Electronic Supplementary Material (ESI) for Chemical Communications

Formation of fluoroalkyl chiral products through Enantioselective Allylic Alkylation catalyzed by NHC ligand.

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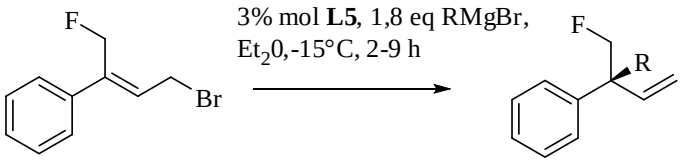
SUPPORTING INFORMATION AVAILABLE

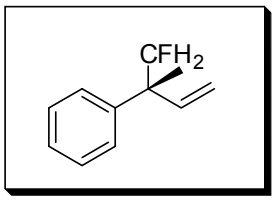
General Remarks.

All reactions were not carried out under nitrogen or argon atmosphere in flame-dried glassware and with dry solvents. Solvents (THF, Et2O, Toluene, DCM, MTBE and CH3CN) were dried over alumina (activated at 350 °C under nitrogen atmosphere for 12 h). Yields refer to chromatographically and spectroscopically (1H NMR) homogeneous materials, unless otherwise stated. Reactions were monitored by GC-MS Hewlet Packard (EI mode) HP6890-5973 on an HP6890 or by TLC carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV lamp as visualizing agent and KMnO4 solution as developing agents. Flash chromatographies were performed using silica gel (particle size 32-63 μm, 60 Å). 1H (300 or 400 MHz) and 13C (75 or 100 MHz) NMR spectra were recorded on Bruker AMX-300 or 400 instrument in CDCl3 and calibrated using residual undeuterated solvent as an internal reference. Chemical shift (δ) are given in ppm relative to tetramethylsilane (0 ppm). Multiplicity is indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br s (broad singlet). Coupling constants *J* are reported in Hz. **^{19}F-(NMR) is proton decoupled unless stated**. Mass spectra (MS) and High resolution mass spectra (HRMS) were obtained by Electrospray Ionisation (ESI) or by electronic impact (EI, 70 eV). Optical rotations were measured at 20°C in a 10 cm cell in the stated solvent; [α]D values are given in 10-1 deg.cm2 g-1 (concentration c given as g/100 mL). Enantiomeric excesses were determined by chiral GC measurement on a HP6890 (H2 as vector gas) or HP6850 (He or H2 as vector gas).Temperature programs are described as follows: initial T (°C) – initial times (min) – temperature gradient (°C/min) – final T (°C); retention times (RT) are given in min.).

Catalysis adducts.



In a flame-dried Schlenk under N2 atmosphere, the allyl bromide derivative (0.4 mmol) and the ligand (3 mol %) are suspended in dry Et2O (adjusted to 2.5 ml depending of the Grignard reagent volume) and cooled to -15°C. A solution of Grignard reagent X M in Et2O (1.8 eq) is added dropwise. After complete conversion, the mixture is quenched by addition of NH4Clsat. (2 ml) and stirred at roomtemperature during 15 min. The aqueous layer is separated and extracted with Et2O (3 × 3 ml). The combined organic fractions are dried over Na2SO4, filtered and concentrated in vaccuo. The residue is purified by flash column chromatography to offer a mixture of SN2' and SN2 products. GC or SFC on a chiral stationary phase shows the enantiomeric excess of the SN2' product.



1a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.27 (m,5H) 6.11-6.03 (qd,1H, J=10,86 Hz and J=1,10 Hz) 5.28-5.25 (dd,1H, J=10,82 Hz and J=0,65 Hz) 5.21-5.16 (dd,1H,J=18,4 Hz and J=0,67 Hz) 4.66-4.59 (q,1H, J= 9 Hz) 4.54-4.47 (q,1H,J=9Hz) 1.50 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.5 (J=3,21 Hz) 142.5 (J=4,04 Hz) 128.5, 127.1, 126.9, 114.7, 114.7, 90.0, 88.3, 46.2 (J=17,69 Hz) 22.2 (J=4,72 Hz).

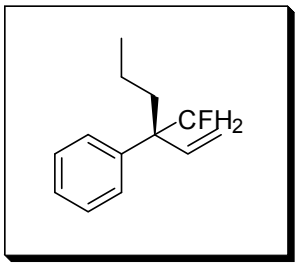
^{19}F NMR (376 MHz, $CDCl_3$) -57, 84.

Yield: 50 %.

85 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:35,8 min. t2:36,2 min.

[α] = -7.02 (*c* 1.33, $CHCl_3$)

HRMS (EI + mode) m:z expected:164,1001 observed:164,1005



1b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.26 (m,5H) 6.09, 6.09-6.02 (qd,1H, J=11,02 Hz and J=1,14 Hz) 5.35-5.32 (dd,1H, J=11,64 Hz and J=0,54 Hz) 5.19-5.14 (dd,1H, J=18,51 Hz and J=0,81 Hz) 4.77-4.66 (q,1H, J=8,99 Hz) 4.63-4.57(q,1H,J=8,99 Hz) 1.93-1.88 (m,2H) 1.37-1.20 (q,2H,J=7,51 Hz) 0.92-0.89 (t,3H,J =7,32 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.6 (J=3,21 Hz) 141.6 (J=4,58 Hz) 128.4, 127.6, 126.7, 115.2 (J=1,27 Hz) , 88.8, 87.0, 49.45(J=17,13 Hz) 37.9 (J=4,19 Hz) 17.5, 14.9.

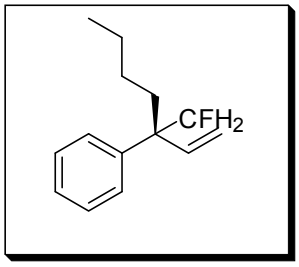
^{19}F NMR (376 MHz, $CDCl_3$) -61,32.

Yield: 55 %.

94 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:46,5 min. t2:47,8 min.

[α] = -6.9 (*c* 4, $CHCl_3$)

HRMS (EI + mode) m:z expected:192,1314 observed:192,1316



1c

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.26 (m,5H) 6.09-6.02 (qd,1H, J=11,06 Hz and J=1,15 Hz) 5.35-5.32 (dd,1H, J=11,1 Hz and J=1 Hz) 5.19-5.14 (dd,1H, J=17,85 Hz and J=0,64 Hz) 4.77-4.66 (q,1H, J=9 Hz) 4.63-4.57(q,1H,J= 9 Hz) 1.93-1.88 (m,2H) 1.37-1.20 (m,4H) 0.92-0.89 (t,3H,J =7,32 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.7 (J=3,18 Hz) 141.6 (J=3,96 Hz) 128.4, 127.6, 127.6, 126.7, 115.2, 115.2, 88.8, 87.1, 49.3 (J=17,12 Hz) 35.3 (J=4,29 Hz) 26.3, 23.6, 14.2.

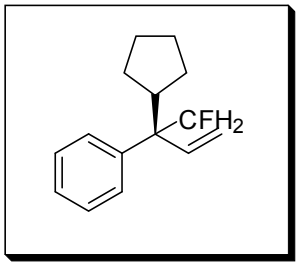
^{19}F NMR (376 MHz, $CDCl_3$) -61,26.

Yield: 54 %.

93 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:56,6 min. t2:57,5 min.

[α] = -7.02 (*c* 4, $CHCl_3$)

HRMS (EI + mode) m:z expected:206,1471 observed:206,1469



1d

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.30, 6.13-6.05(qd,1H, J=11,35 Hz and J=1,58 Hz) 5.52-5.49 (11,36 Hz) 5.30-5.25 (d,1H,J=17,97 Hz) 4.78-4.66 (d,2H,J=47,63 Hz) 2.74-2.67 (quintuplet,1H,J=8,70 Hz) 1,90- 1.29 (m,8H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9 138.4 (J=3,71 Hz) 128.1, 128., 126.5, 116.8 (J=1,41 Hz) 89.0, 87.2, 51.6 (J=16,47 Hz) 43.5 (J=3,73 Hz) 27.8, 27.8, 25.9, 25.4.

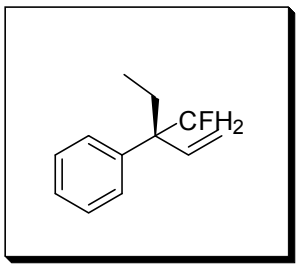
^{19}F NMR (376 MHz, $CDCl_3$) -61,30.

Yield: 60 %.

90 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:49,5 min. t2:51,4 min.

[α] = -27.8 (*c* 1.85, $CHCl_3$)

HRMS (EI + mode) m:z expected:218,1471 observed:218,1470



1e

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.25 (m,5H) 6.07-6.00 (qd,1H, J=11,1 Hz and J=1,22 Hz) 5.35-5.32 dd,1H, J=11,07 Hz and J=1 Hz) 5.19-5.14 (dd,1H,J=18,4 Hz and J=0,64 Hz) 4.77-4.67 (q,1H, J=8,99 Hz) 4.65-4.57 (q,1H, J=8,99 Hz) 2.00-1.93(m,2H) 0.88-0.84 (t,3H, J=7,45 Hz) .

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.4 (J=3,22 Hz) 141.3 (J=4,08 Hz) 128.4, 127.7, 126.7, 115.4 (J=1,61 Hz) 88.5, 49.5 (J=4,54 Hz) 28.0, 27.9, 8.7.

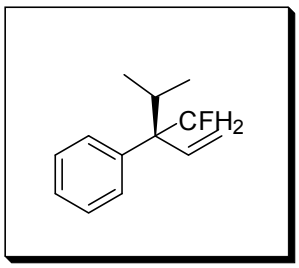
^{19}F NMR (376 MHz, $CDCl_3$) -62,08.

Yield: 48 %.

94 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:42 min. t2:42,8 min.

[α] = -11.8 (*c* 1.94, $CHCl_3$)

HRMS (EI + mode) m:z expected:178,1158 observed:178,1163



1f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.25 (m,5H) 6.10-6.02 (qd,1H,J= 11,41Hz and J=2,09 Hz) 5.45-5.42 (dd,1H, J=11,31Hz and J= 0,99 Hz) 5.19-5.14 (dd,1H, J=18,1 Hz and J=0,85 Hz) 4.85-4.76 (q,1H, J= 9,15 Hz) 4.73-4.64 (q,1H, J=9,19 Hz) 2.48-2.41 (quin,1H, J=6,82 Hz) 0.95-0.93 (d,3H,J=6,82 Hz) 0.85-0.83 (d,3H, J =6,95 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9 (J=1,85 Hz) 138.4 (J=4,81 Hz) 128.2, 128.1, 128.1, 126.6, 116.7, 88.1, 86.3, 52.2 (J=16,25 Hz) 31.9 (J=4,19 Hz) 22.6, 18.1 (J=3,53 Hz).

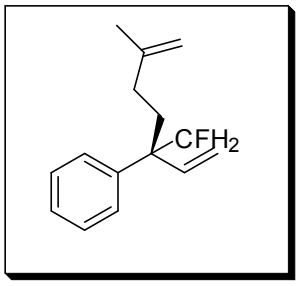
^{19}F NMR (376 MHz, $CDCl_3$) -64,02.

Yield: 40 %.

84 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:47 min. t2:48,7 min.

[α] = -26.5 (*c* 1.72, $CHCl_3$)

HRMS (EI + mode) m:z expected:192,1314 observed:192,1318



1g

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.30 (m,5H) 6.14-6.06 (qd,1H, J=11,1 Hz and J= 1,14 Hz) 5.40 (dd,1H, J=5,4 Hz and J= 0,4 Hz) 5.23 (d,1H, J=17,3 Hz) 4.81-4.73 (m,3H) 4.69-4.62 (q,1H, J=9,03 Hz) 2.12-2.08 (m,2H) 2.08-1.96 (m,2H) 1.78 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.9, 142.0 (J=2,89 Hz) 141.0 (J=4,09 Hz) 128.4, 127.4, 126.7, 115.4 (J=1,58 Hz) 109.7, 88.6, 86.7, 49.0 (J=17,38 Hz) 33.5 (J=4,19 Hz) 32.1, 22.7.

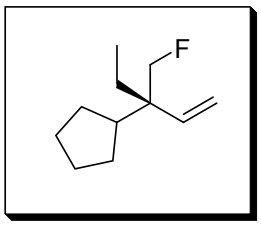
^{19}F NMR (MHz, $CDCl_3$) -60,92.

Yield: 54 %.

88 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:65,8 min. t2:66,7 min.

[α] = -3 (*c* 2.16, $CHCl_3$)

HRMS (EI + mode) m:z expected:218,1471 observed:218,1470
HRMS (EI + mode) m:z expected:192,1314 observed:192,1315



1h

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.72 (qd,1H, J=11,36 Hz and J=2,15 Hz) 5.20 (dd,1H, J=11,23 Hz and J=1,19 Hz) 4.98 (dd,1H, J= 11 Hz and J=1,20 Hz) 4.52 (q, J= 9,21 Hz) 4.33 (q, J=9,12 Hz) 2.04-1.95 (m,1H) 1.67-1.46 (m,9H) 1.33-1.24 (m,2H) 0.90 (t,1H, J=7,48 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.5 (J=2,89 Hz), 115.3, 87.2, 85.5, 45.7, 44.0, 4 26.9, 26.7, 26.4, 25.6, 8.3.

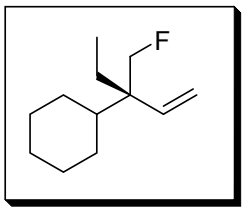
^{19}F NMR (376 MHz, $CDCl_3$) δ -68.71.

Yield: 42 %.

90 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:30,1 min. t2:31 min.

[α] = -2.4 (*c* 0.640, $CHCl_3$)

HRMS (EI + mode) m:z expected:170,1471 observed:170,1475



1i

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.69-5.61 (qd,1H, J=11,31 Hz and J=2,06 Hz) 5.17 (dd,1H, J=11,39 Hz and J=1,14 Hz) 4.94 (dd,1H, J=18 Hz and J=1,16 Hz) 4.51 (q,1H, J=9,30 Hz) 4.37 (q,1H, J=9,30 Hz) 1.80-1.00 (m,14H) 0.99(t,3H, J=7,48 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.7, 114.5 (J=1,58 Hz), 86.5, 84.8, 46.2, 42.1, 27.6, 26.8, 25.1, 8.2.

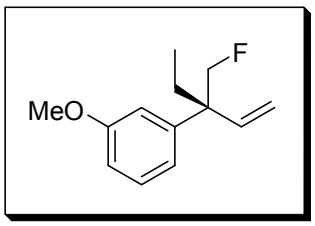
^{19}F NMR (376 MHz, $CDCl_3$) δ -65.

Yield: 60 %.

87 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:41,2 min. t2:41,8 min.

[α] = -1.80 (*c* 2.06, $CHCl_3$)

HRMS (EI + mode) m:z expected:184,1627 observed:184,1630



1j

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (s,1H) 6.90 (m,2H) 6.78 (dd,1H, J=2,56 Hz and J=0,85 Hz) 6.04 (qd,1H, J=11,09 Hz and J=1,16 Hz) 5.33 (d,1H,J=10,89 Hz) 5.18 (d,1H, J=17,84 Hz) 4.66 (q,1H, J= 9,06 Hz) 4.54 (q,1H, J=9,04 Hz) 3.81 (s,3H) 1.96-1.89 (m,2H) 0.83 (t,3H,J=7,48 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 144.1 (J=2,89 Hz), 141.1 (J=4,09 Hz), 129.3, 120.0, 115.4 (J=1,70 Hz), 114.41, 111.4, 88.4, 86.7, 55.3, 49.4 (J=17,38 Hz), 28.0, 8.7.

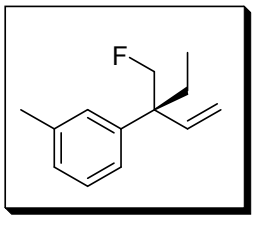
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.

Yield: 53 %.

65 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-180-1-170. t1:146,6 min. t2:147,7 min.

[α] = -4.8 (*c* 1, $CHCl_3$)

HRMS (EI + mode) m:z expected:208,1263 observed:208,1265



1k

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.08 (m,4H) 6.08-6.00 (qd,1H, J= 11.07 Hz and J=1,17 Hz) 5.36-5.33 (d,1H, J=11,13 Hz) 5.20-5.15 (d,1H, J=17,87 Hz) 4.77-4.69 (q,1H, J= 9,02 Hz) 4.65-4.57 (q,1H, J= 9,02 Hz) 2.40 (s,3H) 2.00-1.92 (m,2H) 0.85 (t,3H, J=7,48 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0(J=2,89 Hz), 140.7(J=4,20 Hz), 132.6, 129.2 128.5, 115.9 (J=1,58 Hz) ,88.3, 86.5, 77.5, 77.2, 76.9, 49.3 (J=17,50 Hz), 28.1, 8.6.

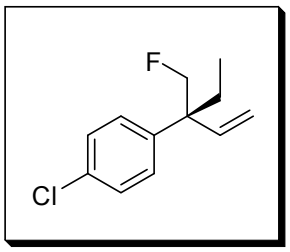
^{19}F NMR (376 MHz, $CDCl_3$) δ -61.99.

Yield: 60 %.

95 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:49,8 min. t2:50,1 min.

[α] = -9.3 (*c* 2.66, $CHCl_3$)

HRMS (EI + mode) m:z expected:192,1314 observed:192,1318



1l

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.26 (m,4H) 6.01-5.96 (qd,1H, J=11,09 Hz and 1,27 Hz) 5.35-5.32 (d,1H, J=11,09 Hz) 5.15-5.11 (d,1H, J=17,71 Hz) 4.72-4.63 (d,1H, J=9,1 Hz) 4.60-4.51 (d,1H, J=9,1 Hz) 1.94-1.87 (m,2H) 0.82 (t,3H, J=7,5 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.7(J=2,89 Hz), 132.6, 129.2, 128.5, 115.9(J=1,80 Hz), 88.3, 86.5, 49.3 (J=17,70 Hz), 28.1, 8.6.

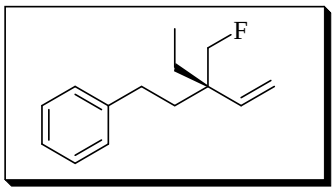
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.11.

Yield: 42 %.

94 % ee separated on GC: Hydrodex beta 6-TBDMS: 60-1-170. t1:70, 6 min. t2:71, 8 min.

[α] = -7.03 (*c* 1.56, $CHCl_3$)

HRMS (EI + mode) m:z expected:212,0768 observed:212,0770



1m

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.20 (m,5H) 5.77-5.70 (dd,1H, J=6,73 Hz and J=11,1 Hz) 5.26-5.23 (d,1H,J=11,23 Hz) 5.09-5.04 (d,1H, J=17,9 Hz) 4.46-4.34 (d,2H,J=47,73 Hz) 2.61-2.56 (t,2H, J=4,20 Hz) 1.75-1.71 (m,2H) 1.59-1.53 (m,2H) 0.94 (t,3H, J=7,54 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9 (J=2,89 Hz), 141.6, 128.6, 128.5, 126.0, 114.9 (J=1,60 Hz), 87.8, 86.1, 44.1 (J=17,40 Hz), 36.3 (J=4,40 Hz), 30.2, 26.7, 8.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -67.

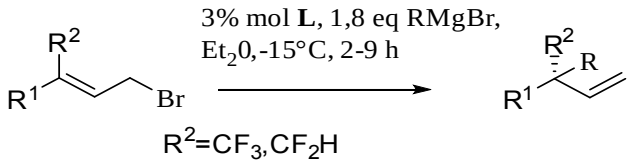
Yield: 60 %.

18 % ee mesured using SFC.

[α] = -1.50 (*c* 2.18, $CHCl_3$)

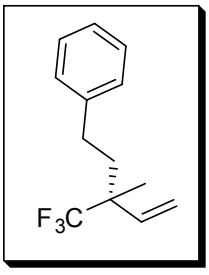
HRMS (EI + mode) m:z expected:206,1471 observed:206,1470

Catalysis adducts.



In a flame-dried Schlenk under N2 atmosphere, the allyl bromide derivative (0.2 mmol) and the ligand (3 mol %, **L4** for CF_3, **L5** for CF_2H) are suspended in dry Et2O (adjusted to 2.5 ml depending of the Grignard reagent volume) and cooled to -15°C. A solution of Grignard reagent X M in Et2O (1.8 eq) is added dropwise. After complete conversion, the mixture is quenched by addition of NH4Clsat. (2 ml) and stirred at roomtemperature during 15 min. The aqueous layer is separated and extracted with Et2O (3 × 3 ml). The combined organic fractions are dried over Na2SO4, filtered and concentrated in vaccuo. The residue is purified by flash column chromatography to offer a mixture of SN2' and SN2 products. GC or SFC on a chiral stationary phase shows the enantiomeric excess of the SN2' product. The absolute configuration was determined by comparison with described compounds. For the new products it was assigned by analogy.

N.B: Respecting the dilution is highly important for the reproducibility of the results.



1n

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.22 (m,5H) 6.00-5.92 (q,1H, J=10,81 Hz) 5.47 (d,1H,J=11,08 Hz) 5.40(d,1H,J=17,54 Hz) 2.64-2.59 (m,2H) 2.00-1.94 (m,2H) 1.37 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.7, 136.40, 128.5, 126.0, 118.0, 46.5 (J=24,07 Hz), 36.1, 29.9, 16.2, 16.2.

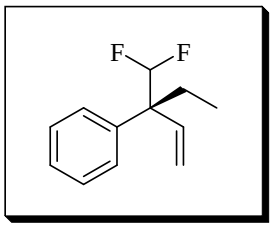
^{19}F NMR (376 MHz, $CDCl_3$) δ 85.54.

Yield: 80%.

79 % ee separated on GC: Hydrodex beta 3P: 60-1-170. t1:47 min. t2:47,6 min.

[α] = 26.6 (*c* 1.50, $CHCl_3$)

HRMS (EI + mode) m:z expected:228,1126 observed:228,1129



1o

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (m,5H) 6.08-6.03 (q,1H,J=12,54 Hz) 6.02 (t,1H, J= 50 Hz) 5.49-5.47 (d,1H,J=11,24 Hz) 5.30-5.26 (d,1H, J=16 Hz) 2.10-1.99 (m,2H) 0.88 (t,3H, J=7,47 Hz).
^{13}C NMR (101 MHz, $CDCl_3$) δ 138.7, 137.4, 128.7, 128.1, 127.3, 121.0, 118.5, 117.8, 116.1, 53.1, 29.9, 26.2, 8.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ 37.07(dd, J=90,58 Hz and J=275 Hz) ^{19}F-(NMR) coupled with hydrogen.

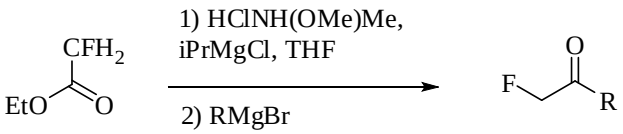
Yield:60 %.

95 % ee separated on GC: Hydrodex beta 3P: 60-120-1-170. t1:130 min. t2:132 min.

[α] = -3.3 (*c* 1.16, $CHCl_3$)

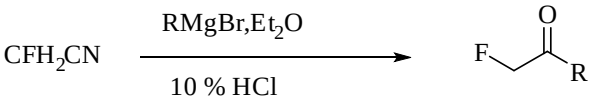
HRMS (EI + mode) m:z expected:196,1064 observed:196,1067

Fluorinated ketone.



Method A : For aryl Grignard.

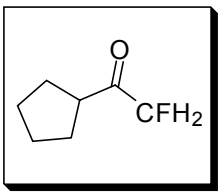
To a suspension of N,0-Dimethylhydroxylamine hydrochloride (1,2 eq) in 40 mL of dry THF at -15 °C a solution of iPrMgCl in THF (2,4 eq) is added dropwise. After 10 minutes at -15°C ethylfluoroacetate (10mmol, 1eq) is added dropwise and the resulting mixture is stirred at -15°C for 45 minutes. A solution of the appropriate Grignard is added dropwise and the mixture is stirred at -15°C for 30 min and at 0°C for an additional 1 hour. The mixture is carefully quenched with a saturated solution of NH4Cl, extracted two times with EtOAc, dried over sodium sulfate and concentrated in vacuo. The crude product is purified on SiO2 chromatography to afford compounds as slightly yellow oil.



Method B : For aryl and alkyl Grignard.

To a solution of fluoroacetonitrile (5 mmol,1 eq) in 30 mL of dry Et2O at 0°C the appropriate Grignard reagent (1,2 eq) is added dropwise. The mixture is stirred an additional 2 h at 0°C and the mixture is carefully quenched with a 10 % HCl solution, extracted two times with Et2O, dried over sodium sulfate and concentrated in vacuo. The crude product is purified on SiO2 chromatography to afford compounds as slightly yellow oil.

N.B: Those two methods are complementary and can be use when the other is inefficient.



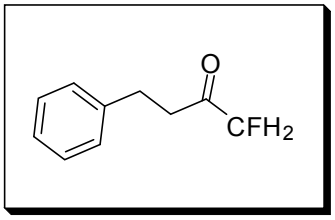
2a

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.98 (dd,1H, J= 1,10 Hz) 4.87 (dd,1H, J= 1,10 Hz) 3.18-3.09 (m,1H) 1.90-1.84 (m,4H) 1.72-1.67 (m,4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 208.9, 208.8, 85.3, 83.5, 46.7, 28.3, 26.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -65.77.

Yield: 50 % using Method B.



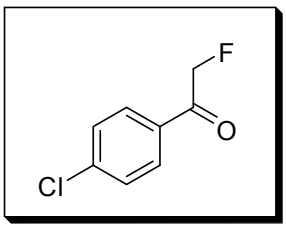
2b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.25 (m,5H) 4.89 (s,1H) 4.77 (s,1H) 3.04-2.96 (m,2H) 2.95-2.91 (m,2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 206.3, 140.4, 128.6, 128.3, 126.3, 86.0, 84.1, 39.9, 28.7, 28.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ -65.71.

Yield: 55 % using Method B.



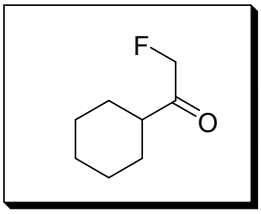
2c

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d,2H,J=8,5 Hz) 7.49(d,2H,J=8,60 Hz) 5.55-5.43 (d,2H,J=46,87 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.8, 132.2, 129.5, 84.7, 82.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ -67.82.

Yield: 48 % using Method A.



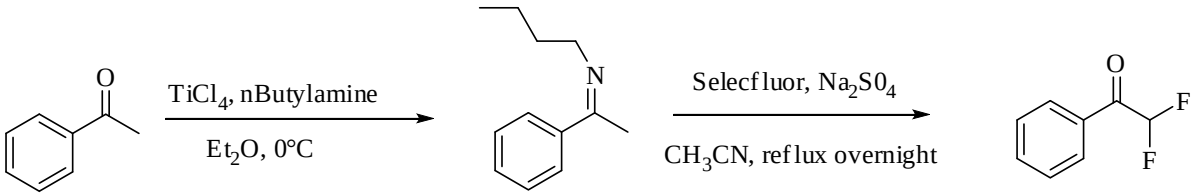
2d

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.94-4.82 (d,2H,J=47,66 Hz) 2.65 (m,1H) 1.87-1.67 (m,5H) 1.4-1.12 (m,5H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 209.4, 209.3, 85.1, 83.2, 46.4, 27.9, 25.9, 25.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -68.86.

Yield: 52 % using Method B.

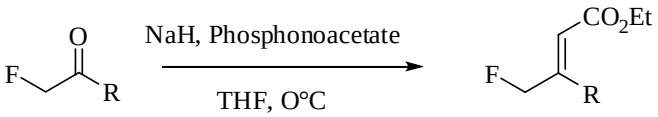
Diflluorinated ketone



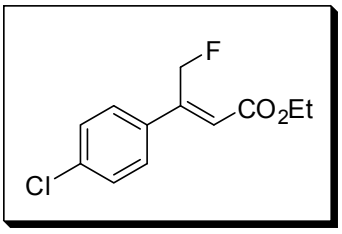
Titanium chloride (1M in dichloromethane,1 eq) was added dropwise to a mixture of acetophenone (1eq,15 mmol) and *n*butylamine (3eq) in dry Et_2O at 0°C. When the addition is finished the ice bath is removed and the white suspension is stirred at RT overnight. The reaction is quenched by the addition of 1M NaOH. The mixture is extracted with diethyl ether, dried over sodium sulfate and concentrated in vacuo. The crude product is pure enough to be engaged in the next step without any purification. Spectral and yield data identical to the existing literature.

Selecfluor (2 eq), dry sodium sulfate (300 mg) and the imine (1eq,3 mmol) is dissolved into dry acetonitrile. A reflux condenser is fitted and the mixture is refluxed overnight. The solution is cooled down and treated with 10 % aqueous HCl. The biphasic mixture is vigorously stirred for 30 min. The water phase is extracted with two portions of ether, dried over sodium sulfate and concentrated carefully in vacuo (volatil conpound 100 mBar,40 °C). the crude mixture is quickly filtered over a short plus of silica gel (silica gel sensible) in a Buchner using pentane/ether as eluant. After careful evaporation the compound is recovered as a yellow oil. Molecule 2f. Yield: 80 %. Spectral data comparable to literature.

Monofluoro and difluoro allylic ester.



To a suspension of NaH (1,2 eq) in 30 mL of dry THF at 0°C, triethylphosphonoacetate (1,1 eq) is added dropwise in order that the gas evolution is not too strong. After complete addition of the phosphonoacetate the mixture is stirred at 0°C for an additional 20 minutes and the ketone (1 eq) dissolved in 2 mL of THF is added. The ice bath is removed and the mixture is stirred overnight at RT. The mixture is carefully quenched with a saturated solution of NH4Cl, extracted two times with EtOAc, dried over sodium sulfate and concentrated in vacuo. The crude product is purified on SiO2 chromatography to afford compounds as slightly yellow oil with *E* stereochemistry.



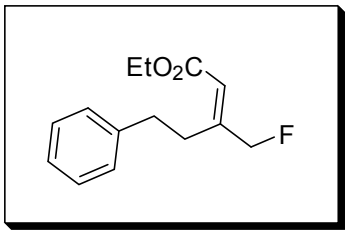
3a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.37 (m,4H) 6.15 (s,1H) 5.95-5.83 (dd,2H.J=47,47 Hz and J=1,23 Hz) 4.27-4.21 (q,2H,J=7,21 Hz) 1.31 (t,3H,J=7,15 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 165.5, 152.3, 152.1, 135.9, 135.8, 135.7, 129.0, 128.89, 128.8, 120.5, 120.5, 79.9, 78.3, 60.9, 14.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -55.28.

Yield: 55 %.



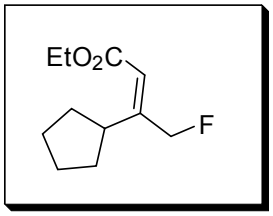
3b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.38 (t,2H, J=8,19 Hz) 7.33-7.29 (t,3H, J=8,02 Hz) 5.83, 5.83 (d,1H, J=1,66 Hz) 5.78 (d,1H, J= 1,65 Hz) 5.66 (d,1H, J= 1,75 Hz) 4.27-4.22 (q,2H, J= 7,12 Hz) 2.95-2.91 (t,2H, J= 7,24 Hz) 2.77-2.73 (t,2H, J= 7,5 Hz) 1.40-1.35 (t,3H, J=7,18 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 165.7, 158.2, 158.0, 140.9, 128.5, 128.5, 128.5, 128.40, 126.2, 116.4, 116.3, 83.0, 81.3, 60.2, 35.6, 35.5, 34.4, 14.2.

^{19}F NMR (376 MHz, $CDCl_3$) δ -63.27.

Yield: 65 %.



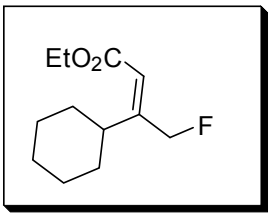
3c

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 (s,1H) 5.61 (s,1H) 5.49 (s,1H) 4.16-4.10 (q,2H, J=7,12 Hz) 2.79 (quintuplet,1H, J=6,57 Hz) 1.95-1.91 (m,2H) 1.72-1.61 (m,4H) 1.47.1.44 (m,2H) 1.28-1,24 (m,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 165.9, 161.7, 161.5, 113.8, 113.8, 82.6, 80.9, 59.9, 43.1, 43.0, 31.3, 24.7, 14.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -61.87.

Yield: 57 %.



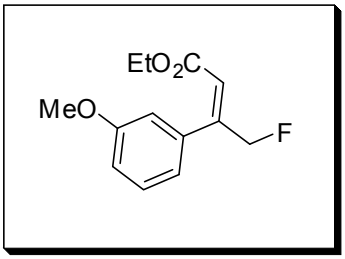
3d

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.69 (s,1H) 5.63-5.50 (dd,1H, J= 48,25 Hz and J=1,71 Hz) 4.17-4.12 (q,2H, J=7,12 Hz) 2.47-2.39 (m,1H) 1.88-1.61 (m,6H) 1.37-1.15 (m,10H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 166.4, 163.2, 163.1, 114.8, 114.7, 82.3, 80.7, 60.3, 40.9, 40.8, 32.2, 26.7, 26.3, 14.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.56.

Yield: 50 %.



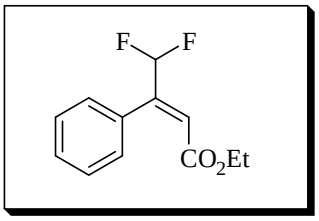
3e

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-6.94 (m,4H) 6.19 (s,1H) 5.95-5.83 (dd,2H,J=47,39 Hz and J=1,14 Hz) 4.27-4.22 (q,2H, J=7,15 Hz) 3.84 (s,3H) 1.36-1.32 (t,3H,J=7,15 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 165.7, 159.8, 153.1, 153.0, 129.7, 119.9, 115.1, 113.1, 80.0, 78.4, 77.5, 60.8, 55.5, 14.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -54.62.

Yield: 62 %.



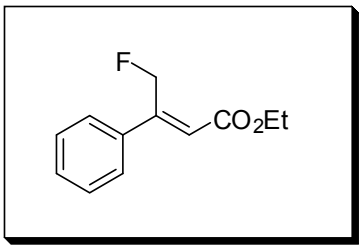
3f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (t,1H, J=54,71 Hz) 7.56 (m,2H) 7.43 (m,3H) 6.31 (s,1H) 4.31 (q,2H,J=7,46 Hz) 1.37 (t,3H,J=7,12 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.8, 149.2, 134.1, 129.9, 128.7, 128.2, 125.0, 124.95, 124.8, 113.1, 110.7, 108.4, 61.4, 29.9, 14.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ 45.84.

Yield: 49 %.

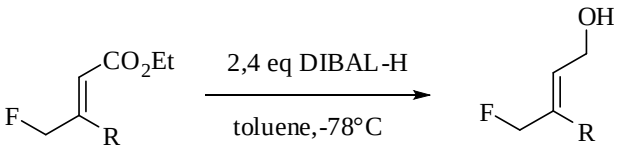


3g

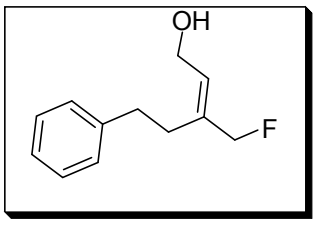
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.53 (m,2H) 7.47-7.45 (m,3H) 6.22 (s,1H) 6.03-5.90 (dd,2H, J=1,16 Hz and J=47,48 Hz) 4.31-4.26 (q,2H,J=7,17 Hz) 1.40-1.36 (t,3H,J=7,10 Hz).

Yield: 58 %.

Fluorinated allylic alcohol.



The ester (1eq) is dissolved in toluene (8mL/mmol) and cooled to -78°C using a dry ice/acetone bath. DIBAL-H (2,4eq, 1 M in hexane) is then added over 5 min. The mixture is stirred at -78°C for 30 minutes and at 0°C for an additional 30 minutes. The mixture is carefully quenched with an aqueous tartaric acid solution, extracted two times with EtOAc, dried over sodium sulfate and concentrated in vacuo. The crude product is purified on SiO2 chromatography (pentane/ether 1/1) to afford compounds as an oil.



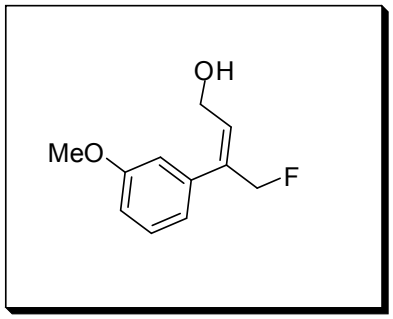
4a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (m,2H) 7.22 (m,2H) 5.69 (t,1H, J= 6,75 Hz) 5.03-4.91 (d,1H, J= 47,49 Hz) 4.20 (q,2H, J= 3,31 hz) 2.83 (t,2H,J=6,15 Hz) 2.50 (t,2H, J= 7,99 Hz) 1.28 (s,1H,OH).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.6, 137.6, 137.5, 129.9, 129.8, 128.6, 128.6, 128.5, 128.5, 128.5, 126.2, 81.3, 79.7, 77.5, 58.7, 36.6, 34.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -52.50.

Yield: 88 %.



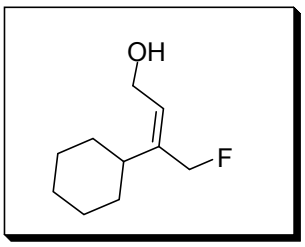
4b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (t,1H, J= 8,01 Hz) (t,2H, J= 7,44 Hz) 6.90 (dd,1H, J= 2,68 Hz and J= 8,34 Hz) 6.24 (sextuplet,1H, J= 3,63 Hz) 5.37 (d,1H, J=47,65 Hz) 4.44 (q,2H, J= 4,21 Hz) 3.84 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 140.9, 136.6 133.80, 129.6, 118.7, 113.2, 112.1, 80.4, 78.8, 58.9, 55.2.

^{19}F NMR (376 MHz, $CDCl_3$) δ -46.14.

Yield: 90 %.



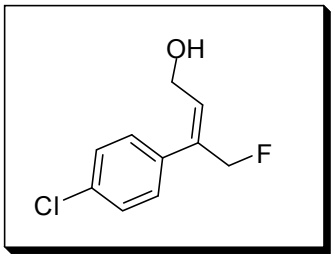
4c

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.70-5.65 (m,1H) 4.99-4.87 (d,1H, J=47,75 Hz) 4.24 (m,2H) 2.07-2.00 (t,1H, J= 11,65 Hz) 1.80-1.15 (m,10H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.1, 128.6, 80.7, 79.1, 59.0, 43.0, 32.2, 26.8, 26.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ -47.94.

Yield: 91 %.



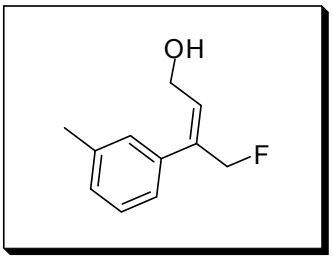
4d

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.31 (m,4H) 6.26-6.23 (m,2H) 5.39-5.25 (d,1H,47,6 Hz) 4.52-4.48 (m,2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.7, 136.0 (J=214,31 Hz), 133.8, 133.6 (J=7,61 Hz), 128.7, 127.6, 80.2, 78.6, 59.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -46.94.

Yield: 85 %.

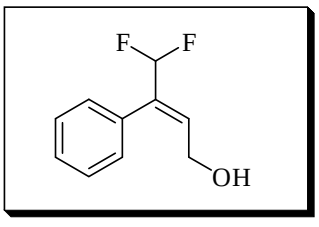


4e

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m,1H) 7.20-7.18 (m,1H) 6.29-6.24 (sextuplet,1H, J= 1,07 Hz) 5.42-5.29 (d,1H, J= 47,64 Hz) 4.52- 4.49 (q,2H, J= 4,12 Hz) 2.43 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.6, 139.5, 138.4, 137.4, 137.3, 133.3, 133.2, 128.8, 128.6, 127.2, 123.5, 80.8, 79.1, 59.4, 21.7.

.Yield: 89 %.



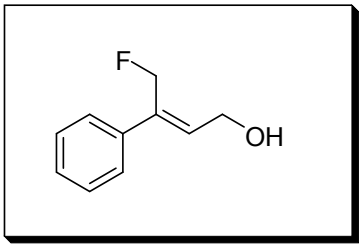
4f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.36 (m,5H) 6.86-6.59 (t,1H,J=54,85 Hz) 6.22 (t,1H,J=6,31 Hz) 4.56-4.53 (quintuplet,2H, J=2,95 Hz) 1.75 (s,1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.6, 136.6, 136.5, 136.5, 136.2, 135.8, 128.6, 128.4, 127.69, 115.5, 113.1, 110.8, 58.9.

^{19}F NMR (376 MHz, $CDCl_3$) δ 50.13.

Yield: 81 %.

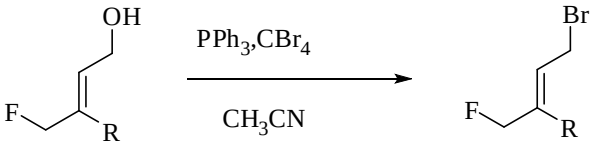


4g

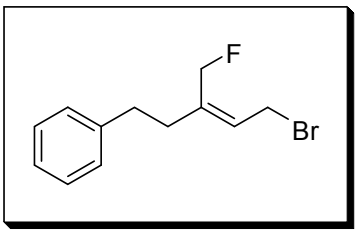
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.31 (m,5H) 6.30 (sextuplet,1H, J=3,66 Hz) 5.31 (d,2H, J=47,63 Hz) 4.54-4.50 (q,2H,J=4,1 Hz) 1.65 (t,1H, J=5,68 Hz).

Yield: 94 %.

Fluorinated allylic bromide.



In a conditioned Schlenk the alcohol (1 eq) is dissolved into 10 mL of dry acetonitrile. At 0°C $P(Ph)_3$ (1,5 eq) and CBr_4 (1,5 eq) are added in this order and portionwise. The ice bath is removed and the resulting mixture is stirred for 1 h at RT. The solvent is evaporated and the residue purified by silica gel chromatography using pentane as eluant.



5a

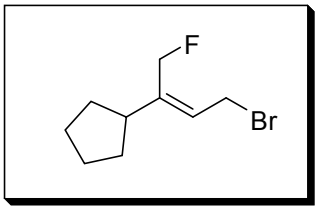
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.21 (m,5H) 5.79, 5.78-5.73 (t,1H, J= 8,61 Hz) 5.08-4.96, (d,J=47,25 Hz) 4.04-4.01 (dd,2H, J=1,99 Hz and J=8,60 Hz) 2.83-2.79 (t,2H, J=7,50 Hz) 2.53-2.49 (t,2H,J= 6,30 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.2, 140.1, 140.0, 128.7, 128.7, 128.6, 128.6, 128.6, 126.43, 126.3, 126.3, 80.6, 79.0, 36.5, 36.5, 34.5, 26.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ 52.69 ..

Yield: 89 %.

HRMS (EI + mode) m:z expected:256,0263 observed:256,0260



5b

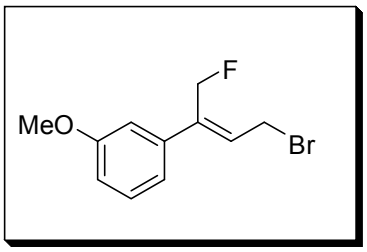
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.84-5.79 (td,1H, J= 2,65 Hz and J=7,17 Hz) 5.79,5.06-4.92 (dd,2H, J=2,13 Hz and J= 47,54 Hz) J= 4.09-4.04 (m,2H) 2.62-2.54 (quin,1H, J= 8,09 Hz) 1.88-1.27 (m,8H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.6, 143.5, 124.8, 124.7, 80.2, 78.5, 45.1, 45.0, 31.3, 27.4, 25.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -51.69 .

Yield: 80 %.

HRMS (EI + mode) m:z expected:220,0263 observed:220,0264



5c

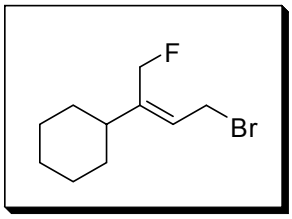
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.27 (m,2H) 7.04-7.02 (d,1H, J= 7,73 Hz) 6.90-6.87 (dd,1H, J= 0,9 Hz and J= 8,28 Hz) 6.34-6.29 8td,1H, J= 3,49 Hz and J=8,65 Hz) 5.40-5.29 (d,2H, J=47,52 Hz) 4.25-4.23 (dd,2H, J= 2,52 Hz and J= 8,64 Hz) 3.84 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 140.4, 140.4, 139.2, 139.0, 129.8, 129.6, 129.5, 119.07, 113.9, 112.4, 79.8, 78.2, 55.5, 26.9.

^{19}F NMR (376 MHz, $CDCl_3$) δ -48.41.

Yield: 86 %.

HRMS (EI + mode) m:z expected:258,0056 observed:258,0060



5d

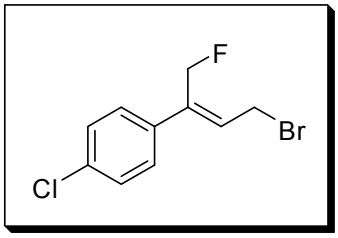
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82-5.78 (td,1H, J=1,06 Hz and J=8,62 Hz) 5.09-4.97 (d,2H,J=47,6 Hz) 4.13 (dd,2H, J= 2,53 Hz and J=8,52 Hz) 2.15--1.81 (m,3H) 1.50-1.20 (m,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.4, 145.3, 125.2, 125.1, 79.9, 78.2, 77.5, 43.1, 43.1, 31.9, 27.5, 26.7, 26.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ -51.19.

Yield: 92 %.

HRMS (EI + mode) m:z expected:234,0419 observed:234,0420



5e

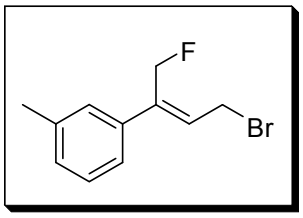
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (m,4H) 6.32-6.27 (td,1H, J= 3,24 Hz and J=8,63 Hz) 5.39-5.27 (d,2H, J= 47,46 Hz) 4.23-4.21 (dd,2H, J=2,50 Hz and J= 8,63 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.2, 138.1, 137.3, 137.3, 134.5, 129.7, 129.6, 129.0, 127.9, 79.5, 77.8, 26.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -49.13 .

Yield: 73 %.

HRMS (EI + mode) m:z expected:261,9560 observed: 261,9562



5f

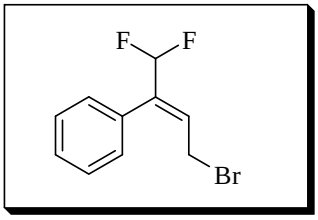
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.18 (m,4H) 6.33-6.28 (td,1H, J=3,46 hz and J=8,66 Hz) 5.42-5.30 (d,2H, J=47,58 Hz) 4.26-4.24 (dd,2H, J=2,51 Hz and J=8,67 Hz) 2.39 (s,3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.4, 139.3, 138.9, 138.9, 138.5, 129.3, 129.2, 129.1, 128.75, 127.3, 123.7, 79.9, 78.2, 27.1, 21.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ -48.46.

Yield: 80 %.

HRMS (EI + mode) m:z expected:242,0106 observed: 242,0108



5g

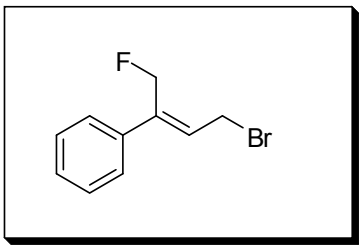
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (m,5H) 6.69 (t,1H,J=54,43 Hz) 6.31 (t,1H, J=8,79 Hz) 4.27 (dt,2H, J=1,72 Hz and J= 8,70 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.7, 137.5, 137.3, 135.7, 135.6, 135.6, 132.2, 132.2, 132.15, 128.9, 128.8, 128.7, 127.6, 114.9, 112.5, 110.2, 25.2.

^{19}F NMR (376 MHz, $CDCl_3$) δ 49.88.

Yield: 70 %.

HRMS (EI + mode) m:z expected:245,9856 observed: 245,9857



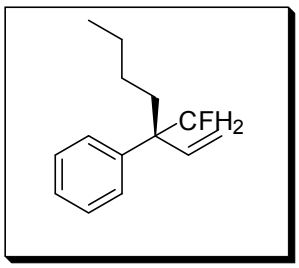
5h

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.28 (m,5H) 6.29 (td,1H,J=3,37 Hz and J= 8,62 Hz) 5.46- 5.30 (d,2H, J=47,51 Hz) 4.24 (dd,2H, J=2,45 Hz and J=8,66 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -48.66.

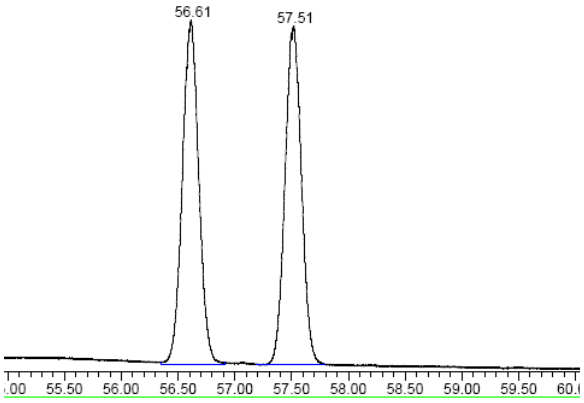
Yield: 81 %.

HRMS (EI + mode) m:z expected:227,9950 observed: 227,9951



1c

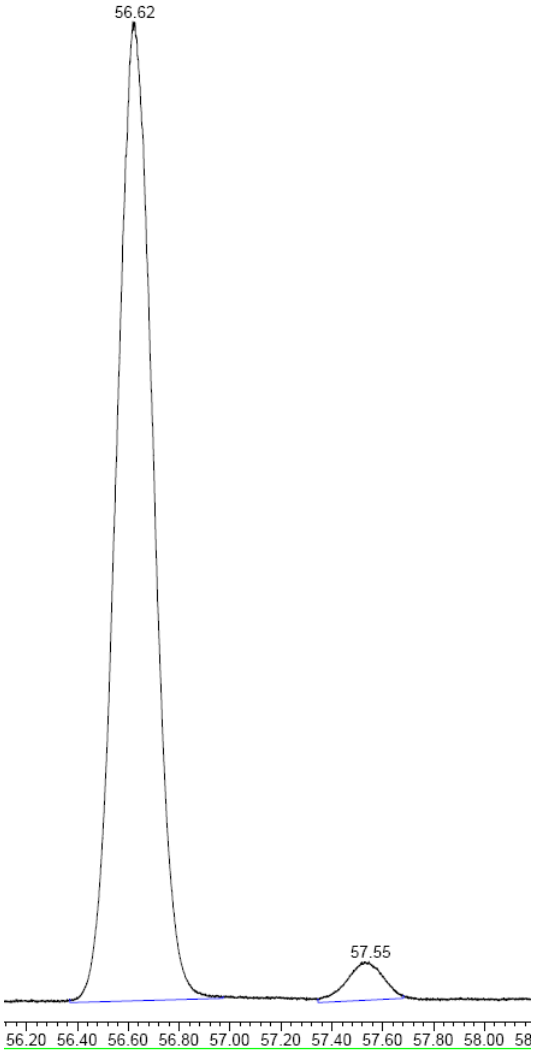
```
File       :C:\MSDChem\2\DATA\GRA699R.D
Operator   : DG
Acquired   : 22.03.2012 03:29:18 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA699R
Misc Info  :
Vial Number: 3
```




```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA699R.D
Signal(s) : FID1A.CH
Acq On    : 22.03.2012 03:29:18 PM
Sample    : GRA699R
Misc      :
ALS Vial  : 3   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	56.614	56.351	56.912	M	188793	18935991	100.00%	50.505%
2	57.514	57.185	57.793	M	185469	18556938	98.00%	49.495%

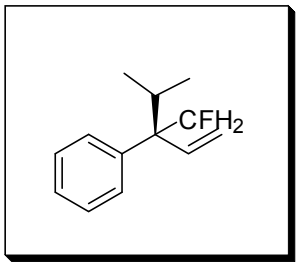
Sum of corrected areas: 37492929




```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA698.D
 Signal(s) : FID1A.CH
 Acq On    : 22.03.2012 06:10:16 PM
 Sample    : GRA698
 Misc      :
 ALS Vial  : 1   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

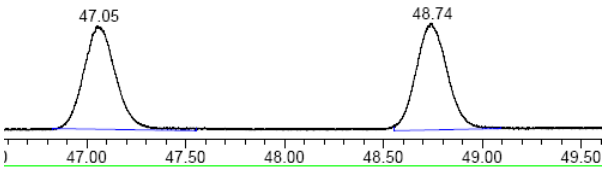
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	56.618	56.370	56.972	M	236659	23947853	100.00%	96.272%
2	57.545	57.346	57.682	M	9346	927279	3.87%	3.728%

Sum of corrected areas: 24875131



1f

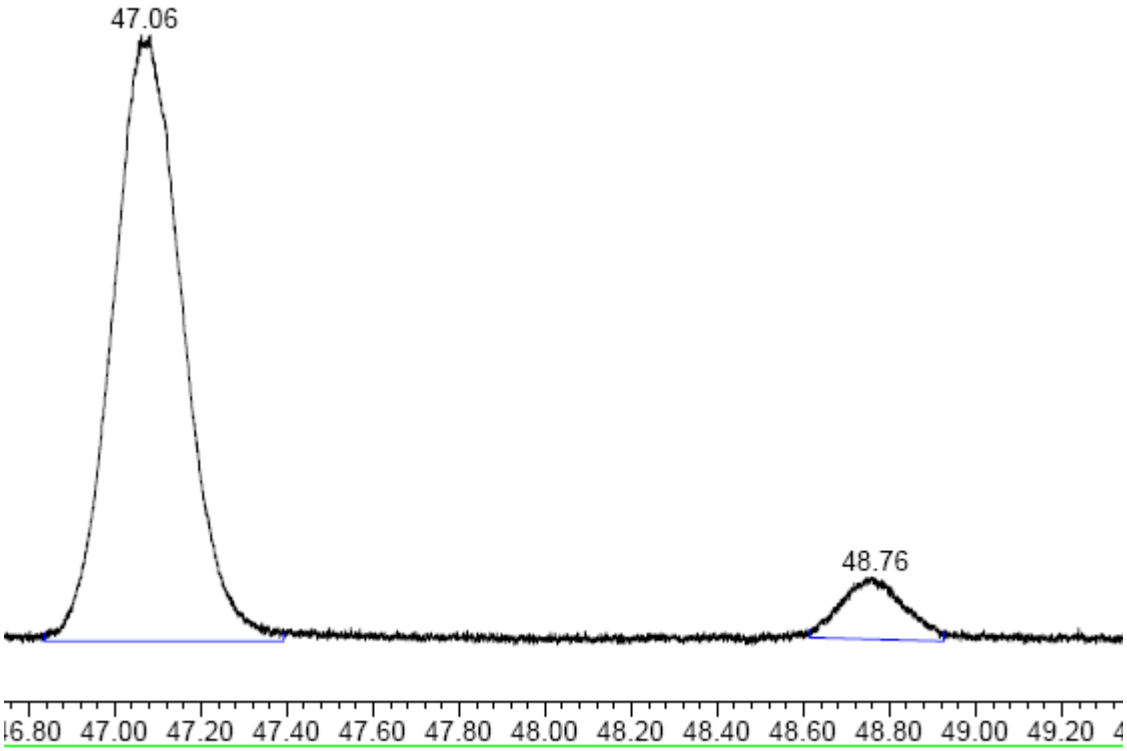
```
File       :C:\MSDChem\2\DATA\gra720.D
Operator   : DG
Acquired   : 03.04.2012 07:00:28 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: gra720
Misc Info  :
Vial Number: 5
```




```
Data Path : C:\MSDChem\2\DATA\
Data File : gra720.D
Signal(s) : FID1A.CH
Acq On    : 03.04.2012 07:00:28 PM
Sample    : gra720
Misc      :
ALS Vial  : 5   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

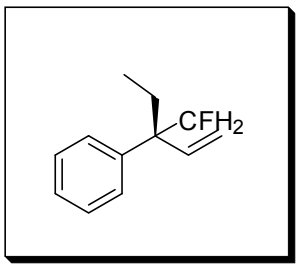
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	47.051	46.828	47.551	M	31724	3602740	99.89%	49.972%
2	48.737	48.554	49.091	M	32764	3606805	100.00%	50.028%
			Sum of corrected areas:			7209544		

```
File       :C:\MSDChem\2\DATA\gra721c.D
Operator   : dm
Acquired   : 05.04.2012 01:35:03 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: gra721c
Misc Info  :
Vial Number: 5
```



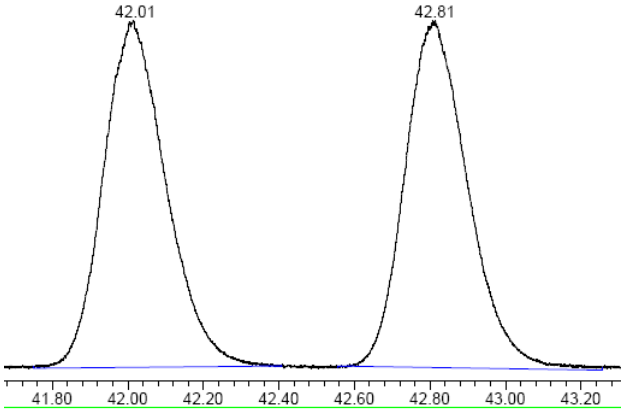

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : gra721c.D
 Signal(s) : FID1A.CH
 Acq On    : 05.04.2012 01:35:03 PM
 Sample    : gra721c
 Misc      :
 ALS Vial  : 5   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	47.062	46.837	47.392	M	29871	3389925	100.00%	91.772%
2	48.760	48.614	48.926	M	3064	303920	8.97%	8.228%
			Sum of corrected areas:			3693846		



1e

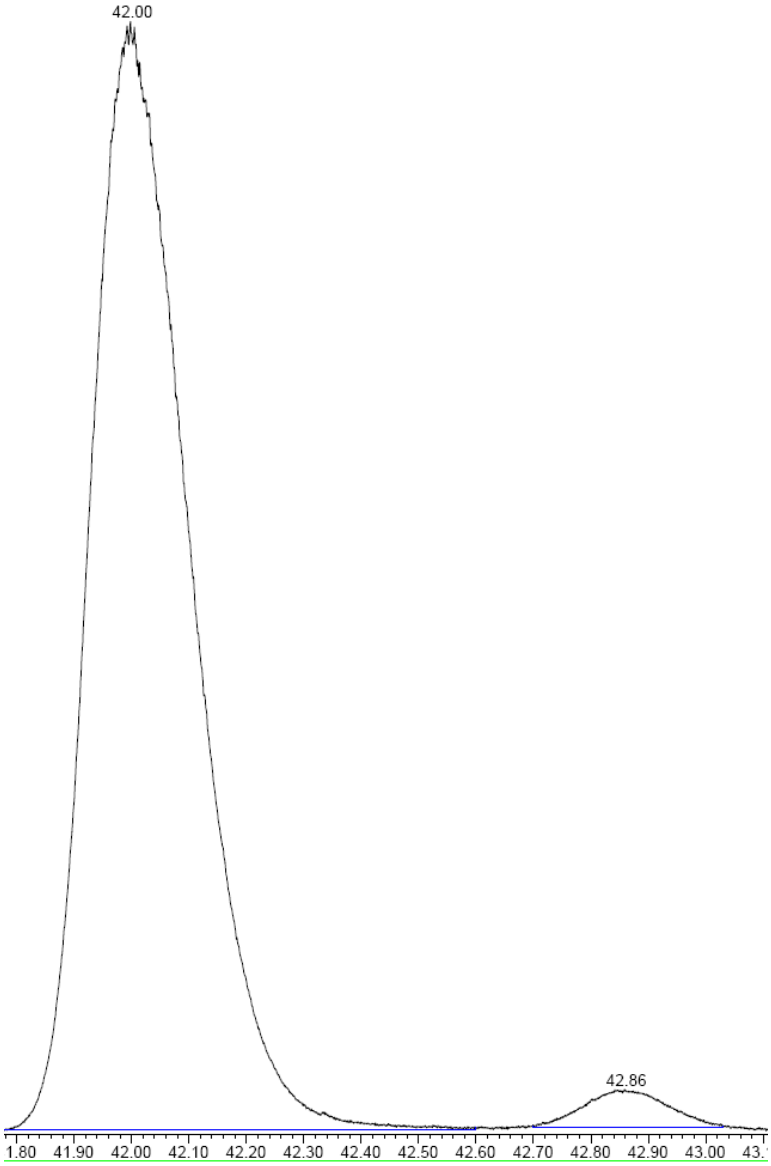
```
File       :C:\MSDChem\2\DATA\GRA658R1.D
Operator   : DG
Acquired   : 28.02.2012 09:20:02 AM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA658R1
Misc Info  :
Vial Number: 1
```




```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA658R1.D
Signal(s) : FID1A.CH
Acq On    : 28.02.2012 09:20:02 AM
Sample    : GRA658R1
Misc      :
ALS Vial  : 1   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	42.014	41.749	42.407	M	63431	7321395	99.51%	49.877%
2	42.807	42.555	43.257	M	63557	7357410	100.00%	50.123%
			Sum of corrected areas:			14678805		

