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# Efficient access to β-Amino Acid Ester/β-Amino Ketone Derivatives via Photocatalytic Radical Alkoxycabonylimidation/ Carbonylimidation of Alkenes

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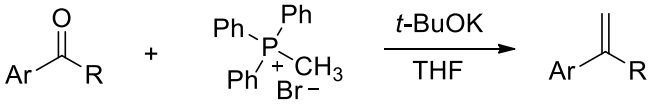
## General Experimental Informations

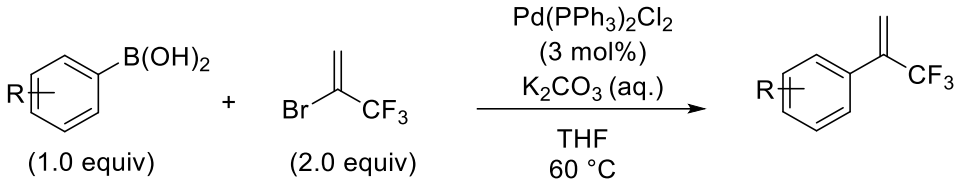
Unless otherwise noted, all the reagents were purchased from commercial suppliers and used without further purification. $^{1}H$ NMR spectra were recorded at 400 or 600 MHz. The chemical shifts were recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t =triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. $^{13}C$ NMR data were collected at 100 or 150 MHz with complete proton decoupling. $^{19}F$ NMR data were collected at 375 or 576 MHz. The HRMS spectrum was measured by micromass $QTOF_2$ Quadrupole/Time of Flight Tandem mass spectrometer with electron spray ionization. Cyclic voltammograms were recorded on a CHI 660E potentiostat.

## General procedure for the preparation of alkenes[a]



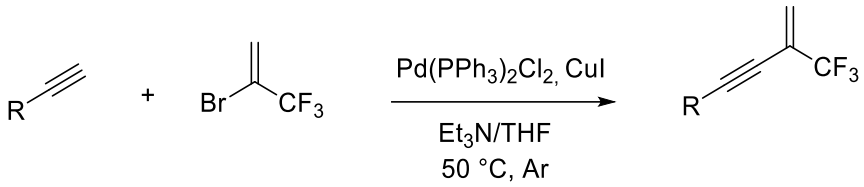

An oven-dried flask was charged with ketone (1.0 equiv.), menthyl triphenyl (1.2 equiv.) and THF (2.5 mL/mmol). Then *t*-BuOK (1.2 equiv.) was added to the mixture and stirred at room temperature for 3 hours. The residue was partitioned between ethyl acetate (50 mL) and saturated brine (50 mL). The organic layer was washed with water, dried over $NaSO_4$ and concentrated. The residue was purified by column chromatography to afford the corresponding alkene.

## General procedure for the preparation of trifluoromethyl alkenes[b]



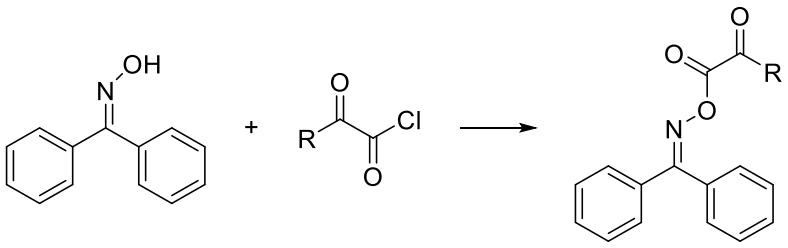

Trifluoromethyl alkenes were prepared according to reported synthetic procedures.( *Org. Lett.* **2017**, *19*, 946-949; *Angew. Chem. Int. Ed.*, 2020, **59**, 6706-6710.). To a Schlenk tube equipped with stir bar, arylboronic acid (1.0 equiv., 10 mmol) and $Pd(PPh_3)_2Cl_2$ (3 mol%, 0.3 mmol, 210.6 mg) were added. The vessel was evacuated and filled with argon (three times), and then aqueous $K_2CO_3$ (2.0 M, 20 mL) and THF (30 mL) were added. After addition of 2-bromo-3,3,3-trifluoropropene (2.0 equiv., 20 mmol, 2.1 mL), the solution was stirred at 60°C for 12 hours (TLC tracking detection). The solvent was removed under reduced pressure and the residue was purified by column chromatography to afford the corresponding trifluoromethyl alkene (PE - PE/EA=100:1).

## General procedure for the preparation of β--$CF_3$-1,3-Enynes[c]



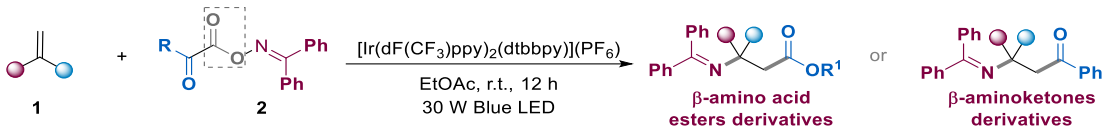

To an oven dried Schlenk flask equipped with a magnetic stir bar was successively added $PdCl_2(PPh_3)_2$ (0.01 equiv.) and CuI (0.02 equiv.). The system was purged with argon. Dry THF (50 mL) was then added followed by $Et_3N$ (5 mL), alkyne (5 mmol, 1 equiv.), 2-bromo-3,3,3-trifluoroprop-1-ene (6 mmol, 1.2 equiv.) via syringe. The reaction mixture was stirred at 50 °C for 12 h. The reaction was then cooled to room temperature, quenched with saturated $NH_4Cl$ solution and extracted with EtOAc. Combined extracts were dried over $Na_2SO_4$. After evaporation under vacuum, the residue was purified by flash chromatography over silica gel to give the desired $CF_3$-substituted 1,3-enyne.

## General procedure for the preparation of oxime oxalate 2a and 2b



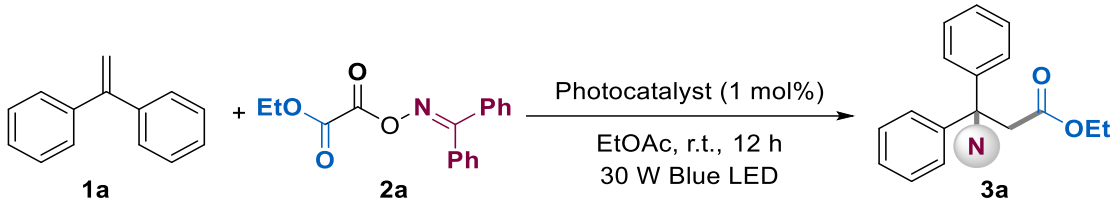

To a stirred solution of oxime (20 mmol, 1.0 equiv.) and pyridine (30 mmol, 1.5 equiv.) in anhydrous THF (1 M) at 0 °C was dropwise added corresponding Oxalyl chloride (30 mmol, 1.5 equiv.). The reaction was stirred for another 1 h at room temperature. After that, the reaction mixture was diluted with EtOAc and washed brine. The organic layers were dried with anhydrous $Na_2SO_4$ and concentrated in vacuo. The residue was purified by chromatography on silica gel to afford the corresponding oxime carbonate (79%-86%).

## Procedure for the preparation of compounds 3 and 4




To a 15 mL Schlenk flask equipped with a magnetic stirring bar, **1** (0.2 mmol, 1.0 equiv.), **2** (0.3 mmol, 1.5 equiv.), $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ (1 mmol %) and EtOAc (4 mL) were added. The vessel was evacuated and backfilled with Ar three times. The tube was screw-capped and stirred at room temperature under irradiation of 30 W blue LED (distance app. 3 cm) for 12 h. The solvent was removed under reduced pressure, and then the residue was purified by flash column chromatography (PE-PE/EtOAc = 1:9 to 1:4) to afford the desired product **3** or **4**.

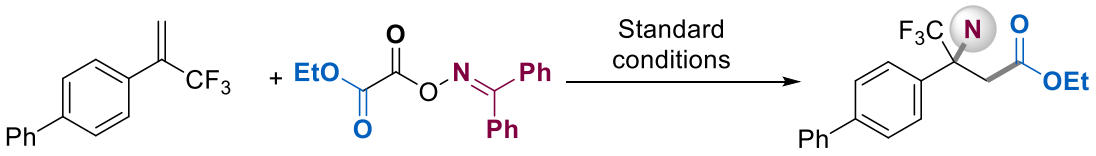
## Optimization of reaction conditions




| Entry | Photocatalyst | Solvent | Ratio (**1a**:**2a**) | Yield (%) |
|---|---|---|---|---|
| 1 | $[Mes\text{-}Acr](ClO_4)$ | EtOAc | 1:1.2 | 0 |
| 2 | $[Ru(bpy)_3](PF_6)_2$ | EtOAc | 1:1.2 | 0 |
| 3 | *fac*-$[Ir(ppy)_3]$ | EtOAc | 1:1.2 | 0 |
| 4 | $[Ir(ppy)_2(dtbbpy)][PF_6]$ | EtOAc | 1:1.2 | trace |
| 5 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | EtOAc | 1:1.2 | 62 |
| 6 | Thioxanthone | EtOAc | 1:1.2 | trace |
| 7 | Eosin Y | EtOAc | 1:1.2 | 0 |
| 8 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | THF | 1:1.2 | 28 |
| 9 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | PhMe | 1:1.2 | 44 |
| 10 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | MeCN | 1:1.2 | trace |
| 11 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | DMF | 1:1.2 | 41 |
| 12 | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | EtOAc | 1:1.5 | 71 |
| 13[b] | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | EtOAc | 1:1.5 | 76 |
| 14 | – | EtOAc | 1:1.5 | 0 |
| 15[c] | $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ | EtOAc | 1:1.5 | 0 |

[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), cat (1 mmol%), solvent (2 mL), 30 W blue LED (450 nm), 12 hours, argon atmosphere, rt. [b] EtOAc (4 mL). [c] Without LED.

**Time Profile of the Transformation with the Light ON/OFF over Time[d]**




Standard reactions were set up parallel on a 0.40 mmol scale according to the stand condition with $PhCF_3$ (0.4 mmol) as an internal standard. After being irradiated for 2 h, an aliquot (200 μL) from the reaction mixture was transferred into a nuclear magnetic tube charged with 0.5 mL of $CDCl_3$-$d_1$. The yield of product **3t** was determined by $^{19}F$ NMR. Then the reaction mixture was stirred for 2 h with light-off. All of the following yields were analyzed in the identical way after a 2 hour light on or off.

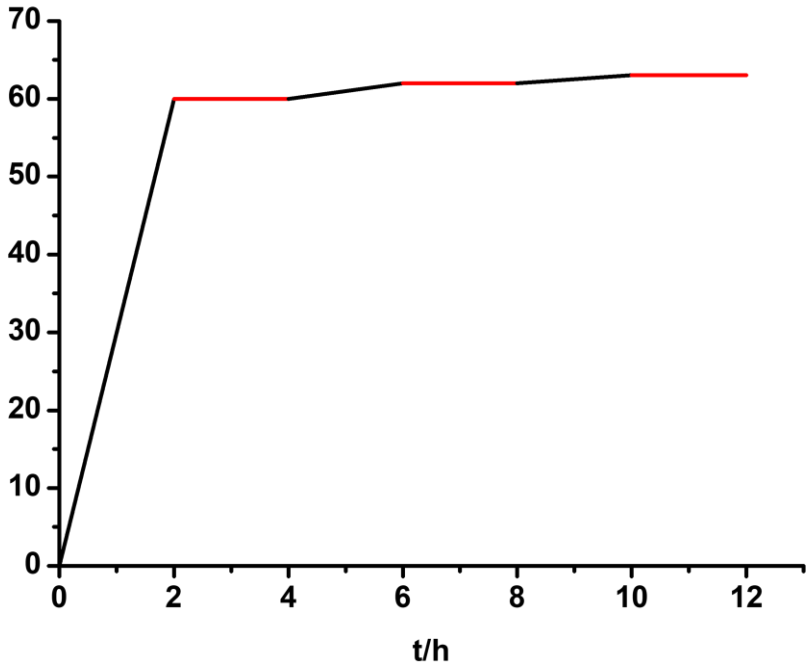



**Figure S1**. Time profile of the transformation with the light ON/OFF over time.

## Emission Quenching Experiments (Stern–Volmer Studies)[e]

All fluorescence measurements were recorded using a Hitachi FL-7000 Fluorometer. Quenching studies were conducted in EtOAc. All $[Ir(dFCF_3ppy)_2dtbbpy]PF_6$ solutions (concentration of 5 μM ) were excited at 320 nm and the emission intensity was collected at 517 nm. Measurements using corresponding quenchers were taken in triplicate at different concentrations.

## 2a of Fluorescence Spectra

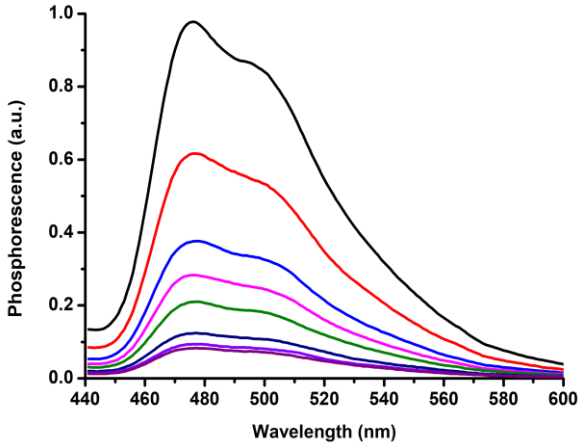



**Figure S2.** Fluorescence emission spectra of [Ir{dFCF3ppy}2(dtbbpy)]PF6 (33.3 μM) in deoxygenated EtOAc with different concentration of **2a** The excitation wavelength was 392 nm.

## 2b of Fluorescence Spectra

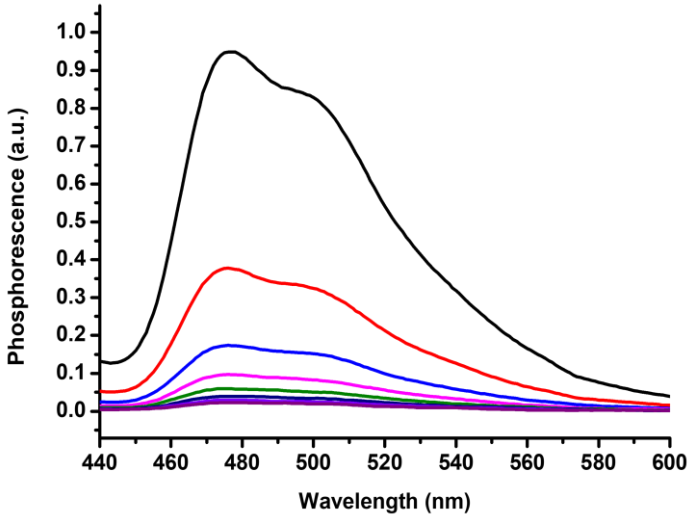


**Figure S3.** Fluorescence emission spectra of [Ir{dFCF3ppy}2(dtbbpy)]PF6 (33.3 μM) in deoxygenated EtOAc with different concentration of **2a** The excitation wavelength was 392 nm.

## CV studies for [Ir-F], 2a and 2b

Cyclic voltammetry Cyclic Voltammetry was performed using a CHI 660E potentiostat using a glassy carbon working electrode, Ag/AgCl in 3 M NaCl reference electrode, and a platinum counter electrode. The solution for test of [Ir-F], 2a and 2b was prepared by dissolving the sample (0.2 mmol) into a 0.1 M solution of tetrabutylammonium hexafluorophosphate ($TBAPF_6$) in EtOAc (10 mL). The potential range scanned was typically -2V and 2V at a 100mV/s.

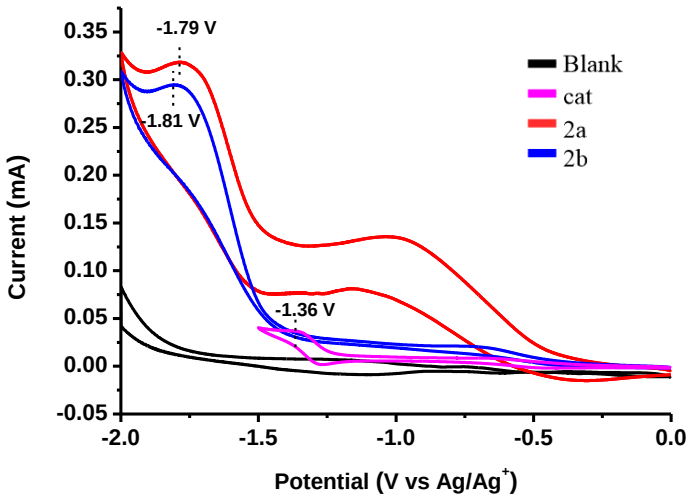
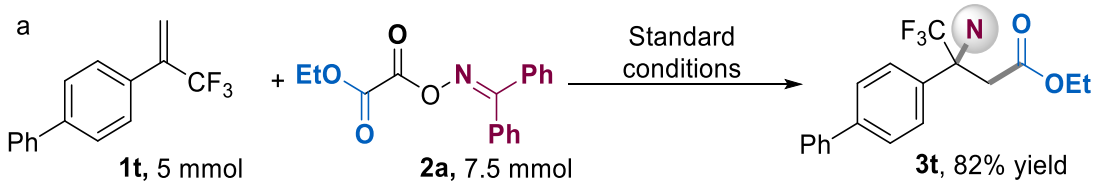


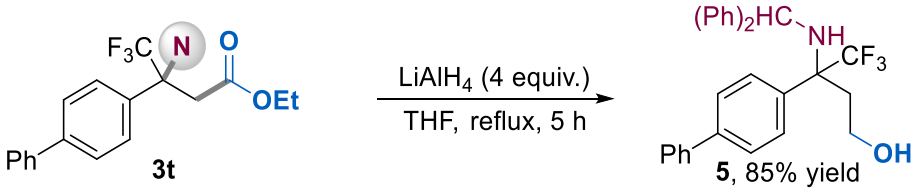

**Figure S4.** UV-Vis absorption spectra of photocatalyst [Ir{dFCF$_3$ppy}$_2$(dtbbpy)]PF$_6$ (15 μM), **2a** (40 μM), **2b** (40 μM), in deoxygenated EtOAc.

## Scale-up reaction

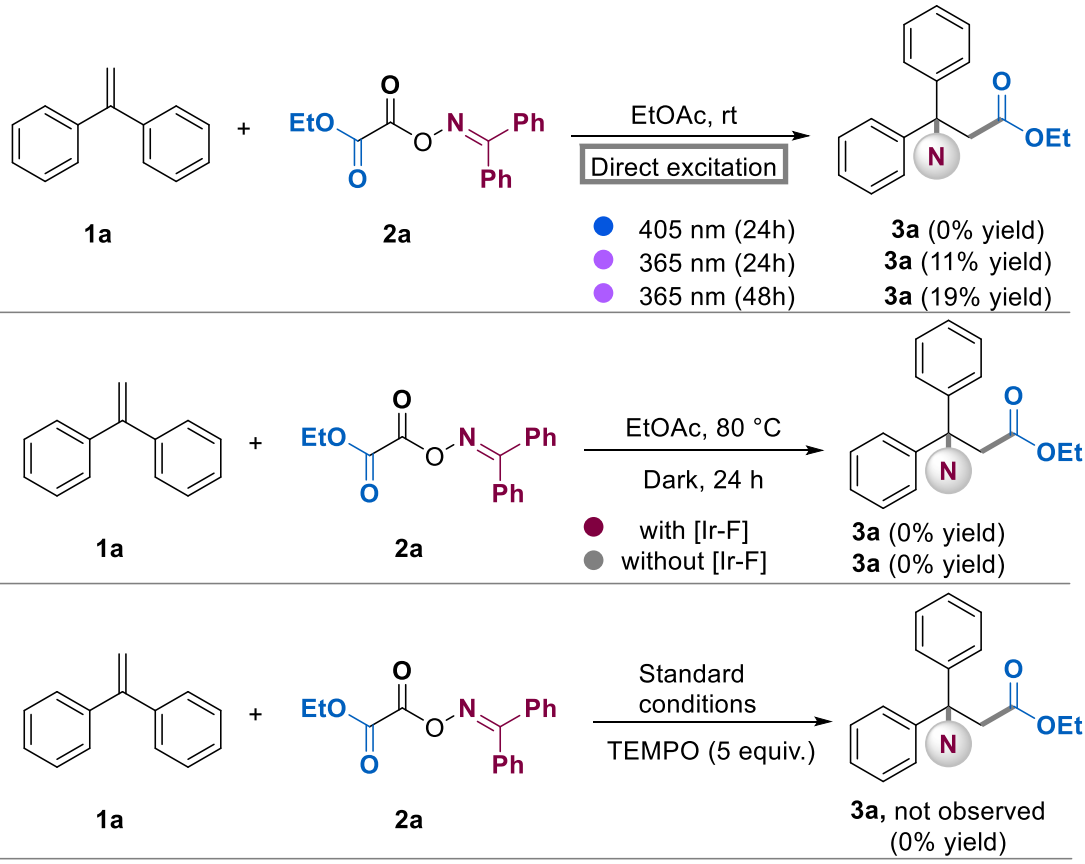



## Conversion of 3t

b




## Control reaction




## Unsuccessful substrates

## References:


*a.* M. L. Conner, M. K. Brown, *J. Org. Chem.* **2016**, *81*, 8050–8060.

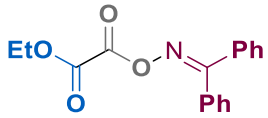
*b.* .Y. Liu, Y.-H. Zhou, Y.-L. Zhao, J.-P. Qu, *Org. Lett.* **2017**, *19*, 946-949.

c. N. J. Adamson, H. Jeddi, S. J. Malcolmson, *J. Am. Chem. Soc.* **2019**, *141*, 8574–8583.

d. M. A.Cismesiaa, T. P. Yoon, *Chem. Sci.* **2015**, *6*, 5426-5434.

e. J. D. Slinker, A. A. Gorodetsky, M. S. Lowry, J. Wang, S. Parker, R. Rohl, S. Bernhard, G. G. Malliaras, *J. Am. Chem. Soc*. **2004**, *126*, 2763-2767.

## Characterization data of compounds 2a-2c, 3a-3z, 4a-4r and 5.




**2a**

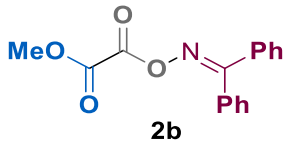
**2a**: light yellow solid, yield: 82%;

**m.p.** 75 – 76 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.57 – 7.60 (m, 2H), 7.44 – 7.52 (m, 4H), 7.37 – 7.41 (m, 4H), 4.31 (q, $J$ = 7.2 Hz, 2H), 1.31 (t, $J$ = 7.2 Hz, 1H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) $\delta$ 167.3, 157.3, 156.3, 134.1, 131.6, 131.5, 130.3, 129.4, 129.3, 128.6, 128.3, 63.2, 13.9 ppm;

HRMS (ESI): $C_{17}H_{15}NNaO_4^+$ $[M+Na]^+$ Calcd 320.0893, Found 320.0894.




**2b**

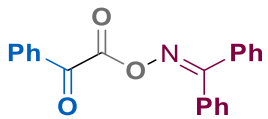
**2b**: light yellow solid, yield: 86%;

**m.p.** 80 – 81 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.56 – 7.59 (m, 2H), 7.45 – 7.50 (m, 4H), 7.37 – 7.40 (m, 4H), 3.85 (s, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) $\delta$ 167.5, 157.7, 156.1, 134.0, 131.6, 131.5, 130.4, 129.4, 129.2, 128.6, 128.3, 53.6 ppm;

HRMS (ESI): $C_{16}H_{13}NNaO_4^+$ $[M+Na]^+$ Calcd 306.0737, Found 306.0738.




**2c**

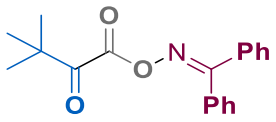
**2c**: white solid, yield: 79%;

**m.p.** 98 – 99 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.93 – 7.96 (m, 2H), 7.61 – 7.66 (m, 1H), 7.42 – 7.50 (m, 8H), 7.32 – 7.39 (m, 4H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 186.1, 166.7, 163.5, 135.1, 134.0, 132.5, 131.6, 131.5, 130.3, 129.80, 129.2, 129.2, 128.6, 128.4 ppm;

HRMS (ESI): $C_{21}H_{15}NNaO_3^+$ $[M+Na]^+$ Calcd 352.0944, Found 352.0940.




2d

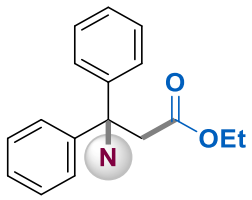
**2d**: white solid, yield: 73%;

**m.p.** 93 – 94 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.53 – 7.56 (m, 2H), 7.43 – 7.50 (m, 4H), 7.31 – 7.39 (m, 4H), 1.21 (s, 9H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 202.2, 166.4, 163.7, 134.0, 131.8, 131.5, 130.1, 129.1, 128.8, 128.6, 128.4, 42.7, 26.6 ppm;

HRMS (ESI): $C_{19}H_{19}NNaO_3^+$ $[M+Na]^+$ Calcd 332.1257, Found 332.1257.



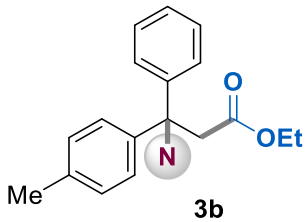

3a

**3a**: 65.8 mg, light yellow oil, yield: 76%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.67 – 7.70 (m, 2H), 7.28 – 7.37 (m, 7H), 7.11 – 7.20 (m, 7H), 7.06 (t, $J$ = 7.6 Hz, 2H), 6.59 – 6.62 (m, 2H), 3.86 (q, $J$ = 7.2 Hz, 2H), 3.18 (s, 2H), 0.93 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.3, 167.8, 148.9, 141.9, 138.5, 130.0, 128.5, 128.3, 128.0, 127.8, 127.7, 127.6, 127.4, 127.3, 126.3, 125.8, 67.3, 60.2, 45.1, 13.9 ppm;

HRMS (ESI): $C_{30}H_{28}NO_2^+$ $[M+H]^+$ Calcd 434.2115, Found 434.2114.



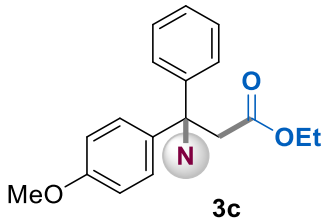

3b

**3b**: 66.3 mg, light yellow oil, yield: 78%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.60 – 7.61 (m, 2H), 7.27 – 7.30 (m, 1H), 7.20 – 7.25 (m, 4H), 7.04 – 7.12 (m, 6H), 6.98 (t, $J$ = 7.2 Hz, 2H), 6.91 (d, $J$ = 8.4 Hz, 2H), 6.53 – 6.54 (m, 2H), 3.79 (q, $J$ = 7.2 Hz, 2H), 3.08 (s, 2H), 2.22 (s, 3H), 0.86 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 169.3, 166.4, 147.9, 144.9, 140.9, 137.5, 134.6, 128.8, 127.4, 127.36, 126.9, 126.7, 126.6, 126.5, 126.2, 126.1, 125.1, 66.1, 59.0, 44.0, 20.0, 12.9 ppm;

HRMS (ESI): $C_{31}H_{30}NO_2^+$ $[M+H]^+$ $[M+Na]^+$ Calcd 448.2271, Found 448.2273.



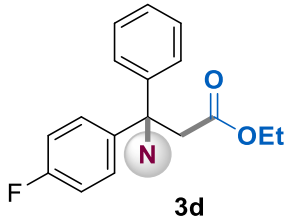

**3c**

**3c**: 64.8 mg, light yellow oil, yield: 74%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.66 – 7.69 (m, 2H), 7.29 – 7.36 (m, 5H), 7.13 – 7.21 (m, 6H), 7.06 – 7.10 (m, 2H), 6.70 – 6.73 (m, 2H), 6.61 – 6.63 (m, 2H), 3.87 (q, $J$ = 7.2 Hz, 2H), 3.77 (s, 3H), 0.94 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.3, 167.5, 157.9, 149.1, 141.9, 141.1, 138.6, 129.9, 128.8, 128.4, 127.9, 127.8, 127.7, 127.6, 127.4, 127.3, 126.2, 113.1, 66.9, 60.1, 55.3, 45.3, 14.0 ppm;

HRMS (ESI): $C_{31}H_{30}NO_2^+$ $[M+Na]^+$ Calcd 461.0560, Found 461.0564.




**3d**

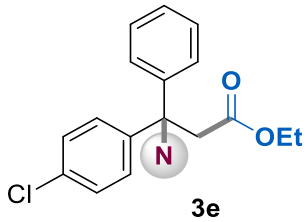
**3d**: 70.4 mg, light yellow oil, yield: 78%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.66 – 7.68 (m, 2H), 7.36 – 7.39 (m, 1H), 7.29 – 7.32 (m, 4H), 7.14 – 7.22 (m, 6H), 7.08 (t, $J$ = 7.8 Hz, 2H), 6.88 (t, $J$ = 9.0 Hz, 2H), 6.22 (d, $J$ = 7.2 Hz, 2H), 3.87 (q, $J$ = 7.2 Hz, 2H), 3.13 – 3.19 (m, 2H), 0.94 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -117.1 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.1, 167.9, 161.3 ( d, $^1J_{C\text{-}F}$ = 243.6 Hz), 148.5, 144.7 ( d, $^3J_{C\text{-}F}$ = 3.9 Hz), 141.7, 138.4, 130.0, 129.3 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 128.4, 127.9, 127.8, 127.6, 127.4, 127.4, 127.3, 126.4, 114.5, 114.3, 66.9, 60.2, 45.2, 13.9 ppm;

HRMS (ESI): $C_{30}H_{27}FNO_2^+$ $[M+H]^+$ Calcd 452.2020, Found 452.2038.



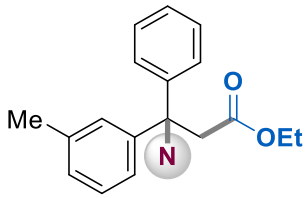

**3e**

**3e**: 64.4 mg, light yellow oil, yield: 69%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.65 – 7.70 (m, 2H), 7.36 – 7.39 (m, 1H), 7.29 – 7.34 (m, 4H), 7.14 – 7.22 (m, 8H), 7.09 (t, $J$ = 7.8 Hz, 2H), 6.62 (d, $J$ = 4.8 Hz, 2H), 3.88 (q, $J$ = 7.2 Hz, 2H), 3.12 – 3.20 (m, 2H), 0.95 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 167.0, 168.1, 148.2, 147.5, 141.6, 138.3, 131.9, 130.1, 129.2, 128.4, 127.9, 127.9, 127.8, 127.6, 127.4, 127.38, 127.35, 126.4, 66.9, 60.2, 45.0, 13.9 ppm;

HRMS (ESI): $C_{30}H_{27}ClNO_2^+$ $[M+H]^+$ Calcd 468.1725, Found 461.1724.



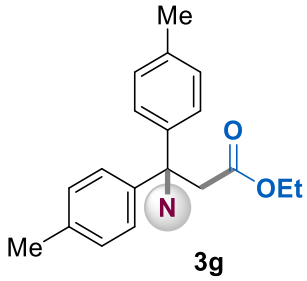

**3f**

**3e**: 63.5 mg, light yellow oil, yield: 71%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.67 – 7.70 (m, 2H), 7.29 – 7.38 (m, 5H), 7.14 – 7.22 (m, 4H), 7.00 – 7.08 (m, 5H), 6.93 – 6.95 (m, 1H), 6.60 (d, $J$ = 7.2 Hz, 2H), 3.86 (q, $J$ = 7.2 Hz, 2H), 3.11 – 3.21 (m, 2H), 2.21 (s, 3H), 0.95 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.4, 167.6, 149.0, 148.6, 142.0, 138.5, 137.1, 129.9, 128.5, 128.3, 127.9, 127.8, 127.7, 127.7, 127.6, 127.3, 127.2, 127.0, 126.2, 125.1, 67.3, 60.1, 45.2, 21.6, 13.9 ppm;

HRMS (ESI): $C_{31}H_{30}NO_2^+$ $[M+H]^+$ Calcd 448.2271, Found 448.2276.




**3g**

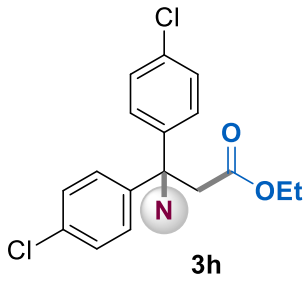
**3g**: 76.4 mg, white solid, yield: 83%;

**m.p.** 124 – 125 ℃;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.66 – 7.70 (m, 2H), 7.27 – 7.36 (m, 3H), 7.15 – 7.19 (m, 5H), 7.05 (t, $J$ = 7.6 Hz, 2H), 6.97 (d, $J$ = 7.6 Hz, 4H), 6.62 (d, $J$ = 7.2 Hz, 2H), 3.87 (q, $J$ = 7.2 Hz, 2H), 3.13 (s, 2H), 2.28 (s, 6H), 0.94 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.5, 167.3, 146.1, 142.0, 138.7, 135.6, 129.9, 128.5, 128.5, 127.9, 127.6, 127.6, 127.3, 127.2, 67.0, 60.1, 45.2, 21.1, 14.0 ppm;

HRMS (ESI): $C_{32}H_{31}NO_2^+$ $[M+H]^+$ Calcd 461.2355, Found 461.2389.



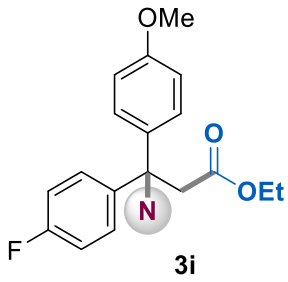

3h

**3h**: 71.1mg, light yellow oil, yield: 71%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.63 – 7.66 (m, 2H), 7.30 – 7.40 (m, 3H), 7.09 – 7.24 (m, 11H), 6.63 – 6.65 (m, 2H), 3.88 (q, $J$ = 7.2 Hz, 2H), 3.14 (s, 2H), 2.28 (s, 6H), 0.95 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 169.7, 168.5, 146.9, 141.4, 138.3, 132.3, 130.3, 129.2, 128.4, 128.1, 128.0, 127.6, 127.5, 127.4, 66.7, 60.4, 45.2, 14.0 ppm;

HRMS (ESI): $C_{30}H_{26}Cl_2NO_2^+$ $[M+H]^+$ Calcd 502.1335, Found 502.1339.




3i

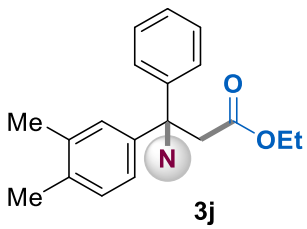
**3i**: 76.0 mg, light yellow oil, yield: 79%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.65 – 7.67 (m, 2H), 7.29 – 7.38 (m, 5H), 7.18 – 7.23 (m, 1H), 7.08 – 7.12 (m, 4H), 6.86 – 6.90 (m, 2H), 6.68 – 6.72 (m, 2H), 6.63 (d, $J$ = 8.0 Hz, 2H), 3.88 (q, $J$ = 7.2 Hz, 2H), 3.76 (s, 3H), 3.09 – 3.17 (m, 2H), 0.95 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -117.2 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.2, 167.7, 161.3 (d, $^{1}J_{C\text{-}F}$ = 245.0 Hz), 158.1, 145.0, 144.98 (d, $^{3}J_{C\text{-}F}$ = 3.0 Hz), 141.8, 140.8, 138.5, 130.1, 129.4 (d, $^{3}J_{C\text{-}F}$ = 7.6 Hz), 128.7, 128.4, 128.0, 127.5, 127.4, 127.3, 114.4 (d, $^{2}J_{C\text{-}F}$ = 21.2 Hz), 113.2, 66.6, 60.2, 55.3, 45.5, 14.0 ppm;

HRMS (ESI): $C_{31}H_{29}FNO_3^+$ $[M+H]^+$ Calcd 482.2126, Found 482.2123.



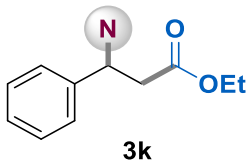

3j

**3j**: 69.2 mg, light yellow oil, yield: 75%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.67 – 7.69 (m, 2H), 7.29 – 7.38 (m, 5H), 7.12 – 7.22 (m, 4H), 7.03 – 7.07 (m, 2H), 6.90– 6.96 (m, 3H), 6.58 – 6.61 (m, 2H), 3.87 (q, $J$ = 7.2 Hz, 2H), 3.11 – 3.19 (m, 2H), 2.19 (s, 3H), 2.12 (s, 3H), 0.93 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.4, 167.3, 149.1, 146.0, 142.0, 138.6, 135.6, 134.4, 129.9, 129.0, 128.9, 128.4, 127.9, 127.7, 127.6, 127.2, 127.1, 126.1, 125.2, 67.1, 60.1, 45.2, 20.0, 19.4, 13.9 ppm;

HRMS (ESI): $C_{32}H_{32}NO_2^+$ $[M+H]^+$ Calcd 462.2428, Found 462.2428.




**3k**

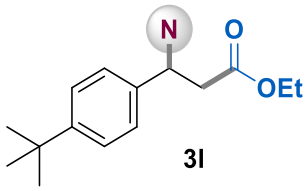
**3k**: 46.4 mg, light yellow solid, yield: 65%;

**m.p.** 102 – 103 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.64 – 7.66 (m, 2H), 7.26 – 7.43 (m, 9H), 7.20 – 7.24 (m, 2H), 7.03 – 7.07 (m, 2H), 7.03 – 7.06 (m, 2H), 4.87 (dd, $J$ = 9.2, 4.4 Hz, 1H), 3.98 – 4.12 (m, 2H), 3.06 (dd, $J$ = 15.0, 9.2 Hz, 1H), 2.77 (dd, $J$ = 15.0, 4.4 Hz, 1H), 1.16 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.5, 168.3, 143.7, 139.9, 136.8, 130.1, 129.4, 128.8, 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 127.8, 127.1, 126.7, 63.1, 60.3, 44.6, 14.3 ppm;

HRMS (ESI): $C_{24}H_{24}NO_2^+$ $[M+H]^+$ Calcd 358.1802, Found 358.1799.



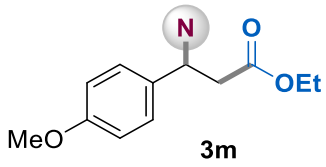

**3l**

**3l**: 56.2 mg, light yellow oil, yield: 68%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.62 – 7.65 (m, 2H), 7.40 – 7.43 (m, 3H), 7.27 – 7.35 (m, 5H), 7.19 – 7.21 (m, 2H), 7.06 – 7.09 (m, 2H), 4.85 (dd, $J$ = 9.2, 4.4 Hz, 1H), 3.99 – 4.11 (m, 2H), 3.05 (dd, $J$ = 15.0, 9.2 Hz, 1H), 2.76 (dd, $J$ = 15.0, 4.6 Hz, 1H), 1.30 (s, 9H), 1.14 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.6, 167.8, 149.8, 140.4, 140.0, 136.8, 130.1, 129.4, 128.7, 128.5, 128.4, 128.0, 127.9, 126.7, 125.3, 62.7, 60.3, 44.5, 34.5, 31.4, 14.2 ppm;

HRMS (ESI): $C_{28}H_{32}NO_2^+$ $[M+H]^+$ Calcd 414.2428, Found 414.2424.



3m

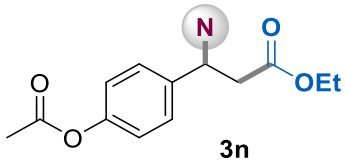
**3m**: 54.2 mg, light yellow solid, yield: 70%;

**m.p.** 110 – 111 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.62 – 7.64 (m, 2H), 7.38 – 7.43 (m, 3H), 7.28 – 7.36 (m, 3H), 7.16 – 7.19 (m, 2H), 7.04 – 7.06 (m, 2H), 6.80 – 6.83 (m, 2H), 4.82 (dd, $J$ = 9.2, 4.8 Hz, 1H), 3.99 – 4.12 (m, 2H), 3.77 (s, 3H), 3.03 (dd, $J$ = 15.0, 9.2 Hz, 1H), 2.74 (dd, $J$ = 15.0, 4.8 Hz, 1H), 1.16 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.5, 167.9, 158.6, 140.0, 136.8, 135.9, 130.0, 128.7, 128.5, 128.2, 128.1, 128.0, 127.9, 127.5, 127.3, 113.8, 62.4, 60.3, 55.2, 44.6, 14.3 ppm;

HRMS (ESI): $C_{25}H_{26}NO_3^+$ $[M+H]^+$ Calcd 388.1907, Found 388.1906.



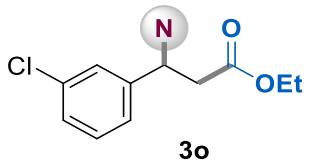
3n

**3n**: 60.6 mg, light yellow oil, yield: 73%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.62 – 7.65 (m, 2H), 7.37 – 7.43 (m, 3H), 7.25 – 7.37 (m, 5H), 7.03 – 7.07 (m, 2H), 6.98 – 7.02 (m, 2H), 4.87 (dd, $J$ = 9.2, 4.6 Hz, 1H), 3.99 – 4.11 (m, 2H), 3.03 (dd, $J$ = 15.0, 9.2 Hz, 1H), 2.75 (dd, $J$ = 15.0, 4.8 Hz, 1H), 2.73 (s, 3H), 1.15 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.3, 169.6, 168.4, 149.6, 141.2, 139.8, 136.6, 130.2, 128.7, 128.6, 128.3, 128.1, 128.0, 127.8, 121.5, 62.5, 60.4, 44.6, 21.2, 14.2 ppm;

HRMS (ESI): $C_{26}H_{26}NO_4^+$ $[M+H]^+$ Calcd 416.1856, Found 416.1858.



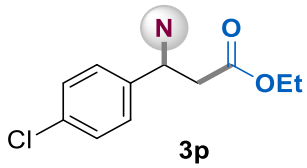
3o

**3o**: 48.5 mg, light yellow oil, yield: 62%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.63 – 7.66 (m, 2H), 7.28 – 7.44 (m, 7H), 7.18 – 7.21 (m, 2H), 7.10 – 7.13 (m, 1H), 7.02 – 7.05 (m, 2H), 4.83 (dd, $J$ = 9.0, 4.6 Hz, 1H), 4.00 – 4.11 (m, 2H), 3.01 (dd, $J$ = 15.0, 9.0 Hz, 1H), 2.75 (dd, $J$ = 15.0, 4.8 Hz, 1H), 2.73 (s, 3H), 1.16 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.1, 168.9, 145.7, 139.7, 136.5, 134.2, 130.3, 129.8, 129.3, 128.8, 128.7, 128.4, 128.3, 128.2, 128.0, 127.8, 127.3, 125.2, 62.5, 60.4, 44.4, 14.2 ppm;

HRMS (ESI): $C_{24}H_{23}ClNO_2^+$ $[M+H]^+$ Calcd 392.1412, Found 392.1412.




**3p**

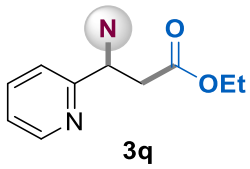
**3p**: 52.4 mg, white solid, yield: 67%;

**m.p.** 116 – 117 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) *δ* 7.62 – 7.65 (m, 2H), 7.29 – 7.42 (m, 6H), 7.23 – 7.26 (m, 2H), 7.18 – 7.20 (m, 2H), 7.01 – 7.04 (m, 2H), 4.84 (dd, *J* = 9.0, 4.8 Hz, 1H), 3.98 – 4.11 (m, 2H), 3.01 (dd, *J* = 15.0, 9.0 Hz, 1H), 2.74 (dd, *J* = 15.0, 4.8 Hz, 1H), 2.73 (s, 3H), 1.16 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.2, 168.7, 142.2, 139.7, 136.6, 132.7, 130.2, 128.7, 128.6, 128.4, 128.3, 128.1, 127.7, 62.3, 60.4, 44.4, 14.2 ppm;

HRMS (ESI): $C_{24}H_{23}ClNO_2^+$ $[M+H]^+$ Calcd 392.1412, Found 392.1410.



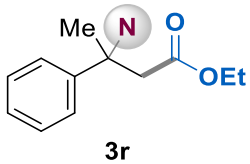

**3q**

**3q**: 47.3 mg, light yellow oil, yield: 66%;

**$^1$H NMR** (400 MHz, Chloroform-d) *δ* 8.51 – 8.53 (m, 1H), 7.67 – 7.71 (m, 2H), 7.60 – 7.64 (m, 1H), 7.37 – 7.42 (m, 4H), 7.31 – 7.35 (m, 3H), 7.08 – 7.15 (m, 3H), 5.07 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.00 – 4.11 (m, 2H), 3.00 – 3.12 (m, 2H), 1.15 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.5, 169.7, 162.1, 149.1, 139.8, 136.6, 136.4, 130.3, 128.9, 128.7, 128.4, 128.3, 128.1, 127.8, 122.1, 121.8, 64.4, 60.3, 42.5, 14.2 ppm;

HRMS (ESI): $C_{23}H_{23}ClN_2O_2^+$ $[M+H]^+$ Calcd 359.1754, Found 359.1756.



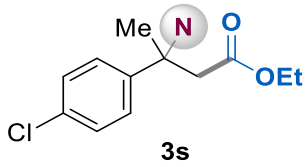

**3r**

**3r**: 58.6 mg, light yellow oil, yield: 79%;

**$^1$H NMR** (400 MHz, Chloroform-d) *δ* 7.57 – 7.59 (m, 2H), 7.27 – 7.37 (m, 3H), 7.14 – 7.20 (m, 6H), 7.07 (t, *J* = 7.6 Hz, 2H), 6.61 (t, *J* = 7.4 Hz, 2H), 4.03 (q, *J* = 7.2 Hz, 2H), 3.17 (d, *J* = 13.6 Hz, 1H), 2.93 (d, *J* = 13.6 Hz, 1H), 1.57 (s, 3H), 1.12 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.3, 166.7, 148.2, 141.5, 138.6, 129.8, 128.4, 128.3, 127.9, 127.8, 127.5, 127.3, 126.4, 126.4, 62.9, 60.0, 52.1, 25.5, 14.2 ppm;

HRMS (ESI): $C_{25}H_{26}NO_2^+$ $[M+H]^+$ Calcd 372.1958, Found 372.1967.



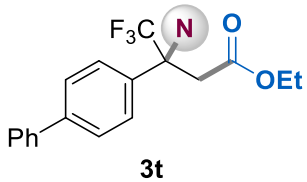

**3s**

**3s**: 62.4 mg, light yellow oil, yield: 77%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.50 – 7.51 (m, 2H), 7.26 – 7.30 (m, 1H), 7.20 – 7.24 (m, 2H), 7.12 – 7.16 (m, 1H), 7.01 – 7.07 (m, 6H), 6.56 (d, *J* = 7.2 Hz, 2H), 3.94 (q, *J* = 7.2 Hz, 2H), 3.05 (d, *J* = 13.8 Hz, 1H), 2.87 (d, *J* = 13.8 Hz, 1H), 1.49 (s, 3H), 1.05 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 169.9, 166.0, 145.4, 140.2, 137.4, 131.1, 129.1, 128.9, 127.3, 127.3, 126.9, 126.8, 126.7, 126.6, 126.4, 61.4, 59.0, 50.7, 24.5, 13.1 ppm;

HRMS (ESI): $C_{25}H_{25}ClNO_2^+$ $[M+H]^+$ Calcd 406.1568, Found 406.1568.




**3t**

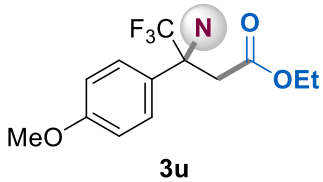
**3t**: 86.2 mg, light yellow oil, yield: 86%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.65 – 7.68 (m, 2H), 7.58 – 7.61 (m, 2H), 7.31 – 7.50 (m, 10H), 7.17 – 7.21 (m, 1H), 7.04 (t, *J* = 7.6 Hz, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.20 (d, *J* = 14.6 Hz, 1H), 2.76 (d, *J* = 14.6 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) *δ* -76.7 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.5, 168.8, 141.3, 140.9, 140.2, 139.1, 137.7, 130.6, 128.9, 128.8, 128.5, 128.3, 128.2, 128.1, 127.8, 127.7, 127.4, 127.1, 126.8, 126.2 (q, $^{1}J_{C\text{-}F}$ = 283.5 Hz), 67.9 (q, $^{2}J_{C\text{-}F}$ = 25.8 Hz), 60.8, 36.1, 29.8, 14.0 ppm;

HRMS (ESI): $C_{31}H_{27}F_3NO_2^+$ $[M+H]^+$ Calcd 502.1988, Found 502.1987.




**3u**

**3u**: 74.6 mg, light yellow oil, yield: 82%;

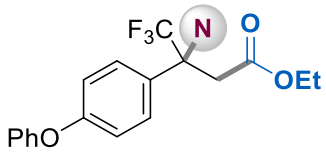
**$^{1}$H NMR** (600 MHz, Chloroform-d) *δ* 7.63 – 7.64 (m, 2H), 7.37 – 7.40 (m, 1H), 7.30 – 7.33 (m, 2H), 7.19 – 7.22 (m, 3H), 7.07 (t, *J* = 7.8 Hz, 2H), 6.77 – 6.80 (m, 2H), 6.64 – 6.66 (m, 2H), 4.11

(q, $J$ = 7.2 Hz, 2H), 3.80 (s, 3H), 3.12 (d, $J$ = 14.6 Hz, 1H), 2.65 (d, $J$ = 14.6 Hz, 1H), 1.18 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -77.1 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.1, 168.8, 159.6, 140.9, 137.7, 132.3, 130.4, 129.1, 128.8, 128.1, 128.0, 127.8, 127.3, 126.1 (q, $^{1}J_{C\text{-}F}$ = 282.3 Hz), 113.5, 67.6 (q, $^{2}J_{C\text{-}F}$ = 26.0 Hz), 60.7, 55.3, 35.9, 29.7, 14.0 ppm;

HRMS (ESI): $C_{26}H_{25}F_3NO_3^+$ $[M+H]^+$ Calcd 456.1781, Found 456.1782.




**3v**

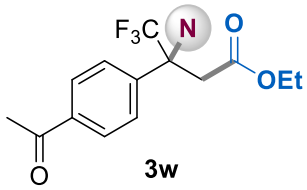
**3v**: 87.9 mg, light yellow oil, yield: 85%;

**$^{1}$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.63 – 7.64 (m, 2H), 7.30 – 7.40 (m, 5H), 7.22 – 7.25 (m, 3H), 7.10 – 7.14 (m, 3H), 7.02 (d, $J$ = 8.1 Hz, 2H), 6.88 (d, $J$ = 8.8 Hz, 2H), 6.64 – 6.78 (m, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 3.14 (d, $J$ = 14.5 Hz, 1H), 2.71 (d, $J$ = 14.5 Hz, 1H), 1.18 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -76.8 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.4, 168.7, 157.5, 156.6, 140.8, 137.7, 134.6, 130.5, 129.9, 129.5, 128.8, 128.3, 128.2, 128.0, 127.8, 127.4, 126.3 (q, $^{1}J_{C\text{-}F}$ = 285.4 Hz), 123.7, 119.1, 118.1, 67.6 (q, $^{2}J_{C\text{-}F}$ = 25.9 Hz), 60.8, 36.1, 29.7, 14.0 ppm;

HRMS (ESI): $C_{31}H_{26}F_3NNaO_3^+$ $[M+Na]^+$ Calcd 540.1757, Found 540.1776.




**3w**

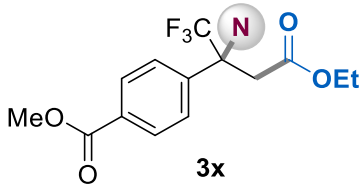
**3w**: 75.7 mg, light yellow oil, yield: 81%;

**$^{1}$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.89 – 7.91 (m, 1H), 7.82 – 7.84 (m, 1H), 7.62 – 7.65 (m, 3H), 7.38 – 7.41 (m, 2H), 7.30 – 7.33 (m, 2H), 7.16 – 7.19 (m, 1H), 7.01 – 7.05 (m, 2H), 6.54 – 6.74 (m, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 3.23 (d, $J$ = 14.6 Hz, 1H), 2.82 (d, $J$ = 14.6 Hz, 1H), 2.53 (s, 3H), 1.18 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -76.6 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 197.5, 171.0, 168.5, 140.5, 137.5, 136.8, 133.2, 130.7, 128.8, 128.6, 128.5, 128.3, 128.3, 128.1, 127.6, 127.4, 127.2, 124.3 (q, $^{1}J_{C\text{-}F}$ = 285.3 Hz), 68.0 (q, $^{2}J_{C\text{-}F}$ = 25.9 Hz), 60.9, 36.3, 26.7, 13.9 ppm;

HRMS (ESI): $C_{27}H_{24}F_3NNaO_3^+$ $[M+Na]^+$ Calcd 490.1600, Found 490.1605.




**3x**

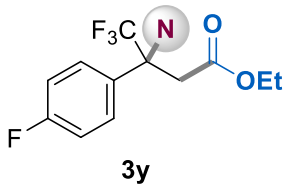
**3x**: 77.3 mg, light yellow oil, yield: 80%;

**$^{1}$H NMR** (600 MHz, Chloroform-d) *δ* 7.93 – 7.94 (m, 2H), 7.63 – 7.65 (m, 2H), 7.39 – 7.42 (m, 3H), 7.32 – 7.35 (m, 2H), 7.19 – 7.22 (m, 1H), 7.04 – 7.08 (m, 2H), 6.52 – 6.74 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.92 (s, 3H), 3.18 (d, *J* = 14.6 Hz, 1H), 2.74 (d, *J* = 14.6 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) *δ* -76.6 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.9, 168.4, 166.6, 144.9, 140.6, 137.5, 130.7, 130.2, 129.3, 128.9, 128.4, 128.2, 128.1, 127.7, 127.5, 126.0 (q, $^{1}J_{C\text{-}F}$ = 285.4 Hz), 68.0 (q, $^{2}J_{C\text{-}F}$ = 25.8 Hz), 60.8, 52.3, 36.0, 29.7, 14.0 ppm;

HRMS (ESI): $C_{27}H_{25}F_3NO_4^+$ $[M+H]^+$ Calcd 484.1730, Found 484.1730.




