

Supporting Information

Metal-free photocatalytic intermolecular trifluoromethylation-gem-difluoroallylation of unactivated alkenes

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1. General Information:

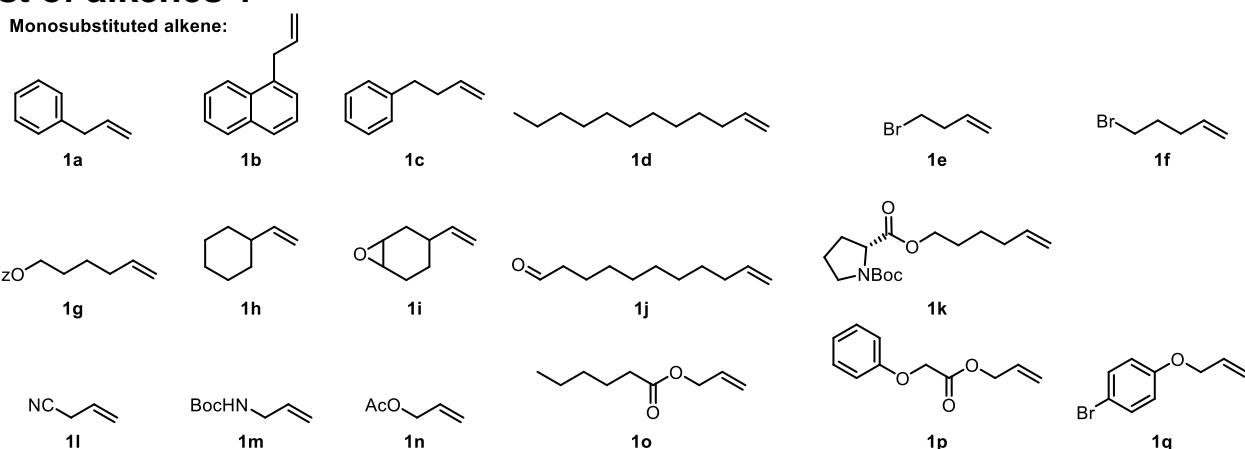
Unless otherwise noted, all reactions were performed in a 4 mL test tube at room temperature under nitrogen atmosphere. Photo-irradiation was carried out with a 30 W blue LED. Solvents were dried by passage through an activated alumina column under argon. Liquids and solutions were transferred via syringe. For chromatography, 200-300 mesh silica gel (Qingdao, China) was employed. ^1H NMR and ^{13}C NMR spectra were measured in CDCl_3 and recorded on Bruker ARX 600 spectrometer. ^{19}F NMR spectra were measured in CDCl_3 and recorded on Varian 400 spectrometer. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of CDCl_3 (7.26), to the carbon resonance of CDCl_3 (77.16). Coupling constants (J) were given in Hertz (Hz). The term m, t, d, s, dd referred to multiplet, triplet, doublet, singlet, doublet or doublet. Gas chromatography-mass spectrometry (GC-MS) was performed on an Thermo Fisher Trace ISQ 7000. Gas chromatography (GC) was performed on a Shimadzu GC 2010-pro system equipped with a split-mode capillary injection system and flame ionization detectors. High-resolution mass spectra (HRMS) were recorded on a Bruker Daltonics MicroTOF-Q II from Sichuan University. α -CF₃ alkenes were prepared according to literature reported procedures.⁶⁻⁸ All reagents and solvents were commercially available and directly used without any further purification.

Reactions were monitored through thin layer chromatography [Merck 60 F254 precoated silica gel plate (0.2 mm thickness)]. Subsequent to elution, spots were visualized using UV radiation (254 nm) on Spectroline Model ZF-7 254 nm. Other visualization methods include staining with a basic solution of potassium permanganate or acidic solution of ceric ammonium molybdate, followed by heating. Visible light irradiation was performed with a 30 W Led lamp at $\lambda_{\text{ir}} = 450 \pm 10$ nm for photocatalytic reactions. The Led lamps used in this research were brought from Sichuan Zhiyan Technology Co., Ltd. (Figure S1)

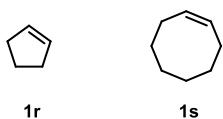
2. Preparation of Substrates:

List of alkenes 1

Monosubstituted alkene:



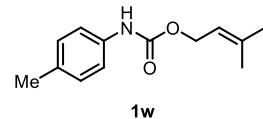
Cyclic alkene:



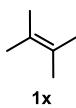
Disubstituted alkene:



Trisubstituted alkene:



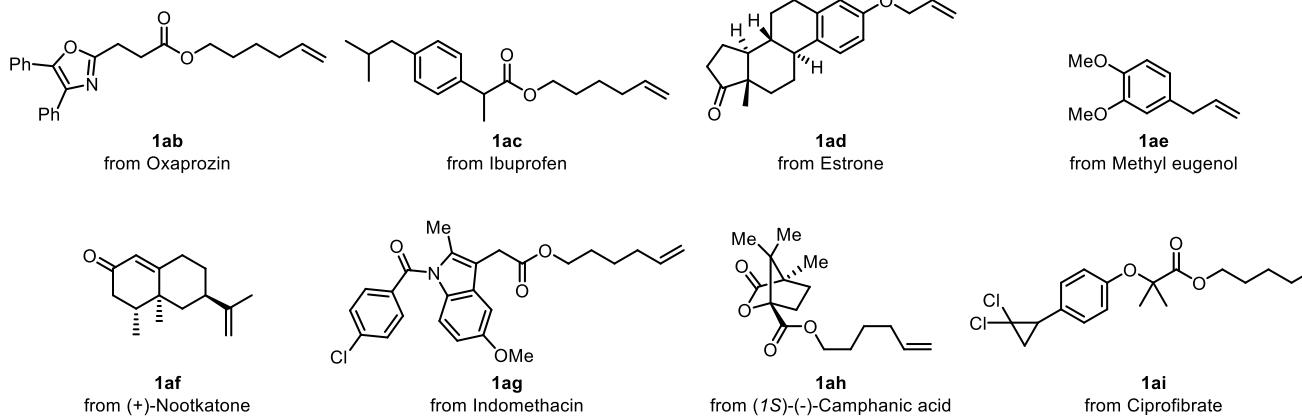
Tetrasubstituted alkene:



Enamide and enol ether:

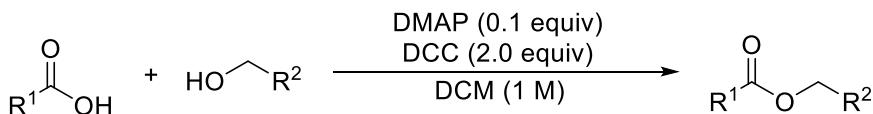


Natural product and drug derivatives:



Alkenes **1a-1j**, **1l-1o**, **1r-1u**, **1x-1aa**, **1ae**, **1af** are commercially available compounds. Alkenes **1k**, **1p**, **1q**, **1v**, **1w**, **1ab-1ad**, **1ag-1ai** were prepared according to the reported literatures.¹⁻³

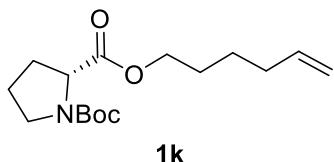
Method A:(For compounds **1k**, **1p**, **1v**, **1ab**, **1ac**, **1ag**, **1ah**, **1ai**)



According to the reported procedure,¹ a flame-dried round-bottomed flask was charged with acid (5.0 mmol, 1.0 equiv), alcohol (10 mmol, 2.0 equiv), 4-(dimethylamino)pyridine (61.0 mg, 0.5 mmol, 0.1 equiv), and dry DCM (5.0 mL, 1 M). The reaction mixture was then cooled to

0 °C, and dicyclohexylcarbodiimide solution (1M in CH₂Cl₂, 10 mL, 10 mmol, 2.0 equiv) was added dropwise. The reaction mixture was allowed to warm at 23 °C, stirred for 12 hours at this temperature, filtered. The filtrate was concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the desired product.

1-(*Tert*-butyl) 2-(hex-5-en-1-yl) (*R*)-pyrrolidine-1,2-dicarboxylate (1k)



1k was prepared according to **General Method A** from commercially available (*tert*-butoxycarbonyl)-D-proline and hex-5-en-1-ol.

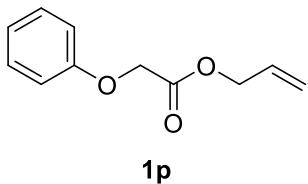
TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 5.75 (tt, J = 10.7, 6.8 Hz, 1H), 4.98 (d, J = 17.1 Hz, 1H), 4.93 (t, J = 8.6 Hz, 1H), 4.40 – 3.80 (m, 3H), 3.78 – 3.19 (m, 2H), 2.27 – 2.09 (m, 1H), 2.05 (dd, J = 13.7, 6.7 Hz, 2H), 1.91 (ddd, J = 20.0, 13.3, 7.0 Hz, 2H), 1.65 – 1.59 (m, 2H), 1.52 – 1.40 (m, 3H), 1.38 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 173.3, 153.8, 138.1, 114.9, 79.8, 64.8, 59.2, 46.3, 33.2, 30.9, 28.3, 28.1, 25.1, 23.6.

This matched literature characterization.¹

Allyl 2-phenoxyacetate (1p)



1p was prepared according to **General Method A** from commercially available 2-phenoxyacetic acid and prop-2-en-1-ol.

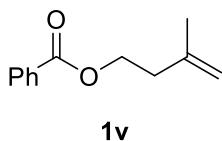
TLC R_f = 0.60 (Hexane/EtOAc = 50:1, v/v).

¹H NMR (400 MHz, CDCl₃) δ 7.35 – 7.27 (m, 2H), 7.00 (t, J = 7.4 Hz, 1H), 6.92 (d, J = 7.9 Hz, 2H), 5.93 (ddt, J = 16.3, 10.4, 5.8 Hz, 1H), 5.34 (dd, J = 17.2, 1.4 Hz, 1H), 5.27 (dd, J = 10.4, 1.2 Hz, 1H), 4.71 (d, J = 5.8 Hz, 2H), 4.66 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 168.7, 157.8, 131.4, 129.6, 121.8, 119.1, 114.7, 65.9, 65.4.

This matched literature characterization.¹

3-Methylbut-3-en-1-yl benzoate (1v)



1v was prepared according to **General Method A** from commercially available benzoic acid and 3-methylbut-3-en-1-ol.

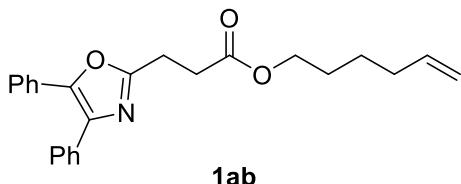
TLC R_f = 0.80 (Hexane/EtOAc = 50:1, v/v).

^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, J = 7.4 Hz, 2H), 7.59 – 7.51 (m, 1H), 7.43 (t, J = 7.6 Hz, 2H), 4.83 (d, J = 10.9 Hz, 2H), 4.44 (t, J = 6.8 Hz, 2H), 2.49 (t, J = 6.8 Hz, 2H), 1.82 (s, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 166.6, 141.7, 132.9, 130.4, 129.6, 128.4, 112.4, 63.2, 36.8, 22.6.

This matched literature characterization.²

Hex-5-en-1-yl 3-(4,5-diphenyloxazol-2-yl)propanoate (1ab)



1ab was prepared according to **General Method A** from commercially available oxaprozin and hex-5-en-1-ol.

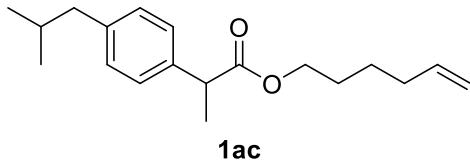
TLC R_f = 0.45 (Hexane/EtOAc = 20:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.66 – 7.61 (m, 2H), 7.60 – 7.54 (m, 2H), 7.39 – 7.29 (m, 6H), 5.76 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 4.99 (ddd, J = 17.1, 3.4, 1.6 Hz, 1H), 4.94 (ddt, J = 10.2, 2.1, 1.1 Hz, 1H), 4.13 (t, J = 6.6 Hz, 2H), 3.19 (t, J = 7.5 Hz, 2H), 2.91 (t, J = 7.6 Hz, 2H), 2.05 (dd, J = 14.3, 7.2 Hz, 2H), 1.71 – 1.58 (m, 2H), 1.50 – 1.37 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 172.1, 161.8, 145.4, 138.3, 135.1, 132.5, 129.0, 128.6, 128.5, 128.4, 128.1, 127.9, 126.5, 114.9, 64.8, 33.3, 31.2, 28.0, 25.2, 23.6.

This matched literature characterization.³

Hex-5-en-1-yl 2-(4-isobutylphenyl)propanoate (1ac)



1ac was prepared according to **General Method A** from commercially available ibuprofen and hex-5-en-1-ol.

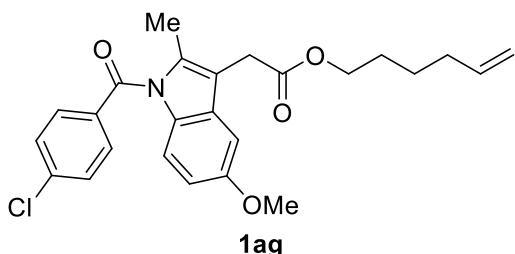
TLC R_f = 0.70 (Hexane/EtOAc = 50:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.20 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 5.73 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 4.97 (ddd, *J* = 17.1, 3.4, 1.7 Hz, 1H), 4.93 (ddt, *J* = 10.2, 2.0, 1.1 Hz, 1H), 4.06 (t, *J* = 6.6 Hz, 2H), 3.68 (q, *J* = 7.2 Hz, 1H), 2.44 (d, *J* = 7.2 Hz, 2H), 2.01 (dd, *J* = 14.3, 7.3 Hz, 2H), 1.84 (dp, *J* = 13.6, 6.8 Hz, 1H), 1.70 – 1.52 (m, 2H), 1.48 (d, *J* = 7.2 Hz, 3H), 1.35 (dq, *J* = 15.1, 7.6 Hz, 2H), 0.89 (d, *J* = 6.6 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 174.8, 140.5, 138.4, 137.9, 129.3, 127.1, 114.7, 64.5, 45.2, 45.0, 33.2, 30.2, 28.0, 25.0, 22.4, 18.5.

This matched literature characterization.⁴

Hex-5-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (1ag)



1ag was prepared according to **General Method A** from commercially available indomethacin and hex-5-en-1-ol.

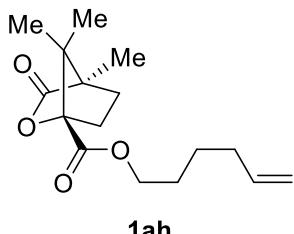
TLC R_f = 0.40 (Hexane/EtOAc = 20:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.66 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 6.96 (d, *J* = 2.5 Hz, 1H), 6.86 (d, *J* = 9.0 Hz, 1H), 6.67 (dd, *J* = 9.0, 2.5 Hz, 1H), 5.74 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.08 – 4.89 (m, 2H), 4.10 (t, *J* = 6.7 Hz, 2H), 3.83 (s, 3H), 3.66 (s, 2H), 2.39 (s, 3H), 2.03 (dd, *J* = 14.3, 7.2 Hz, 2H), 1.63 (dt, *J* = 14.9, 6.7 Hz, 2H), 1.46 – 1.33 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 170.9, 168.3, 156.0, 139.3, 138.2, 135.9, 133.9, 131.2, 130.8, 130.7, 129.1, 115.0, 114.9, 112.7, 111.7, 101.3, 65.0, 55.7, 33.2, 30.4, 28.0, 25.2, 13.4.

This matched literature characterization.³

Hex-5-en-1-yl (1*S*,4*R*)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (1ah)



1ah was prepared according to **General Method A** from commercially available (1*S*)-(–)-Camphanic acid and hex-5-en-1-ol.

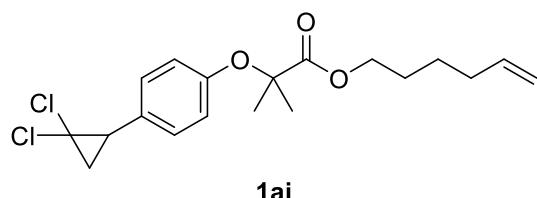
TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 5.77 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 5.00 (dd, J = 17.1, 1.3 Hz, 1H), 4.95 (d, J = 10.2 Hz, 1H), 4.22 (td, J = 6.6, 2.9 Hz, 2H), 2.47 – 2.32 (m, 1H), 2.07 (q, J = 7.1 Hz, 2H), 2.04 – 1.96 (m, 1H), 1.91 (ddd, J = 13.6, 10.9, 4.6 Hz, 1H), 1.84 – 1.57 (m, 3H), 1.50 – 1.39 (m, 2H), 1.10 (s, 3H), 1.04 (s, 3H), 0.94 (s, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 178.2, 167.6, 138.1, 115.1, 91.2, 65.5, 54.8, 54.1, 33.2, 30.6, 29.0, 28.0, 25.1, 16.8, 16.8, 9.7.

This matched literature characterization.³

Hex-5-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (1ai)



1ai was prepared according to **General Method A** from commercially available ciprofibrate and hex-5-en-1-ol.

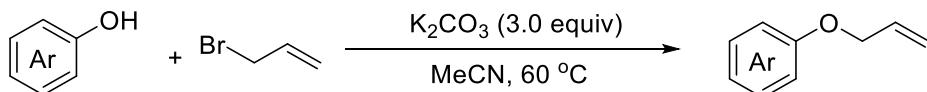
TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.10 (d, J = 8.6 Hz, 2H), 6.80 (d, J = 8.7 Hz, 2H), 5.72 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 5.03 – 4.91 (m, 2H), 4.15 (t, J = 6.6 Hz, 2H), 2.82 (dd, J = 10.6, 8.4 Hz, 1H), 2.00 (q, J = 7.2 Hz, 2H), 1.93 (dd, J = 10.7, 7.4 Hz, 1H), 1.86 – 1.67 (m, 1H), 1.65 – 1.53 (m, 2H), 1.60 (s, 6H), 1.33 (dq, J = 15.1, 7.6 Hz, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 174.3, 155.0, 138.2, 129.6, 128.0, 118.5, 114.9, 79.2, 65.4, 60.9, 60.4, 34.8, 33.2, 27.8, 25.8, 25.5, 25.0.

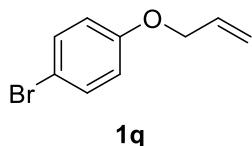
This matched literature characterization.²

Method B:(For compounds **1q**, **1ad**)



According to the reported procedure,² a flame-dried round-bottomed flask was charged with aryl phenol (5.0 mmol, 1.0 equiv), allyl bromide (10 mmol, 2.0 equiv), K_2CO_3 (2.07 g, 15.0 mmol, 3.0 equiv), and dry MeCN (20.0 mL, 0.25 M). The reaction mixture was allowed to heat to 60 °C, stirred for 16 hours at this temperature, filtered. The filtrate was concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the desired product.

1-(Allyloxy)-4-bromobenzene (1q)



1q was prepared according to **General Method B** from commercially available 4-bromophenol and 3-bromoprop-1-ene.

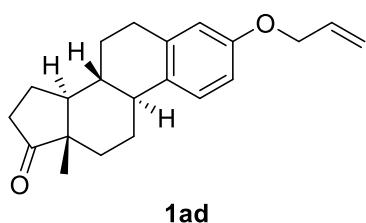
TLC R_f = 0.90 (Hexane/EtOAc = 50:1, v/v).

¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, J = 9.0 Hz, 2H), 6.80 (d, J = 9.0 Hz, 2H), 6.03 (ddd, J = 22.5, 10.5, 5.3 Hz, 1H), 5.40 (dd, J = 17.3, 1.4 Hz, 1H), 5.30 (dd, J = 10.5, 1.2 Hz, 1H), 4.51 (d, J = 5.3 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 157.7, 132.9, 132.3, 117.9, 116.6, 113.0, 69.0.

This matched literature characterization.²

(8*R*,9*S*,13*S*,14*S*)-3-(Allyloxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[a]phenanthren-17-one (1ad)



1ad was prepared according to **General Method B** from commercially available estrone and 3-bromoprop-1-ene.

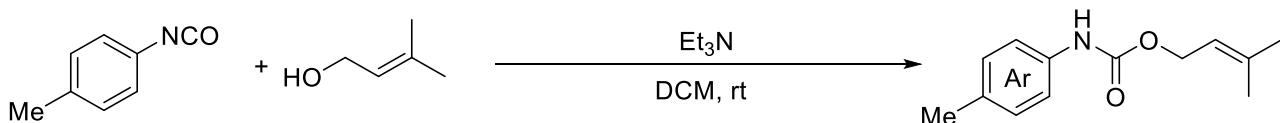
TLC R_f = 0.70 (Hexane/EtOAc = 50:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.19 (d, J = 8.6 Hz, 1H), 6.73 (dd, J = 8.6, 2.7 Hz, 1H), 6.66 (d, J = 2.7 Hz, 1H), 6.05 (ddt, J = 17.2, 10.5, 5.3 Hz, 1H), 5.41 (dq, J = 17.3, 1.5 Hz, 1H), 5.27 (dd, J = 10.5, 1.4 Hz, 1H), 4.51 (dt, J = 5.3, 1.4 Hz, 2H), 3.03 – 2.74 (m, 2H), 2.50 (dd, J = 19.1, 8.5 Hz, 1H), 2.42 – 2.32 (m, 1H), 2.25 (td, J = 10.8, 4.2 Hz, 1H), 2.19 – 2.09 (m, 1H), 2.08 – 2.03 (m, 1H), 2.02 – 1.97 (m, 1H), 1.97 – 1.82 (m, 1H), 1.76 – 1.35 (m, 6H), 0.91 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 156.6, 137.7, 133.5, 132.2, 126.3, 117.5, 114.8, 112.3, 68.8, 50.4, 48.0, 44.0, 38.4, 35.9, 31.6, 29.7, 26.6, 25.9, 21.6, 13.9.

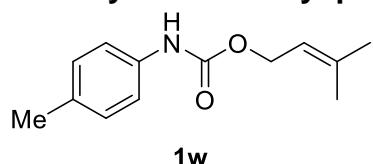
This matched literature characterization.²

Method C:(For compounds 1w)



According to the reported procedure,⁵ a flame-dried was charged with phenyl isocyanate (2.5 mmol, 1 equiv), dry DCM (5 mL), Et₃N (7.5 mmol, 1.1 equiv) and alcohol (2.5 mmol, 1 equiv). The reaction mixture was stirred at room temperature until the alcohol was fully consumed (monitored by TLC). The reaction mixture was washed with 1 M HCl, water and brine and then dried with Na₂SO₄. Then, the organic phase was concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to afford the desired product.

3-Methylbut-2-en-1-yl p-tolylcarbamate (1w)



1w was prepared according to **General Method C** from commercially available 1-isocyanato-4-methylbenzene and 3-methylbut-2-en-1-ol.

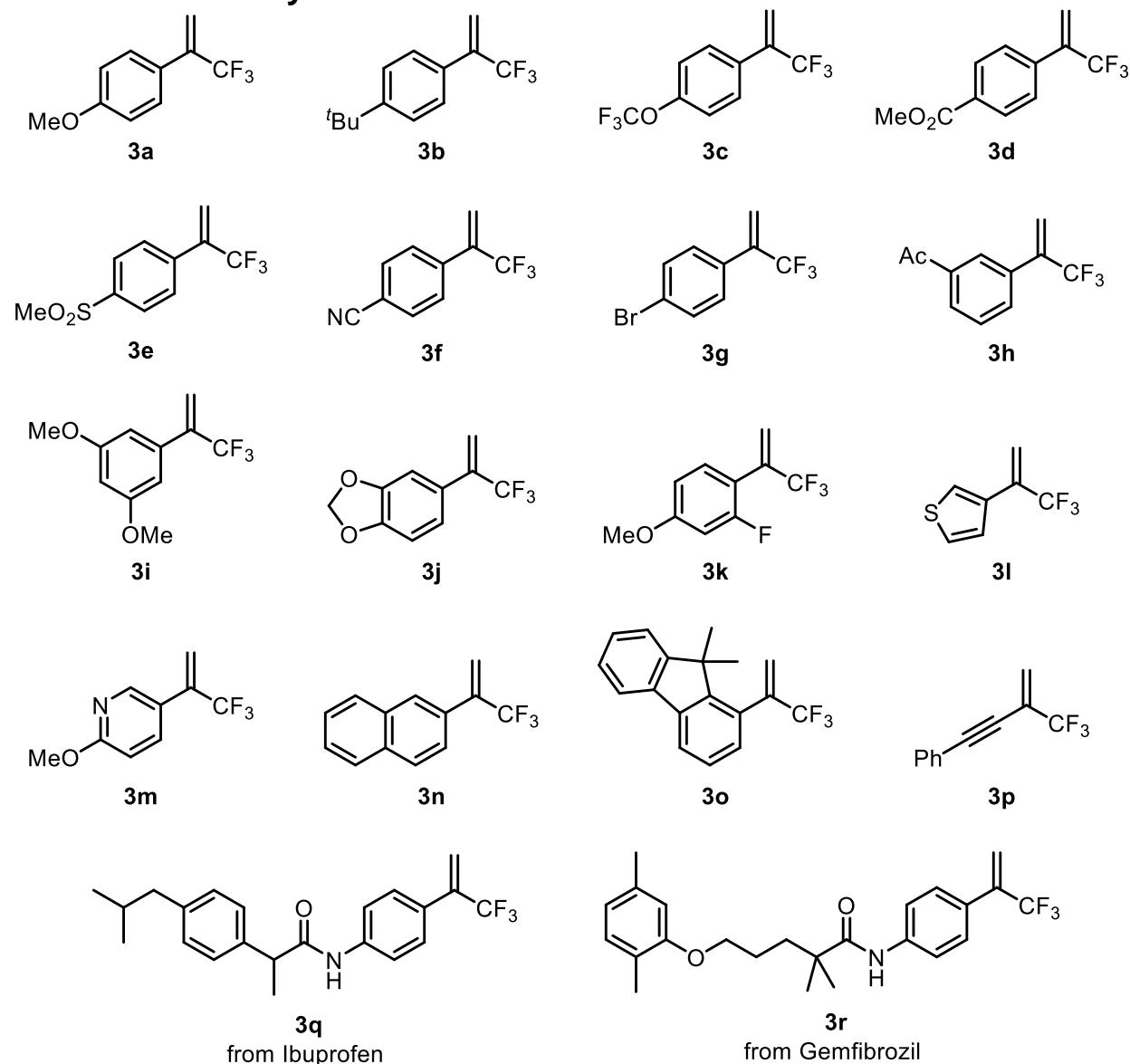
TLC R_f = 0.40 (Hexane/EtOAc = 10:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.25 (d, *J* = 9.7 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 6.51 (s, 1H), 5.39 (t, *J* = 7.2 Hz, 1H), 4.65 (d, *J* = 7.2 Hz, 2H), 2.30 (s, 3H), 1.78 (s, 3H), 1.74 (s, 3H).

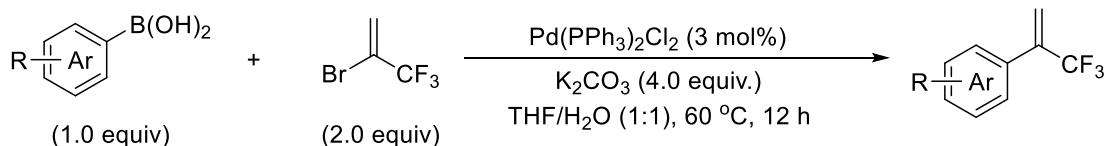
¹³C NMR (151 MHz, CDCl₃) δ 139.4, 135.4, 133.0, 129.6, 118.8, 61.9, 25.8, 20.8, 18.1.

This matched literature characterization.⁵

List of trifluoromethyl alkenes 3



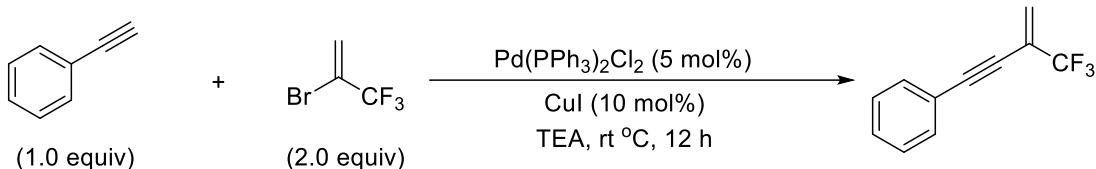
Method D:(For compounds 3a-3o)



According to the reported procedure,⁶ to a 100 mL Schlenk tube equipped a magnetic stir bar, boronic acid (5.0 mmol, 1.0 equiv), and Pd(PPh₃)₂Cl₂ (63.2 mg, 3 mol%) were added. The vessel was evacuated and filled with argon (three times), and then THF (20 mL) and aqueous K₂CO₃ (2.0 M, 10 mL, 4.0 equiv) were added. After the addition of 2-bromo-3,3,3-trifluoropropene (1.04 mL, 10 mmol, 2.0 equiv), the reaction mixture was stirred at 60 °C overnight under an argon atmosphere. The resultant mixture was cooled to room temperature, quenched with saturated aqueous NH₄Cl, and extracted with EtOAc (3 × 15 mL). The combined organic phases were dried over anhydrous Na₂SO₄, filtered, and concentrated.

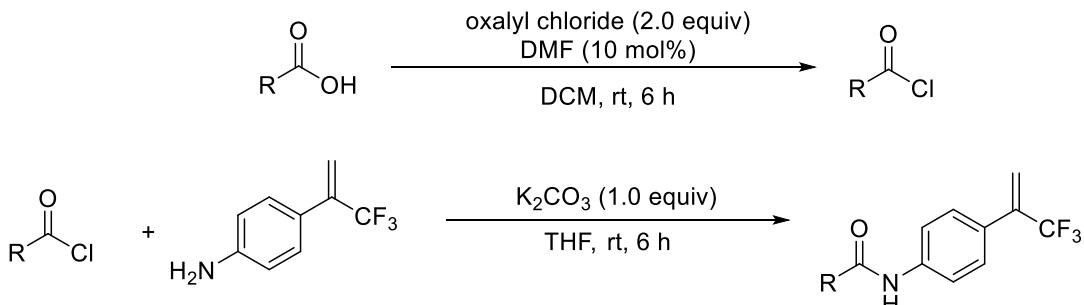
under reduced pressure. The residue was purified by column chromatography on silica gel (Hexane/EtOAc) to give the desired trifluoromethyl alkene.

Procedure E: (For compounds 3p)



According to the reported procedure,⁷ CuI (57.2 mg, 10 mol%) and Pd(PPh_3)₂Cl₂ (105.3 mg, 5 mol%) were dissolved in Et₃N (30 mL) under argon at room temperature. To the solution were added 2-bromo-3,3,3-trifluoroprop-1-ene (0.62 mL, 6.0 mmol, 2.0 equiv) and phenylacetylene (0.33 mL, 3.0 mmol, 1.0 equiv). The reaction mixture was left to stir at room temperature for 12 hours. The resultant mixture was diluted with saturated aqueous NH₄Cl (20 mL) followed by extraction with CH₂Cl₂ (3 X 20 mL). The combined organic phases were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (Hexane/EtOAc = 100 : 1) to give the desired enyne **3p**.

Method E:(For compounds 3q-3r)

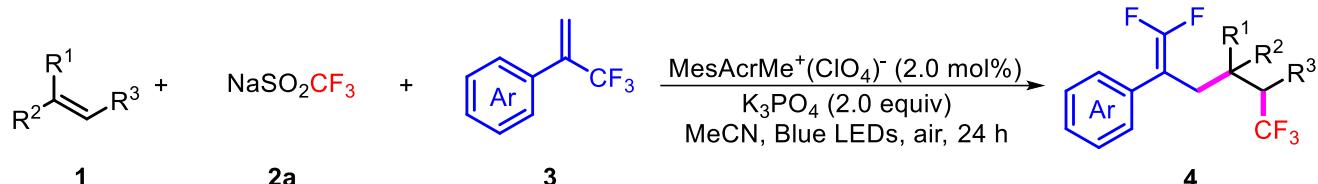


According to the reported procedure,⁸ to a mixture of acid (5.0 mmol, 1.0 equiv) and oxalyl chloride (0.847 mL, 10 mmol, 2.0 equiv) in dry CH₂Cl₂ (20 mL) was added dropwise DMF (39 μ L, 10 mol%). The reaction mixture was stirred at room temperature for 6 hours. Removal of the solvent *in vacuo* afforded the desired acid chloride which was used in the next step without further purification.

To a mixture of 3-(3,3,3-trifluoroprop-1-en-2-yl)aniline (0.94 g, 5.0 mmol, 1.0 equiv) and K₂CO₃ (0.69 g, 5.0 mmol, 1.0 equiv) in dry THF (10 mL) was added dropwise a solution of the freshly prepared acid chloride (5.0 mmol, 1.0 equiv) in dry THF (10 mL). This mixture was stirred at room temperature for 6 hours before water was added to quench the reaction. The resultant mixture was extracted with EtOAc (3 X 20 mL). The combined organic phases were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The

resultant crude product was purified by column chromatography on silica gel (Hexane/EtOAc) to give the desired trifluoromethyl alkene.

3. Standard Reaction Conditions:



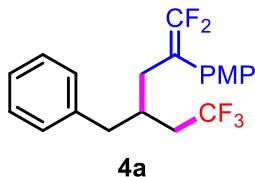
To an oven-dried 4 mL reaction vial equipped with a stir bar was added MesAcrMe⁺(ClO₄)⁻ (0.8 mg, 0.002 mmol, 2 mol%), NaSO₂CF₃ (46.8 mg, 0.30 mmol, 3.0 equiv), and K₃PO₄ (42.4 mg, 0.20 mmol, 2.0 equiv). The vial was then charged with the alkene **1** (0.30 mmol, 3.0 equiv) and CF₃ alkene **3** (0.10 mmol, 1.0 equiv) in anhyd MeCN (1 mL) *via* a syringe. The cap was sealed with Parafilm®, and the solution was irradiated with a 30 W blue LED light at room temperature for 24 hours. The temperature of the reaction was maintained at approximately 27 °C *via* a fan. The solution was stirred vigorously while being irradiated. Once judged to be complete, the solution was transferred to a separatory funnel and diluted with deionized H₂O (20 mL) and Et₂O (20 mL). The layers were separated, and the aq layer was extracted with Et₂O (3 X 20 mL). The combined organic layers were washed with deionized H₂O (2 X 50 mL) followed by brine (100 mL). The combined organic layers were dried (Na₂SO₄), and the solvent was removed *in vacuo* by rotary evaporation. Further purification was accomplished by SiO₂ column chromatography (gradient Hexane/EtOAc) to give the desired product.



Figure S1. Blue LED reactors

4. Characterization Data of Products:

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4a)



4a was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4a** (31.1 mg, 84% yield) was isolated as a clear oil.

TLC $R_f = 0.50$ (Hexane/EtOAc = 50:1, v/v).

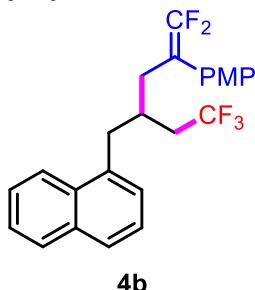
^1H NMR (600 MHz, CDCl_3) δ 7.26 (t, $J = 7.4$ Hz, 2H), 7.21 (t, $J = 7.3$ Hz, 1H), 7.08 (dd, $J = 8.7$, 1.0 Hz, 2H), 7.00 (d, $J = 7.1$ Hz, 2H), 6.87 (d, $J = 8.8$ Hz, 2H), 3.82 (s, 3H), 2.67 (dd, $J = 13.8$, 7.0 Hz, 1H), 2.61 (dd, $J = 13.8$, 7.2 Hz, 1H), 2.49 (ddd, $J = 14.5$, 5.7, 3.6 Hz, 1H), 2.39 (ddd, $J = 14.6$, 5.7, 3.6 Hz, 1H), 2.01 (dtt, $J = 33.3$, 13.3, 6.6 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 154.0 (dd, $J = 290.2$, 286.3 Hz), 139.0, 129.3 (t, $J = 2.7$ Hz), 129.1, 128.5, 127.1 (q, $J = 277.6$ Hz), 126.4, 124.6 – 124.5 (m), 114.1, 90.0 (dd, $J = 21.1$, 14.2 Hz), 55.3, 39.7, 36.5 (q, $J = 27.6$ Hz), 32.6, 31.5.

^{19}F NMR (565 MHz, CDCl_3) δ -62.78 (t, $J = 11.6$ Hz, 3F), -91.19 (d, $J = 44.7$ Hz, 1F), -91.57 (d, $J = 44.7$ Hz, 3F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{20}\text{H}_{19}\text{F}_5\text{ONa}$ 393.1248; found 393.1257.

1-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)naphthalene (4b)



4b was prepared according to the general procedure from 1-allylnaphthalene **1b** (50.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4b** (31.5 mg, 75% yield) was isolated as a clear oil.

TLC $R_f = 0.50$ (Hexane/EtOAc = 50:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.83 (d, $J = 8.2$ Hz, 1H), 7.74 (d, $J = 8.2$ Hz, 1H), 7.51 (d, $J = 8.5$ Hz, 1H), 7.43 (t, $J = 7.5$ Hz, 1H), 7.41 – 7.33 (m, 1H), 7.28 (t, $J = 7.6$ Hz, 1H), 7.20 (d, $J = 6.9$ Hz, 1H), 7.04 (d, $J = 8.0$ Hz, 2H), 6.79 (d, $J = 8.7$ Hz, 2H), 3.80 (s, 3H), 3.18 (dd, $J = 14.0$, 6.9

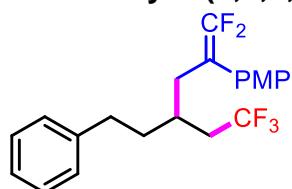
Hz, 1H), 2.98 (dd, J = 14.0, 8.3 Hz, 1H), 2.74 – 2.57 (m, 1H), 2.42 (dd, J = 14.6, 7.6 Hz, 1H), 2.23 (dt, J = 14.2, 7.1 Hz, 1H), 2.17 – 1.88 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.8, 154.0 (dd, J = 290.8, 285.9 Hz), 135.1, 134.0, 131.8, 129.2 (t, J = 3.2 Hz), 128.8, 127.7, 127.4, 127.1 (d, J = 277.2 Hz), 125.9, 125.6, 125.2, 124.2 (t, J = 3.4 Hz), 123.5, 114.0, 89.9 (dd, J = 21.1, 13.9 Hz), 55.3, 39.1 – 35.2 (m), 31.6, 31.5, 29.7.

^{19}F NMR (565 MHz, CDCl_3) δ -62.48 (t, J = 11.9 Hz, 3F), -90.90 (d, J = 44.8 Hz, 1F), -91.19 (d, J = 44.6 Hz, 1F).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{24}\text{H}_{21}\text{F}_5\text{ONa}$ 443.1405; found 443.1412.

1-methoxy-4-(1,1,6,6,6-pentafluoro-4-phenethylhex-1-en-2-yl)benzene (4c)



4c

4c was prepared according to the general procedure from but-3-en-1-ylbenzene **1c** (39.6 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4c** (30.2 mg, 79% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

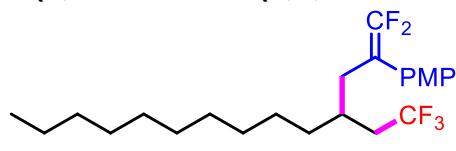
^1H NMR (600 MHz, CDCl_3) δ 7.25 (t, J = 7.5 Hz, 2H), 7.17 (dd, J = 17.9, 7.7 Hz, 3H), 7.07 (d, J = 7.2 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 2.68 – 2.34 (m, 4H), 2.29 – 1.99 (m, 2H), 1.79 (dt, J = 13.0, 6.5 Hz, 1H), 1.68 (dd, J = 14.8, 7.5 Hz, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 154.0 (dd, J = 289.7, 286.3 Hz), 141.5, 129.4 (t, J = 2.6 Hz), 128.4, 128.3, 127.1 (q, J = 277.6 Hz), 126.0, 125.1 – 124.4 (m), 114.1, 90.1 (dd, J = 21.4, 14.5 Hz), 55.3, 36.9 (q, J = 27.4 Hz), 34.7, 32.2, 31.9, 30.3.

^{19}F NMR (565 MHz, CDCl_3) δ -63.13 (t, J = 11.9 Hz, 3F), -91.31 (d, J = 44.9 Hz, 1F), -91.67 (d, J = 44.9 Hz, 1F).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{F}_5\text{ONa}$ 407.1405; found 407.1413.

1-(1,1-difluoro-4-(2,2,2-trifluoroethyl)tetrade-1-en-2-yl)-4-methoxybenzene (4d)



4d

4d was prepared according to the general procedure from dodec-1-ene **1d** (50.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4d** (23.1 mg, 55% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

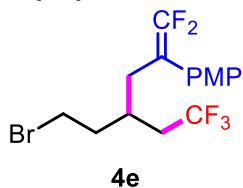
^1H NMR (600 MHz, CDCl_3) δ 7.20 (d, J = 8.0 Hz, 2 H), 6.90 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 2.61 – 2.29 (m, 2H), 2.14 – 1.85 (m, 2H), 1.70 (dt, J = 13.1, 6.6 Hz, 1H), 1.42 – 1.07 (m, 18H), 0.88 (t, J = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 154.0 (dd, J = 289.9, 286.1 Hz), 129.3 (t, J = 2.6 Hz), 127.2 (d, J = 277.1 Hz), 125.4 – 124.6 (m), 114.0, 90.2 (dd, J = 21.6, 14.1 Hz), 55.2, 36.9 (q, J = 27.1 Hz), 32.8, 32.0, 31.9, 30.5, 29.6, 29.5, 29.3, 25.7, 22.7, 14.1.

^{19}F NMR (565 MHz, CDCl_3) δ -63.23 (t, J = 12.5 Hz, 3F), -91.61 (d, J = 45.3 Hz, 1F), -91.93 (d, J = 46.4 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{23}\text{H}_{33}\text{F}_5\text{ONa}$ 443.2344; found 443.2352.

1-(4-(2-bromoethyl)-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (**4e**)



4e was prepared according to the general procedure from 4-bromobut-1-ene **1e** (40.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4e** (27.8 mg, 72% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

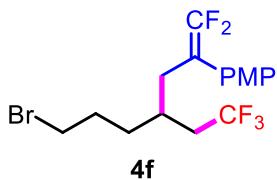
^1H NMR (600 MHz, CDCl_3) δ 7.21 (d, J = 7.8 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 3.46 – 3.24 (m, 2H), 2.47 (d, J = 5.4 Hz, 2H), 2.17 – 2.07 (m, 2H), 1.98 – 1.82 (m, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.0, 154.1 (dd, J = 290.2, 286.8 Hz), 129.4 (t, J = 3.0 Hz), 126.8 (q, J = 277.5 Hz), 124.6 – 124.2 (m), 114.2, 89.7 (dd, J = 21.1, 14.6 Hz), 55.3, 36.6 (q, J = 28.0 Hz), 36.1, 31.6, 29.9, 29.7.

^{19}F NMR (565 MHz, CDCl_3) δ -62.97 (t, J = 12.3 Hz, 3F), -90.75 (d, J = 43.8 Hz, 1F), -91.27 (d, J = 44.4 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{15}\text{H}_{16}\text{BrF}_5\text{ONa}$ 409.0200; found 409.0211.

1-(7-bromo-1,1-difluoro-4-(2,2,2-trifluoroethyl)hept-1-en-2-yl)-4-methoxybenzene (**4f**)



4f was prepared according to the general procedure from 5-bromopent-1-ene **1f** (44.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4f** (33.2 mg, 83% yield) was isolated as a clear oil.

TLC $R_f = 0.50$ (Hexane/EtOAc = 50:1, v/v).

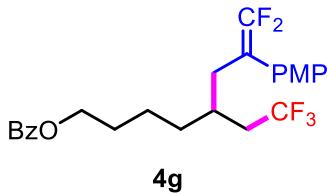
^1H NMR (600 MHz, CDCl_3) δ 7.21 (d, $J = 8.5$ Hz, 2H), 6.91 (d, $J = 8.7$ Hz, 2H), 3.82 (s, 3H), 3.56 – 2.97 (m, 2H), 2.66 – 2.44 (m, 1H), 2.40 (dd, $J = 14.5, 7.6$ Hz, 1H), 2.15 – 1.97 (m, 2H), 1.94 – 1.76 (m, 2H), 1.76 – 1.69 (m, 1H), 1.54 – 1.38 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.0, 154.1 (dd, $J = 290.0, 286.8$ Hz), 129.3 (t, $J = 2.6$ Hz), 127.0 (d, $J = 277.6$ Hz), 124.8 – 124.6 (m), 114.2, 89.9 (dd, $J = 21.4, 14.5$ Hz), 55.3, 37.0 (q, $J = 27.5$ Hz), 33.2, 32.0, 31.4, 30.1, 29.1.

^{19}F NMR (565 MHz, CDCl_3) δ -63.31 (t, $J = 12.0$ Hz, 3F), -91.16 (d, $J = 44.6$ Hz, 1F), -91.51 (d, $J = 44.7$ Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{18}\text{BrF}_5\text{ONa}$ 423.0353; found 423.0359.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl benzoate (4g)



4g was prepared according to the general procedure from hex-5-en-1-yl benzoate **1g** (61.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4g** (31.4 mg, 69% yield) was isolated as a clear oil.

TLC $R_f = 0.40$ (Hexane/EtOAc = 20:1, v/v).

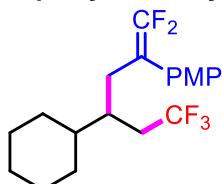
^1H NMR (600 MHz, CDCl_3) δ 8.11 – 7.84 (m, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 6.88 (d, $J = 8.8$ Hz, 2H), 4.28 (t, $J = 6.5$ Hz, 2H), 3.80 (s, 3H), 2.54 – 2.28 (m, 2H), 2.04 (qd, $J = 11.4, 6.3$ Hz, 2H), 1.81 – 1.71 (m, 2H), 1.69 (dt, $J = 13.5, 6.7$ Hz, 2H), 1.52 – 1.40 (m, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 166.6, 158.9, 154.0 (dd, $J = 289.9, 286.4$ Hz), 132.9, 130.4, 129.5, 129.3 (t, $J = 2.6$ Hz), 128.4, 127.1 (d, $J = 277.3$ Hz), 124.9 – 124.8 (m), 114.1, 90.1 (dd, $J = 21.1, 14.3$ Hz), 64.7, 55.2, 36.9 (q, $J = 27.2$ Hz), 32.5, 31.9, 30.5, 28.7, 22.4.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.23 (t, *J* = 12.6 Hz, 3F), -91.36 (d, *J* = 44.9 Hz, 1F), -91.76 (d, *J* = 44.9 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₄H₂₅F₅O₃Na 479.1616; found 479.1608.

1-(4-cyclohexyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4h)



4h

4h was prepared according to the general procedure from vinylcyclohexane **1h** (33.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4h** (31.1 mg, 86% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

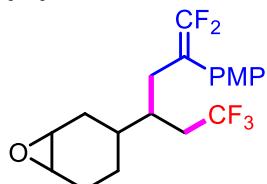
¹H NMR (600 MHz, CDCl₃) δ 7.20 (d, *J* = 7.9 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.46 (dd, *J* = 14.5, 7.6 Hz, 1H), 2.39 (ddd, *J* = 9.8, 6.1, 2.2 Hz, 1H), 2.24 – 2.02 (m, 1H), 1.97 – 1.81 (m, 1H), 1.74 (d, *J* = 13.1 Hz, 2H), 1.67 (d, *J* = 12.6 Hz, 1H), 1.53 (d, *J* = 12.4 Hz, 1H), 1.44 (dd, *J* = 19.8, 7.6 Hz, 2H), 1.25 – 0.86 (m, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 158.8, 154.0 (dd, *J* = 289.6, 285.9 Hz), 129.3 (t, *J* = 2.7 Hz), 127.5 (d, *J* = 277.0 Hz), 124.9 – 124.8 (m), 114.0, 90.4 (dd, *J* = 21.4, 13.8 Hz), 55.2, 39.1, 35.5, 34.1 (q, *J* = 27.4 Hz), 29.3, 29.0, 28.3, 26.6.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.59 (t, *J* = 12.6 Hz, 3F), -91.57 (d, *J* = 45.9 Hz, 1F), -92.34 (d, *J* = 45.3 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₉H₂₃F₅ONa 385.1561; found 385.1567.

3-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)-7-oxabicyclo[4.1.0]heptane (4i)



4i

4i was prepared according to the general procedure from 3-vinyl-7-oxabicyclo[4.1.0]heptane **1i** (37.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4i** (27.7 mg, 74% yield, dr = 1.6:1) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

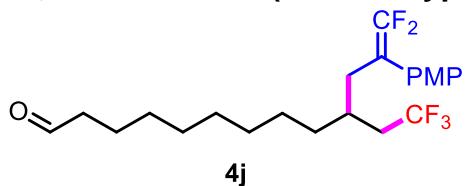
^1H NMR (400 MHz, CDCl_3) δ 7.19 (d, J = 7.7 Hz, 2H), 6.90 (dd, J = 8.8, 2.0 Hz, 2H), 3.82 (s, 3H), 3.14 (s, 2H), 2.43 (d, J = 6.8 Hz, 1H), 2.36 (d, J = 6.8 Hz, 1H), 2.17 (d, J = 14.4 Hz, 1H), 2.05 (ddd, J = 18.6, 11.2, 5.8 Hz, 1H), 1.96 – 1.81 (m, 2H), 1.78 – 1.56 (m, 2H), 1.33 – 0.93 (m, 4H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 155.0 (d, J = 290.1 Hz), 129.4 – 129.2 (m), 127.3 (d, J = 276.8 Hz), 124.5, 114.1, 90.1 (dd, J = 21.7, 14.3 Hz), 55.3, 52.5, 51.8, 34.8 – 34.0 (m), 28.8, 26.8, 25.4, 20.5, 19.3.

^{19}F NMR (565 MHz, CDCl_3) δ -63.68 (t, J = 12.3 Hz, 3F), -91.25 (d, J = 45.1 Hz, 1F), -92.05 (d, J = 11.2 Hz, 1F).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{19}\text{H}_{21}\text{F}_5\text{O}_2\text{Na}$ 399.1354; found 399.1362.

13,13-difluoro-12-(4-methoxyphenyl)-10-(2,2,2-trifluoroethyl)tridec-12-enal (4j)



4j was prepared according to the general procedure from undec-10-enal **1j** (50.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4j** (35.3 mg, 84% yield) was isolated as a clear oil.

TLC R_f = 0.45 (Hexane/EtOAc = 50:1, v/v).

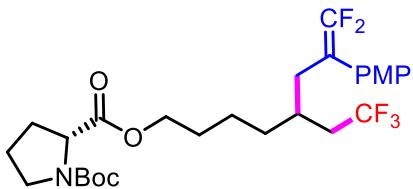
^1H NMR (600 MHz, CDCl_3) δ 9.76 (s, 1H), 7.20 (d, J = 8.0 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 2.42 (td, J = 7.4, 1.9 Hz, 4H), 2.13 – 1.89 (m, 2H), 1.69 (dd, J = 13.0, 6.5 Hz, 1H), 1.65 – 1.52 (m, 3H), 1.36 – 1.18 (m, 11H).

^{13}C NMR (151 MHz, CDCl_3) δ 202.9, 158.9, 154.0 (dd, J = 290.1, 286.3 Hz), 129.3 (t, J = 3.0 Hz), 127.2 (q, J = 277.2 Hz), 125.1 – 124.9 (m), 114.0, 90.2 (dd, J = 21.6, 14.2 Hz), 55.3, 43.9, 36.9 (q, J = 27.1 Hz), 32.8, 32.0, 30.5, 29.7, 29.5, 29.3, 29.2, 29.1, 25.6, 22.1.

^{19}F NMR (565 MHz, CDCl_3) δ -63.23 (t, J = 11.8 Hz, 3F), -91.55 (d, J = 45.1 Hz, 1F), -91.89 (d, J = 44.4 Hz, 1F).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{22}\text{H}_{29}\text{F}_5\text{O}_2\text{Na}$ 443.1980; found 443.1985.

1-(*tert*-butyl) 2-(8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl) (2*R*)-pyrrolidine-1,2-dicarboxylate (4k)



4k

4k was prepared according to the general procedure from 1-(tert-butyl) 2-(hex-5-en-1-yl) (R)-pyrrolidine-1,2-dicarboxylate **1k** (89.1 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4k** (42.2 mg, 77% yield, dr = 1:1) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 5:1, v/v).

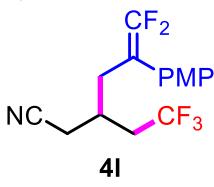
^1H NMR (600 MHz, CDCl_3) δ 7.13 (d, J = 8.4 Hz, 2H), 6.83 (d, J = 8.8 Hz, 2H), 4.29 – 4.08 (m, 1H), 4.09 – 3.92 (m, 2H), 3.75 (s, 3H), 3.57 – 3.19 (m, 2H), 2.59 – 2.26 (m, 2H), 2.31 – 2.07 (m, 1H), 2.05 – 1.91 (m, 2H), 1.86 (ddd, J = 19.6, 12.6, 6.9 Hz, 2H), 1.63 (dd, J = 8.0, 5.3 Hz, 2H), 1.49 (dt, J = 14.0, 7.2 Hz, 2H), 1.38 (s, 4H), 1.33 (s, 5H), 1.32 – 1.14 (m, 4H).

^{13}C NMR (151 MHz, CDCl_3) δ 173.3, 158.9, 154.1 (dd, J = 316.6, 257.8 Hz), 153.8, 129.3, 127.1 (d, J = 277.4 Hz), 124.8, 114.1, 90.0 (dd, J = 18.2, 10.2 Hz), 79.9, 64.6, 59.2, 55.3, 46.3, 38.6 – 35.0 (m), 32.4, 30.9, 30.5, 28.7, 28.3, 24.3, 23.6, 22.2.

^{19}F NMR (565 MHz, CDCl_3) δ -63.24 – -63.29 (m, 3F), -91.28 (d, J = 44.8 Hz, 1F), -91.77 (d, J = 45.1 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{27}\text{H}_{36}\text{F}_5\text{O}_5\text{NNa}$ 572.2406; found 572.2415.

6,6-difluoro-5-(4-methoxyphenyl)-3-(2,2,2-trifluoroethyl)hex-5-enenitrile (**4l**)



4l

4l was prepared according to the general procedure from but-3-enenitrile **1l** (20.1 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4l** (12.8 mg, 40% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 50:1, v/v).

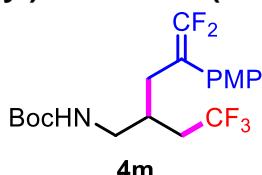
^1H NMR (600 MHz, CDCl_3) δ 7.22 (d, J = 8.4 Hz, 2H), 6.93 (d, J = 8.7 Hz, 2H), 3.82 (s, 3H), 2.75 – 2.53 (m, 2H), 2.45 (dd, J = 5.2, 2.4 Hz, 2H), 2.39 – 2.17 (m, 2H), 2.10 (dt, J = 12.6, 6.3 Hz, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 159.3, 154.3 (dd, *J* = 291.7, 288.6 Hz), 129.2 (t, *J* = 2.7 Hz), 126.1 (d, *J* = 277.0 Hz), 123.6 (t, *J* = 3.1 Hz), 116.9, 114.5, 88.8 (dd, *J* = 20.7, 16.1 Hz), 55.3, 36.4 (q, *J* = 28.7 Hz), 31.7, 28.3, 21.4.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.49 (t, *J* = 11.6 Hz, 3F), -89.38 (d, *J* = 40.5 Hz, 1F), -89.55 (d, *J* = 40.8 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₅H₁₄F₅NONa 342.0888; found 342.0880.

tert-butyl (5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)carbamate (4m)



4m was prepared according to the general procedure from *tert*-butyl allylcarbamate **1m** (47.1 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4m** (24.5 mg, 60% yield) was isolated as a clear oil.

TLC *Rf* = 0.30 (Hexane/EtOAc = 5:1, v/v).

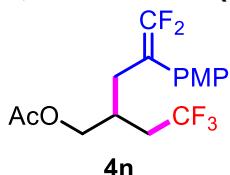
¹H NMR (600 MHz, CDCl₃) δ 7.22 (d, *J* = 8.3 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.49 (s, 1H), 3.81 (s, 3H), 3.15 (d, *J* = 5.3 Hz, 2H), 2.48 (d, *J* = 7.4 Hz, 2H), 2.30 – 2.09 (m, 1H), 2.09 – 1.95 (m, 1H), 1.91 – 1.73 (m, 1H), 1.43 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 159.0, 156.2, 156.0 – 151.1 (m), 129.3, 126.9 (d, *J* = 277.9 Hz), 124.5, 114.2, 89.6 (dd, *J* = 21.3, 14.7 Hz), 79.6, 55.3, 42.8, 35.2, 32.1, 30.1, 28.3.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.67 (t, *J* = 11.7 Hz, 3F), -89.51 (d, *J* = 42.2 Hz, 1F), -90.52 (d, *J* = 42.7 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₉H₂₄F₅NO₃Na 432.1569; found 432.1577.

5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl acetate (4n)



4n was prepared according to the general procedure from allyl acetate **1n** (30.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4n** (22.9 mg, 65% yield) was isolated as a clear oil.

TLC *Rf* = 0.40 (Hexane/EtOAc = 20:1, v/v).

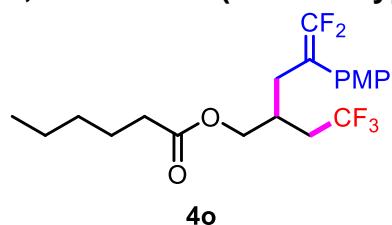
¹H NMR (600 MHz, CDCl₃) δ 7.22 (d, *J* = 7.9 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.00 (ddd, *J* = 26.0, 11.4, 4.5 Hz, 2H), 3.82 (s, 3H), 2.54 (dd, *J* = 7.2, 2.0 Hz, 2H), 2.38 – 2.20 (m, 1H), 2.15 – 2.08 (m, 1H), 2.05 (s, 3H), 2.06 – 1.99 (m, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 170.8, 159.1, 154.1 (dd, *J* = 290.6, 287.1 Hz), 129.3 (t, *J* = 2.7 Hz), 126.7 (q, *J* = 277.0 Hz), 124.3 (t, *J* = 3.3 Hz), 114.2, 89.3 (dd, *J* = 21.0, 15.1 Hz), 64.7, 55.3, 34.9 (q, *J* = 28.4 Hz), 30.5, 29.4, 20.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.65 (t, *J* = 10.9 Hz, 3F), -89.84 (d, *J* = 41.4 Hz, 1F), -90.10 (d, *J* = 41.7 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₆H₁₇F₅O₃Na 375.0990; found 375.0997.

5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl hexanoate (4o)



4o was prepared according to the general procedure from allyl hexanoate **1o** (46.8 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4o** (29.0 mg, 71% yield) was isolated as a clear oil.

TLC *Rf* = 0.60 (Hexane/EtOAc = 20:1, v/v).

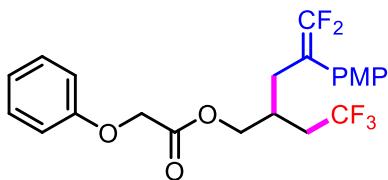
¹H NMR (600 MHz, CDCl₃) δ 7.22 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.00 (qd, *J* = 11.4, 4.4 Hz, 2H), 3.82 (s, 3H), 2.53 (d, *J* = 7.3 Hz, 2H), 2.30 (t, *J* = 7.6 Hz, 2H), 2.20 (s, 1H), 2.12 (ddd, *J* = 26.3, 16.1, 10.5 Hz, 1H), 2.05 (dt, *J* = 10.7, 3.8 Hz, 1H), 1.61 (dt, *J* = 14.9, 7.6 Hz, 2H), 1.44 – 1.19 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 173.6, 159.0, 154.1 (dd, *J* = 290.7, 287.3 Hz), 129.3 (t, *J* = 2.7 Hz), 126.7 (d, *J* = 277.0 Hz), 124.3 (t, *J* = 3.5 Hz), 114.2, 89.3 (dd, *J* = 21.3, 14.9 Hz), 64.5, 55.3, 34.9 (q, *J* = 28.4 Hz), 34.1, 31.3, 30.6, 29.5, 24.6, 22.3, 13.9.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.65 (t, *J* = 10.8 Hz, 3F), -90.25 (d, *J* = 42.0 Hz, 1F), -90.72 (d, *J* = 41.9 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₀H₂₅F₅O₃Na 431.1616; found 431.1624.

5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl 2-phenoxyacetate (4p)



4p

4p was prepared according to the general procedure from allyl 2-phenoxyacetate **1p** (57.6 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4p** (22.2 mg, 50% yield) was isolated as a clear oil.

TLC R_f = 0.60 (Hexane/EtOAc = 20:1, v/v).

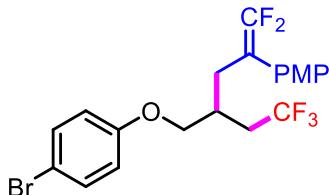
^1H NMR (600 MHz, CDCl_3) δ 7.31 (t, J = 7.9 Hz, 2H), 7.18 (d, J = 8.5 Hz, 2H), 7.01 (t, J = 7.4 Hz, 1H), 6.95 – 6.82 (m, 4H), 4.65 (s, 2H), 4.25 – 3.99 (m, 2H), 3.81 (s, 3H), 2.58 – 2.30 (m, 2H), 2.16 – 1.83 (m, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 168.9, 159.1, 157.7, 154.1 (dd, J = 290.9, 287.4 Hz), 129.7, 129.3 (t, J = 3.0 Hz), 126.5 (d, J = 276.9 Hz), 124.6 – 123.9(m), 121.9 114.5, 114.3, 89.2 (dd, J = 20.9, 15.2 Hz), 65.5, 65.0, 55.3, 34.6 (q, J = 28.5 Hz), 30.5, 29.4.

^{19}F NMR (565 MHz, CDCl_3) δ -63.68 (t, J = 10.8 Hz, 3F), -90.28 (d, J = 42.0 Hz, 1F), -90.75 (d, J = 41.9 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{22}\text{H}_{21}\text{F}_5\text{O}_4\text{Na}$ 467.1252; found 467.1264.

1-bromo-4-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)benzene (**4q**)



4q

4q was prepared according to the general procedure from 1-(allyloxy)-4-bromobenzene **1q** (63.3 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4q** (28.8 mg, 62% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

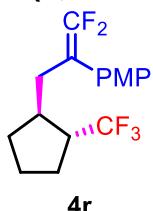
^1H NMR (600 MHz, CDCl_3) δ 7.35 (d, J = 8.6 Hz, 2H), 7.21 (d, J = 8.4 Hz, 2H), 6.87 (d, J = 8.5 Hz, 2H), 6.69 (d, J = 8.6 Hz, 2H), 3.89 – 3.76 (m, 2H), 3.80 (s, 3H), 2.73 – 2.62 (m, 2H), 2.39 (qd, J = 17.8, 8.5 Hz, 1H), 2.26 – 2.10 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 159.0, 157.6, 154.1 (dd, *J* = 290.8, 287.7 Hz), 132.3, 129.2 (t, *J* = 3.0 Hz), 126.9 (d, *J* = 277.0 Hz), 124.5 (t, *J* = 3.4 Hz), 116.3, 114.2, 113.3, 89.5 (dd, *J* = 21.0, 14.9 Hz), 68.3, 55.3, 34.7 (q, *J* = 28.4 Hz), 31.4, 29.4.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.59 (t, *J* = 12.2 Hz, 3F), -90.50 (d, *J* = 42.7 Hz, 1F), -90.76 (d, *J* = 42.7 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₀H₁₈BrF₅O₂Na 487.0302; found 487.0314.

1-(1,1-difluoro-3-(2-(trifluoromethyl)cyclopentyl)prop-1-en-2-yl)-4-methoxybenzene (4r)



4r was prepared according to the general procedure from cyclopentene **1r** (20.1 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4r** (25.9 mg, 81% yield) was isolated as a clear oil.

TLC *Rf* = 0.40 (Hexane/EtOAc = 50:1, v/v).

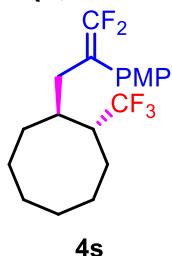
¹H NMR (600 MHz, CDCl₃) δ 7.23 (d, *J* = 7.9 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.65 (ddt, *J* = 14.1, 5.4, 3.5 Hz, 1H), 2.34 (ddd, *J* = 14.2, 9.8, 2.3 Hz, 1H), 2.25 (ddt, *J* = 13.3, 9.9, 5.0 Hz, 1H), 2.11 – 2.01 (m, 1H), 1.93 – 1.83 (m, 1H), 1.79 – 1.68 (m, 2H), 1.64 (dt, *J* = 12.7, 6.3 Hz, 1H), 1.56 (dt, *J* = 13.0, 7.4 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 158.8, 154.0 (dd, *J* = 289.9, 285.6 Hz), 129.3 (t, *J* = 3.2 Hz), 128.6 (q, *J* = 277.7 Hz), 125.1 (t, *J* = 3.3 Hz), 114.0, 90.8 (dd, *J* = 21.5, 13.8 Hz), 55.3, 48.1 (q, *J* = 26.1 Hz), 38.5, 33.2, 32.3, 26.7 (d, *J* = 2.1 Hz), 24.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -70.40 (d, *J* = 10.6 Hz, 3F), -92.04 (d, *J* = 46.6 Hz, 1F), -92.35 (dd, *J* = 45.3, 4.1 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₆H₁₇F₅ONa 343.1092; found 343.1099.

1-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-2-(trifluoromethyl)cyclooctane (4s)



4s was prepared according to the general procedure from (Z)-cyclooctene **1s** (33.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-

methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4s** (31.5 mg, 87% yield, dr = 1.2:1) was isolated as a clear oil. TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

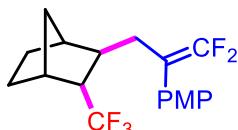
¹H NMR (600 MHz, CDCl₃) δ 7.20 (d, J = 8.4 Hz, 2H), 6.89 (d, J = 7.3 Hz, 2H), 3.82 (s, 3H), 2.27 (dd, J = 18.1, 5.9 Hz, 2H), 2.12 (ddd, J = 19.0, 16.9, 9.5 Hz, 1H), 2.00 – 1.67 (m, 4H), 1.67 – 1.55 (m, 2H), 1.53 – 1.29 (m, 7H).

¹³C NMR (151 MHz, CDCl₃) δ 158.7, 154.0 (dd, J = 303.1, 287.4 Hz), 129.4 (t, J = 3.0 Hz), 128.7 (d, J = 279.8 Hz), 125.7 (d, J = 10.9 Hz), 113.9, 90.8 (dd, J = 19.6, 15.6 Hz), 55.3, 42.7 (q, J = 24.2 Hz), 35.6, 35.1, 31.7, 29.1, 26.1, 25.2, 24.6, 23.9.

¹⁹F NMR (565 MHz, CDCl₃) δ -73.25 (d, J = 10.9 Hz, 1.5F), -73.38 (d, J = 10.9 Hz, 1.5F), -92.36 (d, J = 18.1 Hz, 2F).

HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₉H₂₃F₅ONa 385.1561; found 385.1564.

2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)bicyclo[2.2.1]heptane (**4t**)



4t

4t was prepared according to the general procedure from (1R,4S)-bicyclo[2.2.1]hept-2-ene **1t** (28.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4t** (29.7 mg, 86% yield, dr = 1.7:1) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

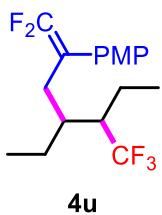
¹H NMR (400 MHz, CDCl₃) δ 7.25 – 7.08 (m, 2H), 6.98 – 6.84 (m, 2H), 3.82 (s, 3H), 2.68 – 2.33 (m, 4H), 2.17 – 2.01 (m, 2H), 1.94 – 1.65 (m, 2H), 1.65 – 1.55 (m, 1H), 1.51 – 1.30 (m, 1H), 1.25 – 1.16 (m, 1H), 1.12 (d, J = 10.7 Hz, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 158.8, 153.8 (dd, J = 255.6, 252.8 Hz), 129.5 (t, J = 3.1 Hz), 127.8 (d, J = 280.1 Hz), 125.4 – 125.2 (m), 114.0, 91.4 (dd, J = 21.6, 14.3 Hz), 55.3, 49.5 (q, J = 24.9 Hz), 42.8, 39.5, 38.6, 34.1, 29.9, 28.5, 21.3.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.95 (t, J = 11.7 Hz, 3F), -90.71 (d, J = 43.8 Hz, 1F), -91.24 (d, J = 44.1 Hz, 1F).

HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₈H₁₉F₅ONa 369.1248; found 369.1240.

1-(4-ethyl-1,1-difluoro-5-(trifluoromethyl)hept-1-en-2-yl)-4-methoxybenzene (**4u**)



4u was prepared according to the general procedure from (E)-hex-3-ene **1u** (25.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4u** (13.4 mg, 40% yield, dr = 2:1) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.24 – 7.04 (m, 2H), 6.93 – 6.83 (m, 2H), 3.82 (s, 3H), 2.60 – 2.41 (m, 1H), 2.41 – 2.23 (m, 1H), 2.21 – 1.88 (m, 1H), 1.57 – 1.10 (m, 5H), 0.91 (t, J = 7.5 Hz, 3H), 0.85 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 157.9 – 153.5 (m), 129.4 (t, J = 2.6 Hz), 128.8 (d, J = 282.1 Hz), 125.3 – 124.8 (m), 114.0, 90.7 (dd, J = 21.6, 14.2 Hz), 55.3, 45.1 (dd, J = 47.6, 23.9 Hz), 36.9, 28.9, 22.6, 17.7, 12.9, 12.0.

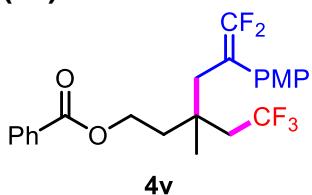
^{19}F NMR (565 MHz, CDCl_3):

For major: δ -65.88 (d, J = 11.8 Hz, 2F), -91.52 (d, J = 45.8 Hz, 0.66F), -92.54 (d, J = 45.5 Hz, 0.66F).

For minor: δ -65.46 (d, J = 12.0 Hz, 1F), -91.65 (d, J = 44.5 Hz, 0.33F), -92.05 (d, J = 45.8 Hz, 0.33F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{17}\text{H}_{21}\text{F}_5\text{ONa}$ 359.1405; found 359.1411.

6,6-difluoro-5-(4-methoxyphenyl)-3-methyl-3-(2,2,2-trifluoroethyl)hex-5-en-1-yl benzoate (**4v**)



4v was prepared according to the general procedure from 3-methylbut-3-en-1-yl benzoate **1v** (57.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4v** (36.2 mg, 82% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

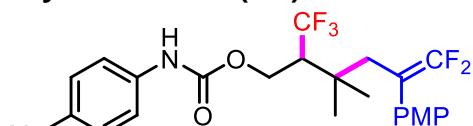
^1H NMR (600 MHz, CDCl_3) δ 7.98 (d, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.22 (d, J = 8.1 Hz, 2H), 6.87 (d, J = 8.7 Hz, 2H), 4.28 (dd, J = 10.4, 4.3 Hz, 2H), 3.74 (s, 3H), 2.57 (q, J = 14.5 Hz, 2H), 2.25 – 1.95 (m, 2H), 1.92 – 1.75 (m, 2H), 1.00 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 166.5, 158.9, 157.0 – 152.4 (m), 133.0, 130.2, 130.1, 129.5, 128.4, 126.9 (dd, *J* = 590.0, 311.4 Hz), 126.5 (dd, *J* = 4.1, 2.0 Hz), 114.1, 89.0 (dd, *J* = 20.7, 15.3 Hz), 61.2, 55.2, 42.1 (q, *J* = 26.4 Hz), 38.2, 37.4, 36.0, 24.9.

¹⁹F NMR (565 MHz, CDCl₃) δ -58.76 (t, *J* = 12.6 Hz, 3F), -89.07 (d, *J* = 40.6 Hz, 1F), -90.77 (d, *J* = 40.3 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₃H₂₃F₅O₃Na 465.1460; found 465.1472.

6,6-difluoro-5-(4-methoxyphenyl)-3,3-dimethyl-2-(trifluoromethyl)hex-5-en-1-yl p-tolylcarbamate (4w)



4w

4w was prepared according to the general procedure from 3-methylbut-2-en-1-yl p-tolylcarbamate **1w** (65.7 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4w** (36.7 mg, 78% yield) was isolated as a white solid.

TLC *Rf* = 0.45 (Hexane/EtOAc = 5:1, v/v).

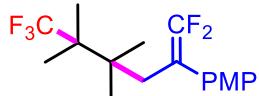
¹H NMR (600 MHz, CDCl₃) δ 7.26 (s, 2H), 7.22 (d, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.44 (s, 1H), 4.36 (d, *J* = 11.1 Hz, 1H), 4.23 (dd, *J* = 12.0, 6.7 Hz, 1H), 3.75 (s, 3H), 2.56 (s, 2H), 2.31 (s, 3H), 2.29 – 2.15 (m, 1H), 0.98 (s, 3H), 0.95 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 158.8, 154.6 (t, *J* = 289.0 Hz), 134.9, 133.3, 129.6, 129.5, 127.7 (d, *J* = 258.9 Hz), 127.3, 126.6, 118.8 – 118.5 (m), 114.1, 89.2 (dd, *J* = 21.0, 14.8 Hz), 60.7, 55.2, 50.1 (q, *J* = 23.5 Hz), 38.3, 36.9, 25.7, 25.4, 20.8.

¹⁹F NMR (565 MHz, CDCl₃) δ -61.84 (t, *J* = 11.6 Hz, 3F), -89.14 (d, *J* = 40.6 Hz, 1F), -91.18 (d, *J* = 40.8 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₄H₂₆F₅NO₃Na 494.1725; found 494.1729.

1-methoxy-4-(1,1,6,6,6-pentafluoro-4,4,5,5-tetramethylhex-1-en-2-yl)benzene (4x)



4x

4x was prepared according to the general procedure from 2,3-dimethylbut-2-ene **1x** (25.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4x** (19.2 mg, 57% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

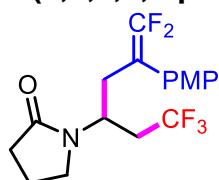
^1H NMR (600 MHz, CDCl_3) δ 7.22 (d, J = 7.6 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 3.81 (s, 3H), 2.54 (s, 2H), 1.11 (s, 6H), 0.79 (s, 6H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.5, 154.4 (dd, J = 289.5, 287.7 Hz), 130.2 (d, J = 286.6 Hz), 129.4 – 129.3 (m), 128.0 – 127.7 (m), 113.9, 89.9 (dd, J = 21.0, 14.0 Hz), 55.3, 46.2 (q, J = 21.3 Hz), 39.7, 34.4, 23.0, 18.7.

^{19}F NMR (565 MHz, CDCl_3) δ -68.68 (s, 3F), -89.60 (d, J = 42.0 Hz, 1F), -91.86 (d, J = 41.7 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{17}\text{H}_{21}\text{F}_5\text{ONa}$ 359.1405; found 359.1411.

1-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)pyrrolidin-2-one (4y)



4y

4y was prepared according to the general procedure from 1-vinylpyrrolidin-2-one **1y** (33.3 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4y** (30.5 mg, 84% yield) was isolated as a clear oil.

TLC R_f = 0.35 (Hexane/EtOAc = 5:1, v/v).

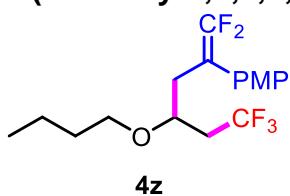
^1H NMR (600 MHz, CDCl_3) δ 7.21 (d, J = 8.5 Hz, 2H), 6.90 (d, J = 8.6 Hz, 2H), 4.15 (s, 1H), 3.81 (s, 3H), 3.16 (t, J = 7.0 Hz, 2H), 2.93 – 2.77 (m, 1H), 2.66 (dt, J = 15.0, 10.2 Hz, 1H), 2.60 – 2.42 (m, 1H), 2.24 (ddd, J = 15.0, 10.6, 4.2 Hz, 1H), 2.19 (t, J = 8.1 Hz, 2H), 1.81 (tt, J = 14.6, 7.5 Hz, 1H), 1.71 (tt, J = 15.3, 7.7 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 175.1, 159.1, 154.2 (dd, J = 291.1, 288.1 Hz), 129.3 (t, J = 2.6 Hz), 125.8 (q, J = 277.1 Hz), 124.5 (t, J = 3.5 Hz), 114.1, 89.0 (dd, J = 20.9, 16.1 Hz), 55.3, 46.7, 44.8, 35.0 (q, J = 28.2 Hz), 31.3, 30.6, 18.3.

^{19}F NMR (565 MHz, CDCl_3) δ -64.52 (t, J = 10.7 Hz, 3F), -90.33 (d, J = 41.1 Hz, 1F), -90.58 (d, J = 42.1 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{17}\text{H}_{18}\text{NF}_5\text{O}_2\text{Na}$ 386.1150; found 386.1157.

1-(4-butoxy-1,1,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4z)



4z

4z was prepared according to the general procedure from 1-(vinyloxy)butane **1z** (30.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4z** (31.7 mg, 90% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

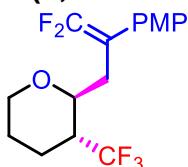
^1H NMR (600 MHz, CDCl_3) δ 7.24 (d, J = 8.7 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 3.71 – 3.43 (m, 1H), 3.35 (t, J = 6.5 Hz, 2H), 2.82 – 2.59 (m, 1H), 2.58 – 2.45 (m, 1H), 2.29 (tdd, J = 18.3, 9.2, 6.0 Hz, 1H), 2.23 – 1.99 (m, 1H), 1.53 – 1.37 (m, 2H), 1.31 (dd, J = 15.1, 7.5 Hz, 2H), 0.88 (t, J = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 154.3 (t, J = 288.9 Hz), 129.3 (t, J = 3.1 Hz), 126.1 (q, J = 277.0 Hz), 125.1, 114.1, 88.7 (t, J = 18.2 Hz), 72.2, 69.8, 55.3, 38.6 (q, J = 27.5 Hz), 33.4, 31.9, 19.2, 13.8.

^{19}F NMR (565 MHz, CDCl_3) δ -63.48 – -63.52 (m, 3F), -90.72 (s, 2F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{17}\text{H}_{21}\text{F}_5\text{O}_2\text{Na}$ 375.1354; found 375.1360.

2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)tetrahydro-2H-pyran (**4aa**)



4aa

4aa was prepared according to the general procedure from 3,4-dihydro-2H-pyran **1aa** (25.2 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4aa** (25.2 mg, 75% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

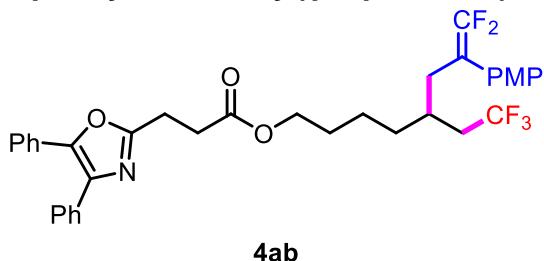
^1H NMR (600 MHz, CDCl_3) δ 7.23 (d, J = 8.2 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 3.98 – 3.86 (m, 1H), 3.82 (s, 3H), 3.32 – 3.18 (m, 1H), 3.15 (ddd, J = 11.5, 7.8, 4.8 Hz, 1H), 2.77 (dd, J = 14.9, 3.3 Hz, 1H), 2.54 (dd, J = 14.4, 11.0 Hz, 1H), 2.12 (ddd, J = 8.9, 8.0, 3.6 Hz, 1H), 2.09 – 2.00 (m, 1H), 1.69 – 1.53 (m, 2H), 1.51 – 1.37 (m, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.7, 154.1 (t, J = 287.2 Hz), 129.6 (t, J = 2.8 Hz), 126.6 (q, J = 279.9 Hz), 125.4 – 125.3 (m), 113.9, 88.9 (dd, J = 21.7, 16.4 Hz), 74.0, 67.7, 55.2, 44.9 (q, J = 24.8 Hz), 32.4, 24.4, 23.3.

^{19}F NMR (565 MHz, CDCl_3) δ -67.54 (d, J = 9.8 Hz, 3F), -91.37 (dd, J = 44.9, 5.7 Hz, 1F), -92.23 (d, J = 46.5 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{17}\text{F}_5\text{O}_2\text{Na}$ 359.1041; found 359.1048.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 3-(4,5-diphenyloxazol-2-yl)propanoate (4ab)



4ab was prepared according to the general procedure from hex-5-en-1-yl 3-(4,5-diphenyloxazol-2-yl)propanoate **1ab** (112.5 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ab** (48.3 mg, 77% yield) was isolated as a white solid.

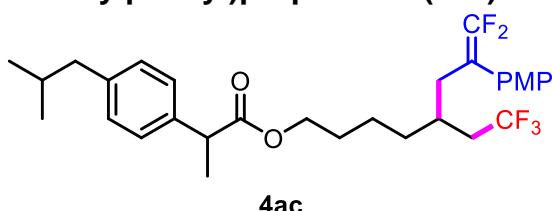
TLC R_f = 0.50 (Hexane/EtOAc = 10:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.69 – 7.59 (m, 2H), 7.57 (dd, J = 5.2, 3.3 Hz, 2H), 7.43 – 7.28 (m, 6H), 7.19 (d, J = 8.0 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 4.23 – 3.95 (m, 2H), 3.80 (s, 3H), 3.18 (t, J = 7.6 Hz, 2H), 2.90 (t, J = 7.6 Hz, 2H), 2.46 – 2.32 (m, 2H), 2.00 (qd, J = 11.4, 6.3 Hz, 2H), 1.71 (dd, J = 12.8, 6.4 Hz, 1H), 1.57 – 1.49 (m, 2H), 1.30 (ddd, J = 20.2, 13.0, 5.8 Hz, 4H).
 ^{13}C NMR (151 MHz, CDCl_3) δ 172.0, 161.8, 158.9, 154.0 (dd, J = 290.0, 286.5 Hz), 145.4, 135.1, 132.5, 129.3 (t, J = 2.9 Hz), 129.0, 128.7, 128.6, 128.5, 128.0 (d, J = 11.1 Hz), 127.9, 127.1 (d, J = 277.1 Hz), 126.5, 125.3 – 124.6 (m), 114.1, 90.1 (dd, J = 21.1, 14.3 Hz), 64.5, 55.3, 36.8 (q, J = 27.6 Hz), 32.4, 31.9, 31.2, 30.5, 29.7, 28.6, 23.6, 22.1.

^{19}F NMR (565 MHz, CDCl_3) δ -63.22 (t, J = 12.0 Hz, 3F), -91.33 (d, J = 44.8 Hz, 1F), -91.71 (d, J = 44.6 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{35}\text{H}_{34}\text{F}_5\text{NO}_4\text{Na}$ 650.2300; found 650.2311.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-isobutylphenyl)propanoate (4ac)



4ac was prepared according to the general procedure from hex-5-en-1-yl 2-(4-isobutylphenyl)propanoate **1ac** (86.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ac** (43.2 mg, 80% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

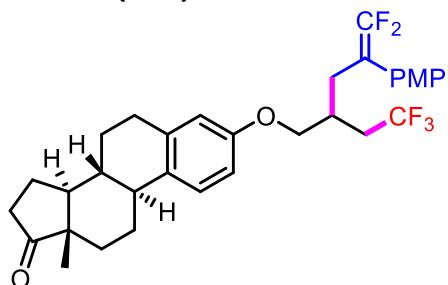
¹H NMR (600 MHz, CDCl₃) δ 7.23 – 7.13 (m, 4H), 7.07 (d, *J* = 7.4 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 4.01 (ddd, *J* = 9.0, 6.1, 2.7 Hz, 2H), 3.81 (s, 3H), 3.67 (q, *J* = 7.1 Hz, 1H), 2.42 (d, *J* = 7.2 Hz, 2H), 2.47 – 2.37 (m, 1H), 2.34 (dd, *J* = 14.2, 7.4 Hz, 1H), 2.08 – 1.89 (m, 2H), 1.83 (dt, *J* = 13.5, 6.7 Hz, 1H), 1.66 (dt, *J* = 13.0, 6.5 Hz, 1H), 1.53 – 1.44 (m, 5H), 1.35 – 1.23 (m, 2H), 1.24 – 1.07 (m, 2H), 0.88 (d, *J* = 6.6 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 174.8, 158.9, 154.0 (dd, *J* = 290.0, 286.6 Hz), 140.5, 137.8, 127.1, 127.1 (d, *J* = 277.3 Hz), 125.3 – 124.4 (m), 114.1, 90.1 (dd, *J* = 21.4, 14.2 Hz), 64.3, 55.3, 45.2, 45.0, 36.8 (dd, *J* = 51.4, 27.4 Hz), 32.4, 31.8, 30.4, 30.2, 28.6, 22.4, 22.0, 18.4 (d, *J* = 7.1 Hz).

¹⁹F NMR (565 MHz, CDCl₃) δ -63.24 (t, *J* = 15.6 Hz, 3F), -91.38 (d, *J* = 46.0 Hz, 1F), -91.76 (d, *J* = 45.2 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₃₀H₃₇F₅O₃Na 563.2555; found 563.2567.

(8R,9S,13S,14S)-3-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ad)



4ad

4ad was prepared according to the general procedure from (8R,9S,13S,14S)-3-(allyloxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one **1ad** (93.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ad** (42.1 mg, 75% yield) was isolated as a white solid.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 8.6 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.65 (dd, *J* = 8.6, 2.6 Hz, 1H), 6.57 (d, *J* = 2.5 Hz, 1H), 3.85 (dd, *J* = 7.7, 4.7 Hz, 2H), 3.81 (s, 3H), 2.88 (dd, *J* = 11.0, 4.7 Hz, 2H), 2.78 – 2.58 (m, 2H), 2.51 (dd, *J* = 18.8, 8.5 Hz, 1H), 2.47 – 2.32 (m, 2H), 2.24 (dd, *J* = 17.8, 7.3 Hz, 2H), 2.20 – 2.11 (m, 4H), 2.10 – 1.92 (m, 2H), 1.72 – 1.48 (m, 3H), 1.49 – 1.29 (m, 2H), 0.91 (s, 3H).

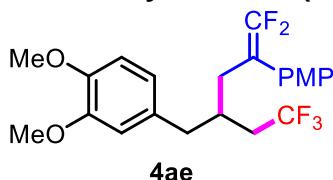
¹³C NMR (151 MHz, CDCl₃) δ 158.9, 156.6, 154.1 (dd, *J* = 290.5, 287.5 Hz), 137.8, 132.5, 129.3 (t, *J* = 2.6 Hz), 127.0 (d, *J* = 276.9 Hz), 126.4, 114.5, 114.2, 112.2, 112.1, 89.6 (dd, *J* =

21.0, 14.9 Hz), 68.2, 55.3, 50.4, 48.0, 44.0, 38.4, 35.9, 34.6 (q, J = 28.2 Hz), 31.6, 31.4, 29.6, 29.5, 26.6, 25.9, 21.6, 13.9.

^{19}F NMR (565 MHz, CDCl_3) δ -63.53 (t, J = 12.3 Hz, 3F), -90.62 (d, J = 42.9 Hz, 1F), -90.81 (d, J = 44.5 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{32}\text{H}_{35}\text{F}_5\text{O}_3\text{Na}$ 585.2399; found 585.2390.

4-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)-1,2-dimethoxybenzene (4ae)



4ae was prepared according to the general procedure from 4-allyl-1,2-dimethoxybenzene **1ae** (53.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ae** (36.1 mg, 84% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

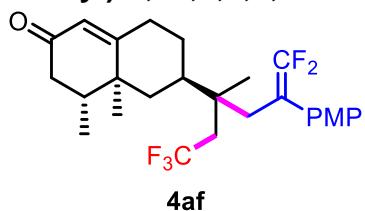
^1H NMR (600 MHz, CDCl_3) δ 7.10 (d, J = 7.9 Hz, 2H), 6.87 (d, J = 8.8 Hz, 2H), 6.76 (d, J = 8.1 Hz, 1H), 6.57 (dd, J = 8.1, 1.9 Hz, 1H), 6.43 (d, J = 1.9 Hz, 1H), 3.86 (s, 3H), 3.81 (s, 3H), 3.77 (s, 3H), 2.67 – 2.53 (m, 2H), 2.48 (dd, J = 14.7, 7.2 Hz, 1H), 2.39 (dd, J = 14.9, 7.2 Hz, 1H), 2.04 (qd, J = 11.4, 6.3 Hz, 2H), 1.98 – 1.86 (m, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 158.9, 154.0 (dd, J = 290.1, 286.3 Hz), 148.9, 147.6, 131.6, 129.4 (t, J = 3.0 Hz), 127.1 (q, J = 277.8 Hz), 124.6 – 124.5 (m), 121.2, 114.0, 111.9, 111.1, 90.1 (dd, J = 21.1, 14.1 Hz), 55.9, 55.8, 55.3, 39.2, 36.6 (q, J = 27.6 Hz), 32.7, 31.4.

^{19}F NMR (565 MHz, CDCl_3) δ -62.75 (t, J = 12.6 Hz, 3F), -91.20 (d, J = 45.0 Hz, 1F), -91.63 (d, J = 45.0 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{22}\text{H}_{23}\text{F}_5\text{O}_3\text{Na}$ 453.1460; found 453.1465.

(4R,4aS,6R)-4,4a-dimethyl-6-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)-3-methylhex-5-en-3-yl)-4,4a,5,6,7,8-hexahydronaphthalen-2(3H)-one (4af)



4af was prepared according to the general procedure from (4R,4aS,6R)-4,4a-dimethyl-6-(prop-1-en-2-yl)-4,4a,5,6,7,8-hexahydronaphthalen-2(3H)-one **1af** (65.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-

(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4af** (35.2 mg, 75% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

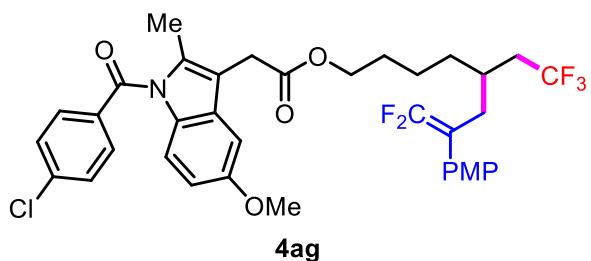
^1H NMR (600 MHz, CDCl_3) δ 7.20 (t, J = 7.3 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 5.70 (d, J = 29.5 Hz, 1H), 3.80 (s, 3H), 2.58 – 2.39 (m, 2H), 2.36 (ddd, J = 20.1, 8.5, 5.6 Hz, 1H), 2.28 – 2.13 (m, 3H), 2.00 – 1.89 (m, 3H), 1.91 – 1.83 (m, 1H), 1.82 – 1.58 (m, 2H), 1.19 – 1.01 (m, 2H), 1.06 – 0.85 (m, 9H).

^{13}C NMR (151 MHz, CDCl_3) δ 199.5, 170.1, 158.9, 154.4 (t, J = 289.1 Hz), 129.6, 127.3 (d, J = 279.0 Hz), 127.0, 124.4, 114.1, 89.3 (dd, J = 11.9, 8.7 Hz), 60.4, 55.3, 42.0, 40.6, 39.5 (dd, J = 49.5, 27.6 Hz), 38.8, 34.7, 32.8, 27.3, 23.2, 16.7, 15.0.

^{19}F NMR (565 MHz, CDCl_3) δ -57.94 (t, J = 12.7 Hz, 3F), -89.20 (d, J = 40.9 Hz, 1F), -90.75 (d, J = 40.9 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{26}\text{H}_{31}\text{F}_5\text{O}_2\text{Na}$ 493.2136; found 493.2144.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (4ag)



4ag was prepared according to the general procedure from hex-5-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate **1ag** (131.7 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ag** (55.9 mg, 81% yield) was isolated as a white solid.

TLC R_f = 0.30 (Hexane/EtOAc = 5:1, v/v).

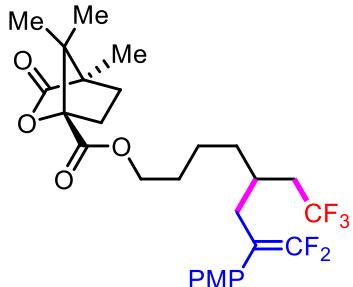
^1H NMR (600 MHz, CDCl_3) δ 7.64 (d, J = 8.5 Hz, 2H), 7.46 (d, J = 8.5 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 6.96 (d, J = 2.5 Hz, 1H), 6.88 (d, J = 8.8 Hz, 2H), 6.85 (d, J = 9.0 Hz, 1H), 6.66 (dd, J = 9.0, 2.5 Hz, 1H), 4.06 (t, J = 6.5 Hz, 2H), 3.82 (s, 3H), 3.80 (s, 3H), 3.65 (s, 2H), 2.39 (s, 3H), 2.43 – 2.34 (m, 1H), 2.32 (dd, J = 14.5, 7.4 Hz, 1H), 2.05 – 1.84 (m, 2H), 1.67 (dt, J = 13.1, 6.5 Hz, 1H), 1.57 – 1.45 (m, 2H), 1.37 – 1.28 (m, 2H), 1.28 – 1.14 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 170.9, 168.3, 158.9, 156.0, 154.0 (dd, J = 290.3, 286.1 Hz), 139.3, 136.0, 133.9, 131.2, 130.8, 130.7, 129.3 (t, J = 2.6 Hz), 129.1, 127.1 (d, J = 277.1 Hz), 124.8 (t, J = 3.4 Hz), 115.0, 114.1, 112.6, 111.5, 101.4, 90.1 (dd, J = 21.3, 14.4 Hz), 64.7, 55.7, 55.3, 36.7 (q, J = 27.4 Hz), 32.4, 31.7, 30.4, 28.6, 22.1, 13.3.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.22 (t, *J* = 11.9 Hz, 3F), -91.32 (d, *J* = 44.7 Hz, 1F), -91.70 (d, *J* = 44.6 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₃₆H₃₅F₅CINO₅Na 714.2016; found 714.2028.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl (1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (4ah)



4ah was prepared according to the general procedure from hex-5-en-1-yl (1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate **1ah** (84.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ah** (46.3 mg, 87% yield) was isolated as a clear oil.

TLC *Rf* = 0.40 (Hexane/EtOAc = 10:1, v/v).

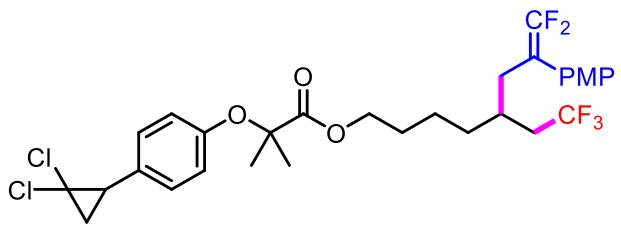
¹H NMR (600 MHz, CDCl₃) δ 7.19 (d, *J* = 8.3 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 4.24 – 4.13 (m, 2H), 3.81 (s, 3H), 2.52 – 2.31 (m, 3H), 2.02 (ddd, *J* = 14.5, 9.7, 6.6 Hz, 2H), 1.92 (ddd, *J* = 13.2, 10.8, 4.6 Hz, 1H), 1.69 (ddd, *J* = 17.6, 8.9, 4.6 Hz, 2H), 1.65 – 1.55 (m, 3H), 1.42 – 1.35 (m, 2H), 1.31 (dt, *J* = 21.0, 7.1 Hz, 2H), 1.11 (s, 3H), 1.04 (s, 3H), 0.94 (d, *J* = 2.7 Hz, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 178.2, 167.6, 158.9, 154.0 (dd, *J* = 290.1, 286.3 Hz), 129.3 (t, *J* = 2.5 Hz), 127.1 (q, *J* = 277.2 Hz), 124.9 – 124.7 (m), 114.1, 91.1, 90.0 (dd, *J* = 21.6, 14.2 Hz), 65.3, 65.3, 60.4, 55.3, 54.8, 54.1, 36.8 (q, *J* = 27.5 Hz), 32.3, 32.0, 30.6, 29.0, 28.6, 22.2, 16.8, 9.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -63.23 (t, *J* = 12.6 Hz, 3F), -91.26 (d, *J* = 46.8 Hz, 1F), -91.68 (d, *J* = 45.0 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₇H₃₃F₅O₅Na 555.2140; found 555.2151.

8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (4ai)



4ai

4ai was prepared according to the general procedure from hex-5-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate **1ai** (111.0 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **4ai** (51.0 mg, 82% yield) was isolated as a white solid.

TLC $R_f = 0.40$ (Hexane/EtOAc = 20:1, v/v).

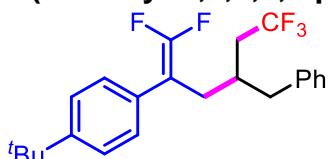
^1H NMR (600 MHz, CDCl_3) δ 7.18 (d, $J = 8.3$ Hz, 2H), 7.09 (d, $J = 7.7$ Hz, 2H), 6.89 (d, $J = 8.7$ Hz, 2H), 6.79 (d, $J = 8.6$ Hz, 2H), 4.10 (t, $J = 6.5$ Hz, 2H), 3.81 (s, 3H), 2.88 – 2.60 (m, 1H), 2.39 (dd, $J = 14.5, 7.0$ Hz, 1H), 2.33 (dd, $J = 14.5, 7.5$ Hz, 1H), 2.12 – 1.83 (m, 3H), 1.76 (td, $J = 8.0, 2.4$ Hz, 1H), 1.65 (dd, $J = 13.1, 6.6$ Hz, 1H), 1.60 (s, 6H), 1.55 – 1.42 (m, 2H), 1.39 – 1.25 (m, 2H), 1.25 – 1.06 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 174.3, 158.9, 155.0, 154.0 (dd, $J = 290.1, 286.3$ Hz), 129.6, 129.3 (t, $J = 2.9$ Hz), 128.1, 126.9 (dd, $J = 508.0, 323.7$ Hz), 124.9 – 124.7 (m), 118.4, 114.1, 90.1 (dd, $J = 21.1, 14.2$ Hz), 79.1, 65.1, 60.9, 55.3, 36.8 (q, $J = 27.4$ Hz), 34.8, 32.3, 31.8, 30.4, 28.4, 25.8, 25.4, 22.0.

^{19}F NMR (565 MHz, CDCl_3) δ -63.24 (t, $J = 12.7$ Hz, 3F), -91.42 (d, $J = 44.9$ Hz, 1F), -91.79 (d, $J = 44.4$ Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{30}\text{H}_{33}\text{F}_5\text{Cl}_2\text{O}_4\text{Na}$ 645.1568; found 645.1579.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(*tert*-butyl)benzene (**5a**)



5a

5a was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-(*tert*-butyl)-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3b** (22.8 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5a** (32.5 mg, 82% yield) was isolated as a clear oil.

TLC $R_f = 0.40$ (Hexane/EtOAc = 50:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.34 (d, $J = 8.5$ Hz, 2H), 7.29 – 7.23 (m, 2H), 7.21 (dt, $J = 9.4, 4.3$ Hz, 1H), 7.11 (dd, $J = 8.4, 1.2$ Hz, 2H), 7.03 – 6.93 (m, 2H), 2.68 (dd, $J = 14.0, 6.2$ Hz, 1H),

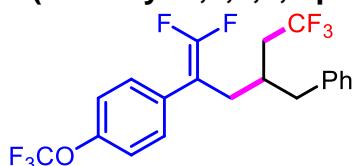
2.60 (dd, J = 14.0, 6.7 Hz, 1H), 2.56 – 2.48 (m, 1H), 2.48 – 2.31 (m, 1H), 2.17 – 1.93 (m, 3H), 1.33 (s, 9H).

^{13}C NMR (151 MHz, CDCl_3) δ 154.1 (dd, J = 291.1, 286.7 Hz), 150.5, 139.0, 129.6 – 129.3 (m), 129.1, 128.5, 127.8 (t, J = 3.0 Hz), 126.4, 125.5, 90.3 (dd, J = 21.0, 13.8 Hz), 39.6, 36.5 (q, J = 27.5 Hz), 34.6, 32.7, 31.4, 31.3.

^{19}F NMR (565 MHz, CDCl_3) δ -62.64 (t, J = 12.1 Hz, 3F), -90.38 (d, J = 43.0 Hz, 1F), -90.65 (d, J = 44.5 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{23}\text{H}_{25}\text{F}_5\text{Na}$ 419.1769; found 419.1775.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(trifluoromethoxy)benzene (5b)



5b

5b was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-(trifluoromethoxy)-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3c** (25.6 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5b** (37.3 mg, 88% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

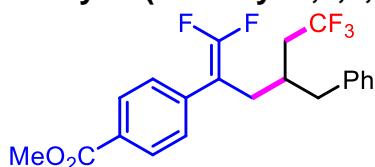
^1H NMR (600 MHz, CDCl_3) δ 7.29 – 7.24 (m, 2H), 7.24 – 7.20 (m, 1H), 7.19 – 7.12 (m, 4H), 7.00 – 6.96 (m, 2H), 2.76 – 2.59 (m, 2H), 2.56 – 2.47 (m, 1H), 2.42 (ddt, J = 14.8, 6.9, 2.2 Hz, 1H), 2.13 – 1.99 (m, 2H), 1.94 (dt, J = 13.6, 6.8 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 154.2 (dd, J = 291.9, 287.8 Hz), 148.4, 138.8, 131.3 – 130.9 (m), 129.6 (t, J = 3.1 Hz), 129.0, 128.6, 127.0 (dd, J = 554.7, 277.1 Hz), 126.6, 121.0, 89.7 (dd, J = 21.4, 14.5 Hz), 39.8, 36.7 (q, J = 27.5 Hz), 32.7, 31.3, 29.7.

^{19}F NMR (565 MHz, CDCl_3) δ -57.84 (s), -62.88 (t, J = 12.3 Hz, 3F), -89.37 (d, J = 40.5 Hz, 1F), -89.50 (d, J = 40.9 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{20}\text{H}_{16}\text{F}_8\text{ONa}$ 447.0966; found 447.0960.

methyl 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzoate (5c)



5c

5c was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and

methyl 4-(3,3,3-trifluoroprop-1-en-2-yl)benzoate **3d** (23.0 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5c** (33.0 mg, 83% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

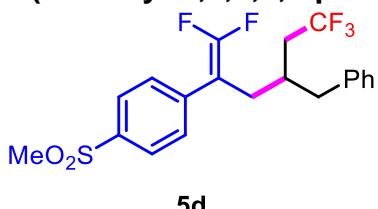
^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 8.4 Hz, 2H), 7.29 – 7.21 (m, 5H), 6.99 (d, J = 6.9 Hz, 2H), 3.93 (s, 3H), 2.64 (d, J = 7.1 Hz, 2H), 2.60 – 2.51 (m, 1H), 2.47 (dd, J = 14.8, 6.9 Hz, 1H), 2.13 – 1.98 (m, 2H), 1.94 (dt, J = 13.5, 6.8 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 166.7, 154.4 (dd, J = 293.4, 288.7 Hz), 138.8, 137.3 (t, J = 3.7 Hz), 129.8, 129.3, 129.0, 128.6, 128.1 (t, J = 3.1 Hz), 127.1 (dd, J = 590.3, 313.0 Hz), 126.6, 90.4 (dd, J = 21.8, 13.2 Hz), 52.2, 39.8, 36.7 (q, J = 27.7 Hz), 32.8, 31.1.

^{19}F NMR (565 MHz, CDCl_3) δ -62.87 (t, J = 12.4 Hz, 3F), -88.01 (d, J = 37.5 Hz, 1F), -88.20 (d, J = 38.2 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{21}\text{H}_{19}\text{F}_5\text{O}_2\text{Na}$ 421.1197; found 421.1204.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(methylsulfonyl)benzene (**5d**)



5d was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-(methylsulfonyl)-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3e** (25.0 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5d** (34.3 mg, 82% yield) was isolated as a white solid.

TLC R_f = 0.30 (Hexane/EtOAc = 10:1, v/v).

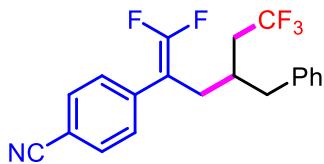
^1H NMR (600 MHz, CDCl_3) δ 7.88 (d, J = 8.5 Hz, 2H), 7.36 – 7.16 (m, 5H), 7.01 (d, J = 7.1 Hz, 2H), 3.08 (s, 3H), 2.70 (dd, J = 13.9, 6.9 Hz, 1H), 2.62 (dd, J = 13.9, 7.6 Hz, 1H), 2.56 (dd, J = 15.0, 7.4 Hz, 1H), 2.52 – 2.40 (m, 1H), 2.07 (ddd, J = 11.5, 9.6, 6.5 Hz, 2H), 1.91 (dt, J = 13.7, 6.9 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 154.6 (dd, J = 294.6, 289.5 Hz), 139.5, 138.6, 138.5 – 138.3 (m), 129.0, 129.0 (t, J = 3.3 Hz), 128.7, 127.7, 126.7, 90.0 (dd, J = 22.5, 12.5 Hz), 44.5, 40.0, 36.8 (q, J = 28.0 Hz), 32.9, 31.0.

