Supporting Information

Direct oxidative isoperfluoropropylation of terminal alkenes via hexafluoropropylene (HFP) and silver fluoride

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Table of Contents

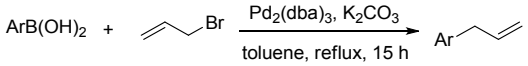
1 General information

Unless noted otherwise, the reactions were performed in oven-dried glassware containing a Teflon-coated stirrer bar and dry septum under a nitrogen atmosphere. Room temperature (rt) was range from 17 °C to 26 °C. ^{1}H NMR spectra were recorded on a Agilent 400 spectrometer (400 MHz) spectrometer with residual solvent peak as internal reference. ^{19}F NMR spectra were taken on a Agilent 400 spectrometer (376 MHz). ^{13}C NMR spectra were taken a Bruker AM-400 spectrometer (100.5 MHz) or Agilent 400 spectrometer (100.5 MHz) with residual solvent peak as internal reference. $CDCl_3$ was referenced to 7.26 ppm in ^{1}H NMR and 77.16 ppm in ^{13}C spectra. ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ as outside standard. Chemical shifts (δ) are reported in ppm, and coupling constants (*J*) are in Hertz (Hz). The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad. Melting points were measured on a Melt-Temp apparatus and were uncorrected. IR spectra were obtained with a Nicolet AV-360 spectrophotometer. High resolution mass spectra (HRMS) were performed using FinniganMIT-8430 (EI) and Bruker ApeXIII 7.0 TESLA FTMS, IonSpec 4.7 Tesla FTMS (ESI) high-resolution mass spectrometer. TLC analysis was performed on silica gel plates, Column chromatography over silica gel (mesh 300-400) and hexane (petroleum ether)/ethyl acetate or pentane/ dichloromethane were used as the eluent.

Acetonitrile was dried by refluxing over CaH_2 and subsequent distillation. Benzoyl peroxide and cuprous iodide were freshly purified according to the purification handbook *Purification of Laboratory Chemicals* before using. Unless otherwise noted, all other reagents were purchased from commercial suppliers and used as received.

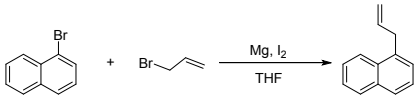
2 Preparation of Substrates

2.1 General procedure for preparation of **1c**, **1d**, **1e**.[1]



[$Pd_2(dba)_3$] (48.1 mg, 0.0525 mol), allyl bromide (90.7 mg, 0.75 mmol), K_2CO_3 (932 mg, 6.75 mmol), arylboronic acid (198 mg, 1 mmol) and dry toluene (5 mL) were placed in a two-necked round-bottom flask under nitrogen. The reaction mixture was heated at reflux for 15h, monitoring by GC. Then hydrogen peroxide (10 mL) was added, the mixture was stirred at room temperature for 30 min and the toluene layer was separated. The aqueous layer was extracted three times with diethyl ether. The combined organic extracts were washed with brine and dried with $MgSO_4$. The solvent was removed under reduced pressure and the crude product was purified by column chromatography on silica gel to give desired product.

2.2 Procedure for preparation of **1f**.[2]

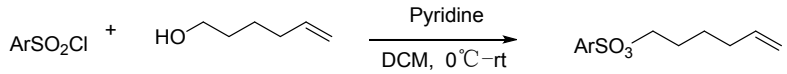


A flame-dried vial equipped with a magnetic stir bar was charged with Mg granules (230 mg, 9.6 mmol) and iodide grain and sealed with a septum. The vial was evacuated and backfilled with N_2 for three times. Part of a solution of 1-bromonaphthalene (1657 mg, 8 mmol) in THF (10 mL) was added into the vial via a syringe. After the color suddenly faded, the rest of solution was added. The solution was stirred for 2 h under N_2. The mixture was decanted into another dry flask. It was then cooled to 0℃ and allyl bromide (1452 mg, 12 mmol) was added carefully. The resulting solution was stirred for 45 min at 0 ℃ and quenched with saturated solution of NH_4Cl. The solution was then extracted with 3×10 mL diethyl ether and the combined organic layers were dried over $MgSO_4$. The solvent was then removed in vacuo and the residue was purified by flash column chromatography to afford the corresponding alkene.

2.3 General procedure for preparation of **1i**, **1j**, **1k**.[3]

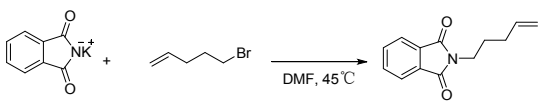
To a solution of hex-5-en-1-ol (2 mmol), Et_3N (4 mmol), DMAP (10 mol %) in CH_2Cl_2 (5 mL) was added dropwise with the corresponding acyl chloride (3 mmol) dissolved in CH_2Cl_2 at 0℃. The resulted mixture was vigorously stirred at room temperature. The reaction was monitored by TLC. The reaction mixture was treated with saturated solution of $NaHCO_3$ (10 mL). After stirring at room temperature for 20 min, ethyl acetate (20 mL) was added. The organic layer was separated, and washed with water (3×10 mL). The combined organic extracts were washed with brine (50 mL), and dried over $MgSO_4$. After evaporation of the solvent, the crude product was purified by chromatography on silica gel to give the product.

2.4 General procedure for preparation of substrates **1l**, **1m**.[2]



Hex-5-en-1-ol (2.2 mmol, 1.1 equiv.) and pyridine (2.2 mmol, 2.2 equiv.) were dissolved in 2 mL dry DCM. Sulfonyl chloride (2.0 mmol, 1equiv.) was then added dropwise to the solution at 0℃. After stirring for 1h at room temperature, the salt was filtered and the solvent was removed in vacuo. The residue was purified by silica gel flash column chromatography to provide the corresponding alkenes.

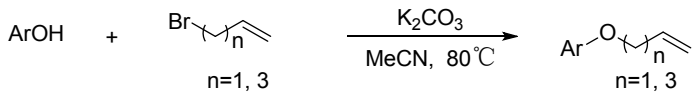
2.5 Procedure for preparation of **1n**.[2]



5-Bromopent-1-ene (1.19 g, 8 mmol) and potassium phthalimide (1.48 g, 8 mmol) were then dissolved in 14 mL DMF. With stirring, the solution was heated overnight (45℃). After filtration,

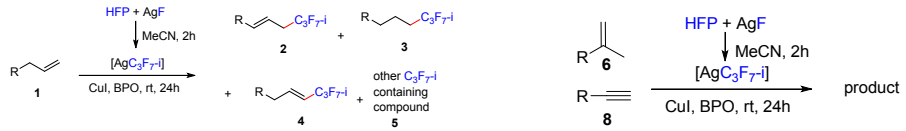
the mixture was washed with 50 mL brine and was extracted with 50 mL Et_2O. The solution was dried over Na_2SO_4 and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to provide the corresponding alkene.

2.6 General procedure for preparation of **1g**, **1o**, **1p**, **1q**, **1r**, **1s**, **1t**, **1u**, **1v**, **1x**.[4]



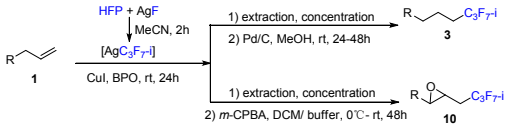
To a solution of aromatic alcohol (2.5 mmol, 1.0 eqiv.) and K_2CO_3 (7.5 mmol, 3 equiv.) in CH_3CN (10.0 mL) was added allyl bromide/5-bromopent-1-ene (5.0 mmol, 2.0 equiv.), and the mixture was refluxed for 12h. It was then cooled to 25°C and the solvent was removed in vacuo. The residue was partitioned between diethyl ether and water, and the aqueous layer was extracted with diethyl ether (3 x 10 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$ and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to provide the corresponding alkenes.

3 General produce of direct oxidative isoperfluoropropylation of terminal alkenes, 1,1-disubstituted alkenes and terminal alkynes via hexafluoropropylene and silver fluoride (Scheme 2 and 3)



In a nitrogen-filled glove box, an oven-dried crimp cap vessel with Teflon-coated stirrer bar was charged with silver fluoride (50.8 mg, 0.4 mmol) and was brought under an atmosphere of dry nitrogen. To this vessel, anhydrous acetonitrile (2 mL) and hexafluoropropylene (balloon, adequate) were added, and the mixture was stirred at ice-water bath in the dark until silver fluoride precipitate dissolved completely. Then this solution was added to another oven-dried vessel, which filling with copper iodide (61 mg, 0.32 mmol) and benzoyl peroxide (72.7 mg, 0.3 mmol). In the end, olefin or alkyne (**1**, **6** or **8**, 0.2 mmol) was added. The reaction mixture was stirred at ambient temperature for 24 hours. The resulting mixture was diluted with diethyl ether (10 mL), then filtered through a short pad of celite and rinsed with diethyl ether. The resulting organic solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The residue was further purified by flash chromatography on silica gel to give the desired oxidative isoperfluoropropylation products.

4 Further conversion of the allylic isoperfluoropropylated compounds (Scheme 4)



4.1 Reduction of the allylic isoperfluoropropylated compounds

In a nitrogen-filled glove box, an oven-dried mL crimp cap vessel with Teflon-coated stirrer bar was charged with silver fluoride (101.6 mg, 0.8 mmol) and was brought under an atmosphere of dry nitrogen. To this vessel, anhydrous acetonitrile (3 mL) and hexafluoropropylene (balloon, adequate) were added, and the mixture was stirred at ice-water bath in the dark until silver fluoride precipitate dissolved completely. Then this solution was added to another oven-dried vessel, which filling with copper iodide (122 mg, 0.64 mmol) and benzoyl peroxide (145.4 mg, 0.6 mmol). In the end, olefin (**1**, 0.4 mmol) was added. The reaction mixture was stirred at ambient temperature for 24 hours. The resulting mixture was diluted with diethyl ether (20 mL), then filtered through a short pad of celite and rinsed with diethyl ether. The resulting organic solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated.

The residue was added MeOH (5 mL) and 5% Pd/C (85.2 mg, 0.04 mmol), then introduced hydrogen and heated to 25℃ at constant pressure for 24-48 h. The resulting mixture was diluted with diethyl ether (10 mL), then filtered through a short pad of celite and rinsed with diethyl ether. The resulting organic solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The residue was further purified by flash chromatography on silica gel to give the desired products **3a** and **3x**.

4.2 Epoxidation of the allylic isoperfluoropropylated compound[17]

In a nitrogen-filled glove box, an oven-dried mL crimp cap vessel with Teflon-coated stirrer bar was charged with silver fluoride (50.8 mg, 0.4 mmol) and was brought under an atmosphere of dry nitrogen. To this vessel, anhydrous acetonitrile (2 mL) and hexafluoropropylene (balloon, adequate) were added, and the mixture was stirred at ice-water bath in the dark until silver fluoride precipitate dissolved completely. Then this solution was added to another oven-dried vessel, which filling with copper iodide (61 mg, 0.32 mmol) and benzoyl peroxide (72.7 mg, 0.3 mmol). In the end, Allylbenzene (**1a**, 0.2 mmol) was added. The reaction mixture was stirred at ambient temperature for 24 hours. The resulting mixture was diluted with diethyl ether (10 mL), then filtered through a short pad of celite and rinsed with diethyl ether. The resulting organic solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated.

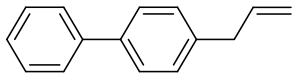
The residue was added CH_2Cl_2 (8 mL) and phosphate buffer (8 mL, pH= 7.0). Then 3-Chloroperbenzoic acid (*m*-CPBA, 172.6 mg, 1 mmol) was added at 0℃. After stirring for 48 h, the resulting solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The residue was further purified by flash chromatography on silica gel to give the epoxide **10**.

5 Radical trapping experiments (Scheme 5)

In a nitrogen-filled glove box, an oven-dried crimp cap vessel with Teflon-coated stirrer bar was charged with silver fluoride (50.8 mg, 0.4 mmol) and was brought under an atmosphere of dry nitrogen. To this vessel, anhydrous acetonitrile (2 mL) and hexafluoropropylene (balloon, adequate) were added, and the mixture was stirred at ice-water bath in the dark until silver fluoride precipitate dissolved completely. Then this solution was added to another oven-dried vessel, which filling with: **a)** copper iodide (61 mg, 0.32 mmol), benzoyl peroxide (72.7 mg, 0.3 mmol) and 2,2,6,6-Tetramethylpiperidinooxy (93.6 mg, 0.6 mmol); or **b)** copper iodide (61 mg, 0.32 mmol), benzoyl peroxide (72.7 mg, 0.3 mmol) and benzoquinone (43.2 mg, 0.4 mmol); or **c)** benzoyl peroxide (72.7 mg, 0.3 mmol) and4-(ethoxycarbonyl)benzenediazonium tetrafluoroborate (79.2 mg, 0.3 mmol). In the end, olefin (**1**, 0.2 mmol) was added. The reaction mixture was stirred at ambient temperature for 24 hours. The resulting mixture was added $PhCF_3$ as internal standard then conducted ^{19}F NMR and GC-MS analysis. The reaction mixture of **b)** and **c)** was diluted with Et_2O (10 mL) respectively, then filtered through a short pad of celite and rinsed with diethyl ether. The resulting organic solution was add into water (10 mL) and extracted by diethyl ether (3×10 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated. The residue was further purified by flash chromatography on silica gel to give the desired products **11** and **12**.

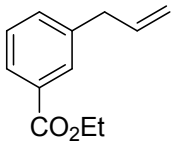
6 Spectral data for substrates

1c: 4-allyl-1,1'-biphenyl



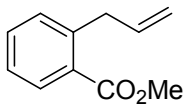
Known compound.[1] **1c** was obtained as a colorless oil in 67% yield (129 mg); hexane. ^1H NMR (400 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 20.3, 7.7 Hz, 4H), 7.47 (t, *J* = 7.3 Hz, 2H), 7.37 (t, *J* = 7.3 Hz, 1H), 7.31 (d, *J* = 7.6 Hz, 2H), 6.11-5.97 (m, 1H), 5.21 – 5.10 (m, 2H), 3.48 (d, *J* = 6.5 Hz, 2H).

1d: ethyl 3-allylbenzoate



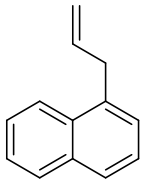
Known compound.[5] **1d** was obtained as a light yellow oil in 63% yield (119 mg); hexane/ ethyl acetate = 30/1. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.86 (m, 2H), 7.40 – 7.34 (m, 2H), 6.03 – 5.91 (m, 1H), 5.14 – 5.04 (m, 2H), 4.38 (q, *J* = 7.1 Hz, 2H), 3.44 (d, *J* = 6.6 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H).

1e: methyl 2-allylbenzoate



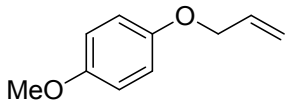
Known compound.[6] **1e** was obtained as a yellow oil in 33% yield (43 mg); petroleum ether/ ethyl acetate = 15/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.31 – 7.23 (m, 2H), 6.01 (ddt, *J* = 16.9, 10.3, 6.4 Hz, 1H), 5.02 (ddd, *J* = 16.8, 8.6, 1.6 Hz, 2H), 3.89 s, 3H), 3.76 (d, *J* = 6.4 Hz, 2H).

1f: 1-allylnaphthalene



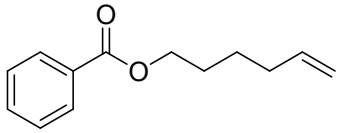
Known compound.[7] **1f** was obtained as a colorless oil in 77% yield (1.03 g); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.1 Hz, 1H), 7.88 (t, *J* = 6.6 Hz, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.52 (p, *J* = 6.7 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 7.0 Hz, 1H), 6.15 (ddt, *J* = 16.7, 10.7, 6.4 Hz, 1H), 5.18 – 5.09 (m, 2H), 3.88 (d, *J* = 6.3 Hz, 2H).

1g: 1-(allyloxy)-4-methoxybenzene



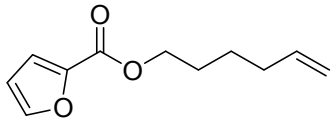
Known compound.[8] **1g** was obtained as a colorless oil in 79% yield (323 mg); hexane/ ethyl acetate = 20/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (q, *J* = 9.3 Hz, 4H), 6.06 (ddd, *J* = 22.5, 10.5, 5.3 Hz, 1H), 5.35 (ddd, *J* = 13.9, 11.7, 1.3 Hz, 2H), 4.50 (d, *J* = 5.3 Hz, 2H), 3.78 (s, 3H).

1i: hex-5-en-1-yl benzoate



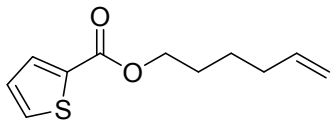
Known compound.[9] **1i** was obtained as a colorless oil in 93% yield (378 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.02 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 2H), 5.82 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 5.01 (ddd, *J* = 13.7, 11.2, 1.3 Hz, 2H), 4.33 (t, *J* = 6.6 Hz, 2H), 2.13 (q, *J* = 7.2 Hz, 2H), 1.84 – 1.72 (m, 2H), 1.61 – 1.51 (m, 2H).

1j: hex-5-en-1-yl furan-2-carboxylate



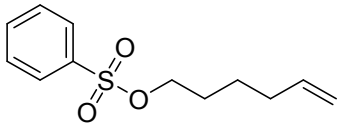
Known compound.[10] **1j** was obtained as a colorless oil in 45% yield (260 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.16 (d, *J* = 3.8 Hz, 1H), 6.51 – 6.47 (m, 1H), 5.79 (ddt, *J* = 16.9, 10.3, 6.7 Hz, 1H), 4.99 (dd, *J* = 22.4, 13.6 Hz, 2H), 4.30 (t, *J* = 6.7 Hz, 2H), 2.10 (q, *J* = 7.1 Hz, 2H), 1.81 – 1.70 (m, 2H), 1.56 – 1.46 (m, 2H).

1k: hex-5-en-1-yl thiophene-2-carboxylate



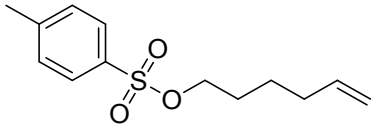
Known compound.[11] **1k** was obtained as a colorless oil in 66% yield (414 mg); hexane/ ethyl acetate = 15/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 3.5 Hz, 1H), 7.54 (d, *J* = 4.8 Hz, 1H), 7.09 (t, *J* = 3.5 Hz, 1H), 5.87 – 5.74 (m, 1H), 5.00 (dd, *J* = 23.0, 13.6 Hz, 2H), 4.30 (t, *J* = 6.6 Hz, 2H), 2.12 (q, *J* = 6.9 Hz, 2H), 1.81 – 1.71 (m, 2H), 1.58 – 1.48 (m, 2H).

1l: hex-5-en-1-yl benzenesulfonate



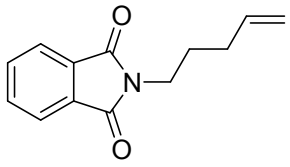
1l was obtained as a colorless oil in 57% yield (277 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.87 (m, 2H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.55 (t, *J* = 7.8 Hz, 2H), 5.70 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 4.98 – 4.89 (m, 2H), 4.04 (t, *J* = 6.4 Hz, 2H), 1.99 (q, *J* = 7.2 Hz, 2H), 1.70 – 1.60 (m, 2H), 1.40 (dt, *J* = 14.7, 7.4 Hz, 2H).

1m: hex-5-en-1-yl 4-methylbenzenesulfonate



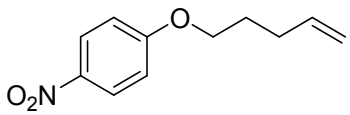
Known compound.[12] **1m** was obtained as a colorless oil in 64% yield (323 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 5.77 – 5.64 (m, 1H), 4.99 – 4.89 (m, 2H), 4.02 (t, *J* = 6.4 Hz, 2H), 2.44 (s, 3H), 1.99 (q, *J* = 7.5 Hz, 2H), 1.69 – 1.59 (m, 2H), 1.40 (dt, *J* = 14.7, 7.4 Hz, 2H).

1n：2-(pent-4-en-1-yl)isoindoline-1,3-dione



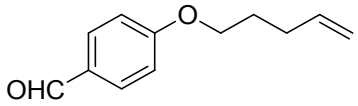
Known compound.[2] **1n** was obtained as a colorless oil in 38% yield (659 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.79 (m, 2H), 7.72 – 7.66 (m, 2H), 5.86 – 5.73 (m, 1H), 5.07 – 4.92 (m, 2H), 3.71 – 3.65 (m, 2H), 2.14 – 2.05 (m, 2H), 1.82 – 1.72 (m, 2H).

1o: 1-nitro-4-(pent-4-en-1-yloxy)benzene



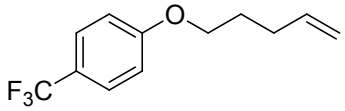
Known compound.[4] **1o** was obtained as a colorless oil in 96% yield (495 mg); hexane/ ethyl acetate = 20/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 9.3 Hz, 2H), 6.93 (d, *J* = 9.3 Hz, 2H), 5.84 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.11 – 4.98 (m, 2H), 4.06 (t, *J* = 6.4 Hz, 2H), 2.25 (q, *J* = 7.3 Hz, 2H), 1.97 – 1.88 (m, 2H).

1p: 4-(pent-4-en-1-yloxy)benzaldehyde



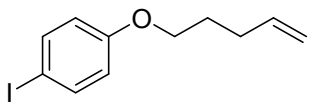
Known compound.[4] **1p** was obtained as a colorless oil in 76% yield (361 mg); hexane/ ethyl acetate = 20/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.81 (d, *J* = 8.5 Hz, 2H), 6.98 (d, *J* = 8.6 Hz, 2H), 5.90 – 5.77 (m, 1H), 5.11 – 4.97 (m, 2H), 4.04 (t, *J* = 6.4 Hz, 2H), 2.24 (q, *J* = 7.3 Hz, 2H), 1.96 – 1.86 (m, 2H).

1q: 1-(pent-4-en-1-yloxy)-4-(trifluoromethyl)benzene



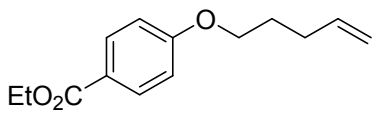
New compound. **1q** was obtained as a colorless oil in 55% yield (185 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.6 Hz, 2H), 6.95 (d, *J* = 8.6 Hz, 2H), 5.86 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.05 (dd, *J* = 21.0, 13.7 Hz, 2H), 4.01 (t, *J* = 6.4 Hz, 2H), 2.25 (q, *J* = 7.2 Hz, 2H), 1.95 – 1.87 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.50 (s, 3F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ161.67, 137.68, 127.00 (q, *J* = 3.7 Hz), 124.65 (q, *J* = 270.6 Hz), 122.86 (q, *J* = 32.7 Hz), 115.56, 114.59, 67.51, 30.16, 28.39; IR (film, cm^{-1}): ν 3080, 2945, 2877, 1617, 1520, 1331, 1259, 1162, 1110, 1068, 1009, 916, 836, 639; MS (EI) m/z (relative intensity) 230 (66) [M^+], 162 (100); HRMS (EI) calcd. For $C_{12}H_{13}OF_3$: 230.0918, Found: 230.0919.

1r: 1-iodo-4-(pent-4-en-1-yloxy)benzene



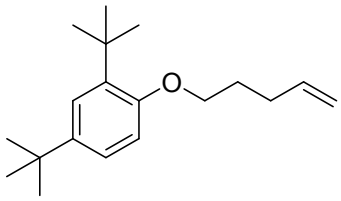
Known compound.[13] **1r** was obtained as a yellow oil in 91% yield (657 mg); hexane/ ethyl acetate = 50/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.9 Hz, 2H), 6.67 (d, *J* = 8.9 Hz, 2H), 5.84 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.10 – 4.98 (m, 2H), 3.93 (t, *J* = 6.4 Hz, 2H), 2.23 (q, *J* = 7.0 Hz, 2H), 1.92 – 1.82 (m, 2H).

1s: ethyl 4-(pent-4-en-1-yloxy)benzoate



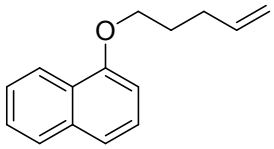
Known compound.[4] **1s** was obtained as a light yellow oil in 94% yield (551 mg); hexane/ ethyl acetate = 15/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.9 Hz, 2H), 6.90 (d, *J* = 8.9 Hz, 2H), 5.85 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.10 – 4.97 (m, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 4.01 (t, *J* = 6.4 Hz, 2H), 2.24 (q, *J* = 7.1 Hz, 2H), 1.94 – 1.85 (m, 2H), 1.37 (t, *J* = 7.1 Hz, 3H).

1t: 2,4-di-*tert*-butyl-1-(pent-4-en-1-yloxy)benzene



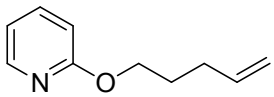
New compound. **1t** was obtained as a colorless oil in 98% yield (637 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (s, 1H), 7.19 (d, *J* = 7.7 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 5.90 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.06 (dd, *J* = 26.9, 13.7 Hz, 2H), 4.00 (t, *J* = 6.3 Hz, 2H), 2.32 (q, *J* = 7.1 Hz, 2H), 1.97 (dd, *J* = 14.0, 6.8 Hz, 2H), 1.43 (s, 9H), 1.33 (s, 9H); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 155.64, 142.37, 138.10, 137.22, 124.02, 123.37, 115.28, 111.15, 66.98, 35.20, 34.38, 31.76, 30.73, 30.04, 28.95; IR (film, cm^{-1}): ν 3078, 2955, 2858, 1642, 1604, 1499, 1470, 1236, 1201, 1094, 1020, 913, 809; MS (EI) m/z (relative intensity) 274 (42) [M^+], 259 (100); HRMS (EI) calcd. For $C_{19}H_{30}O$: 274.2297, Found: 274.2296.

1u: 1-(pent-4-en-1-yloxy)naphthalene



Known compound.[14] **1u** was obtained as a colorless oil in 82% yield (437 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.81 (d, *J* = 8.5 Hz, 2H), 6.98 (d, *J* = 8.6 Hz, 2H), 5.90 – 5.77 (m, 1H), 5.11 – 4.97 (m, 2H), 4.04 (t, *J* = 6.4 Hz, 2H), 2.24 (q, *J* = 7.3 Hz, 2H), 1.96 – 1.86 (m, 2H); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 154.90, 138.02, 134.63, 127.56, 126.46, 125.85, 125.20, 122.19, 120.16, 115.36, 104.68, 67.37, 30.50, 28.64; MS (EI) m/z (relative intensity) 212 (52) [M^+], 144 (100); HRMS (EI) calcd. For $C_{15}H_{16}O$: 212.1201, Found: 212.1205.

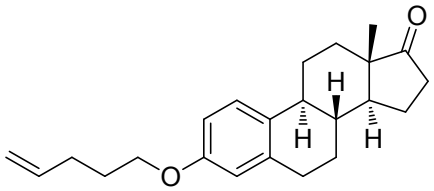
1v: 2-(pent-4-en-1-yloxy)pyridine



Known compound.[15] **1v** was obtained as a light yellow oil in 12% yield (50 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 5.0 Hz, 1H), 7.55 (t, *J* = 8.5 Hz, 1H), 6.86 – 6.81 (m, 1H), 6.72 (d, *J* = 8.4 Hz, 1H), 5.86 (ddt, *J* = 16.9, 10.3, 6.6 Hz, 1H), 5.02 (dd, *J* = 29.2, 13.7 Hz, 2H), 4.29 (t, *J* = 6.6 Hz, 2H), 2.22 (q, *J* = 7.2 Hz, 2H), 1.92 – 1.83 (m, 2H); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 164.12, 147.03, 138.60, 138.13, 116.64, 115.11, 111.19, 65.35, 30.35, 28.46; MS (EI) m/z (relative intensity) 163 (31) [M^+], 95 (100); HRMS (EI) calcd. For $C_{10}H_3NO$: 163.0997, Found:

163.0994.

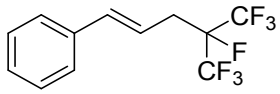
1x: (*8R,9S,13S,14S*)-13-methyl-3-(pent-4-en-1-yloxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one



Known compound.[16] **1x** was obtained as a white solid in 55% yield (467 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.6 Hz, 1H), 6.72 (dd, *J* = 8.6, 2.4 Hz, 1H), 6.66 (s, 1H), 5.86 (ddt, *J* = 16.9, 10.1, 6.6 Hz, 1H), 5.04 (dd, *J* = 25.7, 13.4 Hz, 2H), 3.95 (t, *J* = 6.4 Hz, 2H), 2.90 (dd, *J* = 10.9, 4.6 Hz, 2H), 2.51 (dd, *J* = 18.8, 8.6 Hz, 1H), 2.40 (d, *J* = 9.9 Hz, 1H), 2.24 (dd, *J* = 14.0, 6.8 Hz, 3H), 2.16 (dd, *J* = 18.4, 9.3 Hz, 1H), 2.11 – 1.94 (m, 3H), 1.93 – 1.83 (m, 2H), 1.67 – 1.40 (m, 6H), 0.92 (s, 3H).

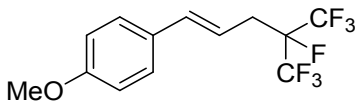
7 Spectral data for products

2a (*E*)-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene



2a was obtained as a colorless liquid in 83% yield (48 mg); pentane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.27 (m, 5H), 6.61 (d, *J* = 15.8 Hz, 1H), 6.18 – 6.07 (m, 1H), 3.03 (dd, *J* = 19.9, 7.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.02 (d, *J* = 7.1 Hz, 6F), -181.72 – -182.22 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 136.76, 136.38, 128.82, 128.30, 126.62, 121.08 (qd, *J* = 288.4, 27.7 Hz), 117.20 (d, *J* = 6.0 Hz), 93.14 – 89.86 (m), 32.98 (d, *J* = 20.9 Hz); IR (film, cm^{-1}): ν 3030, 1288, 1225, 1148, 967, 746, 720; MS (EI) m/z (relative intensity) 286 (27) [M$^+$], 117 (100); HRMS (EI) calcd. For $C_{12}H_9F_7$: 286.0592, Found: 286.0587.

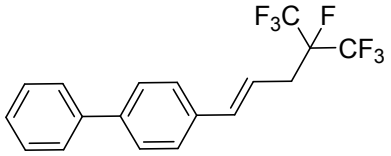
2b (*E*)-1-methoxy-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene



2b was obtained as a light yellow liquid in 67% yield (42 mg); pentane/ dichloromethane = 13/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.5 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.53 (d, *J* = 15.7 Hz, 1H), 6.02 – 5.91 (m, 1H), 3.82 (s, 1H), 2.99 (dd, *J* = 19.9, 7.4 Hz, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.99 (d, *J* = 7.1 Hz, 6F), -181.94 – -182.13 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 159.79, 136.15, 129.22, 127.83, 121.10 (qd, *J* = 285.4, 25.1 Hz), 114.85 (d, *J* = 5.9 Hz), 114.23, 91.53 (ddt, *J* = 203.3, 62.6, 31.3 Hz), 55.46, 33.01 (d, *J* = 21.0 Hz); IR (film, cm^{-1}): ν 2960, 2840, 1609,

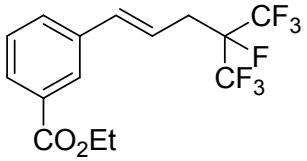
1513, 1294, 1224, 1175, 1146, 1036, 968, 805; MS (EI) m/z (relative intensity) 316 (37) [M^+], 147 (100); HRMS (EI) calcd. For $C_{13}H_{11}OF_7$: 316.0698, Found: 316.0699.

2c (*E*)-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)-1,1'-biphenyl



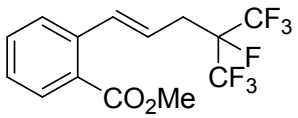
2c was obtained as a white solid in 58% yield (42 mg); hexane. mp: 92-94℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (t, *J* = 8.3 Hz, 4H), 7.46 (dd, *J* = 7.4, 5.1 Hz, 4H), 7.37 (t, *J* = 7.3 Hz, 1H), 6.65 (d, *J* = 15.8 Hz, 1H), 6.23 – 6.12 (m, 1H), 3.06 (dd, *J* = 19.9, 7.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.96 (d, *J* = 7.1 Hz, 6F), -181.92 – -182.15 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 141.15, 140.71, 136.31, 135.36, 128.98, 127.61, 127.52, 127.09 (d, *J* = 7.1 Hz), 121.09 (qd, *J* = 287.1, 27.8 Hz), 117.23 (d, *J* = 5.9 Hz), 91.52 (ddt, *J* = 125.7, 62.7, 31.3 Hz), 33.03 (d, *J* = 20.9 Hz); IR (film, cm^{-1}): ν 3031, 2929, 1478, 1225, 1163, 971, 759, 741, 698; MS (EI) m/z (relative intensity) 362 (43) [M^+], 178 (100); HRMS (EI) calcd. For $C_{18}H_{13}F_7$: 362.0905, Found: 362.0898.

2d (*E*)-ethyl 3-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzoate



2d was obtained as a light yellow liquid in 74% yield (53 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.95 (d, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 6.64 (d, *J* = 15.8 Hz, 1H), 6.24 – 6.14 (m, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 3.03 (dd, *J* = 19.9, 7.4 Hz, 2H), 1.41 (t, *J* = 7.1 Hz, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.03 (d, *J* = 7.1 Hz, 6F), -182.10 – -182.33 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 166.50, 136.60, 135.86, 131.18, 130.67 , 129.25, 128.85, 127.74, 121.04 (qd, *J* = 286.2, 27.8 Hz), 118.55 (d, *J* = 6.0 Hz), 93.08 – 89.80 (m), 61.27, 32.90 (d, *J* = 20.9 Hz), 14.45; IR (film, cm^{-1}): ν 2985, 1721, 1445, 1368, 1285, 1224, 1148, 1129, 1025, 968, 750, 722; MS (EI) m/z (relative intensity) 358 (51) [M^+], 313 (100); HRMS (EI) calcd. For $C_{15}H_{13}O_2F_7$: 358.0804, Found: 358.0799.

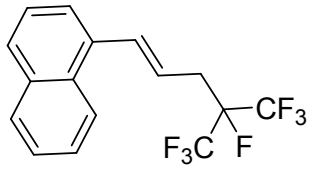
2e (*E*)-methyl 2-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzoate



2e was obtained as a yellow liquid in 59% yield (41 mg); petroleum ether/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 4.0 Hz, 2H), 7.43 – 7.37 (m, 1H), 7.37 – 7.32 (m, 1H), 5.98 (dt, *J* = 15.0, 7.4 Hz, 1H), 3.90 (d, *J* = 3.5 Hz, 3H), 3.07 (dd, *J* = 20.2, 7.3 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.98 (d, *J* = 7.1 Hz, 6F), -182.05 – -182.25 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 167.67, 138.47, 135.94, 132.49, 130.70, 128.49, 127.90 (d, *J* = 2.3 Hz), 121.07 (dd, *J* = 286.2, 27.4 Hz), 119.79 (d, *J* = 5.6 Hz), 91.54 (d, *J* = 203.5 Hz), 52.22,

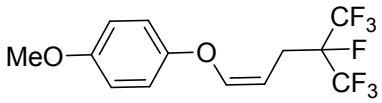
33.02 (d, J = 20.9 Hz); IR (film, cm^{-1}): ν 2955, 1725, 1599, 1571, 1483, 1435, 1352, 1294, 1224, 1149, 1084, 968, 749, 722; MS (EI) m/z (relative intensity) 344 (21) [M^+], 161 (100); HRMS (EI) calcd. For $C_{14}H_{11}O_2F_7$: 344.0647, Found: 344.0650.

2f (*E*)-1-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)naphthalene



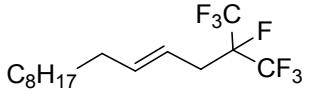
2f was obtained as a colorless liquid in 45% yield (30 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.1 Hz, 1H), 7.85 (dd, J = 20.1, 8.5 Hz, 2H), 7.54 (dd, J = 14.8, 7.5 Hz, 3H), 7.47 (t, J = 7.7 Hz, 1H), 7.36 (d, J = 15.4 Hz, 1H), 6.15 (dt, J = 14.8, 7.4 Hz, 1H), 3.16 (dd, J = 19.8, 7.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.86 (d, J = 7.1 Hz, 6F), -181.70 – -181.93 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 134.50, 134.28, 133.72, 131.18, 128.69 (d, J = 8.7 Hz), 126.44, 126.08, 125.74, 124.45, 123.75, 121.14 (qd, J = 286.7, 27.6 Hz), 120.53 (d, J = 5.9 Hz), 93.22 – 89.95 (m), 33.27 (d, J = 20.9 Hz); IR (film, cm^{-1}): ν 3062, 1509, 1303, 1224, 1147, 969, 795, 776, 721; MS (EI) m/z (relative intensity) 336 (30) [M^+], 167 (100); HRMS (EI) calcd. For $C_{14}H_{11}F_7$: 336.0749, Found: 336.0755.

2g (*Z*)-1-methoxy-4-((4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)oxy)benzene



2g was obtained as a colorless liquid in 94% yield (60 mg); pentane/ dichloromethane = 20/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (d, J = 9.0 Hz, 2H), 6.86 (d, J = 9.1 Hz, 2H), 6.51 (d, J = 6.1 Hz, 1H), 4.79 – 4.71 (m, 1H), 3.79 (s, 3H), 3.08 (dd, J = 19.6, 7.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.32 (d, J = 7.0 Hz, 6F), -182.27 – -182.47 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 155.95, 151.17, 146.11, 121.15 (dd, J = 287.7, 27.3 Hz), 118.05, 114.91, 98.13 (d, J = 7.0 Hz), 92.80 – 90.16 (m), 55.84, 34.28; IR (film, cm^{-1}): ν 2957, 2839, 1672, 1507, 1293, 1224, 1181, 1055, 827, 751, 721; MS (EI) m/z (relative intensity) 332 (100) [M^+]; HRMS (EI) calcd. For $C_{13}H_{11}O_2F_7$: 332.0647, Found: 332.0649.

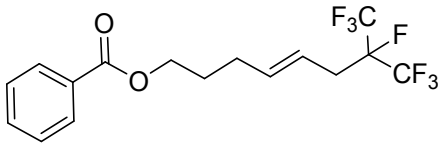
2h (*E*)-1,1,1,2-tetrafluoro-2-(trifluoromethyl)tetradec-4-ene



2h was obtained as a colorless liquid in 86% yield (58 mg); pentane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 – 5.64 (m, 1H), 5.42 – 5.32 (m, 1H), 2.79 (dd, J = 19.9, 7.2 Hz, 2H), 2.05 (dd, J = 14.0, 7.0 Hz, 2H), 1.27 (s, 14H), 0.89 (dd, J = 6.9, 6.1 Hz, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.09 (d, J = 7.0 Hz, 6F), -182.11 (dqd, J = 20.6, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 138.73, 121.16 (qd, J = 288.2, 27.7 Hz), 117.40 (d, J = 6.0 Hz), 91.54 (ddt, J = 202.7, 62.5, 31.3 Hz), 32.78

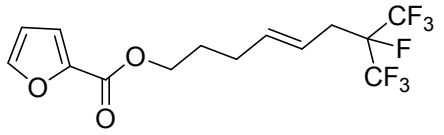
(d, J = 21.0 Hz), 32.62, 32.09 ,29.74, 29.62, 29.49, 29.19, 29.14, 22.86, 14.21; IR (film, cm^{-1}): ν 2928, 2857, 1467, 1287, 1224, 1152, 971, 719; MS (EI) m/z (relative intensity) 336 (5) [M^+], 57 (100); HRMS (EI) calcd. For $C_{15}H_{23}F_7$: 336.1688, Found: 336.1691.

2i (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzoate



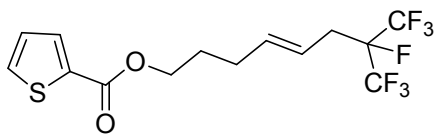
2i was obtained as a light yellow liquid in 79% yield (59 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.02 (m, 2H), 7.59 – 7.53 (m, 1H), 7.47 – 7.41 (m, 2H), 5.73 (dt, J = 13.8, 6.8 Hz, 1H), 5.51 – 5.41 (m, 1H), 4.32 (t, J = 6.4 Hz, 2H), 2.80 (dd, J = 20.0, 7.2 Hz, 2H), 2.28 – 2.19 (m, 2H), 1.90 – 1.83 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.03 (d, J = 7.1 Hz, 6F), -181.78 – -182.32 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 166.70, 137.01, 133.04, 130.50, 130.46, 129.68, 128.50, 121.06 (qd, J = 287.8, 27.6 Hz), 118.68 (d, J = 5.9 Hz), 92.73 – 90.09 (m), 64.25, 32.61 (d, J = 21.0 Hz), 29.10, 28.18; IR (film, cm^{-1}): ν 2956, 1721, 1603, 1452, 1272, 1223, 1151, 1114, 1027, 972, 719; MS (ESI) m/z (relative intensity) 372.9 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{16}H_{16}O_2F_7$ $[M+H]^+$: 373.1033, Found: 373.1032.

2j (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl furan-2-carboxylate



2j was obtained as a colorless liquid in 63% yield (46 mg); hexane/ ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.17 (d, J = 3.1 Hz, 1H), 6.50 (s, 1H), 5.76 – 5.65 (m, 1H), 5.50 – 5.39 (m, 1H), 4.29 (t, J = 6.6 Hz, 2H), 2.79 (dd, J = 19.9, 7.1 Hz, 2H), 2.20 (dd, J = 14.4, 7.1 Hz, 2H), 1.88 – 1.79 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.05 (d, J = 7.0 Hz, 6F), -182.09 – -182.32 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 158.85, 146.40, 144.90, 121.04 (qd, J = 287.6, 27.7 Hz), 118.73 (d, J = 6.0 Hz), 117.96, 111.94, 91.39 (ddt, J = 203.1, 62.7, 31.3 Hz), 64.20, 32.60 (d, J = 21.0 Hz), 28.94, 28.11; IR (film, cm^{-1}): ν 2956, 1733, 1581, 1475, 1297, 1224, 1180, 1121, 1013, 973, 763; MS (ESI) m/z (relative intensity) 362.9 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{14}H_{13}O_3F_7$ $[M+H]^+$: 363.0826, Found: 363.0820.

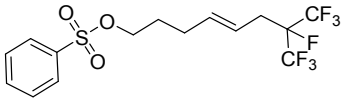
2k (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl thiophene-2-carboxylate



2k was obtained as a colorless liquid in 80% yield (61 mg); hexane/ ethyl acetate = 40/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 3.6 Hz, 1H), 7.55 (d, J = 4.9 Hz, 1H), 7.10 (t, J = 4.3 Hz, 1H),

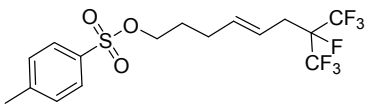
5.78 – 5.68 (m, 1H), 5.45 (dt, *J* = 14.8, 7.3 Hz, 1H), 4.29 (t, *J* = 6.5 Hz, 2H), 2.80 (dd, *J* = 19.9, 7.2 Hz, 2H), 2.22 (dd, *J* = 14.3, 7.1 Hz, 2H), 1.89 – 1.79 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.99 (d, *J* = 7.0 Hz, 6F), -182.04 – -182.27 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 162.34, 136.94, 134.02, 133.49, 132.42, 127.87, 121.04 (qd, *J* = 286.2, 27.9 Hz), 118.72 (d, *J* = 6.1 Hz), 91.40 (ddt, *J* = 203.0, 62.7, 31.4 Hz), 64.38, 32.60 (d, *J* = 20.9 Hz), 29.02, 28.14; IR (film, cm^{-1}): ν 2956, 1712, 1527, 1419, 1261, 1223, 1151, 1097, 973, 751, 719; MS (ESI) m/z (relative intensity) 378.9 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{14}H_{14}O_2F_7S$ $[M+H]^+$: 379.0597, Found: 379.0591.

2l (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzenesulfonate



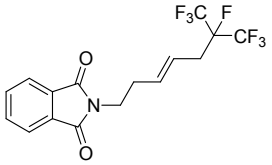
2l was obtained as a colorless liquid in 67% yield (55 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.3 Hz, 2H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 2H), 5.62 – 5.50 (m, 1H), 5.38 – 5.27 (m, 1H), 4.02 (t, *J* = 6.3 Hz, 2H), 2.73 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.78 – 1.68 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.08 (d, *J* = 7.1 Hz, 6F), -181.80 – -182.43 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 136.27, 136.14, 133.87, 129.37, 127.95, 120.98 (qd, *J* = 288.8, 27.8 Hz), 119.22 (d, *J* = 6.0 Hz), 91.31 (ddt, *J* = 203.0, 62.6, 31.3 Hz), 69.85, 32.48 (d, *J* = 20.9 Hz), 28.23 (d, *J* = 5.3 Hz); IR (film, cm^{-1}): ν 2955, 1448, 1363, 1288, 1224, 1188, 970, 754, 590; MS (ESI) m/z (relative intensity) 430.9 (100) $[M+Na]^+$; HRMS (ESI) calcd. For $C_{15}H_{16}O_3F_7S$ $[M+H]^+$: 409.0703, Found: 409.0700.

2m (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl 4-methylbenzenesulfonate



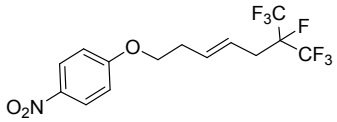
2m was obtained as a colorless liquid in 68% yield (57 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 5.60 – 5.51 (m, 1H), 5.32 (dt, *J* = 14.6, 7.2 Hz, 1H), 3.99 (t, *J* = 6.3 Hz, 2H), 2.73 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.43 (s, 3H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.76 – 1.68 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.08 (d, *J* = 7.1 Hz, 6F), -182.00 – -182.46 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 144.94, 136.21, 133.27, 129.96, 128.00, 120.99 (qd, *J* = 288.3, 27.7 Hz), 119.16 (d, *J* = 5.6 Hz), 91.33 (ddt, *J* = 203.4, 62.8, 31.4 Hz), 69.61, 32.49 (d, *J* = 20.9 Hz), 28.23 (d, *J* = 7.0 Hz), 21.65; IR (film, cm^{-1}): ν 2956, 1598, 1364, 1224, 1177, 971, 664, 555; MS (ESI) m/z (relative intensity) 422.9 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{16}H_{18}O_3F_7S$ $[M+H]^+$: 423.0859, Found: 423.0854.

2n (*E*)-2-(6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)isoindoline-1,3-dione



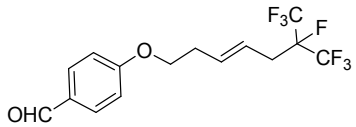
2n was obtained as a white solid in 71% yield (54 mg); hexane/ ethyl acetate = 12/1. mp: 56-58℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.77 (m, 2H), 7.72 – 7.66 (m, 2H), 5.71 – 5.61 (m, 1H), 5.41 (dt, *J* = 14.6, 7.1 Hz, 1H), 3.74 (t, *J* = 6.9 Hz, 2H), 2.72 (dd, *J* = 20.2, 7.1 Hz, 2H), 2.45 (dd, *J* = 13.6, 6.8 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.22 (d, *J* = 7.1 Hz, 6F), -182.44 – -182.64 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 168.40, 134.14 (d, *J* = 3.0 Hz), 134.04, 132.17, 123.39 (d, *J* = 6.3 Hz), 123.30, 120.89 (qd, *J* = 287.5, 28.2 Hz), 120.86 (d, *J* = 5.6 Hz), 91.22 (ddt, *J* = 125.6, 63.0, 31.6 Hz), 37.15, 32.46 (d, *J* = 20.9 Hz), 31.80; IR (film, cm^{-1}): ν 2942, 2360, 1716, 1396, 1288, 1223, 1156, 1064, 719; MS (ESI) m/z (relative intensity) 384.0 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{16}H_{13}O_2NF_7$ $[M+H]^+$: 384.0829, Found: 384.0822.

2o (*E*)-1-nitro-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



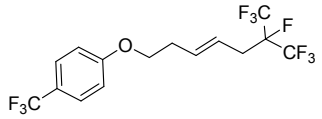
2o was obtained as a colorless liquid in 67% yield (50 mg); hexane/ ethyl acetate = 25/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 8.3, 0.9 Hz, 2H), 6.93 (d, *J* = 9.2 Hz, 2H), 5.79 (dt, *J* = 14.0, 6.8 Hz, 1H), 5.58 (dt, *J* = 14.7, 7.2 Hz, 1H), 4.08 (t, *J* = 6.4 Hz, 2H), 2.84 (dd, *J* = 19.9, 7.2 Hz, 2H), 2.59 (q, *J* = 6.6 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.04 (d, *J* = 6.4 Hz, 6F), -182.28 (dqd, *J* = 20.8, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 163.96, 141.73, 133.21, 126.04, 121.01 (d, *J* = 6.0 Hz), 121.00 (qd, *J* = 288.5, 27.4 Hz), 114.54, 91.37 (ddt, *J* = 203.2, 62.7, 31.4 Hz), 67.75, 32.61 (d, *J* = 20.9 Hz), 32.25; IR (film, cm^{-1}): ν 3088, 2940, 1594, 1499, 1343, 1264, 1223, 1047, 974, 846, 753, 720, 658; MS (EI) m/z (relative intensity) 375 (8) $[M^+]$, 237 (100); HRMS (EI) calcd. For $C_{14}H_{12}NO_3F_7$: 375.0705, Found: 375.0699.

2p (*E*)-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzaldehyde



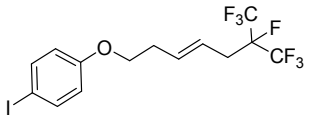
2p was obtained as a colorless liquid in 52% yield (37 mg); hexane/ ethyl acetate = 25/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.82 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 5.84 – 5.74 (m, 1H), 5.63 – 5.51 (m, 1H), 4.07 (t, *J* = 6.4 Hz, 2H), 2.84 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.58 (q, *J* = 6.5 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.03 (d, *J* = 7.0 Hz, 6F), -182.16 – -182.36 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 190.90, 163.96, 133.45, 132.12, 130.18, 121.02 (qd, *J* = 286.6, 27.5 Hz), 120.79 (d, *J* = 6.0 Hz), 114.88, 93.03 – 89.73 (m), 67.31, 32.63 (d, *J* = 21.0 Hz), 32.33; IR (film, cm^{-1}): ν 2940, 2830, 2739, 1696, 1602, 1579, 1510, 1312, 1223, 1027, 974, 833, 719; MS (EI) m/z (relative intensity) 358 (34) $[M^+]$, 237 (100); HRMS (EI) calcd. For $C_{15}H_{13}O_2F_7$: 358.0804, Found: 358.0806.

2q (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-4-(trifluoromethyl) benzene



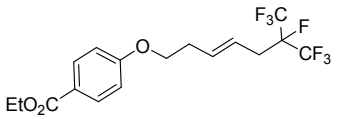
2q was obtained as a colorless liquid in 64% yield (51 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 8.6 Hz, 2H), 5.85 – 5.75 (m, 1H), 5.63 – 5.52 (m, 1H), 4.03 (t, *J* = 6.5 Hz, 2H), 2.85 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.57 (q, *J* = 6.6 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.61 (s, 3F), -76.04 (d, *J* = 7.0 Hz, 6F), -182.25 (dqd, *J* = 20.9, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 161.40, 133.62, 127.05 (q, *J* = 3.7 Hz), 124.61 (q, *J* = 270.6 Hz), 123.17 (q, *J* = 32.7 Hz), 121.07 (qd, *J* = 286.0, 28.2 Hz), 120.70 (d, *J* = 6.0 Hz), 114.62, 93.10 – 89.78 (m), 67.23, 32.68 (d, *J* = 20.9 Hz), 32.40; IR (film, cm^{-1}): ν 2937, 2879, 1616, 1520, 1259, 1225, 1162, 1123, 1069, 973, 837, 719; MS (EI) m/z (relative intensity) 398 (15) [M^{+}], 237 (100); HRMS (EI) calcd. For $C_{15}H_{12}OF_{10}$: 398.0728, Found: 398.0725.

2r (*E*)-1-iodo-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



2r was obtained as a light yellow liquid in 90% yield (82 mg); hexane/ ethyl acetate = 60/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.4 Hz, 2H), 6.66 (d, *J* = 8.4 Hz, 2H), 5.83 – 5.73 (m, 1H), 5.55 (dt, *J* = 14.8, 7.4 Hz, 1H), 3.95 (t, *J* = 6.5 Hz, 2H), 2.84 (dd, *J* = 19.9, 7.2 Hz, 2H), 2.54 (q, *J* = 6.6 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.01 (d, *J* = 7.0 Hz, 6F), -182.21 (dqd, *J* = 20.8, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 158.82, 138.38, 133.77, 121.05 (qd, *J* = 286.1, 27.3 Hz), 120.47 (d, *J* = 6.0 Hz), 117.08, 91.40 (ddt, *J* = 203.2, 62.8, 31.3 Hz), 82.96, 67.12, 32.66 (d, *J* = 20.9 Hz), 32.43; IR (film, cm^{-1}): ν 2929, 2876, 1586, 1471, 1283, 1242, 1151, 1033, 972, 820, 719; MS (EI) m/z (relative intensity) 456 (15) [M^{+}], 220 (100); HRMS (EI) calcd. For $C_{14}H_{12}OF_7I$: 455.9821, Found: 455.9824.

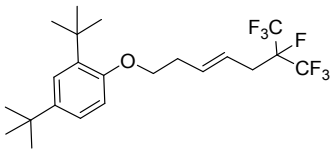
2s (*E*)-ethyl 4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzoate



2s was obtained as a colorless liquid in 68% yield (55 mg); hexane/ ethyl acetate = 35/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 5.79 (dt, *J* = 14.0, 6.8 Hz, 1H), 5.56 (dt, *J* = 14.8, 7.3 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 4.03 (t, *J* = 6.5 Hz, 2H), 2.83 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.56 (q, *J* = 6.6 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.04 (d, *J* = 7.0 Hz, 6F), -182.24 (dqd, *J* = 20.7, 14.1, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 166.49, 162.61, 133.65, 131.68, 123.23, 121.04 (qd, *J* = 287.1, 27.5 Hz), 120.60 (d, *J* = 6.0 Hz), 114.15, 91.40 (ddt, *J* = 203.3, 62.8, 31.5 Hz), 67.12, 60.75, 32.65 (d, *J* = 20.9 Hz), 32.40, 14.47; IR (film, cm^{-1}): ν 2984, 2939, 1713, 1607, 1511, 1279, 1253, 1224, 1168, 1104, 1029, 974,

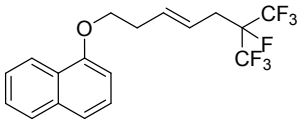
848, 720, 699; MS (EI) m/z (relative intensity) 402 (20) [M^+], 121 (100); HRMS (EI) calcd. For $C_{17}H_{17}O_3F_7$: 402.1066, Found: 402.1061.

2t (*E*)-2,4-di-tert-butyl-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



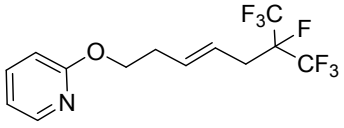
2t was obtained as a colorless liquid in 53% yield (45 mg); hexane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 2.4 Hz, 1H), 7.20 (dd, *J* = 8.4, 2.5 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 5.92 – 5.83 (m, 1H), 5.66 – 5.56 (m, 1H), 4.05 (t, *J* = 6.3 Hz, 2H), 2.87 (dd, *J* = 19.9, 7.2 Hz, 2H), 2.64 (q, *J* = 6.4 Hz, 2H), 1.43 (s, 9H), 1.34 (s, 9H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.03 (d, *J* = 7.0 Hz, 6F), -182.13 – -182.36 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 155.42, 142.78, 137.38, 134.71, 124.13, 123.43, 121.13 (qd, *J* = 289.7, 27.2 Hz), 120.22 (d, *J* = 5.8 Hz), 111.26, 93.05 – 89.78 (m), 67.03, 35.21, 34.42, 33.02, 32.70 (d, *J* = 20.9 Hz), 31.75, 30.00; IR (film, cm^{-1}): ν 2959, 2909, 2869, 1500, 1472, 1361, 1286, 1225, 1162, 1093, 1035, 972, 809, 720; MS (EI) m/z (relative intensity) 442 (16) [M^+], 191 (100); HRMS (EI) calcd. For $C_{22}H_{29}OF_7$: 442.2107, Found: 442.2101.

2u (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)naphthalene



2u was obtained as a yellow liquid in 81% yield (62 mg); hexane/ ethyl acetate = 100/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 – 8.27 (m, 1H), 7.87 – 7.80 (m, 1H), 7.54 – 7.45 (m, 3H), 7.40 (t, *J* = 7.9 Hz, 1H), 6.82 (d, *J* = 7.5 Hz, 1H), 5.97 – 5.87 (m, 1H), 5.71 – 5.61 (m, 1H), 4.19 (t, *J* = 6.4 Hz, 2H), 2.89 (dd, *J* = 20.0, 7.2 Hz, 2H), 2.72 (q, *J* = 6.4 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.97 (d, *J* = 7.1 Hz, 6F), -182.22 (dqd, *J* = 20.7, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 154.70, 134.68, 134.27, 127.58, 126.56, 125.95, 125.85, 125.29, 122.13, 121.09 (qd, *J* = 287.1, 28.0 Hz), 120.49, 120.31 (d, *J* = 6.0 Hz), 104.78, 91.43 (ddt, *J* = 202.4, 62.4, 31.3 Hz), 67.23, 32.70 (t, *J* = 10.4 Hz); IR (film, cm^{-1}): ν 3054, 2933, 2875, 1581, 1509, 1460, 1405, 1270, 1224, 1152, 1101, 972, 791, 771, 719; MS (EI) m/z (relative intensity) 380 (11) [M^+], 144 (100); HRMS (EI) calcd. For $C_{18}H_{15}OF_7$: 380.1011, Found: 380.1004.

