Supplementary Information

Photoinduced Fe-Catalyzed Bromination and Iodination of Unstrained Cyclic Alcohols

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Materials: Acetonitrile (MeCN) was purified by a Vigor solvent purification system. Anhydrous $Fe(OAc)_2$ and $NaBrO_3$ were purchased from Energy Chemical. *t*-BuONa was purchased from Adamas-beta. $Fe(OAc)_2$ and *t*-BuONa were stored and weighed in the glovebox. Other commercially available chemicals were purchased and used without additional purification unless noted otherwise.

Infrared spectra were recorded on a Nicolet iS5 using neat thin film technique. High-resolution mass spectra (HRSM) were obtained on a Waters I-Class VION IMS QTof and are reported as m/z (relative intensity). Accurate masses are reported for the molecular ion $[M+Na]^+$, $[M+H]^+$, $[M-OH]^+$, $[M-H]^-$ or $[M]^+$. ^1H NMR spectra were recorded on a Bruker-400 MHz spectrometer, ^{13}C NMR spectra were recorded at 101 MHz, and ^{19}F NMR spectra were recorded at 376 MHz. Unless otherwise noted, all spectra were acquired in $CDCl_3$. Chemical shifts are reported in parts per million (ppm, δ), downfield from tetramethylsilane (TMS, δ = 0.00 ppm) and are referenced to residual solvent ($CDCl_3$, δ = 7.26 ppm (^1H) and 77.00 ppm (^{13}C)). Coupling constants were reported in Hertz (Hz). Data for NMR spectra were reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, m = multiplet, coupling constant (Hz), and integration.

The tertiary alcohols including **1a-1d**,[1] **1f**,[1] **1g**,[1] **1k**,[1] **1r**,[1] **1e**,[2] **1l**,[2] **1h**,[3] **1n**,[3] **1o**,[3] **1i**,[4] **1k**,[5] **1m**,[6] **1p**,[7] **1q**[7] and **1s**[8], are known and were prepared according to the known literature.

The LED light (100 W, emitting area: 30 × 30 mm) was assembled using the 390-395 nm chips purchased from GuangHong Chips. The emission spectra of the LED light is shown below (**Figure S1**) and wavelength of peak intensity is 390-395 nm. The material of the reaction vessels is regular borosilicate glass. The distance from the light source to the reaction vessel is 5 cm (**Figure S2**).

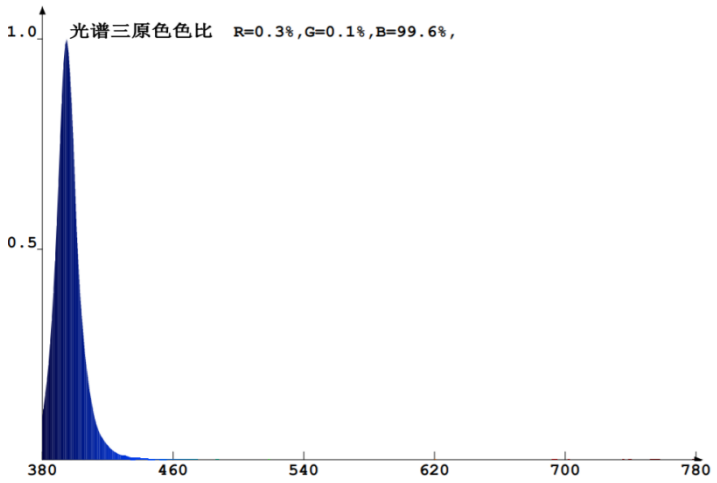


Figure S1: The emission spectrum of the LED light.

