width = 9.11[us]  
X Acq Time = 1.06430464[s]  
X Angle = 30[deg]  
X Atn = 4.9[dB]  
X Pulse = 3.03666667[us]  
Irr Atn Dec = 22.255[dB]  
Irr Atn Noe = 22.255[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.06430464[s]

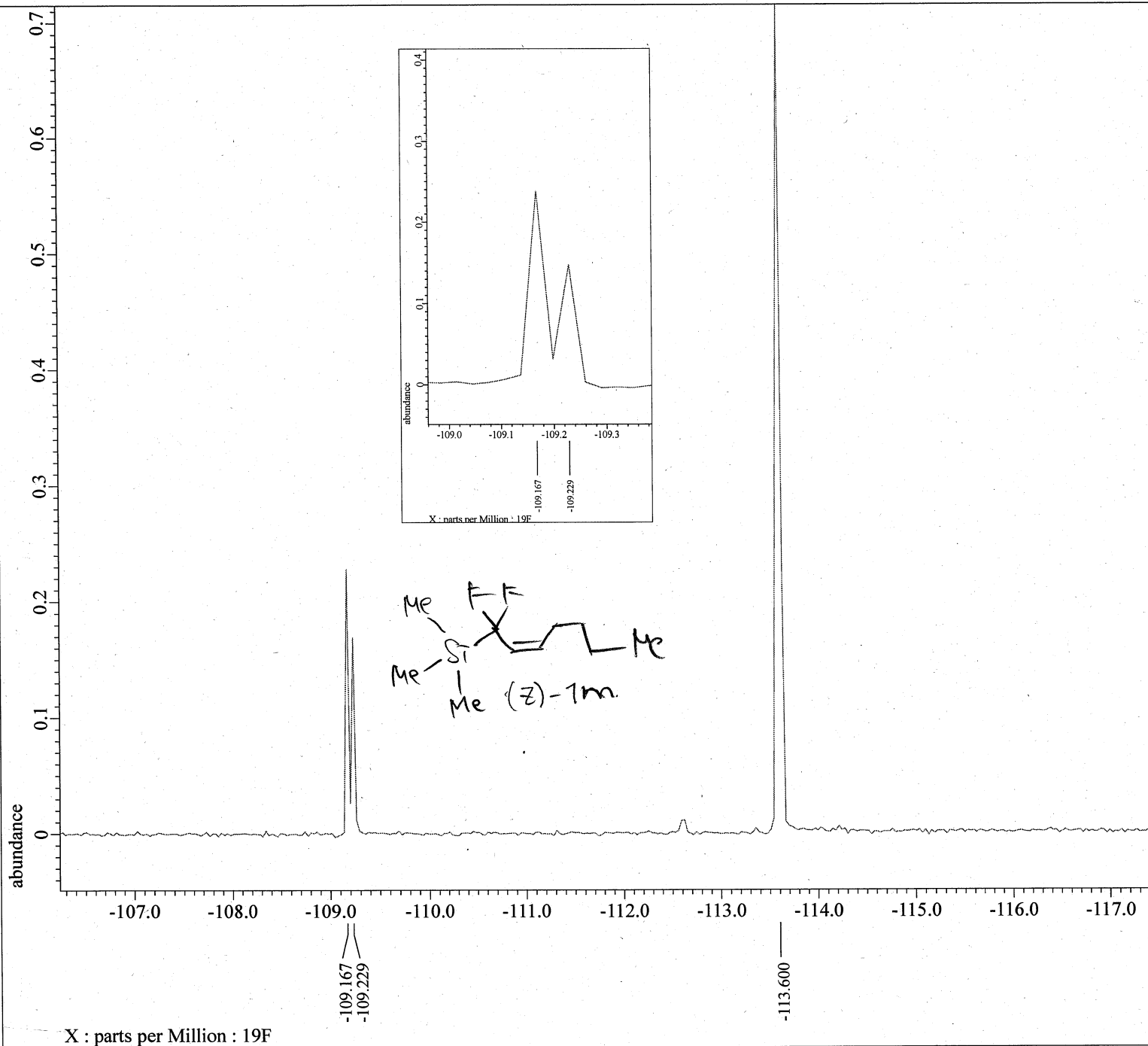


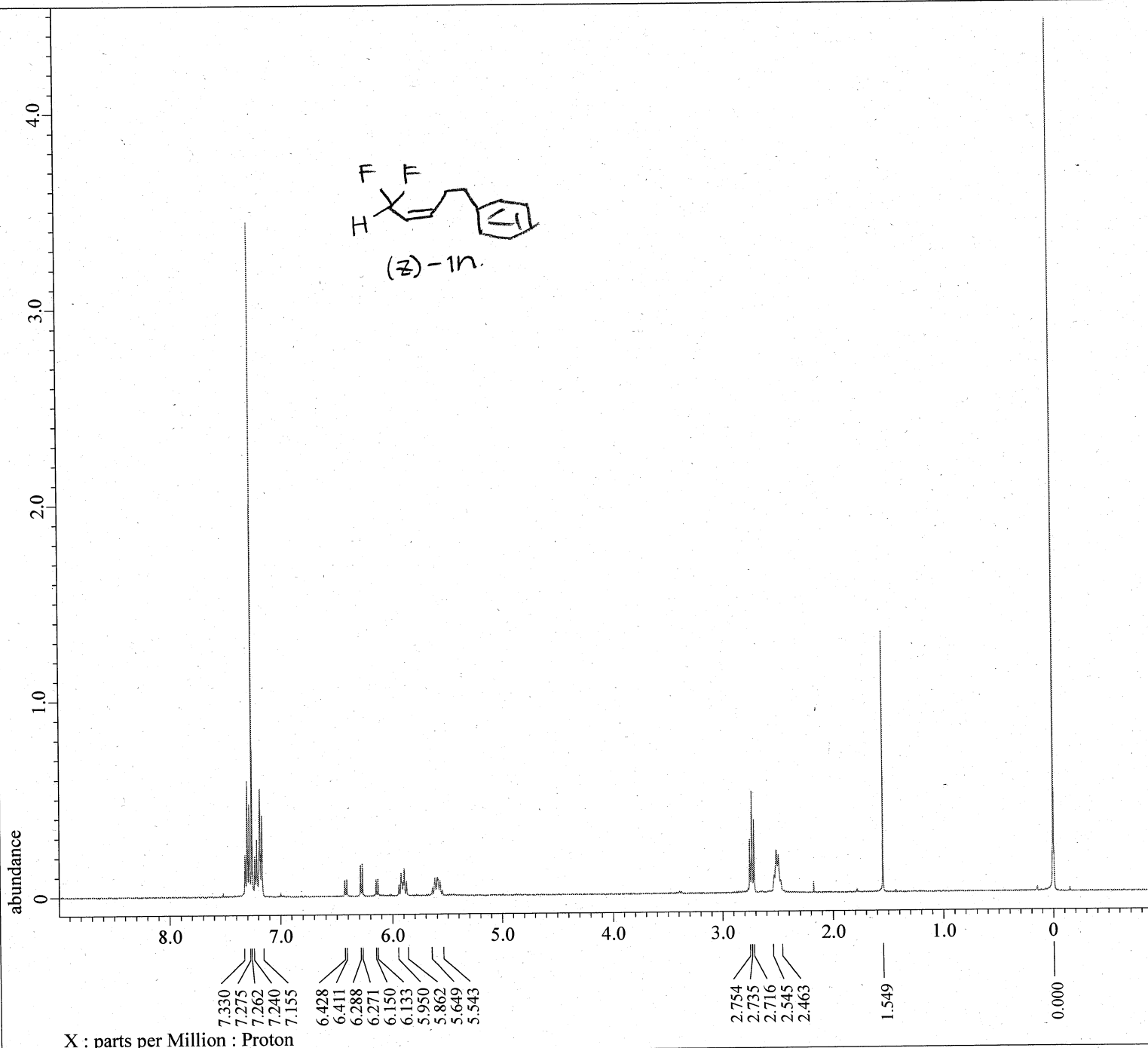
Filename = AKY639-pure-fnmr-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#348065  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 7-AUG-2018 18:15:04  
Revision\_Time = 13-NOV-2018 14:10:17

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECK 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 22.4[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 5.08781824[s]





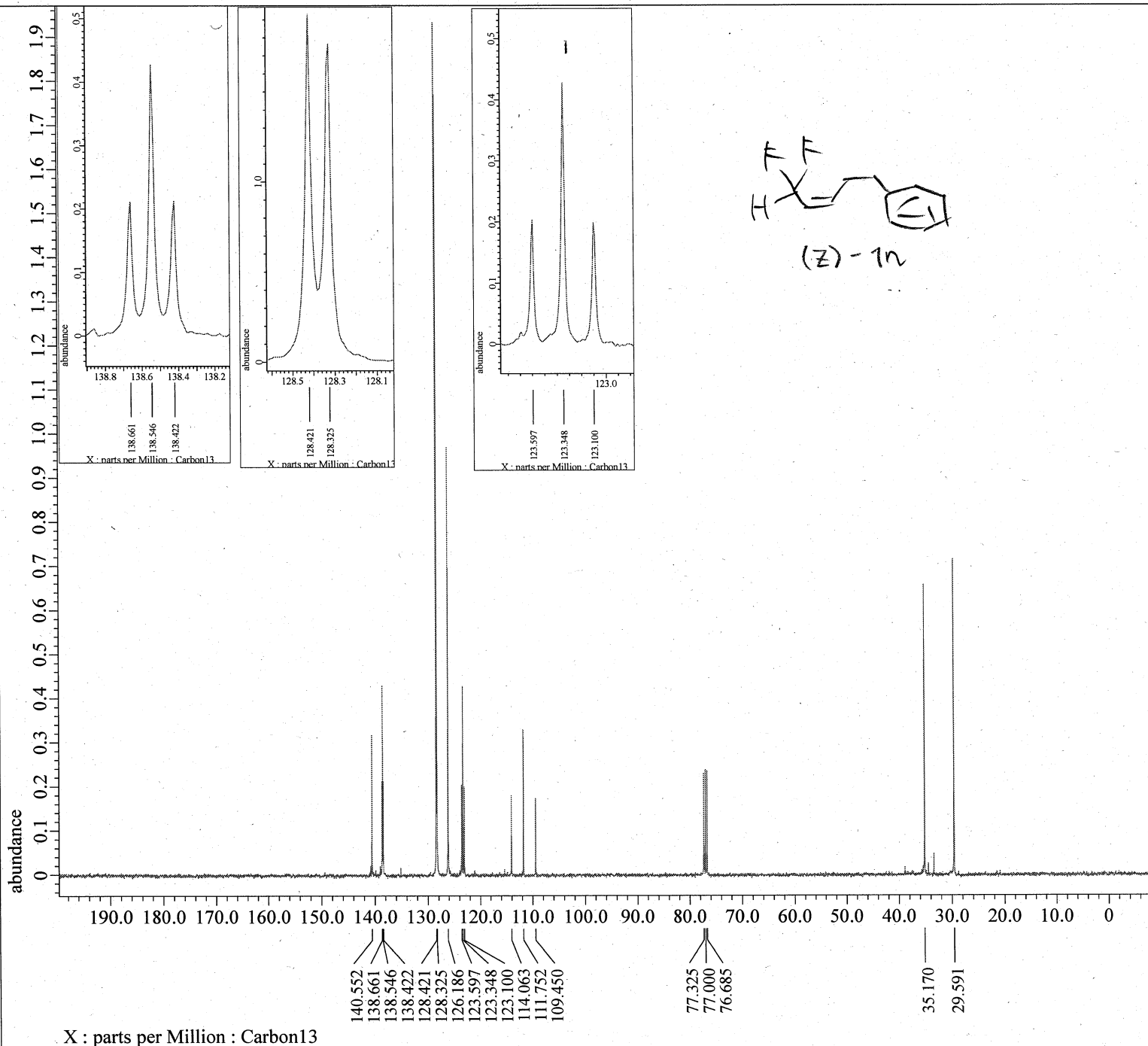
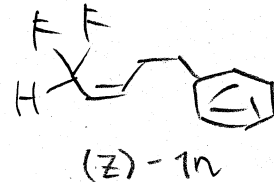
Filename = AKY206-cugel\_Proton-2-4.j  
Author = element  
Experiment = proton.jxp  
Sample\_Id = AKY206-pure  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 20-FEB-2017 11:32:25  
Revision\_Time = 29-MAR-2019 20:13:09

Comment = single pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 26214  
X\_Domain = Proton  
Dim\_Title = Proton  
Dim\_Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.4073814 [T] (400 [MHz])  
X\_Acq\_Duration = 2.18103808 [s]  
X\_Domain = 1H  
X\_Freq = 400.53219825 [MHz]  
X\_Offset = 5 [ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45849727 [Hz]  
X\_Sweep = 7.51201923 [kHz]  
X\_Sweep\_Clipped = 6.00961538 [kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825 [MHz]  
Irr\_Offset = 5 [ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825 [MHz]  
Tri\_Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

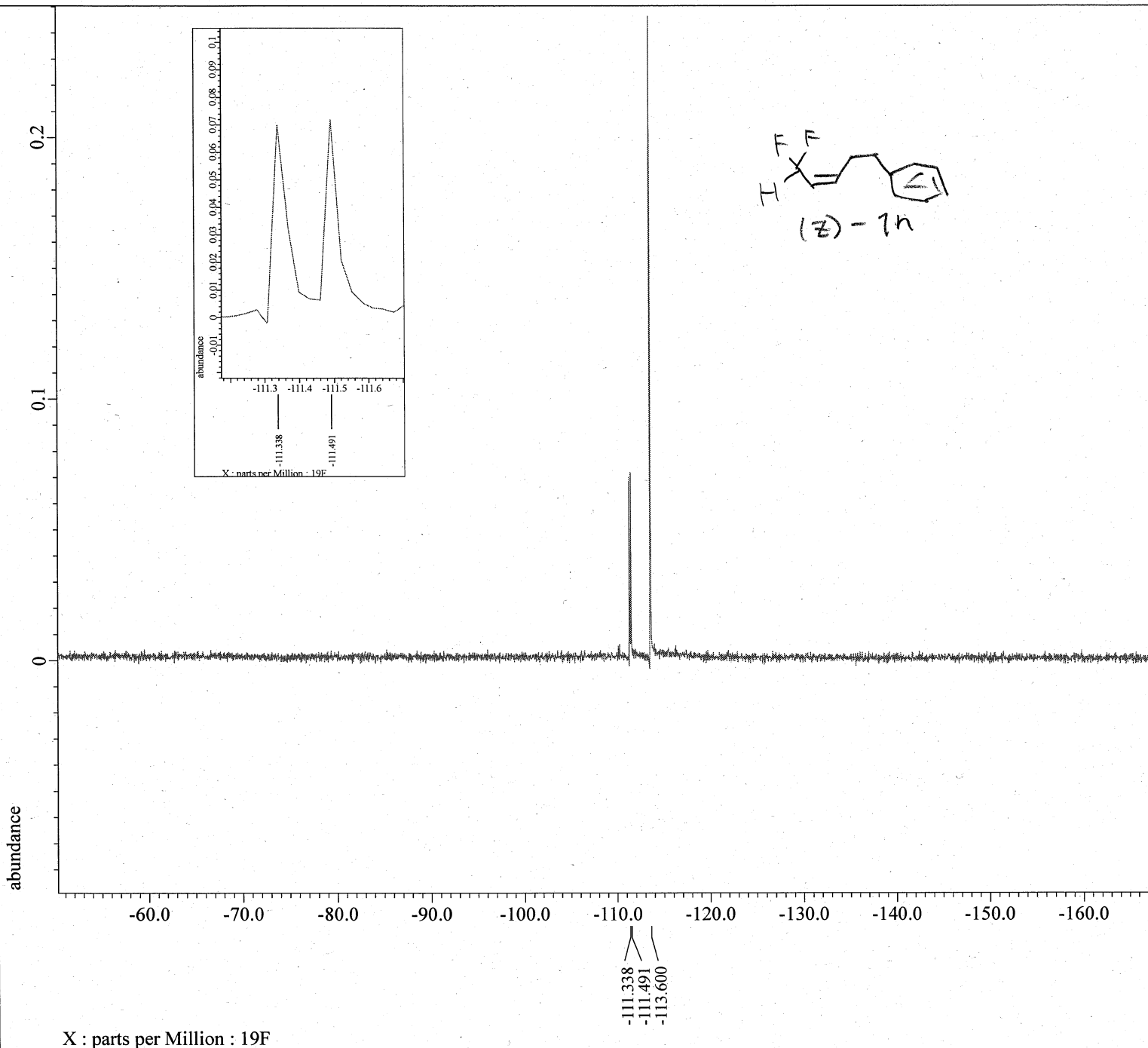
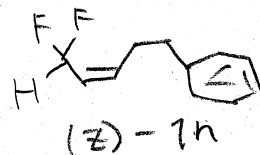
Relaxation\_Delay = 5 [s]  
Recvr\_Gain = 50  
Temp\_Get = 18.4 [dC]  
X\_90\_Width = 6 [us]  
X\_Acq\_Time = 2.18103808 [s]  
X\_Angle = 45 [deg]  
X\_Atn = 0.8 [dB]  
X\_Pulse = 3 [us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1 [s]  
Repetition\_Time = 7.18103808 [s]





Filename = AKY206-carbon128\_Carbon-1  
Author = element  
Experiment = carbon.jxp  
Sample Id = AKY206-carbon128  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 7-MAR-2019 16:45:00  
Revision\_Time = 22-MAR-2019 16:55:21  
Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Carbon  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR  
Field Strength = 9.37221[T] (400[MHz])  
X\_Acq\_Duration = 1.0433312[s]  
X\_Domain = 13C  
X\_Freq = 100.33735165[MHz]  
X\_Offset = 100.0[ppm]  
X\_Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.95846665[Hz]  
X\_Sweep = 31.40703518[kHz]  
X\_Sweep\_Clippped = 25.12562814[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 399.03472754[MHz]  
Irr\_Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128  
Relaxation\_Delay = 2[s]  
Recvr\_Gain = 50  
Temp\_Get = 17.3[dC]  
X\_90\_Width = 10.9[us]  
X\_Acq\_Time = 1.0433312[s]  
X\_Angle = 30[deg]  
X\_Atn = 5.4[dB]  
X\_Pulse = 3.6333333[us]  
Irr\_Atn\_Dec = 25.823[dB]  
Irr\_Atn\_No = 25.823[dB]  
Irr\_Noise = WALTZ  
Irr\_Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.0433312[s]

X : parts per Million : Carbon13

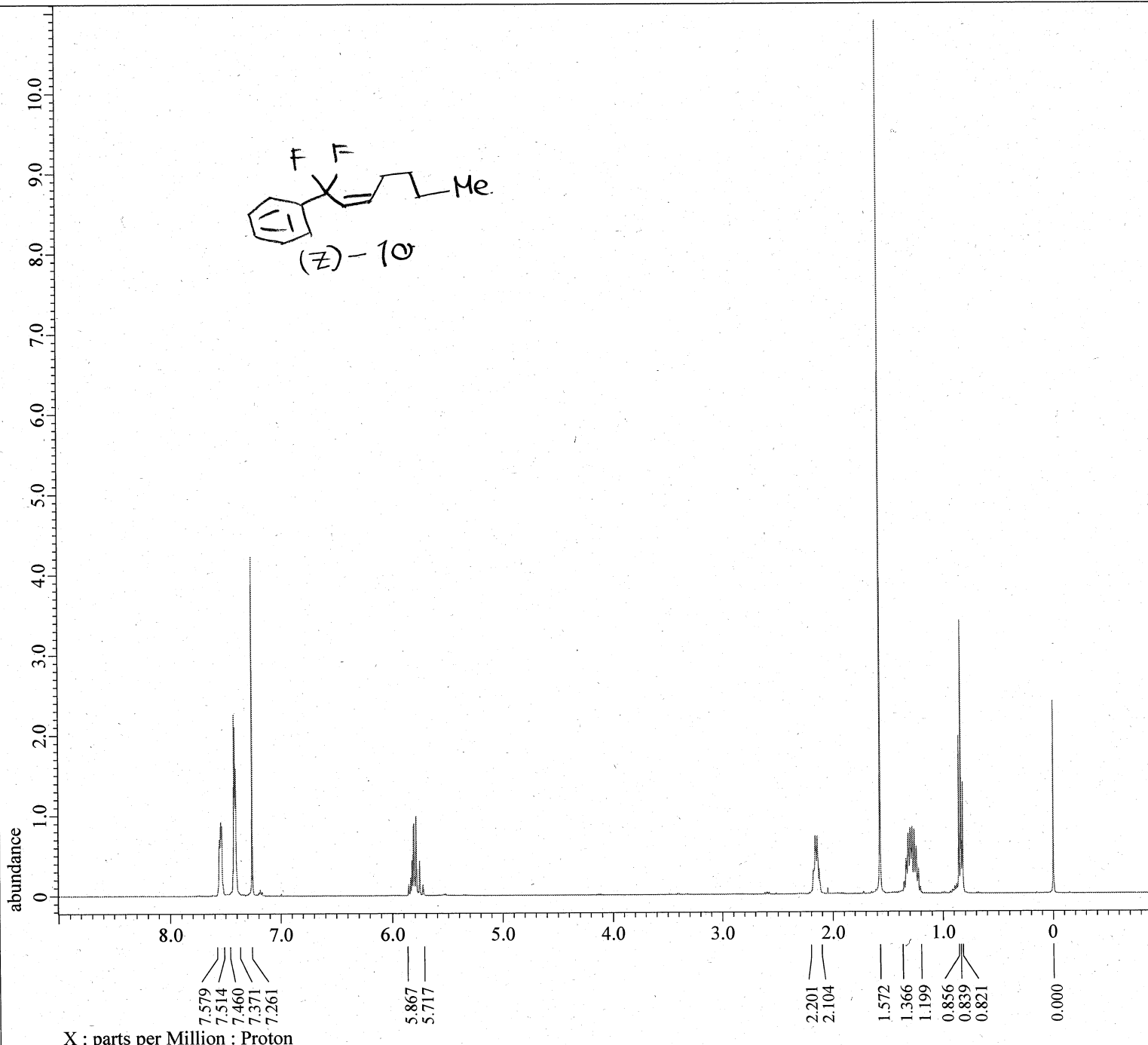


Filename = AKY206-pure-FNMR-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#595868  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 8-MAR-2019 01:03:38  
Revision\_Time = 22-MAR-2019 16:57:42

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 19.1[dC]  
X 90 Width = 13.9[us]  
X Acq Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]

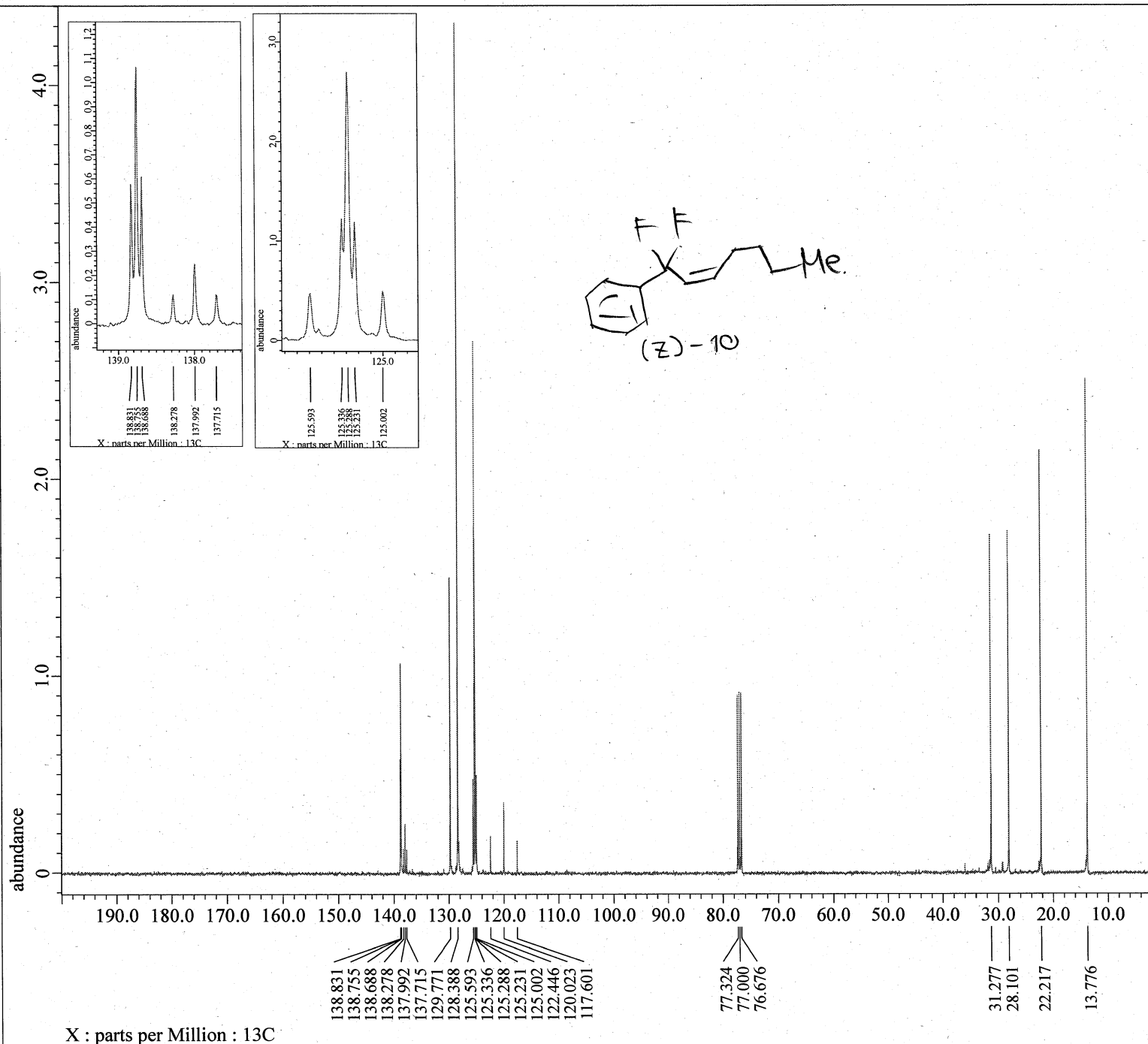


Filename = AKY640-gpc-2\_Proton-1-2.j  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY640-gpc-2  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 3-AUG-2018 21:48:11  
Revision\_Time = 29-MAR-2019 20:17:26

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814 [T] (400 [MHz])  
X Acq\_Duration = 2.18103808 [s]  
X Domain = 1H  
X Freq = 400.53219825 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45849727 [Hz]  
X Sweep = 7.51201923 [kHz]  
X Sweep\_Clippped = 6.00961538 [kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825 [MHz]  
Irr\_Offset = 5 [ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825 [MHz]  
Tri\_Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr\_Gain = 48  
Temp\_Get = 19.8 [dC]  
X 90\_Width = 6.22 [us]  
X Acq\_Time = 2.18103808 [s]  
X\_Angle = 45 [deg]  
X\_Atn = 0.8 [dB]  
X Pulse = 3.11 [us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1 [s]  
Repetition\_Time = 7.18103808 [s]

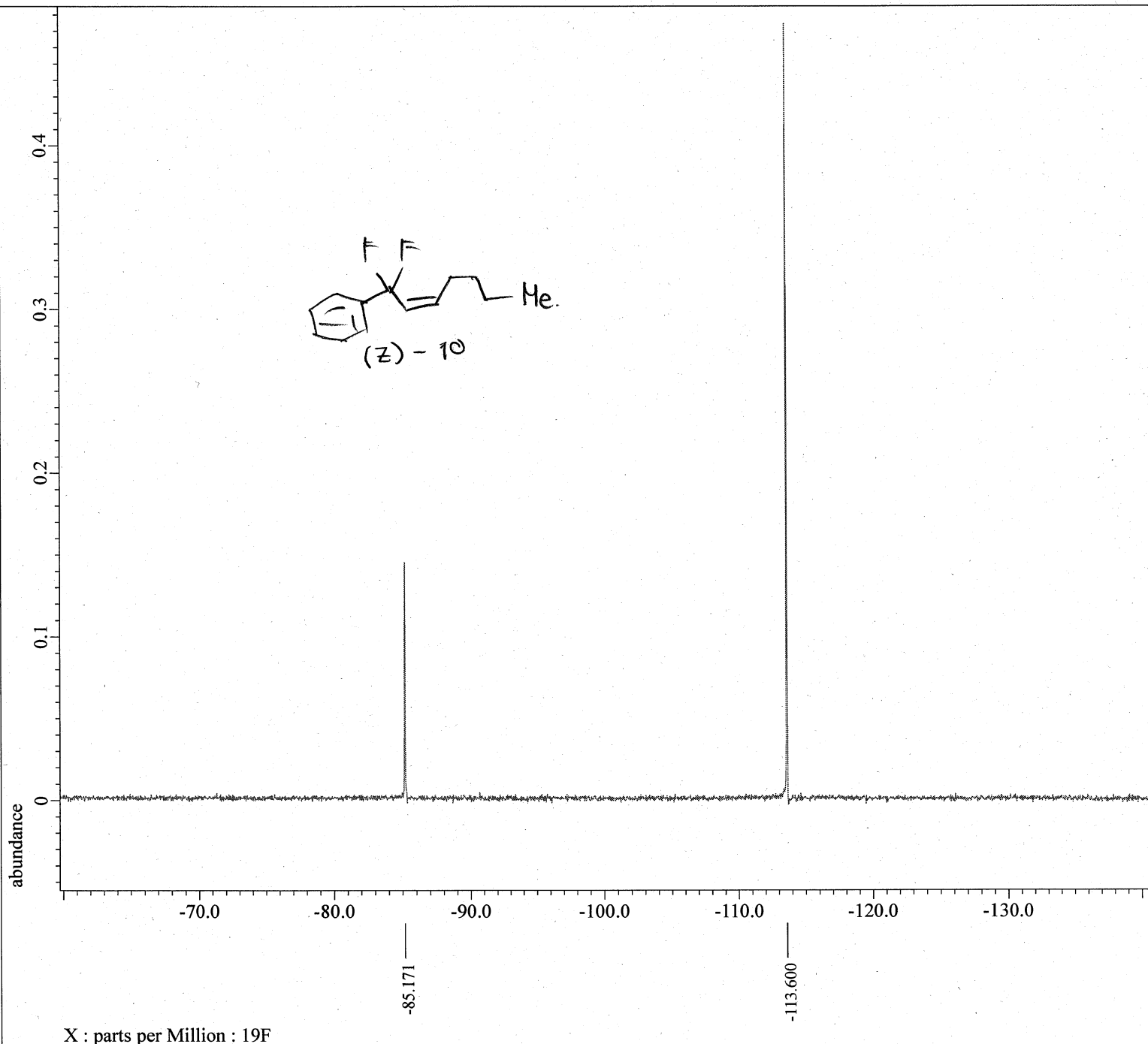


Filename = AKY640-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = S#515720  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 4-AUG-2018 21:54:12  
Revision\_Time = 13-NOV-2018 13:51:10

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 1.06430464[s]  
X Domain = 13C  
X Freq = 98.51479726[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061[Hz]  
X Sweep = 30.78817734[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total\_Scans = 256

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 60  
Temp\_Get = 22.1[dC]  
X\_90\_Width = 9.11[us]  
X\_Acq\_Time = 1.06430464[s]  
X\_Angle = 30[deg]  
X\_Atn = 4.9[dB]  
X\_Pulse = 3.03666667[us]  
Irr\_Atn\_Dec = 22.255[dB]  
Irr\_Atn\_No = 22.255[dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.06430464[s]

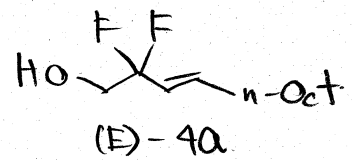


Filename = AKY640-fnmr-1real-1.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#639198  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 21-AUG-2018 02:20:24  
Revision\_Time = 13-NOV-2018 13:53:46

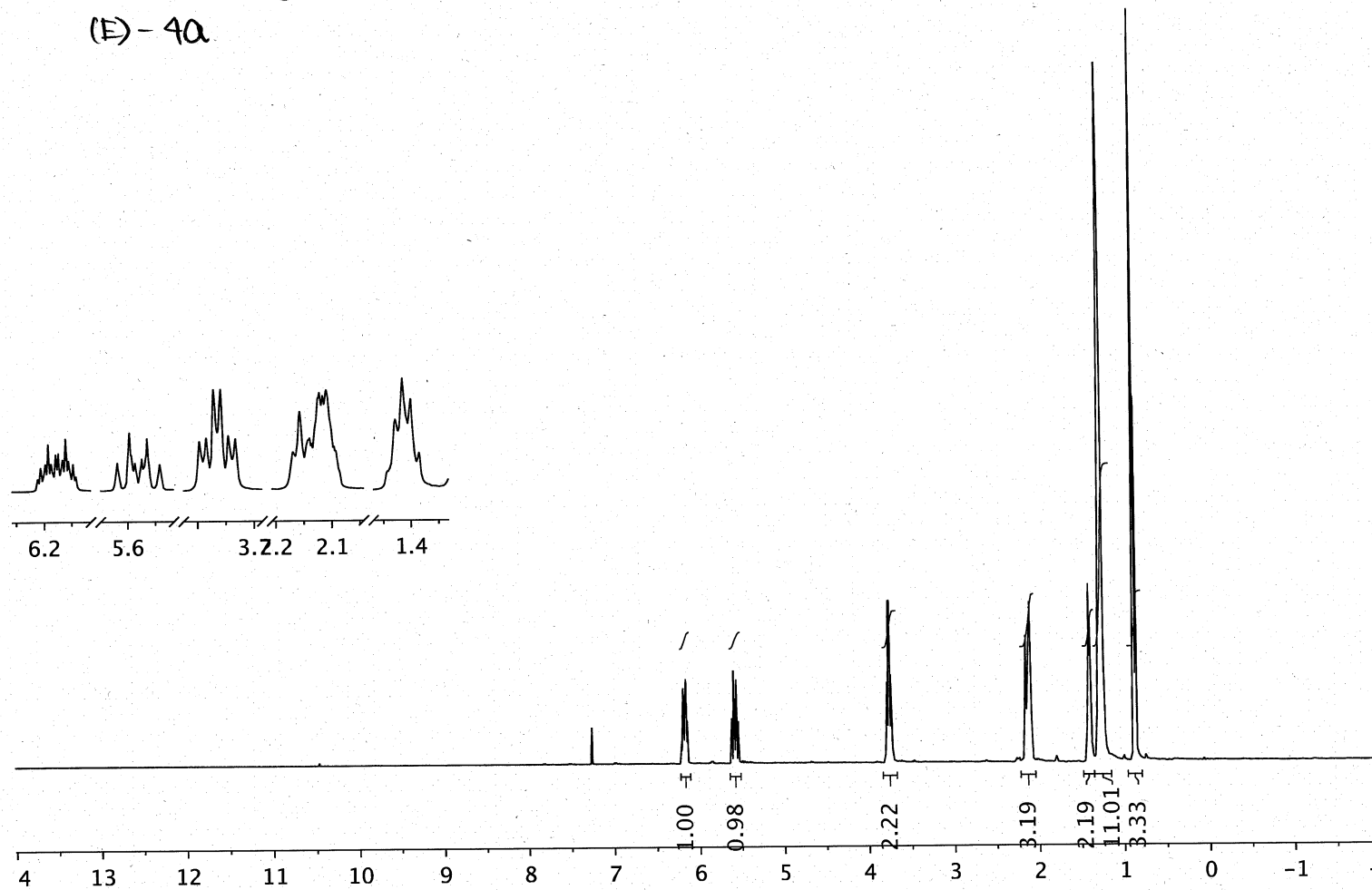
Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

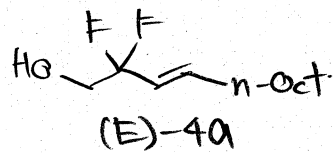
Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 22.2[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

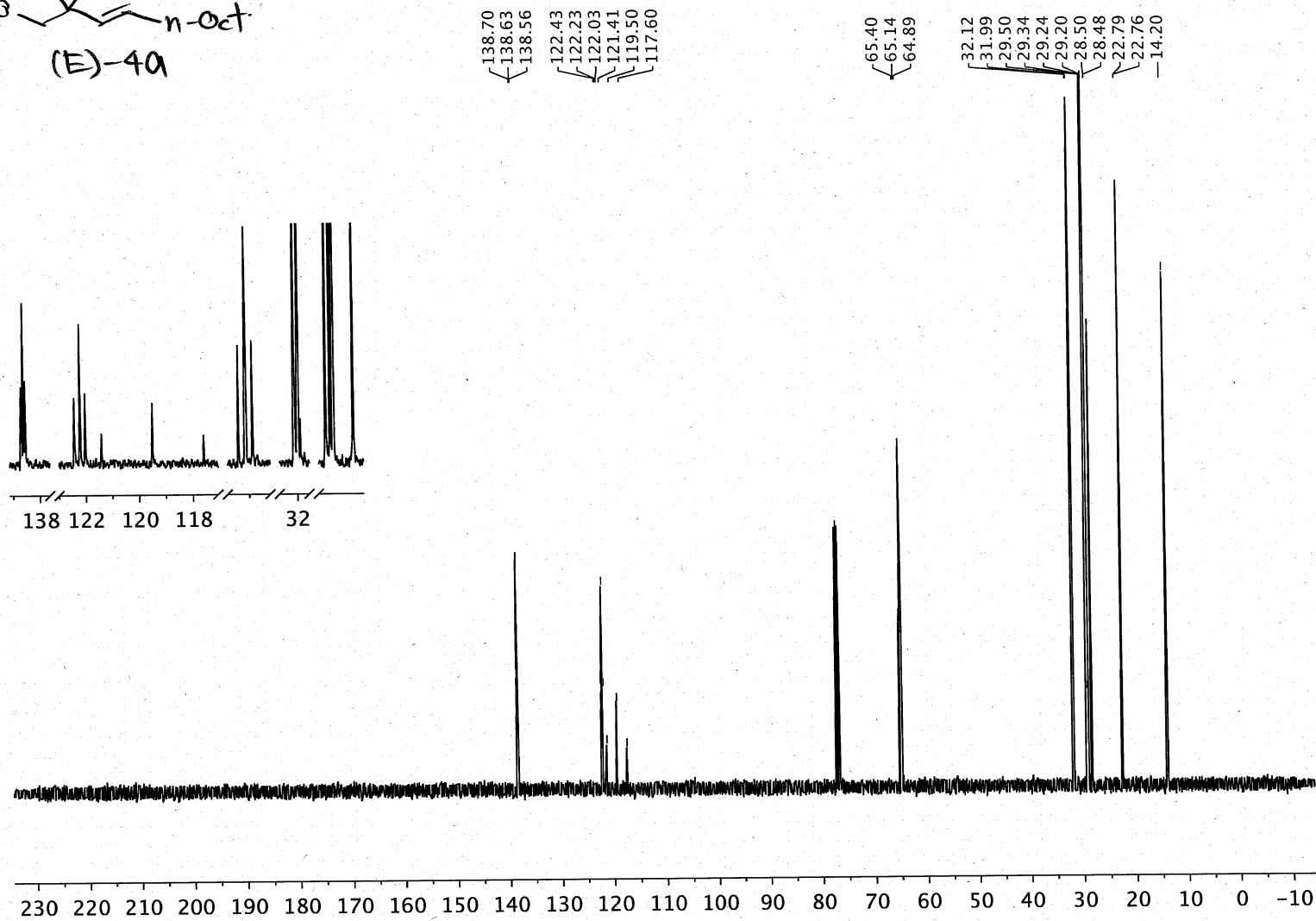


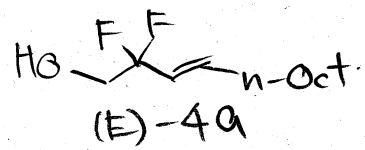
4a <sup>1</sup>H NMR Spectrum



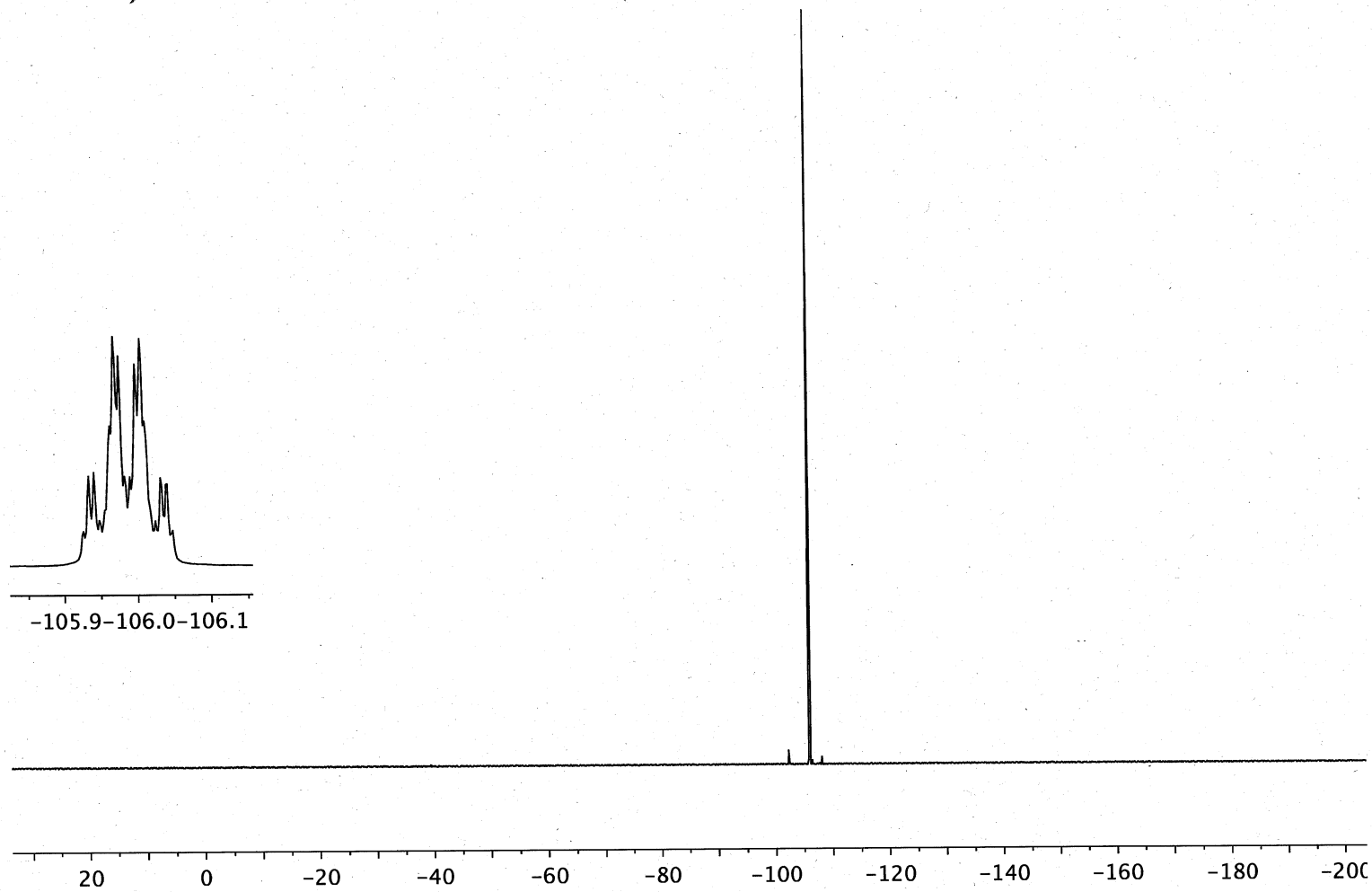


4a <sup>13</sup>C NMR Spectrum

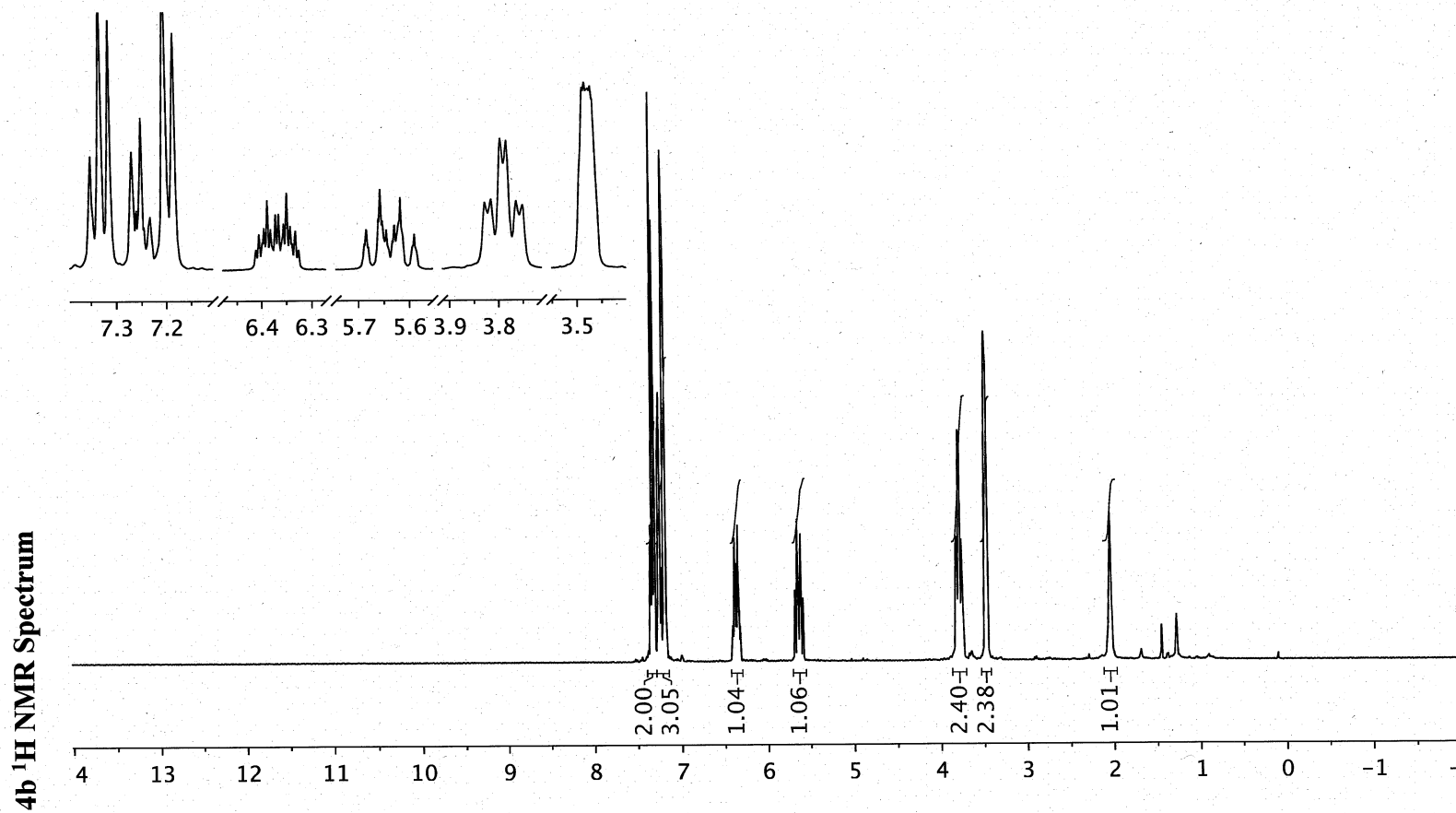
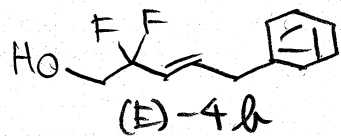


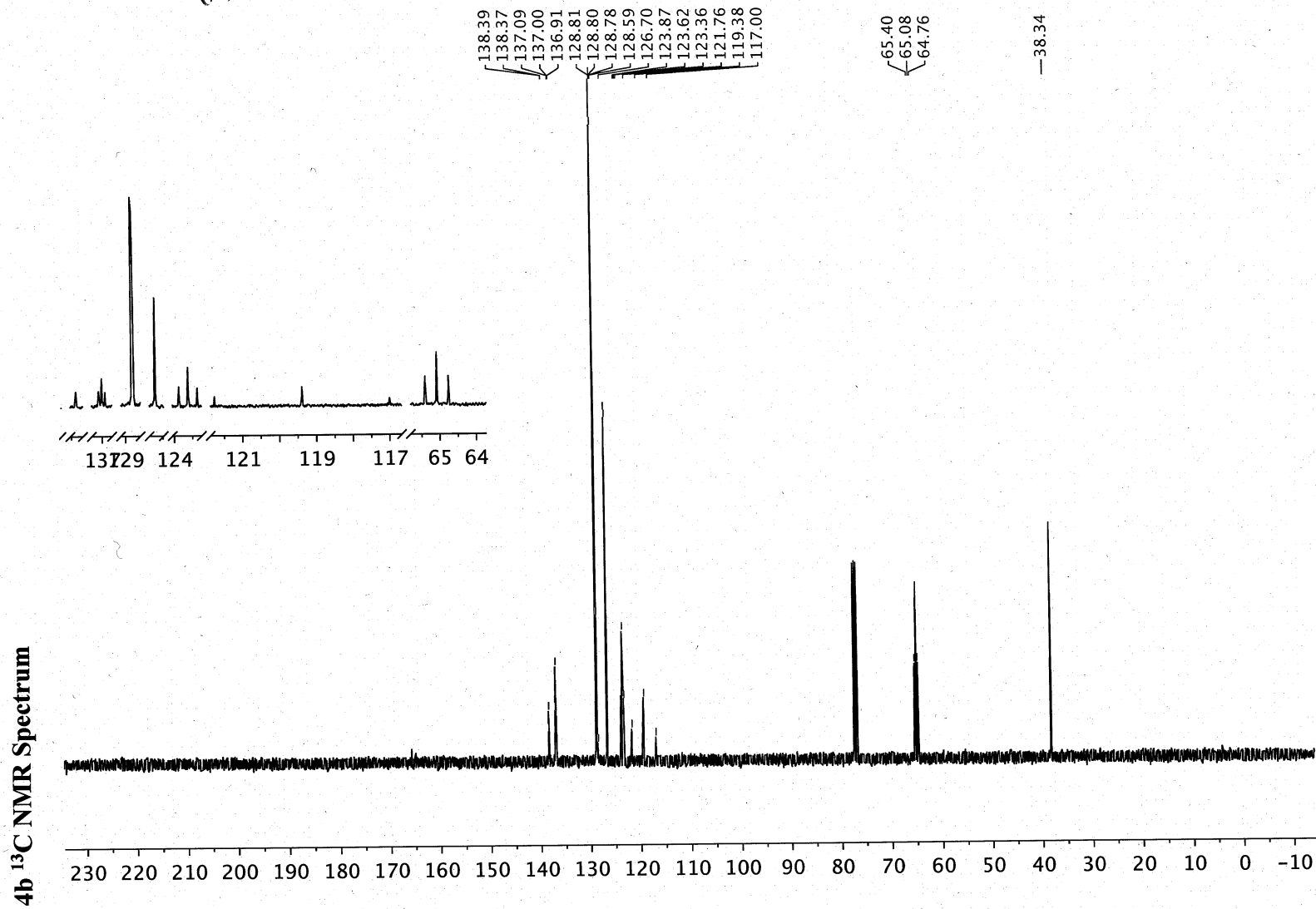
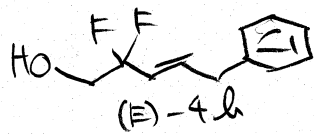


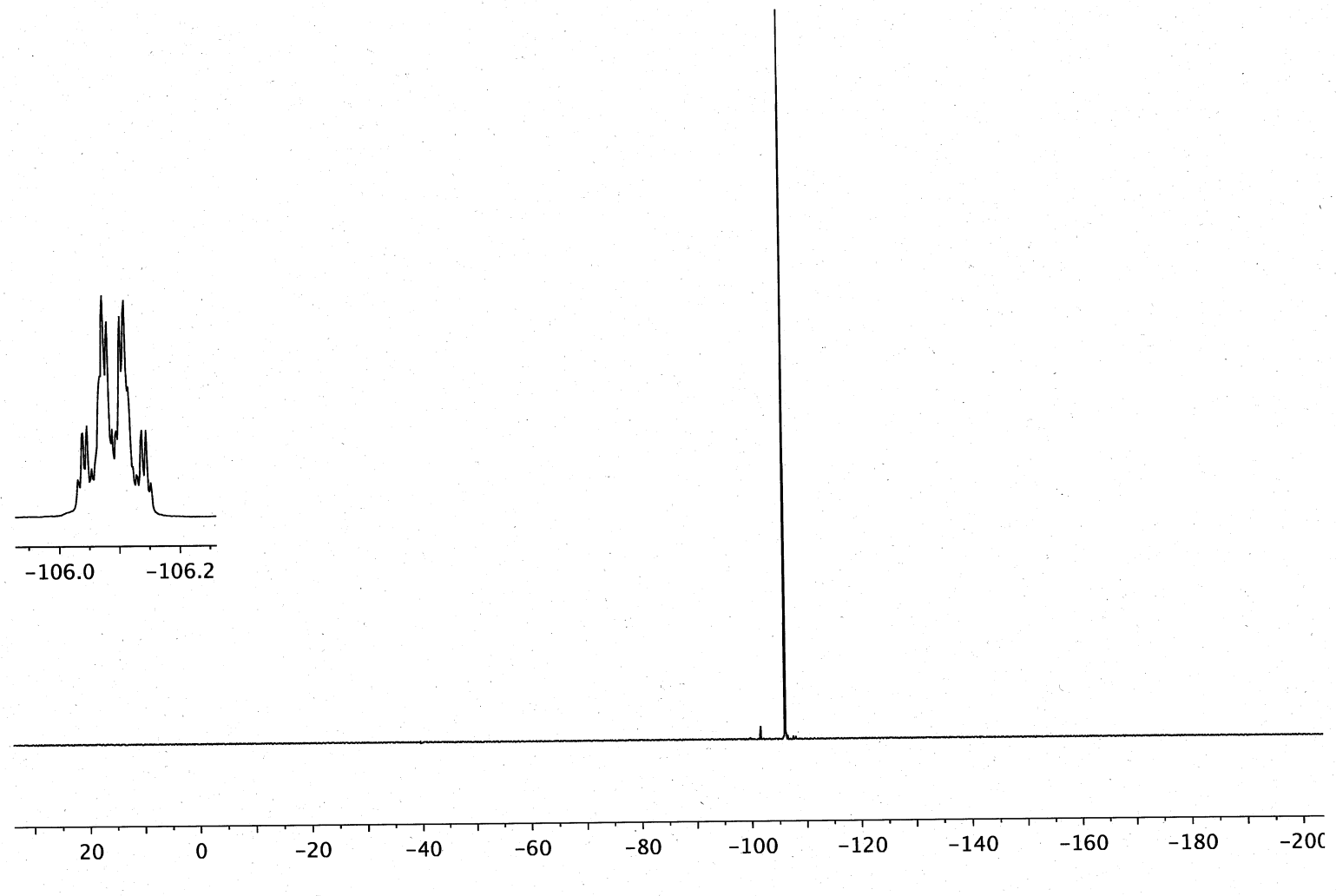
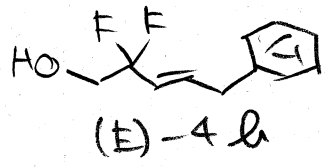
4a <sup>19</sup>F NMR Spectrum



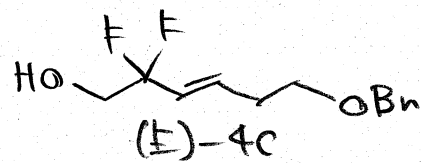




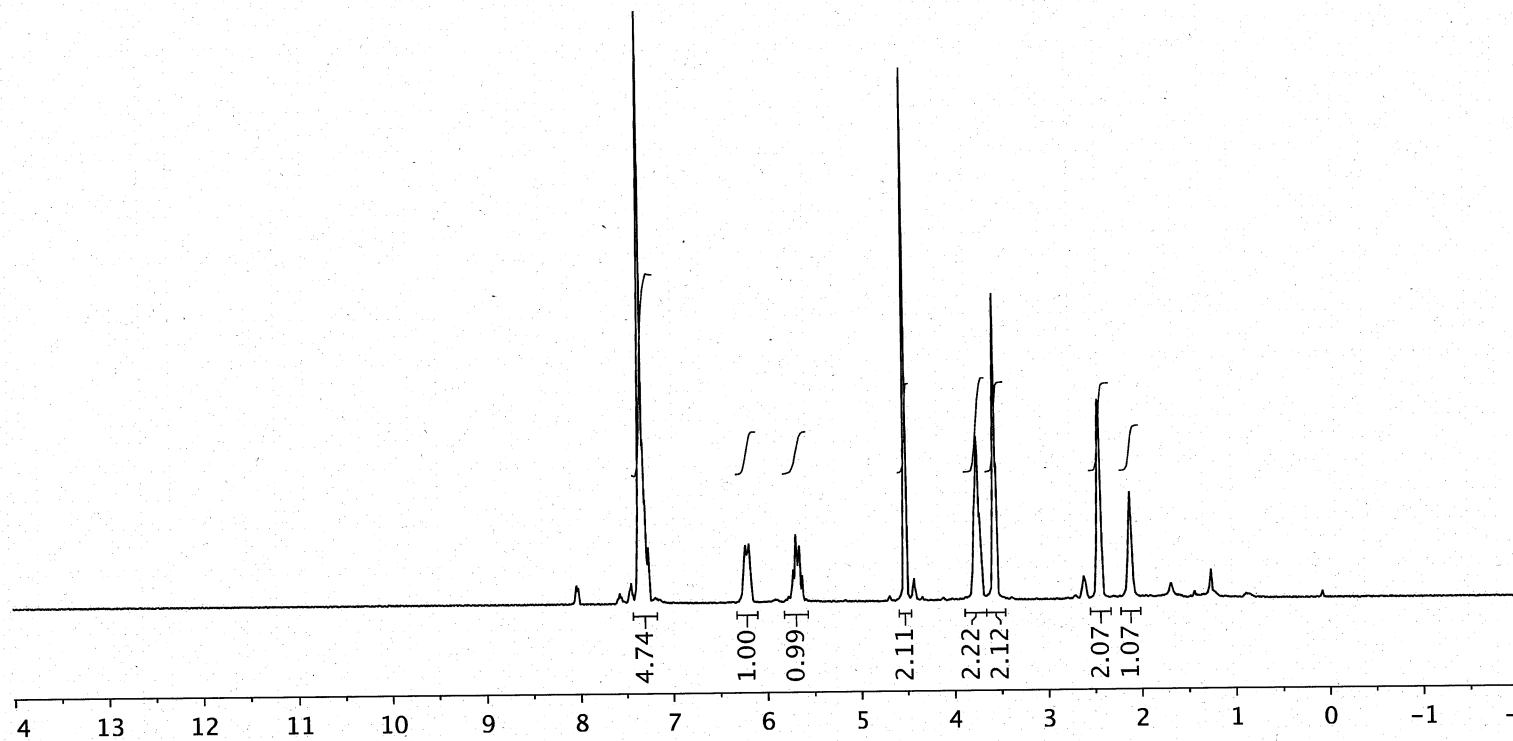


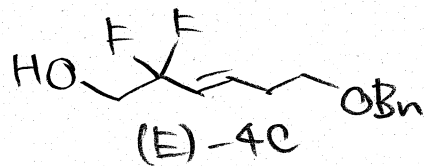


4b  $^{19}\text{F}$  NMR Spectrum



4c <sup>1</sup>H NMR Spectrum

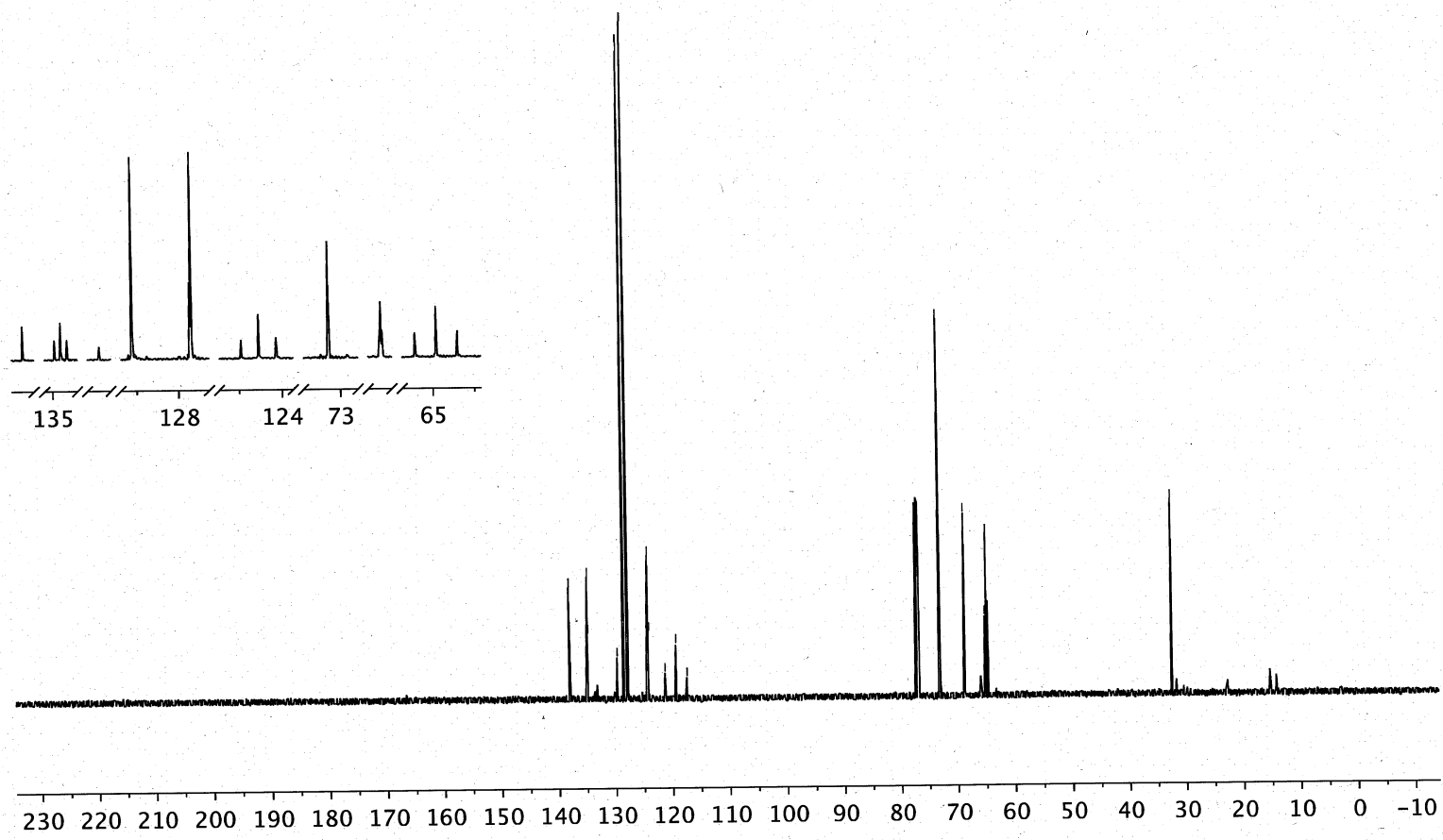




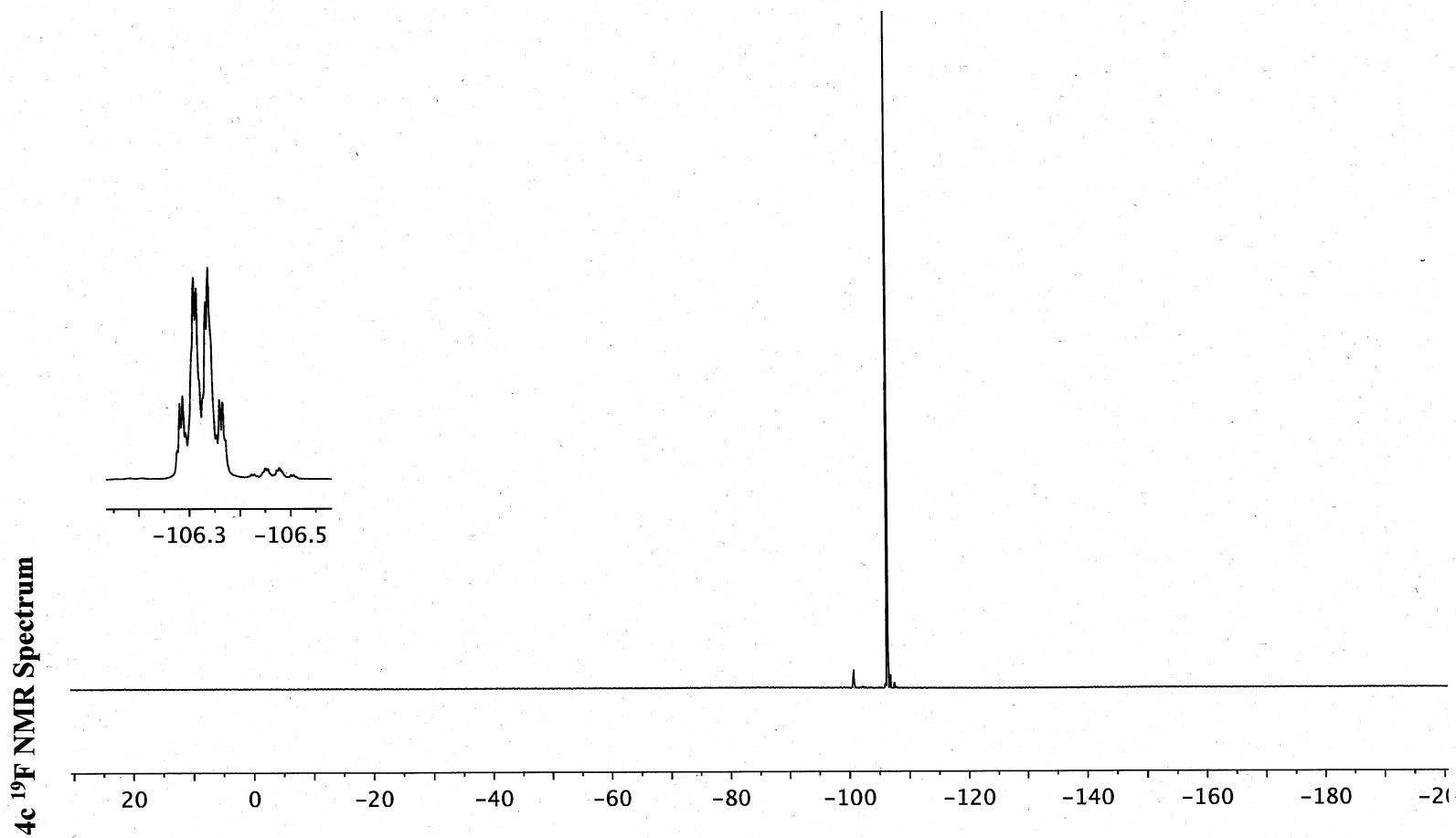
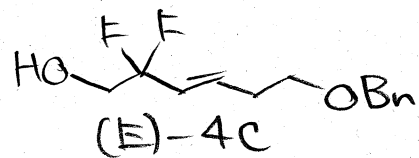
138.16  
 134.98  
 134.91  
 134.83  
 129.69  
 128.56  
 128.55  
 127.87  
 127.85  
 127.84  
 124.48  
 124.27  
 124.07  
 121.25  
 119.35  
 117.44

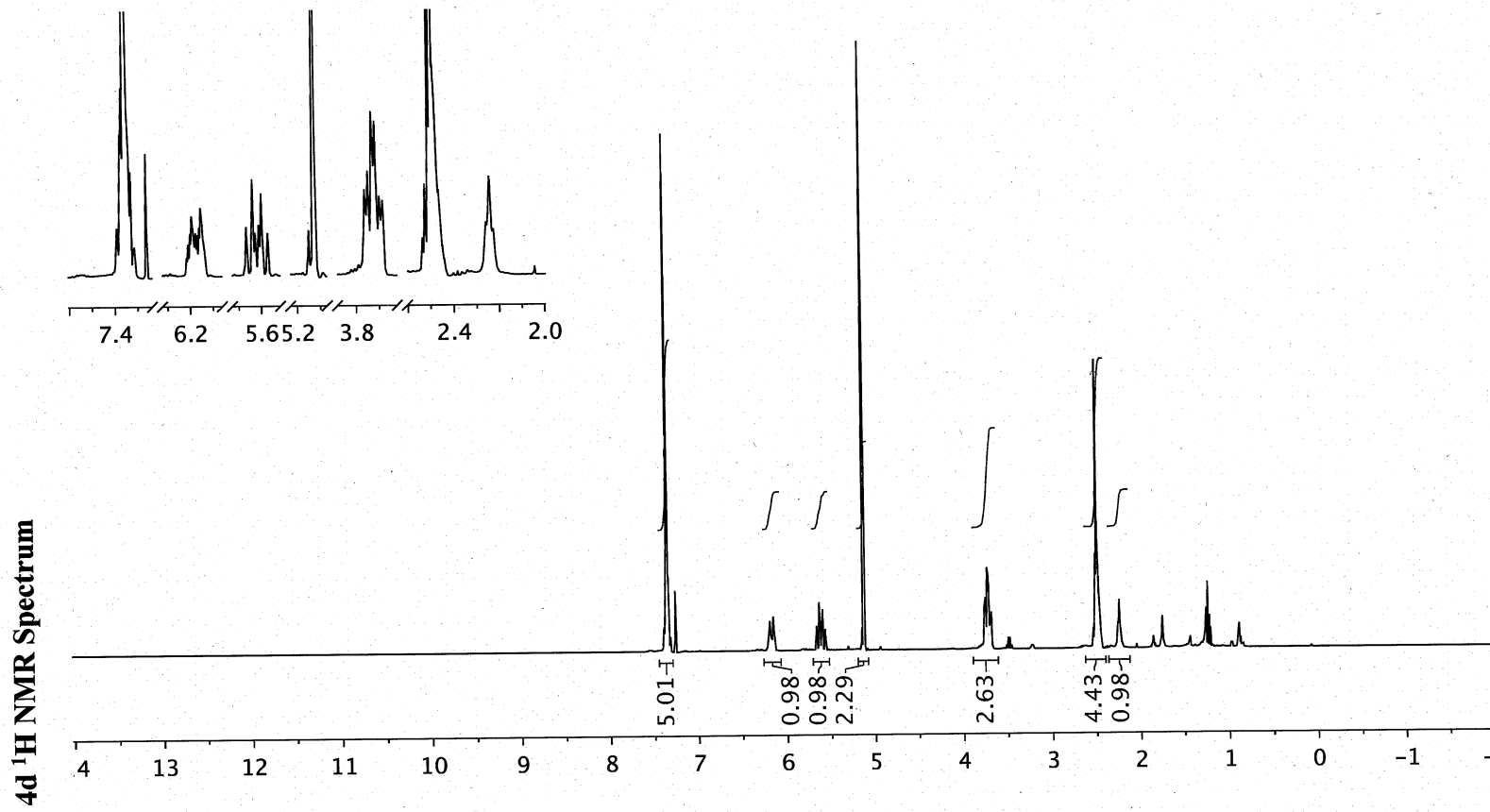
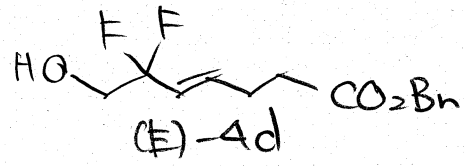
73.14  
 68.75  
 68.73  
 68.72  
 65.22  
 64.96  
 64.71