**3y**

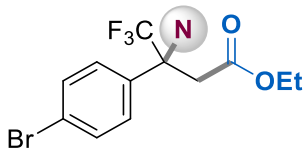
**3y**: 69.1 mg, light yellow oil, yield: 78%;

**$^{1}$H NMR** (600 MHz, Chloroform-d) *δ* 7.62 – 7.64 (m, 2H), 7.39 – 7.41 (m, 1H), 7.32 – 7.34 (m, 2H), 7.28 – 7.30 (m, 2H), 7.21 – 7.24 (m, 1H), 7.09 (t, *J* = 7.7 Hz, 2H), 6.95 – 6.98 (m, 2H), 6.55 – 6.72 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.12 (d, *J* = 14.6 Hz, 1H), 2.70 (d, *J* = 14.6 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) *δ* -77.0, -113.12 – 113.16 (m) ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.6, 168.5, 162.6 (d, $^{1}J_{C\text{-}F}$ = 249.3 Hz), 140.7, 137.6, 136.0, 135.9, 130.6, 129.9 (d, $^{3}J_{C\text{-}F}$ = 7.9 Hz), 128.8, 128.3, 128.0, 127.6, 127.4, 126.2 (q, $^{1}J_{C\text{-}F}$ = 284.9 Hz), 115.1 (d, $^{2}J_{C\text{-}F}$ = 21.7 Hz), 67.6 (q, $^{2}J_{C\text{-}F}$ = 26.1 Hz), 60.8, 36.1, 13.9 ppm;

HRMS (ESI): $C_{25}H_{22}F_4NO_2^+$ $[M+H]^+$ Calcd 444.1581, Found 444.1580.




**3z**

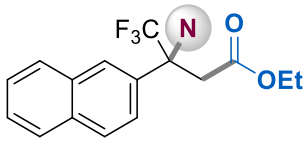
**3z**: 79.6 mg, light yellow oil, yield: 79%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.62 – 7.64 (m, 2H), 7.39 – 7.42 (m, 3H), 7.32 – 7.34 (m, 2H), 7.22 – 7.25 (m, 2H), 7.18 – 7.20 (m, 1H), 7.10 (t, $J$ = 7.7 Hz, 2H), 6.60 – 6.75 (m, 2H), 4.11 (q, $J$ = 7.2 Hz, 2H), 3.11 (d, $J$ = 14.9 Hz, 1H), 2.69 (d, $J$ = 14.9 Hz, 1H), 1.18 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -76.9 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.8, 168.5, 140.6, 139.1, 137.5, 131.3, 130.7, 129.8, 128.9, 128.4, 128.3, 128.1, 127.7, 127.5, 126.0 (q, $^1J_{C\text{-}F}$ = 284.8 Hz), 122.9, 67.4 (q, $^2J_{C\text{-}F}$ = 26.0 Hz), 60.9, 36.0, 14.0 ppm

HRMS (ESI): $C_{25}H_{22}BrF_3NO_2^+$ $[M+H]^+$ Calcd 504.0781, Found 504.0780.




**4a**

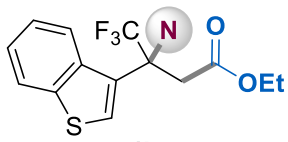
**4a**: 68.4 mg, light yellow oil, yield: 72%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.82 (d, $J$ = 8.0 Hz, 1H), 7.77 (d, $J$ = 8.7 Hz, 1H), 7.70 (d, $J$ = 8.1 Hz, 1H), 7.68 – 7.65 (m, 2H), 7.62 (d, $J$ = 2.0 Hz, 1H), 7.56 (d, $J$ = 8.8 Hz, 1H), 7.46 – 7.53 (m, 2H), 7.40 – 7.43 (m, 1H), 7.32 – 7.35 (m, 2H), 7.07 – 7.10 (m, 2H), 6.87 (s, 2H ), 6.45 – 6.70 (m, 2H), 4.14 (q, $J$ = 7.2 Hz, 2H), 3.32 (d, $J$ = 14.6 Hz, 1H), 2.83 (d, $J$ = 14.6 Hz, 1H), 1.21 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -76.5 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.6, 168.8, 140.9, 137.5, 137.4, 132.9, 132.6, 130.6, 128.9, 128.4, 128.3, 128.2, 128.0, 127.9, 127.7, 127.4, 127.2, 126.9, 126.8, 126.3, 126.0, 126.0 (q, $^1J_{C\text{-}F}$ = 285.3 Hz), 68.1 (q, $^2J_{C\text{-}F}$ = 26.0 Hz), 60.8, 36.1, 29.7, 14.0 ppm

HRMS (ESI): $C_{29}H_{25}F_3NO_2^+$ $[M+H]^+$ Calcd 476.1832, Found 476.1842.




**4b**

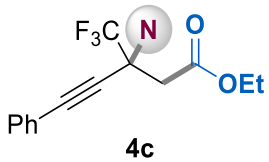
**4b**: 66.4 mg, light yellow oil, yield: 69%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 8.08 (d, $J$ = 8.3 Hz, 1H), 7.81 (d, $J$ = 8.1 Hz, 1H), 7.64 (dd, $J$ = 8.4, 1.4 Hz, 2H), 7.39 – 7.42 (m, 1H), 7.29 – 7.34 (m, 4H), 7.24 – 7.26 (m, 2H), 7.14 – 7.17 (m, 1H), 6.93 – 7.04 (m, 3H), 4.17 (q, $J$ = 7.2 Hz, 2H), 3.24 (d, $J$ = 13.8 Hz, 1H), 2.83 (d, $J$ = 13.8 Hz, 1H), 1.21 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -75.5 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 170.6, 168.5, 140.2, 140.1, 137.9, 136.8, 134.4, 130.6, 128.7, 128.2, 128.0, 127.2, 126.8, 126.7, 126.4 (q, $^1J_{C\text{-}F}$ = 284.6 Hz), 125.1, 124.4, 124.4, 122.6, 67.8 (q, $^2J_{C\text{-}F}$ = 27.4 Hz), 60.9, 38.1, 29.7, 13.9 ppm

HRMS (ESI): $C_{27}H_{23}F_3NO_2S^+$ $[M+H]^+$ Calcd 482.1396, Found 482.1395.




**4c**

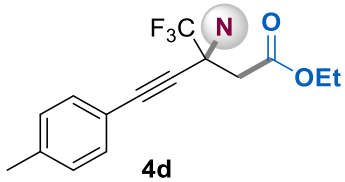
**4c**: 59.3 mg, light yellow oil, yield: 66%;

**$^1$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.58 – 7.60 (m, 2H), 7.35 – 7.40 (m, 3H), 7.29 – 7.32 (m, 4H), 7.22 – 7.25 (m, 2H), 7.17 – 7.20 (m, 2H), 7.03 – 7.04 (m, 2H), 4.14 (q, $J$ = 7.2 Hz, 2H), 3.28 (d, $J$ = 14.2 Hz, 1H), 3.06 (d, $J$ = 14.2 Hz, 1H), 1.19 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -77.5 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 171.8, 168.0, 140.6, 136.2, 131.8, 130.7, 129.0, 128.7, 128.6, 128.5, 128.0, 127.8, 127.5, 124.4 (q, $^1J_{C\text{-}F}$ = 283.9 Hz), 121.9, 91.2, 82.6, 63.4 (q, $^2J_{C\text{-}F}$ = 28.4 Hz), 60.8, 42.7, 29.7, 14.1 ppm;

HRMS (ESI): $C_{27}H_{23}F_3NO_2^+$ $[M+H]^+$ Calcd 450.1675, Found 450.1681.




**4d**

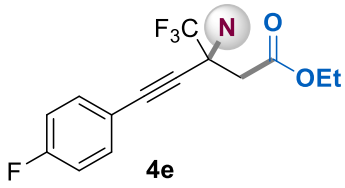
**4d**: 64.8 mg, light yellow oil, yield: 70%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.57 – 7.60 (m, 2H), 7.28 – 7.41 (m, 7H), 7.22 – 7.25 (m, 1H), 7.00 (d, $J$ = 8.0 Hz, 2H), 6.93 (d, $J$ = 8.0 Hz, 2H), 4.13 (q, $J$ = 7.2 Hz, 2H), 3.26 (d, $J$ = 14.2 Hz, 1H), 3.04 (d, $J$ = 14.2 Hz, 1H), 2.31 (s, 3H), 1.18 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -77.6 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.8, 168.1, 140.7, 138.7, 136.3, 132.5, 131.7, 130.7, 130.1, 129.4, 129.0, 128.7, 128.6, 128.3, 128.1, 128.0, 127.9, 127.5, 118.9, 91.3, 81.9, 63.4 (q, $^{2}J_{C\text{-}F}$ = 28.4 Hz), 60.8, 42.7, 21.5, 14.1 ppm;

HRMS (ESI): $C_{28}H_{25}F_3NO_2^+$ $[M+H]^+$ Calcd 464.1832, Found 464.1839.




**4e**

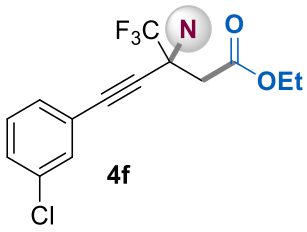
**4e**: 63.5 mg, light yellow oil, yield: 68%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.56 – 7.59 (m, 2H), 7.28 – 7.41 (m, 7H), 7.21 – 7.25 (m, 1H), 7.00 – 7.03 (m, 2H), 6.86 – 6.91 (m, 2H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.29 (d, *J* = 14.2 Hz, 1H), 3.05 (d, *J* = 14.2 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) *δ* -77.5, -110.39 – 110.42 (m) ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.8, 168.0, 162.6 (d, $^{1}J_{C\text{-}F}$ = 250.0 Hz), 140.6, 136.3, 133.8, 133.7, 130.8, 129.0, 128.7, 128.6, 128.0, 127.5, 115.1 (d, $^{2}J_{C\text{-}F}$ = 22.1 Hz), 90.3, 82.3, 63.4 (q, $^{2}J_{C\text{-}F}$ = 28.4 Hz), 60.9, 42.8, 14.2 ppm;

HRMS (ESI): $C_{27}H_{22}F_4NO_2^+$ $[M+H]^+$ Calcd 468.1581, Found 468.1588.




**4f**

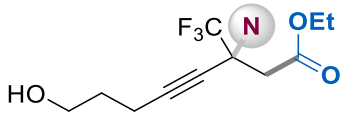
**4f**: 59.9 mg, light yellow oil, yield: 62%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.56 – 7.59 (m, 2H), 7.46 – 7.49 (m, 1H), 7.28 – 7.41 (m, 6H), 7.21 – 7.24 (m, 1H), 7.12 (t, *J* = 7.8 Hz, 2H), 6.97 (t, *J* = 1.8 Hz, 2H), 6.91 – 6.93 (m, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.31 (d, *J* = 14.2 Hz, 1H), 3.06 (d, *J* = 14.2 Hz, 1H), 1.20 (t, *J* = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) *δ* -77.4 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 172.0, 167.9, 159.0, 140.5, 138.2, 136.2, 135.5, 133.6, 131.8, 130.8, 130.1, 129.9, 129.6, 129.4, 129.1, 129.0, 128.9, 128.8, 128.7, 128.6, 128.3, 128.1, 127.9, 127.6, 124.3 (q, $^{1}J_{C\text{-}F}$ = 282.9 Hz), 123.6, 90.1, 83.8, 63.5 (q, $^{2}J_{C\text{-}F}$ = 28.5 Hz), 60.9, 42.8, 14.2 ppm;

HRMS (ESI): $C_{27}H_{22}ClF_3NO_2^+$ $[M+H]^+$ Calcd 484.1286, Found 484.1291.




**4g**

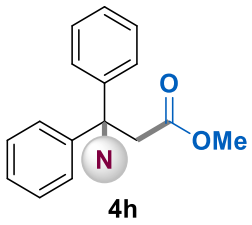
**4g**: 51.7 mg, light yellow oil, yield: 60%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.55 – 7.57 (m, 2H), 7.36 – 7.43 (m, 4H), 7.28 – 7.32 (m, 4H), 4.12 (q, $J$ = 7.2 Hz, 2H), 3.52 (t, $J$ = 6.1 Hz, 1H), 3.18 (d, $J$ = 14.2 Hz, 1H), 2.93 (d, $J$ = 14.2 Hz, 1H), 1.76 – 1.88 (m, 2H), 1.40 – 1.47 (m, 2H), 1.19 (t, $J$ = 7.2 Hz, 3H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -77.8 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 171.4, 168.4, 140.6, 136.4, 130.7, 128.9, 128.6, 128.5, 128.0, 127.4, 124.4 (q, $^{1}J_{C\text{-}F}$ = 284.0 Hz), 92.0, 73.9, 63.0 (q, $^{2}J_{C\text{-}F}$ = 28.3 Hz), 61.3, 60.9, 42.9, 30.2, 15.4, 14.1 ppm;

HRMS (ESI): $C_{24}H_{25}F_3NO_3^+$ $[M+H]^+$ Calcd 432.1781, Found 432.1782.



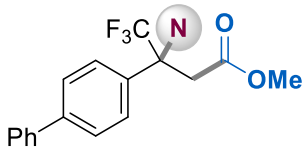

**4h**

**4h**: 67.9 mg, light yellow oil, yield: 81%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.67 – 7.69 (m, 2H), 7.28 – 7.37 (m, 7H), 7.14 – 7.21 (m, 7H), 7.05 – 7.08 (m, 2H), 6.58 – 6.61 (m, 2H), 3.40 (s, 3H), 3.19 (s, 2H), ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.7, 167.8, 148.8, 141.9, 138.5, 130.0, 128.5, 128.0, 127.8, 127.7, 127.5, 127.4, 127.3, 126.3, 67.3, 51.4, 44.9 ppm;

HRMS (ESI): $C_{29}H_{26}NO_2^+$ $[M+H]^+$ Calcd 420.1958, Found 420.1957.




**4i**

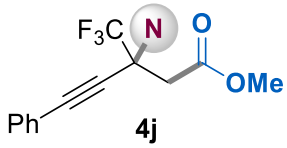
**4i**: 80.8 mg, light yellow oil, yield: 83%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.64 – 7.67 (m, 2H), 7.58 – 7.61 (m, 2H), 7.31 – 7.51 (m, 10H), 7.17 – 7.22 (m, 1H), 7.05 (t, $J$ = 7.6 Hz, 2H), 6.62 – 6.76 (m, 2H), 3.67 (s, 3H), 3.21 (d, $J$ = 14.6 Hz, 1H), 2.75 (d, $J$ = 14.6 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -76.9 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 170.7, 169.3, 141.3, 140.9, 140.2, 139.0, 137.7, 130.6, 128.9, 128.9, 128.4, 128.2, 128.1, 127.8, 127.7, 127.4, 127.1, 126.8, 126.4 (q, $^{1}J_{C\text{-}F}$ = 285.4 Hz), 67.9 (q, $^{2}J_{C\text{-}F}$ = 26.1 Hz), 52.0, 35.6 ppm;

HRMS (ESI): $C_{30}H_{25}F_3NO_2^+$ $[M+H]^+$ Calcd 488.1832, Found 488.1832.



**4j**

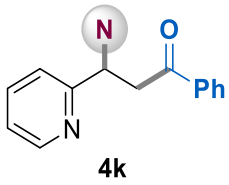
**4j**: 60.9 mg, light yellow oil, yield: 70%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.57 – 7.60 (m, 2H), 7.27 – 7.41 (m, 8H), 7.17 – 7.24 (m, 3H), 7.02 – 7.05 (m, 2H), 3.69 (s, 3H), 3.31 (d, $J$ = 14.4 Hz, 1H), 3.08 (d, $J$ = 14.4 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -77.6 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 172.0, 168.5, 140.6, 136.2, 131.8, 130.8, 129.0, 128.7, 128.6, 128.5, 128.1, 127.8, 127.5, 124.4 (q, $^1J_{C\text{-}F}$ = 284.2 Hz), 121.9, 91.4, 82.5, 63.4 (q, $^2J_{C\text{-}F}$ = 28.2 Hz), 52.0, 42.5 ppm;

HRMS (ESI): $C_{26}H_{21}F_3NO_2^+$ $[M+H]^+$ Calcd 436.1519, Found 436.1520.



**4k**

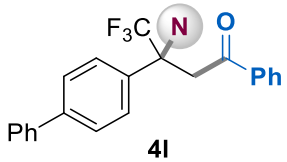
**4k**: 52.4 mg, light yellow oil, yield: 67%;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 8.53 – 8.54 (m, 1H), 7.93 – 7.95 (m, 2H), 7.60 – 7.64 (m, 2H), 7.47 – 7.52 (m, 1H), 7.27 – 7.41 (m, 10H), 7.06 – 7.15 (m, 3H), 5.28 (dd, $J$ = 8.0, 4.8 Hz, 1H), 3.72 – 3.83 (m, 2H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -77.6 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 198.4, 162.4, 149.2, 139.7, 137.3, 136.6, 136.6, 132.9, 130.2, 128.9, 128.6, 128.5, 128.4, 128.3, 128.1, 128.0, 127.7, 122.1, 122.0, 64.3, 46.4 ppm;

HRMS (ESI): $C_{27}H_{23}F_3N_2O^+$ $[M+H]^+$ Calcd 391.1805, Found 391.1806.



**4l**

**4l**: 87.4 mg, white solid, yield: 82%;

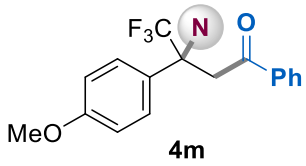
**m.p.** 145 – 146 ℃;

**$^1$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.80 – 7.82 (m, 2H), 7.71 – 7.74 (m, 2H), 7.60 – 7.63 (m, 2H), 7.53 – 7.57 (m, 4H), 7.32 – 7.48 (m, 9H), 7.08 – 7.12 (m, 1H), 6.93 (t, $J$ = 7.6 Hz, 2H), 6.65 – 6.76 (m, 2H), 4.11 (d, $J$ = 16.6 Hz, 1H), 3.15 (d, $J$ = 16.6 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -75.4 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 194.3, 169.6, 141.1, 141.0, 140.3, 140.1, 138.0, 137.7, 133.0, 130.6, 129.7, 129.4, 129.0, 128.9, 128.7, 128.5, 128.4, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.5, 127.2, 126.9, 126.5 (q, $^{1}J_{C\text{-}F}$ = 284.9 Hz), 68.4 (q, $^{2}J_{C\text{-}F}$ = 25.9 Hz), 38.6 ppm;

HRMS (ESI): $C_{35}H_{27}F_3NO^+$ $[M+H]^+$ Calcd 534.2039, Found 534.2037.



**4m**

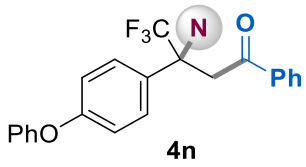
**4m**: 74.0 mg, light yellow oil, yield: 76%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.76 – 7.79 (m, 2H), 7.68 – 7.71 (m, 2H), 7.49 – 7.57 (m, 1H), 7.32 – 7.44 (m, 7H), 7.09 – 7.13 (m, 1H), 6.93 – 6.97 (m, 2H), 6.83 – 6.87 (m, 2H), 6.62 – 6.73 (m, 2H), 4.05 (d, *J* = 16.6 Hz, 1H), 3.83 (s,3H), 3.02 (d, *J* = 16.6 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) *δ* -75.9 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 194.3, 169.2, 159.5, 141.1, 138.1, 137.7, 133.3, 132.9, 130.4, 129.3, 129.1, 128.9, 128.5, 128.5, 128.4, 128.1, 128.0, 127.9, 127.8, 127.4, 126.4 (q, $^{1}J_{C\text{-}F}$ = 284.7 Hz), 113.4, 68.0 (q, $^{2}J_{C\text{-}F}$ = 25.8 Hz), 55.3, 38.4 ppm;

HRMS (ESI): $C_{30}H_{25}F_3NO_2^+$ $[M+H]^+$ Calcd 488.1832, Found 488.1833.



**4n**

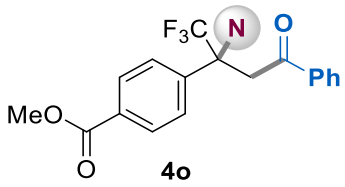
**4n**: 85.6 mg, light yellow oil, yield: 78%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) *δ* 7.77 – 7.79 (m, 2H), 7.68 – 7.71 (m, 2H), 7.50 – 7.54 (m, 1H), 7.31 – 7.44 (m, 9H), 7.11 – 7.16 (m, 2H), 6.93 – 7.07 (m, 6H), 6.65 – 6.79 (m, 2H), 4.05 (d, *J* = 16.6 Hz, 1H), 3.83 (s,3H), 3.02 (d, *J* = 16.6 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) *δ* -75.7 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 194.3, 169.5, 157.4, 156.7, 141.0, 138.1, 137.6, 135.6, 133.0, 130.5, 129.9, 129.5, 128.9, 128.5, 128.2, 128.1, 128.0, 127.8, 127.5, 126.5 (q, $^{1}J_{C\text{-}F}$ = 284.8 Hz), 123.7, 119.2, 118.2, 68.2 (q, $^{2}J_{C\text{-}F}$ = 25.8 Hz), 38.5 ppm;

HRMS (ESI): $C_{35}H_{27}F_3NO_2^+$ $[M+H]^+$ Calcd 550.1988, Found 550.1990.




**4o**

**4o**: 81.4 mg, light yellow solid, yield: 79%;

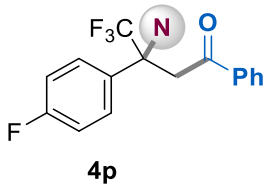
**m.p.** 155 – 156 ℃;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.99 – 8.02 (m, 2H), 7.73 – 7.76 (m, 2H), 7.69 – 7.72 (m, 2H), 7.63 (d, $J$ = 8.2 Hz, 2H), 7.50 – 7.54 (m, 1H), 7.32 – 7.44 (m, 5H), 7.08 – 7.12 (m, 1H), 6.64 (t, $J$ = 7.6 Hz, 2H), 6.62 – 6.78 (m, 2H), 4.06 (d, $J$ = 16.9 Hz, 1H), 3.93 (s,3H), 3.20 (d, $J$ = 16.9 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -75.2 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 193.9, 169.8, 166.7, 145.9, 140.9, 137.8, 137.4, 133.1, 130.7, 130.0, 129.4, 129.0, 128.5, 128.3, 128.1, 127.9, 127.8, 127.7, 126.1 (q, $^{1}J_{C\text{-}F}$ = 285.7 Hz), 68.6 (q, $^{2}J_{C\text{-}F}$ = 25.9 Hz), 52.3, 38.7 ppm;

HRMS (ESI): $C_{31}H_{25}F_3NO_3^+$ $[M+H]^+$ Calcd 516.1781, Found 516.1775.




**4p**

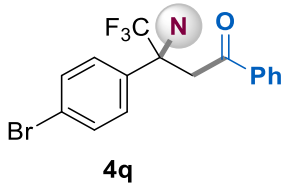
**4p**: 71.3 mg, light yellow oil, yield: 75%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.75 – 7.77 (m, 2H), 7.68 – 7.71 (m, 2H), 7.46 – 7.54 (m, 4H), 7.27 – 7.43 (m, 9H), 7.10 – 7.14 (m, 1H), 6.95 – 7.04 (m, 4H), 6.63 – 6.76 (m, 2H), 4.03 (d, $J$ = 16.7 Hz, 1H), 3.12 (d, $J$ = 16.7 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -75.7, -113.5 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 194.1, 169.6, 162.6 (d, $^{1}J_{C\text{-}F}$ = 248.6 Hz), 159.0, 140.9, 138.2, 137.9, 137.5, 136.9 (d, $^{4}J_{C\text{-}F}$ = 3.3 Hz), 135.6, 133.1, 130.6, 129.8 (d, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 130.0, 129.4, 129.0, 128.7, 128.5, 128.2, 128.1, 128.0, 127.9, 127.7, 127.6, 127.5, 126.2 (q, $^{1}J_{C\text{-}F}$ = 284.6 Hz), 115.2 (d, $^{2}J_{C\text{-}F}$ = 21.3 Hz), 68.1 (q, $^{2}J_{C\text{-}F}$ = 26.0 Hz), 52.3, 38.7 ppm;

HRMS (ESI): $C_{29}H_{22}F_4NO^+$ $[M+H]^+$ Calcd 476.1632, Found 476.1633.




**4q**

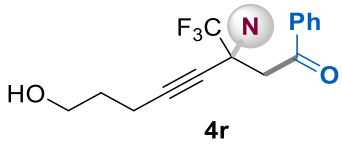
**4q**: 82.4 mg, light yellow oil, yield: 77%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.72 – 7.74 (m, 2H), 7.67 – 7.69 (m, 2H), 7.26 – 7.54 (m, 10H), 7.10 – 7.14 (m, 1H), 6.98 (t, $J$ = 7.6 Hz, 1H), 6.62 – 6.78 (m, 2H), 3.99 (d, $J$ = 16.9 Hz, 1H), 3.15 (d, $J$ = 16.9 Hz, 1H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -75.4 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 193.9, 169.7, 159.1, 140.9, 140.1, 138.2, 137.8, 137.4, 135.5, 133.1, 131.4, 130.7, 129.7, 129.4, 129.0, 128.8, 128.5, 128.3, 128.1, 127.9, 127.6, 126.2 (q, $^{1}J_{C\text{-}F}$ = 284.6 Hz), 122.7, 68.3 (q, $^{2}J_{C\text{-}F}$ = 25.6 Hz), 38.6 ppm;

HRMS (ESI): $C_{29}H_{22}BrF_3NO^+$ $[M+H]^+$ Calcd 536.0831, Found 536.083.




**4r**

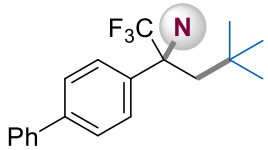
**4r**: 59.3 mg, light yellow oil, yield: 64%;

**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.93 – 7.94 (m, 2H), 7.48 – 7.53 (m, 3H), 7.33 – 7.41 (m, 6H), 7.25 – 7.28 (m, 4H), 3.99 (d, $J$ = 14.9 Hz, 1H), 3.53 (d, $J$ = 14.9 Hz, 1H), 3.39 – 3.49 (m, 2H), 1.68 – 1.77 (m, 2H), 1.31 – 1.36 (m, 2H) ppm;

**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -77.6 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 194.9, 171.1, 140.6, 137.3, 136.3, 133.3, 132.5, 130.6, 130.1, 128.9, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 127.9, 127.2, 124.5 (q, $^{1}J_{C\text{-}F}$ = 284.2 Hz), 92.8, 63.6 (q, $^{2}J_{C\text{-}F}$ = 27.4 Hz), 61.4, 45.4, 30.1, 15.4 ppm;

HRMS (ESI): $C_{28}H_{24}F_3NNaO_2^+$ $[M+Na]^+$ Calcd 486.1651, Found 486.1651.




**4s**

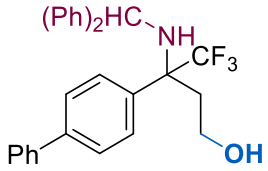
**4s**: 87.2 mg, light yellow oil, yield: 85%;

**$^{1}$H NMR** (600 MHz, Chloroform-d) $\delta$ 7.58 – 7.63 (m, 4H), 7.43 – 7.62 (m, 6H), 7.31 – 7.40 (m, 4H), 7.14 – 7.17 (m, 1H), 7.03 (t, $J$ = 7.6 Hz, 2H), 6.62 (t, $J$ = 7.5 Hz, 2H), 2.38 (d, $J$ = 15.0 Hz, 1H), 2.08 (dd, $J$ = 15.0, 2.0 Hz, 1H) ppm;

**$^{19}$F NMR** (565 MHz, Chloroform-*d*) $\delta$ -71.8 ppm;

**$^{13}$C NMR** (150 MHz, Chloroform-*d*) δ 167.4, 141.4, 141.3, 140.6, 140.4, 137.7, 130.3, 129.4, 128.9, 128.8, 128.1, 128.0, 127.8, 127.5, 127.1, 126.3, 69.4 (q, $^{2}J_{C\text{-}F}$ = 26.5 Hz), 44.8, 31.9, 31.4 ppm;

HRMS (ESI): $C_{32}H_{31}F_3N^+$ $[M+H]^+$ Calcd 486.2403, Found 486.2464.




**5**

**5**: 79.3 mg, light yellow oil, yield: 86%;

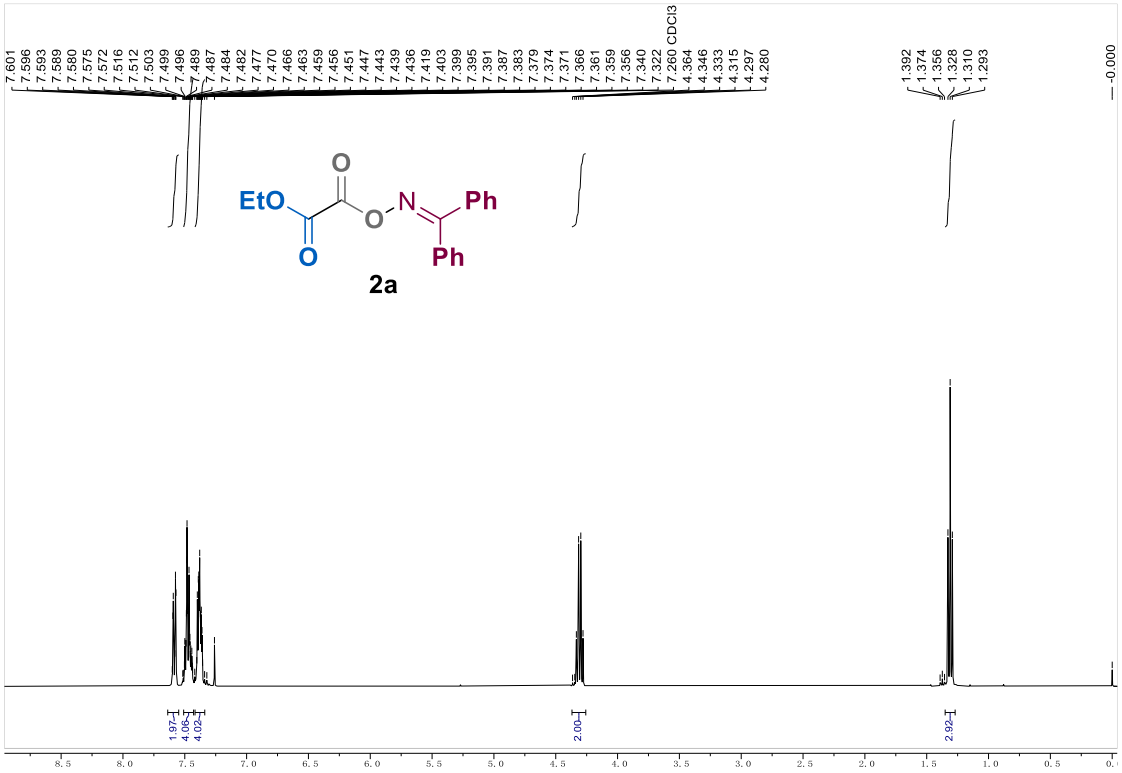
**$^{1}$H NMR** (400 MHz, Chloroform-d) $\delta$ 7.51 – 7.58 (m, 6H), 7.41 – 7.44 (m, 2H), 7.32 – 7.38 (m, 5H), 7.25 – 7.29 (m, 4H), 7.16 – 7.21 (m, 2H), 5.02(s, 1H), 3.39 – 3.52 (m, 2H), 2.32 – 2.39 (m, 1H), 2.18 – 2.24 (m, 1H) ppm;

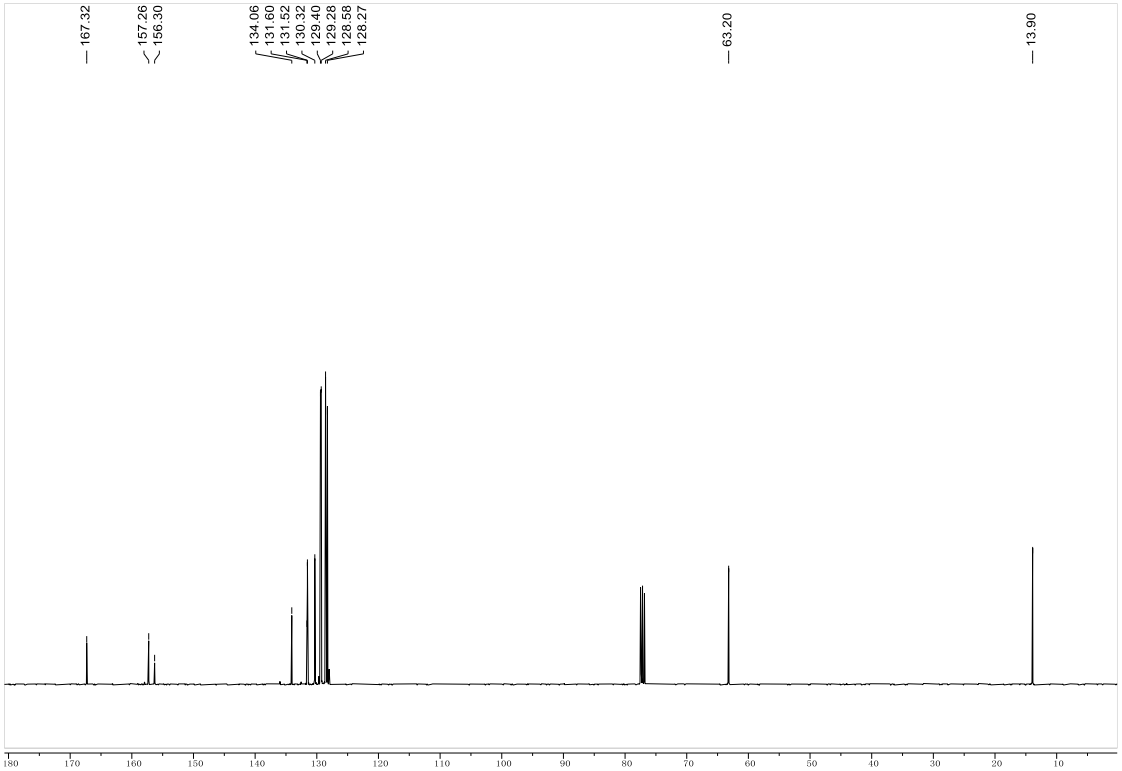
**$^{19}$F NMR** (375 MHz, Chloroform-*d*) $\delta$ -71.0 ppm;

**$^{13}$C NMR** (100 MHz, Chloroform-*d*) δ 145.6, 144.9, 141.0, 140.2, 135.5, 128.9, 128.8, 128.7, 128.1, 127.7, 127.1, 127.05, 127.03, 127.0 (q, $^{1}J_{C\text{-}F}$ = 289.5 Hz), 126.9, 66.9 (q, $^{2}J_{C\text{-}F}$ = 24.9 Hz), 61.7, 58.7, 37.6 ppm;

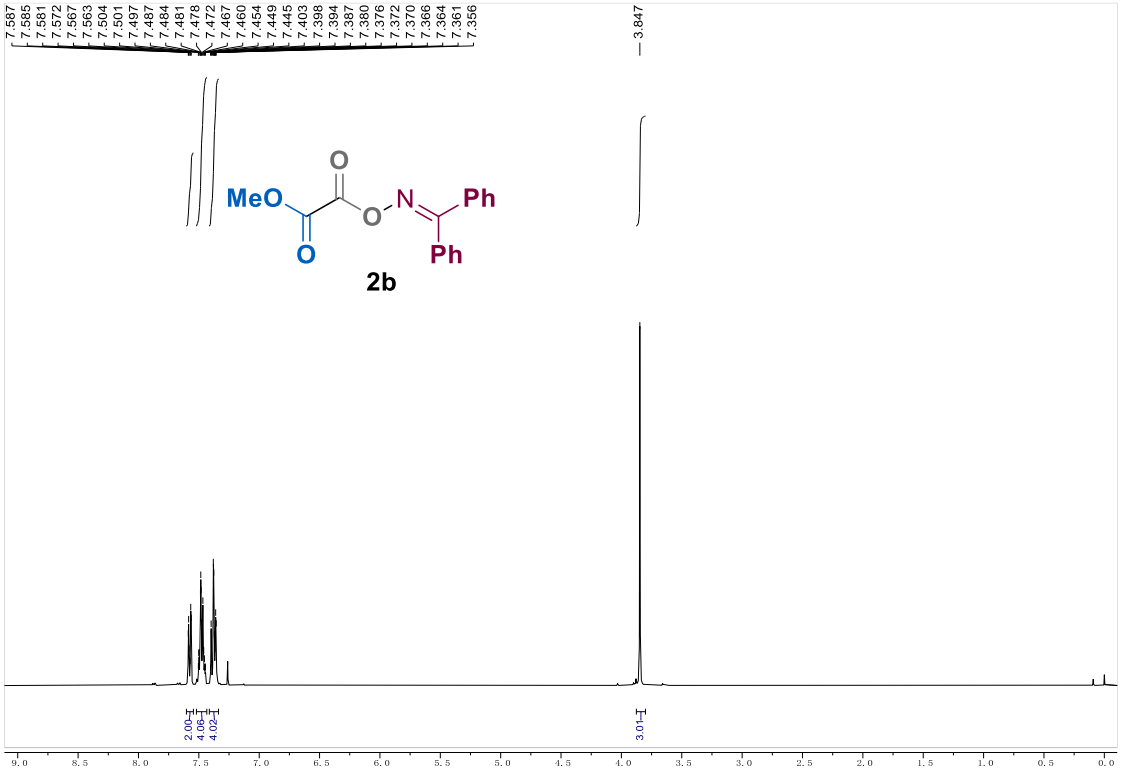
HRMS (ESI): $C_{29}H_{26}F_3NNaO_2^+$ $[M+Na]^+$ Calcd 484.1859, Found 484.1858.

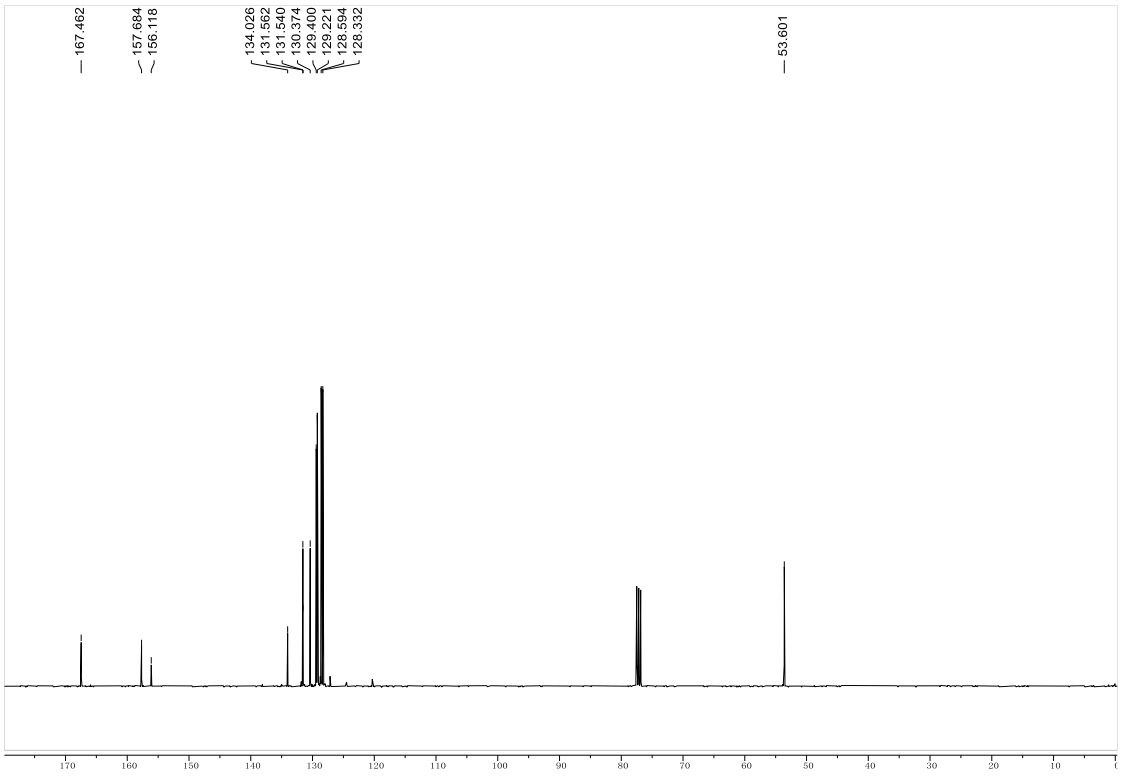
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 2a**



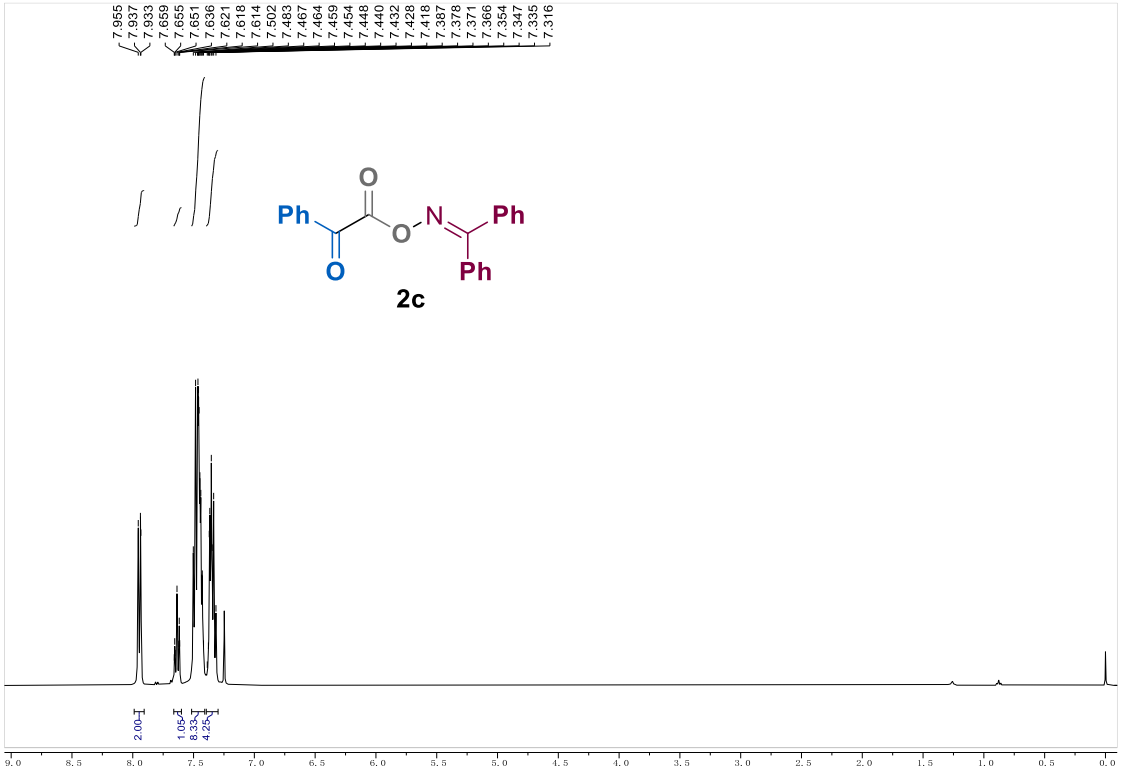



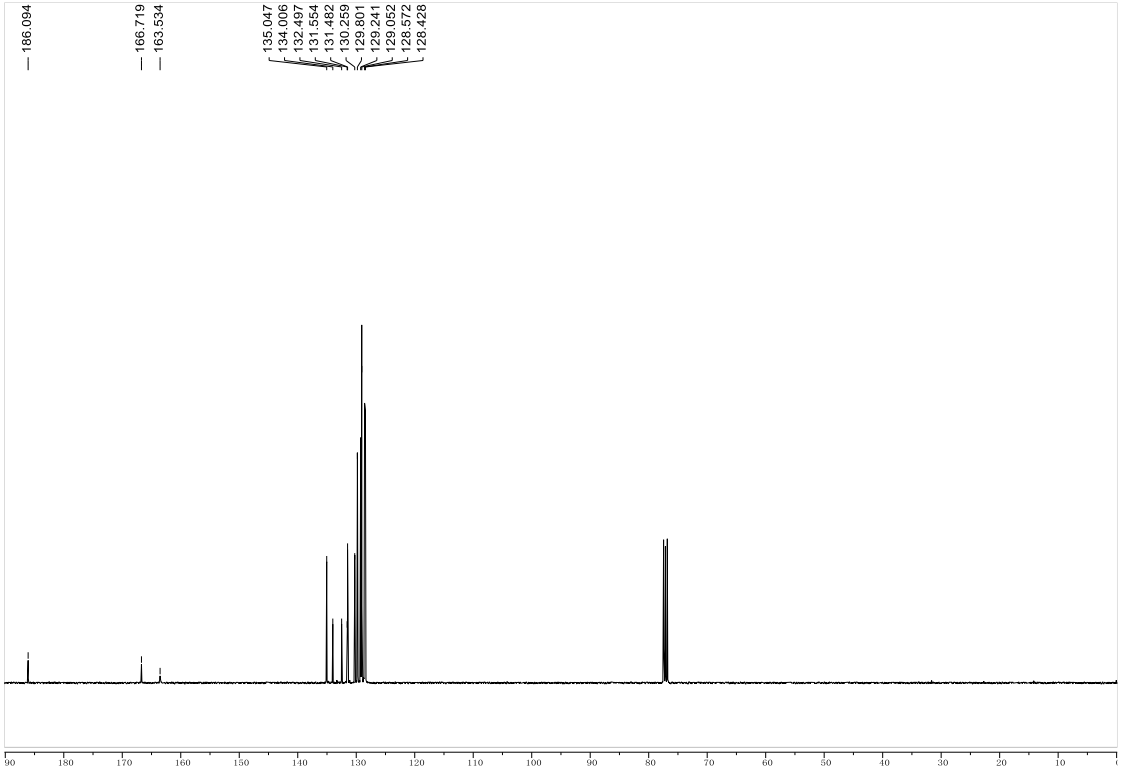
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 2b**



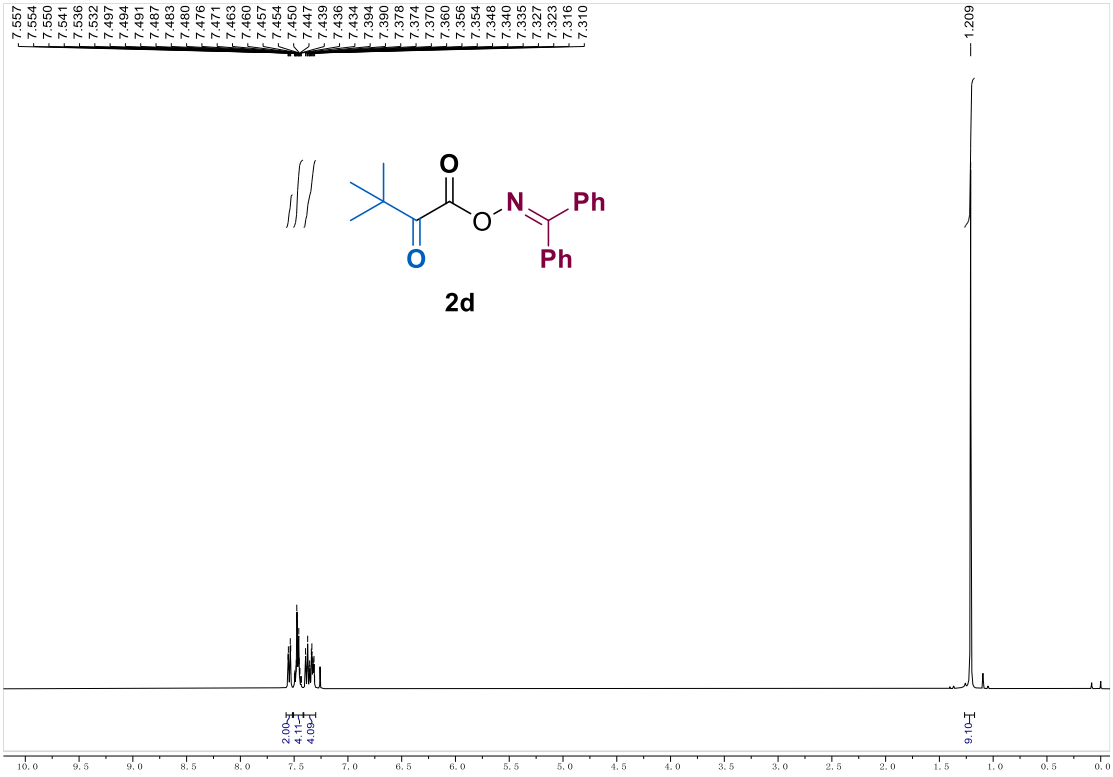



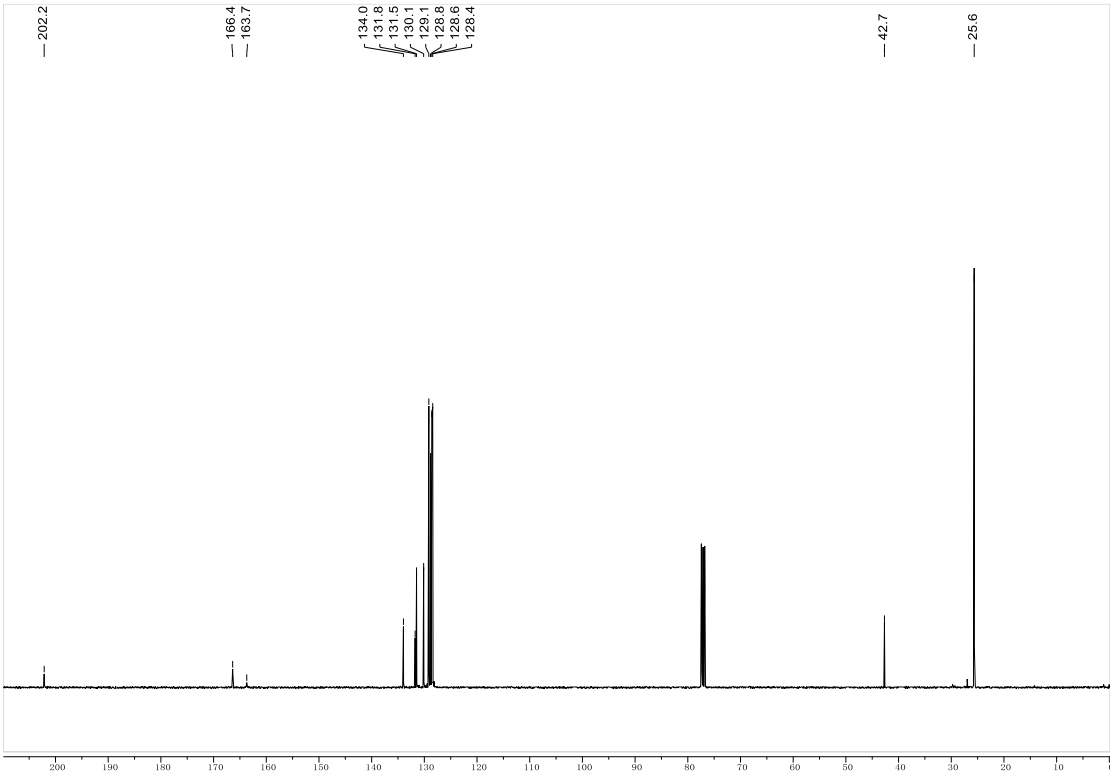
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 2c**



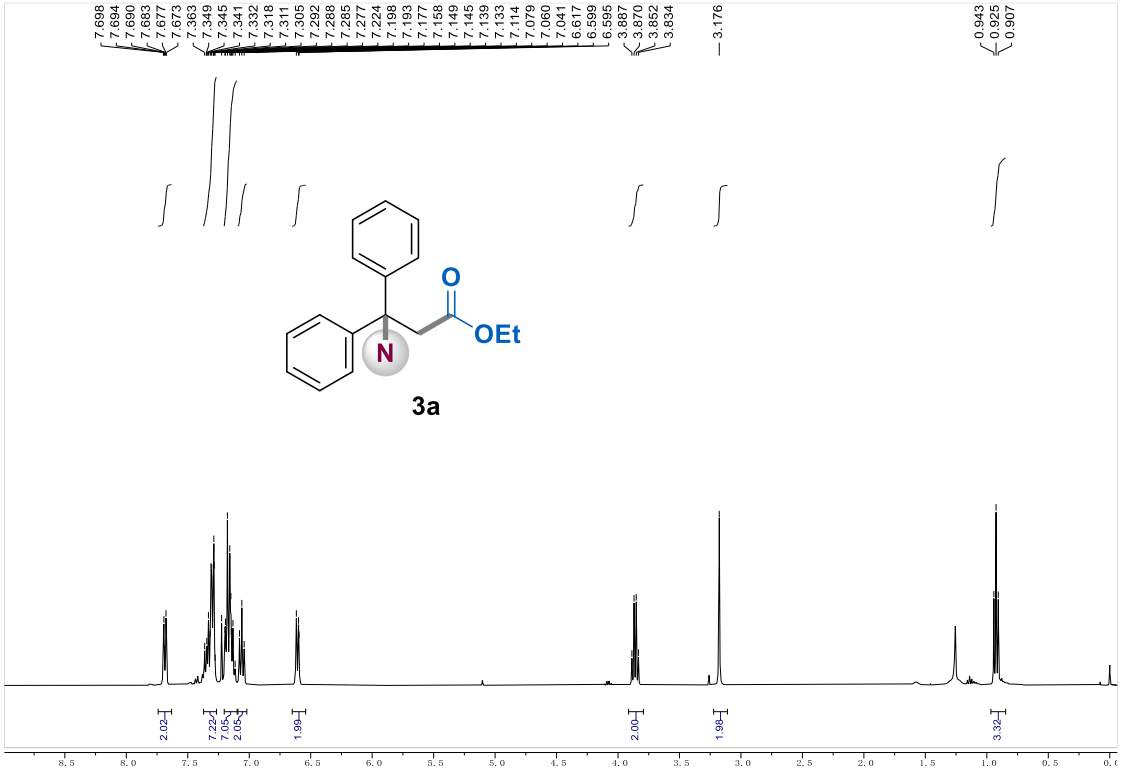



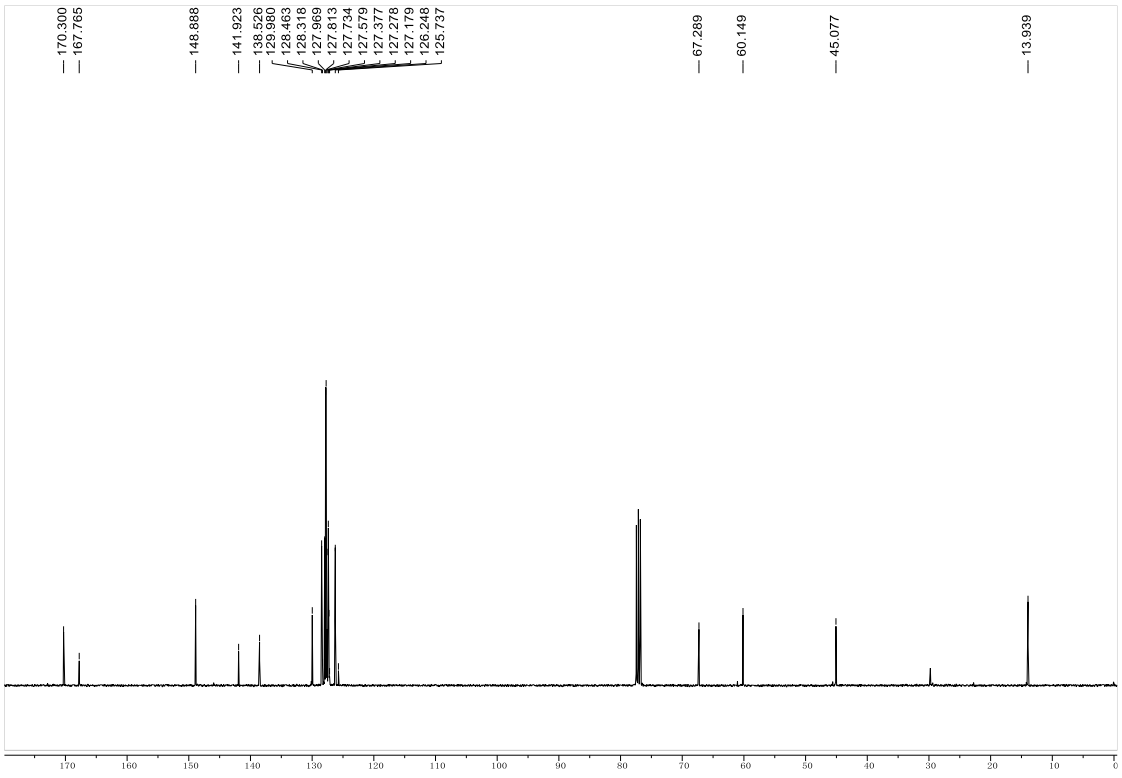
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 2d**



