Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Selective Nickel-catalyzed Fluoroalkylations of Olefins

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Table of contents

1. General comments

All reactions were carried out under an atmosphere of dry argon using standard Schlenk technique, Ace pressure tube or 300 mL autoclave equipped with 10 mL vials. Chemicals were purchased from *Aldrich*, *TCI*, *Alfa*, or *ABCR.* 3,3-Dimethylbutene was freezed and vacuumed for 3 times to get rid of H_2O and O_2 and stored in a Schlenk tube or stored in the fridge of the glovebox as received. Allylbenzene was distilled at 40 mbar with a 70 ºC oil bath and stored in a Schlenk tube. Perfluorobenzene was transferred to a Schlenk tube in glovebox as received. Other solvents were dried and degassed and stored in ©Aldrich Sure/Stor flasks or Schlenk tubes under argon. NaO(*t*-Bu), NaH, and TMEDA were stored in glovebox as received. Other chemicals were used as received. Products were purified by fast chromatography using silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck). Multiplets of NMR were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublets), dt (doublet of triplets), td (triplet of doublets), ddt (doublet of doublet of triplets), dtt (doublet of triplet of triplets), br.s (broad singlet), and m (multiplet).

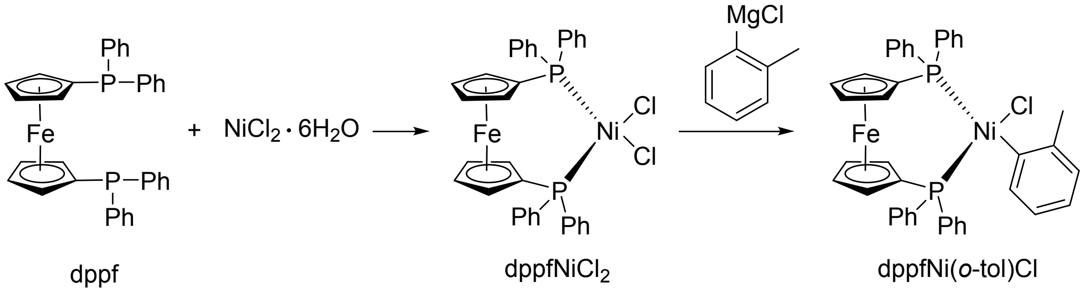
2. Analytical methods

NMRs were recorded on Bruker AV 300, AV 400, and Fourier 300 spectrometers at room temperature. For quantitative ^{19}F NMR a puls delay of 30s are required. NMR chemical shifts are reported in parts per million (ppm) downfield from TMS. ^{13}C and ^{1}H NMR spectra were referenced to signals of deutero solvents and residual protiated solvents, respectively. EPR spectra were recorded on a Bruker EMX CW-micro spectrometer (X-band) with a microwave power of Ca 6.9 mW and modulation frequency and amplitude of 100 kHz and up to 5 G, respectively. For monitoring the EPR spectra at low temperature, the EPR spectrometer was equipped with a temperature controller and liquid N_2 cryostat. The g values were calculated using $h\nu = g B_0\beta$ equation being h = Planck's constant, ν = frequency, B_0 = resonance field and β= Bohr magneton. High resolution mass spectra were recorded on a MAT 95XP ThermoFisher Mass Spectrometer using Electrospray Ionization mode. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). The data are given as mass units per charge (m/z).

3. General procedures

3.1 Synthesis of dppfNi(*o*-tol)Cl

dppfNi(*o*-tol)Cl was synthesized using the slightly modified method from a literature.[1]



Step 1: $NiCl_2 \cdot 6H_2O$ (8.5 mmol, 2.02 g) and EtOH (25 mL) were placed in an argon flushed 100 mL round Schlenk. Then the septum was removed, and dppf (8.5 mmol, 4.712 g) was added in one portion. The flask was fitted with

a reflux condenser, and the mixture was refluxed for 30 min with an 80 °C oil bath and then cooled to room temperature. Once cool, the flask was chilled to 0 °C with an ice bath for 10 min, after which the solid was collected by vacuum filtration, washed twice with EtOH (20 mL) and twice with ether (20 mL). Drying under vacuum yielded dppfNiCl_2 (4.98 g, 85 %) as a deep green powder.

Step 2: dppfNi(*o*-tol)Cl (6.81 mmol, 4.658 g) and 180 mL CH_2Cl_2 were placed in an argon flushed 100 mL round Schlenk. The solution was cooled to 0 °C with an ice bath, and *o*-tolylmagnesium chloride (6.81 mmol, 0.945 M in THF, 7.21 mL) was added dropwise with vigorous stirring. Near the end of the addition, the solution began to change in color from green to orange. The solution was stirred for 15 min at 0 °C, after which the solution was evaporated to dryness under reduced pressure. MeOH (25 mL) was added and the mixture was stirred for 5 min to form a suspension. After the suspension was cooled to 0 °C, the solvent was filtered out. The residue was washed with two portions of cold MeOH (10 mL), and dried under high vacuum to yield dppfNi(*o*-tol)Cl (4.63 g, 92 %) as a fine, bright yellow powder.

dppfNi(*o*-tol)Cl: **^{1}H NMR (400 MHz, CD_2Cl_2)** δ = 8.23 – 8.12 (m, 4H), 8.02 – 7.93 (m, 2H), 7.51 – 7.38 (m, 7H), 7.27 (td, *J* = 8.3, 2.0 Hz, 2H), 7.21 – 7.15 (m, 1H), 6.99 (t, *J* = 7.4 Hz, 1H), 6.76 (td, *J* = 8.2, 2.6 Hz, 2H), 6.66 – 6.54 (t, 2H), 6.43 (t, *J* = 7.4 Hz, 1H), 6.30 (t, *J* = 6.8 Hz, 1H), 6.10 (d, *J* = 7.1 Hz, 1H), 5.15 (s, 1H), 4.54 (m, 1H), 4.25 (s, 1H), 4.19 (s, 1H), 4.02 (d, *J* = 10.1 Hz, 2H), 3.52 (m, 1H), 3.33 (m, 1H), 2.44 (s, 3H).

^{31}P NMR (162 MHz, CD_2Cl_2) δ = 29.51 (d, *J* = 25.9 Hz, 1P), 12.12 (d, *J* = 25.9 Hz, 1P).

3.2 Synthesis of perfluoroalkylated alkenes

Step 1: *For the reactions of $C_{10}F_{21}I$*: 0.2 mmol $C_{10}F_{21}I$, 5 mol% dppfNi(*o*-tol)Cl and 2 equiv NaO(*t*-Bu) were placed in an Ace pressure tube. After flushed with argon, 0.5 mL perfluorobenzene and 2 equiv alkene was added. *For the reactions of other perfluoro reagents except CF_3Br and CF_3I*: 5 mol% dppfNi(*o*-tol)Cl and 2 equiv NaO(*t*-Bu) were placed in an Ace pressure tube. After flushed with argon, 1 mL perfluorobenzene, 0.5 mmol perfluoro reagent and 2 equiv alkene was added. *For the reactions of CF_3I*: 5 mol% dppfNi(*o*-tol)Cl and 2 equiv NaO(*t*-Bu) were placed in an Ace pressure tube. After flushed with argon, 2 equiv alkene and 1 mL perfluorobenzene (or DCE) stock solution containing 0.5 mmol CF_3I were added.

Step 2: Then the tube was sealed and heated for 16 h at specific temperature. After cooling to room temperature, 20 μL 1,4-difluorobenzene and 4 mL heptane or pentane were added to the reaction mixture. Then 250 μL of the solution was taken to measure the conversion and yield by ^{19}F NMR. Then, the products were isolated by fast chromatography using silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck) (Heptane or pentane elution).

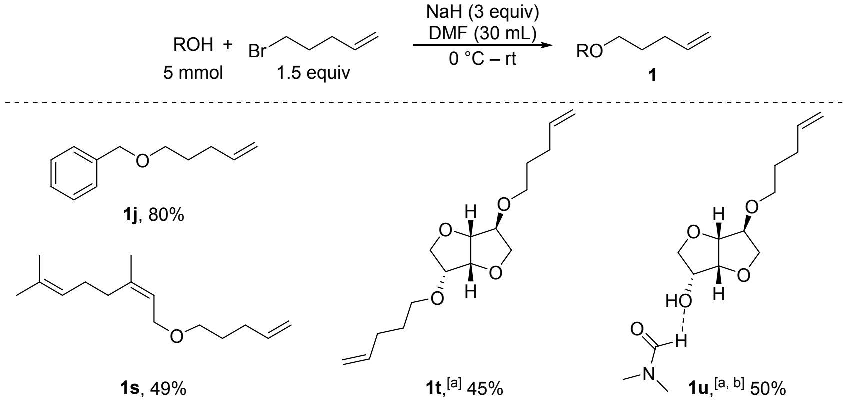
3.3 Synthesis of 3,3-dimethyl-trifluoromethyl butene using CF_3Br

The reaction was carried out in a Parr Instruments 4560 series 300 mL autoclave containing an alloy plate with wells for five 10 mL glass vials. Under argon atmosphere, 0.5 mmol 3,3-dimethylbutene, 5 mol% dppfNi(*o*-tol)Cl and 2 equiv NaO(*t*-Bu), 1 mL perfluorobenzene and a magnetic stirring bar were placed in the vial, which were then capped with a septum equipped with an inlet needle and flushed with argon. The vial was placed in an alloy plate, which was then placed in the autoclave. Once sealed, the autoclave was purged three times with 15 bar nitrogen and then pressurized with 5 bar CF_3Br and 40 bar N_2 at room temperature and heated at 50 °C for 16 h. It was then cooled to room temperature and vented to discharge the excess gas. 20 μL 1,4-difluorobenzene and 4 mL pentane were then added to the reaction mixture. And 250 μL of the pentane solution was taken and mixed with

350 µL $CDCl_3$ to measure the conversion and yield by ^{19}F NMR.

3.4 Synthesis of alkenes

In this project, we synthesized 4 alkenes which are not commercially available using slightly modified procedures from literature.[2] The structure of these olefins are shown in Scheme S1. Firstly, the corresponding alcohol (0.5 mmol), NaH (3 equiv) and a magnetic stirring bar were placed in a 100 mL round Schlenk. After flushed with argon, DMF (30 mL) was added. Then the flask was cooled to 0 °C using an ice bath. Once cool, 5-bromopentene was added dropwise using a syringe pump. Thereafter, the reaction could warm slowly to rt and was further stirred overnight. After isolated by fast chromatography using silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck) (heptane and ethyl acetate elution), the pure products were afforded in 45% to 80% yields.



Scheme S1. Synthesis of the alkenes. Reactions were performed under Ar, yields were of the isolated products. [a] These two products were obtained by one reaction. [b] As indicated by NMR, The -OH group of the product was supposed to be protected by DMF. It will release -OH group again during the perfluoroalkylation reaction.

4. Kinetic time course

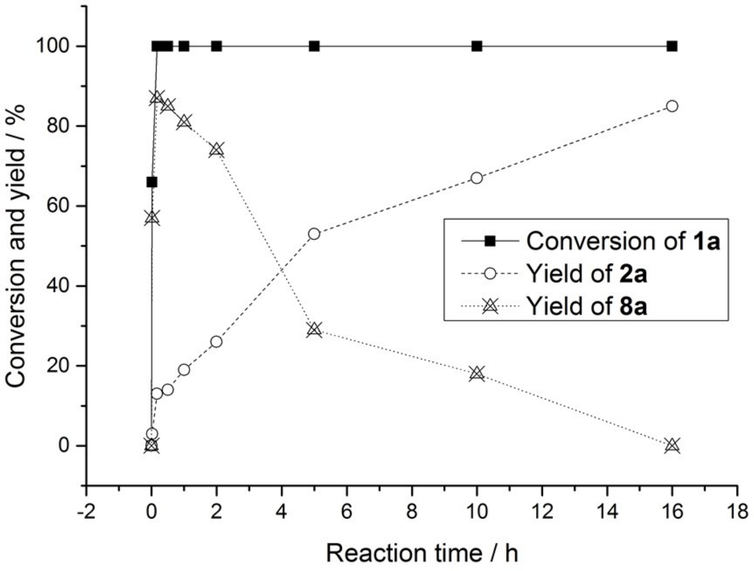


Figure S1. Kinetic time course of the nickel-catalyzed perfluorodecylation of 3,3-dimethylbutene. Reaction conditions: $C_{10}F_{21}I$ (0.2 mmol), **1a** (0.5 mL), dppfNi(*o*-tol)Cl (5 mol%), NaO(*t*-Bu) (2 equiv), 50 °C, Ar, conversions and yields were determined by ^{19}F NMR using 1,4-difluorobenzene as the standard. The conversion of **1a** was completed within only 10 mins. There were two inflection points of the yield of **2a**. The first inflection point at 10 mins was caused by the ending of direct perfluoroalkylation. The second inflection point at 5 h was caused by the low concentration of **8a**.

5. In situ EPR

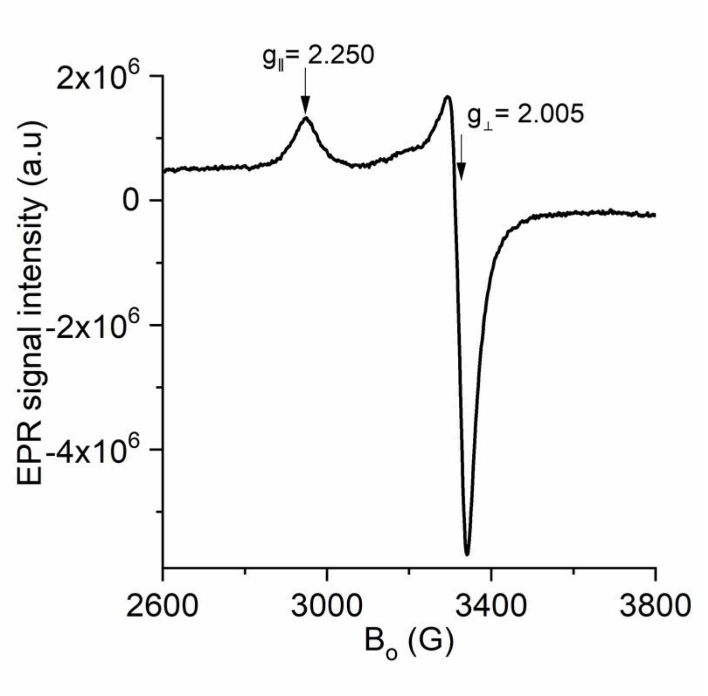


Figure S2. EPR spectrum of the reaction mixture **1a** (0.5 mL), dppfNi(o-tol)Cl (5 mol%) and NaO(*t*-Bu) (2 equiv) measured at 100 K under Ar. The spectrum shows an axial EPR signal due to the formation of Ni(I) species (**A**).

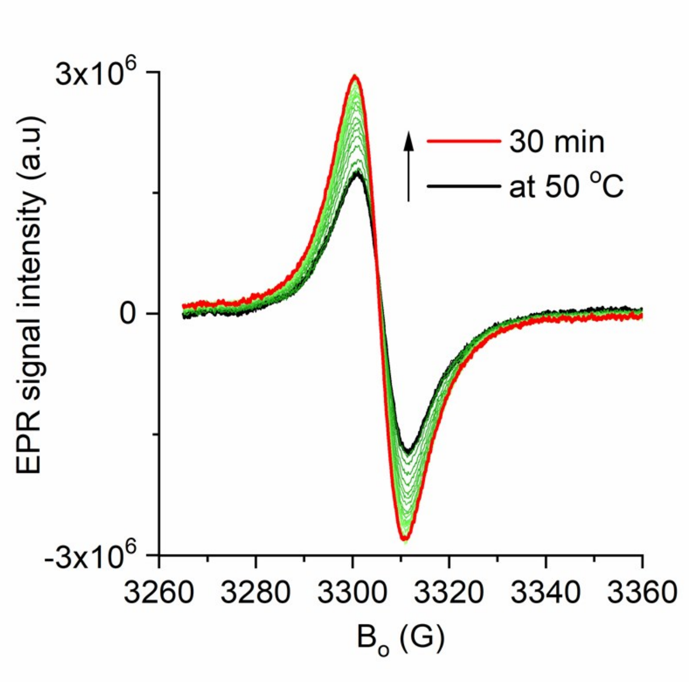
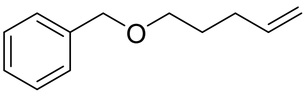


Figure S3. In situ EPR spectra with time of the reaction mixture $C_{10}F_{21}I$ (0.2 mmol), **1a** (0.5 mL), dppfNi(o-tol)Cl (5 mol%) and NaO(*t*-Bu) (2 equiv) measured at 50 °C under Ar. The spectra show EPR signal at g = 2.005 without hyperfine splitting due to the formation of organic radicals (**B and C**). The Ni(I) signal is not observable at reaction temperature due to the short relaxation time.[3]

6. Characterization data

6.1 Characterization data of alkenes



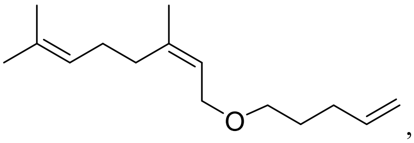
1j, , 80%, colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.38 – 7.03 (m, 5H), 5.74 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 5.06 – 4.74 (m, 2H), 4.41 (s, 2H), 3.40 (t, *J* = 6.5 Hz, 2H), 2.20 – 1.94 (m, 2H), 1.75 – 1.43 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 138.57, 138.22, 128.27, 127.54, 127.42, 114.66, 72.82, 69.65, 30.30, 28.92.

MS (EI, 70 eV): m/z (%) = 176.1 (7) [M]+, 175.1 (14) [M-H]+, 107.1 (13), 91.1 (100), 79.1 (5), 65.1 (9), 55.1 (3).

TLC (silica gel 60 F_{254}): Ethyl acetate/heptane = 1:10, Rf = 0.24 (KMnO4 stain).



1u, , 49%, clolorless oil.

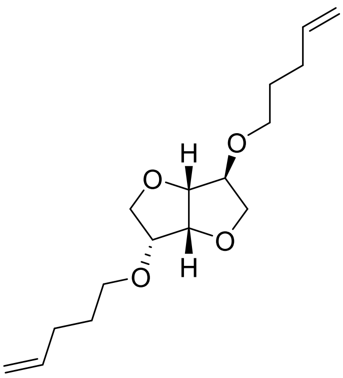
^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.89 (ddt, *J* = 16.8, 9.9, 6.5 Hz, 1H), 5.43 (t, *J* = 6.9 Hz, 1H), 5.25 – 4.94 (m, 3H), 4.01 (d, *J* = 6.9 Hz, 2H), 3.49 (t, *J* = 6.6 Hz, 2H), 2.26 – 2.07 (m, 6H), 1.87 – 1.64 (m, 9H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 140.04, 138.28, 131.76, 123.86, 122.02, 114.56, 69.50, 66.95, 32.20, 30.34, 28.97, 26.67, 25.62, 23.41, 17.56.

HRMS (EI): [M]+ calcd for $C_{15}H_{26}O_1$, 222.19782; found, 222.19802.

TLC (silica gel 60 F_{254}): Ethyl acetate/heptane = 1:10, Rf = 0.67.

TLC (silica gel 60 F_{254}): Ethyl acetate/heptane = 1:2, Rf = 0.76.

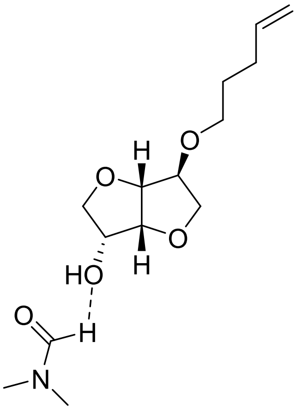


1v, , 45%, clolorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.91 – 5.61 (m, 2H), 5.11 – 4.83 (m, 4H), 4.66 – 4.39 (m, 2H), 4.05 – 3.79 (m, 5H), 3.74 – 3.35 (m, 5H), 2.10 (m, 4H), 1.78 – 1.51 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 138.03, 137.93, 114.83, 114.72, 86.23, 84.35, 80.32, 80.08, 73.37, 70.02, 69.68, 68.83, 30.09, 30.05, 28.82, 28.74.

MS (EI, 70 eV): m/z (%) = 282.2 (2) [M]+, 179.1 (12), 84.1 (42), 69.1 (100).



1w, , 50%, clolorless oil.

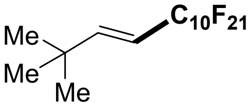
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.91 (s, 1H), 5.73 (ddt, *J* = 14.2, 10.6, 7.0 Hz, 1H), 5.11 – 4.77 (m, 2H), 4.60 (t, *J* = 3.5 Hz, 1H), 4.36 (d, *J* = 3.1 Hz, 1H), 4.20 (s, 1H), 3.98 – 3.66 (m, 5H), 3.66 – 3.25 (m, 3H), 2.88 (s, 3H), 2.80 (s, 3H), 2.04 (q, *J* = 6.9 Hz, 2H), 1.63 (p, *J* = 6.9 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.38, 137.78, 114.46, 88.10, 80.16, 79.67, 76.06, 75.65, 69.74, 69.50, 36.25, 31.15, 29.78, 28.54.

MS (EI, 70 eV): m/z (%) = 214.2 (1) [M-DMF]+, 159.1 (14), 128.1 (14), 85.1 (24), 84.1 (60), 69.1 (100), 68.1 (73), 67.2 (21).

6.2 Characterization data of perfluorodecylated alkenes

Notes: For the ^{13}C NMR (F coupled), peaks for the C of perfluoroalkyl chain and C adjacent to perfluoroalkyl chain (in most cases) are too broad to be assigned (not shown in the following data).



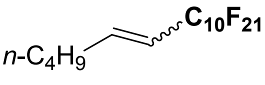
2a, , colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.39 (dt, *J* = 16.0, 2.2 Hz, 1H), 5.48 (dt, *J* = 16.0, 12.0 Hz, 1H), 1.08 (s, 9H).

^{19}F NMR (282 MHz, $CDCl_3$): δ = -81.03 (t, *J* = 10.0 Hz, 3F), -111.31 (q, *J* = 12.3 Hz, 2F), -120.65 – -124.69 (14F), -126.33 (br.s 2F).

TLC (silica gel 60 F_{254}): pentane, Rf = 0.9, $KMnO_4$ stain.

HRMS (EI): [M]+ calcd for $C_{16}H_{11}F_{21}$, 602.05199; found, 602.05119.



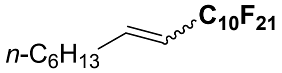
2b, , colorless oil, mixture of *E* and *Z* isomers (84:16).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.52 – 5.99 (m, =C*H*CF_2- (*E/Z*), 1H), 5.74 – 5.27 (m, -C*H*=CHCF_2- (*E/Z*), 1H), 2.47 – 1.99 (m, -CH_2-CH= (*E/Z*), 2H), 1.50 – 1.12 (m, -CH_2-, 4H), 0.92 (t, *J* = 7.2 Hz, -CH_3, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 143.29, 116.76, 77.00, 31.69, 30.07, 22.03, 13.74.

^{19}F NMR (282 MHz, $CDCl_3$): δ = -80.80 (t, *J* = 10.0 Hz, -CF_3, 3F), -106.67 (q, *J* = 13.7, 13.3 Hz, =CHCF_2-, *Z*-isomer), -111.22 (q, *J* = 11.2, 10.0 Hz, =CHCF_2-, *E*-isomer), -121.48 (br.s, -CF_2-, 2F), -121.84 (br.s, -CF_2-, 8F) -122.72 (br.s, -CF_2-, 2F), -123.56 (br.s, =CHCF_2CF_2-, *E*-isomer), -123.74 (br.s, =CHCF_2CF_2-, *Z*-isomer), -126.14 (br.s, -CF_2-, 2F).

HRMS (EI): [M]+ calcd for $C_{16}H_{11}F_{21}$, 602.05199; found, 602.05121.

2c, 

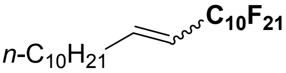
, yellow sticky oil, mixture of *E* and *Z* isomers (87:13).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.58 – 5.98 (m, =C*H*CF$_2$- (*E/Z*), 1H), 5.74 – 5.26 (m, -C*H*=CHCF$_2$- (*E/Z*), 1H), 2.41 – 2.08 (m, -C*H*$_2$-CH= (*E/Z*), 2H), 1.44 (m, -C*H*$_2$-, 2H), 1.29 (br.s, -C*H*$_2$-, 6H), 0.89 (t, *J* = 6.9 Hz, -C*H*$_3$, 3H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 143.22 (t, *J* = 9.1 Hz, =*C*HCF$_2$-), 116.74 (t, *J* = 22.9 Hz, -*C*H=CHCF$_2$-), 76.94, 31.96, 31.48, 28.58, 27.90, 22.46, 13.84.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.16 (t, *J* = 9.9 Hz, -C*F*$_3$, 3F), -106.86 (q, *J* = 13.2 Hz, =CHC*F*$_2$-, *Z*-isomer), -111.46 (q, *J* = 11.7, 12.8 Hz, =CHC*F*$_2$-, *E*-isomer), -121.65 (br.s, -C*F*$_2$-, 2F), -122.05 (br.s, -C*F*$_2$-, 8F), -122.96 (br.s, -C*F*$_2$-, 2F), -123.77 (br.s, =CHCF$_2$C*F*$_2$-, *E*-isomer), -123.94 (br.s, =CHCF$_2$C*F*$_2$-, *Z*-isomer), -126.41 (br.s, -C*F*$_2$-, 2F).

HRMS (EI): [M]+ calcd for $C_{18}H_{15}F_{21}$, 630.08329; found, 630.08205.

2d, 

, colorless oil, mixture of *E* and *Z* isomers (88:12).

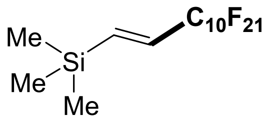
^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.55 – 6.04 (m, =C*H*CF$_2$- (*E/Z*), 1H), 5.94 – 5.34 (m, -C*H*=CHCF$_2$- (*E/Z*), 1H), 2.50 – 2.09 (m, -C*H*$_2$-CH= (*E/Z*), 2H), 1.56 – 1.38 (m, -C*H*$_2$-, 2H), 1.28 (br.s, -C*H*$_2$-, 14H), 0.96 – 0.74 (t, *J* = 6.9 Hz, -C*H*$_3$, 3H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 143.27 (t, *J* = 9.0 Hz, =*C*HCF$_2$-), 116.94 (t, *J* = 23.0 Hz, -*C*H=CHCF$_2$-), 32.07, 32.00, 29.67, 29.61, 29.42, 29.06, 28.08, 22.74, 13.98.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.31 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -106.94 (q, *J* = 13.5 Hz, =CHC*F*$_2$-, *Z*-isomer), -111.56 (q, *J* = 11.4, 10.4 Hz, =CHC*F*$_2$-, *E*-isomer), -121.71 (br.s, -C*F*$_2$-, 2F), -122.12 (br.s, -C*F*$_2$-, 8F), -123.04 (br.s, -C*F*$_2$-, 2F), -123.82 (br.s, =CHCF$_2$C*F*$_2$-, *E*-isomer), -124.01 (br.s, =CHCF$_2$C*F*$_2$-, *Z*-isomer), -126.52 (br.s, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 131.1 (17), 111.2 (31), 98.1 (30), 97.1 (61), 85.1 (46), 84.1 (61), 83.1 (58), 71.1 (64), 70.1 (91), 69.1 (95), 57.1 (100), 56.1 (92), 55.1 (64).

HRMS (EI): [M]+ calcd for $C_{22}H_{23}F_{21}$, 686.14589; found, 686.14522.

2e, 

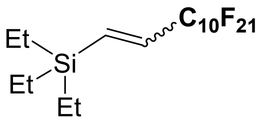
, light yellow oil.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.57 (dt, *J* = 19.0, 2.3 Hz, =C*H*CF$_2$-, 1H), 5.87 (dt, *J* = 19.1, 11.0 Hz, -C*H*=CHCF$_2$-, 1H), 0.00 (s, -C*H*$_3$, 9H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 145.99 (t, *J* = 5.9 Hz, -*C*=CHCF$_2$-), 132.04 (t, *J* = 24.1 Hz, -C=*C*HCF$_2$-), 0.00.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.12 (C*F*$_3$, 3F), -113.43 (q, *J* = 11.6, 11.2 Hz, alkenyl- C*F*$_2$, 2F), -120.90 – -124.77 (m, 14F), -126.40 (br.s, 2F). -81.12 (t, *J* = 9.9 Hz, -C*F*$_3$, 3F), -113.43 (q, *J* = 11.6, 11.2 Hz, =CHC*F*$_2$-, 2F), -121.64 (br.s, -C*F*$_2$-, 2F), -122.03 (br.s, -C*F*$_2$-, 8F), -122.94 (br.s, -C*F*$_2$-, 2F), -123.67 (br.s, =CHCF$_2$C*F*$_2$-, 2F), -126.40 (br.s, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 603.1 (27) [M-Me]+, 214.1 (4), 184.1 (10), 153.1 (77), 131.0 (27), 103.1 (17), 77.1 (100), 53.1 (15).

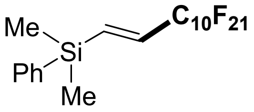


2f, , colorless oil, mixture of *E* and *Z* isomers (84:16).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.67 (dt, *J* = 19.2, 2.3 Hz, =C*H*CF$_2$- (*E*), 0.8H), 6.49 – 6.28 (m, -C*H*=C*H*CF$_2$- (*Z*), 0.4H), 6.05 (dt, *J* = 19.2, 10.9 Hz, -C*H*=CCF$_2$- (*E*),0.8H), 0.96 (t, *J* = 7.9 Hz, -C*H*$_3$, 9H), 0.66 (q, *J* = 8.4 Hz, -C*H*$_2$-, 6H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.98 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -110.46 (q, *J* = 12.1 Hz, alkenyl- C*F*$_2$ (*Z*), 0.4F), -113.29 (q, *J* = 11.4, alkenyl- C*F*$_2$ (*E*), 1.6F), -121.22 – -123.80 (14F), -126.26 (br.s, 2F).

MS (EI, 70 eV): m/z (%) = 631.2 (100) [M-Et]+, 169.0 (19), 167.1 (19), 131.0 (23), 119.0 (26), 105.1 (49), 95.1 (33), 77.1 (22), 69.1 (27).



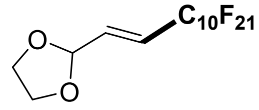
2g, , colorless oil.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 7.52 – 7.46 (m, -Ph, 2H), 7.43 – 7.35 (m, -Ph, 3H), 6.85 (dt, *J* = 19.0, 2.3 Hz, =C*H*CF$_2$-, 1H), 6.07 (dt, *J* = 19.0, 10.9 Hz, -C*H*=CCF$_2$-, 1H), 0.44 (s, -C*H*$_3$, 6H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 142.05 (t, *J* = 5.6 Hz, -*C*=CHCF$_2$-), 135.73, 133.77, 131.30 (t, *J* = 24.0 Hz, -C=*C*HCF$_2$-), 129.72, 128.09, -3.32.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.83 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -113.24 (q, *J* = 12.0 Hz, =CHC*F*$_2$-, 2F), -121.44 (br.s, -C*F*$_2$-, 2F), -121.84 (br.s, -C*F*$_2$-, 10F), -122.73 (br.s, -C*F*$_2$-, 2F), -123.39 (br.s, -C*F*$_2$-, 2F), -126.17 (br.s, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 665.1 (29) [M-Me]+, 584.1 (22), 215.1 (51), 165.1 (100), 139.0 (49), 135.1 (25), 115.1 (24), 77.1 (64).



2h, , colorless sticky oil.

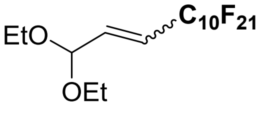
^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.37 (ddt, *J* = 15.8, 4.2, 2.1 Hz, 1H), 6.05 (dt, *J* = 16.1, 12.1 Hz, 1H), 5.47 (m, 1H), 4.08 – 3.84 (m, 4H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 137.94, 120.45, 100.57, 65.11.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.79 (t, *J* = 10.0 Hz, 3F), -112.52 (q, *J* = 12.1 Hz, alkenyl- C*F*$_2$, 2F), -120.13 – -124.31 (m, 14F), -126.12 (2F).

HRMS (EI): [M-H]+ calcd for $C_{15}H_6O_2F_{21}$, 617.00270; found, 616.99916.

TLC (silica gel 60 F$_{254}$): Ethyl acetate/heptane = 1:10, Rf = 0.49 ($KMnO_4$ stain).



2i, , colorless oil, mixture of *E* and *Z* isomers (87:13).

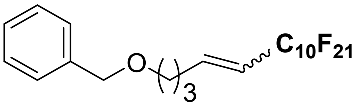
^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.39 – 6.32 (m, =C*H*CF$_2$- (*E*)), 6.20 – 6.12 (m, =C*H*CF$_2$- (*Z*)), 6.12 – 5.90 (m, -C*H*=CHCF$_2$- (*E*)), 5.78 – 5.55 (m, -C*H*=CHCF$_2$- (*Z*)), 5.33 – 5.25 (m, -C*H*(OEt)$_2$ (*Z*)), 5.11 – 5.02 (m, -C*H*(OEt)$_2$

(*E*)), 3.76 – 3.44 (m, -C*H*$_2$-, 4H), 1.22 (t, *J* = 7.1 Hz, -C*H*$_3$, 6H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 139.28 (t, *J* = 8.7 Hz, -*C*=CHCF$_2$-), 120.24 (t, *J* = 23.5 Hz, -C=*C*HCF$_2$-), 98.15, 77.00, 61.16, 15.06.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.02 (t, *J* = 10.0 Hz, C*F*$_3$, 3F), -106.89 (q, *J* = 13.7, 13.1 Hz, =CHC*F*$_2$- (*Z*)), -112.41 (q, *J* = 12.4 Hz, =CHC*F*$_2$- (*E*)), -121.59 (br.s, -C*F*$_2$-, 2F), -121.96 (br.s, -C*F*$_2$-, 10F), -122.87 (br.s, -C*F*$_2$-, 2F), -123.46 (br.s, -C*F*$_2$-, 2F), -126.31 (br.s, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 603.1 (48) [M-EtO]+, 575.1 (100), 238.1 (5), 218.9 (8), 169.0 (24), 131.1 (47), 106.1 (66), 69.0 (54).

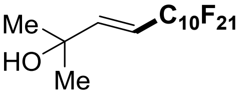
2j, 

, colorless oil, mixture of *E* and *Z* isomers (82:18).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 7.37 – 7.28 (m, -Ph, 5H), 6.49 – 5.41 (m, -C*H*=C*H*CF$_2$-, *E*/*Z*, 2H), 4.52 (s, Ph-C*H*$_2$-, 2H), 3.54 – 3.49(t, *J* = 6.2 Hz, -OC*H*$_2$CH$_2$-, 2H), 2.45 – 2.30 (m, -C*H*$_2$-, 2H), 1.83 – 1.64 (m, -C*H*$_2$-, 2H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.98 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -106.81 (q, *J* = 13.5 Hz, =CHC*F*$_2$- (*Z*)), -111.39 (q, *J* = 11.5, 10.5 Hz, =CHC*F*$_2$- (*E*)), -121.53 (br.s, -C*F*$_2$-, 2F), -121.92 (br.s, -C*F*$_2$-, 8F), -122.82 (br.s, -C*F*$_2$-, 2F), -123.57 and -123.78 (br.s, =CHCF$_2$C*F*$_2$- (*E*/*Z*), 2F), -126.27 (br.s, -C*F*$_2$-, 2F).

HRMS (EI): [M]+ calcd for $C_{22}H_{15}O_1F_{21}$, 694.07821; found, 694.07752.

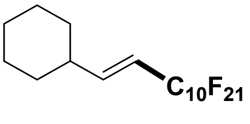
2k, 

, colorless oil.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.71 – 6.41 (m, =C*H*CF$_2$-, 1H), 5.87 (dt, *J* = 15.4, 11.5 Hz, -C*H*=CHCF$_2$-, 1H), 1.54 (s, -OH), 1.51 (s, -C*H*$_3$, 6H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.87 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -111.49 (q, *J* = 12.2 Hz, =CHC*F*$_2$-, 2F), -121.44 (br.s, -C*F*$_2$-, 2F), -121.87 (br.s, -C*F*$_2$-, 8F), -122.76 (br.s, -C*F*$_2$-, 2F), -123.56 (br.s, -C*F*$_2$-, 2F), -126.19 (br.s, -C*F*$_2$-, 2F).

HRMS (EI): [M-Me]+ calcd for $C_{14}H_6O_1F_{21}$, 589.00778; found, 589.00799.

2l, 

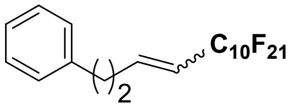
, colorless oil.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.35 (ddt, *J* = 15.9, 6.7, 2.2 Hz, =C*H*CF$_2$-, 1H), 5.72 – 5.22 (m, -C*H*=CHCF$_2$-, 1H), 2.20 – 2.04 (m, 1H), 1.79 – 1.65 (m, 4H), 1.37 – 1.08 (m, 6H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 148.16 (t, *J* = 8.8 Hz, -*C*=CHCF$_2$-), 114.64 (t, *J* = 23.0 Hz, -C=*C*HCF$_2$-), 40.22, 31.69, 25.87, 25.65.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.08 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -111.42 (q, *J* = 12.1 Hz, =CHC*F*$_2$-, 2F), -121.55 (br.s, -C*F*$_2$-, 2F), -122.01 (br.s, -C*F*$_2$-, 8F), -122.91 (br.s, -C*F*$_2$-, 2F), -123.78 (br.s, =CHCF$_2$C*F*$_2$-, 2F), -126.37 (br.s, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 628.1 (9) [M]+, 609.1 (15), 589.1 (1), 567.1 (9), 169.0 (6), 131.0 (8), 103.1 (14), 82.1 (100), 81.1 (32), 69.1 (25), 67.1 (52).

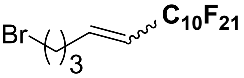
2m,  , colorless oil, mixture of *E* and *Z* isomers (83:17).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 7.64 – 6.84 (m, -Ph, 5H), 6.70 – 6.39 (m, =C*H*CF$_2$- (*E*)), 6.21 – 6.03 (m, =C*H*CF$_2$- (*Z*)), 5.99 – 5.82 (m, -C*H*=CHCF$_2$- (*Z*)), 5.69 – 5.33 (m, -C*H*=CHCF$_2$- (*E*)), 2.81 – 2.74 (m, PhC*H$_2$*-, 2H), 2.72 – 2.64 (m, -C*H$_2$*CH= (*Z*)), 2.57 – 2.43 (m, -C*H$_2$*CH= (*E*)).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 142.07 (t, *J* = 8.9 Hz, -*C*=CHCF$_2$-), 140.36, 128.52, 128.35, 126.29, 117.62 (t, *J* = 22.9 Hz, -C=*C*HCF$_2$-), 34.40, 33.74.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.98 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -106.98 (q, *J* = 13.3 Hz, =CHC*F$_2$*- (*Z*)), -111.47 (q, *J* = 11.5, 10.6 Hz, =CHC*F$_2$*- (*E*)), -121.55 (br.s, -C*F$_2$*-, 2F), -121.94 (br.s, -C*F$_2$*-, 8F), -122.84 (br.s, -C*F$_2$*-, 2F), -123.61 and -123.83 (br.s, =CHCF$_2$C*F$_2$*- (*E/Z*), 2F), -126.27 (br.s, -C*F$_2$*-, 2F).

HRMS (EI): [M-]+ calcd for $C_{20}H_{11}F_{21}$, 650.05199; found, 650.05178.

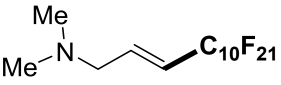
2n,  , colorless oil, mixture of *E* and *Z* isomers (78:22).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.37 (dtt, *J* = 16.2, 7.1, 2.3 Hz, =C*H*CF$_2$- (*E*)), 6.15 – 6.05 (m, =C*H*CF$_2$- (*Z*)), 5.76 – 5.50 (m, -C*H*=CHCF$_2$- (*E/Z*), 1H), 3.19 (t, *J* = 6.7 Hz, BrC*H$_2$*-, 2H), 2.49 – 2.31 (m, -C*H$_2$*CH= (*E/Z*), 2H), 1.98 (p, *J* = 6.8 Hz, BrCH$_2$C*H$_2$*-, 2H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.74 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -106.79 (q, *J* = 13.1 Hz, =CHC*F$_2$*- (*Z*)), -111.44 (q, *J* = 11.9, 11.2 Hz, =CHC*F$_2$*- (*E*)), -121.40 (br.s, -C*F$_2$*-, 2F), -121.78 (br.s, -C*F$_2$*-, 8F), -122.68 (br.s, -C*F$_2$*-, 2F), -123.39 and -123.63 (br.s, =CHCF$_2$C*F$_2$*- (*E/Z*), 2F), -126.09 (br.s, -C*F$_2$*-, 2F).

MS (EI, 70 eV): m/z (%) = 631.2 (100), 169.0 (19), 167.1 (19), 131.0 (23), 119.0 (26), 105.1 (49), 95.1 (33), 77.1 (22), 69.1 (27).

TLC (silica gel 60 F$_{254}$): Ethyl acetate/heptane = 1:10, Rf = 0.71.

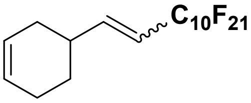
2o,  , brown oil

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.38 – 6.47 (m, =C*H*CF$_2$-, 1H), 5.88 – 5.74 (m, -C*H*=CHCF$_2$-, 1H), 3.06 (s, -C*H$_2$*-, 2H), 2.24 (s, -Me, 6H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 140.17 (t, *J* = 8.9 Hz, -*C*=CHCF$_2$-), 118.89 (t, *J* = 23.3 Hz, C=*C*HCF$_2$-), 60.11, 45.23.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.97 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -111.75 (q, *J* = 11.1, 10.2 Hz, =CHC*F$_2$*-, 2F), -121.57 (br.s, -C*F$_2$*-, 2F), -121.94 (br.s, -C*F$_2$*-, 8F), -122.84 (br.s, -C*F$_2$*-, 2F), -123.58 (br.s, =CHCF$_2$C*F$_2$*-, 2F), -126.28 (br.s, -C*F$_2$*-, 2F).

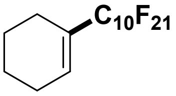
MS (EI, 70 eV): m/z (%) = 603.1 (56) [M]+, 602.1 (66) [M-H]+, 584.1 (100), 484.1 (1), 355.1 (1), 284.1 (1), 134.1 (23), 84.1 (24), 58.1 (44).

2p,  , colorless oil, mixture of *E* and *Z* isomers (89:11).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.42 (ddt, *J* = 15.9, 7.0, 2.2 Hz, =C*H*CF$_2$- (*E*)), 5.83 – 5.53 (m, -C*H*=CHCF$_2$- (*E*) and -CH$_2$C*H*=C*H*CH$_2$-), 5.15 – 4.97 (m, =C*H*CF$_2$- (*Z*) and -C*H*=CHCF$_2$- (*Z*)), 2.50 – 2.40 (m, 1H), 2.22 – 2.04 (m, 3H), 1.97 – 1.79 (m, 2H), 1.51 – 1.41 (m, 1H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 147.19 (t, *J* = 8.7 Hz, -*C*=CHCF$_2$-), 127.04, 125.02, 115.39 (t, *J* = 22.9 Hz, -C=*C*HCF$_2$-), 36.21, 30.07, 27.47, 24.28.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.03 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -109.61 (br.s, =CHC*F*$_2$- (*Z*)), -111.44 (q, *J* = 12.0 Hz, =CHC*F*$_2$- (*E*)), -121.52 (br.s, -C*F*$_2$-, 2F), -121.97 (br.s, -C*F*$_2$-, 8F), -122.88 (br.s, -C*F*$_2$-, 2F), -123.70 (br.s, =CHCF$_2$C*F*$_2$-, 2F), -126.31 (br.s, -C*F*$_2$-, 2F).

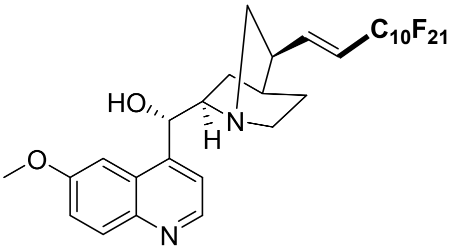


2q,

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.26 (tt, *J* = 3.9, 1.7 Hz, 1H), 2.15 – 2.02 (m, 4H), 1.66 – 1.53 (m, 4H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.80 (t, *J* = 9.9 Hz, 3F), -113.42 (s, alkenyl- C*F*$_2$, 2F), -121.92 (12F), -122.73 (br.s, 2F), -126.14 (br.s, 2F).

HRMS (EI): [M-H]+ calcd for $C_{16}H_8F_{21}$, 599.02852; found, 599.02776.

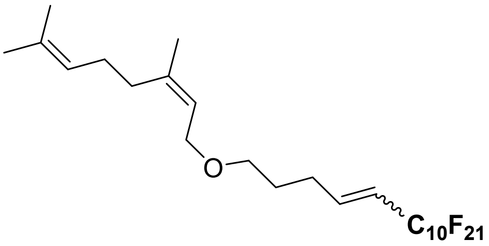


2r, , light brown sticky oil, mixture of *E* and *Z* isomers (81:19).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 8.73 (d, *J* = 4.5 Hz, -Ar*H*, 1H), 8.01 (d, *J* = 9.2 Hz, -Ar*H*, 1H), 7.51 – 7.32 (m, -Ar*H*, 2H), 7.25 (s, -Ar*H*, 1H), 6.73 – 6.51 (m, =C*H*CF$_2$- (*E* and *Z*), 1H), 6.31 (br.s, -O*H*, 1H), 6.13 – 5.56 (m, -C*H*=CHCF$_2$- (*E* and *Z*), 1H), 3.93 (s, -OC*H*$_3$, 3H), 3.45 (s, 1H), 3.33 (m, 1H), 3.06 (m, 2H), 2.82 (m, 2H), 2.57 – 2.12 (m, 2H), 2.04 – 1.83 (m, 1H), 1.58 (d, *J* = 9.3 Hz, 3H).

^{13}C NMR (75 MHz, CDCl$_3$): (only the signals of *E* isomer are shown) δ = 158.28, 147.31, 144.67, 141.73, 132.01, 126.73, 122.04, 100.41, 60.16, 55.51, 50.44, 49.92, 49.81, 48.92, 38.46, 27.36, 25.91.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.95 (t, *J* = 10.0 Hz, -C*F*$_3$, 3F), -106.21 (q, *J* = 11.1, 10.2 Hz, =CHC*F*$_2$-, (*Z*)), -111.37 (q, *J* = 11.5, 10.9 Hz, =CHC*F*$_2$-, (*E*)), -121.45 (br.s, -C*F*$_2$-, 2F), -121.93 (br.s, -C*F*$_2$-, 8F), -122.82 (br.s, -C*F*$_2$-, 2F), -123.48 (br.s, =CHCF$_2$C*F*$_2$-, (*E*)), -123.75 (br.s, =CHCF$_2$C*F*$_2$-, (*Z*)), -126.25 (br.s, -C*F*$_2$-, 2F).

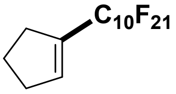


2s, , colorless oil, mixture of *E* and *Z* isomers (83:17).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.58 – 6.01 (m, 1H), 5.93 – 5.48 (m, 1H), 5.34 (m, 1H), 5.17 – 4.99 (m, 1H), 3.94 (d, *J* = 7.5 Hz, 2H), 3.42 (t, *J* = 6.2 Hz, 2H), 2.46 – 2.20 (m, 2H), 2.08 (m, 2H), 1.75 (s, 3H), 1.67 (s, 3H), 1.60 (s, 3H), 1.42 – 0.99 (m, 4H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.87 (t, *J* = 10.0 Hz, 3F), -106.77 (q, *J* = 13.3 Hz, alkenyl- C*F*$_2$ (*Z*), 0.3F), -111.34 (q, *J* = 11.5, 10.7 Hz, alkenyl- C*F*$_2$ (*E*), 1.7F), -121.50 – -123.53 (14F), -126.20 (br.s, 2F).

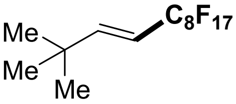
HRMS (EI): [M]+ calcd for $C_{25}H_{25}O_1F_{21}$, 740.15646; found, 740.15590.


2t,

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.93 (t, *J* = 9.9 Hz, 3F), -110.27 (br.s, alkenyl- C*F*$_2$-, 2F), -120.93 (br.s, -C*F*$_2$-, 2F), -121.87 (br.s, -C*F*$_2$-, 8F), -122.35 (br.s, -C*F*$_2$-, 2F), -122.82 (br.s, =CHCF$_2$C*F*$_2$-, 2F), -126.25 (br.s, -C*F*$_2$-, 2F).

6.3 Characterization data of other perfluoroalkylated alkenes

Notes: For the ^{13}C NMR (F coupled), peaks for the C of perfluoroalkyl chain and C adjacent to perfluoroalkyl chain (in most cases) are too broad to be assigned (not shown in the following data).

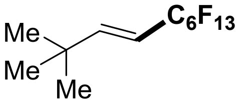

4a,

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.40 (dt, *J* = 16.0, 2.2 Hz, =C*H*CF$_2$-, 1H), 5.49 (dt, *J* = 16.0, 11.9 Hz, -C*H*=CHCF$_2$-, 1H), 1.08 (s, -C*H*$_3$, 9H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 152.93 (t, *J* = 8.3 Hz, -*C*=CHCF$_2$-), 112.47 (t, *J* = 23.0 Hz, -C=*C*HCF$_2$-), 33.46, 28.34.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.18 (t, *J* = 10.1 Hz, -C*F*$_3$, 3F), -111.41 (q, *J* = 12.2 Hz, =CHC*F*$_2$-, 2F), -121.61 (br.s, -C*F*$_2$-, 2F), -122.18 (m, -C*F*$_2$-, 4F), -122.99 (br.s, -C*F*$_2$-, 2F), -123.89 (br.s, =CHCF$_2$C*F*$_2$-, 2F), 126.42 (m, -C*F*$_2$-, 2F).

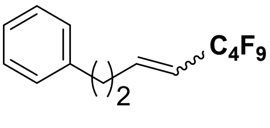
HRMS (EI) [M]+ calcd for $C_{14}H_{11}F_{17}$, 502.05838; found, 502.05844.


4b,

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.42 (dt, *J* = 16.0, 2.2 Hz, =C*H*CF$_2$-, 1H), 5.51 (dt, *J* = 16.0, 11.9 Hz, -C*H*=CHCF$_2$-, 1H), 1.11 (s, -C*H*$_3$, 9H).

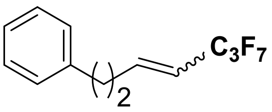
^{19}F NMR (282 MHz, CDCl$_3$): δ = -81.05 (tt, *J* = 10.1, 2.5 Hz, -C*F*$_3$, 3F), -111.29 (q, *J* = 12.3 Hz, =CHC*F*$_2$-, 2F), -121.75 (m, -C*F*$_2$-, 2F), -123.05 (m, -C*F*$_2$-, 2F), -123.82 (m, -C*F*$_2$-, 2F), -126.34 (m, -C*F*$_2$-, 2F).

MS (EI, 70 eV): m/z (%) = 402.1 (2) [M]+, 401.1 (13) [M-H]+, 387.1 (2), 295.0 (1), 131.0 (1), 83.1 (5), 69.1 (10), 57.0 (100).


4c, mixture of *E* and *Z* isomers (81:19).

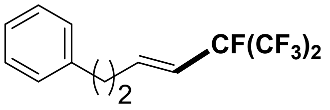
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46 – 7.29 (m, Ar-*H*, 2H), 7.28 – 7.14 (m, Ar-*H*, 3H), 6.47 (dtt, J = 15.9, 6.8, 2.3 Hz, $=C\mathit{H}CF_2$- (*E*)), 6.17 (dtt, J = 12.1, 7.3, 2.4 Hz, $=C\mathit{H}CF_2$- (*Z*)), 5.82 – 5.38 (m, $-C\mathit{H}=CHCF_2$- (*E* and *Z*), 1H), 2.79 (m, $PhC\mathit{H_2}$-, 2H), 2.74 – 2.62 (m, $-C\mathit{H_2}CH=$ (*Z*)), 2.61 – 2.49 (m, $-C\mathit{H_2}CH=$ (*E*)).