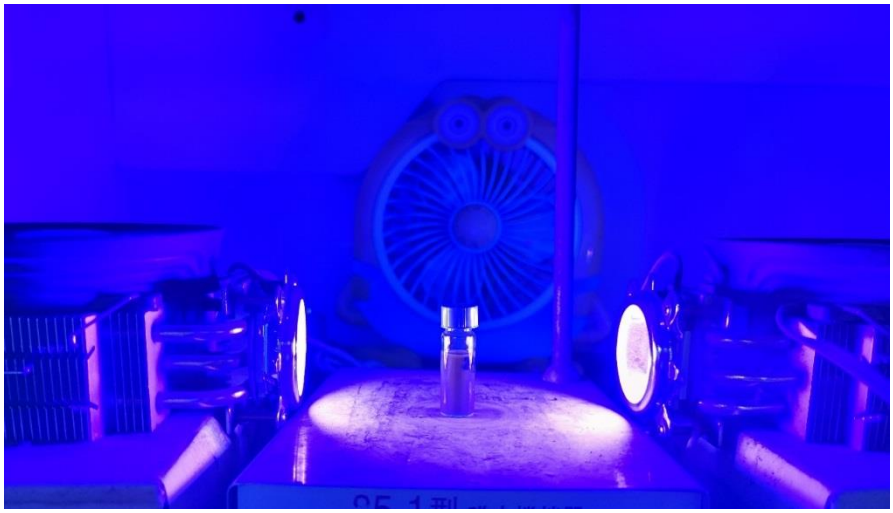
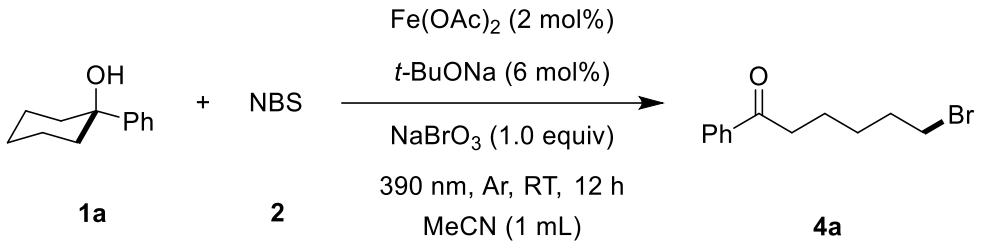


Figure S2: The setting-up reactions.

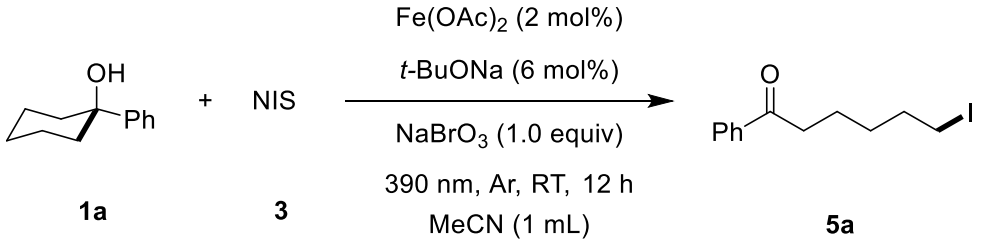
Bromination of Cyclic Alcohols



1a **2** **4a**

Typical Procedure 1: To a 4 mL vial were added $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1a** (70.4 mg, 0.40 mmol), **2** (106.2 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. Under irradiation at 390 nm LEDs, the resulting mixture was stirred for 12 hours at rt. Evaporation and flash chromatography on silica gel afforded **4a** (88.0 mg, 86%): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2 H, Ar-H), 7.59 – 7.54 (m, 1 H, Ar-H), 7.50 – 7.44 (m, 2 H, Ar-H), 3.43 (t, *J* = 6.7 Hz, 2 H, CH_2), 3.00 (t, *J* = 7.28 Hz, 2 H, CH_2), 1.99 – 1.86 (m, 2 H, CH_2), 1.85 – 1.71 (m, 2 H, CH_2), 1.58 – 1.50 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.0, 136.9, 133.0, 128.6, 128.0, 38.2, 33.6, 32.6, 27.8, 23.3. IR ν (neat, cm^{-1}) 2933, 2863, 1681, 1447, 1205. The spectra are matching with the known literature.[3]

Iodination of Cyclic Alcohols

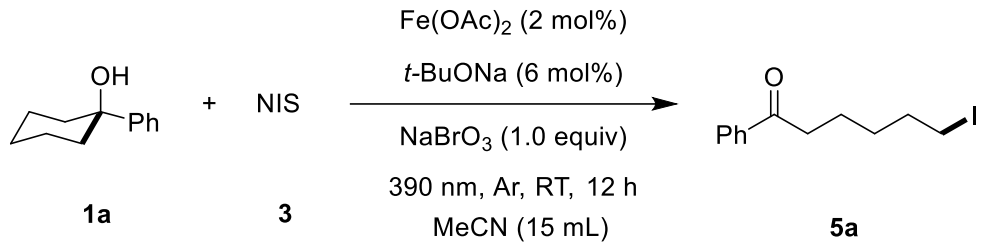


1a **3** **5a**

Typical Procedure 2: To a 4 mL vial were added $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1a** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. Under irradiation at 390 nm LEDs, the resulting mixture was stirred for 12 hours at rt. Evaporation and flash chromatography on silica gel afforded **5a** (104.6 mg, 87%, 96% purity) (eluent: PE/EA = 10/1): light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2 H, Ar-H), 7.59 – 7.54 (m, 1 H, Ar-H),