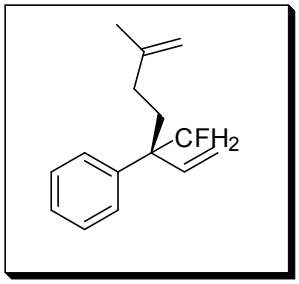
```
File       :C:\MSDChem\2\DATA\GR716R.D
Operator   : DG
Acquired   : 02.04.2012 04:07:51 PM using AcqMethod F60-1-130.M
Instrument :   GC CHIRAL
Sample Name: GR716C
Misc Info  :
Vial Number: 1
```




```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GR716R.D
 Signal(s) : FID1A.CH
 Acq On    : 02.04.2012 04:07:51 PM
 Sample    : GR716C
 Misc      :
 ALS Vial  : 1   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

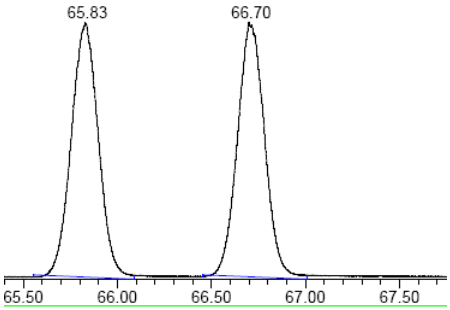
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	41.998	41.777	42.468	M	199461	23943132	100.00%	97.077%
2	42.859	42.696	43.028	M	6980	720860	3.01%	2.923%

Sum of corrected areas: 24663992



1g

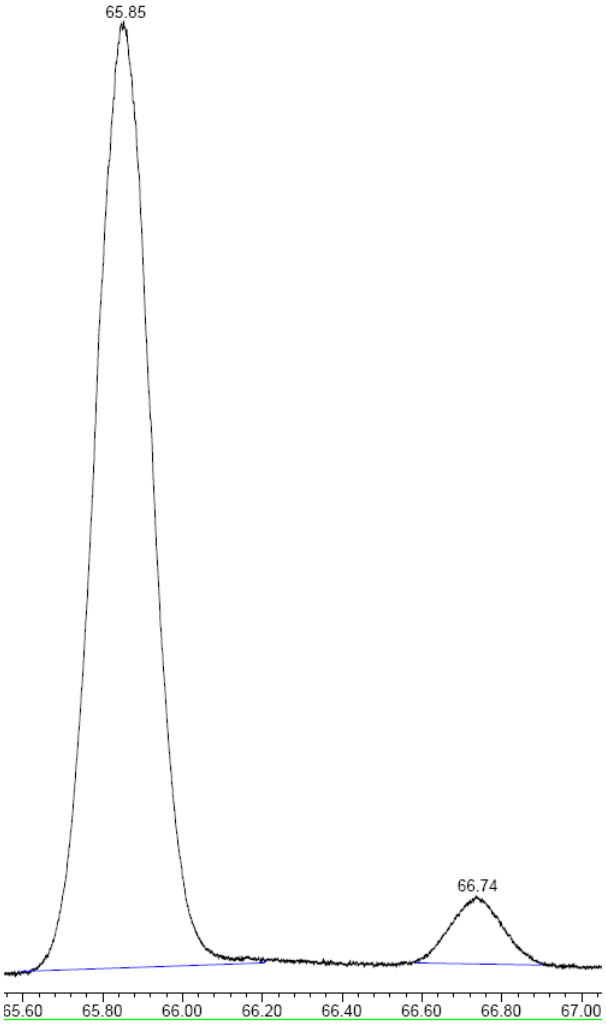
```
File       :C:\MSDChem\2\DATA\GRA704R.D
Operator   : LH
Acquired   : 23.03.2012 09:45:30 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA704R
Misc Info  :
Vial Number: 3
```




```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA704R.D
 Signal(s) : FID1A.CH
 Acq On    : 23.03.2012 09:45:30 PM
 Sample    : GRA704R
 Misc      :
 ALS Vial  : 3   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

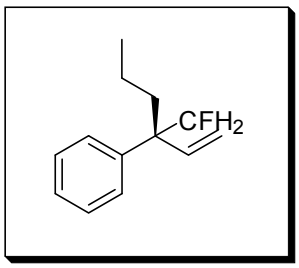
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	65.828	65.552	66.088	M	251864	25083260	99.35%	49.838%
2	66.698	66.453	67.010	M	251410	25246231	100.00%	50.162%
			Sum of corrected areas:			50329491		

```
File       :C:\MSDChem\2\DATA\gra719.D
Operator   : DG
Acquired   : 03.04.2012 03:04:54 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: gra719
Misc Info  :
Vial Number: 4
```



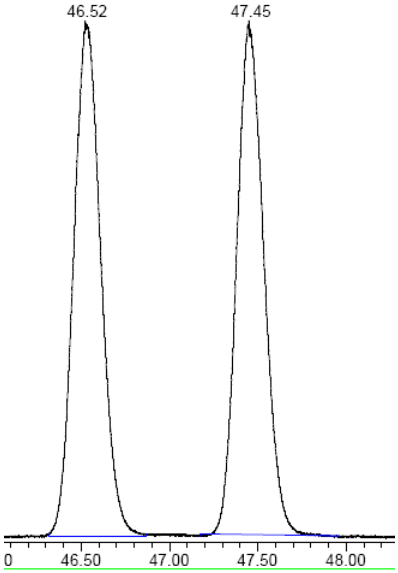

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : gra719.D
 Signal(s) : FID1A.CH
 Acq On    : 03.04.2012 03:04:54 PM
 Sample    : gra719
 Misc      :
 ALS Vial  : 4   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	65.852	65.597	66.205	M	85501	8567640	100.00%	94.023%
2	66.735	66.586	66.898	M	6181	544660	6.36%	5.977%
			Sum of corrected areas:			9112300		



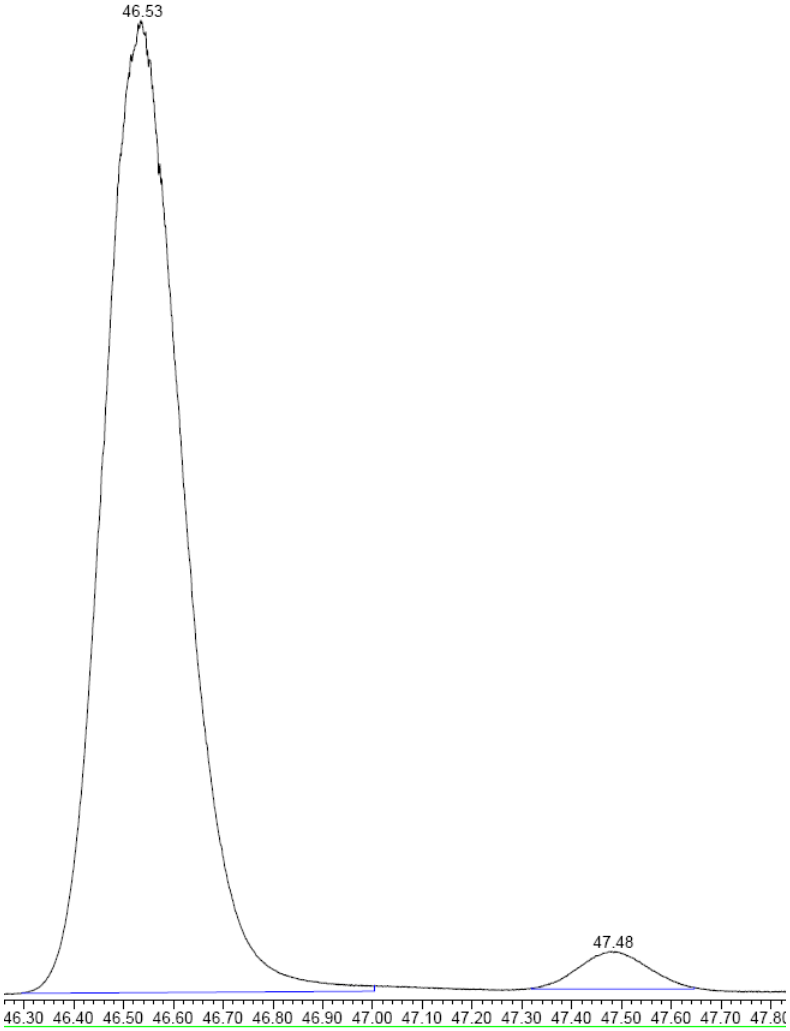
1b

```
File       :C:\MSDChem\2\DATA\GRA702R.D
Operator   : LH
Acquired   : 23.03.2012 05:50:06 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA702R
Misc Info  :
Vial Number: 1
```



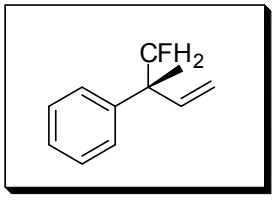

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA702R.D
 Signal(s) : FID1A.CH
 Acq On    : 23.03.2012 05:50:06 PM
 Sample    : GRA702R
 Misc      :
 ALS Vial  : 1   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH

peak  R.T.   Start     End   PK  peak      peak     peak    % of
  #   min     min      min   TY height     area    % max.   total
--- -----   -----    ----  --- -------   -------  ------  ------
  1 46.523  46.315  46.869     M 202707  21544308 100.00% 50.159%
  2 47.449  47.175  47.959     M 202276  21407853  99.37% 49.841%
                    Sum of corrected areas:    42952162
```

```
File       :C:\MSDChem\2\DATA\gra794.D
Operator   : dm
Acquired   : 25.05.2012 01:14:23 PM using AcqMethod F60-1-130.M
Instrument :   GC CHIRAL
Sample Name: gra794
Misc Info  :
Vial Number: 7
```



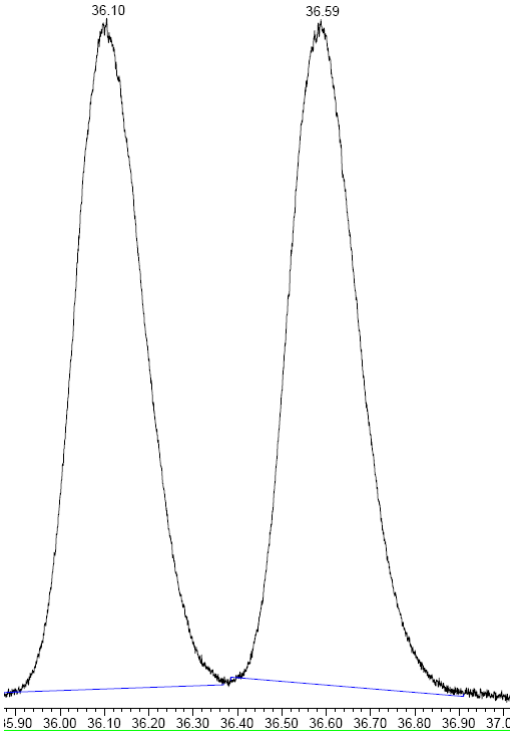

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : gra794.D
 Signal(s) : FID1A.CH
 Acq On    : 25.05.2012 01:14:23 PM
 Sample    : gra794
 Misc      :
 ALS Vial  : 7   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	46.535	46.296	47.004	M	431873	48337400	100.00%	96.755%
2	47.480	47.319	47.645	M	16756	1621136	3.35%	3.245%
				Sum of corrected areas:		49958536		



1a

```
File       :C:\MSDChem\2\DATA\GRA695R.D
Operator   : DG
Acquired   : 21.03.2012 05:53:25 PM using AcqMethod BOTH.M
Instrument :   GC CHIRAL
Sample Name: GRA695R
Misc Info  :
Vial Number: 1
```

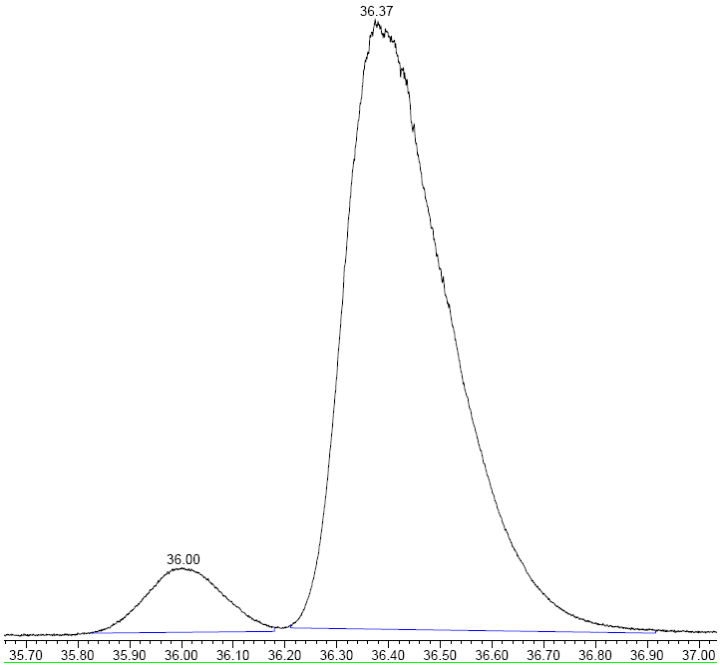



```
File       :C:\MSDChem\2\DATA\GRA715.D
Operator   : DG
Acquired   : 02.04.2012 09:27:08 AM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA715
Misc Info  :
Vial Number: 7
```

```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA695R.D
Signal(s) : Signal #1: FID1A.CH  Signal #2: FID2B.CH
Acq On    : 21.03.2012 05:53:25 PM
Sample    : GRA695R
Misc      :
ALS Vial  : 1   Sample Multiplier: 1

Integration File signal 1: events.e
Integration File signal 2: events2.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal #1 : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	36.105	35.873	36.369	M	40369	4430806	100.00%	50.007%
2	36.588	36.385	36.910	M	40040	4429531	99.97%	49.993%

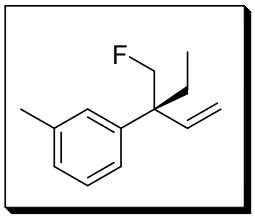
Sum of corrected areas: 8860336




```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA715.D
Signal(s) : FID1A.CH
Acq On    : 02.04.2012 09:27:08 AM
Sample    : GRA715
Misc      :
ALS Vial  : 7   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

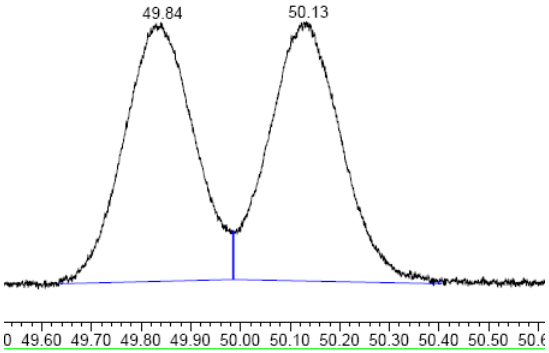
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	36.001	35.829	36.180	M	13606	1418320	7.97%	7.385%
2	36.374	36.211	36.916	M	128290	17787077	100.00%	92.615%

Sum of corrected areas: 19205396



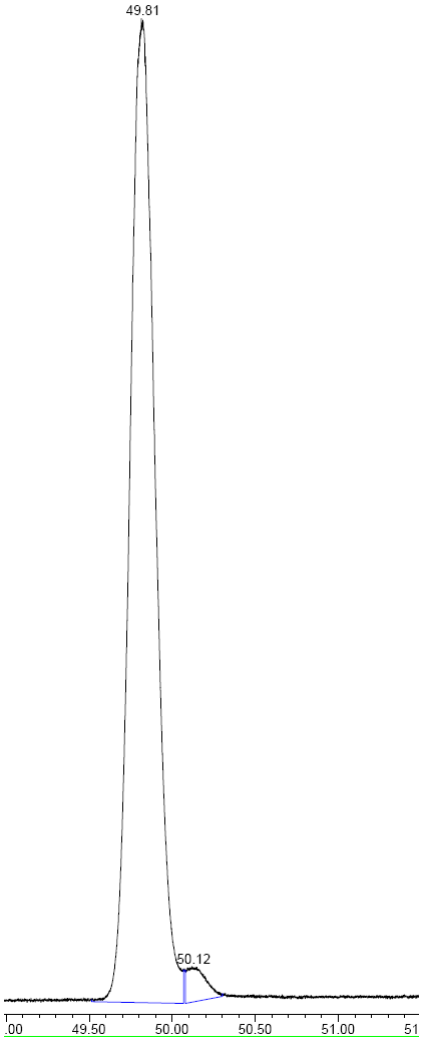
1l

```
File       :C:\MSDChem\2\DATA\GRA775R.D
Operator   : DG
Acquired   : 16.05.2012 06:16:15 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA775R
Misc Info  :
Vial Number: 5
```

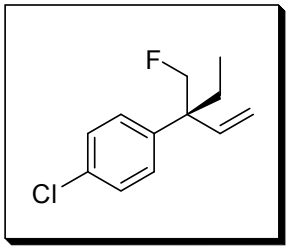



```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA775R.D
 Signal(s) : FID1A.CH
 Acq On    : 16.05.2012 06:16:15 PM
 Sample    : GRA775R
 Misc      :
 ALS Vial  : 5   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH

peak  R.T.   Start     End   PK  peak      peak     peak    % of
  #   min     min      min   TY height     area    % max.   total
---  -----   -----    ----  --- -------   -------  ------  ------
  1 49.839  49.637  49.985     M  14685   1444473  96.50% 49.110%
  2 50.133  49.986  50.404     M  14756   1496818 100.00% 50.890%
                    Sum of corrected areas:     2941292
```

```
File       :C:\MSDChem\2\DATA\GR777.D
Operator   : DG
Acquired   : 18.05.2012 01:27:33 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GR777
Misc Info  :
Vial Number: 5
```

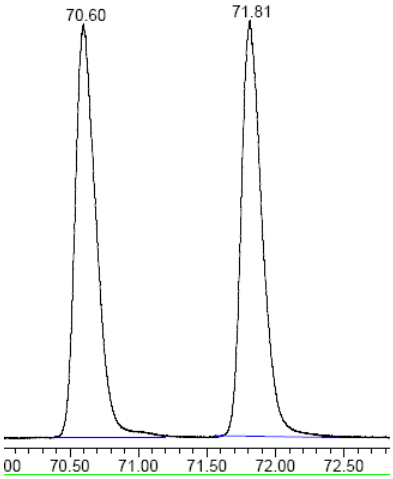



```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GR777.D
 Signal(s) : FID1A.CH
 Acq On    : 18.05.2012 01:27:33 PM
 Sample    : GR777
 Misc      :
 ALS Vial  : 5   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH

peak  R.T.   Start     End   PK  peak      peak     peak    % of
  #   min     min      min   TY height     area    % max.   total
---  -----   -----    ----  --- -------   -------  ------  ------
  1 49.813  49.514  50.069     M 161286  17032987 100.00% 97.374%
  2 50.119  50.079  50.303     M   5855    459345   2.70%  2.626%
                    Sum of corrected areas:    17492332
```



1l

```
File       :C:\MSDChem\2\DATA\GRA770R.D
Operator   : DG
Acquired   : 14.05.2012 01:14:02 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA770R
Misc Info  :
Vial Number: 3
```

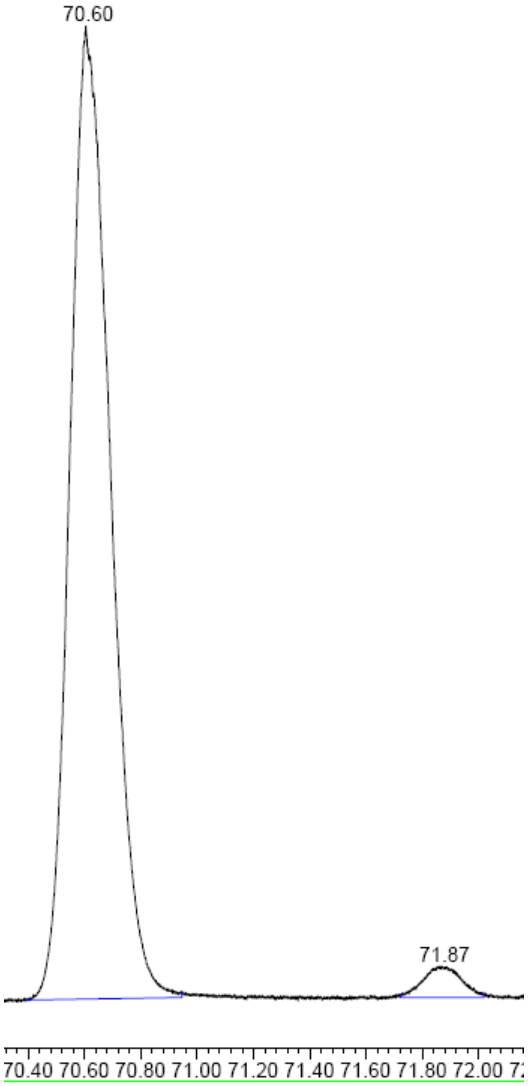



```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA770R.D
Signal(s) : FID1A.CH
Acq On    : 14.05.2012 01:14:02 PM
Sample    : GRA770R
Misc      :
ALS Vial  : 3   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	70.604	70.377	71.195	M	227617	24327168	100.00%	50.082%
2	71.811	71.562	72.450	M	228735	24247624	99.67%	49.918%

Sum of corrected areas: 48574792

```
File       :C:\MSDChem\2\DATA\GRA781.D
Operator   : DG
Acquired   : 19.05.2012 12:36:33 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA781
Misc Info  :
Vial Number: 5
```



Data Path : C:\MSDChem\2\DATA\
Data File : GRA781.D
Signal(s) : FID1A.CH
Acq On : 19.05.2012 12:36:33 PM
Sample : GRA781
Misc :
ALS Vial : 5 Sample Multiplier: 1

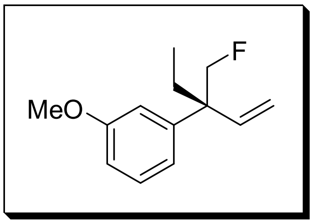
Integration File: events.e

Method : C:\MSDCHEM\2\METHODS\F60-1.M
Title :

Signal : FID1A.CH

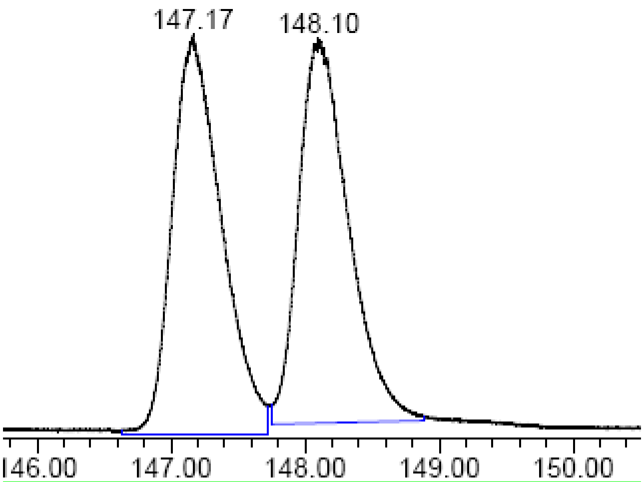
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	70.604	70.396	70.945	M	136235	13613959	100.00%	97.072%
2	71.869	71.722	72.018	M	4382	410606	3.02%	2.928%

Sum of corrected areas: 14024566



1j

```
File       :C:\MSDChem\2\DATA\DG749.D
Operator   : DG
Acquired   : 03.05.2012 05:29:22 PM using AcqMethod F85-120.M
Instrument :   GC CHIRAL
Sample Name: DG749
Misc Info  :
Vial Number: 5
```

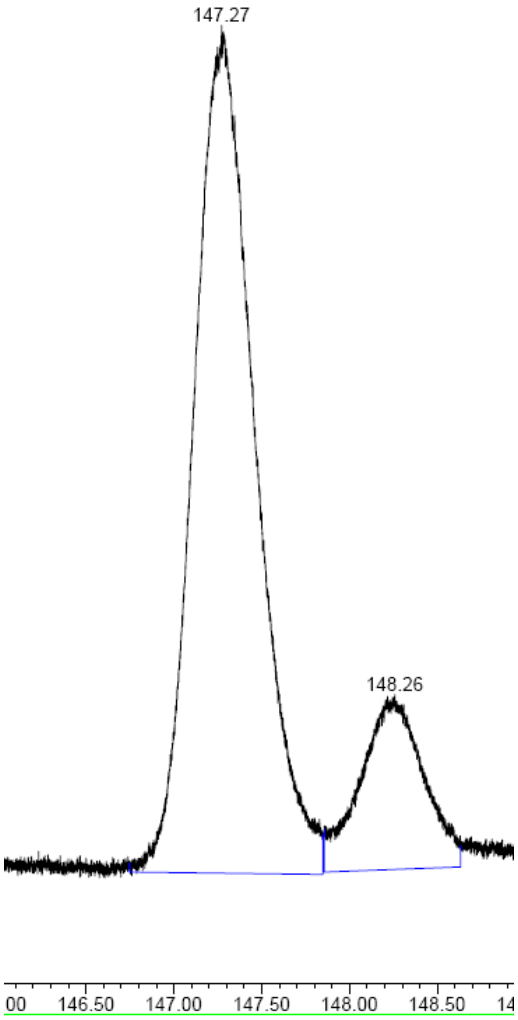



```
Data Path : C:\MSDChem\2\DATA\
Data File : DG749.D
Signal(s) : FID1A.CH
Acq On    : 03.05.2012 05:29:22 PM
Sample    : DG749
Misc      :
ALS Vial  : 5   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	147.165	146.633	147.728	M	112487	28687320	100.00%	50.851%
2	148.102	147.752	148.883	M	107944	27727626	96.65%	49.149%

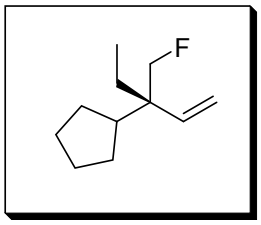
Sum of corrected areas: 56414946

```
File       :C:\MSDChem\2\DATA\GRA757.D
Operator   : LH
Acquired   : 04.05.2012 02:55:35 PM using AcqMethod F85-120.M
Instrument :   GC CHIRAL
Sample Name: GRA757
Misc Info  :
Vial Number: 3
```



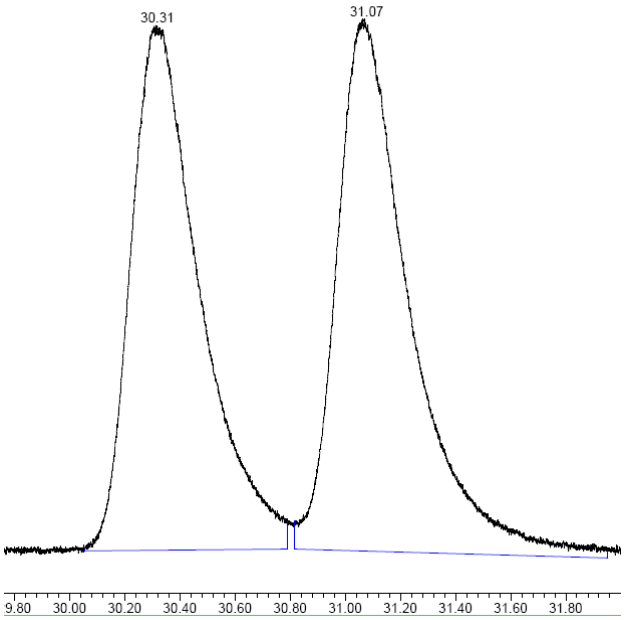

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA757.D
 Signal(s) : FID1A.CH
 Acq On    : 04.05.2012 02:55:35 PM
 Sample    : GRA757
 Misc      :
 ALS Vial  : 3   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	147.272	146.746	147.849	M	25192	6074322	100.00%	82.660%
2	148.257	147.855	148.631	M	5198	1274281	20.98%	17.340%
			Sum of corrected areas:			7348602		



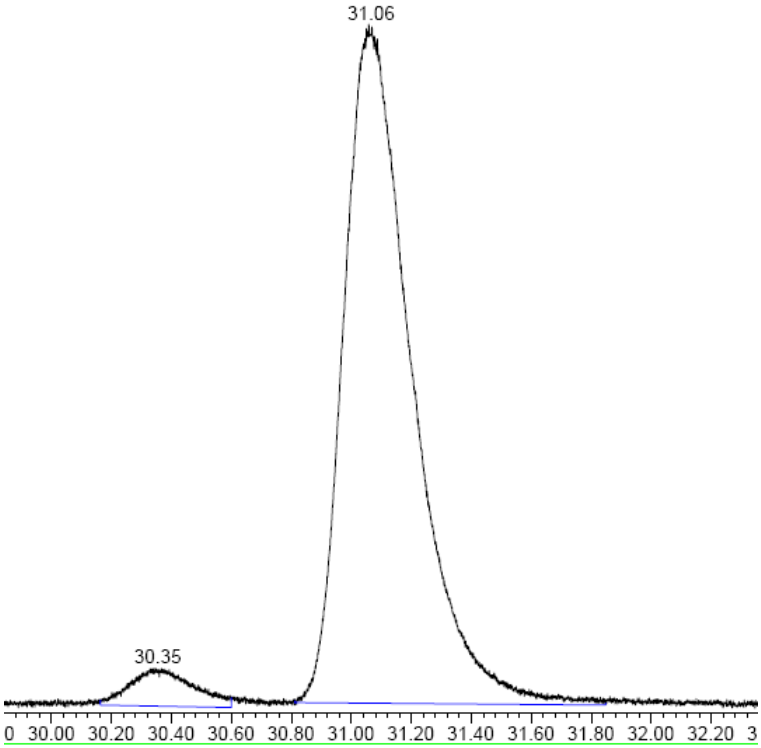
1h

```
File         :C:\MSDChem\2\DATA\GRA734.D
Operator     : DG
Acquired     : 21.04.2012 05:41:34 PM using AcqMethod F60-1.M
Instrument :    GC CHIRAL
Sample Name: GRA734
Misc Info   :
Vial Number: 1
```



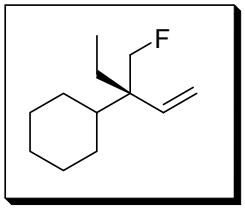

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA734.D
 Signal(s) : FID1A.CH
 Acq On    : 21.04.2012 05:41:34 PM
 Sample    : GRA734
 Misc      :
 ALS Vial  : 1   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	30.311	30.052	30.790	M	37469	6502717	92.70%	48.107%
2	31.071	30.816	31.953	M	37977	7014506	100.00%	51.893%
			Sum of corrected areas:			13517223		



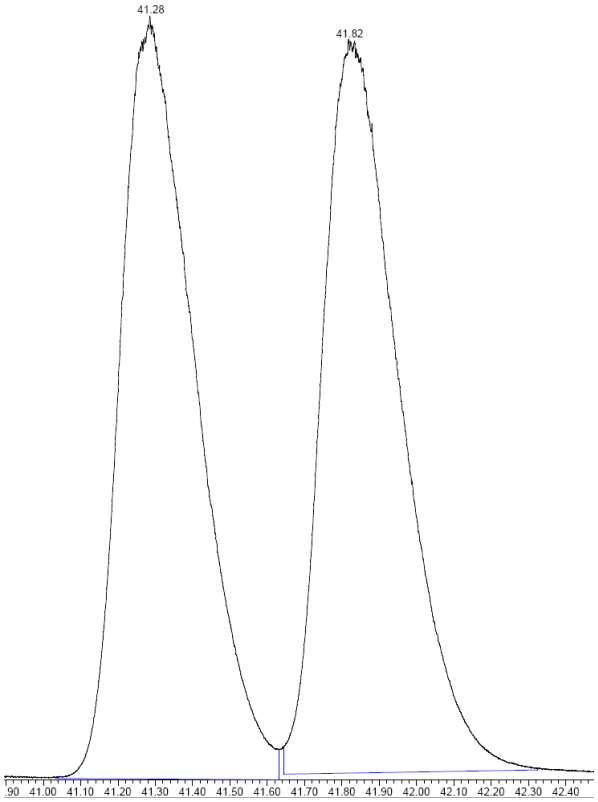

```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA748CAT.D
 Signal(s) : FID1A.CH
 Acq On    : 04.05.2012 06:25:40 PM
 Sample    : GRA748CAT
 Misc      :
 ALS Vial  : 4   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	30.346	30.163	30.600	M	2810	401026	5.09%	4.845%
2	31.058	30.811	31.847	M	50107	7876279	100.00%	95.155%
			Sum of corrected areas:			8277305		



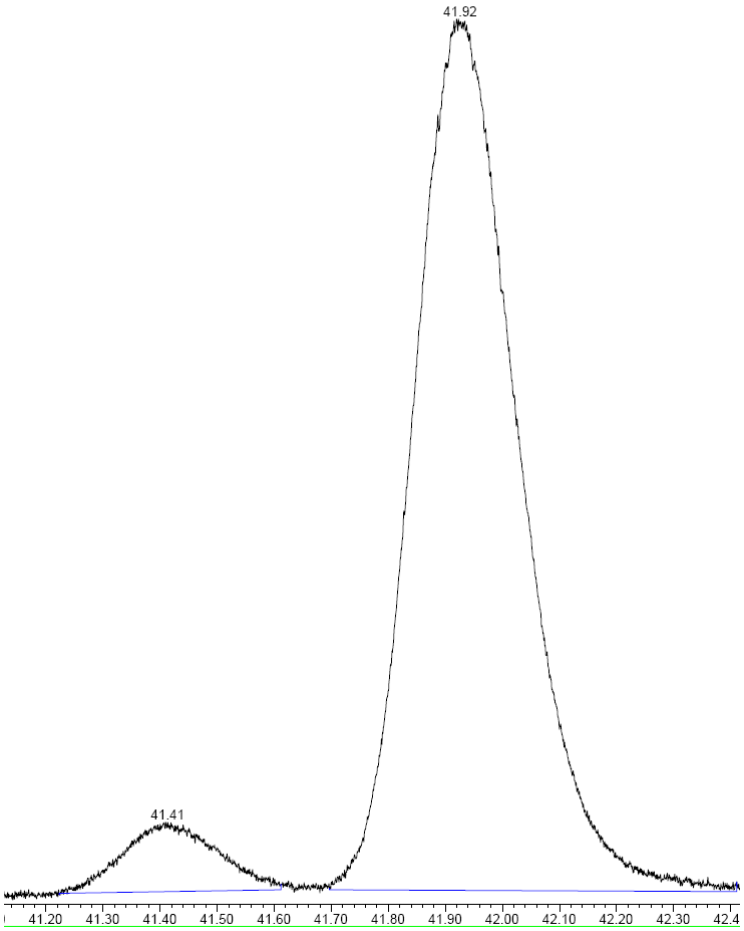
1i

```
File        :C:\MSDChem\2\DATA\GRA754RAC.D
Operator    : DG
Acquired    : 03.05.2012 01:04:09 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA754RAC
Misc Info   :
Vial Number: 1
```




```
 Data Path : C:\MSDChem\2\DATA\
 Data File : GRA754RAC.D
 Signal(s) : FID1A.CH
 Acq On    : 03.05.2012 01:04:09 PM
 Sample    : GRA754RAC
 Misc      :
 ALS Vial  : 1   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH

peak  R.T.   Start     End   PK  peak      peak     peak    % of
  #   min     min      min   TY height     area    % max.   total
---  -----   -----    ----  --- -------   -------  ------  ------
  1 41.285  41.034  41.631     M 292728  40567478 100.00% 50.128%
  2 41.817  41.643  42.324     M 281451  40360297  99.49% 49.872%
                    Sum of corrected areas:    80927775
```

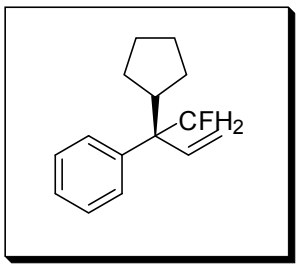
```
File       :C:\MSDChem\2\DATA\GRA756.D
Operator   : LH
Acquired   : 04.05.2012 08:23:30 PM using AcqMethod F60-1.M
Instrument :   GC CHIRAL
Sample Name: GRA756
Misc Info  :
Vial Number: 5
```



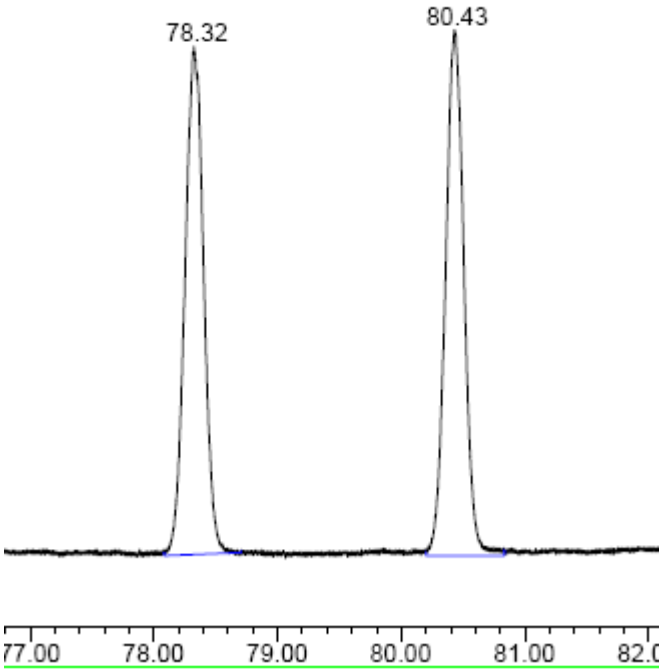

```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA756.D
Signal(s) : FID1A.CH
Acq On    : 04.05.2012 08:23:30 PM
Sample    : GRA756
Misc      :
ALS Vial  : 5   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	41.410	41.223	41.612	M	3517	396612	7.05%	6.585%
2	41.923	41.697	42.411	M	44131	5626450	100.00%	93.415%

Sum of corrected areas: 6023062

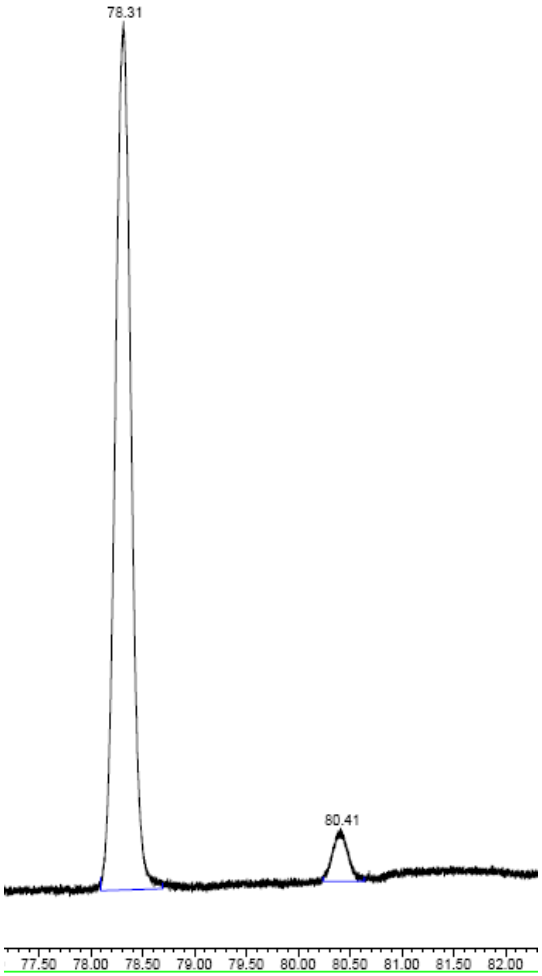


1d




```
Data Path : C:\MSDChem\2\DATA\
Data File : GRA697R.D
Signal(s) : FID1A.CH
Acq On    : 22.03.2012 08:07:55 PM
Sample    : GRA697R
Misc      :
ALS Vial  : 2   Sample Multiplier: 1

Integration File: events.e

Method    : C:\MSDCHEM\2\METHODS\F60-1.M
Title     :

Signal    : FID1A.CH
```

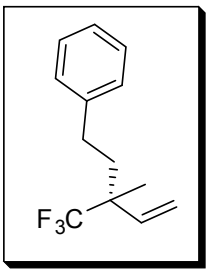
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	78.323	78.082	78.701	M	69412	7299932	97.79%	49.441%
2	80.428	80.200	80.831	M	71813	7465111	100.00%	50.559%
			Sum of corrected areas:			14765043		




```
 Data Path : C:\MSDChem\2\DATA\
 Data File : dga697.D
 Signal(s) : FID1A.CH
 Acq On    : 30.05.2012 06:01:32 PM
 Sample    : dga697
 Misc      :
 ALS Vial  : 6   Sample Multiplier: 1

 Integration File: events.e

 Method    : C:\MSDCHEM\2\METHODS\F60-1.M
 Title     :

 Signal    : FID1A.CH
```

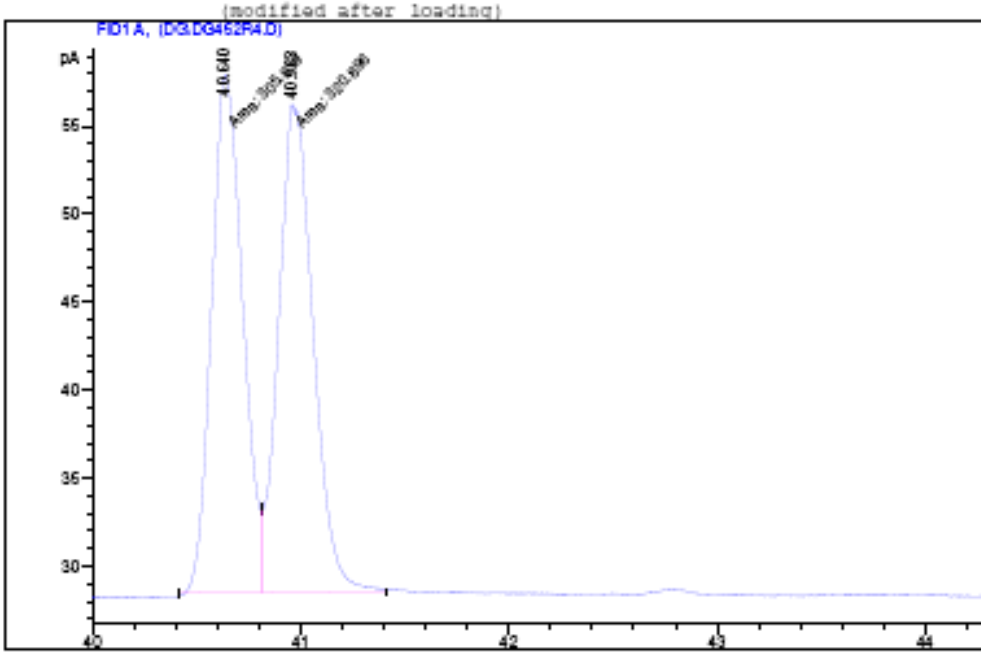
peak #	R.T. min	Start min	End min	PK TY	peak height	peak area	peak % max.	% of total
1	78.311	78.091	78.692	M	54488	5816899	100.00%	94.675%
2	80.406	80.238	80.633	M	3336	327183	5.62%	5.325%

Sum of corrected areas: 6144083



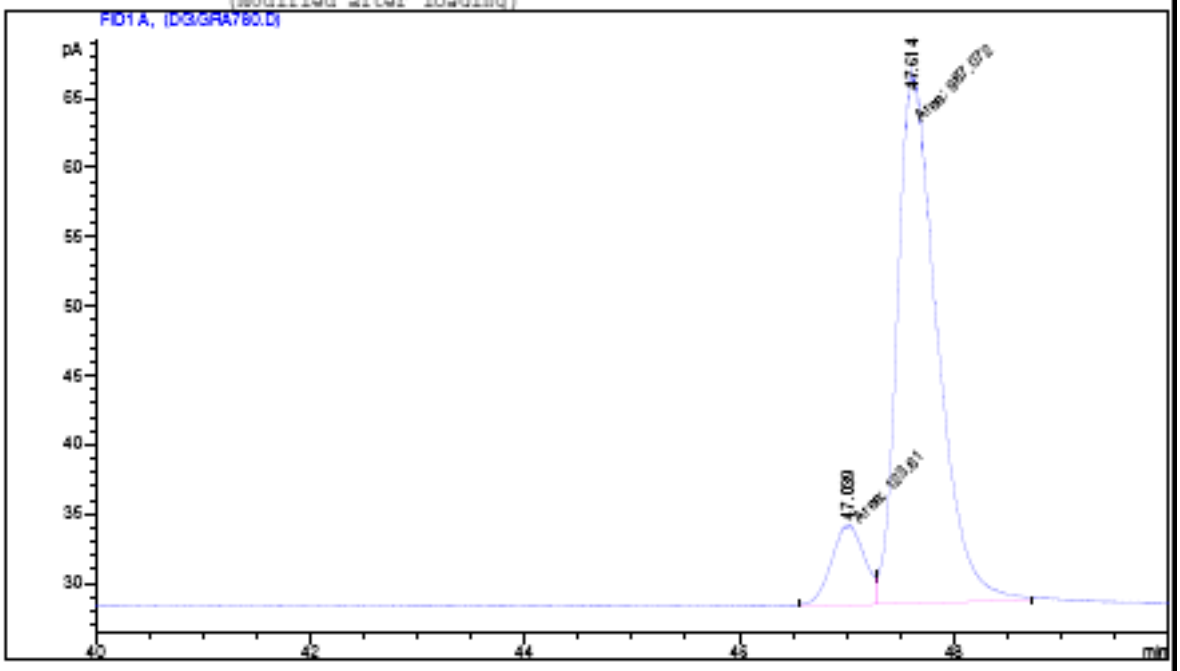
1n

```
Injection Date  : 14-Sep-11 1:56:46 PM
Sample Name     : DG452R4                              Location : Vial 3
Acq. Operator   : DG                                      Inj :   1
                                                      Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\3\METHODS\60-1.M
Last changed    : 14-Sep-11 2:49:32 PM by DG
                  (modified after loading)
Analysis Method : C:\HPCHEM\3\METHODS\60-1.M
Last changed    : 29-May-12 4:20:49 PM by DG
                  (modified after loading)
```

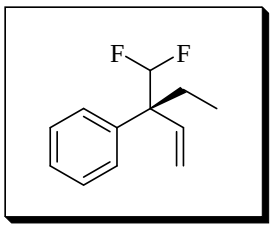



```
                          Area Percent Report

Sorted By             :      Signal
Calib. Data Modified  :      Wednesday, October 24, 2007 10:23:09 AM
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime  Type   Width     Area      Area     Name
  #   [min]           [min]   [pA*s]       %
----|-------|------|-------|---------|--------|--------------------
   1  23.349         0.0000    0.00000  0.00000
   2  24.031         0.0000    0.00000  0.00000
   3  40.640 MF      0.1735  305.95868 48.80855 ?
   4  40.963 FM      0.1930  320.89600 51.19145 ?
```

```
Injection Date  : 19-May-12 2:58:06 PM
Sample Name     : GRA780                              Location : Vial 4
Acq. Operator   : DG                                     Inj :   1
                                                   Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\3\METHODS\60-1.M
Last changed    : 19-May-12 2:41:03 PM by DG
                  (modified after loading)
Analysis Method : C:\HPCHEM\3\METHODS\60-1.M
Last changed    : 29-May-12 4:22:43 PM by DG
                  (modified after loading)
```



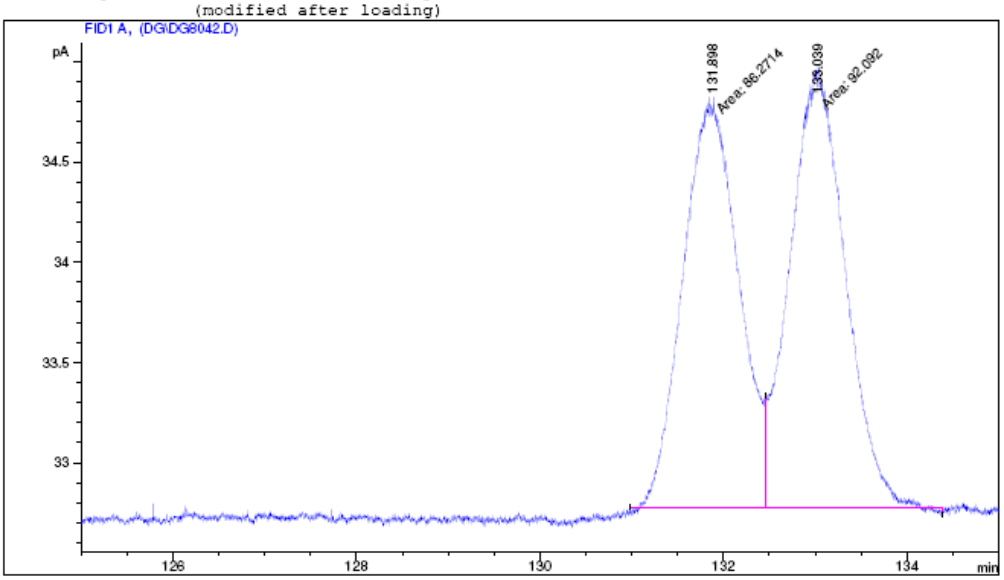

```
                          Area Percent Report

Sorted By             :      Signal
Calib. Data Modified  :      Wednesday, October 24, 2007 10:23:09 AM
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime  Type   Width     Area      Area     Name
  #   [min]          [min]   [pA*s]       %
----|-------|------|-------|---------|--------|-------------------
   1  23.349        0.0000    0.00000  0.00000
   2  24.031        0.0000    0.00000  0.00000
   3  47.039 MF     0.3575  123.61034 11.12923 ?
   4  47.614 FM     0.4228  987.07178 88.87077 ?
```



1o

Data File C:\HPCHEM\3\DATA\DG\DG8042.D Sample Name: DG8042