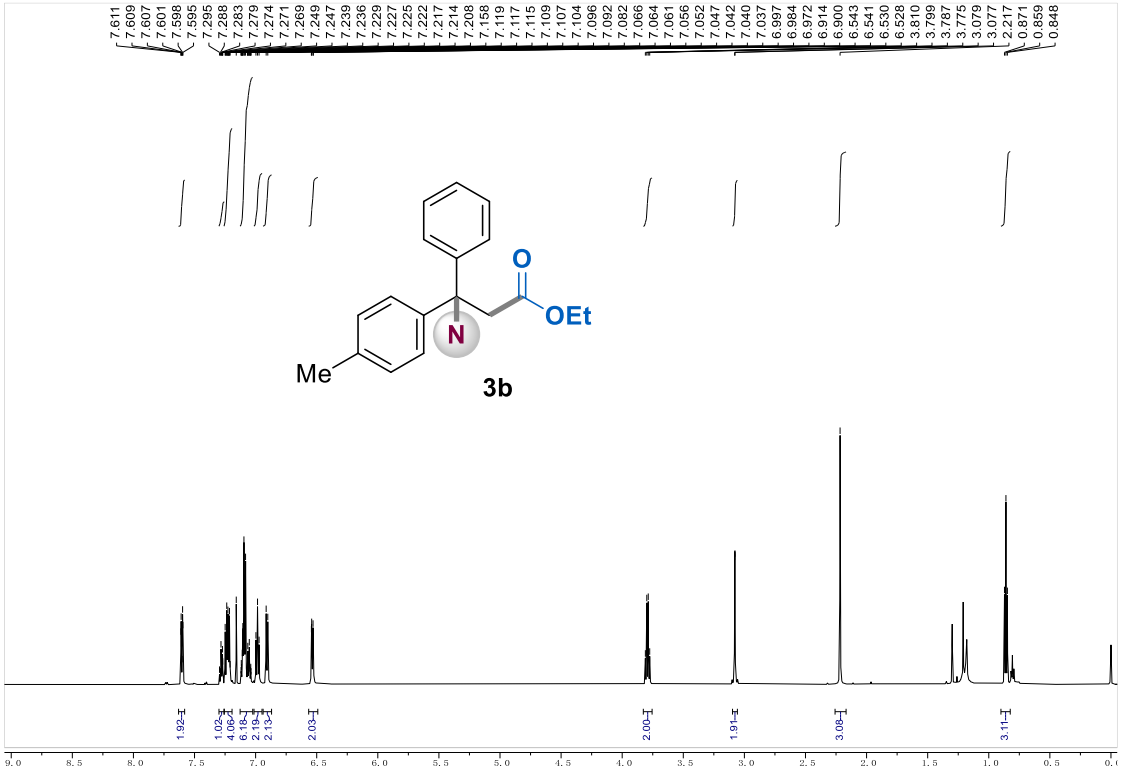



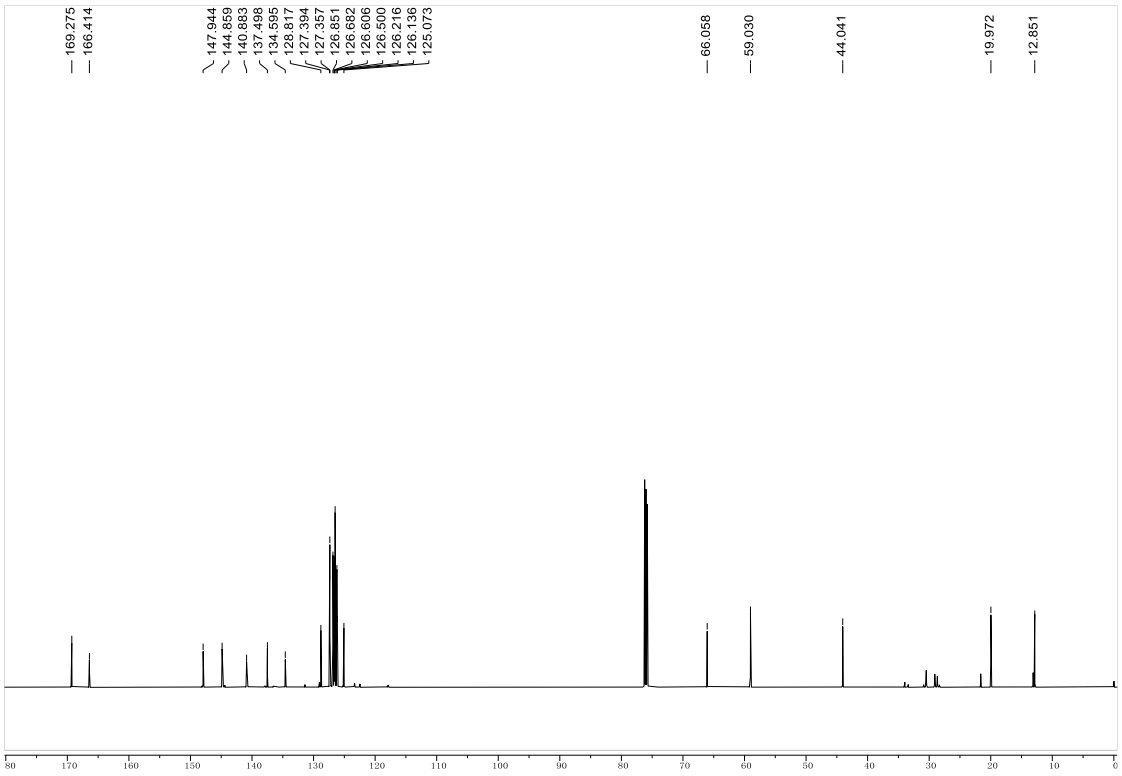
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3a**



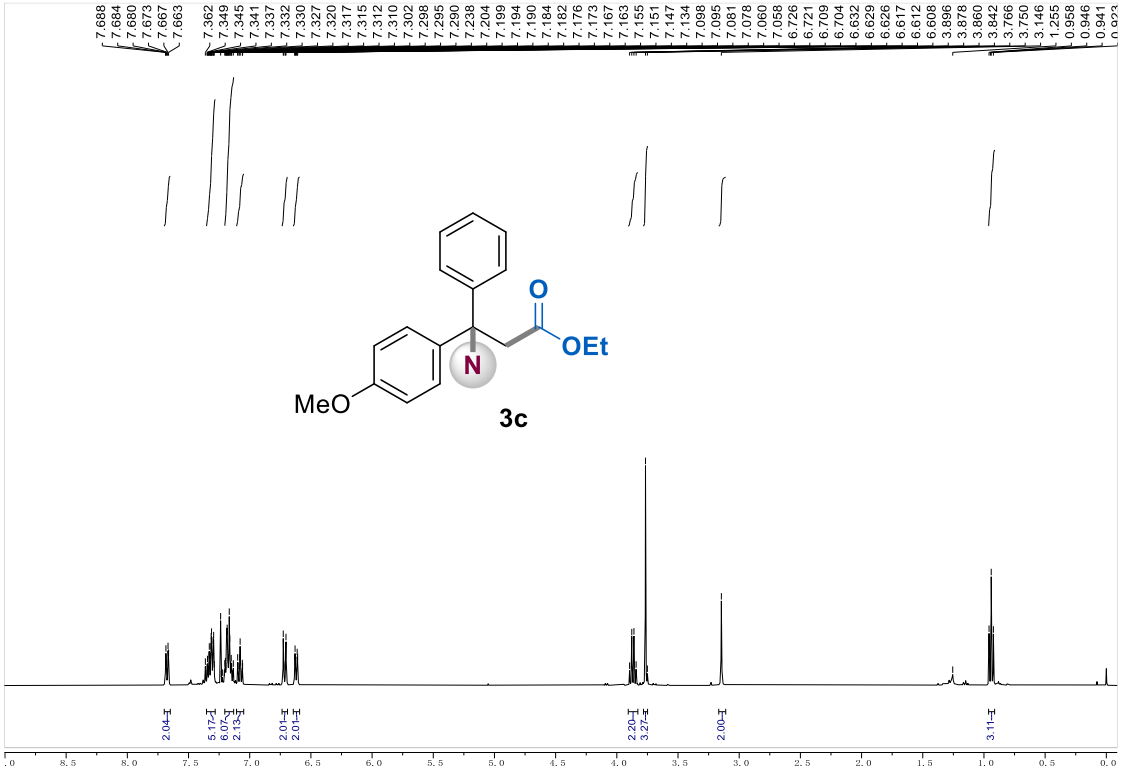



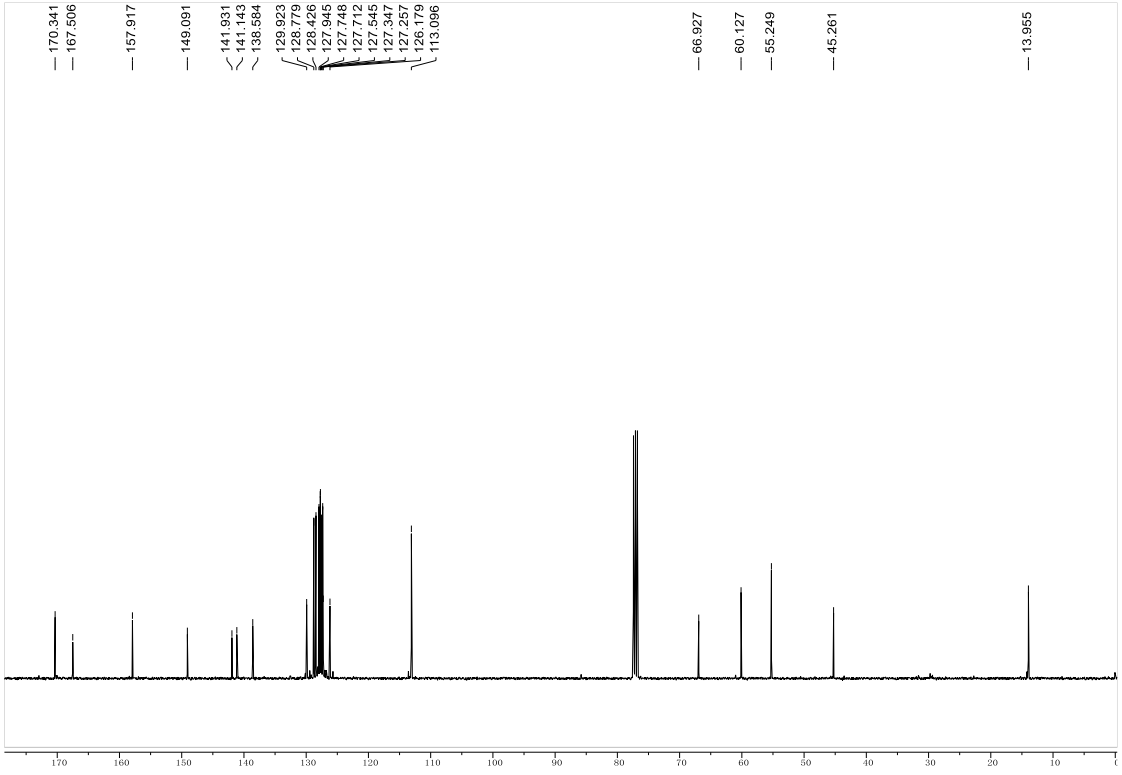
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3b**



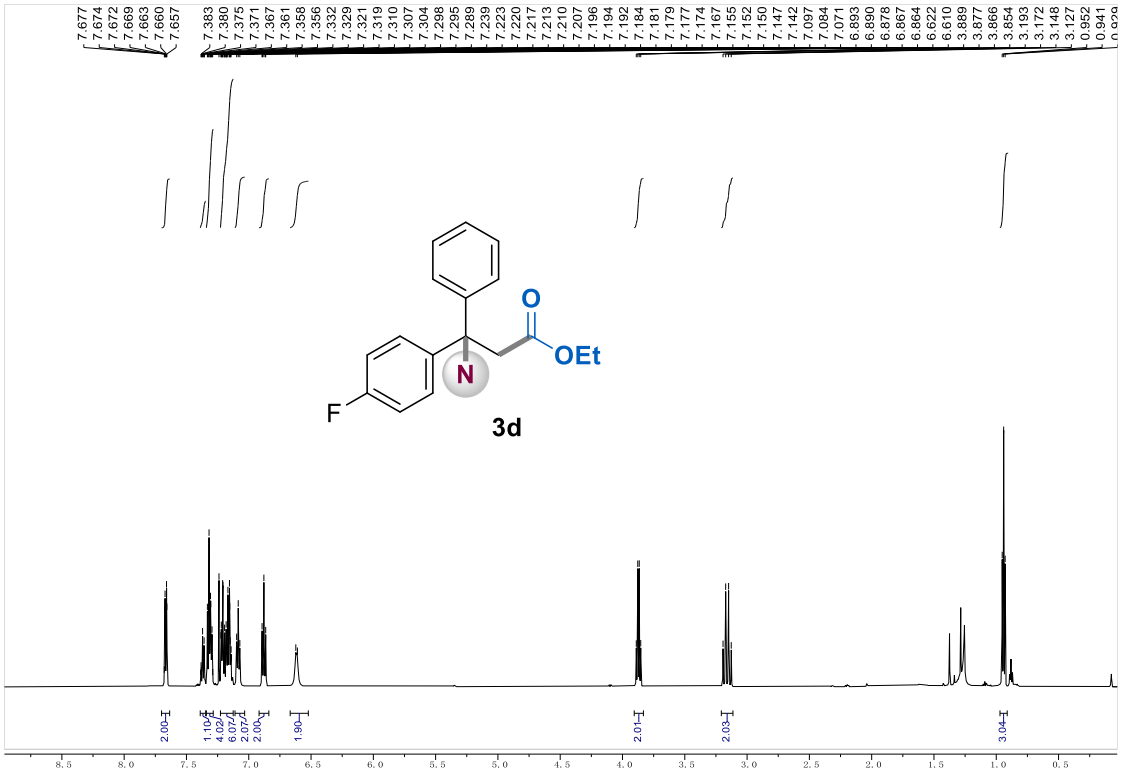



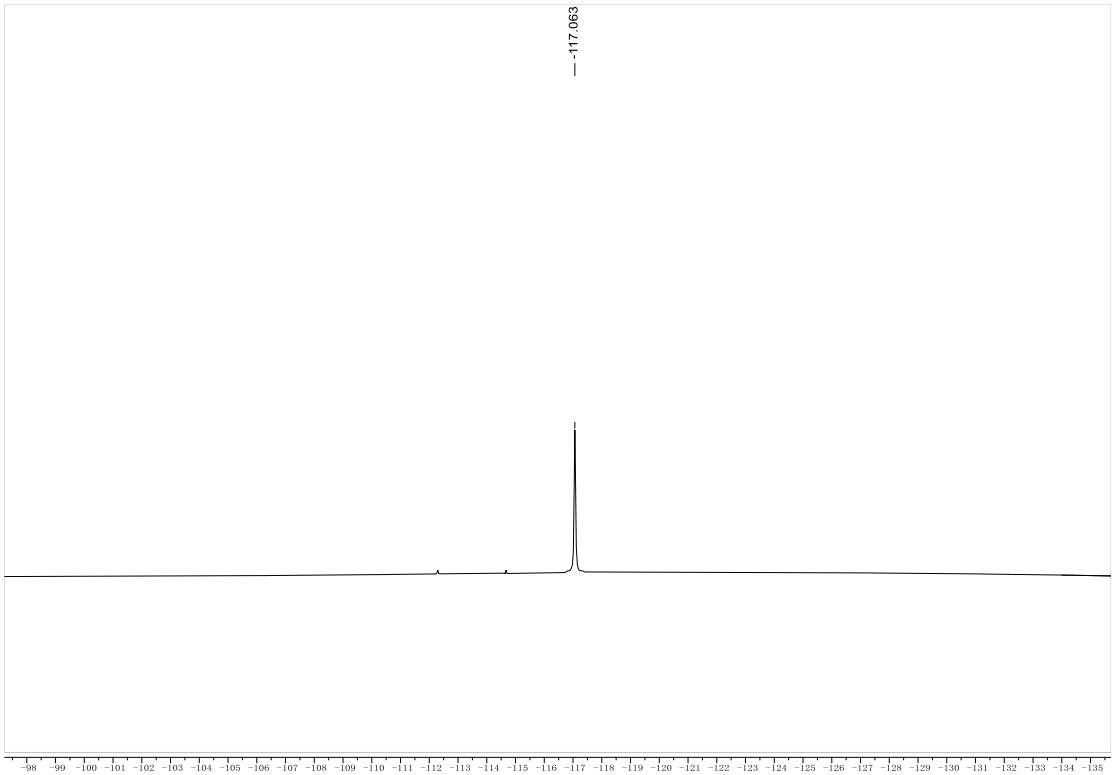
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3c**

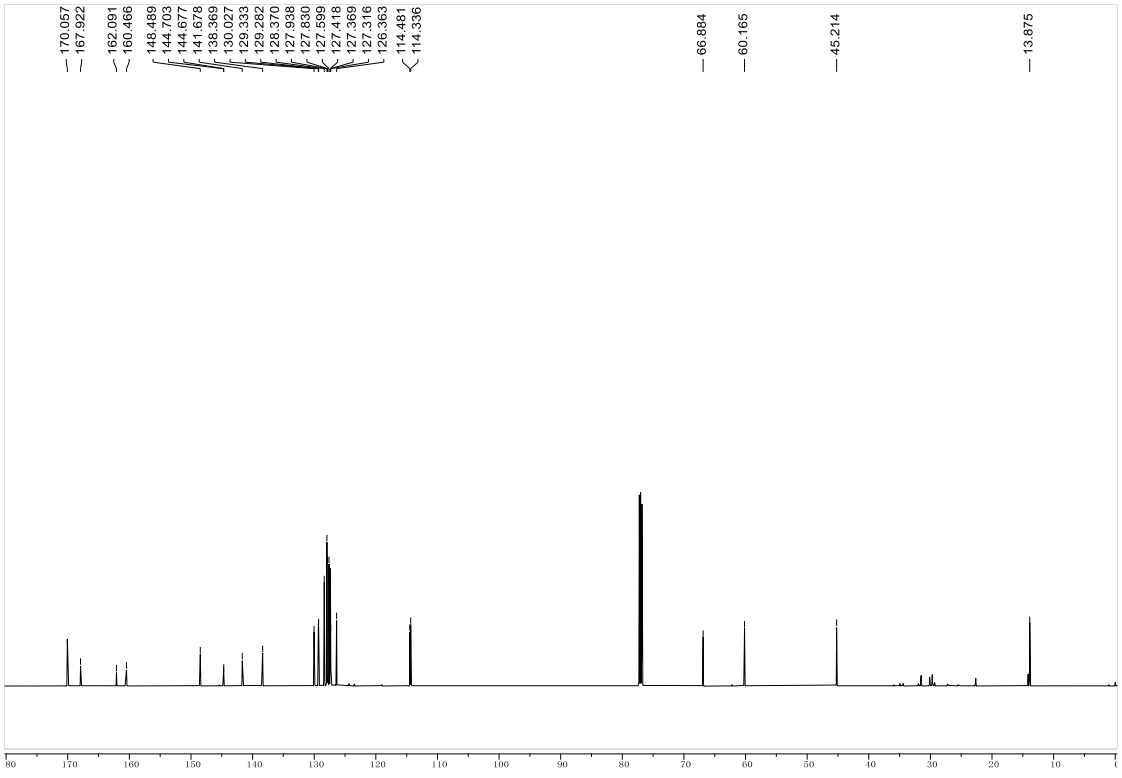


**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3d**

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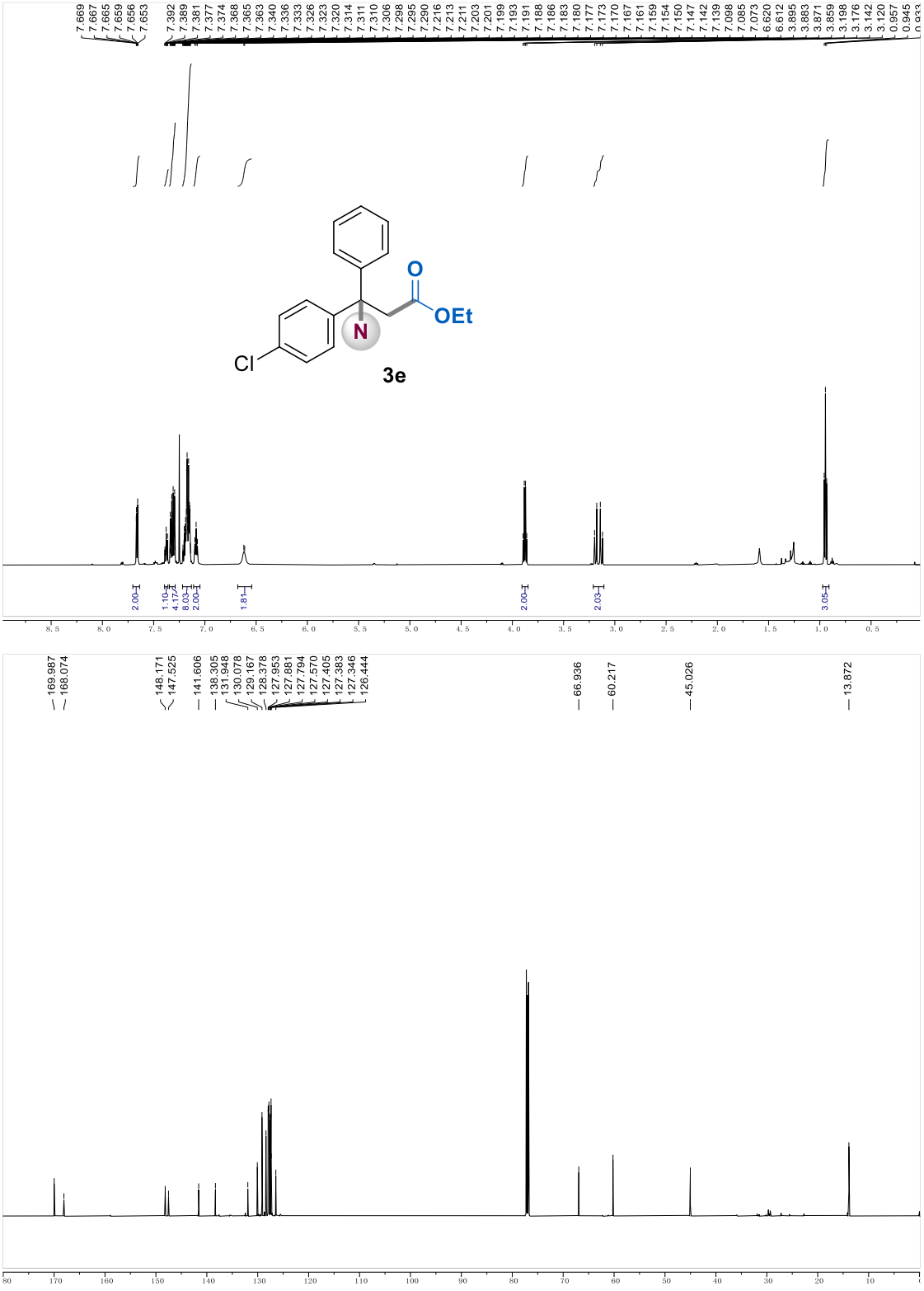
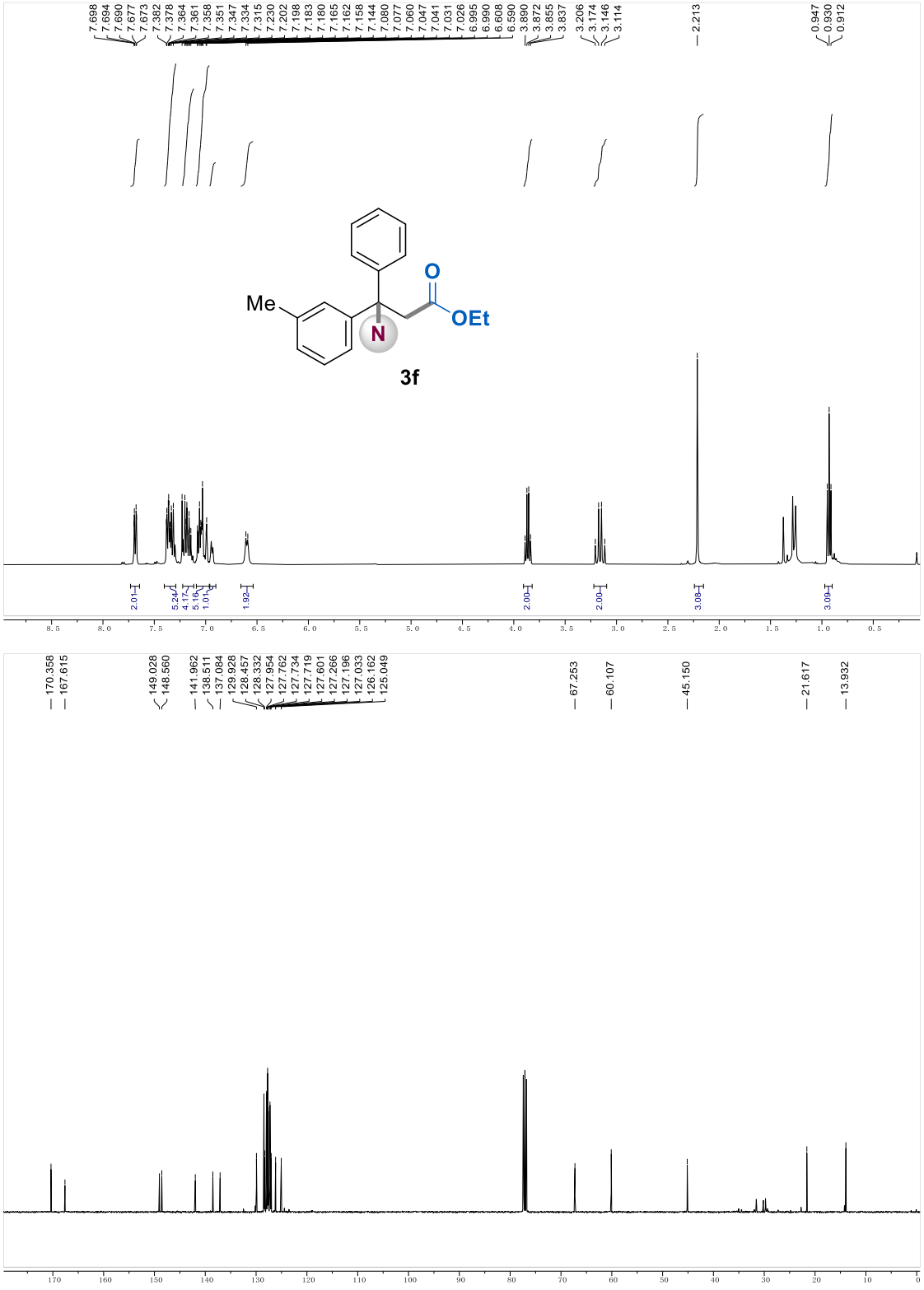
167.922
162.091
160.466
148.489
144.703
144.677
141.678
138.369
130.027
129.333
129.282
128.370
127.938
127.830
127.599
127.418
127.369
127.316
126.363
114.481
114.336
66.884
60.165
45.214
13.875
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

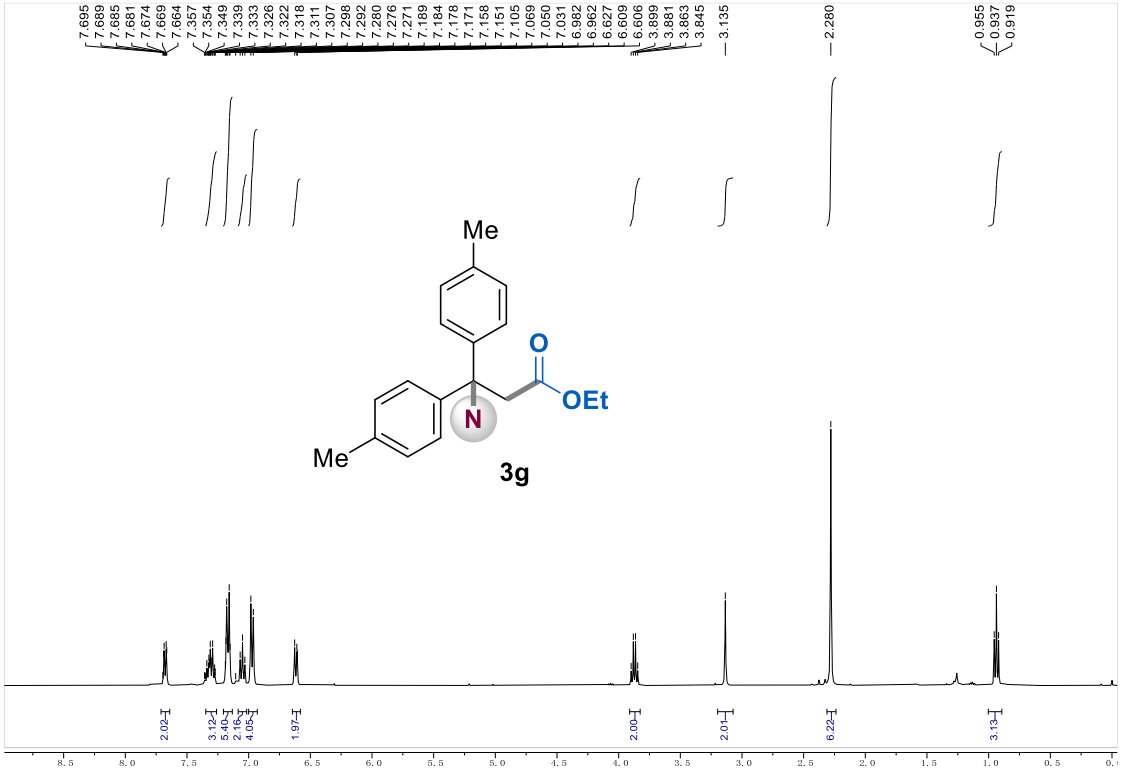
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3e**

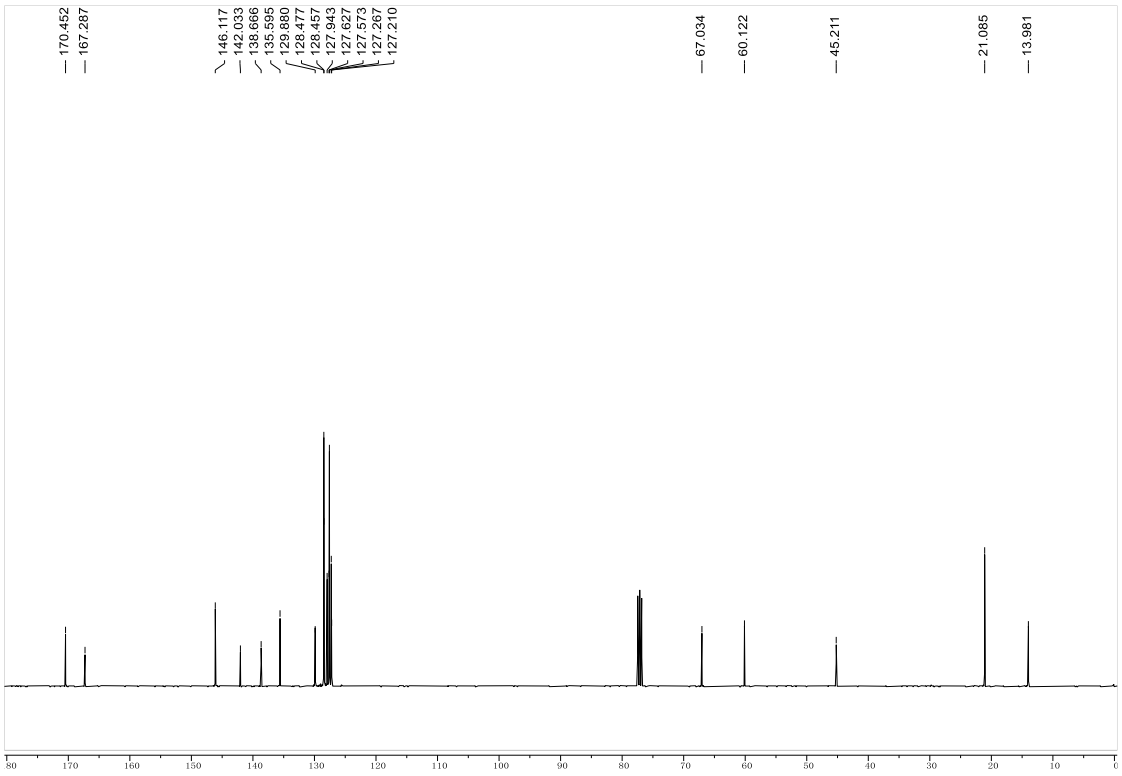


**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3f**

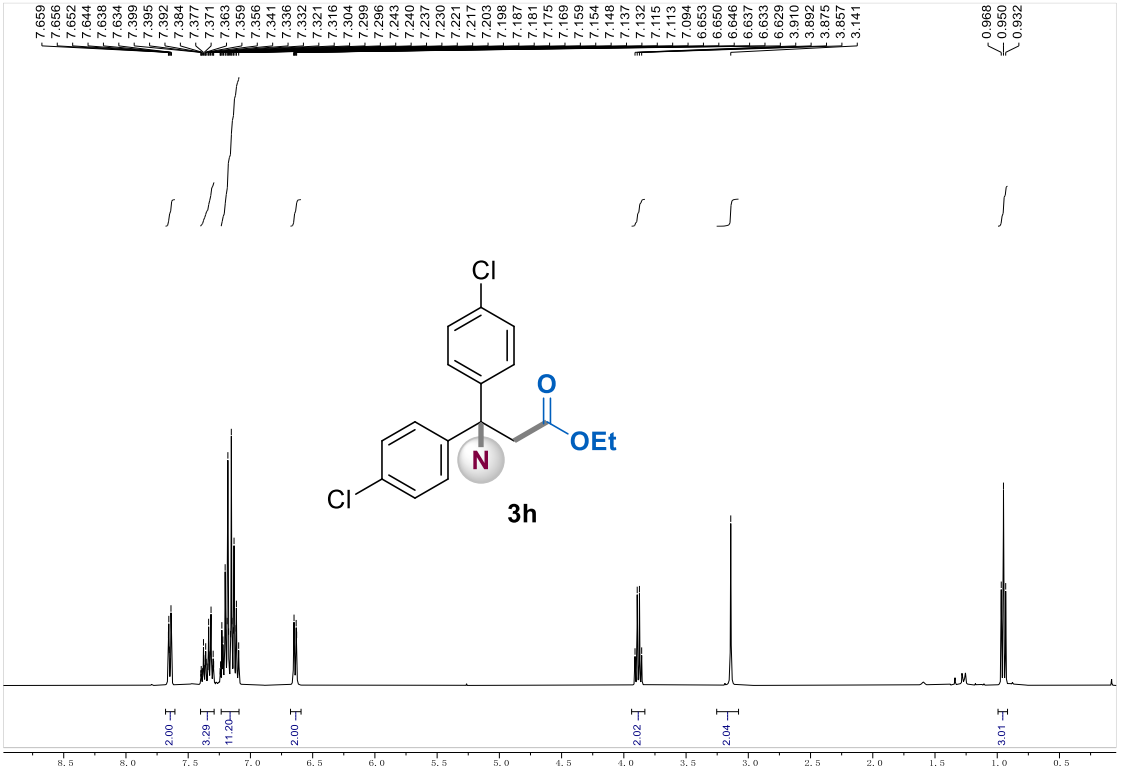


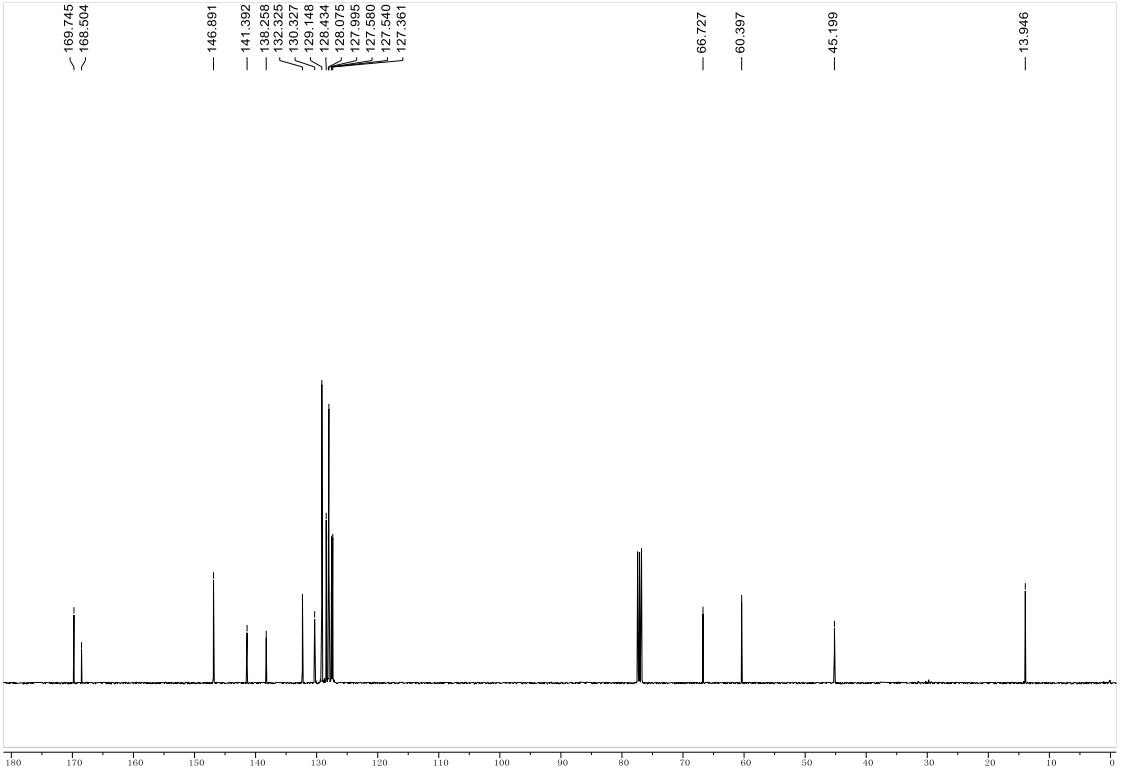
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3g**



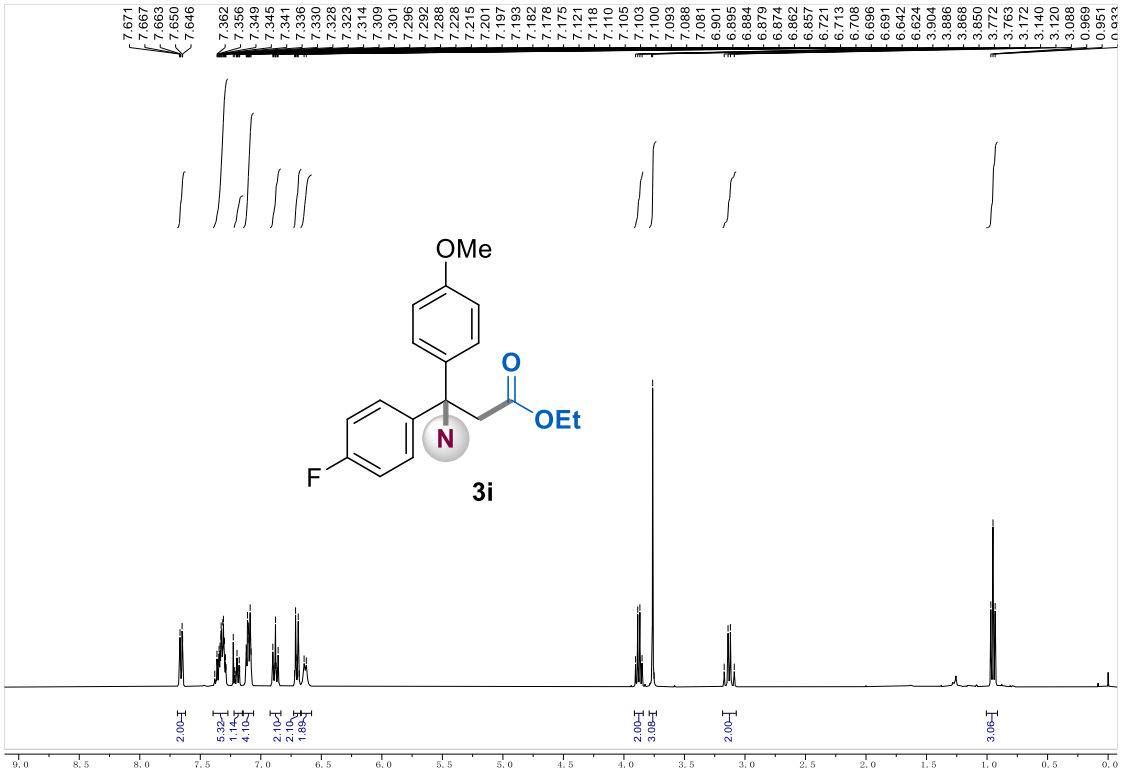



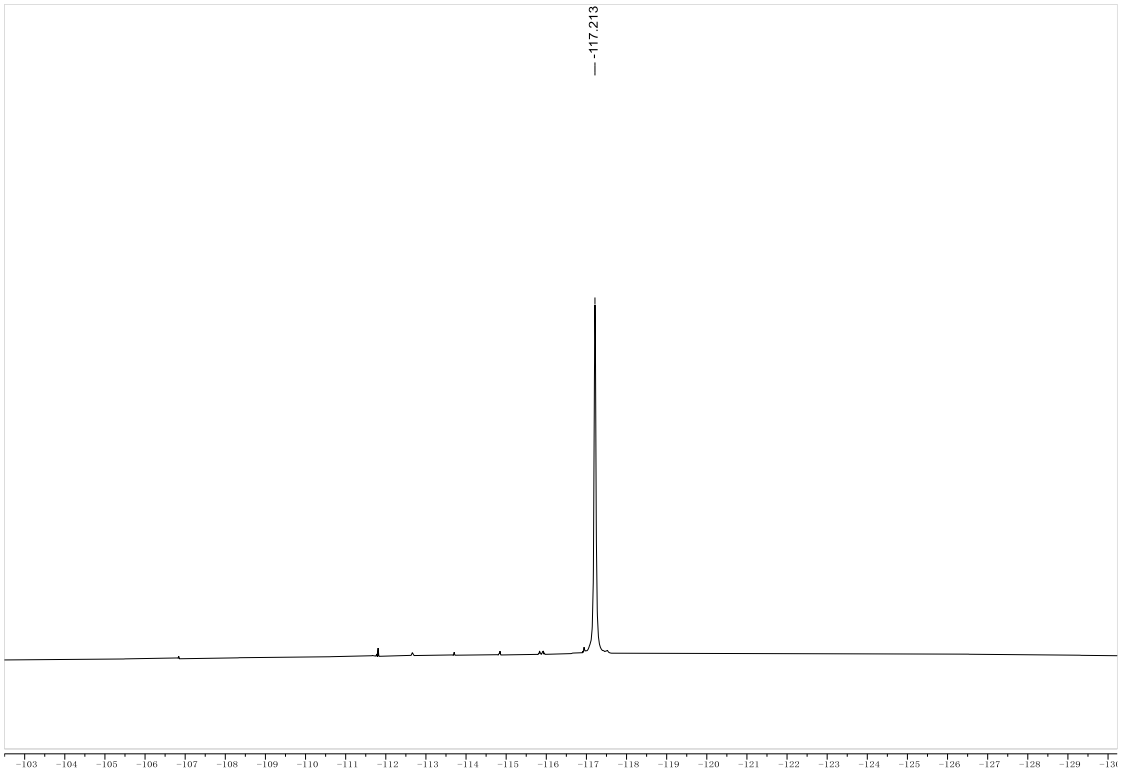
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3h**

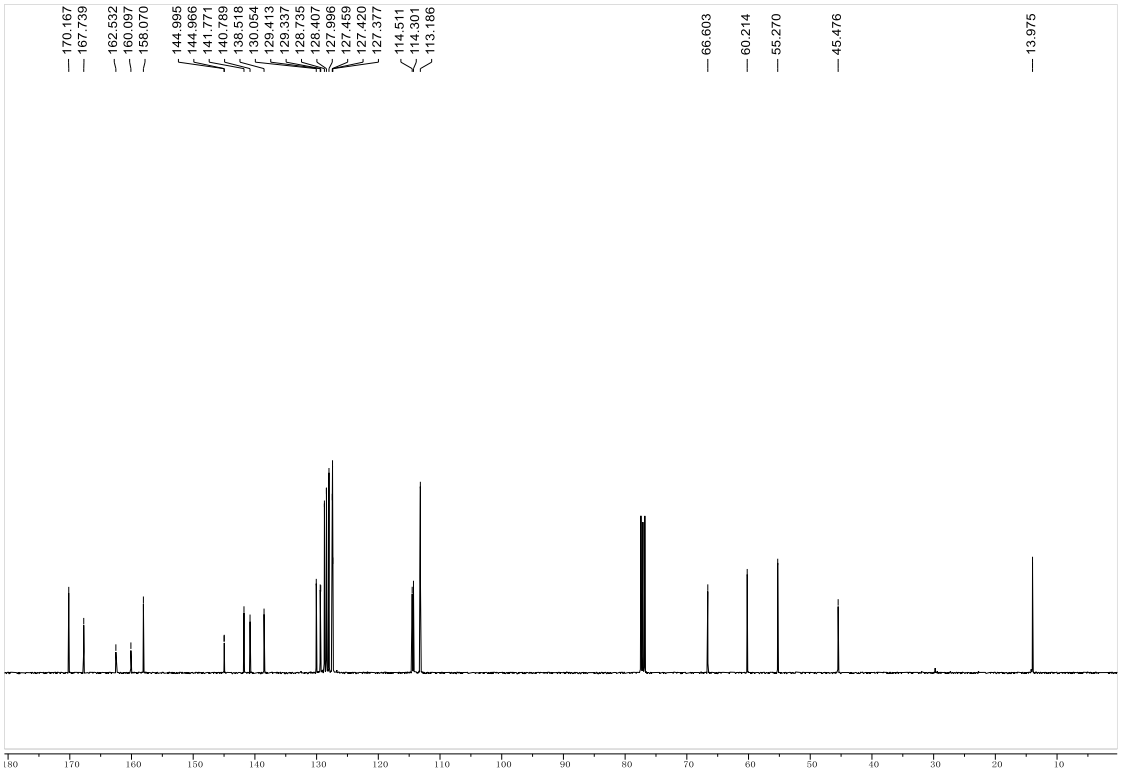


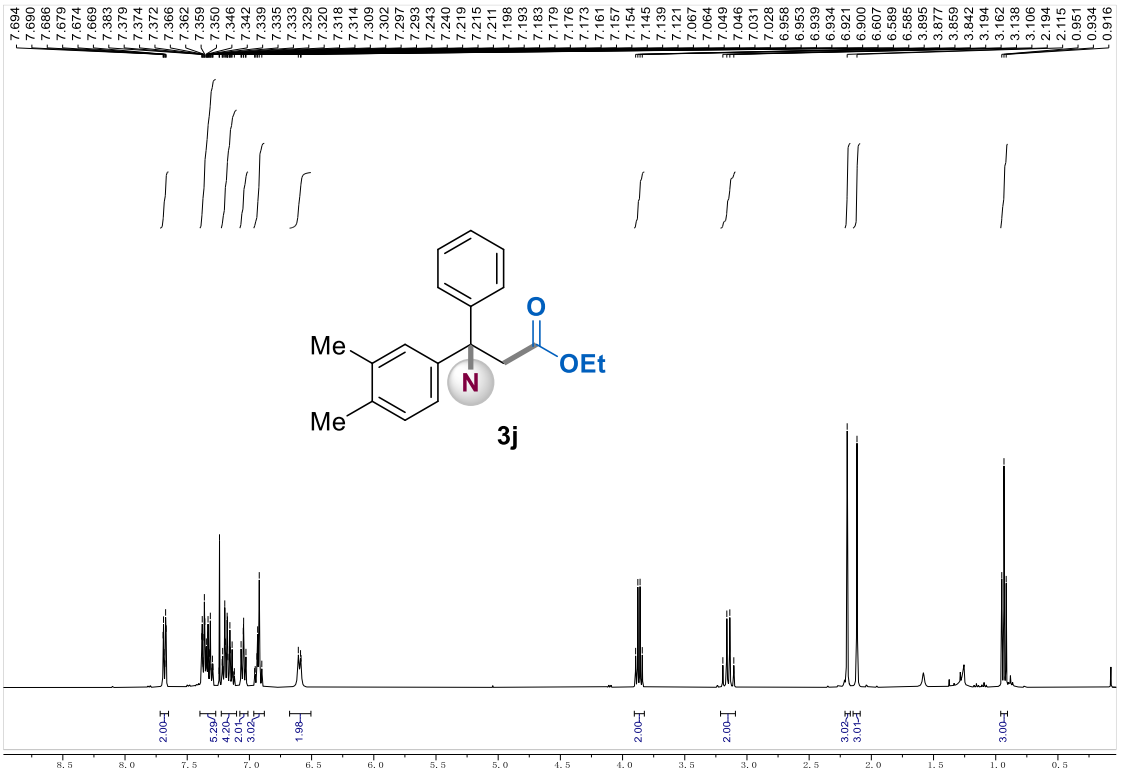
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3i**

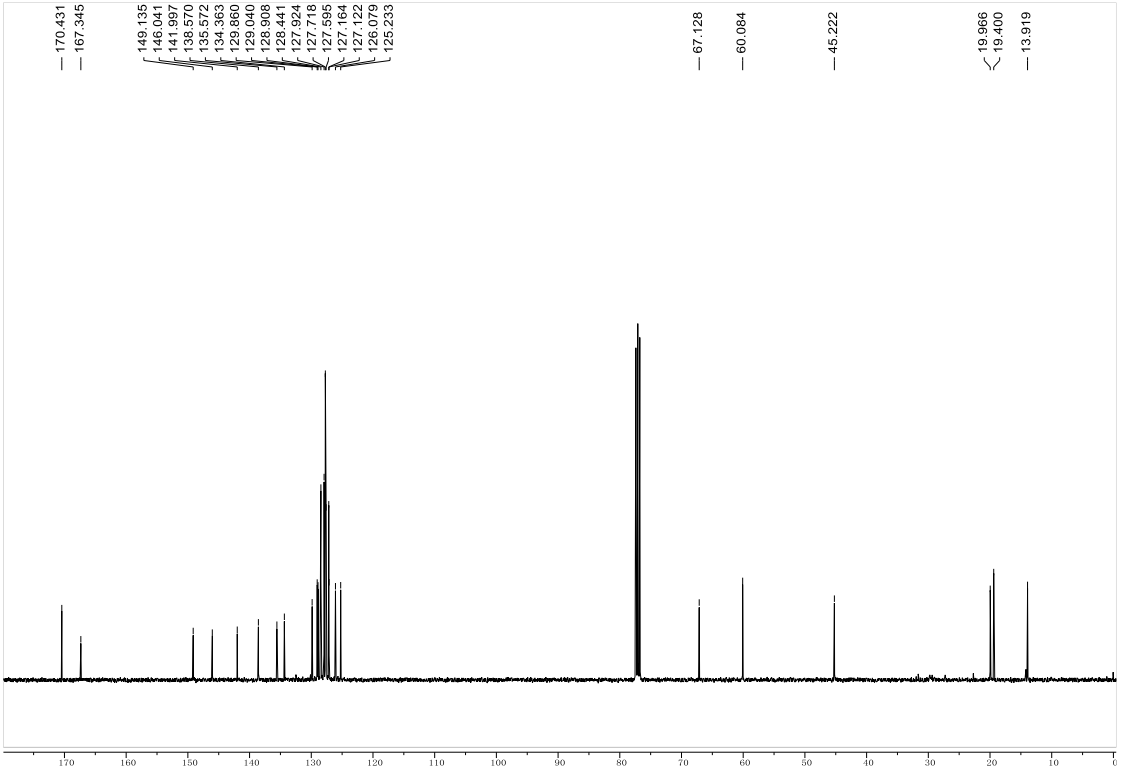


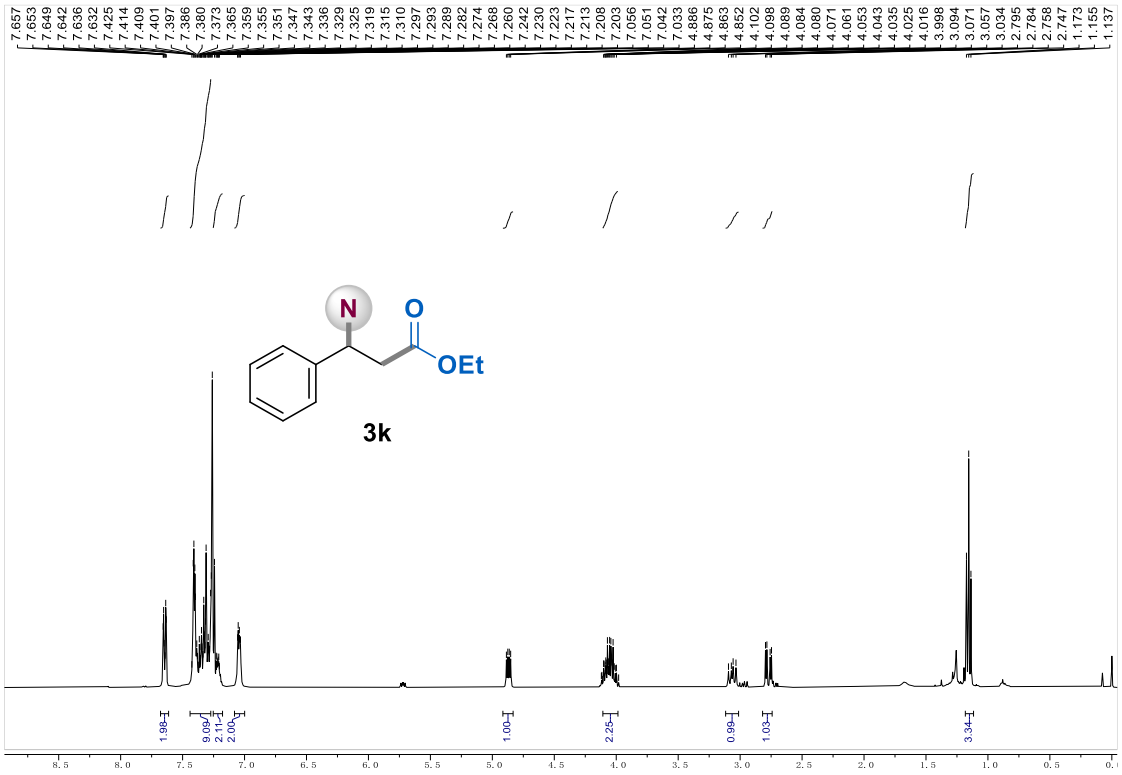

170.167
167.739
162.532
160.097
158.070
144.995
144.966
141.771
140.789
138.518
130.054
129.413
129.337
128.735
128.407
127.996
127.459
127.420
127.377
114.511
114.301
113.186
66.603
60.214
55.270
45.476
13.975
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