^{19}F NMR (282 MHz, $CDCl_3$): δ = -79.93 – -83.15 (m, $-C\mathit{F_3}$, 3F), -105.71 – -108.92 (m, $=CHC\mathit{F_2}$- (*Z*)), -111.55 – -111.68 (m, $=CHC\mathit{F_2}$- (*E*)), -124.38 – -124.54 (m, $=CHCF_2C\mathit{F_2}$- (*E*)), -124.64 – -124.74 (m, $=CHCF_2C\mathit{F_2}$- (*Z*)), -125.75 – -125.89 (m, $-C\mathit{F_2}$-, 2F).

4d,  mixture of *E* and *Z* isomers (80:20).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.45 – 7.31 (m, Ar-*H*, 2H), 7.31 – 7.18 (m, Ar-*H*, 3H), 6.50 (dtt, J = 15.9, 6.8, 2.3 Hz, $=C\mathit{H}CF_2$- (*E*)), 6.20 (dtt, J = 12.0, 7.3, 2.3 Hz, $=C\mathit{H}CF_2$- (*Z*)), 5.81 – 5.45 (m, $-C\mathit{H}=CHCF_2$- (*E* and *Z*), 1H), 2.81 (q, J = 6.9 Hz, $PhC\mathit{H_2}$-, 2H), 2.76 – 2.65 (m, $-C\mathit{H_2}CH=$ (*Z*)), 2.65 – 2.49 (m, $-C\mathit{H_2}CH=$ (*E*)).

^{19}F NMR (282 MHz, $CDCl_3$): δ = -80.43 (q, J = 8.7 Hz, $-C\mathit{F_3}$, 3F), -107.76 – -108.08 (m, $=CHC\mathit{F_2}$- (*Z*)), -112.20 – -112.78 (m, $=CHC\mathit{F_2}$- (*E*)), -127.93 (s, $=CHCF_2C\mathit{F_2}$- (*E*)), -128.18 (s, $=CHCF_2C\mathit{F_2}$- (*Z*)).

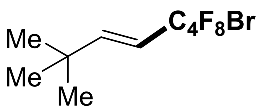
4e, 

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.50 – 7.29 (m, Ar-*H*, 2H), 7.29 – 7.13 (m, Ar-*H*, 3H), 6.40 (dt, J = 15.7, 6.9 Hz, $=C\mathit{H}CF_2$-, 1H), 5.73 – 5.34 (m, $-C\mathit{H}=CHCF_2$-, 1H), 2.81 (t, J = 7.6 Hz, $PhC\mathit{H_2}$-, 2H), 2.64 – 2.40 (m, $-C\mathit{H_2}CH=$, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 140.47 and 140.33 (d, J = 10.3 Hz, $-CH_2\mathit{C}H=$), 140.40, 128.48, 128.40, 126.23, 114.73 (d, *J* = 16.9 Hz, $=\mathit{C}HCF_2$-), 34.58, 34.05.

^{13}C NMR Dept135 (75 MHz, $CDCl_3$): δ = (+): 140.41 (d, J = 10.3 Hz, $-CH_2\mathit{C}H=$), 128.49, 128.41, 126.24, 114.74 (d, J = 17.0 Hz, $=\mathit{C}HCF_2$-). (–): 34.59, 34.06.

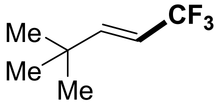
^{19}F NMR (282 MHz, $CDCl_3$): δ = -77.51 (d, J = 7.7 Hz, $-CF(C\mathit{F_3})_2$, 6F), -182.89 – -189.09 (m, $-C\mathit{F}(CF_3)_2$, 1F).

4f, 

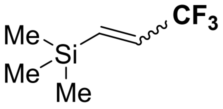
^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.31 (dt, *J* = 16.0, 2.2 Hz, $=C\mathit{H}CF_2$-, 1H), 5.41 (dt, *J* = 16.0, 11.9 Hz, $-C\mathit{H}=CHCF_2$-, 1H), 1.01 (s, $-C\mathit{H_3}$, 9H).

^{19}F NMR (282 MHz, $CDCl_3$): δ = -63.02 – -63.30 (m, $-C\mathit{F_2}Br$, 2F), -111.31 (q, J = 12.2 Hz, $=CHC\mathit{F_2}$-, 2F), -116.26 – -117.04 (m, $-C\mathit{F_2}$-, 2F), -122.44 – -123.19 (m, $-C\mathit{F_2}$-, 2F).

6.4 Characterization data of trifluoromethylated alkenes

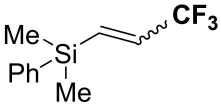
4g, 

^{19}F NMR (282 MHz, $CDCl_3$): δ = -67.30 (t, J = 11.0 Hz, $-C\mathit{F_3}$, 3F).



4h, mixture of *E* and *Z* isomers (84:16).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -62.35 (s, -C*F$_3$* (*Z*)), -67.43 (s, -C*F$_3$* (*E*)).



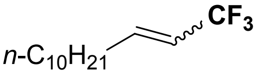
4i, mixture of *E* and *Z* isomers (84:16).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 7.42 – 7.31 (m, Ar-*H*, 2H), 7.29 – 7.14 (m, Ar-*H*, 3H), 6.59 (dq, J = 18.9, 2.0 Hz, =C*H*CF$_2$- (*E*)), 6.35 – 6.06 (m, =C*H*CF$_2$- (*Z*)), 5.97 – 5.79 (m, -C*H*=CHCF$_2$- (*E*)), 5.68 – 5.52 (m, -C*H*=CHCF$_2$- (*Z*)), 0.27 (s, -C*H$_3$*, 6H).

^{13}C NMR (101 MHz, CDCl$_3$): δ = 143.32 (q, *J* = 5.7 Hz, -*C*H=CHCF$_2$- (*Z*)), 140.76 (q, *J* = 4.8 Hz, -*C*H=CHCF$_2$- (*E*)), 137.77 (*C* of -Ph, 1C), 135.77 (*C* of -Ph, 2C), 134.43 (q, J = 34.0 Hz, =*C*HCF$_2$-, 1C), 131.67 (*C* of -Ph, 1C), 130.06 (*C* of -Ph, 2C), 124.13 (q, J = 271.6 Hz, -*C*F$_3$, 1C), -1.36 (-*C*H$_3$, 2C).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -61.37 – -61.42 (m, -C*F$_3$* (*Z*)), -66.49 (dd, J = 5.9, 2.0 Hz, -C*F$_3$* (*E*)).

MS (EI, 70 eV): m/z (%) = 200.1 (21) [M]+, 91.1 (100).



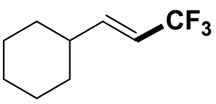
4j, mixture of *E* and *Z* isomers (87:13).

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.54 – 6.28 (m, =C*H*CF$_2$- (*E*)), 5.98 (dt, J = 11.6, 7.9 Hz, =C*H*CF$_2$- (*Z*)), 5.74 – 5.45 (m, -C*H*=CHCF$_2$-, 1H), 2.43 – 2.21 (m, -C*H$_2$*CH= (*Z*)), 2.22 – 2.06 (m, -C*H$_2$*CH= (*E*)), 1.52 – 1.36 (m, -C*H$_2$*-, 2H), 1.27 (br.s, -C*H$_2$*-, 14H), 0.89 (t, *J* = 6.7 Hz, -C*H$_3$*, 3H).

^{13}C NMR (101 MHz, CDCl$_3$): δ = 143.20 (q, J = 5.5 Hz, -*C*H=CHCF$_2$- (*Z*)), 140.81 (q, J = 6.5 Hz, -*C*H=CHCF$_2$- (*E*)), 123.13 (q, J = 269.0 Hz, -*C*F$_3$), 118.25 (q, J = 33.1 Hz, =*C*HCF$_2$- (*E*)), 118.20 (q, J = 33.2 Hz, =*C*HCF$_2$- (*Z*)), 31.90, 31.45, 29.58, 29.52, 29.36, 29.32, 29.03, 27.96, 22.68, 14.09.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -58.09 (dt, *J* = 8.7, 2.3 Hz, -C*F$_3$* (*Z*)), -63.86 – -64.00 (m, -C*F$_3$* (*E*)).

MS (EI, 70 eV): m/z (%) = 236.3 (1) [M]+, 208.2 (9), 193.1 (13), 179.1 (15), 165.1 (20), 151.1 (30), 137.1 (18), 123.1 (11), 111.1 (27), 97.1 (58), 83.1 (56), 69.1 (89), 57.1 (100).

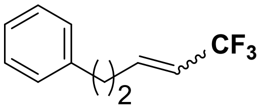


4k,

^{1}H NMR (300 MHz, CDCl$_3$): δ = 6.37 – 6.11 (m, =C*H*CF$_2$-, 1H), 5.64 – 5.31 (m, -C*H*=CHCF$_2$-, 1H), 2.11 – 1.91 (m, -C*H*(CH$_2$-)$_2$, 1H), 1.77 – 1.53 (m, -C*H$_2$*-, 4H), 1.34 – 0.92 (m, -C*H$_2$*-, 6H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -63.78 (dt, *J* = 6.6, 2.1 Hz, -C*F$_3$*, 3F).

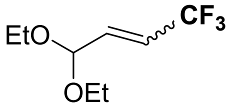
MS (EI, 70 eV): m/z (%) = 178.1 (2) [M]+, 163.1 (4), 136.1 (6), 115.1 (8), 95.1 (7), 82.1 (100), 67.1 (72), 54.1 (14).



4l, mixture of *E* and *Z* isomers (84:16).

^{19}F NMR (282 MHz, $CDCl_3$): δ = -59.08 (s, -CF_3 (*Z*)), -65.00 (s, -CF_3 (*E*)).

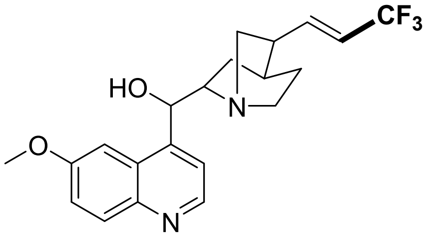
MS (EI, 70 eV): m/z (%) = 229.9 (<1) [M]+, 215.1 (100) [M-Me]+, 153.1 (8), 133.1 (67), 121.1 (23), 105.1 (11), 91.1 (7), 77.1 (31).



4m,

^{19}F NMR (282 MHz, $CDCl_3$): δ = -64.01 – -64.43 (m, -CF_3 (*Z*)), -65.18 – -65.80 (m, -CF_3 (*E*)).

MS (EI, 70 eV): m/z (%) = 198.0 (<1) [M]+, 197.1 (1) [M-H]+, 153.1 (77) [M-EtO]+, 125.1 (100), 103.1 (6), 77.0 (6).



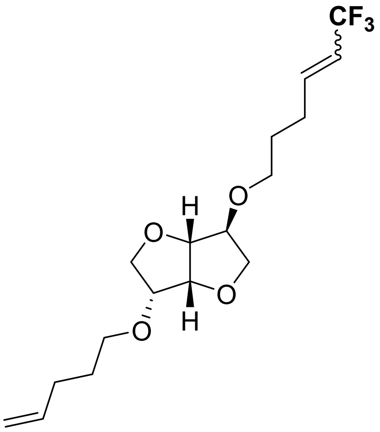
4n,

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.67 (d, *J* = 4.5 Hz, 1H), 7.96 (d, *J* = 9.2 Hz, 1H), 7.38 (d, *J* = 4.5 Hz, 1H), 7.32 (dd, *J* = 9.3, 2.7 Hz, 1H), 7.15 (s, 1H), 6.34 (dd, *J* = 11.6, 10.2 Hz, 1H), 6.22 (s, 1H), 5.76 – 5.53 (m, 1H), 3.87 (s, 3H), 3.38 – 3.12 (m, 1H), 3.07 – 2.58 (m, 5H), 2.31 – 2.10 (m, 1H), 1.86 – 1.76 (m, 2H), 1.58 – 1.43 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ = 158.22, 147.35, 144.70, 143.92 (q, *J* = 4.1 Hz, -*C*H=CHCF$_2$-), 141.86, 132.08, 126.70, 123.13 (q, *J* = 271.9 Hz, -*C*F$_3$), 121.96, 119.01 (q, *J* = 33.3 Hz, =*C*HCF$_2$-), 100.57, 60.20, 55.63, 49.88, 49.80, 34.80, 27.64, 25.83, 21.62.

^{19}F NMR (282 MHz, $CDCl_3$): δ = -57.79 (dd, *J* = 8.6, 1.8 Hz, -CF_3).

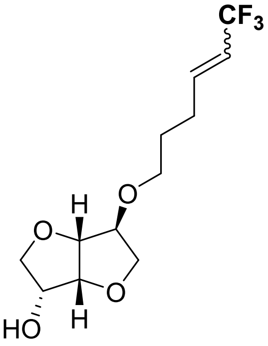
MS (EI, 70 eV): m/z (%) = 391.3 (<1) [M-H]+, 375.2 (100) [M-OH]+, 347.1 (4), 253.1 (7), 204.1 (11), 184.0 (8), 154.1 (4), 136.0 (3), 77.0 (1).



4o,

^{19}F NMR (282 MHz, $CDCl_3$): δ = -58.42 (-CF_3 (*Z*)), -64.33 (-CF_3 (*E*)).

MS (EI, 70 eV): m/z (%) = 350.2 (5) [M]+, 264.2 (8), 136.1 (13), 117.1 (26), 97.1 (13), 84.1 (46), 69.1 (100).



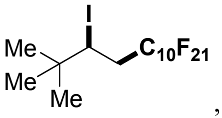
4p,

^{19}F NMR (282 MHz, CDCl$_3$): δ = -58.43 (-CF_3 (*Z*)), -64.35 (-CF_3 (*E*)).

MS (EI, 70 eV): m/z (%) = 282.2 (23) [M]+, 136.1 (37), 117.1 (44), 97.1 (24), 86.1 (36), 85.1 (22), 84.1 (51), 77.1 (21), 69.1 (100), 68.1 (31), 58.1 (20).

6.5 Characterization data of others

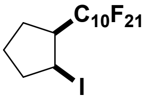
Notes: For the ^{13}C NMR (F coupled), peaks for the C of perfluoroalkyl chain and C adjacent to perfluoroalkyl chain (in most cases) are too broad to be assigned (not shown in the following data).



8a, , White solid.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 4.18 (dd, *J* = 8.5, 2.0 Hz, -CHI-, 1H), 3.05 – 2.66 (m, -CH_2CF$_2$-, 2H), 1.11 (s, -Me, 9H).

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.80 (t, *J* = 10.0 Hz, -CF_3, 3F), -113.85 – -116.41 (m, -CH$_2$CF_2-, 2F), -121.81 (br.s, -CF_2-, 10F), -122.73 (br.s, -CF_2-, 2F), -123.66 (br.s, -CF_2-, 2F), -126.14 (br.s, -CF_2-, 2F).



8b, , White solid.

^{1}H NMR (300 MHz, CDCl$_3$): δ = 4.50 (dt, *J* = 6.8, 4.5 Hz, , -CHI-, 1H), 3.40 – 2.92 (m, -CH_2CF$_2$-, 1H), 2.36 – 1.65 (m, -CH_2-, 6H).

^{13}C NMR (101 MHz, CDCl$_3$): δ = 53.68 (dd, *J* = 21.6, 19.7 Hz), 41.01, 26.35, 25.35, 18.65.

^{19}F NMR (282 MHz, CDCl$_3$): δ = -80.85 (t, *J* = 10.0 Hz, -CF_3, 3F), -114.05 – -120.85 (dd, *J* = 1643.1, 274.4 Hz, -CH$_2$CF_2-, 2F, the right line is overlaped with the signal of -120.88), -120.88 (br.s, -CF_2-, 2F), -121.78 (br.s, -CF_2-, 10F), -122.75 (br.s, -CF_2-, 2F), -126.17 (br.s, -CF_2-, 2F).

7. References

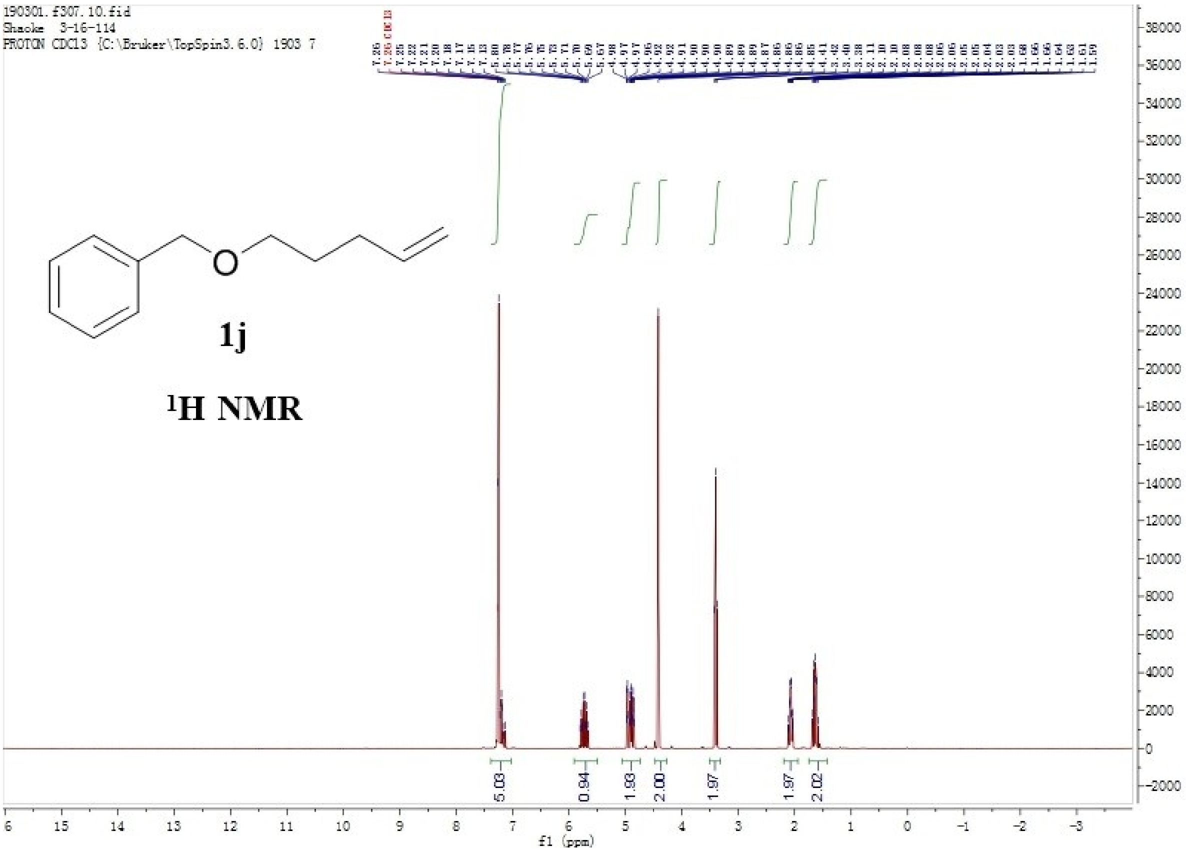
[1] E. A. Standley, S. J. Smith, P. Muller and T. F. Jamison, *Organometallics* **2014**, 33, 2012-2018.

[2] L. Legnani1, G. Prina-Cerai1, T. Delcaillau, S. Willems and B. Morandi, *Science* **2018**, 362, 434-439.

[3] J. Rabeah, J. Radnik, V. Briois, D. Maschmeyer, G. Stochniol, S. Peitz, H. Reeker, C. La Fontaine and A. Brückner, *ACS Catal.* **2016**, 6, 8224-8228.

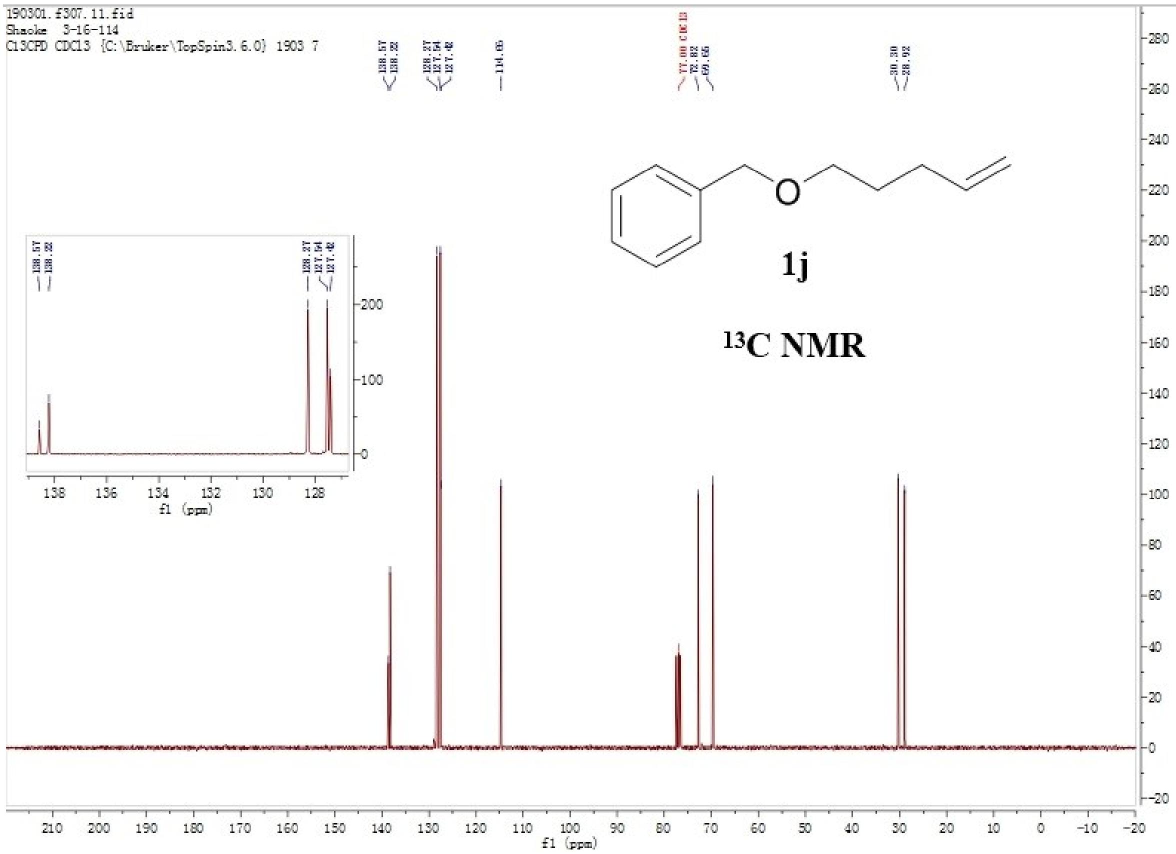
8. Spectra

8.1 Spectra of alkenes



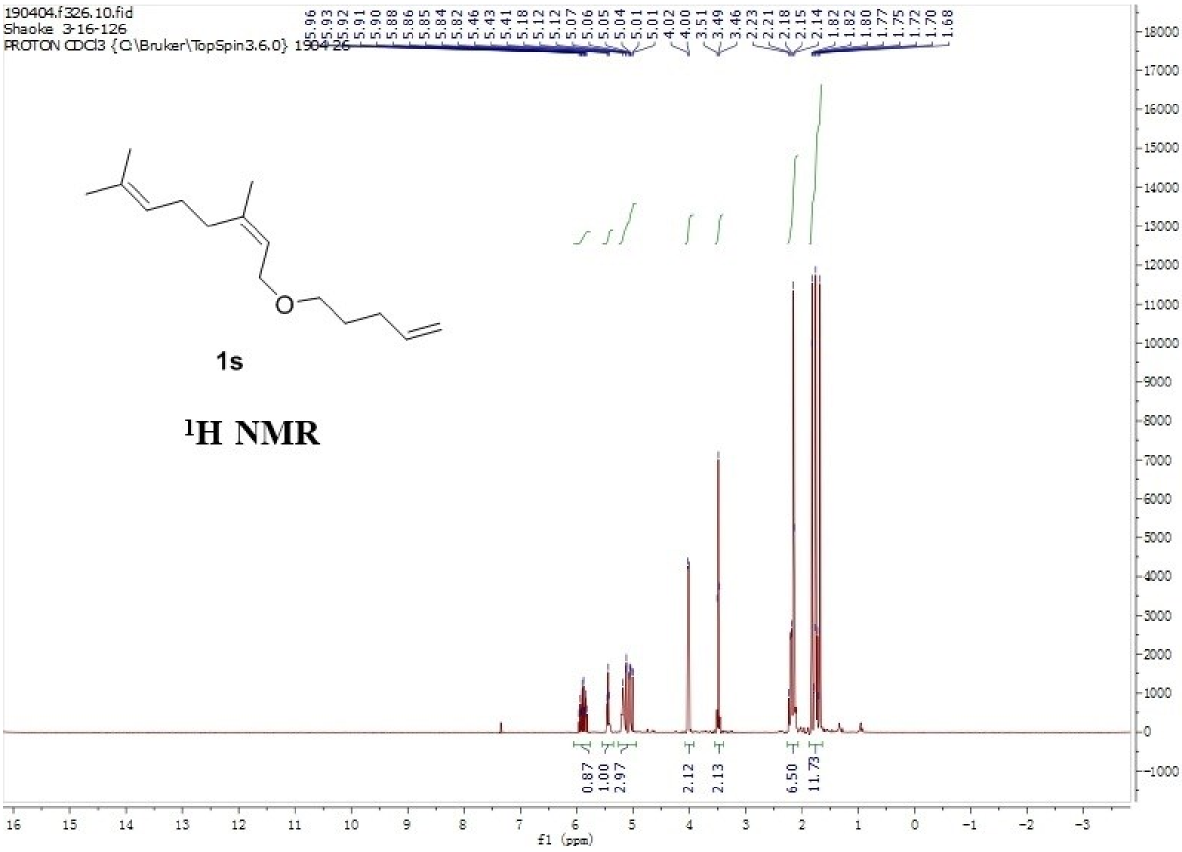
1j

^{1}H NMR



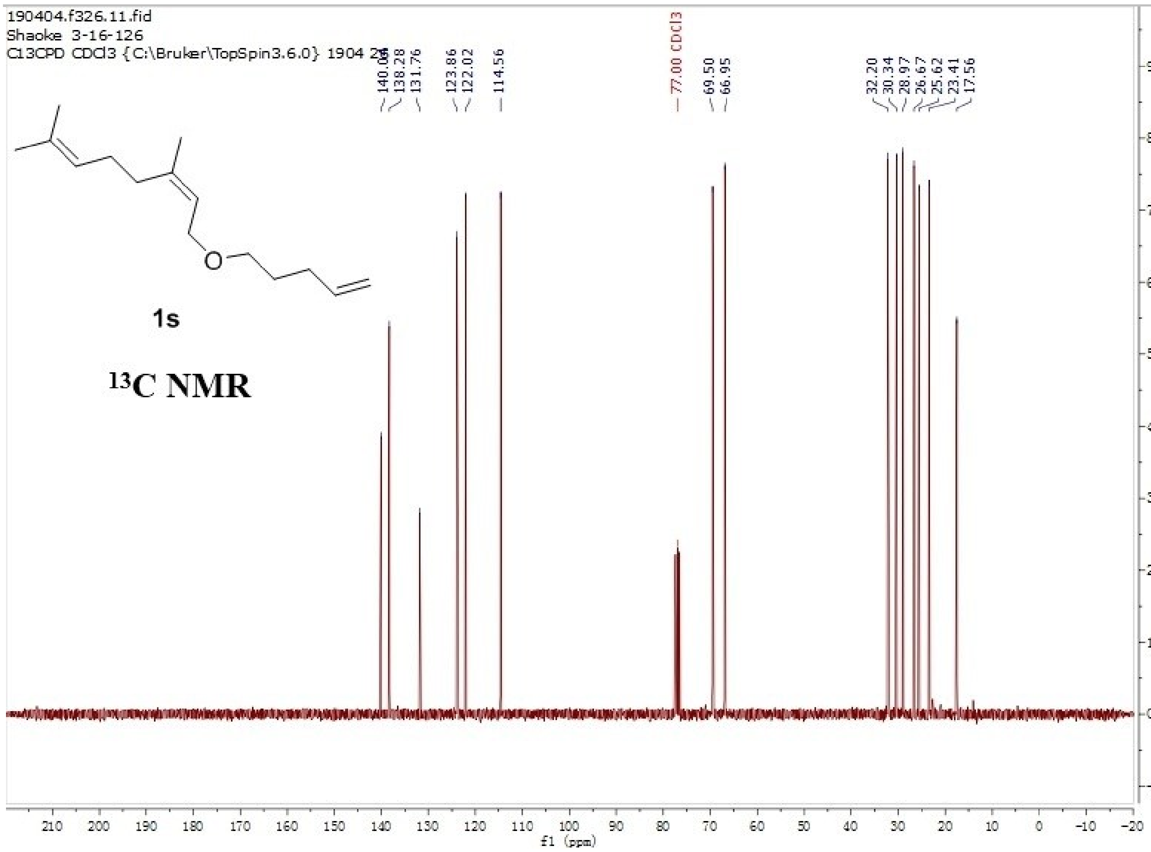
1j

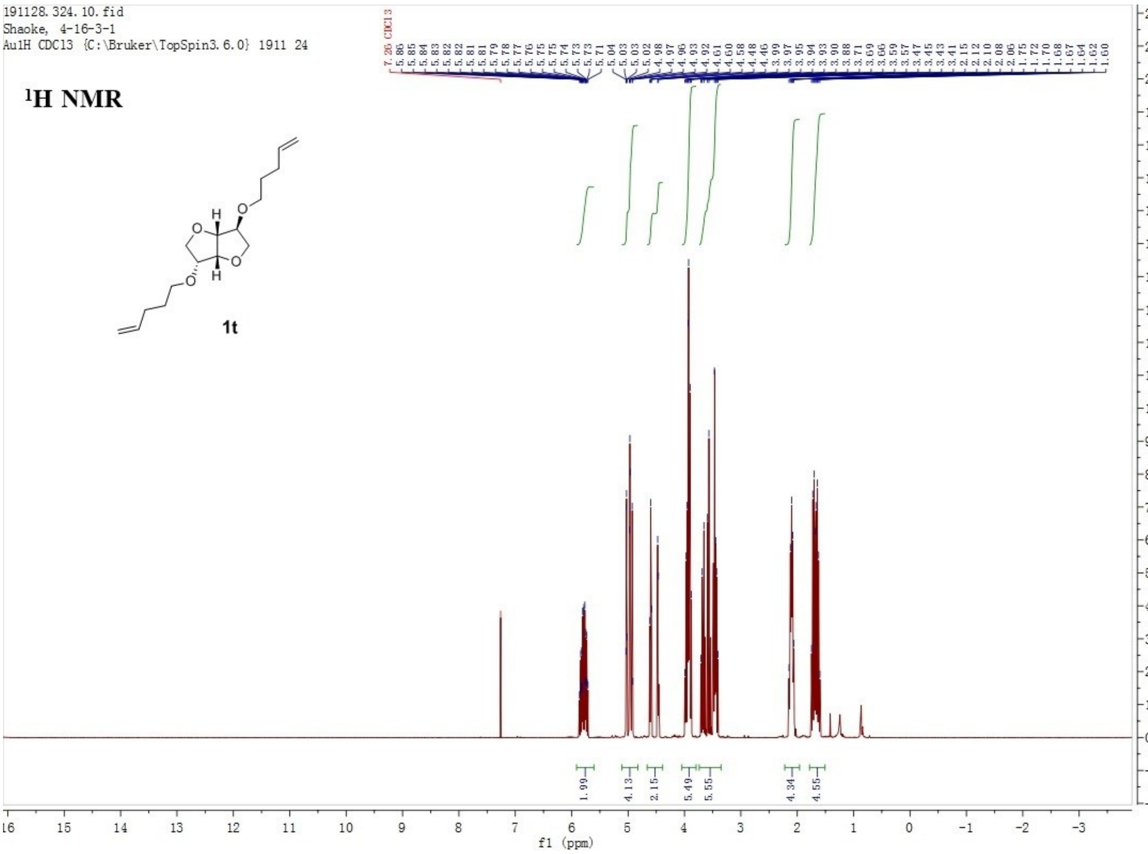
^{13}C NMR

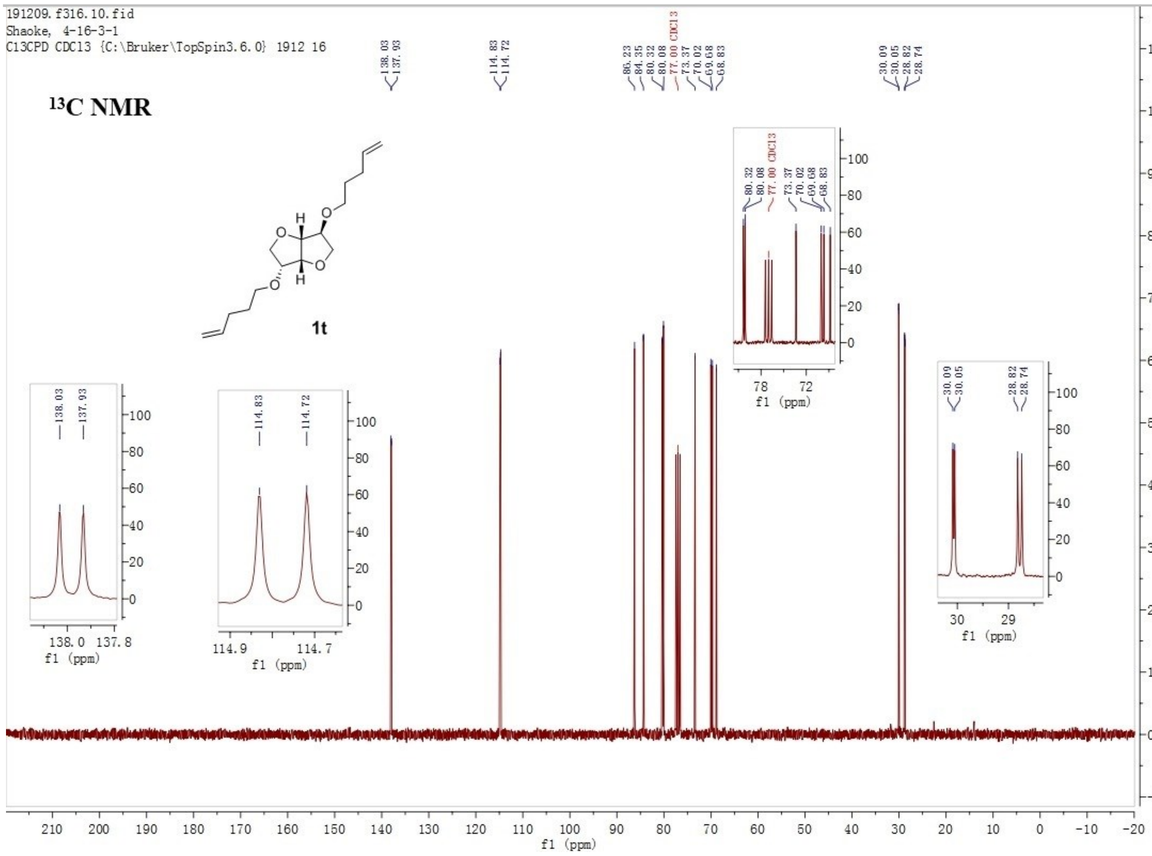


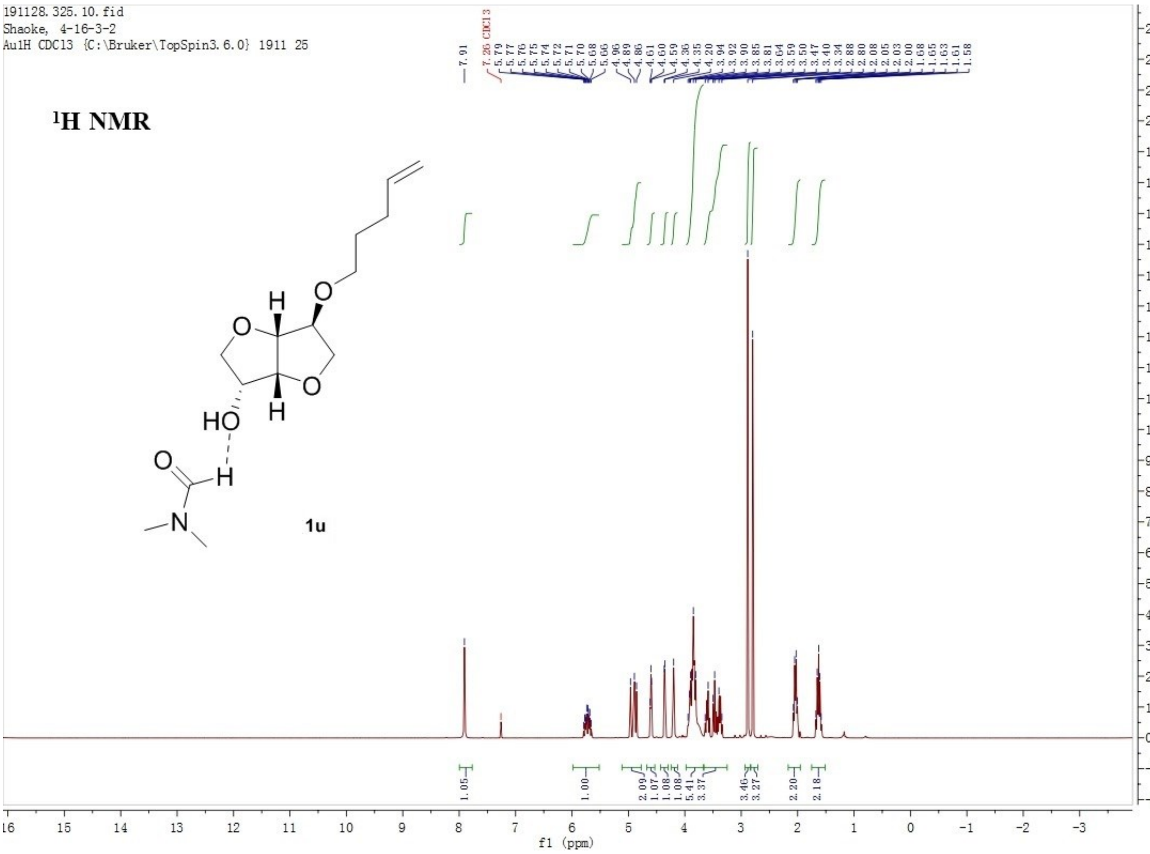
1s

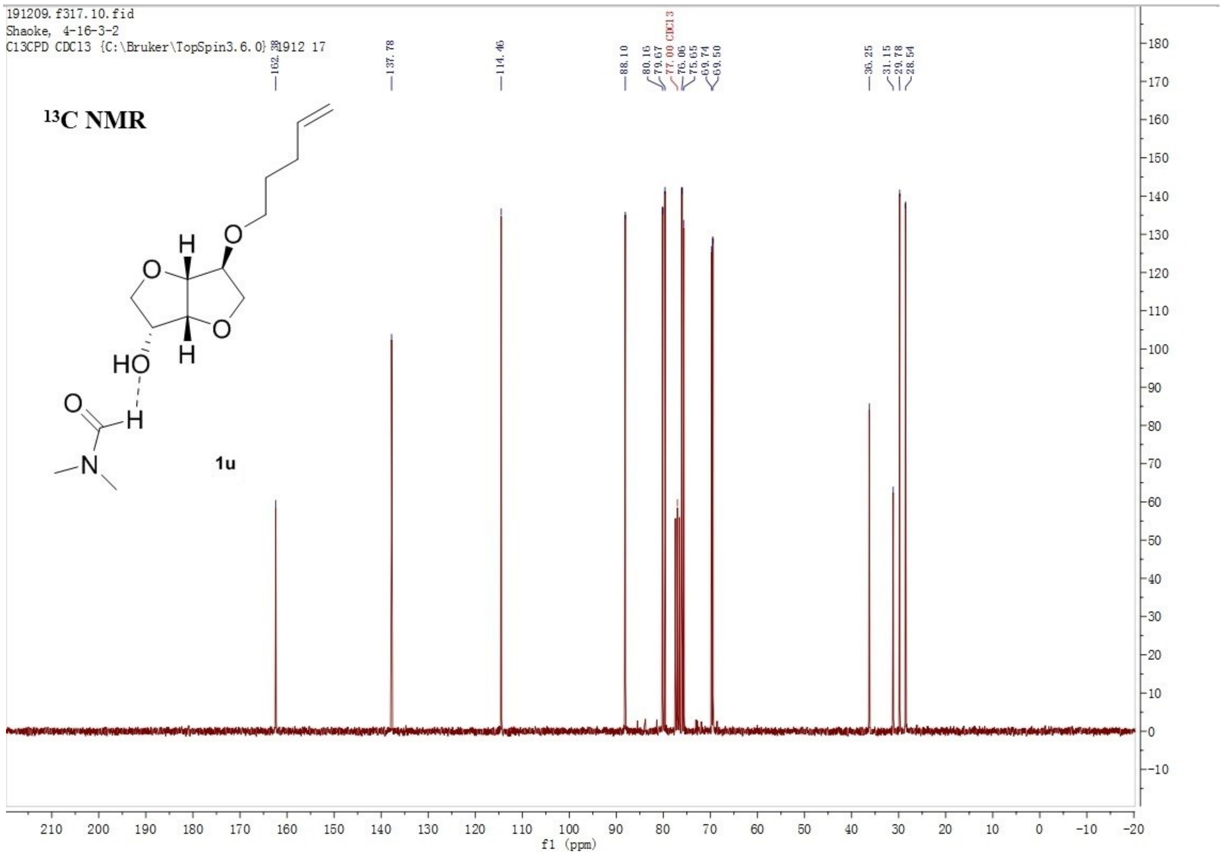
^{1}H NMR


190404.f326.11.fid
Shaoke 3-16-126
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1904 24
140.06
138.28
131.76
123.86
122.02
114.56
77.00 CDCl3
69.50
66.95
32.20
30.34
28.97
26.67
25.62
23.41
17.56
O
1s
13C NMR
90
80
70
60
50
40
30
20
10
0
-10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

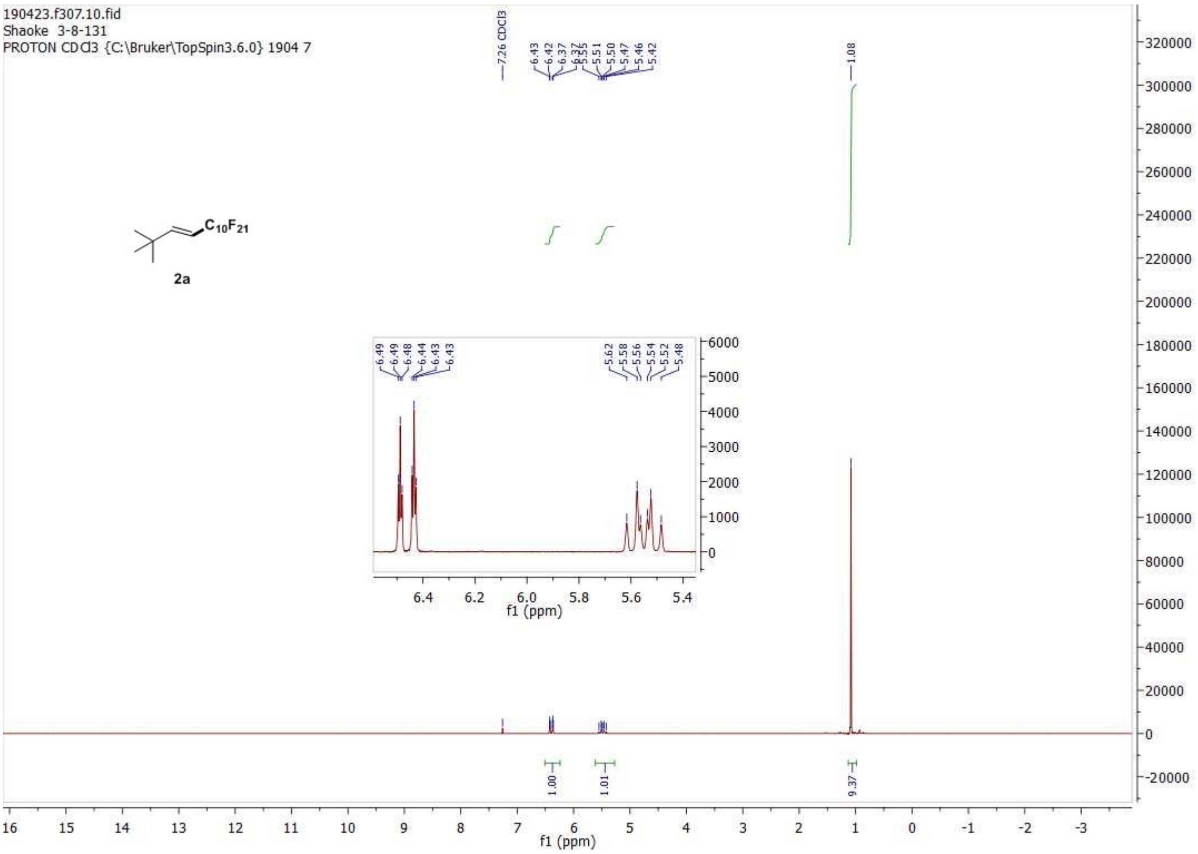

191128.324.10.fid
Shaoke, 4-16-3-1
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 24
7.26 CDCl3
5.86
5.85
5.84
5.83
5.82
5.82
5.81
5.81
5.79
5.78
5.77
5.76
5.75
5.75
5.74
5.73
5.73
5.71
5.04
5.03
5.03
5.02
4.98
4.97
4.96
4.93
4.92
4.61
4.60
4.58
4.48
4.46
3.99
3.97
3.95
3.94
3.93
3.90
3.88
3.71
3.69
3.66
3.59
3.57
3.47
3.45
3.43
3.41
2.15
2.12
2.10
2.08
2.06
1.75
1.72
1.70
1.68
1.67
1.64
1.62
1.60
1H NMR
O
H
O
O
H
O
1t
1.99
4.13
2.15
5.49
5.55
4.34
4.55
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

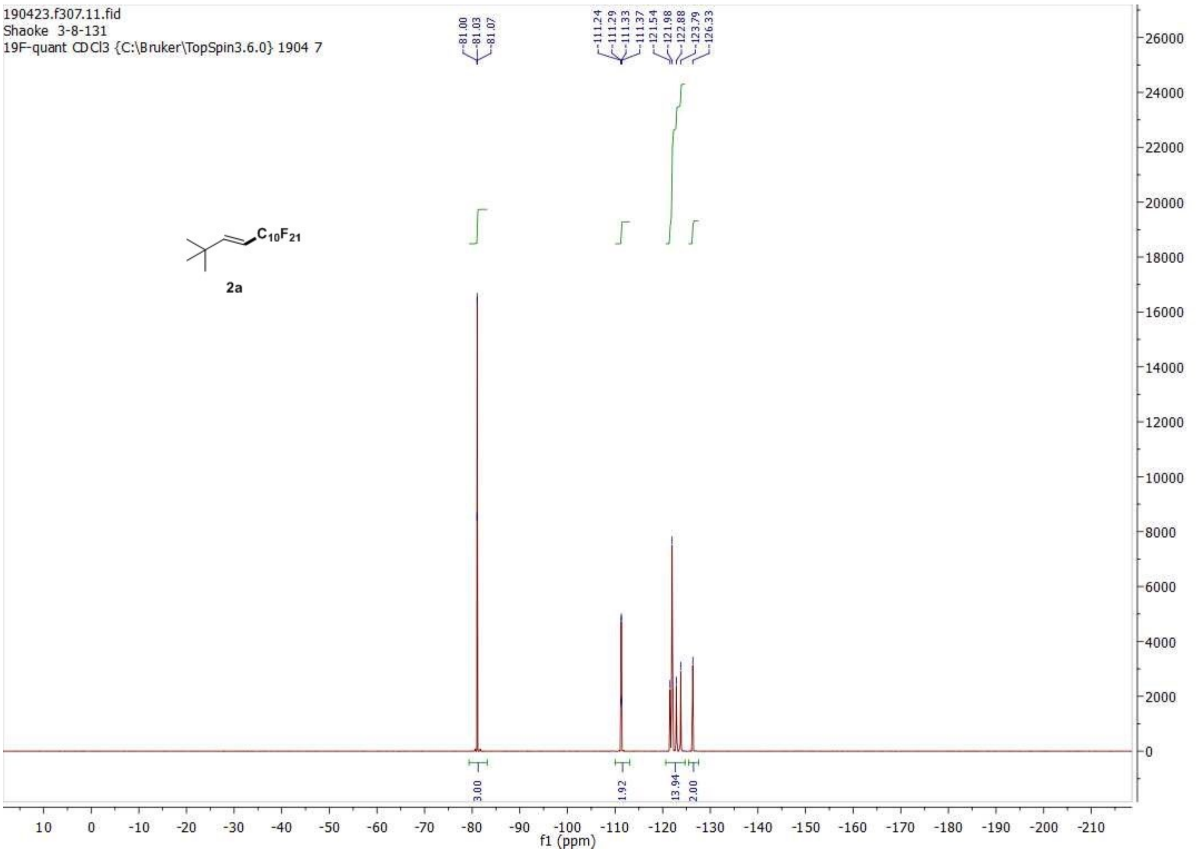

191209.f316.10.fid
Shaoke, 4-16-3-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1912 16
13C NMR
1t
138.03
137.93
114.83
114.72
86.23
84.35
80.32
80.08
77.00 CDCl3
73.37
70.02
69.68
68.83
30.09
30.05
28.82
28.74
f1 (ppm)

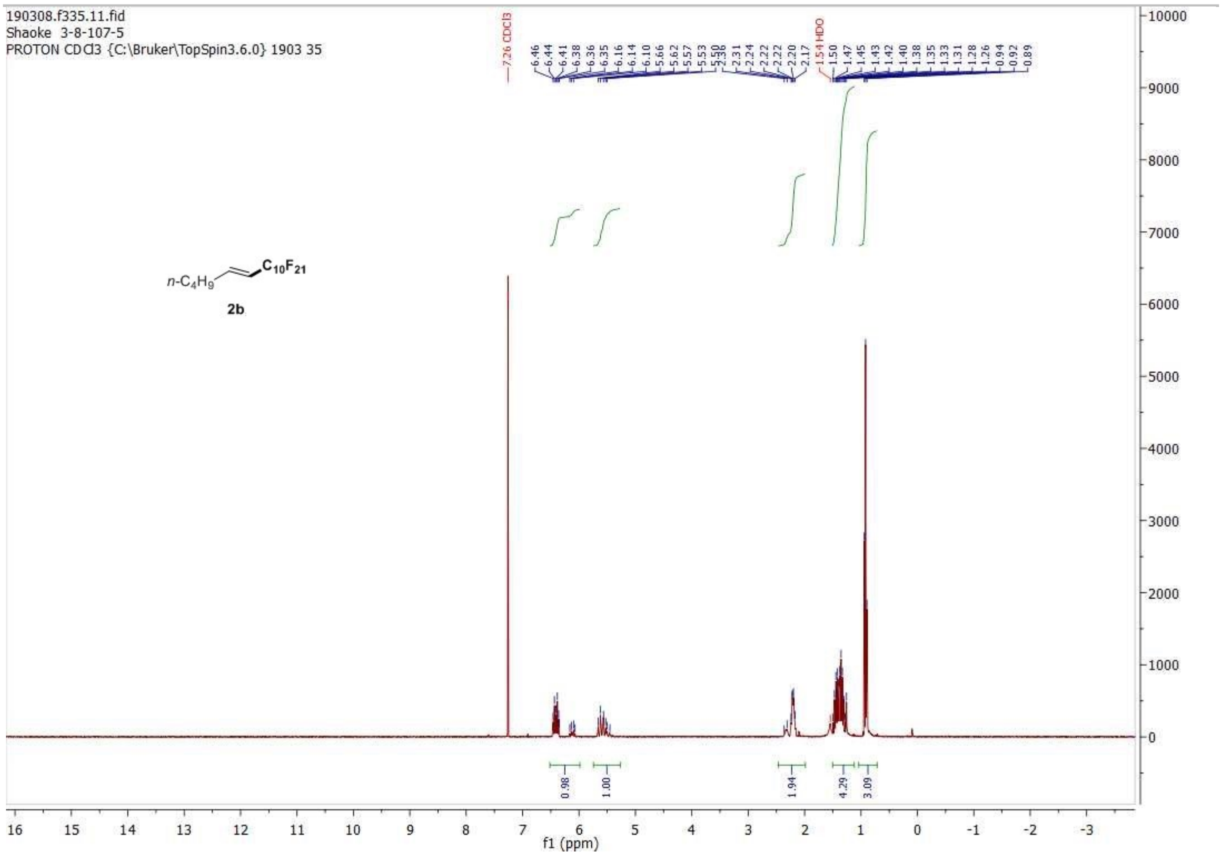

191128.325.10.fid
Shaoke, 4-16-3-2
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 25
1H NMR
1u
HO
O
H
N
7.91
7.26 CDCl3
5.79
5.77
5.76
5.75
5.74
5.72
5.71
5.70
5.68
5.66
4.96
4.89
4.86
4.61
4.60
4.59
4.36
4.35
4.20
3.94
3.92
3.90
3.85
3.81
3.64
3.59
3.50
3.47
3.40
3.34
2.88
2.80
2.08
2.05
2.03
2.00
1.68
1.65
1.63
1.61
1.58
1.05
1.00
2.09
1.07
1.08
1.08
5.41
3.37
3.46
3.27
2.20
2.18
f1 (ppm)