```
Injection Date  : 07-Jun-12 7:03:55 PM
Sample Name     : DG8042                          Location : Vial 3
Acq. Operator   : DG                                 Inj :   1
                                                 Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\3\METHODS\60-100.M
Last changed    : 07-Jun-12 7:01:32 PM by LH
                  (modified after loading)
Analysis Method : C:\HPCHEM\3\METHODS\50-30.M
Last changed    : 16-Jun-12 2:38:14 PM by NG
                  (modified after loading)
```

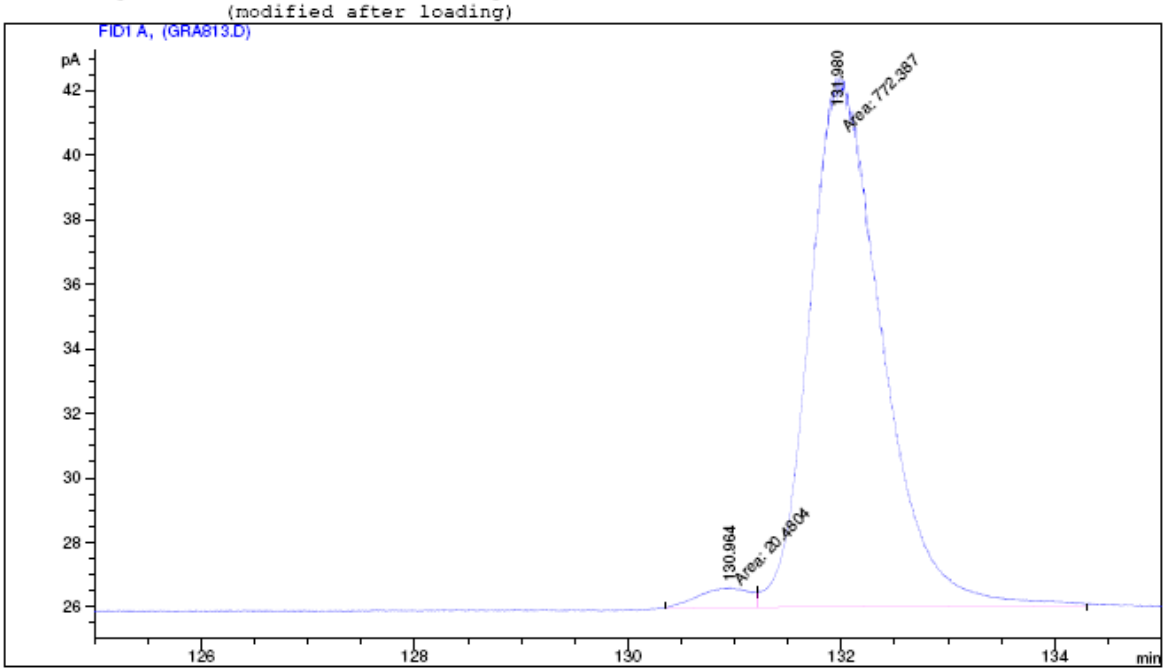



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1 131.898 MF    0.6991   86.27142    2.05671 48.36833
   2 133.039 FM    0.6983   92.09201    2.19805 51.63167

Totals :                   178.36343    4.25476

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

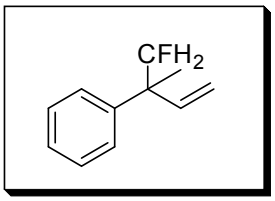
Instrument 3 16-Jun-12 2:42:13 PM NG Page 1 of 1

Data File C:\HPCHEM\3\DATA\GRA813.D Sample Name: GRA813

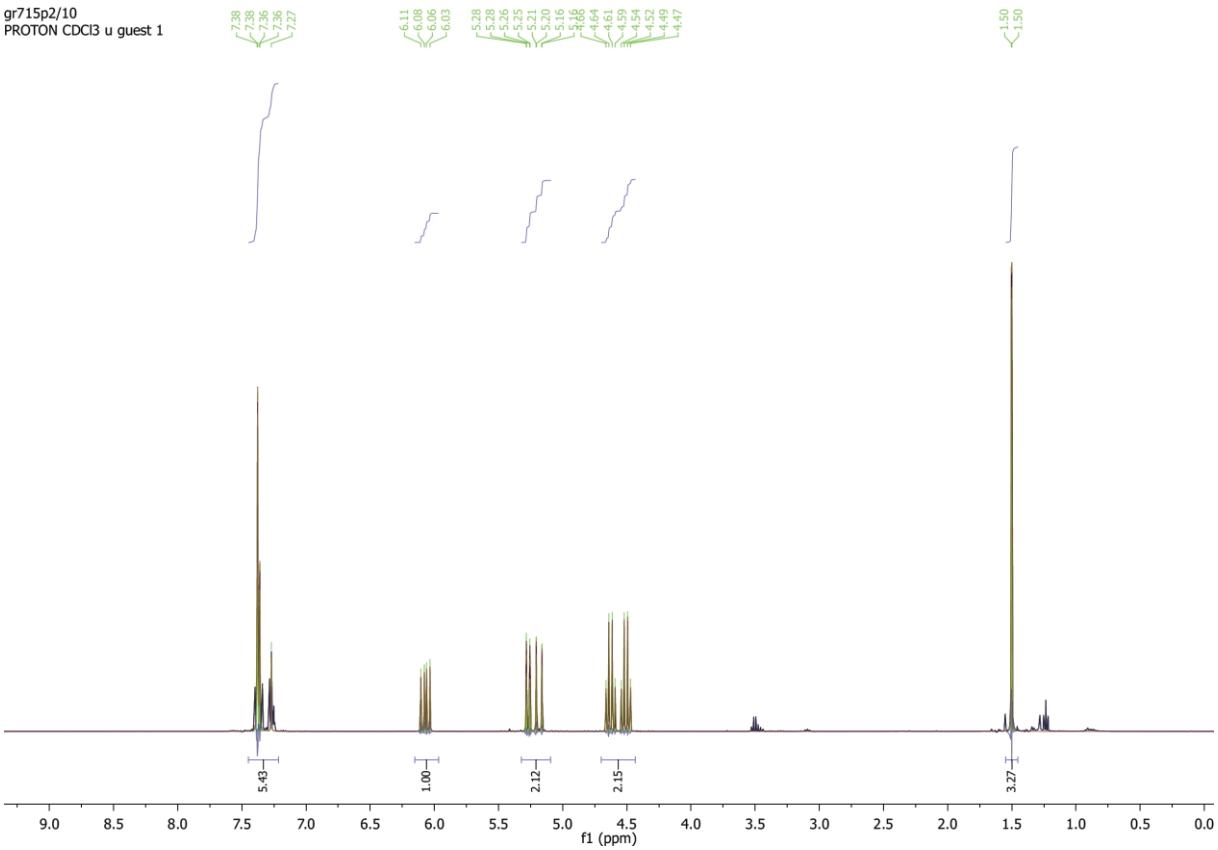
```
Injection Date  : 12-Jun-12 5:16:35 PM          Seq. Line :   2
Sample Name     : GRA813                        Location : Vial 3
Acq. Operator   : DG                               Inj :   1
                                                Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\3\METHODS\60-120.M
Last changed    : 11-Jun-12 4:13:50 PM by MT
Analysis Method : C:\HPCHEM\3\METHODS\50-30.M
Last changed    : 16-Jun-12 2:38:14 PM by NG
                  (modified after loading)
```

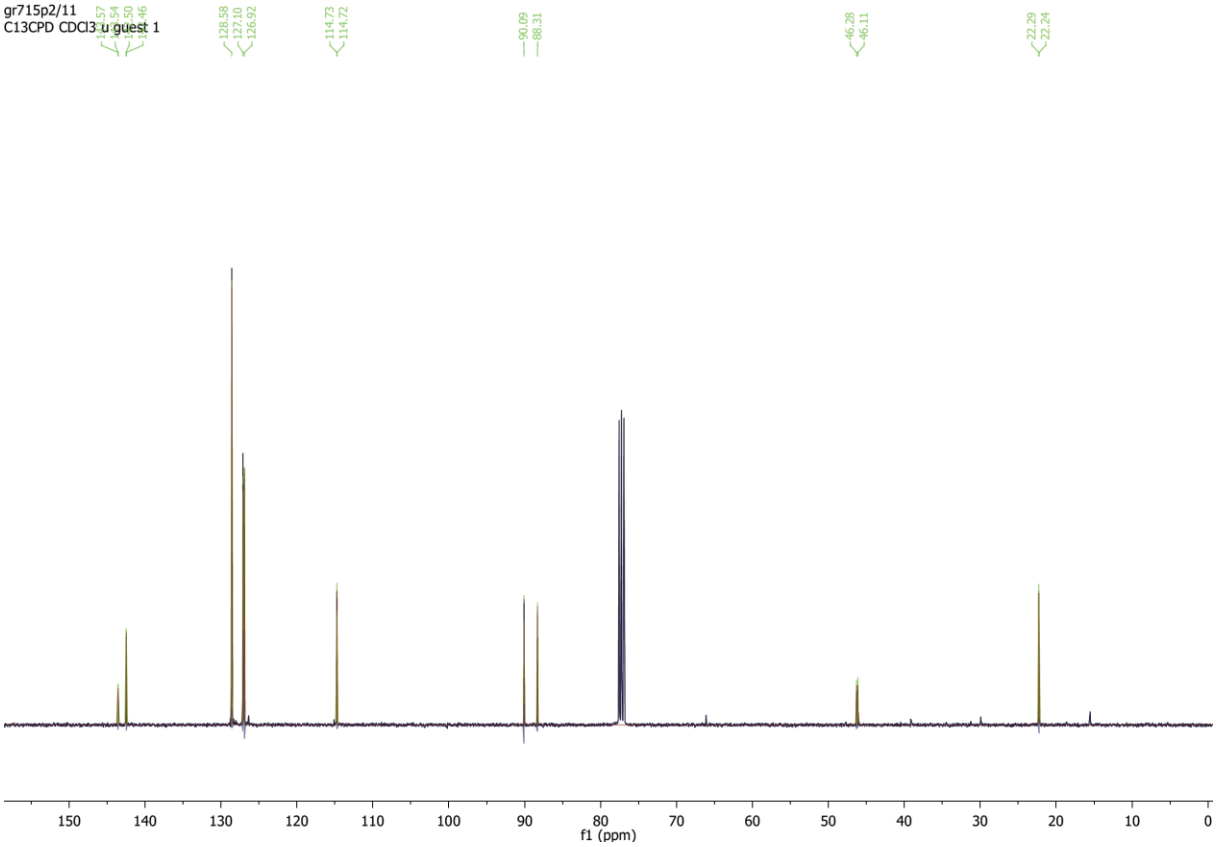


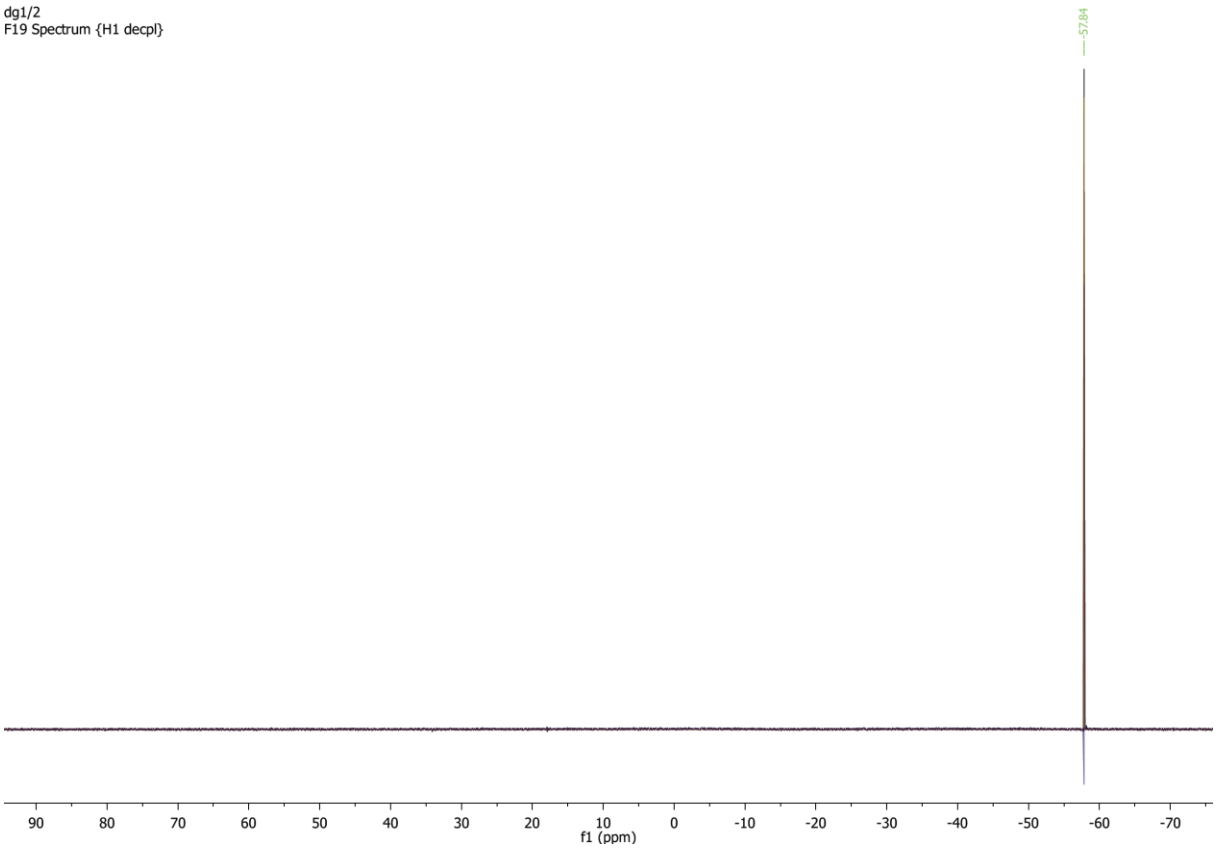

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1 130.964 MF    0.5512   20.48037 6.19291e-1  2.58308
   2 131.980 FM    0.7833  772.38678   16.43530 97.41692

Totals :                   792.86715   17.05459

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

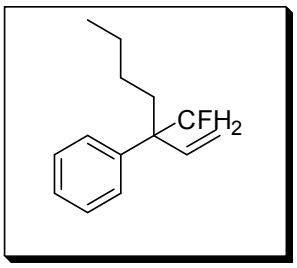

CFH2

1a

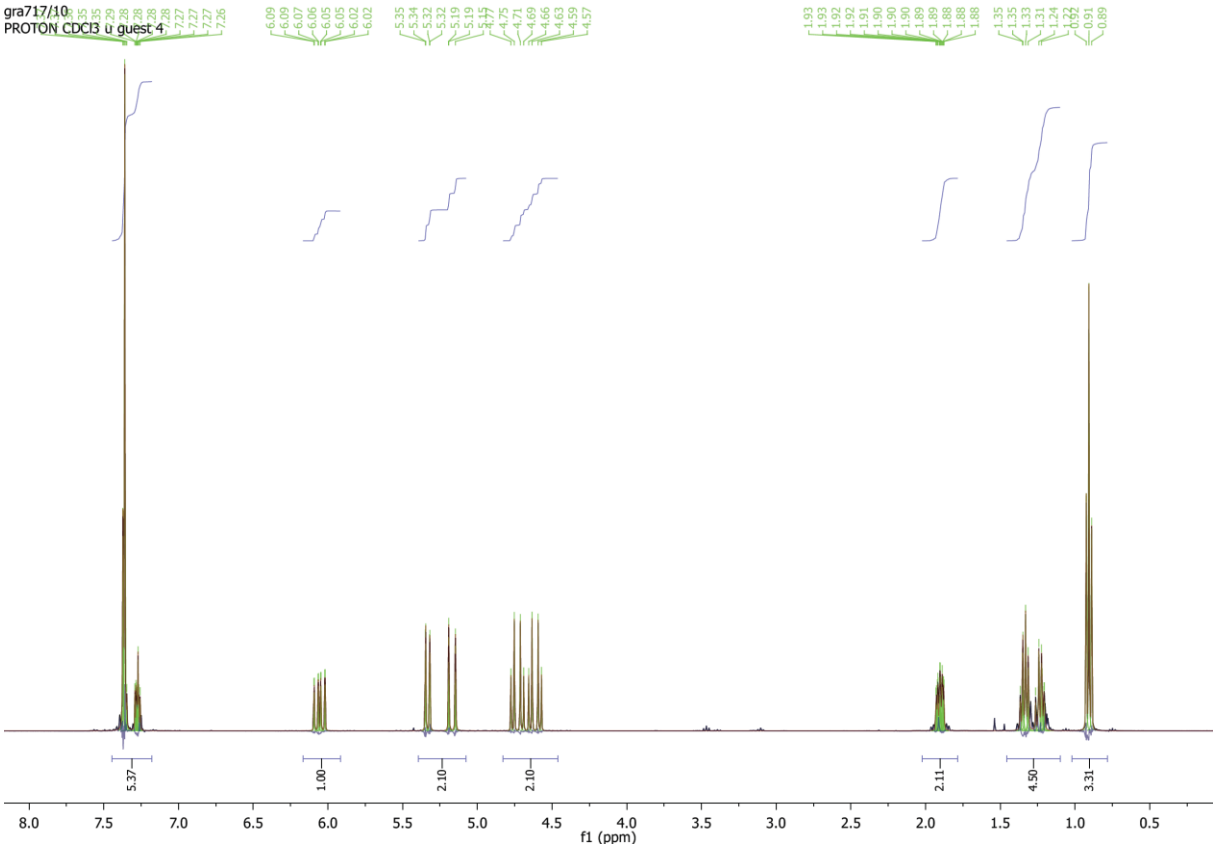

gr715p2/10
PROTON CDCl3 u guest 1
7.38
7.38
7.36
7.36
7.36
7.27
6.11
6.08
6.06
6.03
5.28
5.28
5.26
5.25
5.21
5.20
5.16
4.64
4.61
4.59
4.54
4.52
4.49
4.47
1.50
1.50
5.43
1.00
2.12
2.15
3.27
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

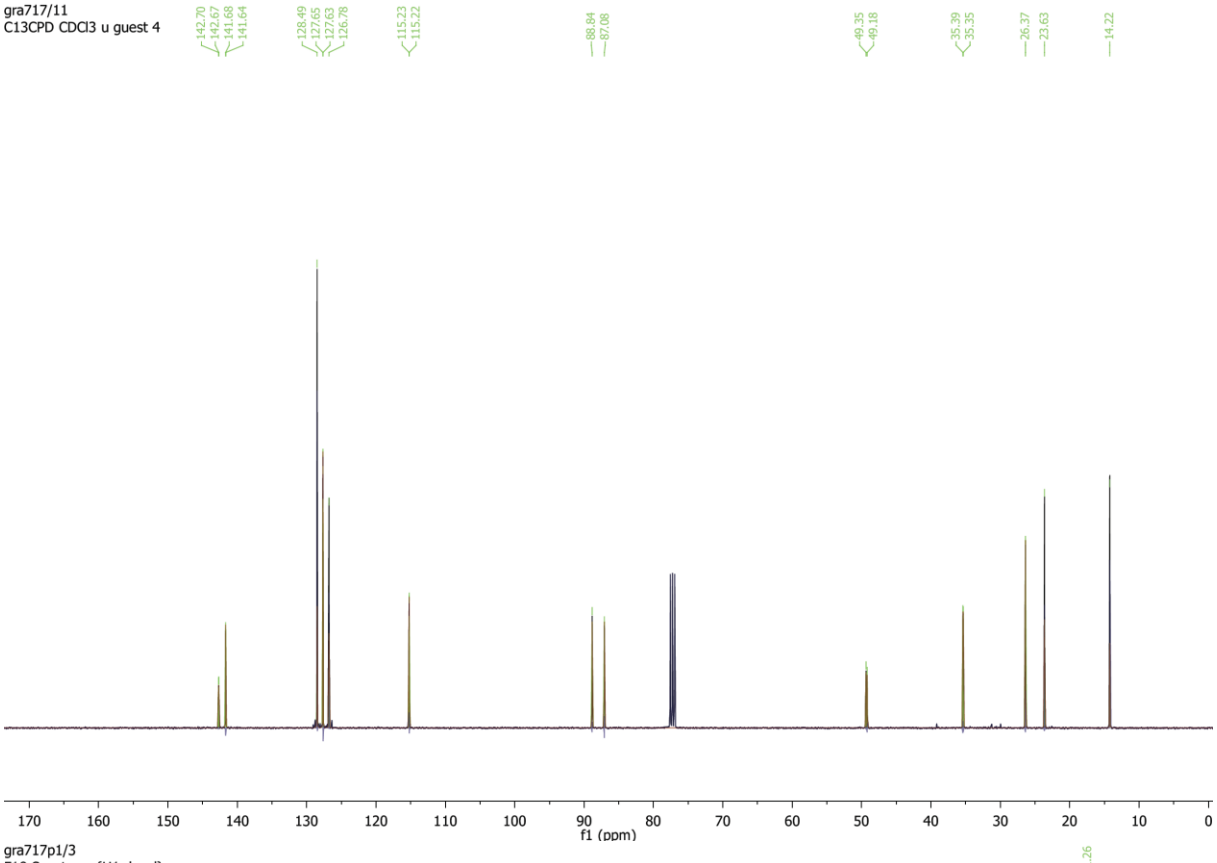

gr715p2/11
C13CPD CDCl3 u guest 1
128.58
127.10
126.92
114.73
114.72
90.09
88.31
46.28
46.11
22.29
22.24
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

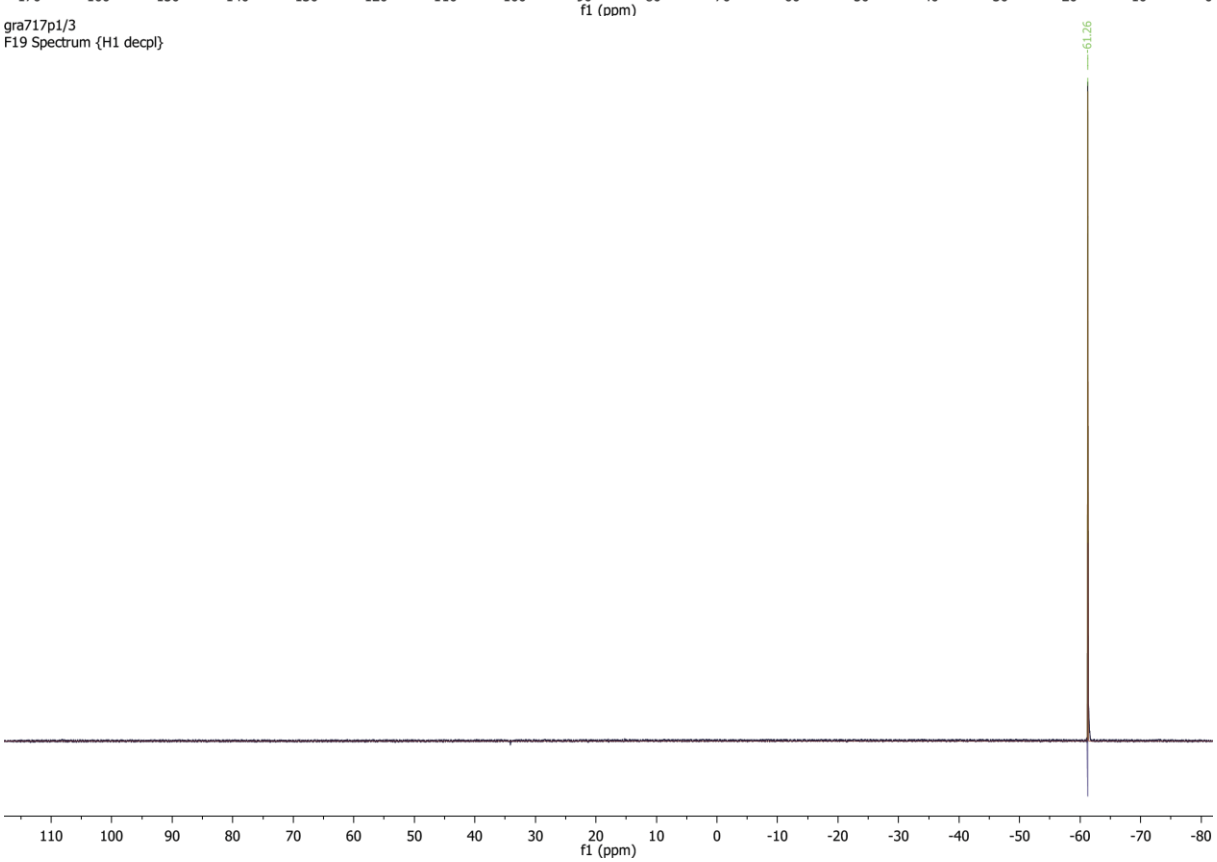

dg1/2
F19 Spectrum {H1 decpl}
-57.84
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70
f1 (ppm)

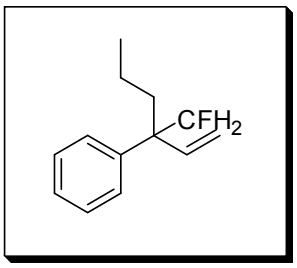

CFH2

1c

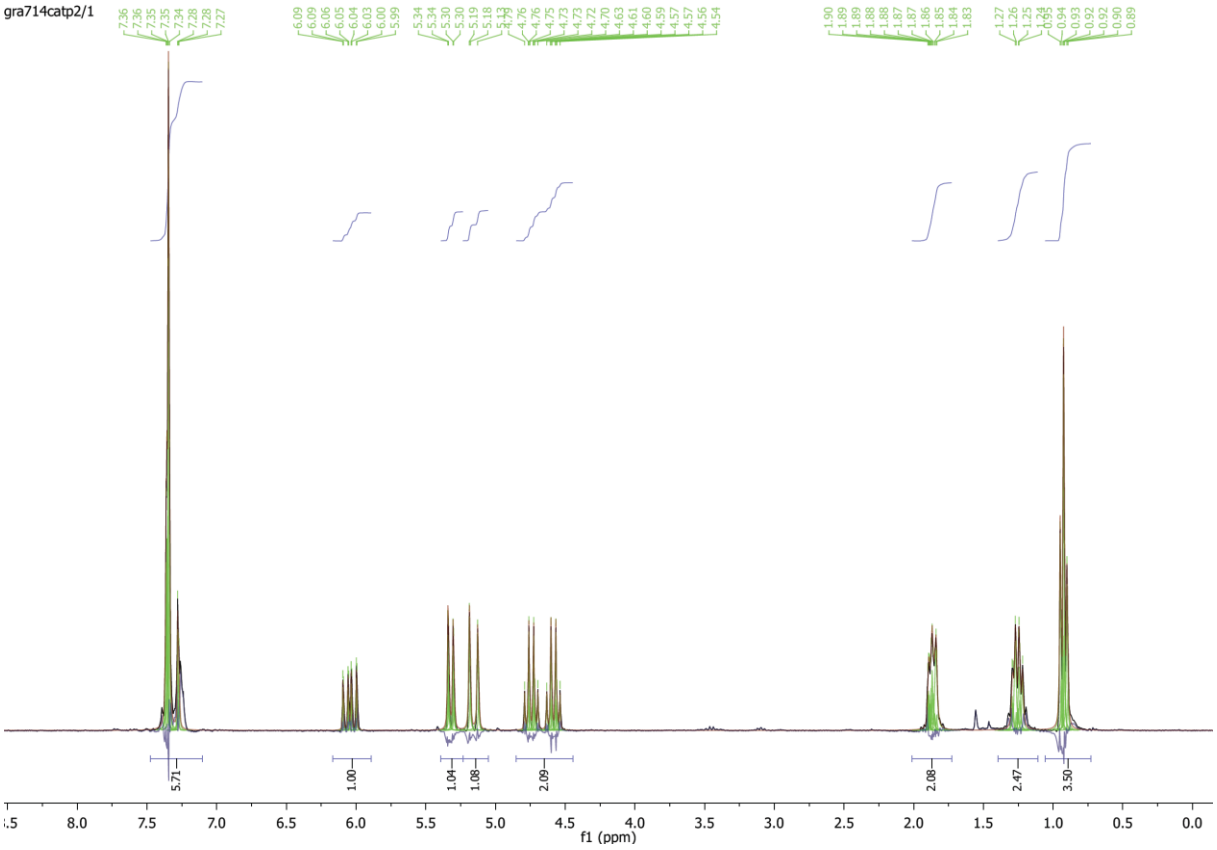

gra717/10
PROTON CDCl3 u guest 4
5.37
1.00
2.10
2.10
2.11
4.50
3.31
f1 (ppm)

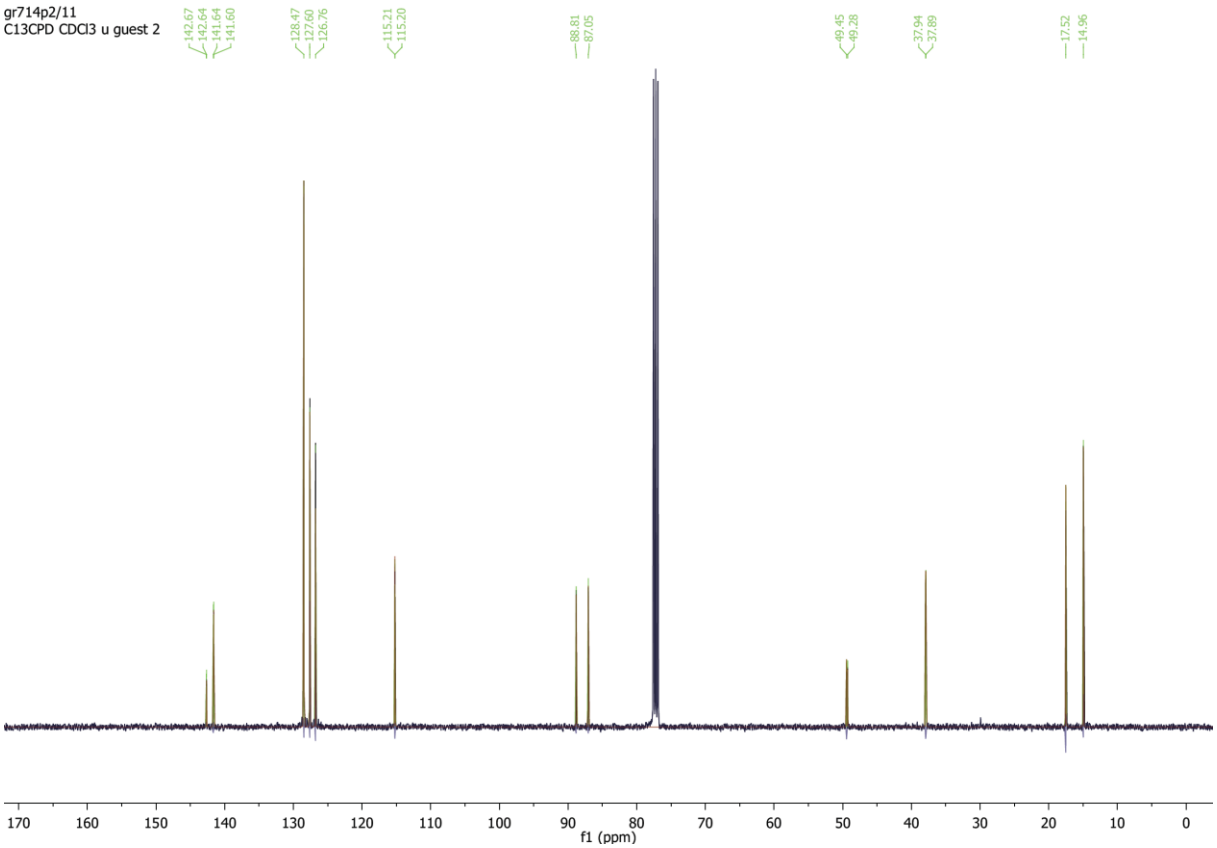

gra717/11
C13CPD CDCl3 u guest 4
142.70
142.67
141.68
141.64
128.49
127.65
127.63
126.78
115.23
115.22
88.84
87.08
49.35
49.18
35.39
35.35
26.37
23.63
14.22
f1 (ppm)

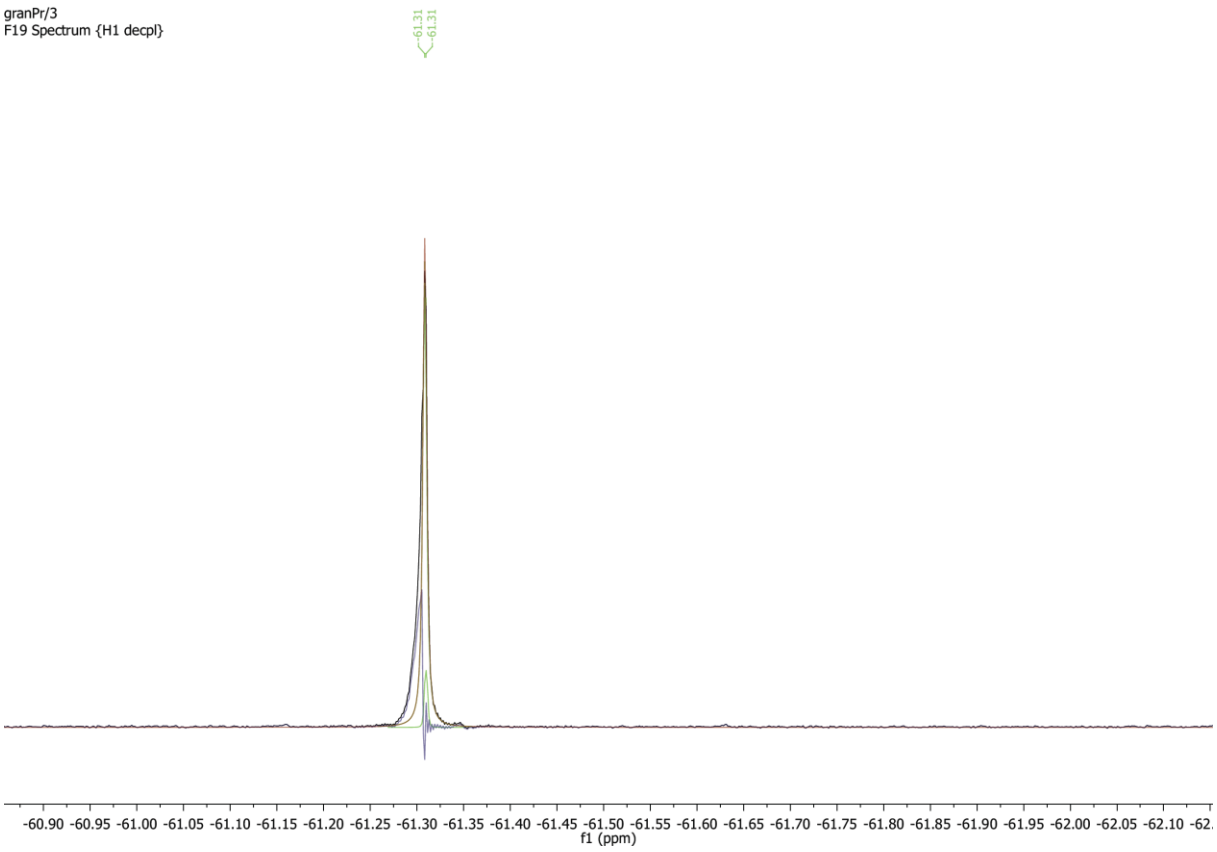

gra717p1/3
F19 Spectrum {H1 decpl}
-61.26
f1 (ppm)

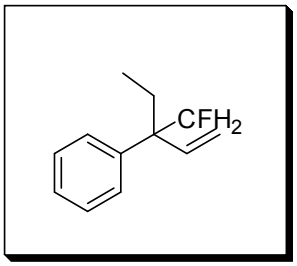


1b

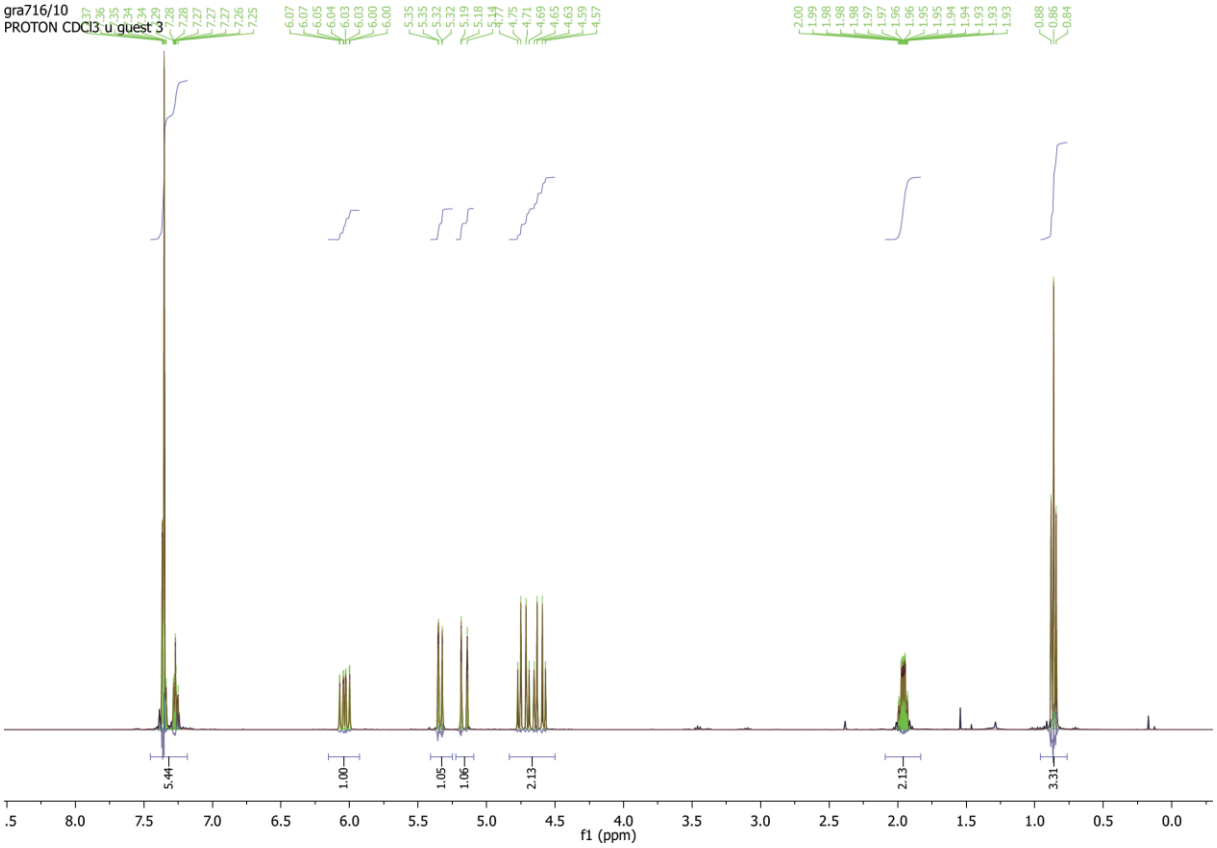


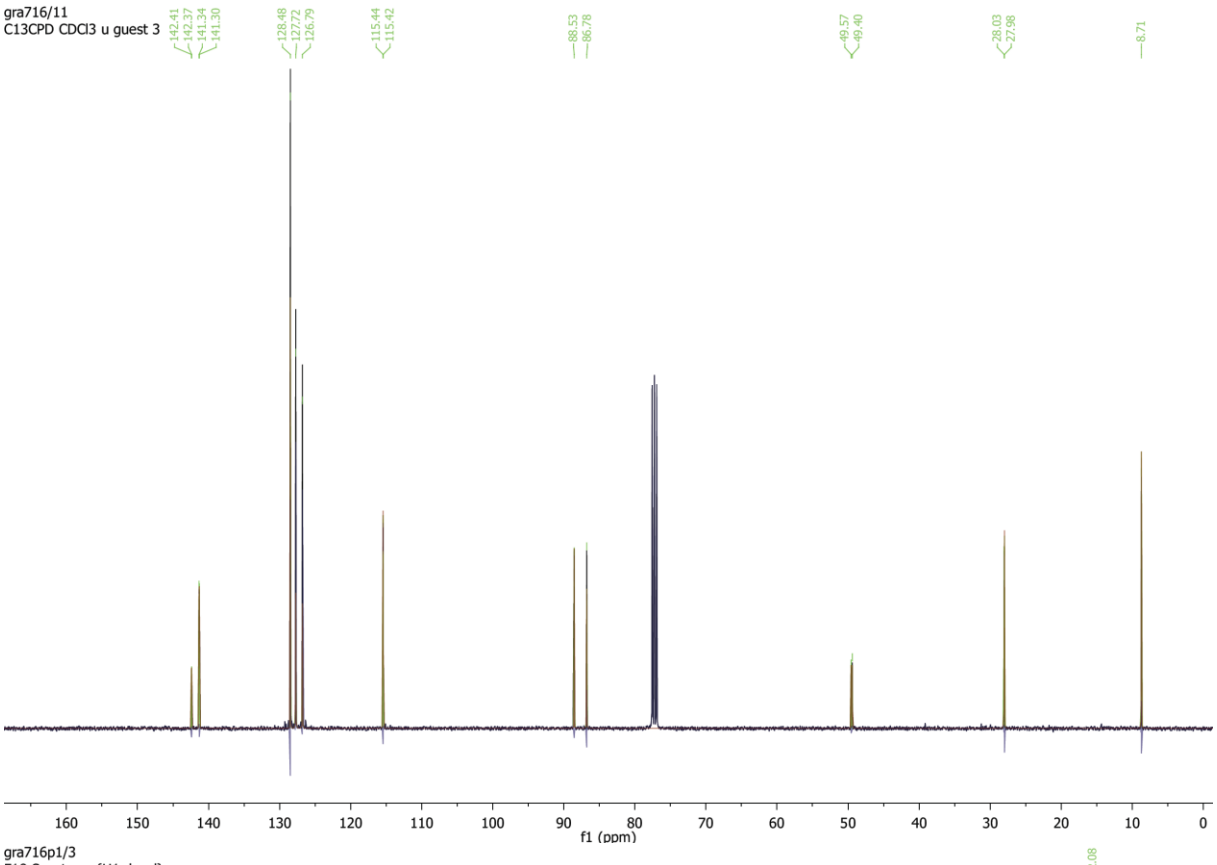

gr714p2/11
C13CPD CDCl3 u guest 2
142.67
142.64
141.64
141.60
128.47
127.60
126.76
115.21
115.20
88.81
87.05
49.45
49.28
37.94
37.89
17.52
14.96
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

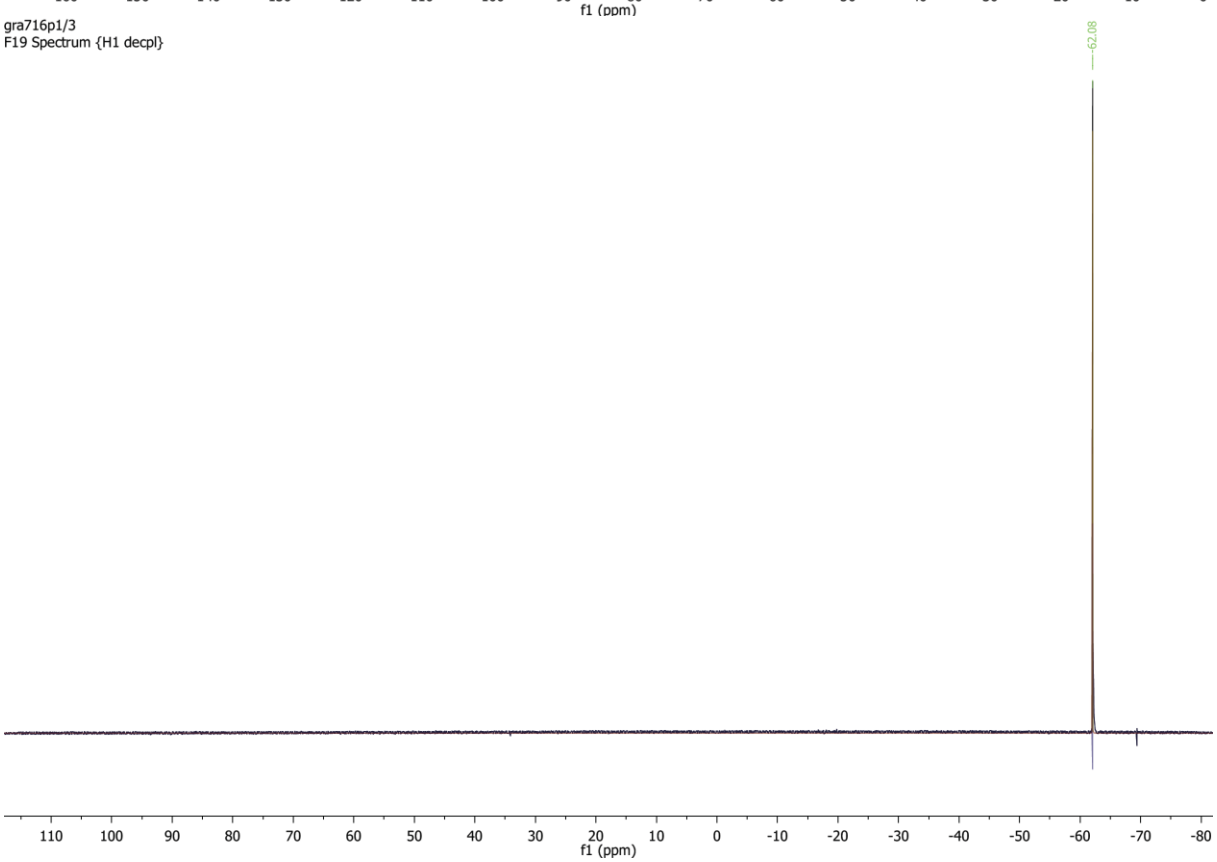

granPr/3
F19 Spectrum {H1 decpl}
-61.31
-61.31
