Supporting Information

Photoredox-catalyzed three-component carbotrifluoromethylation of alkenes via radical-radical coupling

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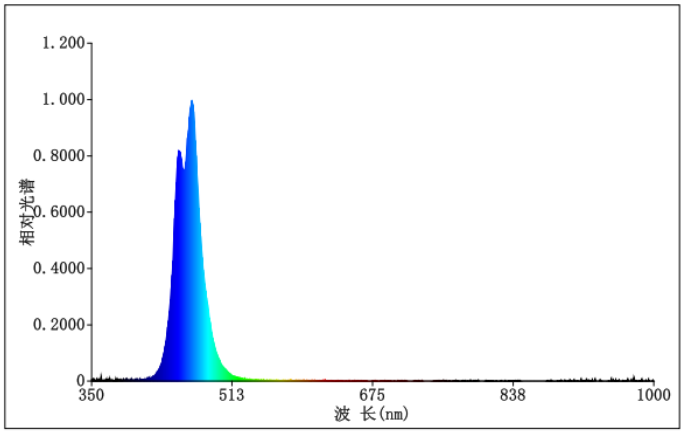
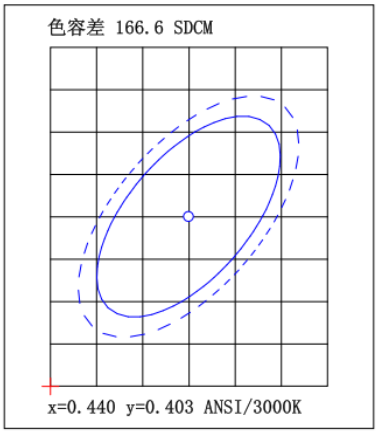
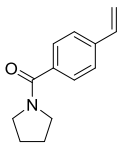
1. General remarks

Proton nuclear magnetic resonance (^{1}H NMR) spectra, carbon nuclear magnetic resonance (^{13}C NMR) spectra and ^{19}F fluorine spectra (^{19}F NMR) were recorded on a JEOL ECZ600R/S3 (^{1}H NMR 600 MHz, ^{13}C NMR 150 MHz, ^{19}F NMR 564 MHz). HRMS were recorded on a MicroMass Waters Xevo G2-XS QTof. GC and MS samples were recorded on an Agilent 7890A-5975C GC-MS system. Fluminescence quenching experiments were recorded on Edinburgh Instruments fluorescence spectrophotometer FLS1000. Unless otherwise indicated, all reagents were purchased commercially without further purification. For the light promoted alkene difunctionalization reaction: use of a blue LEDs panel (40 W, 450-470 nm, manufacturer: Hangzhou Jiadeng Precise Light Source LTD). The distance from the light source to the irradiation glass vial is about 2 cm.

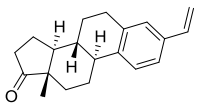




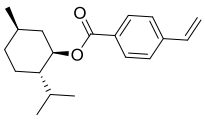
2. Synthesis of alkenes s-16, s-34 and s-35



s-16

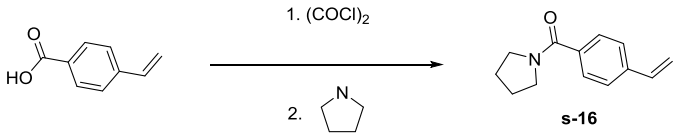


s-34



s-35

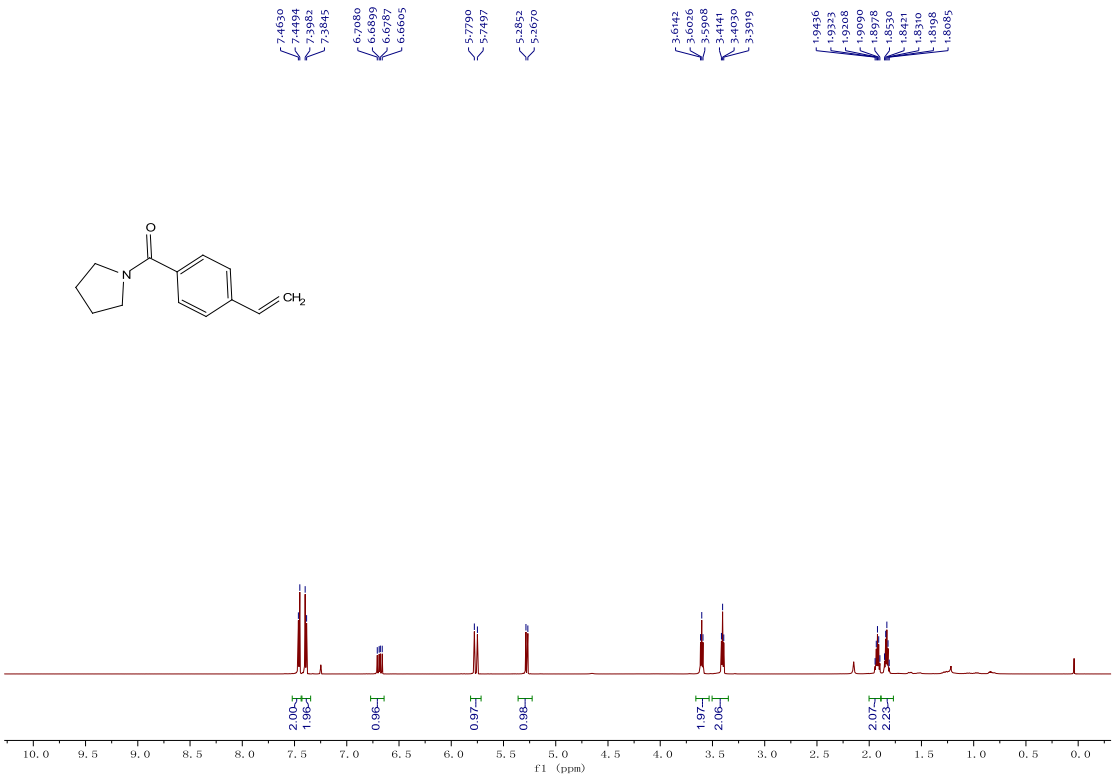
(1) Synthesis of **s-16**



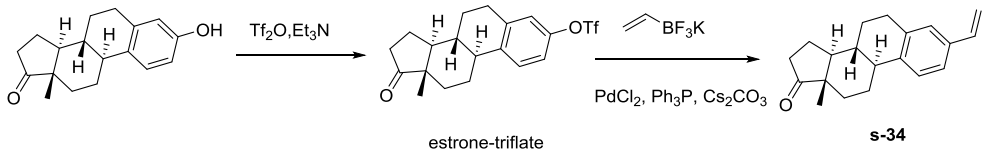
s-16

The compound **s-16** could be prepared according to the reaction procedure: 4-

vinylbenzoic acid (0.74 g, 5 mmol) and a few drops of dimethylformamide were added to CH_2Cl_2 (20 mL), the reaction mixture was cooled in an ice bath, and oxalyl chloride (2.52 g, 20 mmol) was added dropwise. The solution was stirred at room temperature for 4 h. Then the solvent and oxalyl chloride were removed in vacuum to give 4-vinylbenzoyl chloride. Pyrrolidine (0.35 g, 5 mmol) and Et_3N (2.02 g, 20 mmol) were added to CH_2Cl_2 (20 mL). The reaction mixture was cooled in an ice bath, and the as-prepared 4-vinylbenzoyl chloride was added dropwise. The solution was stirred at room temperature for 6 h. The solvent was then evaporated under reduced pressure. The residue was further purified by flash column chromatography using petroleum ether/ethyl acetate as eluant to afford **s-16** as a white solid (0.94 g, 94%). ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.46-7.45 (d, J = 8.2 Hz, 2H), 7.40-7.38 (d, J = 8.2 Hz, 2H), 6.71-6.66 (dd, J = 17.6, 11.0 Hz, 1H), 5.78-5.74 (d, J = 17.6 Hz, 1H), 5.29-5.27 (d, J = 11.0 Hz, 1H), 3.61-3.59 (t, J = 6.9 Hz, 2H), 3.41-3.39 (t, J = 6.8 Hz, 2H), 1.94-1.90 (m, 2H), 1.84-1.81 (m, 2H) (this compound was known, see the ref. *J. Am. Chem. Soc.* **2021**, *143*, 10524).

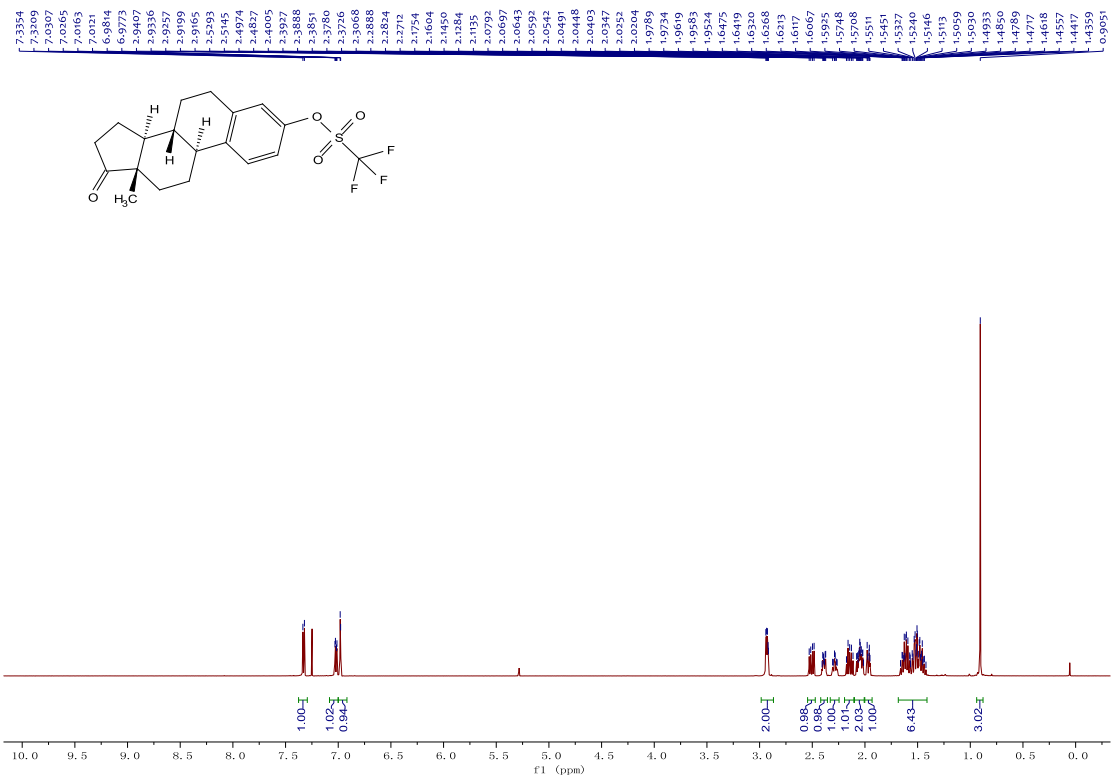


(2) Synthesis of **s-34**



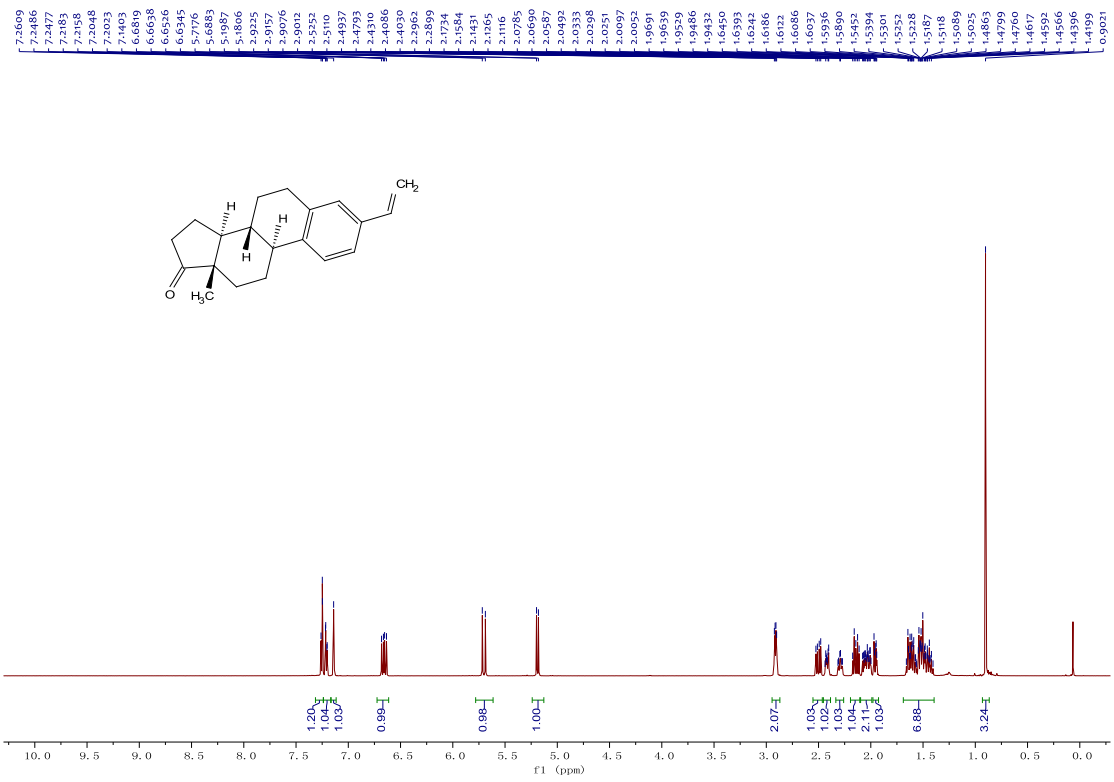
The compound estrone-triflate could be prepared according to the reaction procedure: estrone (0.54 g, 2 mmol) and Et_3N (0.40 g, 4 mmol) were added to CH_2Cl_2 (10 mL), the reaction mixture was cooled in an ice bath, and trifluoromethanesulfonic

anhydride (0.84 g, 3 mmol) was added dropwise. The solution was stirred at room temperature for 8 h. Then a saturated aqueous solution of $NaHCO_3$ was added to the reaction mixture, and the resulted mixture was extracted with ethyl acetate. Combined organic fractions were dried, followed by concentration under reduced pressure. The obtained residue was further purified by flash column chromatography using petroleum ether/ethyl acetate as eluant to afford estrone-triflate as a yellow solid (0.72 g, 90%). ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.34-7.32 (d, J = 8.7 Hz, 1H), 7.03-7.01 (dd, J = 8.7, 2.5 Hz, 1H), 6.98 (d, J = 2.5 Hz, 1H), 2.94-2.92 (m, 2H), 2.53-2.48 (dd, J = 19.1, 8.9 Hz, 1H), 2.41-2.37 (m, 1H), 2.31-2.26 (m, 1H), 2.18-2.11 (m, 1H), 2.08-2.01 (m, 2H), 1.98-1.95 (m, 1H), 1.66-1.42 (m, 6H), 0.91 (s, 3H) (this compound was known, see the ref. *J. Am. Chem. Soc.* **2021**, *143*, 10760).

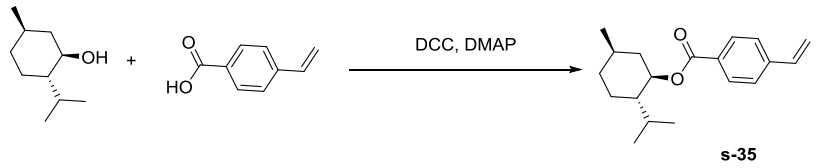


The compound **s-34** could be prepared according to the reaction procedure: To an oven-dried glass vial was added estrone-triflate (603 mg, 1.5 mmol), potassium vinyltrifluoroborate (402 mg, 3 mmol), $PdCl_2$ (26.6 mg, 0.15 mmol), PPh_3 (59 mg, 0.225 mmol) and Cs_2CO_3 (1466 mg, 4.5 mmol). The tube was evacuated and back-filled with nitrogen, which was repeated three times. THF (3 mL) and H_2O (0.5 mL) were subsequently added to the tube via syring. The resulting mixture was stirred at 85 ^{o}C for 20 h. The mixture was extracted with ethyl acetate and concentrated under reduced pressure. The resulting residue was further purified by flash column chromatography using petroleum ether/ethyl acetate as eluant to afford **s-34** as a white solid (327.7 mg, 78%). ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.26-7.25 (d, J = 7.9 Hz, 1H), 7.22-7.20 (d, J = 8.0 Hz, 1H), 7.14 (s, 1H), 6.68-6.63 (dd, J = 17.6, 10.9 Hz, 1H), 5.72-5.69 (d, J = 17.6 Hz, 1H), 5.20-5.18 (d, J = 11.0 Hz, 1H), 2.92-2.90 (m, 2H),

2.52-2.48 (dd, *J* = 19.2, 8.9 Hz, 1H), 2.44-2.40 (m, 1H), 2.31-2.27 (m, 1H), 2.17-2.11 (m, 1H), 2.08-2.00 (m, 2H), 1.97-1.94 (m, 1H), 1.66-1.40 (m, 6H), 0.90 (s, 3H) (this compound was known, see the ref. *J. Am. Chem. Soc.* **2021**, *143*, 10760).

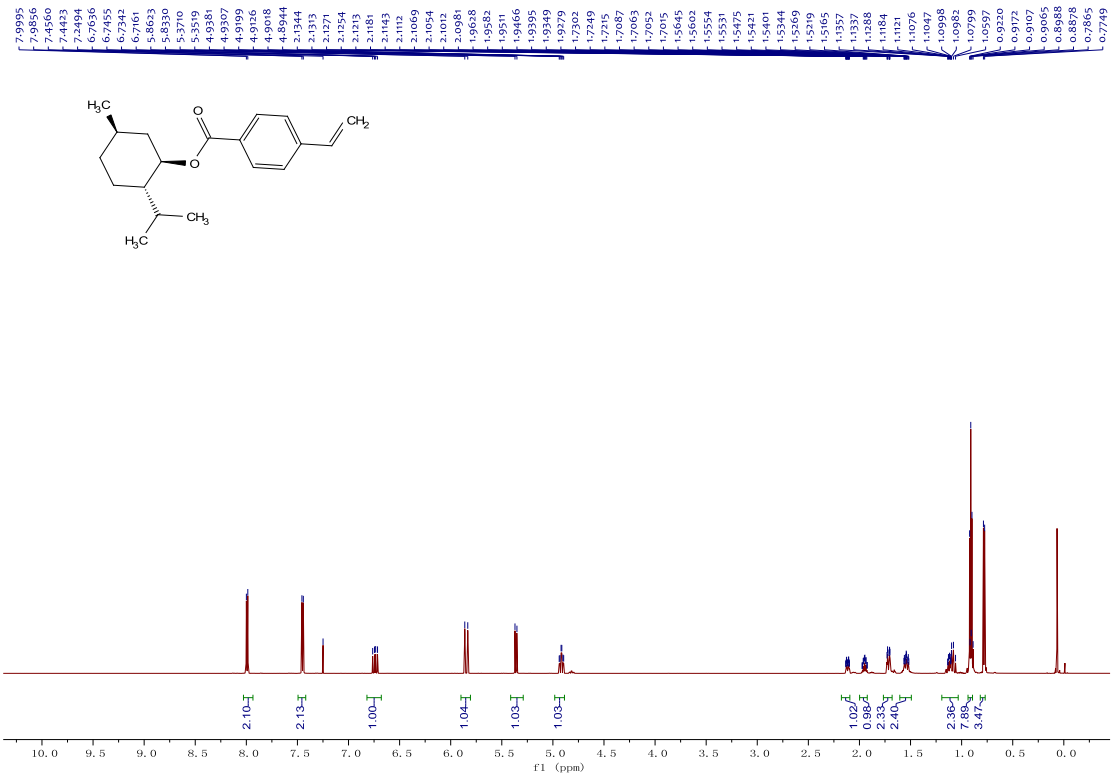


(3) Synthesis of **s-35**

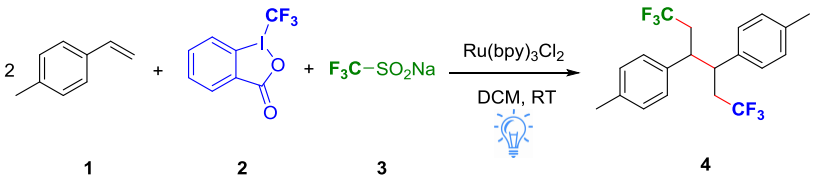


s-35

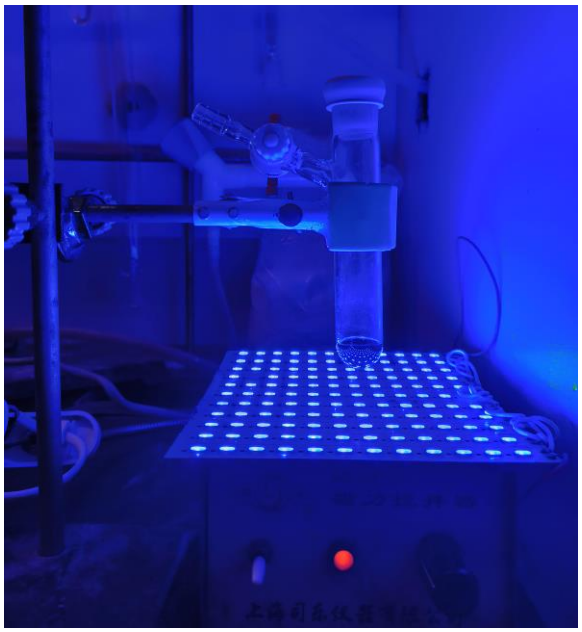
The compound **s-35** could be prepared according to the reaction procedure: 4-vinylbenzoic acid (0.74 g, 5 mmol), 1,3-dicyclohexyl-carbodiimide (1.24 g, 6 mmol) and DMAP (0.06 g, 0.5 mmol) were added to CH_2Cl_2 (25 mL), and menthol (0.78 g, 5 mmol) was added dropwise. The solution was stirred at room temperature for 24 h. The solvent was then evaporated under reduced pressure. The resulted residue was further purified by flash column chromatography using petroleum ether/ethyl acetate as eluant to afford **s-35** as a colorless oil (1.06 g, 74%). ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 8.00-7.99 (d, *J* = 8.3 Hz, 2H), 7.46-7.44 (d, *J* = 8.4 Hz, 2H), 6.76-6.72 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.86-5.83 (d, *J* = 17.6 Hz, 1H), 5.37-5.35 (d, *J* = 10.9 Hz, 1H), 4.94-4.89 (td, *J* = 10.9, 4.4 Hz, 1H), 2.13-2.10 (m, 1H), 1.97-1.92 (m, 1H), 1.73-1.70 (m, 2H), 1.56-1.52 (m, 2H), 1.14-1.06 (m, 2H), 0.92-0.89 (m, 7H), 0.79-0.77 (d, *J* = 6.9 Hz, 3H) (this compound was known, see the ref. *J. Org. Chem.* **2023**, *88*, 16091).



3. Typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation



To an oven-dried 10 mL glass vial was added Togni reagent **2** (126 mg, 0.40 mmol), Langlois reagent **3** (62.4 mg, 0.40 mmol) and $Ru(bpy)_3Cl_2$ (2.6 mg, 2 mol%). The tube was evacuated and back-filled with nitrogen, which was repeated three times. 4-Methylstyrene **1** (47.2 mg, 0.40 mmol) and DCM (1 mL) were then added to the tube via syring. The resulting mixture was stirred at room temperature for 20 h with 40W blue LEDs (450-470 nm) irradiation. After the reaction completion, the mixture was concentrated under reduced pressure. The resulting residue was further purified by flash column chromatography using petroleum ether as eluant to afford the product **4**.



Firure S1 Photograph of the reaction setup

4. “On/off” irradiation experiments for the reaction

To an oven-dried 10 mL glass vial equipped a rubber stopper was added Togni reagent **2** (126 mg, 0.40 mmol), Langlois reagent **3** (62.4 mg, 0.40 mmol) and $Ru(bpy)_3Cl_2$ (2.6 mg, 2 mol%). The tube was evacuated and back-filled with nitrogen, which was repeated three times. 4-Methylstyrene **1** (47.2 mg, 0.40 mmol) and DCM (1 mL) were then added to the tube via syring. Once the mixture was stirred for 2 hours under 40 W blue LEDs radiation, 40 μL of the reaction mixture was taken out via syring. The mixture was monitored by ^{19}F NMR using 1-bromo-4-fluorobenzene as an internal standard. The resulting mixture in the tube continued to react at dark for 2 h, and 40 μL of the reaction mixture was taken out via syring. The mixture was monitored by ^{19}F NMR using 1-bromo-4-fluorobenzene as an internal standard. The above process was repeated for 3 times every 2 hours. The Figure S2 showed that the yield of **4** was obviously increased upon irradiating the reaction with blue LEDs. In contrast, the increase of the yield of **4** was not observed upon performing the reaction in the dark. These results represented the necessity of continuous visible light irradiation for the three-component alkene carbotrifluoromethylation.

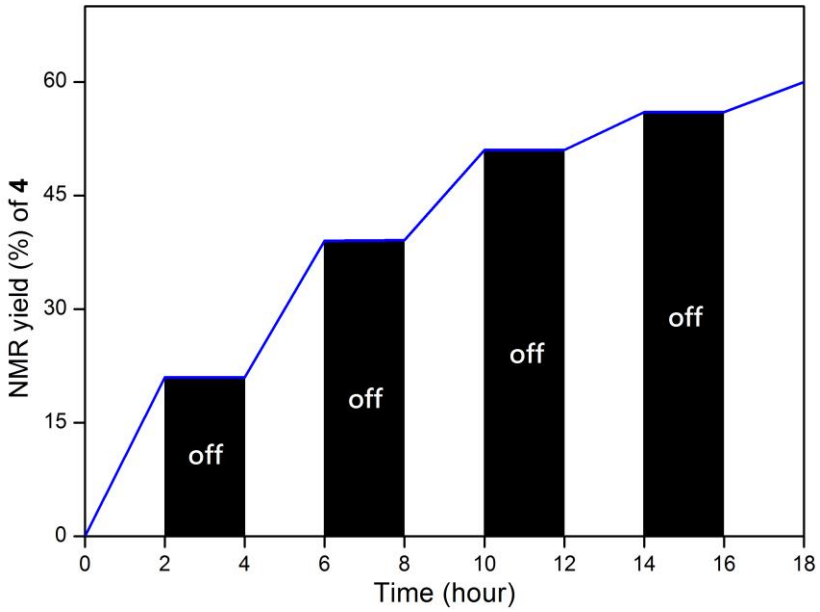
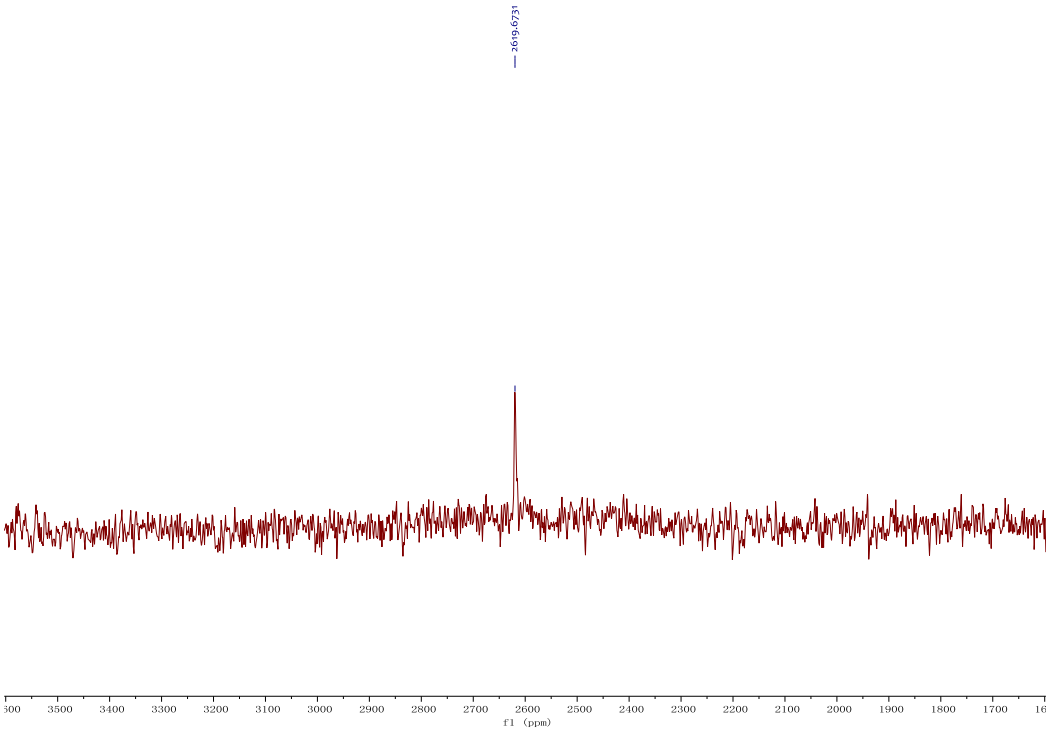


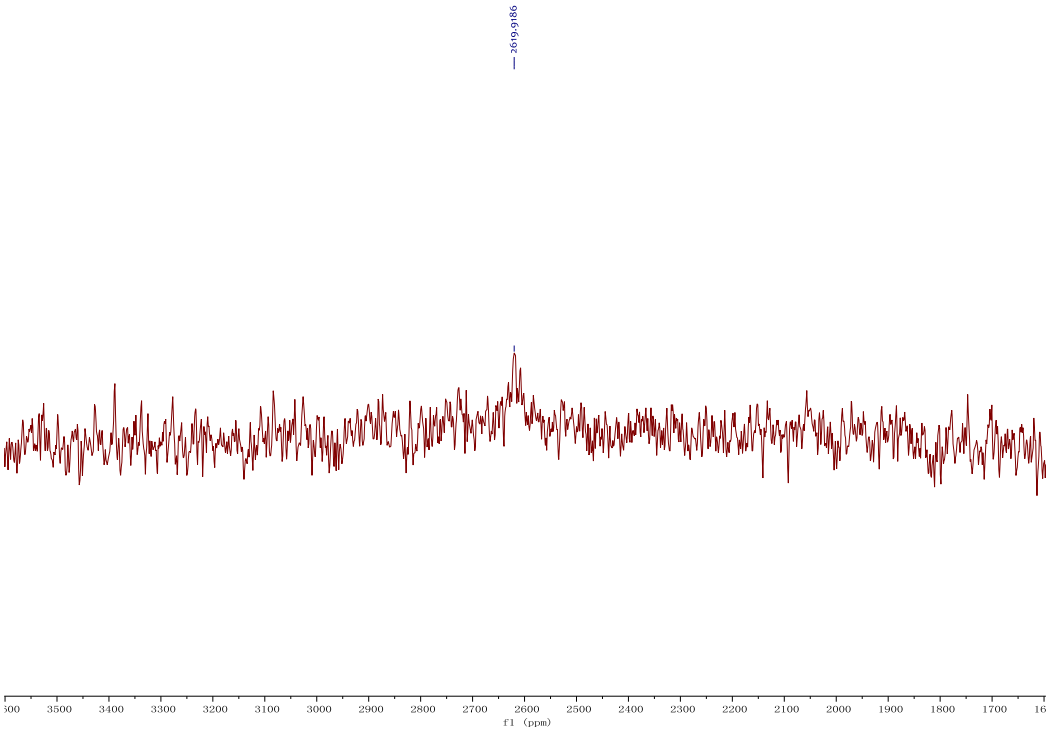
Figure S2. "On/off" irradiation experiments for the reaction.

5. ^{99}Ru NMR experiments

NMR measurements were performed on a 600 MHz NMR spectrometer (JEOL ECZ600R/S3) equipped with a 14.09 T superconducting magnet, an 8.0 mm double-resonance ROYAL probe and a set of low frequency unit (JEOL RESONANCE Inc., Japan). 500 μL sample solutions (before and after reaction) were transferred into 8.0 mm ZrO_2 rotors and the ^{99}Ru NMR spectra were obtained by using Hahn_echo pulse sequence with the following parameters: x_Freq=27.69 MHz, x_sweep=2000 ppm, x_offset=2600 ppm, obs_width_first=1 μs, obs_width_second=2 μs, pre_echo=2 μs, relaxation_delay=5 ms, and scans=108000. Unless otherwise stated, all the NMR experiments were conducted at room temperature (ca. 293K). Spectra of ^{99}Ru NMR represented that lower signal for the photocatalyst $Ru(bpy)_3Cl_2$ was observed after the reaction, which indicated that symmetry of electron cloud of a quadrupolar Ru nucleus was decreased. That was to say, the valence of Ru was changed.



before reaction



after reaction

6. Stern-Volmer fluorescence quenching experiments

Fluorescence measurements for freshly prepared $Ru(bpy)_3Cl_2$ stock solutions in DMF were performed with an excitation irradiation of λ_{ex} = 451 nm and observed for a fluorescence emission of around λ_{em} = 640 nm and an excitation as well as measuring bandwidth of 2 nm. The following fluorescence data were received with measurements of $Ru(bpy)_3Cl_2$ stock solutions of $c = 4 \times 10^{-4}$ M containing Togni reagent in a range of $c = 1 \times 10^{-4}$ M, 2×10^{-4} M, 4×10^{-4} M, 6×10^{-4} M.

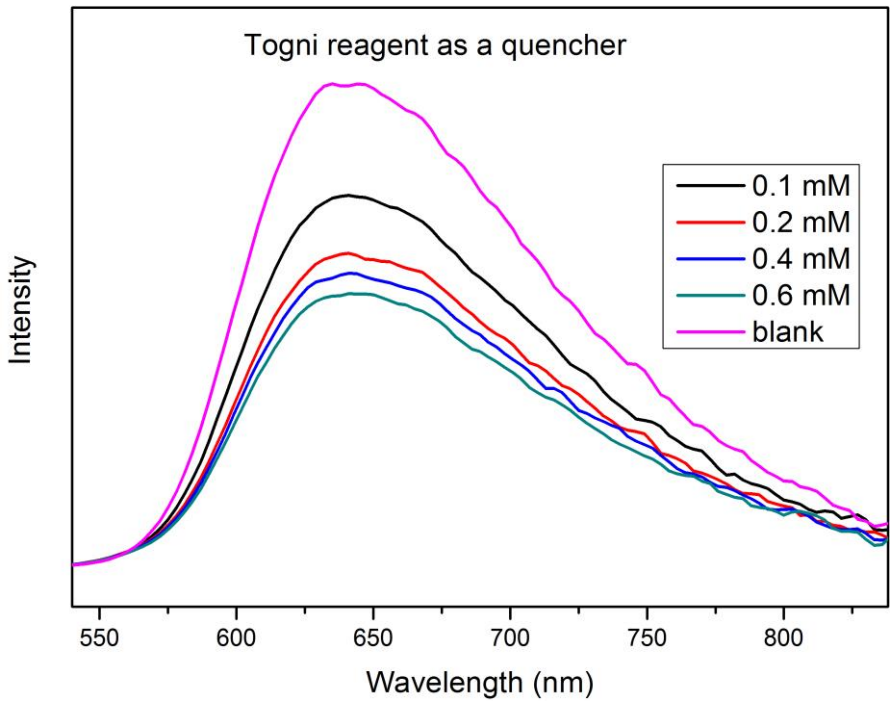


Figure S3. Emission spectra of $Ru(bpy)_3Cl_2$ containing Togni reagent

Fluorescence measurements for freshly prepared $Ru(bpy)_3Cl_2$ stock solutions in DMF were performed with an excitation irradiation of λ_{ex} = 451 nm and observed for a fluorescence emission of around λ_{em} = 640 nm and an excitation as well as measuring bandwidth of 2 nm. The following fluorescence data were received with measurements of $Ru(bpy)_3Cl_2$ stock solutions of $c = 4 \times 10^{-4}$ M containing Langlois reagent in a range of $c = 1 \times 10^{-4}$ M, 2×10^{-4} M, 4×10^{-4} M, 6×10^{-4} M.

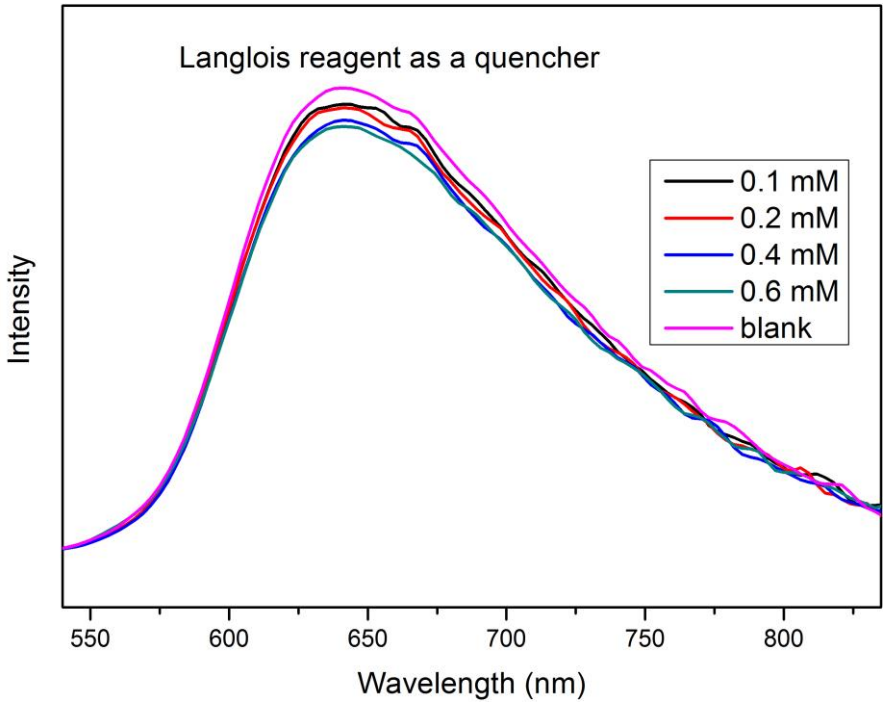


Figure S4. Emission spectra of $Ru(bpy)_3Cl_2$ containing Langlois reagent

Stern-Volmer quenching plot for $Ru(bpy)_3Cl_2$ using Togni and Langlois reagents as quenchers were obtained, respectively. The plot show significant quenching of the excited state of the photocatalyst $Ru(bpy)_3Cl_2$ when using Togni reagent (**2**) as a quencher. These results indicate that photoredox-catalyzed three-component alkene carbotrifluoromethylation proceed through oxidative quenching cycle.