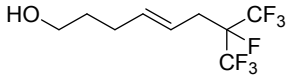
2v (*E*)-2-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)pyridine



2v was obtained as a yellow liquid in 57% yield (36 mg); hexane/ ethyl acetate = 40/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 5.0 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 6.87 – 6.82 (m, 1H), 6.71 (d, *J* = 8.4 Hz, 1H), 5.85 – 5.73 (m, 1H), 5.59 – 5.48 (m, 1H), 4.33 (t, *J* = 6.6 Hz, 2H), 2.82 (dd, *J*

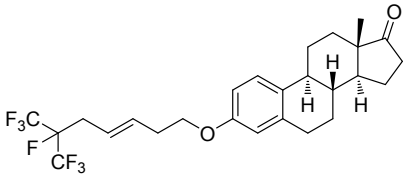
= 19.9, 7.2 Hz, 2H), 2.53 (q, *J* = 6.7 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.05 (d, *J* = 7.0 Hz, 6F), -182.08 – -182.31 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 163.87, 146.98, 138.68, 134.34, 121.06 (qd, *J* = 287.0, 27.8 Hz), 120.00 (d, *J* = 6.1 Hz), 116.83, 111.26, 93.06 – 89.79 (m), 64.72, 32.72 (d, *J* = 20.9 Hz), 32.42; IR (film, cm^{-1}): ν 2961, 2920, 2851, 1597, 1572, 1469, 1434, 1288, 1224, 1127, 1090, 1044, 804, 780, 719; MS (EI) m/z (relative intensity) 331 (4) [M$^+$], 162 (100); HRMS (EI) calcd. For $C_{13}H_{12}NOF_7$: 331.0807, Found: 331.0812.

2w (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-ol



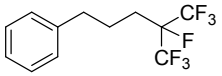
2w was obtained as a colorless liquid in 62% yield (50 mg); dichloromethane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.75 – 5.65 (m, 1H), 5.47 – 5.37 (m, 1H), 3.64 (t, *J* = 6.5 Hz, 2H), 2.80 (dd, *J* = 19.9, 7.2 Hz, 2H), 2.15 (q, *J* = 7.1 Hz, 2H), 1.69 – 1.61 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.03 (d, *J* = 7.0 Hz, 6F), -182.04 – -182.27 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 137.68, 121.07 (qd, *J* = 287.9, 27.8 Hz), 118.18 (d, *J* = 6.0 Hz), 93.03 – 89.80 (m), 62.28, 32.65 (d, *J* = 21.0 Hz), 31.92, 28.86; IR (film, cm^{-1}): ν 3347, 2938, 2867, 1434, 1290, 1224, 1162, 1051, 973, 763; GC-MS (EI) m/z (relative intensity) 268 [M$^+$], 81 (100); GC-HRMS (EI) calcd. For $C_9H_{11}OF_7$: 268.0698, Found: 268.0703.

2x (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-(((*E*)-6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one



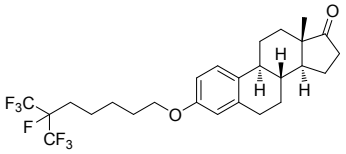
2x was obtained as a white solid in 59% yield (60 mg); hexane/ ethyl acetate = 20/1. mp: 70-72℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.6 Hz, 1H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.64 (s, 1H), 5.86 – 5.75 (m, 1H), 5.62 – 5.50 (m, 1H), 3.97 (t, *J* = 6.4 Hz, 2H), 2.96 – 2.78 (m, 4H), 2.53 (q, *J* = 6.1 Hz, 2H), 2.48 (d, *J* = 8.5 Hz, 1H), 2.45 – 2.36 (m, 1H), 2.30 – 1.93 (m, 5H), 1.70 – 1.38 (m, 6H), 0.92 (s, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.99 (d, *J* = 7.0 Hz, 6F), -182.19 (dqd, *J* = 20.7, 14.0, 7.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 156.94, 137.89, 134.16, 132.34, 126.45, 121.03 (dd, *J* = 287.4, 27.4 Hz), 120.09 (d, *J* = 6.0 Hz), 114.76, 112.29, 93.00 – 89.78 (m), 66.99, 50.56, 48.12, 44.12, 38.51, 35.97, 32.61, 31.73, 29.76, 26.67, 26.05, 21.70, 13.96; IR (film, cm^{-1}): ν 2929, 2867, 1740, 1608, 1573, 1450, 1282, 1223, 1152, 1054, 973, 719; MS (ESI) m/z (relative intensity) 506.9 (100) [M+H]$^+$; HRMS (ESI) calcd. For $C_{26}H_{30}O_2F_7$ [M+H]$^+$: 507.2129, Found: 507.2119.

3a (4,5,5,5-tetrafluoro-4-(trifluoromethyl)pentyl)benzene



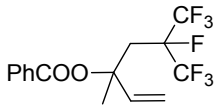
3a was obtained as a colorless liquid in 60% yield (69 mg); pentane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.28 – 7.17 (m, 3H), 2.70 (t, *J* = 7.5 Hz, 2H), 2.18 – 2.04 (m, 2H), 1.94 (dt, *J* = 15.6, 7.7 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.44 (d, *J* = 6.7 Hz, 6F), -183.61 – -183.84 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 140.56, 128.75, 128.46, 126.51, 121.26 (qd, *J* = 286.7, 28.2 Hz), 92.00 (ddt, *J* = 202.3, 63.6, 31.7 Hz), 35.72, 28.63 (d, *J* = 20.6 Hz), 23.21; IR (film, cm^{-1}): ν 3031, 2928, 1310, 1222, 11151, 1092, 946, 751, 719, 698; MS (EI) m/z (relative intensity) 288 (11) $[M]^+$, 91 (100); HRMS (EI) calcd. For $C_{12}H_{11}F_7$: 288.0749, Found: 288.0755.

3x (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)heptyl)oxy)-7,8,9,11,12,13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one



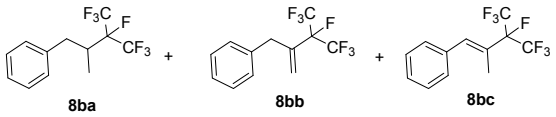
3x was obtained as a white solid in 58% yield (119 mg); hexane/ ethyl acetate = 20/1. mp: 107.1-108.6℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.6 Hz, 1H), 6.72 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.66 (d, *J* = 2.3 Hz, 1H), 3.96 (t, *J* = 6.2 Hz, 2H), 2.95 – 2.87 (m, 2H), 2.51 (dd, *J* = 18.7, 8.6 Hz, 1H), 2.40 (d, *J* = 10.0 Hz, 1H), 2.25 (t, *J* = 10.2 Hz, 1H), 2.20 – 1.93 (m, 6H), 1.87 – 1.76 (m, 2H), 1.72 – 1.38 (m, 10H), 0.92 (s, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ-76.42 (d, *J* = 6.7 Hz, 6F), -183.72 – -183.97 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 220.91, 157.06, 137.84, 132.14, 126.41, 121.21 (qd, *J* = 287.0, 27.4 Hz), 114.59, 112.14, 91.88 (ddt, *J* = 202.4, 63.5, 31.8 Hz), 67.40, 50.48, 48.07, 44.07, 38.47, 35.92, 31.68, 29.73, 29.12, 28.94, 26.63, 26.39, 26.01, 21.64, 21.30 (d, *J* = 4.4 Hz), 13.89; IR (film, cm^{-1}): ν 2933, 2876, 1732, 1612, 1573, 1499, 1474, 1281, 1217, 1157, 1057, 874, 812, 720; MS (ESI) m/z (relative intensity) 509.2 (100) $[M+H]^+$; HRMS (ESI) calcd. For $C_{26}H_{32}O_2F_7$ $[M+H]^+$: 509.2272, Found: 509.2285.

7a 5,6,6,6-tetrafluoro-3-methyl-5-(trifluoromethyl)hex-1-en-3-yl benzoate



7a was obtained as a light yellow liquid in 57% yield (41 mg); petroleum ether / ethyl acetate = 30/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 6.19 (ddd, *J* = 17.4, 11.0, 1.9 Hz, 1H), 5.32 (dd, *J* = 41.5, 14.2 Hz, 2H), 2.96 – 2.73 (m, 2H), 1.86 (s, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.54 – -76.69 (m, 3F), -76.77 – -76.94 (m, 3F), -185.11 – -185.33 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 165.37, 140.86, 133.16, 130.94, 129.66, 128.53, 114.40, 80.37, 37.28 (d, *J* = 17.8 Hz), 25.07 (d, *J* = 2.0 Hz); IR (film, cm^{-1}): ν 2994, 1724, 1602, 1452, 1283，1226, 1163, 1109, 1070, 1049, 1026, 985, 711; MS (ESI) m/z (relative intensity) 420.1 (100) $[M+NH_4]^+$; HRMS (EI) calcd. For $C_{15}H_{14}O_2F_7^+$: 359.0877, Found: $[M+H]^+$: 359.0875.

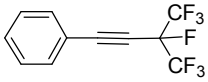
7ba + 7bb + 7bc



8ba **8bb** **8bc**

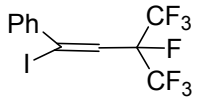
7ba, 7bb and 7bc were obtained as a mixture (46 mg); pentane. Individual yield was determined by integral of ^{19}F NMR (376 MHz, $CDCl_3$). Structures were suggested by NMR and GC-MS. In the ^{1}H NMR (400 MHz, $CDCl_3$) picture, the protons on the aromatic ring were unable to recognize. Protons at δ 2.79 (s, 1H), 2.72 (s, 1H), 2.23 – 2.03 (m, 2H), 1.99 – 1.87 (m, 1H), 1.05 (d, *J* = 6.5 Hz, 3H) and fluorine atoms at δ -76.47 – -76.58 (m, 3F), -77.36 – -77.49 (m, 3F), -182.66 – -182.92 (m, 1F) were associated with **7ba**. Protons at δ 5.13 (d, *J* = 1.4 Hz, 2H), 3.50 (s, 2H), 2.81 – 2.71 (m, 1H), 2.52 (dd, *J* = 13.5, 8.1 Hz, 1H) and fluorine atoms at δ -76.11 (d, *J* = 6.7 Hz, 6F), -185.17 (ttd, *J* = 20.0, 13.4, 6.7 Hz, 1F) were associated with **7bb**. Protons at δ 6.52 (s, 1H), 2.98 (d, *J* = 24.9 Hz, 2H), 2.00 (s, 3H) and fluorine atoms at δ -76.13 (s) were associated with **7bc**. GC-MS (EI) m/z (relative intensity) : 6.933 min, M = 302 (**7ba**), 48.8%; 6.980 min, M = 300 (**7bb**) , 38.1%; 7.058 min, M = 300, 3.3%; 7. 514 min, M = 300 (**7bc**), 9.8%.

9aa (3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)benzene



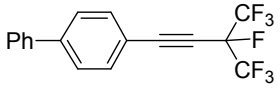
9aa was obtained as a colourless liquid in 31% yield (17 mg); pentane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.1 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.96 (d, *J* = 11.0 Hz, 6F), -165.65 (hept, *J* = 10.9 Hz, 1F); ^{13}C 132.73 (d, *J* = 2.8 Hz), 131.11, 129.50, 128.90 (d, *J* = 2.1 Hz), 128.79, 127.97, 118.97, 93.75; IR (film, cm^{-1}): ν 2923, 2246, 1266，1247, 1177, 1122, 1094, 1062, 984, 756, 725; MS (EI) m/z (relative intensity) 270 (8) [M^+], 45 (100); HRMS (EI) calcd. For $C_{11}H_5F_7$: 270.0279, Found: 270.0281.

9ab: (3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)benzene



9ab: was obtained as a colourless liquid in 14% yield (8 mg); pentane. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (q, *J* = 6.8 Hz, 3H), 7.26 (d, *J* = 7.8 Hz, 2H), 6.44 (d, *J* = 24.1 Hz, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.76 (d, *J* = 7.8 Hz, 6F), -184.49 – -184.69 (m, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 141.88 (d, *J* = 2.8 Hz), 129.07, 128.64, 128.06, 126.62 (d, *J* = 2.9 Hz), 123.45 (d, *J* = 14.0 Hz), 119,71 (qd, *J* = 286.7, 28.1 Hz), 109.61, 94.08– 90.75 (m, 1F); IR (film, cm^{-1}): ν 3061, 1641, 1489，1445, 1301, 1278, 1229, 1199, 1043, 980, 953, 761, 715, 693, 636; MS (EI) m/z (relative intensity) 271 (100) $[M\text{-}I^-]^+$; HRMS (EI) calcd. For $C_{11}H_6F_7I$: 397.9402, Found: 397.9394.

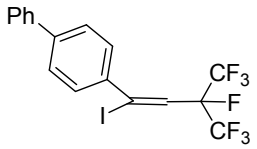
9ba: 4-(3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)-1,1'-biphenyl



9ba was obtained as a white solid in 44% yield (30.5 mg); petroleum ether. mp: 73.7-76.6℃; ^{1}H

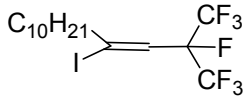
NMR (400 MHz, $CDCl_3$) δ 7.68 – 7.57 (m, 6H), 7.52 – 7.45 (m, 2H), 7.41 (t, *J* = 7.3 Hz, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.90 (d, *J* = 11.0 Hz, 6F), -165.39 (hept, *J* = 11.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 143.96 (d, *J* = 0.6 Hz), 139.84, 133.20 (d, *J* = 2.8 Hz), 129.15, 128.41, 127.43, 127.31, 119.53 (qd, *J* = 286.4, 28.8 Hz), 117.65 (d, *J* = 3.8 Hz), 93.72 (d, *J* = 8.5 Hz); IR (film, cm^{-1}): ν 2242, 1487, 1331, 1268, 1230, 1177, 1116, 1061, 972, 840, 764, 723; MS (EI) m/z (relative intensity) 346 (91) [M^+], 277 (100); HRMS (EI) calcd. For $C_{17}H_9F_7$: 346.0592, Found: 346.0593.

9bb: 4-(3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)-1,1'-biphenyl



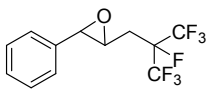
9bb was obtained as a white solid in 23% yield (16 mg); petroleum ether. mp: 78.3-82.6℃; ^1H NMR (400 MHz, $CDCl_3$) δ^1H NMR (400 MHz, cdcl$_3$) δ 7.62 – 7.58 (m, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 2H), 6.46 (d, *J* = 24.0 Hz, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.66 (d, *J* = 7.9 Hz, 6F), -184.27 (ddt, *J* = 23.6, 15.6, 7.8 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 141.92, 140.71 (d, *J* = 2.8 Hz), 140.22, 133.19 (d, *J* = 2.8 Hz), 132.0, 129.15, 128.99, 127.92, 127.44, 127.25 (dd, *J* = 6.9, 4.1 Hz), 126.72, 123.54 (d, *J* = 14.0 Hz), 119.73 (dd, *J* = 287.4, 26.8 Hz), 109.52, 93.79 – 90.84 (m); IR (film, cm^{-1}): ν 1641, 1485, 1448，1402, 1298, 1273, 1231, 1194, 1043, 979, 833, 753, 694; MS (EI) m/z (relative intensity) 347 (100) [$M\text{-}I^-]^+$; HRMS (EI) calcd. For $C_{17}H_{10}F_7I$: 473.9716, Found: 473.9707.

9c: 1,1,1,2-tetrafluoro-4-iodo-2-(trifluoromethyl)tetradec-3-ene



9c was obtained as a colourless liquid in 56% yield (38 mg); pentane. ^1H NMR (400 MHz, $CDCl_3$) δ 6.15 (d, *J* = 24.5 Hz, 1H), 2.68 – 2.59 (m, 2H), 1.56 (dd, *J* = 11.6, 3.7 Hz, 2H), 1.37 – 1.18 (m, 14H), 0.88 (t, *J* = 6.7 Hz, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.19 (d, *J* = 8.1 Hz, 6F), -183.22 (ddq, *J* = 24.3, 16.2, 8.0 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 122.06 (d, *J* = 14.3 Hz), 121.16, 100.14, 41.42 (d, *J* = 8.9 Hz), 32.04, 30.16, 29.68, 29.60, 29.44 (d, *J* = 2.6 Hz), 28.58, 22.83, 14.25; IR (film, cm^{-1}): ν 2927, 2856, 1301, 1228，1181, 1043, 978, 712; MS (EI) m/z (relative intensity) 336 (20) [$M+1\text{-}I^-]^+$, 57 (100); HRMS (EI) calcd. For $C_{15}H_{22}F_7^+$+1: 336.1604, Found: [$M+1\text{-}I^-]^+$, 336.1680.

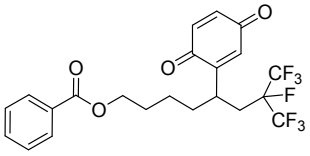
10: 2-phenyl-3-(2,3,3,3-tetrafluoro-2-(trifluoromethyl)propyl)oxirane



10 was obtained as a yellow liquid in 43% yield (26 mg); pentane/ dichloromethane= 1: 10. ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 3H), 7.27 (dd, *J* = 7.7, 1.6 Hz, 2H), 3.73 (s, 1H), 3.20

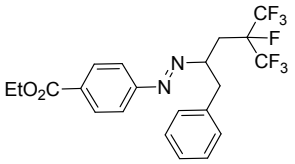
(t, *J* = 5.5 Hz, 1H), 2.62 (ddd, *J* = 20.6, 15.7, 5.2 Hz, 1H), 2.42 – 2.28 (m, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.61 (d, *J* = 6.8 Hz, 6F), -183.27 (tdq, *J* = 20.8, 13.9, 6.9 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 135.91, 128.77, 125.65, 120.86 (qdd, *J* = 34.6, 26.8, 8.3 Hz), 90.91 (ddt, *J* = 204.0, 65.5, 32.8 Hz), 58.62, 55.26 (d, *J* = 6.4 Hz), 32.77 (d, *J* = 20.0 Hz); IR (film, cm^{-1}): ν 2923, 1724, 1464，1325, 1225, 1158, 1127, 1019, 990, 750, 720, 698; MS (EI) m/z (relative intensity) 302 (35) [M$^+$], 90 (100); HRMS (EI) calcd. For $C_{12}H_9OF_7$: 302.0542, Found: 302.0545.

11 5-(3,6-dioxocyclohexa-1,4-dien-1-yl)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)octyl benzoate



11 was obtained as a colorless liquid in 55% yield (53 mg); hexane/ ethyl acetate = 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.56 (t, *J* = 7.0 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 6.73 (s, 2H), 6.55 (s, 1H), 4.28 (t, *J* = 6.3 Hz, 1H), 3.12 (s, 1H), 2.64 (dd, *J* = 24.5, 14.7 Hz, 1H), 2.32 (dd, *J* = 25.9, 15.9 Hz, 1H), 1.83 – 1.65 (m, 4H), 1.46 – 1.28 (m, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.01 – -76.17 (m, 3F), -77.31 – -77.43 (m, 3F), -184.60 (dtt, *J* = 27.5, 13.7, 6.7 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 187.27, 186.66, 166.75, 149.68, 137.21, 136.15, 133.28, 133.11, 130.32, 129.64, 128.51, 120.96 (qdd, *J* = 285.8, 27.8, 18.6 Hz), 93.49 – 90.18 (m), 64.40, 35.50, 31.96 (d, *J* = 18.9 Hz), 28.41, 23.87; IR (film, cm^{-1}): ν 3424, 3065, 2954, 2865, 1719, 1659, 1601, 1508, 1452, 1315, 1279, 1224, 1158, 1118, 1070, 976, 713; MS (ESI) m/z (relative intensity) 502.9 (100) [M+Na]$^+$; HRMS (ESI) calcd. For $C_{22}H_{20}O_4F_7$ [M+H]$^+$: 481.1244, Found: 481.1237.

12 ethyl 4-((4,5,5,5-tetrafluoro-1-phenyl-4-(trifluoromethyl)pentan-2-yl)diazenyl)benzoate



12 was obtained as a yellow liquid in 67% yield (62 mg); hexane/ ethyl acetate = 20/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.5 Hz, 2H), 7.62 (d, *J* = 8.5 Hz, 2H), 7.26 (dq, *J* = 14.4, 7.1 Hz, 3H), 7.15 (d, *J* = 7.0 Hz, 2H), 4.54 (dd, *J* = 13.1, 6.5 Hz, 1H), 4.41 (q, *J* = 7.1 Hz, 2H), 3.21 – 3.01 (m, 3H), 2.51 (dd, *J* = 23.9, 15.5 Hz, 1H), 1.42 (t, *J* = 7.1 Hz, 3H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.43 – -76.63 (m, 3F), -76.92 – -77.09 (m, 3F), -183.67 (pt, *J* = 14.5, 7.1 Hz, 1F); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ 166.06, 154.35, 136.28, 132.37, 130.67, 129.79, 128.77, 127.13, 122.11, 120.99 (qd, *J* = 285.4, 27.5 Hz), 93.36 – 89.66 (m), 72.65, 61.40, 40.80, 30.76 (d, *J* = 19.1 Hz), 14.42; IR (film, cm^{-1}): ν 3030, 2984, 1721, 1605, 1368, 1304, 1275, 1224, 1108, 1016, 955, 773, 699; MS (EI) m/z (relative intensity) 464 (0.5) [M$^+$], 149 (100); HRMS (EI) calcd. For $C_{21}H_{19}N_2O_2F_7$: 464.1335, Found: 464.1330.

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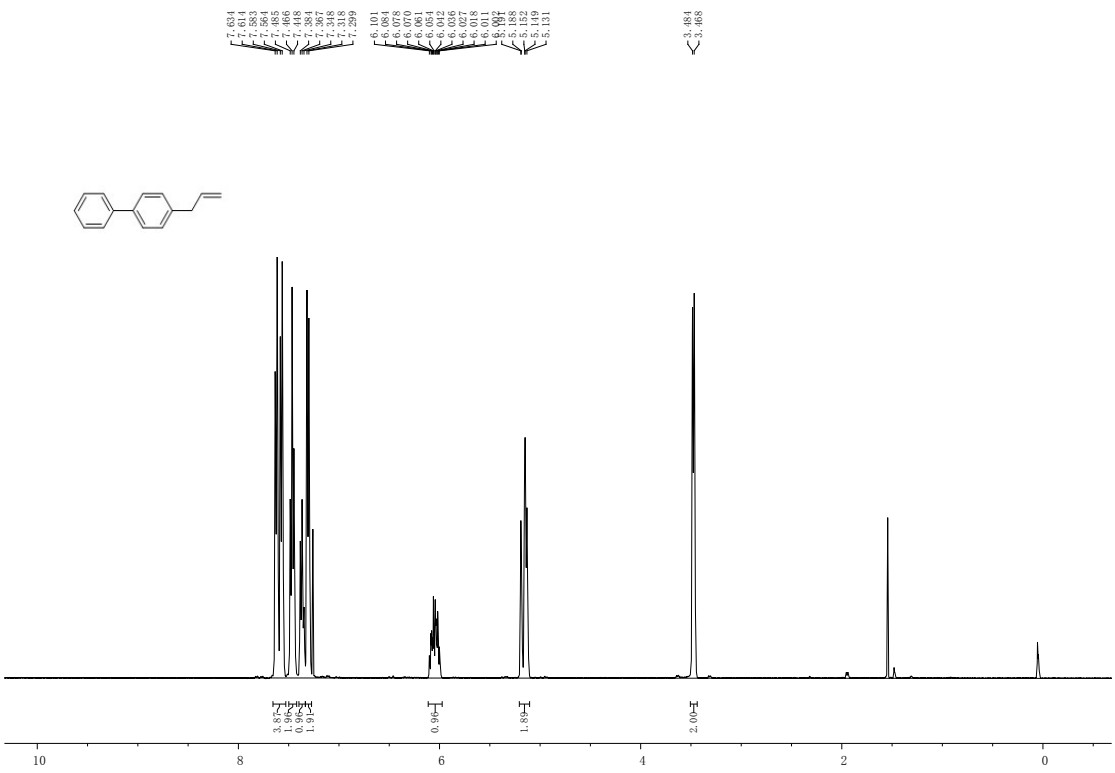
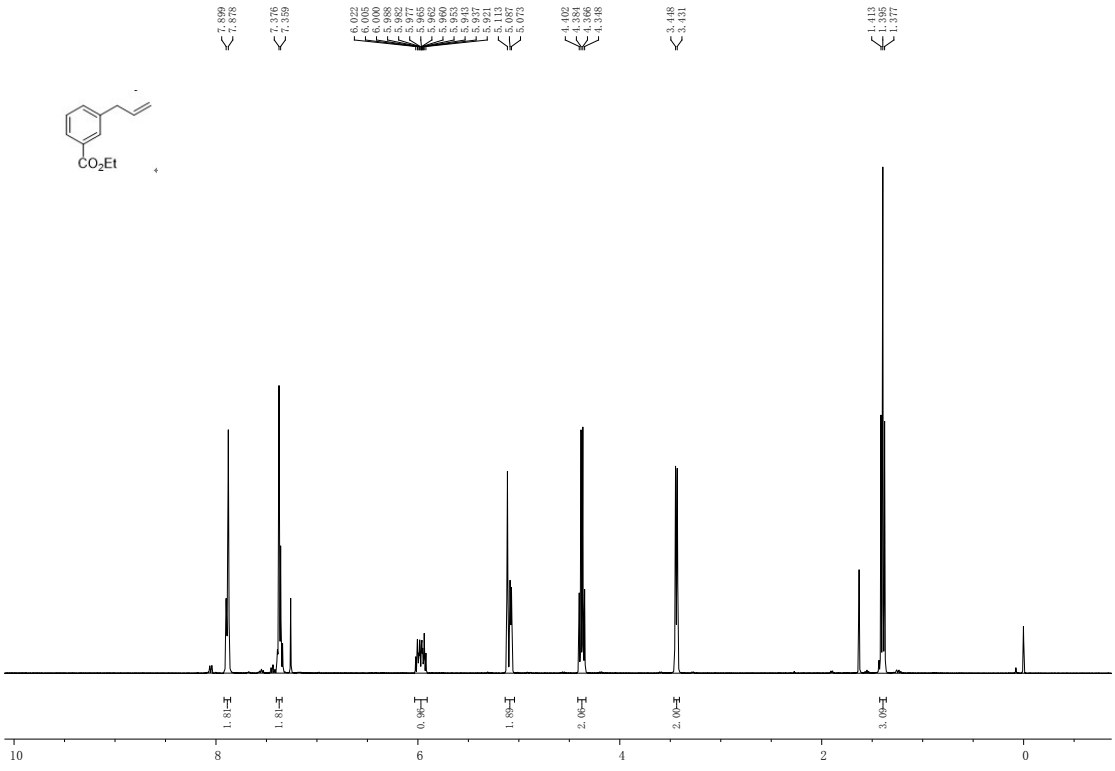
9 Spectral for substrates

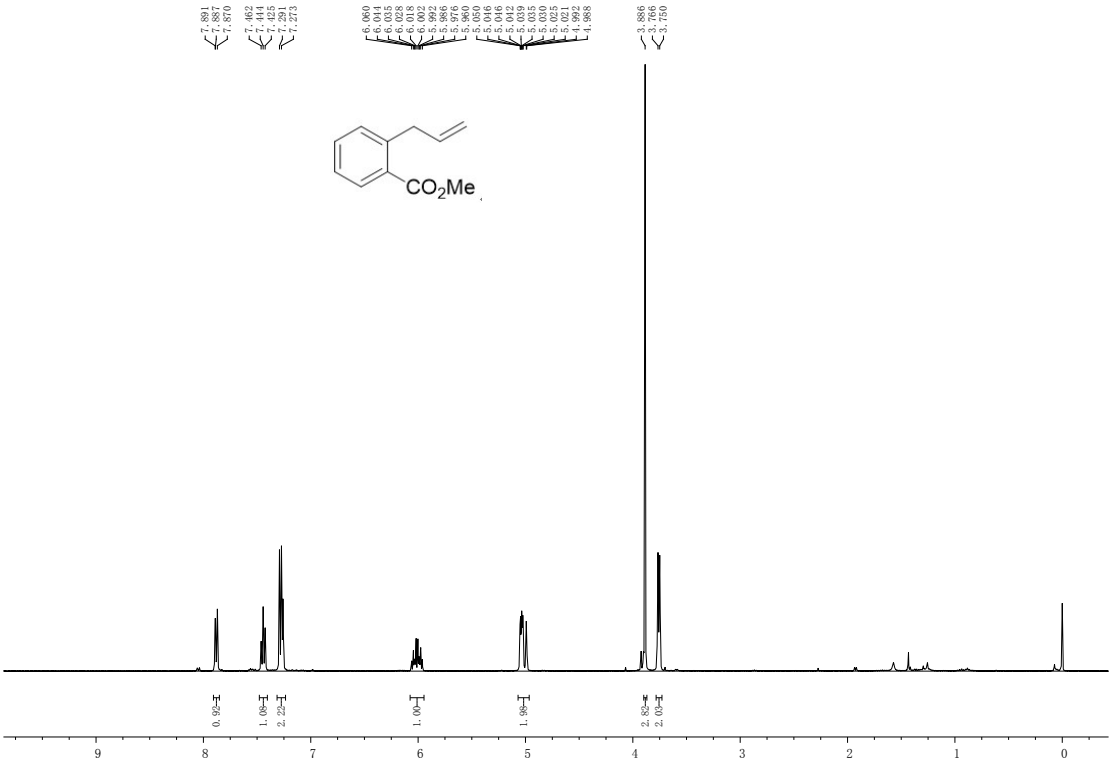
^{1}H NMR spectrum for 1c: 4-allyl-1,1'-biphenyl



^{1}H NMR spectrum for 1d: ethyl 3-allylbenzoate

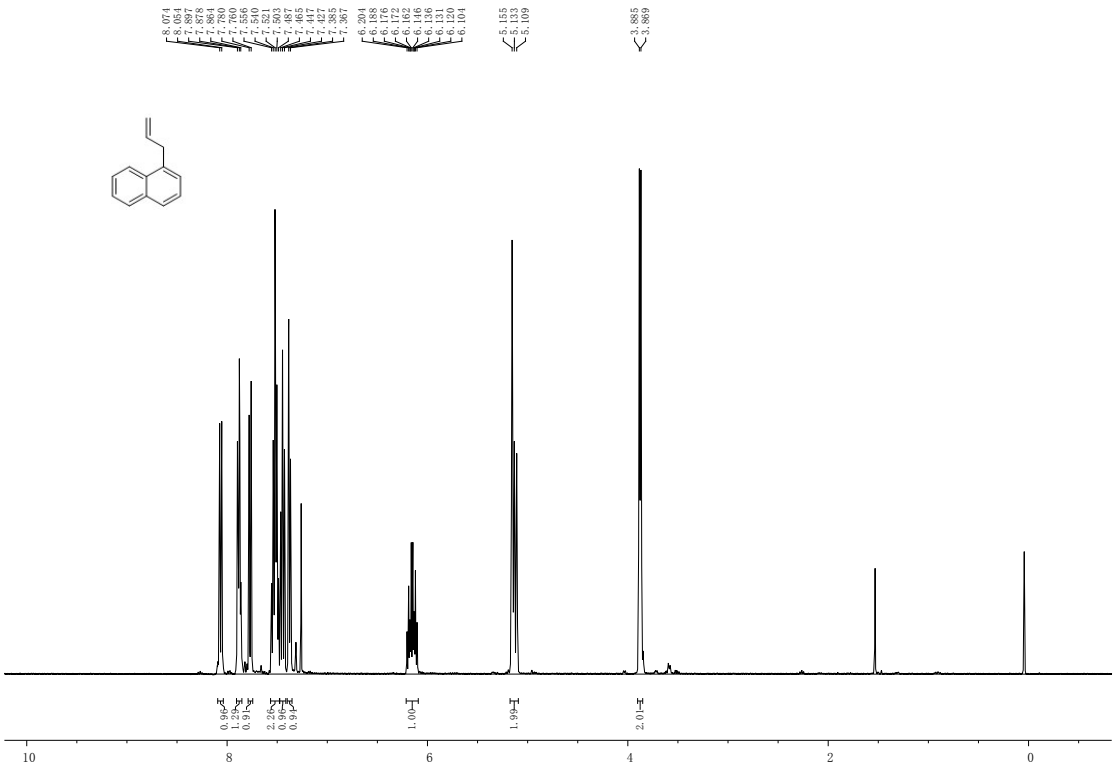


^{1}H NMR spectrum for 1e: methyl 2-allylbenzoate

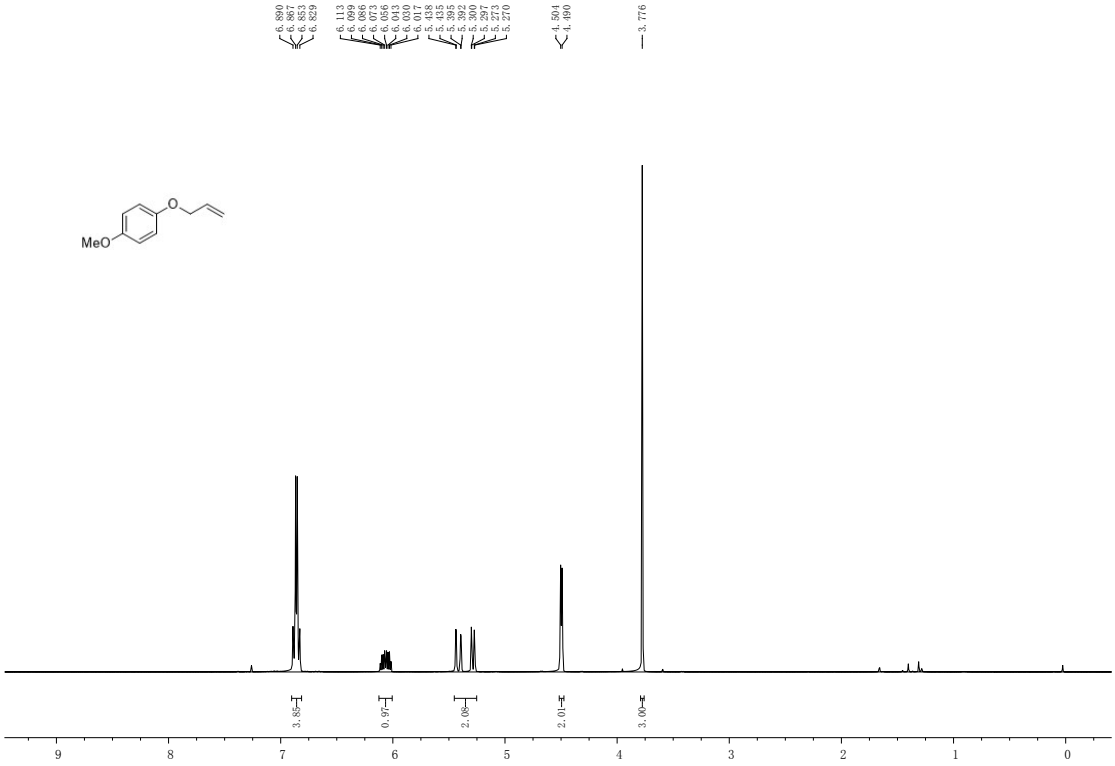


^{1}H NMR spectrum for 1f: 1-allylnaphthalene

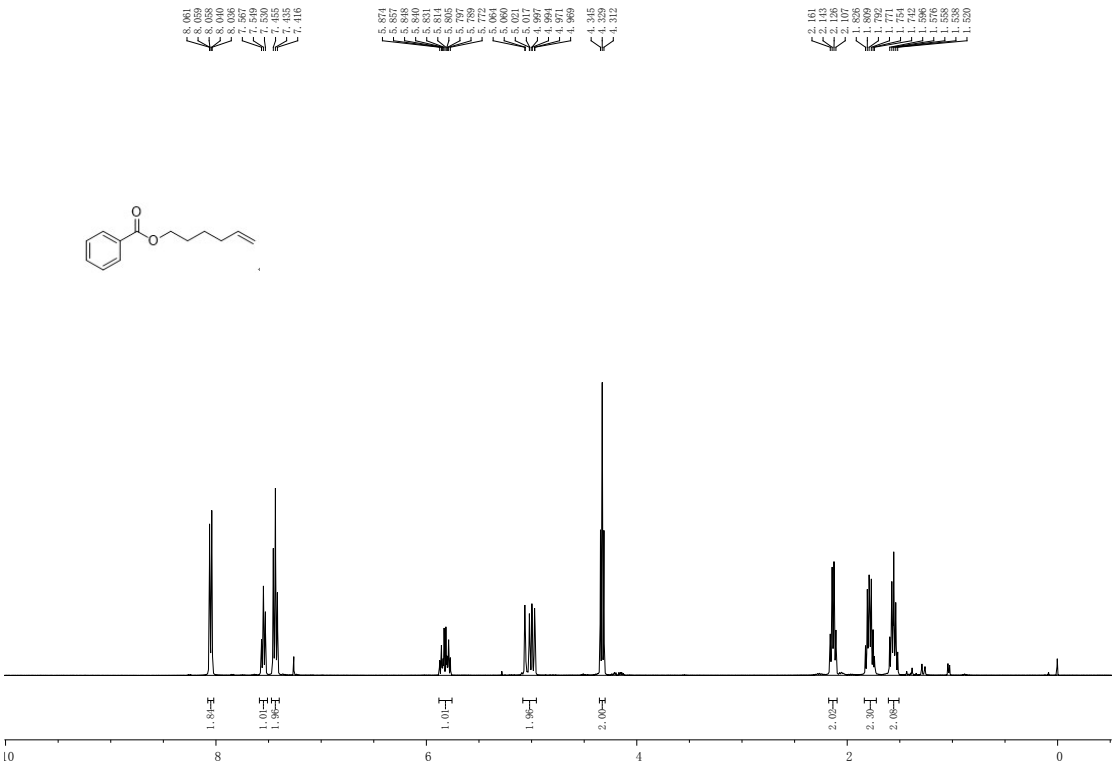
f



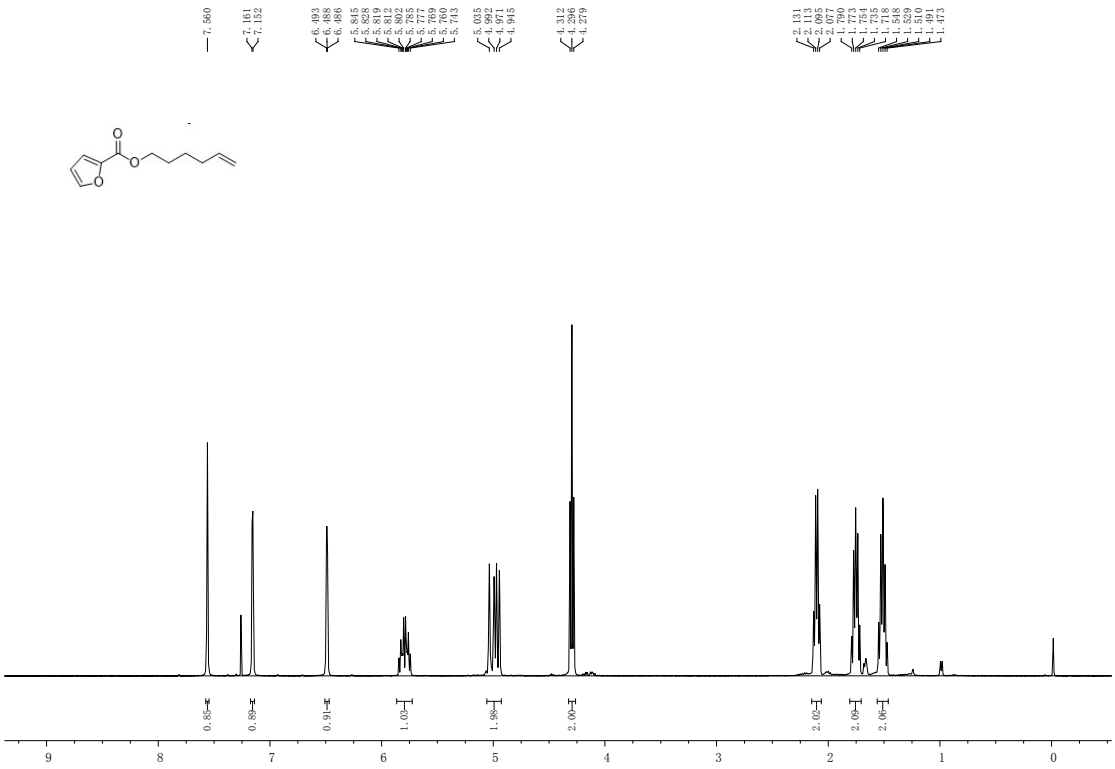
^{1}H NMR spectrum for 1g: 1-(allyloxy)-4-methoxybenzene



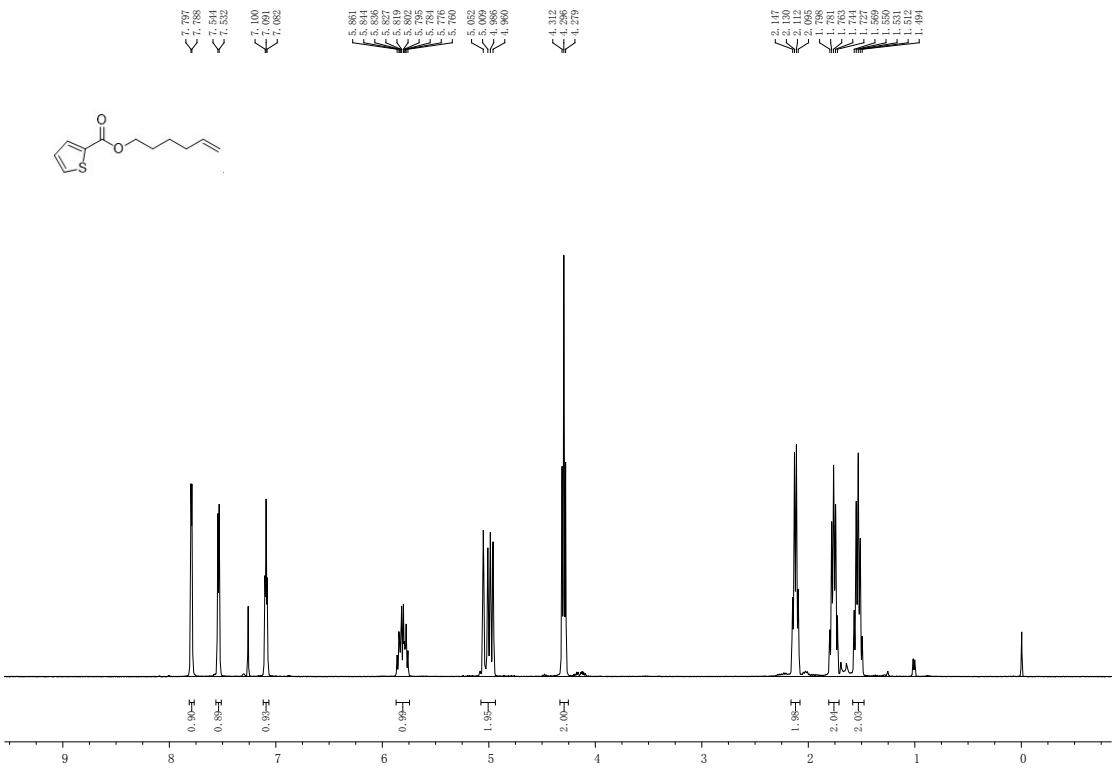
^{1}H NMR spectrum for 1i: hex-5-en-1-yl benzoate



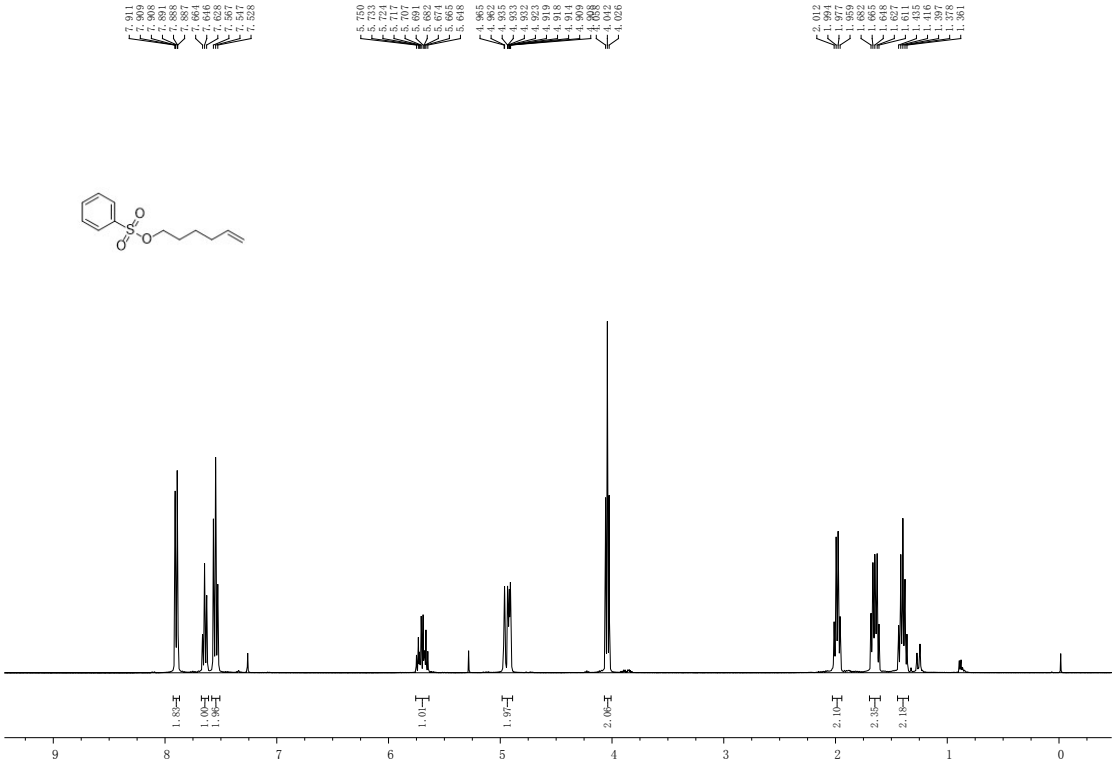
^{1}H NMR spectrum for 1j: hex-5-en-1-yl furan-2-carboxylate



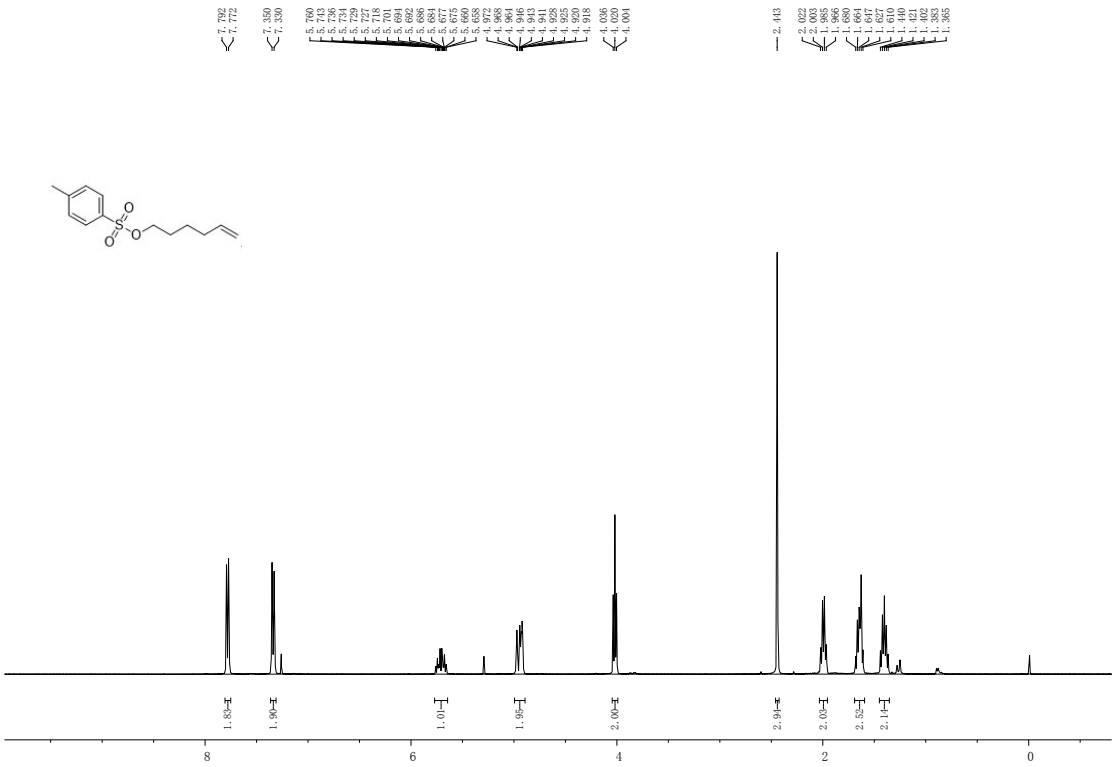
^{1}H NMR spectrum for 1k: hex-5-en-1-yl thiophene-2-carboxylate



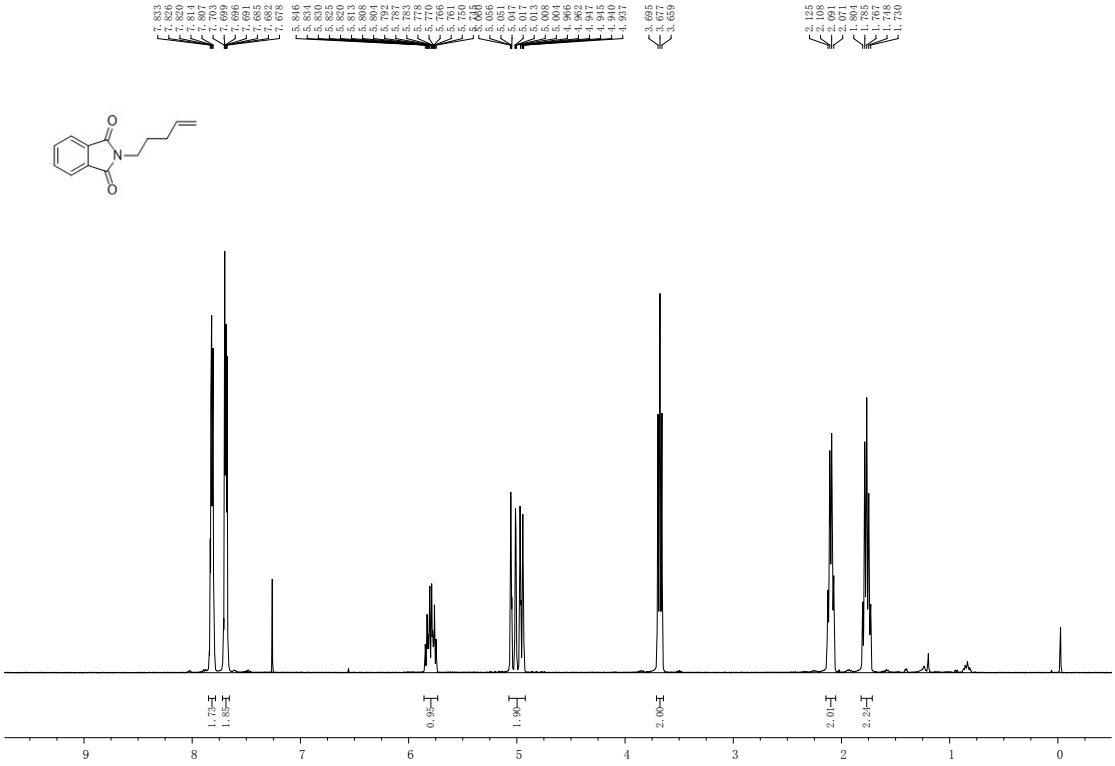
^{1}H NMR spectrum for 1l: hex-5-en-1-yl benzenesulfonate



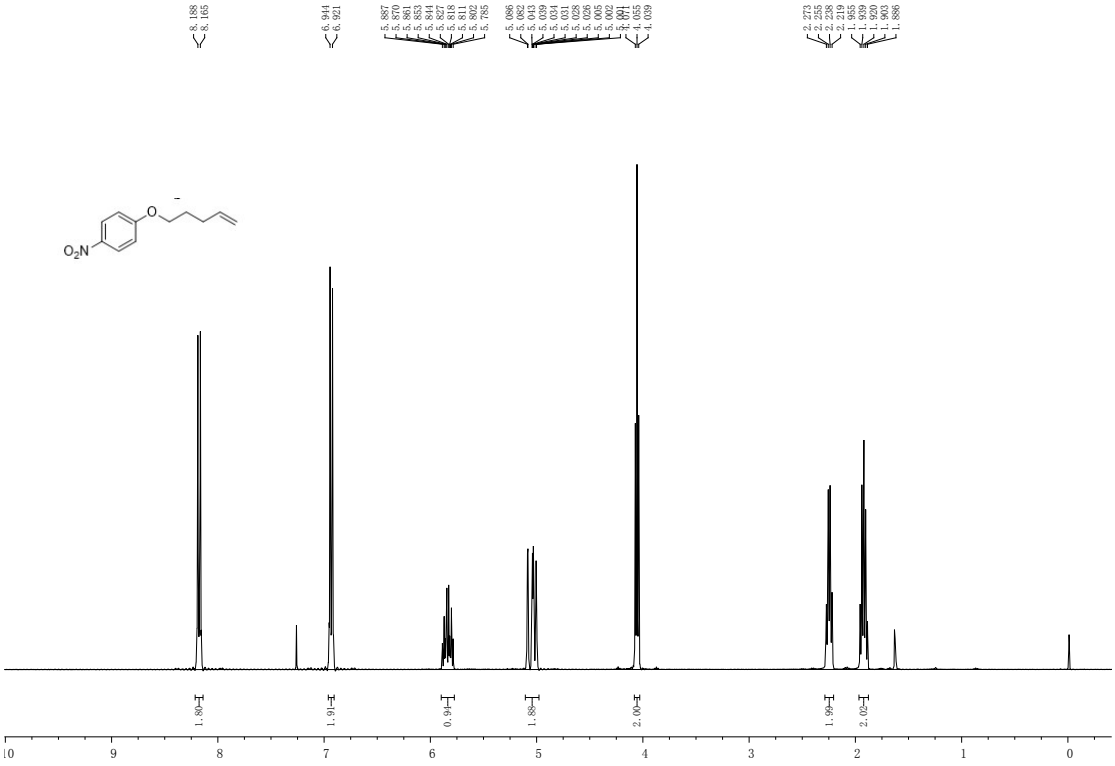
^{1}H NMR spectrum for 1m: hex-5-en-1-yl 4-methylbenzenesulfonate



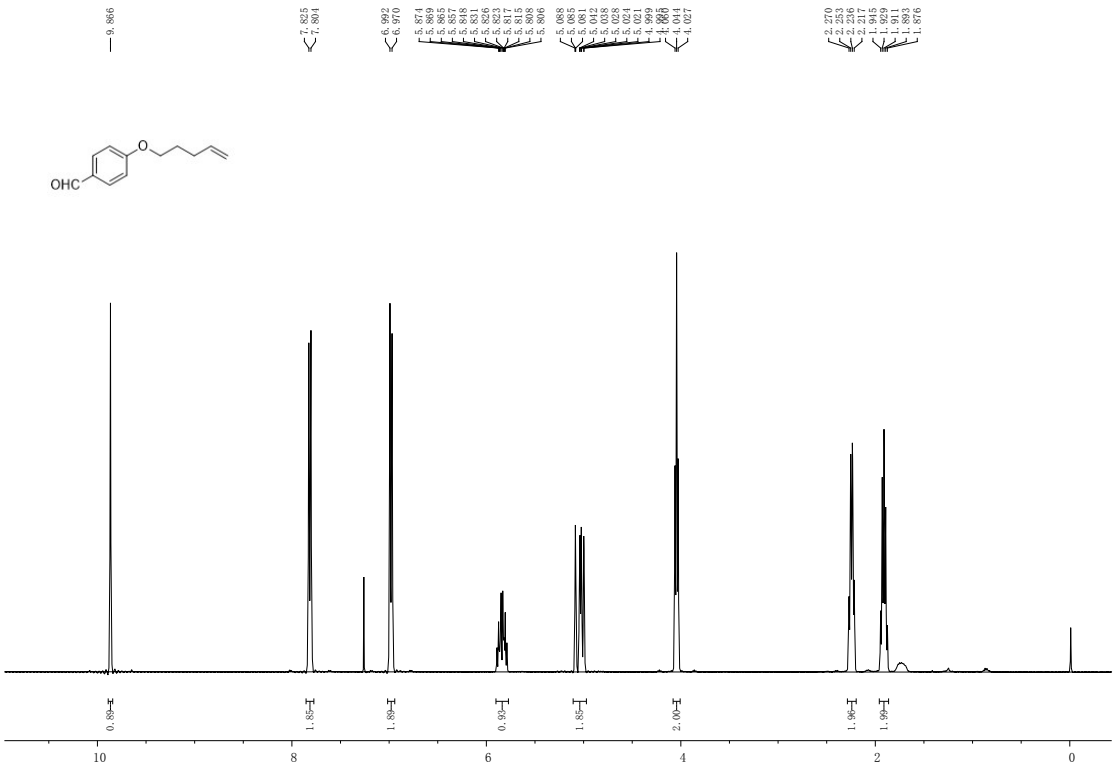
^{1}H NMR spectrum for 1n: 2-(pent-4-en-1-yl)isoindoline-1,3-dione