-60.90 -60.95 -61.00 -61.05 -61.10 -61.15 -61.20 -61.25 -61.30 -61.35 -61.40 -61.45 -61.50 -61.55 -61.60 -61.65 -61.70 -61.75 -61.80 -61.85 -61.90 -61.95 -62.00 -62.05 -62.10 -62.
f1 (ppm)

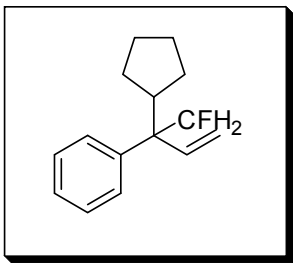

CFH2

1e

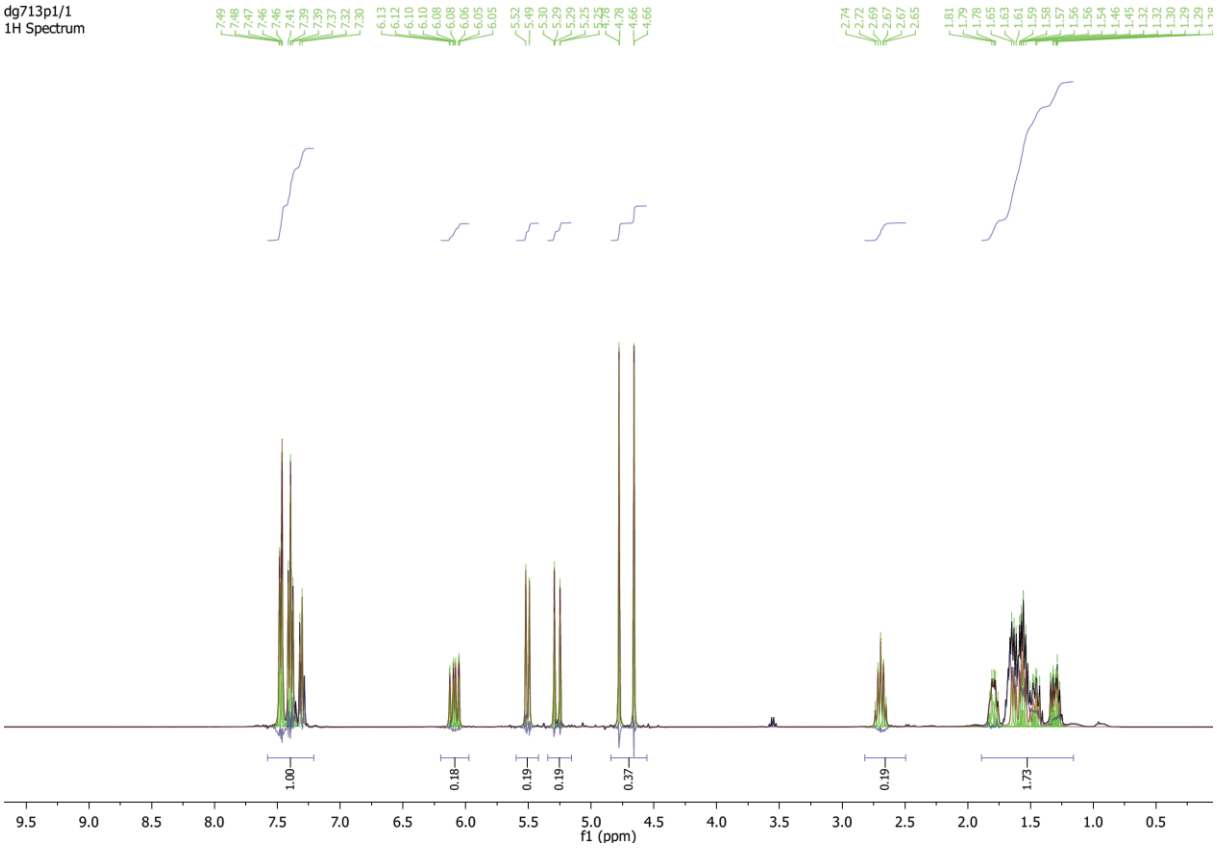

gra716/10
PROTON CDCl3 u guest 3
5.44
1.00
1.05
1.06
2.13
2.13
3.31
f1 (ppm)

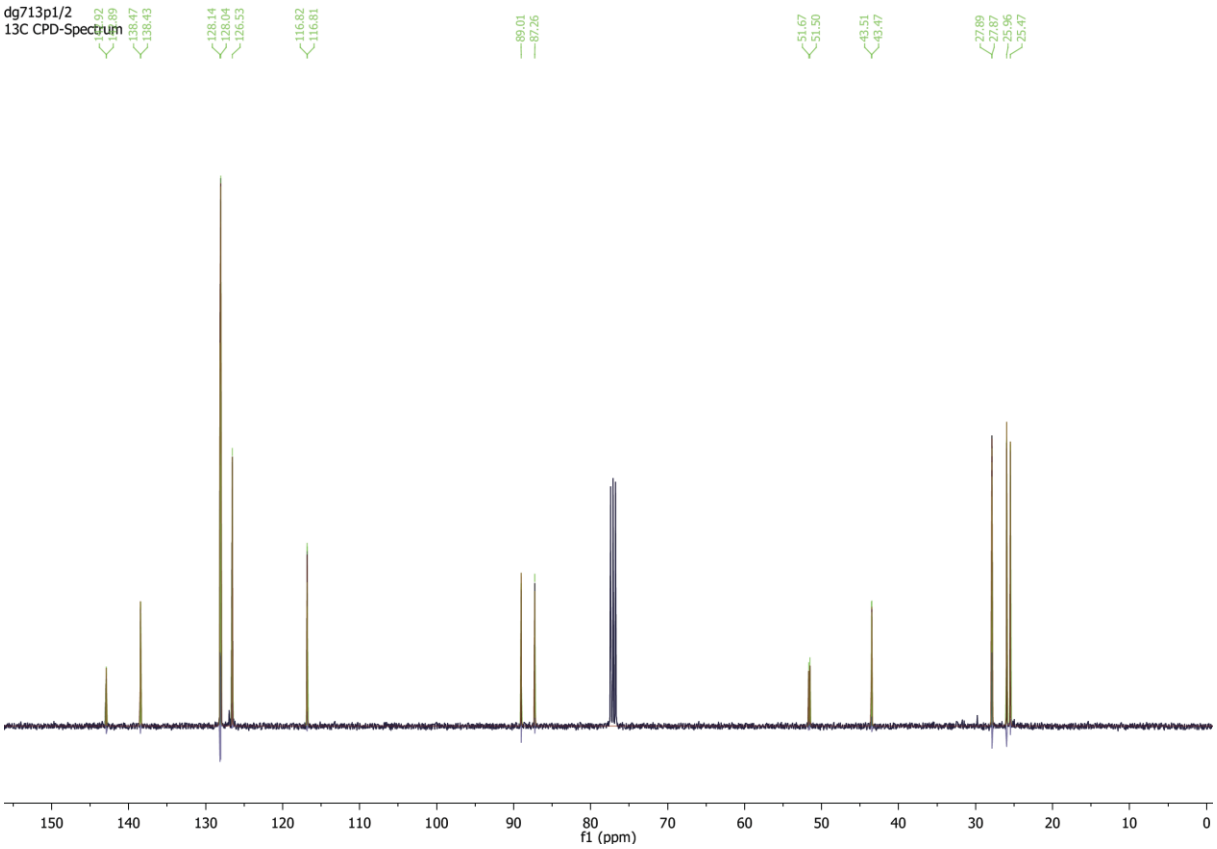

gra716/11
C13CPD CDCl3 u guest 3
142.41
142.37
141.34
141.30
128.48
127.72
126.79
115.44
115.42
88.53
86.78
49.57
49.40
28.03
27.98
8.71
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

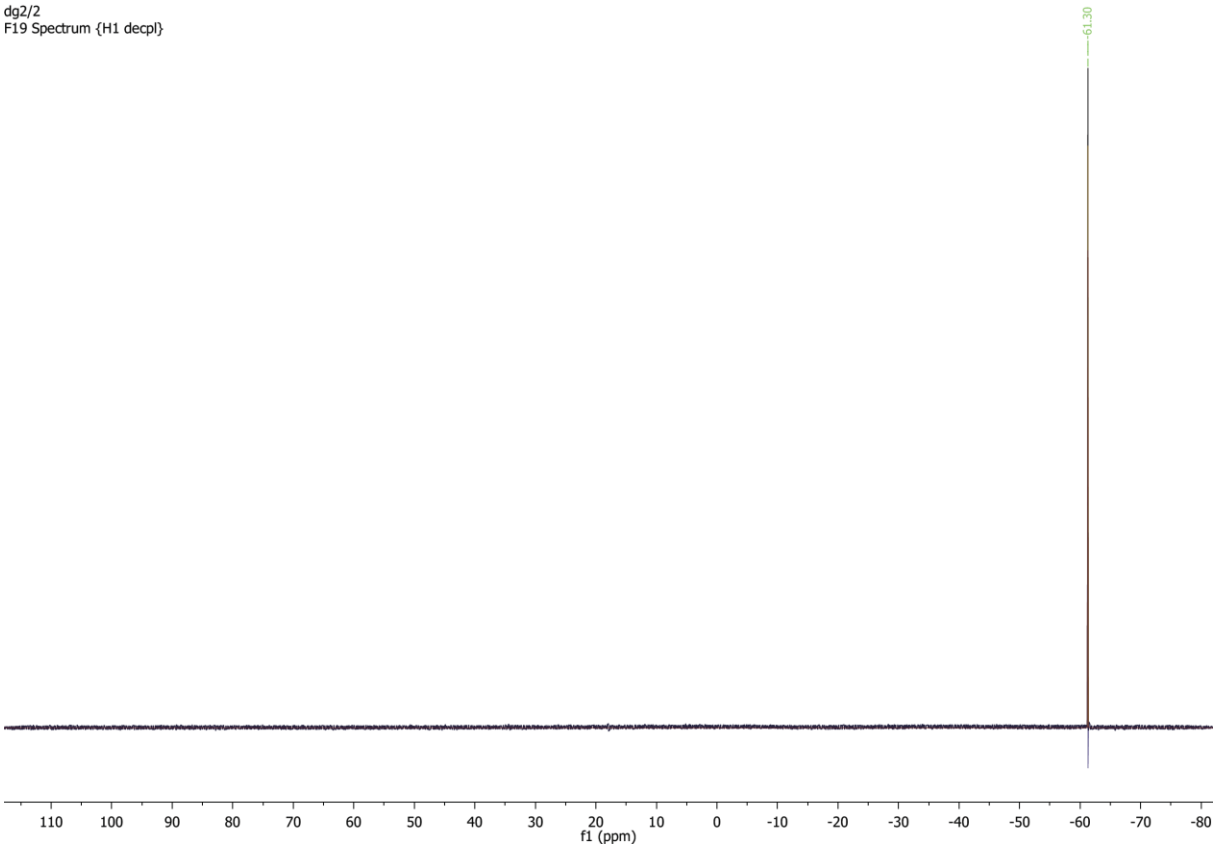

gra716p1/3
F19 Spectrum {H1 decpl}
-62.08
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

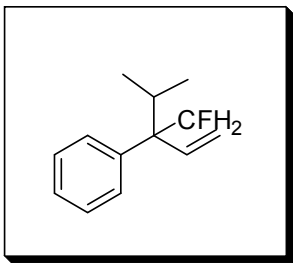


1d

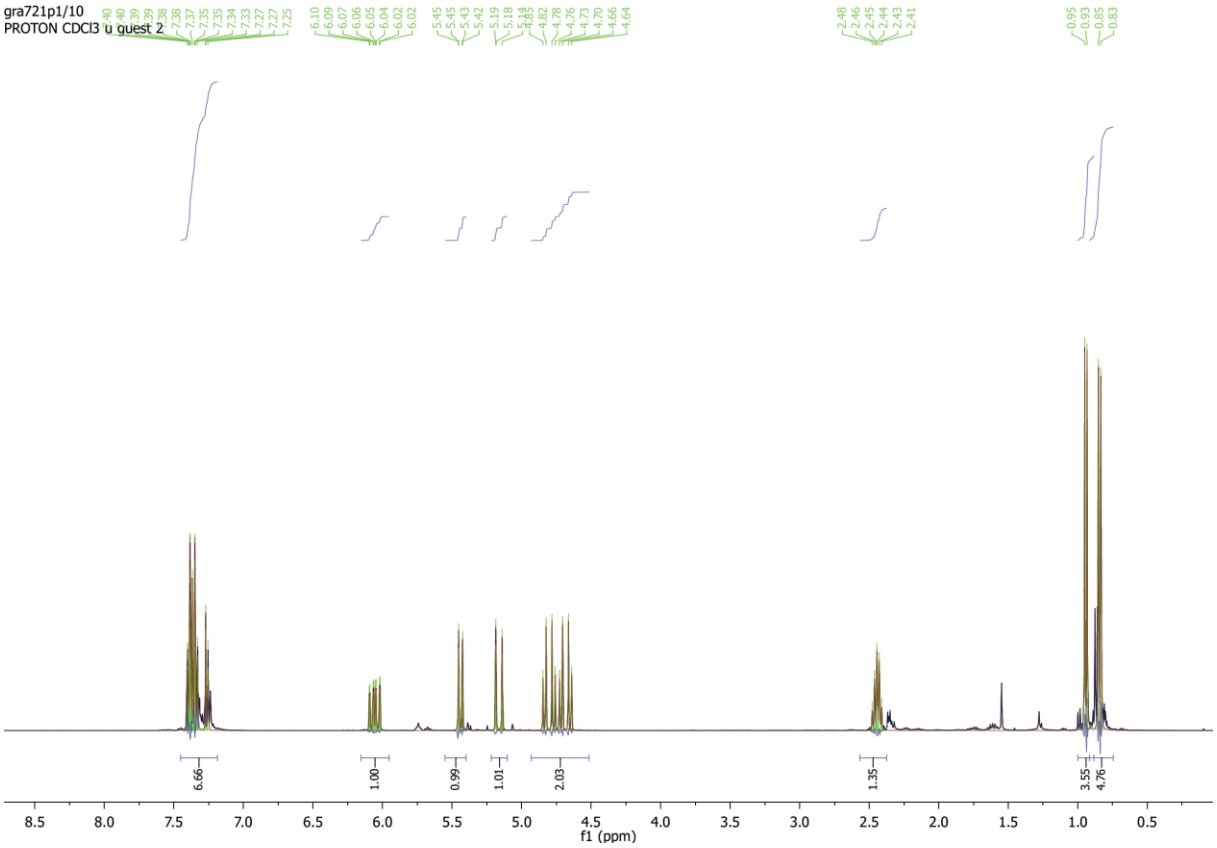


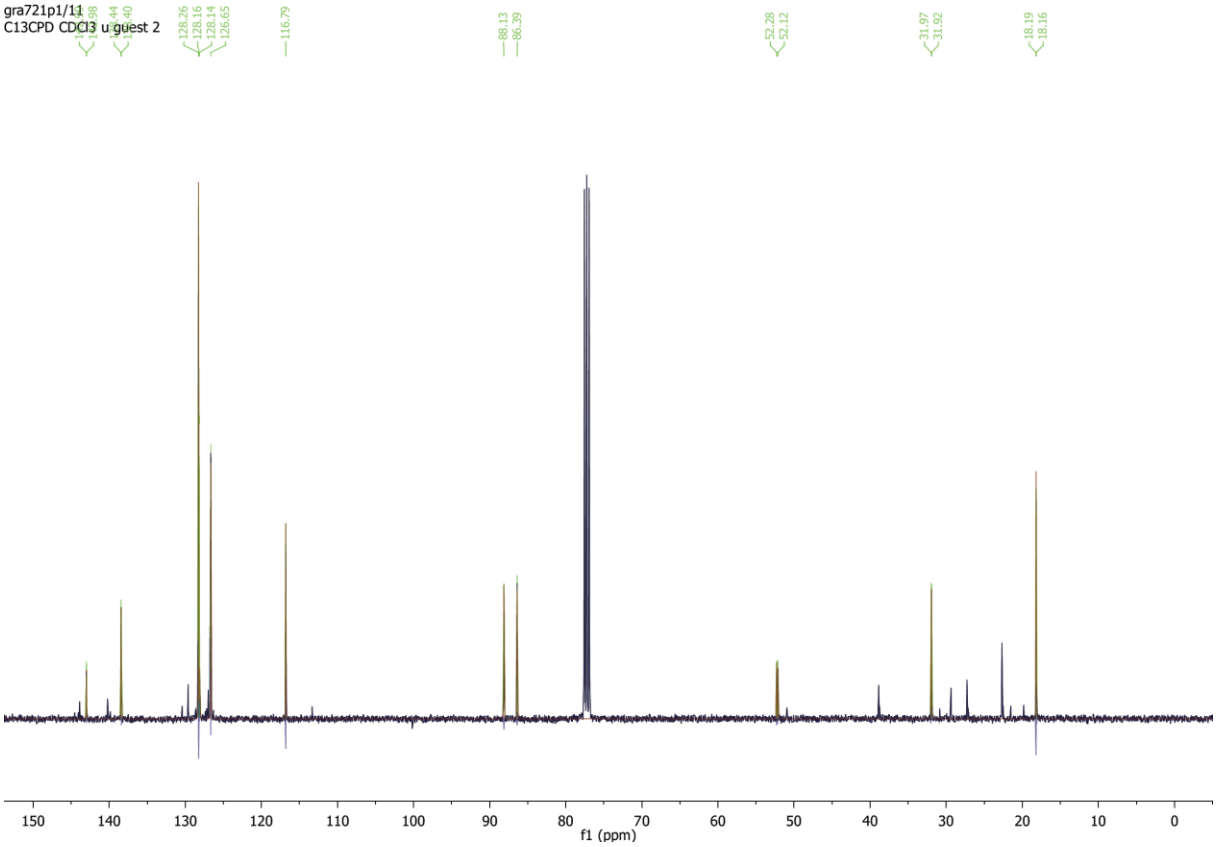

dg713p1/2
13C CPD-Spectrum
139.92
139.89
138.47
138.43
128.14
128.04
126.53
116.82
116.81
89.01
87.26
51.67
51.50
43.51
43.47
27.89
27.87
25.96
25.47
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

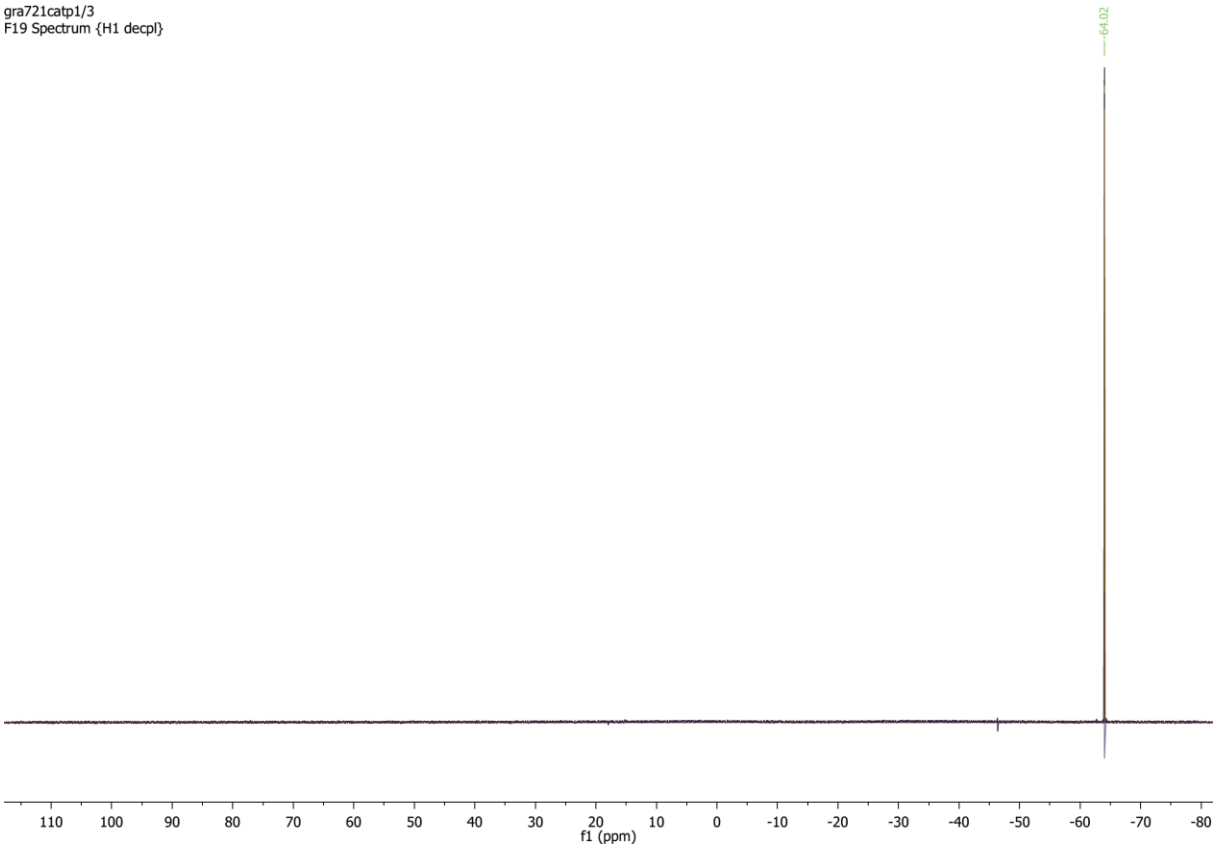

dg2/2
F19 Spectrum {H1 decpl}
-61.30
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

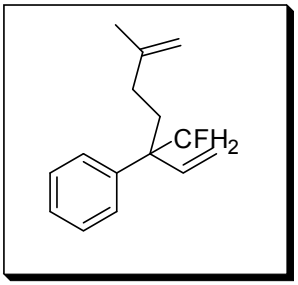

CFH2

1f

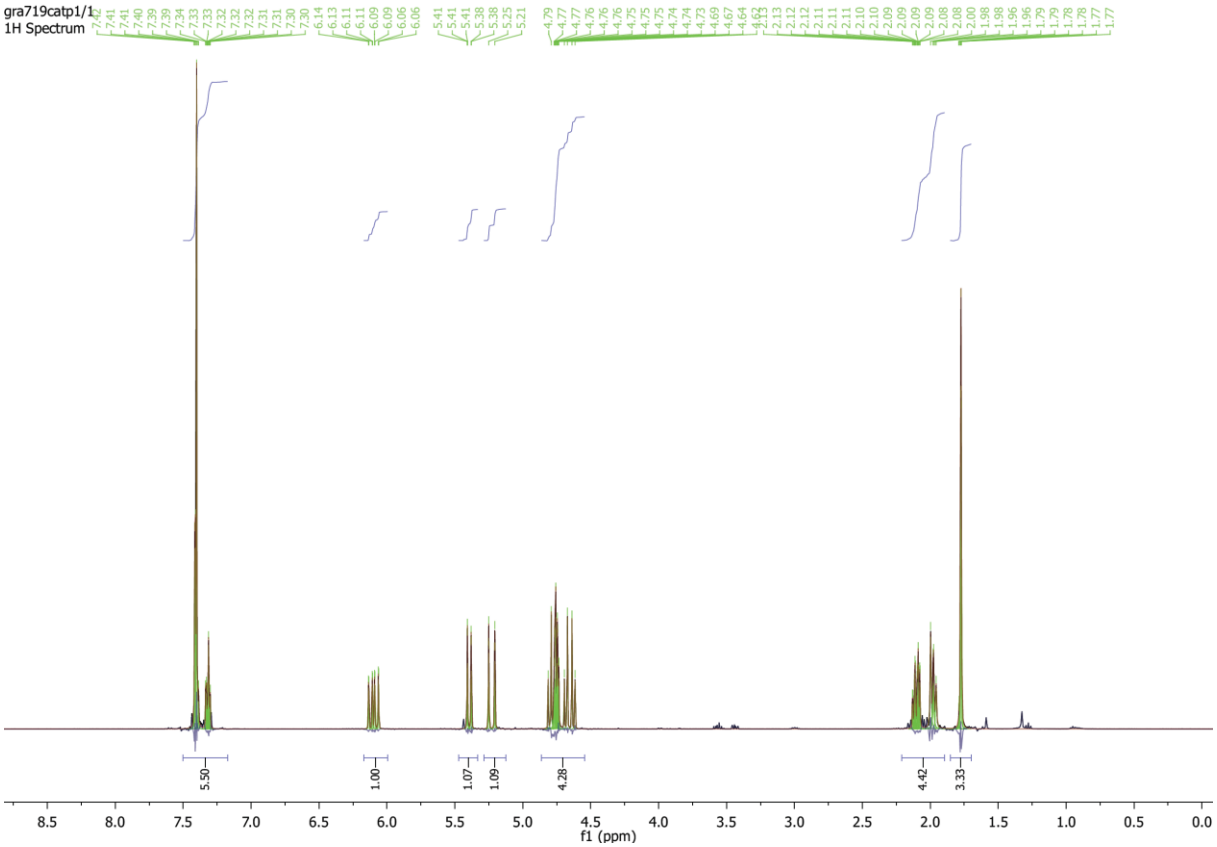

gra721p1/10
PROTON CDCl3 u guest 2
6.66
1.00
0.99
1.01
2.03
1.35
3.55
4.76
f1 (ppm)

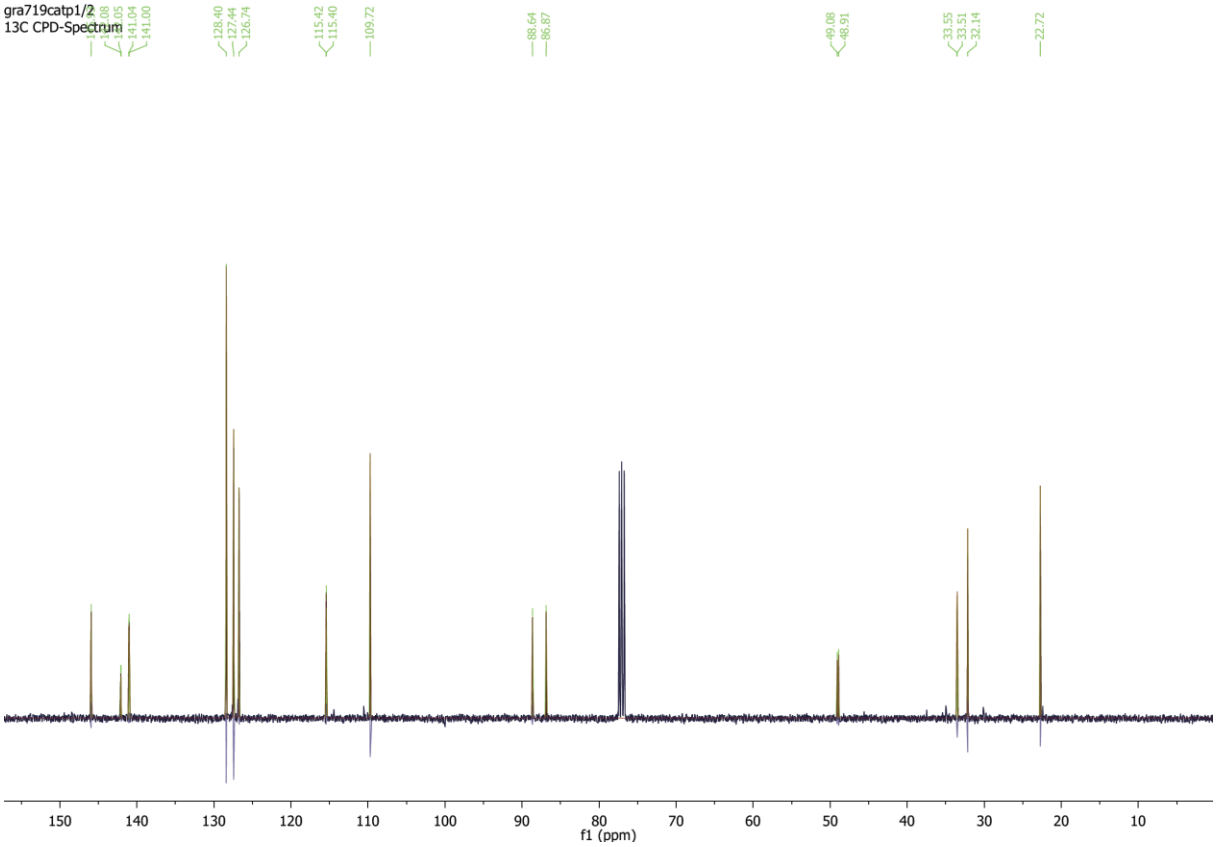

gra721p1/11
C13CPD CDCl3 u guest 2
128.26
128.16
128.14
126.65
116.79
88.13
86.39
52.28
52.12
31.97
31.92
18.19
18.16
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

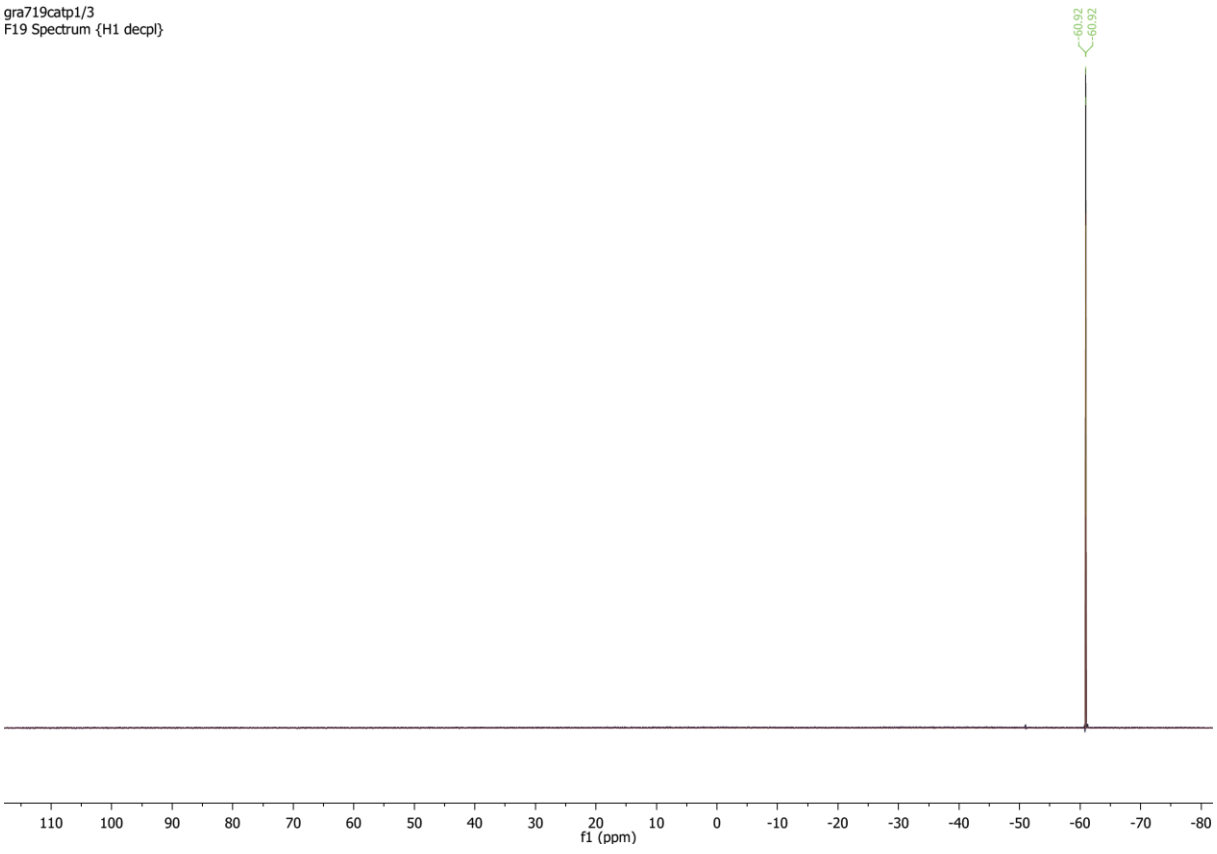

gra721catp1/3
F19 Spectrum {H1 decpl}
-64.02
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

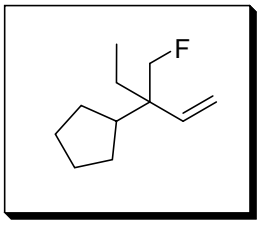

CFH2

1g

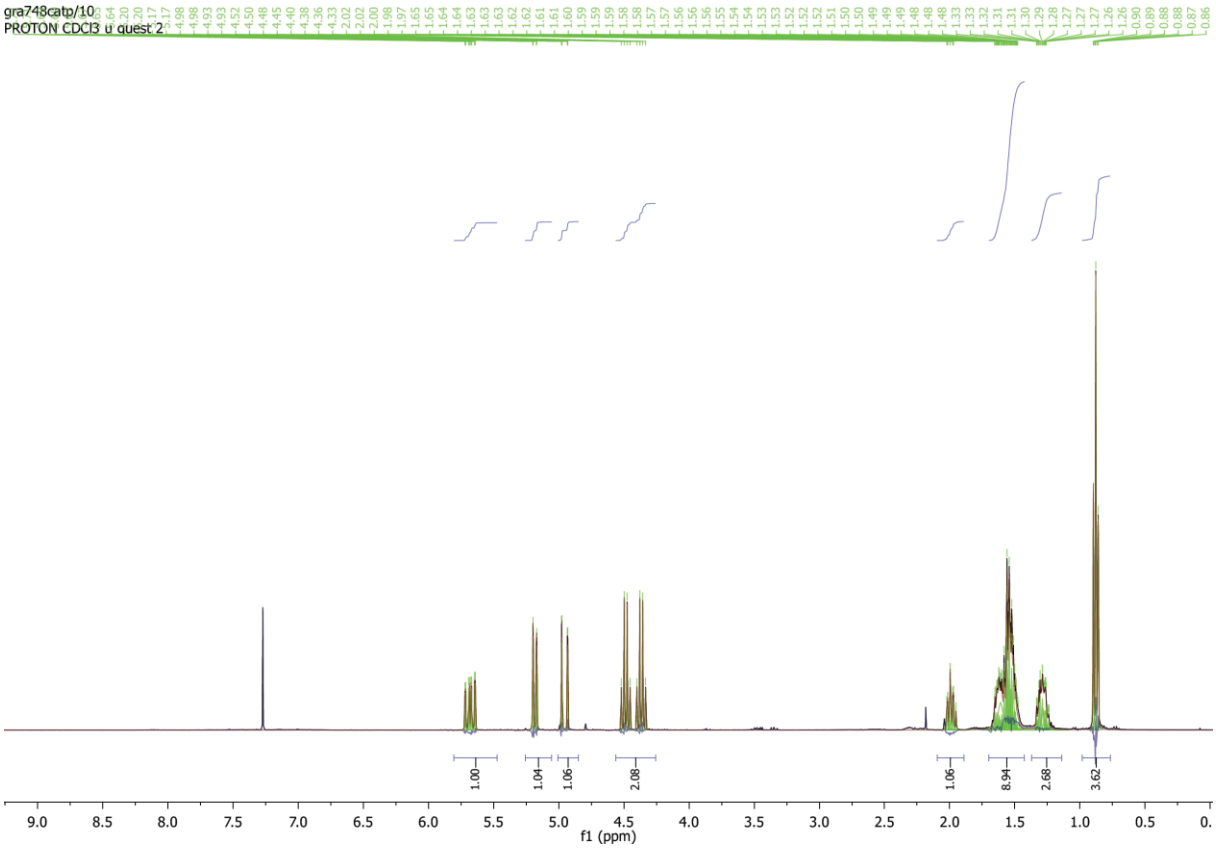

gra719catp1/1
1H Spectrum
5.50
1.00
1.07
1.09
4.28
4.42
3.33
f1 (ppm)

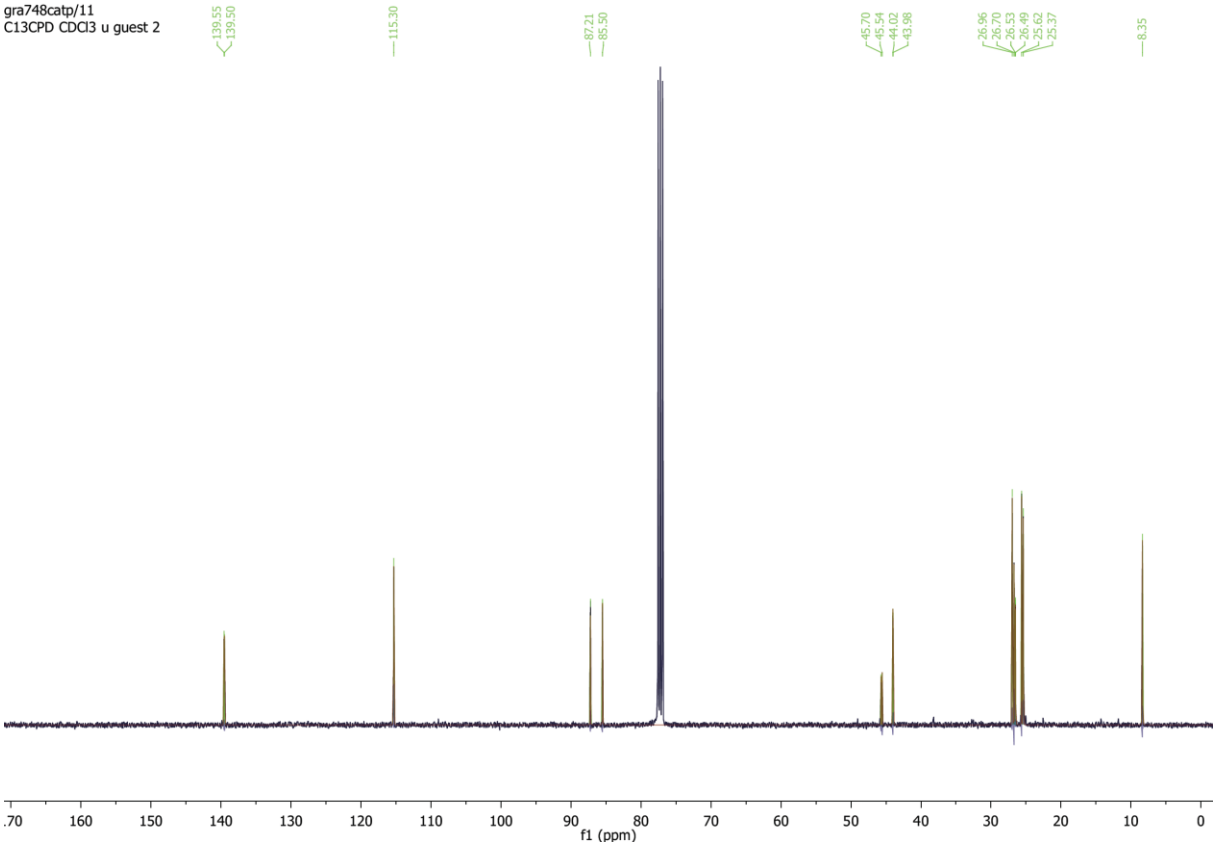

gra719catp1/2
13C CPD-Spectrum
128.40
127.44
126.74
115.42
115.40
109.72
88.64
86.87
49.08
48.91
33.55
33.51
32.14
22.72
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

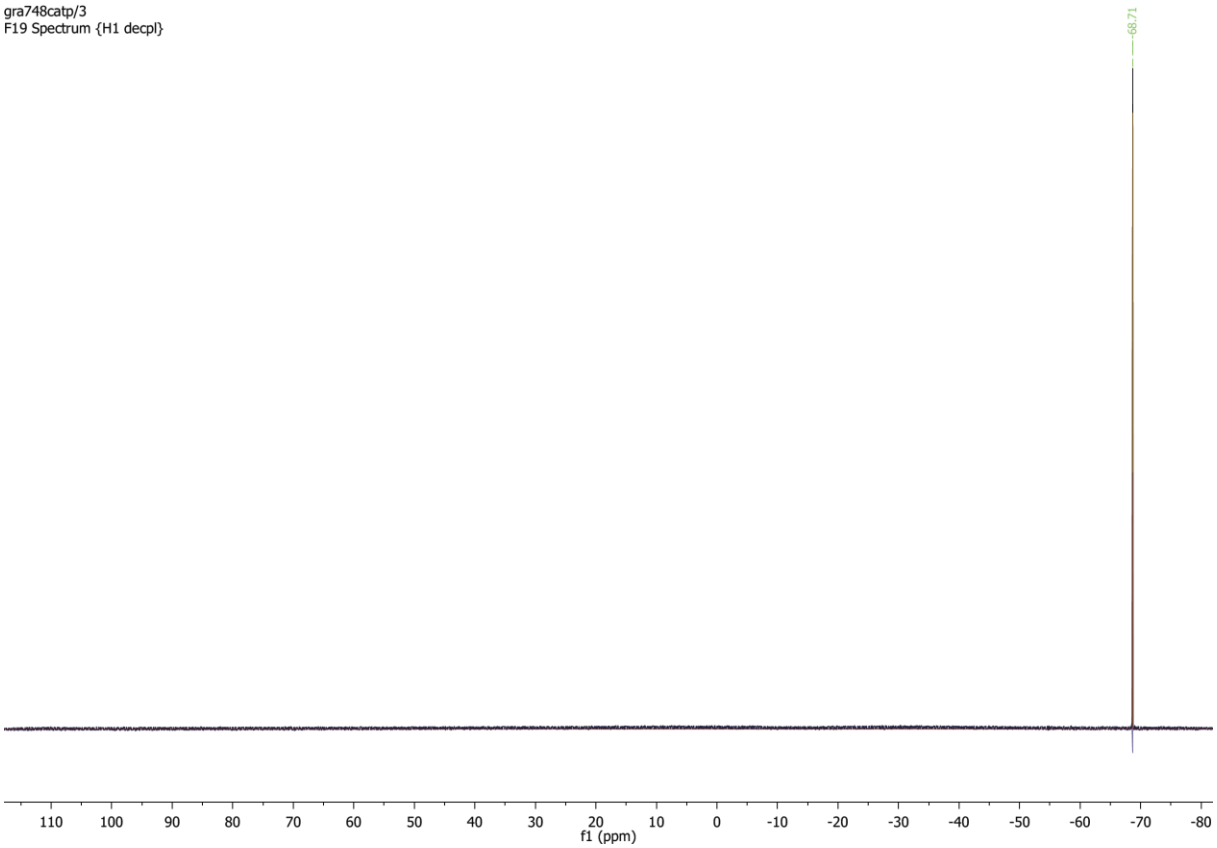

gra719catp1/3
F19 Spectrum {H1 decpl}
60.92
60.92
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

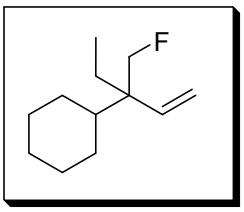

F

1h

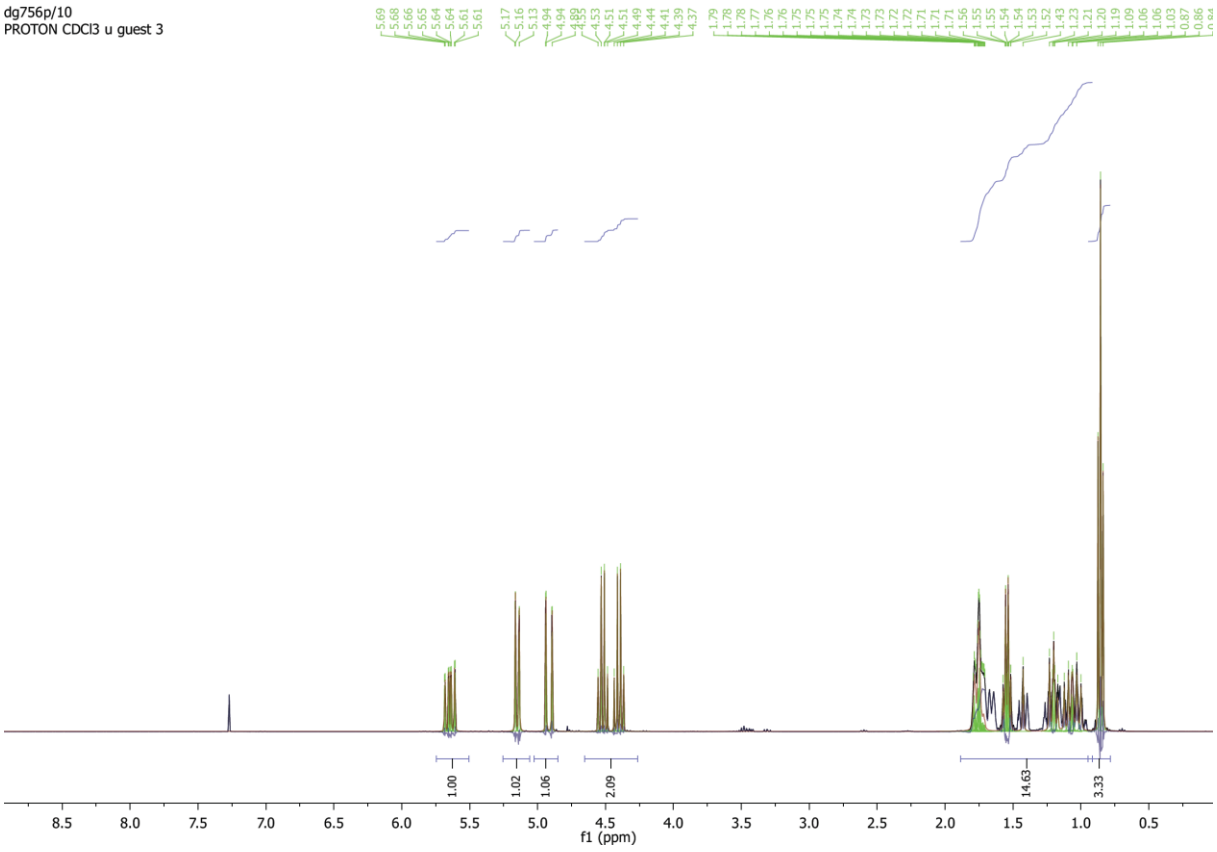

gra748catp/10
PROTON CDCl3 u quest 2
1.00
1.04
1.06
2.08
1.06
8.94
2.68
3.62
f1 (ppm)

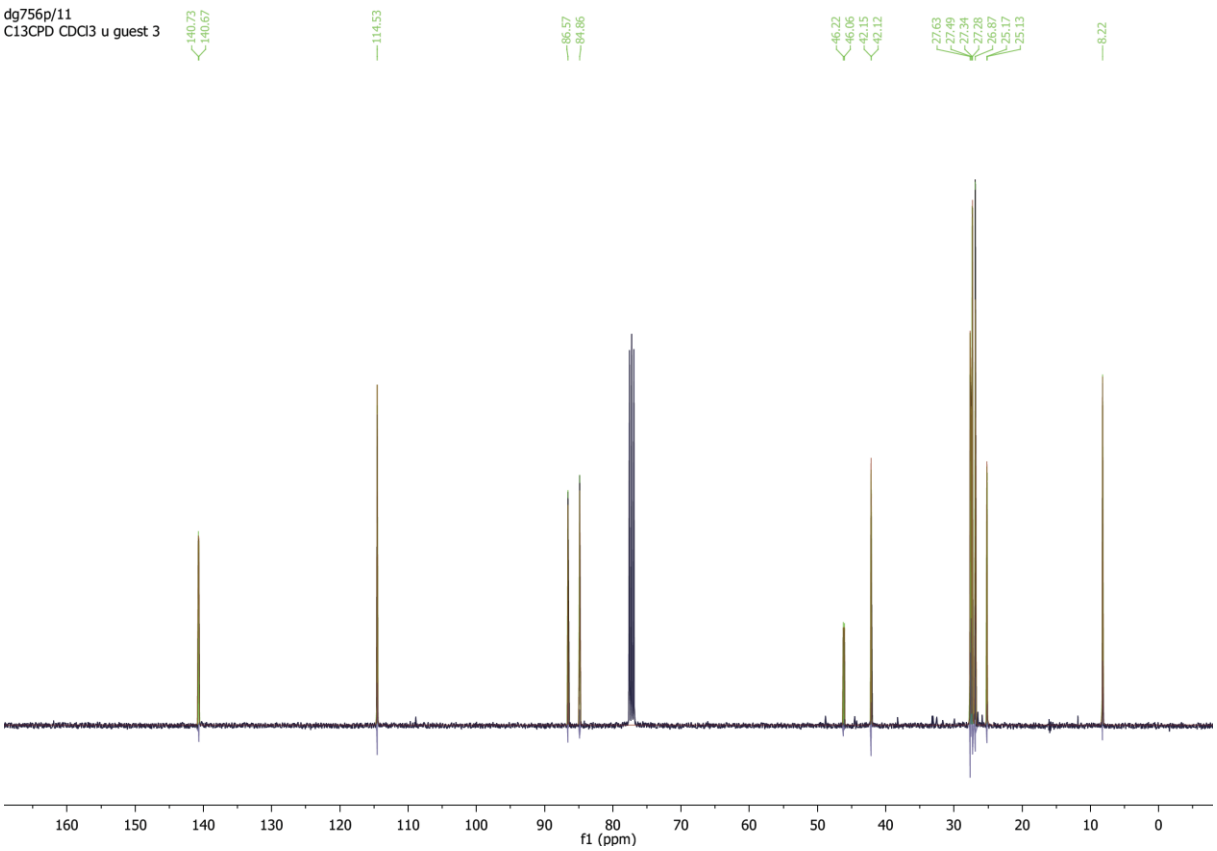

gra748catp/11
C13CPD CDCl3 u guest 2
139.55
139.50
115.30
87.21
85.50
45.70
45.54
44.02
43.98
26.96
26.70
26.53
26.49
25.62
25.37
8.35
f1 (ppm)

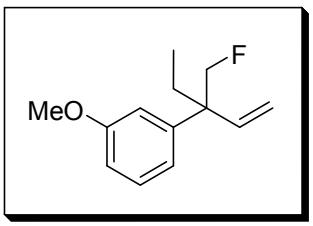

gra748catp/3
F19 Spectrum {H1 decpl}
-68.71
f1 (ppm)


F

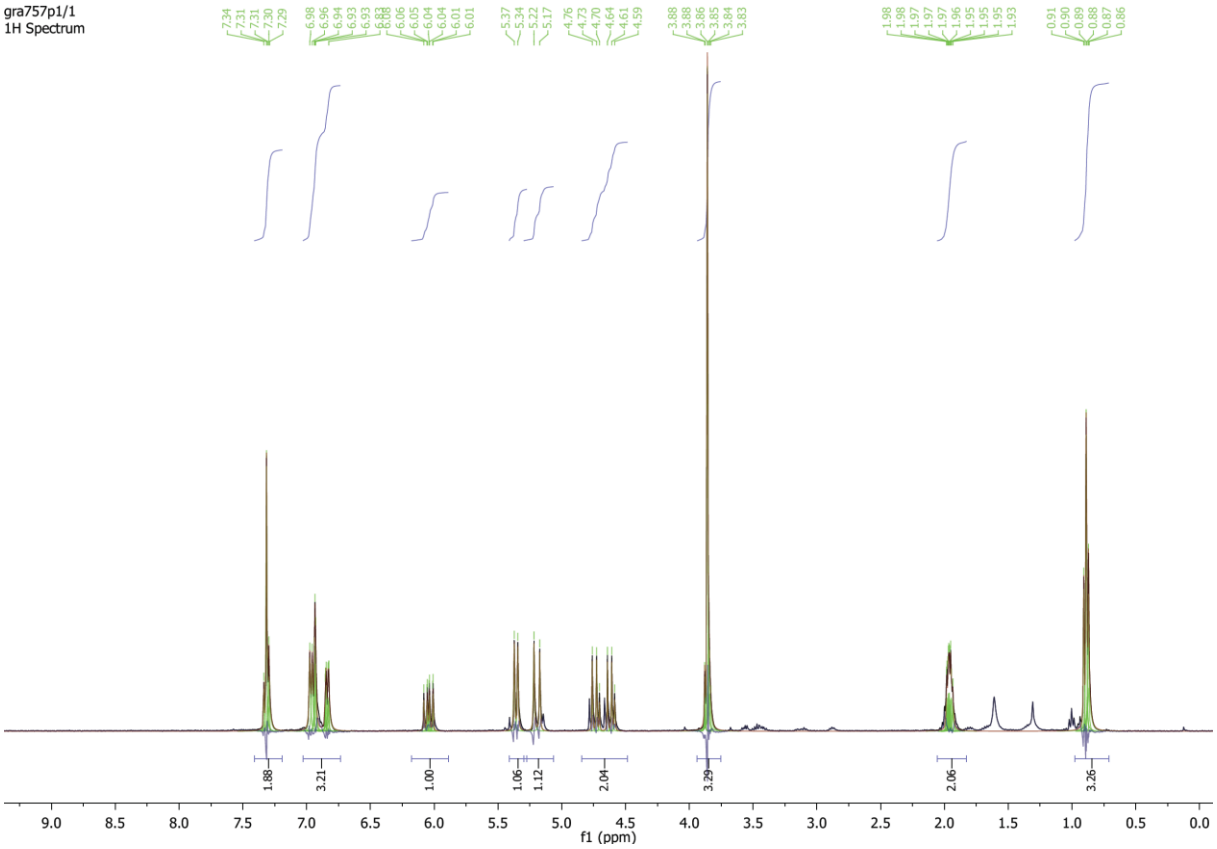
1i

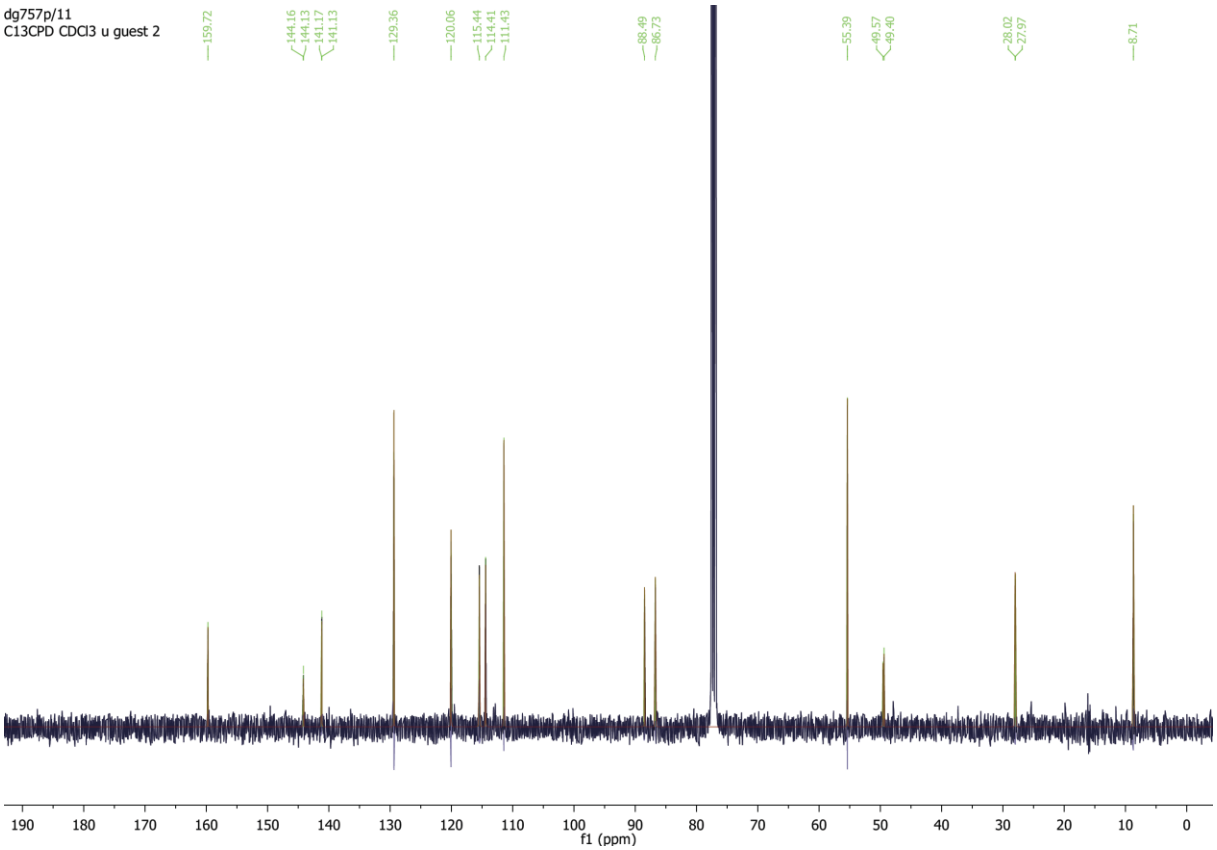

dg756p/10
PROTON CDCl3 u guest 3
1.00
1.02
1.06
2.09
14.63
3.33
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

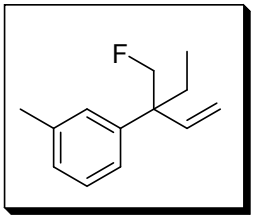

dg756p/11
C13CPD CDCl3 u guest 3
140.73
140.67
114.53
86.57
84.86
46.22
46.06
42.15
42.12
27.63
27.49
27.34
27.28
26.67
25.17
25.13
8.22
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



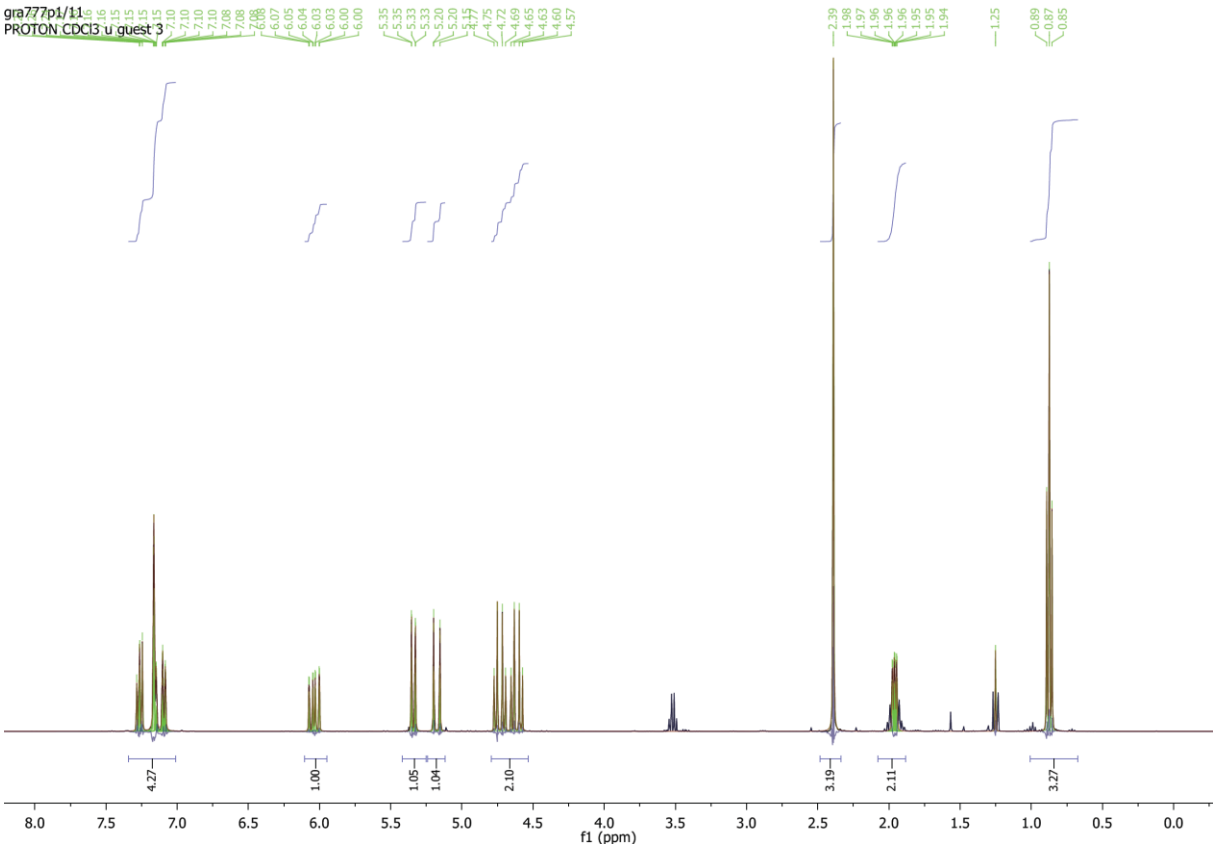
1j

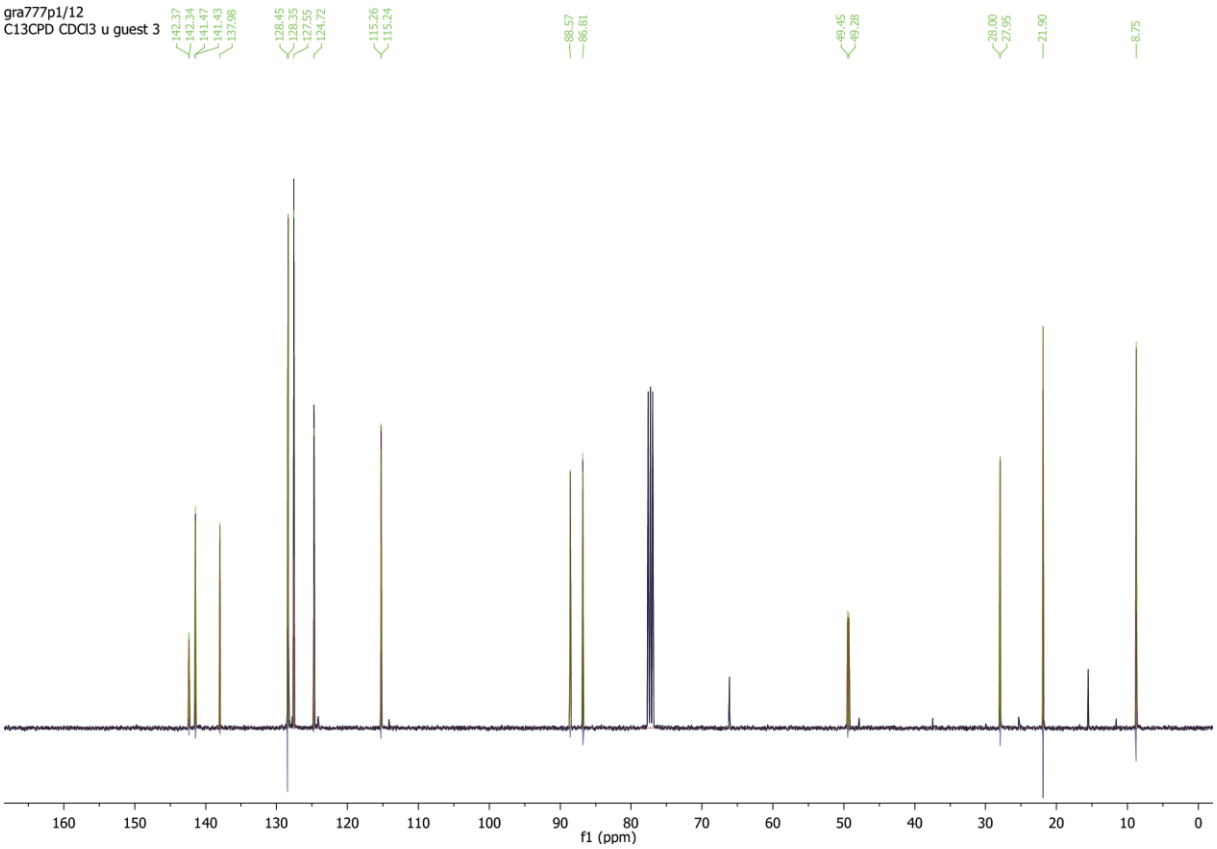


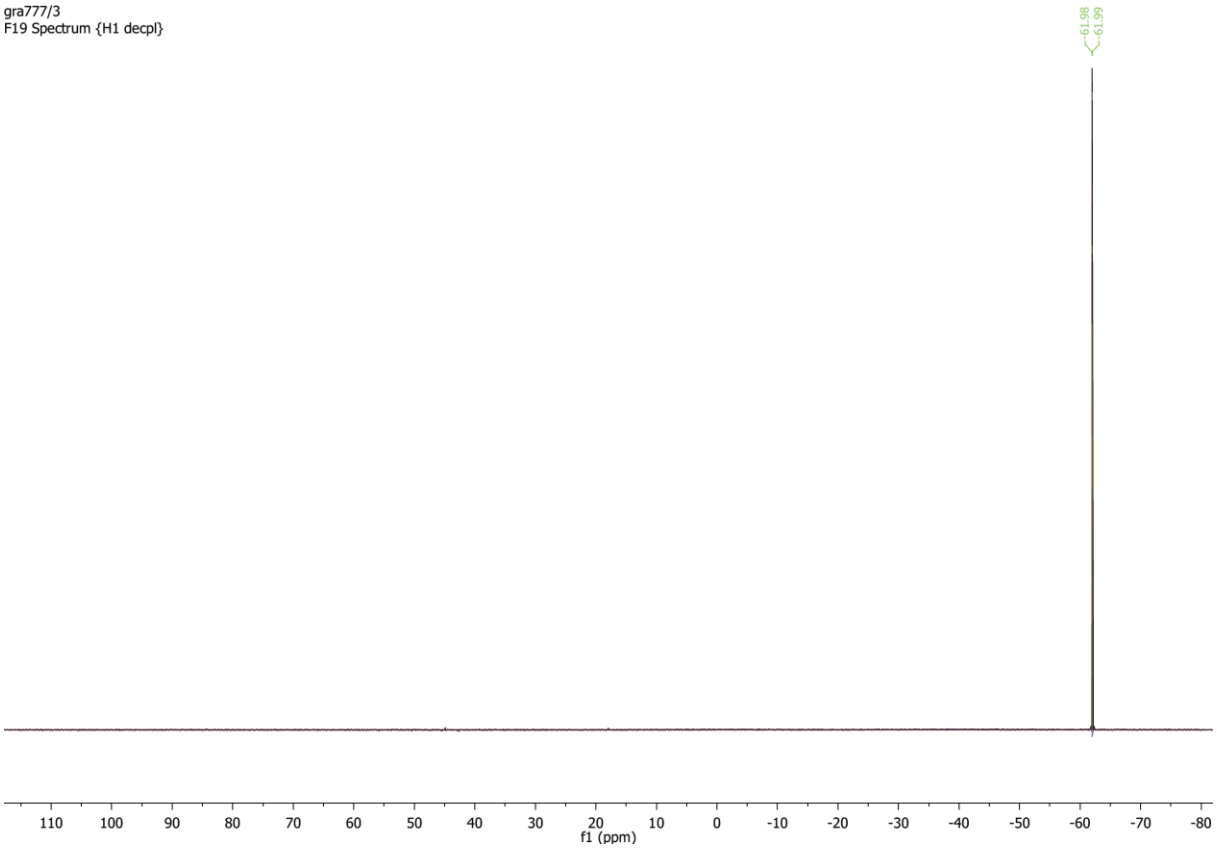

dg757p/11
C13CPD CDCl3 u guest 2
159.72
144.16
144.13
141.17
141.13
129.36
120.06
115.44
114.41
111.43
88.49
86.73
55.39
49.57
49.40
28.02
27.97
8.71
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