^{19}F NMR (565 MHz, CDCl_3) δ -62.88 (t, J = 11.7 Hz, 3F), -86.72 (d, J = 34.6 Hz, 1F), -87.07 (d, J = 34.7 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{20}\text{H}_{19}\text{F}_5\text{O}_2\text{SNa}$ 441.0918; found 441.0925.

4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzonitrile (**5e**)



5e

5e was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 4-(3,3,3-trifluoroprop-1-en-2-yl)benzonitrile **3f** (19.7 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5e** (31.0 mg, 85% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

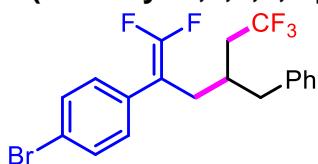
^1H NMR (600 MHz, CDCl_3) δ 7.60 (d, J = 8.5 Hz, 2H), 7.33 – 7.23 (m, 3H), 7.21 (dd, J = 8.5, 1.0 Hz, 2H), 7.06 – 6.94 (m, 2H), 2.69 (dd, J = 13.8, 6.9 Hz, 1H), 2.61 (dd, J = 13.9, 7.6 Hz, 1H), 2.57 – 2.51 (m, 1H), 2.47 (ddt, J = 15.0, 6.7, 2.3 Hz, 1H), 2.18 – 1.96 (m, 2H), 1.89 (dt, J = 13.7, 6.8 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 154.5 (dd, J = 294.8, 289.5 Hz), 138.6, 137.5 – 137.4 (m), 132.4, 129.0, 128.7 (t, J = 3.3 Hz), 128.7, 126.9 (q, J = 277.0 Hz), 126.7, 118.5, 111.4, 90.1 (dd, J = 22.2, 12.9 Hz), 40.0, 36.8 (q, J = 27.8 Hz), 32.9, 30.9, 29.7.

^{19}F NMR (565 MHz, CDCl_3) δ -62.96 (t, J = 12.6 Hz, 3F), -86.79 (d, J = 34.7 Hz, 1F), -86.99 (d, J = 34.7 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{20}\text{H}_{16}\text{F}_5\text{NNa}$ 388.1095; found 388.1104.

1-(4-benzyl-1,6,6,6-pentafluorohex-1-en-2-yl)-4-bromobenzene (**5f**)



5f

5f was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-bromo-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3g** (25.0 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5f** (37.6 mg, 90% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

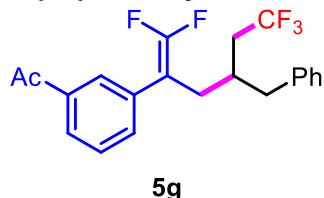
^1H NMR (600 MHz, CDCl_3) δ 7.45 (d, J = 8.5 Hz, 2H), 7.28 (dd, J = 10.1, 4.6 Hz, 2H), 7.24 – 7.18 (m, 1H), 7.02 – 6.96 (m, 4H), 2.72 – 2.57 (m, 2H), 2.50 (dd, J = 14.7, 7.3 Hz, 1H), 2.41 (ddt, J = 14.8, 6.9, 2.2 Hz, 1H), 2.14 – 1.99 (m, 2H), 1.93 (dt, J = 13.6, 6.8 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 154.1 (dd, J = 292.1, 287.8 Hz), 138.8, 131.8, 131.5 – 131.1 (m), 129.7 (t, J = 3.1 Hz), 129.1, 128.6, 127.0 (q, J = 277.0 Hz), 126.6, 121.6, 89.9 (dd, J = 21.8, 14.0 Hz), 39.8, 38.0 – 35.0 (m), 32.7, 31.2, 29.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.85 (t, *J* = 12.0 Hz, 3F), -89.22 (d, *J* = 40.2 Hz, 1F), -89.44 (d, *J* = 39.2 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₆BrF₅Na 441.0248; found 441.0243.

1-(3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)ethan-1-one (5g)



5g was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-(3-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)ethan-1-one **3h** (21.4 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5g** (35.5 mg, 93% yield) was isolated as a clear oil.

TLC *Rf* = 0.30 (Hexane/EtOAc = 50:1, v/v).

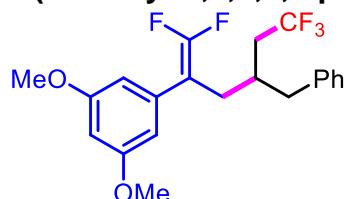
¹H NMR (600 MHz, CDCl₃) δ 8.04 – 7.82 (m, 1H), 7.75 (s, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.35 (d, *J* = 7.7 Hz, 1H), 7.29 – 7.23 (m, 2H), 7.21 (ddd, *J* = 7.4, 3.7, 1.2 Hz, 1H), 6.99 (d, *J* = 6.9 Hz, 2H), 2.66 (dd, *J* = 6.9, 3.6 Hz, 2H), 2.58 (dd, *J* = 7.1, 2.6 Hz, 1H), 2.56 (s, 3H), 2.53 – 2.37 (m, 1H), 2.06 (qd, *J* = 11.3, 6.3 Hz, 2H), 1.94 (dt, *J* = 13.6, 6.8 Hz, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 197.6, 154.3 (dd, *J* = 291.7, 288.6 Hz), 138.8, 137.5, 133.1, 132.8 (t, *J* = 3.0 Hz), 129.0, 129.0, 128.6, 128.0 – 127.8 (m), 127.5 (d, *J* = 433.8 Hz), 127.5, 126.6, 90.1 (dd, *J* = 20.6, 15.2 Hz), 39.9, 36.6 (q, *J* = 28.0 Hz), 32.7, 31.4, 26.6.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.88 (t, *J* = 11.7 Hz, 3F), -89.23 (d, *J* = 40.7 Hz, 1F), -89.32 (d, *J* = 41.3 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₁H₁₉F₅ONa 405.1248; found 405.1255.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-3,5-dimethoxybenzene (5h)



5h was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1,3-dimethoxy-5-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3i** (23.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5h** (35.2 mg, 88% yield) was isolated as a clear oil.

TLC *Rf* = 0.50 (Hexane/EtOAc = 20:1, v/v).

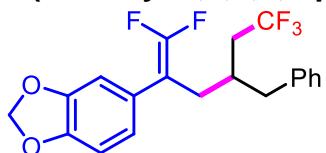
¹H NMR (600 MHz, CDCl₃) δ 7.26 (t, J = 7.4 Hz, 2H), 7.20 (t, J = 7.3 Hz, 1H), 7.02 (d, J = 7.1 Hz, 2H), 6.40 (t, J = 2.2 Hz, 1H), 6.33 (d, J = 1.1 Hz, 2H), 3.74 (s, 6H), 2.65 (qd, J = 13.9, 6.6 Hz, 2H), 2.55 – 2.46 (m, 1H), 2.45 – 2.35 (m, 1H), 2.10 – 1.96 (m, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 160.8, 154.2 (dd, J = 291.7, 286.8 Hz), 139.0, 134.4 – 134.3 (m), 129.1, 128.5, 127.1 (q, J = 277.3 Hz), 126.4, 106.3 (t, J = 3.0 Hz), 100.0, 90.7 (dd, J = 21.6, 13.7 Hz), 55.3, 39.8, 36.5 (q, J = 27.7 Hz), 32.7, 31.5, 29.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.68 (t, J = 11.7 Hz, 3F), -88.83 (d, J = 41.0 Hz, 1F), -90.12 (d, J = 40.5 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₁H₂₁F₅O₂Na 423.1354; found 423.1350.

5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzo[d][1,3]dioxole (5i)



5i

5i was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 5-(3,3,3-trifluoroprop-1-en-2-yl)benzo[d][1,3]dioxole **3j** (21.6 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5i** (27.3mg, 71% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 20:1, v/v).

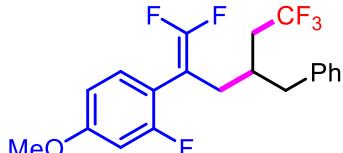
¹H NMR (600 MHz, CDCl₃) δ 7.33 – 7.25 (m, 2H), 7.22 (dd, J = 8.4, 6.3 Hz, 1H), 7.02 (d, J = 7.1 Hz, 2H), 6.77 (d, J = 8.0 Hz, 1H), 6.64 (s, 1H), 6.61 (d, J = 8.1 Hz, 1H), 5.98 (dd, J = 3.6, 1.5 Hz, 2H), 2.72 – 2.58 (m, 2H), 2.51 – 2.43 (m, 1H), 2.41 – 2.31 (m, 1H), 2.14 – 1.96 (m, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 154.1 (dd, J = 290.7, 286.7 Hz), 147.9, 147.0, 139.0, 129.1, 128.5, 127.1 (q, J = 277.1 Hz), 126.5, 126.1 – 126.0 (m), 121.8 (t, J = 2.9 Hz), 108.6 (t, J = 3.2 Hz), 108.4, 101.2, 90.3 (dd, J = 21.9, 13.9 Hz), 39.7, 36.5 (q, J = 27.7 Hz), 32.6, 31.7, 29.7.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.76 (t, J = 11.6 Hz, 3F), -90.39 (d, J = 43.6 Hz, 1F), -91.10 (d, J = 43.2 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₀H₁₇F₅O₂Na 407.1041; found 407.1047.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-fluoro-4-methoxybenzene (5j)



5j

5j was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 2-fluoro-1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3k** (22.0 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5j** (31.0 mg, 80% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

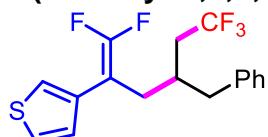
^1H NMR (600 MHz, CDCl_3) δ 7.29 – 7.22 (m, 2H), 7.22 – 7.14 (m, 1H), 7.01 (dd, J = 8.2, 6.8 Hz, 1H), 6.97 (d, J = 7.1 Hz, 2H), 6.72 – 6.55 (m, 2H), 3.69 (s, 3H), 2.73 – 2.56 (m, 2H), 2.46 (dd, J = 14.5, 7.4 Hz, 1H), 2.41 – 2.29 (m, 1H), 2.20 – 2.04 (m, 1H), 2.04 – 1.91 (m, 1H), 1.82 (dt, J = 13.6, 6.8 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 163.5 (d, J = 247.1 Hz), 158.5 – 158.4 (m), 153.8 (t, J = 287.7 Hz), 139.2, 131.5 (d, J = 10.1 Hz), 129.0, 128.4, 127.1 (q, J = 277.2 Hz), 126.3, 117.1, 107.1 (d, J = 21.6 Hz), 99.3 (d, J = 26.1 Hz), 86.7 (dd, J = 23.7, 16.7 Hz), 55.5, 39.6, 36.4 (q, J = 27.4 Hz), 32.6, 31.5, 29.7.

^{19}F NMR (565 MHz, CDCl_3) δ -62.87 (t, J = 12.5 Hz, 3F), -88.03 (d, J = 41.2 Hz, 1F), -92.47 (d, J = 40.7 Hz, 1F), -105.72 – -115.29 (m, 1F).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{20}\text{H}_{18}\text{F}_6\text{ONa}$ 411.1154; found 411.1161.

3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)thiophene (5k)



5k was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 3-(3,3,3-trifluoroprop-1-en-2-yl)thiophene **3l** (17.8 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5k** (27.7 mg, 80% yield) was isolated as a clear oil.

TLC R_f = 0.50 (Hexane/EtOAc = 20:1, v/v).

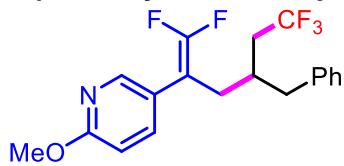
^1H NMR (600 MHz, CDCl_3) δ 7.35 – 7.27 (m, 3H), 7.23 (t, J = 7.4 Hz, 1H), 7.07 (d, J = 7.2 Hz, 2H), 7.01 (ddd, J = 5.0, 2.1, 1.4 Hz, 1H), 6.94 (dd, J = 2.9, 1.1 Hz, 1H), 2.67 (qd, J = 13.8, 7.2 Hz, 2H), 2.50 (dd, J = 14.7, 7.3 Hz, 1H), 2.45 – 2.31 (m, 1H), 2.21 – 2.03 (m, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 153.5 (dd, J = 293.8, 286.1 Hz), 138.0, 131.5 (t, J = 4.3 Hz), 128.1, 127.6, 126.1 (q, J = 277.0 Hz), 125.7 (dd, J = 6.4, 2.0 Hz), 125.5, 124.8, 121.1 (t, J = 5.0 Hz), 85.7 (dd, J = 23.1, 13.1 Hz), 38.8, 35.7 (q, J = 27.5 Hz), 32.3, 30.2.

^{19}F NMR (565 MHz, CDCl_3) δ -62.63 (t, J = 12.2 Hz), -89.37 (d, J = 41.0 Hz), -90.12 (d, J = 41.2 Hz).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{F}_5\text{SNa}$ 369.0707; found 369.0710.

5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-methoxypyridine (5l)



5l

5l was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 2-methoxy-5-(3,3,3-trifluoroprop-1-en-2-yl)pyridine **3m** (20.3 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5l** (31.5 mg, 85% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 20:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.95 (s, 1H), 7.31 – 7.18 (m, 3H), 7.15 (t, J = 7.3 Hz, 1H), 6.95 (d, J = 7.2 Hz, 2H), 6.65 (d, J = 8.6 Hz, 1H), 3.88 (s, 3H), 2.58 (d, J = 7.1 Hz, 2H), 2.43 (dd, J = 14.7, 7.1 Hz, 1H), 2.32 (dd, J = 14.8, 7.1 Hz, 1H), 2.05 – 1.92 (m, 2H), 1.89 (dt, J = 13.6, 6.7 Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 163.4, 154.1 (dd, J = 290.8, 287.9 Hz), 146.4 (t, J = 3.3 Hz), 138.7, 138.1 (t, J = 2.5 Hz), 129.1, 128.6, 127.0 (q, J = 277.5 Hz), 126.6, 121.3 (t, J = 2.6 Hz), 110.9, 87.6 (dd, J = 21.9, 15.2 Hz), 53.6, 39.8, 36.6 (q, J = 27.6 Hz), 32.6, 31.3.

^{19}F NMR (565 MHz, CDCl_3) δ -62.82 (t, J = 12.2 Hz, 3F), -89.83 (d, J = 42.7 Hz, 1F), -89.96 (d, J = 42.5 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{19}\text{H}_{18}\text{F}_5\text{NONa}$ 394.1201; found 394.1208.

2-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)naphthalene (5m)



5m

5m was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 1-(3,3,3-trifluoroprop-1-en-2-yl)naphthalene **3n** (22.2 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5m** (26.5 mg, 68% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

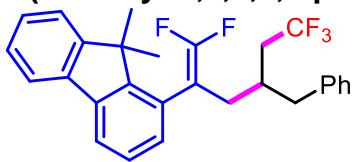
^1H NMR (600 MHz, CDCl_3) δ 7.83 (dd, J = 6.0, 3.4 Hz, 1H), 7.81 (d, J = 8.6 Hz, 1H), 7.74 (dd, J = 6.0, 3.4 Hz, 1H), 7.55 (s, 1H), 7.50 (dd, J = 6.2, 3.2 Hz, 2H), 7.31 (dt, J = 8.5, 1.6 Hz, 1H), 7.28 – 7.17 (m, 3H), 7.00 (d, J = 6.5 Hz, 2H), 2.69 (d, J = 7.0 Hz, 2H), 2.62 (dd, J = 14.6, 7.3 Hz, 1H), 2.59 – 2.49 (m, 1H), 2.16 – 1.96 (m, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 154.4 (dd, *J* = 291.9, 287.5 Hz), 139.0, 133.2, 132.6, 130.2 – 129.6 (m), 129.1, 128.6, 128.3, 128.0, 127.6, 127.3 (t, *J* = 3.1 Hz), 127.1 (d, *J* = 277.3 Hz), 126.5, 126.4, 126.3, 125.8 (t, *J* = 2.7 Hz), 90.7 (dd, *J* = 20.8, 14.1 Hz), 39.9, 36.7 (q, *J* = 27.7 Hz), 32.8 31.3.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.74 (t, *J* = 12.4 Hz, 3F), -89.64 (d, *J* = 41.4 Hz, 1F), -89.79 (d, *J* = 42.7 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₃H₁₉F₅Na 413.1299; found 413.1304.

1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-9,9-dimethyl-9H-fluorene (5n)



5n

5n was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 9,9-dimethyl-1-(3,3,3-trifluoroprop-1-en-2-yl)-9H-fluorene **3o** (28.8 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5n** (28.3 mg, 62% yield) was isolated as a white solid.

TLC *Rf* = 0.40 (Hexane/EtOAc = 50:1, v/v).

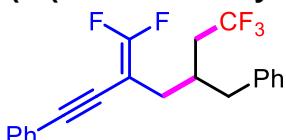
¹H NMR (600 MHz, CDCl₃) δ 7.78 – 7.71 (m, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.49 – 7.42 (m, 1H), 7.34 (pd, *J* = 7.4, 1.5 Hz, 2H), 7.26 – 7.22 (m, 3H), 7.19 (ddt, *J* = 11.0, 7.9, 1.4 Hz, 2H), 7.02 – 6.96 (m, 2H), 2.71 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.62 (dd, *J* = 13.8, 7.3 Hz, 2H), 2.49 (dd, *J* = 14.7, 7.1 Hz, 1H), 2.04 (dd, *J* = 22.1, 19.9, 10.7, 5.4 Hz, 3H), 1.47 (s, 3H), 1.45 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 154.2 (dd, *J* = 290.7, 286.9 Hz), 154.0, 153.8, 139.1, 138.8, 138.6, 131.2 (t, *J* = 3.4 Hz), 129.0, 128.5, 128.2 (d, *J* = 55.8 Hz), 127.5, 127.2 (t, *J* = 2.9 Hz), 127.1, 126.4, 122.7, 122.3 (t, *J* = 2.9 Hz), 120.1, 91.0 (dd, *J* = 20.9, 14.0 Hz), 46.9, 39.8, 36.5 (q, *J* = 27.6 Hz), 32.7, 31.6, 29.7, 27.1, 26.9.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.81 (t, *J* = 11.8 Hz, 3F), -90.00 (d, *J* = 42.6 Hz, 1F), -90.71 (d, *J* = 44.1 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₈H₂₅F₅Na 479.1769; found 479.1761.

(3-(difluoromethylene)-5-(2,2,2-trifluoroethyl)hex-1-yne-1,6-diyl)dibenzene (5o)



5o

5o was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and (3-

(trifluoromethyl)but-3-en-1-yn-1-yl)benzene **3p** (19.6 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5o** (32.4 mg, 89% yield) was isolated as a clear oil.

TLC R_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

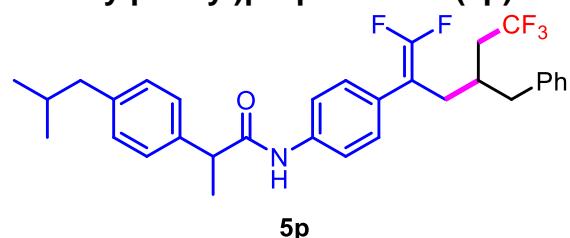
^1H NMR (600 MHz, CDCl_3) δ 7.43 (dd, J = 6.6, 3.0 Hz, 2H), 7.34 (dd, J = 4.5, 2.2 Hz, 3H), 7.31 (t, J = 7.5 Hz, 2H), 7.23 (t, J = 7.4 Hz, 1H), 7.19 (d, J = 7.1 Hz, 2H), 2.82 (dd, J = 13.9, 6.8 Hz, 1H), 2.72 (dd, J = 13.9, 7.5 Hz, 1H), 2.54 – 2.41 (m, 1H), 2.35 – 2.26 (m, 1H), 2.26 – 2.20 (m, 1H), 2.20 – 2.07 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.2 (dd, J = 297.3, 294.1 Hz), 138.8, 131.4, 129.2, 128.6, 128.4, 127.1 (q, J = 277.7 Hz), 126.5, 122.6, 94.4 (t, J = 5.6 Hz), 80.3 (dd, J = 8.0, 3.9 Hz), 39.5, 36.2 (q, J = 27.5 Hz), 33.4, 30.9.

^{19}F NMR (565 MHz, CDCl_3) δ -62.64 (t, J = 11.1 Hz, 3F), -78.07 (d, J = 14.3 Hz, 1F), -83.72 (d, J = 14.2 Hz, 1F).

HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{21}\text{H}_{17}\text{F}_5\text{Na}$ 387.1143; found 387.1147.

N-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-2-(4-isobutylphenyl)propanamide (5p)



5p was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 2-(4-isobutylphenyl)-N-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)propanamide **3q** (37.5 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5p** (38.0 mg, 70% yield) was isolated as a white solid.

TLC R_f = 0.30 (Hexane/EtOAc = 5:1, v/v).

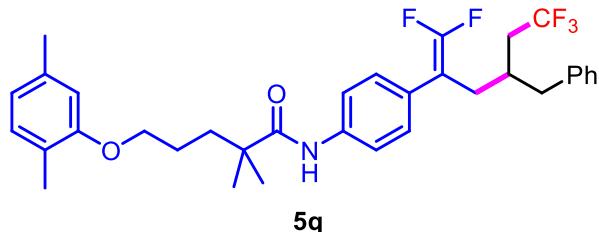
^1H NMR (600 MHz, CDCl_3) δ 7.46 (dd, J = 16.4, 8.1 Hz, 1H), 7.34 – 7.26 (m, 2H), 7.24 (t, J = 8.1 Hz, 2H), 7.21 – 7.13 (m, 4H), 7.08 (d, J = 25.8 Hz, 1H), 6.99 (t, J = 6.8 Hz, 3H), 6.87 (d, J = 7.6 Hz, 1H), 3.69 (qd, J = 7.1, 3.3 Hz, 1H), 2.62 (d, J = 7.0 Hz, 2H), 2.48 (d, J = 7.2 Hz, 2H), 2.47 (dd, J = 12.5, 7.3 Hz, 2H), 2.40 (dd, J = 14.7, 6.9 Hz, 1H), 2.10 – 1.98 (m, 2H), 1.95 (dt, J = 12.0, 5.9 Hz, 1H), 1.91 – 1.82 (m, 1H), 1.60 (dd, J = 7.1, 3.8 Hz, 3H), 0.91 (dd, J = 6.6, 1.7 Hz, 6H).

^{13}C NMR (151 MHz, CDCl_3) δ 172.6, 154.1 (dd, J = 290.5, 288.6 Hz), 141.2, 139.0, 138.2, 138.0, 138.0, 133.3, 129.9, 129.2 (d, J = 2.2 Hz), 128.5, 127.4, 127.4, 127.0 (q, J = 277.4 Hz), 126.4, 123.9, 119.1, 119.0 (d, J = 4.8 Hz), 90.3 (dd, J = 21.8, 13.8 Hz), 47.8, 45.0, 39.7 (d, J = 3.9 Hz), 36.6 (qd, J = 27.5, 6.9 Hz), 32.8, 31.4, 30.2, 29.7, 22.4, 18.6 (d, J = 11.7 Hz).

¹⁹F NMR (565 MHz, CDCl₃) δ -62.69 (t, *J* = 14.1 Hz, 3F), -89.29 (d, *J* = 18.2 Hz, 1F), -89.96 (d, *J* = 40.9 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₃₂H₃₄F₅NONa 566.2453; found 566.2461.

N-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-5-(2,5-dimethylphenoxy)-2,2-dimethylpentanamide (5q)



5q was prepared according to the general procedure from allylbenzene **1a** (35.4 mg, 0.30 mmol, 3.0 equiv), sodium trifluoromethanesulfinate **2a** (46.8 mg, 0.30 mmol, 3.0 equiv) and 5-(2,5-dimethylphenoxy)-2,2-dimethyl-N-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)pentanamide **3r** (41.9 mg, 0.10 mmol, 1.0 equiv). The desired difluoroalkene **5q** (42.8 mg, 73% yield) was isolated as a white solid.

TLC *Rf* = 0.30 (Hexane/EtOAc = 5:1, v/v).

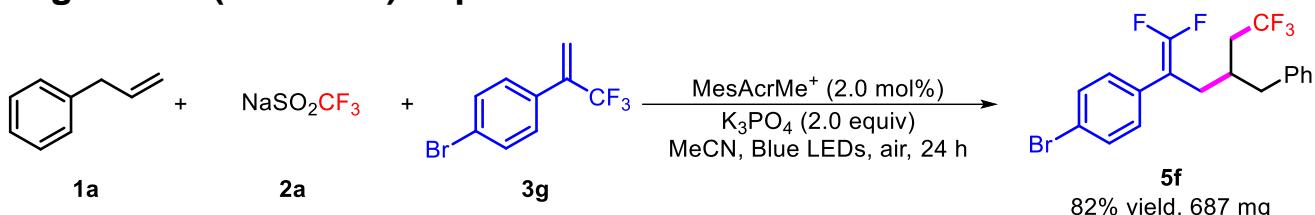
¹H NMR (600 MHz, CDCl₃) δ 7.56 (d, *J* = 8.1 Hz, 1H), 7.39 – 7.24 (m, 5H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 7.7 Hz, 2H), 7.00 (d, *J* = 7.4 Hz, 1H), 6.91 (d, *J* = 7.7 Hz, 1H), 6.67 (d, *J* = 7.4 Hz, 1H), 6.61 (s, 1H), 3.96 (s, 2H), 2.81 – 2.58 (m, 2H), 2.51 (dd, *J* = 14.6, 6.8 Hz, 1H), 2.43 (dd, *J* = 14.7, 6.8 Hz, 1H), 2.29 (s, 3H), 2.17 (s, 3H), 2.10 – 1.93 (m, 3H), 1.84 (d, *J* = 1.3 Hz, 4H), 1.35 (s, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 175.8, 156.9, 154.2 (dd, *J* = 291.8, 287.2 Hz), 139.0, 138.2, 136.6, 133.4, 130.4, 129.3, 129.2, 128.5, 127.1 (q, *J* = 277.4 Hz), 126.41 (s), 124.07 (s), 123.5, 120.9, 119.6, 119.5, 112.2, 90.4 (dd, *J* = 21.6, 13.7 Hz), 67.9, 42.9, 39.7, 37.7, 36.6 (q, *J* = 27.6 Hz), 32.8, 31.5, 29.7, 25.7, 25.6, 25.2, 21.4, 15.8.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.65 (t, *J* = 12.3 Hz, 3F), -89.29 (d, *J* = 40.8 Hz, 1F), -89.99 (d, *J* = 40.9 Hz, 1F).

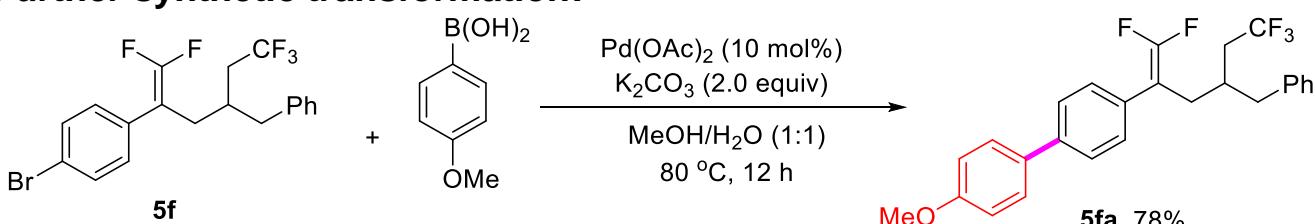
HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₃₄H₃₈F₅NO₂Na 610.2715; found 610.2728.

5. Large-scale experiment and further synthetic transformation: Large Scale (2.0 mmol) Experiment:



To an oven-dried 50 mL reaction vial equipped with a stir bar was added MesAcrMe⁺ (16.0 mg, 0.04 mmol, 2 mol%), NaSO₂CF₃ (936.0 mg, 6.0 mmol, 3.0 equiv), and K₃PO₄ (848.2 mg, 4.0 mmol, 2.0 equiv). The vial was then charged with allylbenzene **1a** (709.1 mg, 6.0 mmol, 3.0 equiv) and 1-bromo-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3g** (500.0 mg, 2.0 mmol, 1.0 equiv) in anhyd MeCN (20 mL) *via* a syringe. The cap was sealed with Parafilm®, and the solution was irradiated with a 30 W blue LED light at room temperature for 24 hours. The temperature of the reaction was maintained at approximately 27 °C *via* a fan. The solution was stirred vigorously while being irradiated. Once judged to be complete, the solution was transferred to a separatory funnel and diluted with deionized H₂O (50 mL) and Et₂O (50 mL). The layers were separated, and the aq layer was extracted with Et₂O (4 X 40 mL). The combined organic layers were washed with deionized H₂O (2 X 50 mL) followed by brine (100 mL). The combined organic layers were dried (Na₂SO₄), and the solvent was removed *in vacuo* by rotary evaporation. Further purification was accomplished by SiO₂ column chromatography (gradient Hexane/EtOAc) to give the desired product **5f** (82% yield, 687 mg) as a white solid.

Further synthetic transformation:



According to the literature,⁵ to a 10 mL Schlenk flask was added difluoroalkene **5f** (41.8 mg, 0.10 mmol, 1.0 equiv), Pd(OAc)₂ (2.3 mg, 0.01 mmol, 10 mol%), potassium carbonate (27.6 mg, 0.20 mmol, 2.0 equiv) and 4-methoxyphenylboronic acid (30.4 mg, 0.20 mmol, 2.0 equiv). Then MeOH (0.5 mL) and H₂O (0.5 mL) was added by syringe. The reaction mixture was stirred at 80 °C for 12 h. After cooling to room temperature, the solvent was removed under vacuum, and the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 50:1) to afford the product as a white solid **5fa** (34.8 mg, 78% yield).

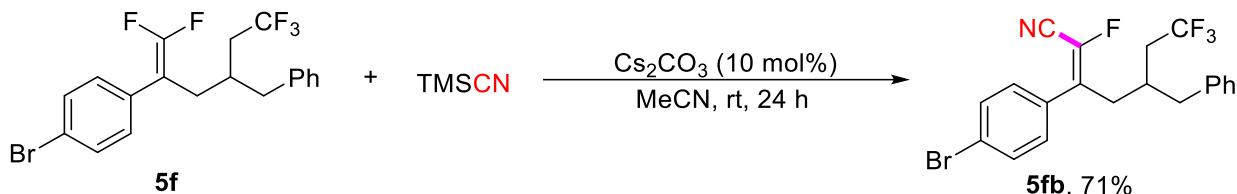
TLC R_f = 0.50 (Hexane/EtOAc = 5:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.57 (d, *J* = 8.7 Hz, 2H), 7.54 (d, *J* = 8.3 Hz, 2H), 7.28 (t, *J* = 7.4 Hz, 2H), 7.23 (t, *J* = 6.8 Hz, 3H), 7.04 (d, *J* = 7.2 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 2H), 3.87 (s, 3H), 2.71 (dd, *J* = 13.8, 6.6 Hz, 1H), 2.66 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.58 (dd, *J* = 14.6, 6.3 Hz, 1H), 2.48 (dd, *J* = 14.6, 6.4 Hz, 1H), 2.22 – 1.81 (m, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 159.3, 154.2 (dd, *J* = 291.5, 287.2 Hz), 139.9, 139.0, 132.9, 130.7, 129.2, 128.5, 128.5 (t, *J* = 3.1 Hz), 128.1, 126.8, 126.5, 114.3, 90.4 (dd, *J* = 21.2, 13.7 Hz), 55.4, 39.7, 36.6 (q, *J* = 27.5 Hz), 32.8, 31.3.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.65 (t, *J* = 11.9 Hz, 3F), -89.84 (d, *J* = 41.8 Hz, 1F), -90.10 (d, *J* = 41.9 Hz, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₆H₂₃F₅ONa 469.1561; found 469.1570.



According to the literature,⁹ to a 10 mL Schlenk flask was added difluoroalkene **5f** (41.8 mg, 0.10 mmol, 1.0 equiv), Cs₂CO₃ (3.3 mg, 0.01 mmol, 10 mol%), and TMSCN (30.0 mg, 0.30 mmol, 3.0 equiv), and then anhydrous MeCN (1.0 mL). The reaction mixture was stirred at rt for 24 h. Then the solvent was removed under vacuum, and the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 30:1) to afford the product as a white solid **5fb** (30.2 mg, 71% yield).

TLC R_f = 0.40 (Hexane/EtOAc = 30:1, v/v).

¹H NMR (600 MHz, CDCl₃) δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.20 (t, *J* = 7.1 Hz, 2H), 7.17 (d, *J* = 6.9 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 7.2 Hz, 2H), 2.69 (ddd, *J* = 14.4, 6.9, 3.5 Hz, 1H), 2.63 – 2.54 (m, 2H), 2.50 (dd, *J* = 13.9, 7.4 Hz, 1H), 2.05 – 1.98 (m, 1H), 1.94 – 1.90 (m, 1H), 1.89 – 1.79 (m, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 138.1, 137.7 (d, *J* = 16.0 Hz), 132.5, 131.3, 130.8 (d, *J* = 3.5 Hz), 129.6 (d, *J* = 2.8 Hz), 129.0, 128.7, 126.9, 126.7 (d, *J* = 277.5 Hz), 124.7, 112.0 (d, *J* = 47.2 Hz), 40.2, 37.0 (q, *J* = 28.0 Hz), 33.6, 32.8.

¹⁹F NMR (565 MHz, CDCl₃) δ -62.92 (t, *J* = 11.7 Hz, 3F), -121.33 (s, 1F).

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₀H₁₆F₄NBrNa 448.0295; found 448.0304.



According to the literature,¹⁰ to a 10 mL Schlenk flask was added difluoroalkene **5f** (41.8 mg, 0.10 mmol, 1.0 equiv), KO^tBu (44.8 mg, 0.20 mmol, 2.0 equiv), and then anhydrous THF (1.0 mL). The reaction mixture was stirred at rt for 24 h. Then the solvent was removed under vacuum, and the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 30:1) to afford the product as a white solid **5fc** (30.0 mg, 63% yield, *E*:*Z* = 1.5:1).

TLC *R*_f = 0.40 (Hexane/EtOAc = 50:1, v/v).

For *Z* isomer:

¹H NMR (600 MHz, CDCl₃) δ 7.37 (d, *J* = 8.5 Hz, 2H), 7.26 (t, *J* = 7.4 Hz, 2H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.08 (d, *J* = 8.5 Hz, 2H), 7.01 (d, *J* = 7.1 Hz, 2H), 2.80 – 2.56 (m, 2H), 2.51 (ddd, *J* = 14.5, 7.8, 2.4 Hz, 1H), 2.41 (ddd, *J* = 14.5, 6.7, 2.7 Hz, 1H), 2.11 – 1.94 (m, 2H), 1.89 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.22 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 155.5, 153.6, 139.4, 135.4 (d, *J* = 5.1 Hz), 131.1, 130.3 (d, *J* = 3.1 Hz), 129.2, 128.4, 126.3, 120.0, 98.3 (d, *J* = 36.0 Hz), 84.0 (d, *J* = 3.2 Hz), 39.8, 36.5 (q, *J* = 27.4 Hz), 33.1, 31.9, 28.6 (d, *J* = 1.7 Hz).

¹⁹F NMR (565 MHz, CDCl₃) δ -62.82 (t, *J* = 12.7 Hz, 3F), -75.14 (s, 1F).

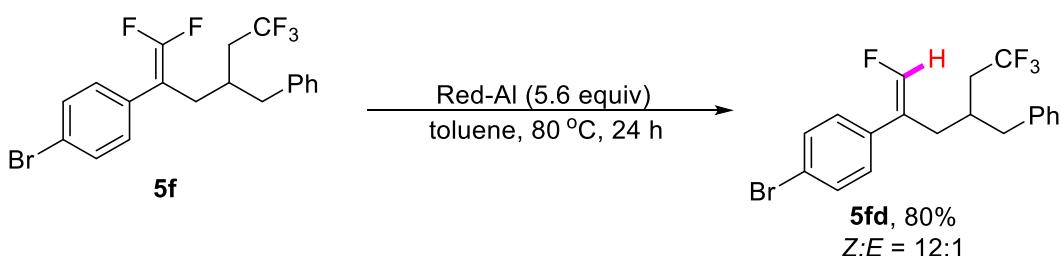
For *E* isomer:

¹H NMR (600 MHz, CDCl₃) δ 7.40 (d, *J* = 8.5 Hz, 2H), 7.29 – 7.23 (m, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.04 (d, *J* = 7.6 Hz, 2H), 7.00 (d, *J* = 7.3 Hz, 2H), 2.69 – 2.51 (m, 2H), 2.43 (dd, *J* = 14.3, 7.3 Hz, 1H), 2.35 (dd, *J* = 14.3, 6.8 Hz, 1H), 2.10 – 1.85 (m, 3H), 1.39 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 154.8, 152.9, 139.4, 134.7, 131.3, 130.1 (d, *J* = 4.1 Hz), 129.2, 128.4, 126.3, 120.2, 97.3 (d, *J* = 27.0 Hz), 83.4 (d, *J* = 2.9 Hz), 39.8, 36.5 (q, *J* = 27.3 Hz), 33.3, 33.0, 28.8 (d, *J* = 1.8 Hz).

¹⁹F NMR (565 MHz, CDCl₃) δ -62.73 (t, *J* = 11.8 Hz, 3F), -78.52 (s, 1F).

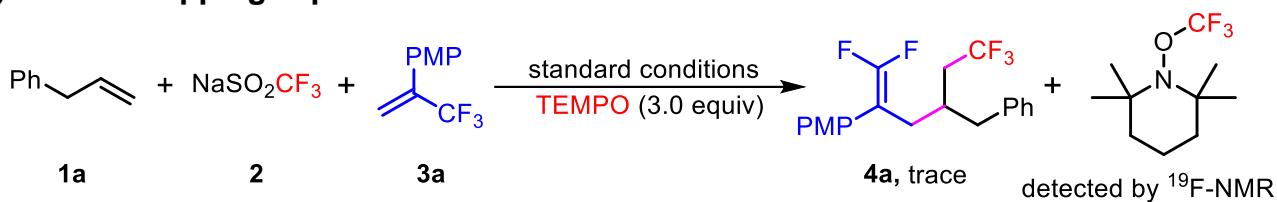
HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₃H₂₅F₄OBrNa 495.0917; found 495.0925.



According to the literature,¹¹ the 25 mL Schlenk tube was added difluoroalkene **5f** (41.8 mg, 0.10 mmol, 1.0 equiv) and toluene (1 mL) under argon. The Red-Al (80 ul, 3.5 M in toluene, 0.28 mol, 2.8 equiv) was added at 0 °C and the reaction was stirred for 2 h at 0 °C. Then the Red-Al (80 ul, 3.5 M in toluene, 0.28 mol, 2.8 equiv) was added and the mixture was stirred at 80 °C for another 24 h. The solution was cooled to room temperature and the crude product was purified by flash column chromatography on silica gel (PE) to afford the corresponding product **5fd** (32 mg, 80% yield).

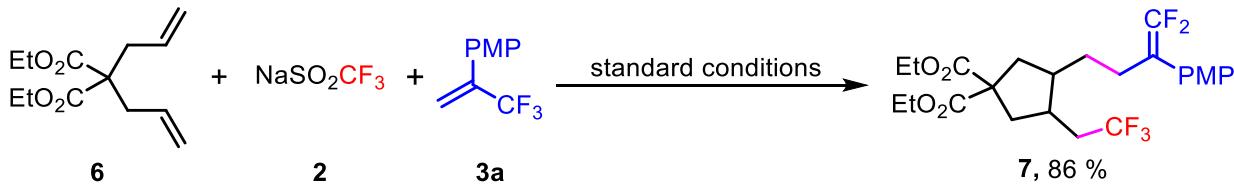
6: Control Experiment:

A) Radical trapping experiment:



To an oven-dried 4 mL reaction vial equipped with a stir bar was added MesAcrMe⁺ (0.8 mg, 0.002 mmol, 2 mol%), NaSO₂CF₃ (46.8 mg, 0.30 mmol, 3.0 equiv), K₃PO₄ (42.4 mg, 0.20 mmol, 2.0 equiv), and TEMPO (46.8 mg, 0.30 mmol, 3.0 equiv). The vial was then charged with allylbenzene **1a** (35.5 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv) in anhyd MeCN (1 mL) via a syringe. The cap was sealed with Parafilm®, and the solution was irradiated with a 30 W blue LED light at room temperature for 24 hours. The temperature of the reaction was maintained at approximately 27 °C via a fan. The solution was stirred vigorously while being irradiated. After the reaction, the mixture was then filtered through a pad of Celite and concentrated under reduced pressure. The residue was then subjected to GC-MS and ^{19}F NMR analysis.

B) Radical clock experiment:



To an oven-dried 4 mL reaction vial equipped with a stir bar was added MesAcrMe⁺ (0.8 mg, 0.002 mmol, 2 mol%), NaSO₂CF₃ (46.8 mg, 0.30 mmol, 3.0 equiv), and K₃PO₄ (42.4 mg, 0.20 mmol, 2.0 equiv). The vial was then charged with diethyl 2,2-diallylmalonate **6** (72.0 mg, 0.30 mmol, 3.0 equiv) and 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene **3a** (20.2 mg, 0.10 mmol, 1.0 equiv) in anhyd MeCN (1 mL) via a syringe. The cap was sealed with Parafilm®, and the solution was irradiated with a 30 W blue LED light at room temperature for 24 hours. The temperature of the reaction was maintained at approximately 27 °C via a fan. The solution was stirred vigorously while being irradiated. Once judged to be complete, the solution was transferred to a separatory funnel and diluted with deionized H₂O (20 mL) and Et₂O (20 mL). The layers were separated, and the aq layer was extracted with Et₂O (3 X 30 mL). The combined organic layers were washed with deionized H₂O (2 X 20 mL) followed by brine (20 mL). The combined organic layers were dried (Na₂SO₄), and the solvent was removed *in vacuo* by rotary evaporation. Further purification was accomplished by SiO₂ column

chromatography (gradient Hexane/EtOAc) to give the desired product **7** (86% yield, 42.3 mg, dr = 11:1) as a colorless oil.

TLC R_f = 0.35 (Hexane/EtOAc = 20:1, v/v).

^1H NMR (600 MHz, CDCl_3) δ 7.21 (d, J = 8.7 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 4.49 – 4.02 (m, 4H), 3.81 (s, 3H), 2.44 (dt, J = 9.1, 6.6 Hz, 2H), 2.40 – 2.20 (m, 3H), 2.21 – 1.81 (m, 4H), 1.41 – 1.28 (m, 1H), 1.26 – 0.94 (m, 8H).

^{13}C NMR (151 MHz, CDCl_3) δ 172.5, 172.4, 158.8, 156.4 – 150.5 (m), 129.3 (t, J = 3.2 Hz), 127.2 (d, J = 277.4 Hz), 125.3, 114.0, 91.4 (dd, J = 19.4, 16.1 Hz), 61.7, 58.5, 55.3, 41.4, 38.4, 37.9, 36.0, 33.2 (q, J = 27.5 Hz), 26.9, 26.0, 14.0 (d, J = 3.4 Hz).

^{19}F NMR (565 MHz, CDCl_3) δ -64.32 (t, J = 11.8 Hz), -92.21 (d, J = 7.8 Hz).

HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{24}\text{H}_{29}\text{F}_5\text{O}_5\text{Na}$ 515.1827; found 515.1839.

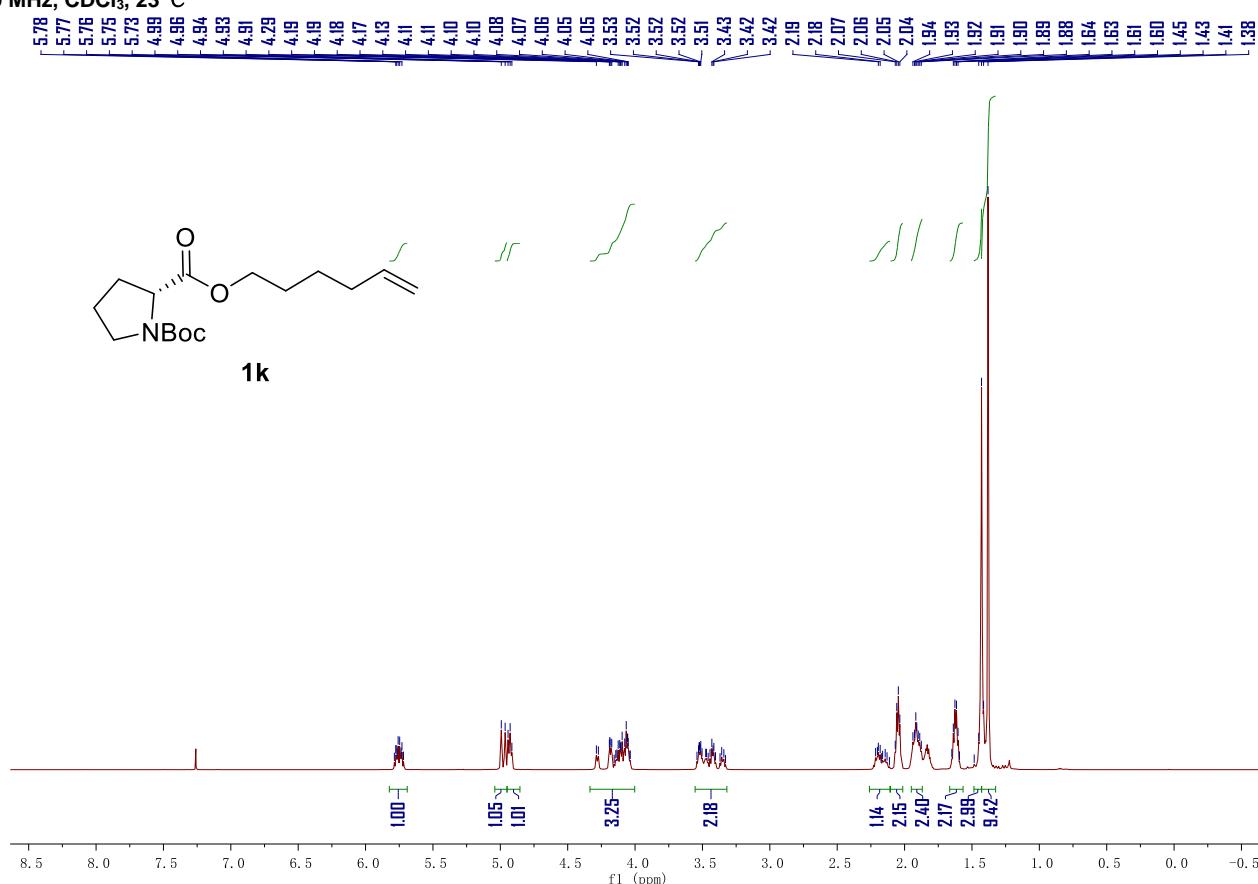
7. References:

- (1) Braun, M.-G.; Doyle, A. G. Palladium-Catalyzed Allylic C–H Fluorination. *J. Am. Chem. Soc.* **2013**, *135*, 12990–12993.
- (2) Lu, Z.-C.; Hennis, O.; Gentry, J.; Xu, B.; Hammond, G. B. Base-Promoted Radical Azofluoromethylation of Unactivated Alkenes. *Org. Lett.* **2020**, *22*, 4383–4388.
- (3) Xu, C.; Huang, W.-Q.; Zhang, R.-Z.; Gao, C.; Li, Y.-X.; Wang, M. Trifluoromethylations of Alkenes Using PhICF₃Cl as Bifunctional Reagent. *J. Org. Chem.* **2019**, *84*, 14209–14216.
- (4) Cheng, B.; Liu, W.-B.; Lu, Z. Iron-Catalyzed Highly Enantioselective Hydrosilylation of Unactivated Terminal Alkenes. *J. Am. Chem. Soc.* **2018**, *140*, 5014–5017.
- (5) Shi, J.; Guo, L.-Y.; Hu, Q.-P.; Liu, Y.-T.; Li, Q.; Pan, F. Photoredox-Catalyzed Difunctionalization of Unactivated Olefins for Synthesizing Lactam-Substituted *gem*-Difluoroalkenes. *Org. Lett.* **2021**, *23*, 8822–8827.
- (6) Anand, D.; Sun, Z.-C.; Zhou, L. Visible-Light-Mediated β -C–H *gem*-Difluoroallylation of Aldehydes and Cyclic Ketones through C–F Bond Cleavage of 1-Trifluoromethyl Alkenes. *Org. Lett.* **2020**, *22*, 2371–2375.
- (7) Guo, Y.-Q.; Wang, R.; Song, H.; Liu, Y.; Wang, Q. Visible-Light-Induced Deoxygenation/Defluorination Protocol for Synthesis of γ,γ -Difluoroallylic Ketones. *Org. Lett.* **2020**, *22*, 709–713.
- (8) Lan, Y.; Yang, F.-Y.; Wang, C. Synthesis of *gem*-Difluoroalkenes via Nickel-Catalyzed Allylic Defluorinative Reductive Cross-Coupling. *ACS Catal.* **2018**, *8*, 9245–9251.
- (9) Jiang, L.-F.; Ren, B.-T.; Li, B.; Zhang, G.-Y.; Peng, Y.; Guan, Z.-Y.; Deng, Q.-H. Nucleophilic Substitution of *gem*-Difluoroalkenes with TMSNu Promoted by Catalytic Amounts of Cs₂CO₃. *J. Org. Chem.* **2019**, *84*, 6557–6564.
- (10) He, Y.-W.; Anand, D.; Sun, Z.-C.; Zhou, L. Visible-Light-Promoted Redox Neutral γ,γ Difluoroallylation of Cycloketone Oxime Ethers with Trifluoromethyl Alkenes via C–C and C–F Bond Cleavage. *Org. Lett.* **2019**, *21*, 3769–3773.
- (11) Poutrel, P.; Pannecoucke, X.; Jubault, P.; Poisson, T. Stereoselective Synthesis of Terminal Monofluoroalkenes from Trifluoromethylated Alkenes. *Org. Lett.* **2020**, *22*, 4858–4863.

8. NMR Spectra:

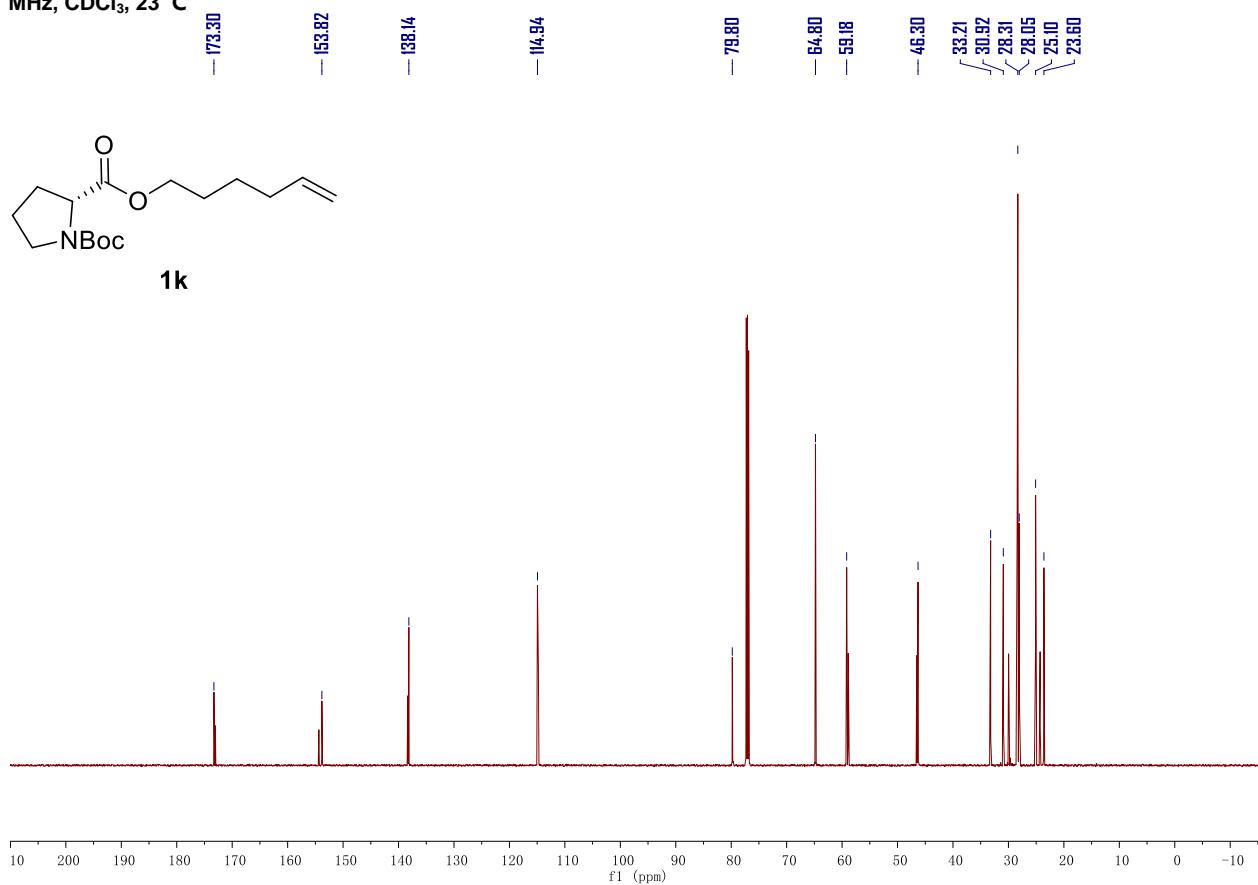
¹H NMR spectrum of 1-(*tert*-butyl) 2-(hex-5-en-1-yl) (*R*)-pyrrolidine-1,2-dicarboxylate (1k)

600 MHz, CDCl₃, 23 °C



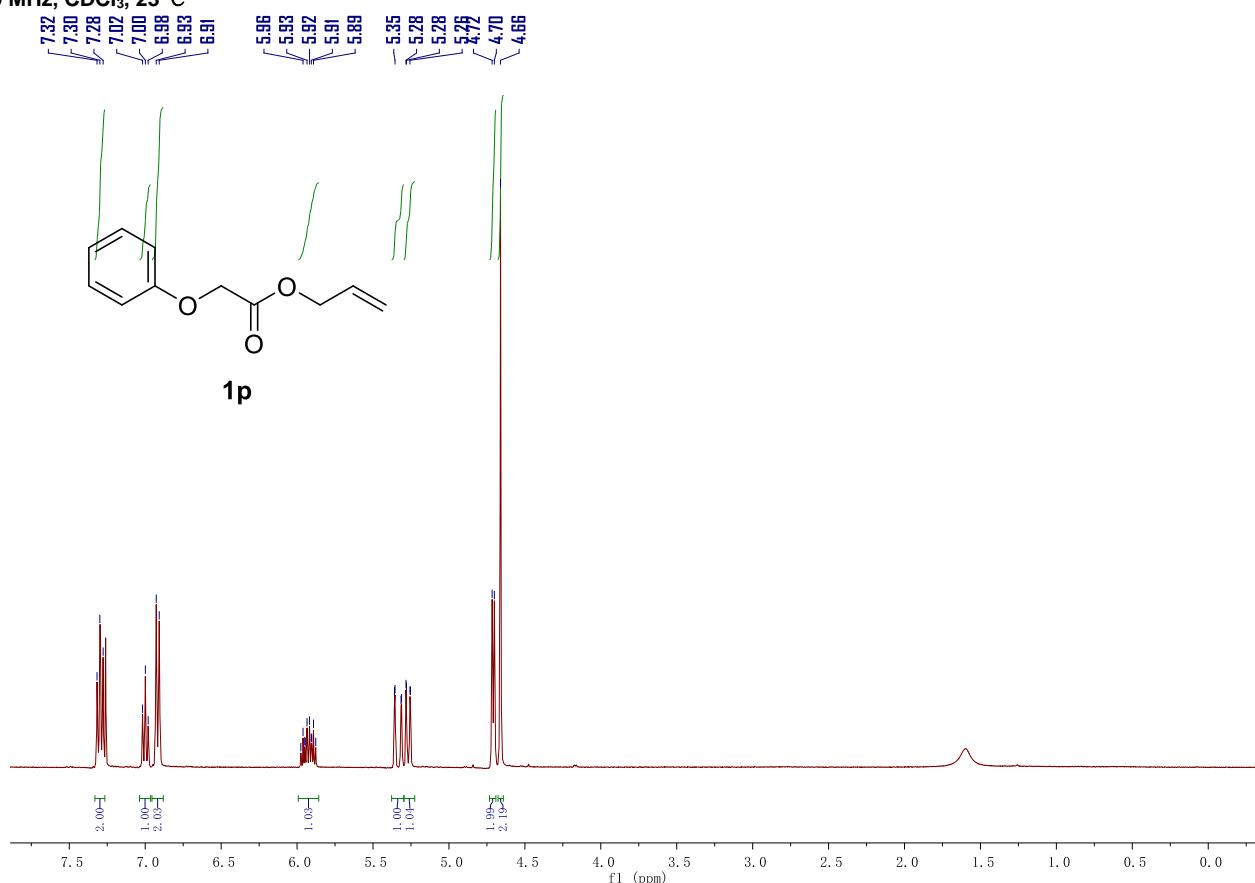
¹³C NMR spectrum of 1-(*tert*-butyl) 2-(hex-5-en-1-yl) (*R*)-pyrrolidine-1,2-dicarboxylate (1k)

151 MHz, CDCl₃, 23 °C



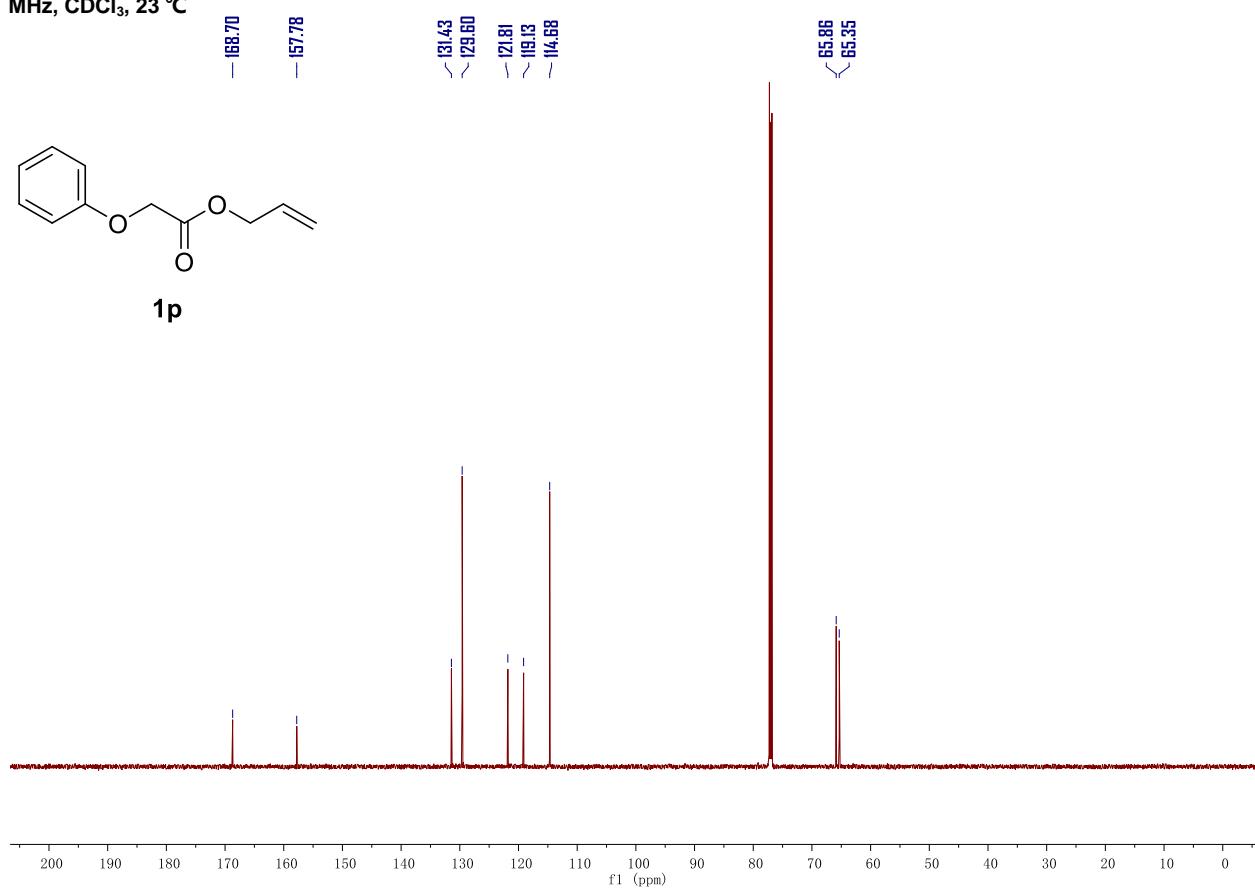
¹H NMR spectrum of allyl 2-phenoxyacetate (1p)

600 MHz, CDCl₃, 23 °C



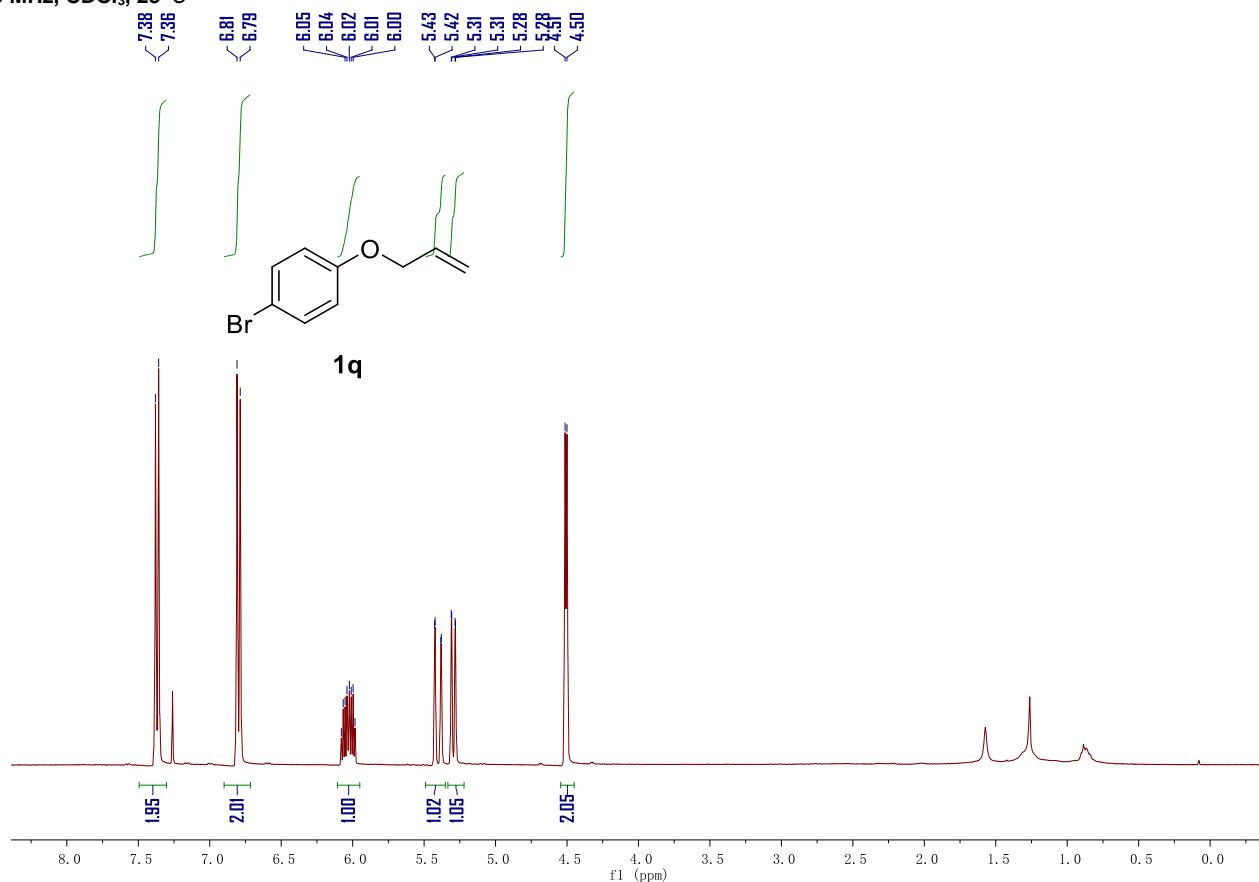
¹³C NMR spectrum of allyl 2-phenoxyacetate (1p)

151 MHz, CDCl₃, 23 °C



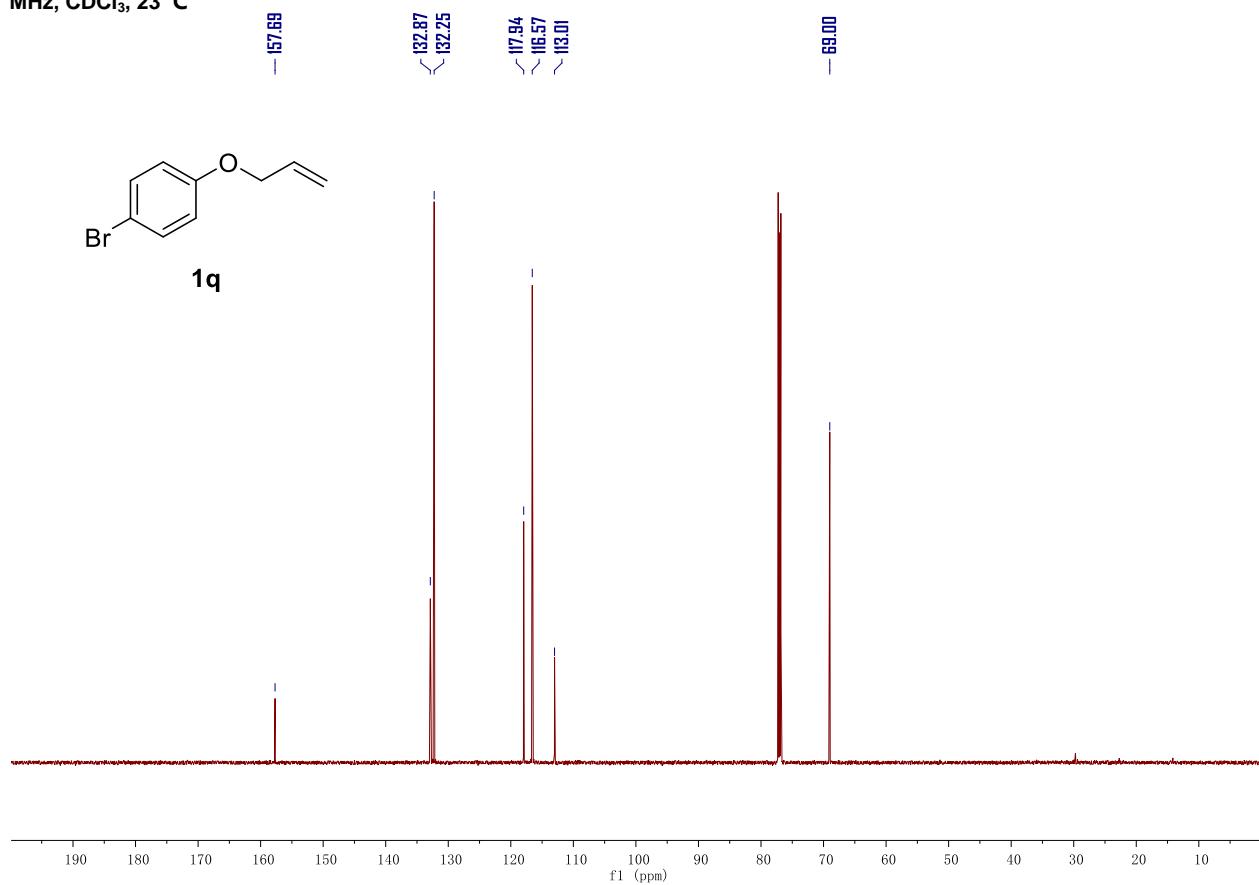
¹H NMR spectrum of 1-(allyloxy)-4-bromobenzene (1q)

600 MHz, CDCl₃, 23 °C



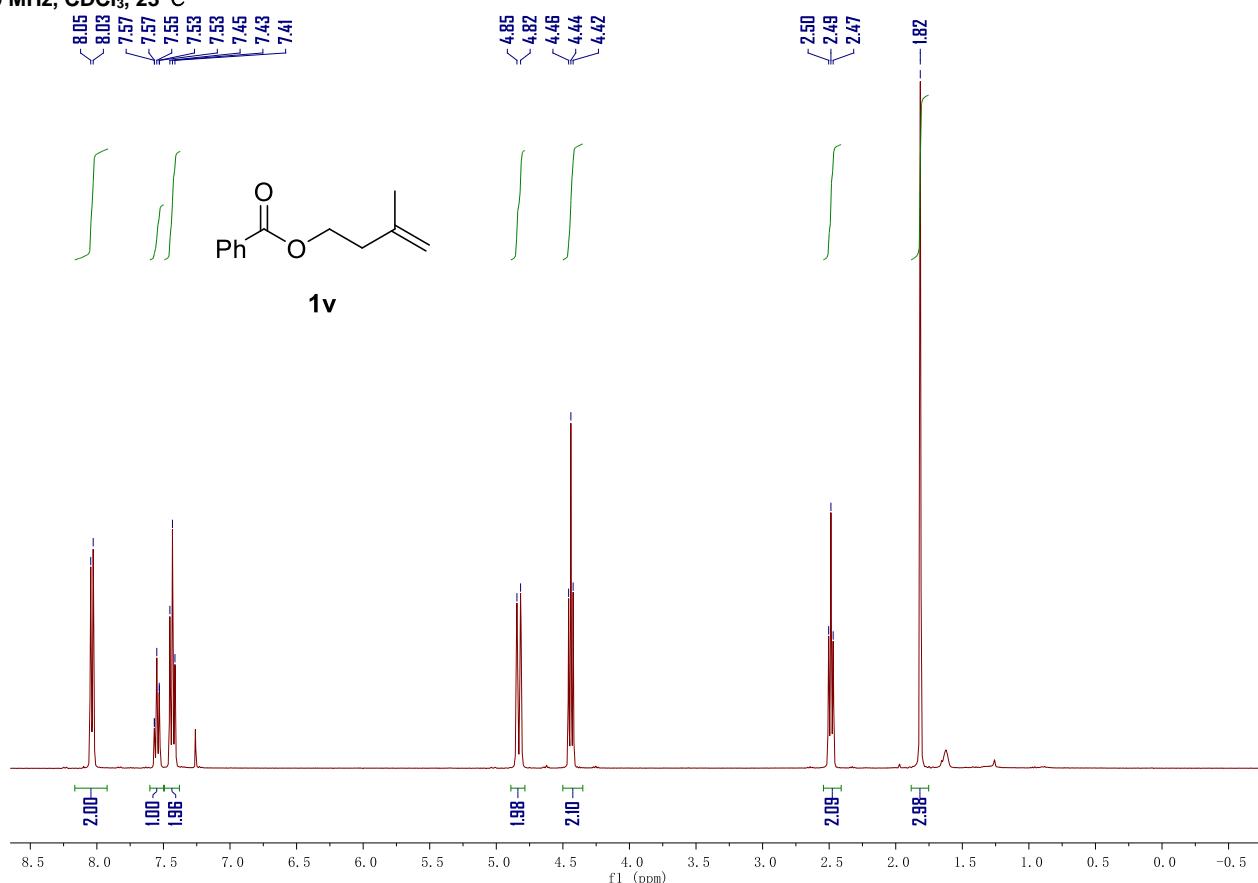
¹³C NMR spectrum of 1-(allyloxy)-4-bromobenzene (1q)

151 MHz, CDCl₃, 23 °C



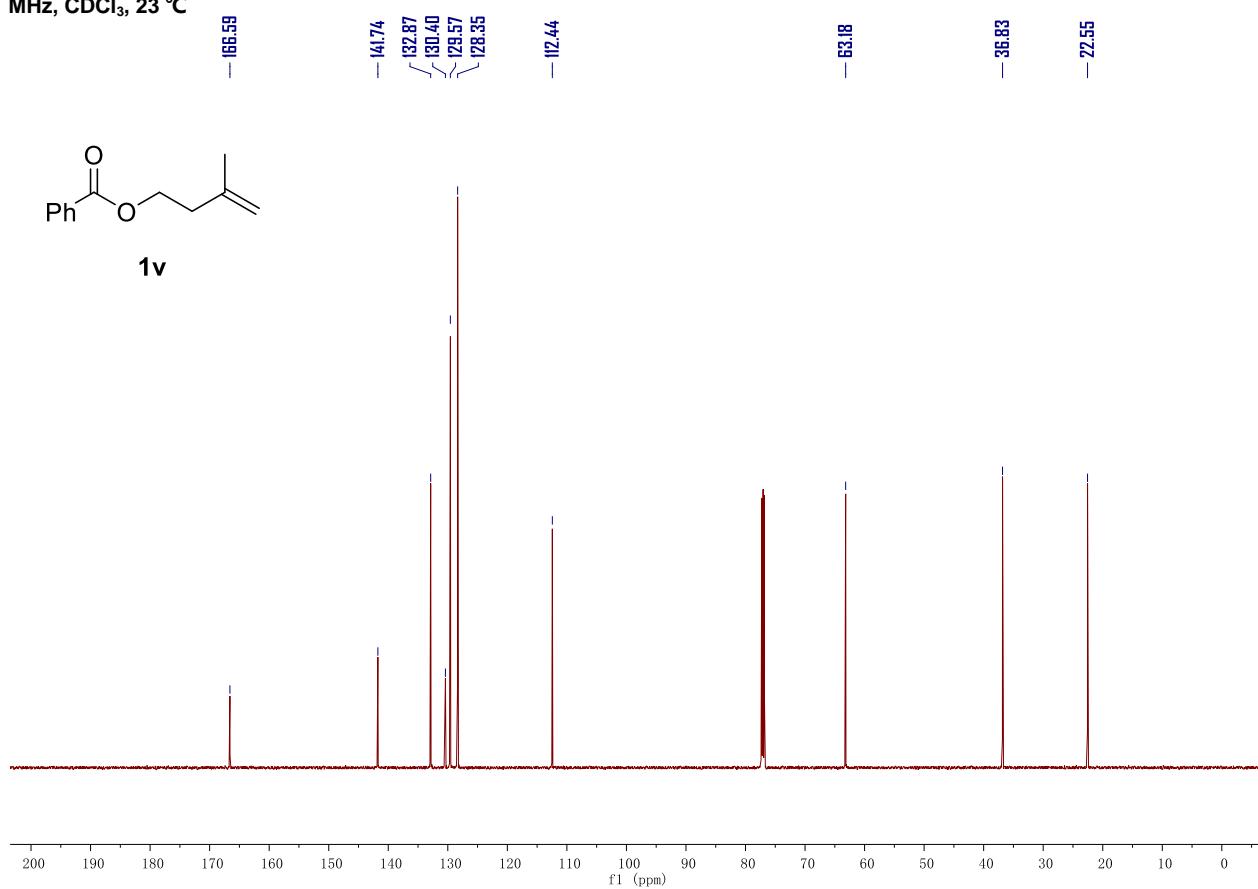
¹H NMR spectrum of 3-methylbut-3-en-1-yl benzoate (1v)

600 MHz, CDCl₃, 23 °C



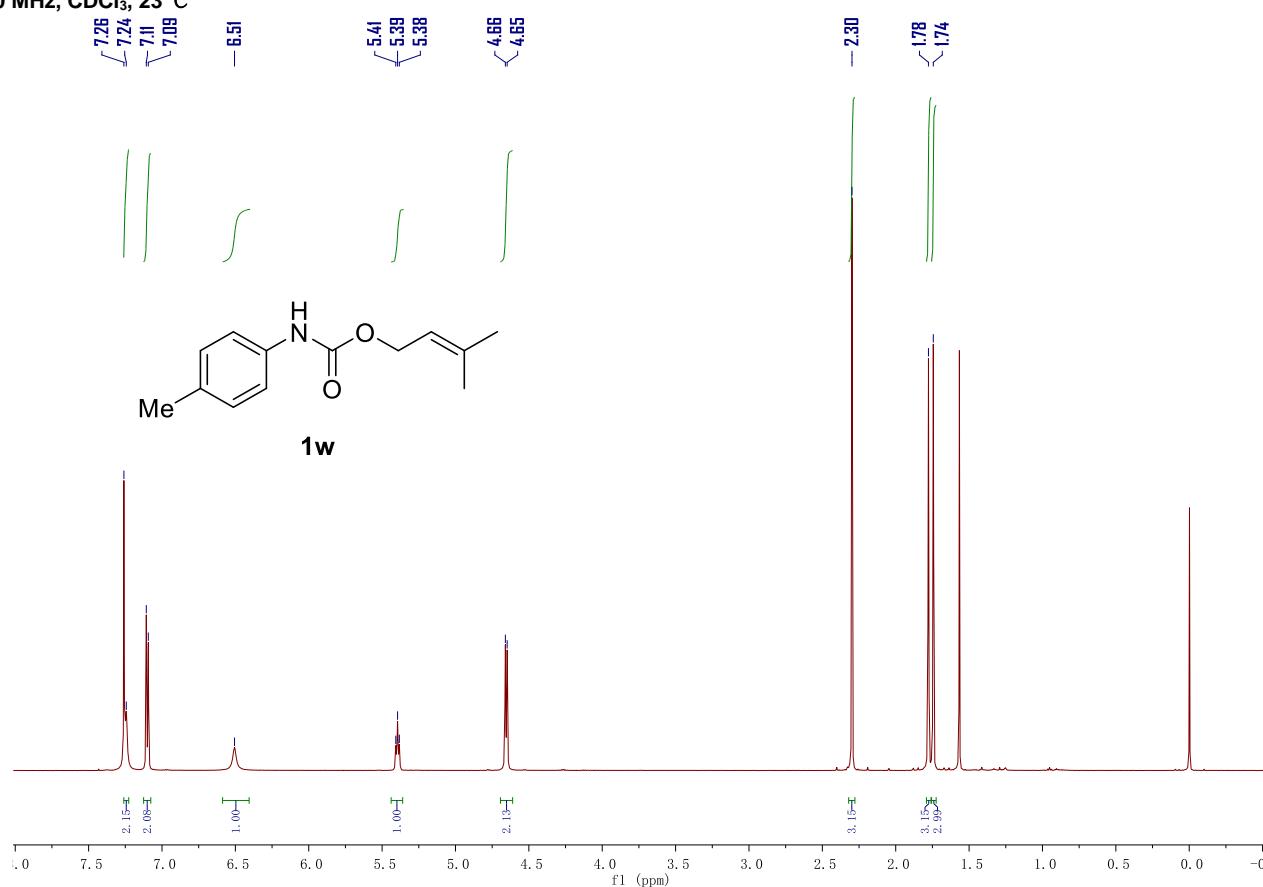
¹³C NMR spectrum of 3-methylbut-3-en-1-yl benzoate (1v)

151 MHz, CDCl₃, 23 °C



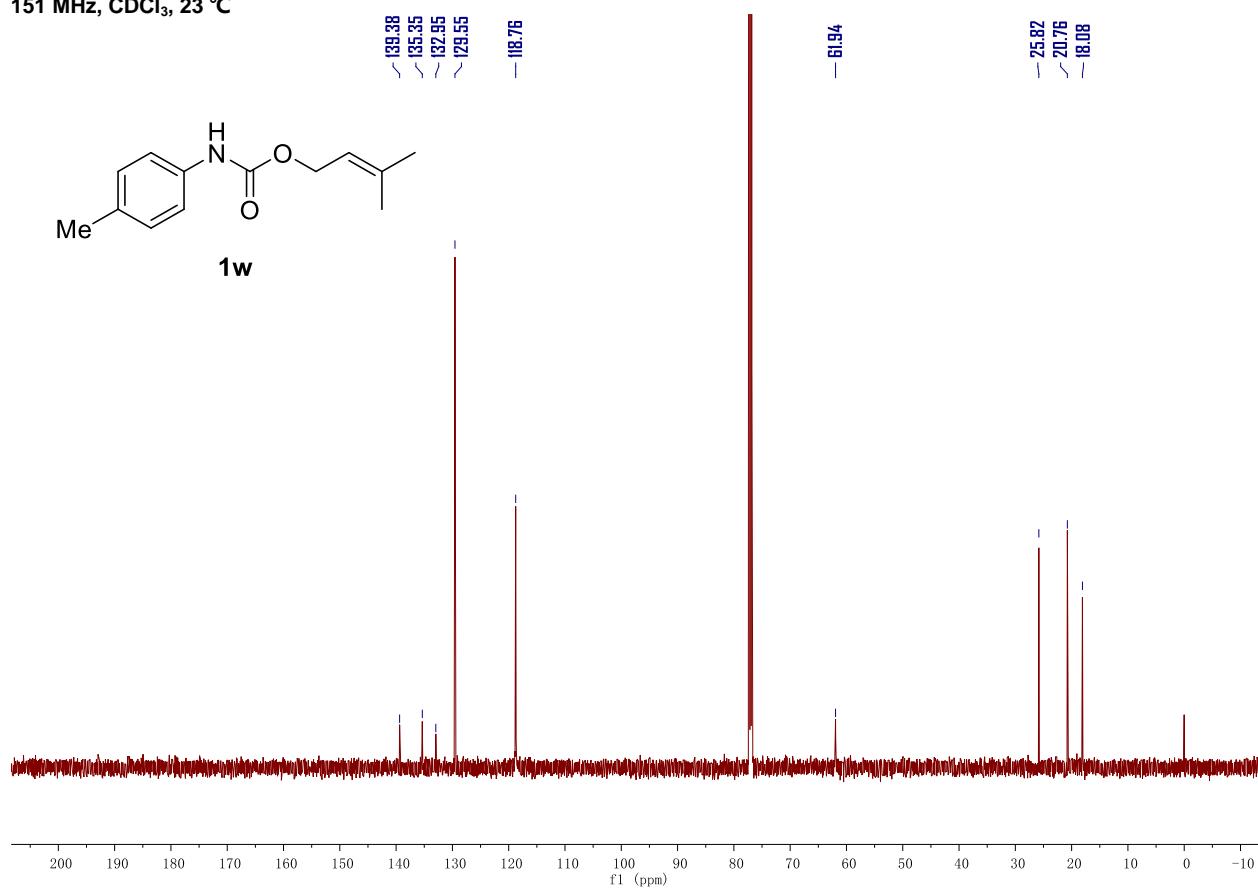
¹H NMR spectrum of 3-methylbut-2-en-1-yl p-tolylcarbamate (1w)

600 MHz, CDCl₃, 23 °C



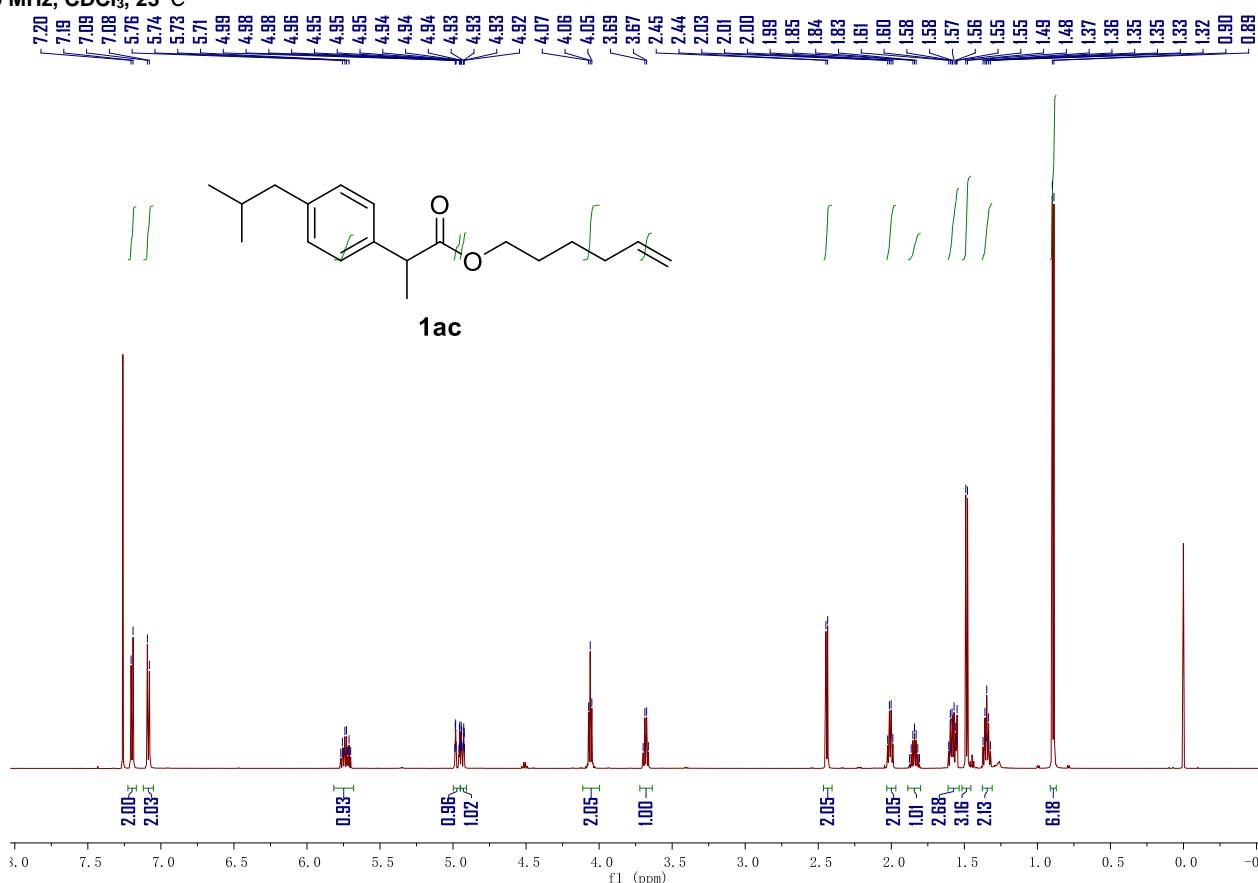
¹³C NMR spectrum of 3-methylbut-2-en-1-yl p-tolylcarbamate (1w)

151 MHz, CDCl₃, 23 °C



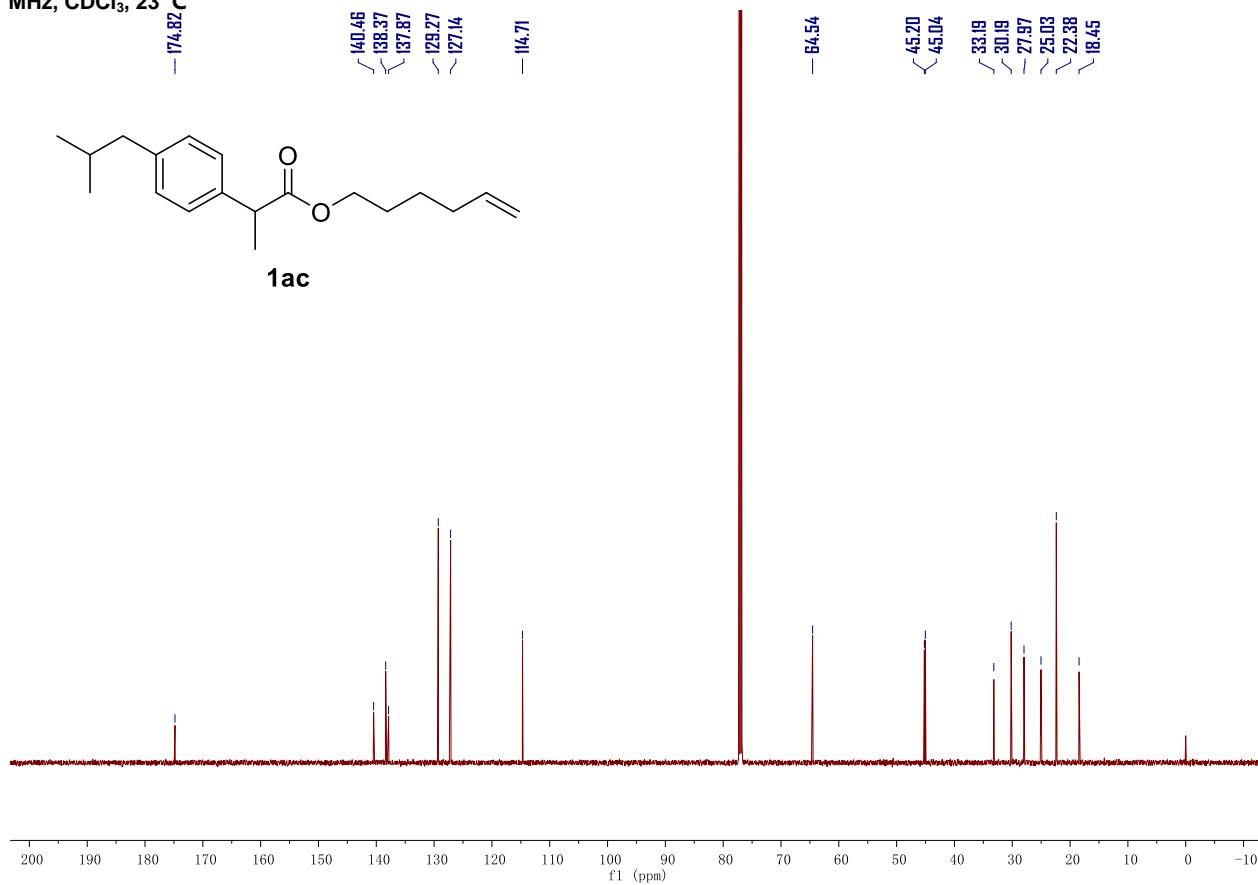
¹H NMR spectrum of hex-5-en-1-yl 2-(4-isobutylphenyl)propanoate (1ac)

600 MHz, CDCl₃, 23 °C



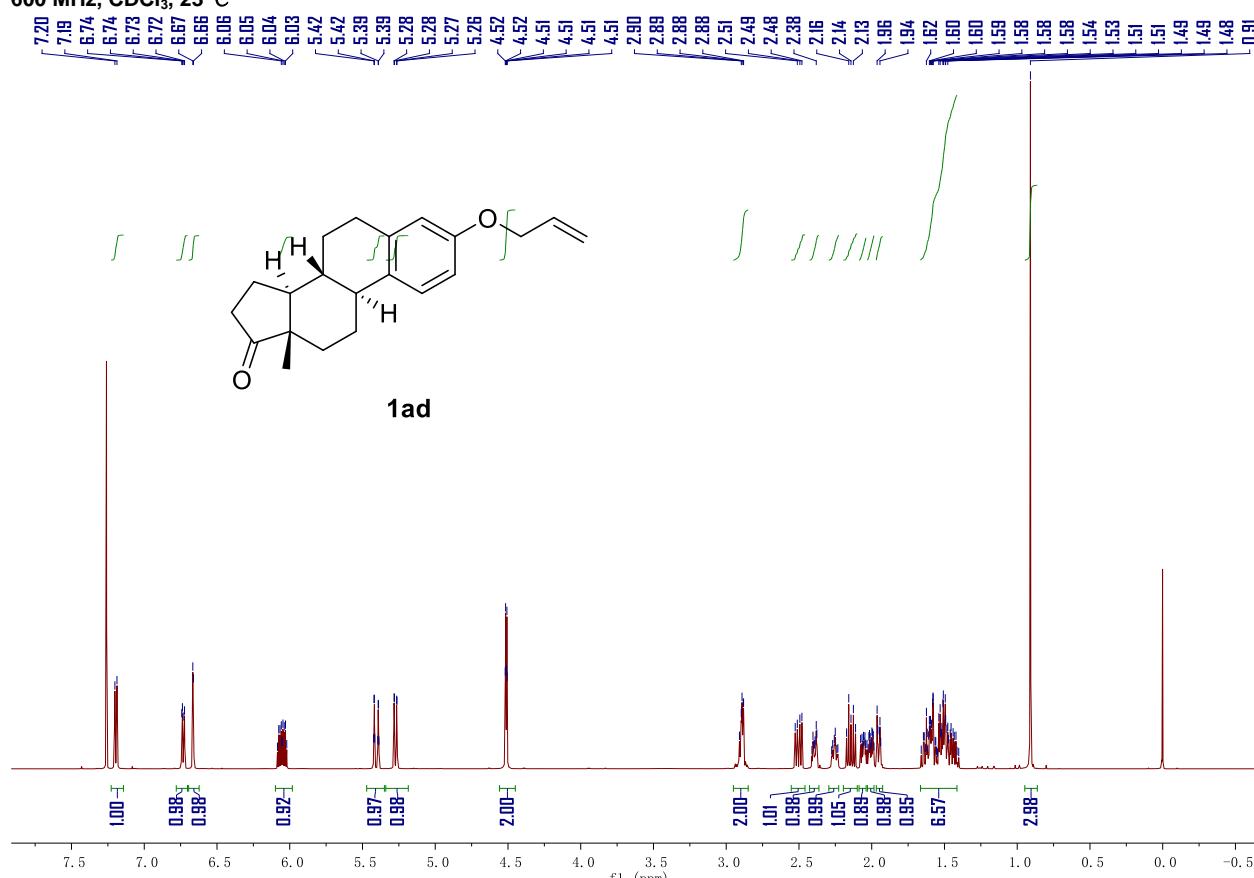
¹³C NMR spectrum of hex-5-en-1-yl 2-(4-isobutylphenyl)propanoate (1ac)

151 MHz, CDCl₃, 23 °C



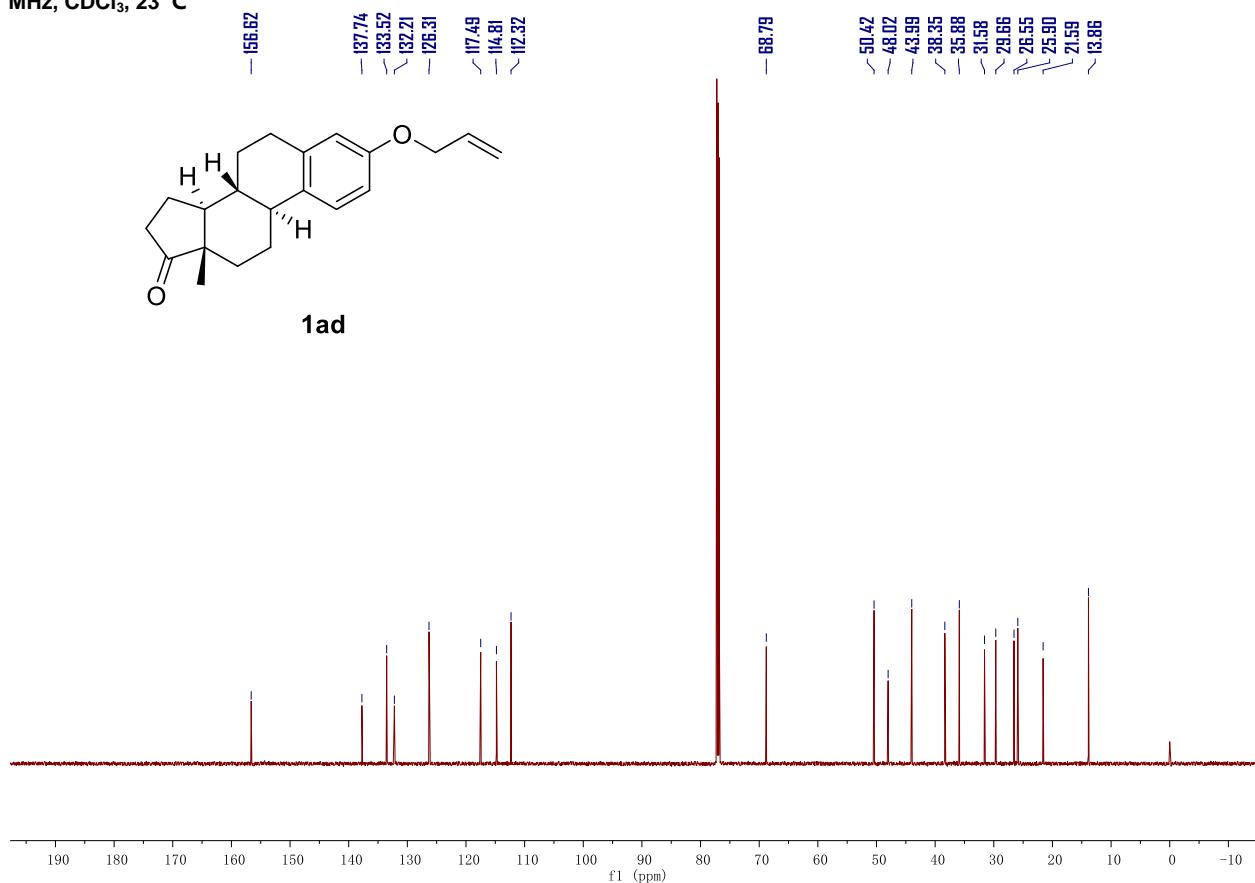
¹H NMR spectrum of 3-(allyloxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (1ad)

600 MHz, CDCl₃, 23 °C

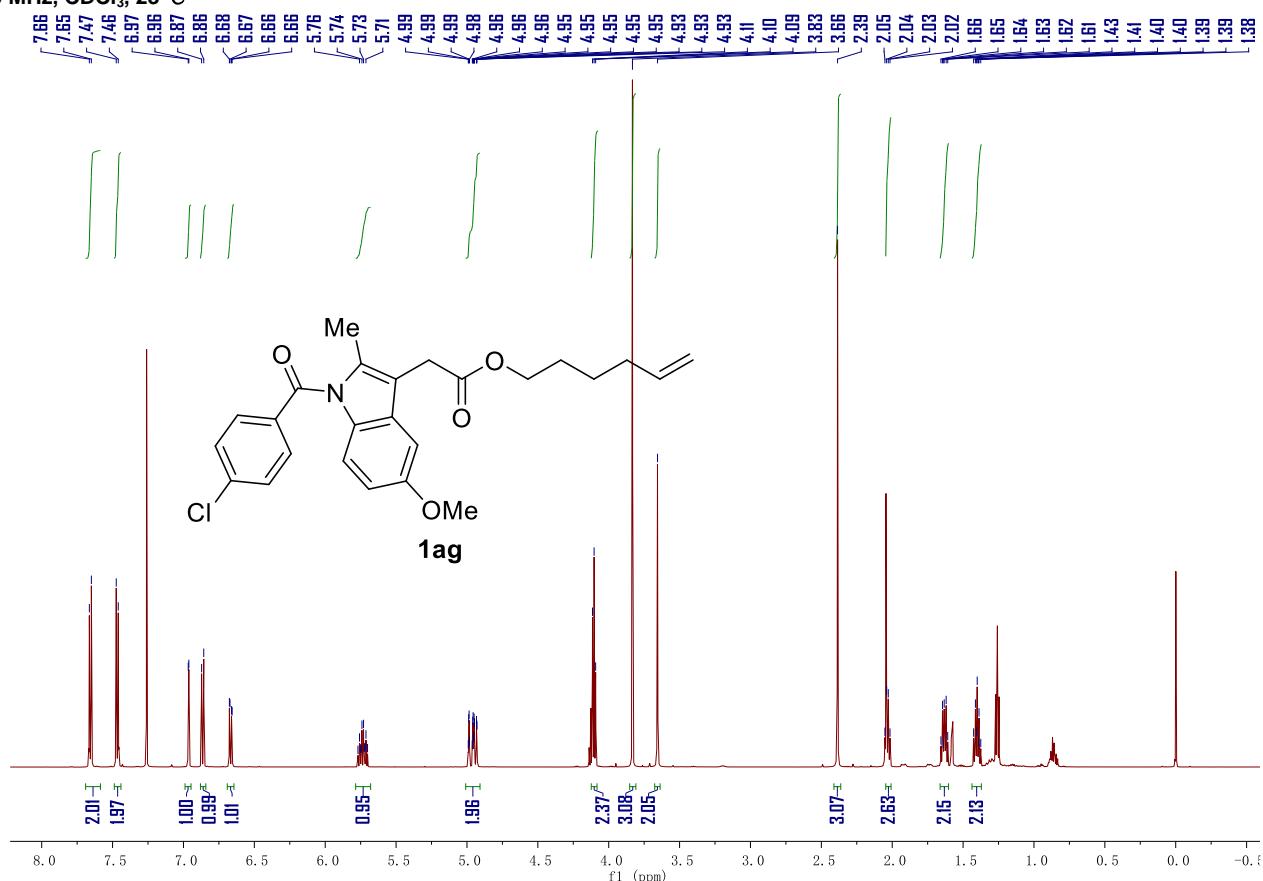


¹³C NMR spectrum of 3-(allyloxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (1ad)

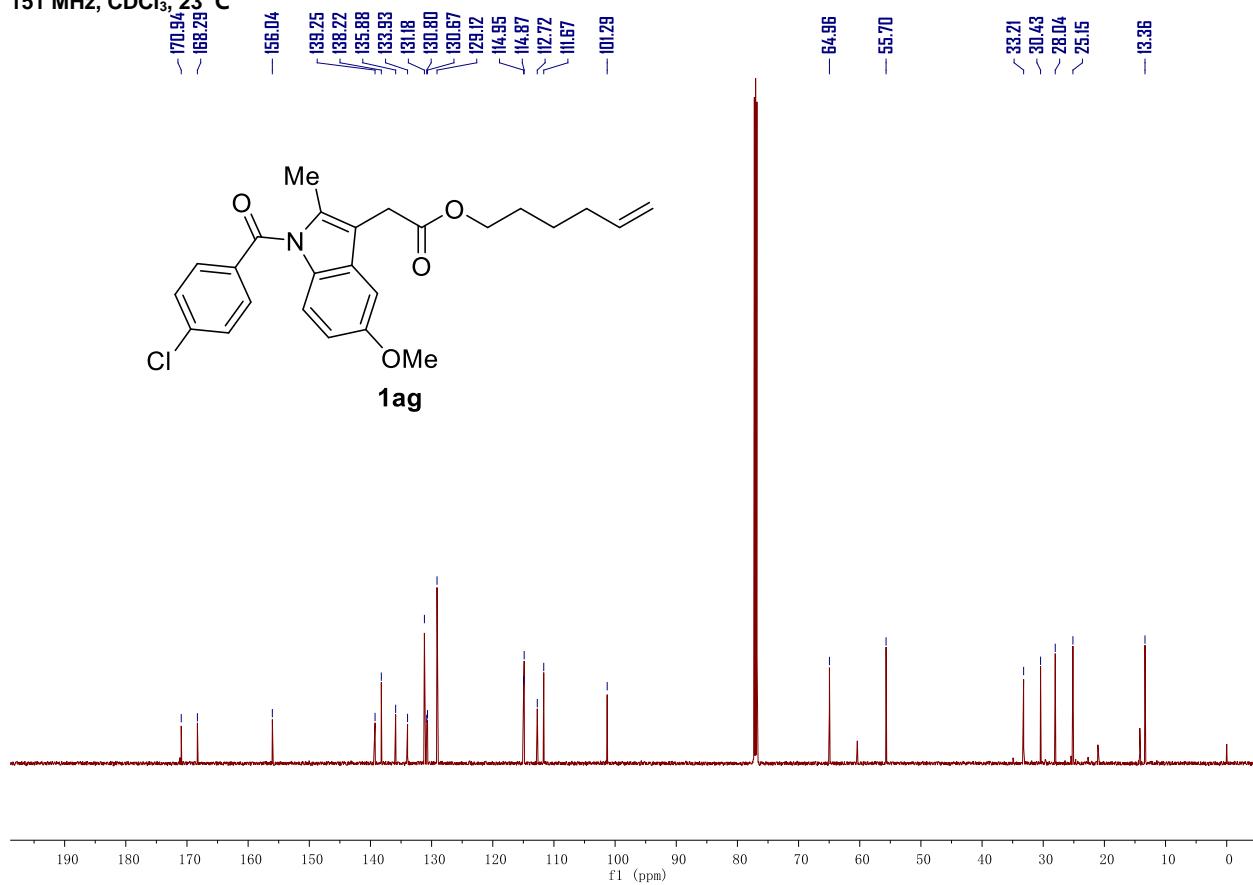
151 MHz, CDCl₃, 23 °C



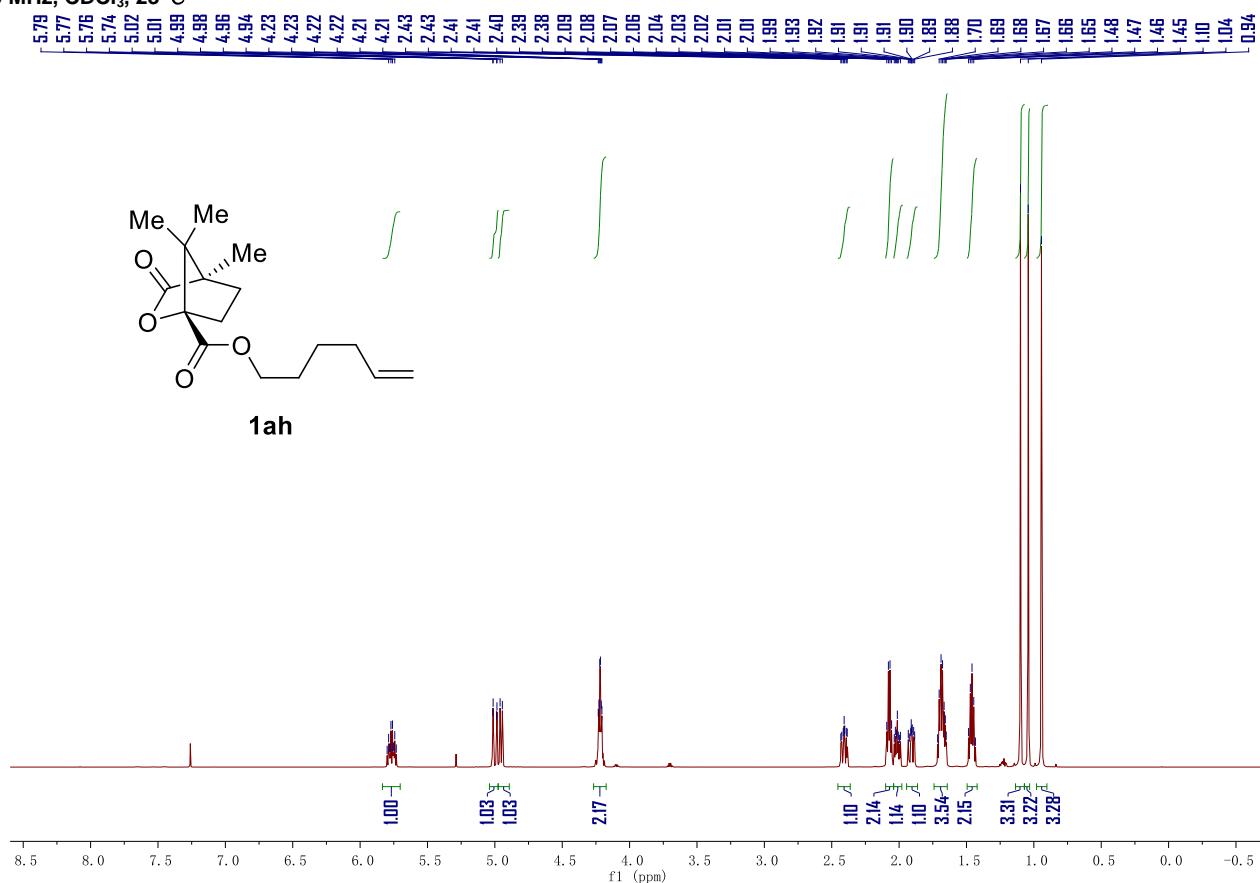
¹H NMR spectrum of hex-5-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (1ag)
600 MHz, CDCl₃, 23 °C