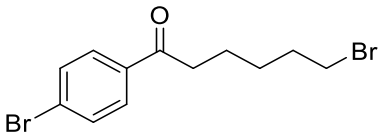
7.50 – 7.44 (m, 2 H, Ar-H), 3.21 (t, J = 6.97 Hz, 2 H, CH_2), 2.99 (t, J = 7.30 Hz, 2 H, CH_2), 1.93 – 1.84 (m, 2 H, CH_2), 1.82 – 1.72 (m, 2 H, CH_2), 1.55 – 1.46 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.0, 136.9, 132.9, 128.5, 128.0, 38.2, 33.3, 30.1, 23.0, 6.8. IR ν (neat, cm^{-1}) 2933, 2863, 1681, 1447, 1205. The spectra are matching with the known literature.[9]

1 gram scale reaction:



1a **3** **5a**

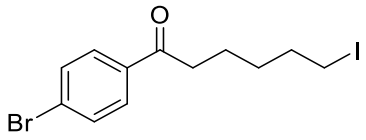
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (20.8 mg, 0.12 mmol), *t*-BuONa (34.6 mg, 0.36 mmol), **1a** (1.06 g, 6 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (0.90 g, 6 mmol) in MeCN (15.0 mL) afforded **5b** (1.6011 g, 88%) (eluent: PE/EA = 10/1, 95% purity): light yellow solid. mp: 41.5–42.3 ºC (DCM/hexane). ^1H MR (400 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2 H, Ar-H), 7.59 – 7.54 (m, 1 H, Ar-H), 7.50 – 7.44 (m, 2 H, Ar-H), 3.21 (t, J = 6.97 Hz, 2 H, CH_2), 2.99 (t, J = 7.30 Hz, 2 H, CH_2), 1.93 – 1.84 (m, 2 H, CH_2), 1.82 – 1.72 (m, 2 H, CH_2), 1.55 – 1.46 (m, 2 H, CH_2).



4b 91%

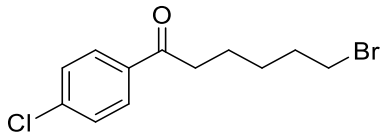
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1b** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4b** (120.2 mg, 91%) (eluent: PE/EA = 20/1 to 10/1): white solid. mp: 39.0–40.2 ºC (DCM/hexane). ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.80 (m, 2 H, Ar-H), 7.63 – 7.57 (m, 2 H, Ar-H), 3.43 (t, J = 6.75 Hz, 2 H, CH_2), 2.96 (t, J = 7.28 Hz, 2 H, CH_2), 1.96 – 1.86 (m, 2 H, CH_2), 1.81 – 1.71 (m, 2 H, CH_2), 1.59 – 1.48 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 135.7,

131.9, 129.5, 128.1, 38.2, 33.6, 32.6, 27.7, 23.1. IR ν (neat, cm^{-1}) 2937, 2865, 1687, 1584, 1396, 1070. The spectra are matching with the known literature.[2]



5b 93%

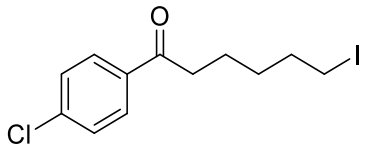
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1b** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5b** (140.0 mg, 93%, 95% purity) (eluent: PE/EA = 10/1): white solid. mp: 63.0–64.2 ºC (DCM/hexane). The pure ^1H NMR could be obtained after preparative thin layer chromatography. ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.80 (m, 2 H, Ar-H), 7.63 – 7.58 (m, 2 H, Ar-H), 3.21 (t, J = 6.95 Hz, 2 H, CH_2), 2.96 (t, J = 7.28 Hz, 2 H, CH_2), 1.93 – 1.84 (m, 2 H, CH_2), 1.80 – 1.71 (m, 2 H, CH_2), 1.54 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 135.6, 131.9, 129.5, 128.1, 38.2, 33.2, 30.1, 22.9, 6.7. IR ν (neat, cm^{-1}) 2988, 2933, 2868, 1682, 1392, 1141. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}BrIO$ 380.9346; found 380.9348.