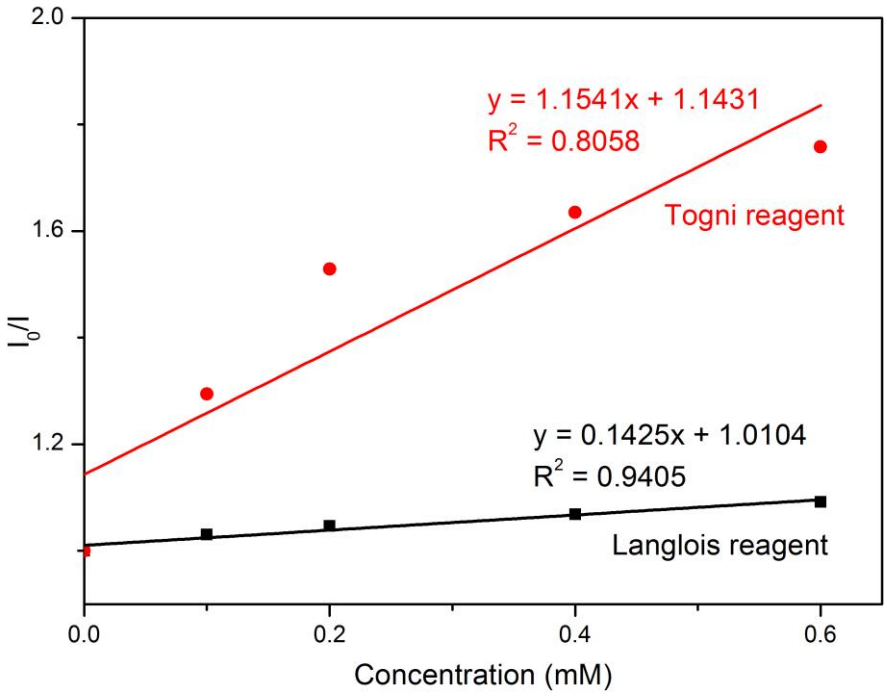
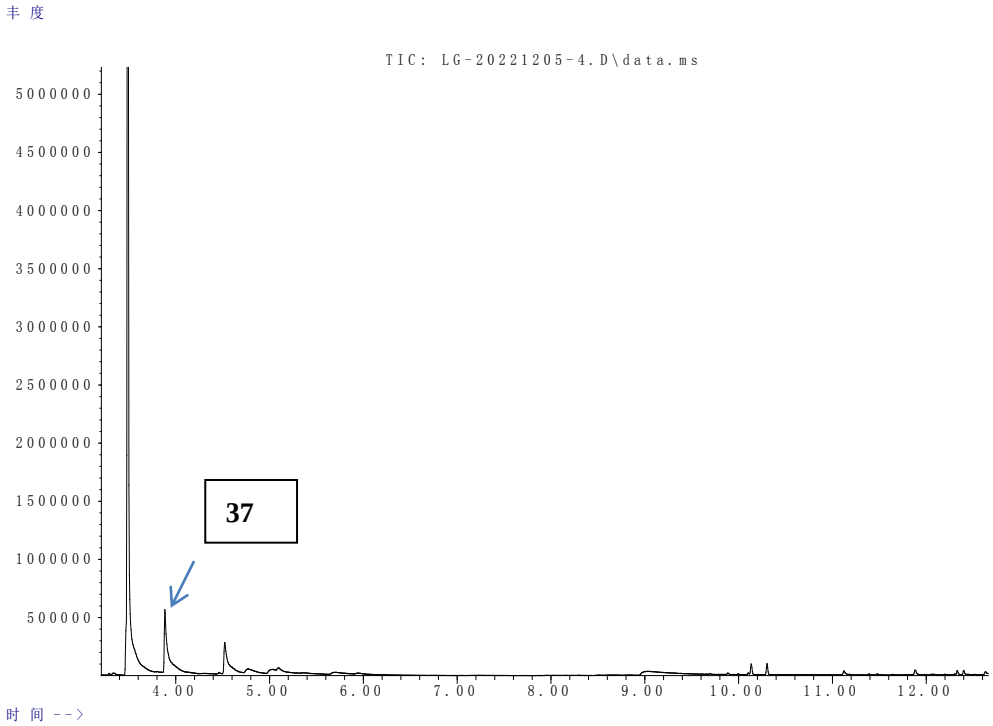
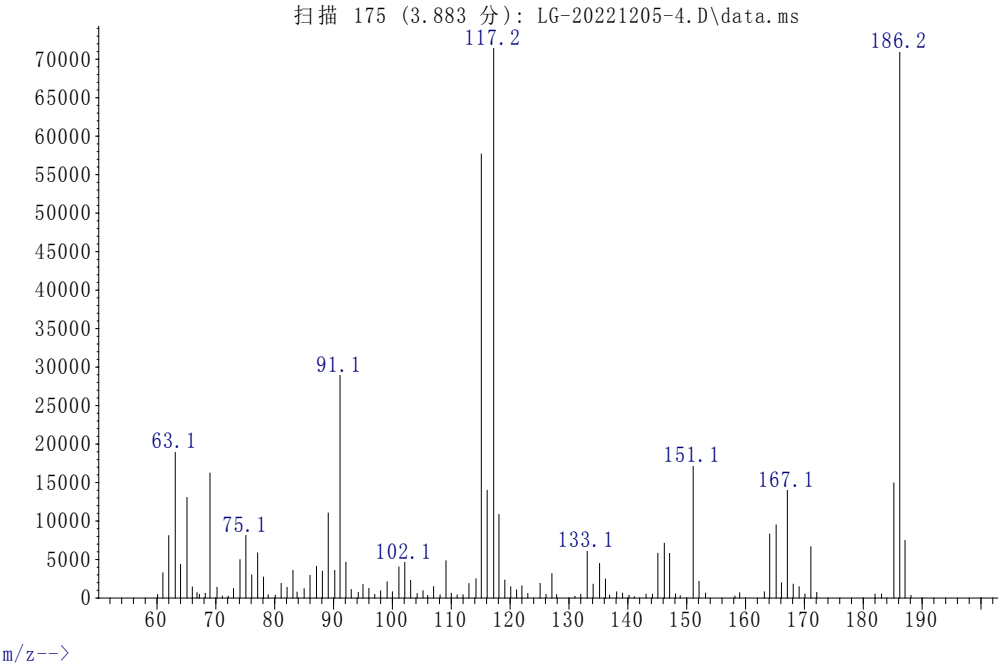
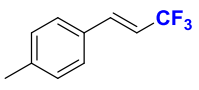


Figure S5. Stern-Volmer quenching plot for $Ru(bpy)_3Cl_2$ using Togni and Langlois reagents as quenchers

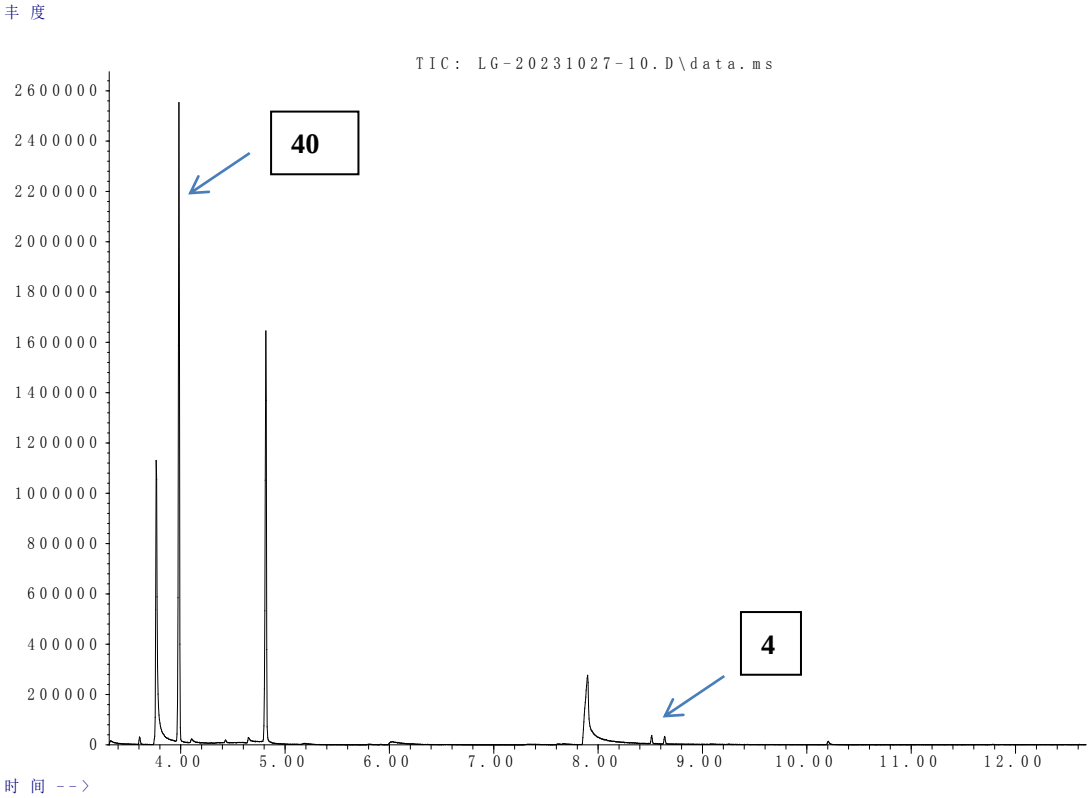
7. GC-MS detection for 37, 40, 41, 42







37, MS (ESI) m/z : 186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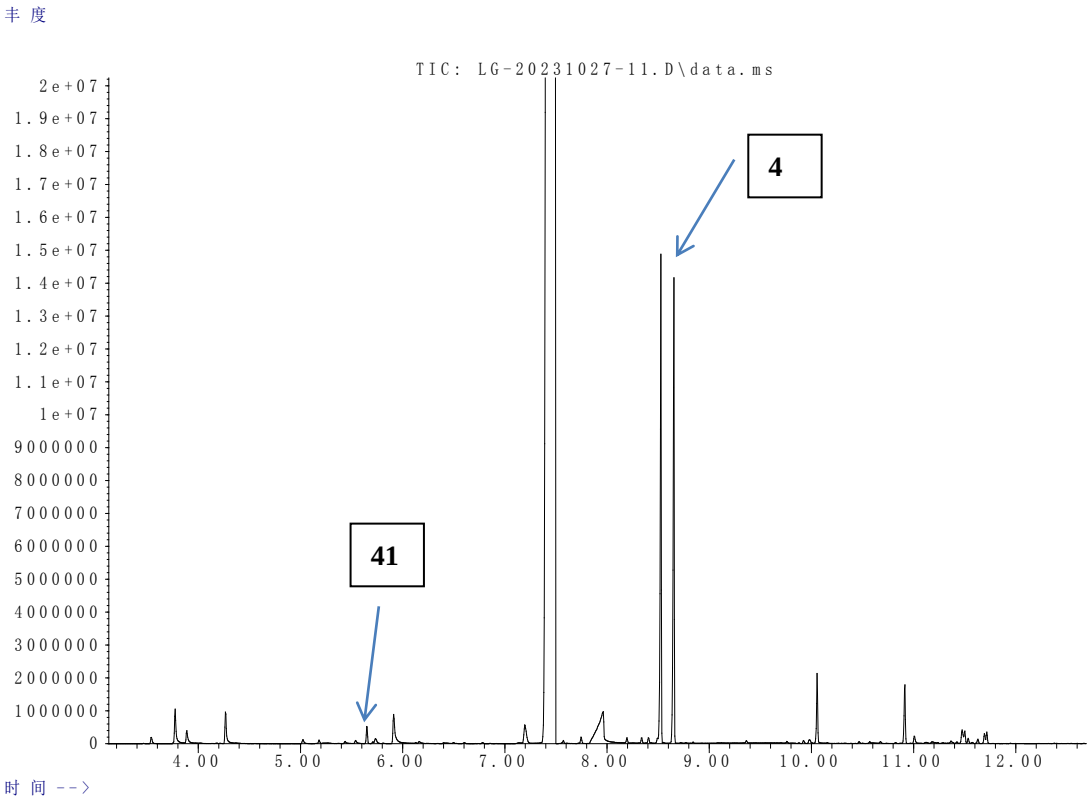
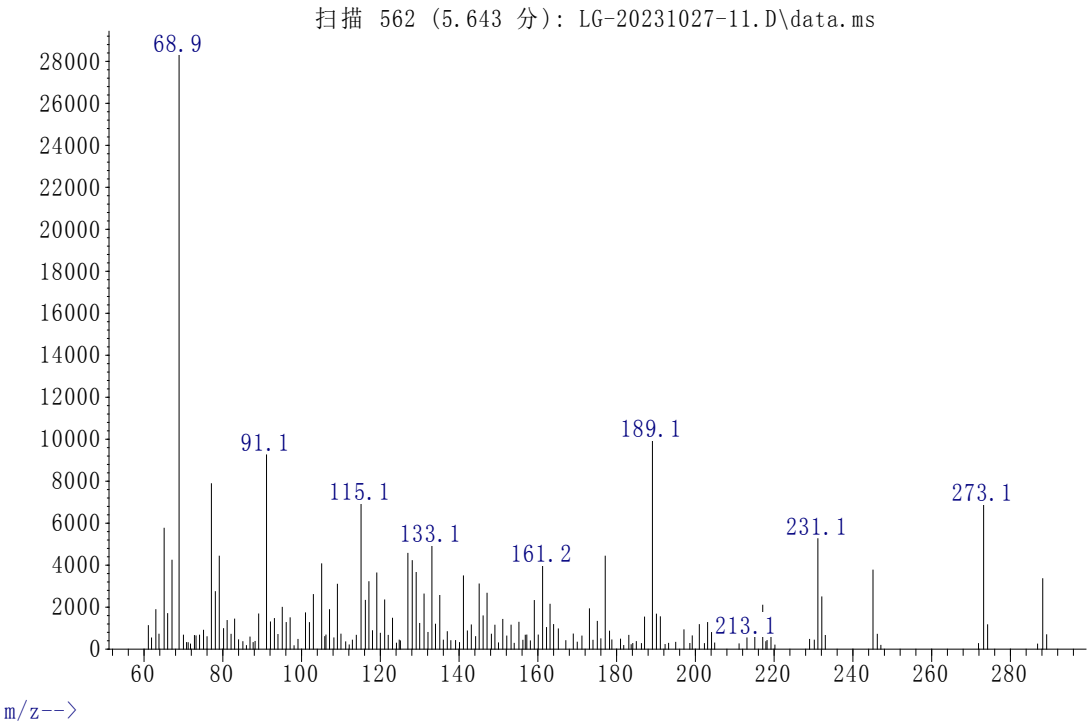
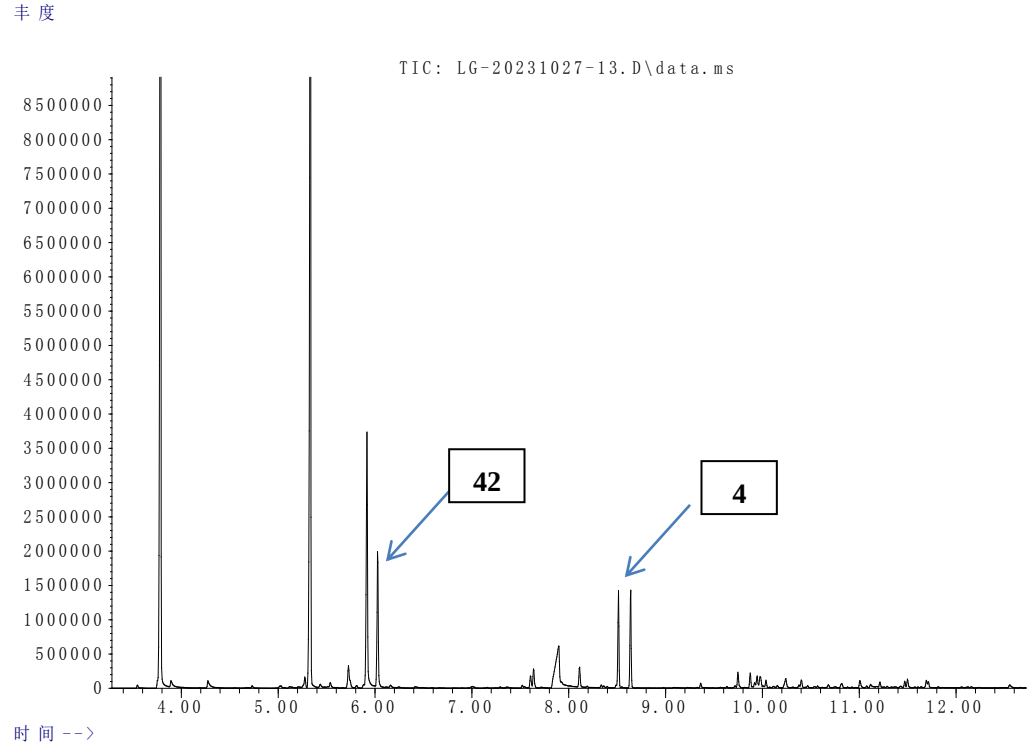
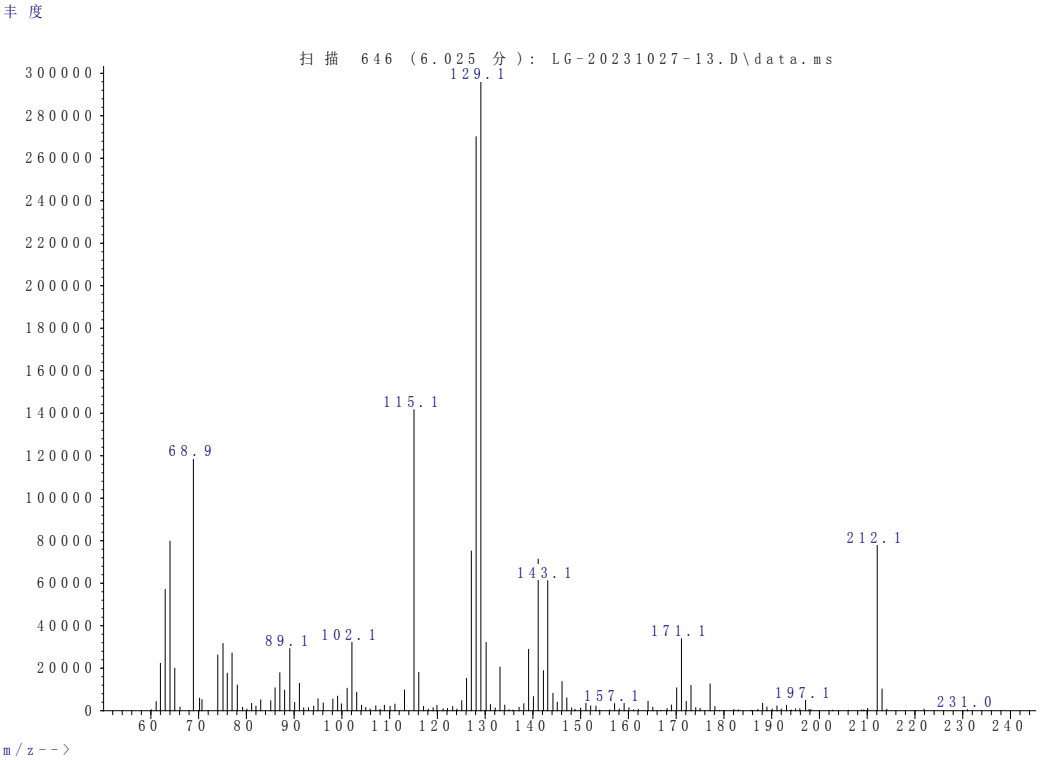
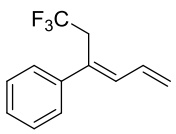




BHT-CF_3 (**41**) MS (ESI) m/z : 288







42, MS (ESI) m/z : 212

8. X-crystal preparation and structure determination

Sample preparation: The obtained **4** (anti) was dissolved with dichloromethane in a test tube. Suitable specimen of **4** was obtained by slow evaporation of the sample from dichloromethane.

Structure determination: The X-ray diffraction data for **4** were collected on a Bruker D8 QUEST ECO diffractometer with graphite–monochromated MoK_{α} radiation (λ = 0.71073 Å) at room temperature. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 by using the program SHELX-2016.[1] Anisotropic thermal factors were assigned to all the non-hydrogen atoms. Generally, the H atoms bonded to C and N atoms were placed geometrically and refined as riding. Further crystallographic details for both compounds are summarized in the following crystallographic data.

Reference:

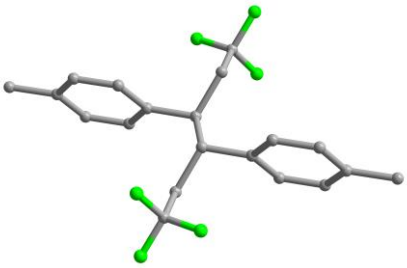
[1] Sheldrick, G. M. *Acta Crystallogr., Sect. C.*, 2015, **71**, 3-8.

9. Crystallographic data

Table S1. Crystal data and structure refinement for 4

Empirical formula	$C_{20}H_{20}F_6$
Formula weight	374.37
Temperature	295(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, $P2_1/c$
Unit cell dimensions	a = 10.0720(9) A b = 10.9544(9) A beta = 93.453(3) deg c = 8.2997(8) A

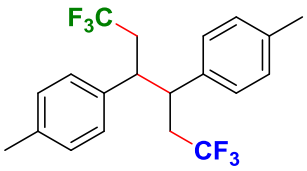
Volume	914.07(14) Å^3
Z, Calculated density	2, 1.360 g cm^{-3}
Absorption coefficient	0.121 mm^{-1}
F(000)	388
Reflections collected / unique	13384 / 1800 [*R*(int) = 0.0524]
Completeness to theta = 25.242	99.8 %
Refinement method	Full-matrix least-squares on F^2
parameters	119
Goodness-of-fit on F^2	1.027
Final *R* indices [$I > 2\text{sigma}(I)$]	$R_1 = 0.0517$, $wR_2 = 0.1242$
R indices (all data)	$R_1 = 0.0766$, $wR_2 = 0.1426$



CCDC: no. 2347065

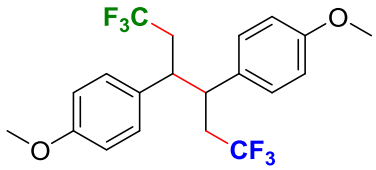
10. Characterization data of products

4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(methylbenzene) **(4)**



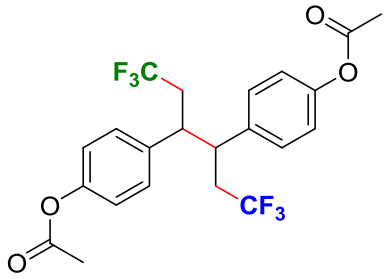
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 34.1 mg, 42%; syn (the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.03-7.02 (d, J = 7.8 Hz, 4H), 6.70-6.68 (d, J = 7.8 Hz, 4H), 3.33-3.29 (m, 2H), 2.55-2.46 (m, 2H), 2.35-2.26 (m, 2H), 2.31 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 137.1, 134.8, 129.4, 128.7, 126.7 (q, J_{CF} = 278.5 Hz), 43.1, 37.5 (q, J_{CF} = 27.2 Hz), 21.2; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6F).
White solid; 24.7 mg, 33%; anti (the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.18-7.17 (d, J = 7.7 Hz, 4H), 7.12-7.10 (d, J = 7.8 Hz, 4H), 3.01-2.96 (m, 2H), 2.35 (s, 6H), 2.25-2.16 (m, 2H), 2.13-2.05 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 137.6, 137.3, 129.9, 127.8, 126.5 (q, J_{CF} = 277.6 Hz), 45.5, 38.3 (q, J_{CF} = 27.6 Hz), 21.2; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.3 (t, J = 10.8 Hz, 6F).[1]

4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(methoxybenzene) **(5)**



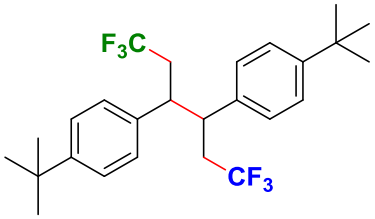
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. White solid; 50.3 mg, 62%; anti : syn = 1.09 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.14-7.13 (d, J = 8.6 Hz, 4H), 6.91-6.90 (d, J = 8.7 Hz, 4H), 6.77-6.75 (d, J = 8.8 Hz, 3.7H), 6.71-6.70 (d, J = 8.7 Hz, 3.7H), 3.82 (s, 6H), 3.78 (s, 5.5H), 3.31-3.28 (m, 1.8H), 2.99-2.94 (m, 2H), 2.53-2.45 (m, 1.8H), 2.32-2.26 (m, 1.8H), 2.20-2.06 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 158.9, 158.8, 132.5, 130.5, 129.8, 128.9, 126.7 (q, J_{CF} = 276.4 Hz), 126.5 (q, J_{CF} = 277.6 Hz), 114.5, 113.3, 55.32, 55.27, 45.3, 42.8, 38.3 (q, J_{CF} = 27.6 Hz), 37.8 (q, J_{CF} = 27.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 5.5F), -63.3 (t, J = 10.6 Hz, 6F).[1]

(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(4,1-phenylene) diacetate **(6)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (30:1) as eluant) to offer the product. White solid; 65.6 mg, 71%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.23-7.21 (d, J = 8.4 Hz, 4H), 7.14-7.13 (d, J = 8.3 Hz, 4H), 6.97-6.95 (d, J = 8.4 Hz, 4H), 6.78-6.77 (d, J = 8.4 Hz, 4H), 3.40-3.37 (m, 2H), 3.10-3.06 (m, 2H), 2.58-2.50 (m, 2H), 2.38-2.30 (m, 2H), 2.30 (s, 6H), 2.27 (s, 6H), 2.24-2.10 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 169.3, 169.2, 150.2, 150.0, 137.6, 135.2, 130.3, 128.9, 126.5 (q, J_{CF} = 273.5 Hz), 126.2 (q, J_{CF} = 274.7 Hz), 122.3, 121.2, 45.2, 42.8, 38.3 (q, J_{CF} = 27.6 Hz), 37.6 (q, J_{CF} = 27.4 Hz), 21.3, 21.2; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.4 Hz, 6F), -63.3 (t, J = 10.6 Hz, 6F).[1]

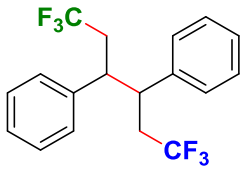
4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(tert-butylbenzene) **(7)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 34.8 mg, 38%; syn (the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.22-7.20 (d, J = 8.3 Hz, 4H), 6.72-6.71 (d, J = 8.3 Hz, 4H), 3.34-3.31 (m, 2H), 2.57-2.48 (m, 2H), 2.37-2.28 (m, 2H), 1.29 (s, 18H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 150.3, 135.0, 129.1, 126.8 (q, J_{CF} = 276.8 Hz), 124.7, 43.0, 37.4 (q, J_{CF} = 27.4 Hz), 34.5, 31.4; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6F).

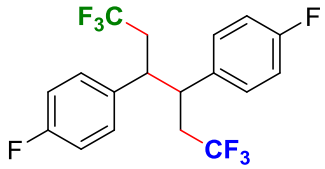
White solid; 36.6 mg, 40%; anti (the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.37-7.36 (d, J = 8.2 Hz, 4H), 7.15-7.13 (d, J = 8.2 Hz, 4H), 3.02-3.00 (m, 2H), 2.21-2.06 (m, 4H), 1.32 (s, 18H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 150.5, 137.5, 127.5, 126.5 (q, J_{CF} = 275.5 Hz), 126.0, 45.4, 38.3 (q, J_{CF} = 27.6 Hz), 34.6, 31.4; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.3 (t, J = 10.6 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{33}F_6$ 459.2486; found 459.2482.

(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)dibenzene **(8)**



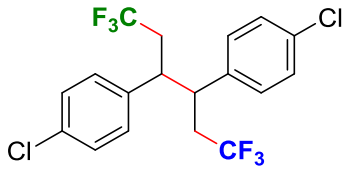
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 50.5 mg, 73%; anti : syn = 3.33 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.40-7.37 (m, 4H), 7.33-7.30 (m, 2H), 7.25-7.23 (m, 4H), 7.22-7.19 (m, 1.8H), 6.80-6.79 (m, 1.2H), 3.37-3.34 (m, 0.6H), 3.09-3.04 (m, 2H), 2.61-2.52 (m, 0.6H), 2.42-2.33 (m, 0.6H), 2.27-2.20 (m, 2H), 2.14-2.06 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 140.6, 138.1, 129.4, 129.2, 128.02, 127.98, 127.8, 127.5, 126.6 (q, J_{CF} = 277.3 Hz), 126.4 (q, J_{CF} = 275.2 Hz), 45.9, 43.7, 38.3 (q, J_{CF} = 27.5 Hz), 37.6 (q, J_{CF} = 27.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 1.8F), -63.4 (t, J = 10.7 Hz, 6F).[1]

4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(fluorobenzene) **(9)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 64.2 mg, 84%; syn (one of the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 6.92-6.90 (m, 4H), 6.76-6.74 (m, 4H), 3.33-3.30 (m, 2H), 2.57-2.49 (m, 2H), 2.38-2.29 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 162.0 (d, J_{CF} = 244.8 Hz), 133.6 (d, J_{CF} = 2.6 Hz), 130.7 (d, J_{CF} = 8.1 Hz), 126.4 (q, J_{CF} = 273.7 Hz), 115.1 (d, J_{CF} = 21.2 Hz), 43.1, 37.9 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6F), -114.5 (s, 2F).[1]

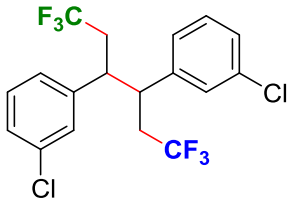
4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(chlorobenzene) **(10)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale

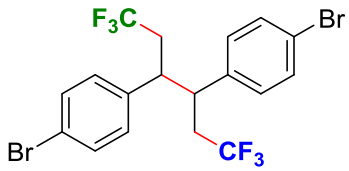
yellow solid; 59.6 mg, 72%; anti : syn = 1.37 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.37-7.36 (d, J = 8.3 Hz, 4H), 7.21-7.19 (d, J = 8.2 Hz, 2.9H), 7.16-7.15 (d, J = 8.3 Hz, 4H), 6.74-6.72 (d, J = 8.3 Hz, 2.9H), 3.33-3.29 (m, 1.5H), 3.05-3.00 (m, 2H), 2.56-2.48 (m, 1.5H), 2.38-2.29 (m, 1.5H), 2.25-2.16 (m, 2H), 2.12-2.04 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 138.6, 136.2, 133.8, 133.6, 130.5, 129.5, 129.2, 128.4, 126.3 (q, J_{CF} = 277.4 Hz), 126.1 (q, J_{CF} = 276.5 Hz), 45.2, 43.2, 38.2 (q, J_{CF} = 27.7 Hz), 37.7 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 4.4F), -63.3 (t, J = 10.6 Hz, 6F).[1]

3,3'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(chlorobenzene) **(11)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale yellow solid; 55.5 mg, 67%; anti : syn = 0.88 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.35-7.30 (m, 4H), 7.23-7.21 (m, 4.5H), 7.18-7.15 (t, J = 7.8 Hz, 2.3H), 7.12-7.10 (m, 2H), 6.77-6.76 (m, 2.3H), 6.71-6.70 (m, 2.3H), 3.32-3.28 (m, 2.3H), 3.05-3.00 (m, 2H), 2.58-2.50 (m, 2.3H), 2.42-2.33 (m, 2.3H), 2.29-2.20 (m, 2H), 2.13-2.05 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 142.2, 139.8, 135.2, 134.2, 130.6, 129.5, 129.2, 128.3, 128.1, 128.0, 127.4, 126.2 (q, J_{CF} = 272.7 Hz), 126.1, 126.0 (q, J_{CF} = 274.2 Hz), 45.5, 43.5, 38.1 (q, J_{CF} = 27.4 Hz), 37.6 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6.8F), -63.3 (t, J = 10.7 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{18}H_{15}Cl_2F_6$ 415.0455; found 415.0463.

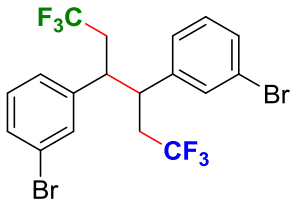
4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(bromobenzene) **(13)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale yellow solid; 65.2 mg, 65%; anti : syn = 0.97 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.53-7.51 (d, J = 7.8 Hz, 4H), 7.36-7.35 (d, J = 7.7 Hz, 4H), 7.10-7.09 (d, J = 7.8 Hz, 4.1H), 6.68-6.67 (d, J = 7.8 Hz, 4.1H), 3.32-3.28 (m, 2.1H), 3.04-2.99 (m, 2H), 2.56-2.48 (m, 2.1H), 2.37-2.30 (m, 2.1H), 2.23-2.16 (m, 2H), 2.12-2.04 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] =

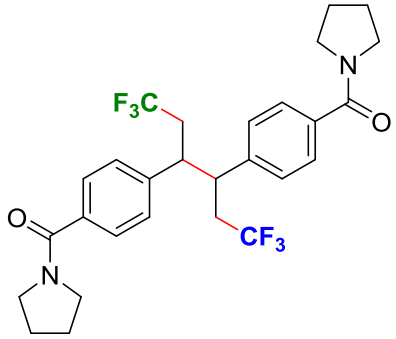
139.1, 136.7, 132.5, 131.4, 130.8, 129.6, 126.3 (q, J_{CF} = 275.8 Hz), 126.1 (q, J_{CF} = 276.2 Hz), 121.9, 121.8, 45.2, 43.2, 38.1 (q, J_{CF} = 27.5 Hz), 37.7 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 6.2F), -63.3 (t, J = 10.6 Hz, 6F).[1]

3,3'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(bromobenzene) **(14)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale yellow oil; 55.2 mg, 55%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.48-7.46 (m, 2H), 7.39-7.37 (m, 4H), 7.29-7.26 (t, J = 7.8 Hz, 2H), 7.16-7.15 (m, 2H), 7.13-7.10 (t, J = 7.8 Hz, 2H), 6.90 (s, 2H), 6.76-6.75 (m, 2H), 3.31-3.28 (m, 2H), 3.03-2.99 (m, 2H), 2.57-2.48 (m, 2H), 2.41-2.31 (m, 2H), 2.28-2.21 (m, 2H), 2.13-2.05 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 142.4, 139.9, 132.1, 131.3, 131.0, 130.93, 130.89, 129.7, 127.9, 126.5, 126.2 (q, J_{CF} = 276.7 Hz), 126.0 (q, J_{CF} = 274.1 Hz), 123.3, 122.3, 45.4, 43.4, 38.2 (q, J_{CF} = 27.6 Hz), 37.6 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6F), -63.3 (t, J = 10.6 Hz, 6F); HRMS (ESI) m/z: $[M+Na]^+$ calcd. for $C_{18}H_{14}Br_2F_6Na$ 524.9264; found 524.9260.

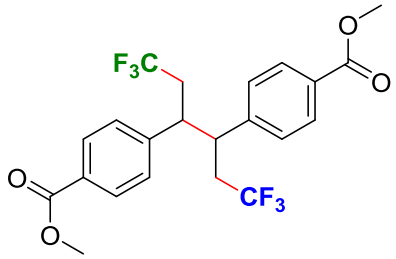
((1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(4,1-phenylene))bis(pyrrolidin-1-ylmethanone) **(16)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (10:1) as eluant) to offer the product. Pale yellow oil; 65% (determind by ^{19}F NMR); anti (Togni reagent as impurity could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.54-7.53 (d, J = 8.0 Hz, 4H), 7.27-7.26 (d, J = 8.0 Hz, 4H), 3.63-3.61 (t, J = 7.0 Hz, 4H), 3.41-3.89 (t, J = 6.9 Hz, 4H), 3.12-3.06 (m, 2H), 2.27-2.18 (m, 2H), 2.11-2.03 (m, 2H), 1.96-1.92 (m, 4H), 1.88-1.83 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] =

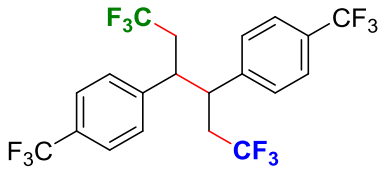
169.2, 142.1, 136.7, 128.1, 127.9, 107.1 (q, J_{CF} = 276.5 Hz), 49.7, 46.4, 45.5, 38.1 (q, J_{CF} = 27.6 Hz), 26.5, 24.5; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.3 (t, J = 10.5 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{28}H_{31}F_6N_2O_2$ 541.2290; found 541.2282.

dimethyl 4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)dibenzoate **(17)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (30:1) as eluant) to offer the product. White solid; 57.3 mg, 62%; anti : syn = 1.07 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 8.08-8.07 (d, J = 8.4 Hz, 4H), 7.86-7.85 (d, J = 8.2 Hz, 3.7H), 7.33-7.31 (d, J = 8.4 Hz, 4H), 6.89-6.87 (d, J = 8.4 Hz, 3.7H), 3.93 (s, 6H), 3.88 (s, 5.6H), 3.41-3.38 (m, 1.9H), 3.19-3.15 (m, 2H), 2.65-2.56 (m, 1.9H), 2.49-2.41 (m, 1.9H), 2.31-2.24 (m, 2H), 2.10-2.02 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 116.7, 116.6, 145.3, 143.3, 130.6, 130.0, 129.5, 129.1, 128.1, 126.2 (q, J_{CF} = 274.5 Hz), 126.0 (q, J_{CF} = 276.8 Hz), 52.4, 52.3, 45.6, 44.0, 38.3 (q, J_{CF} = 27.6 Hz), 37.9 (q, J_{CF} = 27.8 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 5.6F), -63.4 (t, J = 10.6 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{21}F_6O_4$ 463.1344; found 463.1355.

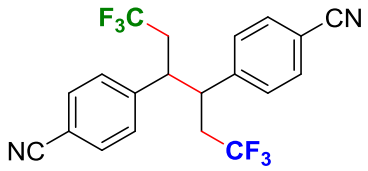
4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis((trifluoromethyl)benzene) **(18)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (100:1) as eluant) to offer the product. Colorless oil; 49.1 mg, 51%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.68-7.67 (d, J = 8.2 Hz, 4H), 7.49-7.48 (d, J = 8.2 Hz, 4H), 7.38-7.36 (d, J = 8.2 Hz, 4H), 6.94-6.93 (d, J = 8.1 Hz, 4H), 3.45-3.41 (m, 2H), 3.22-3.17 (m, 2H), 2.64-2.56 (m, 2H), 2.46-2.37 (m, 2H), 2.33-2.23 (m, 2H), 2.12-2.04 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 144.0, 141.9, 130.5 (q, J_{CF} = 32.7 Hz), 130.1 (q, J_{CF} = 32.6 Hz), 129.5, 128.4, 126.4 (q, J_{CF} = 2.9 Hz), 126.1 (q, J_{CF} = 274.9 Hz), 125.9 (q, J_{CF} = 276.3 Hz), 125.3 (q, J_{CF} = 2.8 Hz), 124.0 (q, J_{CF} = 278.2 Hz), 123.8 (q, J_{CF} = 276.8 Hz), 45.5, 43.7, 38.2 (q, J_{CF} =

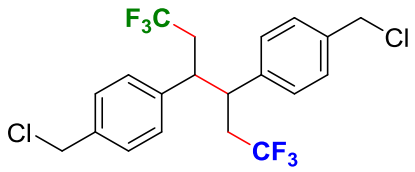
27.2 Hz), 37.7 (q, J_{CF} = 27.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -62.50 (s, 6F), -62.52 (s, 6F), -63.2 (t, J = 10.6 Hz, 6F), -63.4 (t, J = 10.5 Hz, 6F).[1]

4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)dibenzonitrile **(19)**



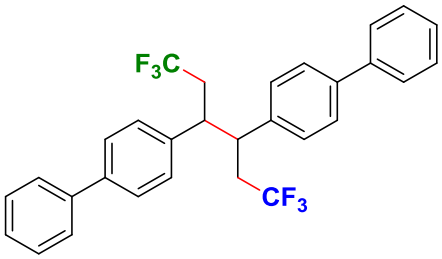
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (100:1) as eluant) to offer the product. Pale yellow solid; 42.7 mg, 54%; anti : syn = 0.33 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.73-7.71 (d, J = 8.2 Hz, 4H), 7.51-7.50 (d, J = 8.3 Hz, 12H), 7.36-7.35 (d, J = 8.2 Hz, 4H), 6.95-6.94 (m, 12H), 3.38-3.34 (m, 6H), 3.20-3.15 (m, 2H), 2.66-2.59 (m, 6H), 2.50-2.41 (m, 6H), 2.33-2.25 (m, 2H), 2.01-1.98 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 145.0, 143.5, 133.2, 132.3, 129.5, 128.8, 125.9 (q, J_{CF} = 279.1 Hz), 125.5 (q, J_{CF} = 276.3 Hz), 118.21, 118.17, 112.5, 112.0, 45.6, 44.3, 38.0 (q, J_{CF} = 27.1 Hz), 37.7 (q, J_{CF} = 27.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.4 Hz, 18F), -63.3 (t, J = 10.7 Hz, 6F).[1]

4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis((chloromethyl)benzene) **(20)**



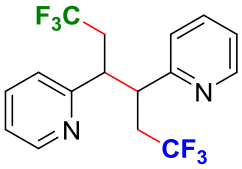
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 65.4 mg, 74%; anti : syn = 0.71 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.42-7.41 (d, J = 8.2 Hz, 4H), 7.25-7.23 (m, 9.6H), 6.80-6.79 (d, J = 8.2 Hz, 5.6H), 4.60 (s, 4H), 4.54 (s, 5.6H), 3.39-3.35 (m, 2.8H), 3.11-3.06 (m, 2H), 2.59-2.50 (m, 2.8H), 2.40-2.32 (m, 2.8H), 2.26-2.18 (m, 2H), 2.13-2.06 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 140.6, 138.1, 137.1, 136.8, 129.7, 129.4, 128.3, 126.5 (q, J_{CF} = 272.4 Hz), 126.3 (q, J_{CF} = 275.1 Hz), 45.8, 45.5, 43.4, 38.2 (q, J_{CF} = 27.6 Hz), 37.6 (q, J_{CF} = 27.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 8.4F), -63.3 (t, J = 10.4 Hz, 6F).[1]

4,4''-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)di-1,1'-biphenyl **(21)**



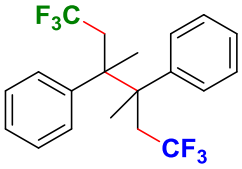
This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale yellow oil; 45.8 mg, 46%; syn (one of the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.59-7.57 (m, 4H), 7.49-7.47 (m, 4H), 7.45-7.42 (m, 4H), 7.36-7.33 (m, 2H), 6.92-6.91 (m, 4H), 3.48-3.44 (m, 2H), 2.67-2.59 (m, 2H), 2.48-2.39 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 140.4, 140.3, 136.9, 129.9, 128.9, 127.5, 127.1, 126.7 (q, J_{CF} = 276.3 Hz), 43.3, 37.6 (q, J_{CF} = 27.3 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.8 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{30}H_{25}F_6$ 499.1860; found 499.1864.

2,2'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)dipyridine **(22)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (8:1) as eluant) to offer the product. Pale yellow oil; 48.7 mg, 70%; syn (one of the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 8.66-8.65 (m, 2H), 7.67-7.64 (m, 2H), 7.23-7.20 (m, 4H), 3.54-3.49 (m, 2H), 2.75-2.67 (m, 2H), 1.91-1.83 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 159.7, 150.3, 136.8, 126.8 (q, J_{CF} = 278.3 Hz), 125.1, 122.6, 45.7, 36.9 (q, J_{CF} = 27.2 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.8 (t, J = 10.6 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{16}H_{15}F_6N_2$ 349.1139; found 349.1146.

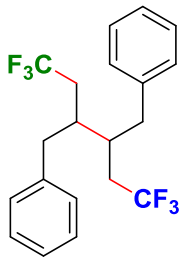
(1,1,1,6,6,6-hexafluoro-3,4-dimethylhexane-3,4-diyl)dibenzene **(23)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless

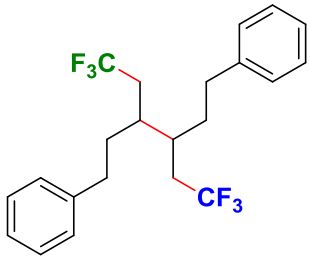
oil; 58.3 mg, 78%; anti : syn = 1.11 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.26-7.25 (m, 12H), 7.06-7.01 (d, J = 34.9 Hz, 7H), 3.13-3.04 (m, 1.8H), 2.62-2.54 (m, 2H), 2.51-2.43 (m, 2H), 2.30-2.22 (m, 1.8H), 1.51 (s, 6H), 1.48 (s, 5.4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 140.5, 140.3, 128.8, 128.6, 127.6 (q, J_{CF} = 275.8 Hz), 127.3, 127.0, 126.8 (q, J_{CF} = 277.4 Hz), 45.9, 45.6, 39.1 (q, J_{CF} = 27.3 Hz), 38.8 (q, J_{CF} = 27.6 Hz), 21.3, 20.8; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -56.2 (t, J = 10.6 Hz, 5.4F), -56.4 (t, J = 10.6 Hz, 6F).[1]

(2,3-bis(2,2,2-trifluoroethyl)butane-1,4-diyl)dibenzene **(24)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 28.4 mg, 38%; anti : syn = 0.88 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.31-7.28 (m, 4.5H), 7.25-7.19 (m, 8.3H), 7.09-7.08 (d, J = 7.0 Hz, 4H), 7.00-6.98 (d, J = 7.1 Hz, 4.5H), 2.83-2.79 (dd, J = 13.9, 7.0 Hz, 2H), 2.68-2.65 (dd, J = 13.9, 7.1 Hz, 2.3H), 2.63-2.57 (m, 4H), 2.35-2.29 (m, 4.5H), 2.23-2.15 (m, 2H), 2.12-2.06 (m, 2H), 2.01-1.94 (m, 4.5H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 138.7, 138.6, 128.9, 128.8, 128.7, 126.8, 126.6, 127.1 (q, J_{CF} = 278.5 Hz), 36.3, 36.2, 35.0, 34.9, 33.8 (q, J_{CF} = 27.2 Hz), 33.7 (q, J_{CF} = 27.3 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.0 (t, J = 10.9 Hz, 6.8F), -63.3 (t, J = 10.9 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{20}H_{21}F_6$ 375.1547; found 375.1550.

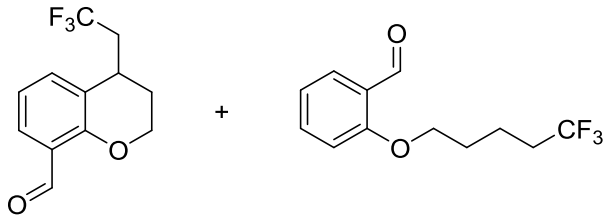
(3,4-bis(2,2,2-trifluoroethyl)hexane-1,6-diyl)dibenzene **(25)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 21.7 mg, 27%; anti : syn = 1.12 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.29-7.27 (m, 8H), 7.21-7.18 (m, 4H), 7.16-7.13 (m, 4H), 7.11-7.08 (m, 4H), 2.62-2.39 (m, 8H), 2.19-1.93 (m, 12H),

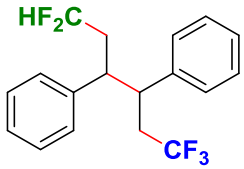
1.68-1.57 (m, 8H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 141.3, 128.63, 128.61, 128.5, 128.4, 127.6 (q, J_{CF} = 276.2 Hz), 127.2 (q, J_{CF} = 278.4 Hz), 126.3, 126.2, 34.9 (q, J_{CF} = 27.5 Hz), 34.7 (q, J_{CF} = 27.4 Hz), 33.9, 33.8, 33.7, 33.6, 32.9, 32.5; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.7 (t, J = 10.8 Hz, 5.4F), -63.8 (t, J = 10.9 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{25}F_6$ 403.1860; found 403.1855.