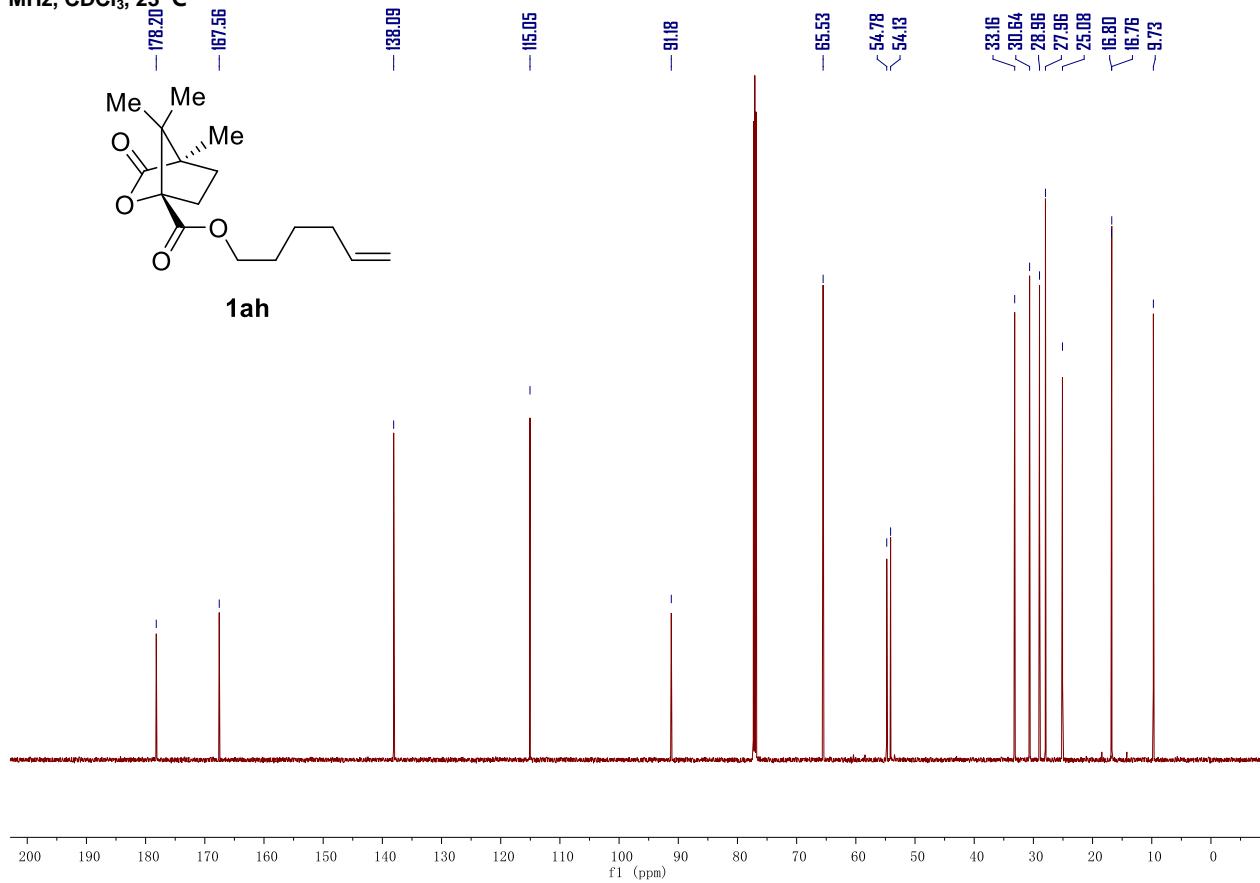
¹³C NMR spectrum of hex-5-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (1ag)
151 MHz, CDCl₃, 23 °C



¹H NMR spectrum of hex-5-en-1-yl-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (1ah)
 600 MHz, CDCl₃, 23 °C

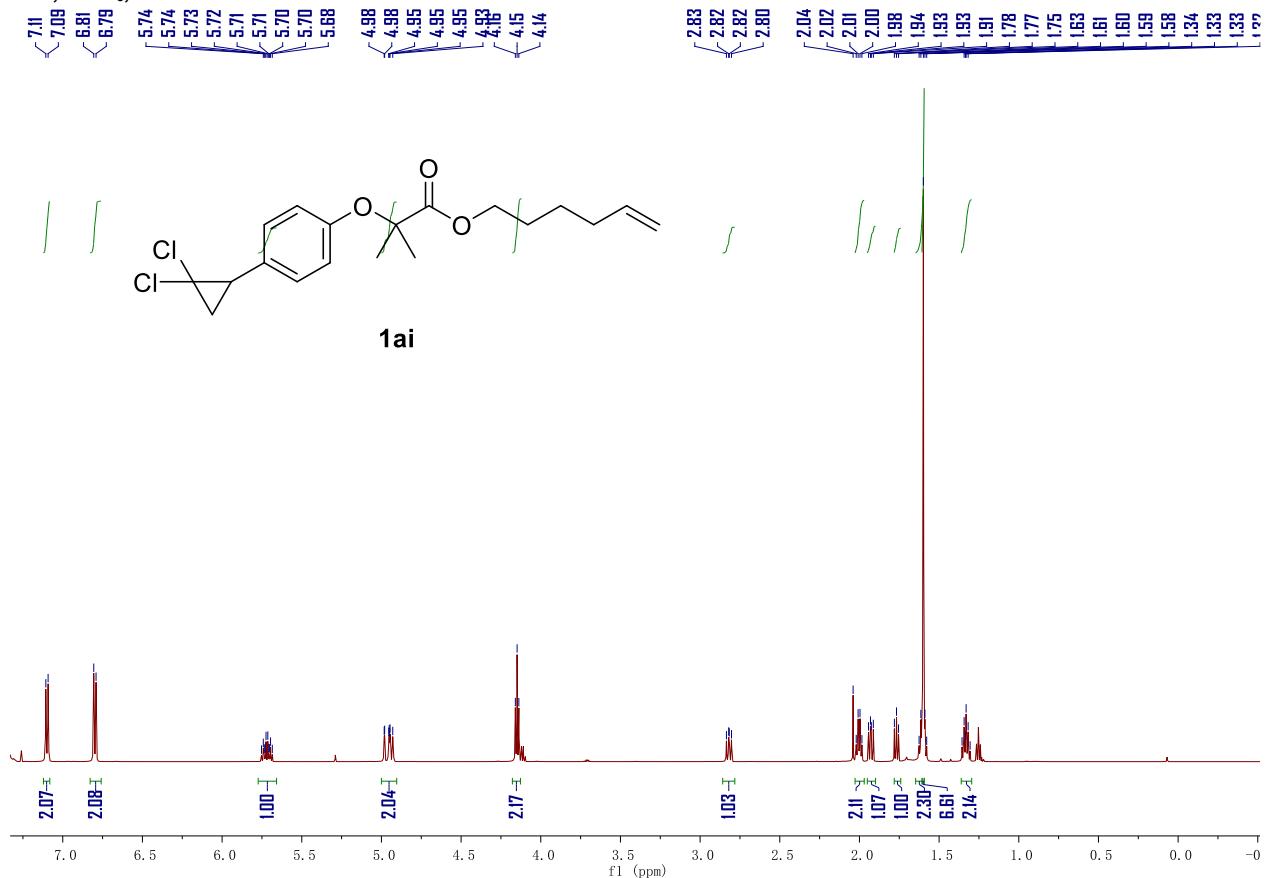


¹³C NMR spectrum of hex-5-en-1-yl-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (1ah)
 151 MHz, CDCl₃, 23 °C



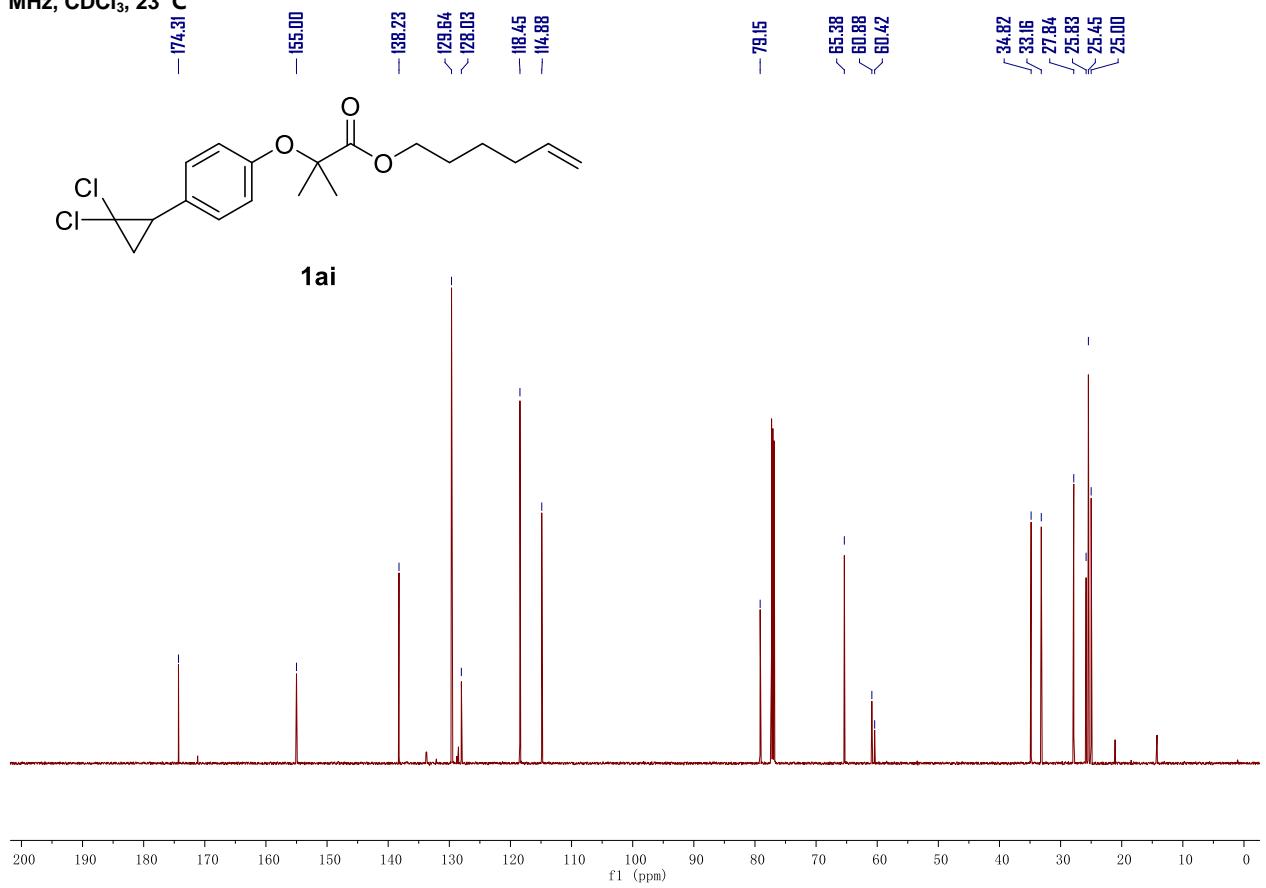
¹H NMR spectrum of hex-5-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (1ai)

600 MHz, CDCl₃, 23 °C



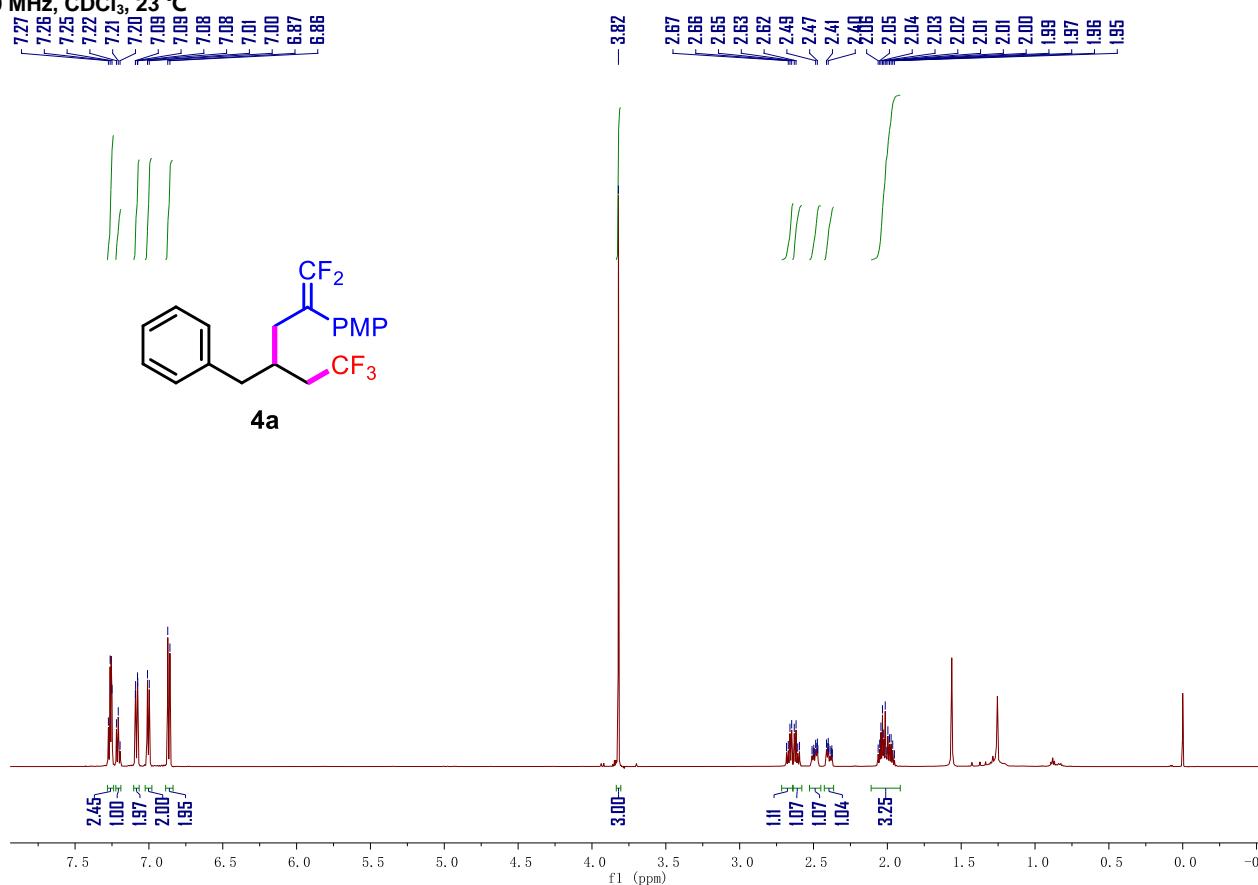
¹³C NMR spectrum of hex-5-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (1ai)

151 MHz, CDCl₃, 23 °C



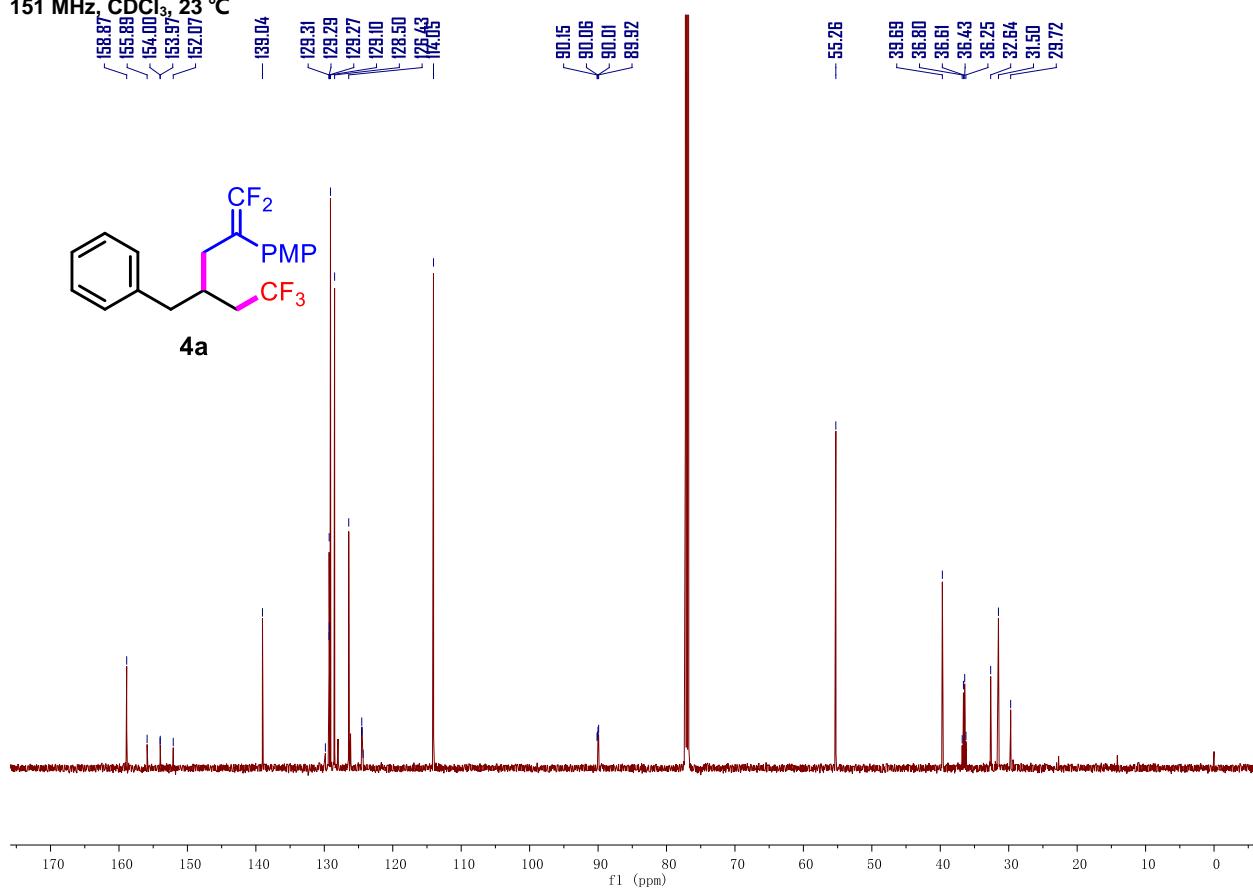
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4a)

600 MHz, CDCl₃, 23 °C

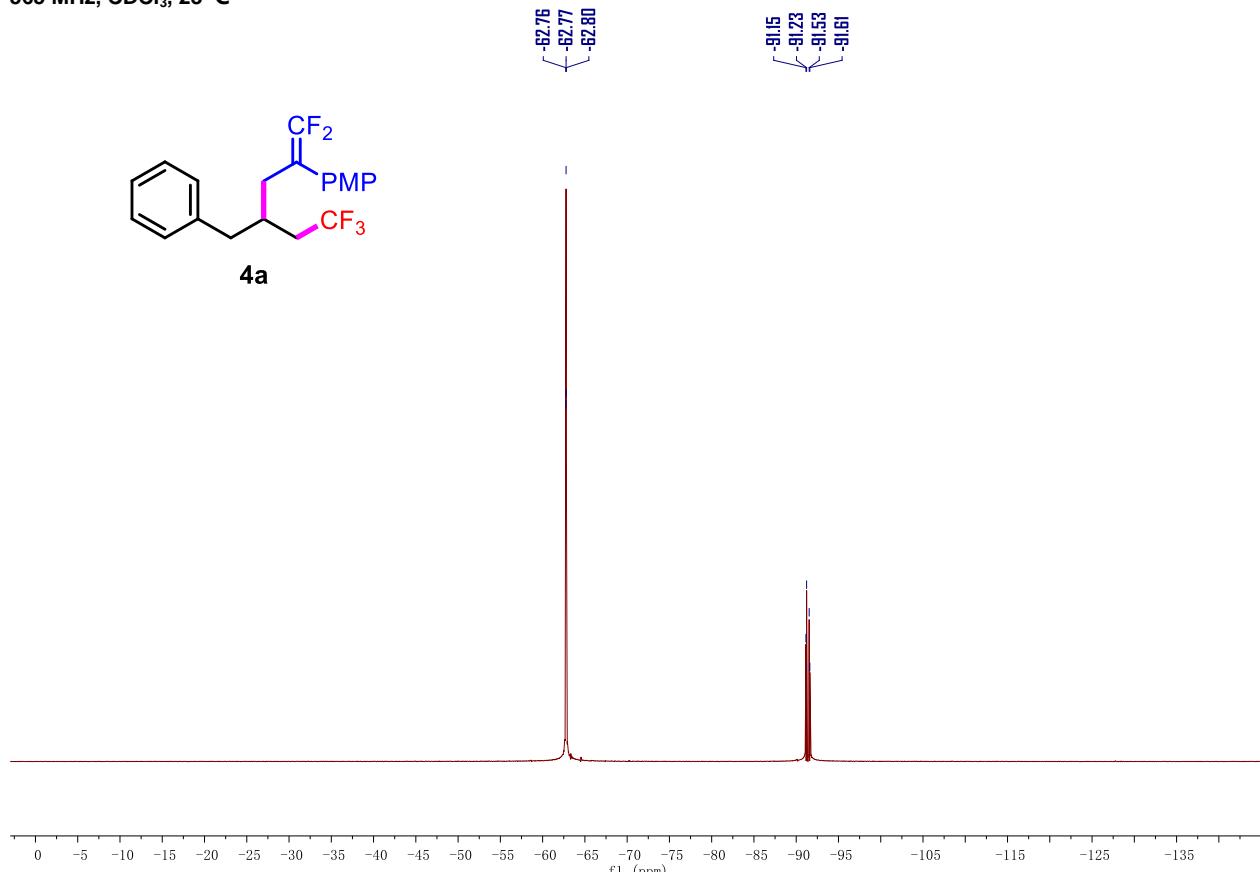


¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4a)

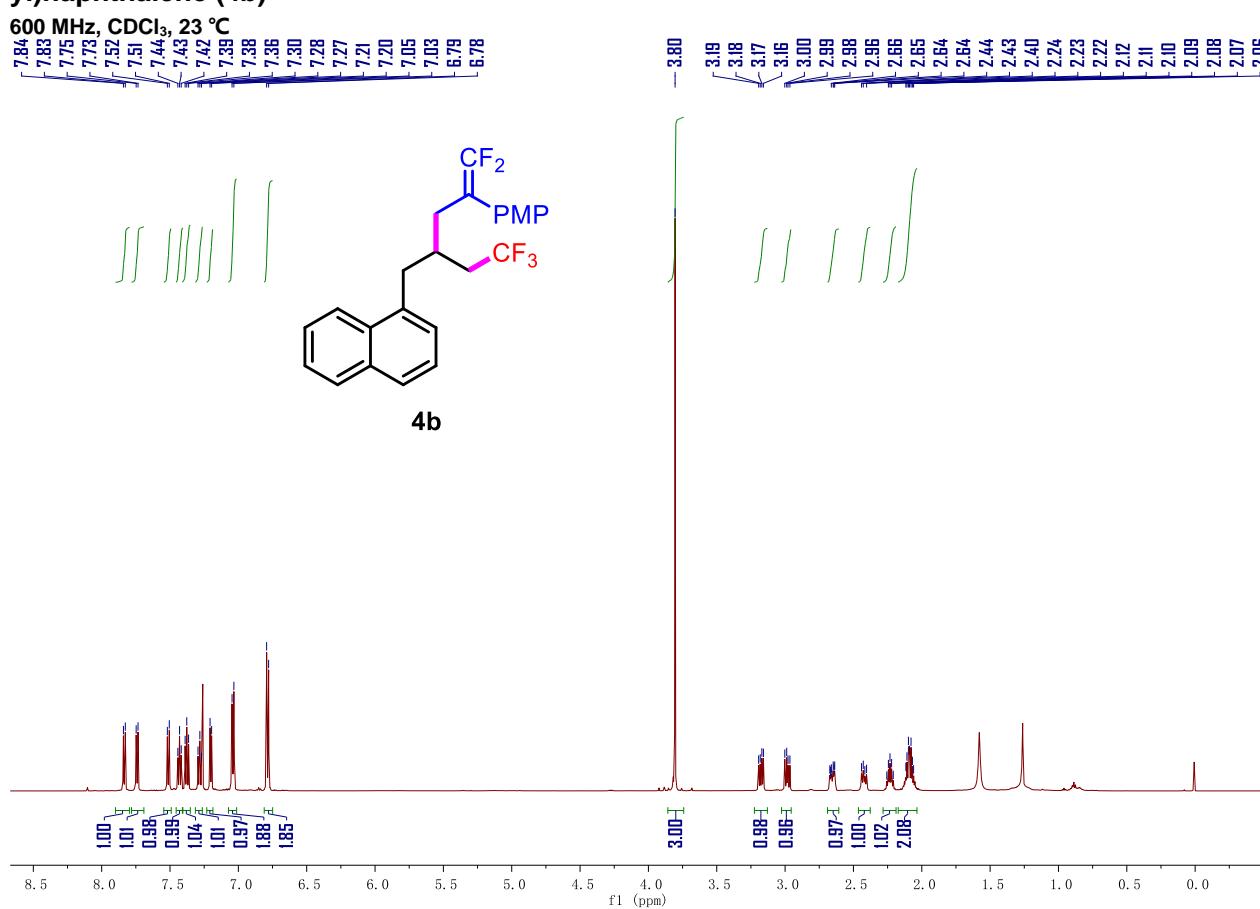
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4a)
 565 MHz, CDCl₃, 23 °C

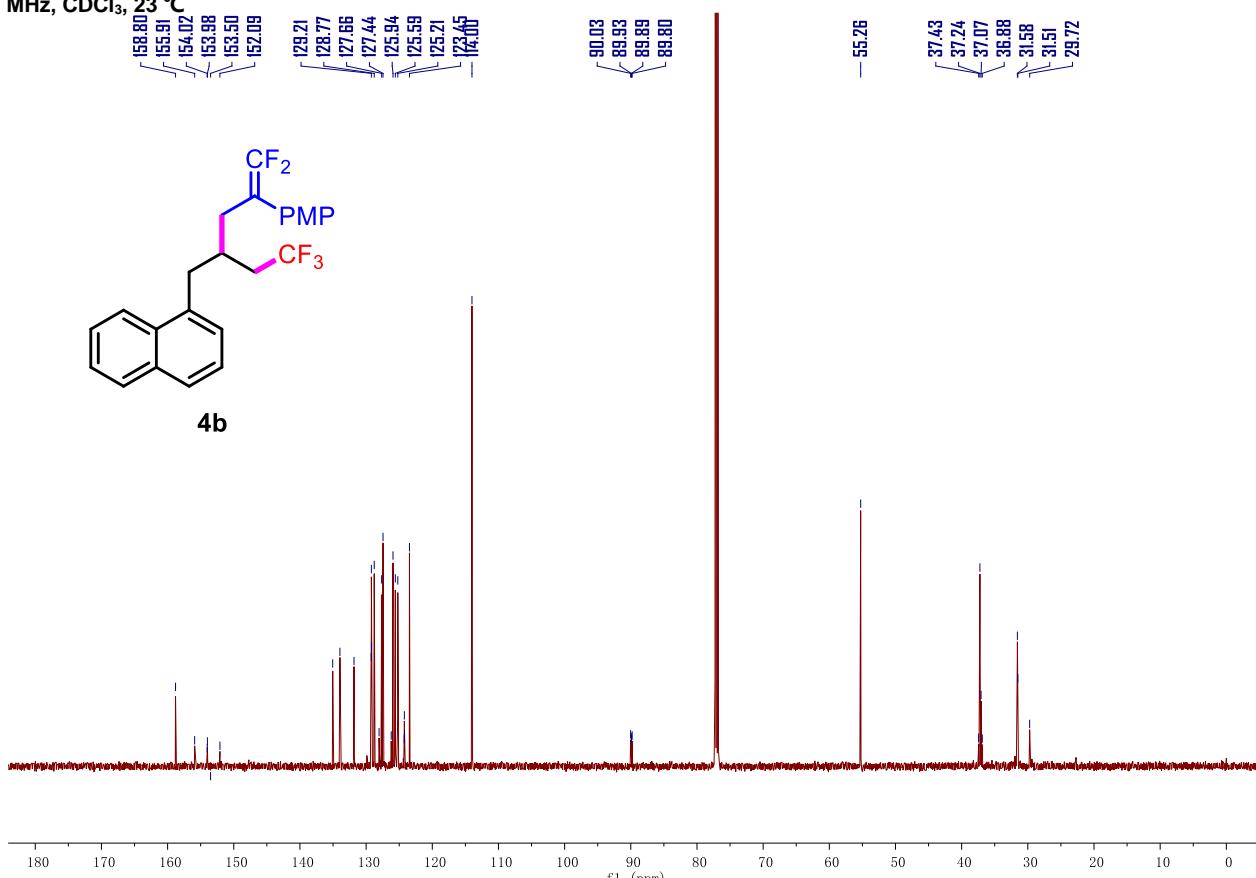


¹H NMR spectrum of 1-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)naphthalene (4b)



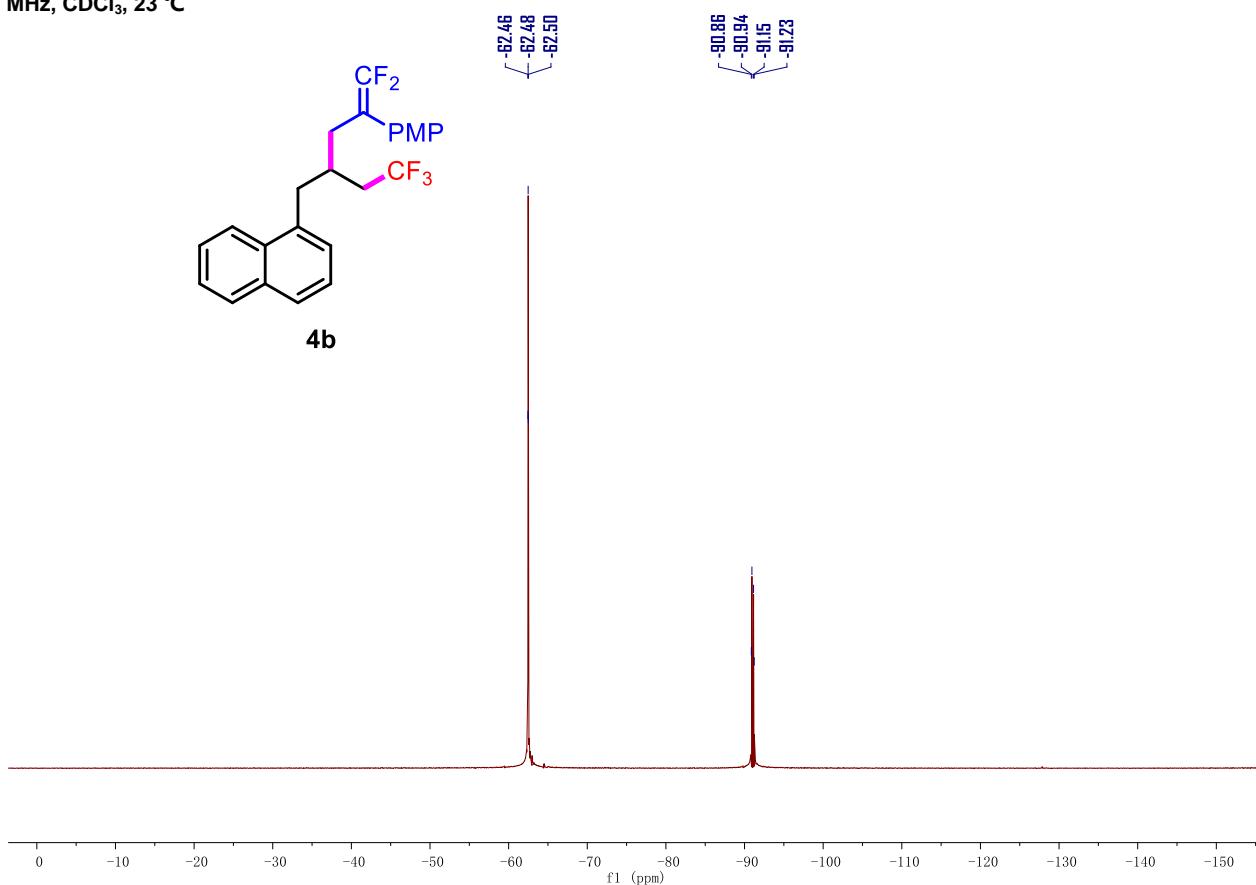
¹³C NMR spectrum of 1-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)naphthalene (4b)

151 MHz, CDCl₃, 23 °C



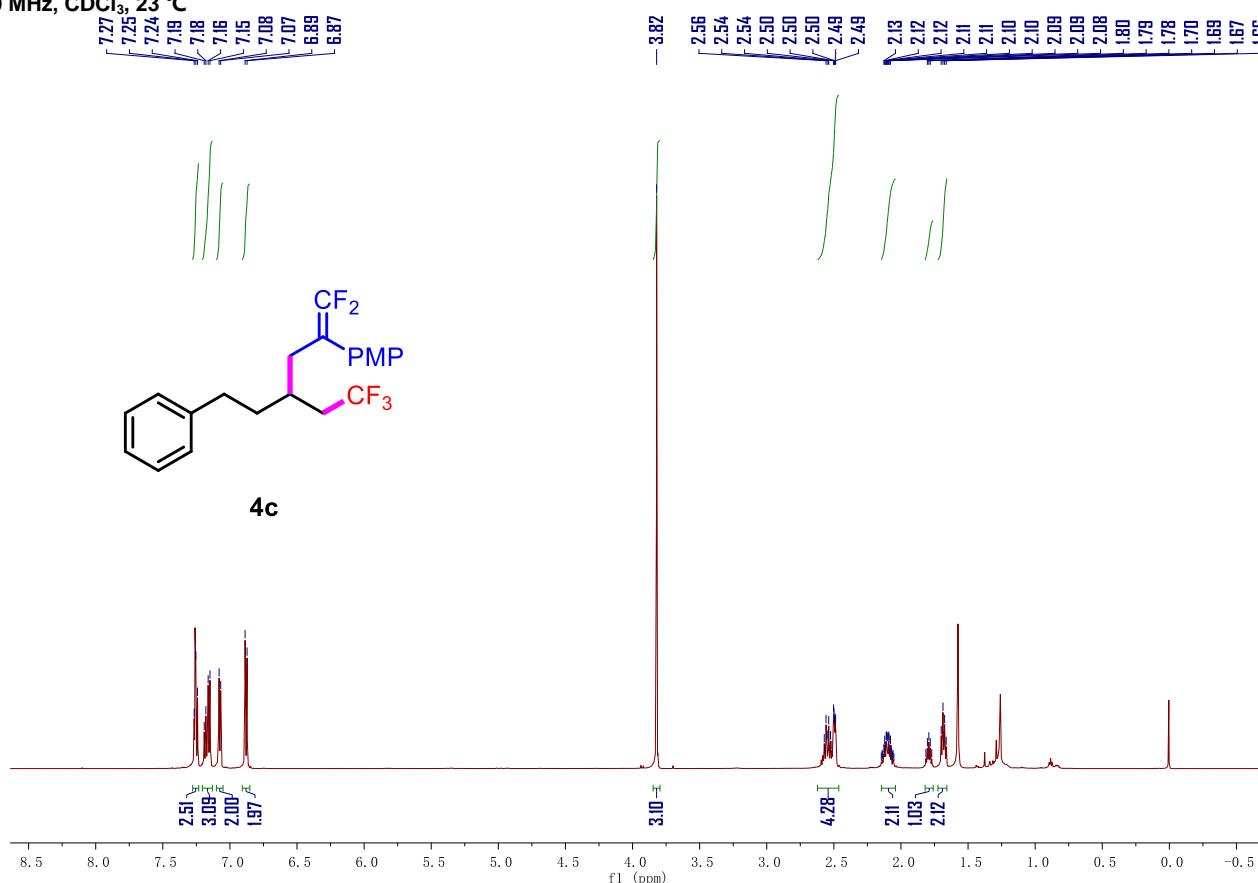
¹⁹F NMR spectrum of 1-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)naphthalene (4b)

565 MHz, CDCl₃, 23 °C



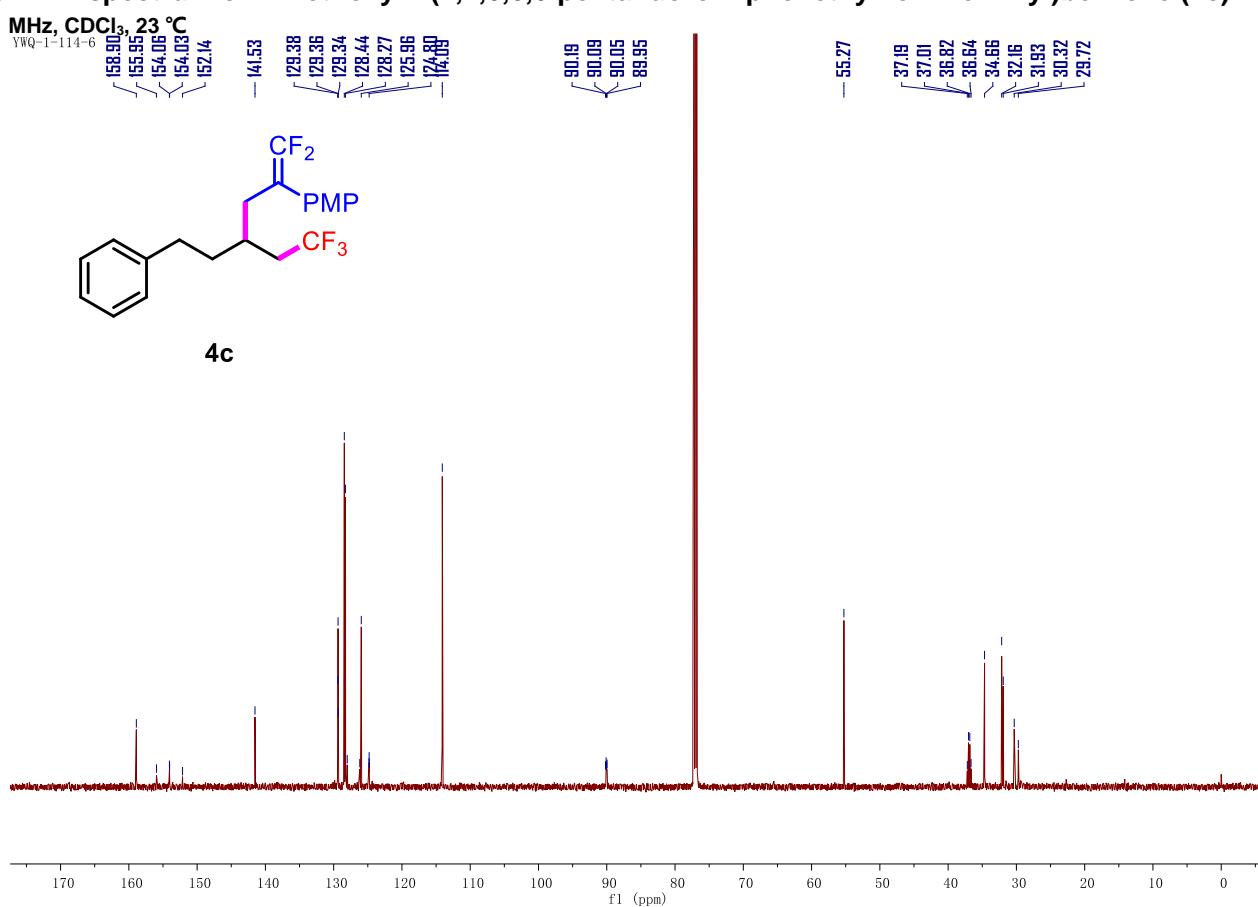
¹H NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4-phenethylhex-1-en-2-yl)benzene (4c)

600 MHz, CDCl₃, 23 °C

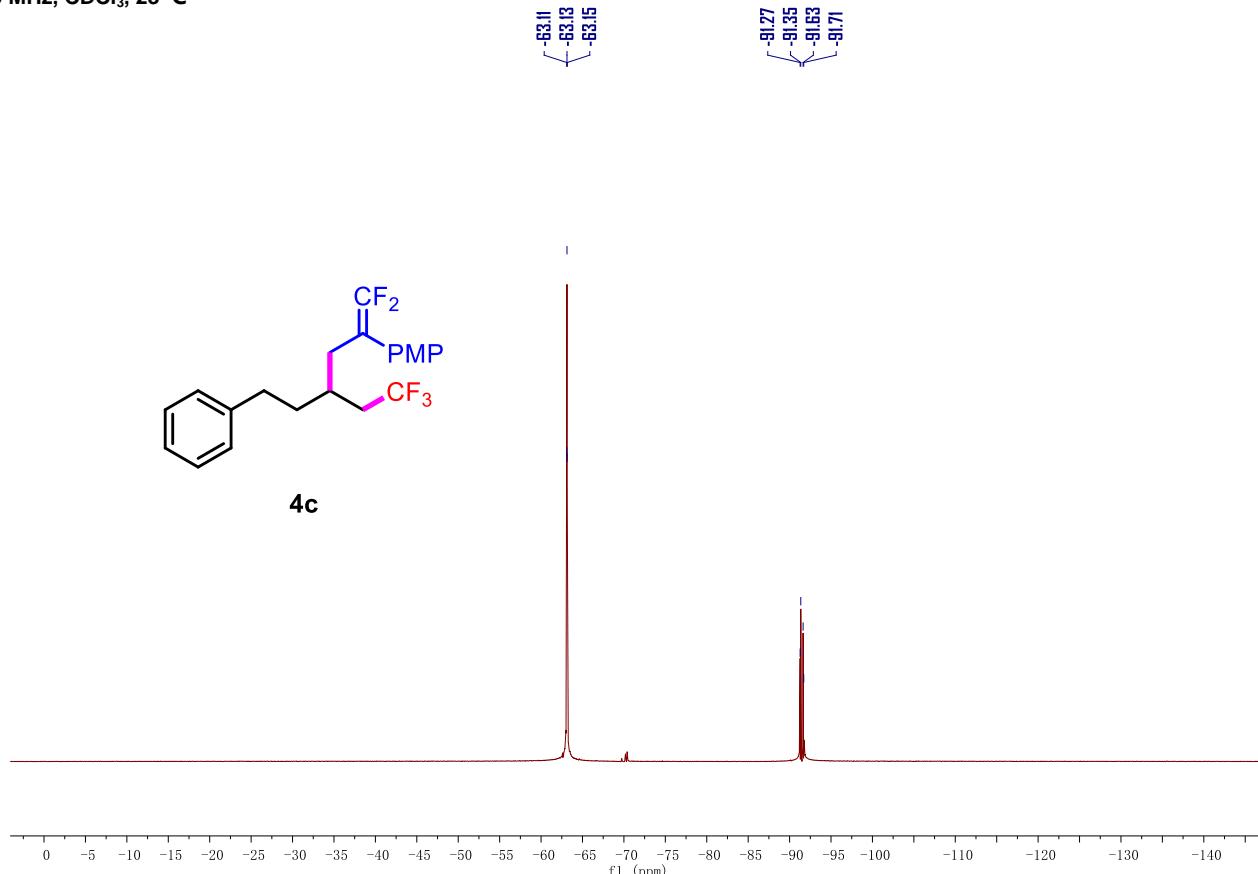


¹³C NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4-phenethylhex-1-en-2-yl)benzene (4c)

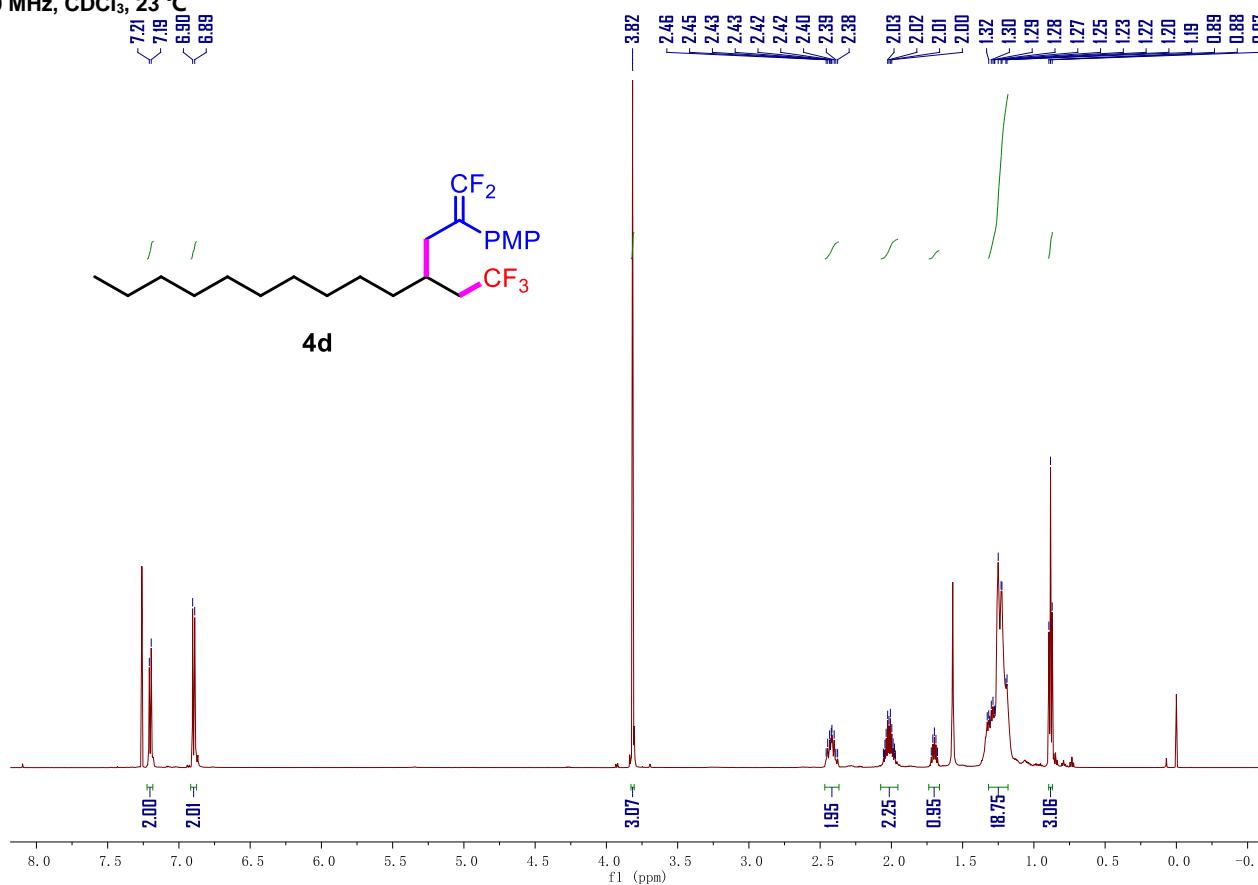
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4-phenethylhex-1-en-2-yl)benzene (4c)
565 MHz, CDCl₃, 23 °C

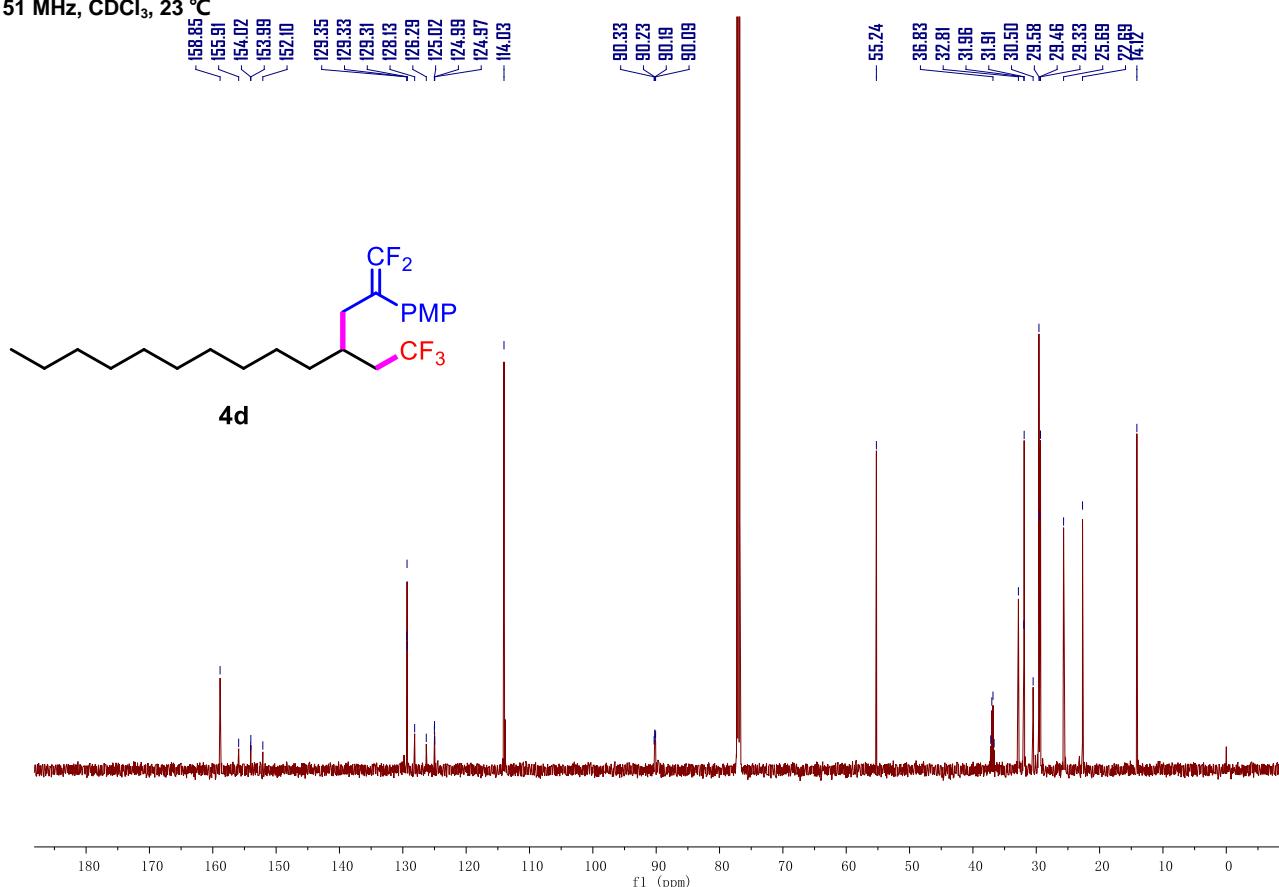


¹H NMR spectrum of 1-(1,1-difluoro-4-(2,2,2-trifluoroethyl)tetradec-1-en-2-yl)-4-methoxybenzene (4d)
600 MHz, CDCl₃, 23 °C



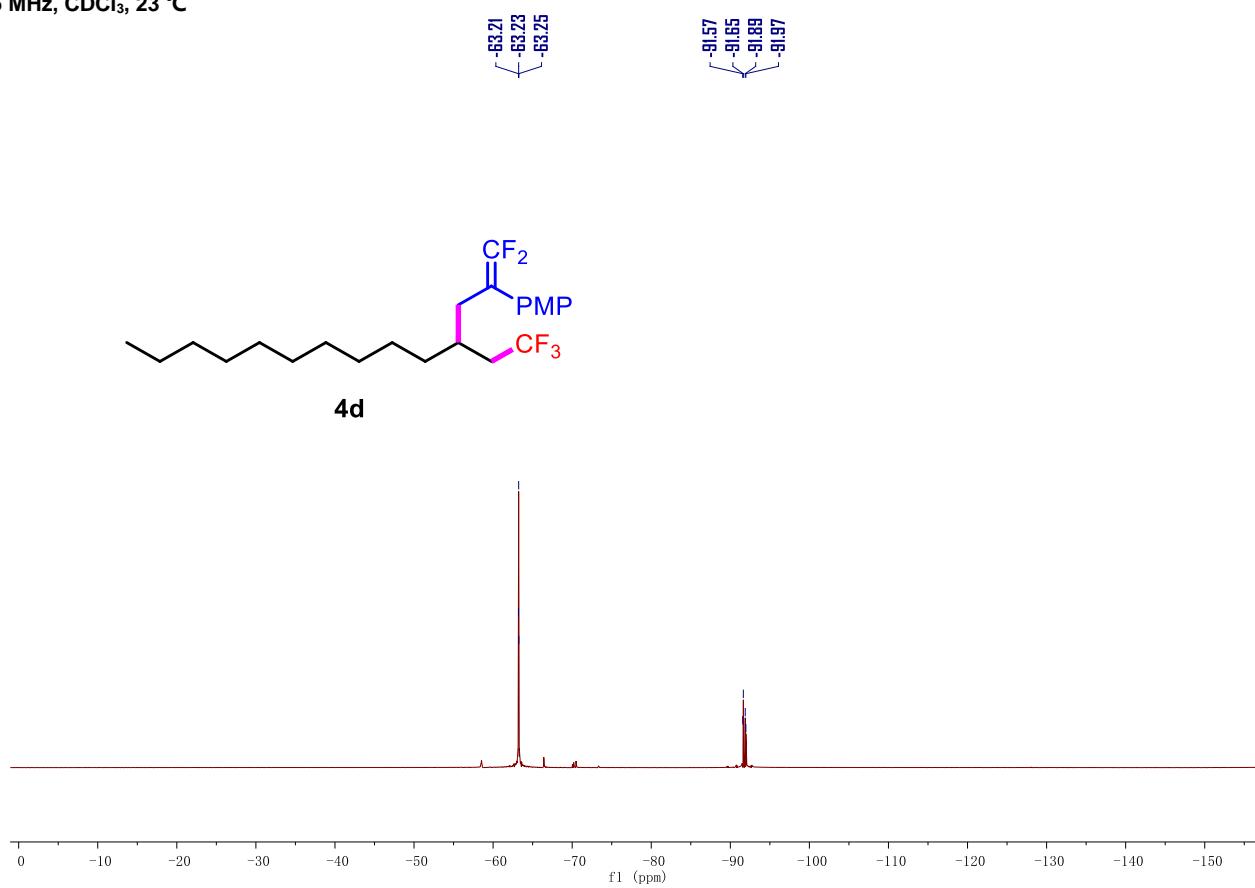
¹³C NMR spectrum of 1-(1,1-difluoro-4-(2,2,2-trifluoroethyl)tetradec-1-en-2-yl)-4-methoxybenzene (4d)

151 MHz, CDCl₃, 23 °C



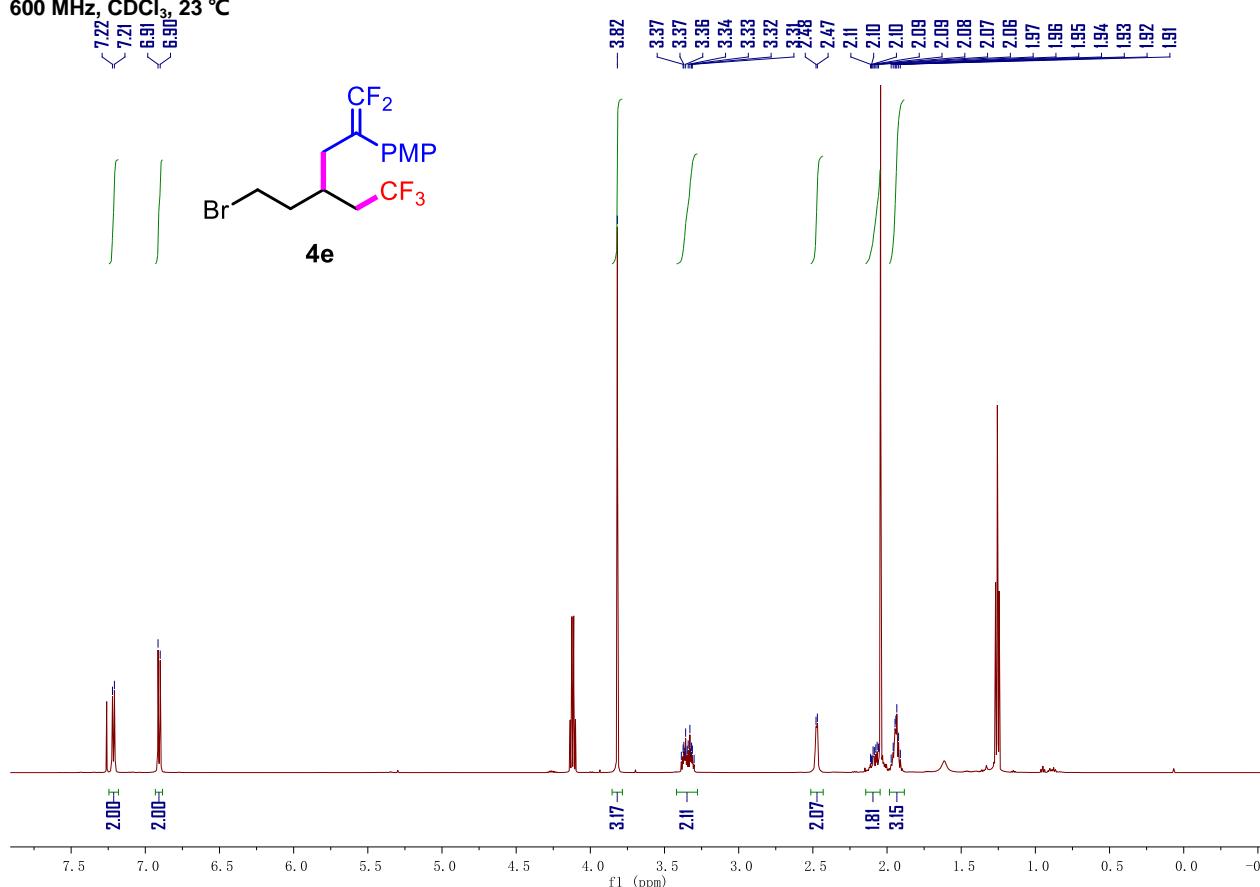
¹⁹F NMR spectrum of 1-(1,1-difluoro-4-(2,2,2-trifluoroethyl)tetradec-1-en-2-yl)-4-methoxybenzene (4d)

565 MHz, CDCl₃, 23 °C



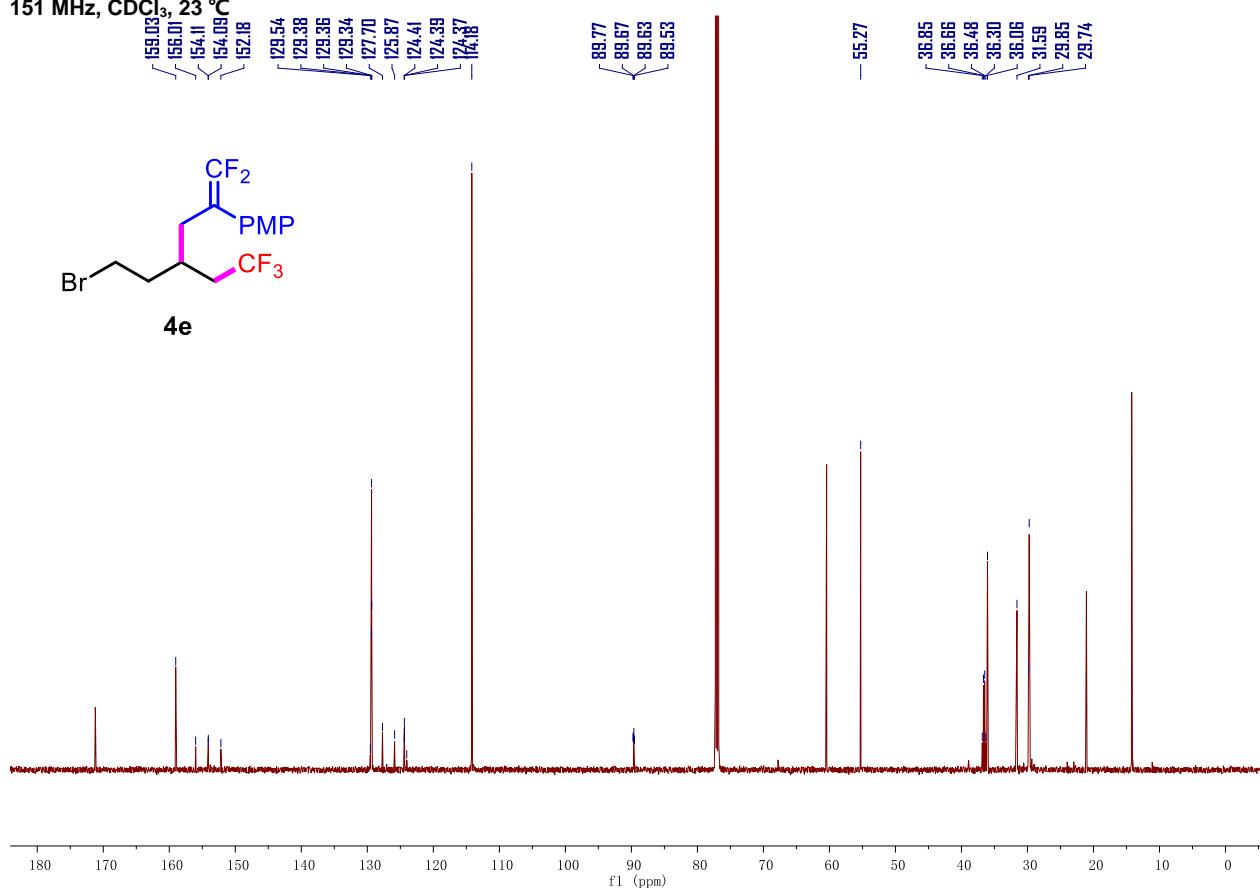
¹H NMR spectrum of 1-(4-(2-bromoethyl)-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4e)

600 MHz, CDCl₃, 23 °C

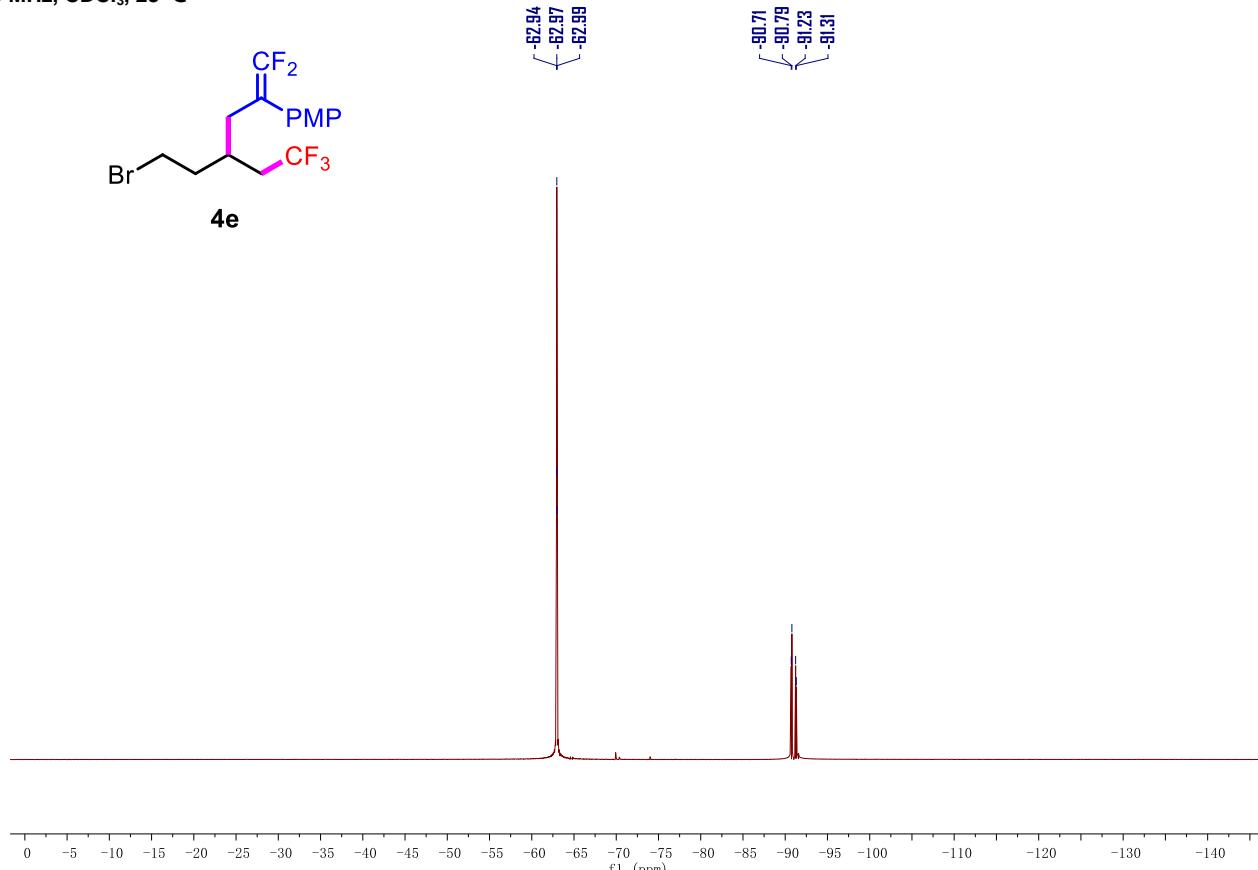


¹³C NMR spectrum of 1-(4-(2-bromoethyl)-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4e)

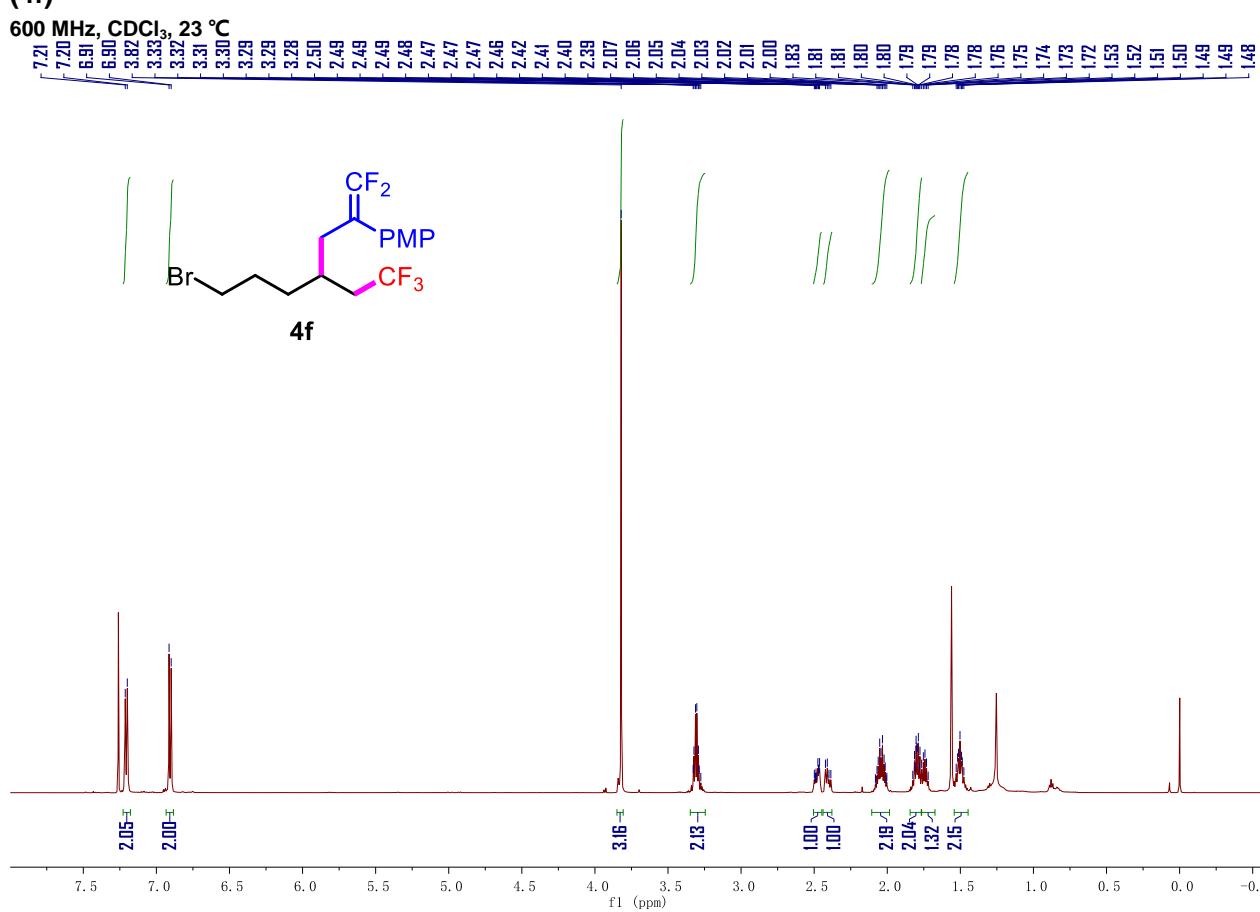
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(4-(2-bromoethyl)-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4e)
565 MHz, CDCl₃, 23 °C

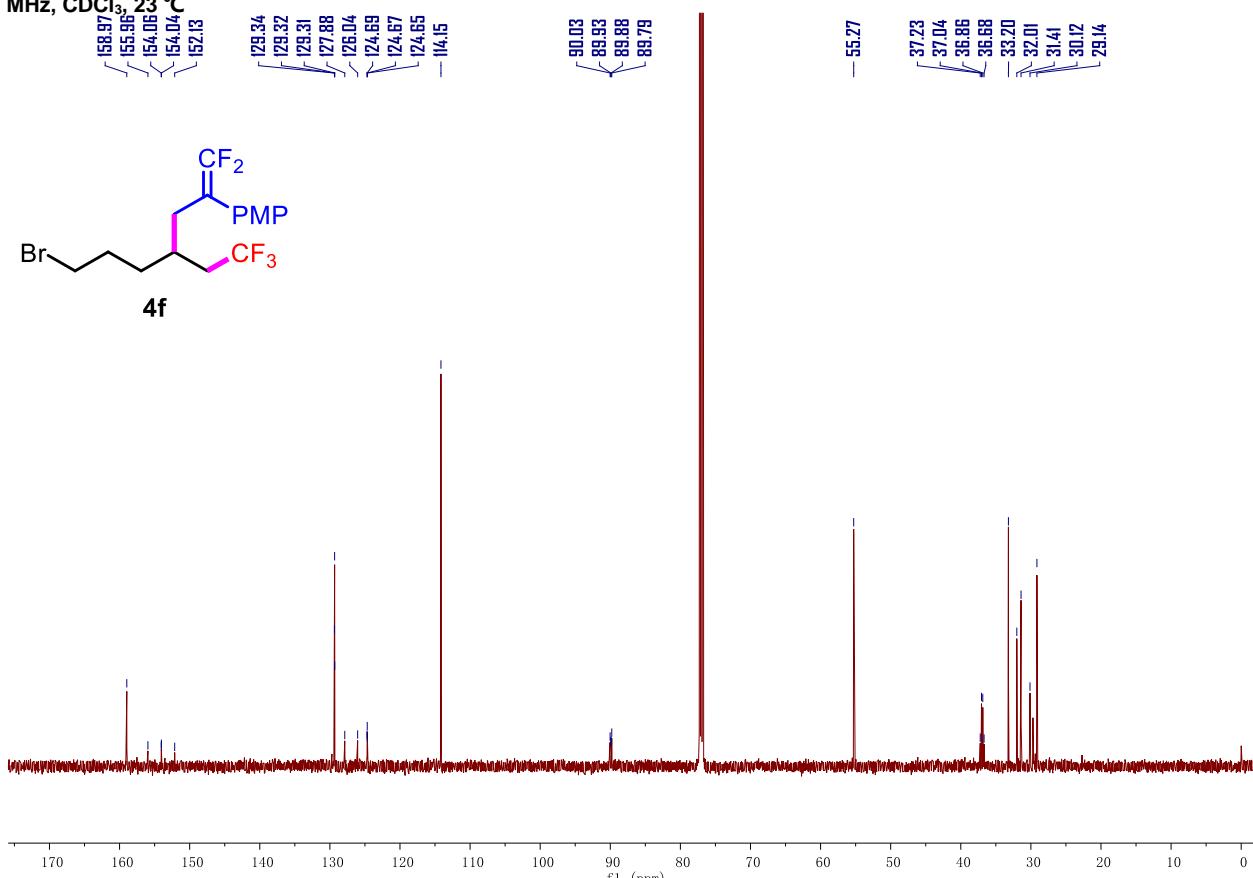


¹H NMR spectrum of 1-(7-bromo-1,1-difluoro-4-(2,2,2-trifluoroethyl)hept-1-en-2-yl)-4-methoxybenzene (4f)



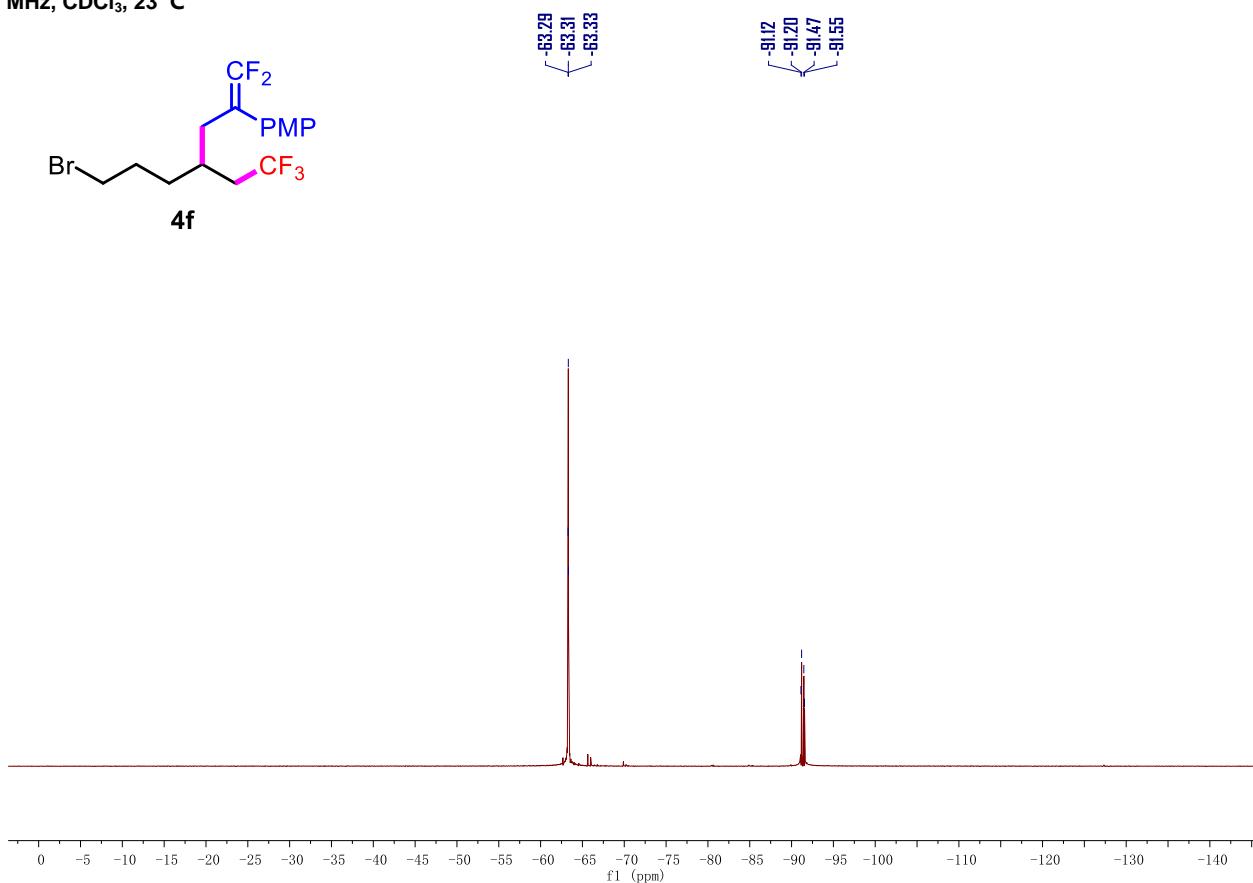
¹³C NMR spectrum of 1-(7-bromo-1,1-difluoro-4-(2,2,2-trifluoroethyl)hept-1-en-2-yl)-4-methoxybenzene (4f)

151 MHz, CDCl₃, 23 °C



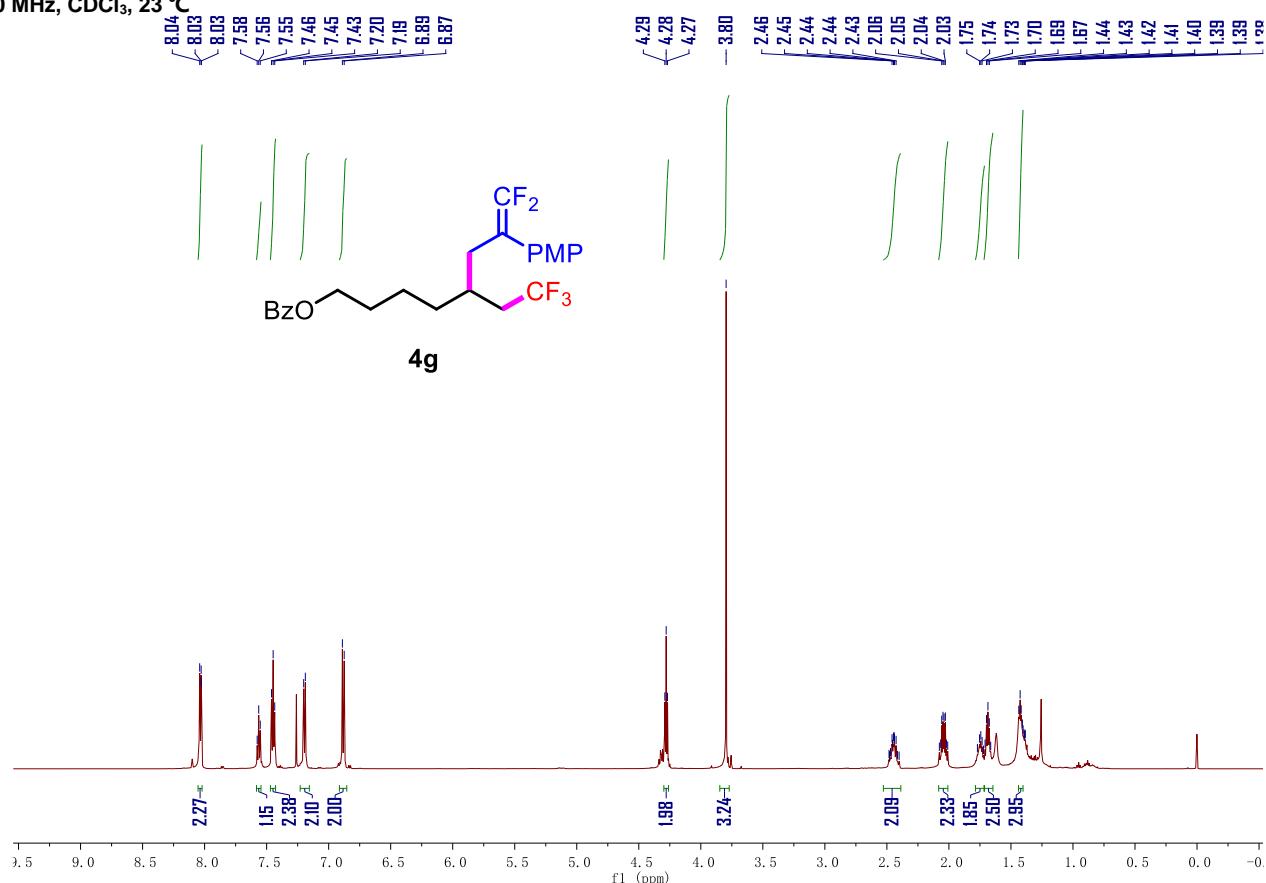
¹⁹F NMR spectrum of 1-(7-bromo-1,1-difluoro-4-(2,2,2-trifluoroethyl)hept-1-en-2-yl)-4-methoxybenzene (4f)

565 MHz, CDCl₃, 23 °C



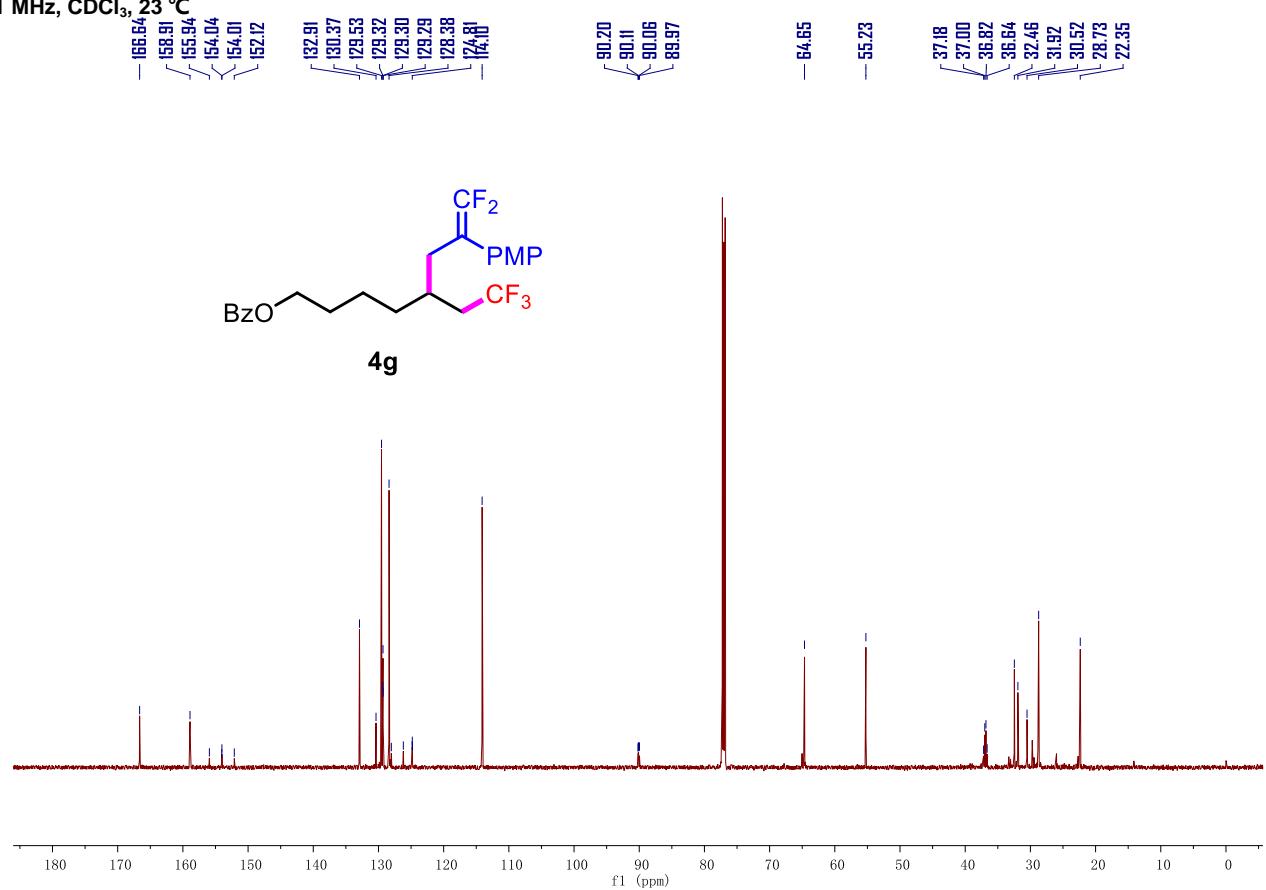
¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl benzoate (4g)

600 MHz, CDCl₃, 23 °C

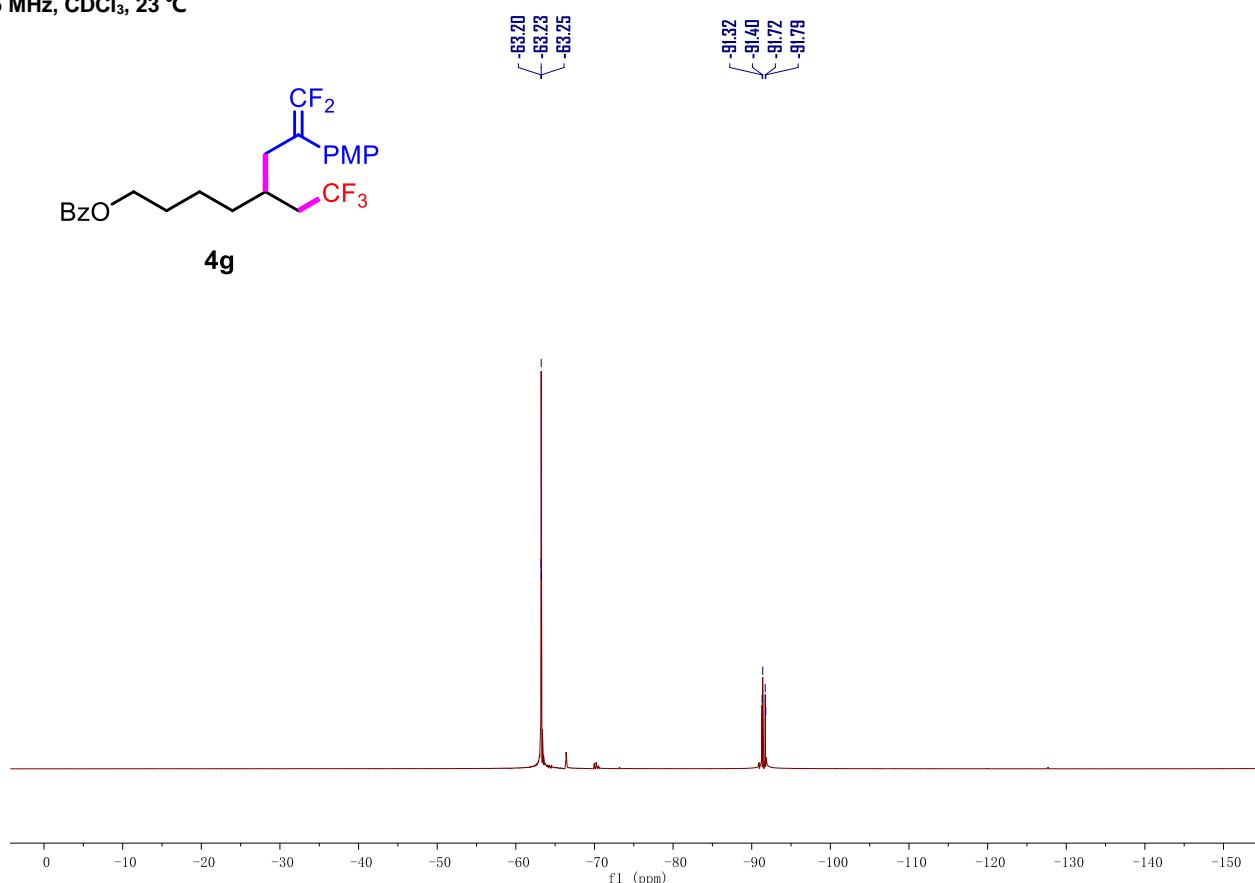


¹³C NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl benzoate (4g)

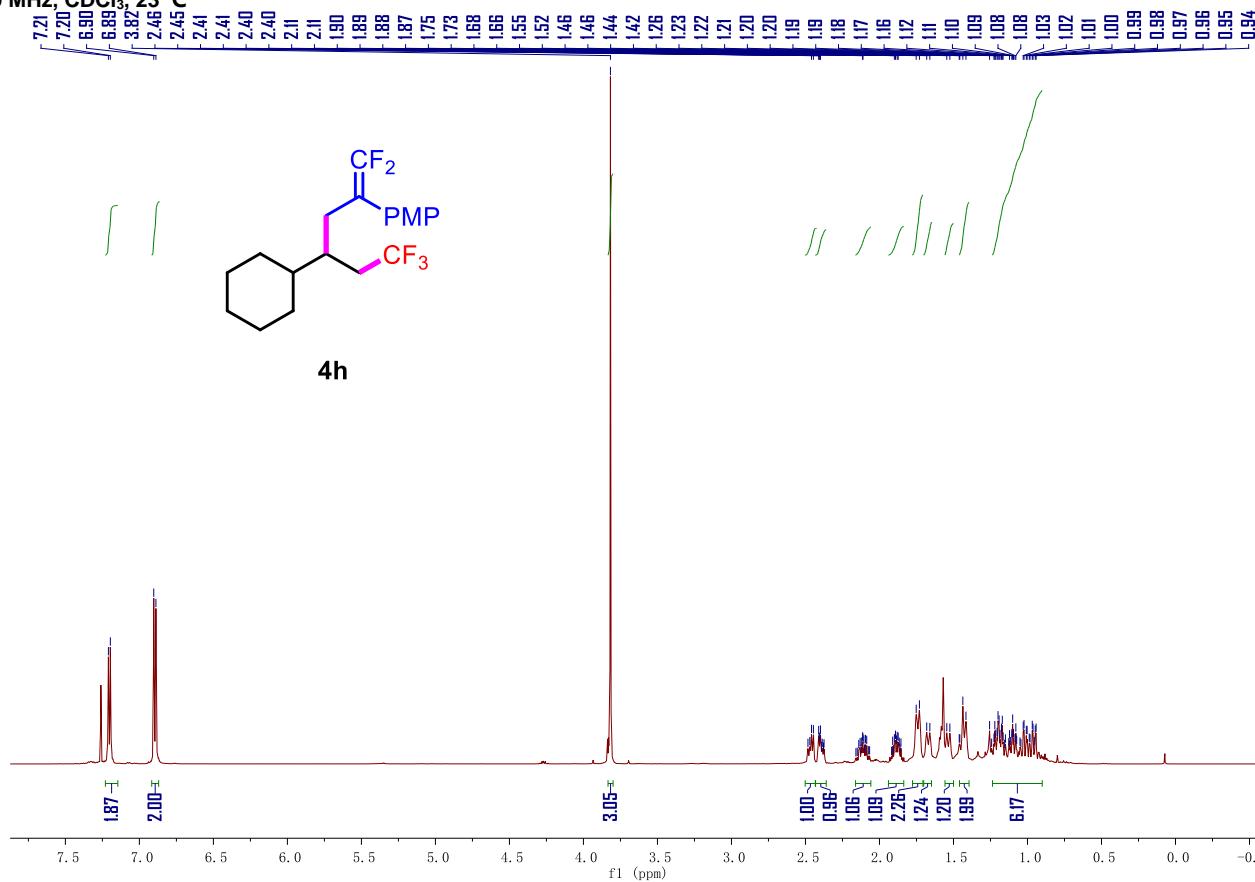
151 MHz, CDCl₃, 23 °C



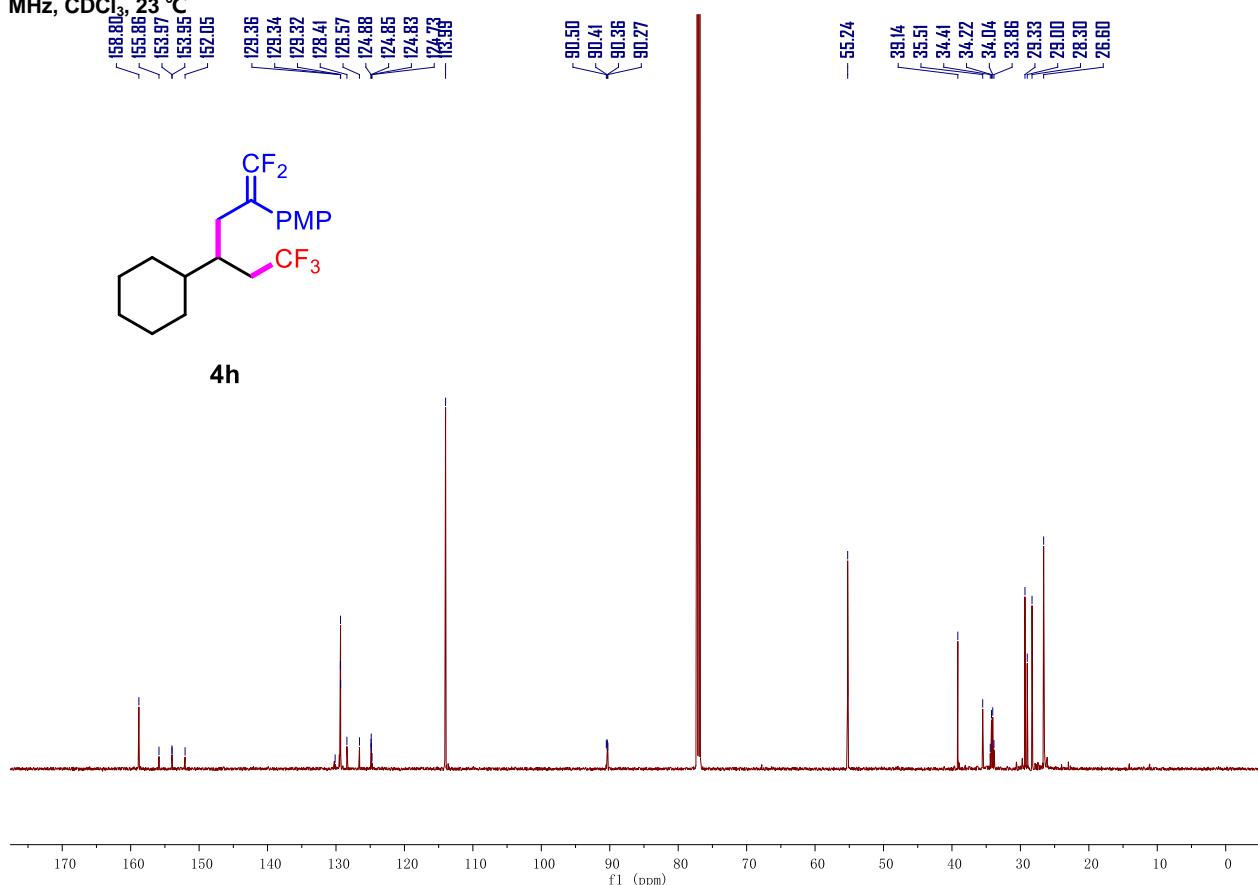
¹⁹F NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl benzoate (4g)
565 MHz, CDCl₃, 23 °C



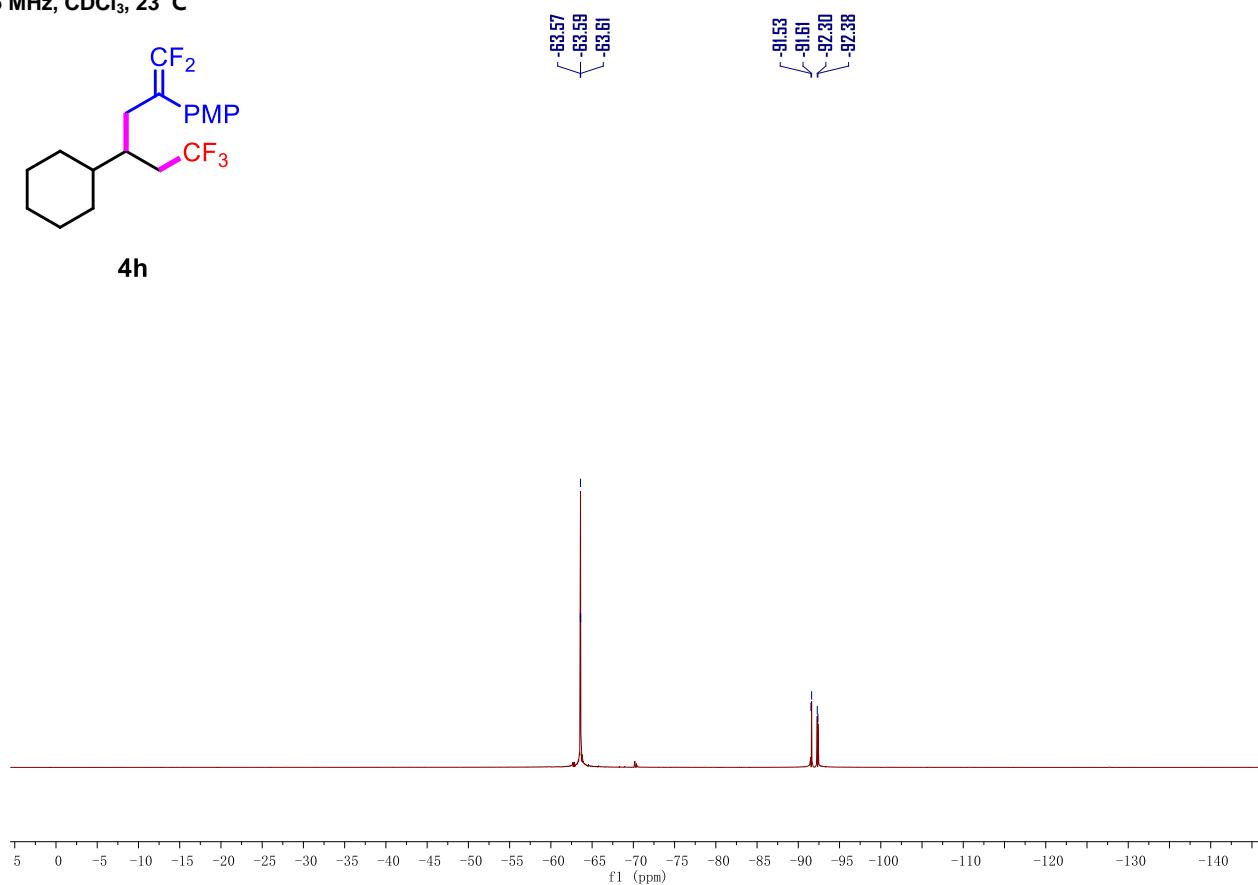
¹H NMR spectrum of 1-(4-cyclohexyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4h)
600 MHz, CDCl₃, 23 °C



¹³C NMR spectrum of 1-(4-cyclohexyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4h)
 151 MHz, CDCl₃, 23 °C



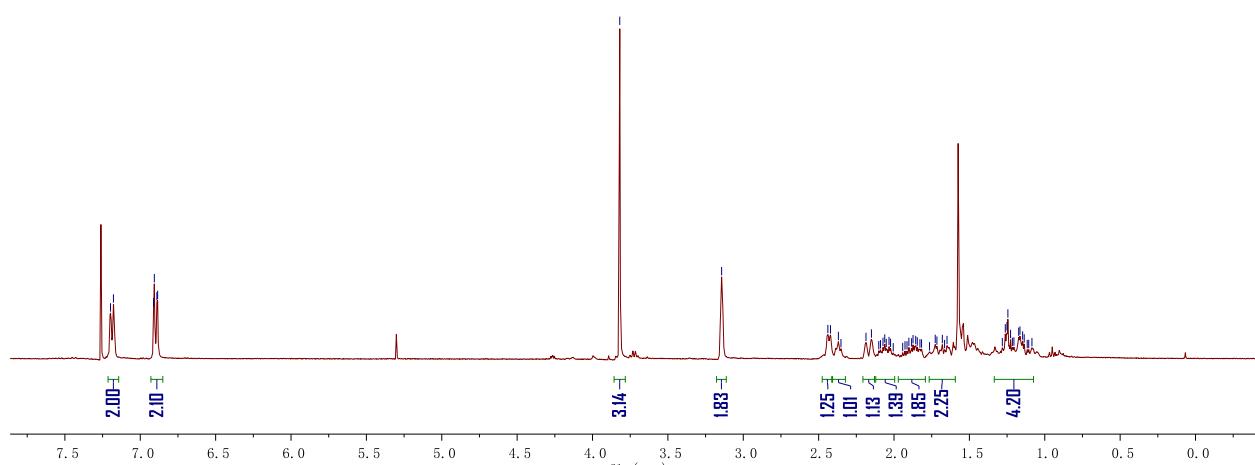
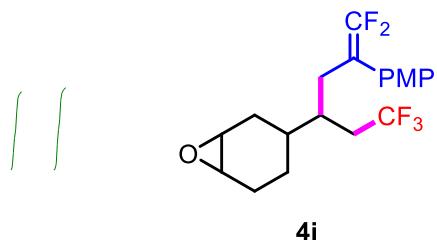
¹⁹F NMR spectrum of 1-(4-cyclohexyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4h)
 565 MHz, CDCl₃, 23 °C



¹H NMR spectrum of 3-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)-7-oxabicyclo[4.1.0]heptane (4i)