4c 84%

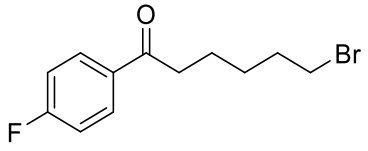
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1c** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4c** (98.2 mg, 84%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.87 (m, 2 H, Ar-H), 7.47 – 7.38 (m, 2 H, Ar-H), 3.43 (t, J = 6.73 Hz, 2 H, CH_2), 2.96 (t, J = 7.25 Hz, 2 H, CH_2), 1.97 – 1.87 (m, 2 H, CH_2), 1.82 – 1.72 (m, 2 H, CH_2), 1.58 – 1.49 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 139.3, 135.1, 129.4, 128.9, 38.2, 33.6, 32.5, 27.7,

23.1. IR ν (neat, cm^{-1}) 2933, 2857, 1681, 1593, 1447, 1205. The pectra are matching with the known literature.[2]



5c 91%

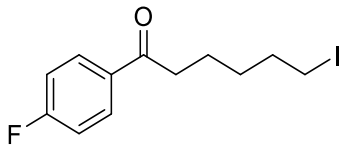
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1c** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5c** (121.2 mg, 91%, 96% purity) (eluent: PE/EA = 10/1): white solid. mp: 59.4–60.9 °C (DCM/hexane). The pure ^1H NMR was obtained after recrystallization (DCM and hexane). ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2 H, Ar-H), 7.46 – 7.42 (m, 2 H, Ar-H), 3.21 (t, J = 6.95 Hz, 2 H, CH_2), 2.97 (t, J = 7.29 Hz, 2 H, CH_2), 1.92 – 1.84 (m, 2 H, CH_2), 1.81 – 1.72 (m, 2 H, CH_2), 1.54 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 139.4, 135.2, 129.4, 128.9, 38.2, 33.2, 30.1, 22.9, 6.7. IR ν (neat, cm^{-1}) 2934, 2862, 1682, 1692, 1586, 1396, 1207. The spectra are matching with the known literature.[3]



4d 90%

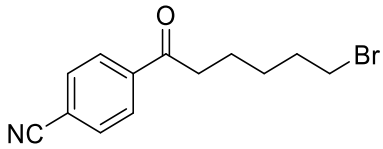
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1d** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4d** (98.0 mg, 90%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.96 (m, 2 H, Ar-H), 7.17 – 7.09 (m, 2 H, Ar-H), 3.21 (t, J = 6.94 Hz, 2 H, CH_2), 2.97 (t, J = 7.25 Hz, 2 H, CH_2), 1.93 – 1.83 (m, 2 H, CH_2), 1.81 – 1.71 (m, 2 H, CH_2), 1.55 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.3, 165.6 (d, J = 254.45 Hz), 133.3 (d, J = 3.08 Hz),

130.6 (d, J = 9.32 Hz), 115.6 (d, J = 21.75 Hz), 38.1, 33.6, 32.5, 27.8, 23.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.30. IR ν (neat, cm^{-1}) 2937, 2864, 1682, 1597, 1226, 1156. The spectra are matching with the known literature.[2]



5d 94%

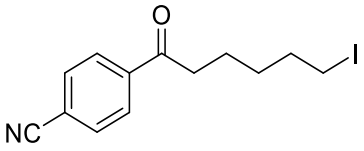
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1d** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5d** (120.3 mg, 91%, 97% purity) (eluent: PE/EA = 10/1): oil. The pure ^{1}H NMR was obtained after second flash chromatography on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.96 (m, 2 H, Ar-H), 7.17 – 7.09 (m, 2 H, Ar-H), 3.21 (t, J = 6.94 Hz, 2 H, CH_2), 2.97 (t, J = 7.24 Hz, 2 H, CH_2), 1.93 – 1.83 (m, 2 H, CH_2), 1.81 – 1.71 (m, 2 H, CH_2), 1.55 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.3, 165.6 (d, J = 254.20 Hz), 133.3, 130.6 (d, J = 8.87 Hz), 115.6 (d, J = 21.82 Hz), 38.1, 33.2, 30.1, 23.0, 6.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.28. IR ν (neat, cm^{-1}) 2932, 2861, 1681, 1597, 1225, 1171, 1159. The spectra are matching with the known literature.[3]