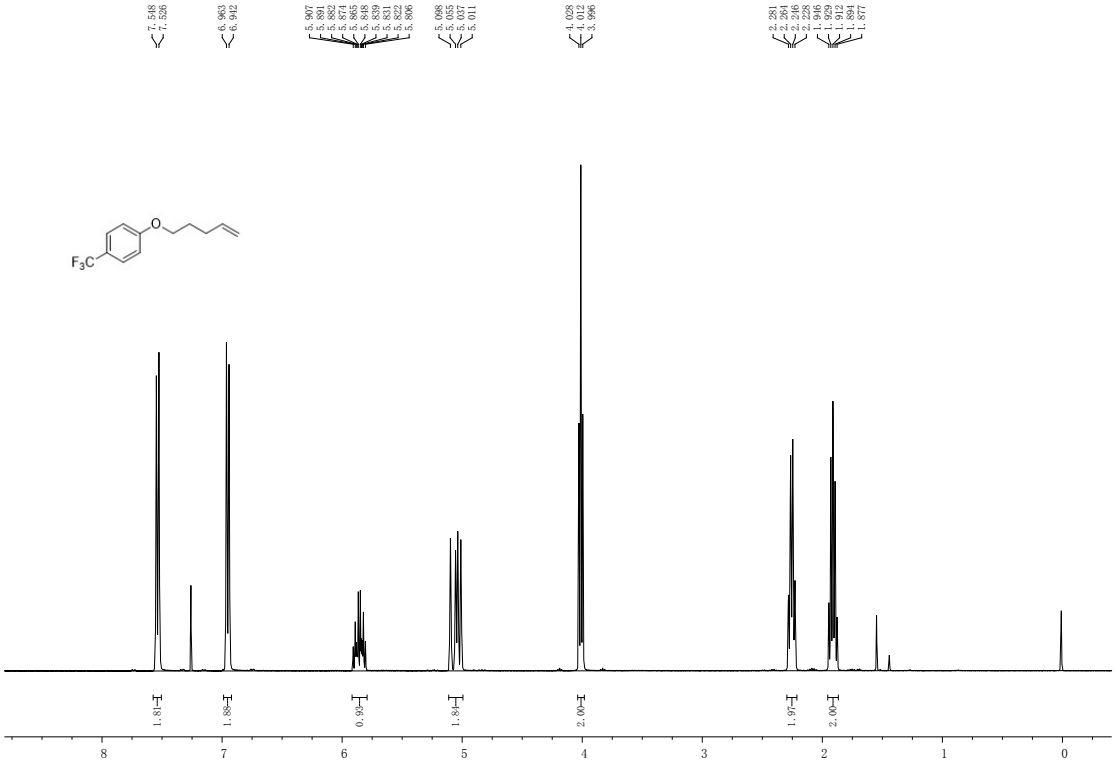
^{1}H NMR spectrum for 1o: 1-nitro-4-(pent-4-en-1-yloxy)benzene



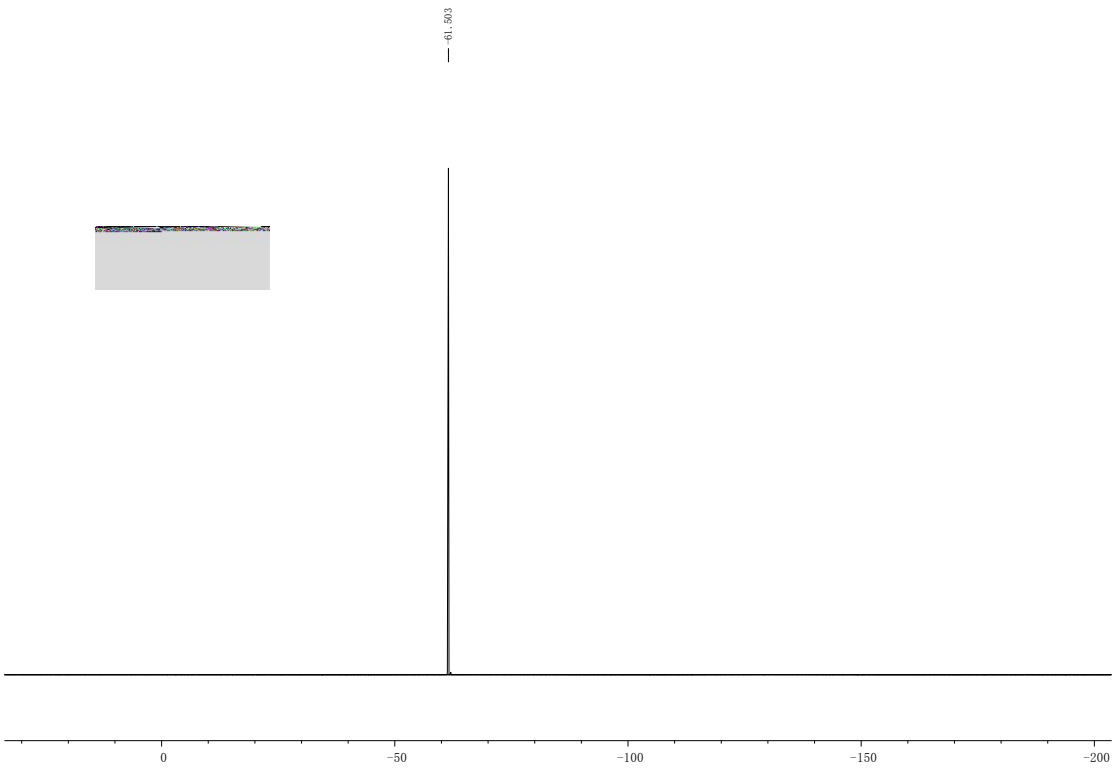
^{1}H NMR spectrum for 1p: 4-(pent-4-en-1-yloxy)benzaldehyde



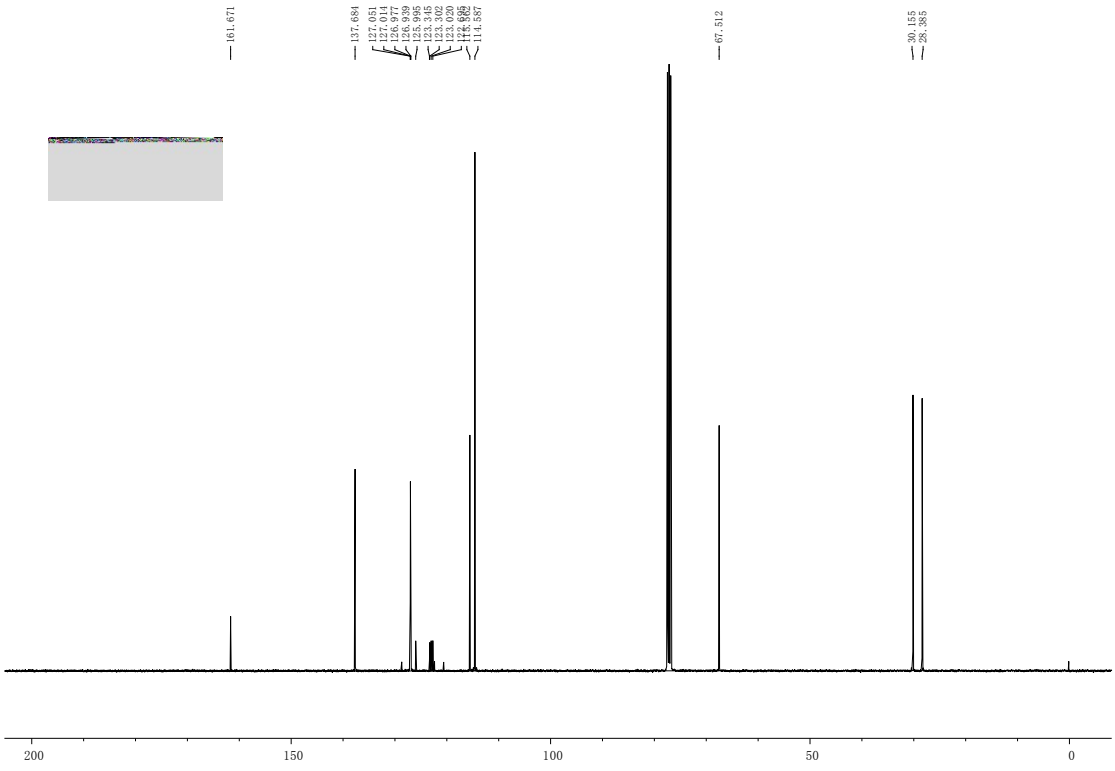
^{1}H NMR spectrum for 1q:1-(pent-4-en-1-yloxy)-4-(trifluoromethyl)benzene



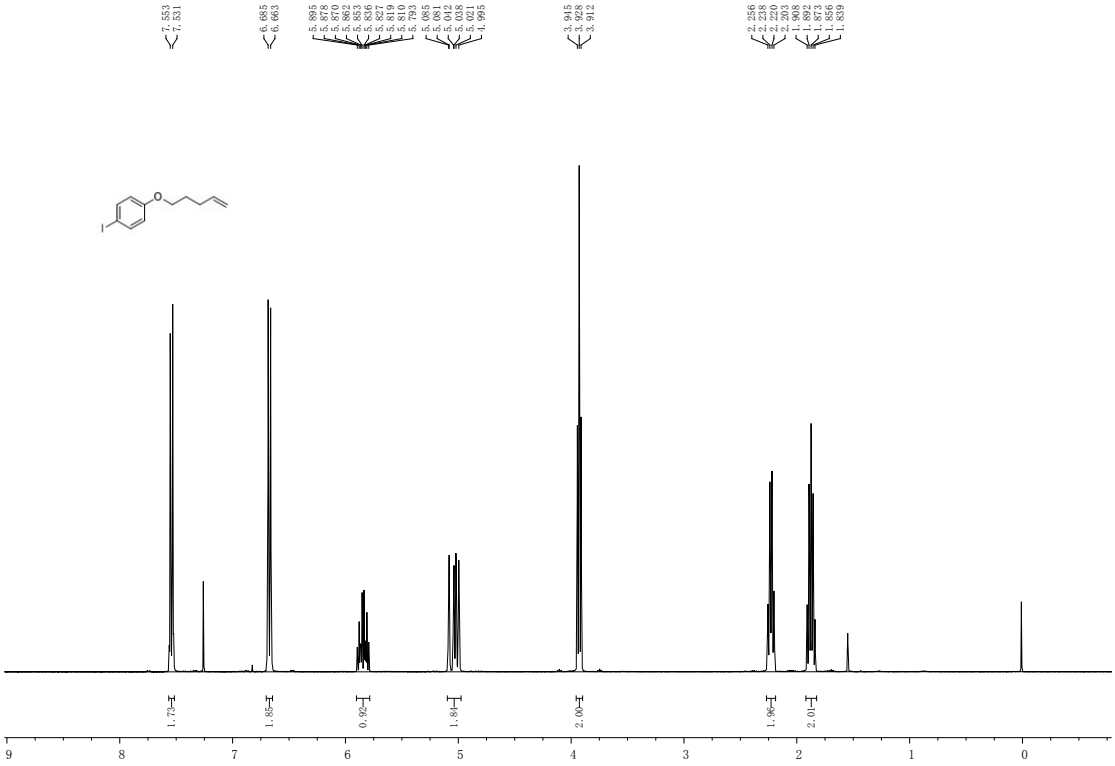
^{19}F NMR spectrum for 1q: 1-(pent-4-en-1-yloxy)-4-(trifluoromethyl)benzene



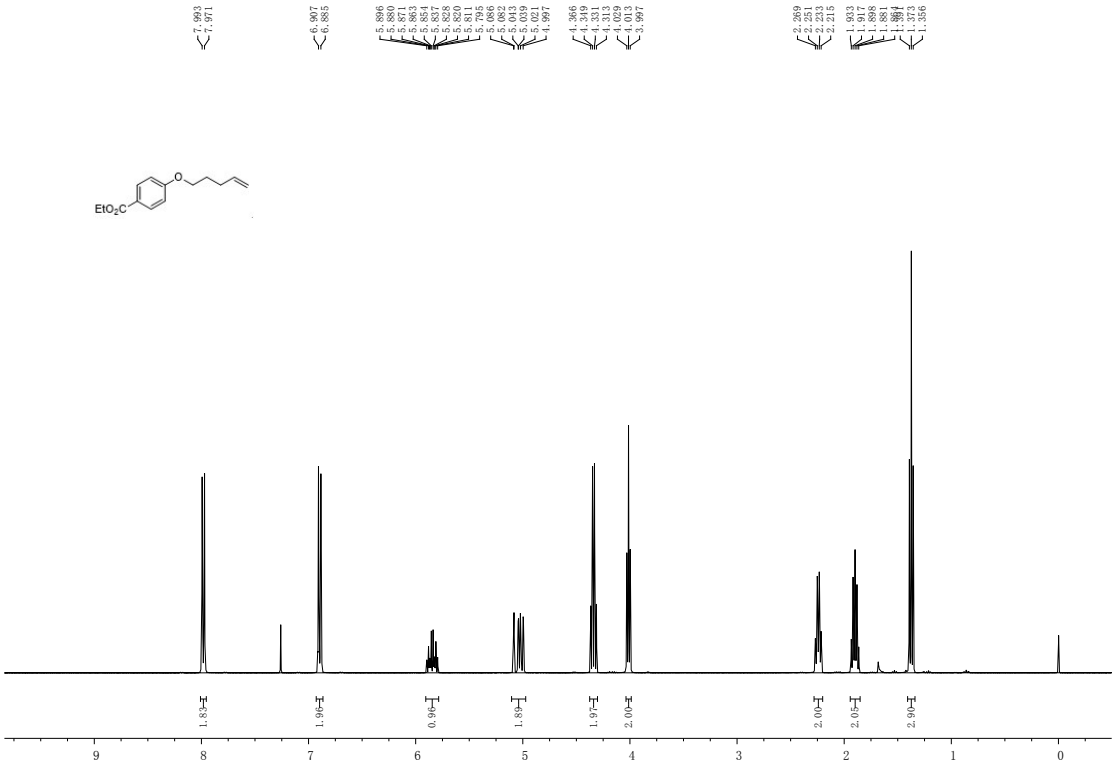
^{13}C NMR spectrum for 1q: 1-(pent-4-en-1-yloxy)-4-(trifluoromethyl)benzene



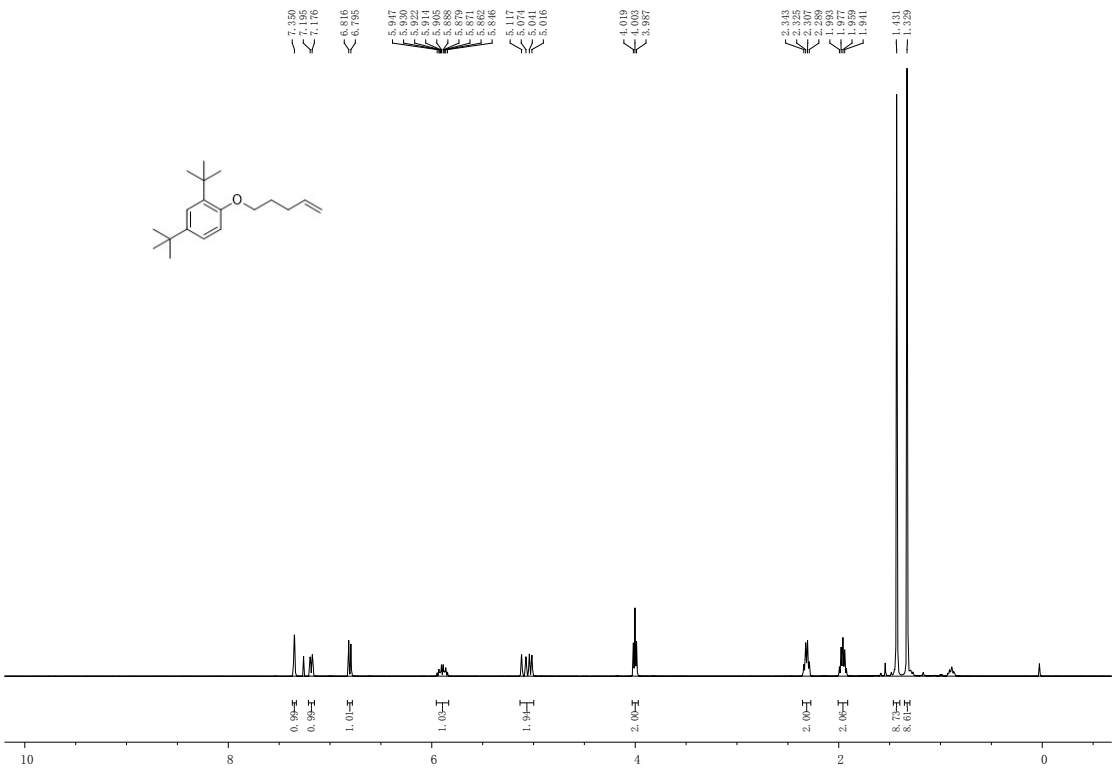
^{1}H NMR spectrum for 1r: 1-iodo-4-(pent-4-en-1-yloxy)benzene



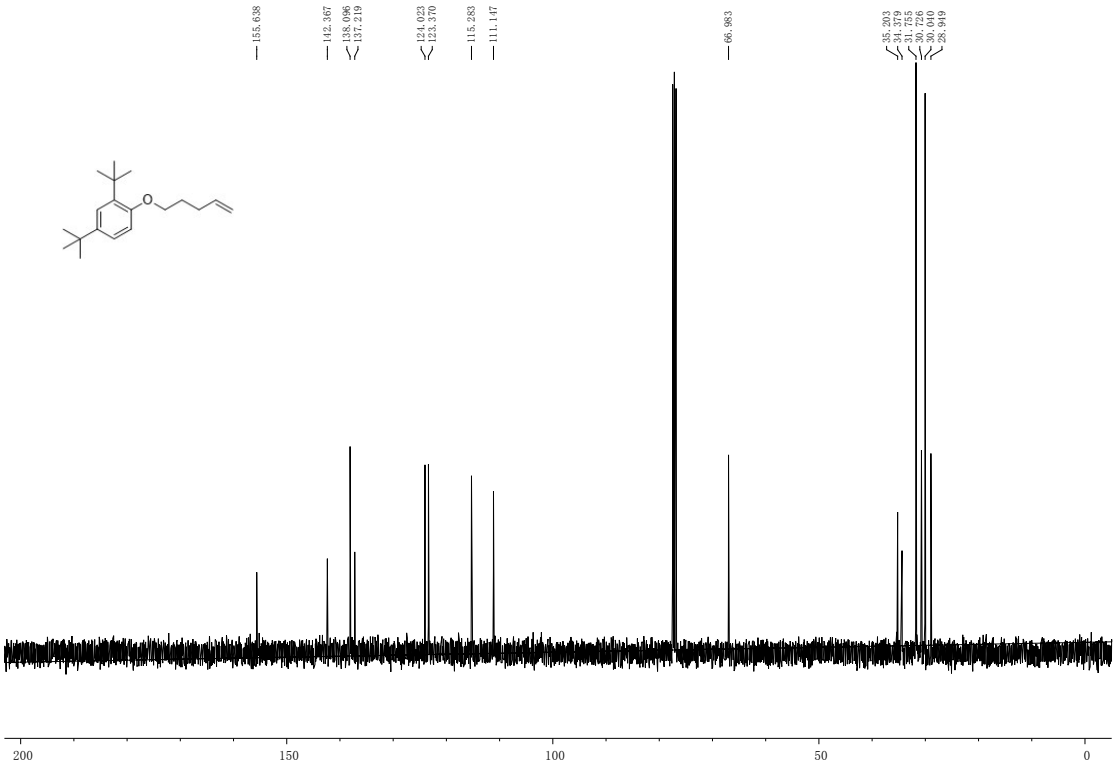
^{1}H NMR spectrum for 1s: ethyl 4-(pent-4-en-1-yloxy)benzoate



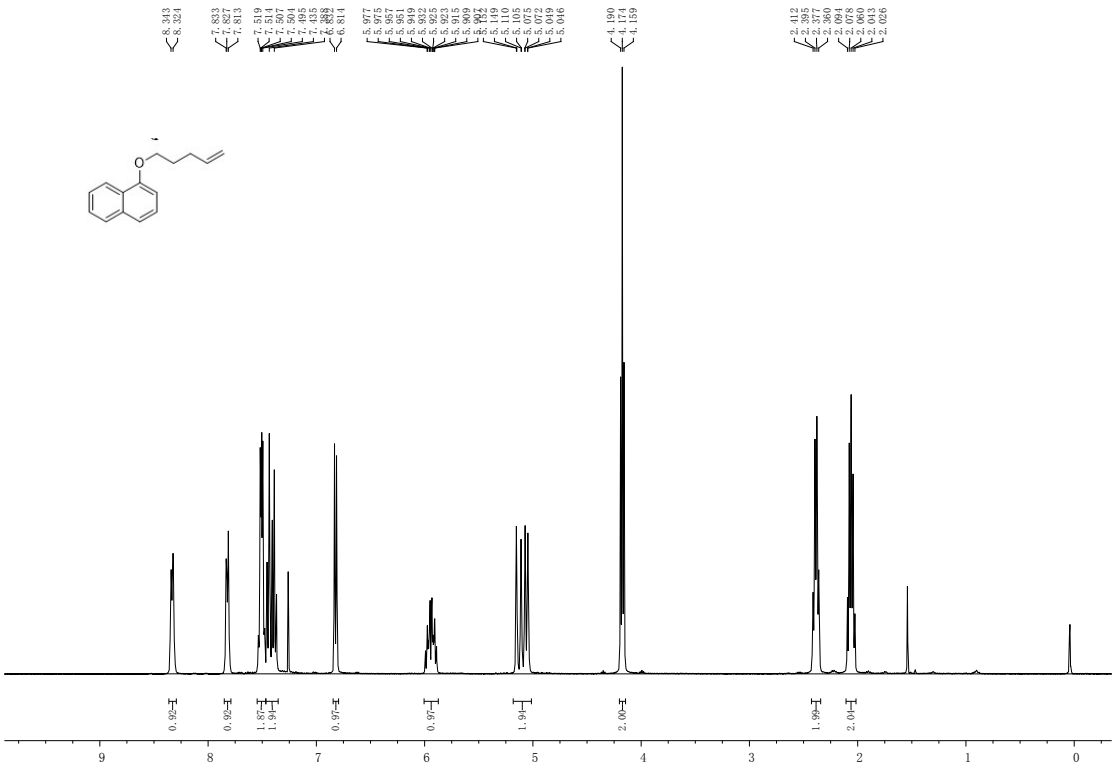
^{1}H NMR spectrum for 1t: 2,4-di-*tert*-butyl-1-(pent-4-en-1-yloxy)benzene



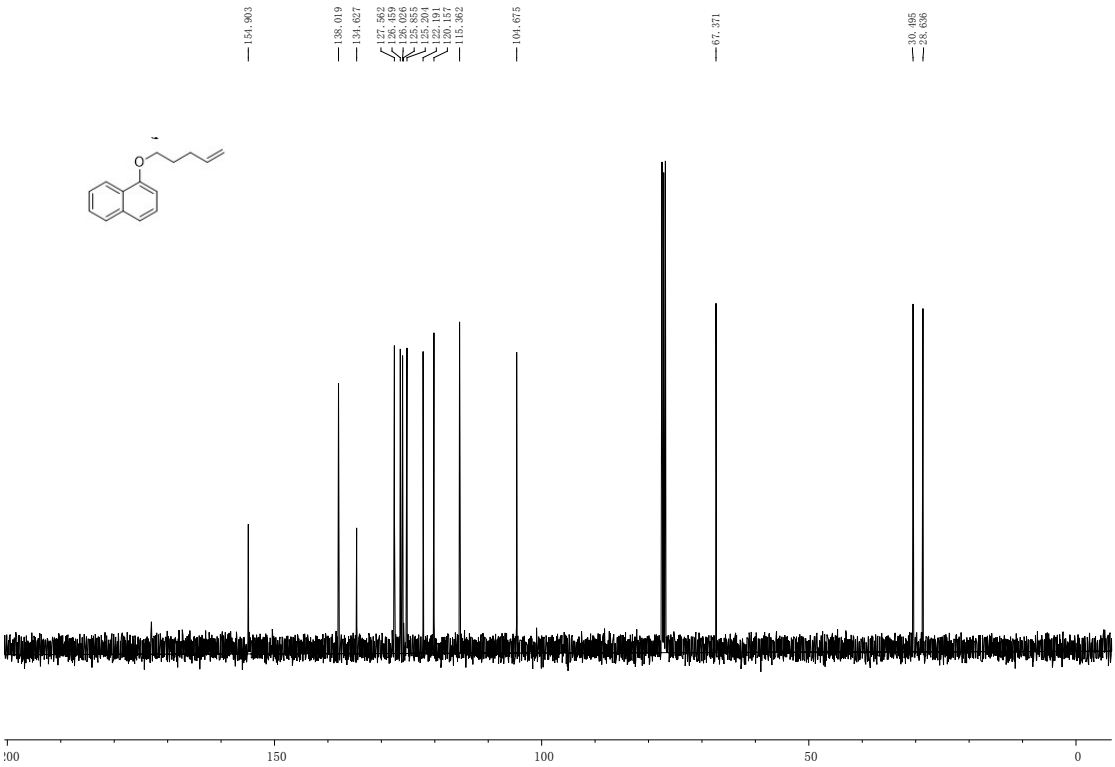
^{13}C NMR spectrum for 1t: 2,4-di-*tert*-butyl-1-(pent-4-en-1-yloxy)benzene



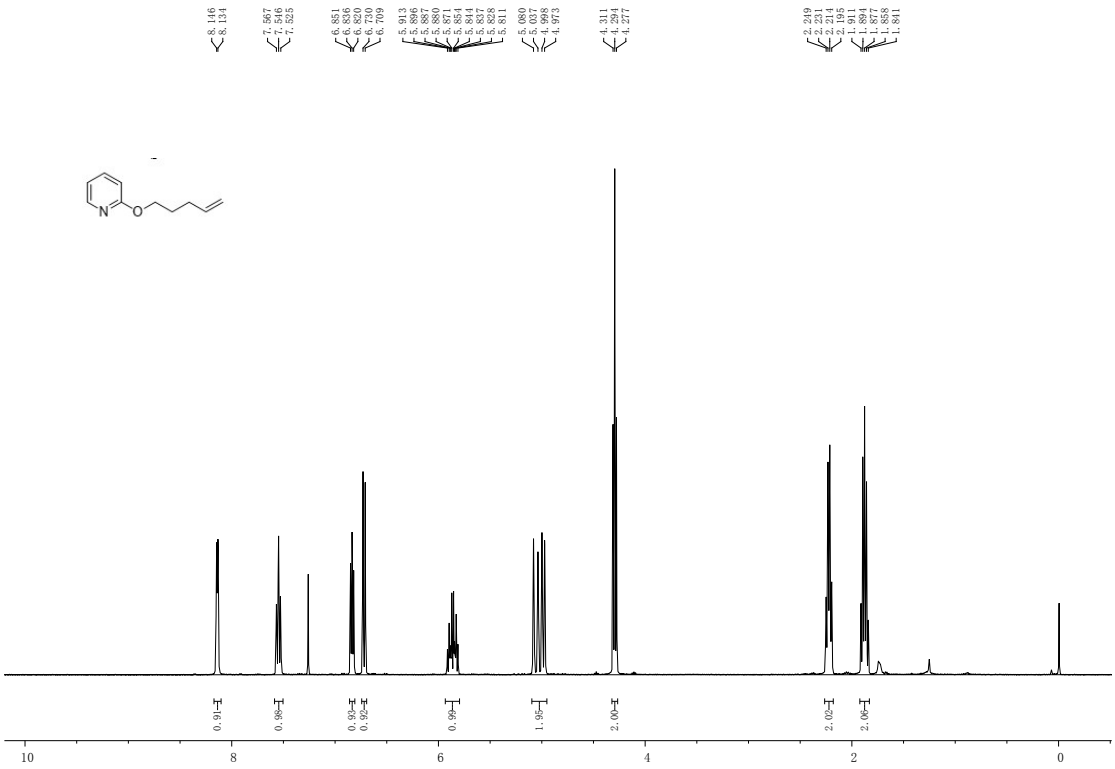
^{1}H NMR spectrum for 1u: 1-(pent-4-en-1-yloxy)naphthalene



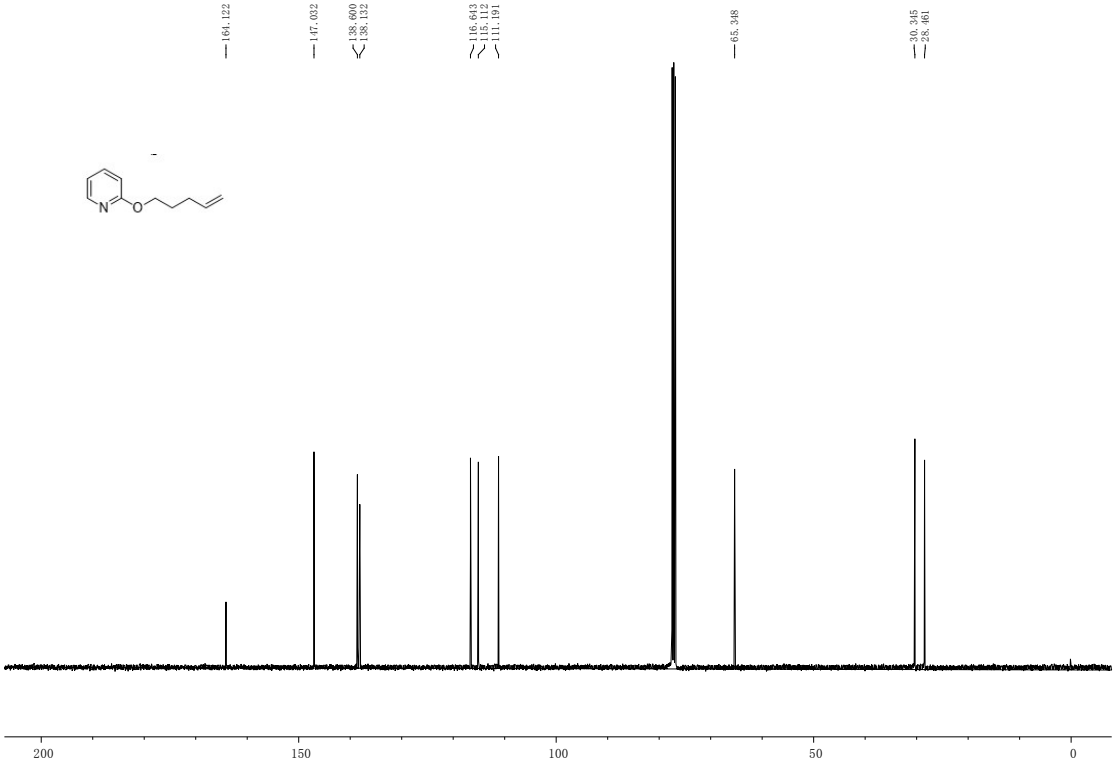
^{13}C NMR spectrum for 1u: 1-(pent-4-en-1-yloxy)naphthalene



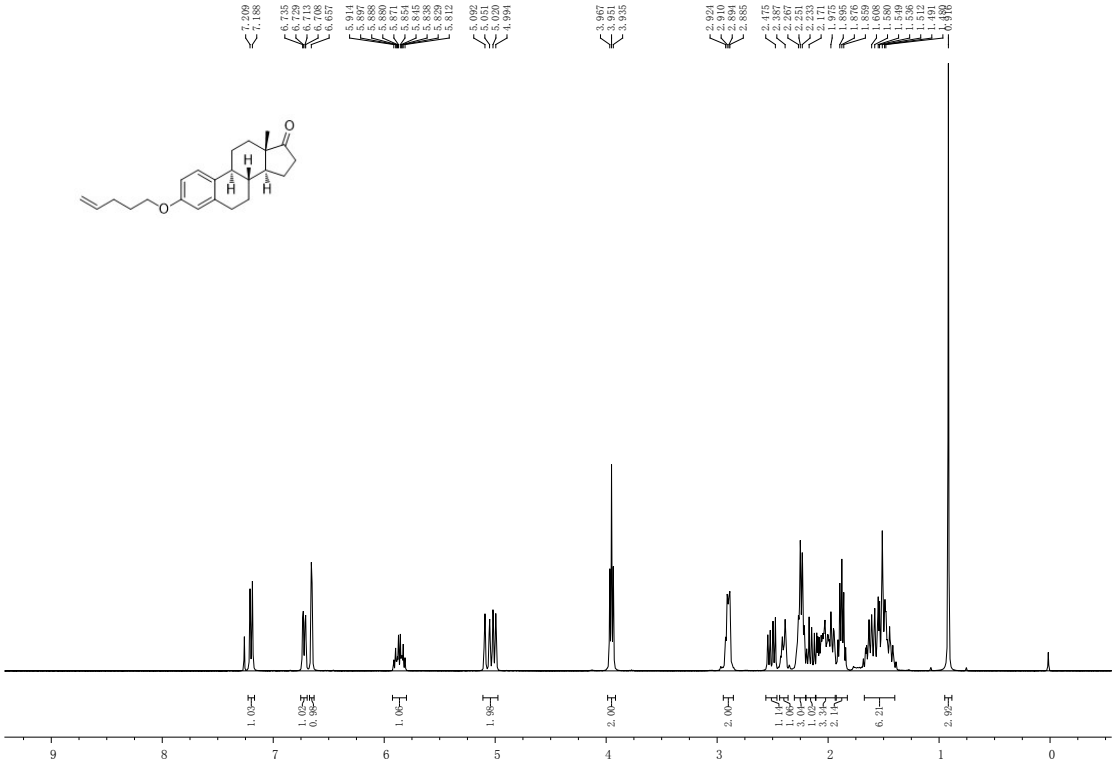
^{1}H NMR spectrum for 1v: 2-(pent-4-en-1-yloxy)pyridine



^{13}C NMR spectrum for 1v: 2-(pent-4-en-1-yloxy)pyridine

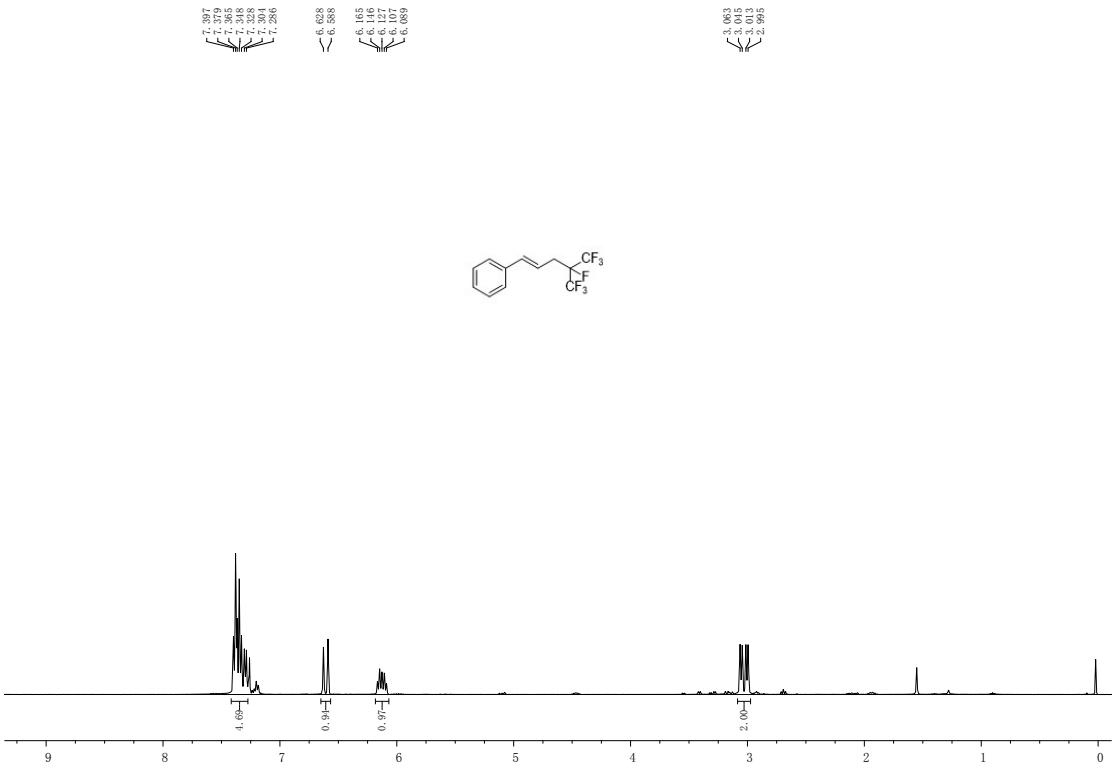


^{1}H NMR spectrum for 1x: (*8R,9S,13S,14S*)-13-methyl-3-(pent-4-en-1-yloxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one

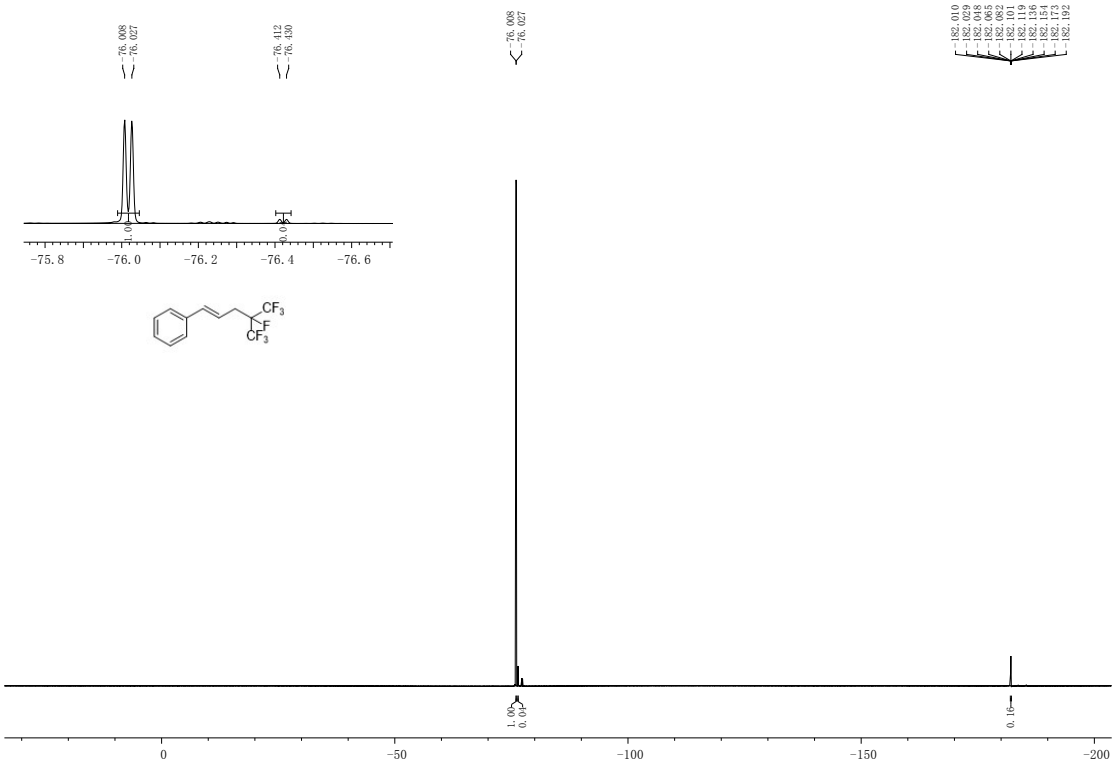


10 Spectral for products

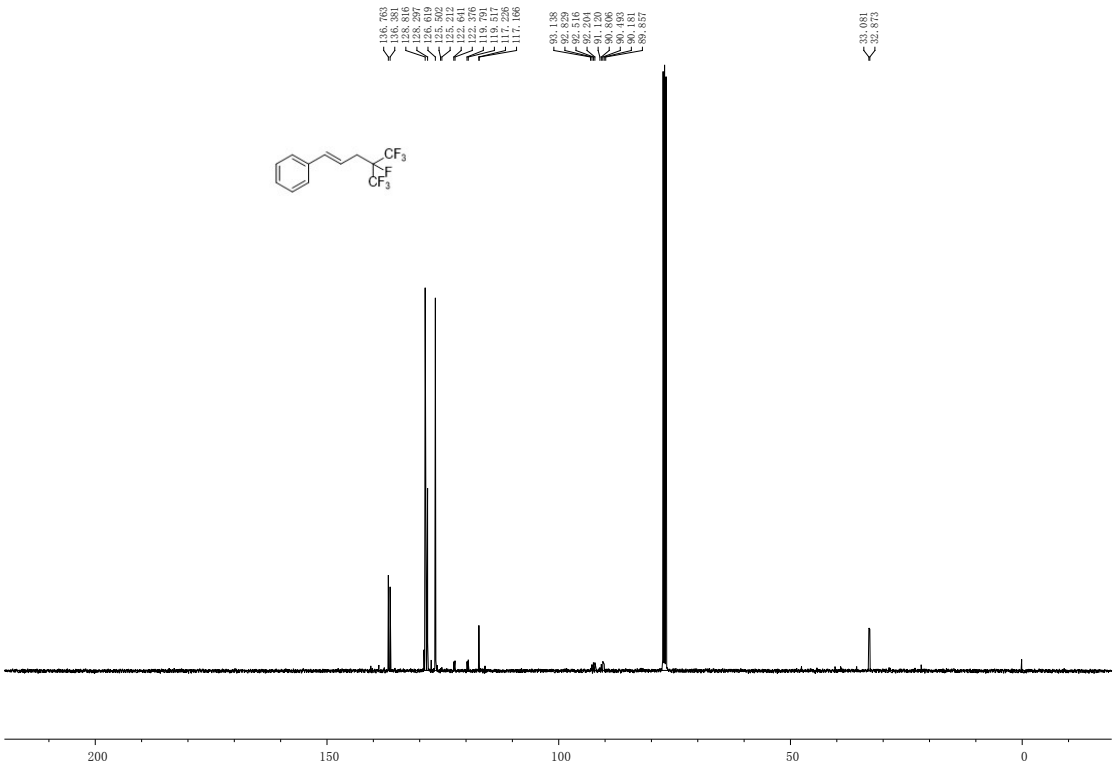
^{1}H NMR spectrum for 2a: (*E*)-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene



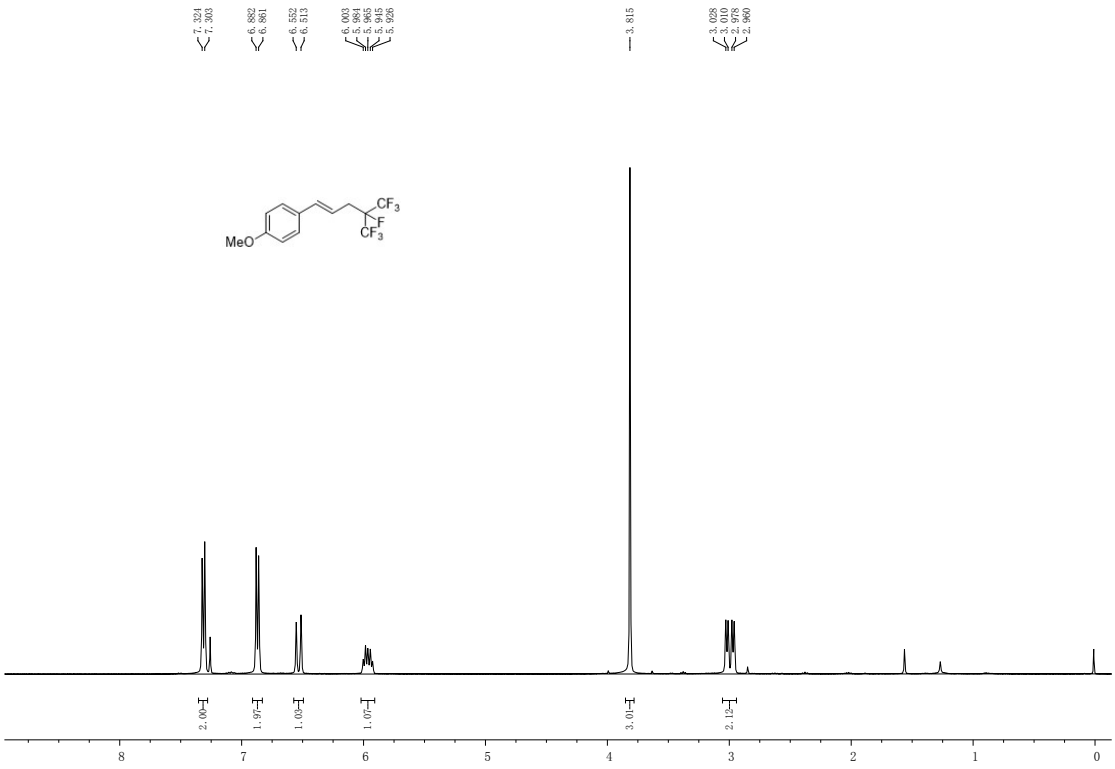
^{19}F NMR spectrum for 2a:(*E*)-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene



^{13}C NMR spectrum for 2a: (*E*)-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene

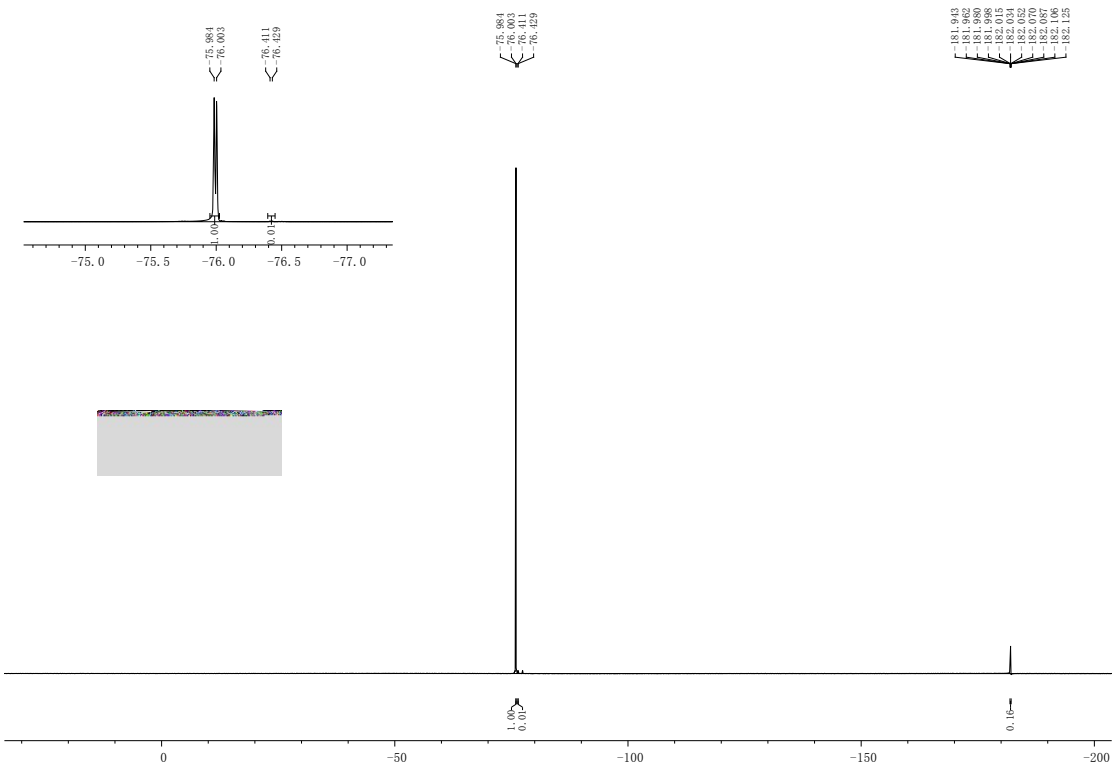


^{1}H NMR spectrum for 2b: (*E*)-1-methoxy-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene

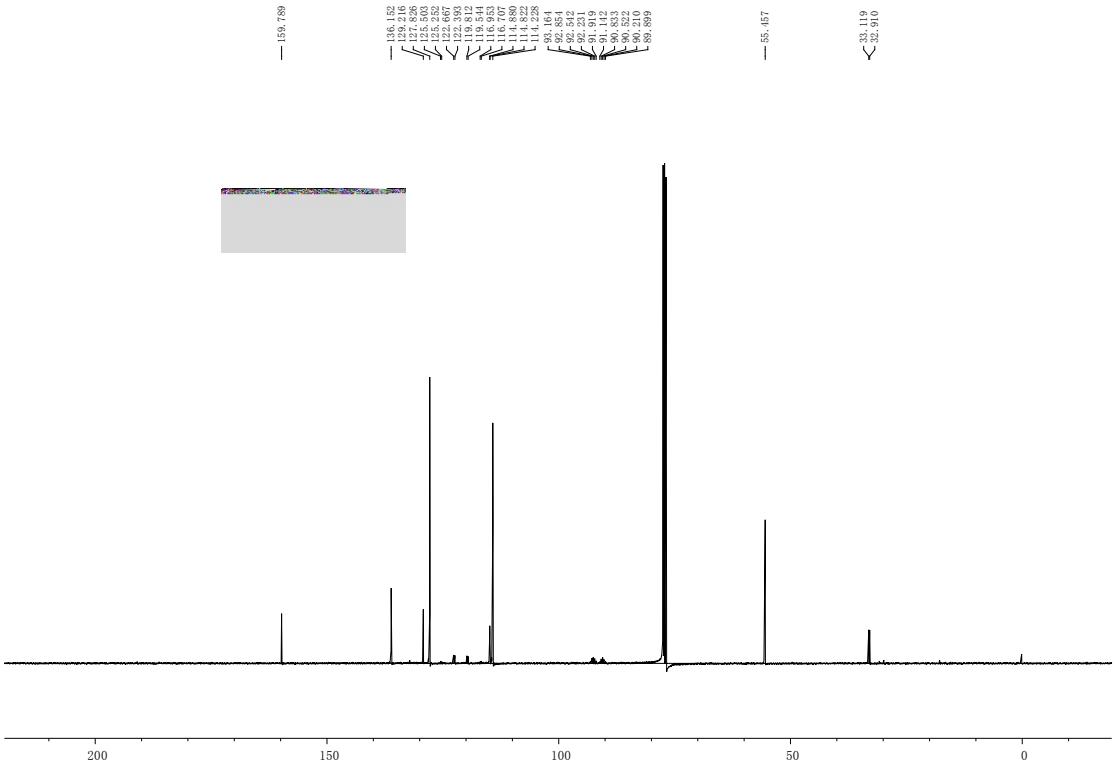


^{19}F NMR spectrum for 2b: (*E*)-1-methoxy-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-

en-1-yl)benzene

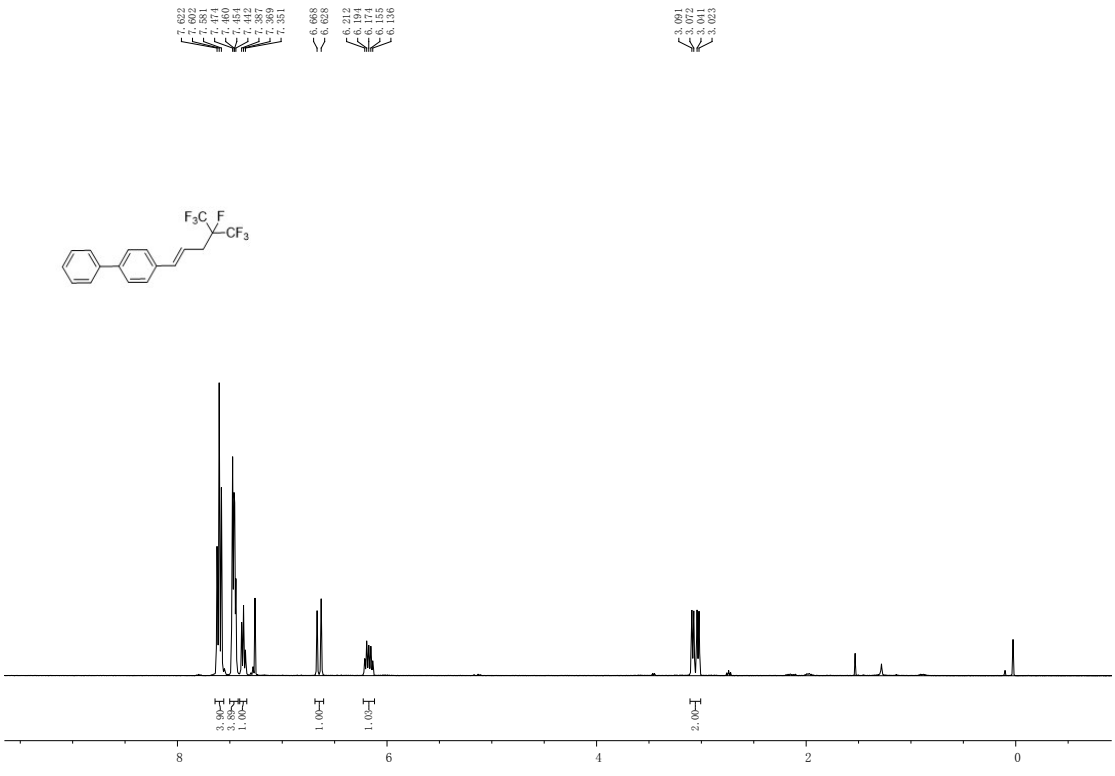


^{13}C NMR spectrum for 2b: (*E*)-1-methoxy-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzene

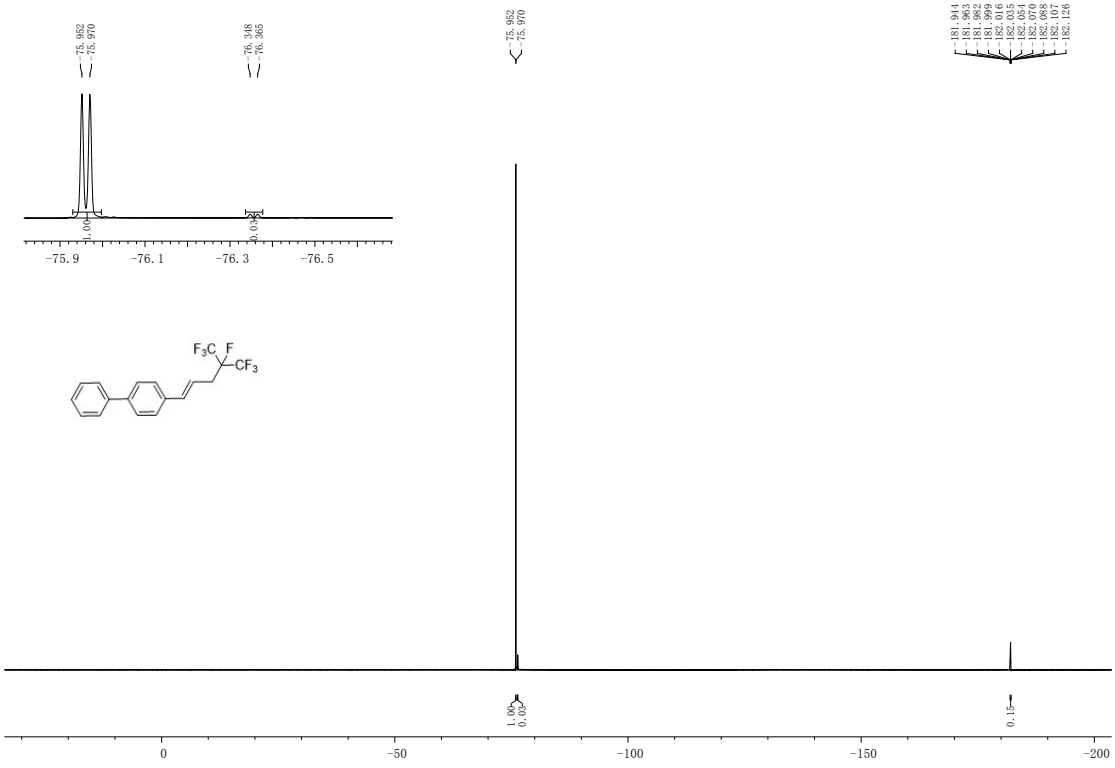


^{1}H NMR spectrum for 2c: (*E*)-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)-1,1'-

biphenyl

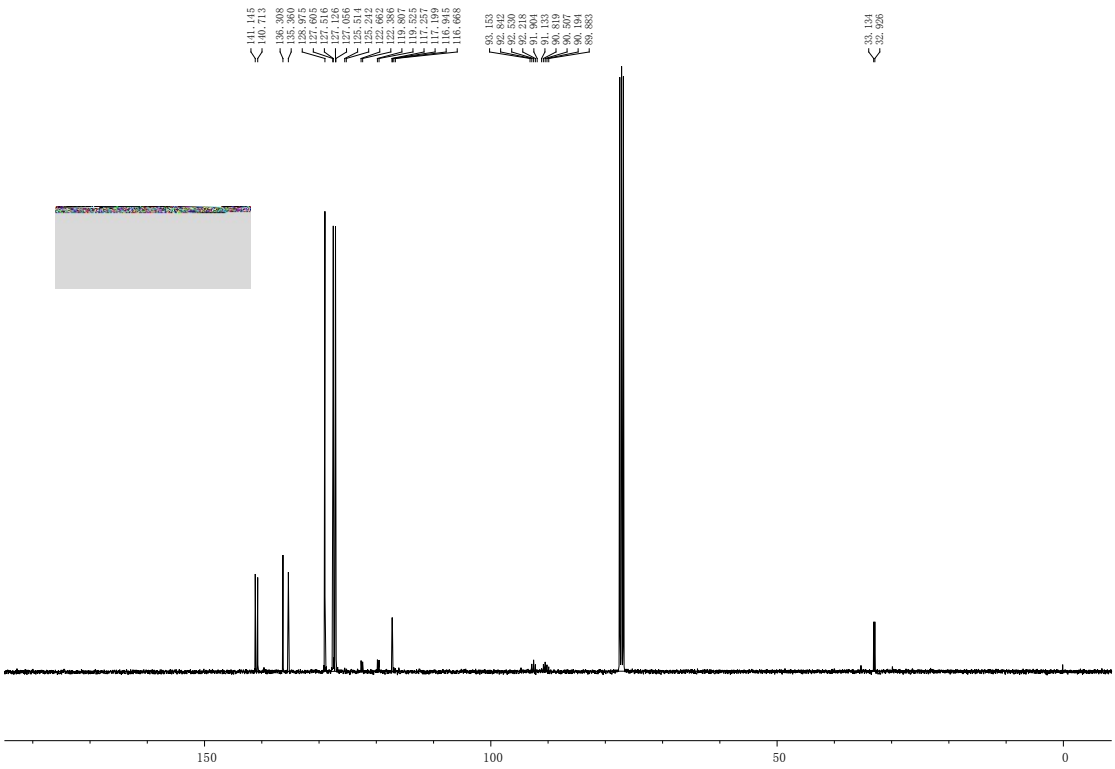


^{19}F NMR spectrum for 2c: (*E*)-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)-1,1'-biphenyl

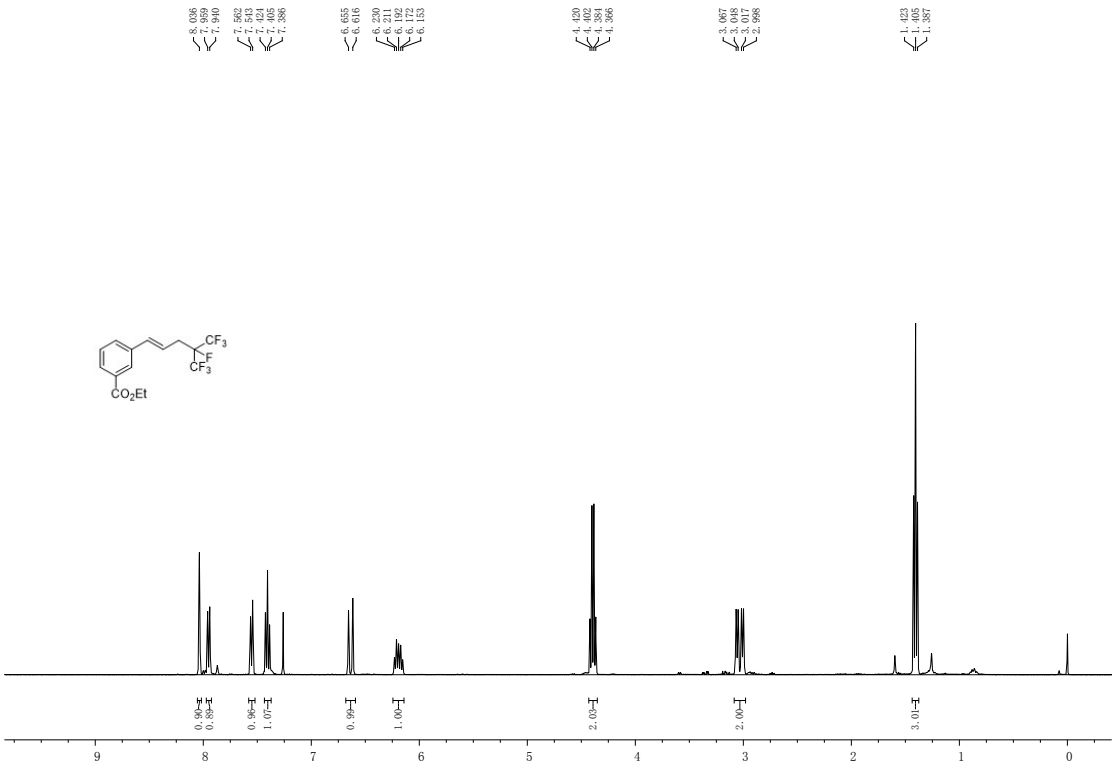


^{13}C NMR spectrum for 2c: (*E*)-4-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)-1,1'-

biphenyl

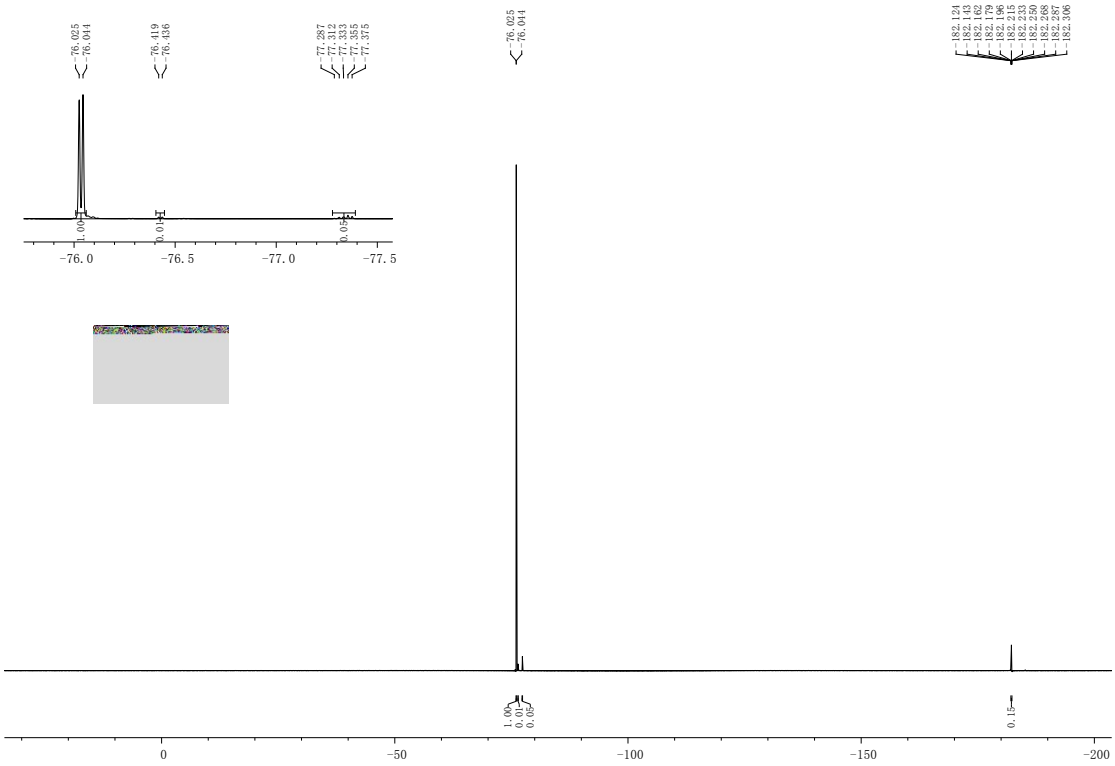


^{1}H NMR spectrum for 2d: (*E*)-ethyl 3-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzoate

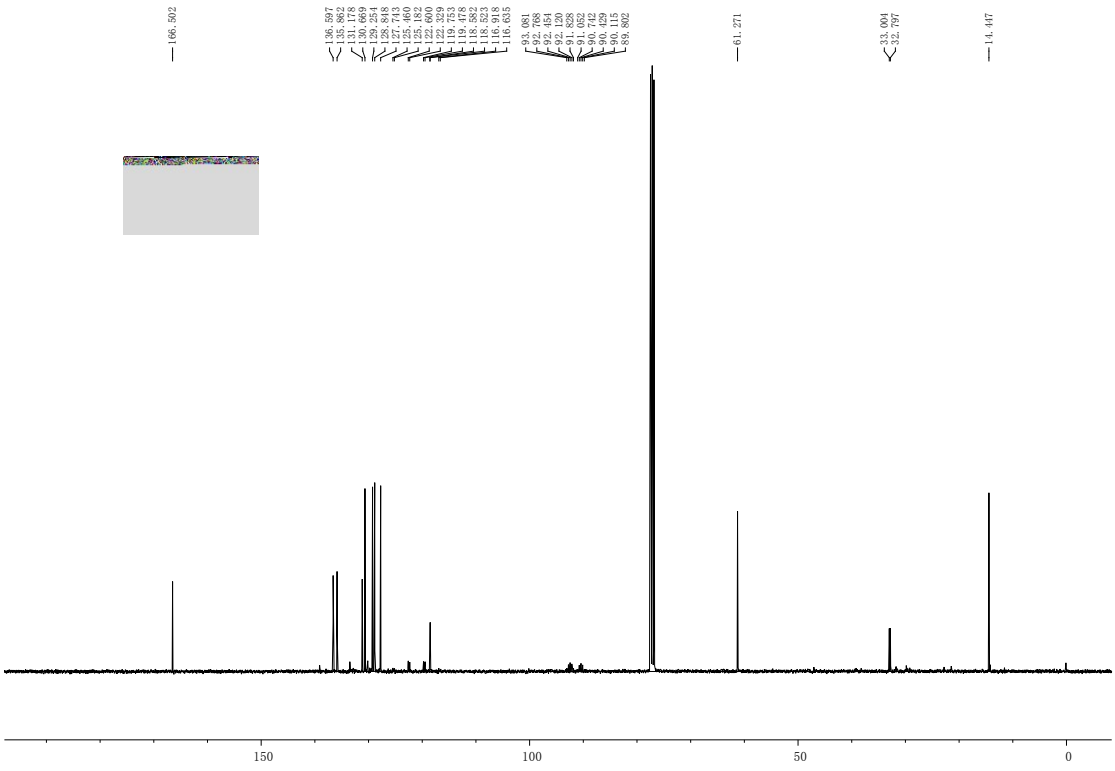


^{19}F NMR spectrum for 2d: (*E*)-ethyl 3-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-

yl)benzoate

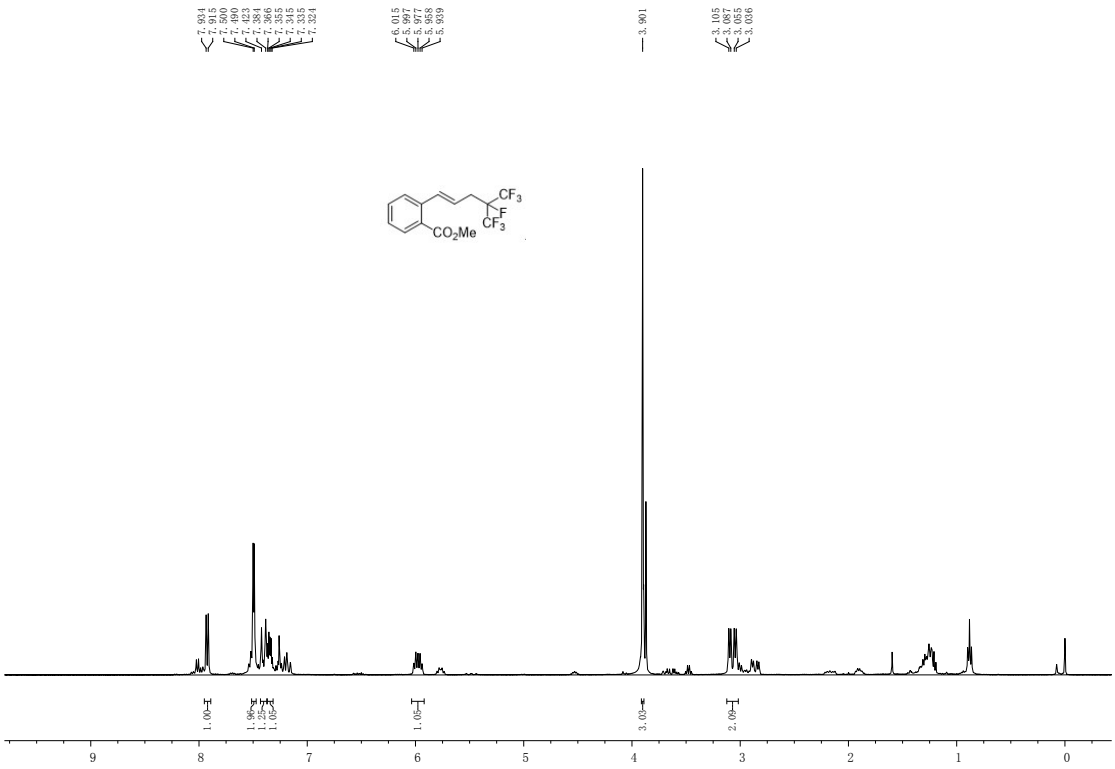


^{13}C NMR spectrum for 2d: (*E*)-ethyl 3-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzoate

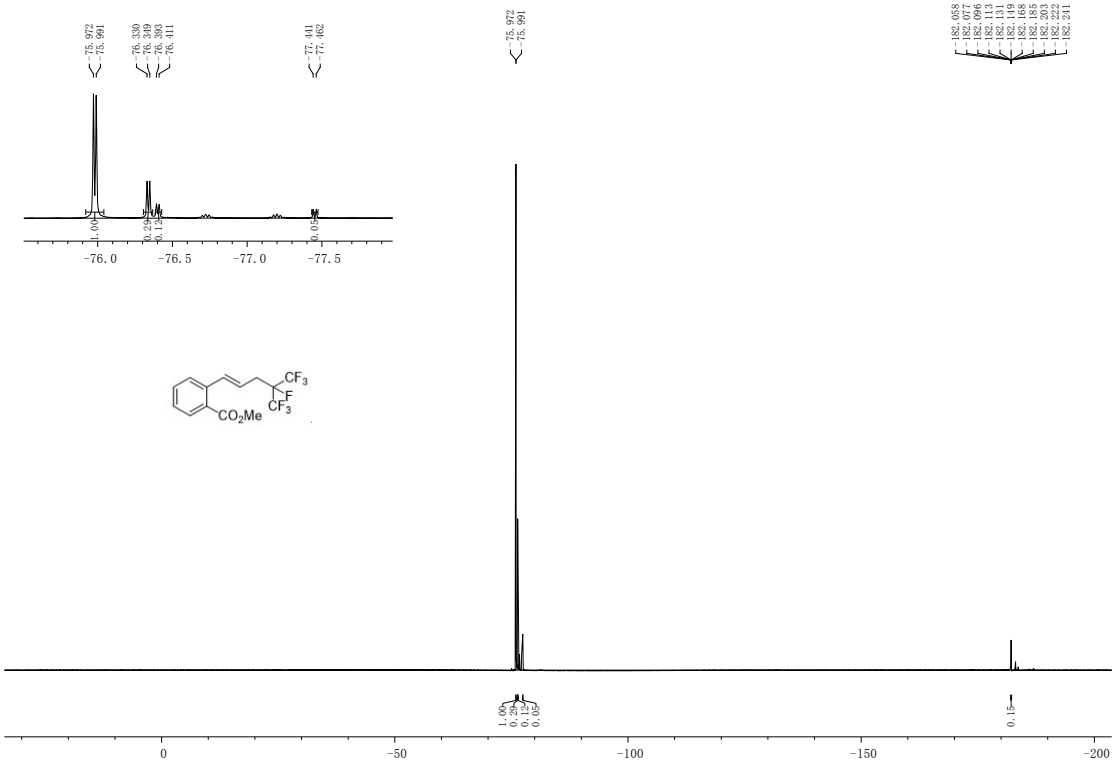


^{1}H NMR spectrum for 2e: (*E*)-methyl 2-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-

yl)benzoate

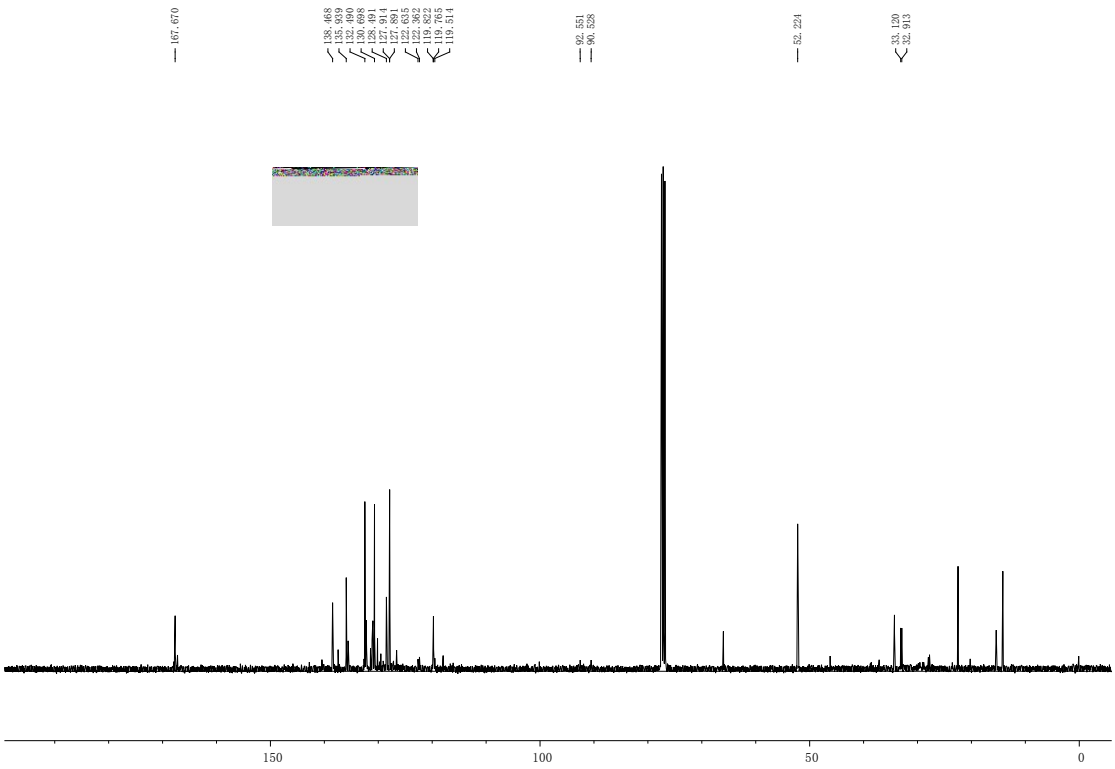


^{19}F NMR spectrum for 2e: (*E*)-methyl 2-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)benzoate

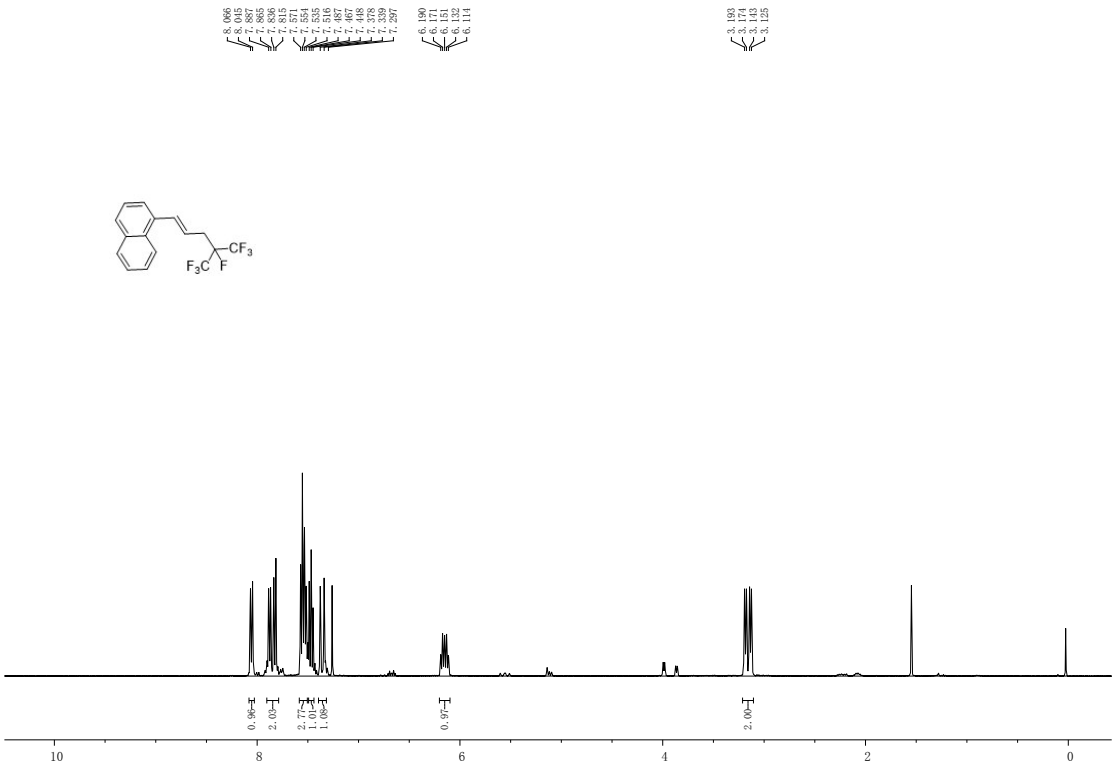


^{13}C NMR spectrum for 2e: (*E*)-methyl 2-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-

yl)benzoate

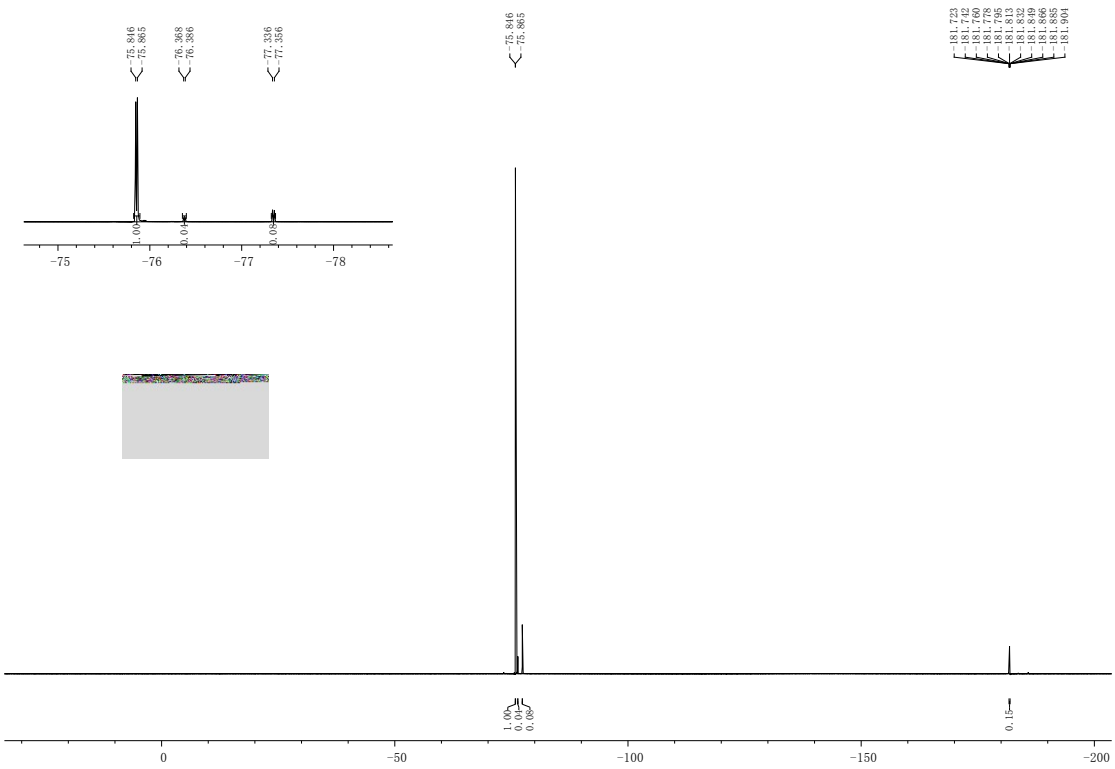


^{1}H NMR spectrum for 2f: (*E*)-1-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)naphthalene

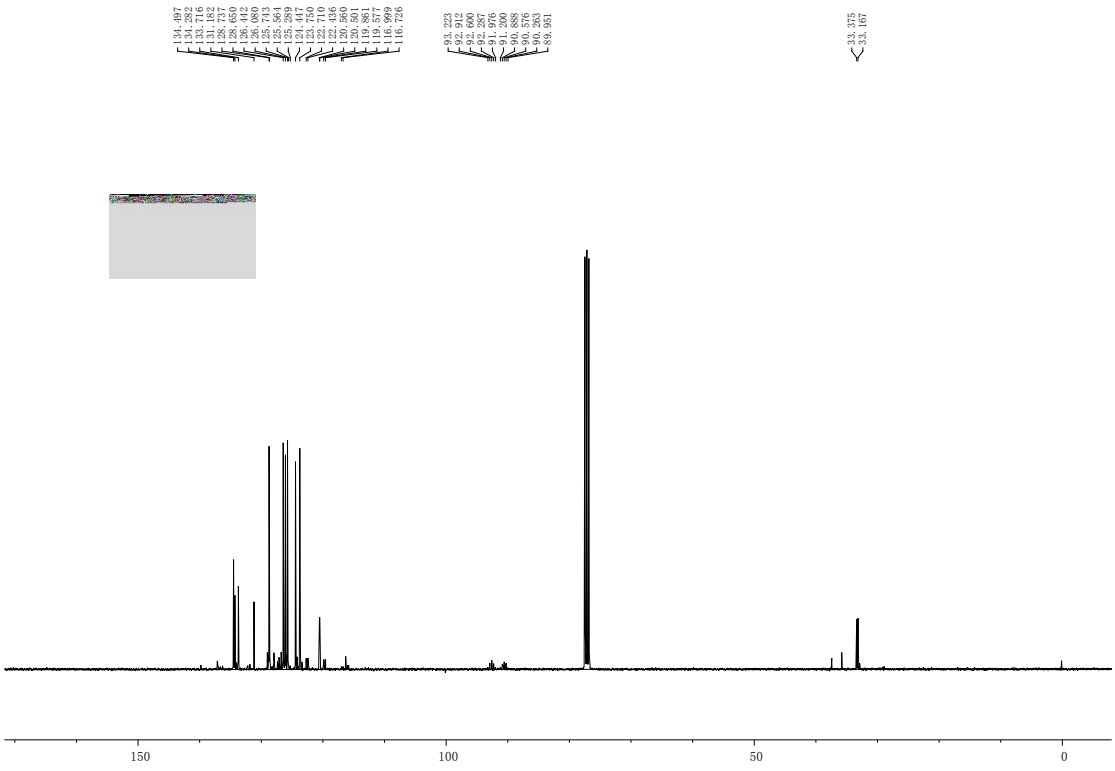


^{19}F NMR spectrum for 2f: (*E*)-1-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-

yl)naphthalene

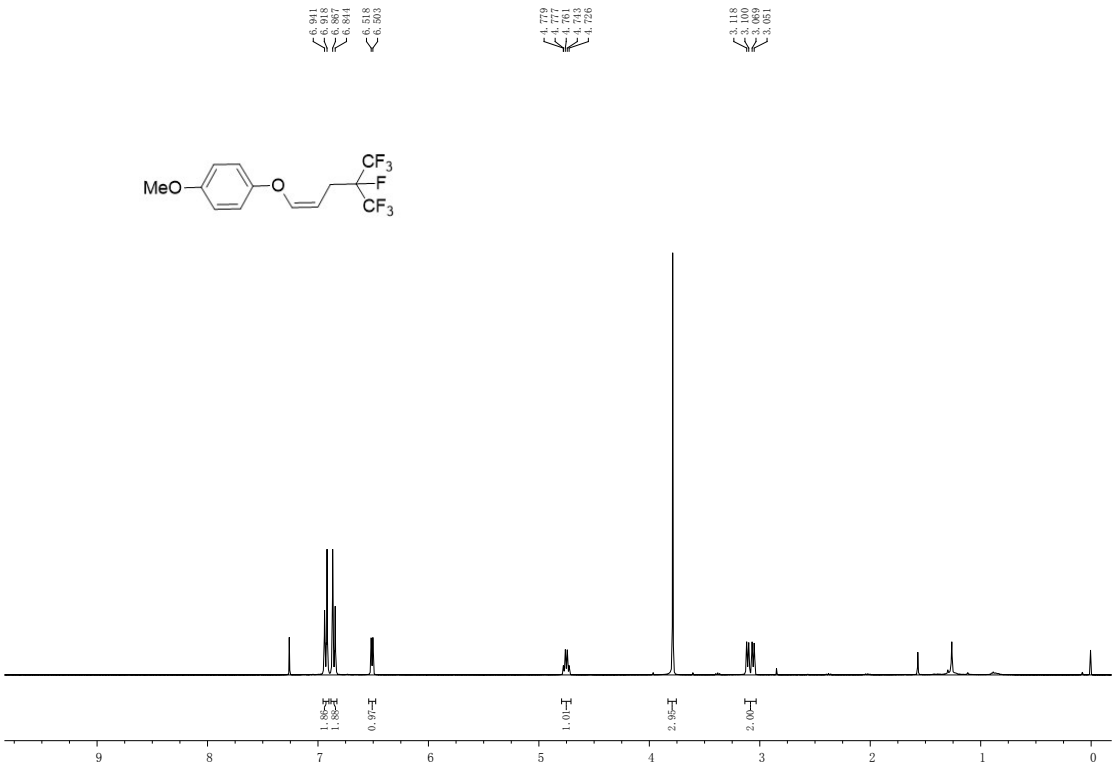


^{13}C NMR spectrum for 2f: (*E*)-1-(4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)naphthalene

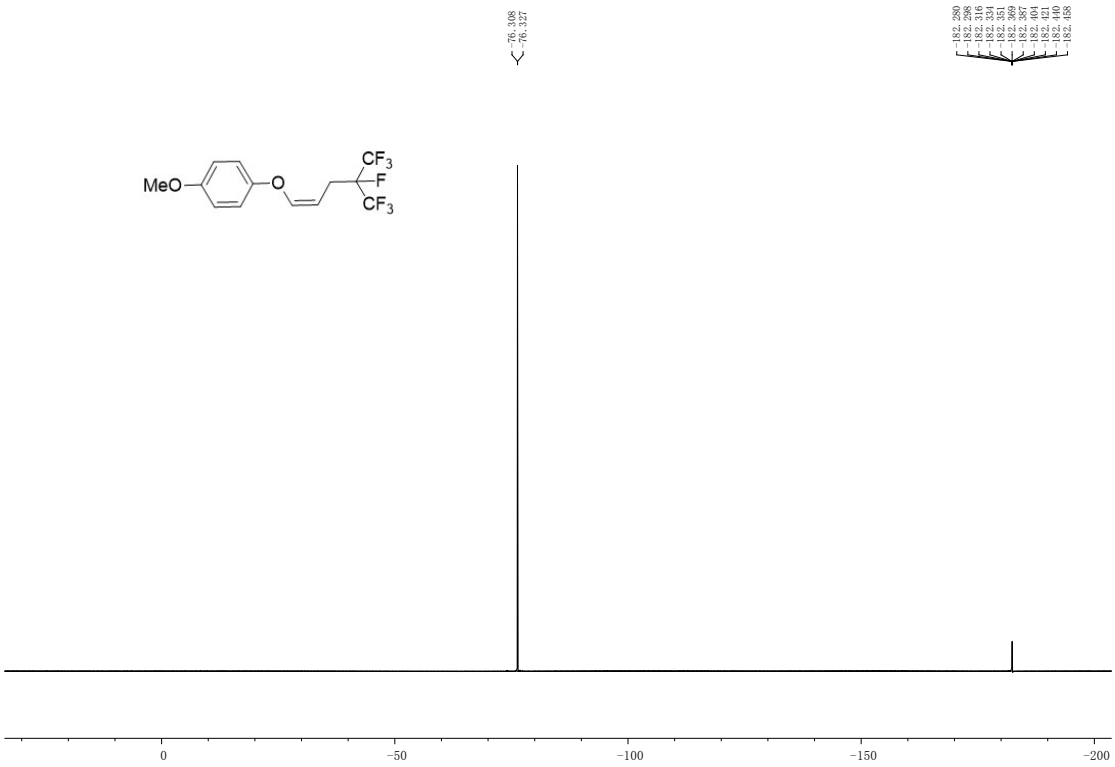


^{1}H NMR spectrum for 2g: (*Z*)-1-methoxy-4-((4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-

en-1-yl)oxy)benzene

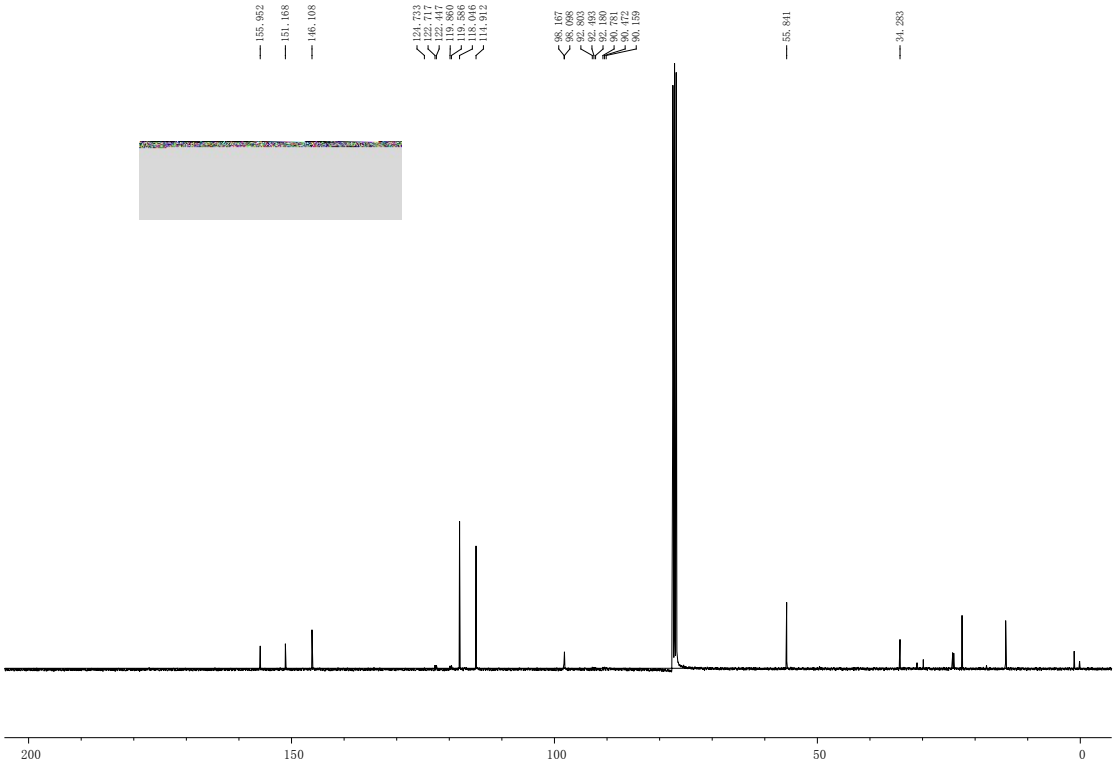


^{19}F NMR spectrum for 2g: (*Z*)-1-methoxy-4-((4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-en-1-yl)oxy)benzene

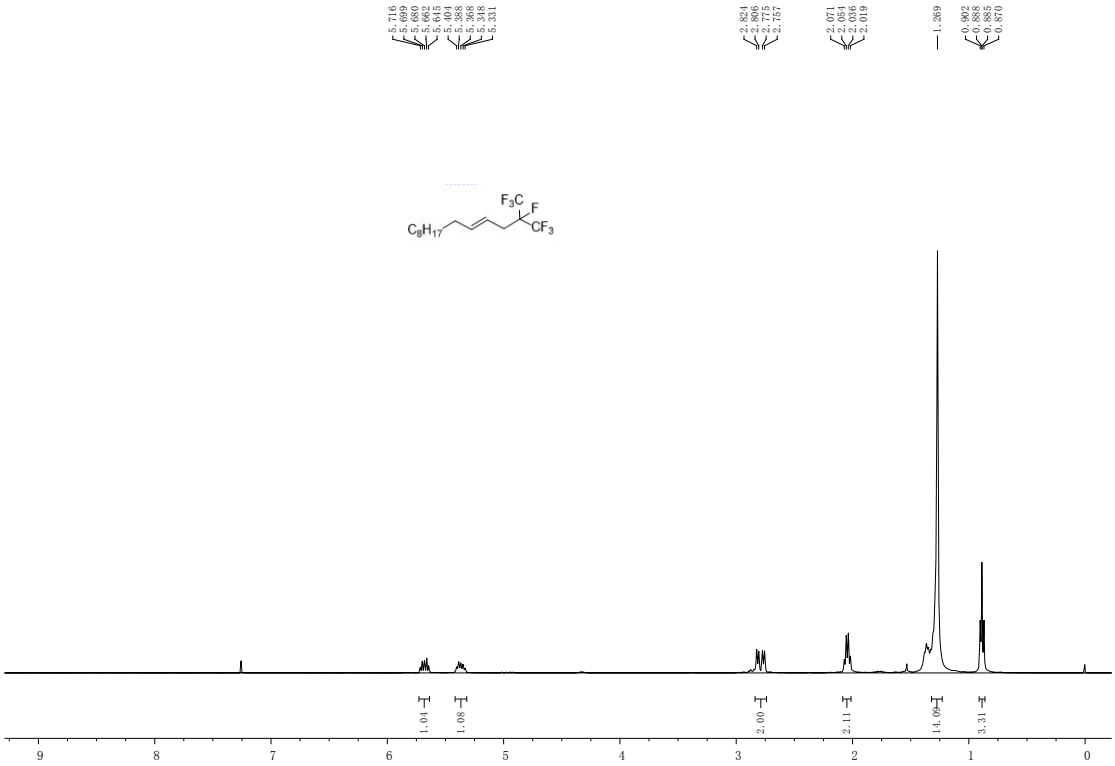


^{13}C NMR spectrum for 2g: (*Z*)-1-methoxy-4-((4,5,5,5-tetrafluoro-4-(trifluoromethyl)pent-1-

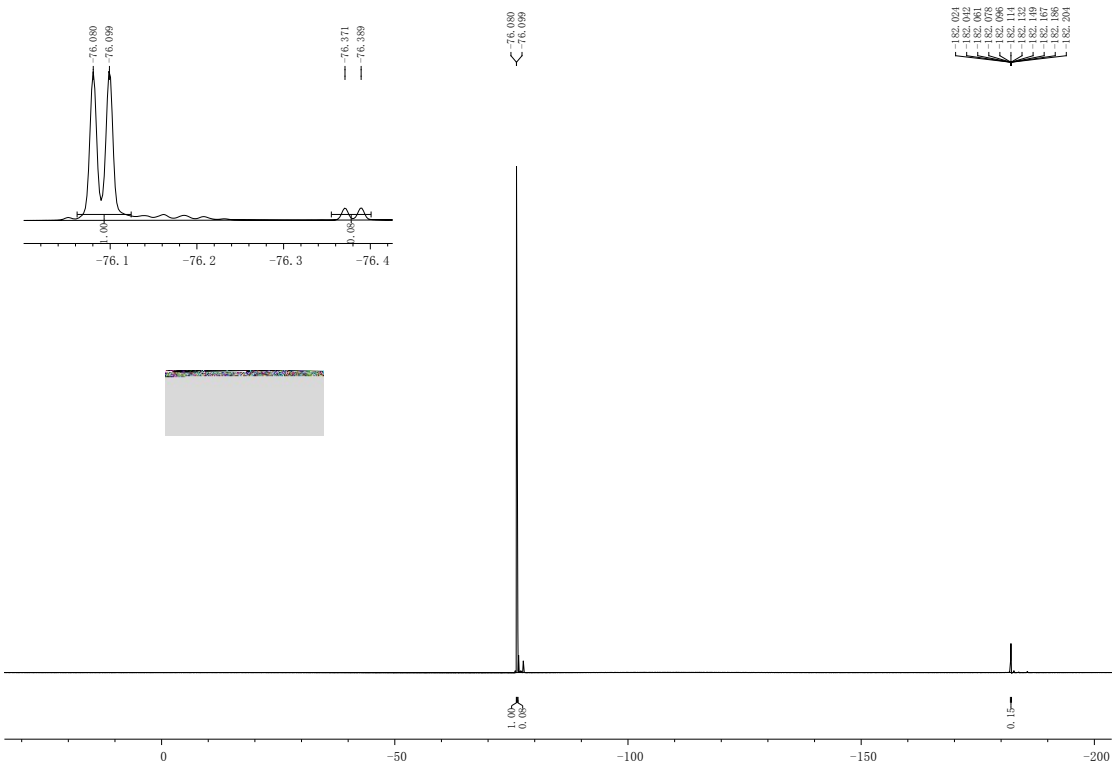
en-1-yl)oxy)benzene



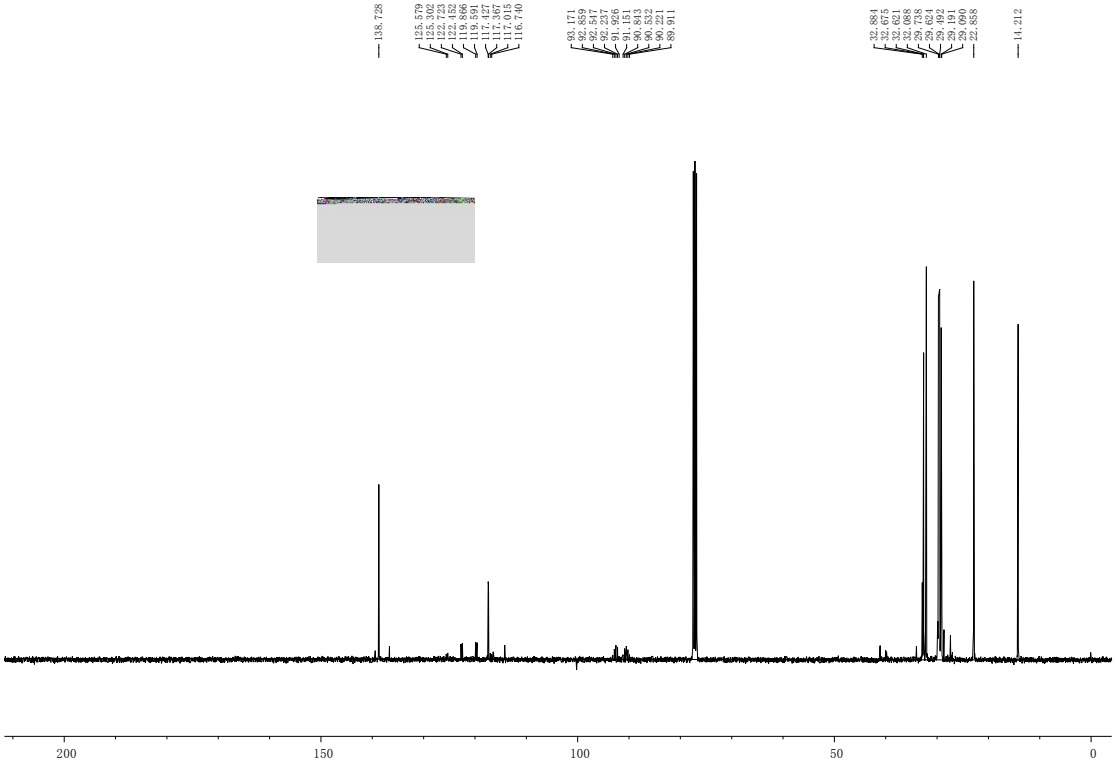
^{1}H NMR spectrum for 2h: (*E*)-1,1,1,2-tetrafluoro-2-(trifluoromethyl)tetradec-4-ene



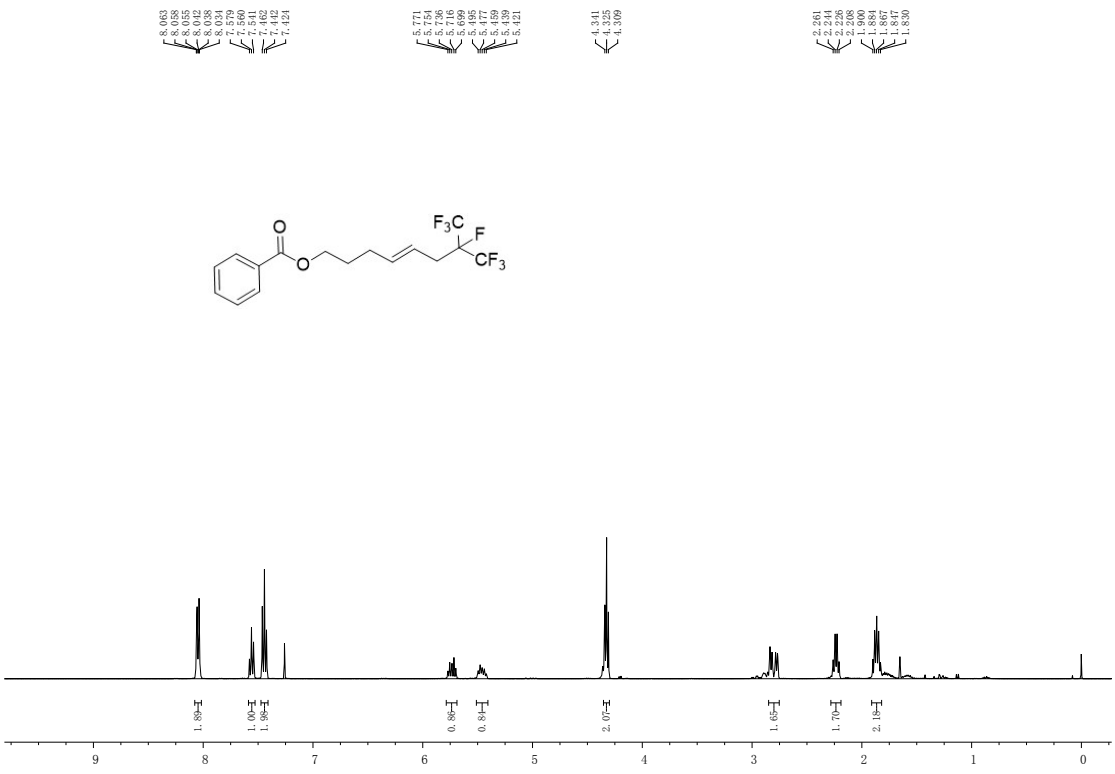
^{19}F NMR spectrum for 2h: (*E*)-1,1,1,2-tetrafluoro-2-(trifluoromethyl)tetradec-4-ene



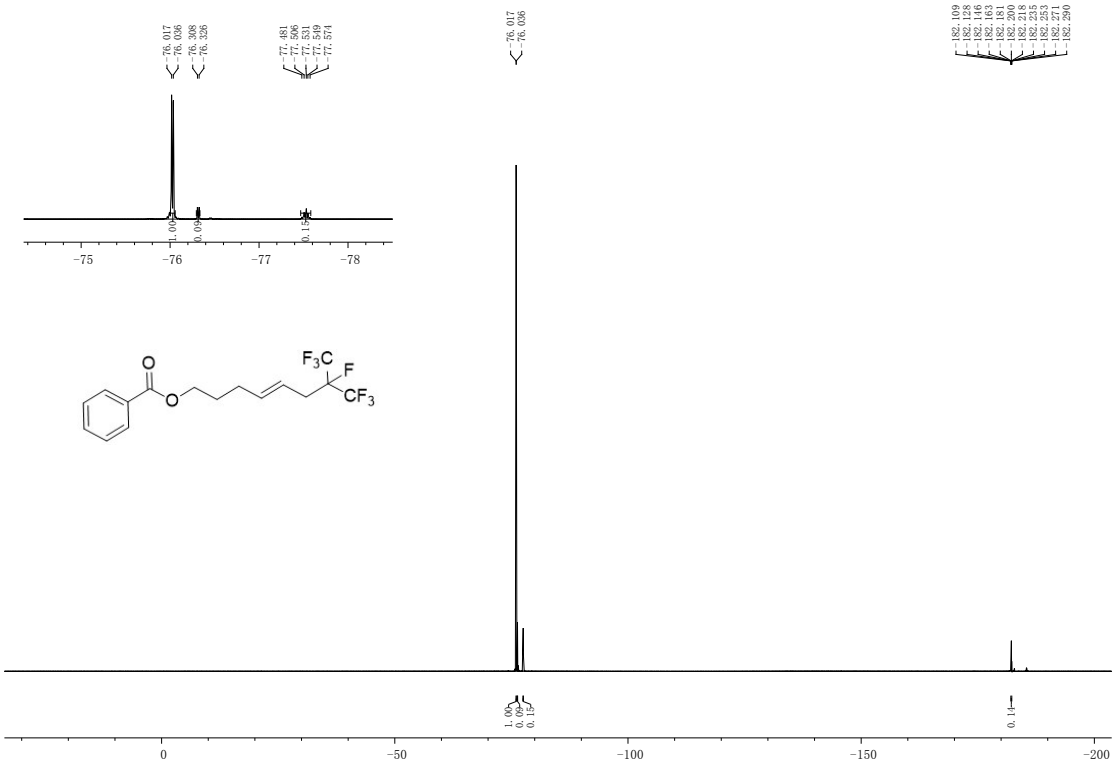
^{13}C NMR spectrum for 2h: (*E*)-1,1,1,2-tetrafluoro-2-(trifluoromethyl)tetradec-4-ene



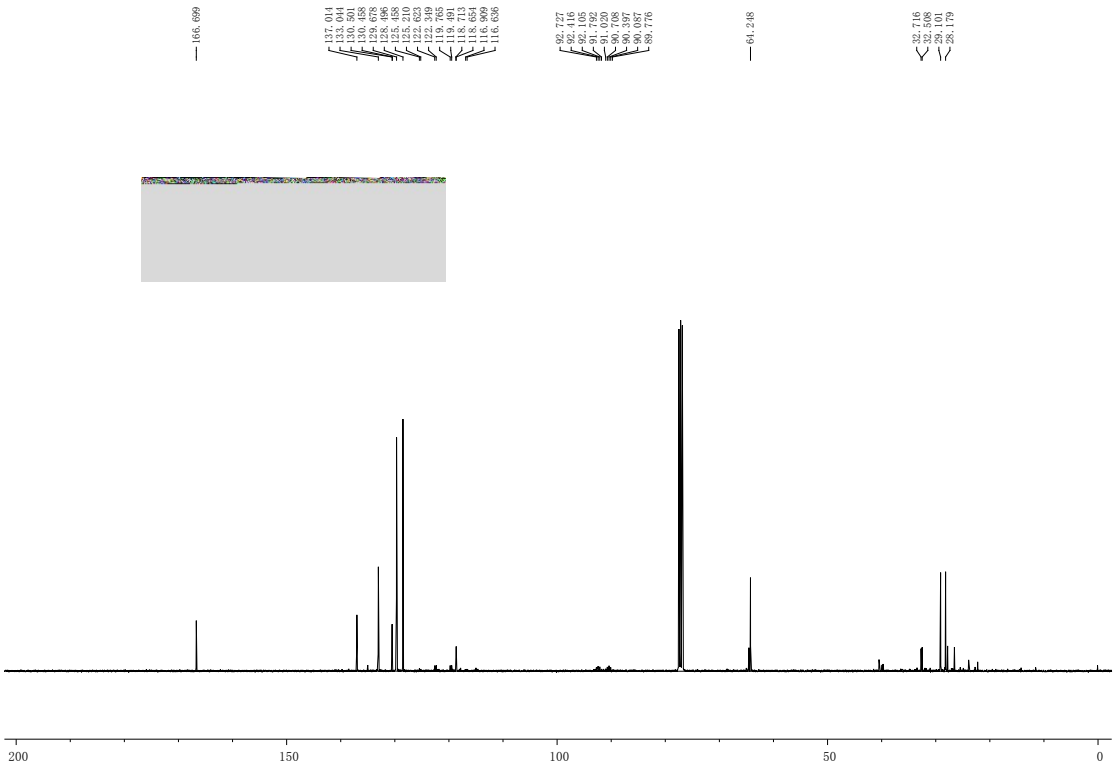
^{1}H NMR spectrum for 2i: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzoate



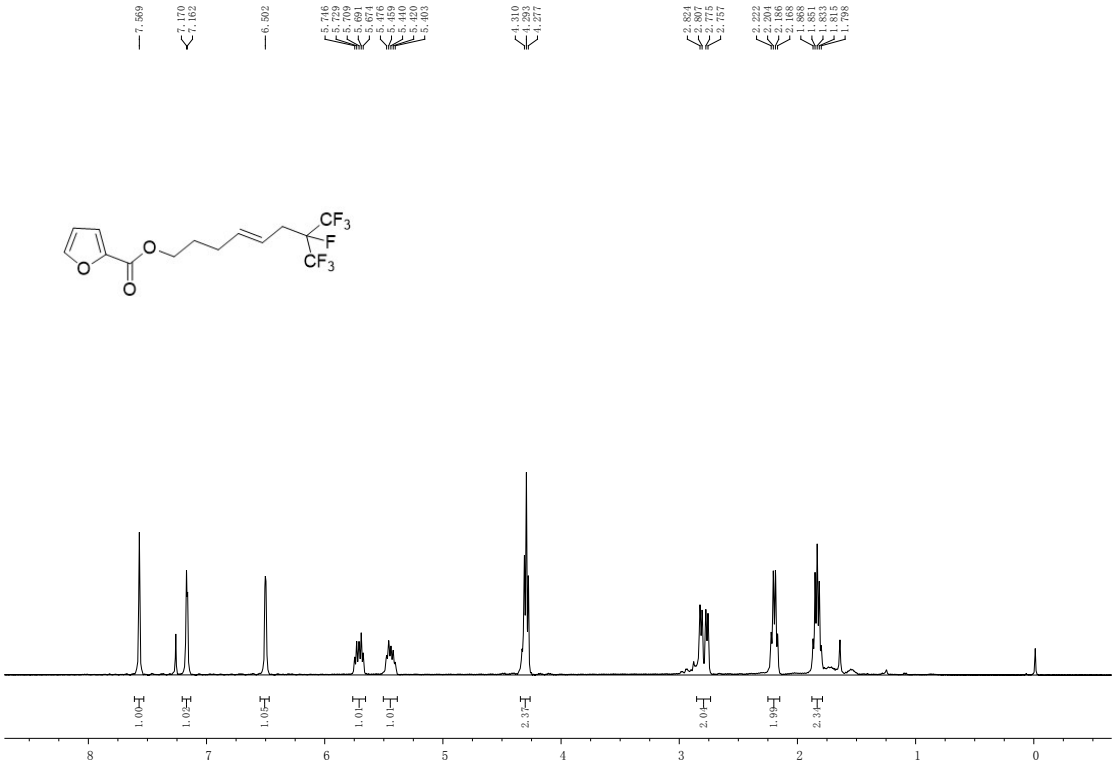
^{19}F NMR spectrum for 2i: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzoate