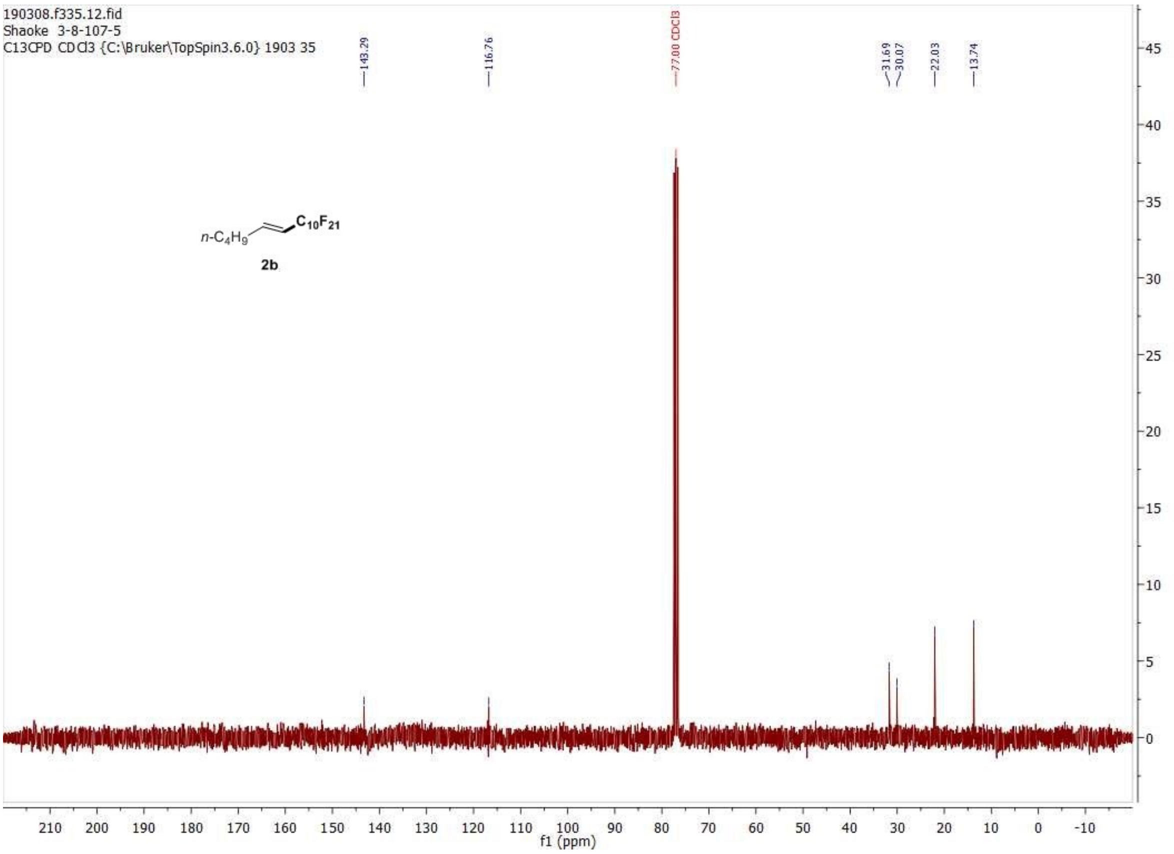

191209.f317.10.fid
Shaoke, 4-16-3-2
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1912 17
162.38
137.78
114.46
88.10
80.16
79.67
77.00 CDCl3
76.06
75.65
69.74
69.50
36.25
31.15
29.78
28.54
^{13}C NMR
O
H
O
H
O
HO
O
H
N
1u
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

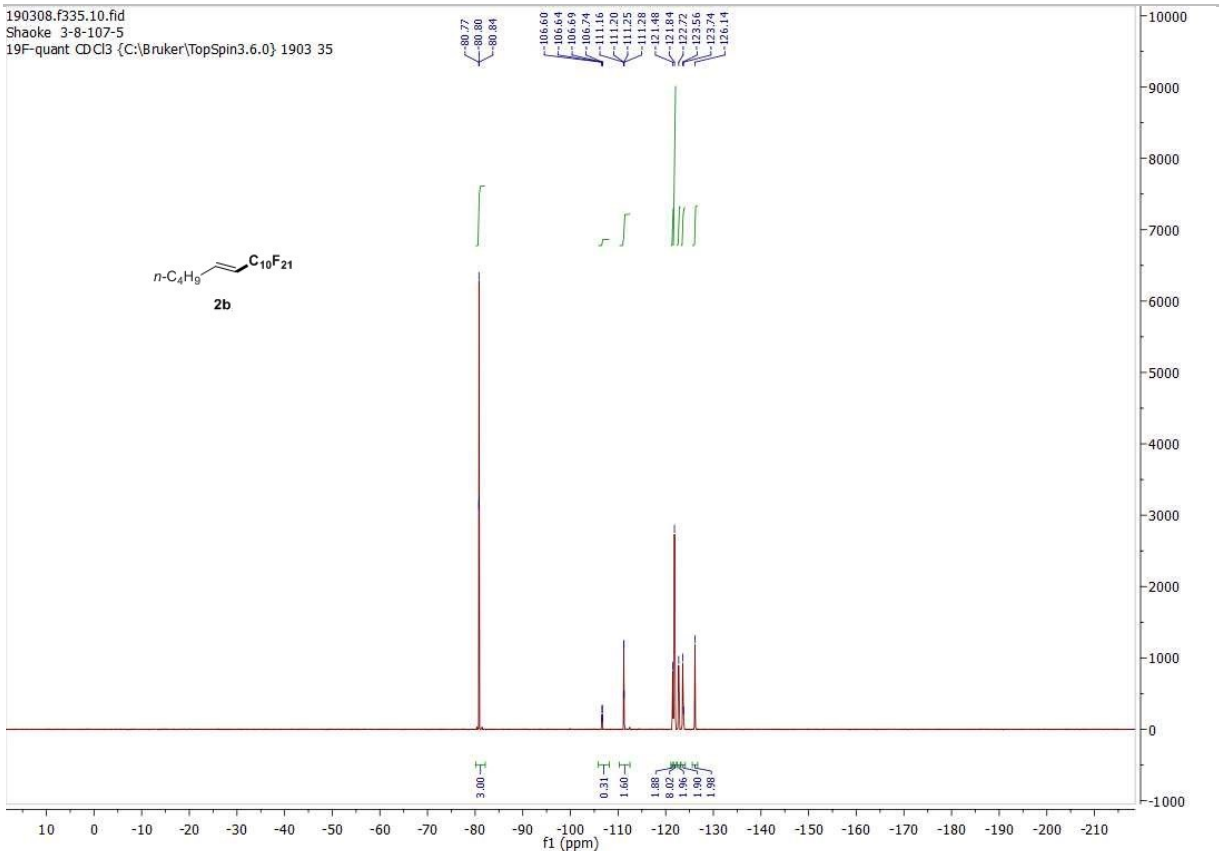
8.2 Spectra of perfluorodecylated alkenes

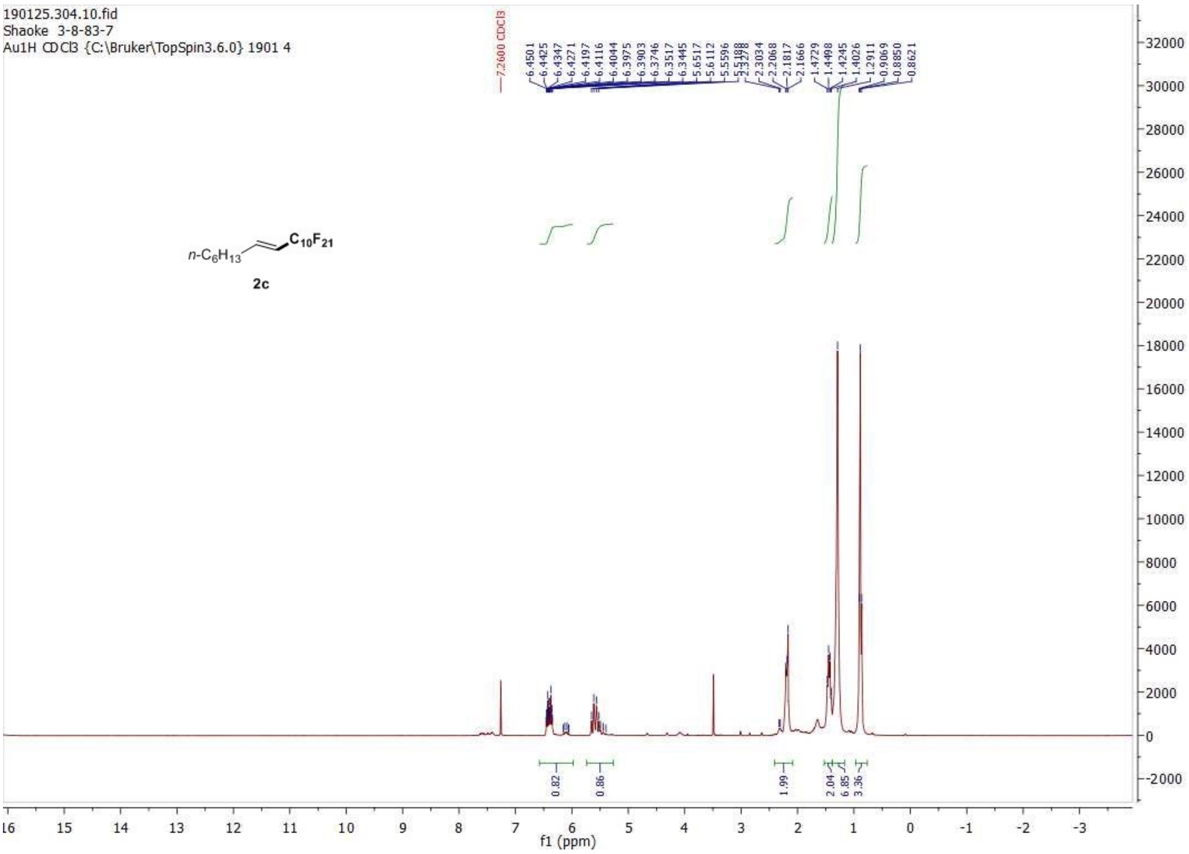


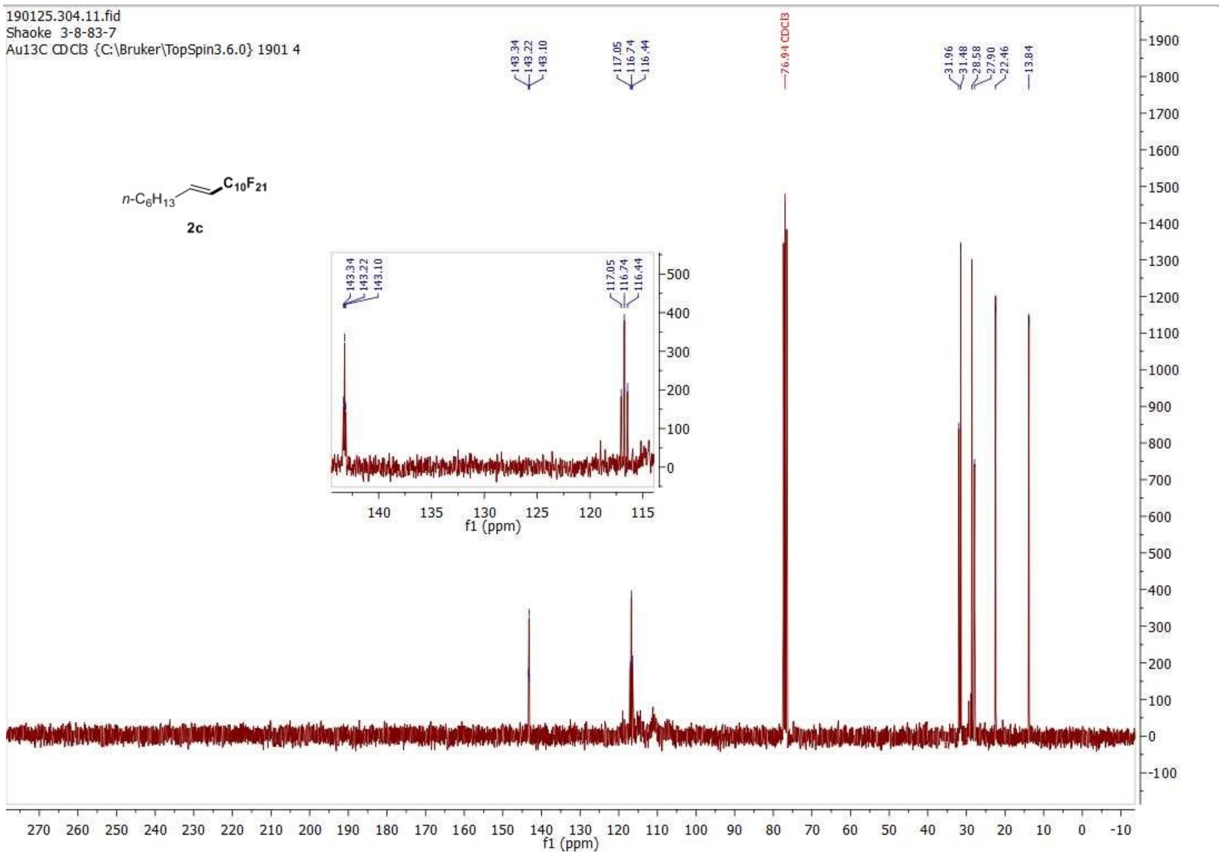


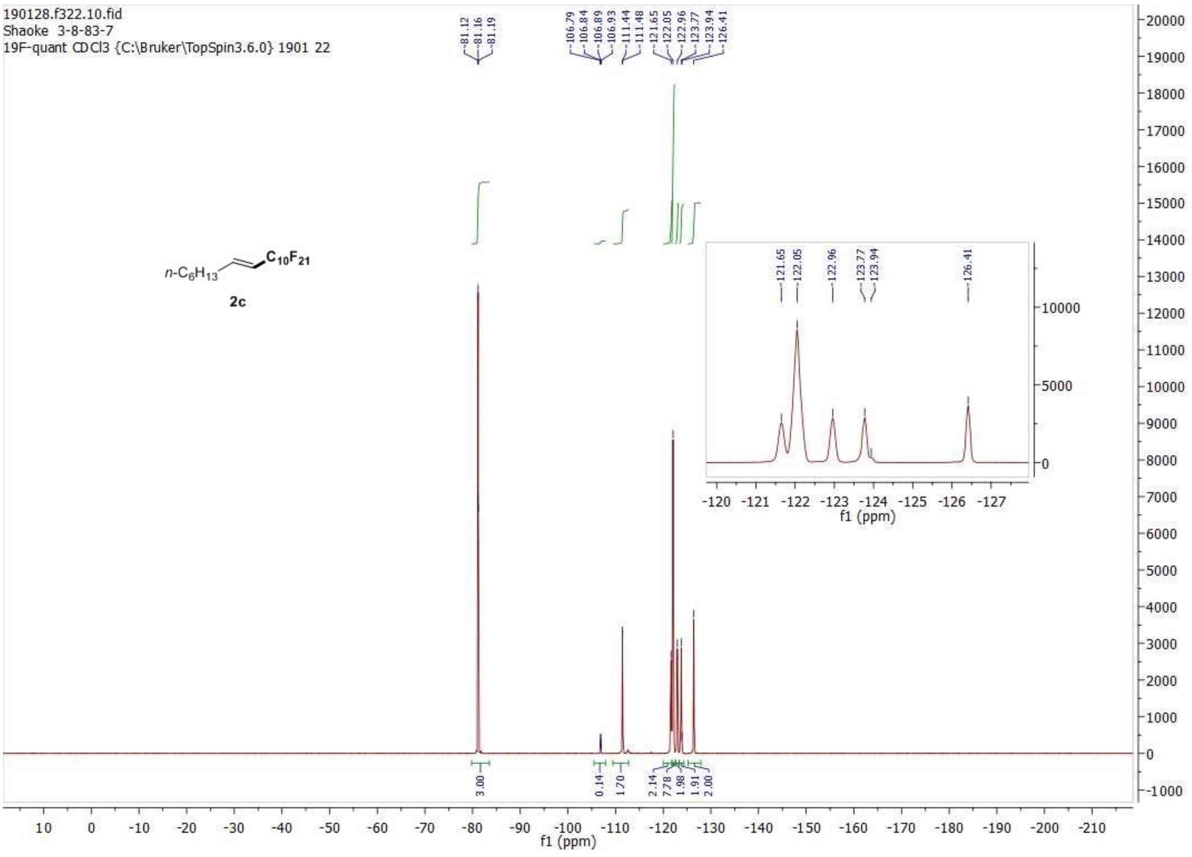

190308.f335.11.fid
Shaoke 3-8-107-5
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1903 35
7.26 CDCl3
6.46
6.44
6.41
6.38
6.36
6.35
6.16
6.14
6.10
5.66
5.62
5.57
5.53
5.50
2.36
2.31
2.24
2.22
2.22
2.20
2.17
1.54 HDO
1.50
1.47
1.45
1.43
1.42
1.40
1.38
1.35
1.33
1.31
1.28
1.26
0.94
0.92
0.89
n-C$_4$H$_9$
C$_{10}$F$_{21}$
2b
0.98
1.00
1.94
4.29
3.09
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0

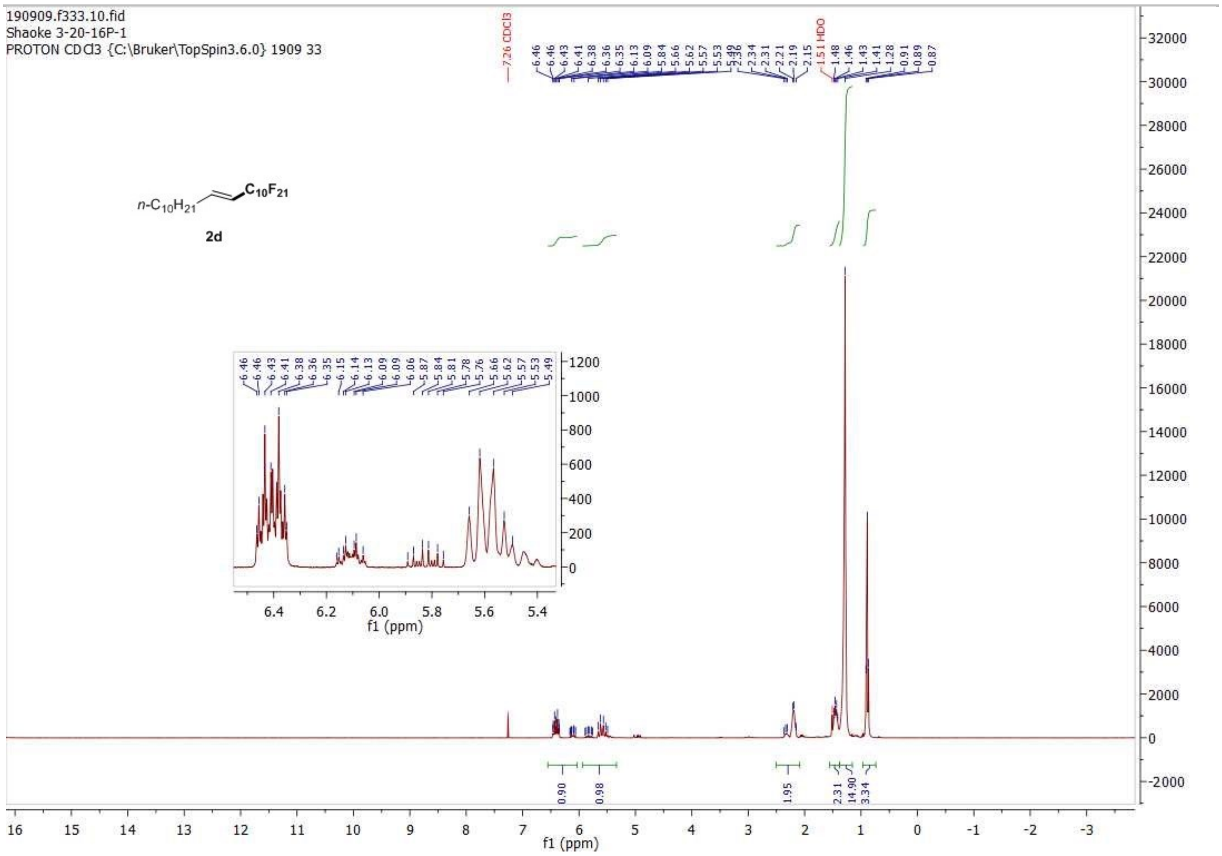

190308.f335.12.fid
Shaoke 3-8-107-5
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1903 35
143.29
116.76
77.00 CDCl3
31.69
30.07
22.03
13.74
n-C$_4$H$_9$
C$_{10}$F$_{21}$
2b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
45
40
35
30
25
20
15
10
5
0

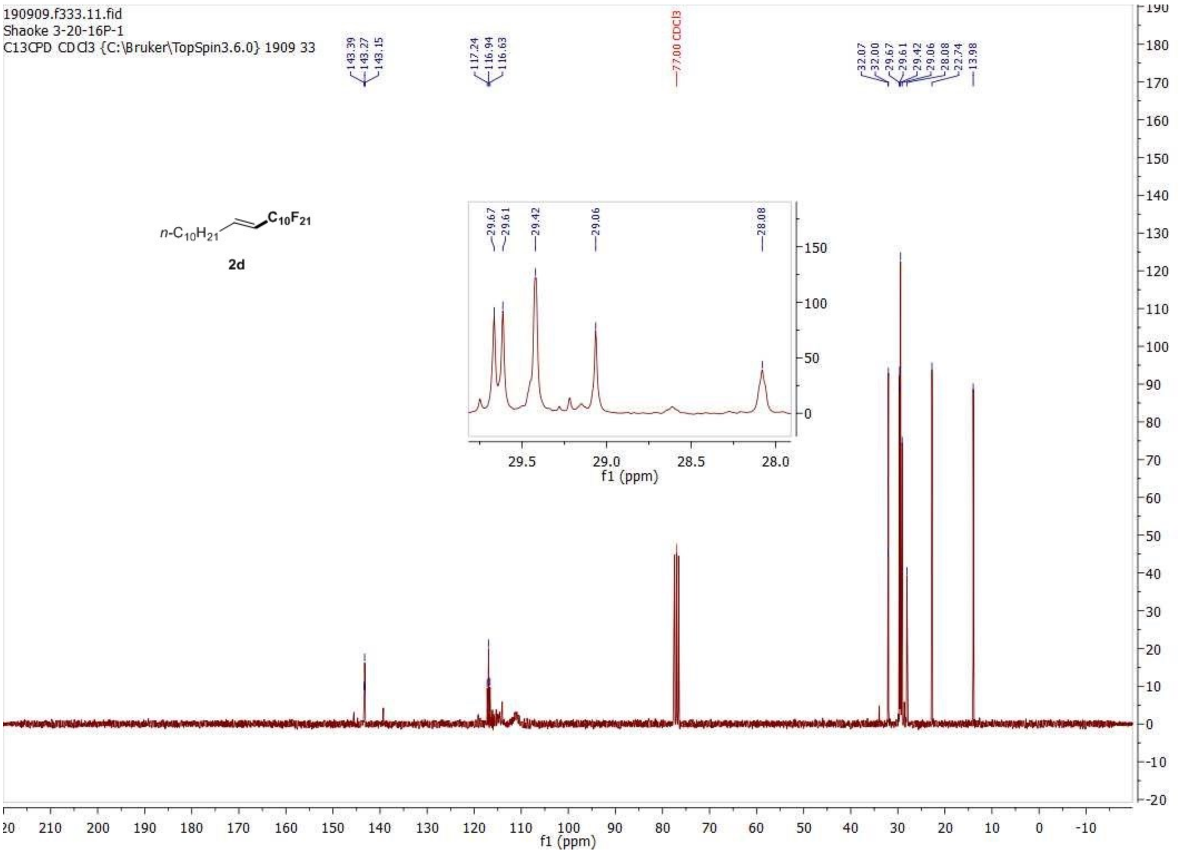

190308.f335.10.fid
Shaoke 3-8-107-5
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1903 35
-80.77
-80.80
-80.84
-106.60
-106.64
-106.69
-106.74
-111.16
-111.20
-111.25
-111.28
-121.48
-121.84
-122.72
-123.56
-123.74
-126.14
n-C4H9
C10F21
2b
3.00
0.31
1.60
1.88
8.02
1.96
1.90
1.98
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

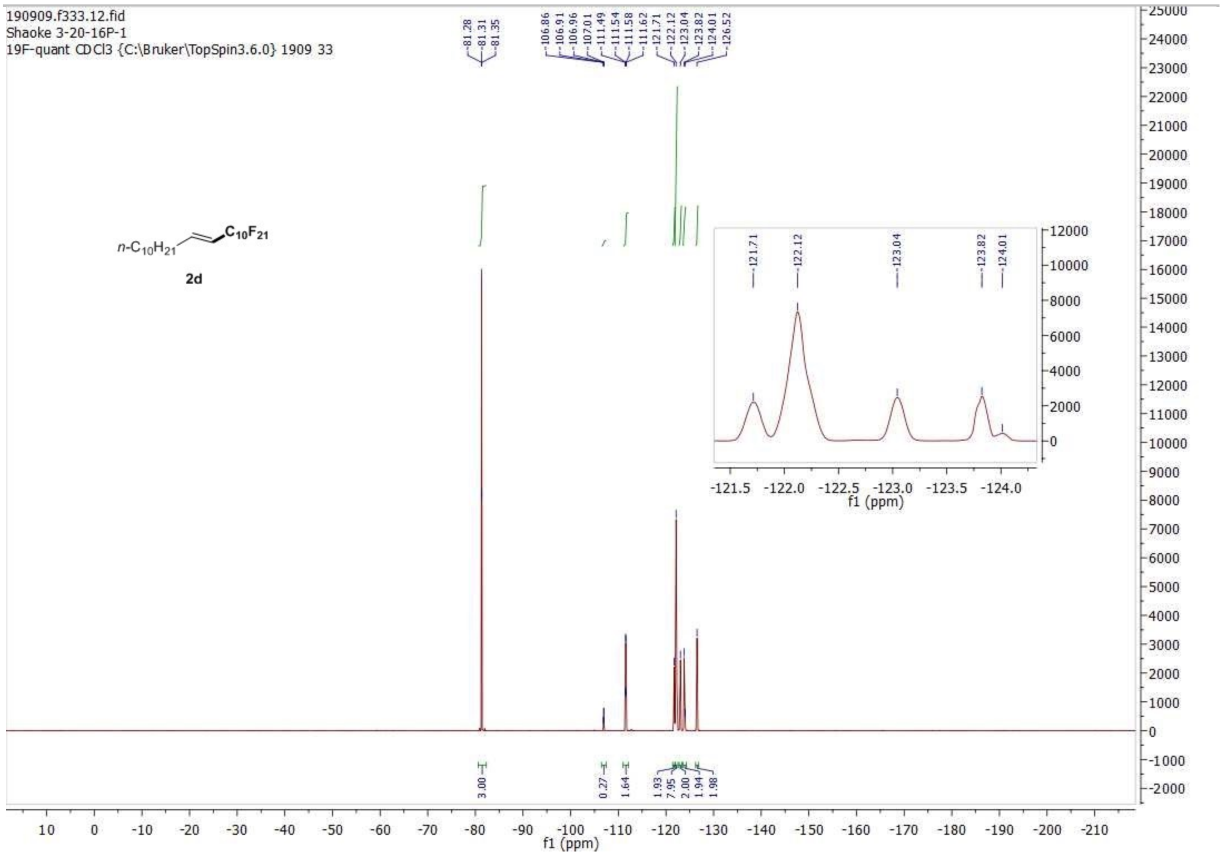

190125.304.10.fid
Shaoke 3-8-83-7
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 4
7.2600 CDCl3
6.4501
6.4425
6.4347
6.4271
6.4197
6.4116
6.4044
6.3975
6.3903
6.3746
6.3517
6.3445
5.6517
5.6112
5.5596
5.5188
2.3278
2.3034
2.2068
2.1817
2.1666
1.4729
1.4498
1.4245
1.4026
1.2911
0.9069
0.8850
0.8621
n-C6H13
C10F21
2c
0.82
0.86
1.99
2.04
6.85
3.36
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

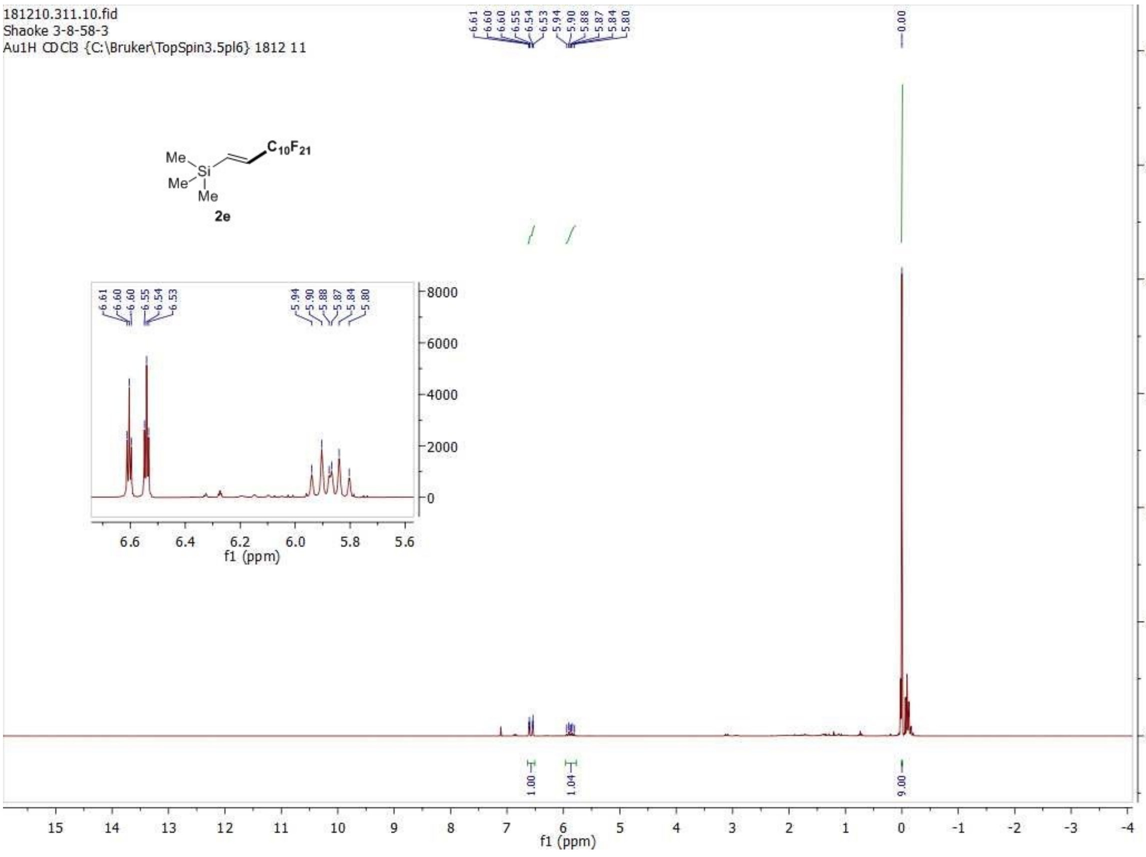

190125.304.11.fid
Shaoke 3-8-83-7
Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 4
143.34
143.22
143.10
117.05
116.74
116.44
76.94 CDCl3
31.96
31.48
28.58
27.90
22.46
13.84
n-C6H13
C10F21
2c
f1 (ppm)

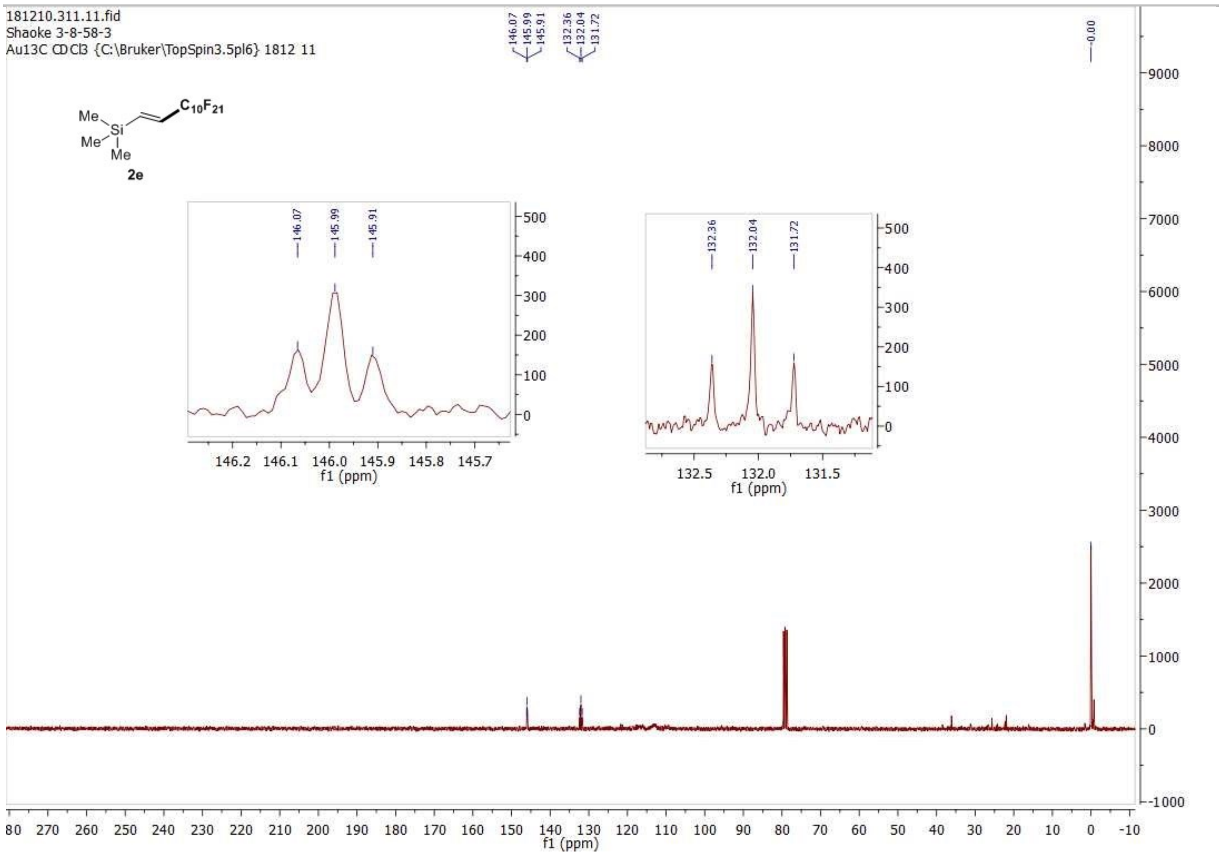

190128.f322.10.fid
Shaoke 3-8-83-7
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 22
-81.12
-81.16
-81.19
-106.79
-106.84
-106.89
-106.93
-111.44
-111.48
-121.65
-122.05
-122.96
-123.77
-123.94
-126.41
n-C6H13
C10F21
2c
3.00
0.14
1.70
2.14
7.78
1.98
1.91
2.00
f1 (ppm)

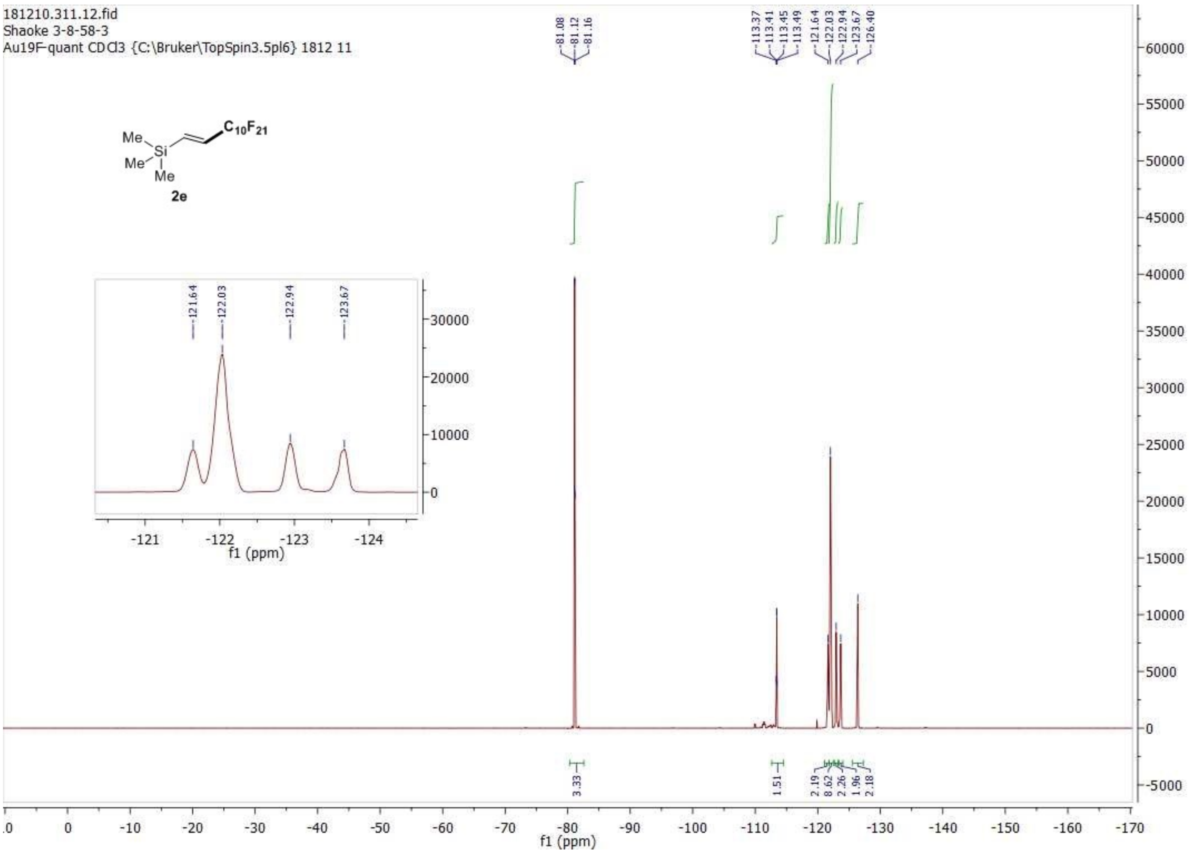

190909.f333.10.fid
Shaoke 3-20-16P-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1909 33
7.26 CDCl3
6.46
6.46
6.43
6.41
6.38
6.36
6.35
6.13
6.09
5.84
5.66
5.62
5.57
5.53
5.49
2.36
2.34
2.31
2.21
2.19
2.15
1.51 HDO
1.48
1.46
1.43
1.41
1.28
0.91
0.89
0.87
n-$C_{10}H_{21}$
$C_{10}F_{21}$
2d
6.46
6.46
6.43
6.41
6.38
6.36
6.35
6.15
6.14
6.13
6.09
6.09
6.06
5.87
5.84
5.81
5.78
5.76
5.66
5.62
5.57
5.53
5.49
1200
1000
800
600
400
200
0
6.4
6.2
6.0
5.8
5.6
5.4
f1 (ppm)
0.90
0.98
1.95
2.31
14.90
3.34
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

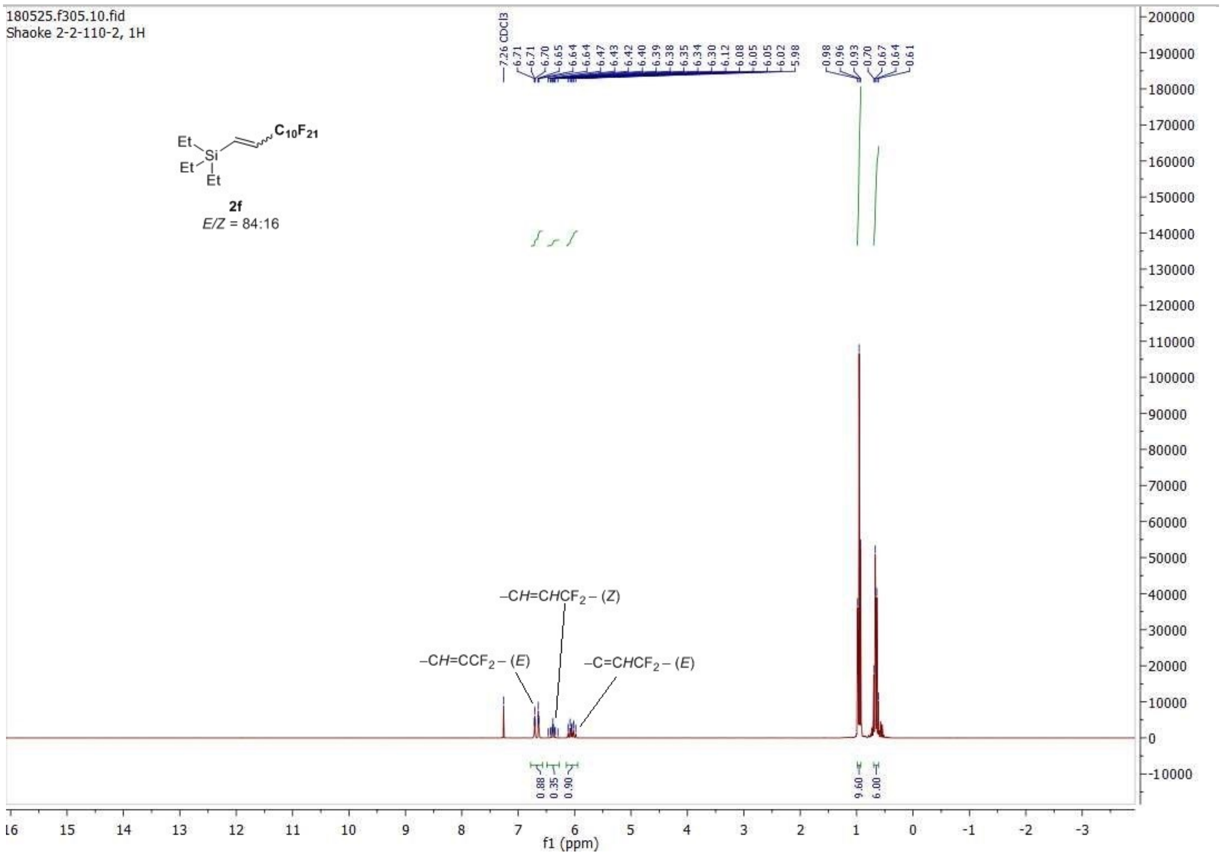

190909.f333.11.fid
Shaoke 3-20-16P-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1909 33
143.39
143.27
143.15
117.24
116.94
116.63
77.00 CDCl3
32.07
32.00
29.67
29.61
29.42
29.06
28.08
22.74
13.98
n-$C_{10}H_{21}$
$C_{10}F_{21}$
2d
29.67
29.61
29.42
29.06
28.08
150
100
50
0
29.5
29.0
28.5
28.0
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

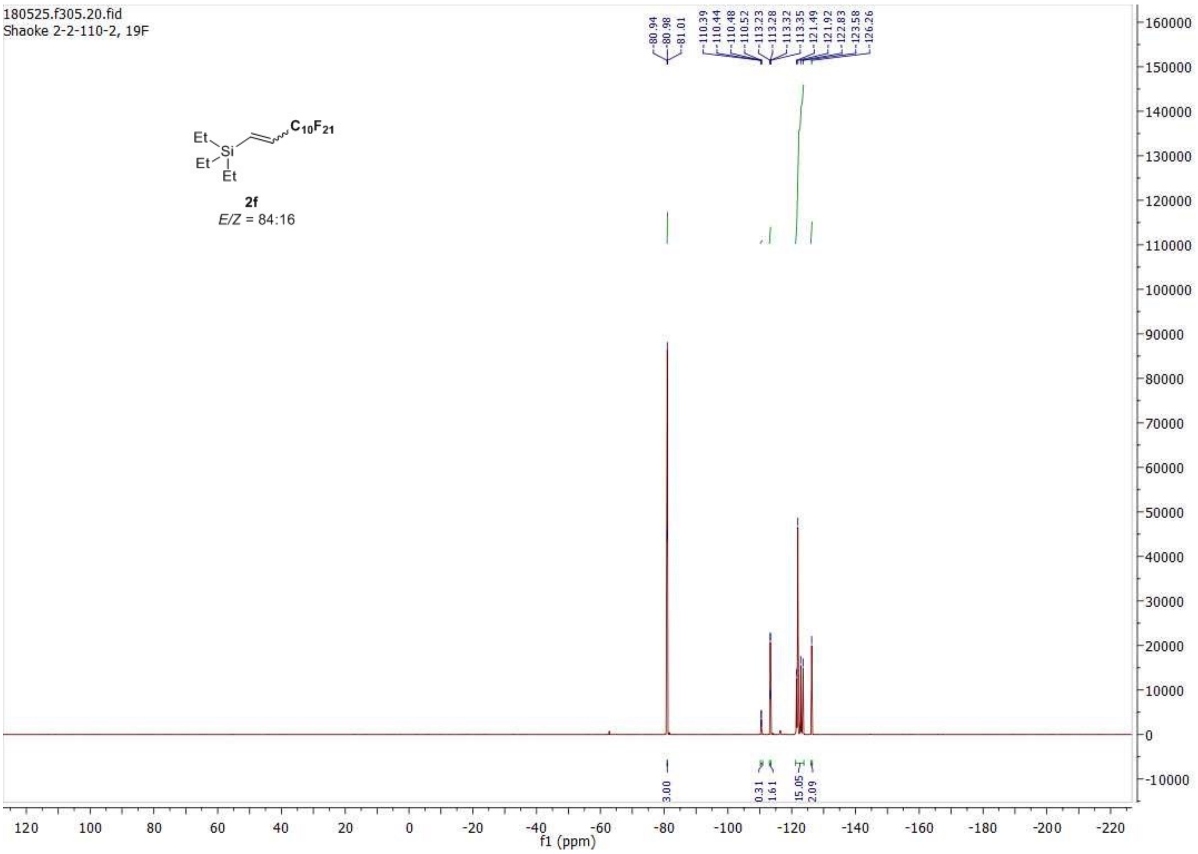

190909.f333.12.fid
Shaoke 3-20-16P-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1909 33
-81.28
-81.31
-81.35
-106.86
-106.91
-106.96
-107.01
-111.49
-111.54
-111.58
-111.62
-121.71
-122.12
-123.04
-123.82
-124.01
-126.52
n-C$_{10}$H$_{21}$
C$_{10}$F$_{21}$
2d
3.00
0.27
1.64
1.93
7.95
2.00
1.94
1.98
f1 (ppm)

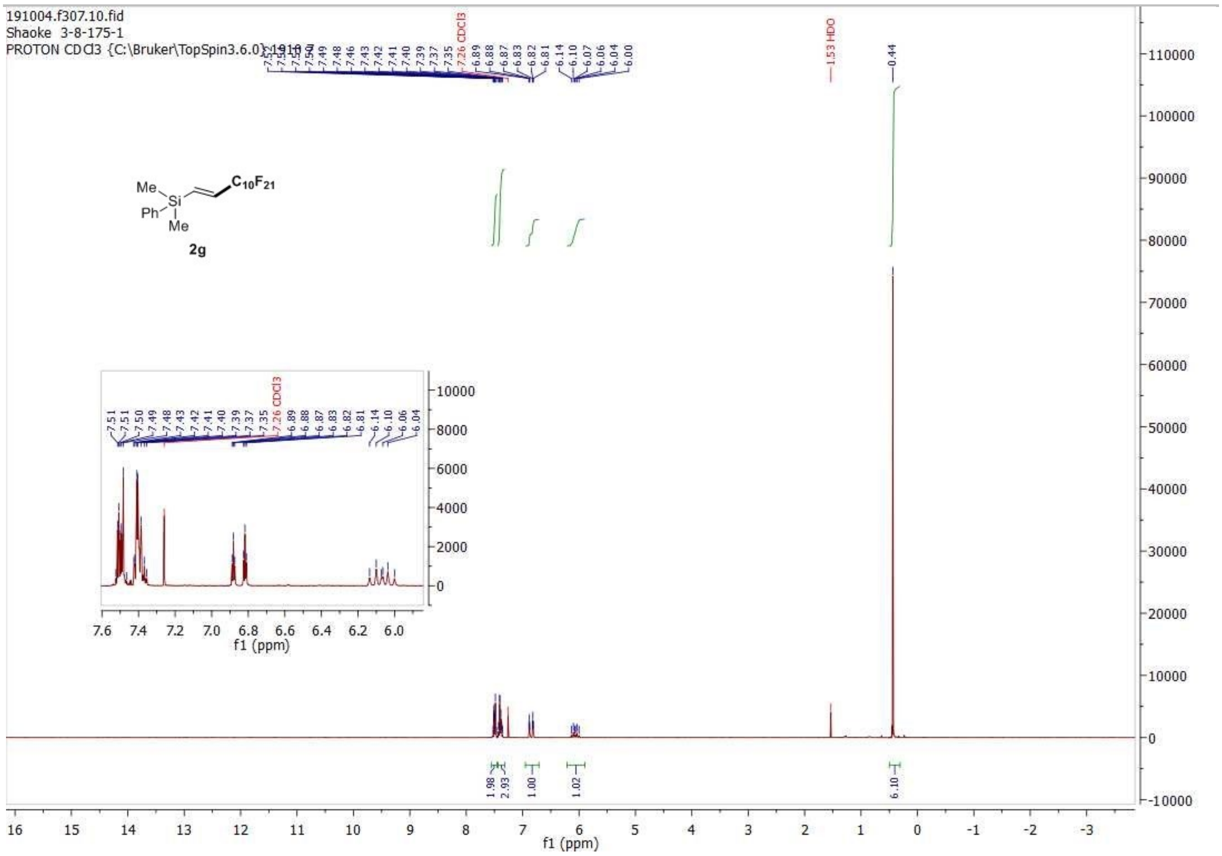

181210.311.10.fid
Shaoke 3-8-58-3
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1812 11
6.61
6.60
6.60
6.55
6.54
6.53
5.94
5.90
5.88
5.87
5.84
5.80
0.00
Me
Si
Me
Me
C$_{10}$F$_{21}$
2e
1.00
1.04
9.00
f1 (ppm)