4-(2,2,2-trifluoroethyl)chromane-8-carbaldehyde (**26**) and 2-((5,5,5-trifluoropentyl)oxy)benzaldehyde **(27)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (10:1) as eluant) to offer the product. Yellow oil; 39.7 mg, 41%; **26** : **27** = 4 : 1 (the mixture could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 10.48 (d, J = 0.8 Hz, 0.3H), 10.39 (d, J = 0.7 Hz, 1H), 7.83-7.82 (dd, J = 7.7, 1.9 Hz, 0.3H), 7.69-7.68 (dd, J = 7.7, 1.8 Hz, 1H), 7.54-7.52 (m, 0.3H), 7.34-7.32 (m, 1H), 7.03-7.01 (m, 0.3H), 6.97-6.94 (m, 1H), 4.39-4.35 (m, 1H), 4.29-4.24 (m, 1H), 4.11-4.09 (t, J = 6.1 Hz, 0.5H), 3.29-3.25 (m, 1H), 2.60-2.51 (m, 1H), 2.43-2.33 (m, 1H), 2.28-2.22 (m, 1H), 2.20-2.15 (m, 0.5H), 2.08-2.03 (m, 1H), 1.96-1.92 (m, 0.5H), 1.82-1.77 (m, 0.5H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 189.8, 189.7, 161.2, 157.3, 136.1, 135.3, 128.6, 127.7, 127.1 (q, J_{CF} = 275.1 Hz), 126.4 (q, J_{CF} = 272.2 Hz), 125.0, 124.8, 120.9, 120.5, 112.4, 67.7, 63.3, 40.2 (q, J_{CF} = 27.1 Hz), 33.5 (q, J_{CF} = 27.4 Hz), 28.3, 26.3, 19.3, 19.0; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.5 (t, J = 10.7 Hz, 3F), -66.2 (t, J = 10.5 Hz, 0.8F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{12}H_{12}F_3O_2$ 245.0789; found 245.0795; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{12}H_{14}F_3O_2$ 247.0946; found 247.0956.

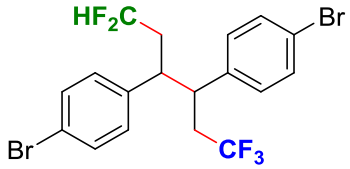
(1,1,1,6,6-pentafluorohexane-3,4-diyl)dibenzene **(30)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Colorless oil; 25.6 mg, 39%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.41-7.36 (m, 5H), 7.33-7.29 (m, 3H), 7.23-7.18 (m, 8H), 6.85-6.83 (m, 4H), 5.51-5.30 (m, 1H), 5.26-5.07 (m, 1H), 3.26-3.23 (m, 1H), 3.18-3.15 (m, 1H), 3.09-3.05 (t, J = 11.0 Hz, 1H), 2.95-2.91 (m, 1H), 2.62-2.54 (m,

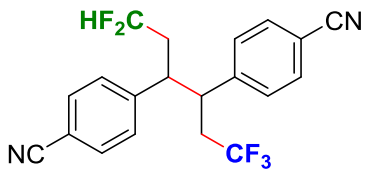
1H), 2.46-2.37 (m, 1H), 2.30-2.24 (m, 2H), 2.18-2.08 (m, 2H), 1.92-1.77 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 140.8, 140.7, 138.9, 138.1, 129.5, 129.2, 129.1, 129.0, 128.4, 128.01, 127.97, 127.9, 127.6, 127.5, 127.4, 127.2, 126.6 (q, J_{CF} = 271.9 Hz), 116.5 (t, J_{CF} = 237.2 Hz), 46.4 (d, J_{CF} = 8.7 Hz), 46.0, 44.7 (d, J_{CF} = 8.7 Hz), 44.6, 38.8 (t, J_{CF} = 21.8 Hz), 38.4 (q, J_{CF} = 27.6 Hz), 38.2 (t, J_{CF} = 21.5 Hz), 37.5 (q, J_{CF} = 27.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 3F), -63.4 (t, J = 10.5 Hz, 3F), -114.3--116.2 (m, 2F), -117.5--119.0 (m, 2F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{18}H_{18}F_5$ 329.1329; found 329.1331.

4,4'-(1,1,1,6,6-pentafluorohexane-3,4-diyl)bis(bromobenzene) **(31)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether as eluant) to offer the product. Pale yellow solid; 33.8 mg, 35%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.54-7.50 (m, 4H), 7.36-7.32 (m, 4H), 7.11-7.08 (t, J = 7.8 Hz, 4H), 6.73-6.70 (t, J = 7.8 Hz, 4H), 5.51-5.10 (m, 2H), 3.21-3.18 (m, 1H), 3.13-3.10 (m, 1H), 3.04-3.00 (m, 1H), 2.91-2.87 (m, 1H), 2.59-2.51 (m, 1H), 2.40-2.33 (m, 1H), 2.31-2.18 (m, 2H), 2.14-2.05 (m, 2H), 1.88-1.76 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 139.4, 139.3, 137.6, 137.0, 132.7, 132.3, 131.7, 131.4, 130.6, 130.5, 129.6, 126.3 (q, J_{CF} = 276.0 Hz), 126.2 (q, J_{CF} = 274.8 Hz), 121.9, 121.67, 121.62, 121.4, 116.0 (t, J_{CF} = 236.8 Hz), 115.9 (t, J_{CF} = 238.1 Hz), 45.63, 45.58, 45.3, 44.1, 44.0, 38.5 (q, J_{CF} = 27.3 Hz), 38.3 (t, J_{CF} = 21.5 Hz), 38.0 (q, J_{CF} = 27.6 Hz), 37.5 (t, J_{CF} = 21.7 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 3F), -63.3 (t, J = 10.5 Hz, 3F), -114.3--116.2 (m, 2F), -117.4--118.8 (m, 2F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{18}H_{16}Br_2F_5$ 484.9539; found 484.9547.

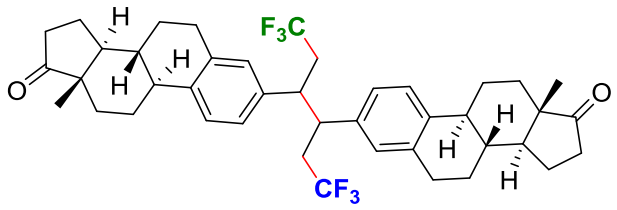
4,4'-(1,1,1,6,6-pentafluorohexane-3,4-diyl)dibenzonitrile **(32)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (100:1) as eluant) to offer the product. Pale yellow oil; 20.4 mg, 27%; anti : syn = 0.52 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.74-7.70 (m, 4H), 7.51-7.47 (m, 7.7H), 7.38-7.34 (m, 4H), 6.99-6.97 (t, J = 8.6 Hz, 7.7H), 5.55-5.34 (m,

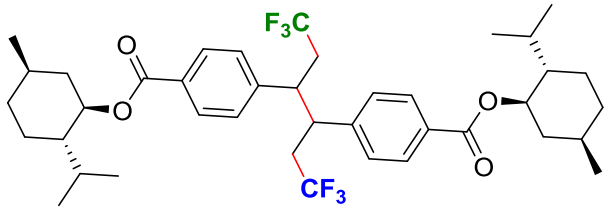
1.9H), 5.33-5.14 (m, 1H), 3.31-3.28 (m, 1.9H), 3.25-3.20 (m, 1.9H), 3.18-3.16 (m, 1H), 3.10-3.06 (m, 1H), 2.71-2.63 (m, 1.9H), 2.52-2.44 (m, 1H), 2.43-2.37 (m, 1.9H), 2.34-2.27 (m, 1H), 2.23-2.14 (m, 1.9H), 2.11-2.05 (m, 1H), 2.00-1.87 (m, 1.9H), 1.81-1.75 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 145.4, 145.3, 144.3, 144.0, 133.4, 133.1, 132.5, 132.3, 129.34, 129.30, 128.83, 128.82, 127.8 (q, J_{CF} = 268.4 Hz), 118.3, 118.1, 115.4, 115.3, 115.2 (t, J_{CF} = 230.8 Hz), 112.5, 112.2, 111.9, 111.7, 45.8, 45.7, 44.93, 44.88, 38.2 (t, J_{CF} = 22.3 Hz), 38.1 (q, J_{CF} = 27.2 Hz), 37.7 (q, J_{CF} = 27.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (t, J = 10.6 Hz, 6F), -63.4 (t, J = 10.6 Hz, 3F), -114.5--116.2 (m, 2F), -117.3--118.2 (m, 4F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{20}H_{16}F_5N_2$ 379.1234; found 379.1227.

3,3'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)bis(13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one) **(34)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (20:1) as eluant) to offer the product. Pale yellow oil; 74.0 mg, 53%; anti : syn = 1 : 1 (the diastereoisomers could not be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.16-7.15 (d, J = 8.0 Hz, 2H), 7.14-7.12 (d, J = 8.1 Hz, 2H), 6.67-6.66 (d, J = 8.0 Hz, 2H), 6.57-6.56 (d, J = 8.1 Hz, 2H), 6.54 (s, 2H), 6.38 (s, 2H), 3.30-3.28 (m, 4H), 2.93-2.78 (m, 8H), 2.53-2.46 (m, 8H), 2.41-2.38 (m, 5H), 2.30-2.25 (m, 9H), 2.18-2.12 (m, 3H), 2.08-2.05 (m, 4H), 1.98-1.95 (m, 4H), 1.65-1.58 (m, 12H), 1.54-1.46 (m, 14H), 0.923 (s, 6H), 0.921 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 221.1, 138.9, 138.8, 135.8, 135.7, 135.2, 135.1, 130.4, 130.3, 127.1, 127.0, 126.8 (q, J_{CF} = 275.8 Hz), 124.72, 124.69, 50.60, 50.57, 48.1, 44.4, 44.3, 42.9, 42.8, 38.2, 37.3 (q, J_{CF} = 27.3 Hz), 37.2 (q, J_{CF} = 27.6 Hz), 36.0, 31.7, 29.5, 26.64, 26.62, 25.8, 25.7, 21.7, 14.0; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.1 (m, 12F).[1]

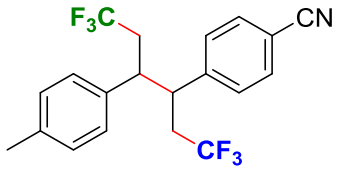
bis((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)
4,4'-(1,1,1,6,6,6-hexafluorohexane-3,4-diyl)dibenzoate **(35)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified

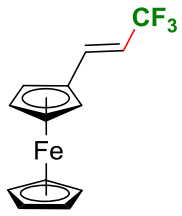
by column chromatography (petroleum ether/ethyl acetate (30:1) as eluant) to offer the product. Colorless oil; 90.9 mg, 64%; syn (one of the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.90-7.88 (t, J = 8.1 Hz, 4H), 6.91-6.88 (d, J = 8.2 Hz, 4H), 4.93-4.88 (m, 2H), 3.45-3.40 (m, 2H), 2.63-2.54 (m, 2H), 2.43-2.36 (m, 2H), 2.12-2.09 (m, 2H), 1.96-1.93 (m, 2H), 1.73-1.71 (d, J = 12.1 Hz, 4H), 1.57-1.51 (m, 4H), 1.15-1.04 (m, 4H), 0.92-0.90 (m, 14H), 0.79-0.78 (d, J = 6.9 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 165.8, 142.8, 130.3, 129.5, 129.0, 126.3 (q, J_{CF} = 274.9 Hz), 75.1, 47.3, 43.8 (q, J_{CF} = 10.9 Hz), 41.0, 37.6 (q, J_{CF} = 27.4 Hz), 34.4, 31.5, 26.5, 23.6, 22.1, 20.9, 16.5; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.2 (t, J = 10.6 Hz, 6F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{40}H_{53}F_6O_4$ 711.3848; found 711.3858.

4-(1,1,1,6,6,6-hexafluoro-4-(p-tolyl)hexan-3-yl)benzonitrile **(36)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (100:1) as eluant) to offer the product. Colorless oil; 25% (determined by ^{19}F NMR, this compound could not be effectively isolated from the reaction mixture); anti (one of the diastereoisomers could be isolated); ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.70-7.69 (d, J = 8.0 Hz, 2H), 7.36-7.35 (d, J = 8.1 Hz, 2H), 7.20-7.18 (d, J = 8.0 Hz, 2H), 7.08-7.07 (d, J = 8.1 Hz, 2H), 3.14-3.11 (m, 1H), 3.05-3.01 (m, 1H), 2.36 (m, 3H), 2.31-2.25 (m, 1H), 2.22-2.14 (m, 2H), 2.0.4-1.99 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 146.3, 137.9, 136.4, 133.0, 130.1, 128.9, 127.7, 126.2 (q, J_{CF} = 264.8 Hz), 118.5, 111.9, 46.0, 45.2, 38.2 (q, J_{CF} = 27.3 Hz), 38.0 (q, J_{CF} = 27.0 Hz), 21.2; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -63.3 (t, J = 10.5 Hz, 3F), -63.5 (t, J = 10.6 Hz, 3F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{20}H_{18}F_6N$ 386.1343; found 386.1349.

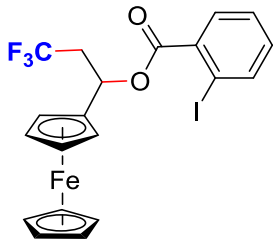
(E)-1-ferrocenyl-3,3,3-trifluoroprop-1-ene **(38)**



This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (80:1) as eluant) to offer the product. Orange solid; 24.7 mg, 22%; ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 6.98-6.95 (m, 1H), 5.82-5.76 (m, 1H), 4.42-4.41 (t, J = 1.8 Hz, 2H), 4.352-4.346 (t, J

= 1.7 Hz, 2H), 4.15 (m, 5H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 137.3 (q, J_{CF} = 6.8 Hz), 123.9 (q, J_{CF} = 276.5 Hz), 112.5 (q, J_{CF} = 33.2 Hz), 78.2, 77.3, 77.1, 76.9, 70.4, 69.6, 68.0; ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -62.7 (d, J = 6.8 Hz, 3F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{13}H_{12}F_3Fe$ 281.0241; found 281.0235.

1-ferrocenyl-3,3,3-trifluoropropyl 2-iodobenzoate (**39**)

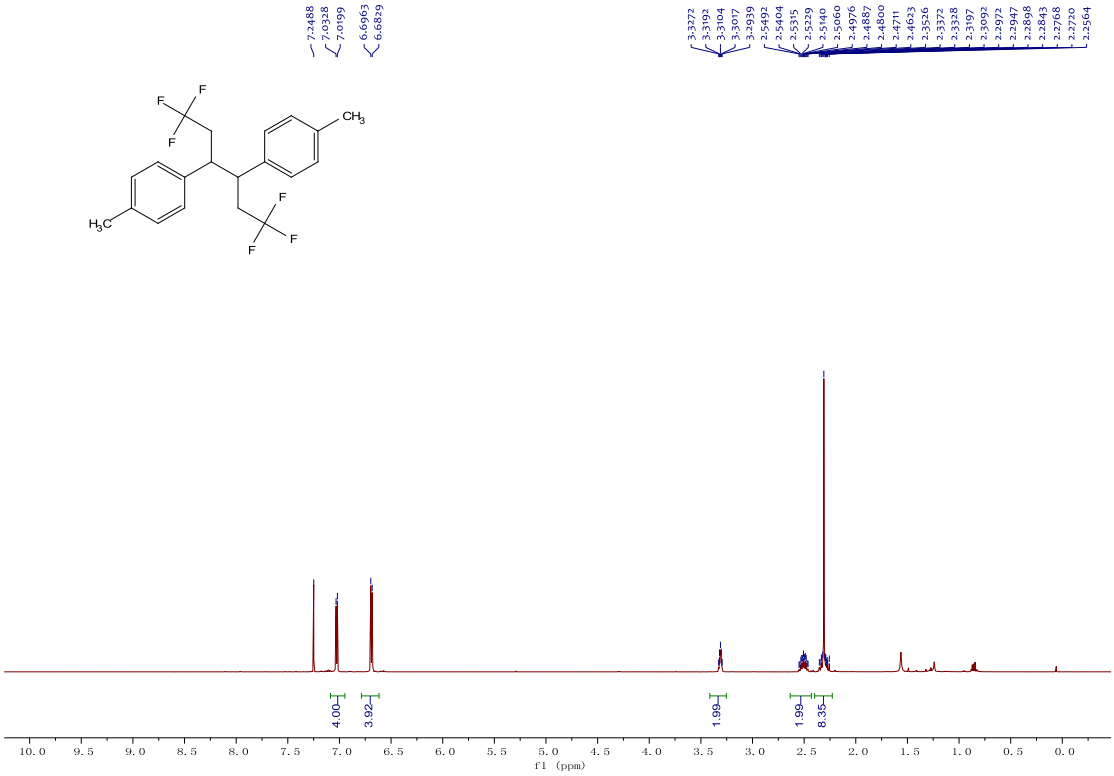


This compound was prepared according to typical reaction procedure of photoredox-catalyzed three-component alkene carbotrifluoromethylation and purified by column chromatography (petroleum ether/ethyl acetate (8:1) as eluant) to offer the product. Orange solid; 12.7 mg, 6%; ^{1}H NMR (600 MHz, $CDCl_3$): δ [ppm] = 7.99-7.97 (d, J = 7.9 Hz, 1H), 7.77-7.76 (dd, J = 7.8, 1.3 Hz, 1H), 7.39-7.37 (t, J = 7.6 Hz, 1H), 7.15-7.12 (td, J = 7.9, 1.5 Hz, 1H), 6.43-6.40 (dd, J = 7.9, 4.1 Hz, 1H), 4.47-4.46 (d, J = 1.3 Hz, 1H), 4.22-4.18 (m, 8H), 2.94-2.88 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ [ppm] = 165.3, 141.5, 134.5, 132.9, 130.9, 128.0, 94.5, 85.3, 69.0, 68.9, 68.8, 68.7, 67.3, 67.2, 66.0, 38.9 (q, J_{CF} = 28.1 Hz); ^{19}F NMR (564 MHz, $CDCl_3$): δ [ppm] = -64.2 (t, J = 10.5 Hz, 3F); HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{20}H_{17}F_3FeIO_2$ 528.9575; found 528.9574.

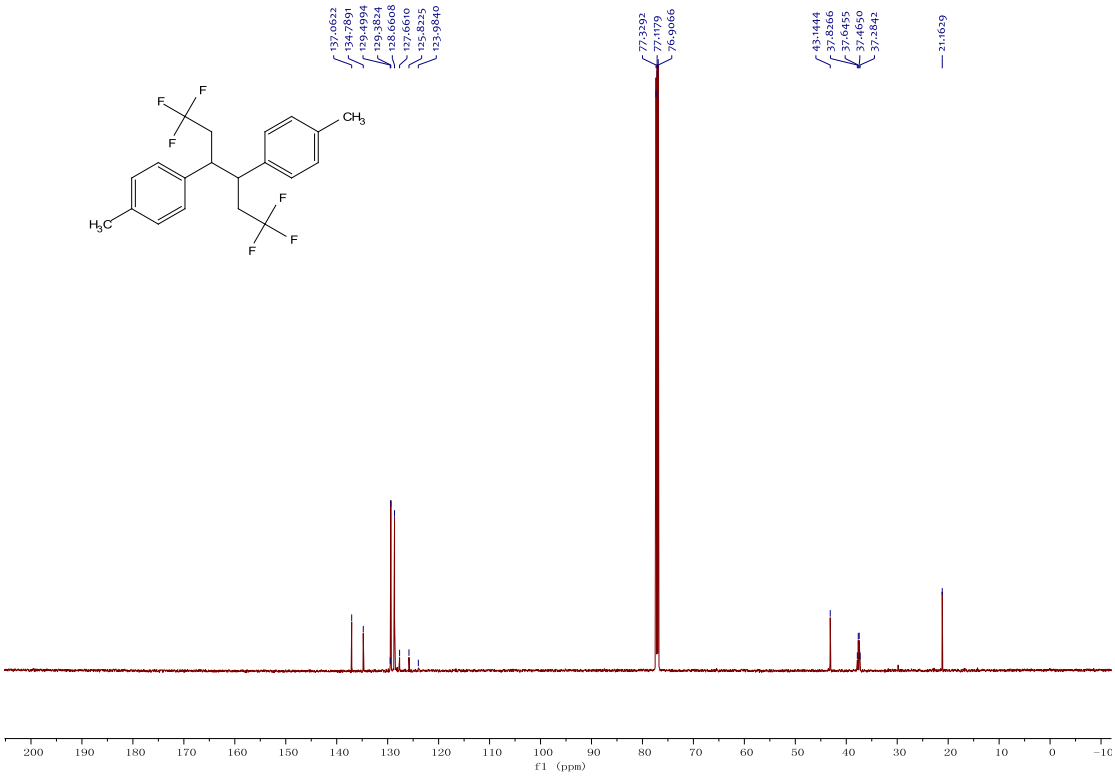
Reference:

[1] D. Louvel, A. Souibgui, A. Taponard, J. Rouillon, M. b. Mosbah, Y. Moussaoui, G. Pilet, L. Khrouz, C. Monnereau, J. C. Vantourout, A. Tlili, *Adv. Synth. Catal*., 2022, **364**, 139-148.

11. NMR Spectra of products

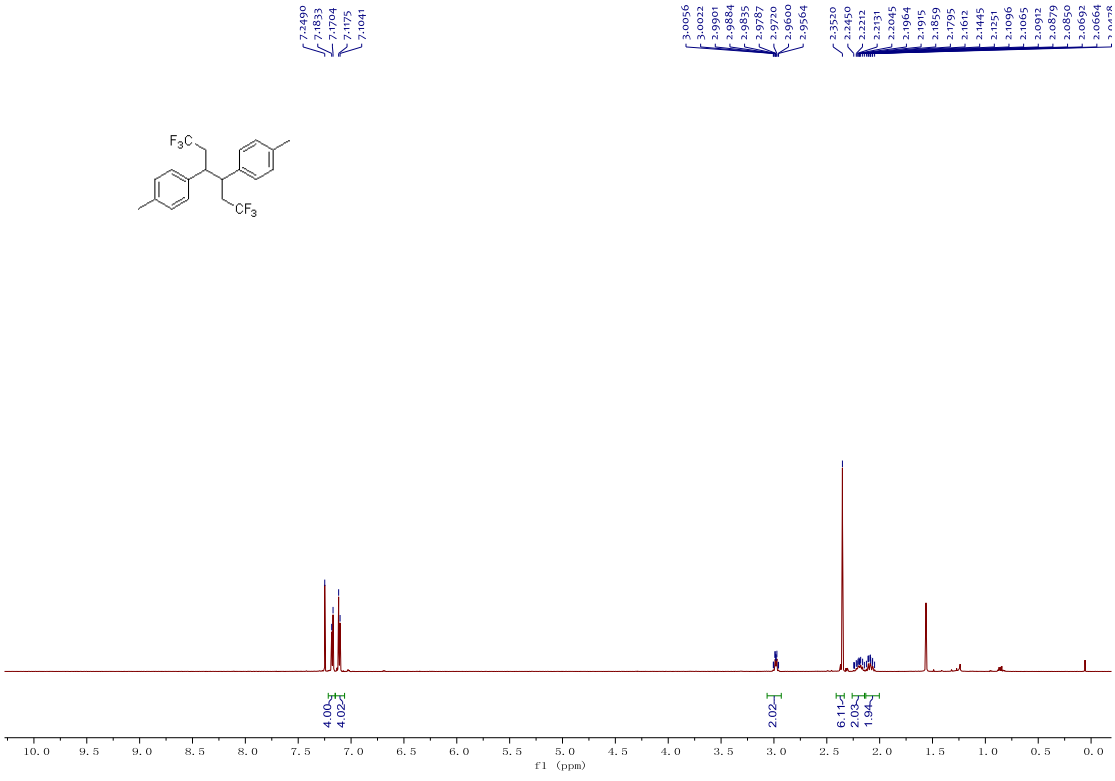


^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **4 (**syn**)**

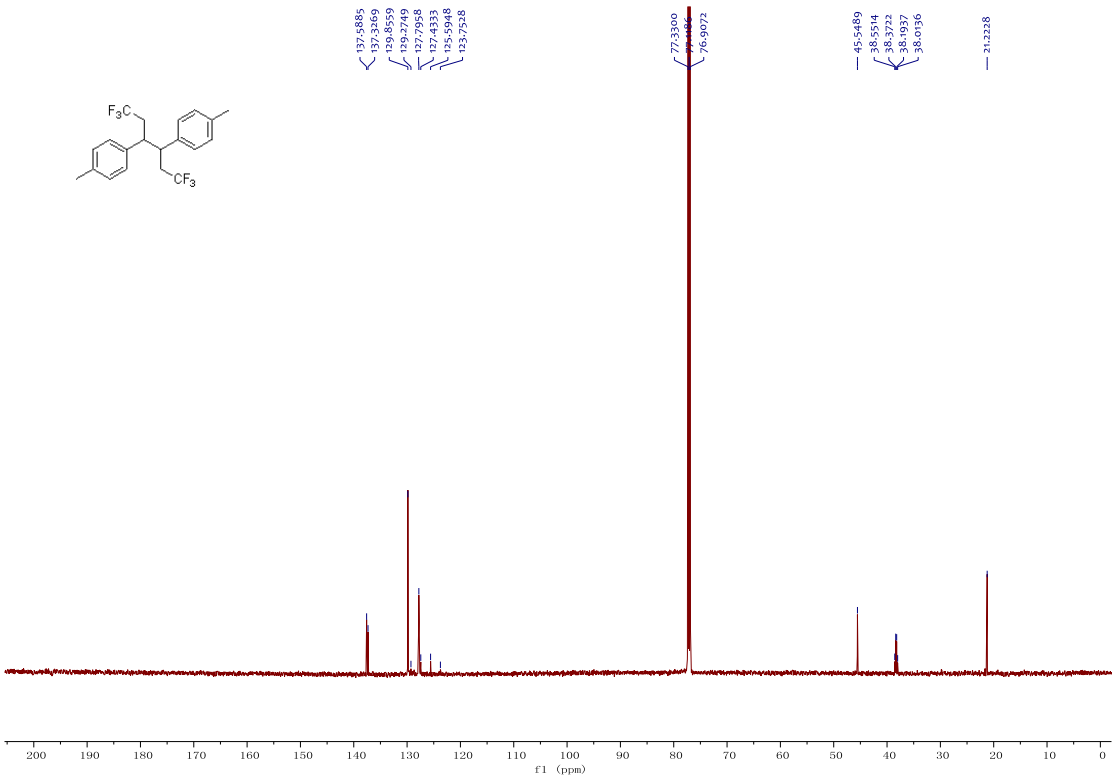


^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **4** (syn)

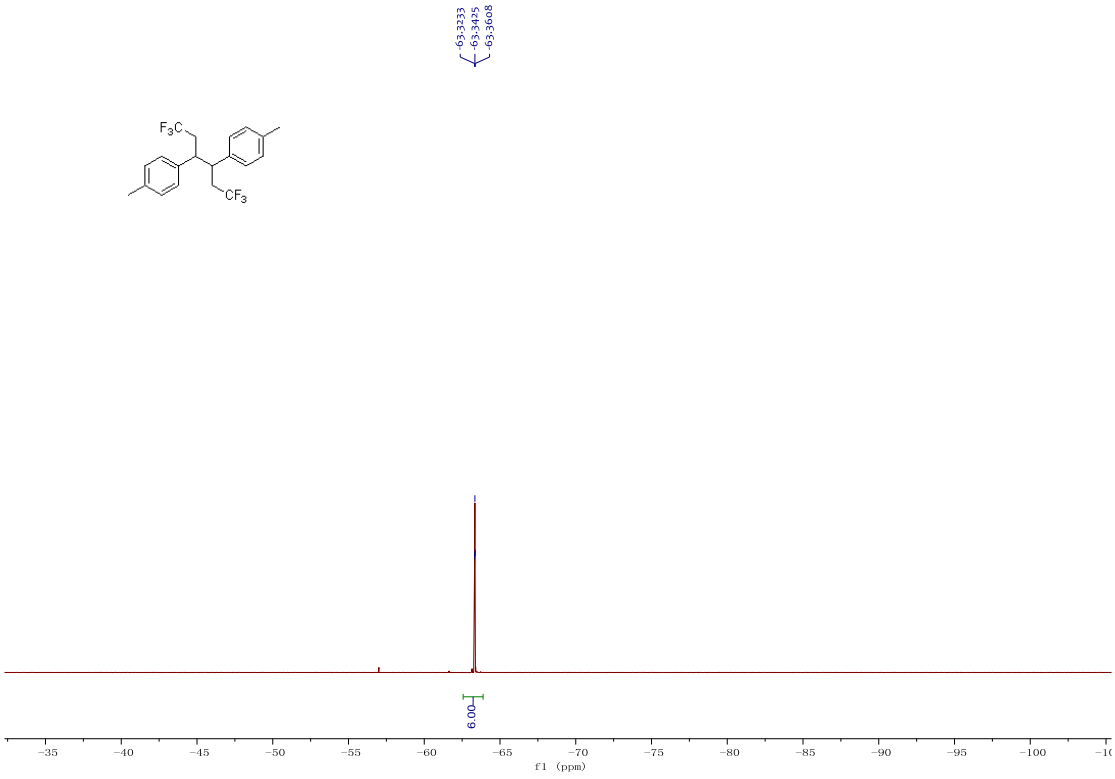
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **4** (syn)



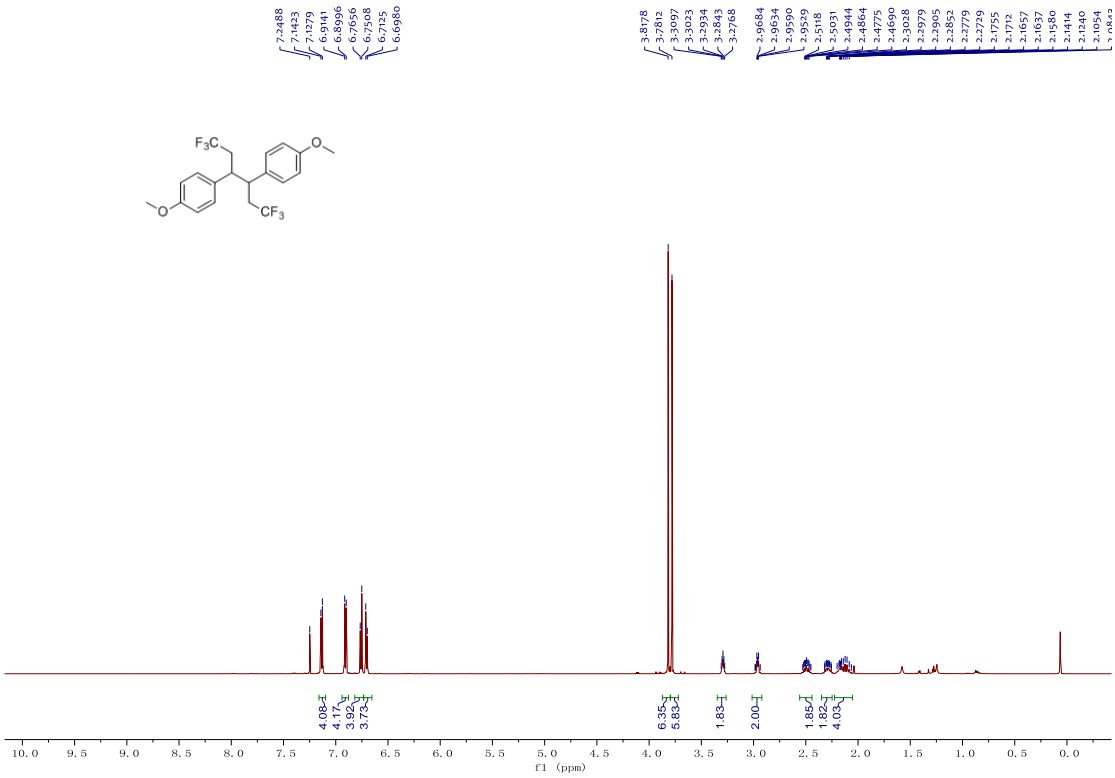
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **4** (anti)



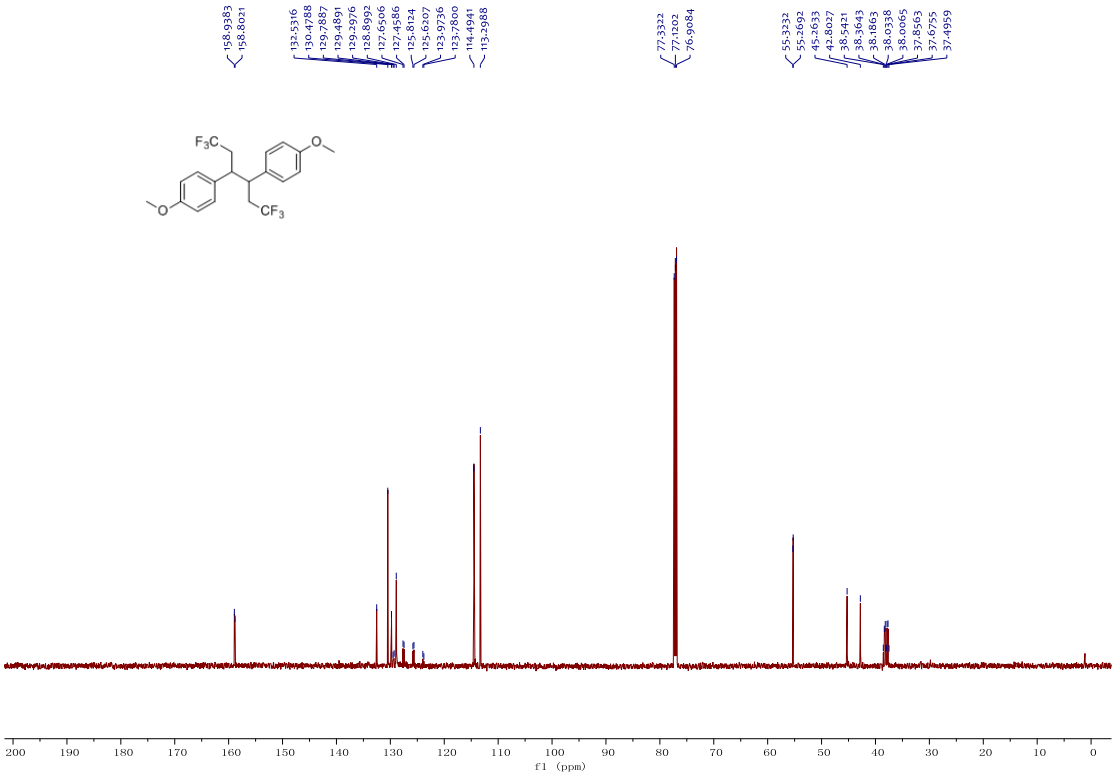
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **4** (anti)



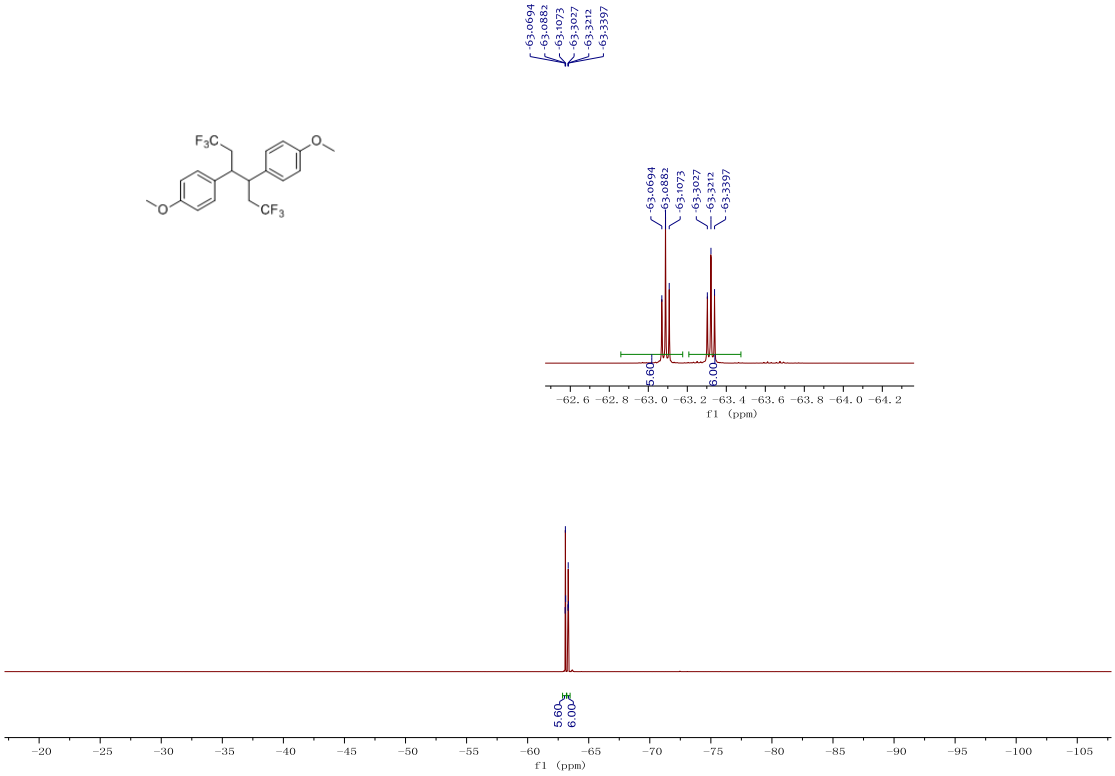
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **4** (anti)



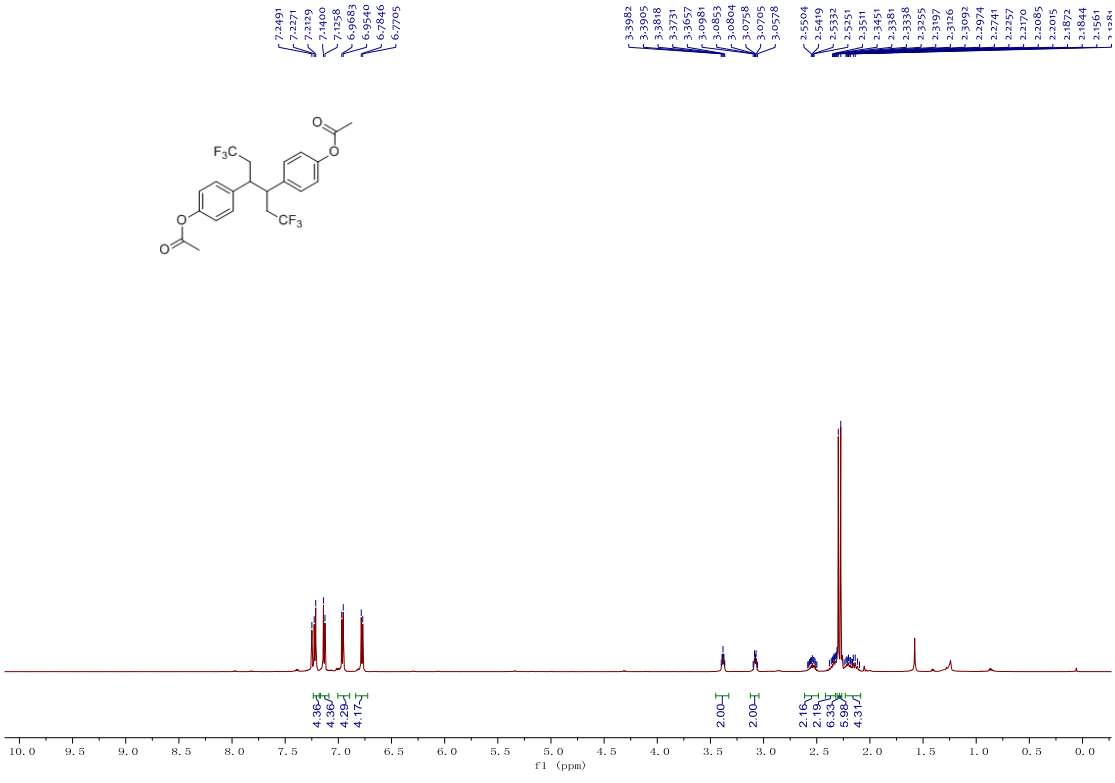
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **5**



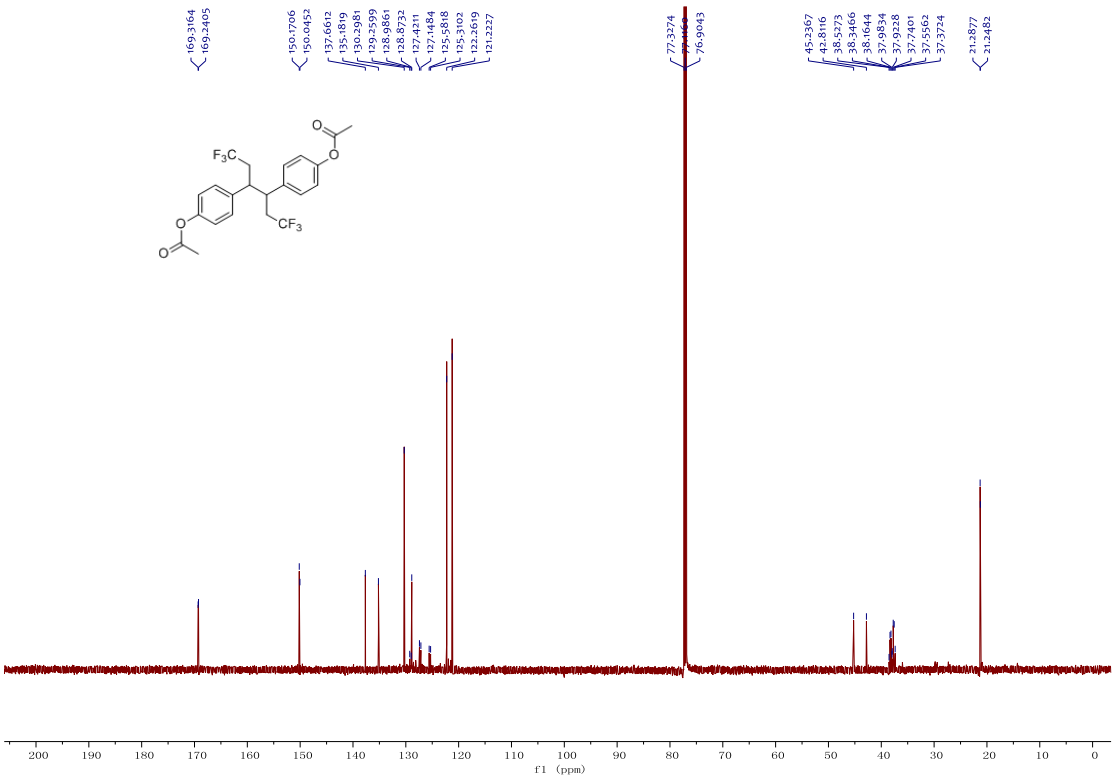
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **5**