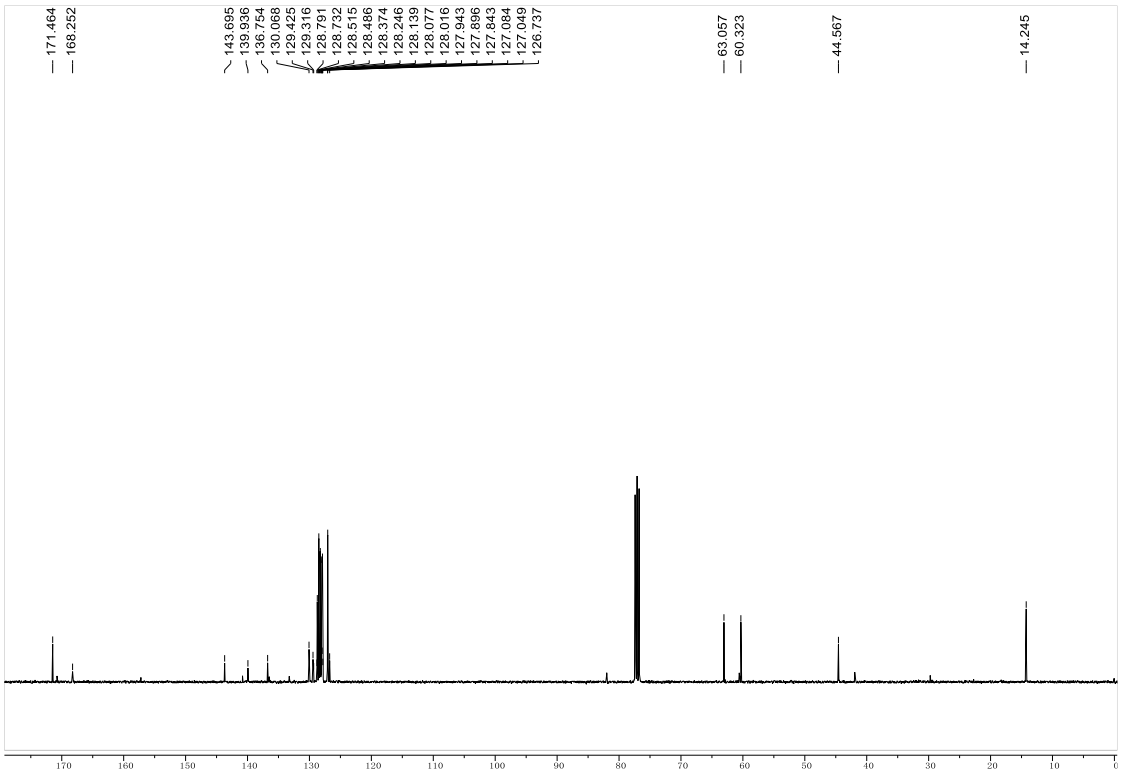
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3j**



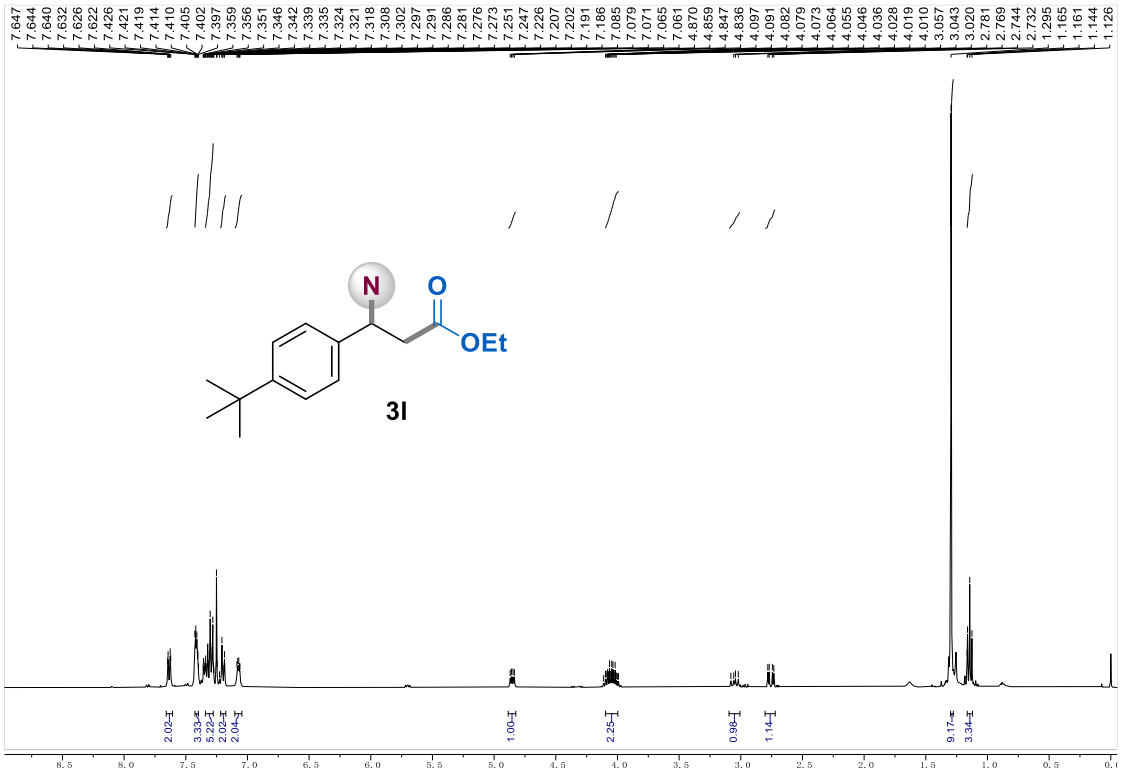



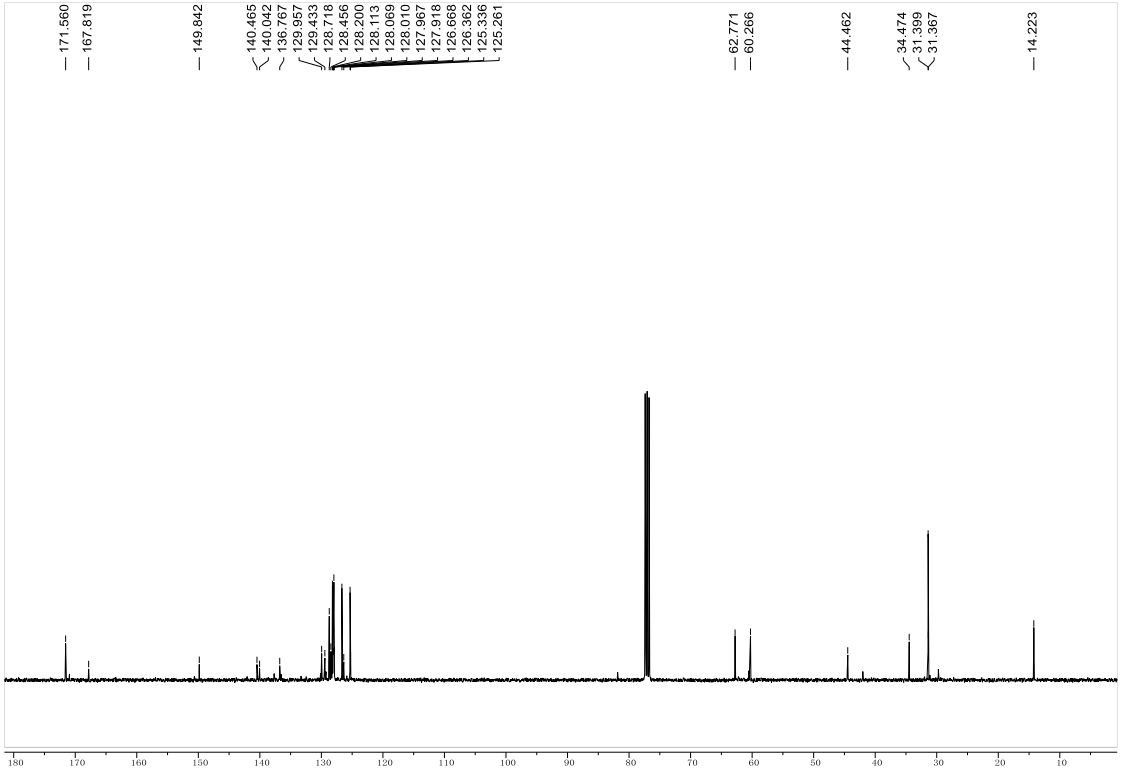
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3k**



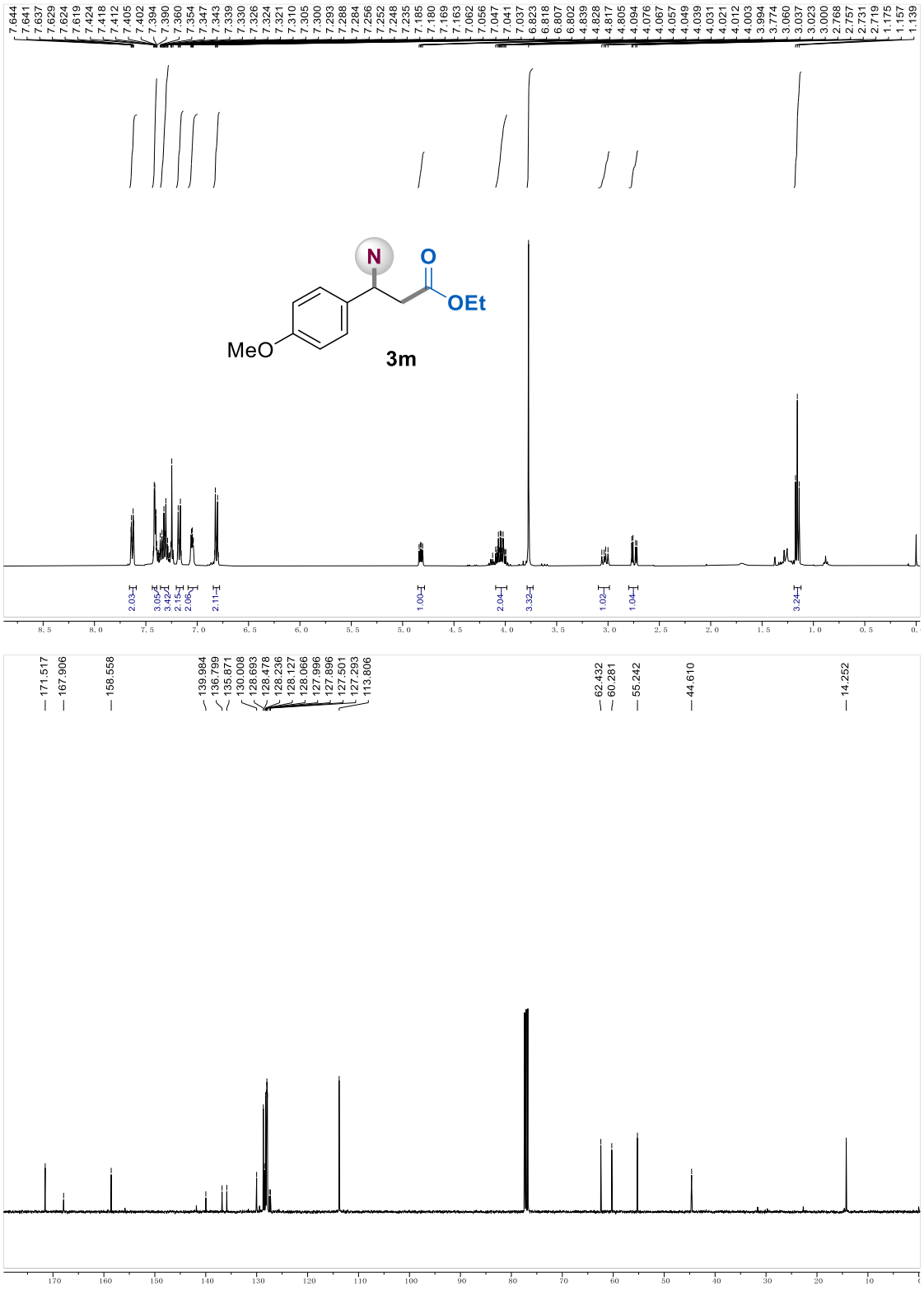



**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3l**

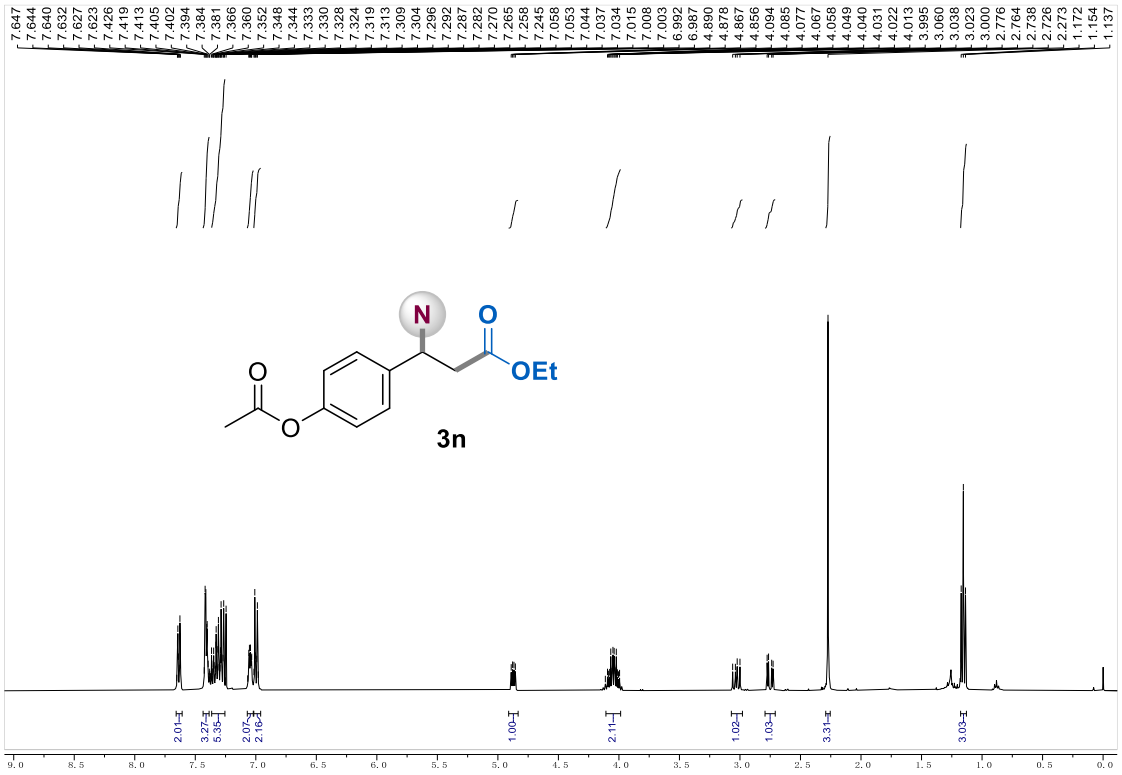


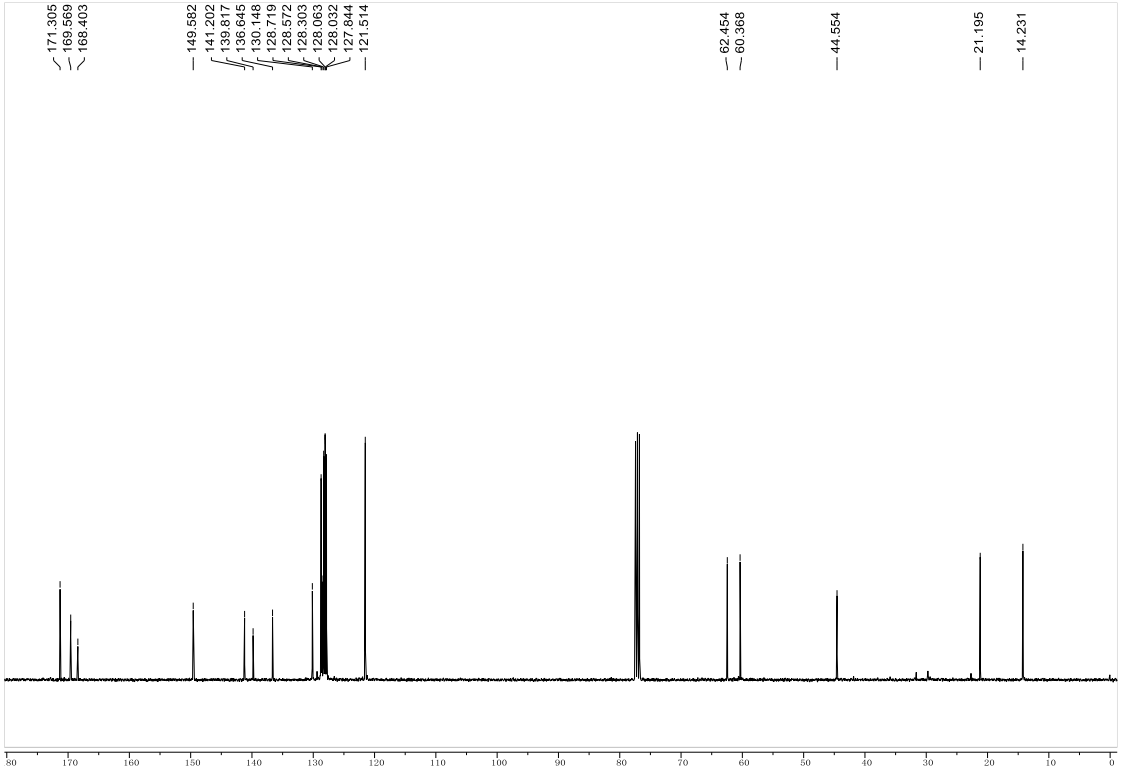



**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3m**

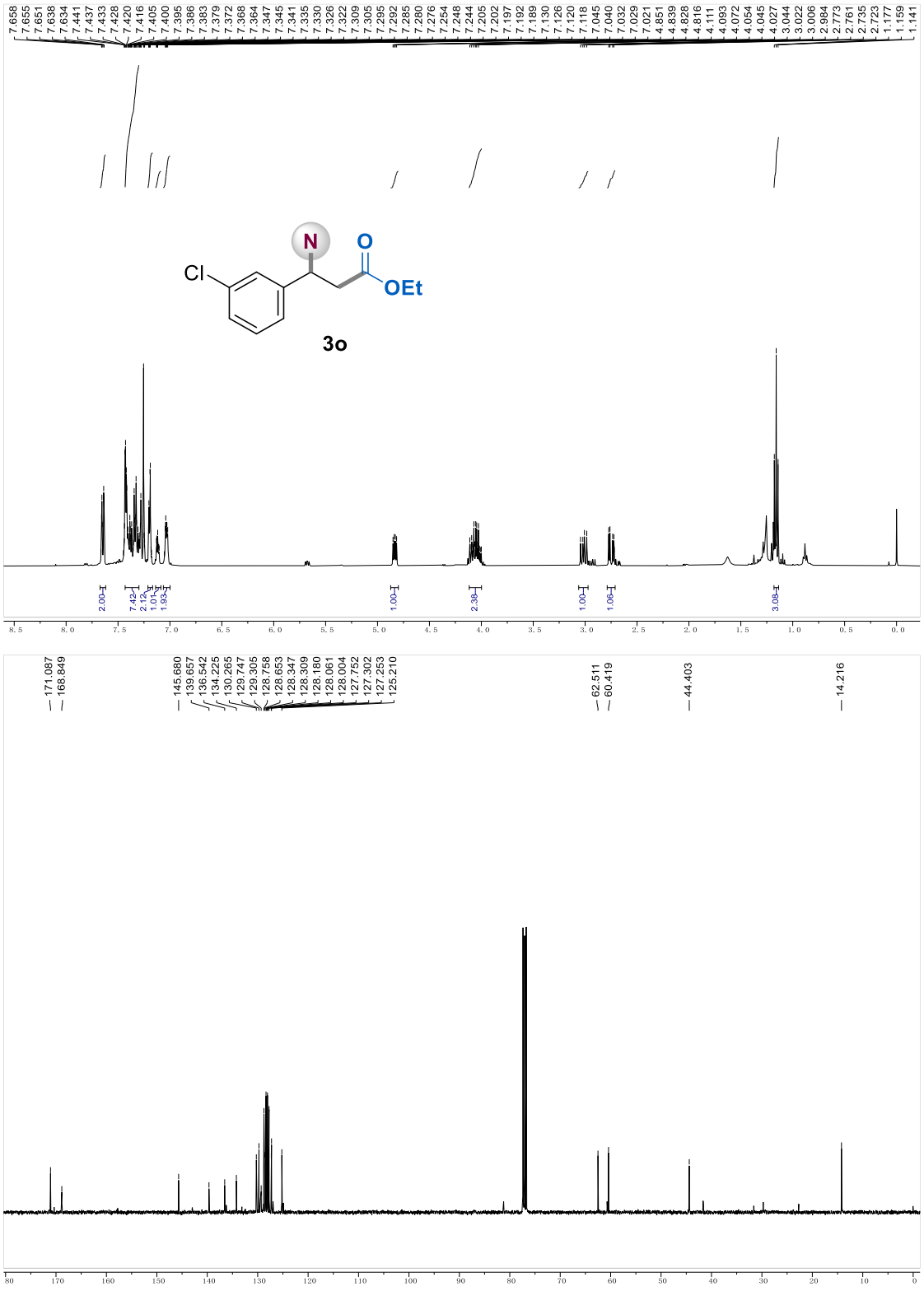


**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3n**

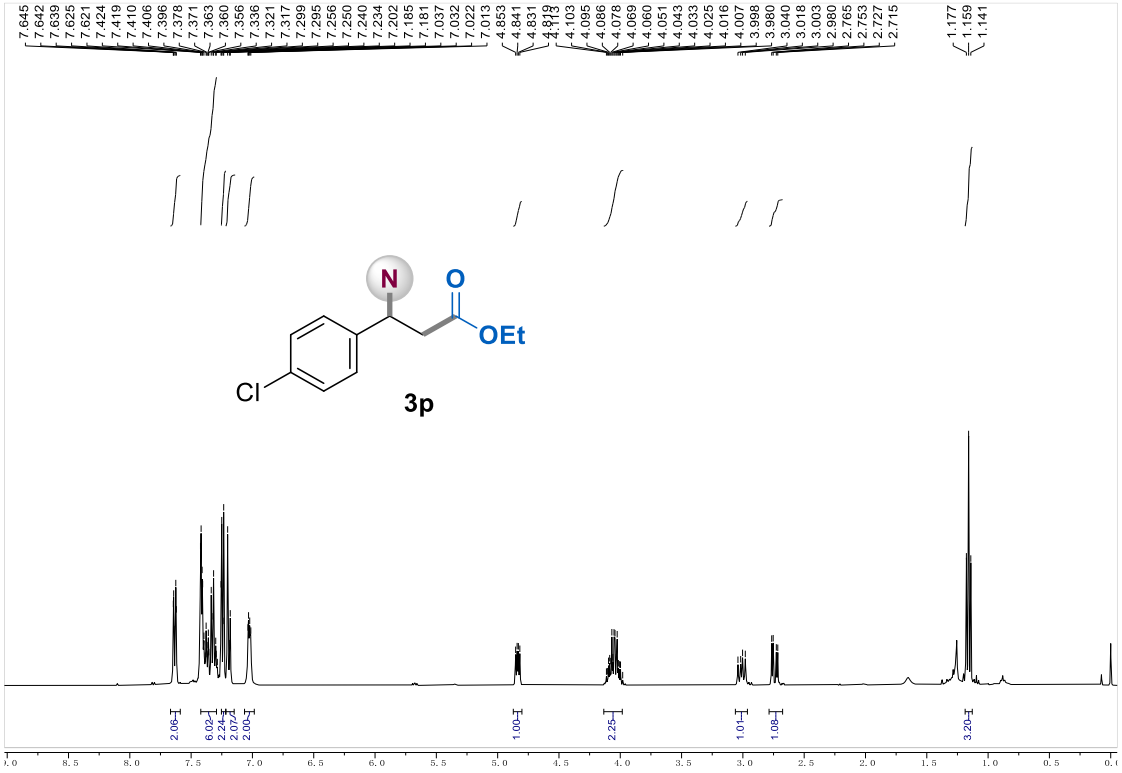


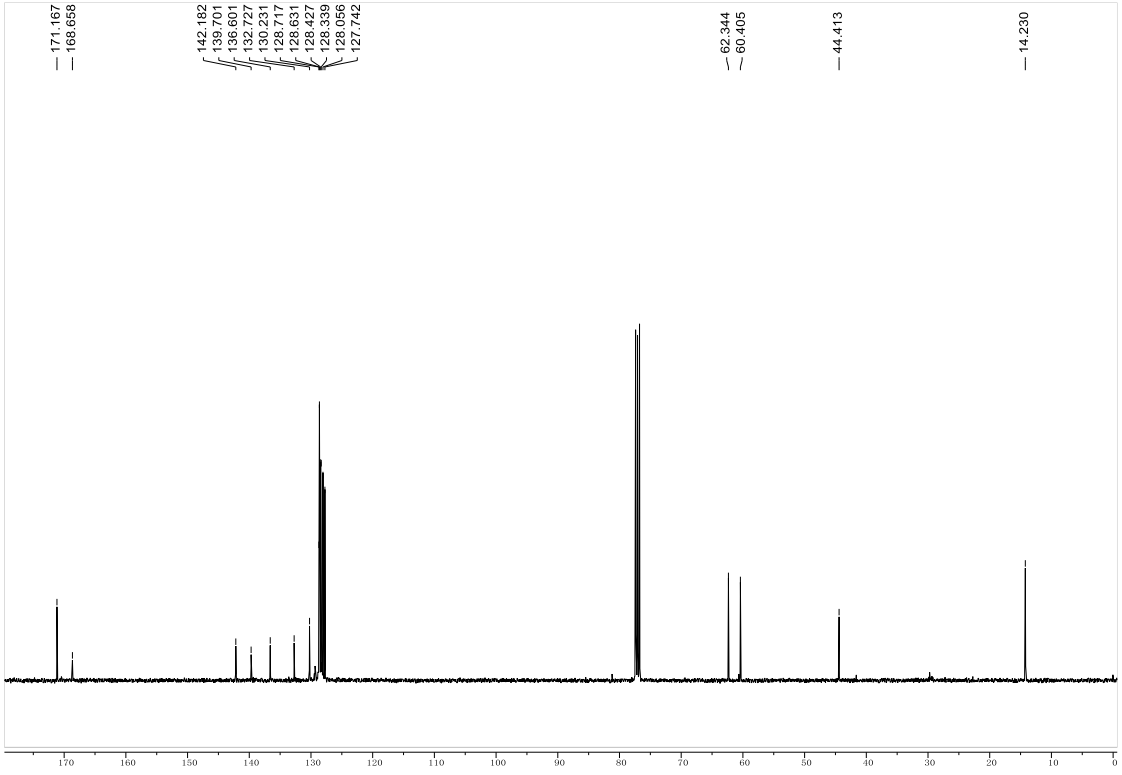



**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3o**

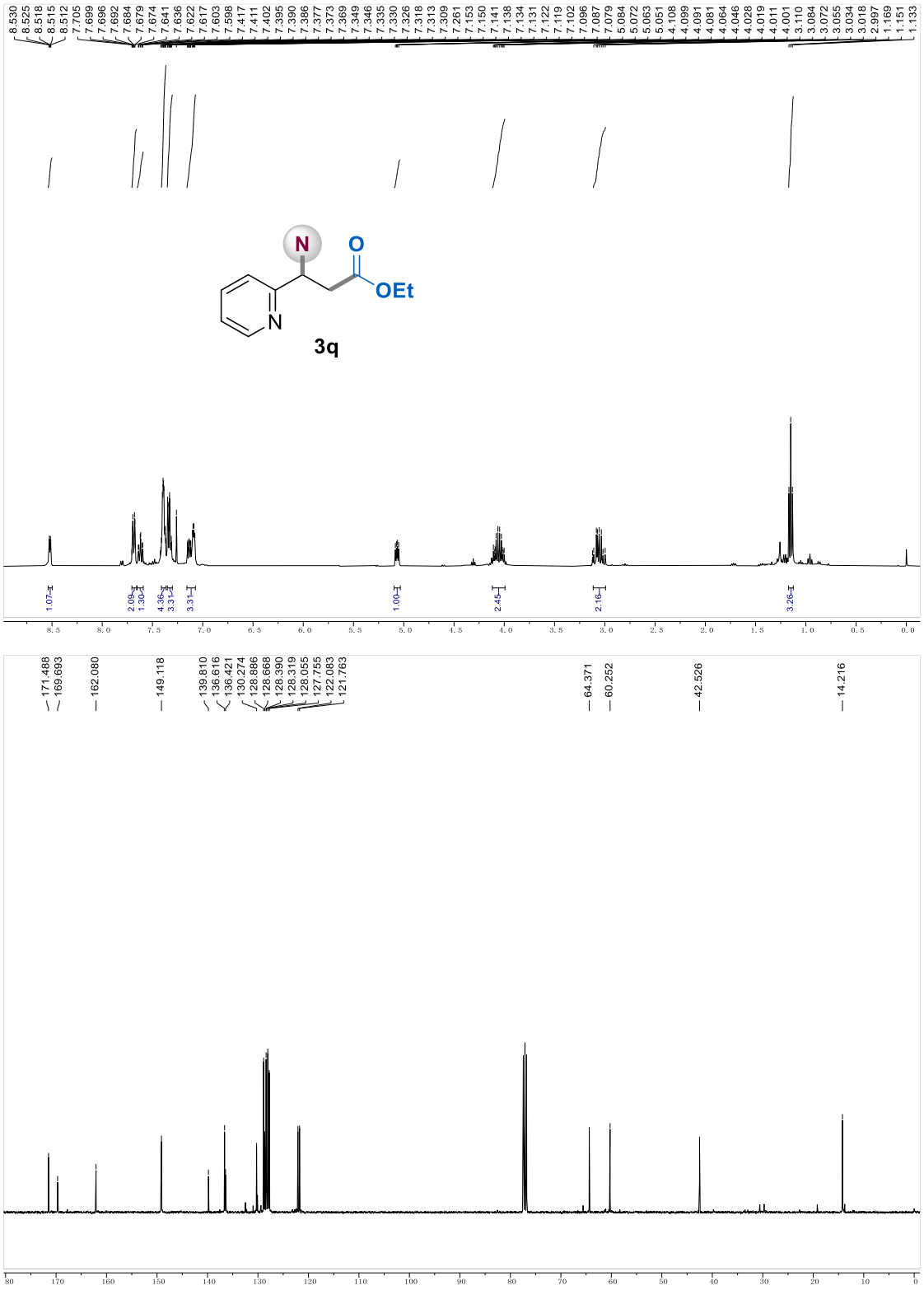


**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3p**

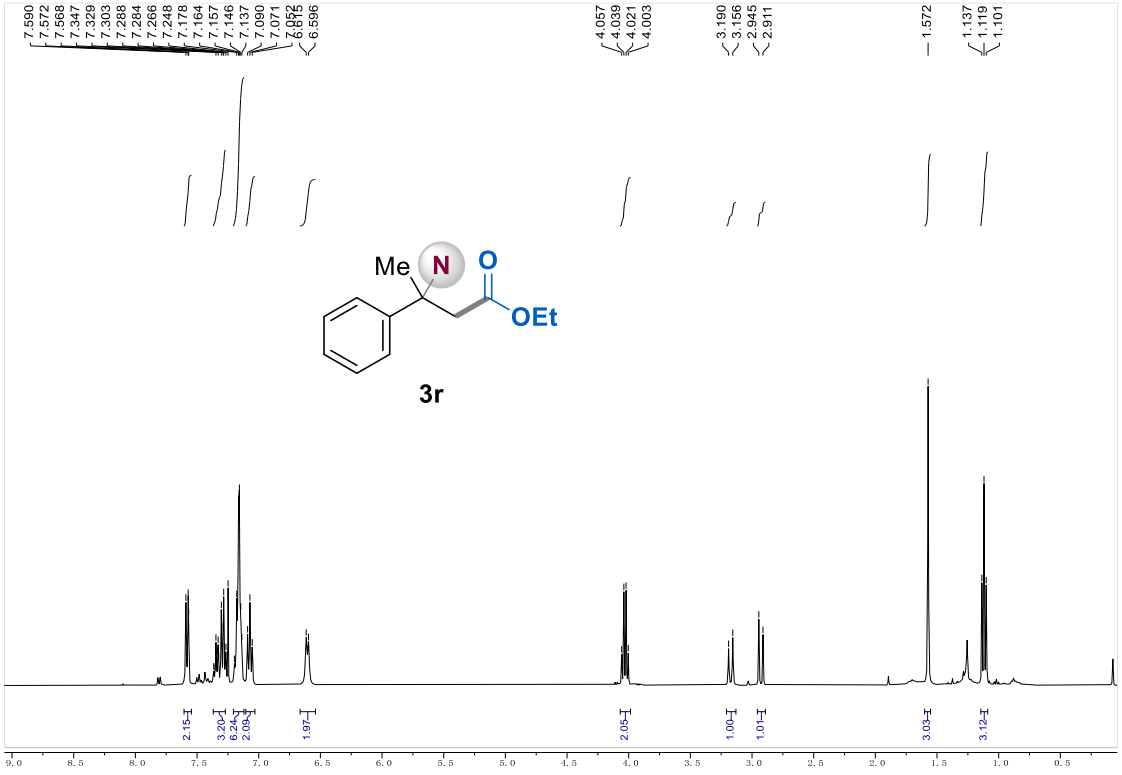


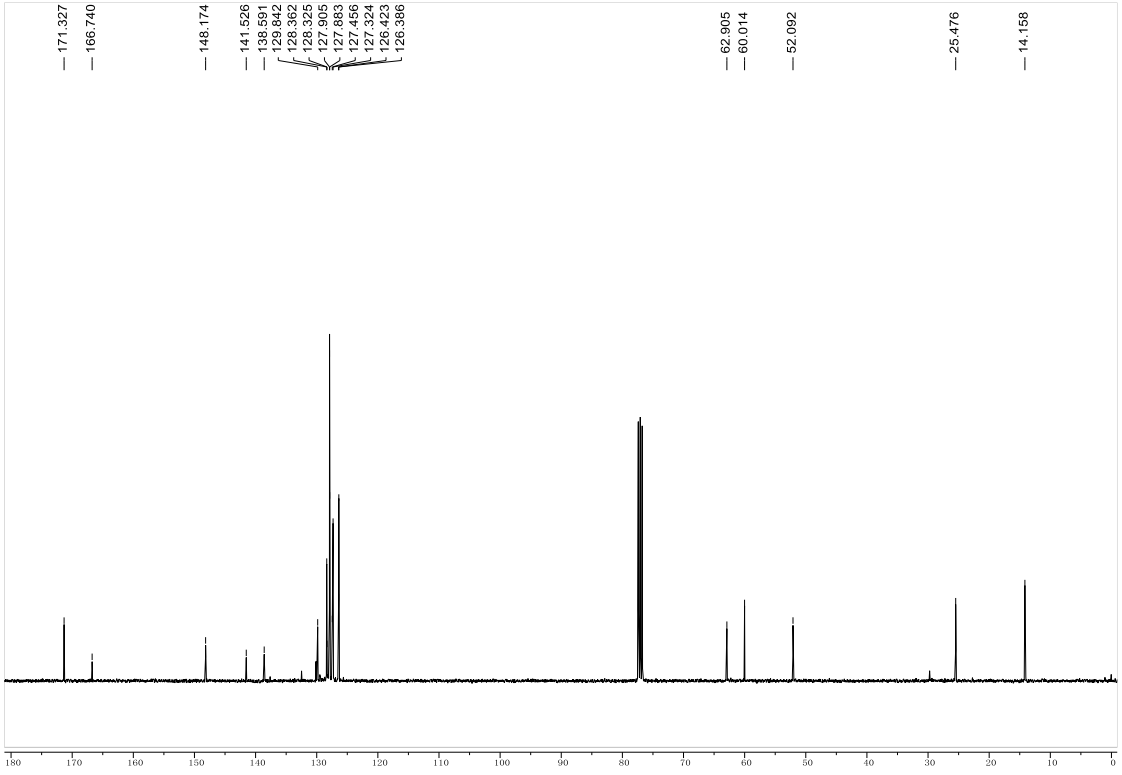



**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3q**

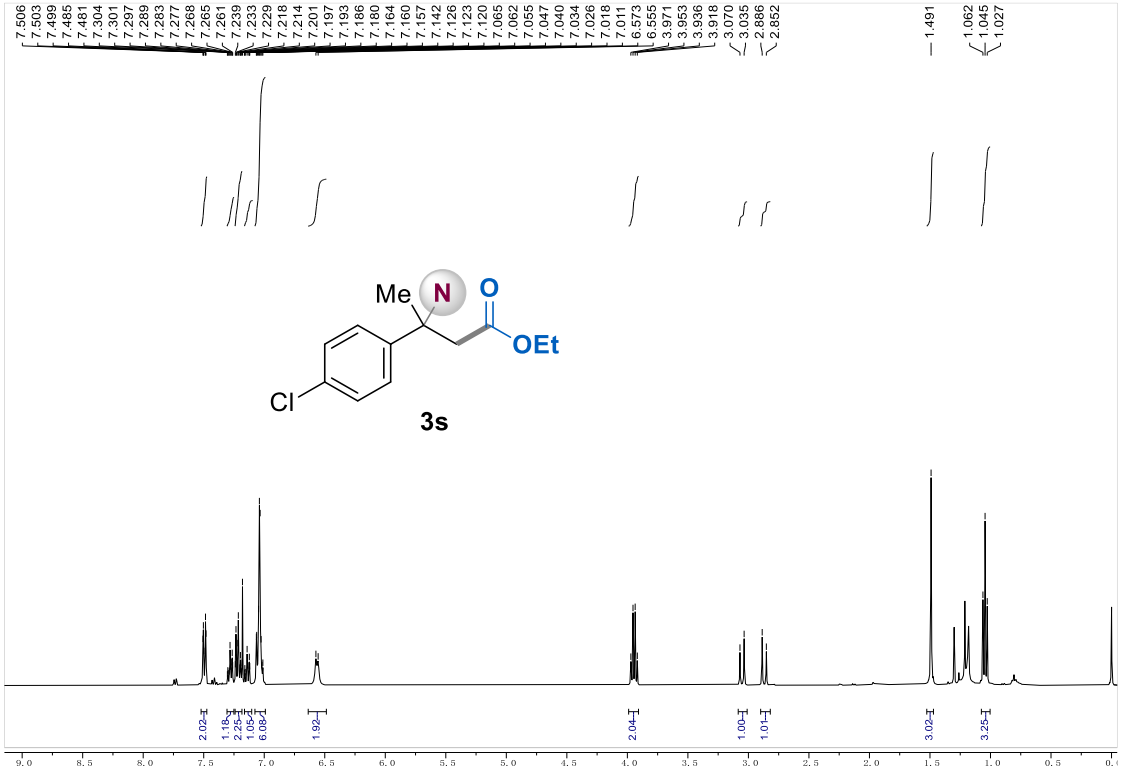


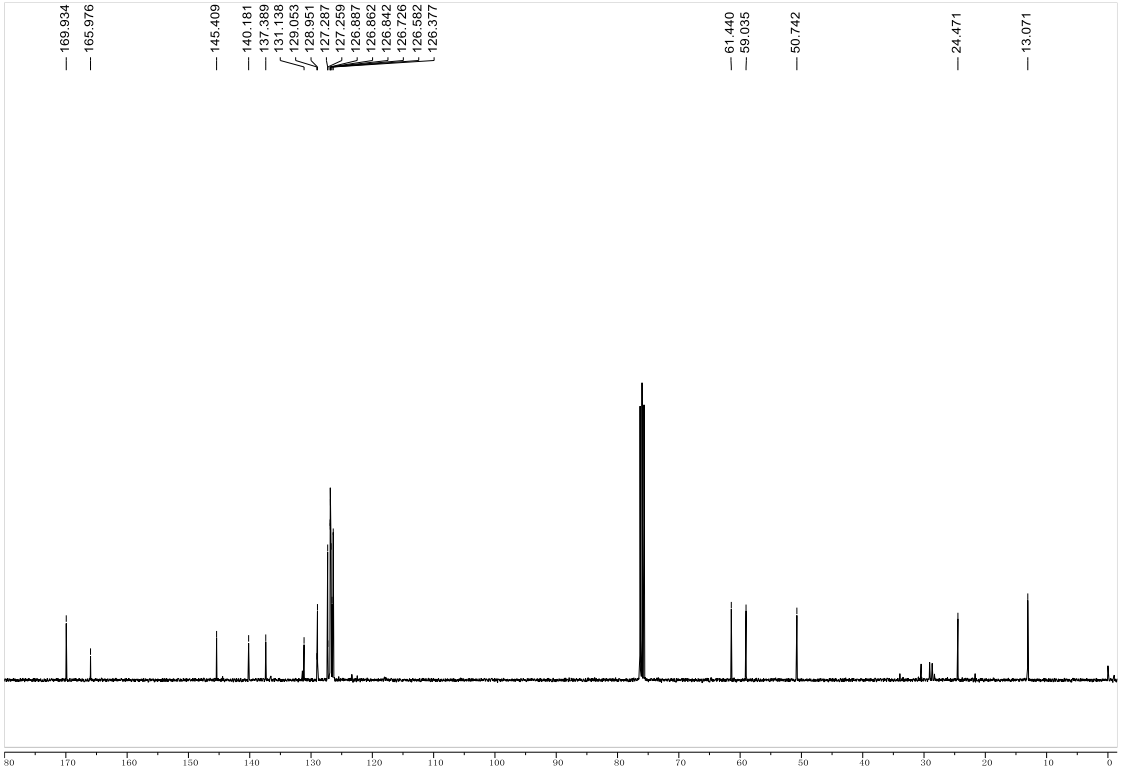
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3r**



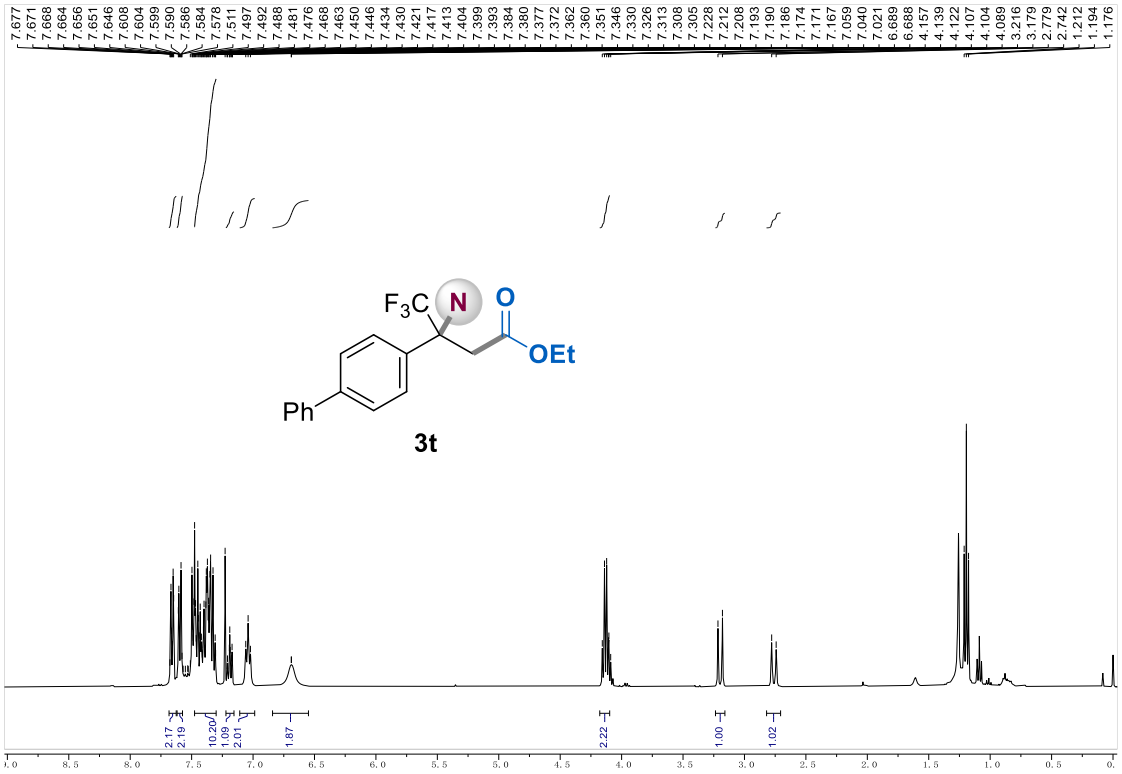



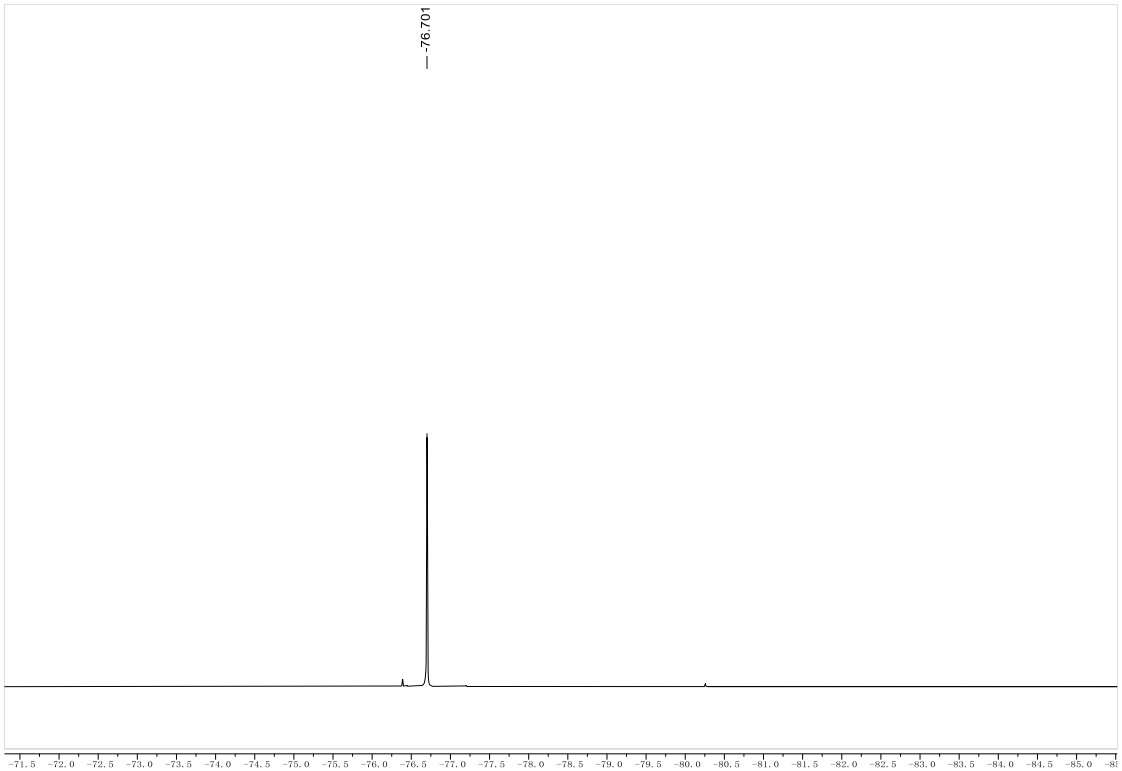
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3s**

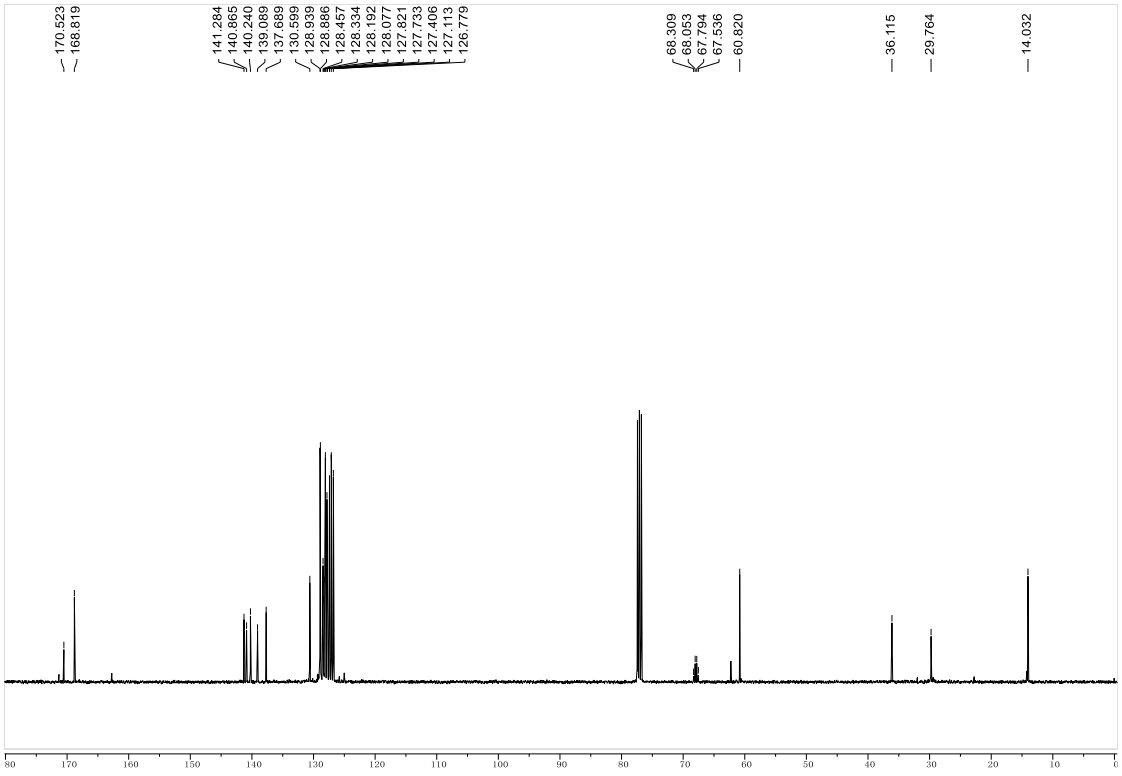


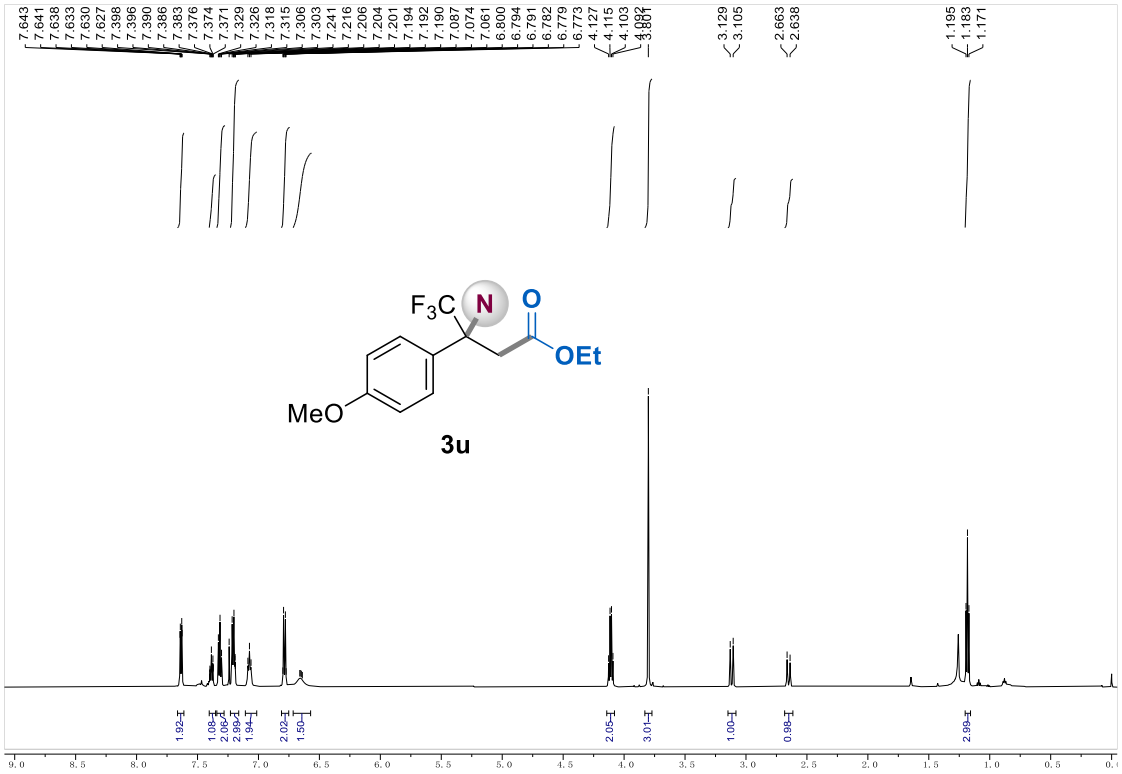
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 3t**