4e 86%

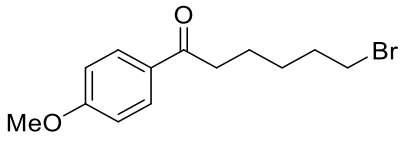
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1e** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4e** (95.8 mg, 86%) (eluent: PE/EA = 10/1): white solid. mp: 55.7–66.8 °C (DCM/hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.31 Hz, 2H, Ar-H), 7.78 (d, J = 8.42 Hz, 2 H, Ar-H), 3.44 (t, J =

6.70 Hz, 2 H, CH_2), 3.02 (t, *J* = 7.24 Hz, 2 H, CH_2), 1.97 – 1.88 (m, 2 H, CH_2), 1.83 – 1.74 (m, 2 H, CH_2), 1.60 – 1.50 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 139.7, 132.5, 128.4, 117.9, 116.2, 38.6, 33.6, 32.4, 27.7, 22.9. IR ν (neat, cm^{-1}) 2931, 2857, 2229, 1685, 1403, 1209, 1045. The spectra are matching with the known literature.[2]



5e 90%

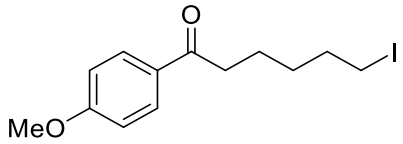
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1e** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5e** (117.7 mg, 90%, 97% purity) (eluent: PE/EA = 10/1): white solid. mp: 74.5–75.4 ºC (DCM/hexane). ^1H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.17 Hz, 2H, Ar-H), 7.78 (d, *J* = 8.27 Hz, 2 H, Ar-H), 3.22 (t, *J* = 7.06 Hz, 2 H, CH_2), 3.02 (t, *J* = 7.19 Hz, 2 H, CH_2), 1.94 – 1.84 (m, 2 H, CH_2), 1.83 – 1.73 (m, 2 H, CH_2), 1.55 – 1.46 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 139.7, 132.5, 128.4, 117.9, 116.2, 38.5, 33.1, 30.0, 22.7, 6.7. IR ν (neat, cm^{-1}) 2949, 2931, 2894, 2232, 1688, 1404, 1254. The spectra are matching with the known literature.[3]



4f 65%

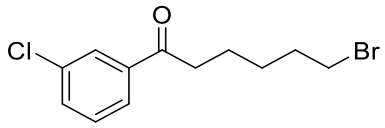
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1f** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4f** (73.5 mg, 65%) (eluent: PE/EA = 10/1): white solid. mp: 55.5–56.4 ºC (DCM/hexane). ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.91 (m, 2 H, Ar-H), 6.97 – 6.90 (m, 2 H, Ar-H), 3.87 (s, 3 H, CH_3),

3.43 (t, J = 6.76 Hz, 2 H, CH_2), 2.94 (t, J = 7.30 Hz, 2 H, CH_2), 1.97 – 1.87 (m, 2 H, CH_2), 1.80 – 1.70 (m, 2 H, CH_2), 1.59 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 163.4, 130.3, 130.0, 113.7, 55.4, 37.9, 33.7, 32.6, 27.9, 23.5. IR ν (neat, cm^{-1}) 2934, 2846, 1676, 1600, 1256, 1170. The spectra are matching with the known literature.[10]



5f 70%

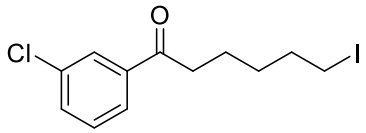
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1f** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5f** (93.6 mg, 70%) (eluent: PE/EA = 10/1): white solid. mp: 59.1–60.3 ºC (DCM/hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.90 Hz, 2 H, Ar-H), 6.93 (d, J = 8.89 Hz, 2 H, Ar-H), 3.87 (s, 3 H, OCH_3), 3.20 (t, J = 6.99 Hz, 2 H, CH_2), 2.93 (t, J = 7.33 Hz, 2 H, CH_2), 1.94 – 1.81 (m, 2 H, CH_2), 1.82 – 1.68 (m, 2 H, CH_2), 1.55 – 1.43 (m, 2 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 163.3, 130.2, 130.0, 113.7, 55.4, 37.9, 33.3, 30.2, 23.3, 6.8. IR ν (neat, cm^{-1}) 2946, 2859, 1678, 1600, 1250, 1173. The spectra are matching with the known literature.[3]