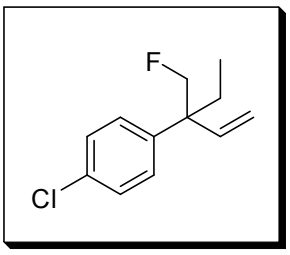

F

1k

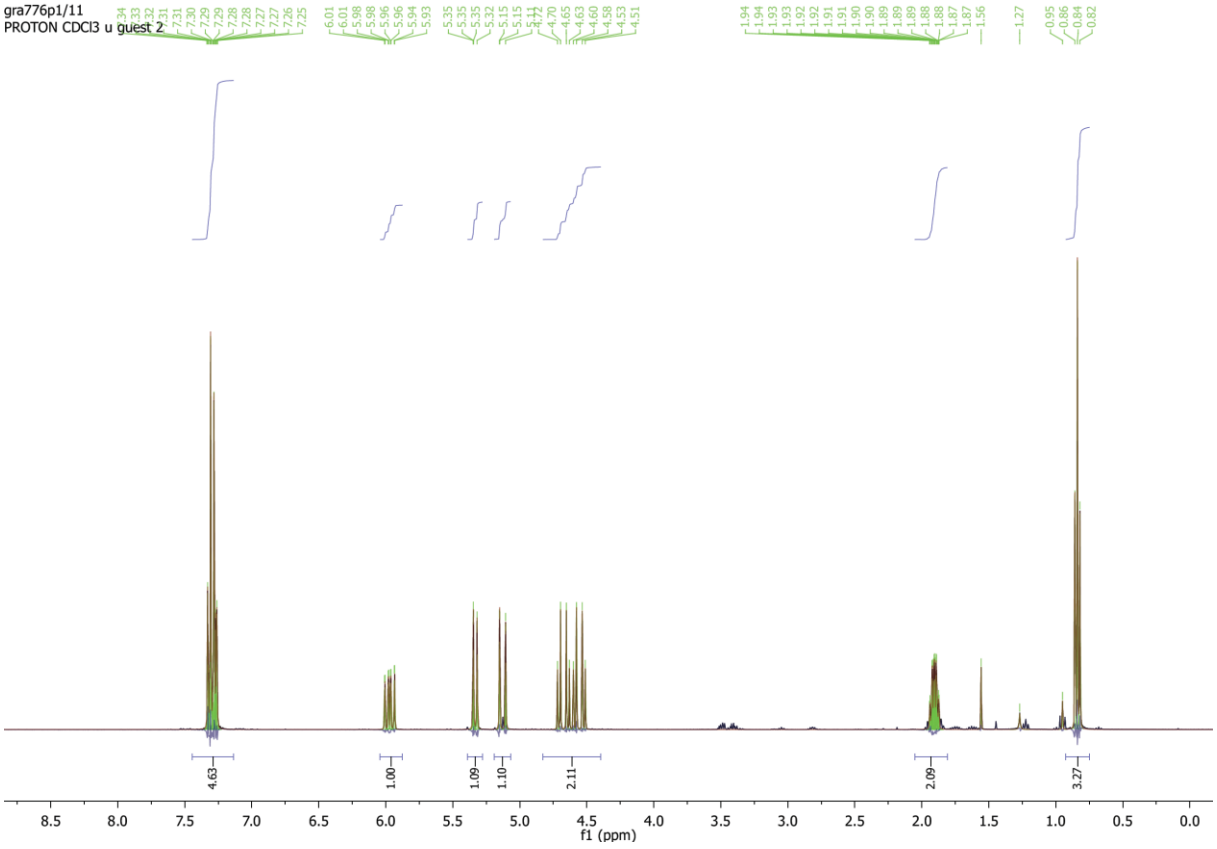

gra777p1/11
PROTON CDCl3 u guest 3
4.27
1.00
1.05
1.04
2.10
3.19
2.11
3.27
f1 (ppm)

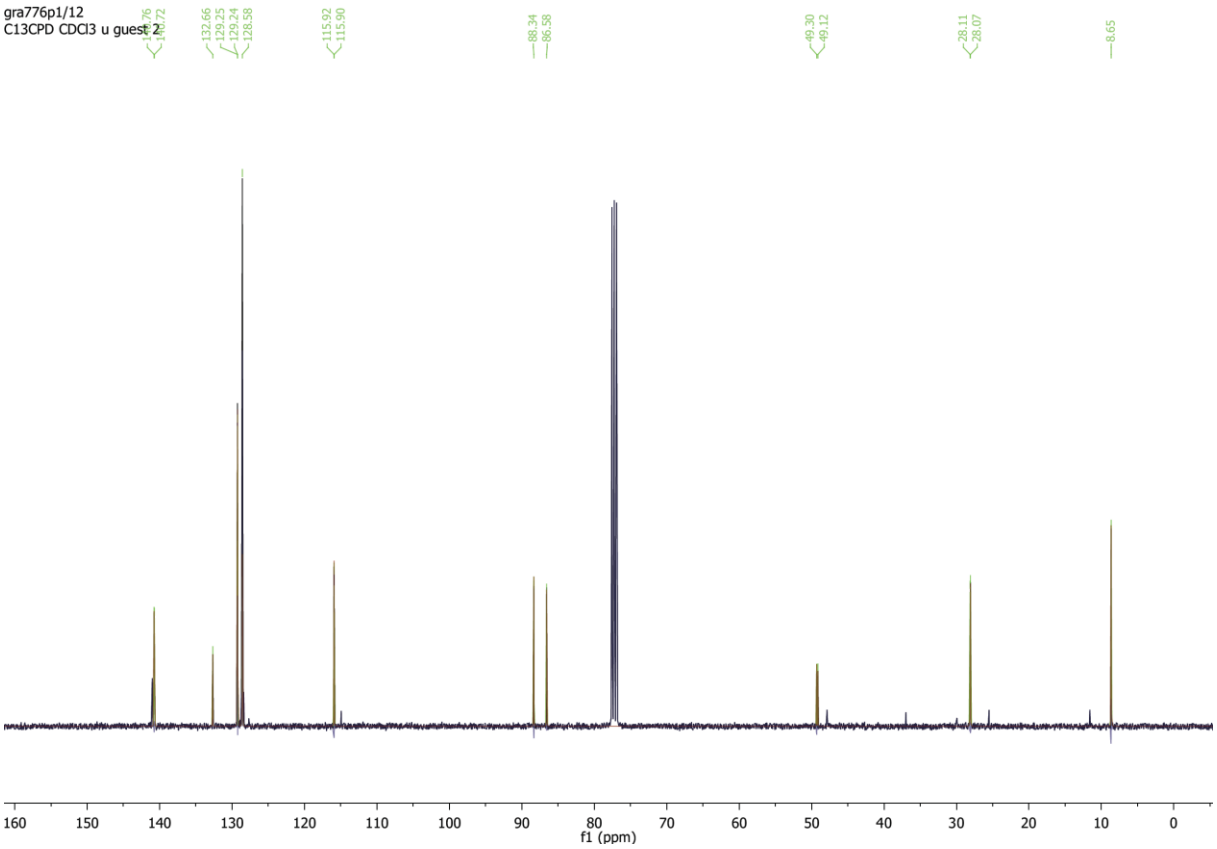

gra777p1/12
C13CPD CDCl3 u guest 3
142.37
142.34
141.47
141.43
137.98
128.45
128.35
127.55
124.72
115.26
115.24
88.57
86.81
49.45
49.28
28.00
27.95
21.90
8.75
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

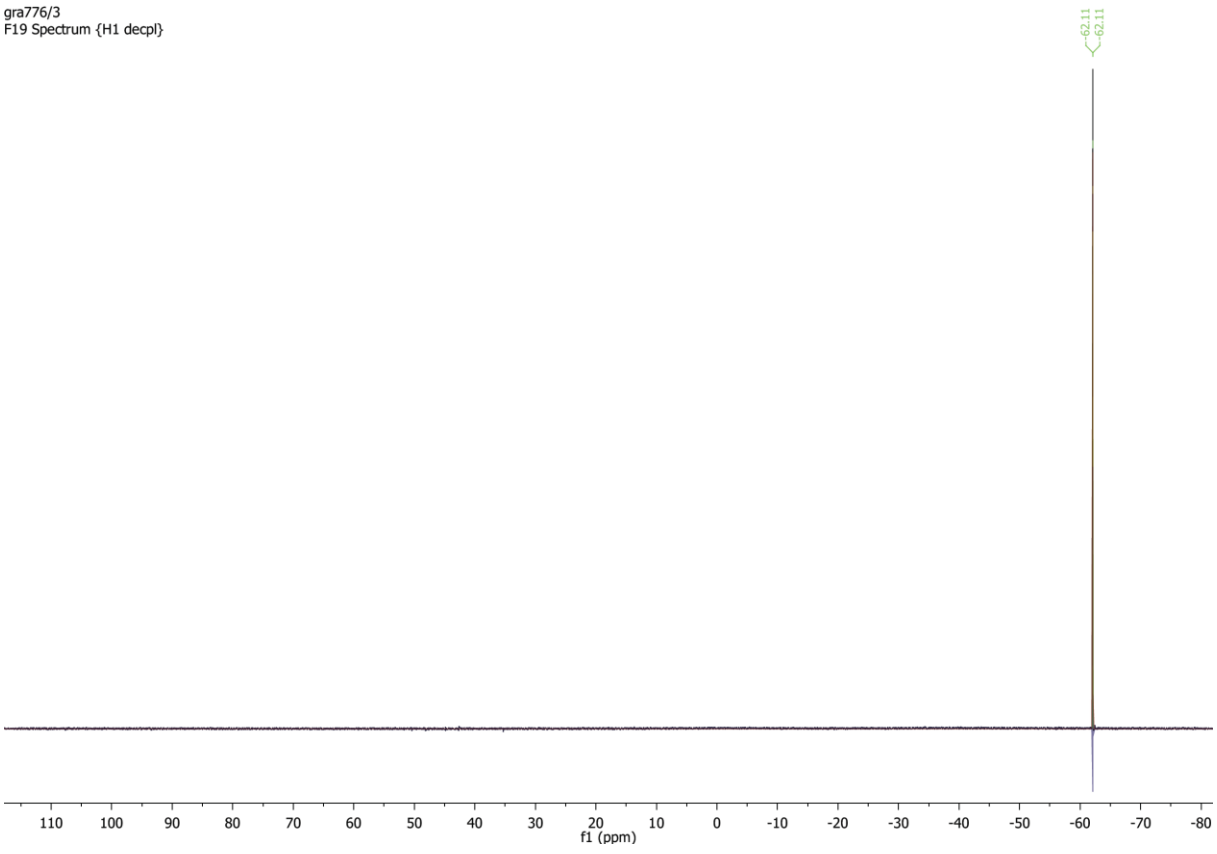

gra777/3
F19 Spectrum {H1 decpl}
61.98
61.99
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

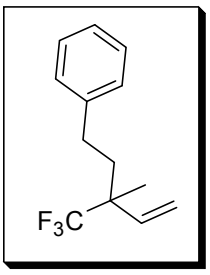

F
Cl

1l

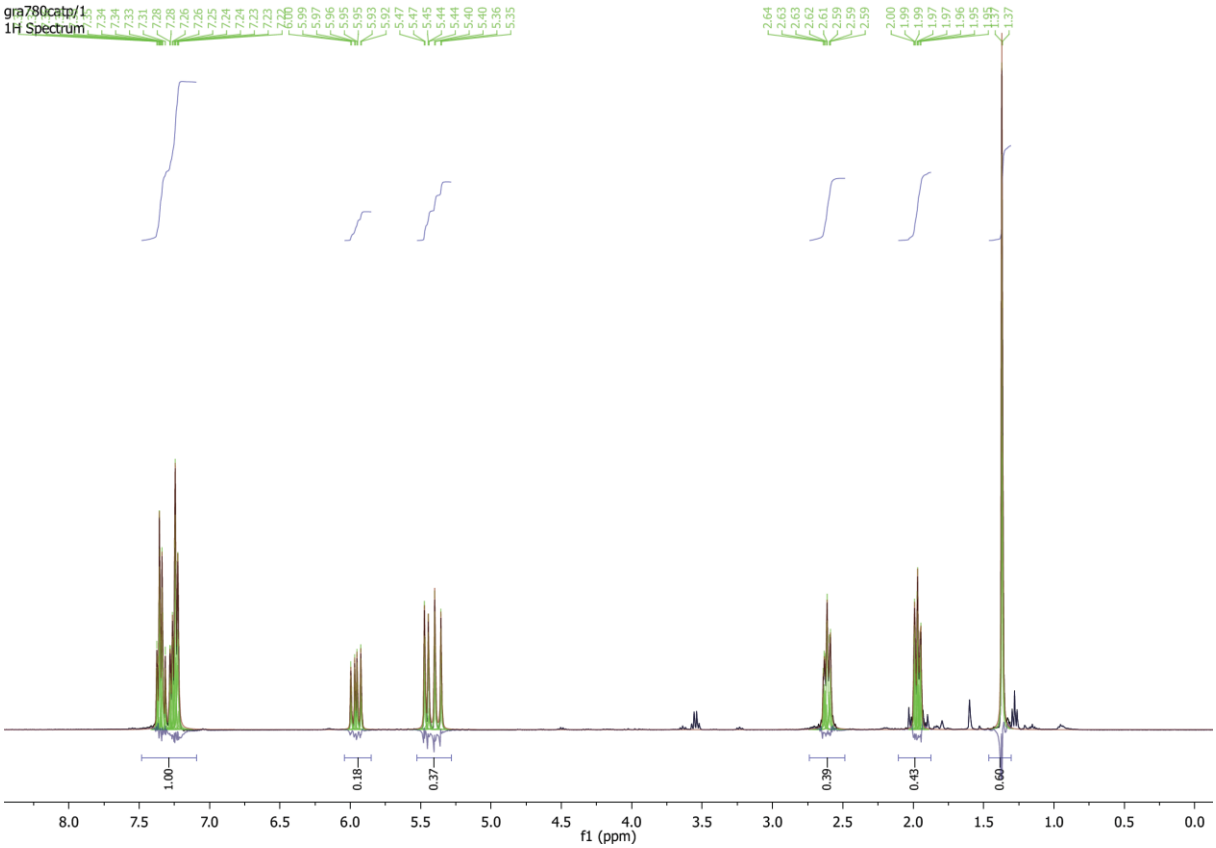

gra776p1/11
PROTON CDCl3 u guest 2
4.63
1.00
1.09
1.10
2.11
2.09
3.27
f1 (ppm)

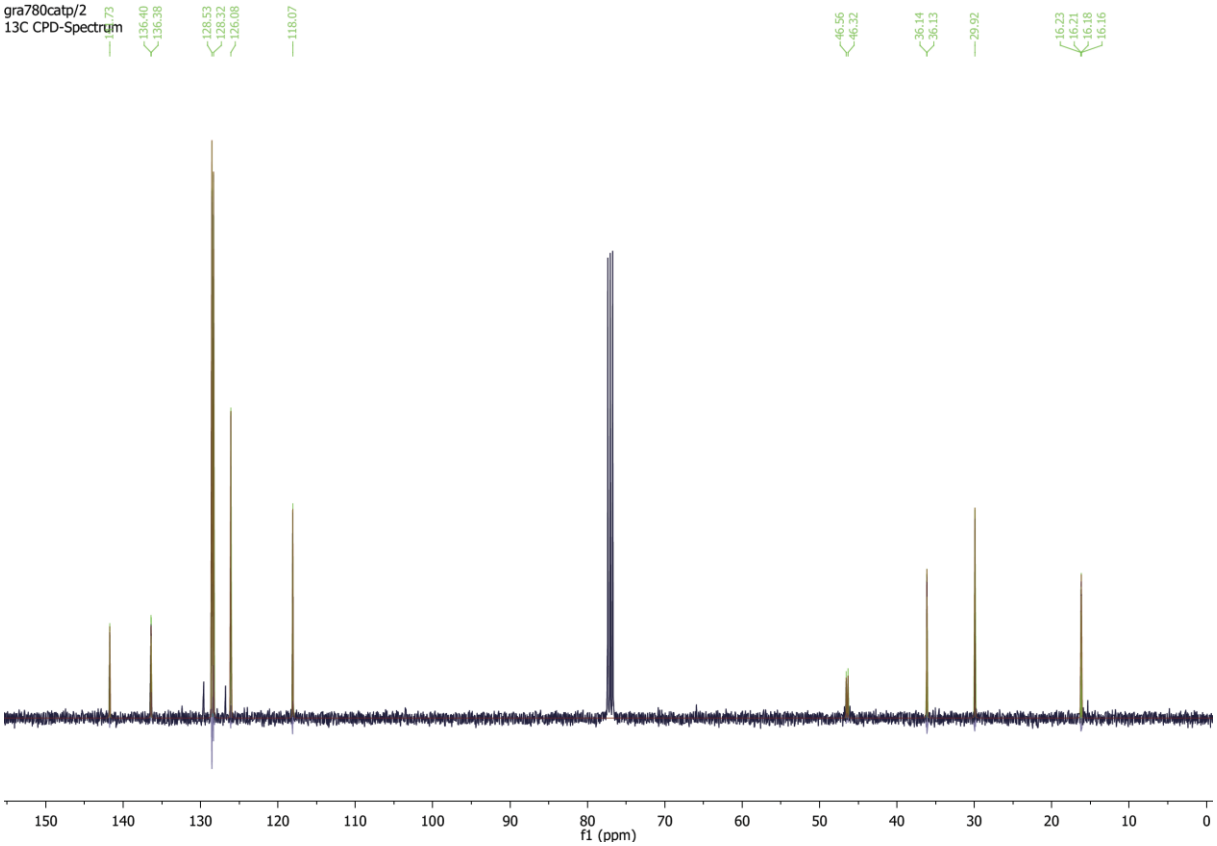

gra776p1/12
C13CPD CDCl3 u guest 2
140.76
140.72
132.66
129.35
129.24
128.58
115.92
115.90
88.34
86.58
49.30
49.12
28.11
28.07
8.65
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

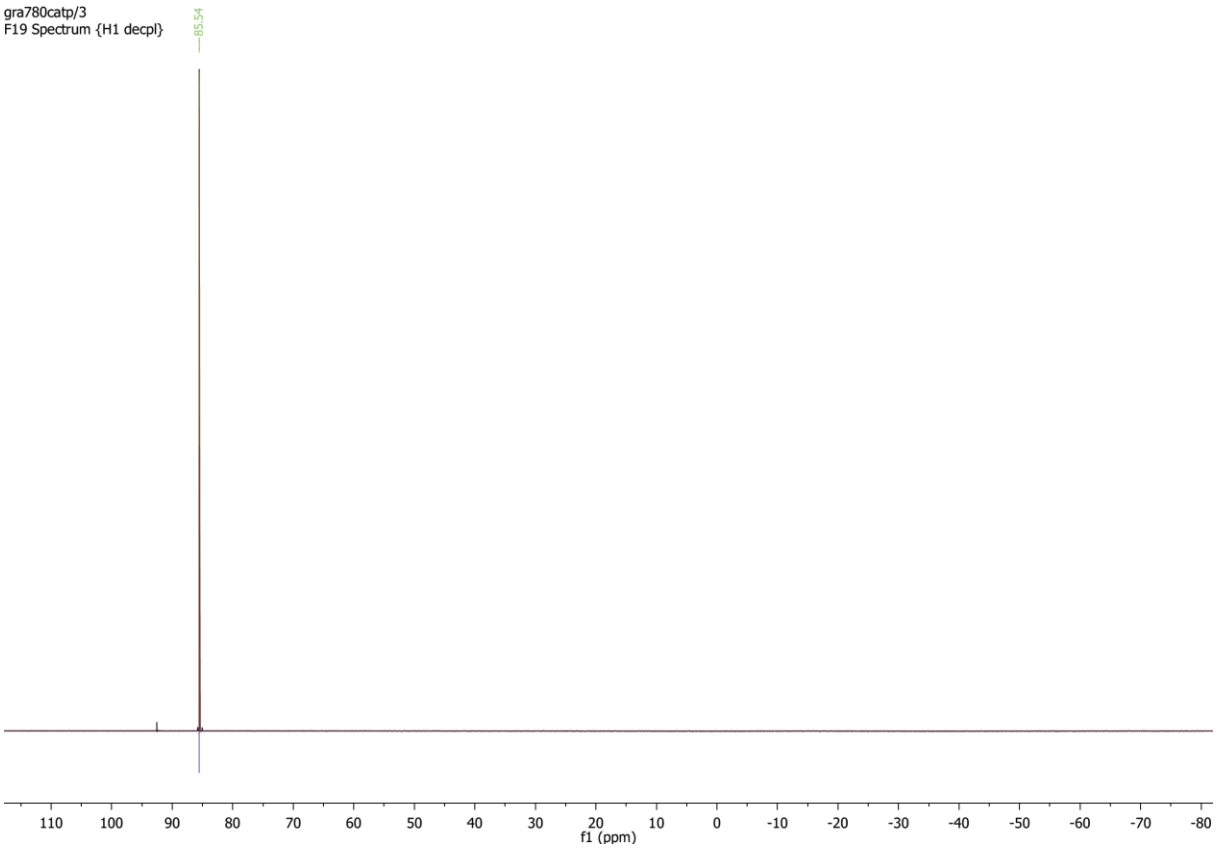

gra776/3
F19 Spectrum {H1 decpl}
62.11
62.11
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

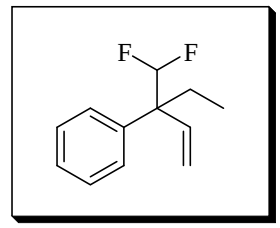


1n

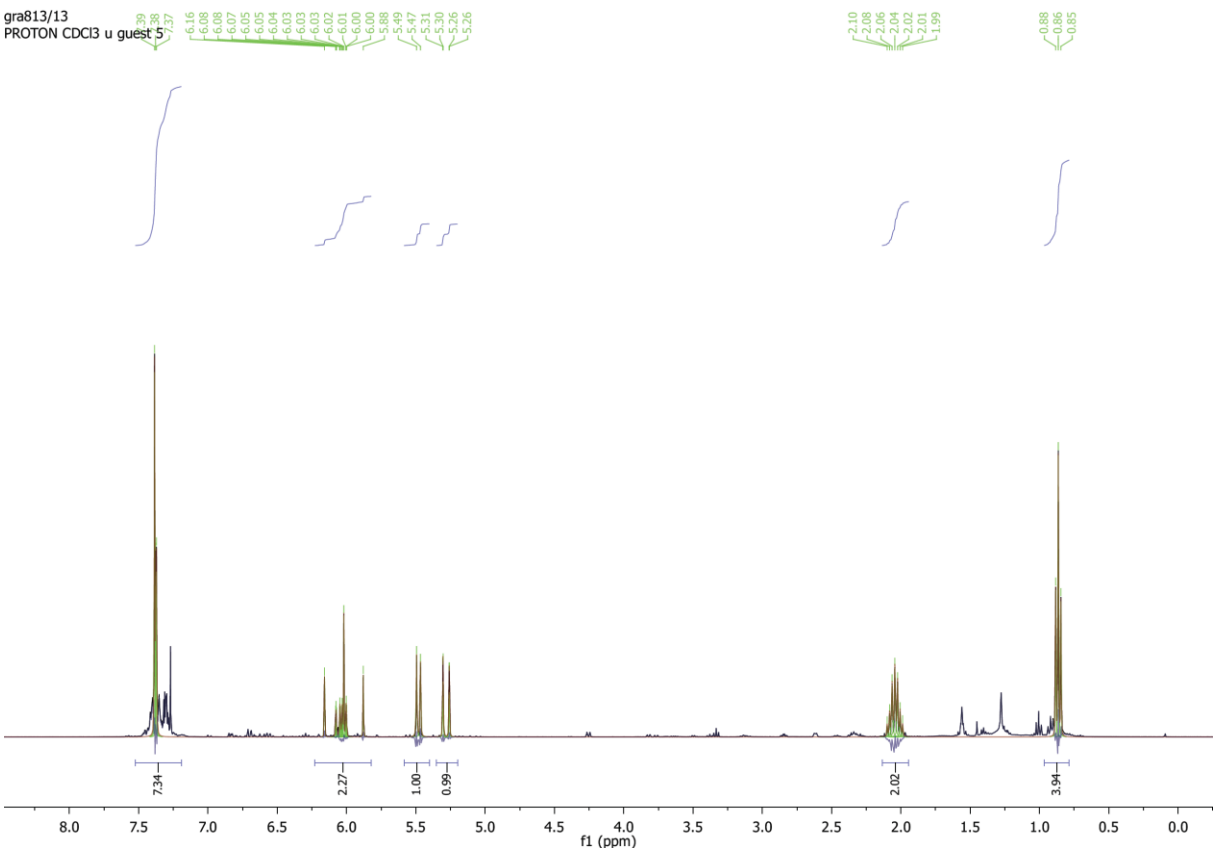


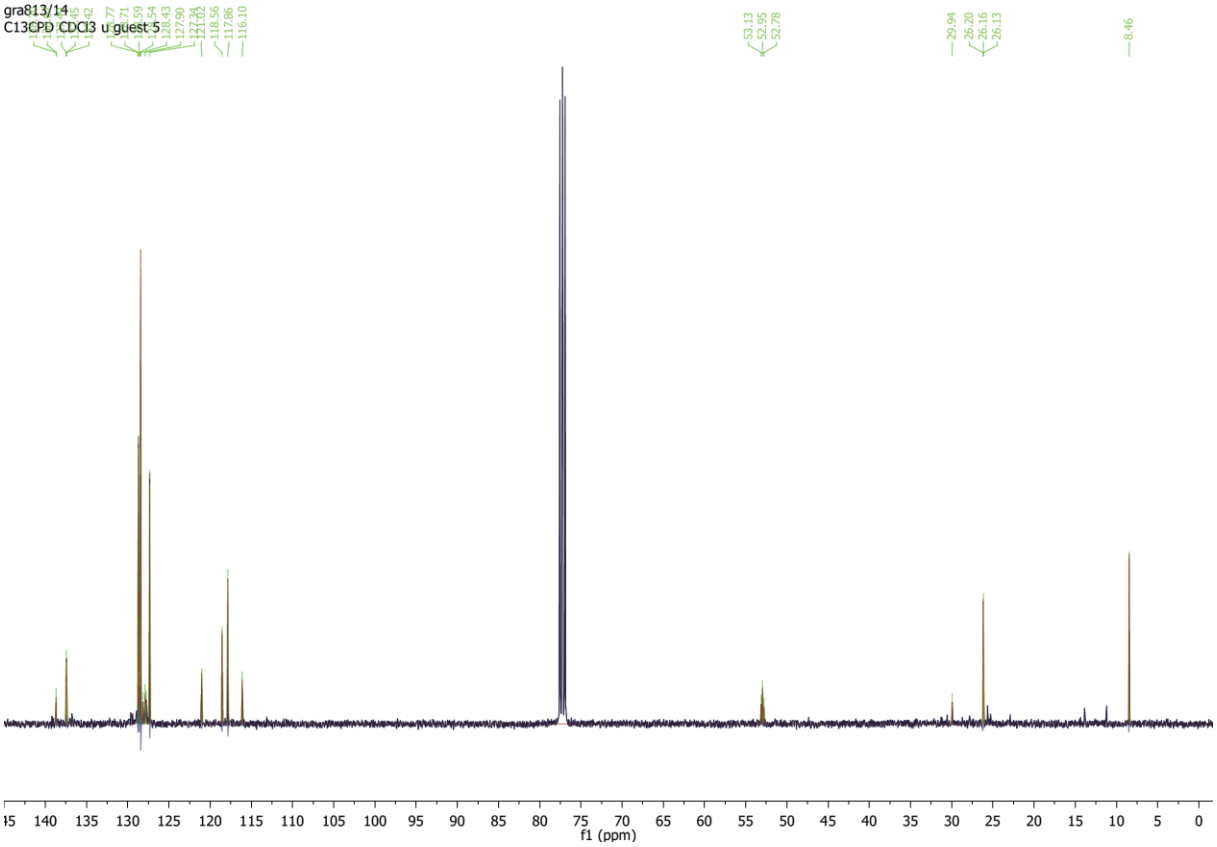

gra780catp/2
13C CPD-Spectrum
136.40
136.38
128.53
128.32
126.08
118.07
46.56
46.32
36.14
36.13
29.92
16.23
16.21
16.18
16.16
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

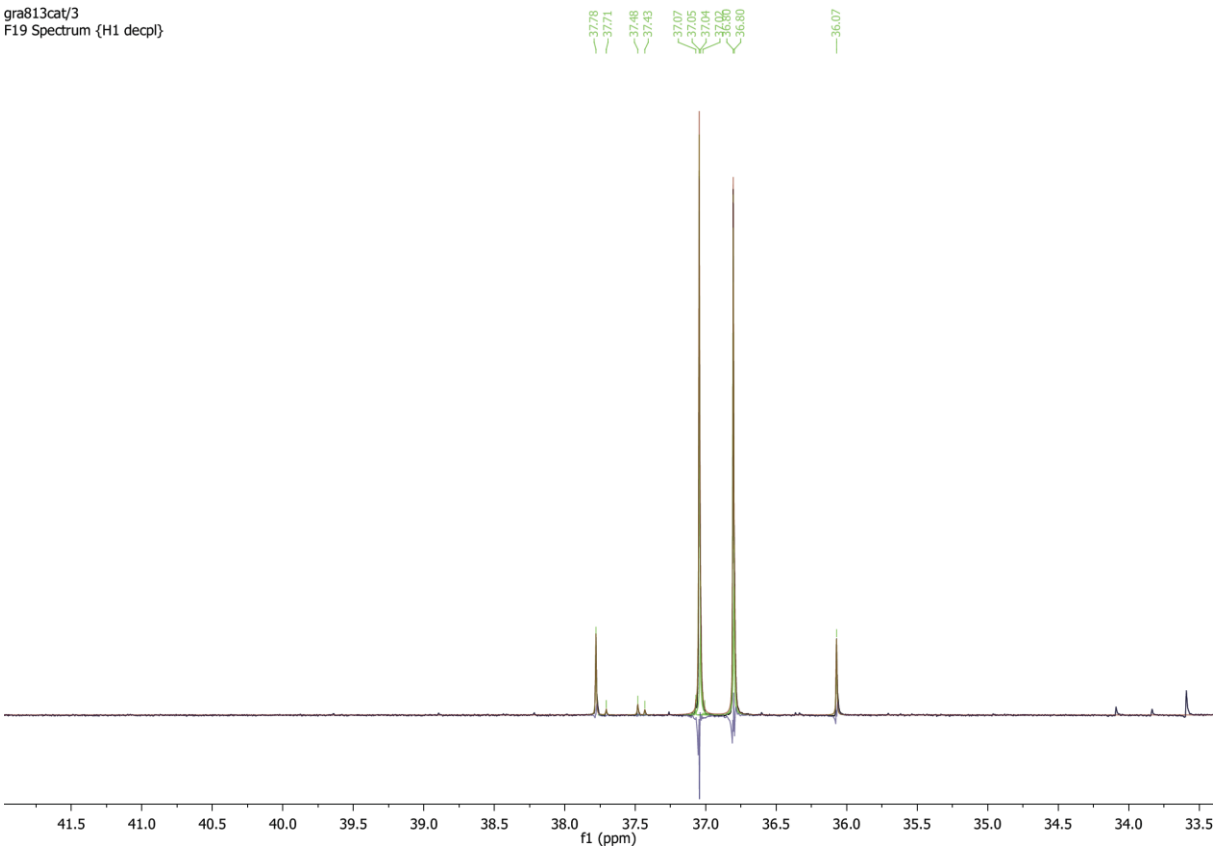

gra780catp/3
F19 Spectrum {H1 decpl}
85.54
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

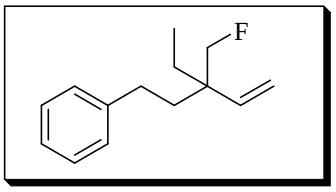

F
F

1o

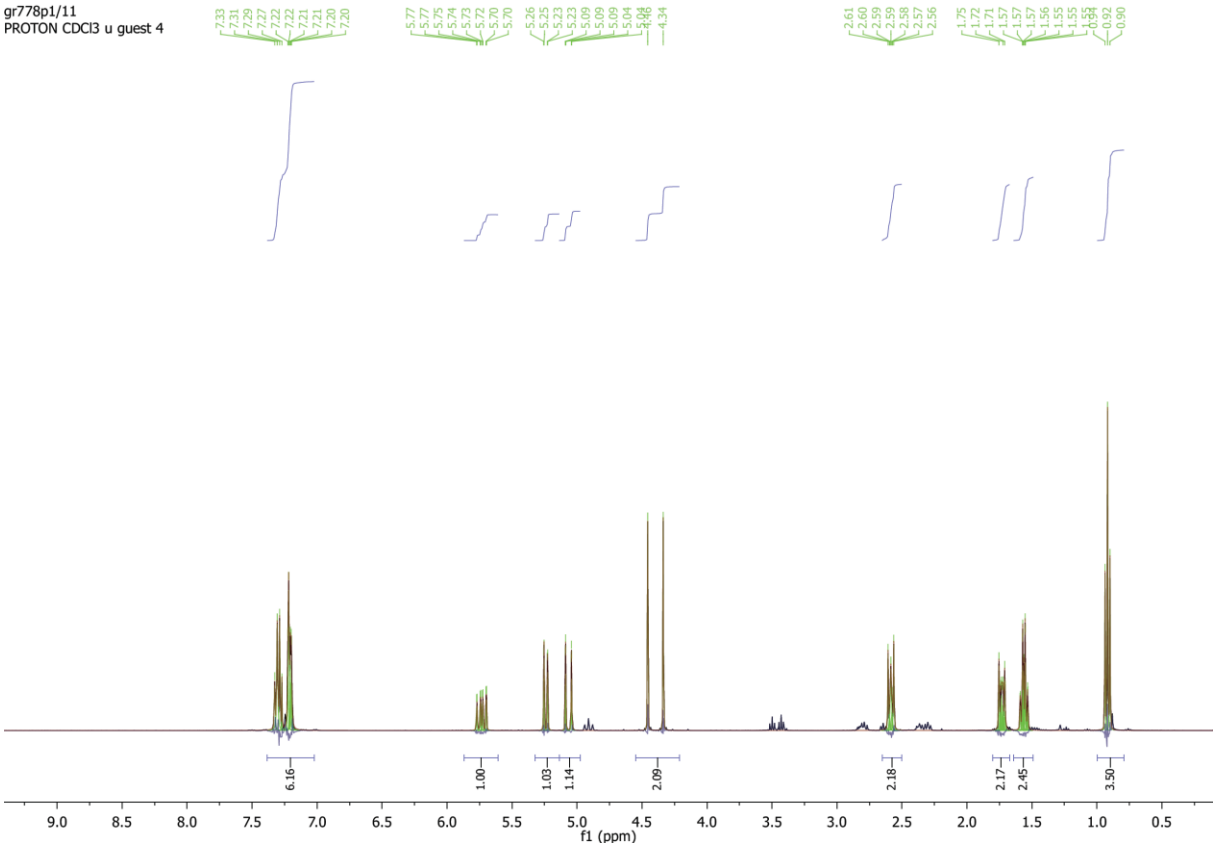

gra813/13
PROTON CDCl3 u guest 5
7.34
2.27
1.00
0.99
2.02
3.94
f1 (ppm)

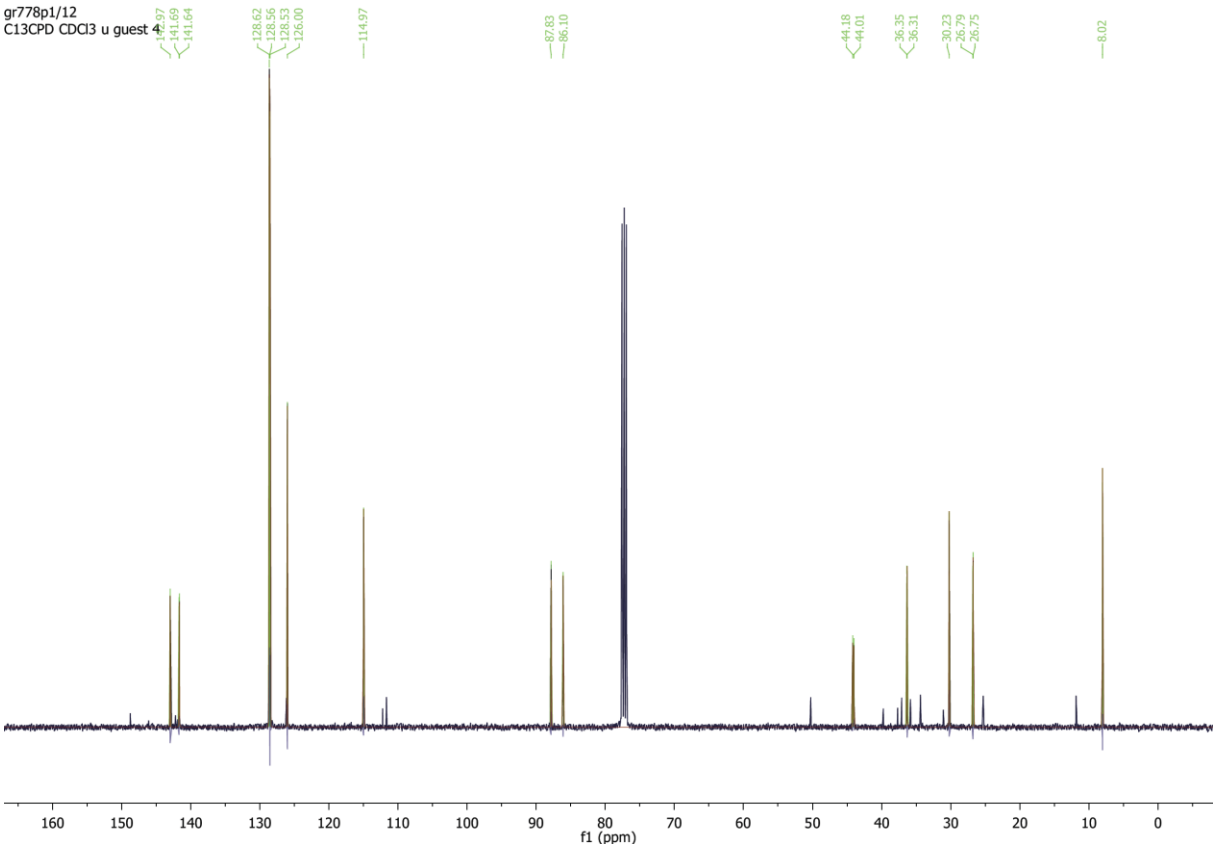

gra813/14
C13CPD CDCl3 u guest 5
53.13
52.95
52.78
29.94
26.20
26.16
26.13
8.46
f1 (ppm)

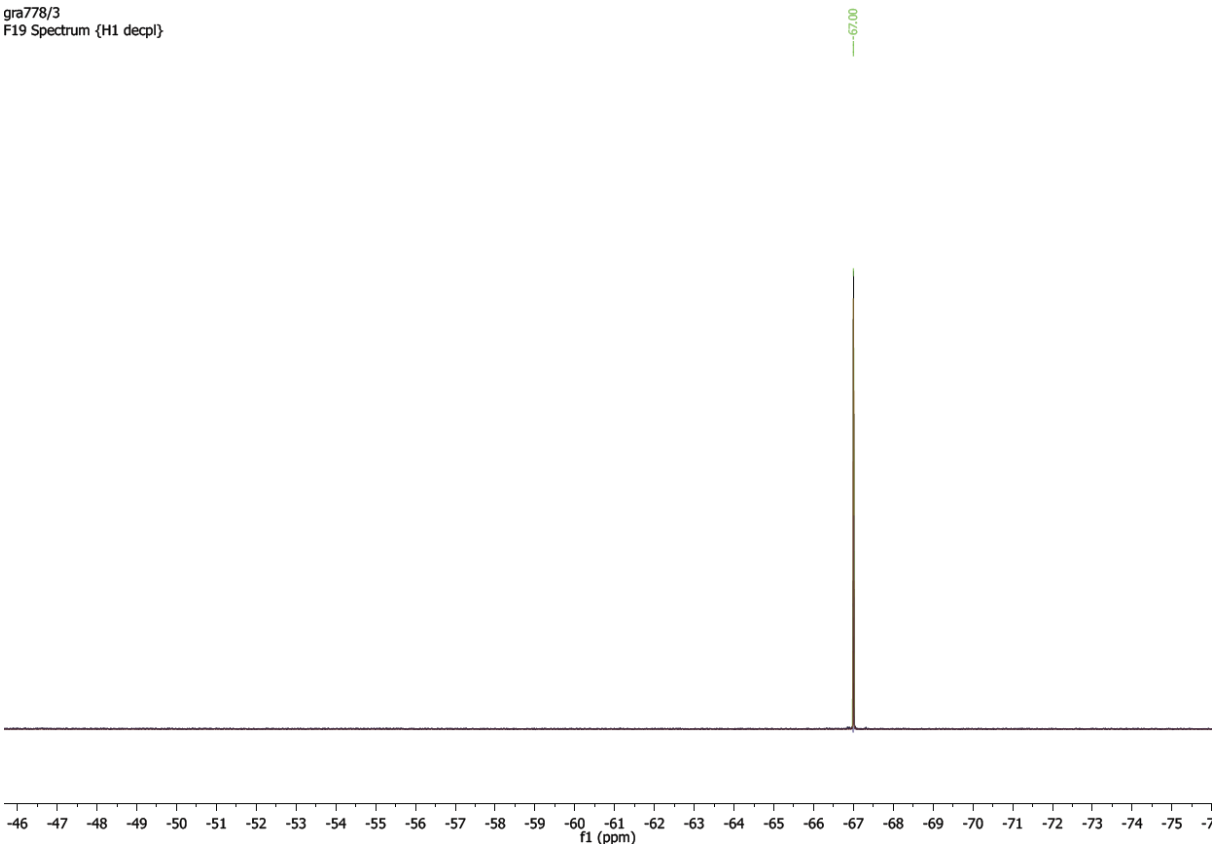

gra813cat/3
F19 Spectrum {H1 decpl}
37.78
37.71
37.48
37.43
37.07
37.05
37.04
36.80
36.80
36.07
f1 (ppm)

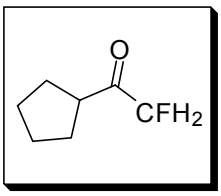

F

1m

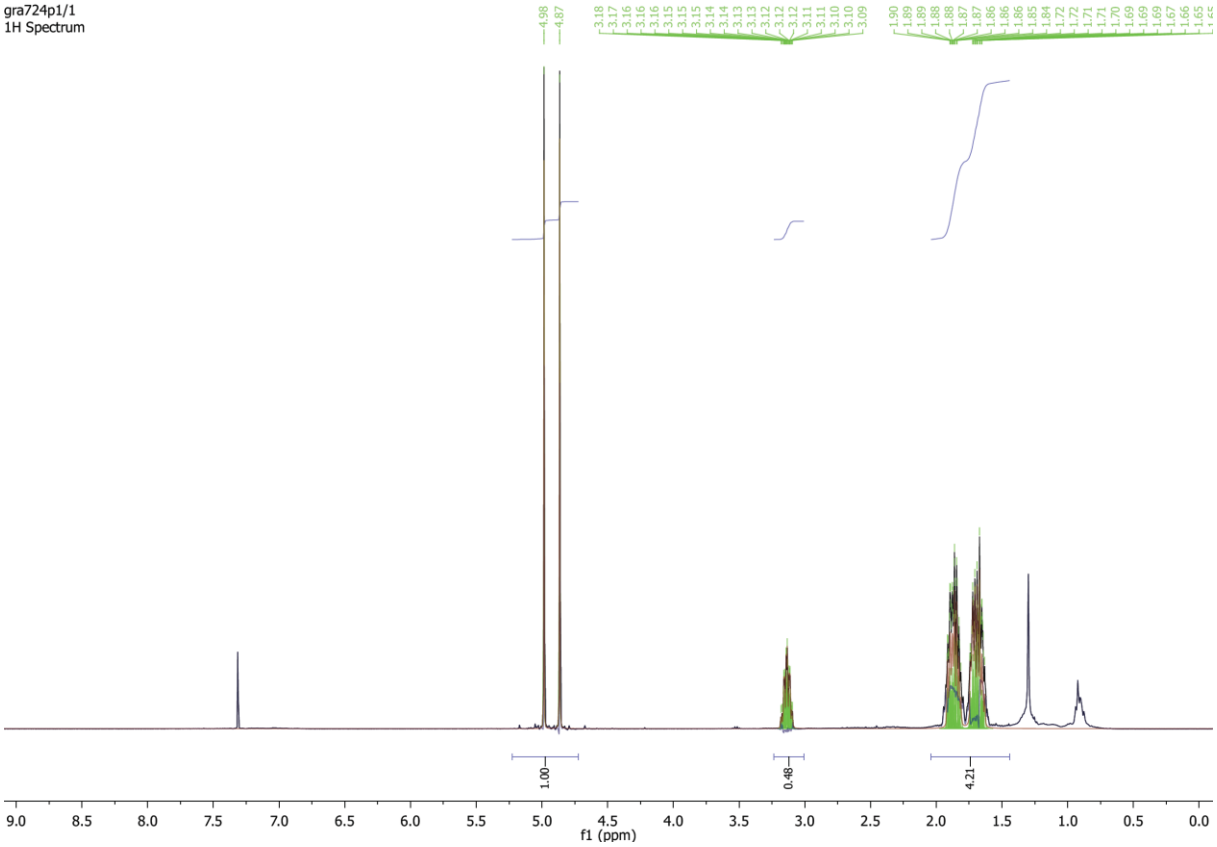

gr778p1/11
PROTON CDCl3 u guest 4
6.16
1.00
1.03
1.14
2.09
2.18
2.17
2.45
3.50
f1 (ppm)

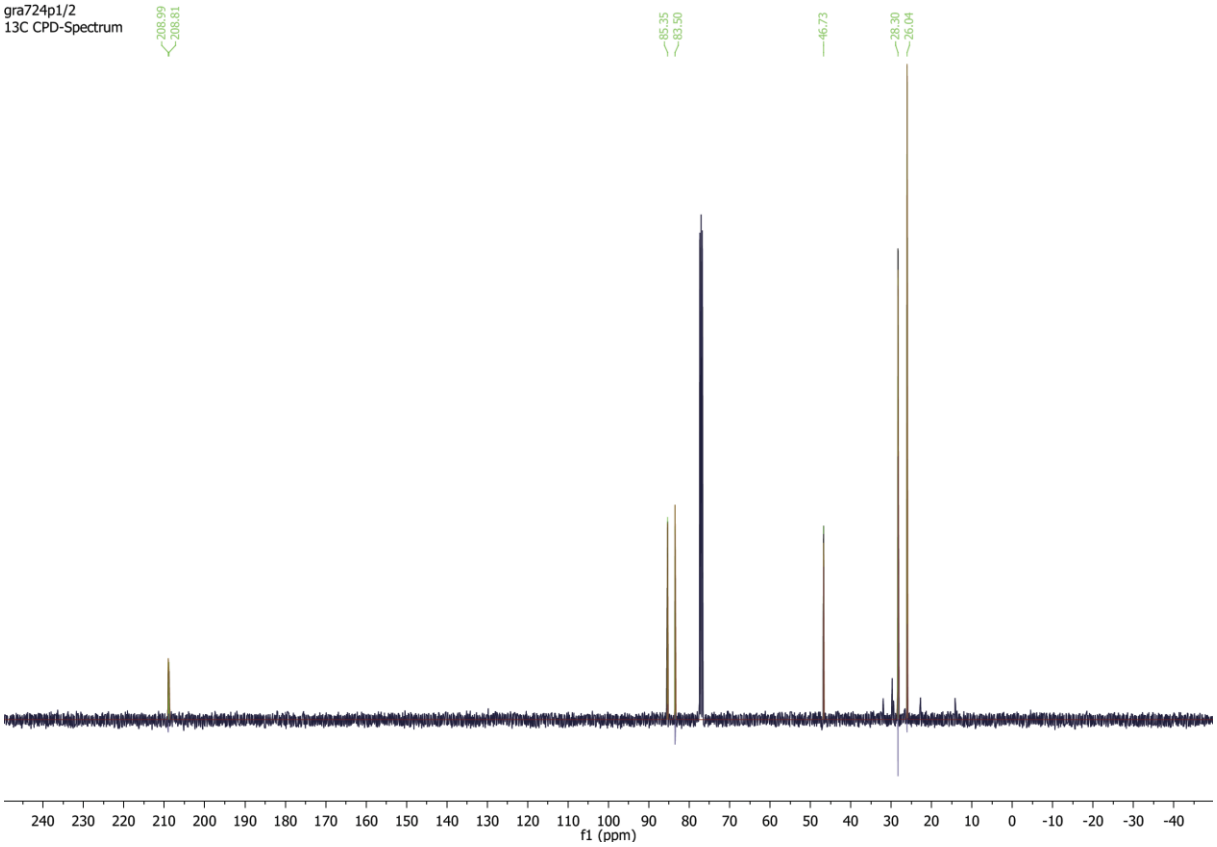

gr778p1/12
C13CPD CDCl3 u guest 4
141.97
141.69
141.64
128.62
128.56
128.53
126.00
114.97
87.83
86.10
44.18
44.01
36.35
36.31
30.23
26.79
26.75
8.02
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

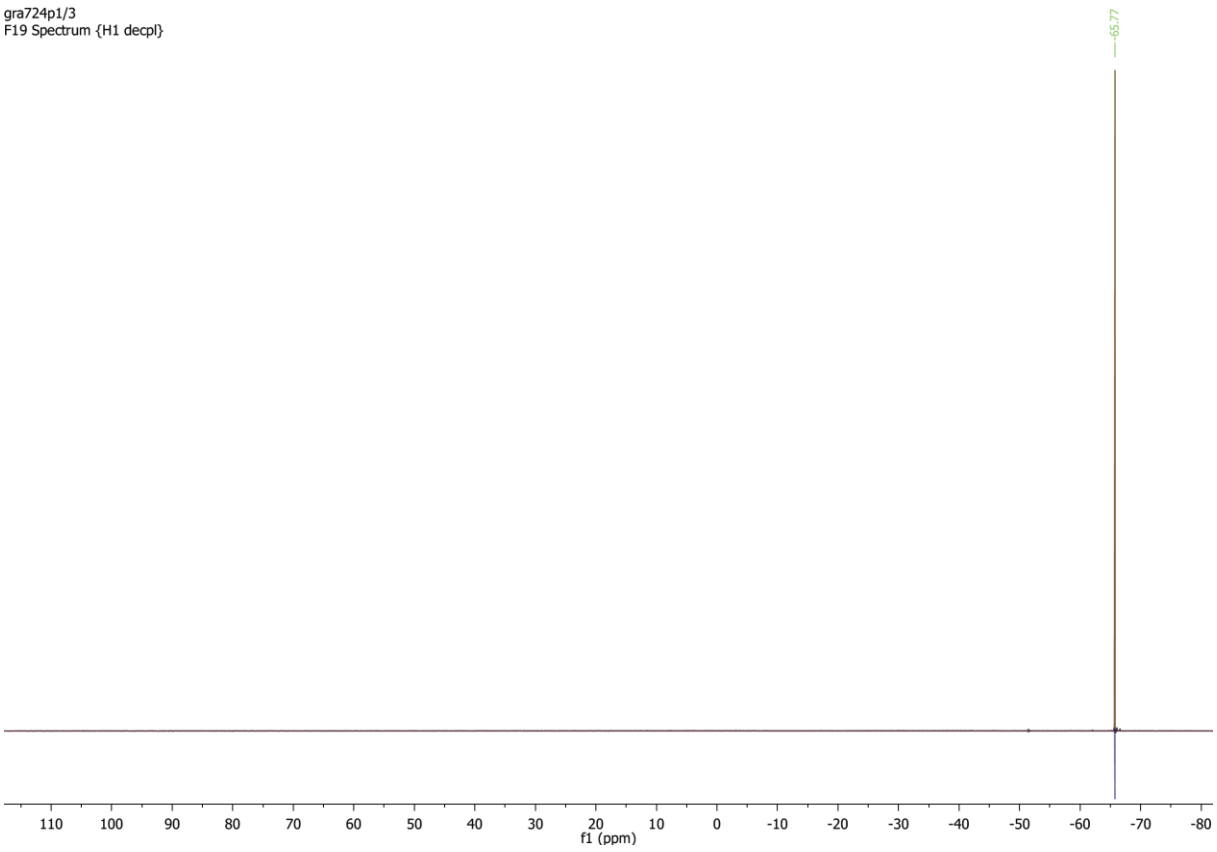

gra778/3
F19 Spectrum {H1 decpl}
-67.00
-46 -47 -48 -49 -50 -51 -52 -53 -54 -55 -56 -57 -58 -59 -60 -61 -62 -63 -64 -65 -66 -67 -68 -69 -70 -71 -72 -73 -74 -75
f1 (ppm)

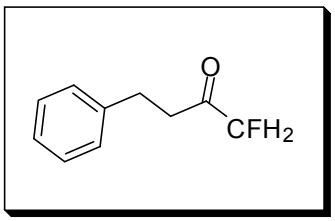


2a

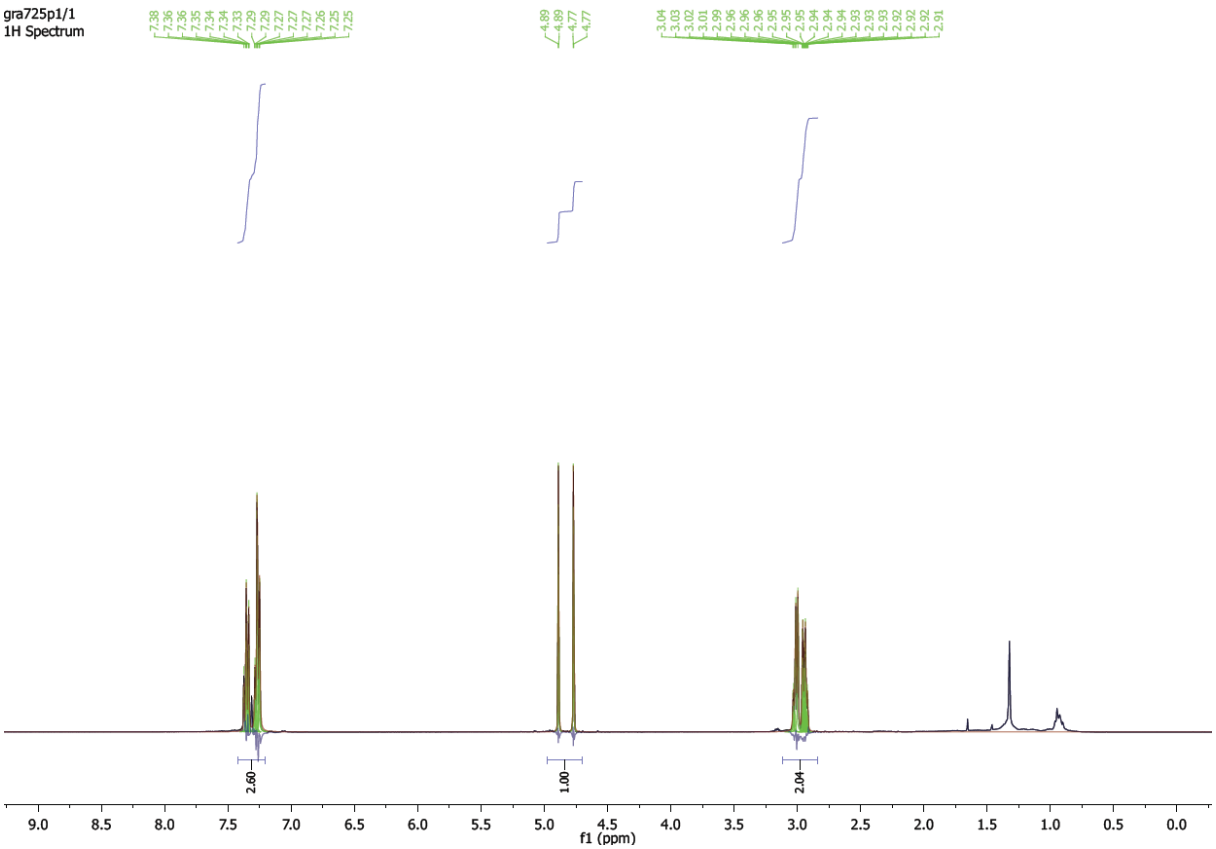


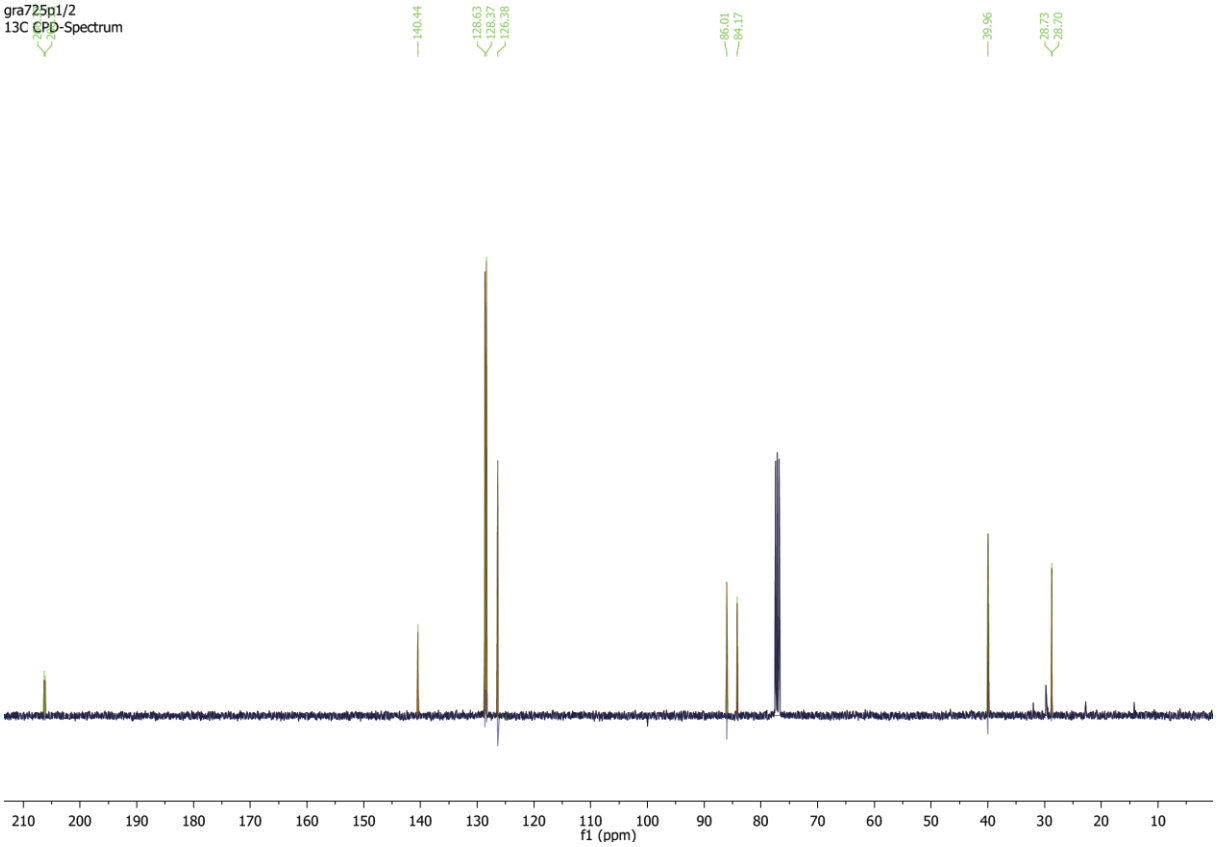

gra724p1/2
13C CPD-Spectrum
208.99
208.81
85.35
83.50
46.73
28.30
26.04
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

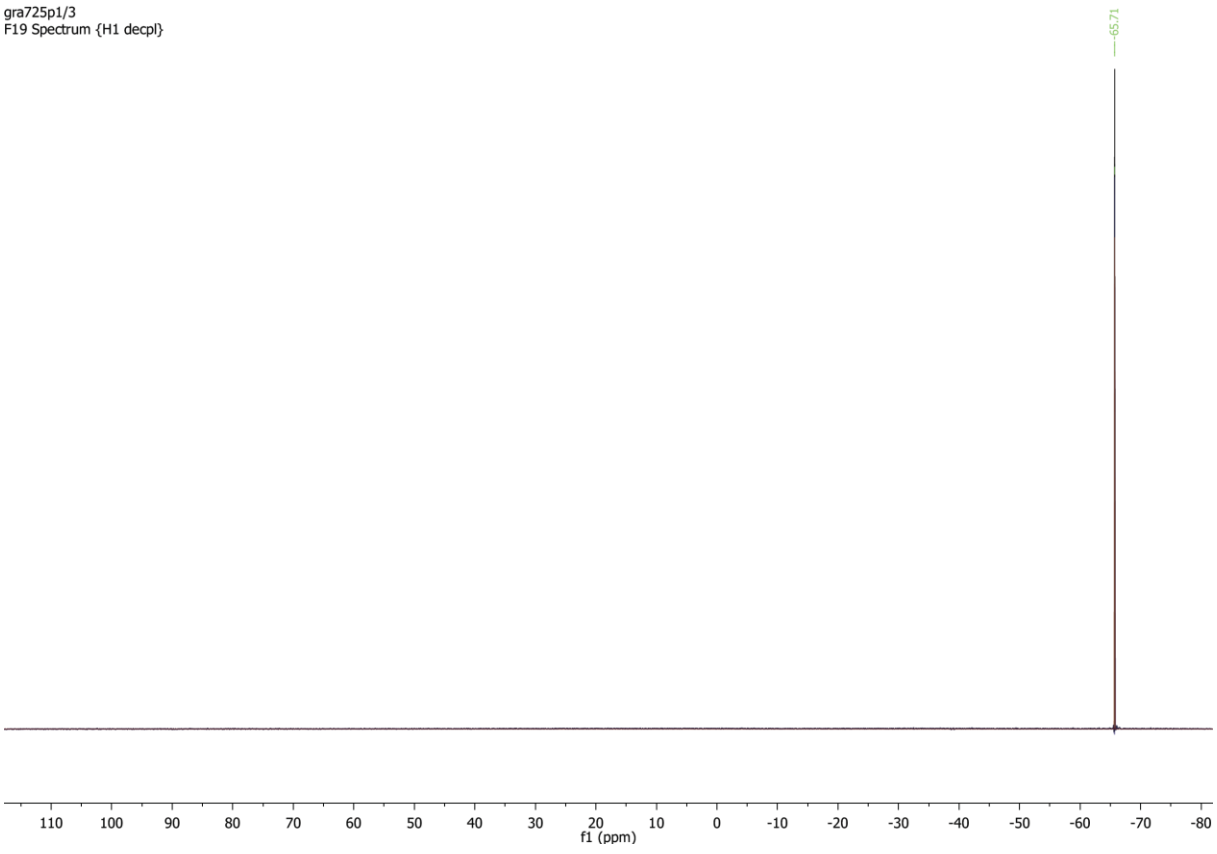

gra724p1/3
F19 Spectrum {H1 decpl}
-65.77
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

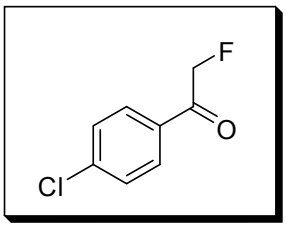


2b

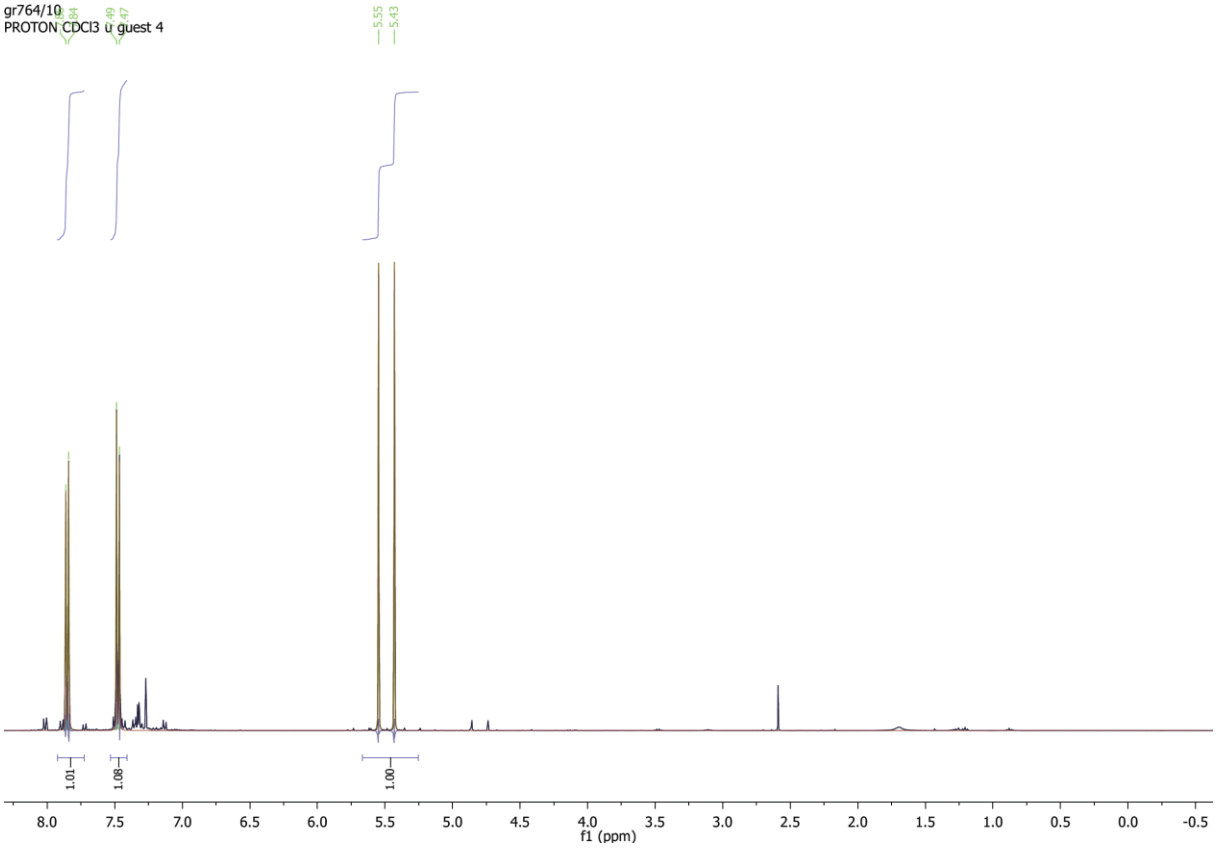


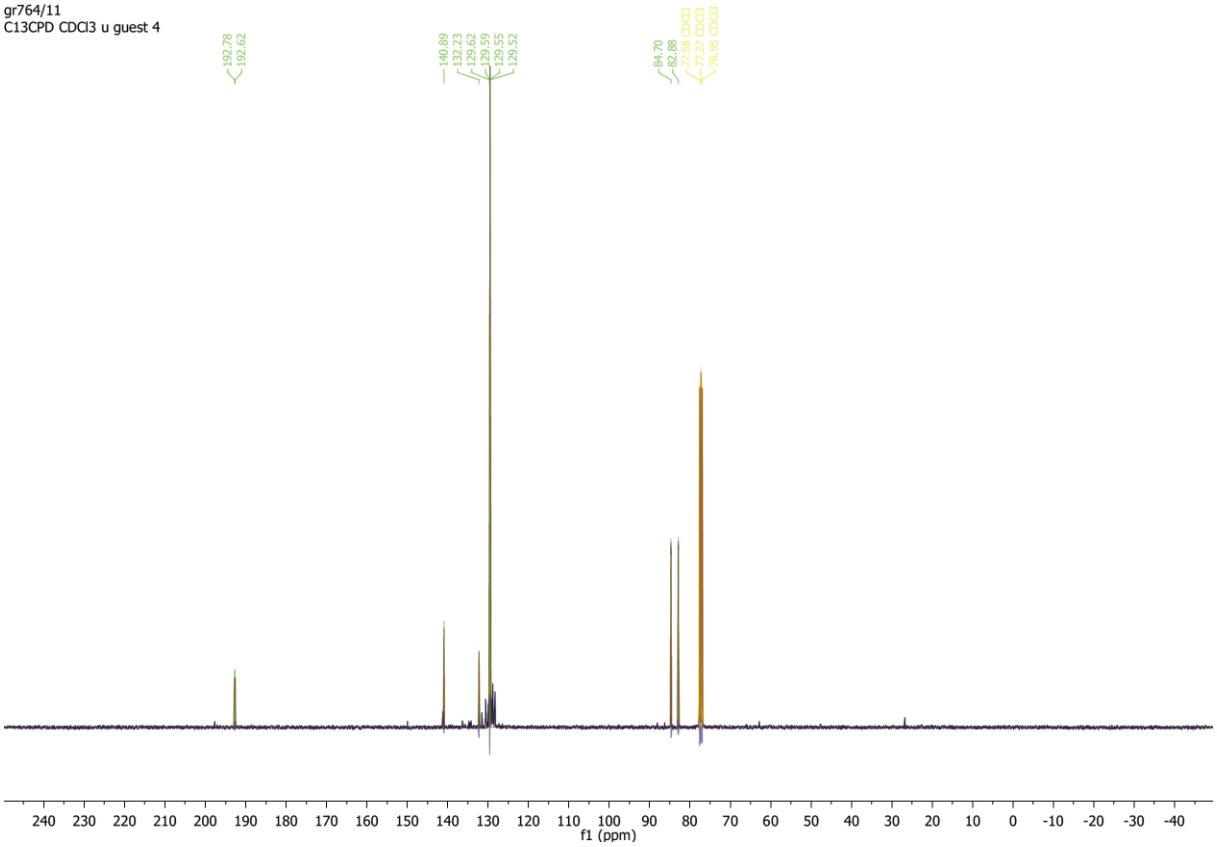

gra725p1/2
13C CPD-Spectrum
140.44
128.63
128.37
126.38
86.01
84.17
39.96
28.73
28.70
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

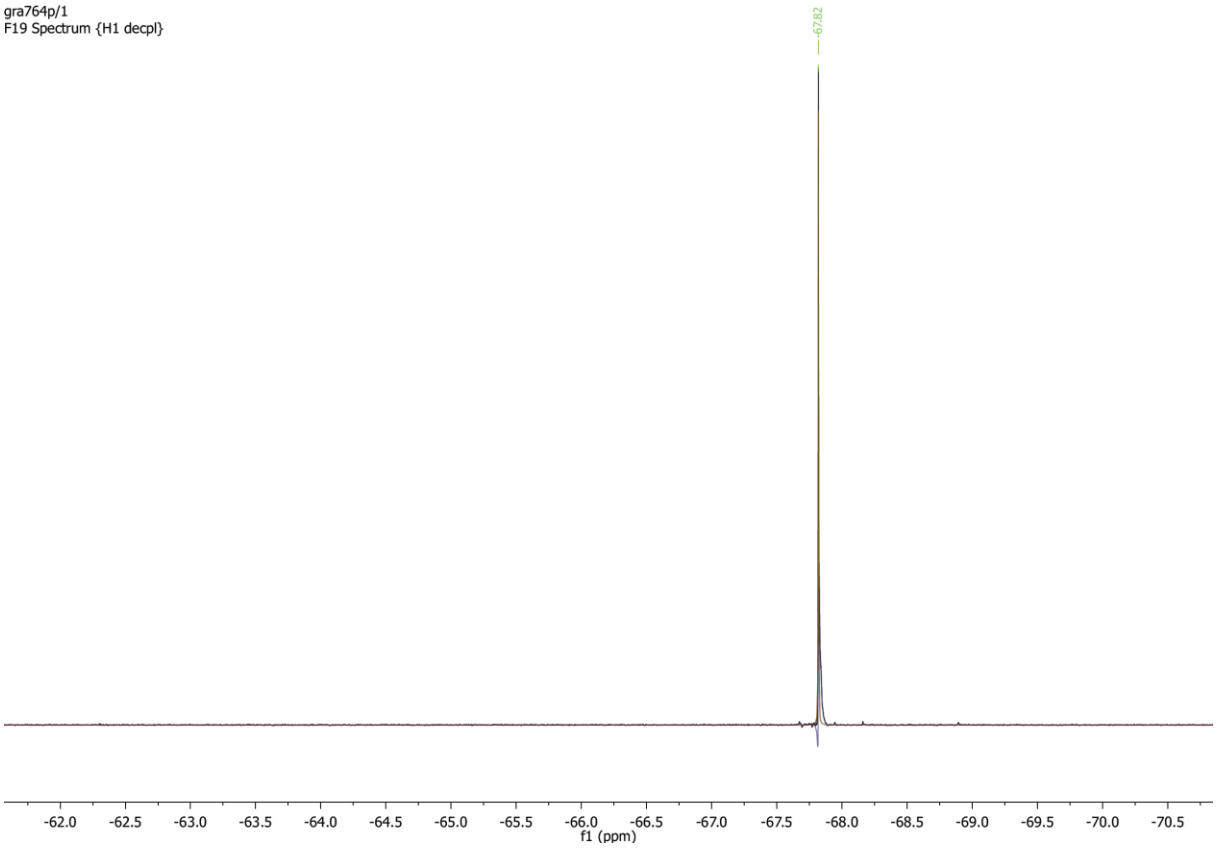

gra725p1/3
F19 Spectrum {H1 decpl}
65.71
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