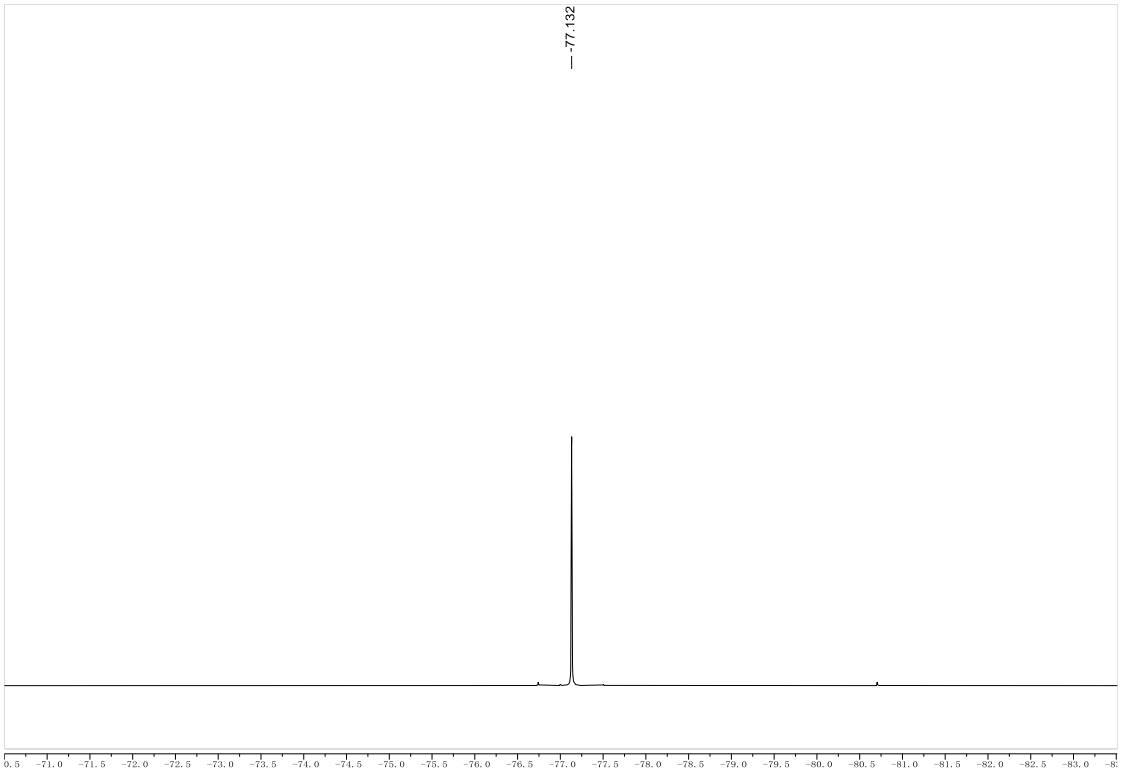


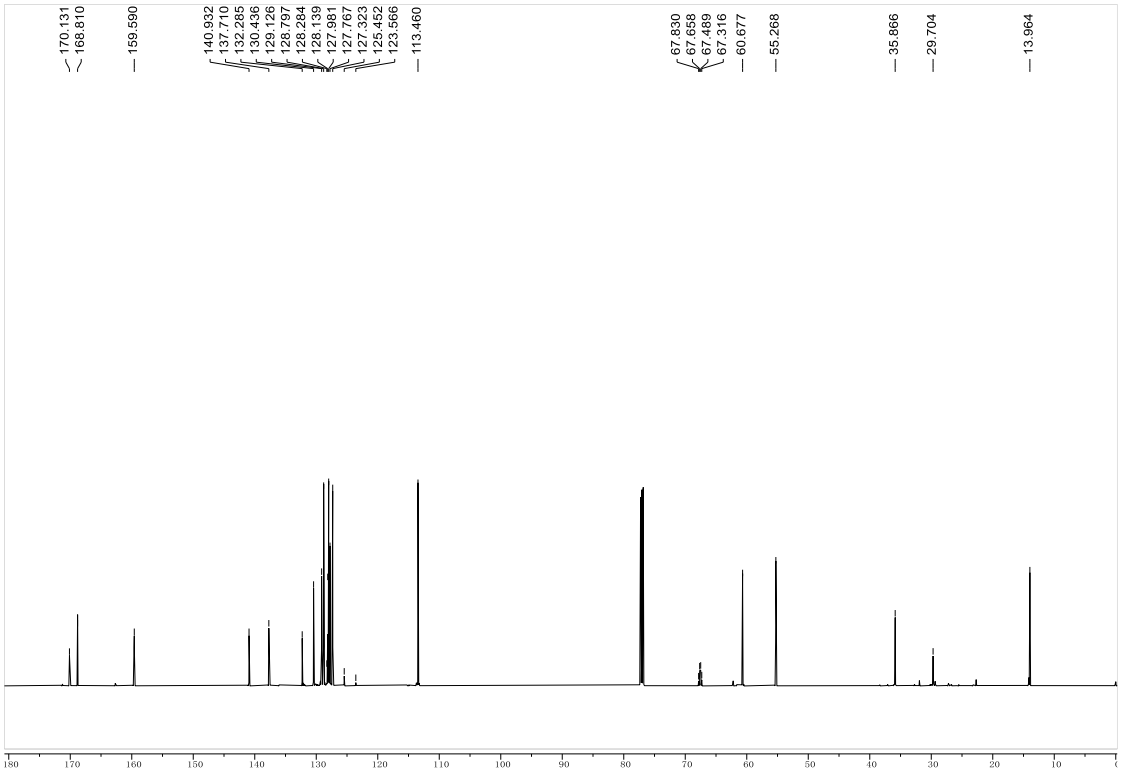




170.523
168.819
141.284
140.865
140.240
139.089
137.689
130.599
128.939
128.886
128.457
128.334
128.192
128.077
127.821
127.733
127.406
127.113
126.779
68.309
68.053
67.794
67.536
60.820
36.115
29.764
14.032
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

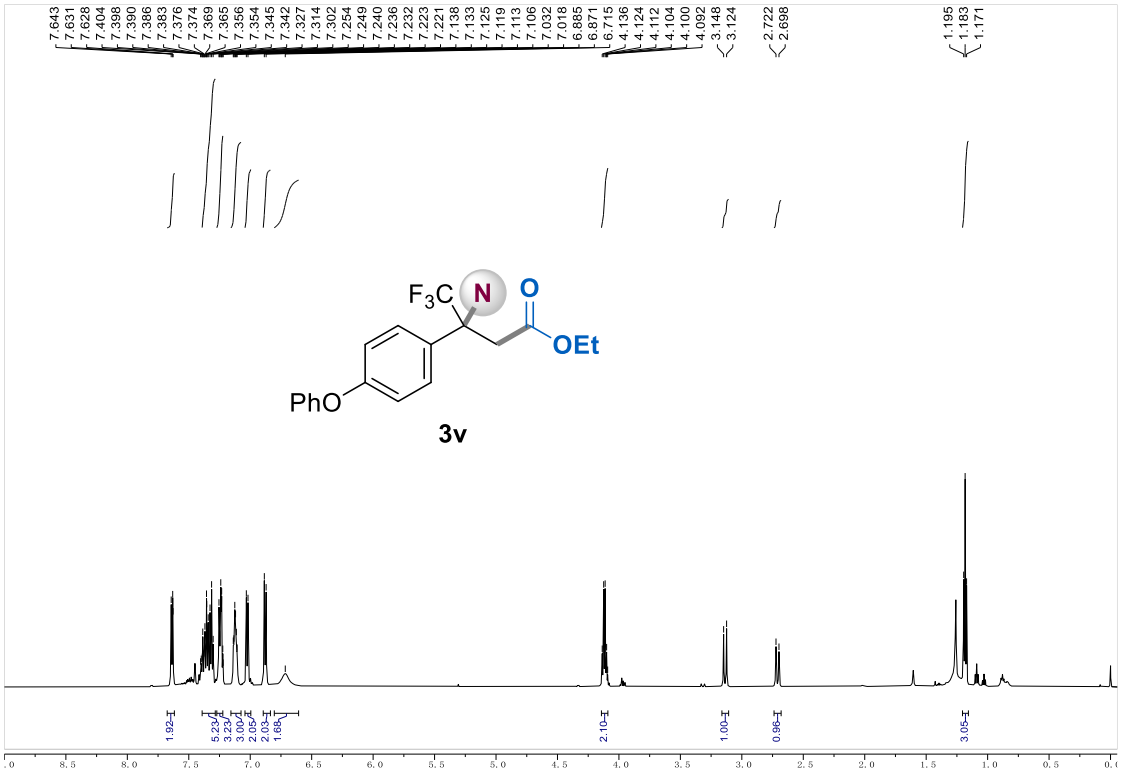
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3u**

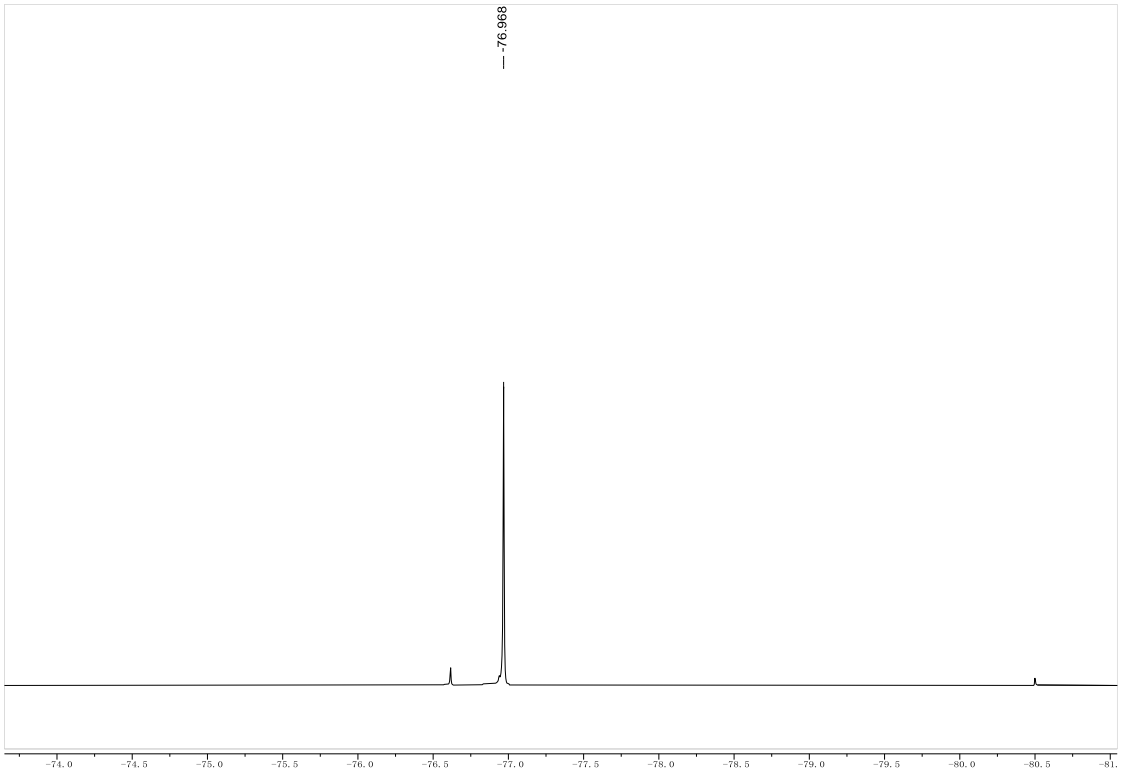


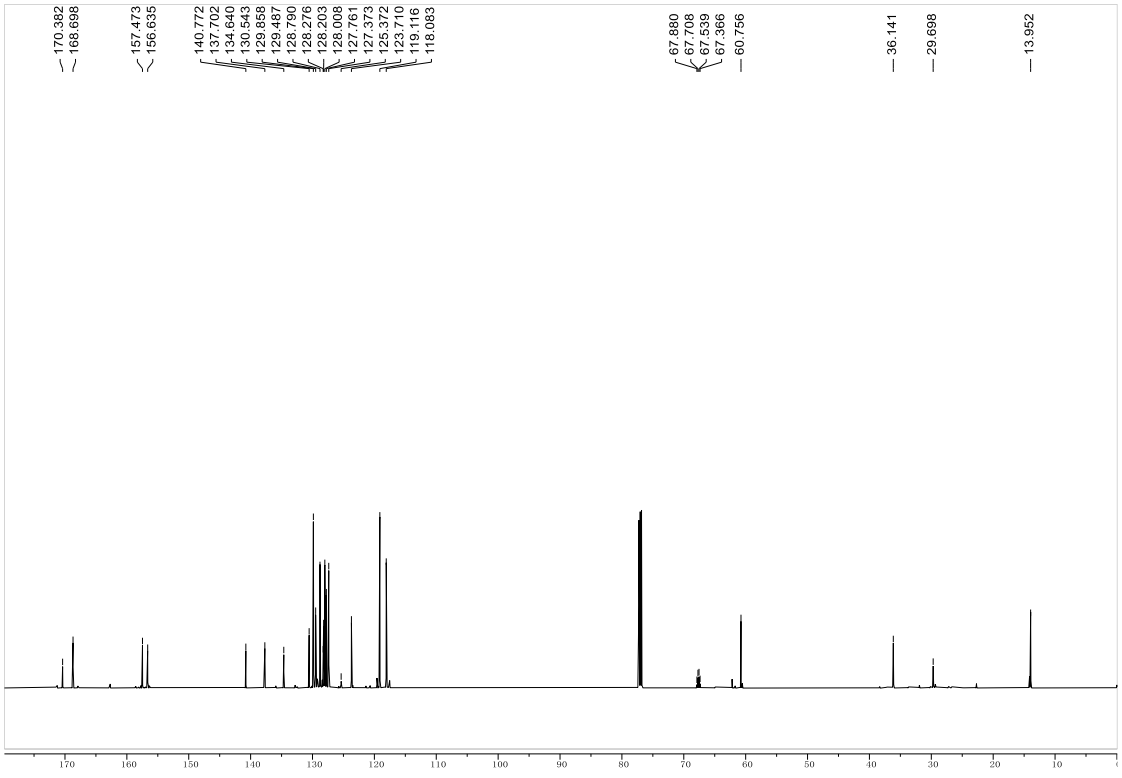




170.131
168.810
159.590
140.932
137.710
132.285
130.436
129.126
128.797
128.284
128.139
127.981
127.767
127.323
125.452
123.566
113.460
67.830
67.658
67.489
67.316
60.677
55.268
35.866
29.704
13.964
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

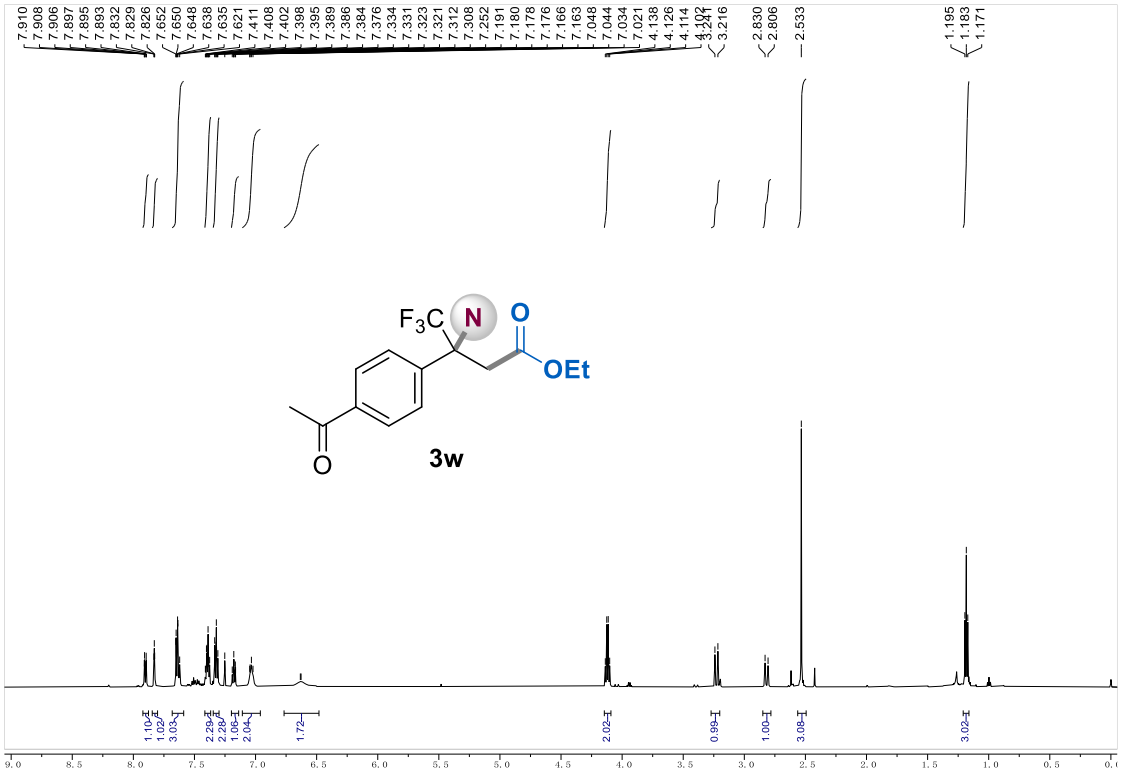
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3v**

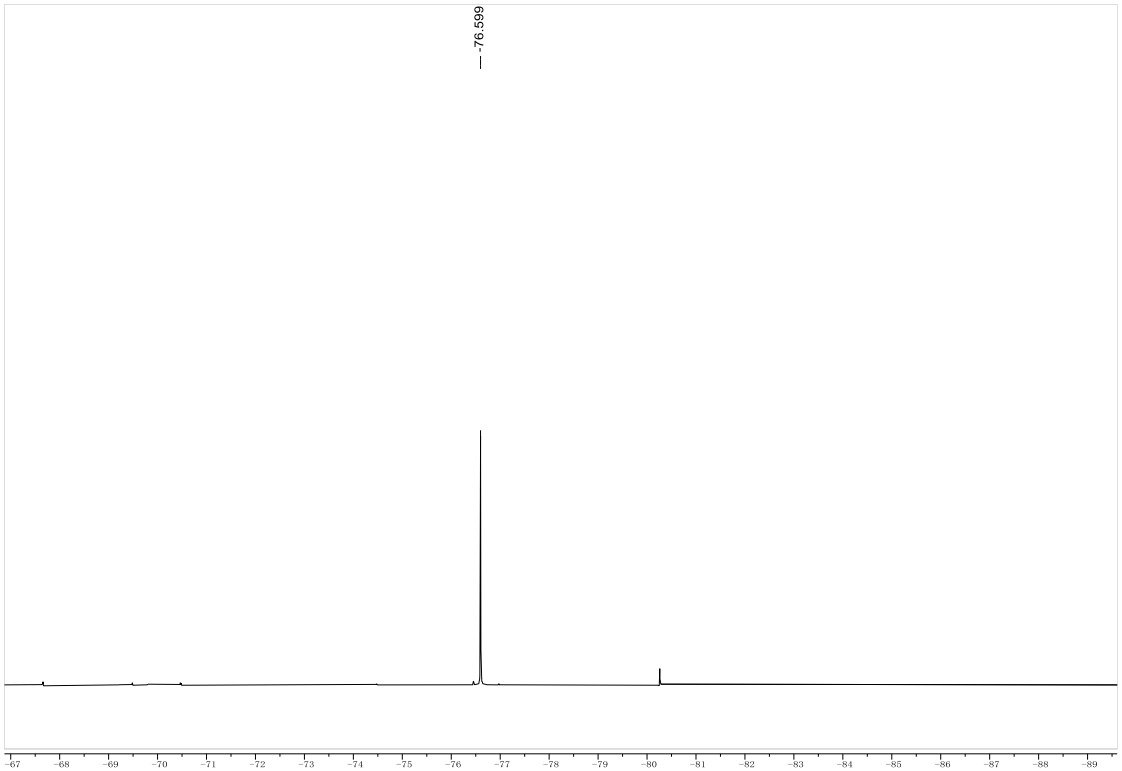


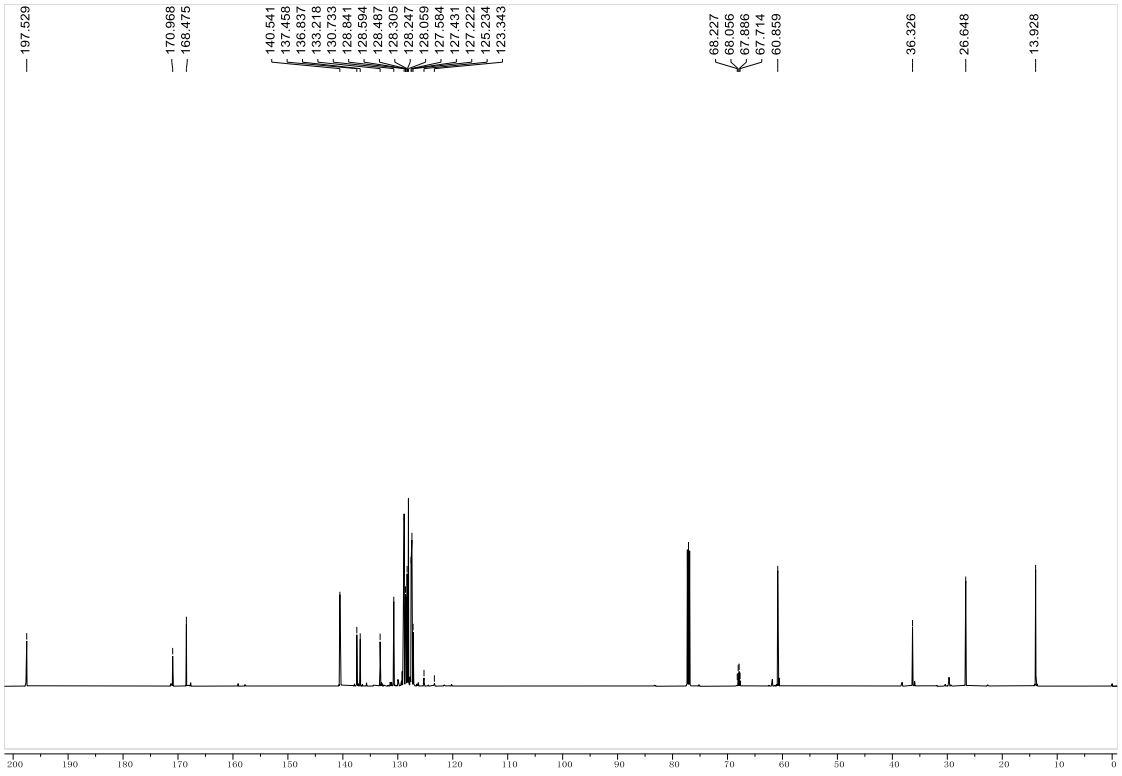




170.382
168.698
157.473
156.635
140.772
137.702
134.640
130.543
129.858
129.487
128.790
128.276
128.203
128.008
127.761
127.373
125.372
123.710
119.116
118.083
67.880
67.708
67.539
67.366
60.756
36.141
29.698
13.952
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

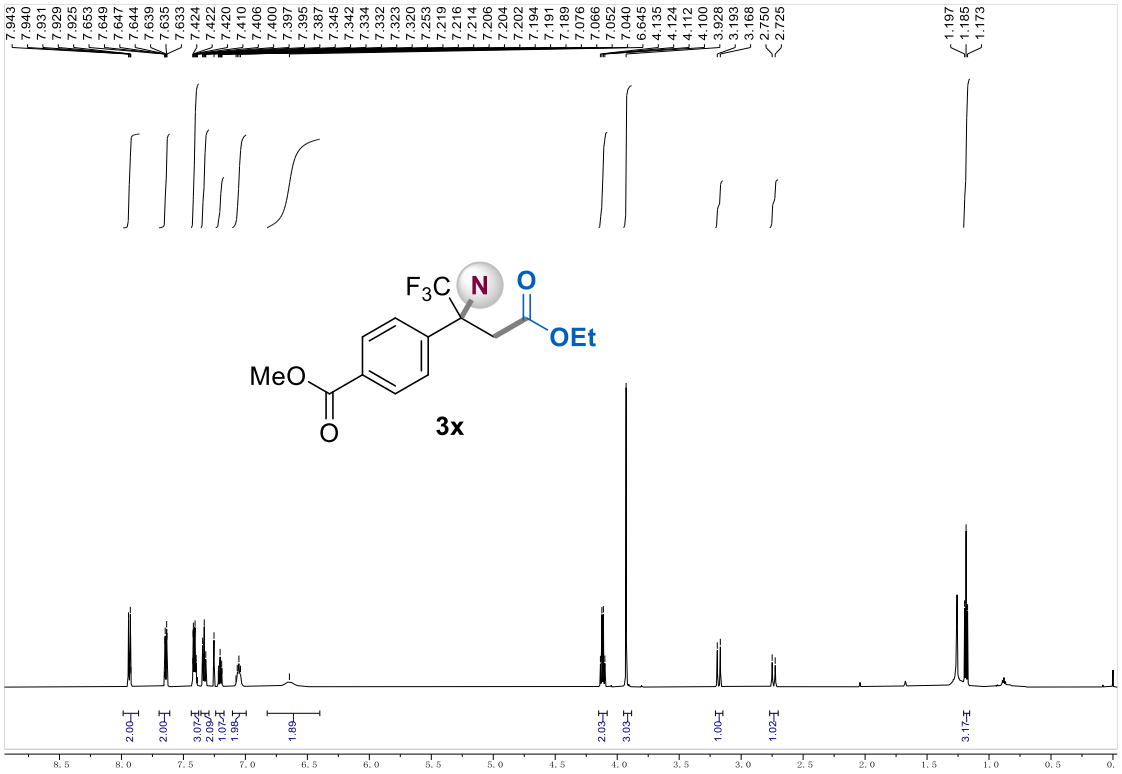
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3w**

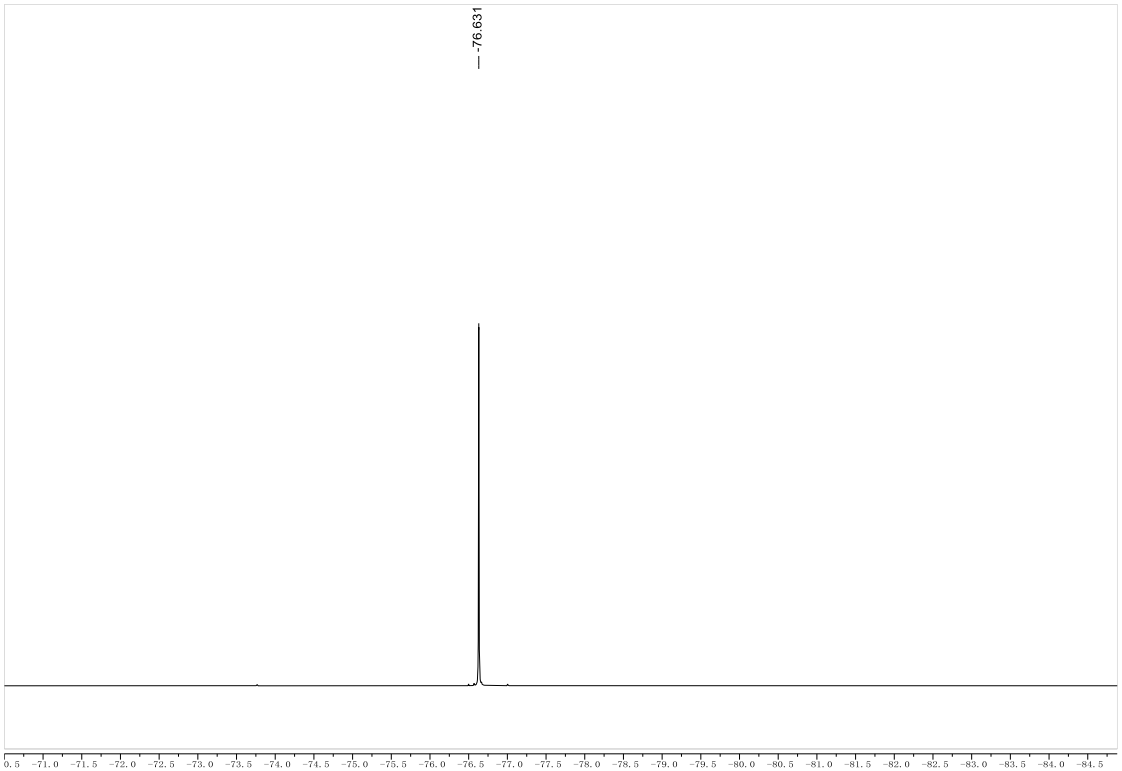


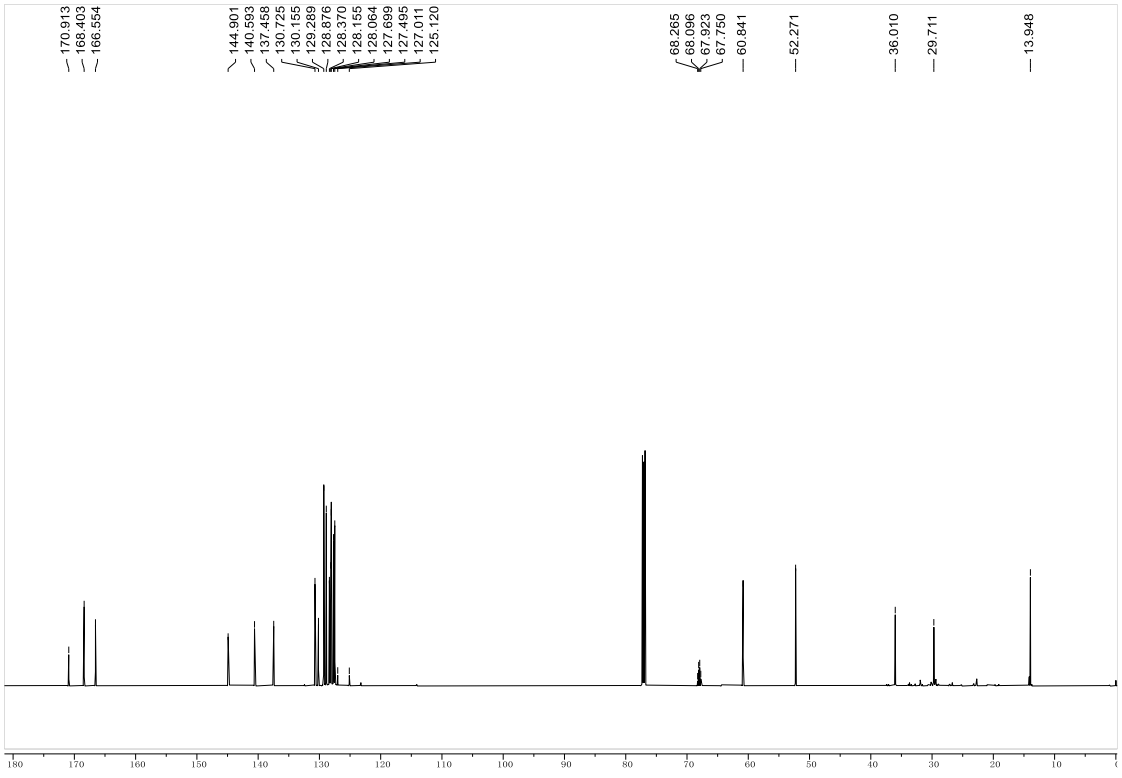




197.529
170.968
168.475
140.541
137.458
136.837
133.218
130.733
128.841
128.594
128.487
128.305
128.247
128.059
127.584
127.431
127.222
125.234
123.343
68.227
68.056
67.886
67.714
60.859
36.326
26.648
13.928
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

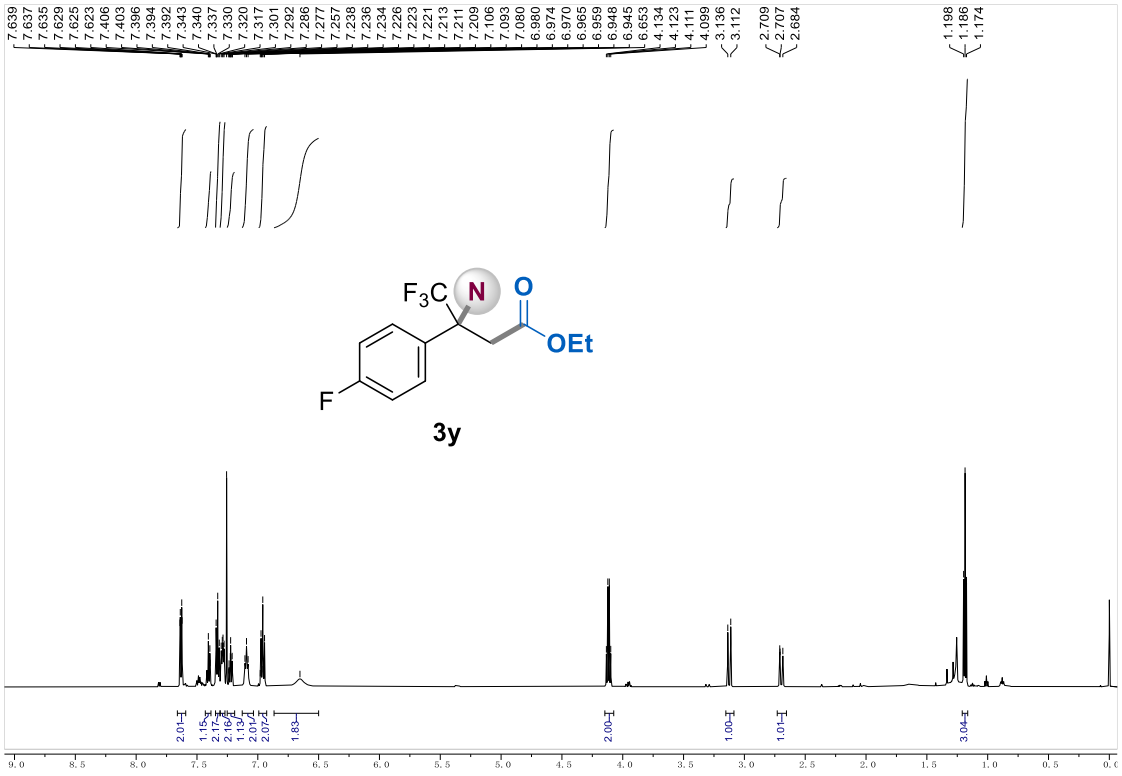
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3x**

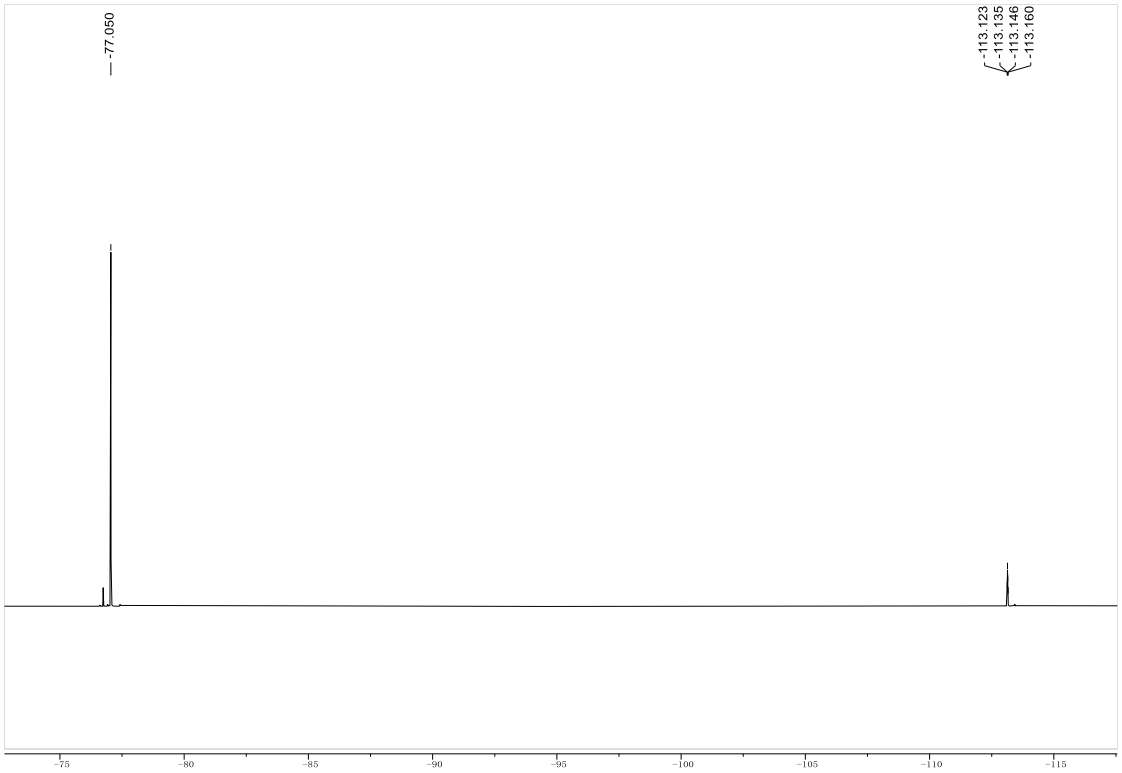


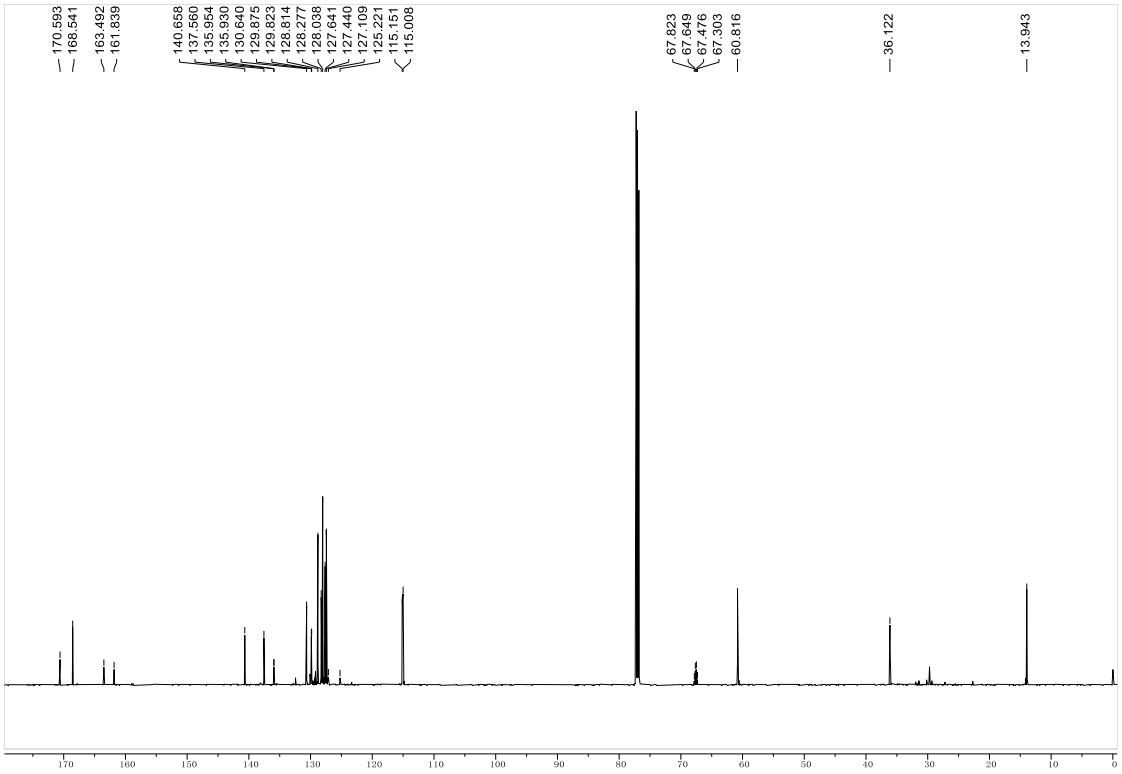




170.913
168.403
166.554
144.901
140.593
137.458
130.725
130.155
129.289
128.876
128.370
128.155
128.064
127.699
127.495
127.011
125.120
68.265
68.096
67.923
67.750
60.841
52.271
36.010
29.711
13.948
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

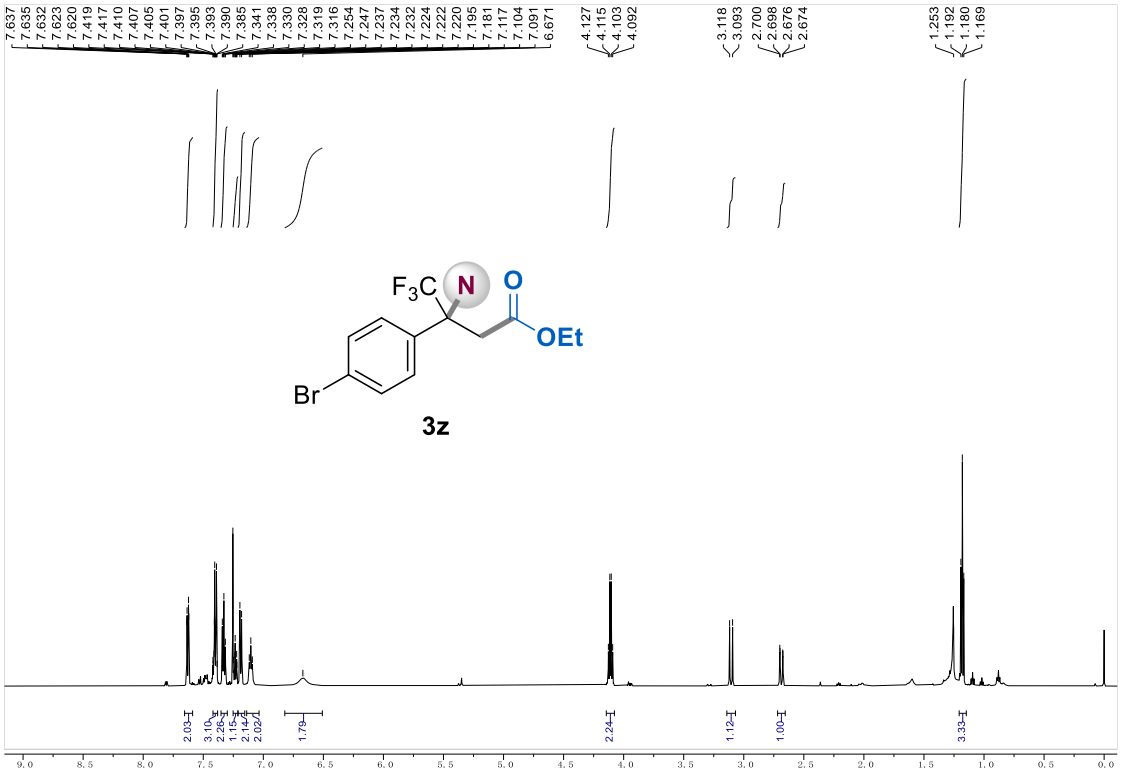
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3y**

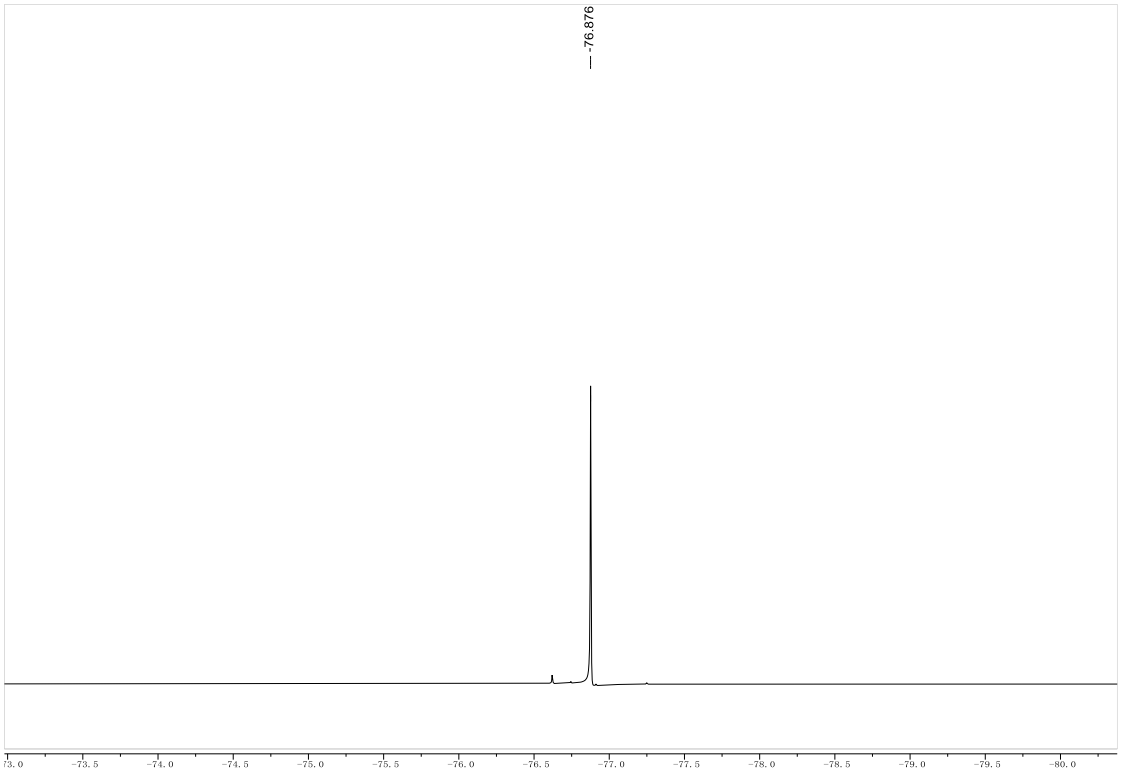


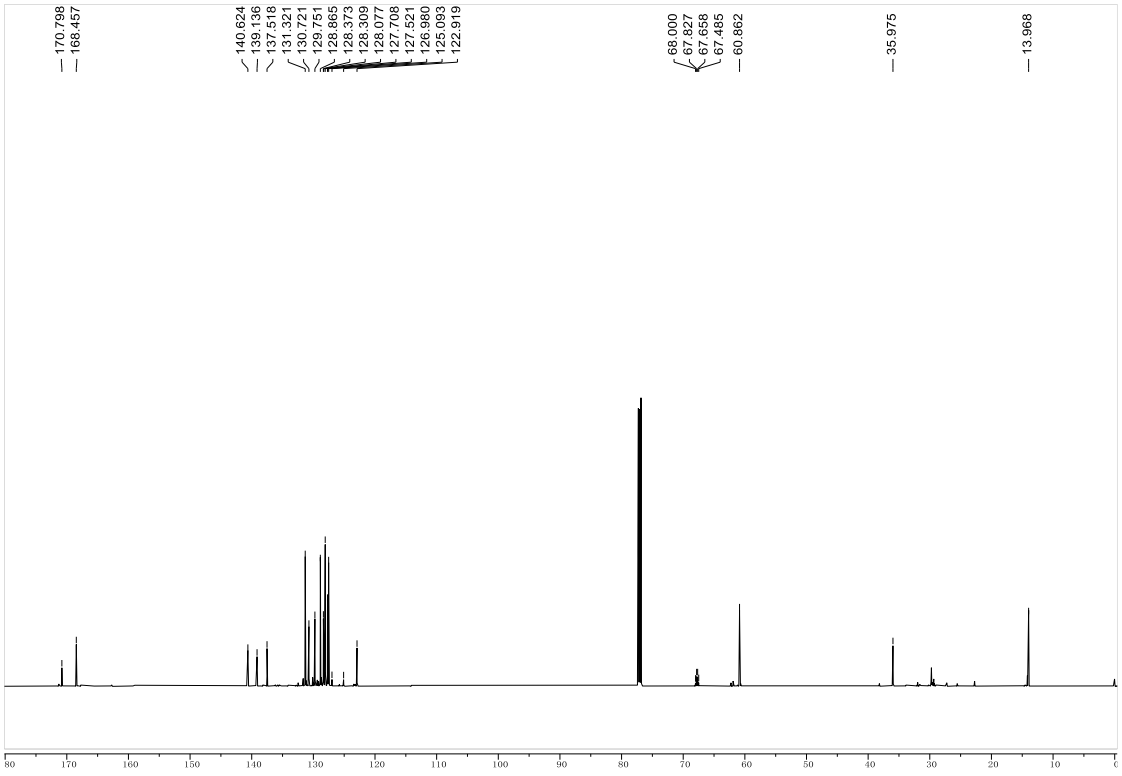




170.593
168.541
163.492
161.839
140.658
137.560
135.954
135.930
130.640
129.875
129.823
128.814
128.277
128.038
127.641
127.440
127.109
125.221
115.151
115.008
67.823
67.649
67.476
67.303
60.816
36.122
13.943
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

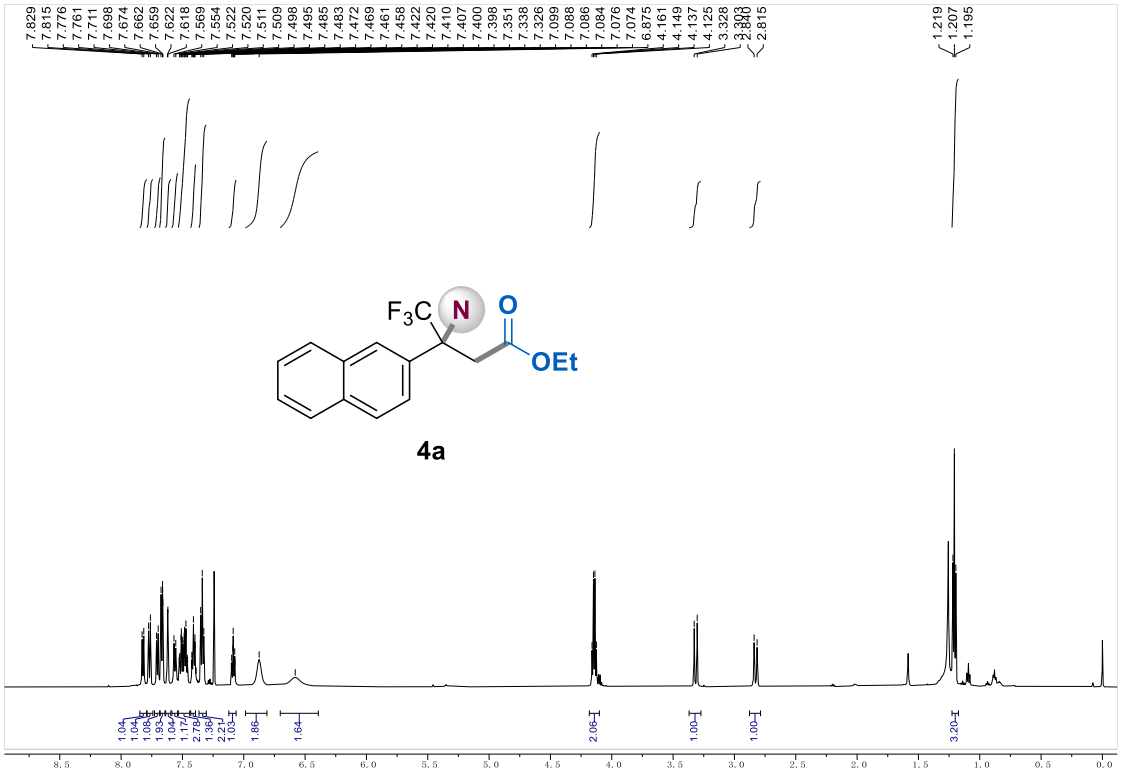
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 3z**

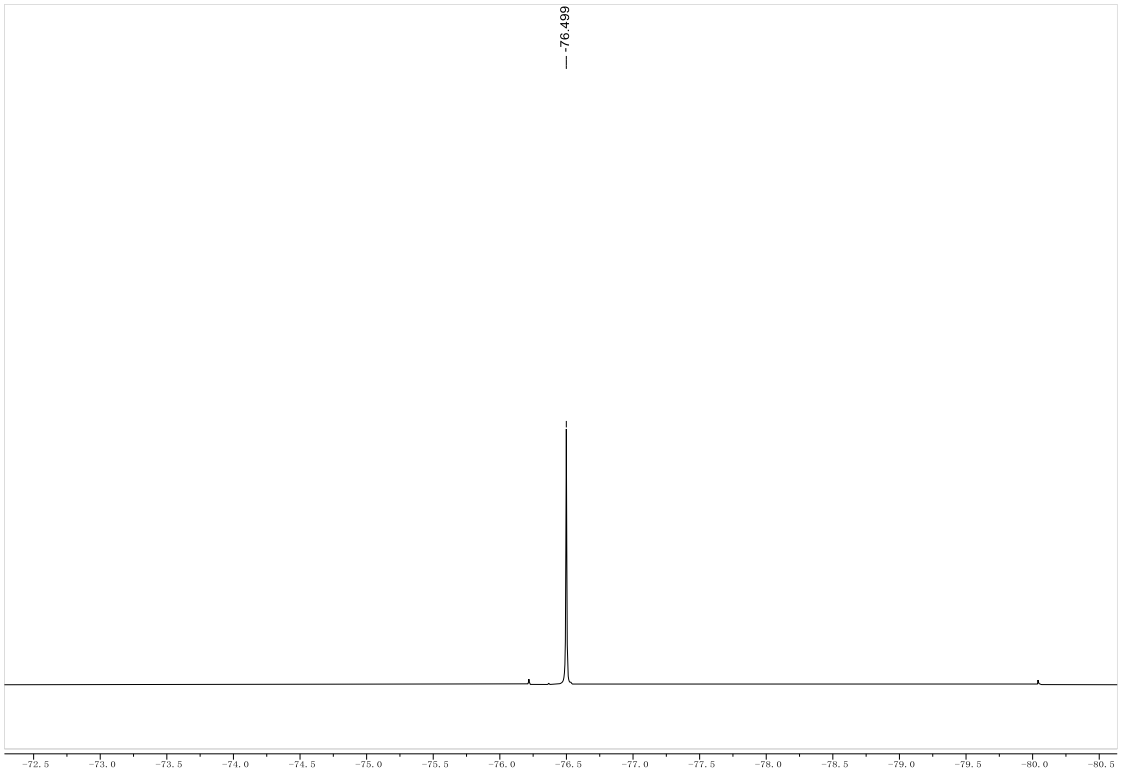


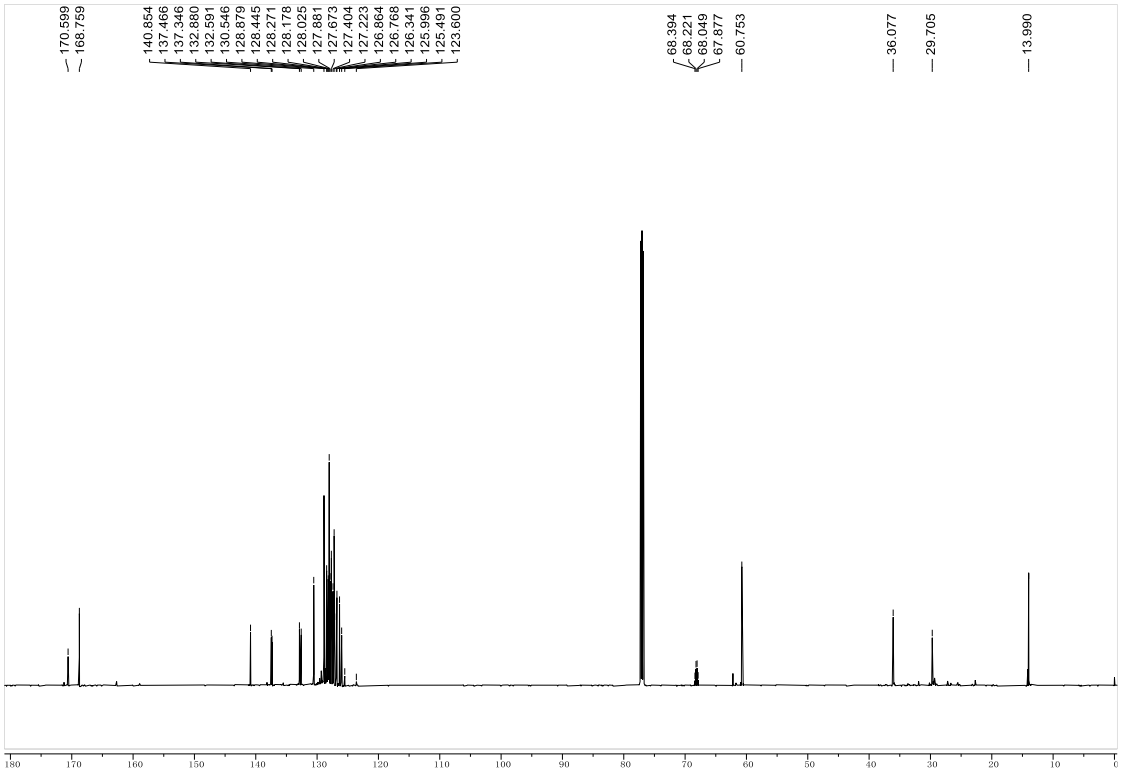




170.798
168.457
140.624
139.136
137.518
131.321
130.721
129.751
128.865
128.373
128.309
128.077
127.708
127.521
126.980
125.093
122.919
68.000
67.827
67.658
67.485
60.862
35.975
13.968
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