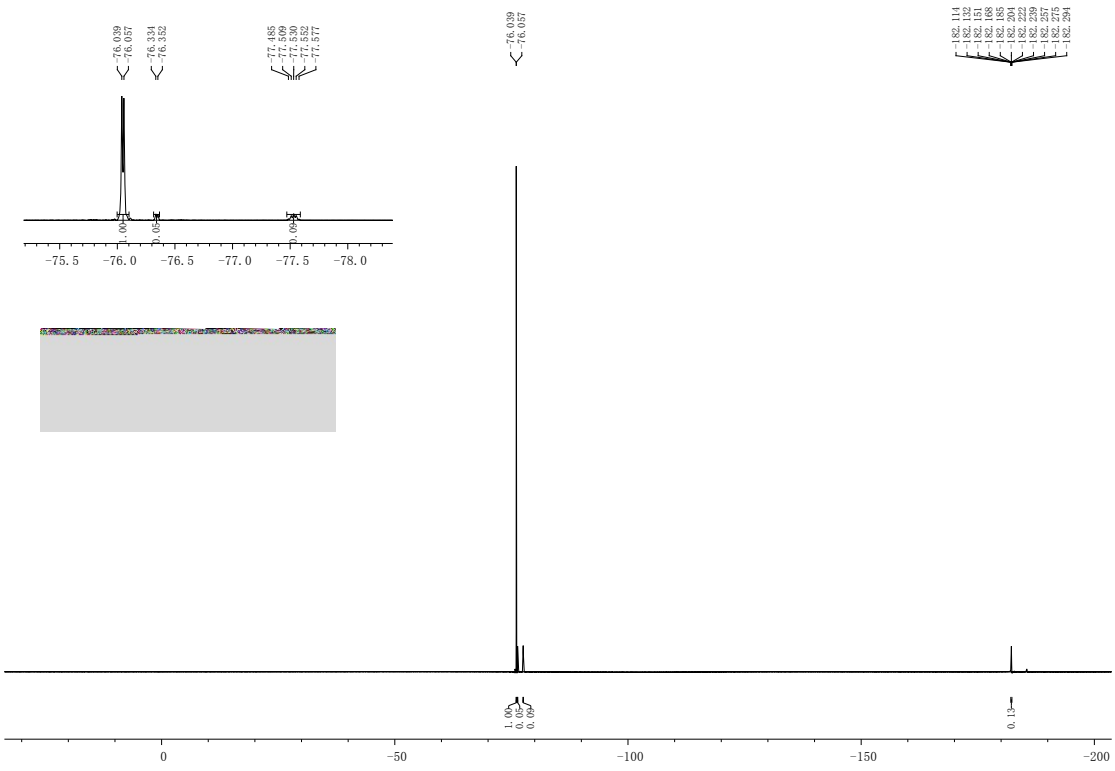
^{13}C NMR spectrum for 2i: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzoate



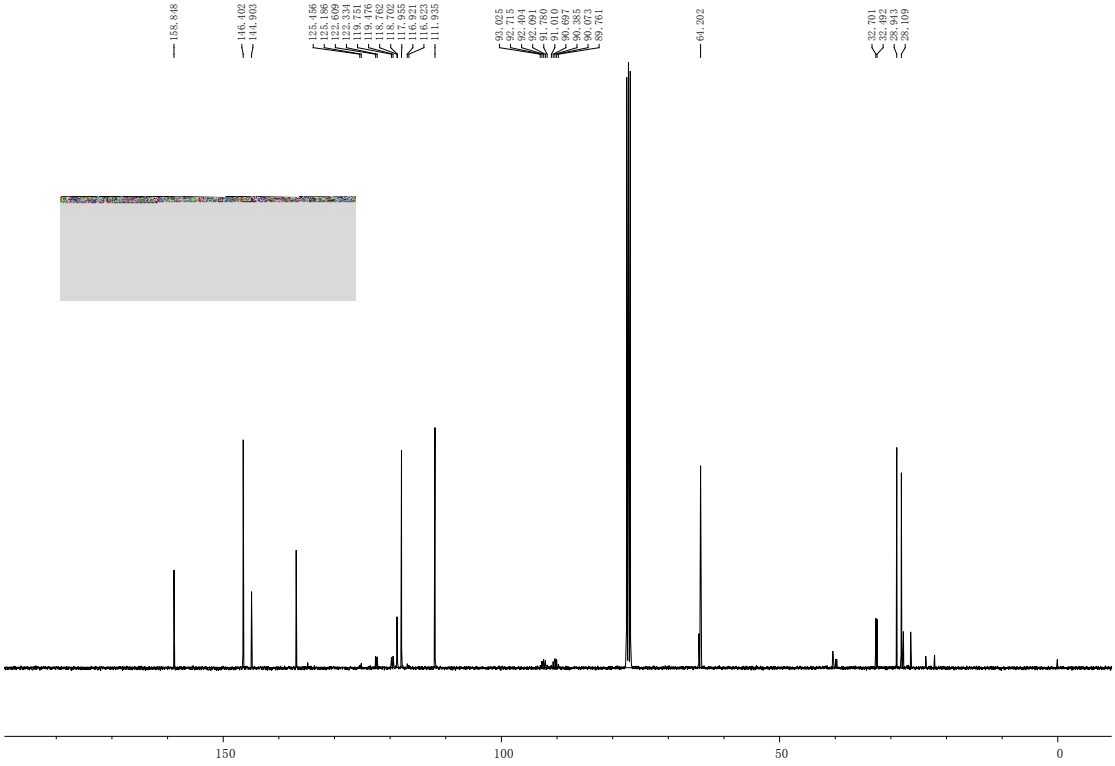
^{1}H NMR spectrum for 2j: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl furan-2-carboxylate



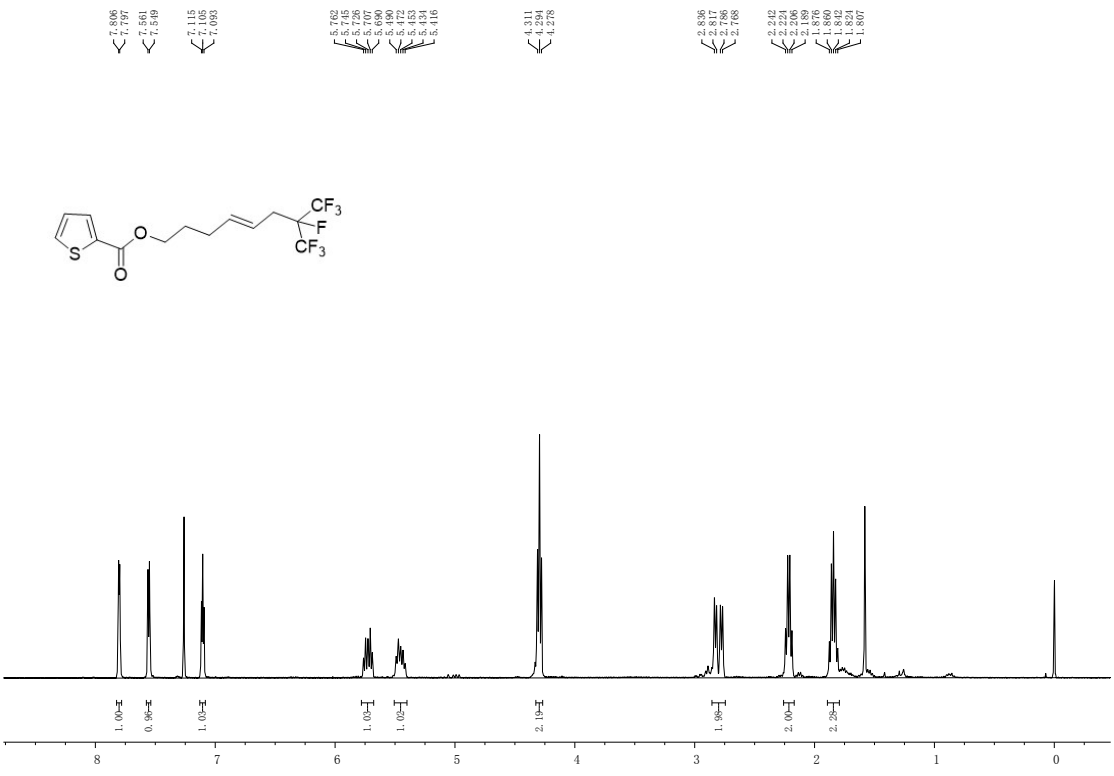
^{19}F NMR spectrum for 2j: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl furan-2-carboxylate



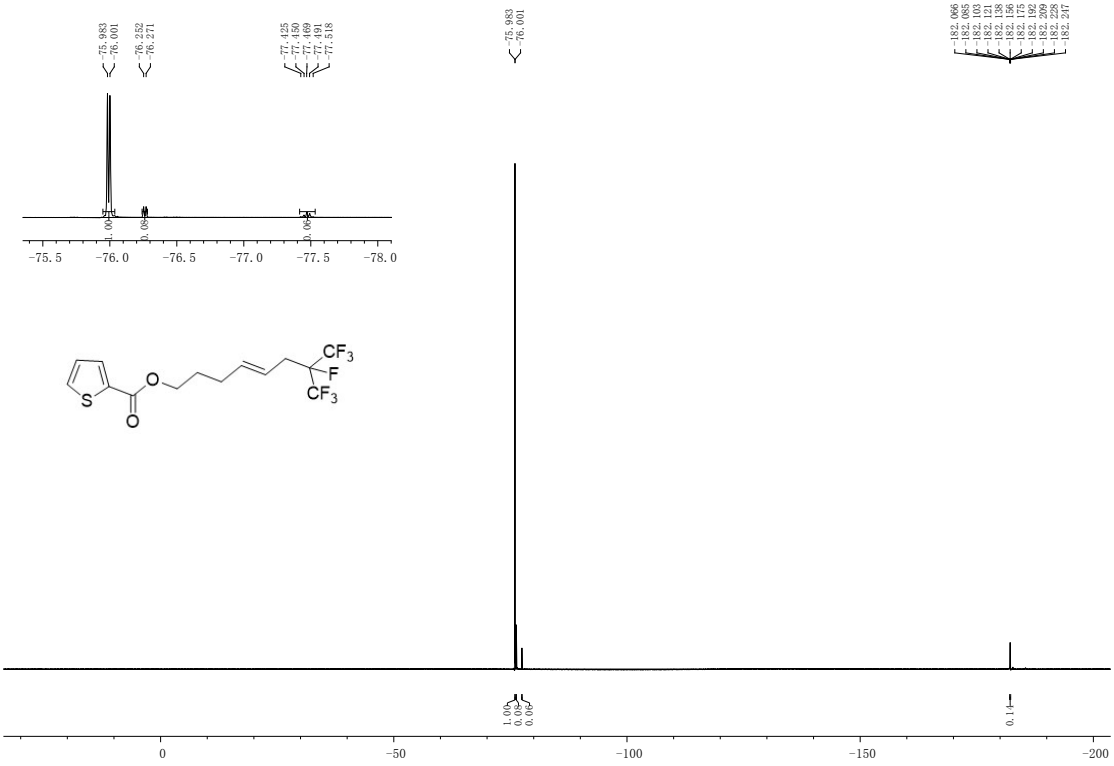
^{13}C NMR spectrum for 2j: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl furan-2-carboxylate



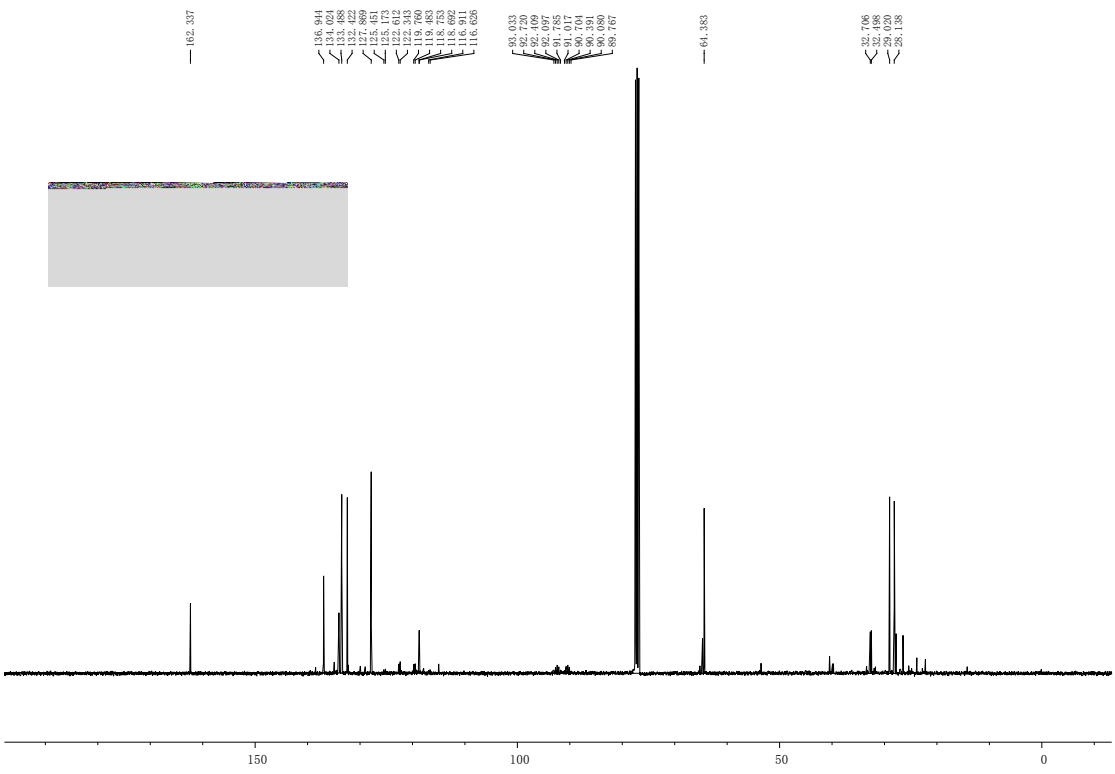
^{1}H NMR spectrum for 2k: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl thiophene-2-carboxylate



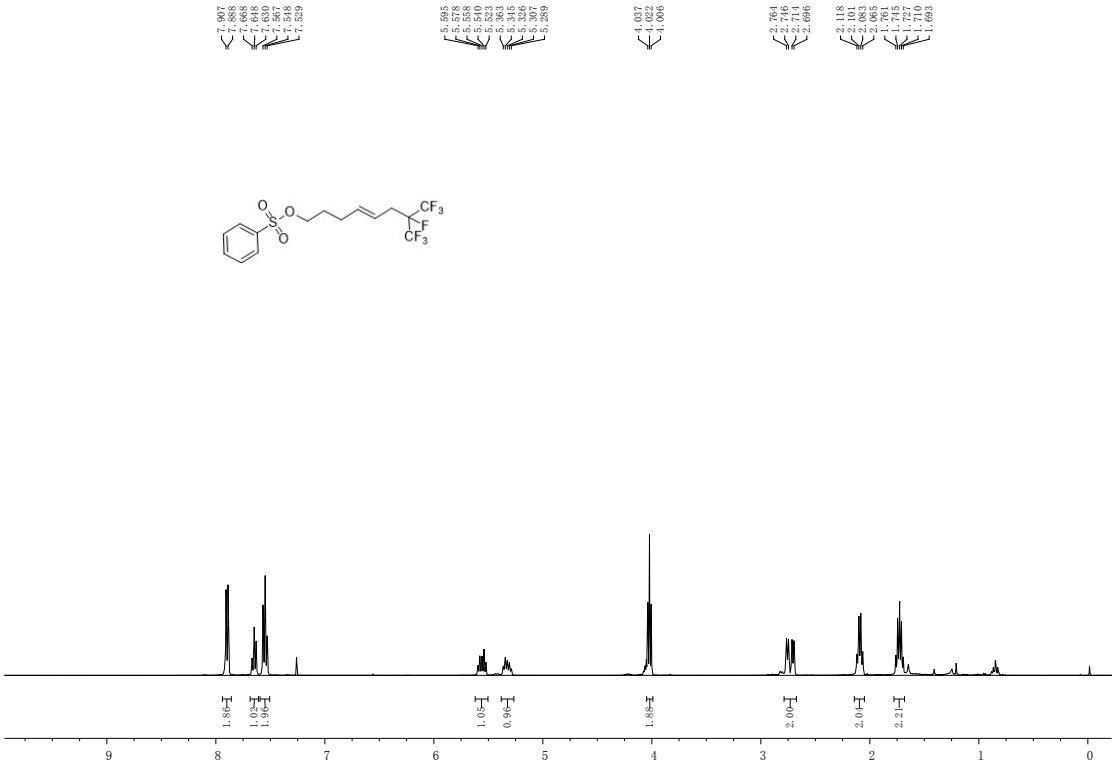
^{19}F NMR spectrum for 2k: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl thiophene-2-carboxylate



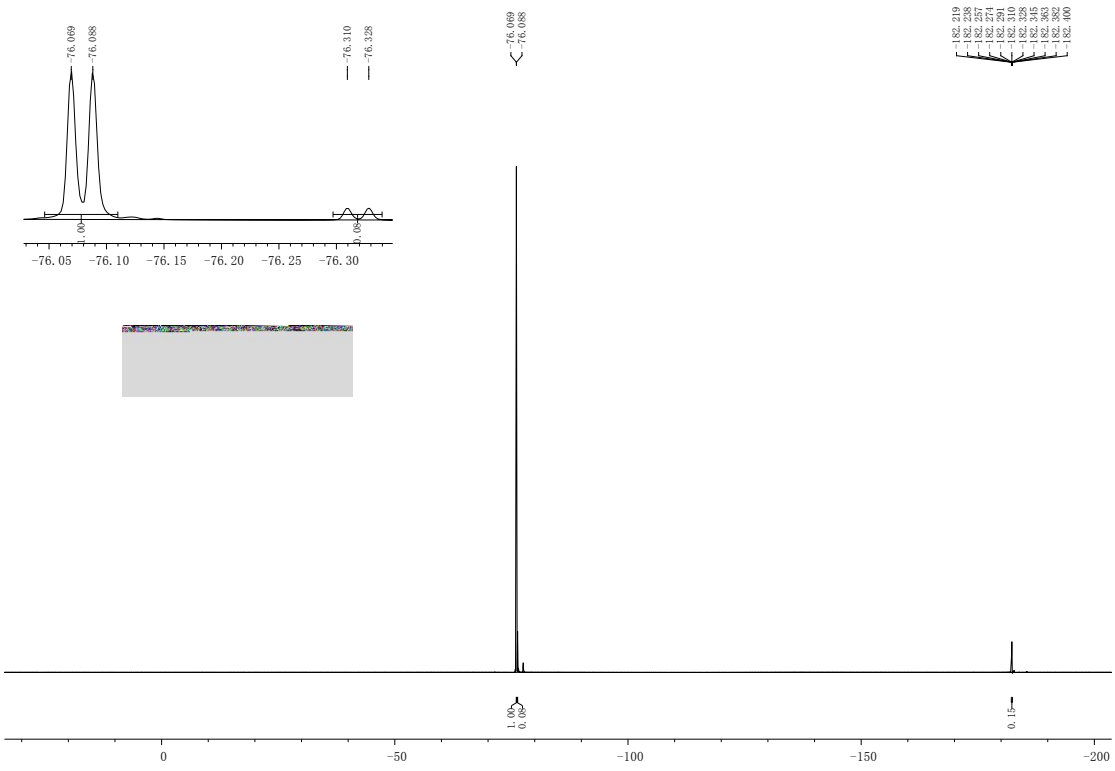
^{13}C NMR spectrum for 2k: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl thiophene-2-carboxylate



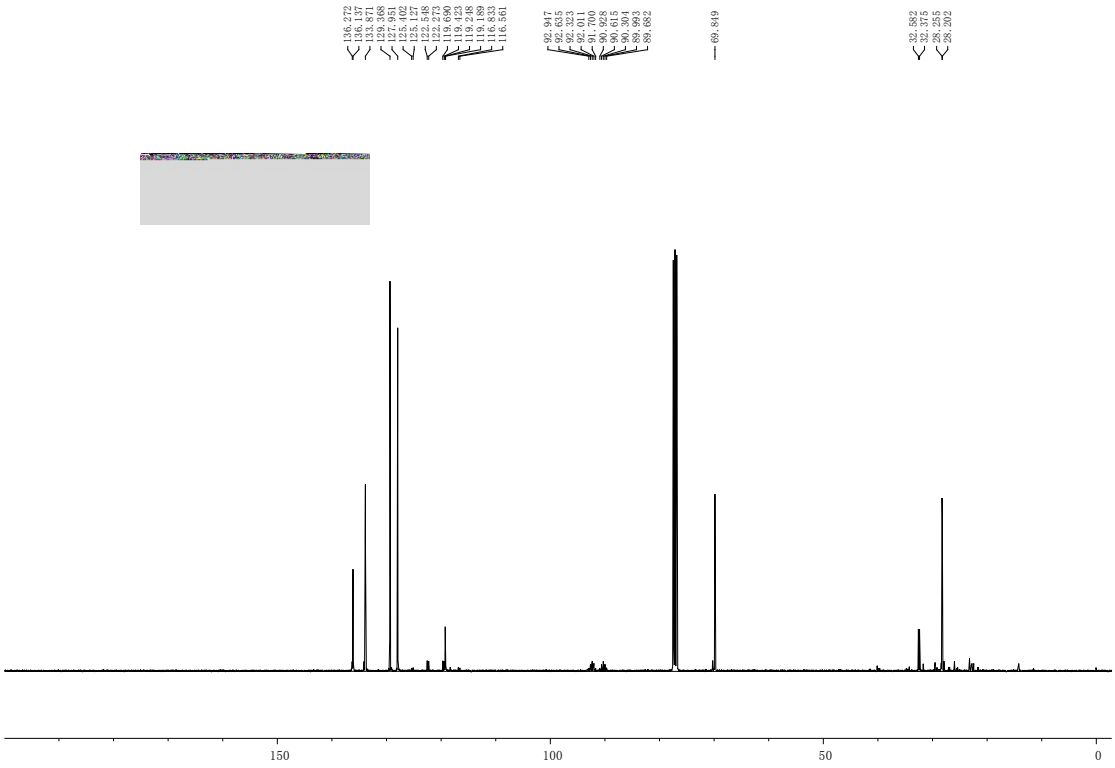
^{1}H NMR spectrum for 2l: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzenesulfonate



^{19}F NMR spectrum for 2l: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzenesulfonate

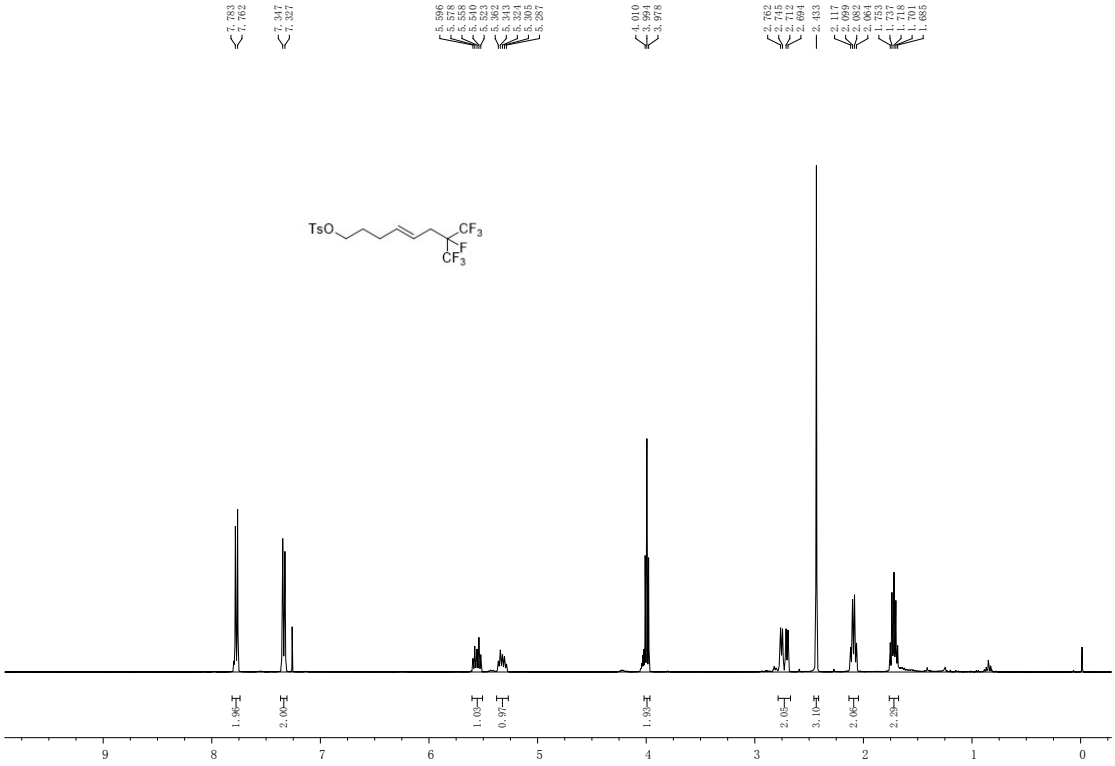


^{13}C NMR spectrum for 2l: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl benzenesulfonate

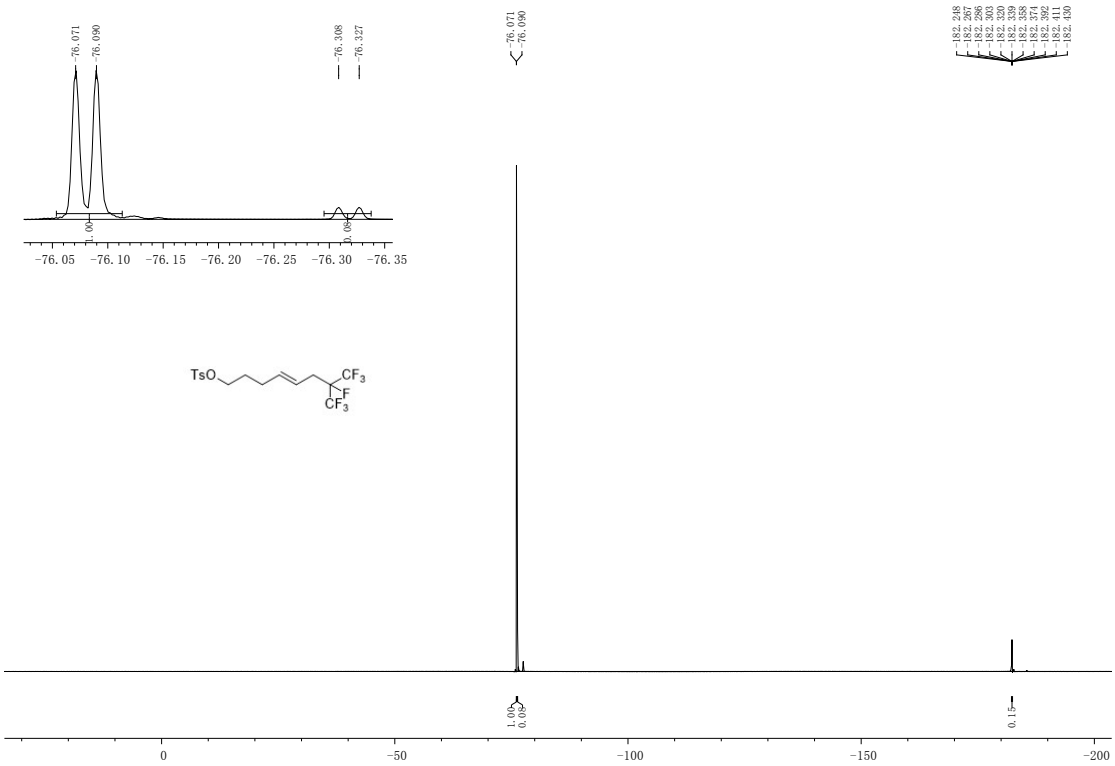


^{1}H NMR spectrum for 2m: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl 4-

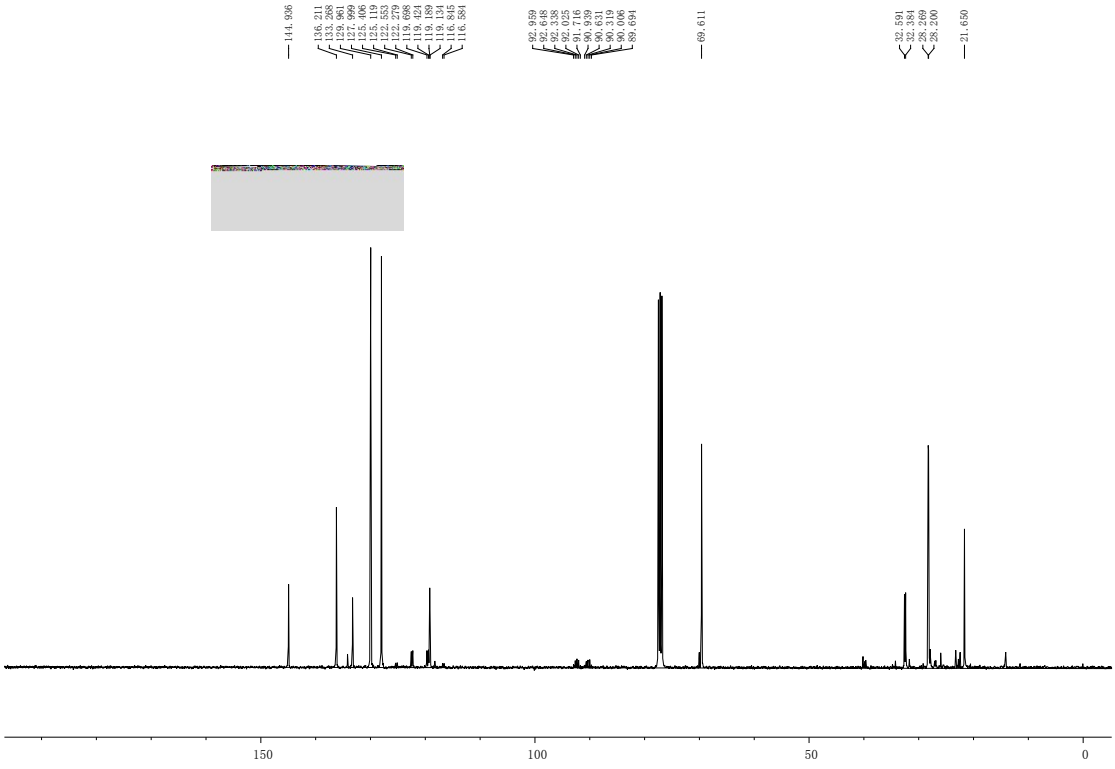
methylbenzenesulfonate



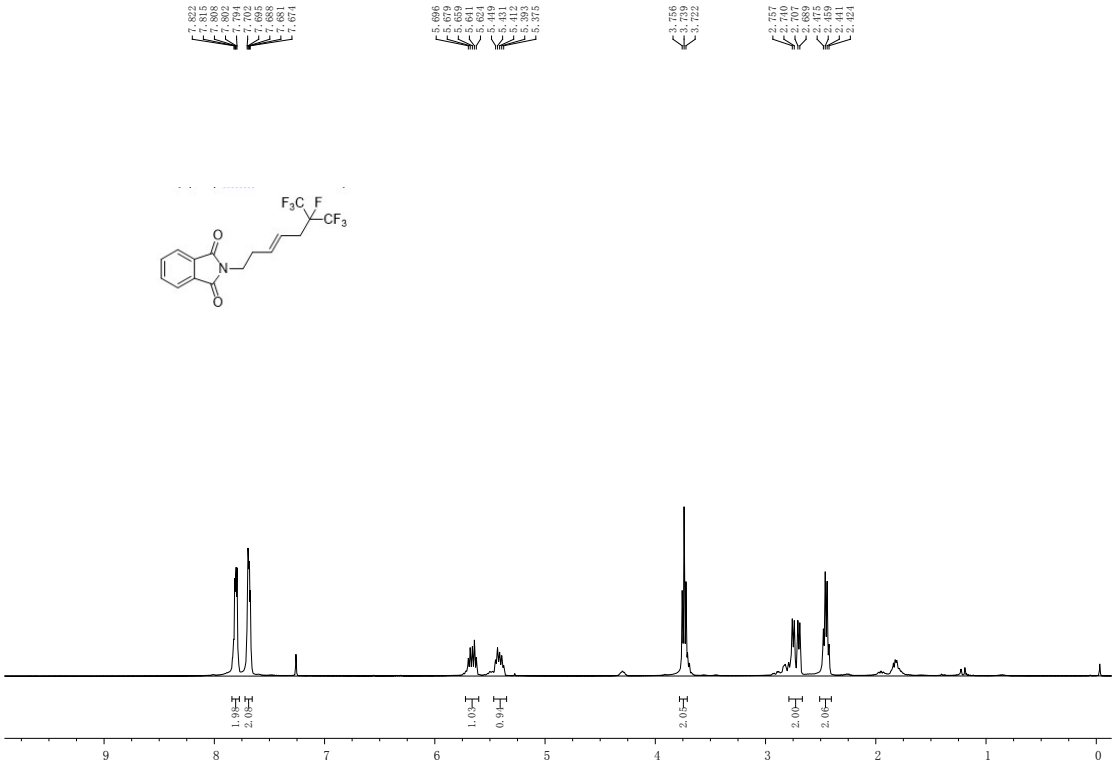
^{19}F NMR spectrum for 2m: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl 4-methylbenzenesulfonate



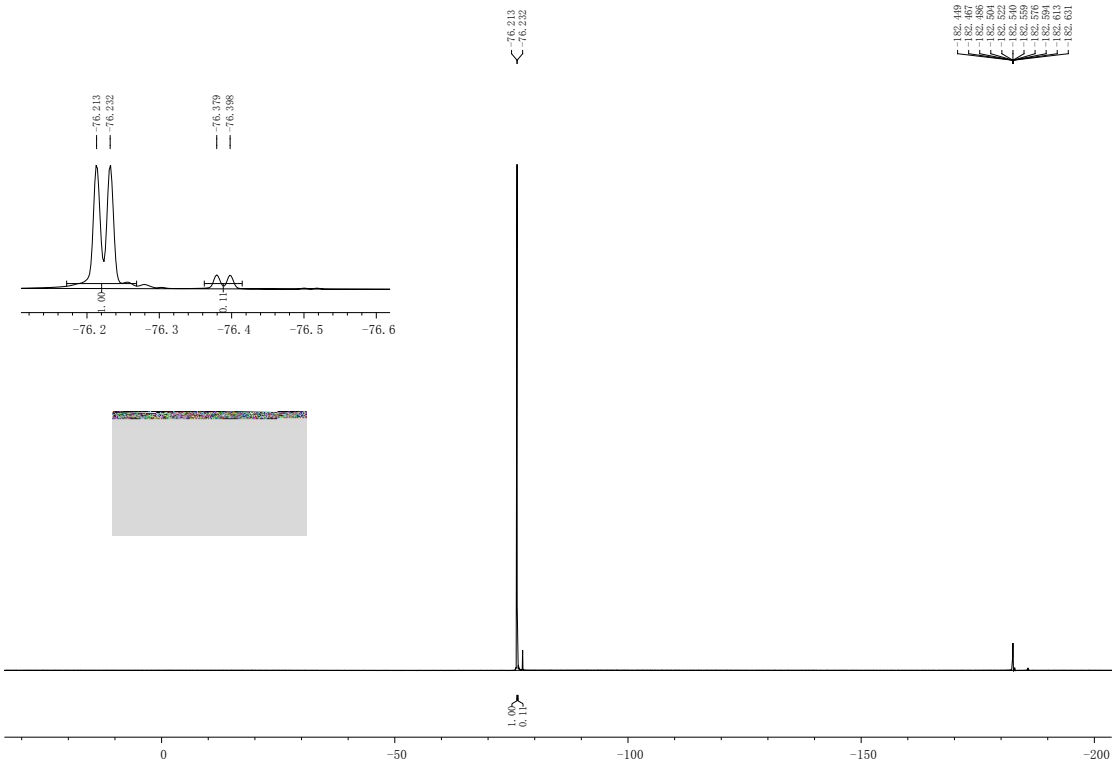
^{13}C NMR spectrum for 2m: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-yl 4-methylbenzenesulfonate



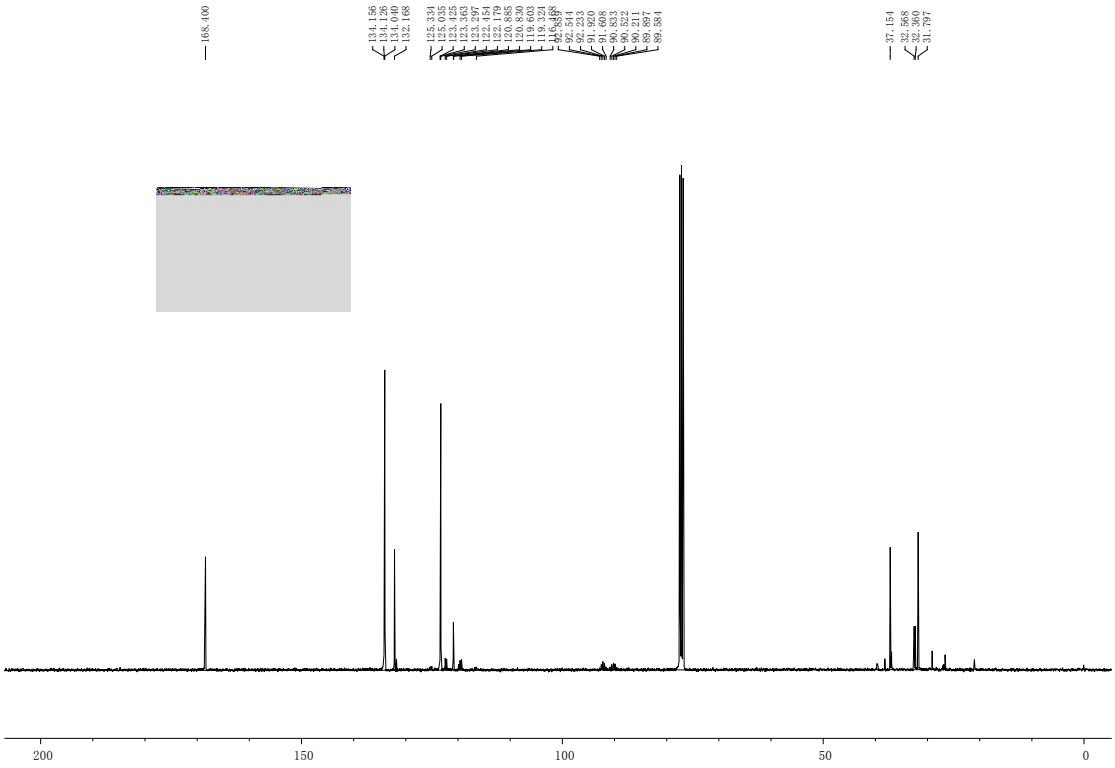
^{1}H NMR spectrum for 2n: (*E*)-2-(6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)isoindoline-1,3-dione



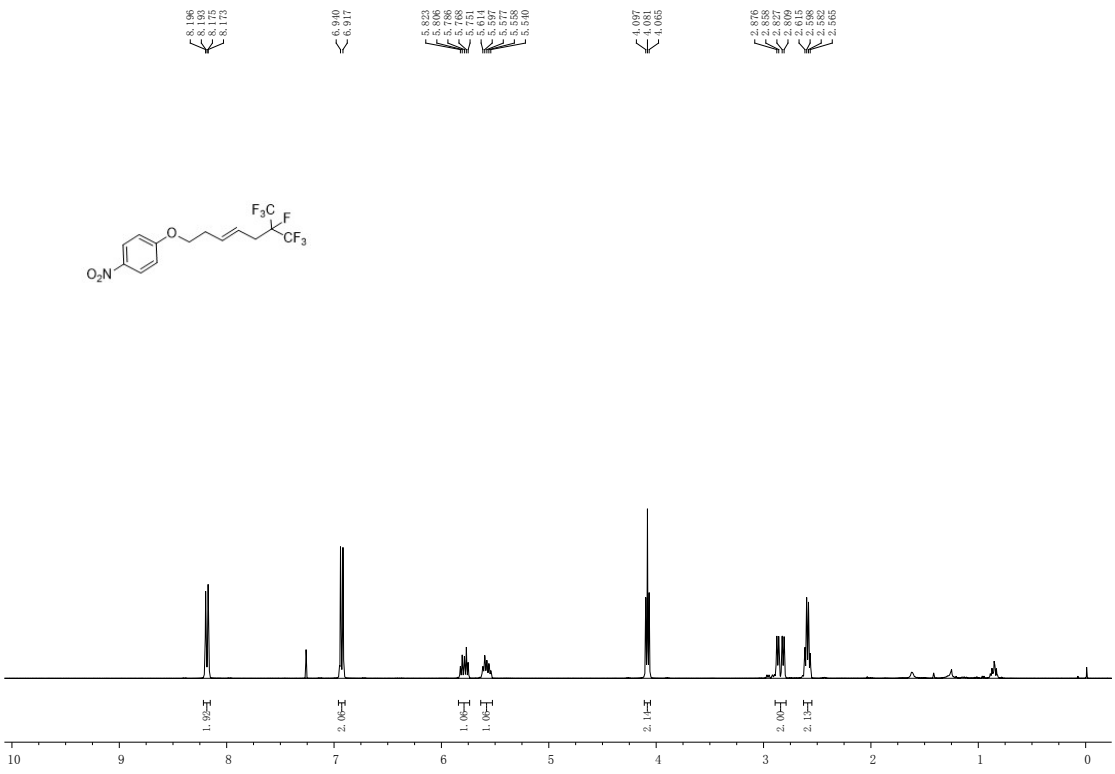
^{19}F NMR spectrum for 2n: (*E*)-2-(6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)isoindoline-1,3-dione



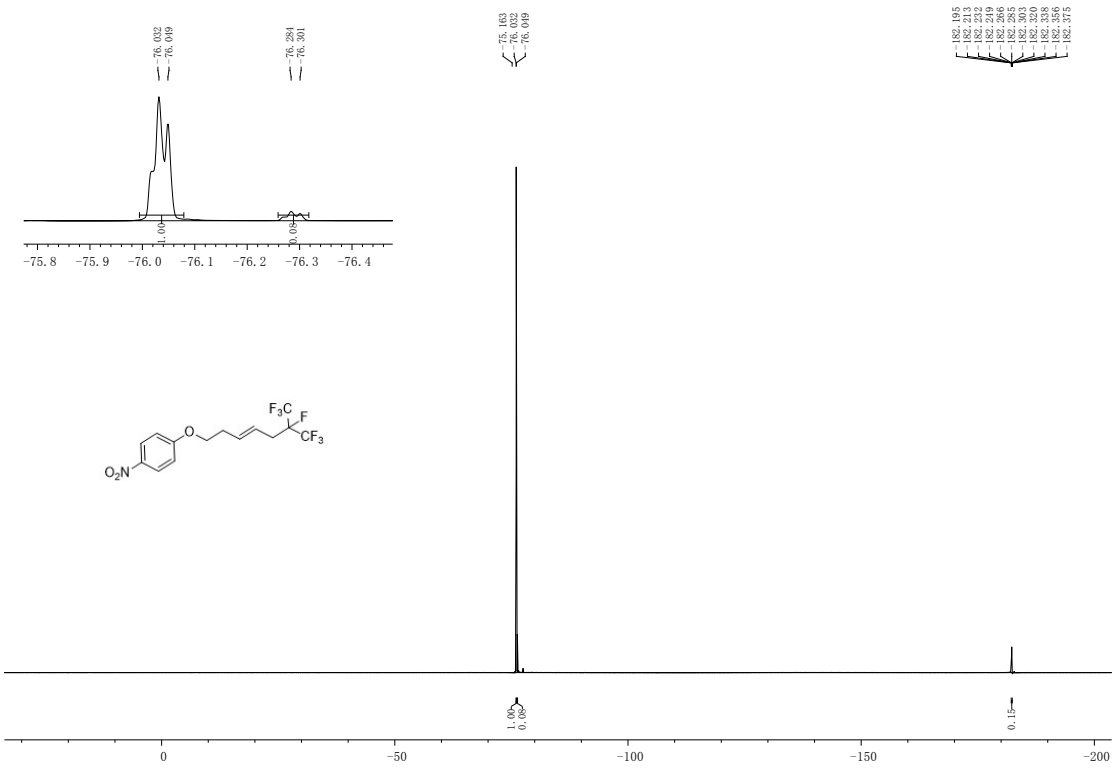
^{13}C NMR spectrum for 2n: (*E*)-2-(6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)isoindoline-1,3-dione



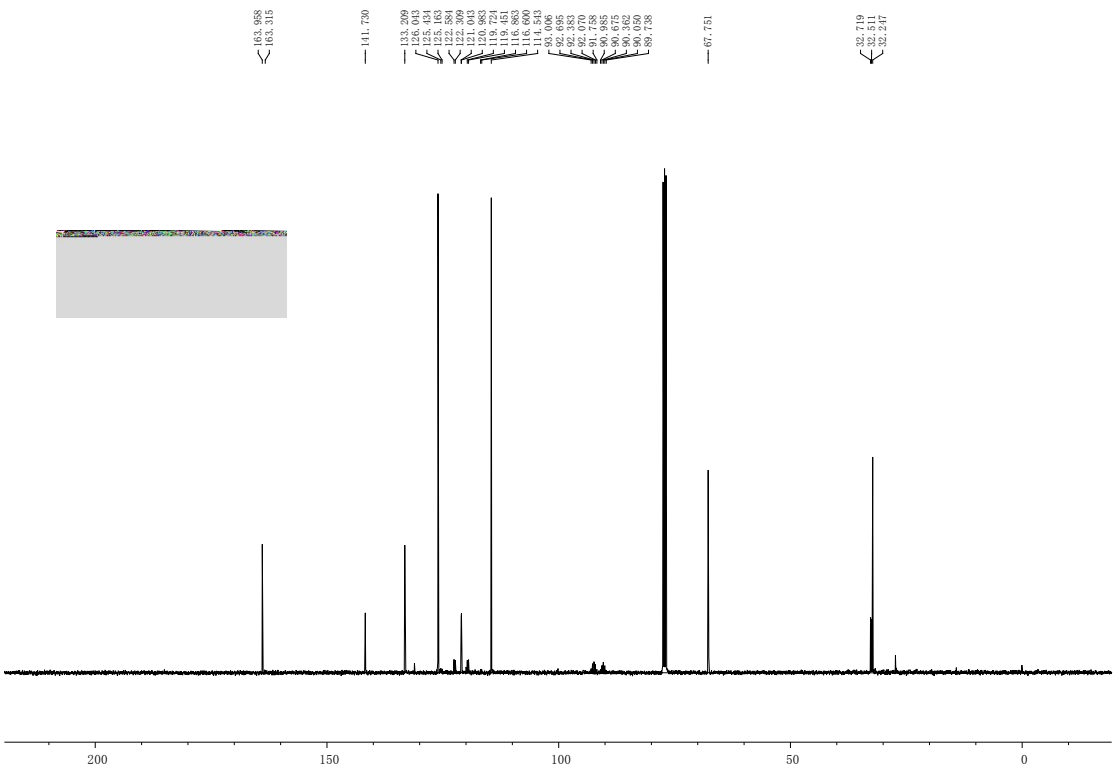
^{1}H NMR spectrum for 2o: (*E*)-1-nitro-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



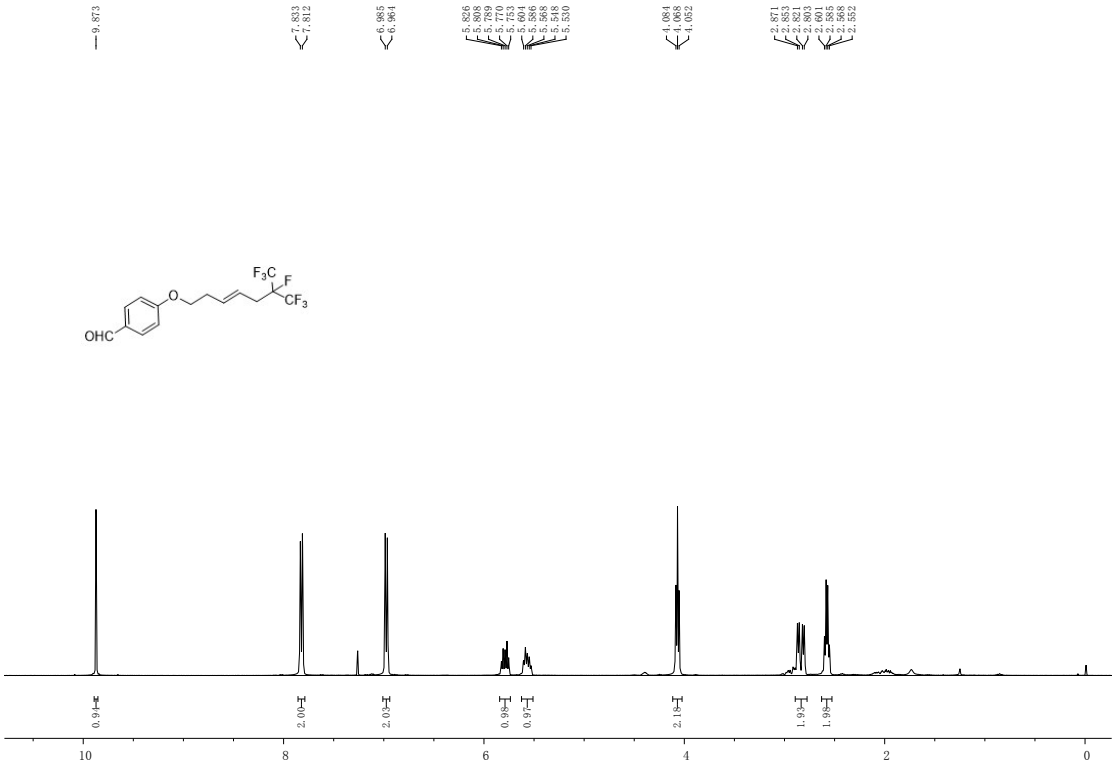
^{19}F NMR spectrum for 2o: (*E*)-1-nitro-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



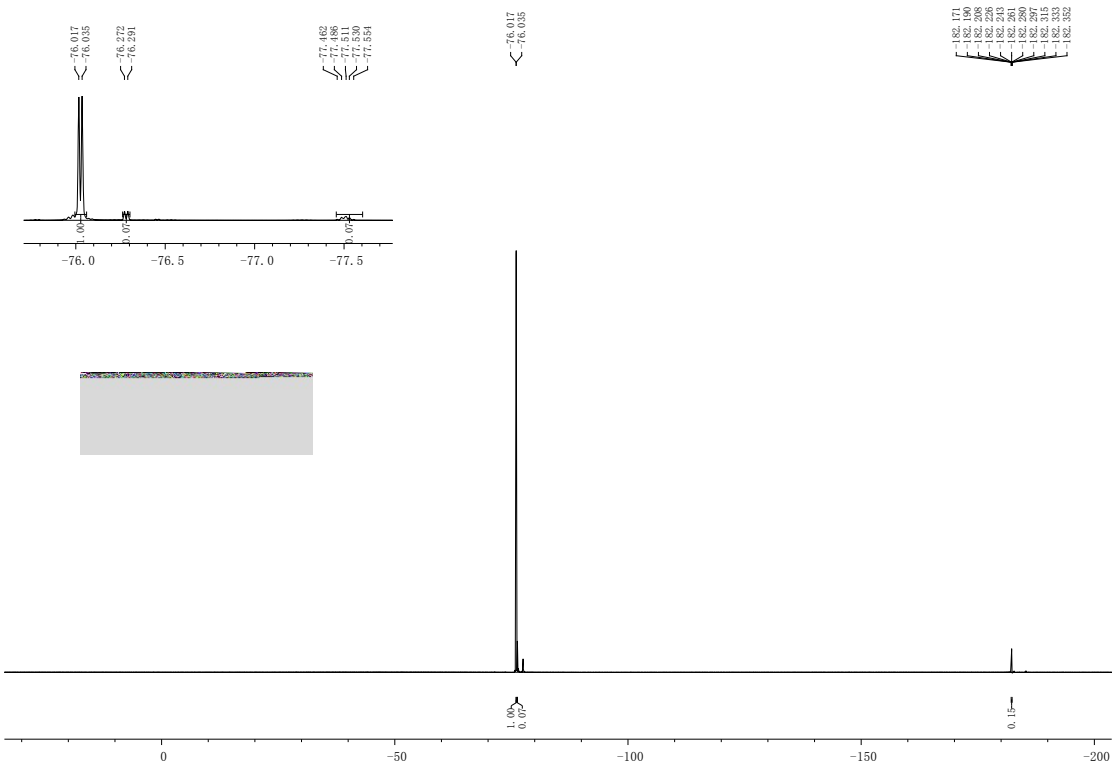
^{13}C NMR spectrum for 2o: (*E*)-1-nitro-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



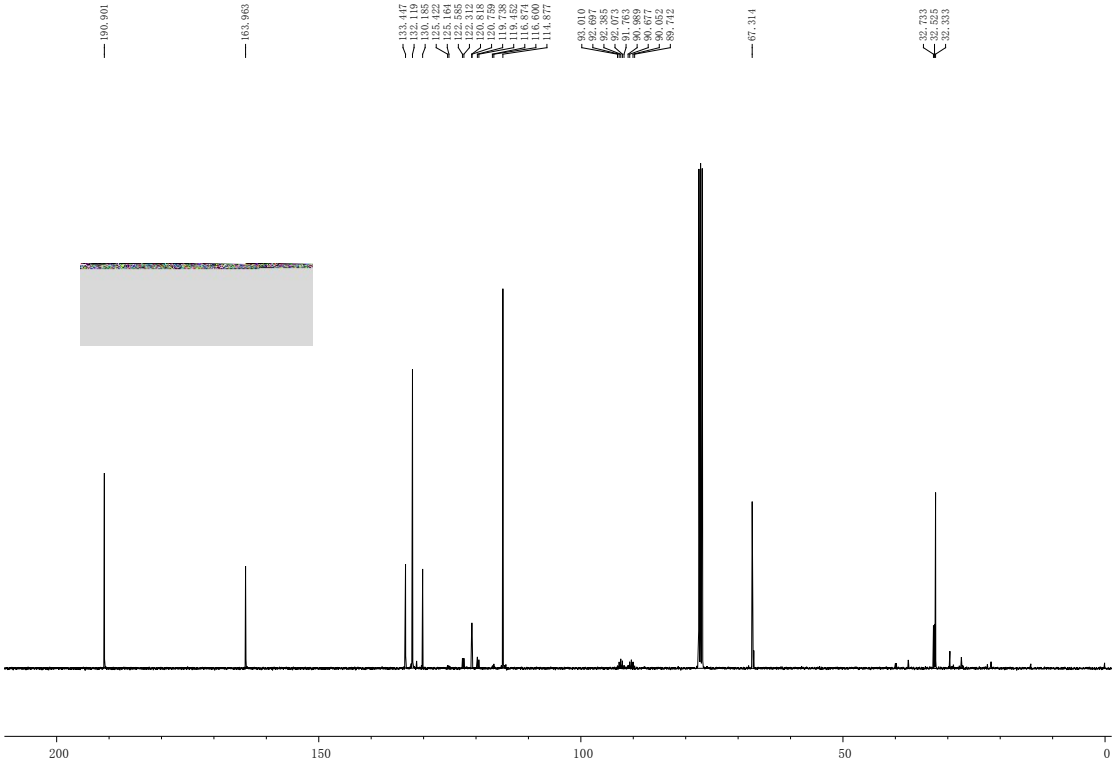
^{1}H NMR spectrum for 2p: (*E*)-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzaldehyde



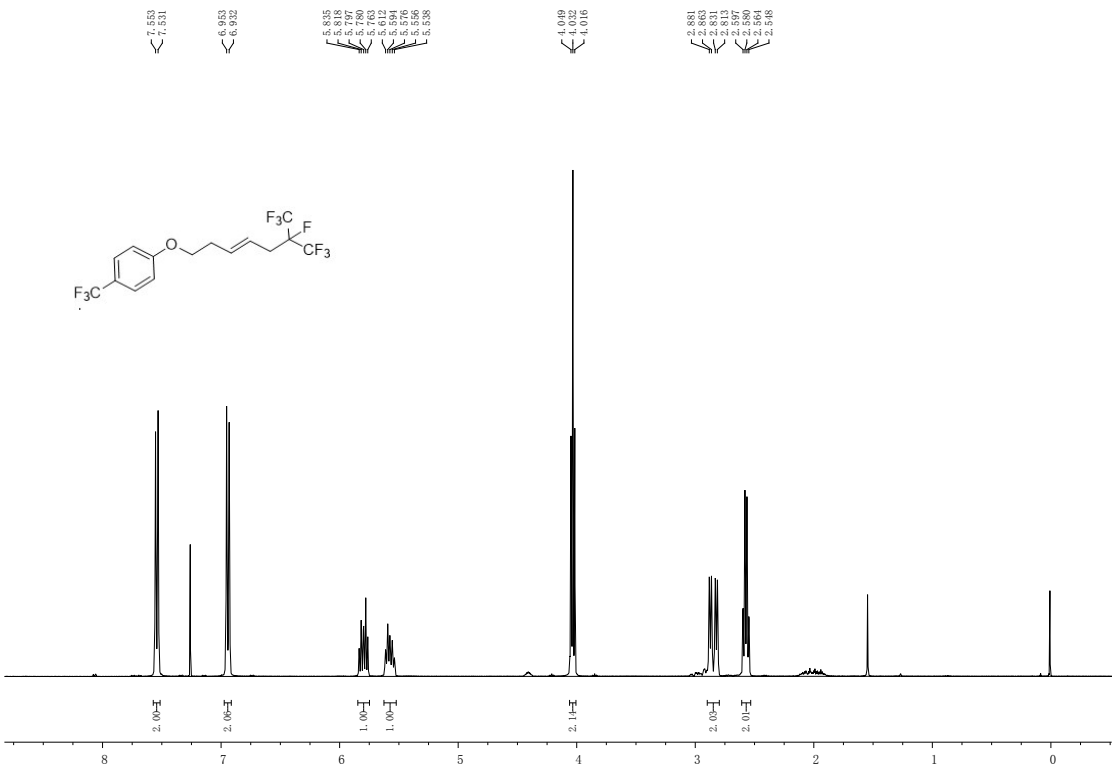
^{19}F NMR spectrum for 2p: (*E*)-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzaldehyde



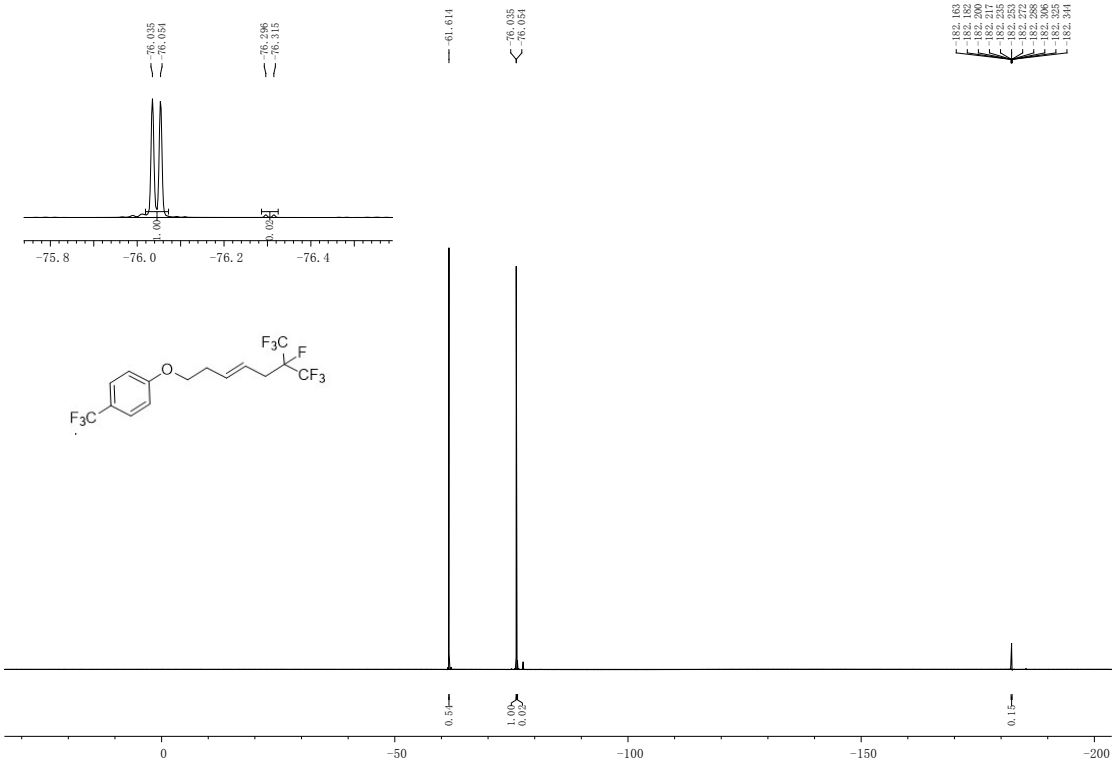
^{13}C NMR spectrum for 2p: (*E*)-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzaldehyde



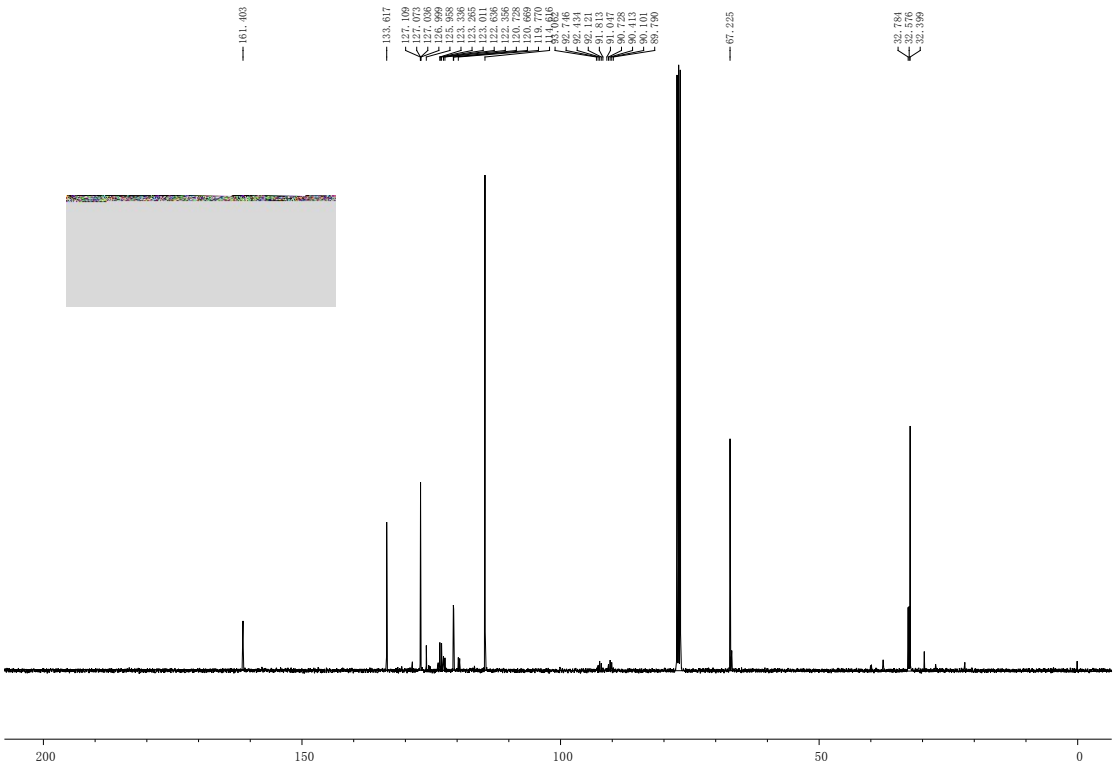
^{1}H NMR spectrum for 2q: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-4-(trifluoromethyl)benzene



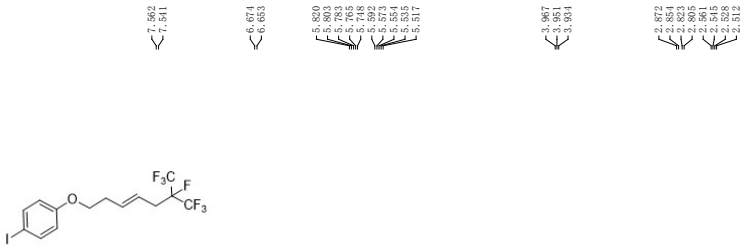
^{19}F NMR spectrum for 2q: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-4-(trifluoromethyl)benzene

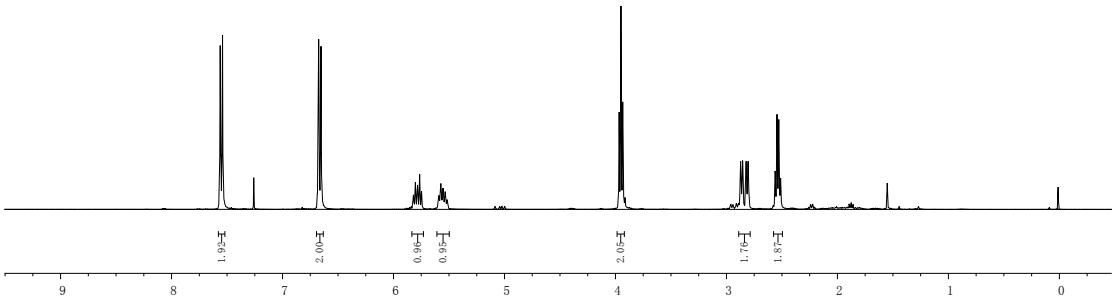


^{13}C NMR spectrum for 2q: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-4-(trifluoromethyl)benzene

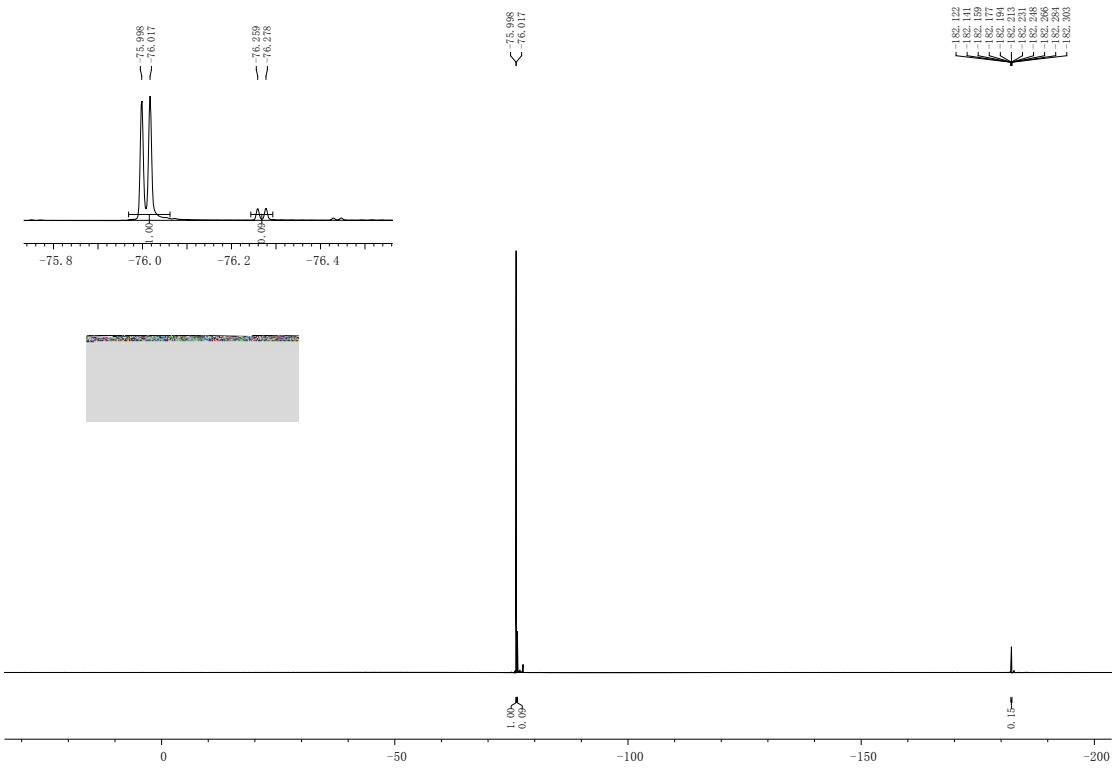


^{1}H NMR spectrum for 2r: (*E*)-1-iodo-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene

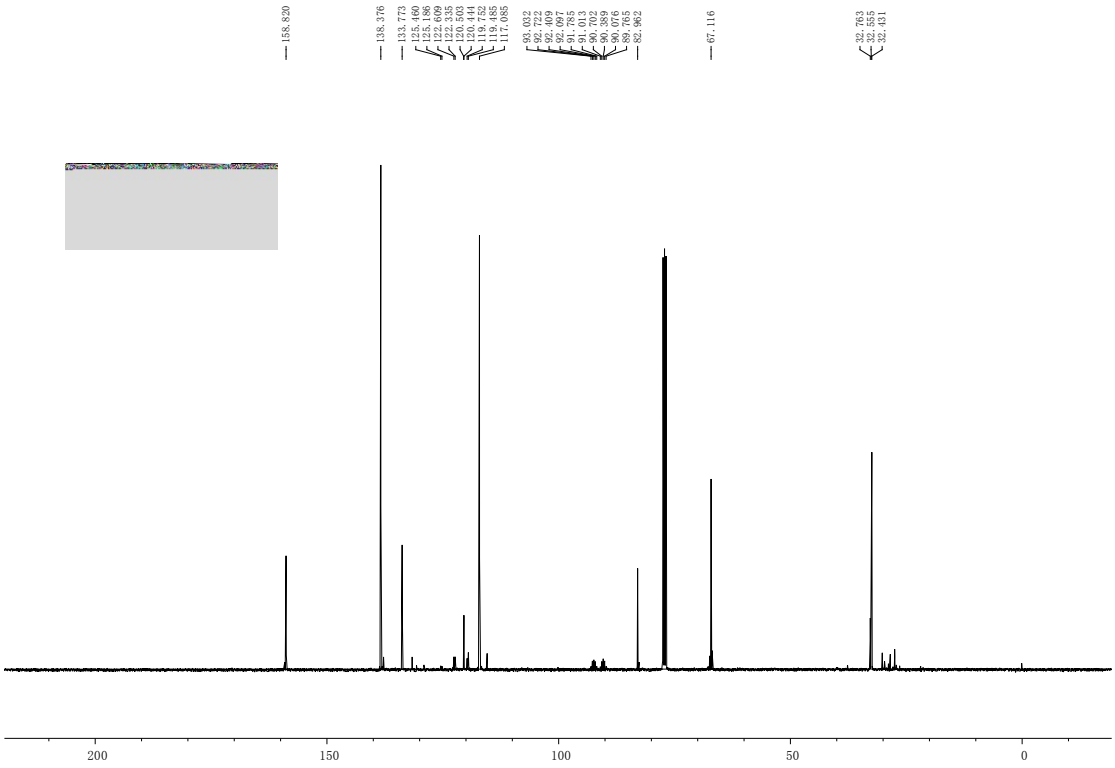




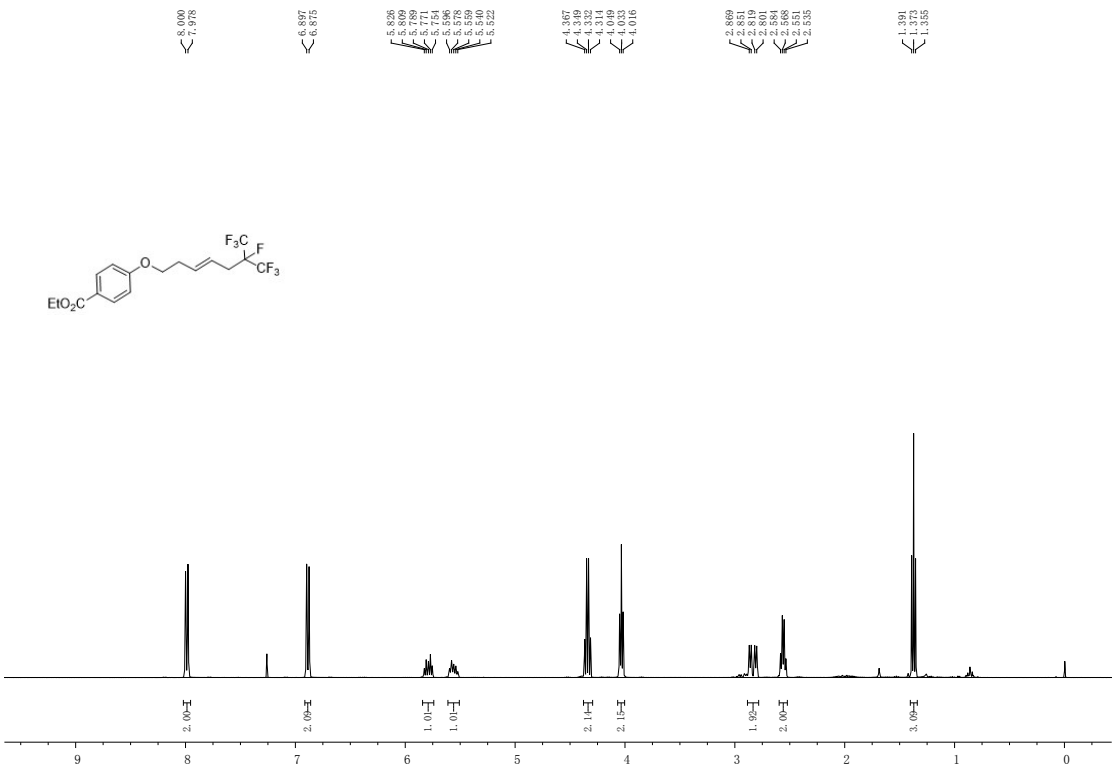
^{19}F NMR spectrum for 2r: (*E*)-1-iodo-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



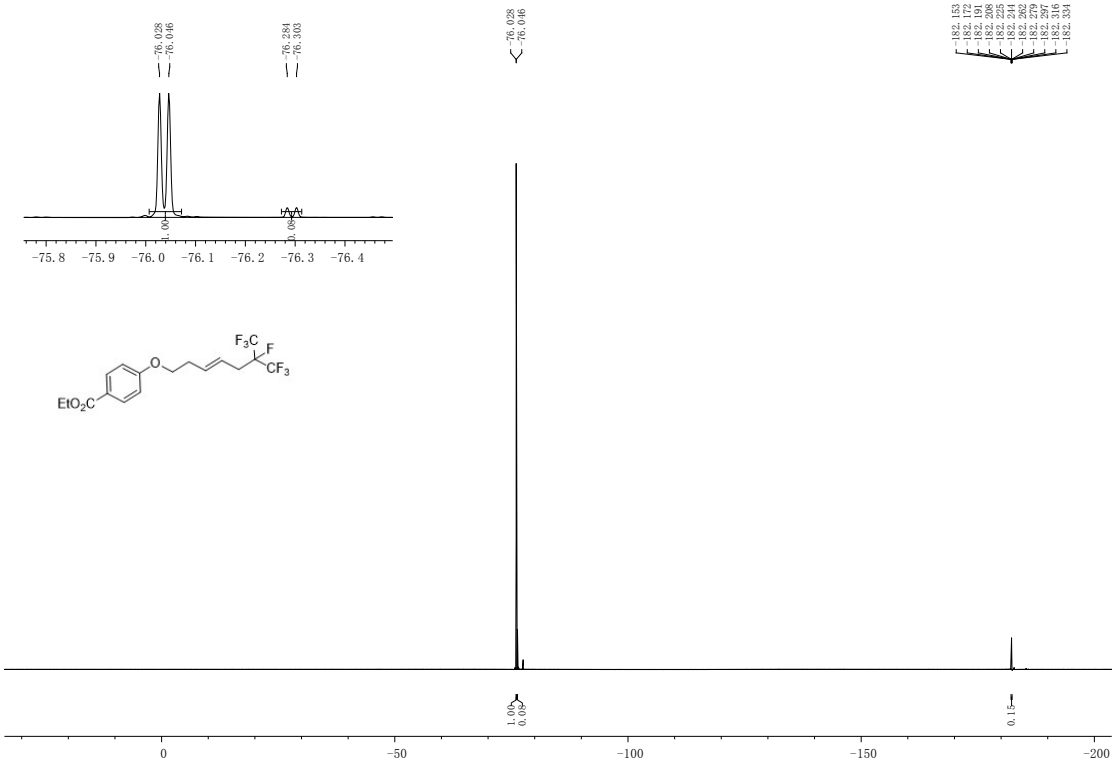
^{13}C NMR spectrum for 2r: (*E*)-1-iodo-4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



^{1}H NMR spectrum for (*E*)-ethyl 2s: 4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzoate

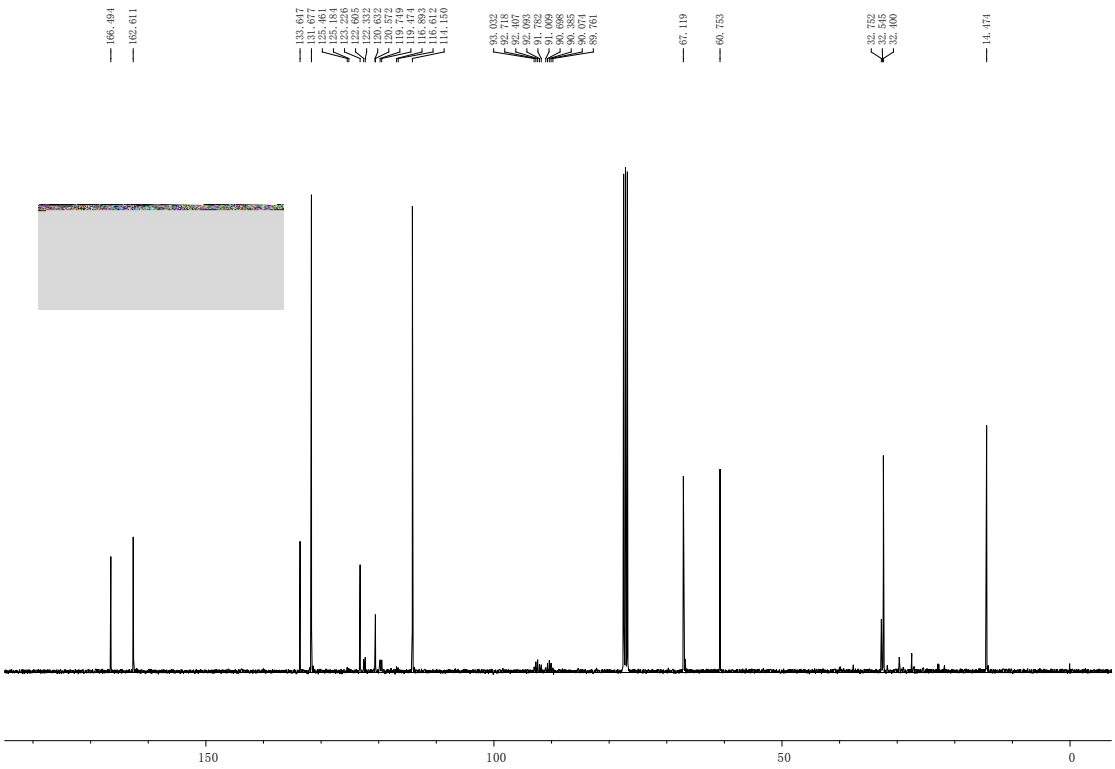


^{19}F NMR spectrum for (*E*)-ethyl 2s: 4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzoate

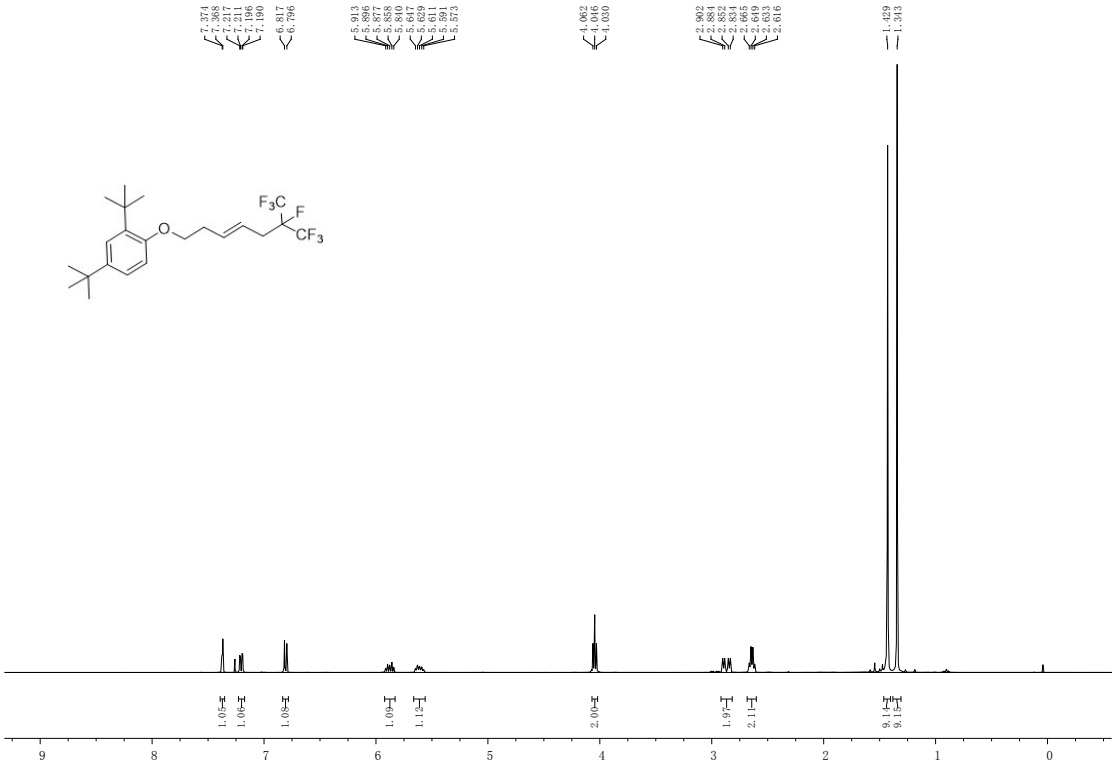


^{13}C NMR spectrum for (*E*)-ethyl 2s: 4-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-

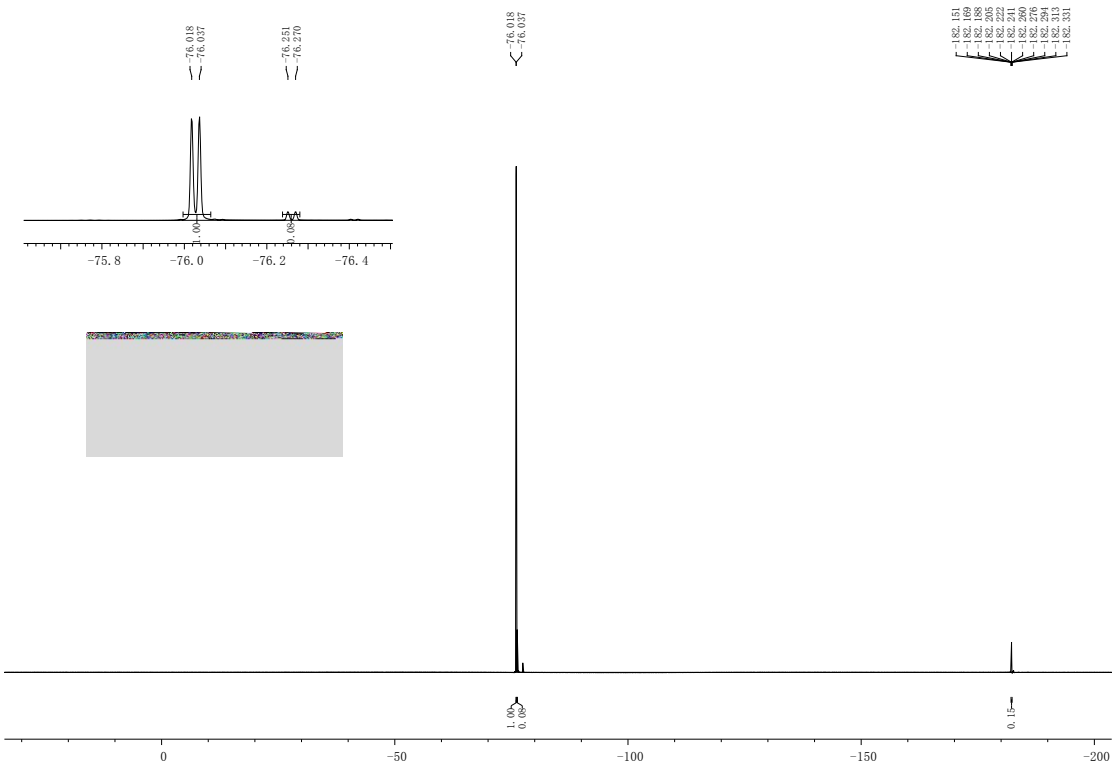
yl)oxy)benzoate



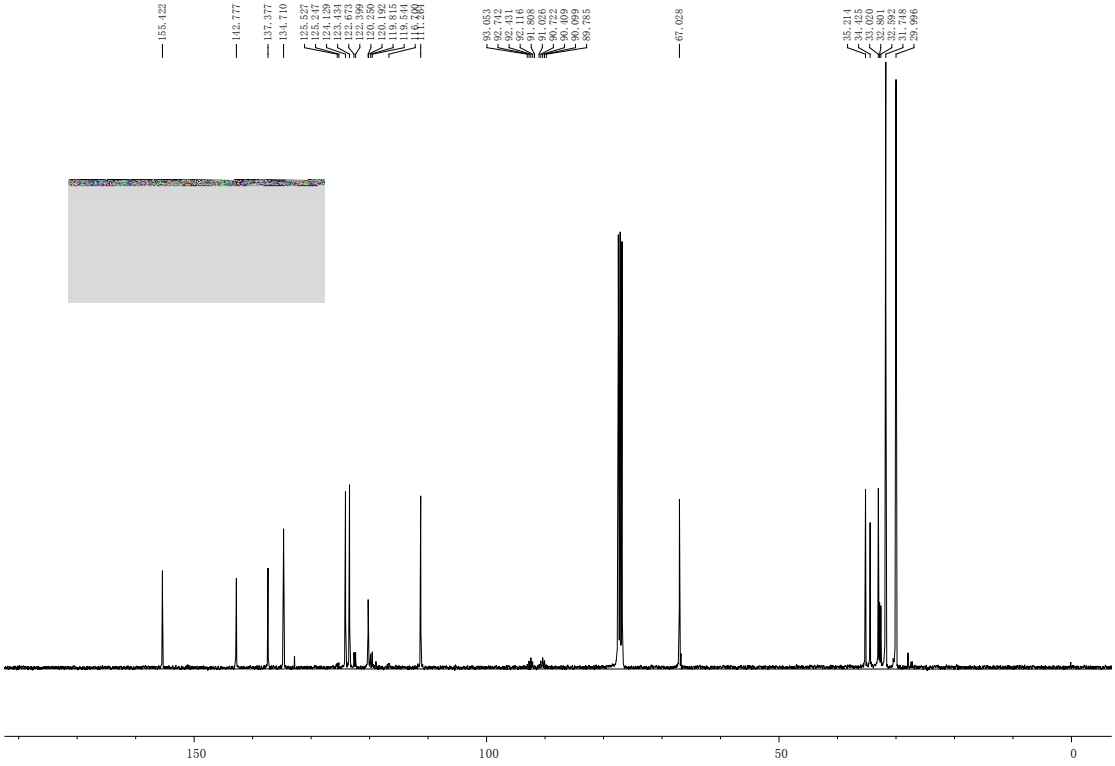
^{1}H NMR spectrum for 2t: (*E*)-2,4-di-tert-butyl-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



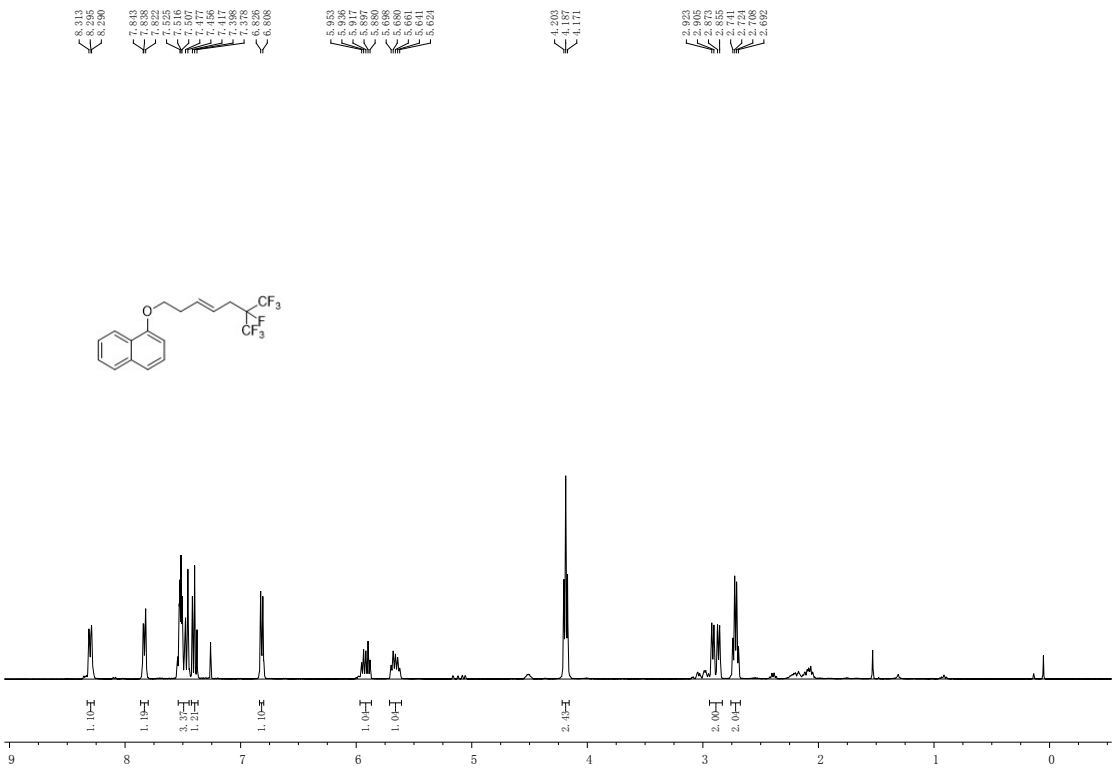
^{19}F NMR spectrum for 2t: (*E*)-2,4-di-tert-butyl-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



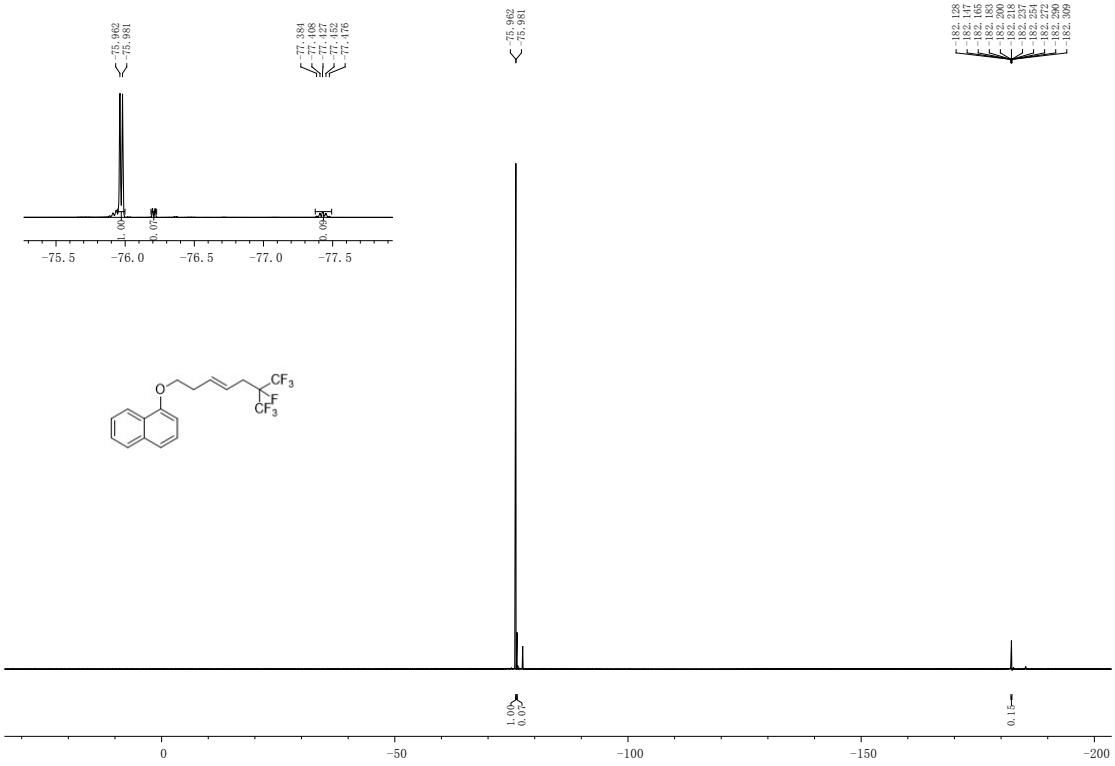
^{13}C NMR spectrum for 2t: (*E*)-2,4-di-tert-butyl-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)benzene



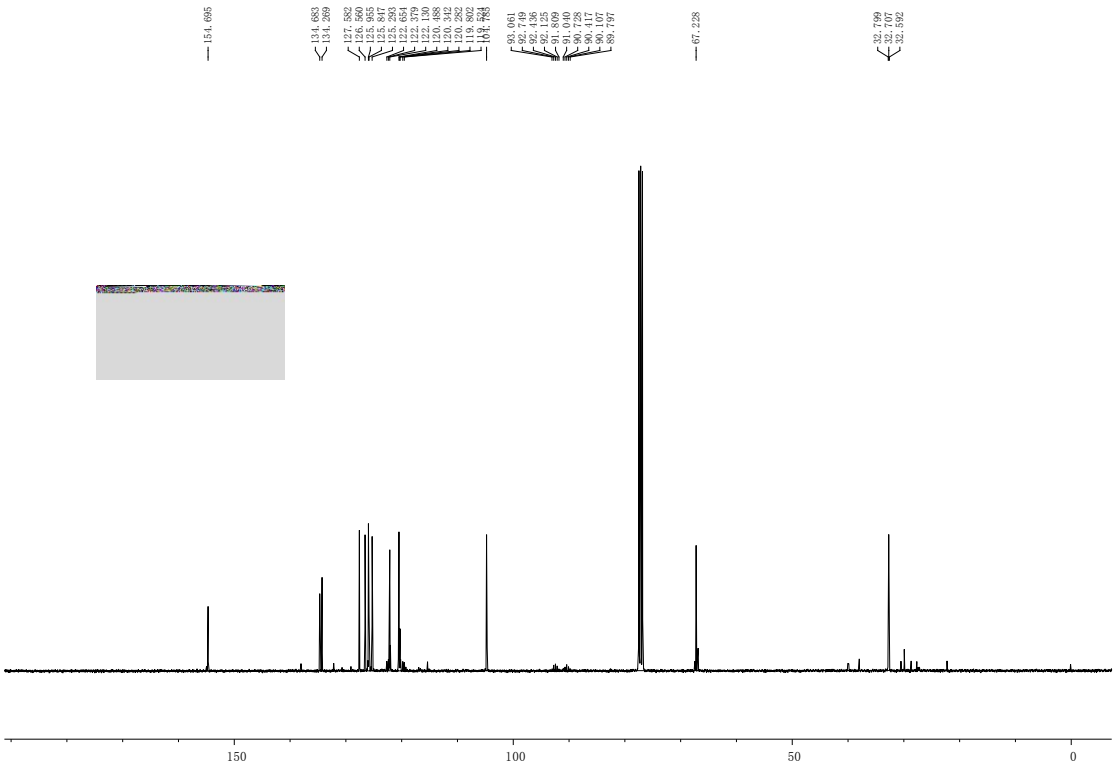
^{1}H NMR spectrum for 2u: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)naphthalene



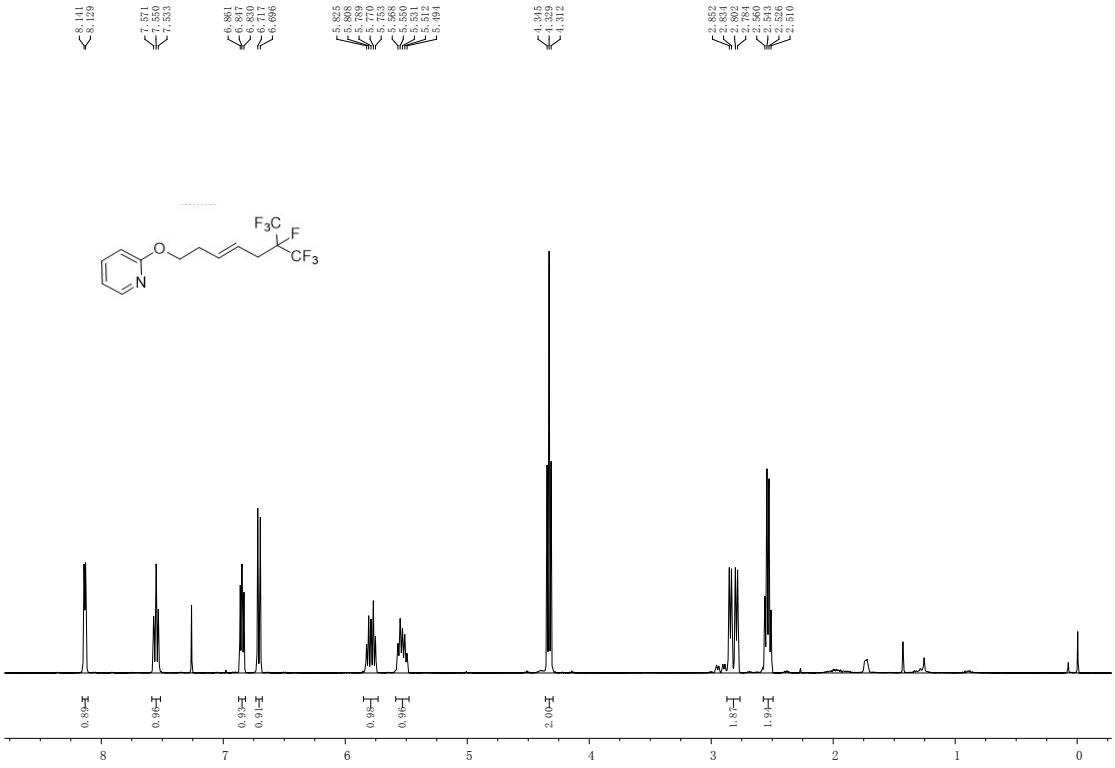
^{19}F NMR spectrum for 2u: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)naphthalene



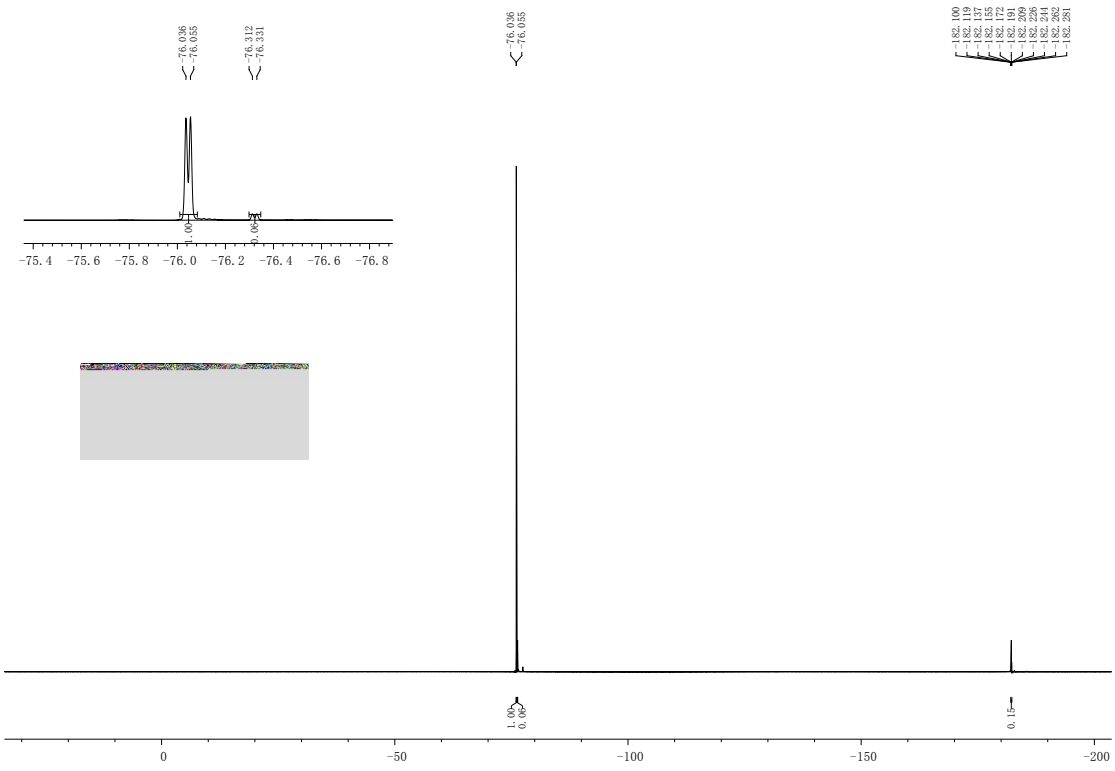
^{13}C NMR spectrum for 2u: (*E*)-1-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)naphthalene



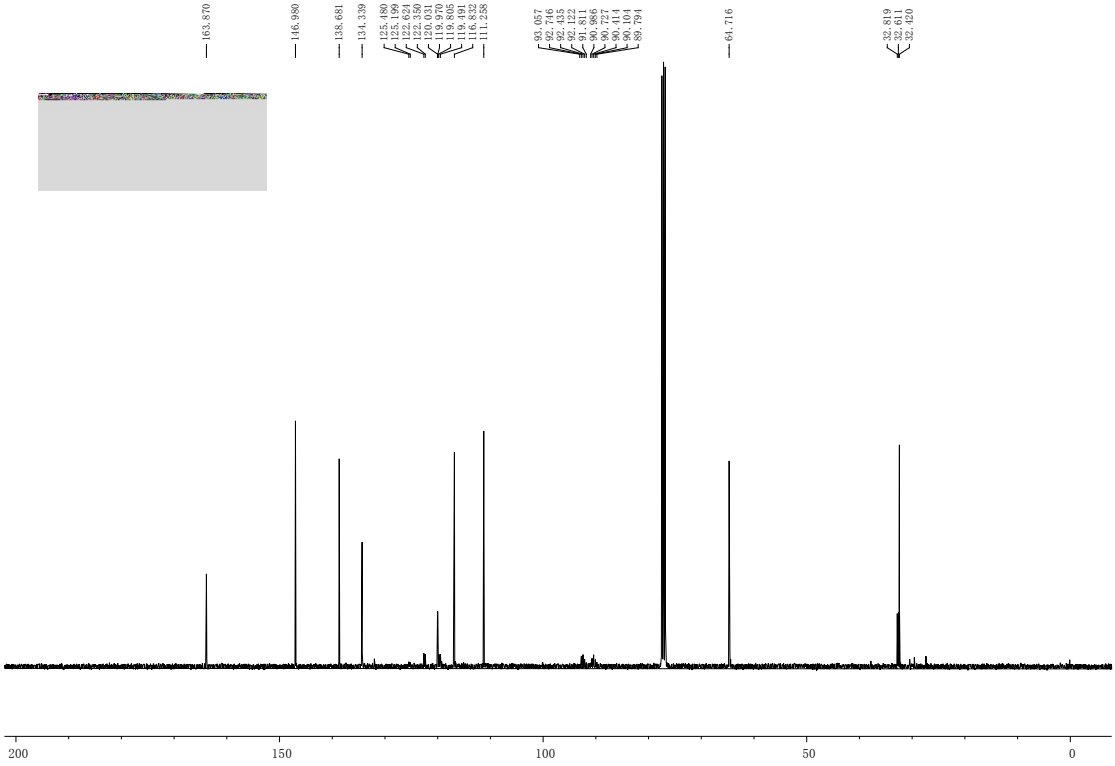
^{1}H NMR spectrum for 2v: (*E*)-2-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)pyridine



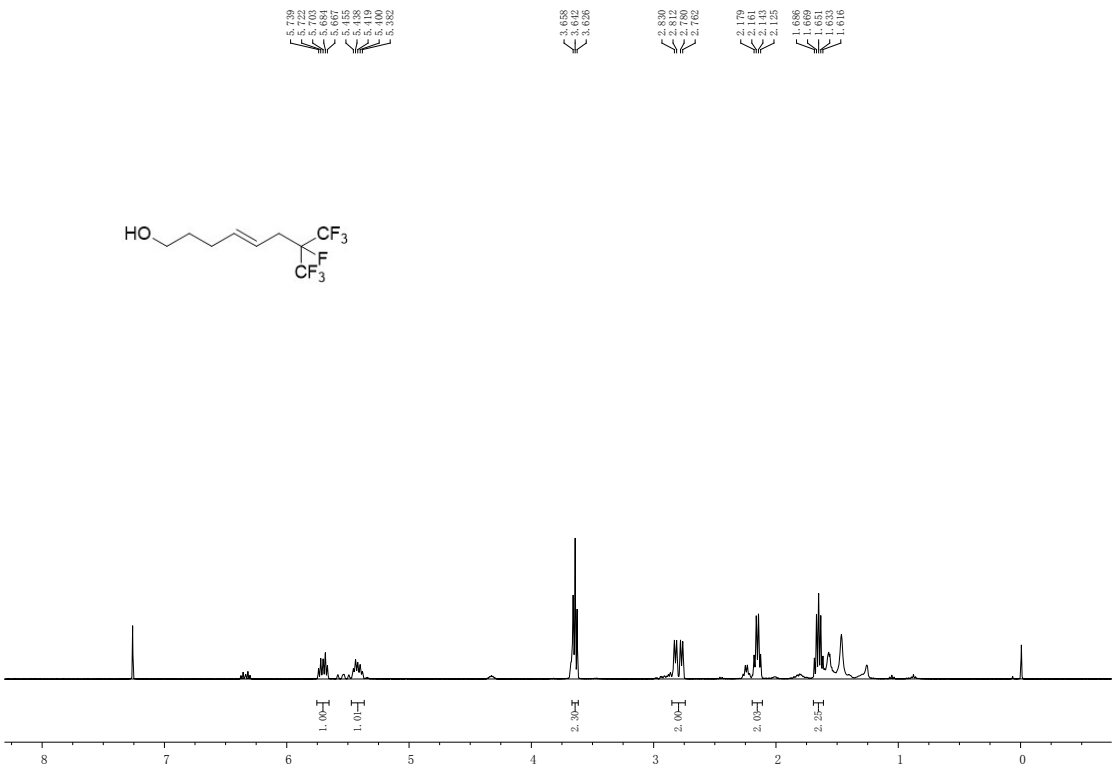
^{19}F NMR spectrum for 2v: (*E*)-2-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)pyridine



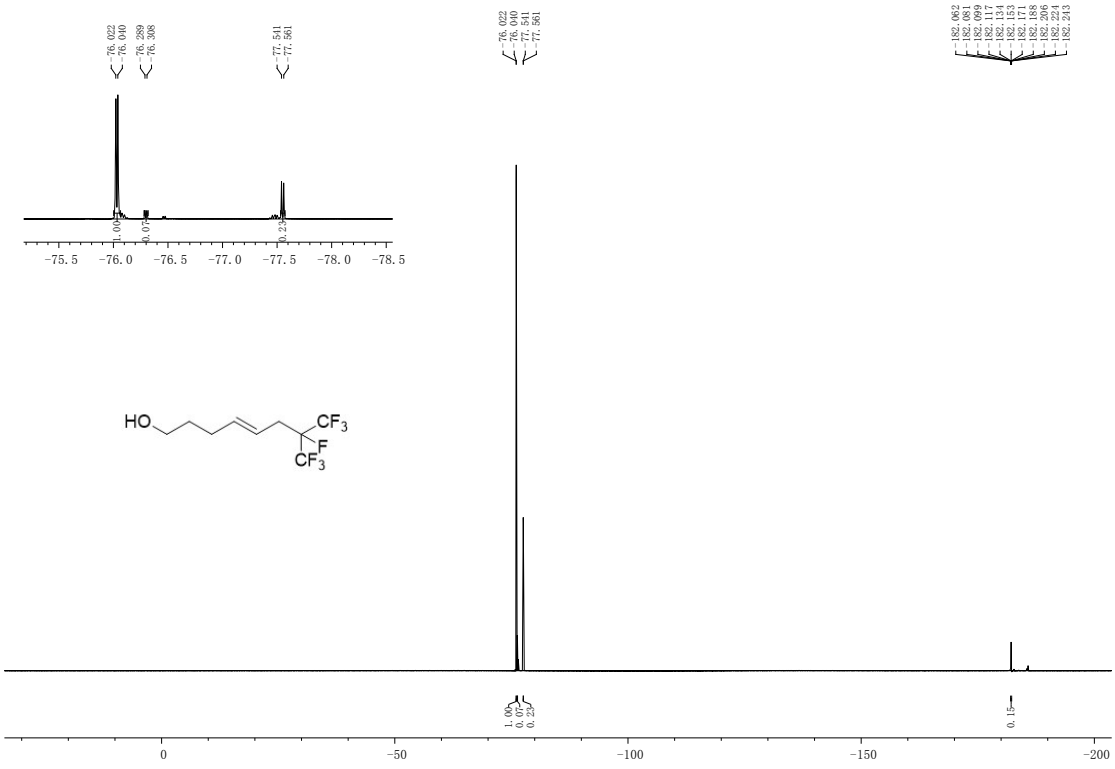
^{13}C NMR spectrum for 2v: (*E*)-2-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)pyridine



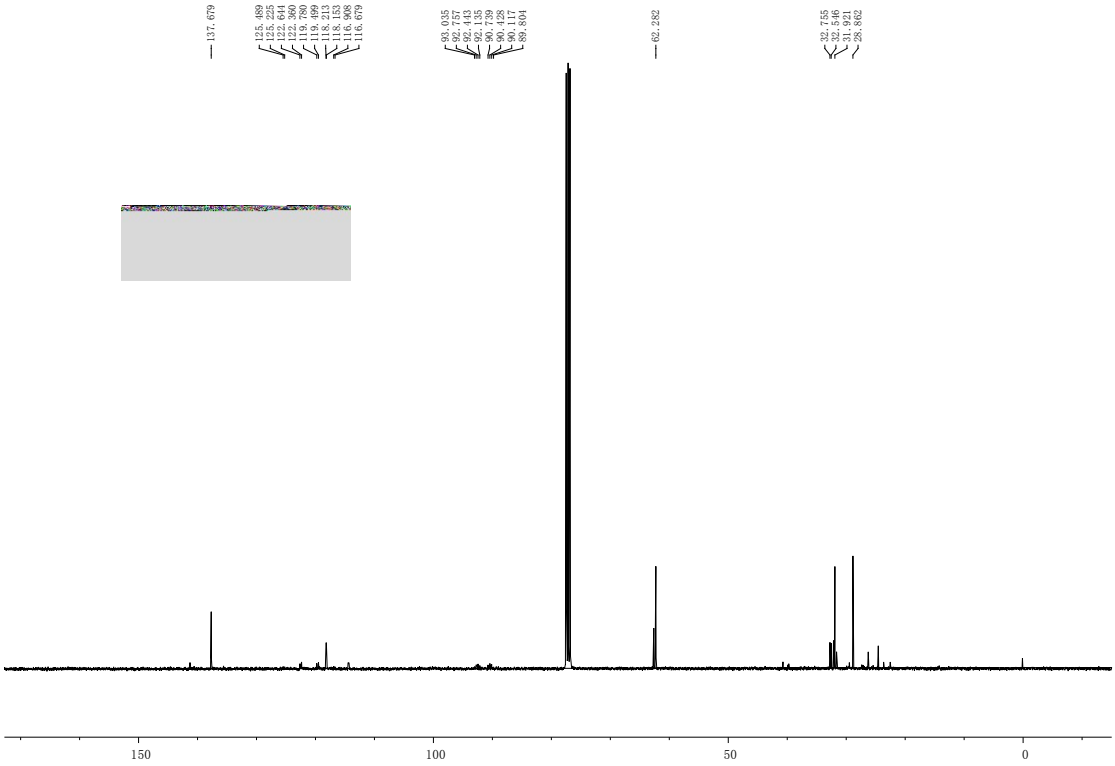
^{1}H NMR spectrum for 2w: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-ol



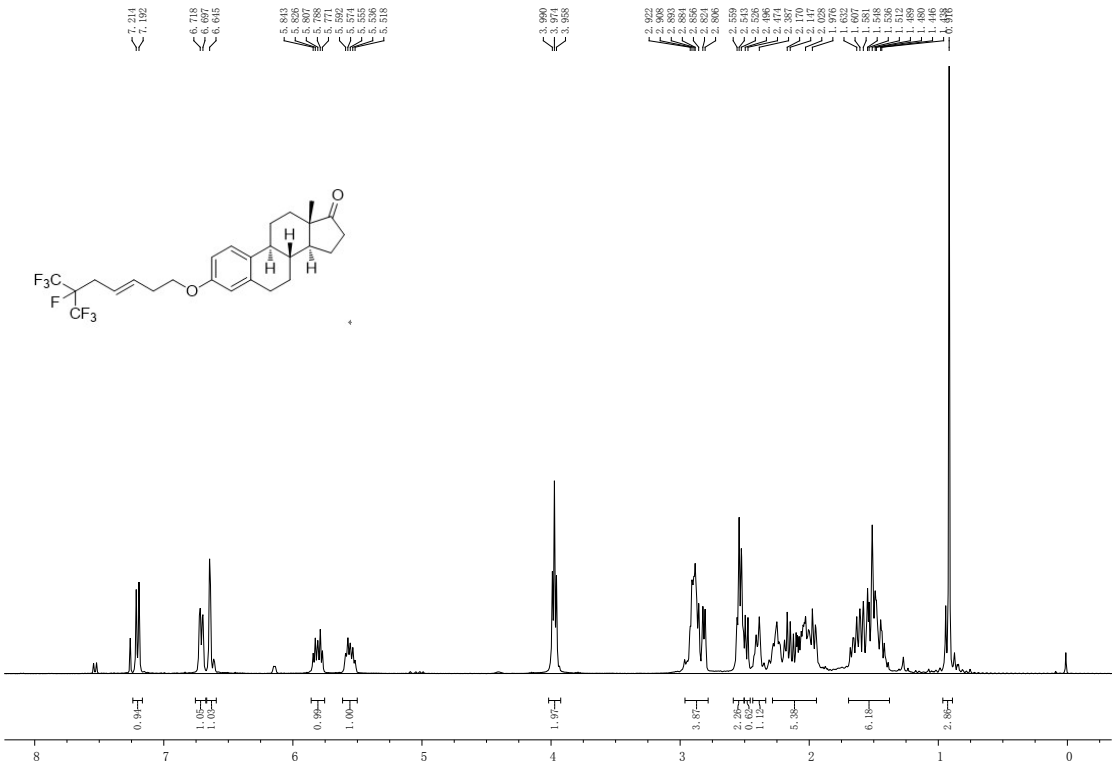
^{19}F NMR spectrum for 2w: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-ol