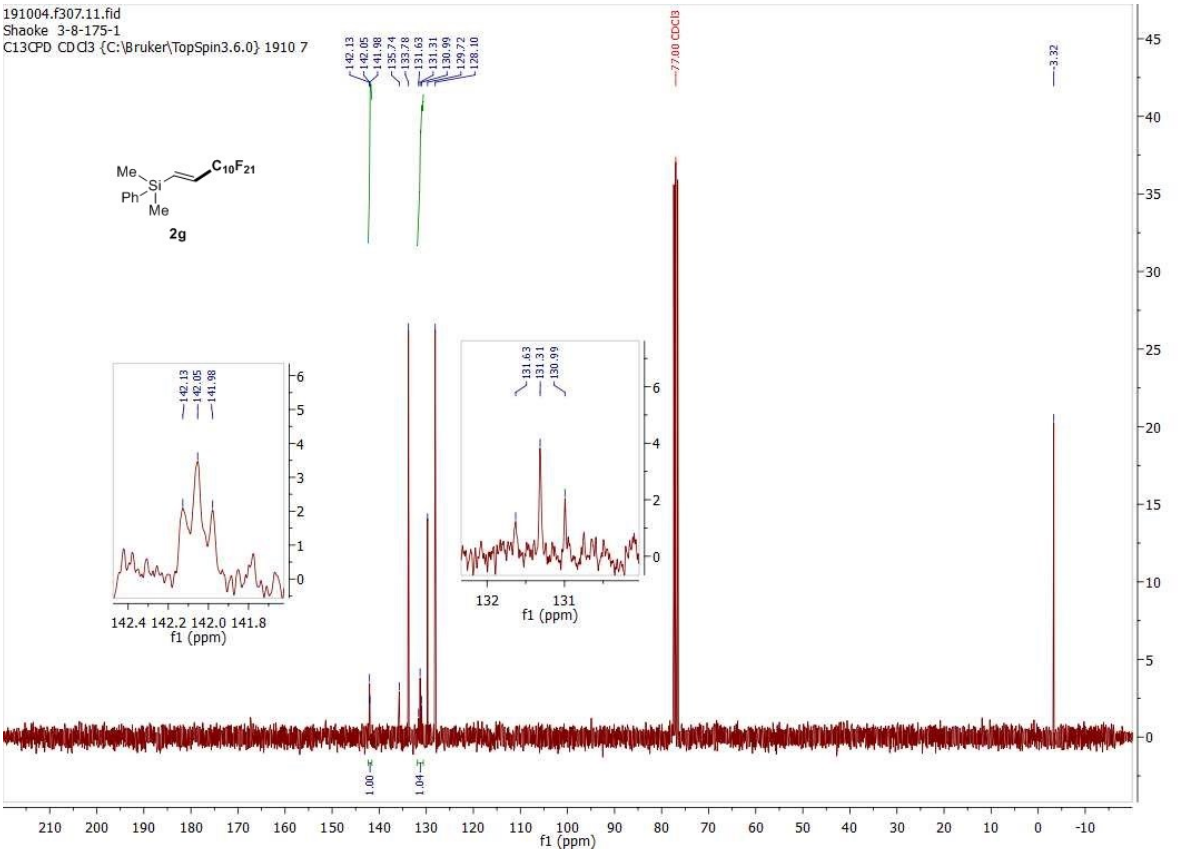

181210.311.11.fid
Shaoke 3-8-58-3
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1812 11
146.07
145.99
145.91
132.36
132.04
131.72
0.00
Me
Si
Me
Me
$C_{10}F_{21}$
2e
146.2 146.1 146.0 145.9 145.8 145.7
f1 (ppm)
132.5 132.0 131.5
f1 (ppm)
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
280 270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

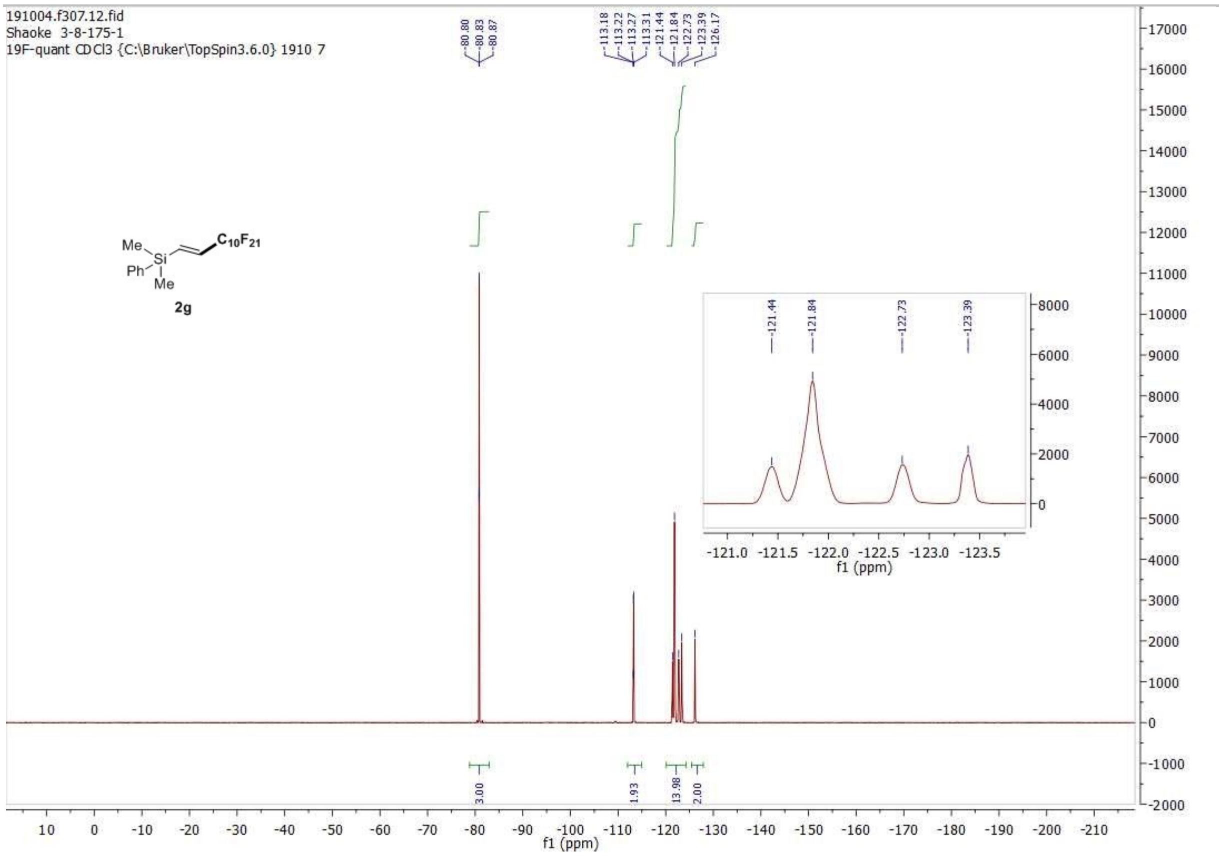

181210.311.12.fid
Shaoke 3-8-58-3
Au19F-quant CDCl3 {C:\Bruker\TopSpin3.5pl6} 1812 11
-81.08
-81.12
-81.16
-113.37
-113.41
-113.45
-113.49
-121.64
-122.03
-122.94
-123.67
-126.40
Me
Si
Me
Me
$C_{10}F_{21}$
2e
-121 -122 -123 -124
f1 (ppm)
3.33
1.51
2.19
8.62
2.26
1.96
2.18
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170
f1 (ppm)

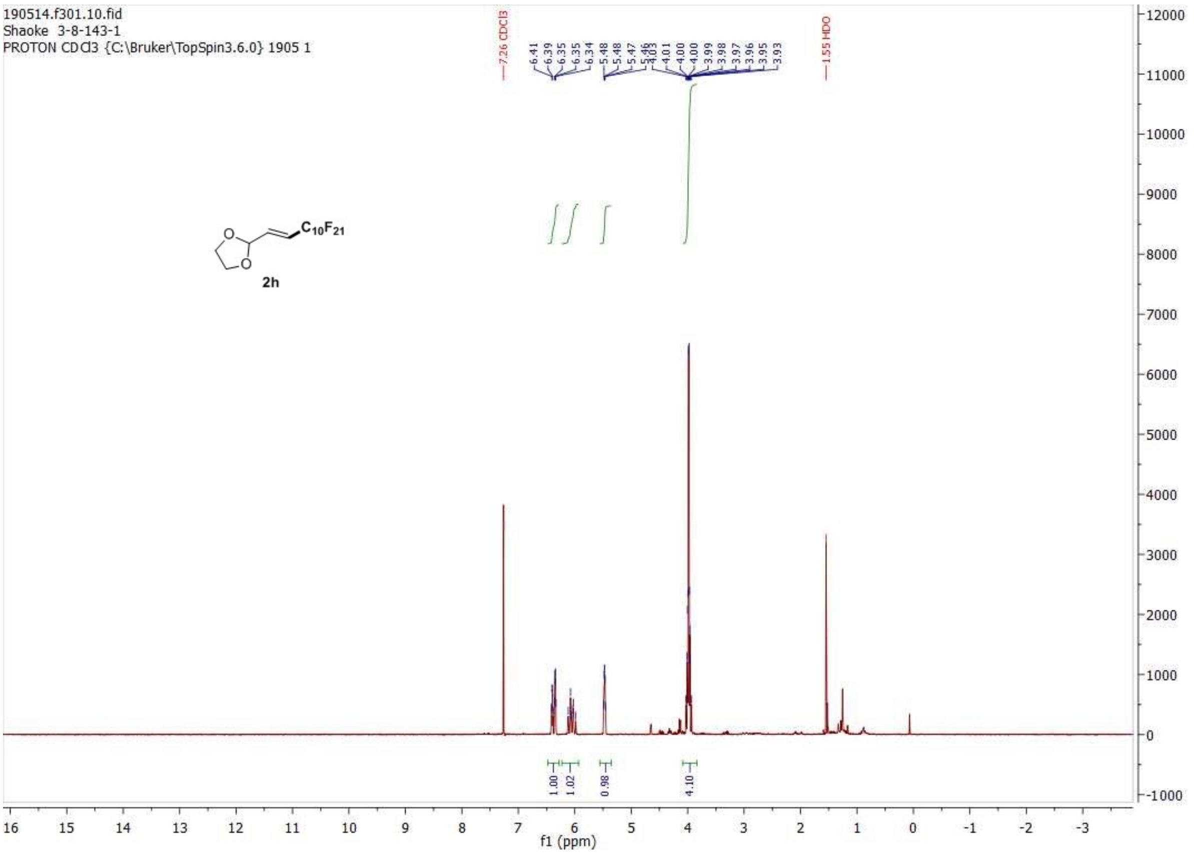

180525.f305.10.fid
Shaoke 2-2-110-2, 1H
7.26 CDCl3
6.71
6.71
6.70
6.65
6.64
6.64
6.47
6.43
6.42
6.40
6.39
6.38
6.35
6.34
6.30
6.12
6.08
6.05
6.05
6.02
5.98
0.98
0.96
0.93
0.70
0.67
0.64
0.61
Et
Si
Et
Et
C10F21
2f
E/Z = 84:16
–CH=CHCF2– (Z)
–CH=CCF2– (E)
–C=CHCF2– (E)
0.88
0.35
0.90
9.60
6.00
f1 (ppm)

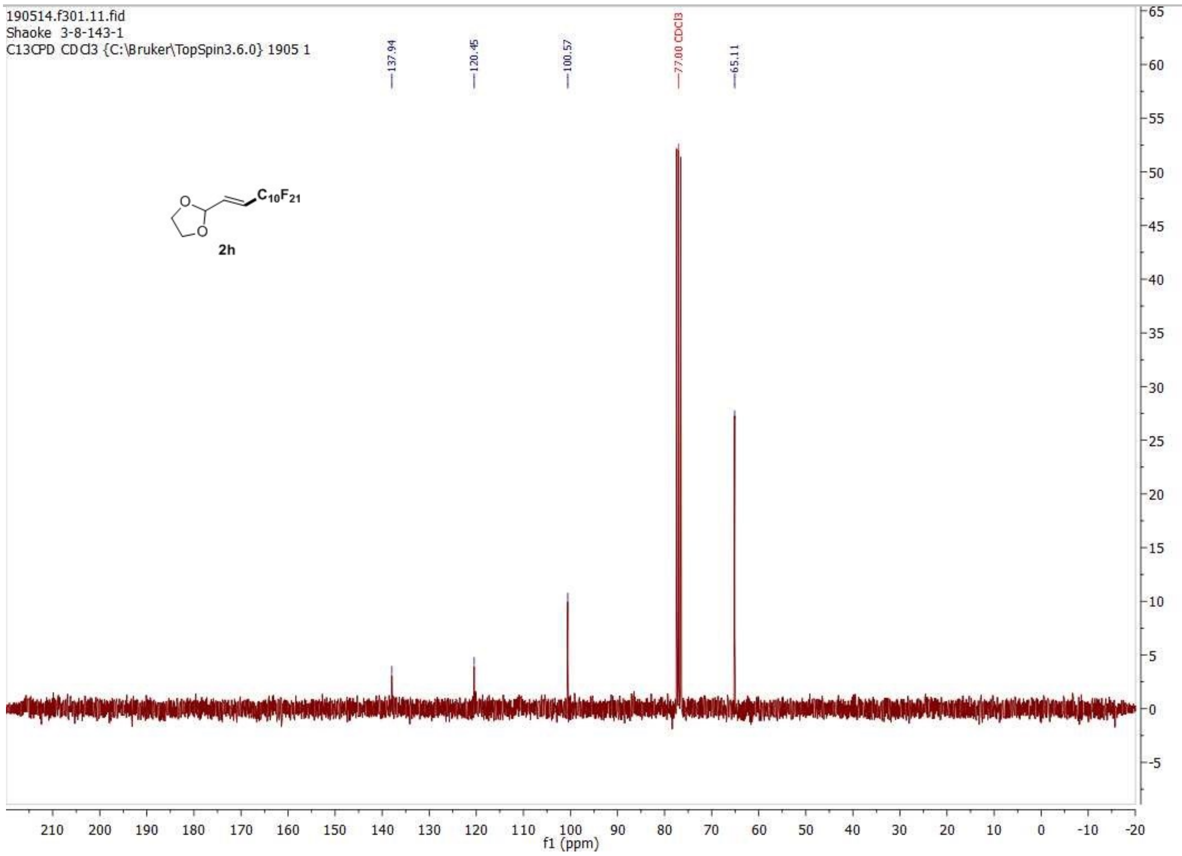

180525.f305.20.fid
Shaoke 2-2-110-2, 19F
-80.94
-80.98
-81.01
-110.39
-110.44
-110.48
-110.52
-113.23
-113.28
-113.32
-113.35
-121.49
-121.92
-122.83
-123.58
-126.26
Et
Si
Et
Et
C10F21
2f
E/Z = 84:16
3.00
0.31
1.61
15.05
2.09
f1 (ppm)

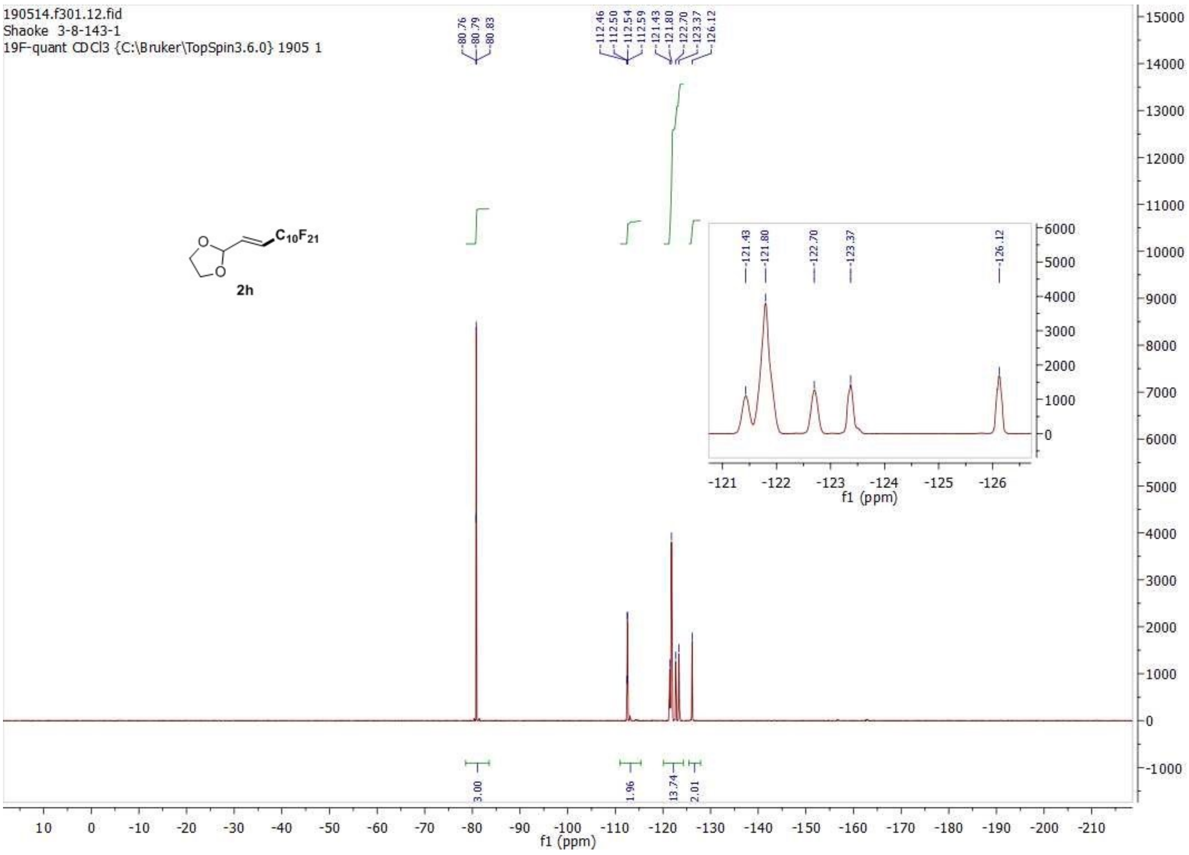

191004.f307.10.fid
Shaoke 3-8-175-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 7
7.51 7.51 7.50 7.49 7.48 7.46 7.43 7.42 7.41 7.40 7.39 7.37 7.35
7.26 CDCl3
6.89 6.88 6.87 6.83 6.82 6.81
6.14 6.10 6.07 6.06 6.04 6.00
1.53 HDO
0.44
Me
Si
Ph
Me
$C_{10}F_{21}$
2g
1.98
2.93
1.00
1.02
6.10
f1 (ppm)

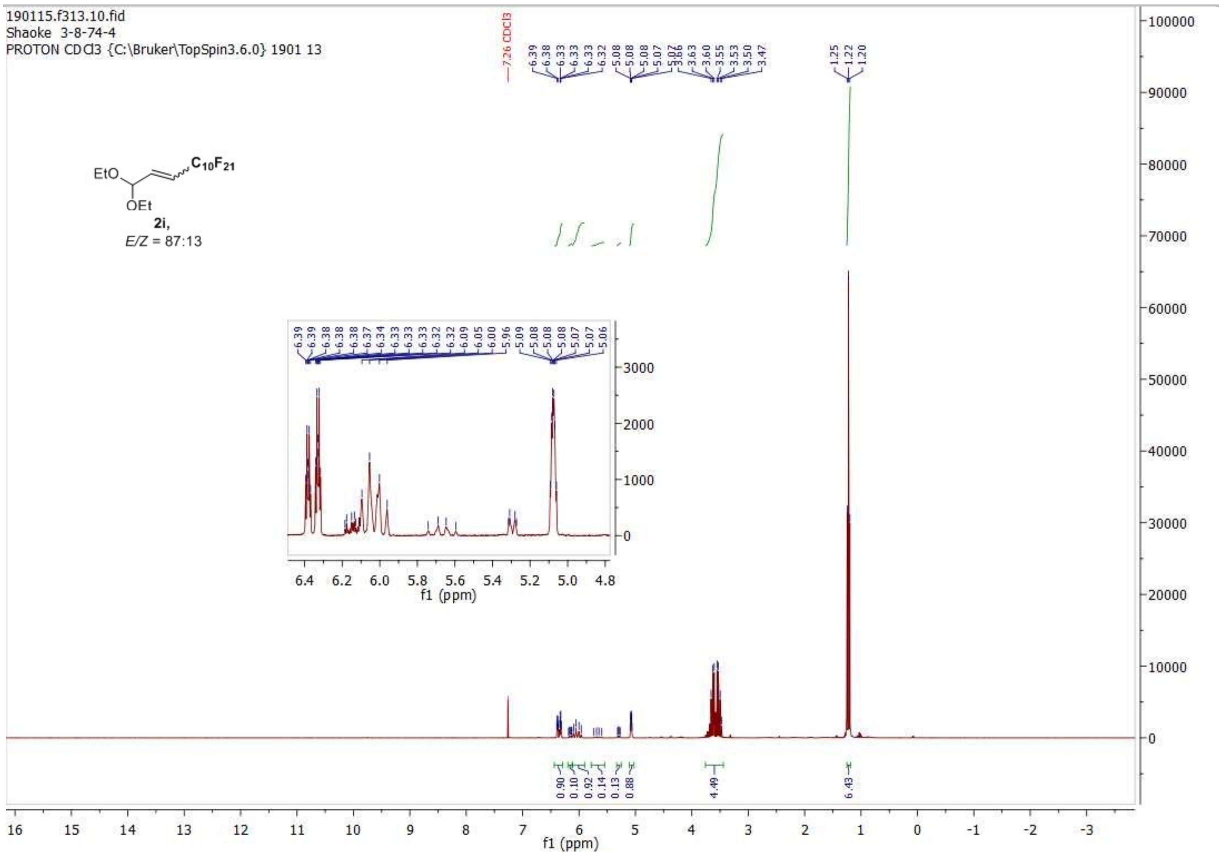

191004.f307.11.fid
Shaoke 3-8-175-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 7
142.13 142.05 141.98 135.74 133.78 131.63 131.31 130.99 129.72 128.10
77.00 CDCl3
-3.32
Me
Si
Ph
Me
$C_{10}F_{21}$
2g
1.00
1.04
f1 (ppm)

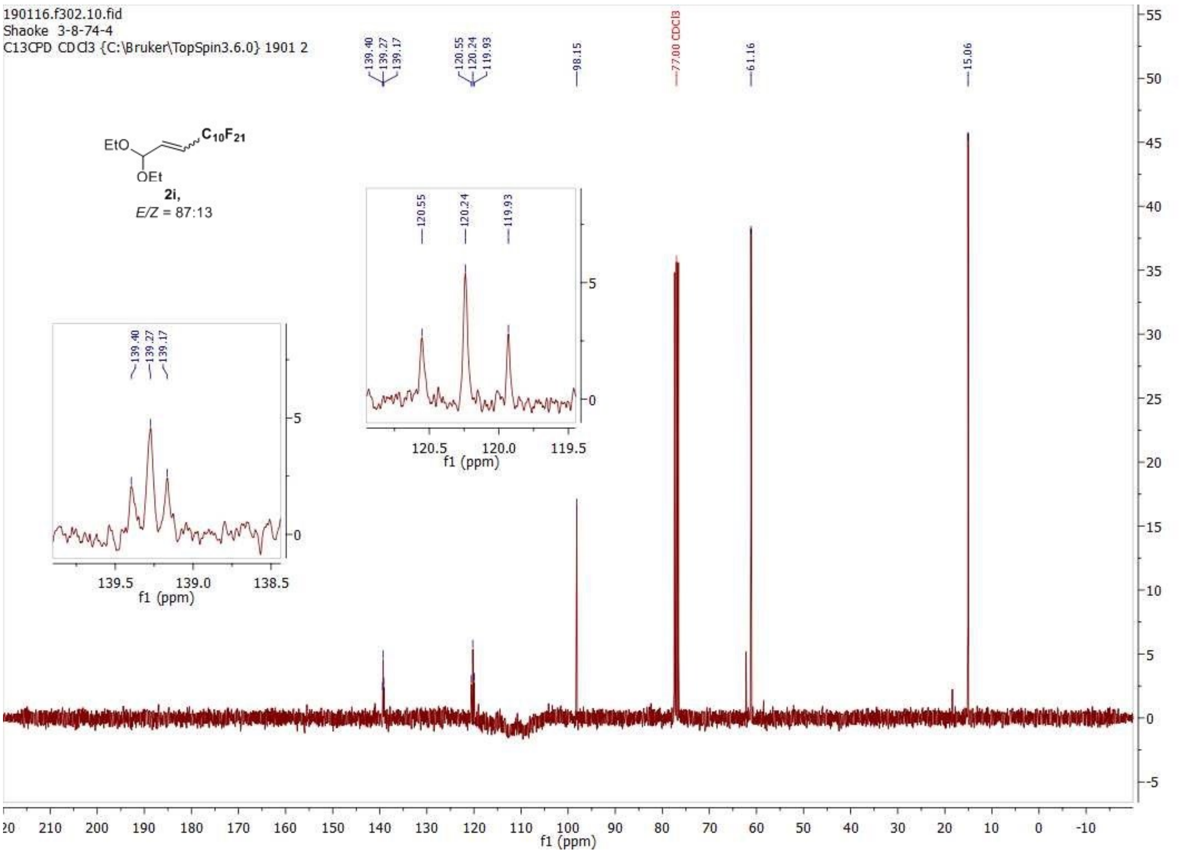

191004.f307.12.fid
Shaoke 3-8-175-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 7
-80.80
-80.83
-80.87
-113.18
-113.22
-113.27
-113.31
-121.44
-121.84
-122.73
-123.39
-126.17
Me
Si
Ph
Me
C10F21
2g
3.00
1.93
13.98
2.00
f1 (ppm)
-121.0 -121.5 -122.0 -122.5 -123.0 -123.5
8000
6000
4000
2000
0
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

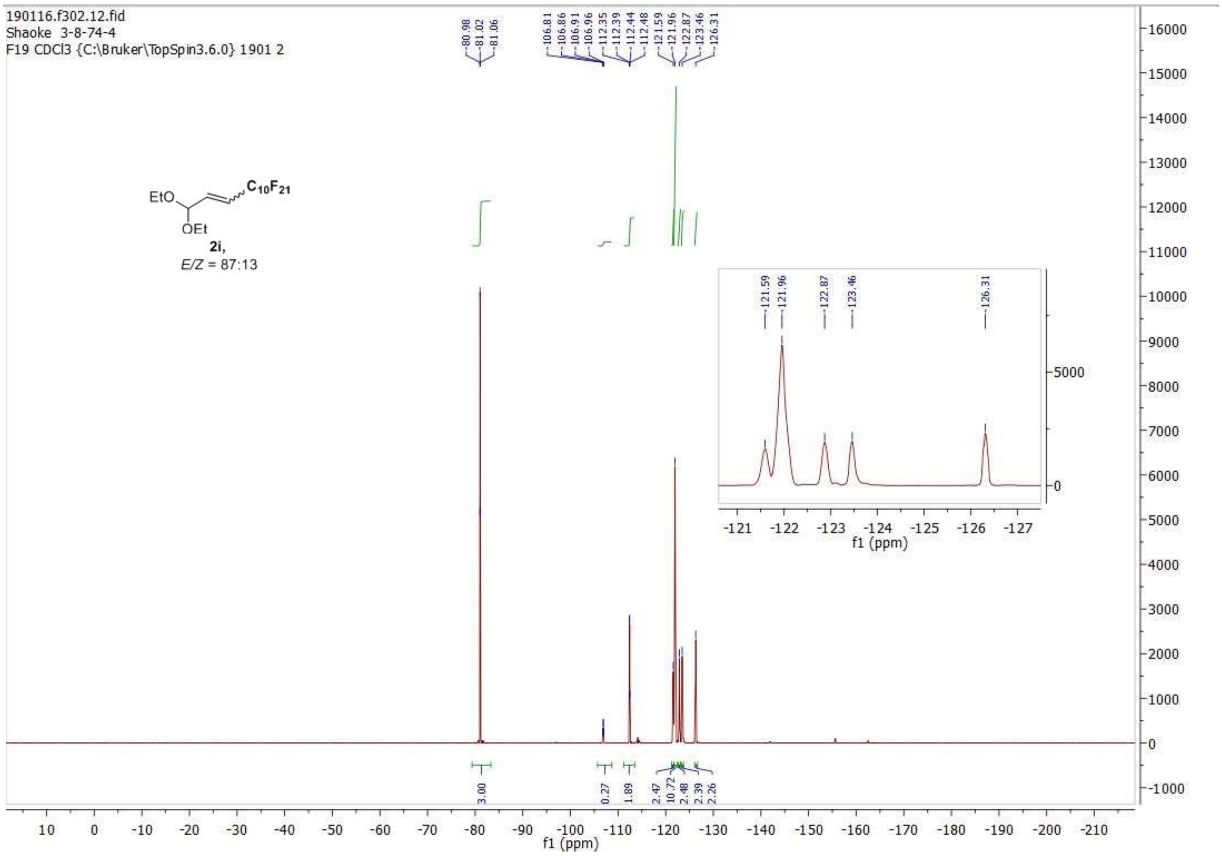

190514.f301.10.fid
Shaoke 3-8-143-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1905 1
7.26 CDCl3
6.41
6.39
6.35
6.35
6.34
5.48
5.48
5.47
5.46
4.03
4.01
4.00
4.00
3.99
3.98
3.97
3.96
3.95
3.93
1.55 HDO
O
O
C10F21
2h
1.00
1.02
0.98
4.10
f1 (ppm)
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3


190514.f301.11.fid
Shaoke 3-8-143-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1905 1
137.94
120.45
100.57
77.00 CDCl3
65.11
C10F21
2h
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5

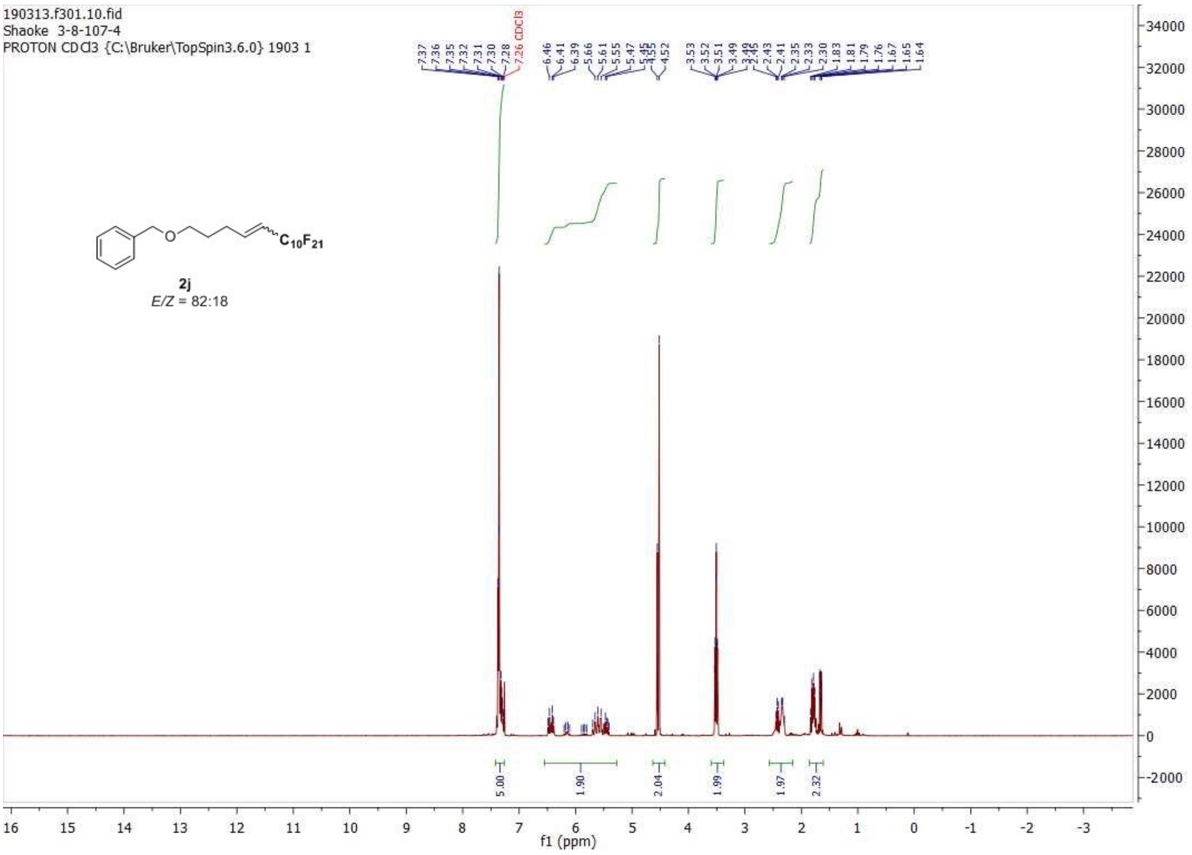

190514.f301.12.fid
Shaoke 3-8-143-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1905 1
-80.76
-80.79
-80.83
-112.46
-112.50
-112.54
-112.59
-121.43
-121.80
-122.70
-123.37
-126.12
C10F21
2h
3.00
1.96
13.74
2.01
f1 (ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
-121 -122 -123 -124 -125 -126
6000 5000 4000 3000 2000 1000 0


190115.f313.10.fid
Shaoke 3-8-74-4
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 13
EtO
OEt
C10F21
2i,
E/Z = 87:13
7.26 CDCl3
6.39
6.38
6.33
6.33
6.33
6.32
5.08
5.08
5.08
5.07
3.66
3.63
3.60
3.55
3.53
3.50
3.47
1.25
1.22
1.20
6.39
6.39
6.38
6.38
6.38
6.37
6.34
6.33
6.33
6.33
6.32
6.32
6.09
6.05
6.00
5.96
5.09
5.08
5.08
5.08
5.07
5.07
5.06
3000
2000
1000
0
6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8
f1 (ppm)
0.90
0.10
0.92
0.14
0.13
0.88
4.49
6.43
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

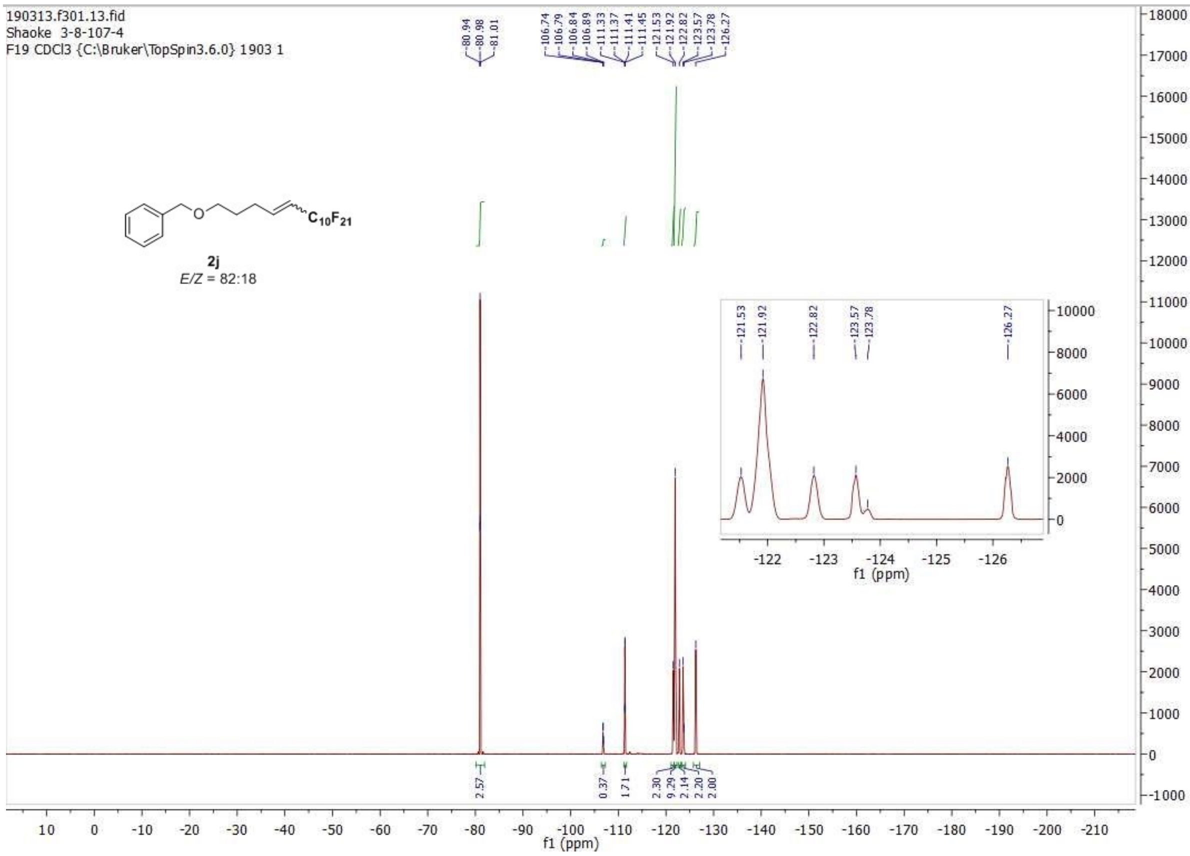

190116.f302.10.fid
Shaoke 3-8-74-4
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 2
139.40
139.27
139.17
120.55
120.24
119.93
98.15
77.00 CDCl3
61.16
15.06
EtO
OEt
C10F21
2i,
E/Z = 87:13
120.55
120.24
119.93
5
0
120.5 120.0 119.5
f1 (ppm)
139.40
139.27
139.17
5
0
139.5 139.0 138.5
f1 (ppm)
55
50
45
40
35
30
25
20
15
10
5
0
-5
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

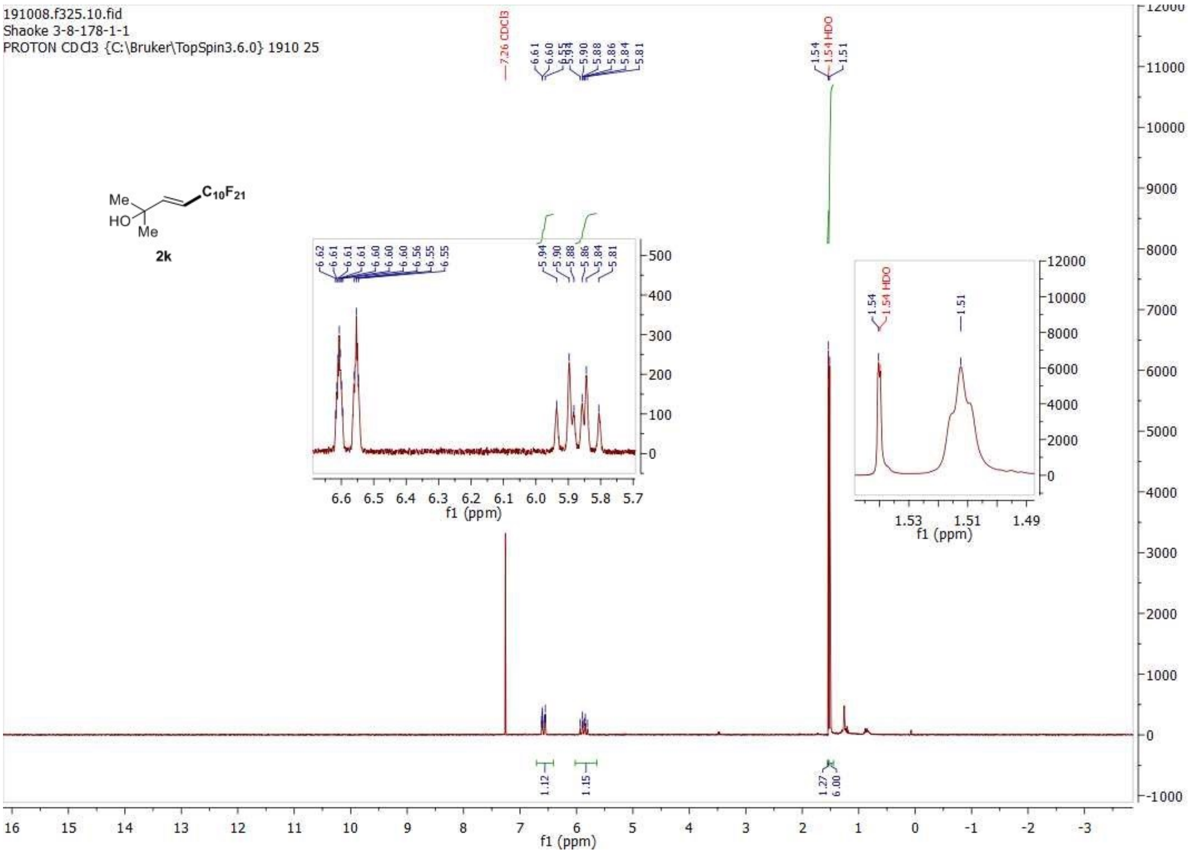


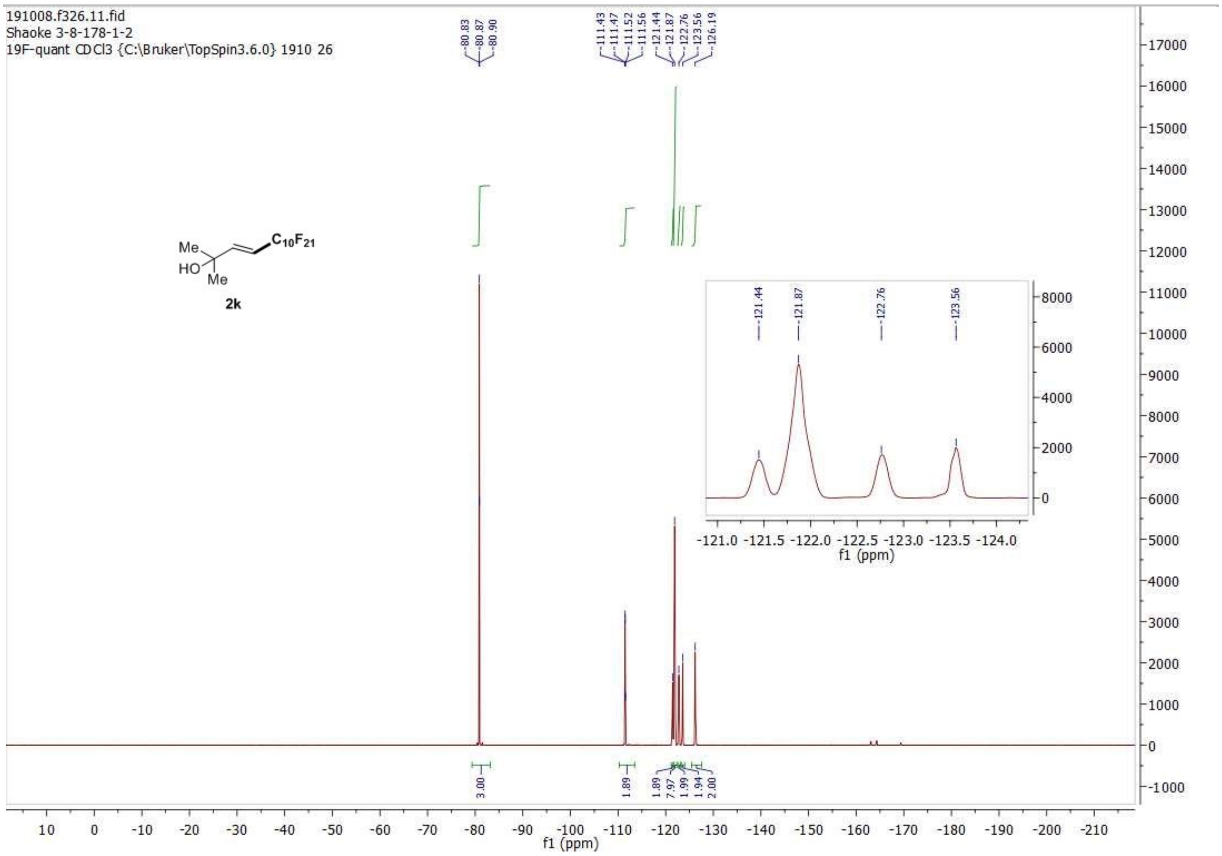
2i,
E/Z = 87:13

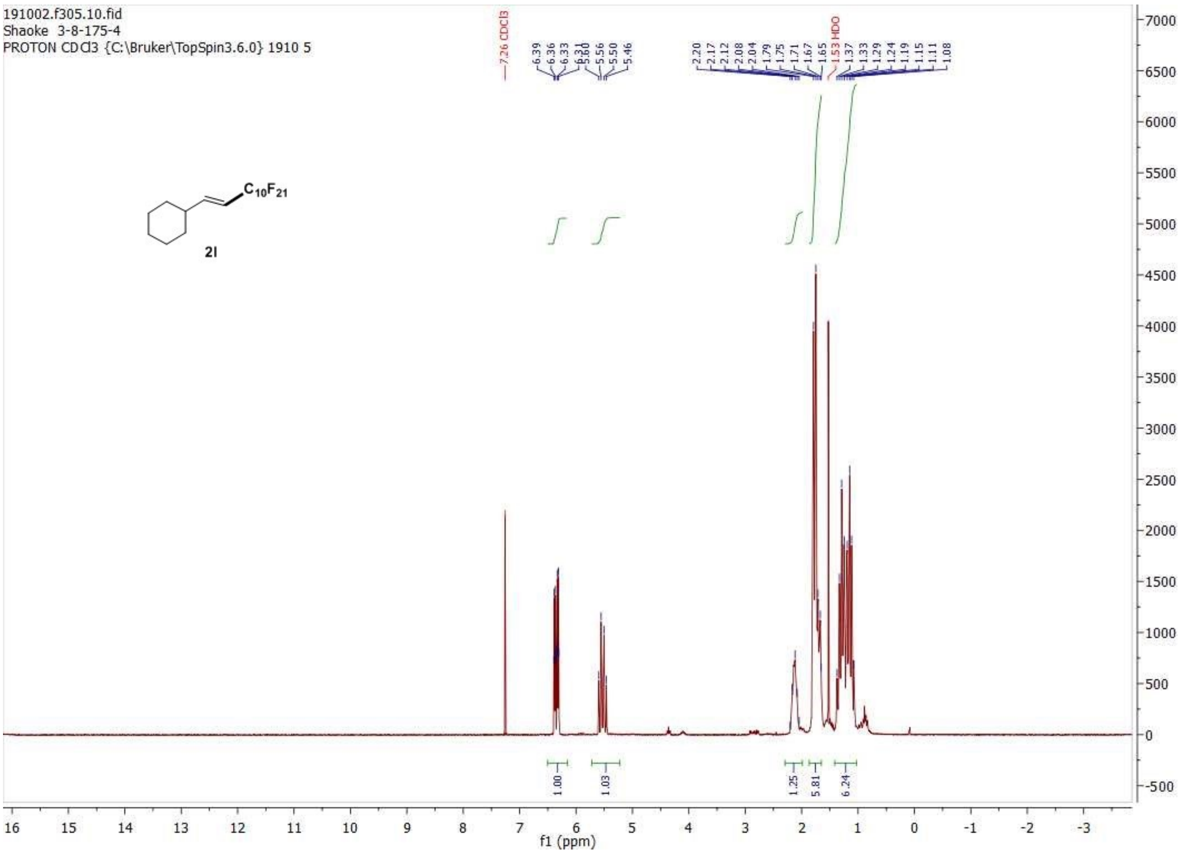


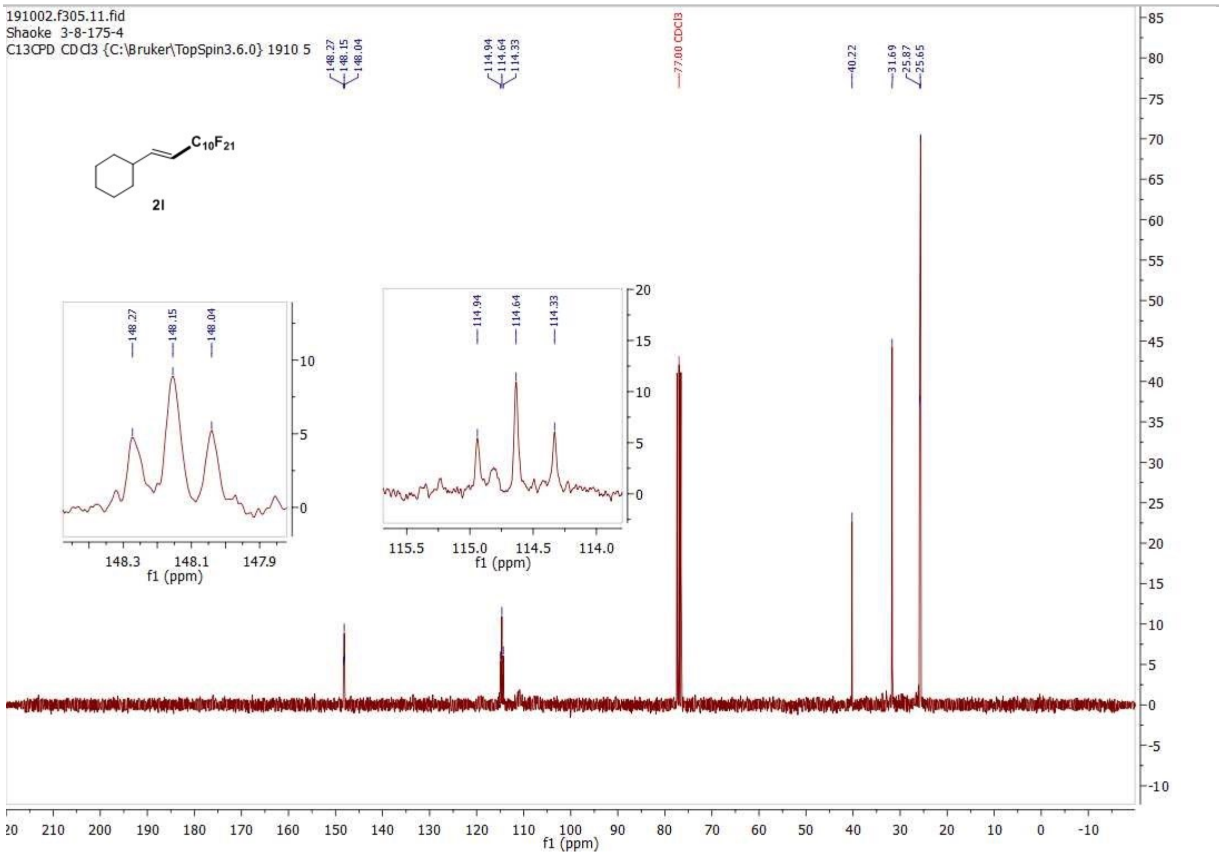
2j
E/Z = 82:18

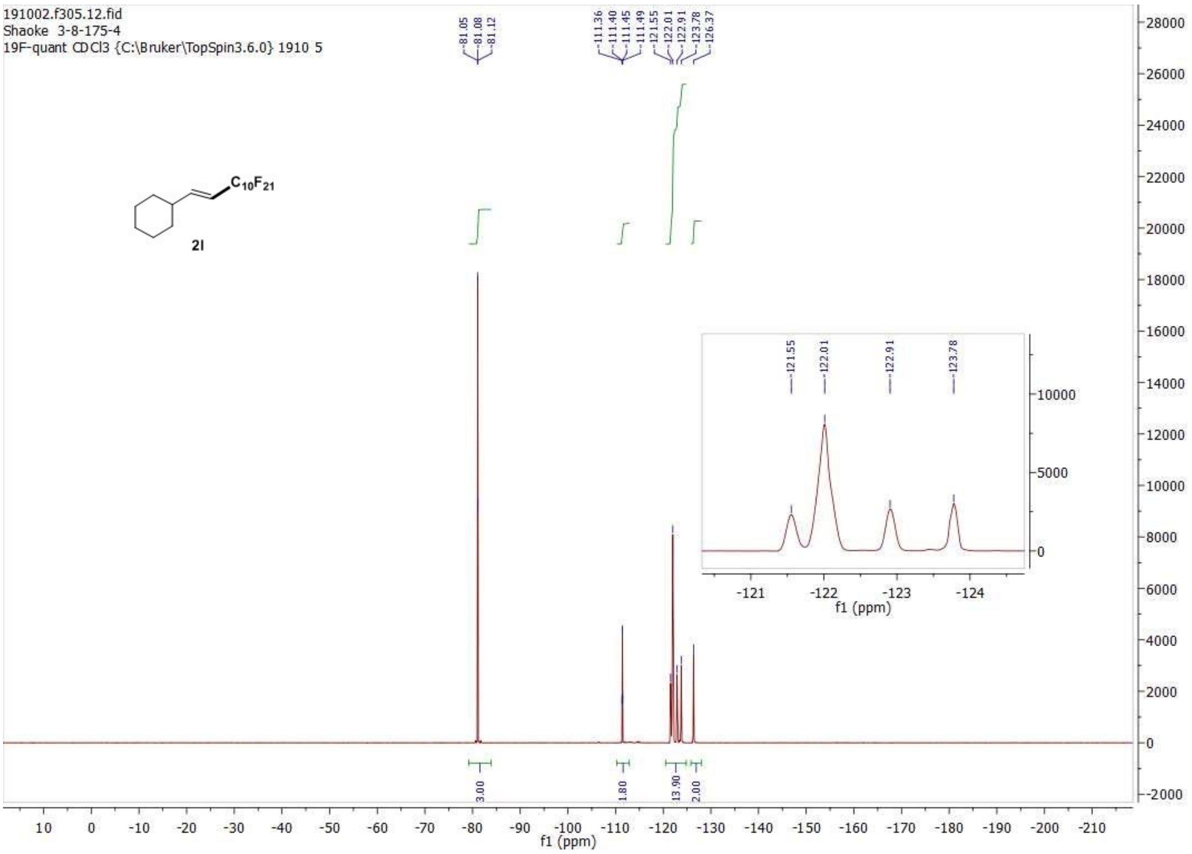

190313.f301.13.fid
Shaoke 3-8-107-4
F19 CDCl3 {C:\Bruker\TopSpin3.6.0} 1903 1
-80.94
-80.98
-81.01
-106.74
-106.79
-106.84
-106.89
-111.33
-111.37
-111.41
-111.45
-121.53
-121.92
-122.82
-123.57
-123.78
-126.27
O
$C_{10}F_{21}$
2j
E/Z = 82:18
2.57
0.37
1.71
2.30
9.29
2.14
2.20
2.00
f1 (ppm)