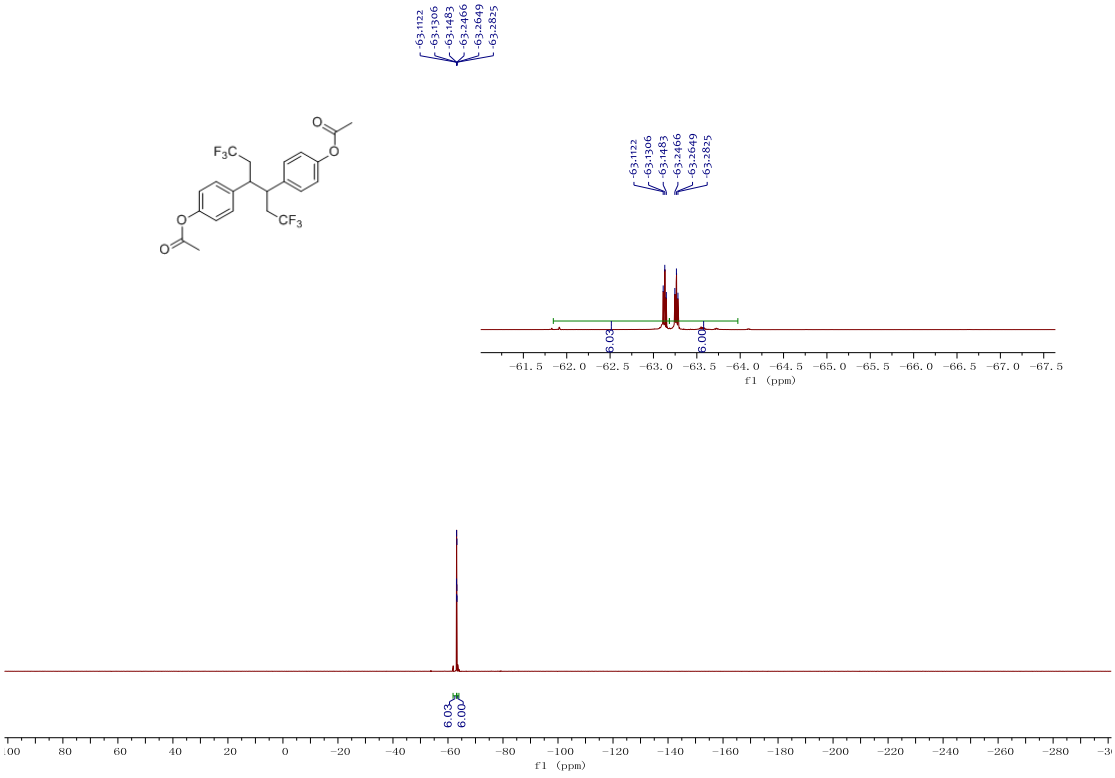
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **5**



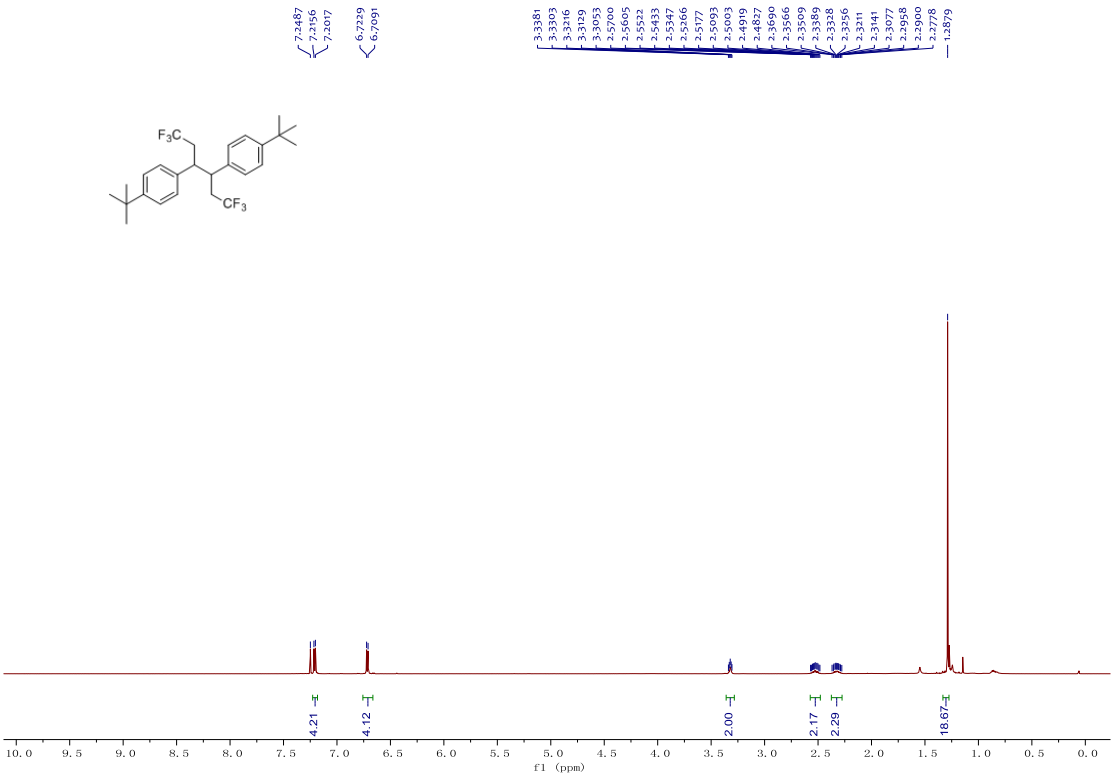
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **6**



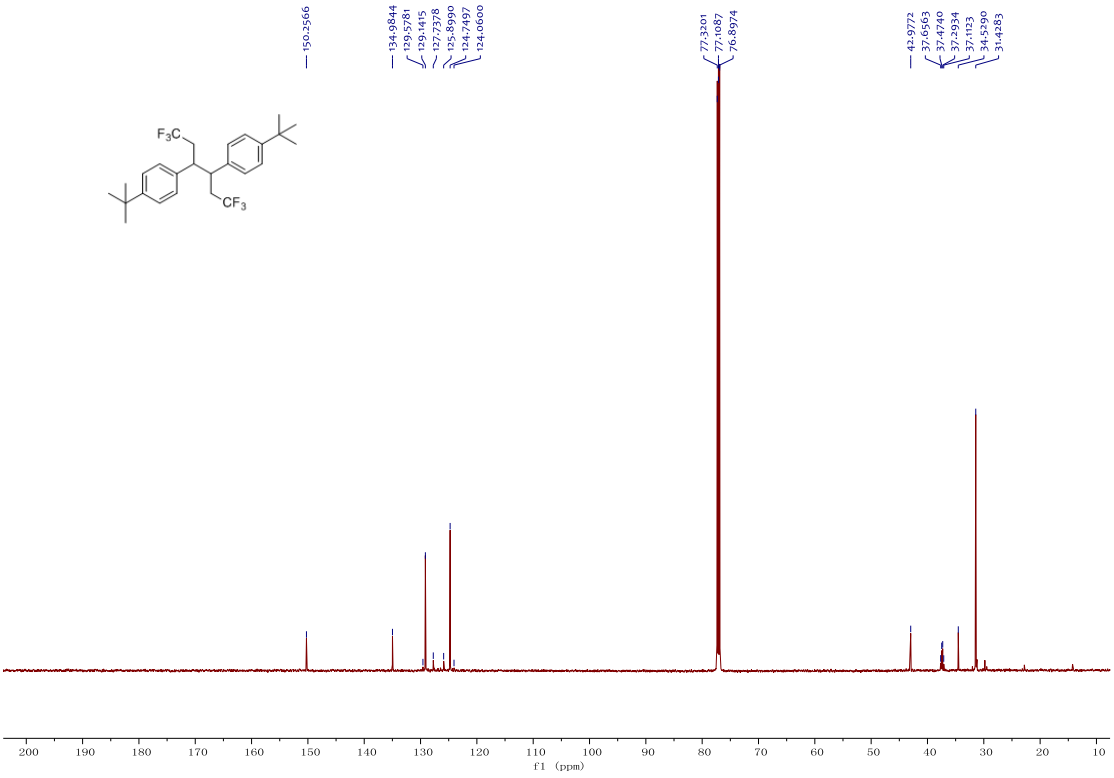
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **6**



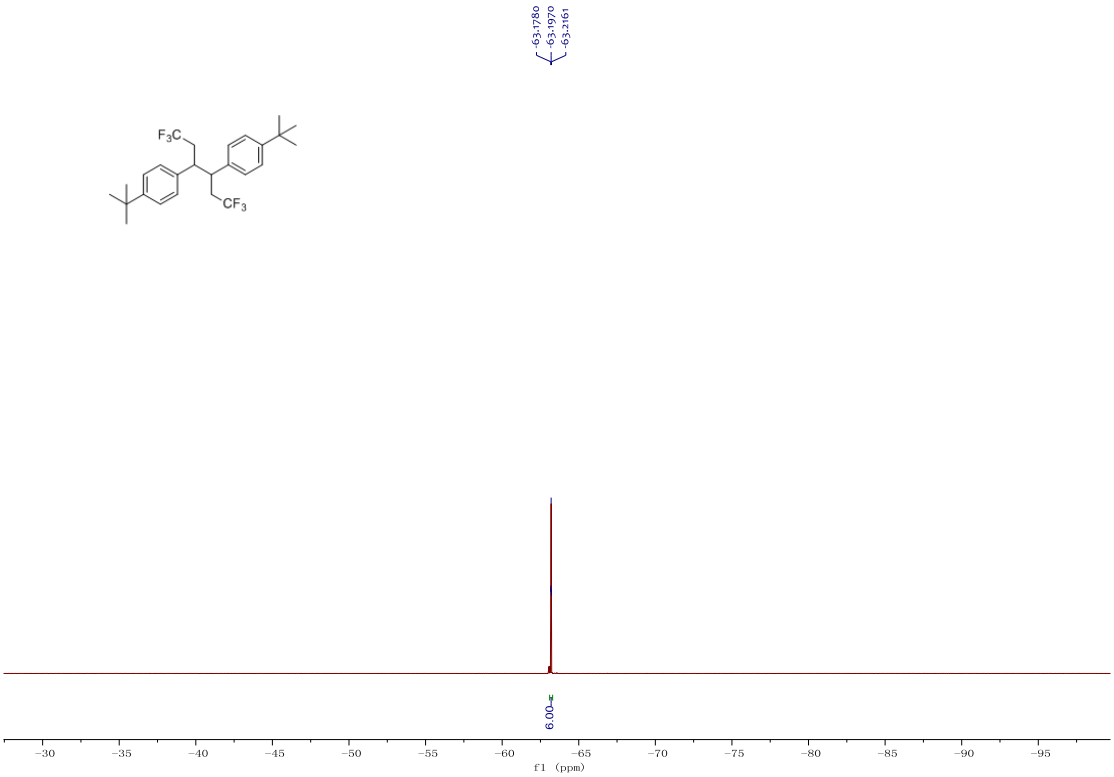
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **6**



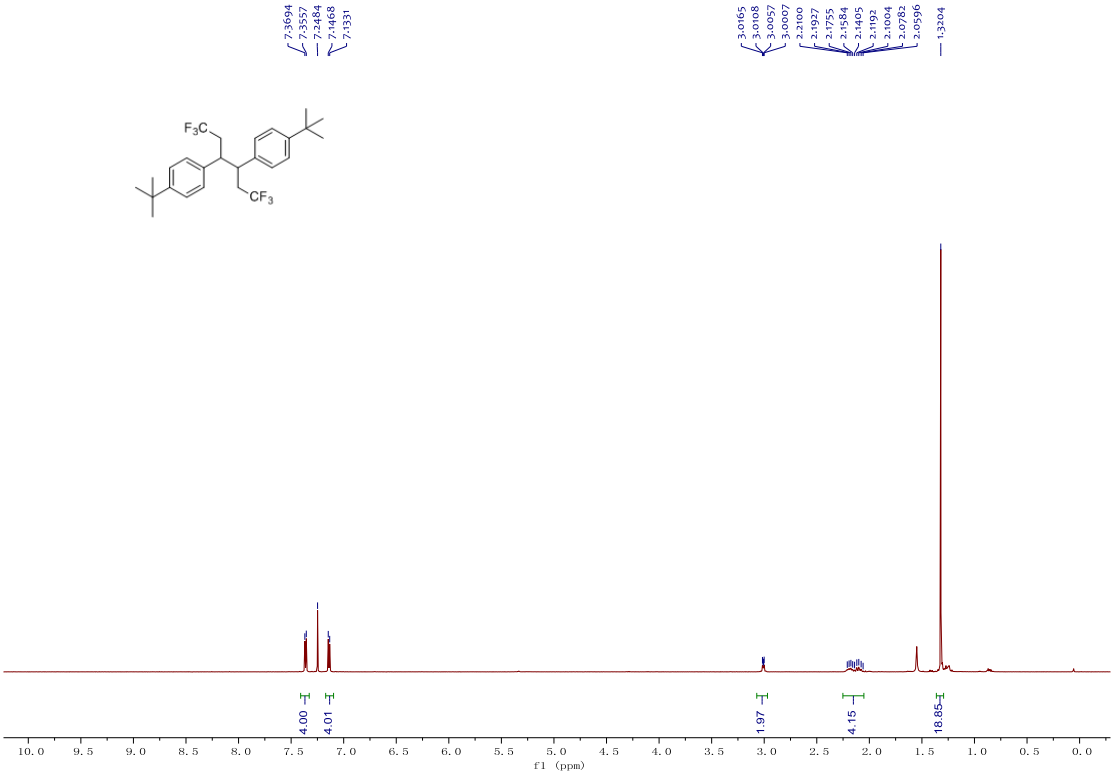
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **7** (syn)



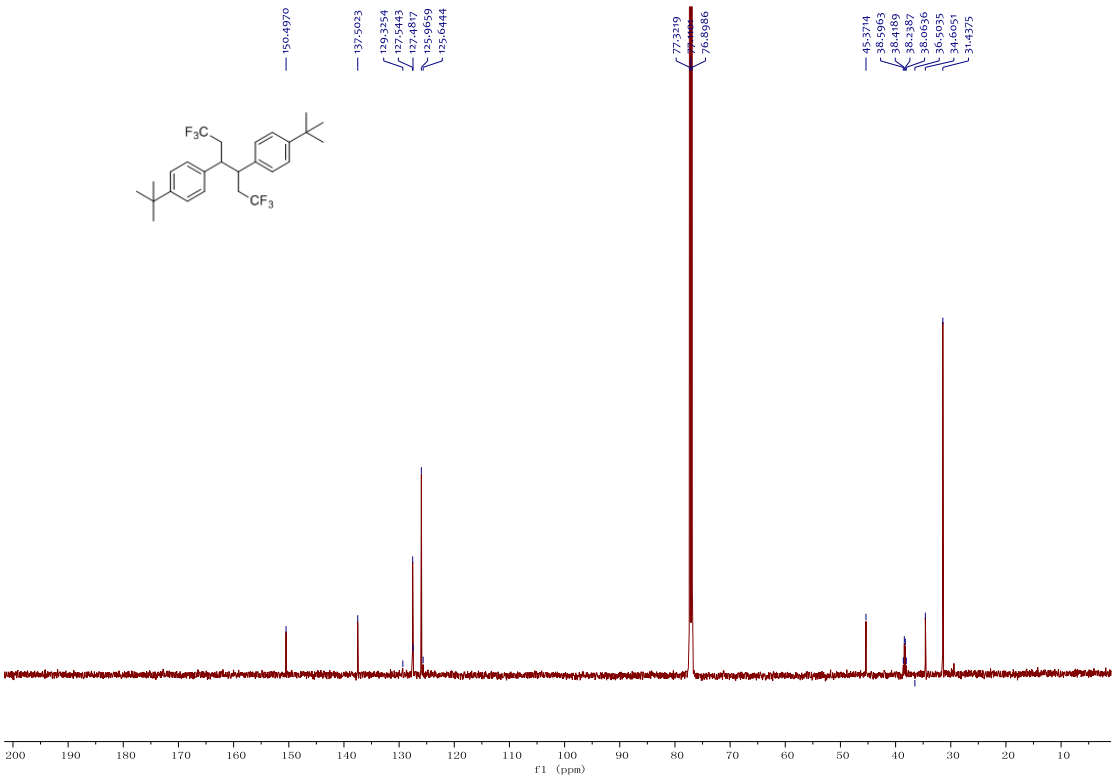
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **7** (syn)



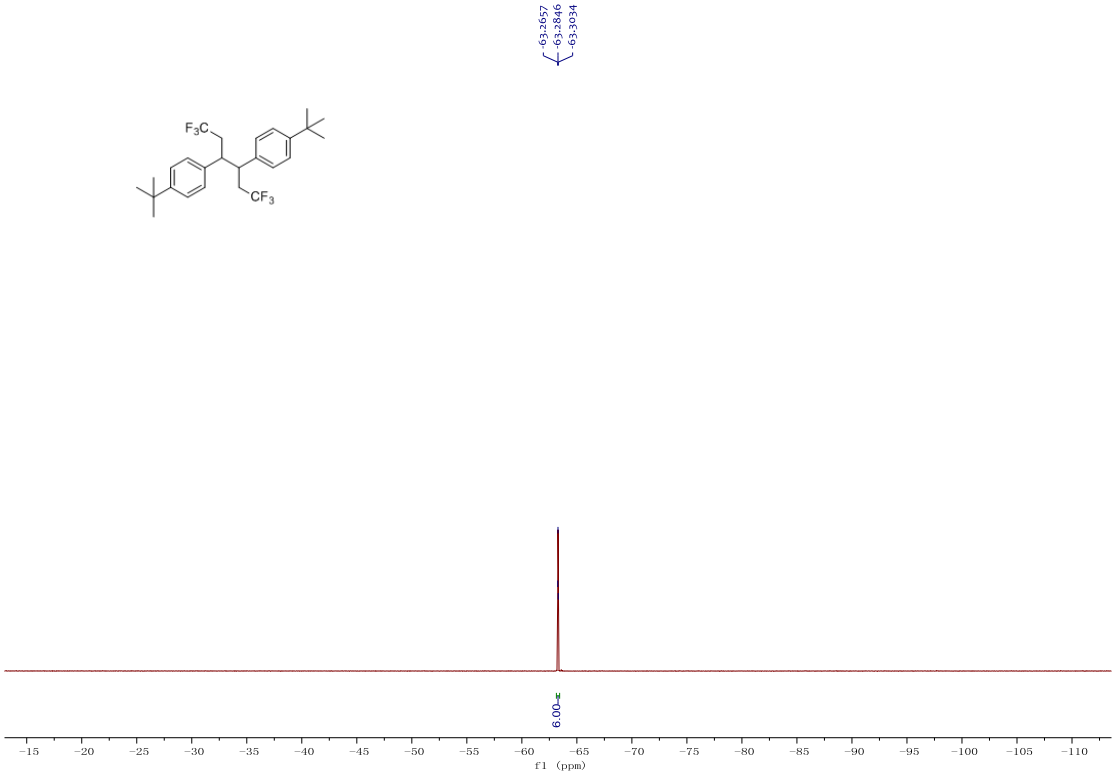
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **7** (syn)



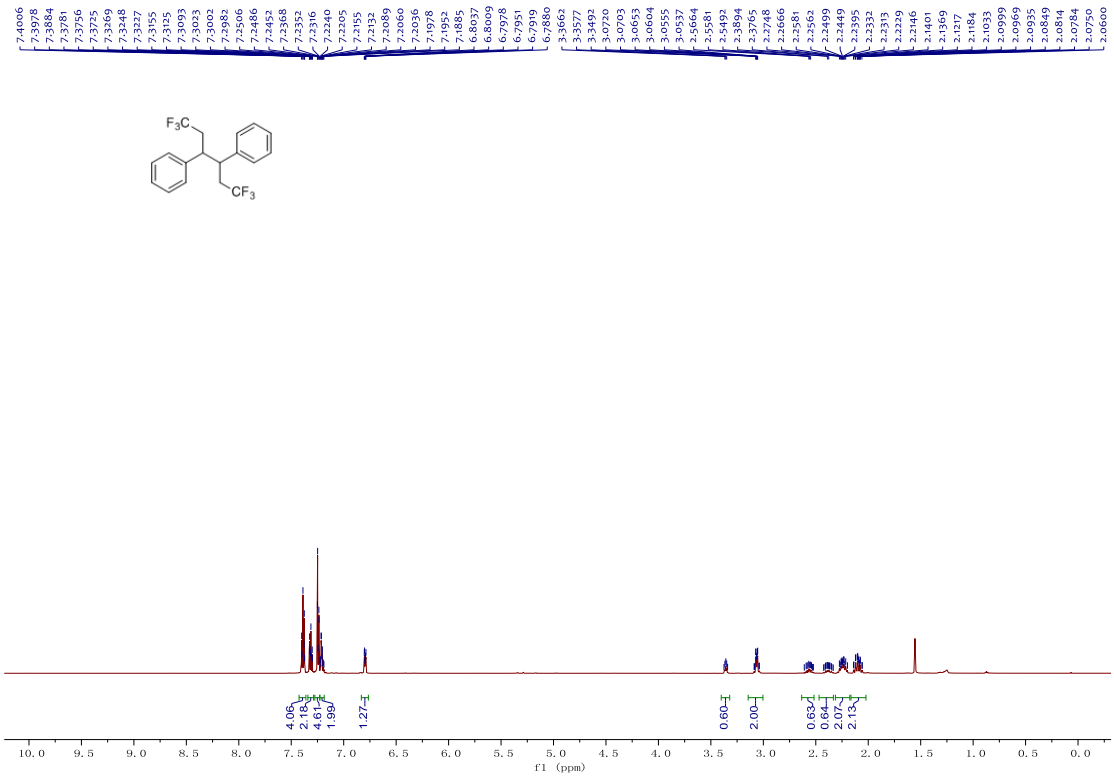
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **7** (anti)



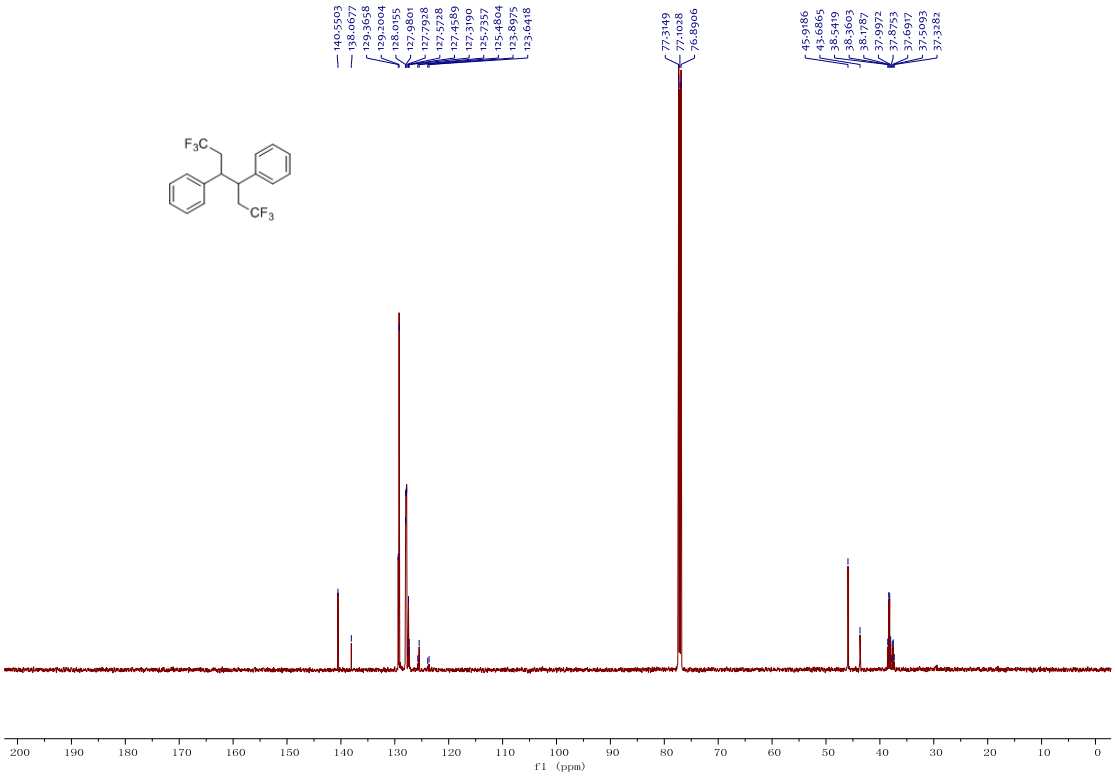
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **7** (anti)



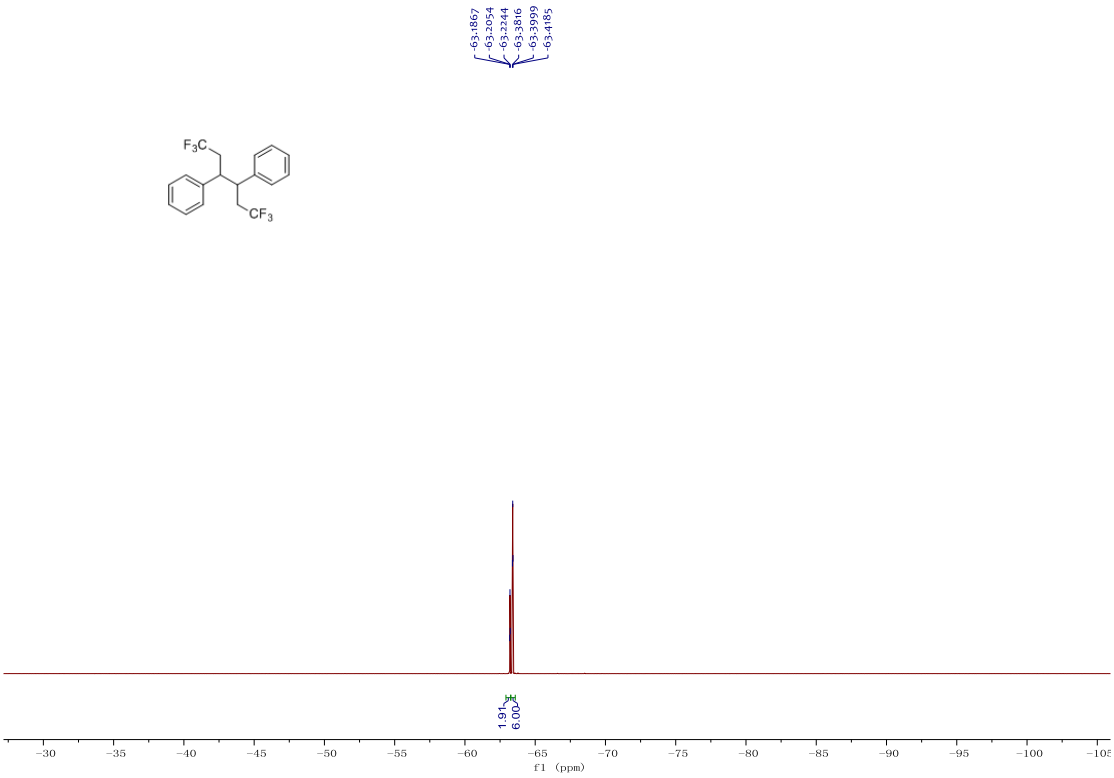
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **7** (anti)



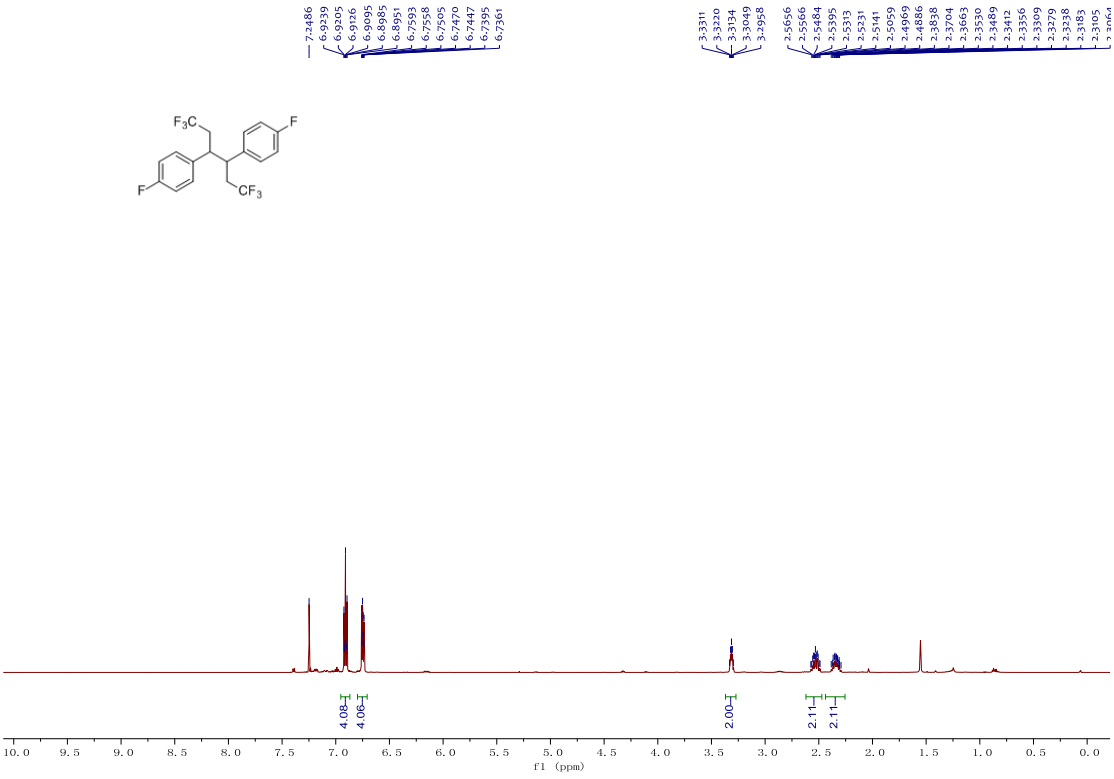
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **8**



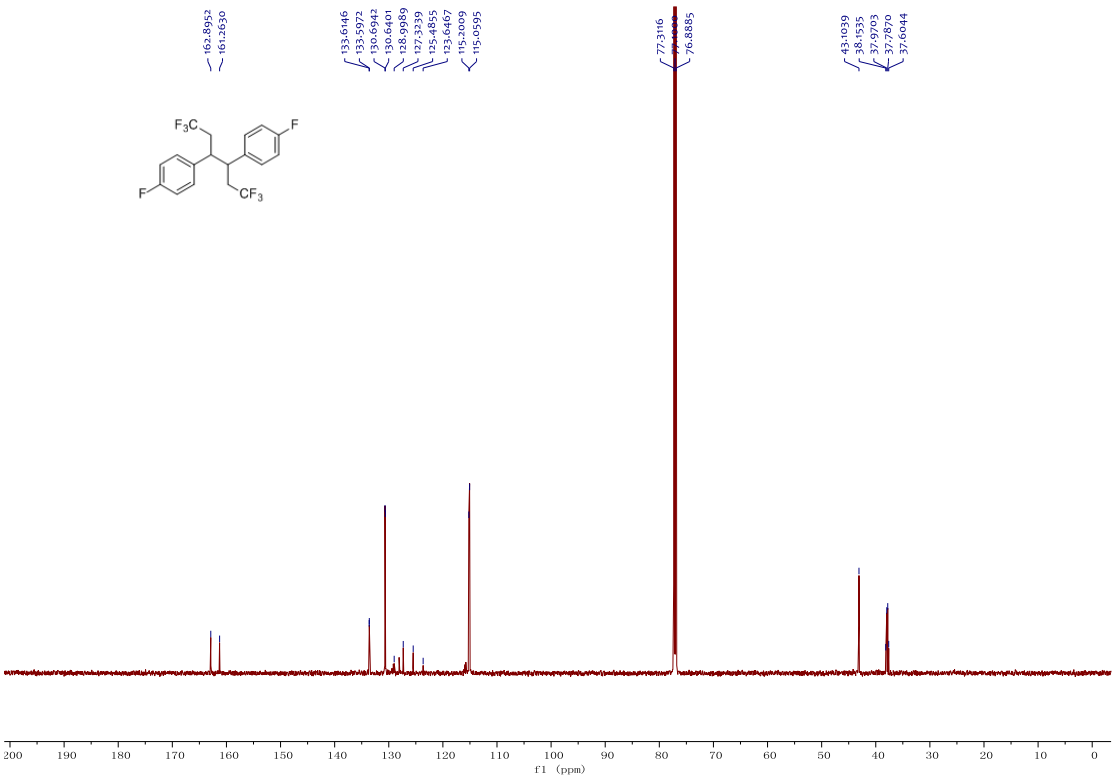
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **8**



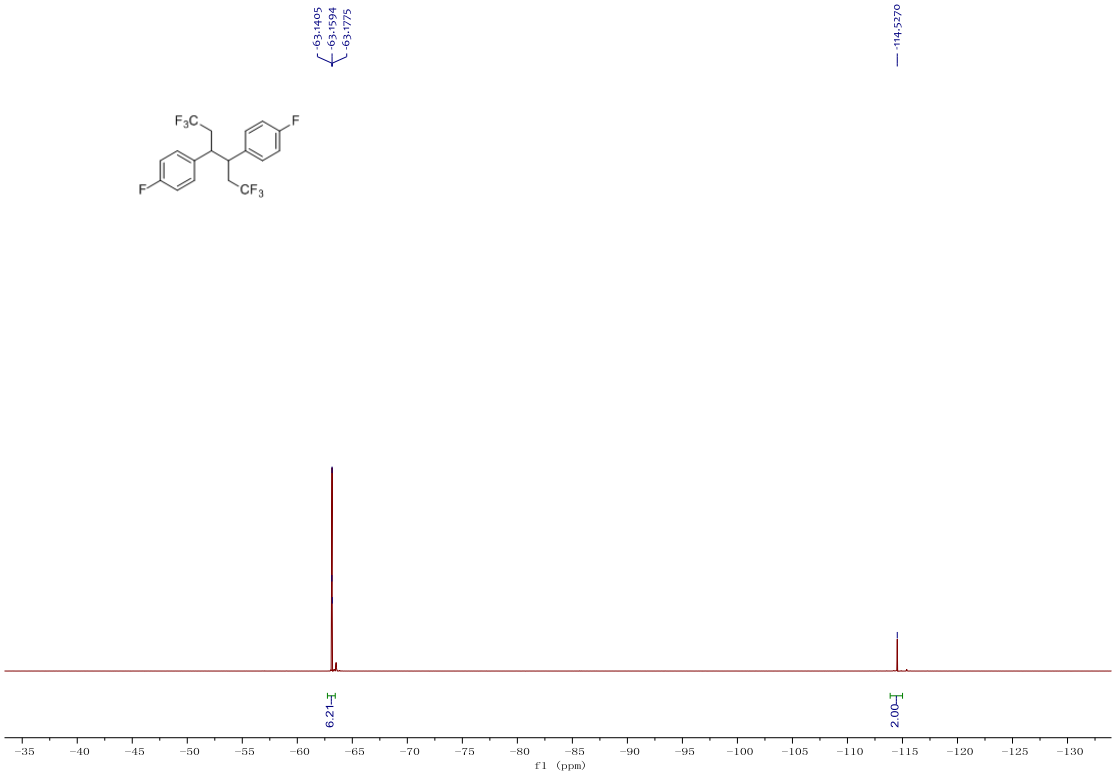
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **8**



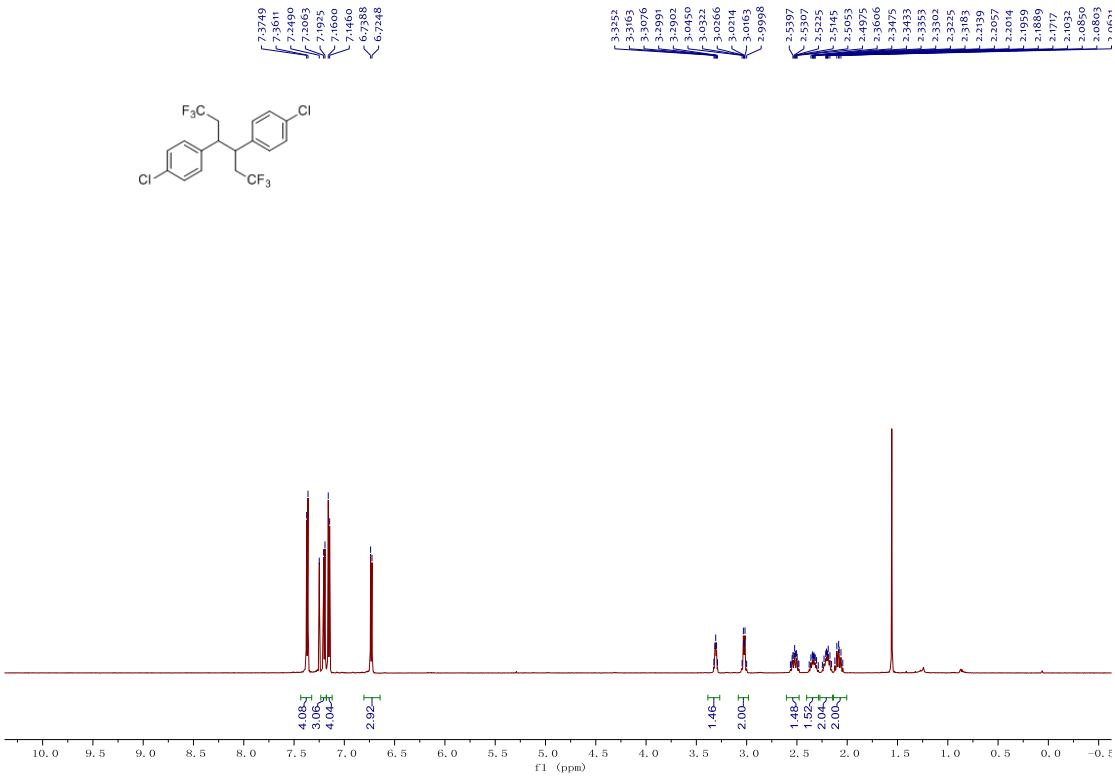
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9**



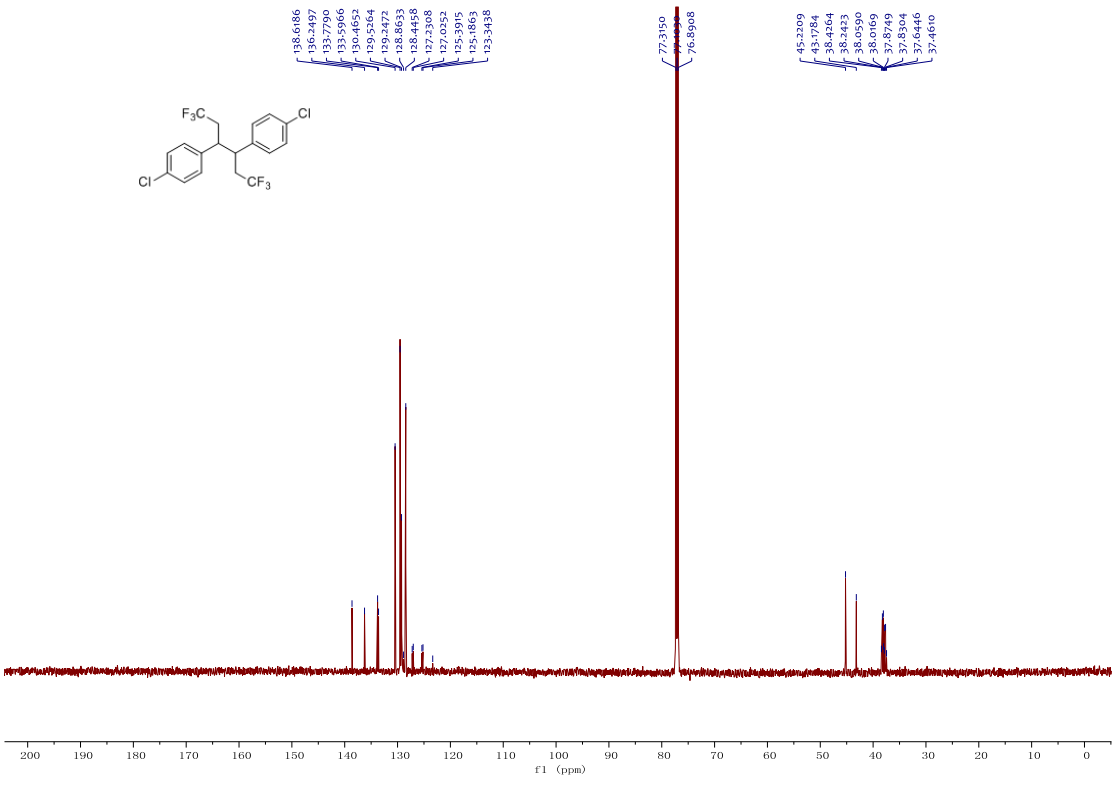
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **9**



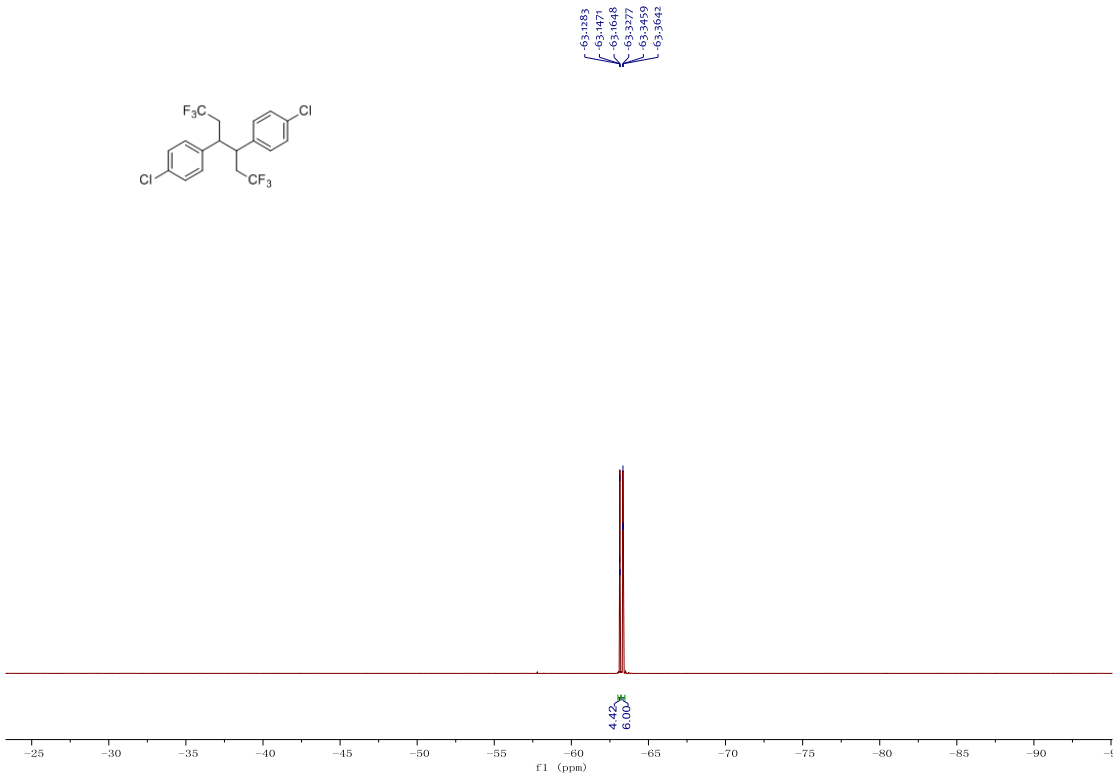
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **9**