—32.52

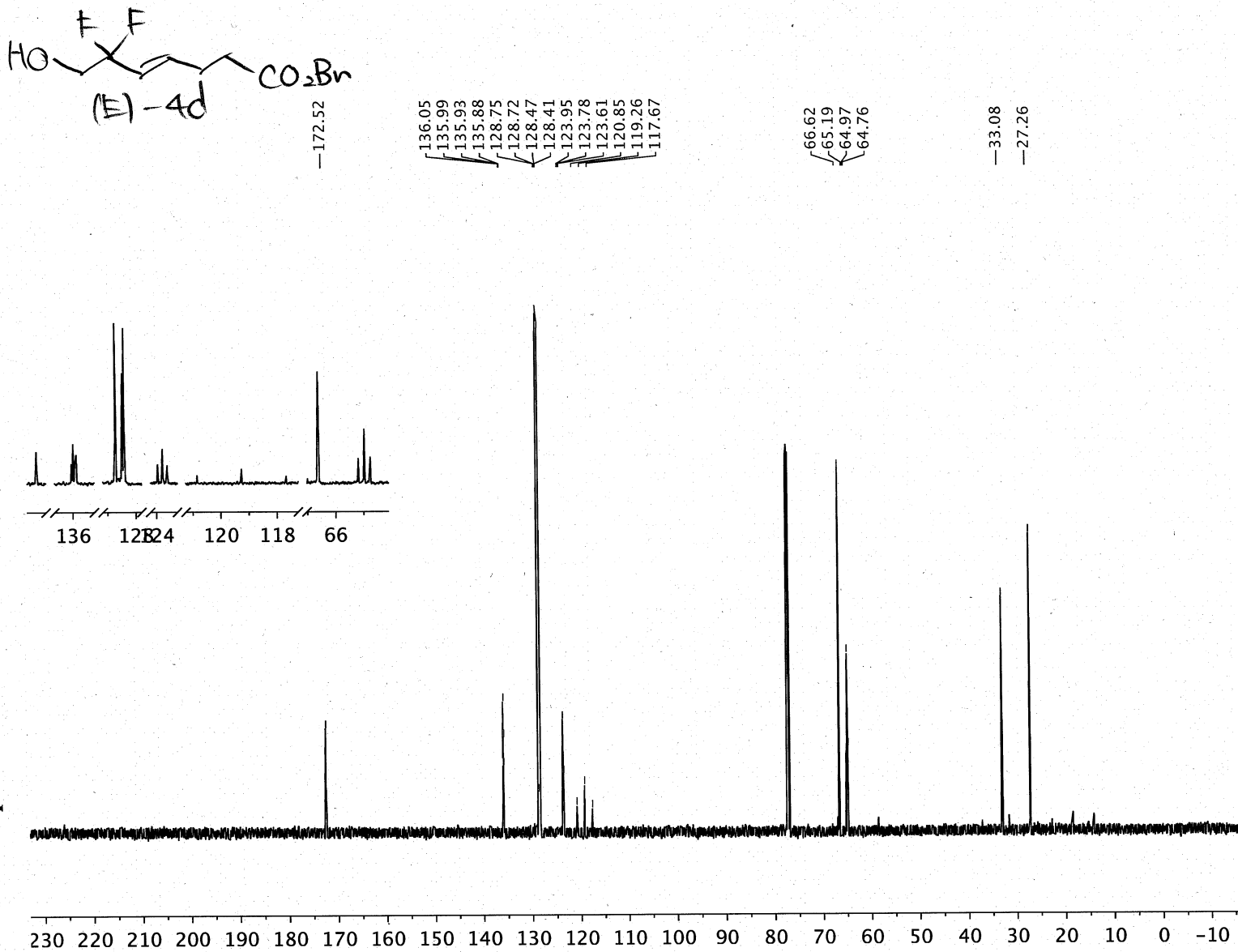


4c <sup>13</sup>C NMR Spectrum

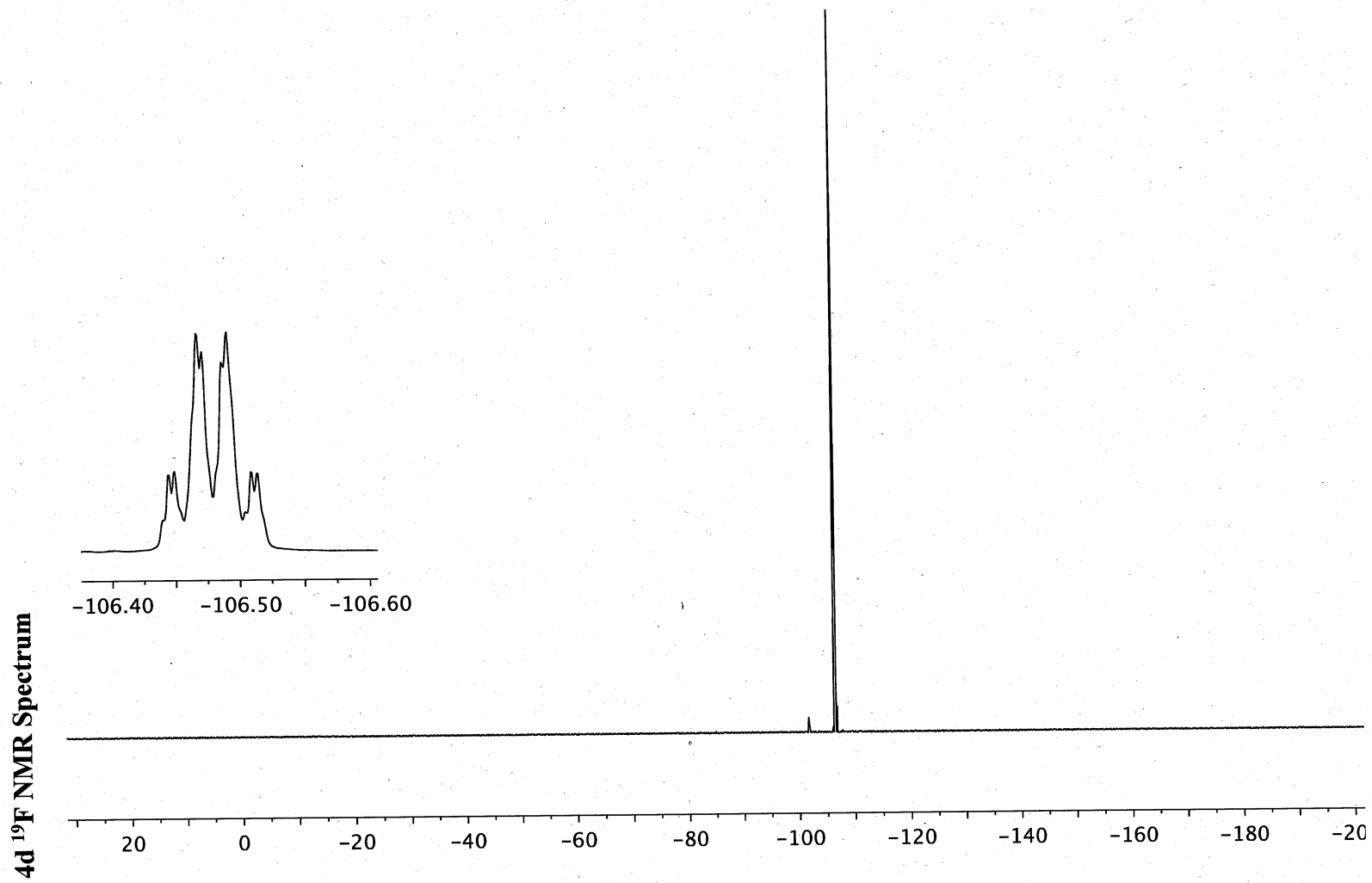
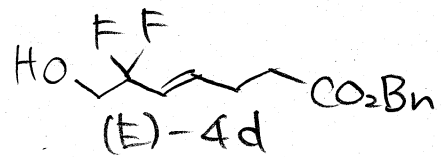




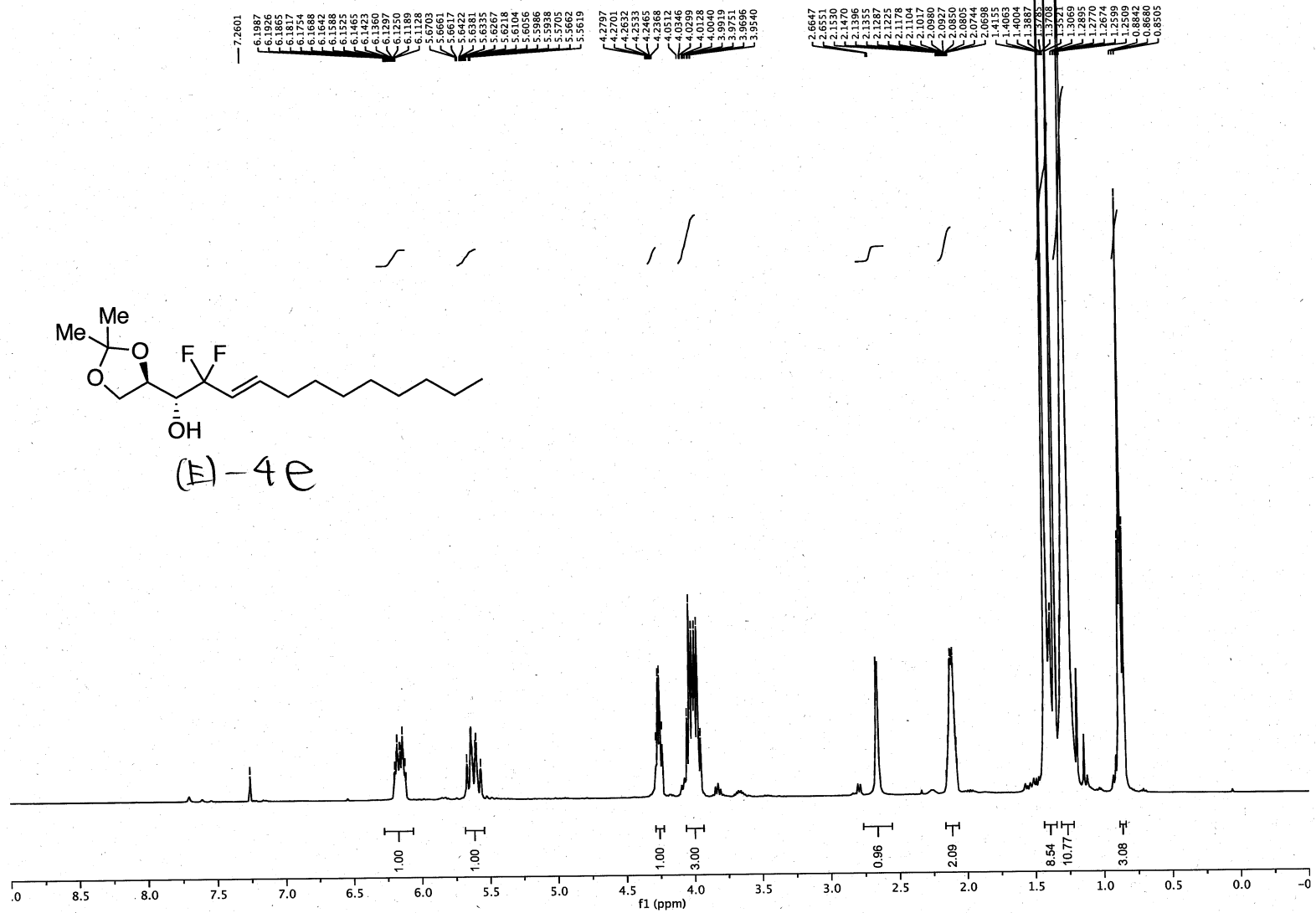
4d <sup>13</sup>C NMR Spectrum



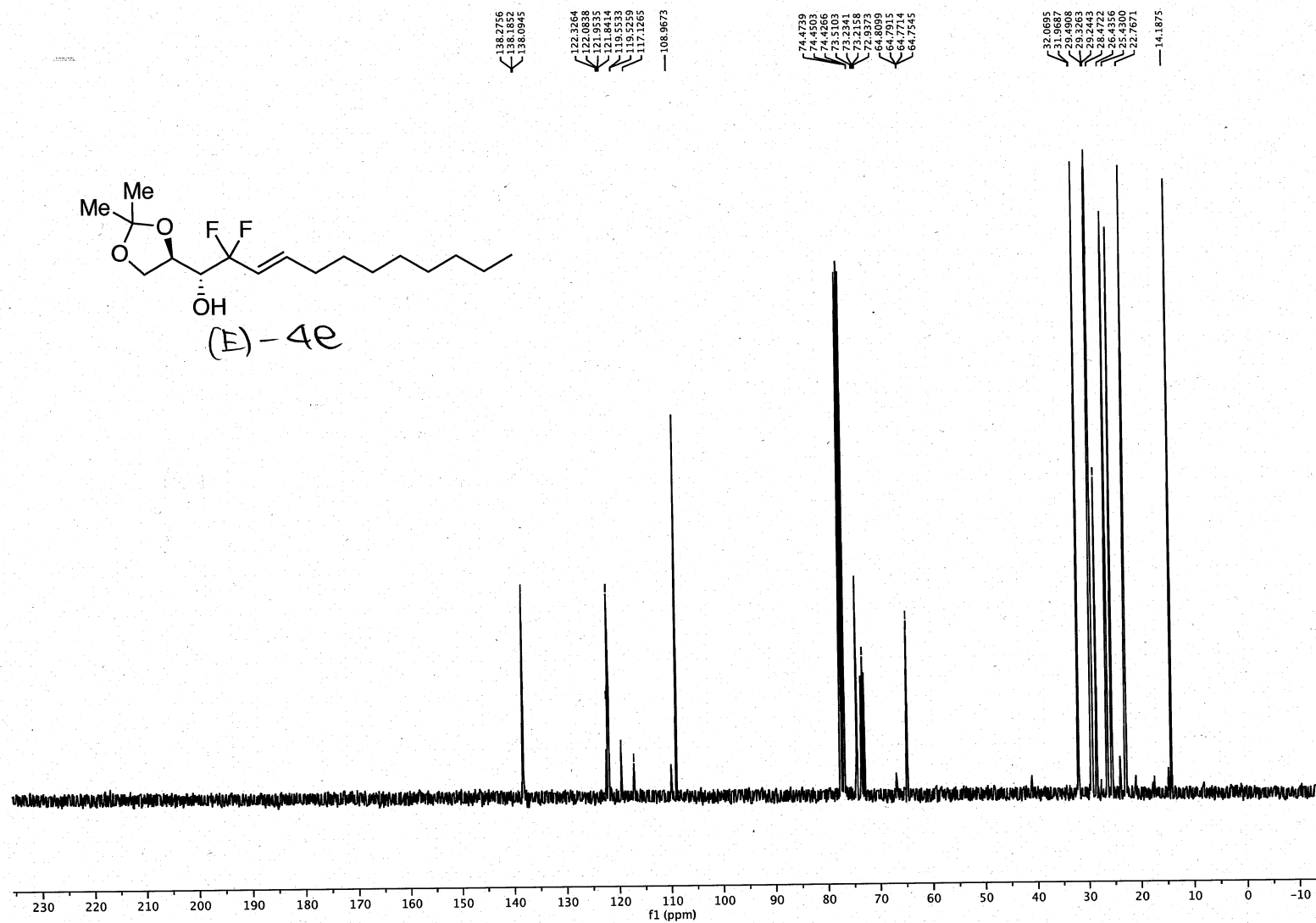




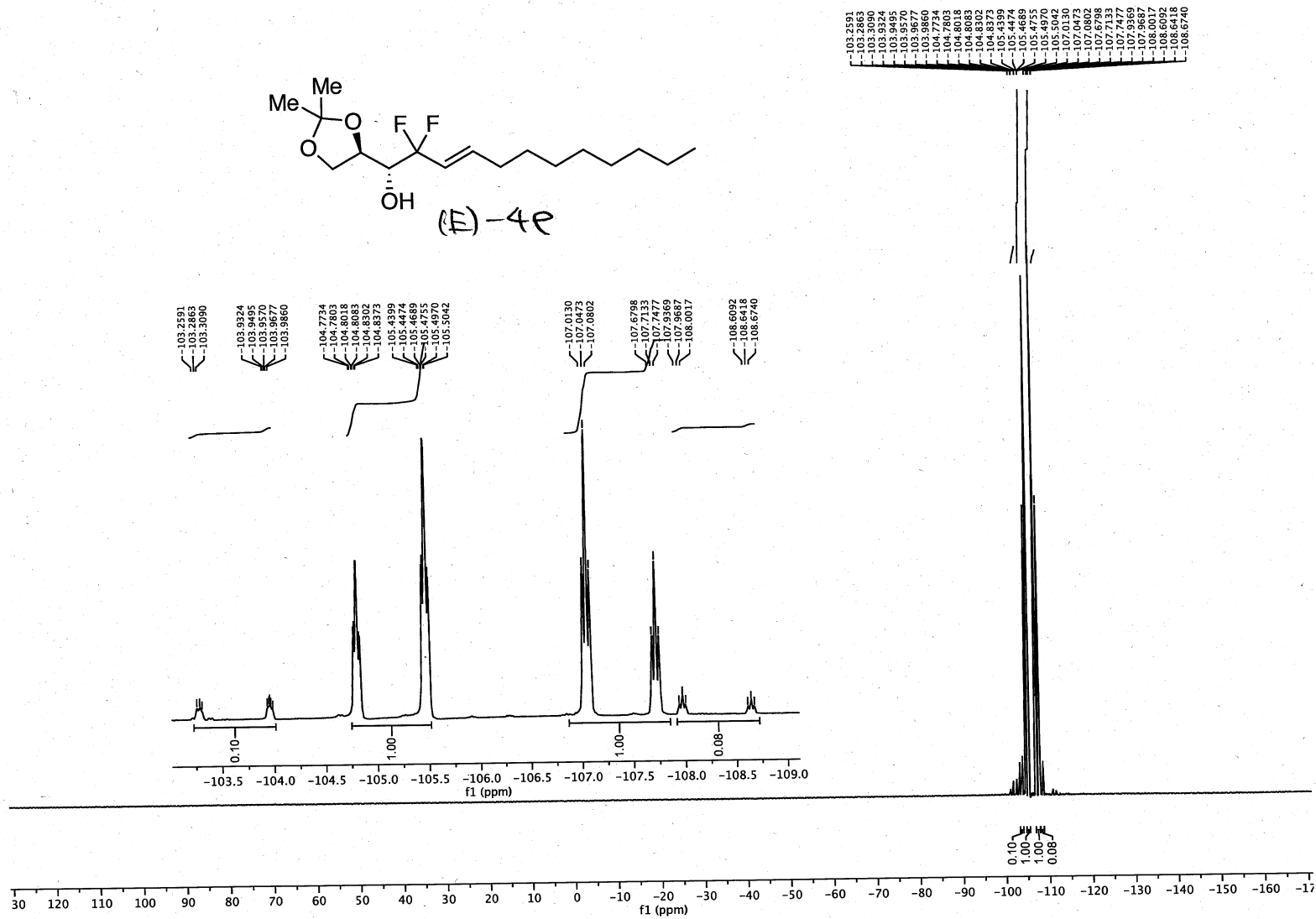
4e <sup>1</sup>H NMR Spectrum

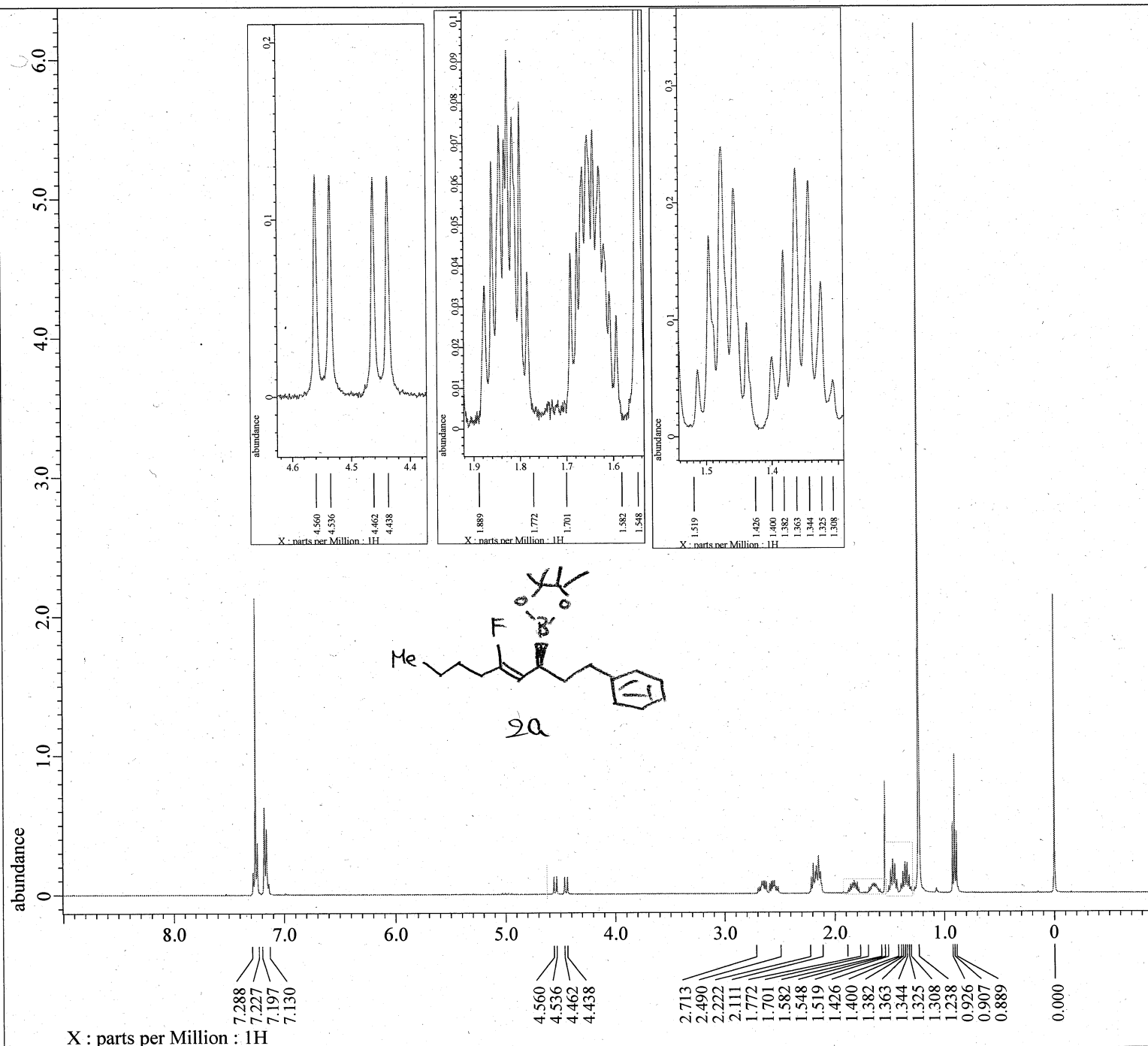


4e <sup>13</sup>C NMR Spectrum



4e <sup>19</sup>F NMR Spectrum



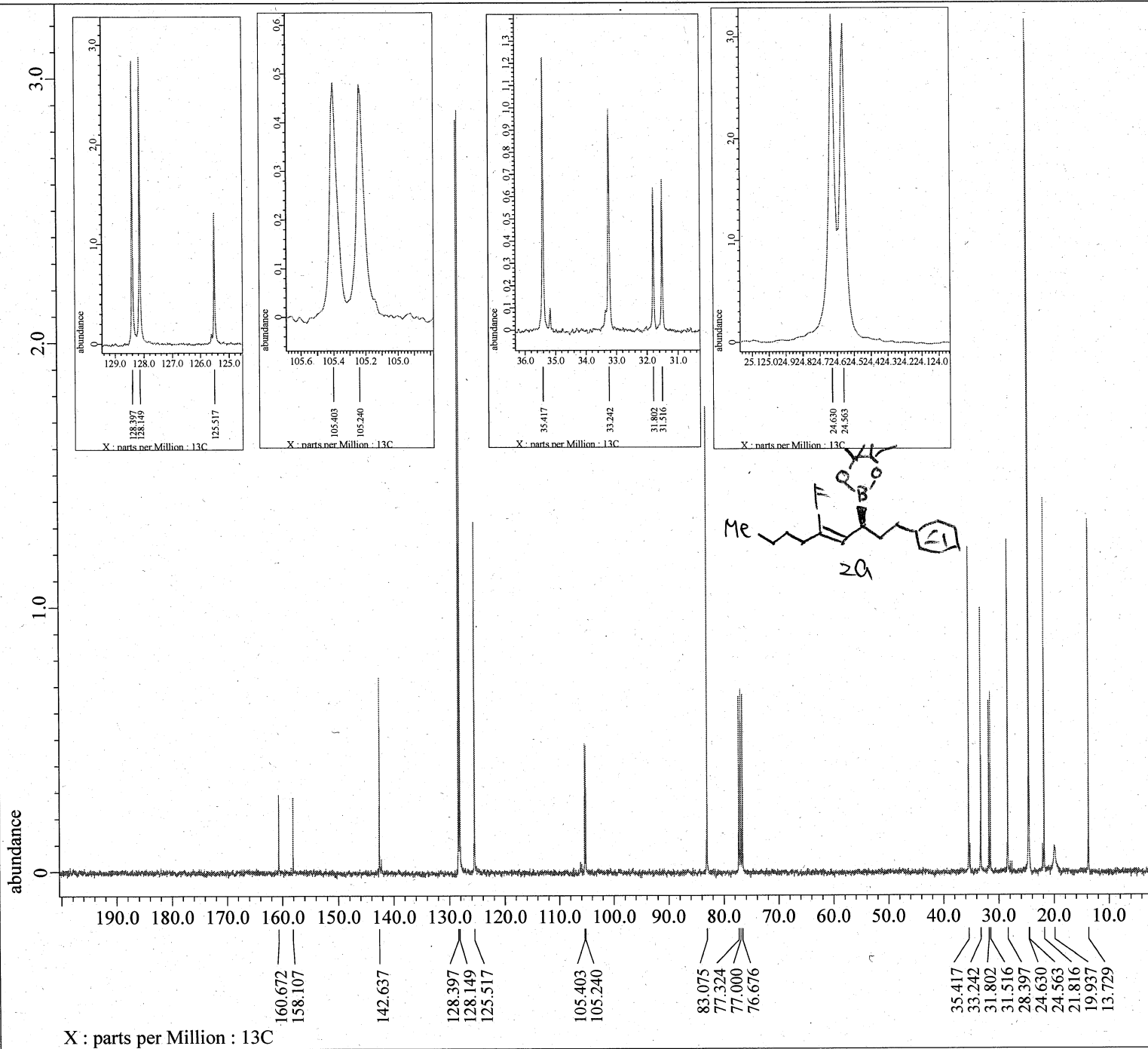


Filename = AKY719-pure-4.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#342377  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 8-NOV-2018 16:57:47  
Revision\_Time = 29-MAR-2019 20:25:14

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 2.228224[s]  
X Domain = 1H  
X\_Freq = 391.78655441[MHz]  
X\_Offset = 5[ppm]  
X Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.44878791[Hz]  
X\_Sweep = 7.35294118[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 1H  
Tri\_Freq = 391.78655441[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 50  
Temp Get = 20.5[dc]  
X 90\_Width = 11.04[us]  
X\_Acq\_Time = 2.228224[s]  
X\_Angle = 45[deg]  
X\_Atn = 1.9[db]  
X\_Pulse = 5.52[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.228224[s]

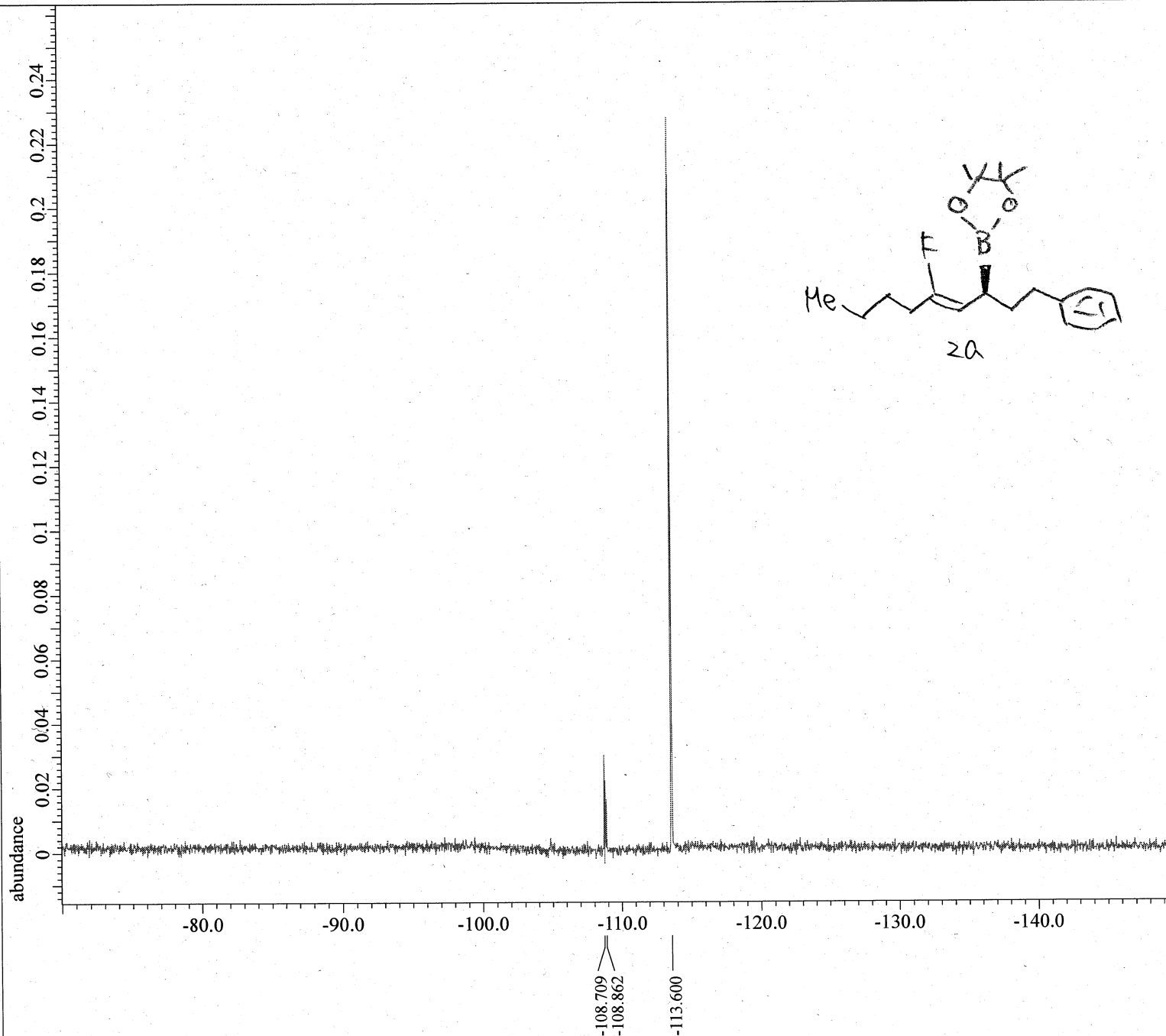


Filename = AKY719-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = S#346250  
Solvent = CHLOROFORM-D  
Actual Start Time = 8-NOV-2018 17:03:21  
Revision Time = 8-NOV-2018 13:14:14

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 1.06430464[s]  
X Domain = 13C  
X Freq = 98.51479726[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061[Hz]  
X Sweep = 30.78817734[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 60  
Temp Get = 20.9[dC]  
X 90 Width = 9.11[us]  
X Acq Time = 1.06430464[s]  
X Angle = 30[deg]  
X Atn = 4.9[dB]  
X Pulse = 3.03666667[us]  
Irr Atn Dec = 22.255[dB]  
Irr Atn Noe = 22.255[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.06430464[s]



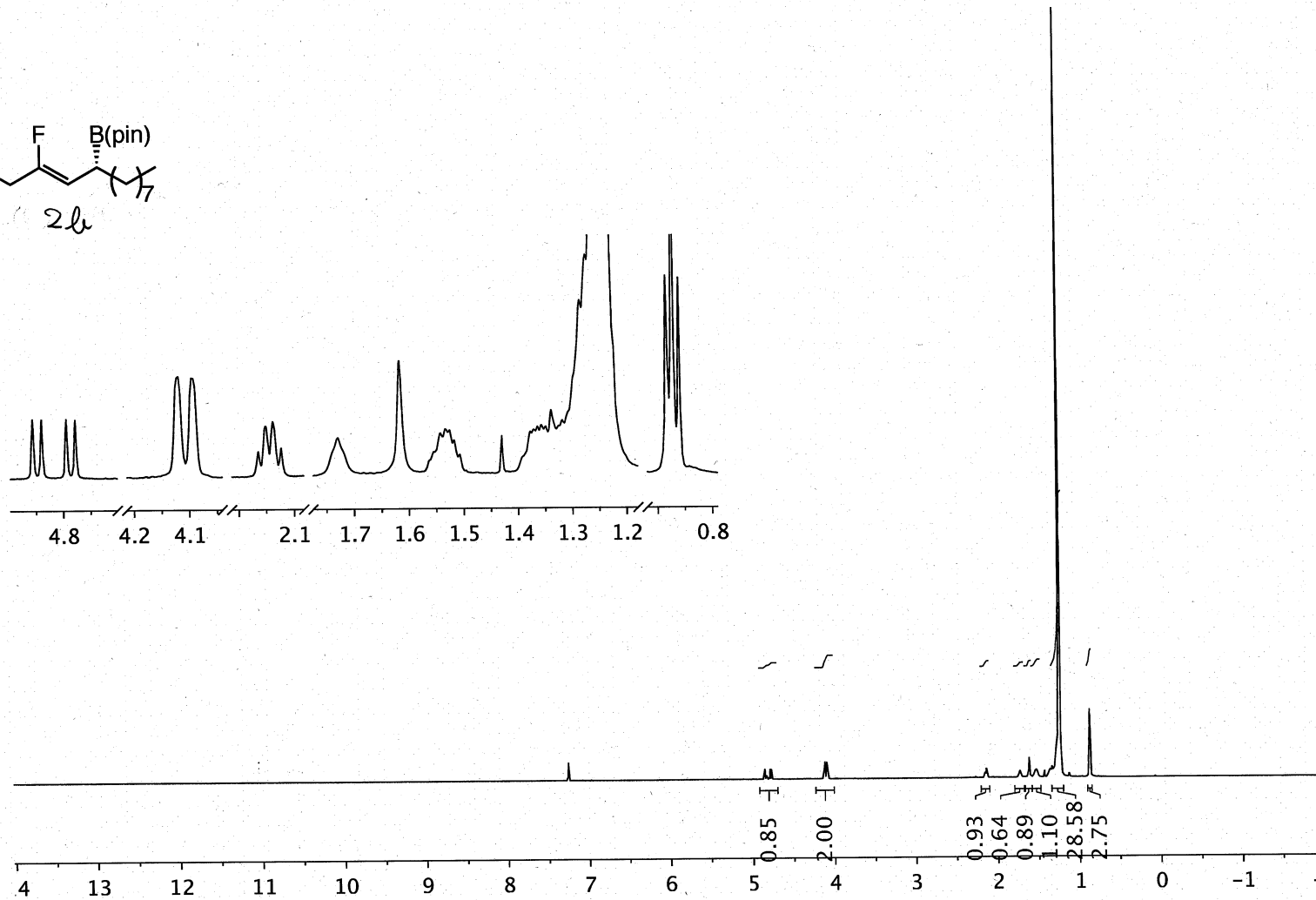
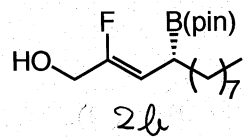
Filename = AKY719-fnmr-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#361400  
Solvent = CHLOROFORM-D  
Actual Start Time = 8-NOV-2018 18:31:57  
RevisiOn Time = 22-MAR-2019 18:09:40

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

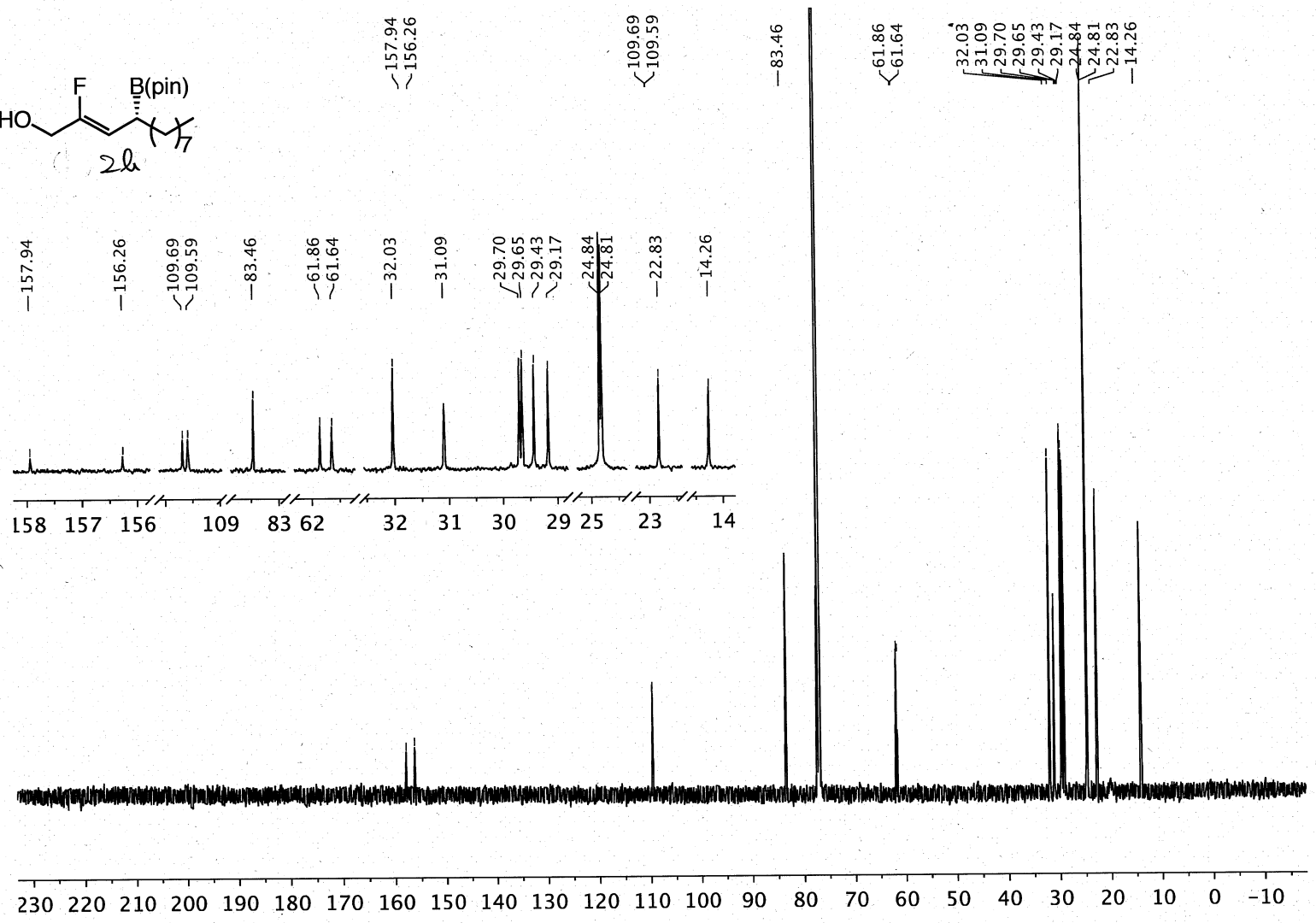
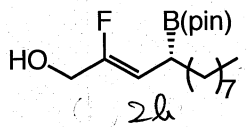
Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

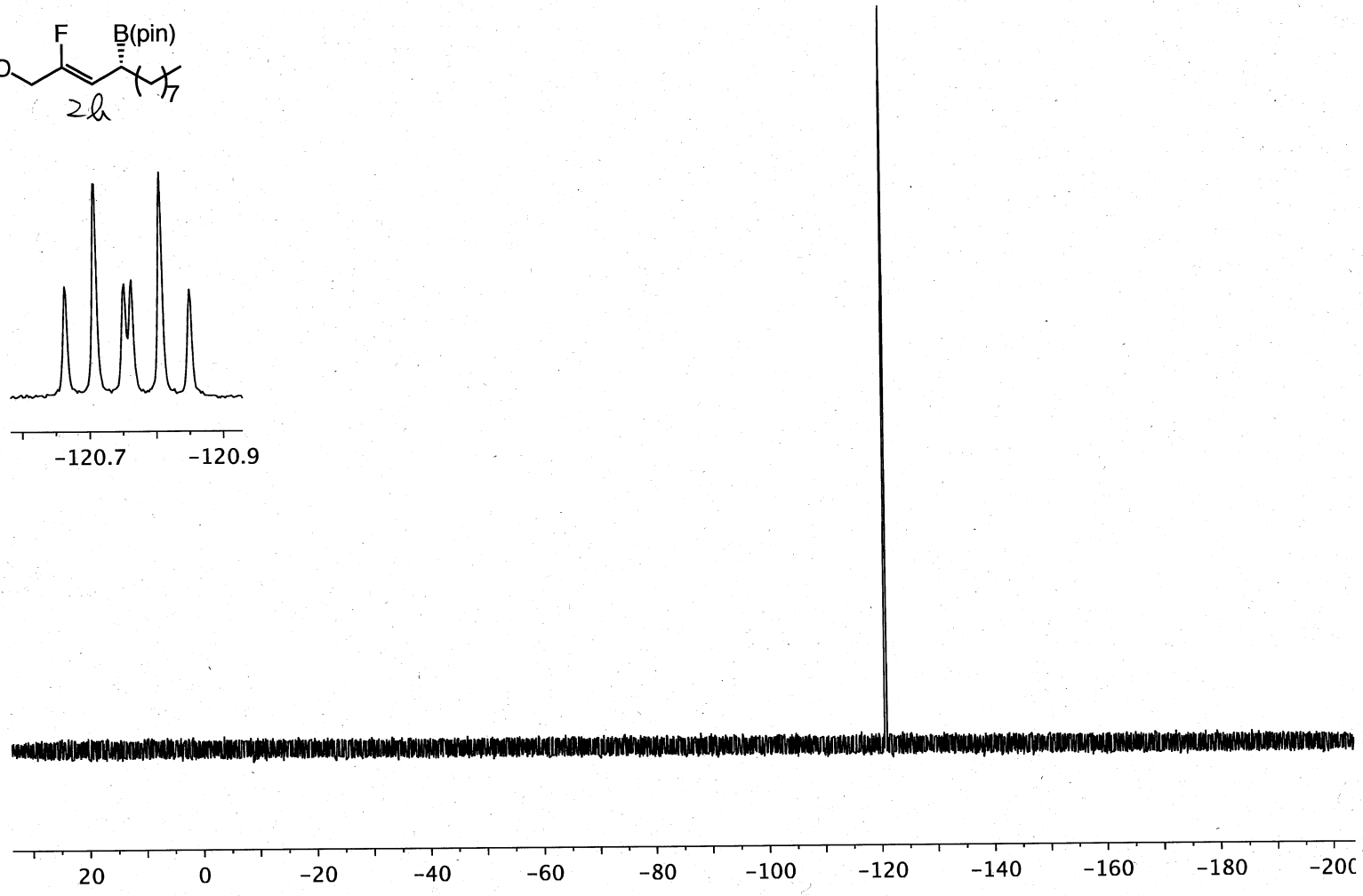
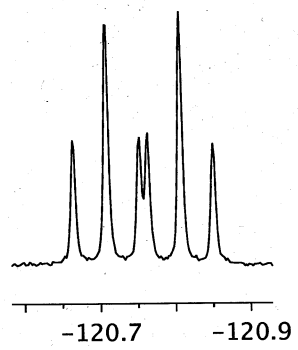
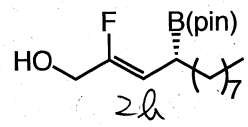
Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 21.8[dC]  
X\_90\_Width = 13.9[us]  
X Acq Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]

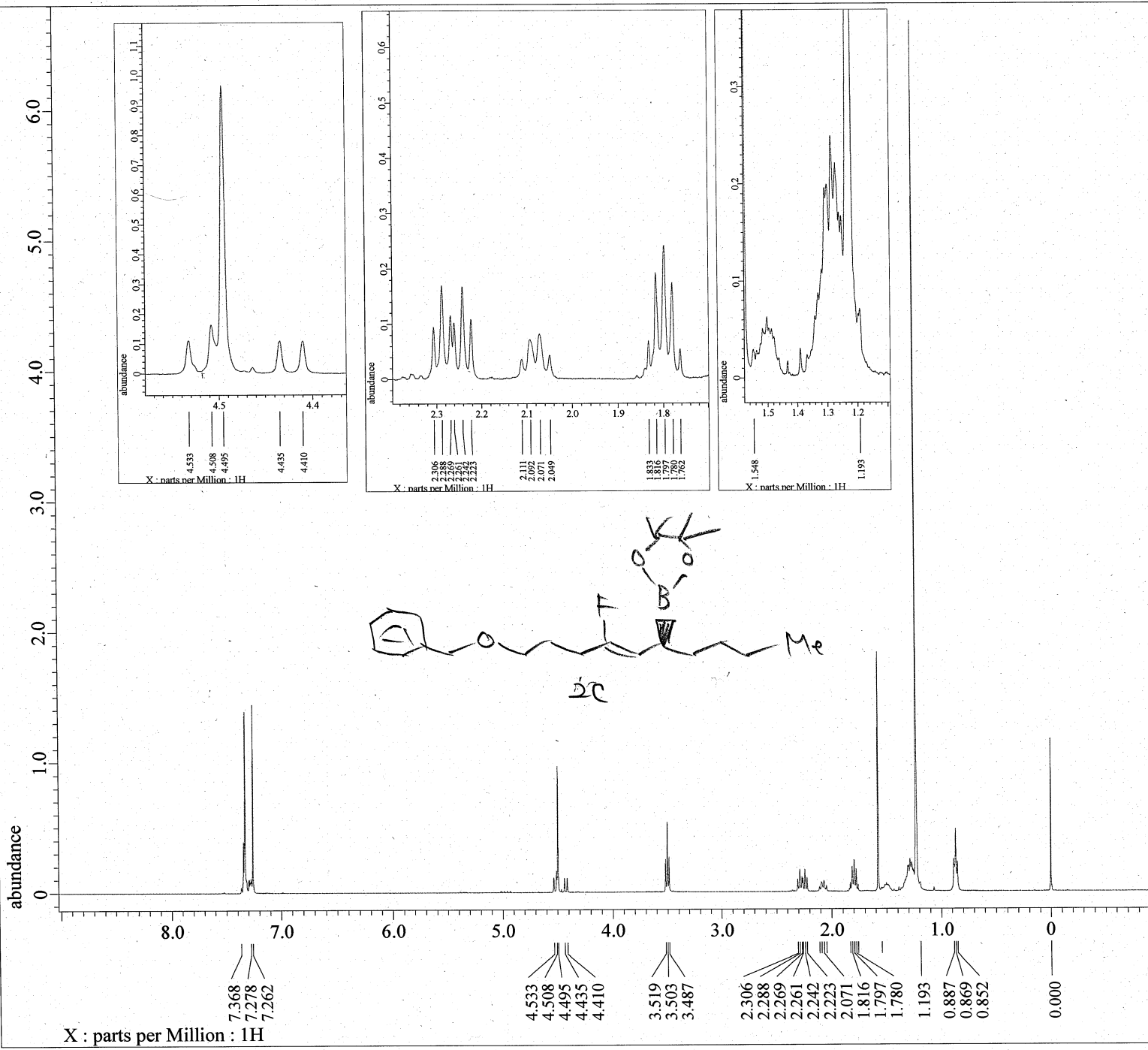
X : parts per Million : 19F









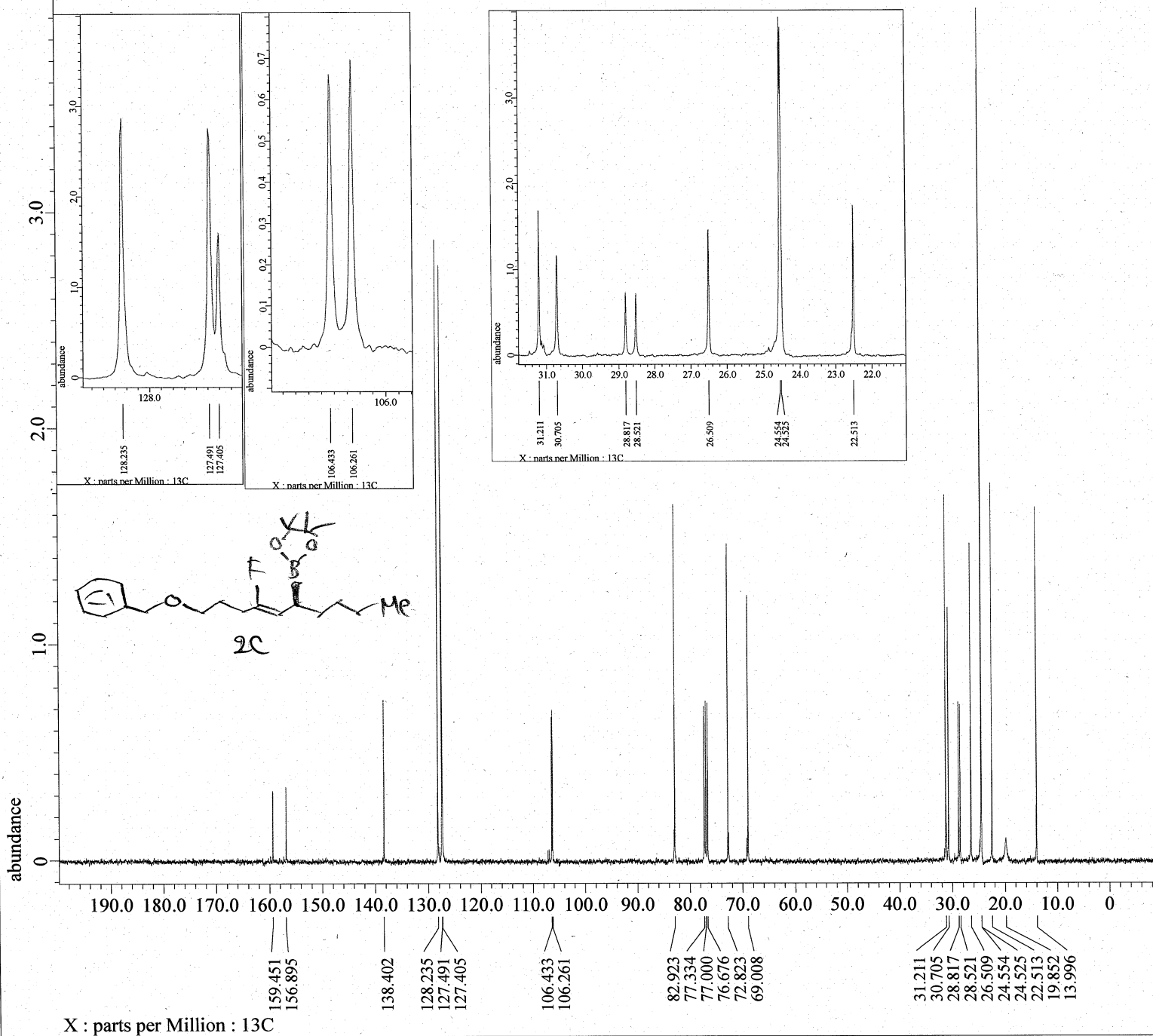


Filename = AKY686-pure-3.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#470146  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 11-OCT-2018 20:34:57  
Revision\_Time = 23-MAR-2019 13:17:03

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390 [MHz])  
X Acq\_Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441 [MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791 [Hz]  
X Sweep = 7.35294118 [kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441 [MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441 [MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

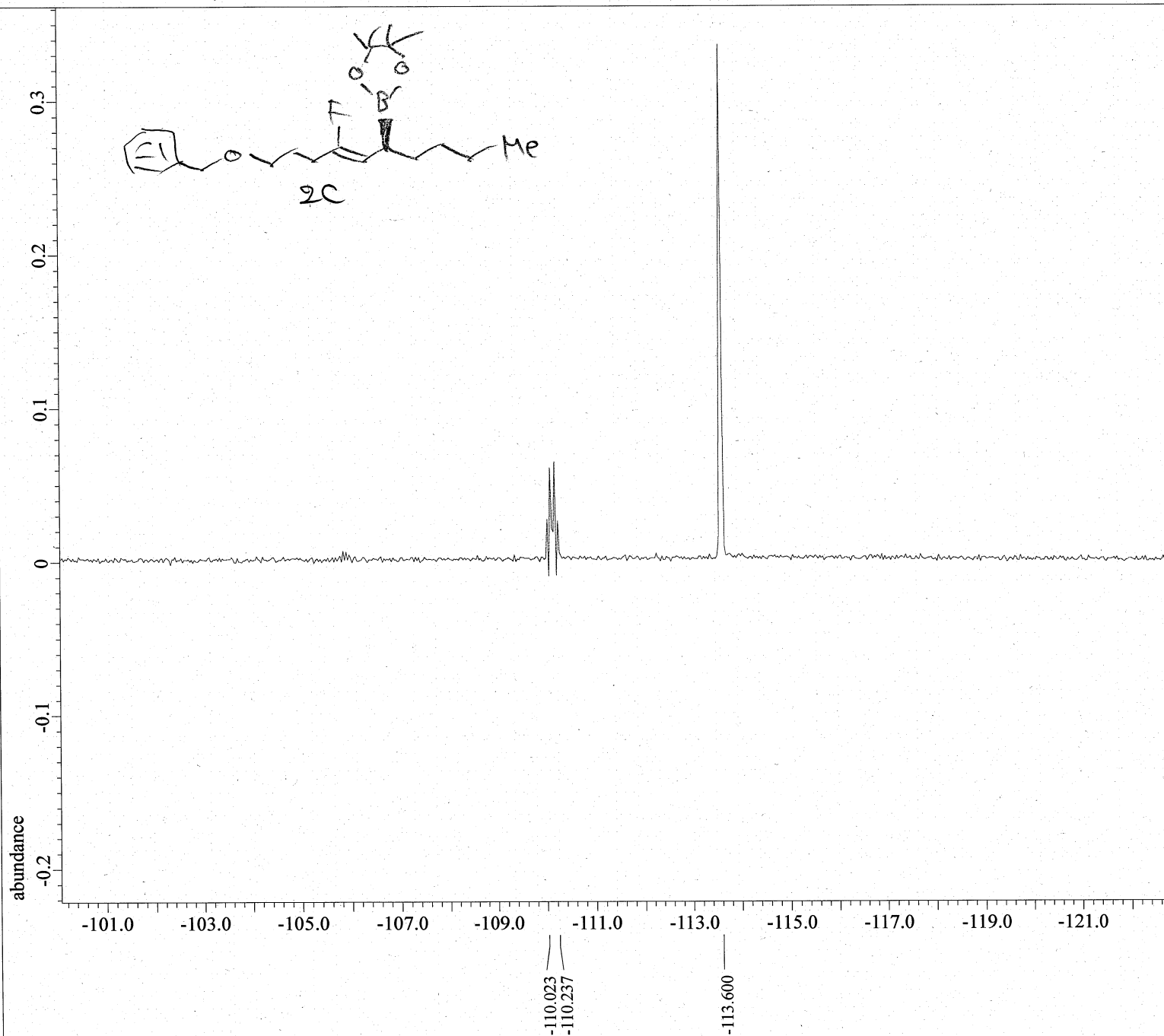
Relaxation\_Delay = 5[s]  
Recvr Gain = 44  
Temp Get = 19.3[dC]  
X 90\_Width = 11.04[us]  
X Acq\_Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.228224[s]



Filename = AKY686-carbon-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#474677  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 11-OCT-2018 20:41:21  
Revision\_Time = 23-MAR-2019 13:19:58

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
X Freq = 98.51479726 [MHz]  
X Offset = 100 [ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061 [Hz]  
X Sweep = 30.78817734 [kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441 [MHz]  
Irr Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation Delay = 2 [s]  
Recvr Gain = 60  
Temp Get = 19.6 [dC]  
X 90 Width = 9.11 [us]  
X Acq Time = 1.06430464 [s]  
X Angle = 30 [deg]  
X Atn = 4.9 [dB]  
X Pulse = 3.03666667 [us]  
Irr Atn Dec = 22.255 [dB]  
Irr Atn Noe = 22.255 [dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1 [s]  
Noe = TRUE  
Noe\_Time = 2 [s]  
Repetition Time = 3.06430464 [s]



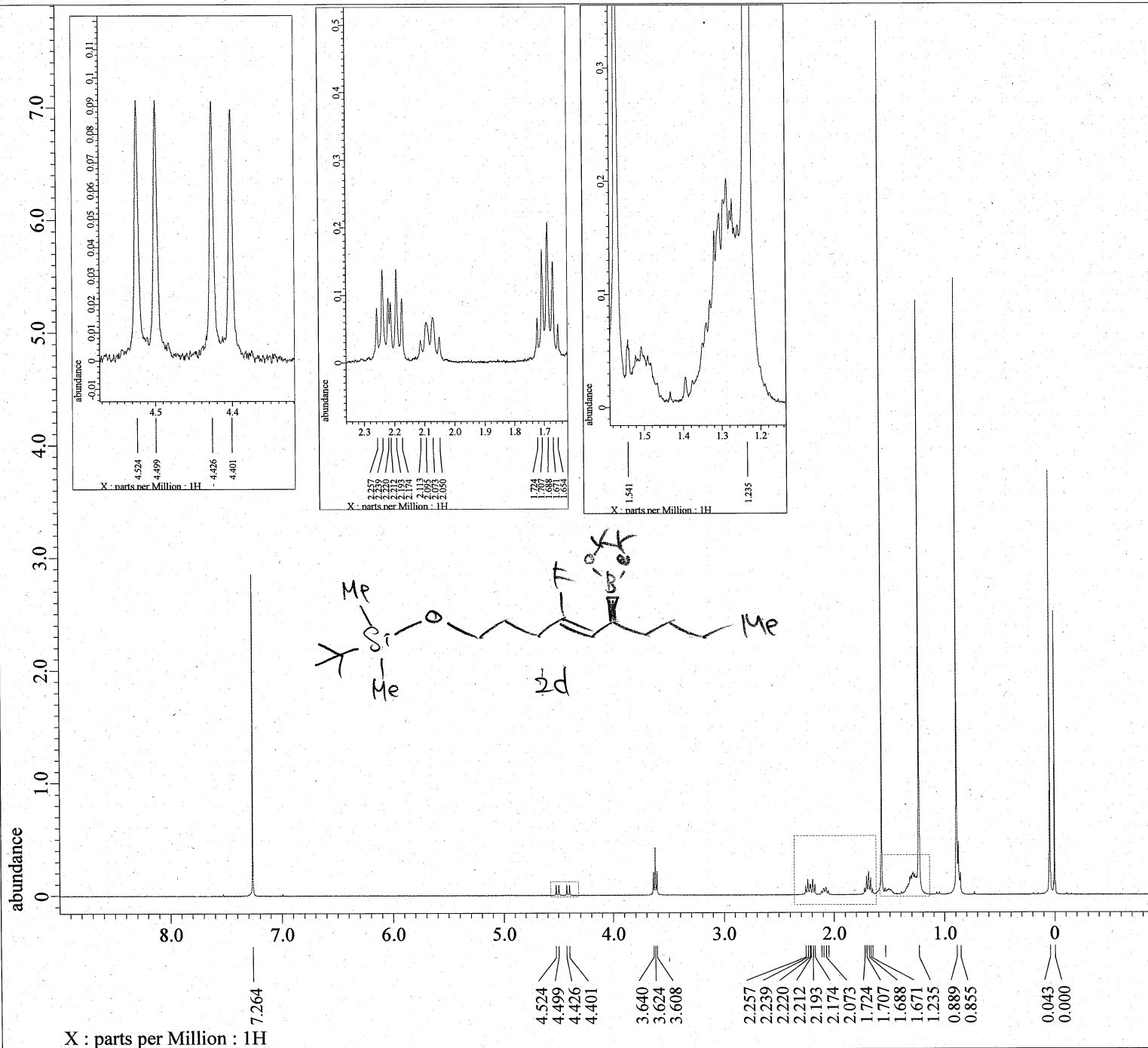
Filename = AKY686-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#506480  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 11-OCT-2018 22:33:45  
Revision\_Time = 23-MAR-2019 13:25:07

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 22.9[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[db]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]

X : parts per Million : 19F

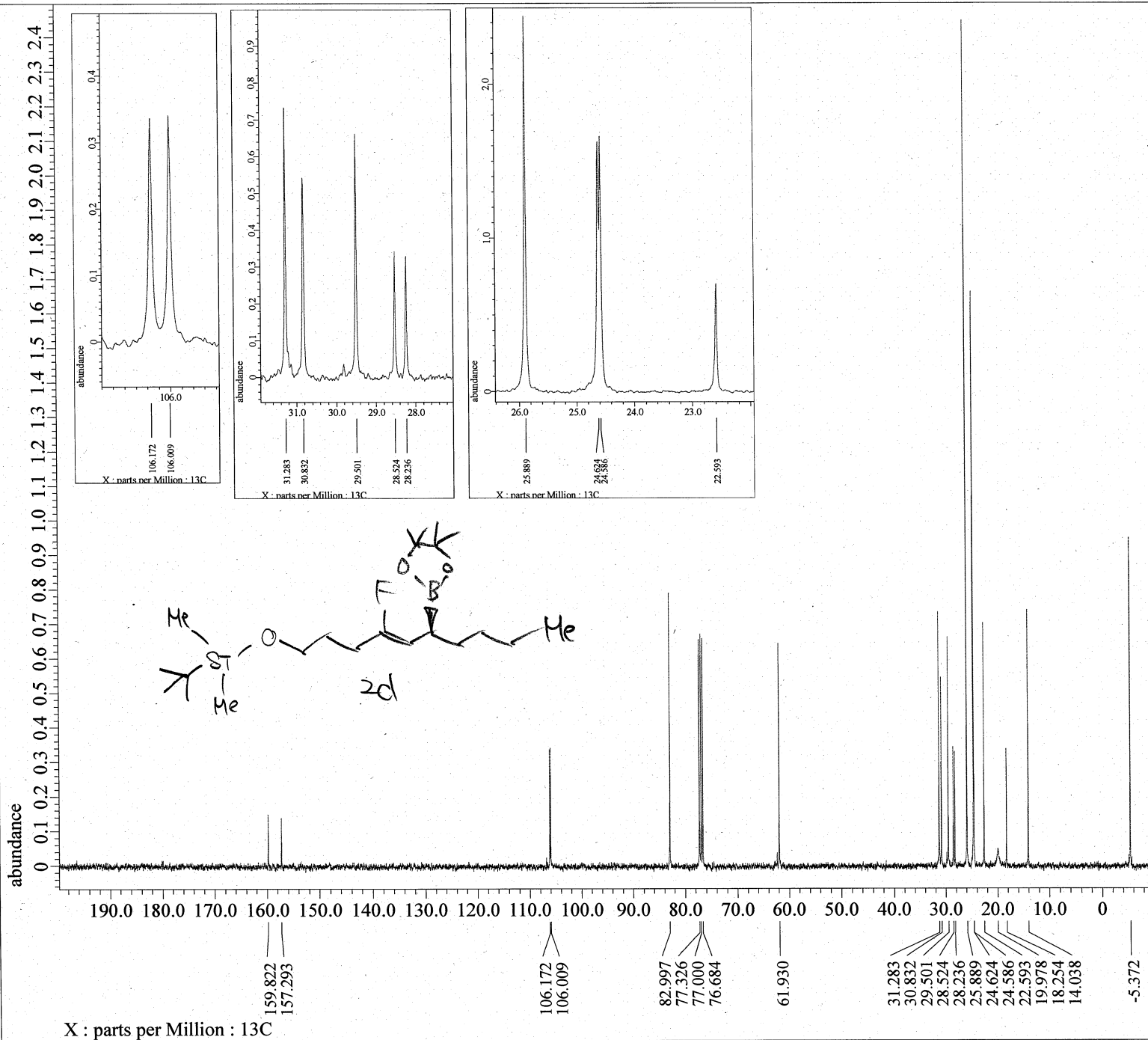


Filename = AKY687-pure-ag-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#400104  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 11-OCT-2018 18:37:59  
Revision\_Time = 23-MAR-2019 14:09:46

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X\_Acq\_Duration = 2.228224[s]  
X\_Domain = 1H  
X\_Freq = 391.78655441[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.44878791[Hz]  
X\_Sweep = 7.35294118[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 1H  
Tri\_Freq = 391.78655441[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 50  
Temp\_Get = 19.6[dC]  
X\_90\_Width = 11.04[us]  
X\_Acq\_Time = 2.228224[s]  
X\_Angle = 45[deg]  
X\_Atn = 1.9[dB]  
X\_Pulse = 5.52[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
DanTe\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.228224[s]

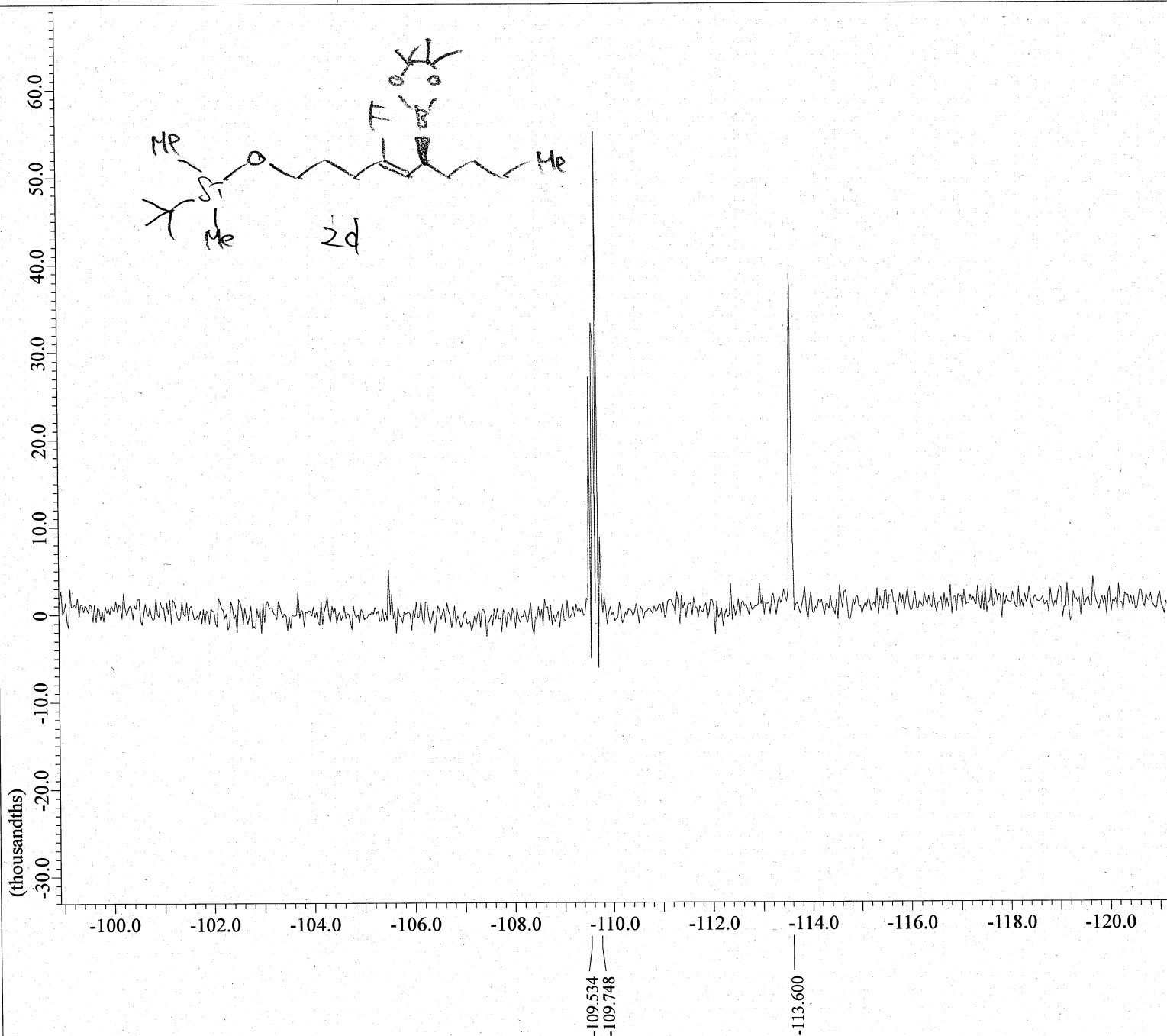


Filename = AKY687-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = 2  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 11-OCT-2018 19:00:49  
Revision\_Time = 23-MAR-2019 14:11:21

Comment = single pulse decoupled ga  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X\_Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 1.048576[s]  
X\_Domain = 13C  
X\_Freq = 99.54517646[MHz]  
X\_Offset = 100[ppm]  
X\_Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.95367432[Hz]  
X\_Sweep = 31.25[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 395.88430144[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 56  
Temp\_Get = 21.6[dc]  
X\_90\_Width = 10.1[us]  
X\_Acq\_Time = 1.048576[s]  
X\_Angle = 30[deg]  
X\_Atn = 3.4[dB]  
X\_Pulse = 3.36666667[us]  
Irr\_Atn\_Dec = 22.3[dB]  
Irr\_Atn\_Noise = 22.3[dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.048576[s]



Filename = AKY687-FNMR--ag-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#423852  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 11-OCT-2018 20:16:02  
Revision\_Time = 23-MAR-2019 14:14:04

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 20.8[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F