4g 84%

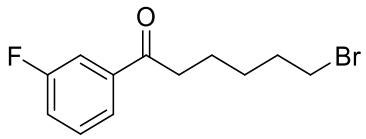
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1f** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4f** (91.2mg, 84%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (t, J = 1.92 Hz, 1 H, Ar-H), 7.83 (dt, J = 7.72, 1.45 Hz, 1 H, Ar-H), 7.53 (ddd, J = 7.96, 2.03, 1.01 Hz, 1 H, Ar-H), 7.41

(t, J = 7.87 Hz, 1 H , Ar-H), 3.43 (t, J = 6.74 Hz, 2 H, CH_2), 2.97 (t, J = 7.25 Hz, 2 H, CH_2), 1.97 – 1.88 (m, 2 H, CH_2), 1.82 – 1.72 (m, 2 H, CH_2), 1.59 – 1.49 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 138.4, 134.9, 132.9, 129.9, 128.1, 126.1, 38.3, 33.6, 32.5, 27.7, 23.1. IR ν (neat, cm^{-1}) 2937, 2863, 1689, 1570, 1254, 1207. The spectra are matching with the known literature.[2]



5g 86%

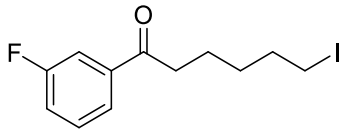
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1g** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5g** (112.0 mg, 86%, 97% purity) (eluent: PE/EA = 10/1): oil. The pure ^1H NMR could be obtained after preparative thin layer chromatography. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 (t, J = 1.92 Hz, 1 H , Ar-H), 7.83 (dt, J = 7.77, 1.41 Hz, 1 H , Ar-H), 7.53 (ddd, J = 8.13, 2.28, 1.09 Hz, 1 H , Ar-H), 7.41 (t, J = 7.84 Hz, 1 H , Ar-H), 3.21 (t, J = 6.95 Hz, 2 H, CH_2), 2.97 (t, J = 7.26 Hz, 2 H, CH_2), 1.93 – 1.84 (m, 2 H, CH_2), 1.81 – 1.72 (m, 2 H, CH_2), 1.55 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 138.4, 134.9, 132.9, 129.9, 128.1, 126.0, 38.3, 33.2, 30.0, 22.8, 6.7. IR ν (neat, cm^{-1}) 2929, 2859, 1689, 1571, 1249, 1201. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}IOCl$ 336.9851; found 336.9852.



4h 81%

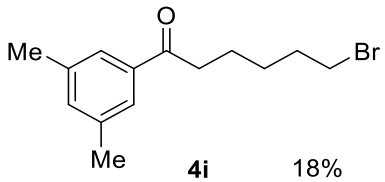
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1h** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4h** (88.5mg, 81%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.74 (dt, J = 7.77, 1.29 Hz, 1 H , Ar-

H), 7.64 (ddd, *J* = 9.43, 2.45, 1.59 Hz, 1 H , Ar-H), 7.45 (td, *J* = 7.99, 5.55 Hz, 1 H , Ar-H), 7.30 – 7.24 (m, 1 H , Ar-H), 3.43 (t, *J* = 6.72 Hz, 2 H, CH_2), 2.98 (t, *J* = 7.26 Hz, 2 H, CH_2), 1.98 – 1.88 (m, 2 H, CH_2), 1.84 – 1.73 (m, 2 H, CH_2), 1.59 – 1.48 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6 (d, *J* = 2.05 Hz), 162.8 (d, *J* = 247.88 Hz), 138.9 (d, *J* = 6.07 Hz), 130.2 (d, *J* = 7.65 Hz), 123.7 (d, *J* = 2.94 Hz), 120.0 (d, *J* = 21.53 Hz), 114.7 (d, *J* = 22.26 Hz), 38.4, 33.6, 32.5, 27.7, 23.1. ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.73. IR ν (neat, cm^{-1}) 2942, 2869, 1685, 1588, 1441, 1256. The spectra are matching with the known literature.[3]