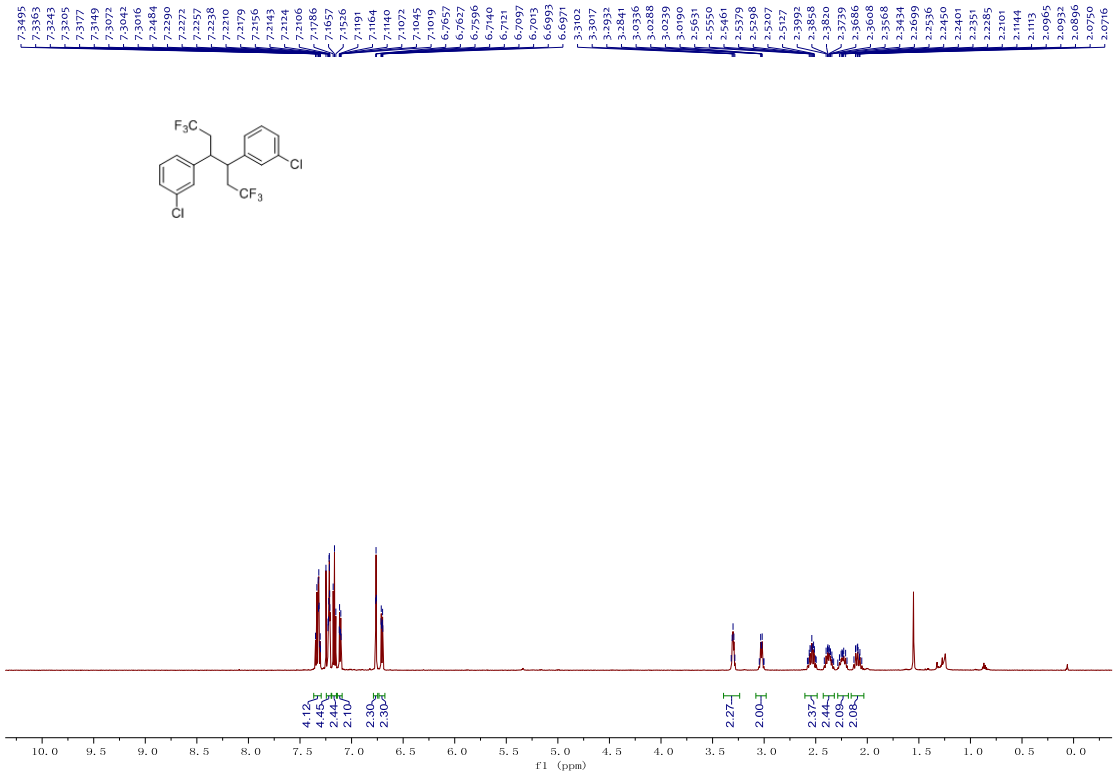
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **10**



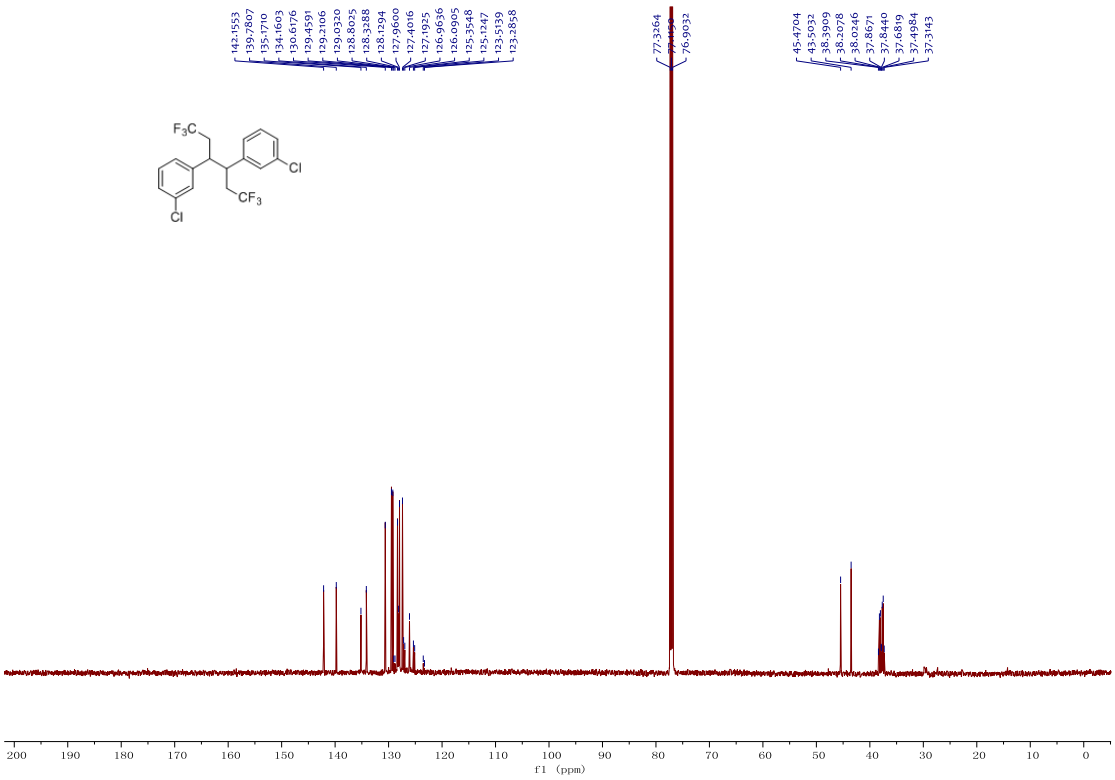
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **10**



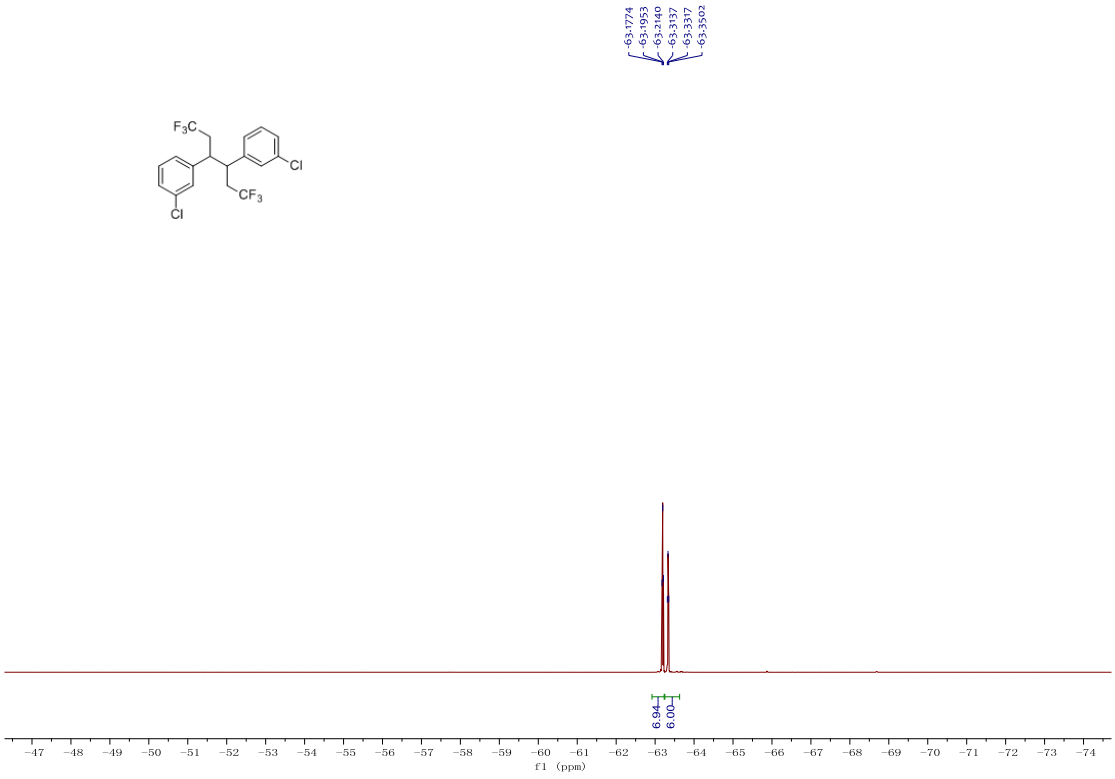
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **10**



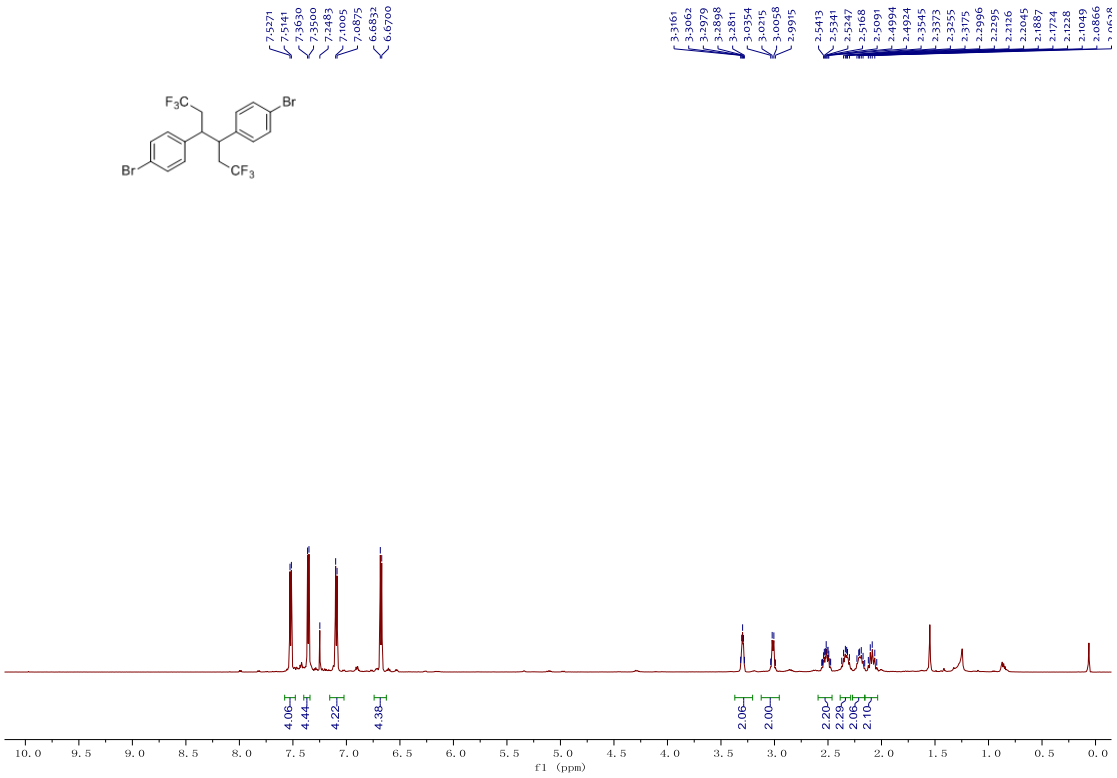
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **11**



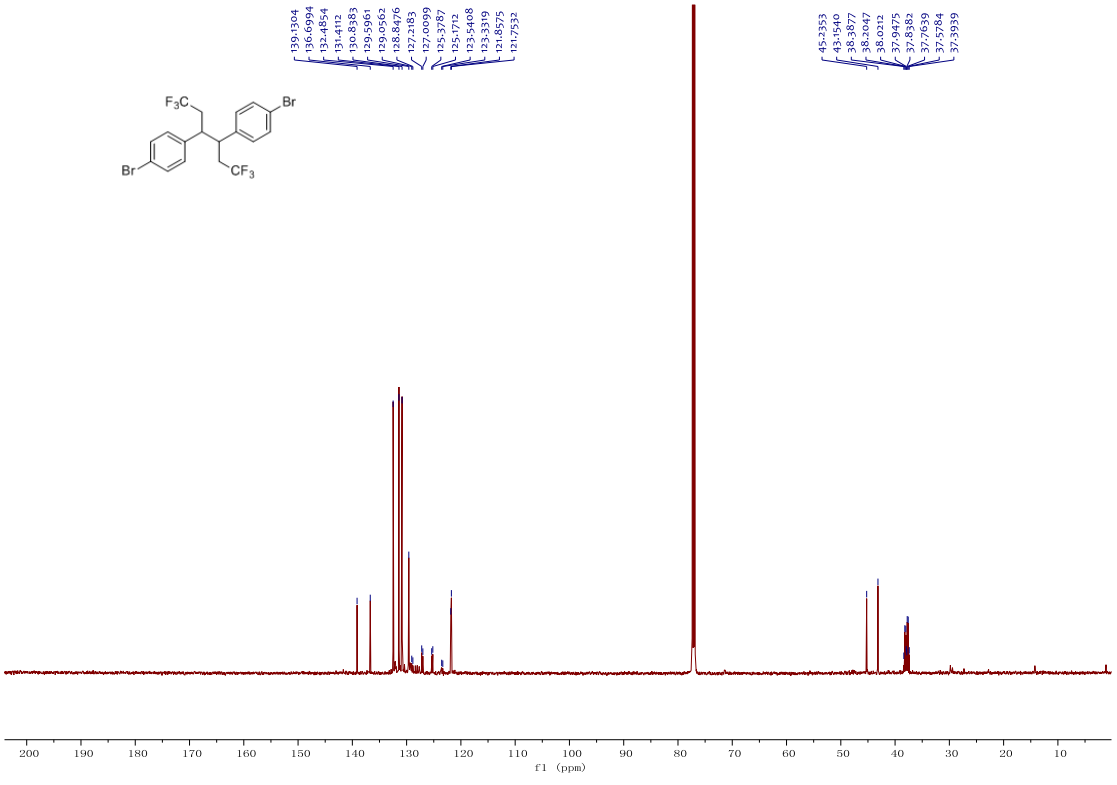
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **11**



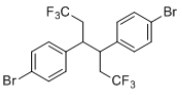
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **11**



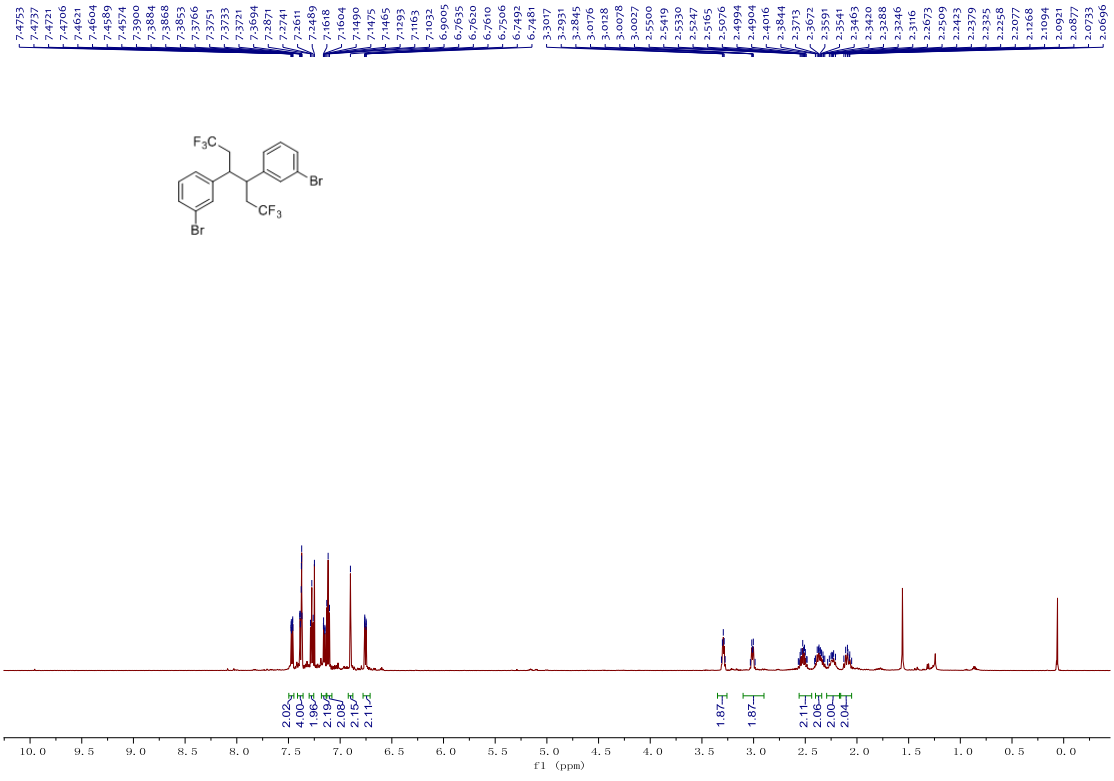
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **13**



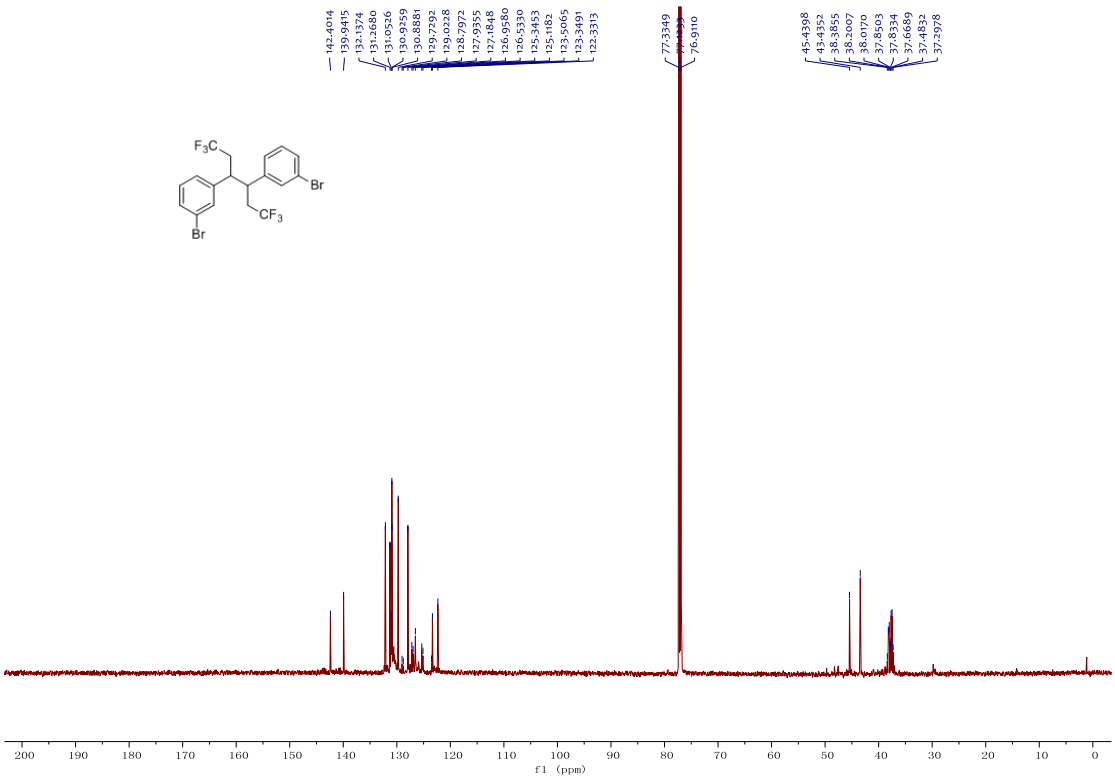
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **13**



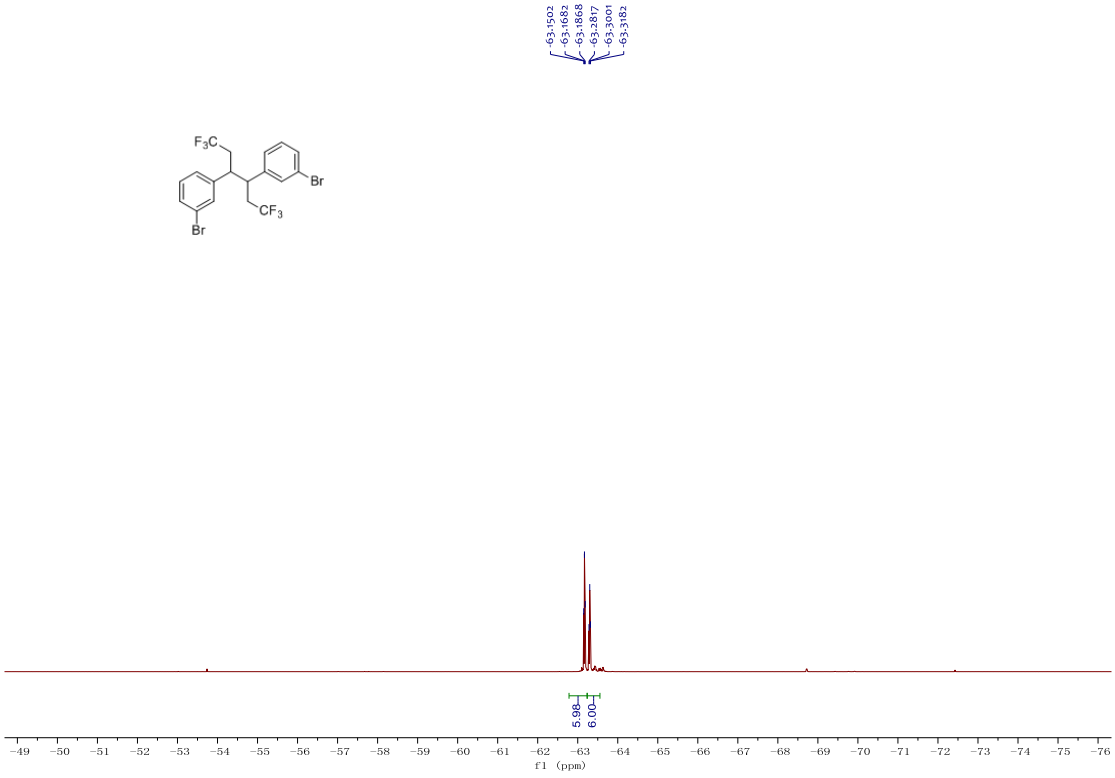
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **13**



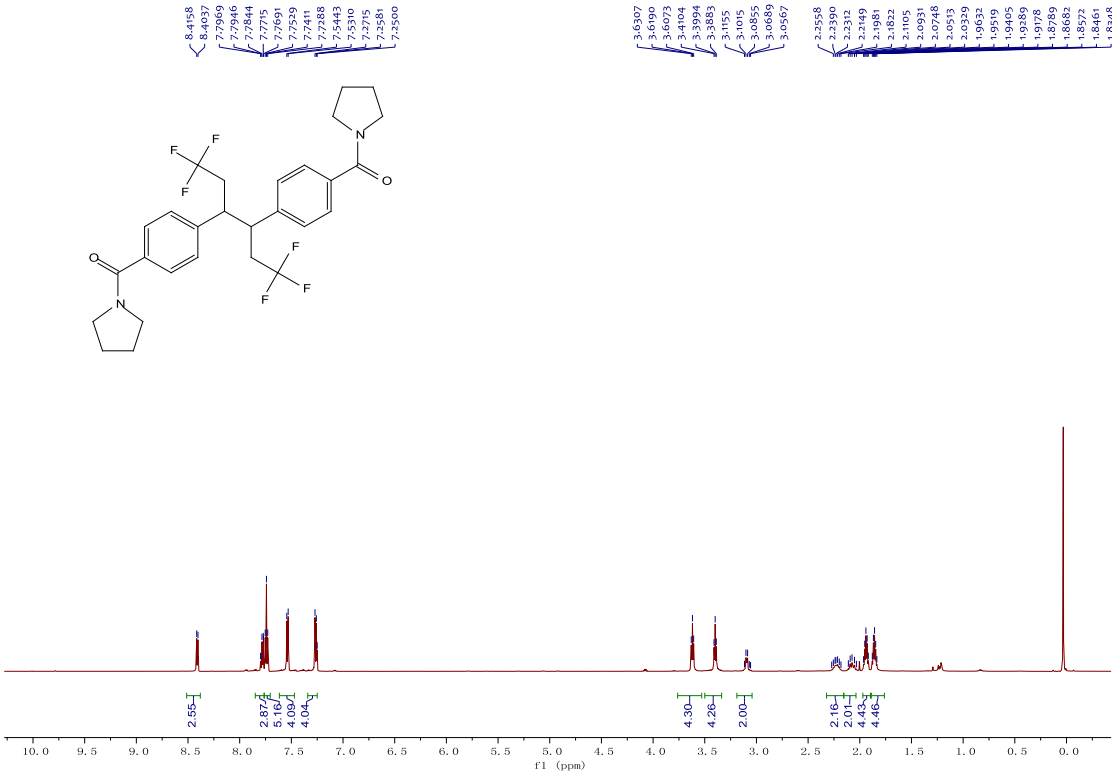
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **14**



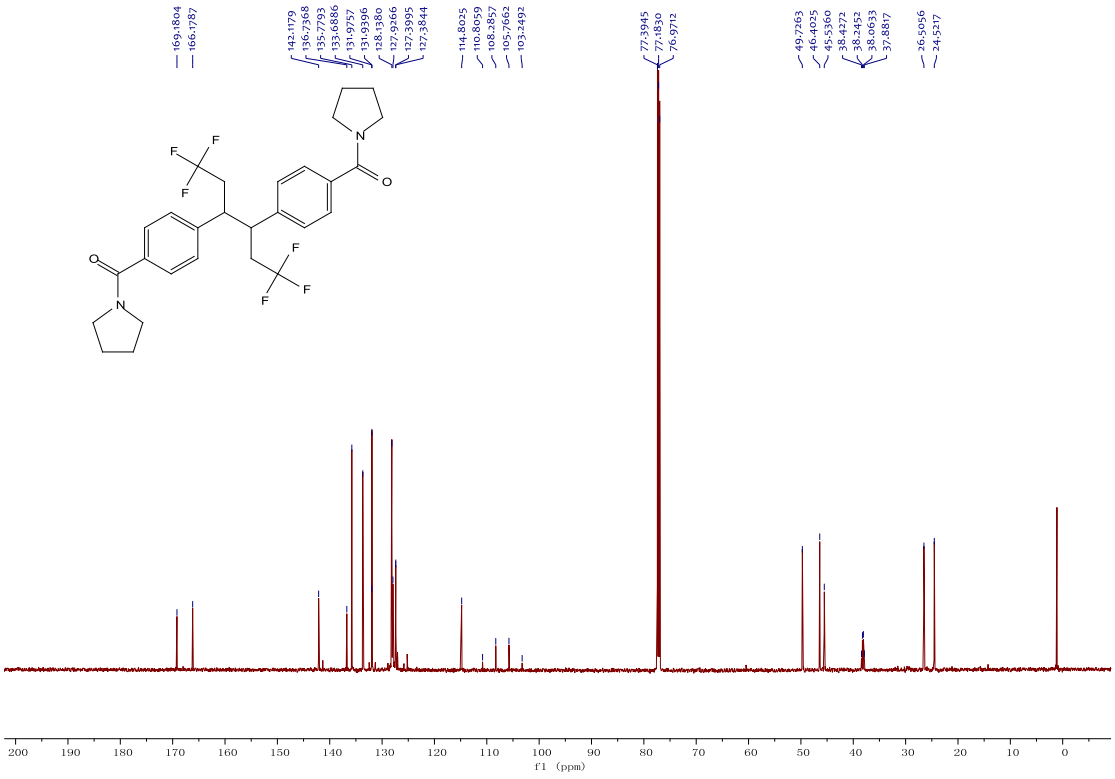
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **14**



^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **14**

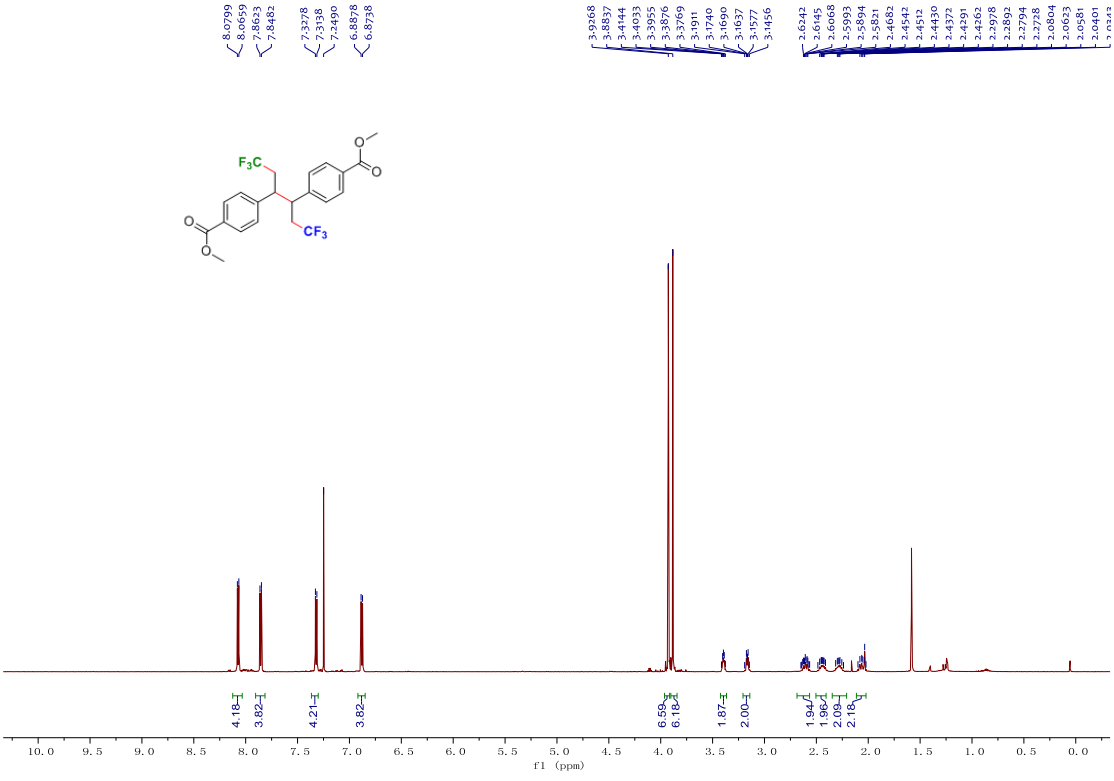


^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **16**

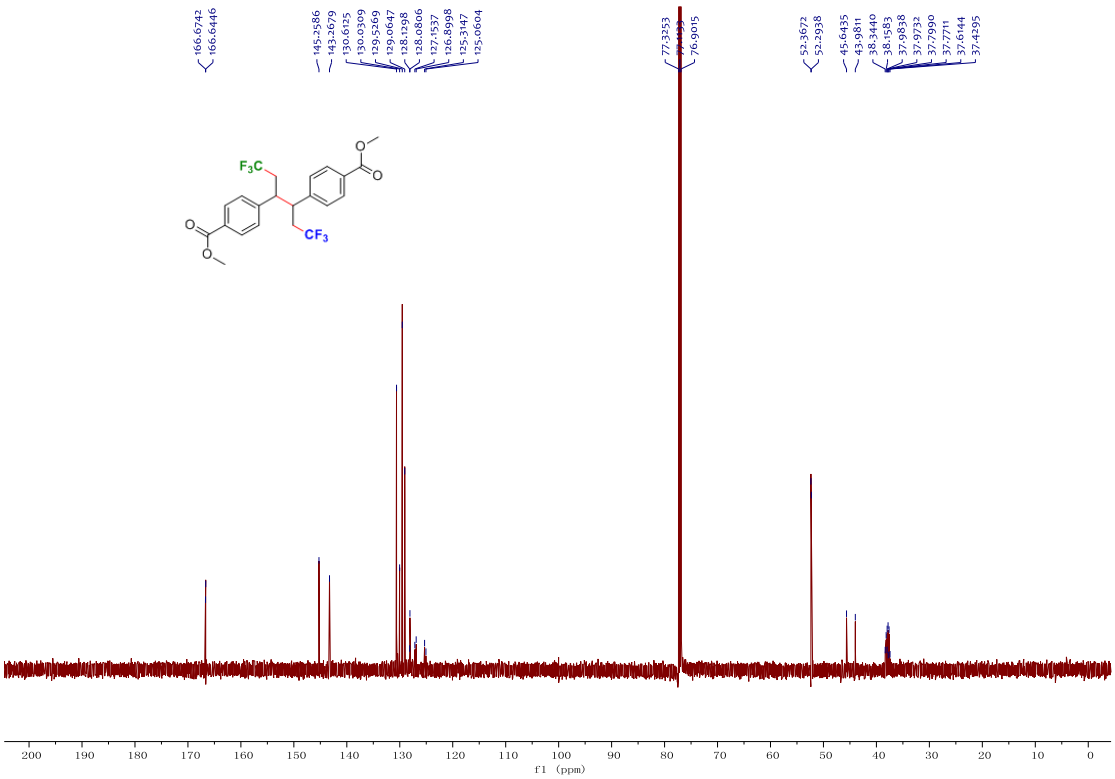


^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **16**

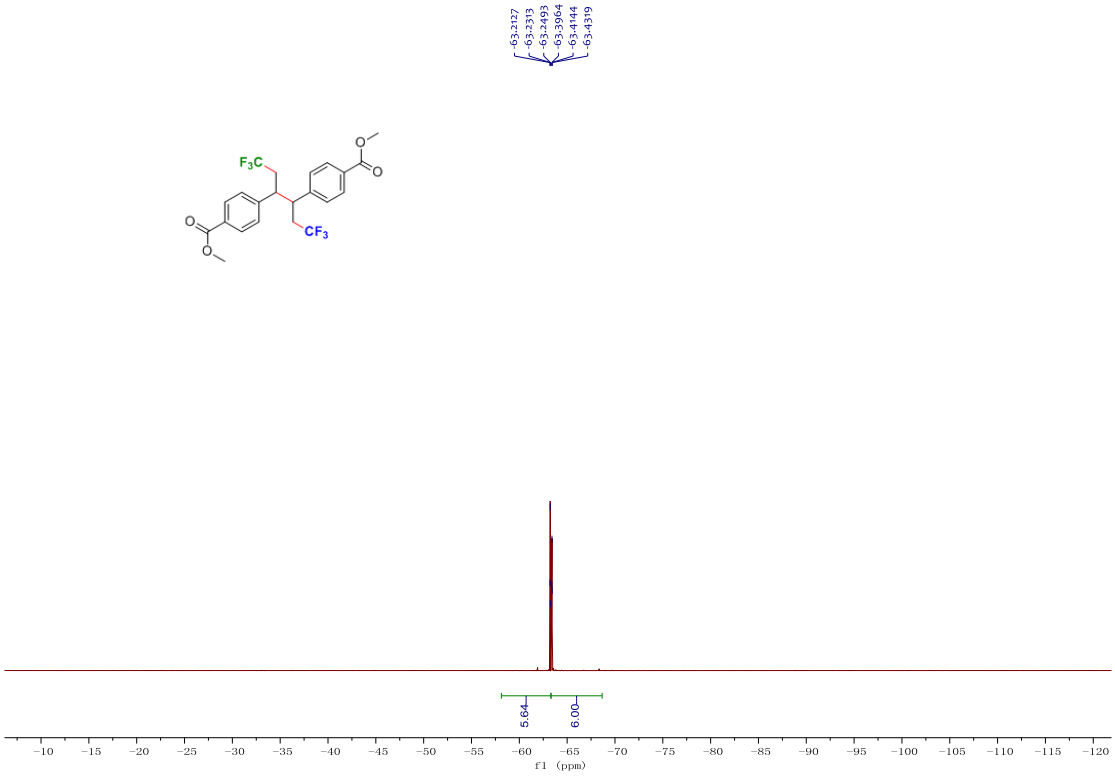
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **16**



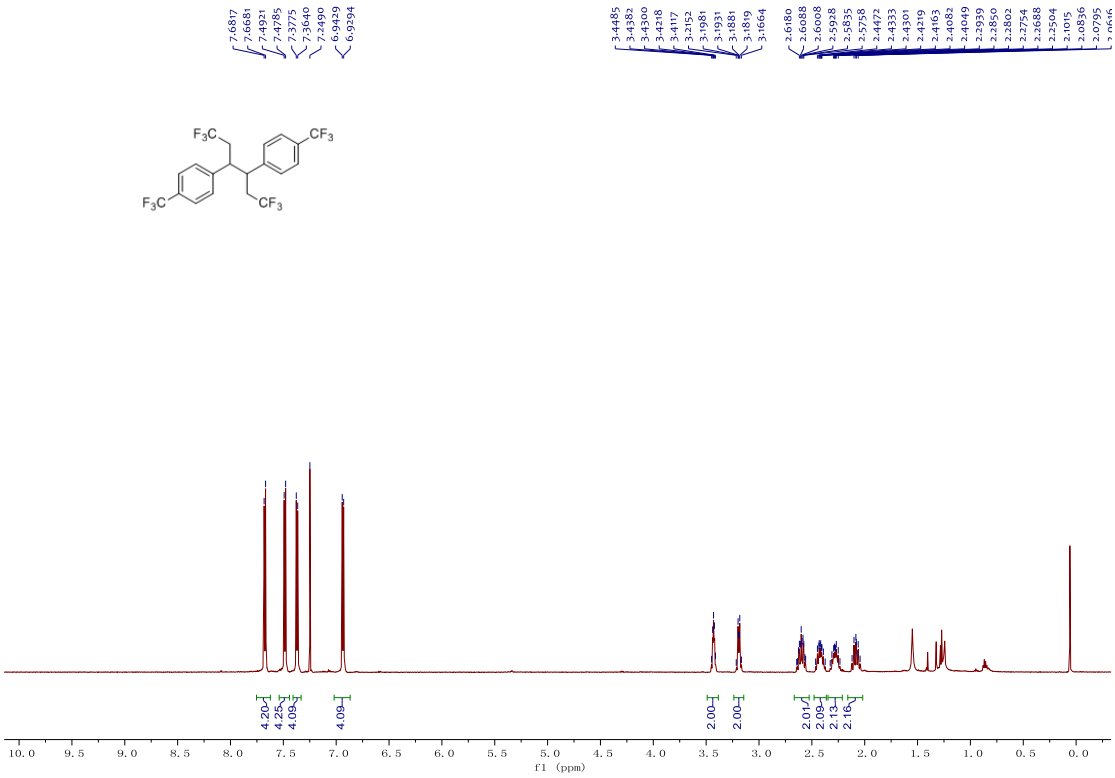
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **17**



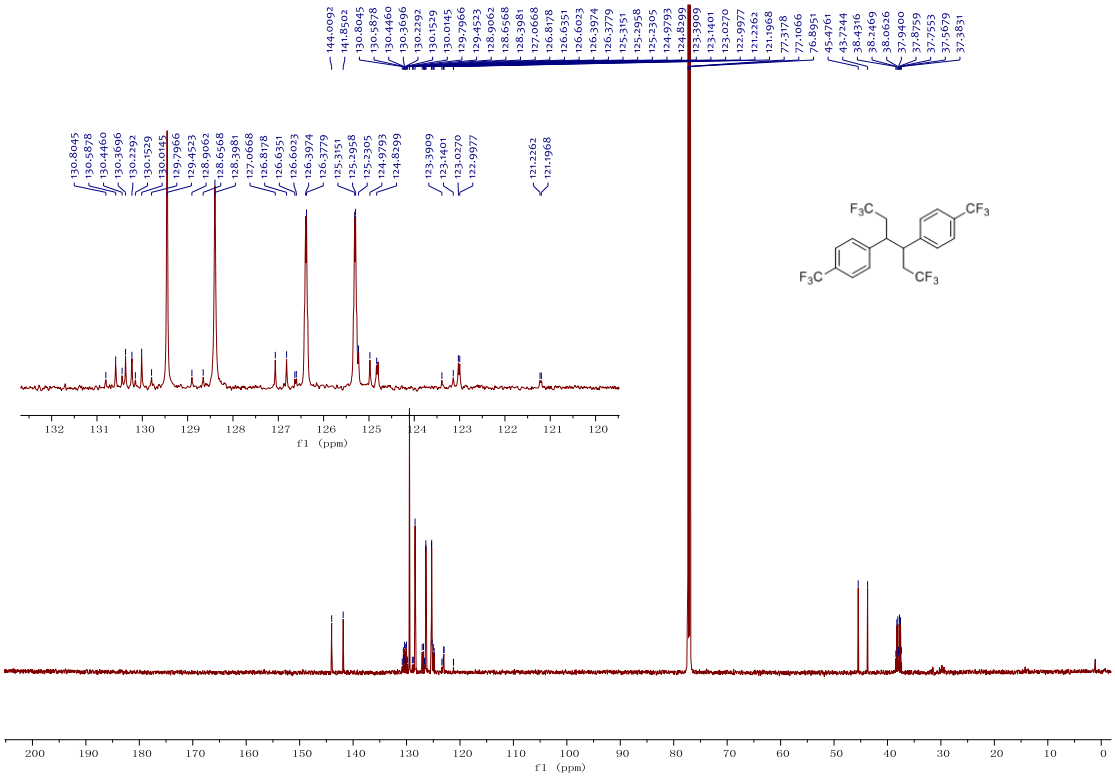
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **17**



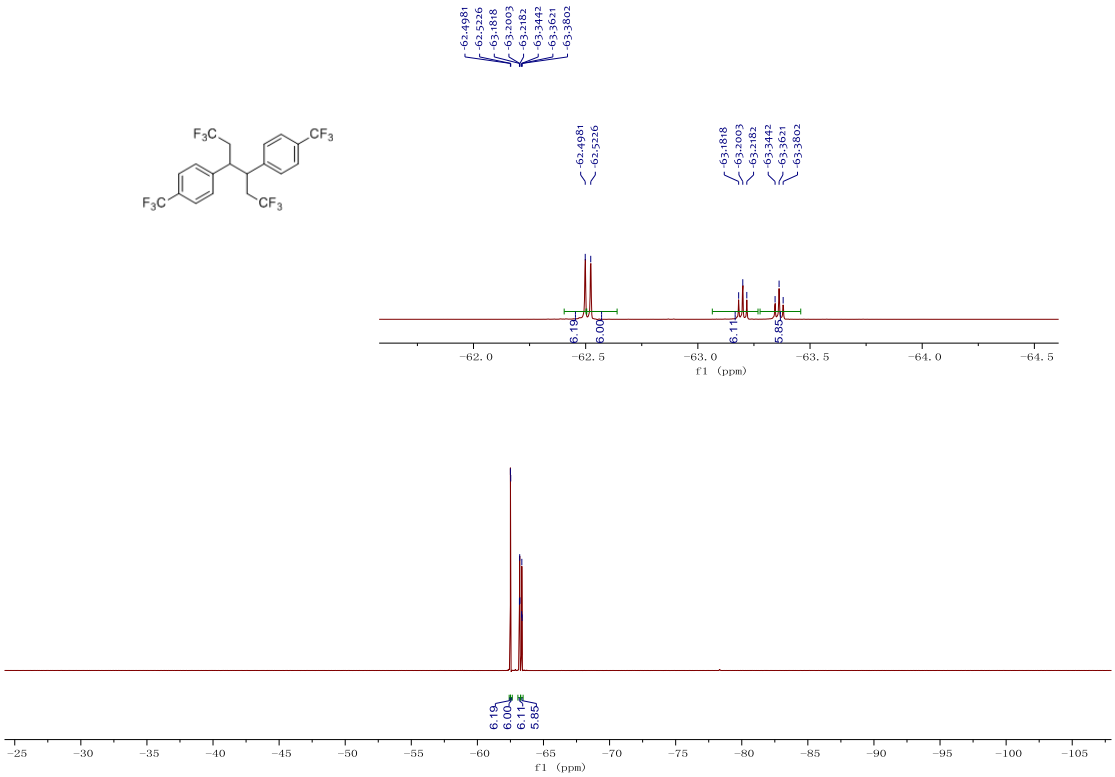
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **17**