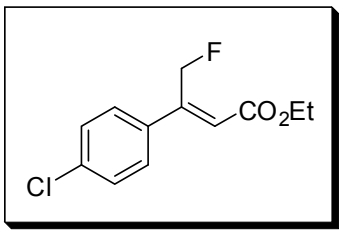

F
O
Cl

2c

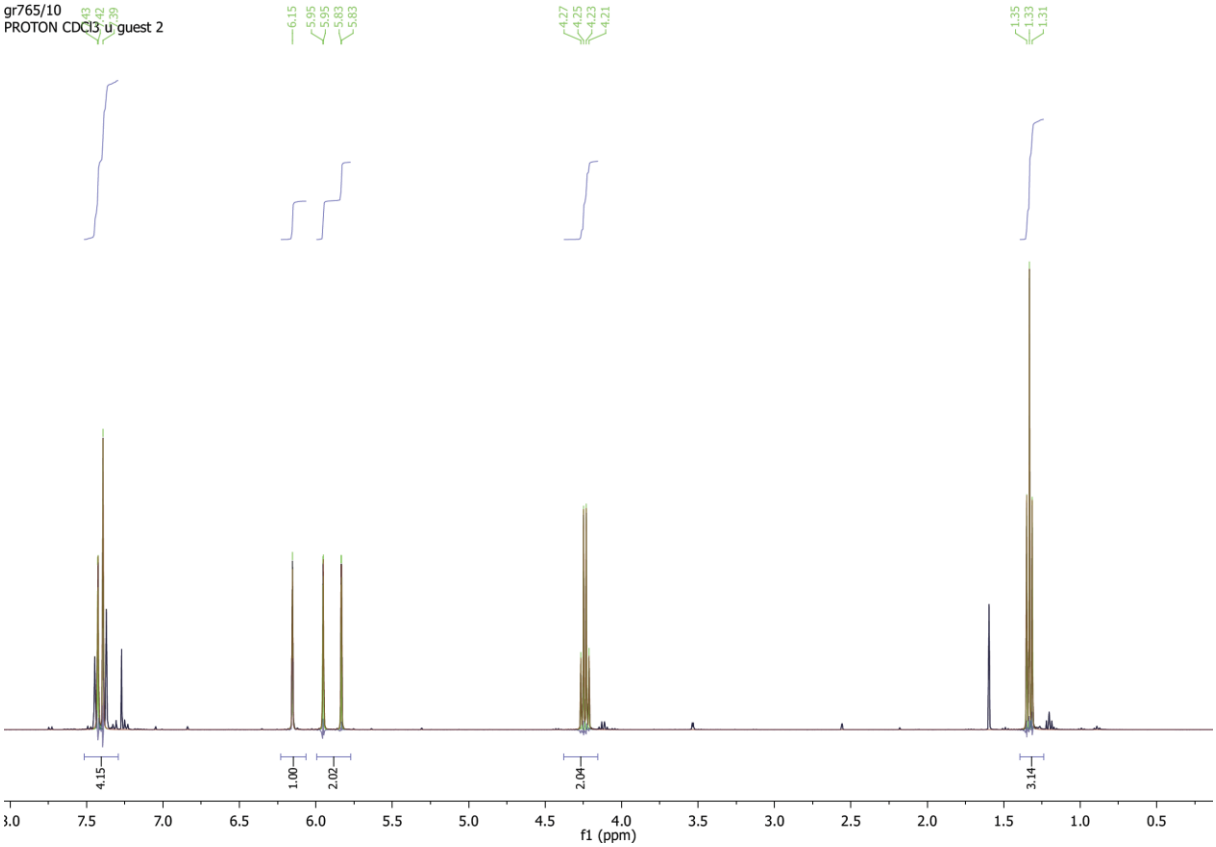

gr764/10
PROTON CDCl3 u guest 4
5.55
5.43
1.01
1.08
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

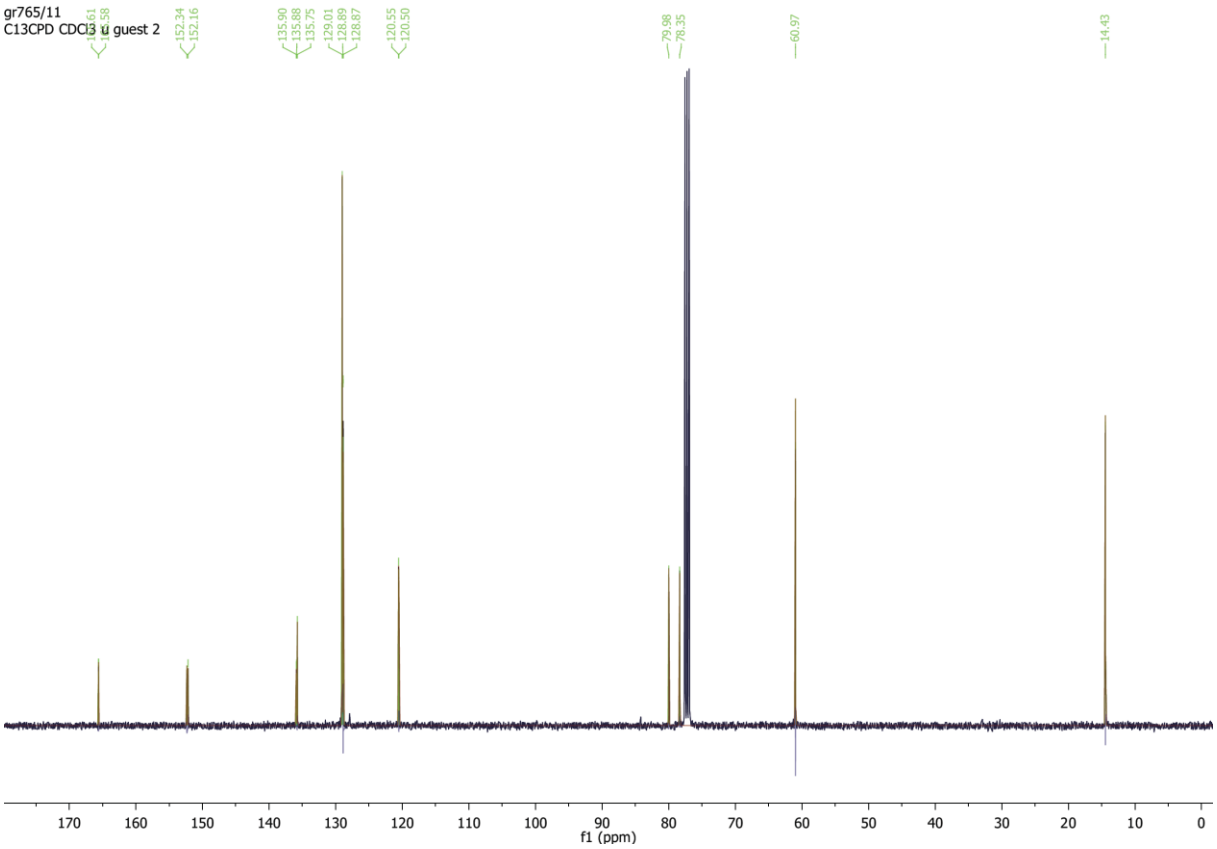

gr764/11
C13CPD CDCl3 u guest 4
192.78
192.62
140.89
132.23
129.62
129.59
129.55
129.52
84.70
82.88
77.58 CDCl3
77.27 CDCl3
76.95 CDCl3
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

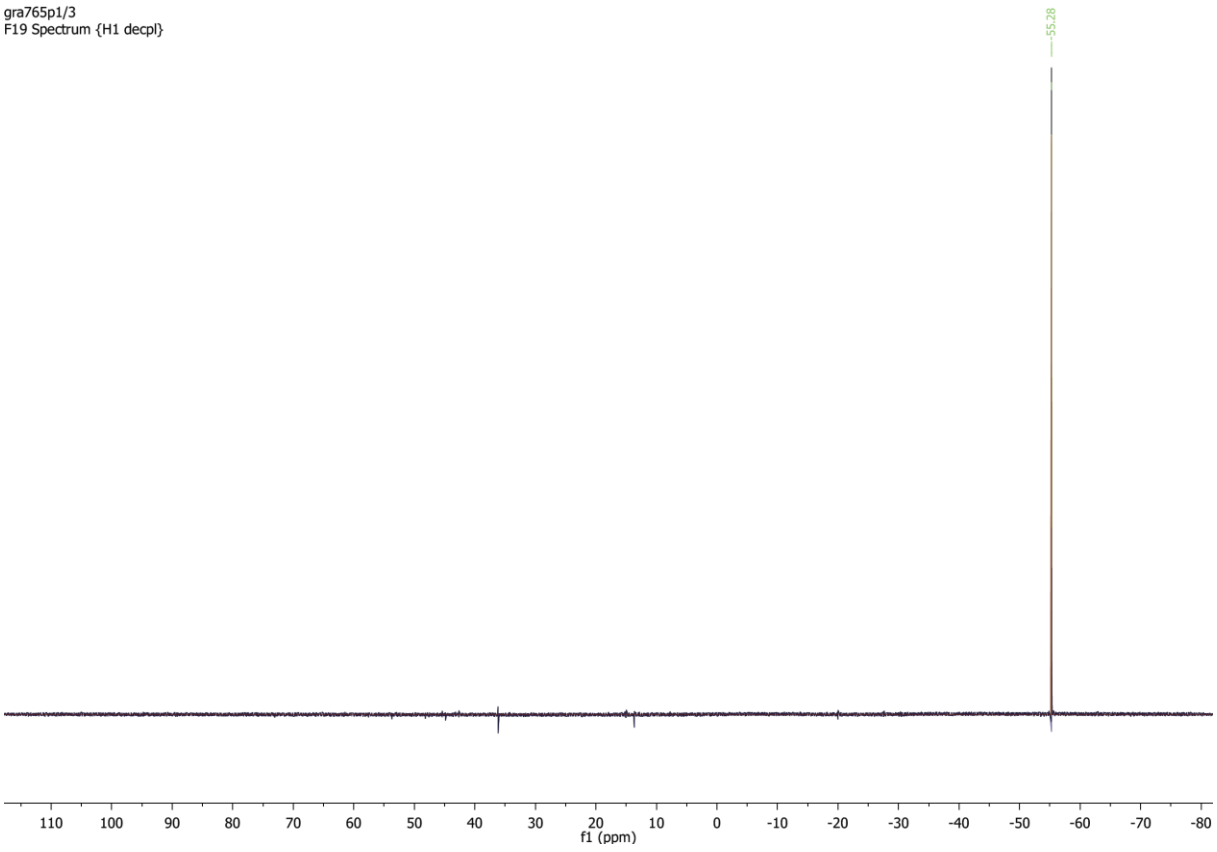

gra764p/1
F19 Spectrum {H1 decpl}
-67.82
-62.0 -62.5 -63.0 -63.5 -64.0 -64.5 -65.0 -65.5 -66.0 -66.5 -67.0 -67.5 -68.0 -68.5 -69.0 -69.5 -70.0 -70.5
f1 (ppm)

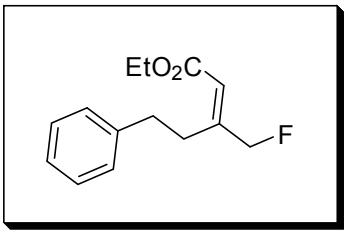

F
CO2Et
Cl

3a

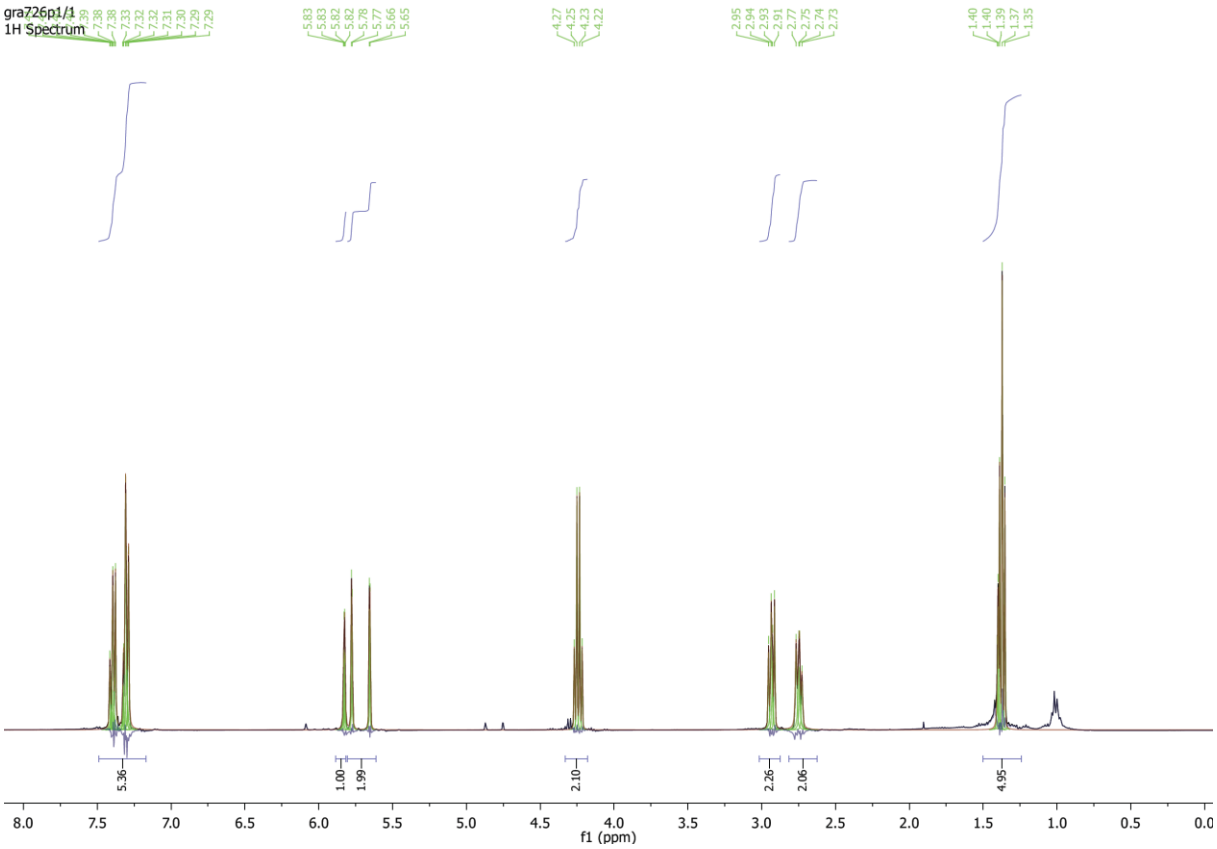

gr765/10
PROTON CDCl3 u guest 2
6.15
5.95
5.95
5.83
5.83
4.27
4.25
4.23
4.21
1.35
1.33
1.31
4.15
1.00
2.02
2.04
3.14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

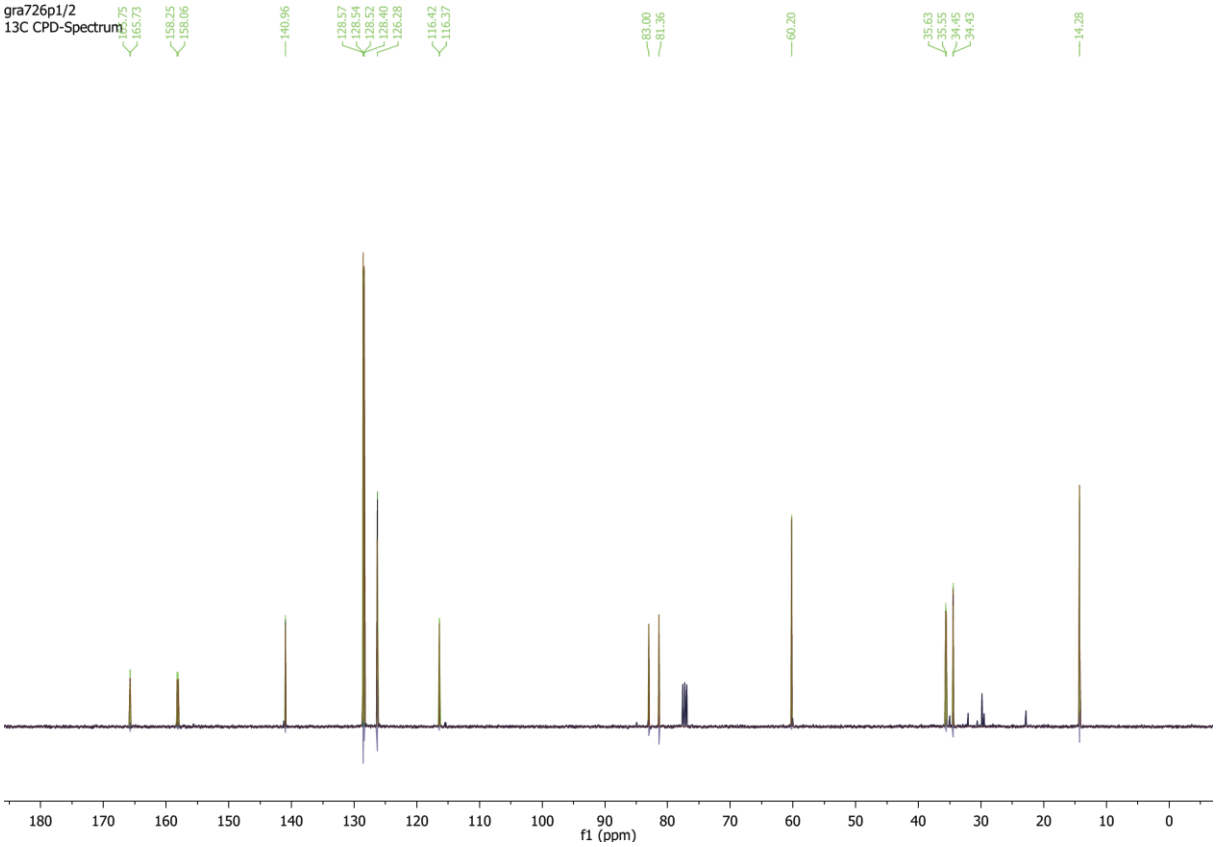

gr765/11
C13CPD CDCl3 u guest 2
152.34
152.16
135.90
135.88
135.75
129.01
128.89
128.87
120.55
120.50
79.98
78.35
60.97
14.43
f1 (ppm)

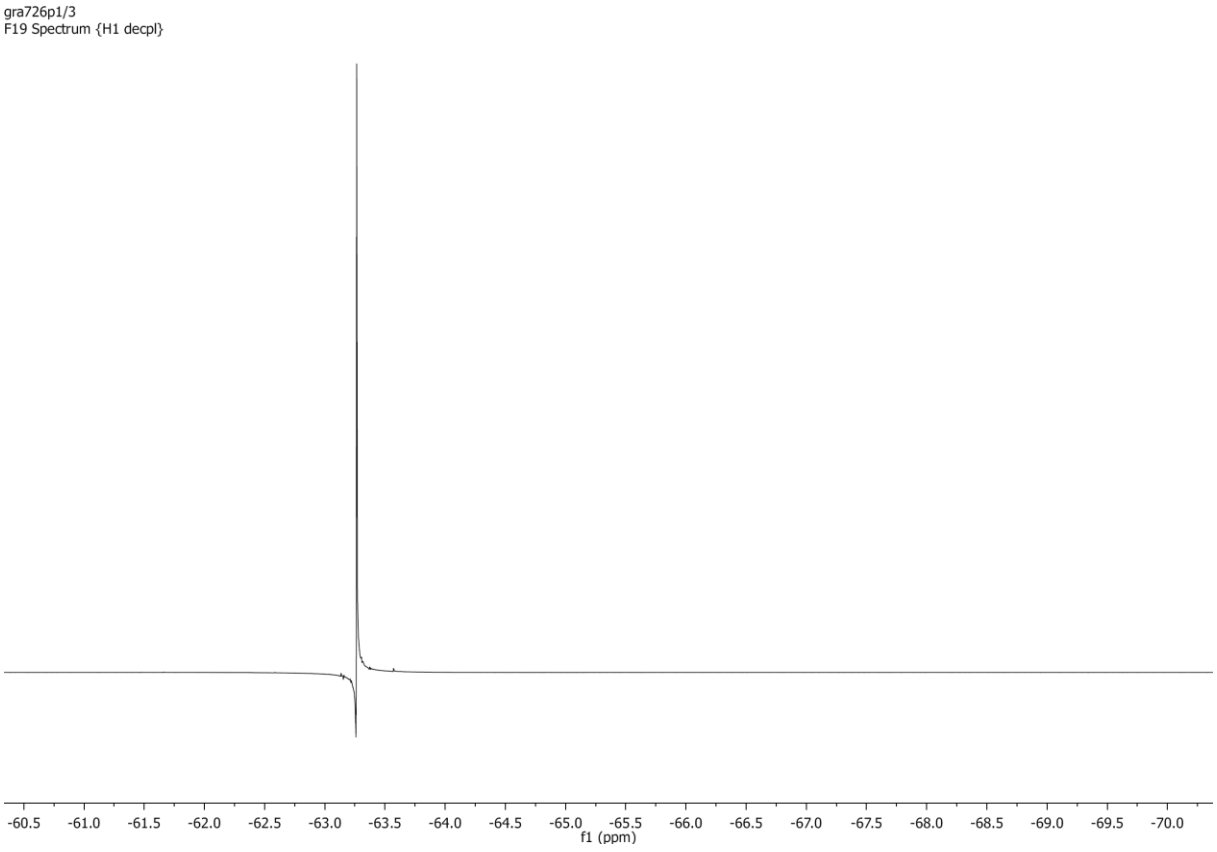

gra765p1/3
F19 Spectrum {H1 decpl}
55.28
f1 (ppm)

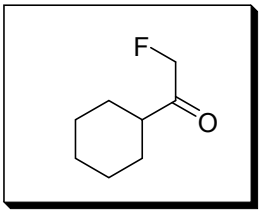

EtO2C
F

3b

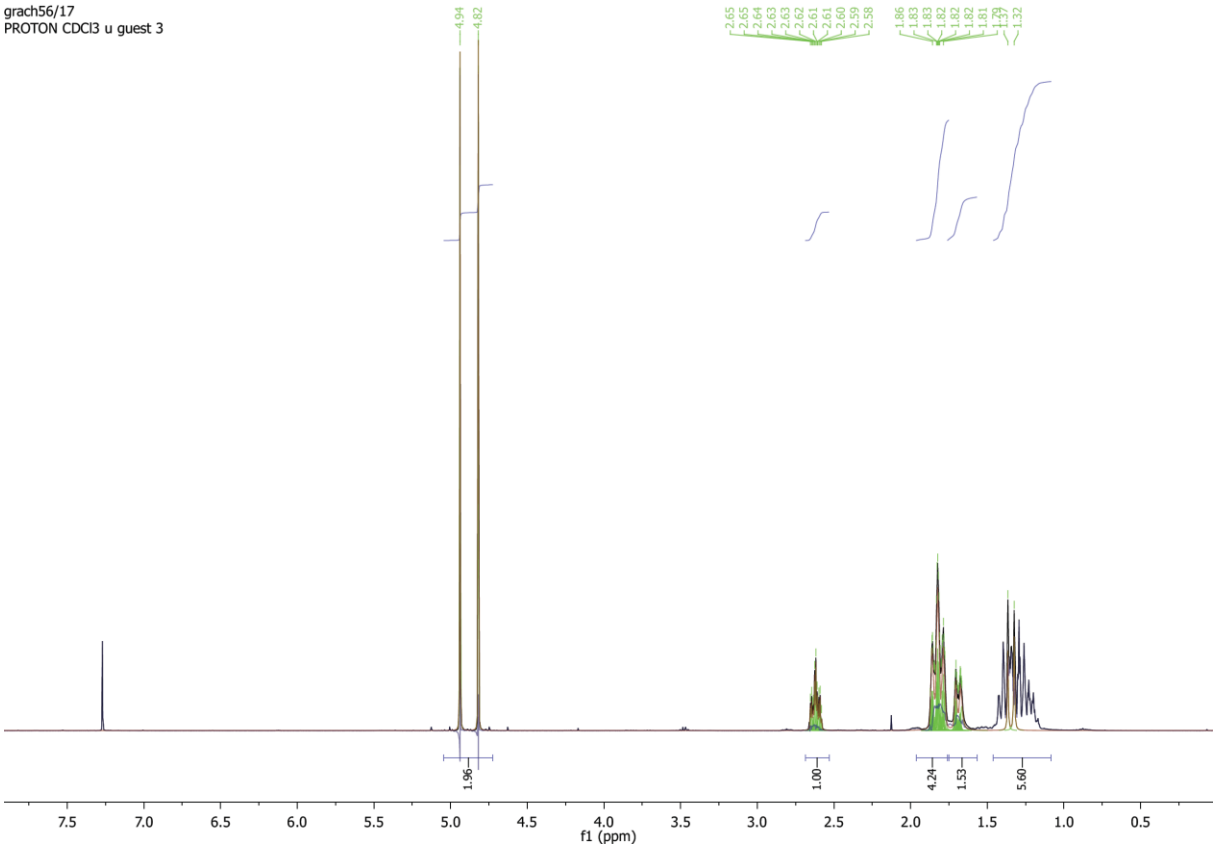

gra726p1/1
1H Spectrum
7.39
7.38
7.38
7.33
7.32
7.32
7.31
7.30
7.29
7.29
5.83
5.83
5.82
5.82
5.78
5.77
5.66
5.65
4.27
4.25
4.23
4.22
2.95
2.94
2.93
2.91
2.77
2.75
2.74
2.73
1.40
1.40
1.39
1.37
1.35
5.36
1.00
1.99
2.10
2.26
2.06
4.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

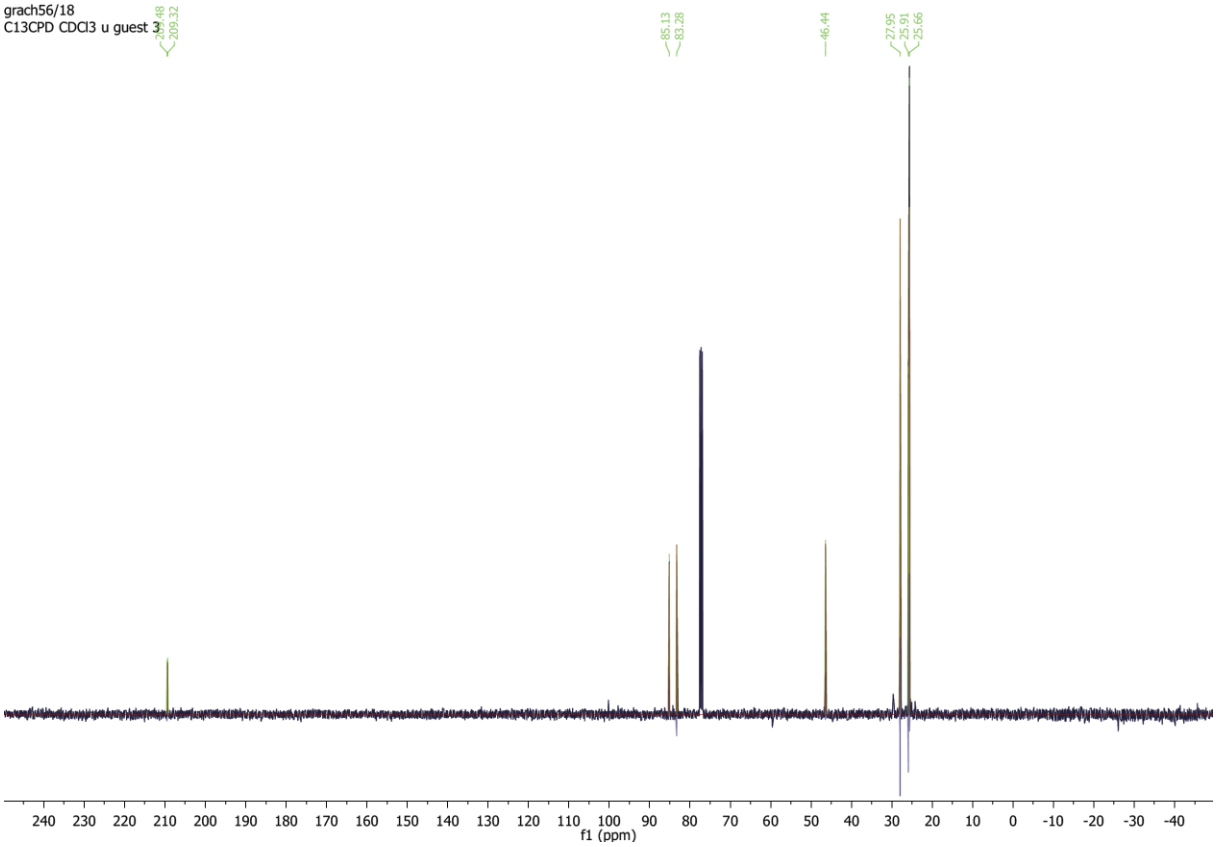

gra726p1/2
13C CPD-Spectrum
165.75
165.73
158.25
158.06
140.96
128.57
128.54
128.52
128.40
126.28
116.42
116.37
83.00
81.36
60.20
35.63
35.55
34.45
34.43
14.28
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

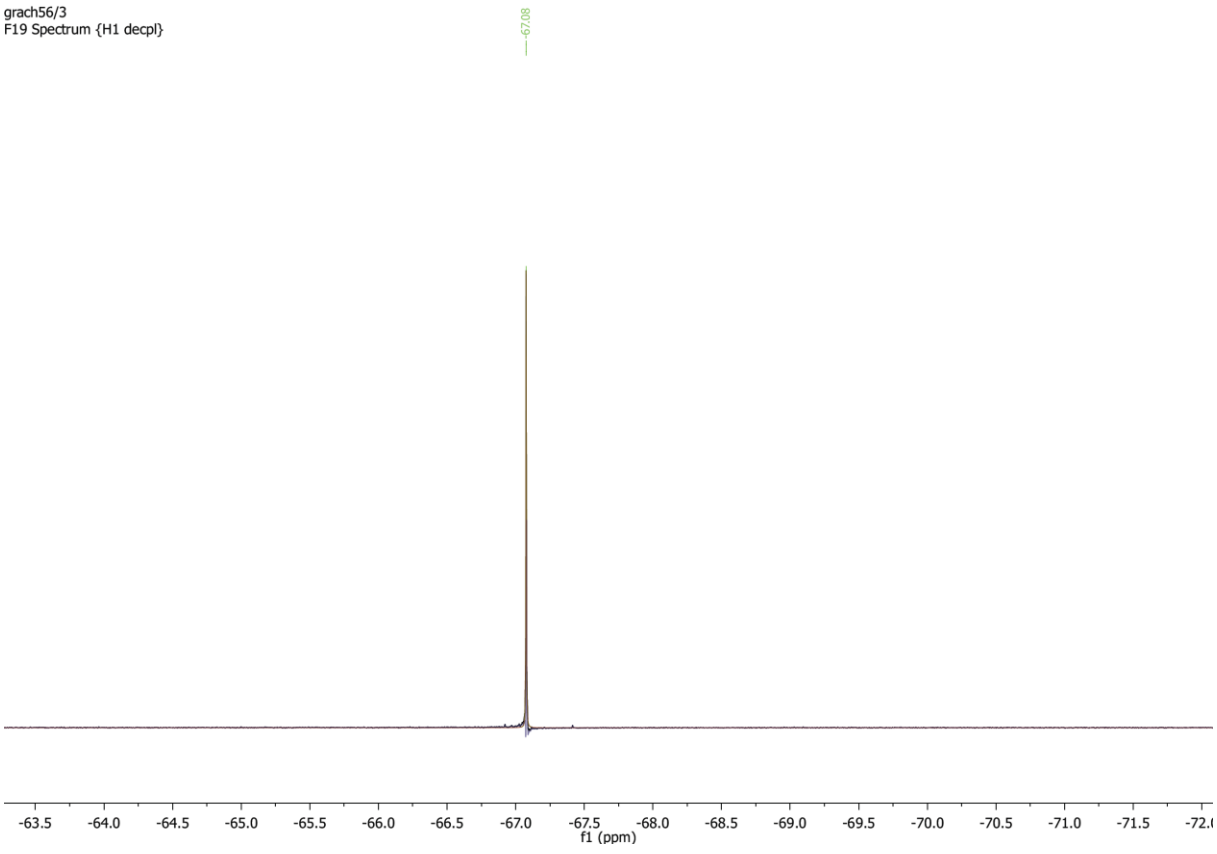

gra726p1/3
F19 Spectrum {H1 decpl}
-60.5 -61.0 -61.5 -62.0 -62.5 -63.0 -63.5 -64.0 -64.5 -65.0 -65.5 -66.0 -66.5 -67.0 -67.5 -68.0 -68.5 -69.0 -69.5 -70.0
f1 (ppm)

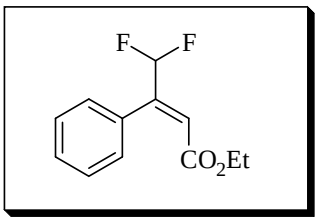

F
O

2d

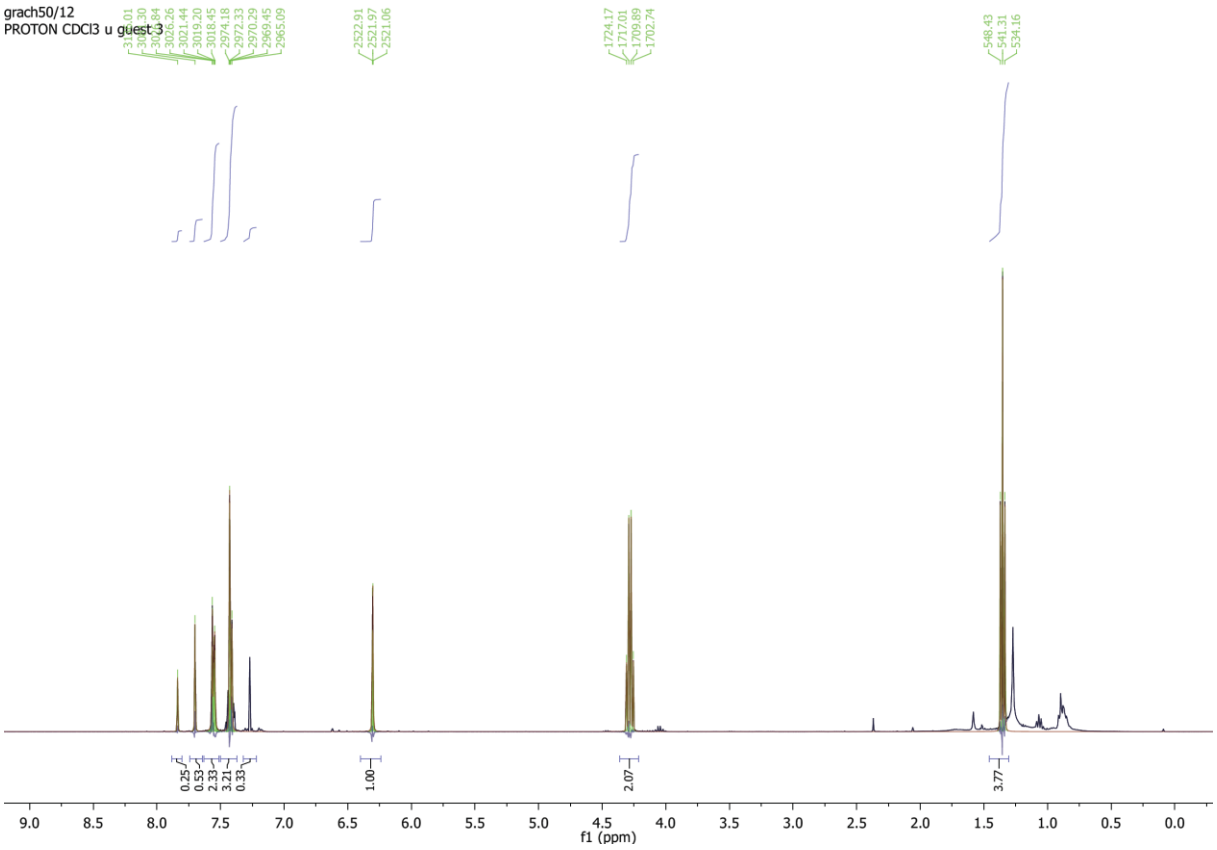

grach56/17
PROTON CDCl3 u guest 3
4.94
4.82
2.65
2.65
2.64
2.63
2.63
2.62
2.61
2.61
2.60
2.59
2.58
1.86
1.83
1.83
1.82
1.82
1.82
1.81
1.79
1.37
1.32
1.96
1.00
4.24
1.53
5.60
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

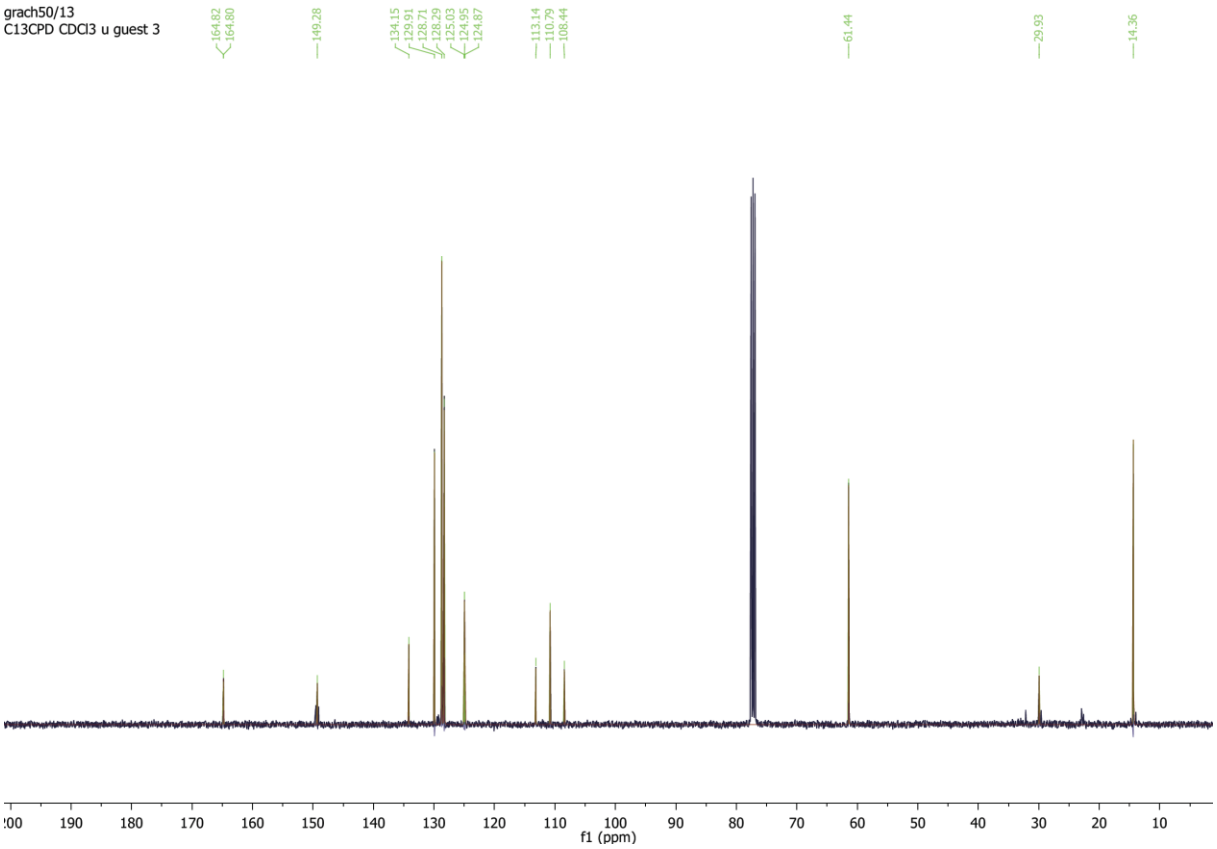

grach56/18
C13CPD CDCl3 u guest 3
209.48
209.32
85.13
83.28
46.44
27.95
25.91
25.66
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)

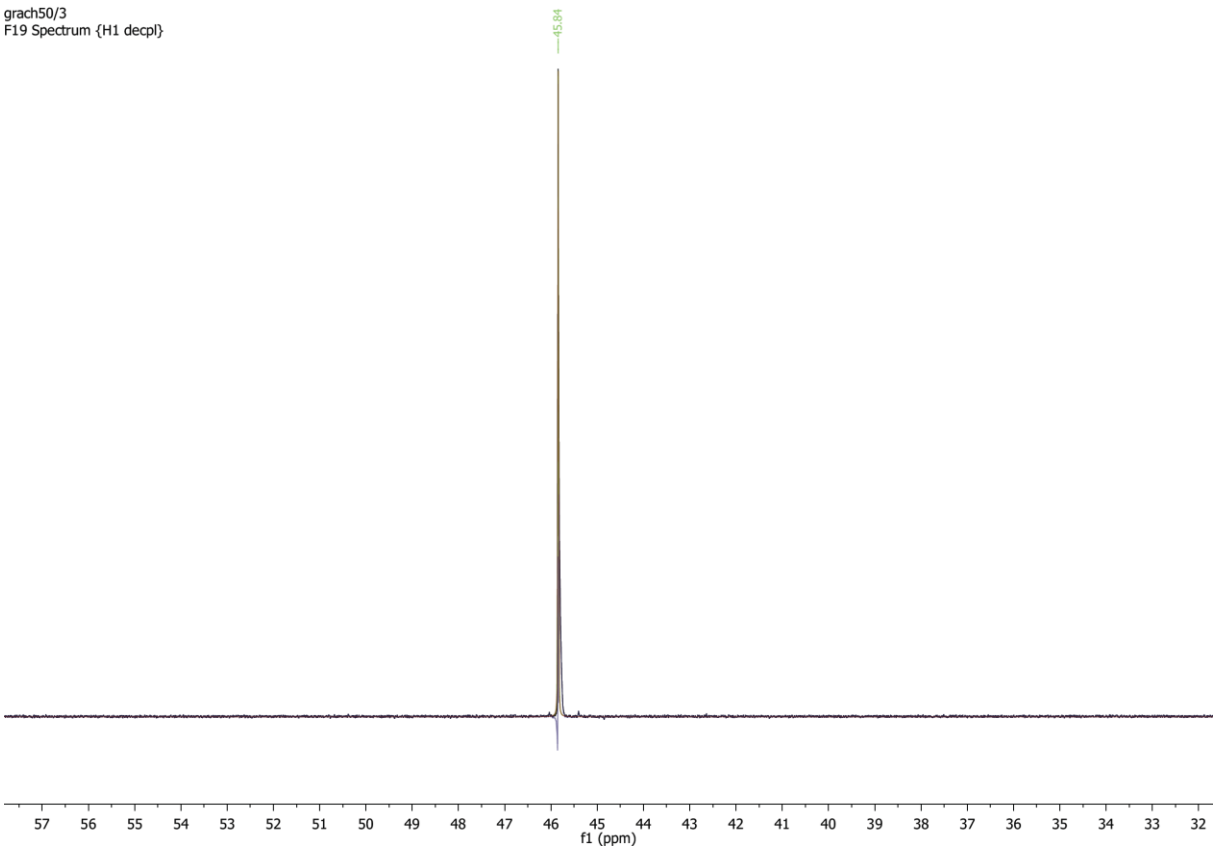

grach56/3
F19 Spectrum {H1 decpl}
-67.08
-63.5 -64.0 -64.5 -65.0 -65.5 -66.0 -66.5 -67.0 -67.5 -68.0 -68.5 -69.0 -69.5 -70.0 -70.5 -71.0 -71.5
f1 (ppm)

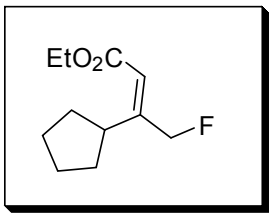


3f

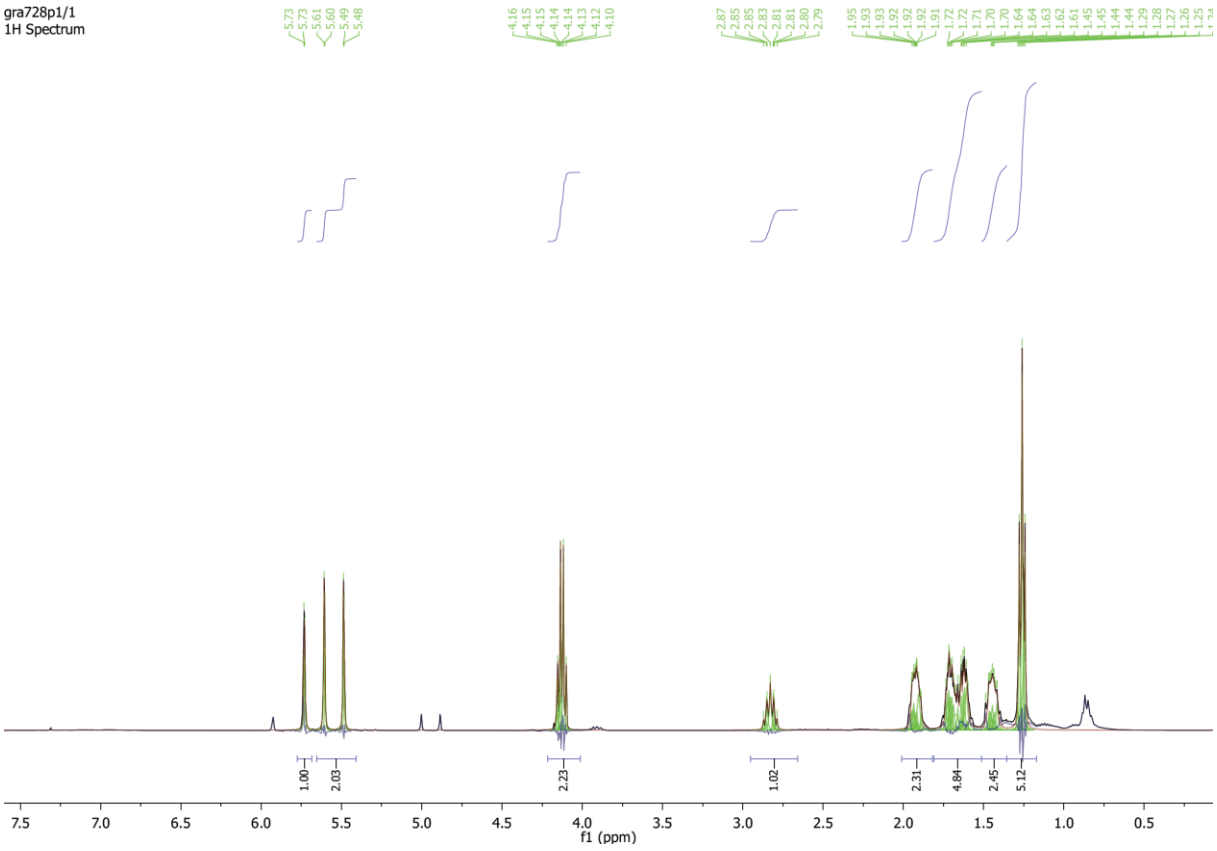


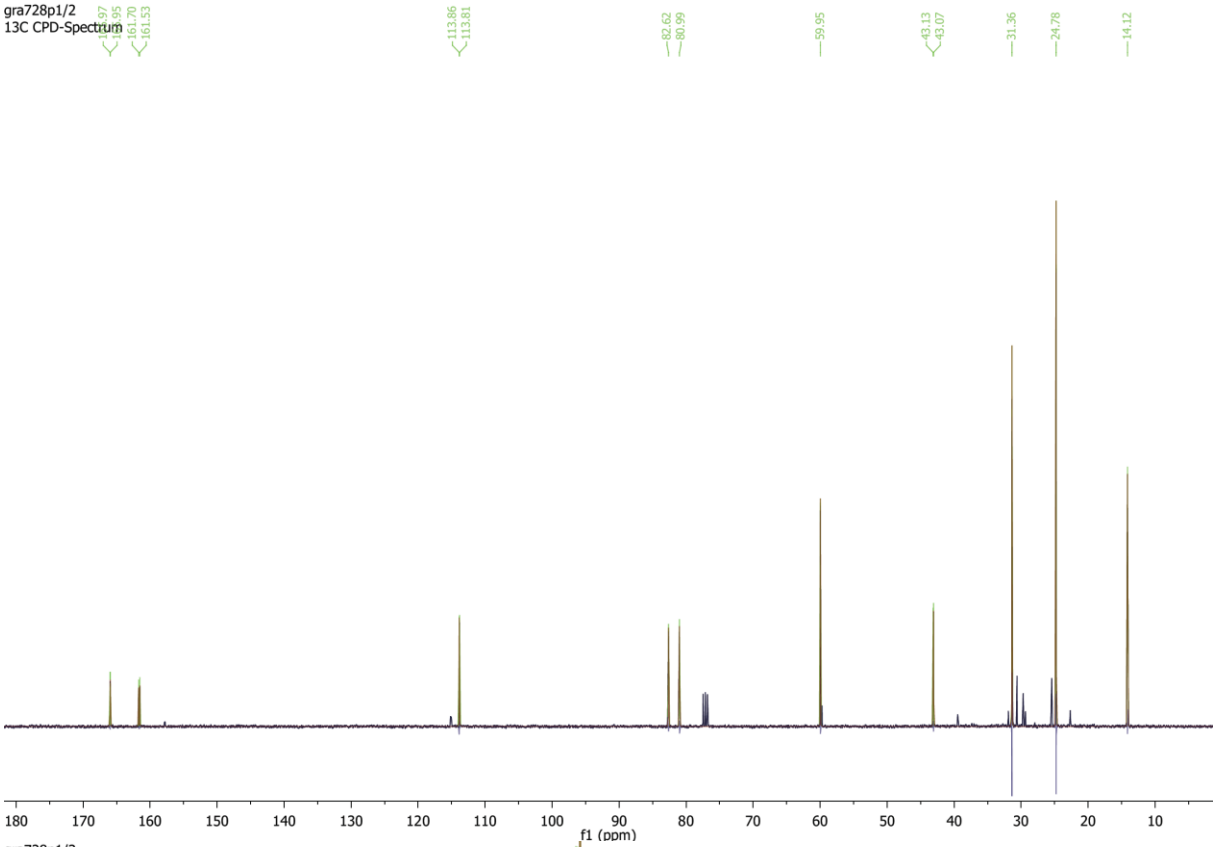

grach50/13
C13CPD CDCl3 u guest 3
164.82
164.80
149.28
134.15
129.91
128.71
128.29
125.03
124.95
124.87
113.14
110.79
108.44
61.44
29.93
14.36
f1 (ppm)

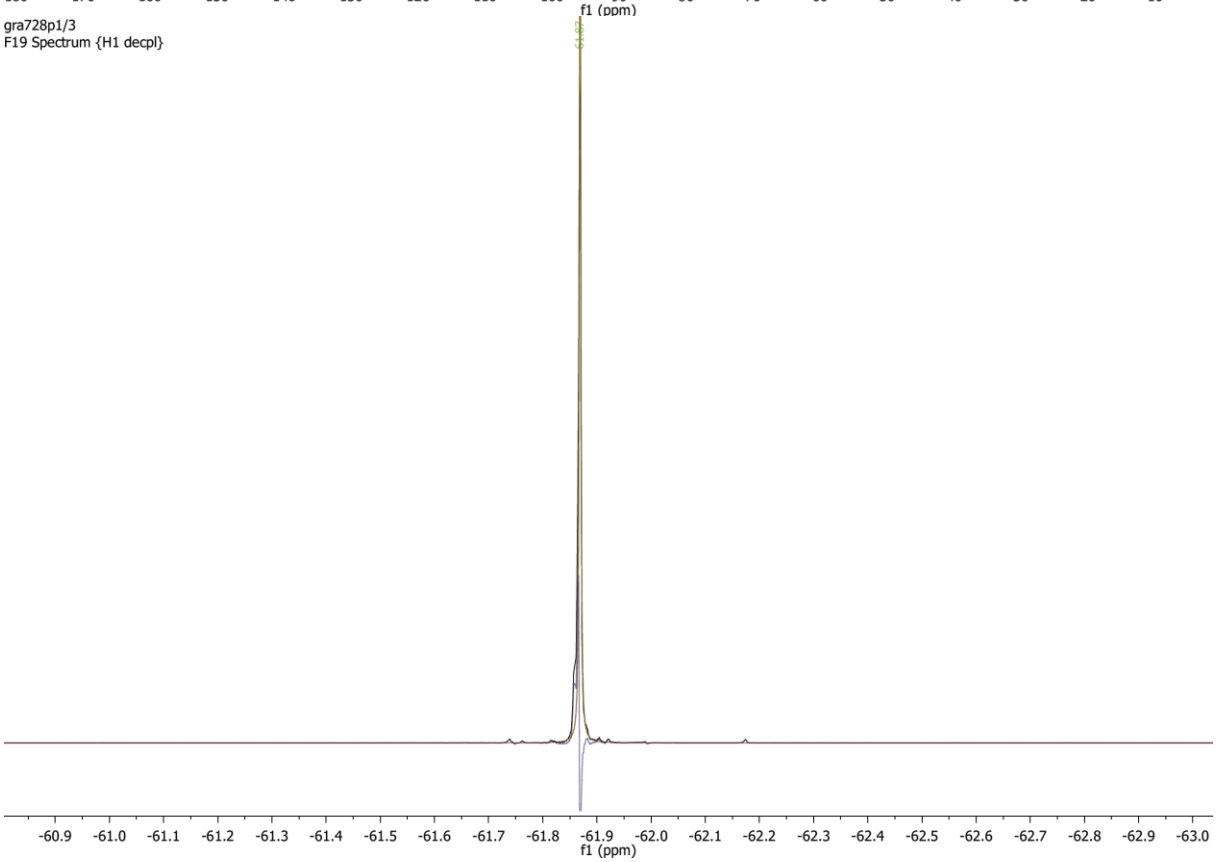

grach50/3
F19 Spectrum {H1 decpl}
45.84
f1 (ppm)

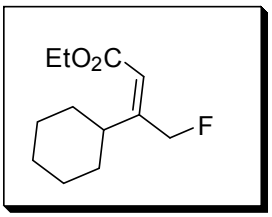


3c

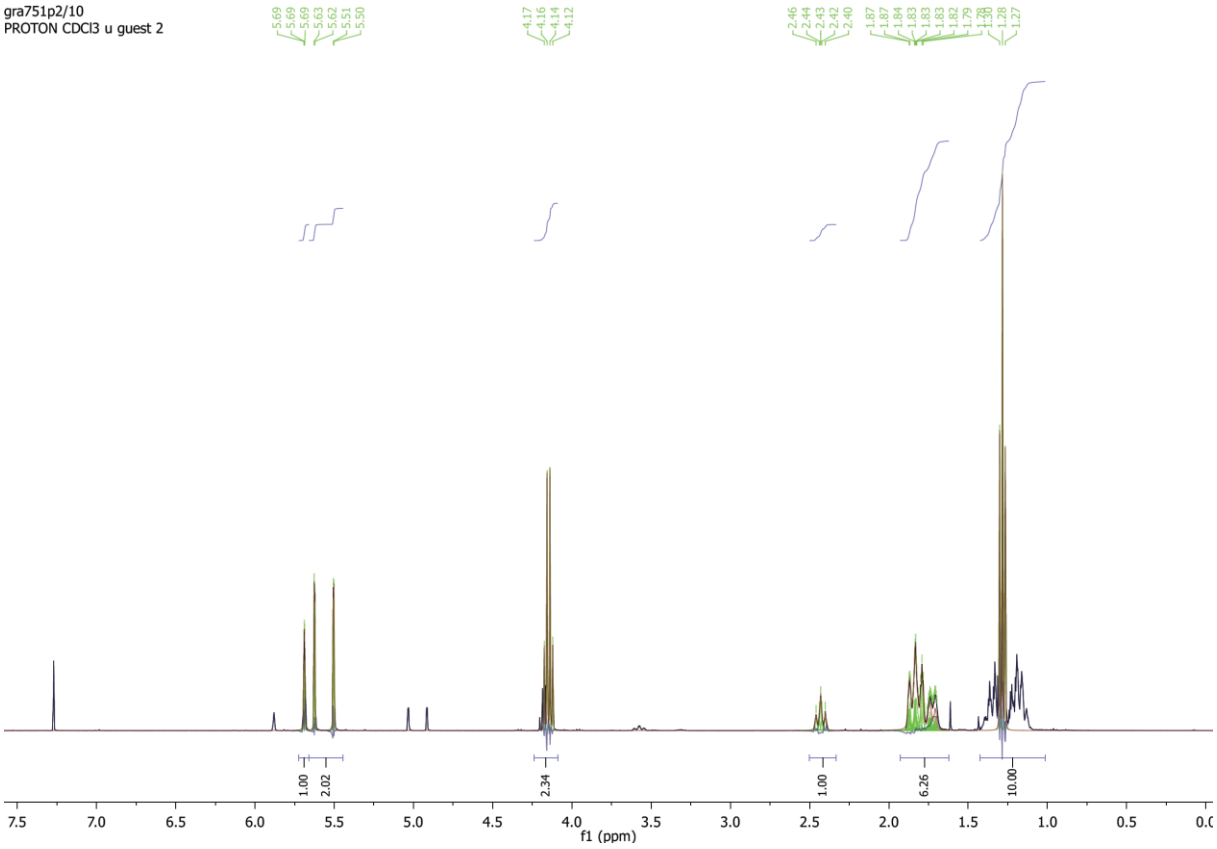


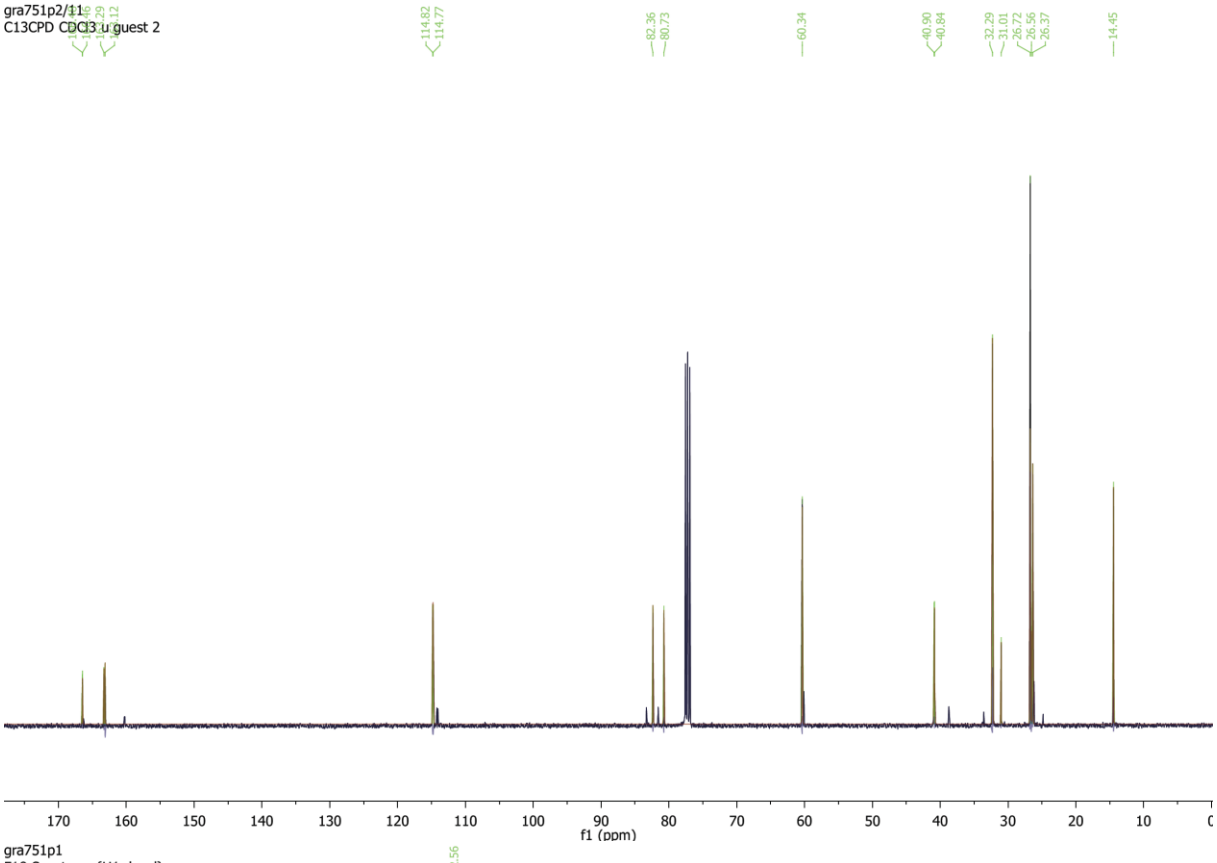

gra728p1/2
13C CPD-Spectrum
161.97
161.95
161.70
161.53
113.86
113.81
82.62
80.99
59.95
43.13
43.07
31.36
24.78
14.12
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

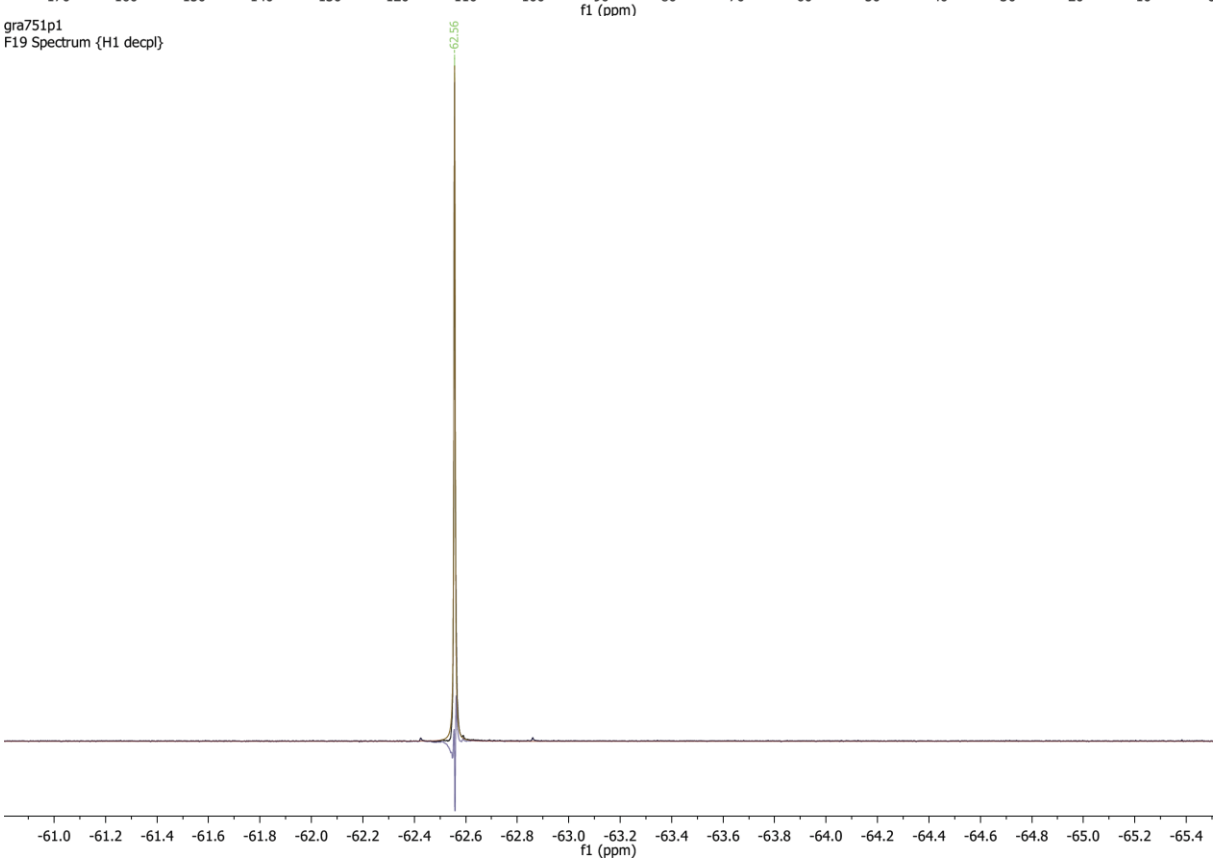

gra728p1/3
F19 Spectrum {H1 decpl}
-61.827