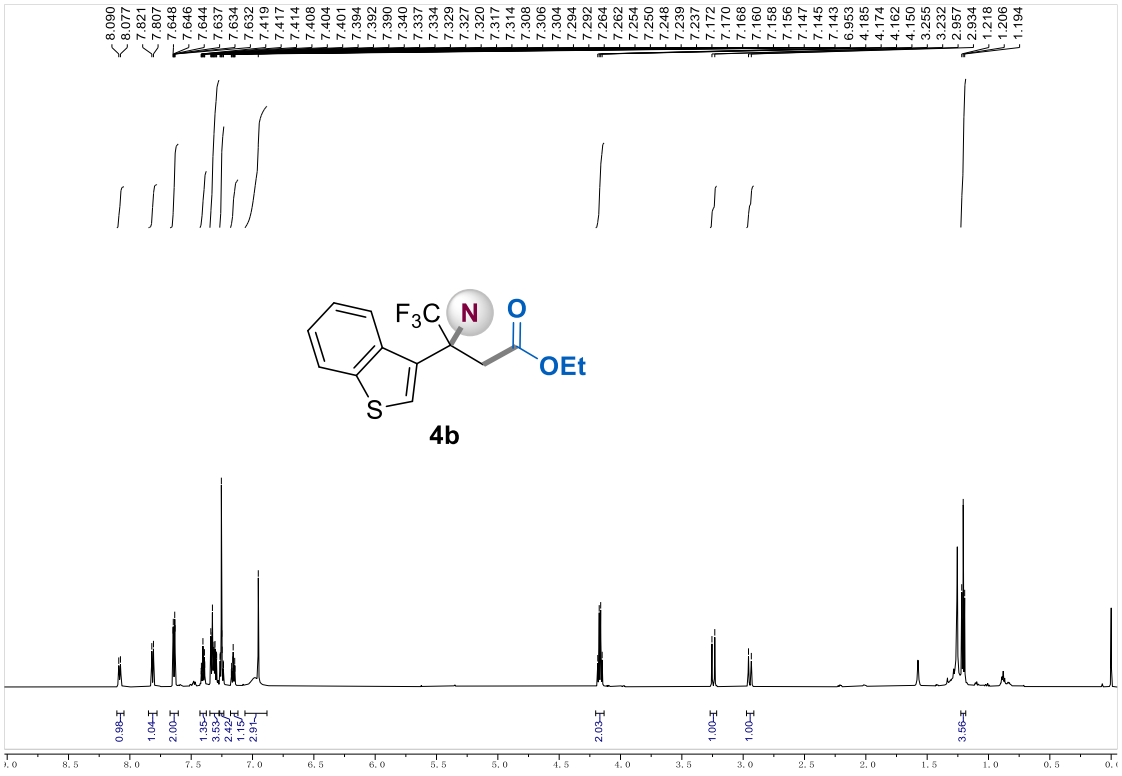
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 4a**

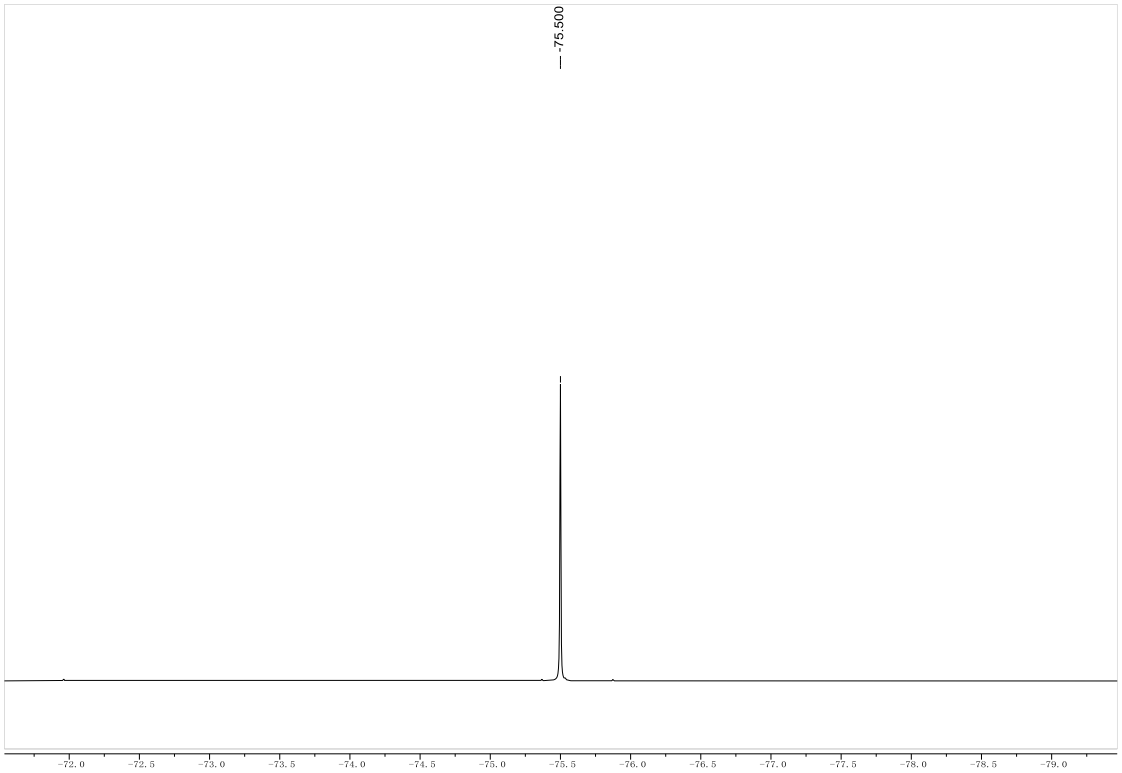


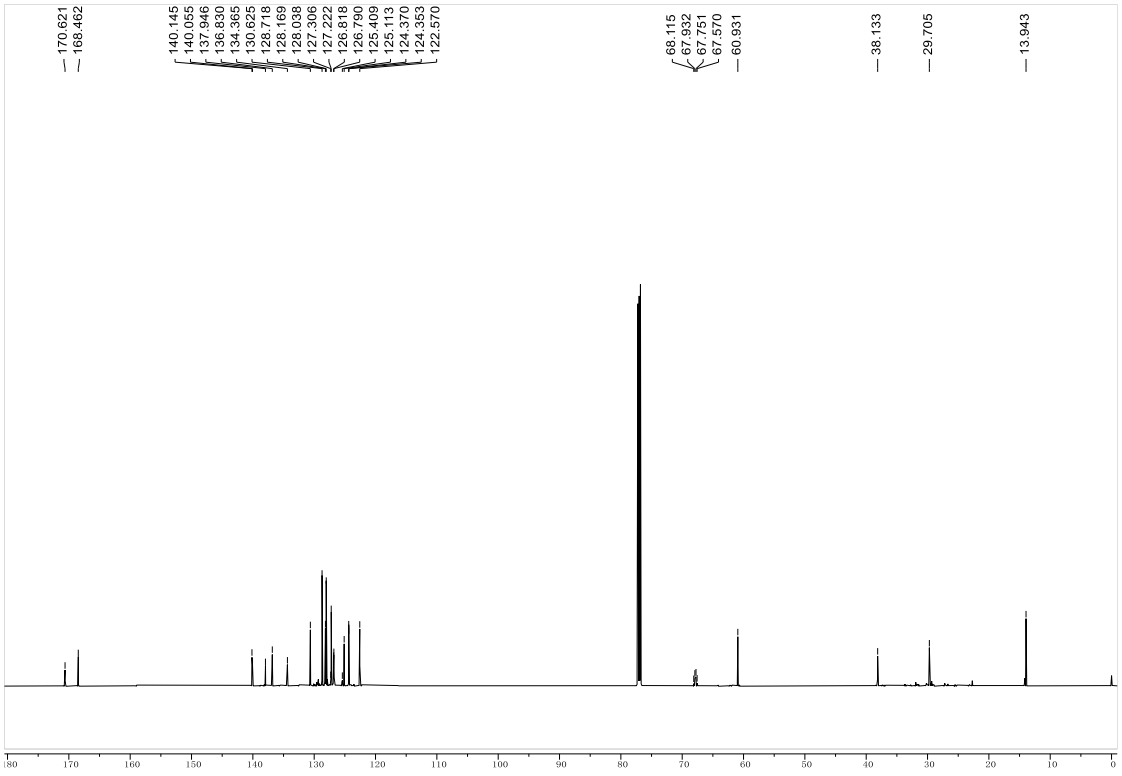




170.599
168.759
140.854
137.466
137.346
132.880
132.591
130.546
128.879
128.445
128.271
128.178
128.025
127.881
127.673
127.404
127.223
126.864
126.768
126.341
125.996
125.491
123.600
68.394
68.221
68.049
67.877
60.753
36.077
29.705
13.990
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

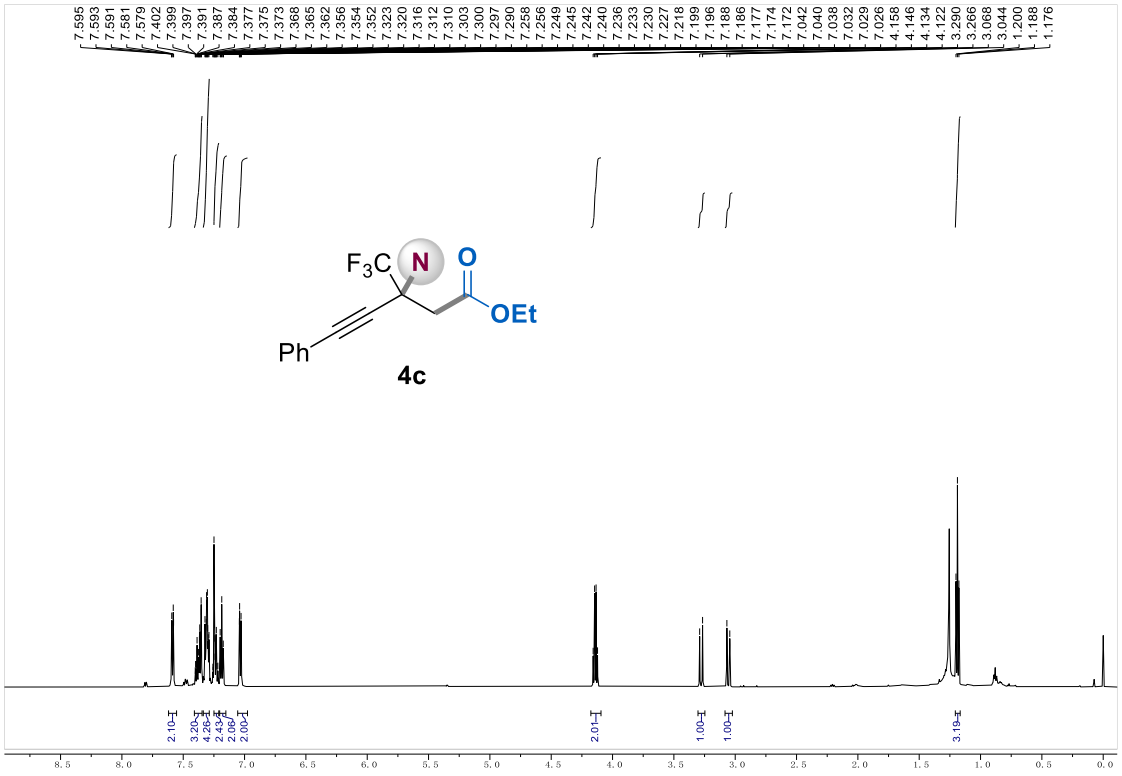
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 4b**

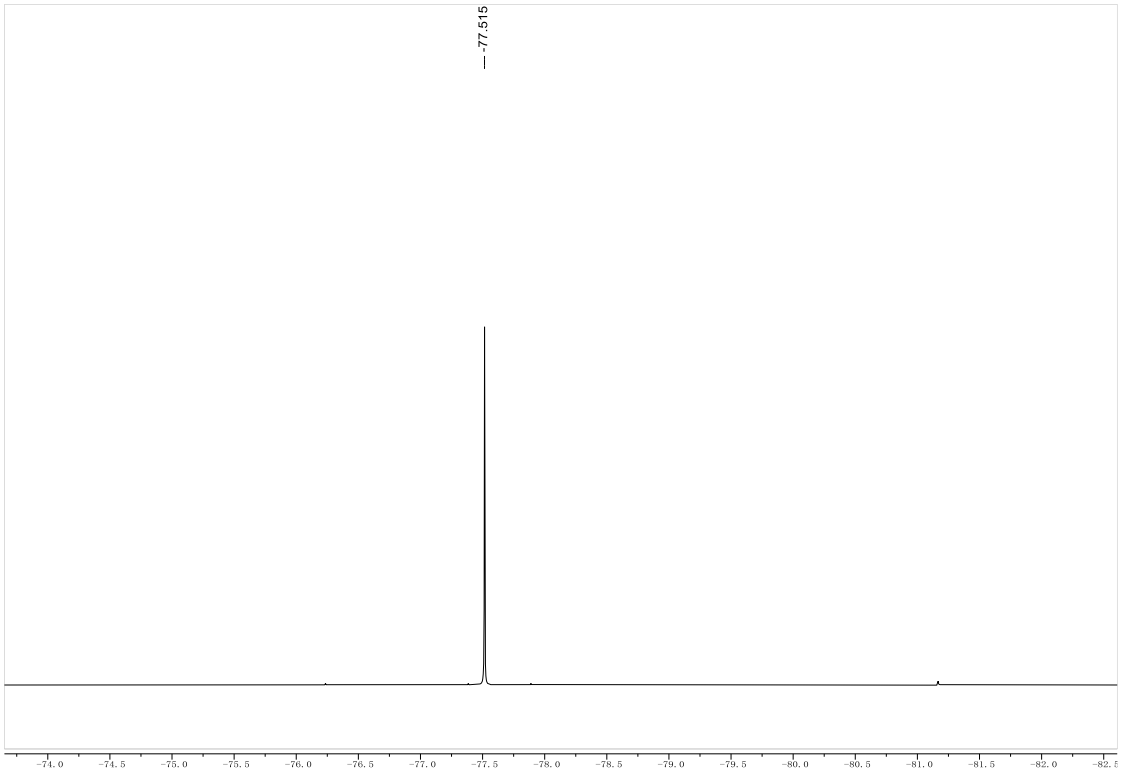


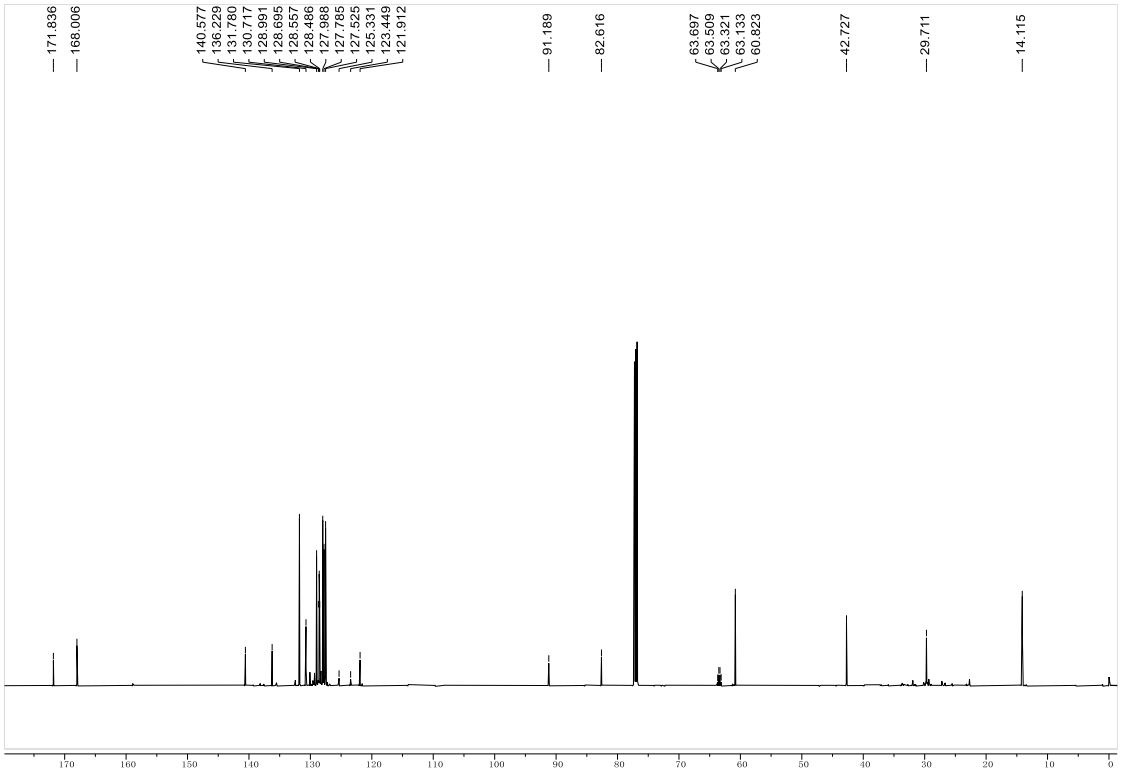




170.621
168.462
140.145
140.055
137.946
136.830
134.365
130.625
128.718
128.169
128.038
127.306
127.222
126.818
126.790
125.409
125.113
124.370
124.353
122.570
68.115
67.932
67.751
67.570
60.931
38.133
29.705
13.943
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

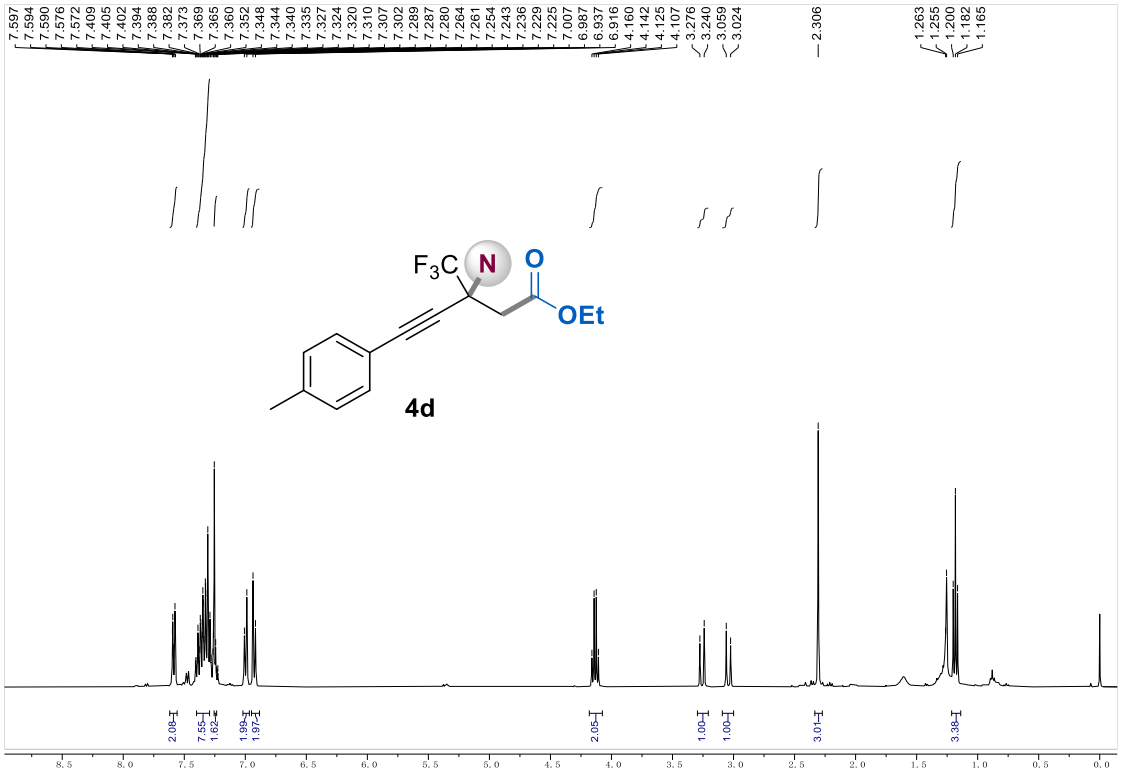
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 4c**

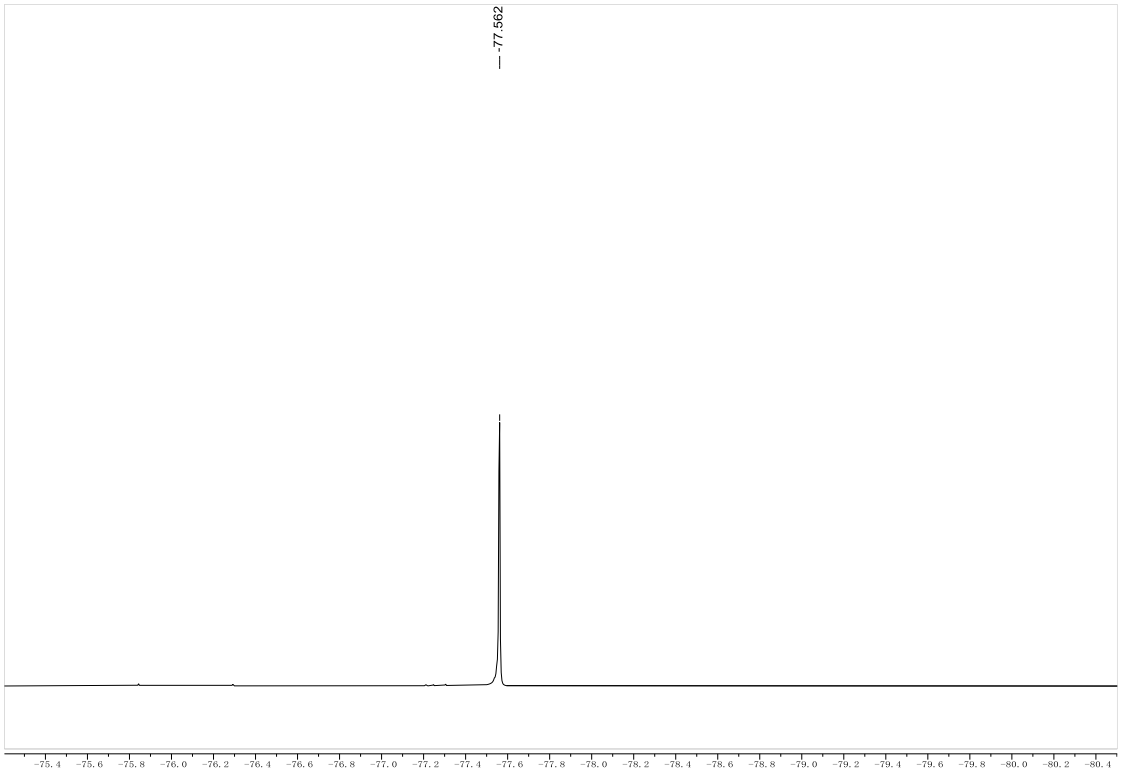


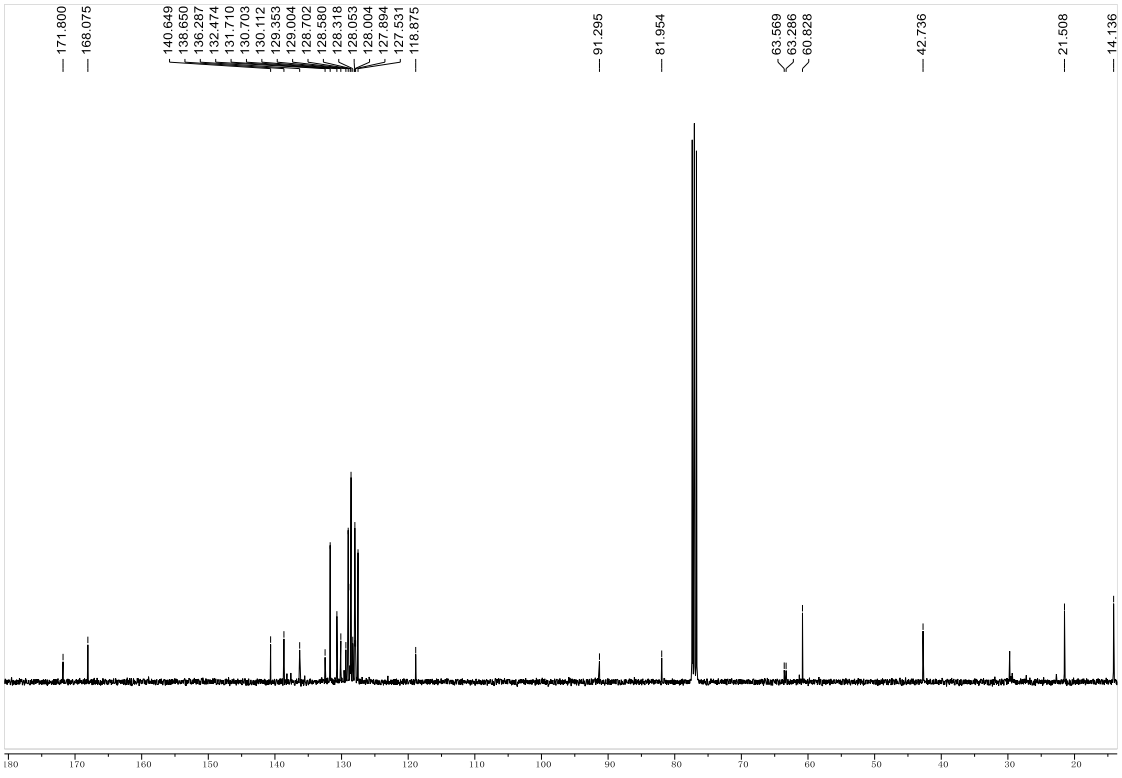




171.836
168.006
140.577
136.229
131.780
130.717
128.991
128.695
128.557
128.486
127.988
127.785
127.525
125.331
123.449
121.912
91.189
82.616
63.697
63.509
63.321
63.133
60.823
42.727
29.711
14.115
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

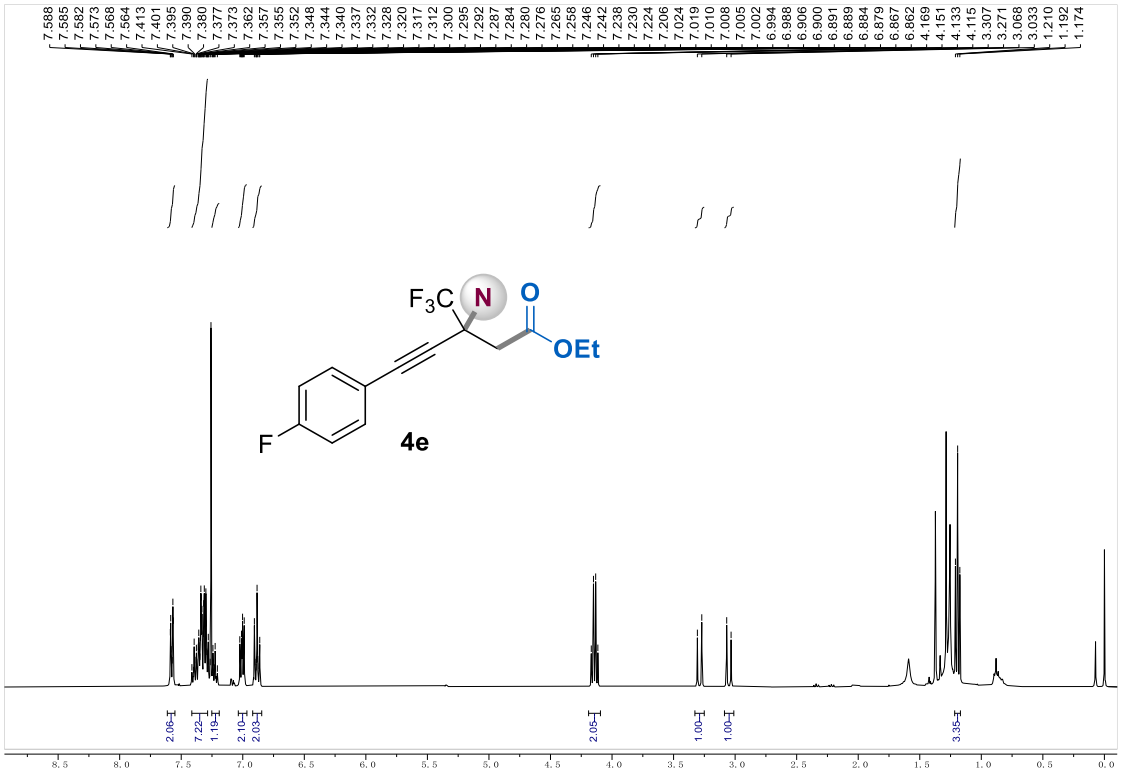
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4d**

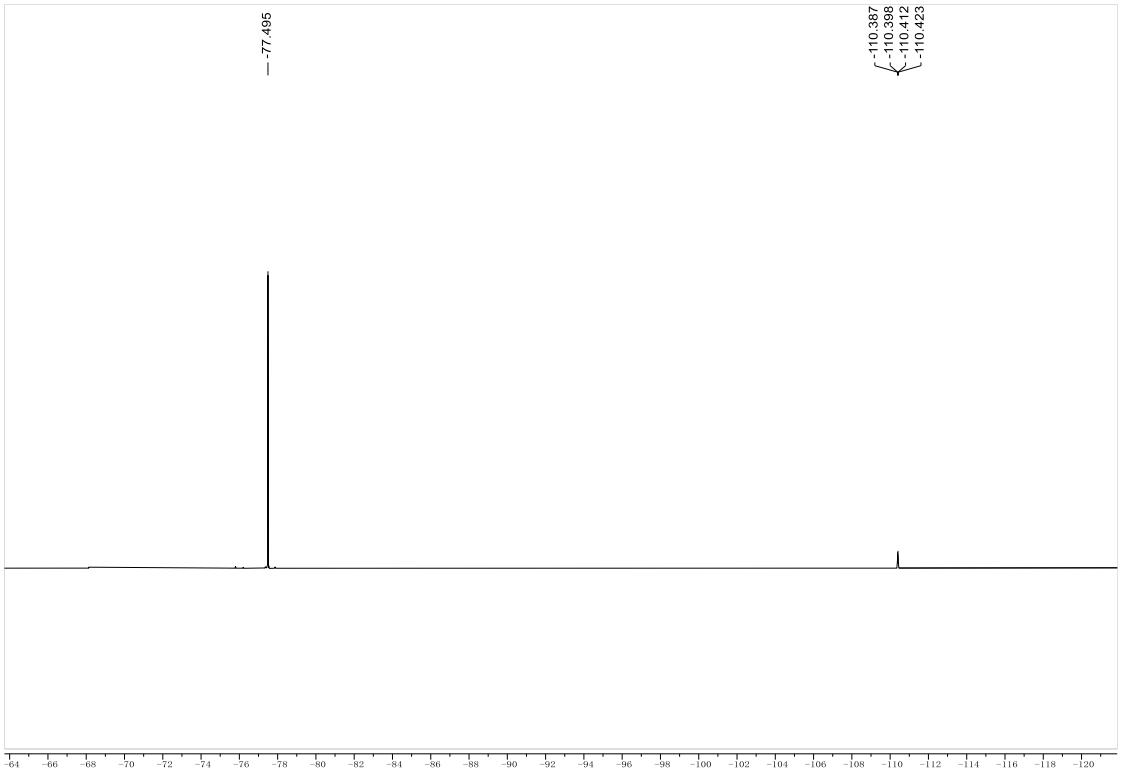


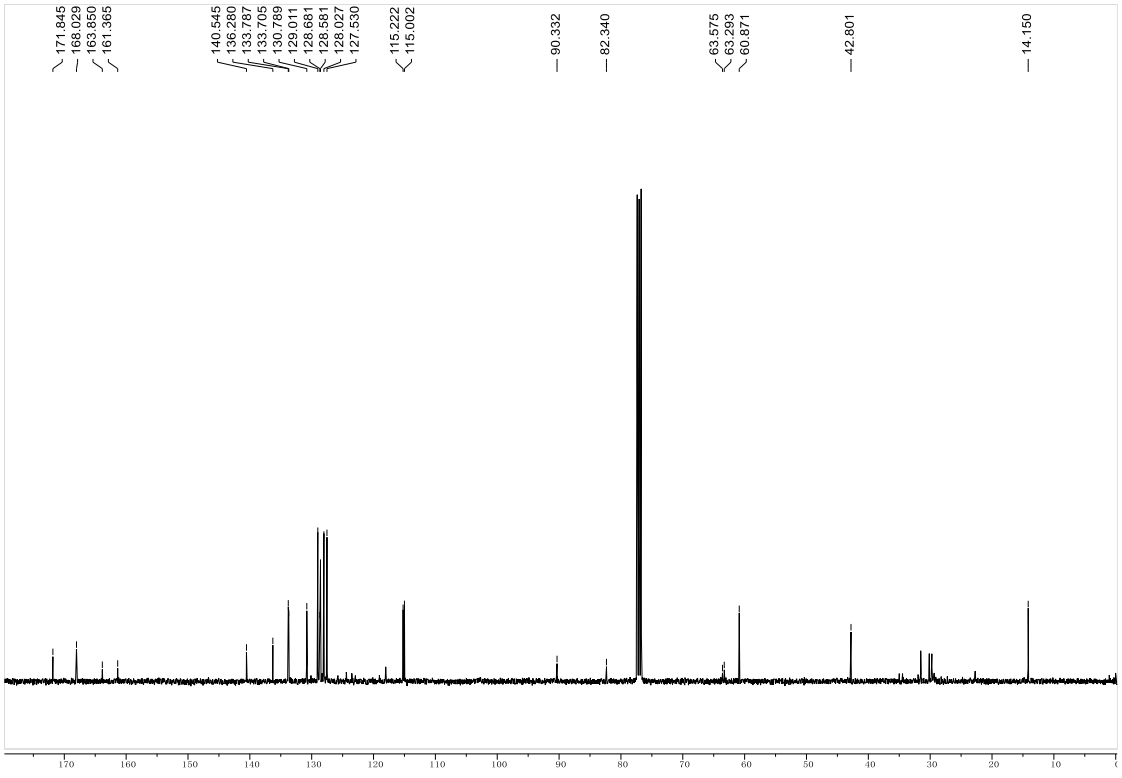




171.800
168.075
140.649
138.650
136.287
132.474
131.710
130.703
130.112
129.353
129.004
128.702
128.580
128.318
128.053
128.004
127.894
127.531
118.875
91.295
81.954
63.569
63.286
60.828
42.736
21.508
14.136
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

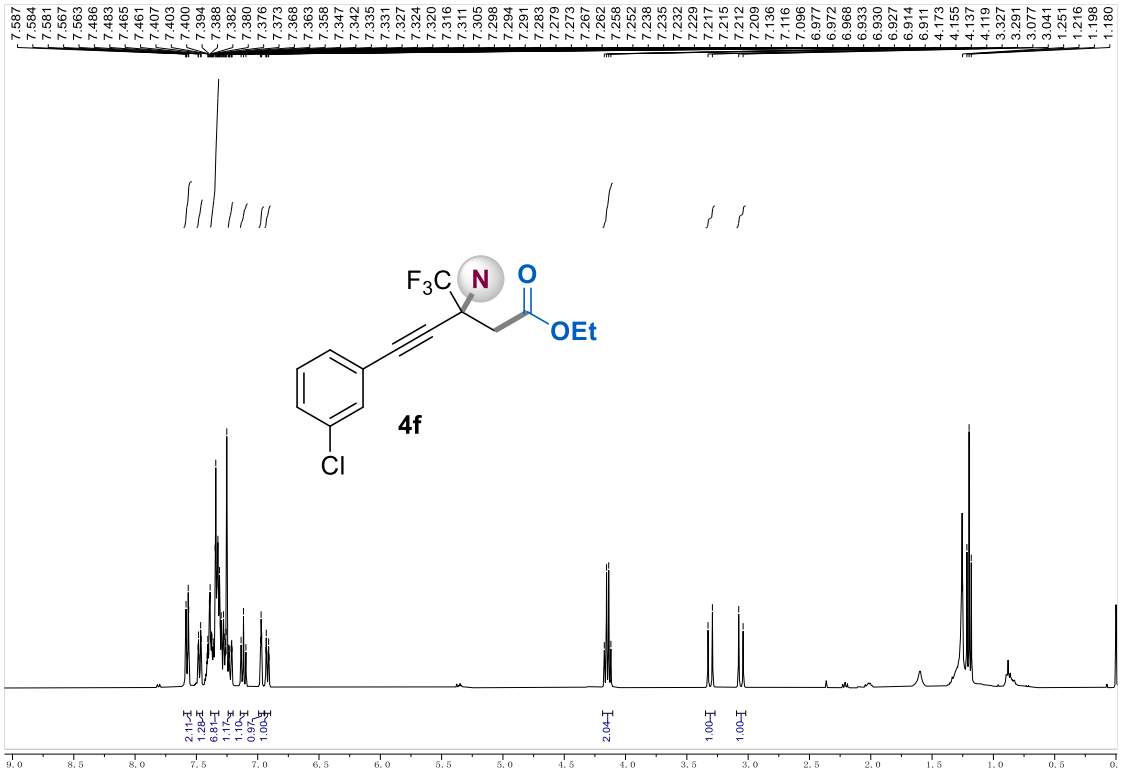
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4e**

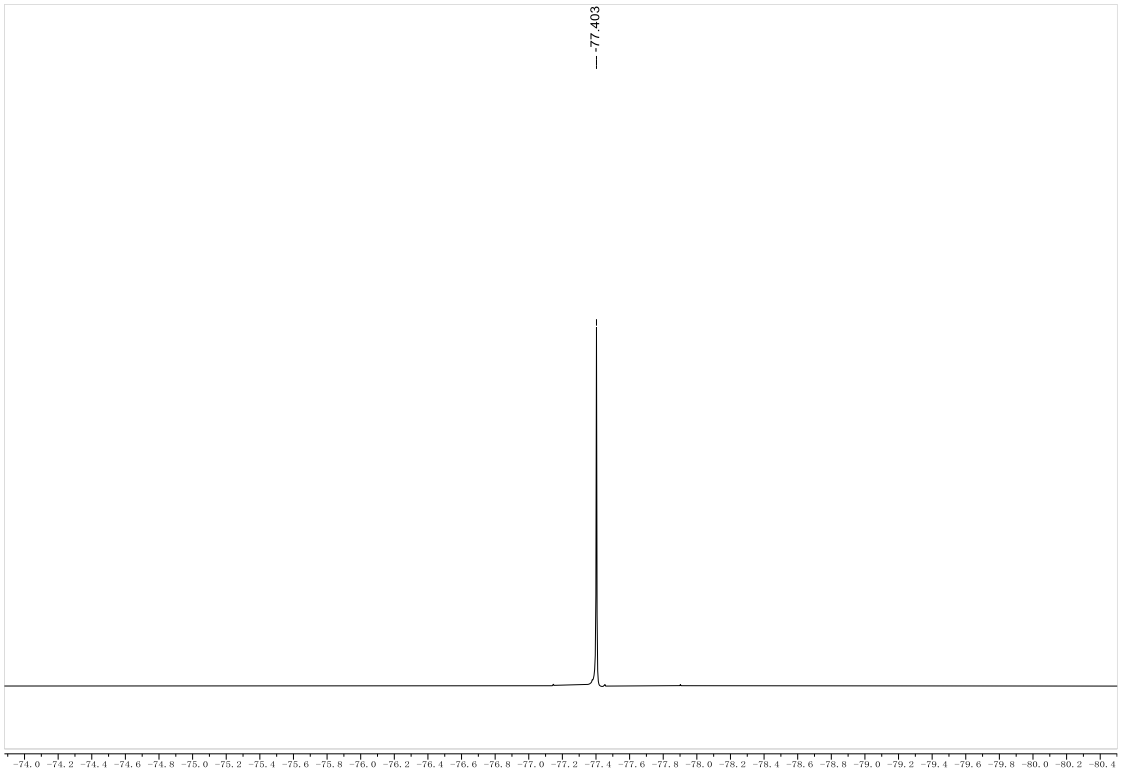


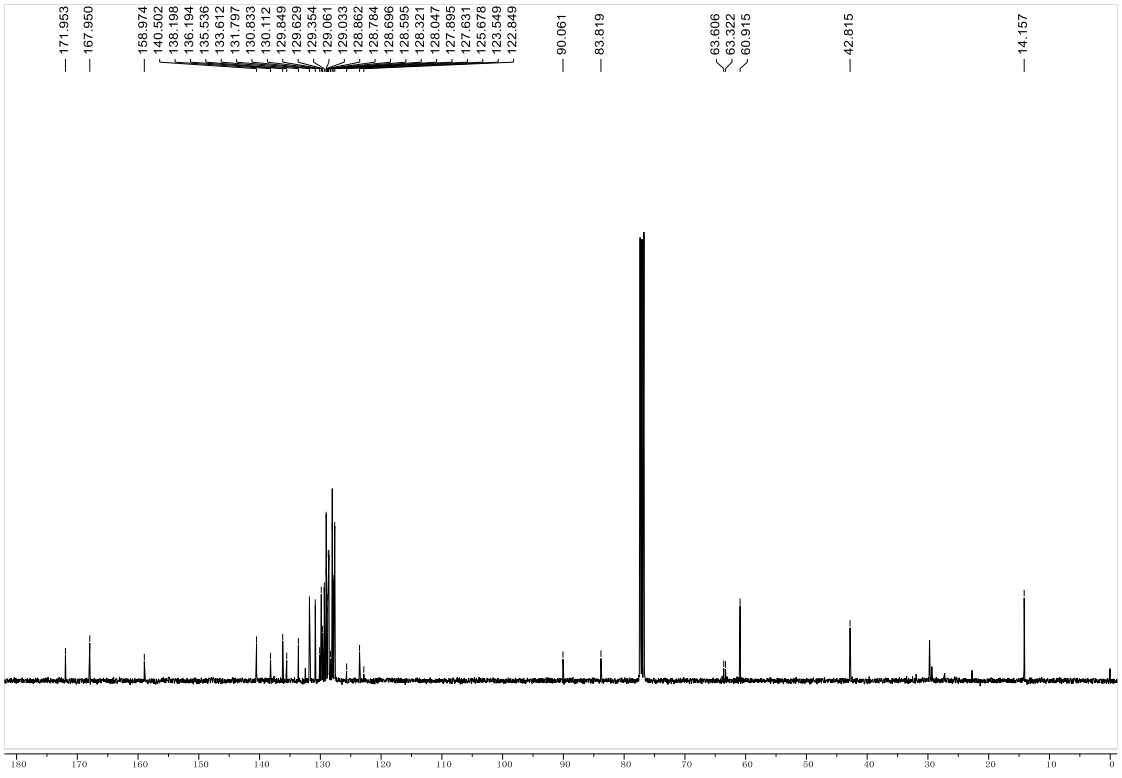




171.845
168.029
163.850
161.365
140.545
136.280
133.787
133.705
130.789
129.011
128.681
128.581
128.027
127.530
115.222
115.002
90.332
82.340
63.575
63.293
60.871
42.801
14.150
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

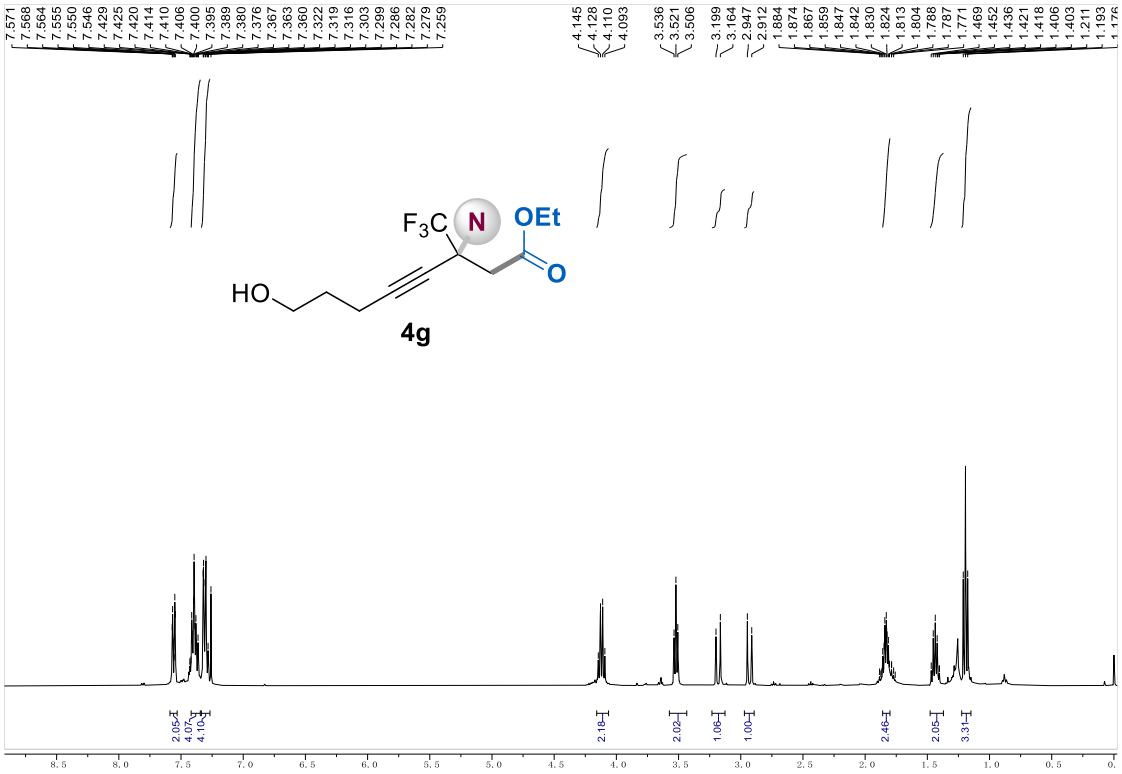
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4f**

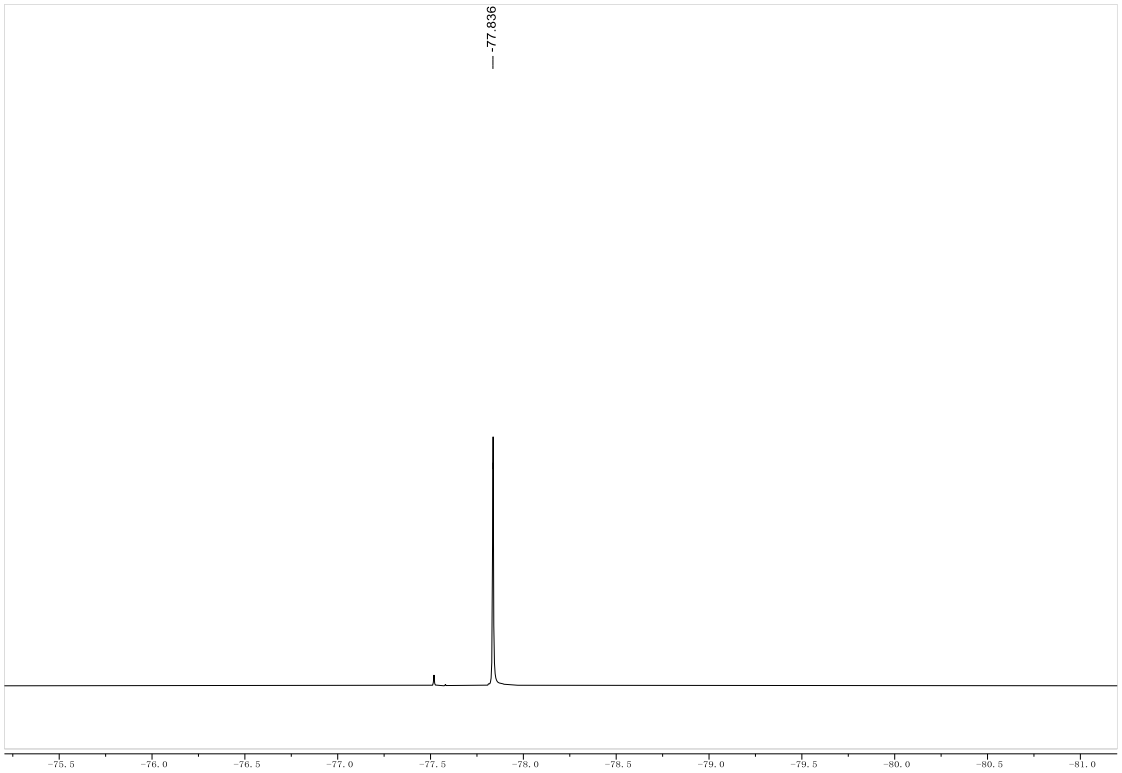


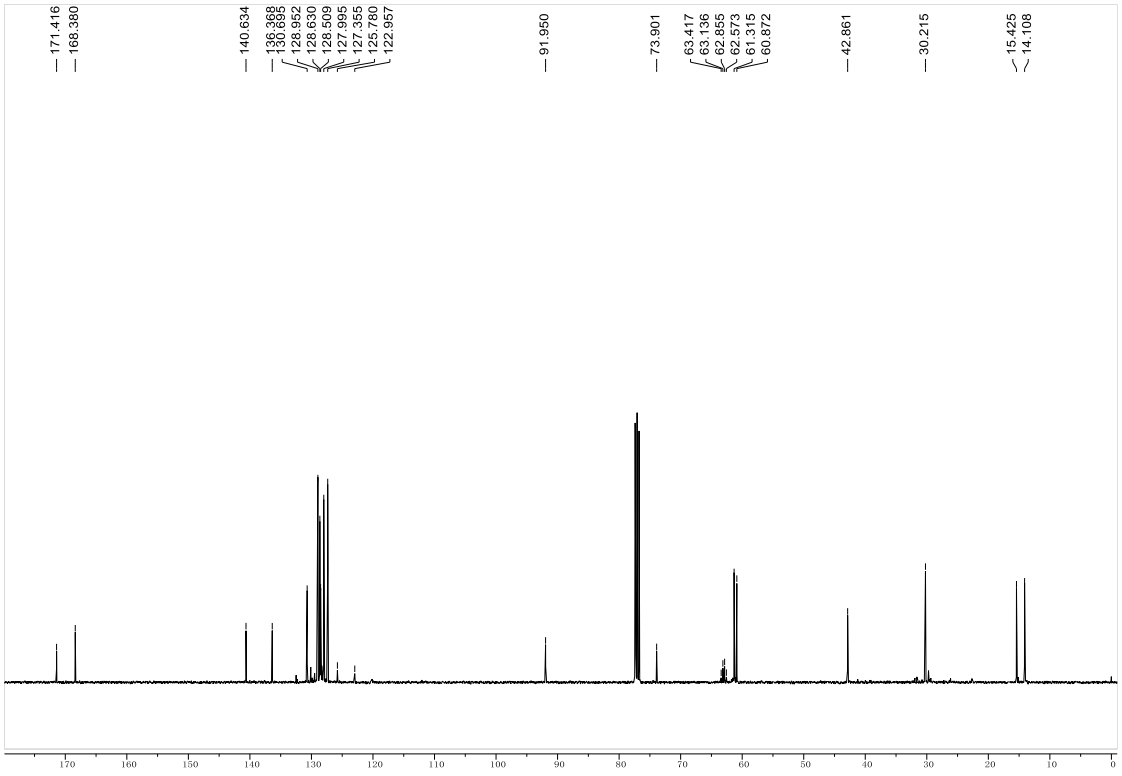




171.953
167.950
158.974
140.502
138.198
136.194
135.536
133.612
131.797
130.833
130.112
129.849
129.629
129.354
129.061
129.033
128.862
128.784
128.696
128.595
128.321
128.047
127.895
127.631
125.678
123.549
122.849
90.061
83.819
63.606
63.322
60.915
42.815
14.157
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