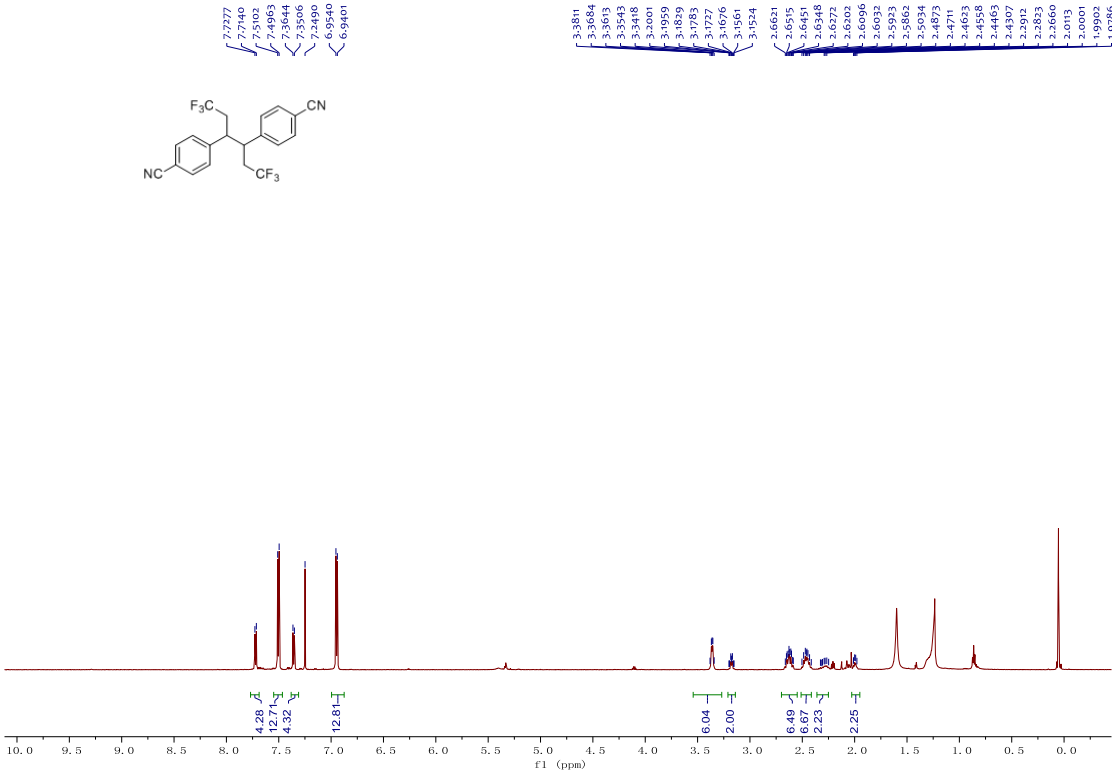
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **18**



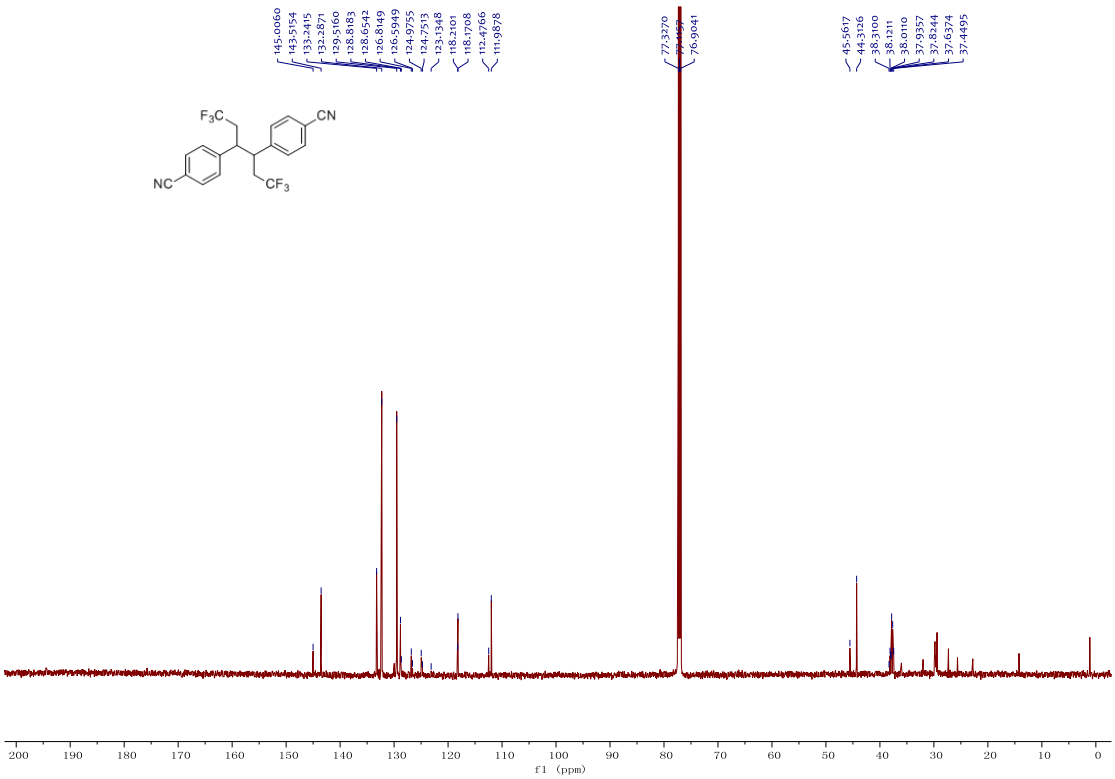
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **18**



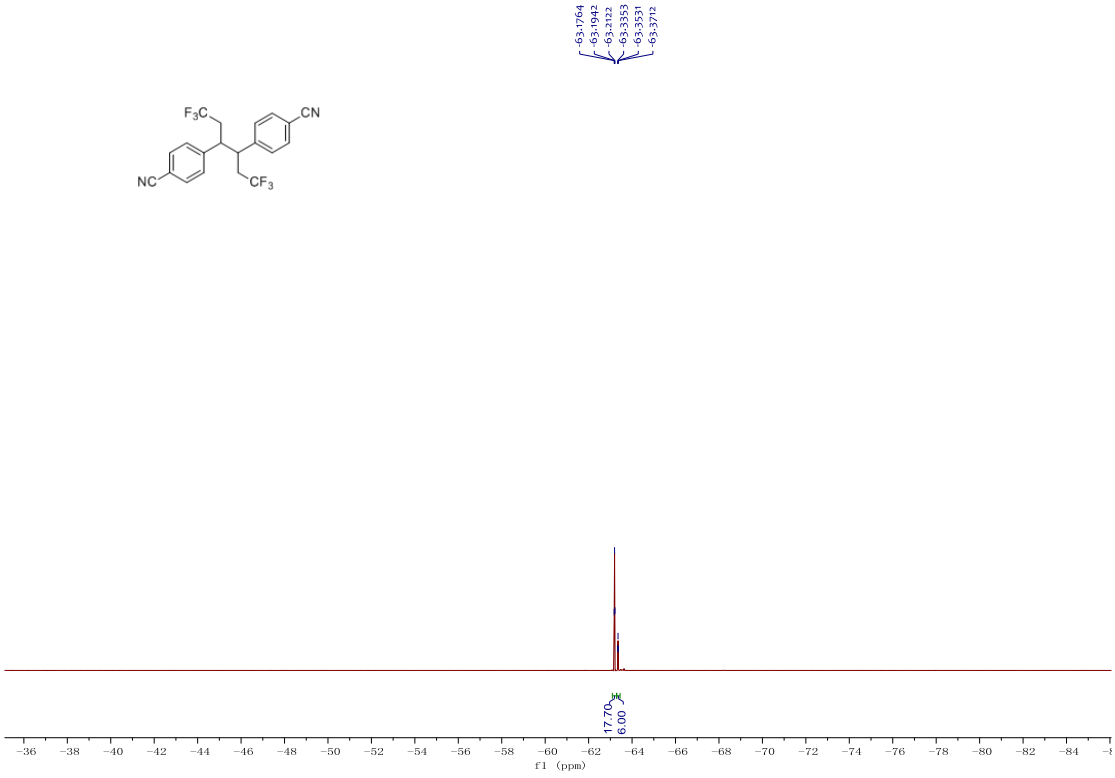
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **18**



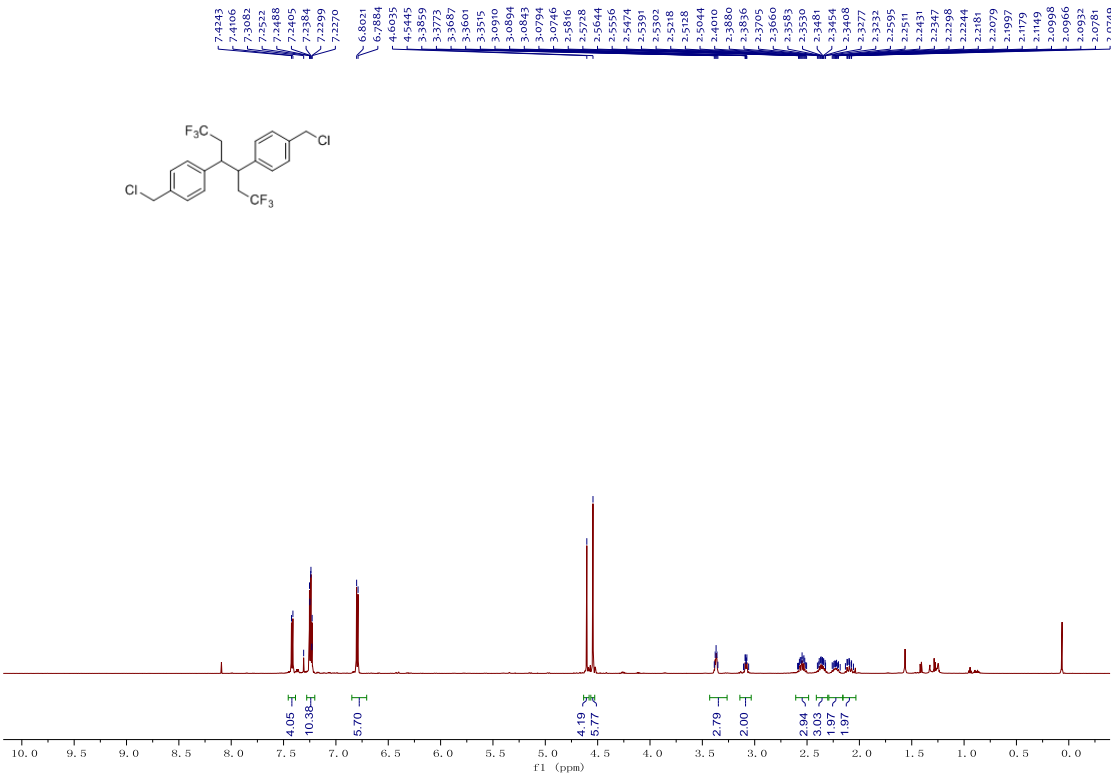
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **19**



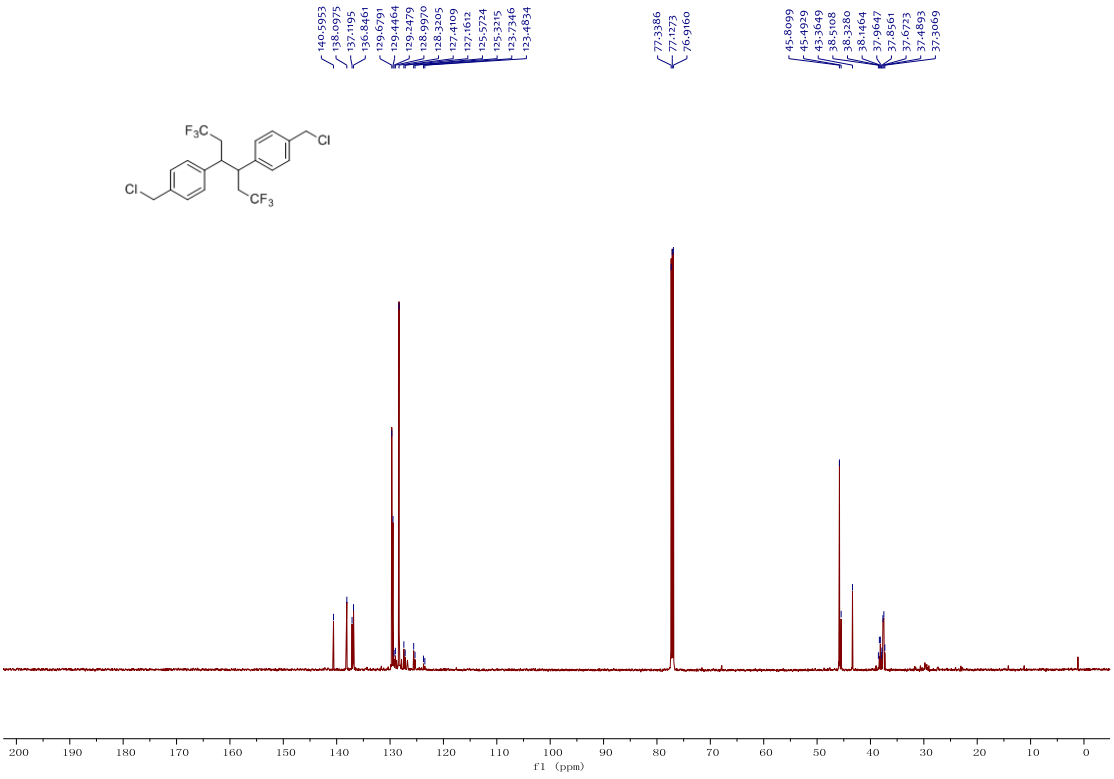
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **19**



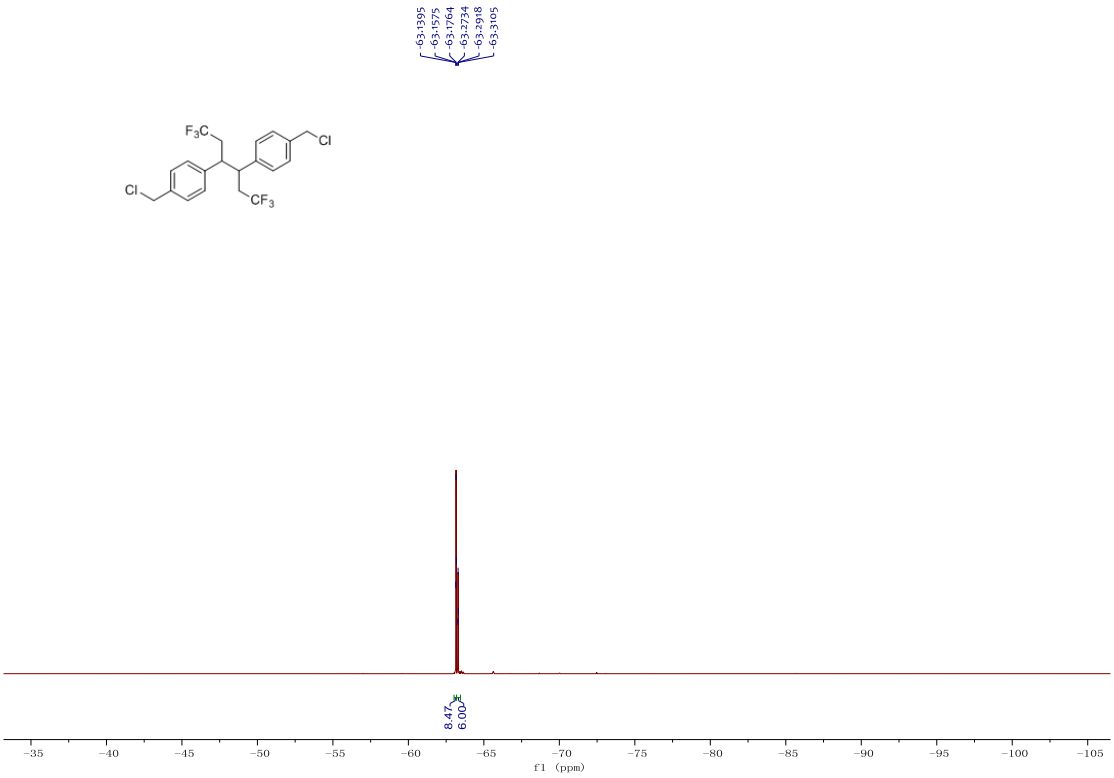
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **19**



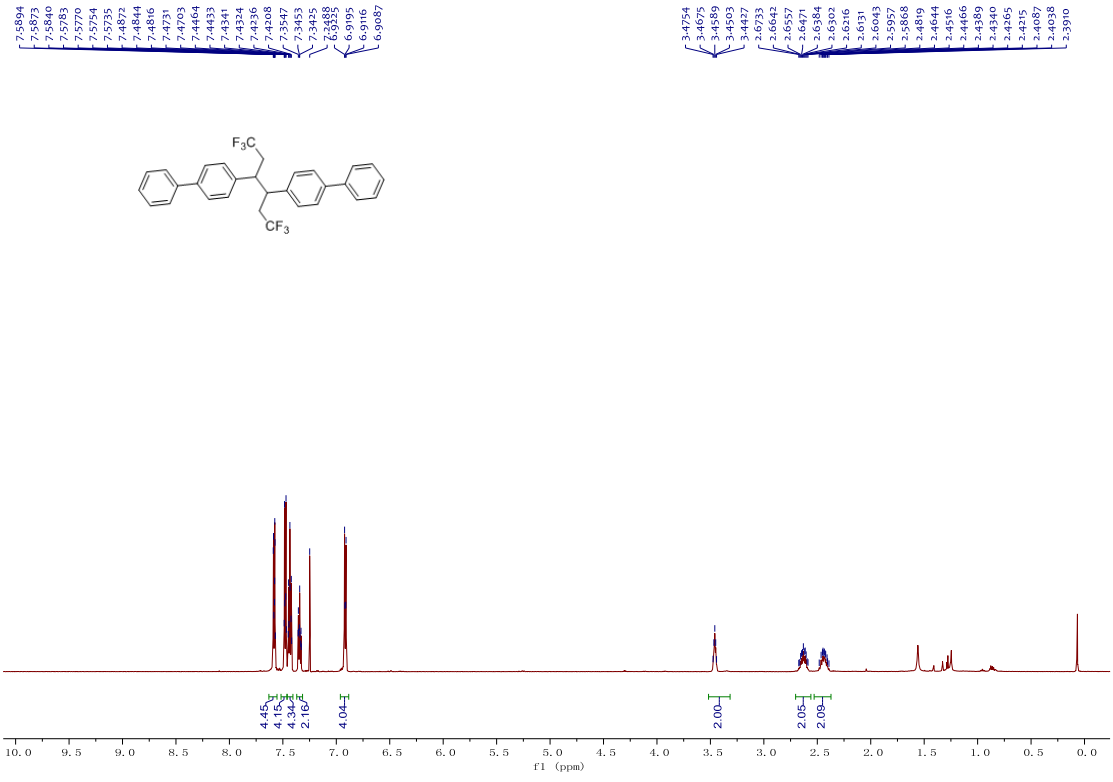
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **20**



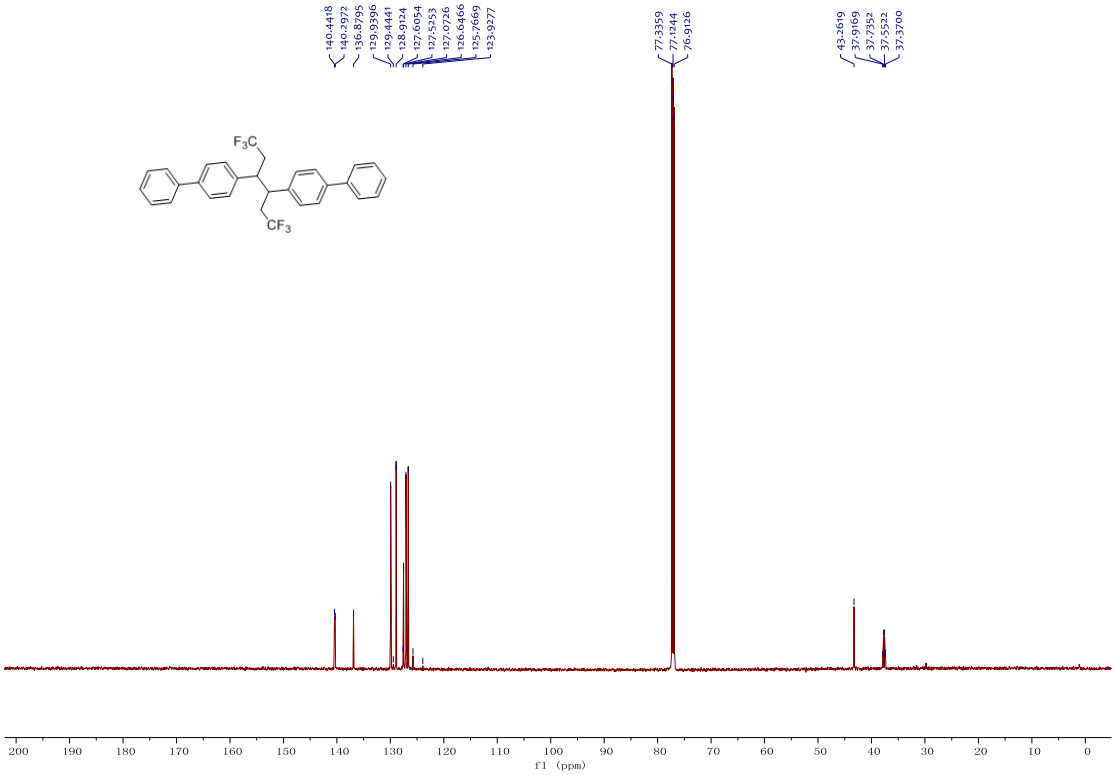
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **20**



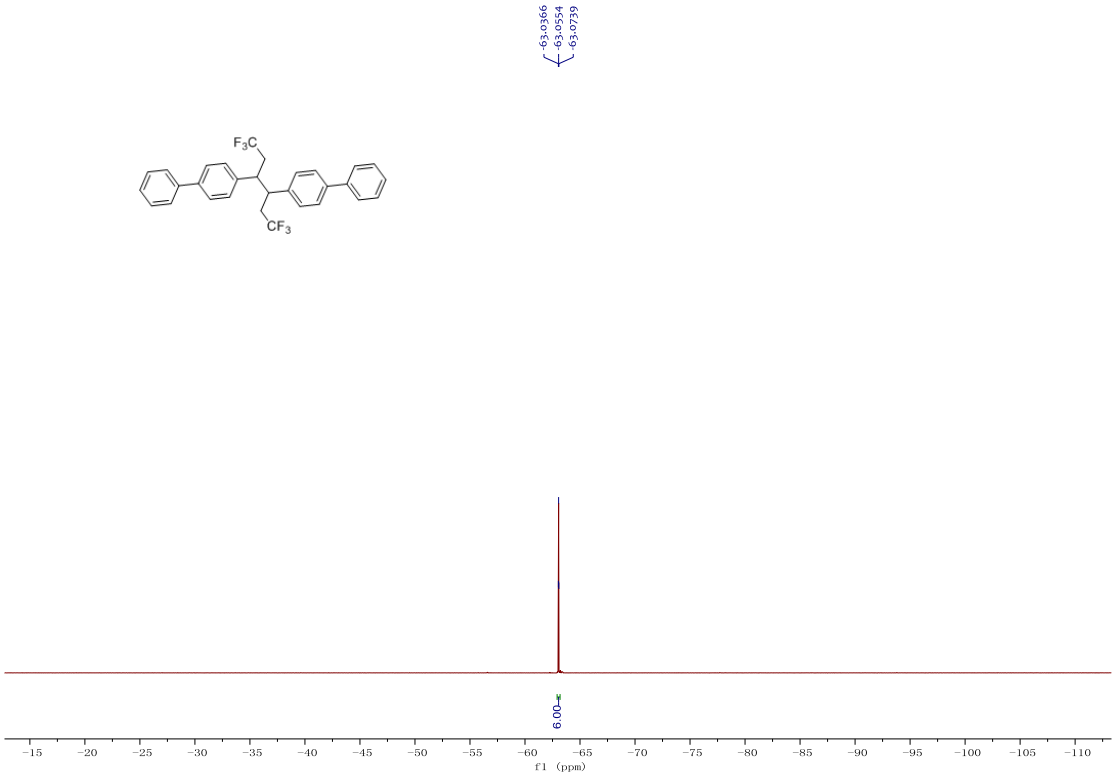
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **20**



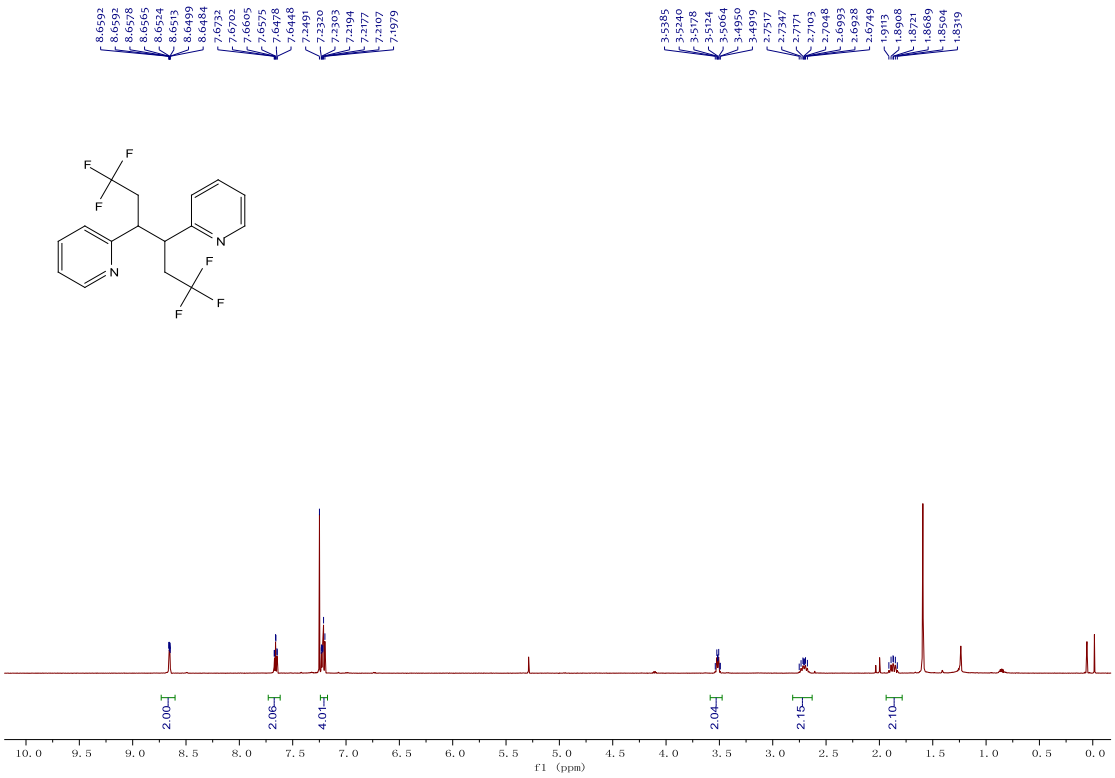
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **21**



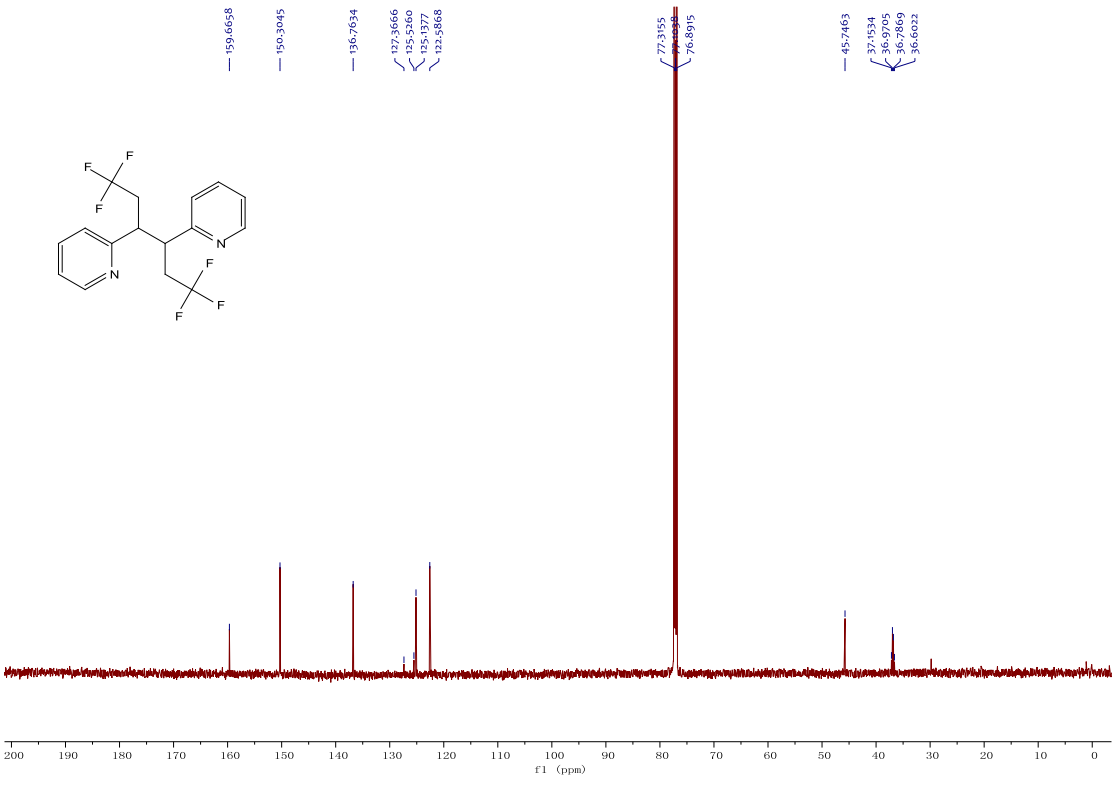
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **21**



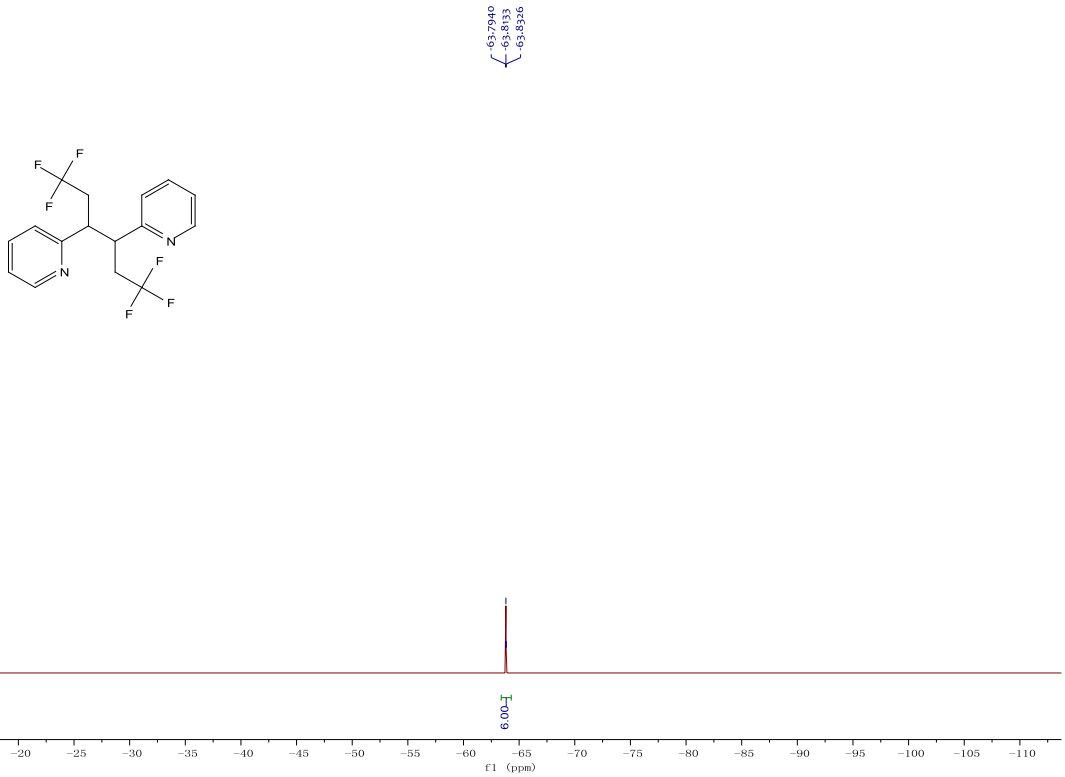
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **21**



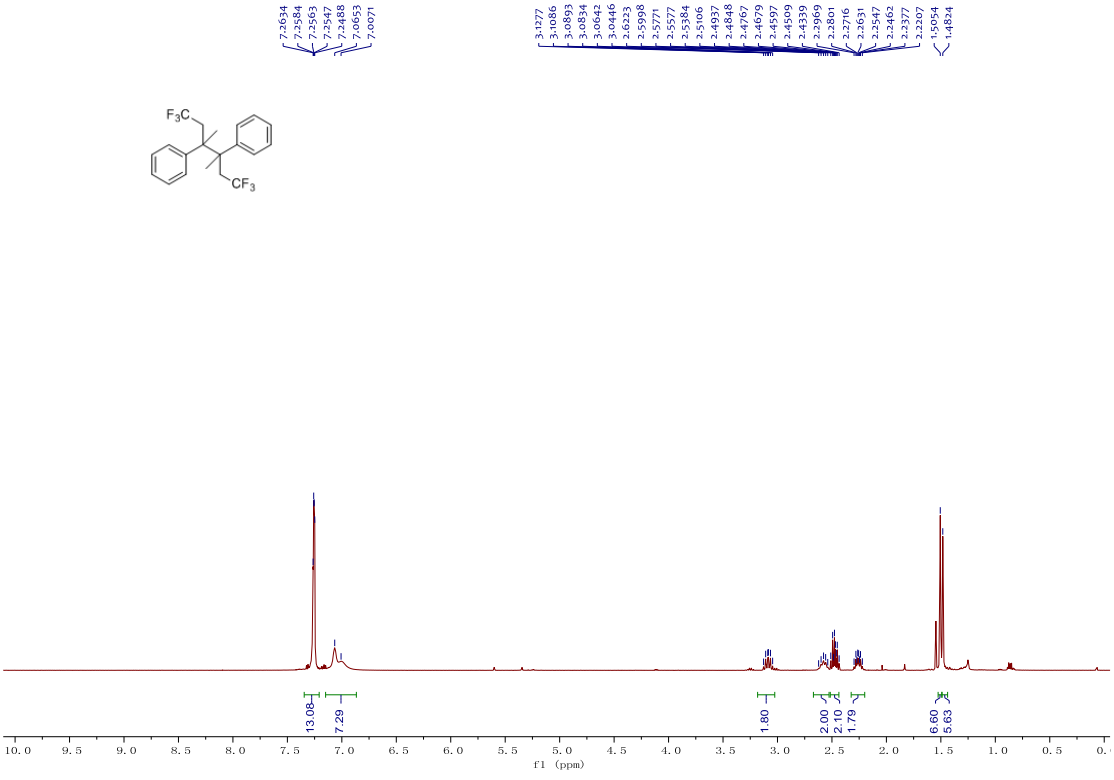
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **22** (syn)



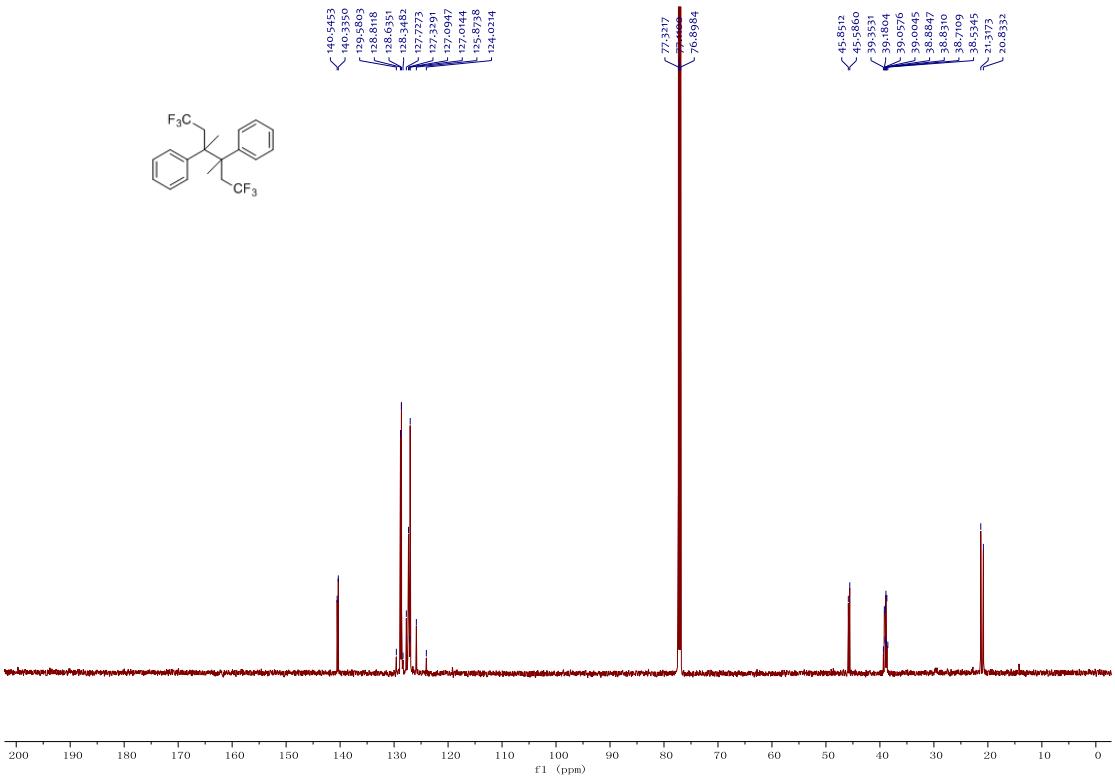
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **22** (syn)



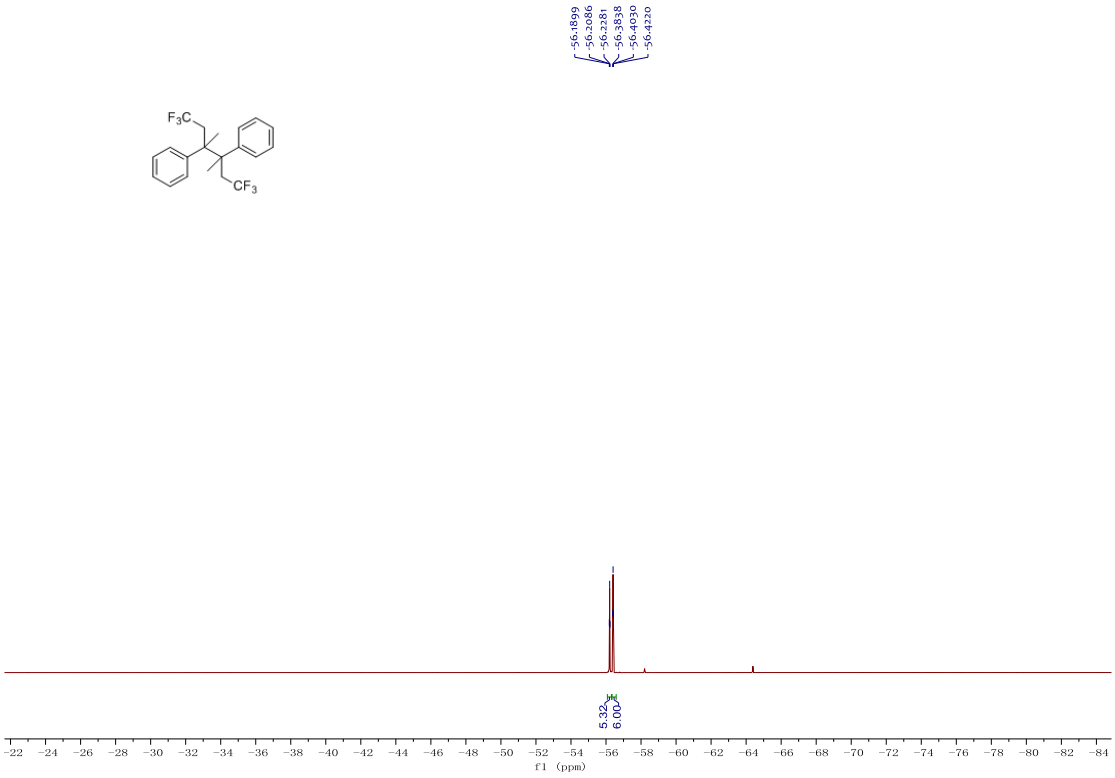
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **22** (syn)



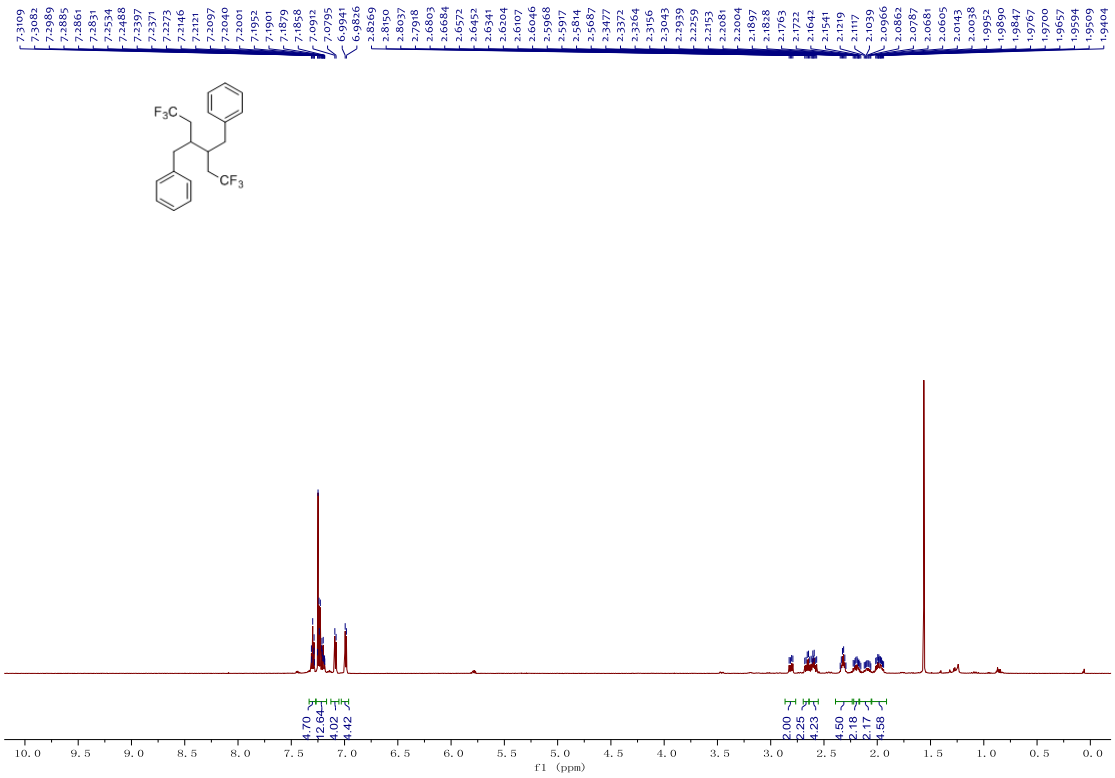
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **23**