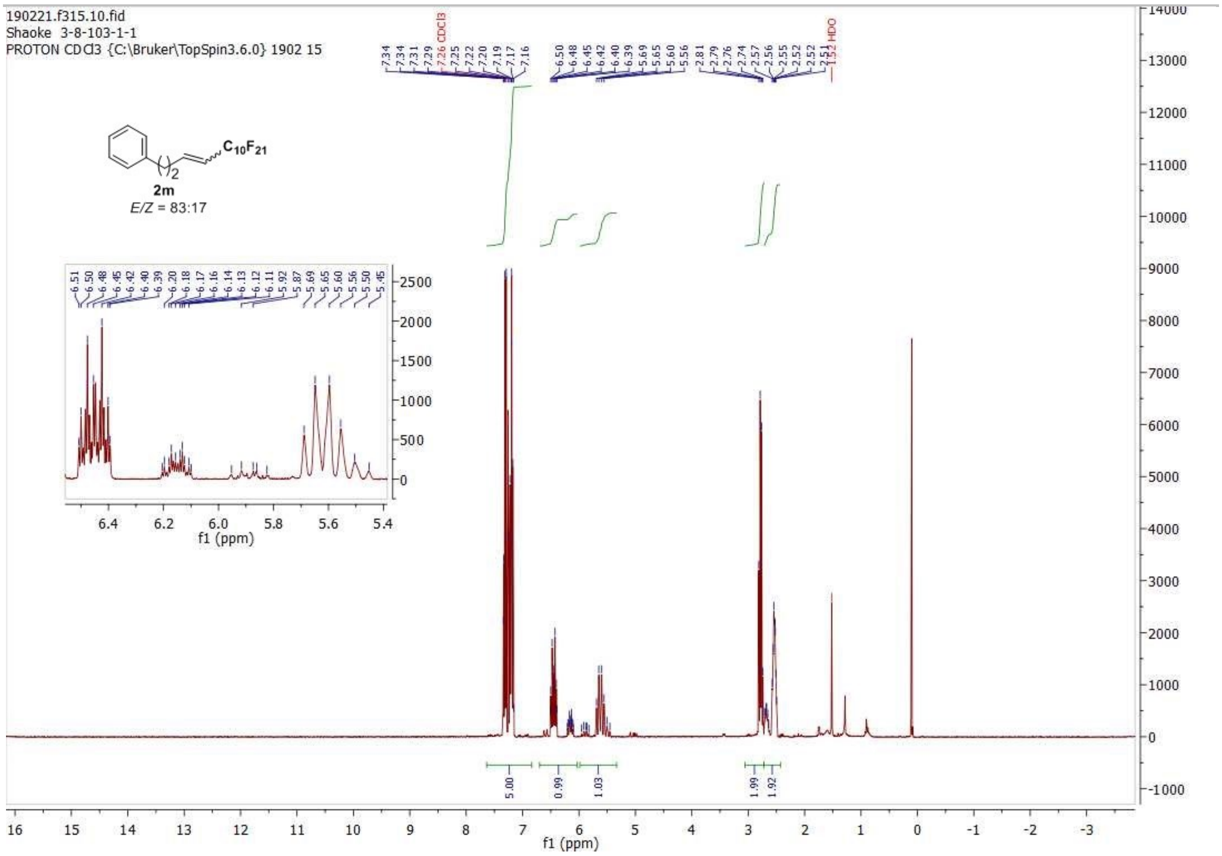

191008.f325.10.fid
Shaoke 3-8-178-1-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 25
7.26 CDCl3
6.61
6.60
6.55
5.94
5.90
5.88
5.86
5.84
5.81
1.54
1.54 HDO
1.51
Me
HO
Me
$C_{10}F_{21}$
2k
1.12
1.15
1.27
6.00
f1 (ppm)

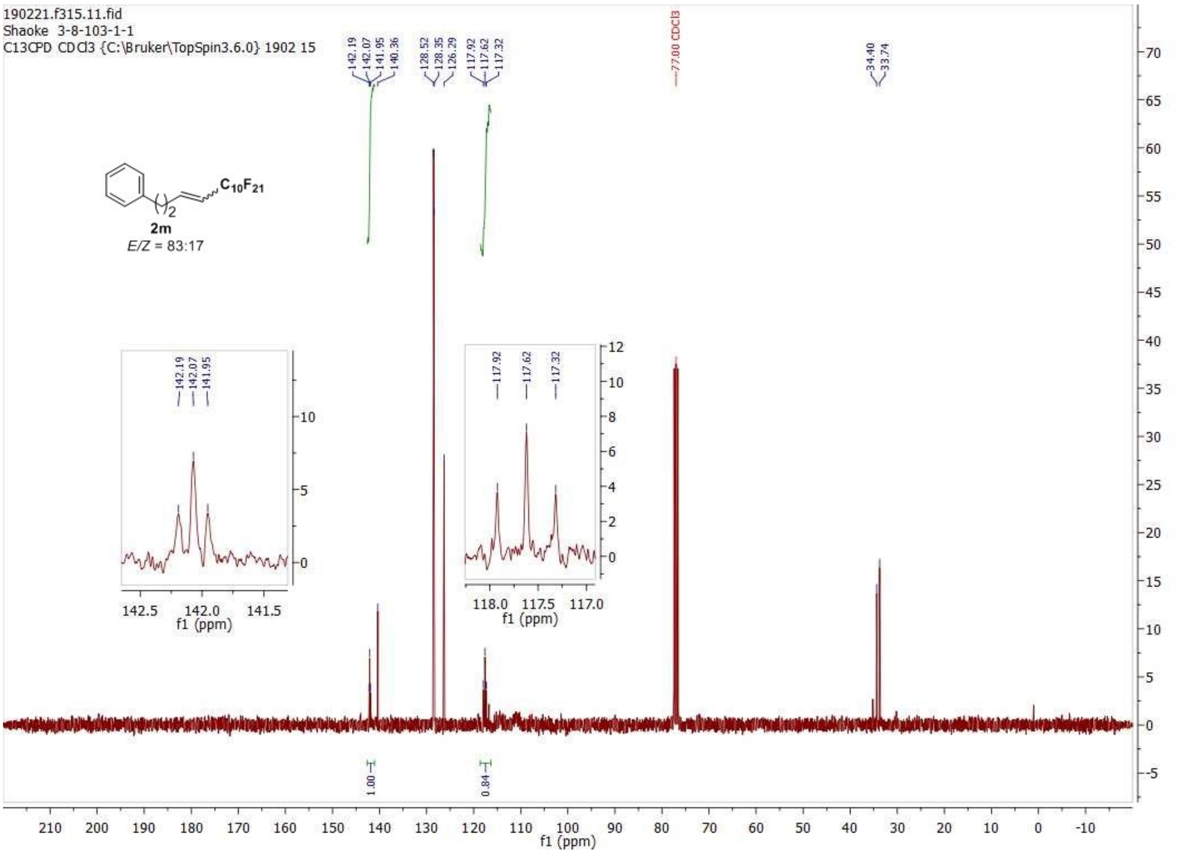

191008.f326.11.fid
Shaoke 3-8-178-1-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 26
-80.83
-80.87
-80.90
-111.43
-111.47
-111.52
-111.56
-121.44
-121.87
-122.76
-123.56
-126.19
Me
HO
Me
$C_{10}F_{21}$
2k
3.00
1.89
1.89
7.97
1.99
1.94
2.00
f1 (ppm)
-121.0 -121.5 -122.0 -122.5 -123.0 -123.5 -124.0

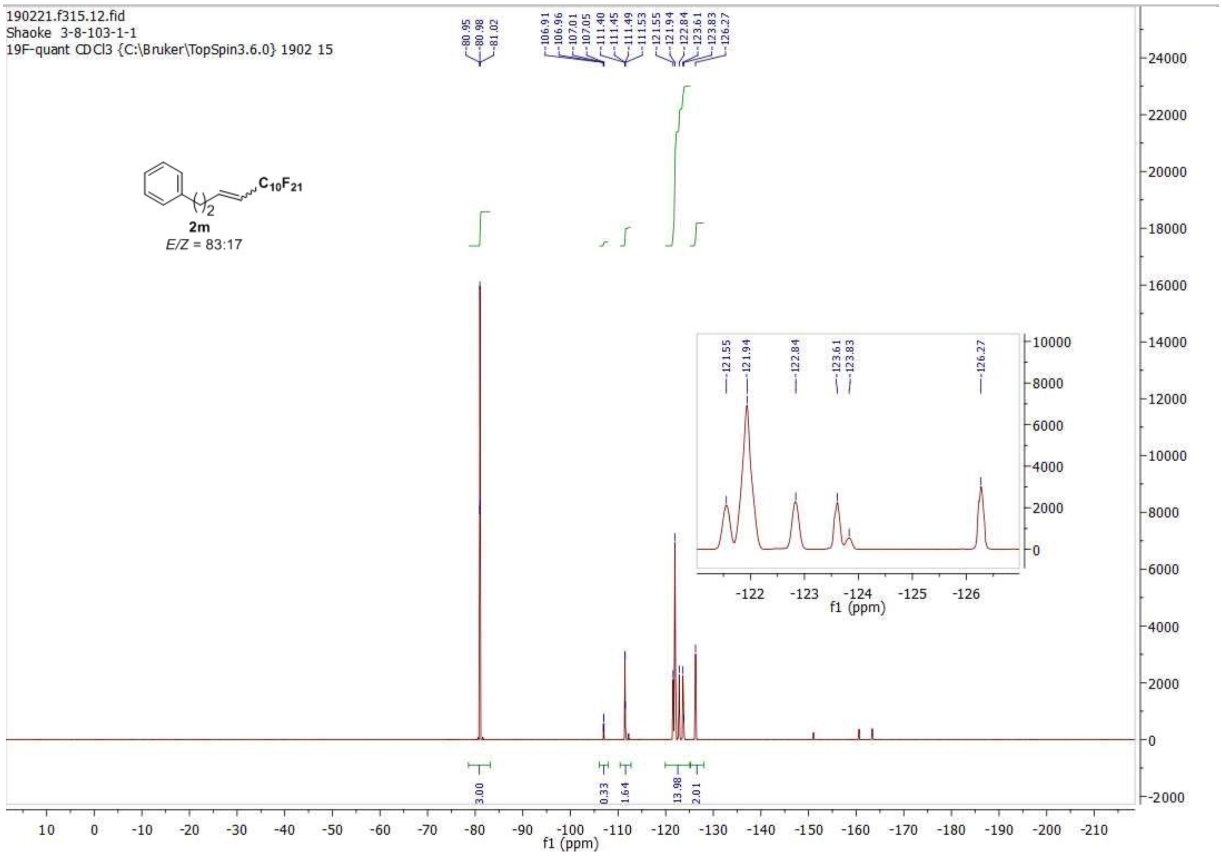

191002.f305.10.fid
Shaoke 3-8-175-4
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 5
7.26 CDCl3
6.39
6.36
6.33
6.31
5.60
5.56
5.50
5.46
2.20
2.17
2.12
2.08
2.04
1.79
1.75
1.71
1.67
1.65
1.53 HDO
1.37
1.33
1.29
1.24
1.19
1.15
1.11
1.08
$C_{10}F_{21}$
2l
1.00
1.03
1.25
5.81
6.24
f1 (ppm)

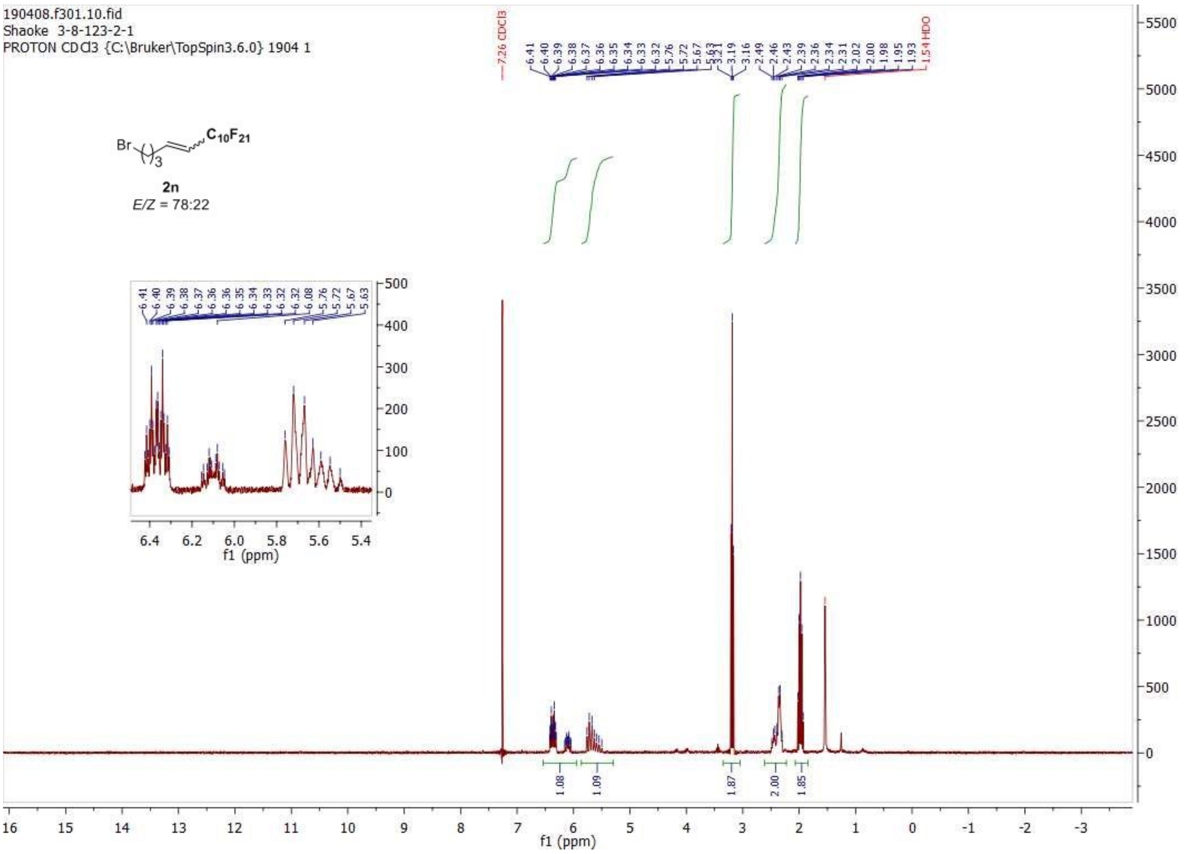

191002.f305.11.fid
Shaoke 3-8-175-4
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 5
148.27
148.15
148.04
114.94
114.64
114.33
77.00 CDCl3
40.22
31.69
25.87
25.65
$C_{10}F_{21}$
2l
f1 (ppm)

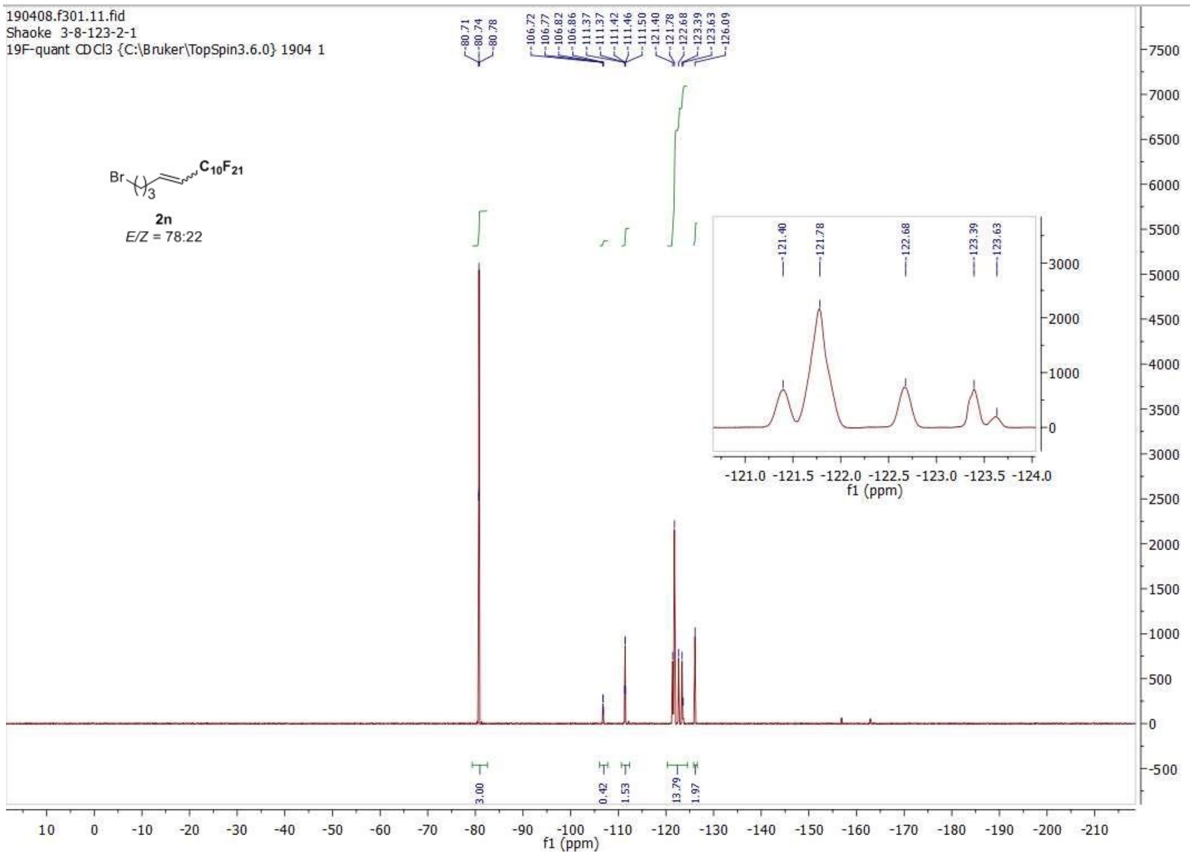

191002.f305.12.fid
Shaoke 3-8-175-4
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 5
-81.05
-81.08
-81.12
-111.36
-111.40
-111.45
-111.49
-121.55
-122.01
-122.91
-123.78
-126.37
$C_{10}F_{21}$
2l
3.00
1.80
13.90
2.00
f1 (ppm)

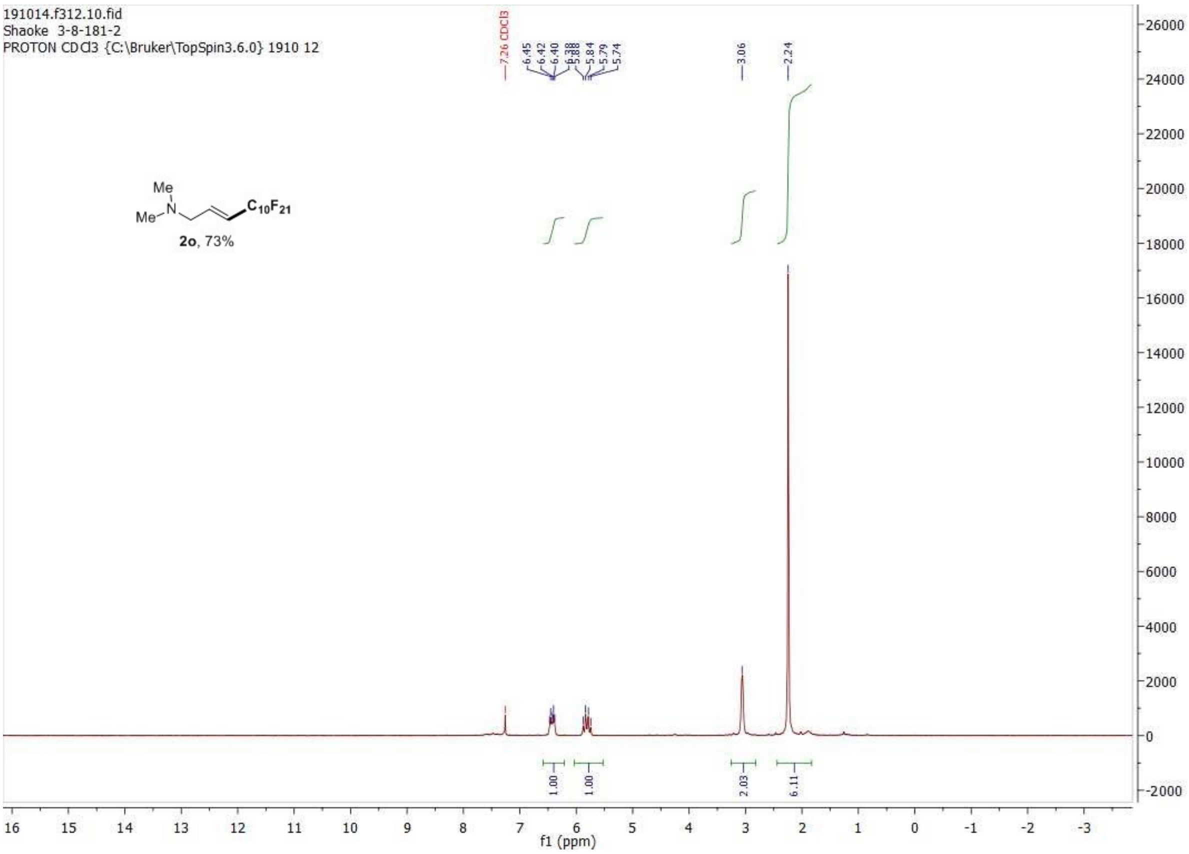

190221.f315.10.fid
Shaoke 3-8-103-1-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 15
7.34
7.34
7.31
7.29
7.26 CDCl3
7.25
7.22
7.20
7.19
7.17
7.16
6.50
6.48
6.45
6.42
6.40
6.39
5.69
5.65
5.60
5.56
2.81
2.79
2.76
2.74
2.57
2.56
2.55
2.52
2.52
2.51
1.52 HDO
C10F21
2
2m
E/Z = 83:17
6.51
6.50
6.48
6.45
6.42
6.40
6.39
6.20
6.18
6.17
6.16
6.14
6.13
6.12
6.11
5.92
5.87
5.69
5.65
5.60
5.56
5.50
5.45
6.4
6.2
6.0
5.8
5.6
5.4
f1 (ppm)
2500
2000
1500
1000
500
0
5.00
0.99
1.03
1.99
1.92
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

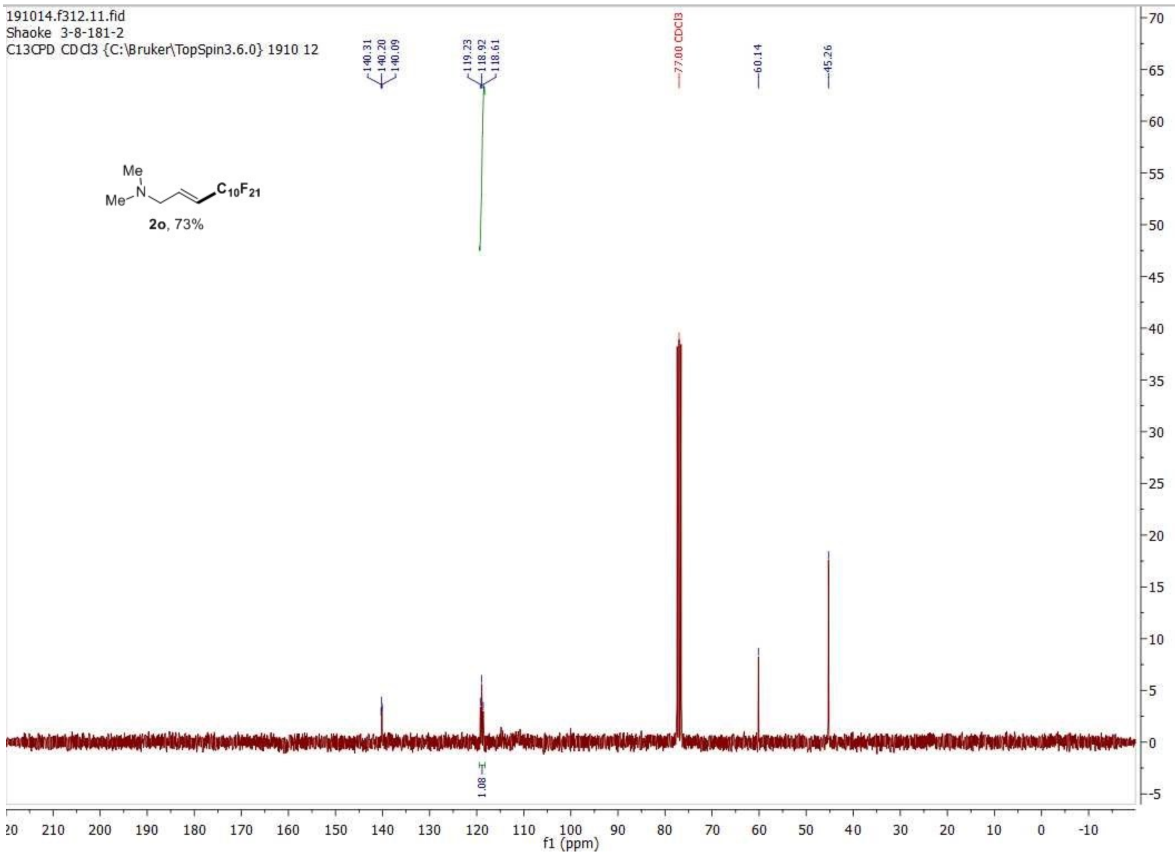

190221.f315.11.fid
Shaoke 3-8-103-1-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 15
142.19
142.07
141.95
140.36
128.52
128.35
126.29
117.92
117.62
117.32
77.00 CDCl3
34.40
33.74
C10F21
2
2m
E/Z = 83:17
142.19
142.07
141.95
10
5
0
142.5
142.0
141.5
f1 (ppm)
117.92
117.62
117.32
12
10
8
6
4
2
0
118.0
117.5
117.0
f1 (ppm)
1.00
0.84
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5

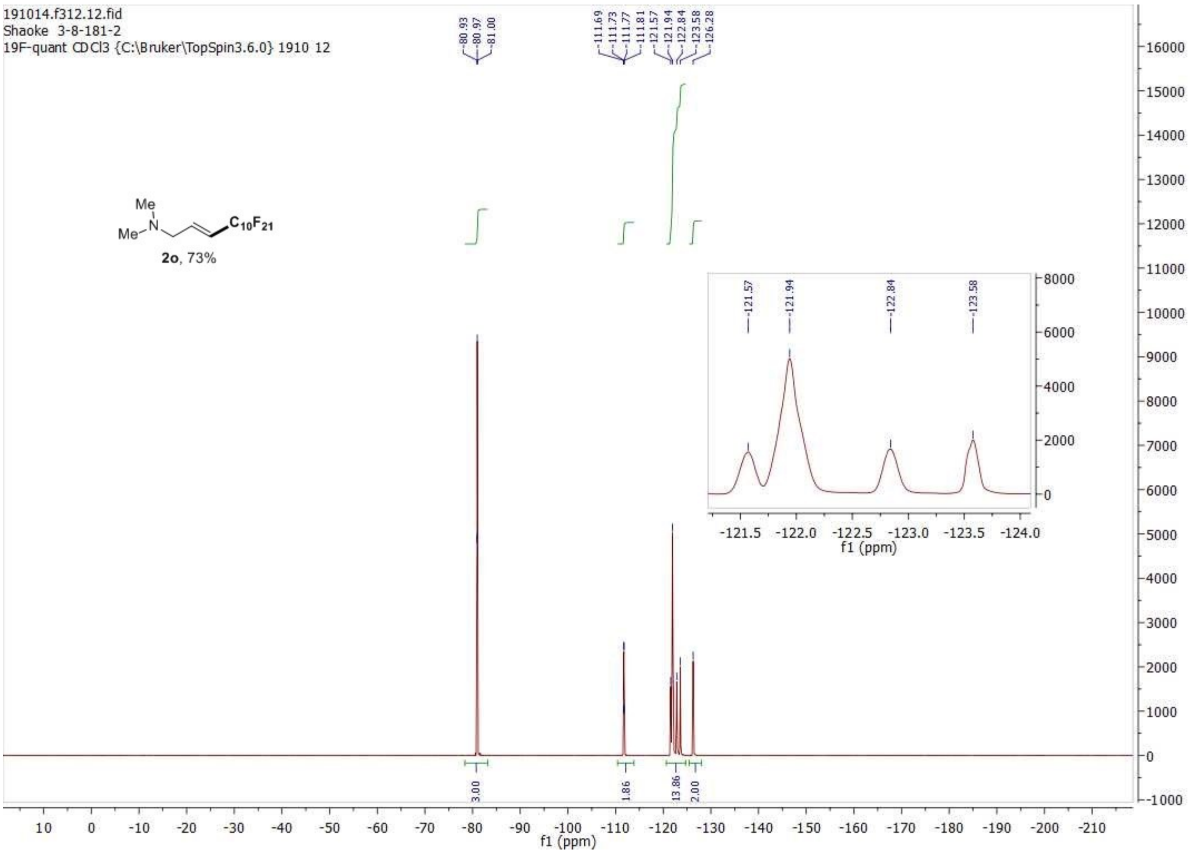

190221.f315.12.fid
Shaoke 3-8-103-1-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 15
-80.95
-80.98
-81.02
-106.91
-106.96
-107.01
-107.05
-111.40
-111.45
-111.49
-111.53
-121.55
-121.94
-122.84
-123.61
-123.83
-126.27
C10F21
2
2m
E/Z = 83:17
3.00
0.33
1.64
13.98
2.01
f1 (ppm)
f1 (ppm)

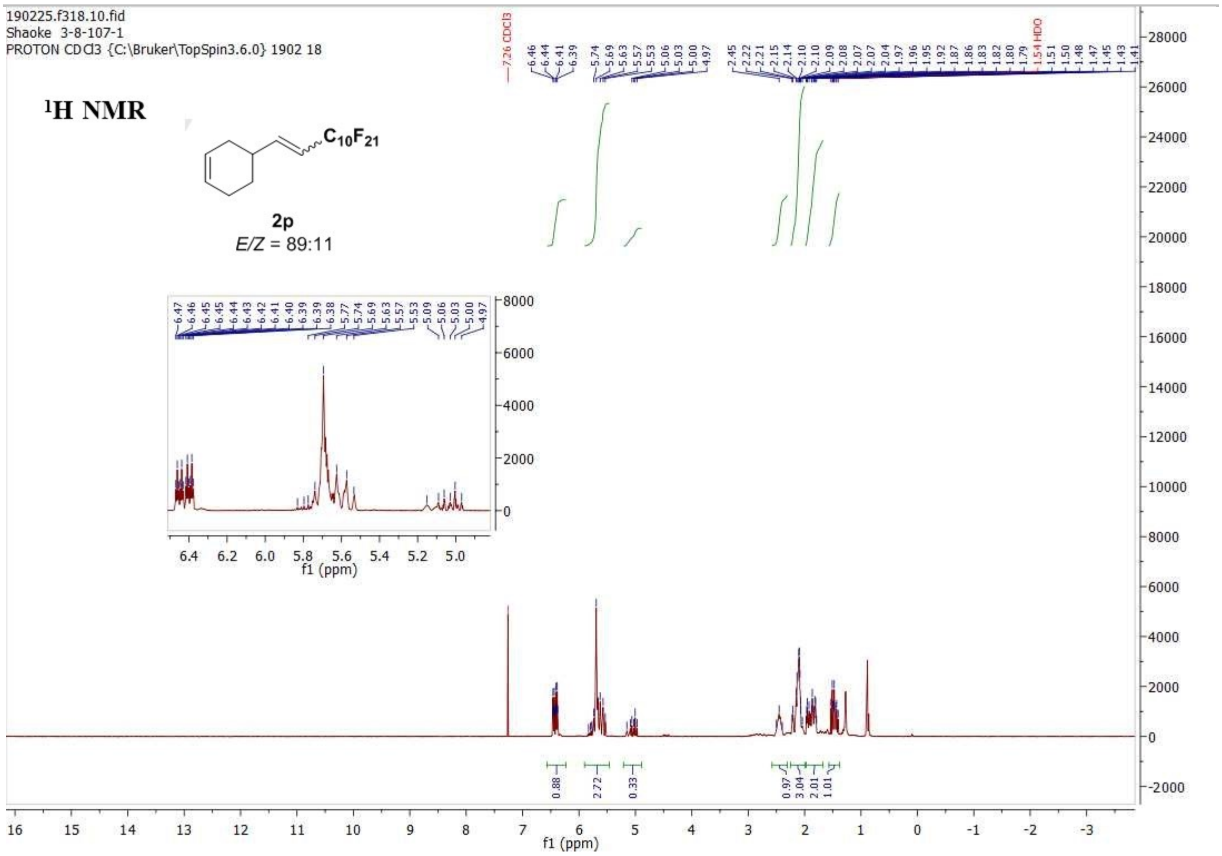

190408.f301.10.fid
Shaoke 3-8-123-2-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1904 1
7.26 CDCl3
1.54 HDO
Br
3
C10F21
2n
E/Z = 78:22
1.08
1.09
1.87
2.00
1.85
f1 (ppm)
f1 (ppm)

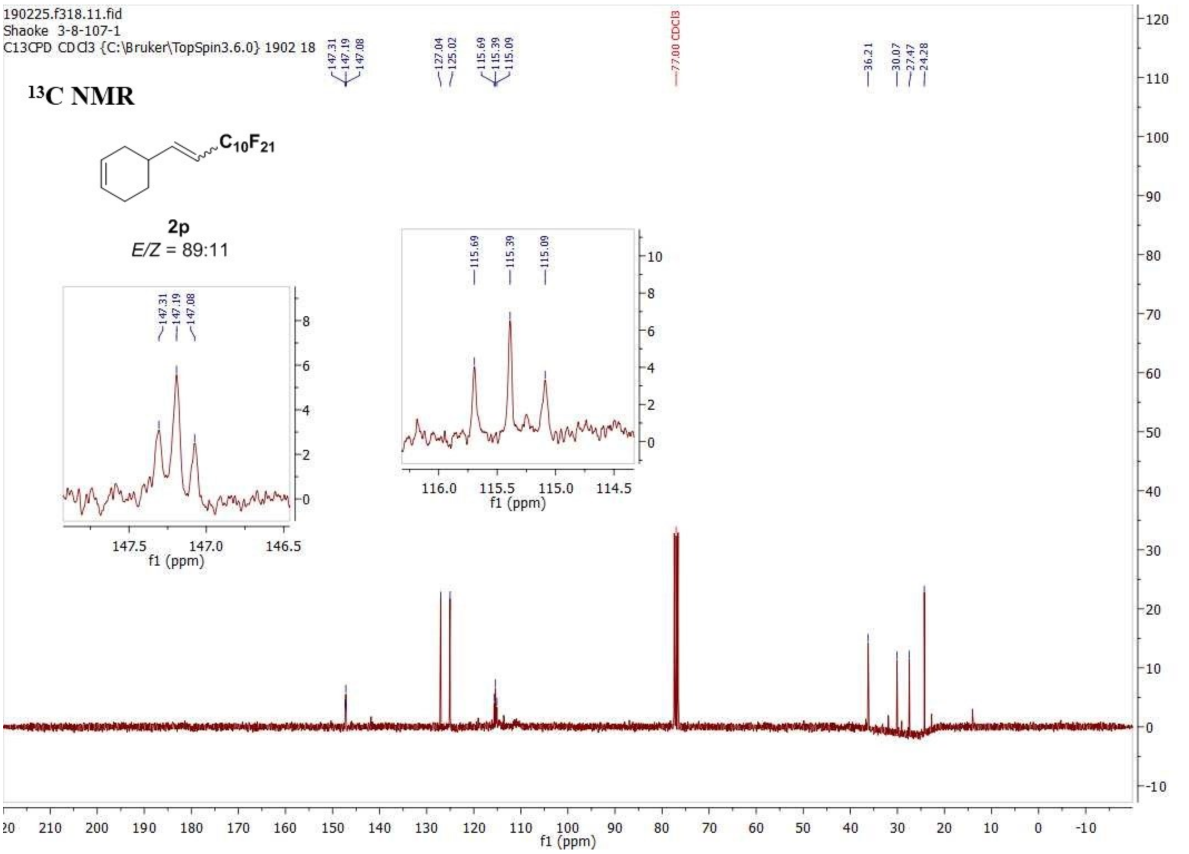

190408.f301.11.fid
Shaoke 3-8-123-2-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1904 1
-80.71
-80.74
-80.78
-106.72
-106.77
-106.82
-106.86
-111.37
-111.37
-111.42
-111.46
-111.50
-121.40
-121.78
-122.68
-123.39
-123.63
-126.09
Br
3
C10F21
2n
E/Z = 78:22
3.00
0.42
1.53
13.79
1.97
-121.0 -121.5 -122.0 -122.5 -123.0 -123.5 -124.0
f1 (ppm)
f1 (ppm)

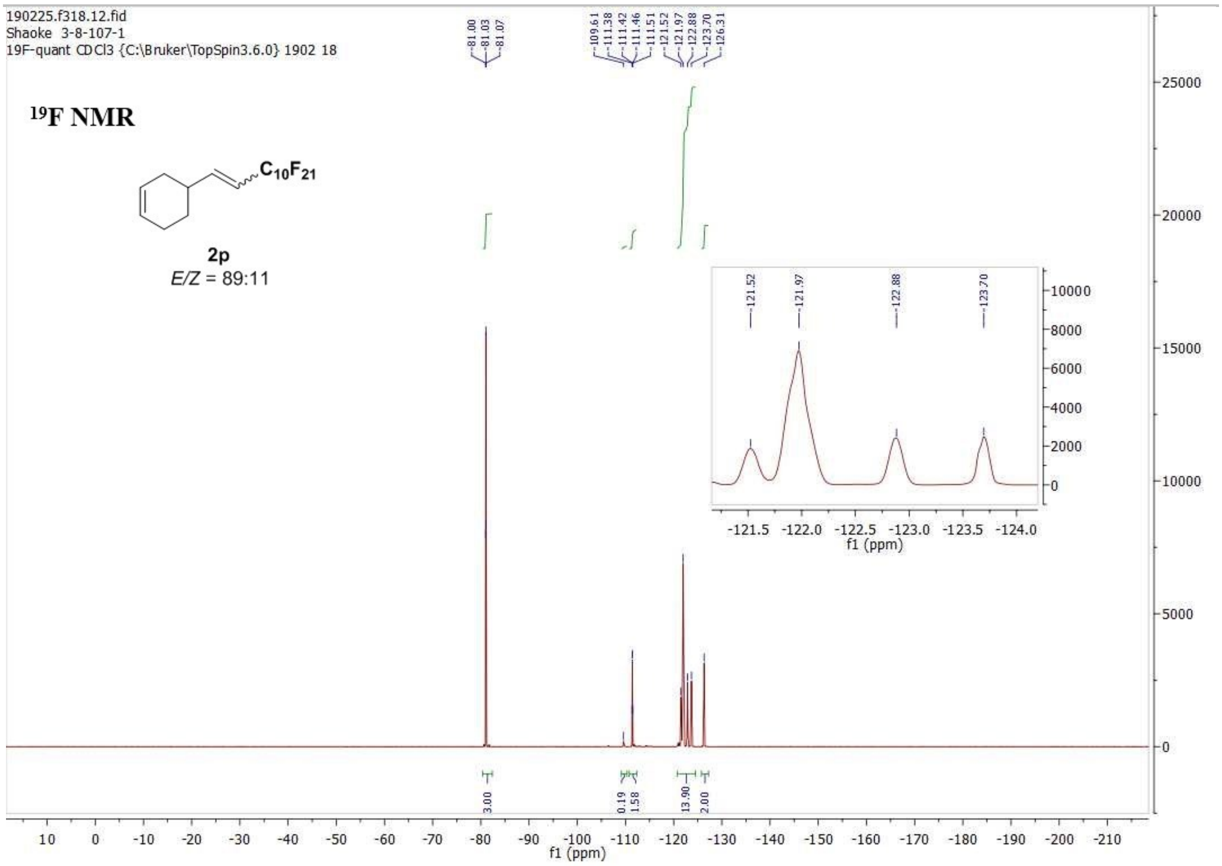

191014.f312.10.fid
Shaoke 3-8-181-2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 12
7.26 CDCl3
6.45
6.42
6.40
5.88
5.88
5.84
5.79
5.74
3.06
2.24
Me
Me
N
C10F21
2o, 73%
1.00
1.00
2.03
6.11
f1 (ppm)

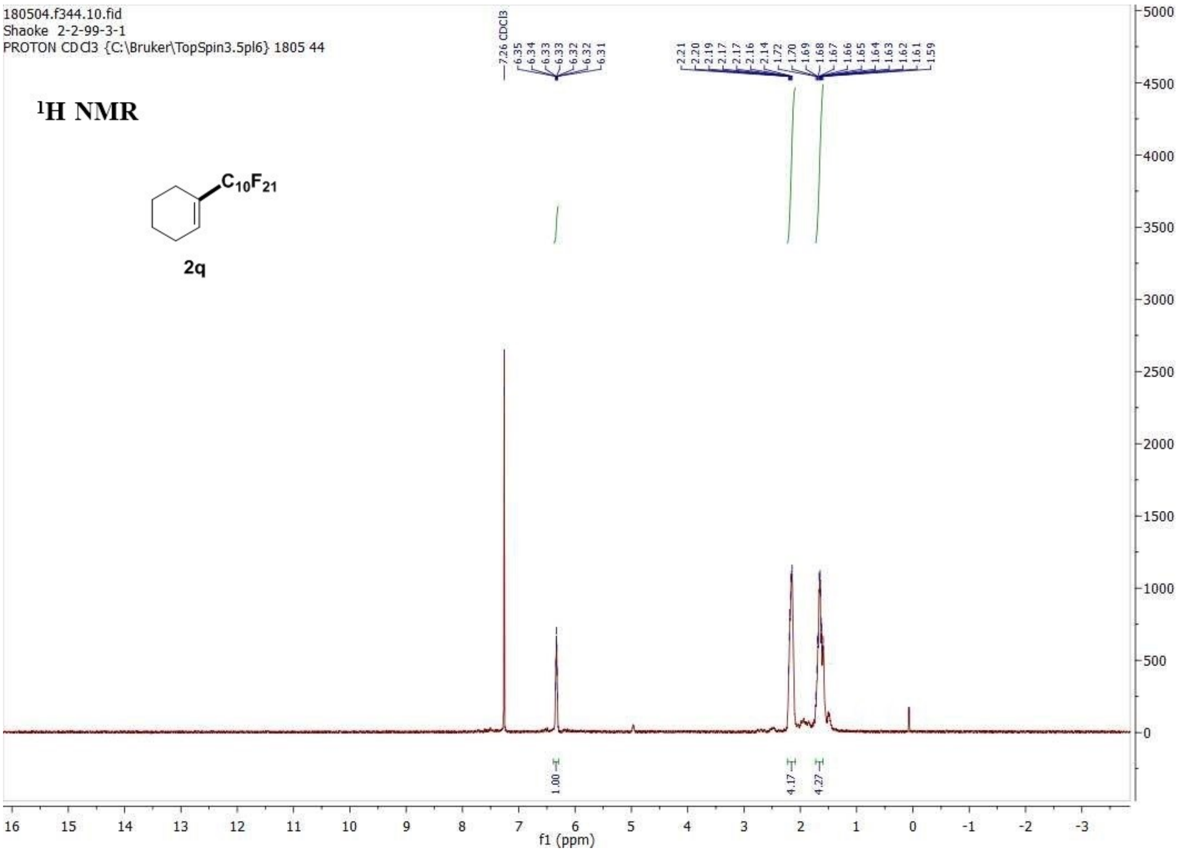

191014.f312.11.fid
Shaoke 3-8-181-2
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 12
140.31
140.20
140.09
119.23
118.92
118.61
77.00 CDCl3
60.14
45.26
Me
N
Me
$C_{10}F_{21}$
2o, 73%
1.08
f1 (ppm)

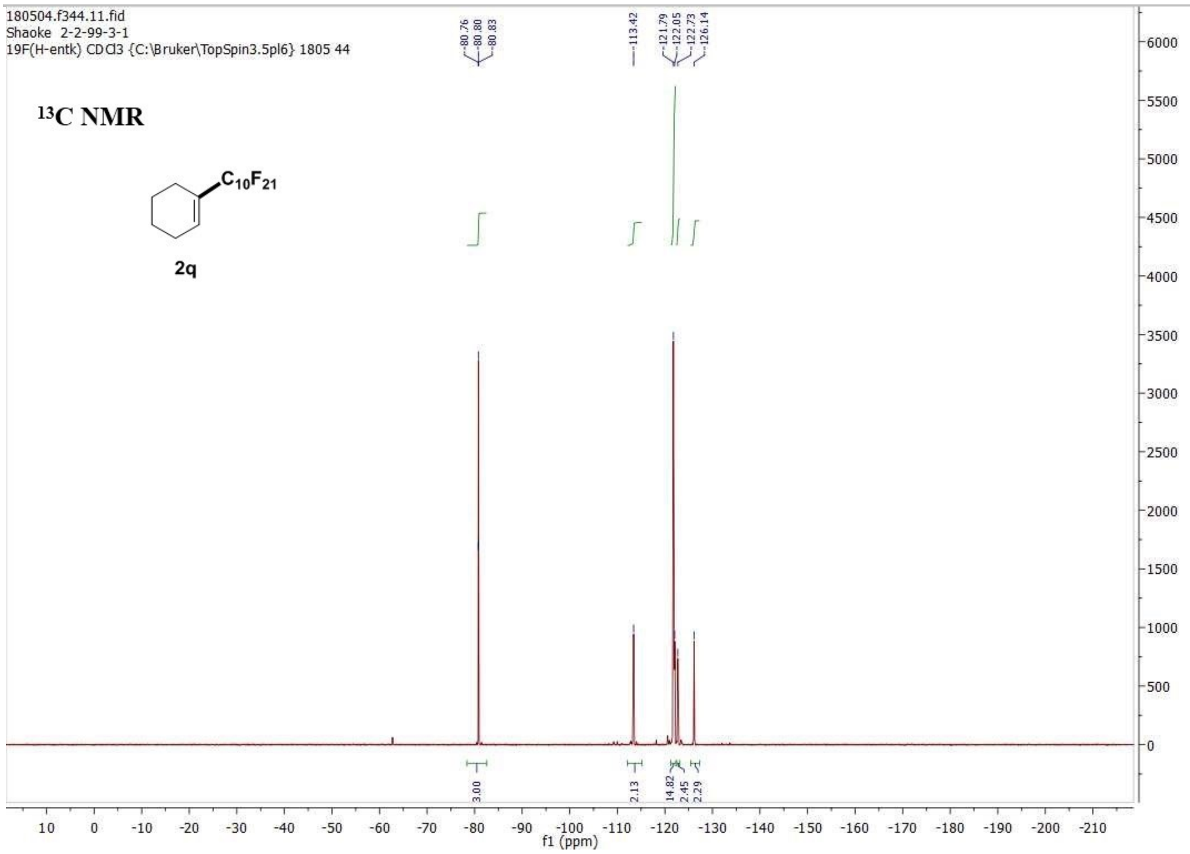

191014.f312.12.fid
Shaoke 3-8-181-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 12
-80.93
-80.97
-81.00
-111.69
-111.73
-111.77
-111.81
-121.57
-121.94
-122.84
-123.58
-126.28
Me
N
Me
$C_{10}F_{21}$
2o, 73%
3.00
1.86
13.86
2.00
f1 (ppm)