600 MHz, CDCl₃, 23 °C

7.20
7.18
6.91
6.89
6.88

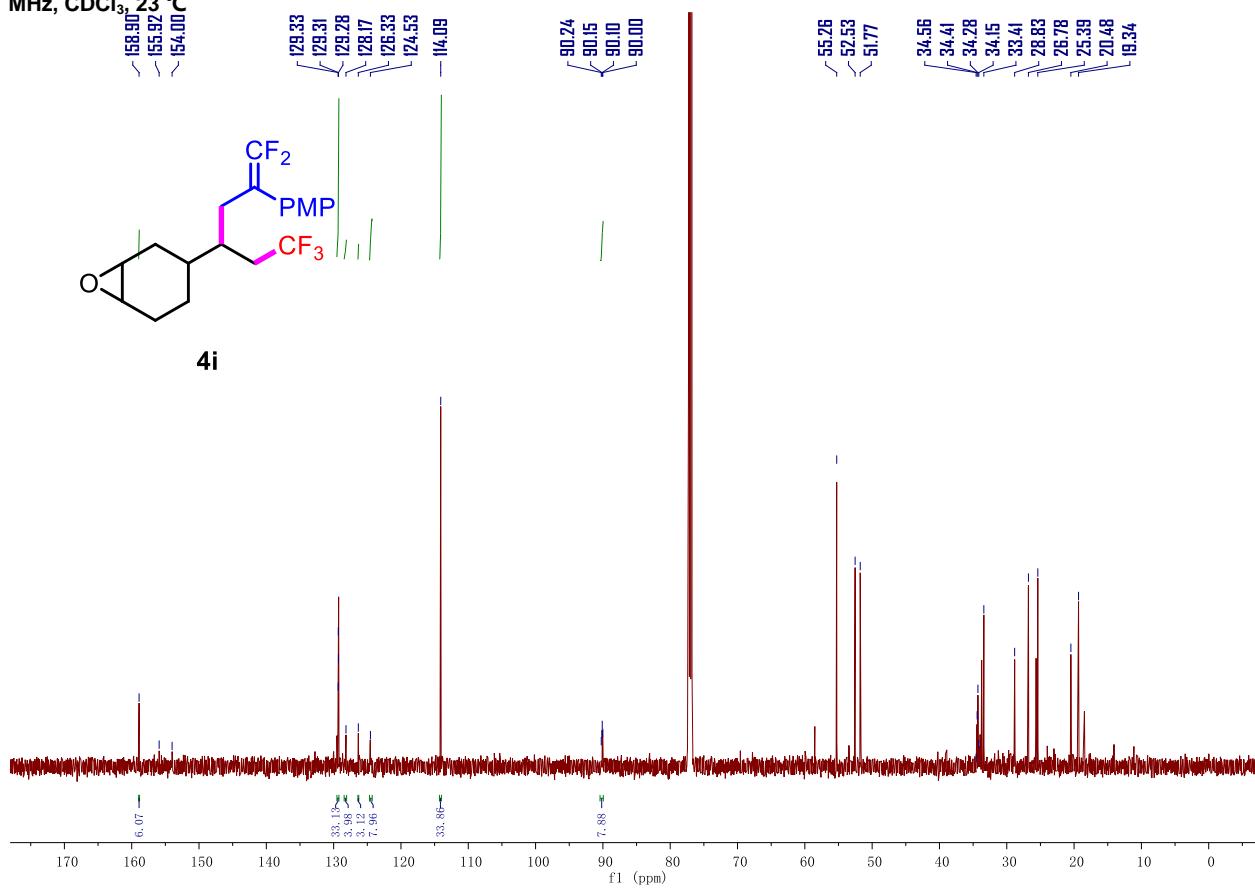
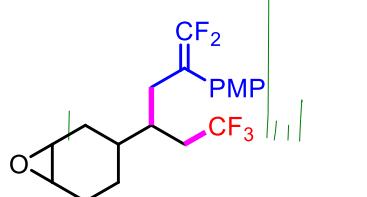


¹³C NMR spectrum of 3-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)-7-oxabicyclo[4.1.0]heptane (4i)

151 MHz, CDCl₃, 23 °C

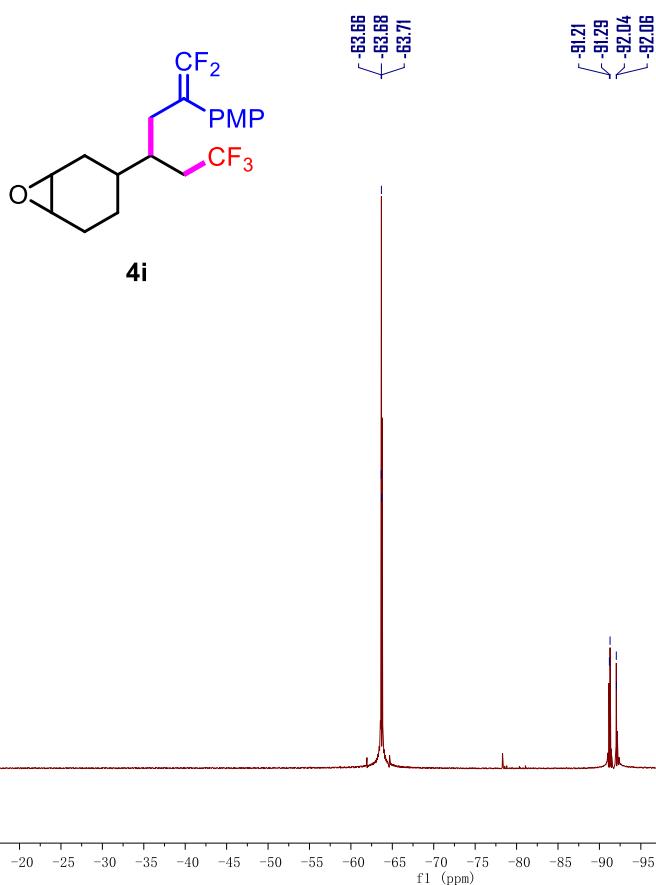
158.90
155.92
154.00

125.33
125.31
122.28
122.17
122.33
124.53
114.09



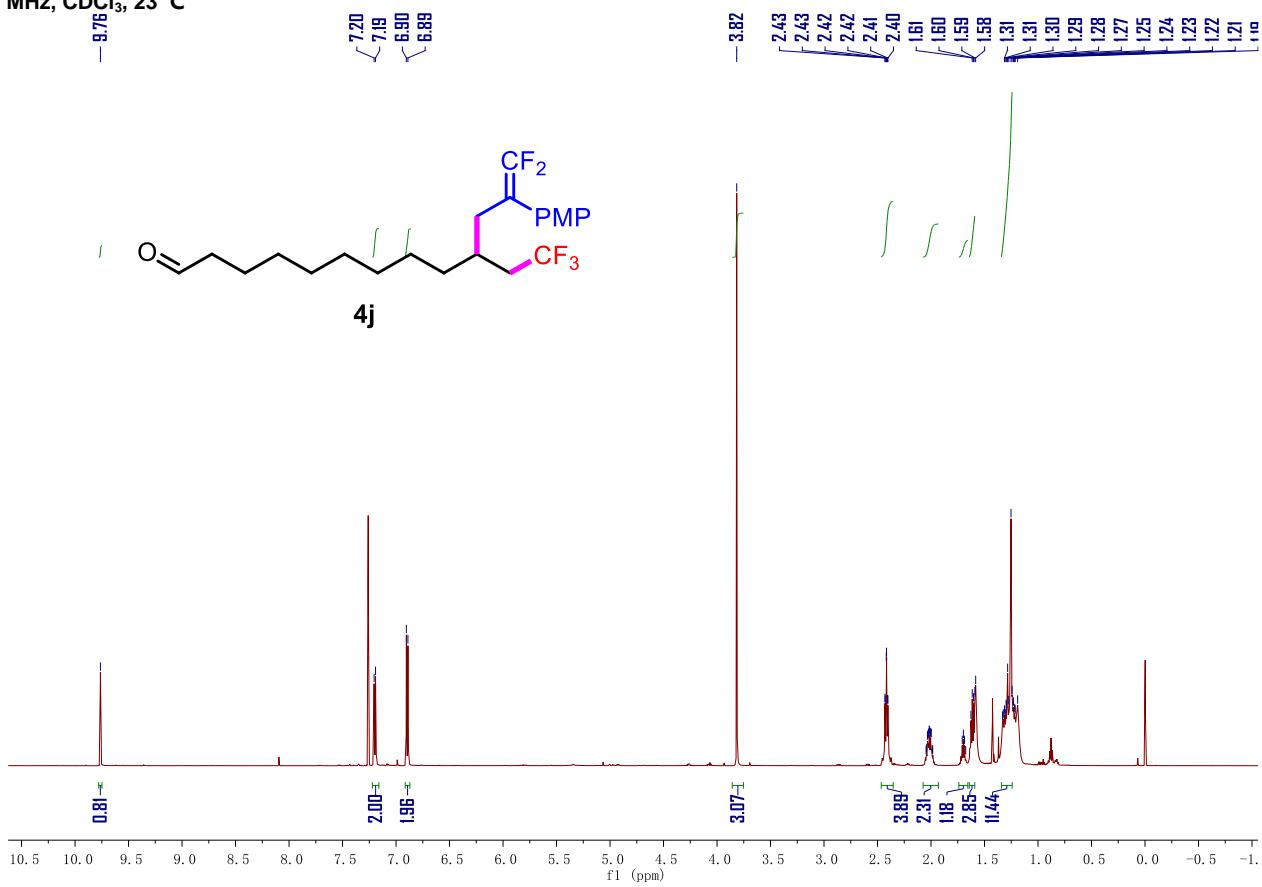
¹⁹F NMR spectrum of 3-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)-7-oxabicyclo[4.1.0]heptane (4i)

565 MHz, CDCl₃, 23 °C



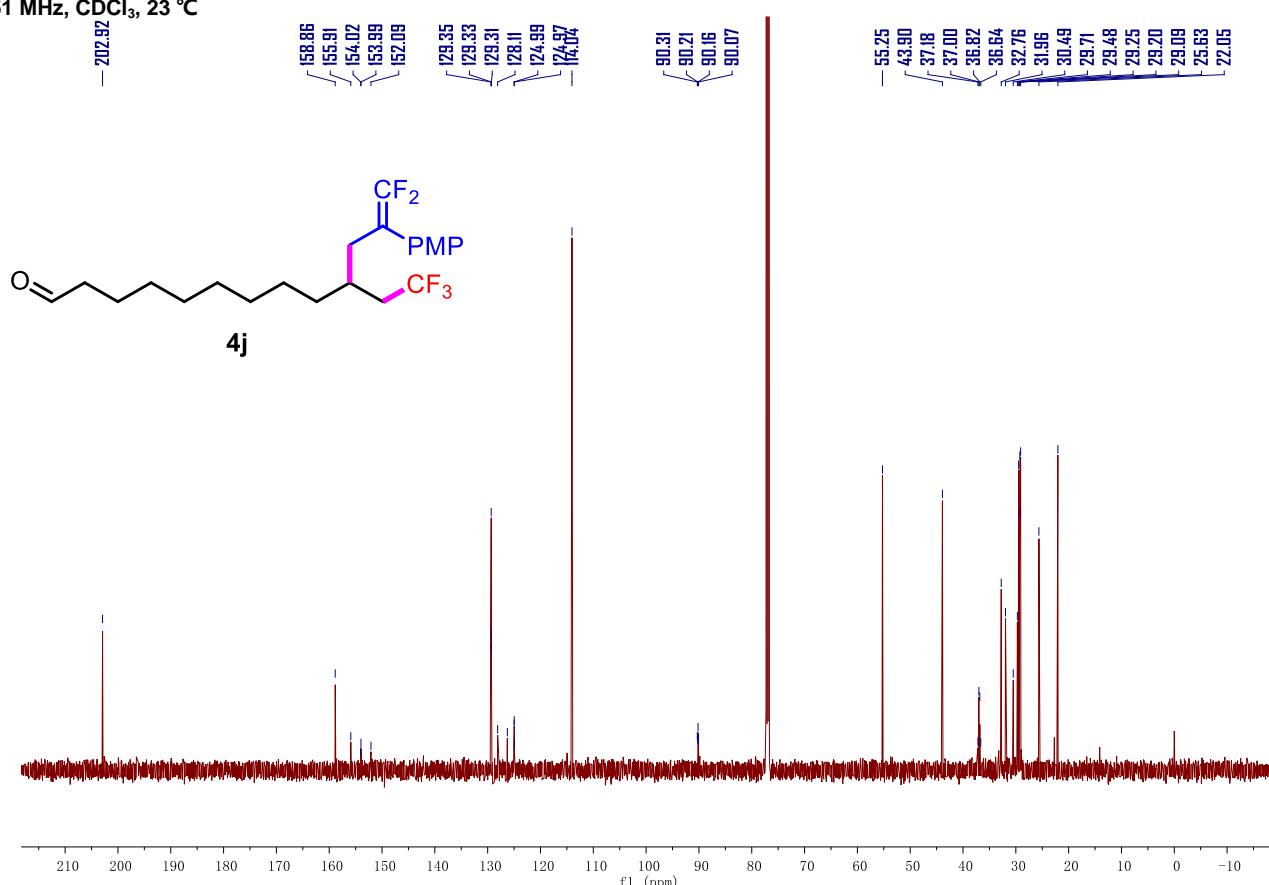
¹H NMR spectrum of 13,13-difluoro-12-(4-methoxyphenyl)-10-(2,2,2-trifluoroethyl)tridec-12-enal (4j)

600 MHz, CDCl₃, 23 °C



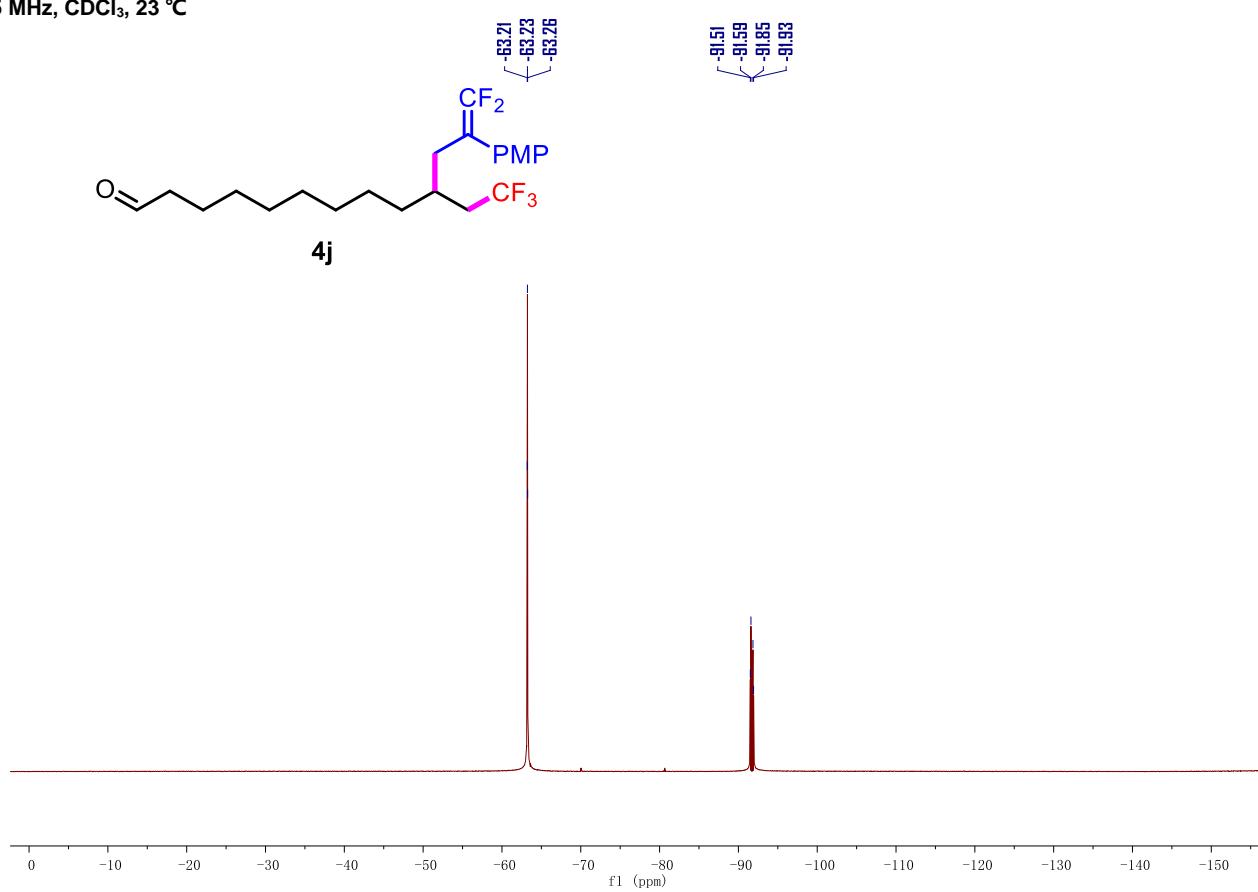
¹³C NMR spectrum of 13,13-difluoro-12-(4-methoxyphenyl)-10-(2,2,2-trifluoroethyl)tridec-12-enal (4j)

151 MHz, CDCl₃, 23 °C



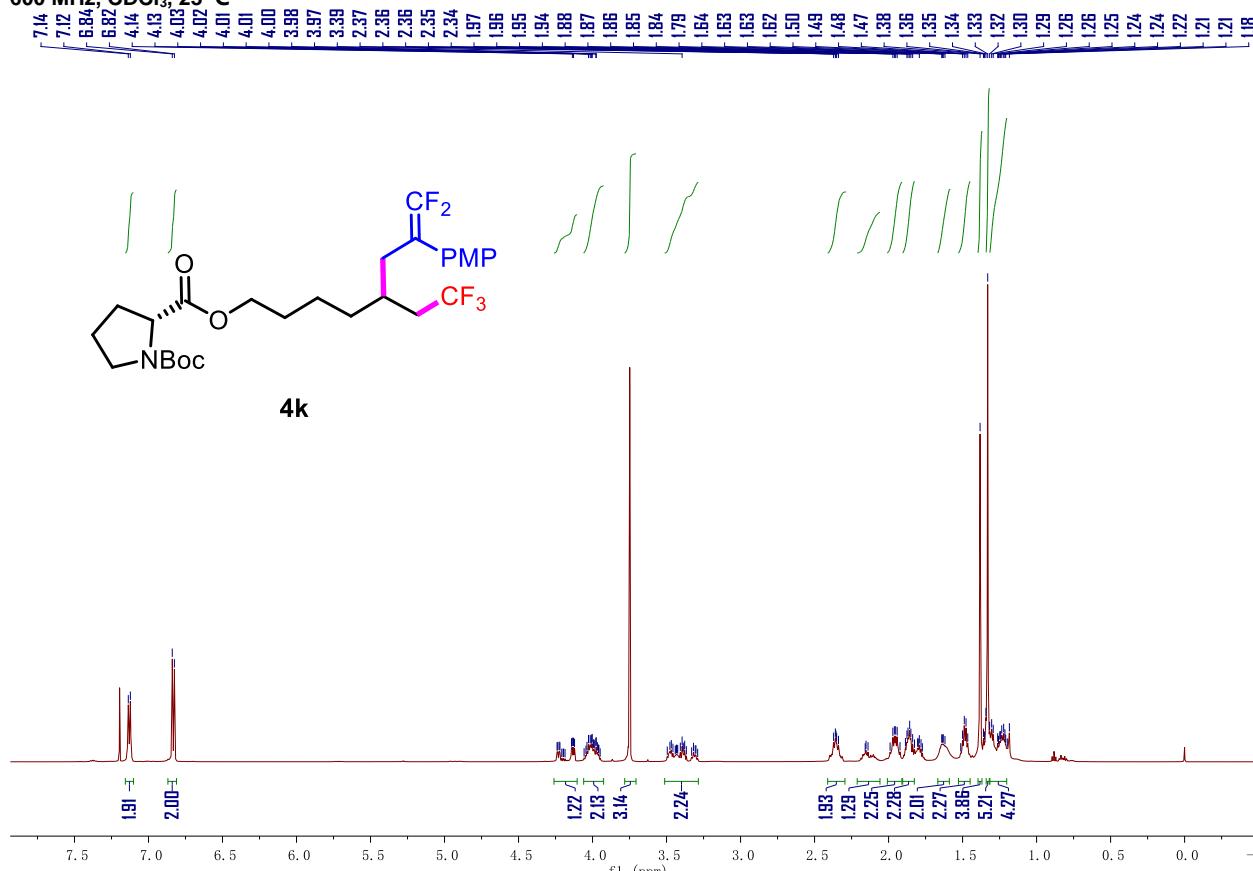
¹⁹F NMR spectrum of 13,13-difluoro-12-(4-methoxyphenyl)-10-(2,2,2-trifluoroethyl)tridec-12-enal (4j)

565 MHz, CDCl₃, 23 °C



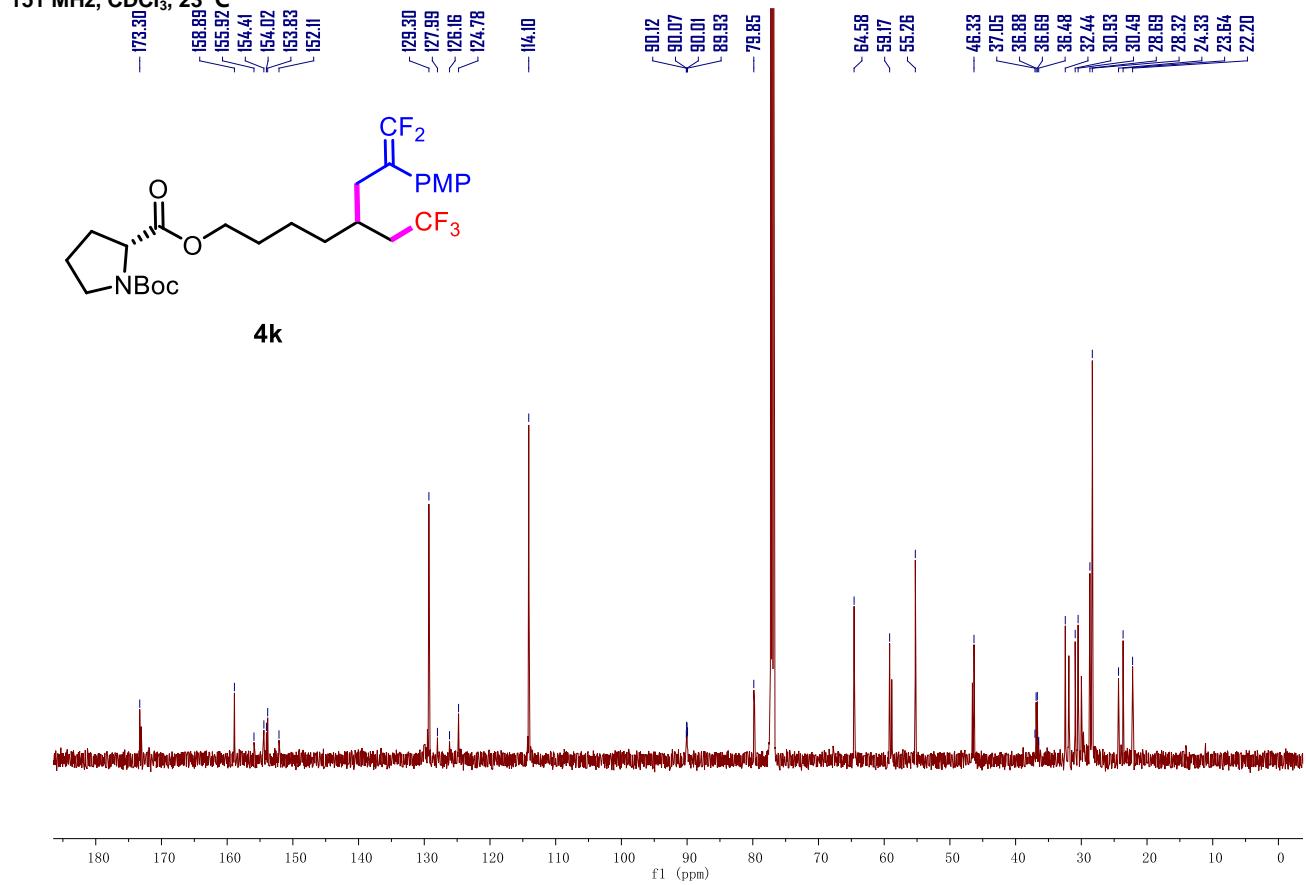
¹H NMR spectrum of 1-(tert-butyl) 2-(8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl) (2R)-pyrrolidine-1,2-dicarboxylate (4k)

600 MHz, CDCl₃, 23 °C

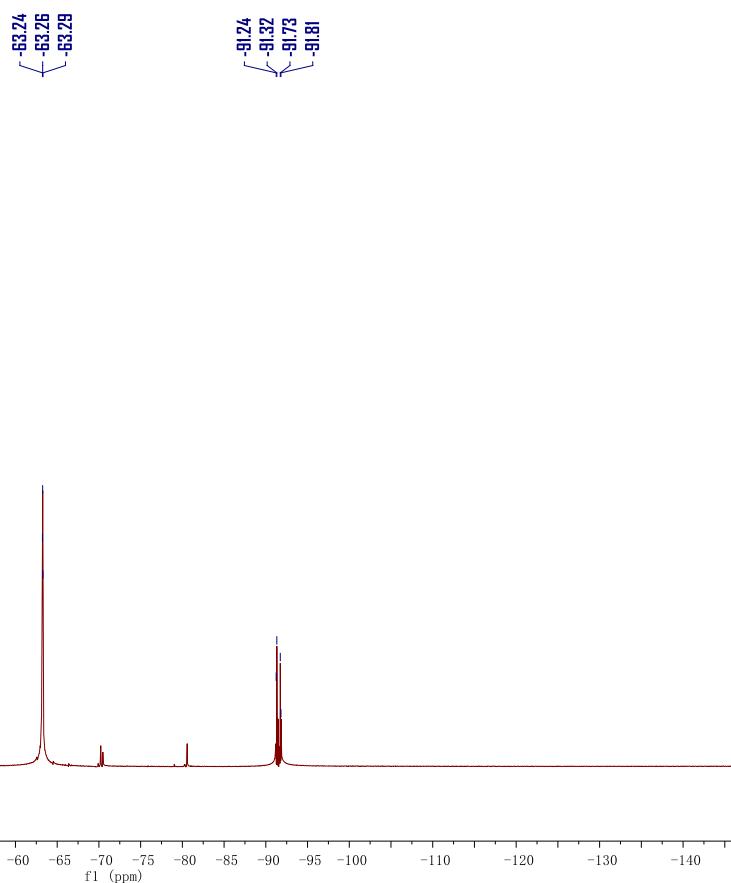
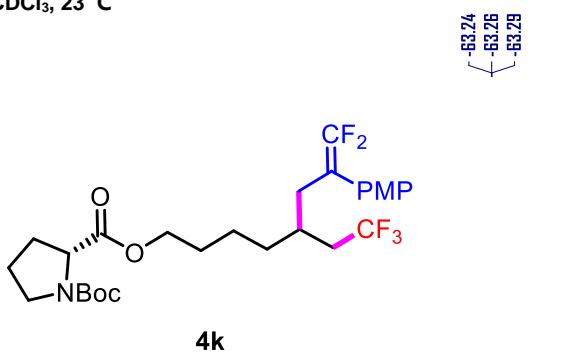


¹³C NMR spectrum of 1-(tert-butyl) 2-(8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl) (2R)-pyrrolidine-1,2-dicarboxylate (4k)

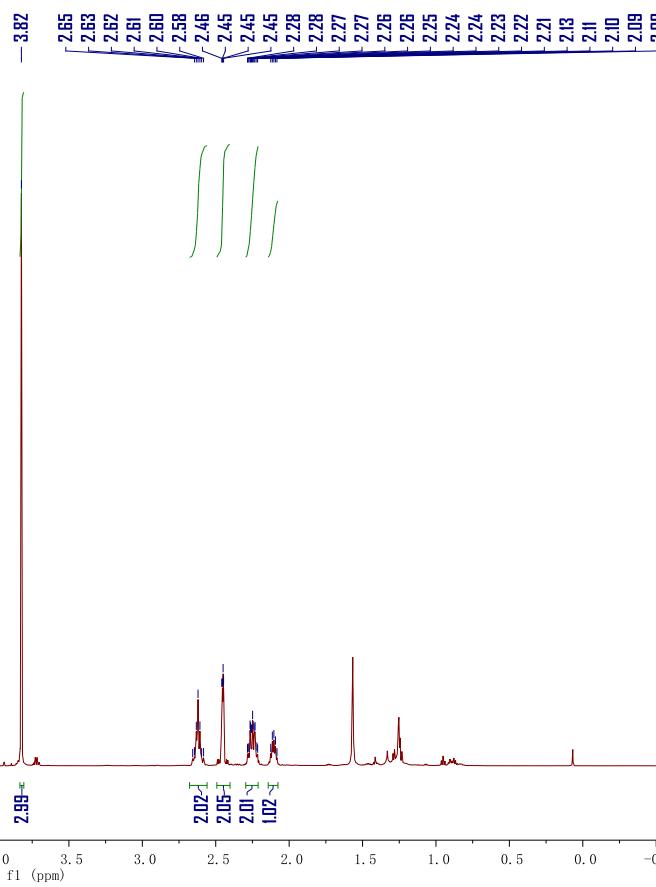
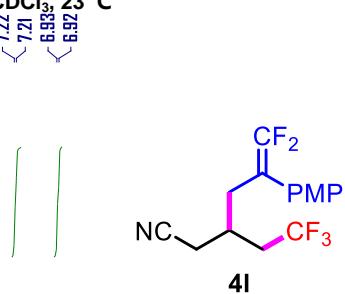
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(*tert*-butyl) 2-(8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl) (2*R*)-pyrrolidine-1,2-dicarboxylate (**4k**)
565 MHz, CDCl₃, 23 °C

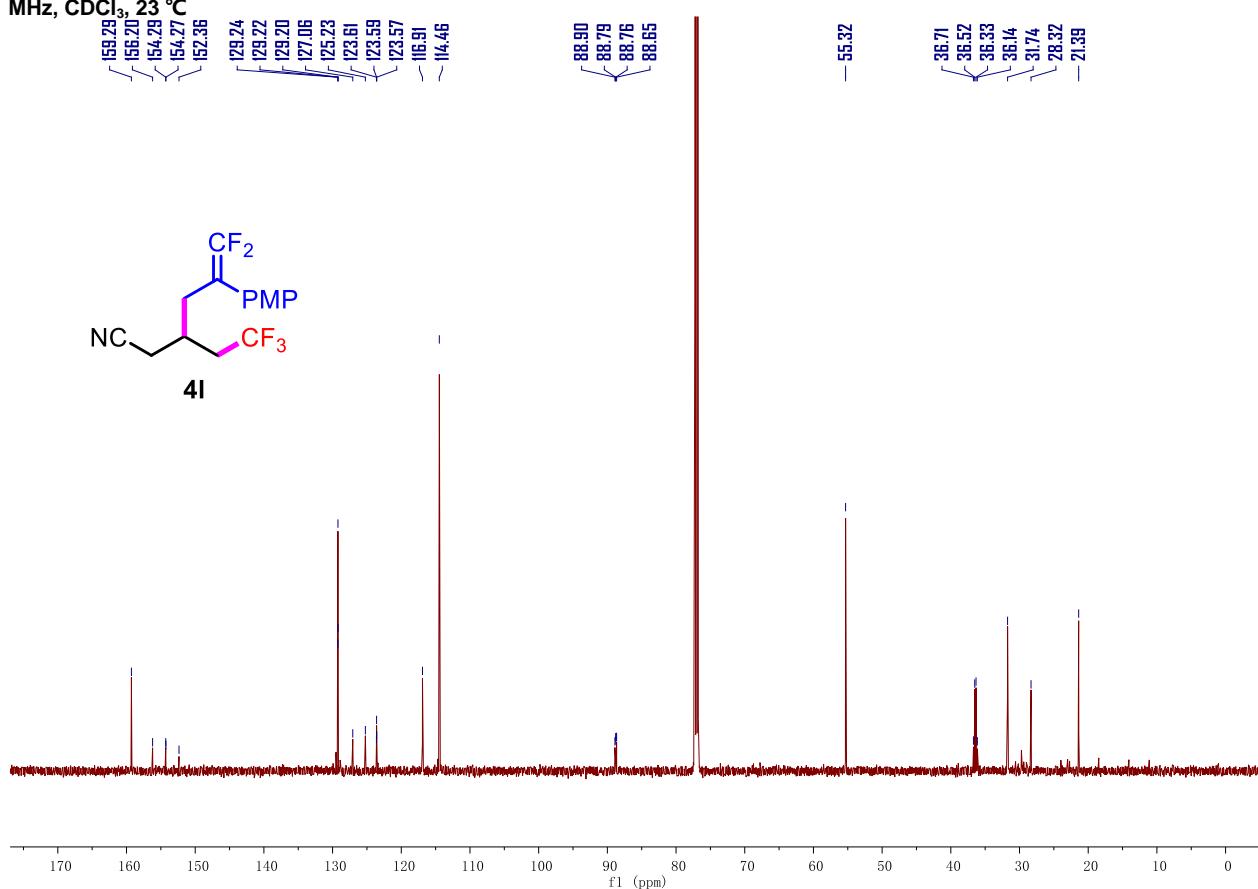


¹H NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-(2,2,2-trifluoroethyl)hex-5-enenitrile (**4l**)
600 MHz, CDCl₃, 23 °C



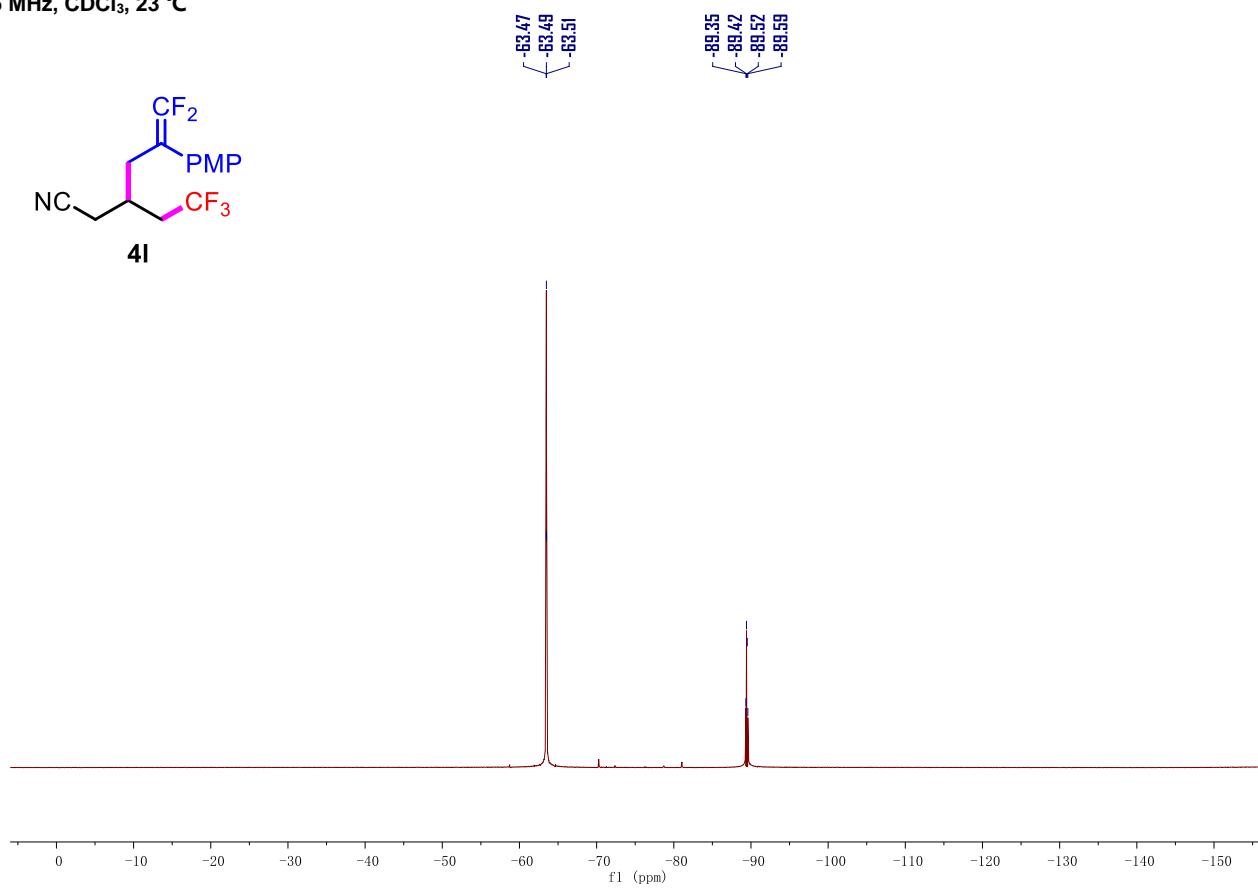
¹³C NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-(2,2,2-trifluoroethyl)hex-5-enenitrile (4l)

151 MHz, CDCl₃, 23 °C



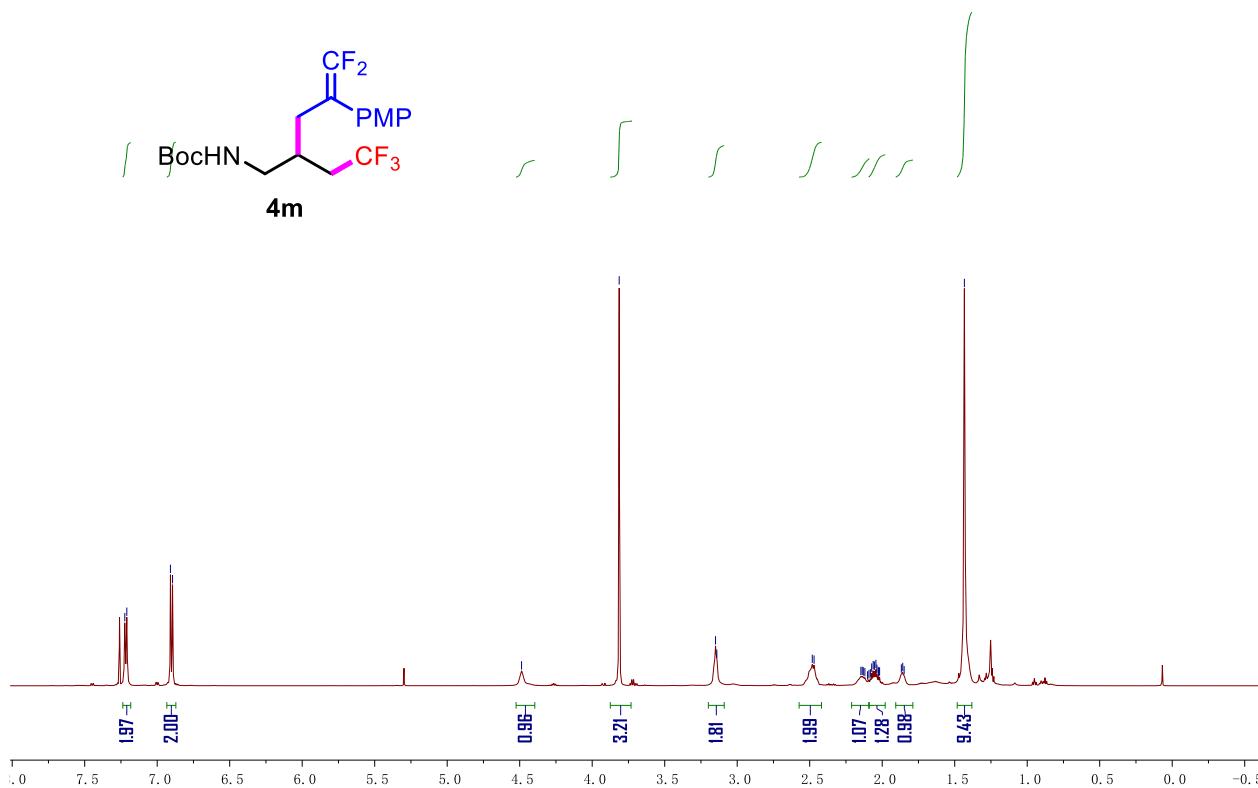
¹⁹F NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-(2,2,2-trifluoroethyl)hex-5-enenitrile (4l)

565 MHz, CDCl₃, 23 °C



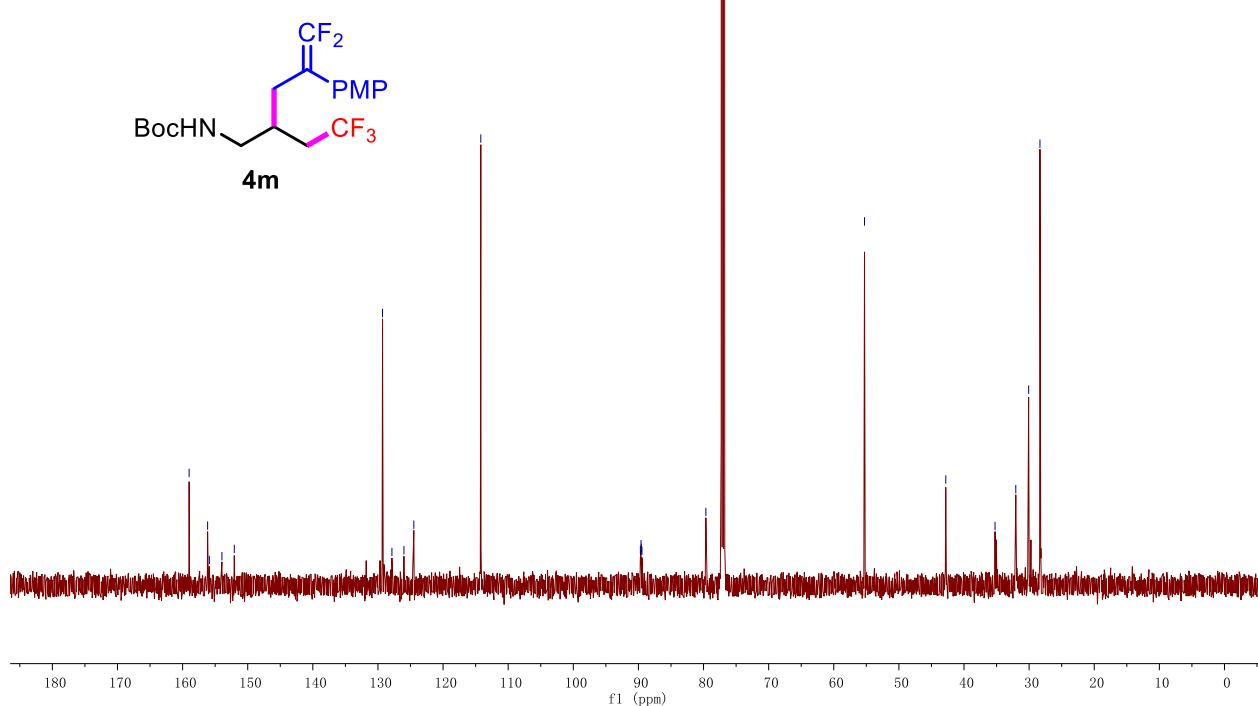
¹H NMR spectrum of tert-butyl (5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)carbamate (4m)

600 MHz, CDCl₃, 23 °C

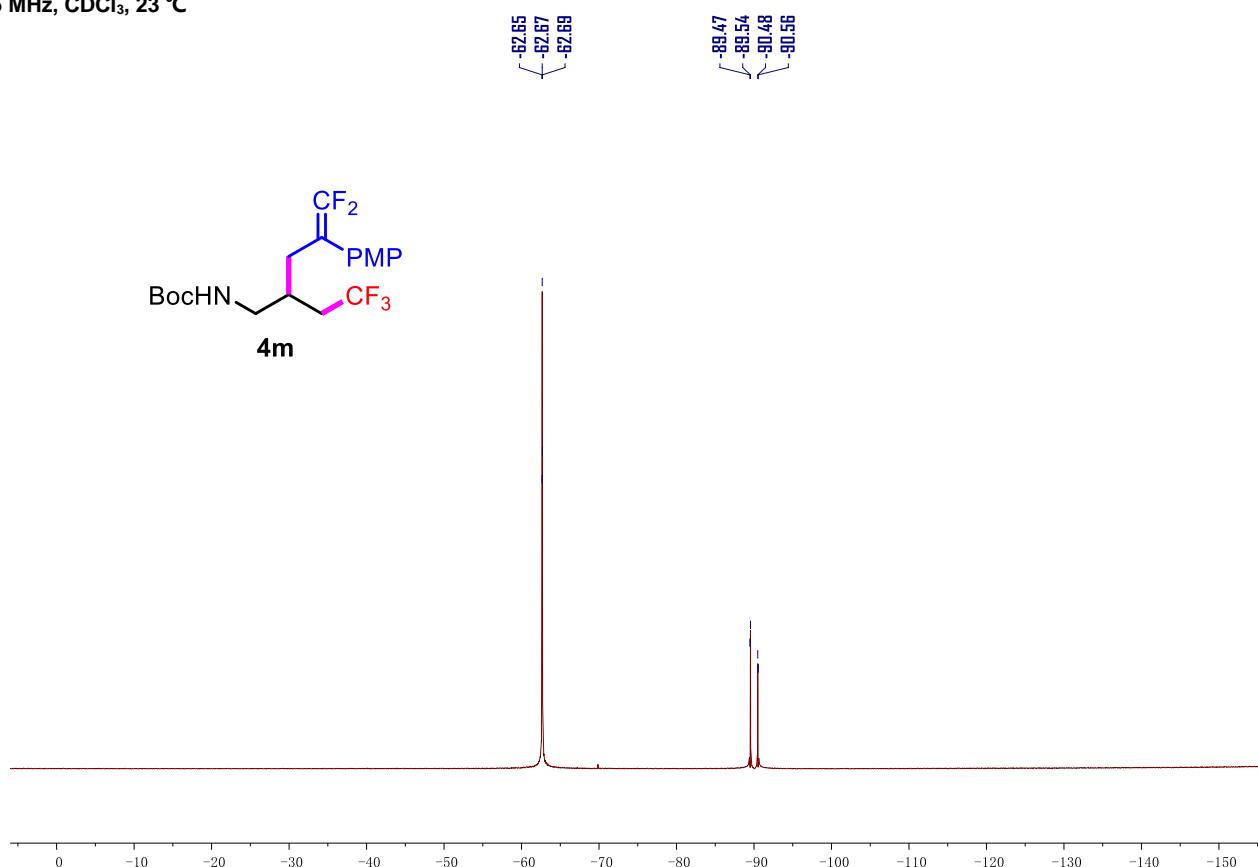


¹³C NMR spectrum of tert-butyl (5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)carbamate (4m)

151 MHz, CDCl₃, 23 °C

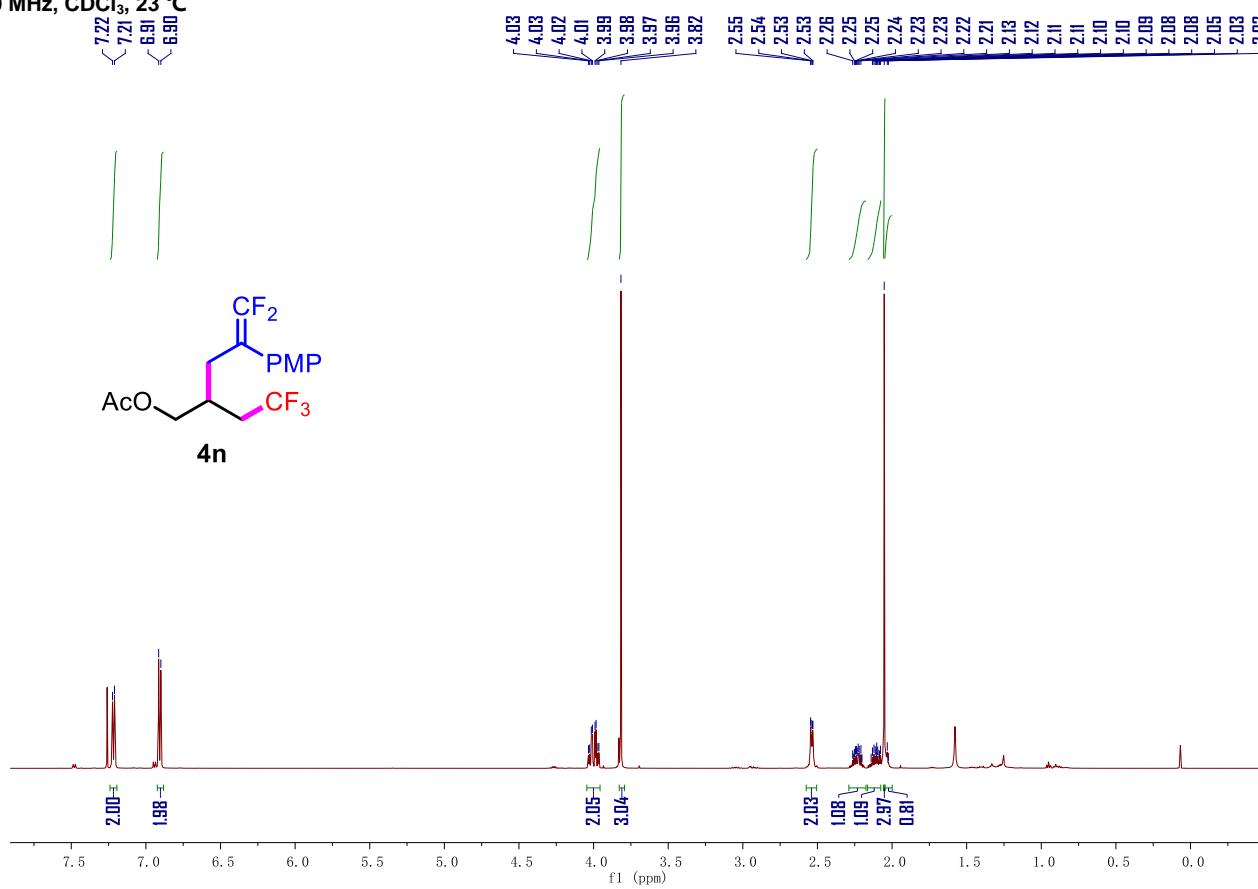


¹⁹F NMR spectrum of tert-butyl (5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)carbamate (4m)
565 MHz, CDCl₃, 23 °C



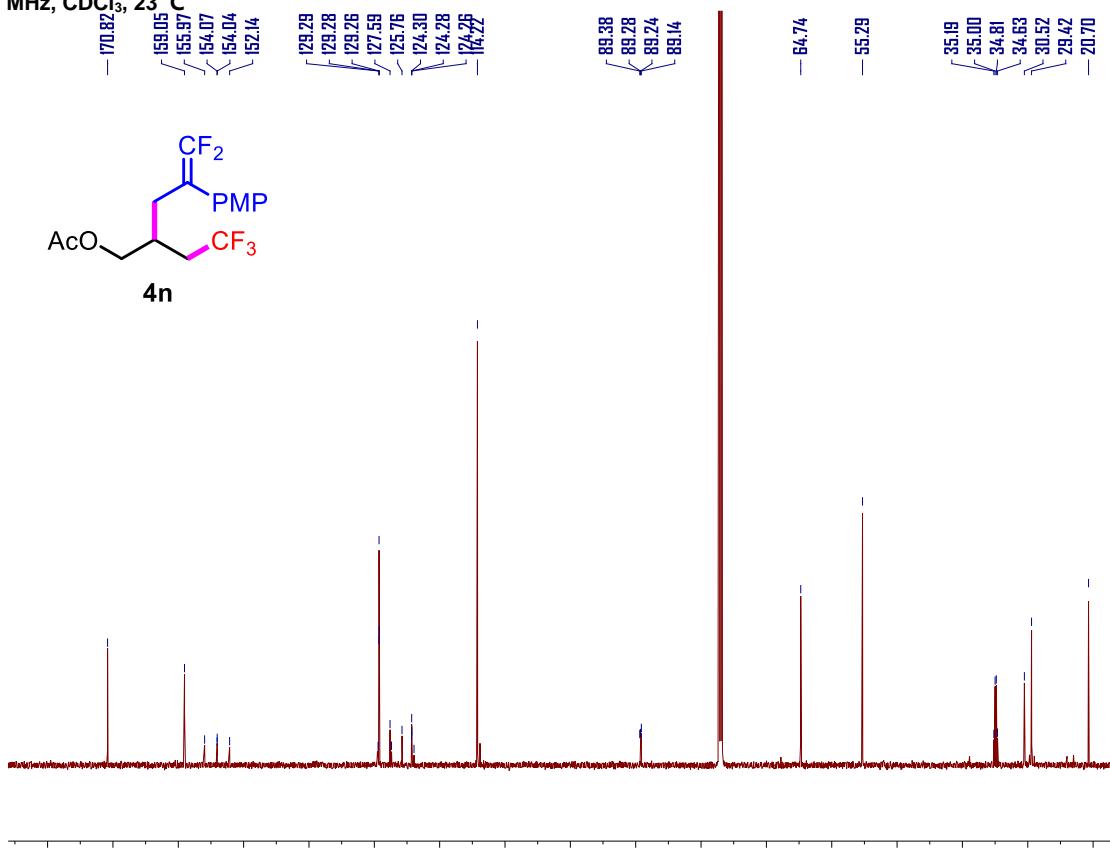
¹H NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl acetate (4n)

600 MHz, CDCl₃, 23 °C



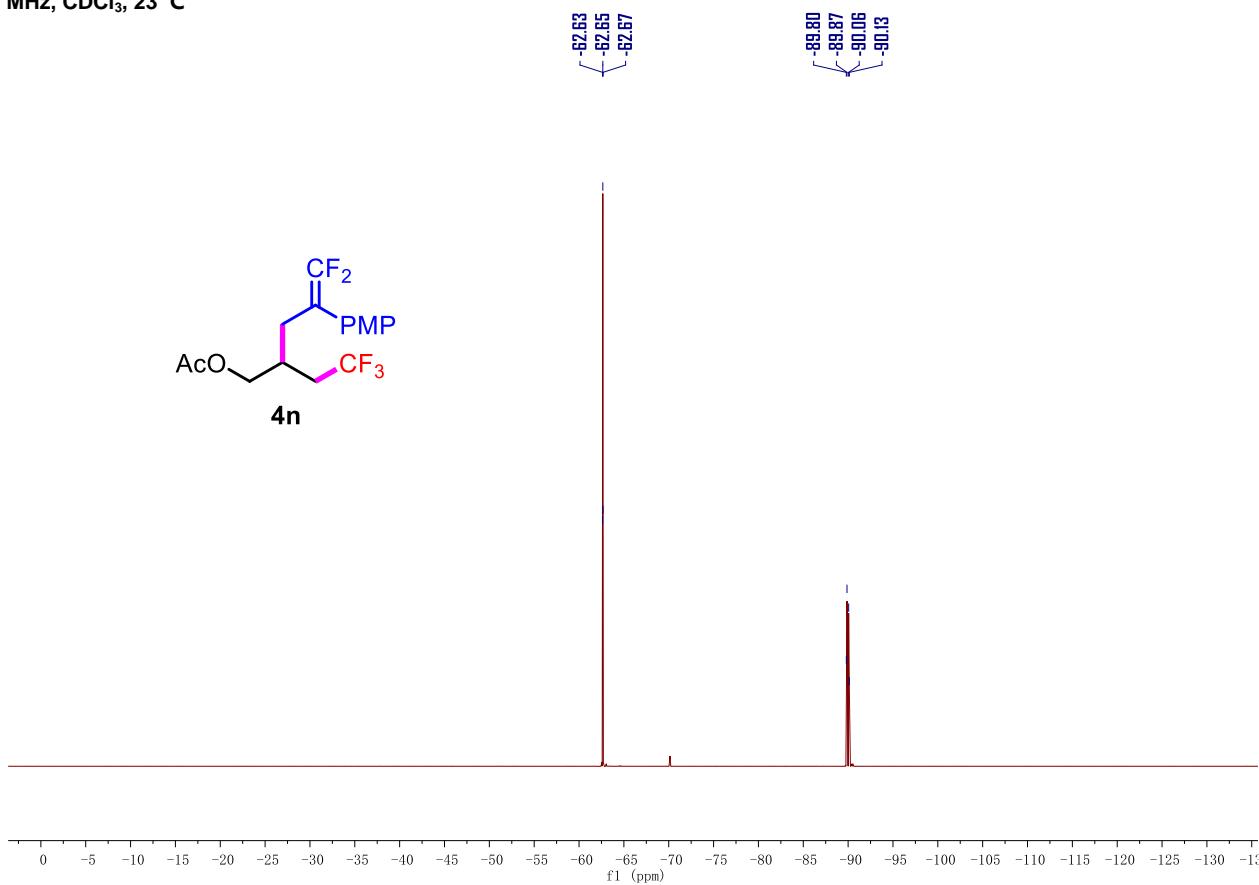
¹³C NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl acetate (4n)

151 MHz, CDCl₃, 23 °C



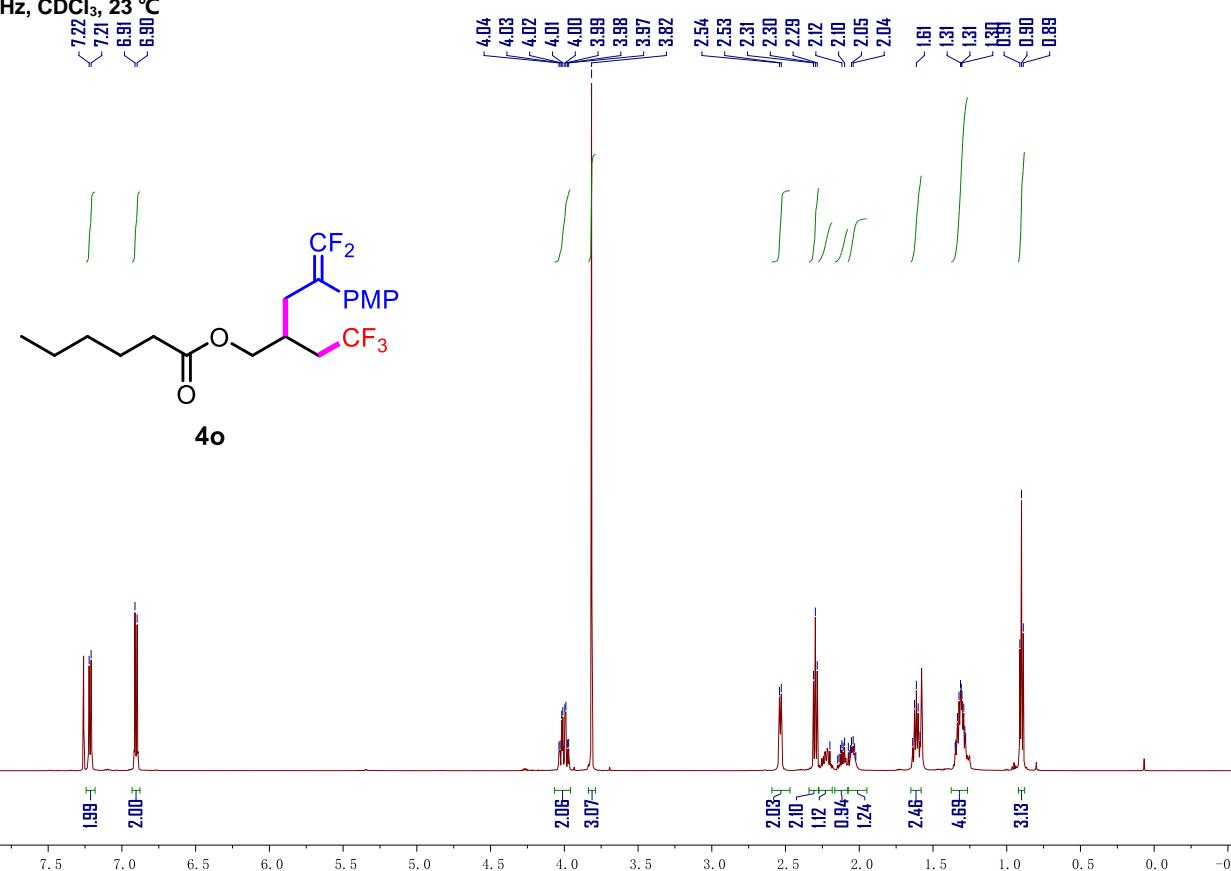
¹⁹F NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl acetate (4n)

565 MHz, CDCl₃, 23 °C



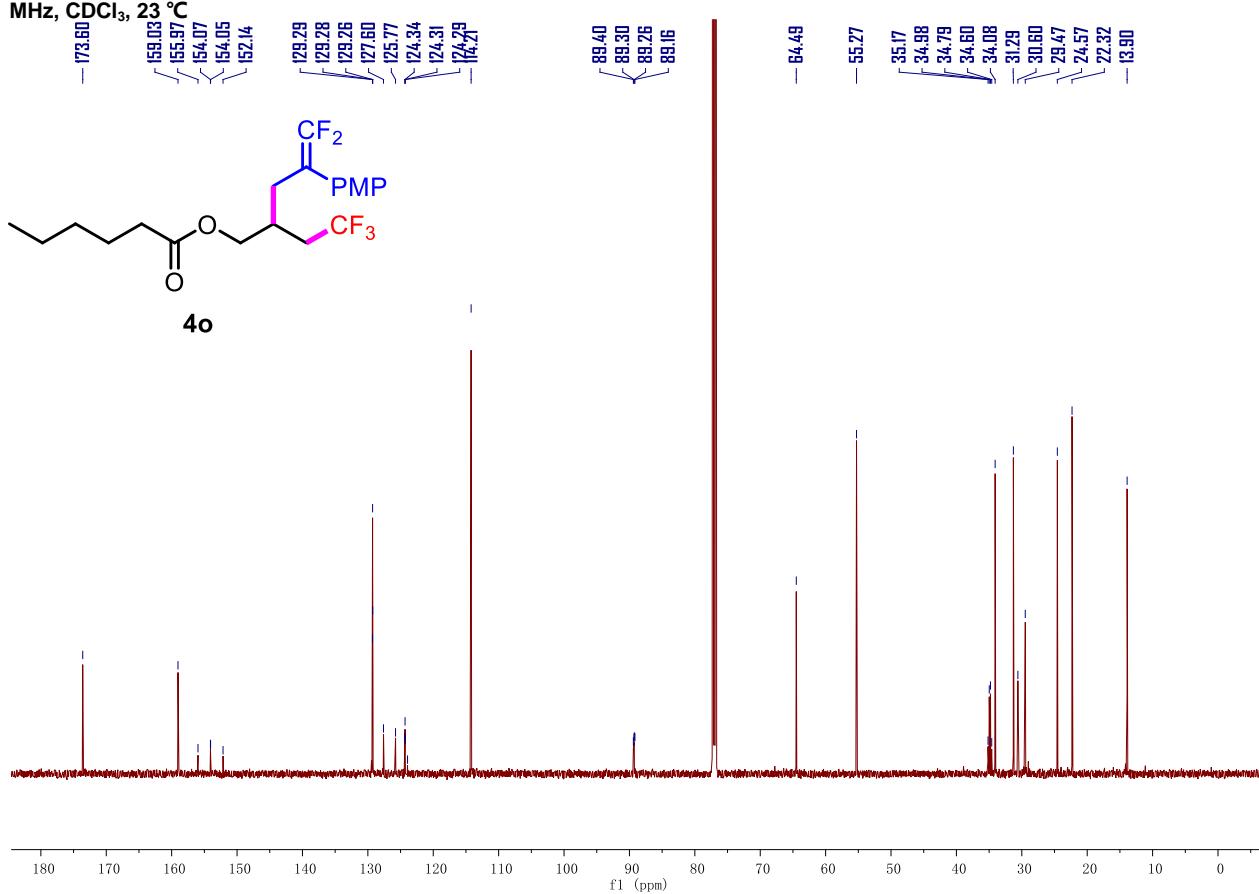
¹H NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl hexanoate (4o)

600 MHz, CDCl₃, 23 °C



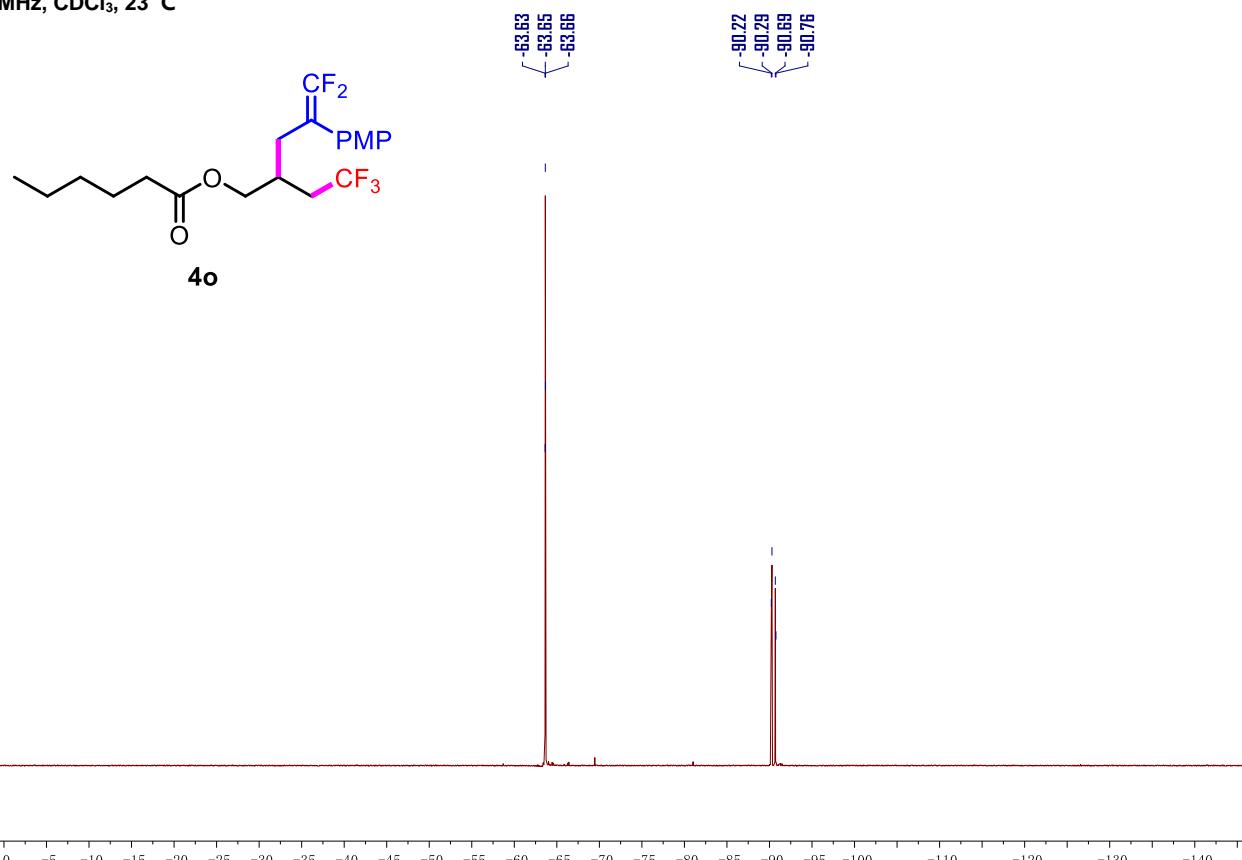
¹³C NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl hexanoate (4o)

151 MHz, CDCl₃, 23 °C



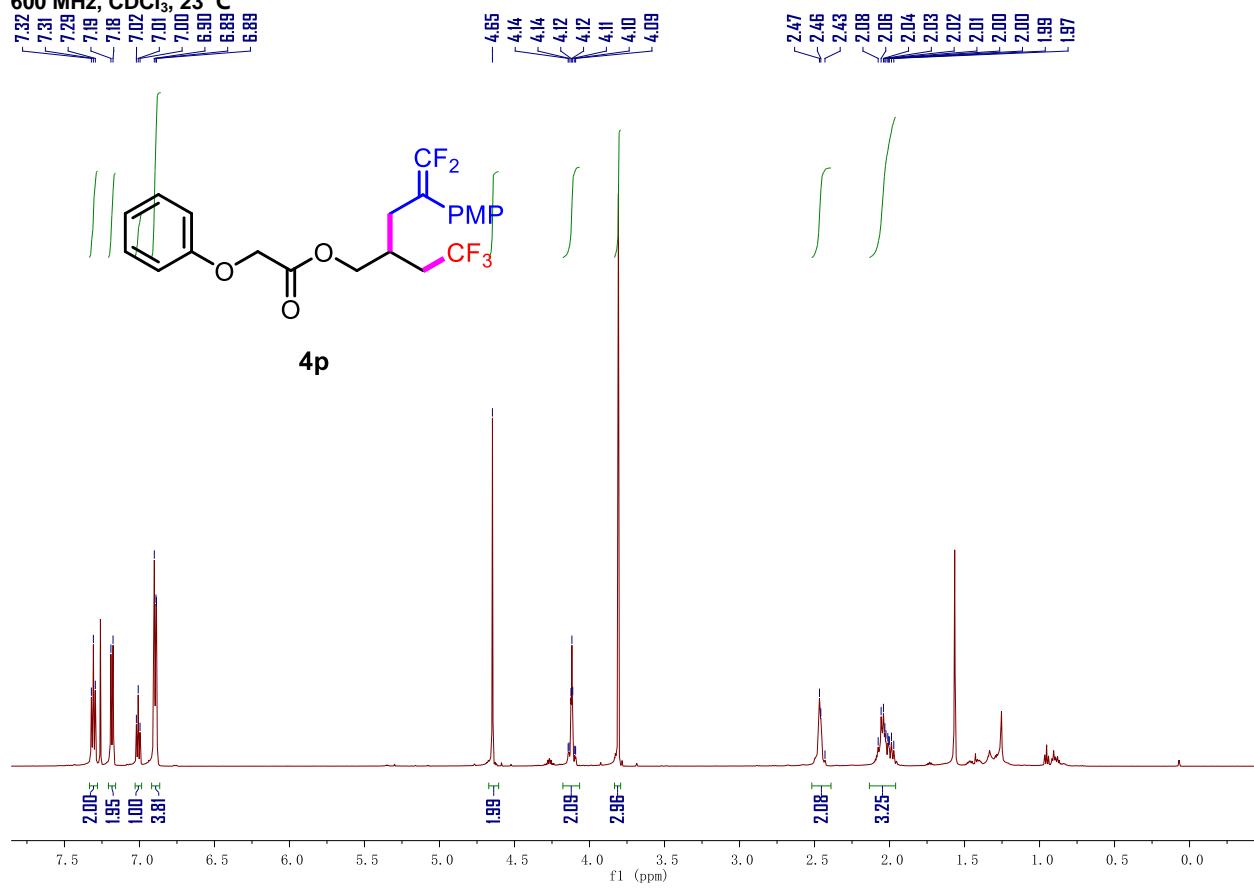
¹⁹F NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl hexanoate (4o)

565 MHz, CDCl₃, 23 °C



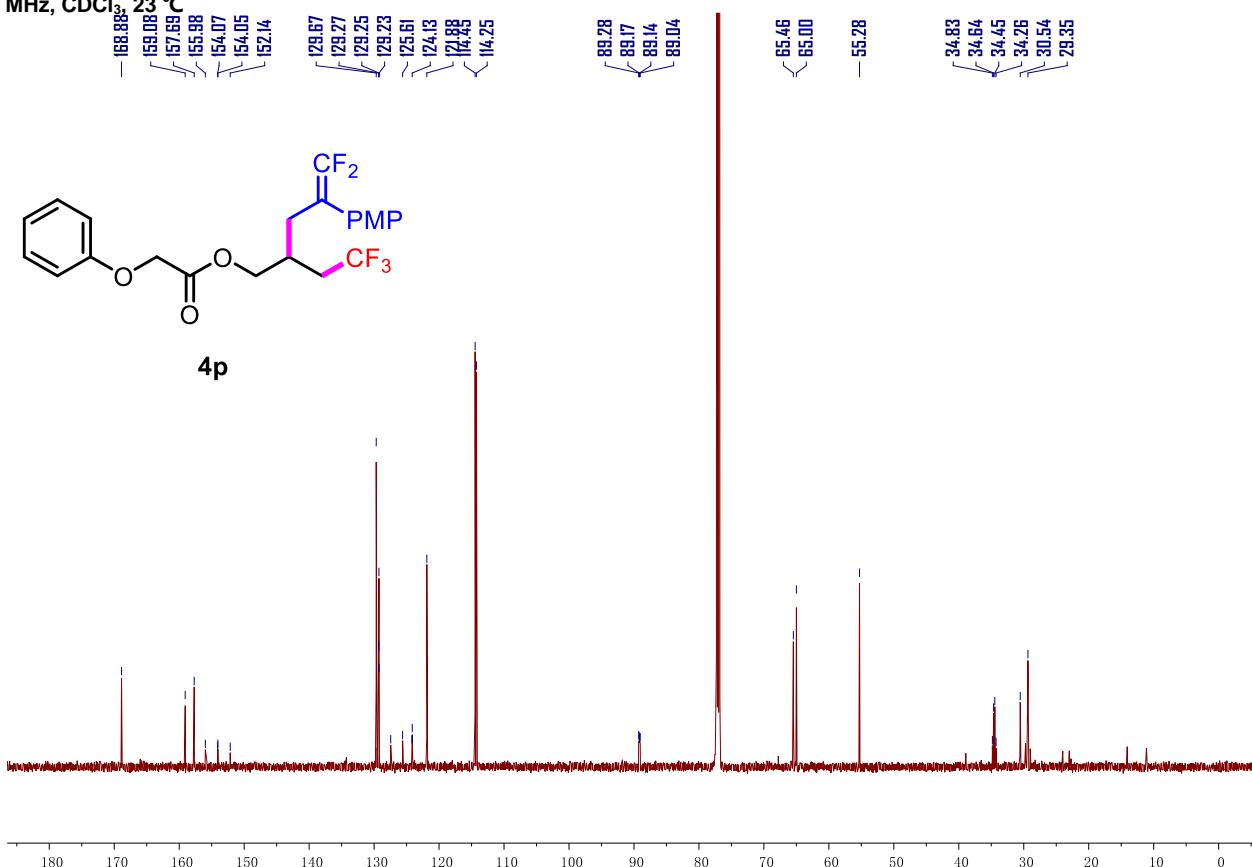
¹H NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl 2-phenoxyacetate (4p)

600 MHz, CDCl₃, 23 °C



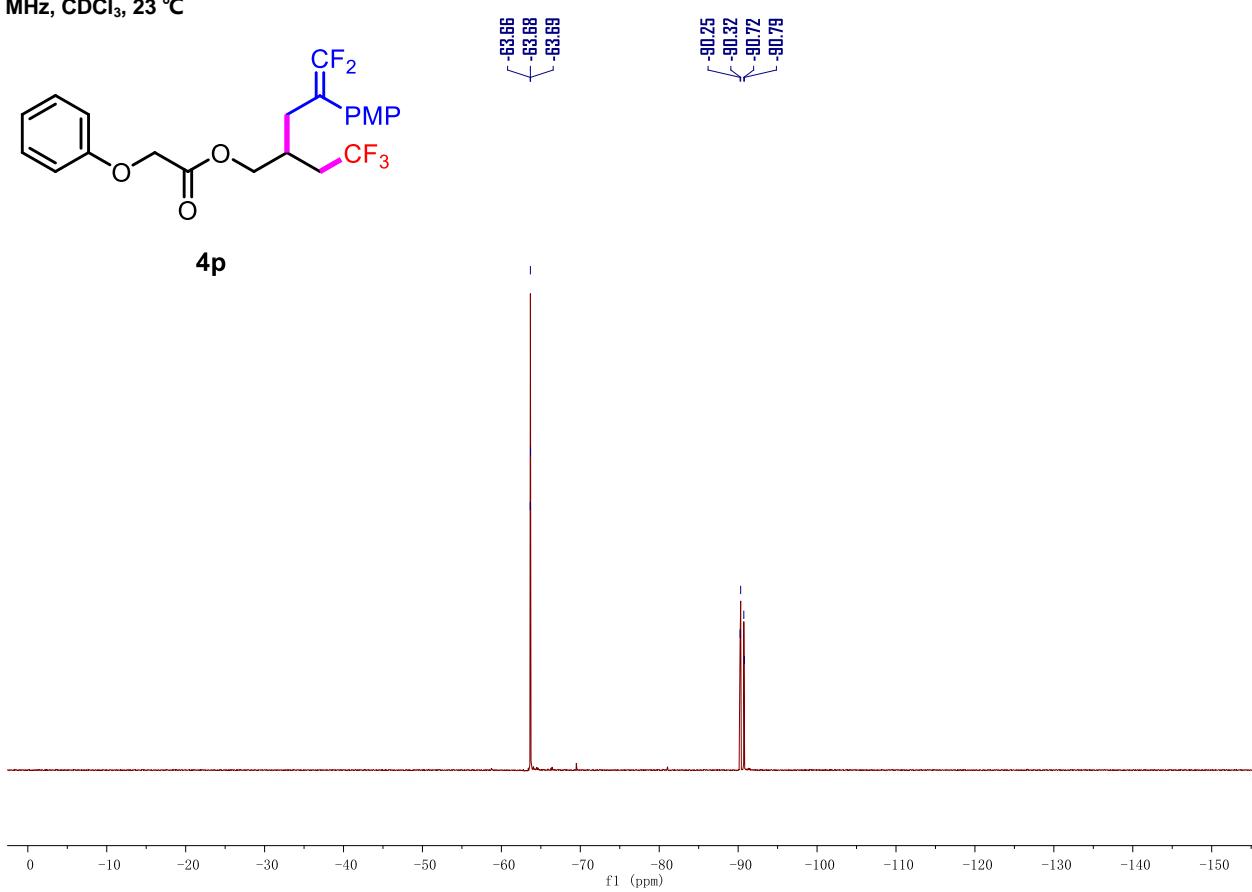
¹³C NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl 2-phenoxyacetate (4p)

151 MHz, CDCl₃, 23 °C



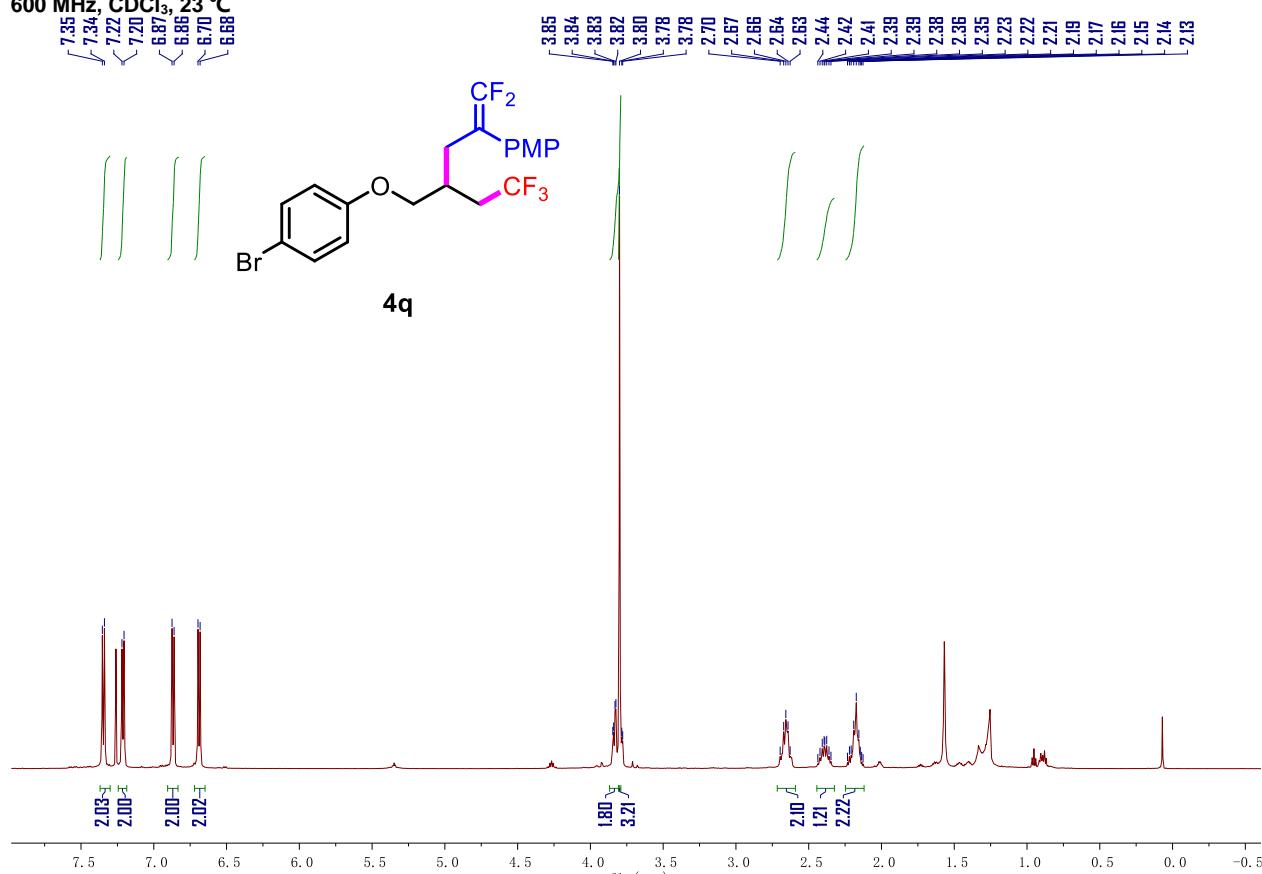
¹⁹F NMR spectrum of 5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl 2-phenoxyacetate (4p)

565 MHz, CDCl₃, 23 °C



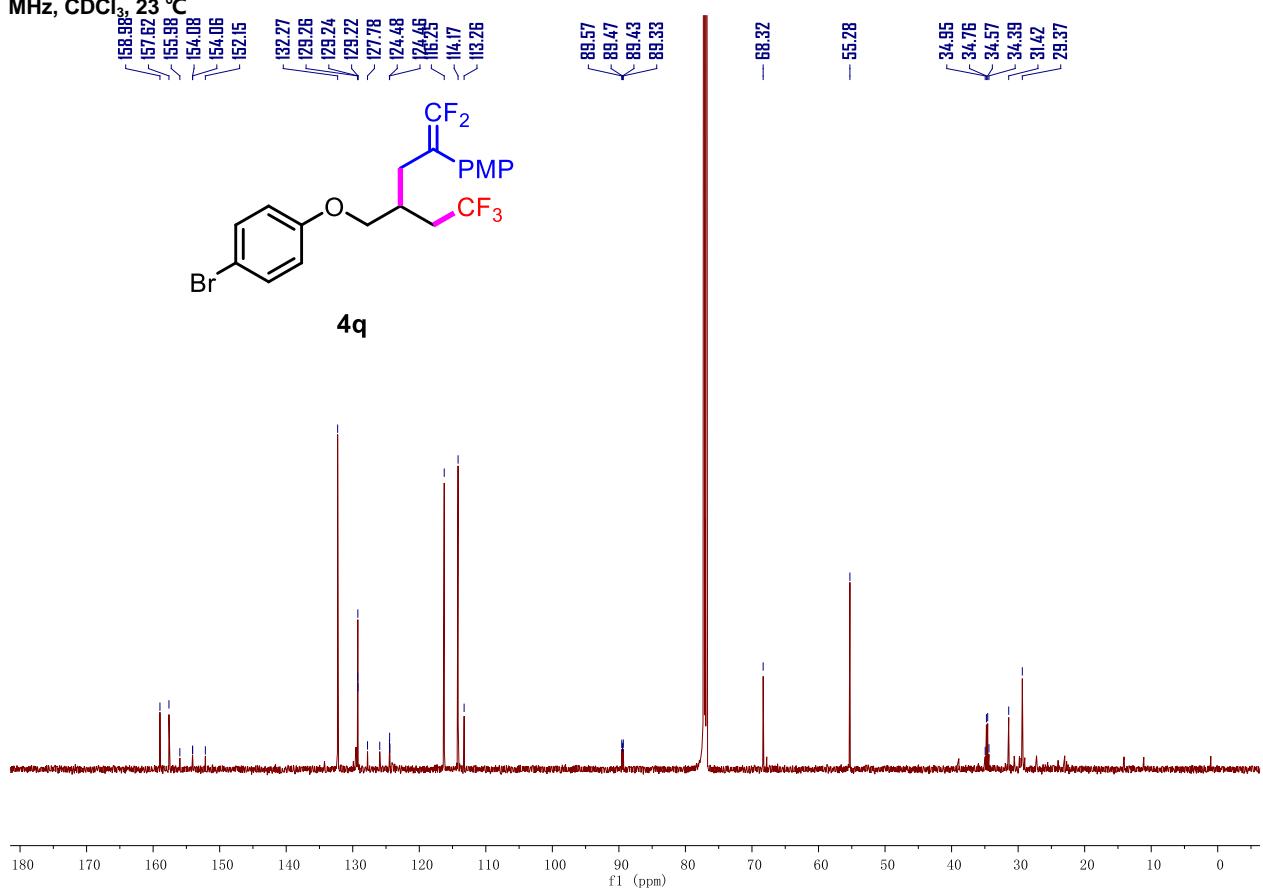
¹H NMR spectrum of 1-bromo-4-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)benzene (4q)

600 MHz, CDCl₃, 23 °C

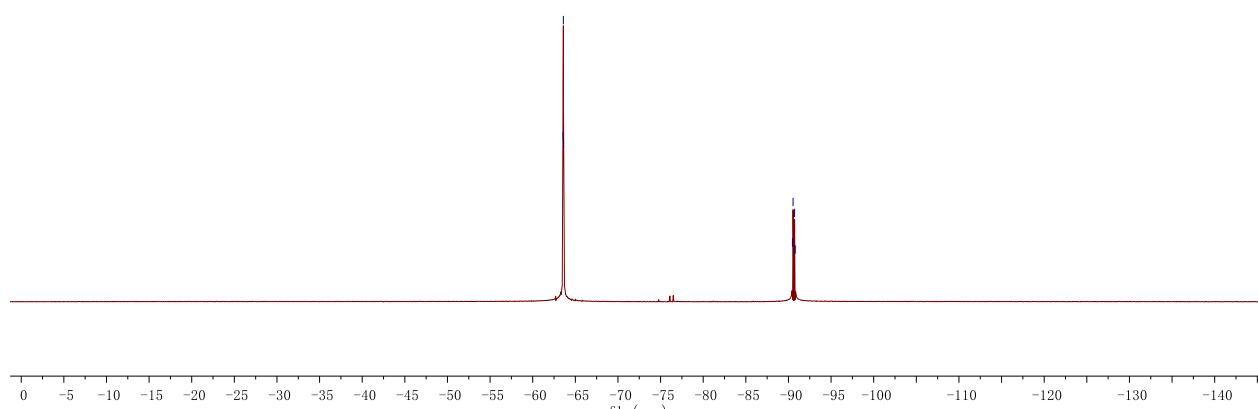
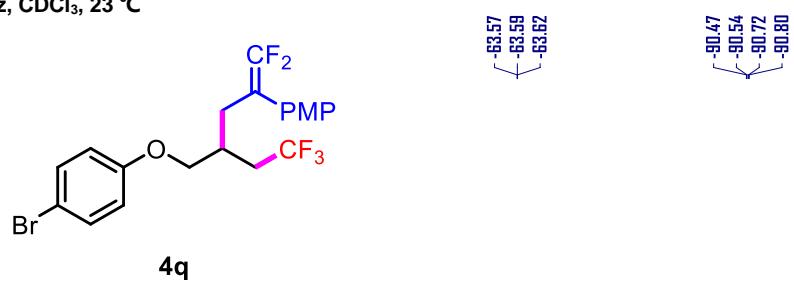


¹³C NMR spectrum of 1-bromo-4-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)benzene (4q)

151 MHz, CDCl₃, 23 °C

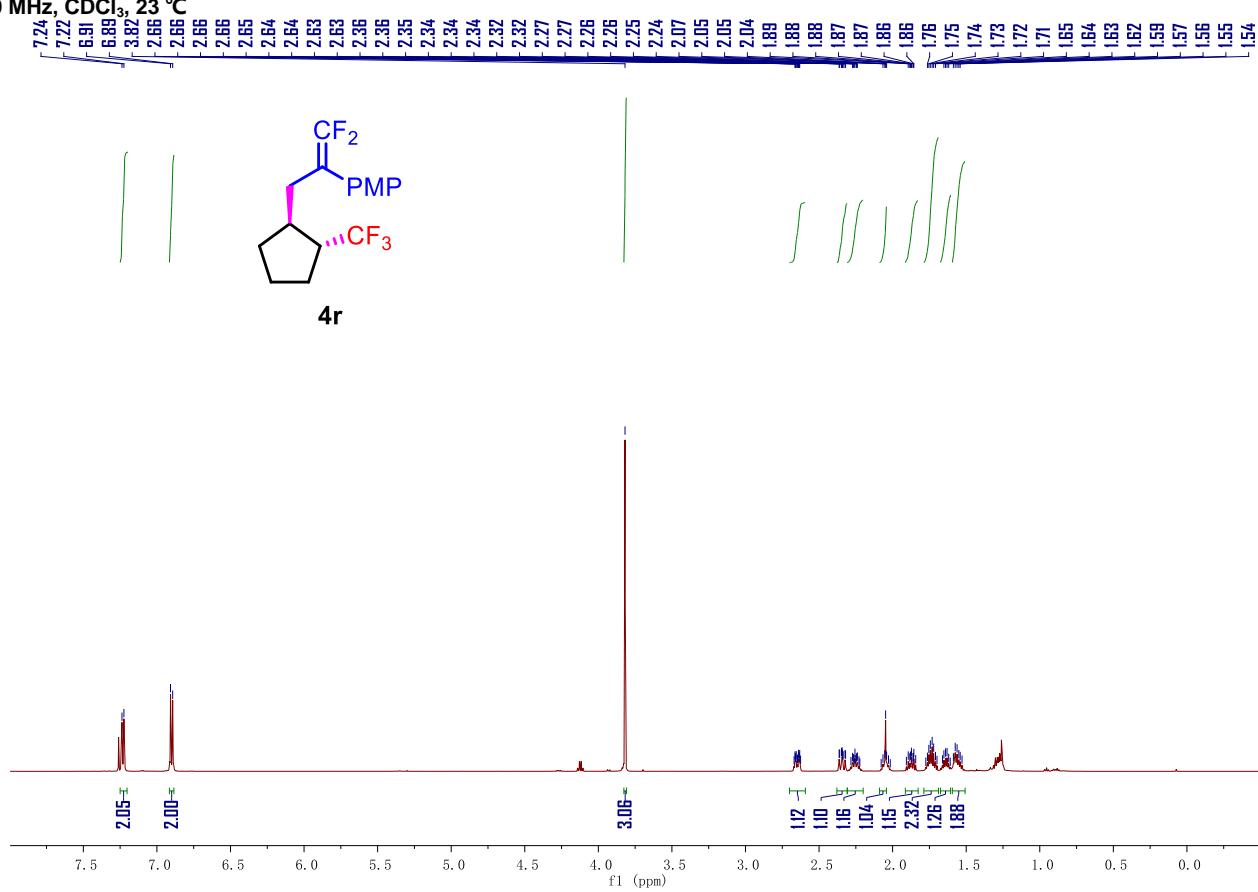


¹⁹F NMR spectrum of 1-bromo-4-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)benzene (4q)
565 MHz, CDCl₃, 23 °C



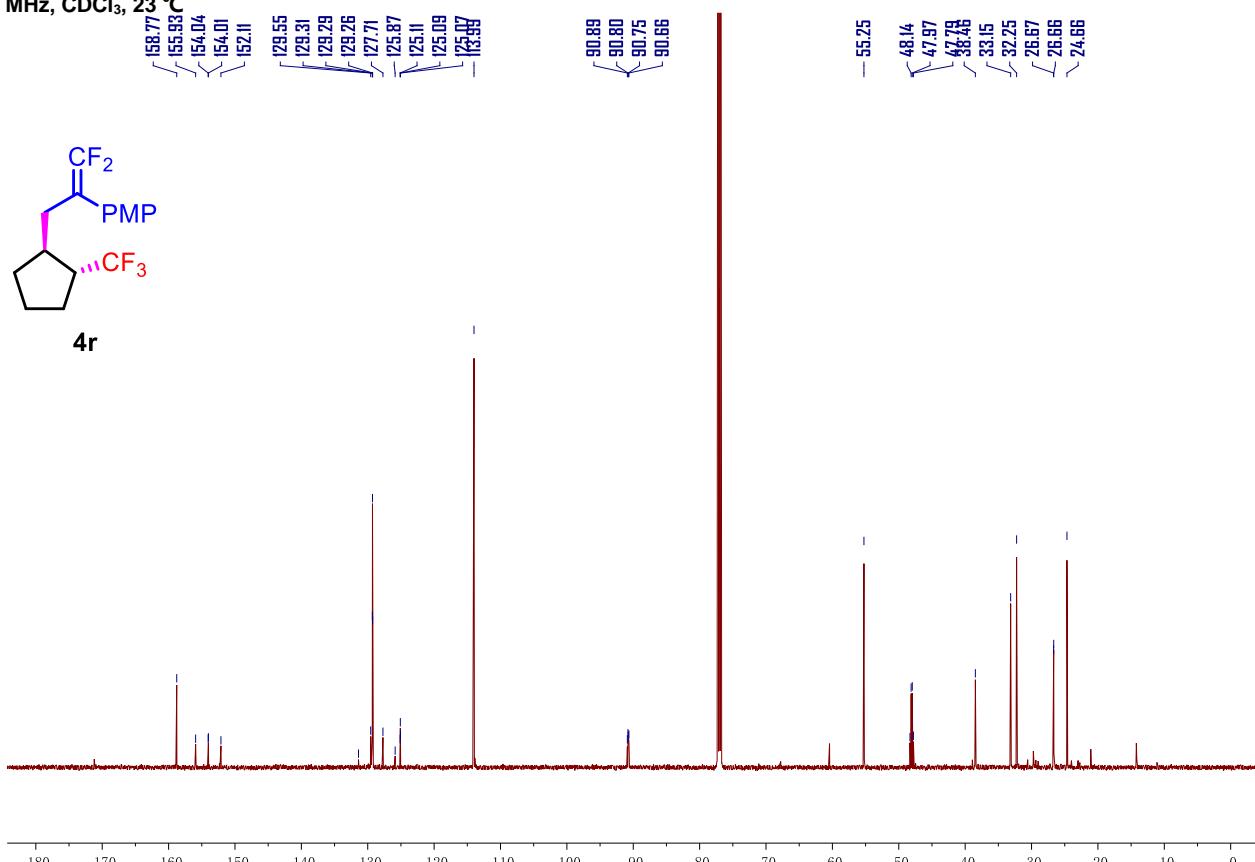
¹H NMR spectrum of 1-(1,1-difluoro-3-(2-(trifluoromethyl)cyclopentyl)prop-1-en-2-yl)-4-methoxybenzene (4r)

600 MHz, CDCl₃, 23 °C



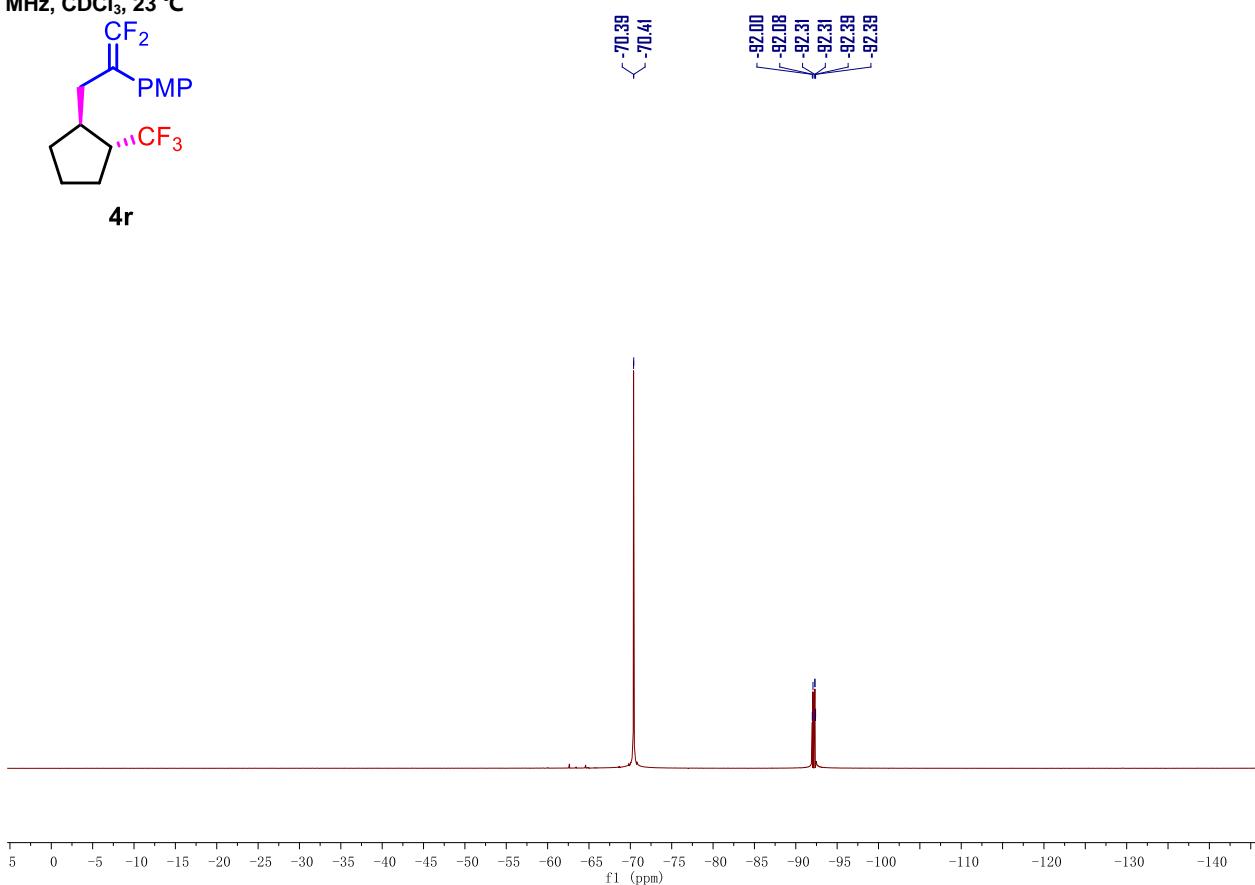
¹³C NMR spectrum of 1-(1,1-difluoro-3-(2-(trifluoromethyl)cyclopentyl)prop-1-en-2-yl)-4-methoxybenzene (4r)

151 MHz, CDCl₃, 23 °C

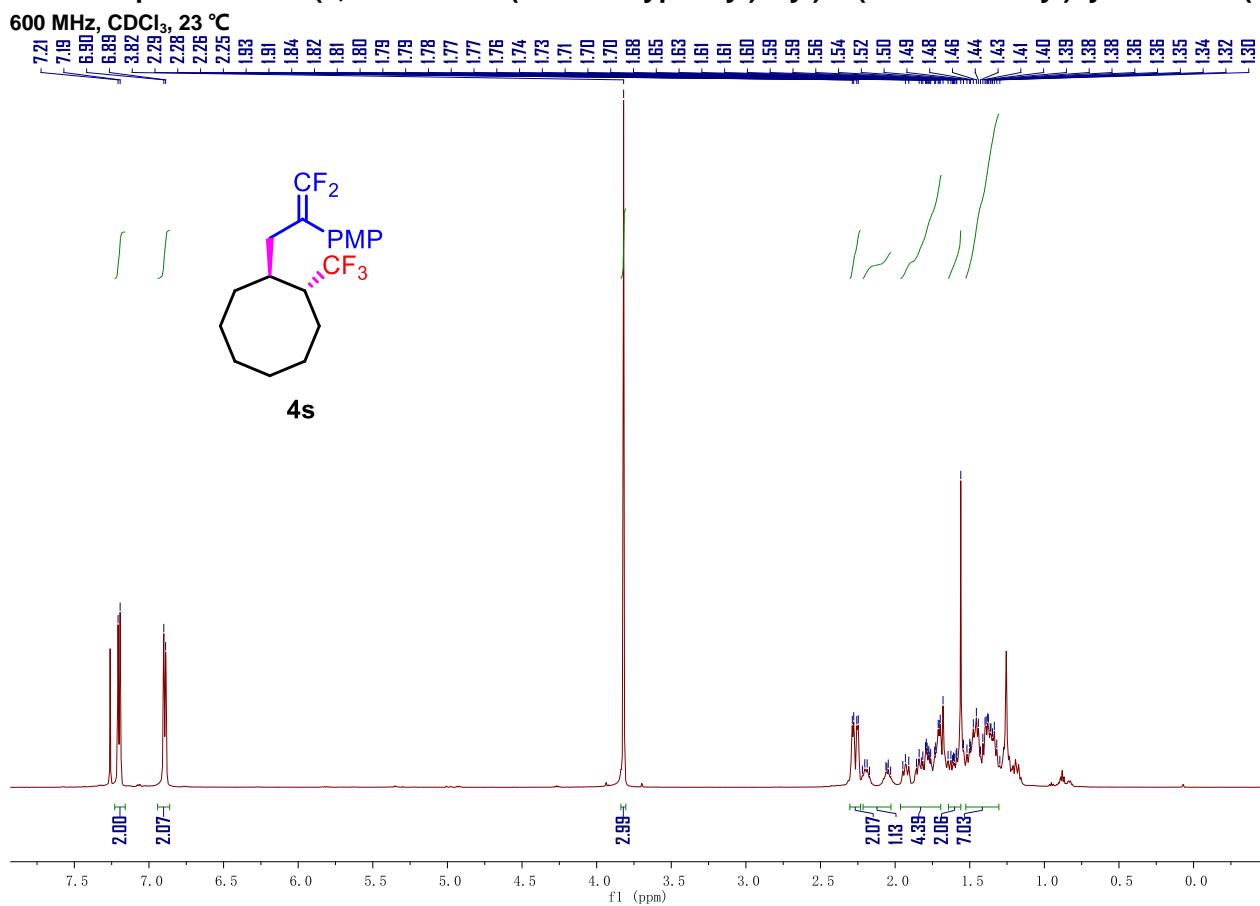


¹⁹F NMR spectrum of 1-(1,1-difluoro-3-(2-(trifluoromethyl)cyclopentyl)prop-1-en-2-yl)-4-methoxybenzene (4r)