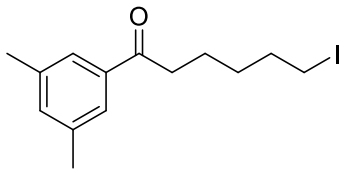
5h 89%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1h** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5h** (115.2 mg, 89%, 97% purity) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dt, *J* = 7.64, 1.27 Hz, 2 H , Ar-H), 7.64 (ddd, *J* = 9.48, 2.67, 1.59 Hz, 1 H , Ar-H), 7.45 (td, *J* = 7.99, 5.53 Hz, 1 H , Ar-H), 7.30 – 7.23 (m, 1 H , Ar-H), 3.21 (t, *J* = 6.95 Hz, 2 H, CH_2), 2.98 (t, *J* = 7.28 Hz, 2H, CH_2), 1.94 – 1.84 (m, 2 H, CH_2), 1.83 – 1.72 (m, 2 H, CH_2), 1.57 – 1.45 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 162.8 (d, *J* = 247.66 Hz), 138.9 (d, *J* = 5.95 Hz), 130.2 (d, *J* = 7.57 Hz), 123.7 (d, *J* = 2.93 Hz), 120.0 (d, *J* = 21.34 Hz), 114.7 (d, *J* = 22.17 Hz), 38.4, 33.2, 30.1, 22.9, 6.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.71. IR ν (neat, cm^{-1}) 2934, 2863, 1690, 1588, 1442, 1251. The spectra are matching with the known literature.[3]



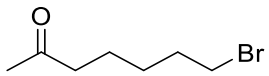
4i 18%

Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1i** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4i** (20.6 mg, 18%) (eluent: PE/EA = 10/1): white solid. mp: 50.7–51.9 ºC (DCM/hexane). ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 2 H , Ar-H), 7.20 (s, 1 H , Ar-H), 3.43 (t, J = 6.77 Hz, 2 H, CH_2), 2.97 (t, J = 7.31 Hz, 2 H, CH_2), 2.37 (s, 6 H, 2 × CH_3), 1.96 – 1.86 (m, 2 H, CH_2), 1.82 – 1.71 (m, 2 H, CH_2), 1.59 – 1.48 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.5, 138.2, 137.0, 134.6, 125.8, 38.3, 33.7, 32.6, 27.8, 23.3, 21.2. IR ν (neat, cm^{-1}) 2937, 2863, 1684, 1604, 1298, 1182. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{19}BrO$ 283.0692; found 283.0688.



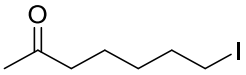
5i 84%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1i** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5i** (111.6 mg, 84%, 93% purity) (eluent: PE/EA = 10/1): white solid. mp: 59.4-60.3 ºC (DCM/hexane). The pure ^1H NMR was obtained after second flash chromatography on silica gel. ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 2 H , Ar-H), 7.19 (s, 1 H , Ar-H), 3.21 (t, J = 7.00 Hz, 2 H, CH_2), 2.96 (t, J = 7.32 Hz, 2 H, CH_2), 2.37 (s, 6 H, 2 × CH_3), 1.95 – 1.84 (m, 2 H, CH_2), 1.80 – 1.70 (m, 2 H, CH_2), 1.54 – 1.43 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 138.1, 137.0, 134.6, 125.8, 38.3, 33.3, 30.1, 23.1, 21.2, 6.8. IR ν (neat, cm^{-1}) 2934, 2861, 1683, 1604, 1181, 1158. The spectra are matching with the known literature.[3]



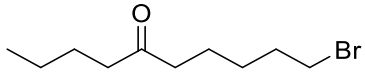
4j 81%

Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.9 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1j** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4j** (63.0 mg, 81%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.41 (t, J = 6.76 Hz, 2 H, CH_2), 2.46 (t, J = 7.32 Hz, 2 H, CH_2), 2.14 (s, 3 H, CH_3), 1.92 – 1.82 (m, 2 H, CH_2), 1.65 – 1.55 (m, 2 H, CH_2), 1.49 – 1.39 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.6, 43.2, 33.5, 32.4, 29.8, 27.5, 22.7. IR ν (neat, cm^{-1}) 2957, 2933, 2868, 1712. The spectra are matching with the known literature.[3]