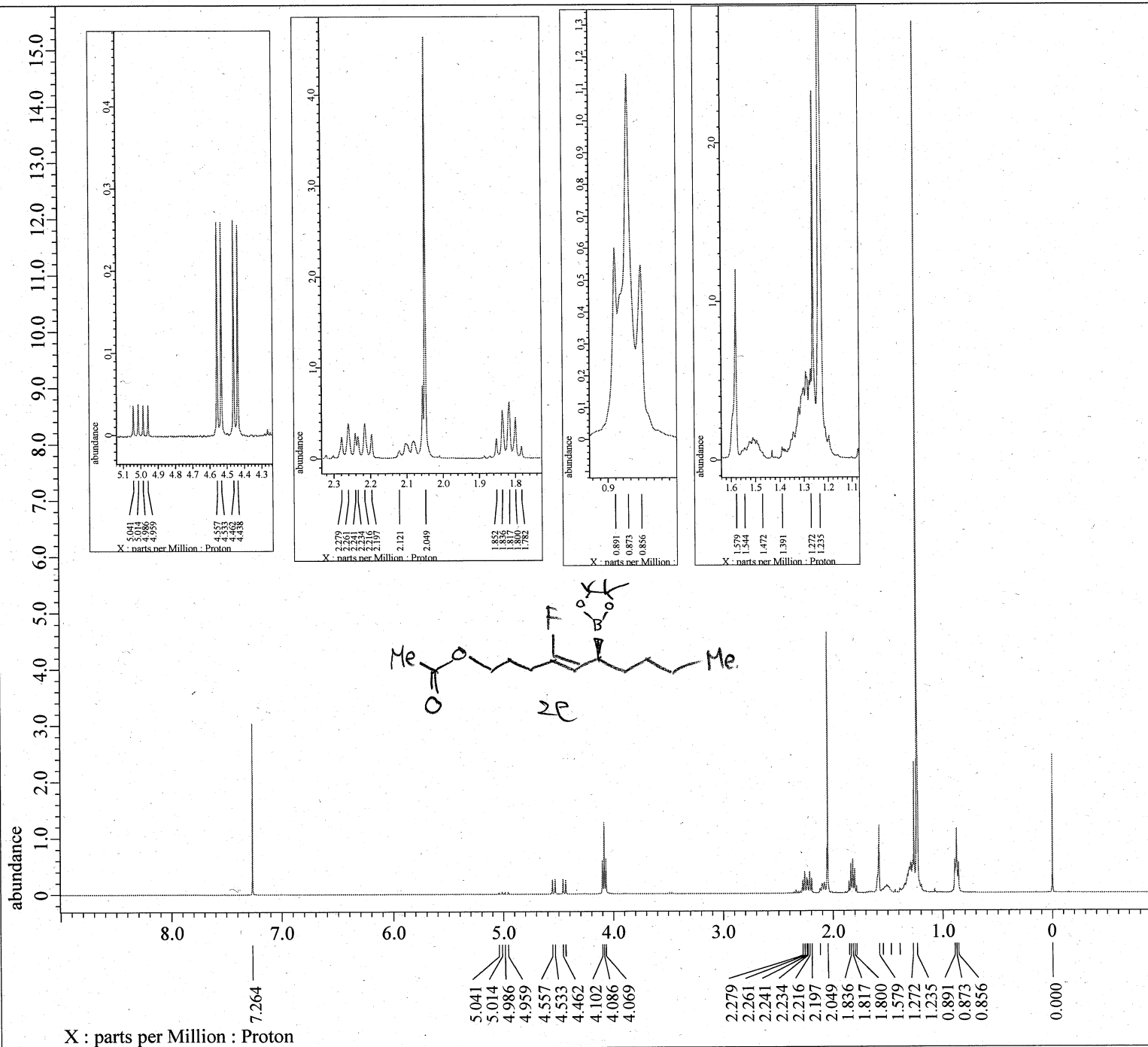


Filename = AKY668-pure\_Proton-1-2.jd  
Author = element  
Experiment = proton.jxp  
Sample\_Id = AKY668-pure  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 20-SEP-2018 18:40:43  
Revision\_Time = 22-MAR-2019 19:31:31

Comment = single pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 13107  
X\_Domain = Proton  
Dim\_Title = Proton  
Dim\_Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.4073814[T] (400[MHz])  
X\_Acq\_Duration = 2.18103808[s]  
X\_Domain = 1H  
X\_Freq = 400.53219825[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45849727[Hz]  
X\_Sweep = 7.51201923[kHz]  
X\_Sweep\_Clipped = 6.00961538[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 38  
Temp\_Get = 20[dC]  
X\_90\_Width = 6.22[us]  
X\_Acq\_Time = 2.18103808[s]  
X\_Angle = 45[deg]  
X\_Atn = 0.8[dB]  
X\_Pulse = 3.11[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.18103808[s]



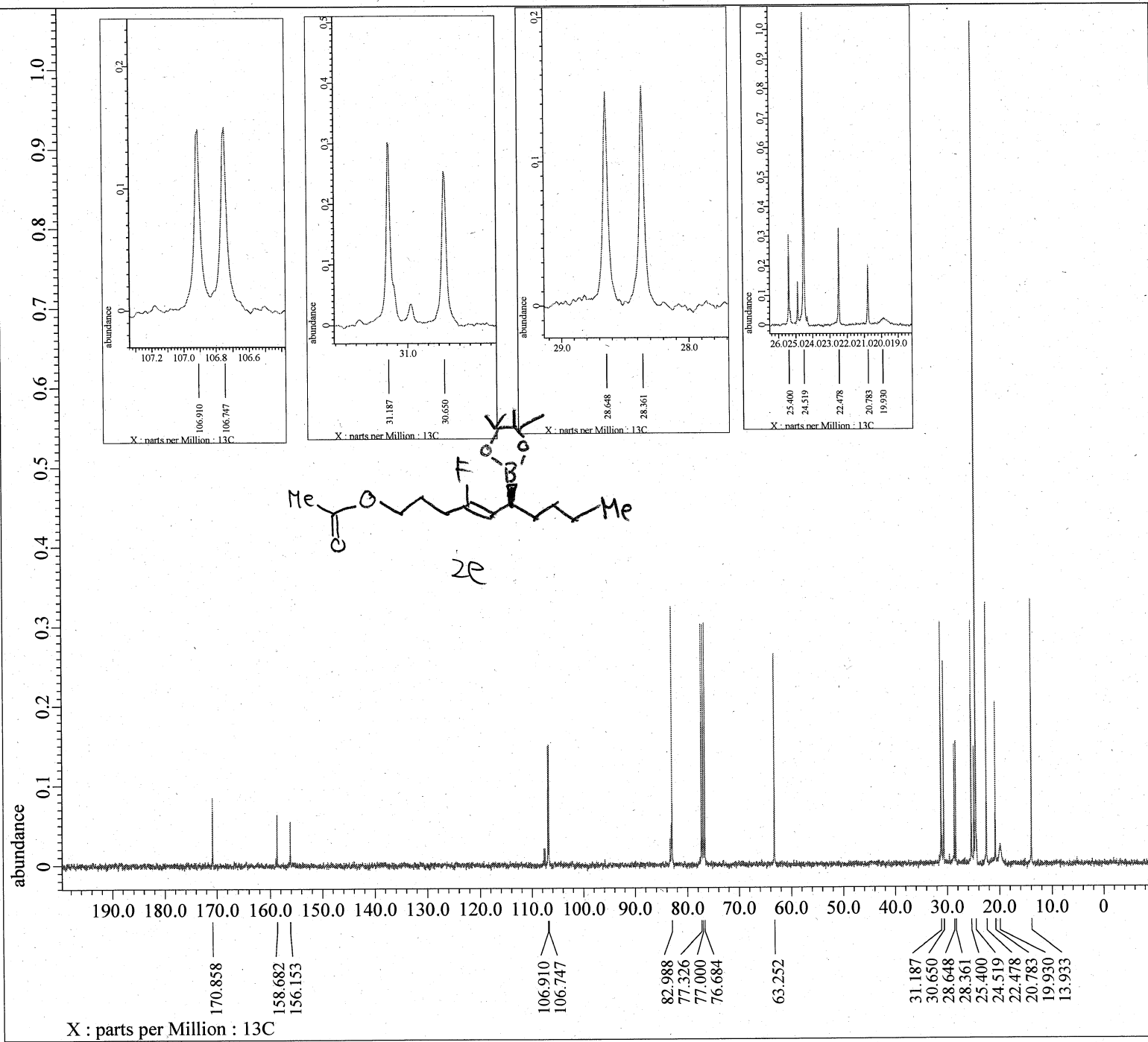


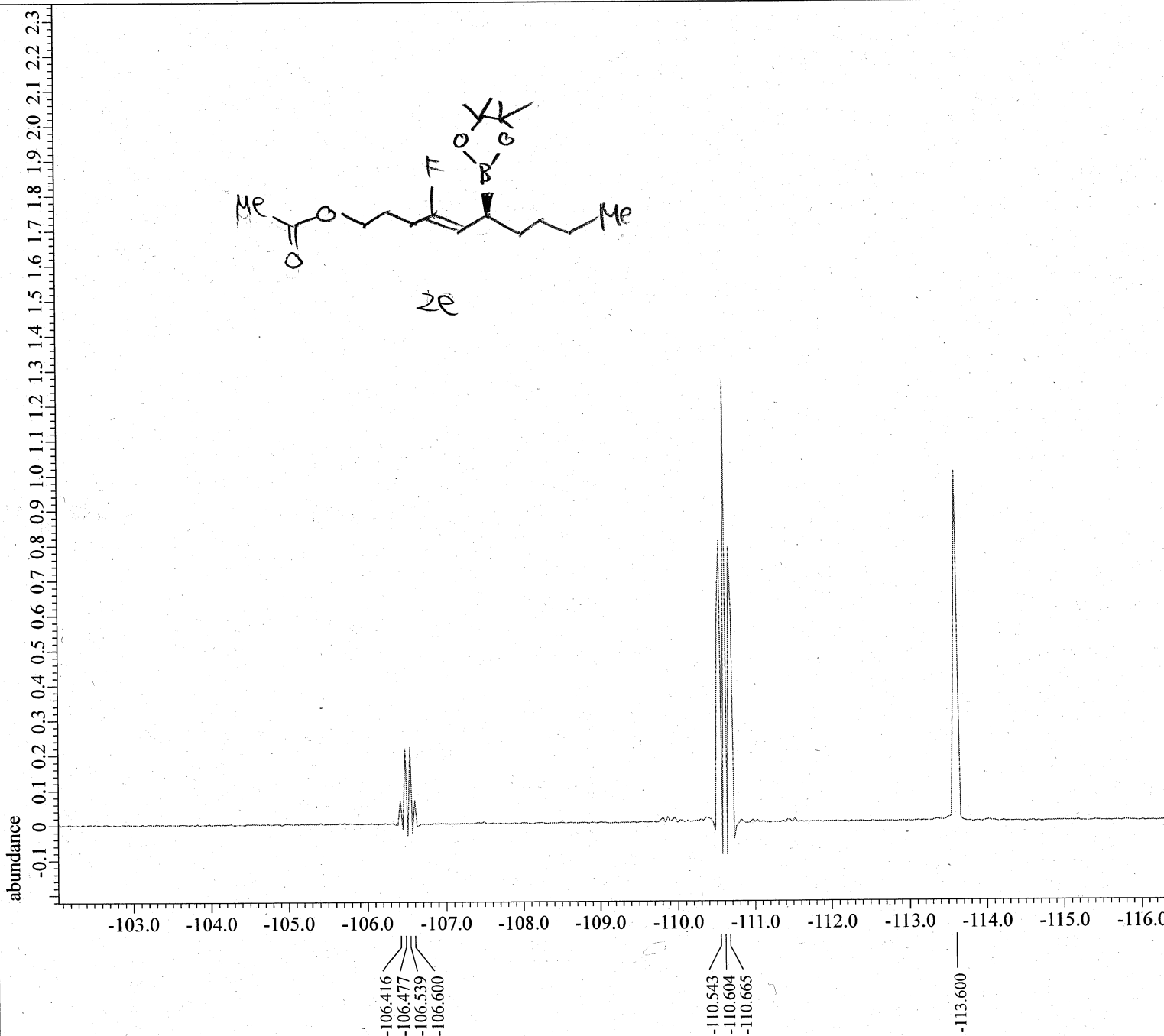
Filename = AKY668-carbon-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#687255  
Solvent = CHLOROFORM-D  
Actual Start Time = 21-SEP-2018 03:33:38  
Revision Time = 22-MAR-2019 19:35:53

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 48  
Temp Get = 21.4[dC]  
X\_90\_Width = 10.1[us]  
X\_Acq Time = 1.048576[s]  
X\_Angle = 30[deg]  
X\_Atn = 3.4[dB]  
X\_Pulse = 3.36666667[us]  
Irr\_Atn Dec = 22.3[dB]  
Irr\_Atn Noe = 22.3[dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]





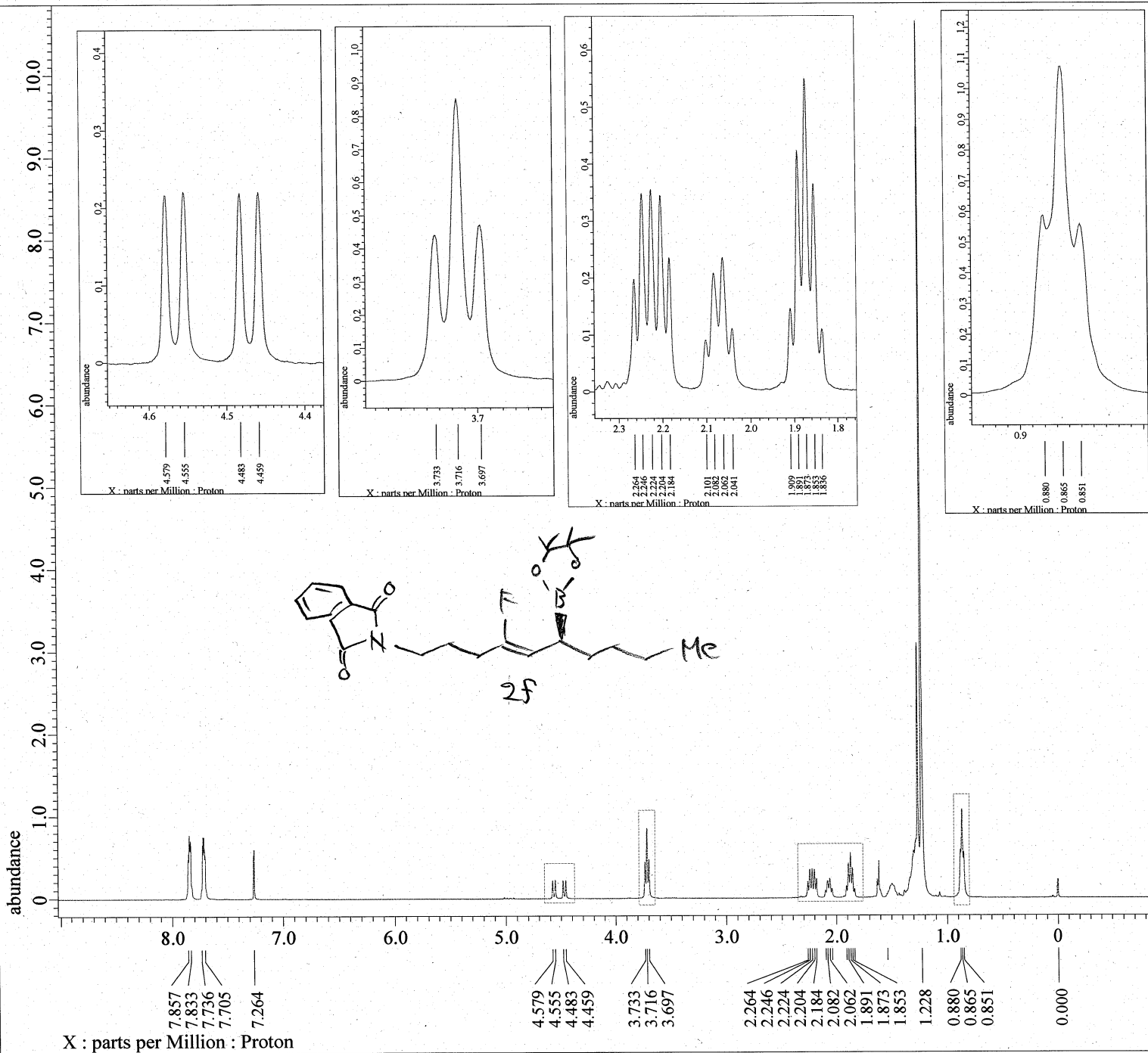
Filename = AKY668-pure-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample\_Id = S#510288  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 20-SEP-2018 22:40:01  
Revision\_Time = 22-MAR-2019 20:34:01

Comment = single pulse  
Data\_Format = 1D COMPLEX  
Dim\_Size = 13107  
X\_Domain = 19F  
Dim\_Title = 19F  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 24  
Temp\_Get = 21.3[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

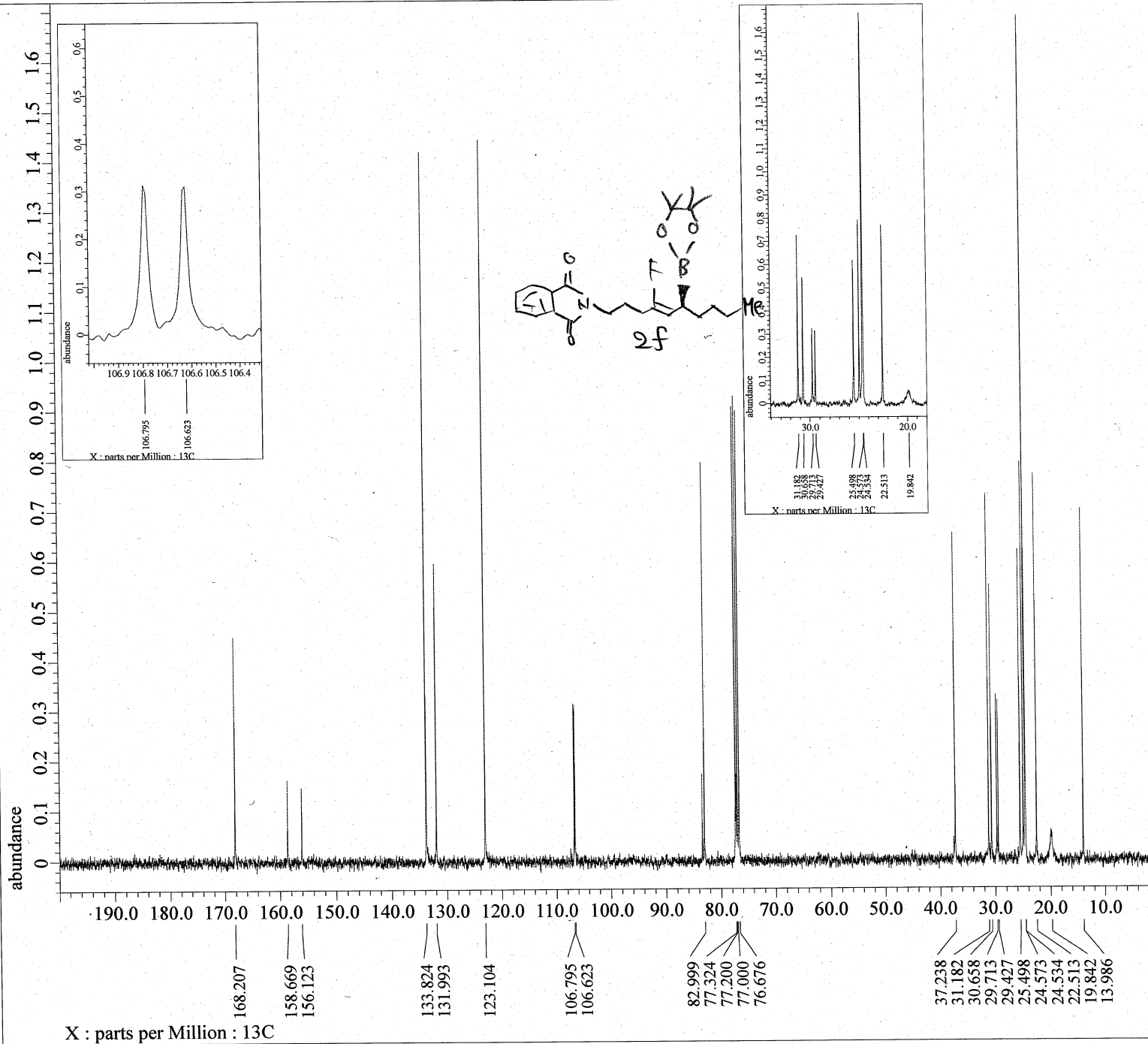


Filename = AKY701-pure-agin\_Proton-1  
Author = element  
Experiment = proton\_jxp  
Sample\_Id = AKY701-pure-agin  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 23-MAR-2019 10:07:42  
Revision\_Time = 23-MAR-2019 10:05:32

Comment = single pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 13107  
X\_Domain = Proton  
Dim\_Title = Proton  
Dim\_Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.4073814[T] (400[MHz])  
X\_Acq\_Duration = 2.18103808[s]  
X\_Domain = 1H  
X\_Freq = 400.53219825[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45849727[Hz]  
X\_Sweep = 7.51201923[kHz]  
X\_Sweep\_Clippped = 6.00961538[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 38  
Temp\_Get = 19.1[dc]  
X\_90\_Width = 6[us]  
X\_Acq\_Time = 2.18103808[s]  
X\_Angle = 45[deg]  
X\_Atn = 0.8[db]  
X\_Pulse = 3[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.18103808[s]

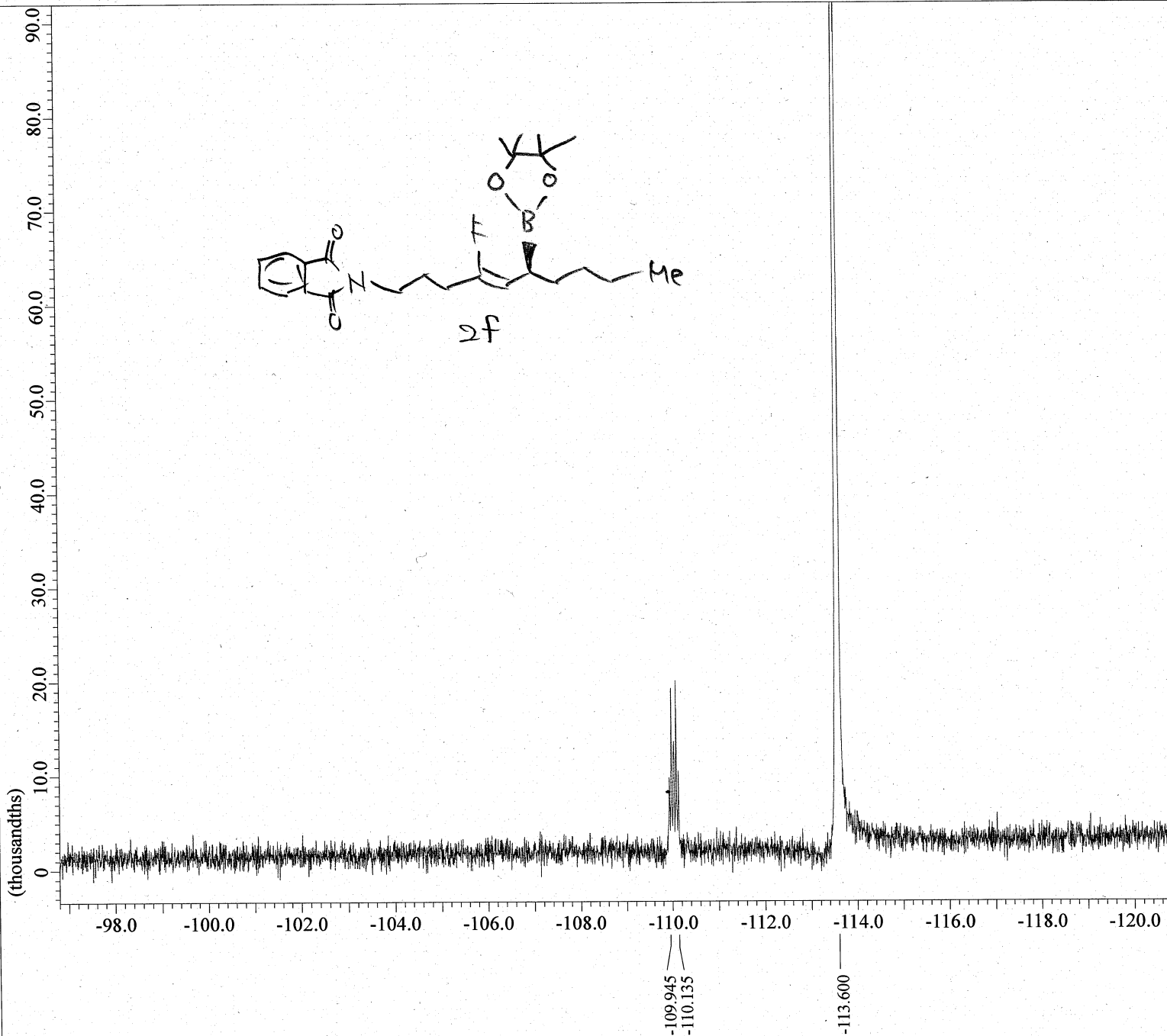


Filename = AKY701-carbon-again-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#347521  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 25-OCT-2018 17:08:11  
Revision\_Time = 23-MAR-2019 11:40:19

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
X Freq = 98.51479726 [MHz]  
X Offset = 100 [ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061 [Hz]  
X Sweep = 30.78817734 [kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441 [MHz]  
Irr Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2 [s]  
Recvr Gain = 60  
Temp Get = 19.4 [dC]  
X 90 Width = 9.11 [us]  
X Acq Time = 1.06430464 [s]  
X Angle = 30 [deg]  
X Atn = 4.9 [dB]  
X Pulse = 3.0366667 [us]  
Irr Atn Dec = 22.255 [dB]  
Irr Atn Noe = 22.255 [dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1 [s]  
Noe = TRUE  
Noe\_Time = 2 [s]  
Repetition\_Time = 3.06430464 [s]

X : parts per Million : 13C



Filename = AKY701-fnmr-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = 2  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 25-OCT-2018 06:35:51  
Revision\_Time = 23-MAR-2019 11:14:53

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 0.43778048[s]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = -90[ppm]  
X Points = 16384  
X\_Prescans = 1  
X\_Resolution = 2.28424986[Hz]  
X\_Sweep = 37.4251497[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 32  
Temp\_Get = 21[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 0.43778048[s]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.43778048[s]

X : parts per Million : 19F

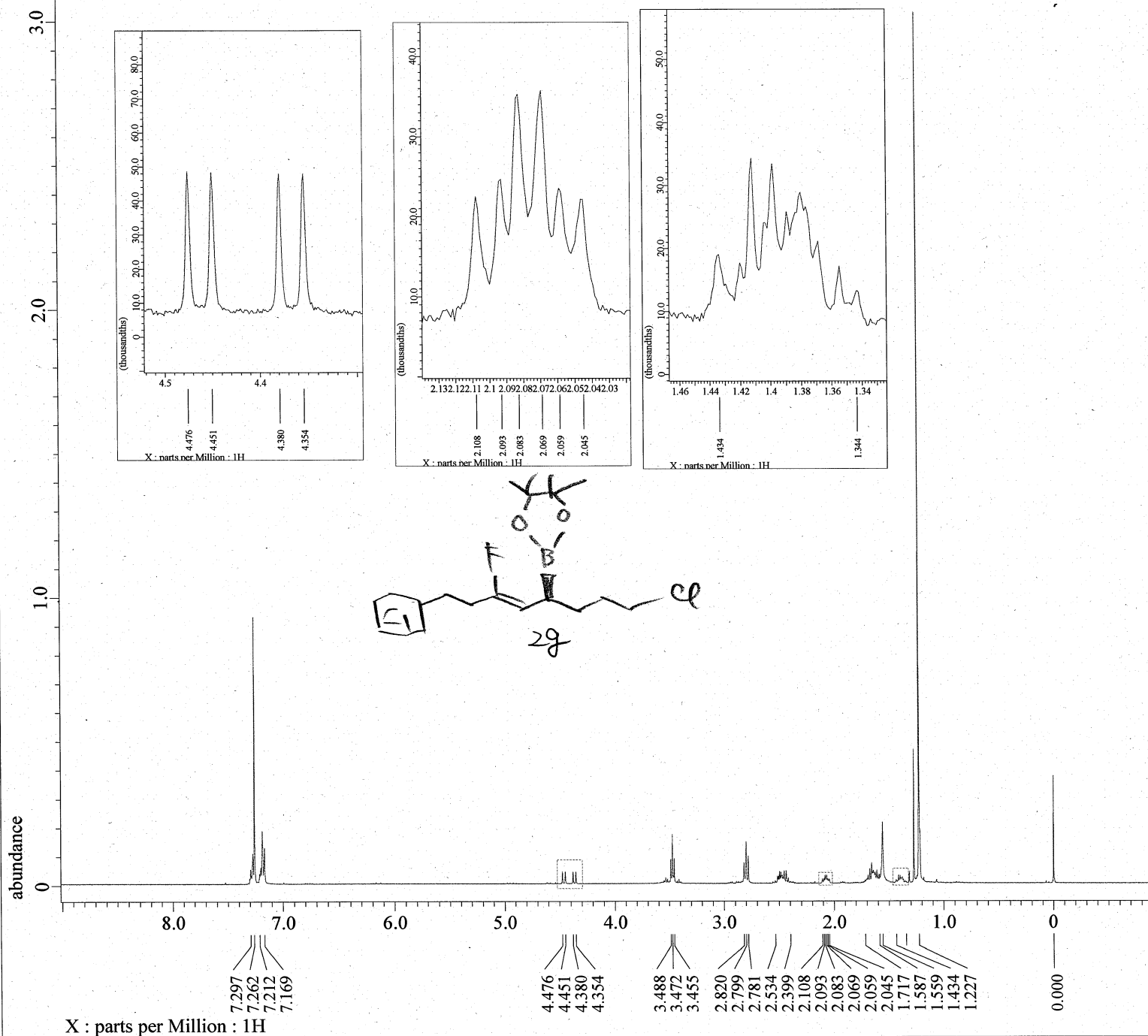


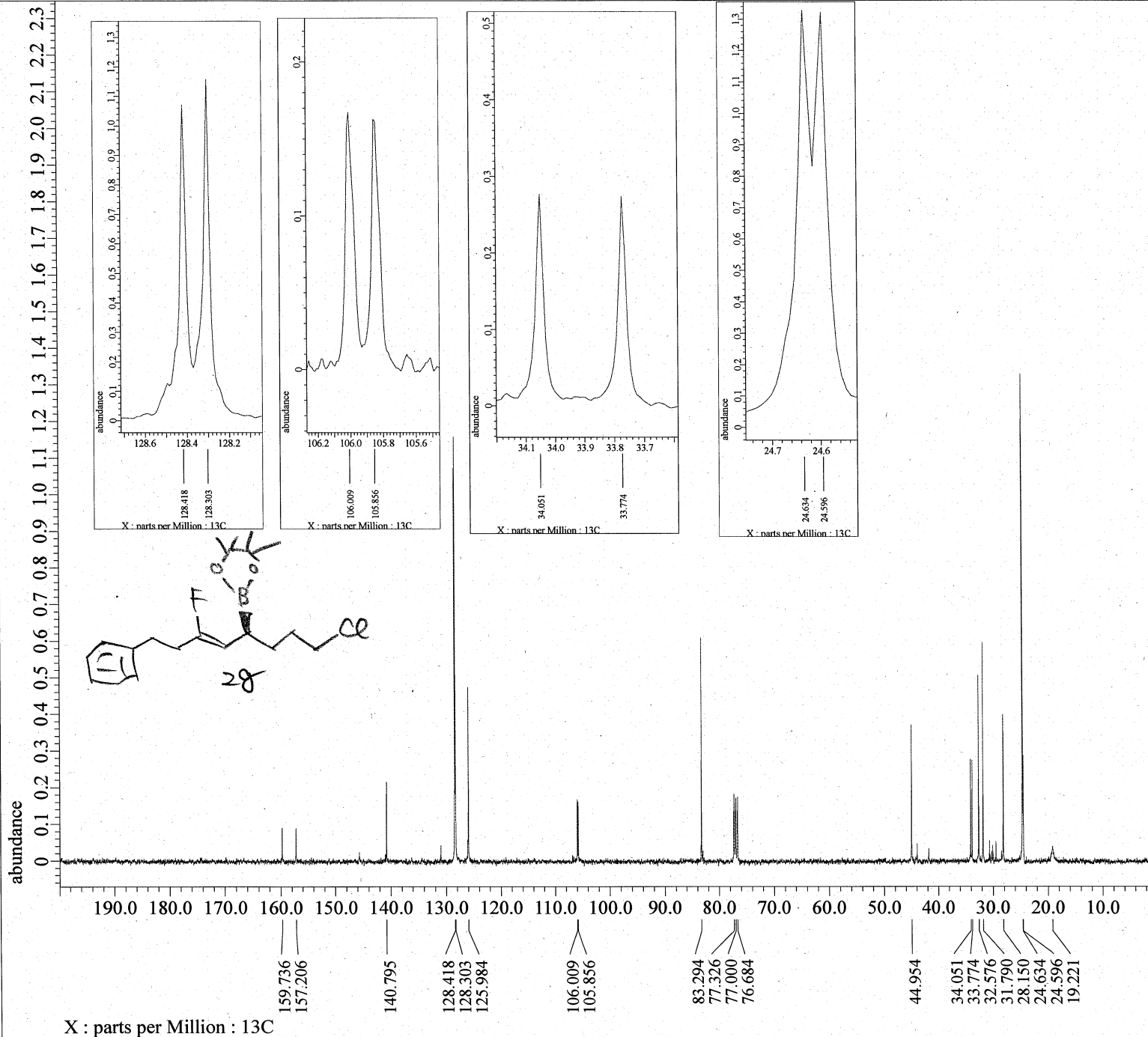
Filename = AKY631-pure-again-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#672344  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 24-MAR-2019 03:03:00  
Revision\_Time = 23-MAR-2019 19:32:50

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq\_Duration = 2.20725248 [s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr Gain = 38  
Temp Get = 20.8 [dC]  
X 90\_Width = 13.2 [us]  
X Acq\_Time = 2.20725248 [s]  
X Angle = 45 [deg]  
X Atn = 3.5 [dB]  
X Pulse = 6.6 [us]  
Irr Mode = Off  
Tri Mode = Off  
DanTe\_Preat = FALSE  
Initial\_Wait = 1 [s]  
Repetition\_Time = 7.20725248 [s]





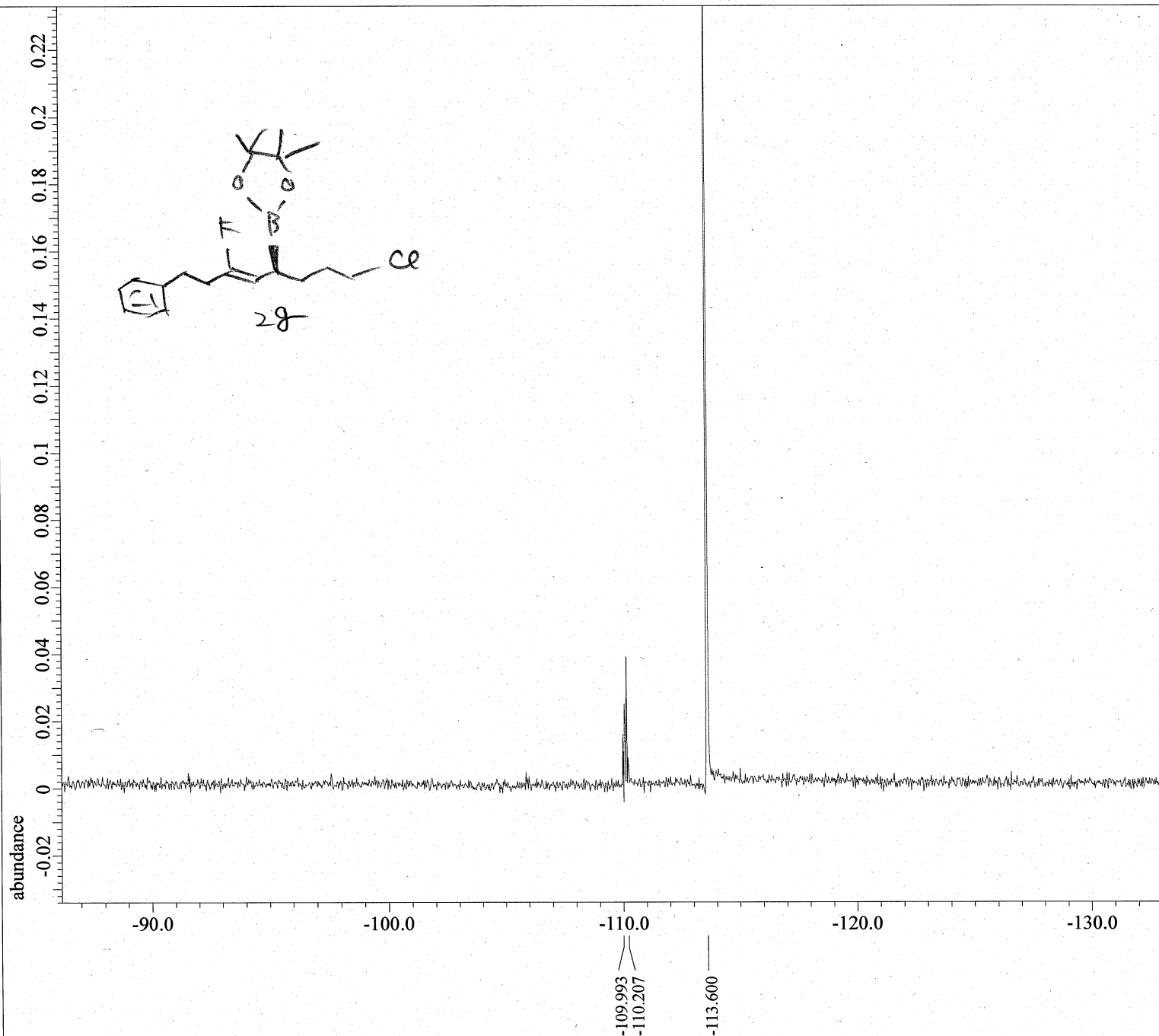
Filename = AKY631-carbon-128-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#681808  
Solvent = CHLOROFORM-D  
Actual Start Time = 24-MAR-2019 03:19:01  
Revision Time = 23-MAR-2019 19:34:56

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total Scans = 128

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 20.9[dc]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[db]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[db]  
Irr Atn Noe = 22.3[db]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]





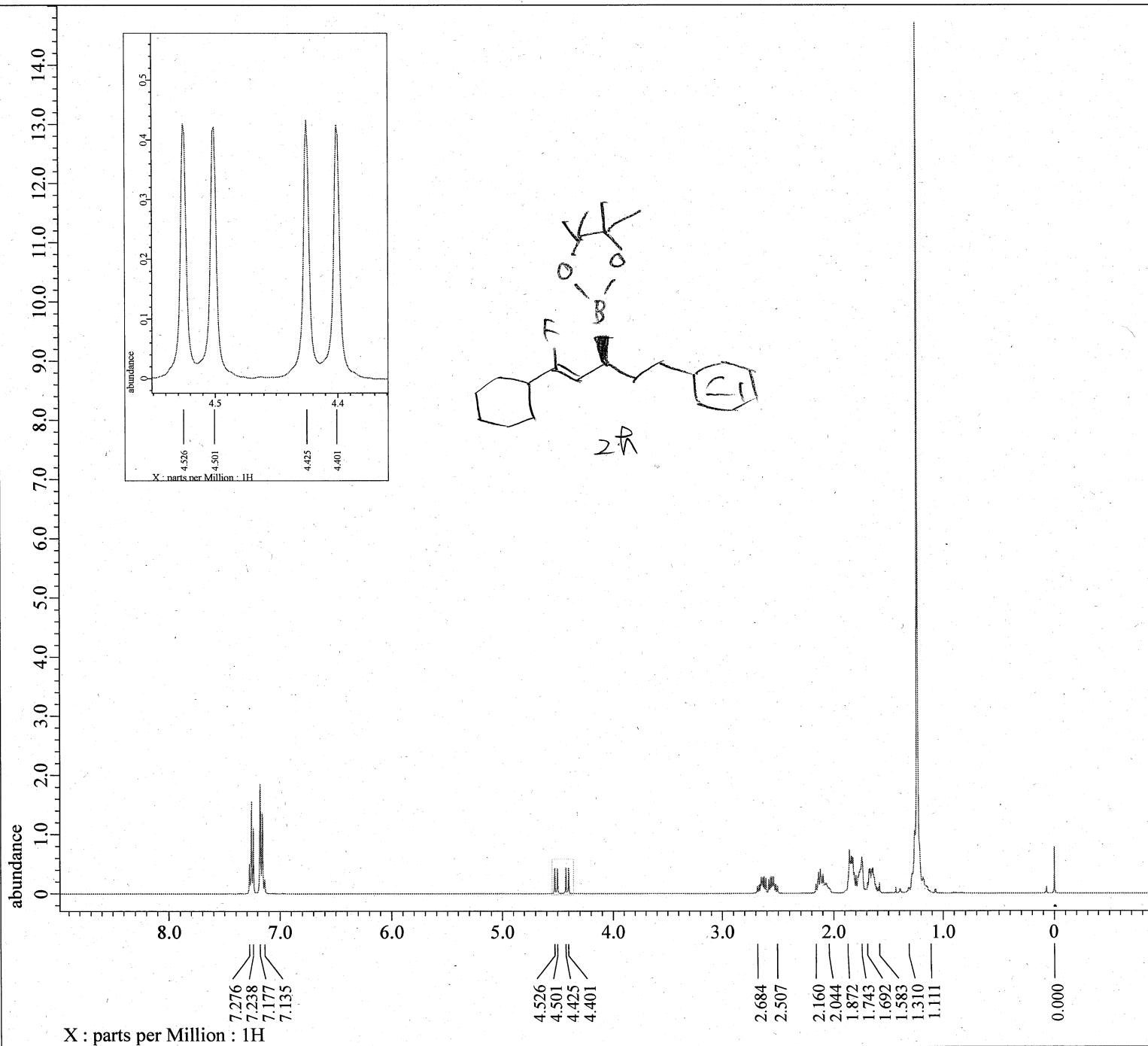
Filename = AKY631-pure-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#677749  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 24-MAR-2019 03:11:57  
Revision Time = 23-MAR-2019 19:37:32

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 20.7[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]

X : parts per Million : 19F

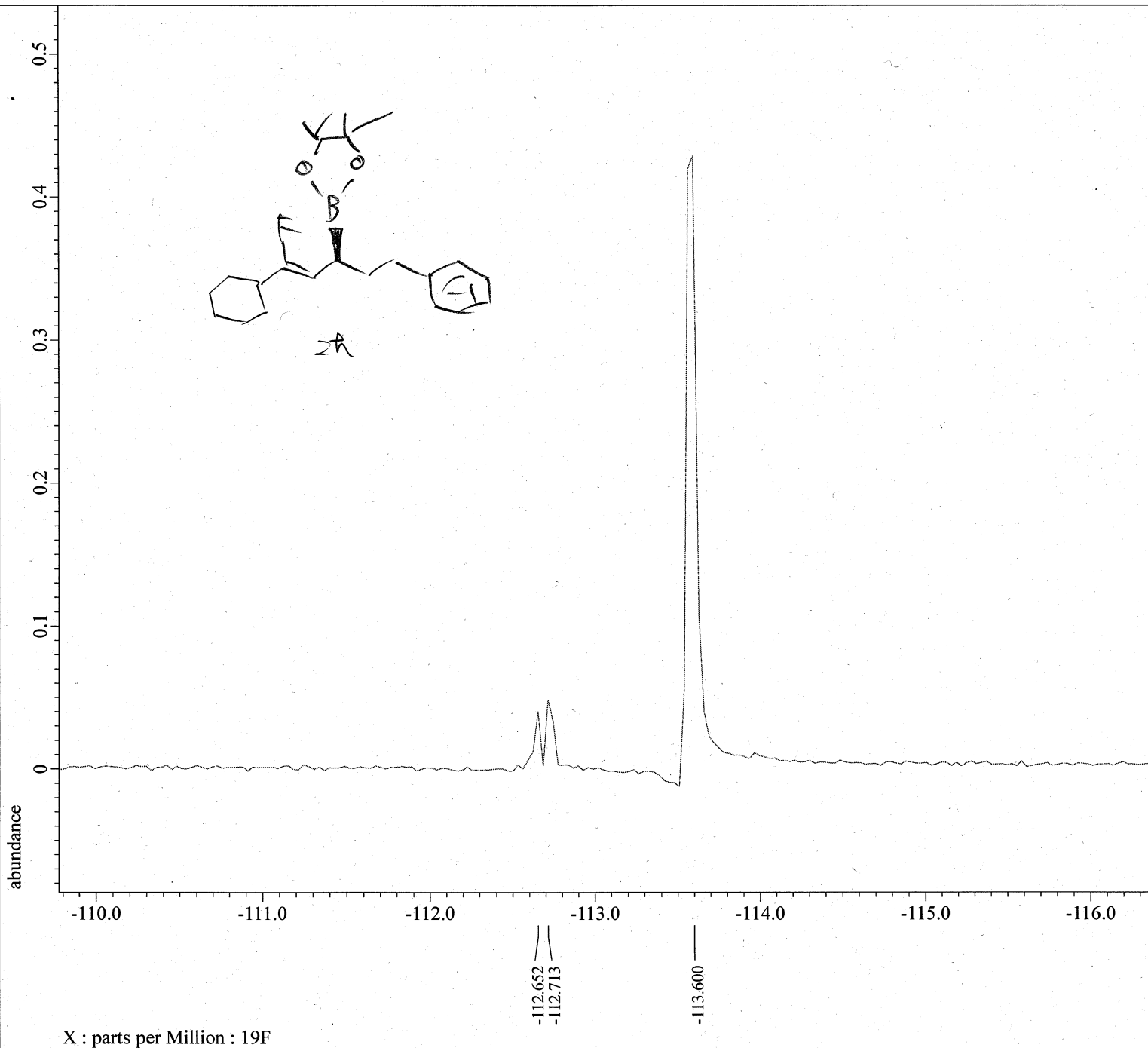


Filename = AKY6XX-Cy-pure-aagin-2.jd  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#574021  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 30-OCT-2018 23:25:33  
Revision\_Time = 31-OCT-2018 09:59:29

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 34  
Temp Get = 18.4[dC]  
X 90\_Width = 11.04[us]  
X Acq\_Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 7.228224[s]

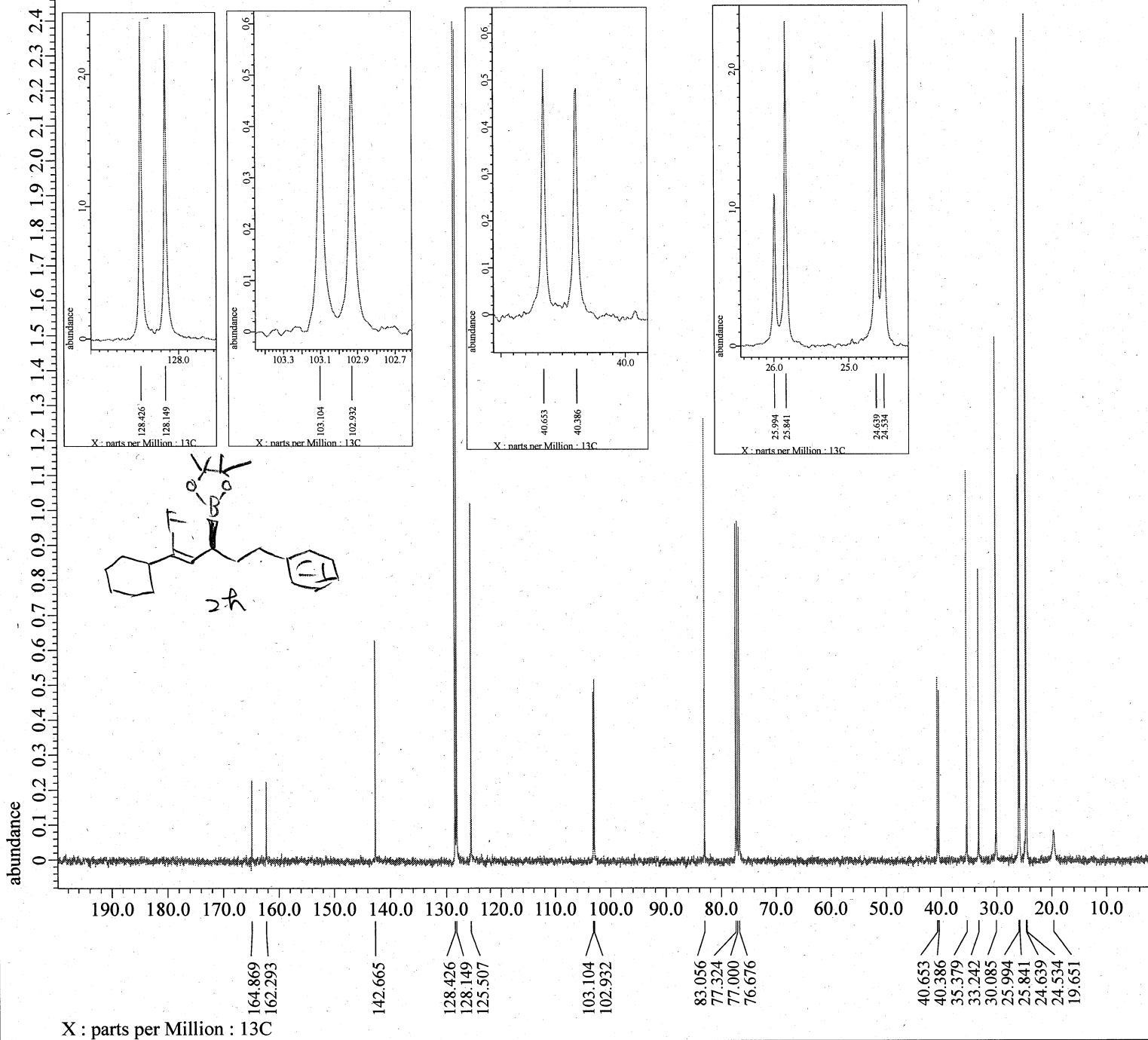


Filename = AKY6XX-Cy-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#568437  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 31-OCT-2018 00:16:57  
Revision\_Time = 31-OCT-2018 10:11:13

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 19.1[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]



Filename = AKY6XX-Cy-carbon-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#578338  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 30-OCT-2018 23:31:24  
Revision\_Time = 31-OCT-2018 10:06:26

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390 [MHz])  
X Acq\_Duration = 1.06430464[s]  
X Domain = 13C  
X\_Freq = 98.51479726 [MHz]  
X\_Offset = 100 [ppm]  
X Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.93958061 [Hz]  
X\_Sweep = 30.78817734 [kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441 [MHz]  
Irr\_Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2 [s]  
Recvr\_Gain = 60  
Temp\_Get = 19.2 [dC]  
X\_90\_Width = 9.11 [us]  
X Acq Time = 1.06430464 [s]  
X\_Angle = 30 [deg]  
X\_Atn = 4.9 [dB]  
X\_Pulse = 3.03666667 [us]  
Irr\_Atn\_Dec = 22.255 [dB]  
Irr\_Atn\_Noise = 22.255 [dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1 [s]  
Noe = TRUE  
Noe Time = 2 [s]  
Repetition\_Time = 3.06430464 [s]

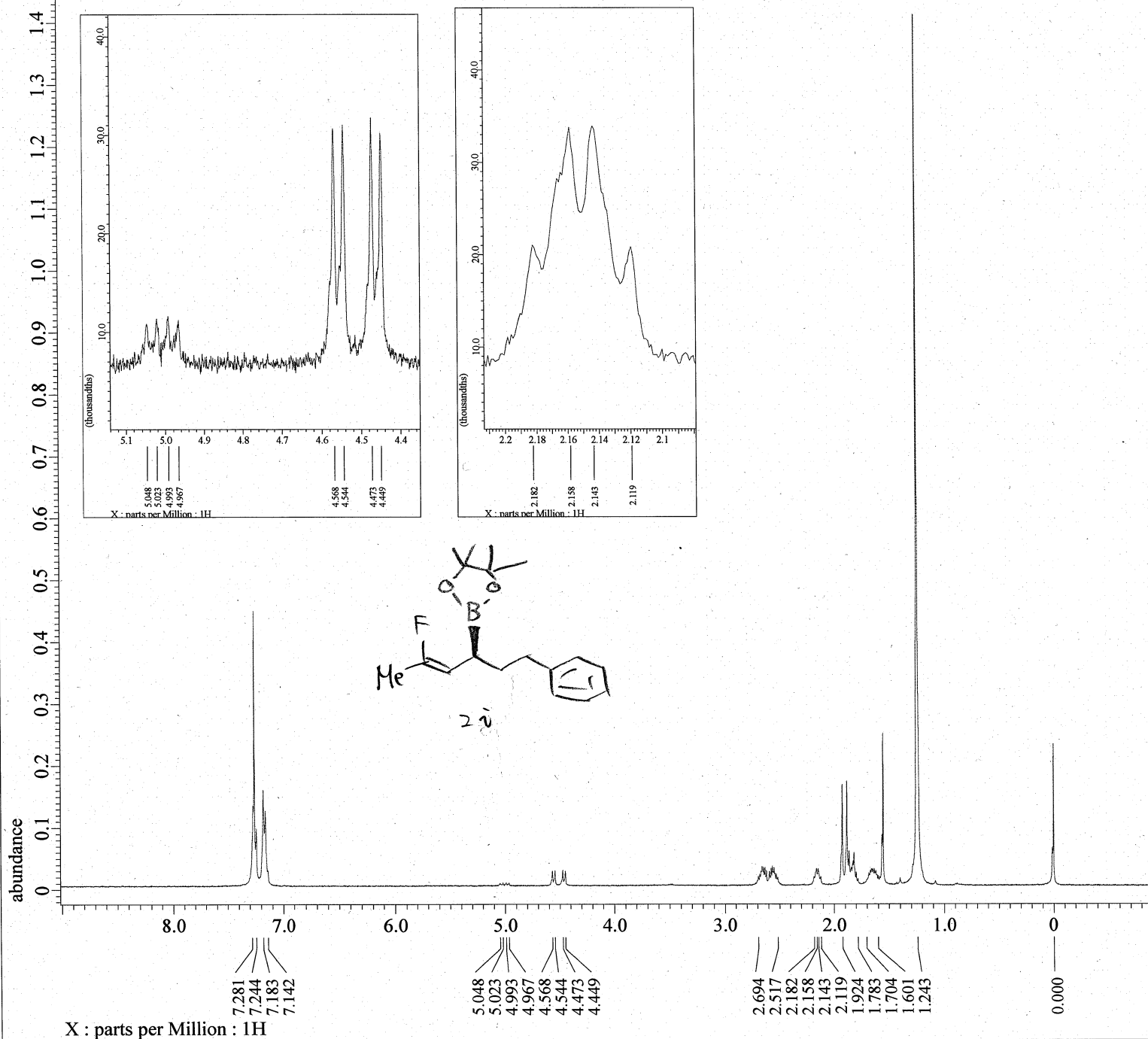


Filename = AKY838-pure-HNMR-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#459729  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 9-MAR-2019 21:16:09  
Revision\_Time = 24-MAR-2019 14:29:03

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 2.20725248[s]  
X Domain = 1H  
X Freq = 395.88430144[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193[Hz]  
X Sweep = 7.42280285[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 38  
Temp Get = 20.4[dc]  
X\_90\_Width = 13.2[us]  
X Acq\_Time = 2.20725248[s]  
X Angle = 45[deg]  
X Atn = 3.5[dB]  
X Pulse = 6.6[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.20725248[s]



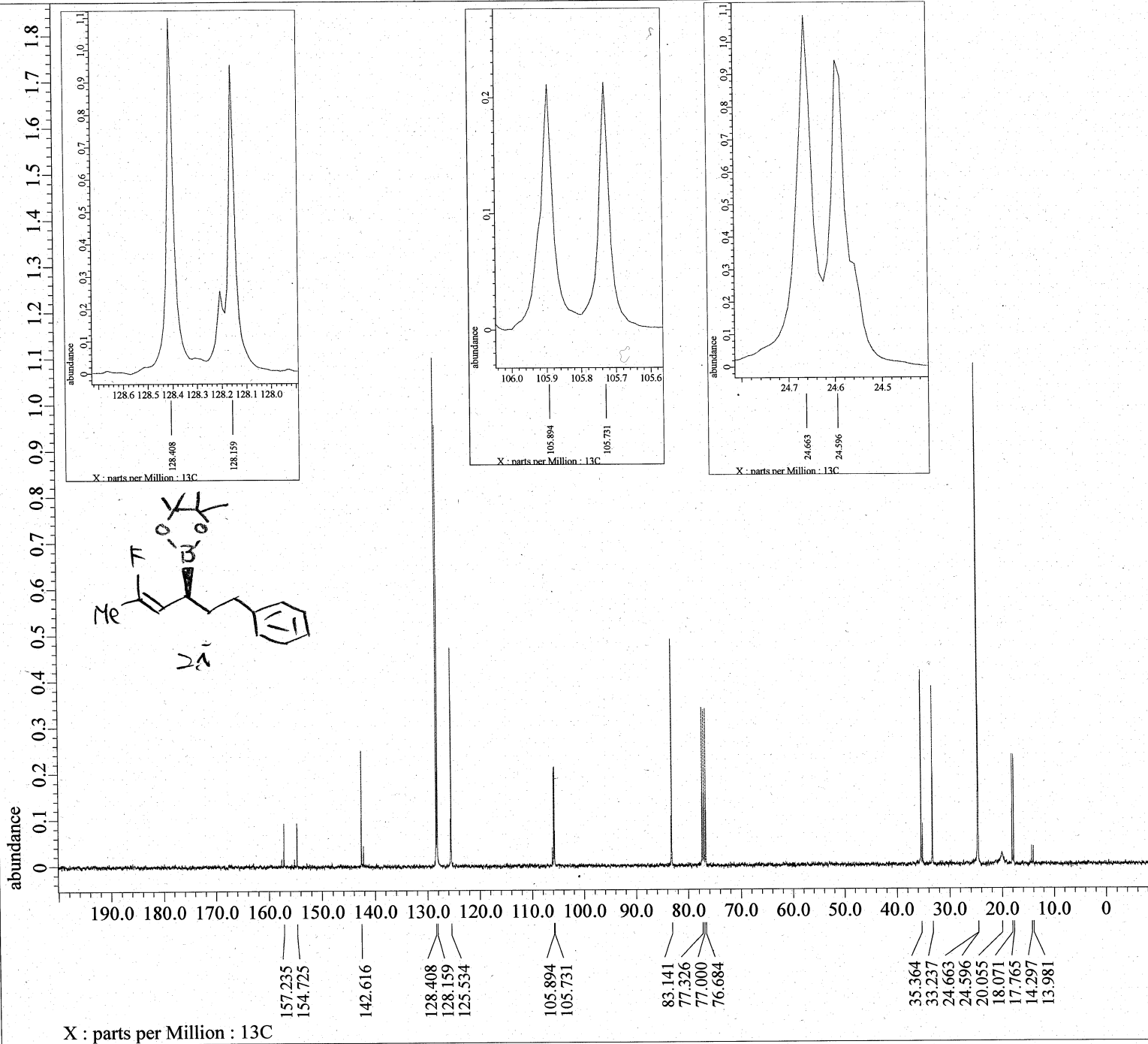


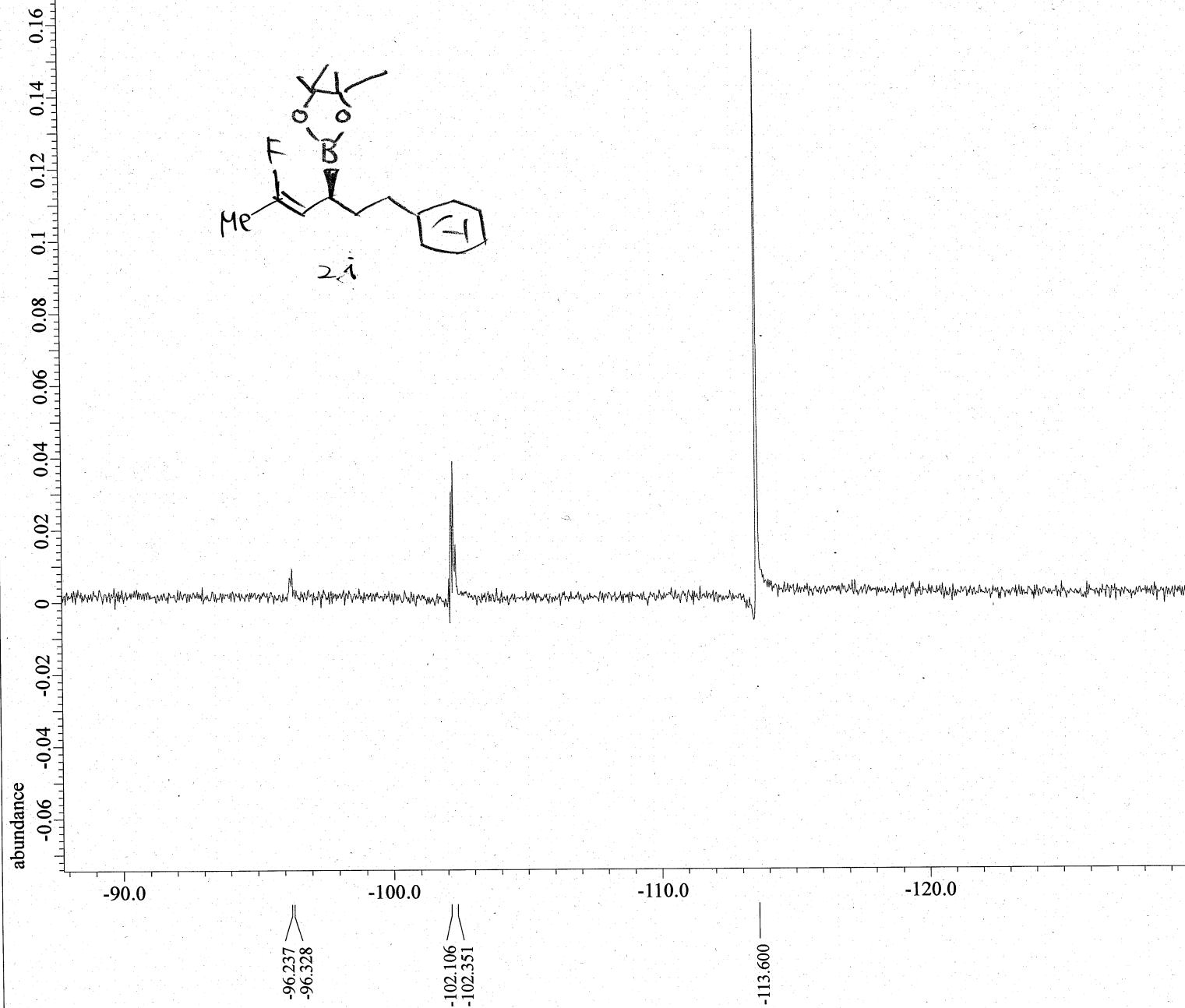
Filename = AKY838-carbon-128-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample\_Id = S#463453  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 9-MAR-2019 21:22:08  
Revision\_Time = 24-MAR-2019 14:35:36

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECK 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2[s]  
Recvr Gain = 50  
Temp Get = 20.8[dc]  
X 90\_Width = 10.1[us]  
X Acq\_Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition\_Time = 3.048576[s]





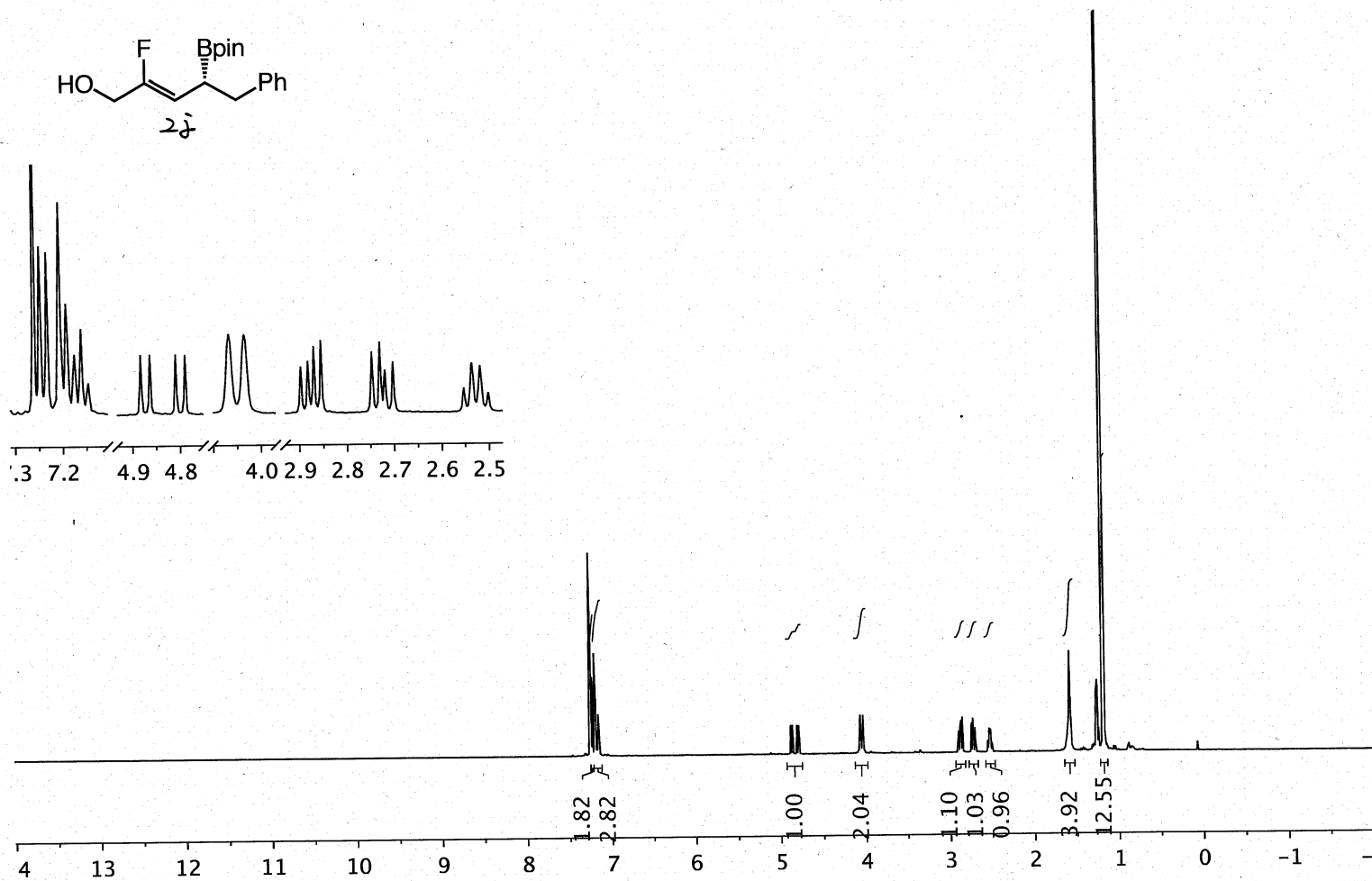
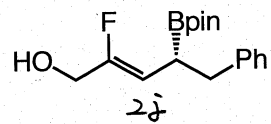
Filename = AKY838-pure-FNMR-ag-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample\_Id = S#496025  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 9-MAR-2019 22:17:18  
Revision\_Time = 24-MAR-2019 14:44:26

Comment = single\_pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 13107  
X\_Domain = 19F  
Dim\_Title = 19F  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

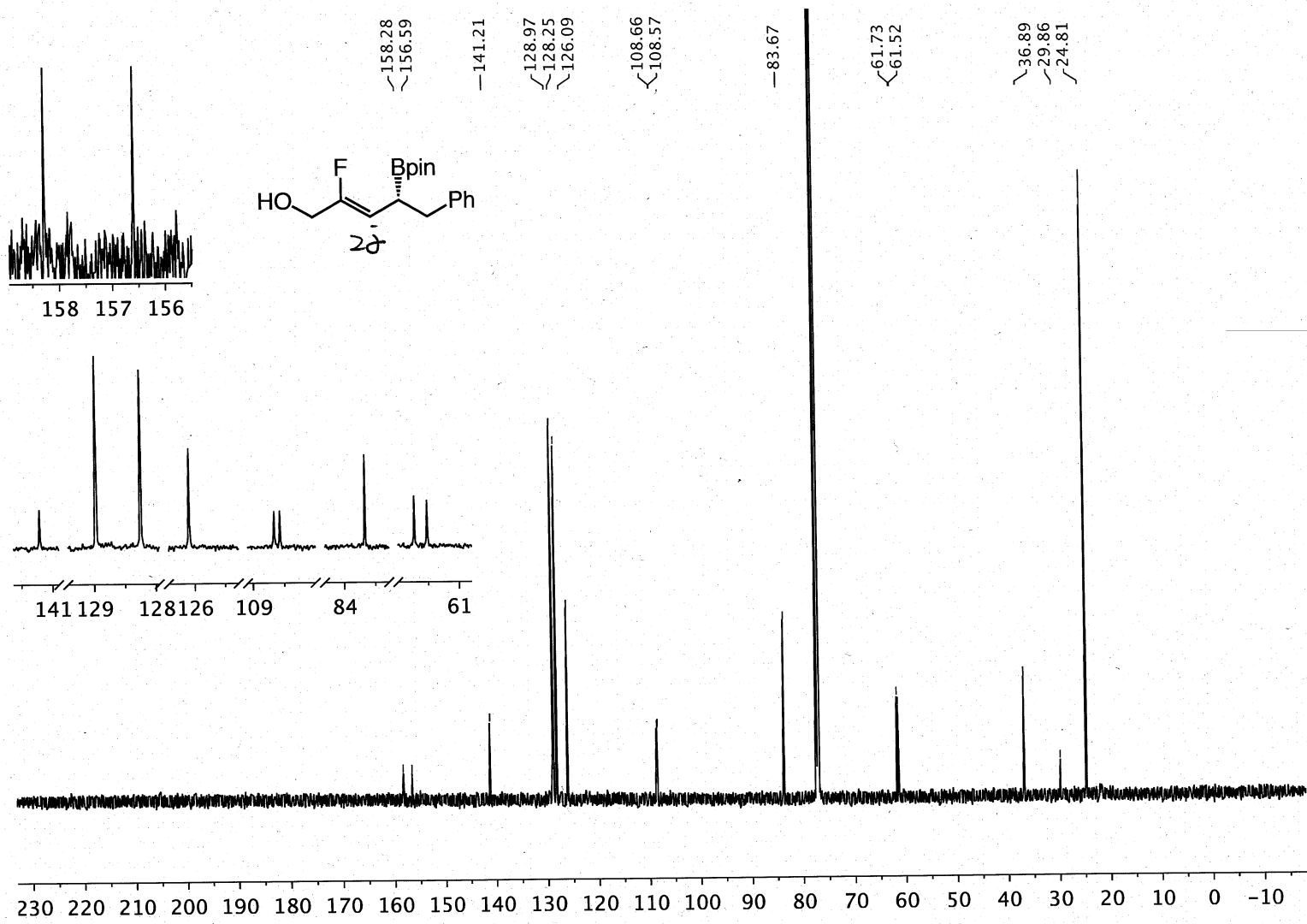
Field\_Strength = 9.2982153[T] (400 [MHz])  
X\_Acq\_Duration = 87.81824 [ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686 [MHz]  
X\_Offset = 0 [ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602 [Hz]  
X\_Sweep = 186.56716418 [kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686 [MHz]  
Irr\_Offset = 5 [ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686 [MHz]  
Tri\_Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