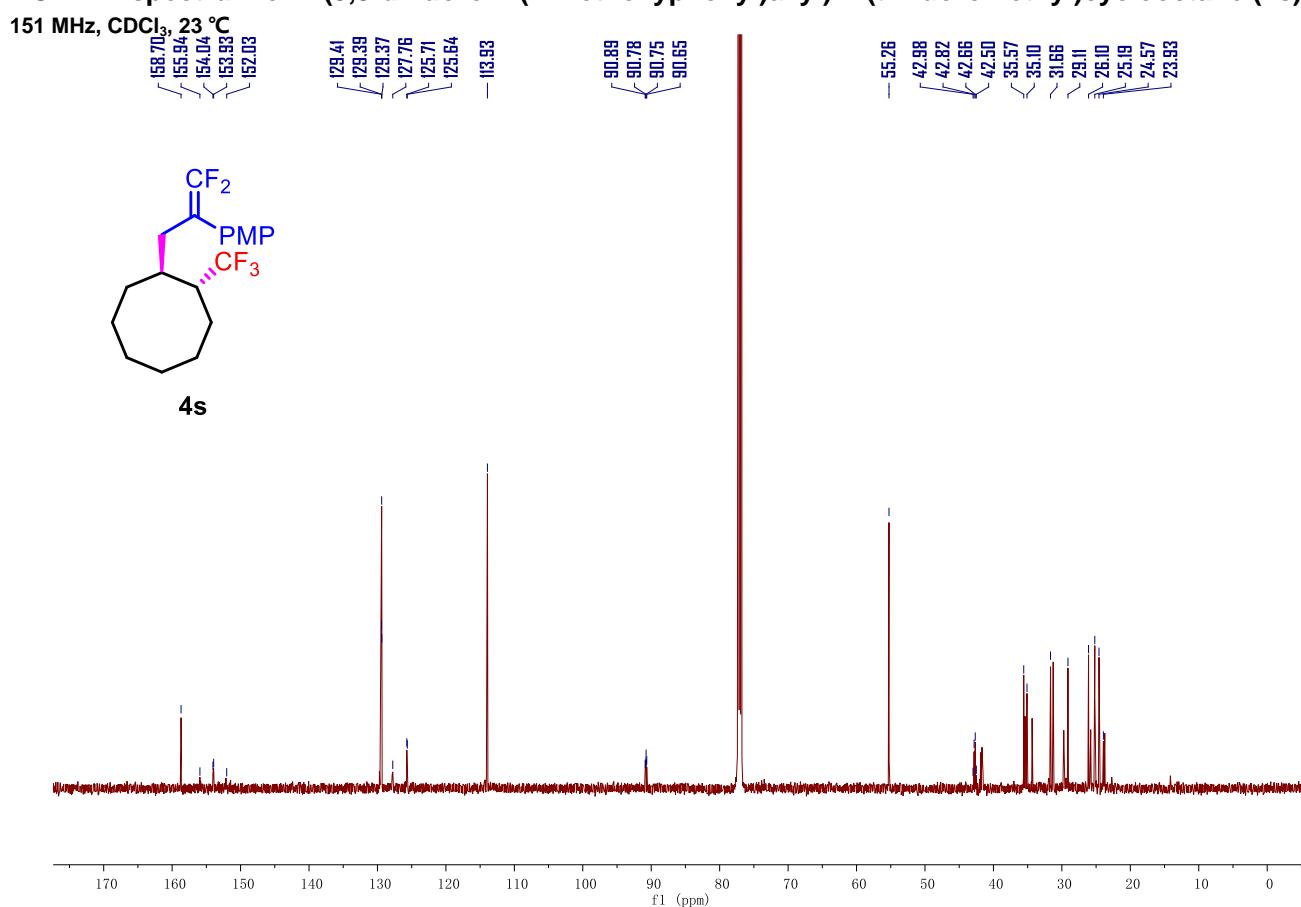
565 MHz, CDCl₃, 23 °C



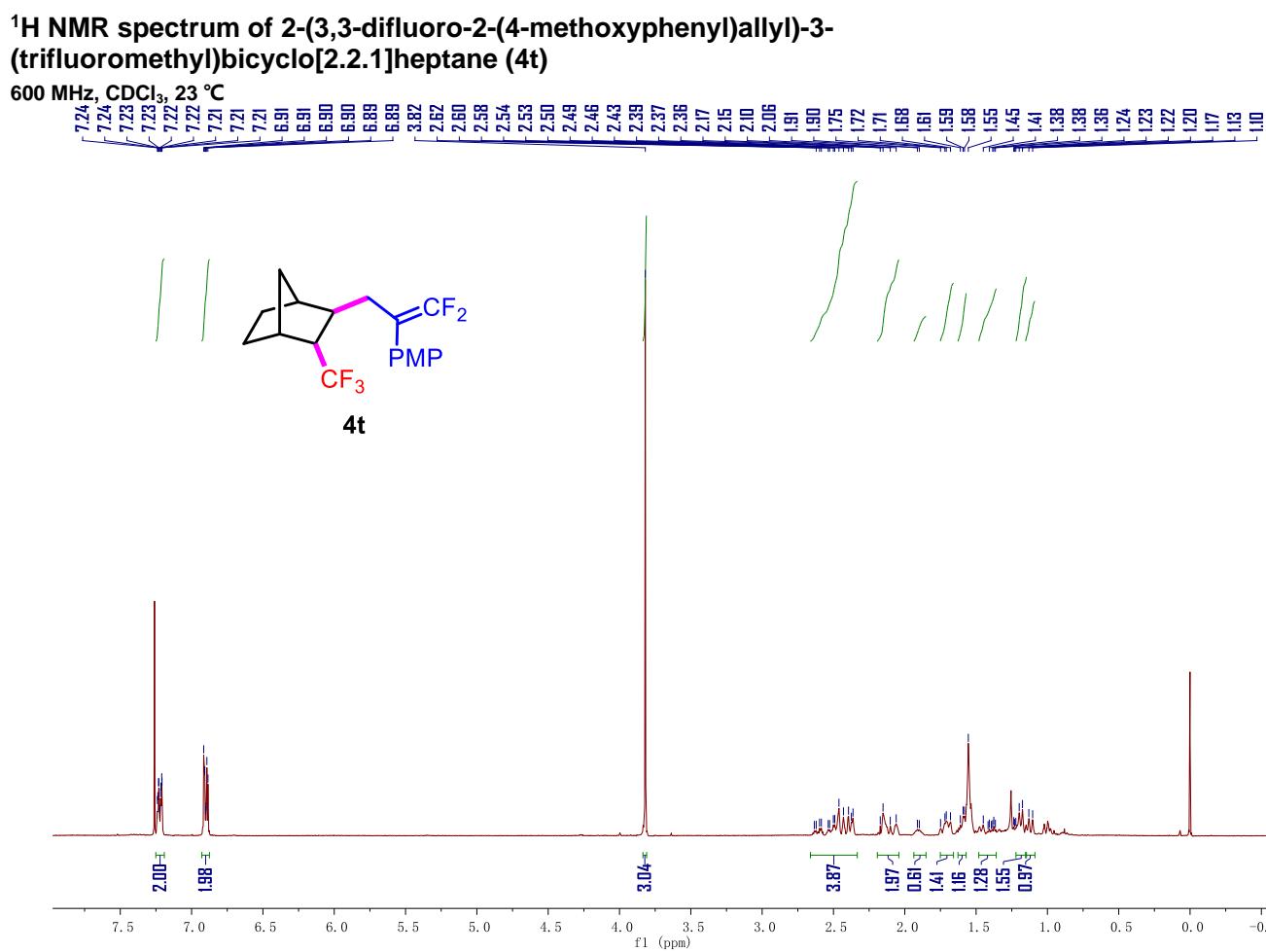
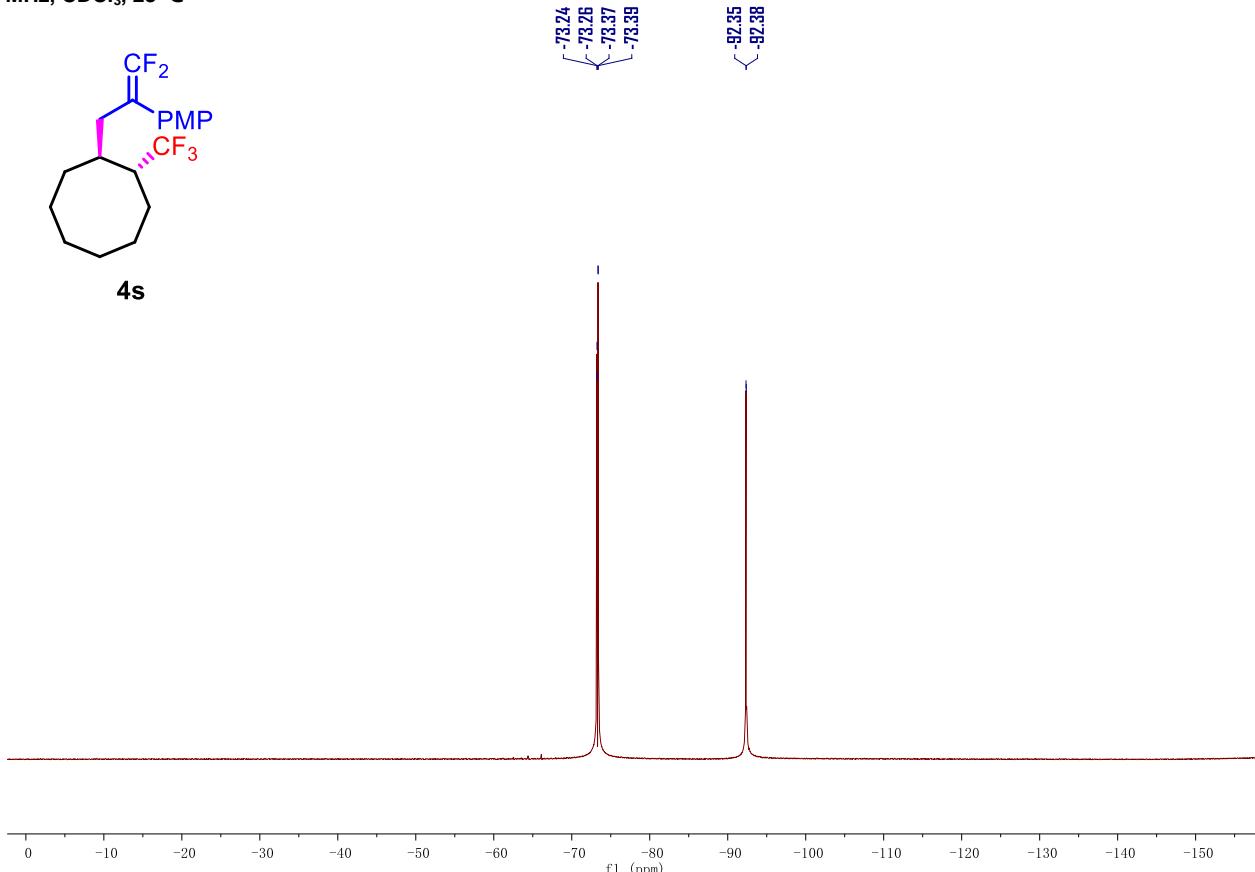
¹H NMR spectrum of 1-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-2-(trifluoromethyl)cyclooctane (4s)



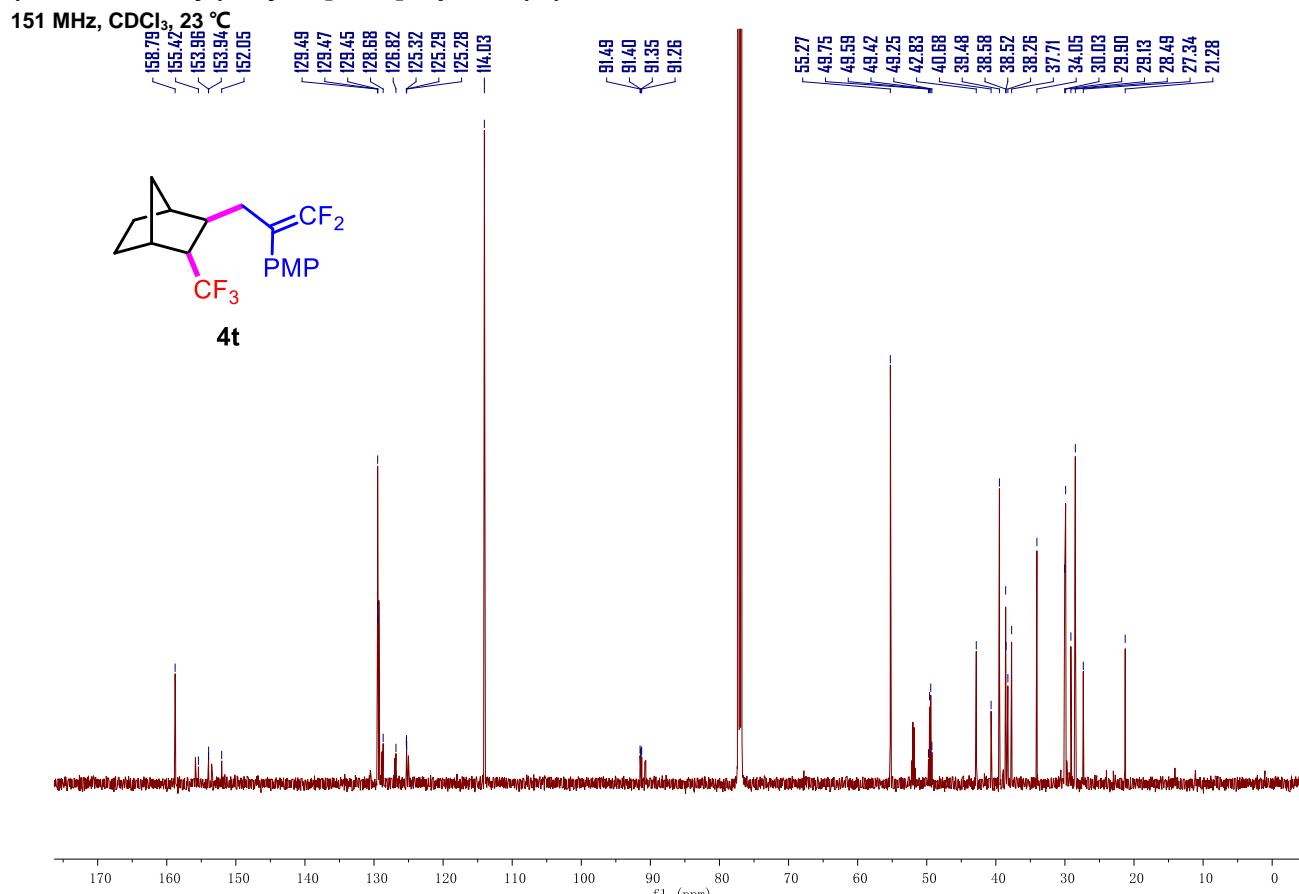
¹³C NMR spectrum of 1-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-2-(trifluoromethyl)cyclooctane (4s)



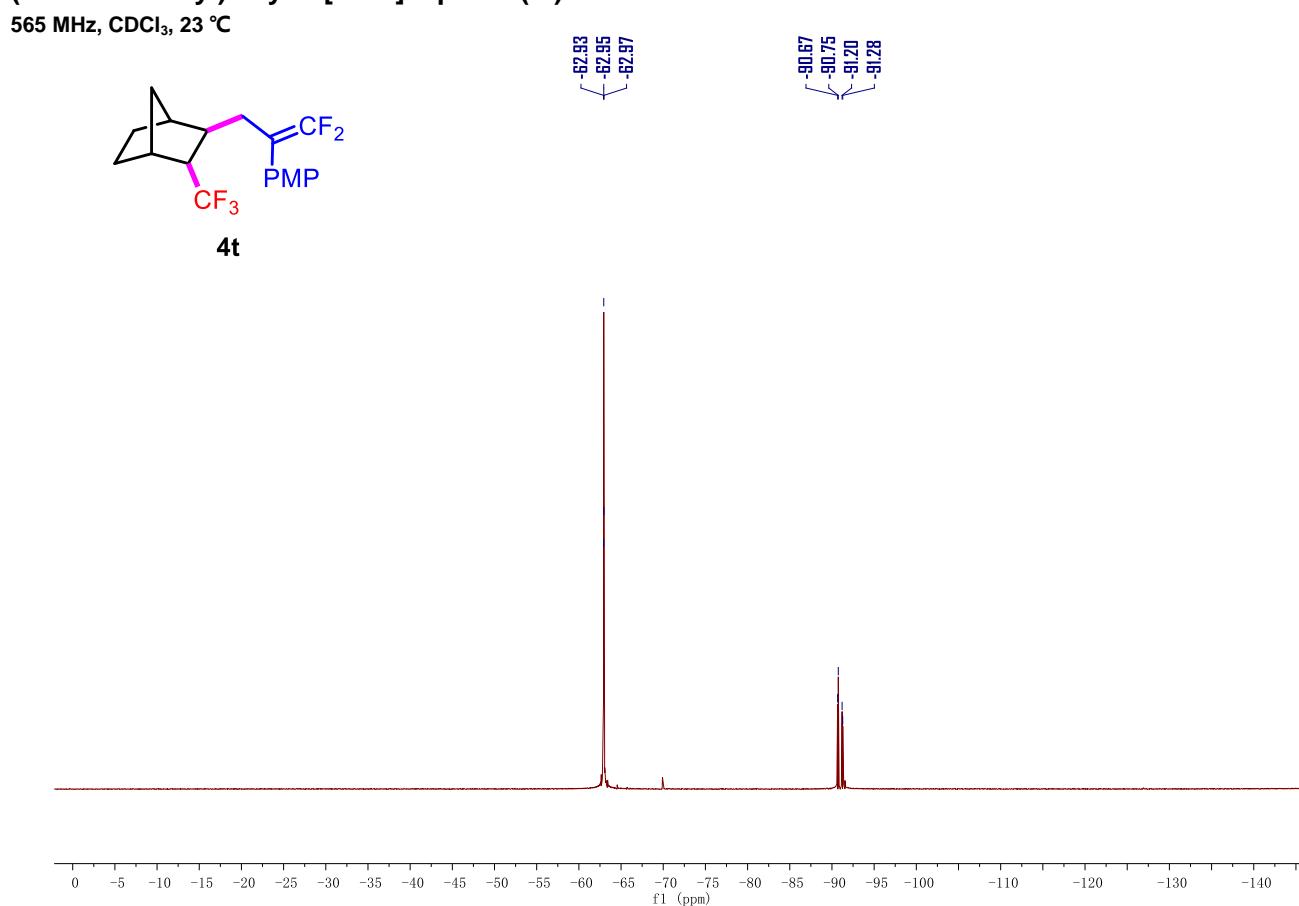
¹⁹F NMR spectrum of 1-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-2-(trifluoromethyl)cyclooctane (4s)
565 MHz, CDCl₃, 23 °C



¹³C NMR spectrum of 2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)bicyclo[2.2.1]heptane (4t)

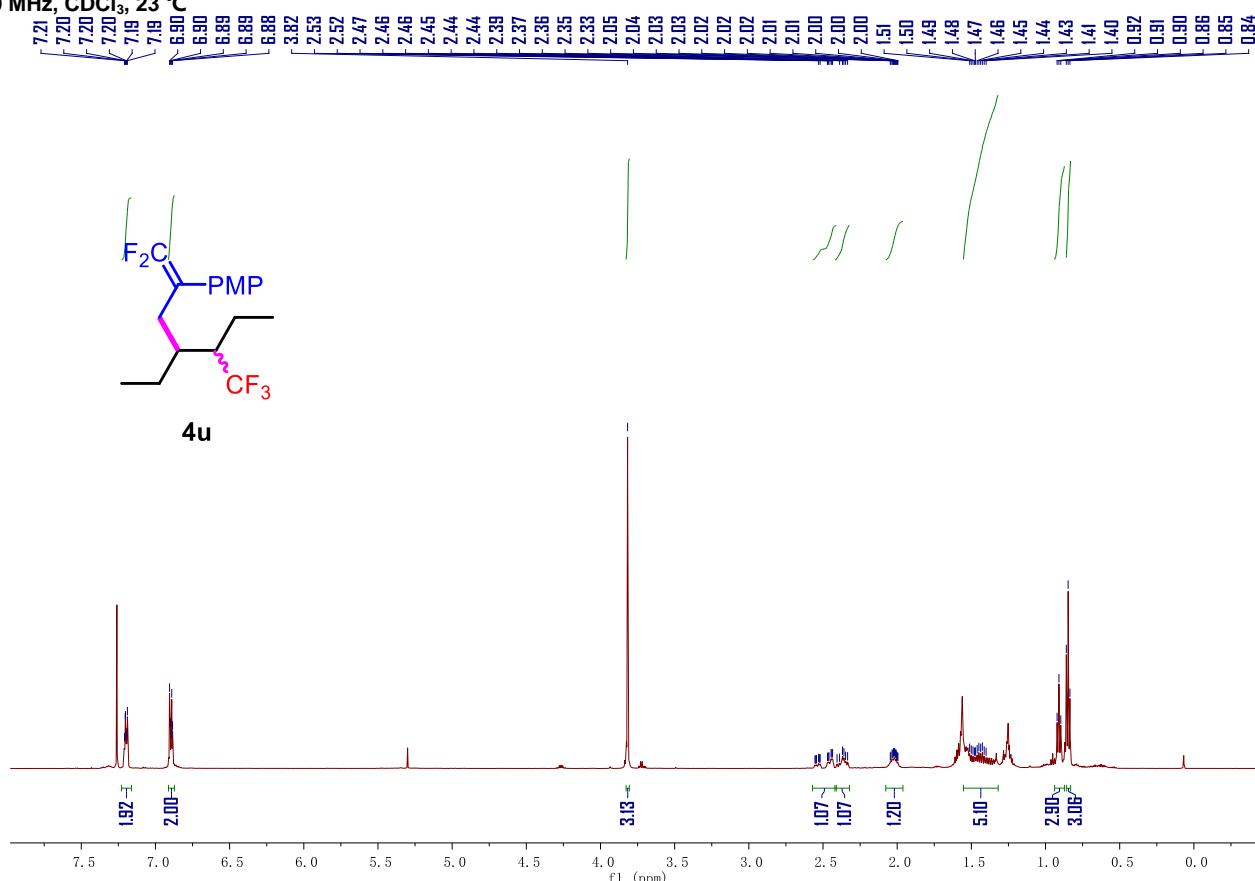


¹⁹F NMR spectrum of 2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)bicyclo[2.2.1]heptane (4t)



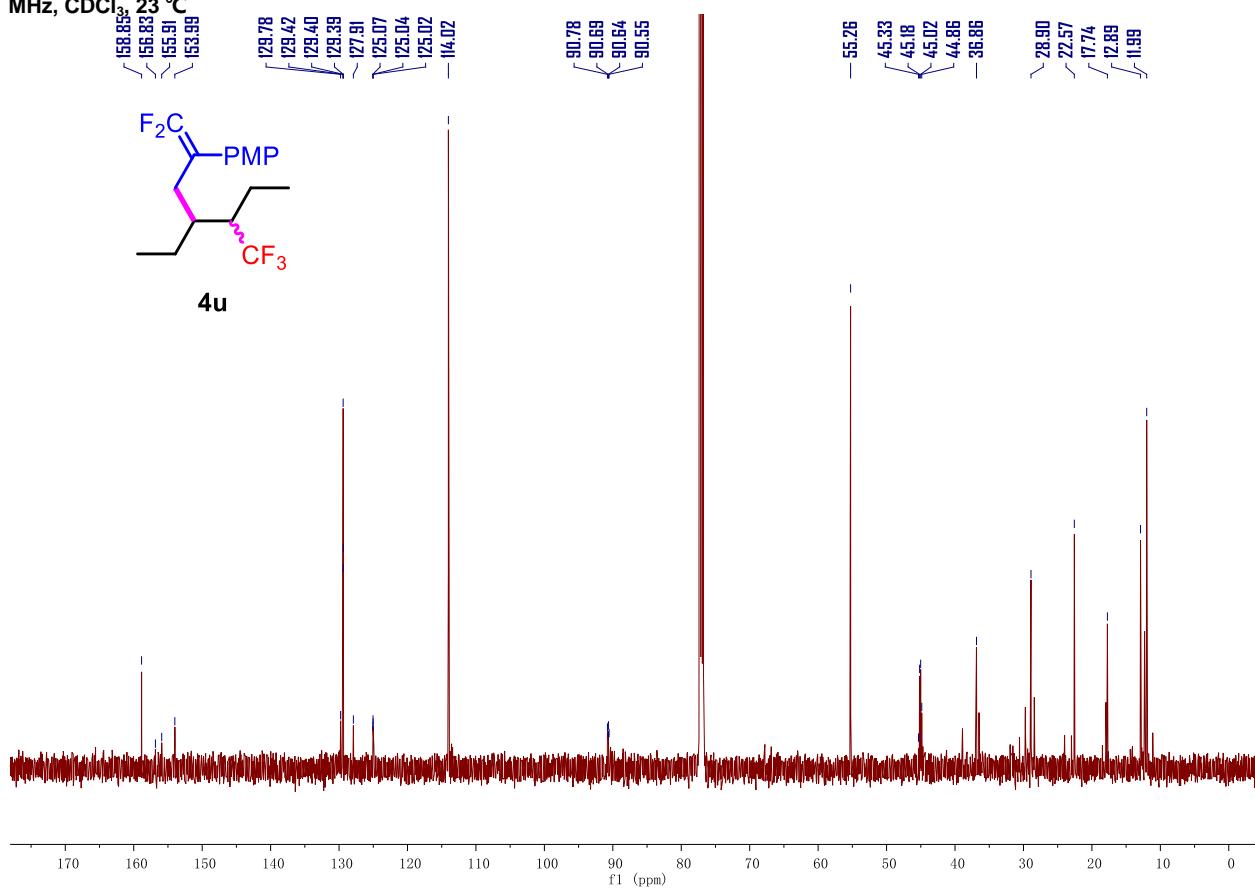
¹H NMR spectrum of 1-(4-ethyl-1,1-difluoro-5-(trifluoromethyl)hept-1-en-2-yl)-4-methoxybenzene (4u)

600 MHz, CDCl₃, 23 °C

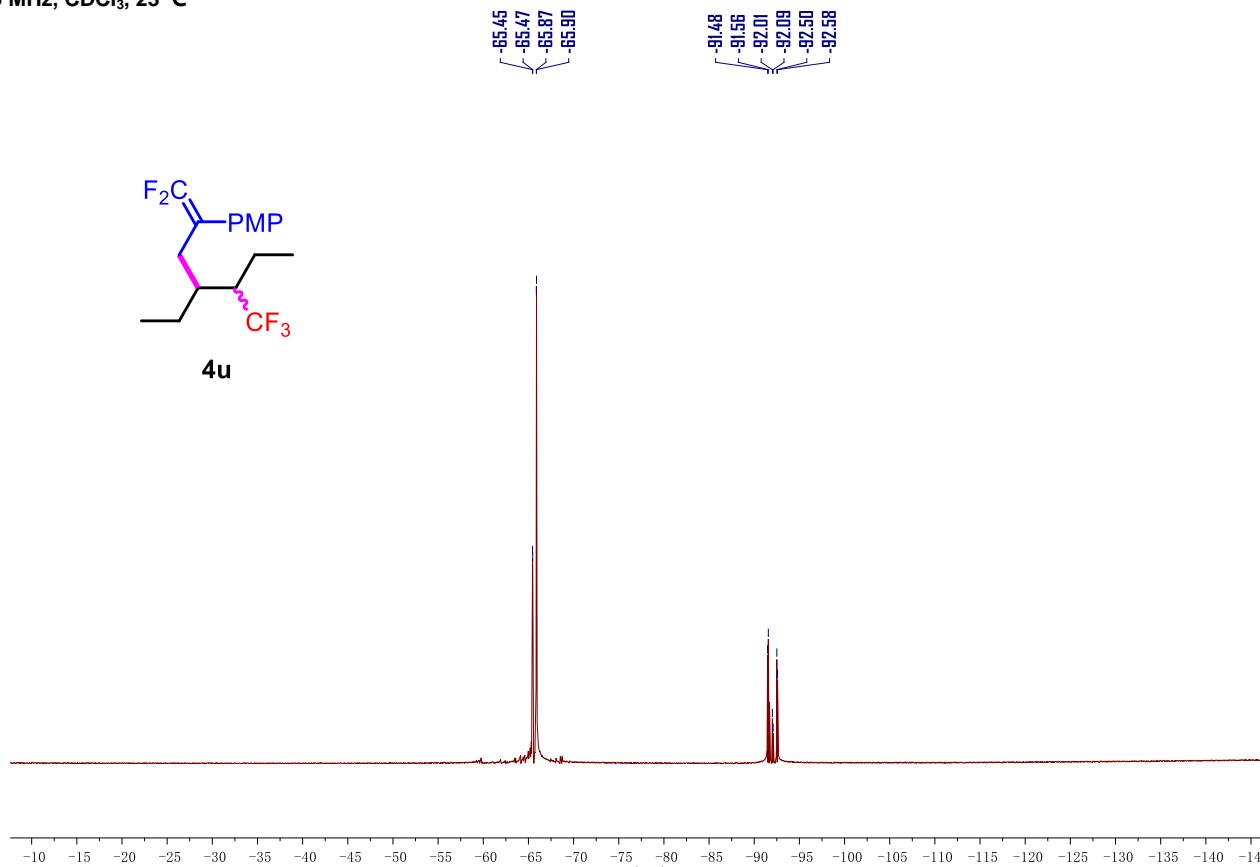


¹³C NMR spectrum of 1-(4-ethyl-1,1-difluoro-5-(trifluoromethyl)hept-1-en-2-yl)-4-methoxybenzene (4u)

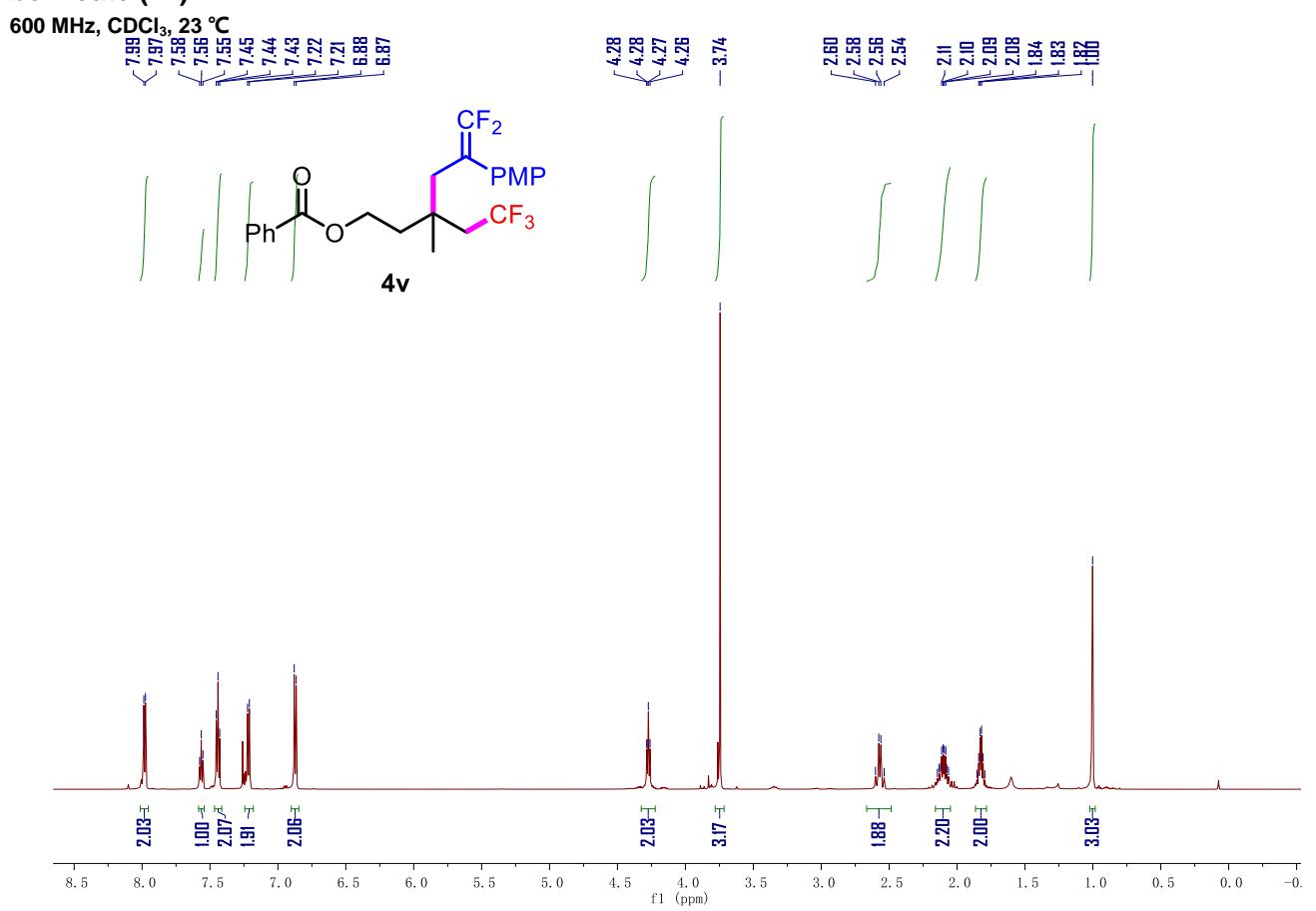
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(4-ethyl-1,1-difluoro-5-(trifluoromethyl)hept-1-en-2-yl)-4-methoxybenzene (4u)
565 MHz, CDCl₃, 23 °C

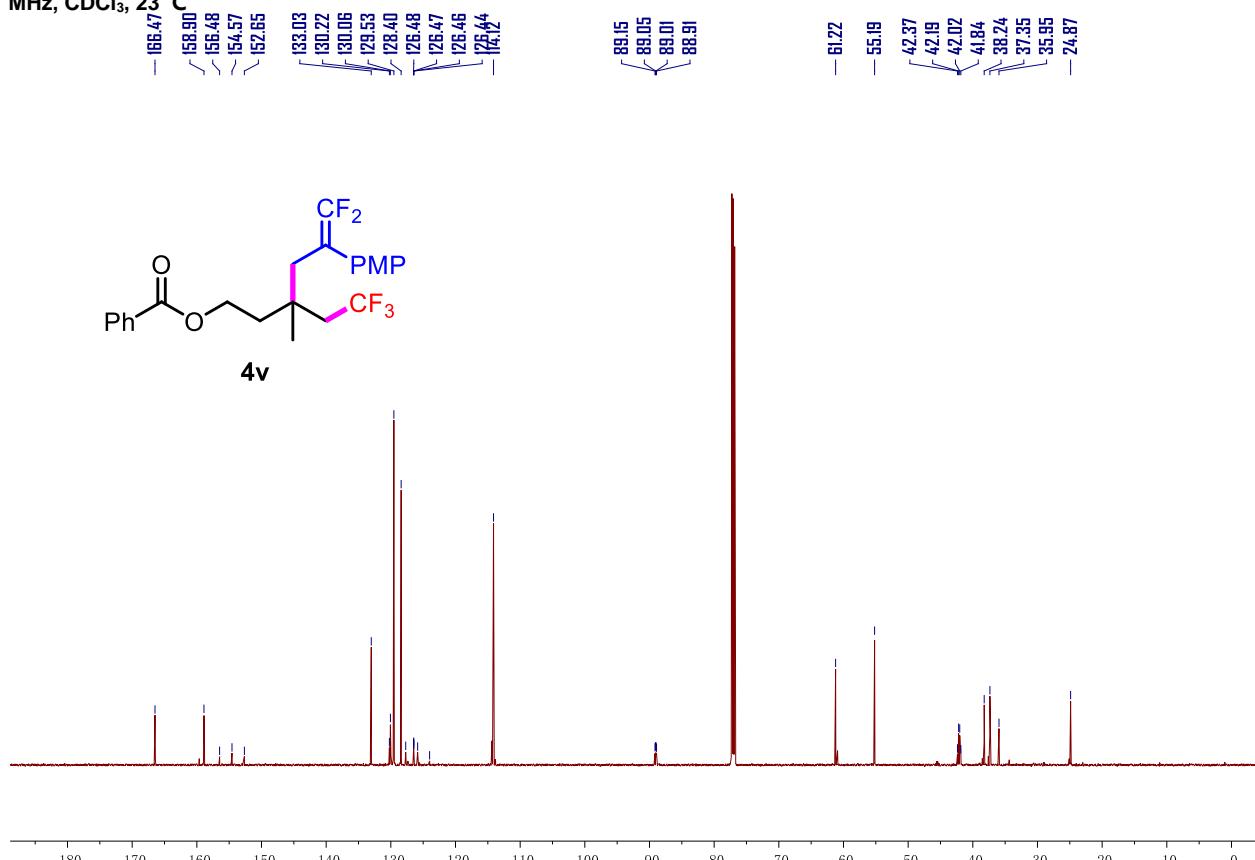


¹H NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-methyl-3-(2,2,2-trifluoroethyl)hex-5-en-1-yl benzoate (4v)



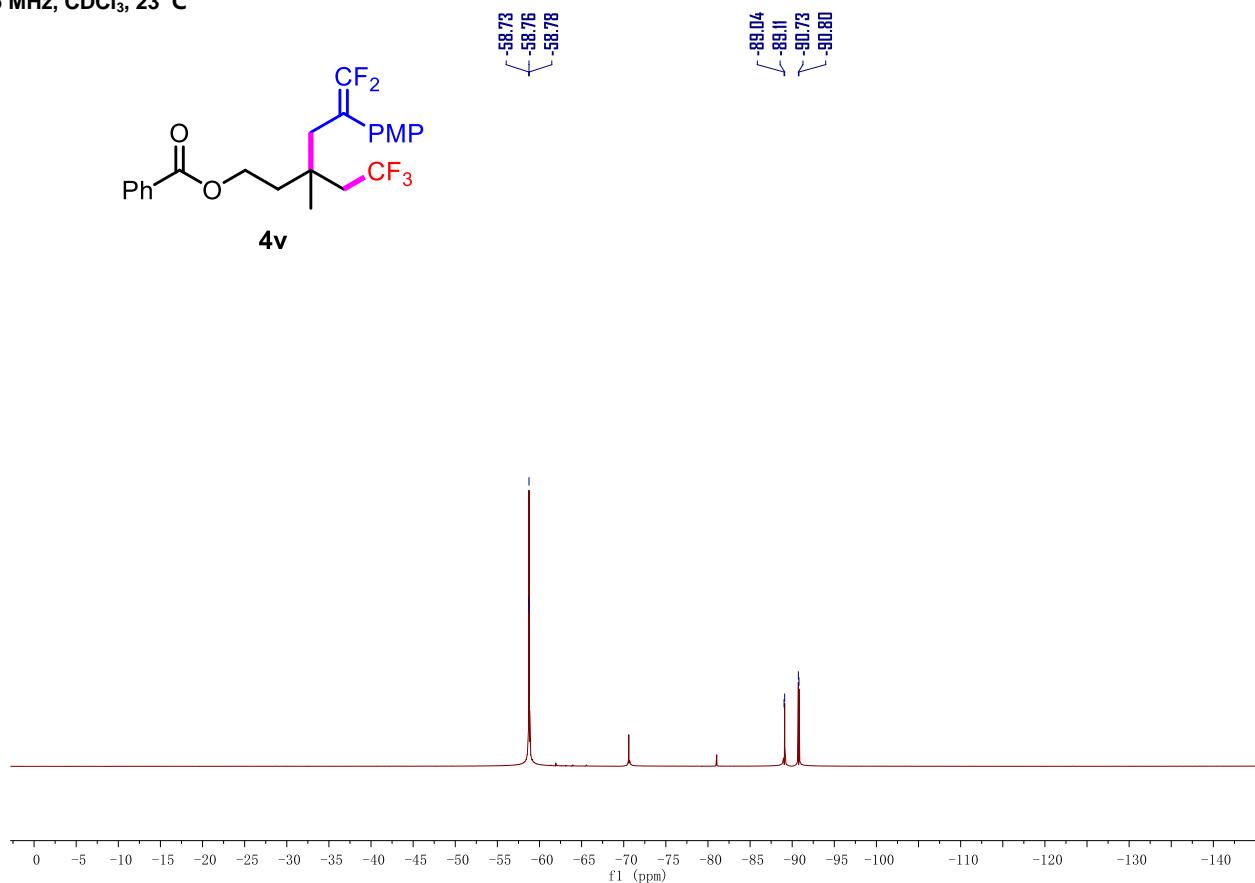
¹³C NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-methyl-3-(2,2,2-trifluoroethyl)hex-5-en-1-yl benzoate (4v)

151 MHz, CDCl₃, 23 °C



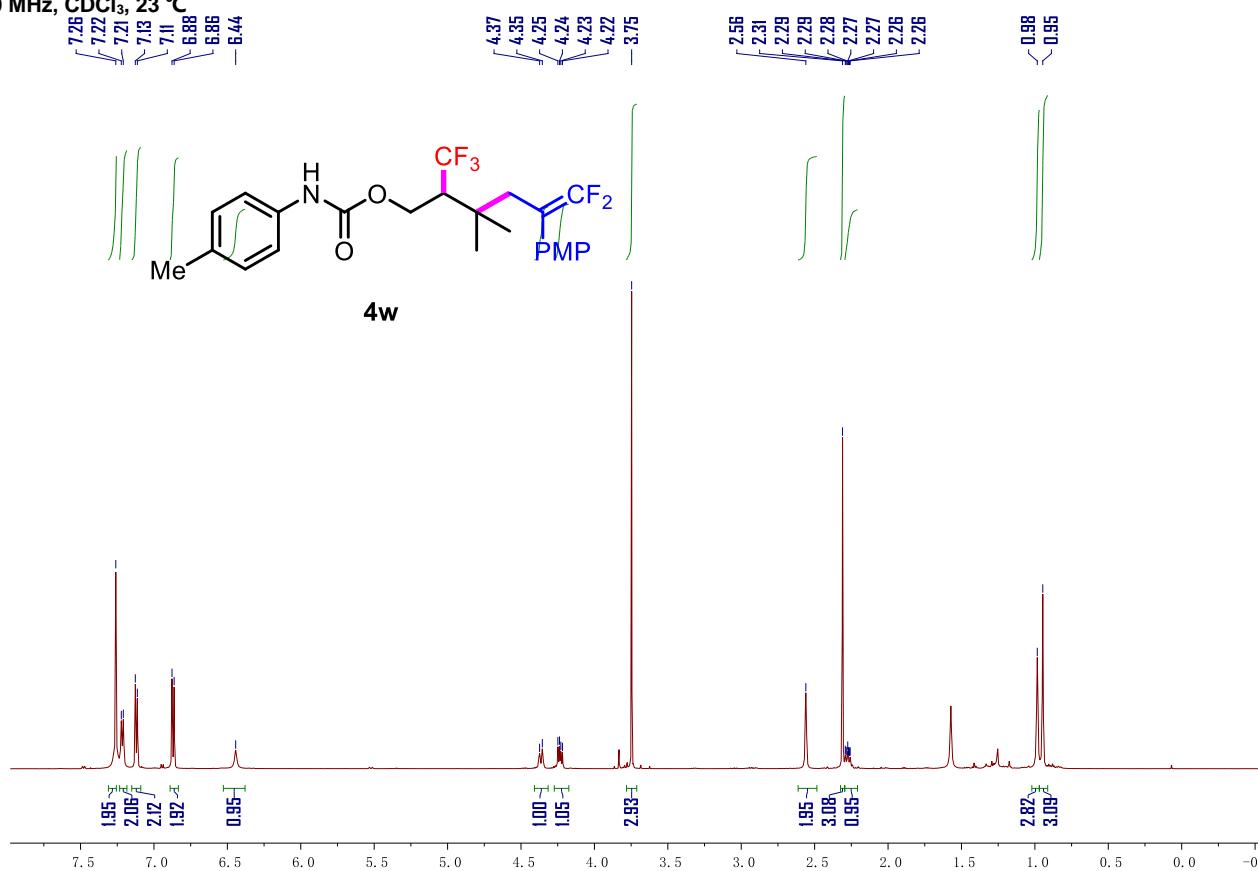
¹⁹F NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3-methyl-3-(2,2,2-trifluoroethyl)hex-5-en-1-yl benzoate (4v)

565 MHz, CDCl₃, 23 °C



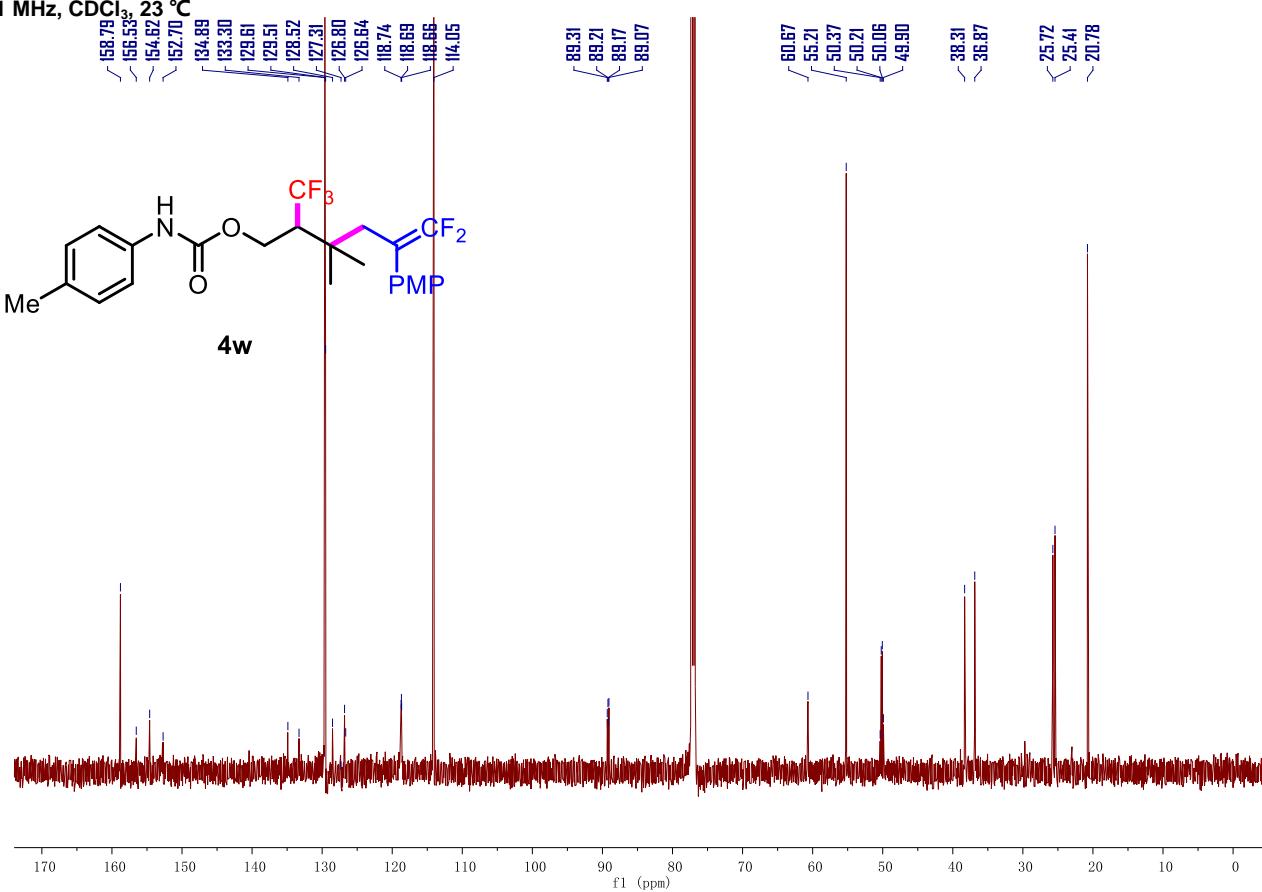
¹H NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3,3-dimethyl-2-(trifluoromethyl)hex-5-en-1-yl *p*-tolylcarbamate (4w)

600 MHz, CDCl₃, 23 °C



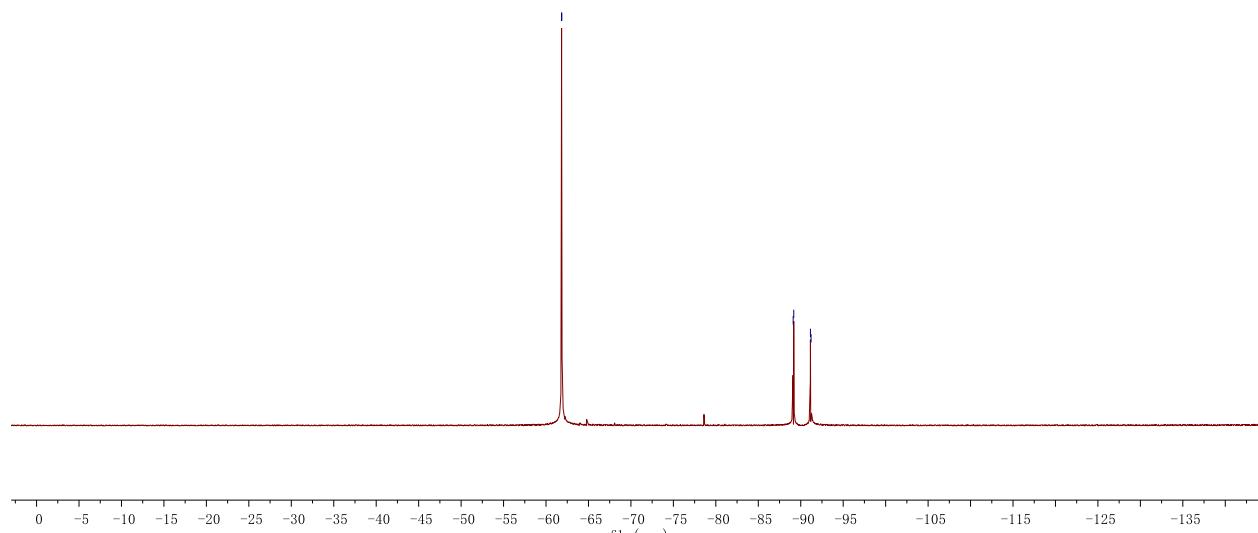
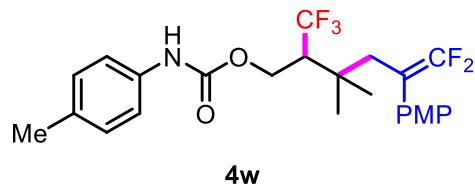
¹³C NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3,3-dimethyl-2-(trifluoromethyl)hex-5-en-1-yl *p*-tolylcarbamate (4w)

151 MHz, CDCl₃, 23 °C



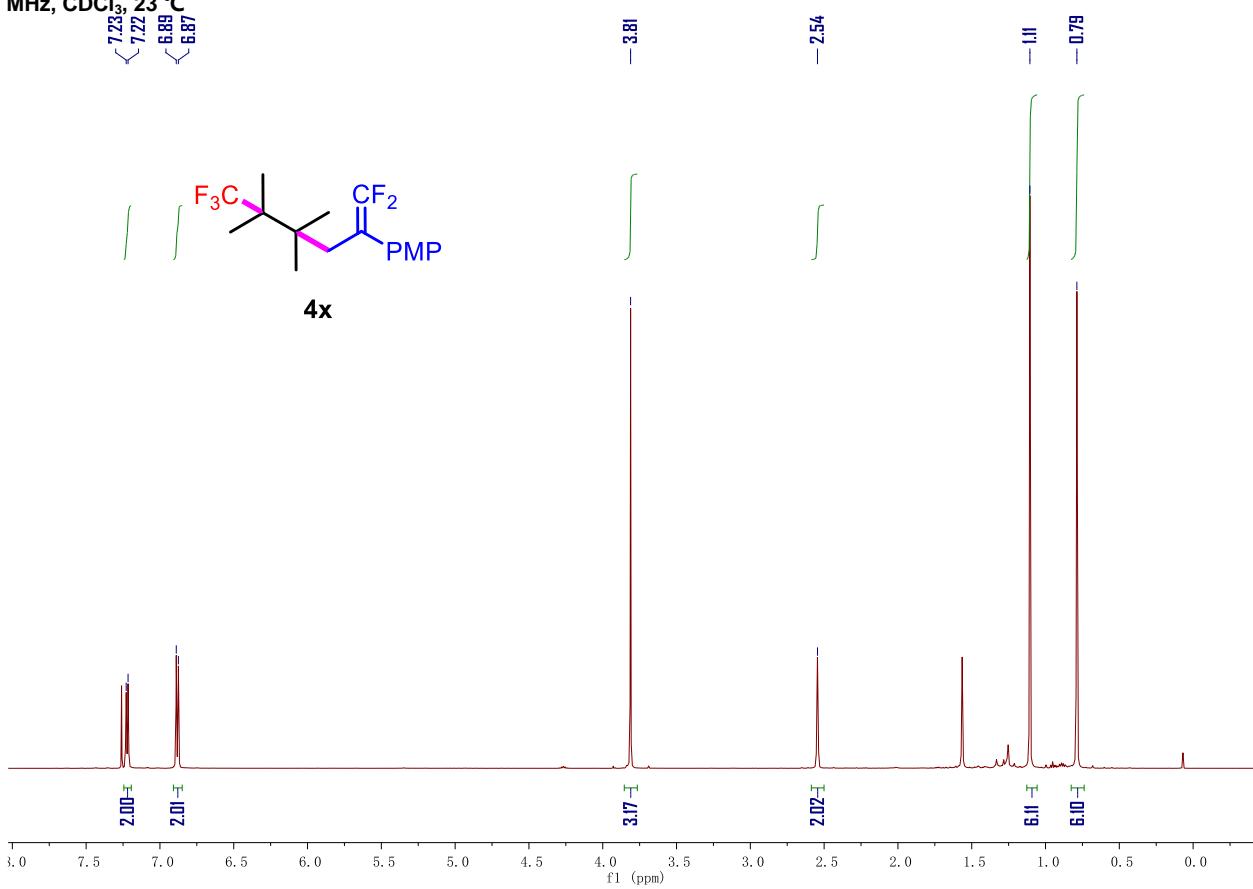
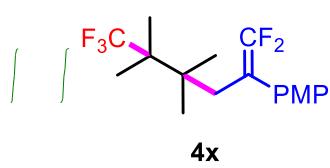
¹⁹F NMR spectrum of 6,6-difluoro-5-(4-methoxyphenyl)-3,3-dimethyl-2-(trifluoromethyl)hex-5-en-1-yl *p*-tolylcarbamate (4w)

565 MHz, CDCl₃, 23 °C



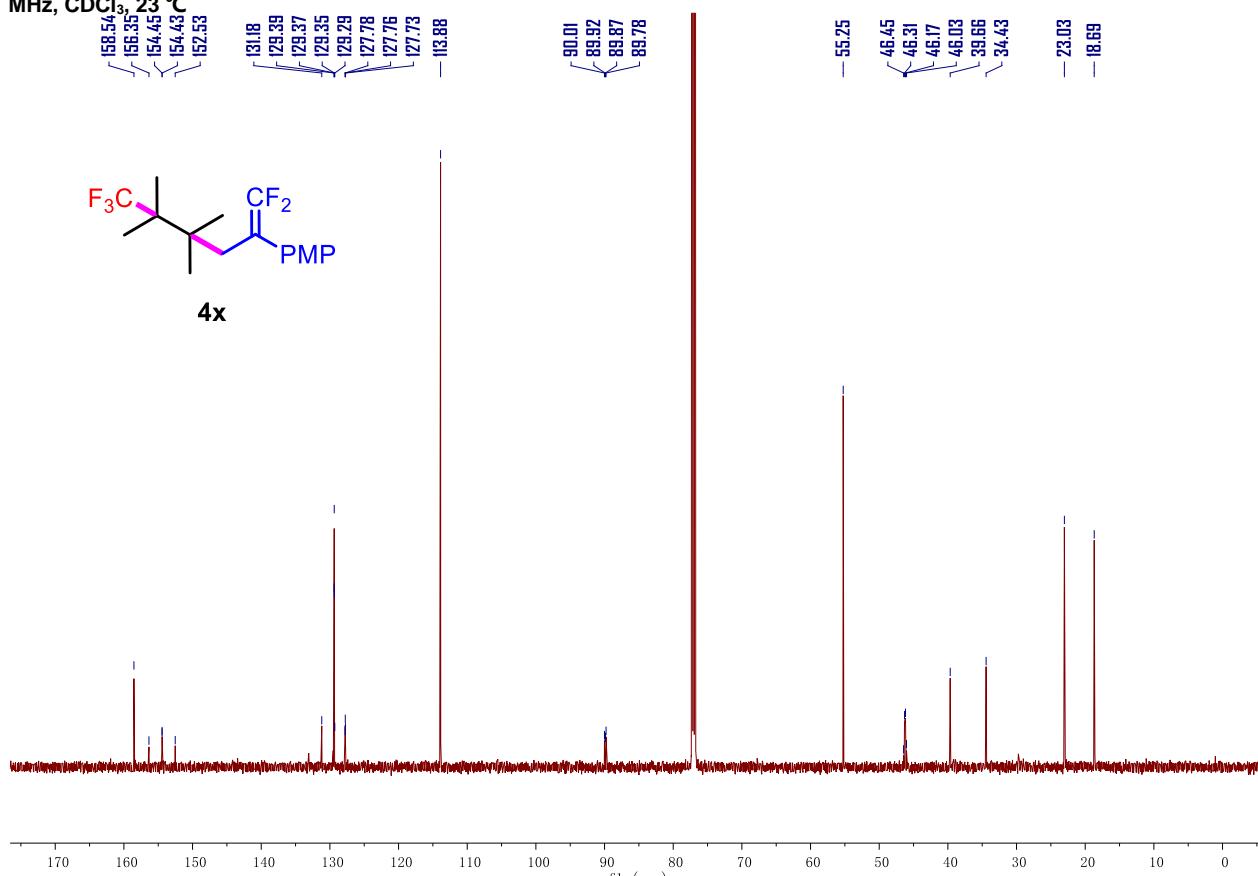
¹H NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4,4,5,5-tetramethylhex-1-en-2-yl)benzene (4x)

600 MHz, CDCl₃, 23 °C



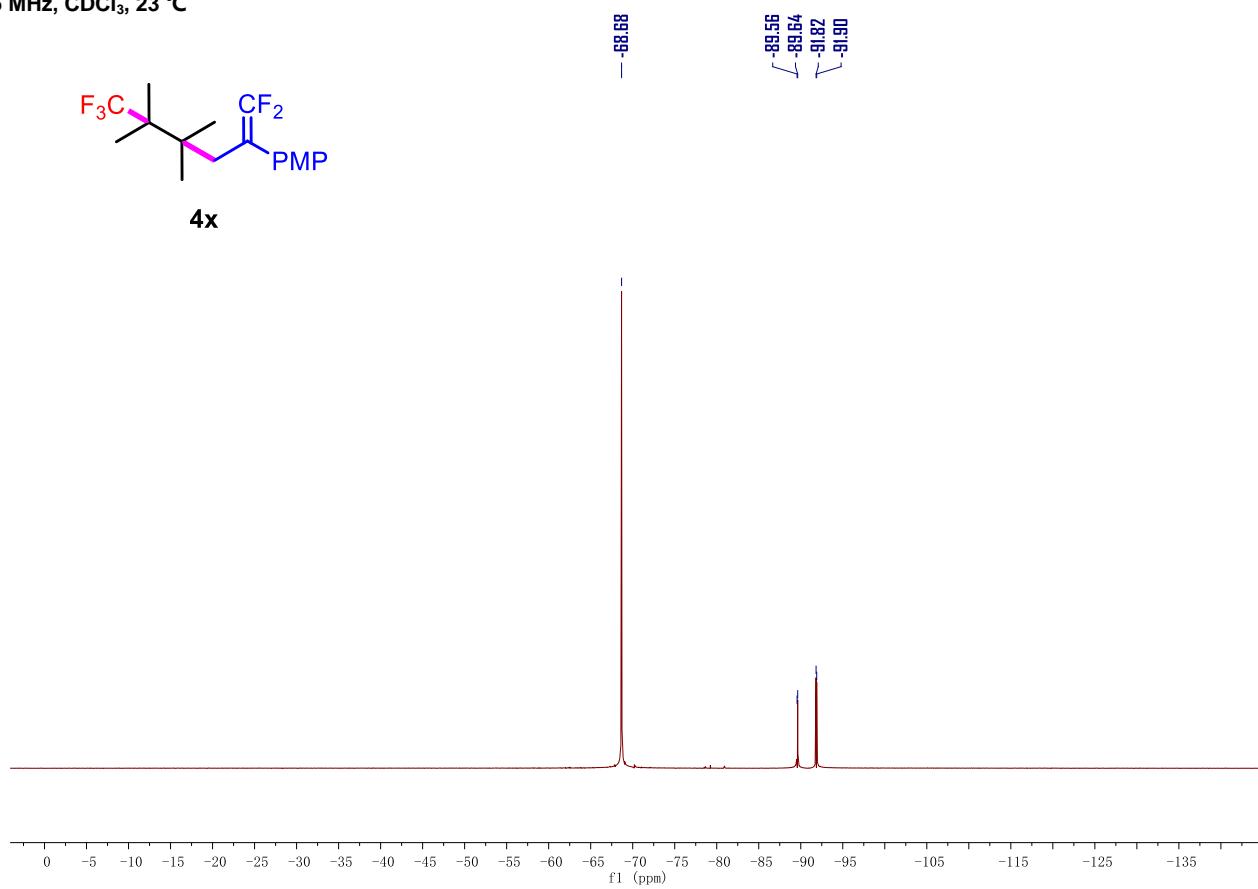
¹³C NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4,4,5,5-tetramethylhex-1-en-2-yl)benzene (4x)

151 MHz, CDCl₃, 23 °C



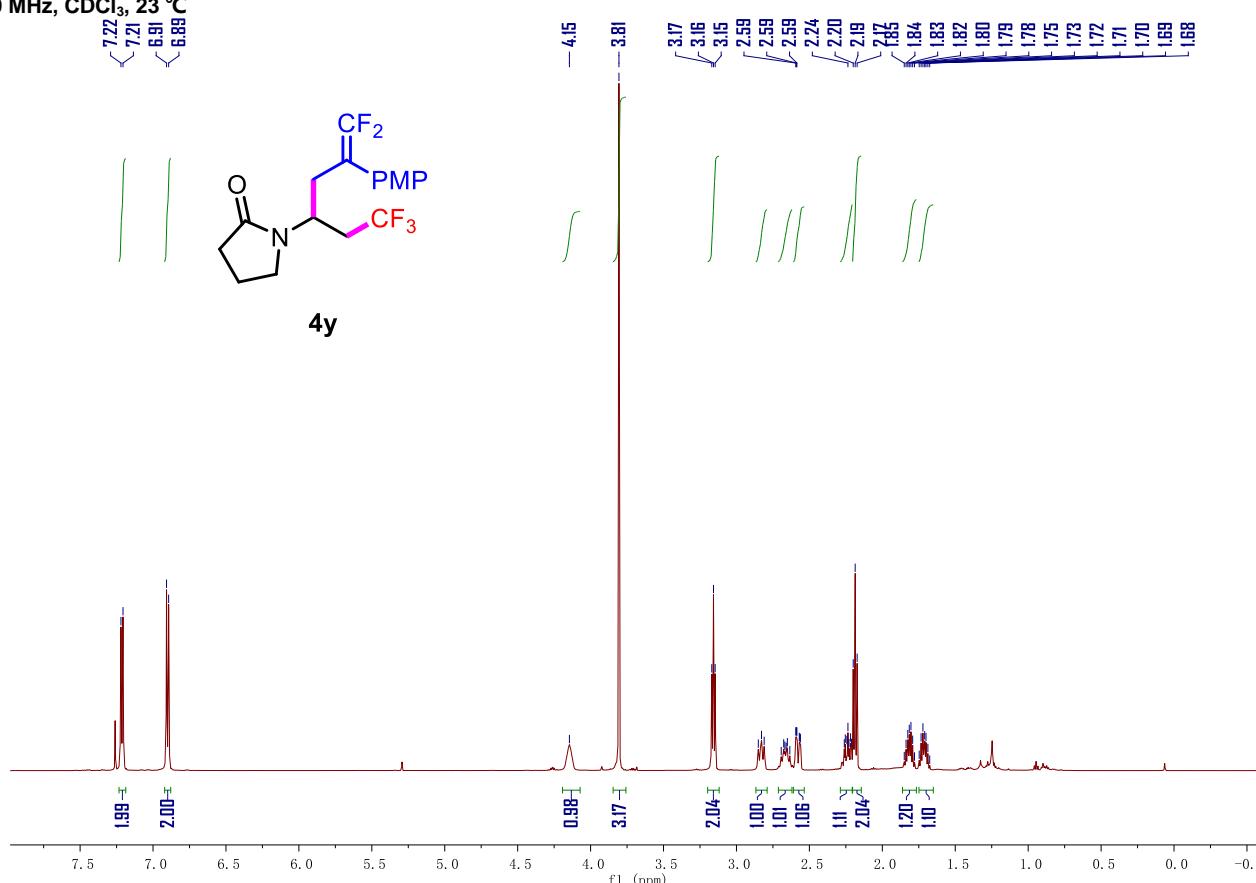
¹⁹F NMR spectrum of 1-methoxy-4-(1,1,6,6,6-pentafluoro-4,4,5,5-tetramethylhex-1-en-2-yl)benzene (4x)

565 MHz, CDCl₃, 23 °C



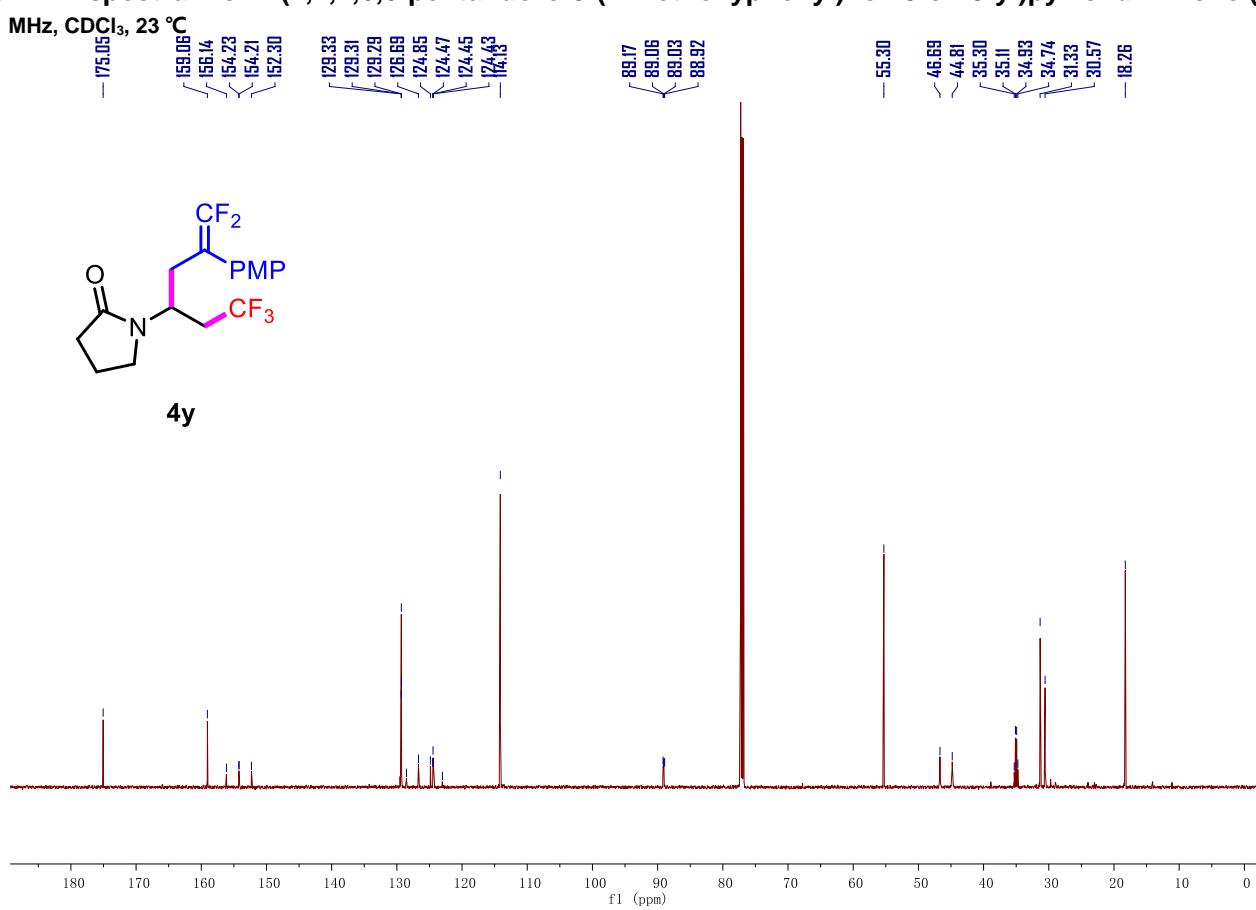
¹H NMR spectrum of 1-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)pyrrolidin-2-one (4y)

600 MHz, CDCl₃, 23 °C

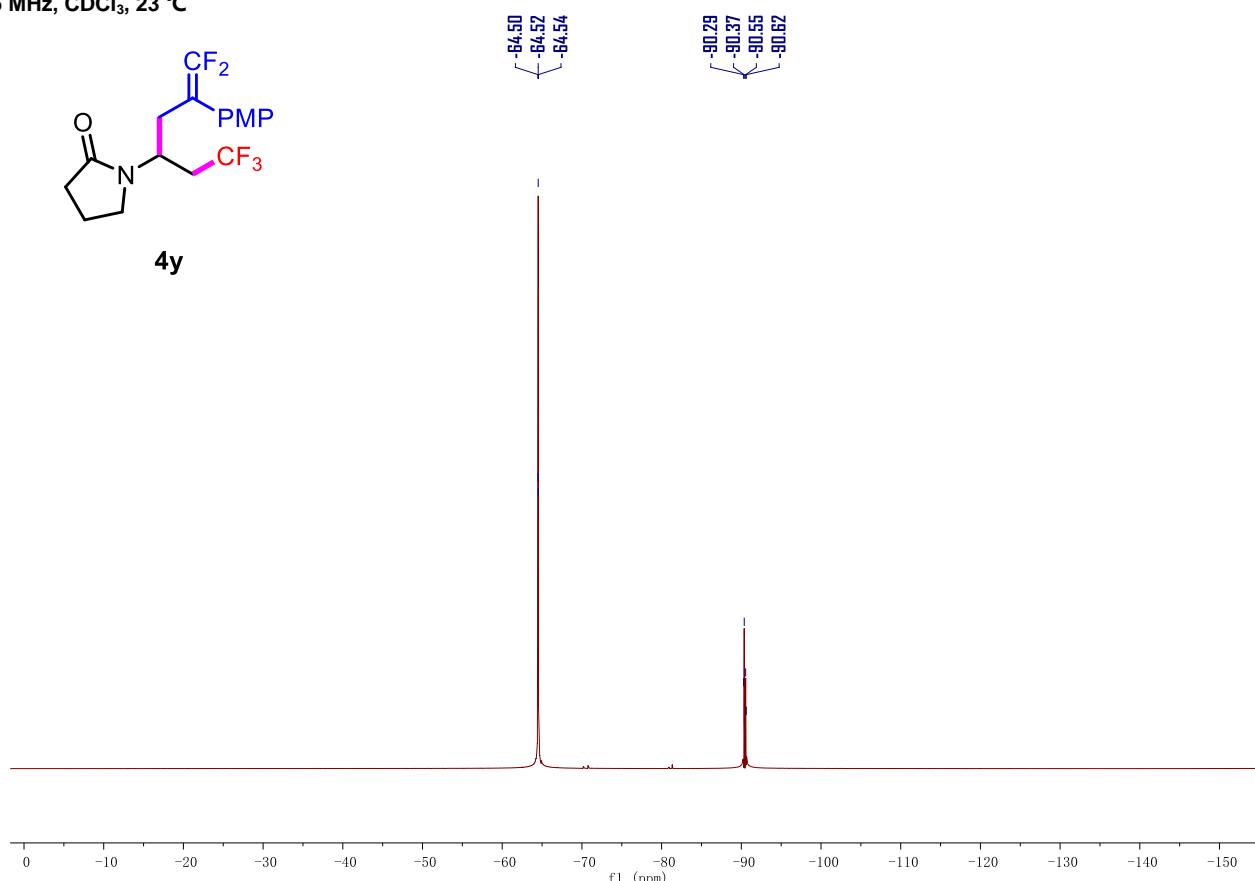


¹³C NMR spectrum of 1-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)pyrrolidin-2-one (4y)

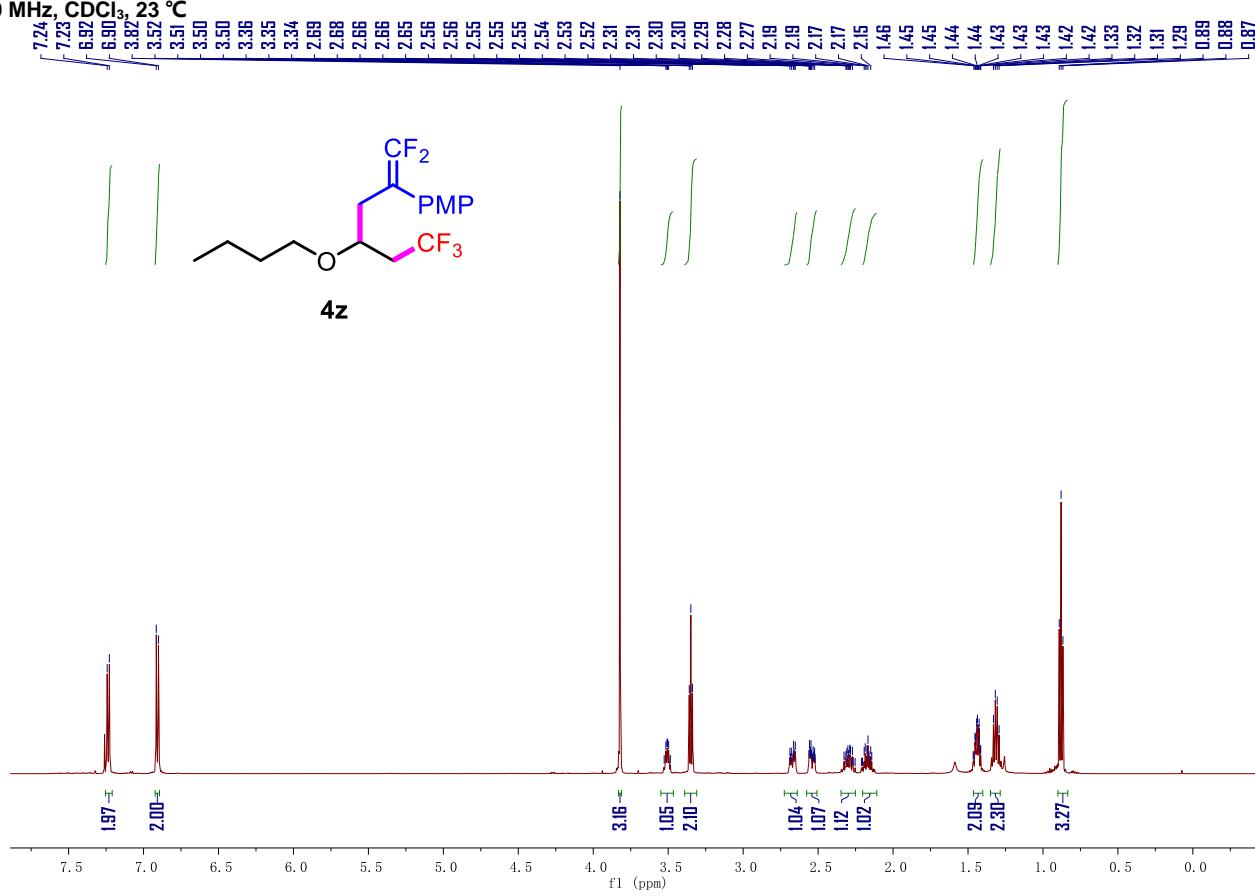
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)hex-5-en-3-yl)pyrrolidin-2-one (4y)
 565 MHz, CDCl₃, 23 °C

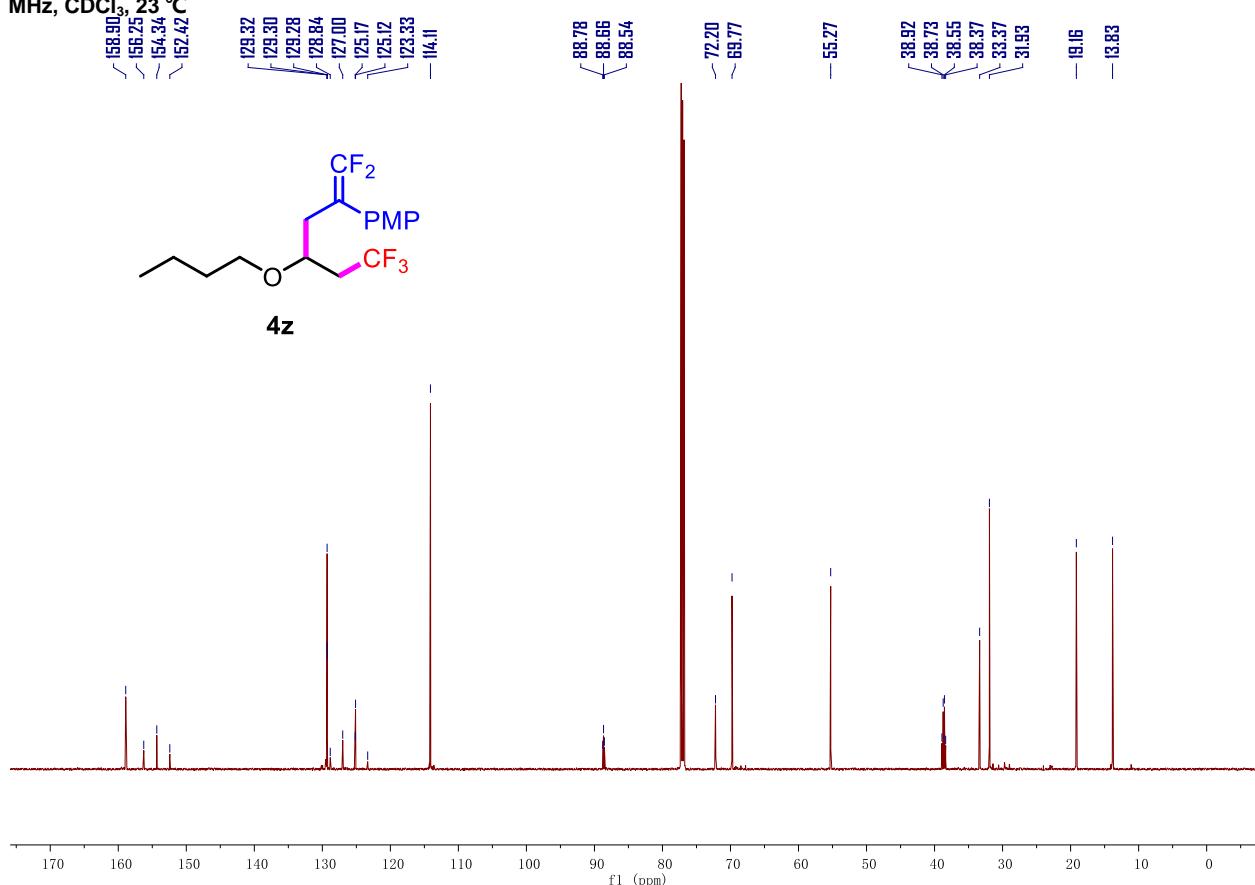


¹H NMR spectrum of 1-(4-butoxy-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4z)
 600 MHz, CDCl₃, 23 °C



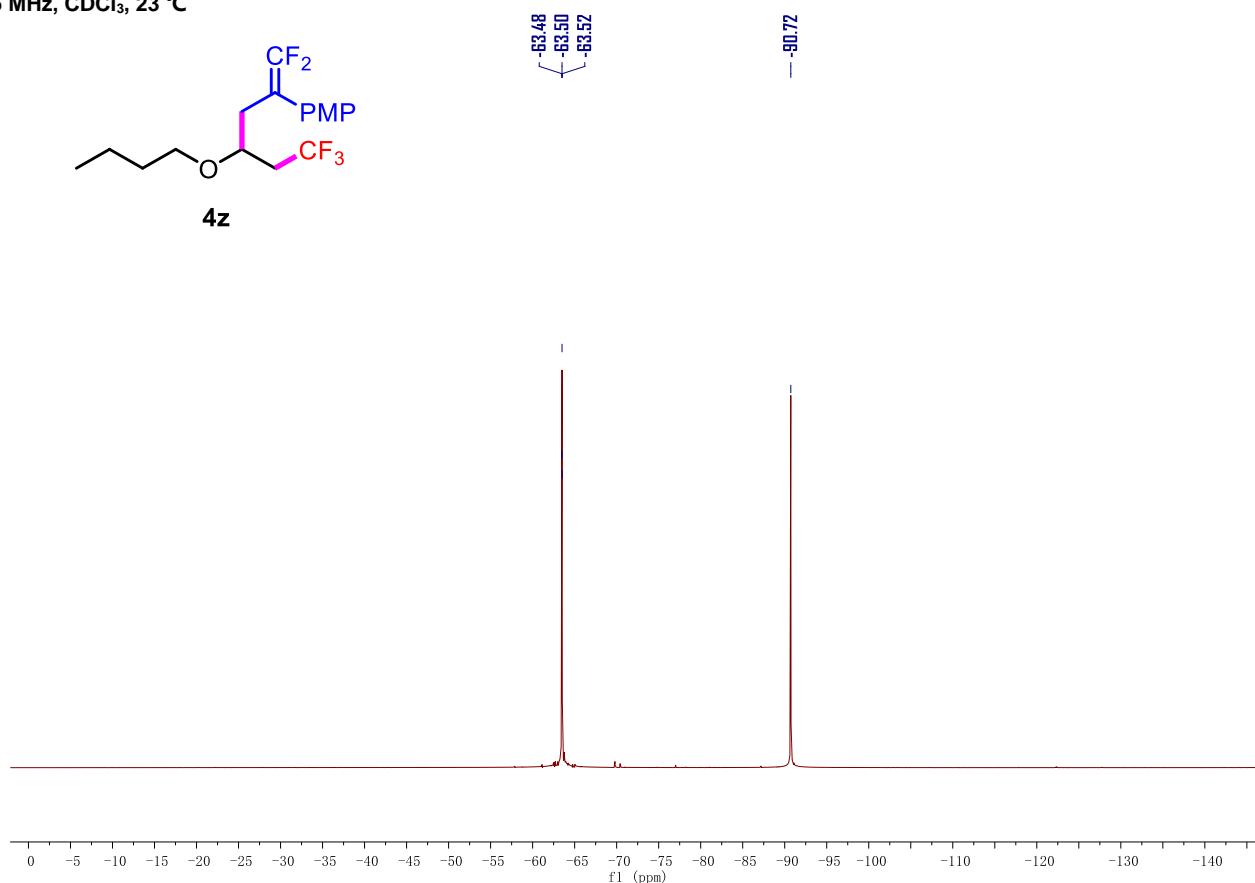
¹³C NMR spectrum of 1-(4-butoxy-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4z)

151 MHz, CDCl₃, 23 °C

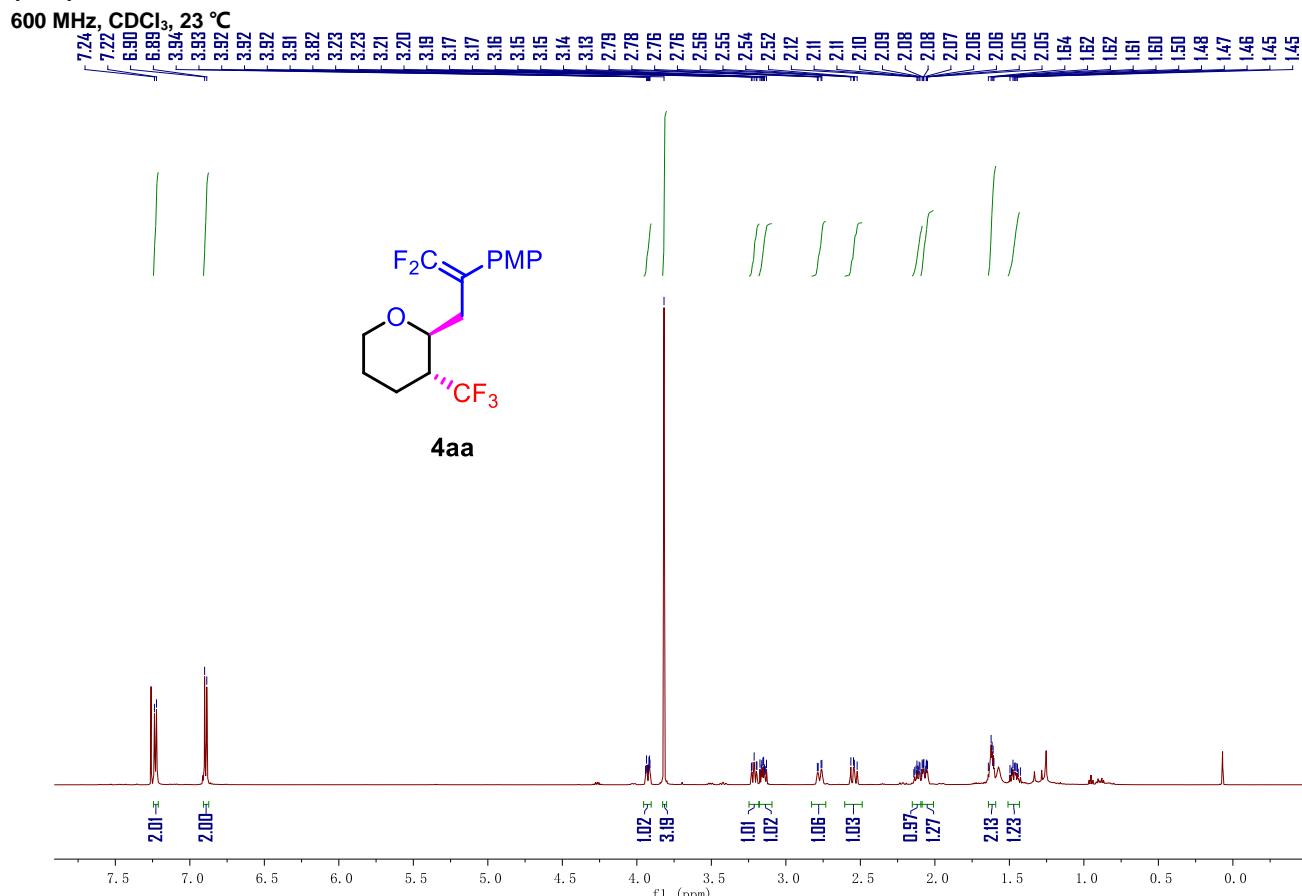


¹⁹F NMR spectrum of 1-(4-butoxy-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-methoxybenzene (4z)

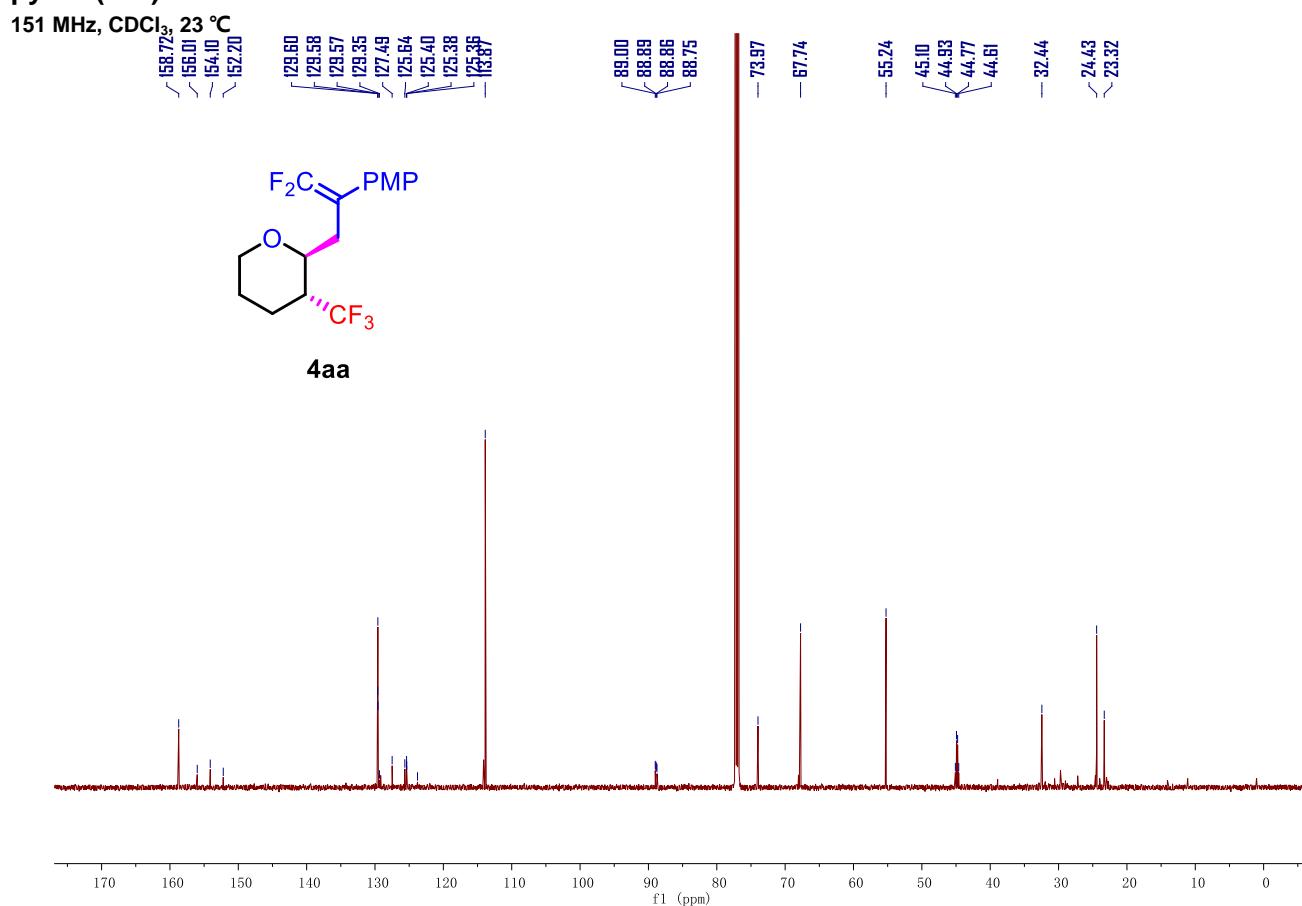
565 MHz, CDCl₃, 23 °C



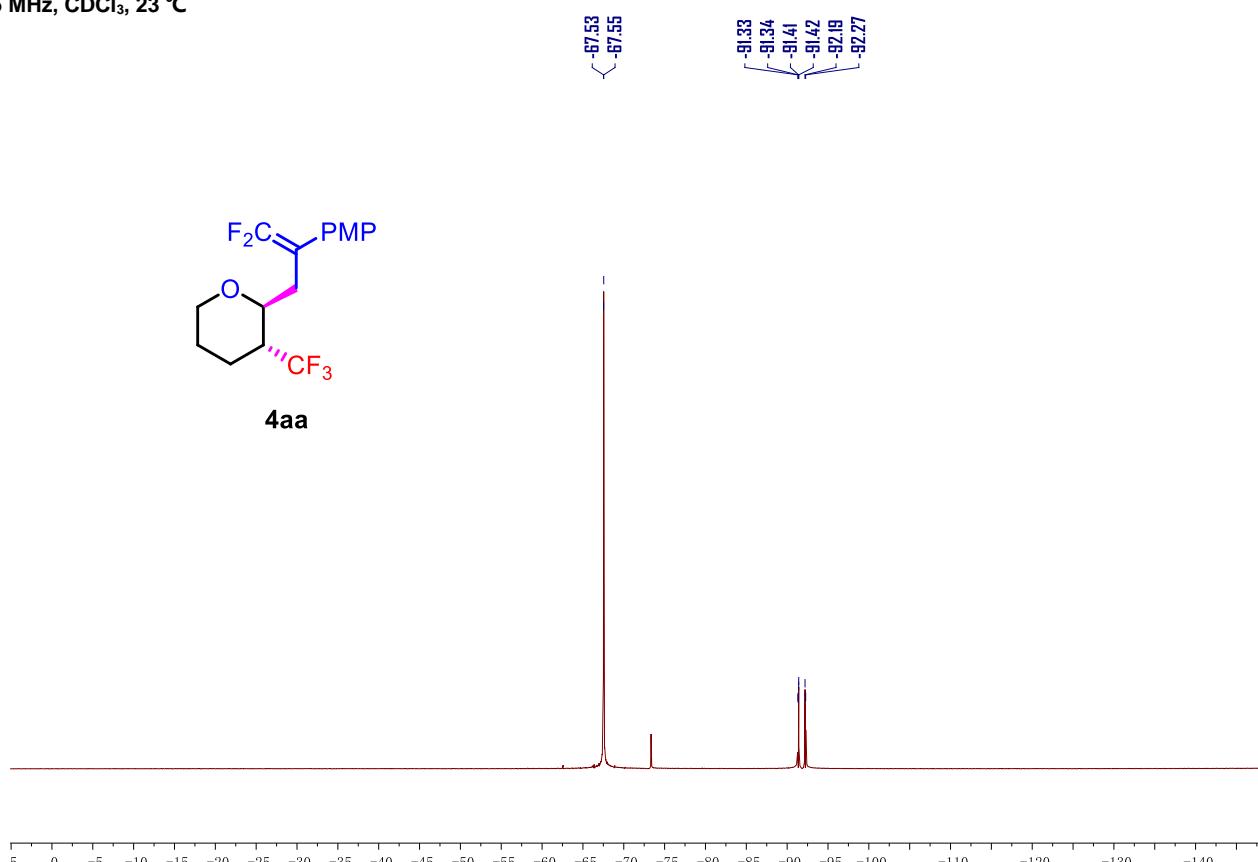
¹H NMR spectrum of 2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)tetrahydro-2H-pyran (4aa)



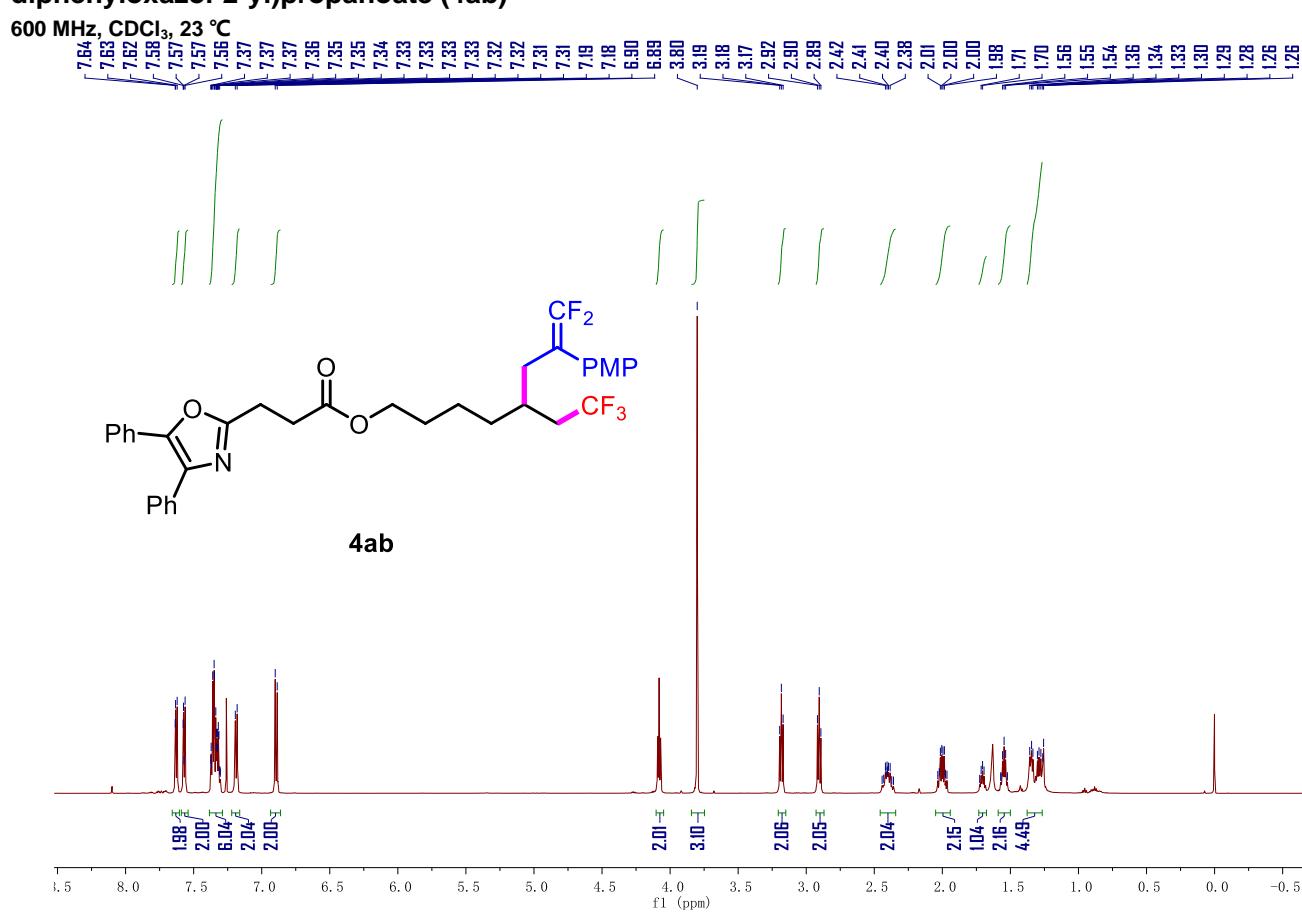
¹³C NMR spectrum of 2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)tetrahydro-2H-pyran (4aa)



¹⁹F NMR spectrum of 2-(3,3-difluoro-2-(4-methoxyphenyl)allyl)-3-(trifluoromethyl)tetrahydro-2H-pyran (4aa)
565 MHz, CDCl₃, 23 °C

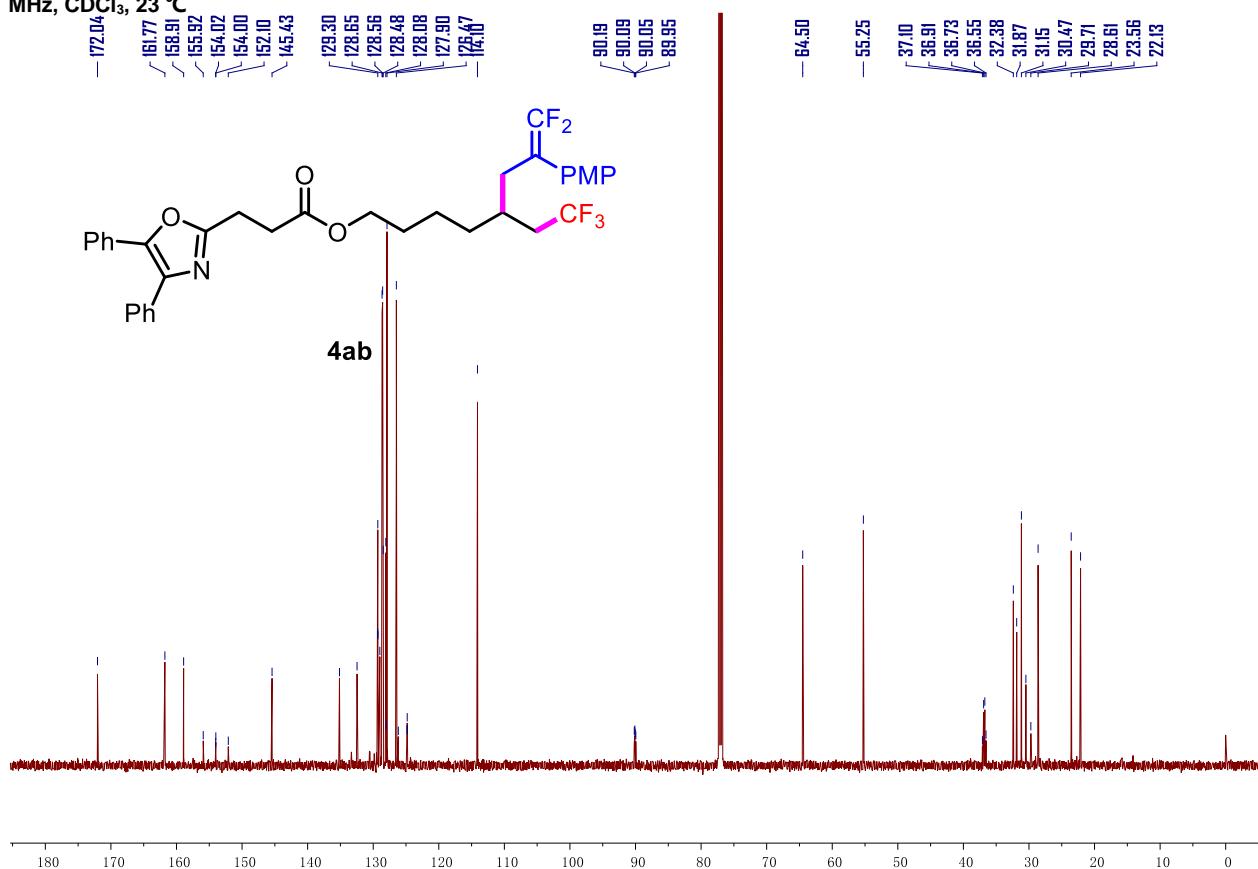


¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 3-(4,5-diphenyloxazol-2-yl)propanoate (4ab)

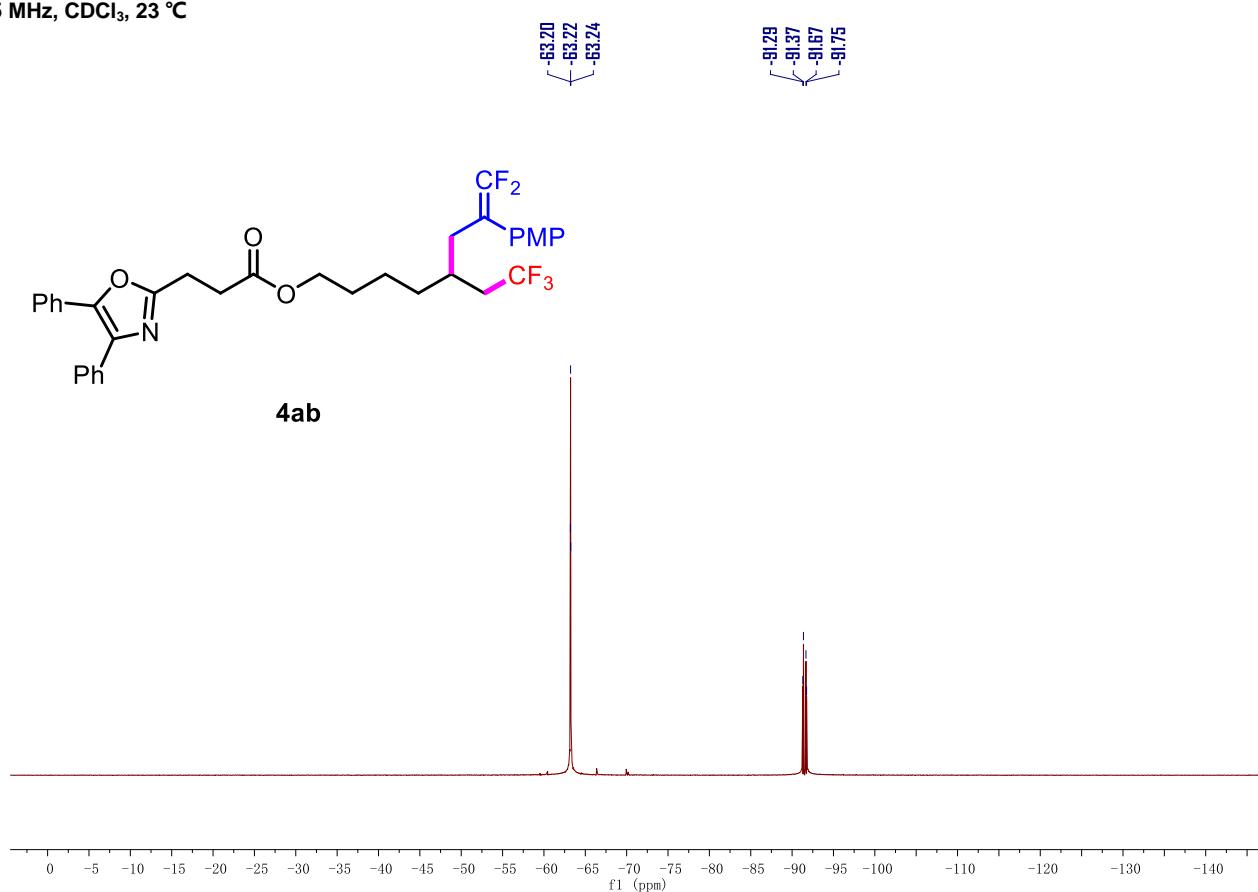


¹³C NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 3-(4,5-diphenyloxazol-2-yl)propanoate (4ab)