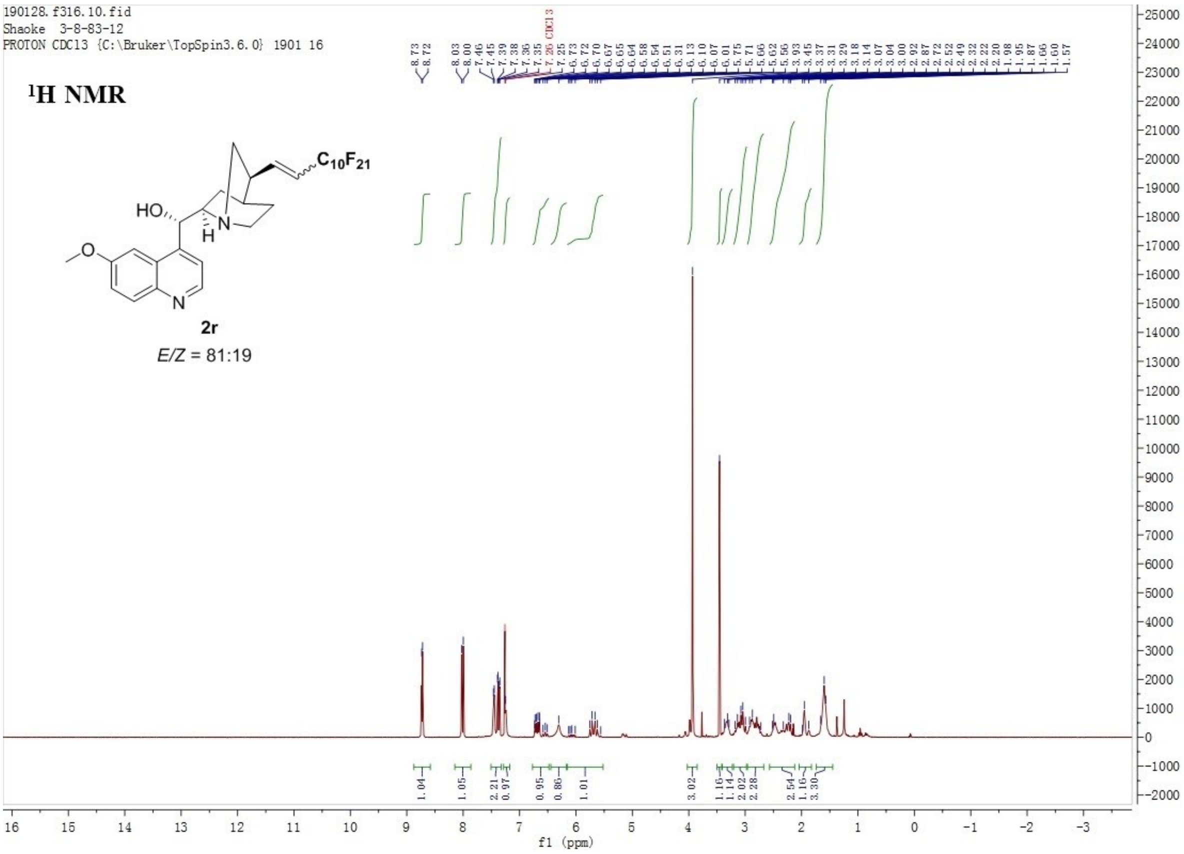

190225.f318.10.fid
Shaoke 3-8-107-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 18
1H NMR
C10F21
2p
E/Z = 89:11
7.26 CDCl3
6.46
6.44
6.41
6.39
5.74
5.69
5.63
5.57
5.53
5.06
5.03
5.00
4.97
2.45
2.22
2.21
2.15
2.14
2.10
2.10
2.09
2.08
2.07
2.07
2.04
1.97
1.96
1.95
1.92
1.87
1.86
1.83
1.82
1.80
1.79
1.54 HDO
1.51
1.50
1.48
1.47
1.45
1.43
1.41
0.88
2.72
0.33
0.97
3.04
2.01
1.01
f1 (ppm)

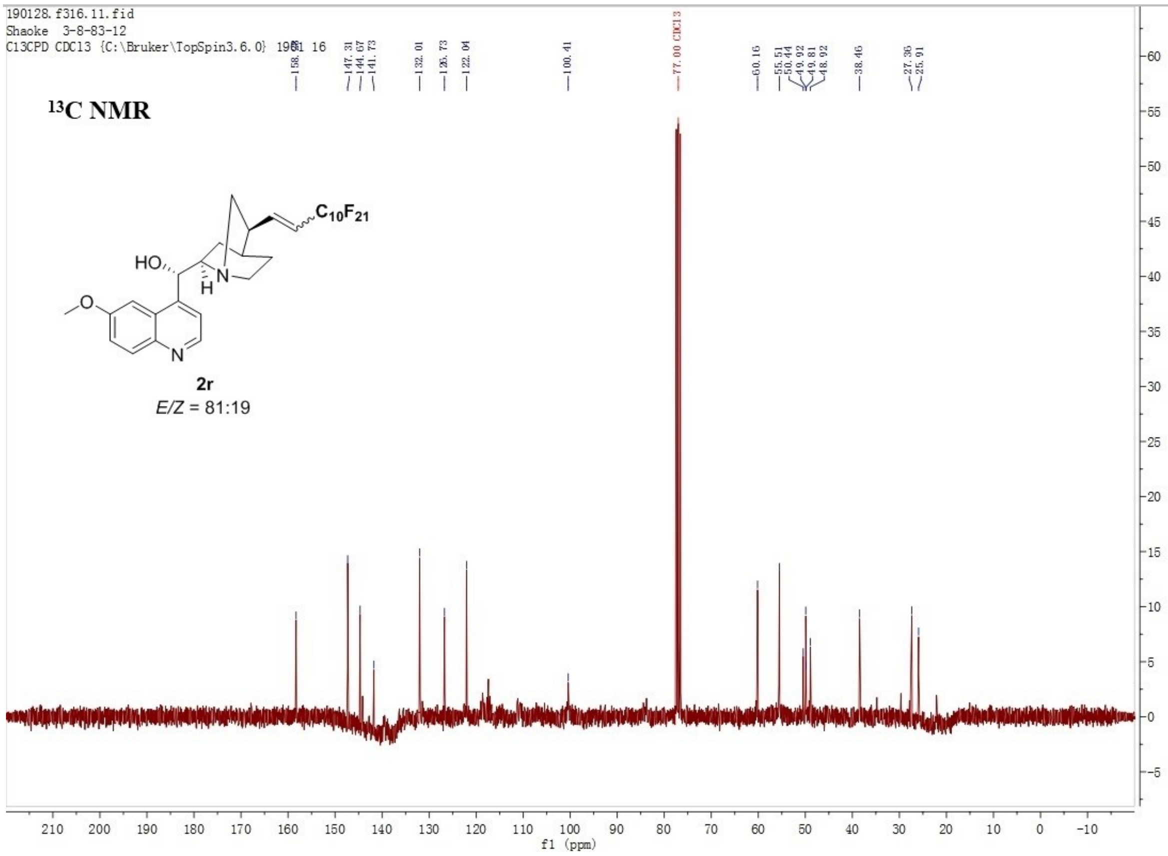

190225.f318.11.fid
Shaoke 3-8-107-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 18
13C NMR
C10F21
2p
E/Z = 89:11
147.31
147.19
147.08
127.04
125.02
115.69
115.39
115.09
77.00 CDCl3
36.21
30.07
27.47
24.28
f1 (ppm)

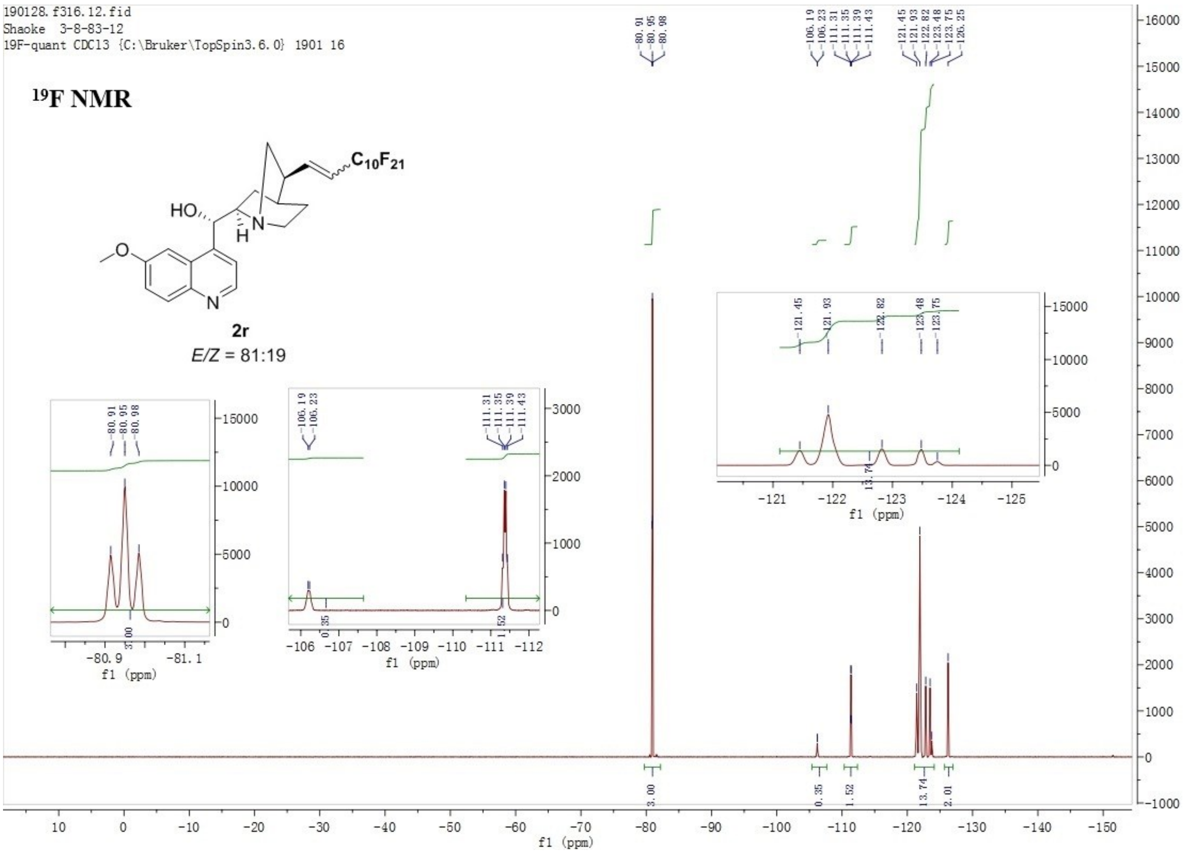

190225.f318.12.fid
Shaoke 3-8-107-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1902 18
19F NMR
C10F21
2p
E/Z = 89:11
-81.00
-81.03
-81.07
-109.61
-111.38
-111.42
-111.46
-111.51
-121.52
-121.97
-122.88
-123.70
-126.31
3.00
0.19
1.58
13.90
2.00
f1 (ppm)
-121.5 -122.0 -122.5 -123.0 -123.5 -124.0
25000 20000 15000 10000 5000 0

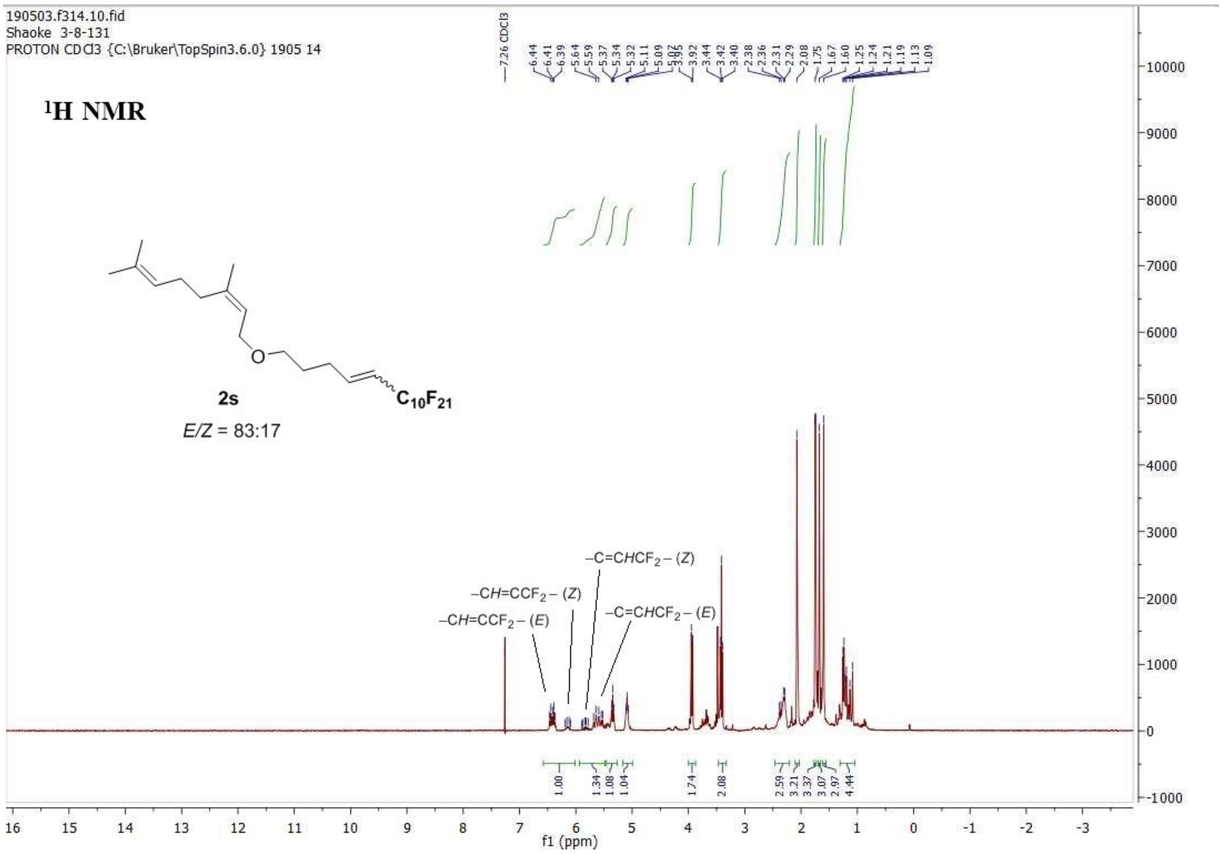

180504.f344.10.fid
Shaoke 2-2-99-3-1
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1805 44
1H NMR
C10F21
2q
7.26 CDCl3
6.35
6.34
6.33
6.33
6.32
6.32
6.31
2.21
2.20
2.19
2.17
2.17
2.16
2.14
1.72
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.61
1.59
1.00
4.17
4.27
f1 (ppm)
5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0

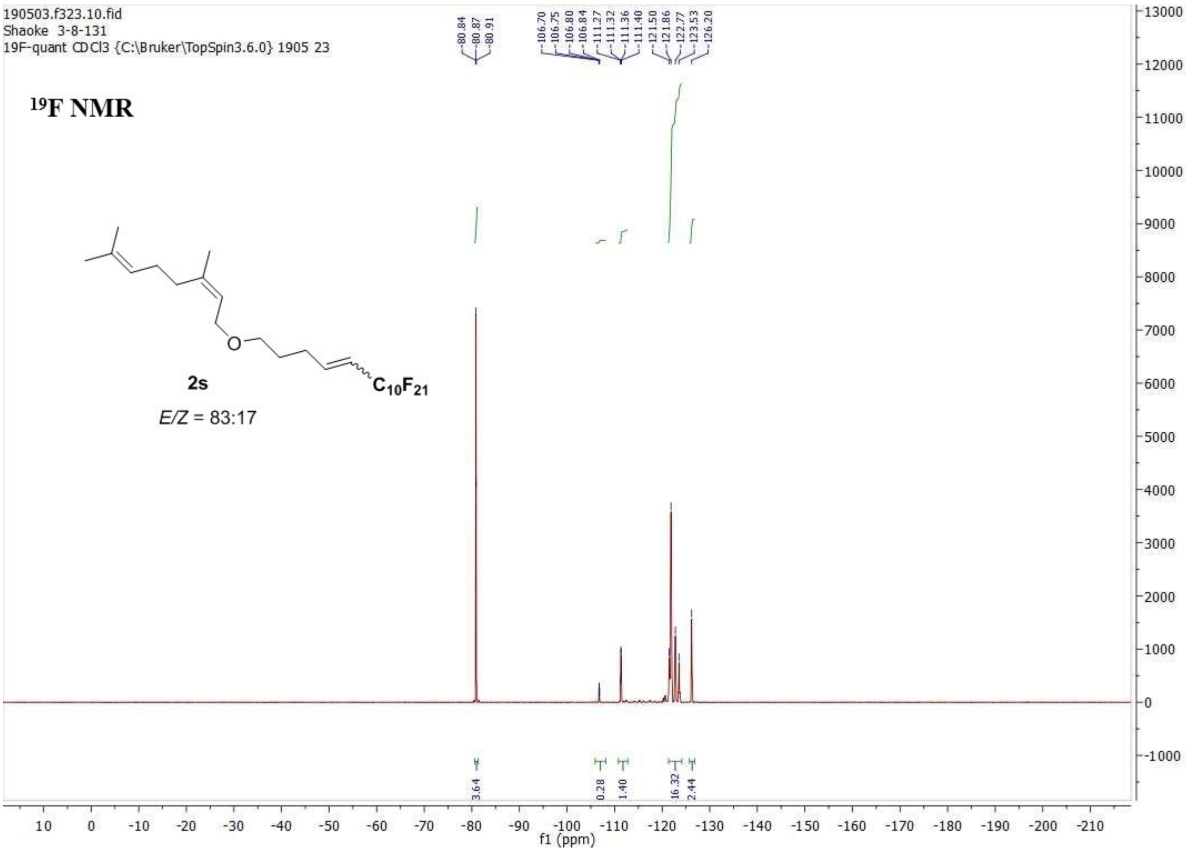

180504.f344.11.fid
Shaoke 2-2-99-3-1
19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.5pl6} 1805 44
13C NMR
C10F21
2q
-80.76
-80.80
-80.83
-113.42
-121.79
-122.05
-122.73
-126.14
3.00
2.13
14.82
2.45
2.29
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)
6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0


190128.f316.10.fid
Shaoke 3-8-83-12
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 16
1H NMR
HO
H
N
O
C10F21
2r
E/Z = 81:19
8.73
8.72
8.03
8.00
7.46
7.45
7.39
7.38
7.36
7.35
7.26 CDCl3
7.25
6.73
6.72
6.70
6.67
6.65
6.64
6.58
6.54
6.51
6.31
6.13
6.10
6.07
6.01
5.75
5.71
5.66
5.62
5.56
3.93
3.45
3.37
3.31
3.29
3.18
3.14
3.07
3.04
3.00
2.92
2.87
2.72
2.52
2.49
2.32
2.22
2.20
1.98
1.95
1.87
1.66
1.60
1.57
1.04
1.05
2.21
0.97
0.95
0.86
1.01
3.02
1.16
1.14
2.02
2.28
2.54
1.16
3.30
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)
25000 24000 23000 22000 21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000

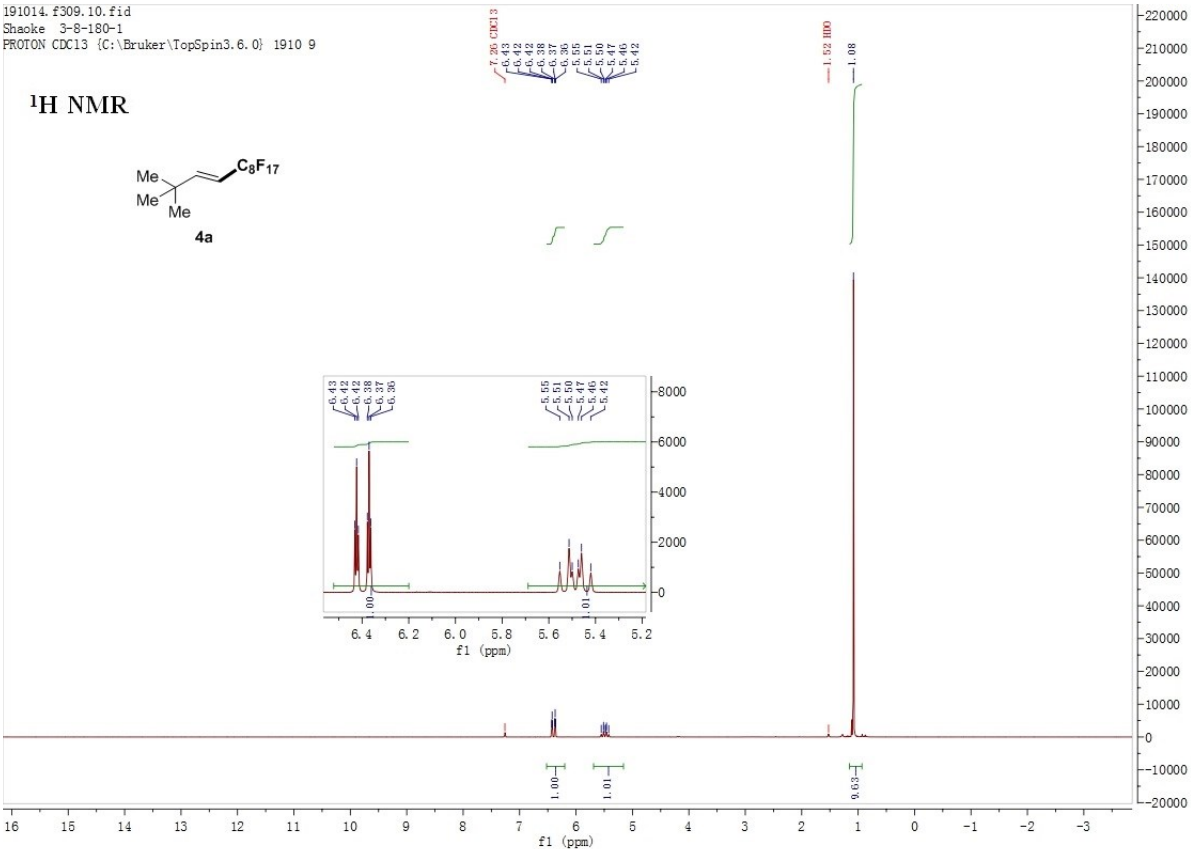

190128.f316.11.fid
Shaoke 3-8-83-12
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 16
158.
147.31
144.67
141.73
132.01
125.73
122.04
100.41
77.00 CDCl3
60.16
55.51
50.44
49.92
49.81
48.92
38.46
27.36
25.91
13C NMR
HO
H
N
O
N
C10F21
2r
E/Z = 81:19
f1 (ppm)

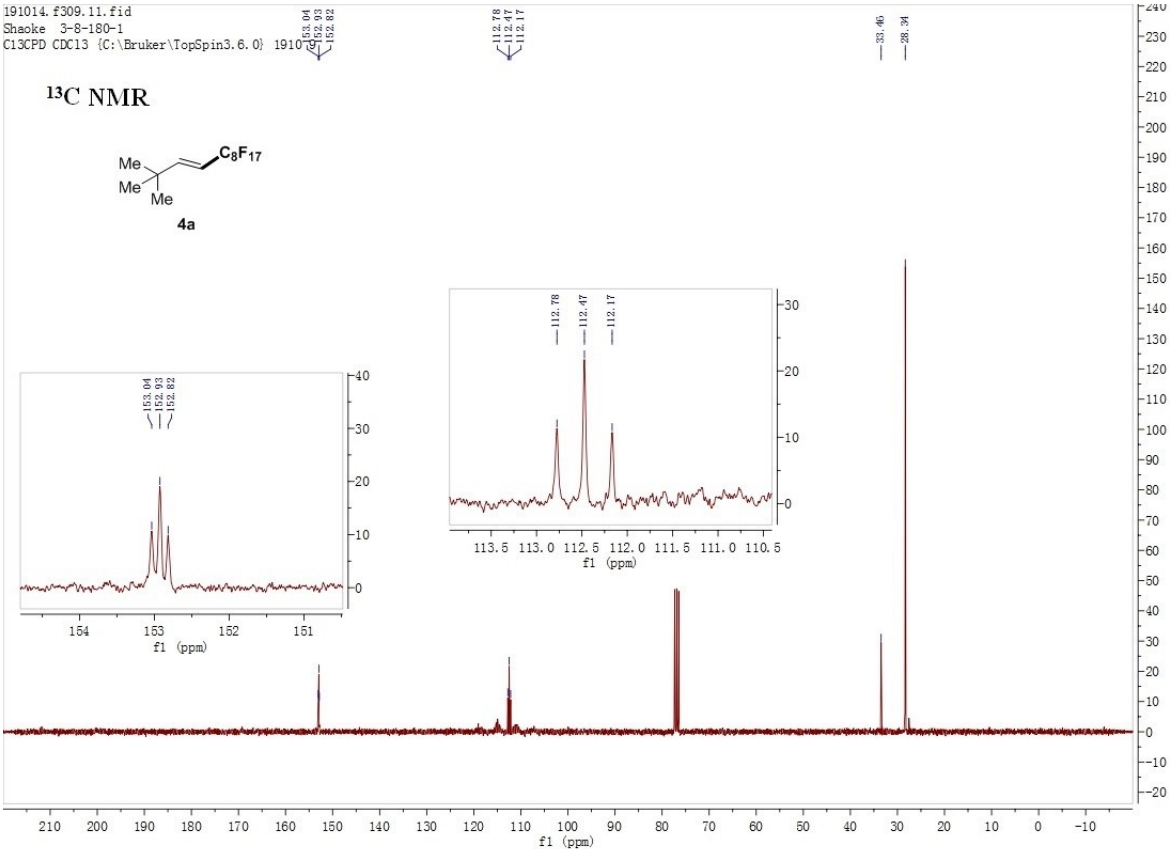

190128.f316.12.fid
Shaoke 3-8-83-12
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1901 16
-80.91
-80.95
-80.98
-106.19
-106.23
-111.31
-111.35
-111.39
-111.43
-121.45
-121.93
-122.82
-123.48
-123.75
-126.25
19F NMR
HO
H
N
O
N
C10F21
2r
E/Z = 81:19
3.00
0.35
1.52
13.74
2.01
f1 (ppm)

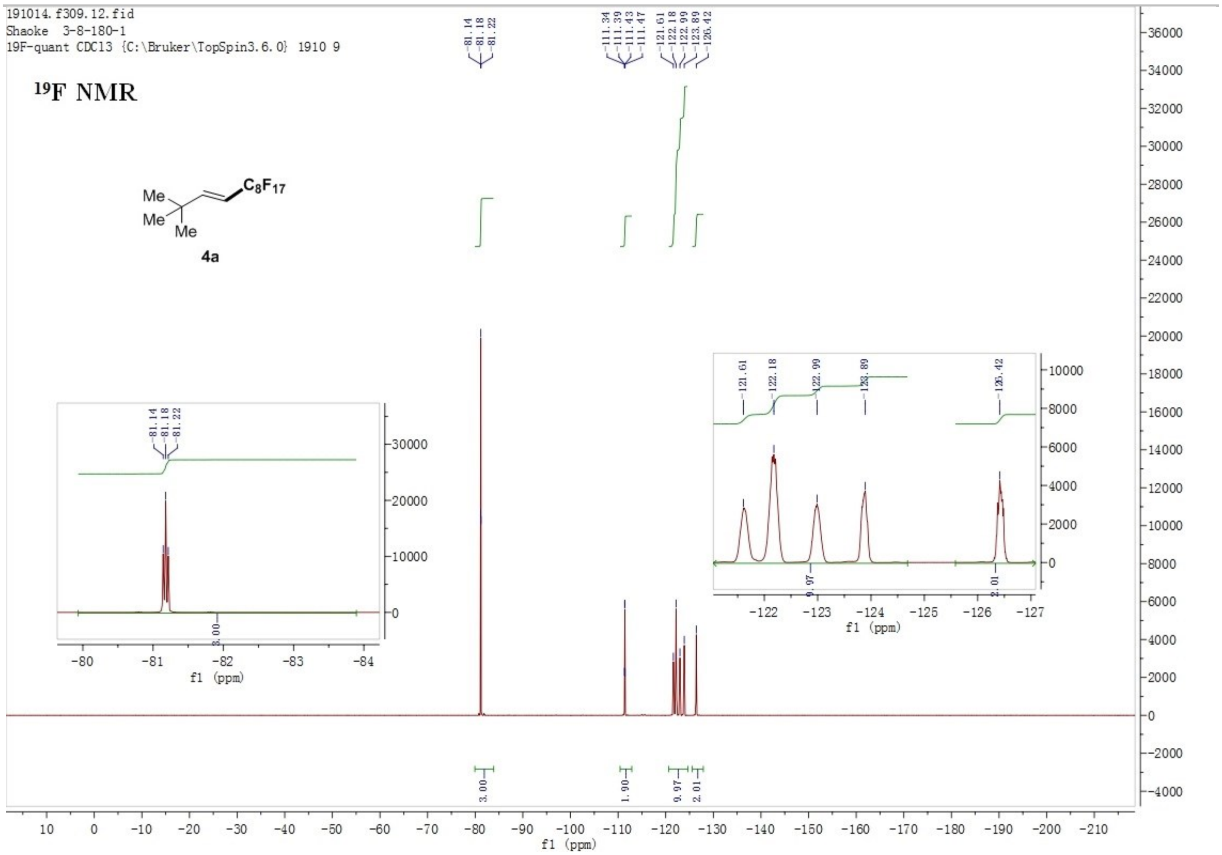

190503.f314.10.fid
Shaoke 3-8-131
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1905 14
7.26 CDCl3
6.44
6.41
6.39
5.64
5.59
5.37
5.34
5.32
5.11
5.09
5.07
3.95
3.92
3.44
3.42
3.40
2.38
2.36
2.31
2.29
2.08
1.75
1.67
1.60
1.25
1.24
1.21
1.19
1.13
1.09
¹H NMR
O
$C_{10}F_{21}$
2s
E/Z = 83:17
–CH=CCF₂– (E)
–CH=CCF₂– (Z)
–C=CHCF₂– (Z)
–C=CHCF₂– (E)
1.00
1.34
1.08
1.04
1.74
2.08
2.59
3.21
3.37
3.07
2.97
4.44
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

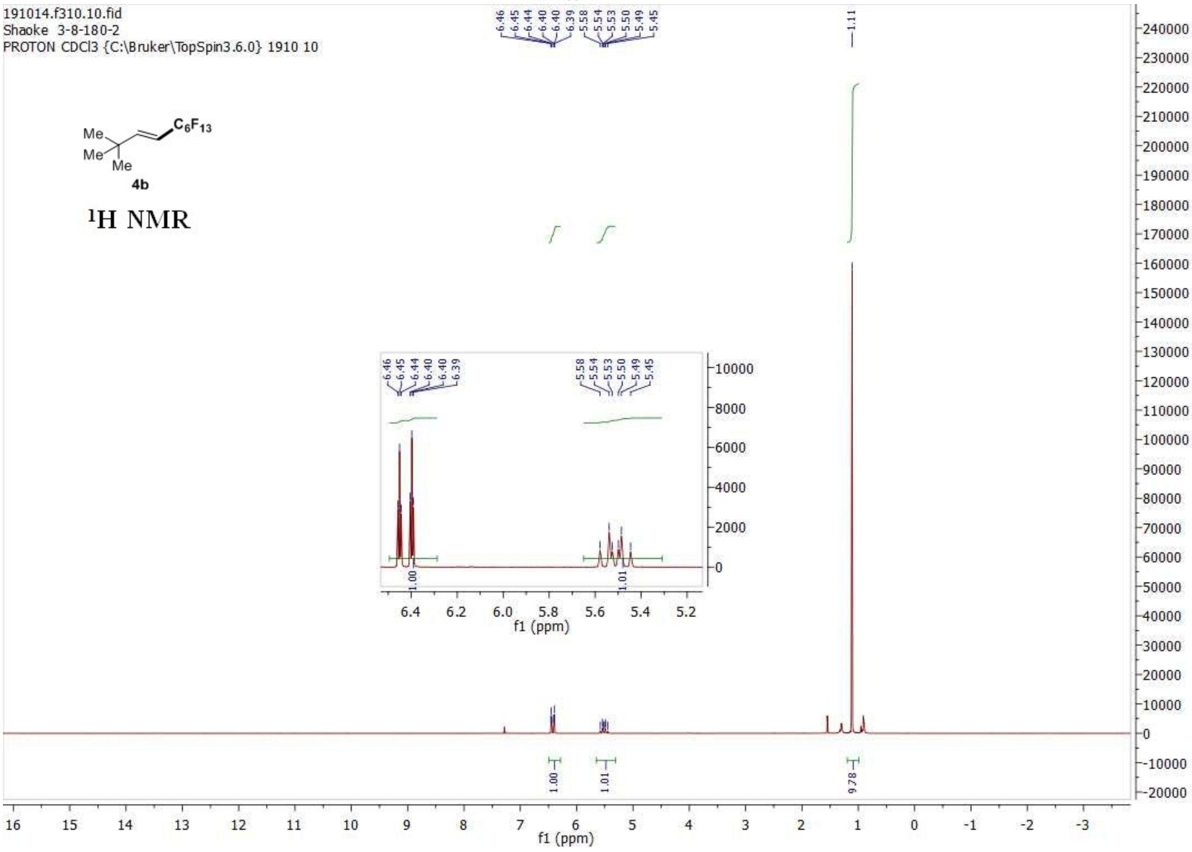

190503.f323.10.fid
Shaoke 3-8-131
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1905 23
-80.84
-80.87
-80.91
-106.70
-106.75
-106.80
-106.84
-111.27
-111.32
-111.36
-111.40
-121.50
-121.86
-122.77
-123.53
-126.20
¹⁹F NMR
O
$C_{10}F_{21}$
2s
E/Z = 83:17
3.64
0.28
1.40
16.32
2.44
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

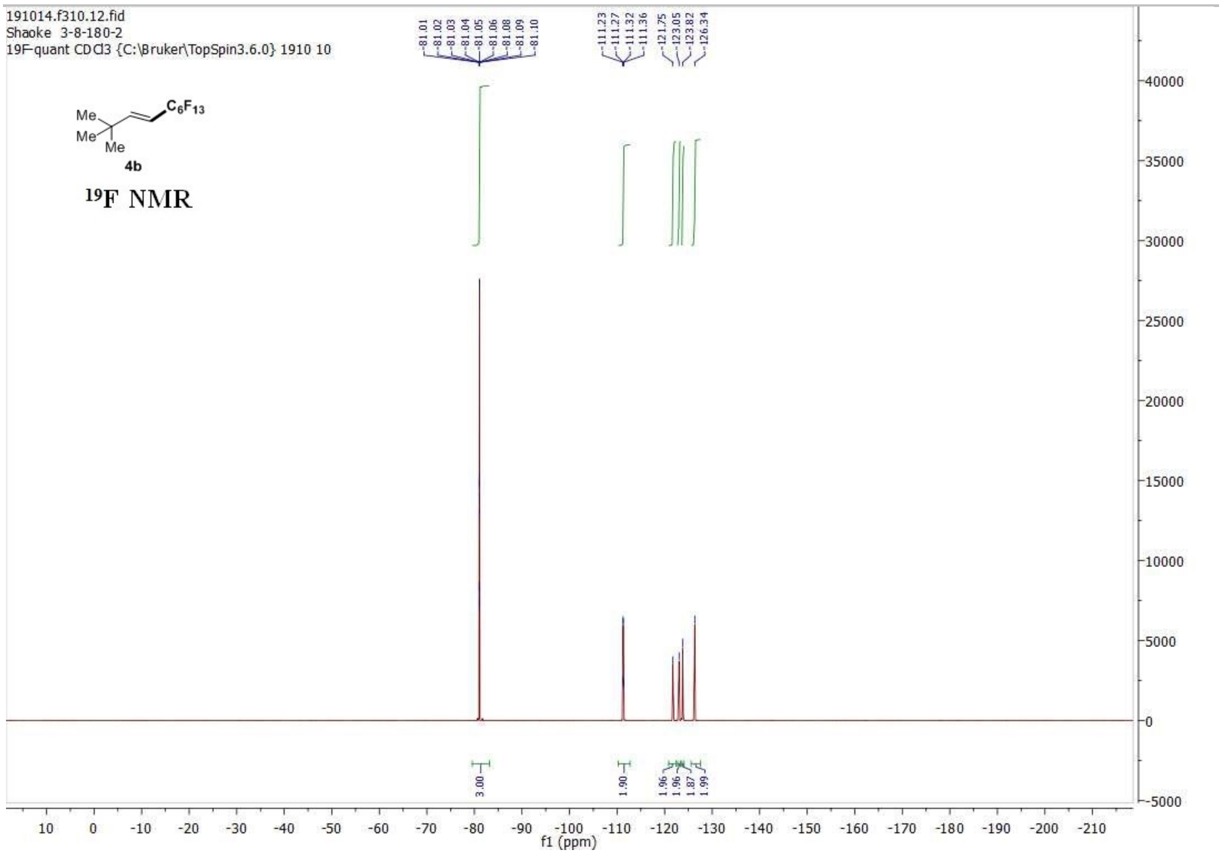
8.3 Spectra of other perfluoroalkylated alkenes

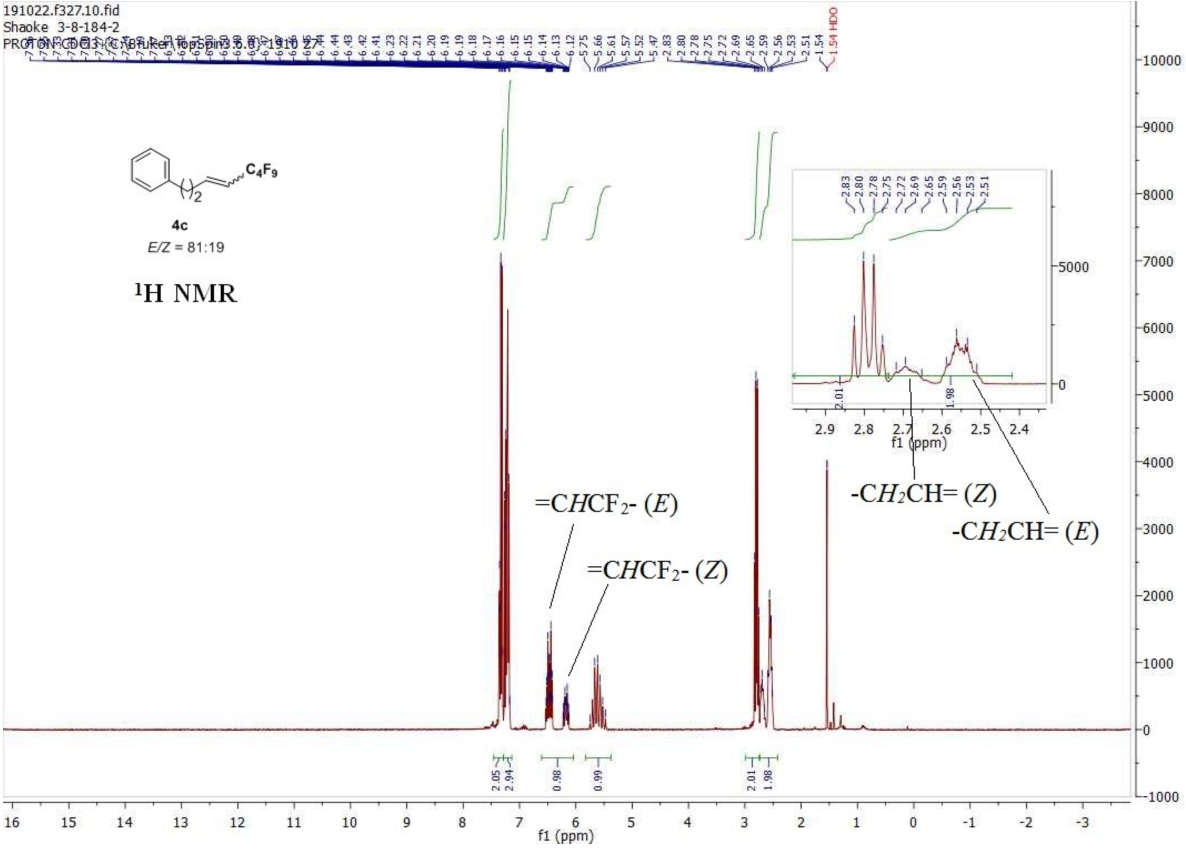


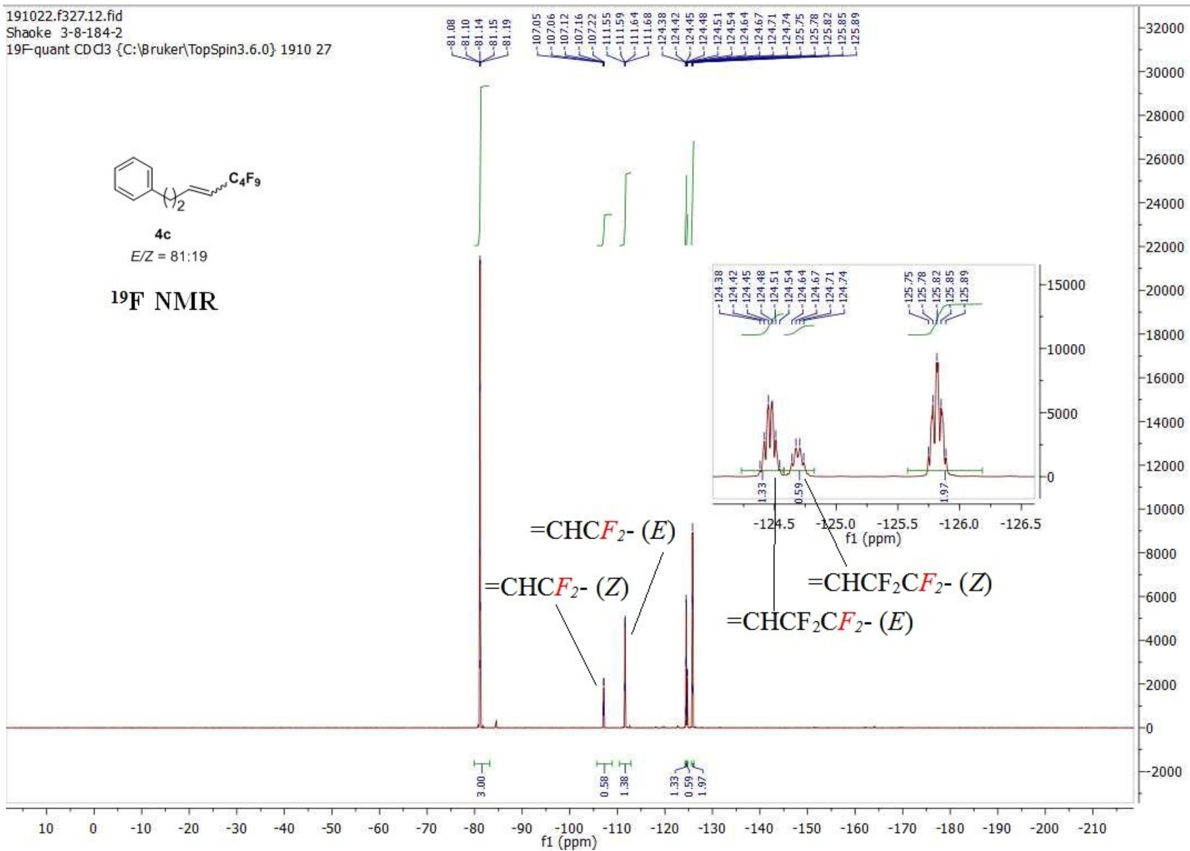


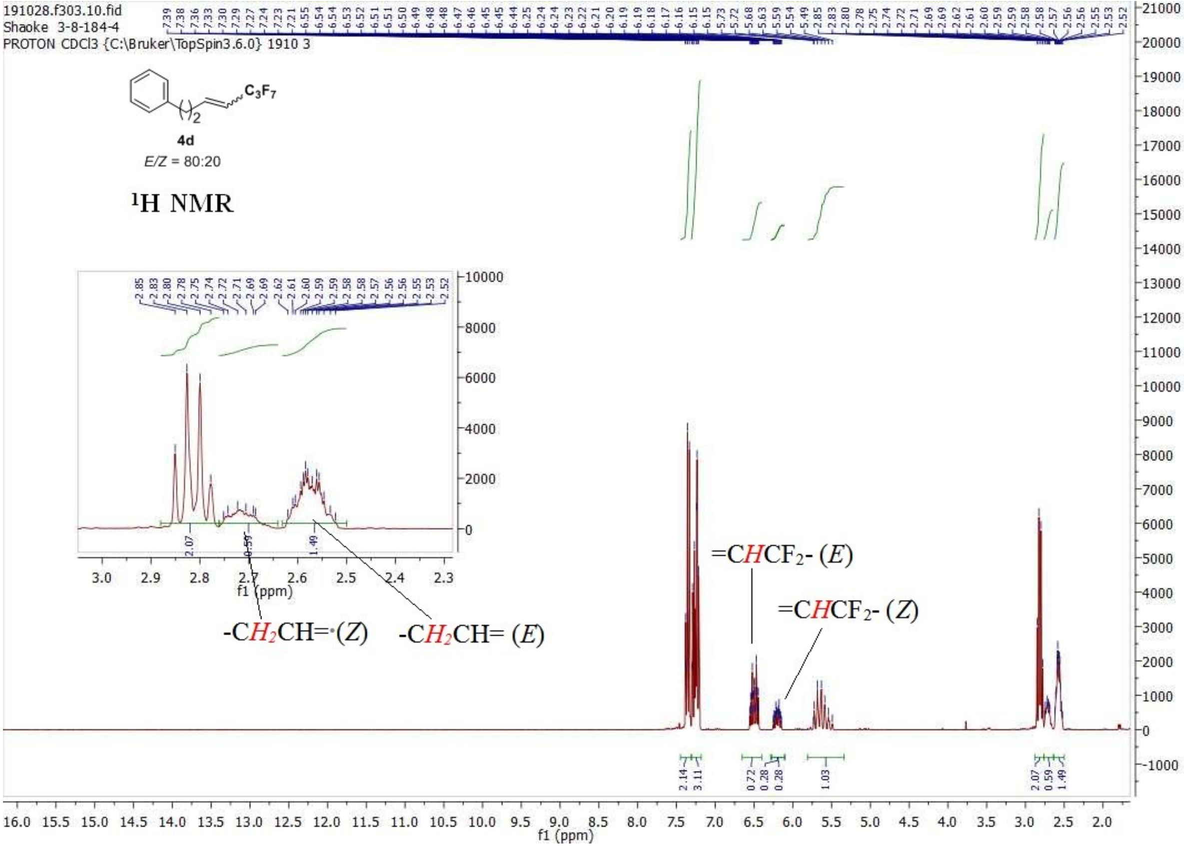

191014.f309.12.fid
Shaoke 3-8-180-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 9
19F NMR
Me
Me
Me
C8F17
4a
-81.14
-81.18
-81.22
-111.34
-111.39
-111.43
-111.47
-121.61
-122.18
-122.99
-123.89
-126.42
3.00
1.90
9.97
2.01
f1 (ppm)

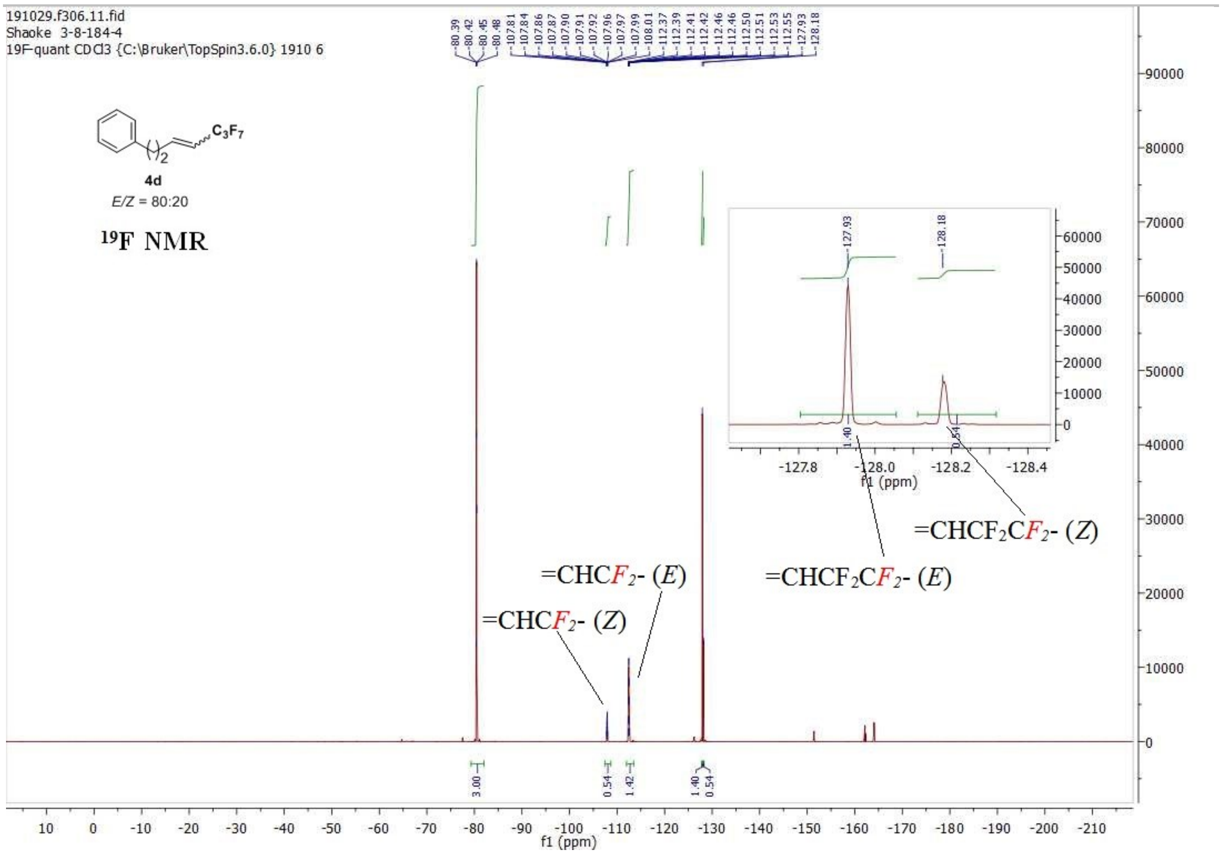

191014.f310.10.fid
Shaoke 3-8-180-2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 10
Me
Me
Me
C6F13
4b
1H NMR
6.46
6.45
6.44
6.40
6.40
6.39
5.58
5.54
5.53
5.50
5.49
5.45
1.11
1.00
1.01
9.78
f1 (ppm)

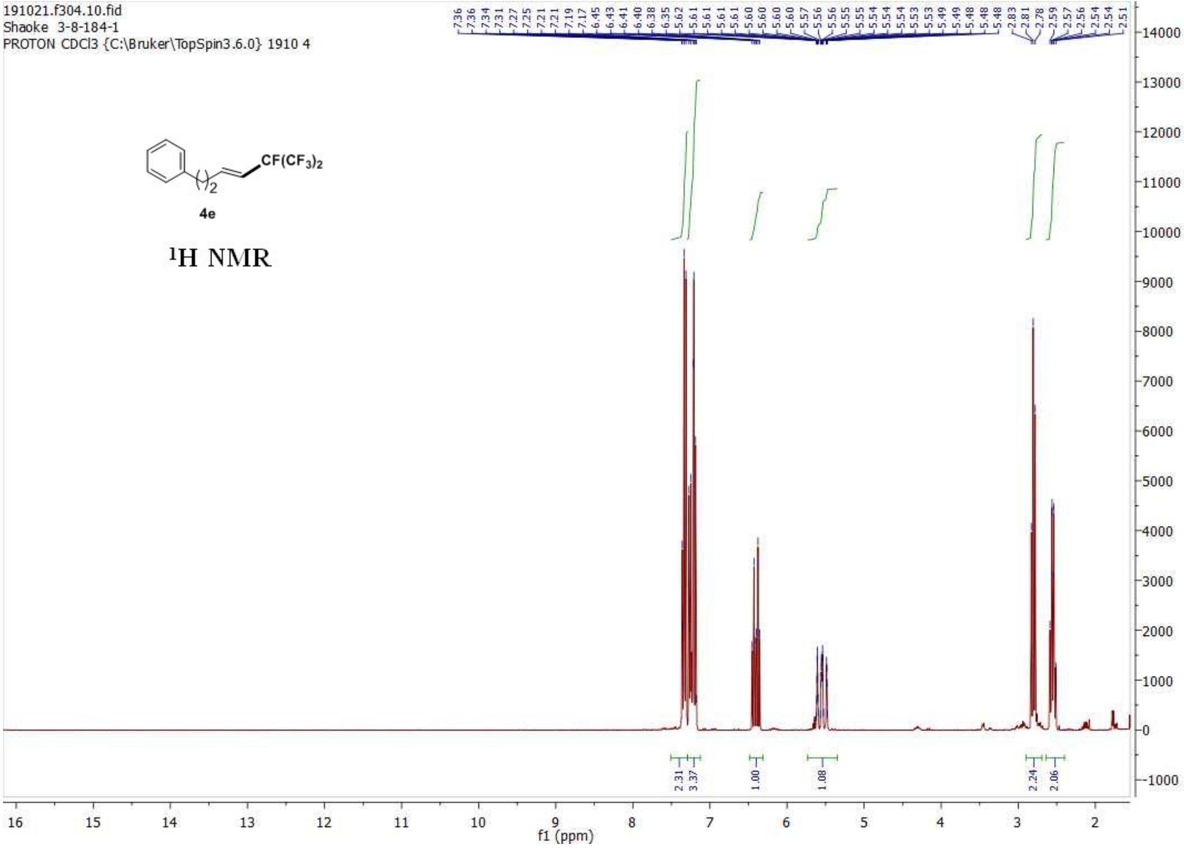

191014.f310.12.fid
Shaoke 3-8-180-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 10
-81.01
-81.02
-81.03
-81.04
-81.05
-81.06
-81.08
-81.09
-81.10
-111.23
-111.27
-111.32
-111.36
-121.75
-123.05
-123.82
-126.34
Me
Me
Me
C6F13
4b
19F NMR
3.00
1.90
1.96
1.96
1.87
1.99
f1 (ppm)
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000