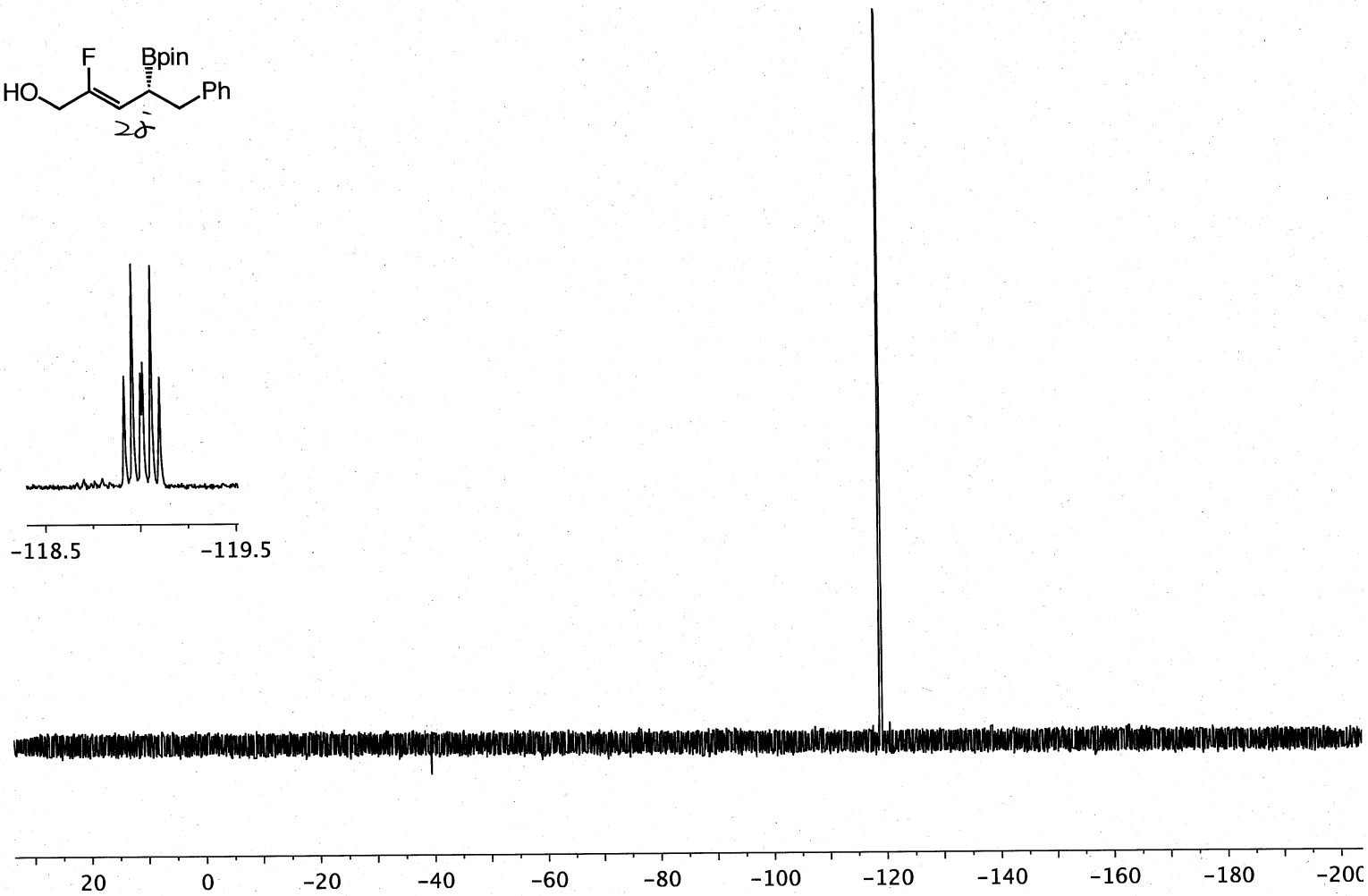
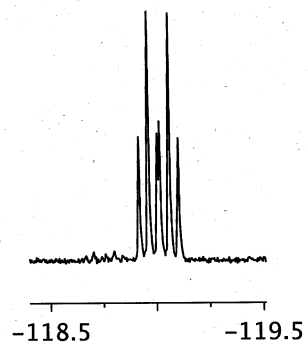
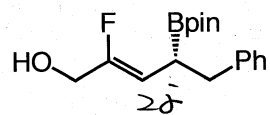
Relaxation\_Delay = 5 [s]  
Recvr\_Gain = 24  
Temp\_Get = 20.6 [dC]  
X\_90\_Width = 13.9 [us]  
X\_Acq\_Time = 87.81824 [ms]  
X\_Angle = 45 [deg]  
X\_Atn = 4 [dB]  
X\_Pulse = 6.95 [us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1 [s]  
Repetition\_Time = 5.08781824 [s]

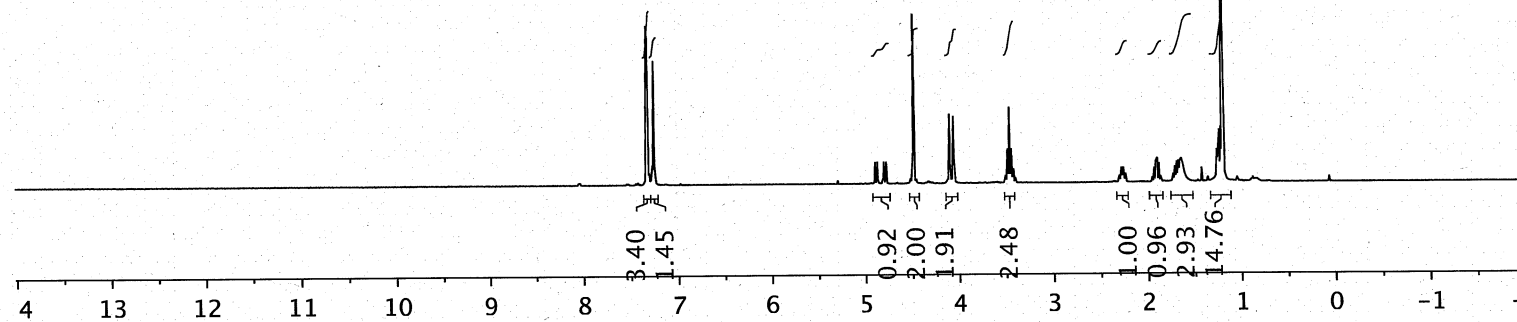
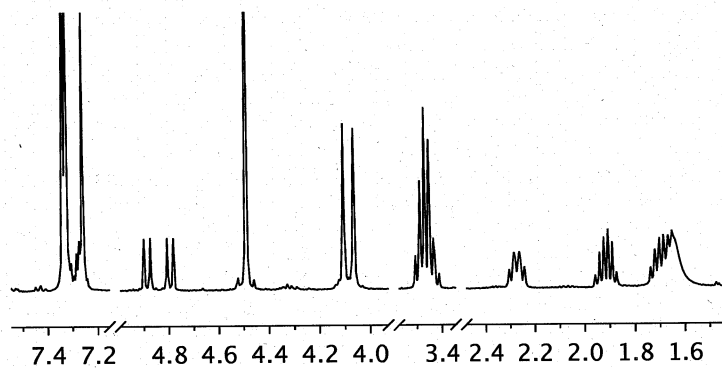
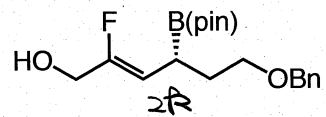
X : parts per Million : 19F

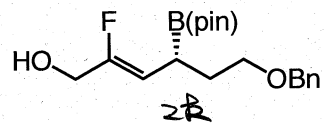












158.47  
156.45

138.75

128.44  
127.86  
127.60

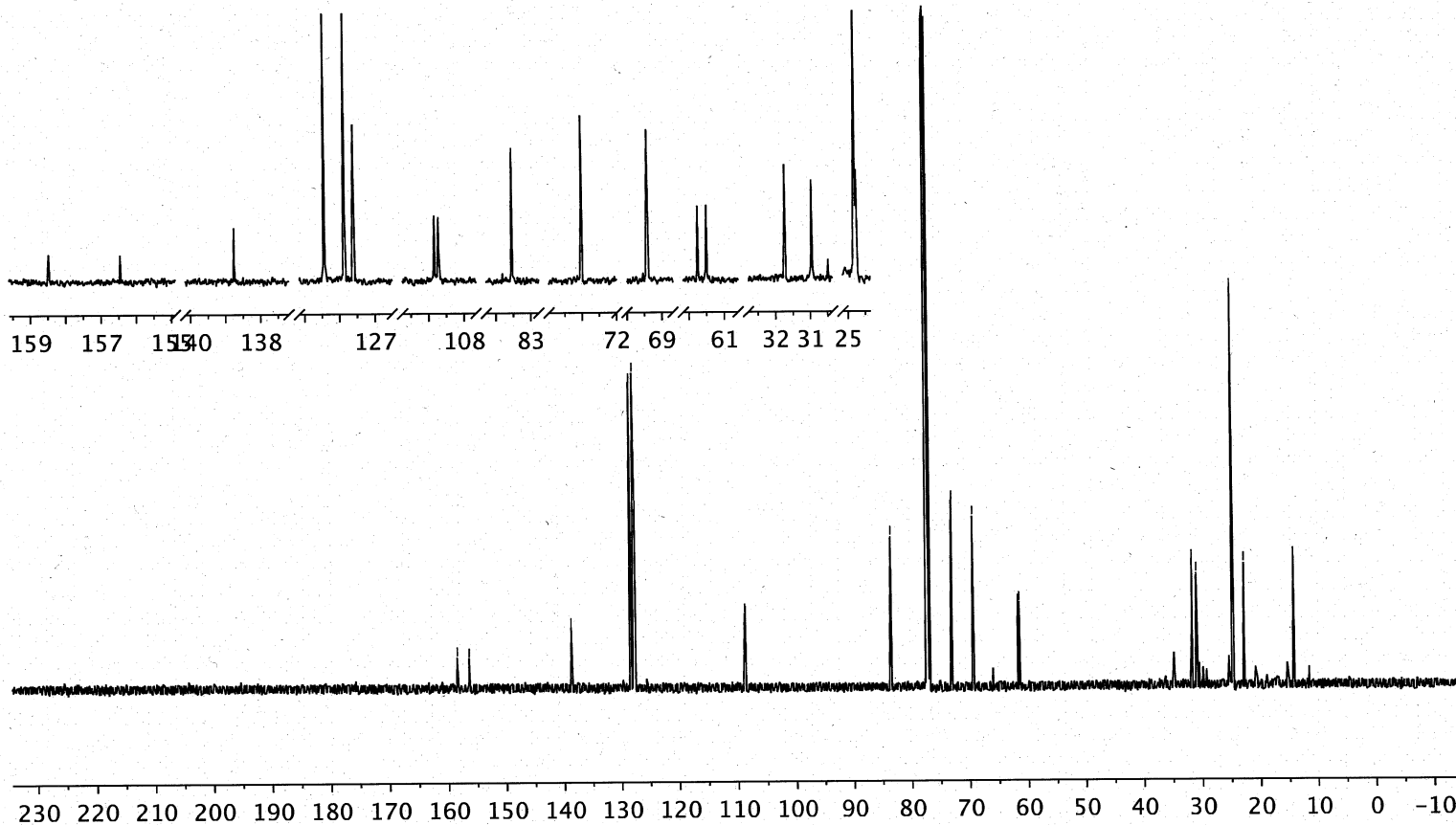
108.83  
108.72

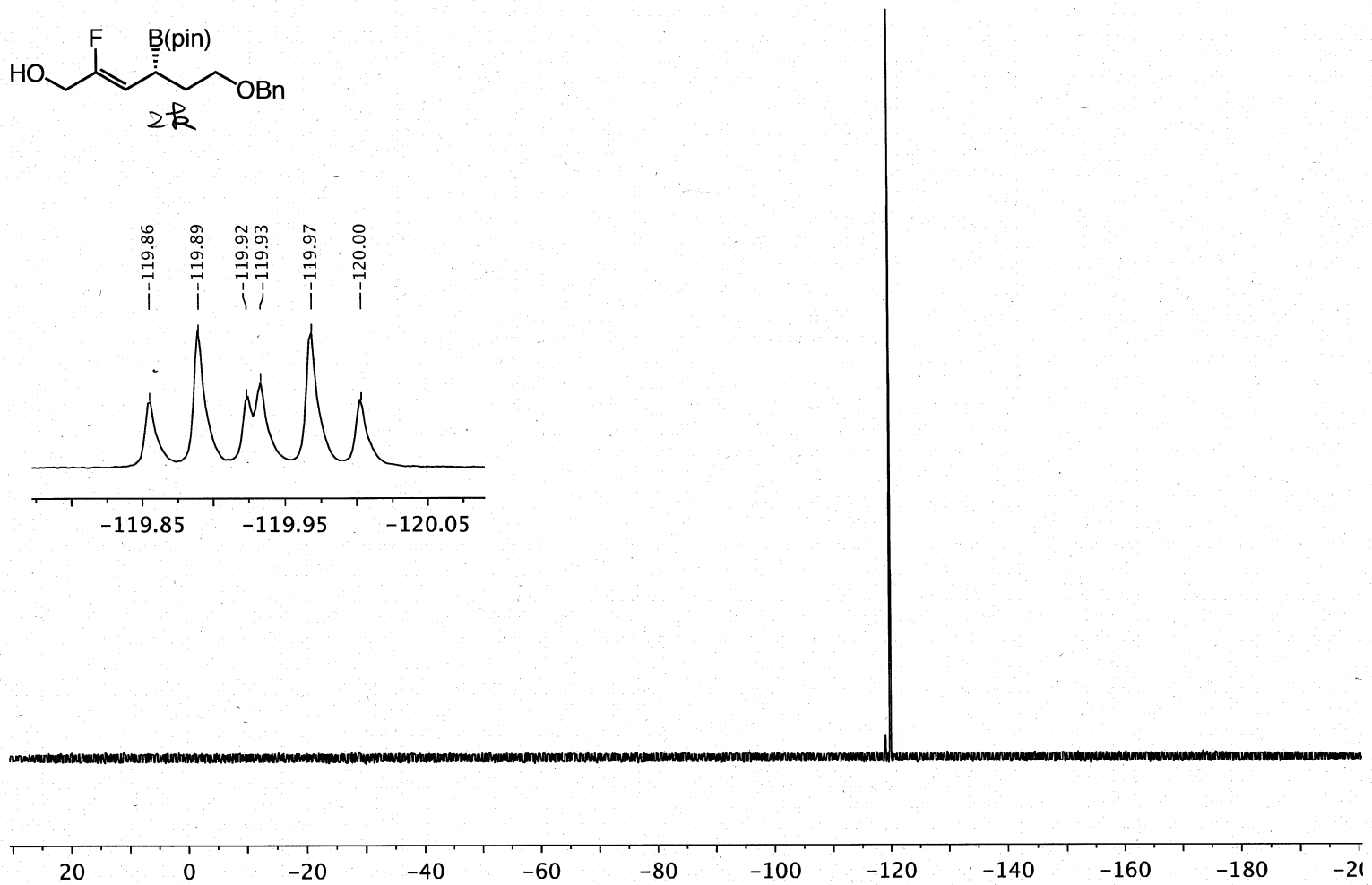
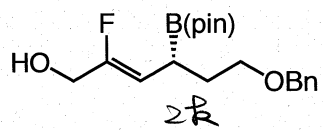
83.54

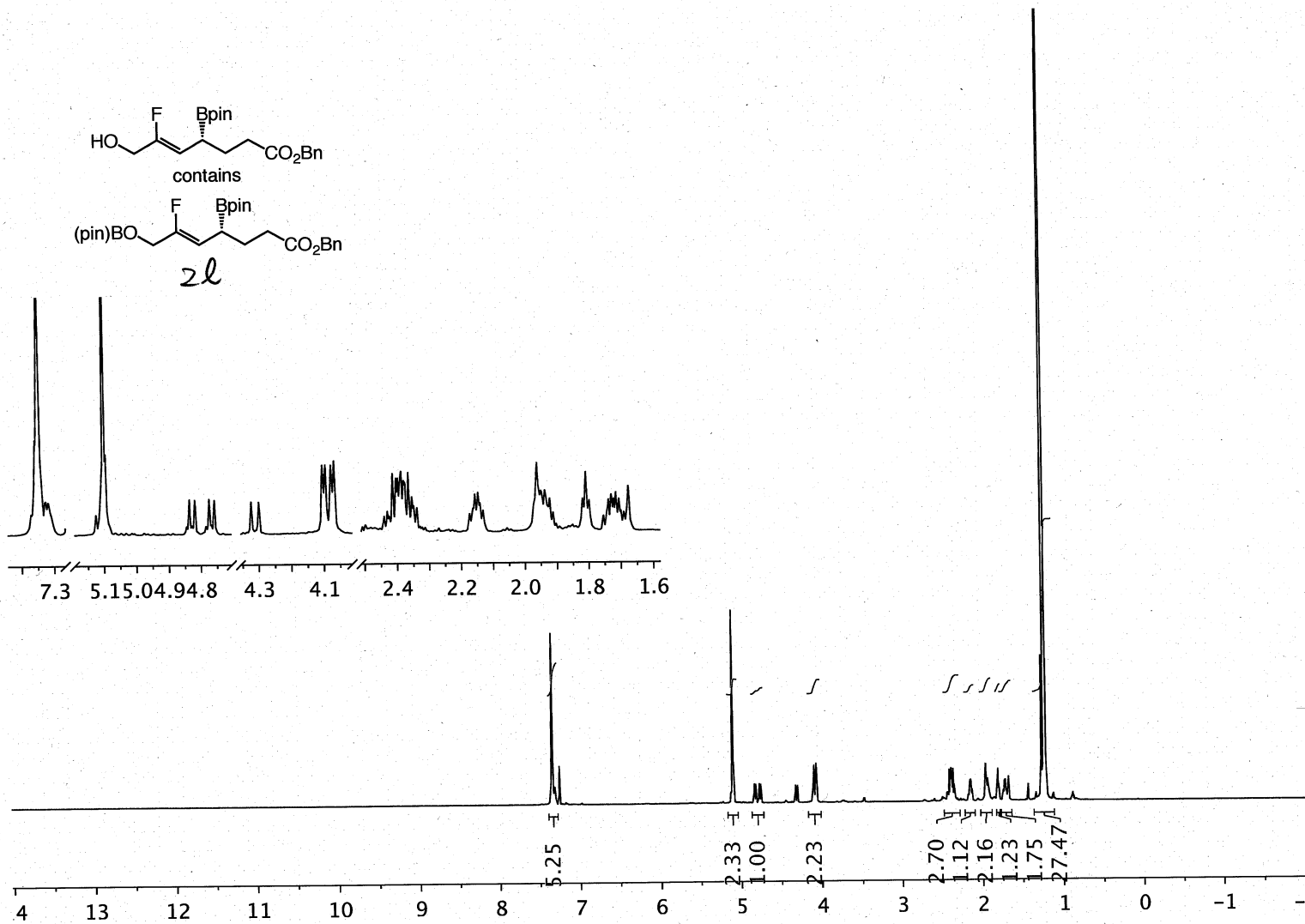
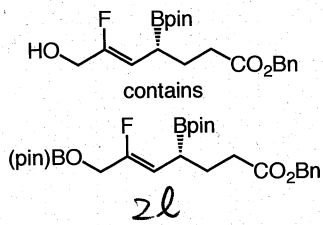
73.02  
69.41

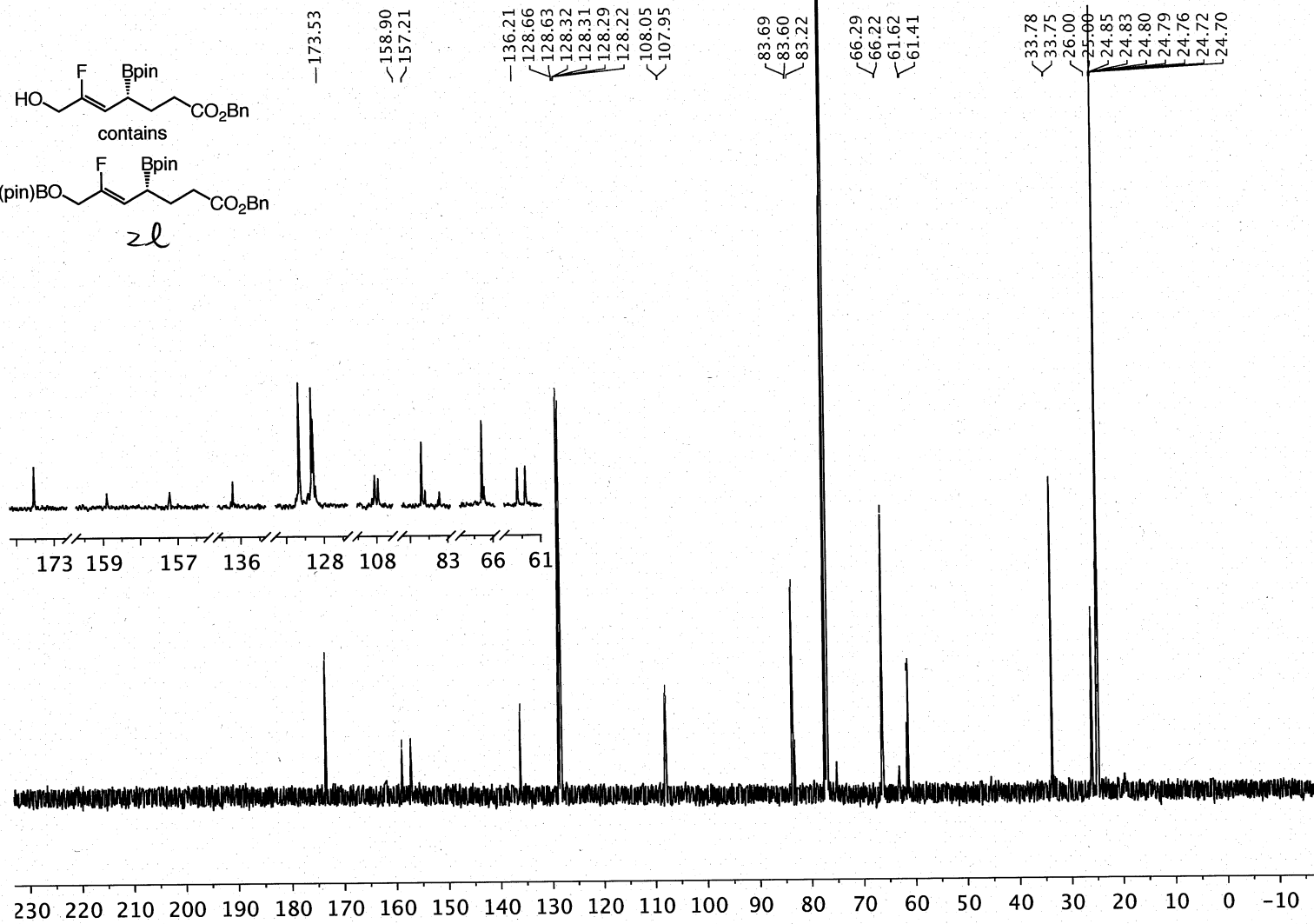
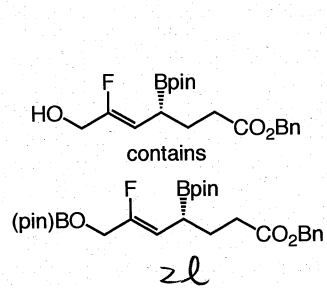
61.75  
61.50

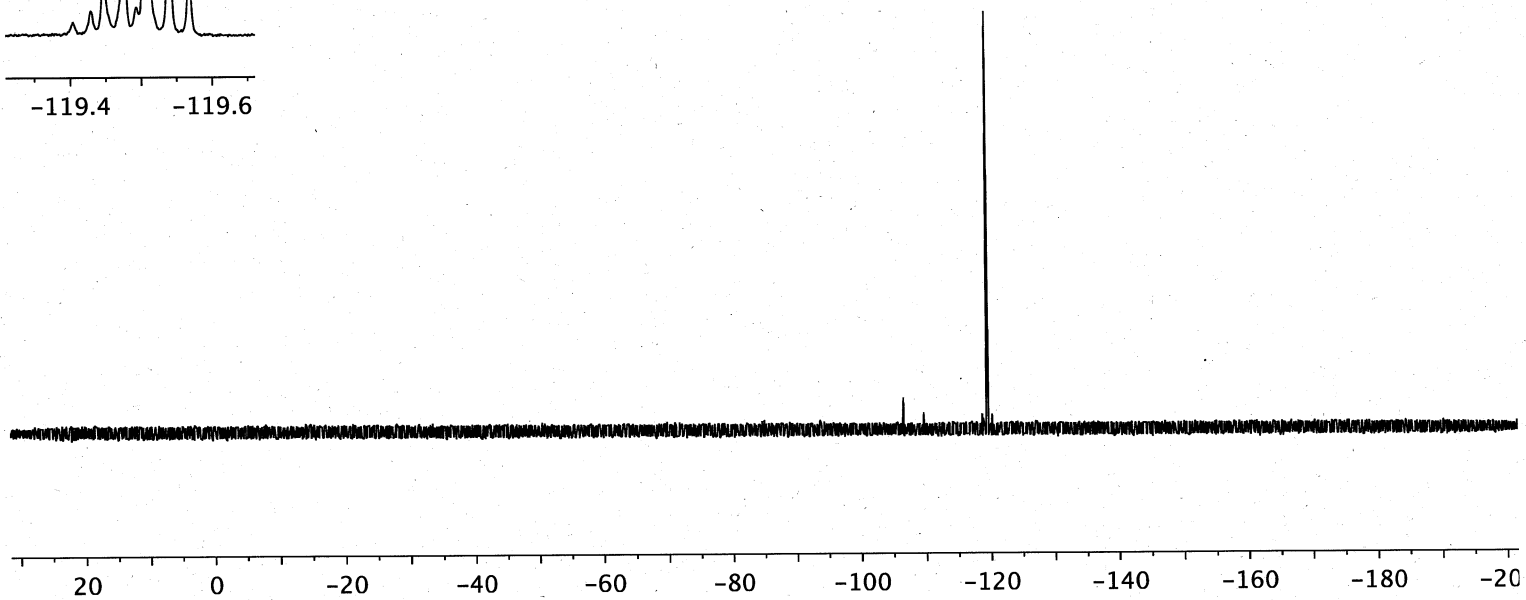
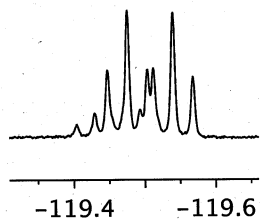
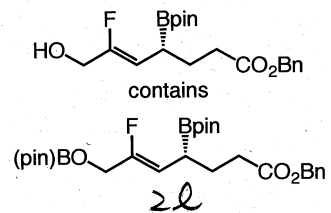
31.74  
30.96  
30.94  
24.81  
24.77  
22.80  
14.26



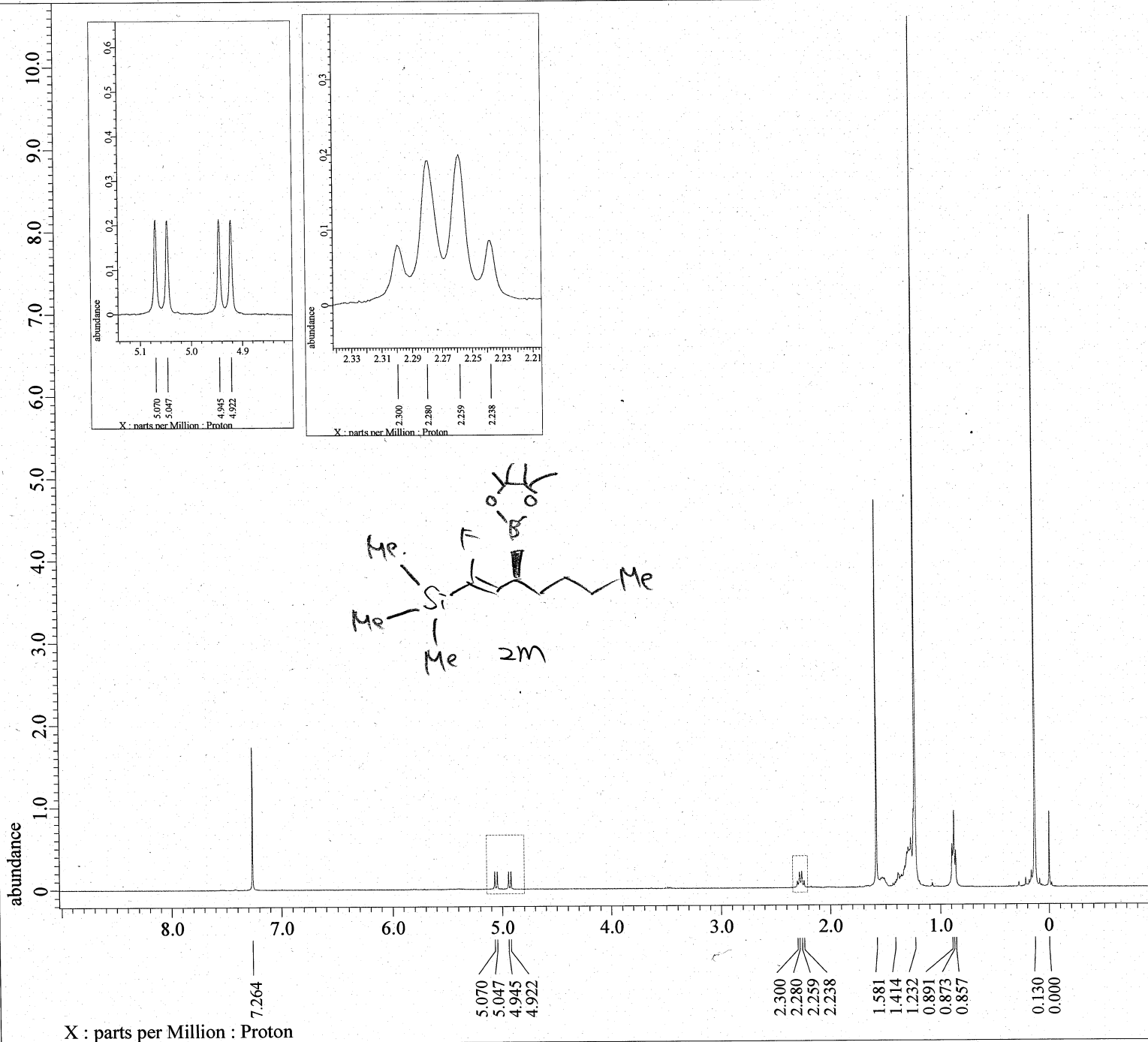










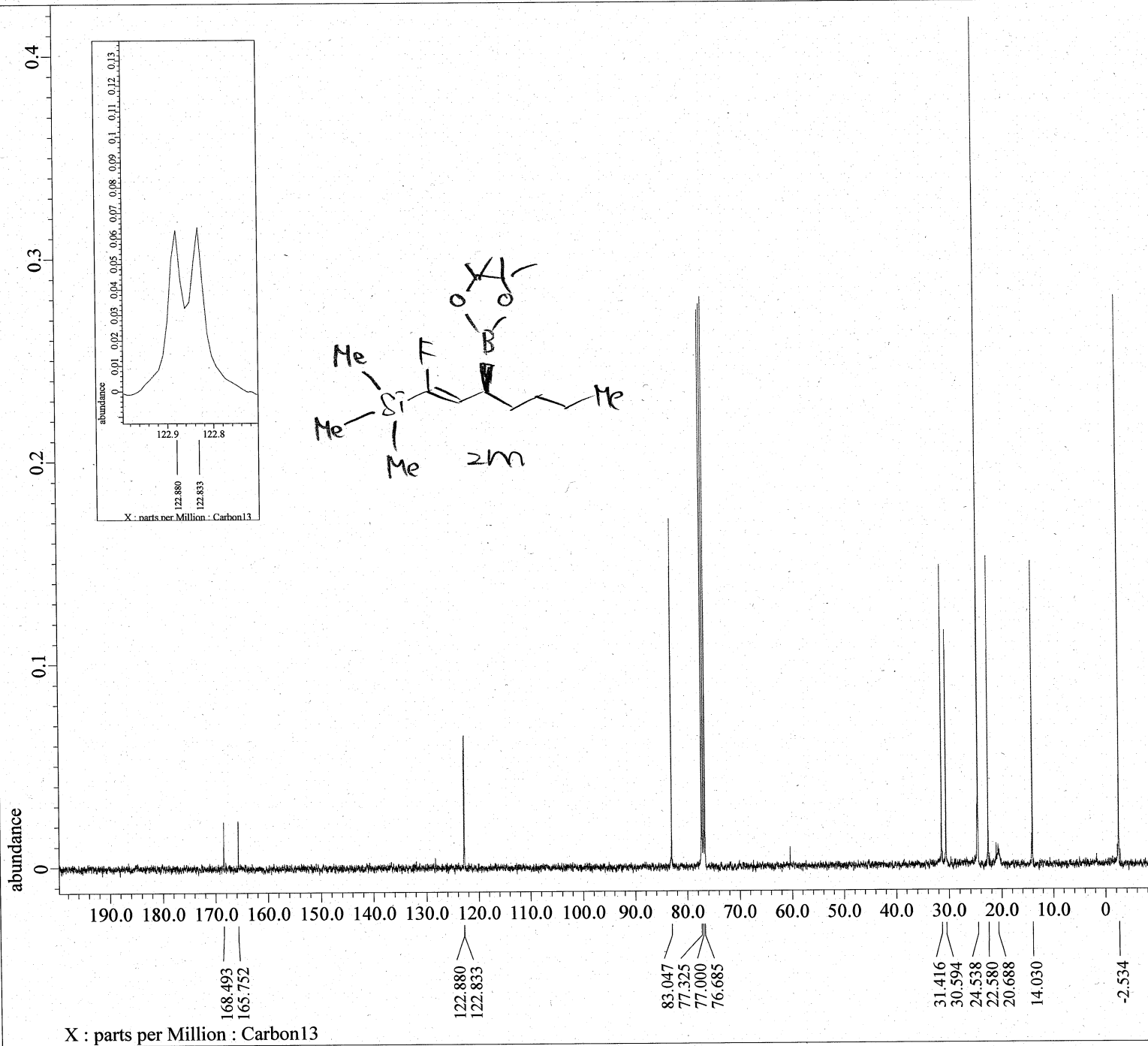


Filename = AKY642-pure\_Proton-1-3.jd  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY642-pure  
Solvent = CHLOROFORM-D  
Actual Start Time = 9-AUG-2018 09:38:52  
Revision Time = 23-MAR-2019 17:24:48

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814 [T] (400 [MHz])  
X Acq Duration = 2.18103808 [s]  
X Domain = 1H  
X Freq = 400.53219825 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45849727 [Hz]  
X Sweep = 7.51201923 [kHz]  
X Sweep Clipped = 6.00961538 [kHz]  
Irr Domain = Proton  
Irr Freq = 400.53219825 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = Proton  
Tri Freq = 400.53219825 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5 [s]  
Recvr Gain = 42  
Temp Get = 19.8 [dC]  
X 90 Width = 6.22 [us]  
X Acq Time = 2.18103808 [s]  
X Angle = 45 [deg]  
X Atn = 0.8 [dB]  
X Pulse = 3.11 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.18103808 [s]



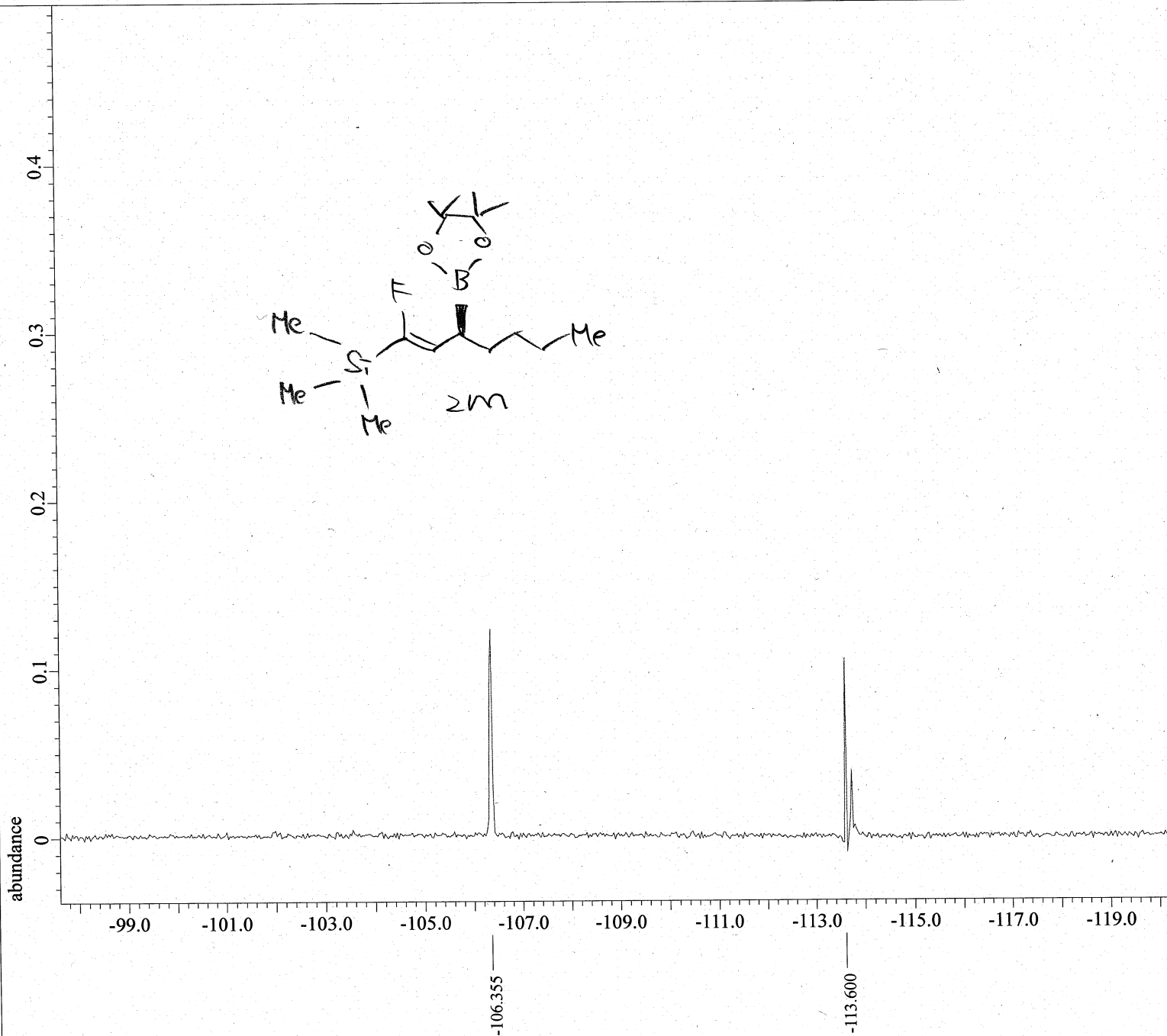
Filename = AKY642-carbon-512\_Carbon-  
Author = element  
Experiment = carbon.jxp  
Sample Id = AKY642-carbon-512  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 9-MAR-2019 17:11:03  
Revision\_Time = 23-MAR-2019 17:26:13

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = Carbon  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X Acq\_Duration = 1.04333312[s]  
X Domain = 13C  
X Freq = 100.33735165[MHz]  
X Offset = 100.0[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95846665[Hz]  
X Sweep = 31.40703518[kHz]  
X Sweep Clipped = 25.12562814[kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754[MHz]  
Irr Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 512  
Total\_Scans = 512

Relaxation\_Delay = 2[s]  
Recvr Gain = 50  
Temp Get = 19.3[dC]  
X 90\_Width = 10.9[us]  
X Acq Time = 1.04333312[s]  
X Angle = 30[deg]  
X Atn = 5.4[dB]  
X Pulse = 3.63333333[us]  
Irr Atn Dec = 25.823[dB]  
Irr Atn Noe = 25.823[dB]  
Irr Noise = WALTZ  
Irr Pwidth = 0.115[ms]  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.04333312[s]

X : parts per Million : Carbon13



Filename = AKY642-pure-FNMR-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample\_Id = S#591976  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 10-MAR-2019 00:57:12  
Revision\_Time = 23-MAR-2019 17:28:57

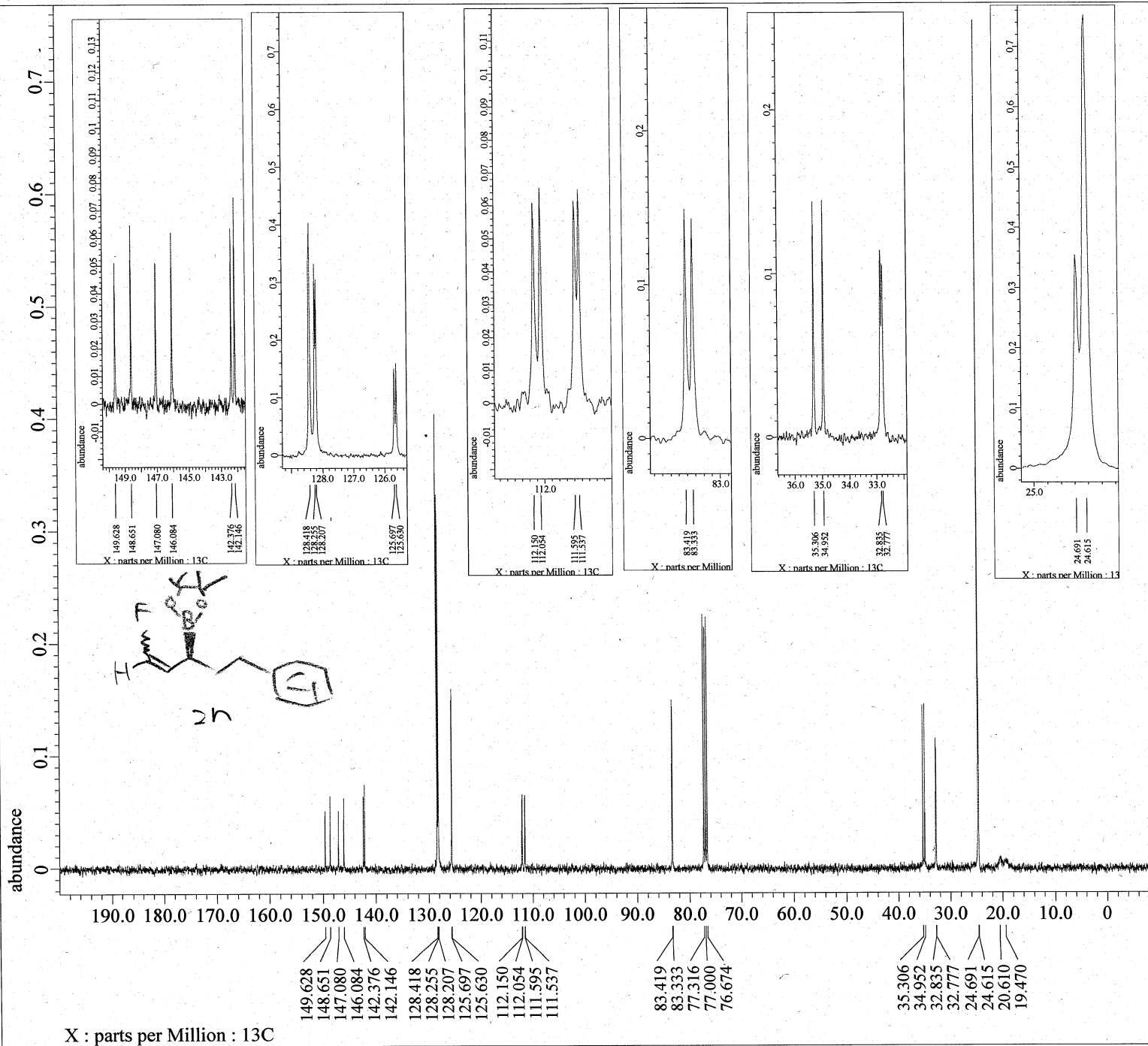
Comment = single\_pulse  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 13107  
X\_Domain = 19F  
Dim\_Title = 19F  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 24  
Temp\_Get = 21[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F





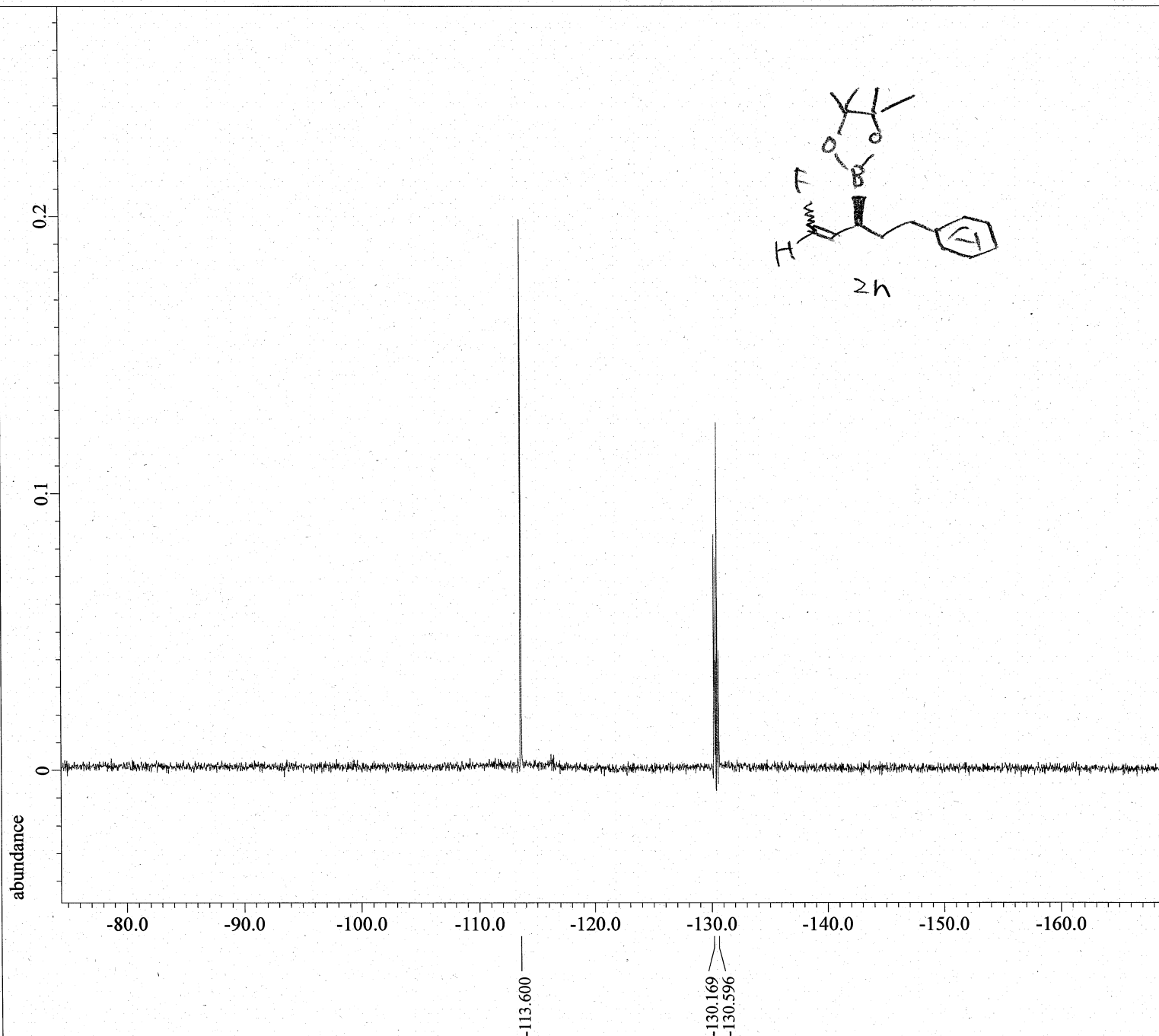
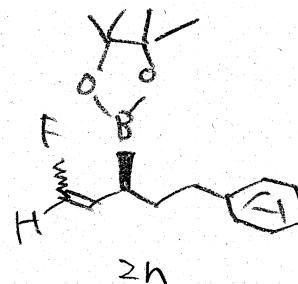
Filename = AKY748-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample\_Id = S#338232  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 20-MAR-2019 17:53:40  
Revision\_Time = 24-MAR-2019 16:07:02

Comment = single\_pulse\_decoupled\_ga  
Data\_Format = 1D\_COMPLEX  
Dim\_Size = 26214  
X\_Domain = 13C  
Dim\_Title = 13C  
Dim\_Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 1.048576[s]  
X\_Domain = 13C  
X\_Freq = 99.54517646[MHz]  
X\_Offset = 100[ppm]  
X\_Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.95367432[Hz]  
X\_Sweep = 31.25[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 395.88430144[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 128  
Total\_Scans = 128

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 48  
Temp\_Get = 21.4[ $^{\circ}$ C]  
X\_90\_Width = 10.1[ $\mu$ s]  
X\_Acq\_Time = 1.048576[s]  
X\_Angle = 30[deg]  
X\_Atn = 3.4[dB]  
X\_Pulse = 3.36666667[ $\mu$ s]  
Irr\_Atn\_Dec = 22.3[dB]  
Irr\_Atn\_Noise = 22.3[dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.048576[s]

X : parts per Million : 13C

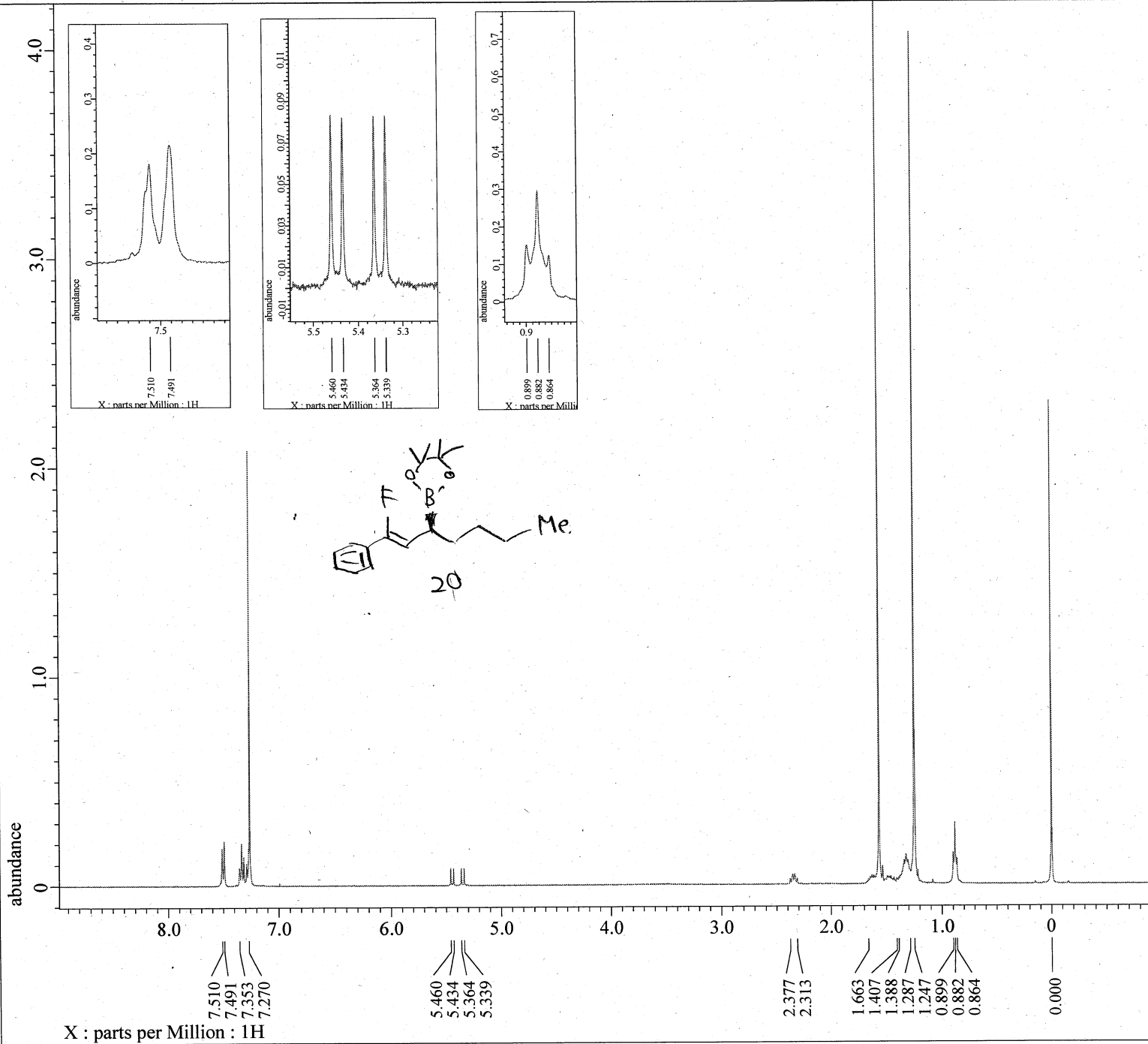


Filename = AKY748-fnmr-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#351315  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 20-MAR-2019 18:16:14  
Revision\_Time = 24-MAR-2019 15:44:57

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 21[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]



Filename = AKY657-pure-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#513078  
Solvent = CHLOROFORM-D  
Actual Start Time = 4-SEP-2018 21:46:24  
Revision Time = 18-FEB-2019 14:13:07

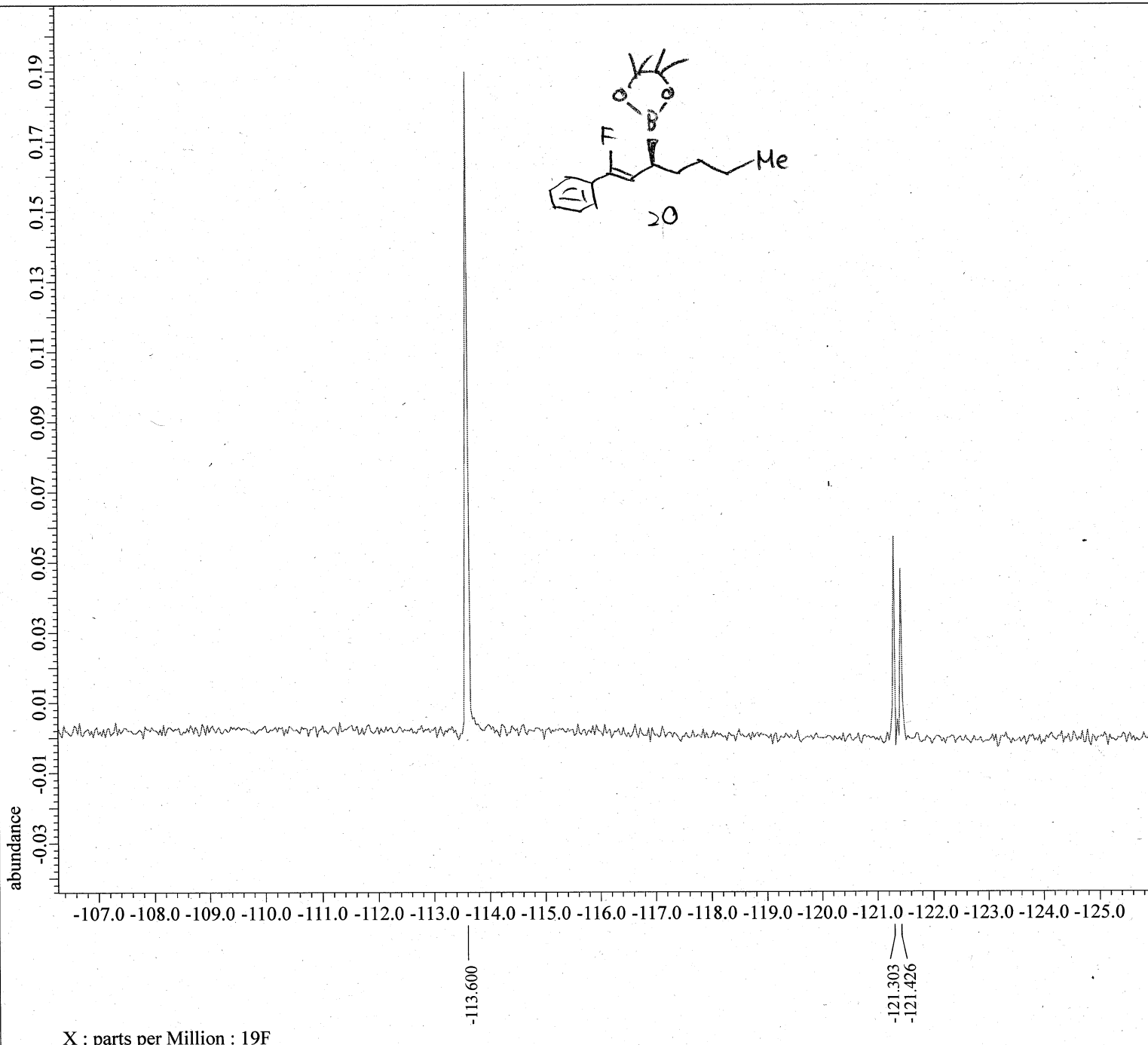
Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441 [MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791 [Hz]  
X Sweep = 7.35294118 [kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441 [MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441 [MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 50  
Temp Get = 20.9 [dC]  
X 90\_Width = 11.04 [us]  
X Acq Time = 2.228224 [s]  
X Angle = 45 [deg]  
X Atn = 1.9 [dB]  
X Pulse = 5.52 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.228224 [s]







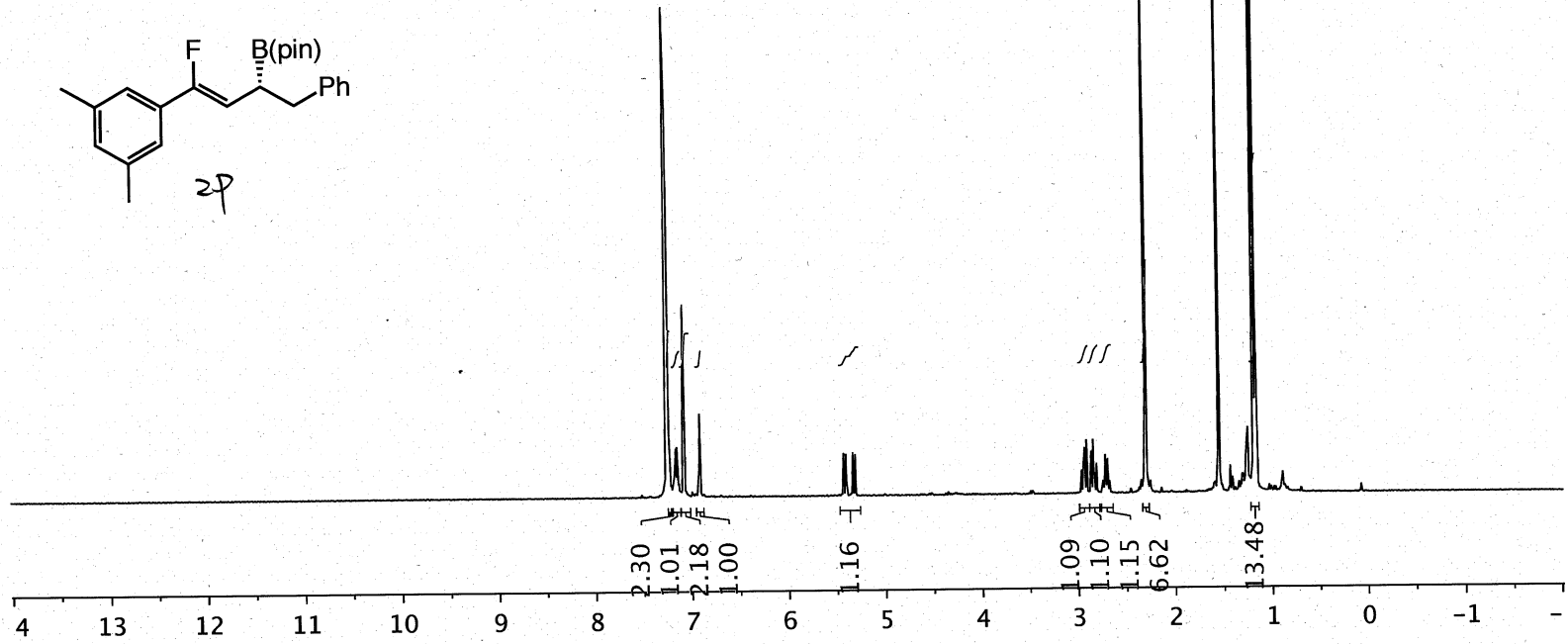
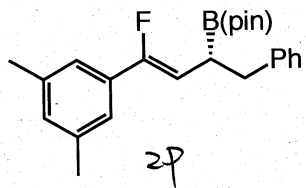
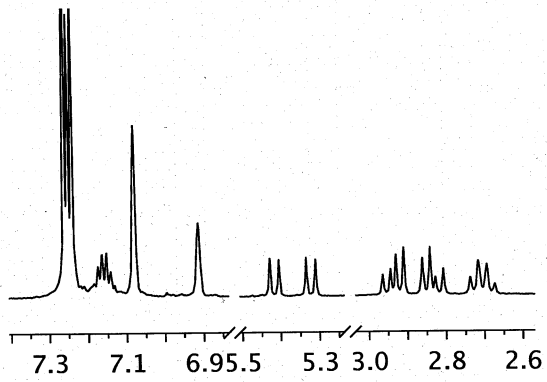
Filename = AKY657-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#545743  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 4-SEP-2018 23:44:47  
Revision\_Time = 18-FEB-2019 14:35:43

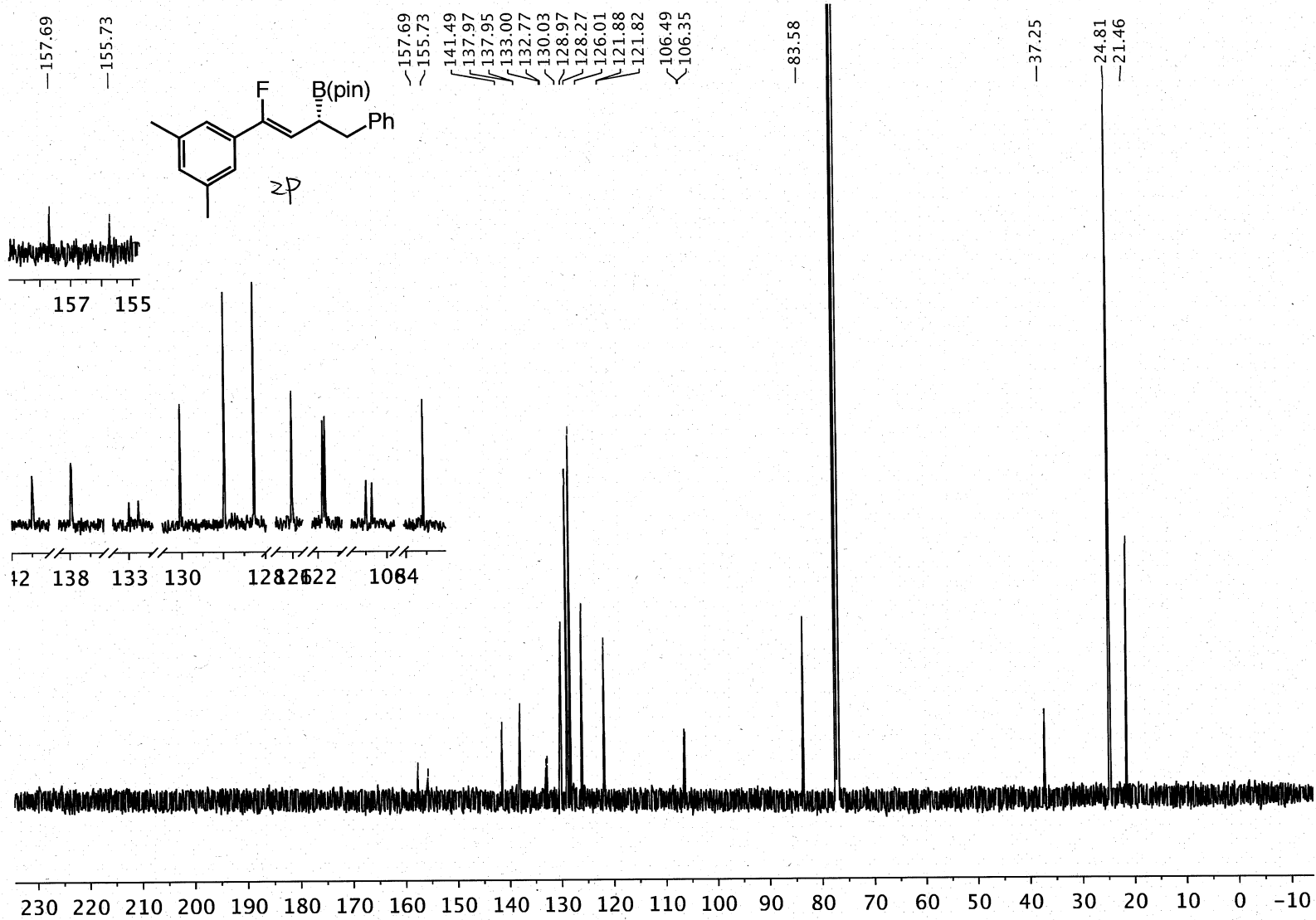
Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

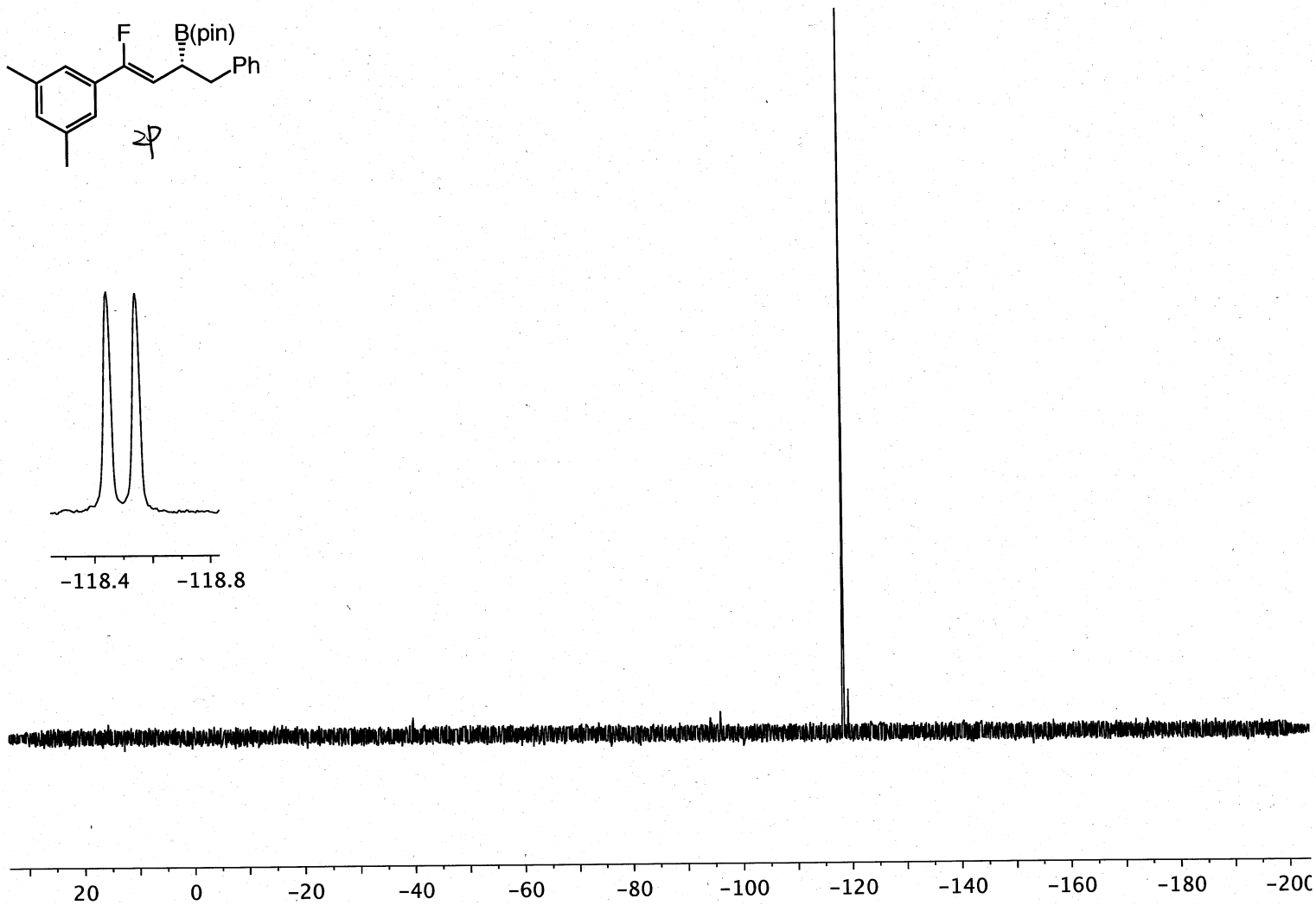
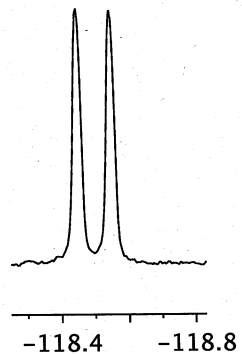
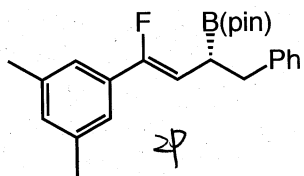
Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