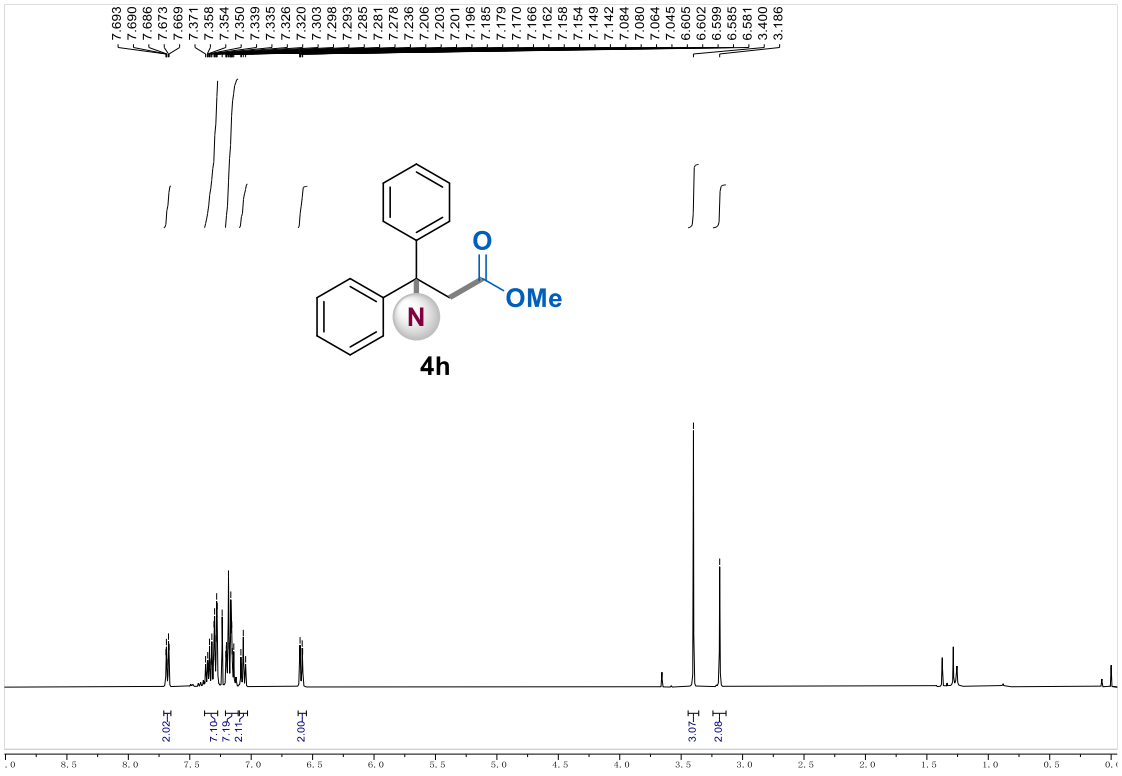
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4g**

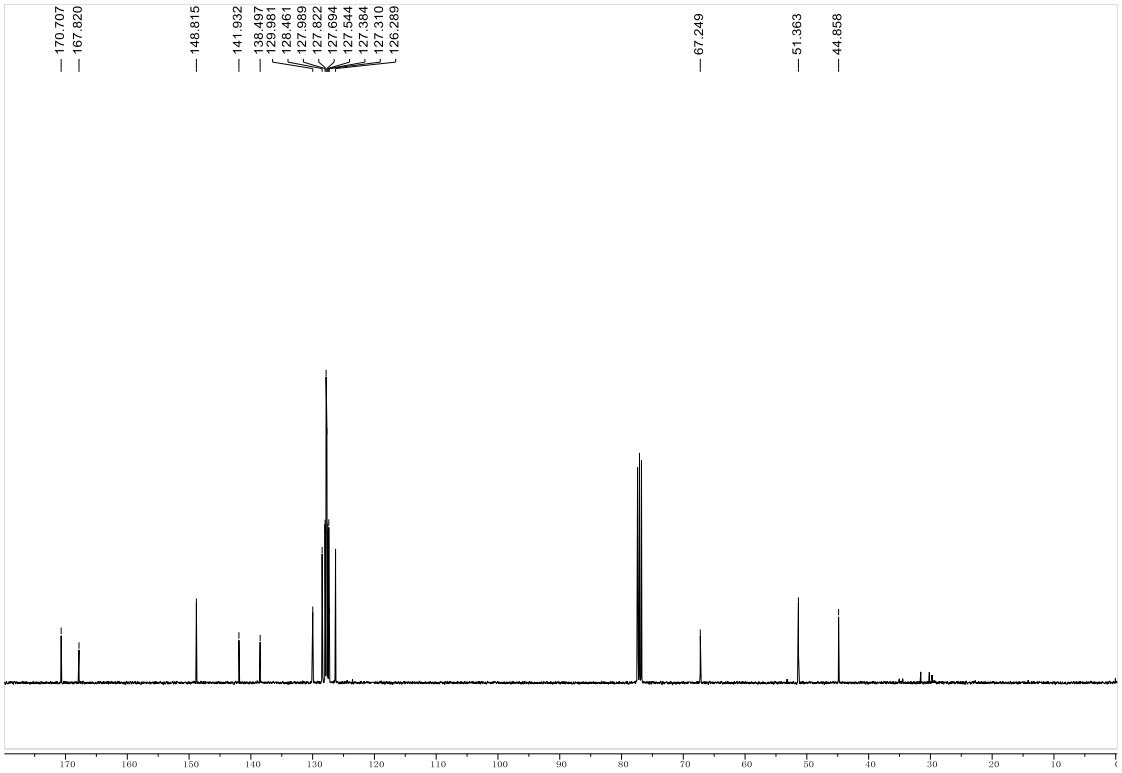


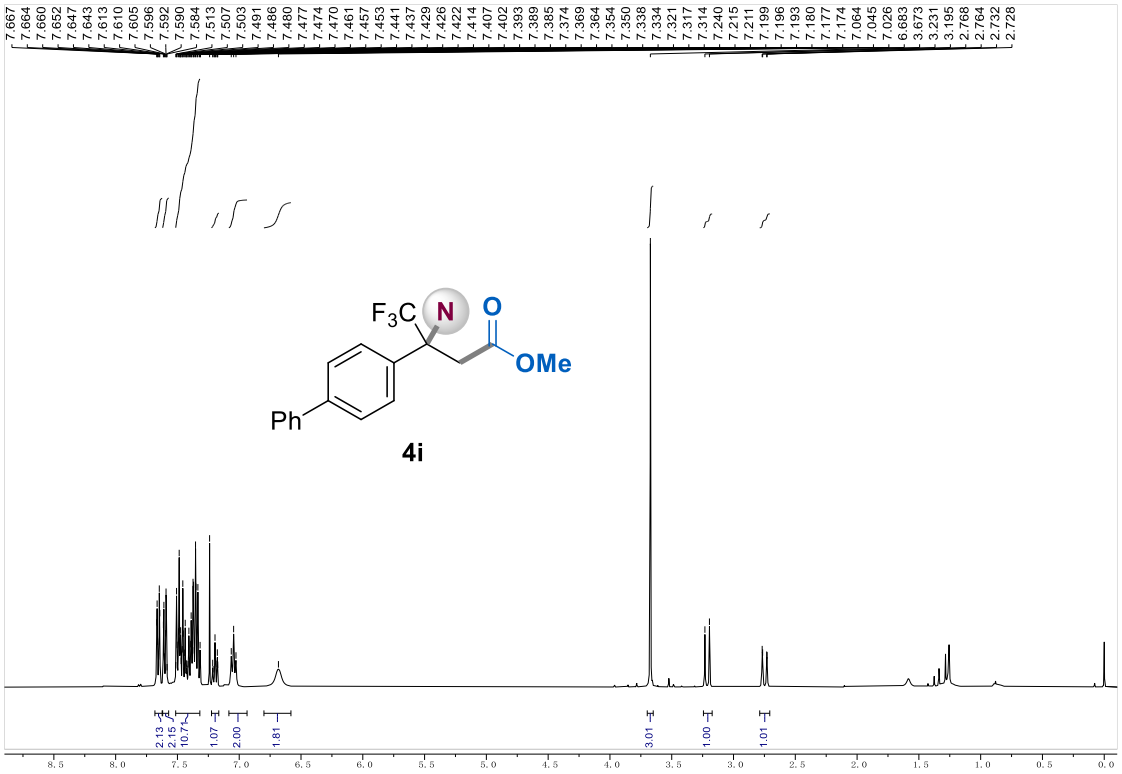

171.416
168.380
140.634
136.868
136.385
128.952
128.630
128.509
127.995
127.355
125.780
122.957
91.950
73.901
63.417
63.136
62.855
62.573
61.315
60.872
42.861
30.215
15.425
14.108
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

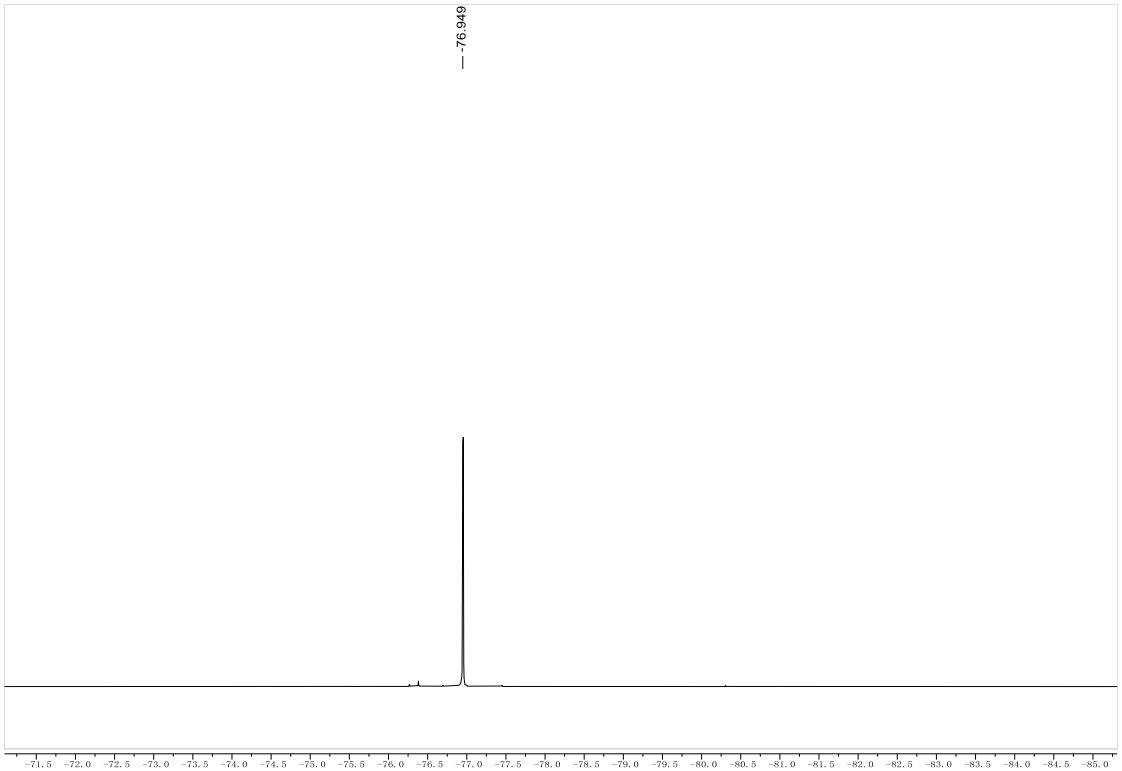
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4h**

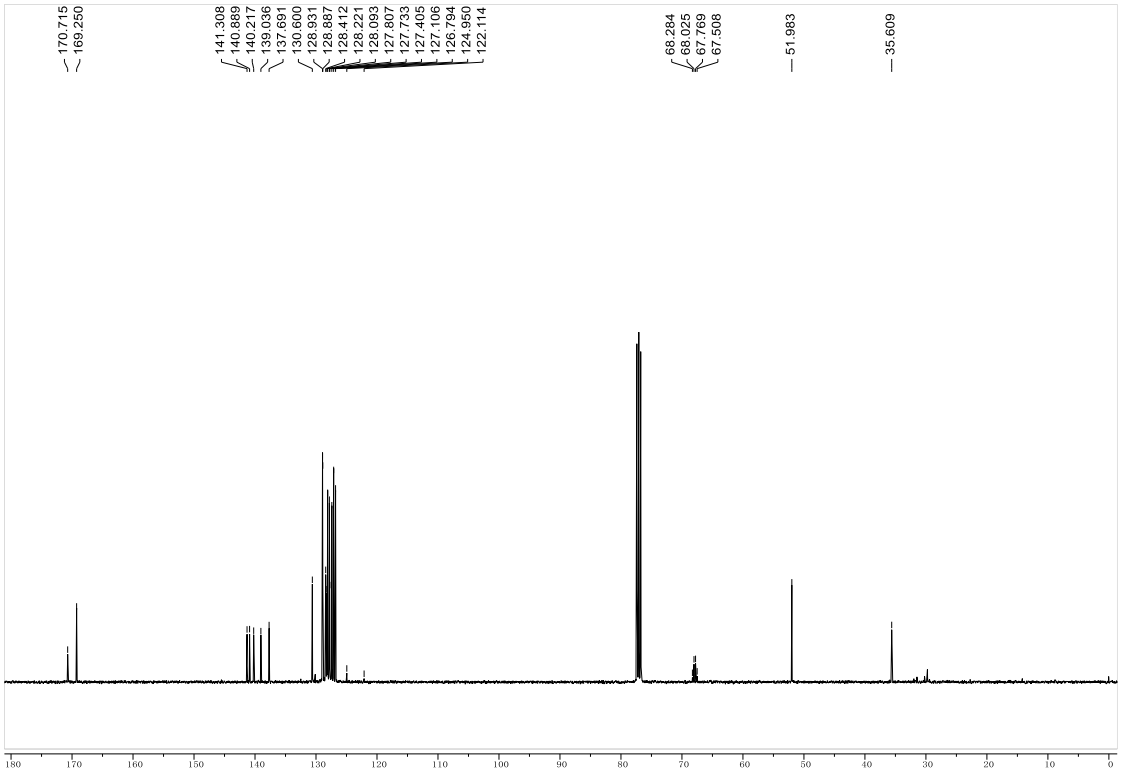


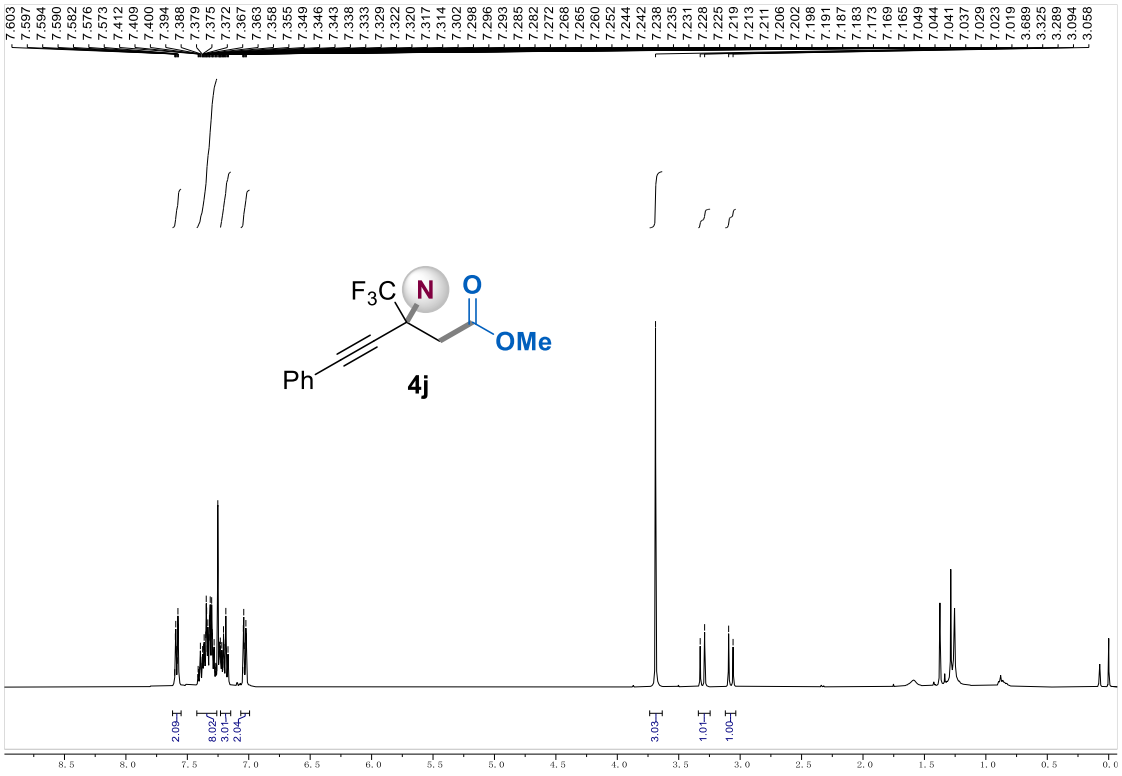
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4i**

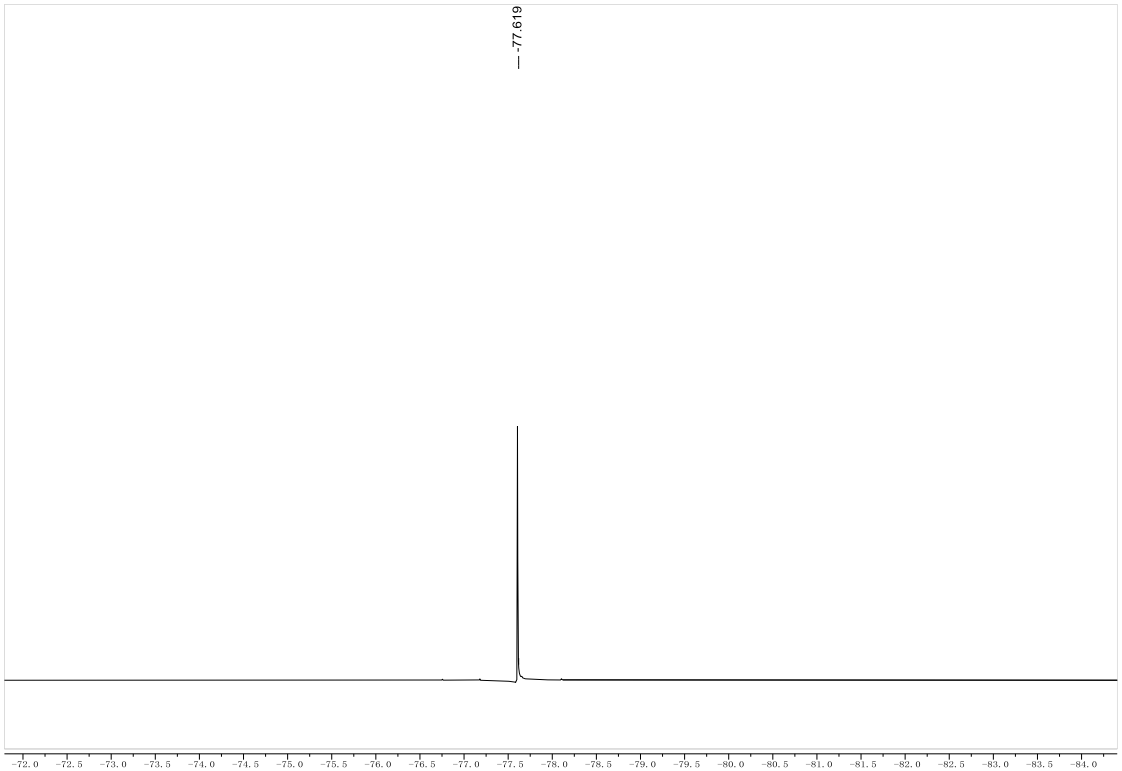


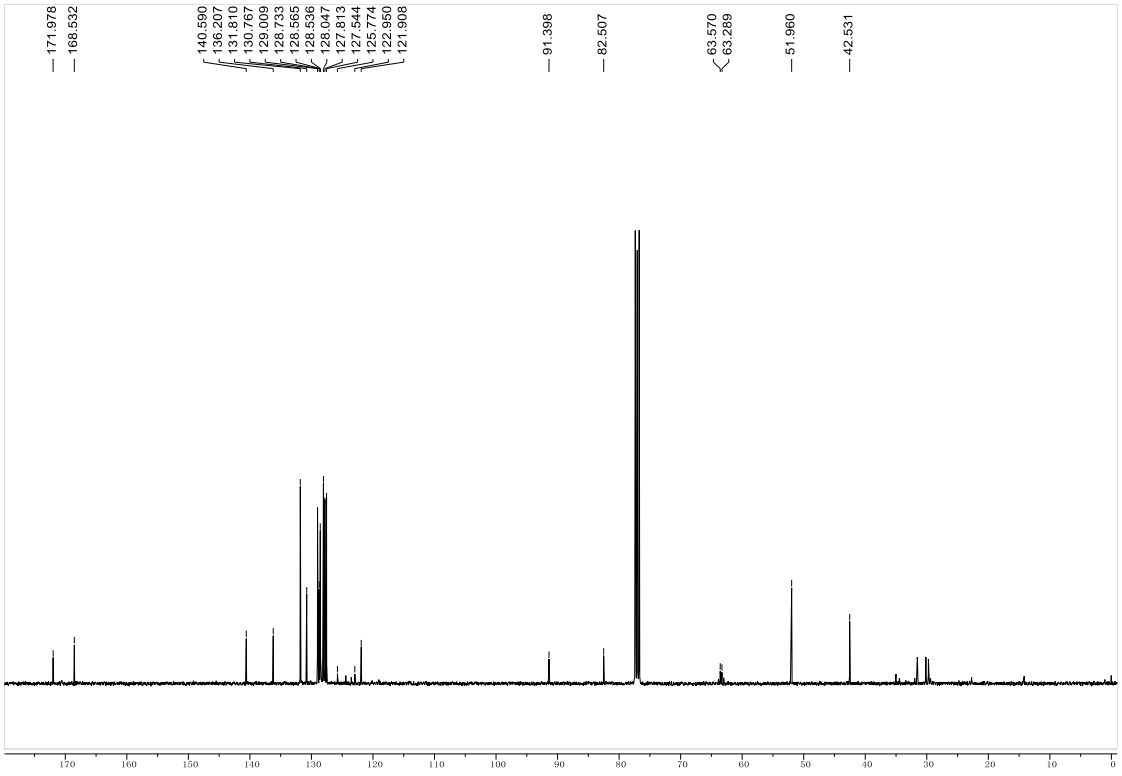




170.715
169.250
141.308
140.889
140.217
139.036
137.691
130.600
128.931
128.887
128.412
128.221
128.093
127.807
127.733
127.405
127.106
126.794
124.950
122.114
68.284
68.025
67.769
67.508
51.983
35.609
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

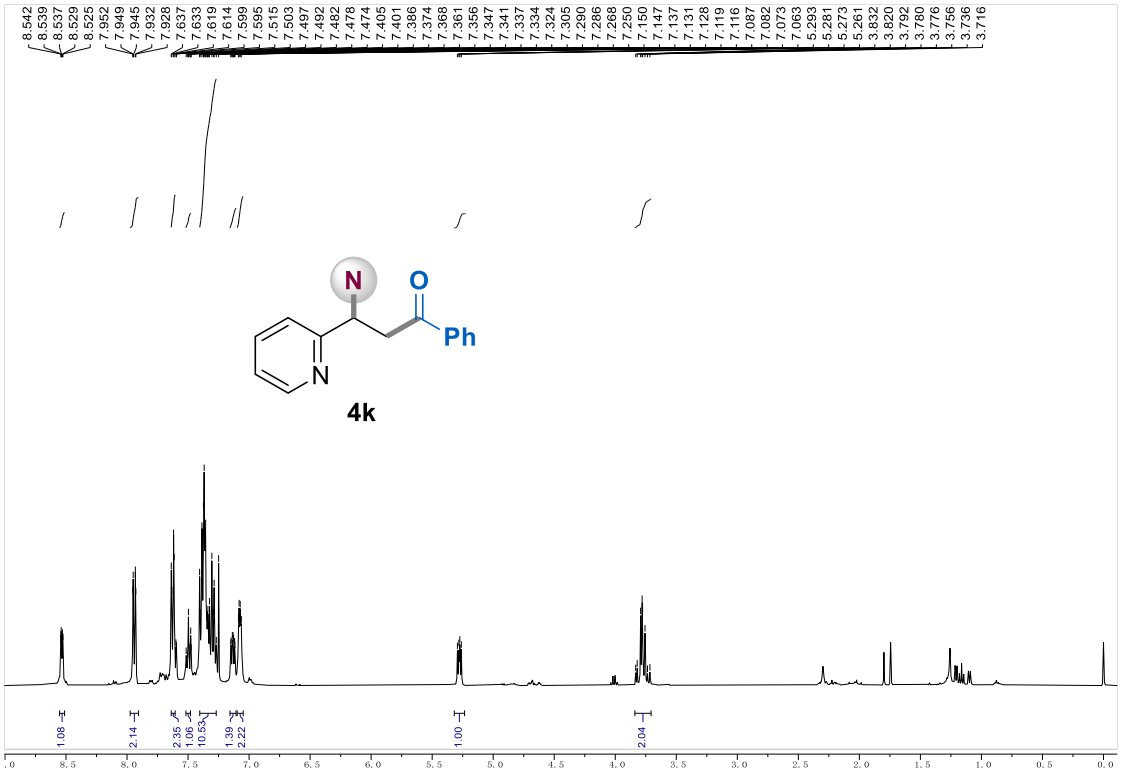
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4j**

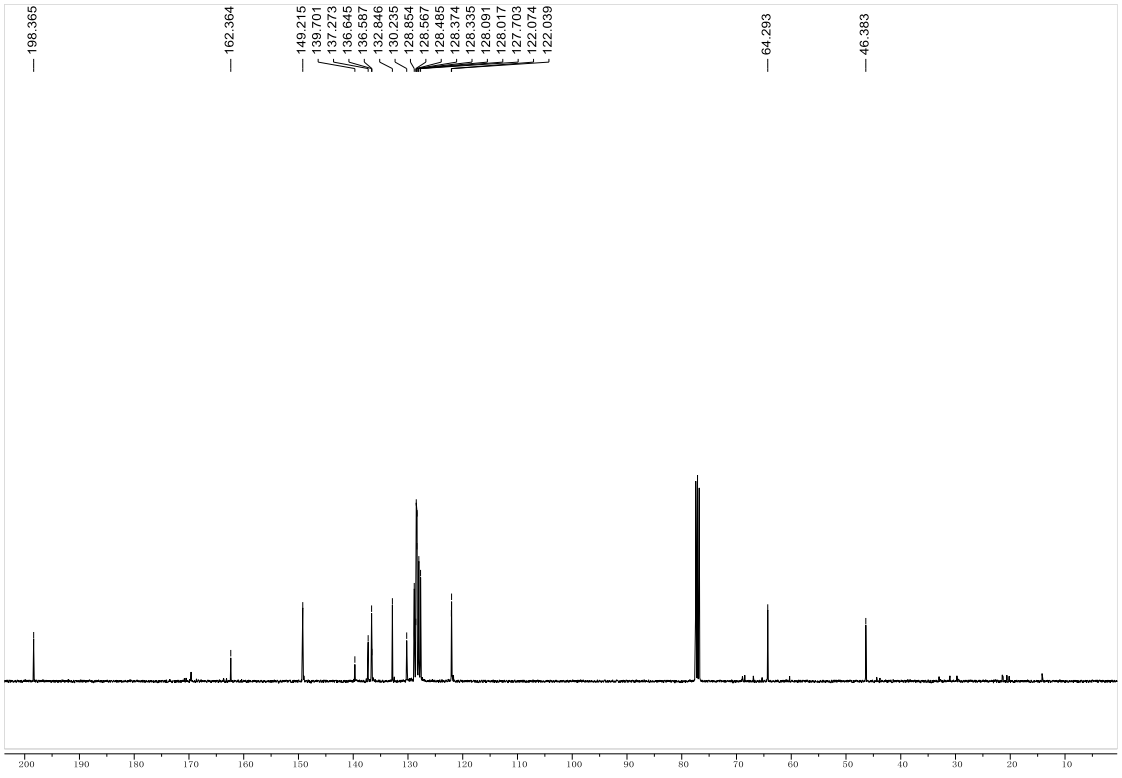


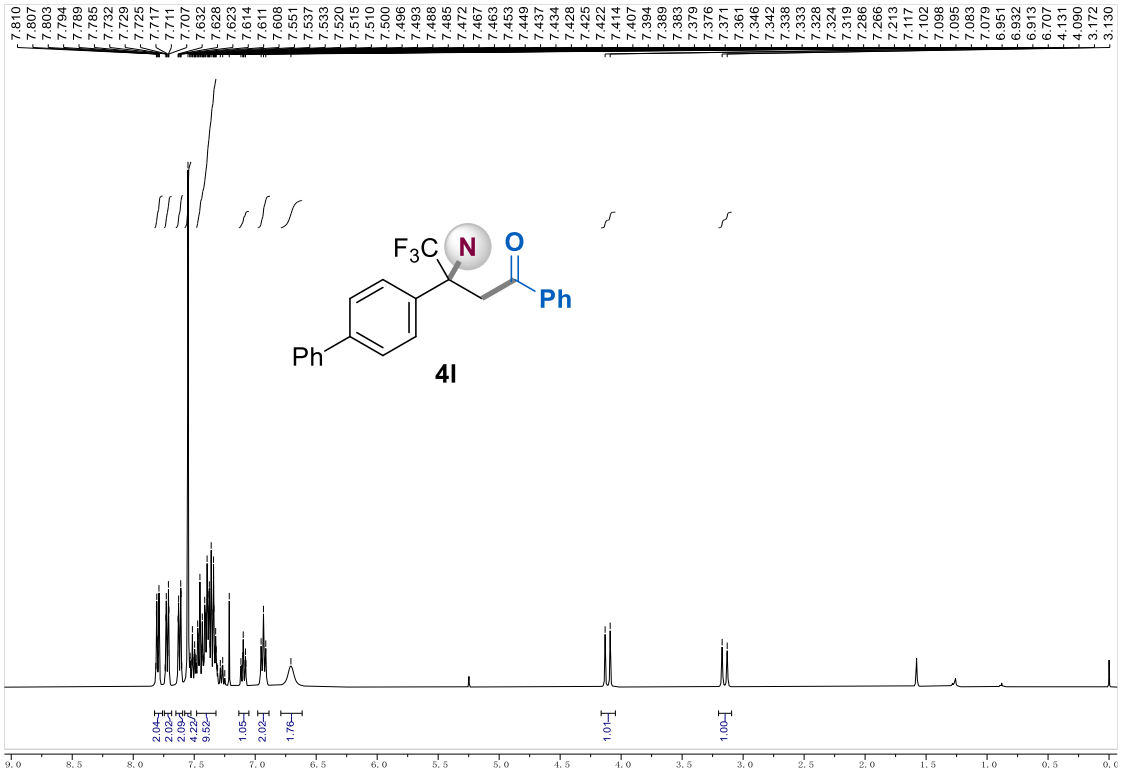

171.978
168.532
140.590
136.207
131.810
130.767
129.009
128.733
128.565
128.536
128.047
127.813
127.544
125.774
122.950
121.908
91.398
82.507
63.570
63.289
51.960
42.531
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

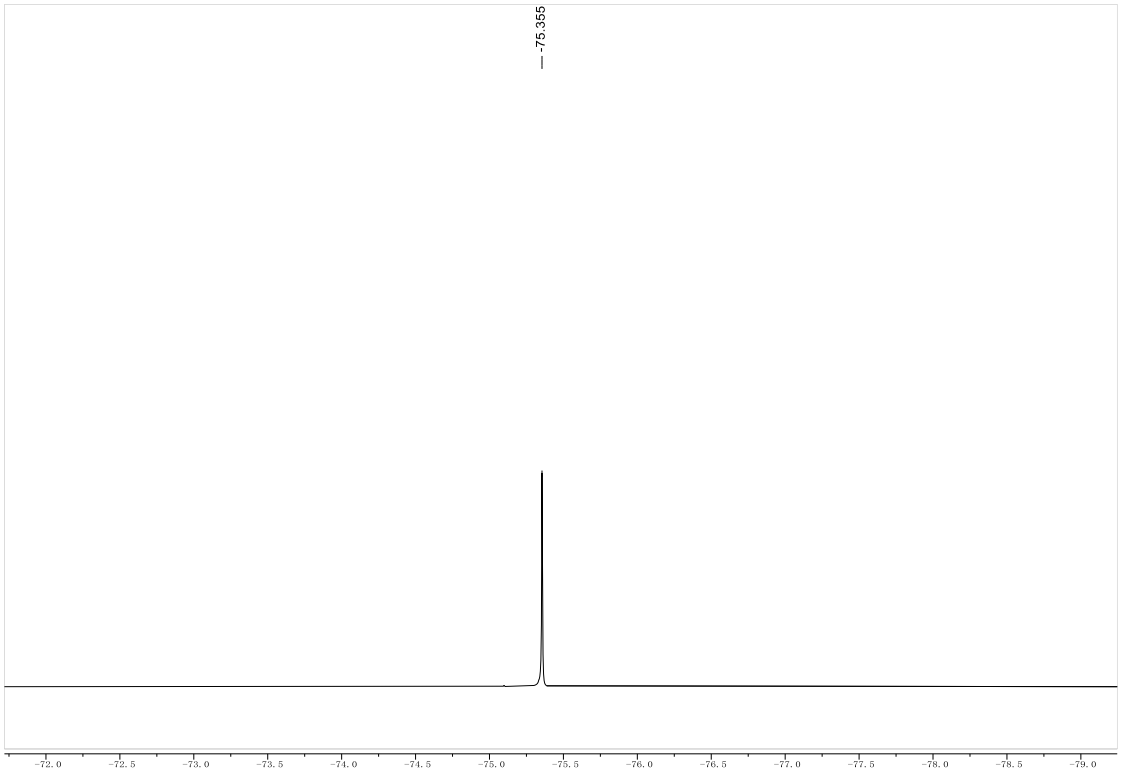
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4k**

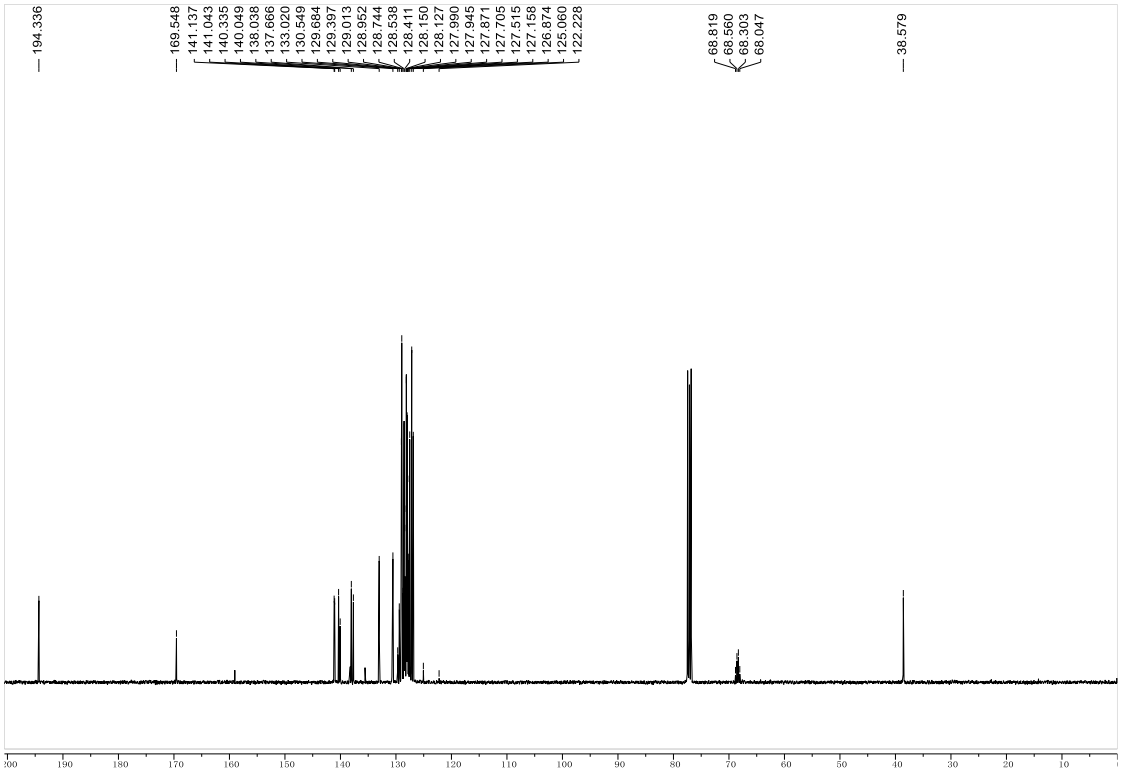


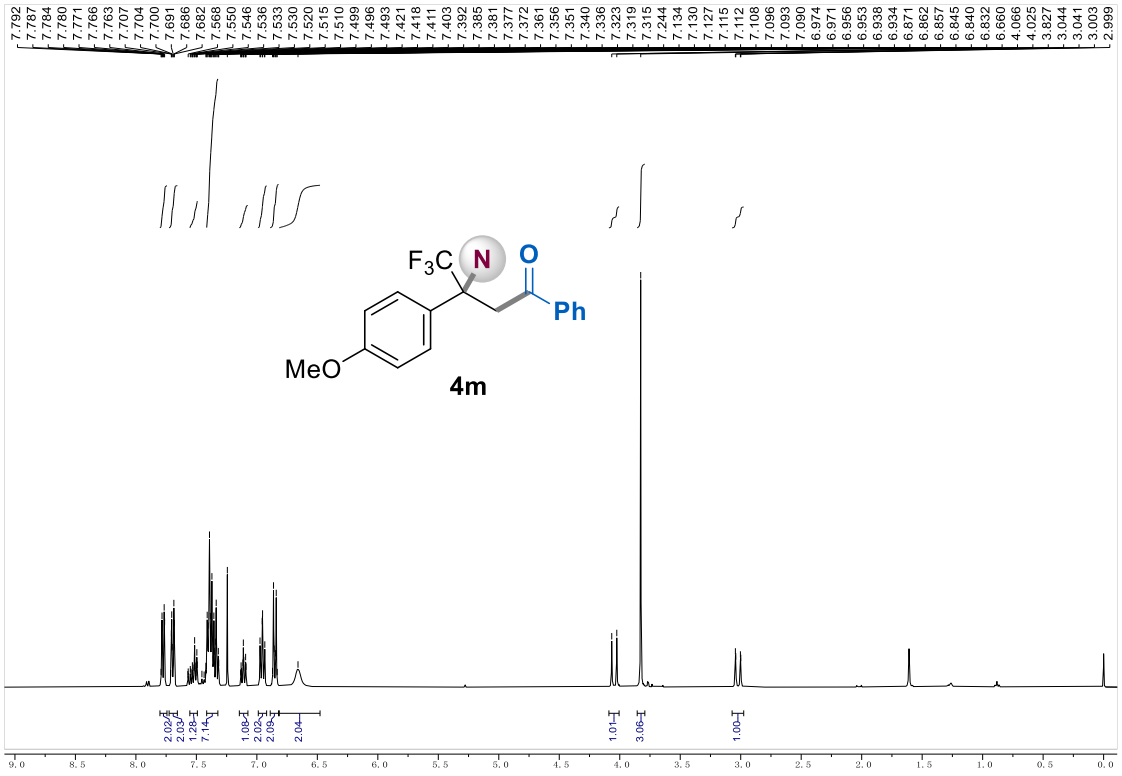
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4l**

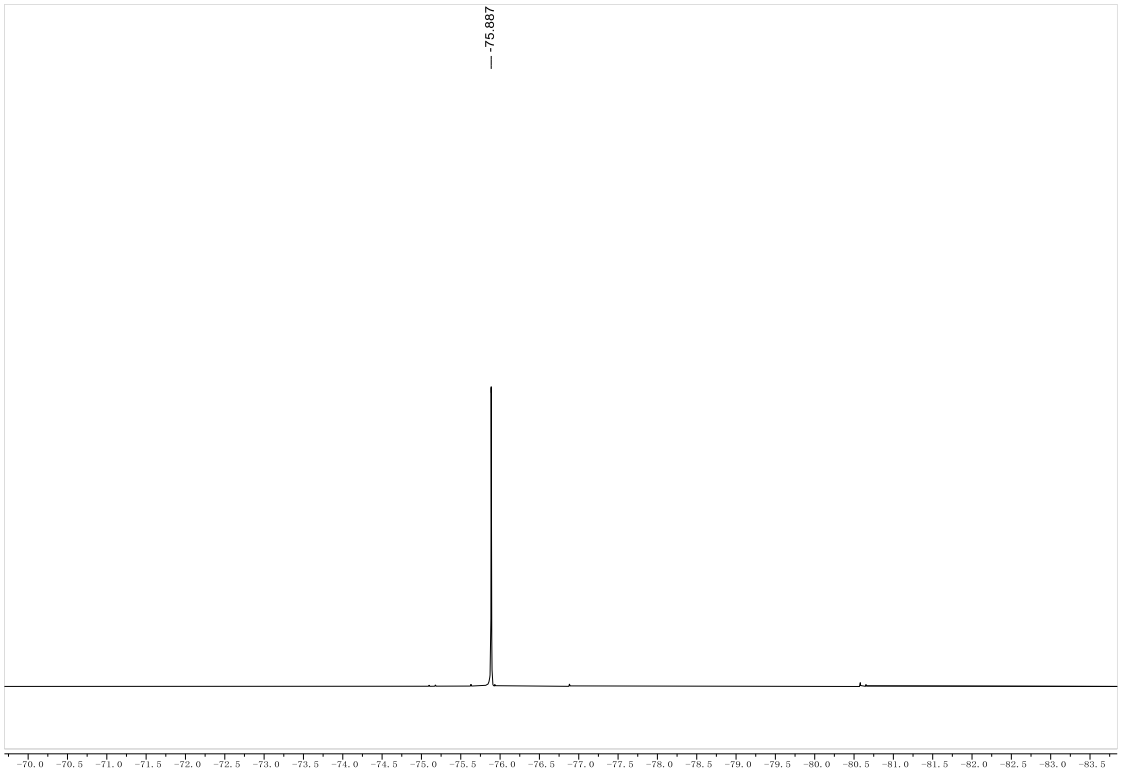


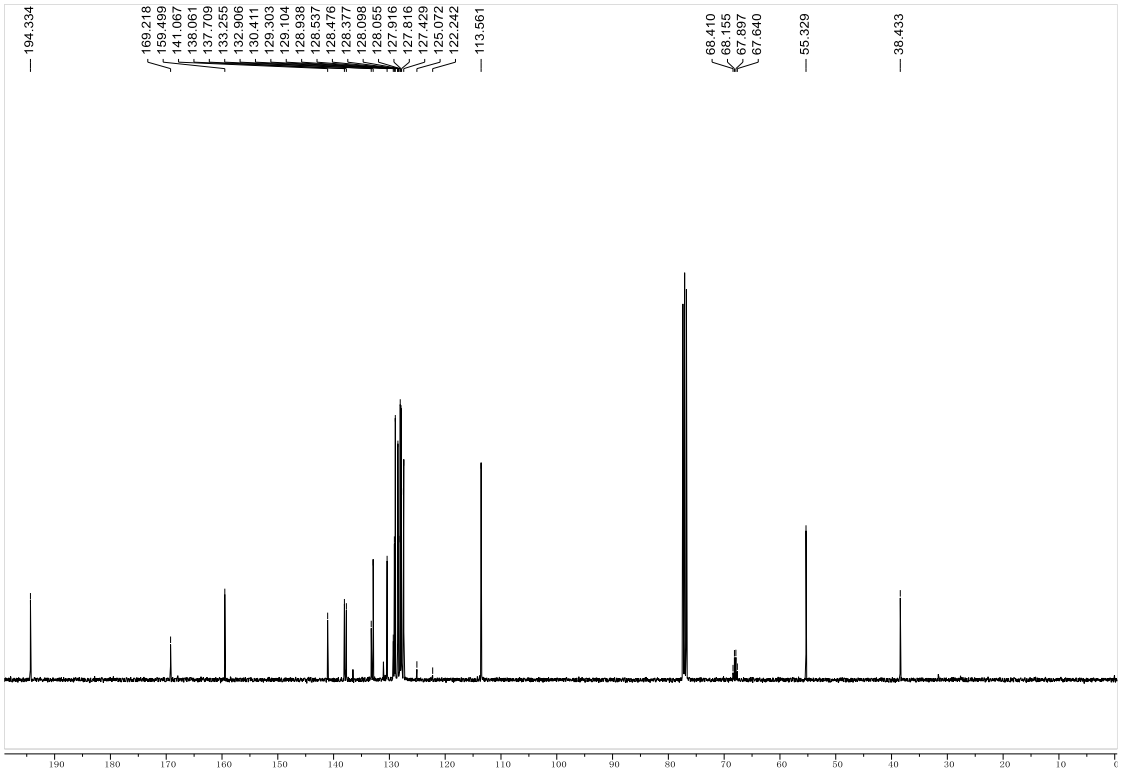




194.336
169.548
141.137
141.043
140.335
140.049
138.038
137.666
133.020
130.549
129.684
129.397
129.013
128.952
128.744
128.538
128.411
128.150
128.127
127.990
127.945
127.871
127.705
127.515
127.158
126.874
125.060
122.228
68.819
68.560
68.303
68.047
38.579
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

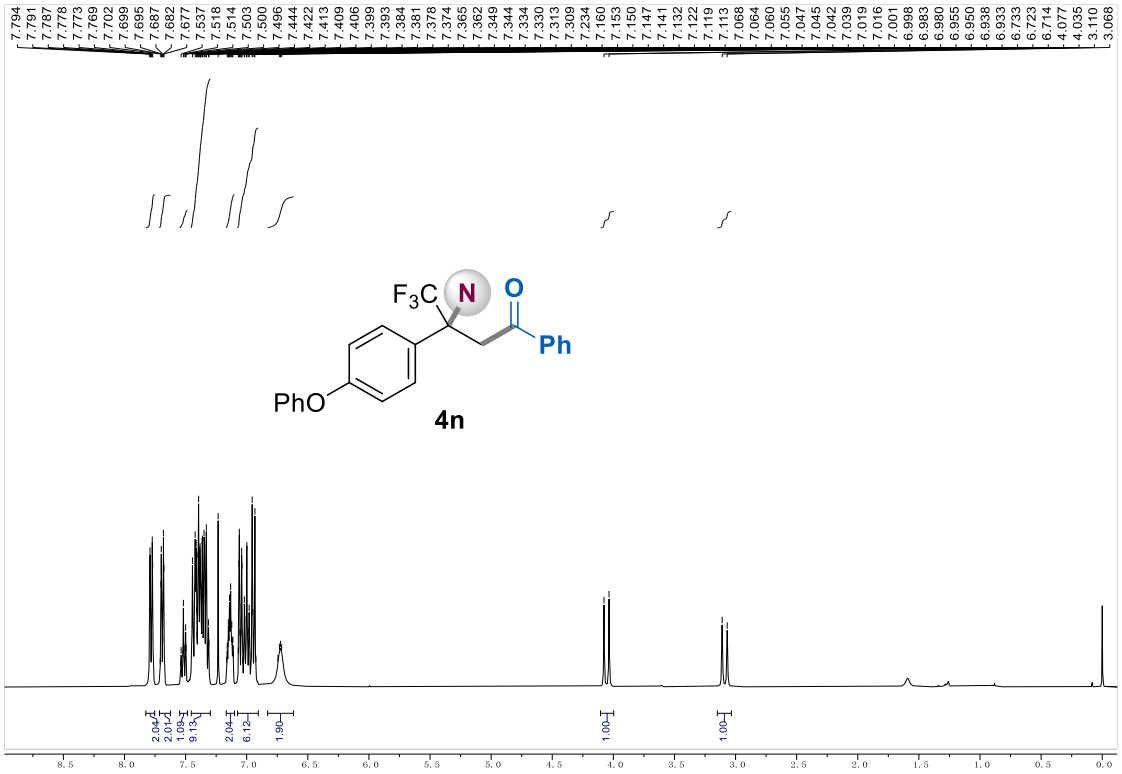
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4m**

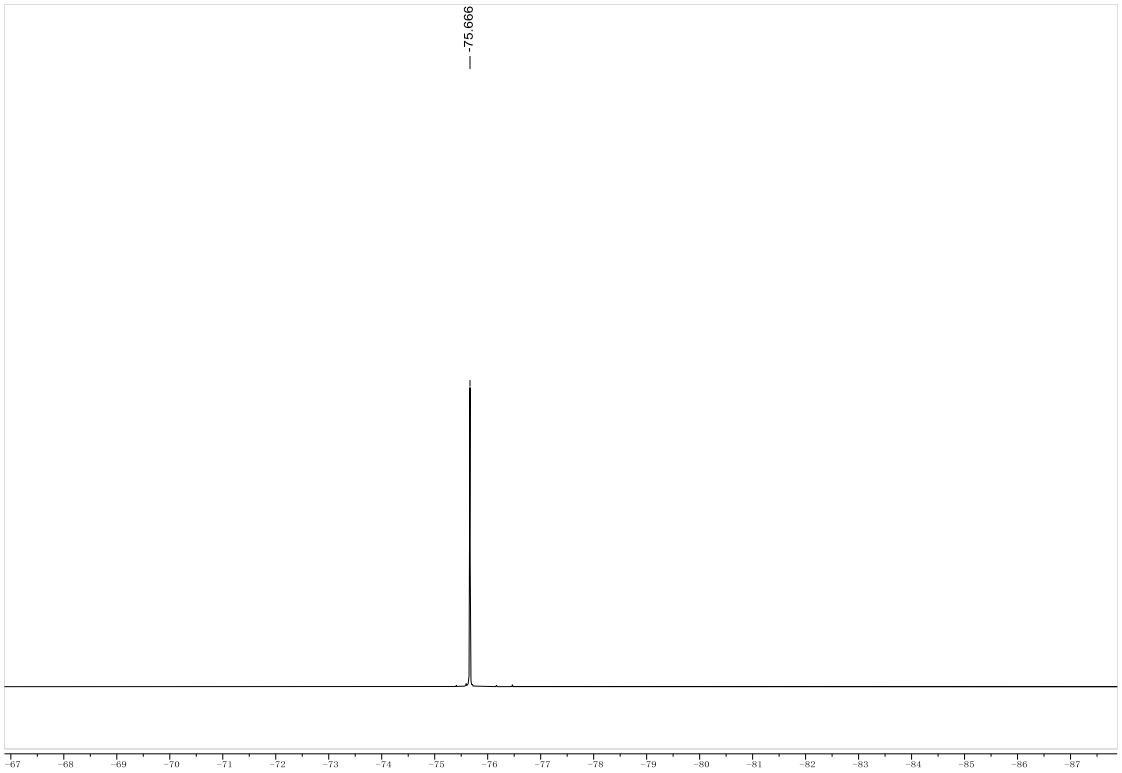


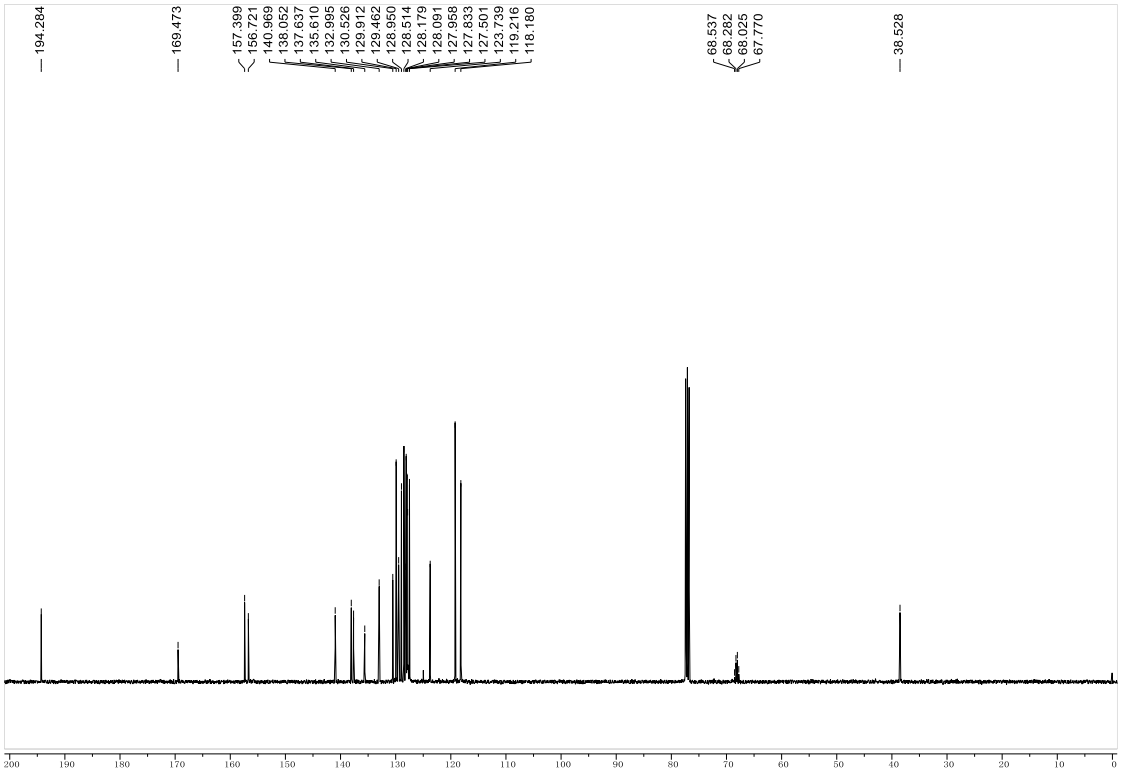




194.334
169.218
159.499
141.067
138.061
137.709
133.255
132.906
130.411
129.303
129.104
128.938
128.537
128.476
128.377
128.098
128.055
127.916
127.816
127.429
125.072
122.242
113.561
68.410
68.155
67.897
67.640
55.329
38.433
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