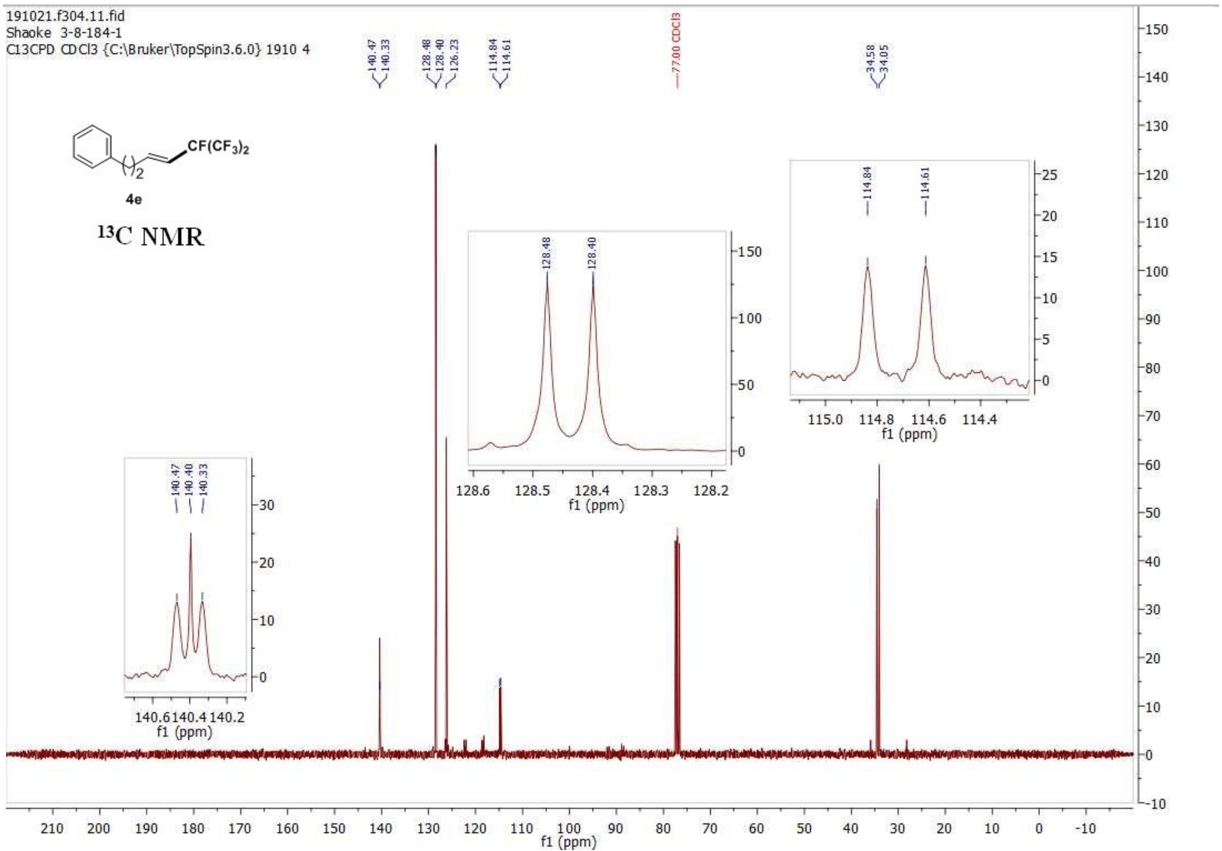

191022.f327.10.fid
Shaoke 3-8-184-2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 27
6.44
6.43
6.42
6.41
6.23
6.22
6.21
6.20
6.19
6.19
6.18
6.17
6.16
6.15
6.15
6.14
6.13
6.12
5.75
5.66
5.61
5.57
5.52
5.47
2.83
2.80
2.78
2.75
2.72
2.69
2.65
2.59
2.56
2.53
2.51
1.54
1.54 HDO
C4F9
2
4c
E/Z = 81:19
1H NMR
=CHCF2- (E)
=CHCF2- (Z)
-CH2CH= (Z)
-CH2CH= (E)
2.01
1.98
2.9
2.8
2.7
2.6
2.5
2.4
f1 (ppm)
5000
0
2.05
2.94
0.98
0.99
2.01
1.98
f1 (ppm)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

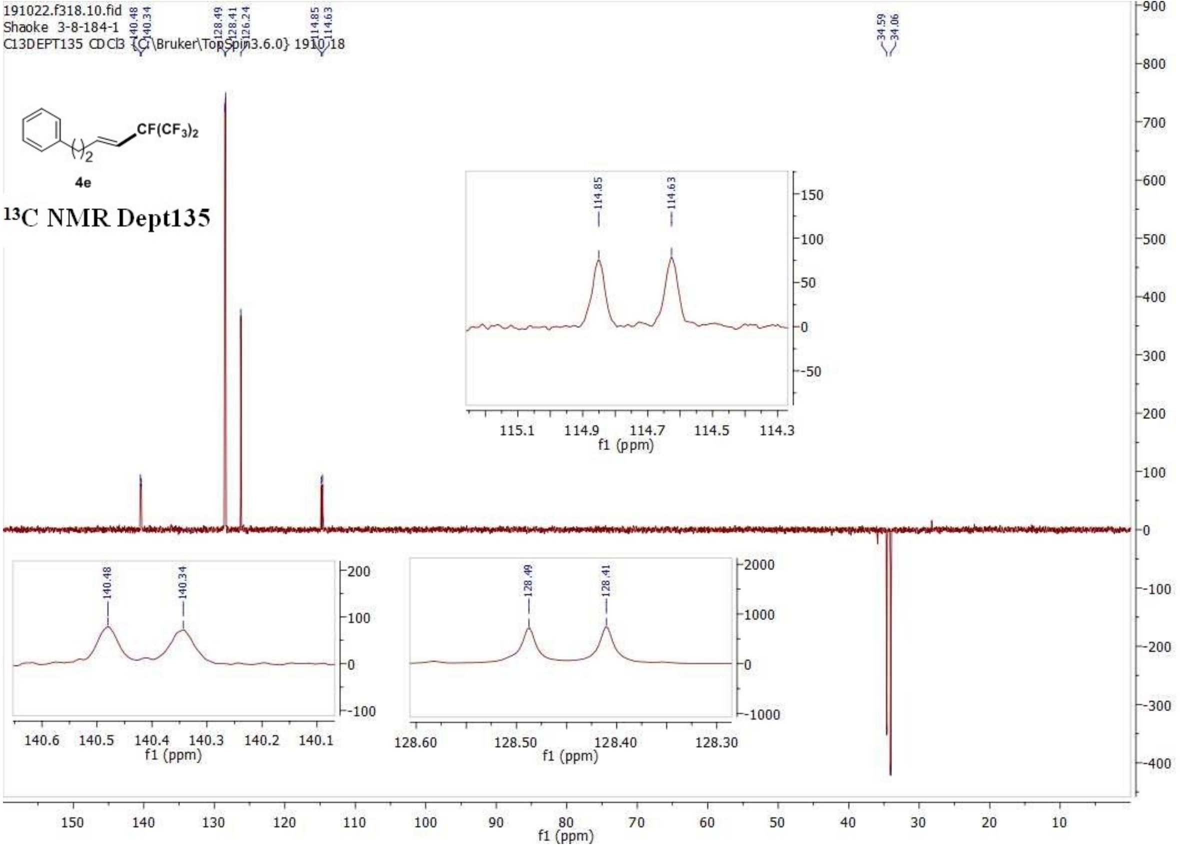

191022.f327.12.fid
Shaoke 3-8-184-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 27
-81.08 -81.10 -81.14 -81.15 -81.19
-107.05 -107.06 -107.12 -107.16 -107.22 -111.55 -111.59 -111.64 -111.68
-124.38 -124.42 -124.45 -124.48 -124.51 -124.54 -124.64 -124.67 -124.71 -124.74 -125.75 -125.78 -125.82 -125.85 -125.89
C_4F_9
4c
E/Z = 81:19
^{19}F NMR
=CHCF_2- (E)
=CHCF_2- (Z)
=CHCF$_2$CF_2- (Z)
=CHCF$_2$CF_2- (E)
1.33
0.59
1.97
-124.5
-125.0
-125.5
-126.0
-126.5
f1 (ppm)
15000
10000
5000
0
3.00
0.58
1.38
1.33
0.59
1.97
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)
32000 30000 28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000

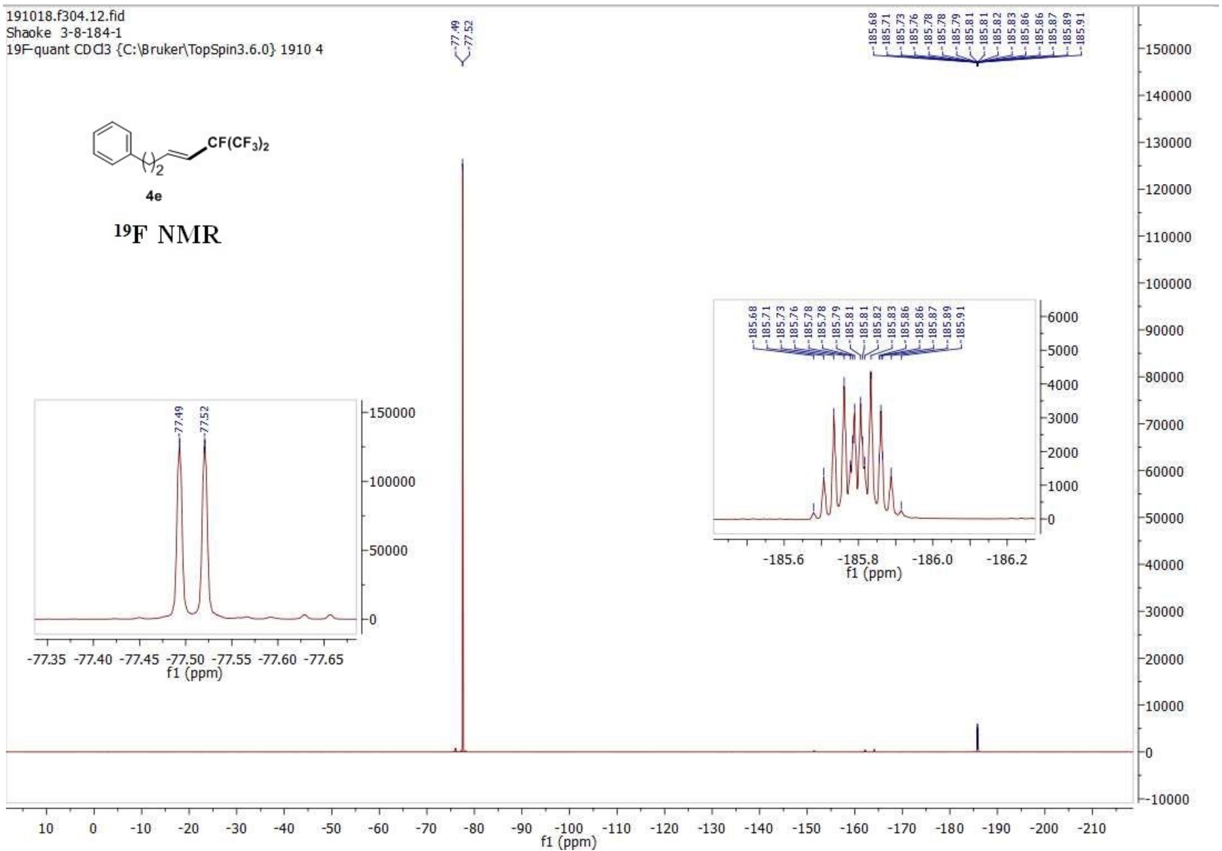

191028.f303.10.fid
Shaoke 3-8-184-4
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 3
7.39 7.38 7.36 7.33 7.30 7.29 7.27 7.24 7.23 7.21 6.55 6.54 6.54 6.53 6.52 6.51 6.51 6.50 6.49 6.48 6.48 6.47 6.46 6.45 6.45 6.44 6.25 6.24 6.24 6.23 6.22 6.21 6.20 6.19 6.19 6.18 6.17 6.16 6.15 6.15 5.73 5.72 5.68 5.63 5.59 5.54 5.49 2.85 2.83 2.80 2.78 2.75 2.74 2.72 2.71 2.69 2.69 2.62 2.61 2.60 2.59 2.59 2.58 2.58 2.57 2.56 2.56 2.55 2.53 2.52
C_3F_7
4d
E/Z = 80:20
^1H NMR
2.85 2.83 2.80 2.78 2.75 2.74 2.72 2.71 2.69 2.69 2.62 2.61 2.60 2.59 2.59 2.58 2.58 2.57 2.56 2.56 2.55 2.53 2.52
10000 8000 6000 4000 2000 0
2.07
0.59
1.49
3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3
f1 (ppm)
-CH_2CH= (Z)
-CH_2CH= (E)
=CHCF$_2$- (E)
=CHCF$_2$- (Z)
2.14
3.11
0.72
0.28
0.28
1.03
2.07
0.59
1.49
16.0 15.5 15.0 14.5 14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)
21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

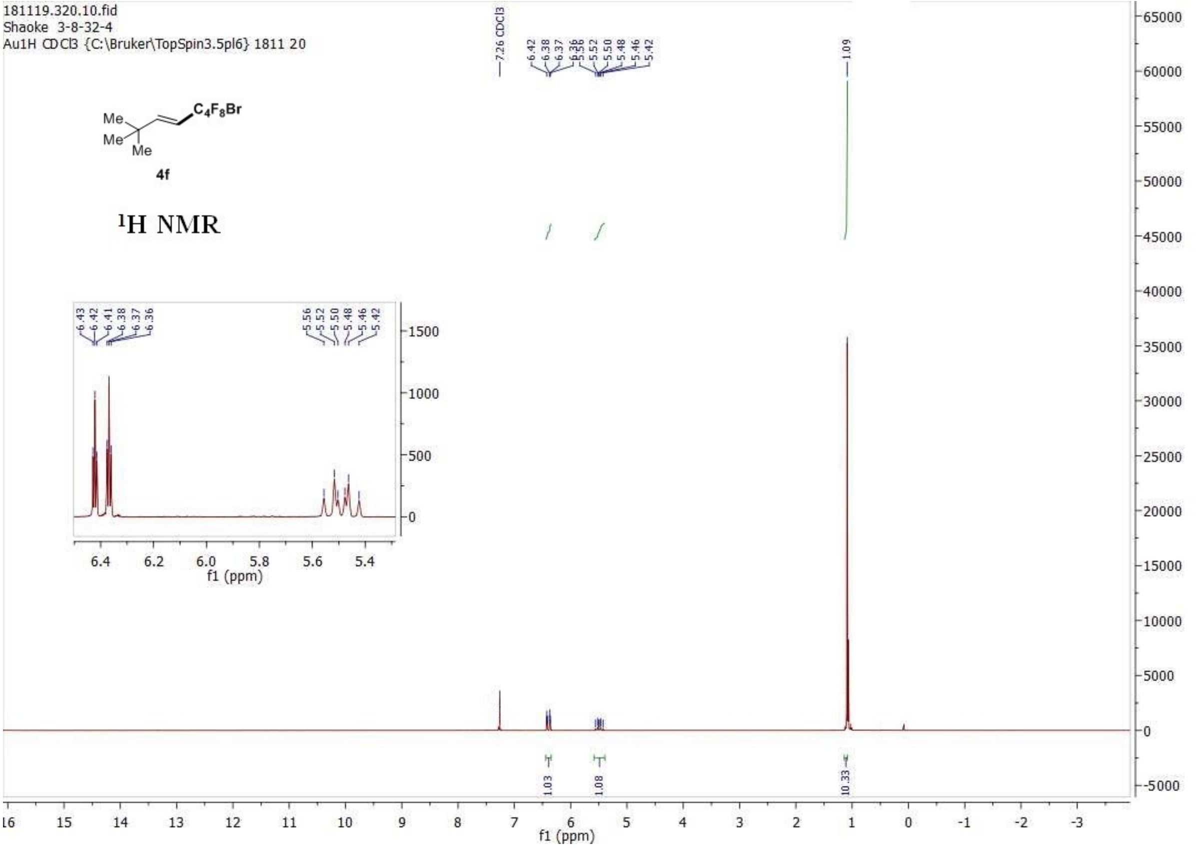

191029.f306.11.fid
Shaoke 3-8-184-4
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 6
-80.39
-80.42
-80.45
-80.48
-107.81
-107.84
-107.86
-107.87
-107.90
-107.91
-107.92
-107.96
-107.97
-107.99
-108.01
-112.37
-112.39
-112.41
-112.42
-112.46
-112.50
-112.51
-112.53
-112.55
-127.93
-128.18
C_3F_7
4d
E/Z = 80:20
^{19}F NMR
=CHCF_2- (Z)
=CHCF_2- (E)
=CHCF$_2$CF_2- (E)
=CHCF$_2$CF_2- (Z)
-127.93
-128.18
1.40
0.54
-127.8
-128.0
-128.2
-128.4
f1 (ppm)
60000
50000
40000
30000
20000
10000
0
3.00
0.54
1.42
1.40
0.54
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)
90000
80000
70000
60000
50000
40000
30000
20000
10000
0

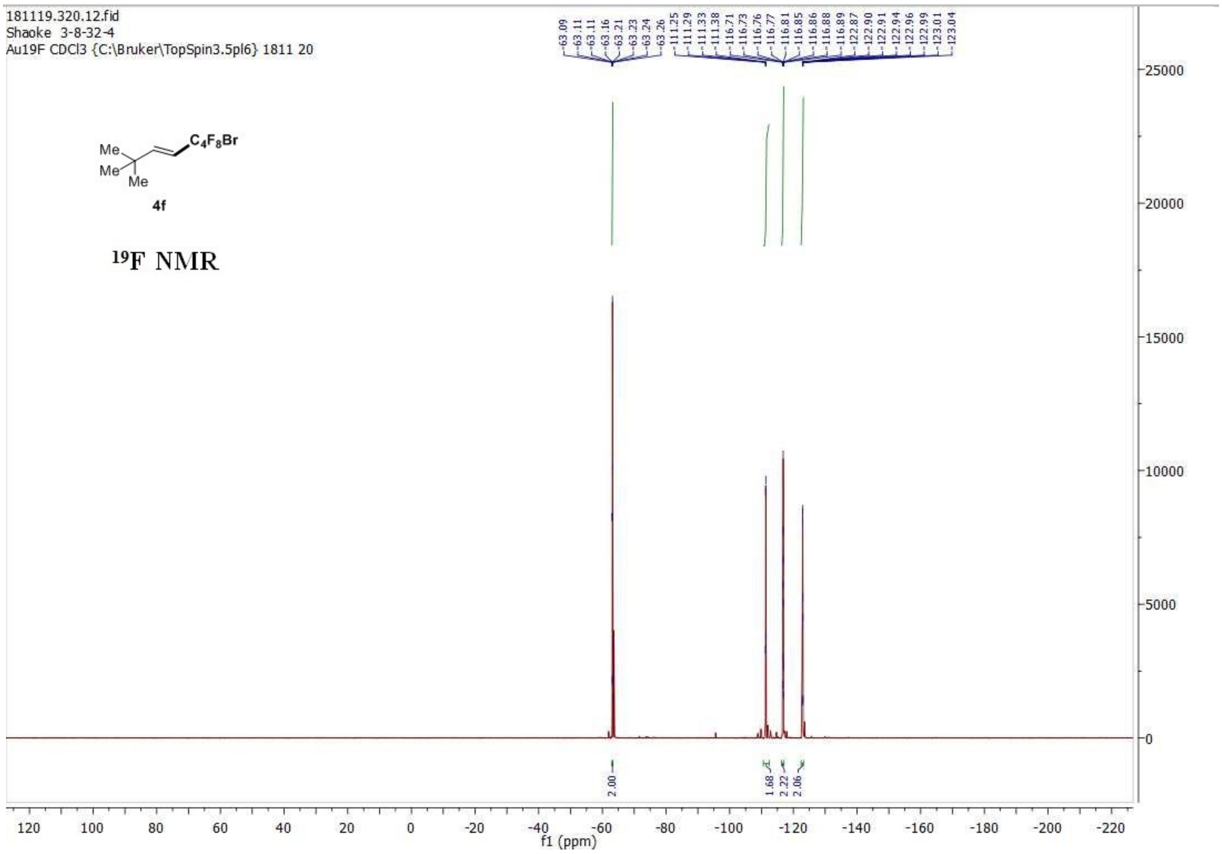

191021.f304.10.fid
Shaoke 3-8-184-1
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 4
7.36
7.36
7.34
7.31
7.27
7.25
7.21
7.21
7.19
7.17
6.45
6.43
6.41
6.40
6.38
6.35
5.62
5.61
5.61
5.61
5.61
5.60
5.60
5.60
5.57
5.56
5.56
5.55
5.55
5.54
5.54
5.53
5.53
5.49
5.48
5.48
5.48
2.83
2.81
2.78
2.59
2.57
2.56
2.54
2.54
2.51
CF(CF$_3$)$_2$
4e
^{1}H NMR
2.31
3.37
1.00
1.08
2.24
2.06
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
f1 (ppm)
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000


191021.f304.11.fid
Shaoke 3-8-184-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 4
140.47
140.33
128.48
128.40
126.23
114.84
114.61
77.00 CDCl3
34.58
34.05
CF(CF3)2
2
4e
13C NMR
140.47
140.40
140.33
128.48
128.40
114.84
114.61
f1 (ppm)

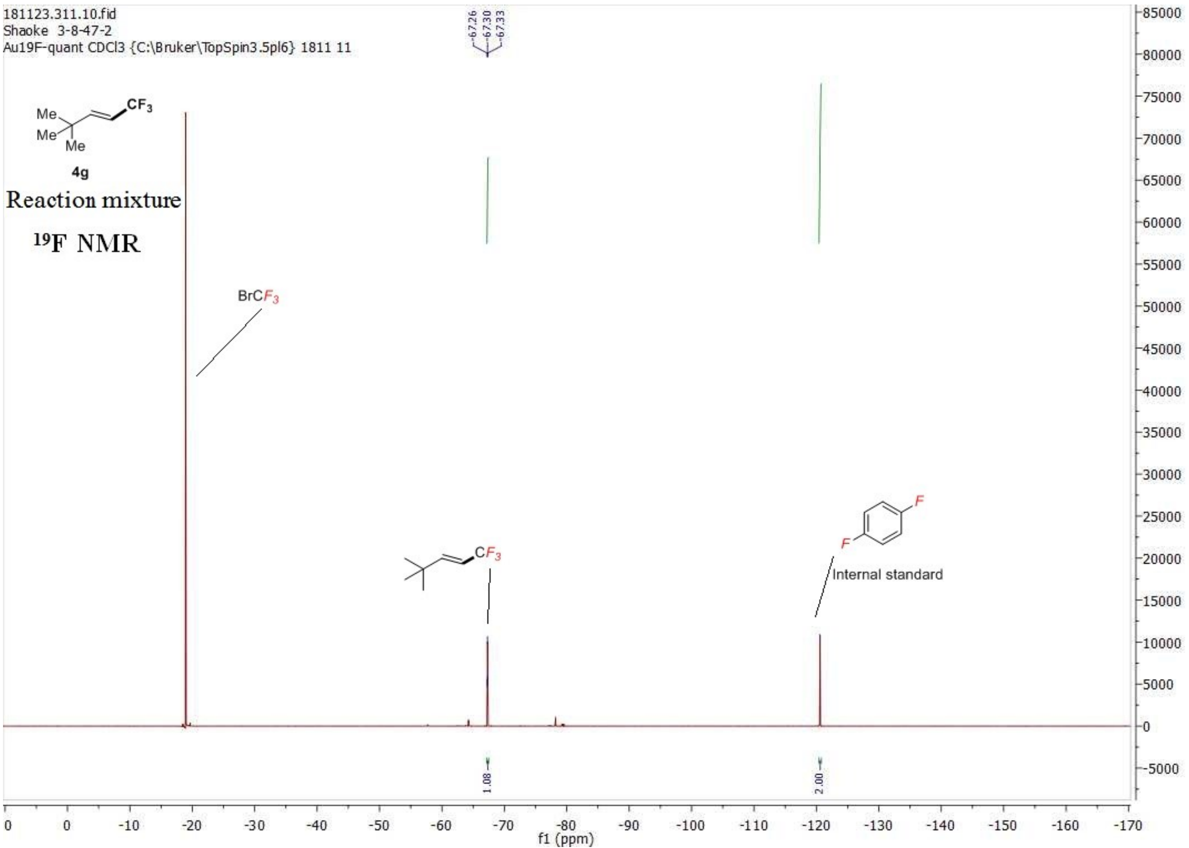

191022.f318.10.fid
Shaoke 3-8-184-1
C13DEPT135 CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 18
140.48
140.34
128.49
128.41
126.24
114.85
114.63
34.59
34.06
CF(CF3)2
2
4e
13C NMR Dept135
114.85
114.63
140.48
140.34
128.49
128.41
f1 (ppm)

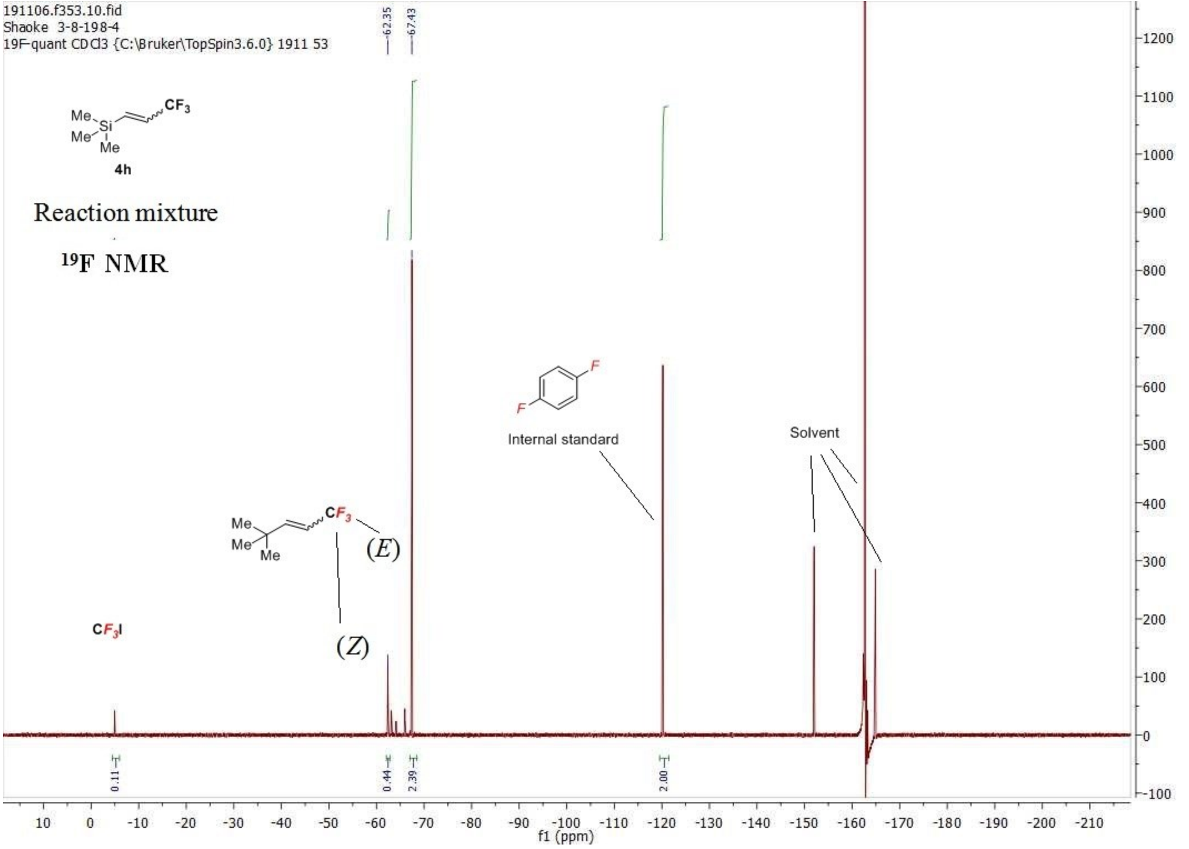

191018.f304.12.fid
Shaoke 3-8-184-1
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1910 4
CF(CF3)2
2
4e
19F NMR
-77.49
-77.52
-185.68 -185.71 -185.73 -185.76 -185.78 -185.78 -185.79 -185.81 -185.81 -185.82 -185.83 -185.86 -185.86 -185.87 -185.89 -185.91
f1 (ppm)

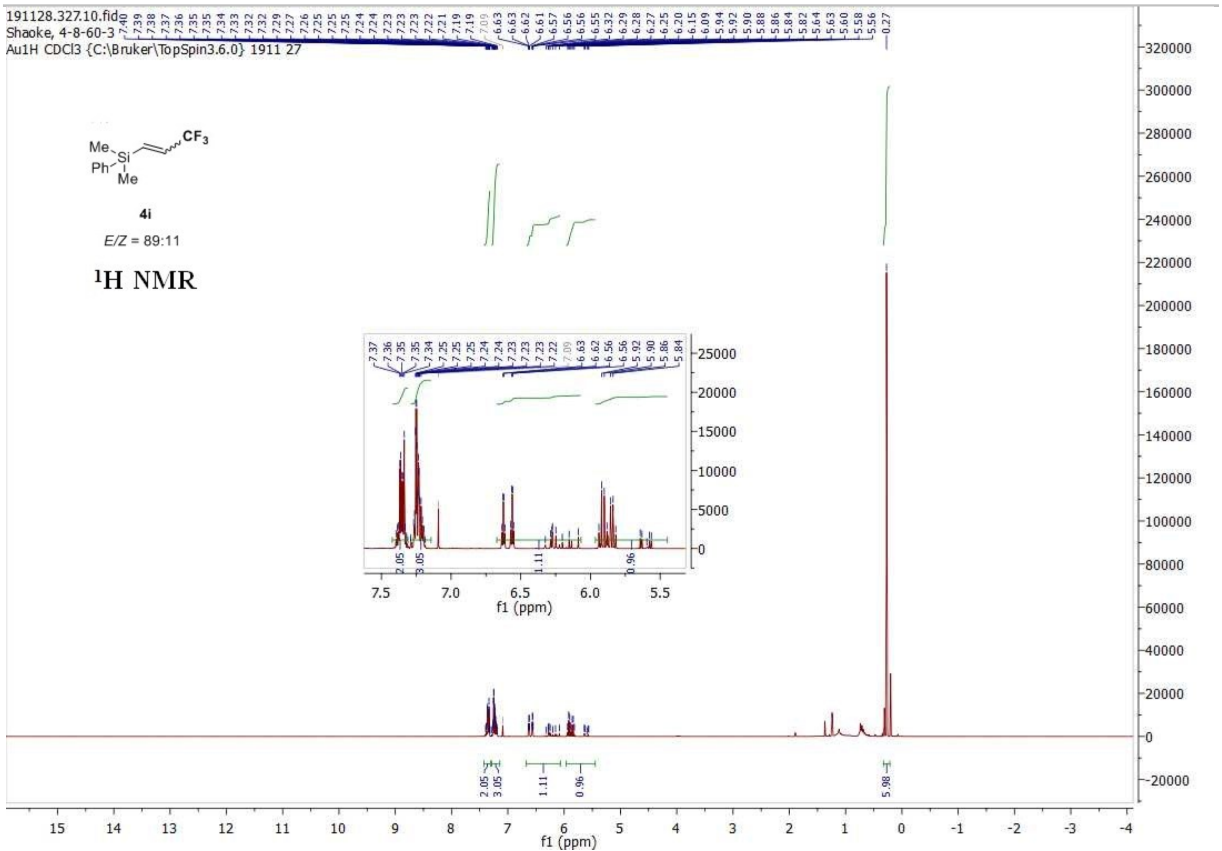

181119.320.10.fid
Shaoke 3-8-32-4
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1811 20
Me
Me
Me
C4F8Br
4f
1H NMR
7.26 CDCl3
6.42 6.38 6.37 6.36 5.56 5.52 5.50 5.48 5.46 5.42
1.09
6.43 6.42 6.41 6.38 6.37 6.36
5.56 5.52 5.50 5.48 5.46 5.42
1.03
1.08
10.33
f1 (ppm)

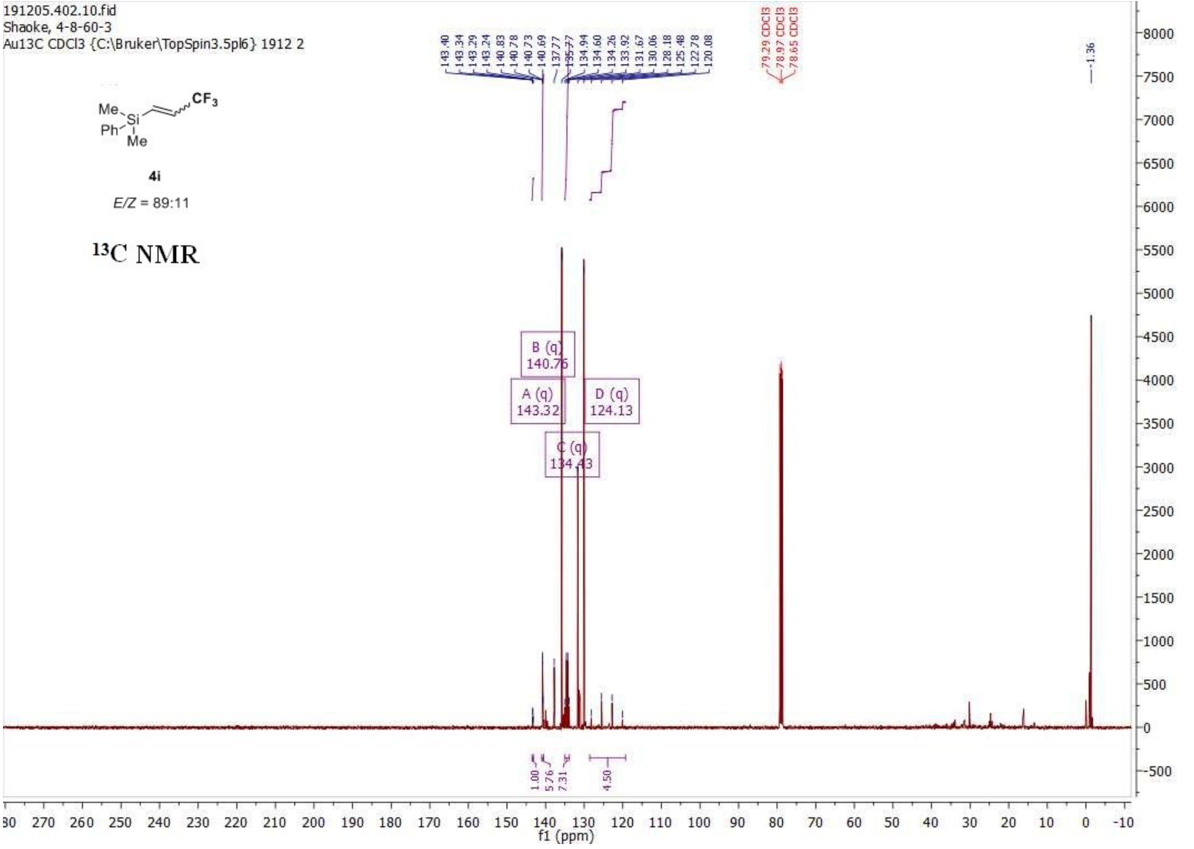

181119.320.12.fid
Shaoke 3-8-32-4
Au19F CDCl3 {C:\Bruker\TopSpin3.5pl6} 1811 20
-63.09
-63.11
-63.11
-63.16
-63.21
-63.23
-63.24
-63.26
-111.25
-111.29
-111.33
-111.38
-116.71
-116.73
-116.76
-116.77
-116.81
-116.85
-116.86
-116.88
-116.89
-122.87
-122.90
-122.91
-122.94
-122.96
-122.99
-123.01
-123.04
Me
Me
Me
C4F8Br
4f
19F NMR
25000
20000
15000
10000
5000
0
2.00
1.68
2.22
2.06
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

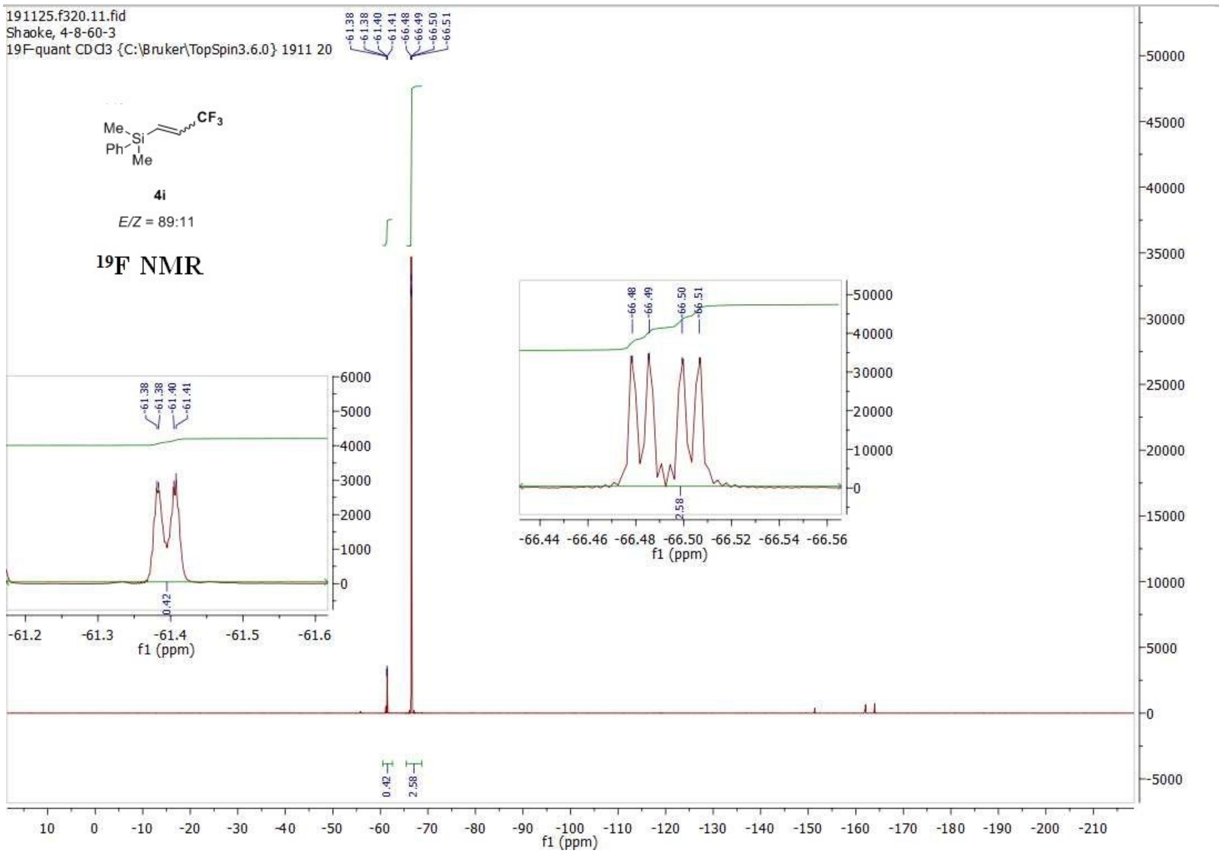
8.4 Spectra of trifluoromethylated alkenes

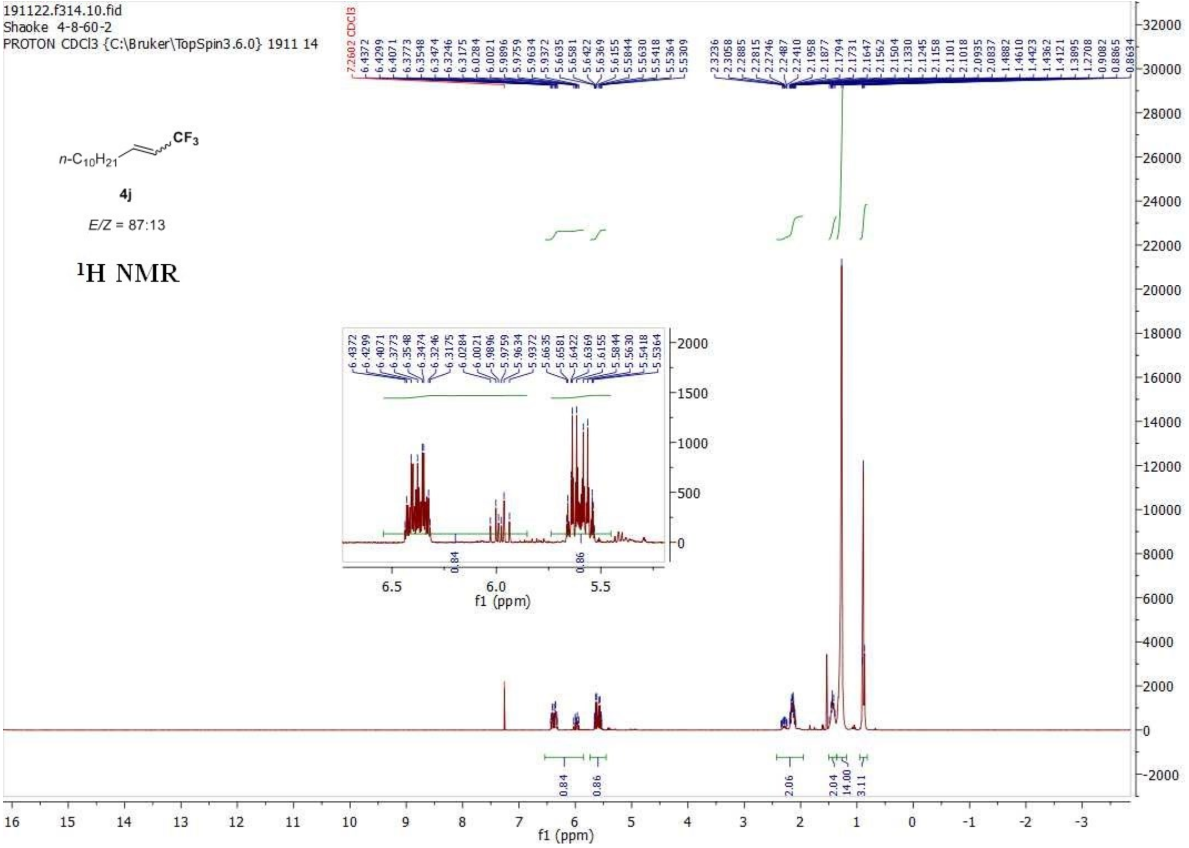


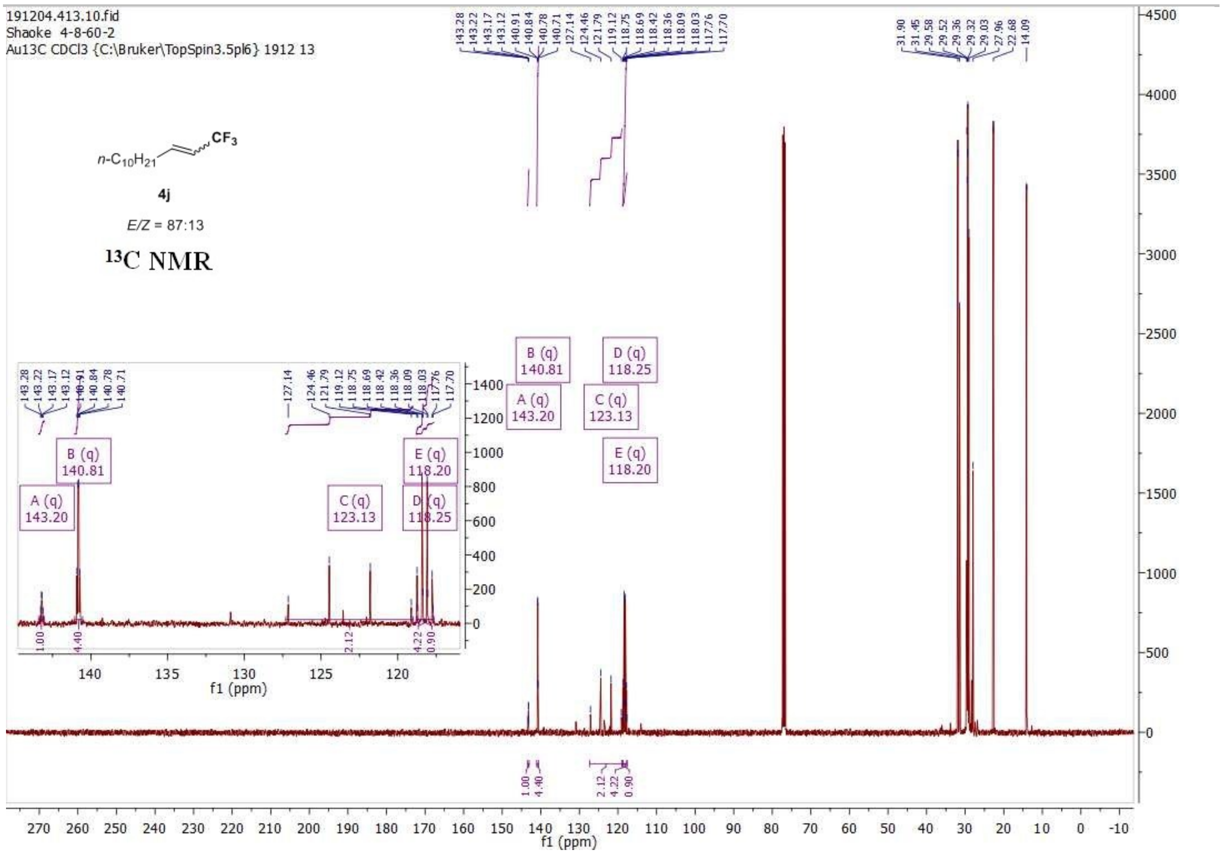


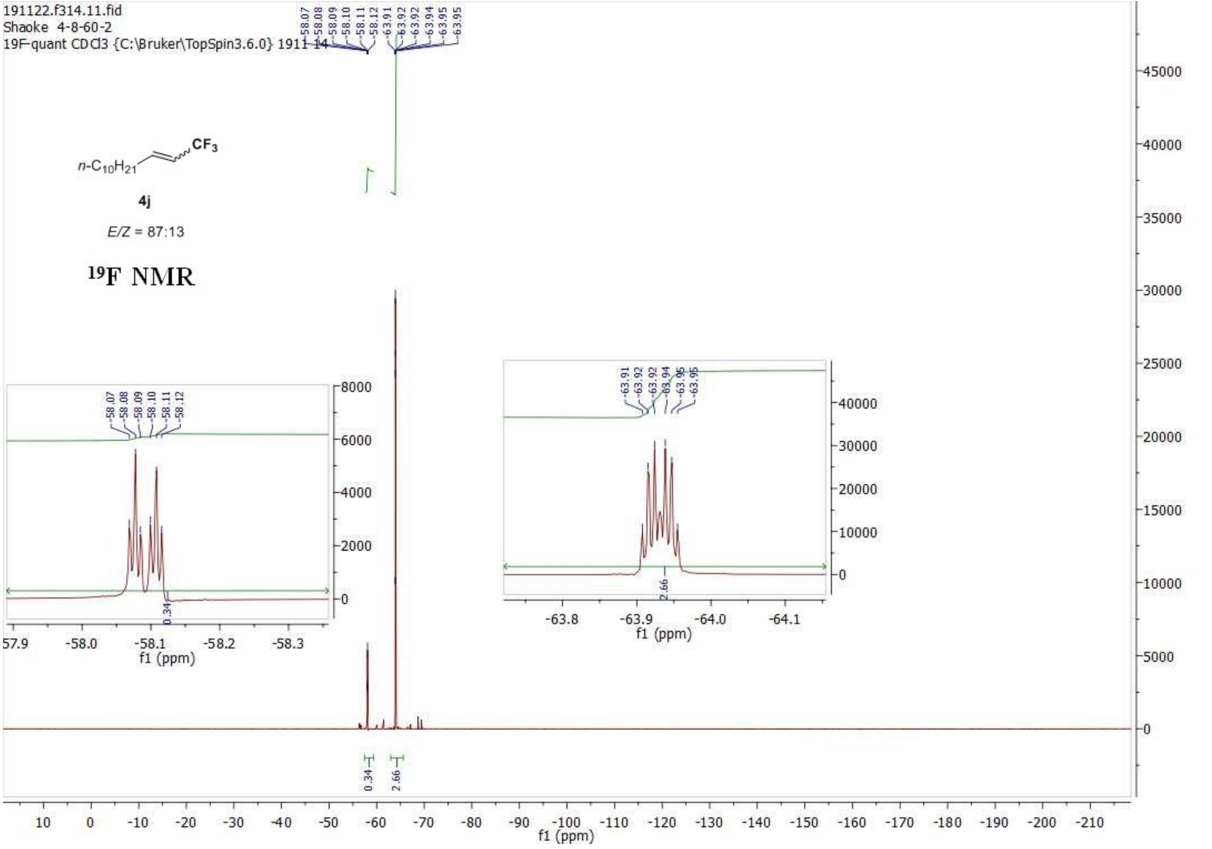

191128.327.10.fid
Shaoke, 4-8-60-3
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 27
7.40 7.39 7.38 7.37 7.36 7.35 7.35 7.34 7.33 7.32 7.32 7.29 7.27 7.26 7.25 7.25 7.25 7.24 7.24 7.23 7.23 7.23 7.22 7.21 7.19 7.19 7.09 6.63 6.62 6.61 6.57 6.56 6.56 6.55 6.32 6.29 6.28 6.27 6.25 6.20 6.15 6.09 5.94 5.92 5.90 5.88 5.86 5.84 5.82 5.64 5.63 5.60 5.58 5.56 0.27
Me
Si
Ph
Me
CF3
4i
E/Z = 89:11
1H NMR
7.37 7.36 7.35 7.35 7.34 7.25 7.25 7.25 7.24 7.23 7.23 7.23 7.22 7.09 6.63 6.62 6.56 6.56 5.92 5.90 5.86 5.84
2.05
3.05
1.11
0.96
5.98
f1 (ppm)