^{13}C NMR spectrum for 2w: (*E*)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)oct-4-en-1-ol

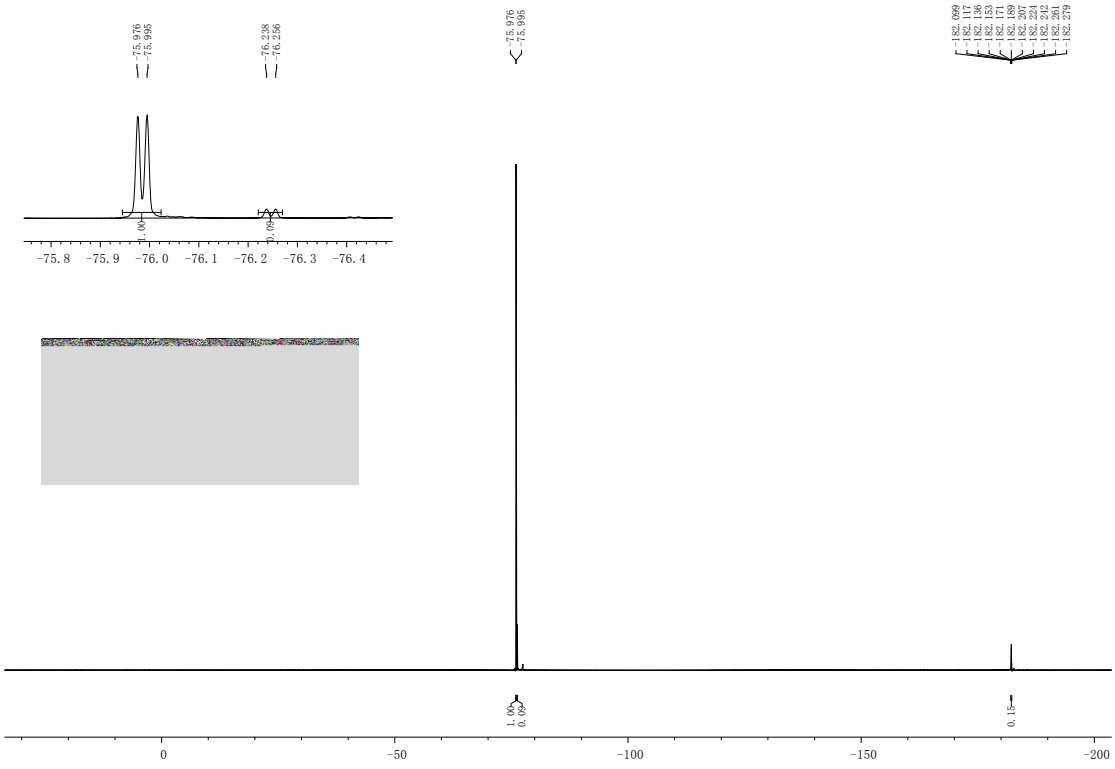


^{1}H NMR spectrum for 2x: (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-(((*E*)-6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one

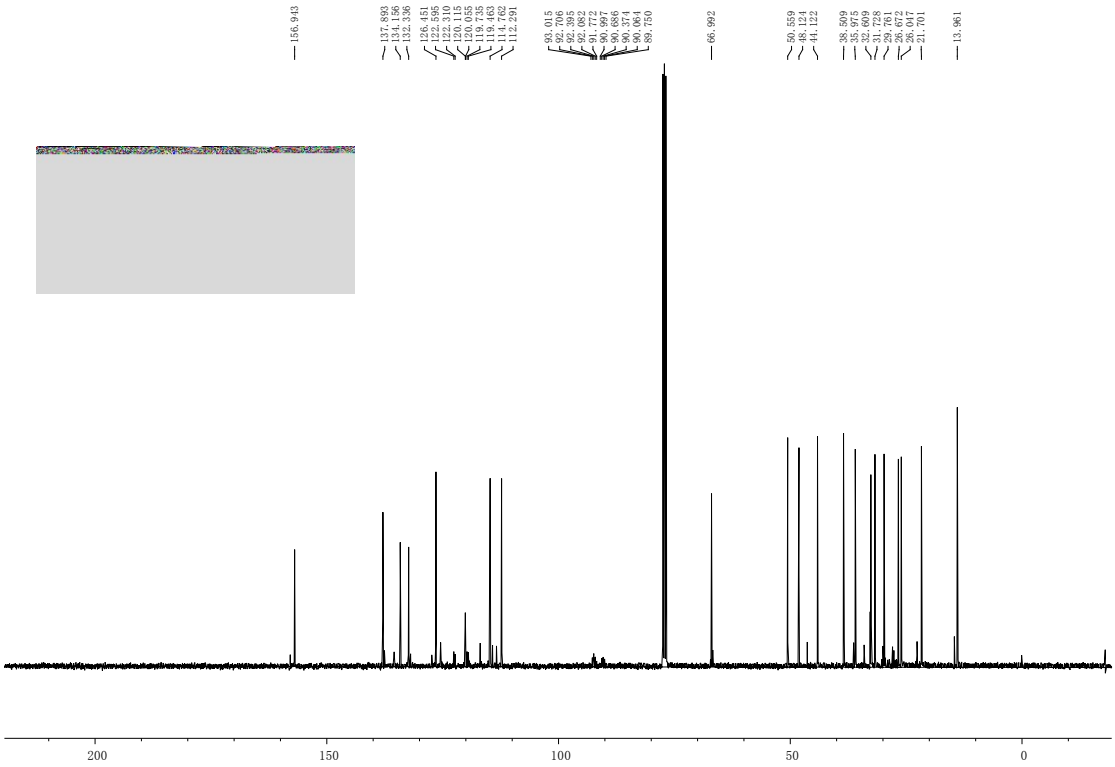


^{19}F NMR spectrum for 2x: (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-(((*E*)-6,7,7,7-tetrafluoro-6-

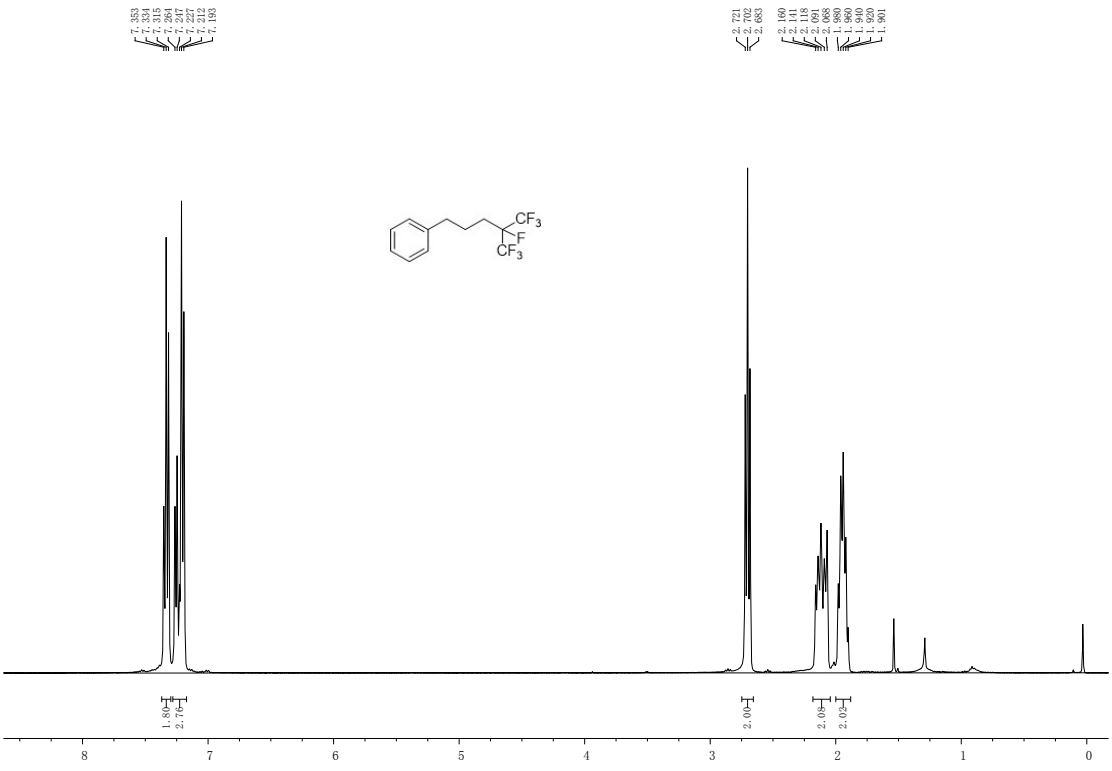
(trifluoromethyl)hept-3-en-1-yl)oxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one



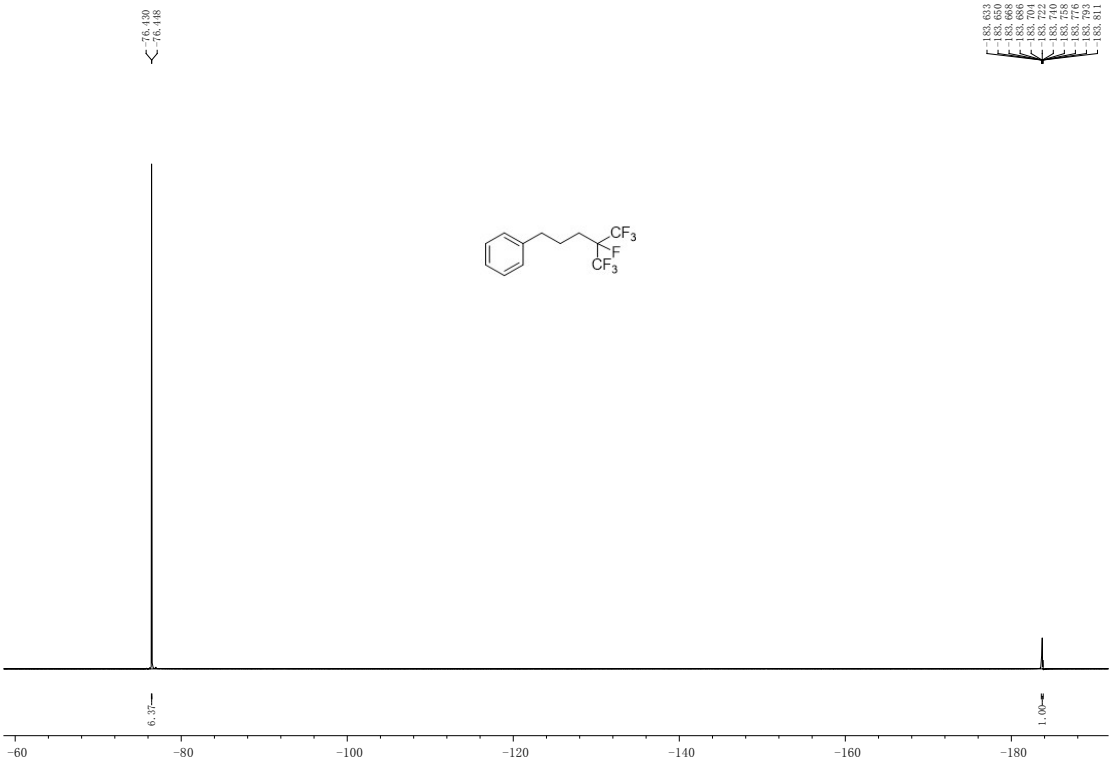
^{13}C NMR spectrum for 2x: (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-(((*E*)-6,7,7,7-tetrafluoro-6-(trifluoromethyl)hept-3-en-1-yl)oxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[a]phenanthren-17(14*H*)-one



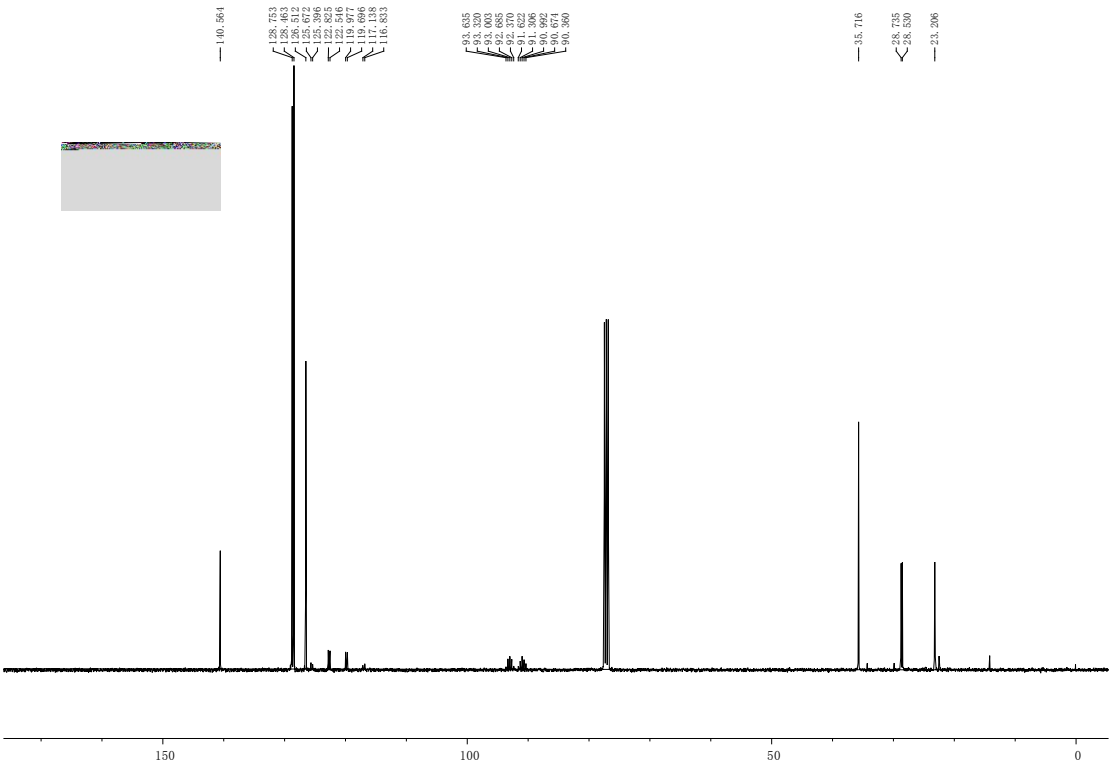
^{1}H NMR spectrum for 3a: (4,5,5,5-tetrafluoro-4-(trifluoromethyl)pentyl)benzene



^{19}F NMR spectrum for 3a: (4,5,5,5-tetrafluoro-4-(trifluoromethyl)pentyl)benzene

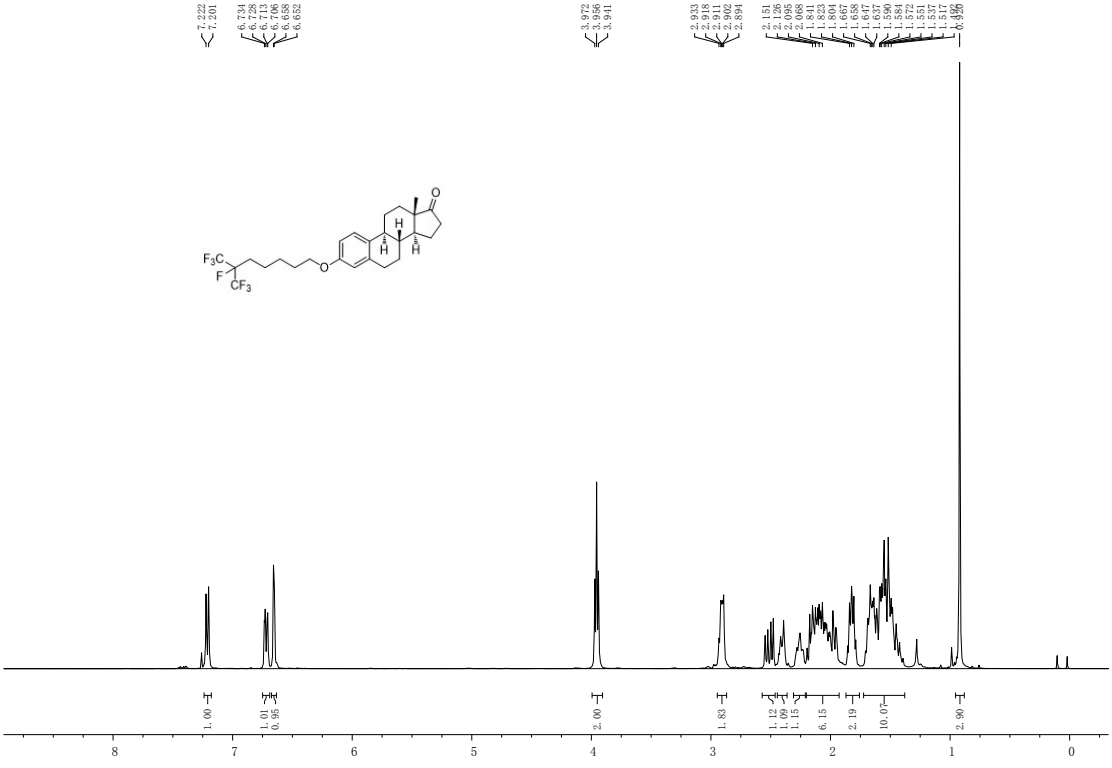


^{13}C NMR spectrum for 3a: (4,5,5,5-tetrafluoro-4-(trifluoromethyl)pentyl)benzene



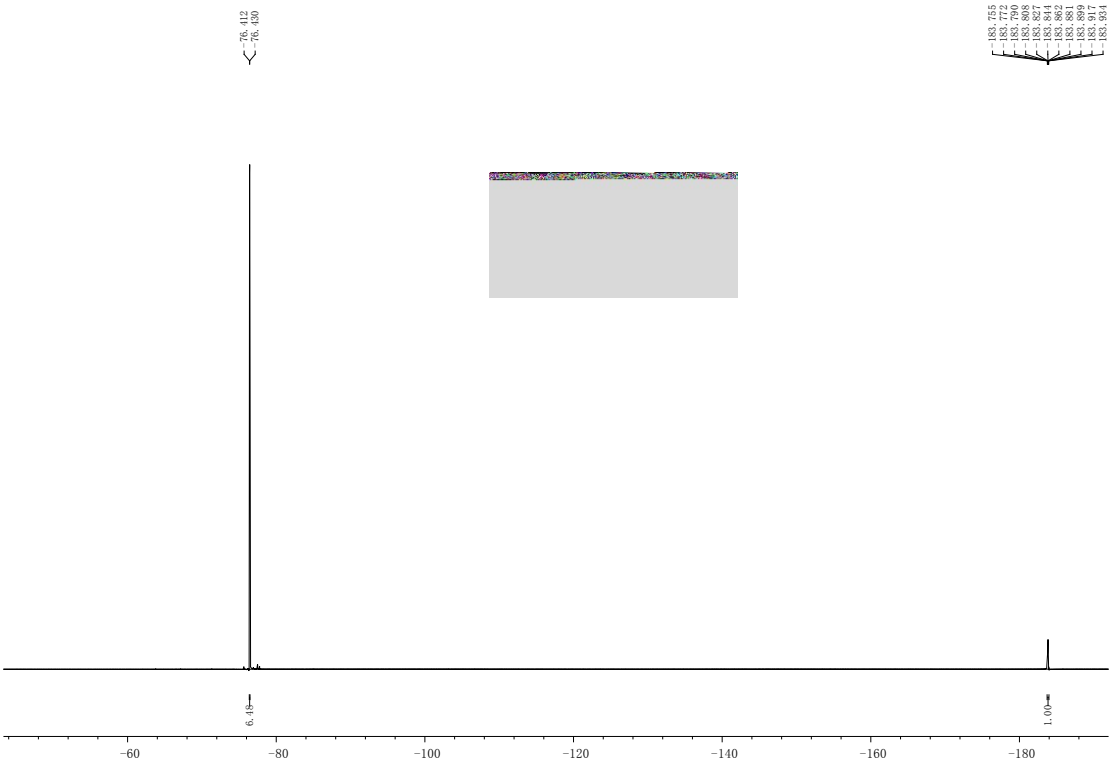
^{1}H NMR spectrum for 3x:

(*8R*,*9S*,*13S*,*14S*)-13-methyl-3-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)heptyl)oxy)-7,8,9,11,12,13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one

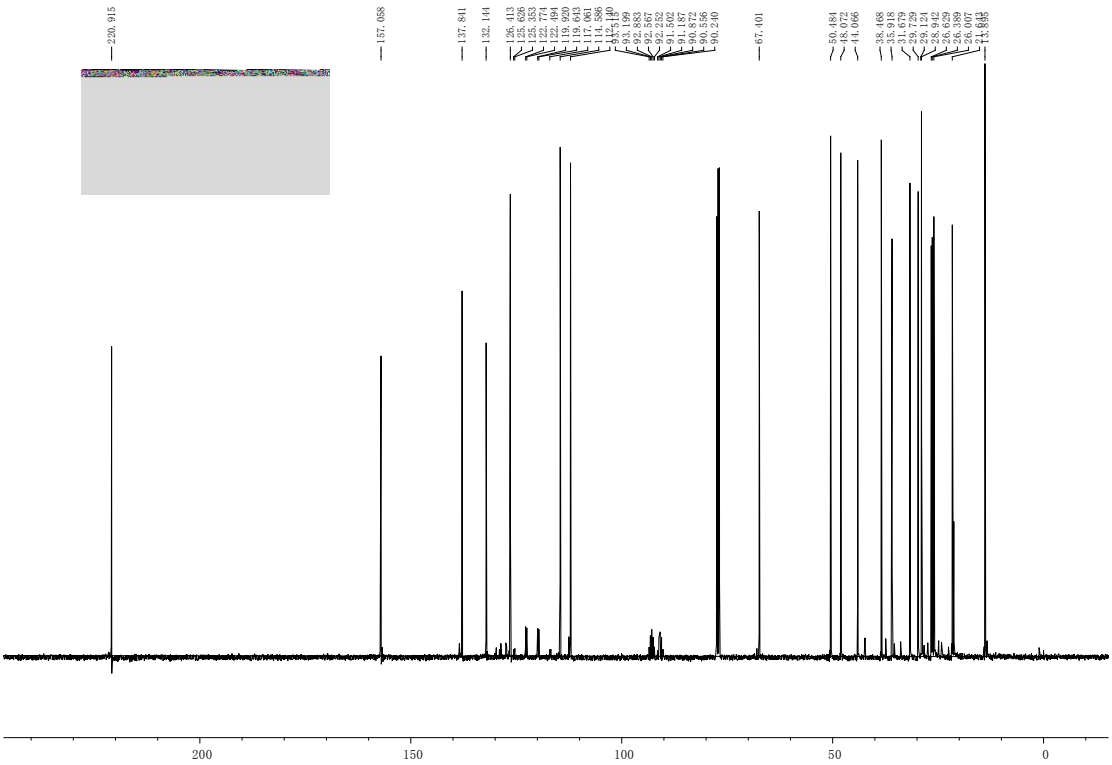


^{19}F NMR spectrum for 3x:

(*8R*,*9S*,*13S*,*14S*)-13-methyl-3-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)heptyl)oxy)-7,8,9,11,12,13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one

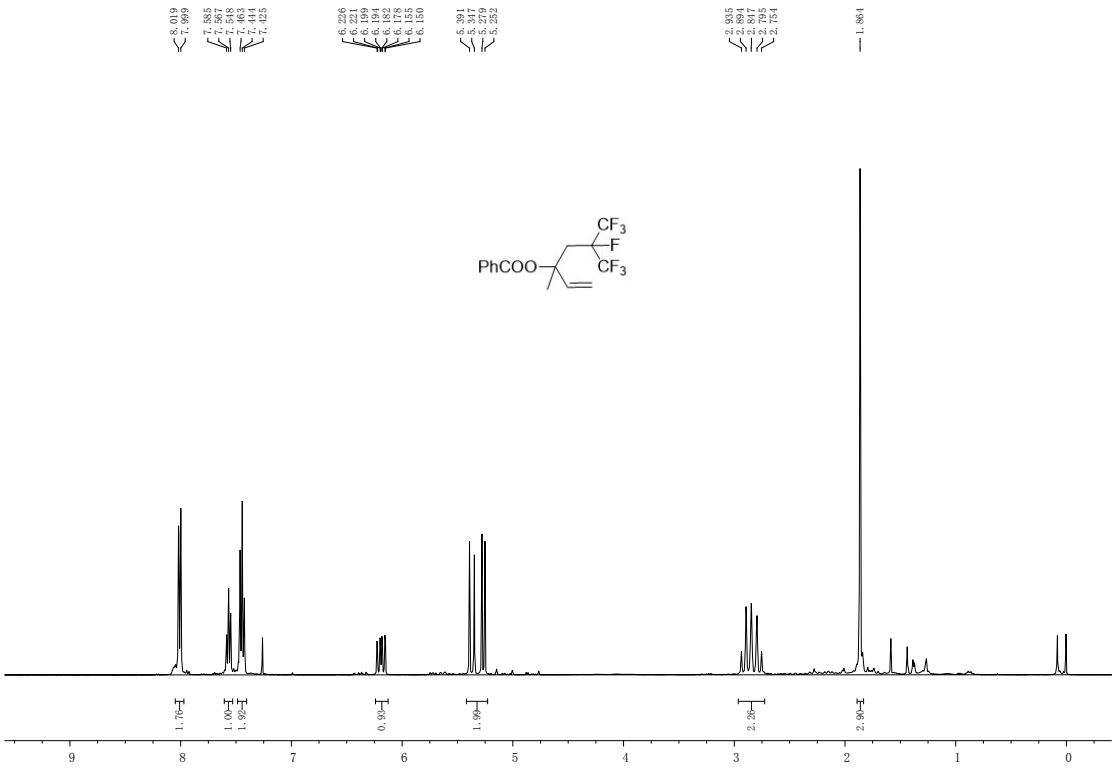


^{13}C NMR spectrum for 3x: (*8R*,*9S*,*13S*,*14S*)-13-methyl-3-((6,7,7,7-tetrafluoro-6-(trifluoromethyl)heptyl)oxy)-7,8,9,11,12,13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one

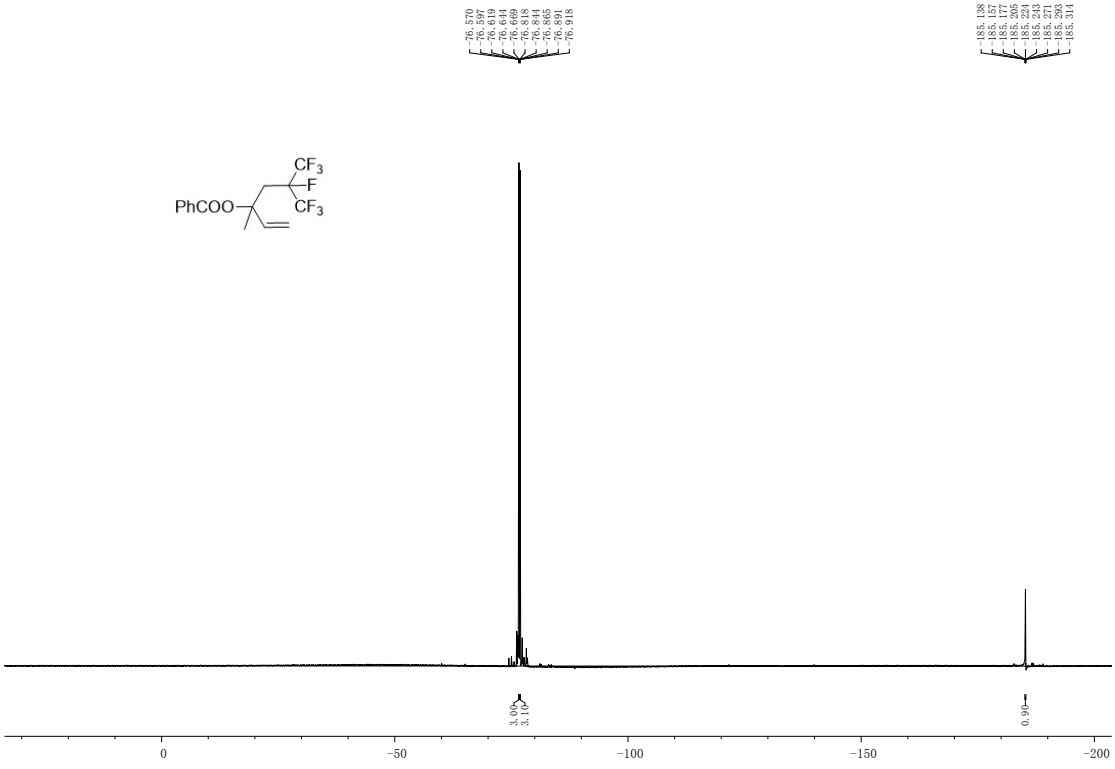


^{1}H NMR spectrum for 7a: 5,6,6,6-tetrafluoro-3-methyl-5-(trifluoromethyl)hex-1-

en-3-yl benzoate

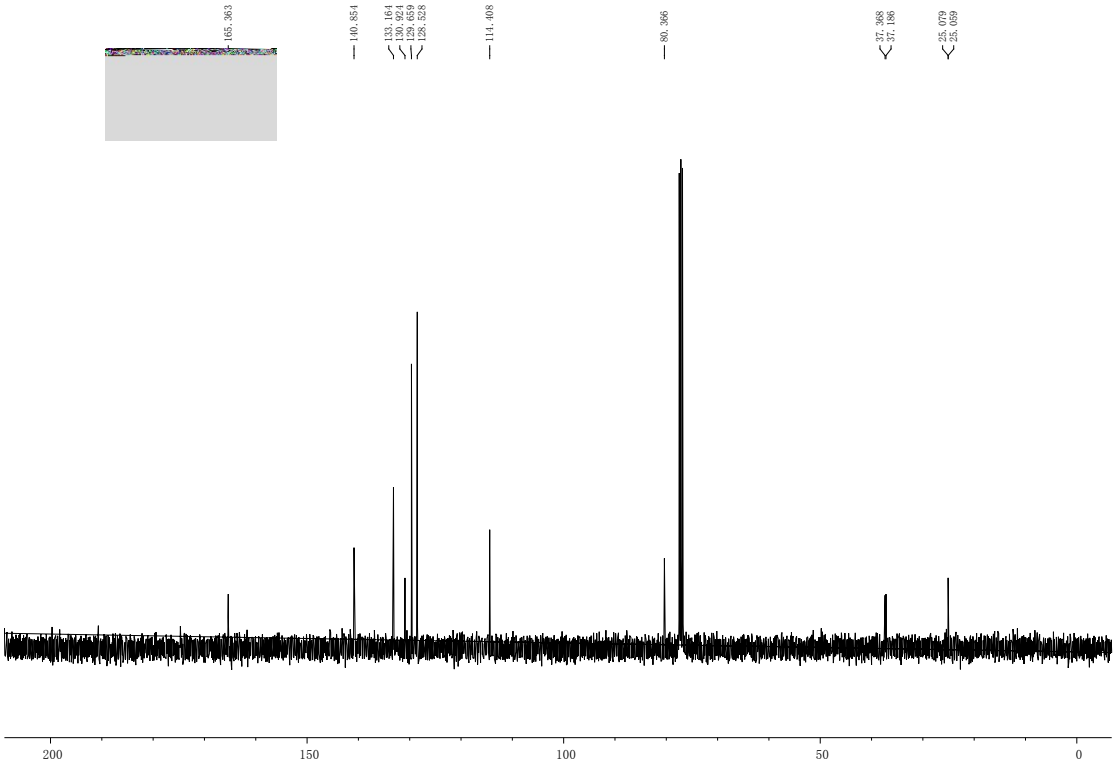


^{19}F NMR spectrum for 7a: 5,6,6,6-tetrafluoro-3-methyl-5-(trifluoromethyl)hex-1-en-3-yl benzoate

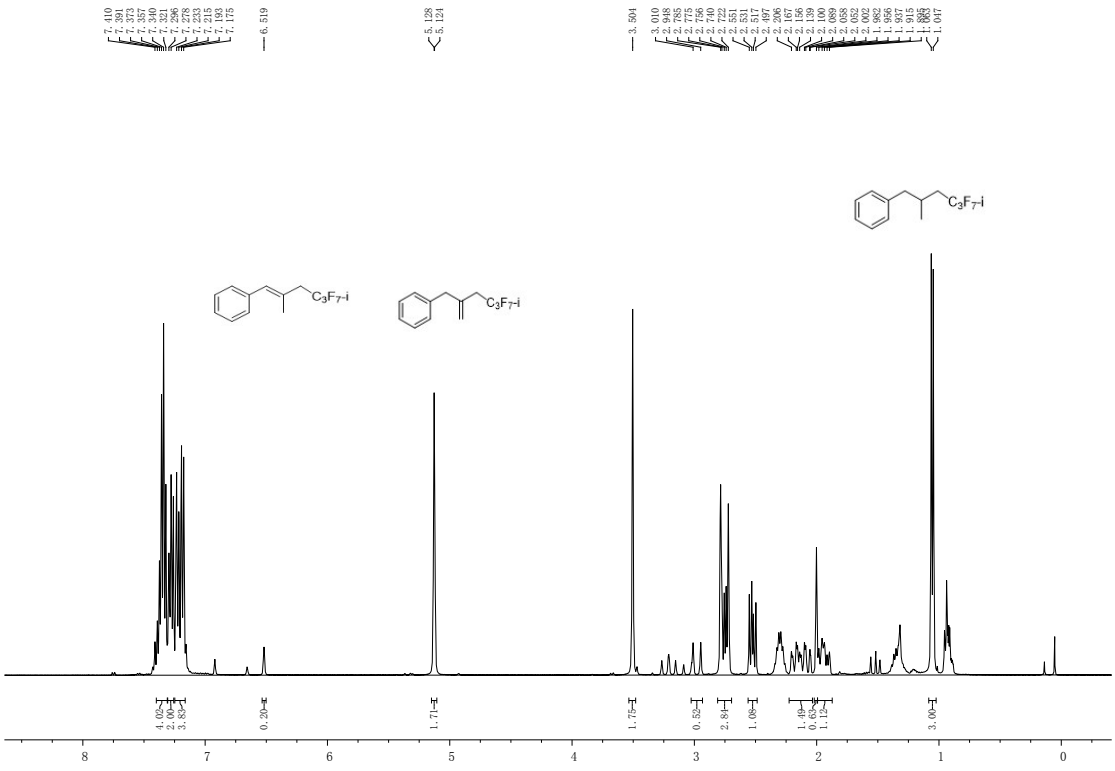


^{13}C NMR spectrum for 7a: 5,6,6,6-tetrafluoro-3-methyl-5-(trifluoromethyl)hex-1-

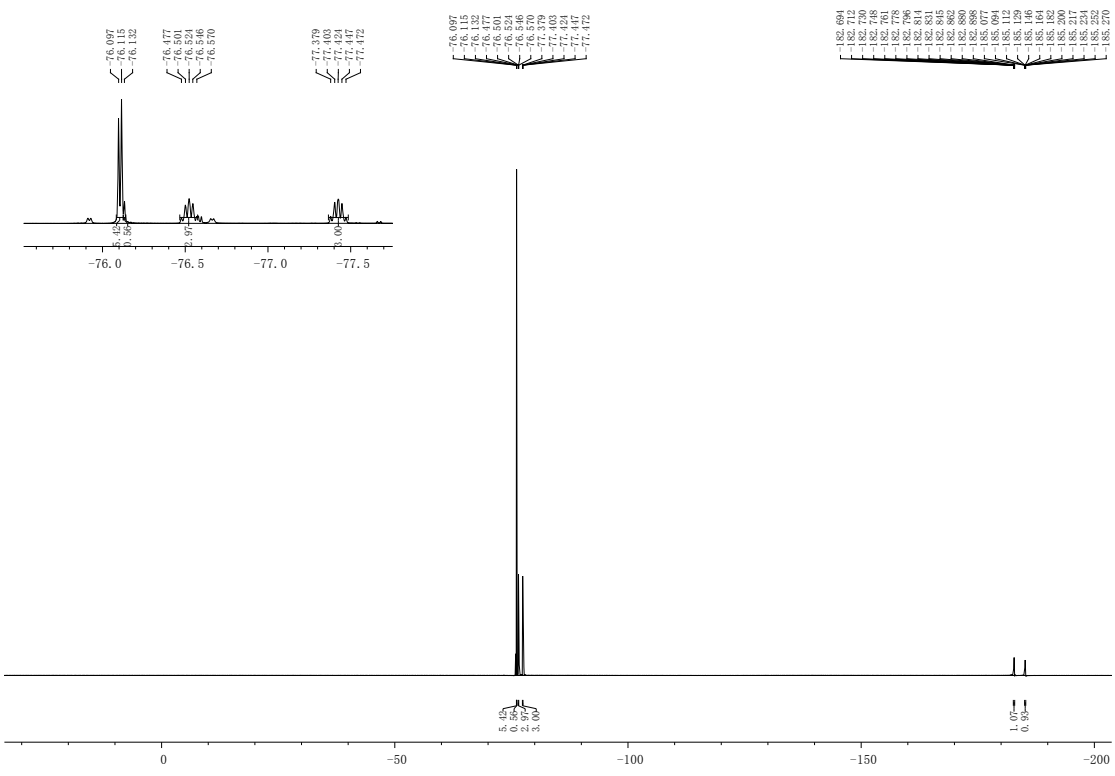
en-3-yl benzoate



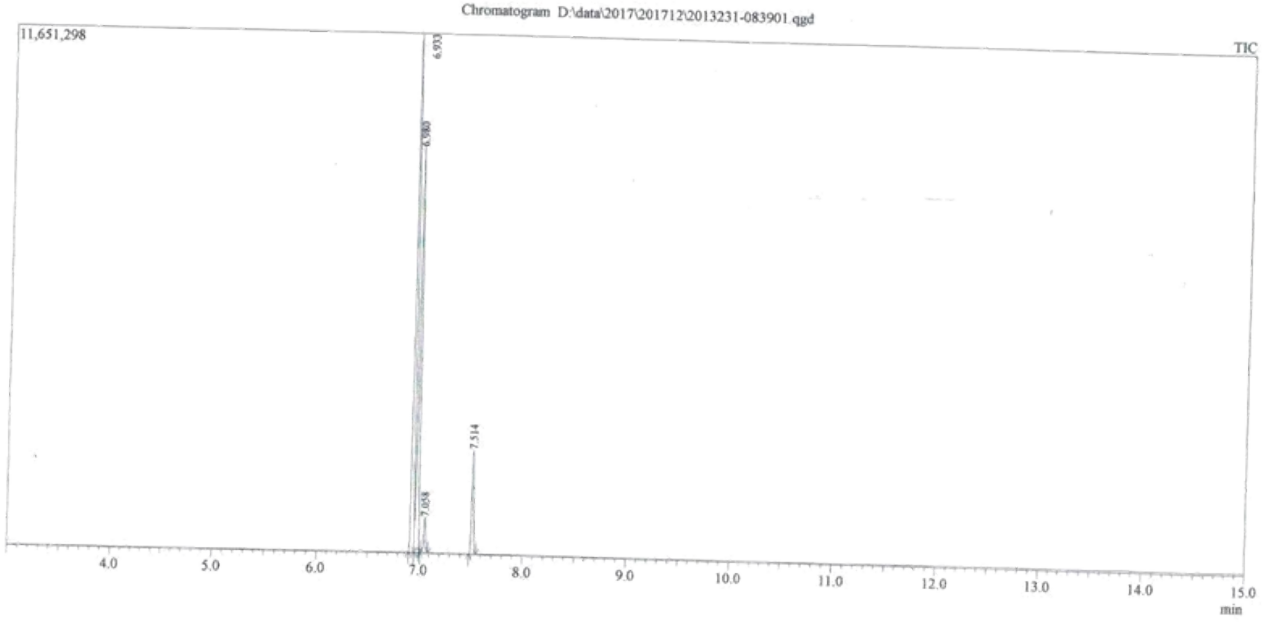
^{1}H NMR spectrum for 7ba + 7bb + 7bc



^{19}F NMR spectrum for 7ba + 7bb + 7bc



GC-MS for 7ba + 7bb + 7bc



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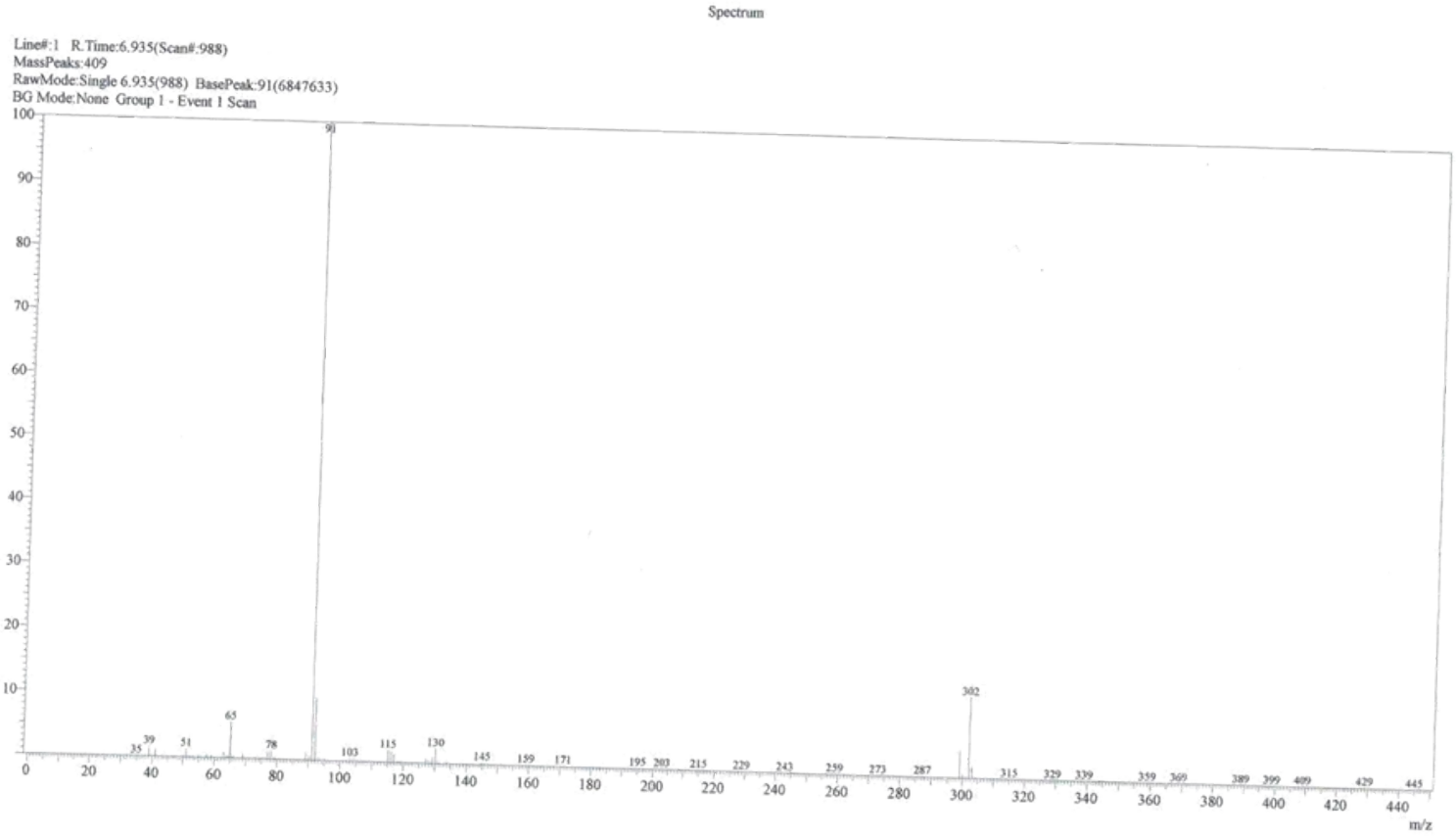
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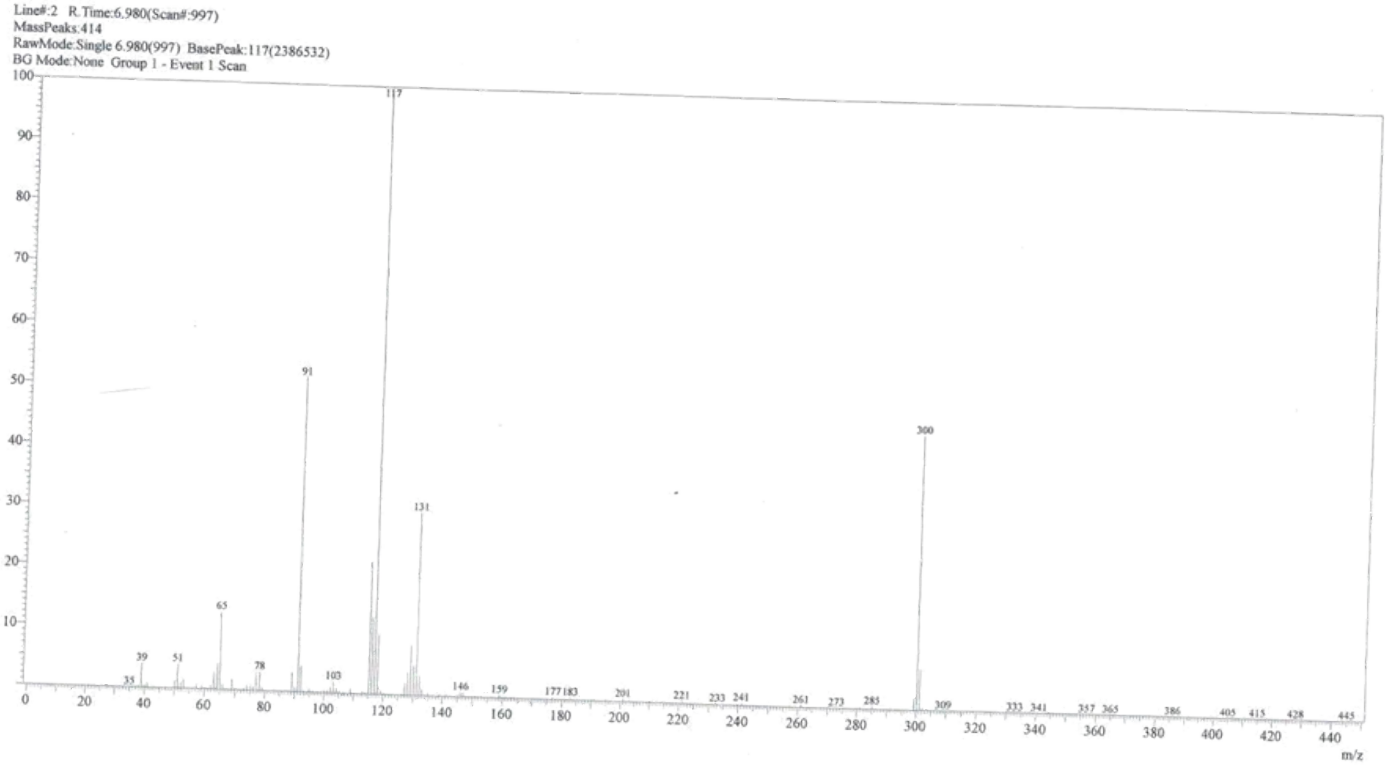
m/z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91
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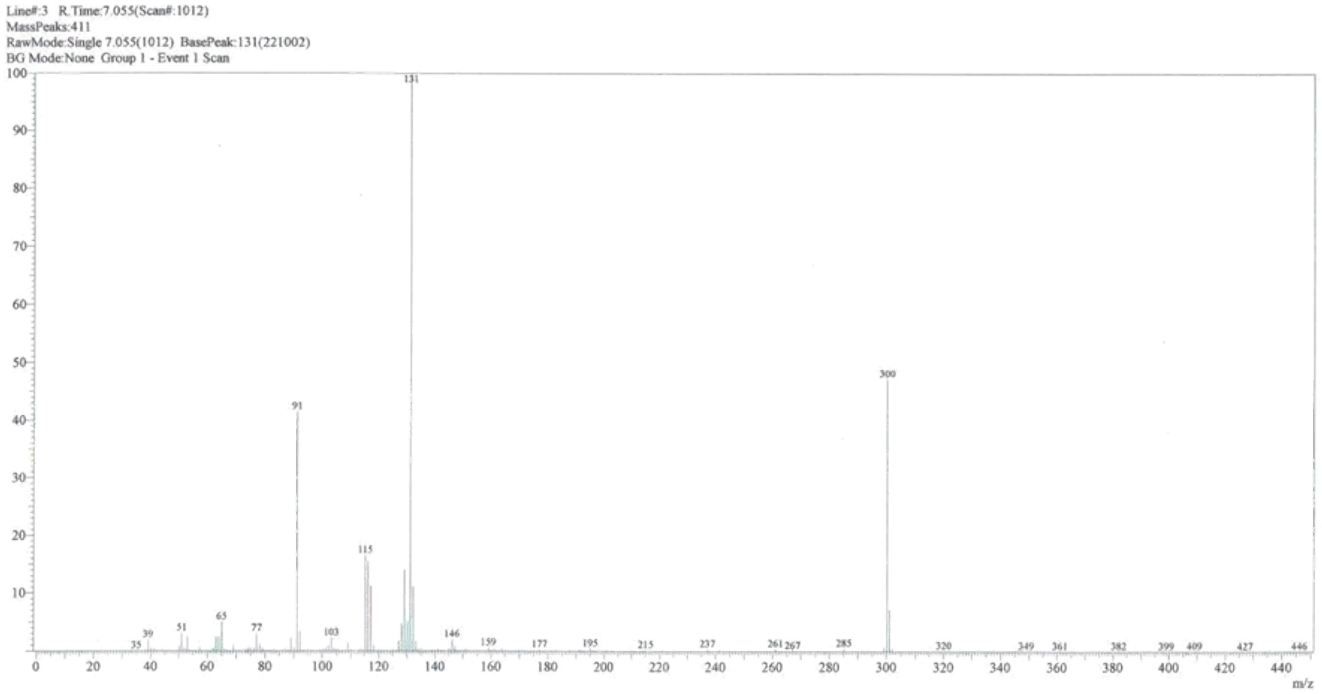
445
m/z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BG Mode:None Group 1 - Event 1 Scan
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300
115
65
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51
77
103
146
159
177
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215
237
261
267
285
320
349
361
382
399
409
427
446
m/z

Line#:3 R.Time:7.055(Scan#:1012)
MassPeaks:411
RawMode:Single 7.055(1012) BasePeak:131(221002)
BG Mode:None Group 1 - Event 1 Scan
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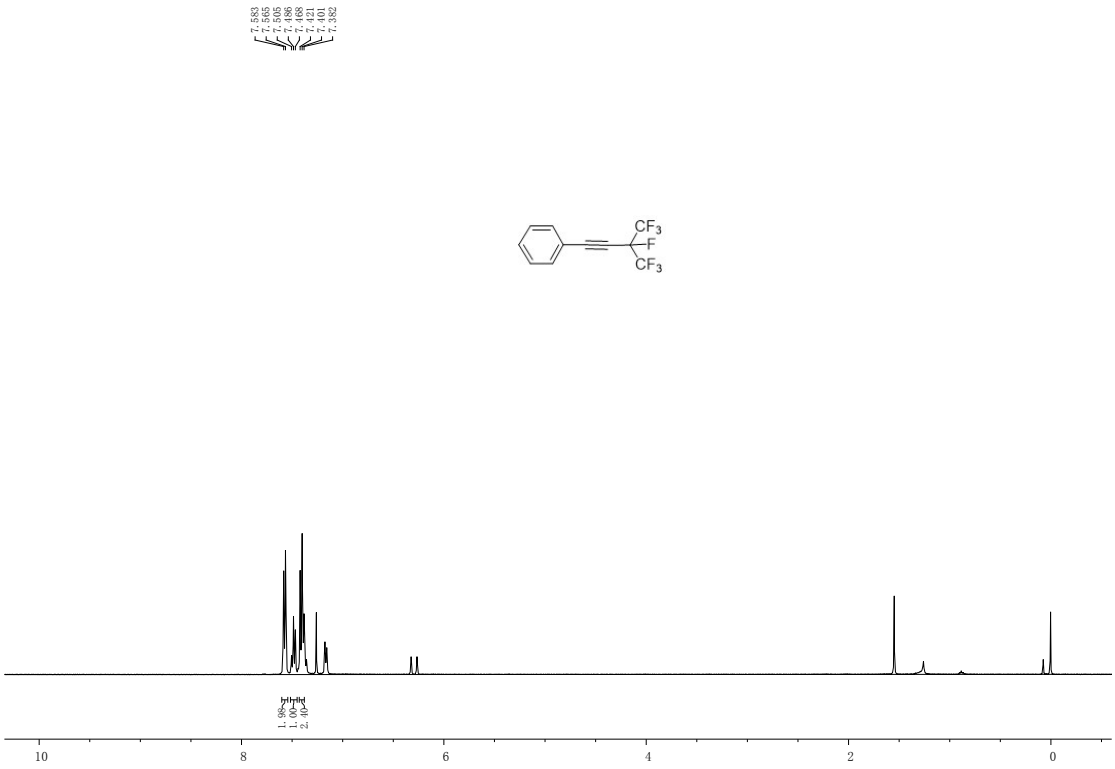
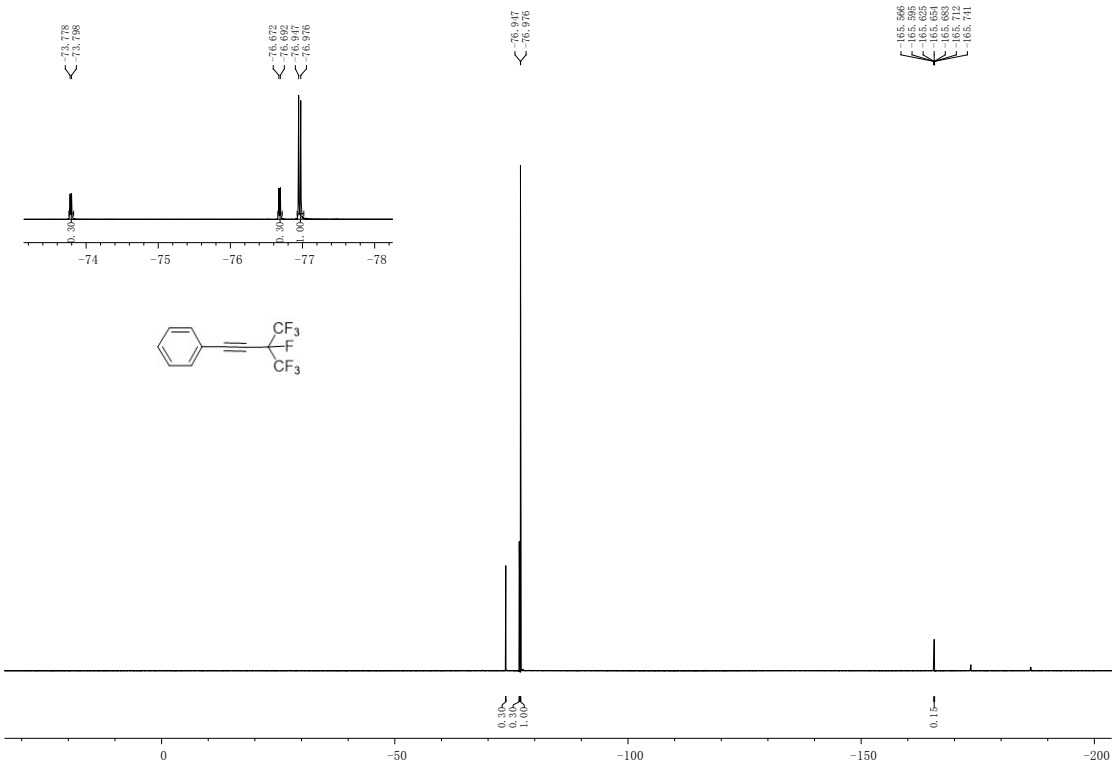
80
70
60
50
40
30
20
10
131
300
91
115
63
0 20 40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440
m/z