151 MHz, CDCl₃, 23 °C

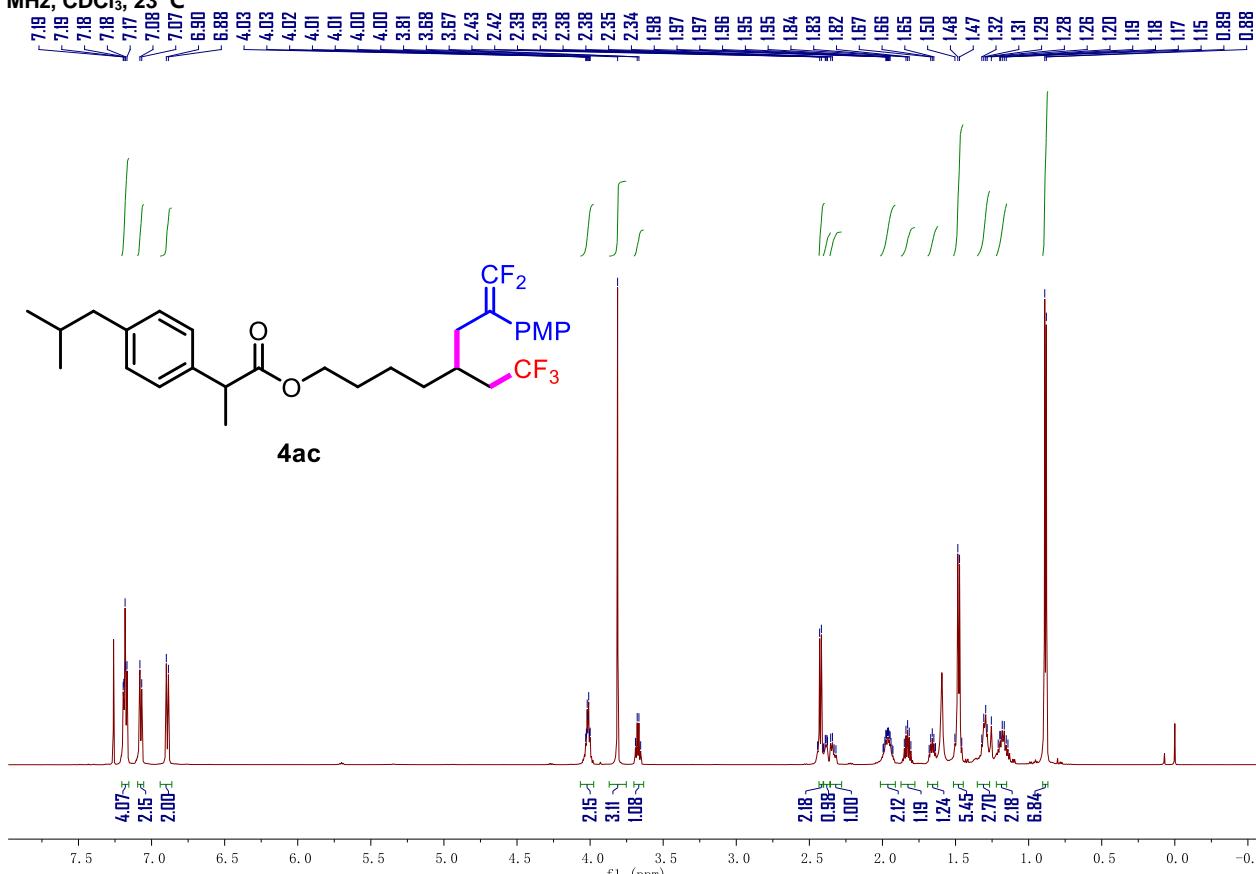


565 MHz, CDCl₃, 23 °C



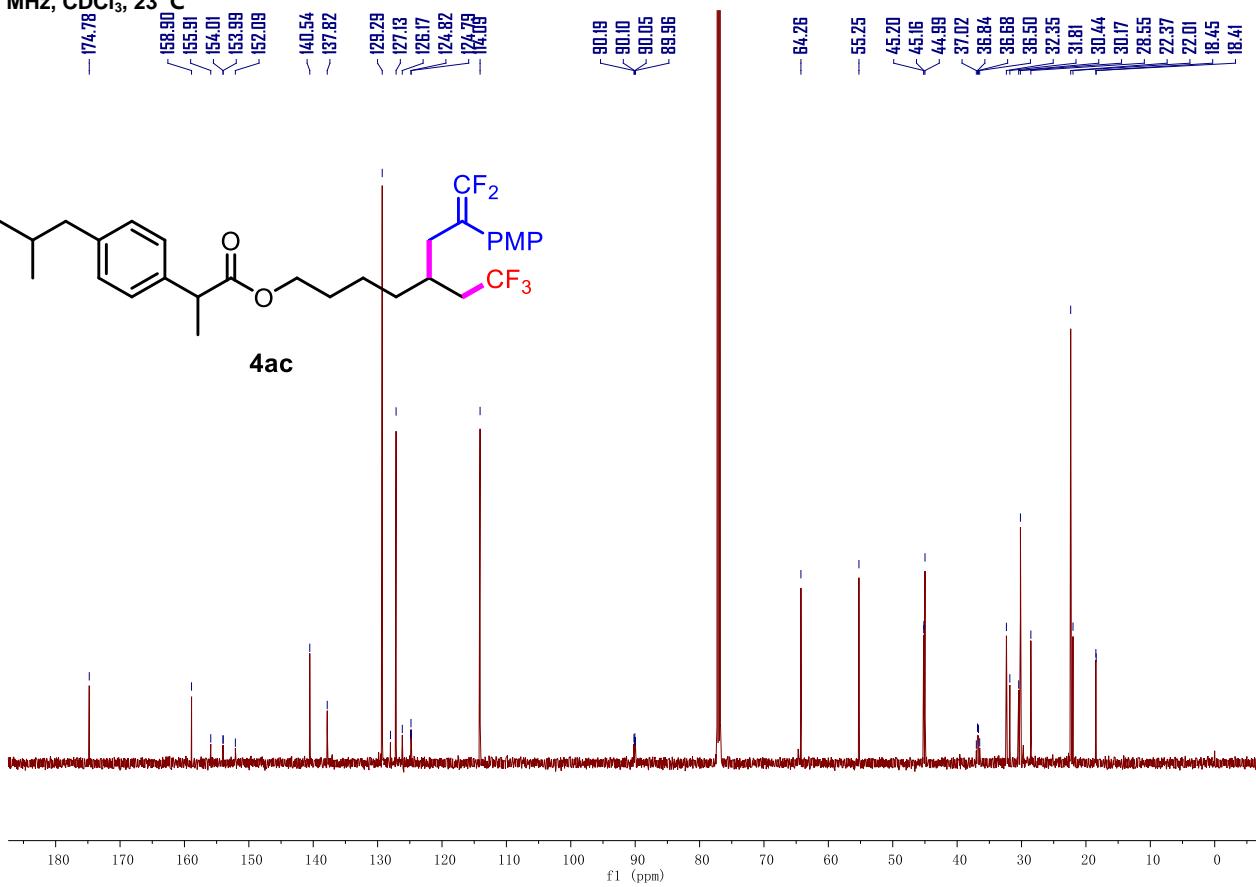
¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-isobutylphenyl)propanoate (4ac)

600 MHz, CDCl₃, 23 °C



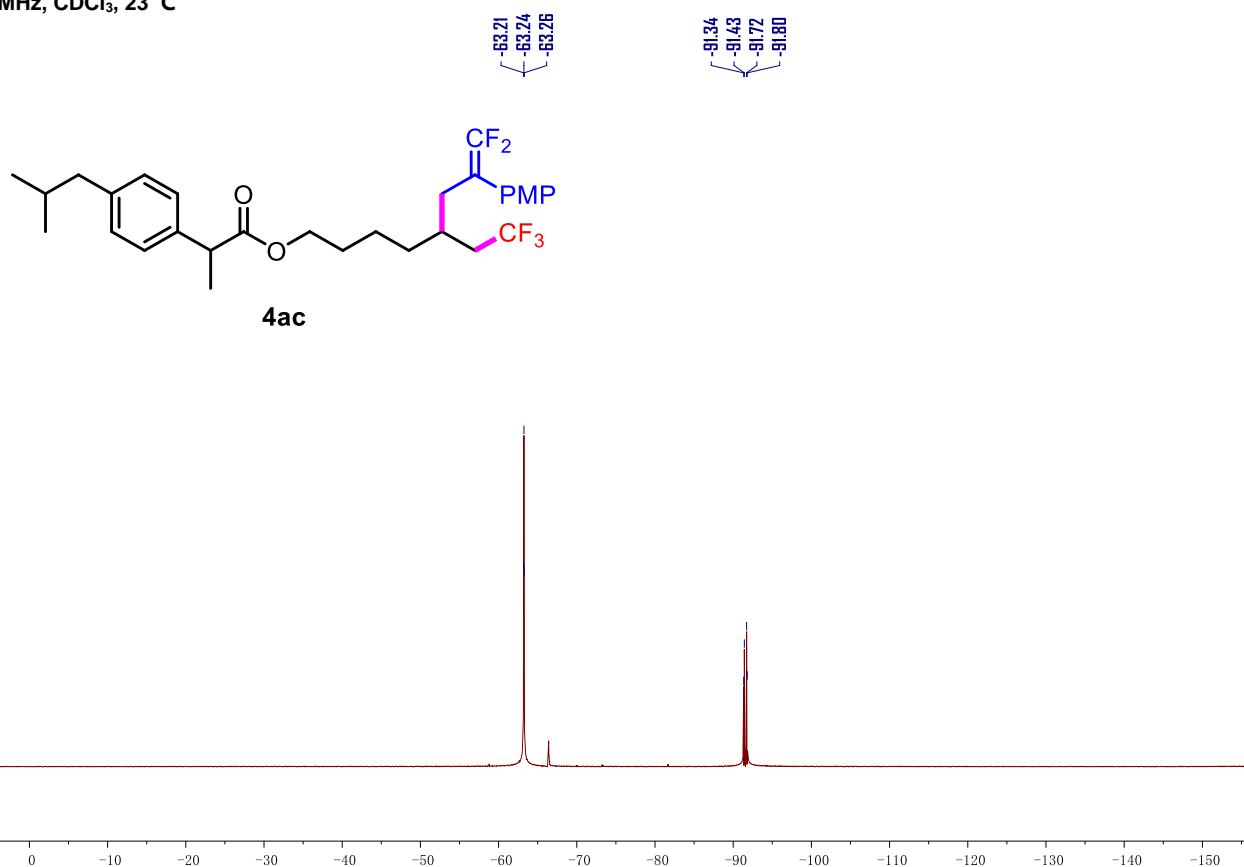
¹³C NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-isobutylphenyl)propanoate (4ac)

151 MHz, CDCl₃, 23 °C

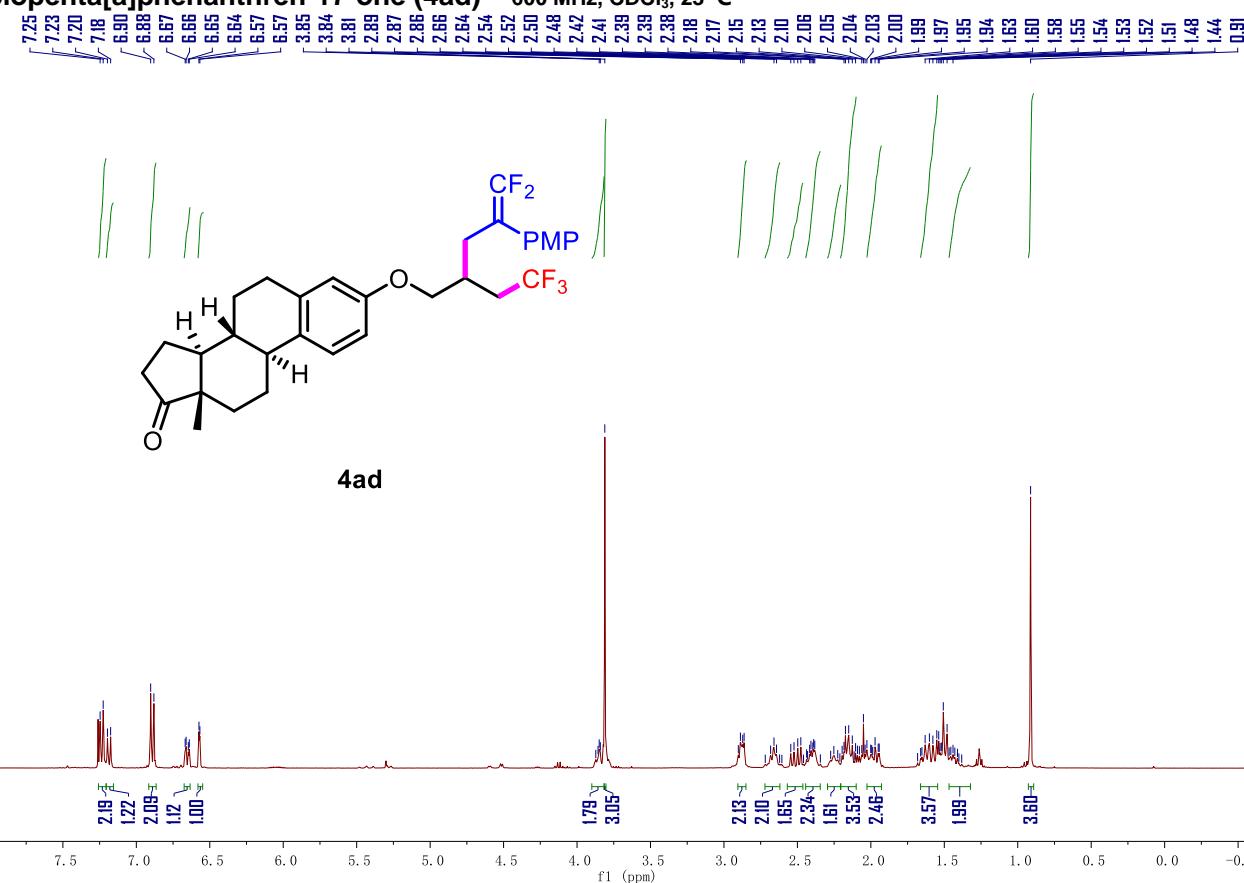


¹⁹F NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-isobutylphenyl)propanoate (4ac)

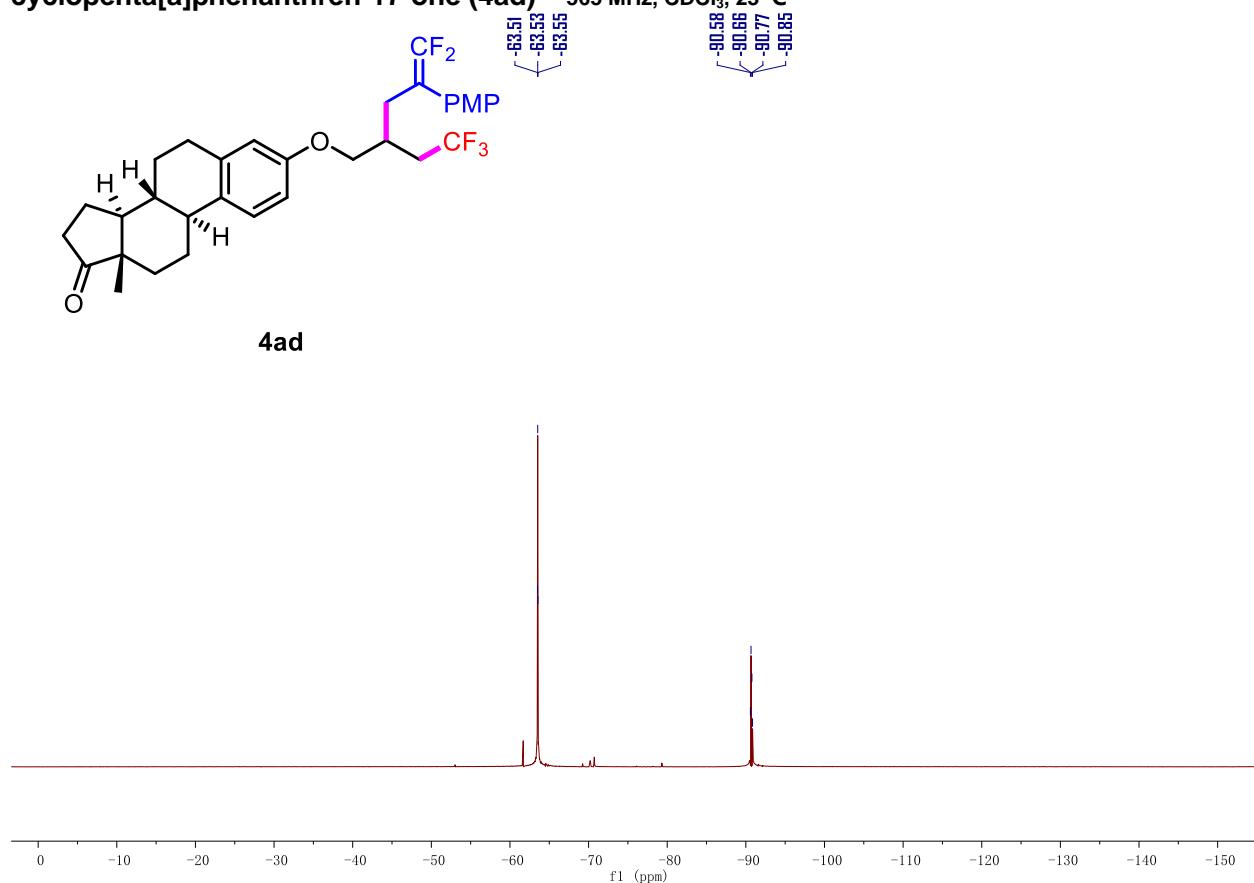
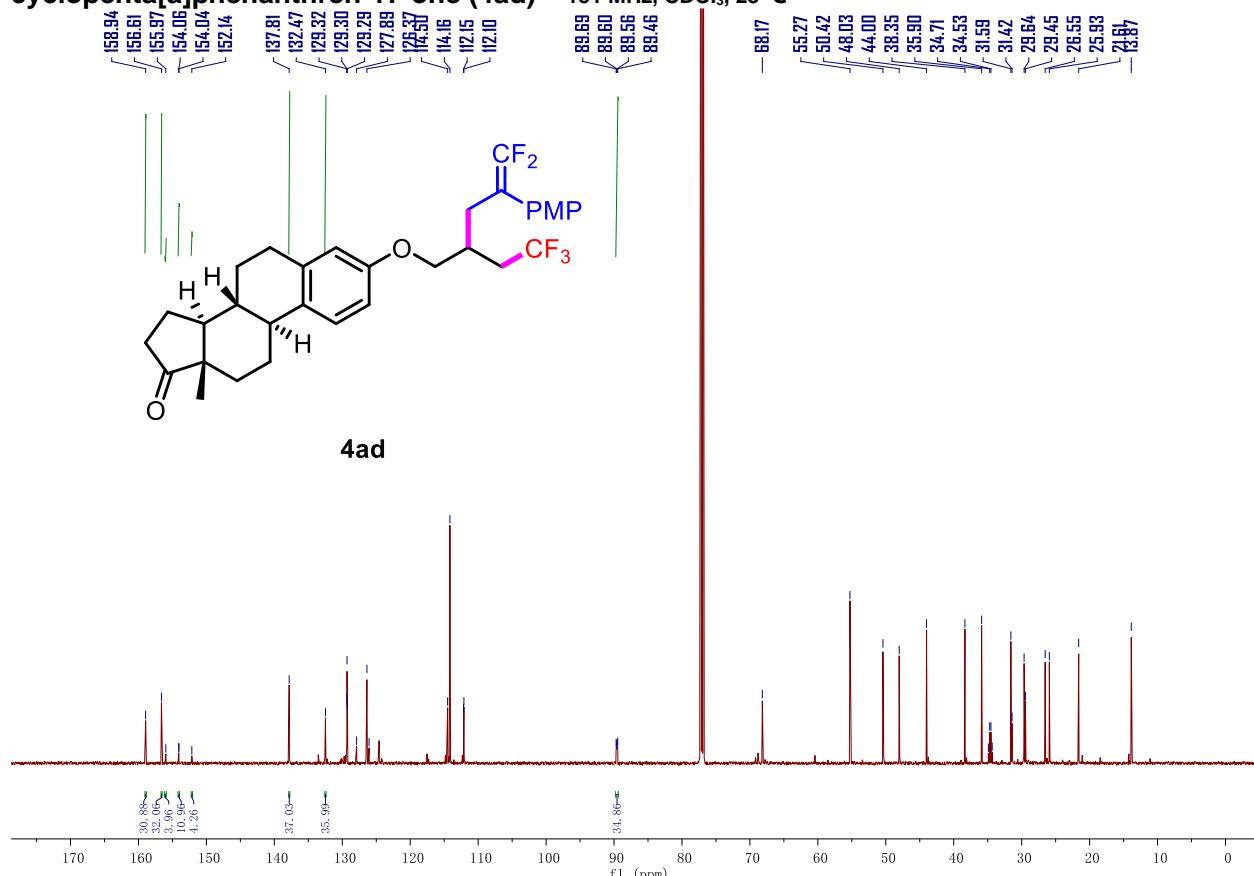
565 MHz, CDCl₃, 23 °C



¹H NMR spectrum of (8R,9S,13S,14S)-3-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ad) 600 MHz, CDCl₃, 23 °C

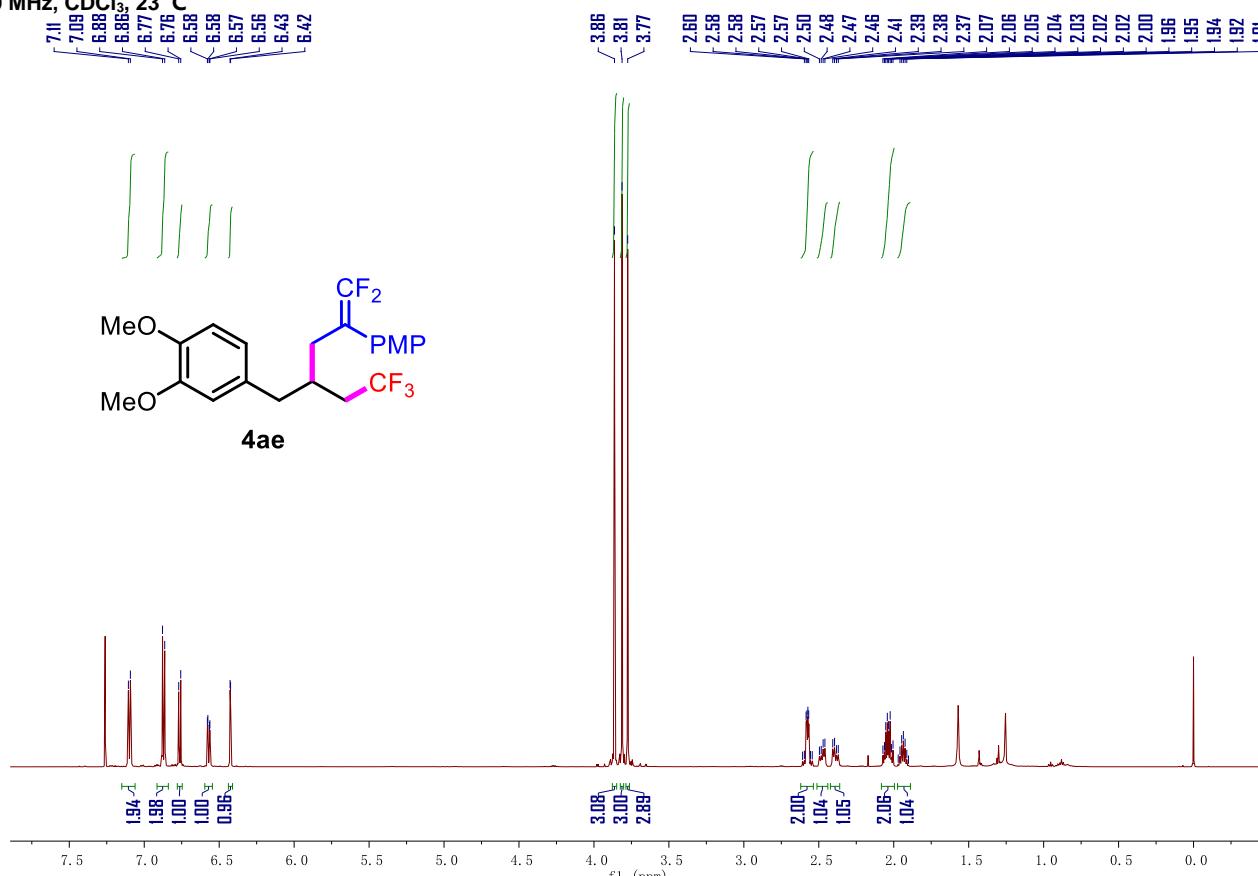


¹³C NMR spectrum of (8R,9S,13S,14S)-3-((5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)oxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4ad) 151 MHz, CDCl₃, 23 °C



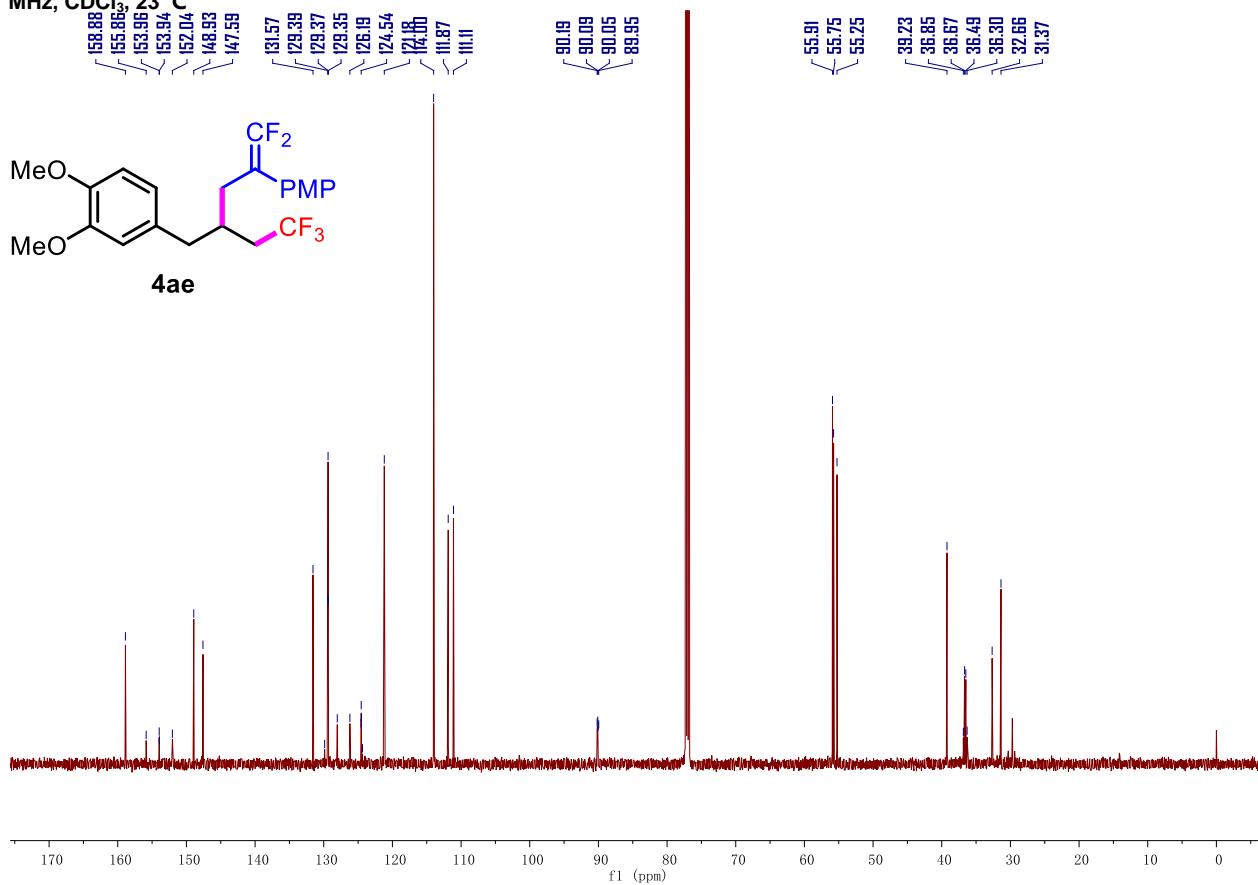
¹H NMR spectrum of 4-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)-1,2-dimethoxybenzene (4ae)

600 MHz, CDCl₃, 23 °C



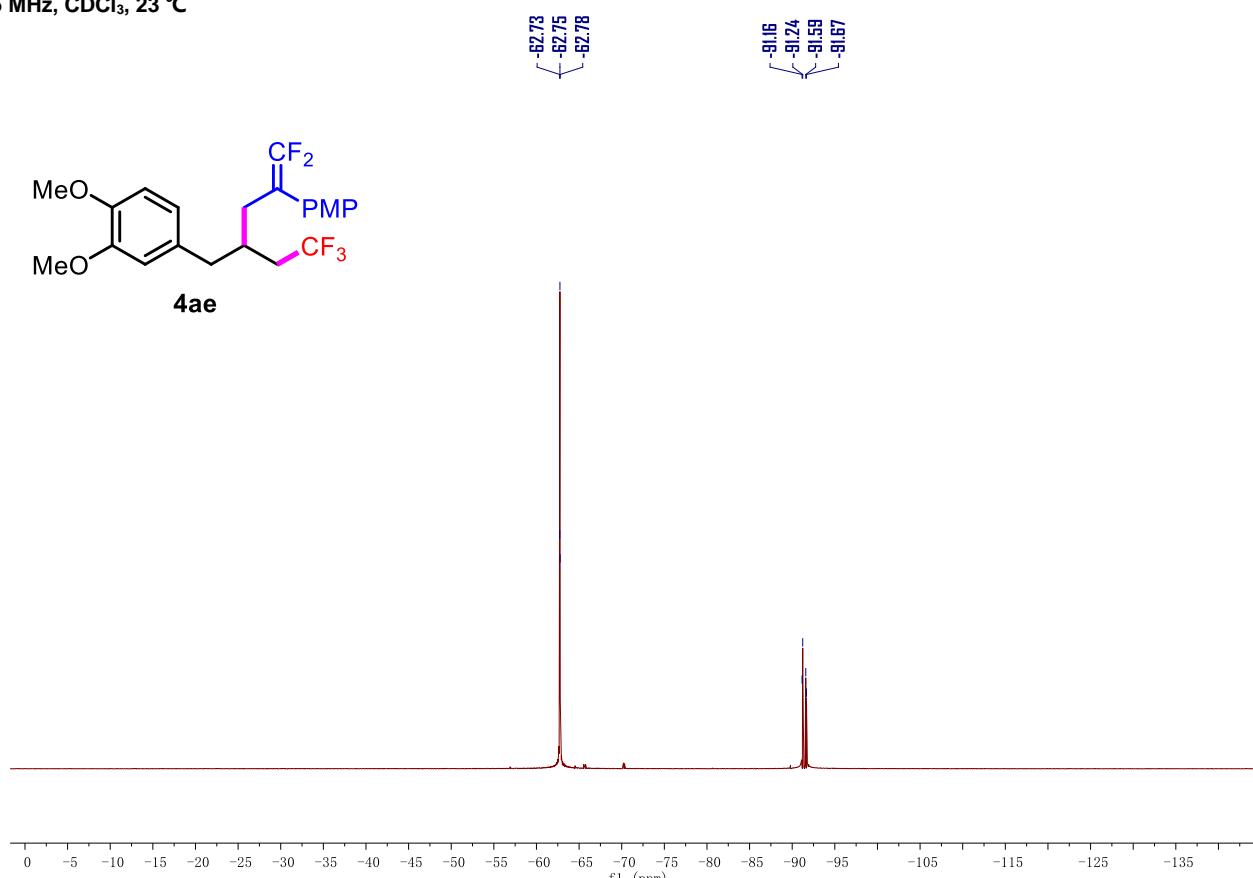
¹³C NMR spectrum of 4-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)-1,2-dimethoxybenzene (4ae)

151 MHz, CDCl₃, 23 °C



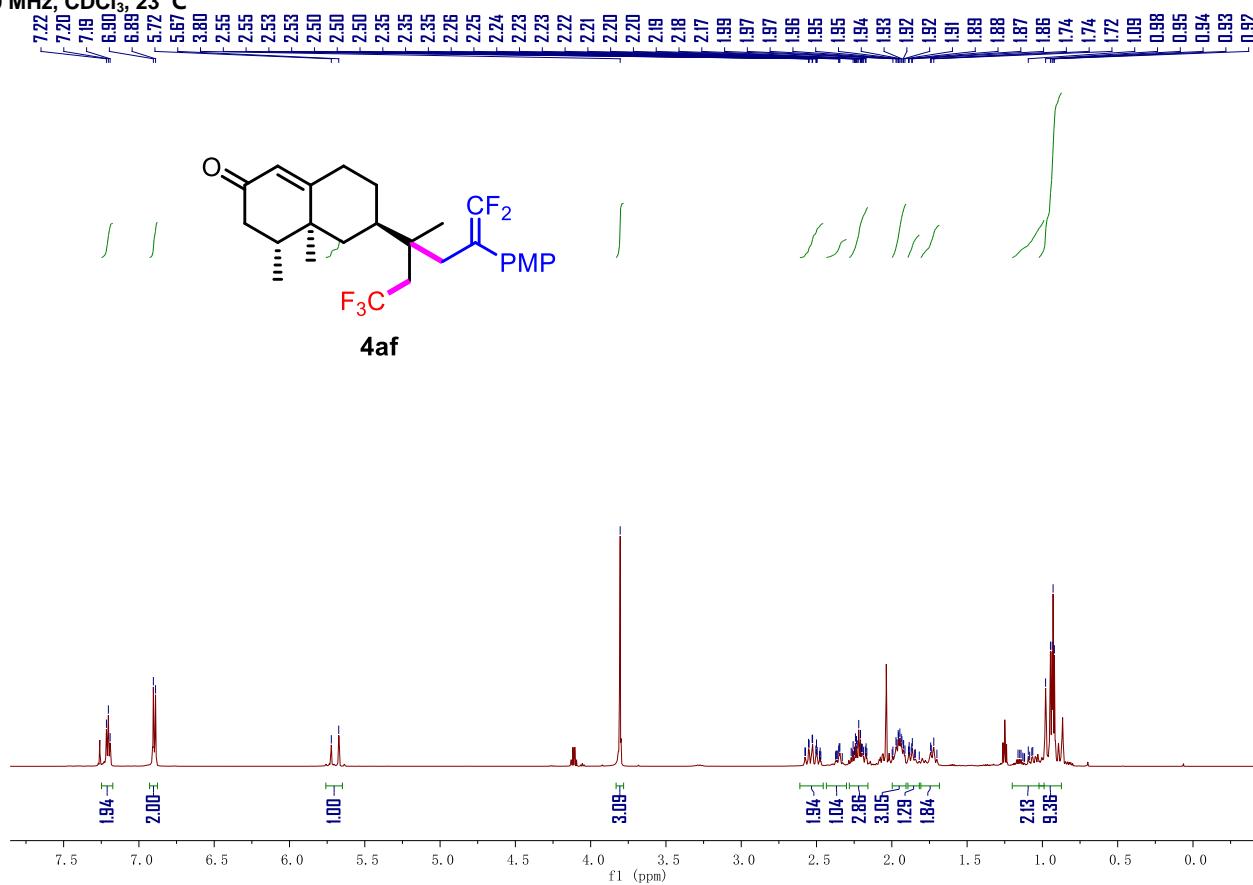
¹⁹F NMR spectrum of 4-(5,5-difluoro-4-(4-methoxyphenyl)-2-(2,2,2-trifluoroethyl)pent-4-en-1-yl)-1,2-dimethoxybenzene (4ae)

565 MHz, CDCl₃, 23 °C

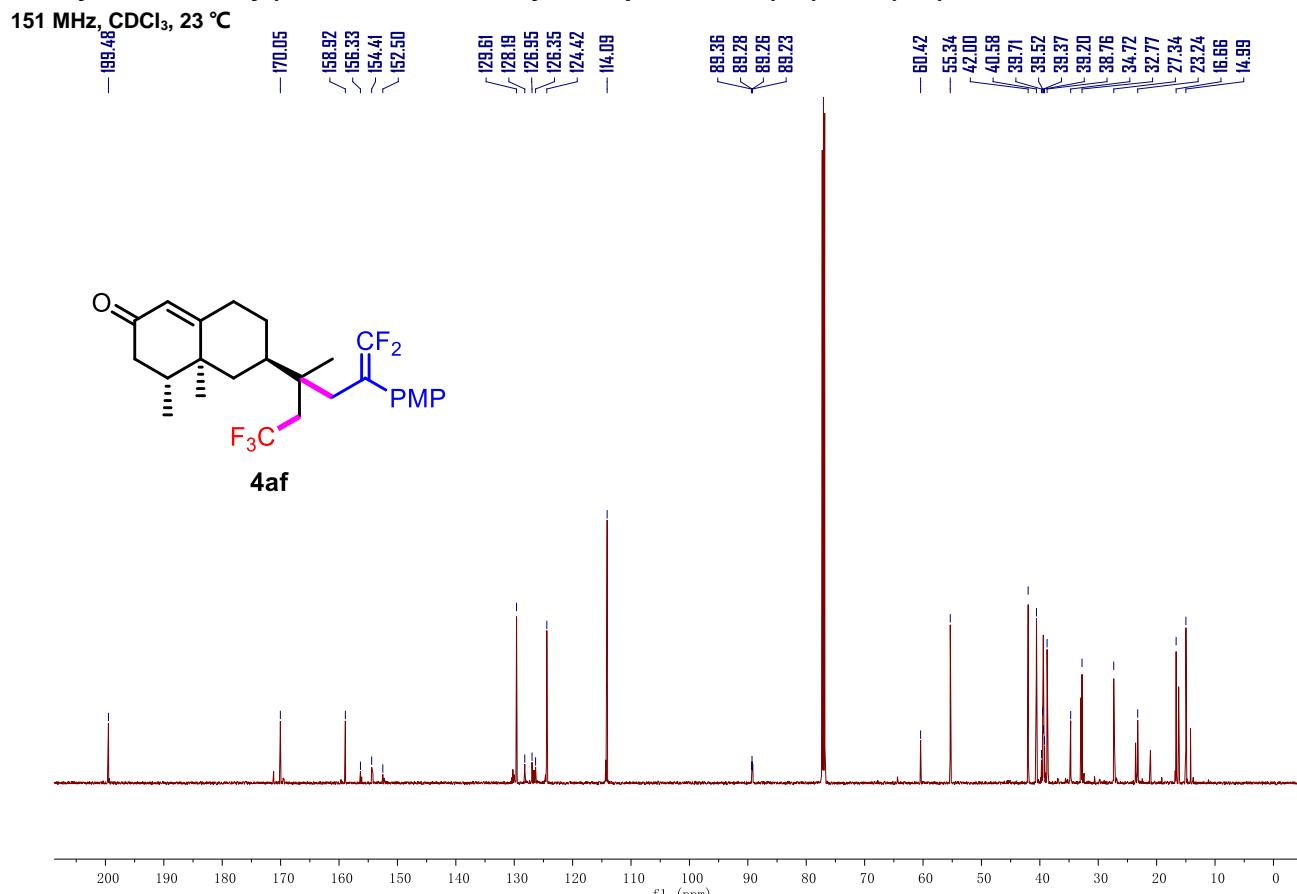


¹H NMR spectrum of (4R,4aS,6R)-4,4a-dimethyl-6-(1,1,1,6,6,6-pentafluoro-5-(4-methoxyphenyl)-3-methylhex-5-en-3-yl)-4,4a,5,6,7,8-hexahydronaphthalen-2(3H)-one (4af)

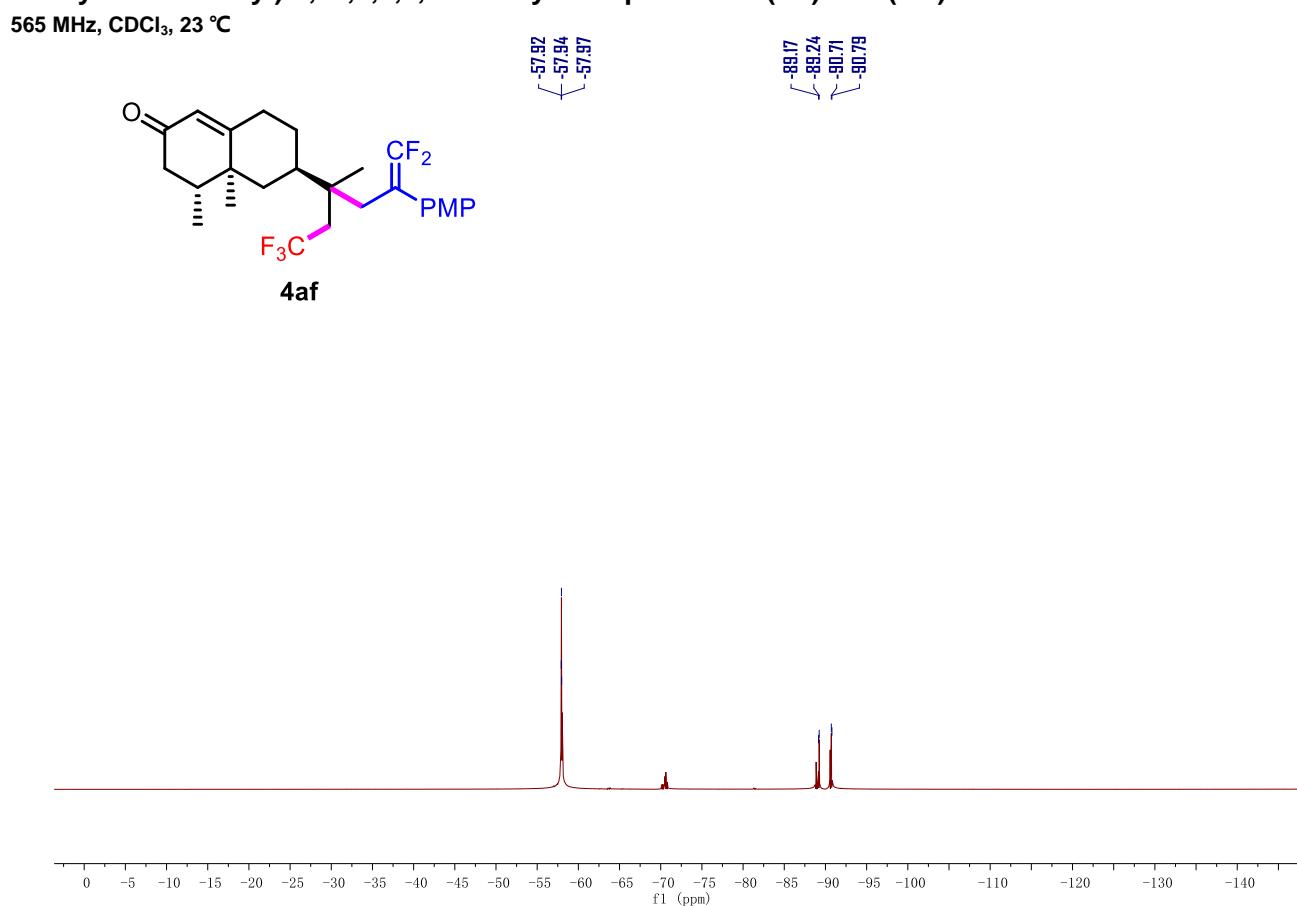
600 MHz, CDCl₃, 23 °C



¹³C NMR spectrum of (4R,4aS,6R)-4,4a-dimethyl-6-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)-3-methylhex-5-en-3-yl)-4,4a,5,6,7,8-hexahydronaphthalen-2(3H)-one (4af)

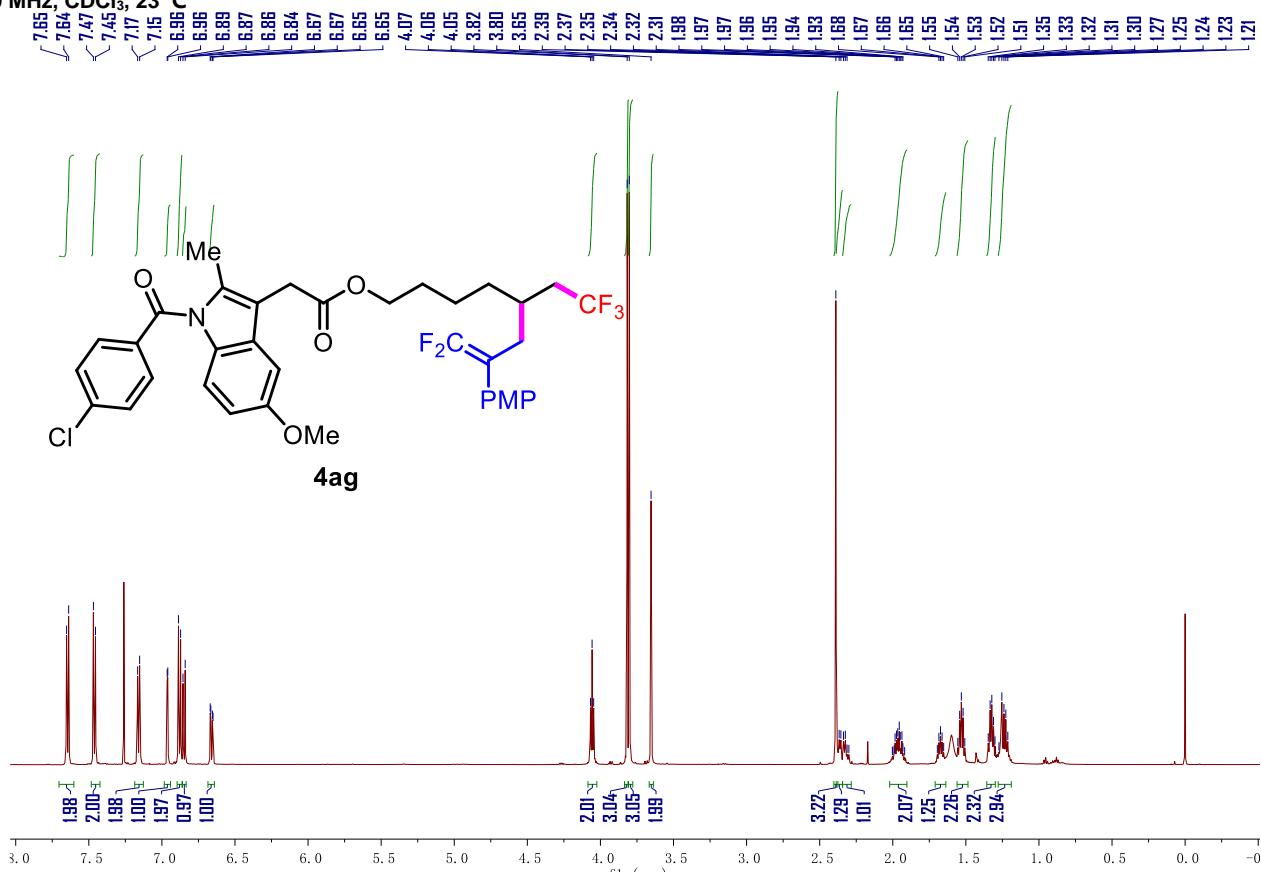


¹⁹F NMR spectrum of (4R,4aS,6R)-4,4a-dimethyl-6-(1,1,1,6,6-pentafluoro-5-(4-methoxyphenyl)-3-methylhex-5-en-3-yl)-4,4a,5,6,7,8-hexahydronaphthalen-2(3H)-one (4af)



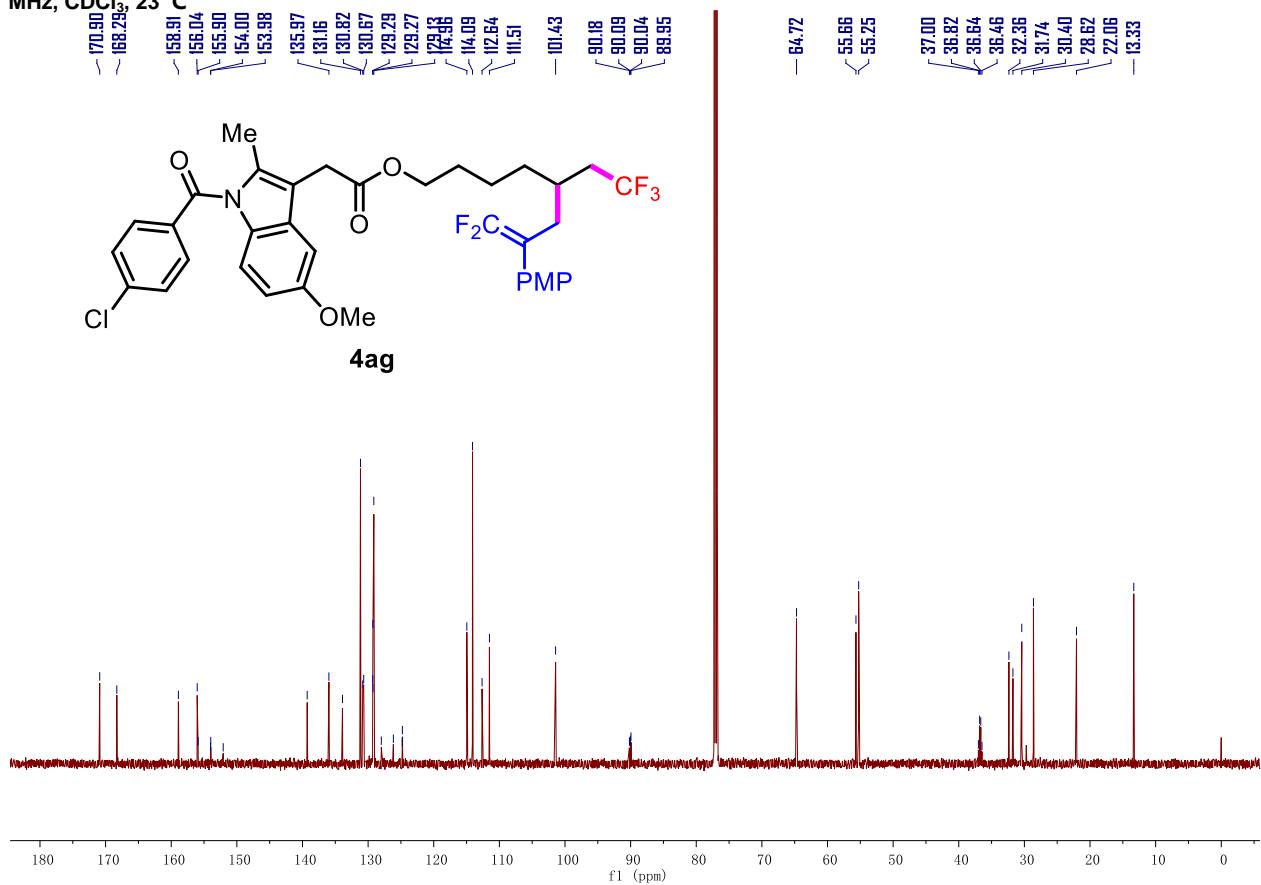
¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (4ag)

600 MHz, CDCl₃, 23 °C



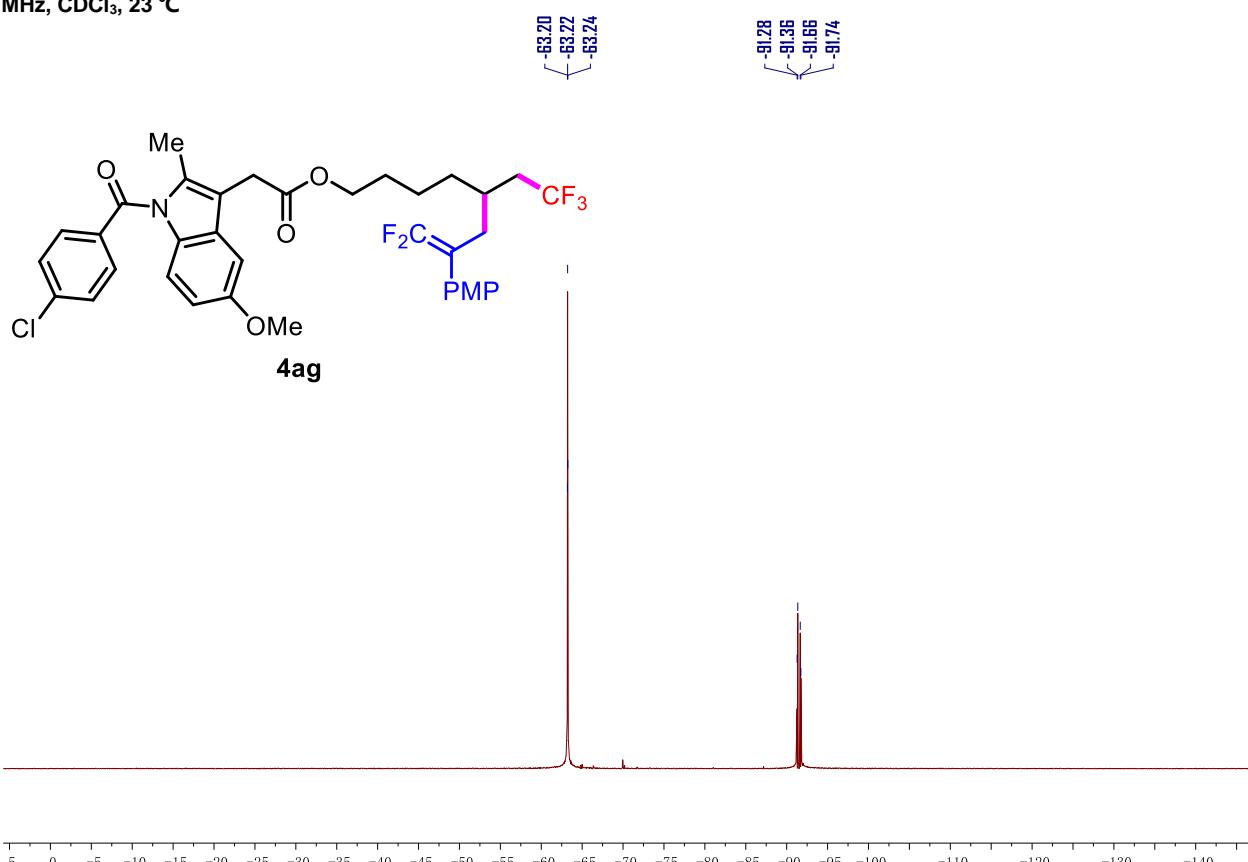
¹³C NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (4ag)

151 MHz, CDCl₃, 23 °C



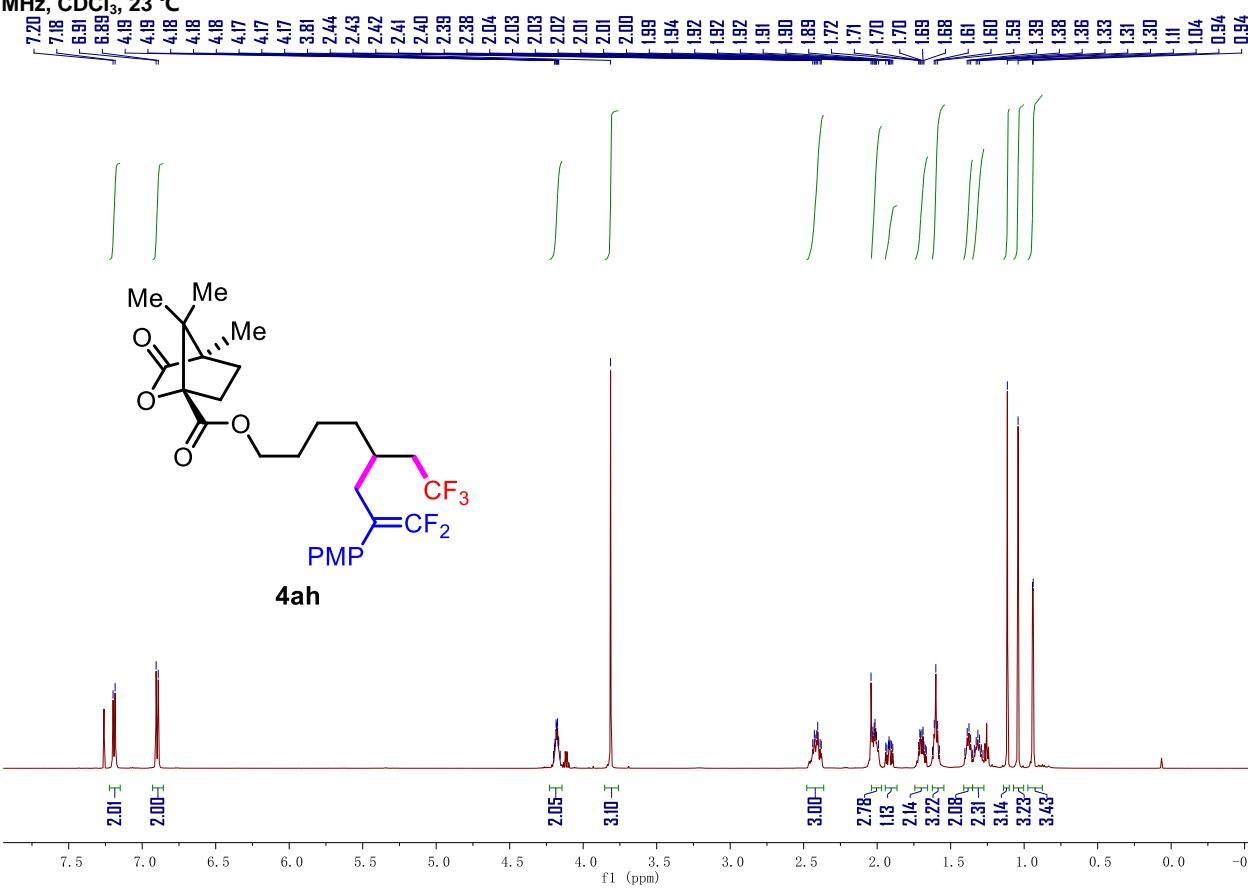
¹⁹F NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (4ag)

565 MHz, CDCl₃, 23 °C

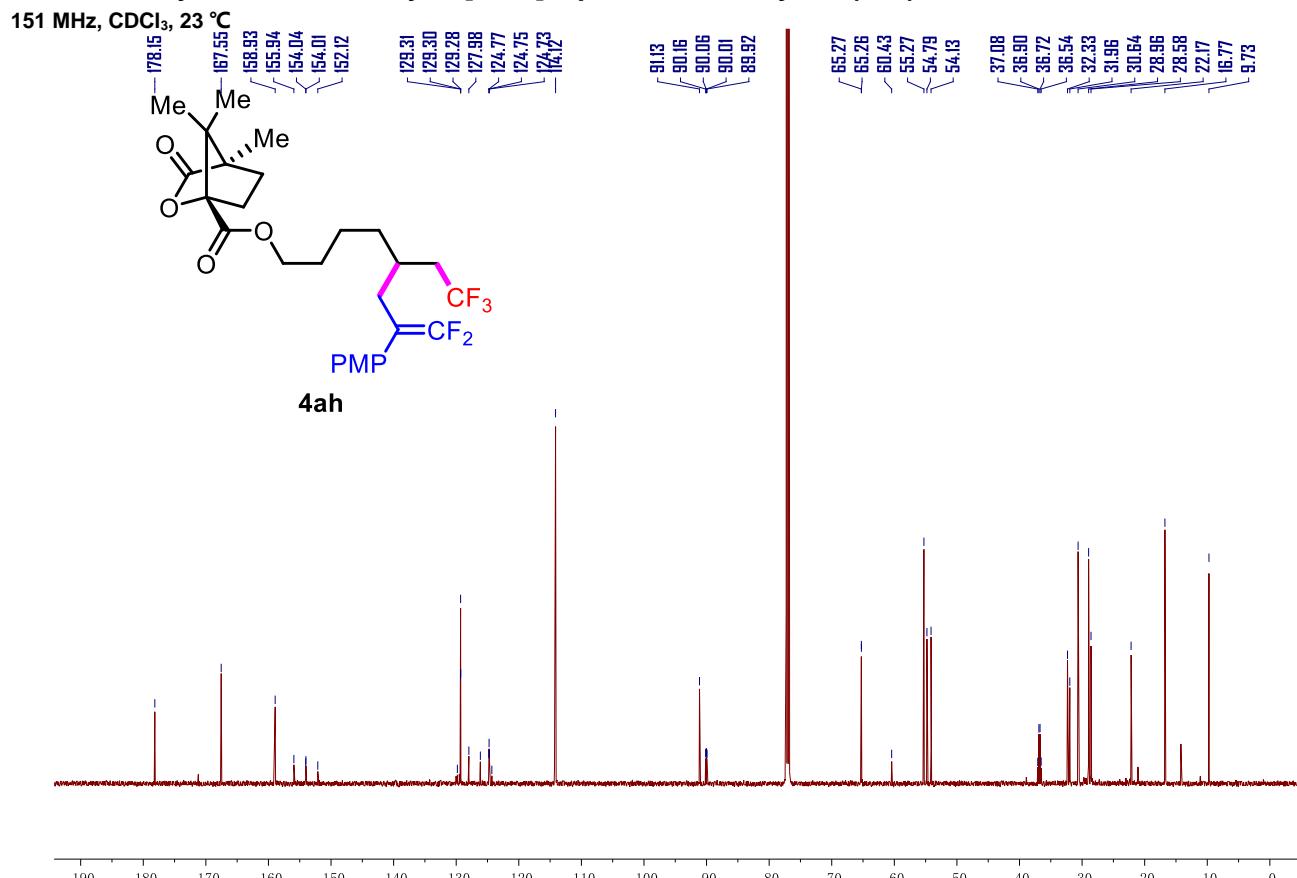


¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl (1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (4ah)

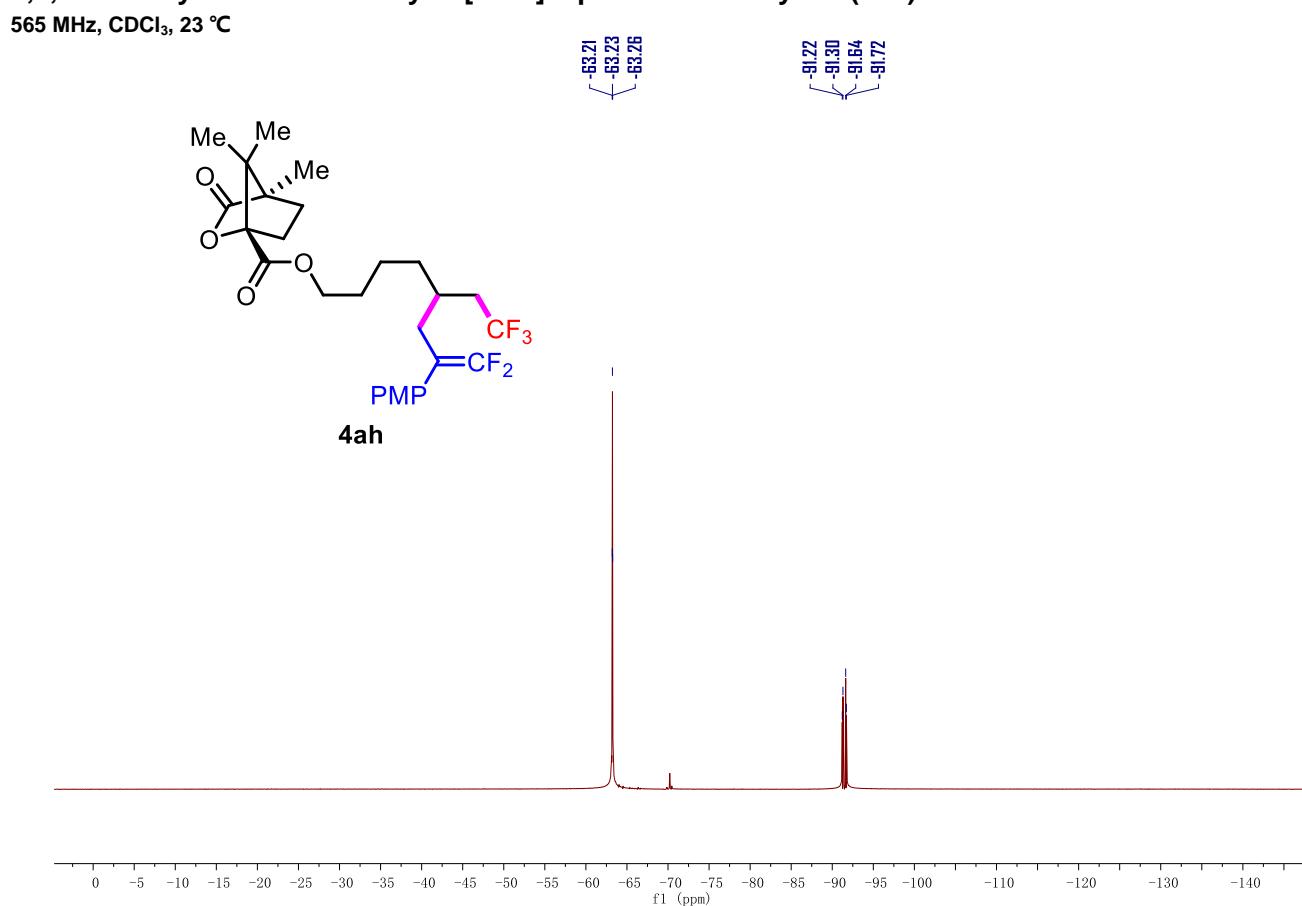
600 MHz, CDCl₃, 23 °C



¹³C NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl (1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (4ah)

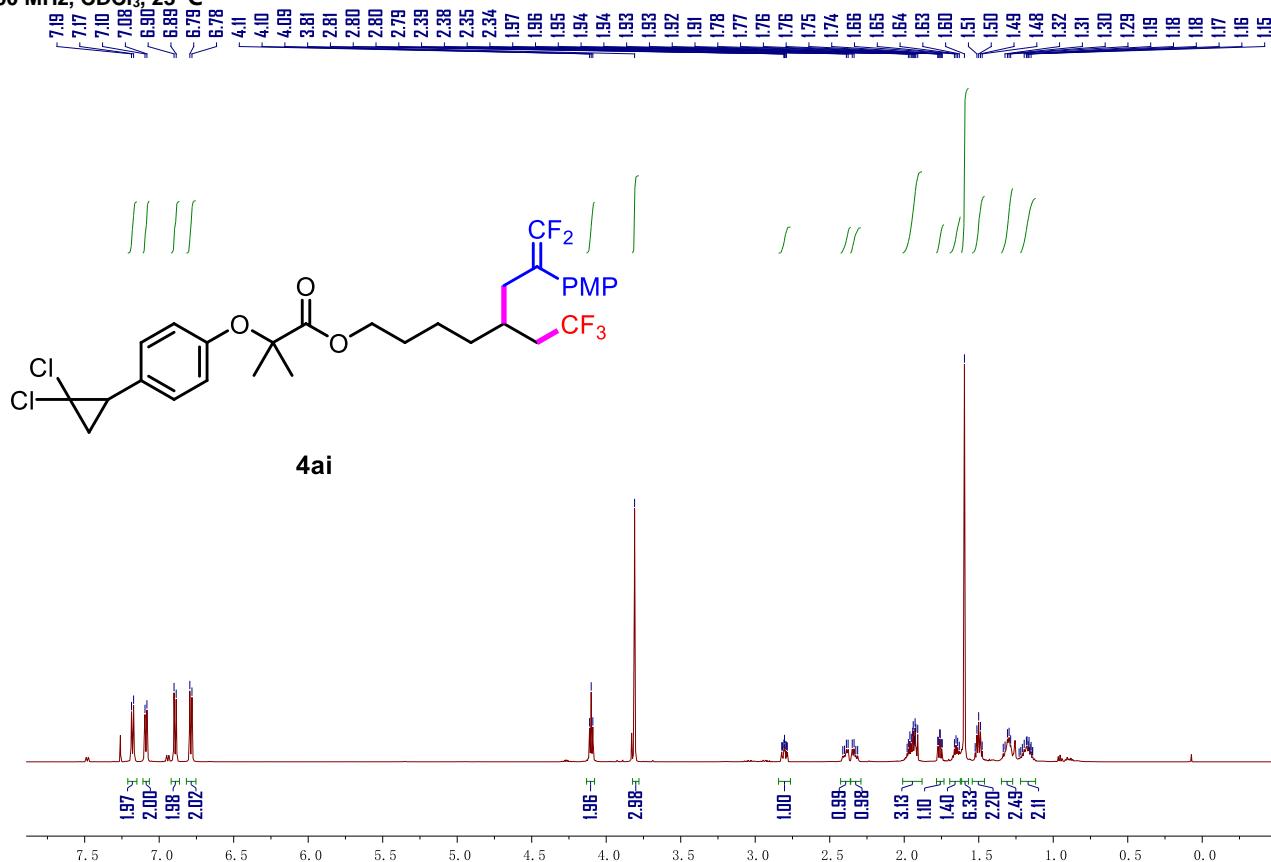


¹⁹F NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl (1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (4ah)

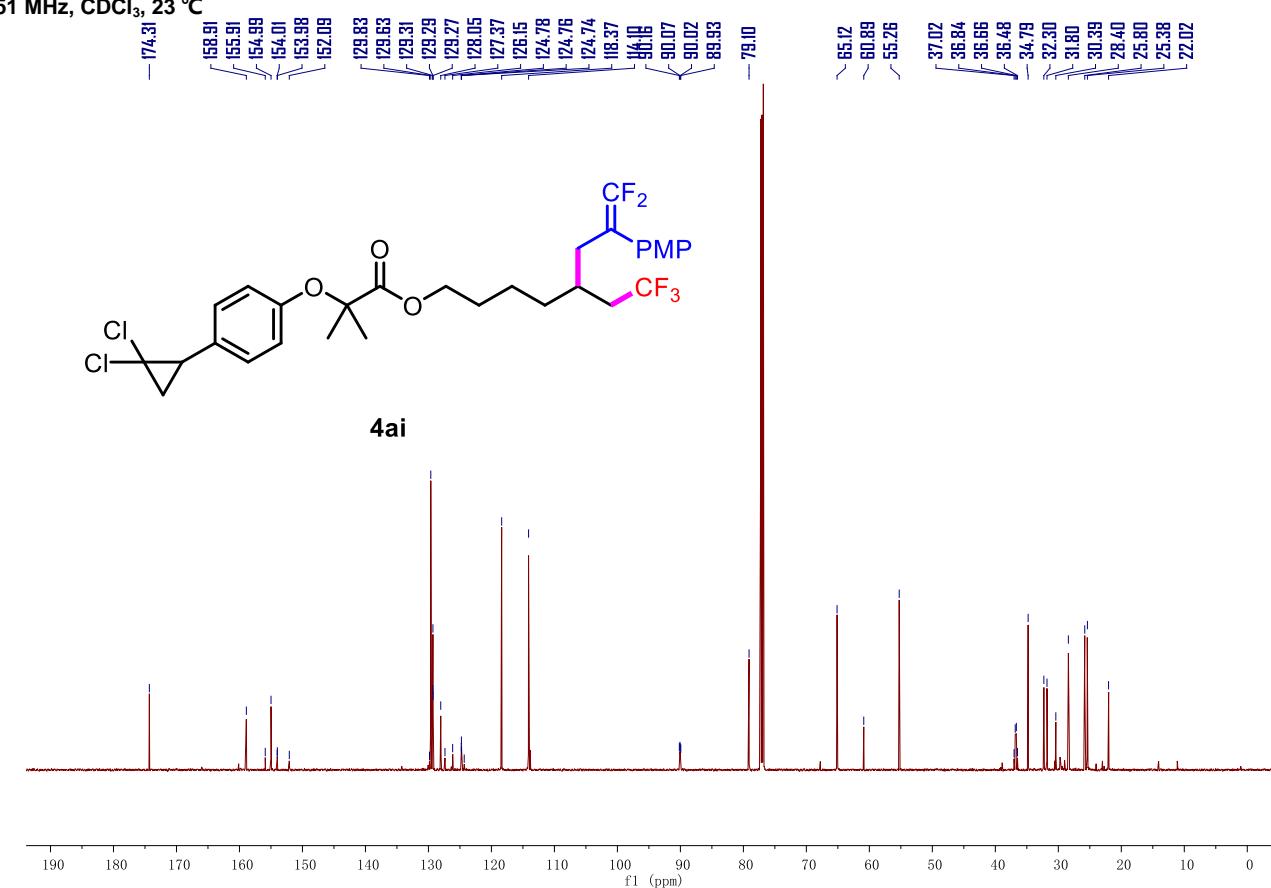


¹H NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (4ai)

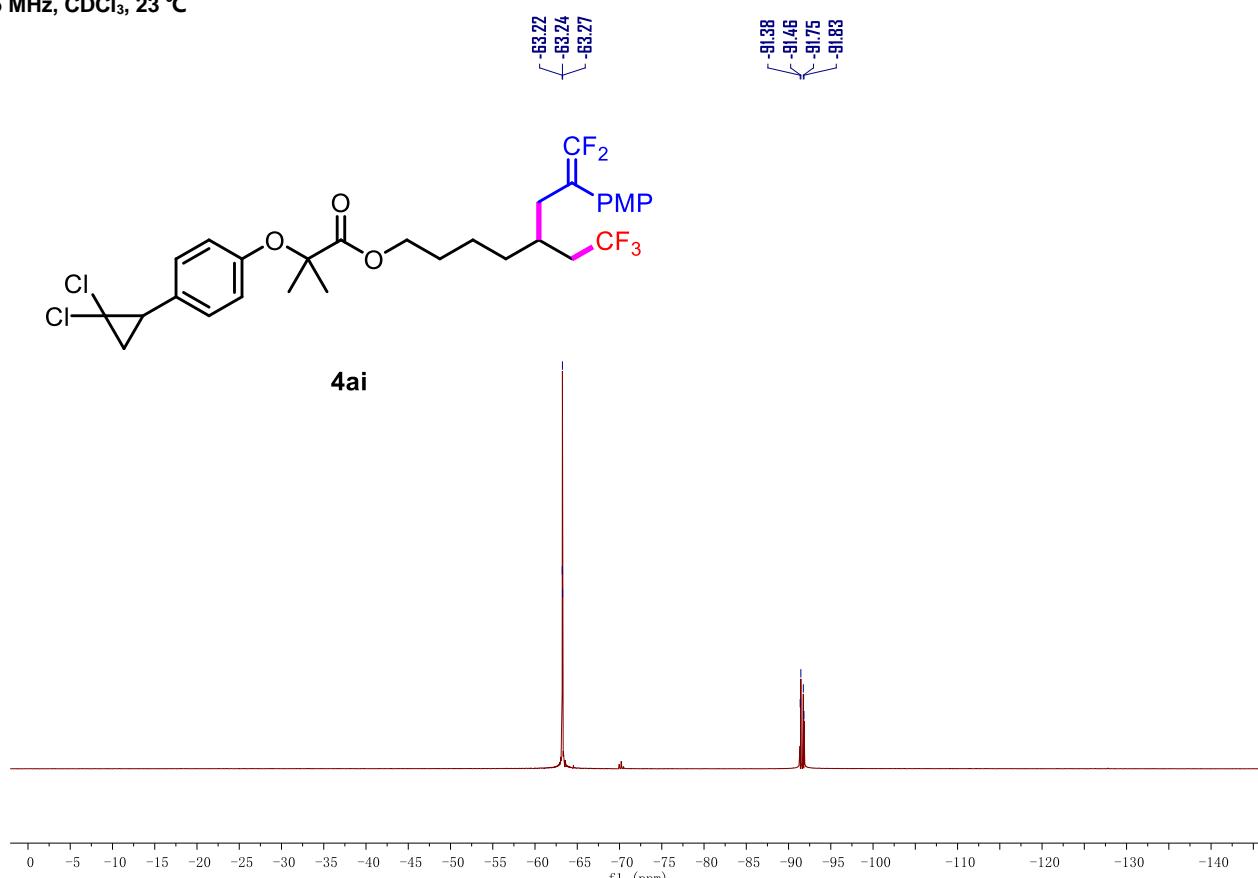
600 MHz, CDCl₃, 23 °C



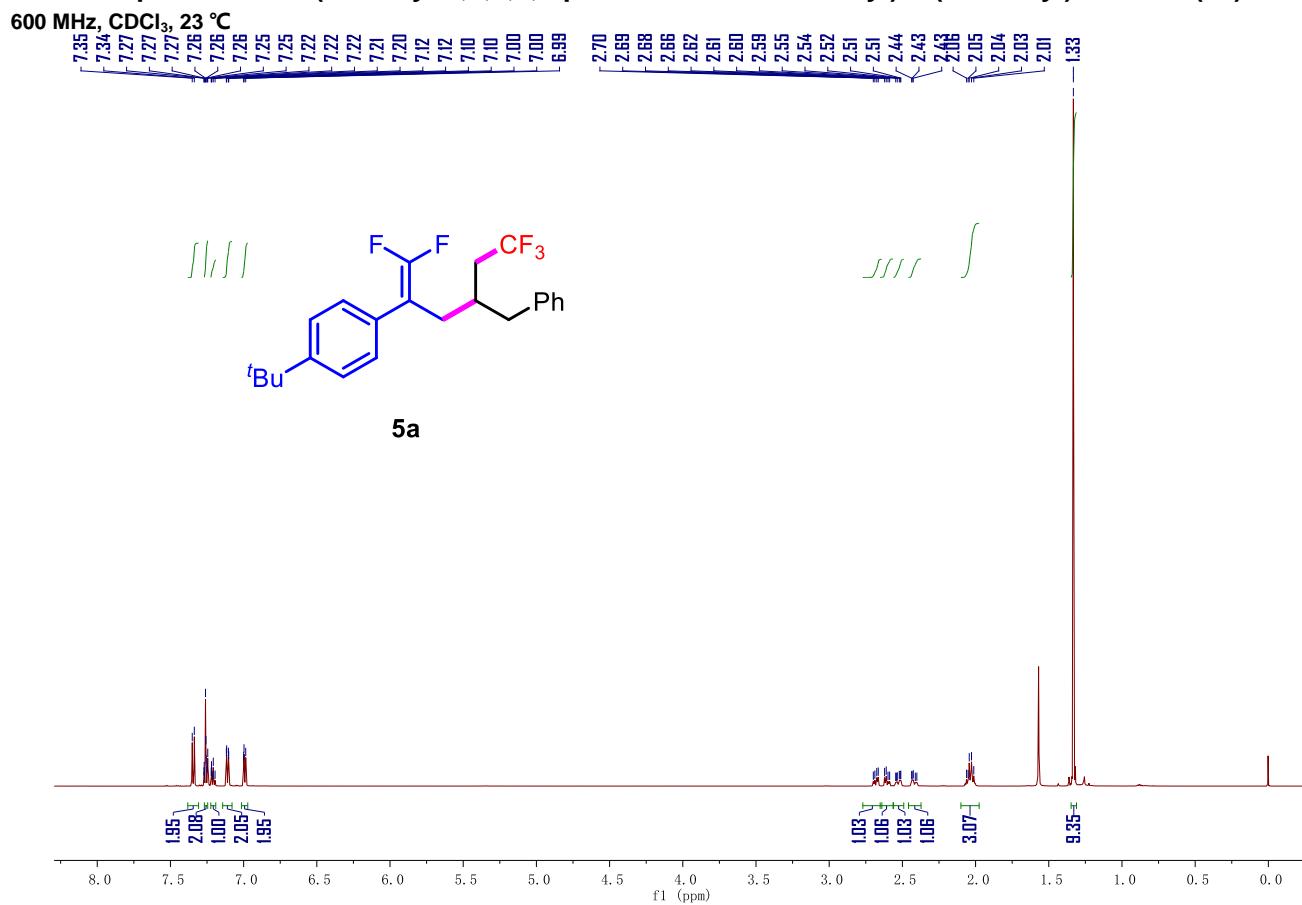
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 8,8-difluoro-7-(4-methoxyphenyl)-5-(2,2,2-trifluoroethyl)oct-7-en-1-yl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (4ai)
565 MHz, CDCl₃, 23 °C

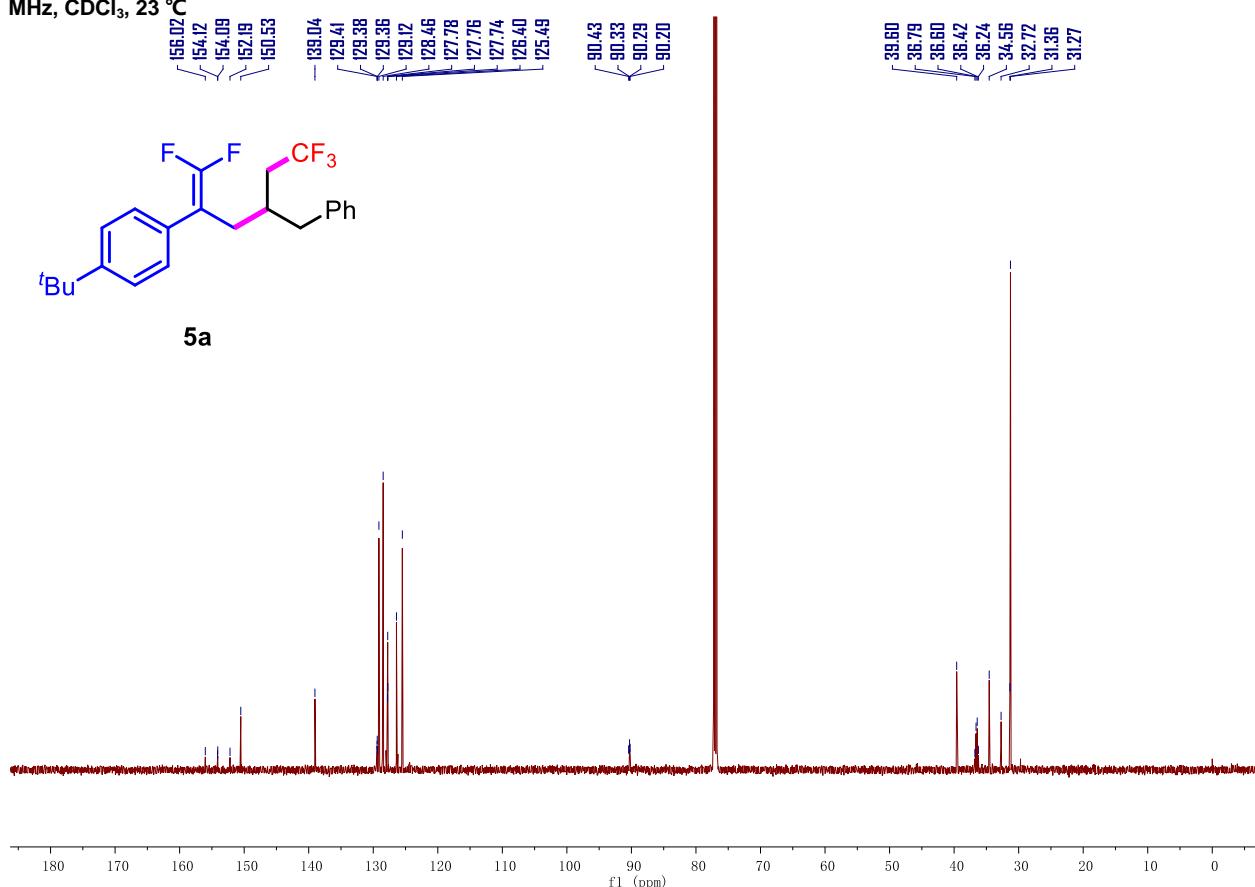


¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(tert-butyl)benzene (5a)



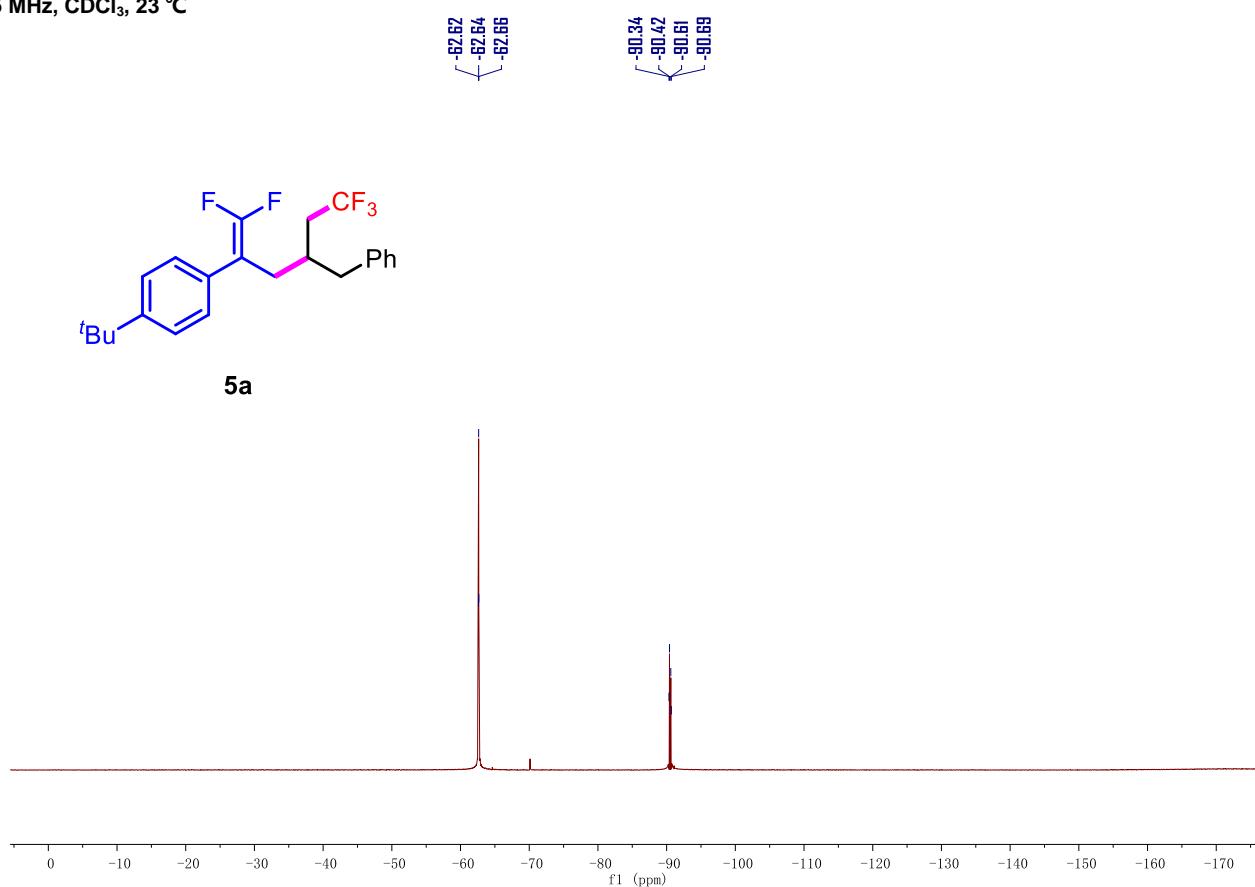
¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(tert-butyl)benzene (5a)

151 MHz, CDCl₃, 23 °C

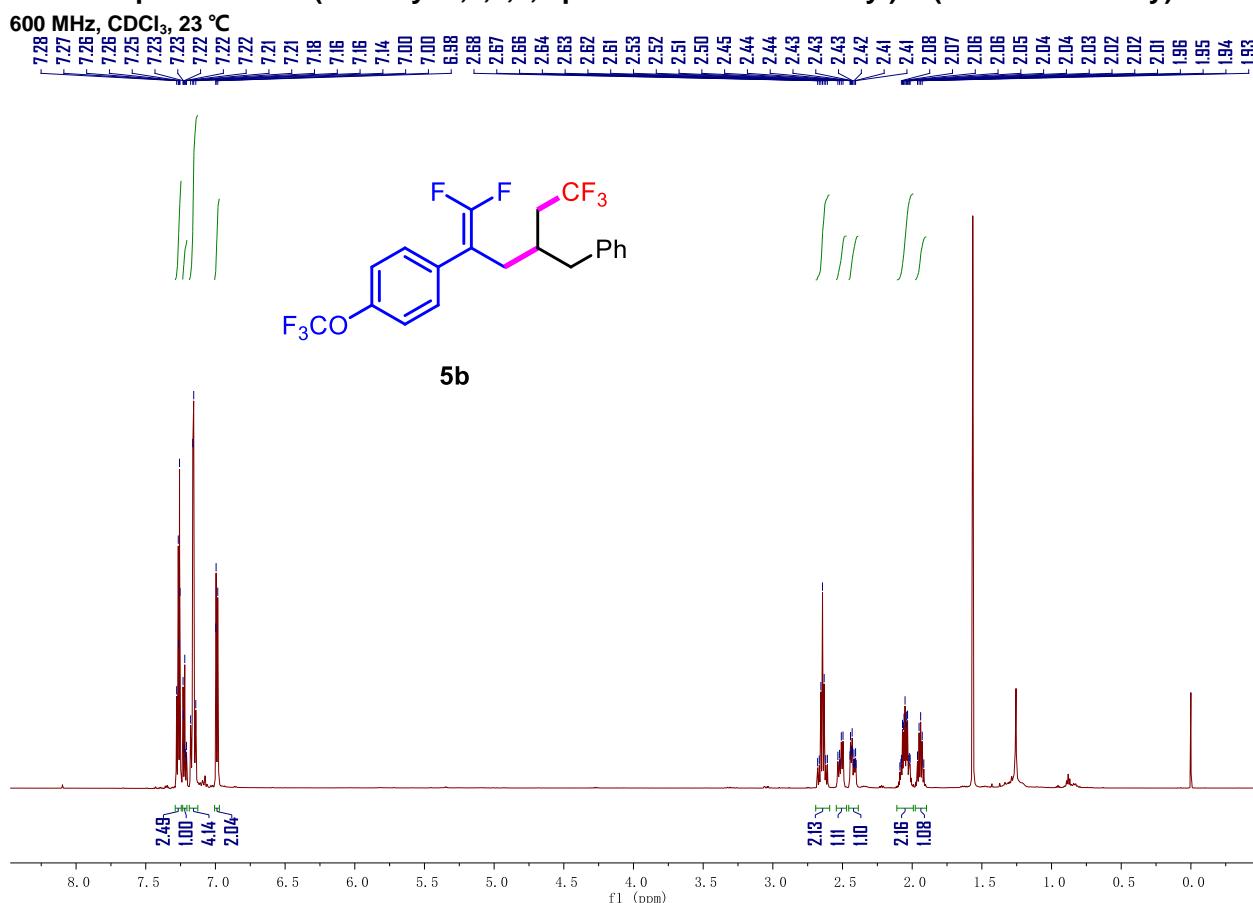


¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(tert-butyl)benzene (5a)

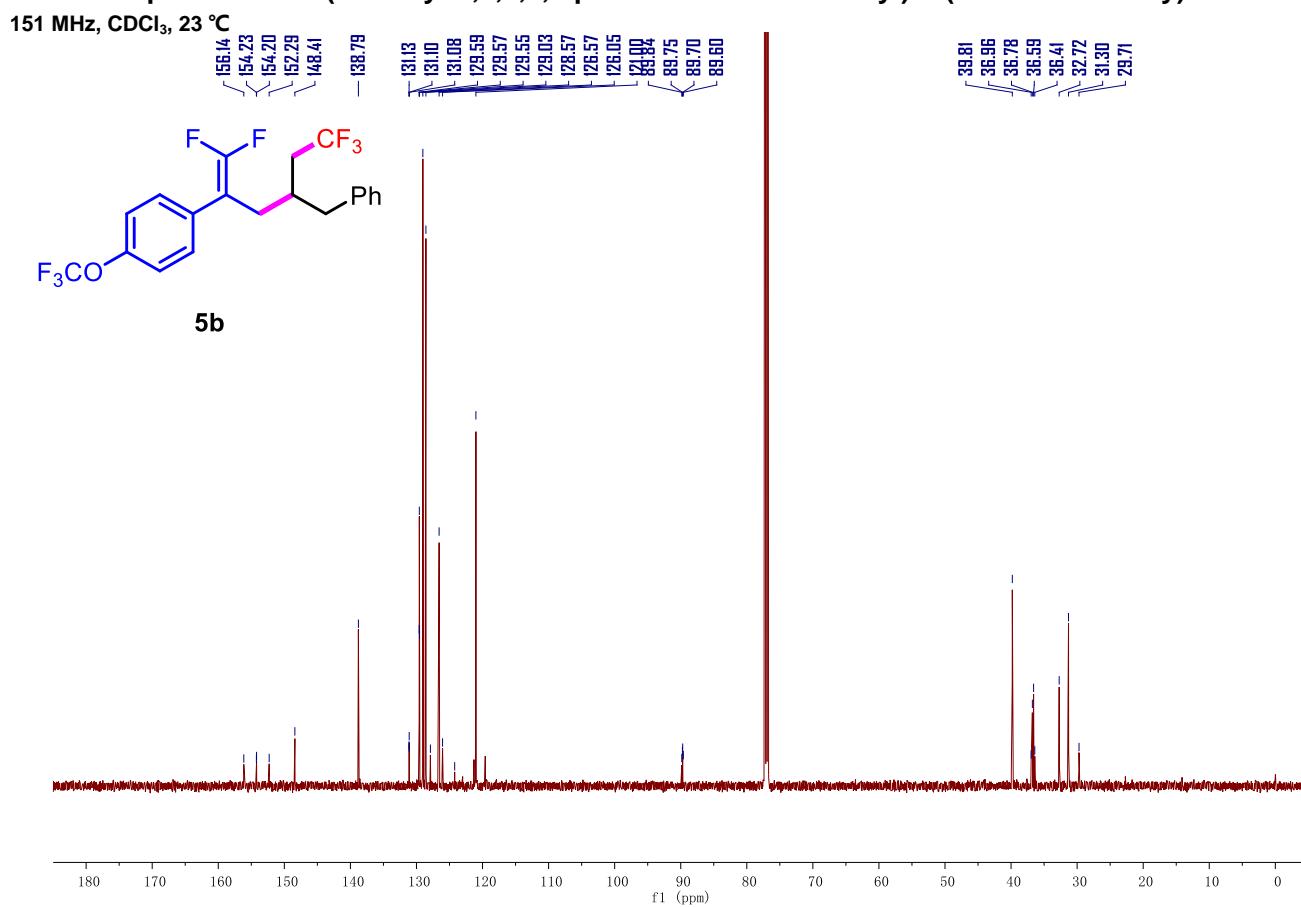
565 MHz, CDCl₃, 23 °C



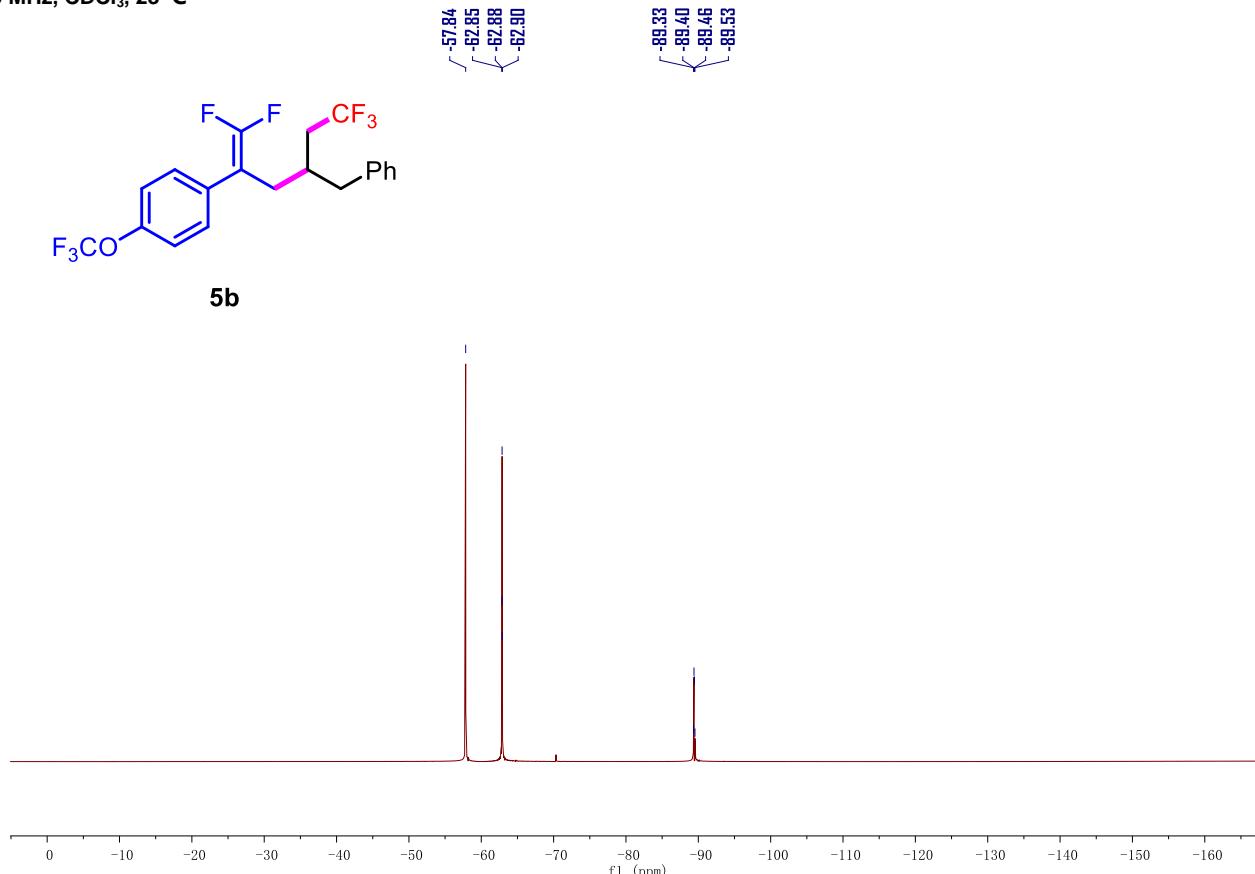
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(trifluoromethoxy)benzene (5b)



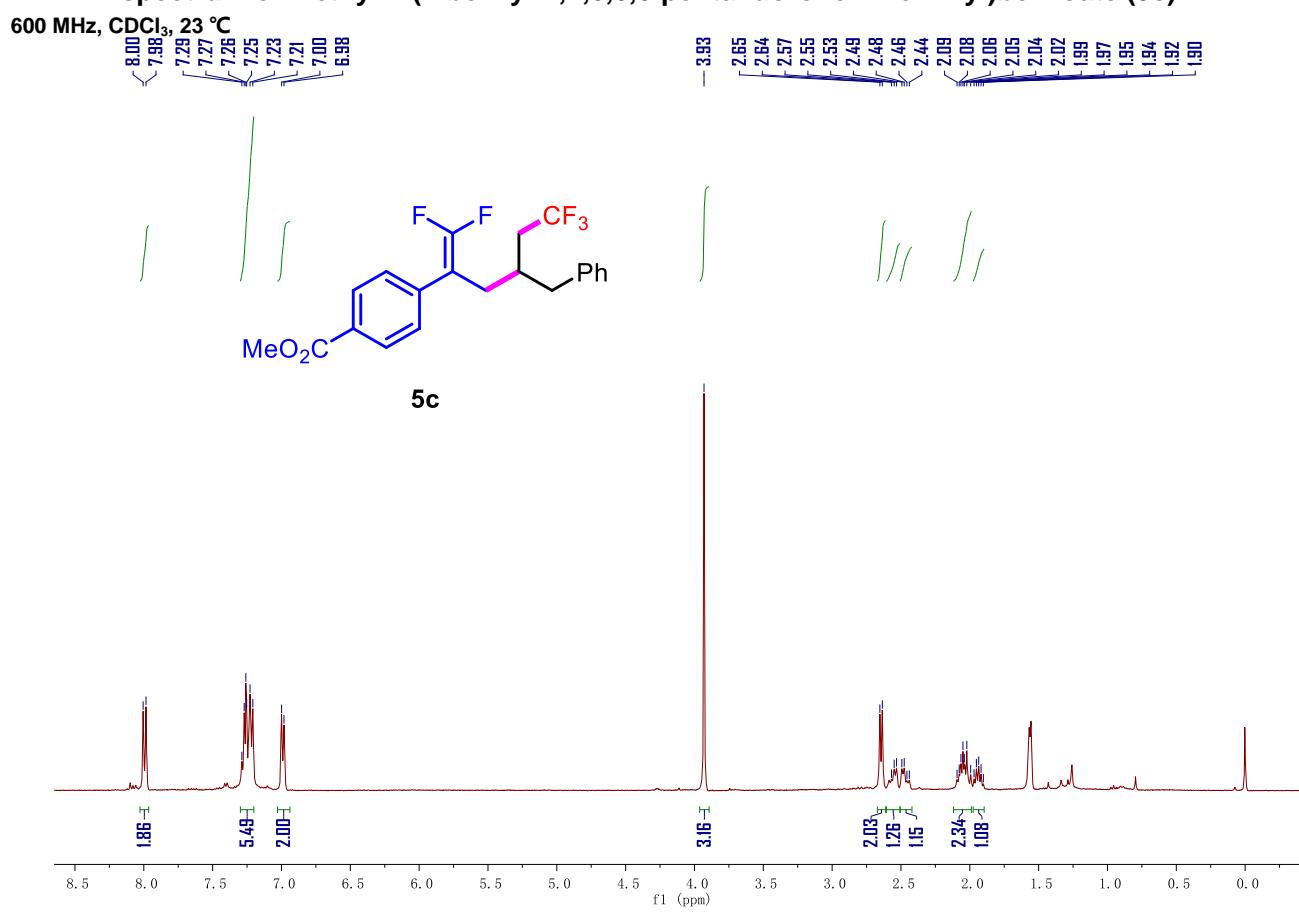
¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(trifluoromethoxy)benzene (5b)



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(trifluoromethoxy)benzene (5b)
565 MHz, CDCl₃, 23 °C

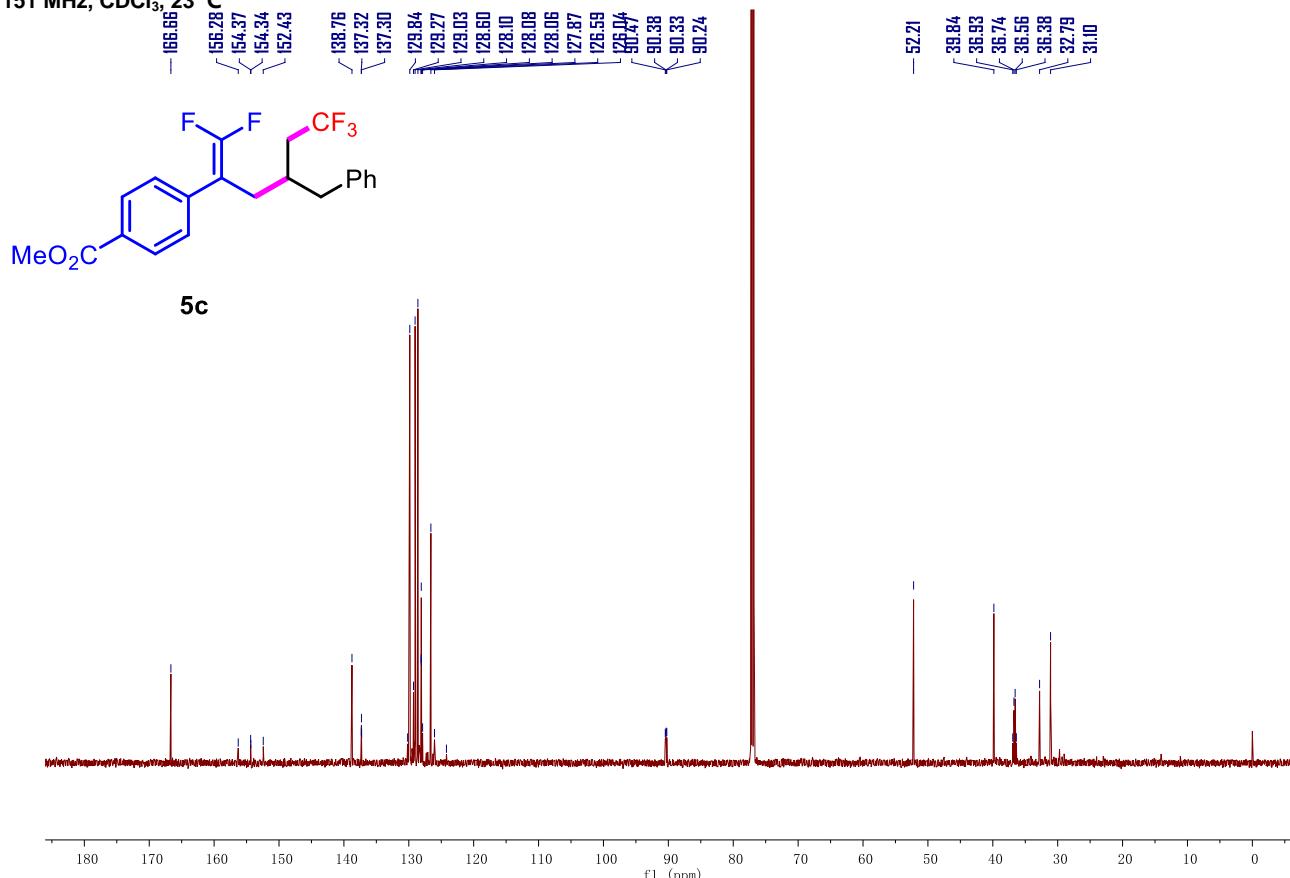


¹H NMR spectrum of methyl 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzoate (5c)



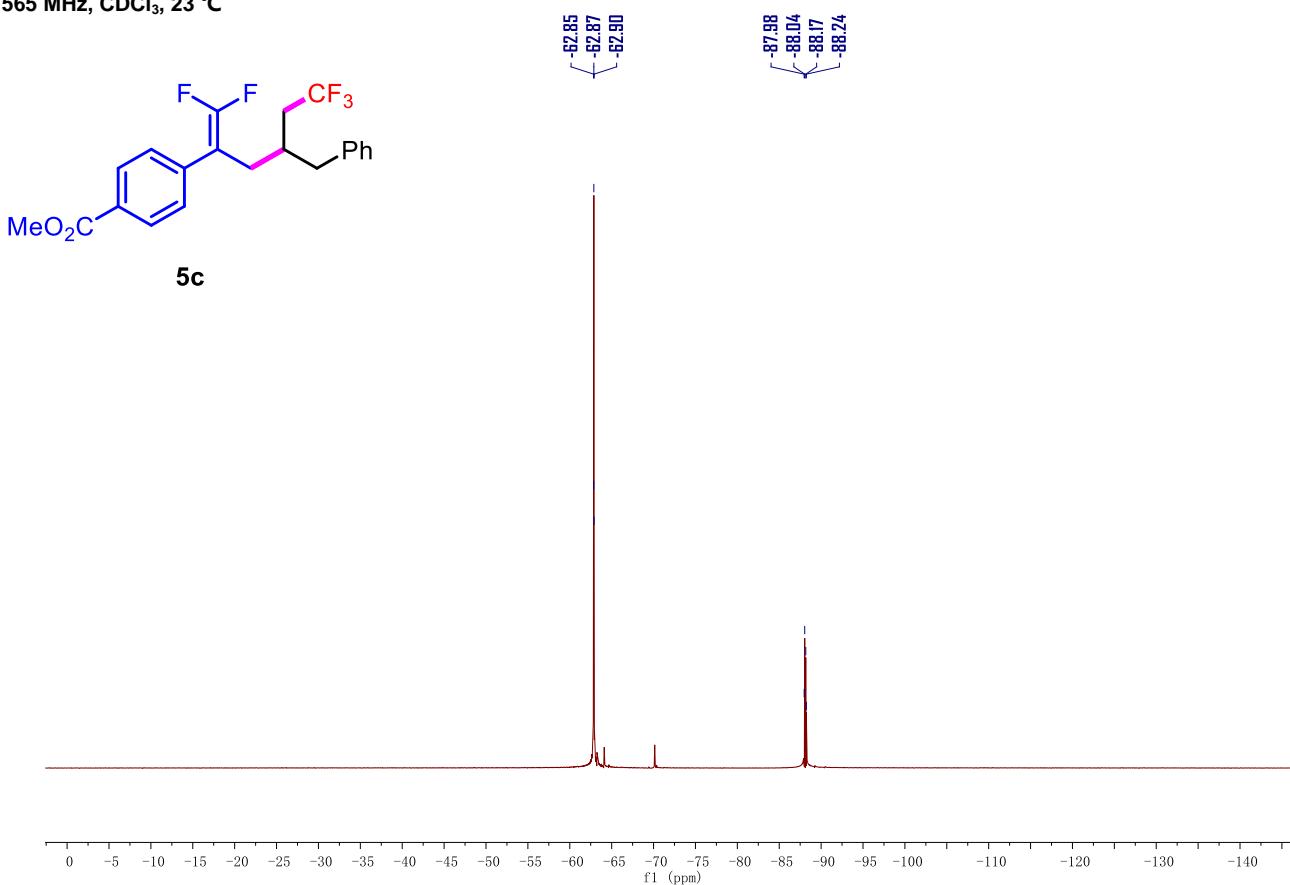
¹³C NMR spectrum of methyl 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzoate (5c)