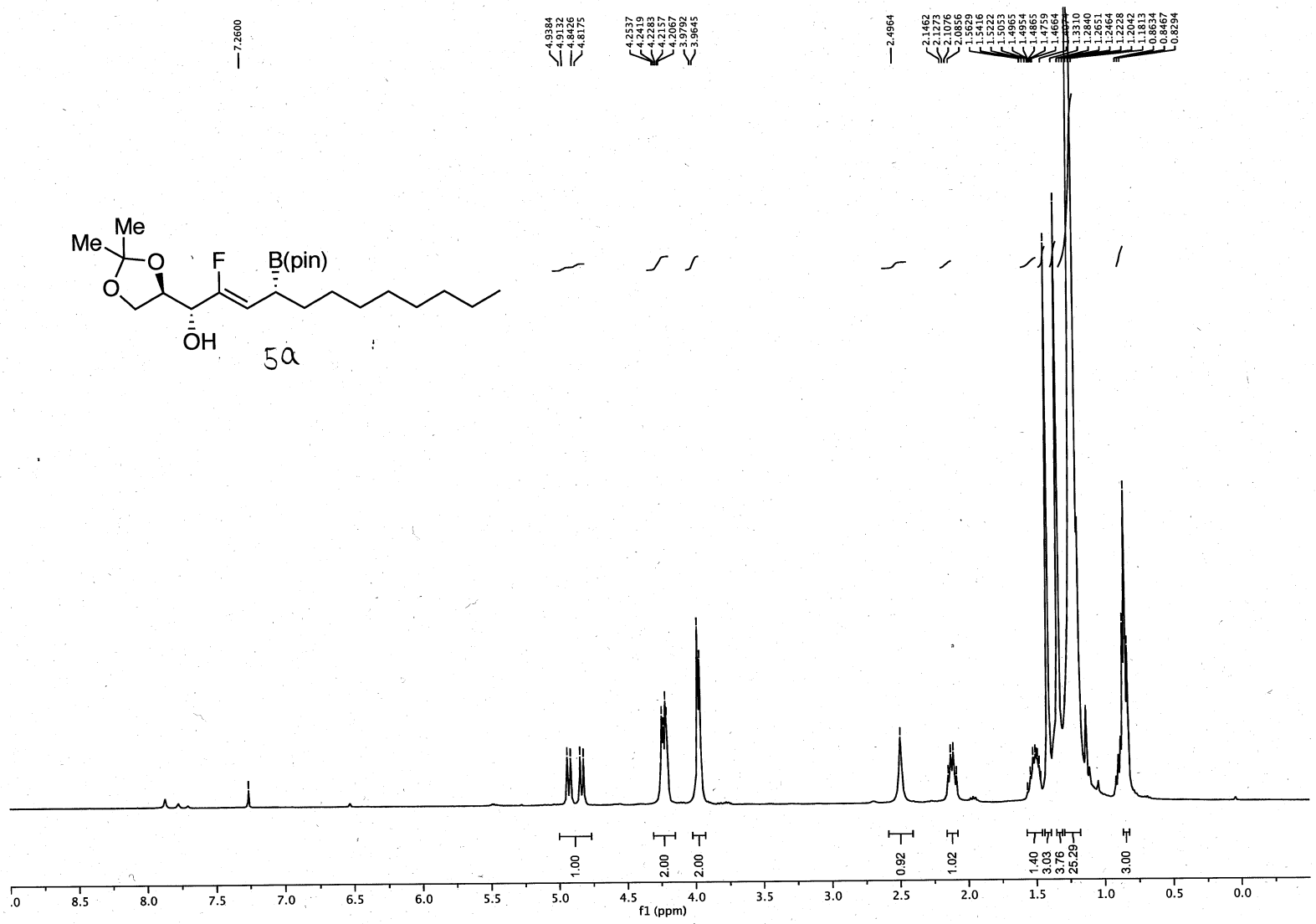
Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 22.5[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

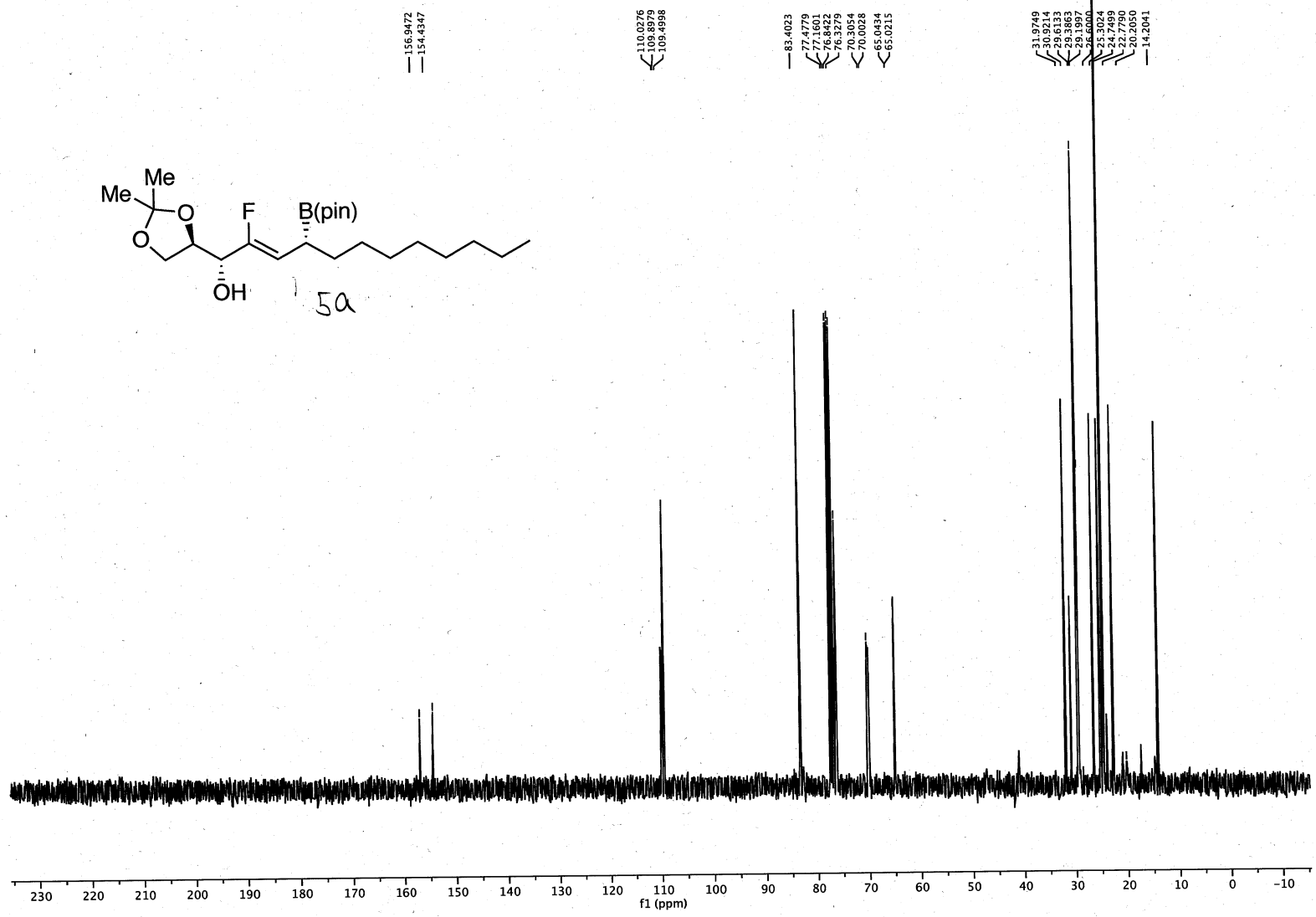
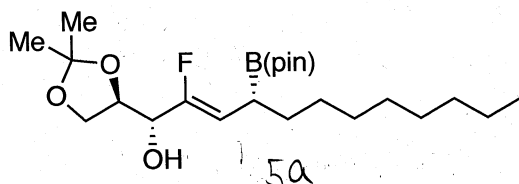
X : parts per Million : 19F

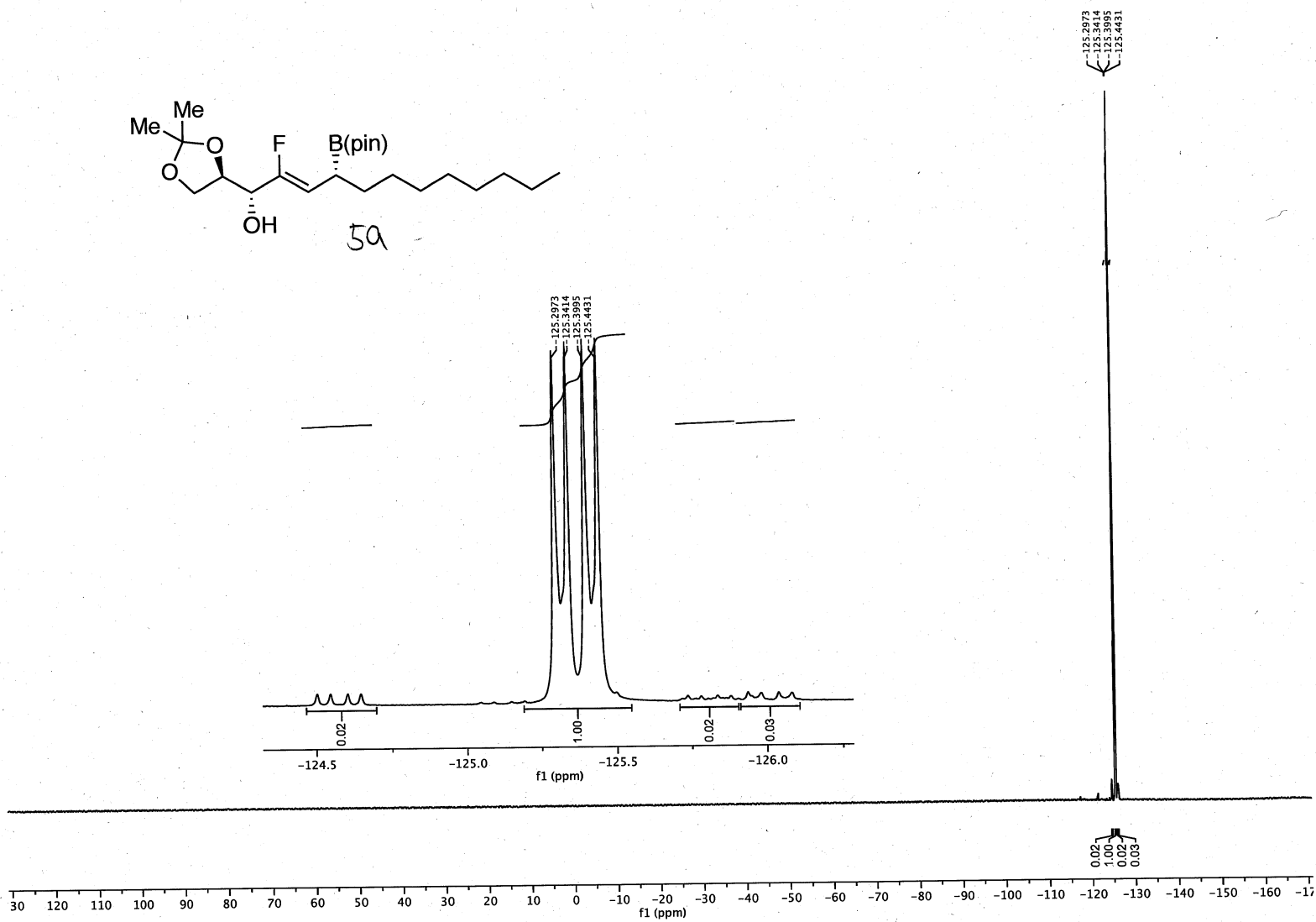
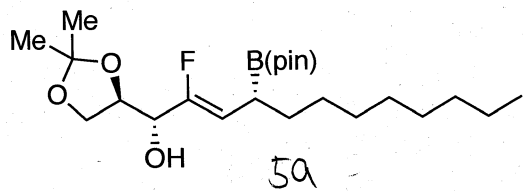


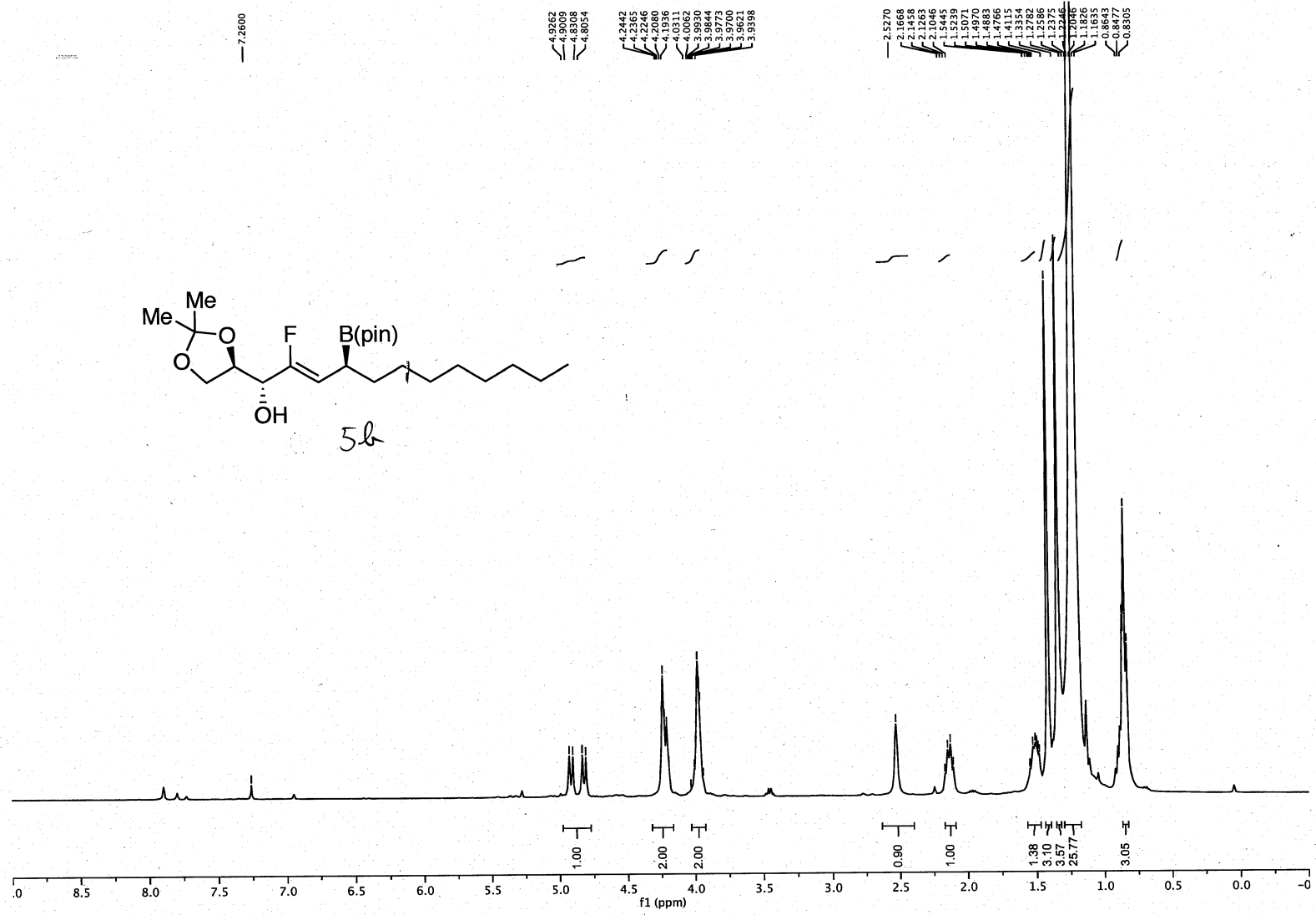




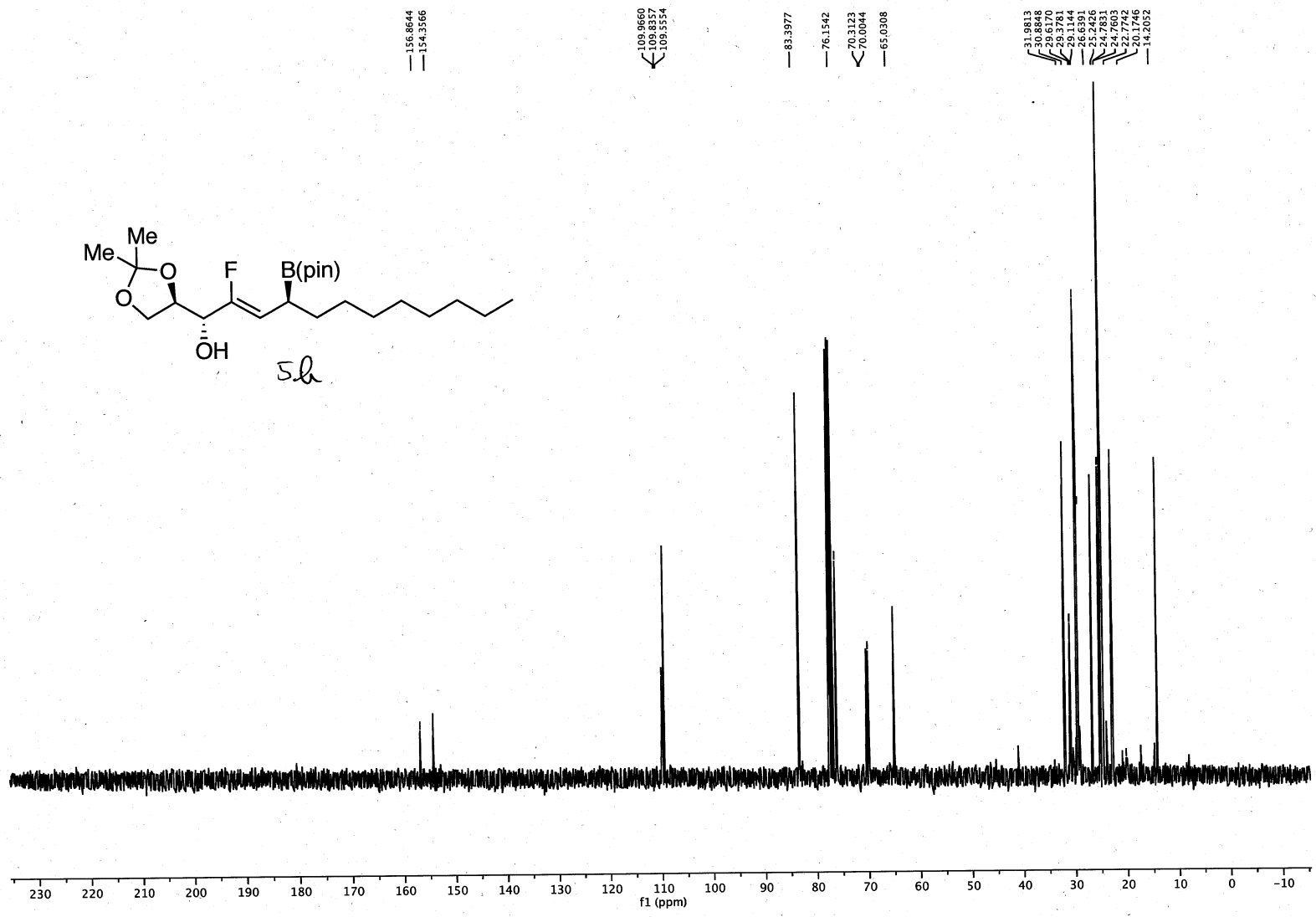
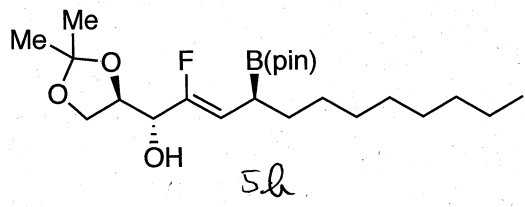


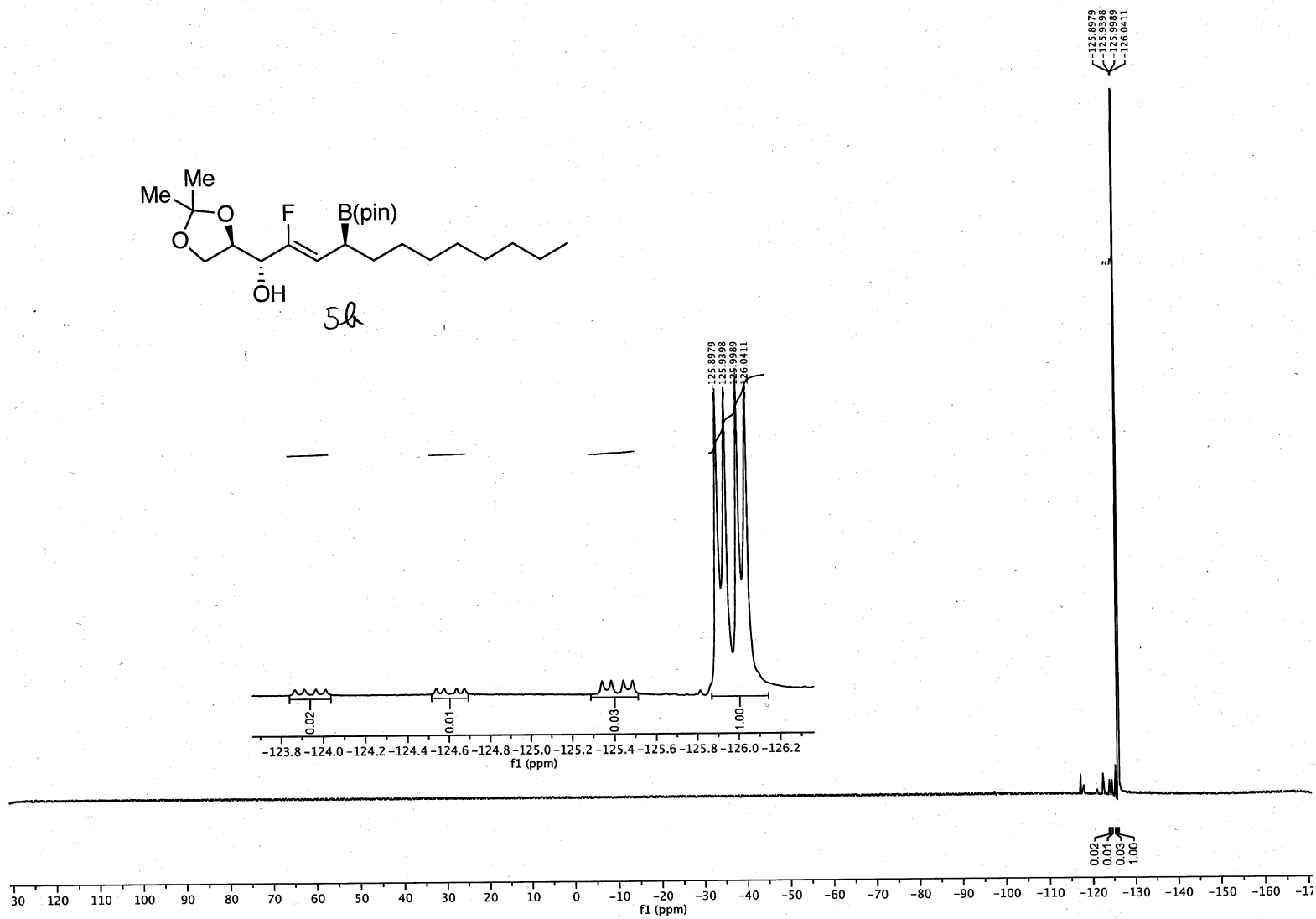
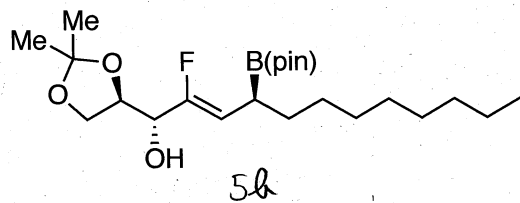


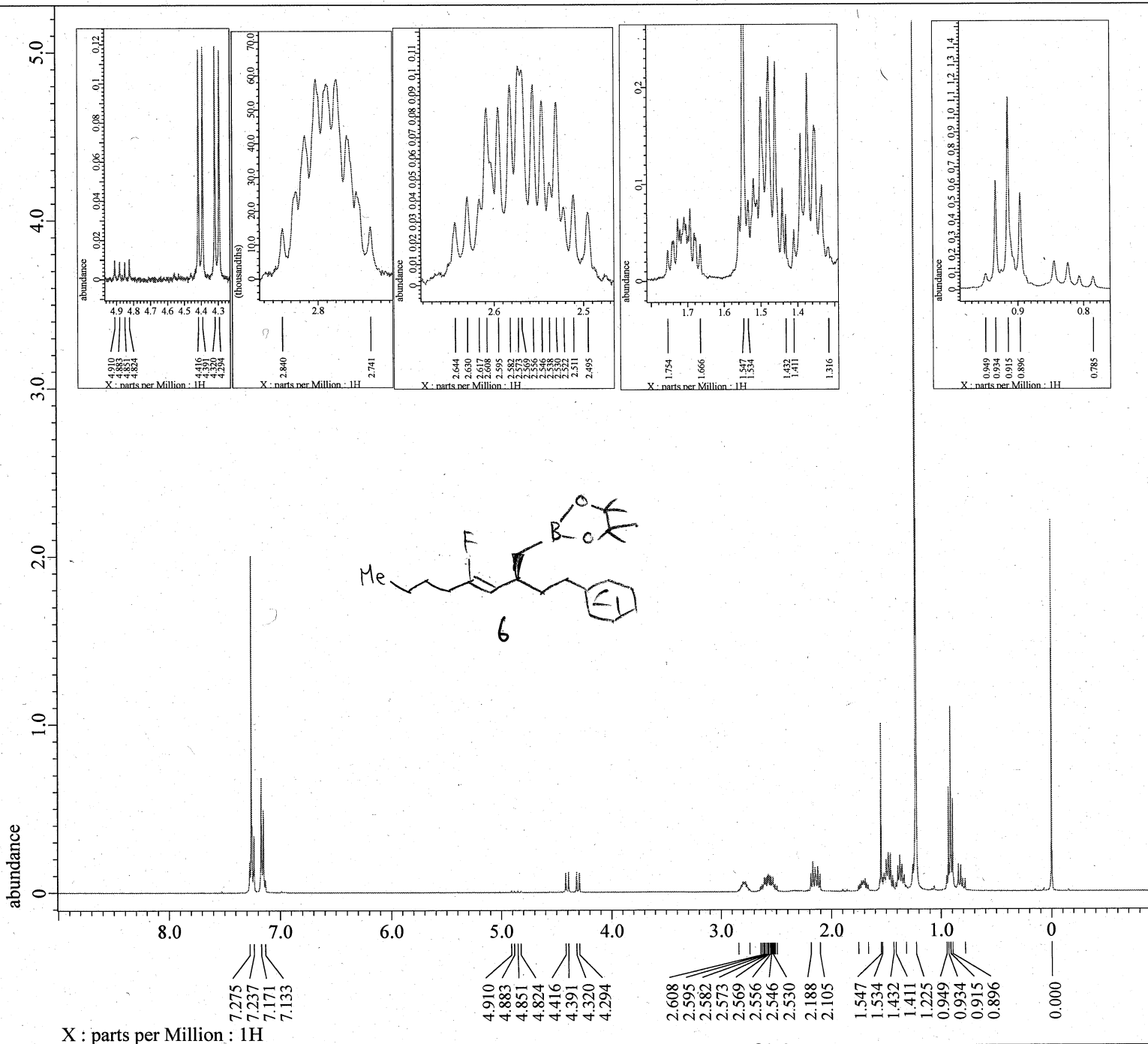










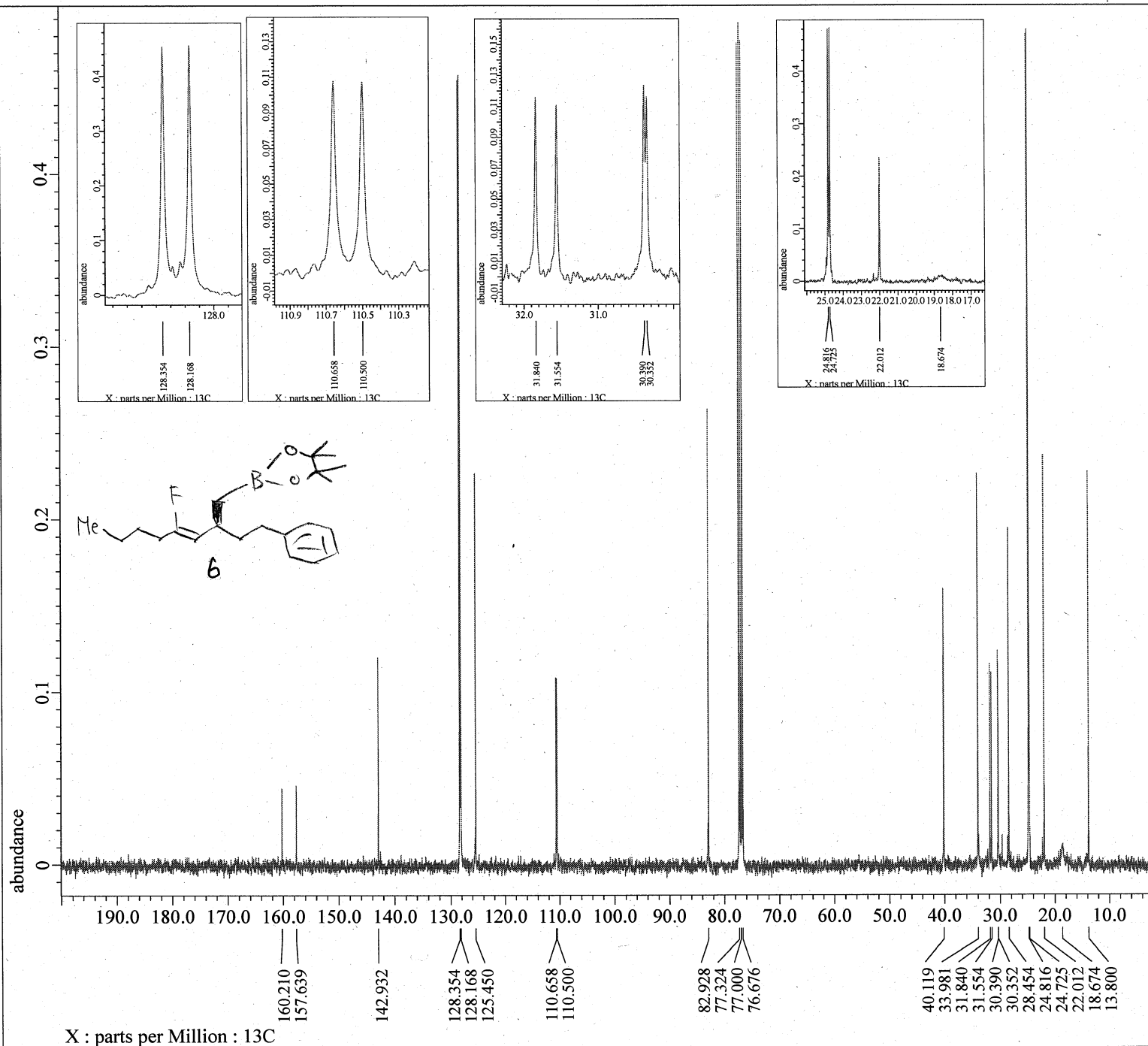


Filename = AKY730-pure-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#412633  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 17-NOV-2018 18:53:49  
Revision\_Time = 17-NOV-2018 14:52:39

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 48  
Temp Get = 21.5[dC]  
X\_90\_Width = 11.04[us]  
X Acq Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[dB]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 7.228224[s]

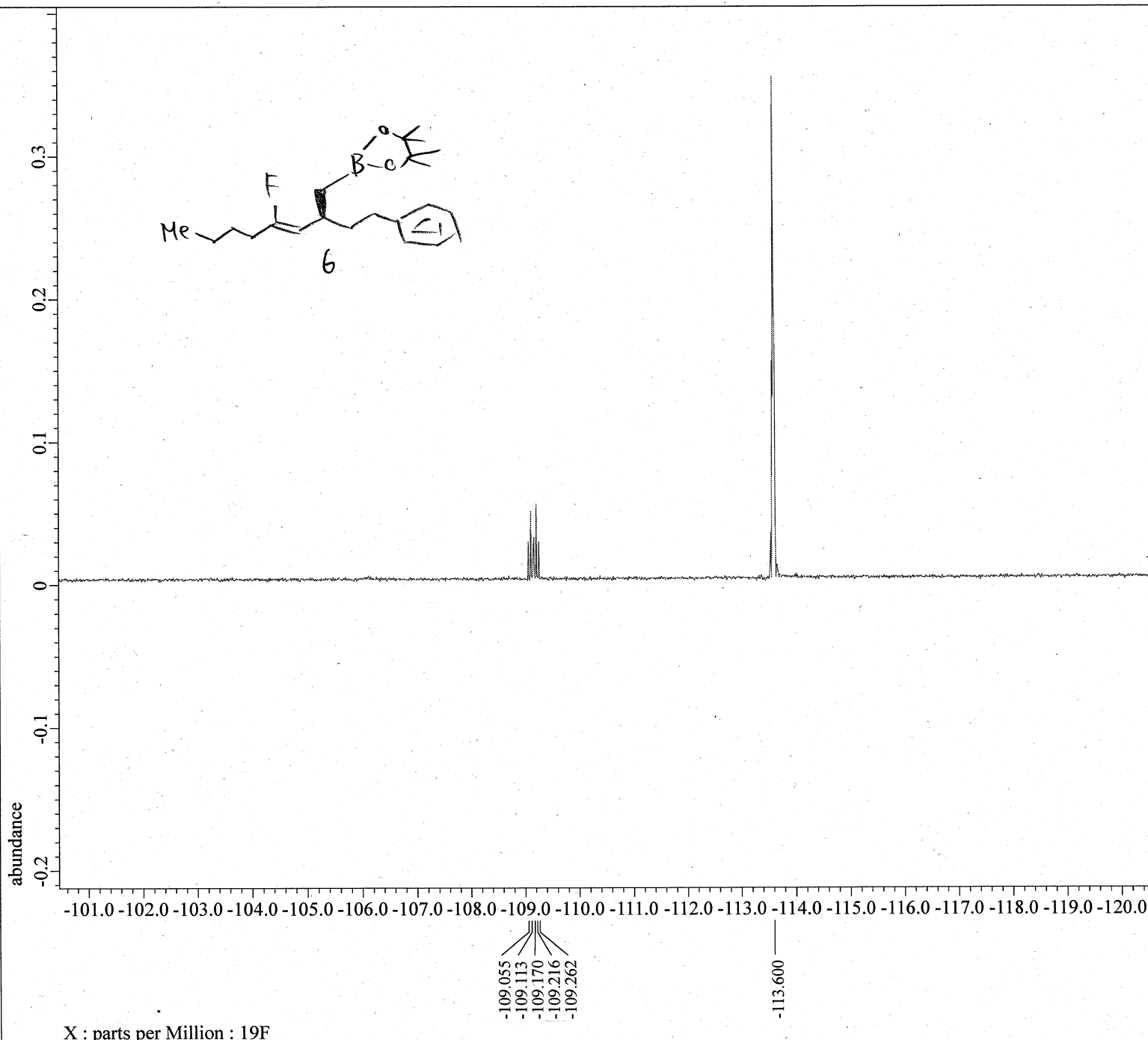


Filename = AKY730-carbon-3.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#417834  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 17-NOV-2018 19:01:22  
Revision\_Time = 17-NOV-2018 13:23:05

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 52428  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 1.06430464[s]  
X Domain = 13C  
X\_Freq = 98.51479726[MHz]  
X\_Offset = 100[ppm]  
X Points = 32768  
X\_Prescans = 4  
X\_Resolution = 0.93958061[Hz]  
X\_Sweep = 30.78817734[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 391.78655441[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total\_Scans = 256

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 60  
Temp\_Get = 21.9[dc]  
X\_90\_Width = 9.11[us]  
X\_Acq\_Time = 1.06430464[s]  
X\_Angle = 30[deg]  
X\_Atn = 4.9[dB]  
X\_Pulse = 3.03666667[us]  
Irr\_Atn\_Dec = 22.255[dB]  
Irr\_Atn\_Noise = 22.255[dB]  
Irr\_Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.06430464[s]



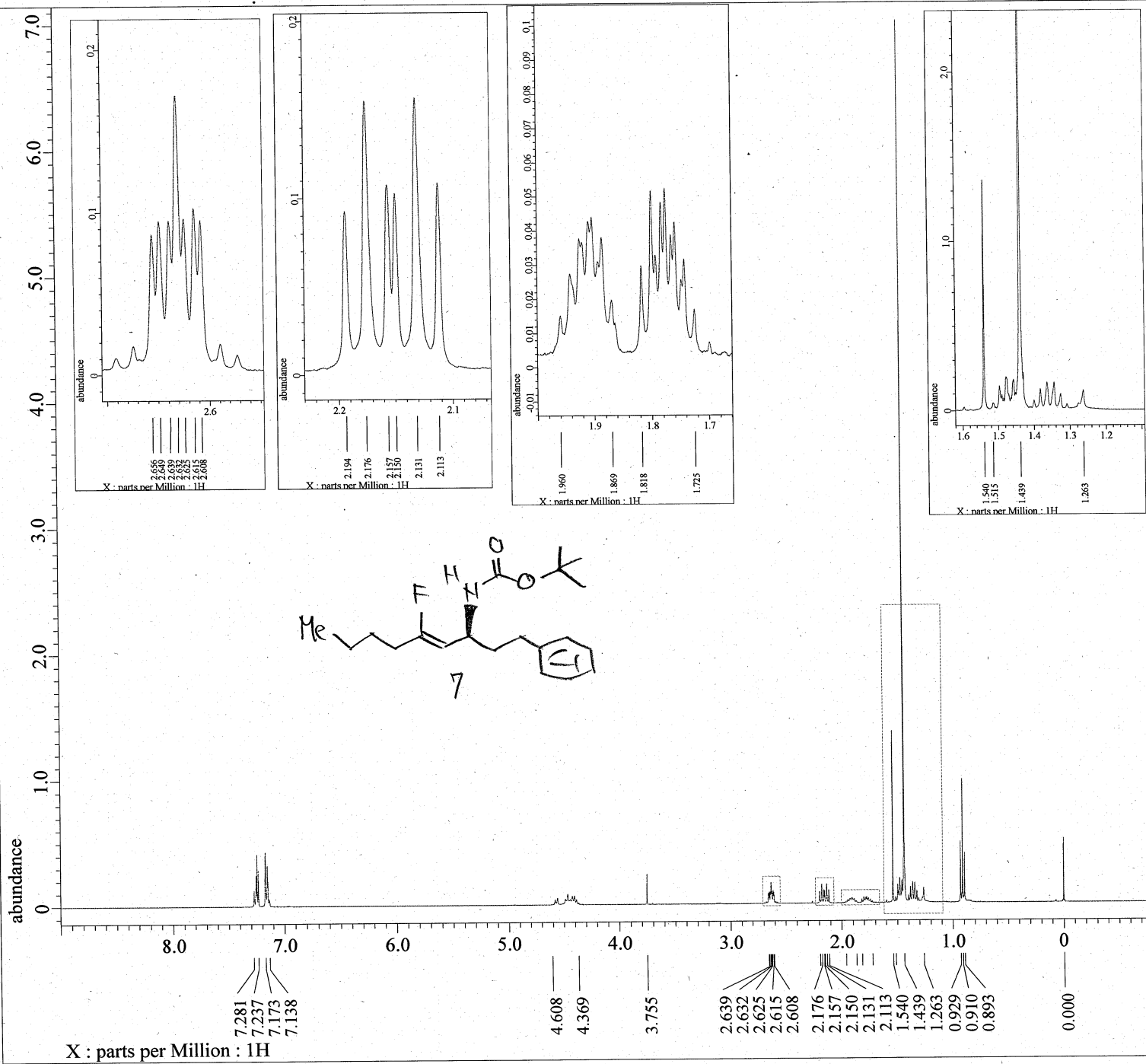
Filename = AKY730-pure-fnmr-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#442060  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 17-NOV-2018 20:46:10  
Revision\_Time = 17-NOV-2018 13:28:53

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 0.23330816[s]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = -60[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 4.2861767[Hz]  
X Sweep = 70.2247191[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 16  
Total\_Scans = 16

Relaxation\_Delay = 5[s]  
Recvr Gain = 30  
Temp Get = 22[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 0.23330816[s]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 5.23330816[s]

X : parts per Million : 19F

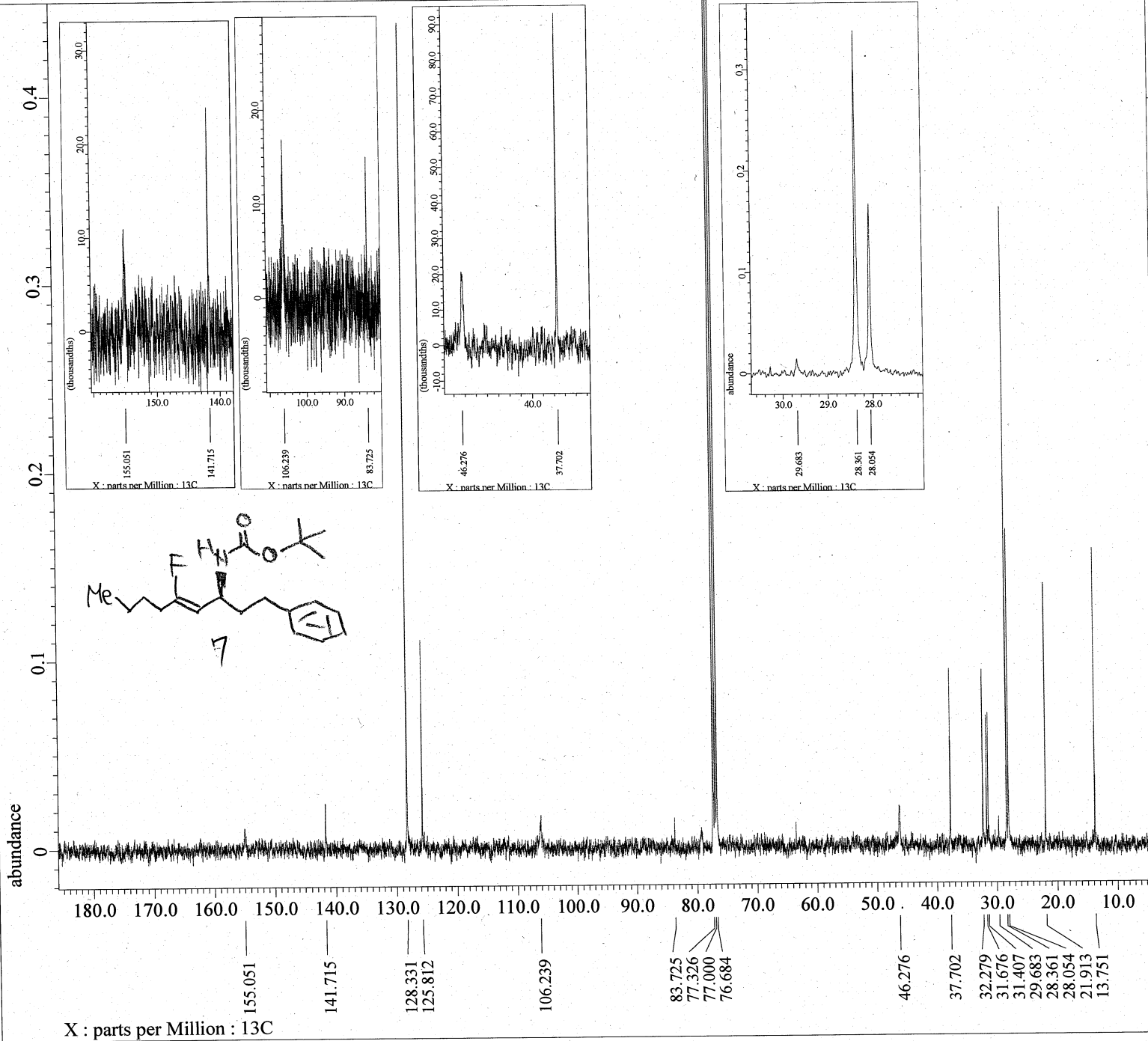


Filename = AKY763-pure-60-3.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#537868  
Solvent = CHLOROFORM-D  
Actual Start Time = 13-DEC-2018 23:26:28  
Revision Time = 25-MAR-2019 10:50:08

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 2.20725248[s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 36  
Temp Get = 60[dc]  
X 90 Width = 13.2[us]  
X Acq Time = 2.20725248[s]  
X Angle = 45[deg]  
X Atn = 3.5[dB]  
X Pulse = 6.6[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.20725248[s]

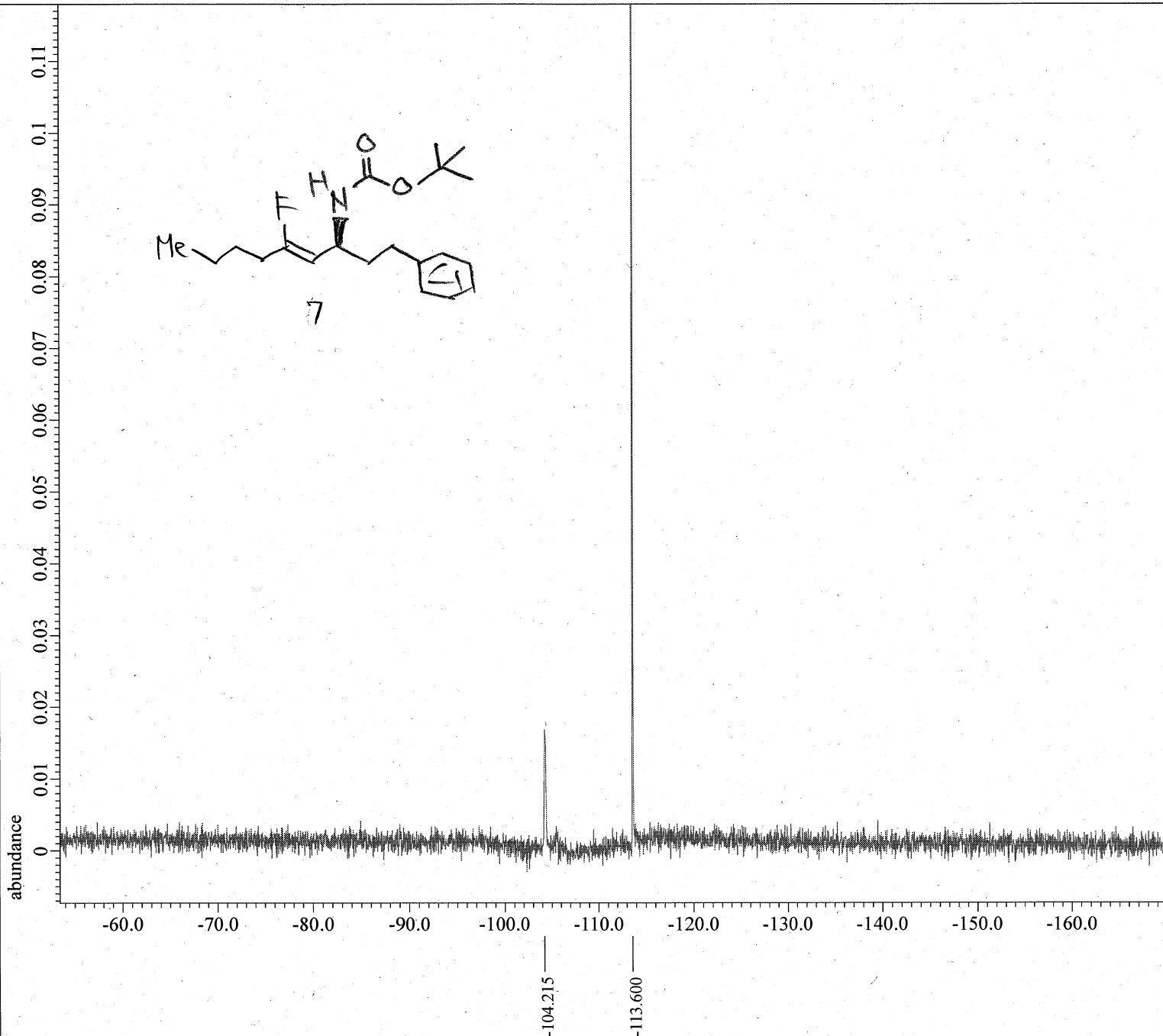


Filename = AKY763-carbonag-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#794162  
Solvent = CHLOROFORM-D  
Actual Start Time = 20-DEC-2018 06:33:08  
Revision Time = 25-MAR-2019 13:18:54

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total Scans = 256

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 27.2[dC]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]



Filename = AKY763-pure-FNMR-again-2.  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#749921  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 8-MAR-2019 05:20:40  
Revision\_Time = 25-MAR-2019 10:55:19

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq\_Duration = 87.81824 [ms]  
X Domain = 19F  
X Freq = 372.50336686 [MHz]  
X Offset = 0 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602 [Hz]  
X Sweep = 186.56716418 [kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr Gain = 24  
Temp Get = 19.1 [dC]  
X 90\_Width = 13.9 [us]  
X Acq\_Time = 87.81824 [ms]  
X Angle = 45 [deg]  
X Atn = 4 [dB]  
X Pulse = 6.95 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 5.08781824 [s]

X : parts per Million : 19F



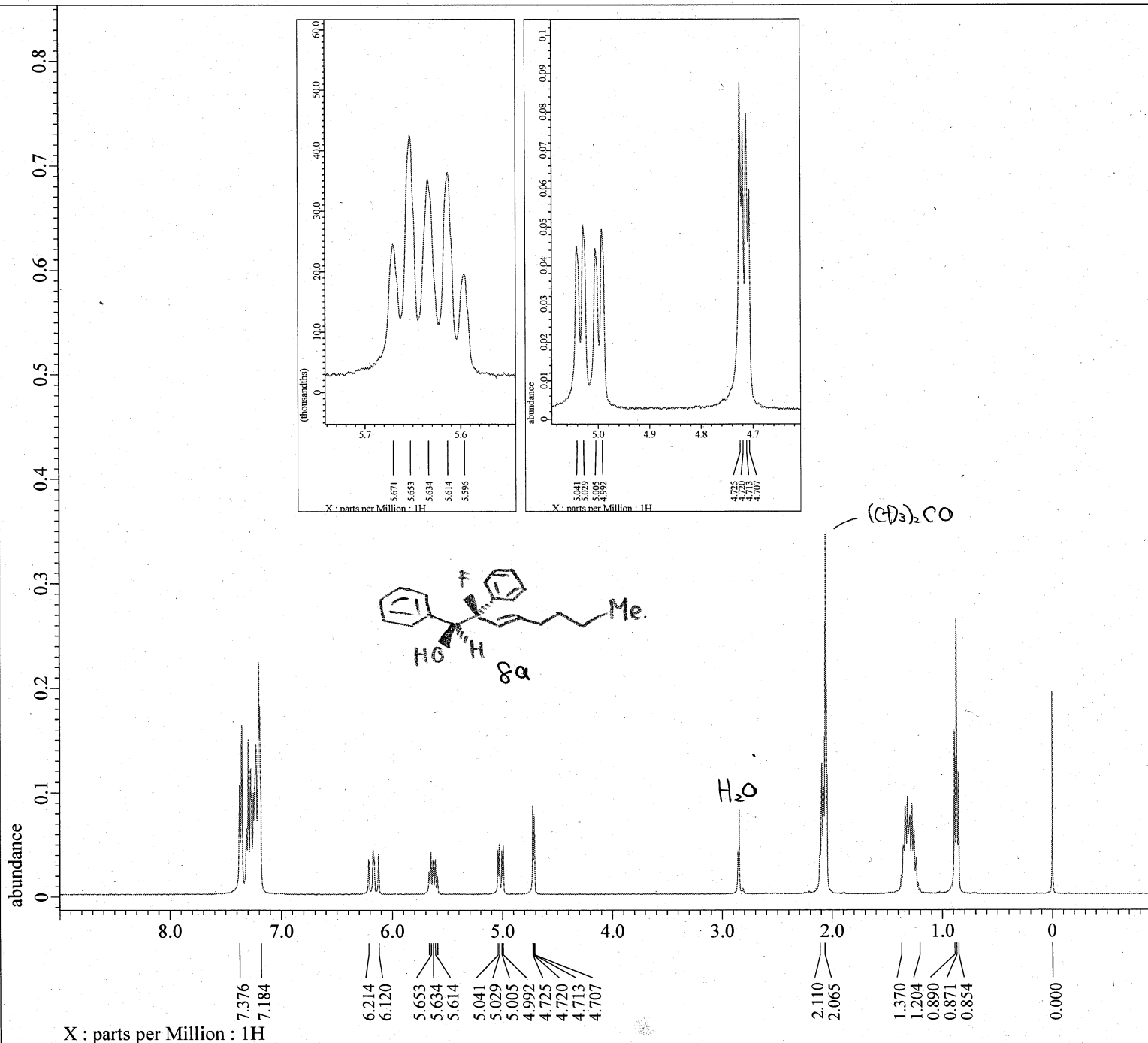


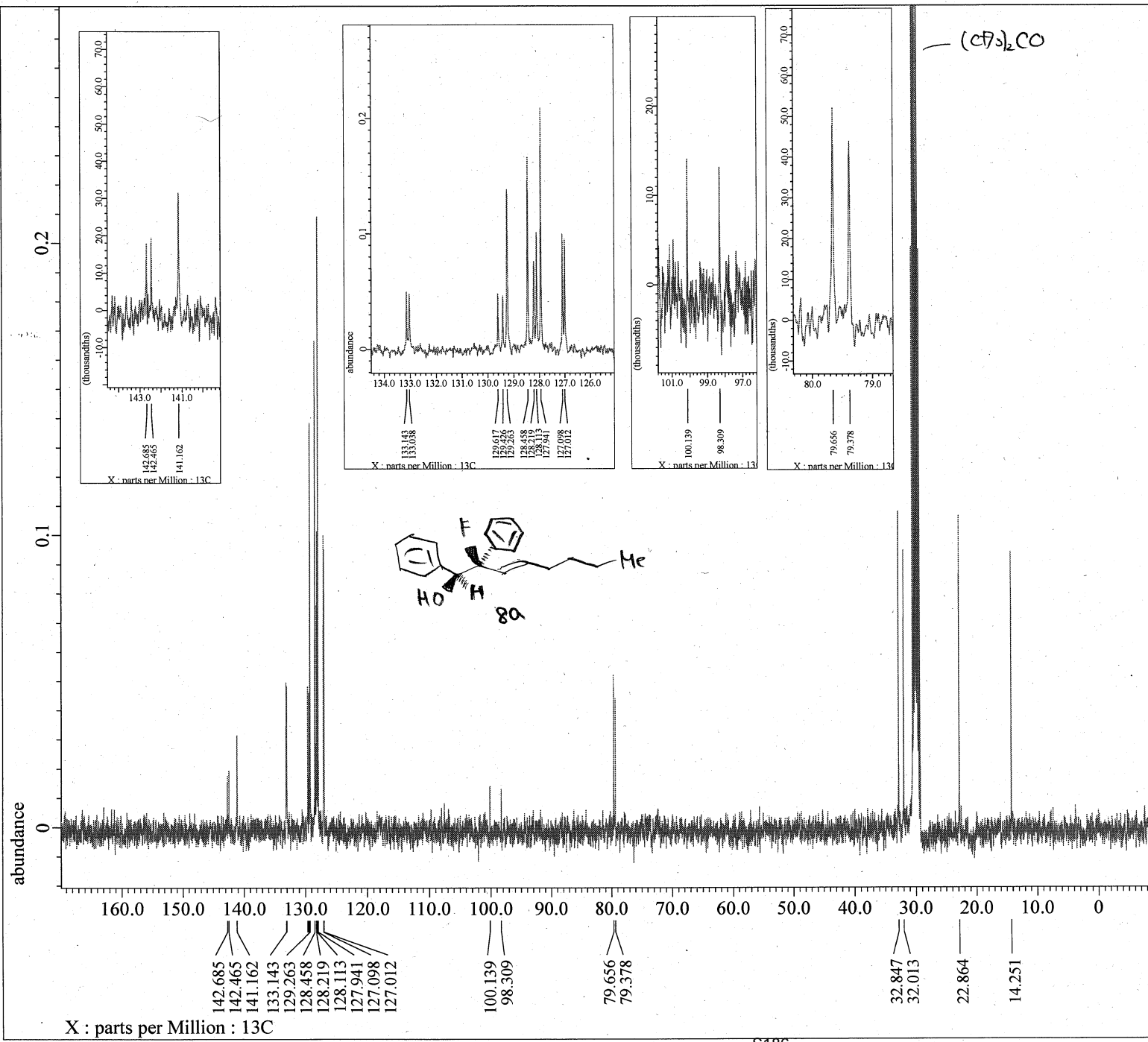
Filename = AKY787-pure-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#650605  
Solvent = ACETONE-D6  
Actual\_Start Time = 13-JAN-2019 02:34:04  
Revision\_Time = 20-FEB-2019 10:31:43

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X\_Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 2.20725248[s]  
X\_Domain = 1H  
X\_Freq = 395.88430144[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45305193[Hz]  
X\_Sweep = 7.42280285[kHz]  
Irr\_Domain = 1H  
Irr\_Freq = 395.88430144[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 1H  
Tri\_Freq = 395.88430144[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 36  
Temp\_Get = 86[dC]  
X\_90\_Width = 13.2[us]  
X\_Acq\_Time = 2.20725248[s]  
X\_Angle = 45[deg]  
X\_Atn = 3.5[dB]  
X\_Pulse = 6.6[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Preat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.20725248[s]

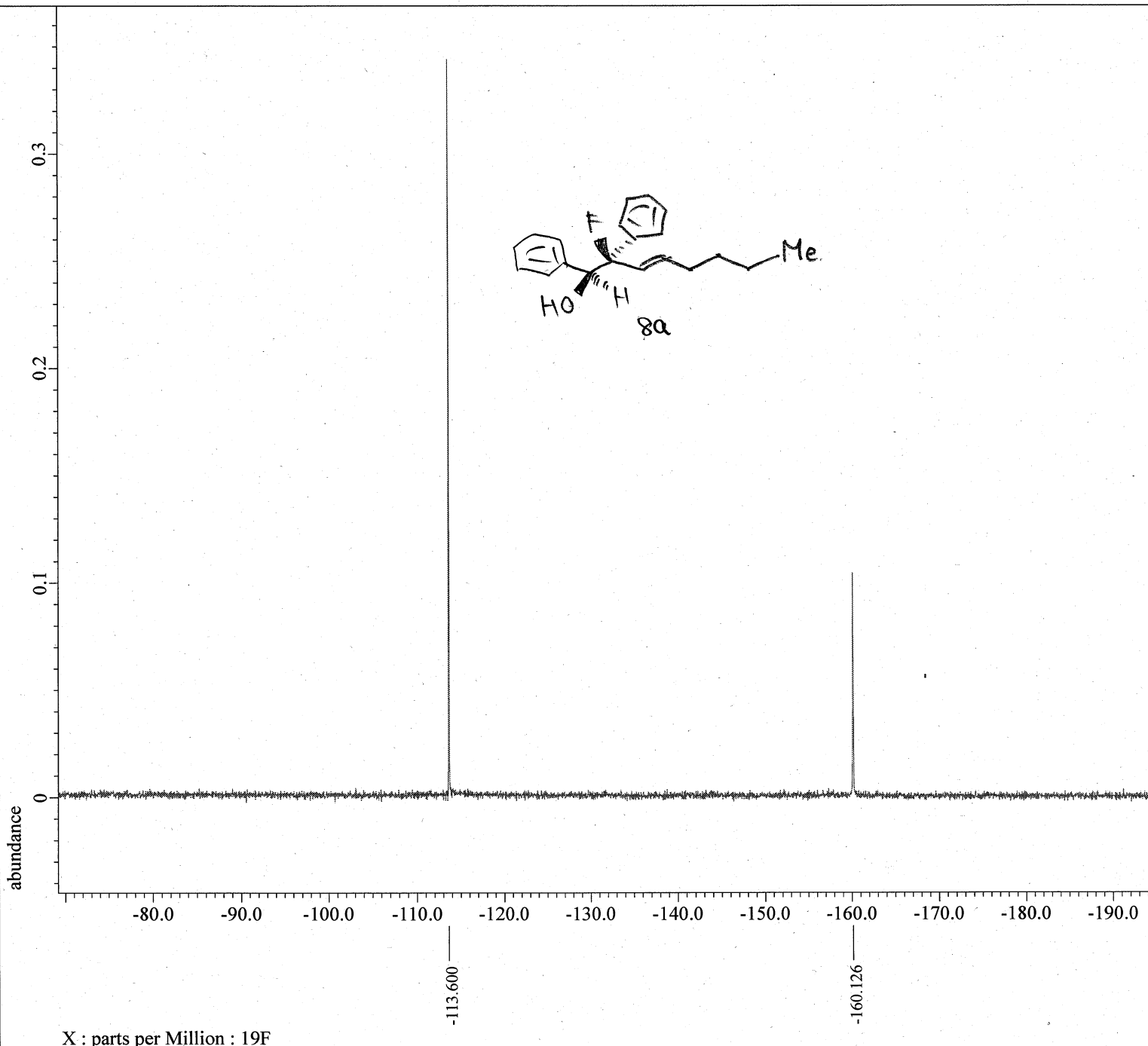




(CF<sub>3</sub>)<sub>2</sub>CO



Filename = AKY787-pure-carbon-2.jdf  
 Author = element  
 Experiment = single pulse\_dec  
 Sample Id = S#655030  
 Solvent = ACETONE-D6  
 Actual Start Time = 13-JAN-2019 02:40:50  
 Revision Time = 20-FEB-2019 10:50:39  
  
 Comment = single pulse decoupled ga  
 Data Format = 1D COMPLEX  
 Dim Size = 26214  
 X Domain = 13C  
 Dim Title = 13C  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECX 400P  
 Spectrometer = DELTA2\_NMR  
  
 Field Strength = 9.2982153[T] (400[MHz])  
 X Acq Duration = 1.048576[s]  
 X Domain = 13C  
 X Freq = 99.54517646[MHz]  
 X Offset = 100[ppm]  
 X Points = 32768  
 X Prescans = 4  
 X Resolution = 0.95367432[Hz]  
 X Sweep = 31.25[kHz]  
 Irr Domain = 1H  
 Irr Freq = 395.88430144[MHz]  
 Irr Offset = 5[ppm]  
 Clipped = FALSE  
 Scans = 256  
 Total\_Scans = 256  
  
 Relaxation\_Delay = 2[s]  
 Recvr Gain = 54  
 Temp Get = 90[dC]  
 X 90\_Width = 10.1[us]  
 X Acq Time = 1.048576[s]  
 X Angle = 30[deg]  
 X Atn = 3.4[dB]  
 X Pulse = 3.36666667[us]  
 Irr Atn Dec = 22.3[dB]  
 Irr Atn Noe = 22.3[dB]  
 Irr Noise = WALTZ  
 Decoupling = TRUE  
 Initial Wait = 1[s]  
 Noe = TRUE  
 Noe Time = 2[s]  
 Repetition Time = 3.048576[s]



Filename = AKY787-pure-FNMR-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#593741  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 19-FEB-2019 01:00:11  
Revision\_Time = 20-FEB-2019 10:58:01

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 24  
Temp\_Get = 19[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

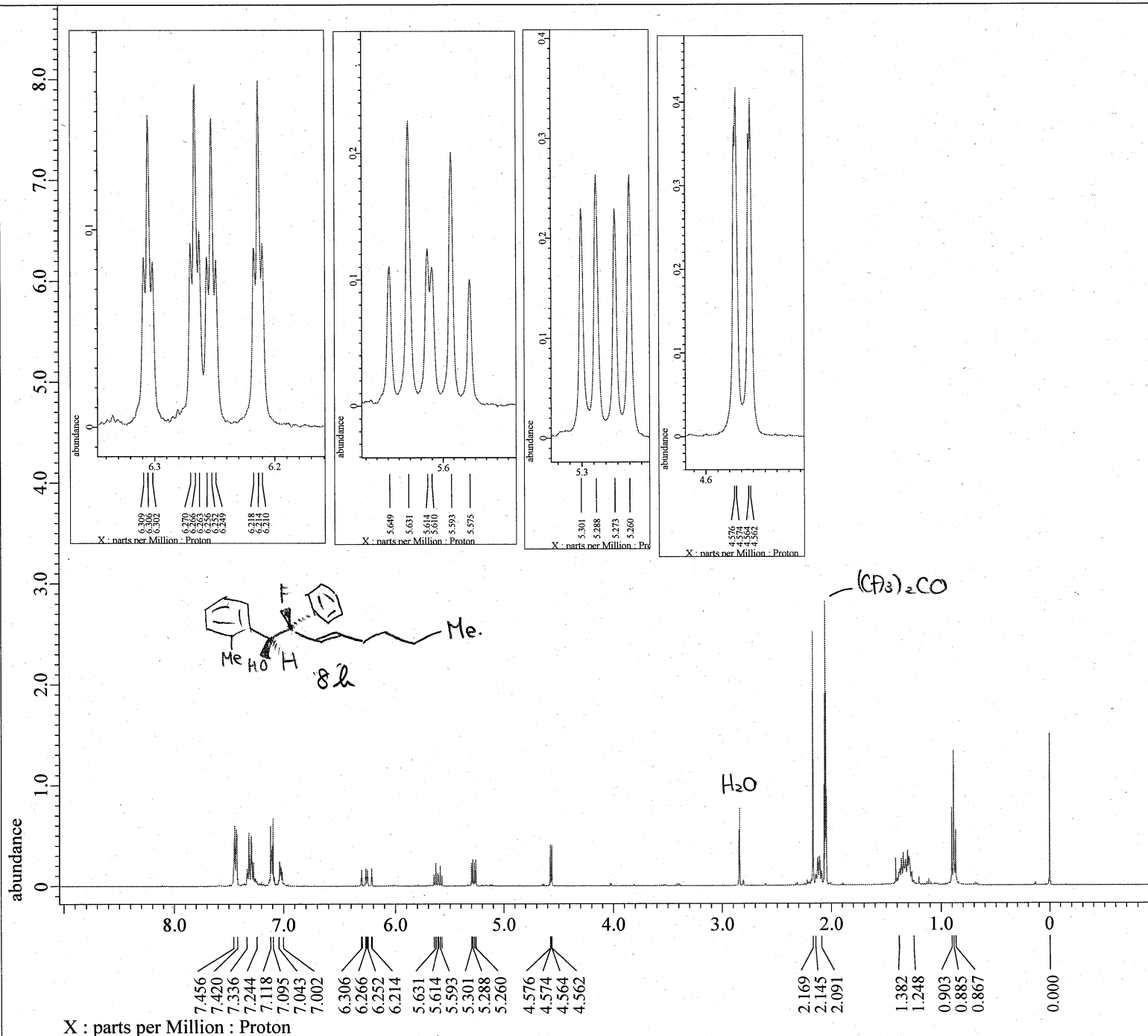


Filename = AKY819-pure-agag\_Proton-1  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY819-pure-agag  
Solvent = ACETONE-D6  
Actual\_Start Time = 26-FEB-2019 15:06:20  
Revision\_Time = 27-FEB-2019 20:56:34

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X\_Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814[T] (400[MHz])  
X\_Acq\_Duration = 2.18103808[s]  
X\_Domain = 1H  
X\_Freq = 400.53219825[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45849727[Hz]  
X\_Sweep = 7.51201923[kHz]  
X\_Sweep\_Clippped = 6.00961538[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 42  
Temp Get = 19.3[dC]  
X\_90\_Width = 6[us]  
X\_Acq\_Time = 2.18103808[s]  
X\_Angle = 45[deg]  
X\_Atn = 0.8[dB]  
X\_Pulse = 3[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
DanE\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.18103808[s]



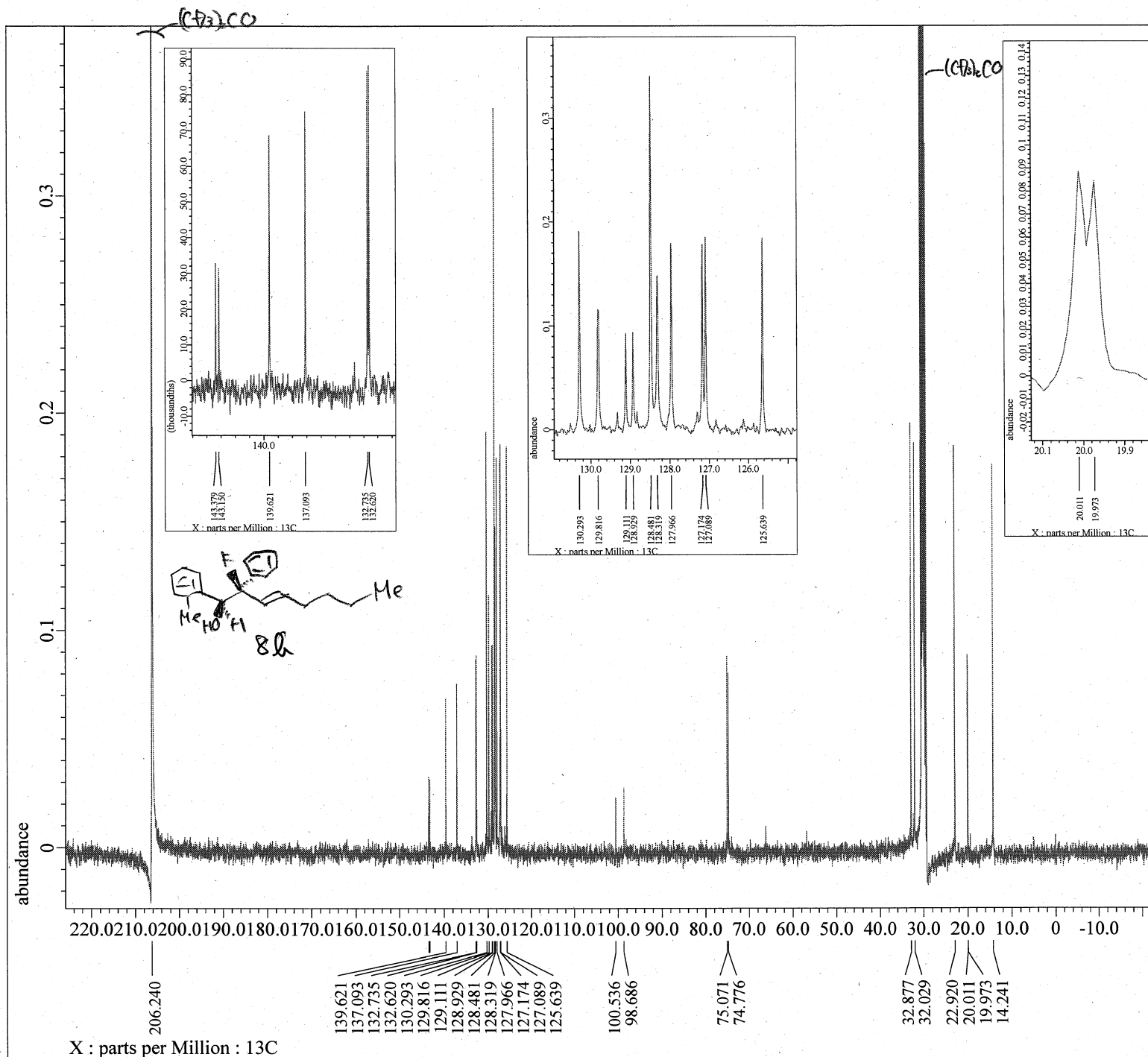


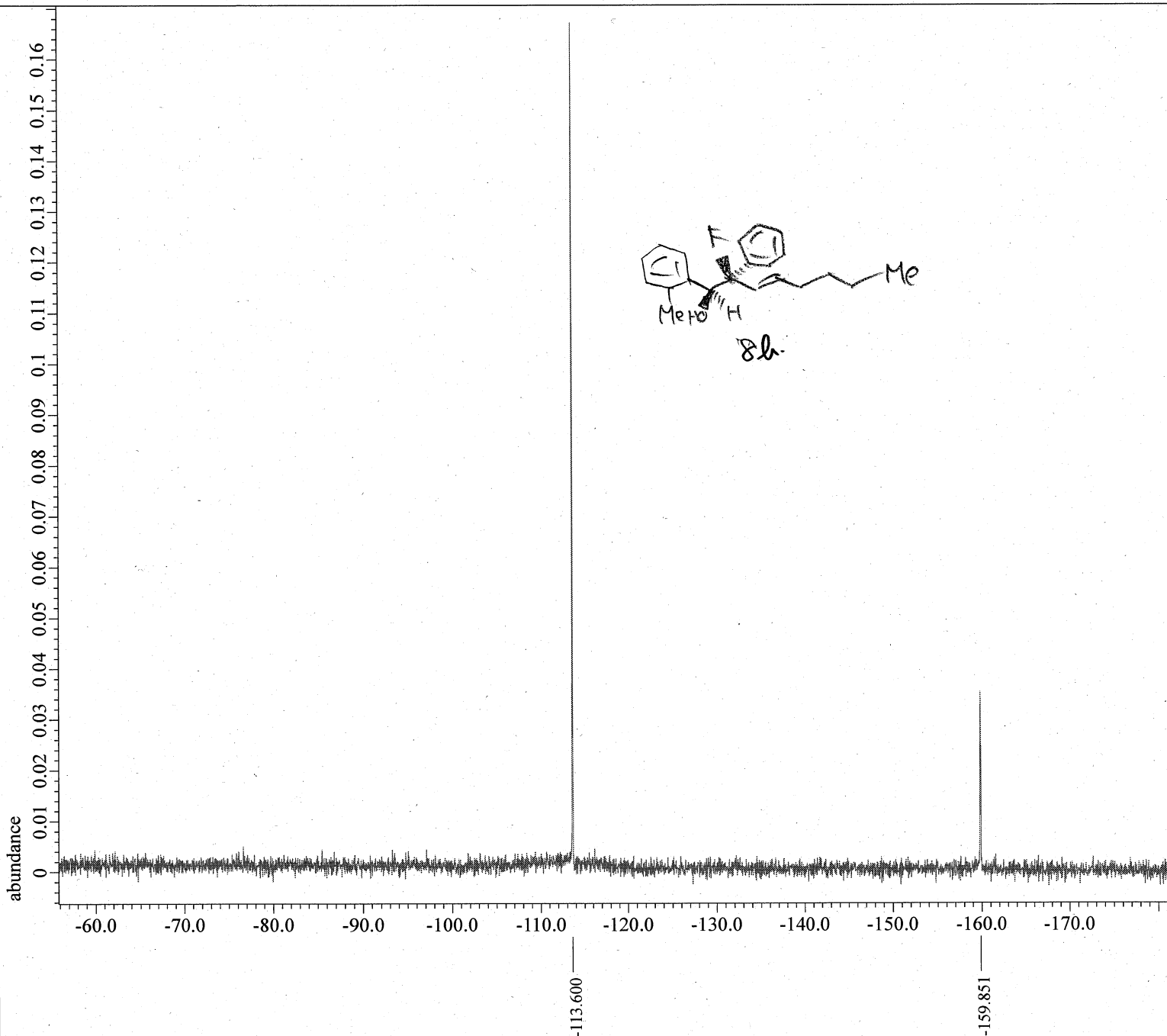
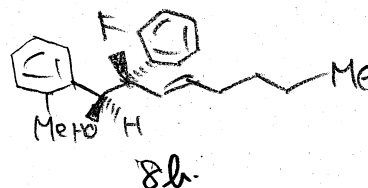
Filename = AKY819-carbon-again-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = S#549012  
Solvent = ACETONE-D6  
Actual Start Time = 26-FEB-2019 22:37:37  
Revision Time = 27-FEB-2019 21:05:06