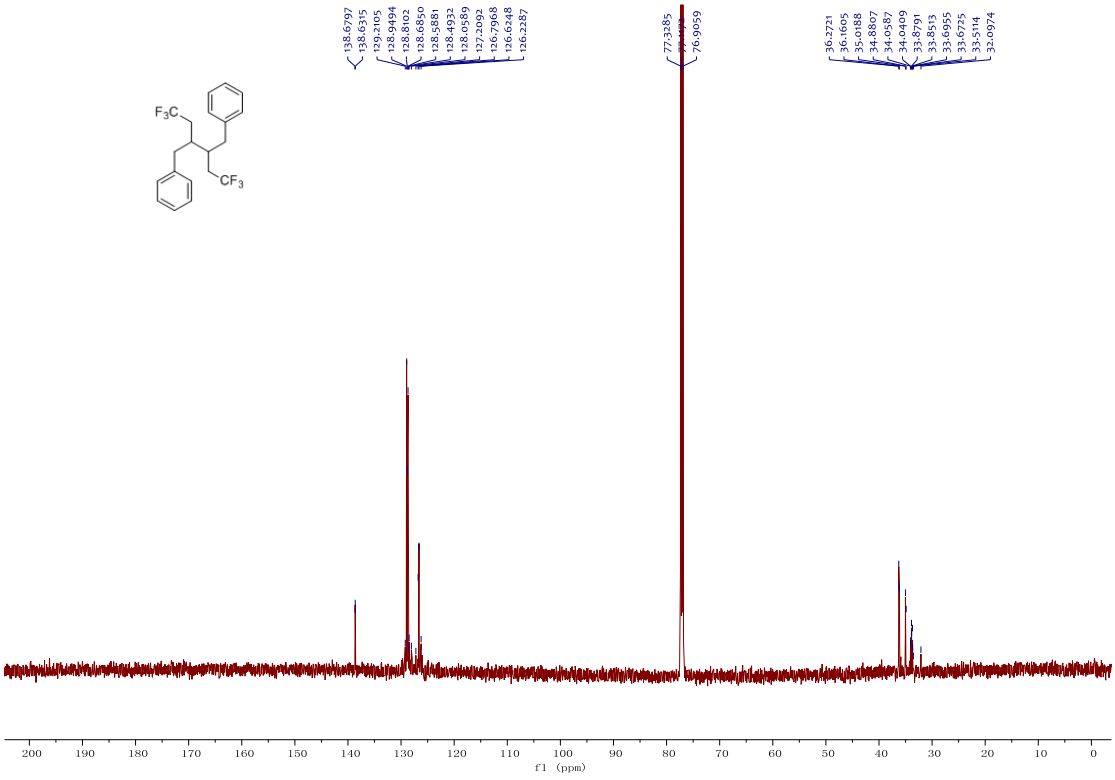
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **23**



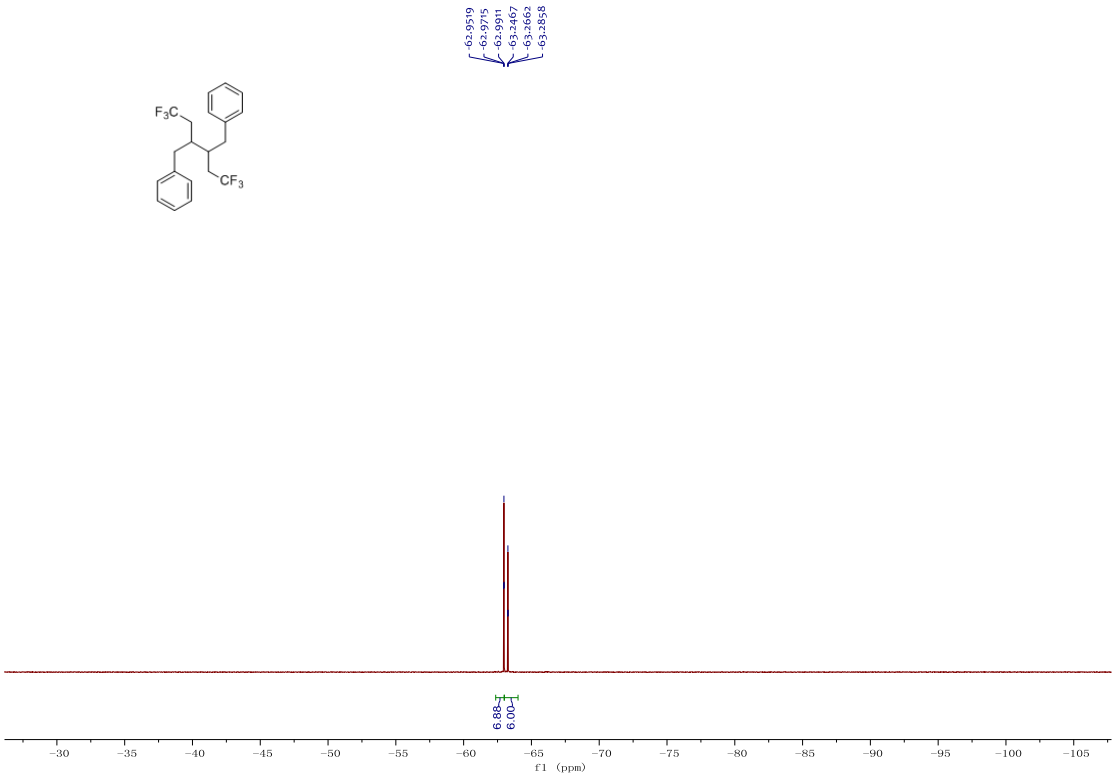
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **23**



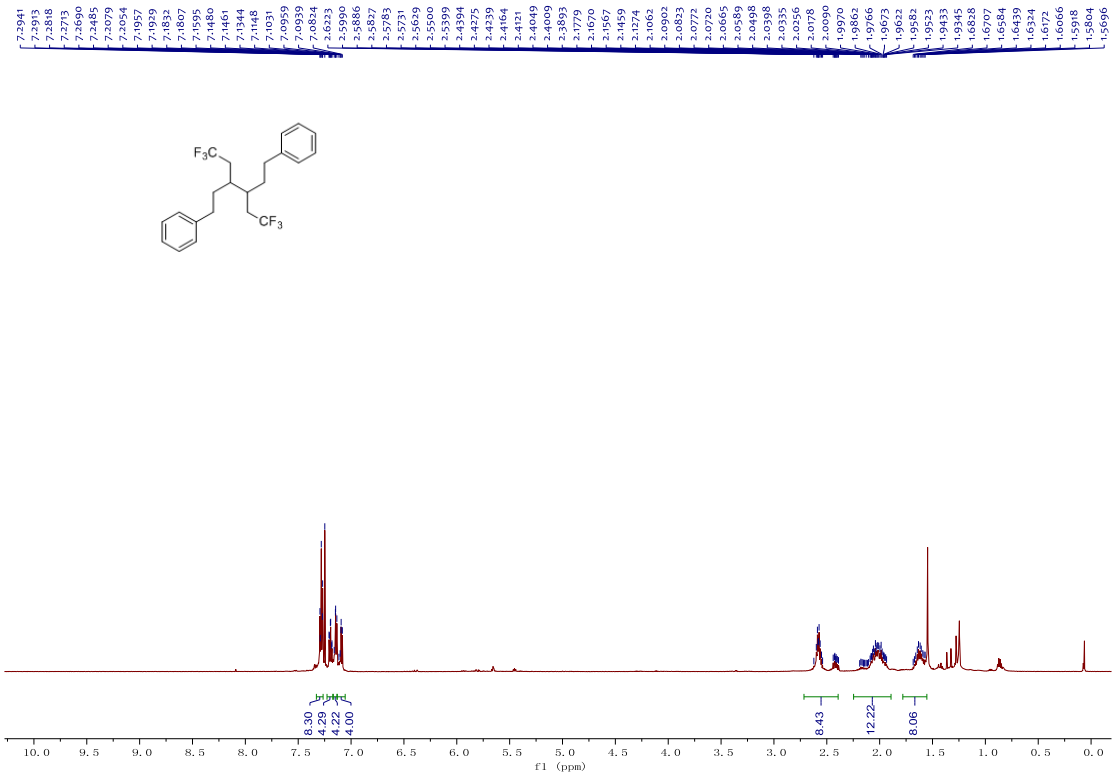
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **24**



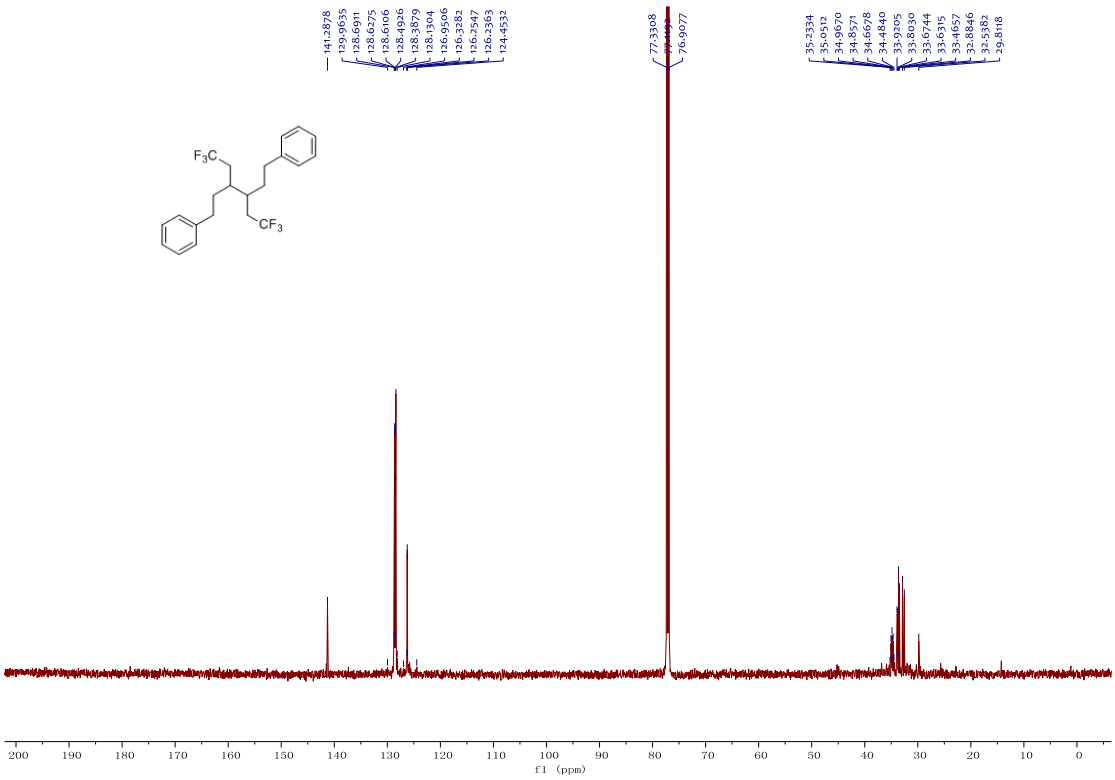
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **24**



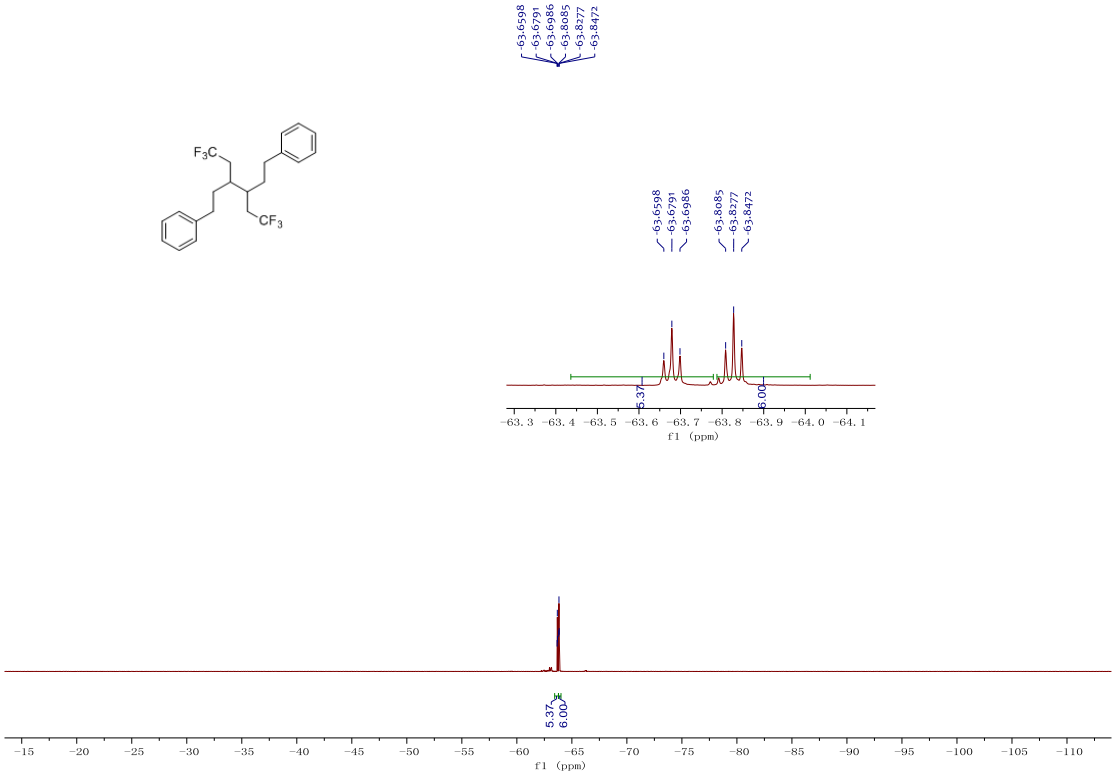
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **24**



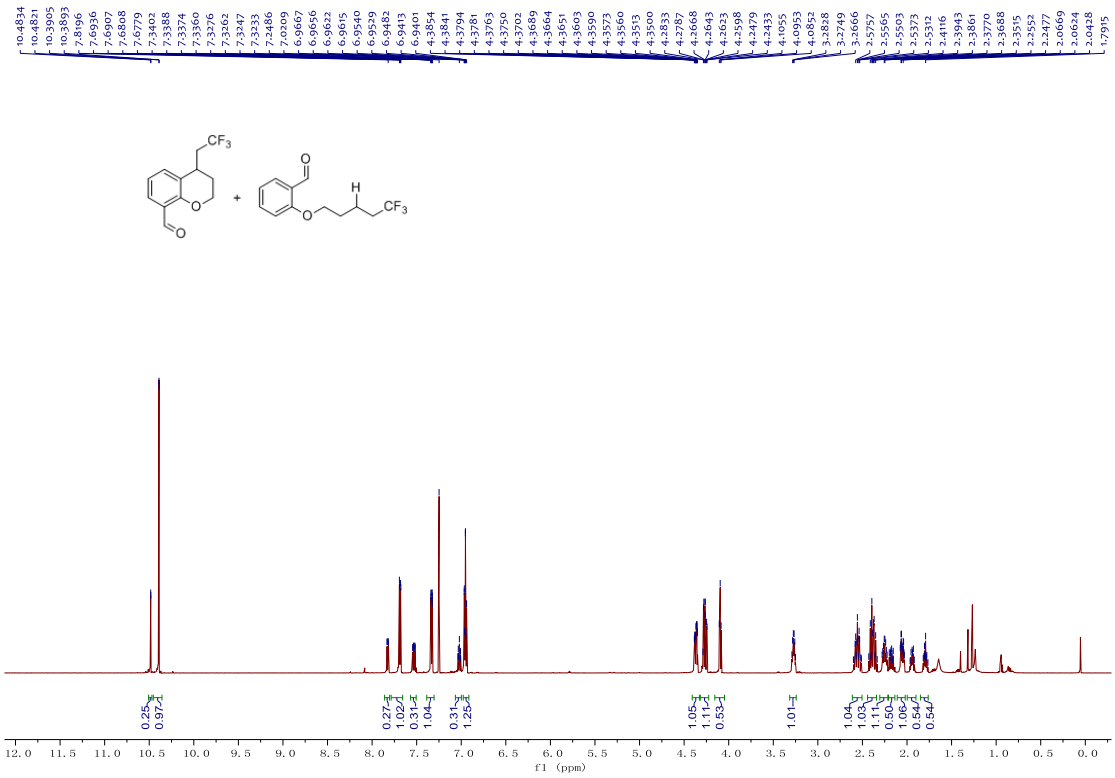
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **25**



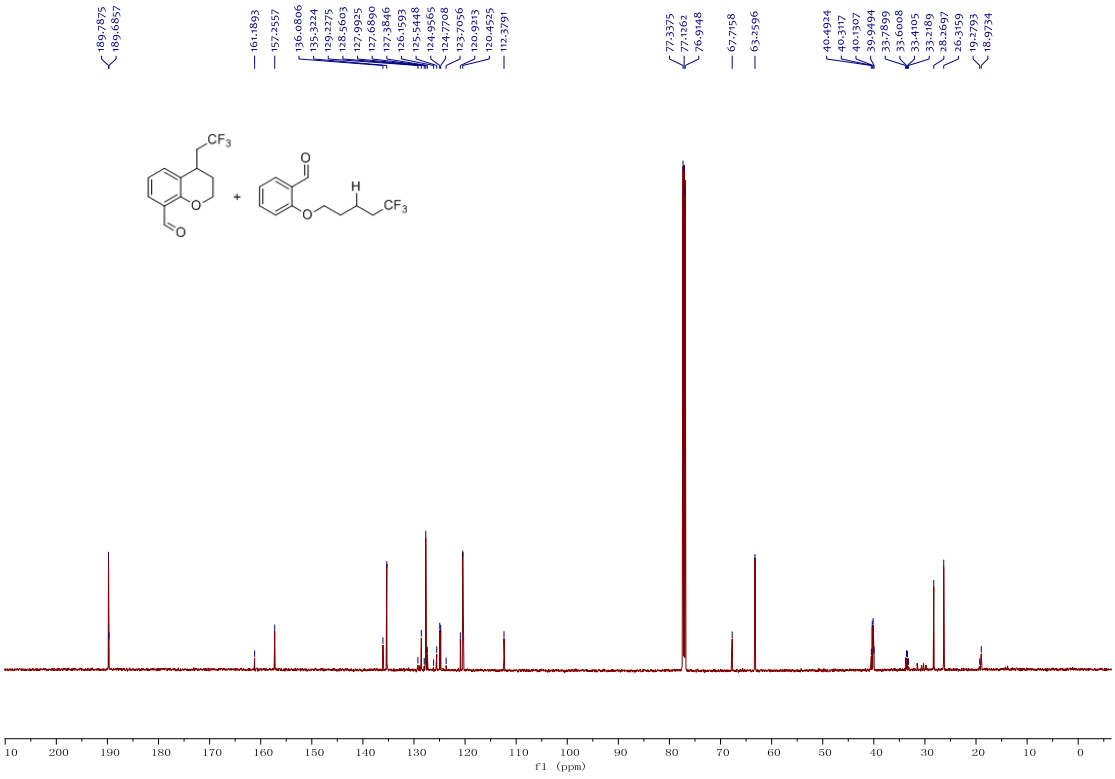
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **25**



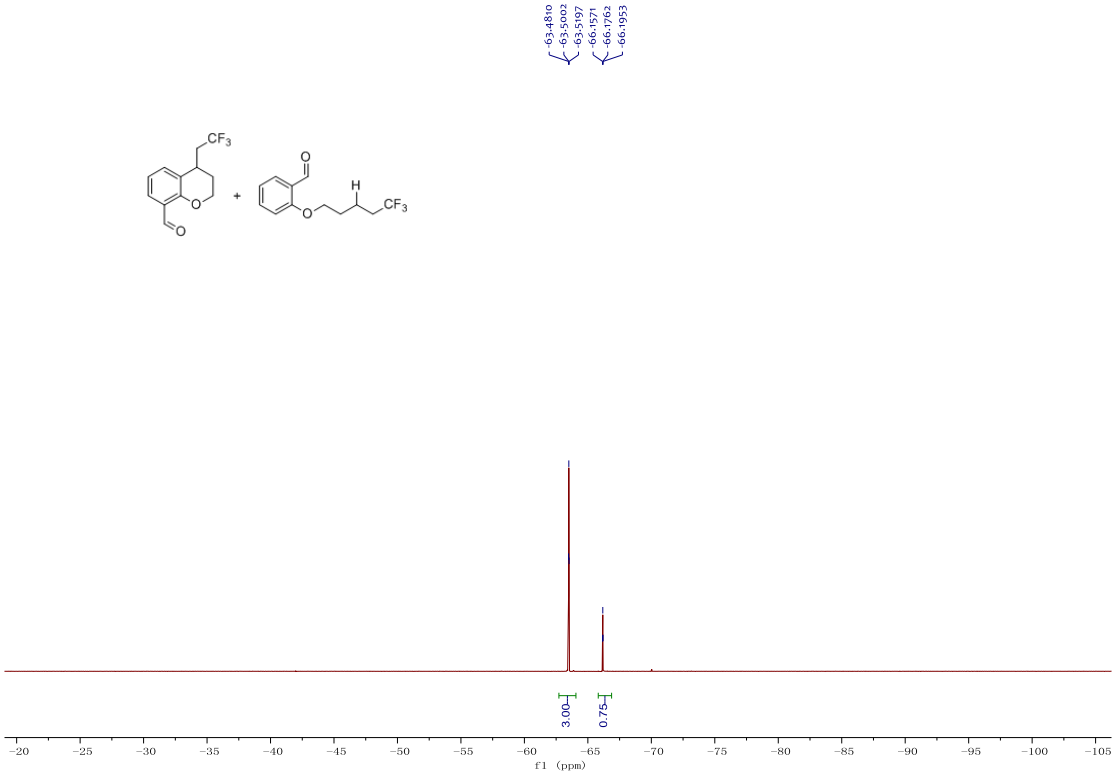
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **25**



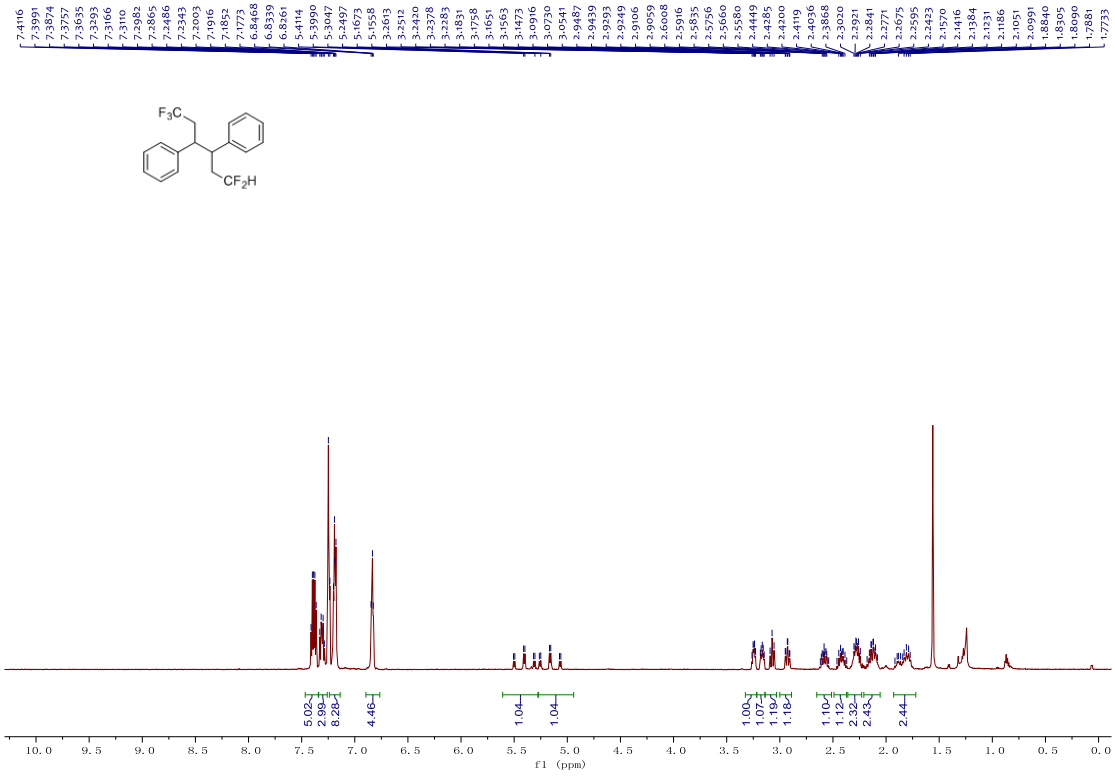
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **26**+**27**



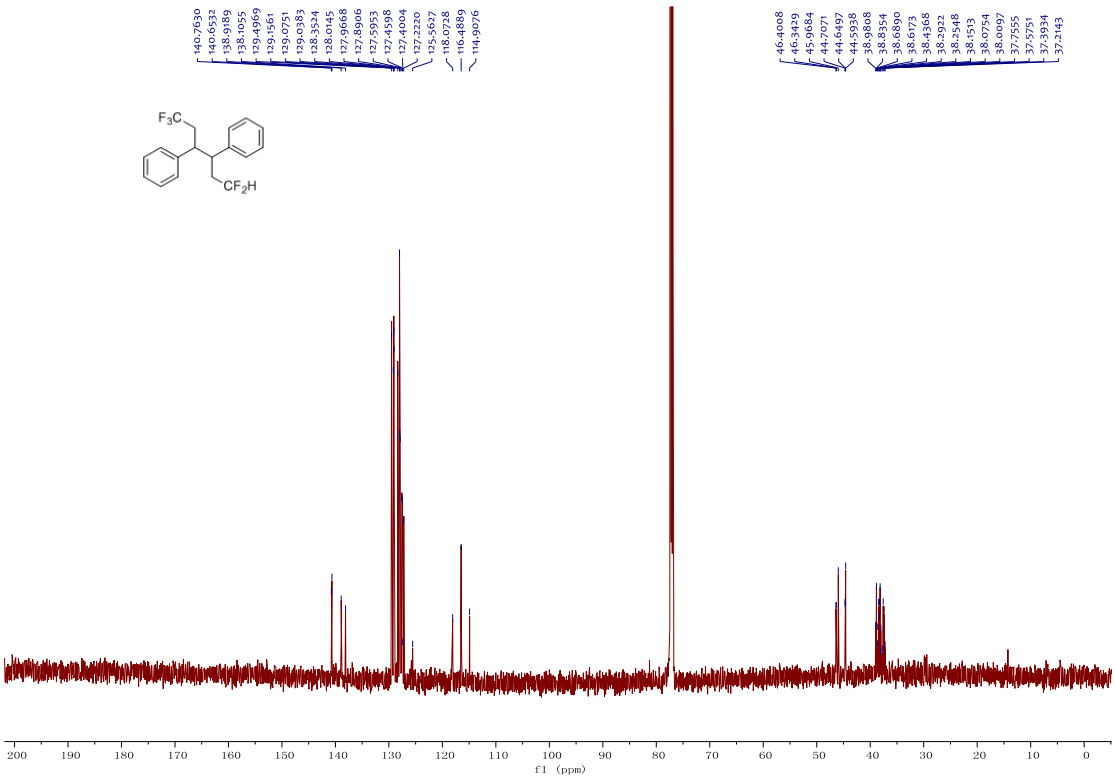
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **26**+**27**



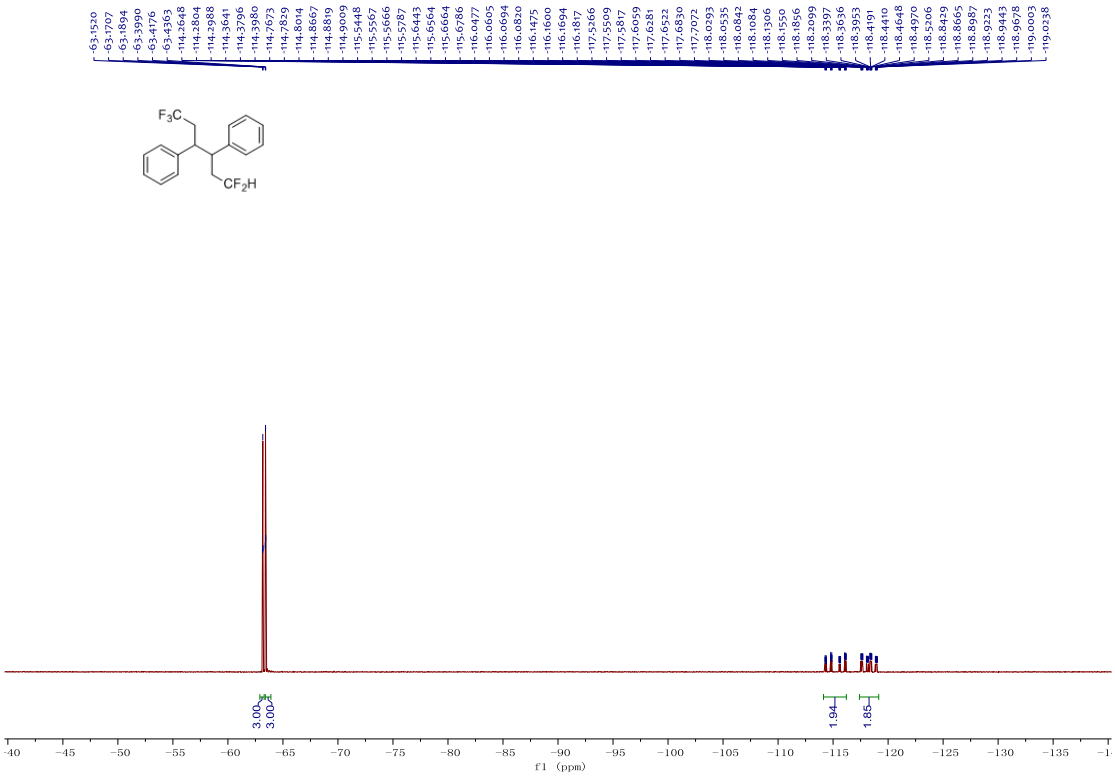
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **26**+**27**



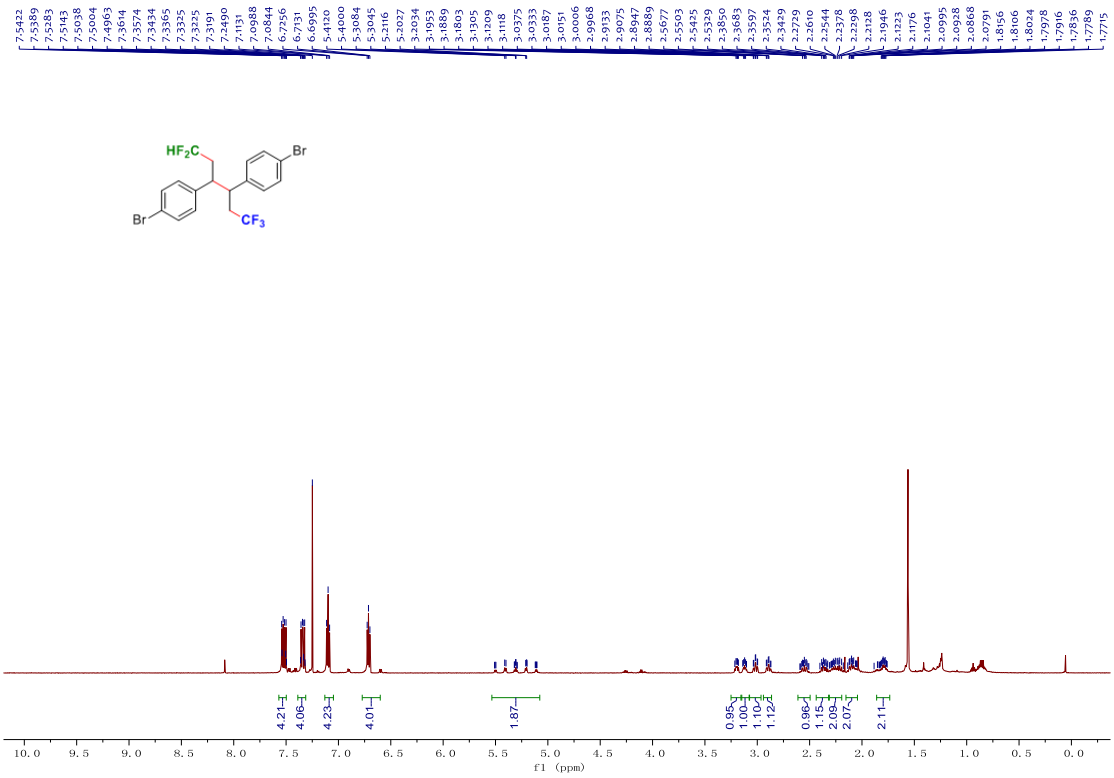
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **30**



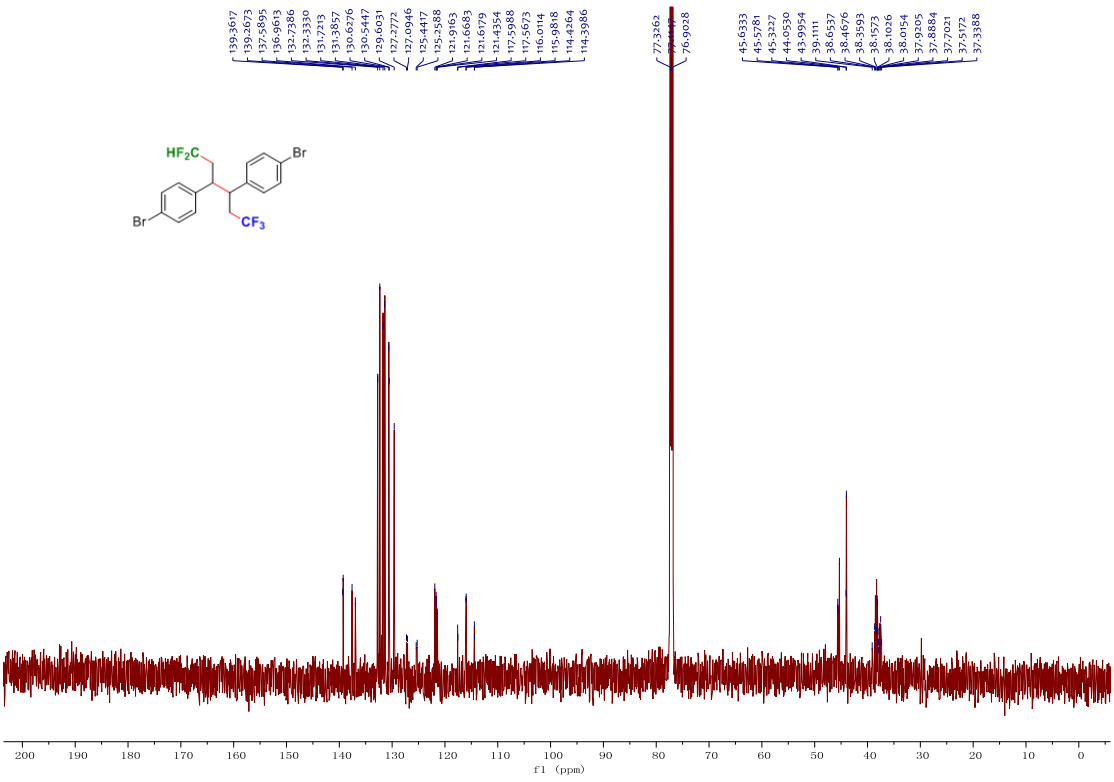
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **30**



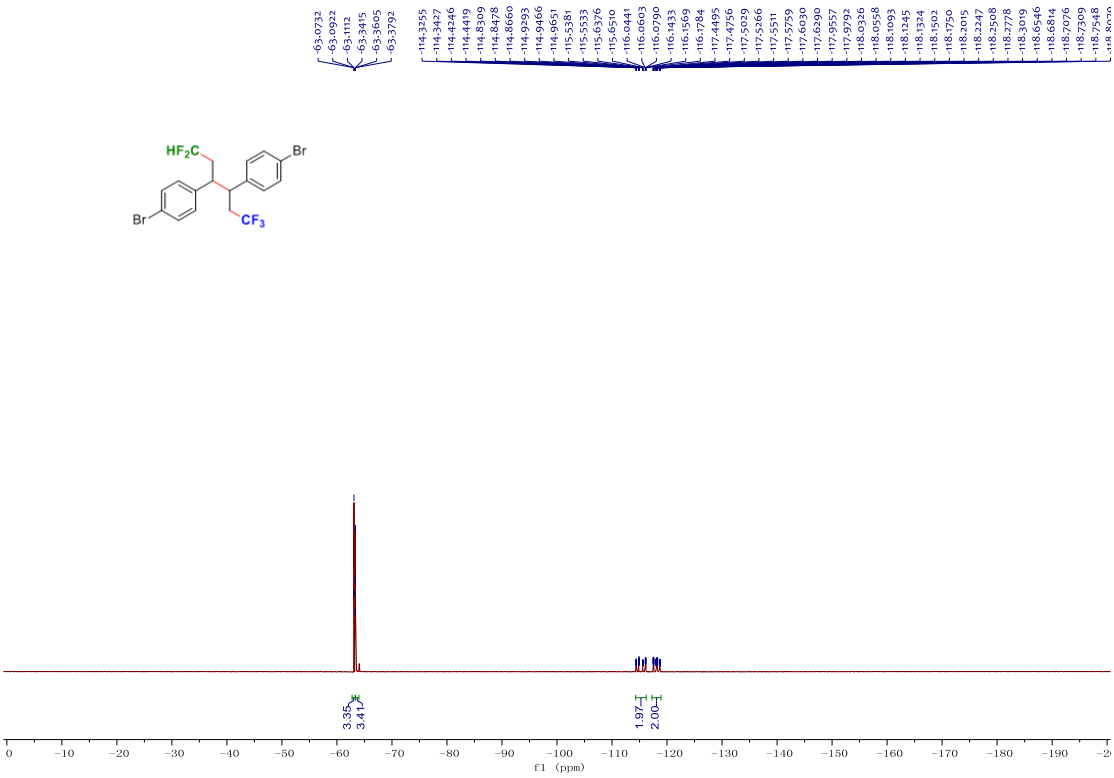
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **30**



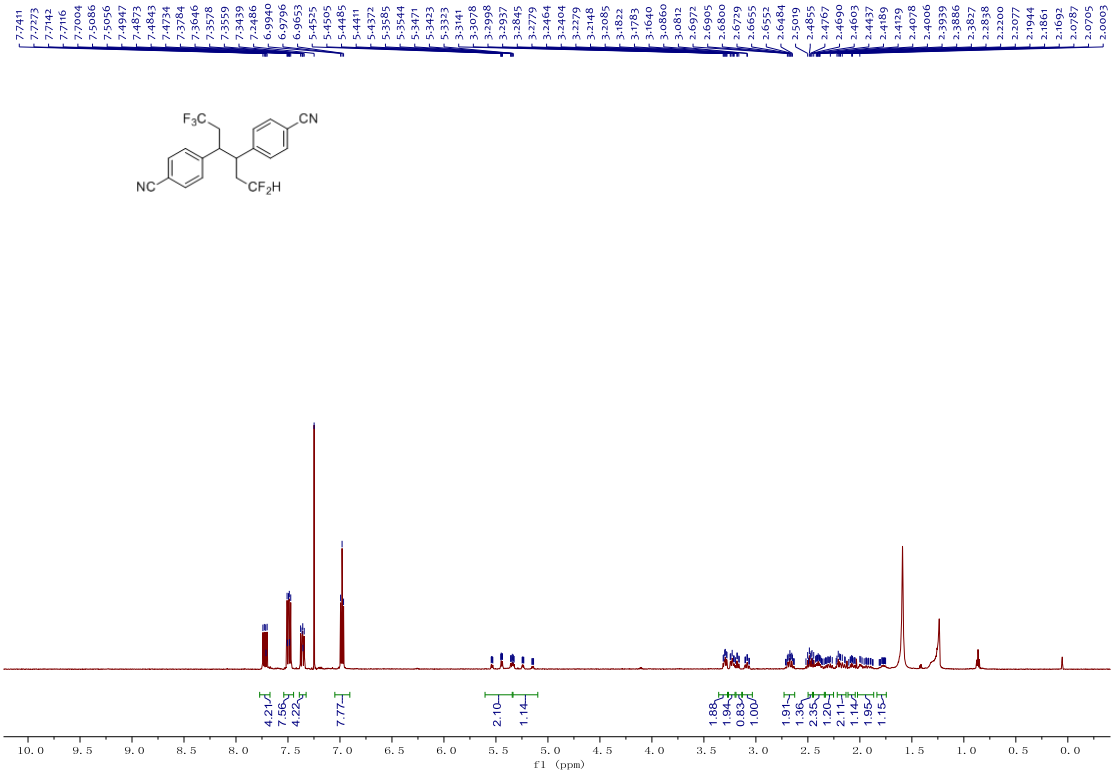
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **31**



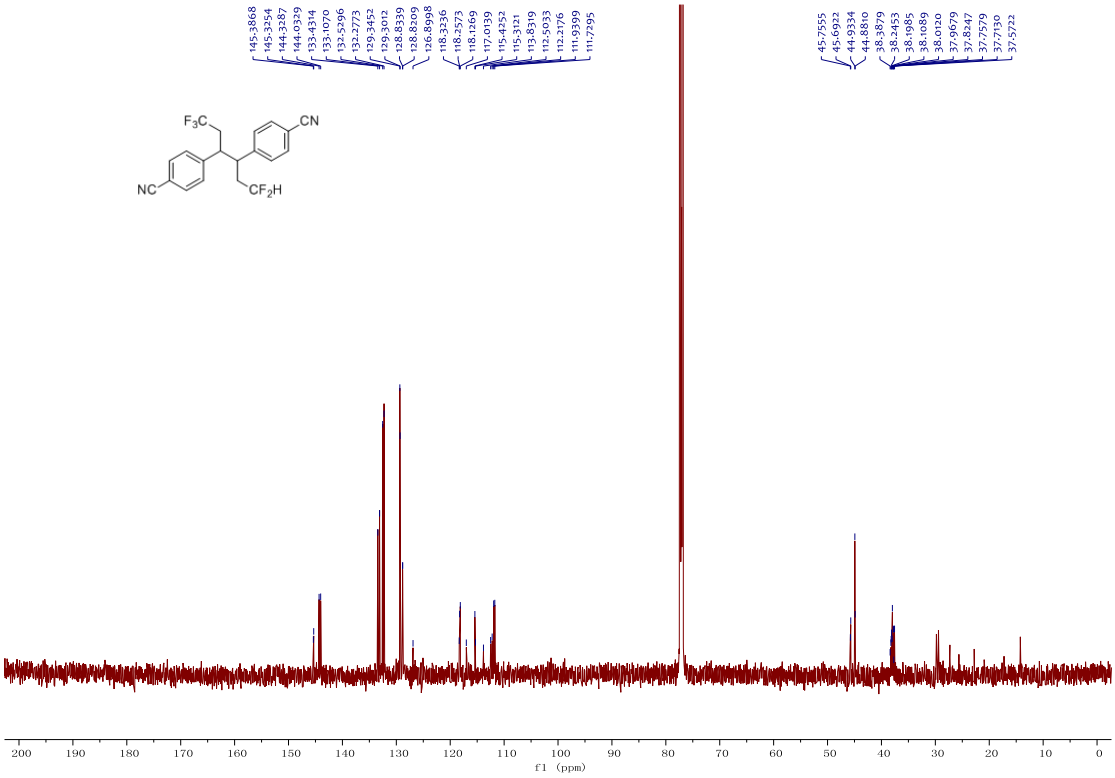
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **31**