-60.9 -61.0 -61.1 -61.2 -61.3 -61.4 -61.5 -61.6 -61.7 -61.8 -61.9 -62.0 -62.1 -62.2 -62.3 -62.4 -62.5 -62.6 -62.7 -62.8 -62.9 -63.0
f1 (ppm)

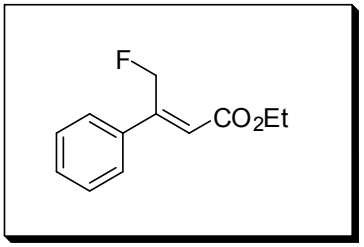

EtO2C
F

3d

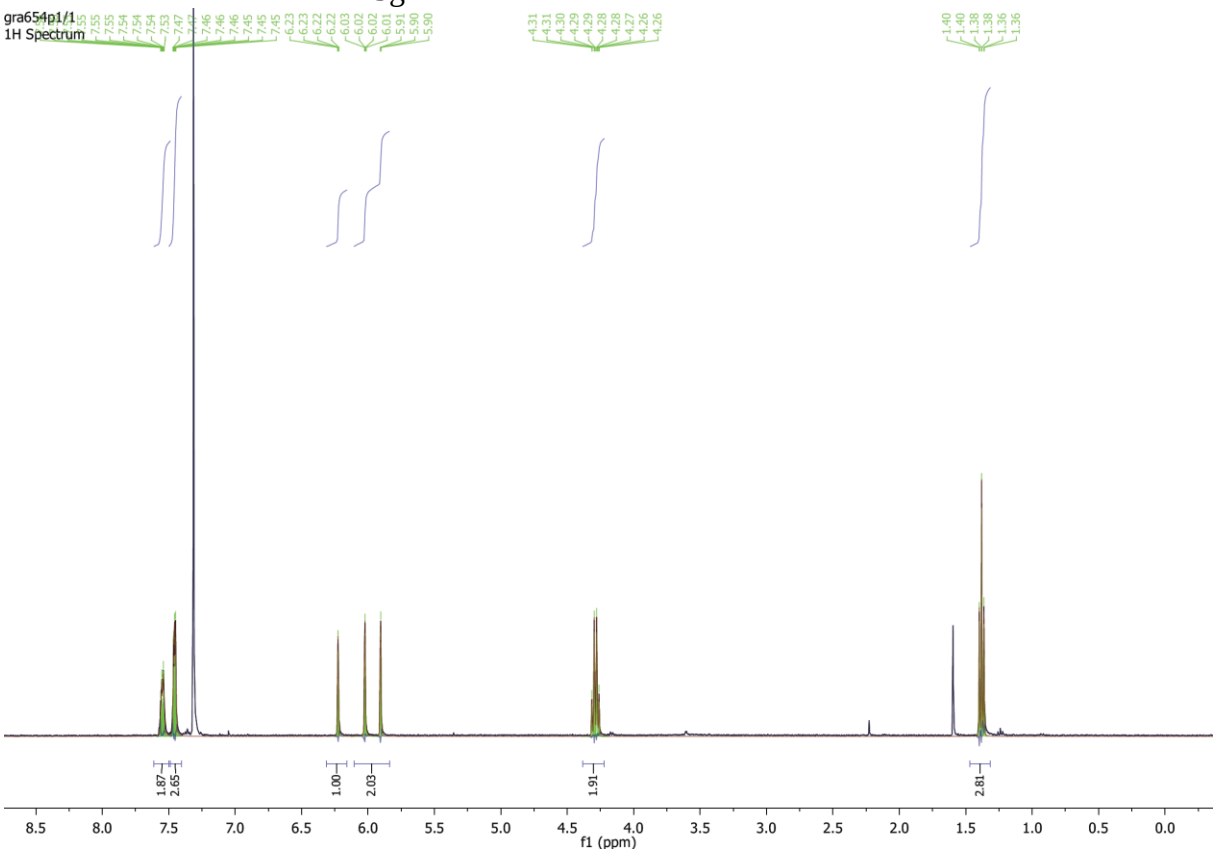

gra751p2/10
PROTON CDCl3 u guest 2
5.69
5.69
5.69
5.63
5.62
5.51
5.50
4.17
4.16
4.14
4.12
2.46
2.44
2.43
2.42
2.40
1.87
1.87
1.84
1.83
1.83
1.83
1.82
1.79
1.38
1.30
1.28
1.27
1.00
2.02
2.34
1.00
6.26
10.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

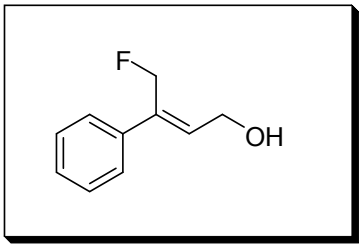

gra751p2/11
C13CPD CDCl3 u guest 2
114.82
114.77
82.36
80.73
60.34
40.90
40.84
32.29
31.01
26.72
26.56
26.37
14.45
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


gra751p1
F19 Spectrum {H1 decpl}
-62.56
-61.0 -61.2 -61.4 -61.6 -61.8 -62.0 -62.2 -62.4 -62.6 -62.8 -63.0 -63.2 -63.4 -63.6 -63.8 -64.0 -64.2 -64.4 -64.6 -64.8 -65.0 -65.2 -65.4
f1 (ppm)

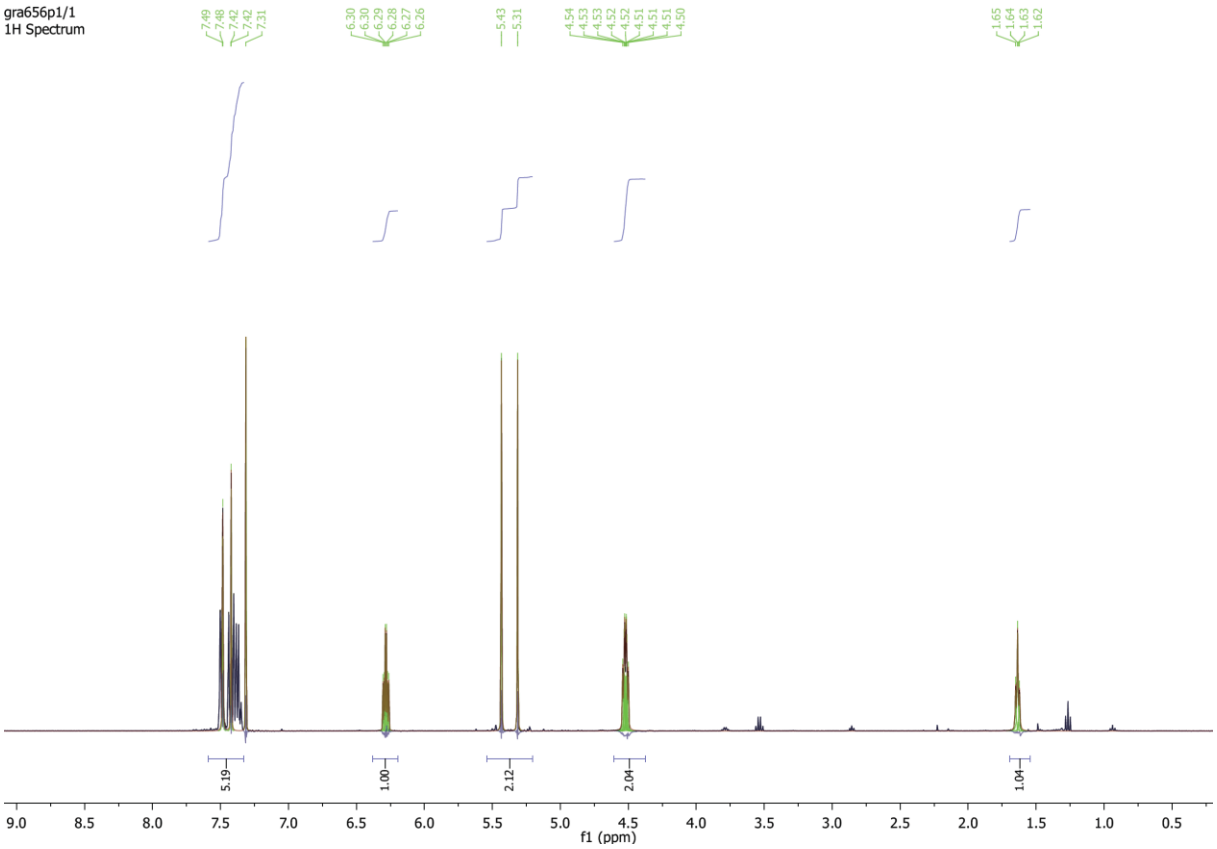

F
CO2Et

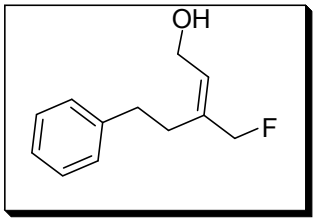
3g


gra654p1/1
1H Spectrum
1.87
2.65
1.00
2.03
1.91
2.81
f1 (ppm)

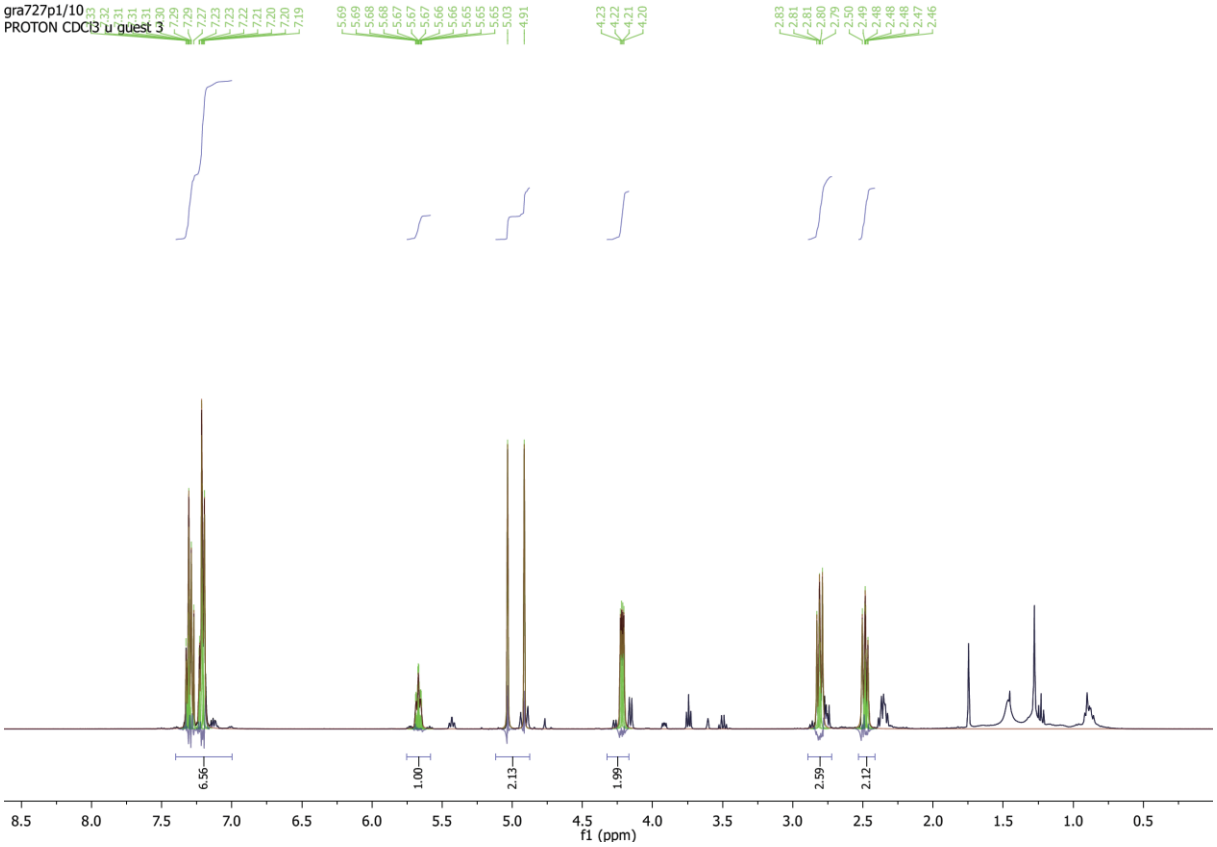

F
OH

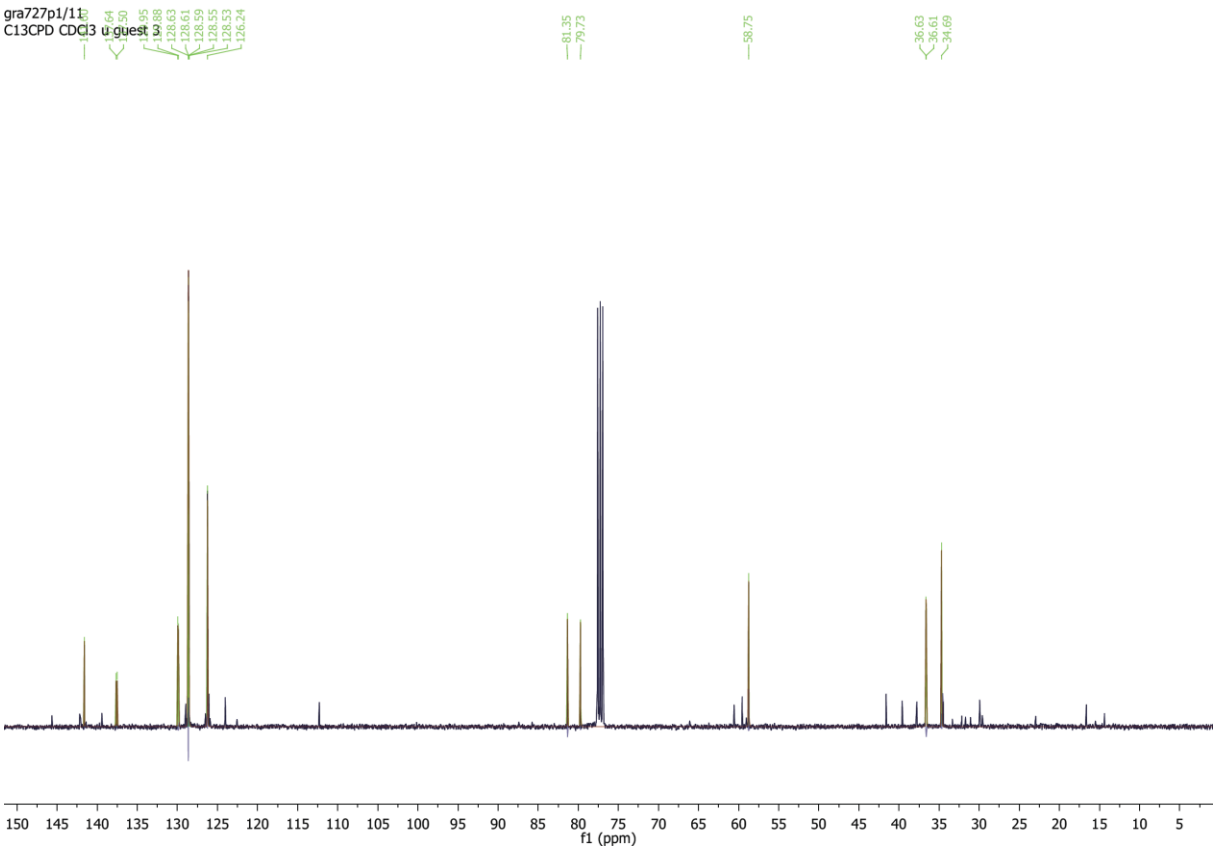
4g

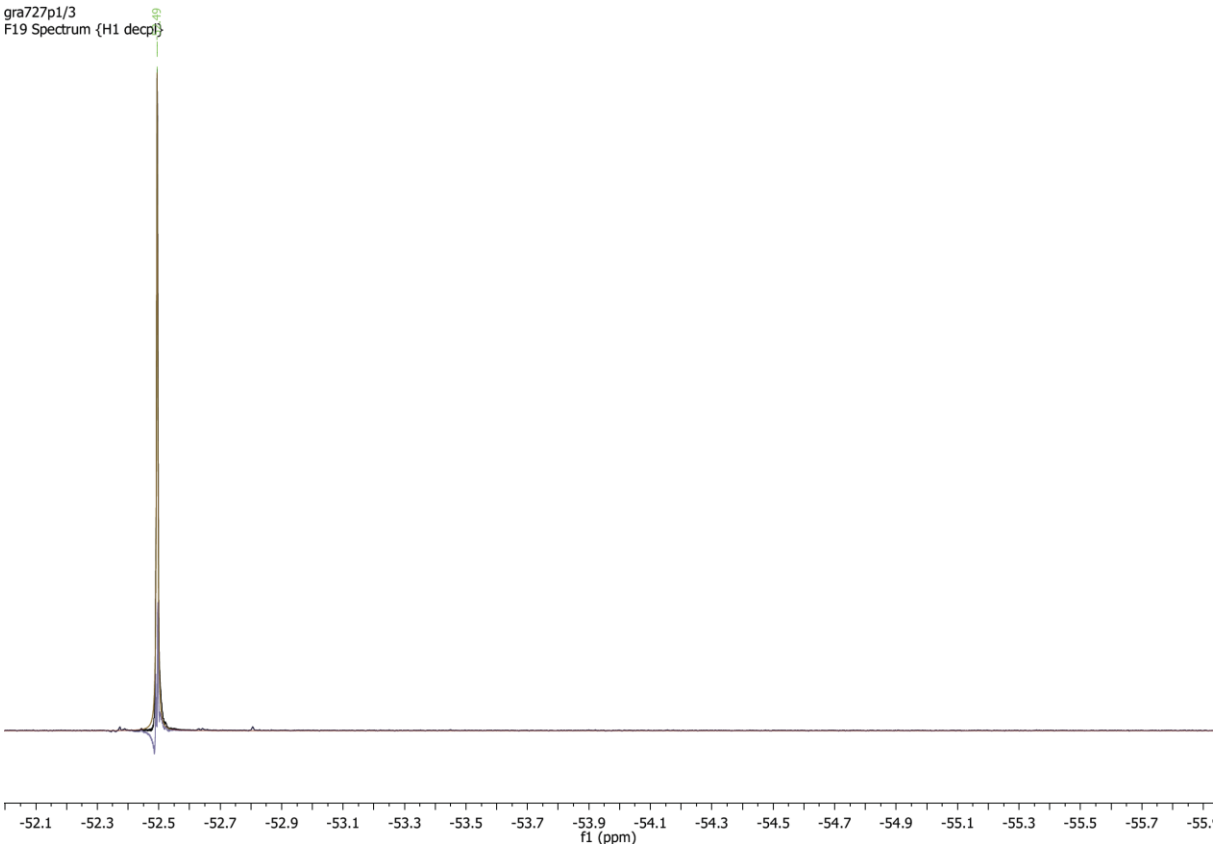

gra656p1/1
1H Spectrum
7.49
7.48
7.42
7.42
7.31
6.30
6.30
6.29
6.28
6.27
6.26
5.43
5.31
4.54
4.53
4.53
4.52
4.52
4.51
4.51
4.51
4.50
1.65
1.64
1.63
1.62
5.19
1.00
2.12
2.04
1.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

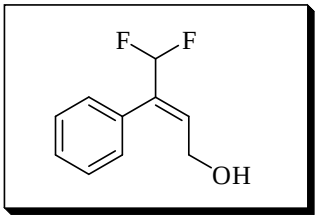

OH
F

4a

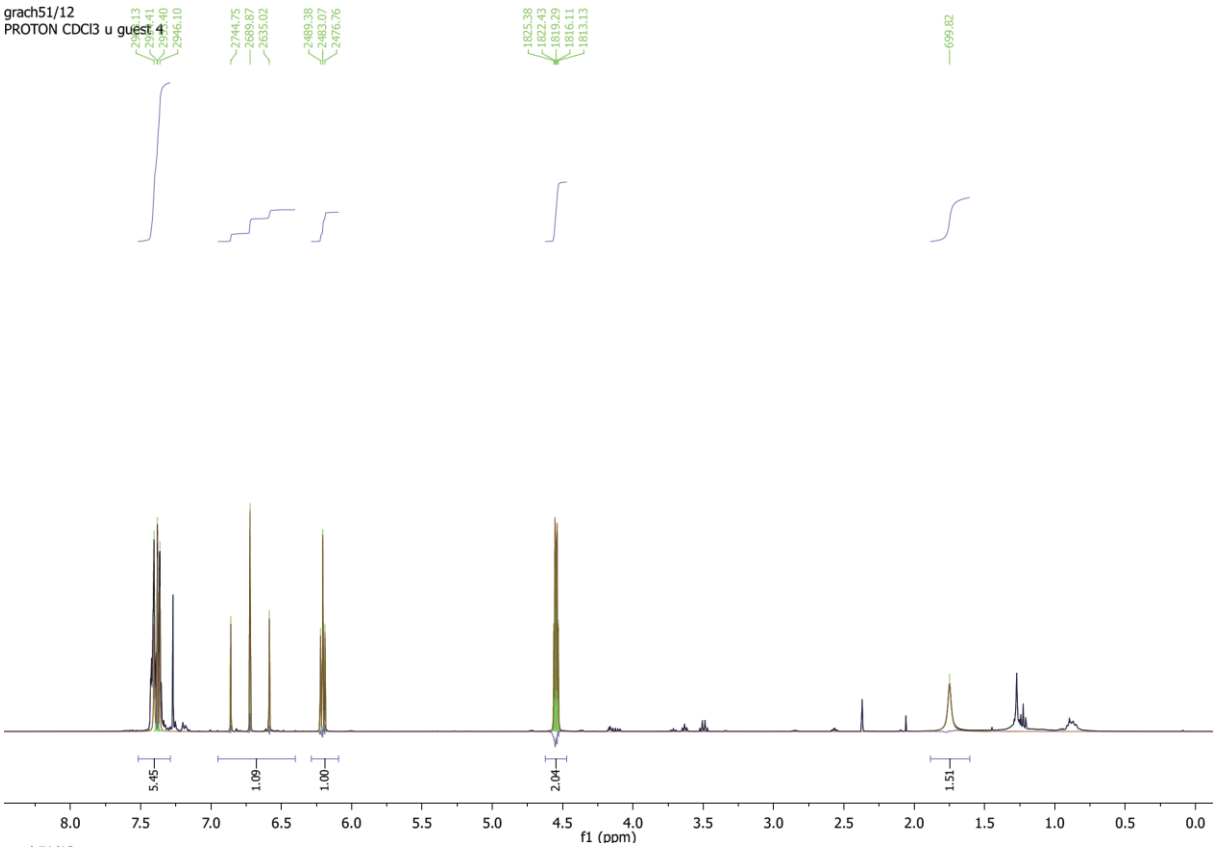

gra727p1/10
PROTON CDCl3 u guest 3
6.56
1.00
2.13
1.99
2.59
2.12
f1 (ppm)

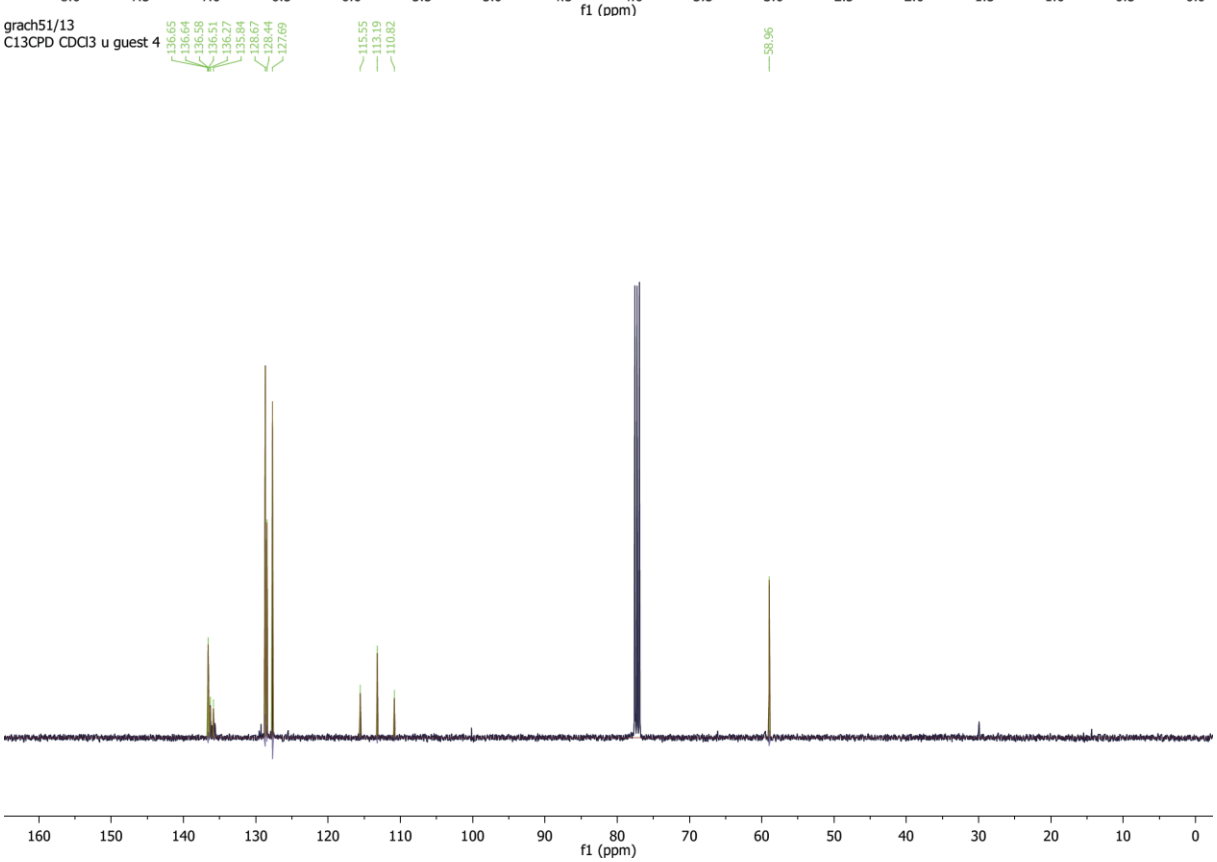

gra727p1/11
C13CPD CDCl3 u guest 3
81.35
79.73
58.75
36.63
36.61
34.69
f1 (ppm)

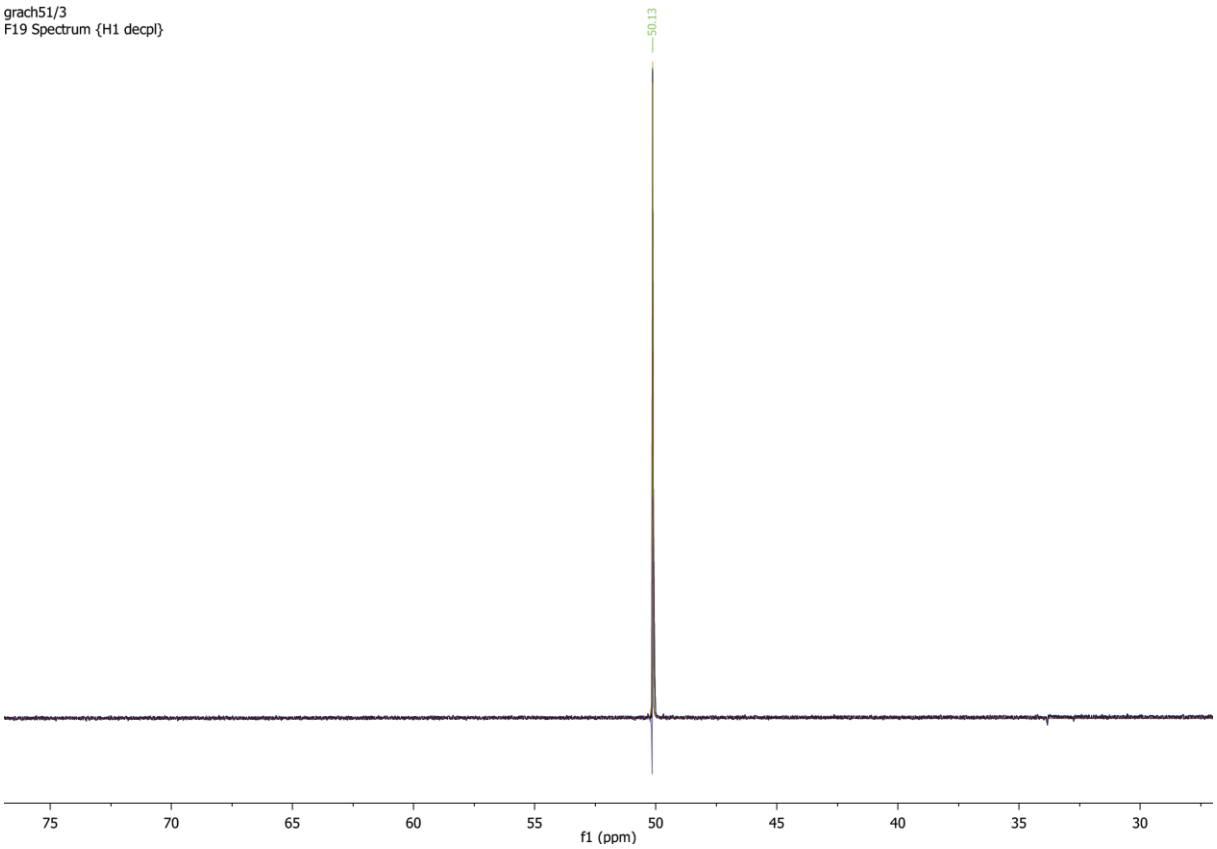


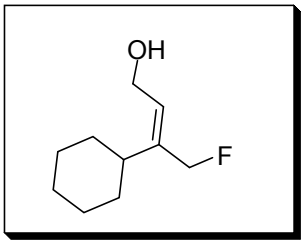


4f

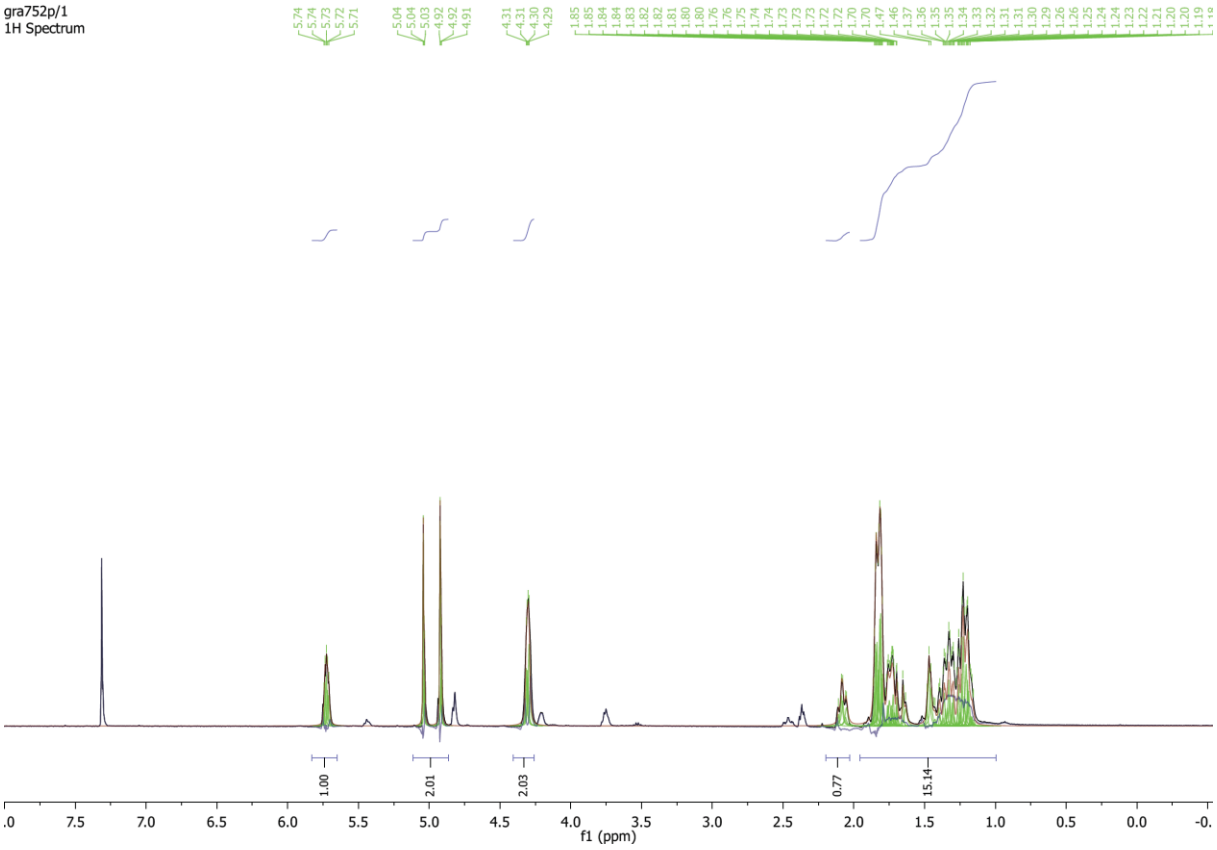

grach51/12
PROTON CDCl3 u guest 4
2946.10
2744.75
2689.87
2655.02
2489.38
2483.07
2476.76
1825.38
1822.43
1819.29
1816.11
1813.13
699.82
5.45
1.09
1.00
2.04
1.51
f1 (ppm)

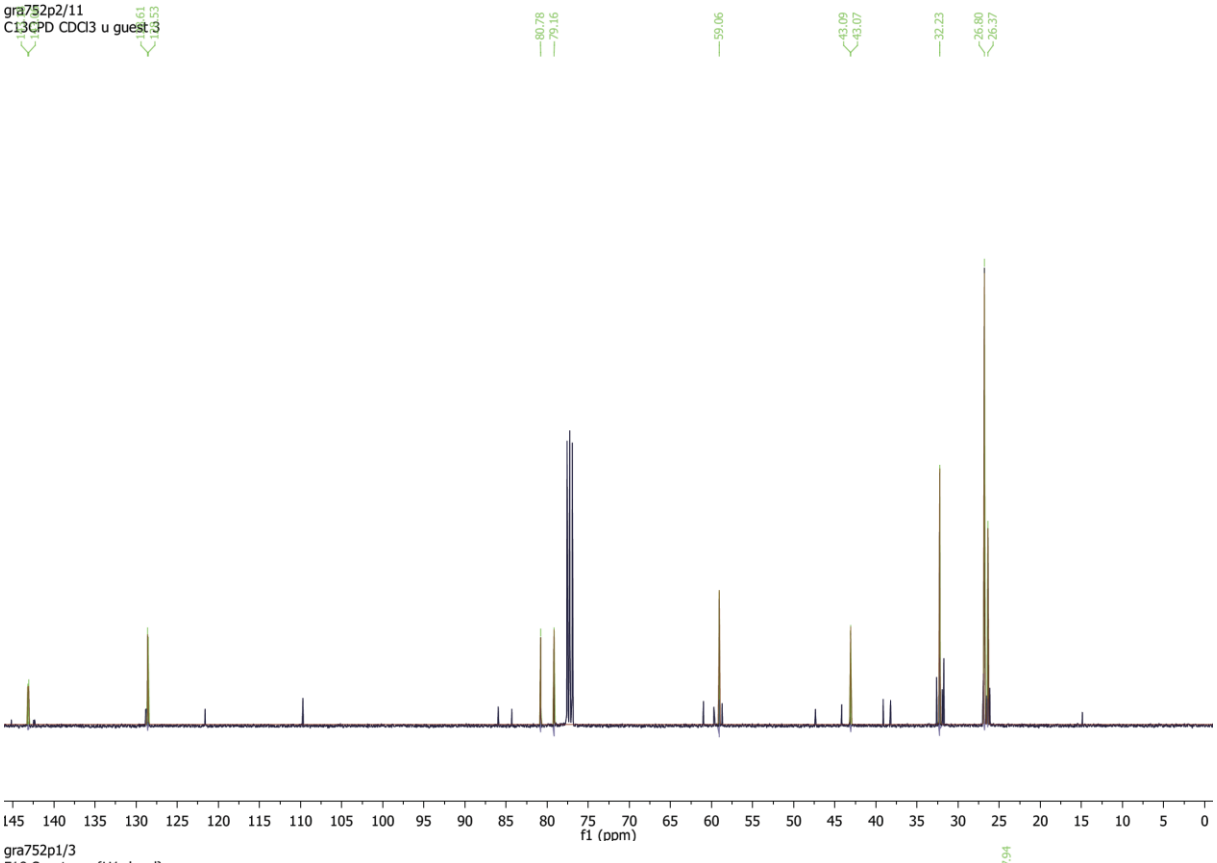

grach51/13
C13CPD CDCl3 u guest 4
136.65
136.64
136.58
136.51
136.27
135.84
128.67
128.44
127.69
115.55
113.19
110.82
58.96
f1 (ppm)

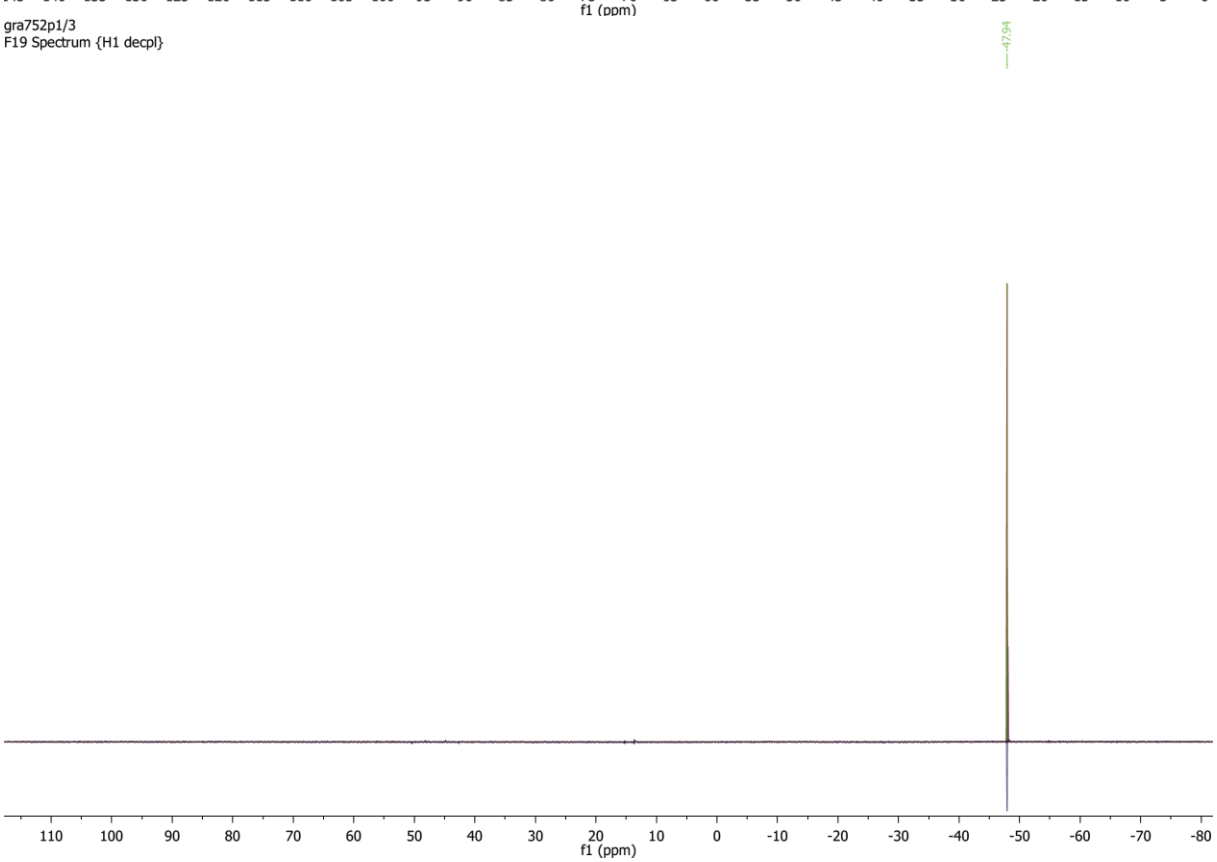


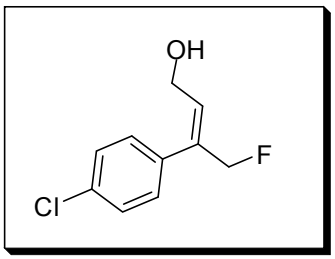


4c

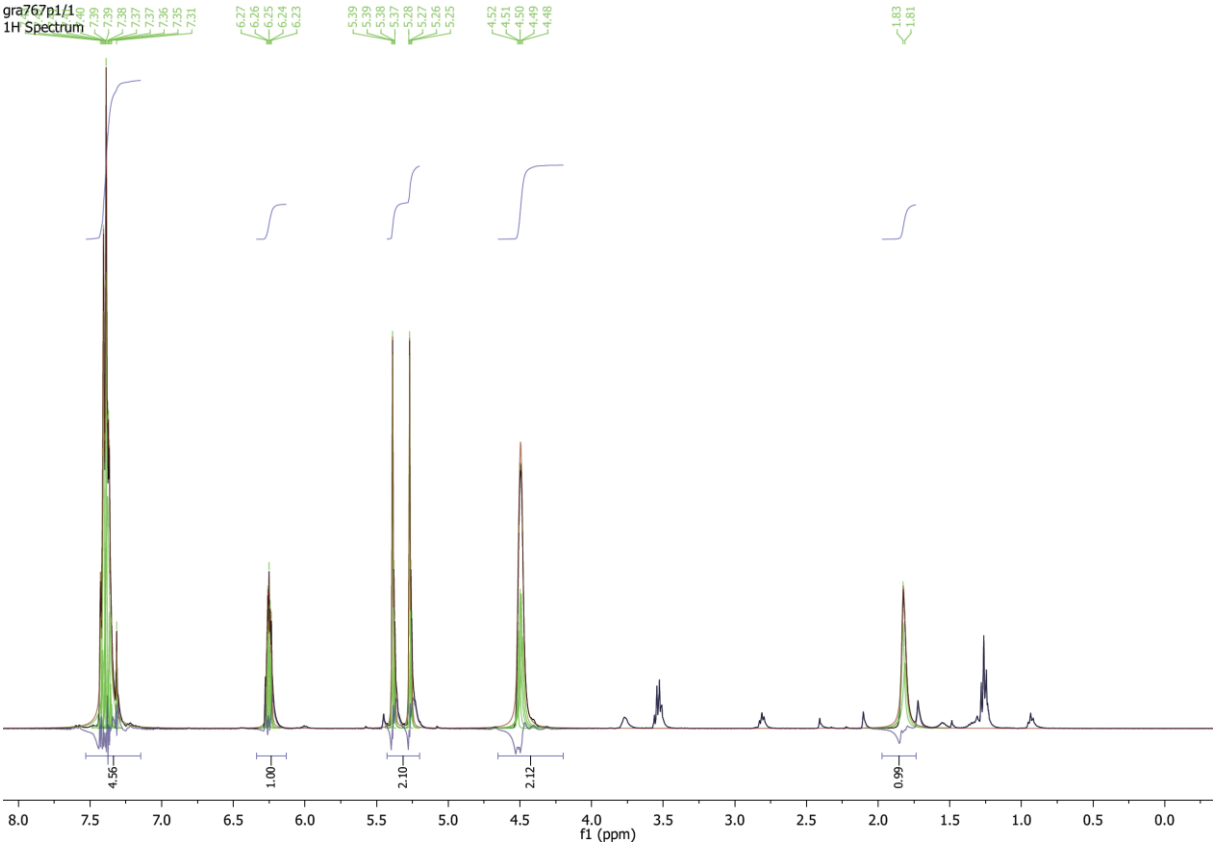


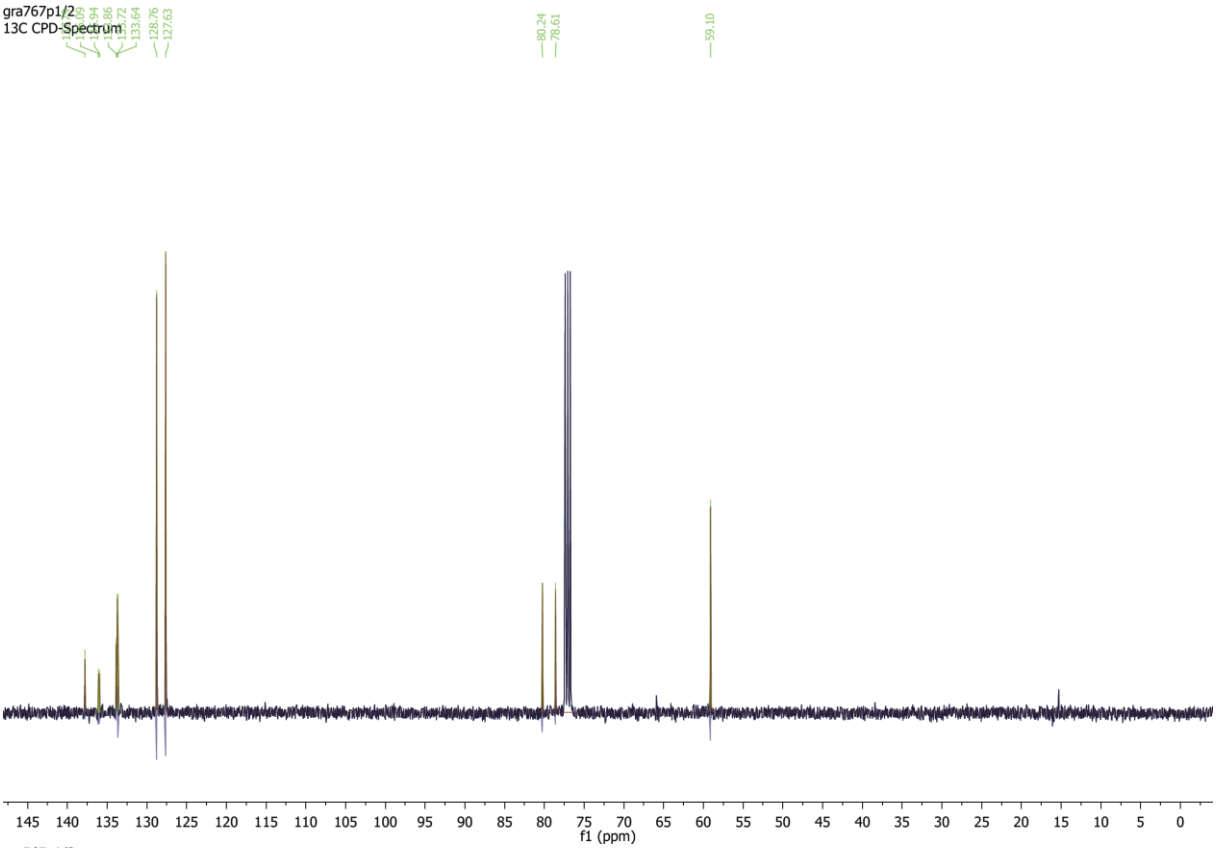

gra752p2/11
C13CPD CDCl3 u guest 3
80.78
79.16
59.06
43.09
43.07
32.23
26.80
26.37
f1 (ppm)

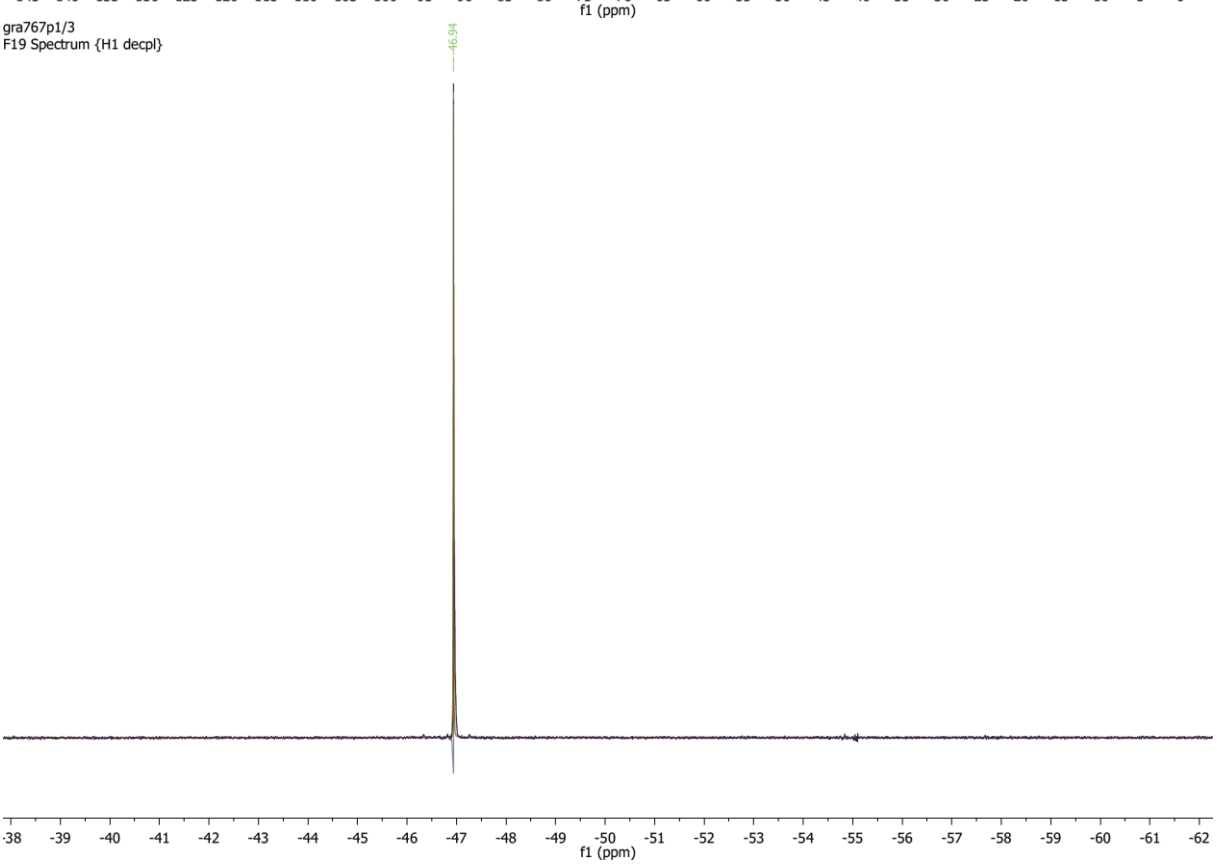

gra752p1/3
F19 Spectrum {H1 decpl}
-47.94
f1 (ppm)

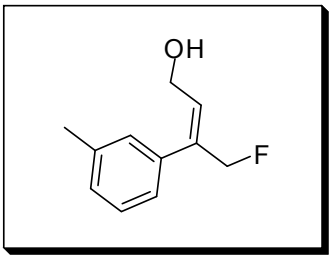


4d

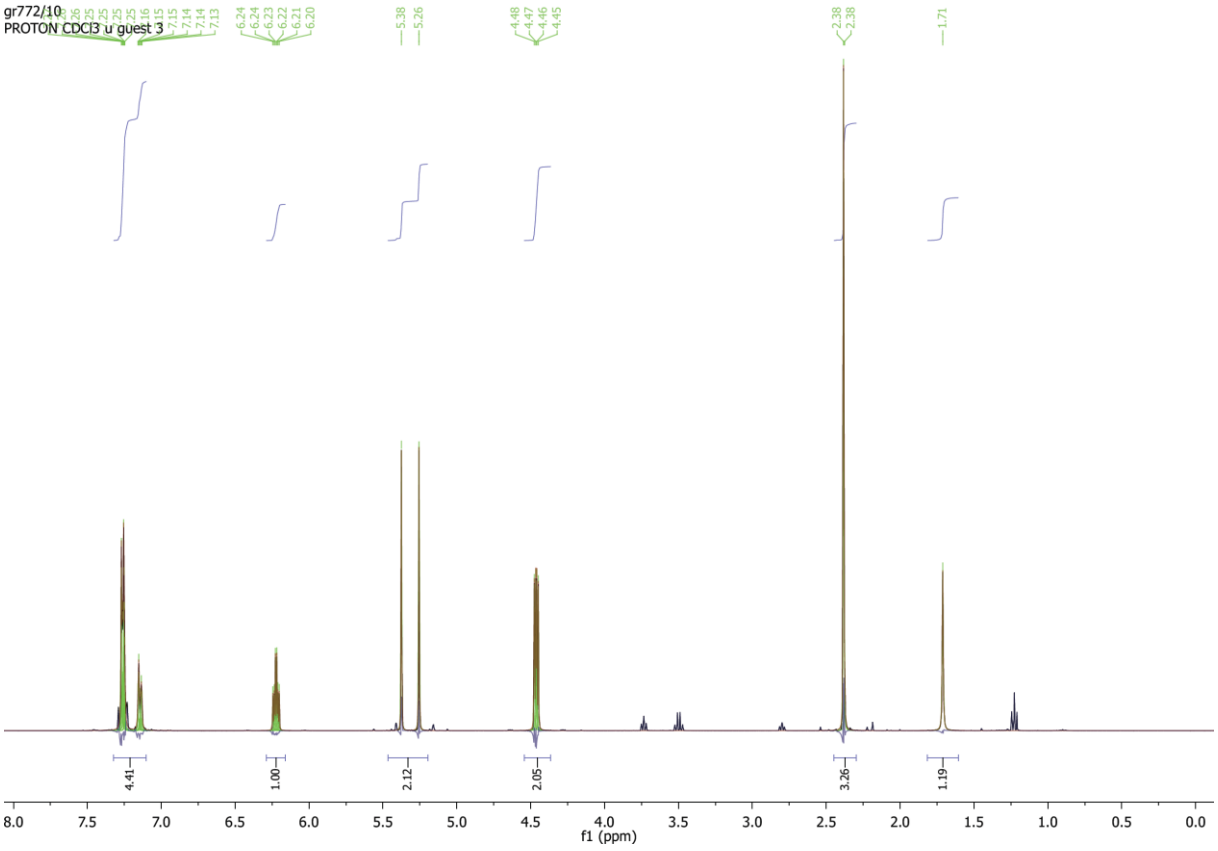


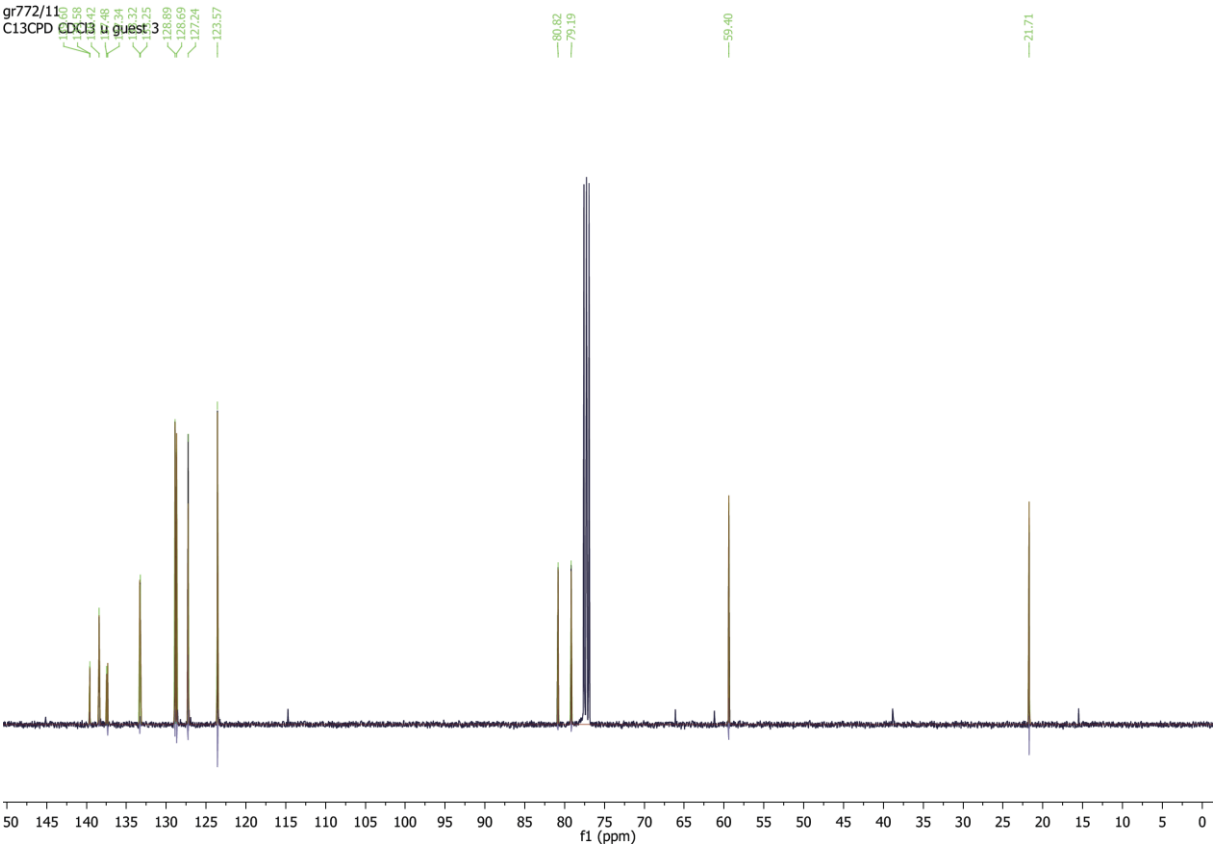

gra767p1/2
13C CPD-Spectrum
80.24
78.61
59.10
f1 (ppm)

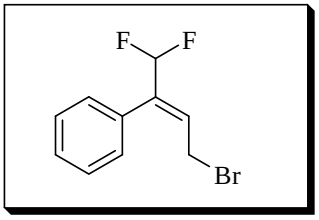

gra767p1/3
F19 Spectrum {H1 decpl}
-46.94
f1 (ppm)


OH
F

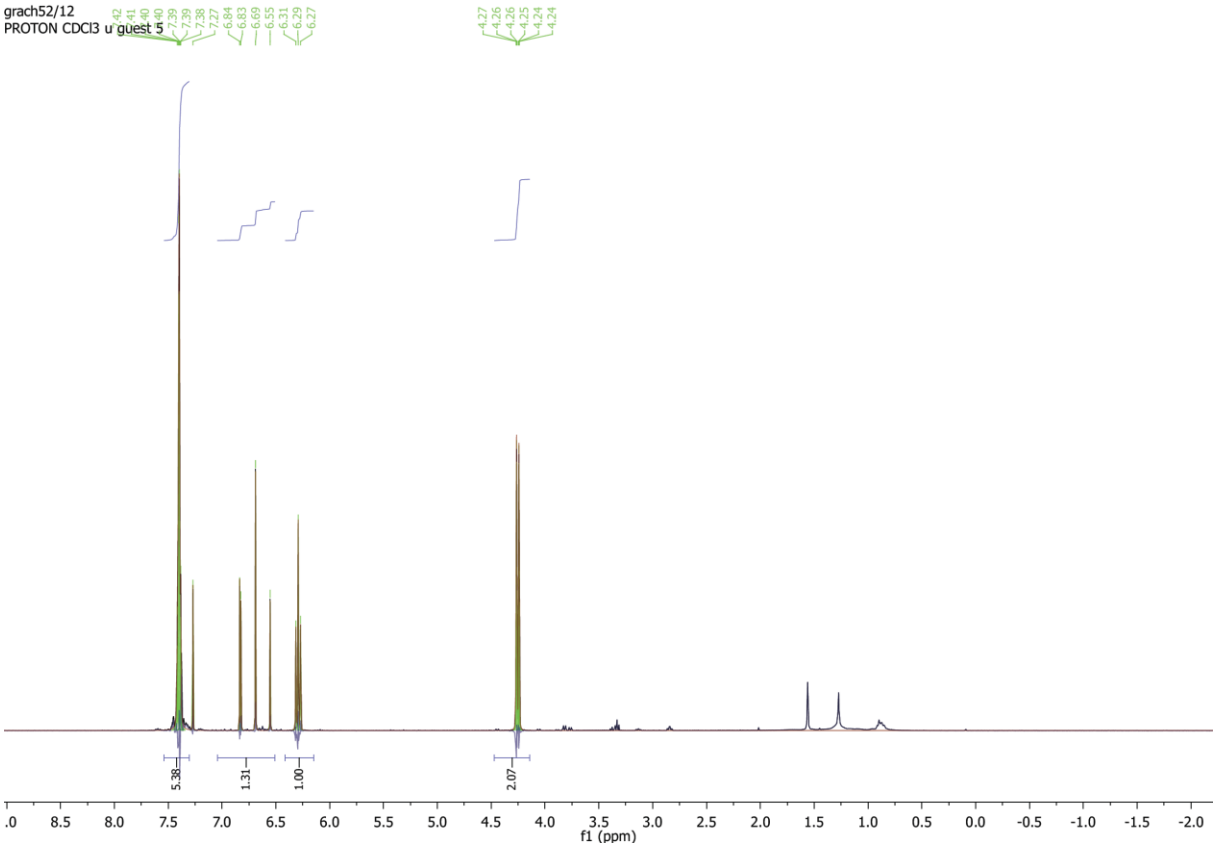
4e

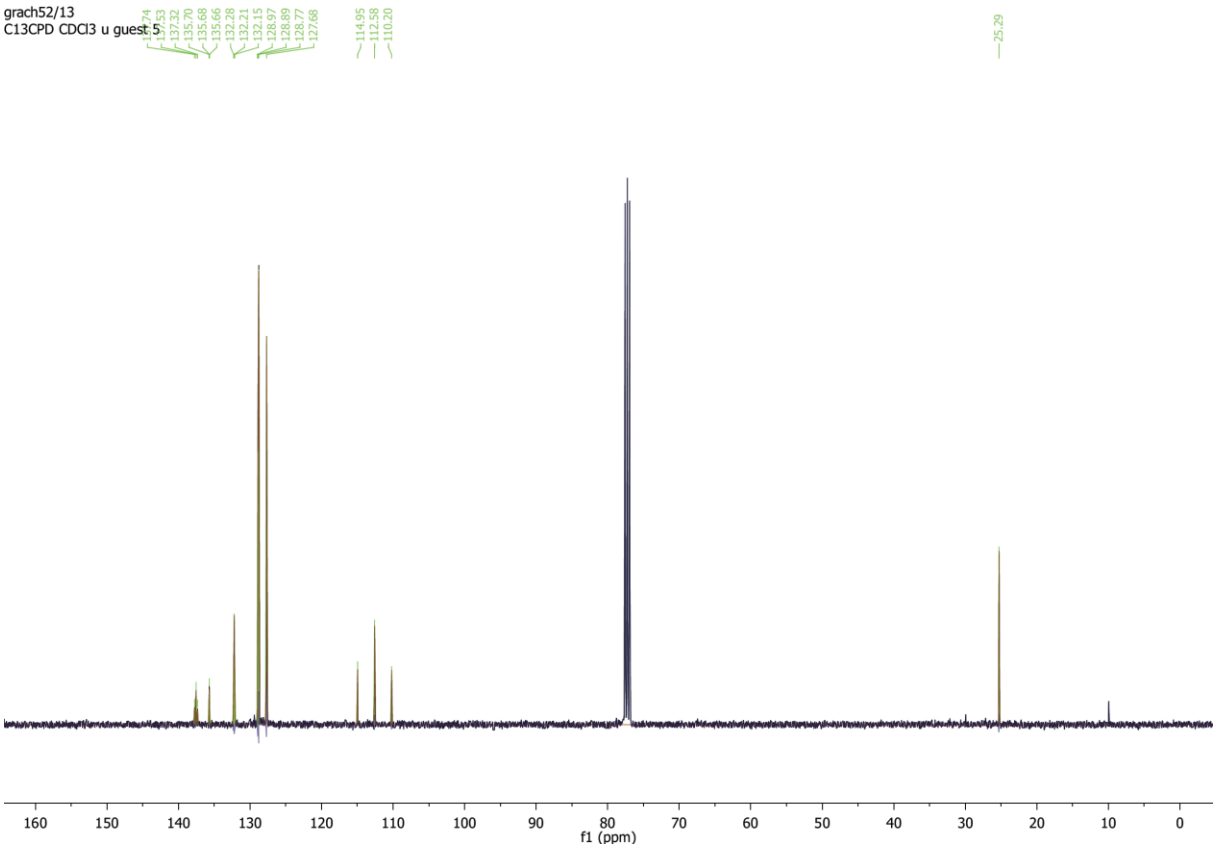

gr772/10
PROTON CDCl3 u guest 3
6.24
6.24
6.23
6.22
6.21
6.20
5.38
5.26
4.48
4.47
4.46
4.45
2.38
2.38
1.71
4.41
1.00
2.12
2.05
3.26
1.19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

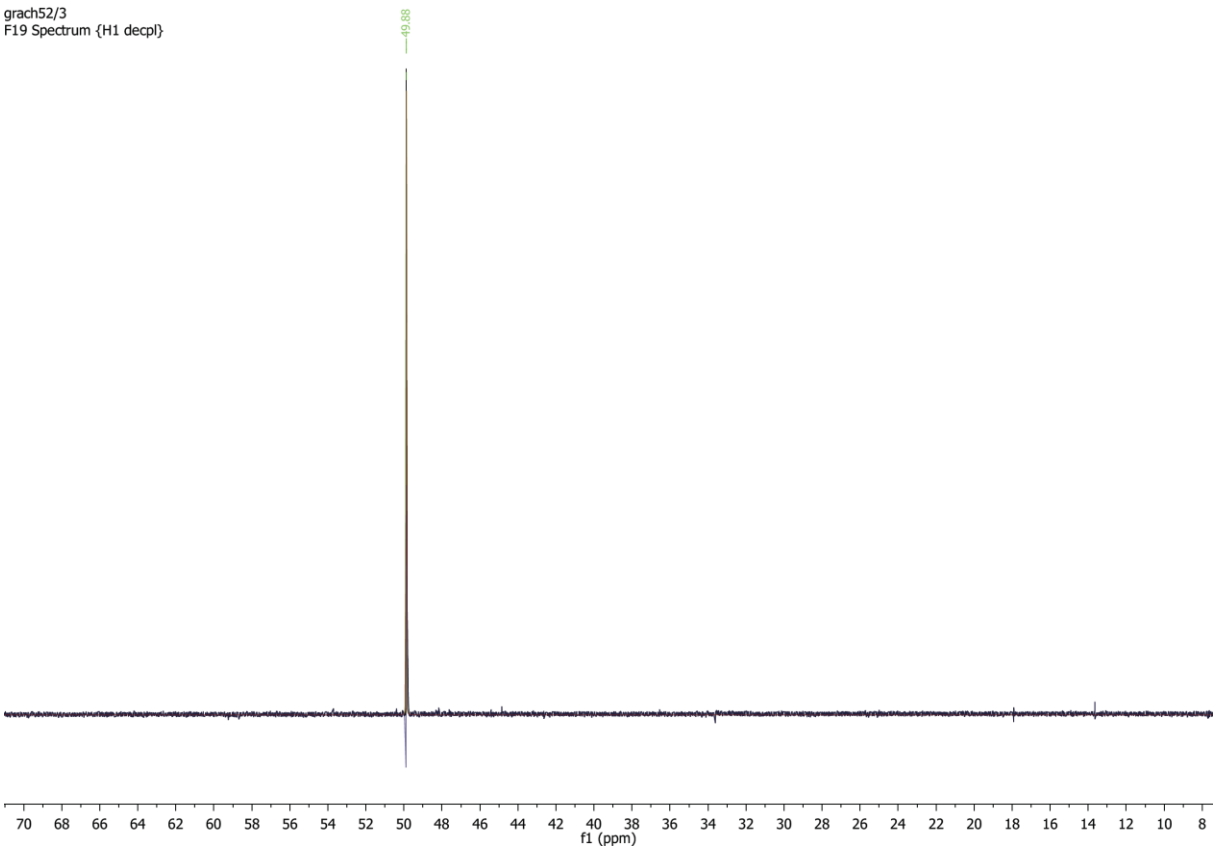

gr772/11
C13CPD CDCl3 u guest 3
128.89
128.69
127.24
123.57
80.82
79.19
59.40
21.71
f1 (ppm)

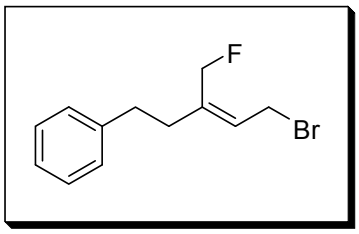


5g

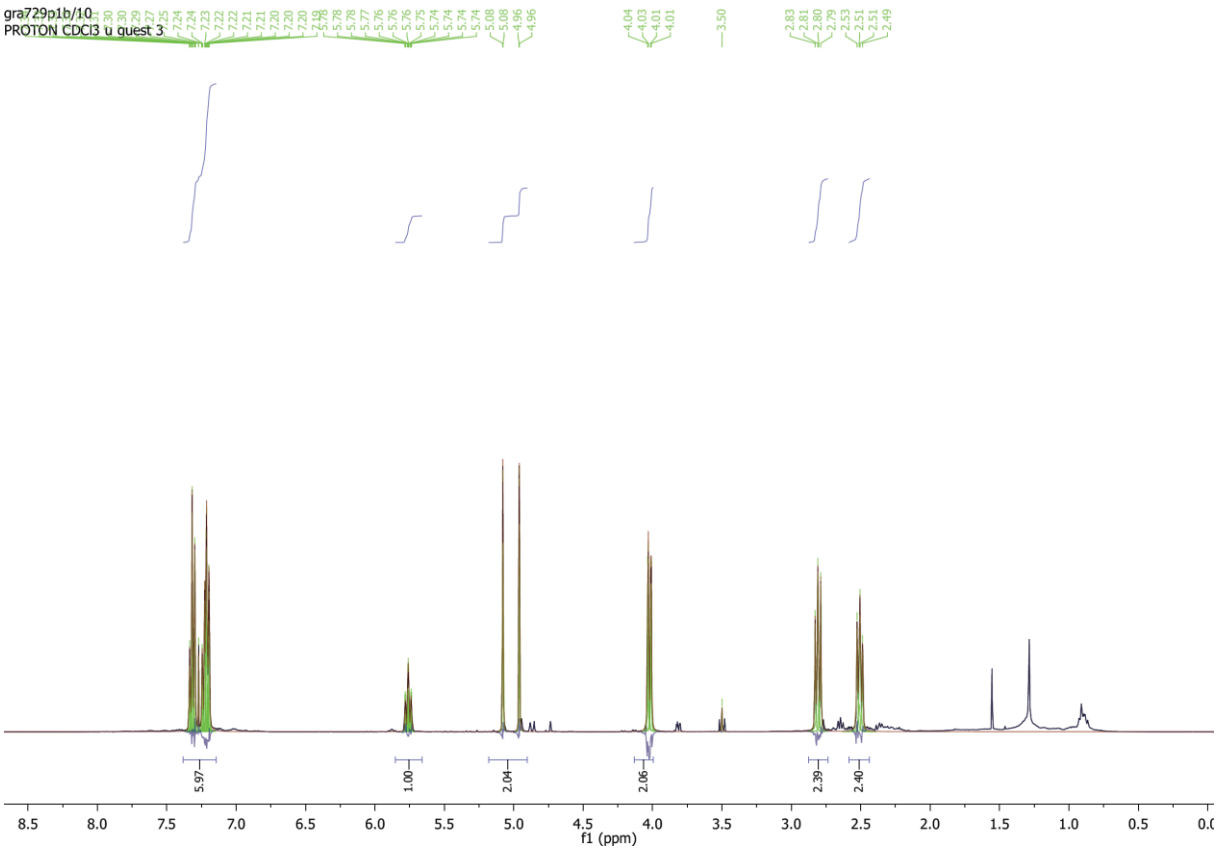


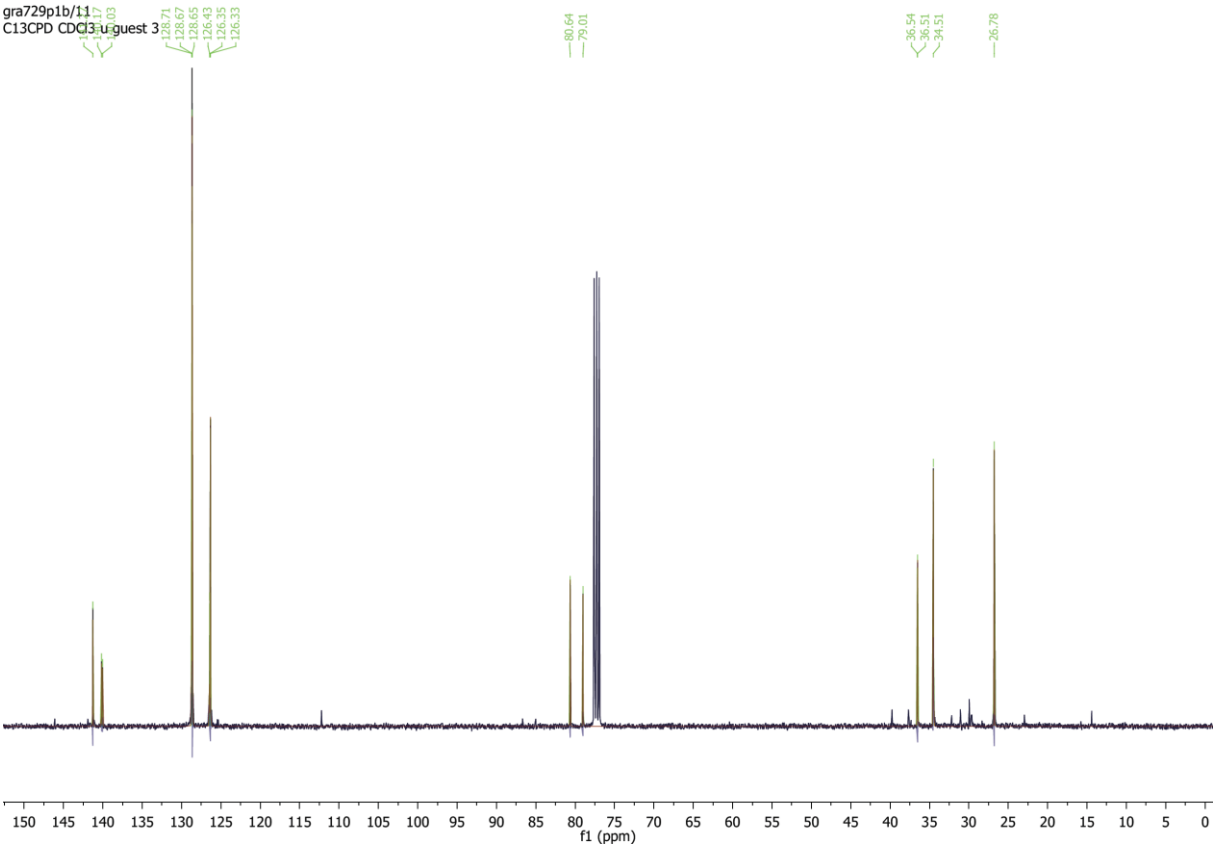

grach52/13
C13CPD CDCl3 u guest 5
137.74
137.53
137.32
135.70
135.68
135.66
132.28
132.21
132.15