Comment = single pulse decoupled ga  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 1.06430464[s]  
X Domain = 13C  
X Freq = 98.51479726[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061[Hz]  
X Sweep = 30.78817734[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 1024  
Total Scans = 1024

Relaxation Delay = 2[s]  
Recvr Gain = 60  
Temp Get = 18.4[dC]  
X 90 Width = 9.11[us]  
X Acq Time = 1.06430464[s]  
X Angle = 30[deg]  
X Atn = 4.9[dB]  
X Pulse = 3.03666667[us]  
Irr Atn Dec = 22.255[dB]  
Irr Atn Noe = 22.255[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.06430464[s]





Filename = AKY819-pure-FNMR-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#506103  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 8-MAR-2019 22:34:07  
Revision\_Time = 25-MAR-2019 14:19:46

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 19.5[degC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

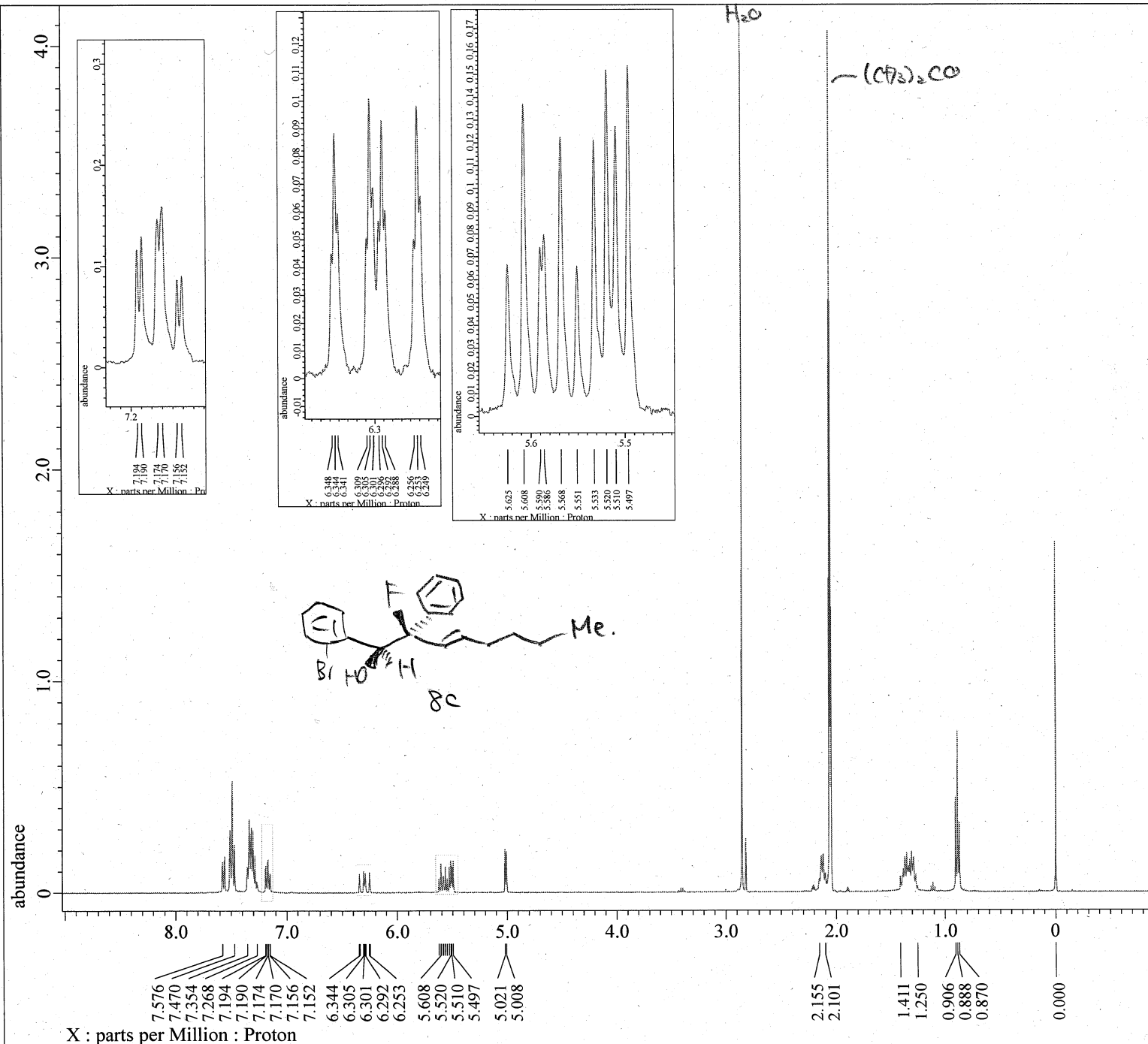


Filename = AKY841-pure-last-HNMR\_Pro  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY841-pure-last-HNMR  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 12-MAR-2019 11:47:42  
Revision\_Time = 25-MAR-2019 14:47:49

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 26214  
X Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECS400  
Spectrometer = DELTA2\_NMR

Field Strength = 9.37221[T] (400[MHz])  
X Acq\_Duration = 2.1889024[s]  
X Domain = 1H  
X Freq = 399.03472754[MHz]  
X Offset = 5.0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45684997[Hz]  
X Sweep = 7.48502994[kHz]  
X Sweep\_Clippped = 5.98802395[kHz]  
Irr Domain = Proton  
Irr Freq = 399.03472754[MHz]  
Irr Offset = 5.0[ppm]  
Tri Domain = Proton  
Tri Freq = 399.03472754[MHz]  
Tri Offset = 5.0[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 48  
Temp Get = 18.6[dC]  
X 90\_Width = 6.6[us]  
X Acq\_Time = 2.1889024[s]  
X Angle = 45[deg]  
X Atn = 1[dB]  
X Pulse = 3.3[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.1889024[s]



X : parts per Million : Proton



Filename = AKY841-pure-carbon1024-2.  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#819755  
Solvent = ACETONE-D6  
Actual\_Start Time = 12-MAR-2019 07:15:30  
Revision\_Time = 25-MAR-2019 14:49:35

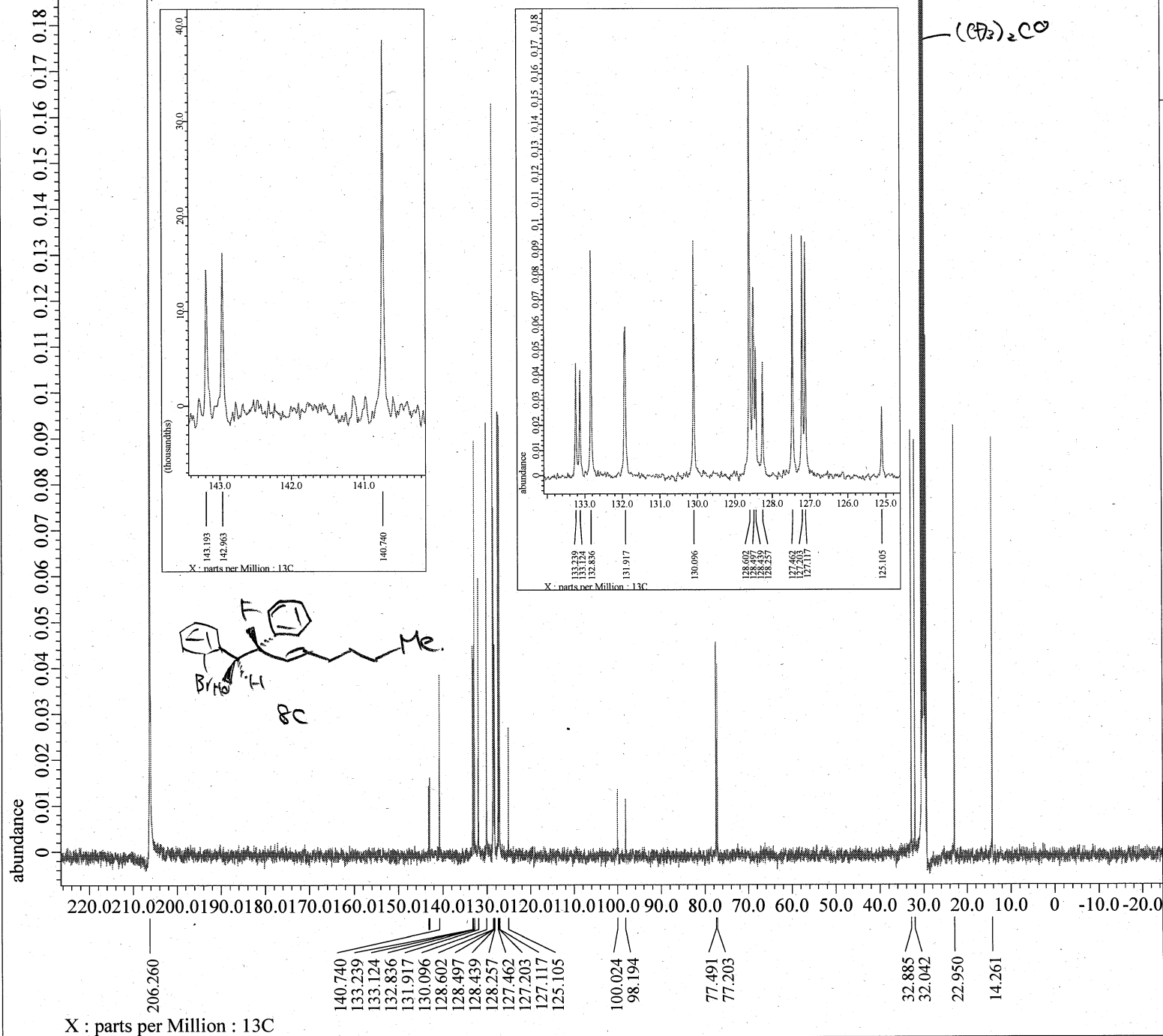
Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq\_Duration = 1.048576 [s]  
X Domain = 13C  
X Freq = 99.54517646 [MHz]  
X Offset = 100 [ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432 [Hz]  
X Sweep = 31.25 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 1024  
Total\_Scans = 1024

Relaxation\_Delay = 2 [s]  
Recvr Gain = 50  
Temp Get = 20.8 [dC]  
X 90\_Width = 10.1 [us]  
X Acq\_Time = 1.048576 [s]  
X Angle = 30 [deg]  
X Atn = 3.4 [dB]  
X Pulse = 3.36666667 [us]  
Irr Atn Dec = 22.3 [dB]  
Irr Atn Noe = 22.3 [dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1 [s]  
Noe = TRUE  
Noe Time = 2 [s]  
Repetition\_Time = 3.048576 [s]

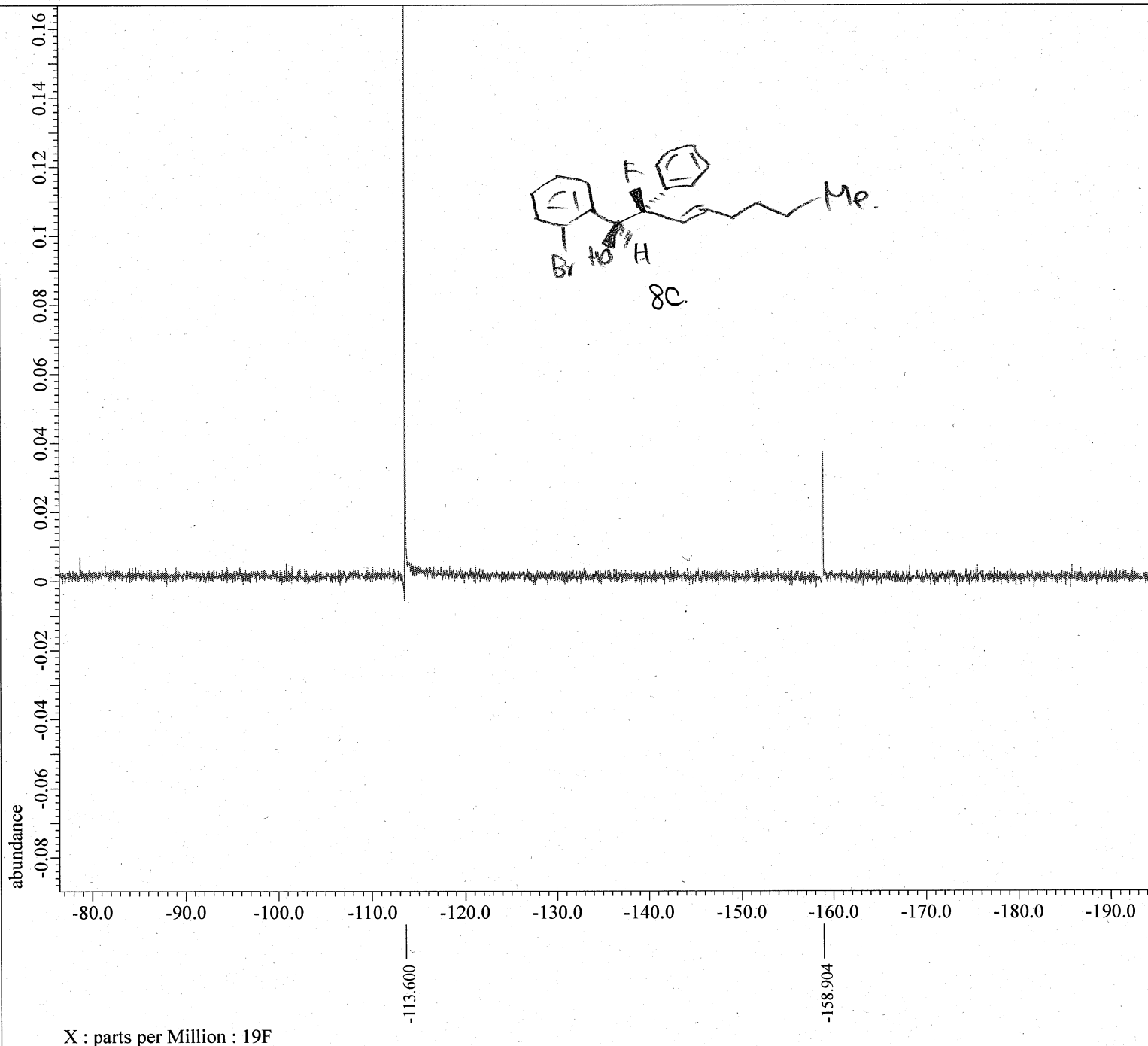
(CF<sub>3</sub>)<sub>2</sub>CO

(CF<sub>3</sub>)<sub>2</sub>CO



X : parts per Million : 13C



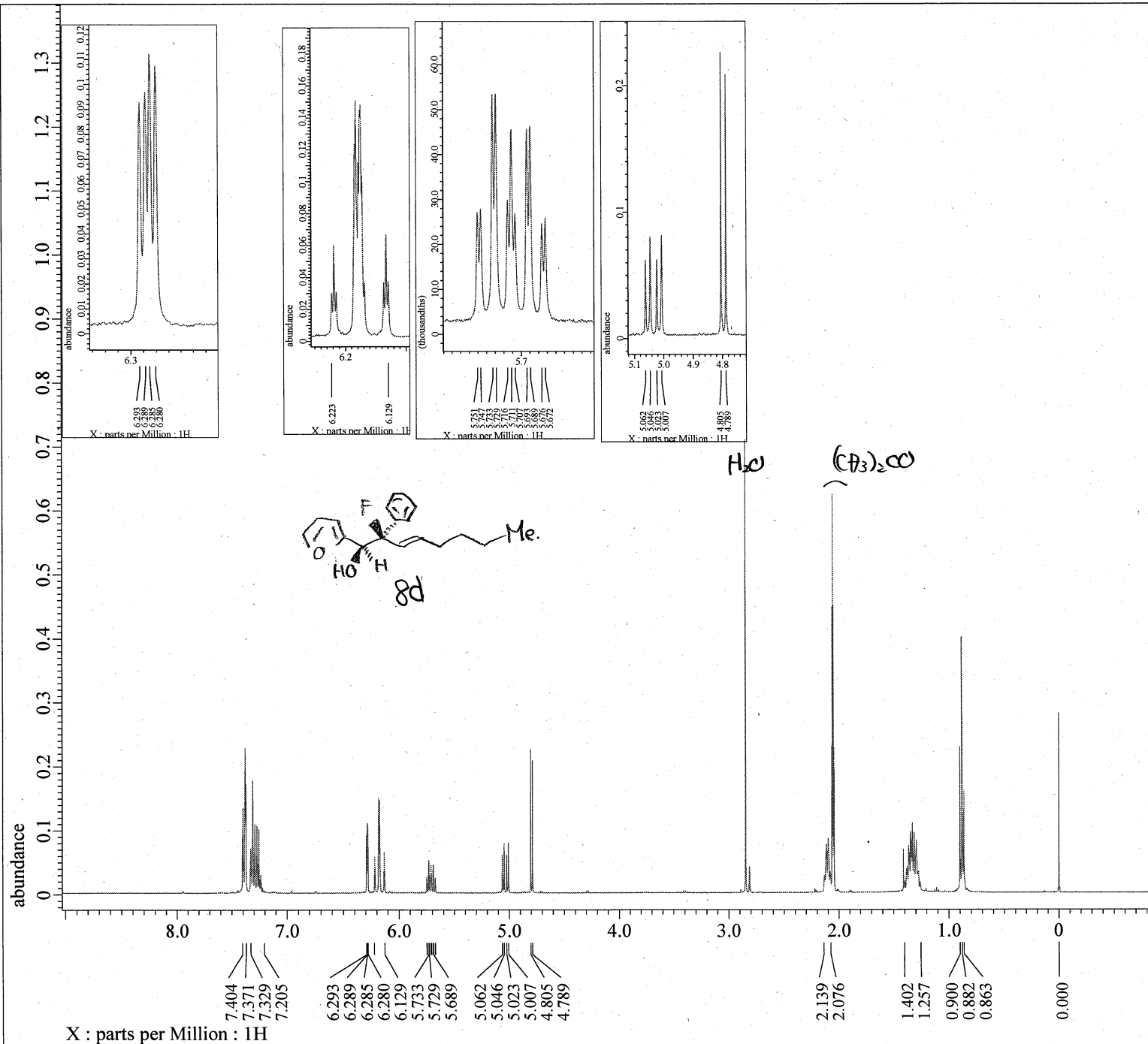


Filename = AKY841-pure-FNMRAGain-2.j  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#348731  
Solvent = ACETONE-D6  
Actual\_Start Time = 12-MAR-2019 18:11:47  
Revision\_Time = 25-MAR-2019 14:51:36

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153 [T] (400 [MHz])  
X Acq\_Duration = 87.81824 [ms]  
X Domain = 19F  
X Freq = 372.50336686 [MHz]  
X Offset = 0 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602 [Hz]  
X Sweep = 186.56716418 [kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr Gain = 24  
Temp Get = 19.9 [dC]  
X 90\_Width = 13.9 [us]  
X Acq\_Time = 87.81824 [ms]  
X Angle = 45 [deg]  
X Atn = 4 [dB]  
X Pulse = 6.95 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 5.08781824 [s]



Filename = AKY820-pure-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#720775  
Solvent = ACETONE-D6  
Actual Start Time = 9-FEB-2019 04:32:03  
Revision Time = 17-FEB-2019 18:47:51

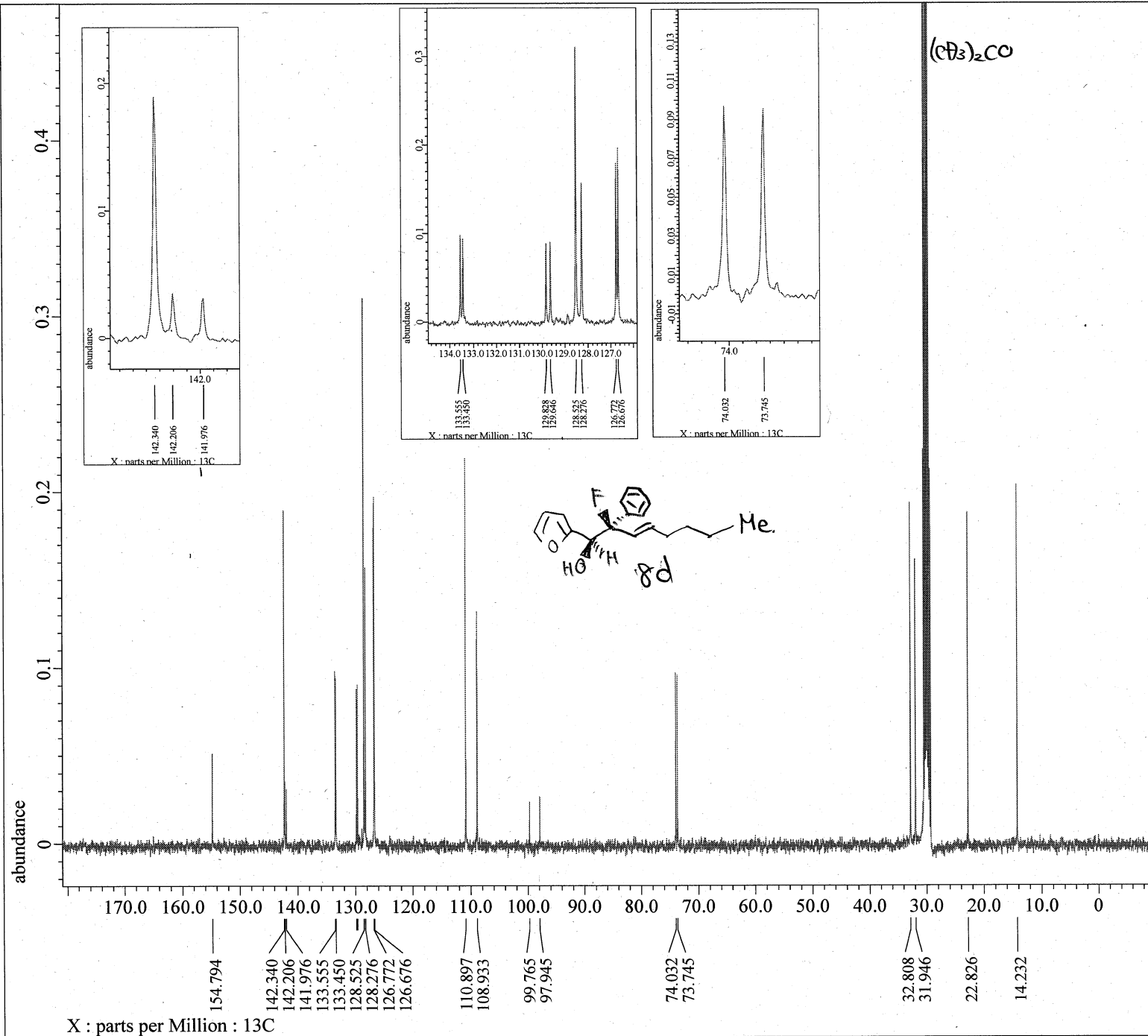
Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 2.20725248[s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5 [s]  
Recvr Gain = 36  
Temp Get = 20.7 [dC]  
X 90 Width = 13.2 [us]  
X Acq Time = 2.20725248 [s]  
X Angle = 45 [deg]  
X Atn = 3.5 [dB]  
X Pulse = 6.6 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.20725248 [s]



$(CD_3)_2CO$

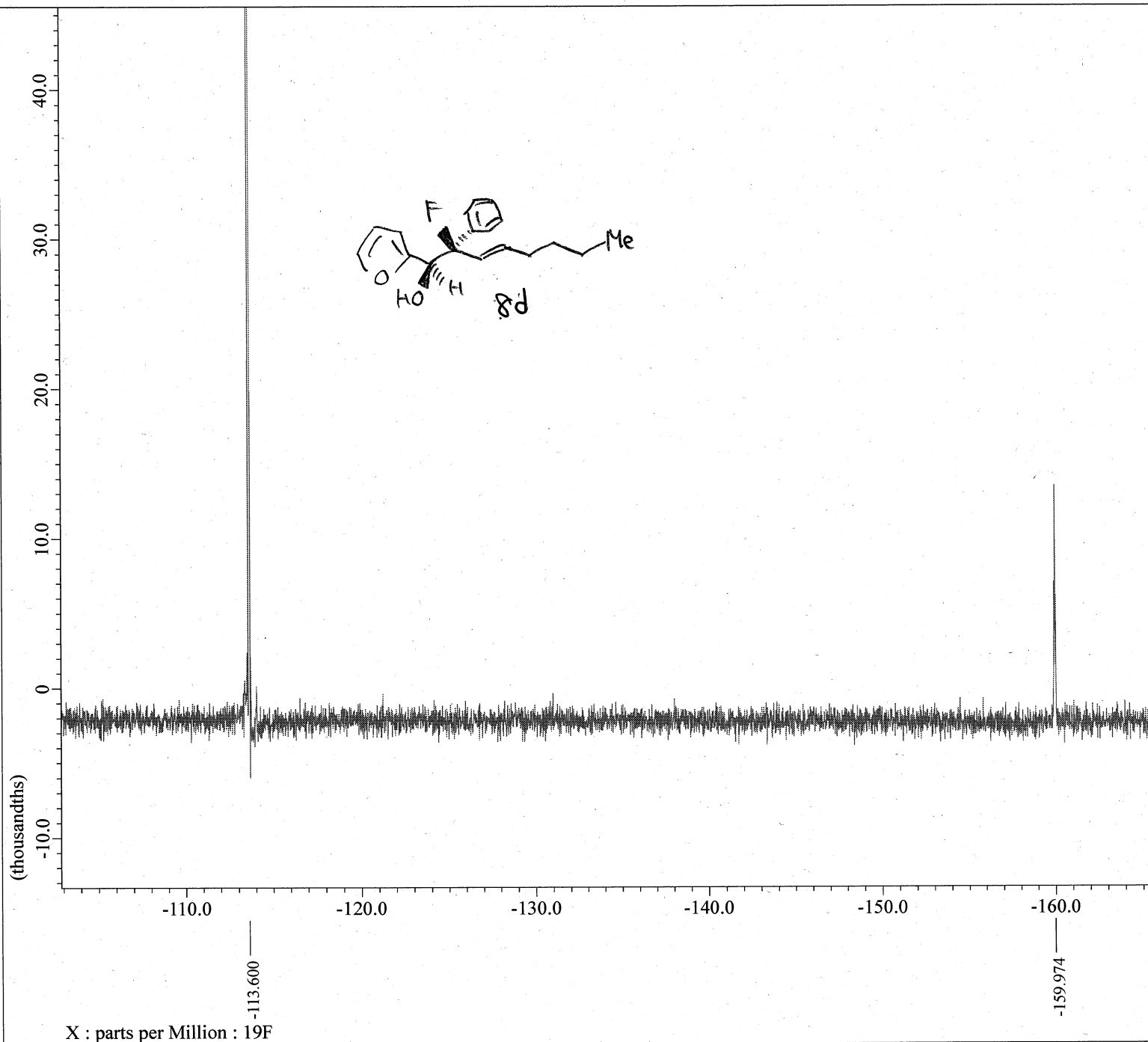


Filename = AKY820-carbon-2.jdf  
Author = element  
Experiment = single\_pulse\_dec  
Sample Id = S#553236  
Solvent = ACETONE-D6  
Actual\_Start Time = 17-FEB-2019 23:52:25  
Revision Time = 17-FEB-2019 18:51:35

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 512  
Total Scans = 512

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 19.3[dC]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]



Filename = AKY820-FNMR-32-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#601516  
Solvent = ACETONE-D6  
Actual\_Start Time = 18-FEB-2019 01:12:46  
Revision Time = 17-FEB-2019 19:06:58

Comment = single\_pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 0.17563648[s]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = -100[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 5.69357801[Hz]  
X Sweep = 93.28358209[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 32  
Total\_Scans = 32

Relaxation\_Delay = 5[s]  
Recvr Gain = 30  
Temp Get = 20[dC]  
X 90\_Width = 13.9[us]  
X Acq\_Time = 0.17563648[s]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.17563648[s]

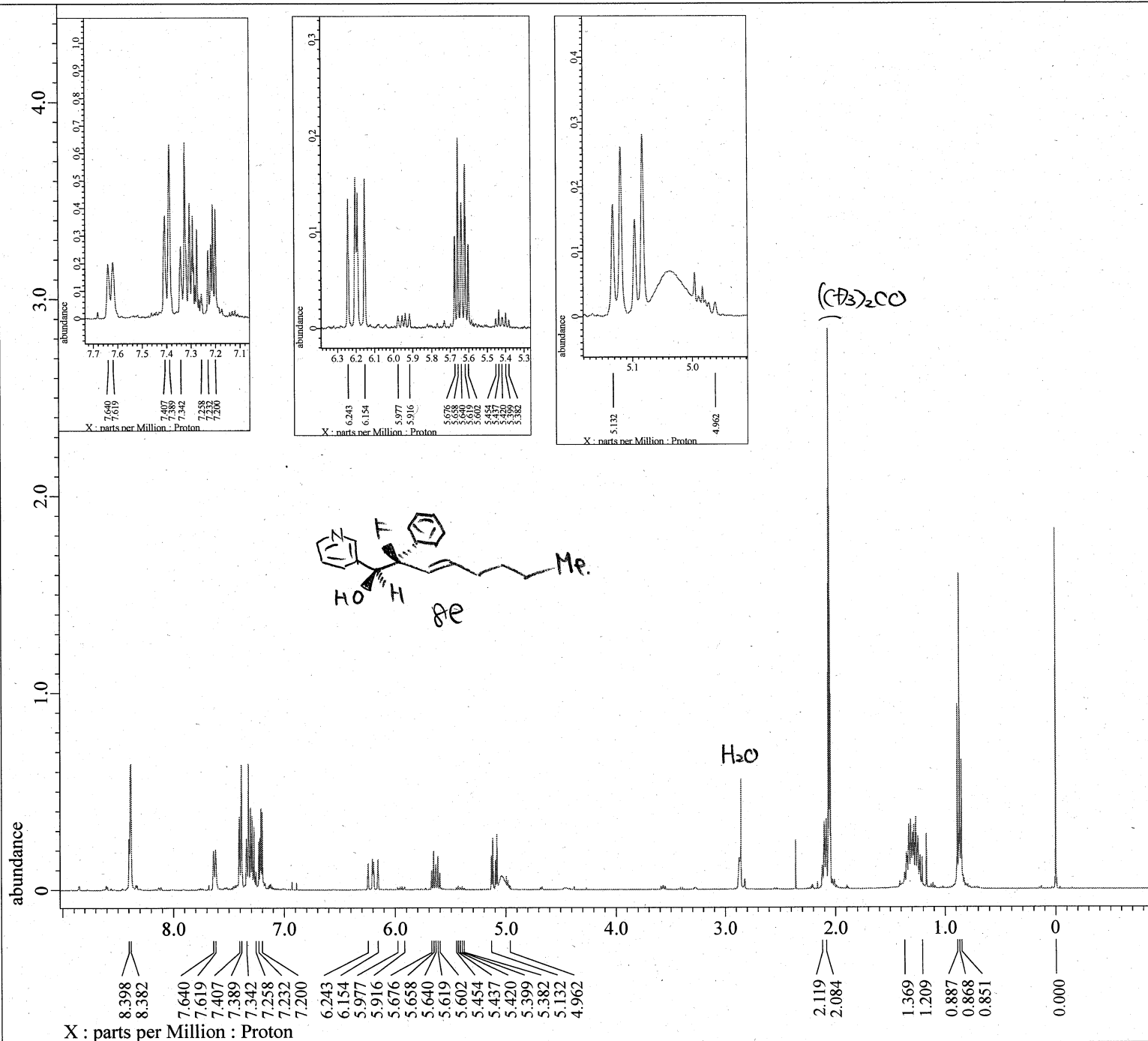


Filename = AKY798-pure1\_Proton-1-2.j  
Author = element  
Experiment = proton.jxp  
Sample Id = AKY798-pure1  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 22-JAN-2019 14:38:04  
Revision\_Time = 20-FEB-2019 15:24:03

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X\_Domain = Proton  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Spectrometer = DELTA2\_NMR

Field Strength = 9.4073814 [T] (400 [MHz])  
X\_Acq\_Duration = 2.18103808 [s]  
X\_Domain = 1H  
X\_Freq = 400.53219825 [MHz]  
X\_Offset = 5 [ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 0.45849727 [Hz]  
X\_Sweep = 7.51201923 [kHz]  
X\_Sweep\_Clipped = 6.00961538 [kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 400.53219825 [MHz]  
Irr\_Offset = 5 [ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 400.53219825 [MHz]  
Tri\_Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr\_Gain = 44  
Temp\_Get = 17.6 [dC]  
X\_90\_Width = 6 [us]  
X\_Acq\_Time = 2.18103808 [s]  
X\_Angle = 45 [deg]  
X\_Atn = 0.8 [dB]  
X\_Pulse = 3 [us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
DanE\_Presat = FALSE  
Initial\_Wait = 1 [s]  
Repetition\_Time = 7.18103808 [s]



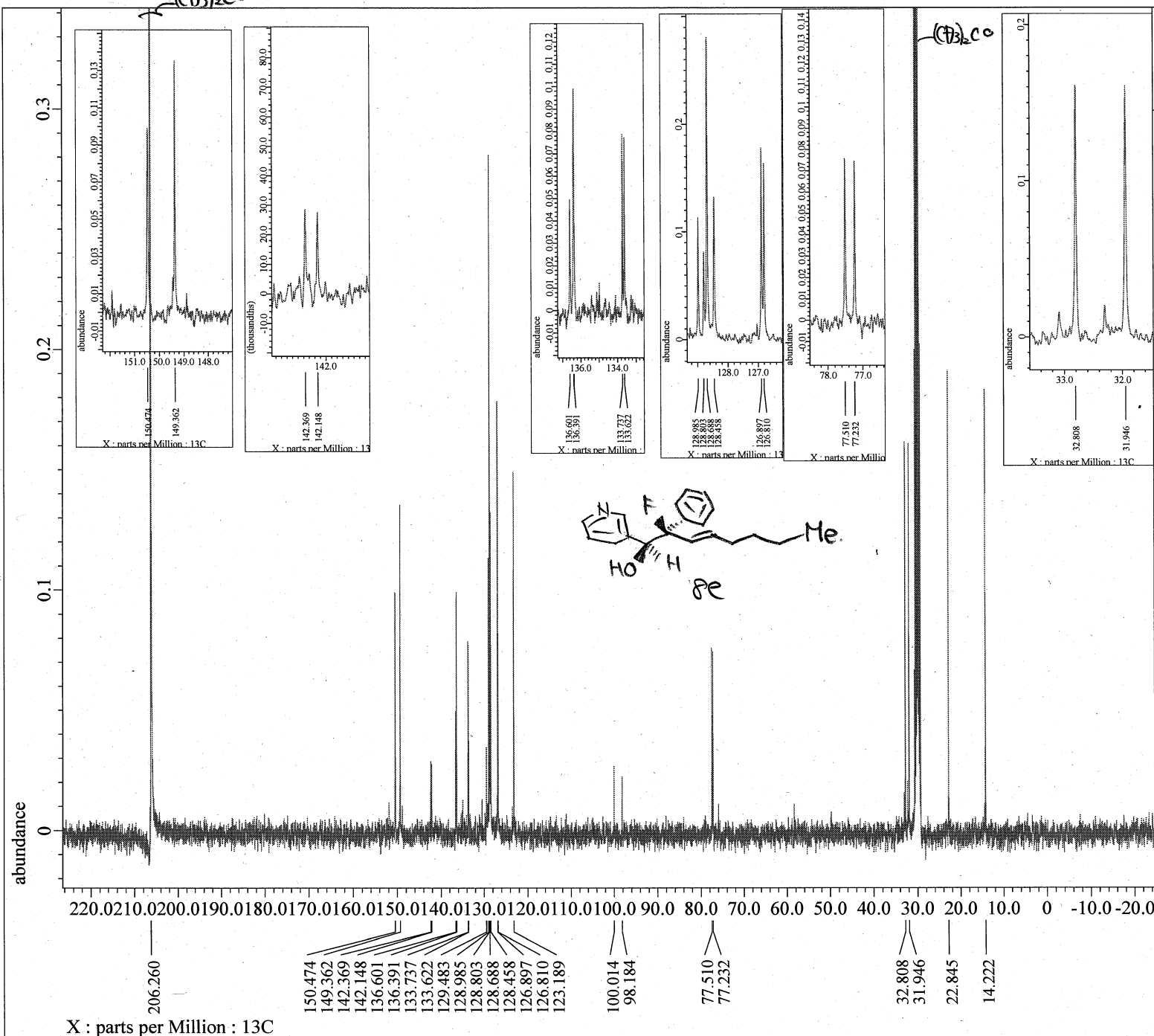


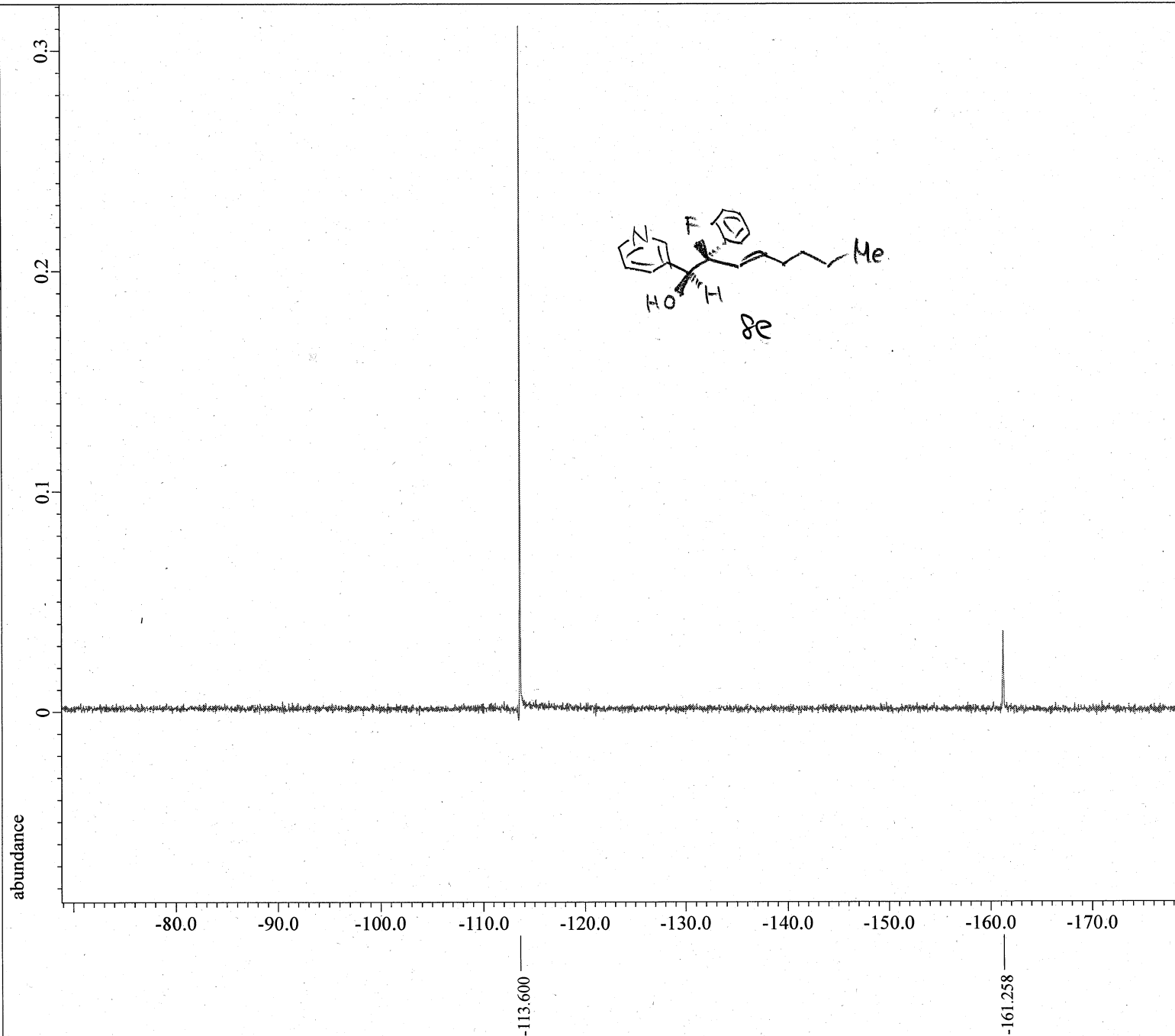
Filename = AKY798-pure-carbon256-2.j  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#748176  
Solvent = ACETONE-D6  
Actual Start Time = 7-FEB-2019 05:16:57  
Revision Time = 20-FEB-2019 15:31:30

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 1.048576[s]  
X Domain = 13C  
X Freq = 99.54517646[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.95367432[Hz]  
X Sweep = 31.25[kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total Scans = 256

Relaxation Delay = 2[s]  
Recvr Gain = 54  
Temp Get = 20.2[dC]  
X 90 Width = 10.1[us]  
X Acq Time = 1.048576[s]  
X Angle = 30[deg]  
X Atn = 3.4[dB]  
X Pulse = 3.36666667[us]  
Irr Atn Dec = 22.3[dB]  
Irr Atn Noe = 22.3[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition Time = 3.048576[s]





Filename = AKY798-pure-FNMR-3.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#782013  
Solvent = ACETONE-D6  
Actual\_Start Time = 7-FEB-2019 06:13:54  
Revision\_Time = 20-FEB-2019 15:52:25

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 20.9[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
DanTe\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

X : parts per Million : 19F

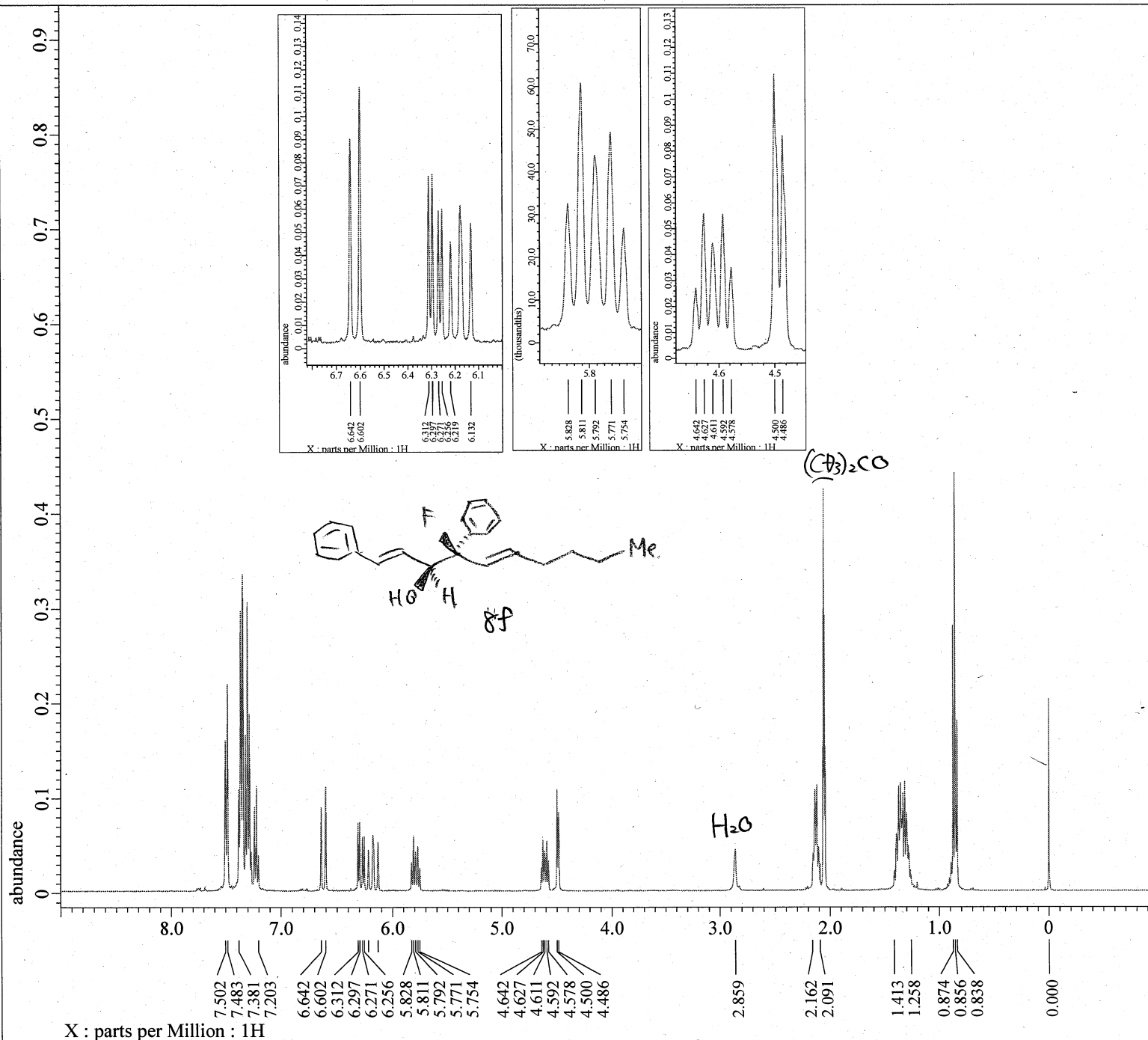


Filename = AKY824-pure-again-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#604771  
Solvent = ACETONE-D6  
Actual Start Time = 17-FEB-2019 01:18:30  
Revision Time = 16-FEB-2019 20:02:55

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 26214  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

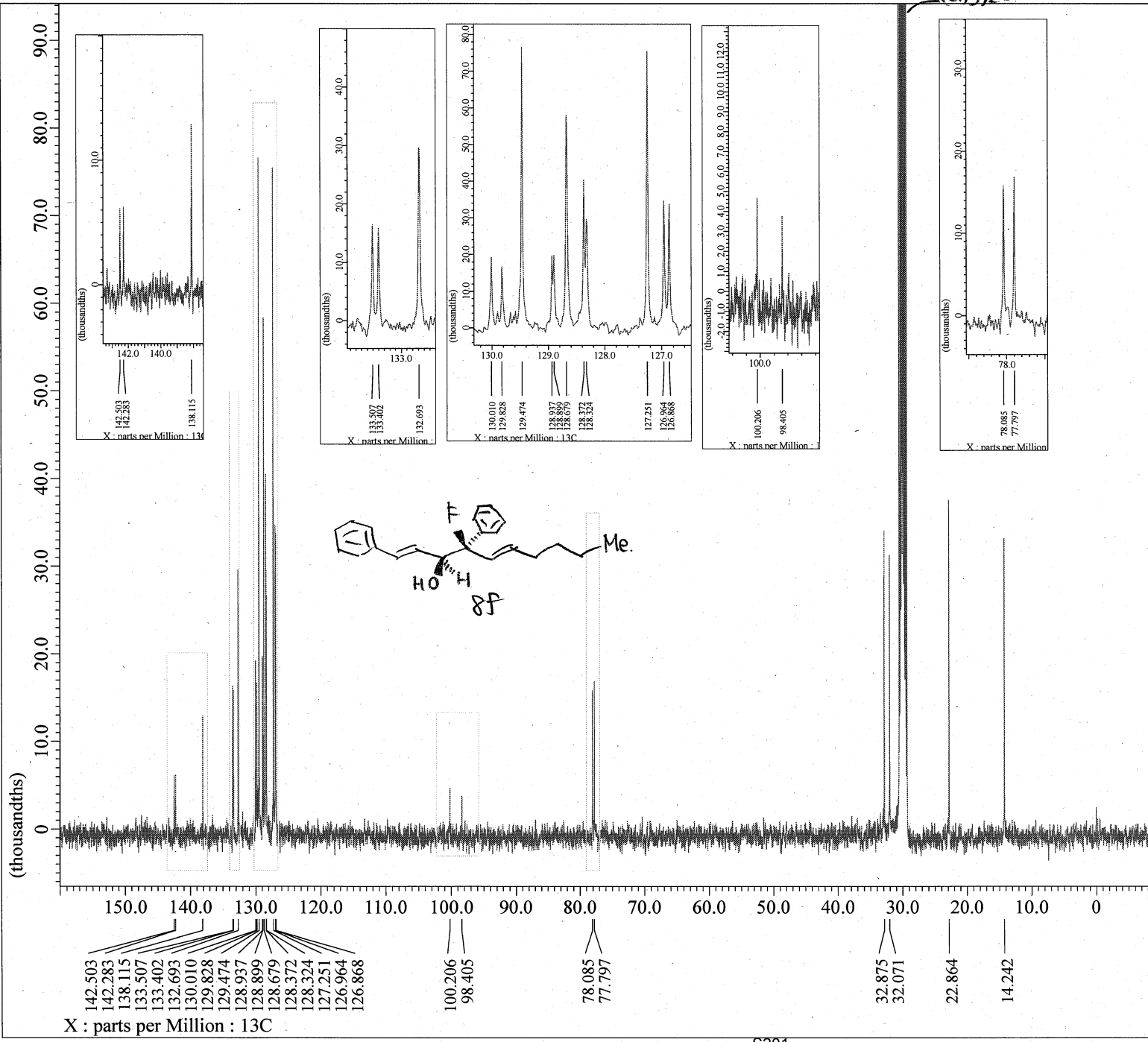
Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 2.20725248[s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 36  
Temp Get = 19.2 [dC]  
X 90 Width = 13.2 [us]  
X Acq Time = 2.20725248 [s]  
X Angle = 45 [deg]  
X Atn = 3.5 [dB]  
X Pulse = 6.6 [us]  
Irr Mode = Off  
Tri Mode = Off  
Date\_Preset = FALSE  
Initial Wait = 1 [s]  
Repetition Time = 7.20725248 [s]

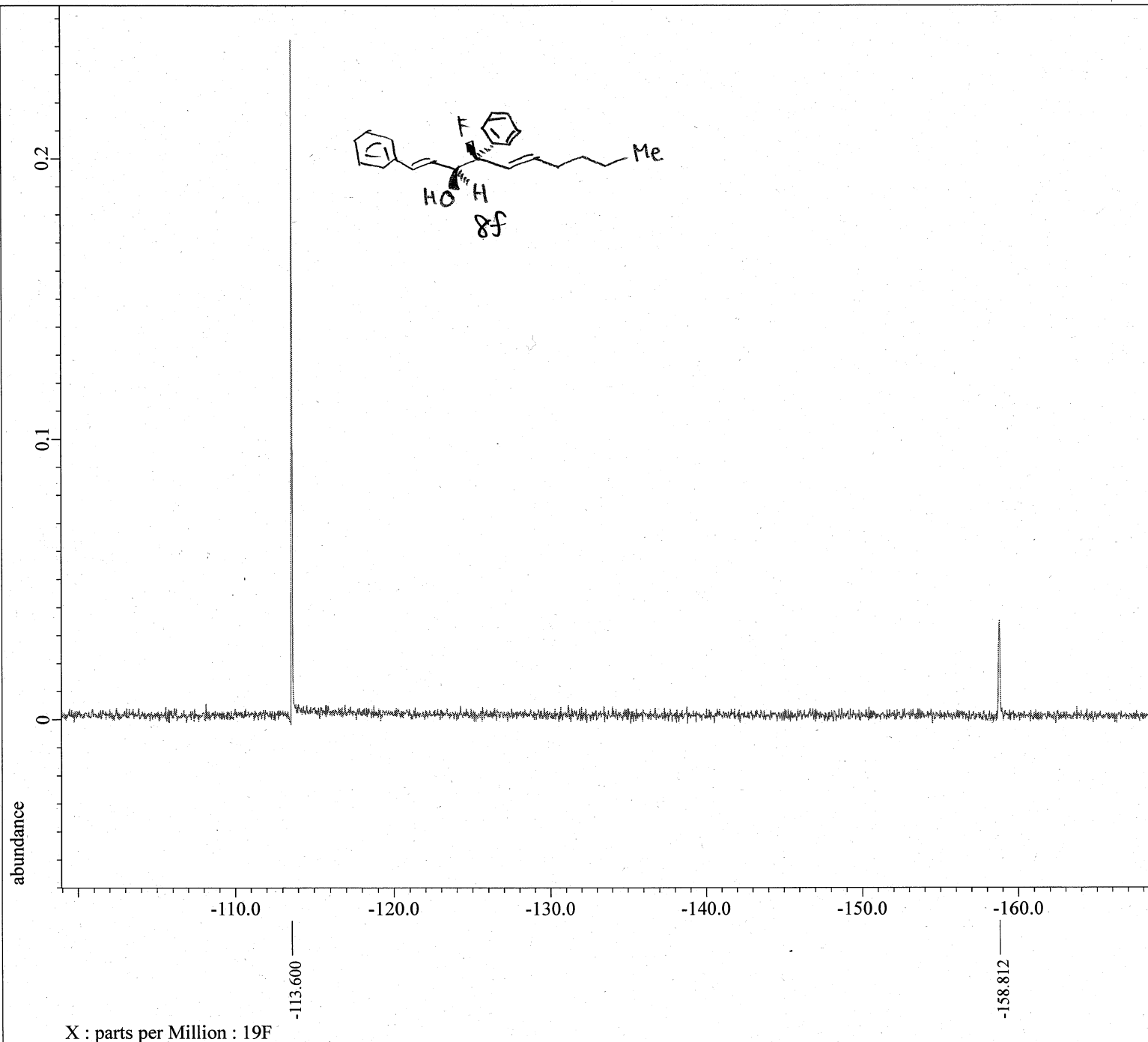
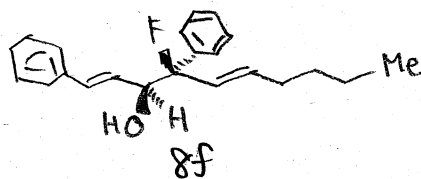




(CF<sub>3</sub>)<sub>2</sub>CO



Filename = AKY824-carbon-2.jdf  
 Author = element  
 Experiment = single\_pulse\_dec  
 Sample Id = S#592065  
 Solvent = ACETONE-D6  
 Actual Start Time = 16-FEB-2019 00:56:04  
 Revision Time = 16-FEB-2019 20:13:59  
  
 Comment = single pulse decoupled ga  
 Data Format = 1D COMPLEX  
 Dim Size = 26214  
 X Domain = 13C  
 Dim Title = 13C  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = EXC 400P  
 Spectrometer = DELTA2\_NMR  
  
 Field Strength = 9.2982153[T] (400[MHz])  
 X Acq Duration = 1.048576[s]  
 X Domain = 13C  
 X Freq = 99.54517646[MHz]  
 X Offset = 100[ppm]  
 X Points = 32768  
 X Prescans = 4  
 X Resolution = 0.95367432[Hz]  
 X Sweep = 31.25[kHz]  
 Irr Domain = 1H  
 Irr Freq = 395.88430144 [MHz]  
 Irr Offset = 5[ppm]  
 Clipped = FALSE  
 Scans = 1024  
 Total\_Scans = 1024  
  
 Relaxation\_Delay = 2[s]  
 Recvr Gain = 48  
 Temp Get = 19.4[dC]  
 X 90\_Width = 10.1[us]  
 X Acq Time = 1.048576[s]  
 X Angle = 30[deg]  
 X Atn = 3.4[dB]  
 X Pulse = 3.36666667[us]  
 Irr Atn Dec = 22.3[dB]  
 Irr Atn Noe = 22.3[dB]  
 Irr Noise = WALTZ  
 Decoupling = TRUE  
 Initial Wait = 1[s]  
 Noe = TRUE  
 Noe Time = 2[s]  
 Repetition Time = 3.048576[s]

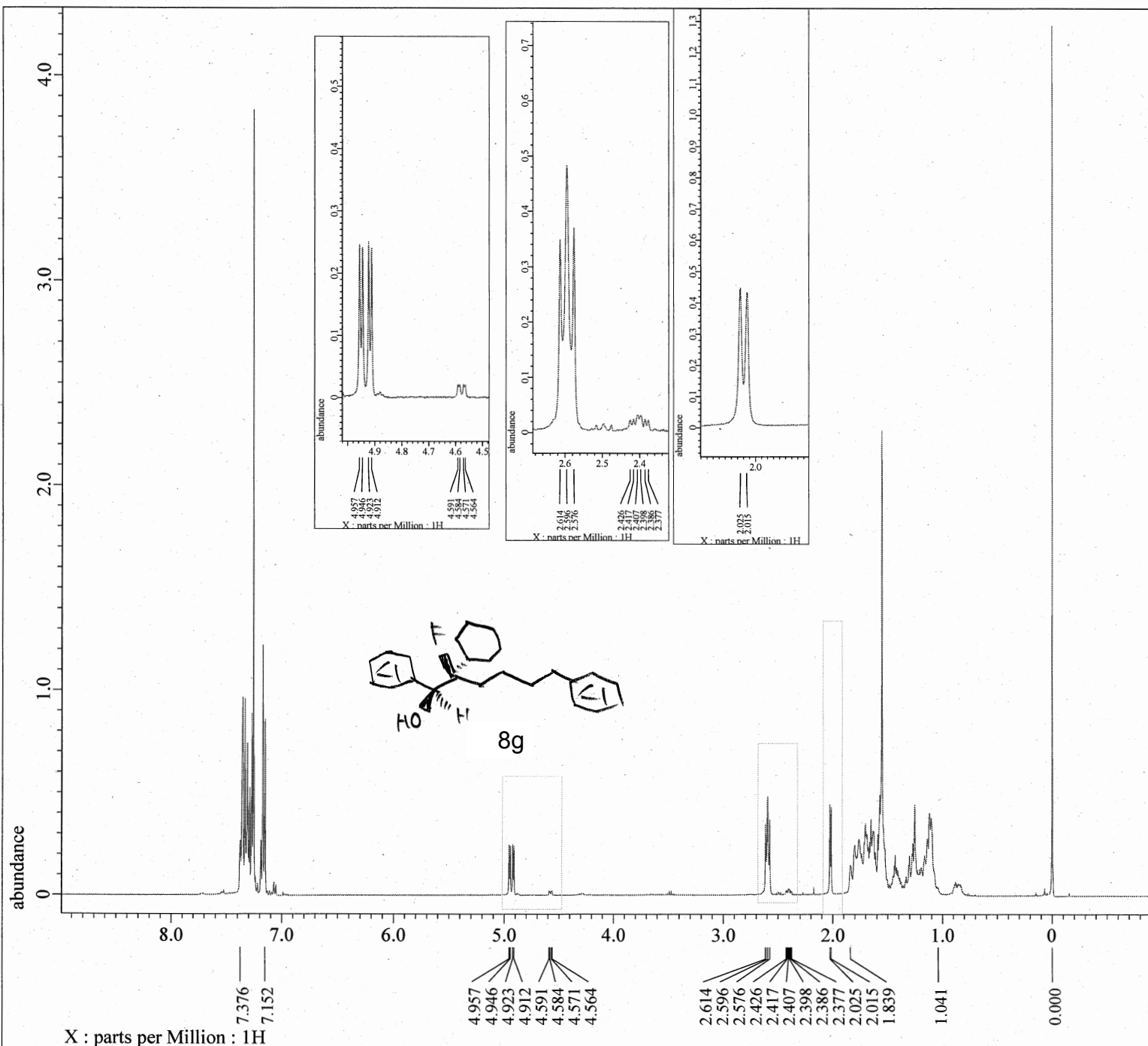


Filename = AKY824-pure-fnmr-again-2.  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#629184  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 17-FEB-2019 01:59:10  
Revision\_Time = 16-FEB-2019 20:38:45

Comment = single pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 26  
Temp\_Get = 19.2[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]



Filename = AKY713-pure-1 2-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#378499  
Solvent = CHLOROFORM-D  
Actual Start Time = 2-NOV-2018 17:59:05  
Revision Time = 25-MAR-2019 19:27:14

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq Duration = 2.228224[s]  
X Domain = 1H  
X Freq = 391.78655441[MHz]  
X Offset = 5[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.44878791[Hz]  
X Sweep = 7.35294118[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 1H  
Tri Freq = 391.78655441[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 32  
Total Scans = 32

Relaxation Delay = 5[s]  
Recvr Gain = 46  
Temp Get = 19.4[dc]  
X 90 Width = 11.04[us]  
X Acq Time = 2.228224[s]  
X Angle = 45[deg]  
X Atn = 1.9[db]  
X Pulse = 5.52[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 7.228224[s]

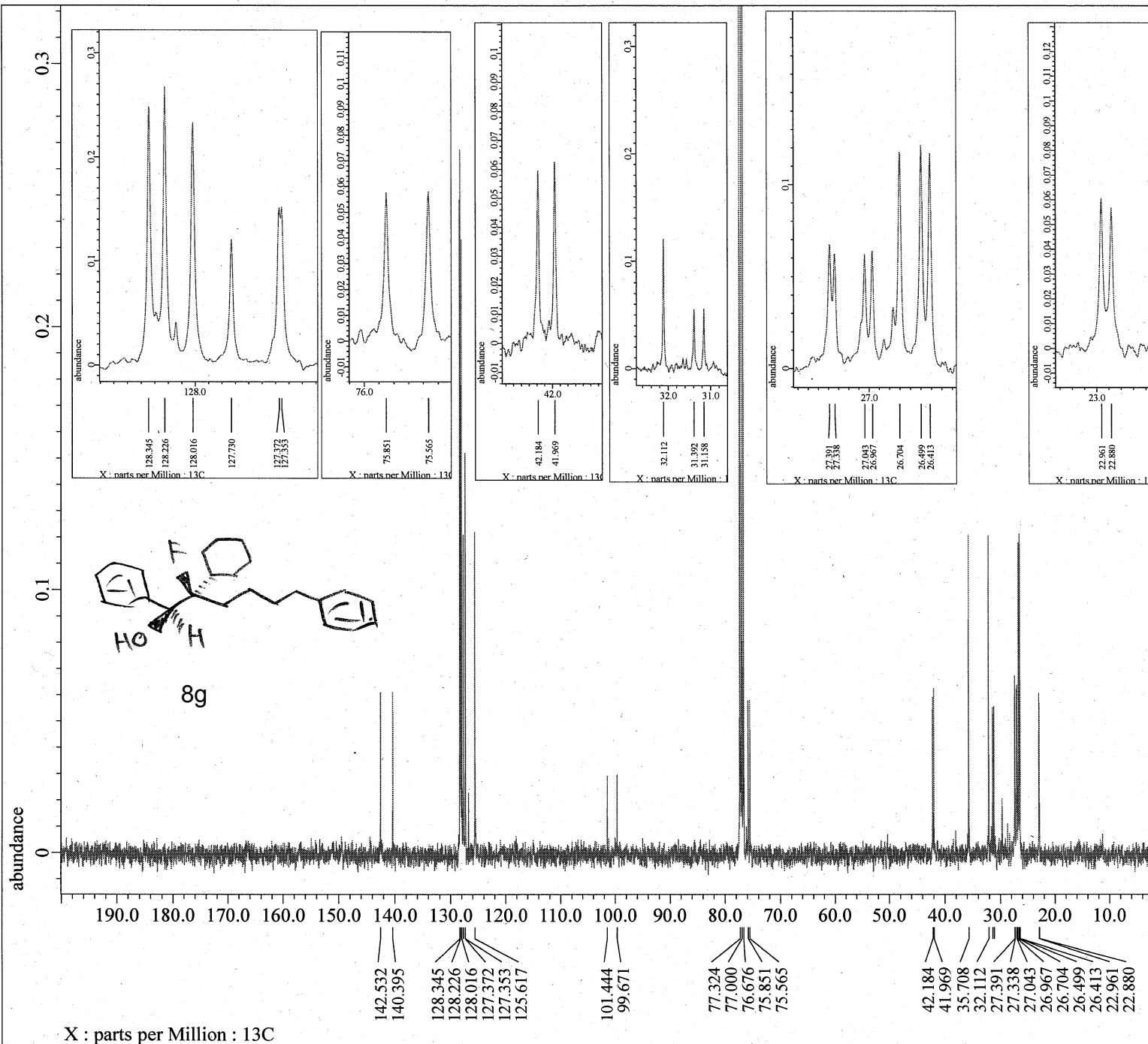


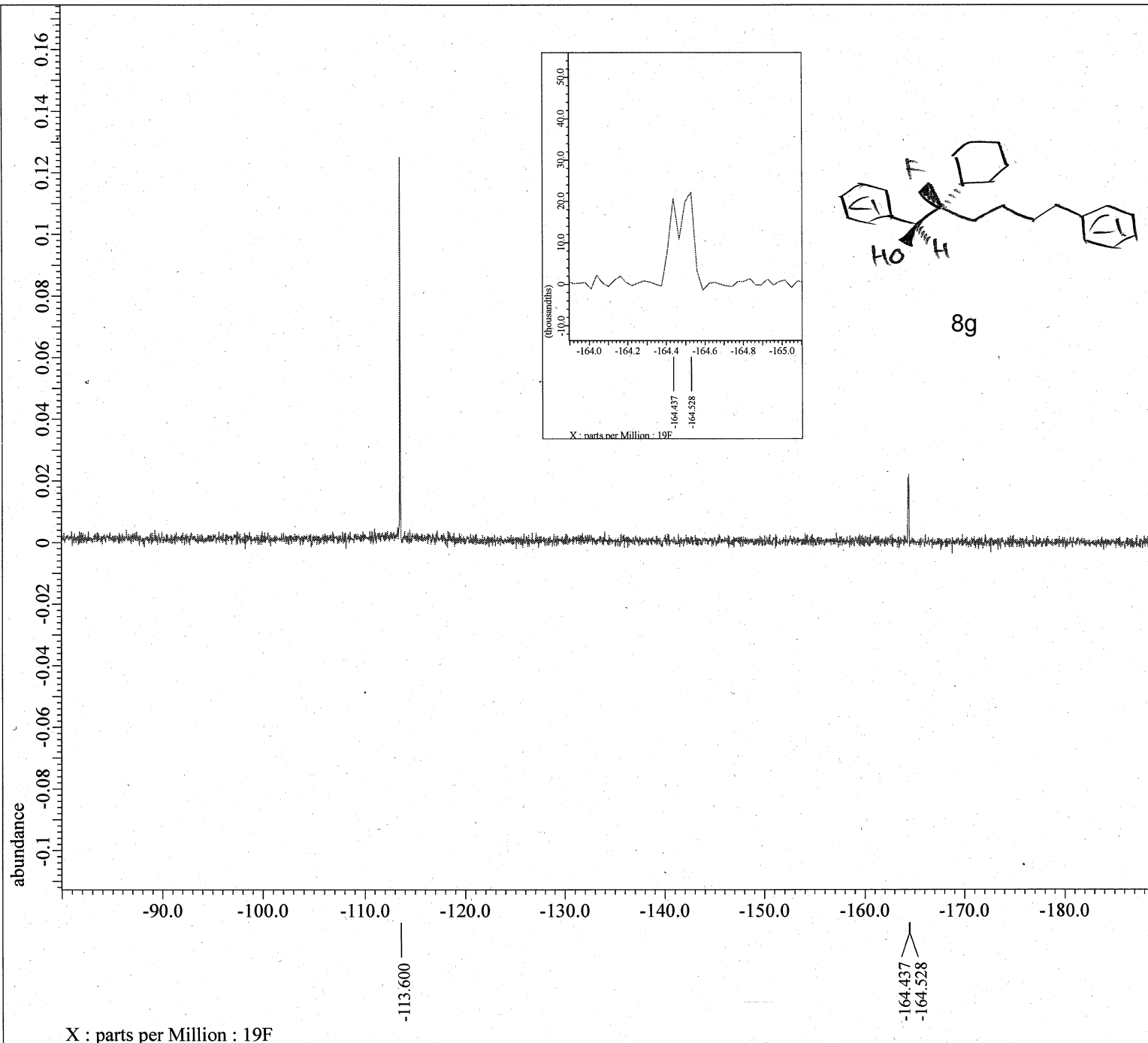
Filename = AKY713-carbon-2.jdf  
Author = element  
Experiment = single pulse\_dec  
Sample Id = S#357964  
Solvent = CHLOROFORM-D  
Actual\_Start Time = 2-NOV-2018 17:23:44  
Revision\_Time = 2-NOV-2018 13:25:53