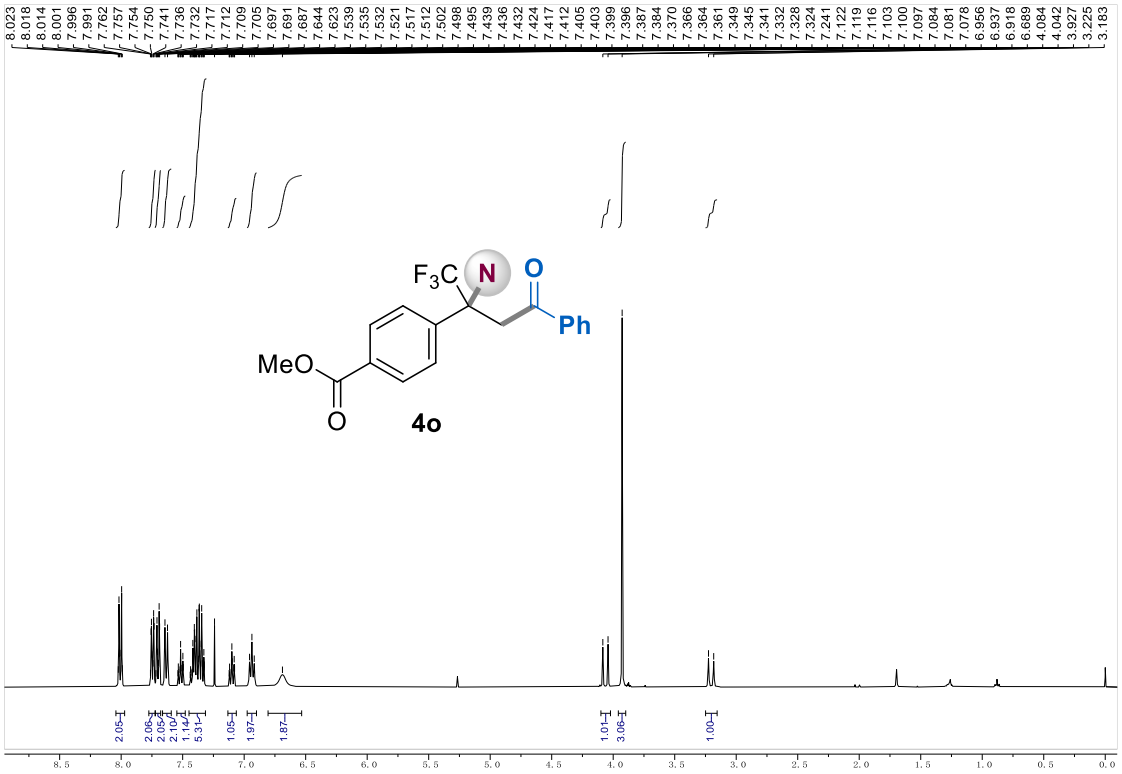
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4n**

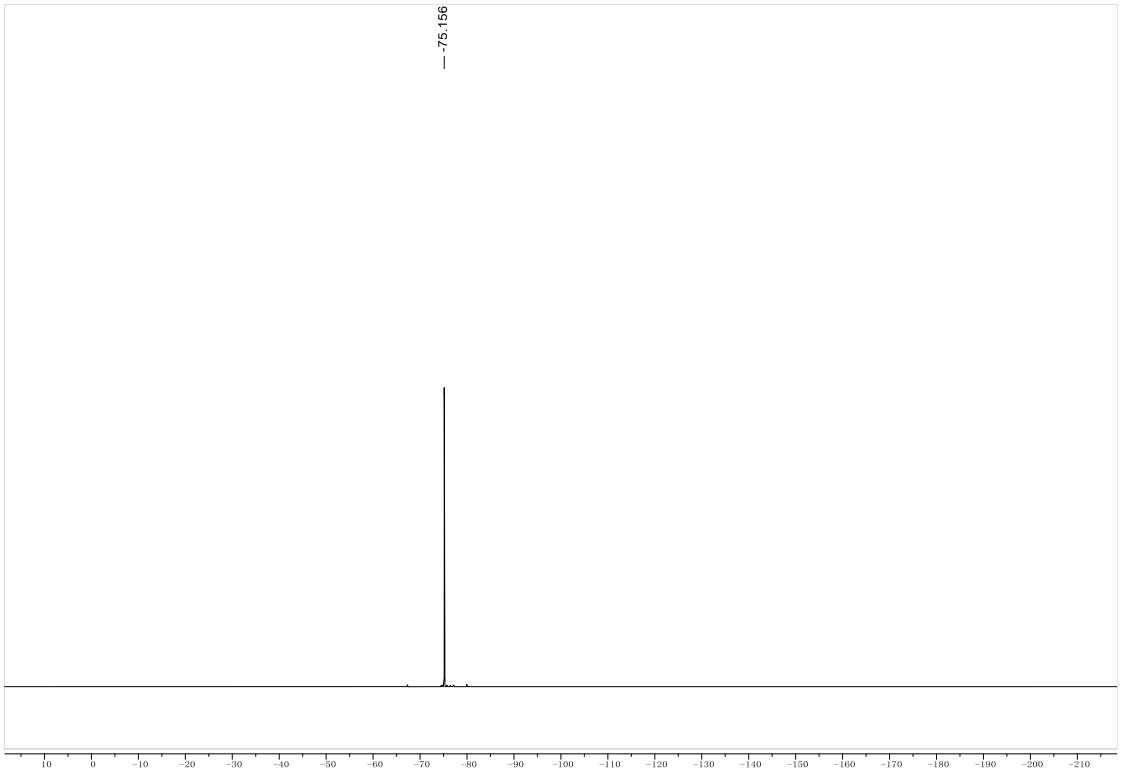


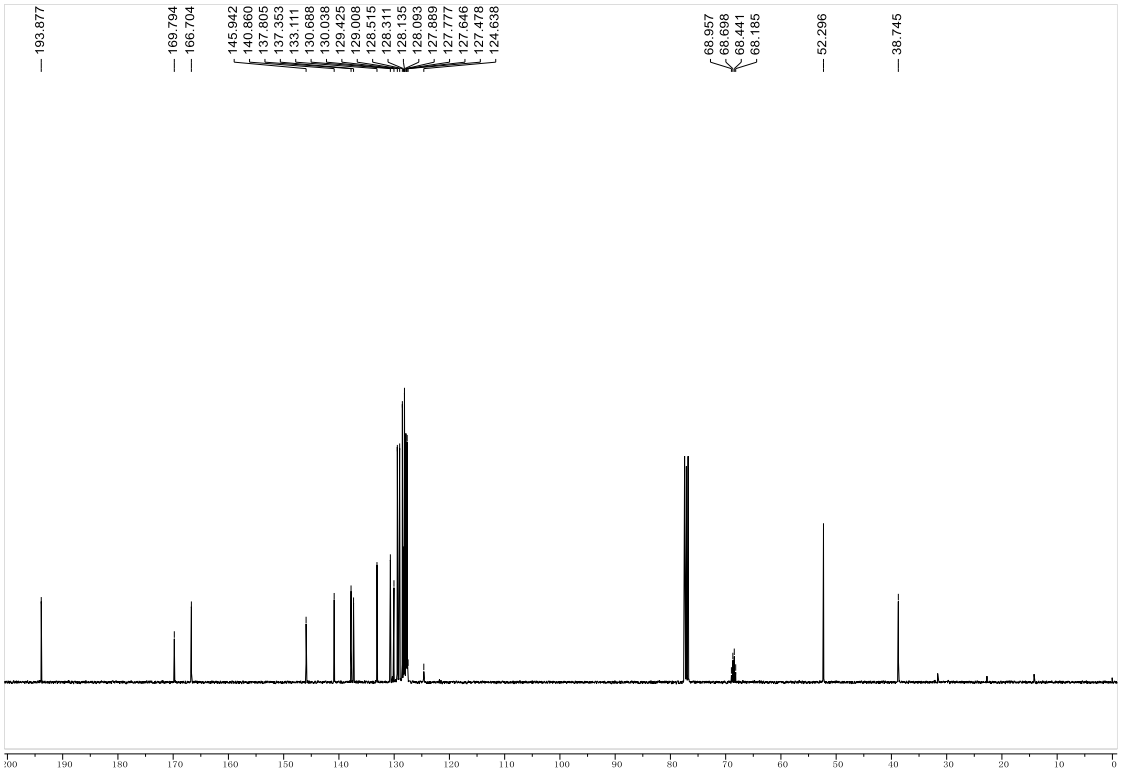




194.284
169.473
157.399
156.721
140.969
138.052
137.637
135.610
132.995
130.526
129.912
129.462
128.950
128.514
128.179
128.091
127.958
127.833
127.501
123.739
119.216
118.180
68.537
68.282
68.025
67.770
38.528
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

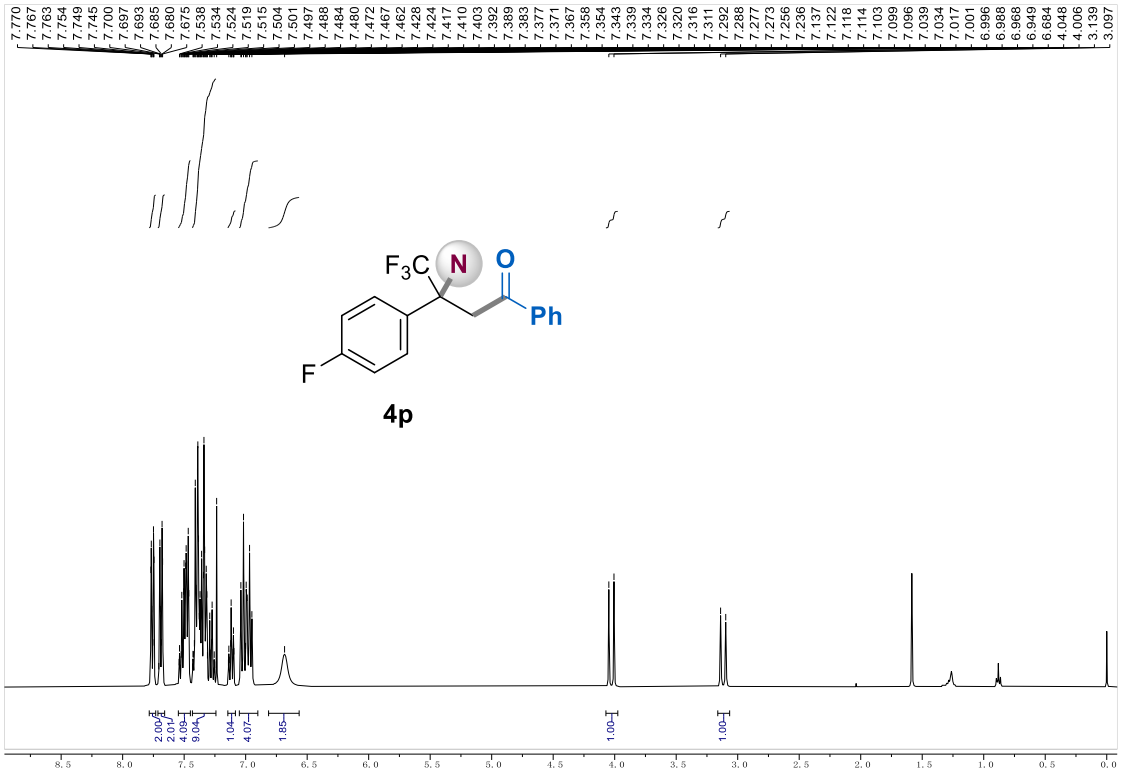
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4o**

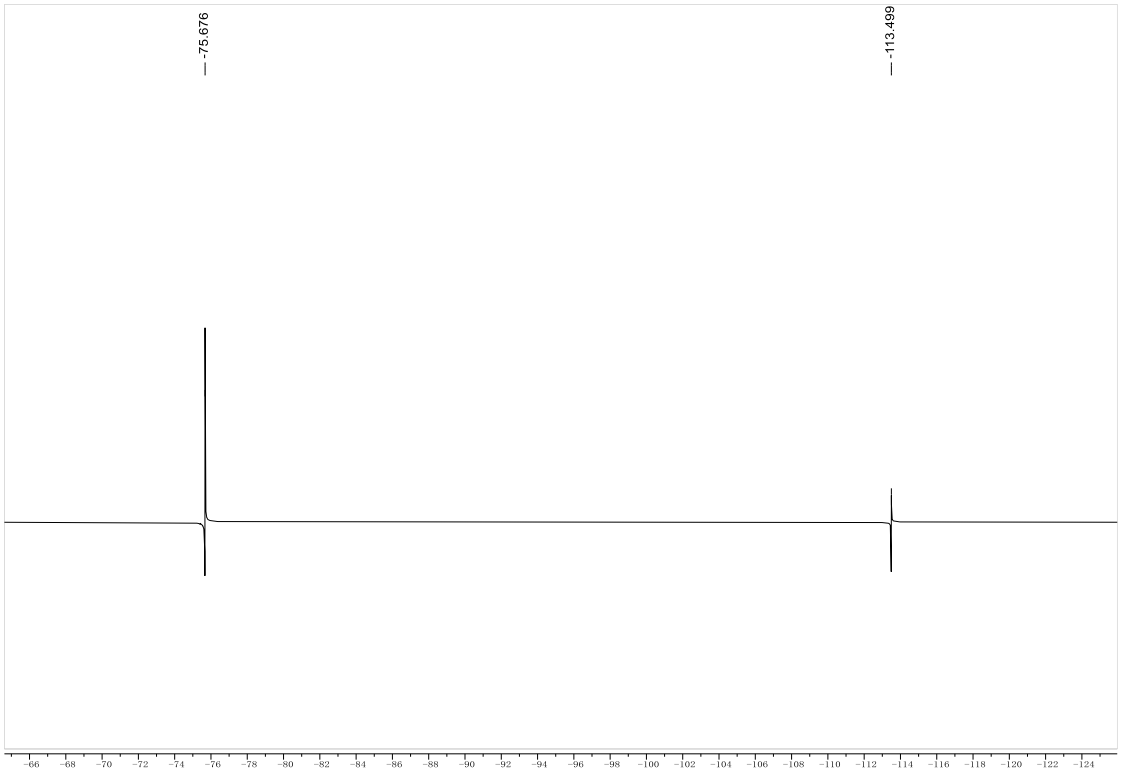


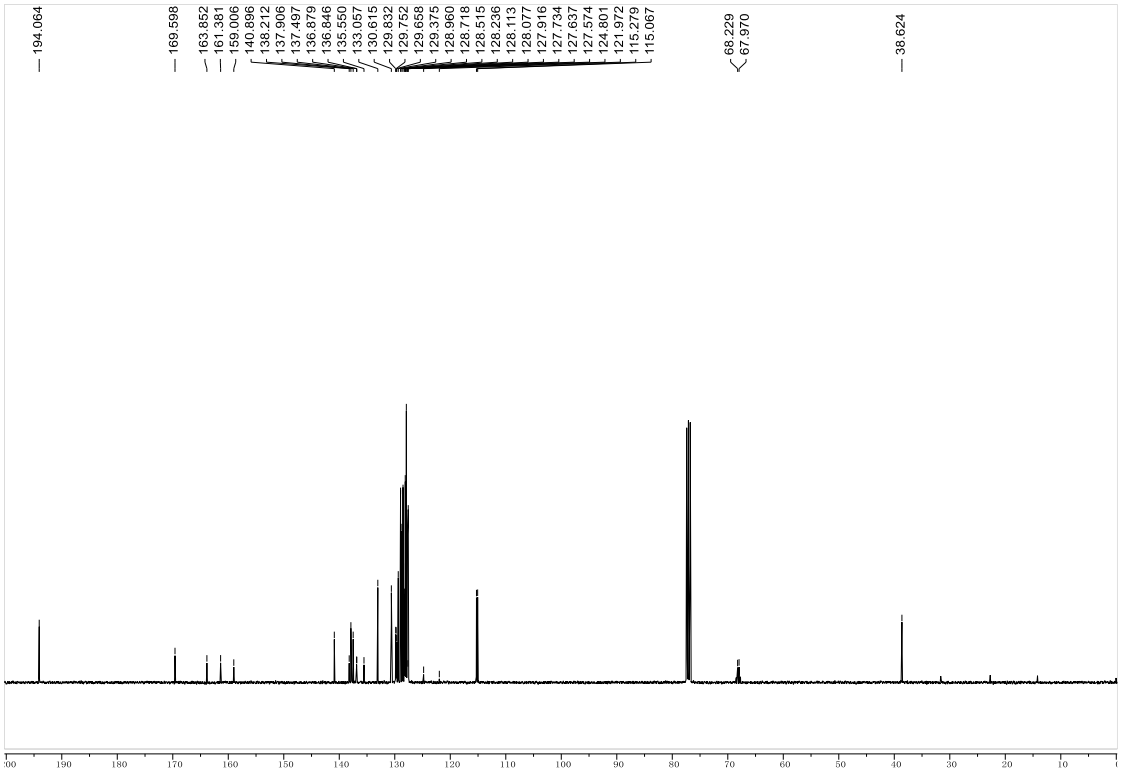




193.877
169.794
166.704
145.942
140.860
137.805
137.353
133.111
130.688
130.038
129.425
129.008
128.515
128.311
128.135
128.093
127.889
127.777
127.646
127.478
124.638
68.957
68.698
68.441
68.185
52.296
38.745
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

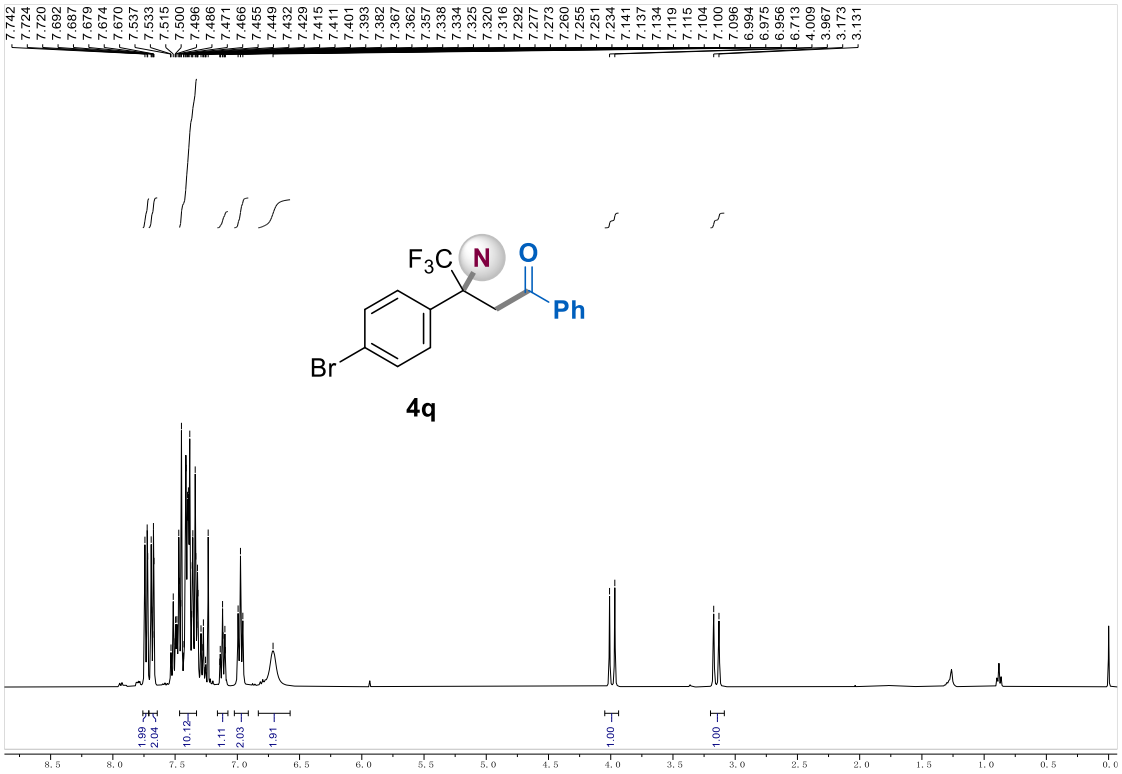
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4p**

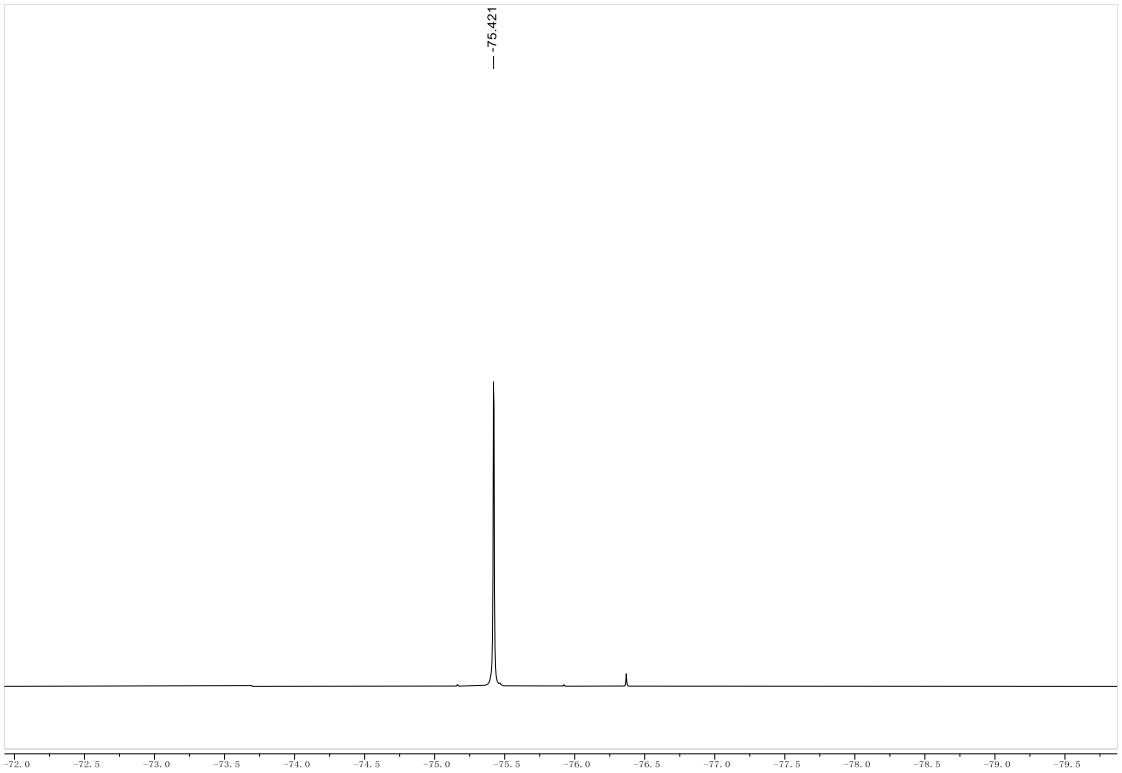


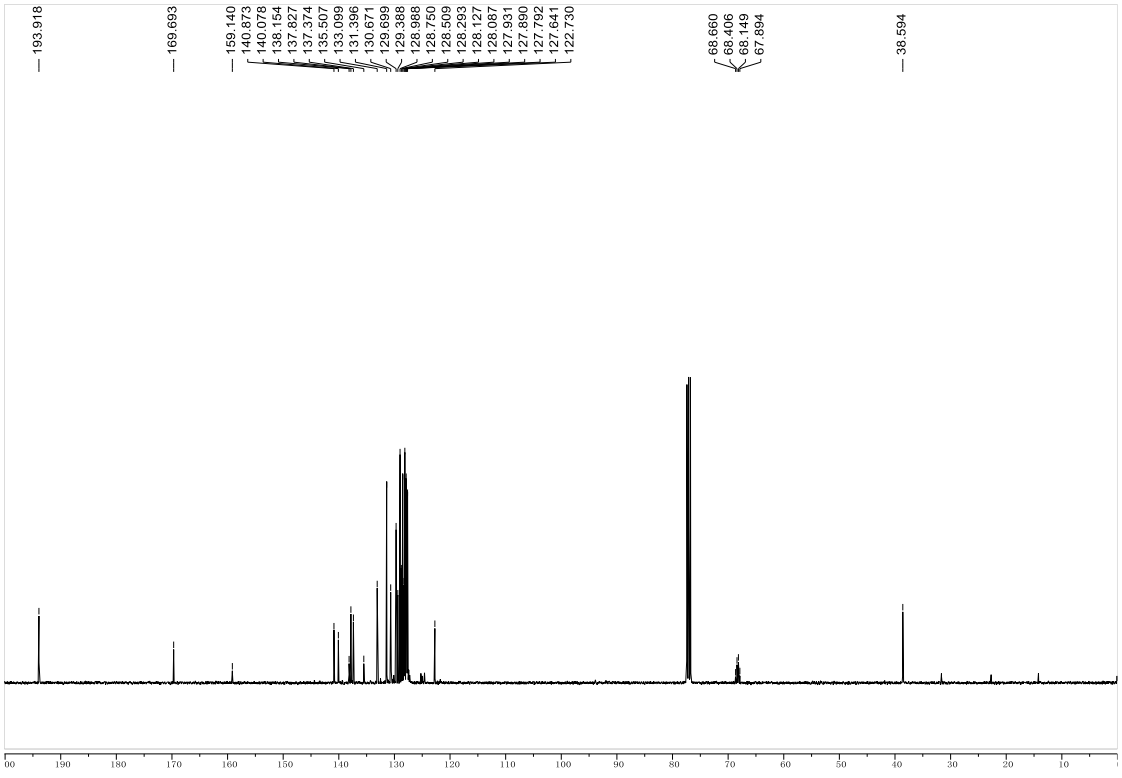




194.064
169.598
163.852
161.381
159.006
140.896
138.212
137.906
137.497
136.879
136.846
135.550
133.057
130.615
129.832
129.752
129.658
129.375
128.960
128.718
128.515
128.236
128.113
128.077
127.916
127.734
127.637
127.574
124.801
121.972
115.279
115.067
68.229
67.970
38.624
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

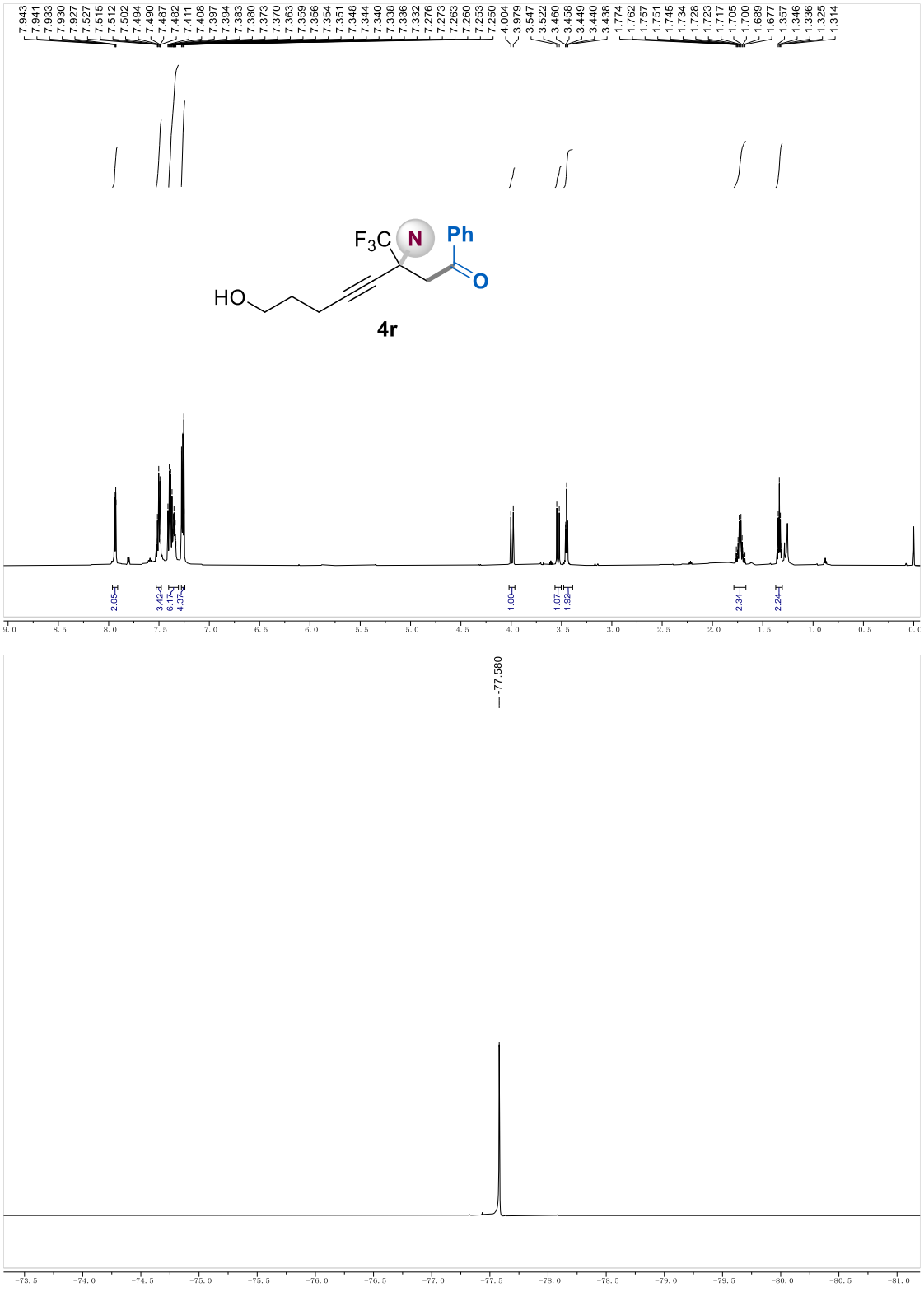
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4q**

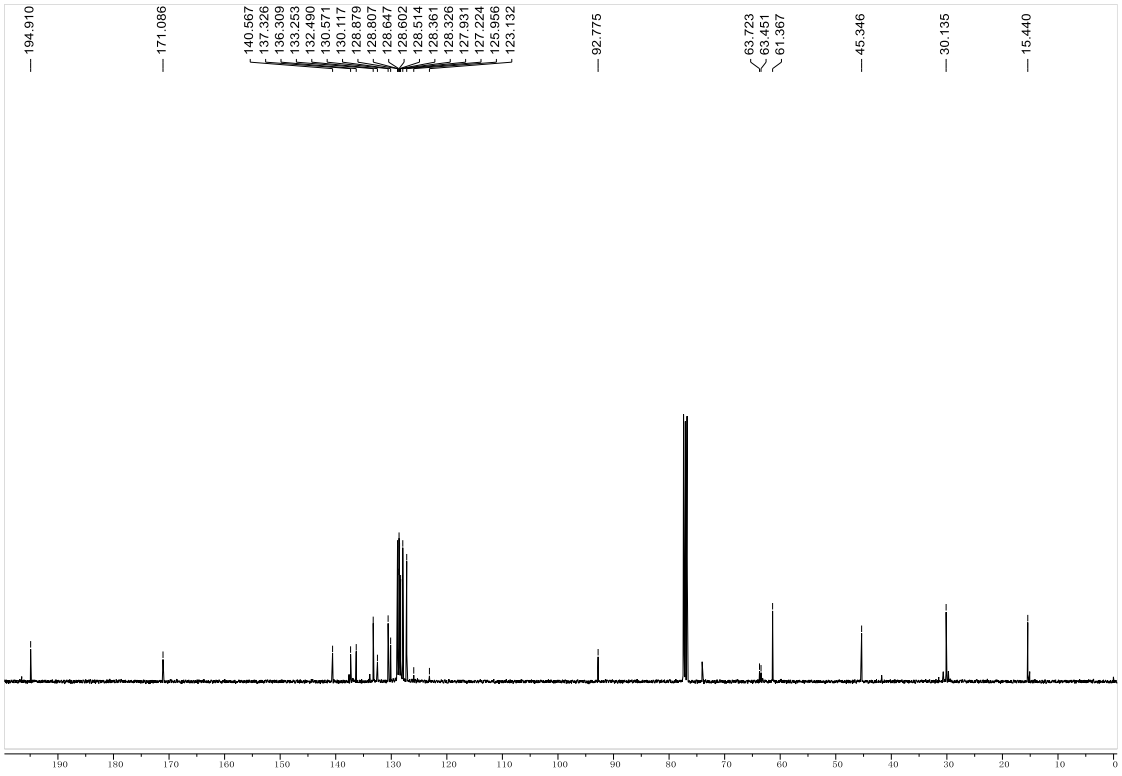


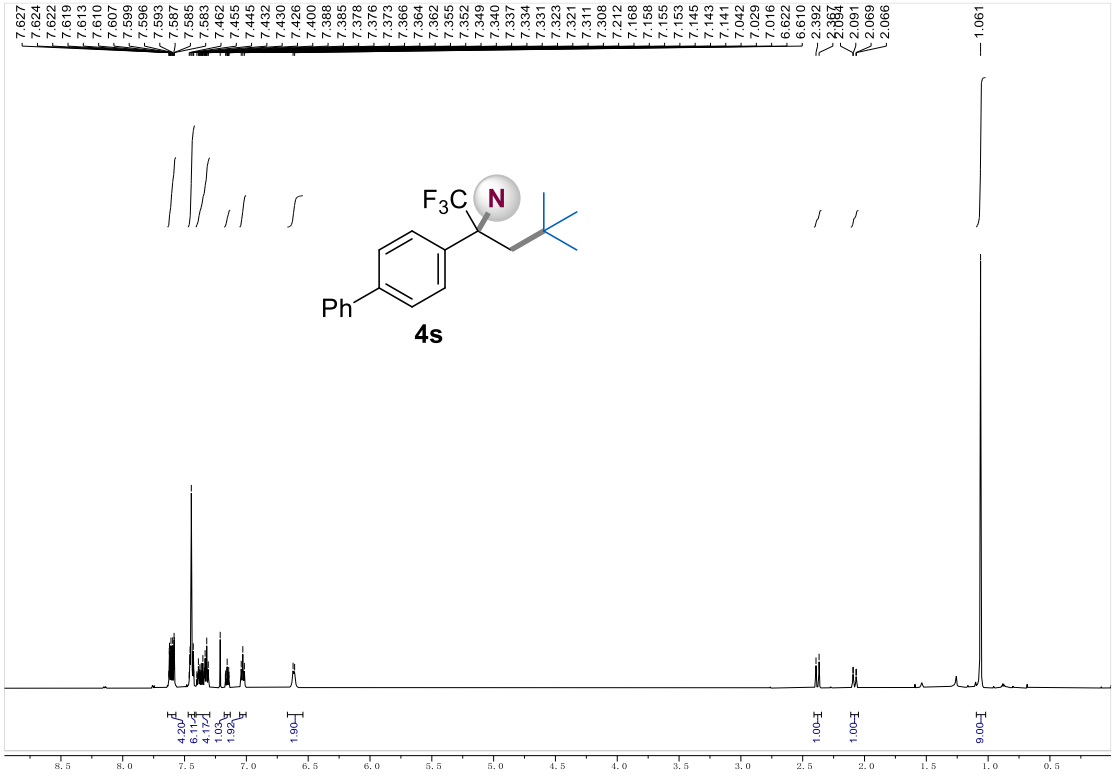

193.918
169.693
159.140
140.873
140.078
138.154
137.827
137.374
135.507
133.099
131.396
130.671
129.699
129.388
128.988
128.750
128.509
128.293
128.127
128.087
127.931
127.890
127.792
127.641
122.730
68.660
68.406
68.149
67.894
38.594
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

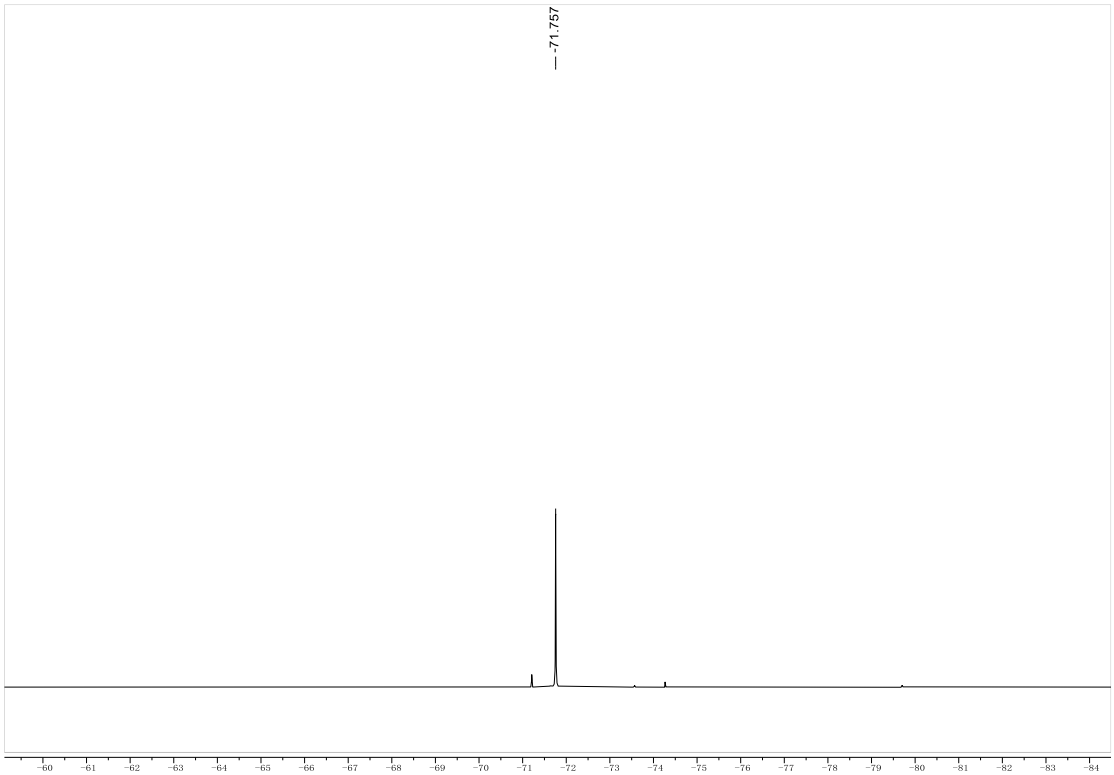
**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 4r**

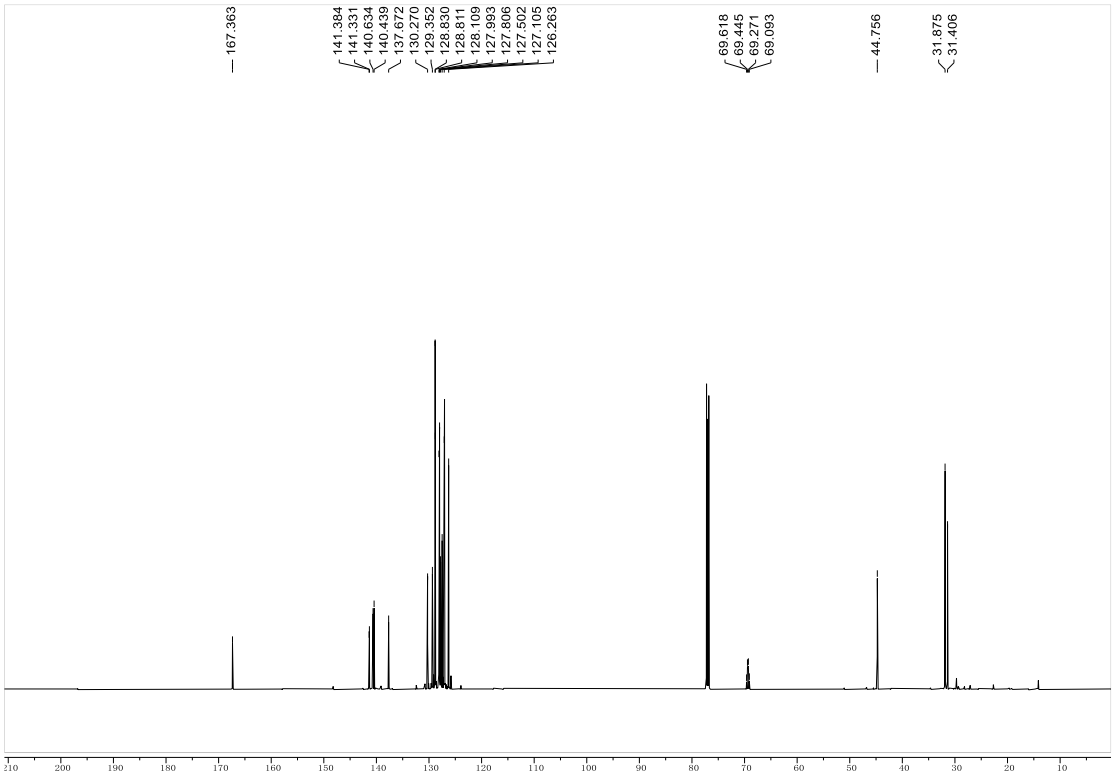



194.910
171.086
140.567
137.326
136.309
133.253
132.490
130.571
130.117
128.879
128.807
128.647
128.602
128.514
128.361
128.326
127.931
127.224
125.956
123.132
92.775
63.723
63.451
61.367
45.346
30.135
15.440
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

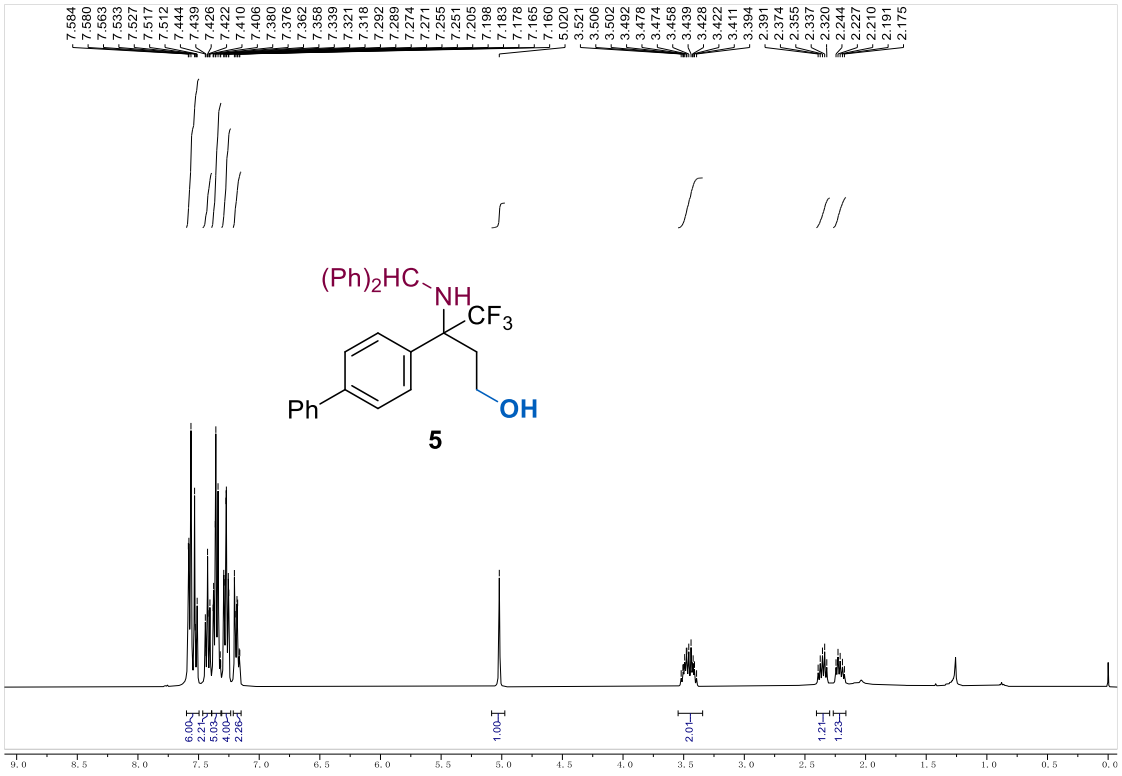
**$^{1}$H NMR (600 MHz, $CDCl_3$), $^{19}$F NMR (565 MHz, $CDCl_3$), $^{13}$C NMR (150 MHz, $CDCl_3$) spectrum of product 4s**

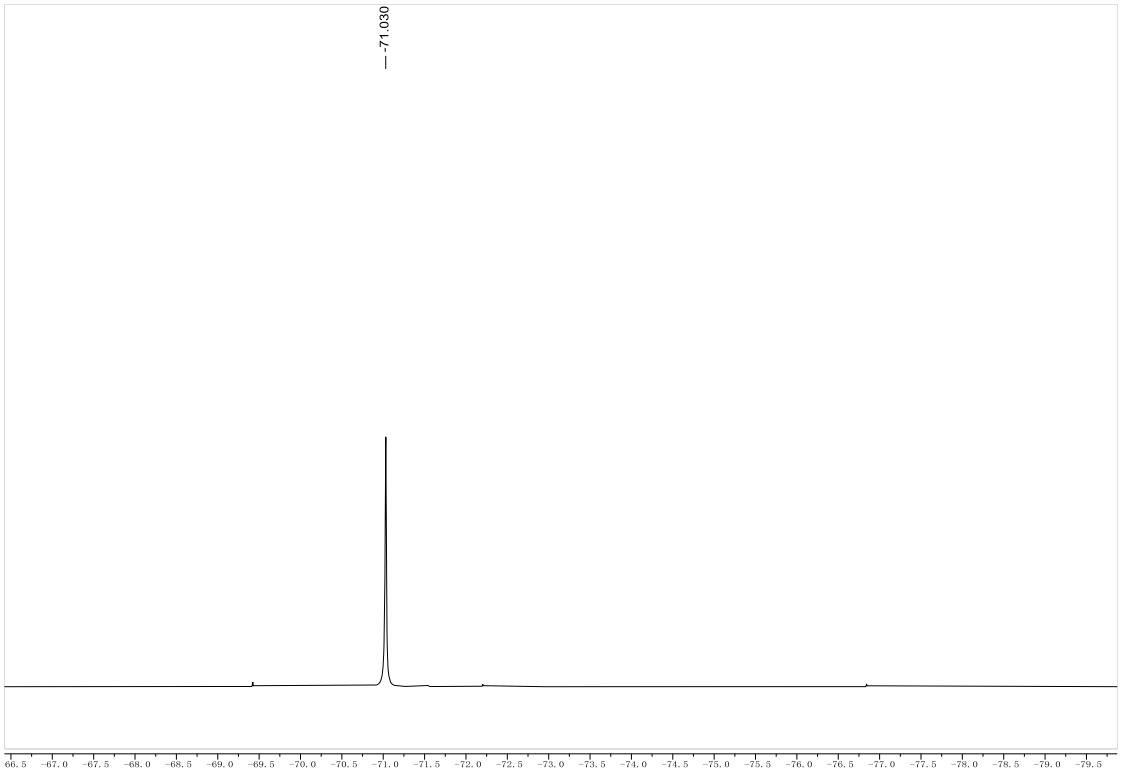


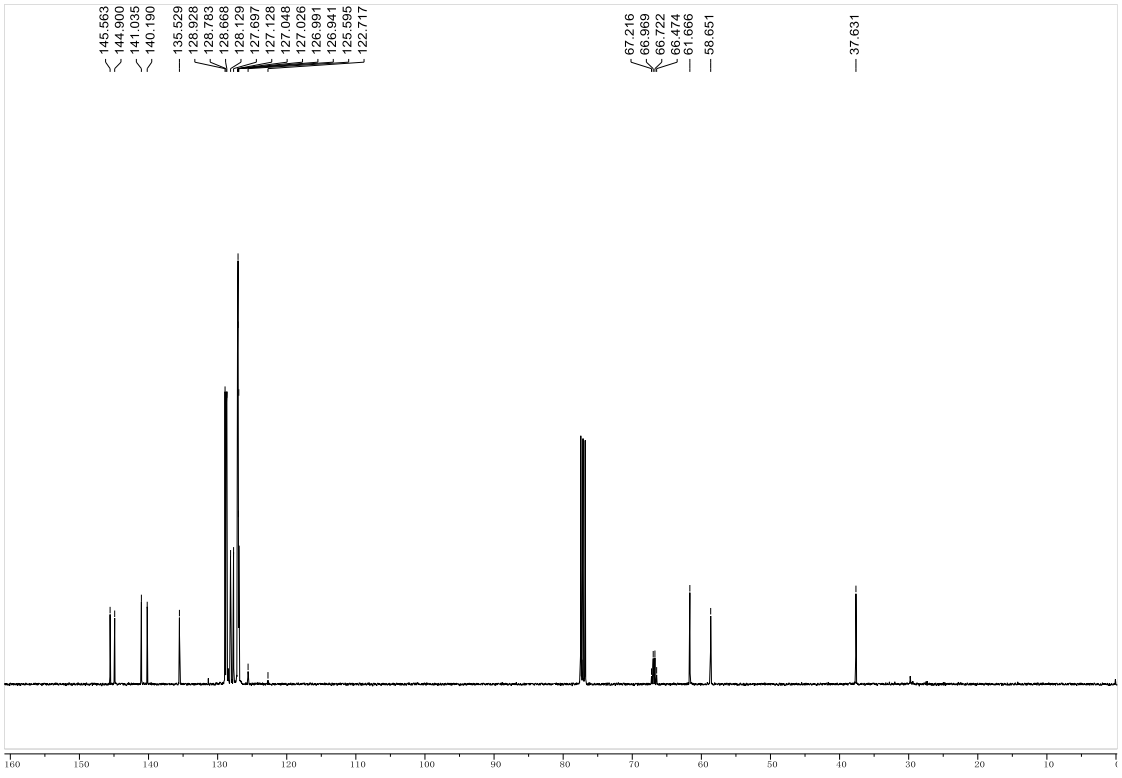




167.363
141.384
141.331
140.634
140.439
137.672
130.270
129.352
128.830
128.811
128.109
127.993
127.806
127.502
127.105
126.263
69.618
69.445
69.271
69.093
44.756
31.875
31.406
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

**$^{1}$H NMR (400 MHz, $CDCl_3$), $^{19}$F NMR (375 MHz, $CDCl_3$), $^{13}$C NMR (100 MHz, $CDCl_3$) spectrum of product 5**

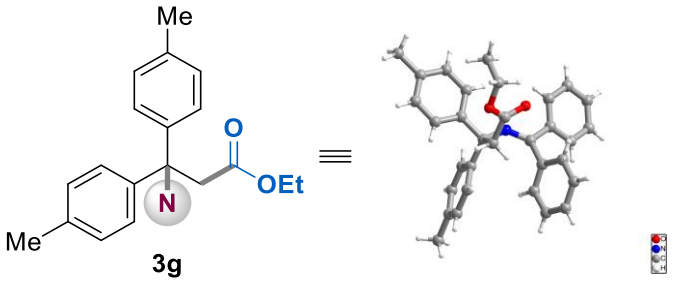



145.563
144.900
141.035
140.190
135.529
128.928
128.783
128.668
128.129
127.697
127.128
127.048
127.026
126.991
126.941
125.595
122.717
67.216
66.969
66.722
66.474
61.666
58.651
37.631
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

# X-ray crystallographic data of compound

X-Ray crystallographic analysis of **3g** (CCDC 2143808) showing the thermal ellipsoids at 30% probability level.




**3g**

## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 20211221-xpj_autoaaa

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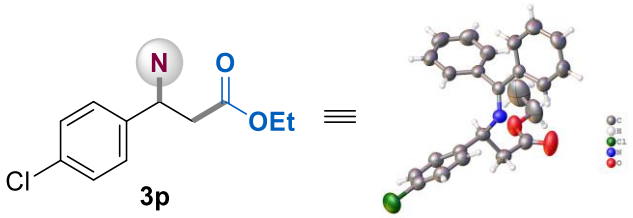
No syntax errors found. CIF dictionary Interpreting this report

## Datablock: 20211221-xpj_autoaaa

---

```
Bond precision:   C-C = 0.0034 A          Wavelength=0.71073

Cell:           a=10.3110(4)      b=11.2258(5)      c=12.8508(5)
                alpha=91.396(3)   beta=99.313(3)    gamma=117.296(4)
Temperature:    293 K

                 Calculated                Reported
Volume           1296.15(11)               1296.15(10)
Space group      P -1                      P -1
Hall group       -P 1                      -P 1
Moiety formula   C32 H31 N O2              C32 H31 N O2
Sum formula      C32 H31 N O2              C32 H31 N O2
Mr               461.58                    461.58
Dx,g cm-3        1.183                     1.183
Z                2                         2
Mu (mm-1)        0.073                     0.073
F000             492.0                     492.0
F000'            492.20
h,k,lmax         13,14,16                  13,14,16
Nref             6188                      5440
Tmin,Tmax                                  0.723,1.000
Tmin'

Correction method= # Reported T Limits: Tmin=0.723 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.879          Theta(max)= 27.873

R(reflections)= 0.0542( 4198)                wR2(reflections)=
                                             0.1553( 5440)
S = 1.061                 Npar= 319
```

X-Ray crystallographic analysis of **3p** (CCDC 2143809) showing the thermal ellipsoids at 30% probability level.




## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 20220108-xpj_auto

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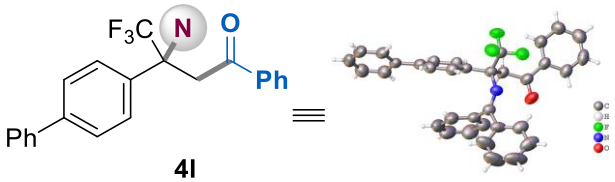
No syntax errors found. CIF dictionary Interpreting this report

## Datablock: 20220108-xpj_auto

---

```
Bond precision:   C-C = 0.0033 A          Wavelength=0.71073

Cell:               a=9.2336(4)      b=20.3550(6)      c=22.5192(7)
                    alpha=90         beta=90           gamma=90
Temperature:        293 K

                  Calculated                  Reported
Volume            4232.5(3)                   4232.5(2)
Space group       P b c a                     P b c a
Hall group        -P 2ac 2ab                  -P 2ac 2ab
Moiety formula    C24 H22 Cl N O2             C24 H22 Cl N O2
Sum formula       C24 H22 Cl N O2             C24 H22 Cl N O2
Mr                391.88                      391.87
Dx,g cm-3         1.230                       1.230
Z                 8                           8
Mu (mm-1)         0.199                       0.199
F000              1648.0                      1648.0
F000'             1649.83
h,k,lmax          12,26,29                    12,26,27
Nref              5054                        4380
Tmin,Tmax                                     0.520,1.000
Tmin'

Correction method= # Reported T Limits: Tmin=0.520 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.867            Theta(max)= 27.900

R(reflections)= 0.0520( 3109)                 wR2(reflections)=
                                              0.1408( 4380)
S = 1.062                  Npar= 254
```

X-Ray crystallographic analysis of **3l** (CCDC 214380) showing the thermal ellipsoids at 30% probability level.




**4l**

## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) aaaa

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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: aaaa

---

```
Bond precision:   C-C = 0.0069 A          Wavelength=0.71073

Cell:             a=12.1957(6)     b=14.1964(6)      c=16.4452(8)
                  alpha=90         beta=94.807(5)    gamma=90
Temperature:      293 K

                  Calculated                  Reported
Volume            2837.2(2)                   2837.2(2)
Space group       P 21/n                      P 1 21/n 1
Hall group        -P 2yn                      -P 2yn
Moiety formula    C35 H26 F3 N O              C35 H26 F3 N O
Sum formula       C35 H26 F3 N O              C35 H27 F3 N O
Mr                533.57                      534.57
Dx,g cm-3         1.249                       1.251
Z                 4                           4
Mu (mm-1)         0.088                       0.088
F000              1112.0                      1116.0
F000'             1112.58
h,k,lmax          16,18,21                    15,18,21
Nref              6774                        5680
Tmin,Tmax                                     0.619,1.000
Tmin'

Correction method= # Reported T Limits: Tmin=0.619 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.839          Theta(max)= 27.889

R(reflections)= 0.0780( 2929)                 wR2(reflections)=
                                              0.3311( 5680)
S = 1.069                 Npar= 361
```