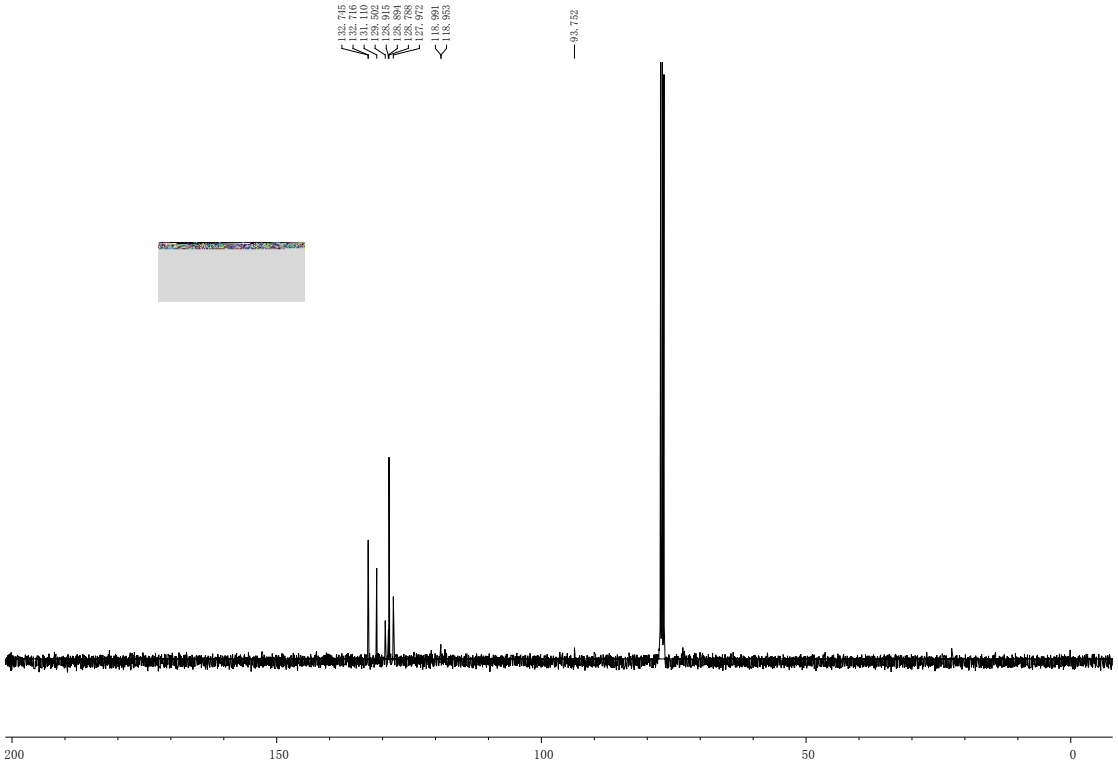
^{1}H NMR spectrum for 9aa: (3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)benzene



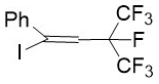
^{19}F NMR spectrum for 9aa: (3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)benzene

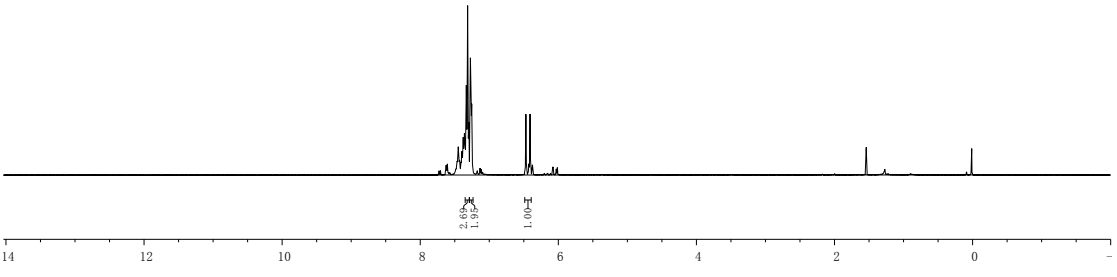


^{13}C NMR spectrum for 9aa: (3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)benzene

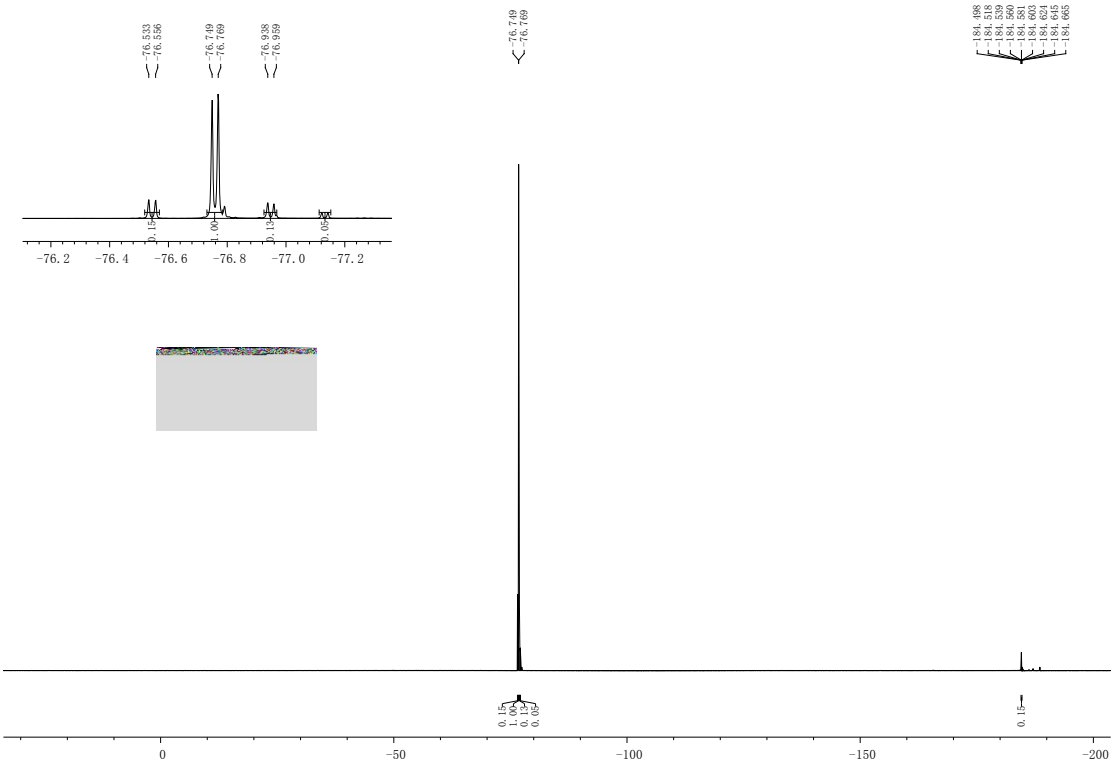


^{1}H NMR spectrum for 9ab: (3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)benzene

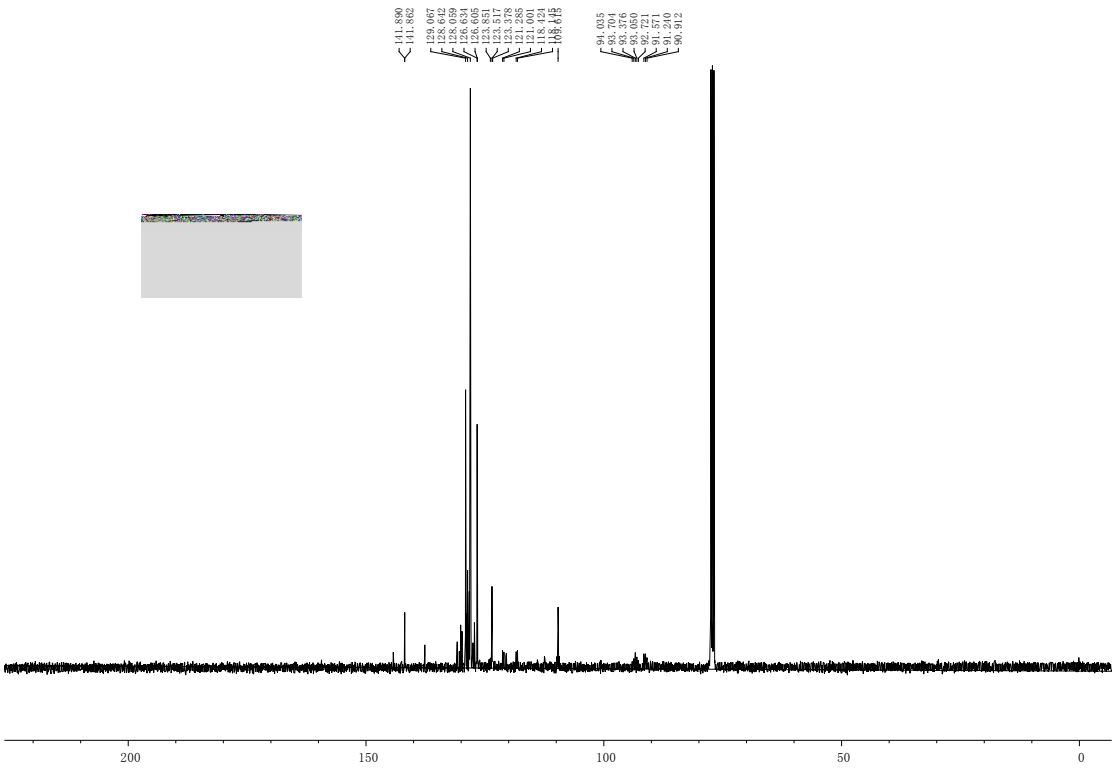




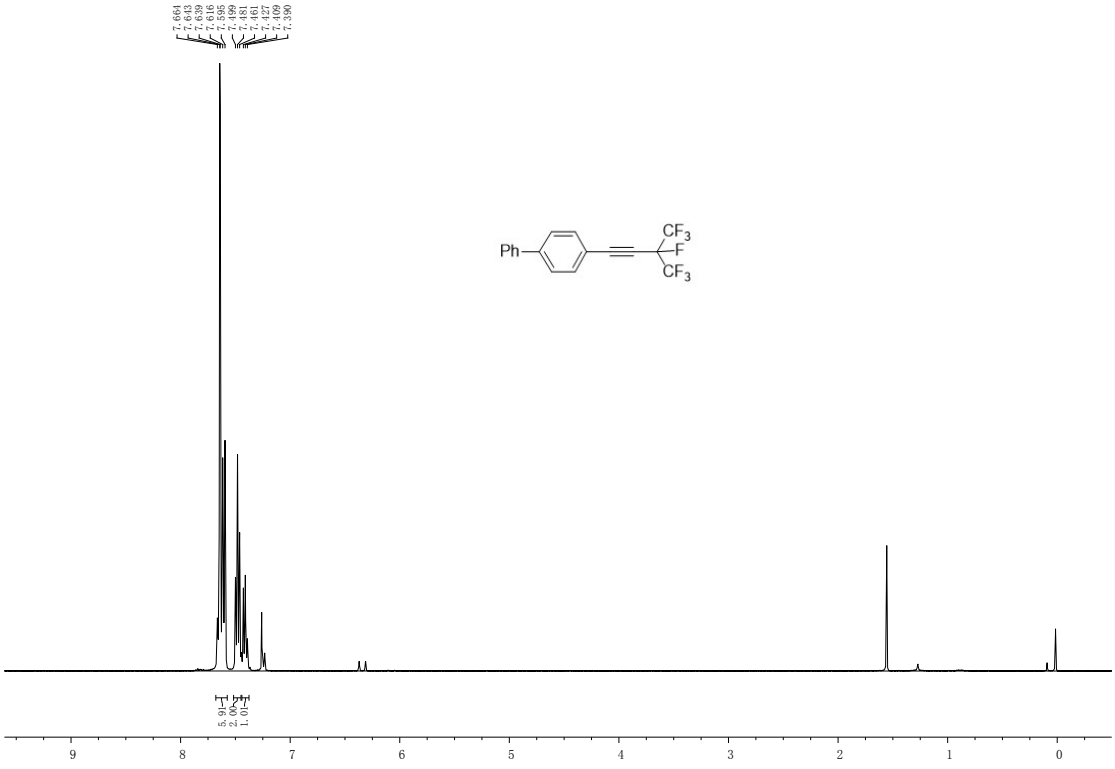
^{19}F NMR spectrum for 9ab: (3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)benzene



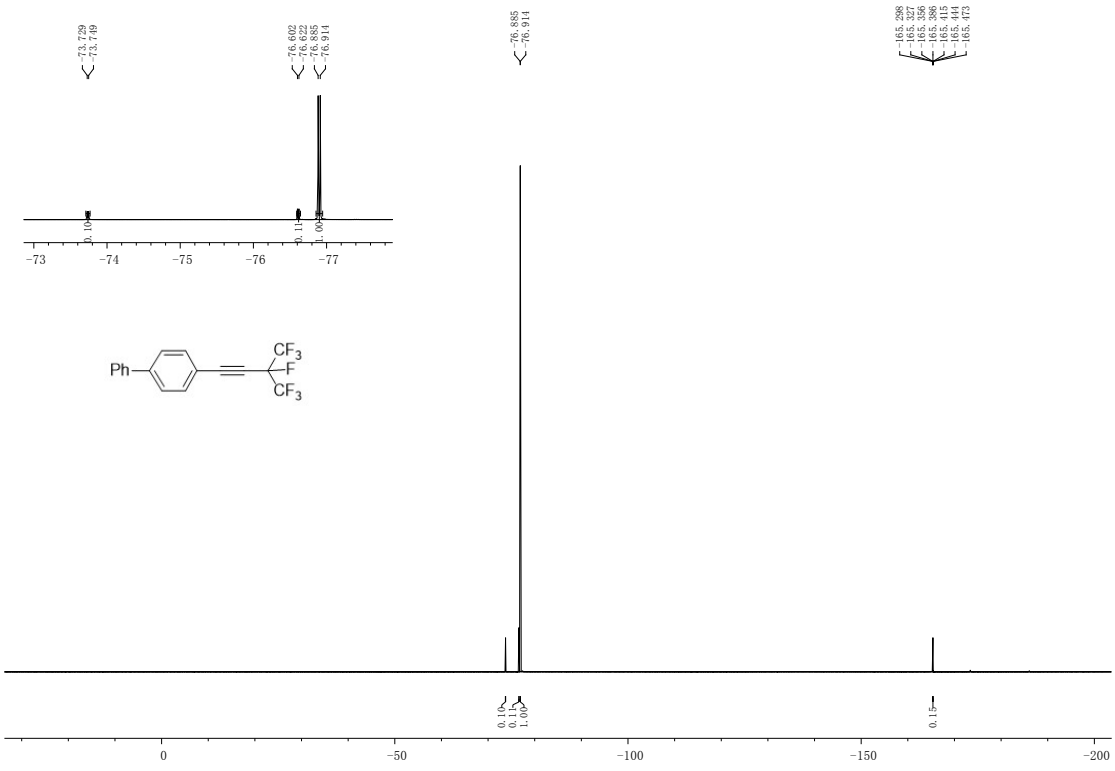
^{13}C NMR spectrum for 9ab: (3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)benzene



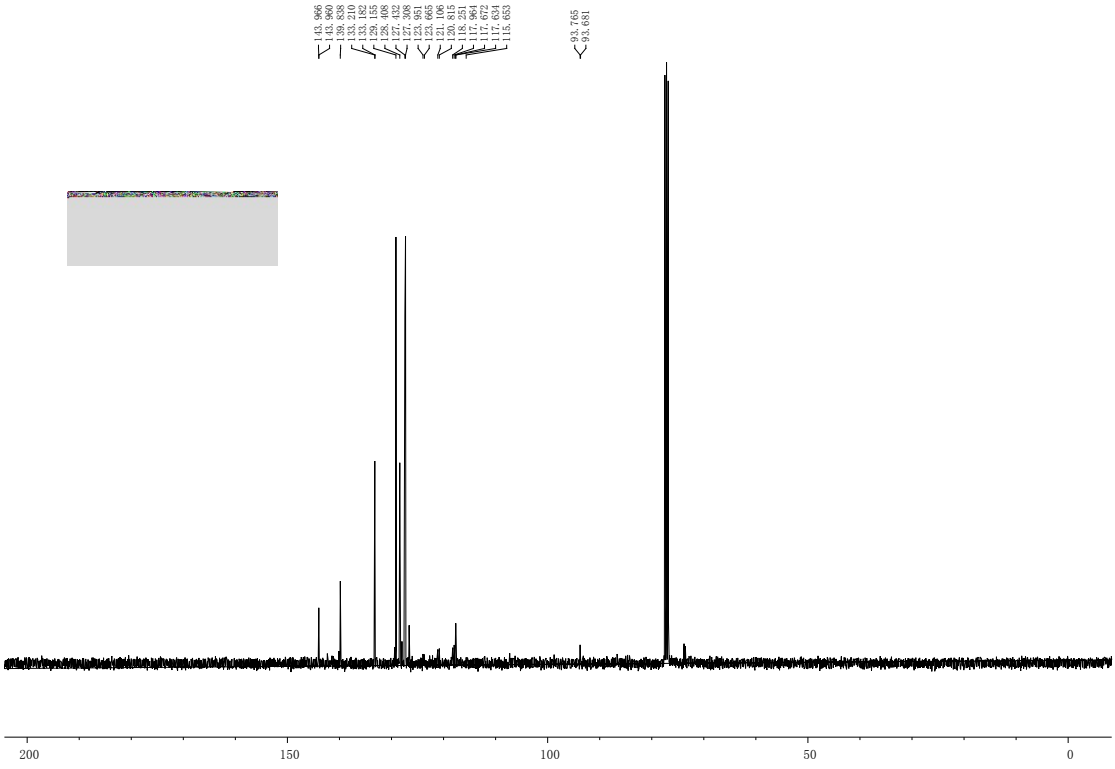
^{1}H NMR spectrum for 9ba: 4-(3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)-1,1'-biphenyl



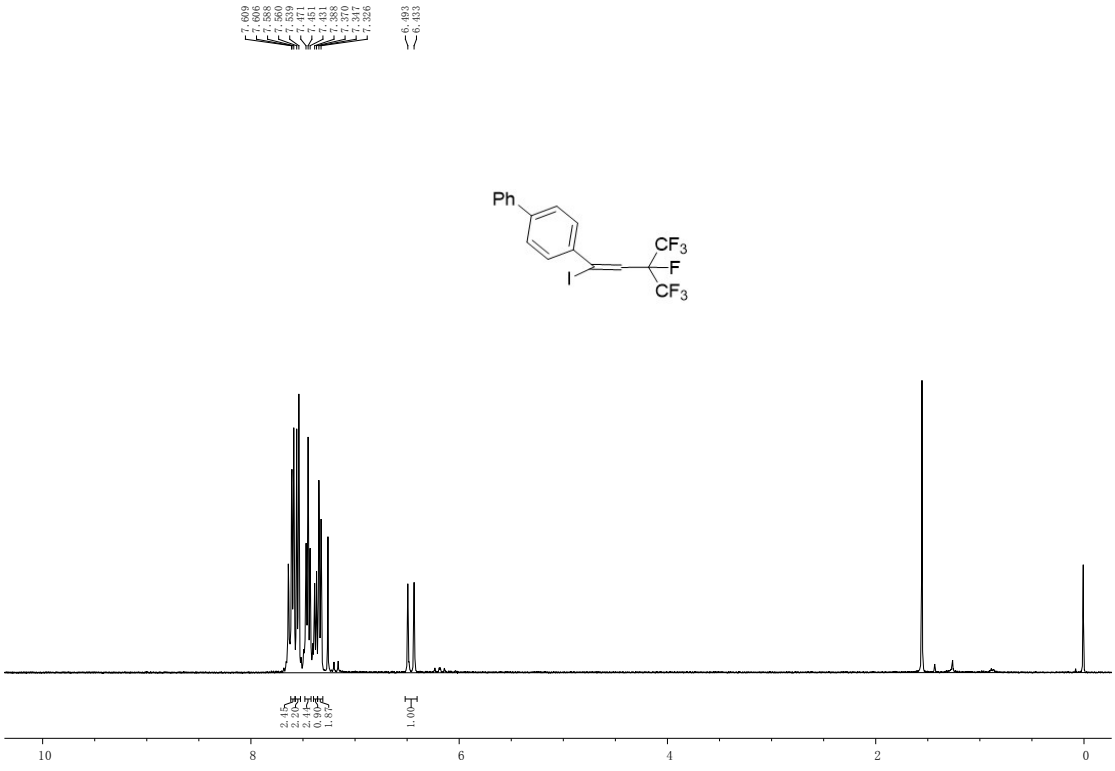
^{19}F NMR spectrum for 9ba: 4-(3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)-1,1'-biphenyl



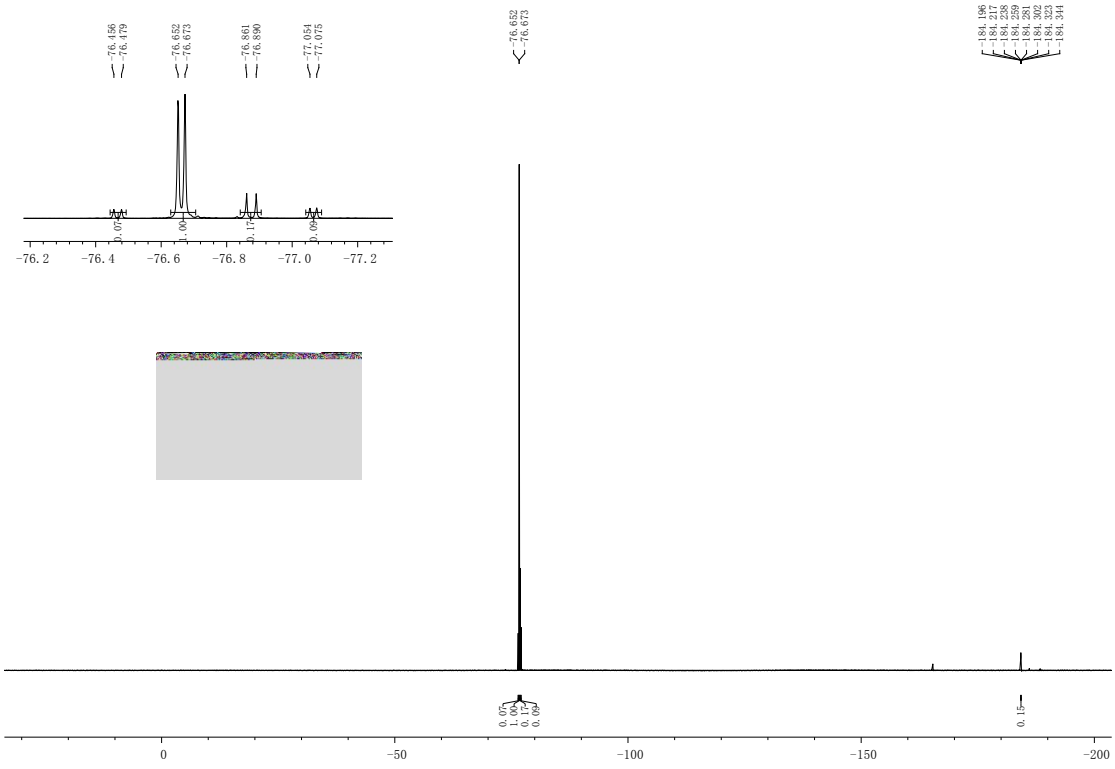
^{13}C NMR spectrum for 9ba: 4-(3,4,4,4-tetrafluoro-3-(trifluoromethyl)but-1-yn-1-yl)-1,1'-biphenyl



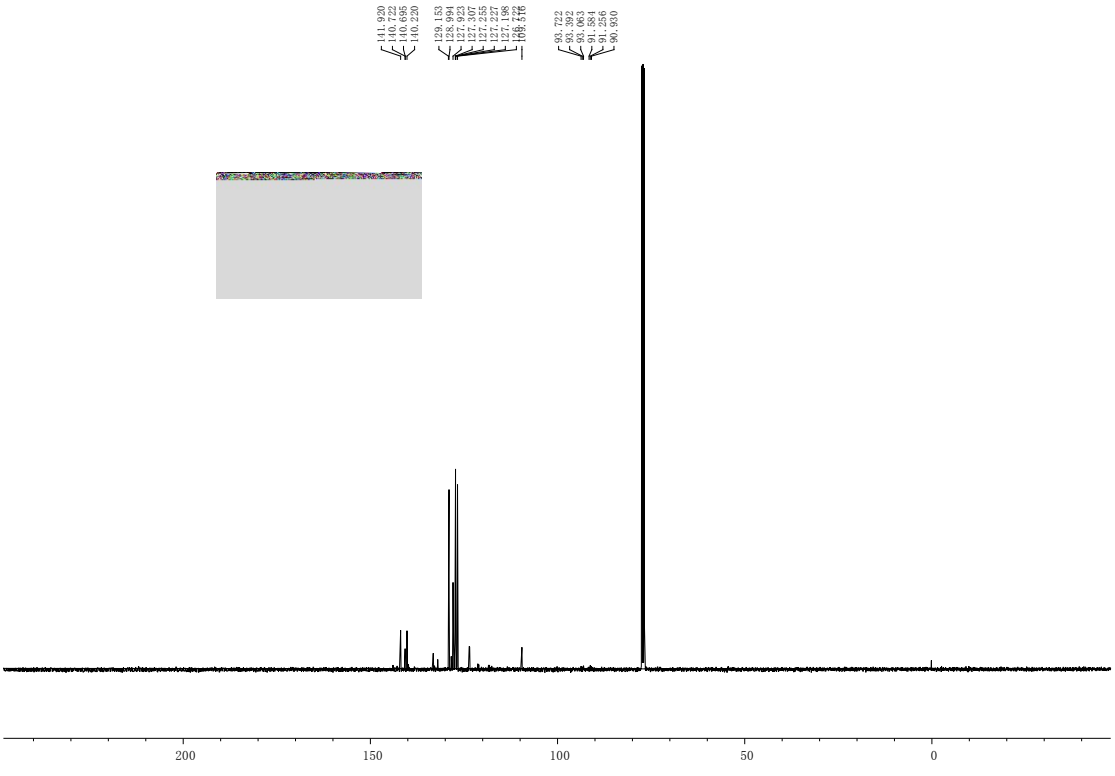
^{1}H NMR spectrum for 9bb: 4-(3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)-1,1'-biphenyl



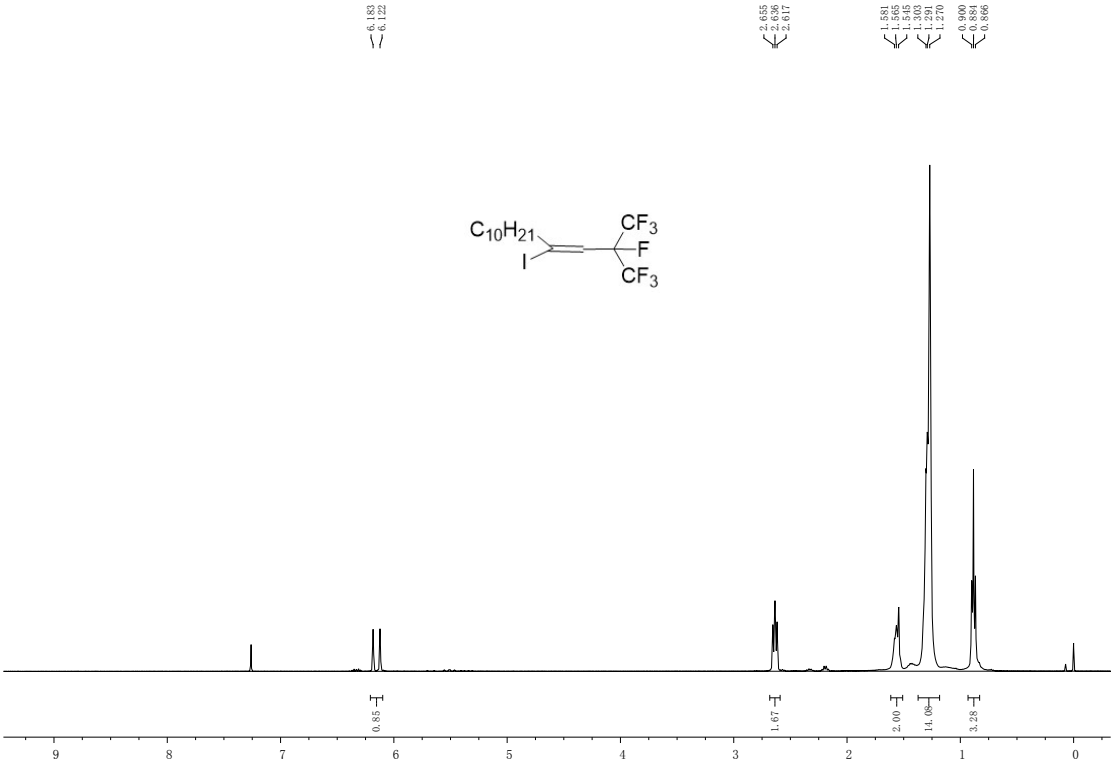
^{19}F NMR spectrum for 9bb: 4-(3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)-1,1'-biphenyl



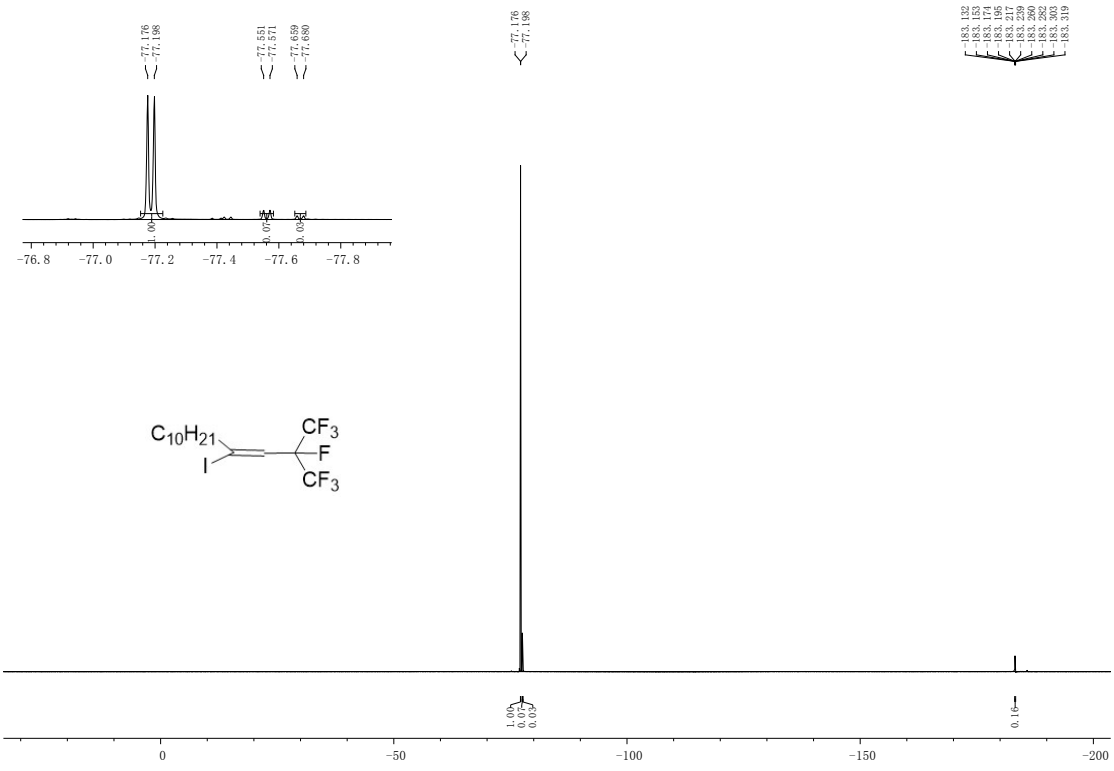
^{13}C NMR spectrum for 9bb: 4-(3,4,4,4-tetrafluoro-1-iodo-3-(trifluoromethyl)but-1-en-1-yl)-1,1'-biphenyl



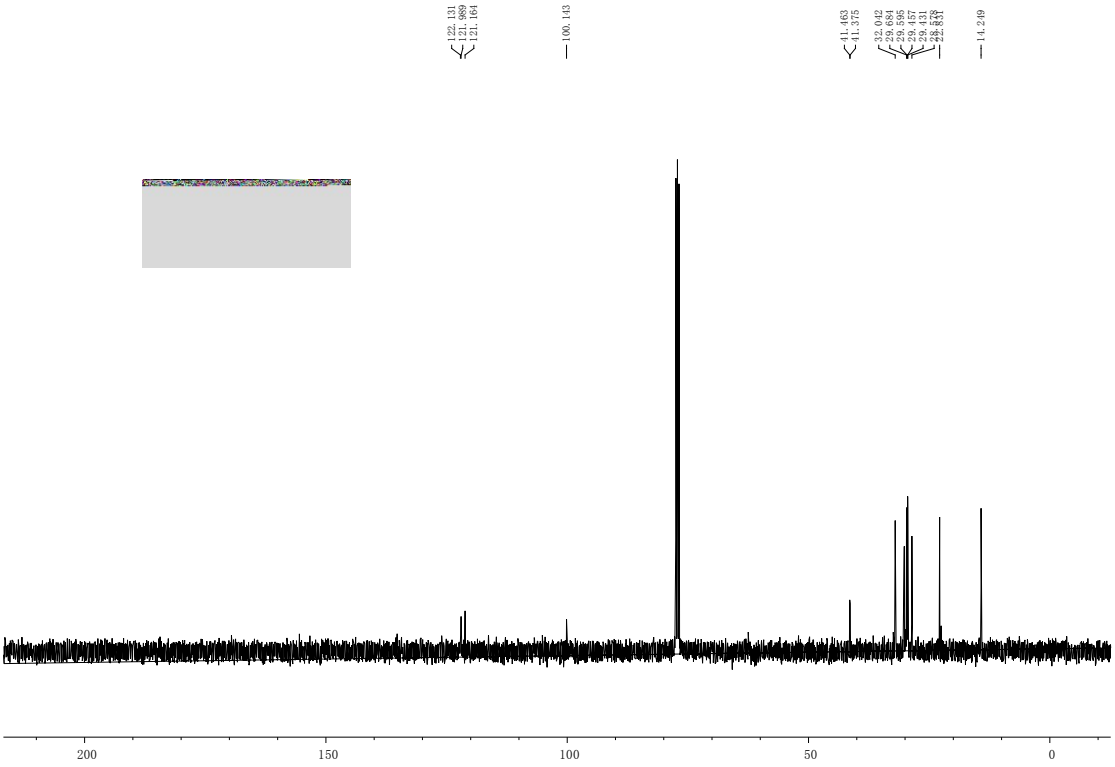
^{1}H NMR spectrum for 9c: 1,1,1,2-tetrafluoro-4-iodo-2-(trifluoromethyl)tetradec-3-ene



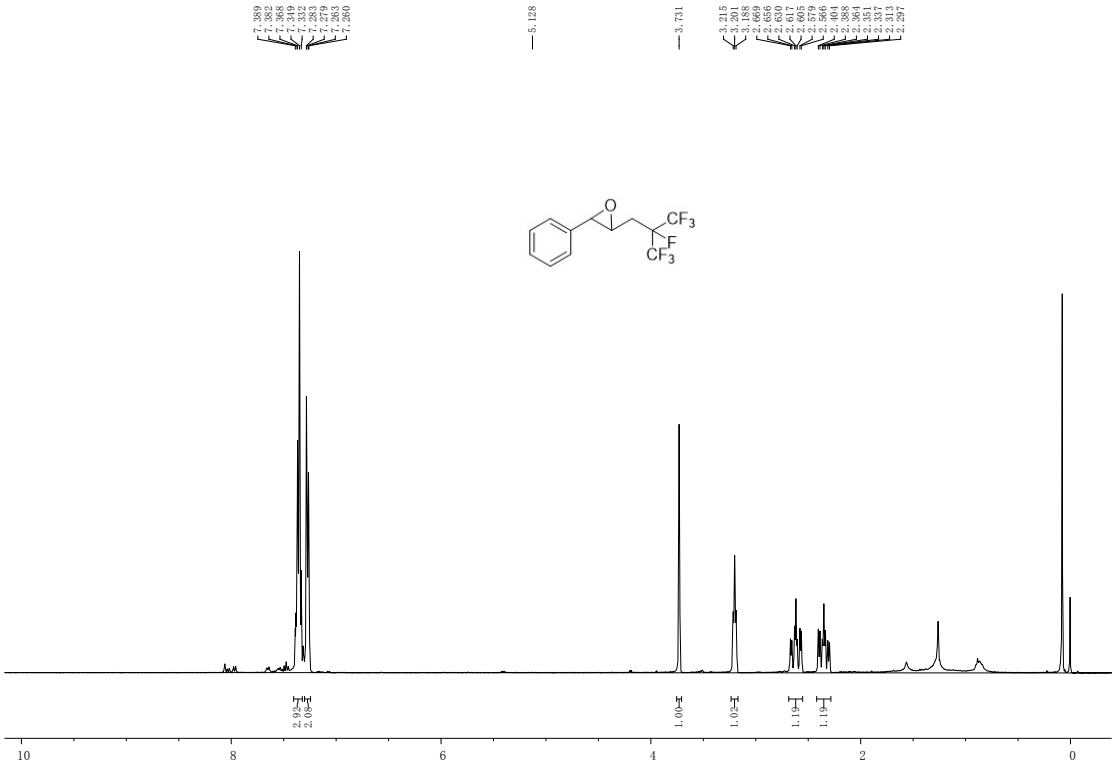
^{19}F NMR spectrum for 9c: 1,1,1,2-tetrafluoro-4-iodo-2-(trifluoromethyl)tetradec-3-ene



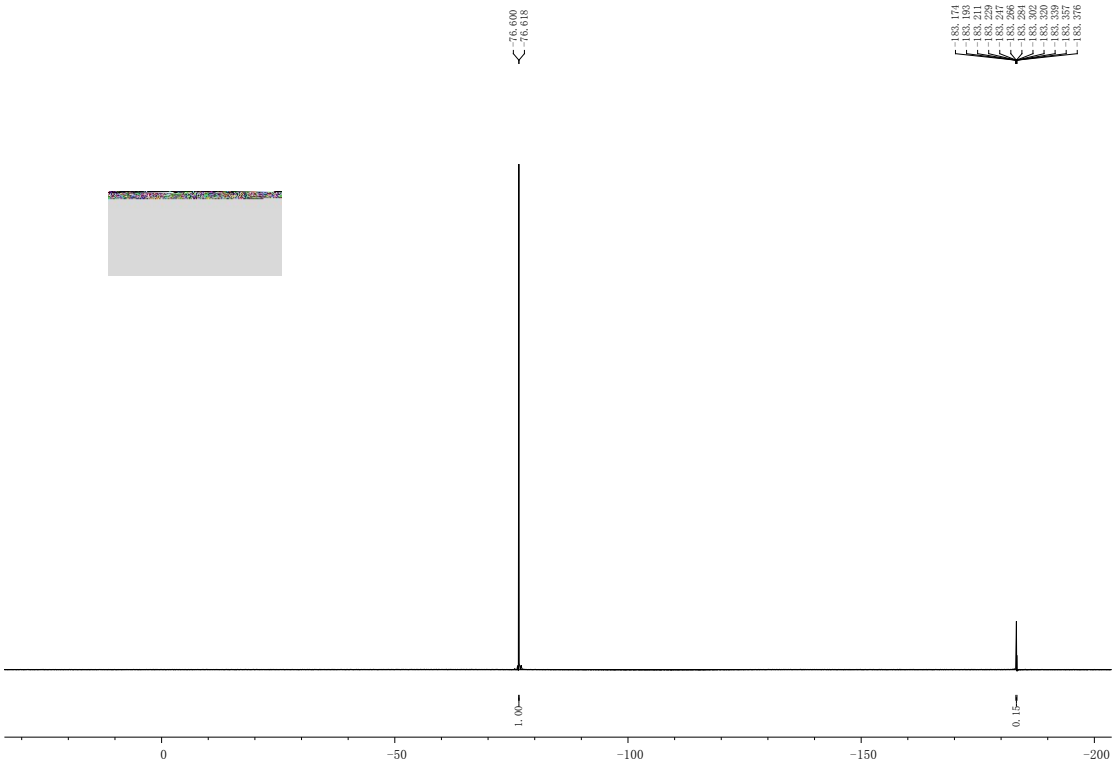
^{13}C NMR spectrum for 9c: 1,1,1,2-tetrafluoro-4-iodo-2-(trifluoromethyl)tetradec-3-ene



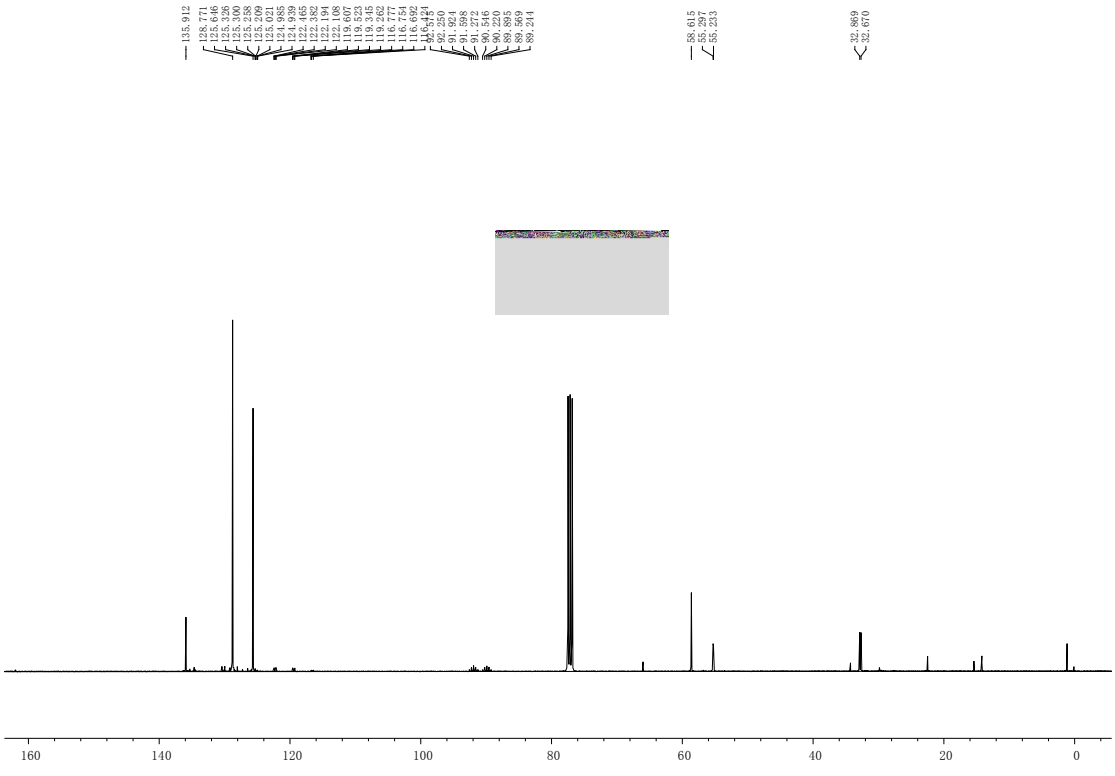
^{1}H NMR spectrum for 10: 2-phenyl-3-(2,3,3,3-tetrafluoro-2-(trifluoromethyl)propyl)oxirane



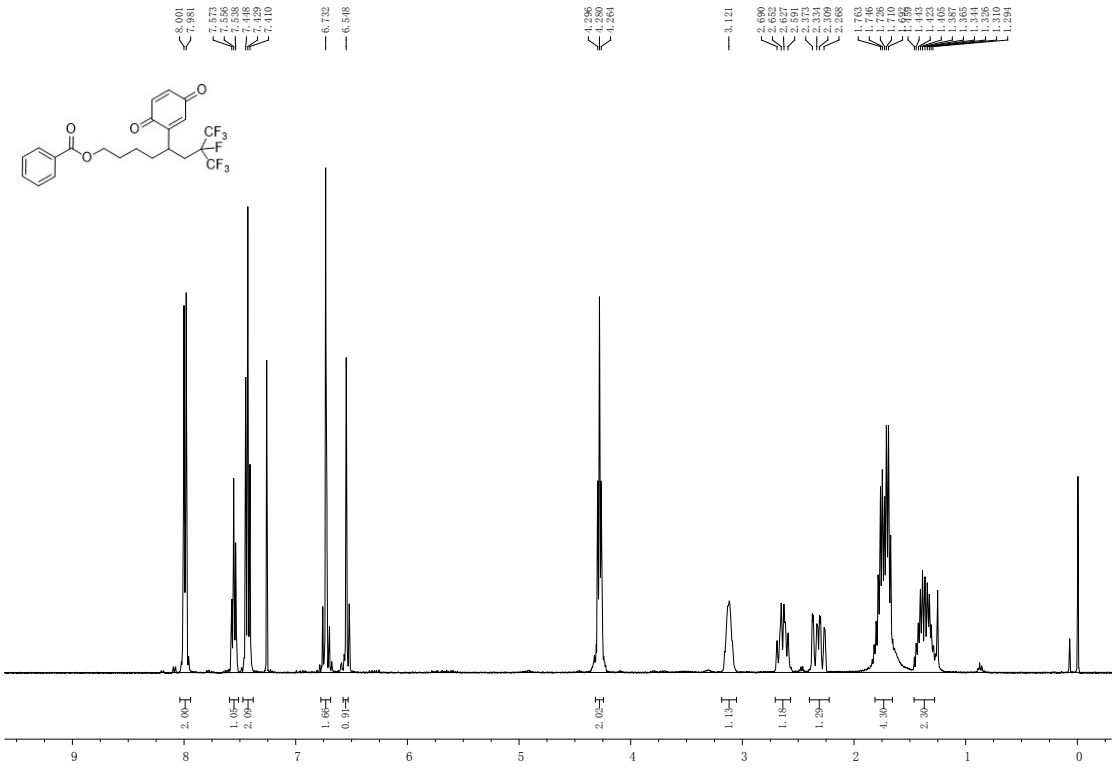
^{19}F NMR spectrum for 10: 2-phenyl-3-(2,3,3,3-tetrafluoro-2-(trifluoromethyl)propyl)oxirane



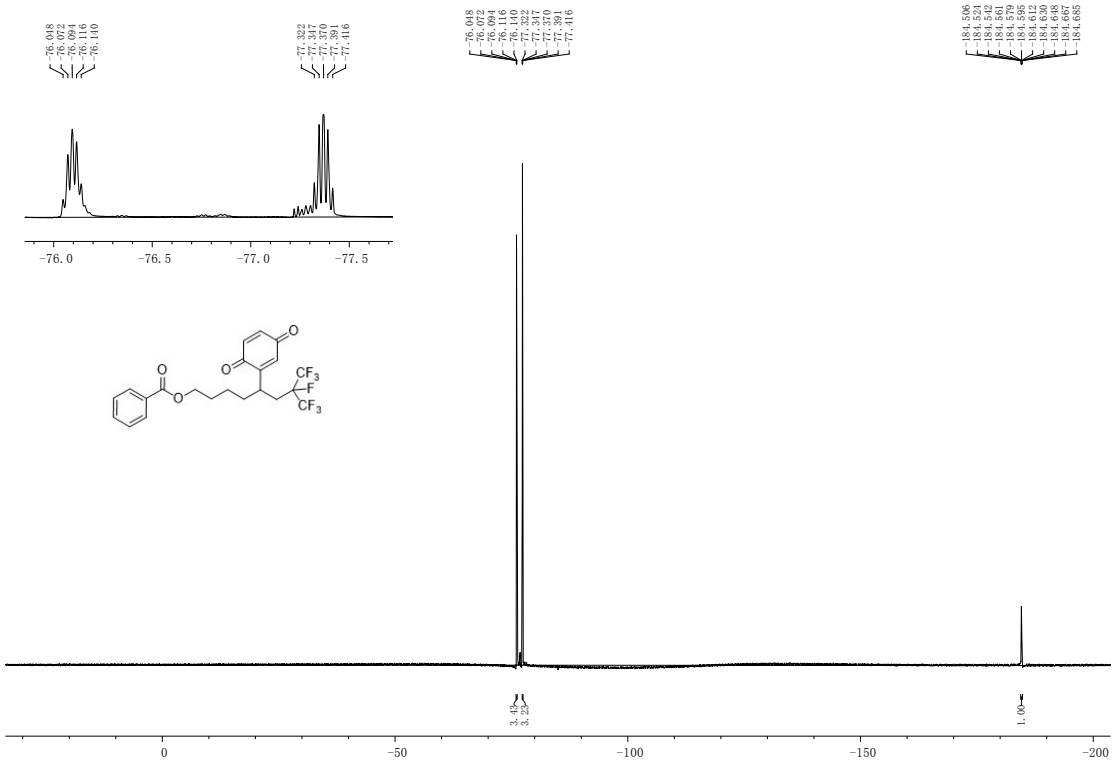
^{13}C NMR spectrum for 10: 2-phenyl-3-(2,3,3,3-tetrafluoro-2-(trifluoromethyl)propyl)oxirane



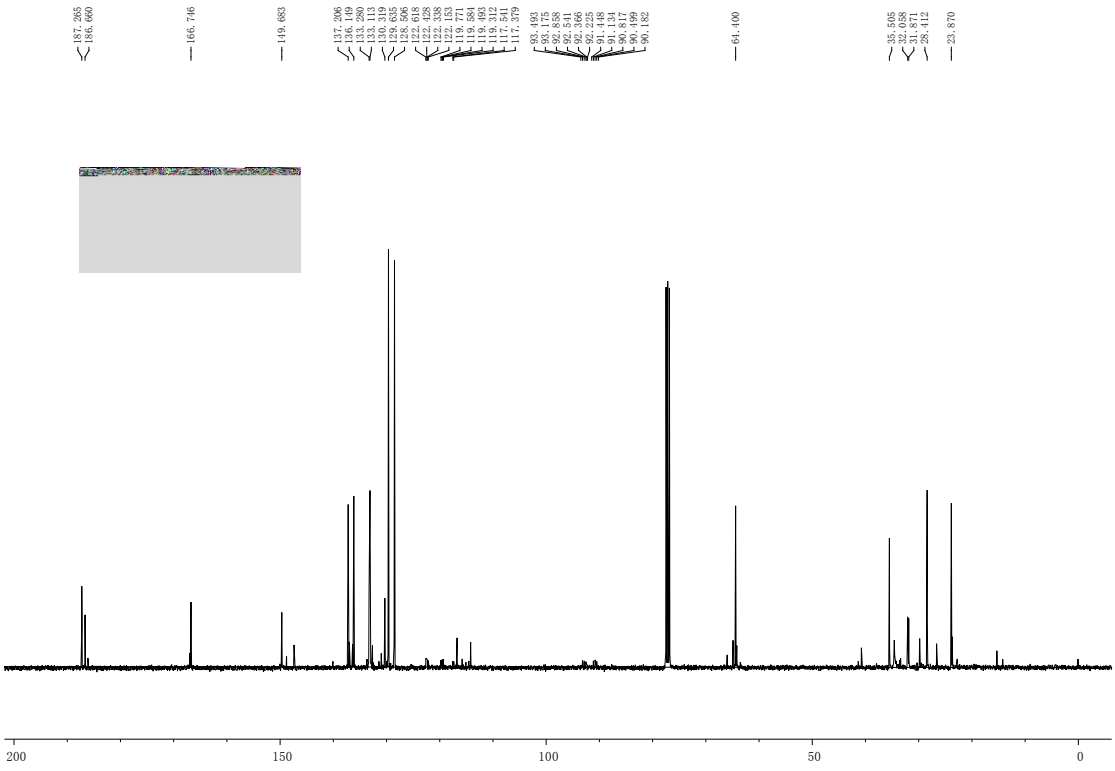
^{1}H NMR spectrum for 11: 5-(3,6-dioxocyclohexa-1,4-dien-1-yl)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)octyl benzoate



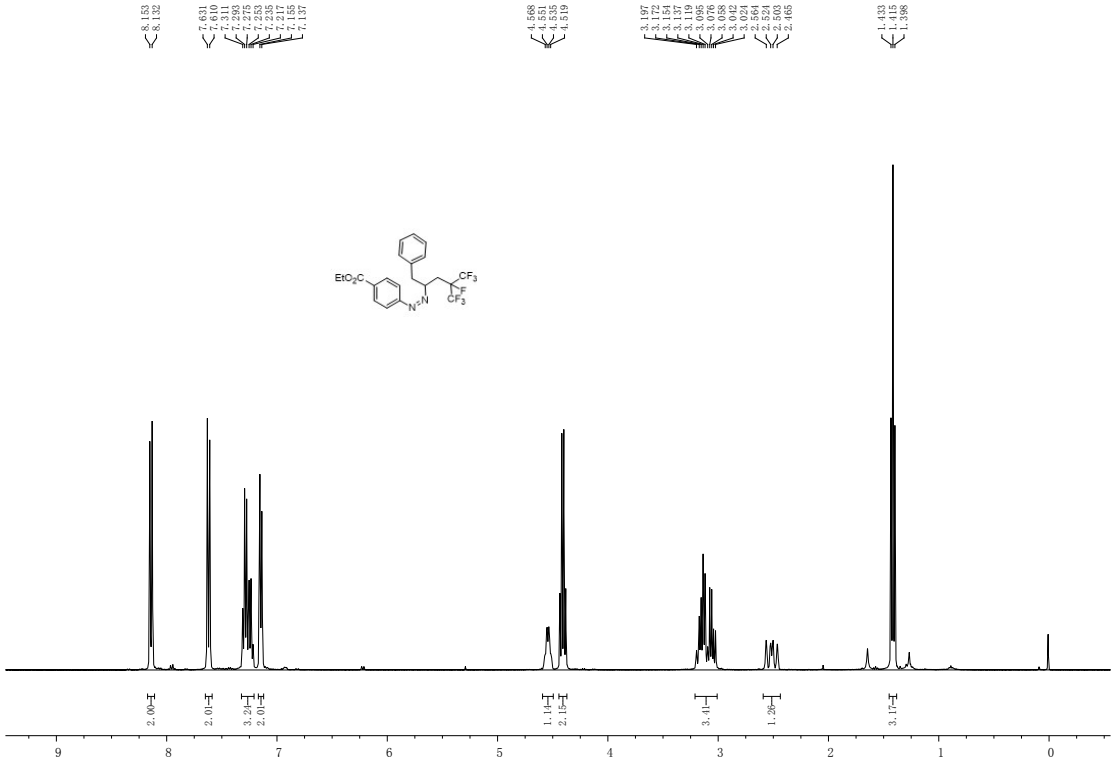
^{19}F NMR spectrum for 11: 5-(3,6-dioxocyclohexa-1,4-dien-1-yl)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)octyl benzoate



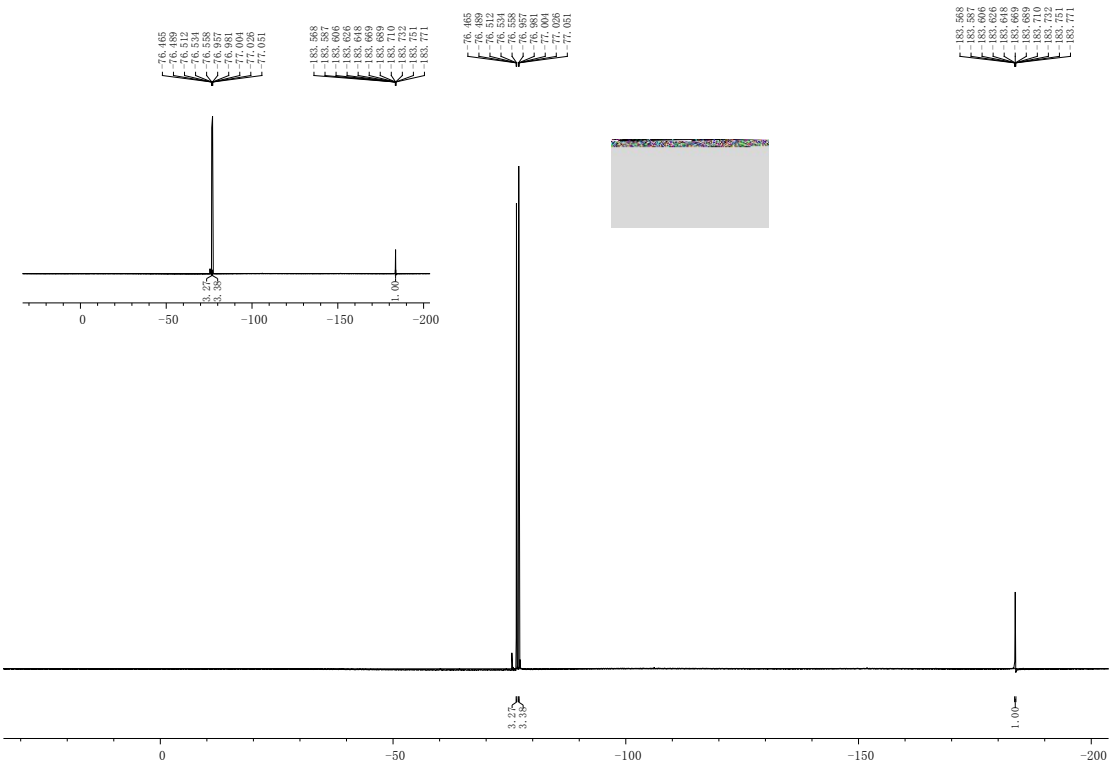
^{13}C NMR spectrum for 11: 5-(3,6-dioxocyclohexa-1,4-dien-1-yl)-7,8,8,8-tetrafluoro-7-(trifluoromethyl)octyl benzoate



^{1}H NMR spectrum for 12: ethyl 4-((4,5,5,5-tetrafluoro-1-phenyl-4-(trifluoromethyl)pentan-2-yl)diazenyl)benzoate



^{19}F NMR spectrum for 12: ethyl 4-((4,5,5,5-tetrafluoro-1-phenyl-4-(trifluoromethyl)pentan-2-yl)diazenyl)benzoate



^{13}C NMR spectrum for 12: ethyl 4-((4,5,5,5-tetrafluoro-1-phenyl-4-(trifluoromethyl)pentan-2-yl)diazenyl)benzoate