Comment = single pulse decoupled ga  
Data Format = 1D COMPLEX  
Dim Size = 52428  
X Domain = 13C  
Dim Title = 13C  
Dim Units = [ppm]  
Dimensions = X  
Site = ECS 400  
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])  
X Acq\_Duration = 1.06430464[s]  
X Domain = 13C  
X Freq = 98.51479726[MHz]  
X Offset = 100[ppm]  
X Points = 32768  
X Prescans = 4  
X Resolution = 0.93958061[Hz]  
X Sweep = 30.78817734[kHz]  
Irr Domain = 1H  
Irr Freq = 391.78655441[MHz]  
Irr Offset = 5[ppm]  
Clipped = FALSE  
Scans = 256  
Total\_Scans = 256

Relaxation\_Delay = 2[s]  
Recvr Gain = 60  
Temp Get = 18.7[dC]  
X 90\_Width = 9.11[us]  
X Acq\_Time = 1.06430464[s]  
X Angle = 30[deg]  
X Atn = 4.9[dB]  
X Pulse = 3.03666667[us]  
Irr Atn Dec = 22.255[dB]  
Irr Atn Noe = 22.255[dB]  
Irr Noise = WALTZ  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition\_Time = 3.06430464[s]





Filename = AKY713-pure-fnmr-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#397178  
Solvent = CHLOROFORM-D  
Actual\_Start\_Time = 2-NOV-2018 19:31:40  
Revision\_Time = 2-NOV-2018 13:15:29

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X\_Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field\_Strength = 9.2982153[T] (400[MHz])  
X\_Acq\_Duration = 87.81824[ms]  
X\_Domain = 19F  
X\_Freq = 372.50336686[MHz]  
X\_Offset = 0[ppm]  
X\_Points = 16384  
X\_Prescans = 1  
X\_Resolution = 11.38715602[Hz]  
X\_Sweep = 186.56716418[kHz]  
Irr\_Domain = 19F  
Irr\_Freq = 372.50336686[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = 19F  
Tri\_Freq = 372.50336686[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 21.5[dC]  
X\_90\_Width = 13.9[us]  
X\_Acq\_Time = 87.81824[ms]  
X\_Angle = 45[deg]  
X\_Atn = 4[dB]  
X\_Pulse = 6.95[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
DanE\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 5.08781824[s]

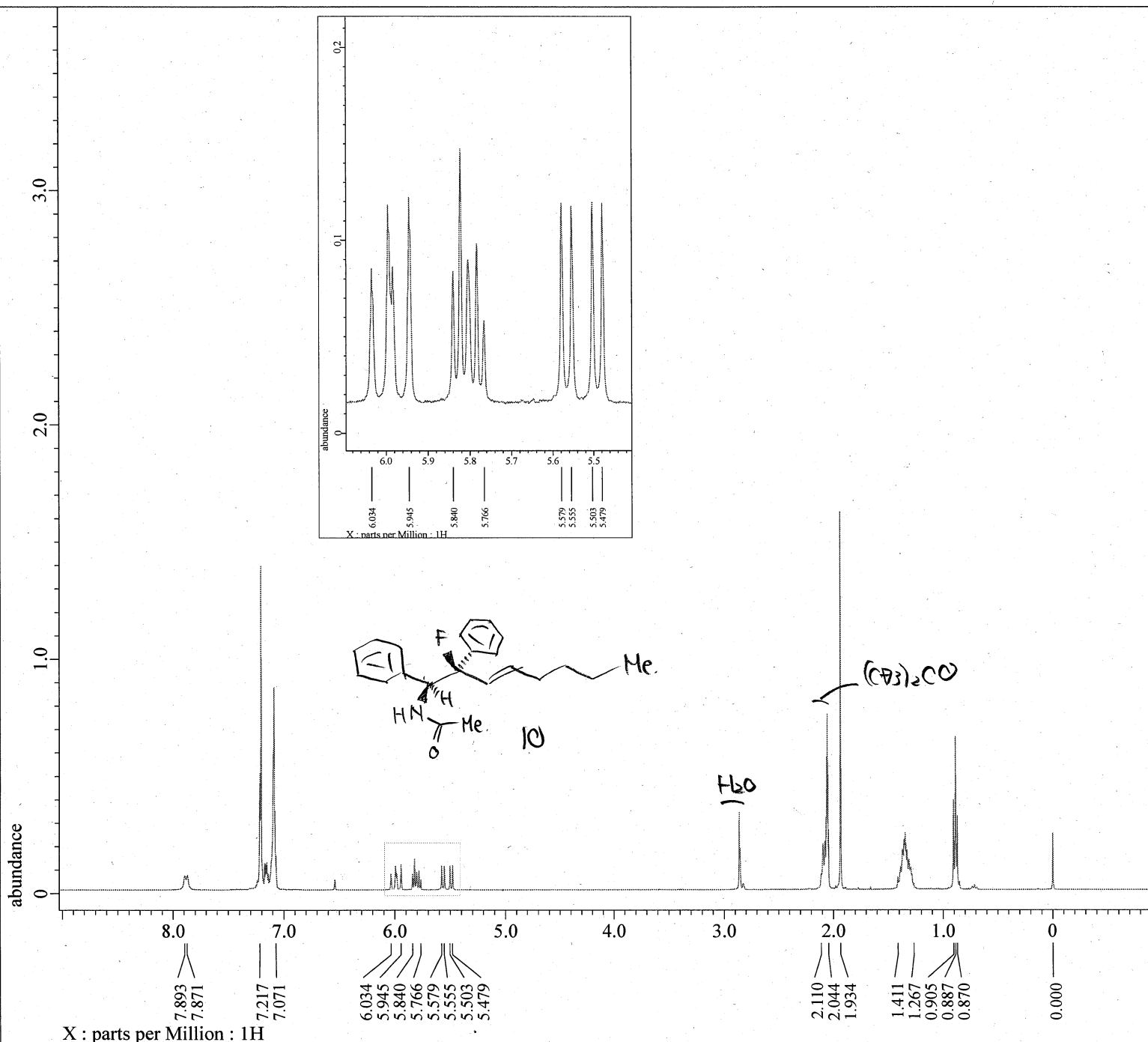
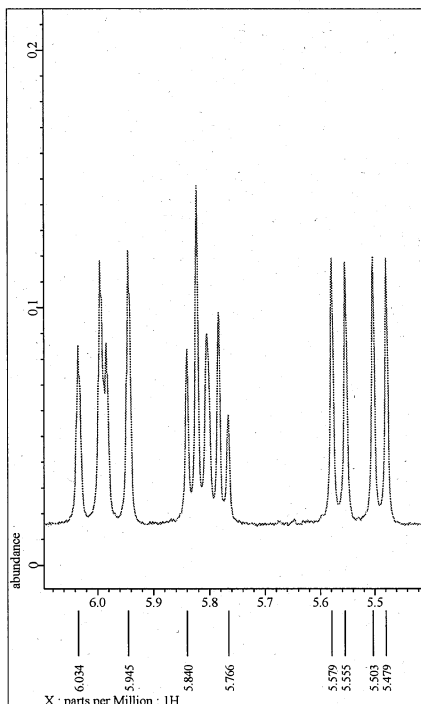


Filename = AKY832-pure-again-2.jdf  
Author = element  
Experiment = single\_pulse.ex2  
Sample Id = S#583695  
Solvent = ACETONE-D6  
Actual\_Start\_Time = 2-MAR-2019 00:43:40  
Revision\_Time = 4-MAR-2019 16:28:22

Comment = single pulse  
Data Format = 1D\_COMPLEX  
Dim Size = 13107  
X Domain = 1H  
Dim Title = 1H  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

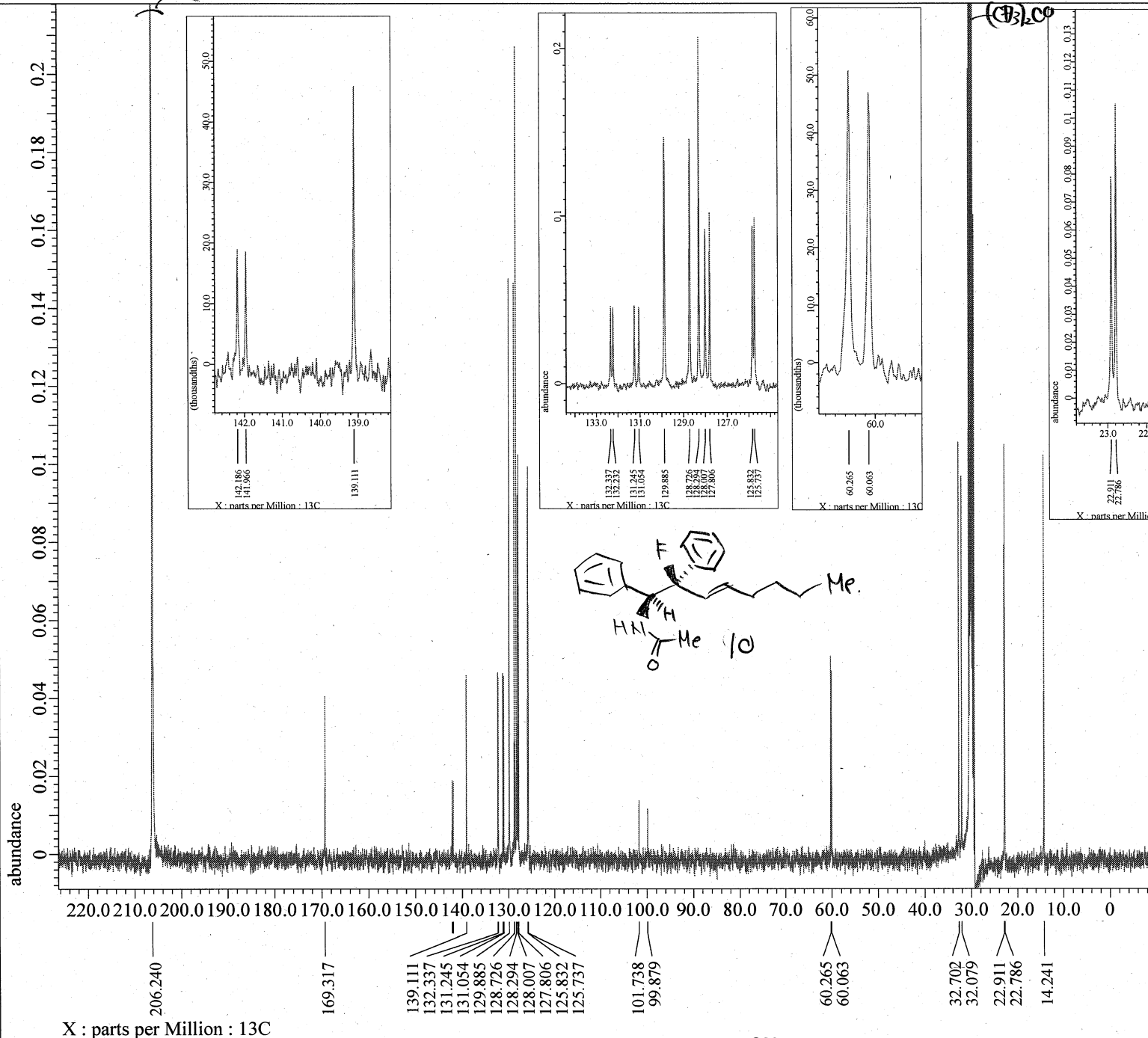
Field Strength = 9.2982153[T] (400[MHz])  
X Acq\_Duration = 2.20725248[s]  
X Domain = 1H  
X Freq = 395.88430144 [MHz]  
X Offset = 5 [ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 0.45305193 [Hz]  
X Sweep = 7.42280285 [kHz]  
Irr Domain = 1H  
Irr Freq = 395.88430144 [MHz]  
Irr Offset = 5 [ppm]  
Tri\_Domain = 1H  
Tri Freq = 395.88430144 [MHz]  
Tri Offset = 5 [ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5 [s]  
Recvr Gain = 36  
Temp Get = 20 [dC]  
X 90\_Width = 13.2 [us]  
X Acq\_Time = 2.20725248 [s]  
X Angle = 45 [deg]  
X Atn = 3.5 [dB]  
X Pulse = 6.6 [us]  
Irr Mode = Off  
Tri Mode = Off  
Dante\_Presat = FALSE  
Initial Wait = 1 [s]  
Repetition\_Time = 7.20725248 [s]



X : parts per Million : 1H

(F3)2CO



Filename = AKY832-carbom1024-2.jdf  
 Author = element  
 Experiment = single\_pulse\_dec  
 Sample Id = S#610014  
 Solvent = ACETONE-D6  
 Actual Start Time = 2-MAR-2019 01:26:46  
 Revision Time = 4-MAR-2019 16:52:28  
  
 Comment = single pulse decoupled ga  
 Data Format = 1D COMPLEX  
 Dim Size = 26214  
 X Domain = 13C  
 Dim Title = 13C  
 Dim Units = [ppm]  
 Dimensions = X  
 Site = ECX 400P  
 Spectrometer = DELTA2\_NMR  
  
 Field Strength = 9.2982153[T] (400 [MHz])  
 X Acq Duration = 1.048576[s]  
 X Domain = 13C  
 X Freq = 99.54517646 [MHz]  
 X Offset = 100 [ppm]  
 X Points = 32768  
 X Prescans = 4  
 X Resolution = 0.95367432 [Hz]  
 X Sweep = 31.25 [kHz]  
 Irf Domain = 1H  
 Irf Freq = 395.88430144 [MHz]  
 Irf Offset = 5 [ppm]  
 Clipped = FALSE  
 Scans = 1024  
 Total\_Scans = 1024  
  
 Relaxation\_Delay = 2[s]  
 Recvr Gain = 54  
 Temp Get = 20.4 [dC]  
 X 90 Width = 10.1 [us]  
 X Acq Time = 1.048576[s]  
 X Angle = 30 [deg]  
 X Atn = 3.4 [dB]  
 X Pulse = 3.36666667 [us]  
 Irf Atn Dec = 22.3 [dB]  
 Irf Atn Noe = 22.3 [dB]  
 Irf Noise = WALTZ  
 Decoupling = TRUE  
 Initial Wait = 1[s]  
 Noe = TRUE  
 Noe Time = 2[s]  
 Repetition Time = 3.048576[s]

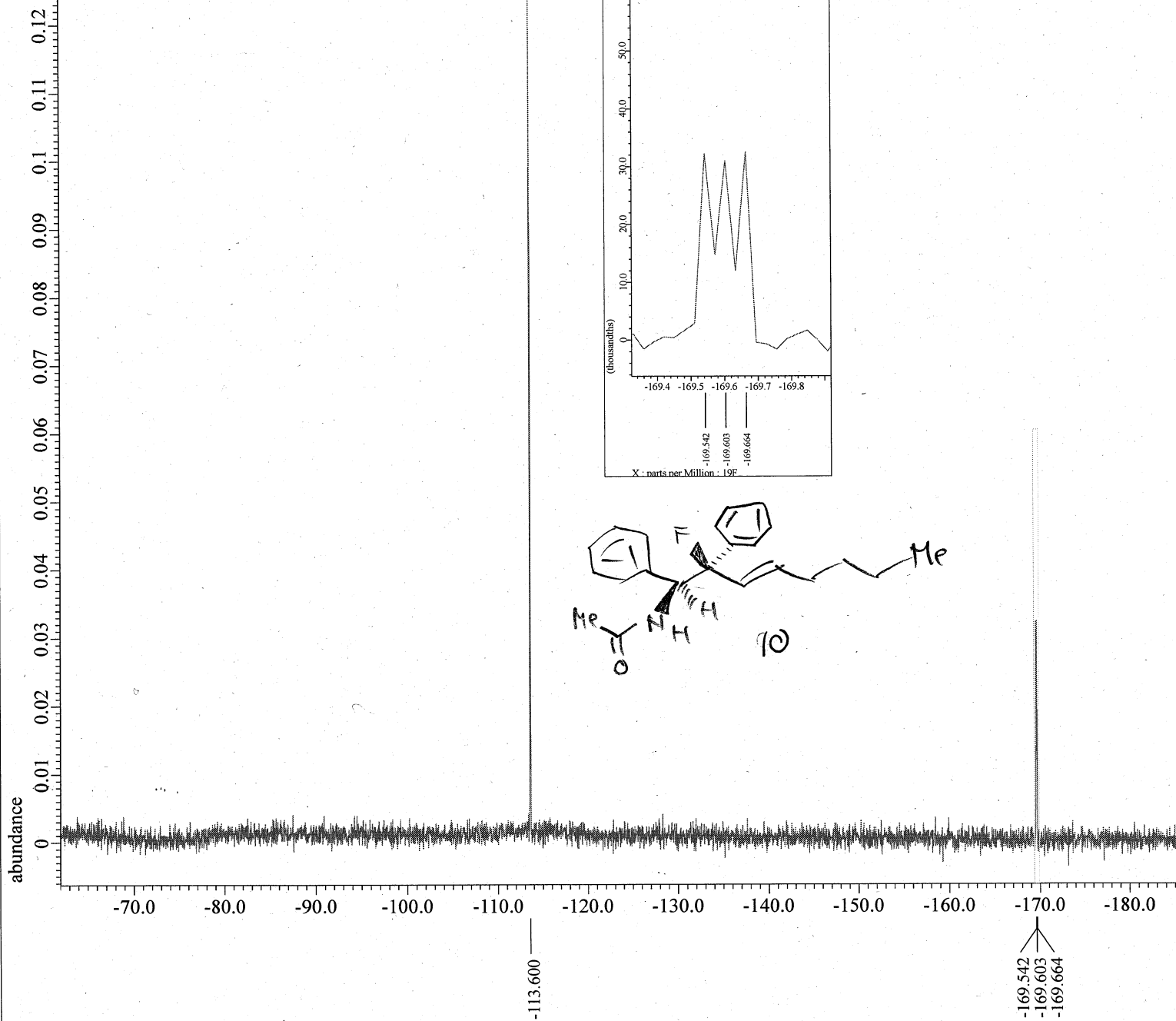
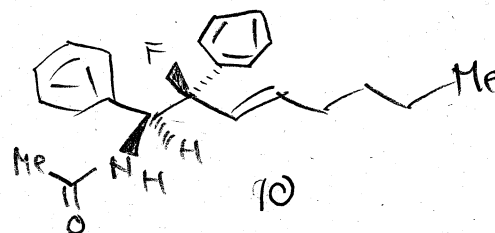
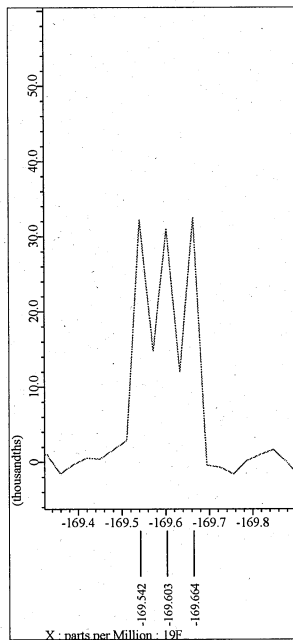


Filename = AKY832-pure-FNMrag-2.jdf  
Author = element  
Experiment = single pulse.ex2  
Sample Id = S#501749  
Solvent = ACETONE-D6  
Actual Start Time = 8-MAR-2019 22:26:55  
Revision Time = 25-MAR-2019 20:55:45

Comment = single pulse  
Data Format = 1D COMPLEX  
Dim Size = 13107  
X Domain = 19F  
Dim Title = 19F  
Dim Units = [ppm]  
Dimensions = X  
Site = ECX 400P  
Spectrometer = DELTA2\_NMR

Field Strength = 9.2982153[T] (400[MHz])  
X Acq Duration = 87.81824[ms]  
X Domain = 19F  
X Freq = 372.50336686[MHz]  
X Offset = 0[ppm]  
X Points = 16384  
X Prescans = 1  
X Resolution = 11.38715602[Hz]  
X Sweep = 186.56716418[kHz]  
Irr Domain = 19F  
Irr Freq = 372.50336686[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = 19F  
Tri Freq = 372.50336686[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 8

Relaxation Delay = 5[s]  
Recvr Gain = 24  
Temp Get = 19.5[dC]  
X 90 Width = 13.9[us]  
X Acq Time = 87.81824[ms]  
X Angle = 45[deg]  
X Atn = 4[dB]  
X Pulse = 6.95[us]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Initial Wait = 1[s]  
Repetition Time = 5.08781824[s]



X : parts per Million : 19F



### Chromaster System Manager Report

Analyzed Date and Time: 2018/11/10  
12:37

Reported Date and Time: 2018/11/10  
14:03:10

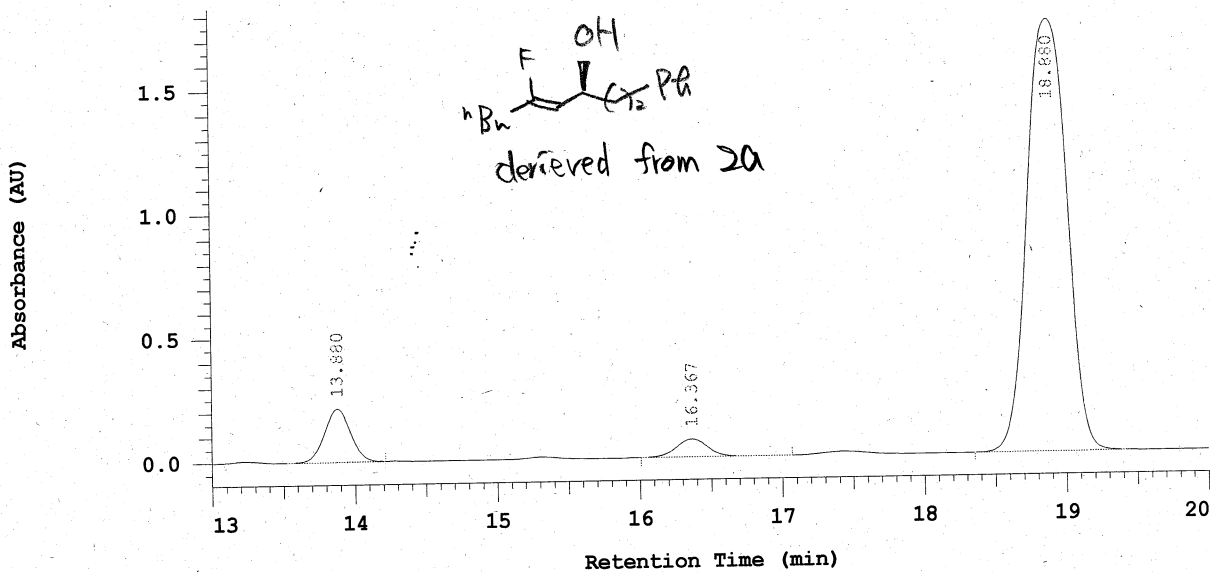
Processed Date and Time: 2018/11/10  
14:02

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0086\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1  
Application(data): AKY  
Sample Name: AKY723-chiral-nBuPh-IC-4%  
Injection from this vial: 1 of 1  
Sample Description:

Series: 0086  
Vial Number: 21  
Vial Type: UNK  
Volume: 10.0 ul

Chrom Type: Fixed WL Chromatogram, 207 nm



Processing Method: column3(IC-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 207 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 13.880 | 1363396  | 7.211   | BB |
| 2   | 16.367 | 548834   | 2.903   | BB |
| 3   | 18.880 | 16994292 | 89.886  | BB |
|     |        | 18906522 | 100.000 |    |

Peak rejection level: 0

## Chromaster System Manager Report

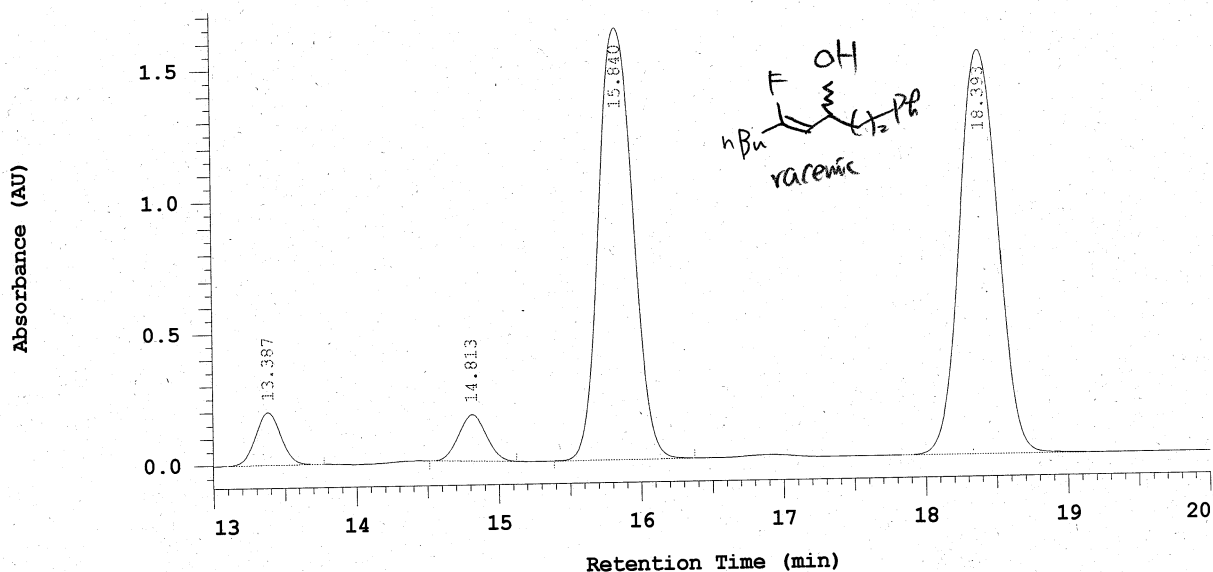
Analyzed Date and Time: 2018/11/10 11:36      Reported Date and Time: 2018/11/10 13:39:49

Processed Date and Time: 2018/11/10 13:39

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0085\  
 Processing Method: column3(IC-3)

System (acquisition): Sys 1      Series: 0085  
 Application(data): AKY      Vial Number: 20  
 Sample Name: AKY721-rac-nBuPh-IC-4%      Vial Type: UNK  
 Injection from this vial: 1 of 1      Volume: 10.0 ul  
 Sample Description:

Chrom Type: Fixed WL Chromatogram, 207 nm



Processing Method: column3(IC-3)  
 Method Developer:  
 Pump 1: 5110

Pump 1 Solvent A: hexane  
 Pump 1 Solvent C:

Pump 1 Solvent B: 2-propanol  
 Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 207 nm

Peak Quantitation: AREA  
 Calculation Method: AREA%

| No.      | RT     | Area     | Conc 1  | BC |
|----------|--------|----------|---------|----|
| 1        | 13.387 | 1247840  | 4.074   | MC |
| 2        | 14.813 | 1190811  | 3.887   | MC |
| 3        | 15.840 | 13771837 | 44.959  | MC |
| 4        | 18.393 | 14421539 | 47.080  | BB |
| 30632027 |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/11 19:31

Reported Date and Time: 2018/10/11 20:32:50

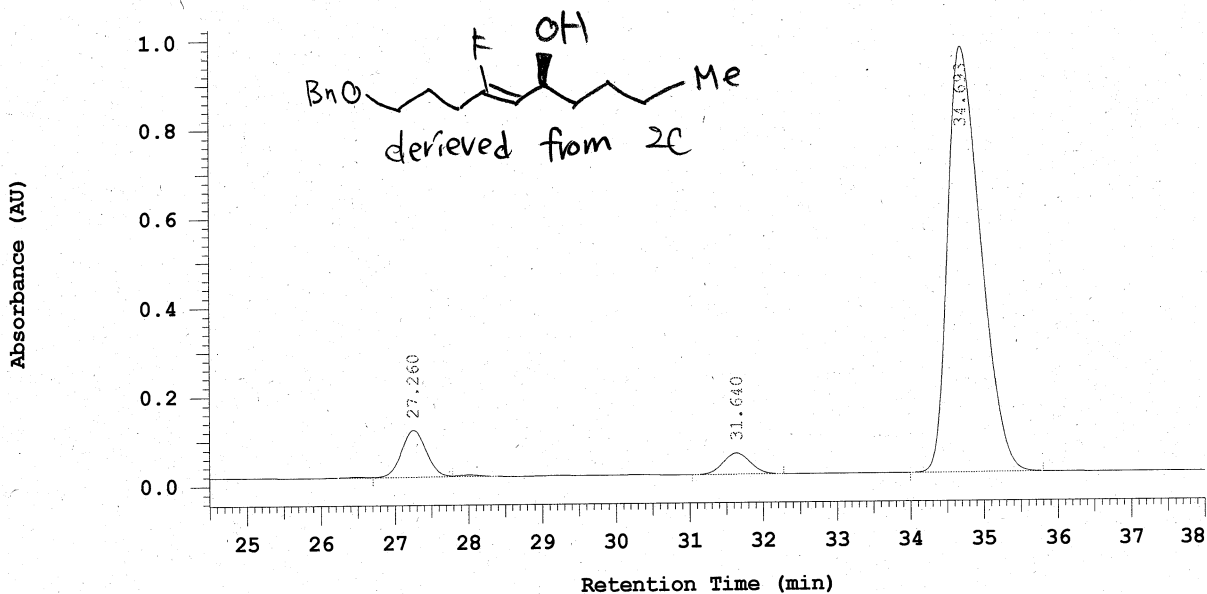
Processed Date and Time: 2018/10/11 20:32

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0047\_Bn-chiral\  
 Processing Method: column2 (IBN-3)

System (acquisition): Sys 1  
 Application (data): AKY  
 Sample Name: AKY688-chiralBn-IBN3-2%  
 Injection from this vial: 1 of 1  
 Sample Description:

Series: 0047\_Bn-chiral  
 Vial Number: 9  
 Vial Type: UNK  
 Volume: 10.0 ul

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column2 (IBN-3)  
 Method Developer:  
 Pump 1: 5110  
 Pump 1 Solvent A: hexane  
 Pump 1 Solvent C:  
 Method Description:

Pump 1 Solvent B: 2-propanol  
 Pump 1 Solvent D:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA  
 Calculation Method: AREA%

| No. | RT     | Area     | Conc 1   | BC      |
|-----|--------|----------|----------|---------|
| 1   | 27.260 | 1245271  | 7.408    | VV      |
| 2   | 31.640 | 632459   | 3.762    | BB      |
| 3   | 34.693 | 14932712 | 88.830   | BB      |
|     |        |          | 16810442 | 100.000 |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/11 16:36

Reported Date and Time: 2018/10/11 20:26:41

Processed Date and Time: 2018/10/11 20:26

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0045\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0045

Application(data): AKY

Vial Number: 10

Sample Name: AKY678-racBn-IBN3-2%-ag

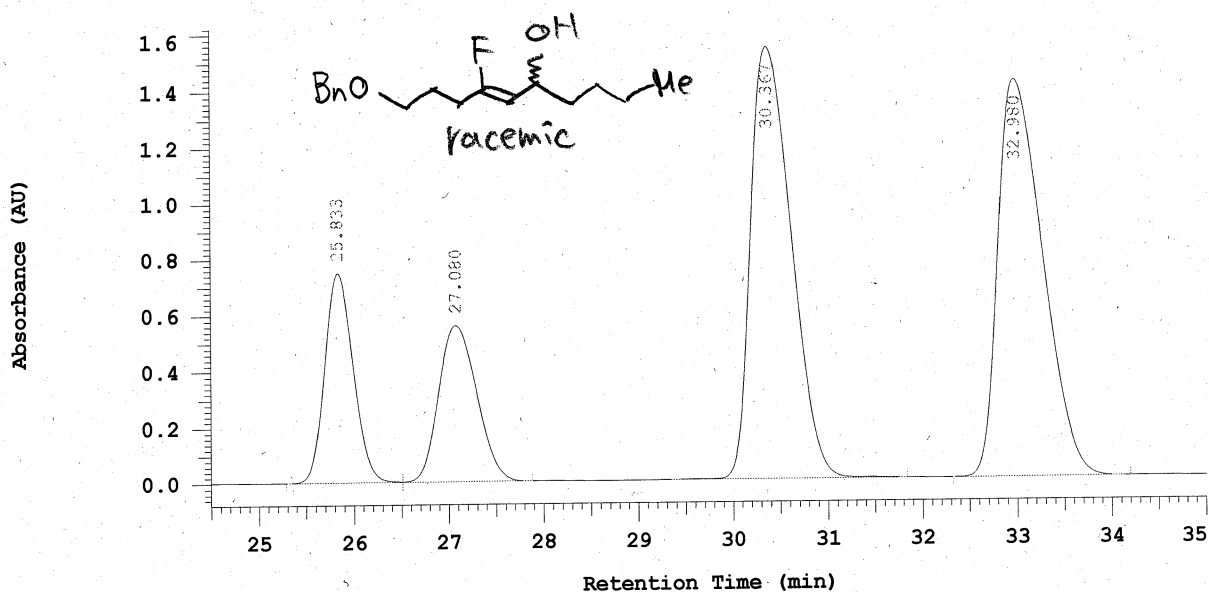
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 25.833 | 7782850  | 12.622  | BV |
| 2   | 27.080 | 7807706  | 12.663  | VB |
| 3   | 30.367 | 22791091 | 36.963  | VB |
| 4   | 32.980 | 23276922 | 37.751  | BB |
|     |        | 61658569 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/12  
01:01

Reported Date and Time: 2018/10/12  
09:18:49

Processed Date and Time: 2018/10/12  
09:17

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0051\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1

Series: 0051

Application(data): AKY

Vial Number: 12

Sample Name: AKY689-chiralTBS-IC3-1.5%

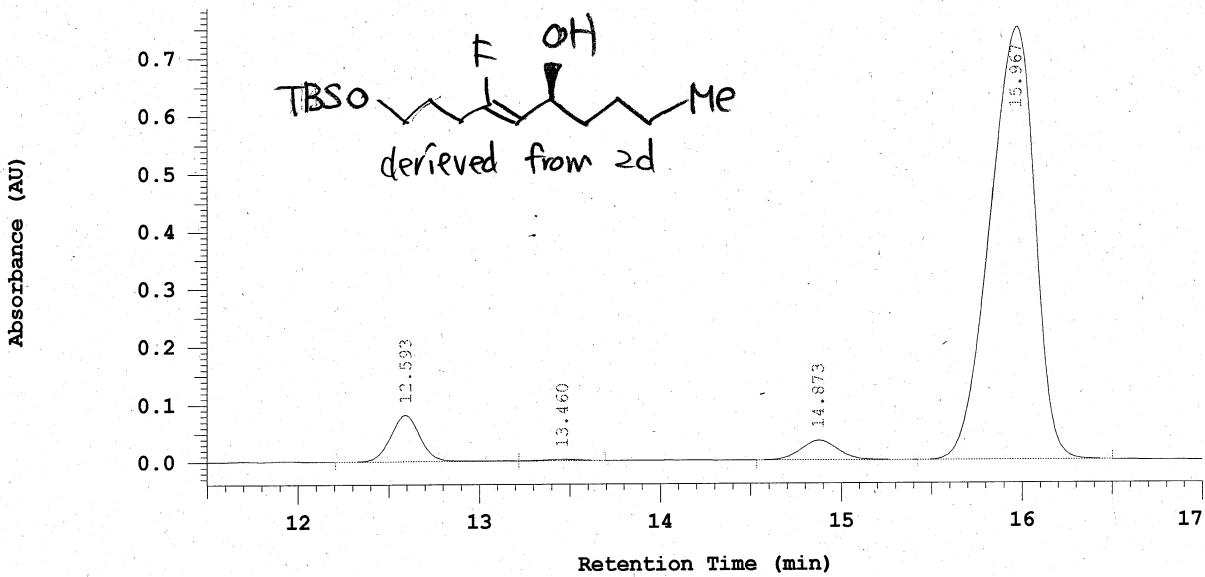
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column3(IC-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 12.593 | 468461  | 6.530   | BB |
| 2   | 13.460 | 15507   | 0.216   | BB |
| 3   | 14.873 | 246761  | 3.440   | BB |
| 4   | 15.967 | 6443397 | 89.814  | BB |
|     |        | 7174126 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/12  
00:09

Reported Date and Time: 2018/10/12  
09:08:31

Processed Date and Time: 2018/10/12  
09:08

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0050\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1

Series: 0050

Application(data): AKY

Vial Number: 11

Sample Name: AKY679-ractTBS-IC3-1.5%-ag

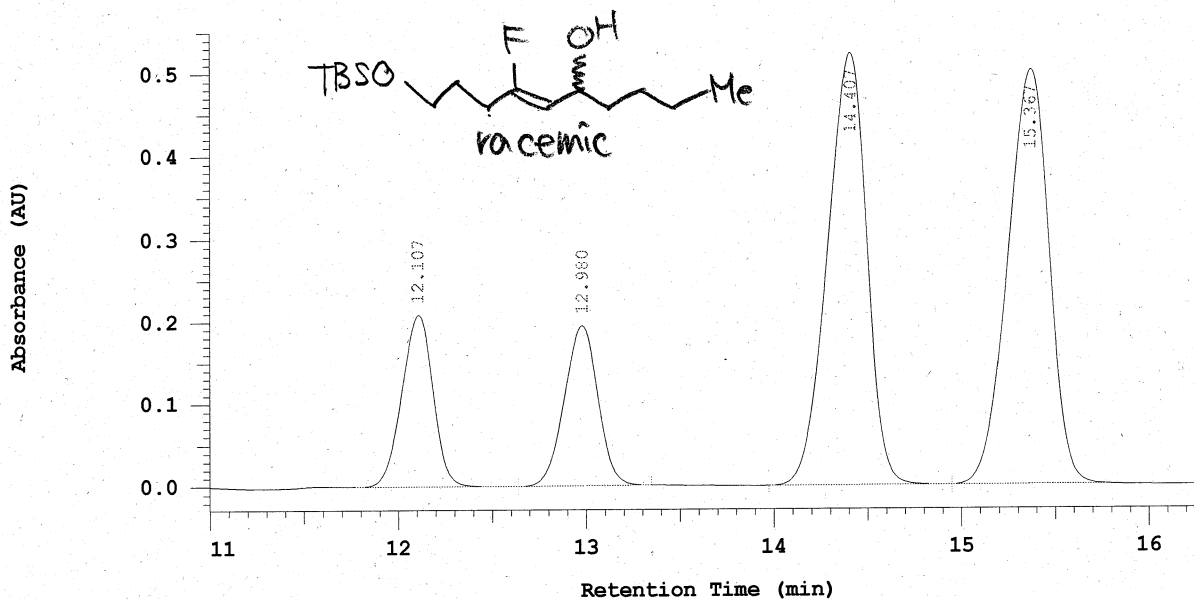
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column3(IC-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 12.107 | 1206899  | 11.970  | BB |
| 2   | 12.980 | 1207290  | 11.974  | BB |
| 3   | 14.407 | 3831661  | 38.002  | BB |
| 4   | 15.367 | 3836825  | 38.054  | BB |
|     |        | 10082675 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/09/21 15:22

Reported Date and Time: 2018/10/02 15:30:14

Processed Date and Time: 2018/10/02 15:29

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0025Ac-chiral\  
 Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0025Ac-chiral

Application(data): AKY

Vial Number: 11

Sample Name: AKY669-chiral-IF-3-4%-60min

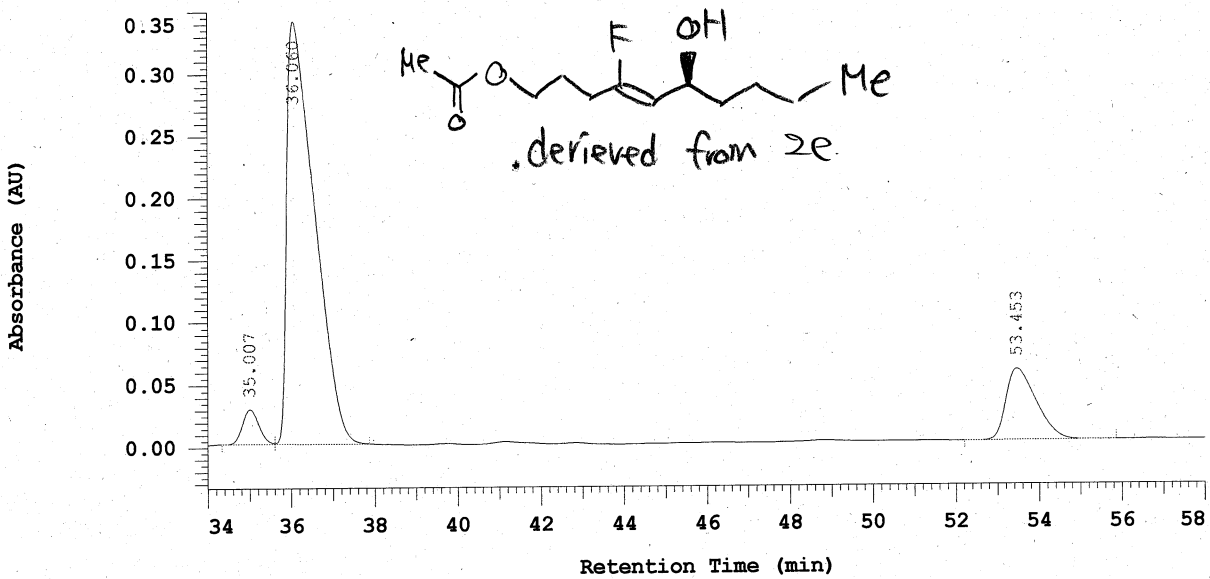
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 35.007 | 386631  | 4.006   | MC |
| 2   | 36.060 | 7816462 | 80.981  | MC |
| 3   | 53.453 | 1449071 | 15.013  | MC |
|     |        |         | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/09/21 14:01

Reported Date and Time: 2018/10/02 15:28:52

Processed Date and Time: 2018/10/02 15:28

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0024Ac-rac\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0024Ac-rac

Application(data): AKY

Vial Number: 10

Sample Name: AKY665-rac-IF-3-4%-60min

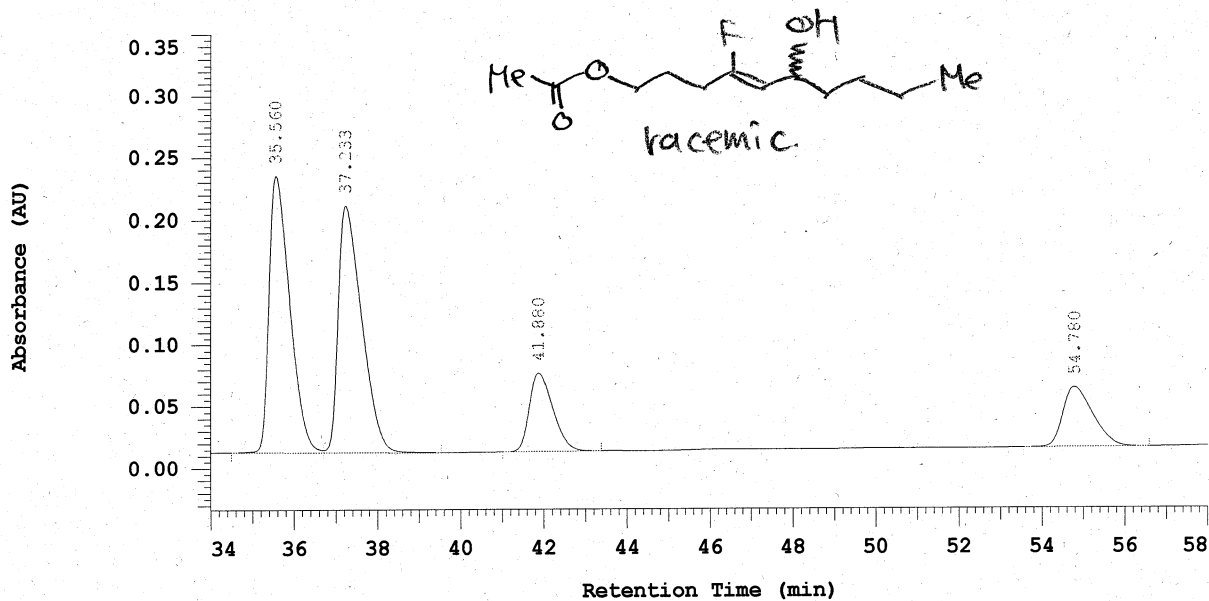
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1   | BC      |
|-----|--------|---------|----------|---------|
| 1   | 35.560 | 3879244 | 37.938   | BV      |
| 2   | 37.233 | 3895764 | 38.100   | VB      |
| 3   | 41.880 | 1228750 | 12.017   | BB      |
| 4   | 54.780 | 1221370 | 11.945   | BB      |
|     |        |         | 10225128 | 100.000 |

Peak rejection level: 0



### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/24  
23:23

Reported Date and Time: 2018/10/25  
09:04:17

Processed Date and Time: 2018/10/25  
09:03

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0066\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1

Series: 0066

Application(data): AKY

Vial Number: 15

Sample Name: AKY701-chiralNPhth-IE3-  
1.5%

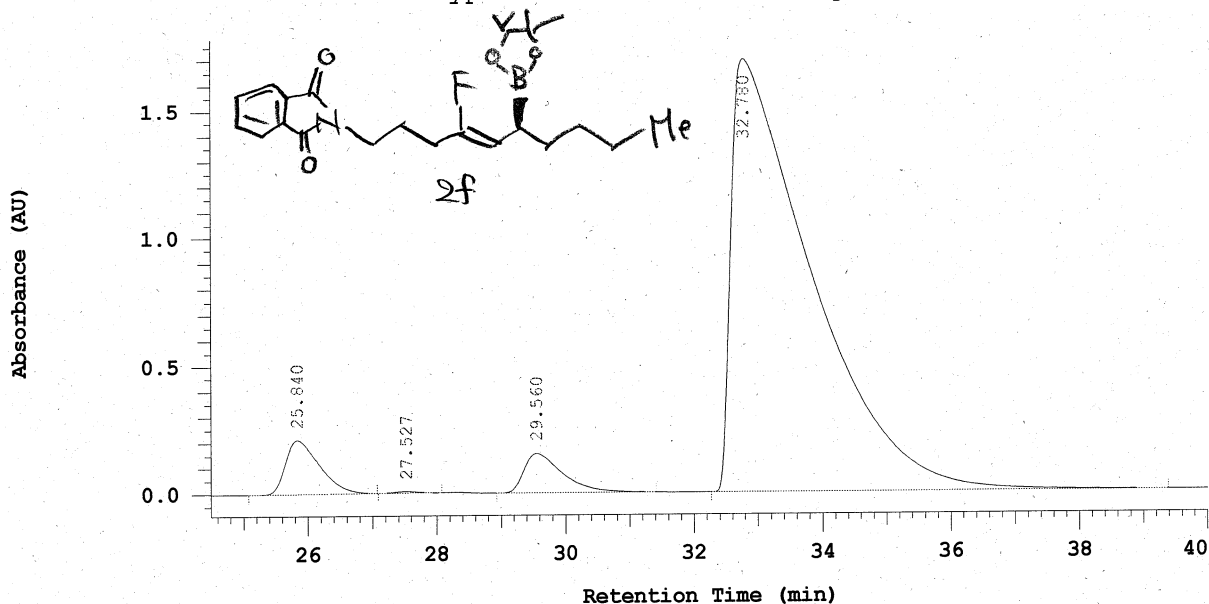
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 216 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 216 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 25.840 | 3903780  | 4.835   | MC |
| 2   | 27.527 | 75138    | 0.093   | MC |
| 3   | 29.560 | 3214553  | 3.981   | MC |
| 4   | 32.780 | 73550499 | 91.091  | BB |
|     |        | 80743970 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/10/24  
21:51

Reported Date and Time: 2018/10/25  
09:01:06

Processed Date and Time: 2018/10/24  
22:53

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0065\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1

Series: 0065

Application(data): AKY

Vial Number: 13

Sample Name: AKY691-racNPhth-IE3-1.5%

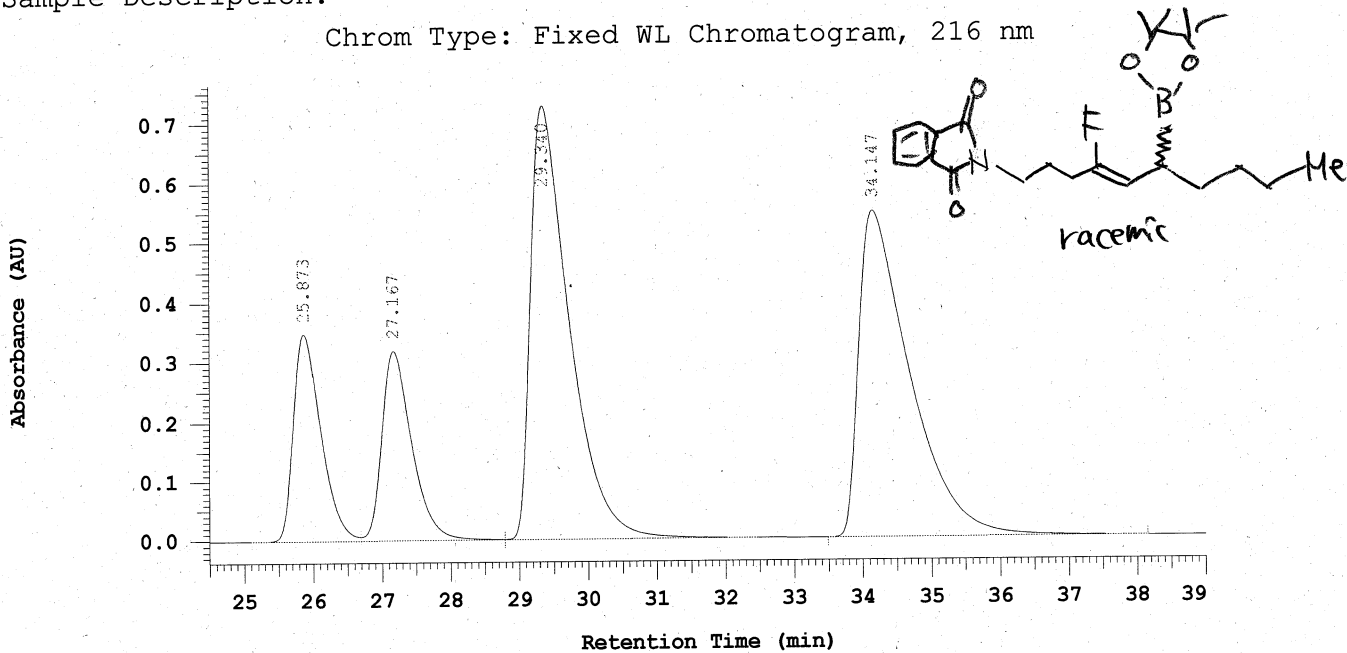
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 216 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 216 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 25.873 | 4837142  | 12.681  | BV |
| 2   | 27.167 | 4898065  | 12.841  | VV |
| 3   | 29.340 | 14192446 | 37.206  | VB |
| 4   | 34.147 | 14217643 | 37.272  | BB |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/08/29  
15:33

Reported Date and Time: 2019/03/14  
11:28:15

Processed Date and Time: 2019/03/14  
11:27

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0005\

Processing Method: 20180824\_column2 (IBN-3)

System (acquisition): Sys 1

Series: 0005

Application(data): AKY

Vial Number: 4

Sample Name: AKY650-IBN3-5%

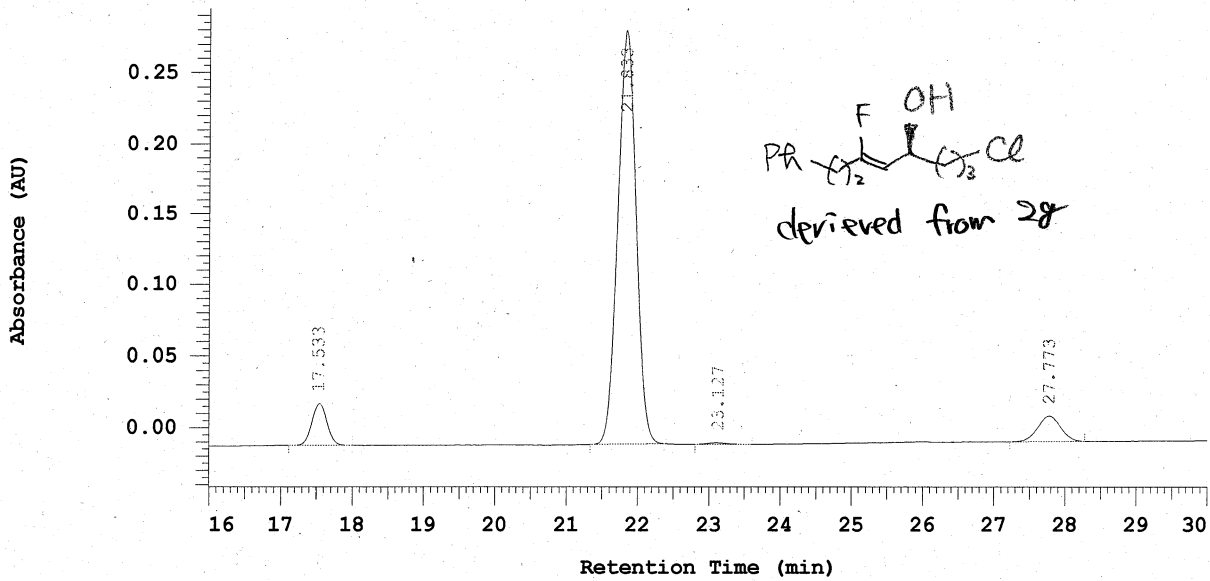
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: 20180824\_column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 17.533 | 211675  | 6.950   | MC |
| 2   | 21.833 | 2626639 | 86.245  | BB |
| 3   | 23.127 | 8354    | 0.274   | MC |
| 4   | 27.773 | 198876  | 6.530   | BB |
|     |        |         | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/08/24  
17:32

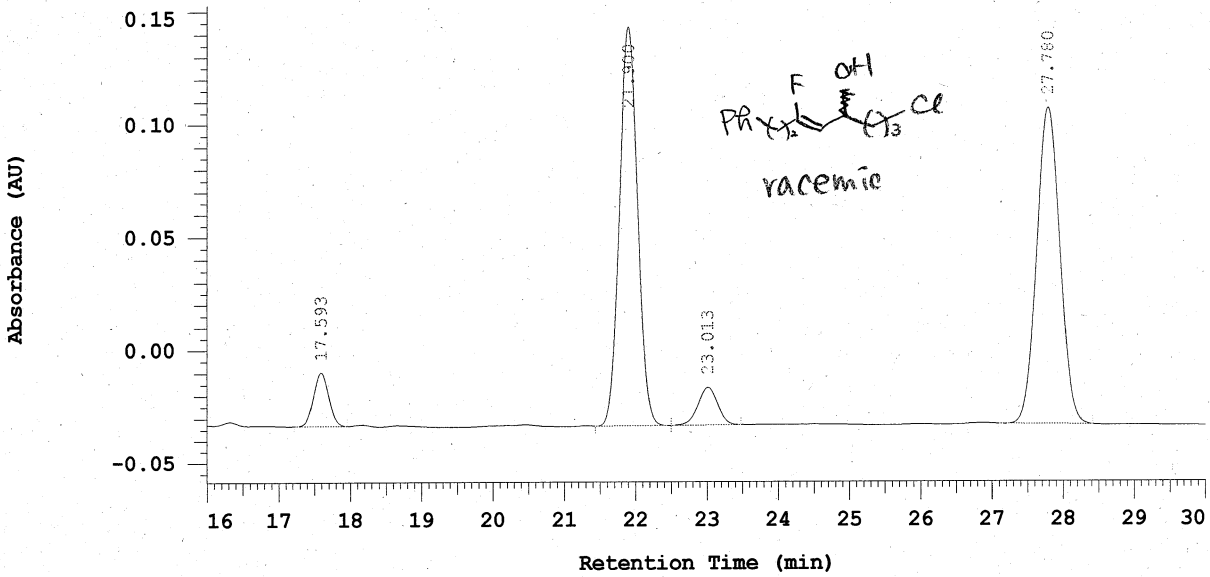
Reported Date and Time: 2019/03/14  
11:28:41

Processed Date and Time: 2019/03/14  
11:28

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0002\  
Processing Method: 20180824\_column2 (IBN-3)

System (acquisition): Sys 1                      Series: 0002  
Application (data): AKY                          Vial Number: 1  
Sample Name: AKY648-IBN3-5%                  Vial Type: UNK  
Injection from this vial: 1 of 1                Volume: 10.0 ul  
Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: 20180824\_column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC      |
|-----|--------|---------|---------|---------|
| 1   | 17.593 | 175114  | 5.009   | MC      |
| 2   | 21.900 | 1575645 | 45.067  | BB      |
| 3   | 23.013 | 160155  | 4.581   | BB      |
| 4   | 27.780 | 1585278 | 45.343  | BB      |
|     |        |         | 3496192 | 100.000 |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/08/24  
21:04

Reported Date and Time: 2019/03/23  
16:18:36

Processed Date and Time: 2019/03/23  
16:18

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0004\  
Processing Method: column1(IA-3)

System (acquisition): Sys 1

Series: 0004

Application(data): AKY

Vial Number: 3

Sample Name: AKY633-IBN3-5%

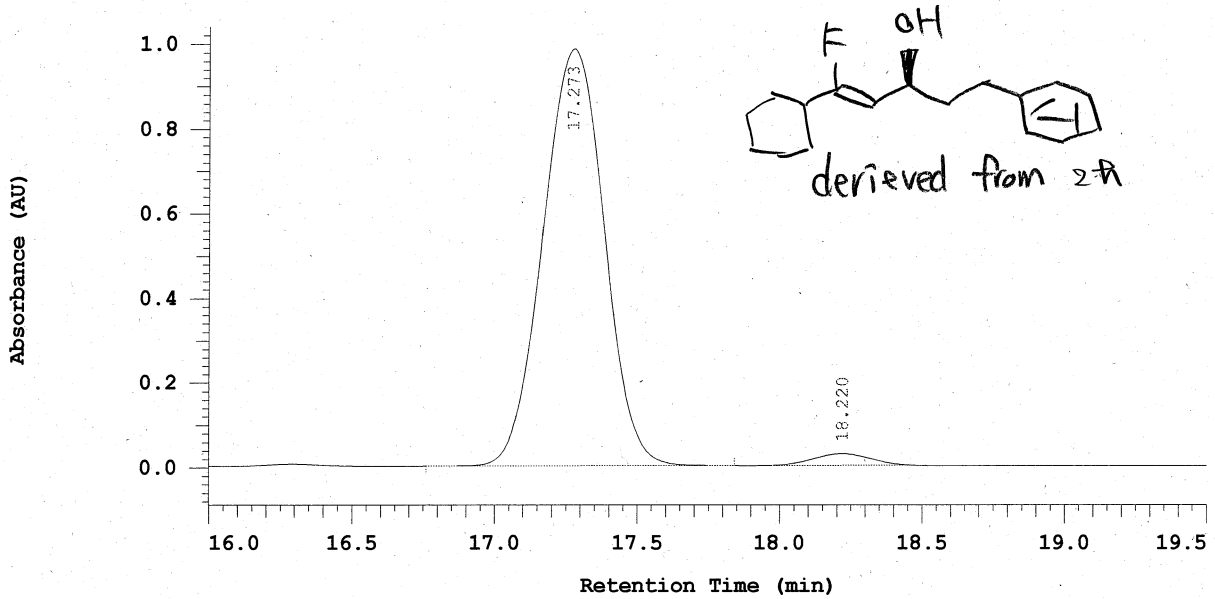
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column1(IA-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 17.273 | 7326901 | 97.486  | MC |
| 2   | 18.220 | 188971  | 2.514   | MC |
|     |        | 7515872 | 100.000 |    |

Peak rejection level: 0

**Chromaster System Manager Report**Analyzed Date and Time: 2018/08/24  
20:12Reported Date and Time: 2019/03/23  
16:17:45Processed Date and Time: 2019/03/23  
16:17

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0003\

Processing Method: column1(IA-3)

System (acquisition): Sys 1

Series: 0003

Application(data): AKY

Vial Number: 2

Sample Name: AKY630-IBN3-5%

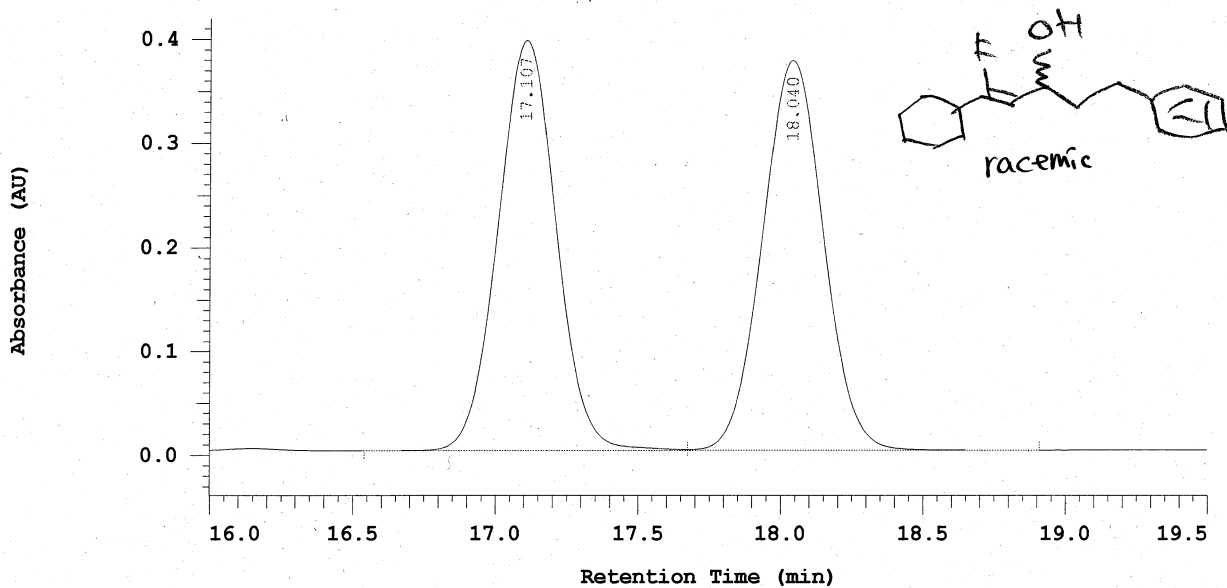
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column1(IA-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 17.107 | 2788372 | 50.135  | BV |
| 2   | 18.040 | 2773376 | 49.865  | VB |
|     |        | 5561748 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/12  
18:59

Reported Date and Time: 2019/03/23  
16:27:03

Processed Date and Time: 2019/03/23  
16:26

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0247\_Me-chiral\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1

Series: 0247\_Me-chiral

Application(data): AKY

Vial Number: 103

Sample Name: AKY840-MeAcchiral-IE-3%

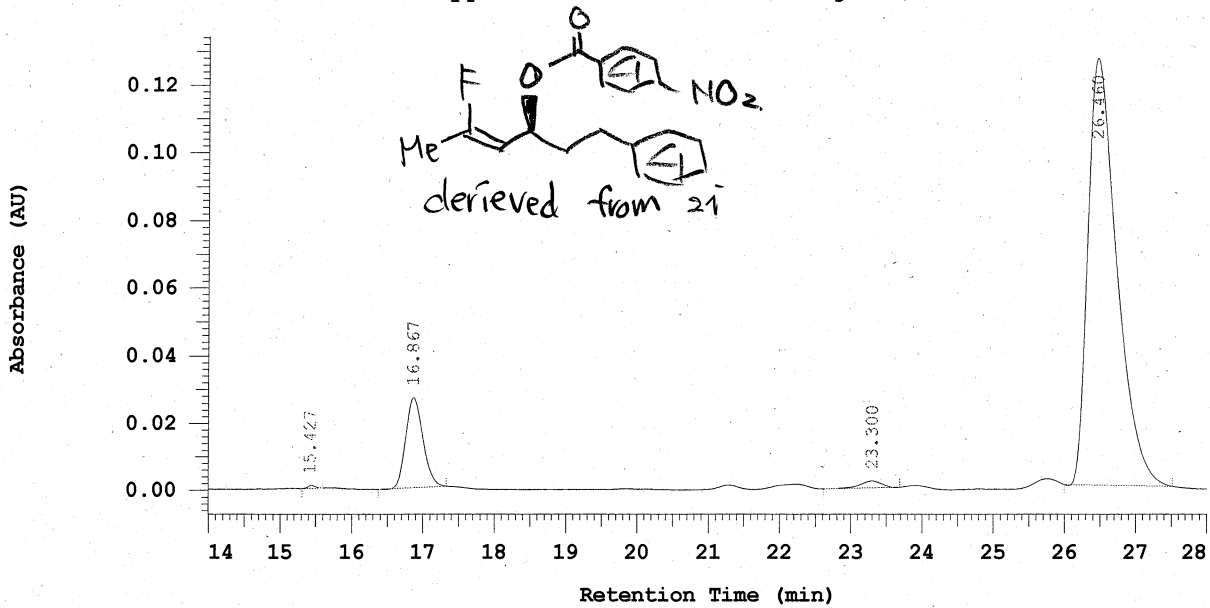
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC      |
|-----|--------|---------|---------|---------|
| 1   | 15.427 | 2929    | 0.144   | MC      |
| 2   | 16.867 | 226530  | 11.103  | MC      |
| 3   | 23.300 | 21534   | 1.056   | MC      |
| 4   | 26.460 | 1789202 | 87.698  | MC      |
|     |        |         | 2040195 | 100.000 |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/12  
13:57

Reported Date and Time: 2019/03/23  
16:26:19

Processed Date and Time: 2019/03/23  
16:26

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0243\_Me-rac\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1

Series: 0243\_Me-rac

Application(data): AKY

Vial Number: 101

Sample Name: AKY588-MeAc-rac-IE-3%

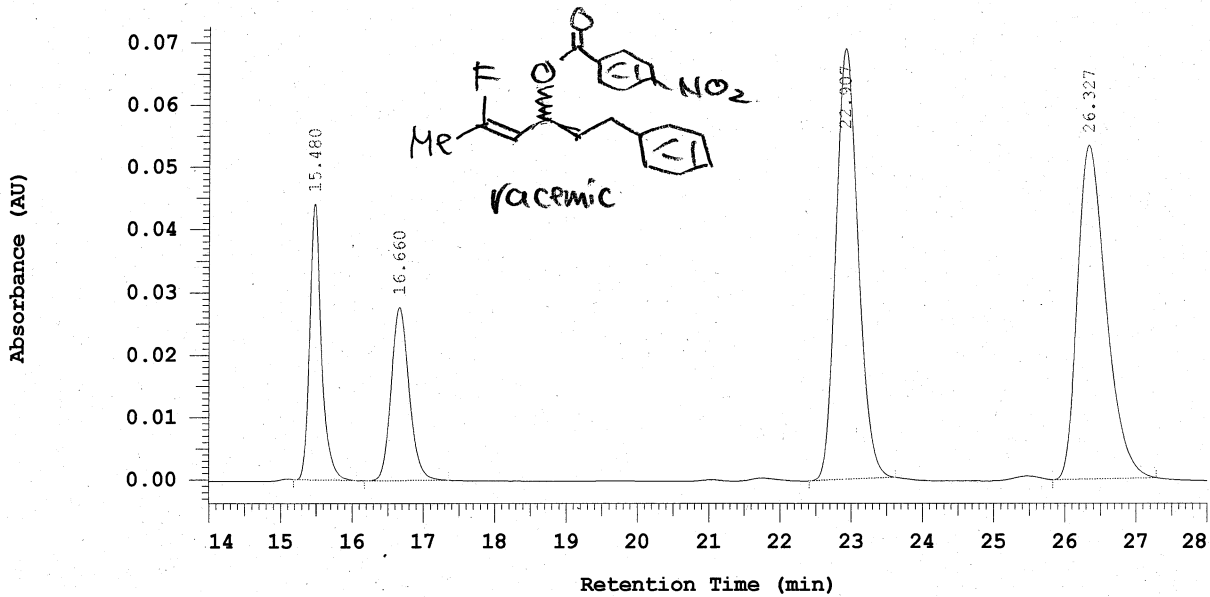
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area   | Conc 1  | BC      |
|-----|--------|--------|---------|---------|
| 1   | 15.480 | 245792 | 12.295  | MC      |
| 2   | 16.660 | 246380 | 12.324  | MC      |
| 3   | 22.907 | 758406 | 37.937  | MC      |
| 4   | 26.327 | 748563 | 37.444  | MC      |
|     |        |        | 1999141 | 100.000 |

Peak rejection level: 0



### Chromaster System Manager Report

Analyzed Date and Time: 2018/08/29  
21:17

Reported Date and Time: 2019/03/23  
16:23:51

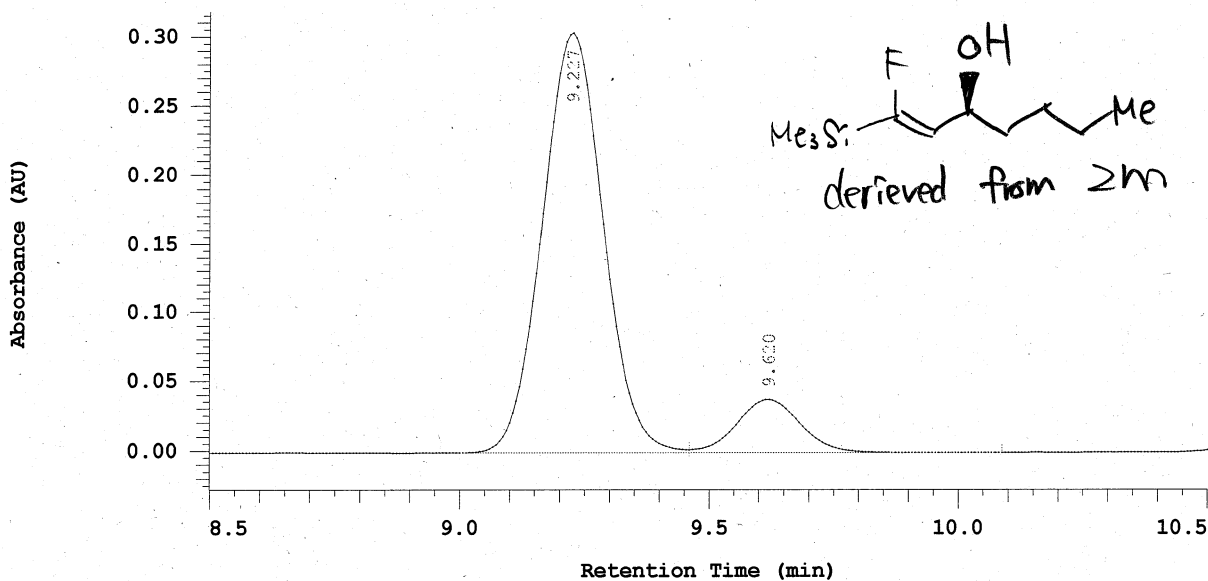
Processed Date and Time: 2019/03/23  
16:23