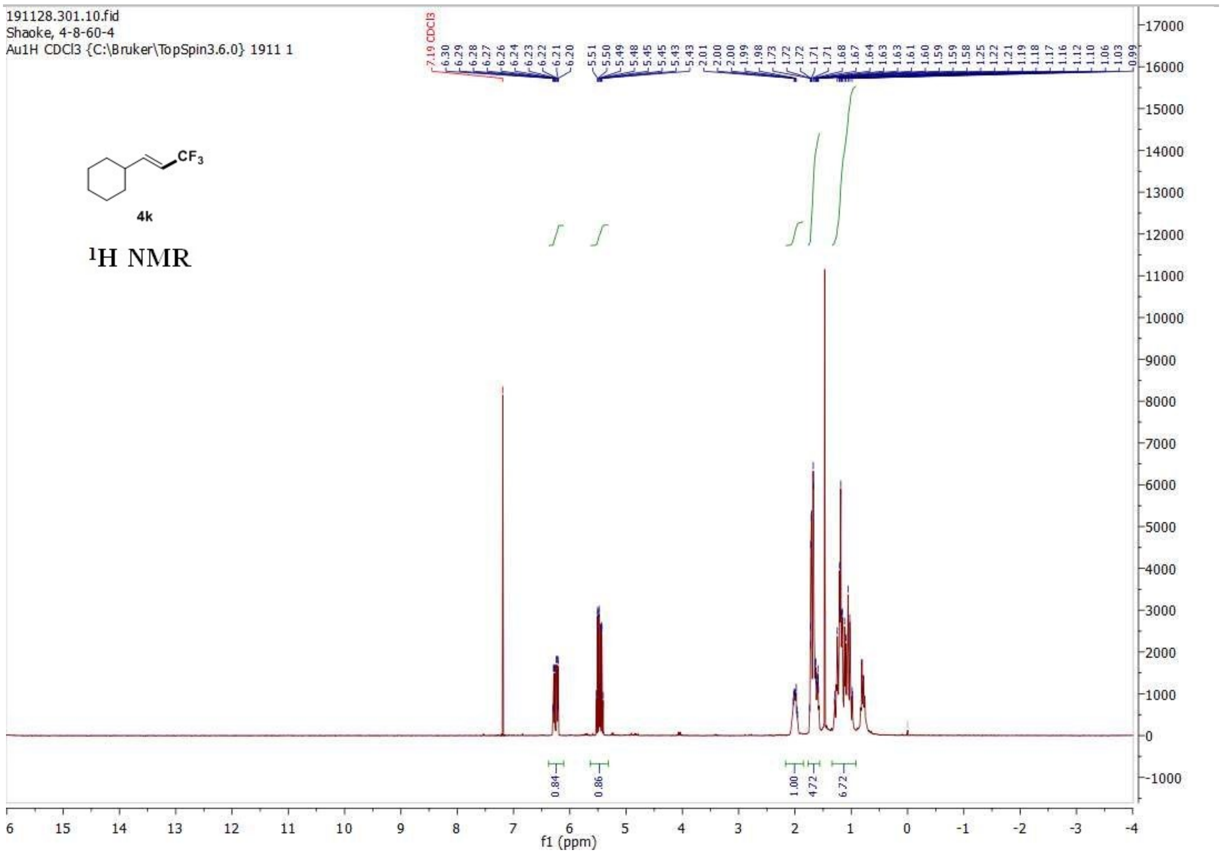

191205.402.10.fid
Shaoke, 4-8-60-3
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1912 2
143.40 143.34 143.29 143.24 140.83 140.78 140.73 140.69 137.77 135.77 134.94 134.60 134.26 133.92 131.67 130.06 128.18 125.48 122.78 120.08
79.29 CDCl3
78.97 CDCl3
78.65 CDCl3
-1.36
Me
Si
Ph
Me
CF3
4i
E/Z = 89:11
13C NMR
B (q)
140.76
A (q)
143.32
D (q)
124.13
C (q)
134.43
1.00
5.76
7.31
4.50
f1 (ppm)

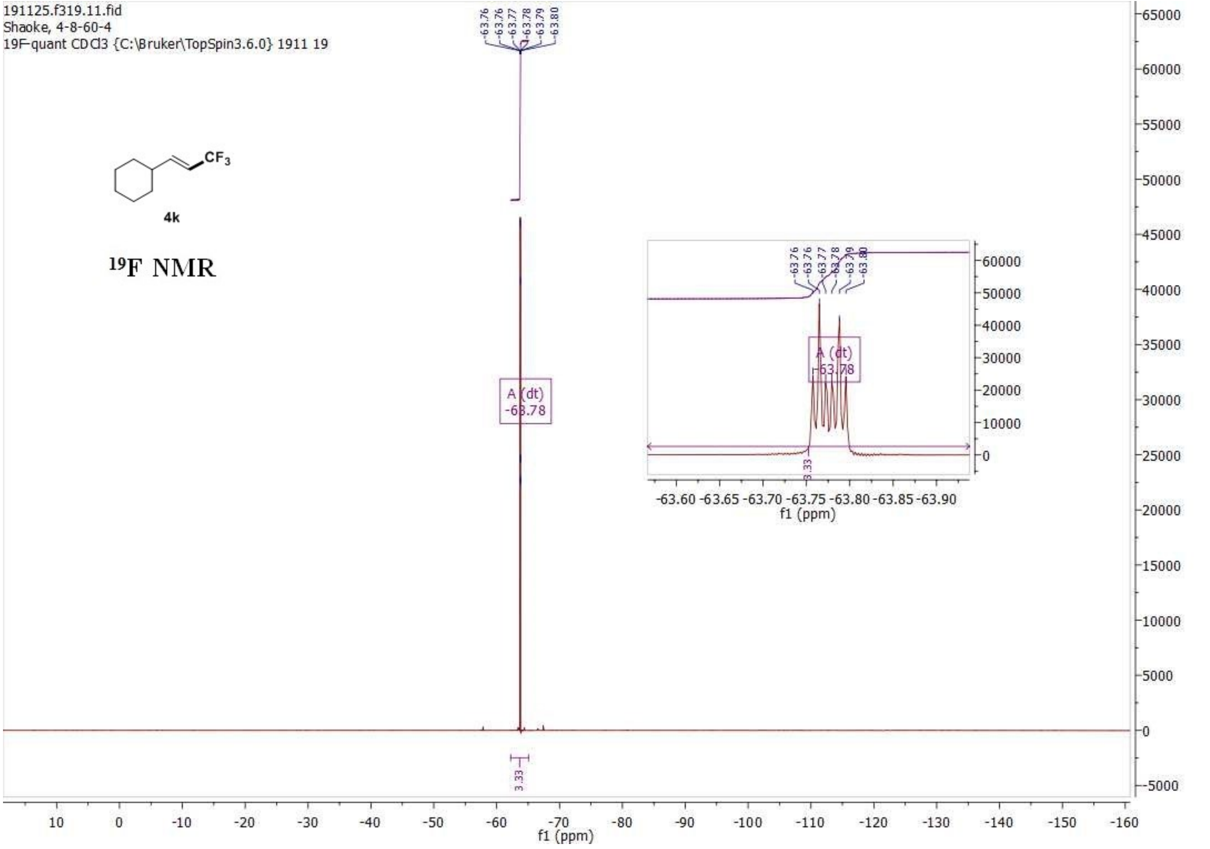

191125.f320.11.fid
Shaoke, 4-8-60-3
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 20
-61.38
-61.38
-61.40
-61.41
-66.48
-66.49
-66.50
-66.51
Me
Si
Ph
Me
CF3
4i
E/Z = 89:11
19F NMR
0.42
2.58
f1 (ppm)

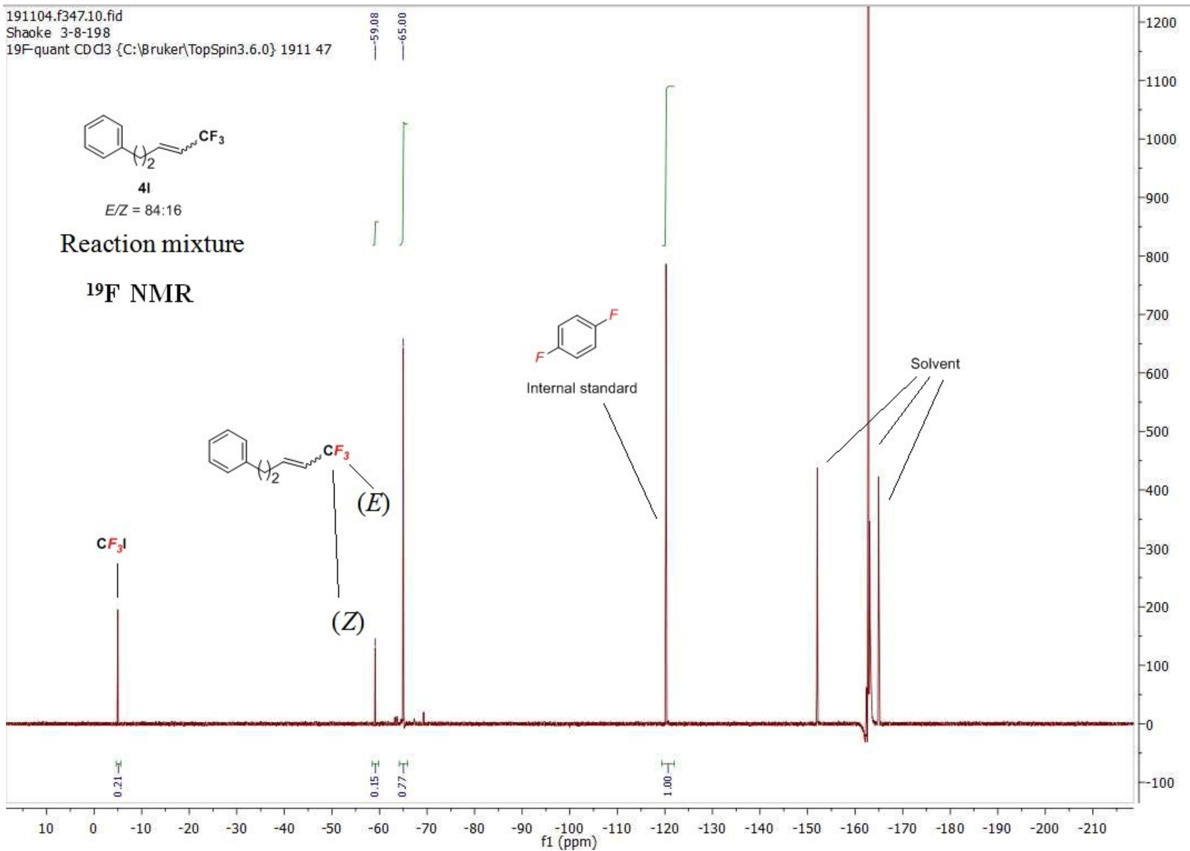

191122.f314.10.fid
Shaoke 4-8-60-2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 14
7.2602 CDCl3
6.4372
6.4299
6.4071
6.3773
6.3548
6.3474
6.3246
6.3175
6.0284
6.0021
5.9896
5.9759
5.9634
5.9372
5.6635
5.6581
5.6422
5.6369
5.6155
5.5844
5.5630
5.5418
5.5364
5.5309
2.3236
2.3058
2.2885
2.2815
2.2746
2.2487
2.2410
2.1958
2.1877
2.1794
2.1731
2.1647
2.1562
2.1504
2.1330
2.1245
2.1158
2.1101
2.1018
2.0935
2.0837
1.4882
1.4610
1.4423
1.4362
1.4121
1.3895
1.2708
0.9082
0.8865
0.8634
n-C10H21
CF3
4j
E/Z = 87:13
1H NMR
0.84
0.86
2.06
2.04
14.00
3.11
f1 (ppm)

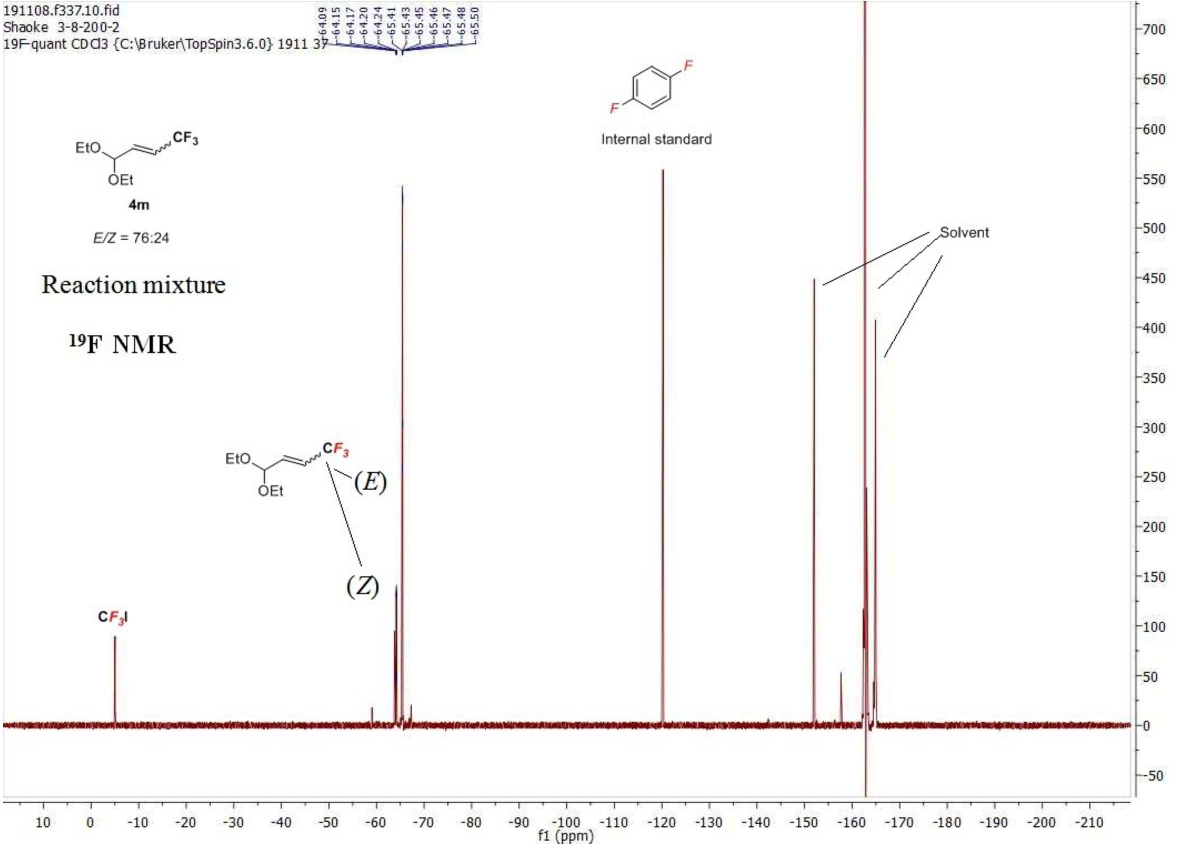

191204.413.10.fid
Shaoke 4-8-60-2
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1912 13
n-C10H21 CF3
4j
E/Z = 87:13
13C NMR
143.28 143.22 143.17 143.12 140.91 140.84 140.78 140.71 127.14 124.46 121.79 119.12 118.75 118.69 118.42 118.36 118.09 118.03 117.76 117.70
31.90 31.45 29.58 29.52 29.36 29.32 29.03 27.96 22.68 14.09
A (q) 143.20
B (q) 140.81
C (q) 123.13
D (q) 118.25
E (q) 118.20
1.00 4.40 2.12 4.22 0.90
f1 (ppm)

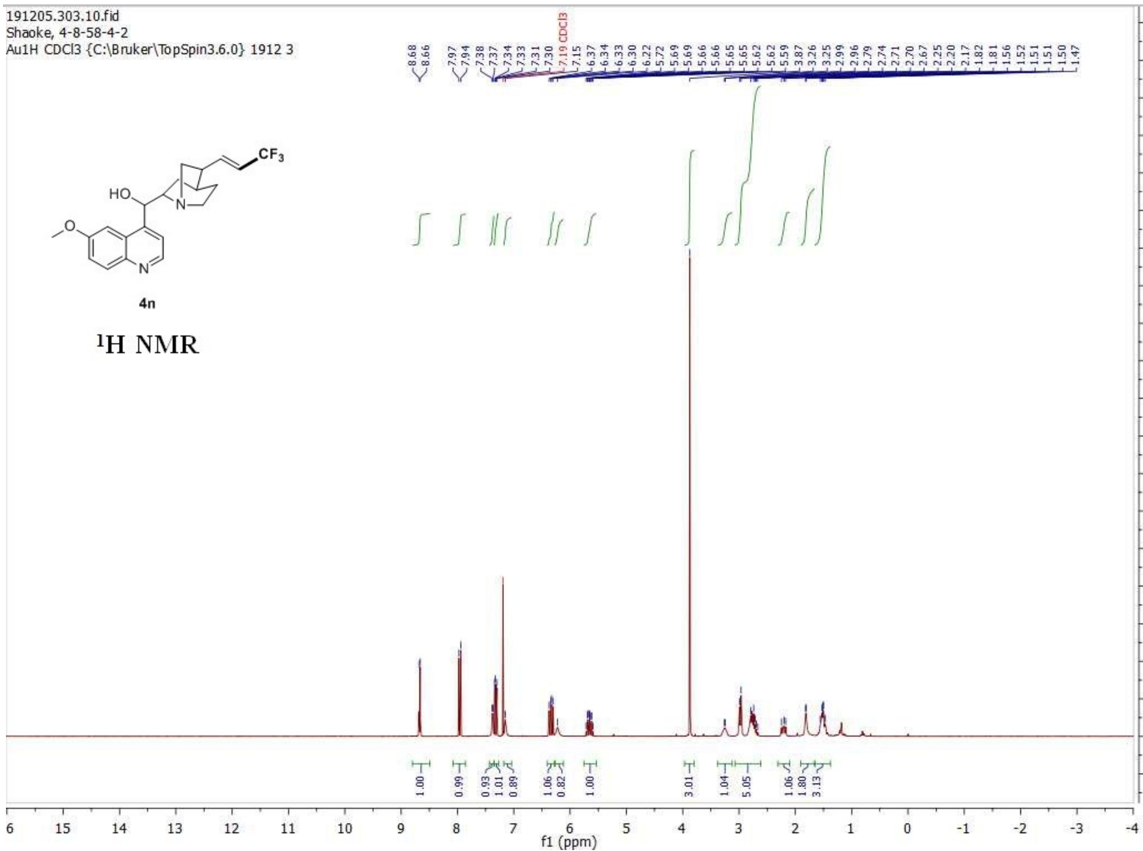

191122.f314.11.fid
Shaoke 4-8-60-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 14
-58.07 -58.08 -58.09 -58.10 -58.11 -58.12 -63.91 -63.92 -63.94 -63.95 -63.95
n-C10H21 CF3
4j
E/Z = 87:13
19F NMR
0.34 2.66
f1 (ppm)

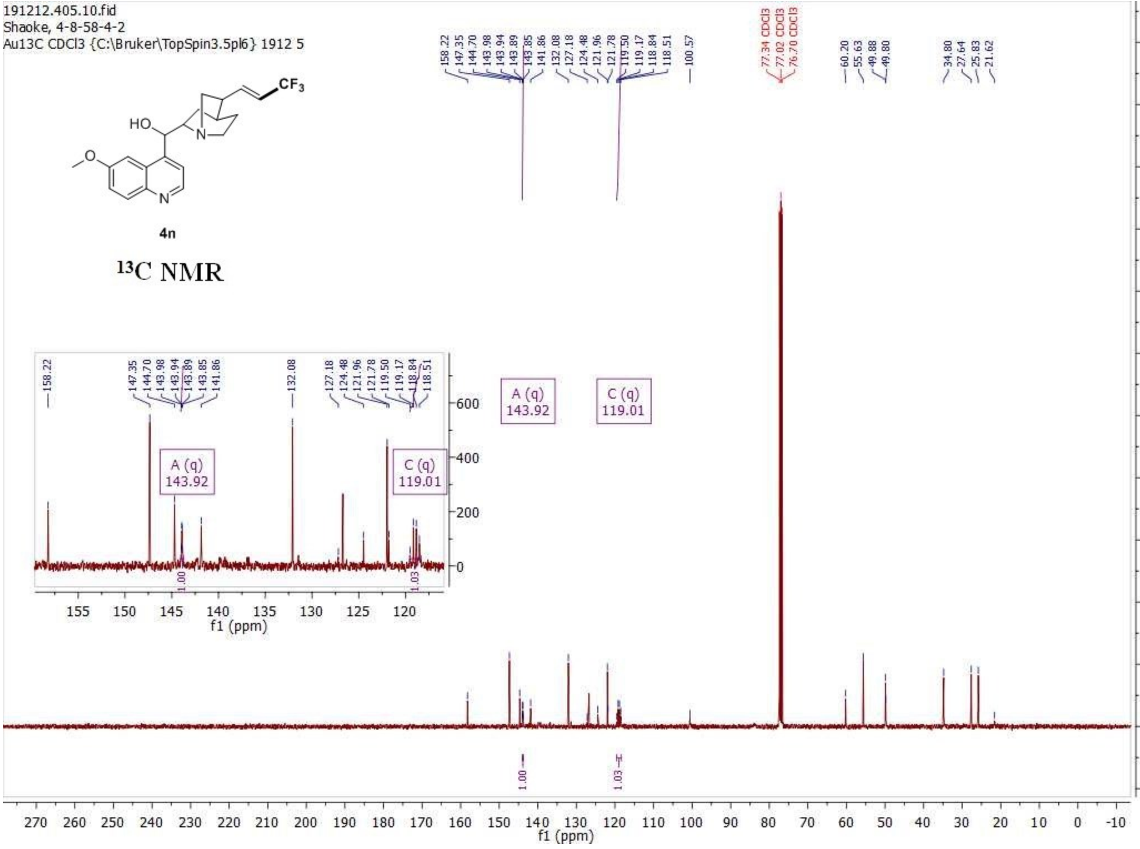

191128.301.10.fid
Shaoke, 4-8-60-4
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 1
7.19 CDCl3
6.30
6.29
6.28
6.27
6.26
6.24
6.23
6.22
6.21
6.20
5.51
5.50
5.49
5.48
5.45
5.45
5.43
5.43
2.01
2.00
1.99
1.98
1.73
1.72
1.72
1.71
1.71
1.68
1.67
1.64
1.63
1.63
1.61
1.60
1.59
1.59
1.58
1.25
1.22
1.21
1.19
1.18
1.17
1.16
1.12
1.10
1.06
1.03
0.99
CF3
4k
1H NMR
0.84
0.86
1.00
4.72
6.72
f1 (ppm)

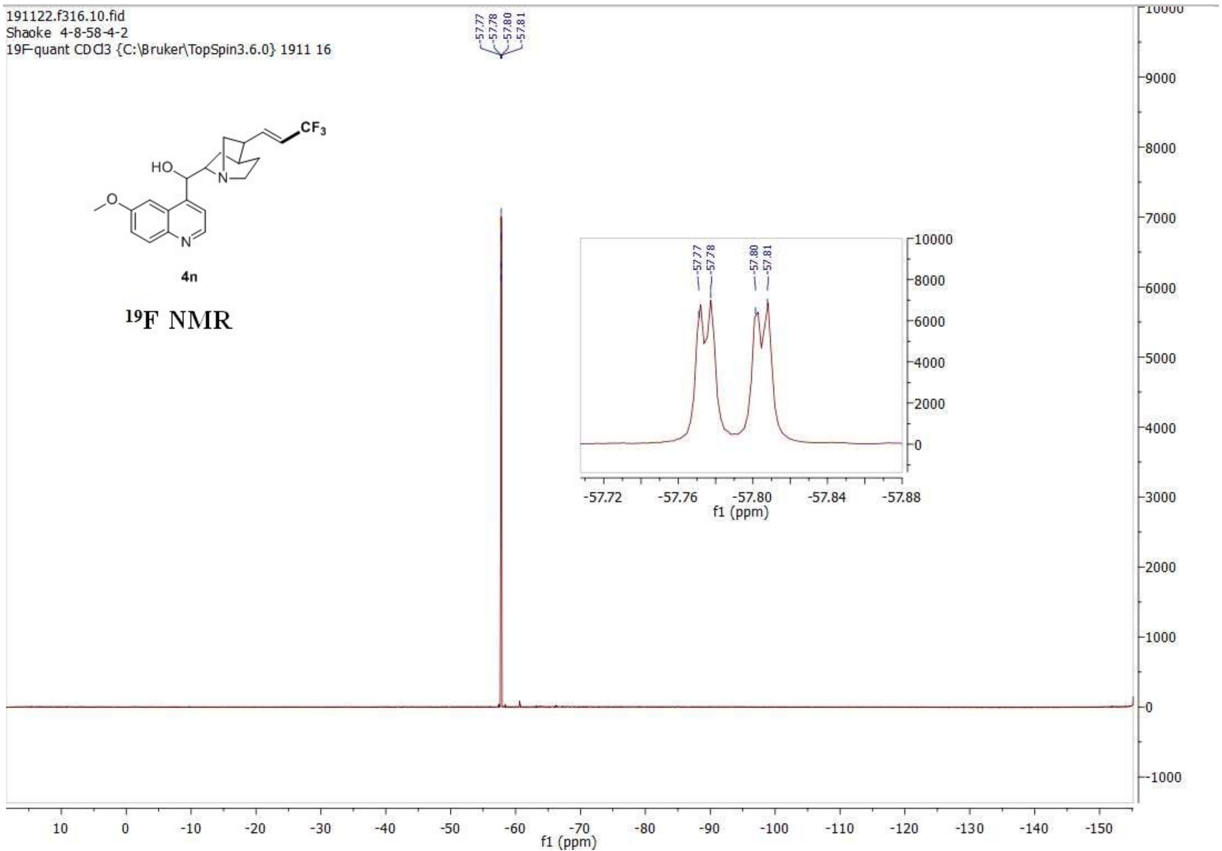

191125.f319.11.fid
Shaoke, 4-8-60-4
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 19
-63.76
-63.76
-63.77
-63.78
-63.79
-63.80
CF3
4k
19F NMR
A (dt)
-63.78
3.33
f1 (ppm)

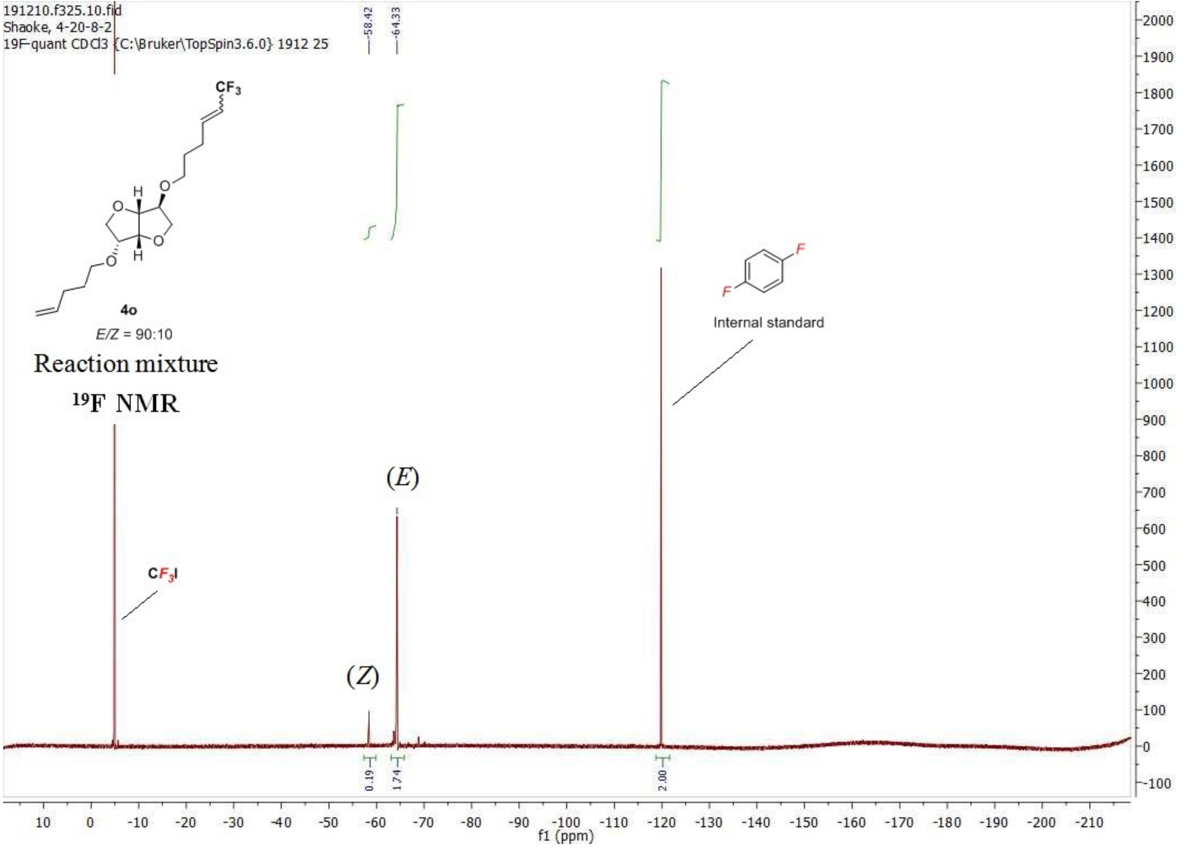

191104.f347.10.fid
Shaoke 3-8-198
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 47
-59.08
-65.00
CF3
4l
E/Z = 84:16
Reaction mixture
19F NMR
CF3I
CF3
(E)
(Z)
F
F
Internal standard
Solvent
0.21
0.15
0.77
1.00
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

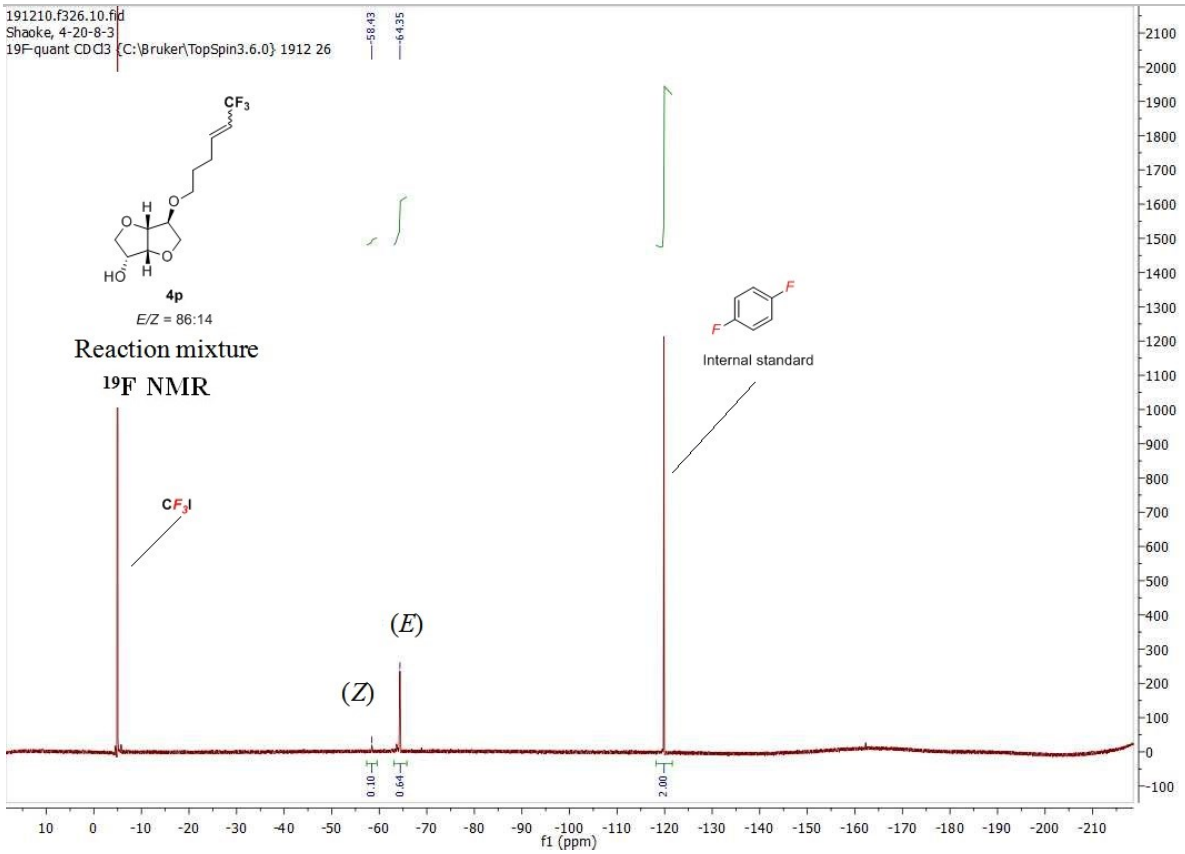

191108.f337.10.fid
Shaoke 3-8-200-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 37
-64.09
-64.15
-64.17
-64.20
-64.24
-65.41
-65.43
-65.45
-65.46
-65.47
-65.48
-65.50
EtO
OEt
CF3
4m
E/Z = 76:24
Reaction mixture
19F NMR
EtO
OEt
CF3
(E)
(Z)
CF3I
F
F
Internal standard
Solvent
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


191205.303.10.fid
Shaoke, 4-8-58-4-2
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1912 3
8.68 8.66 7.97 7.94 7.38 7.37 7.34 7.33 7.31 7.30 7.19 CDCl3 7.15 6.37 6.34 6.33 6.30 6.22 5.72 5.69 5.69 5.66 5.66 5.65 5.65 5.62 5.62 5.59 3.87 3.26 3.25 2.99 2.96 2.79 2.74 2.71 2.70 2.67 2.25 2.20 2.17 1.82 1.81 1.56 1.52 1.51 1.51 1.50 1.47
HO
N
N
CF3
4n
1H NMR
1.00 0.99 0.93 1.01 0.89 1.06 0.82 1.00 3.01 1.04 5.05 1.06 1.80 3.13
f1 (ppm)

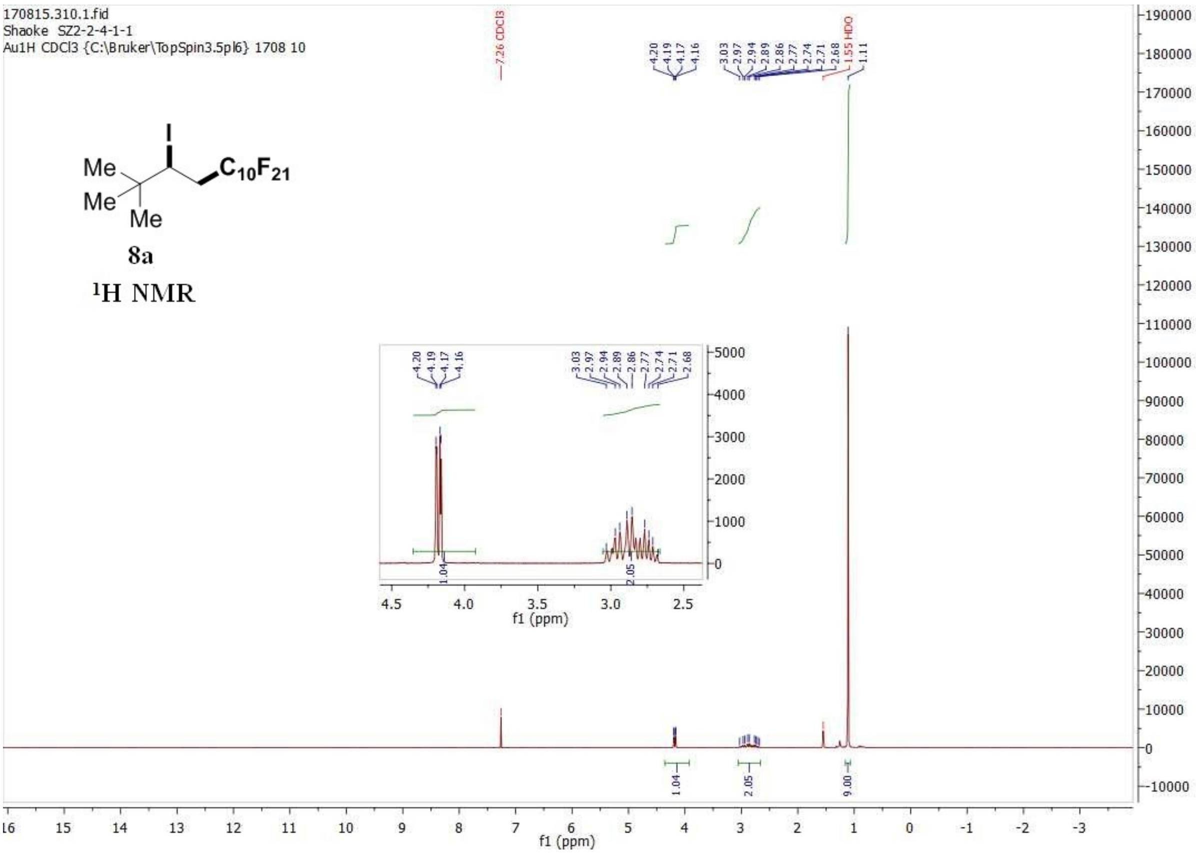

191212.405.10.fid
Shaoke, 4-8-58-4-2
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1912 5
158.22 147.35 144.70 143.98 143.94 143.89 143.85 141.86 132.08 127.18 124.48 121.96 121.78 119.50 119.17 118.84 118.51 100.57 77.34 CDCl3 77.02 CDCl3 76.70 CDCl3 60.20 55.63 49.88 49.80 34.80 27.64 25.83 21.62
HO
N
N
CF3
4n
13C NMR
A (q) 143.92
C (q) 119.01
1.00 1.03
f1 (ppm)

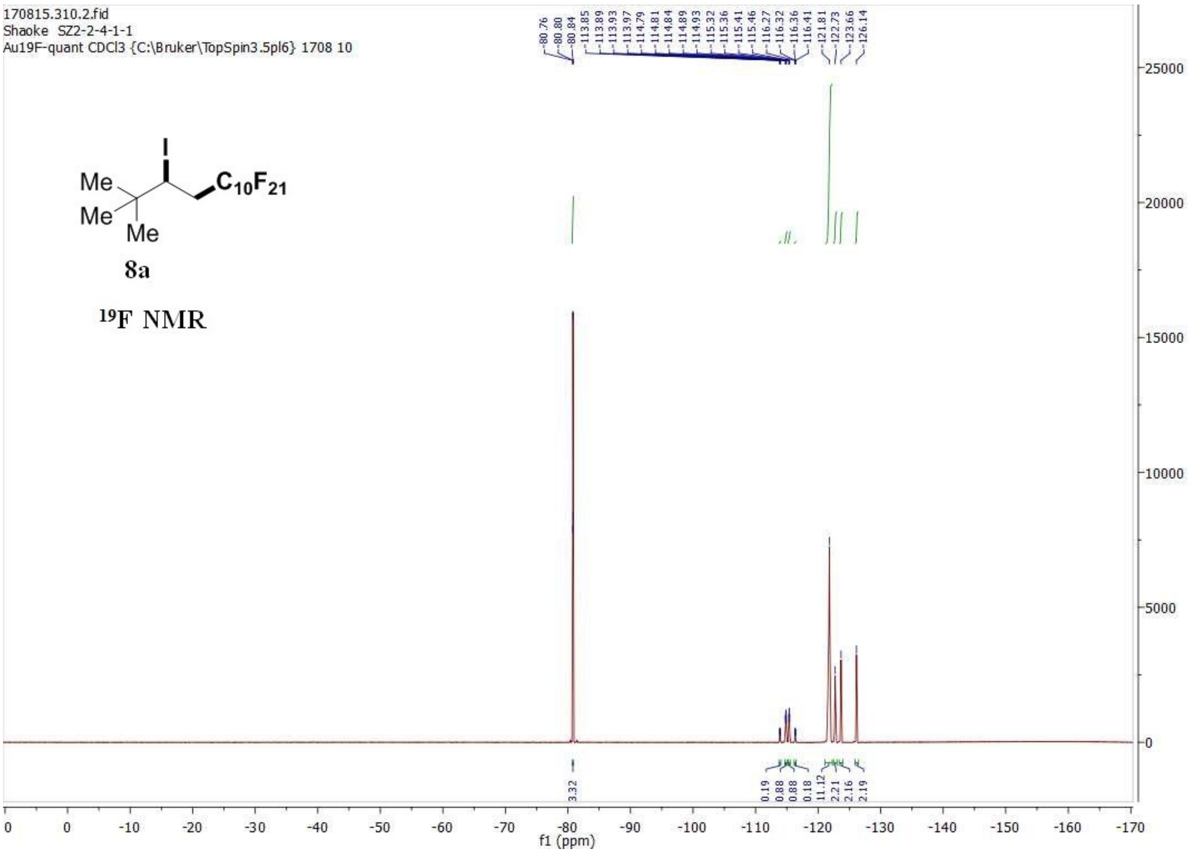

191122.f316.10.fid
Shaoke 4-8-58-4-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 16
-57.77
-57.78
-57.80
-57.81
CF3
HO
O
N
N
4n
19F NMR
-57.77
-57.78
-57.80
-57.81
10000
8000
6000
4000
2000
0
-57.72
-57.76
-57.80
-57.84
-57.88
f1 (ppm)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

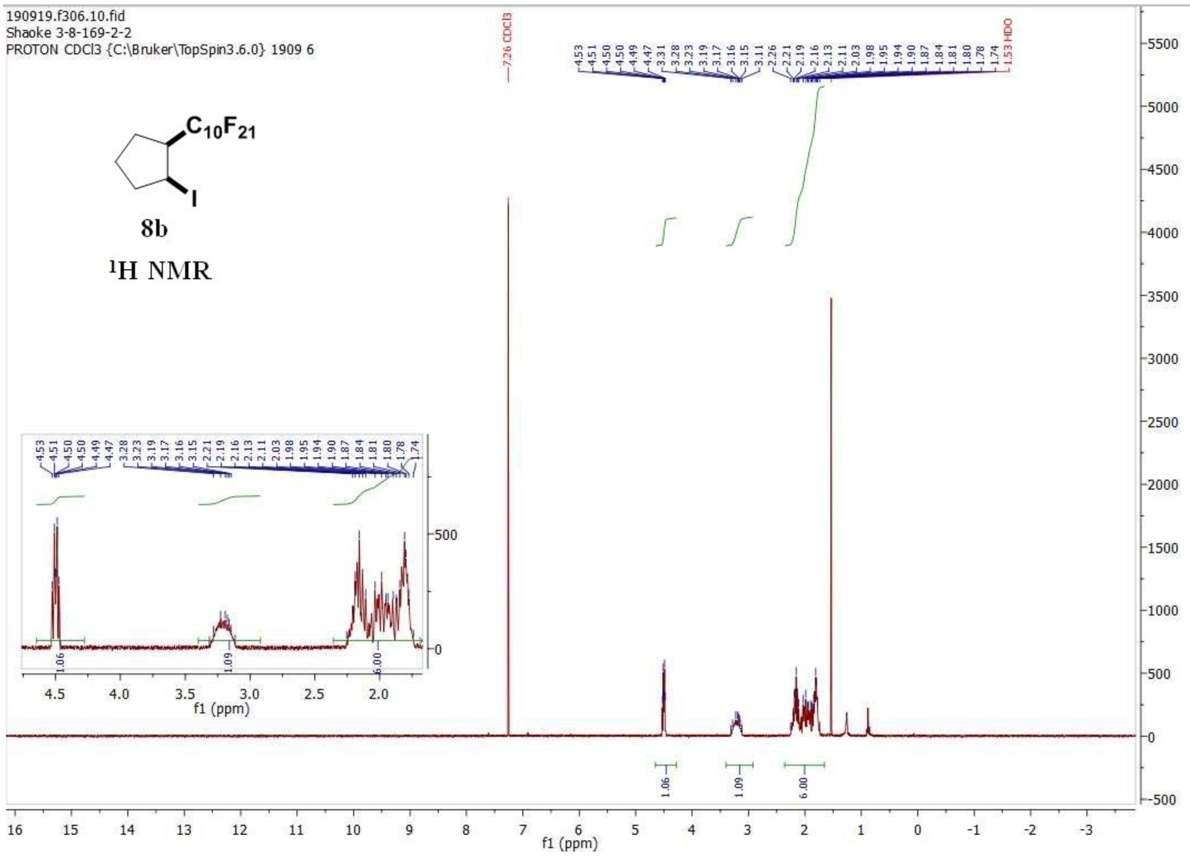

191210.f325.10.fid
Shaoke, 4-20-8-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1912 25
-58.42
-64.33
CF3
H
O
O
O
H
O
4o
E/Z = 90:10
Reaction mixture
19F NMR
F
F
Internal standard
CF3I
(E)
(Z)
0.19
1.74
2.00
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

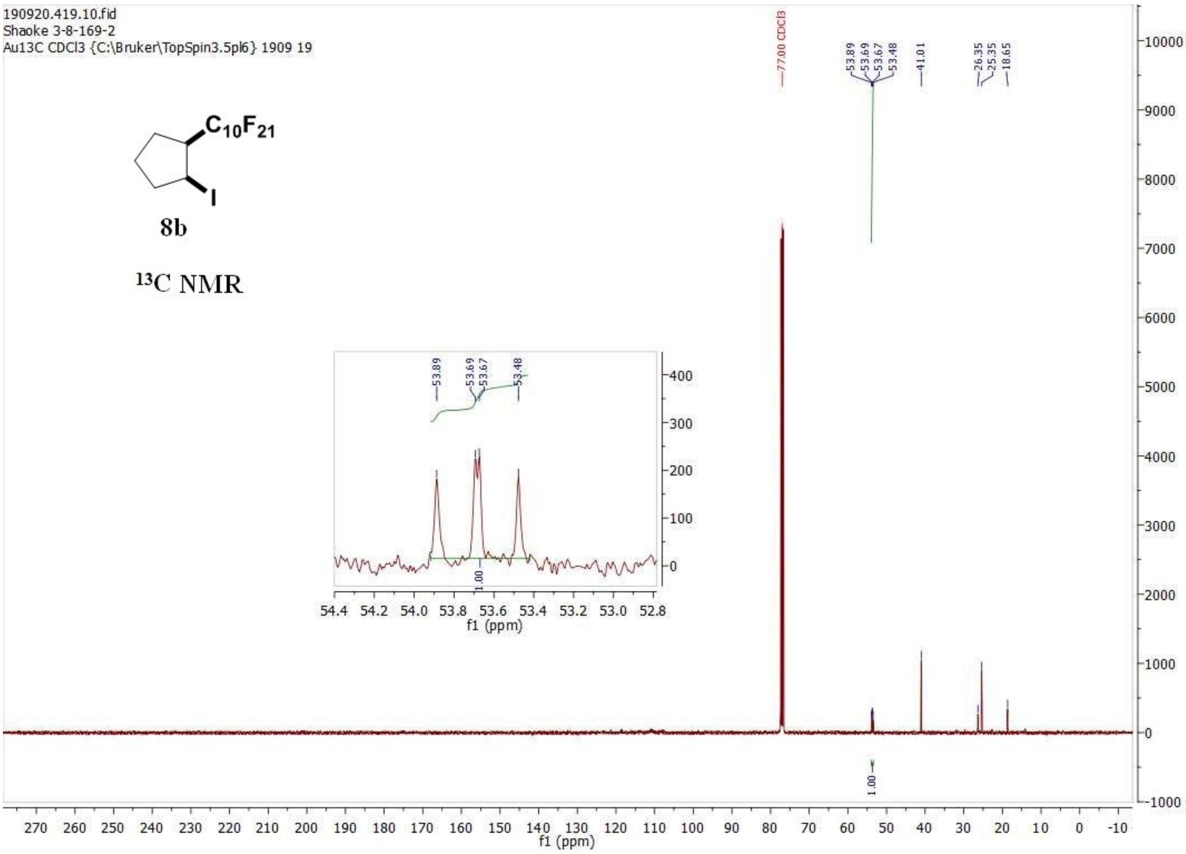

191210.f326.10.fid
Shaoke, 4-20-8-3
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1912 26
-58.43
-64.35
CF3
H
O
O
O
HO
H
4p
E/Z = 86:14
Reaction mixture
19F NMR
CF3I
(Z)
(E)
F
F
Internal standard
0.10
0.64
2.00
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

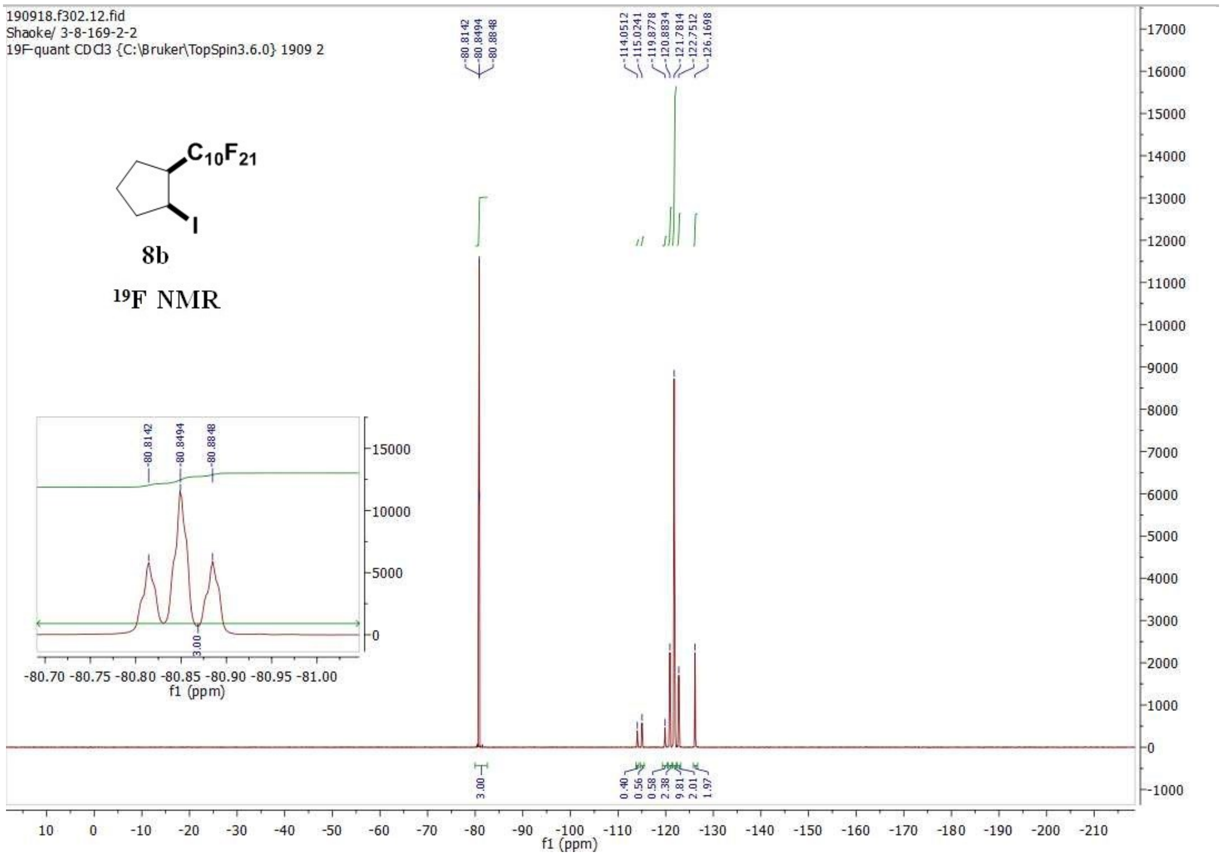
8.5 Spectra of others






190919.f306.10.fid
Shaoke 3-8-169-2-2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 1909 6
C10F21
I
8b
1H NMR
7.26 CDCl3
4.53
4.51
4.50
4.50
4.49
4.47
3.31
3.28
3.23
3.19
3.17
3.16
3.15
3.11
2.26
2.21
2.19
2.16
2.13
2.11
2.03
1.98
1.95
1.94
1.90
1.87
1.84
1.81
1.80
1.78
1.74
1.53 HDO
1.06
1.09
6.00
f1 (ppm)
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
-500 0 500 1000 1500 2000 2500 3000 3500 4000 4500 5000 5500
4.5 4.0 3.5 3.0 2.5 2.0
0 500


190920.419.10.fid
Shaoke 3-8-169-2
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1909 19
C10F21
I
8b
13C NMR
77.00 CDCl3
53.89
53.69
53.67
53.48
41.01
26.35
25.35
18.65
1.00
f1 (ppm)
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
-1000 0 1000 2000 3000 4000 5000 6000 7000 8000 9000 10000
54.4 54.2 54.0 53.8 53.6 53.4 53.2 53.0 52.8
0 100 200 300 400


190918.f302.12.fid
Shaoke/ 3-8-169-2-2
19F-quant CDCl3 {C:\Bruker\TopSpin3.6.0} 1909 2
C10F21
I
8b
19F NMR
-80.8142
-80.8494
-80.8848
-114.0512
-115.0241
-119.8778
-120.8834
-121.7814
-122.7512
-126.1698
3.00
0.40
0.56
0.58
2.38
9.81
2.01
1.97
-80.70 -80.75 -80.80 -80.85 -80.90 -80.95 -81.00
f1 (ppm)
15000
10000
5000
0
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)
17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000