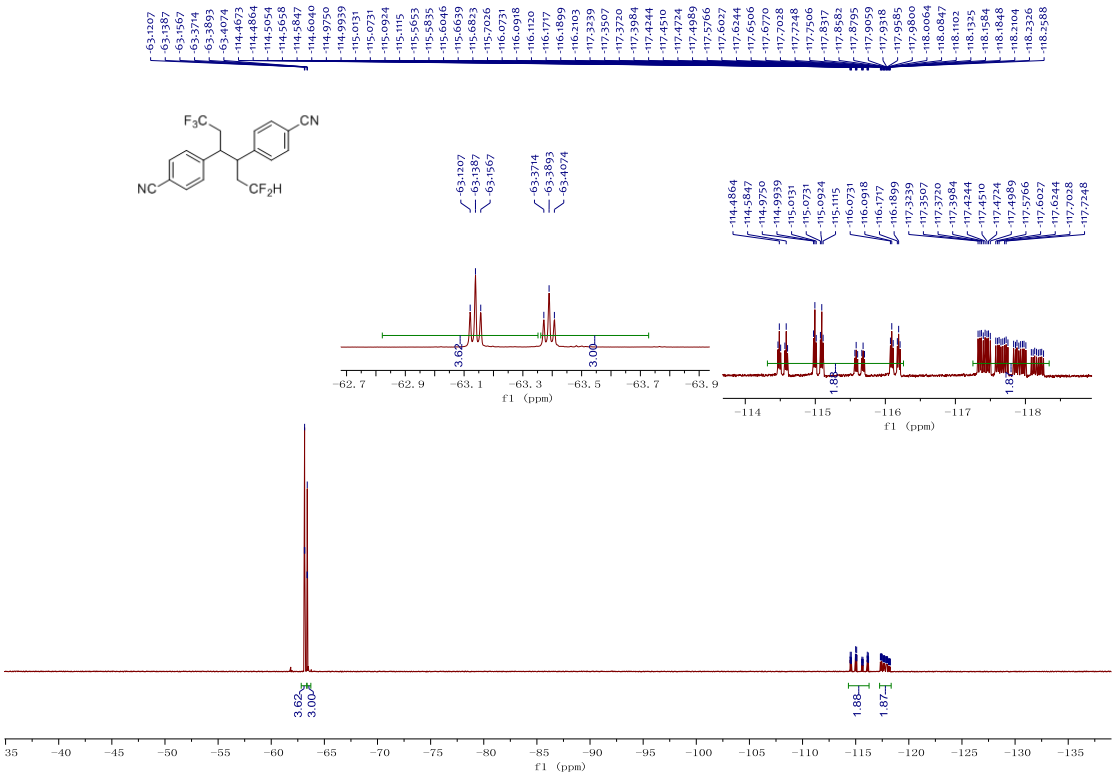
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **31**



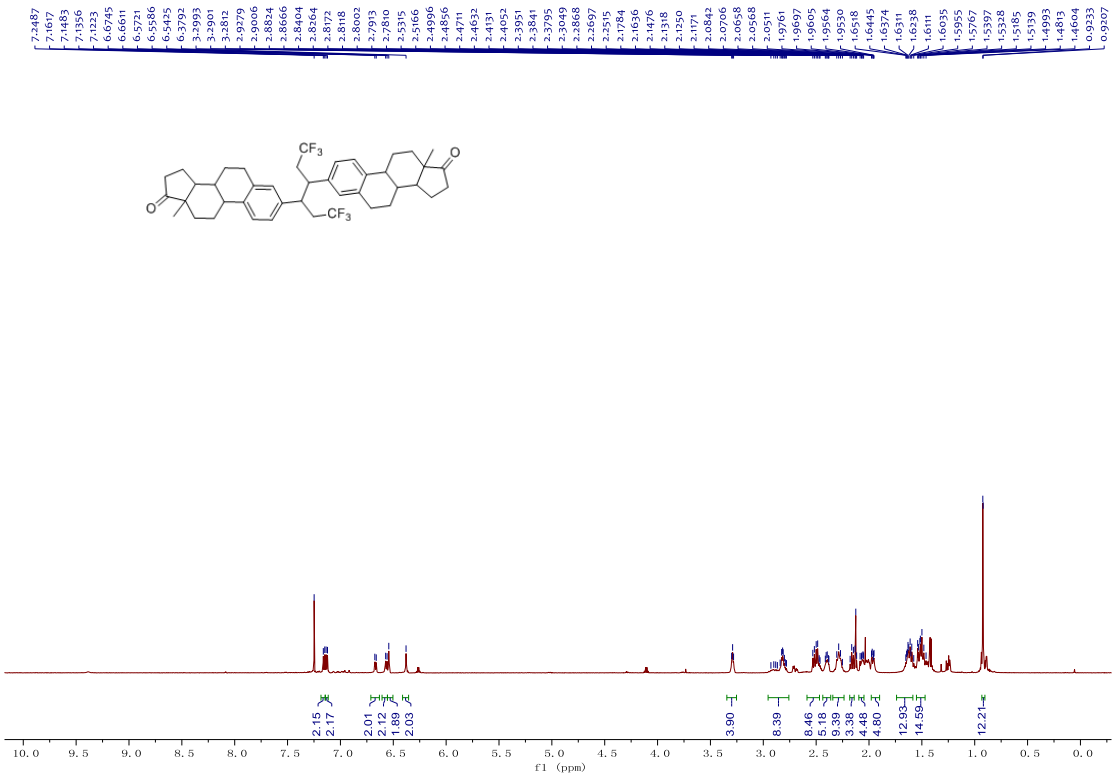
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **32**



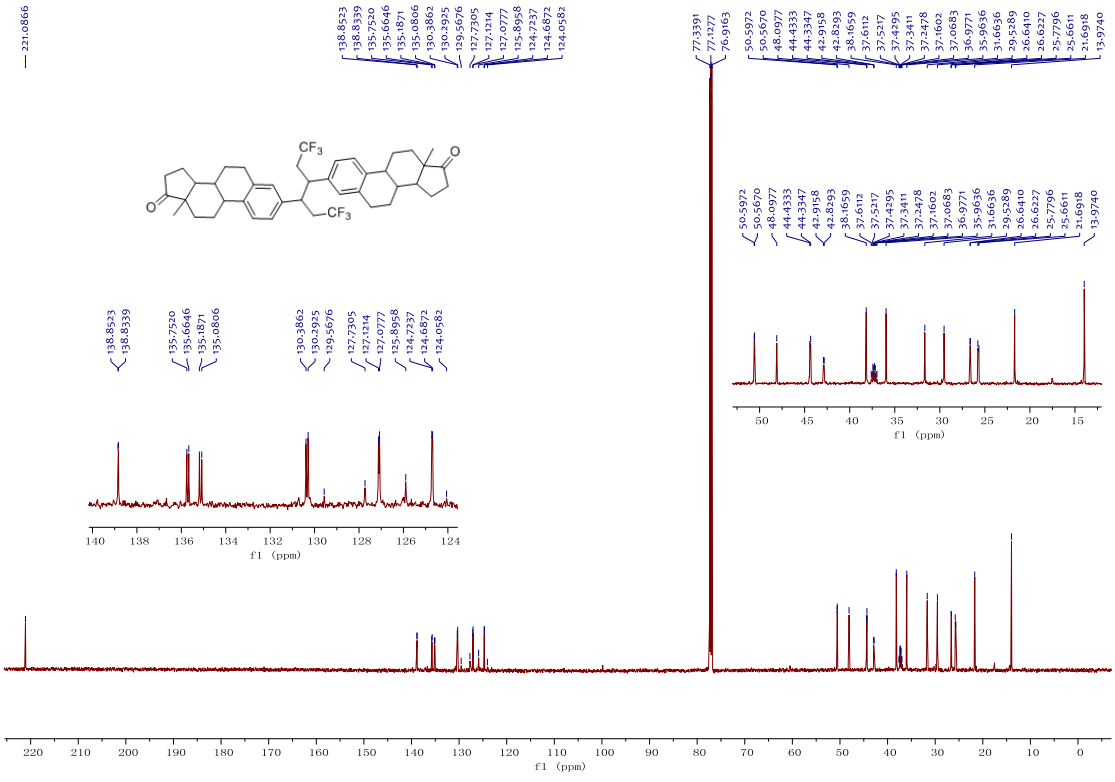
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **32**



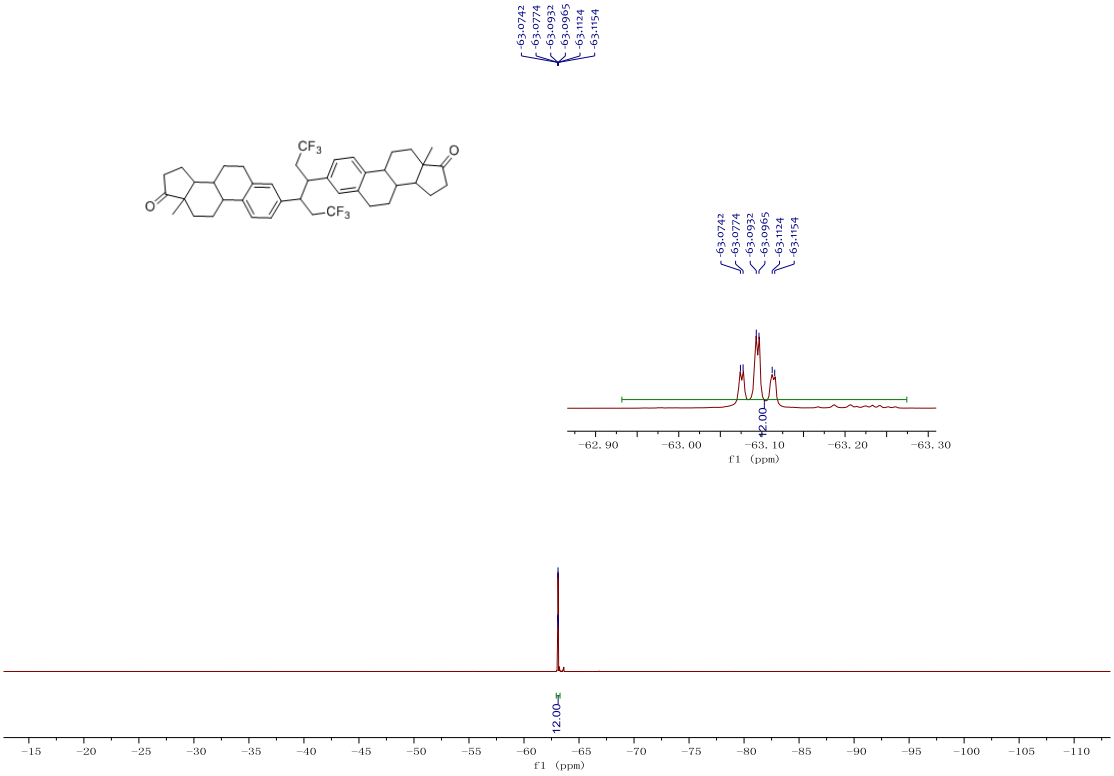
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **32**



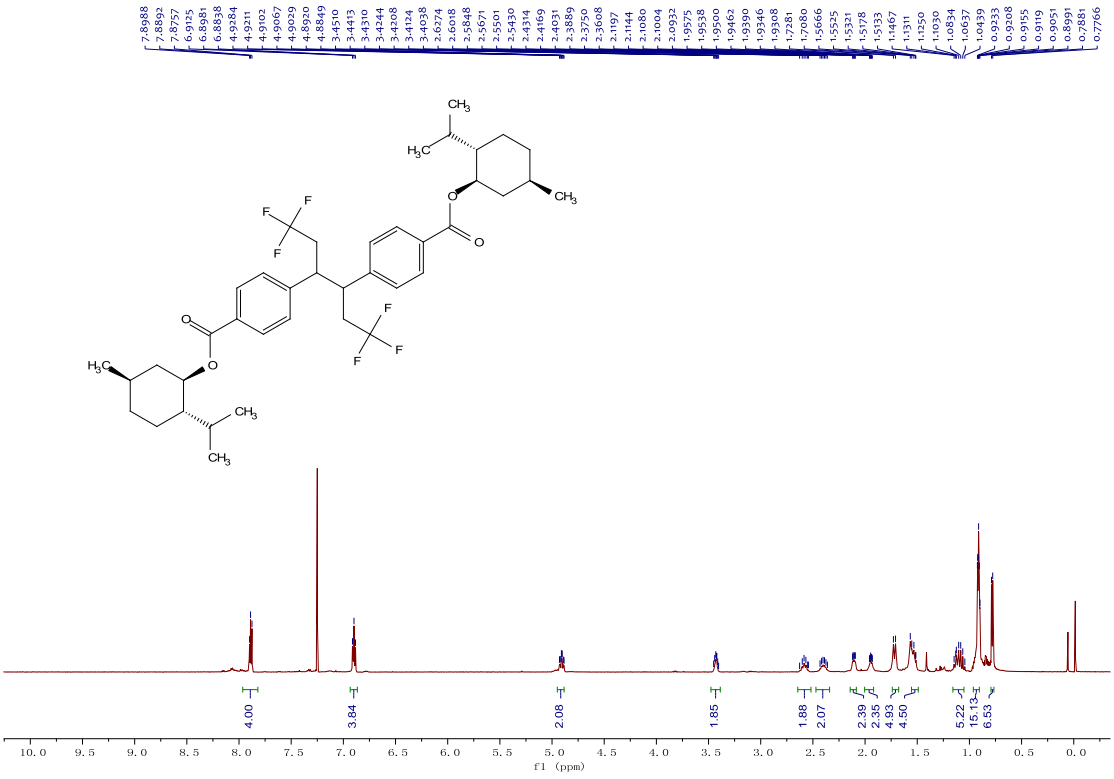
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **34**



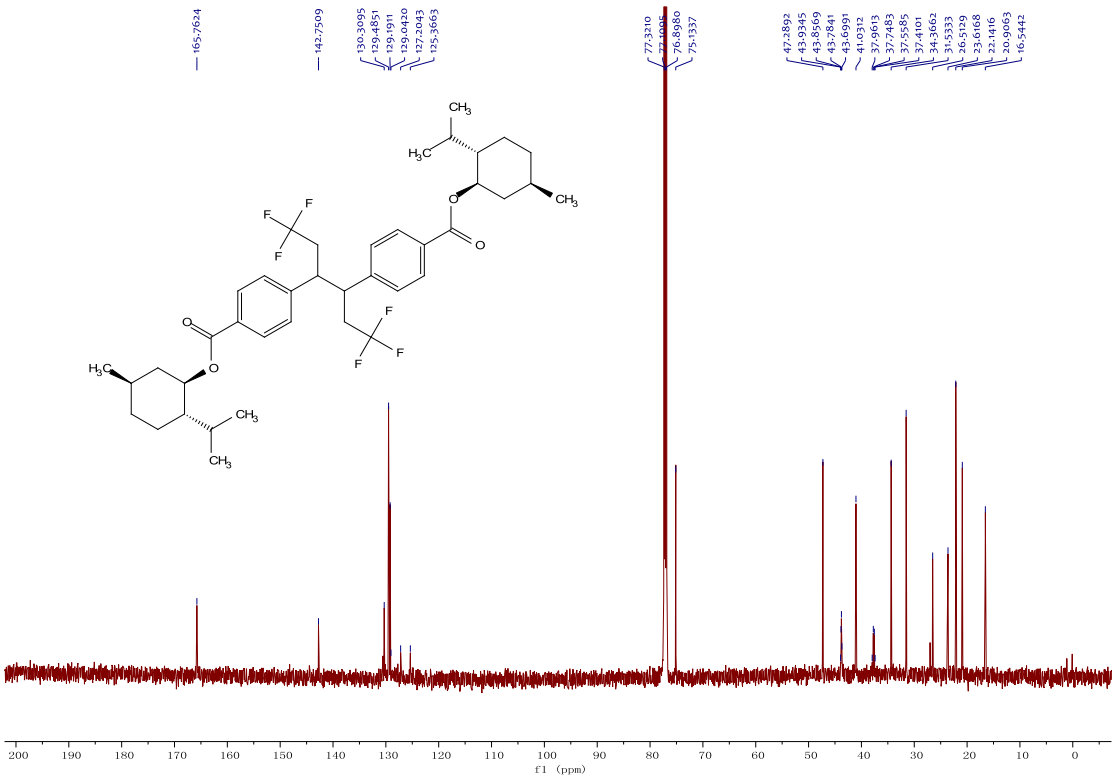
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **34**



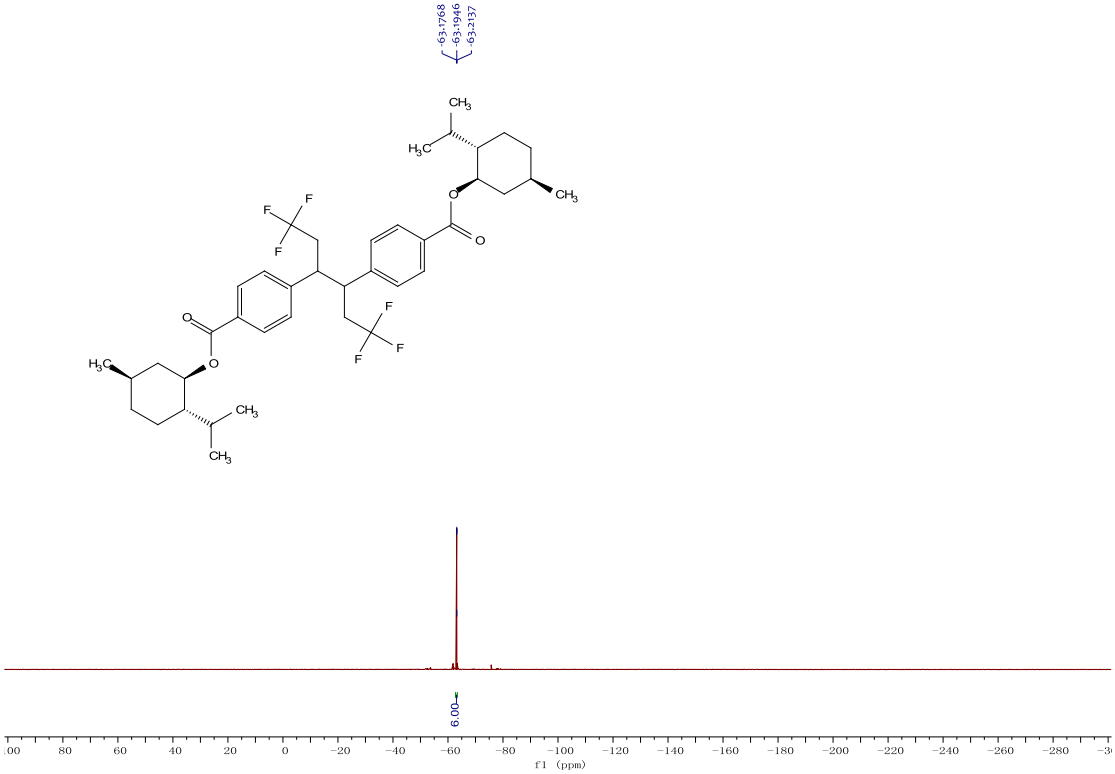
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **34**



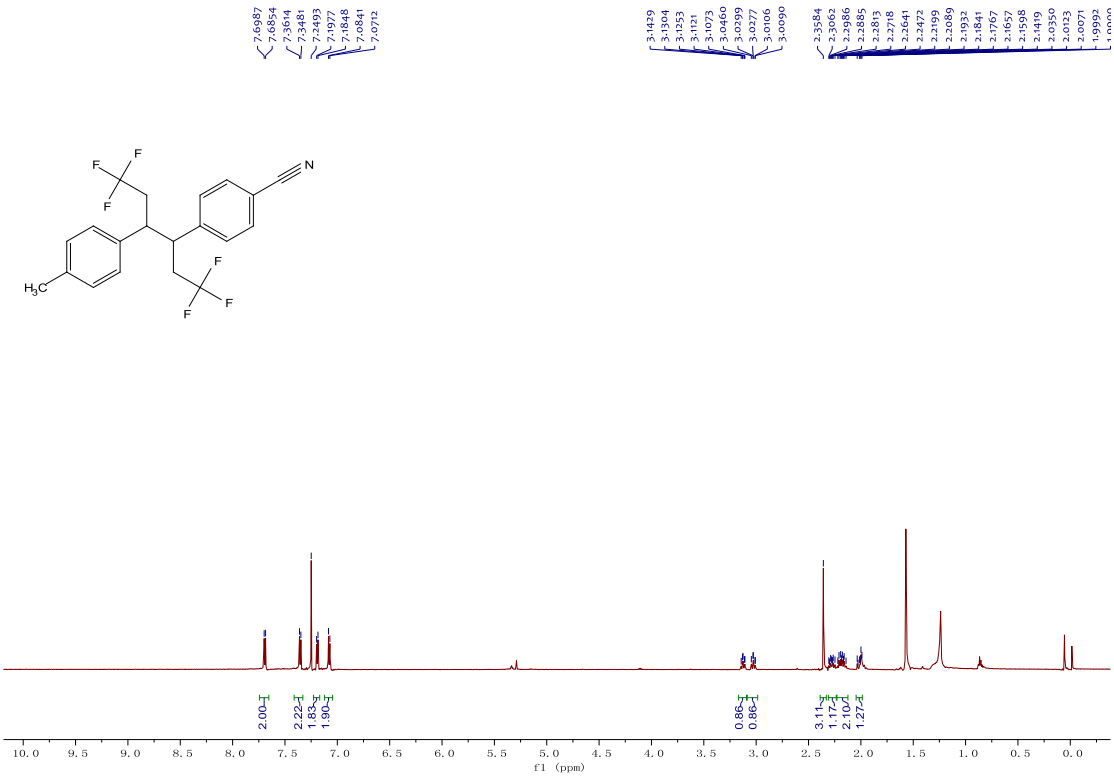
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **35**



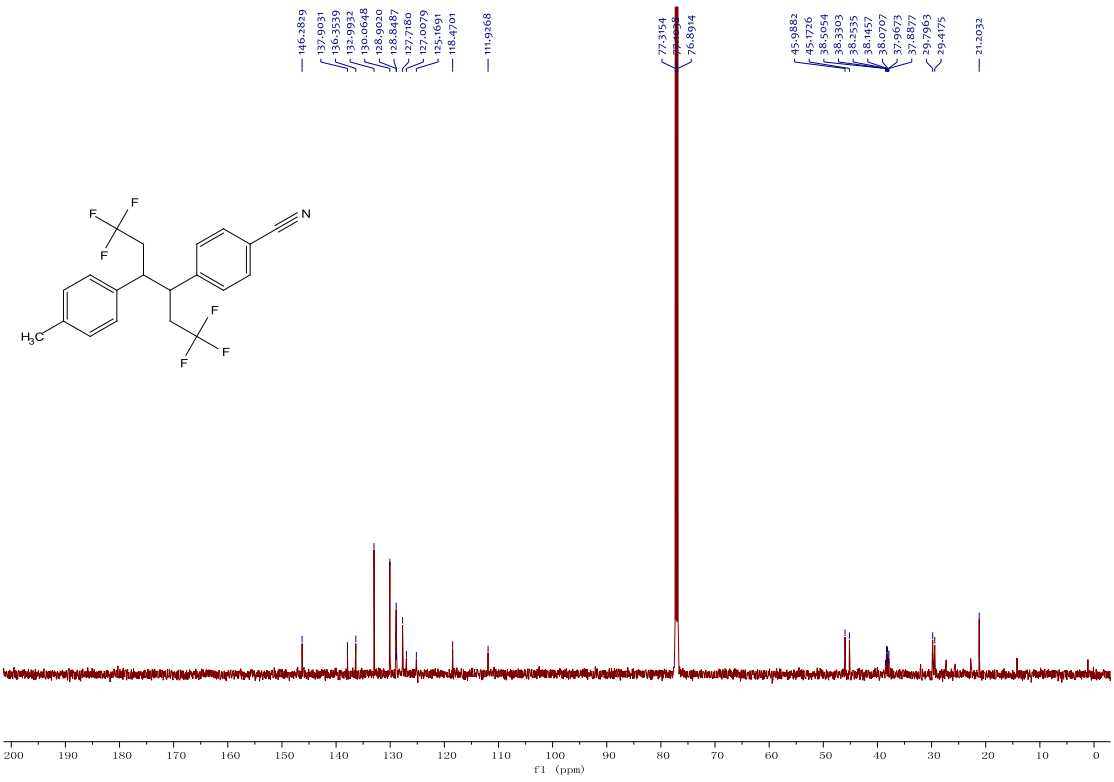
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **35**



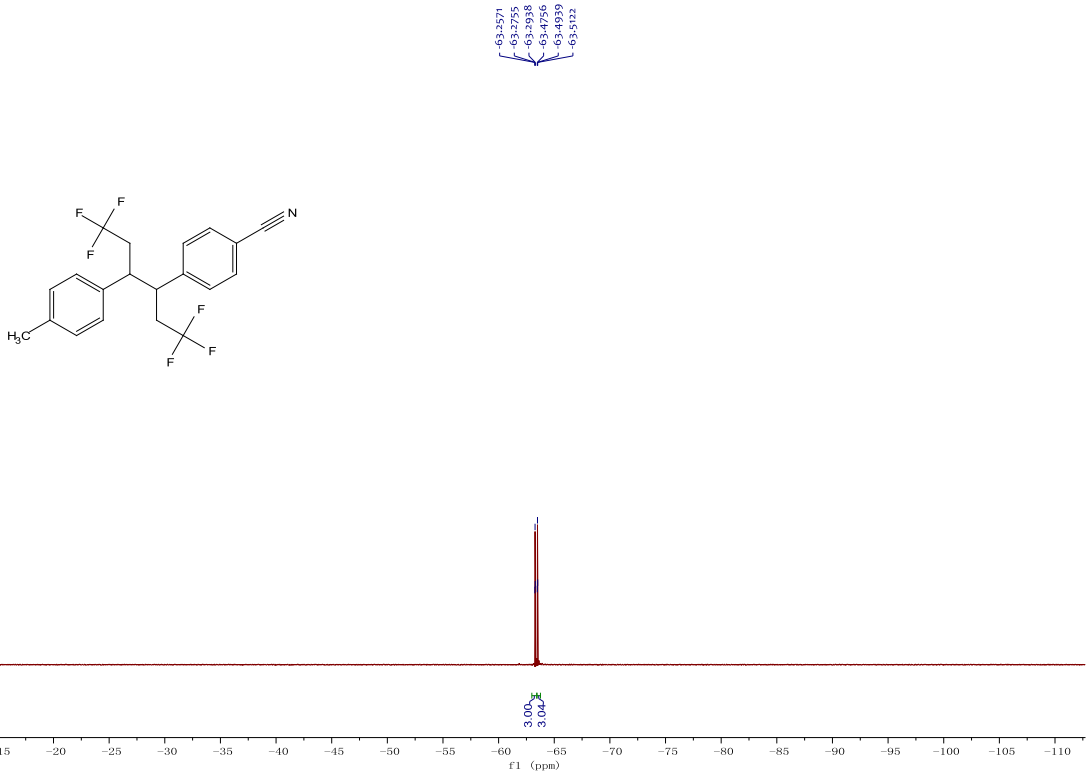
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **35**



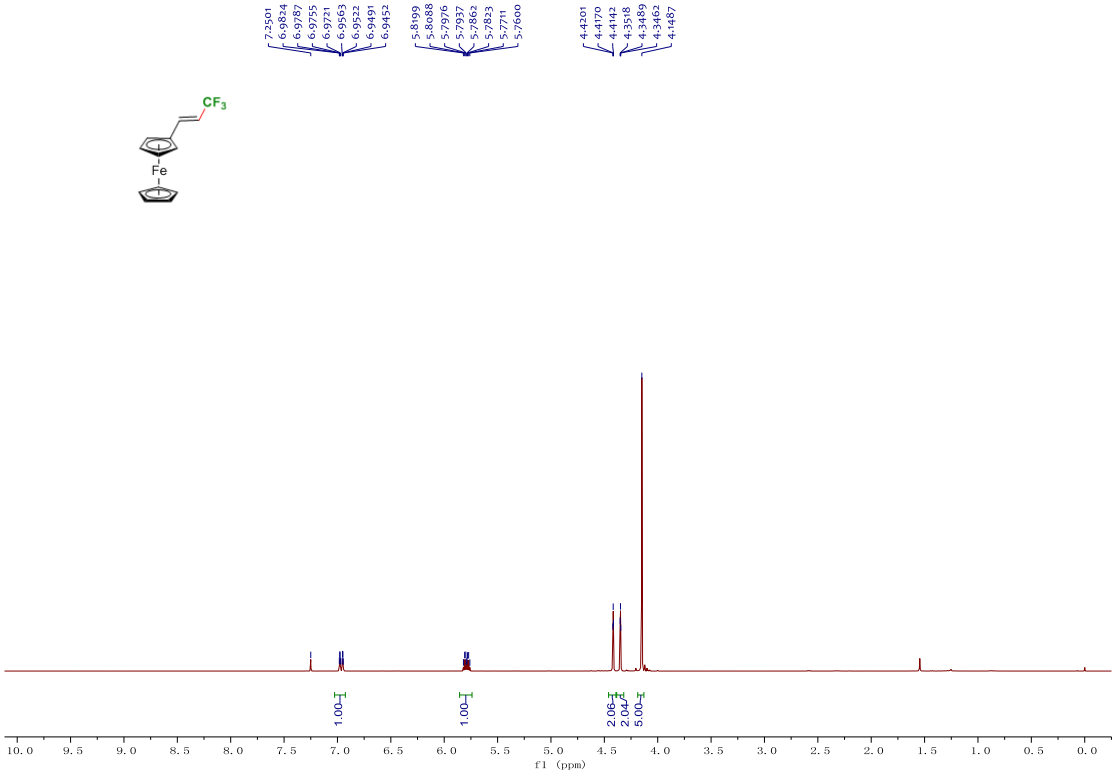
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **36** (anti)



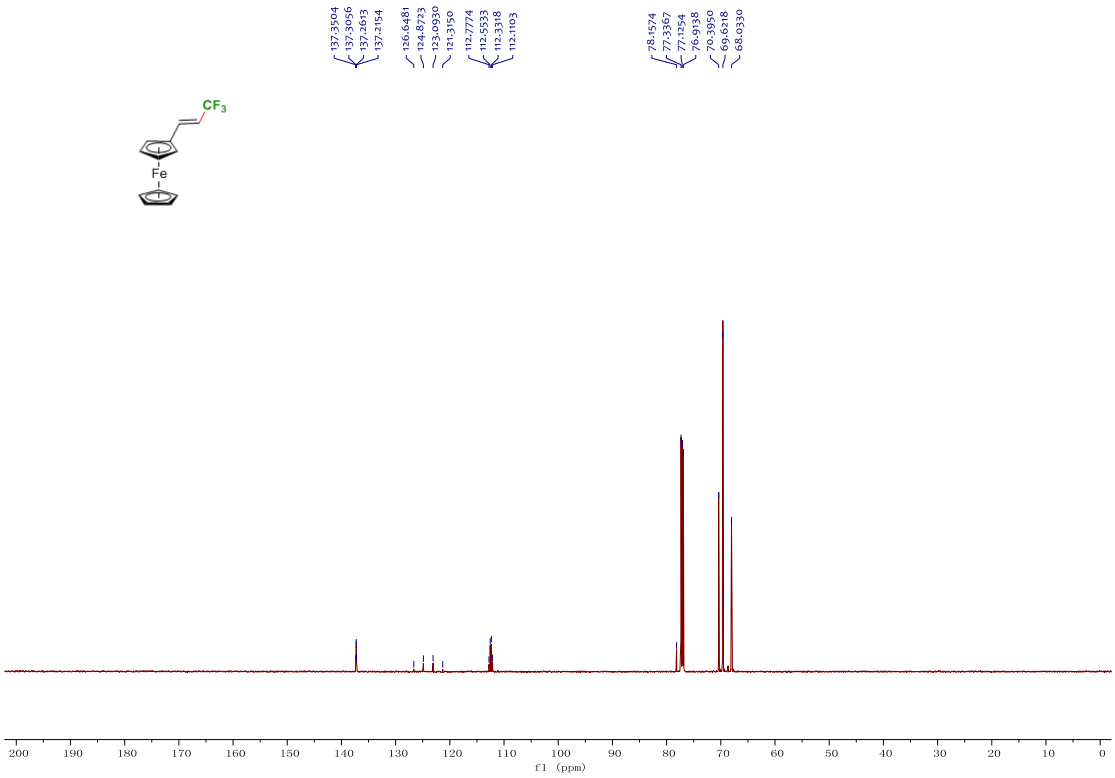
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **36** (anti)



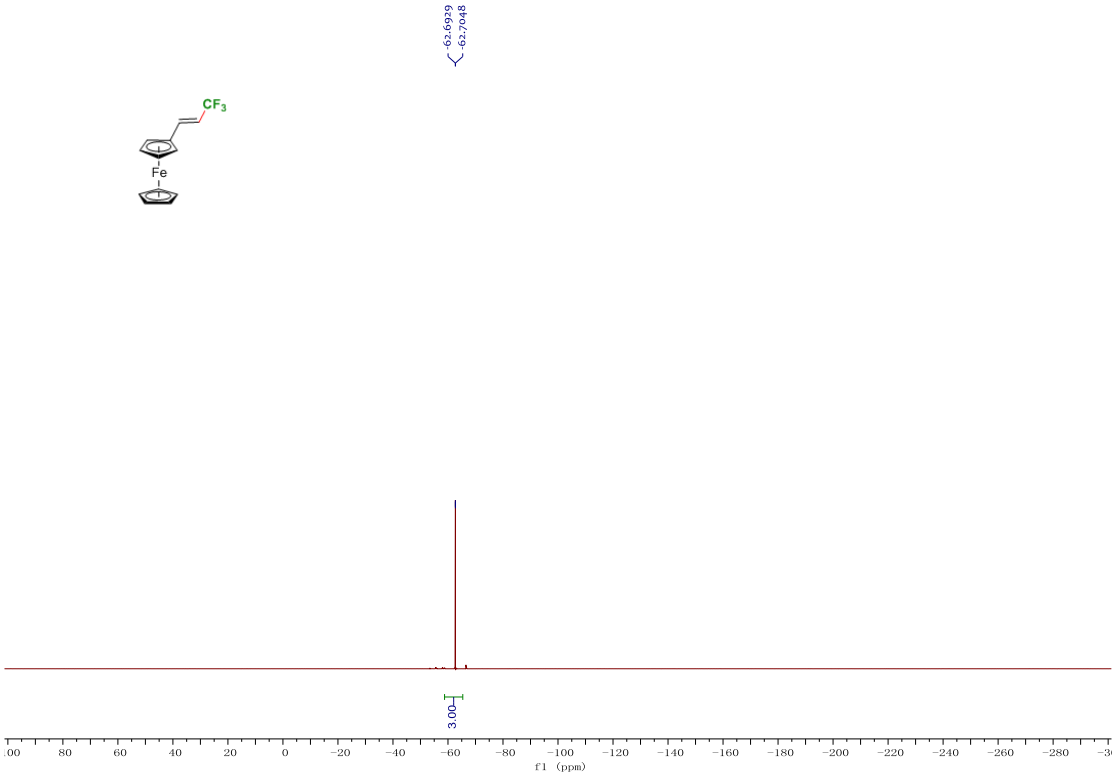
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **36** (anti)



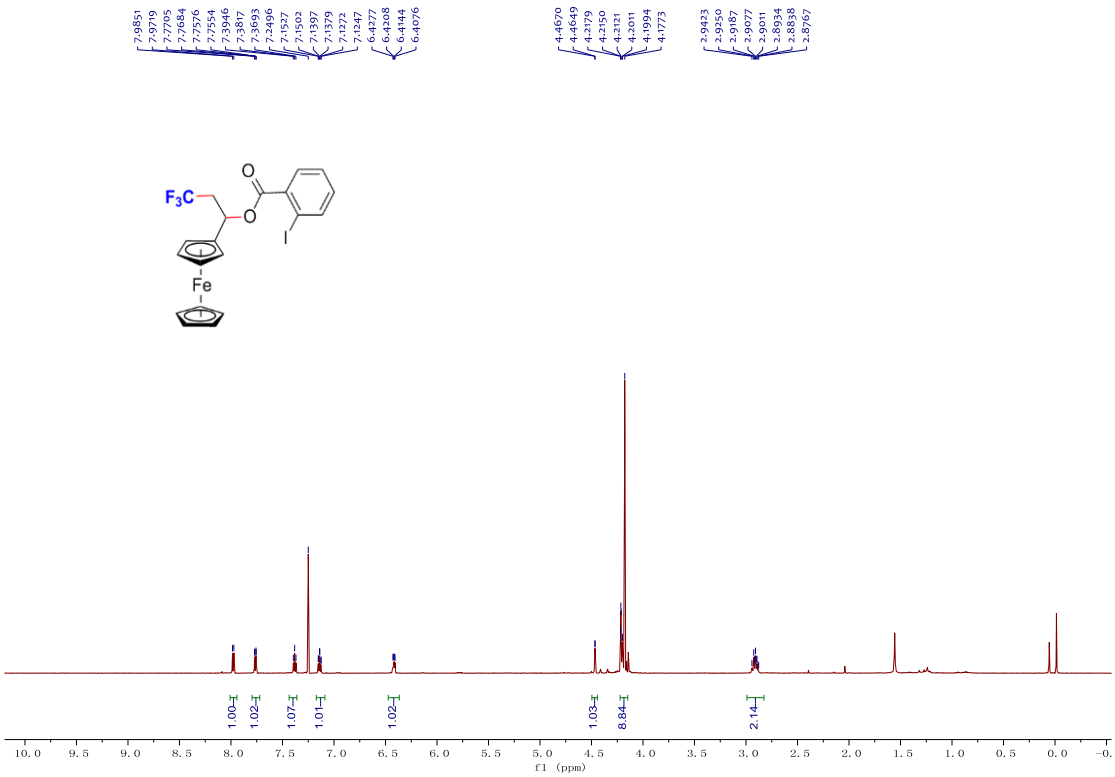
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **38**



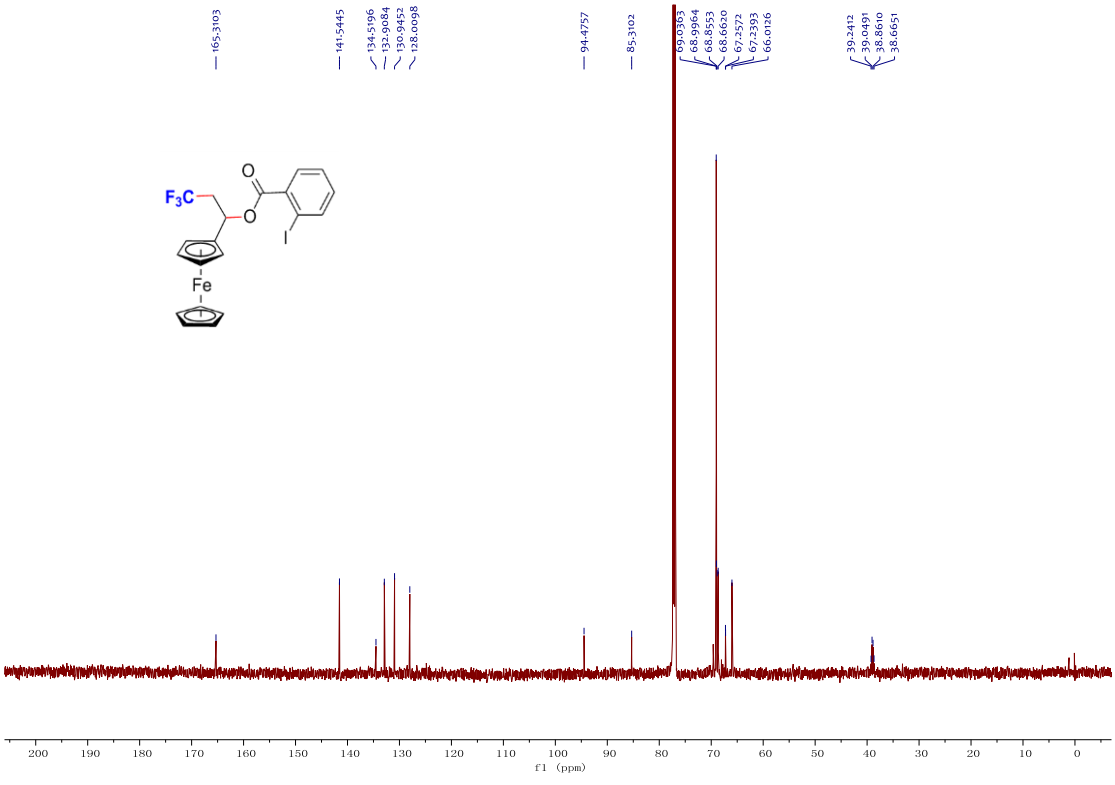
^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **38**



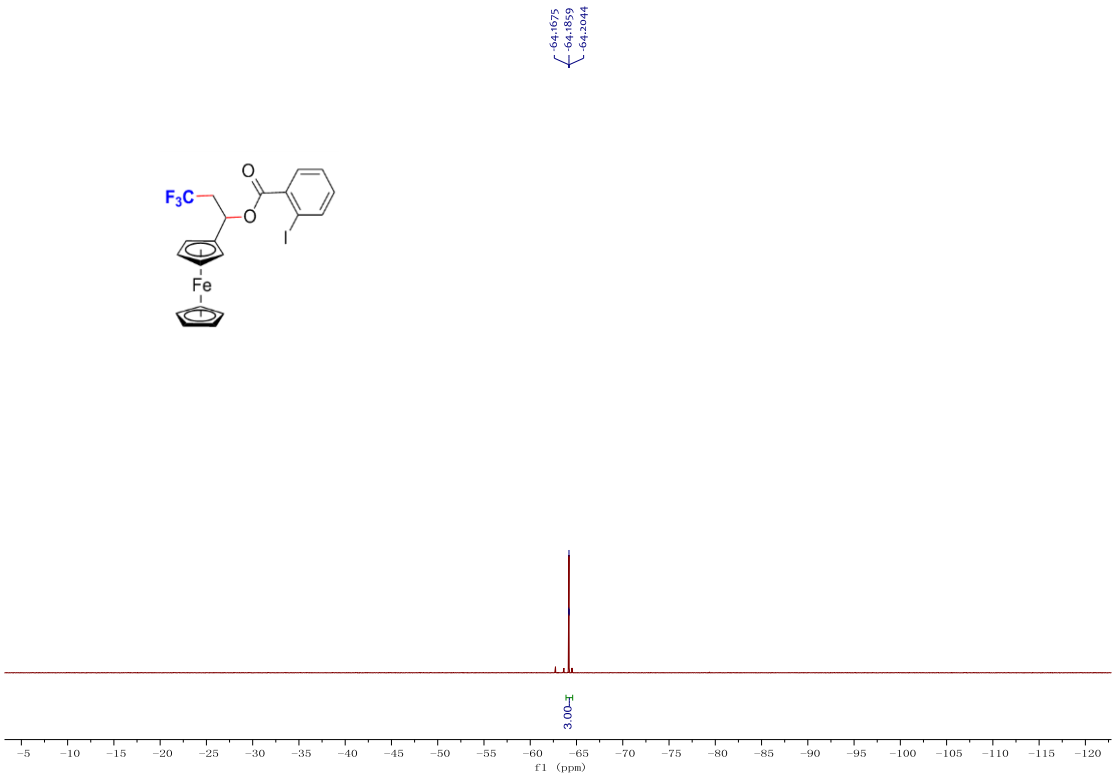
^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **38**



^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **39**



^{13}C NMR spectrum (150 MHz, $CDCl_3$) of **39**



^{19}F NMR spectrum (564 MHz, $CDCl_3$) of **39**