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0011\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1  
Application(data): AKY  
Sample Name: AKY652-IE3-3%-chiral  
Injection from this vial: 1 of 1  
Sample Description:

Series: 0011  
Vial Number: 6  
Vial Type: UNK  
Volume: 10.0 ul

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT    | Area    | Conc 1  | BC |
|-----|-------|---------|---------|----|
| 1   | 9.227 | 1283594 | 88.470  | BV |
| 2   | 9.620 | 167280  | 11.530  | VB |
|     |       | 1450874 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/08/29  
20:38

Reported Date and Time: 2019/03/23  
16:23:06

Processed Date and Time: 2019/03/23  
16:22

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0010\  
Processing Method: column5(IE-3)

System (acquisition): Sys 1

Series: 0010

Application(data): AKY

Vial Number: 5

Sample Name: AKY651-IE3-3%-rac

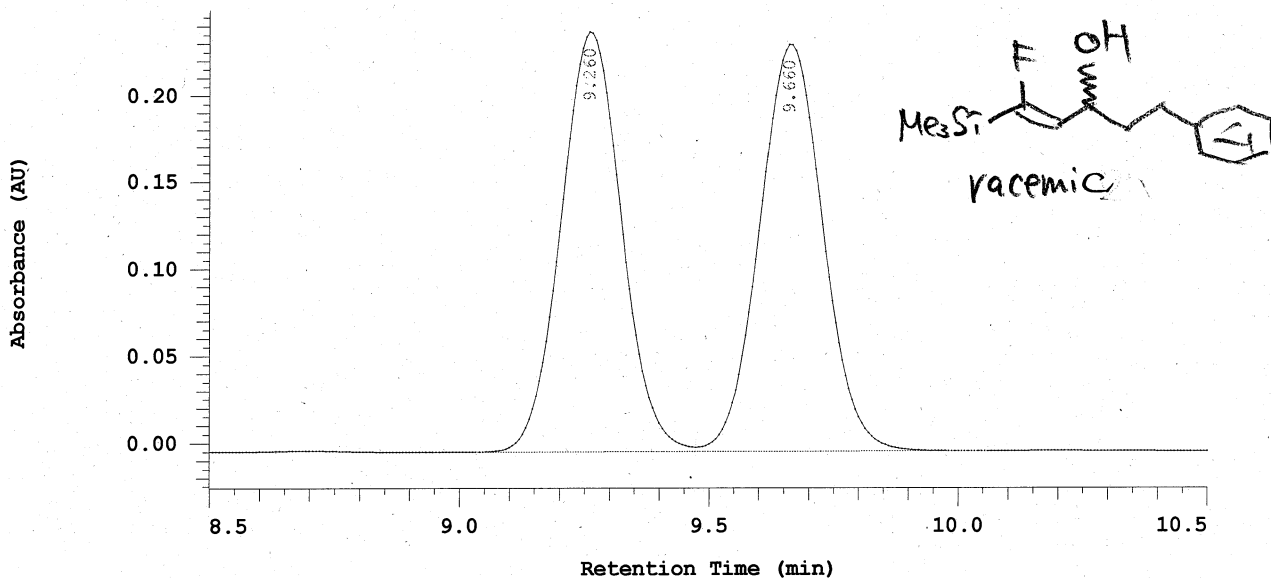
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column5(IE-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT    | Area    | Conc 1  | BC |
|-----|-------|---------|---------|----|
| 1   | 9.260 | 1015375 | 49.640  | BV |
| 2   | 9.660 | 1030087 | 50.360  | VB |
|     |       | 2045462 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/26  
14:17

Reported Date and Time: 2019/03/26  
15:29:17

Processed Date and Time: 2019/03/26  
15:28

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0272\

Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0272

Application (data): AKY

Vial Number: 114

Sample Name: AKY851-chiral-acyl-IBN-3%

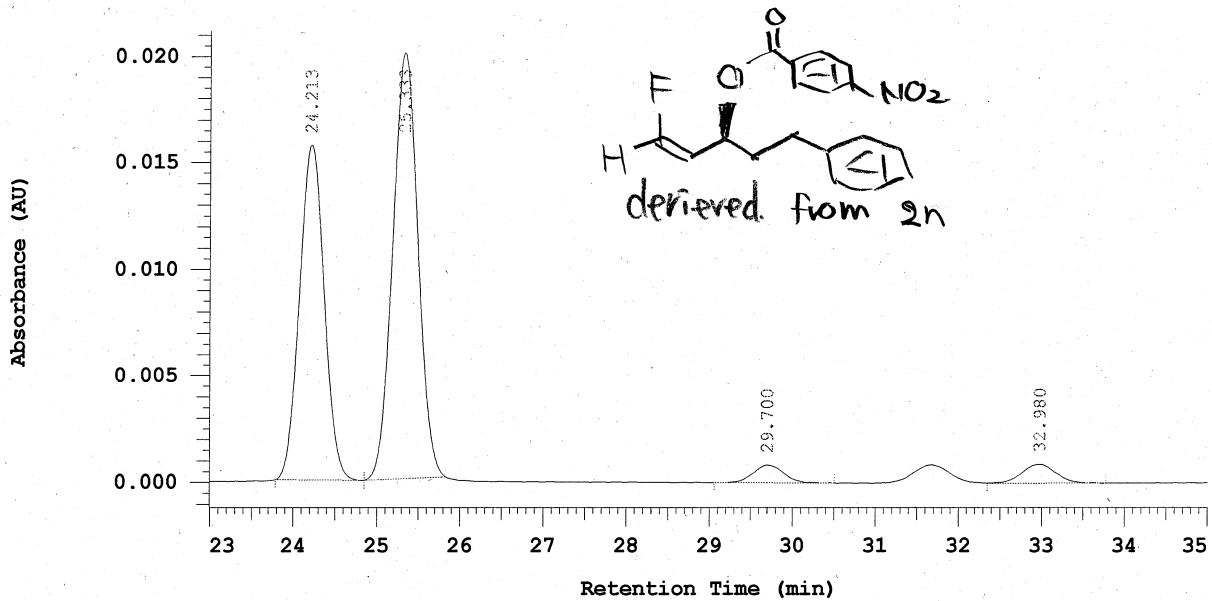
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area   | Conc 1  | BC |
|-----|--------|--------|---------|----|
| 1   | 24.213 | 159751 | 40.529  | BB |
| 2   | 25.333 | 211733 | 53.717  | BB |
| 3   | 29.700 | 10364  | 2.629   | MC |
| 4   | 32.980 | 12316  | 3.125   | MC |
|     |        |        | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/26  
13:25

Reported Date and Time: 2019/03/26  
15:26:04

Processed Date and Time: 2019/03/26  
15:25

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0271\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0271

Application (data): AKY

Vial Number: 113

Sample Name: AKY554-rac-acyl-IBN-3%

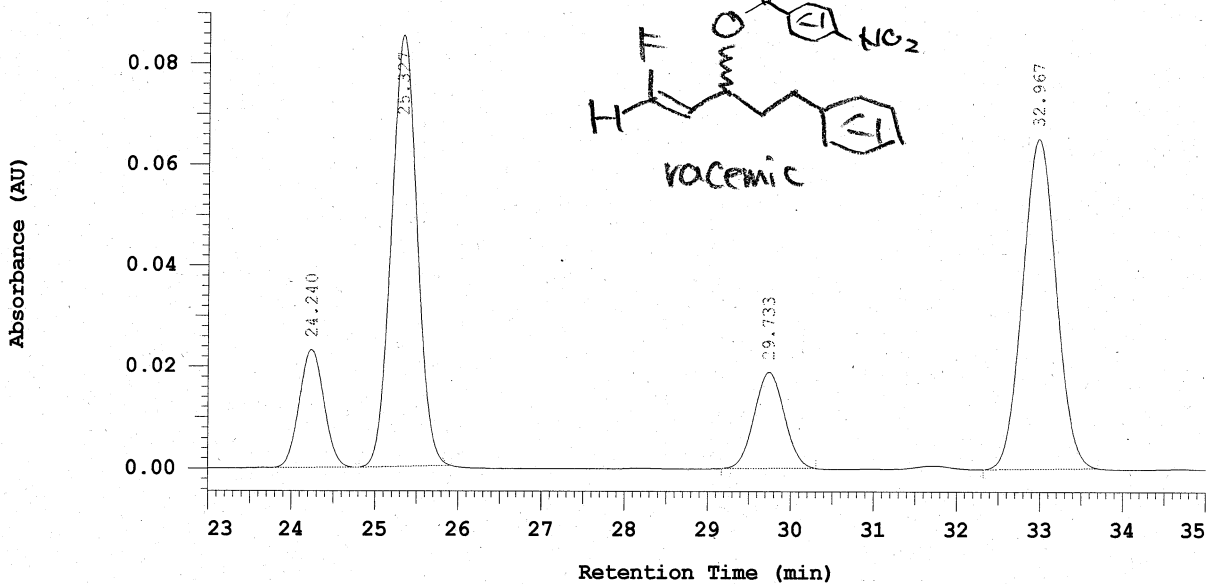
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 24.240 | 234441  | 10.168  | BB |
| 2   | 25.327 | 924308  | 40.087  | BB |
| 3   | 29.733 | 233700  | 10.136  | BB |
| 4   | 32.967 | 913293  | 39.609  | BB |
|     |        | 2305742 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/05  
13:29

Reported Date and Time: 2019/03/05  
16:04:27

Processed Date and Time: 2019/03/05  
16:04

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0234\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0234

Application (data): AKY

Vial Number: 102

Sample Name: AKY819-ph-chiral-Ib-5%

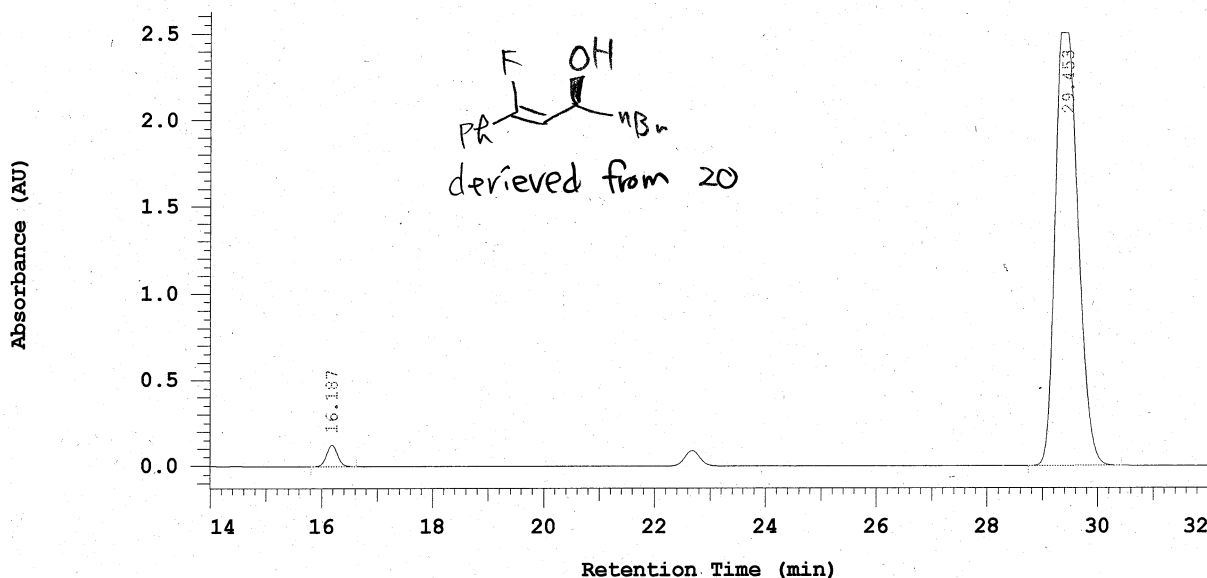
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 16.187 | 853861   | 2.294   | BB |
| 2   | 29.453 | 36363059 | 97.706  | BB |
|     |        | 37216920 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/05  
14:36

Reported Date and Time: 2019/03/05  
16:04:08

Processed Date and Time: 2019/03/05  
16:04

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0235\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0235

Application (data): AKY

Vial Number: 101

Sample Name: AKY659-ph-rac-Ib-5%

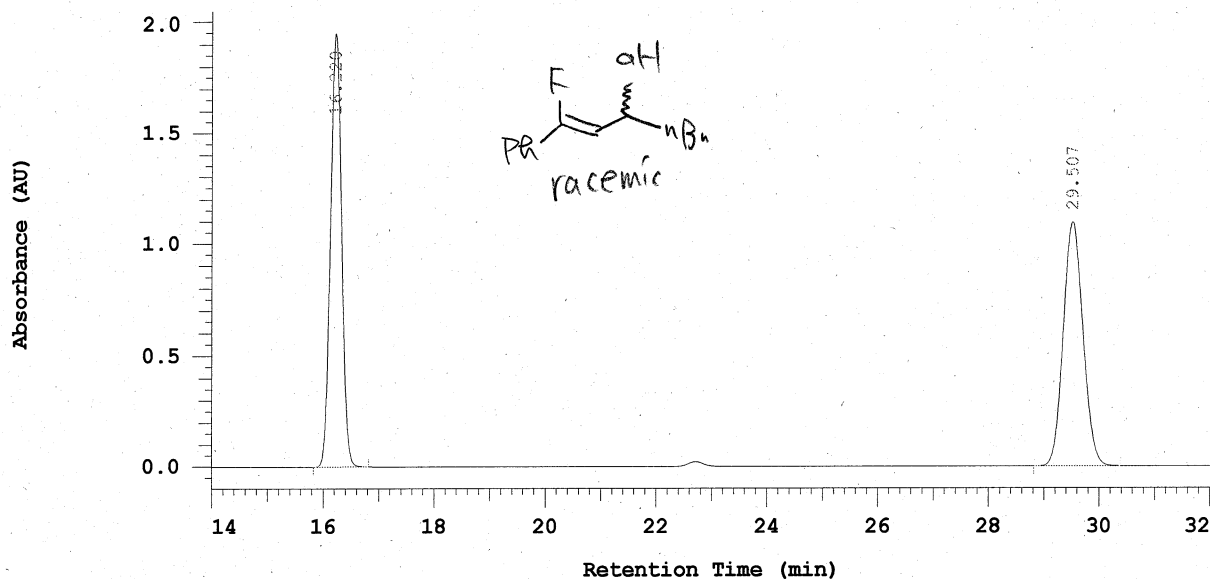
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 16.220 | 13501962 | 49.585  | BB |
| 2   | 29.507 | 13727800 | 50.415  | BB |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/22  
23:33

Reported Date and Time: 2019/02/23  
11:06:48

Processed Date and Time: 2019/02/23  
11:06

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0202\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1

Series: 0202

Application(data): AKY

Vial Number: 9

Sample Name: AKY735,6-Homolchi-IC-2%

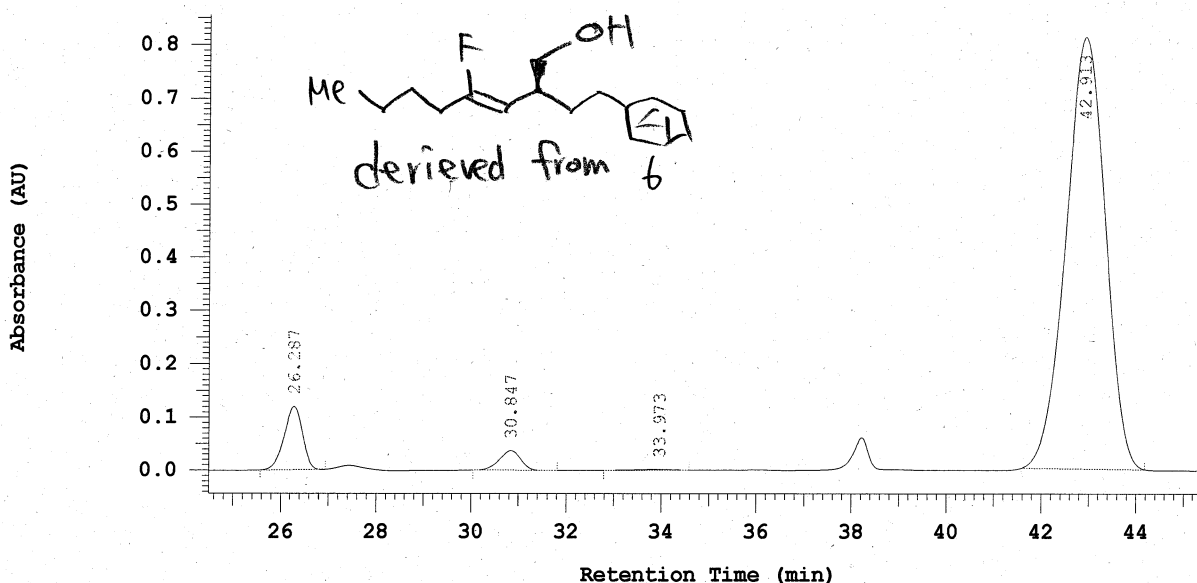
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column3(IC-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 26.287 | 1612403  | 6.270   | BB |
| 2   | 30.847 | 579796   | 2.255   | MC |
| 3   | 33.973 | 35032    | 0.136   | MC |
| 4   | 42.913 | 23488793 | 91.339  | BB |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/22  
22:26

Reported Date and Time: 2019/02/23  
11:03:31

Processed Date and Time: 2019/02/23  
11:03

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0201\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1

Series: 0201

Application(data): AKY

Vial Number: 8

Sample Name: AKY734-Homolrac-IC-2%

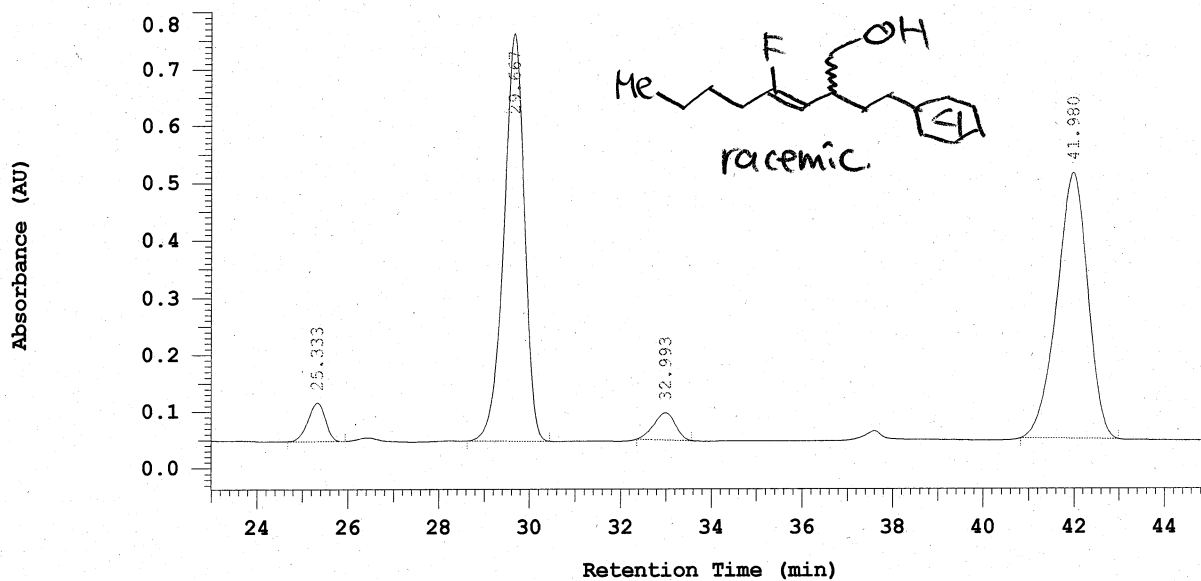
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column3(IC-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1   | BC      |
|-----|--------|----------|----------|---------|
| 1   | 25.333 | 891426   | 3.695    | BB      |
| 2   | 29.667 | 11327996 | 46.959   | BB      |
| 3   | 32.993 | 785381   | 3.256    | BB      |
| 4   | 41.980 | 11118542 | 46.090   | BB      |
|     |        |          | 24123345 | 100.000 |

Peak rejection level: 0



### Chromaster System Manager Report

Analyzed Date and Time: 2018/12/19 22:51

Reported Date and Time: 2018/12/20 09:17:42

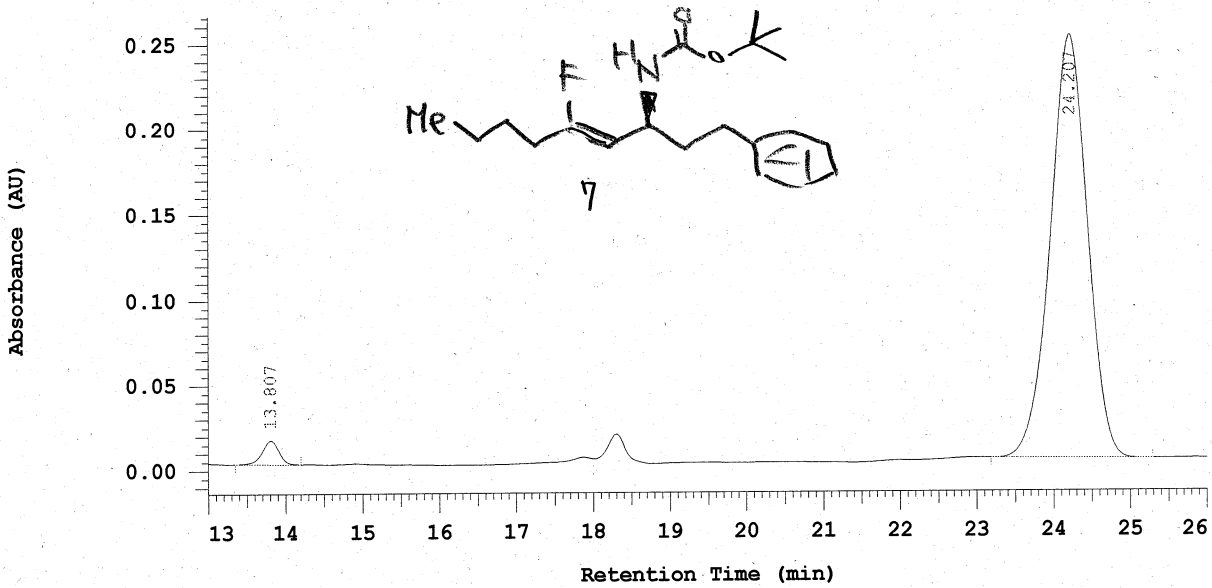
Processed Date and Time: 2018/12/20 09:17

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0133\  
Processing Method: column3(IC-3)

System (acquisition): Sys 1  
Application(data): AKY  
Sample Name: AKY763-chiral-Ic-4%  
Injection from this vial: 1 of 1  
Sample Description:

Series: 0133  
Vial Number: 31  
Vial Type: UNK  
Volume: 10.0 ul

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column3(IC-3)  
Method Developer:  
Pump 1: 5110  
Pump 1 Solvent A: hexane  
Pump 1 Solvent C:  
Method Description:

Pump 1 Solvent B: 2-propanol  
Pump 1 Solvent D:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA  
Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 13.807 | 106630  | 2.482   | MC |
| 2   | 24.207 | 4188887 | 97.518  | MC |
|     |        | 4295517 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/12/19 00:05

Reported Date and Time: 2018/12/20 09:19:43

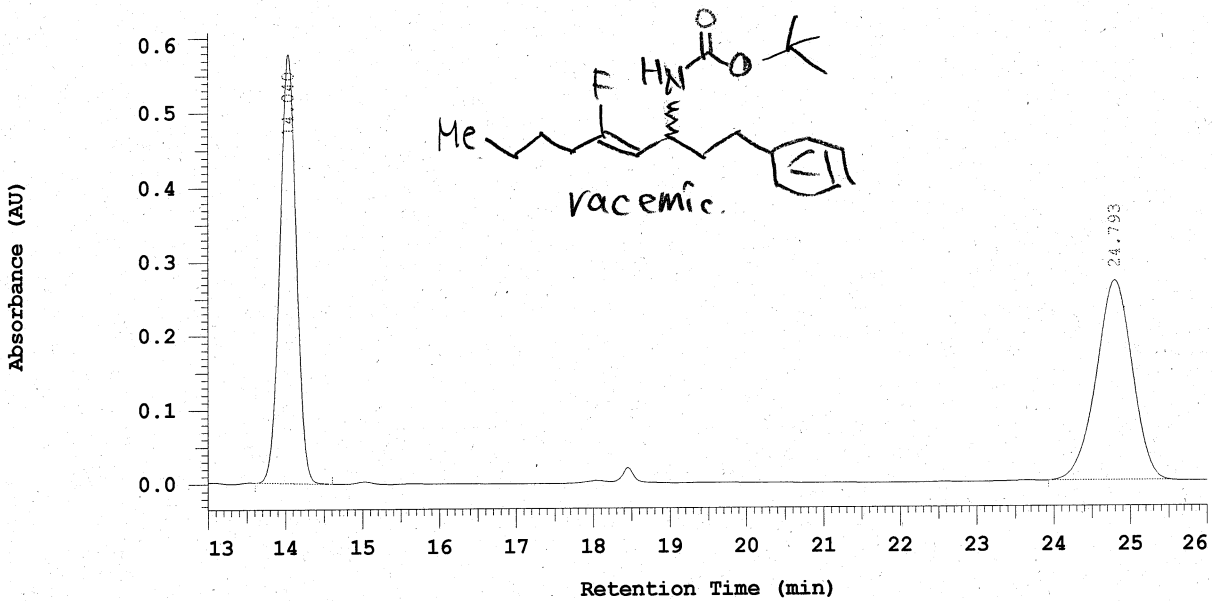
Processed Date and Time: 2018/12/20 09:19

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0129\  
 Processing Method: column3(IC-3)

System (acquisition): Sys 1  
 Application(data): AKY  
 Sample Name: AKY762-rac-Ic-4%  
 Injection from this vial: 1 of 1  
 Sample Description:

Series: 0129  
 Vial Number: 31  
 Vial Type: UNK  
 Volume: 10.0 ul

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column3(IC-3)  
 Method Developer:  
 Pump 1: 5110  
 Pump 1 Solvent A: hexane  
 Pump 1 Solvent C:  
 Method Description:

Pump 1 Solvent B: 2-propanol  
 Pump 1 Solvent D:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA  
 Calculation Method: AREA%

| No. | RT     | Area    | Conc 1  | BC |
|-----|--------|---------|---------|----|
| 1   | 14.040 | 4377410 | 49.881  | BB |
| 2   | 24.793 | 4398346 | 50.119  | BB |
|     |        | 8775756 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/13 17:15

Reported Date and Time: 2019/02/13 19:25:26

Processed Date and Time: 2019/02/13 19:25

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0175\  
Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0175

Application(data): AKY

Vial Number: 49

Sample Name: AKY787-PhPhCHO-IF3-1%rac

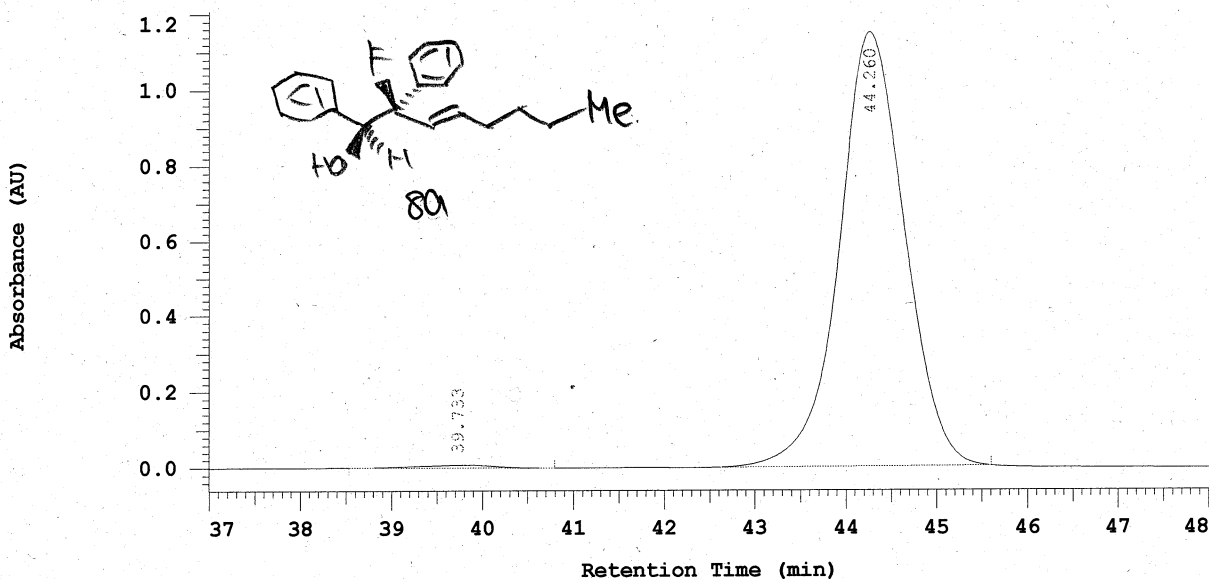
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 39.733 | 224936   | 0.777   | MC |
| 2   | 44.260 | 28711557 | 99.223  | MC |
|     |        | 28936493 | 100.000 |    |

Peak rejection level: 0

**Chromaster System Manager Report**Analyzed Date and Time: 2019/02/13  
18:26Reported Date and Time: 2019/02/13  
19:22:45Processed Date and Time: 2019/02/13  
19:22

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0176\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0176

Application(data): AKY

Vial Number: 48

Sample Name: AKY786-PhPhCHO-IF3-1%rac

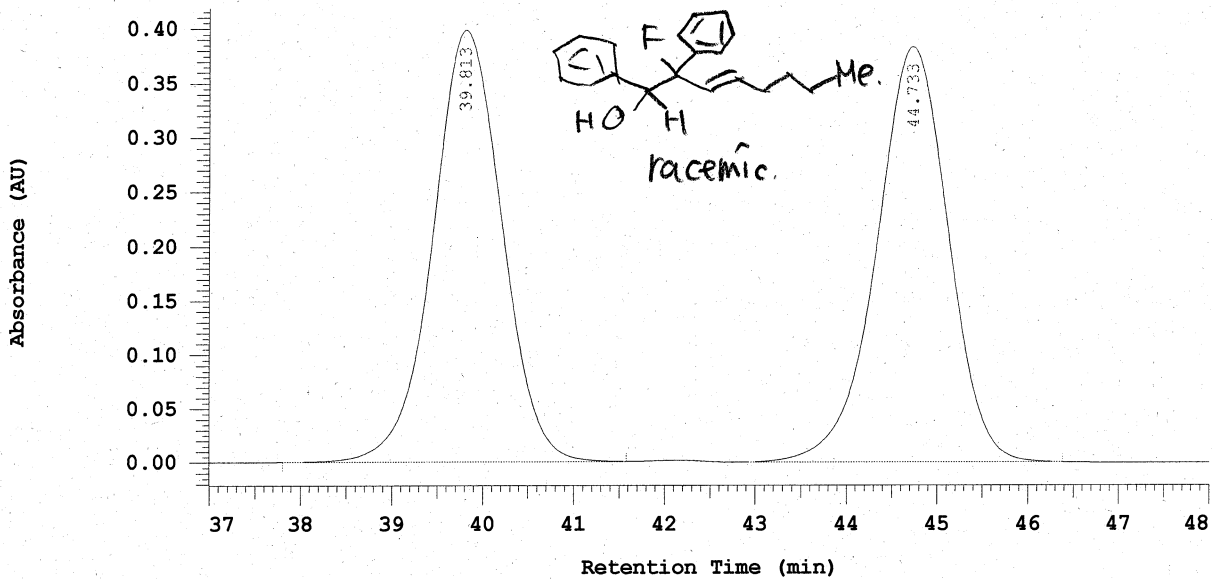
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 39.813 | 10547536 | 50.021  | MC |
| 2   | 44.733 | 10538625 | 49.979  | MC |
|     |        | 21086161 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/25  
14:34

Reported Date and Time: 2019/02/26  
11:40:31

Processed Date and Time: 2019/02/26  
11:40

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0211\  
Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0211

Application(data): AKY

Vial Number: 94

Sample Name: AKY819-Me-chiral-IF-1%

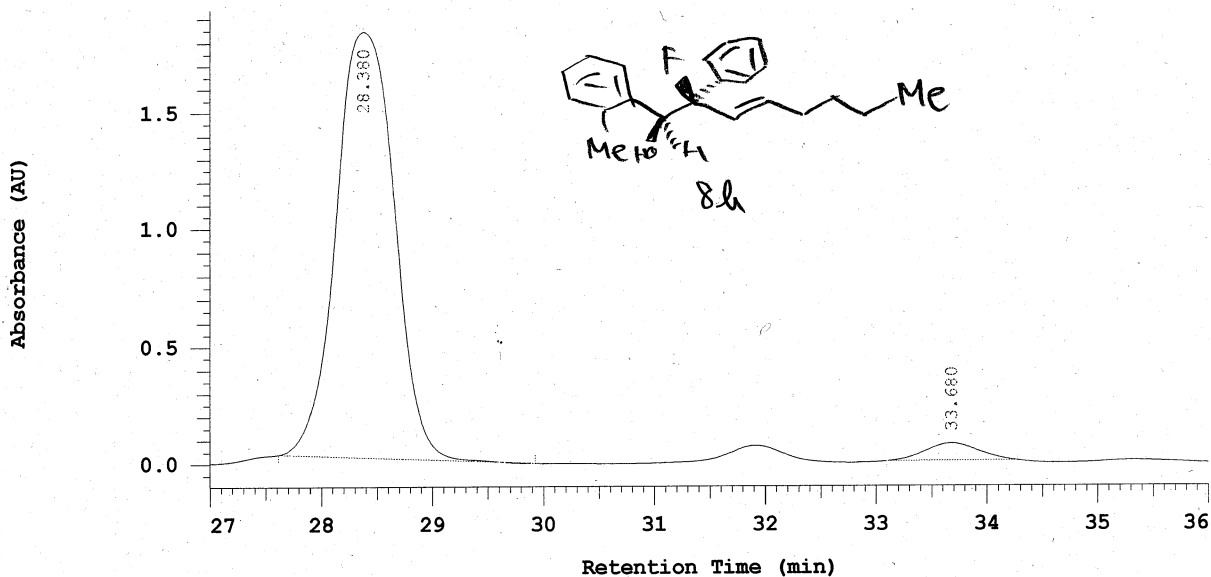
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 28.380 | 33182182 | 96.349  | MC |
| 2   | 33.680 | 1257470  | 3.651   | MC |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/25 19:13

Reported Date and Time: 2019/02/26 11:39:05

Processed Date and Time: 2019/02/26 11:39

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0215\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0215

Application(data): AKY

Vial Number: 93

Sample Name: AKY803-Me-rac-IF-1%

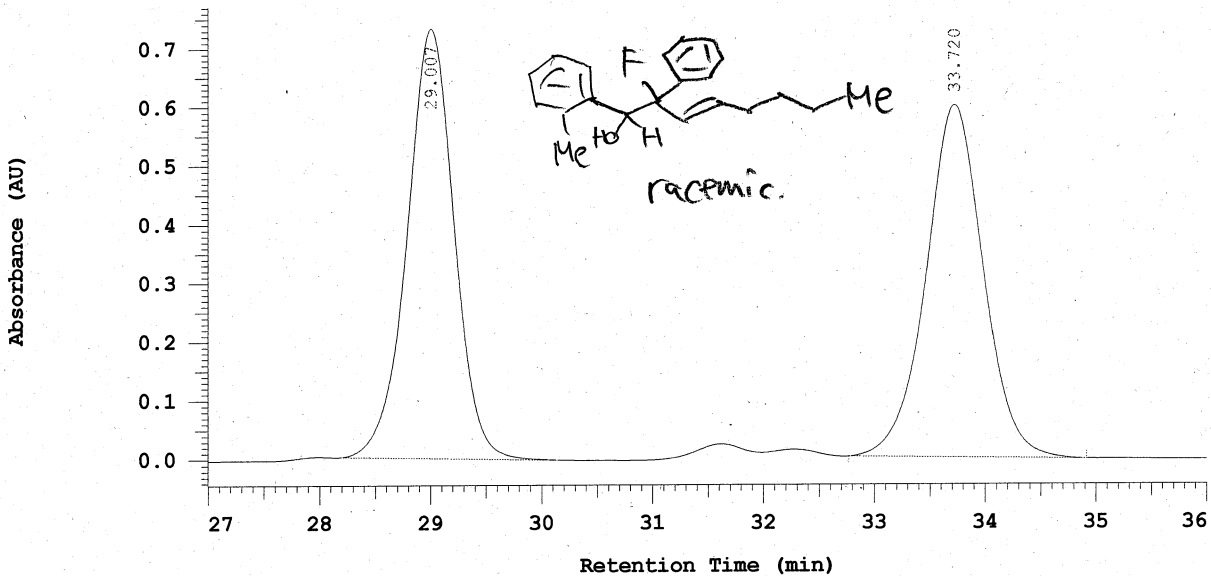
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 29.007 | 10776113 | 50.031  | MC |
| 2   | 33.720 | 10762549 | 49.969  | MC |
|     |        | 21538662 | 100.000 |    |

Peak rejection level: 0

## Chromaster System Manager Report

Analyzed Date and Time: 2019/02/13 14:19      Reported Date and Time: 2019/02/13 15:52:16

Processed Date and Time: 2019/02/13 15:51

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0172\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0172

Application(data): AKY

Vial Number: 46

Sample Name: AKY808-Br-IF3-1%chiral

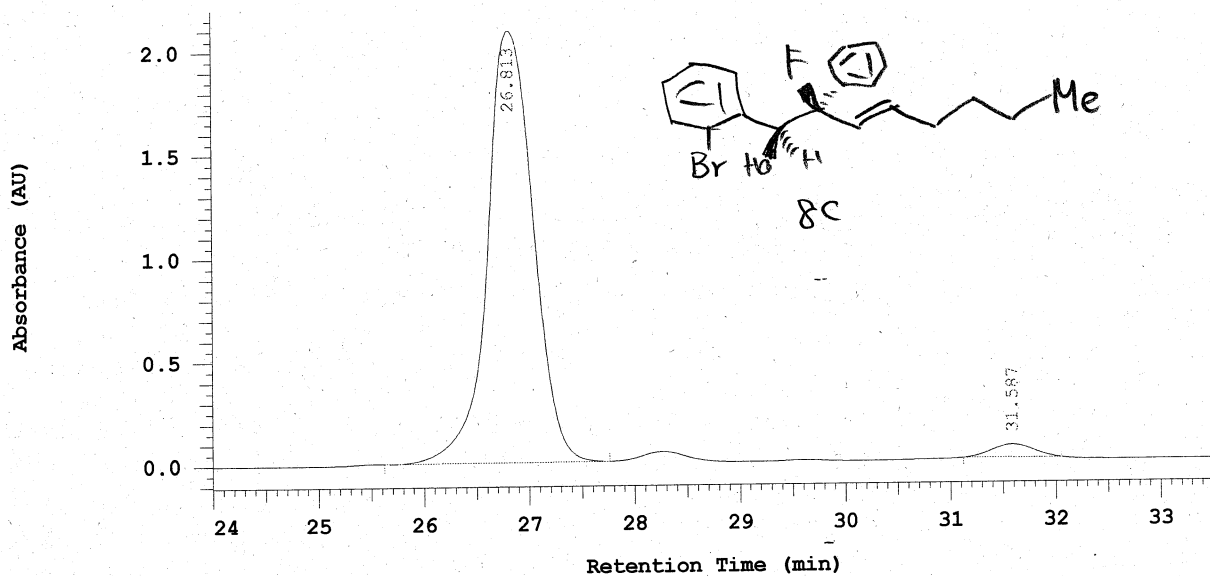
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 26.813 | 31735019 | 97.360  | MC |
| 2   | 31.587 | 860610   | 2.640   | BB |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/13  
15:18

Reported Date and Time: 2019/02/13  
15:56:20

Processed Date and Time: 2019/02/13  
15:56

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0173\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0173

Application(data): AKY

Vial Number: 45

Sample Name: AKY802-Br-IF3-1%rac

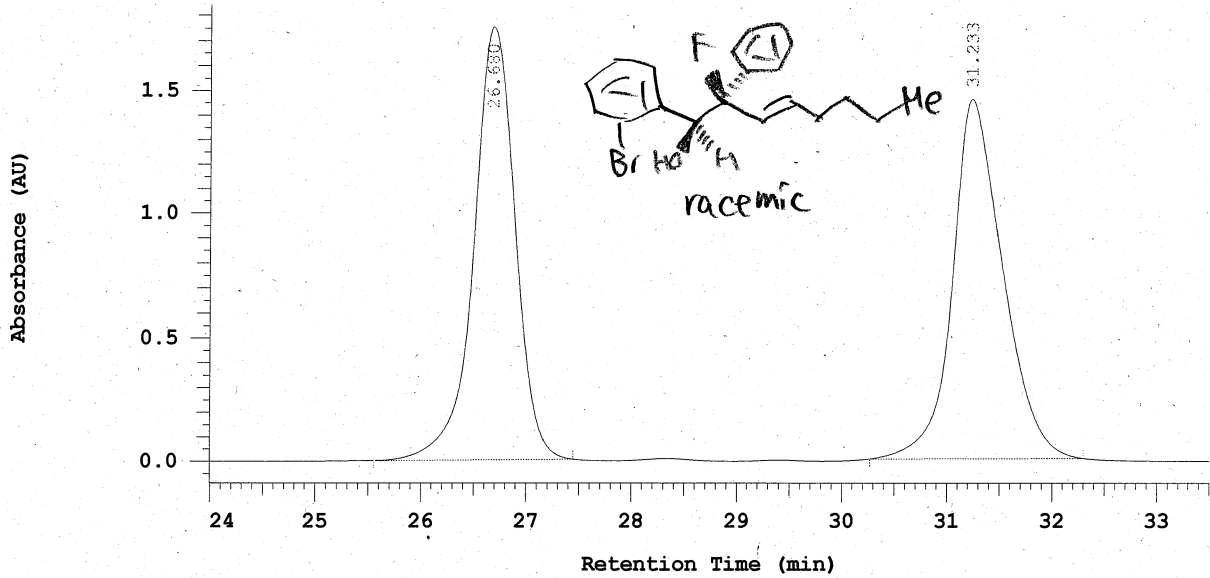
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 26.680 | 23993254 | 49.706  | BB |
| 2   | 31.233 | 24276897 | 50.294  | BB |
|     |        | 48270151 | 100.000 |    |

Peak rejection level: 0



### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/17  
20:12

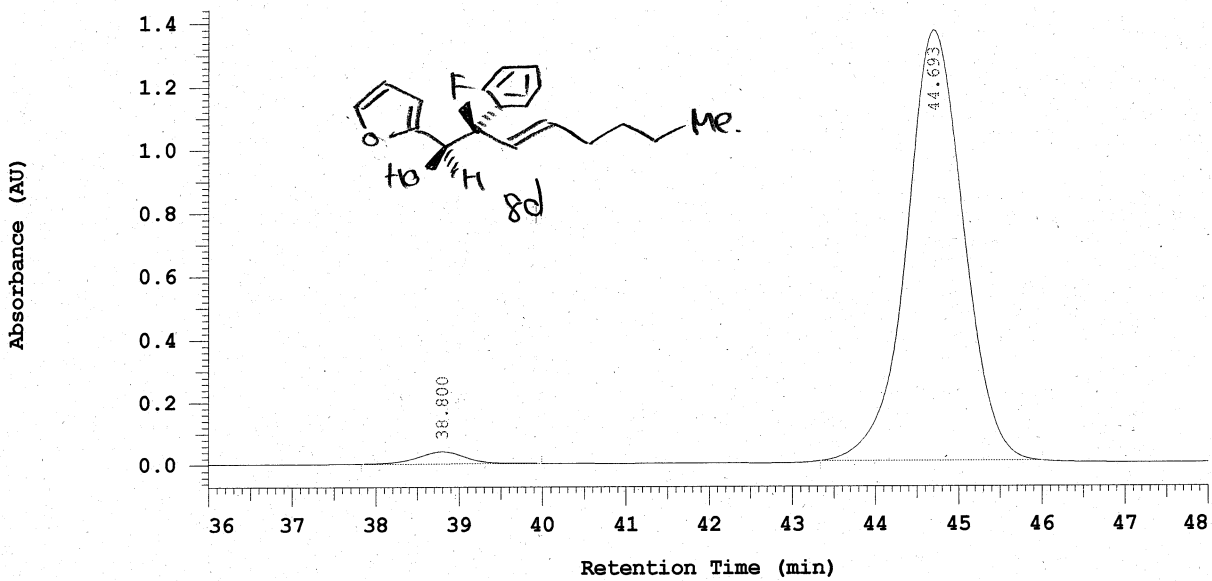
Reported Date and Time: 2019/02/18  
09:08:22

Processed Date and Time: 2019/02/18  
09:08

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0198\  
Processing Method: column6(IF-3)

System (acquisition): Sys 1                      Series: 0198  
Application(data): AKY                              Vial Number: 62  
Sample Name: AKY820-Ph-furan-IF-2%-chir      Vial Type: UNK  
Injection from this vial: 1 of 1                  Volume: 10.0 ul  
Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 38.800 | 763662   | 2.289   | MC |
| 2   | 44.693 | 32596794 | 97.711  | MC |
|     |        | 33360456 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/17  
19:00

Reported Date and Time: 2019/02/18  
09:10:46

Processed Date and Time: 2019/02/18  
09:10

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0197\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0197

Application(data): AKY

Vial Number: 61

Sample Name: AKY805-Ph-furan-IF-2%-rac

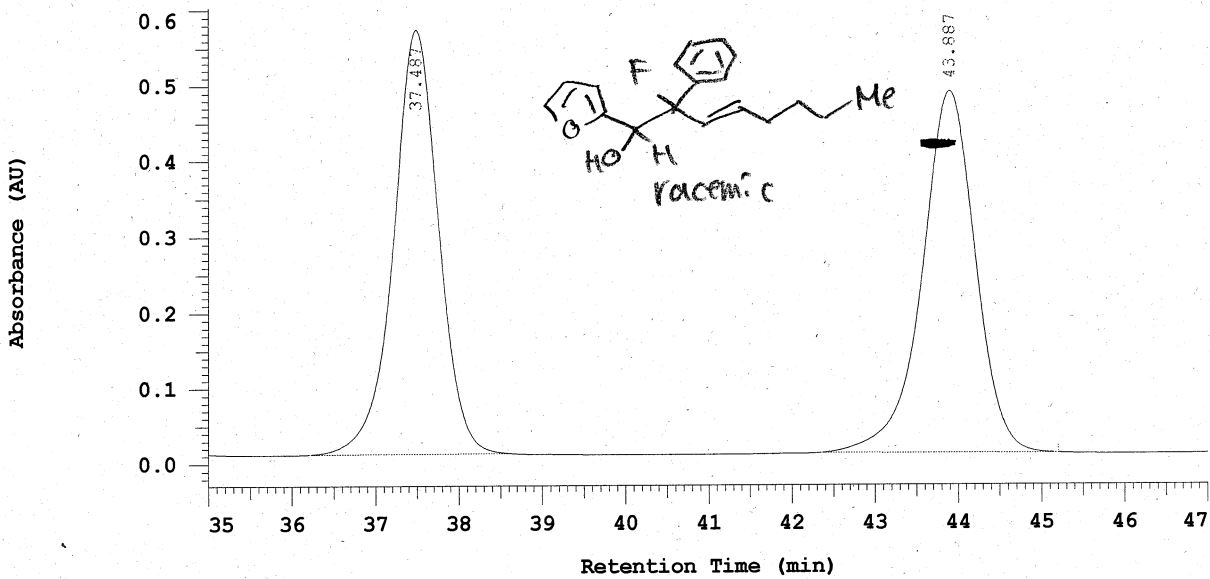
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 37.487 | 10561309 | 49.729  | MC |
| 2   | 43.887 | 10676580 | 50.271  | MC |
|     |        | 21237889 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/23  
11:02

Reported Date and Time: 2019/02/25  
10:38:43

Processed Date and Time: 2019/02/25  
10:38

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0206\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0206

Application(data): AKY

Vial Number: 92

Sample Name: AKY798-pyridine-IF-10%-  
chiral

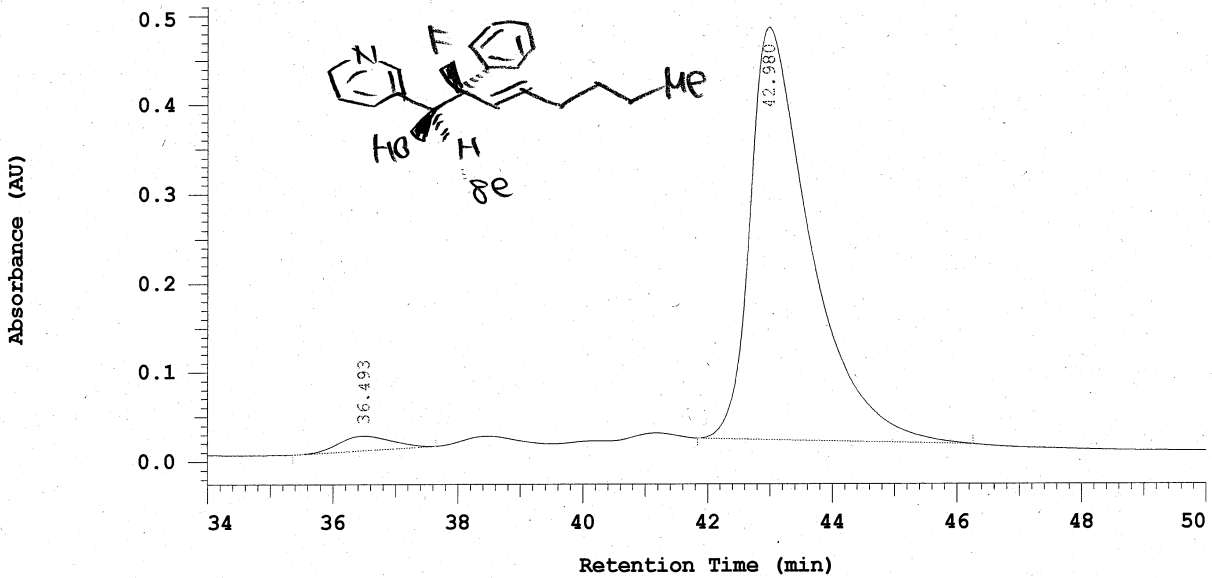
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 36.493 | 476384   | 2.980   | MC |
| 2   | 42.980 | 15510969 | 97.020  | MC |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/23  
16:20

Reported Date and Time: 2019/02/25  
10:43:03

Processed Date and Time: 2019/02/25  
10:42

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0209\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0209

Application(data): AKY

Vial Number: 91

Sample Name: AKY795-pyridine-IF-10%-rac

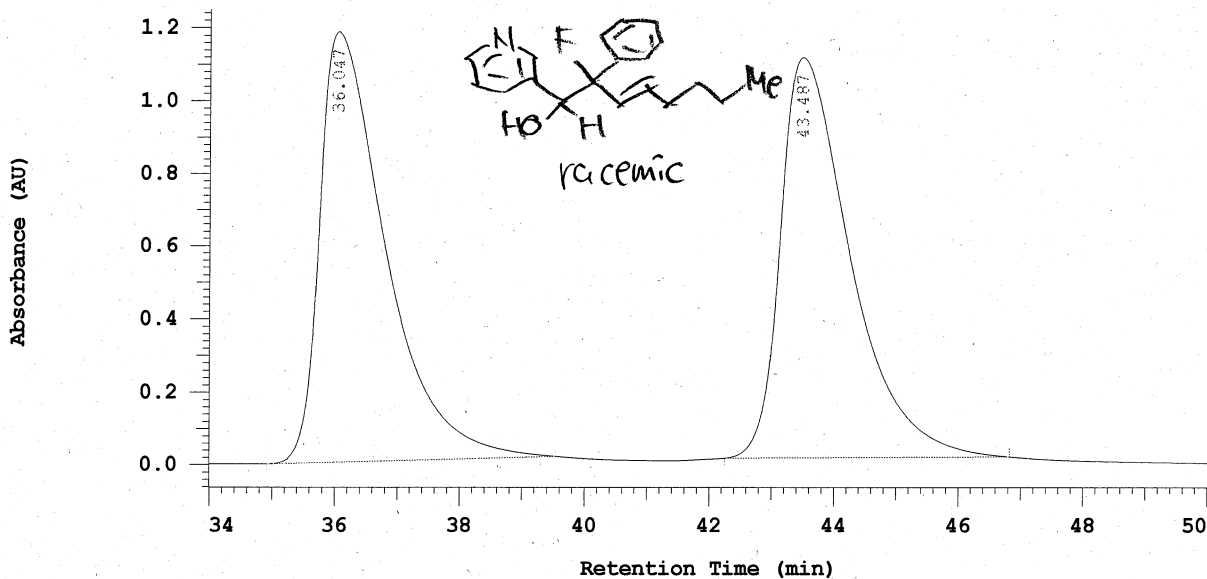
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 36.047 | 43278602 | 50.269  | BB |
| 2   | 43.487 | 42815201 | 49.731  | BB |
|     |        | 86093803 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/16  
21:34

Reported Date and Time: 2019/02/17  
14:52:35

Processed Date and Time: 2019/02/17  
14:49

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0196\  
Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0196

Application(data): AKY

Vial Number: 60

Sample Name: AKY824-Ph-conjugate-IF-  
2%-chir

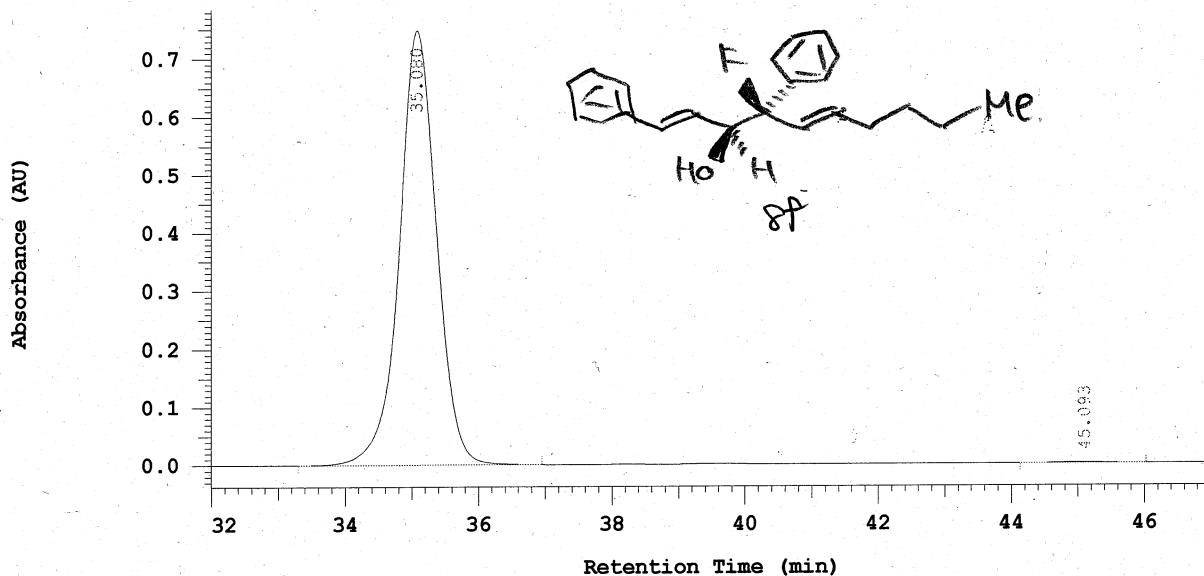
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 35.080 | 14594816 | 99.774  | MC |
| 2   | 45.093 | 33036    | 0.226   | MC |
|     |        | 14627852 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/02/16 20:23

Reported Date and Time: 2019/02/17 14:46:48

Processed Date and Time: 2019/02/17 14:46

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0195\

Processing Method: column6(IF-3)

System (acquisition): Sys 1

Series: 0195

Application(data): AKY

Vial Number: 59

Sample Name: AKY799-Ph-conjugat-IF-2%-rac

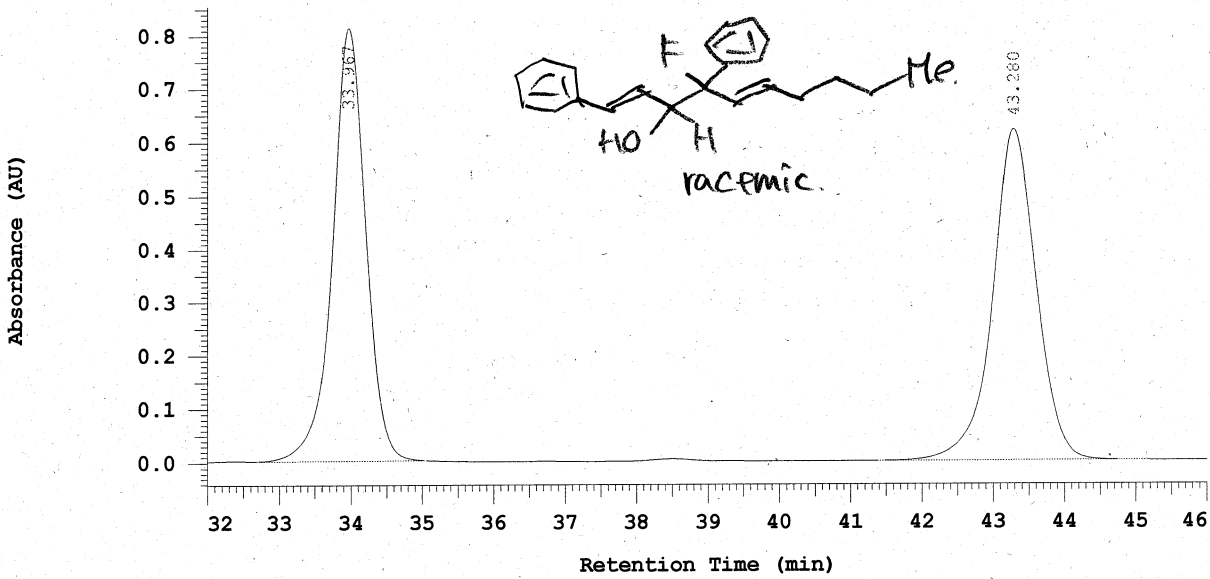
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 250 nm



Processing Method: column6(IF-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 250 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 33.967 | 13039773 | 49.816  | MC |
| 2   | 43.280 | 13135973 | 50.184  | MC |
|     |        |          | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/11/02  
14:34

Reported Date and Time: 2018/11/02  
15:38:46

Processed Date and Time: 2018/11/02  
15:38

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0075\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0075

Application(data): AKY

Vial Number: 17

Sample Name: AKY713-chiraCy-allylb-  
IBN-1.5%

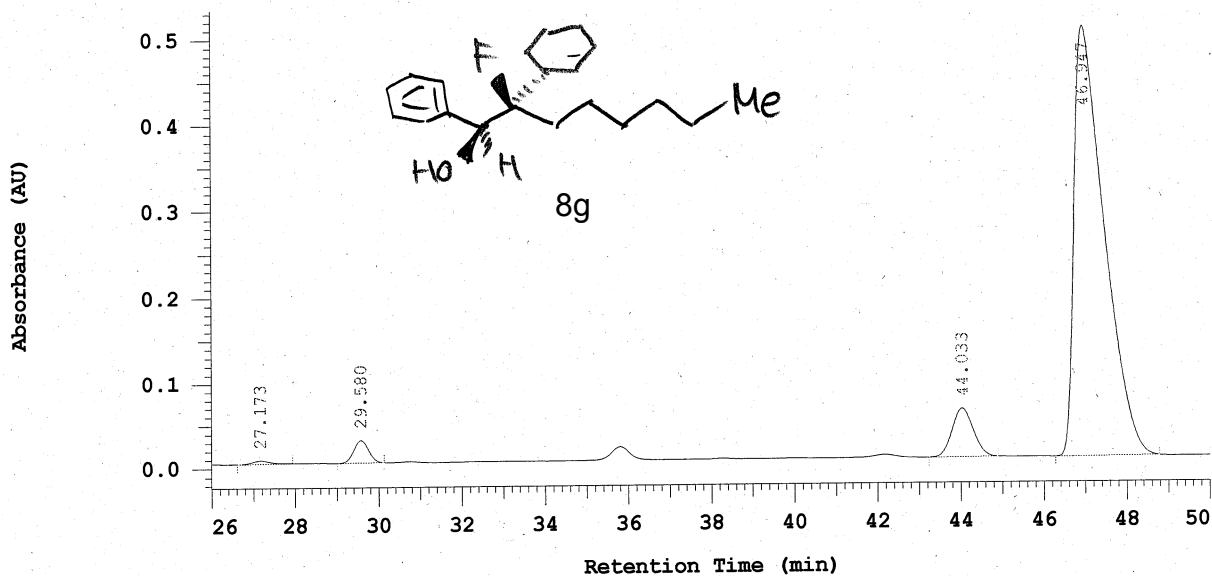
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 206 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 206 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1   | BC      |
|-----|--------|----------|----------|---------|
| 1   | 27.173 | 55280    | 0.393    | MC      |
| 2   | 29.580 | 330545   | 2.350    | MC      |
| 3   | 44.033 | 1026848  | 7.301    | BB      |
| 4   | 46.947 | 12651174 | 89.955   | BB      |
|     |        |          | 14063847 | 100.000 |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2018/11/02  
13:58

Reported Date and Time: 2019/02/22  
15:29:13

Processed Date and Time: 2019/02/22  
15:29

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0073\_Cy-PhCHO-rac\  
Processing Method: column2(IBN-3)

System (acquisition): Sys 1

Series: 0073\_Cy-PhCHO-rac

Application(data): AKY

Vial Number: 16

Sample Name: AKY711-racCy-allylb-IBN-  
1.5%

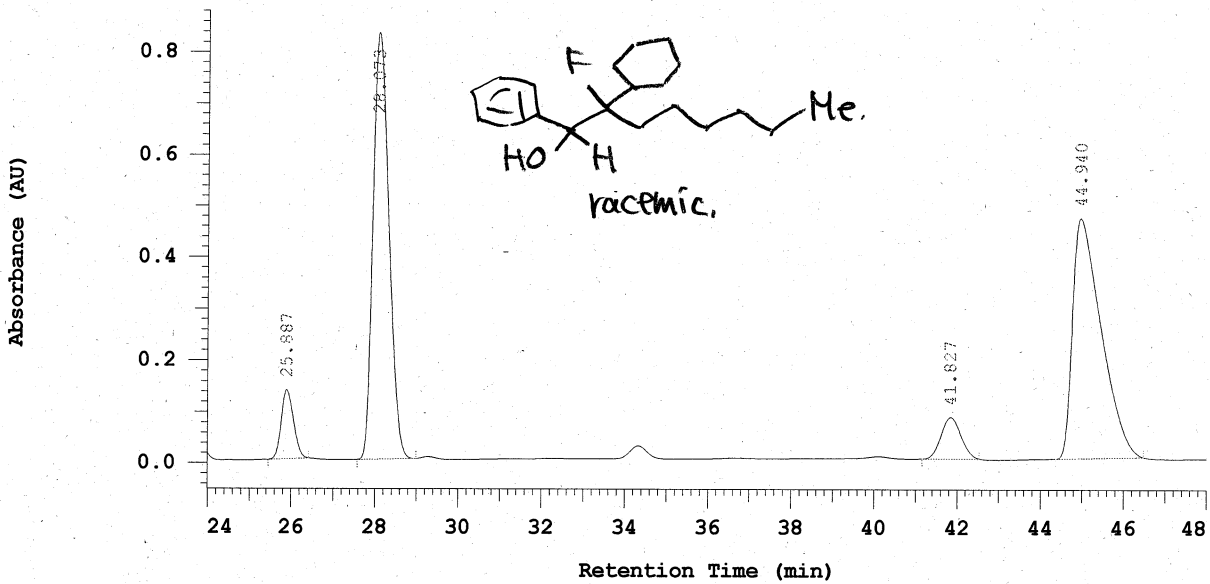
Vial Type: UNK

Volume: 10.0 ul

Injection from this vial: 1 of 1

Sample Description:

Chrom Type: Fixed WL Chromatogram, 202 nm



Processing Method: column2(IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 202 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 25.887 | 1402199  | 5.628   | BB |
| 2   | 28.073 | 10945221 | 43.931  | BB |
| 3   | 41.827 | 1369634  | 5.497   | BB |
| 4   | 44.940 | 11197789 | 44.944  | BB |
|     |        | 24914843 | 100.000 |    |

Peak rejection level: 0



**Chromaster System Manager Report**Analyzed Date and Time: 2019/03/02  
00:30Reported Date and Time: 2019/03/25  
20:34:41Processed Date and Time: 2019/03/25  
20:34

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0228\

Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0228

Application (data): AKY

Vial Number: 100

Sample Name: AKY832-NHAc-chiral-IF-15%

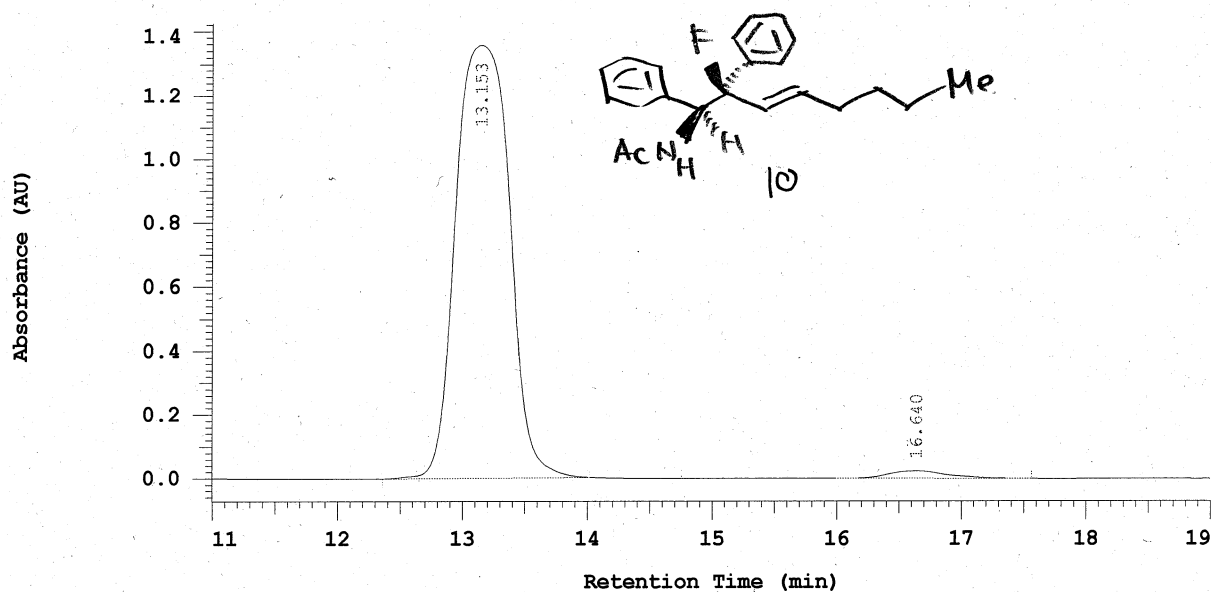
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 13.153 | 20332035 | 98.133  | MC |
| 2   | 16.640 | 386792   | 1.867   | MC |
|     |        | 20718827 | 100.000 |    |

Peak rejection level: 0

### Chromaster System Manager Report

Analyzed Date and Time: 2019/03/01  
22:53

Reported Date and Time: 2019/03/25  
20:38:15

Processed Date and Time: 2019/03/25  
20:38

Data Path: C:\WIN32APP\CHROMASTER\AKY\DATA\0227\  
Processing Method: column2 (IBN-3)

System (acquisition): Sys 1

Series: 0227

Application (data): AKY

Vial Number: 99

Sample Name: AKY830-NHAc-rac-IF-15%

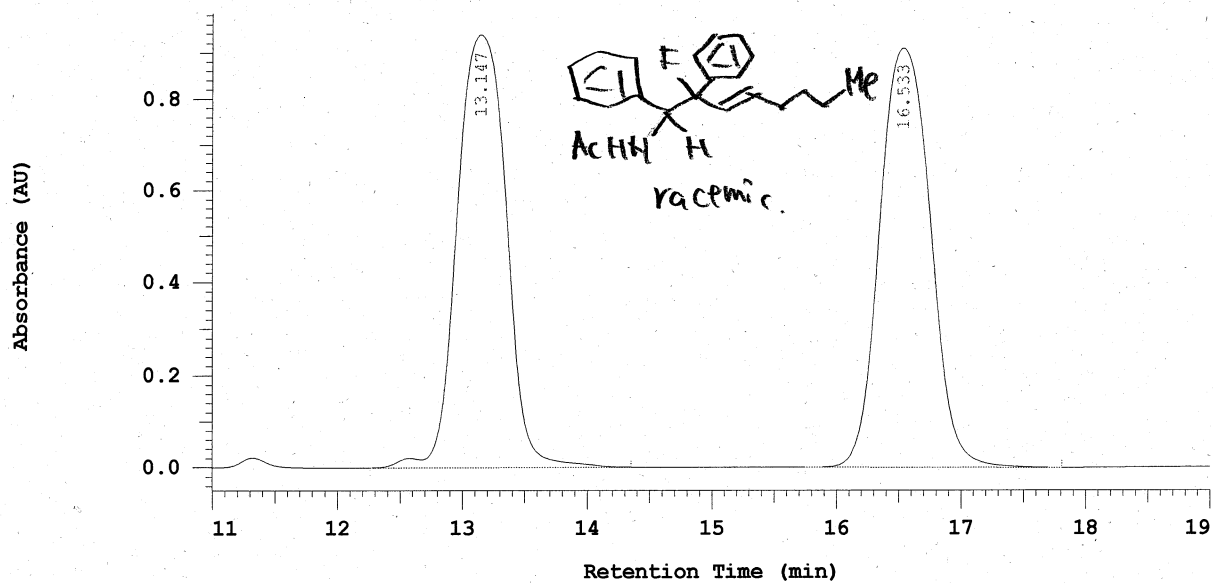
Vial Type: UNK

Injection from this vial: 1 of 1

Volume: 10.0 ul

Sample Description:

Chrom Type: Fixed WL Chromatogram, 203 nm



Processing Method: column2 (IBN-3)

Method Developer:

Pump 1: 5110

Pump 1 Solvent A: hexane

Pump 1 Solvent B: 2-propanol

Pump 1 Solvent C:

Pump 1 Solvent D:

Method Description:

Chrom Type: Fixed WL Chromatogram, 203 nm

Peak Quantitation: AREA

Calculation Method: AREA%

| No. | RT     | Area     | Conc 1  | BC |
|-----|--------|----------|---------|----|
| 1   | 13.147 | 12858525 | 50.002  | MC |
| 2   | 16.533 | 12857301 | 49.998  | BB |
|     |        | 25715826 | 100.000 |    |

Peak rejection level: 0