128.97
128.89
128.77
127.68
114.95
112.58
110.20
25.29
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

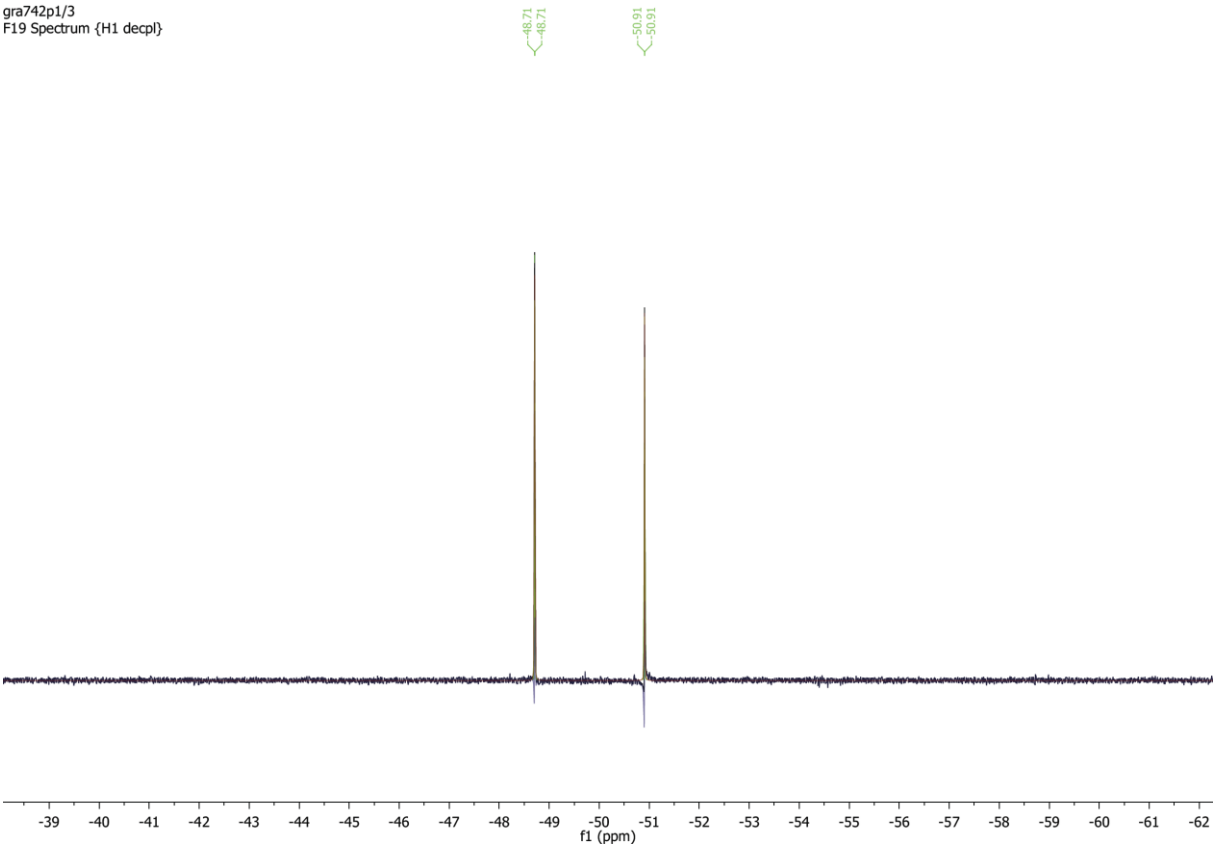

grach52/3
F19 Spectrum {H1 decpl}
-49.88
70
68
66
64
62
60
58
56
54
52
50
48
46
44
42
40
38
36
34
32
30
28
26
24
22
20
18
16
14
12
10
8
f1 (ppm)

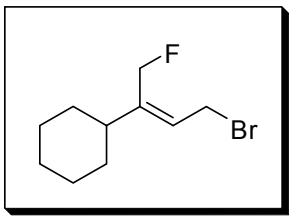

F
Br

5a

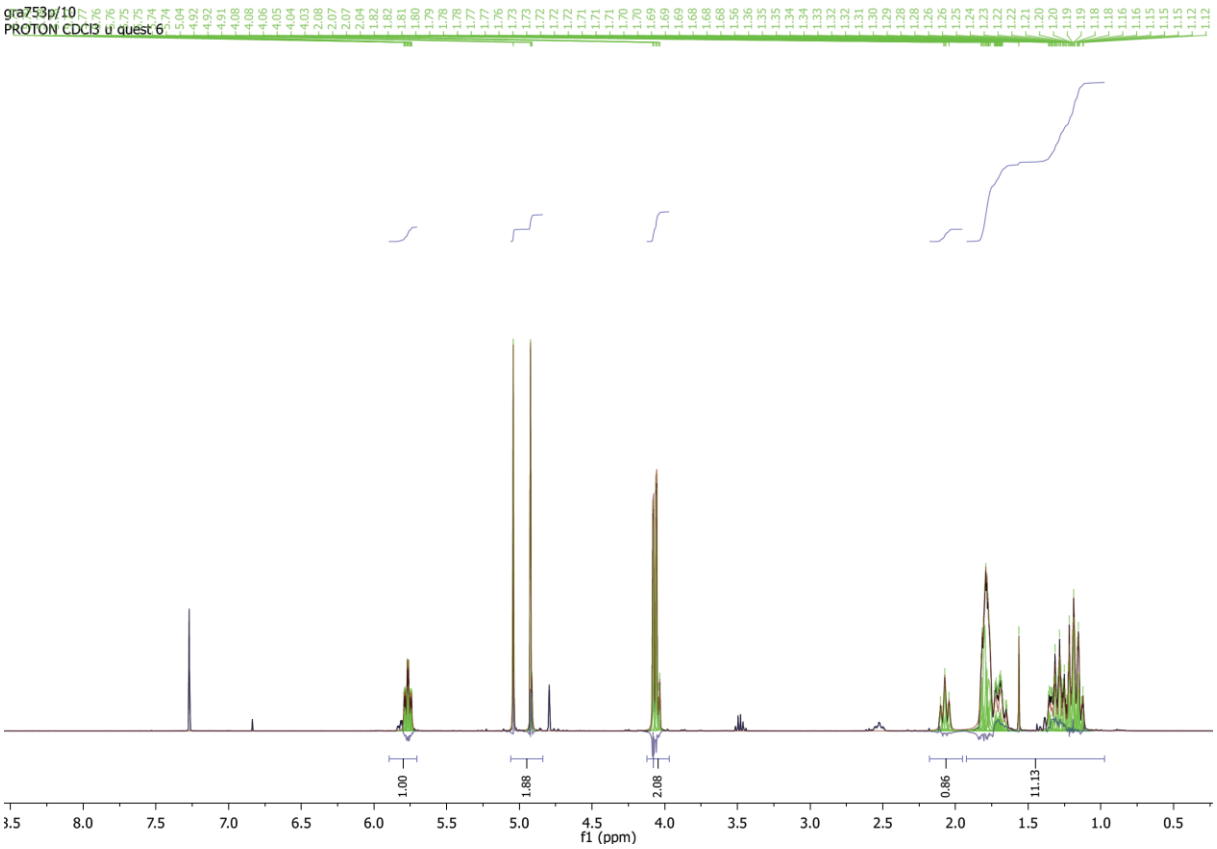

gra729p1b/10
PROTON CDCl3 u quest 3
5.97
1.00
2.04
2.06
2.39
2.40
f1 (ppm)

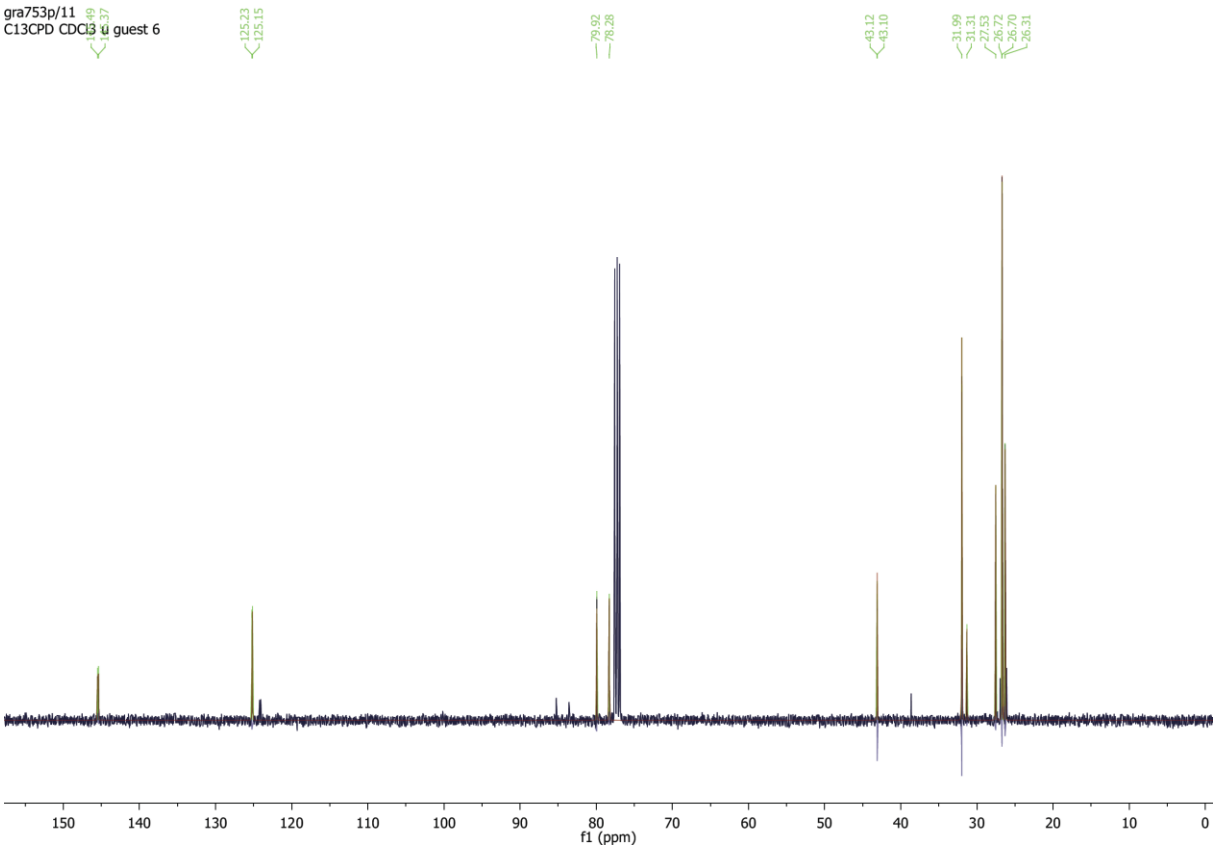

gra729p1b/11
C13CPD CDCl3 u guest 3
140.17
139.03
128.71
128.67
128.65
126.43
126.35
126.33
80.64
79.01
36.54
36.51
34.51
26.78
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

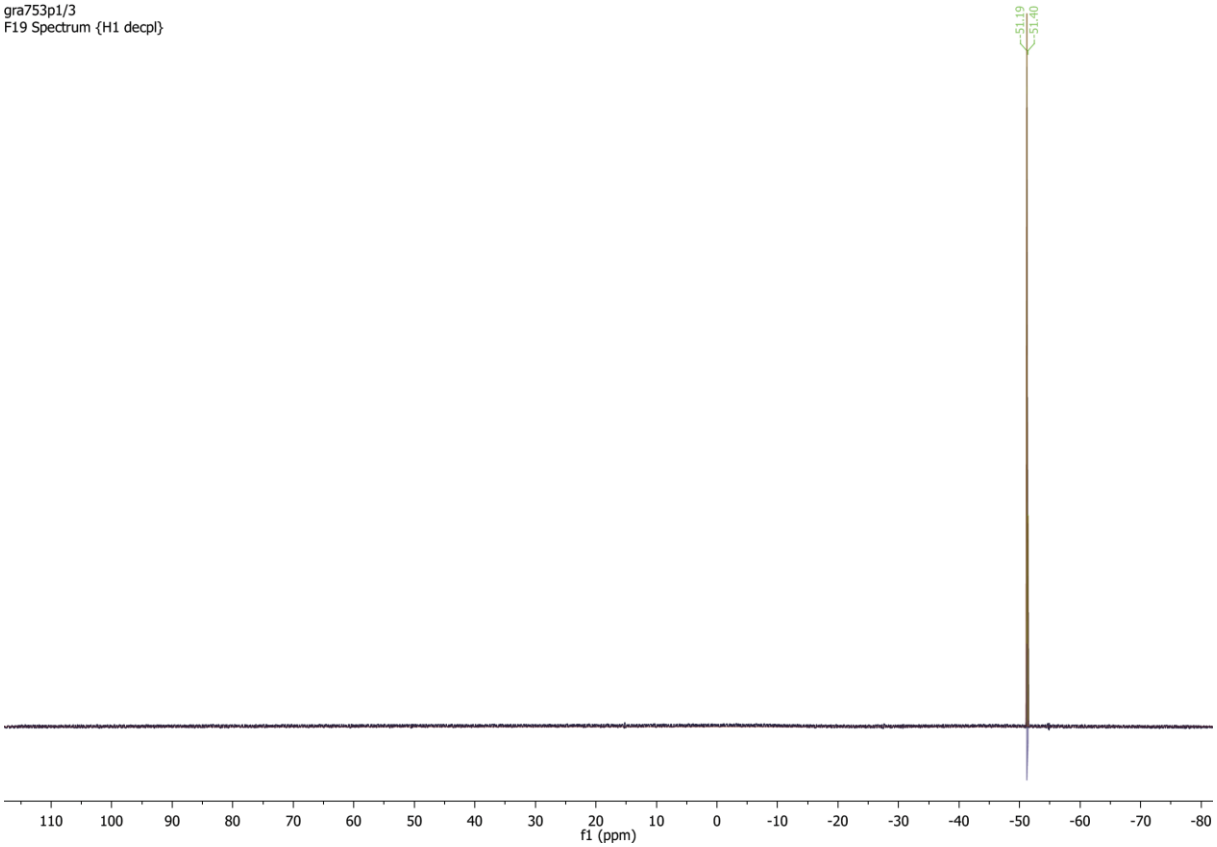

gra742p1/3
F19 Spectrum {H1 decpl}
48.71
48.71
50.91
50.91
-39
-40
-41
-42
-43
-44
-45
-46
-47
-48
-49
-50
-51
-52
-53
-54
-55
-56
-57
-58
-59
-60
-61
-62
f1 (ppm)

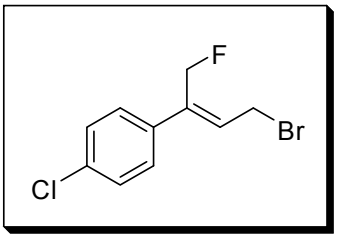

F
Br

5d

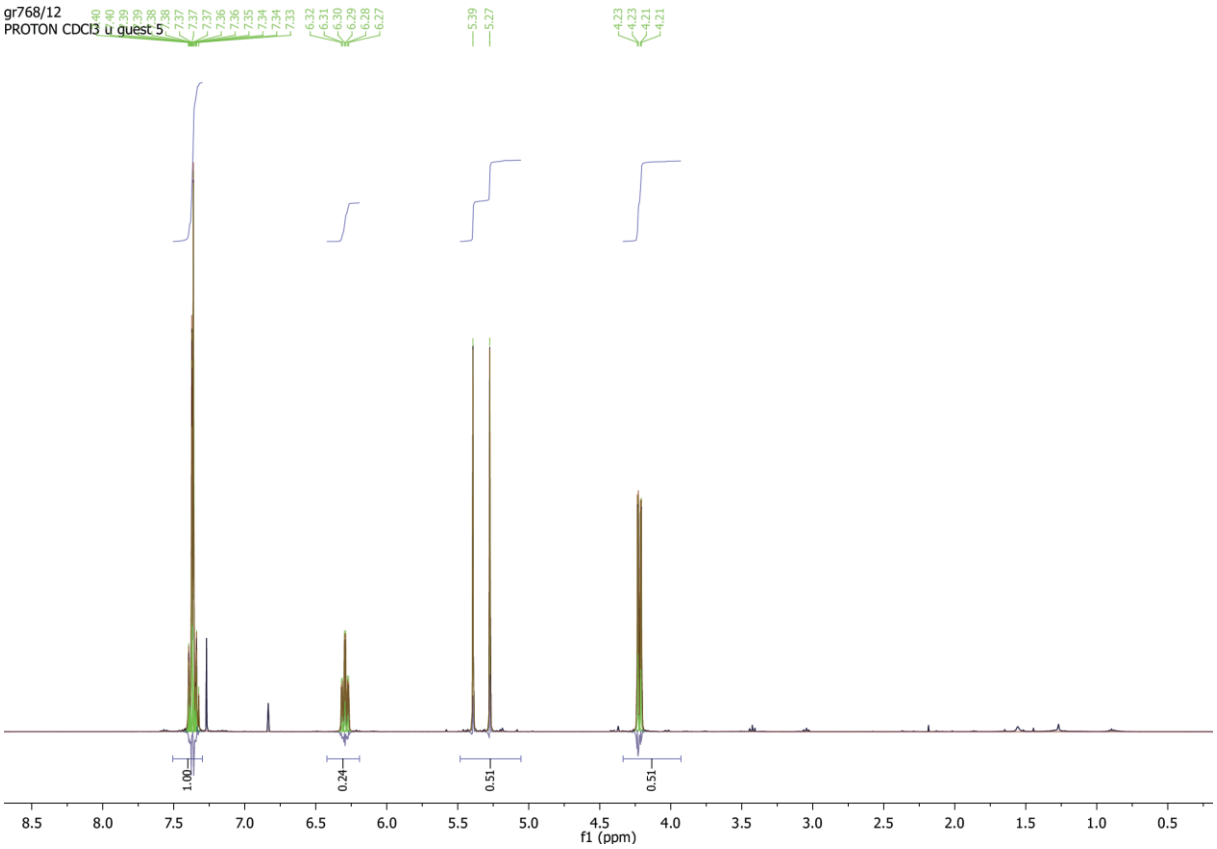

gra753p/10
PROTON CDCl3 u guest 6
1.00
1.88
2.08
0.86
11.13
f1 (ppm)

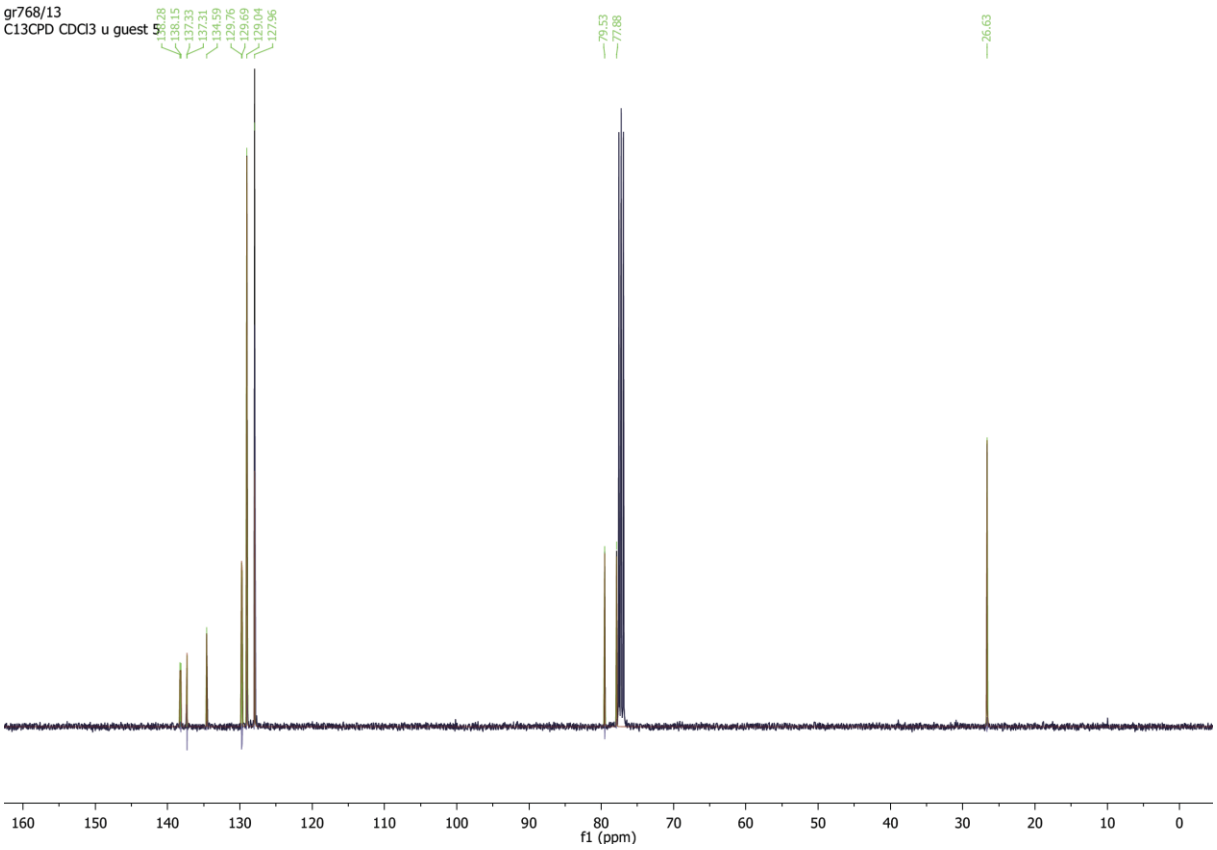

gra753p/11
C13CPD CDCl3 u guest 6
125.23
125.15
79.92
78.28
43.12
43.10
31.99
31.31
27.53
26.72
26.70
26.31
f1 (ppm)

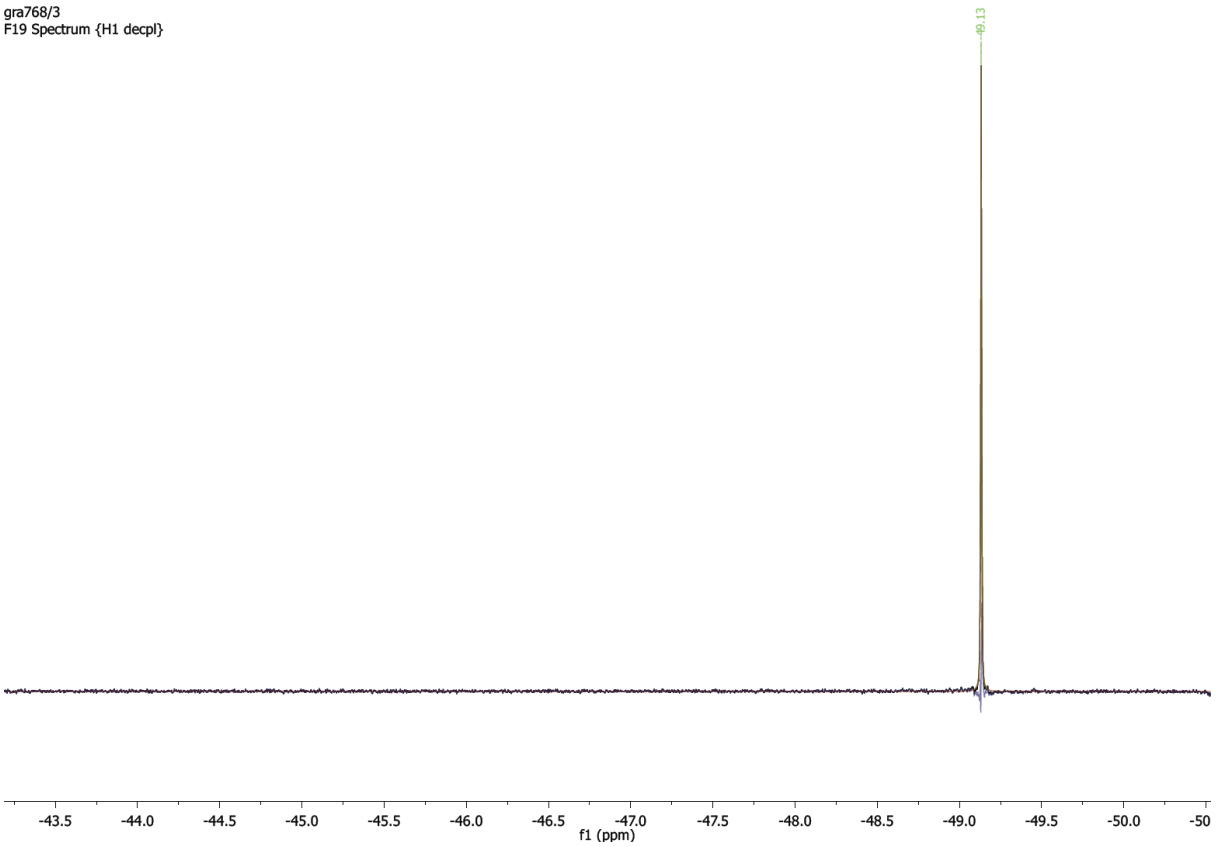

gra753p1/3
F19 Spectrum {H1 decpl}
-51.19
-51.40
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

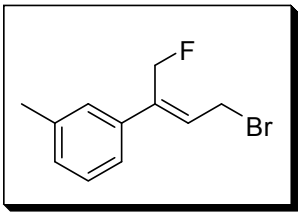

F
Br
Cl

5e

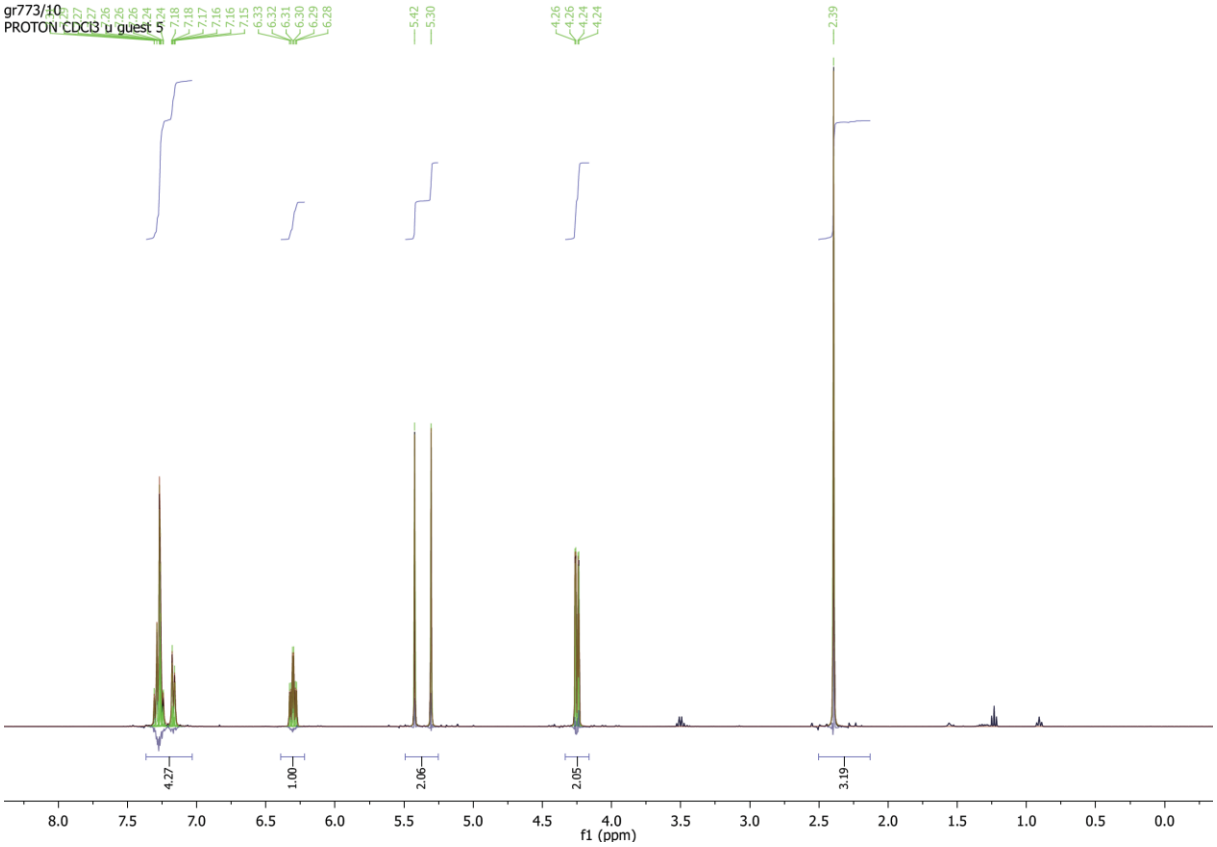

gr768/12
PROTON CDCl3 u guest 5
7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.33
6.32
6.31
6.30
6.29
6.28
6.27
5.39
5.27
4.23
4.23
4.21
4.21
1.00
0.24
0.51
0.51
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

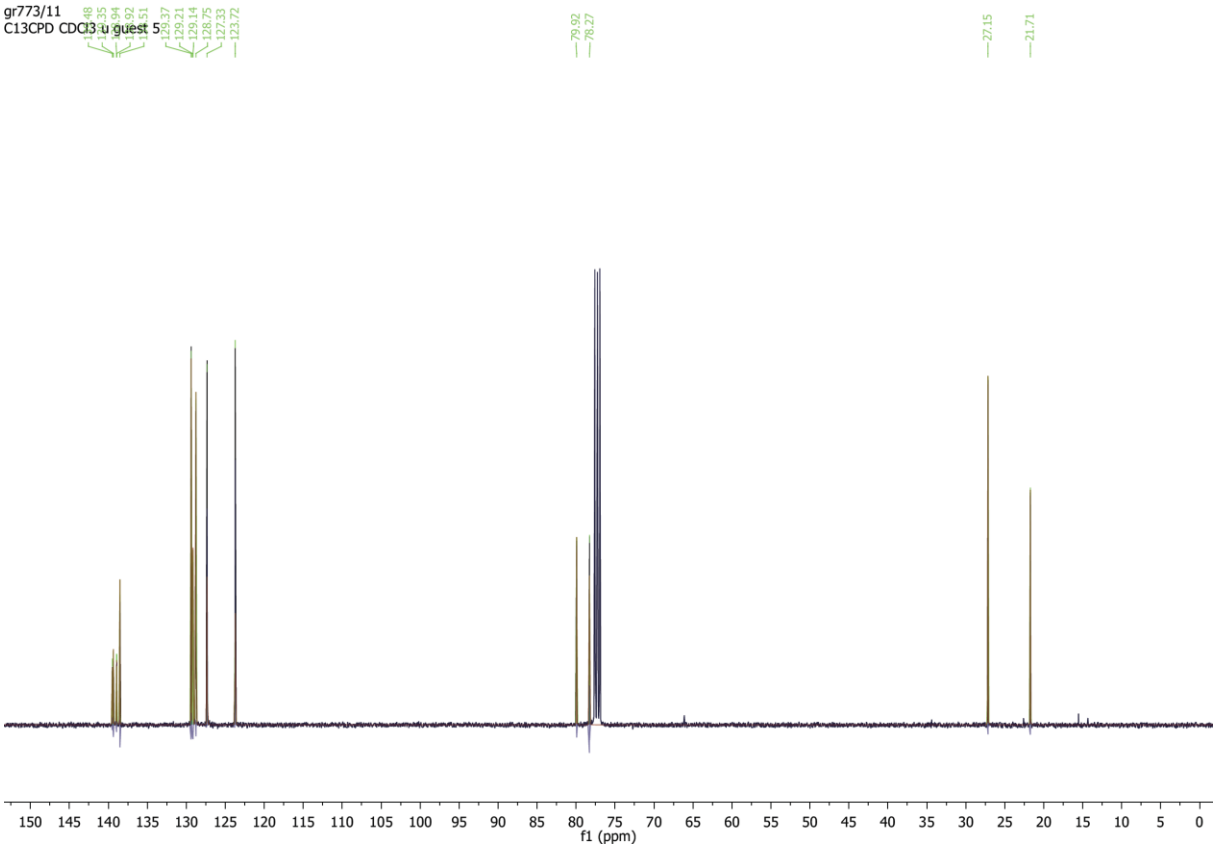

gr768/13
C13CPD CDCl3 u guest 5
138.28
138.15
137.33
137.31
134.59
129.76
129.69
129.04
127.96
79.53
77.88
26.63
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

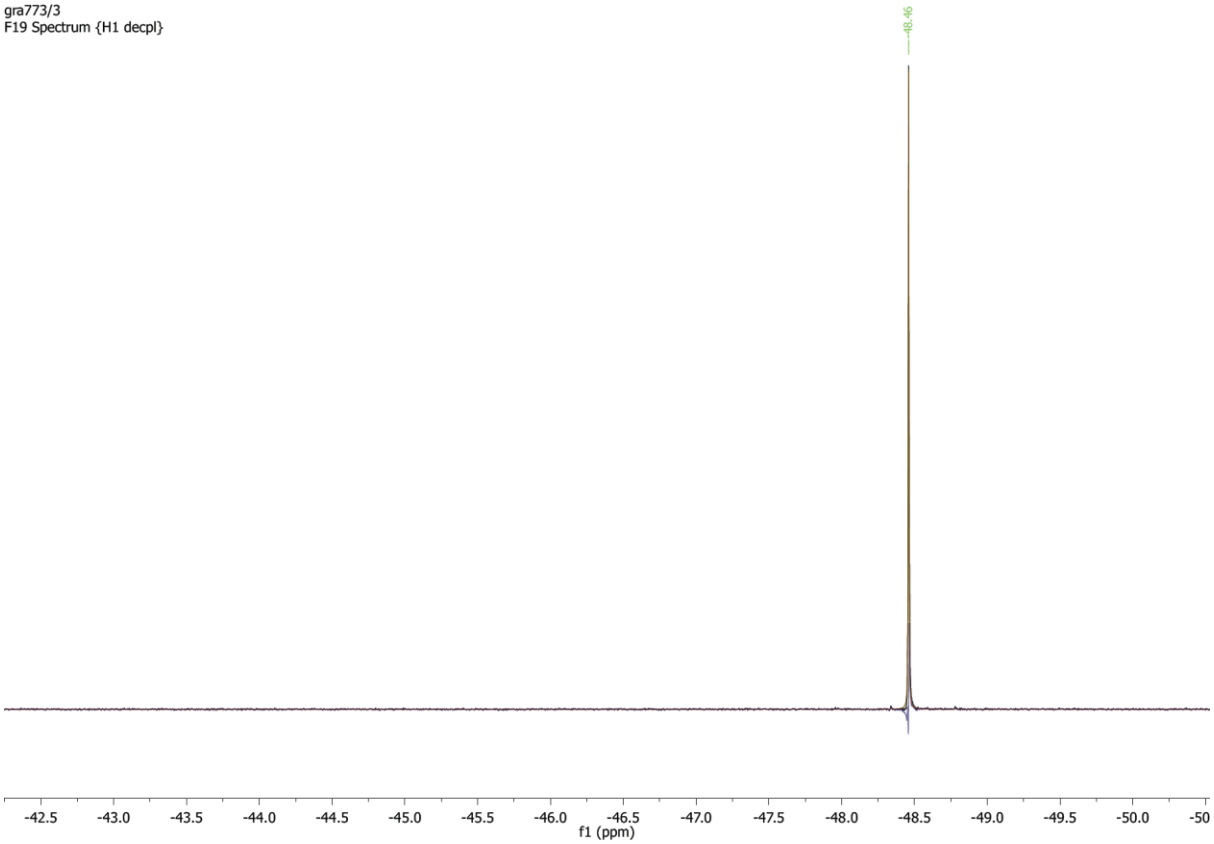

gra768/3
F19 Spectrum {H1 decpl}
-49.13
-43.5
-44.0
-44.5
-45.0
-45.5
-46.0
-46.5
-47.0
-47.5
-48.0
-48.5
-49.0
-49.5
-50.0
-50
f1 (ppm)

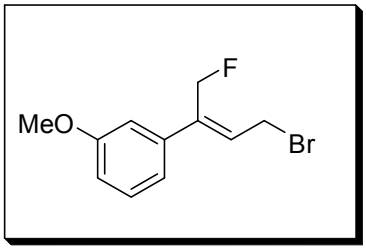

F
Br

5f

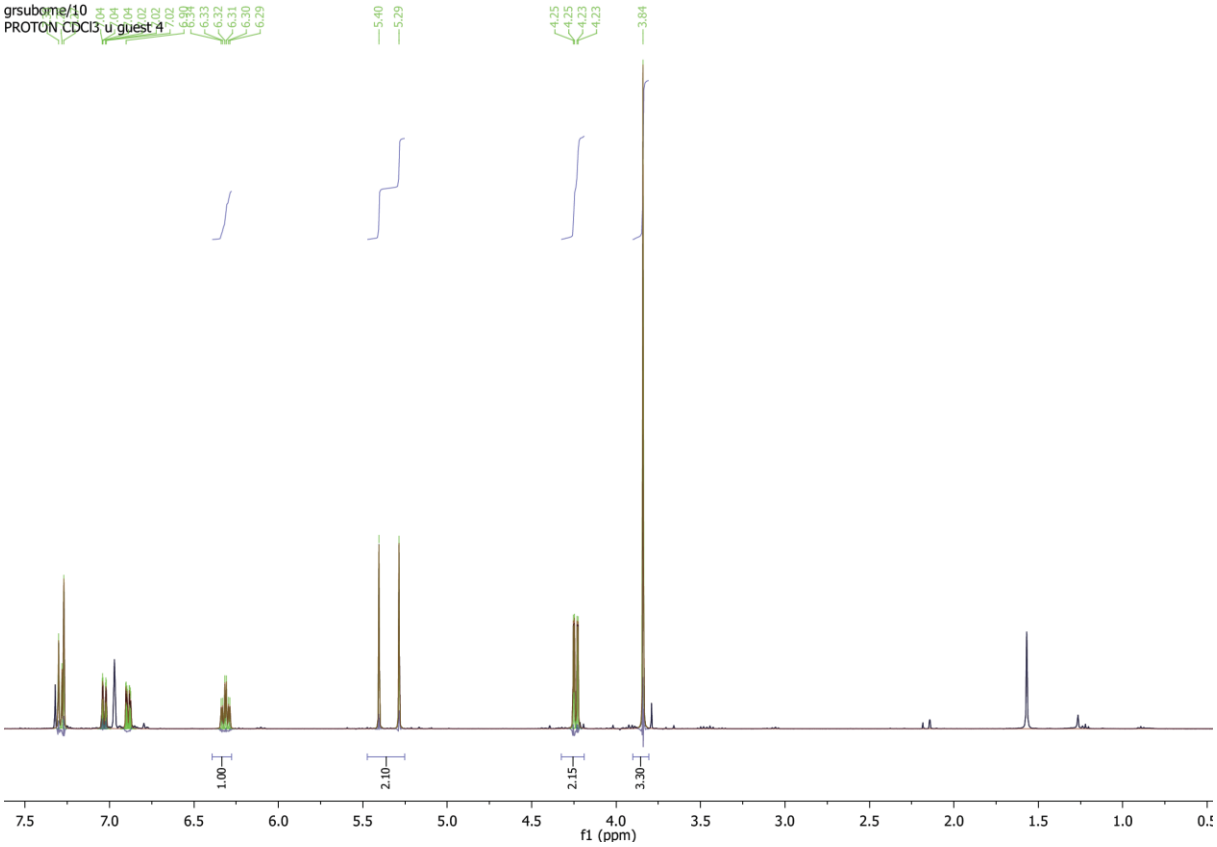

gr773/10
PROTON CDCl3 u guest 5
5.42
5.30
4.26
4.25
4.24
4.24
2.39
4.27
1.00
2.06
2.05
3.19
f1 (ppm)

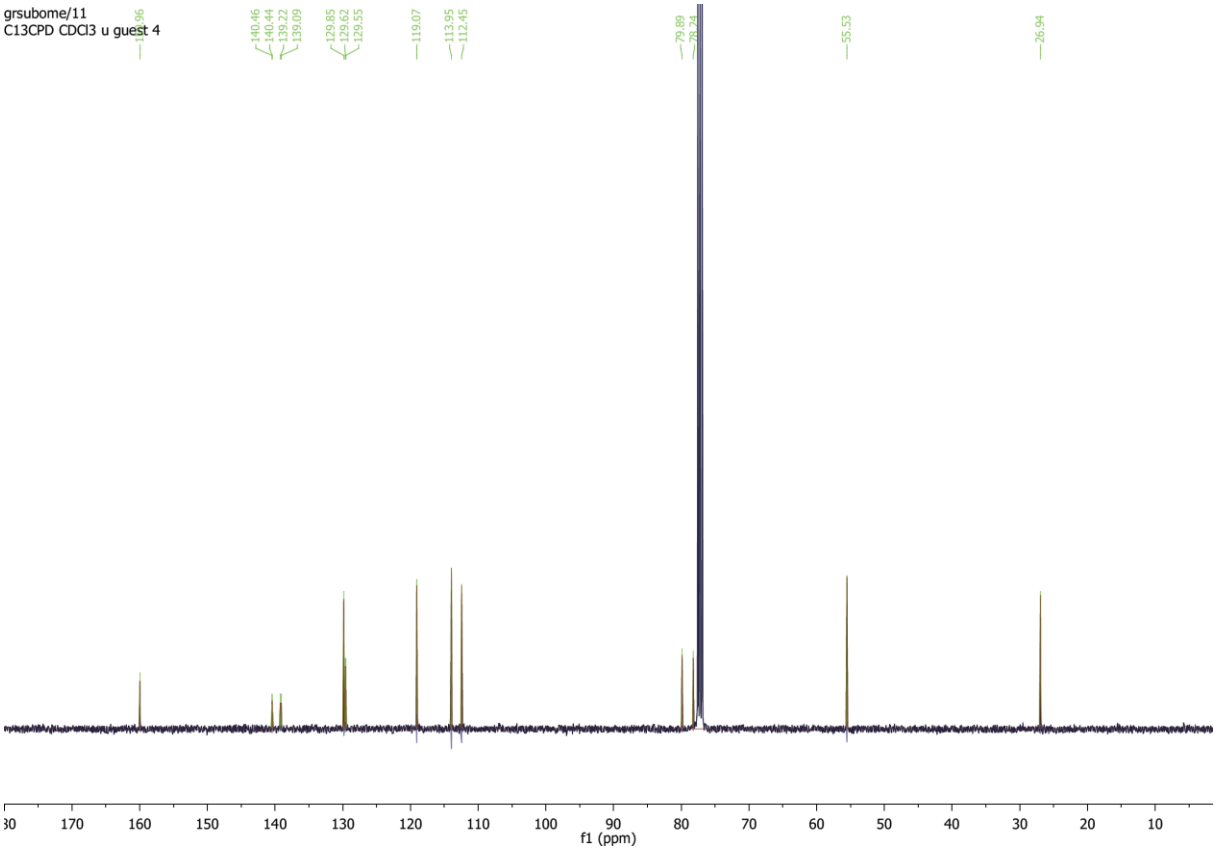

gr773/11
C13CPD CDCl3 u guest 5
79.92
78.27
27.15
21.71
f1 (ppm)

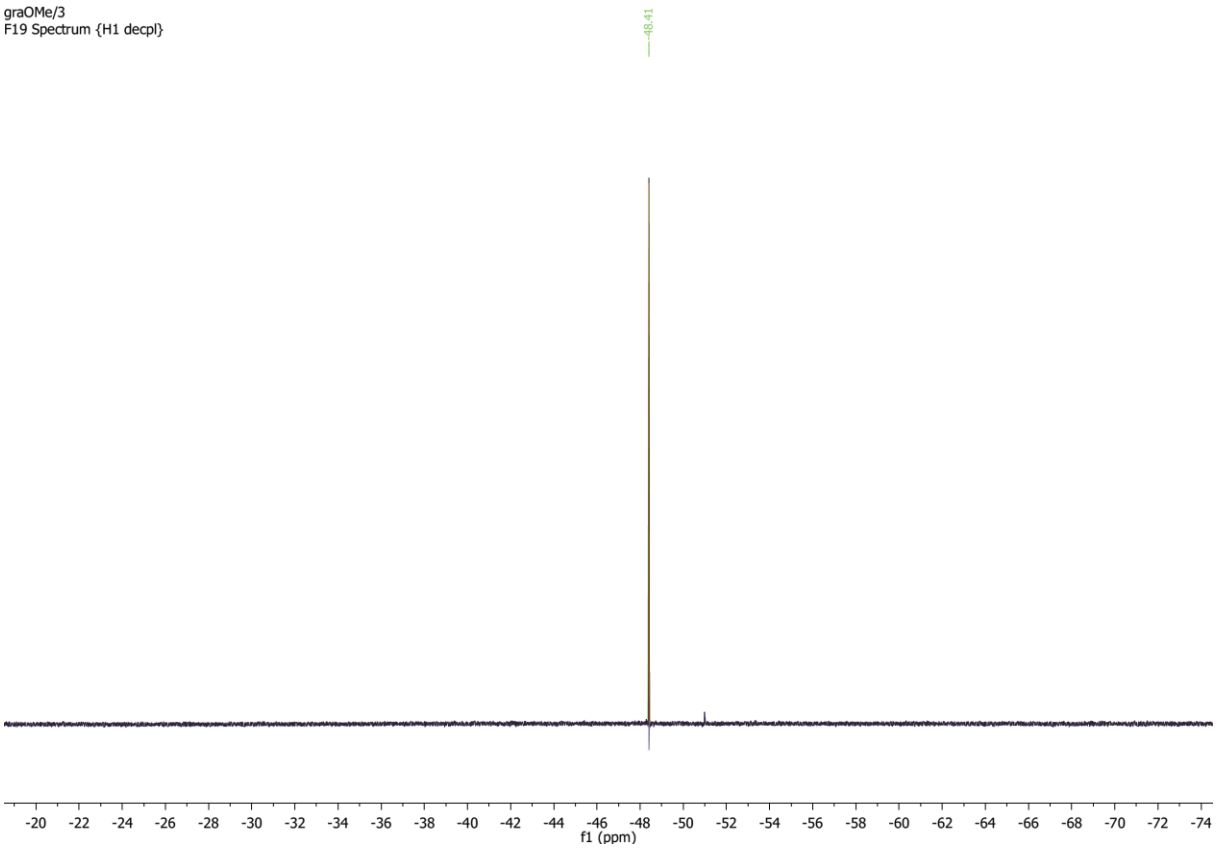

gra773/3
F19 Spectrum {H1 decpl}
-48.46
-42.5
-43.0
-43.5
-44.0
-44.5
-45.0
-45.5
-46.0
-46.5
-47.0
-47.5
-48.0
-48.5
-49.0
-49.5
-50.0
-50
f1 (ppm)

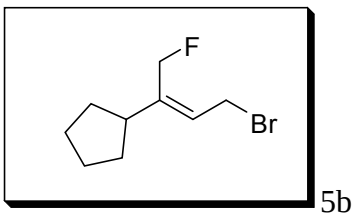

F
MeO
Br

5c

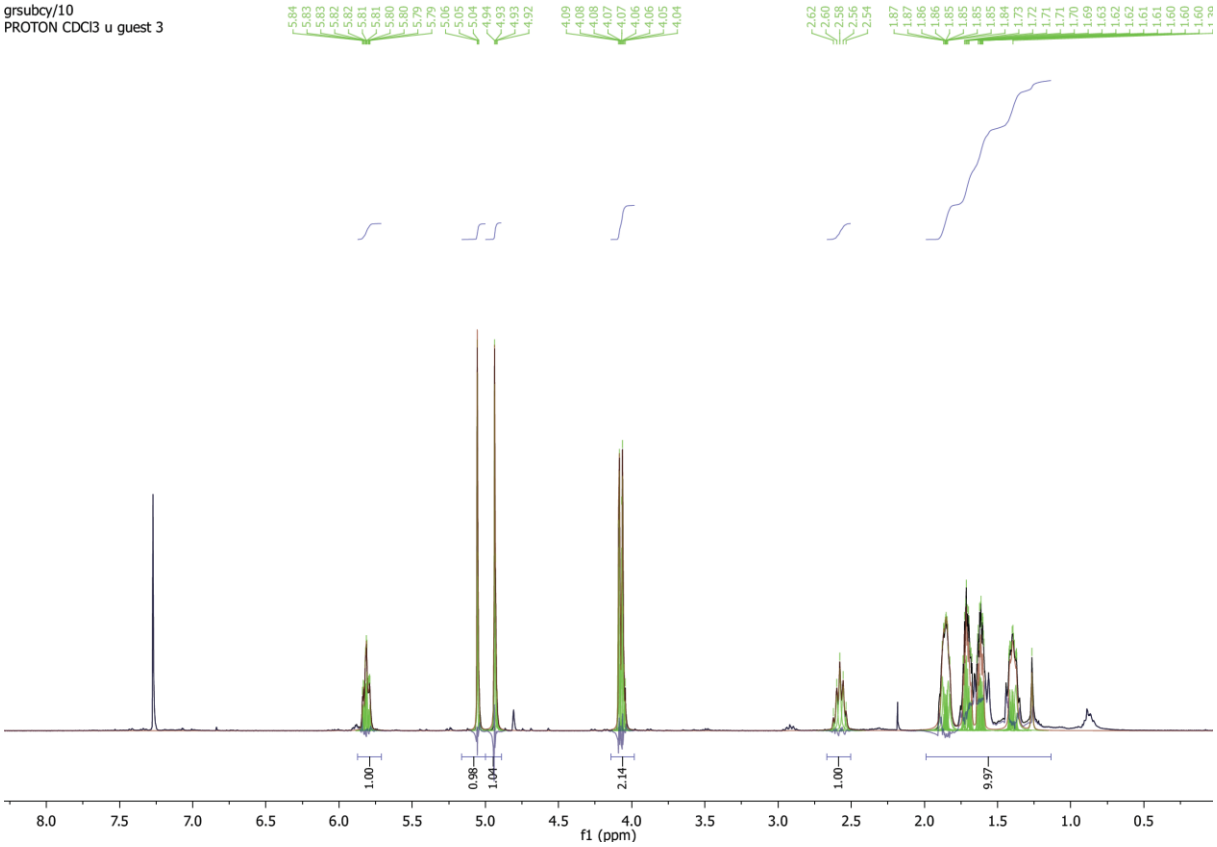

grsubome/10
PROTON CDCl3 u guest 4
5.40
5.29
4.25
4.23
3.84
1.00
2.10
2.15
3.30
f1 (ppm)

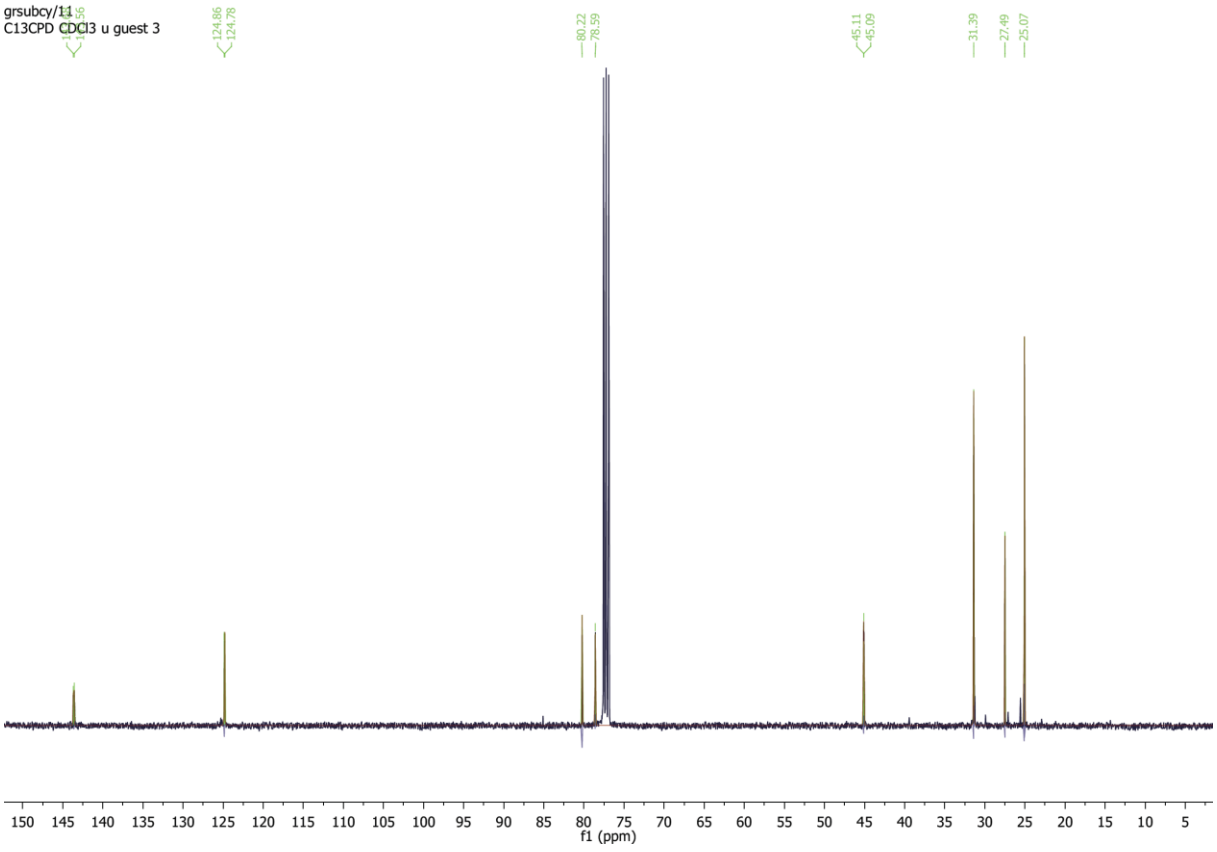

grsubome/11
C13CPD CDCl3 u guest 4
140.46
140.44
139.22
139.09
129.85
129.62
129.55
119.07
113.95
112.45
79.89
28.24
55.53
26.94
f1 (ppm)

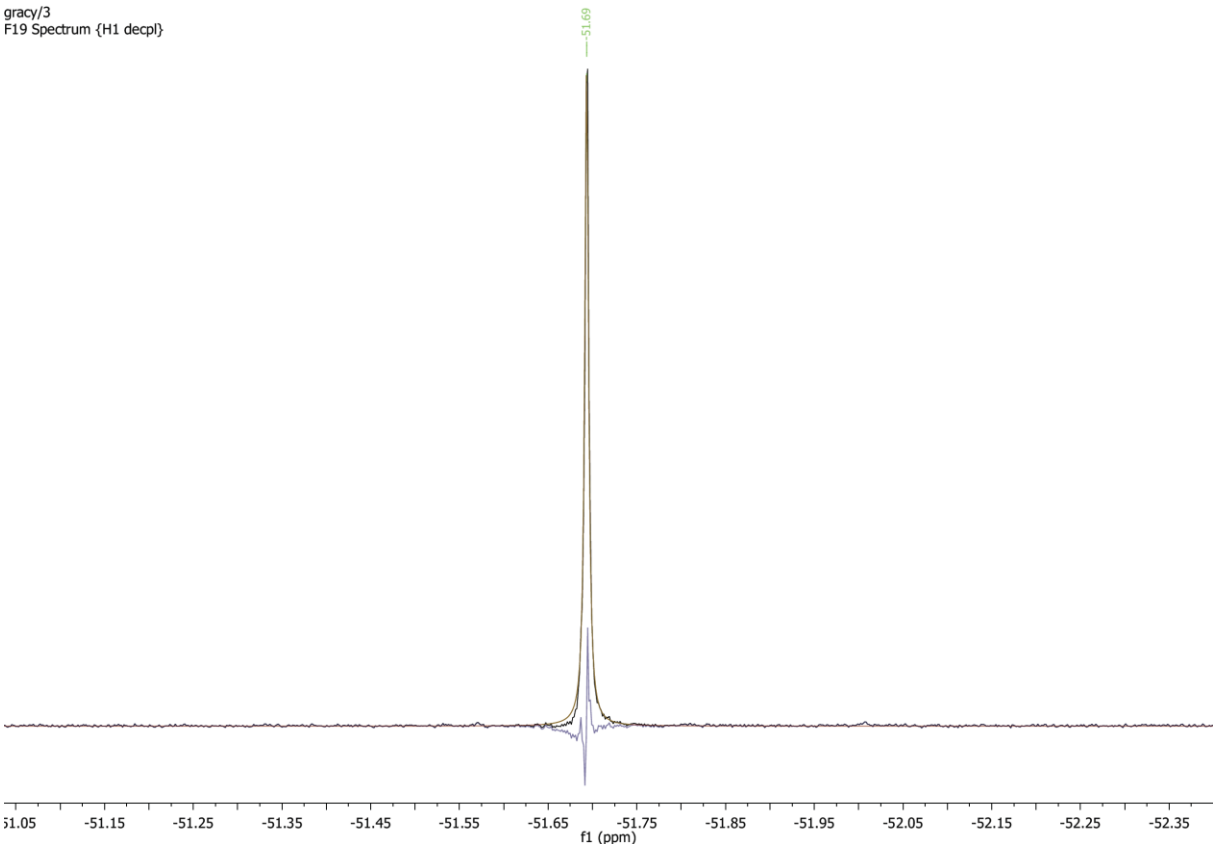


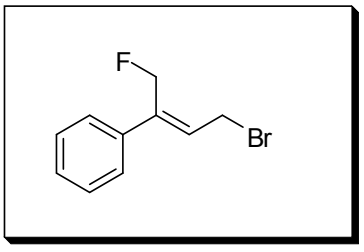


5b

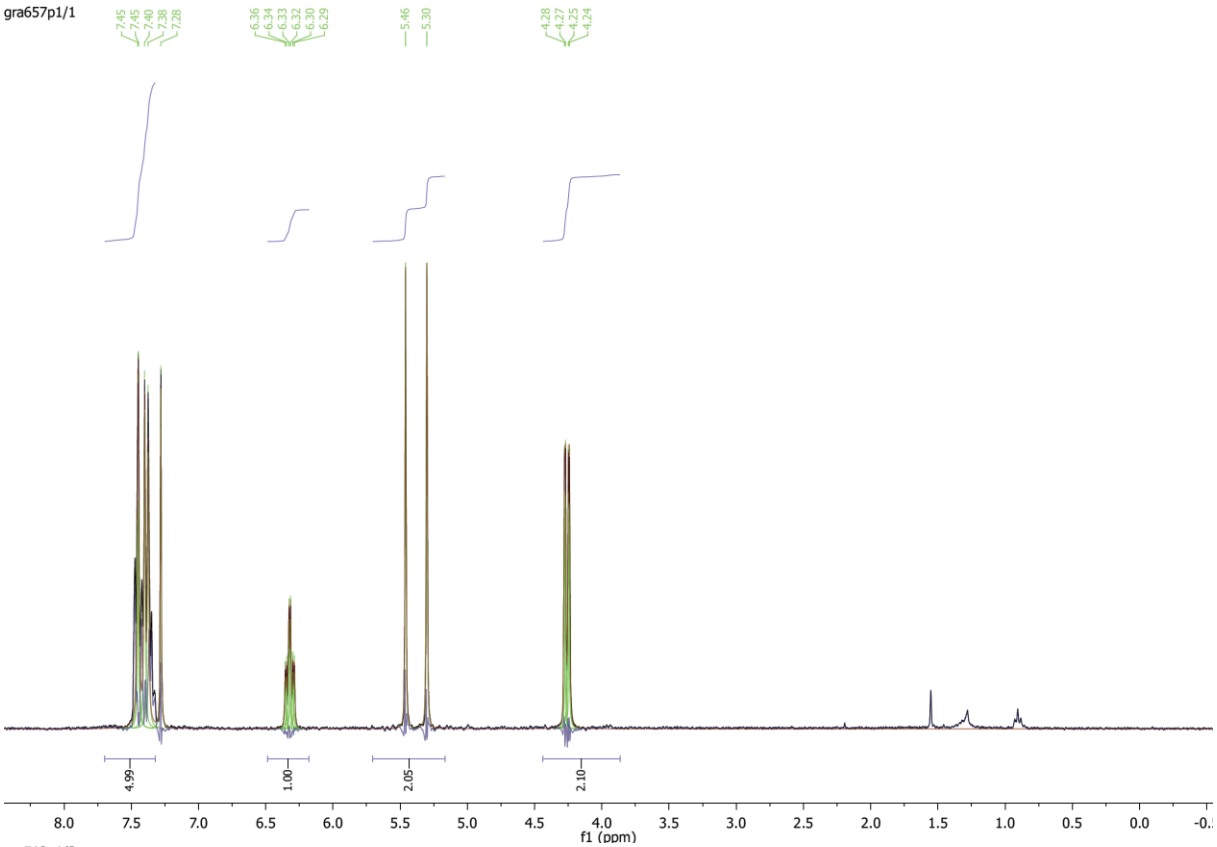

grsubcy/10
PROTON CDCl3 u guest 3
1.00
0.98
1.04
2.14
1.00
9.97
f1 (ppm)

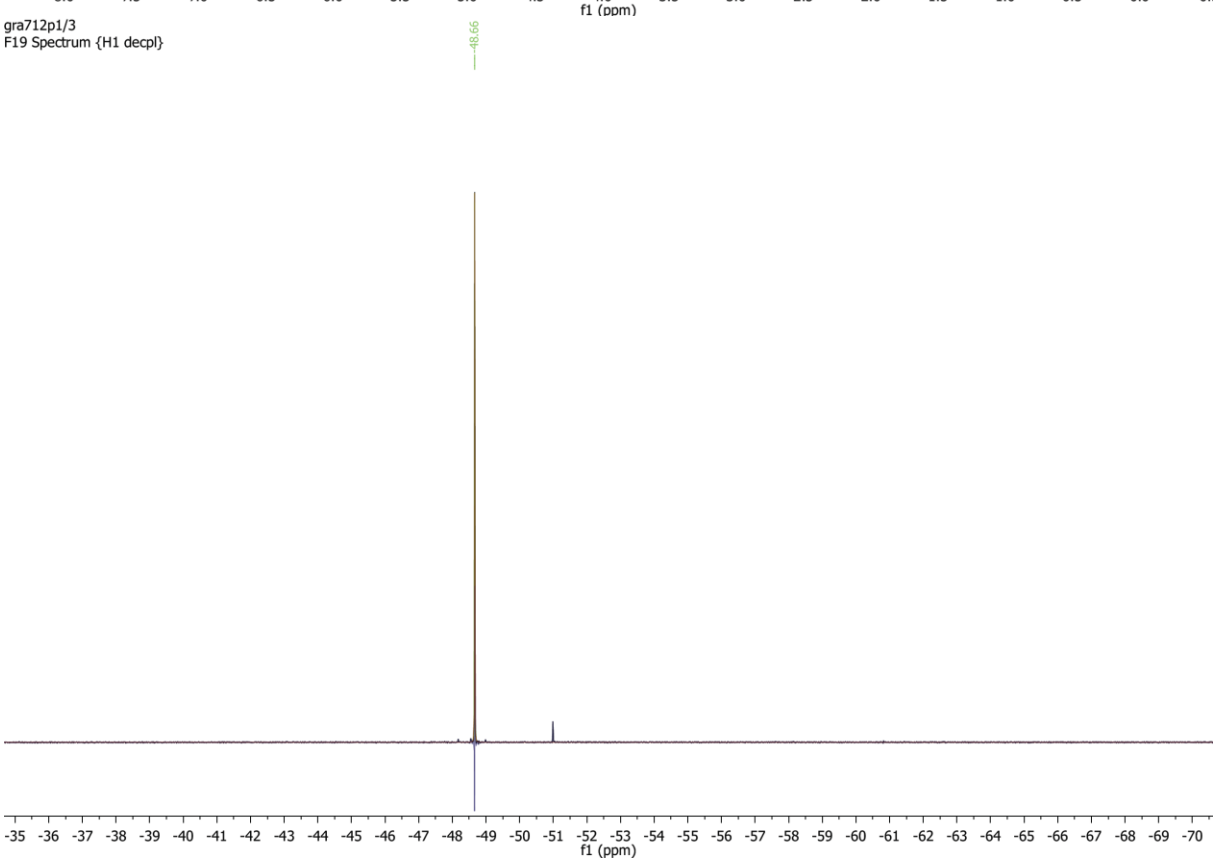

grsubcy/11
C13CPD CDCl3 u guest 3
124.86
124.78
80.22
78.59
45.11
45.09
31.39
27.49
25.07
f1 (ppm)





5h


gra657p1/1
7.45
7.45
7.40
7.38
7.28
6.36
6.34
6.33
6.32
6.30
6.29
5.46
5.30
4.28
4.27
4.25
4.24
4.99
1.00
2.05
2.10
f1 (ppm)


gra712p1/3
F19 Spectrum {H1 decpl}
-48.66
f1 (ppm)