151 MHz, CDCl₃, 23 °C



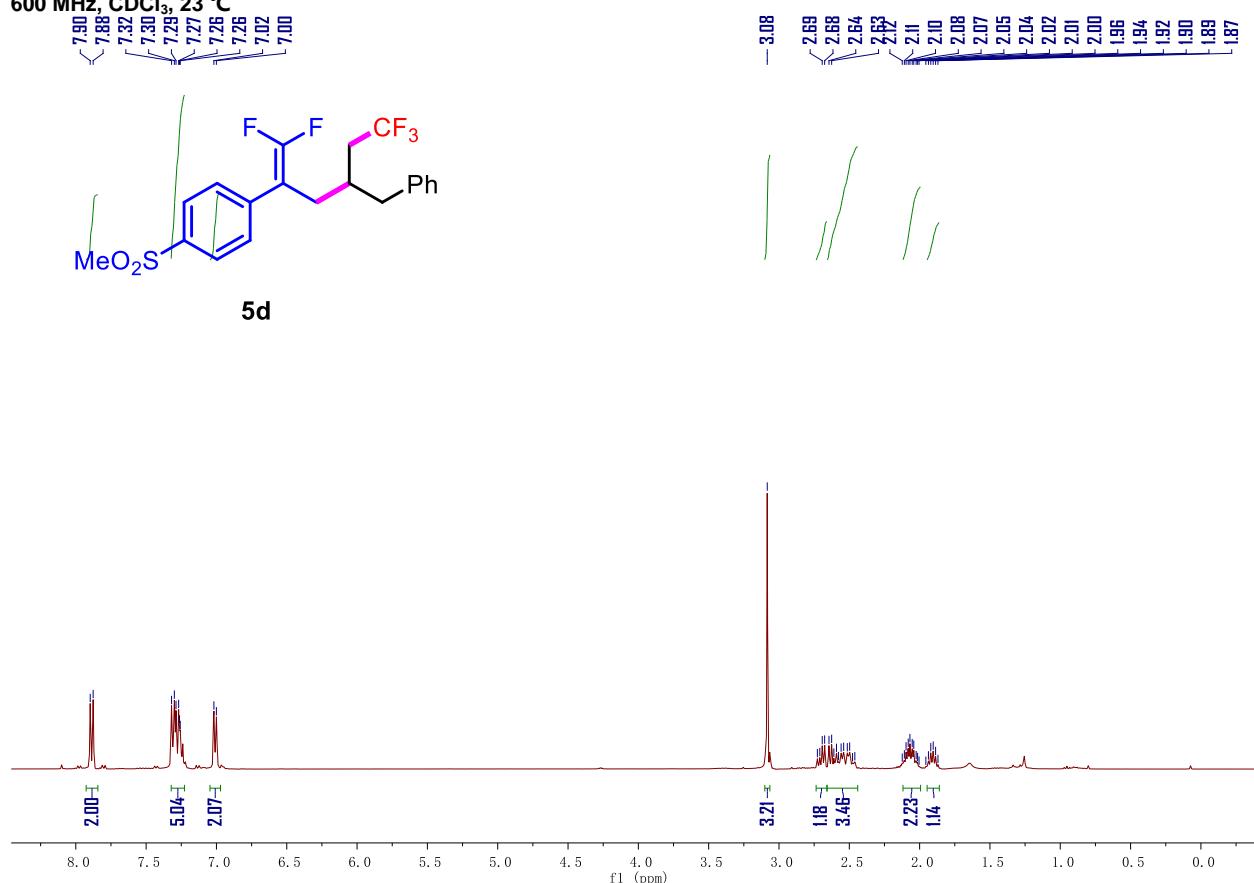
¹⁹F NMR spectrum of methyl 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzoate (5c)

565 MHz, CDCl₃, 23 °C



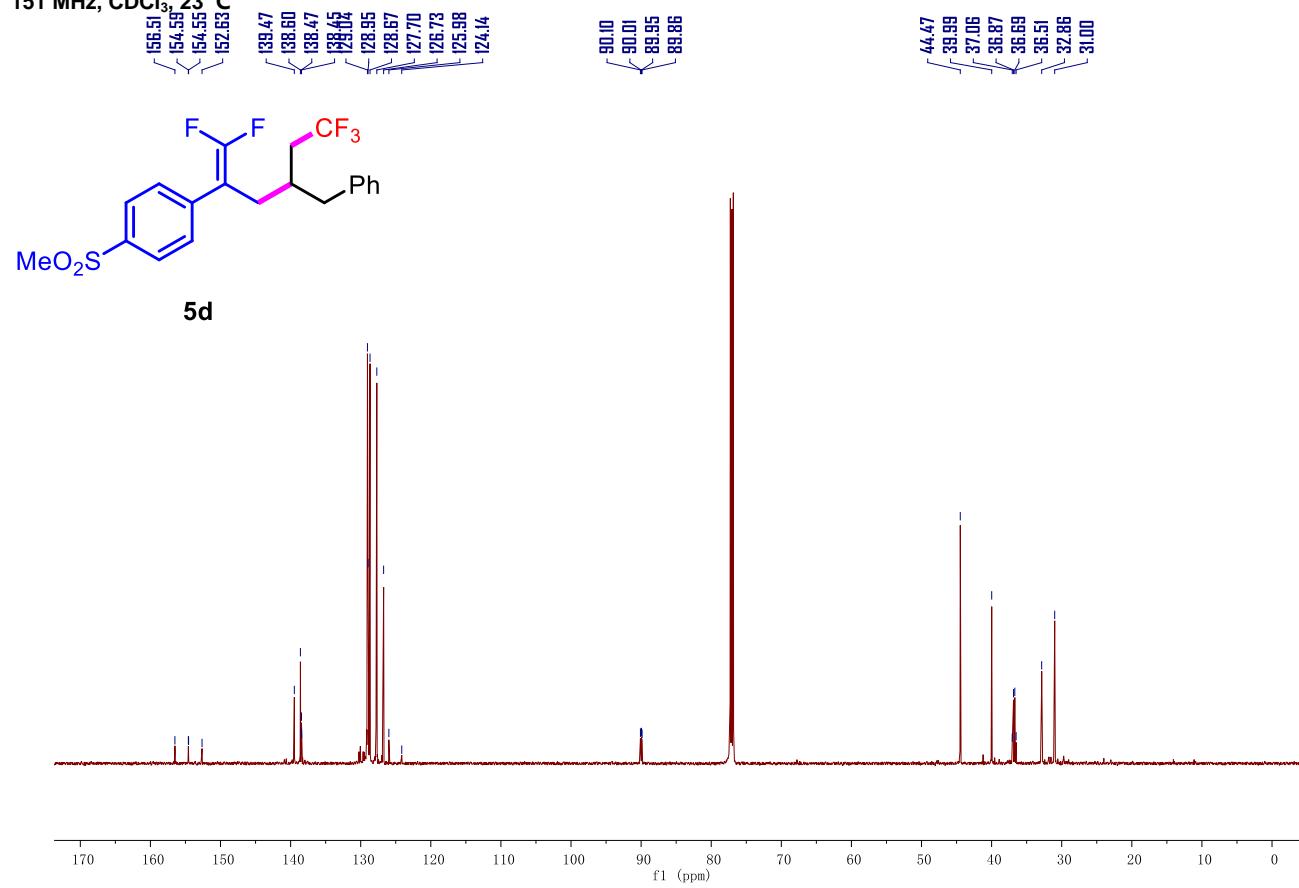
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(methylsulfonyl)benzene (5d)

600 MHz, CDCl₃, 23 °C

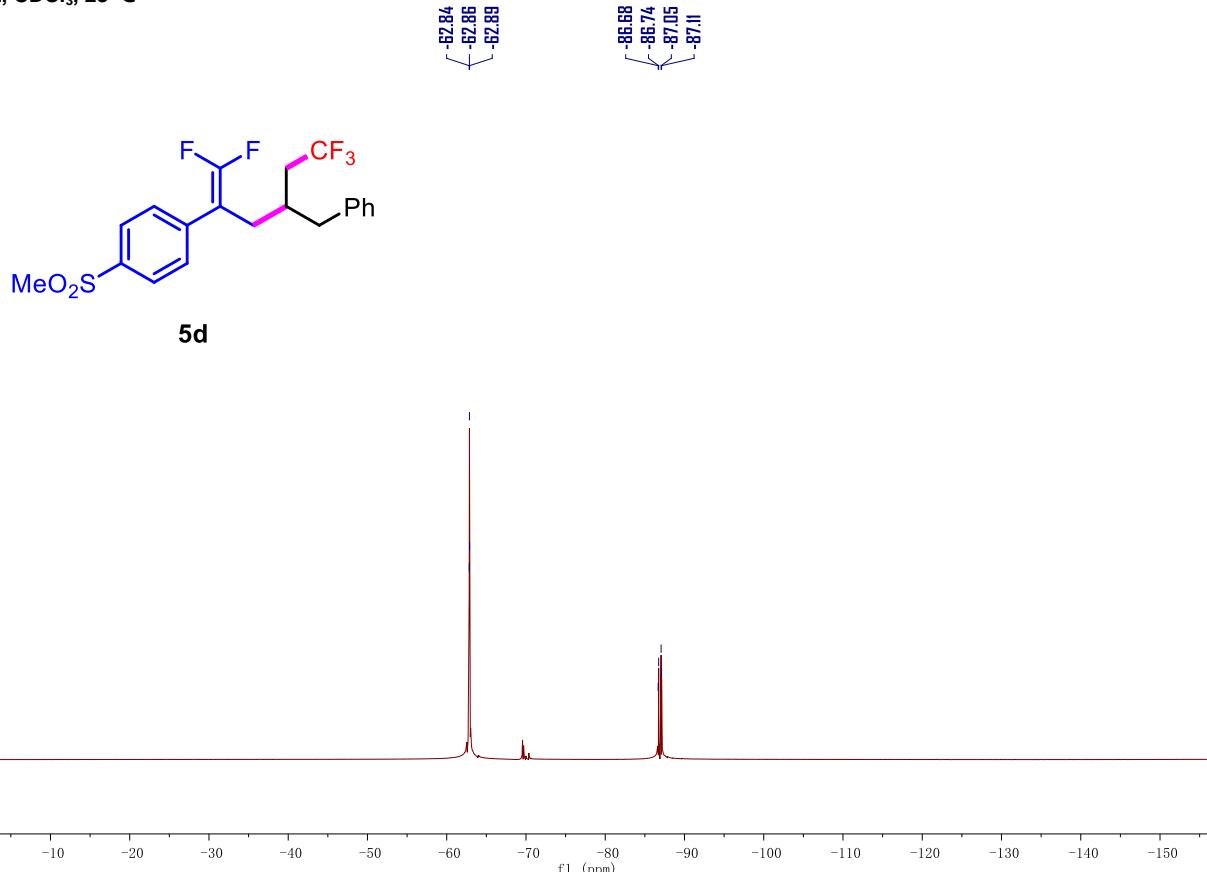


¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(methylsulfonyl)benzene (5d)

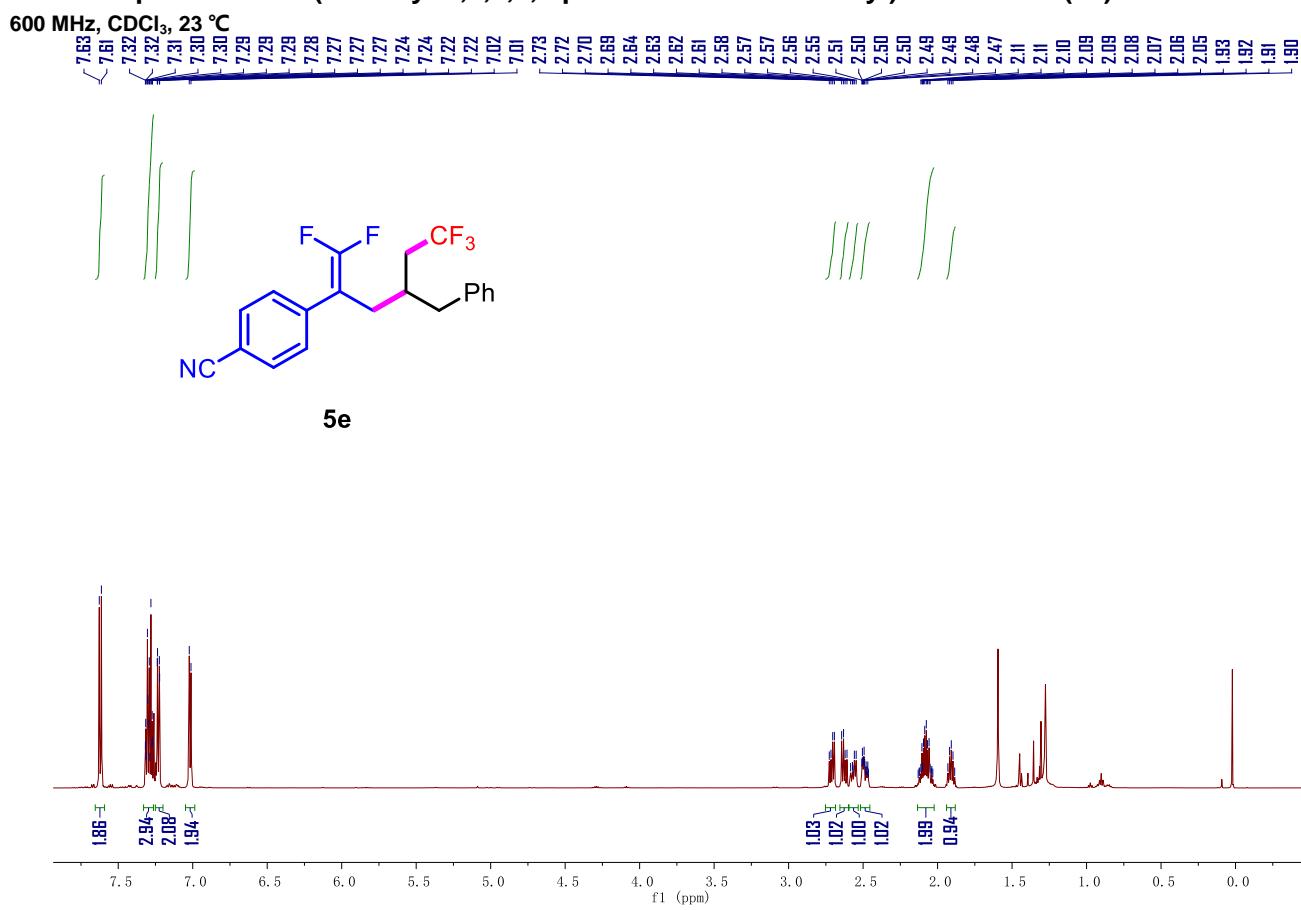
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-(methylsulfonyl)benzene (5d)
565 MHz, CDCl₃, 23 °C

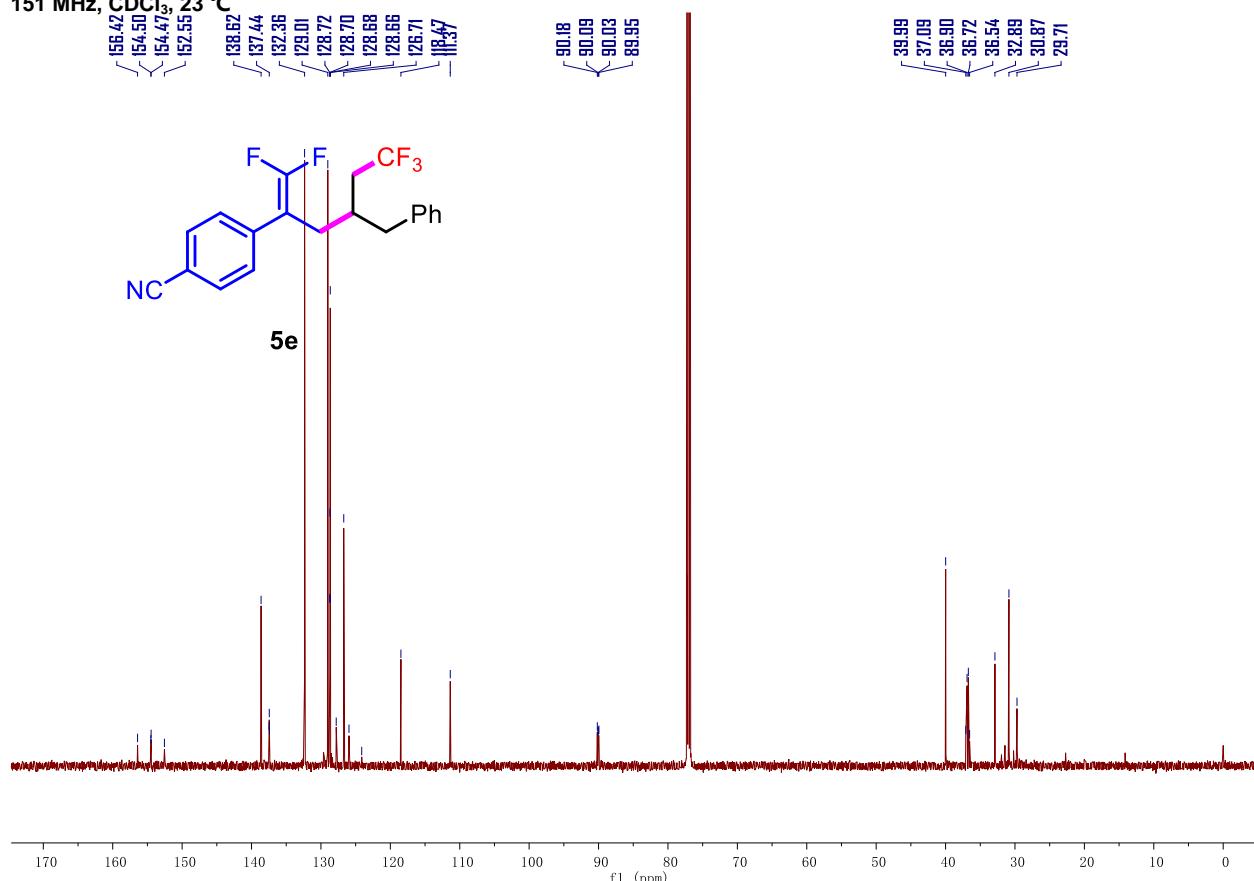


¹H NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzonitrile (5e)



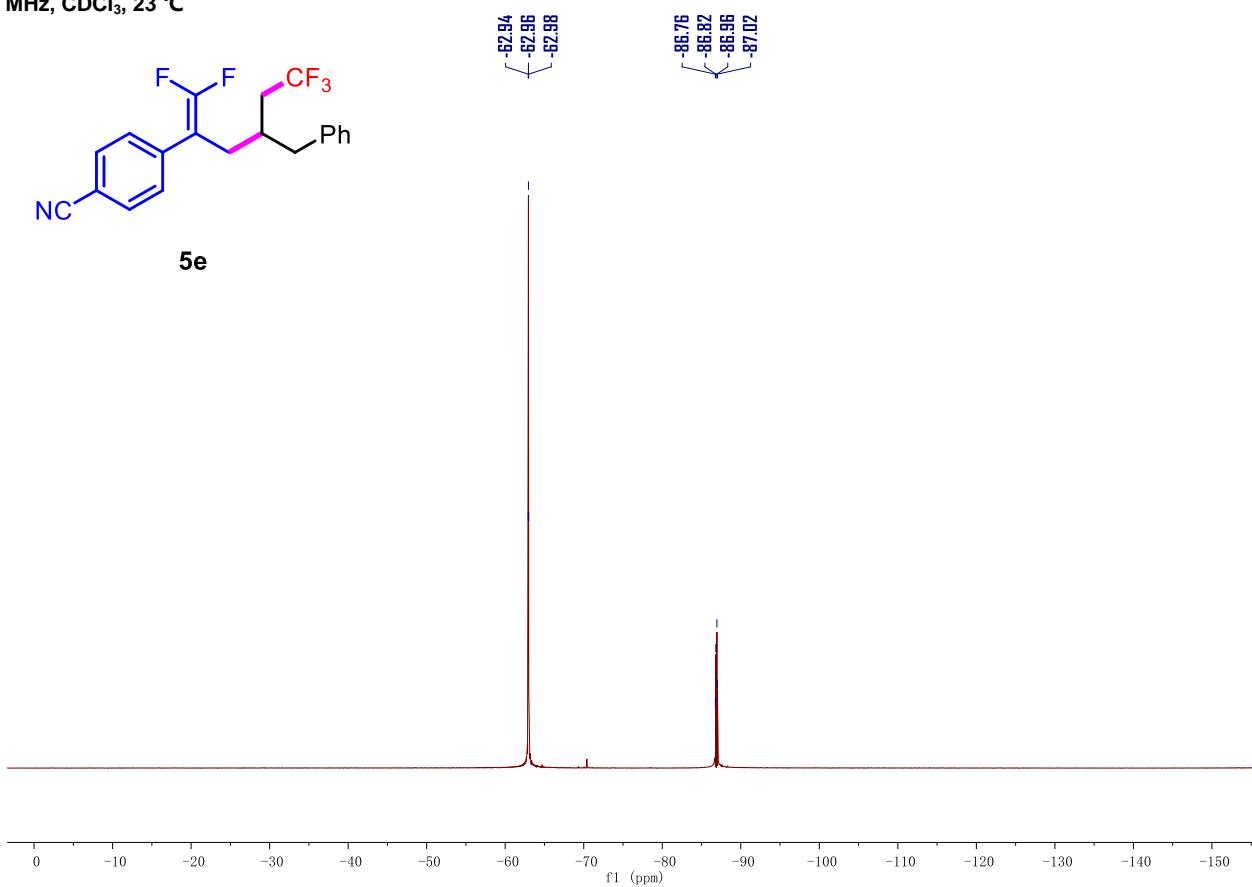
¹³C NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzonitrile (5e)

151 MHz, CDCl₃, 23 °C

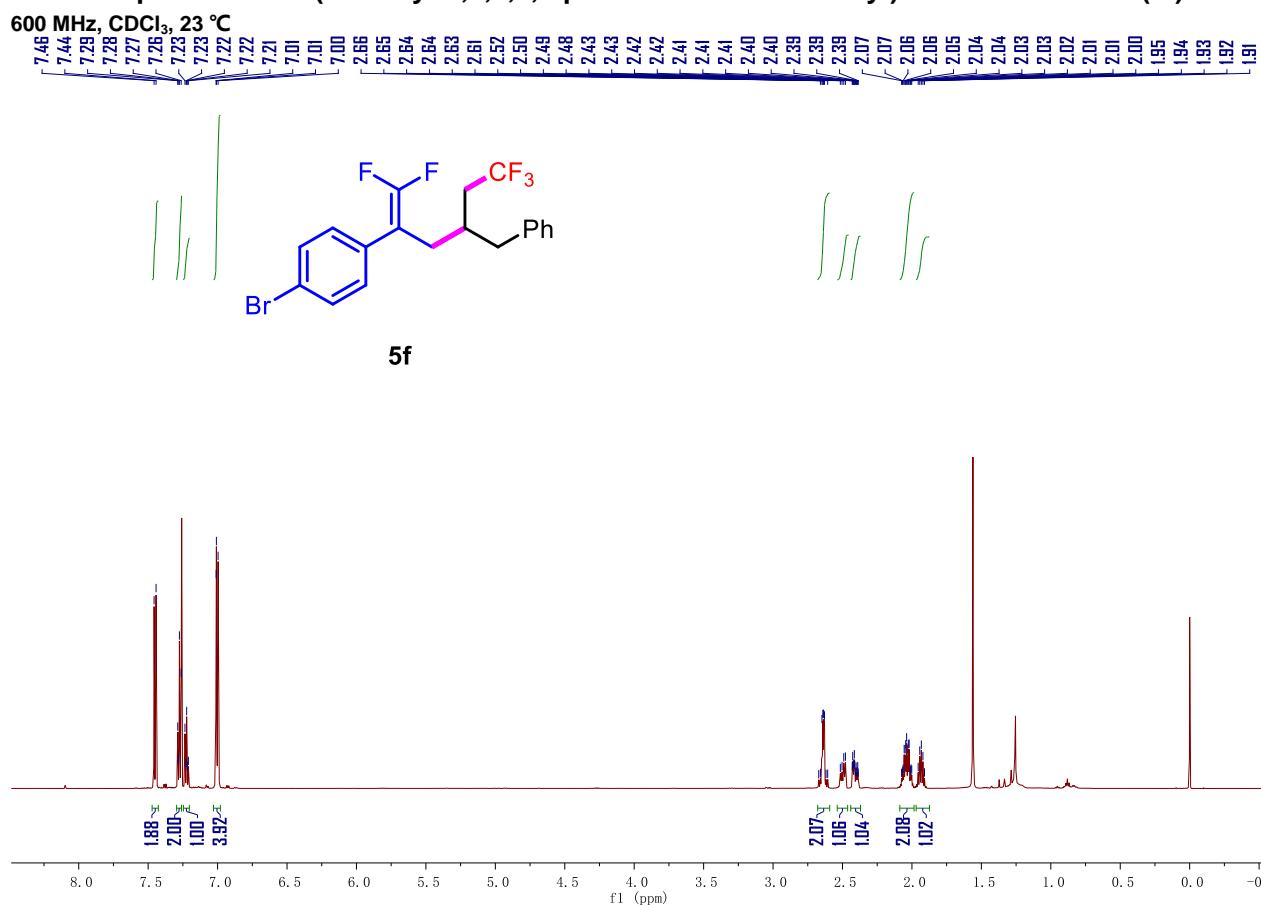


¹⁹F NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzonitrile (5e)

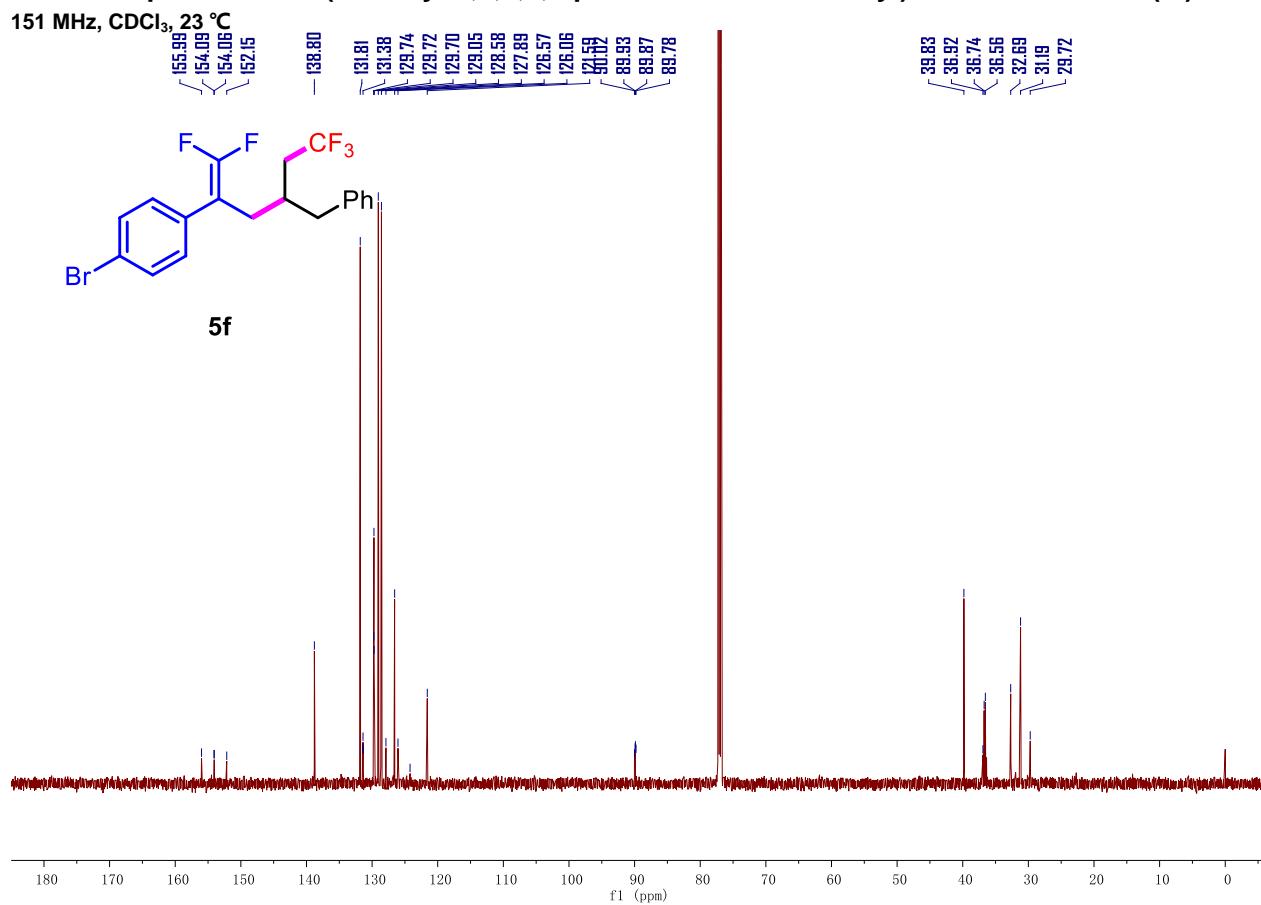
565 MHz, CDCl₃, 23 °C



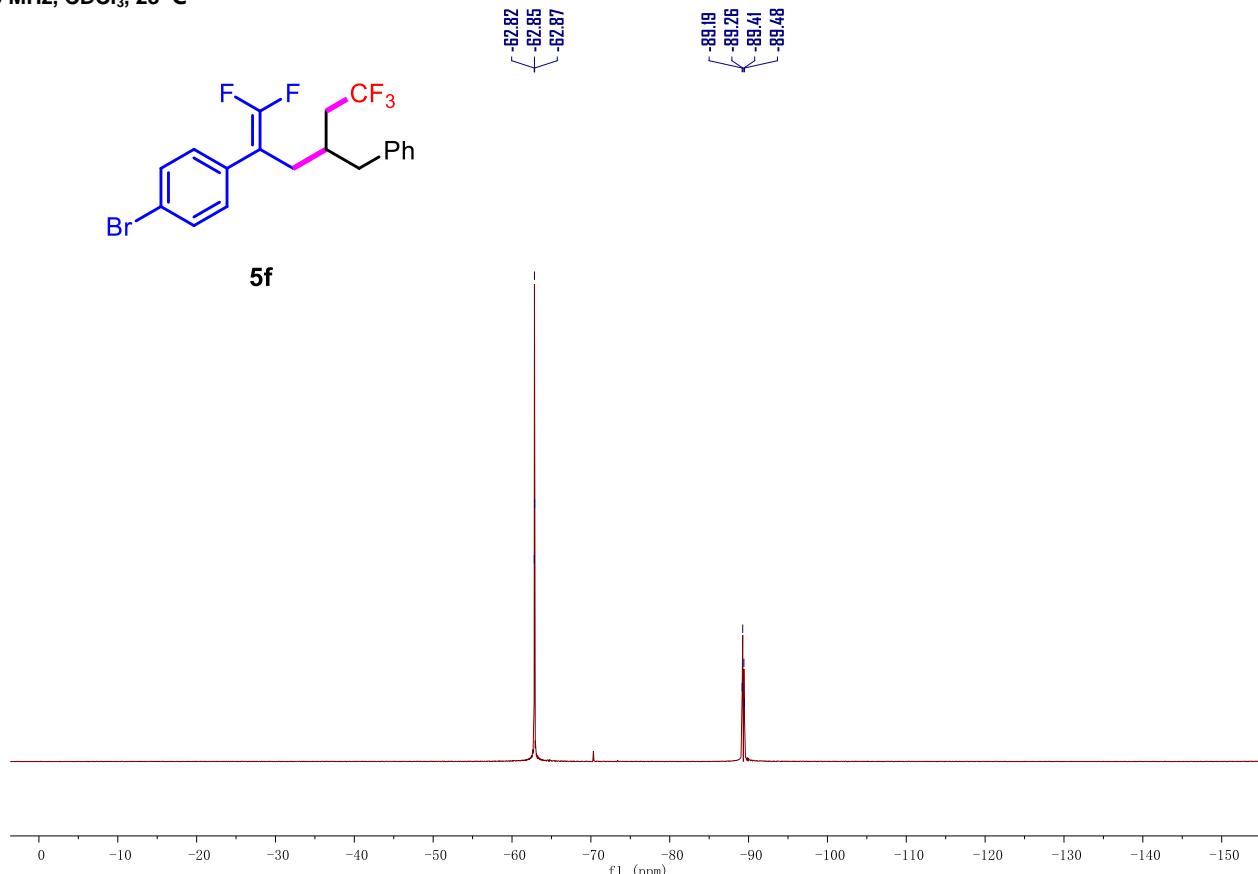
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-bromobenzene (5f)



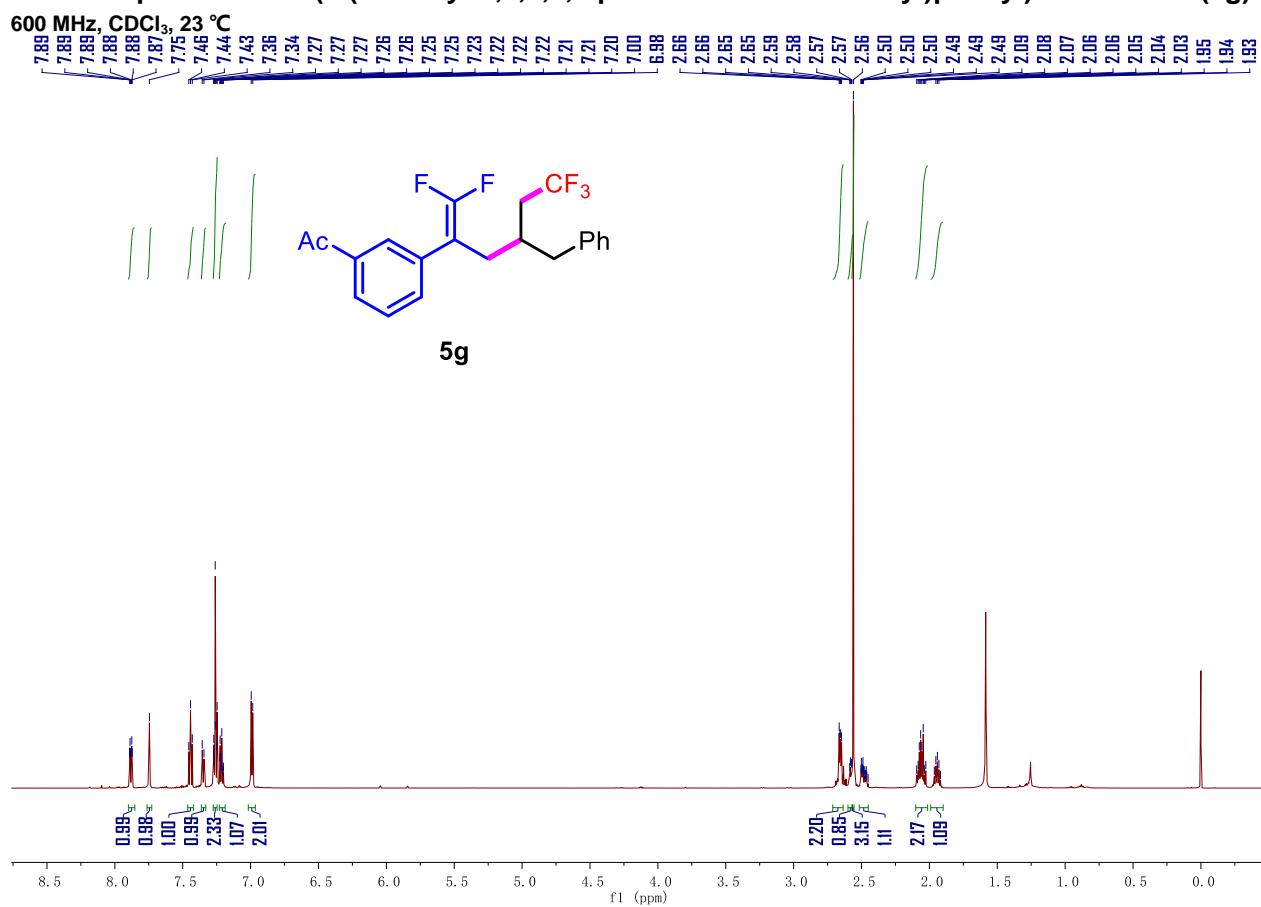
¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-bromobenzene (5f)



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4-bromobenzene (5f)
565 MHz, CDCl₃, 23 °C

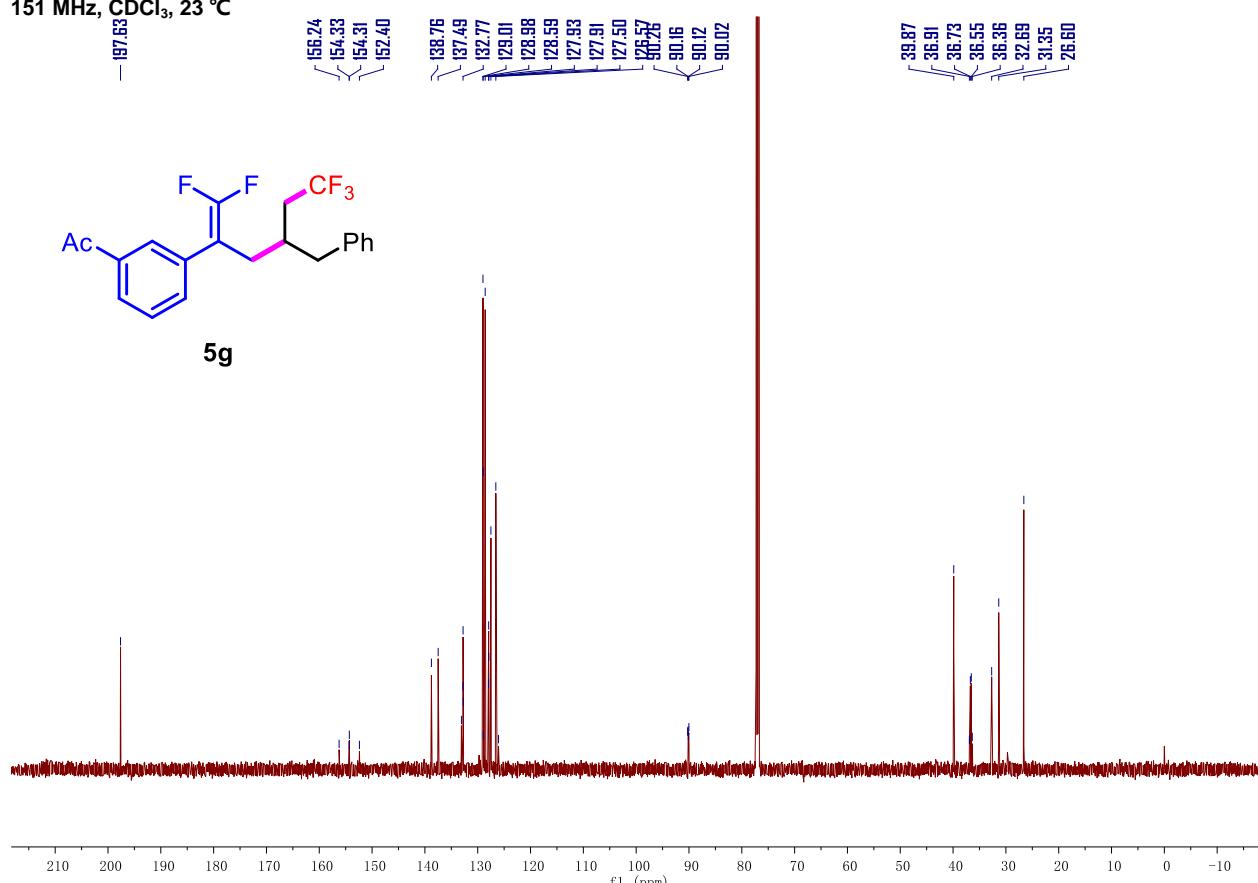


¹H NMR spectrum of 1-(3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)ethan-1-one (5g)



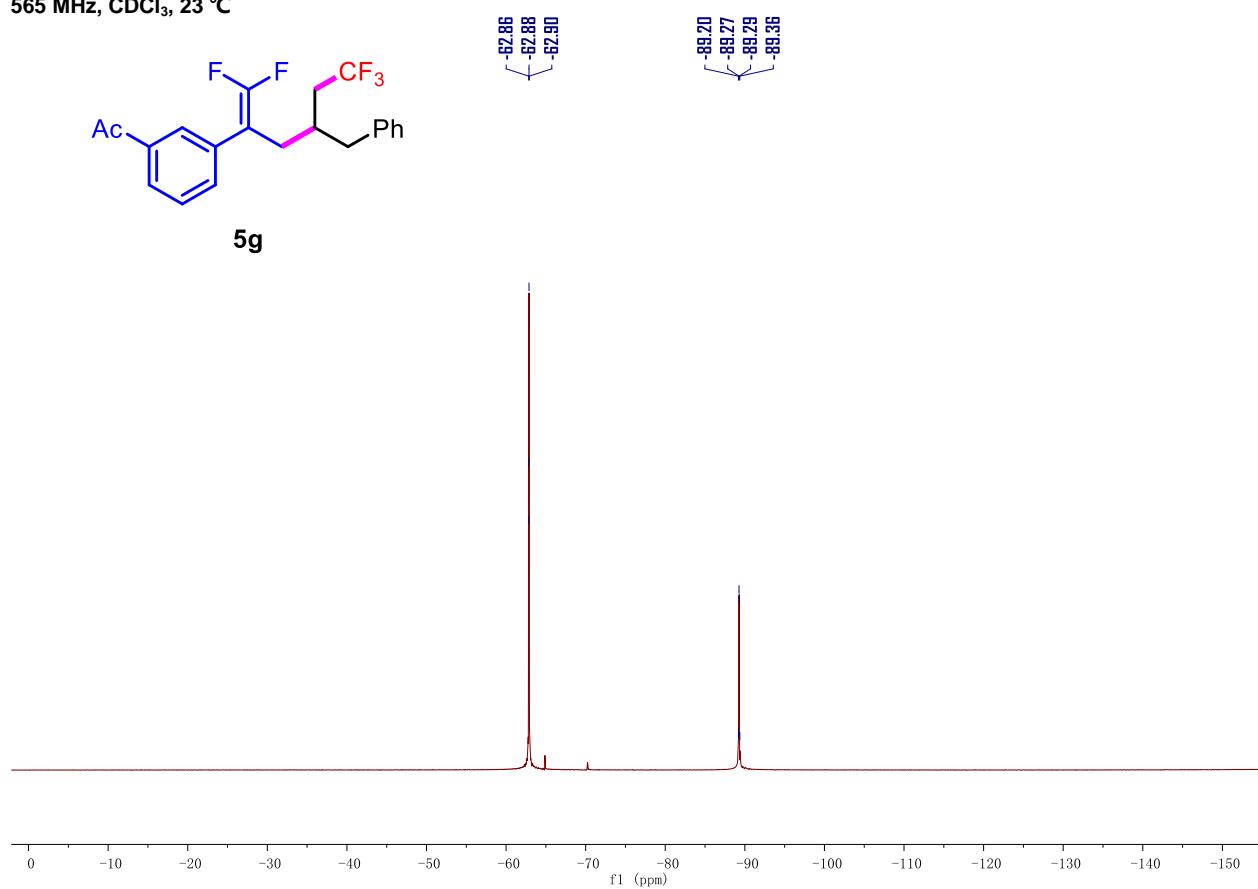
¹³C NMR spectrum of 1-(3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)ethan-1-one (5g)

151 MHz, CDCl₃, 23 °C



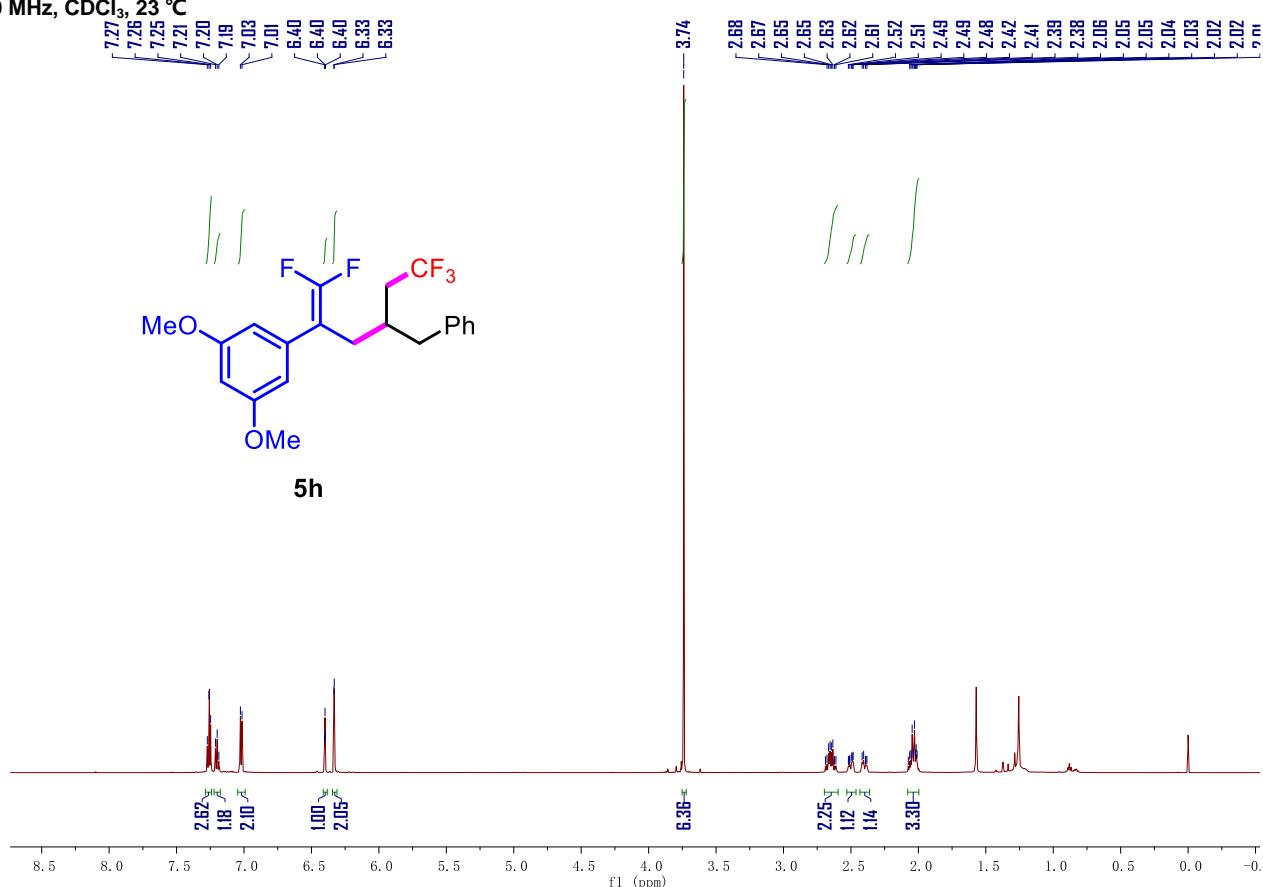
¹⁹F NMR spectrum of 1-(3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)ethan-1-one (5g)

565 MHz, CDCl₃, 23 °C



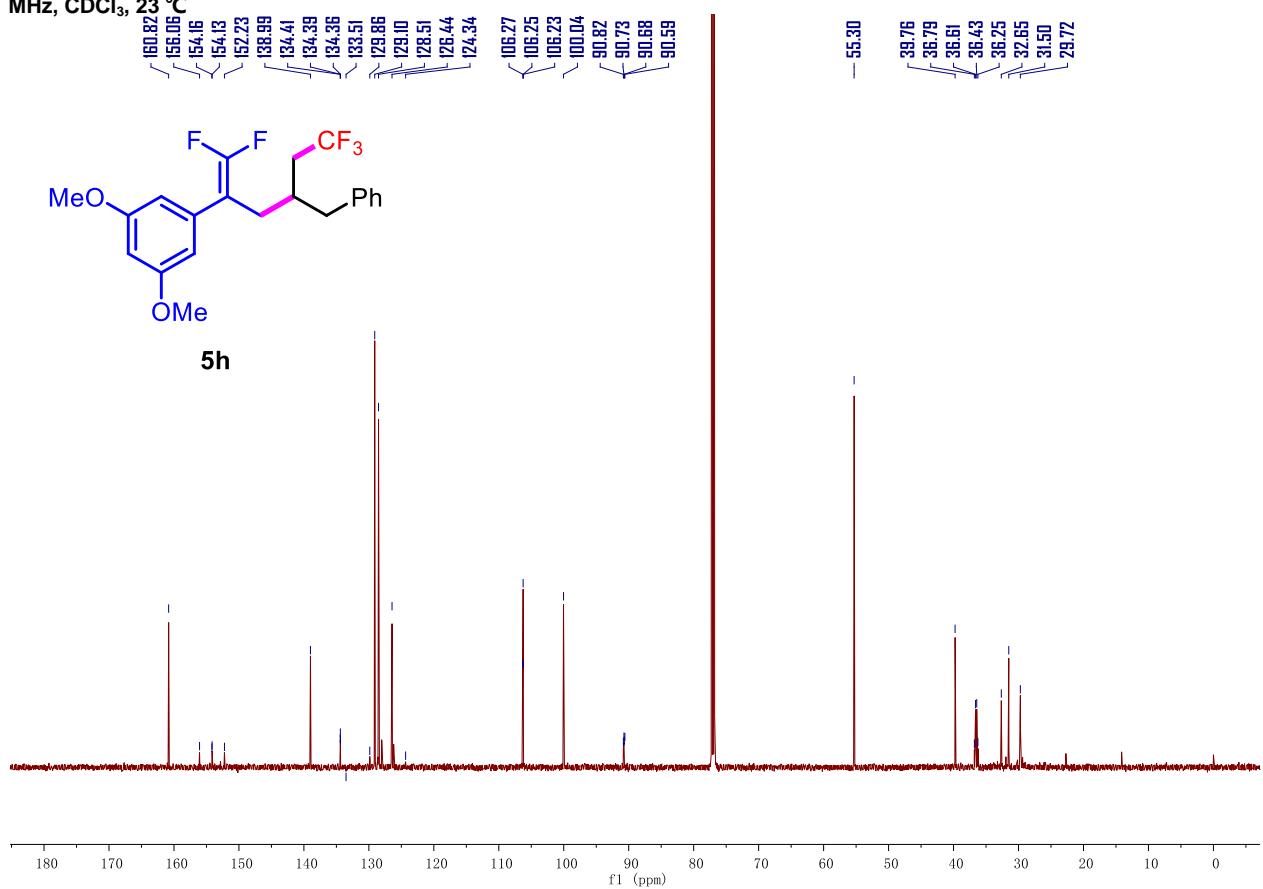
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-3,5-dimethoxybenzene (5h)

600 MHz, CDCl₃, 23 °C

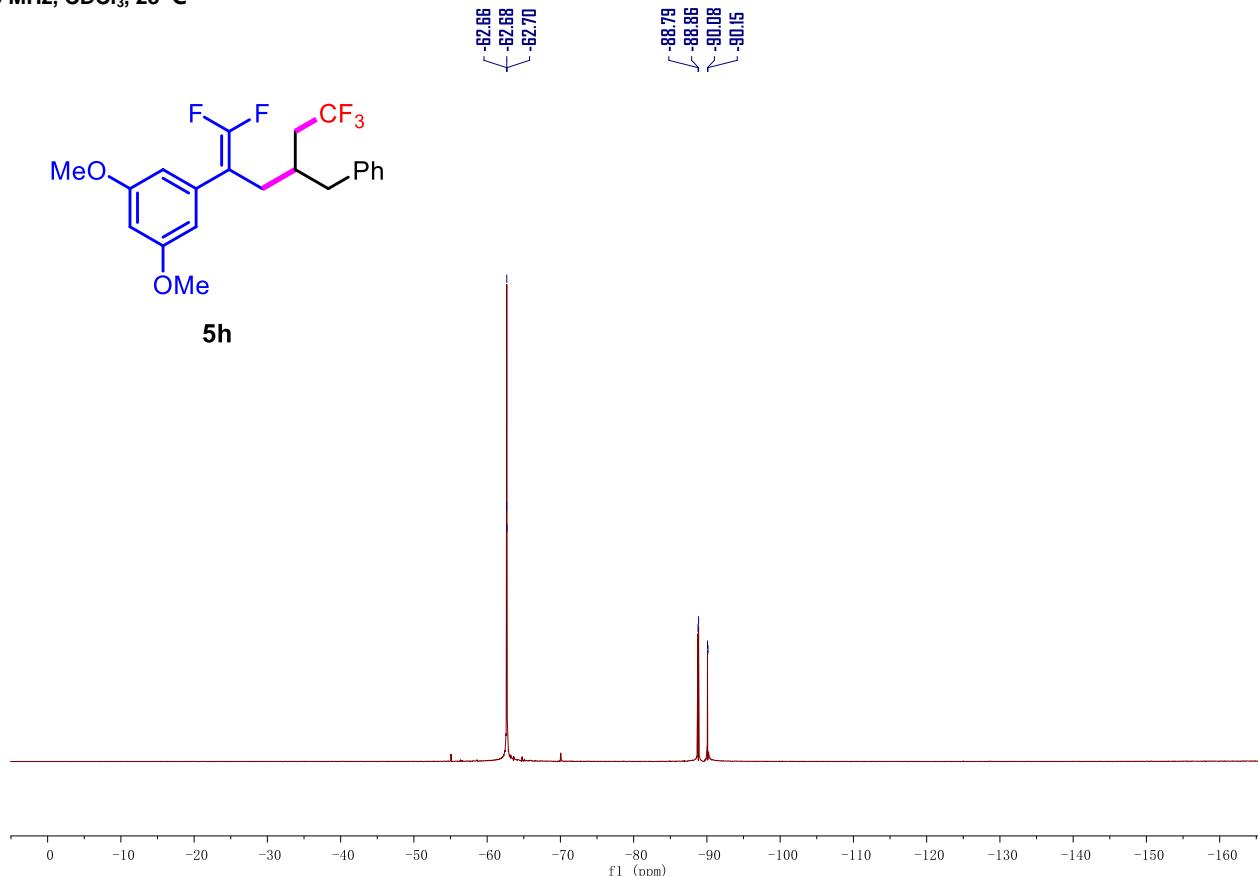


¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-3,5-dimethoxybenzene (5h)

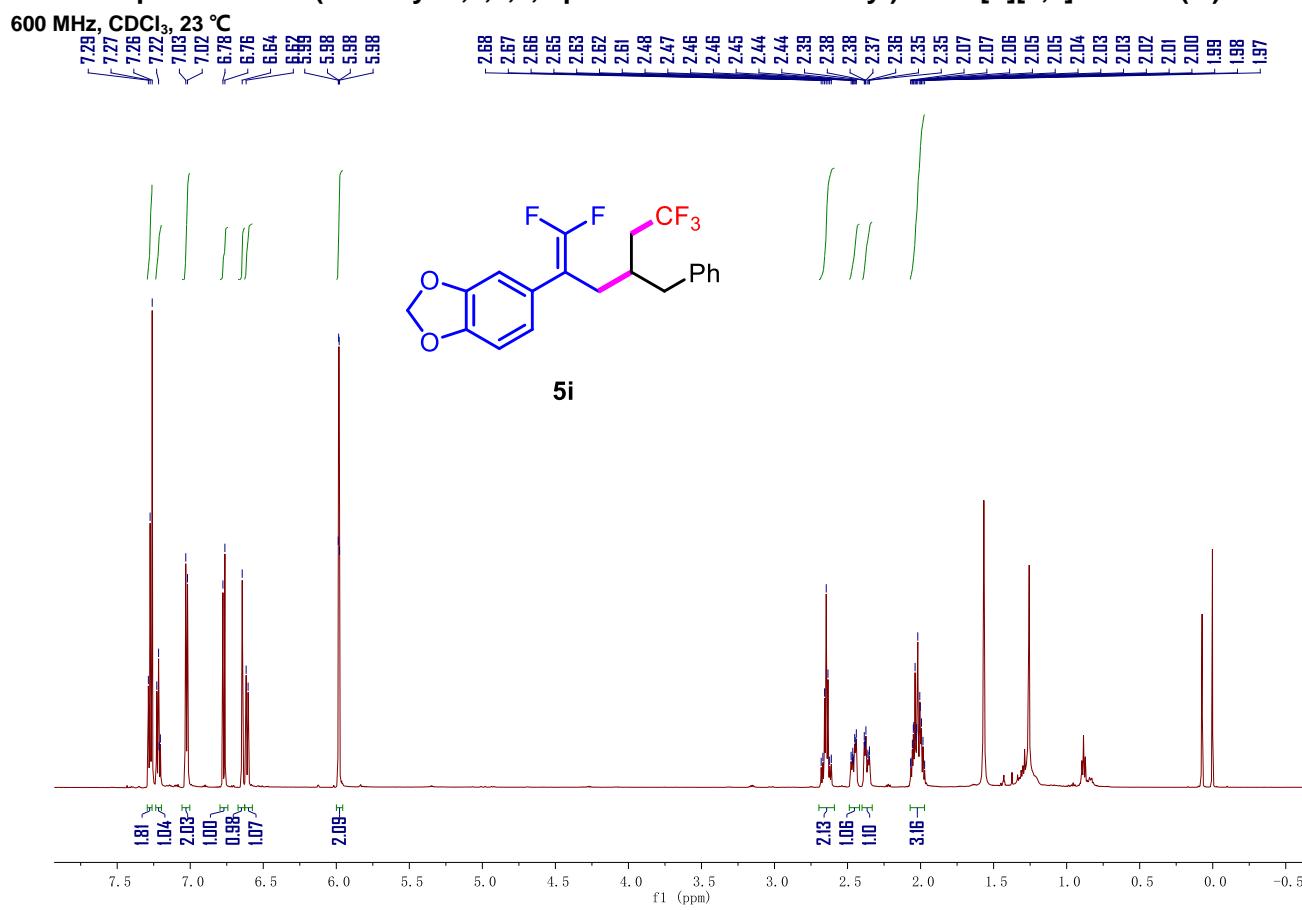
151 MHz, CDCl₃, 23 °C



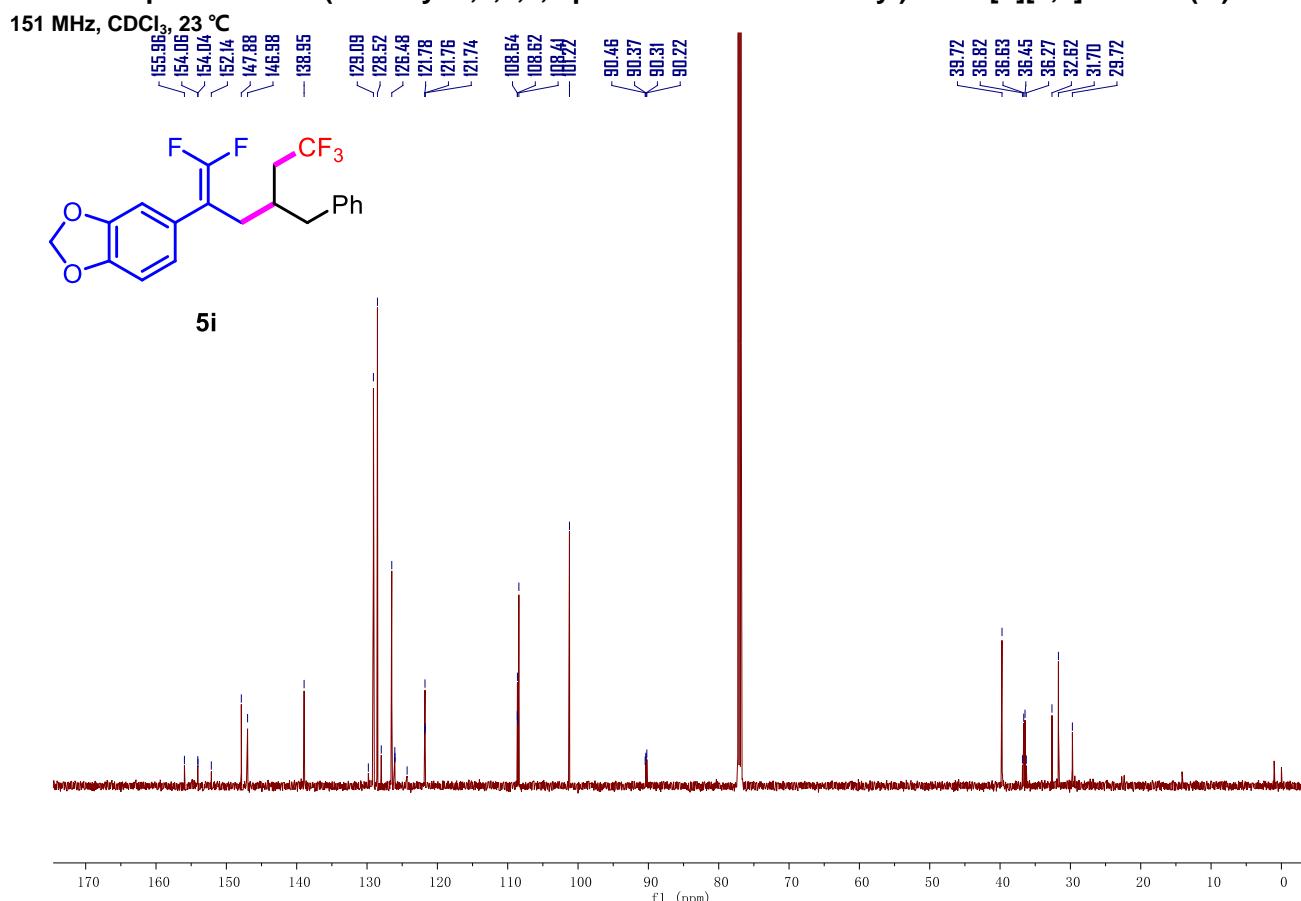
¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-3,5-dimethoxybenzene (5h)
565 MHz, CDCl₃, 23 °C



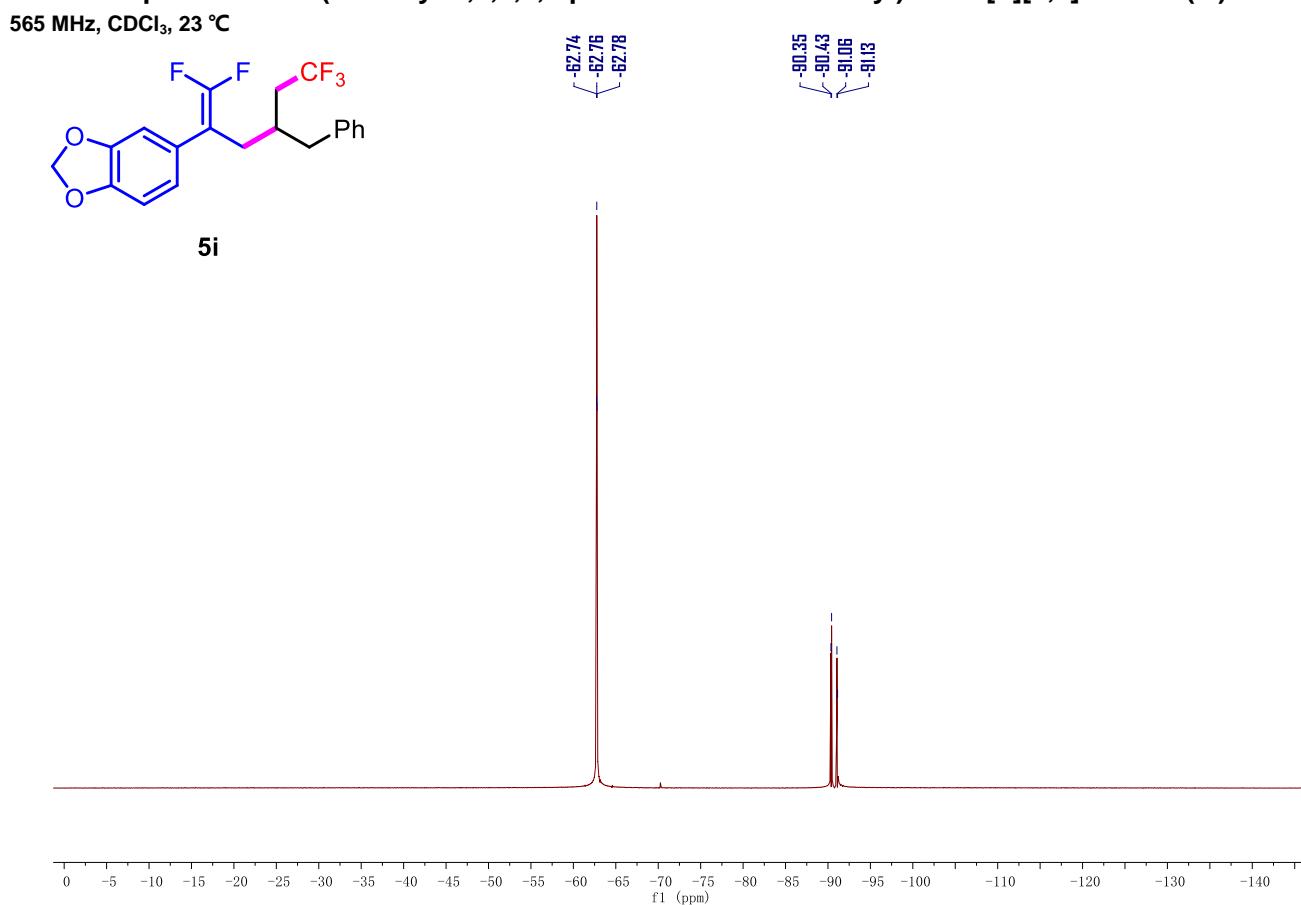
¹H NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzo[d][1,3]dioxole (5i)



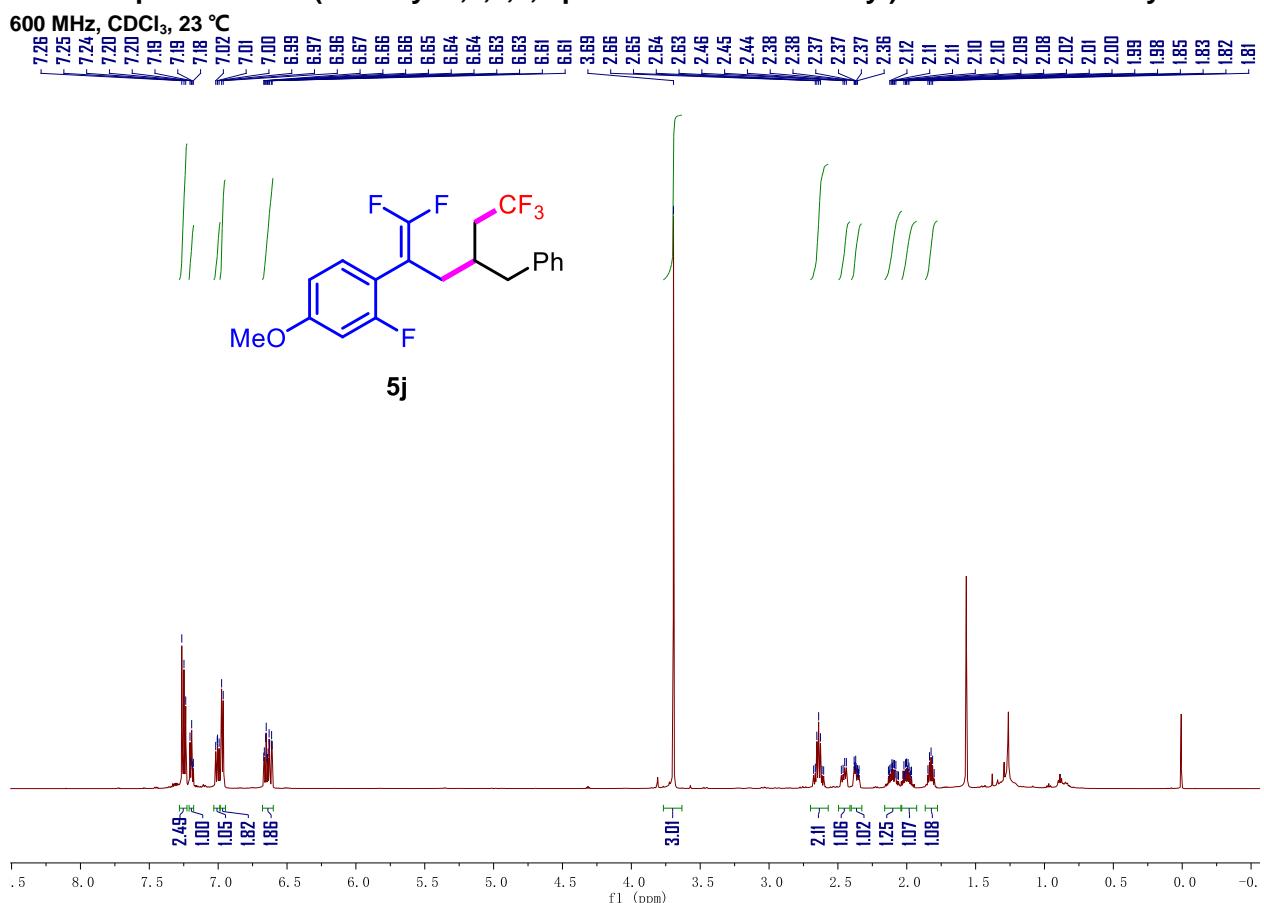
¹³C NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzo[d][1,3]dioxole (5i)



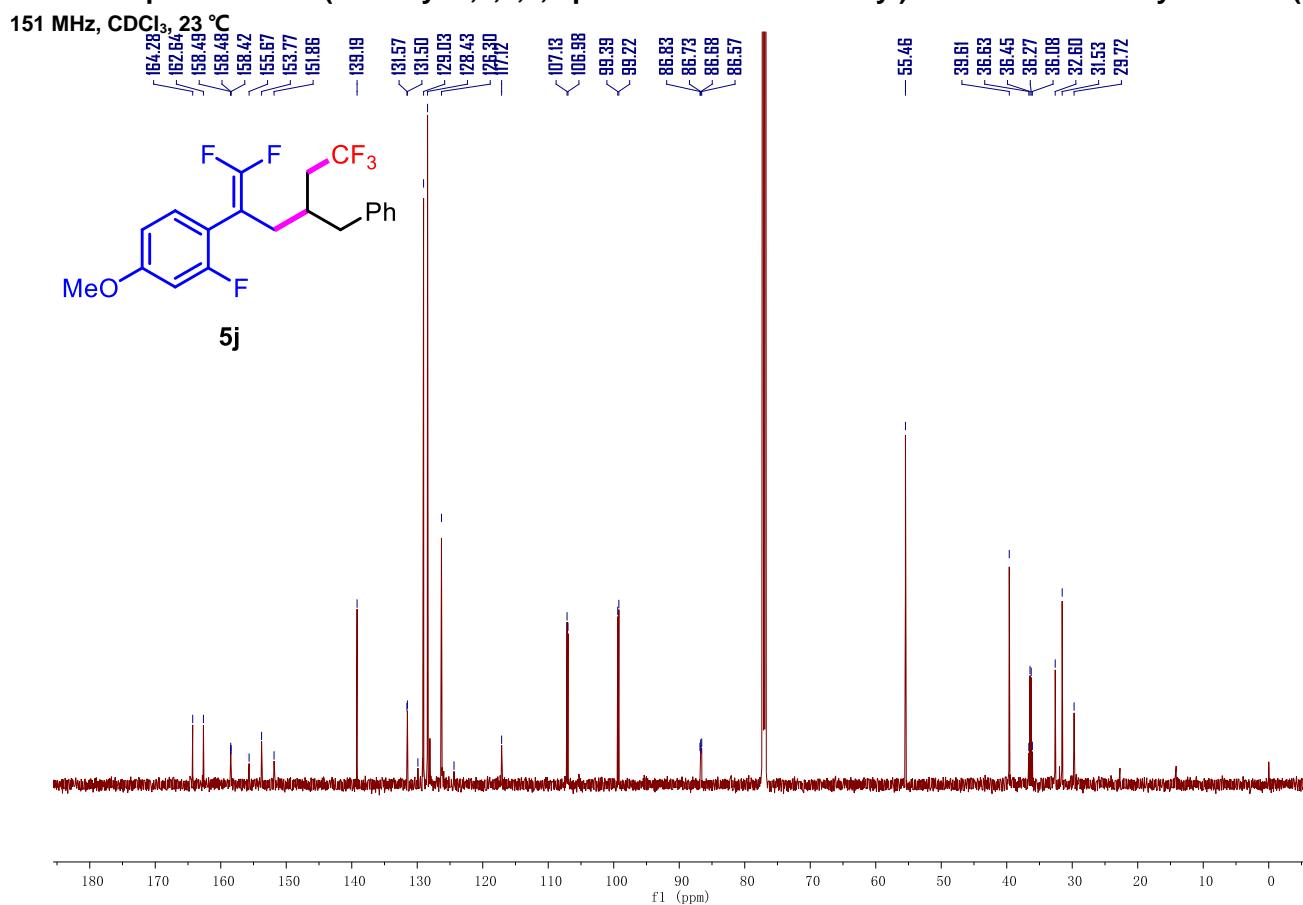
¹⁹F NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)benzo[d][1,3]dioxole (5i)



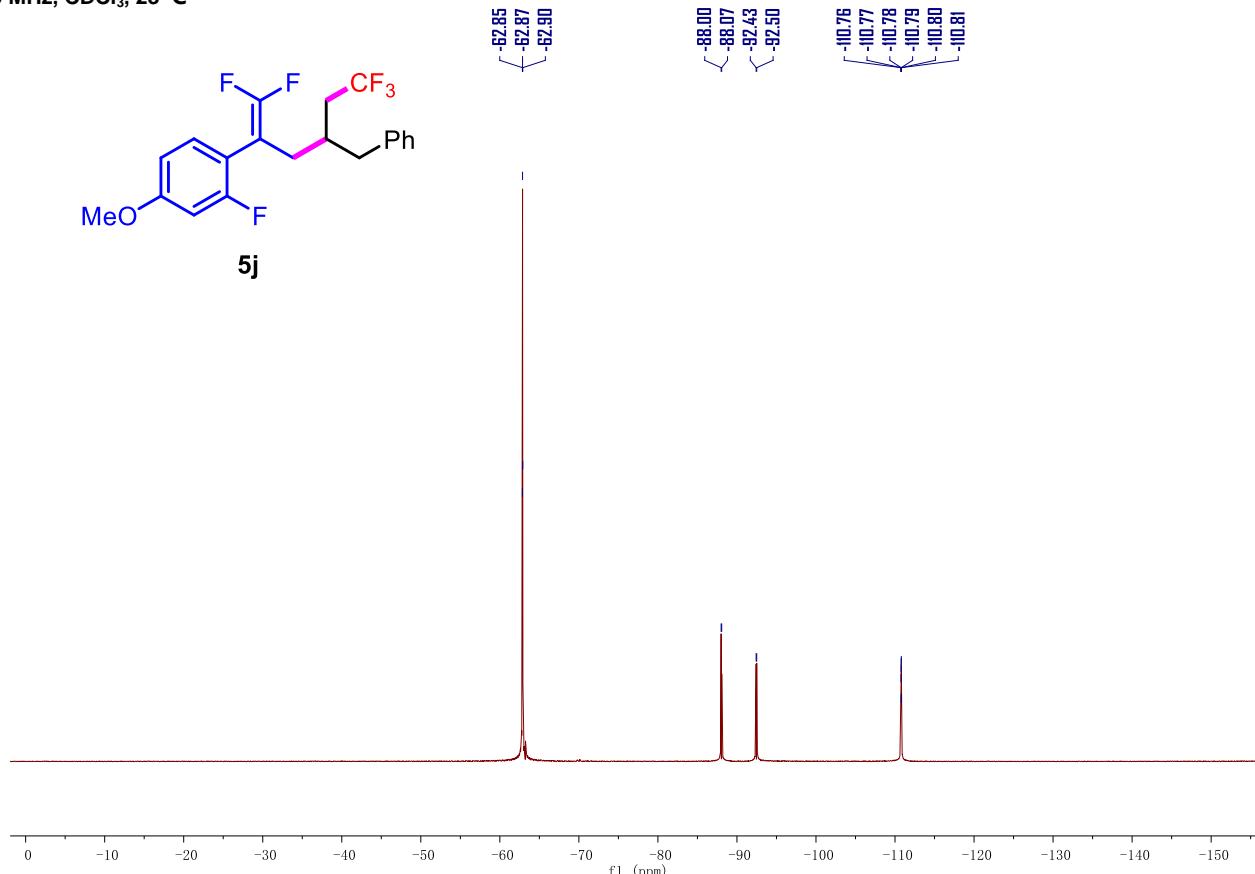
¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-fluoro-4-methoxybenzene (5j)



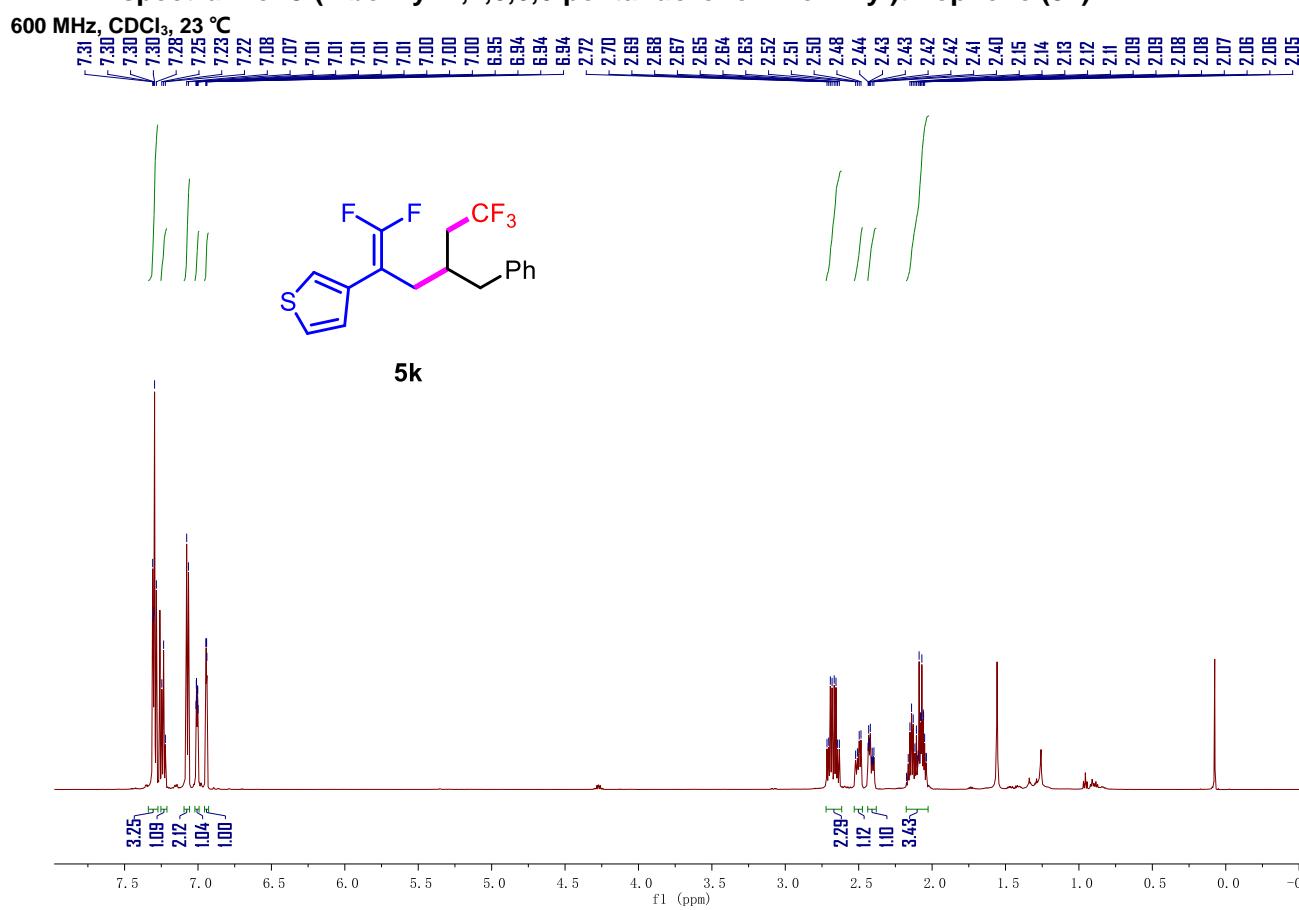
¹³C NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-fluoro-4-methoxybenzene (5j)



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-fluoro-4-methoxybenzene (5j)
565 MHz, CDCl₃, 23 °C

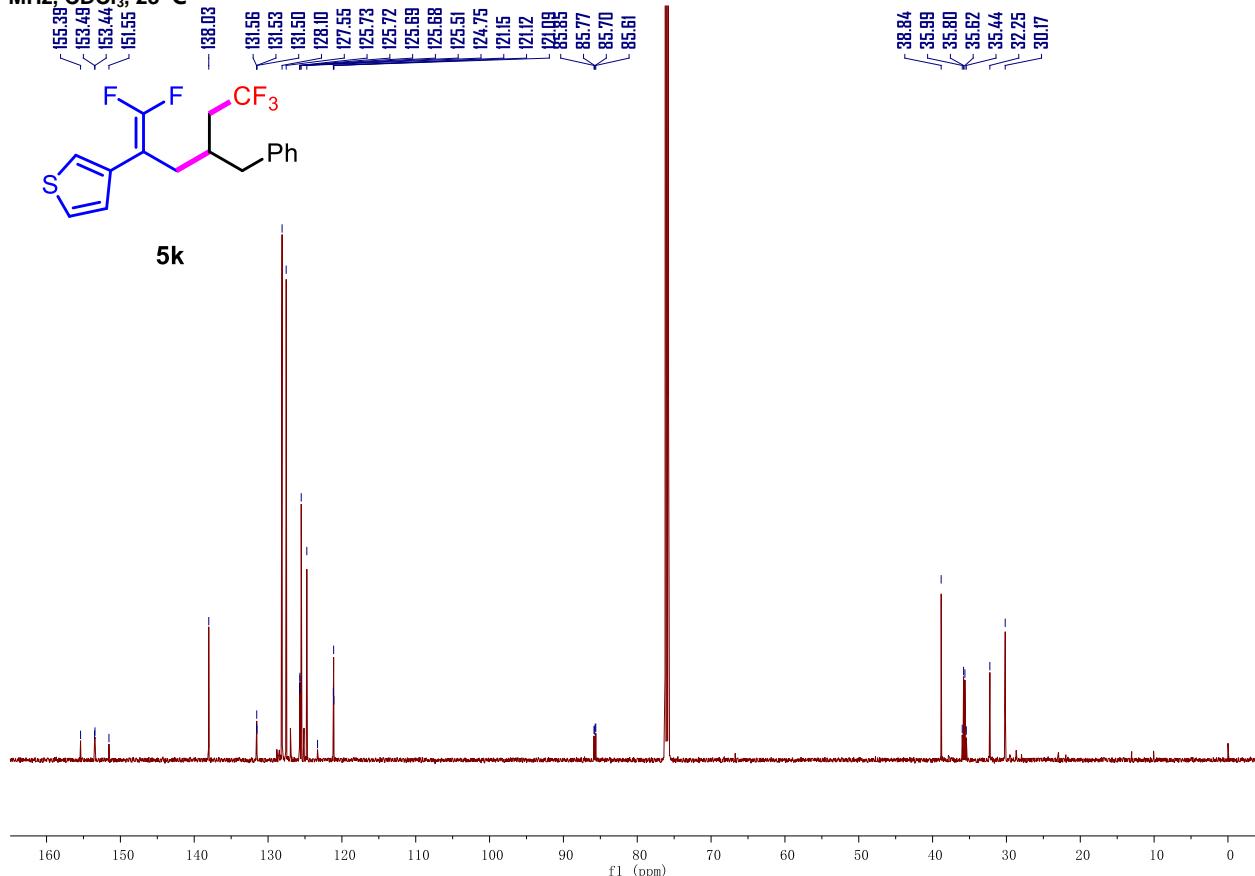


¹H NMR spectrum of 3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)thiophene (5k)



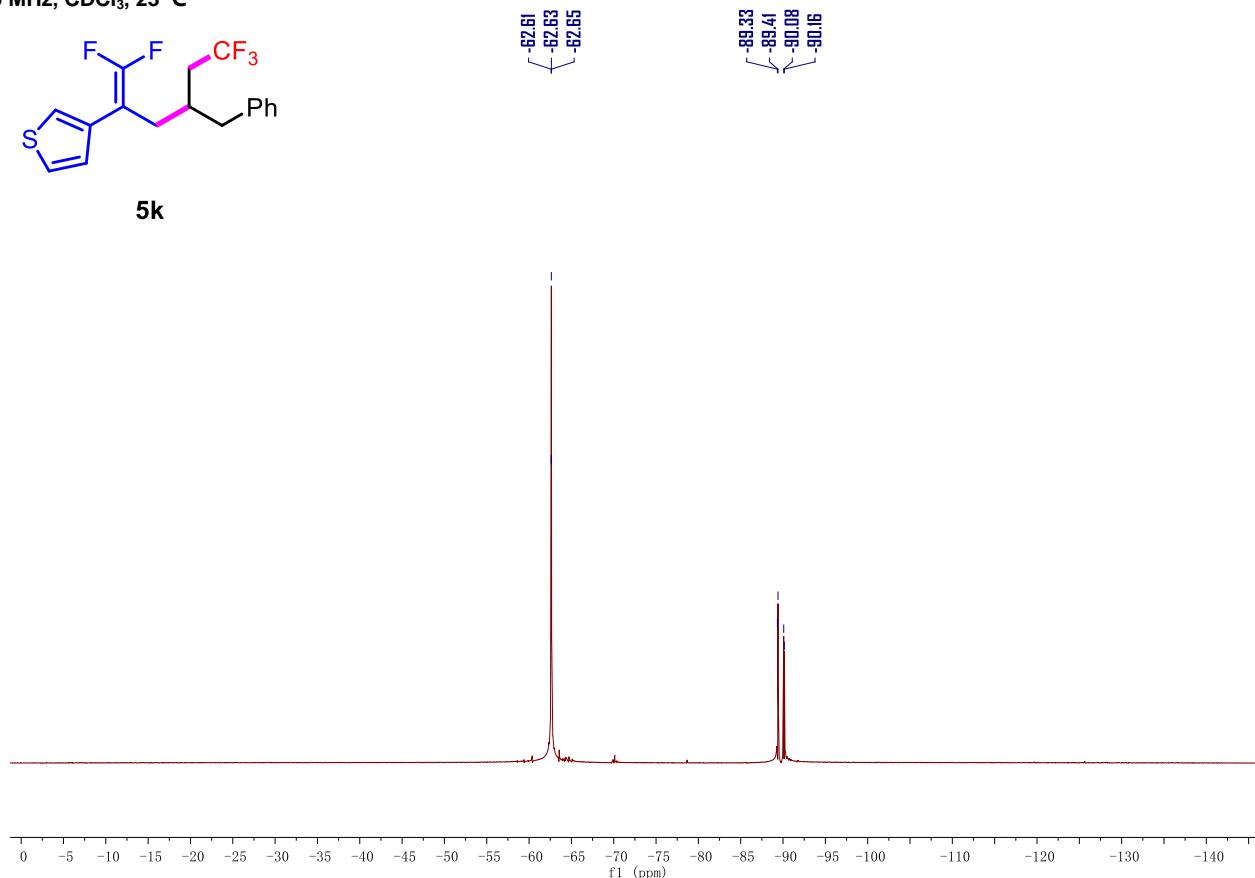
¹³C NMR spectrum of 3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)thiophene (5k)

151 MHz, CDCl₃, 23 °C



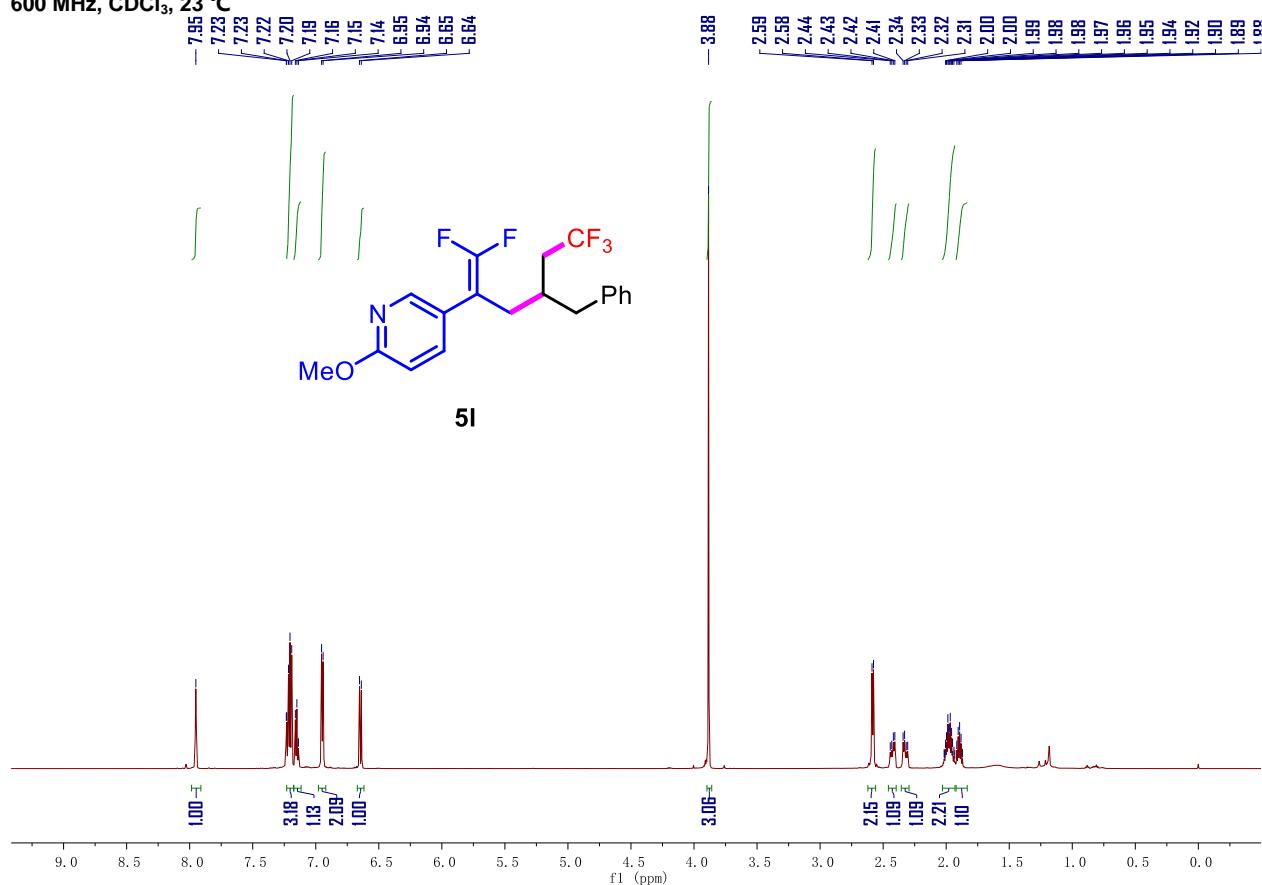
¹⁹F NMR spectrum of 3-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)thiophene (5k)

565 MHz, CDCl₃, 23 °C



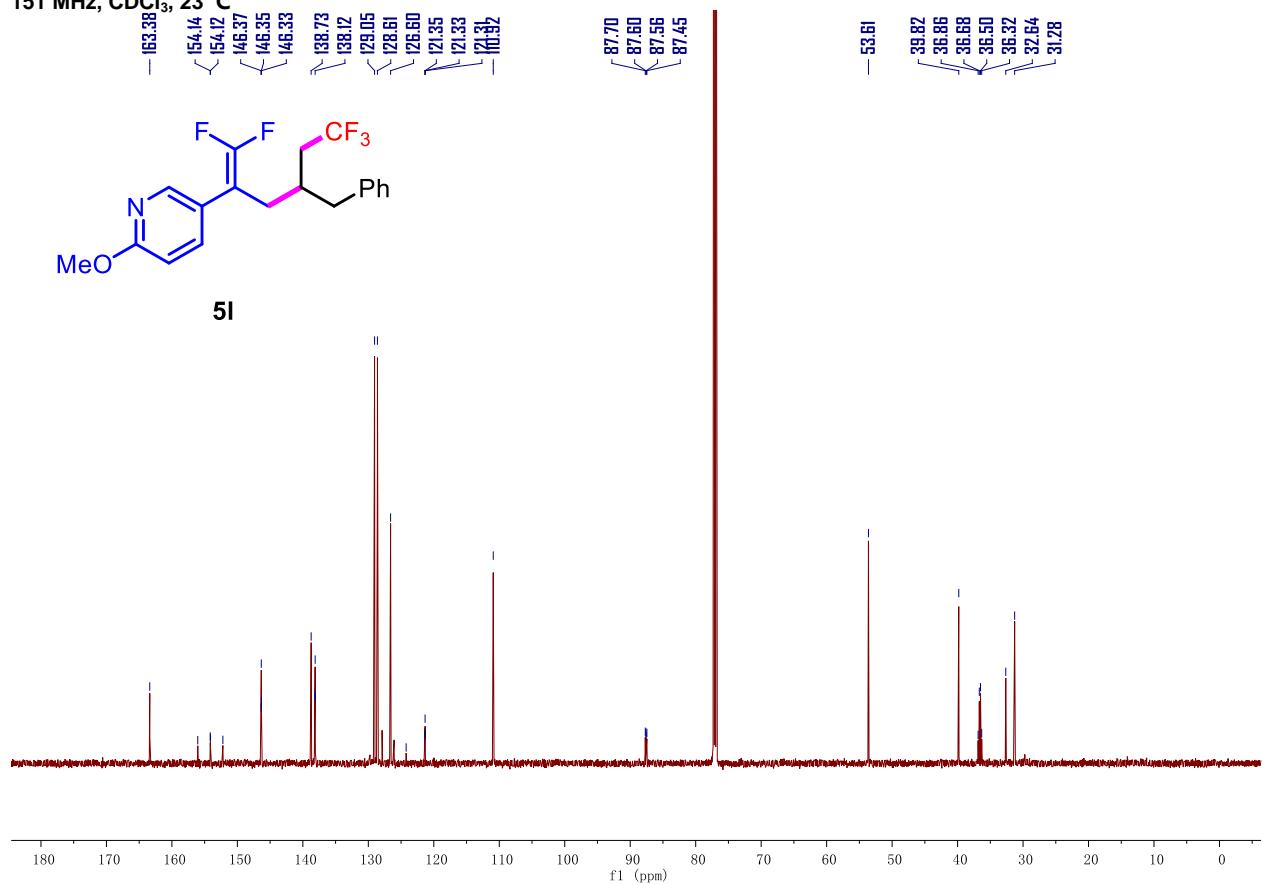
¹H NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-methoxypyridine (5l)

600 MHz, CDCl₃, 23 °C

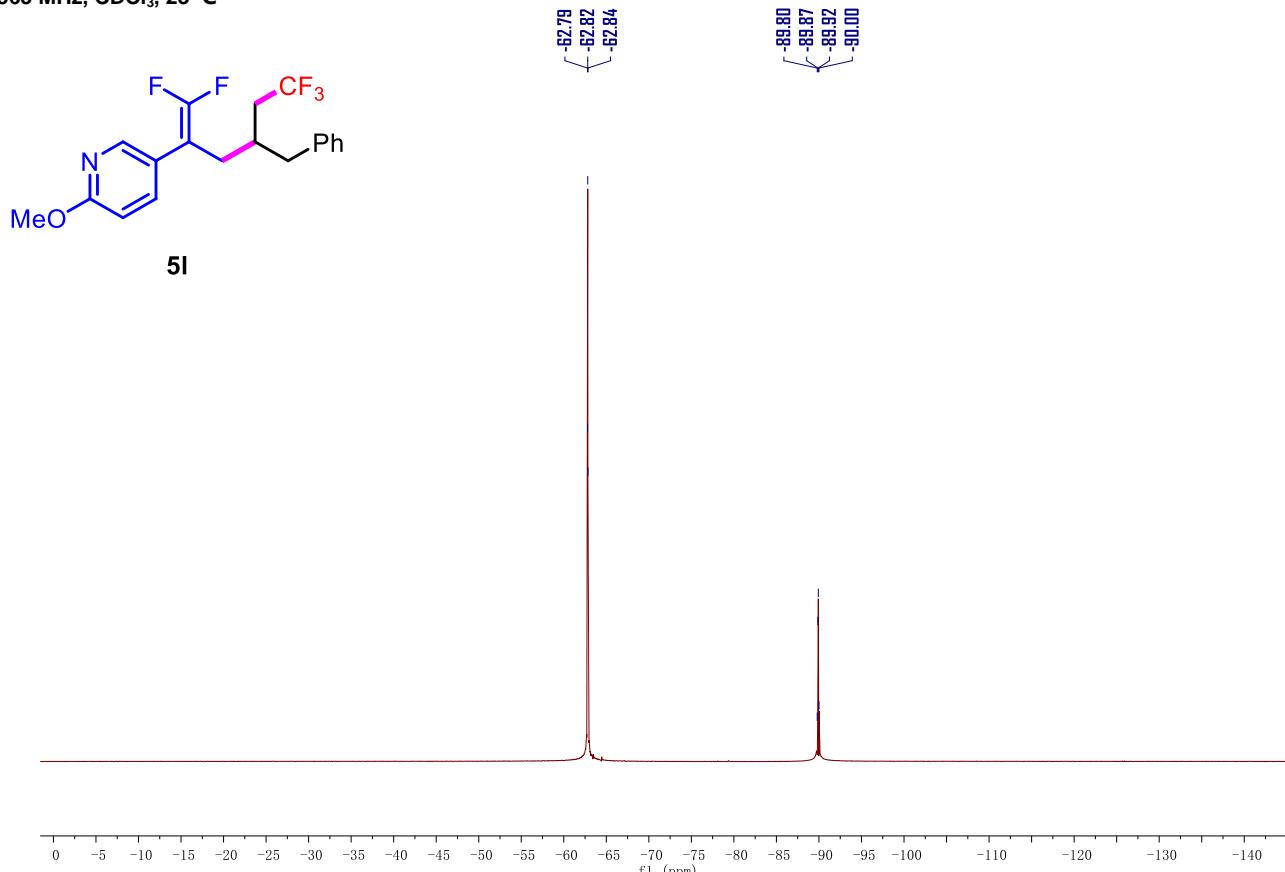


¹³C NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-methoxypyridine (5l)

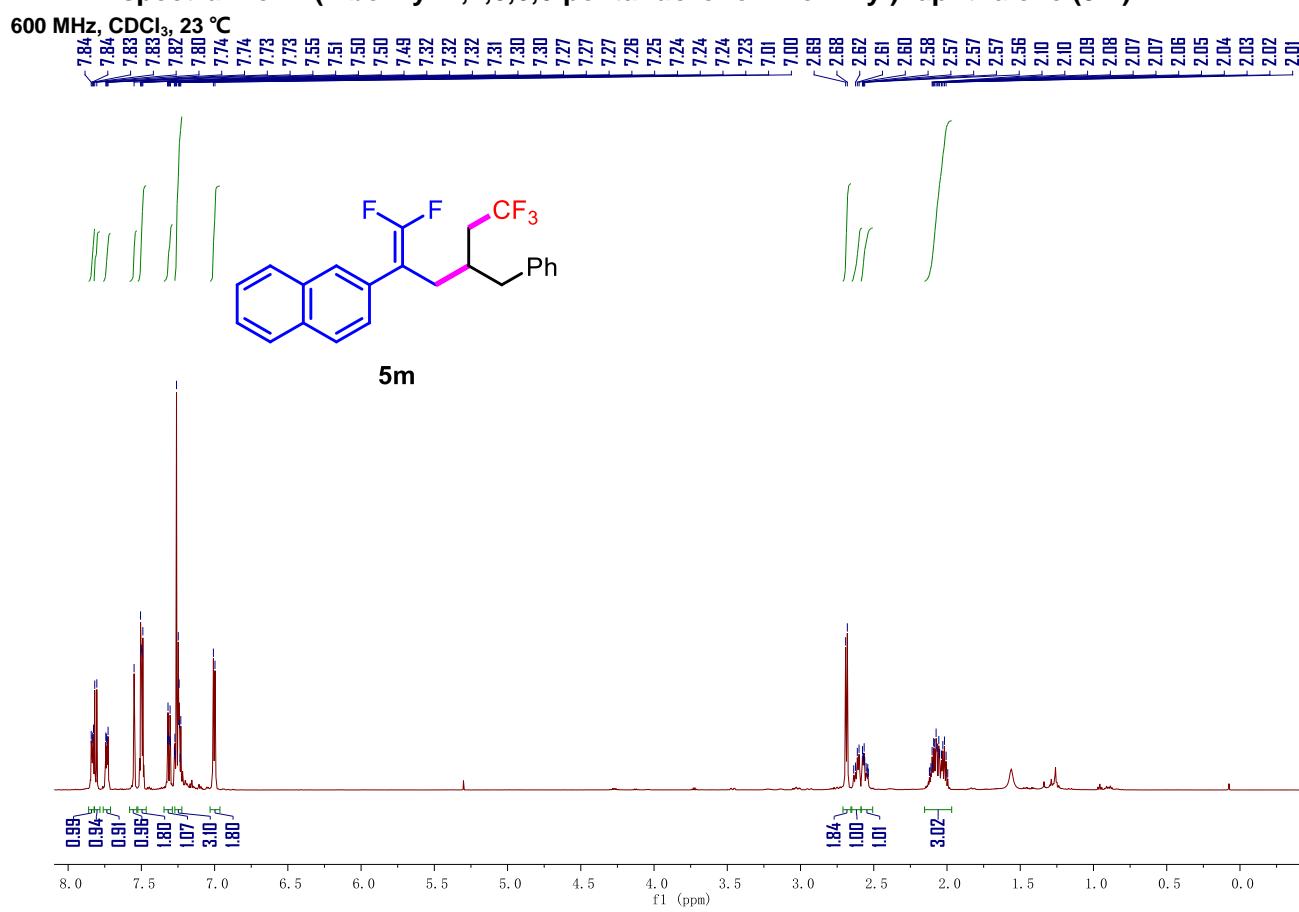
151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 5-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-2-methoxypyridine (5l)
 565 MHz, CDCl₃, 23 °C

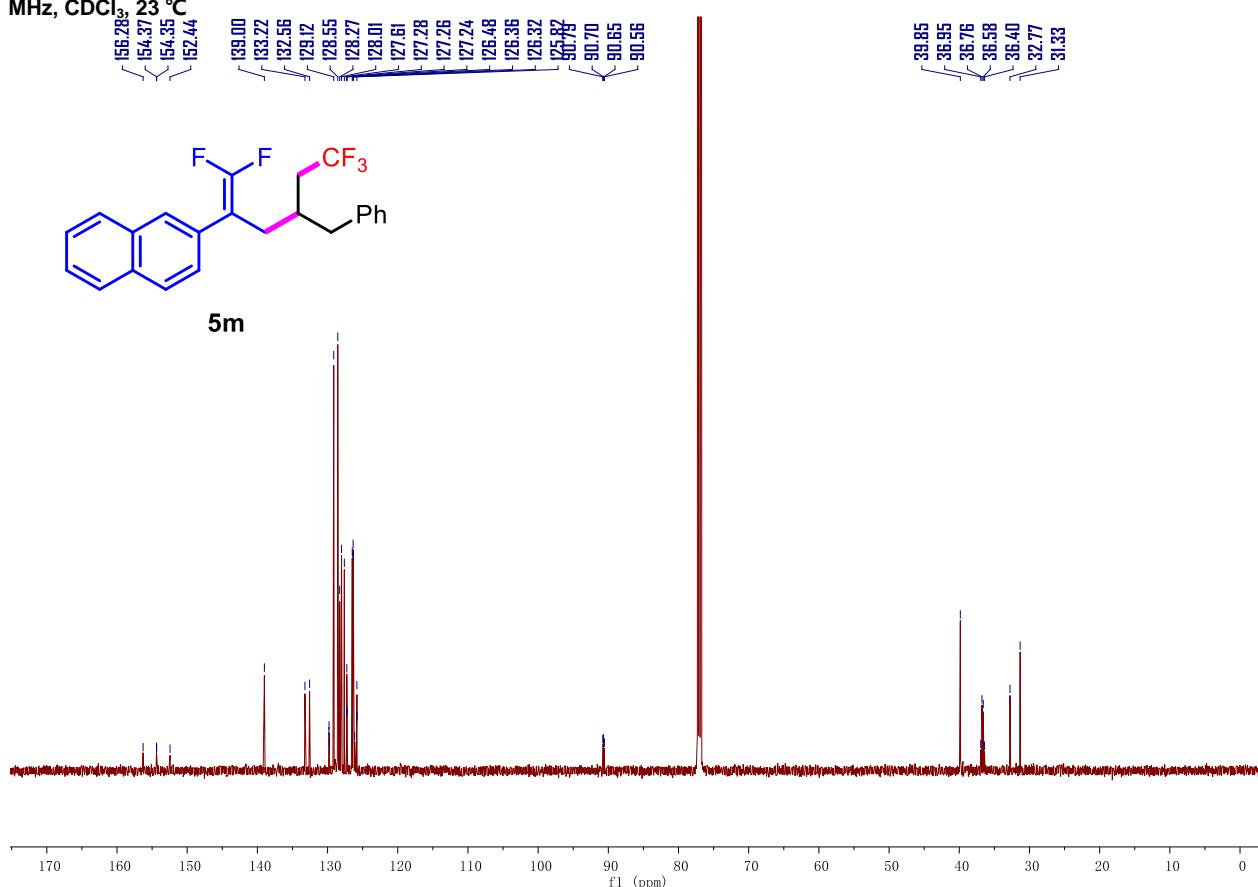


¹H NMR spectrum of 2-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)naphthalene (5m)



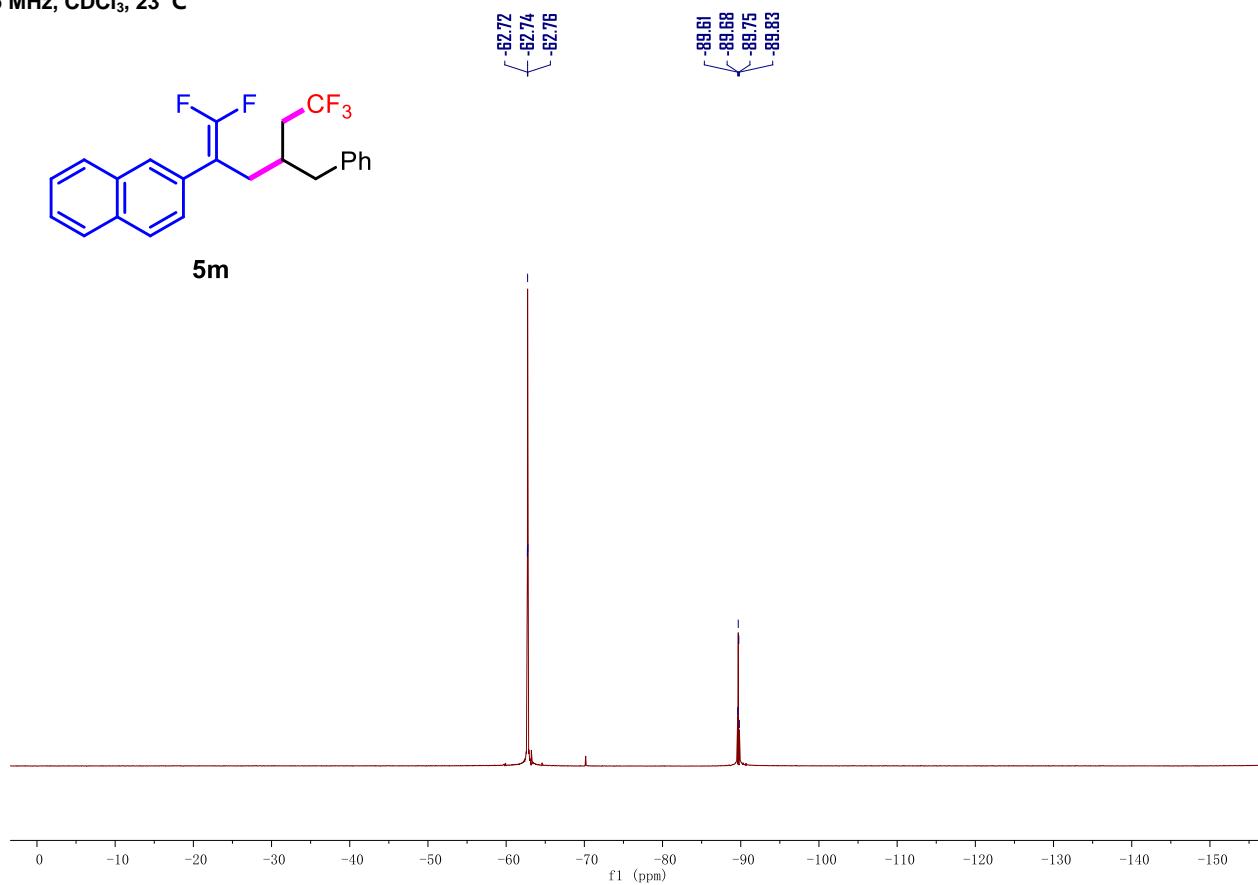
¹³C NMR spectrum of 2-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)naphthalene (5m)

151 MHz, CDCl₃, 23 °C



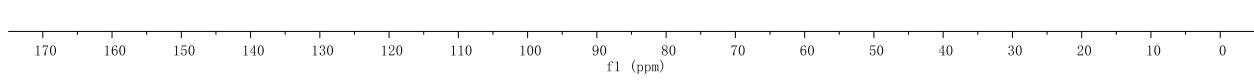
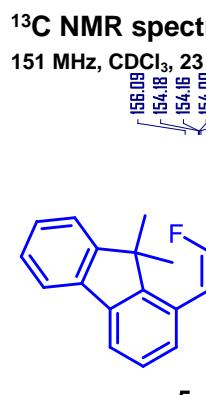
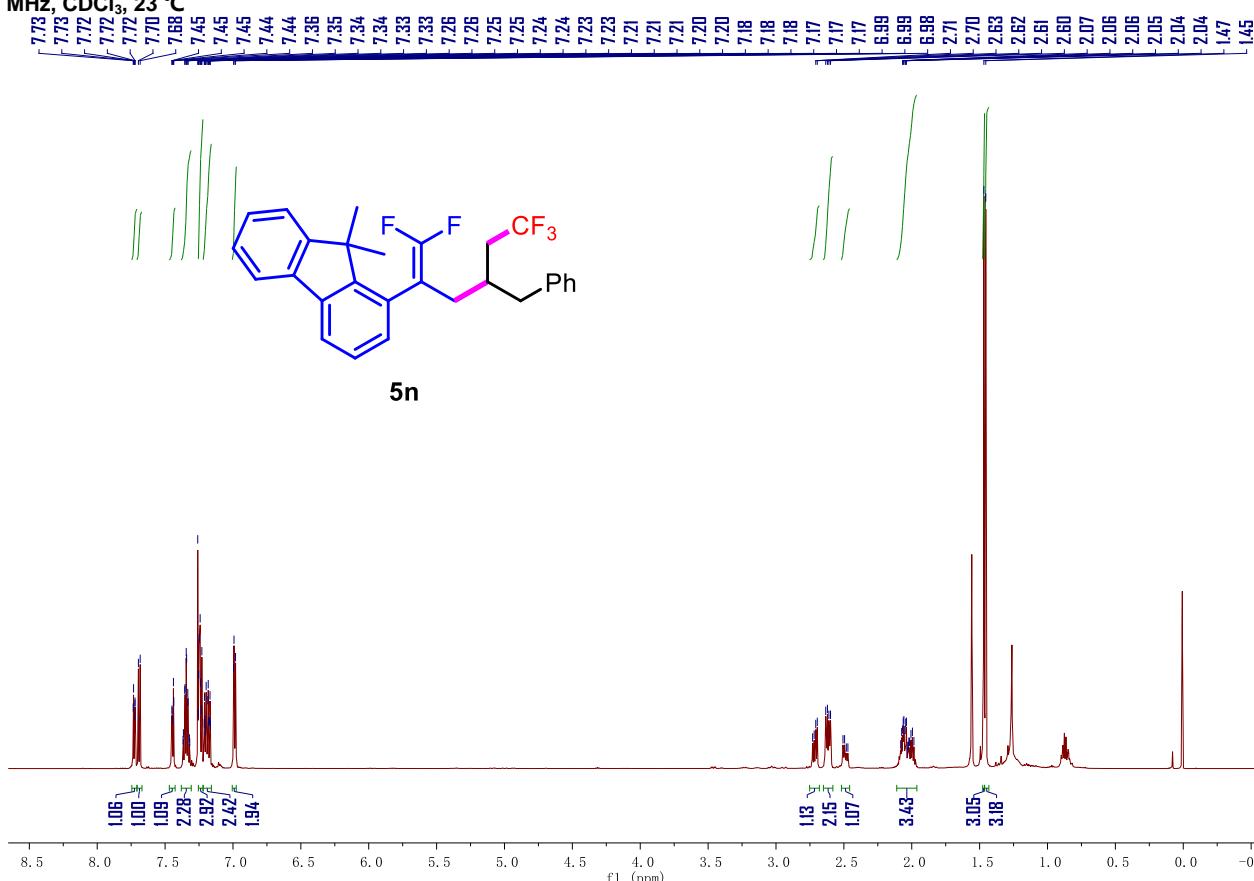
¹⁹F NMR spectrum of 2-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)naphthalene (5m)

565 MHz, CDCl₃, 23 °C

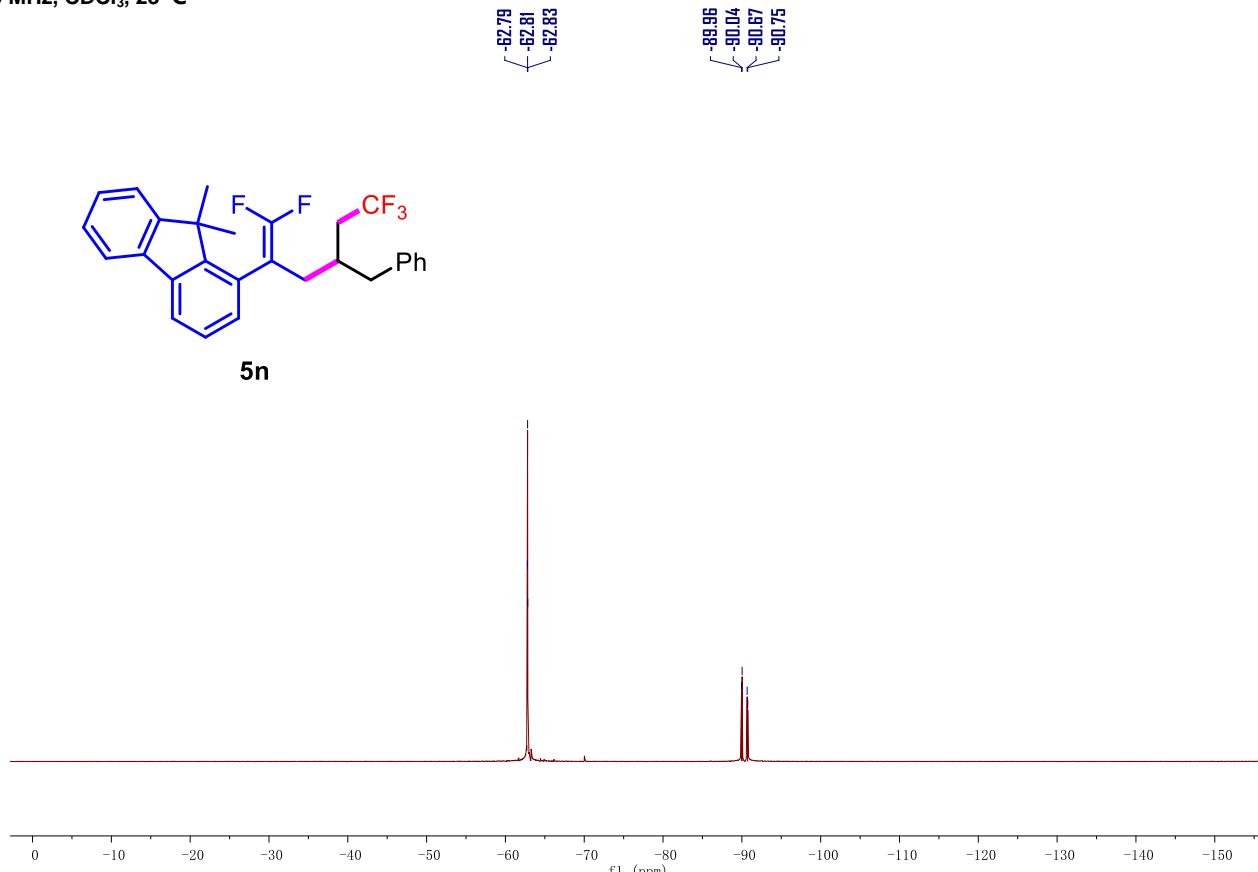


¹H NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-9,9-dimethyl-9H-fluorene (5n)

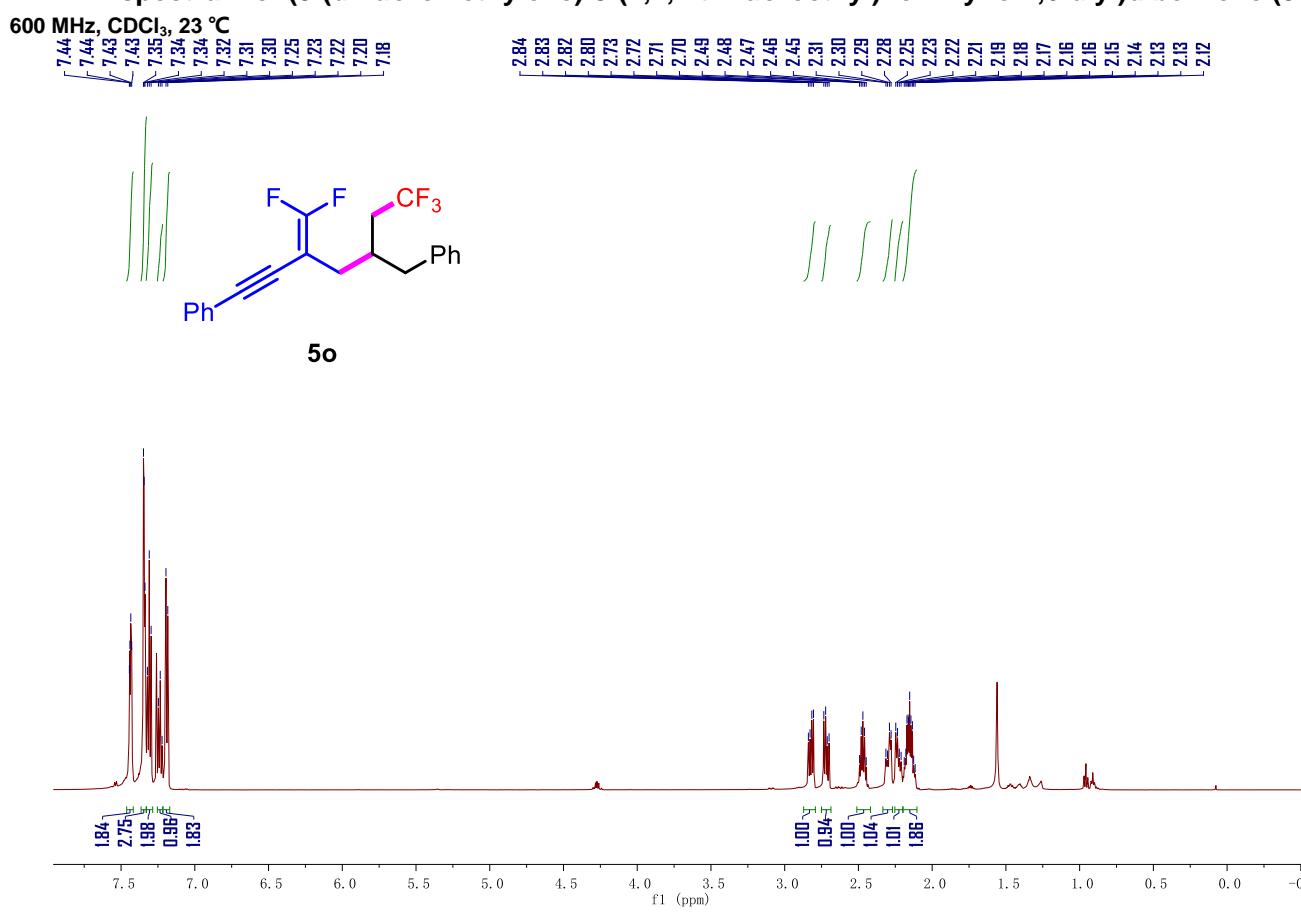
600 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of 1-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-9,9-dimethyl-9H-fluorene (5n)
565 MHz, CDCl₃, 23 °C

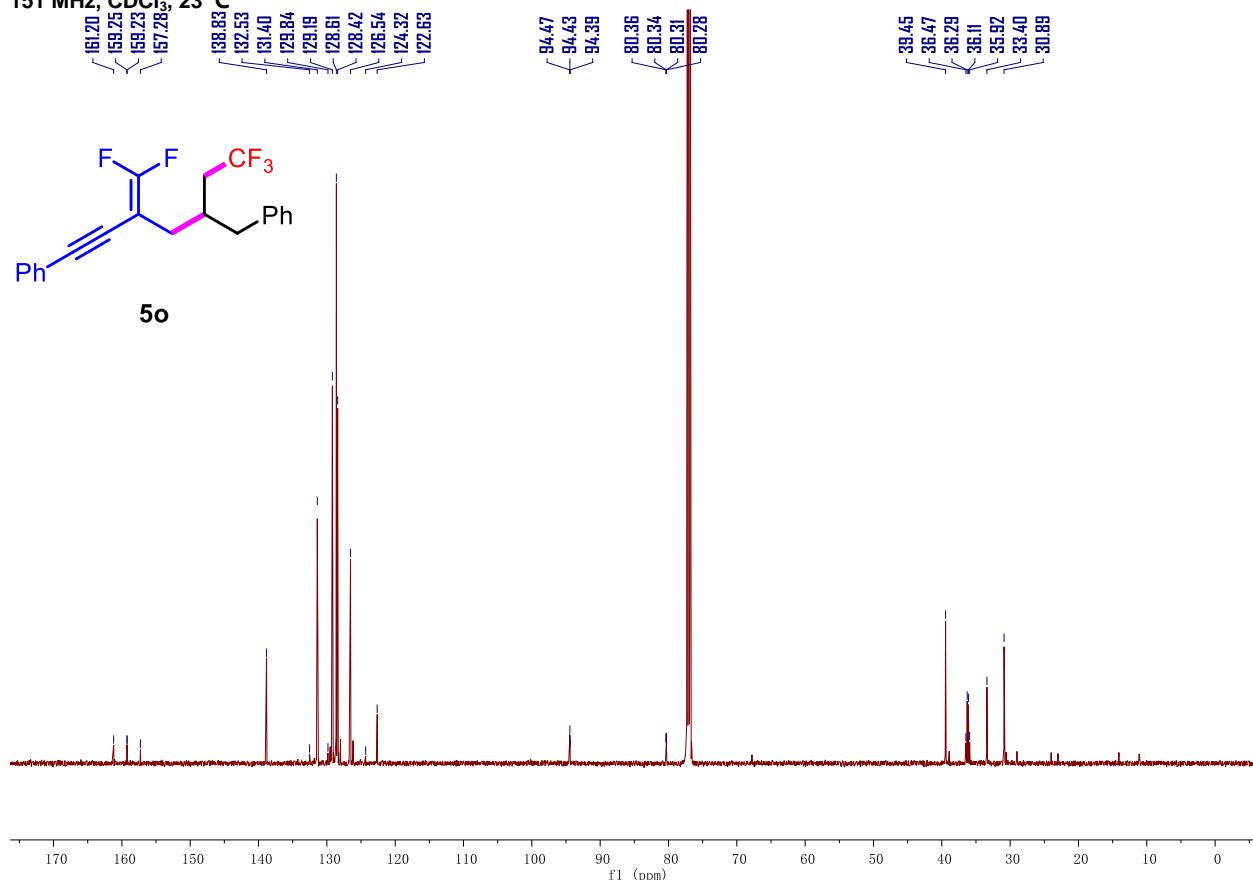


¹H NMR spectrum of (3-(difluoromethylene)-5-(2,2,2-trifluoroethyl)hex-1-yne-1,6-diyldibenzene (5o)



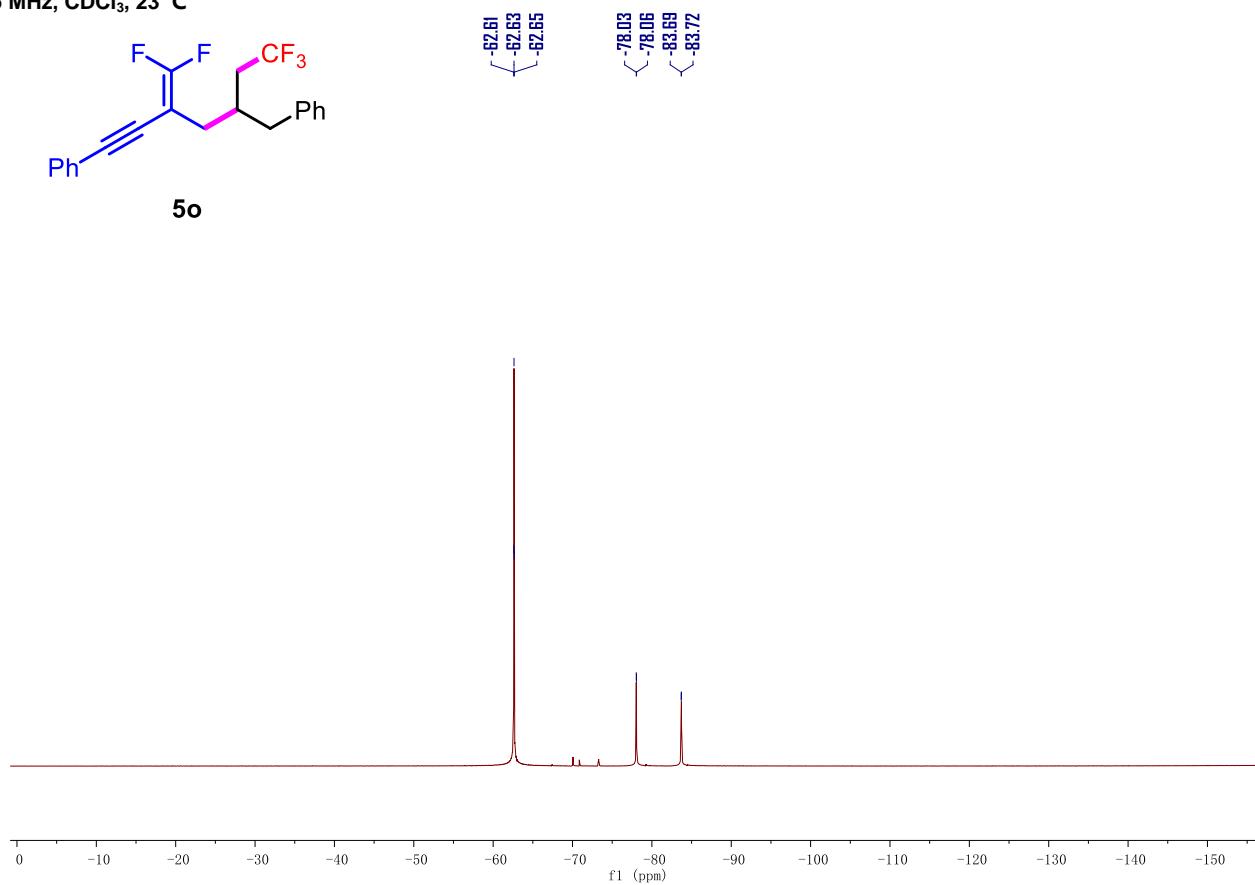
¹³C NMR spectrum of (3-(difluoromethylene)-5-(2,2,2-trifluoroethyl)hex-1-yne-1,6-diyldibenzene (5o)

151 MHz, CDCl₃, 23 °C

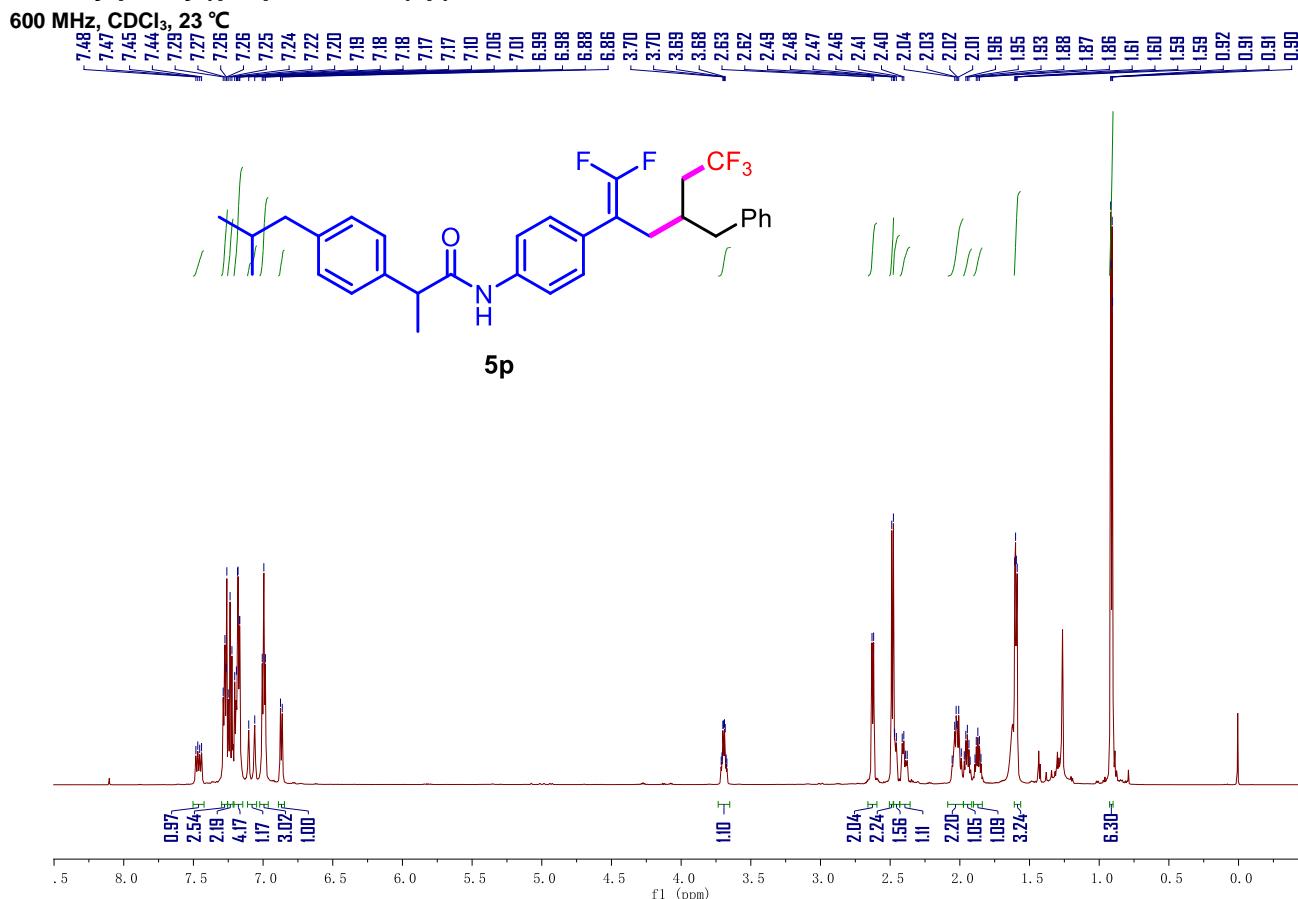


¹⁹F NMR spectrum of (3-(difluoromethylene)-5-(2,2,2-trifluoroethyl)hex-1-yne-1,6-diyldibenzene (5o)

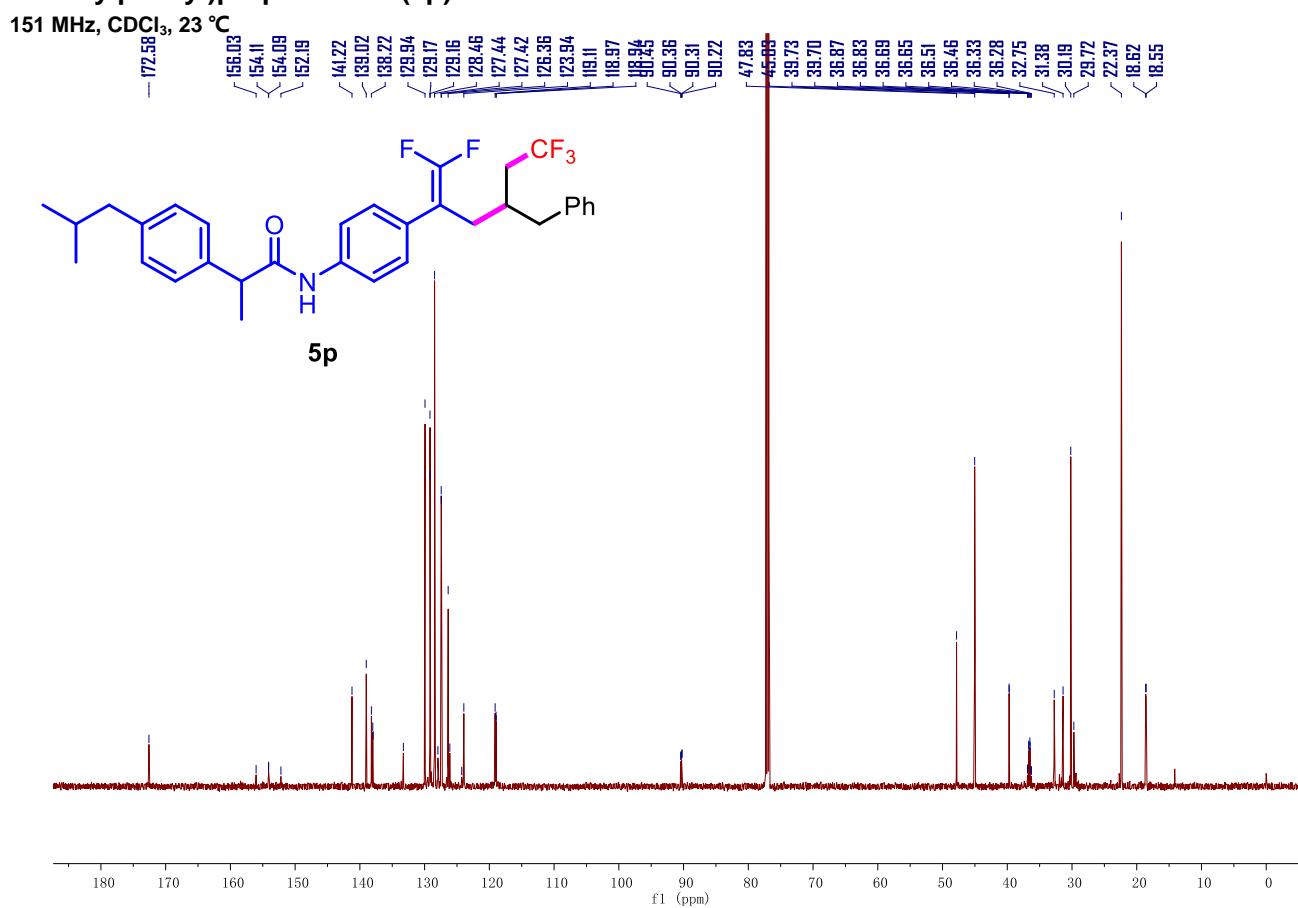
565 MHz, CDCl₃, 23 °C



¹H NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-2-(4-isobutylphenyl)propanamide (5p)

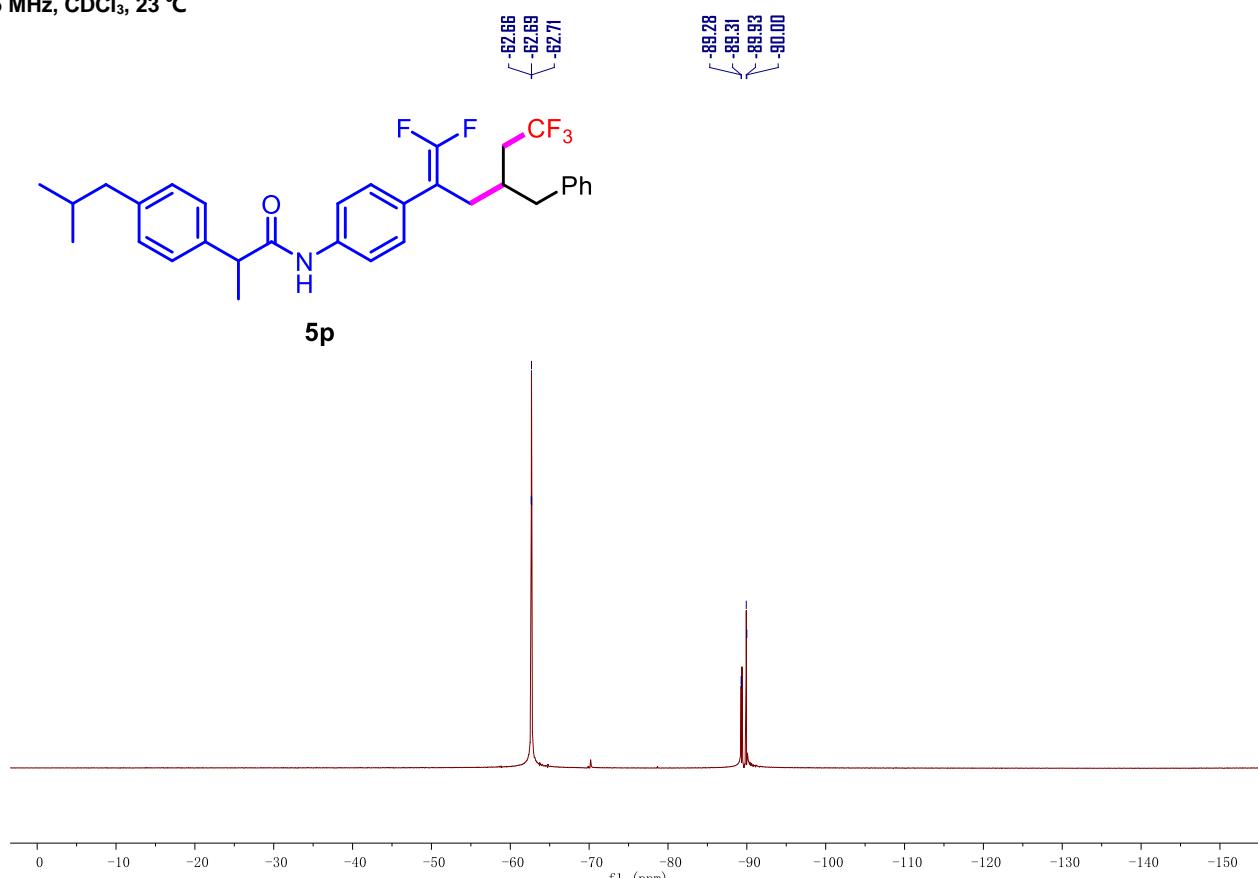


¹³C NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-2-(4-isobutylphenyl)propanamide (5p)



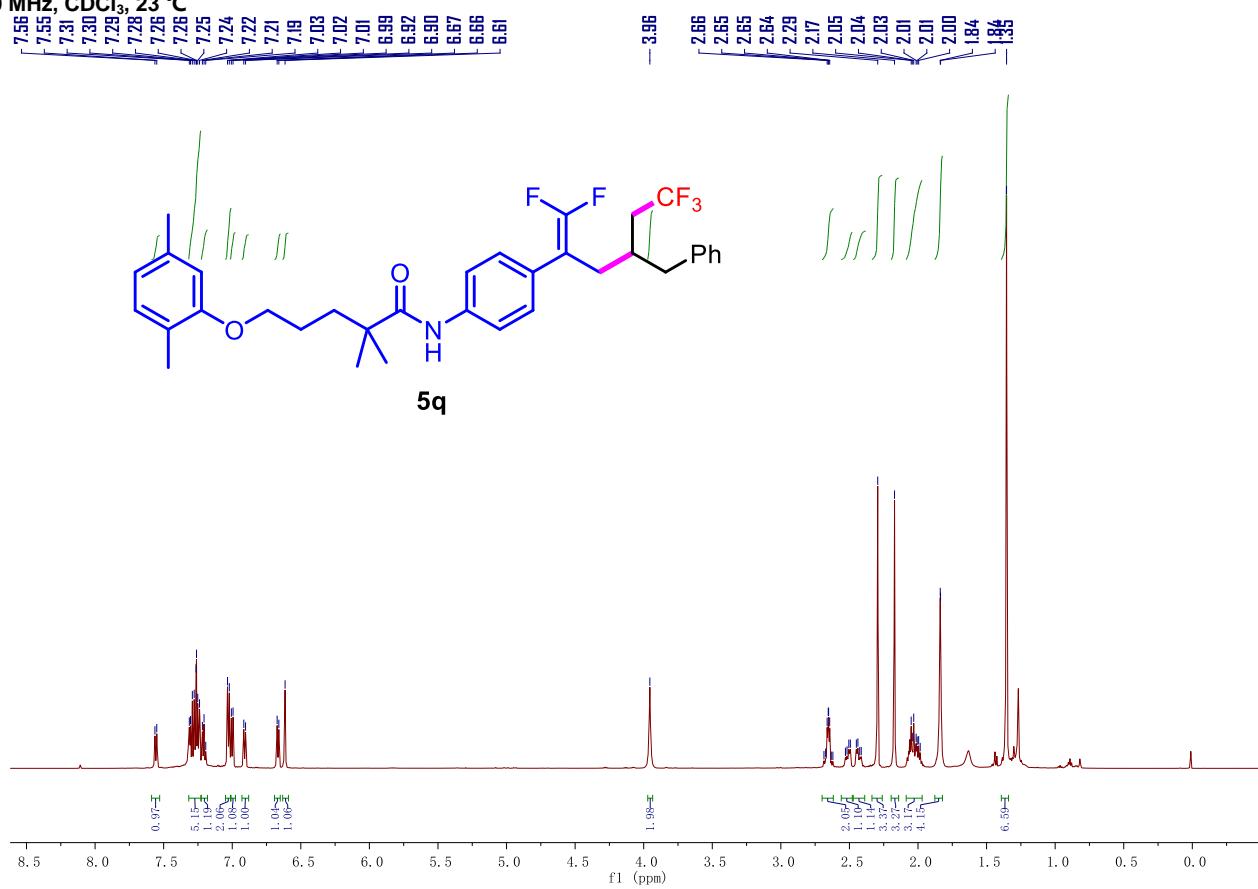
¹⁹F NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-2-(4-isobutylphenyl)propanamide (5p)

565 MHz, CDCl₃, 23 °C

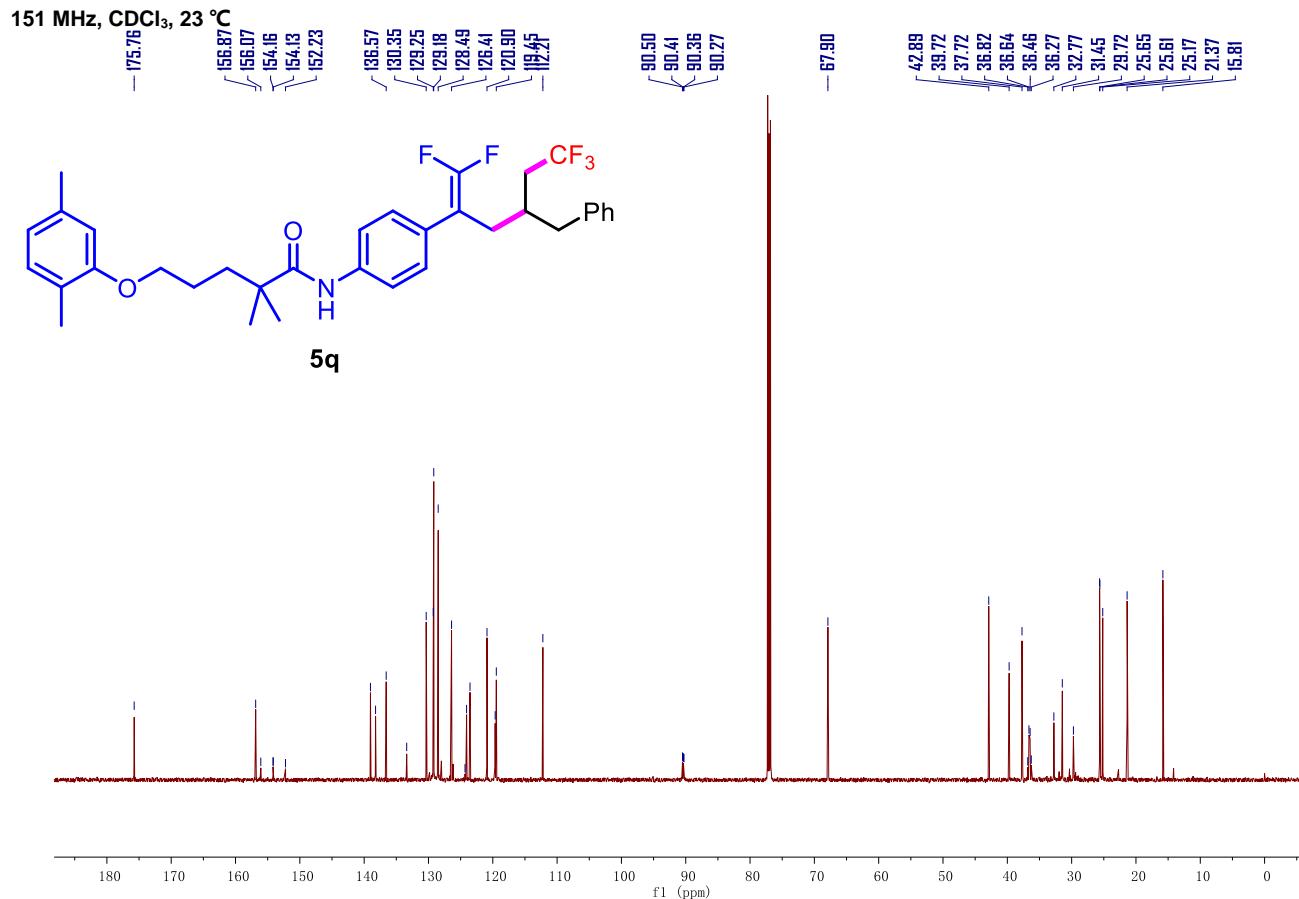


¹H NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-5-(2,5-dimethylphenoxy)-2,2-dimethylpentanamide (5q)

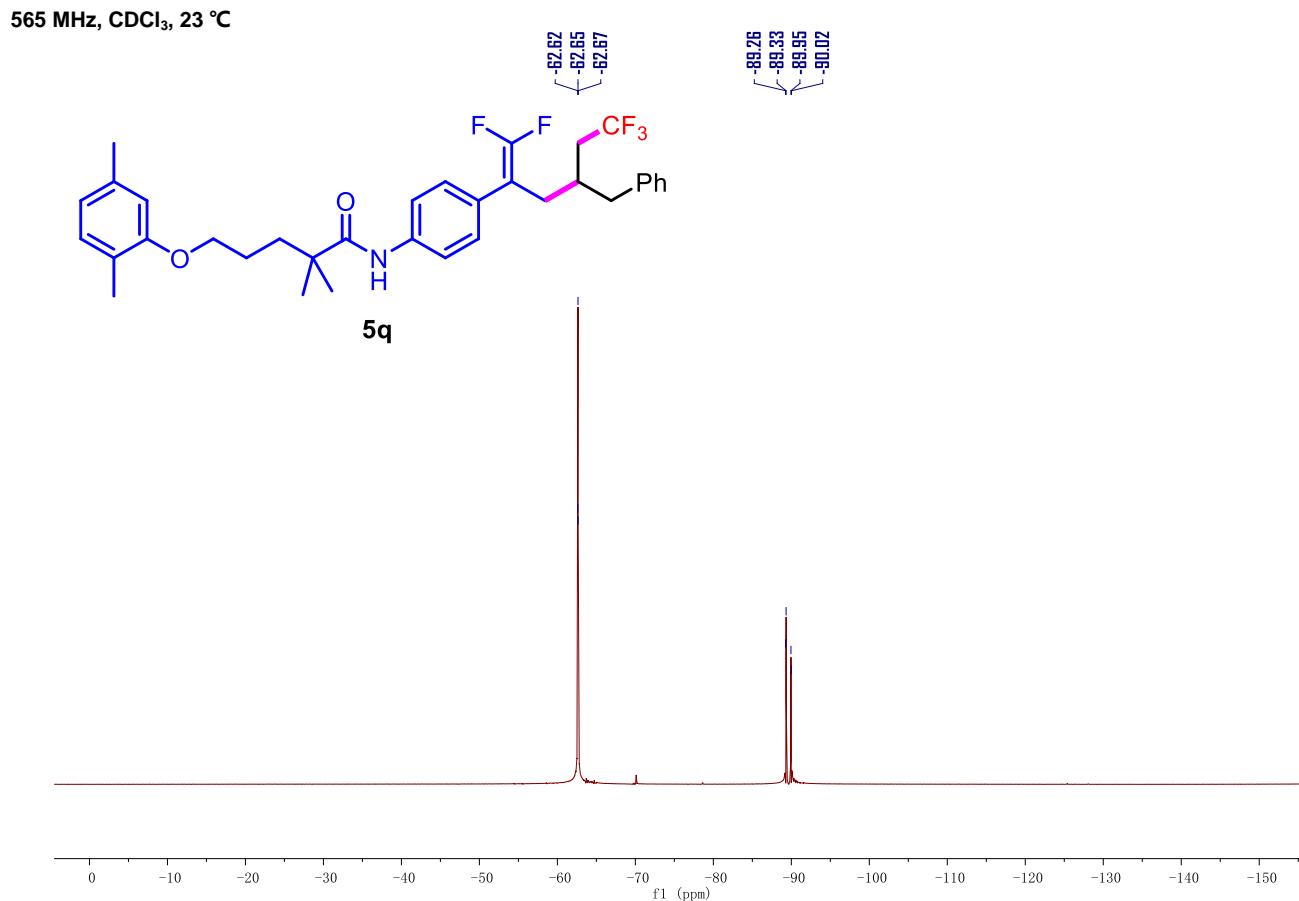
600 MHz, CDCl₃, 23 °C



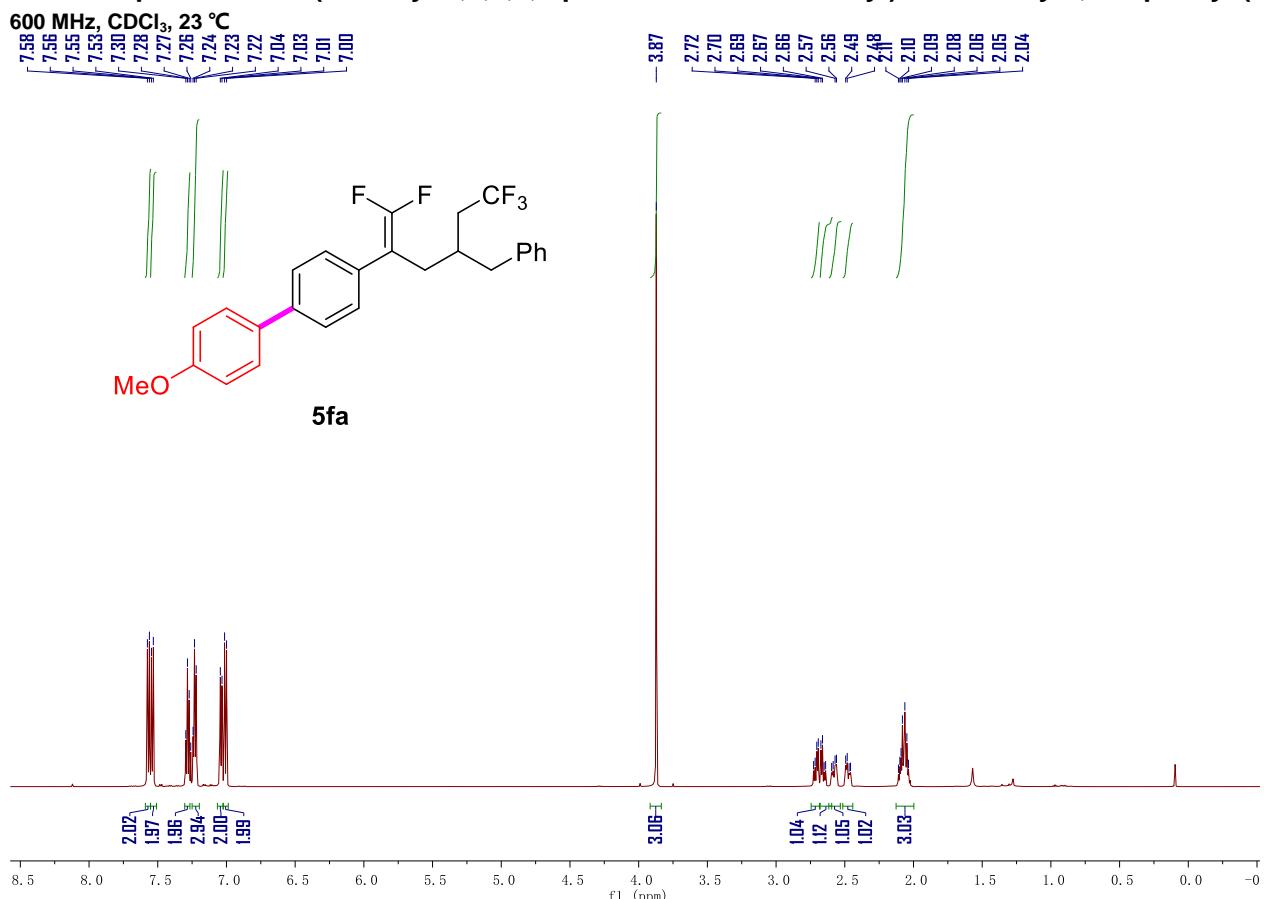
¹³C NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-5-(2,5-dimethylphenoxy)-2,2-dimethylpentanamide (5q)



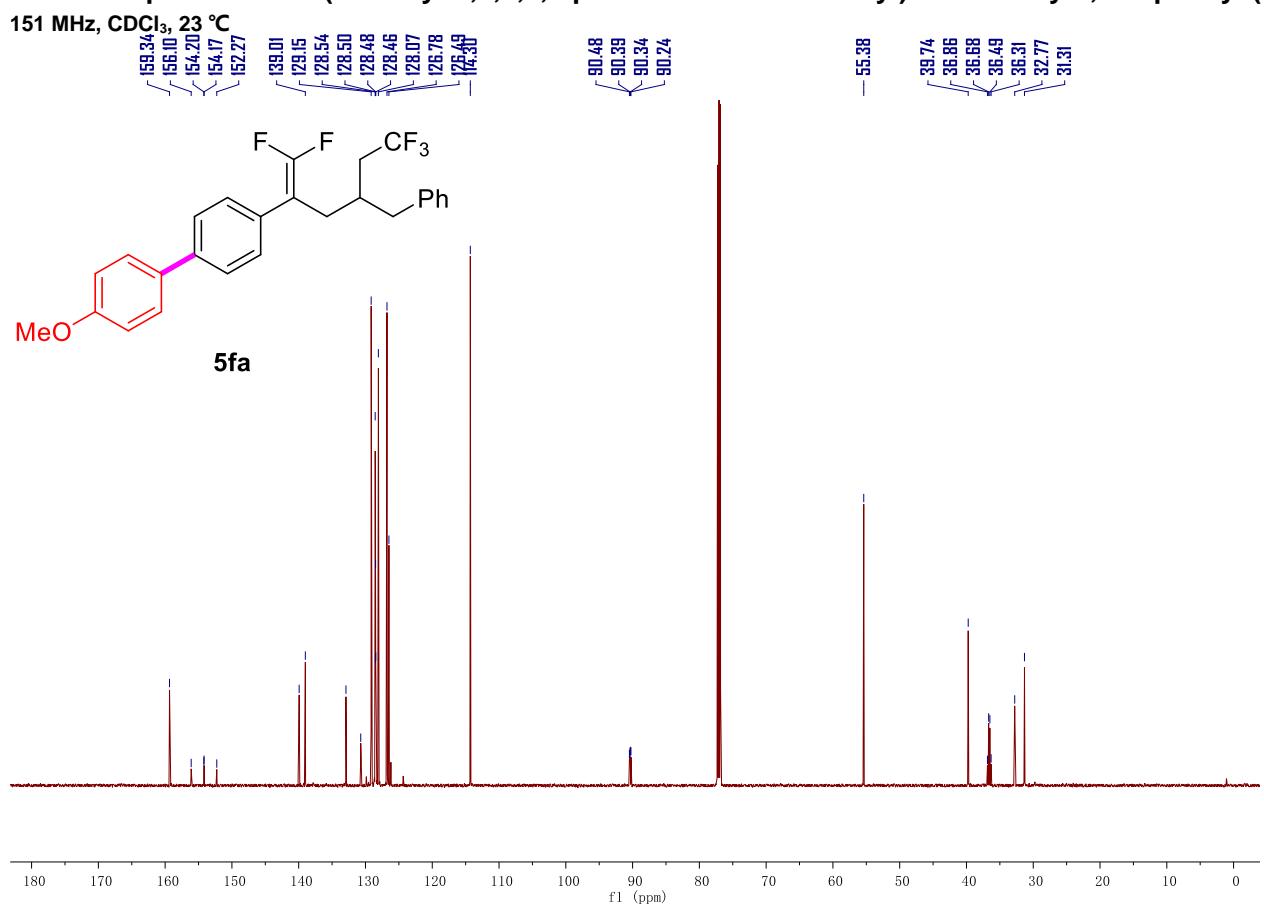
¹⁹F NMR spectrum of *N*-(4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)phenyl)-5-(2,5-dimethylphenoxy)-2,2-dimethylpentanamide (5q)



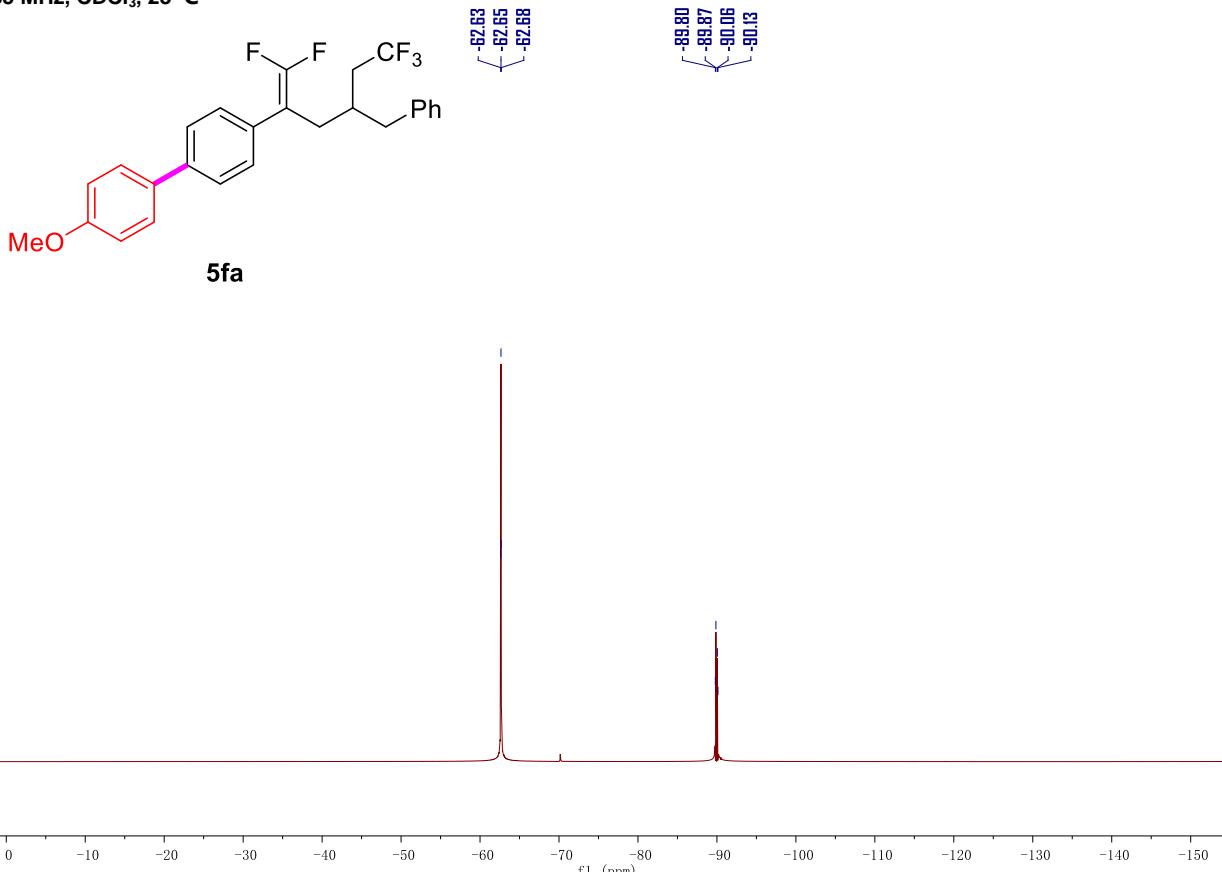
¹H NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4'-methoxy-1,1'-biphenyl (5fa)



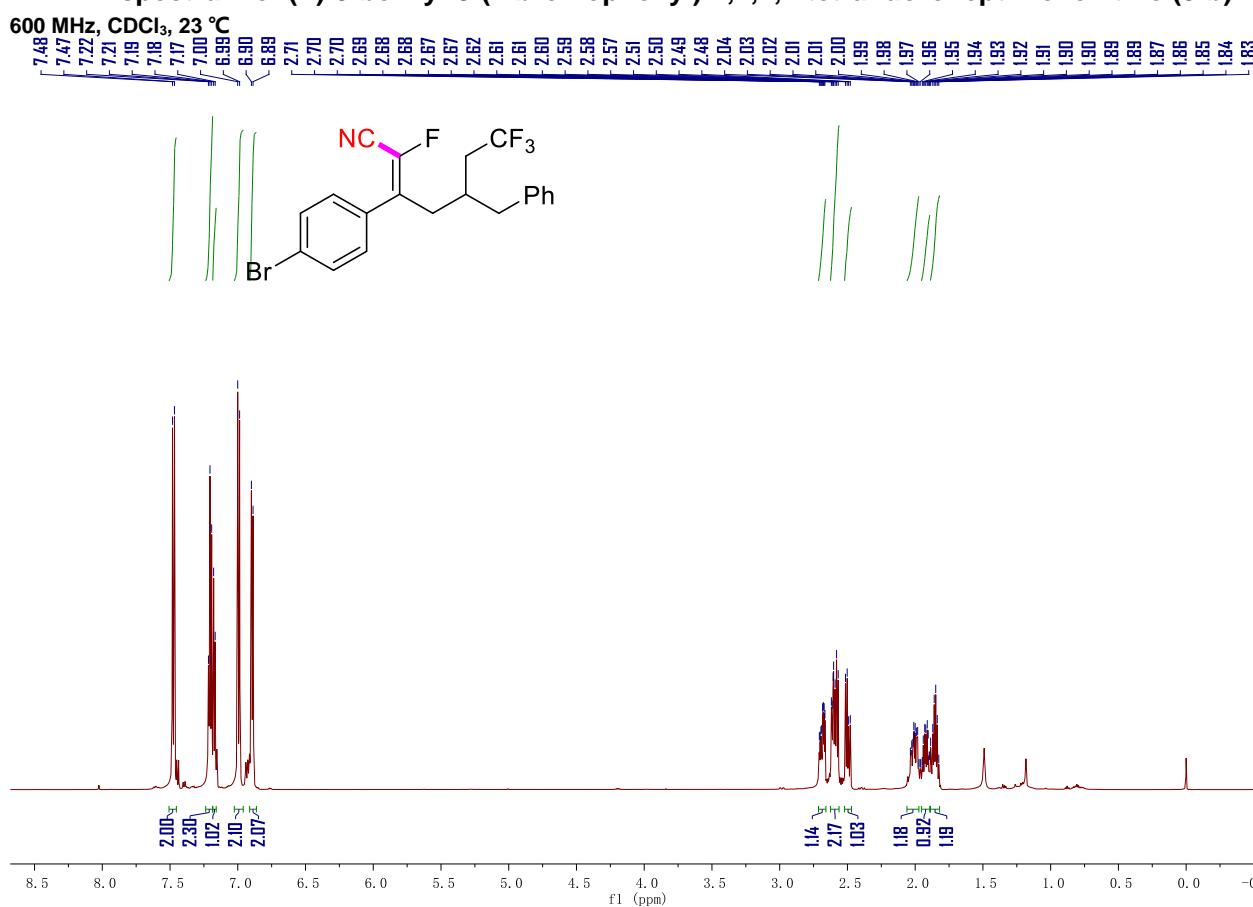
¹³C NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4'-methoxy-1,1'-biphenyl (5fa)



¹⁹F NMR spectrum of 4-(4-benzyl-1,1,6,6,6-pentafluorohex-1-en-2-yl)-4'-methoxy-1,1'-biphenyl (5fa)
565 MHz, CDCl₃, 23 °C

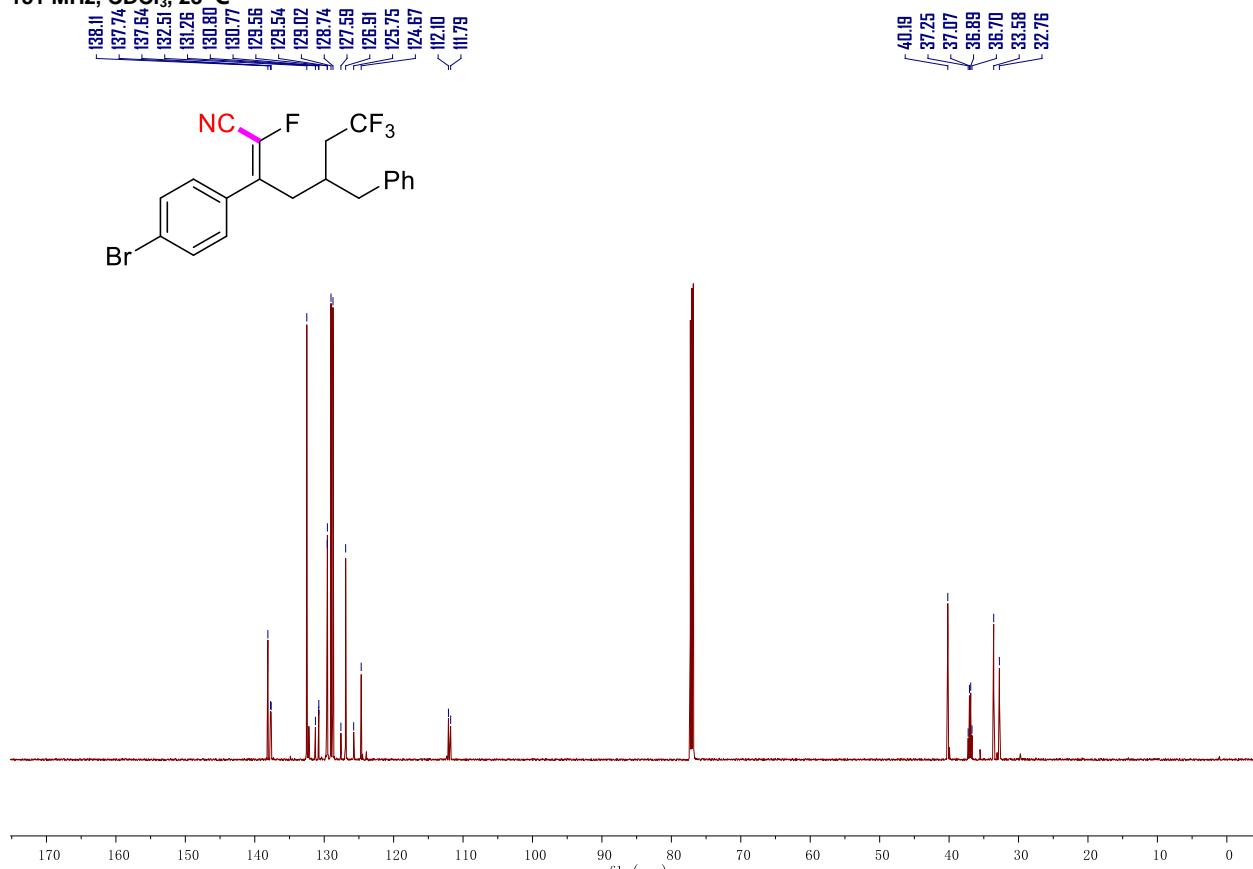


¹H NMR spectrum of (E)-5-benzyl-3-(4-bromophenyl)-2,7,7,7-tetrafluorohept-2-enenitrile (5fb)



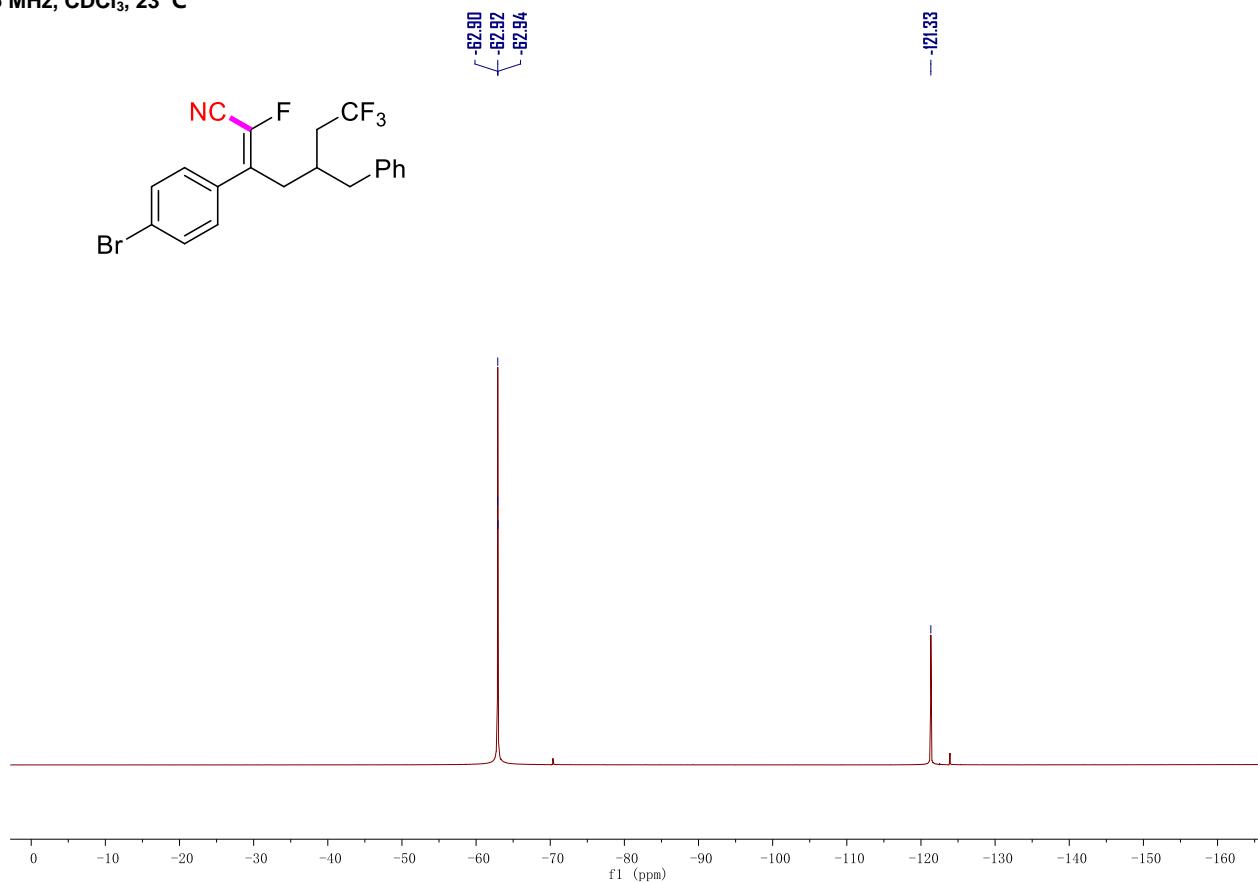
¹³C NMR spectrum of (*E*)-5-benzyl-3-(4-bromophenyl)-2,7,7,7-tetrafluorohept-2-enenitrile (5fb)

151 MHz, CDCl₃, 23 °C

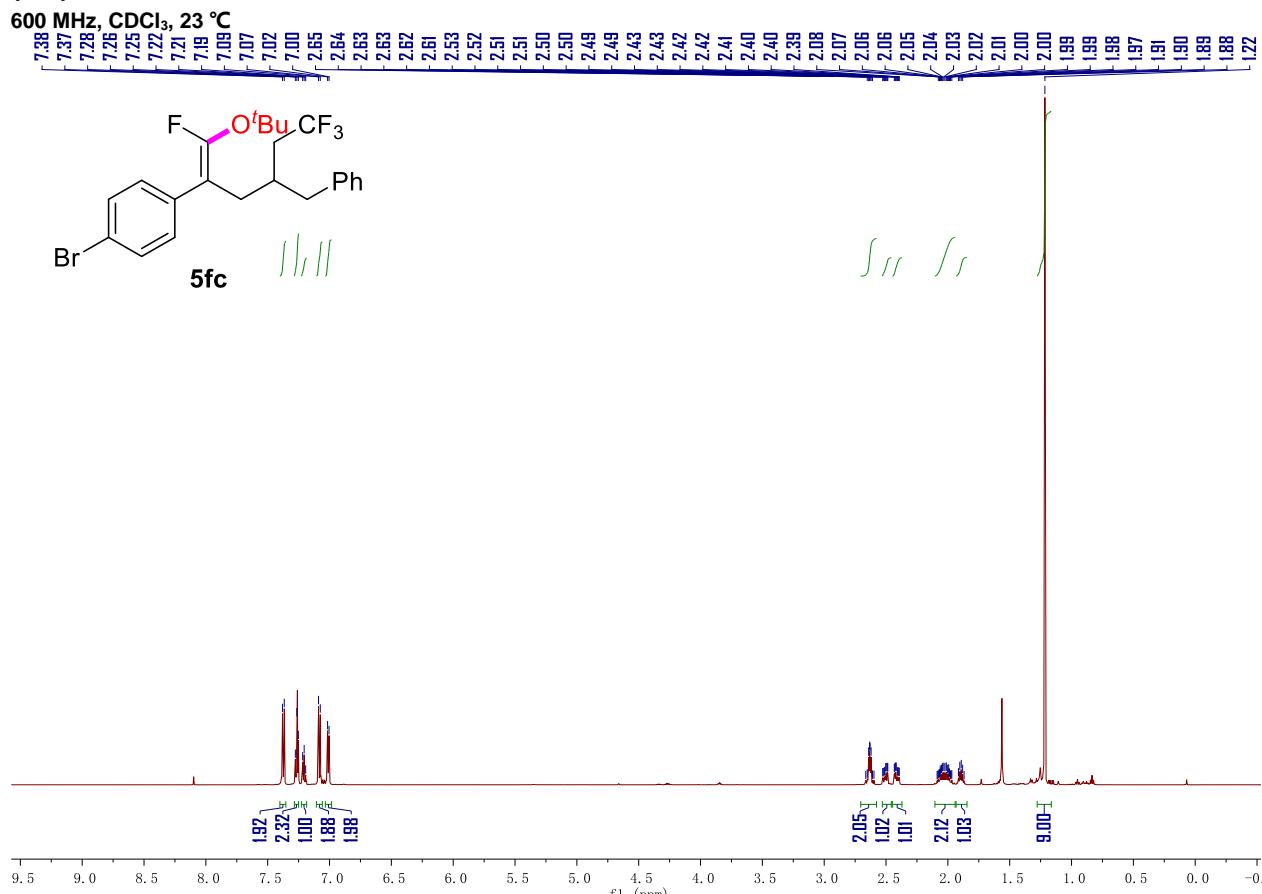


¹⁹F NMR spectrum of (*E*)-5-benzyl-3-(4-bromophenyl)-2,7,7,7-tetrafluorohept-2-enenitrile (5fb)

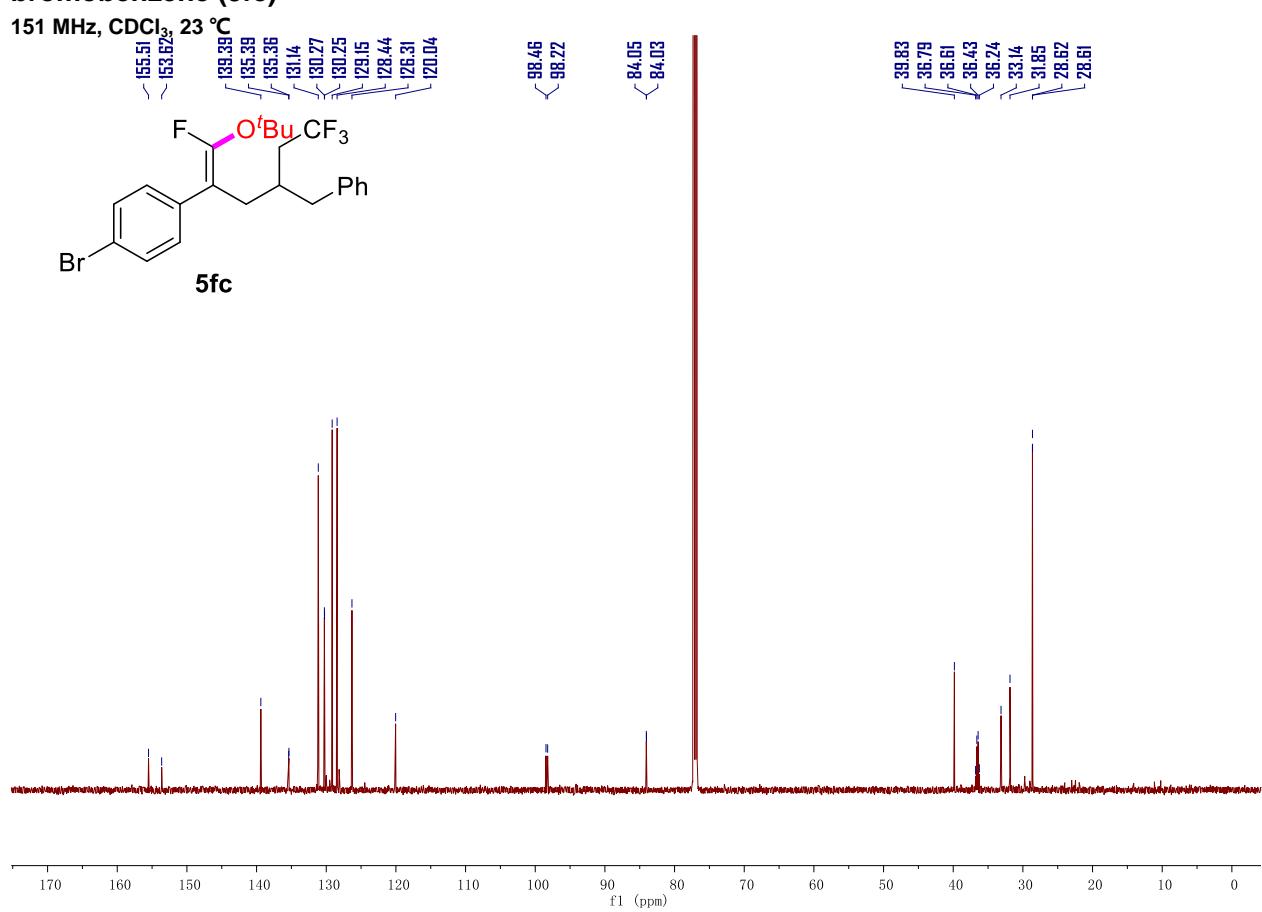
565 MHz, CDCl₃, 23 °C



¹H NMR spectrum of (Z)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)

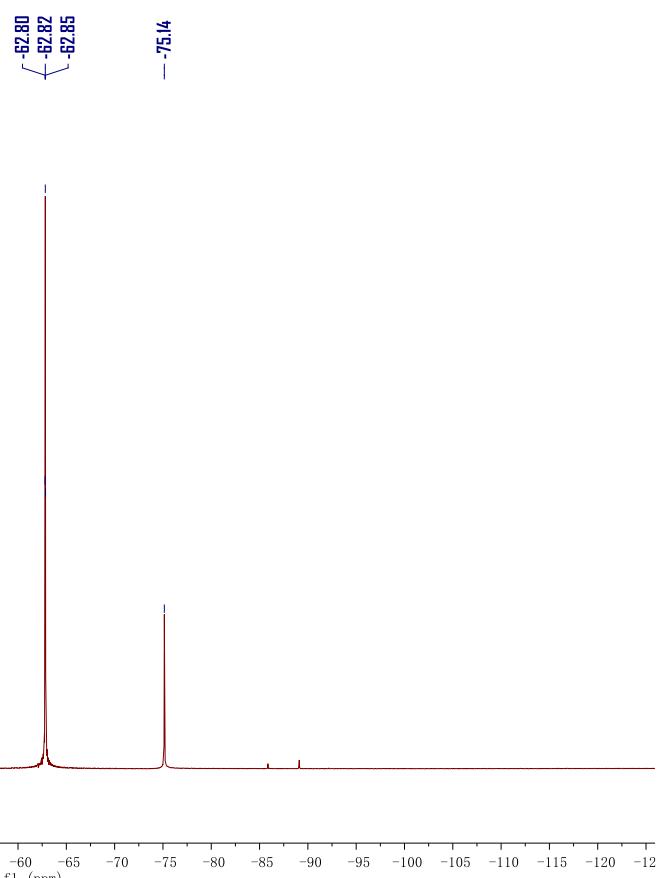
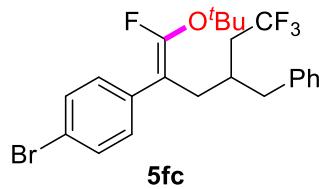


¹³C NMR spectrum of (Z)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)



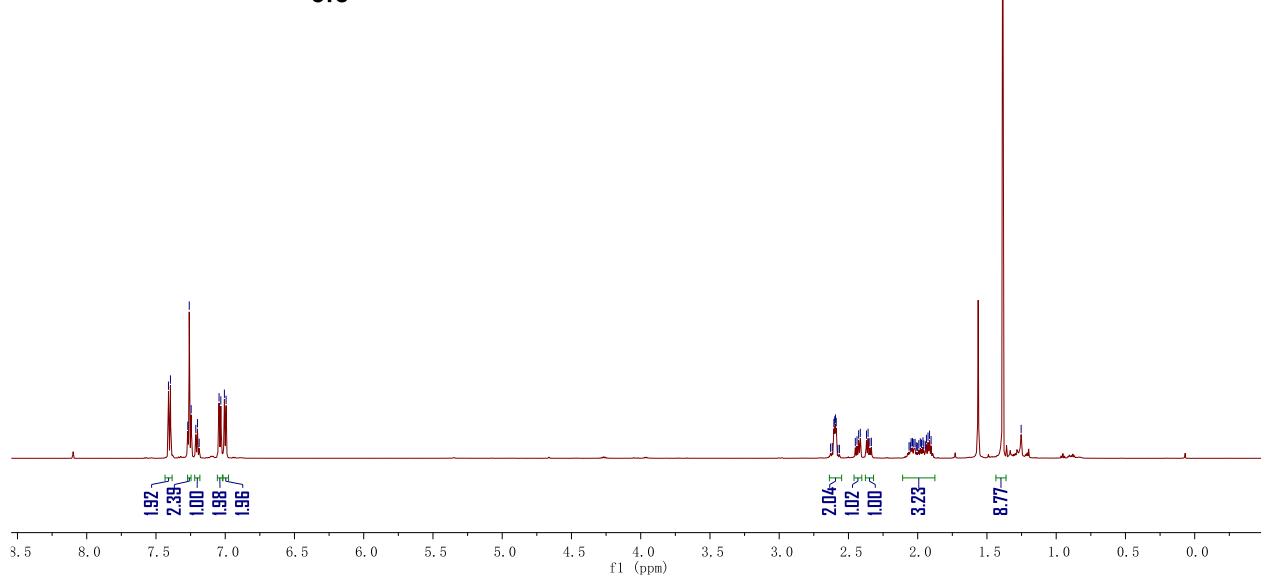
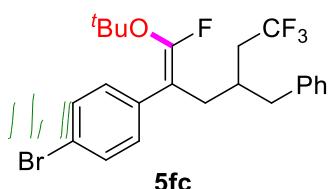
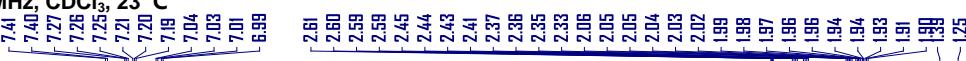
¹⁹F NMR spectrum of (Z)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)

565 MHz, CDCl₃, 23 °C

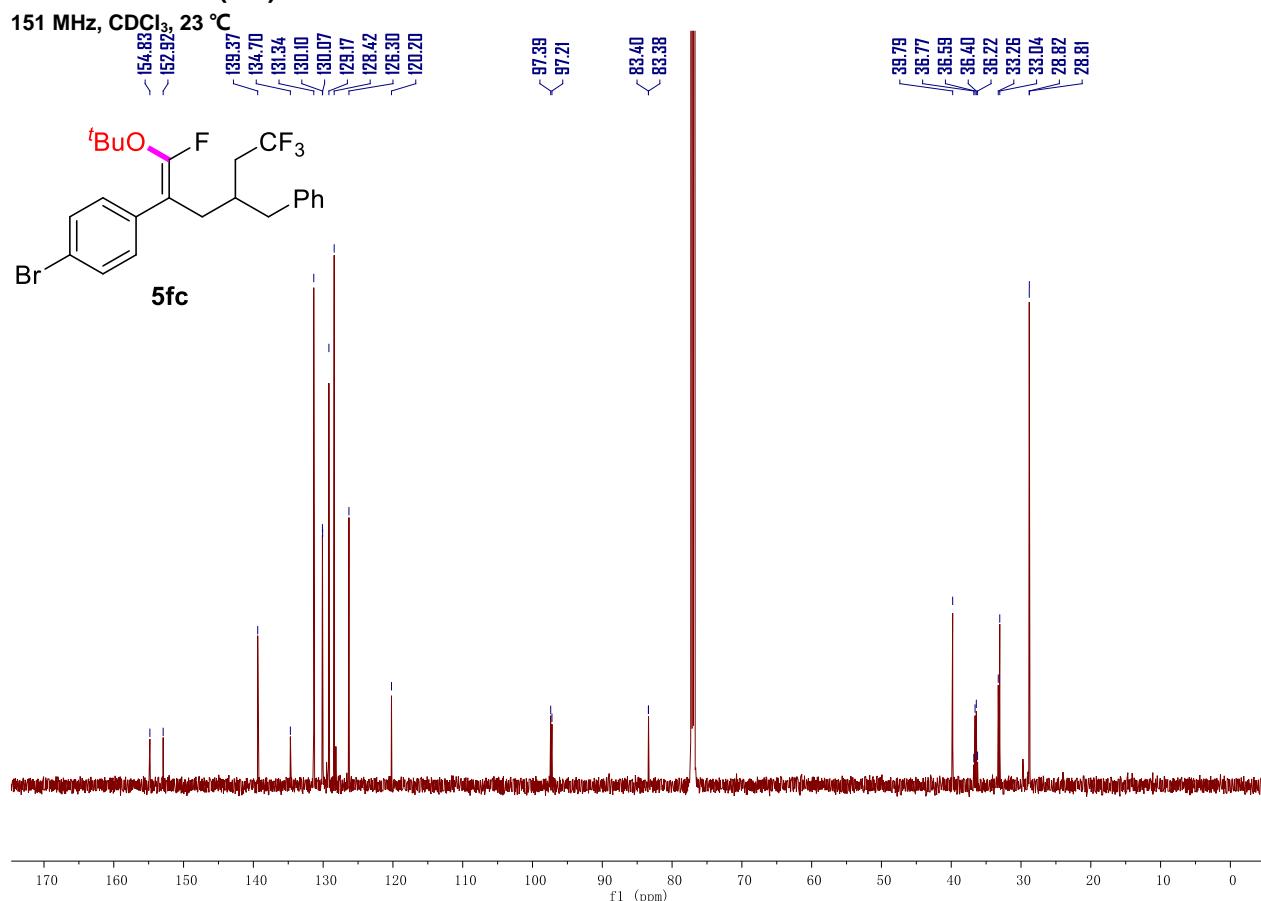


¹H NMR spectrum of (E)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)

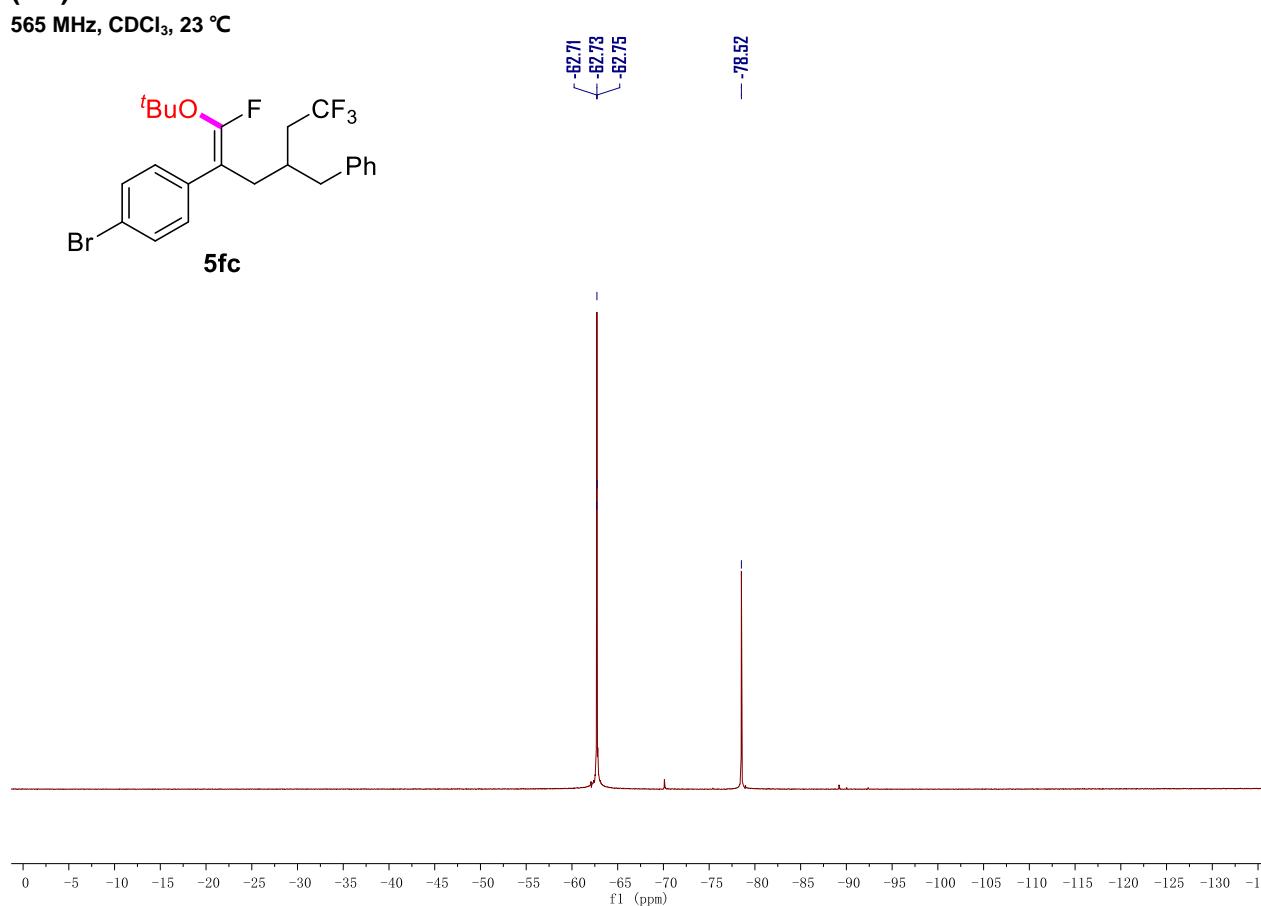
600 MHz, CDCl₃, 23 °C



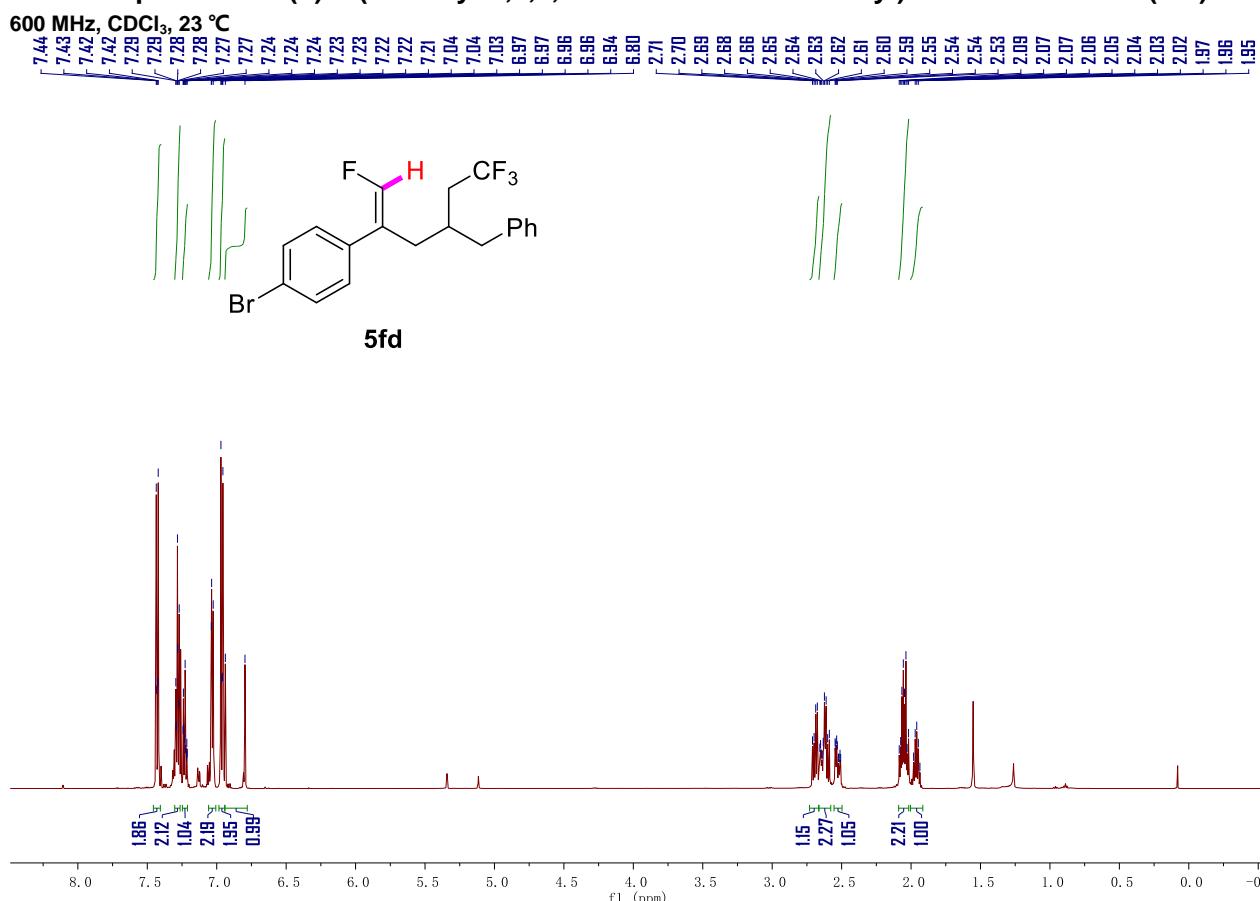
¹³C NMR spectrum of (*E*)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)



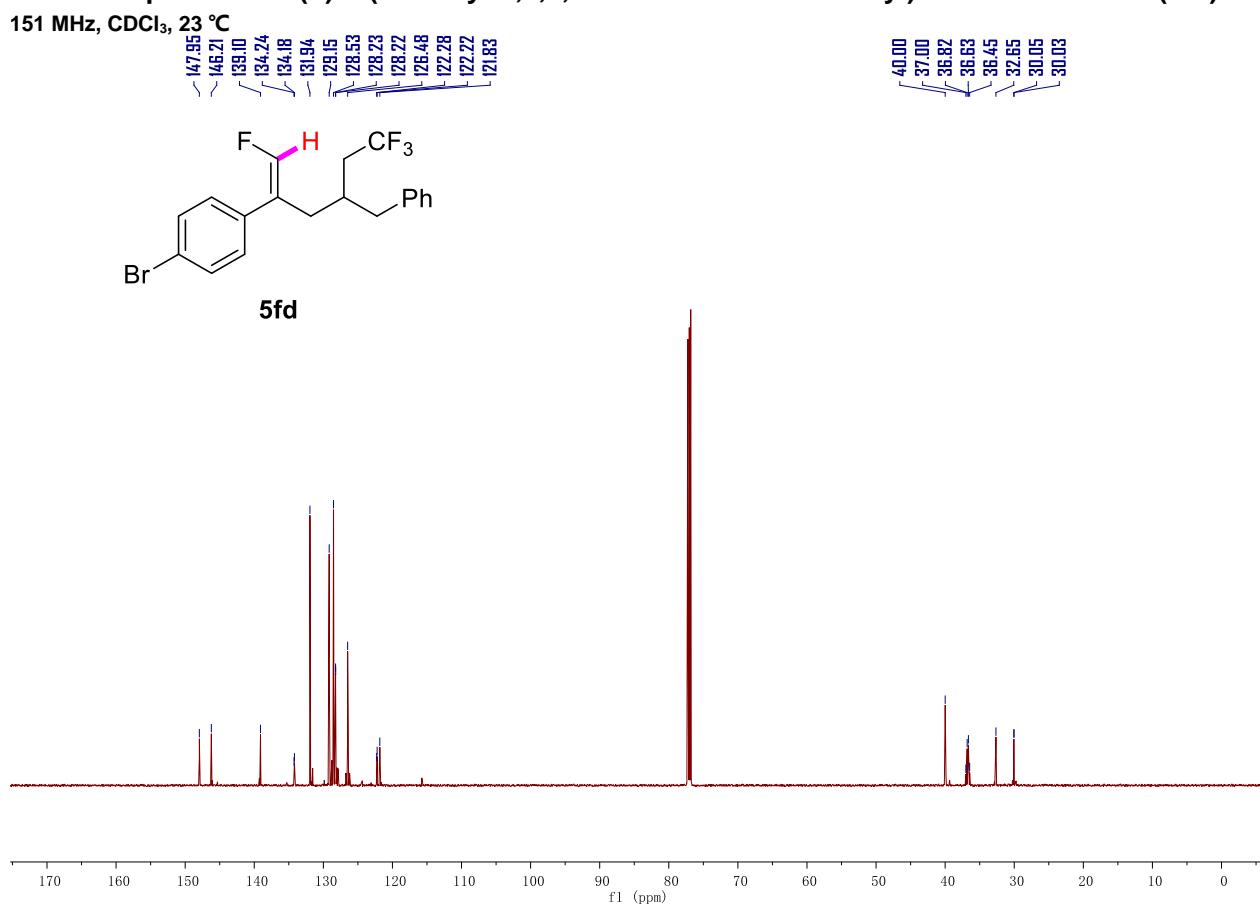
¹⁹F NMR spectrum of (*E*)-1-(4-benzyl-1-(tert-butoxy)-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fc)



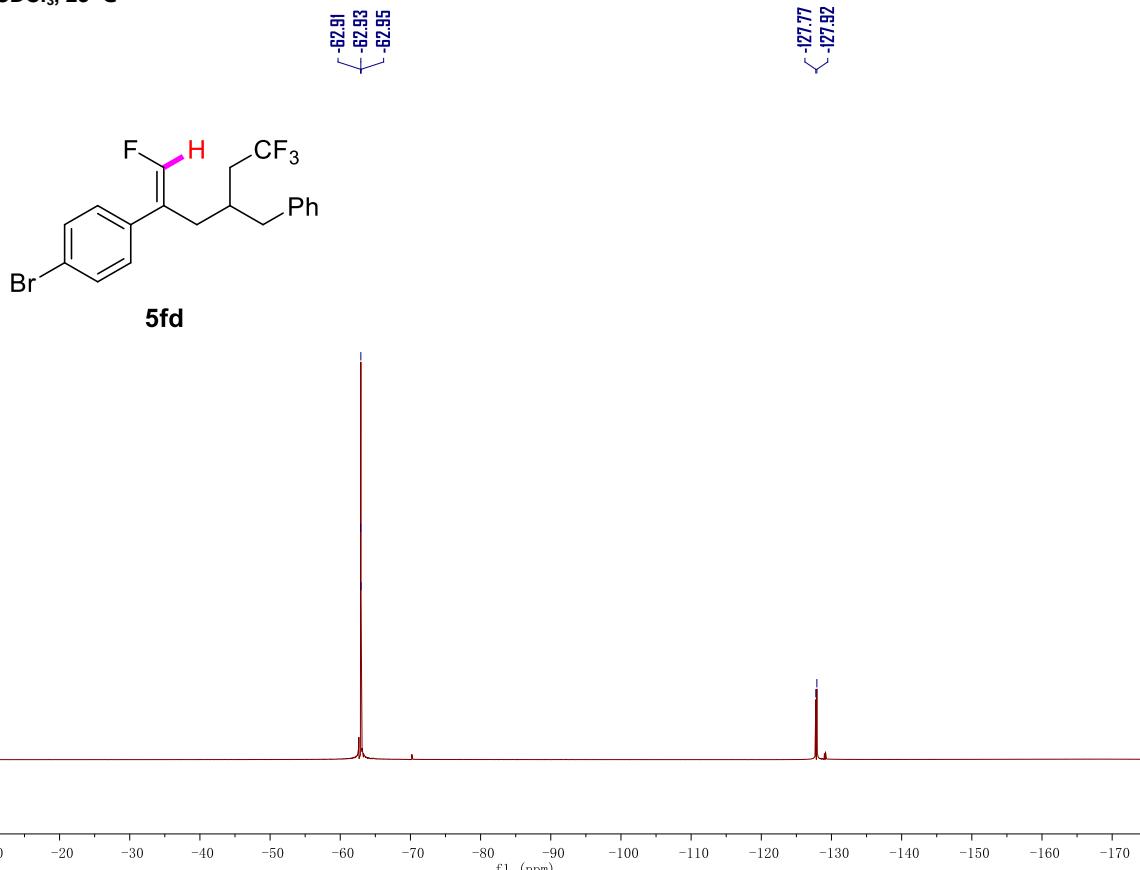
¹H NMR spectrum of (Z)-1-(4-benzyl-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fd)



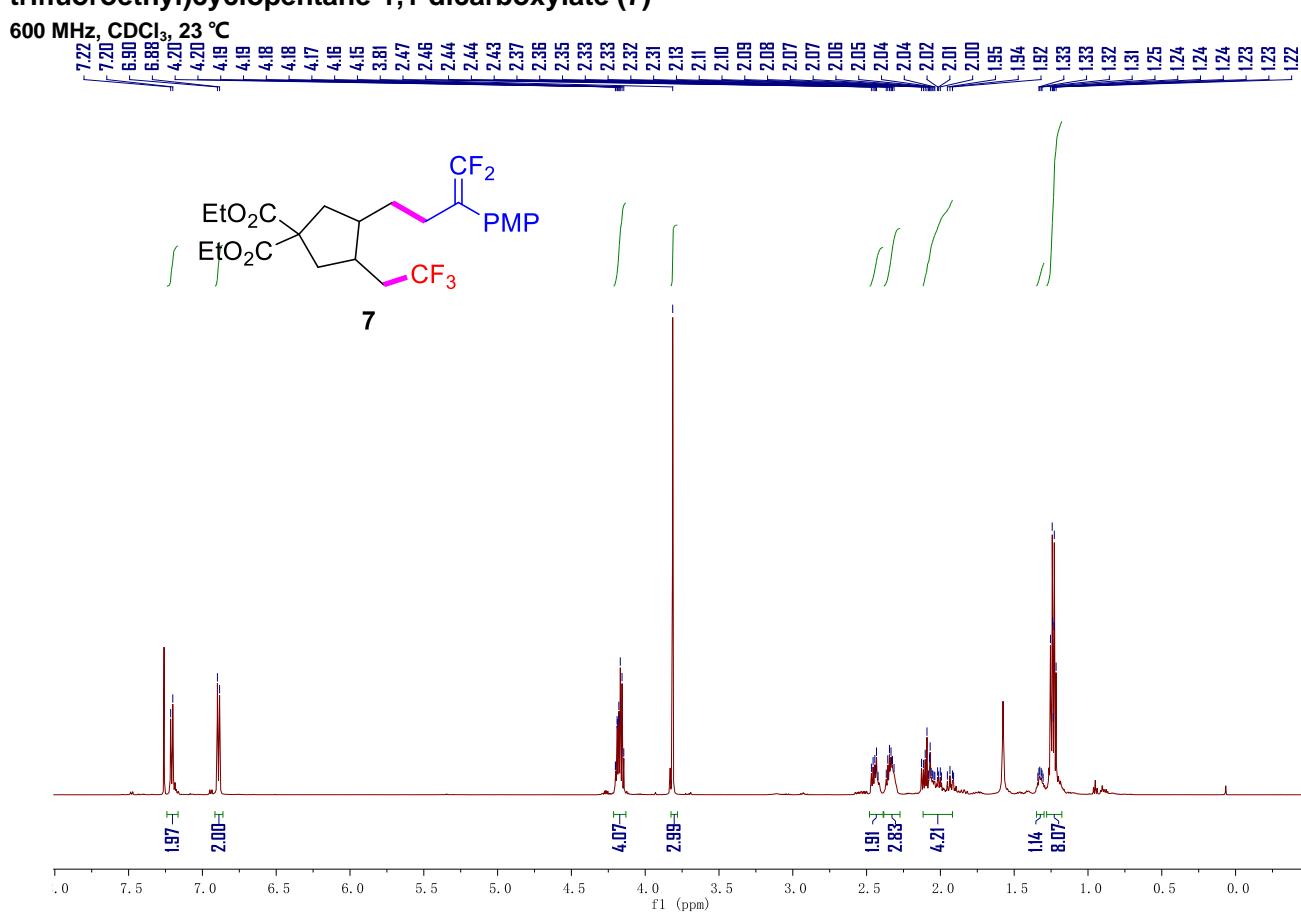
¹³C NMR spectrum of (Z)-1-(4-benzyl-1,6,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fd)



¹⁹F NMR spectrum of (Z)-1-(4-benzyl-1,6,6-tetrafluorohex-1-en-2-yl)-4-bromobenzene (5fd)
565 MHz, CDCl₃, 23 °C

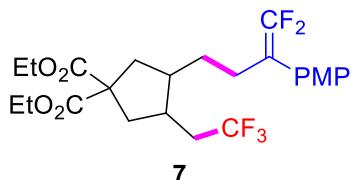


¹H NMR spectrum of diethyl 3-(4,4-difluoro-3-(4-methoxyphenyl)but-3-en-1-yl)-4-(2,2,2-trifluoroethyl)cyclopentane-1,1-dicarboxylate (7)



¹³C NMR spectrum of diethyl 3-(4,4-difluoro-3-(4-methoxyphenyl)but-3-en-1-yl)-4-(2,2,2-trifluoroethyl)cyclopentane-1,1-dicarboxylate (7)

151 MHz, CDCl₃, 23 °C



¹⁹F NMR spectrum of diethyl 3-(4,4-difluoro-3-(4-methoxyphenyl)but-3-en-1-yl)-4-(2,2,2-trifluoroethyl)cyclopentane-1,1-dicarboxylate (7)

565 MHz, CDCl₃, 23 °C