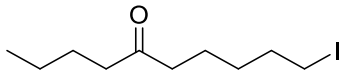
5j 85%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1j** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5j** (81.6 mg, 89%, 95% purity) (eluent: PE/EA = 10/1): oil. The pure ^{1}H NMR was obtained after second flash chromatography on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.19 (t, *J* = 6.93 Hz, 2 H, CH_2), 2.45 (t, *J* = 7.33 Hz, 2 H, CH_2), 2.14 (s, 3 H, CH_3), 1.91 – 1.77 (m, 2 H, CH_2), 1.64 – 1.55 (m, 2 H, CH_2), 1.47 – 1.33 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.8, 43.4, 33.2, 30.0, 29.9, 22.5, 6.8. IR ν (neat, cm^{-1}) 2964, 2930, 2868, 1712. The spectra are matching with the known literature.[3]



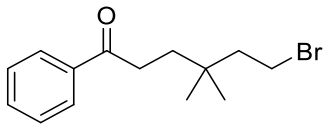
4k 30%

Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1k**(70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4k** (28.1 mg, 30%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 3.41 (t, *J* = 6.87 Hz, 2 H, CH_2), 2.49 – 2.33 (m, 4 H, 2 × CH_2), 1.93 – 1.80 (m, 2 H, CH_2), 1.66 – 1.51 (m, 4 H, 2 × CH_2), 1.48 – 1.39 (m, 2 H, CH_2), 1.36 – 1.26 (m, 2 H, CH_2), 0.91 (t, *J* = 7.33 Hz, 3 H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.1, 42.6, 42.4, 33.6, 32.5, 27.7, 25.9, 22.8, 22.3, 13.8. IR ν (neat, cm^{-1}) 2938, 2857, 1714, 1357, 1585, 1171. The spectra are matching with the known literature.[11]



5k 35%

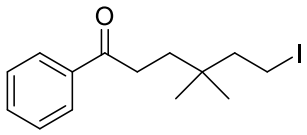
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1k** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5k** (41.8 mg, 35%, 92% purity) (eluent: PE/EA = 10/1): oil. The pure ^1H NMR was obtained after second flash chromatography on silica gel. ^1H NMR (400 MHz, $CDCl_3$) δ 3.19 (t, *J* = 6.97 Hz, 2 H, CH_2), 2.46 – 2.35 (m, 4 H, 2 × CH_2), 1.88 – 1.79 (m, 2 H, CH_2), 1.65 – 1.51 (m, 4 H, 2 × CH_2), 1.44 – 1.25 (m, 4 H, 2 × CH_2), 0.91 (t, *J* = 7.33 Hz, 3 H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.1, 42.6, 42.3, 33.2, 30.0, 25.9, 22.6, 22.3, 13.8, 6.7. IR ν (neat, cm^{-1}) 2933, 2859, 1713, 1426, 1359, 1171. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{10}H_{19}IO$ 283.0653; found 283.0656.



4l 81%

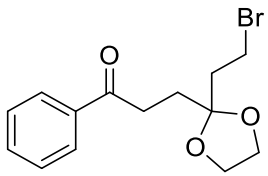
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1l** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and

$NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4l** (90.8 mg, 81%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2 H , Ar-H), 7.61 – 7.54 (m, 1 H , Ar-H), 7.51 – 7.44 (m, 2 H , Ar-H), 3.44 – 3.37 (m, 2 H, CH_2), 2.98 – 2.91 (m, 2 H, CH_2), 1.94 – 1.88 (m, 2 H, CH_2), 1.72 – 1.64 (m, 2 H, CH_2), 0.97 (s, 6 H, 2 × CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 136.9, 133.1, 128.6, 128.0, 45.5, 35.6, 34.1, 33.4, 29.1, 26.6. IR ν (neat, cm^{-1}) 2956, 2868, 1685, 1447, 1217. The spectra are matching with the known literature.[2]



5l 83%

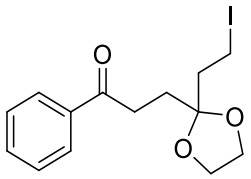
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1l** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5l** (108.9 mg, 83%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.94 (m, 2 H , Ar-H), 7.60 – 7.54 (m, 1 H , Ar-H), 7.52 – 7.43 (m, 2 H , Ar-H), 3.22 – 3.14 (m, 2 H, CH_2), 2.97 – 2.88 (m, 2 H, CH_2), 2.05 – 1.91 (m, 2 H, CH_2), 1.69 – 1.64 (m, 2 H, CH_2), 0.95 (s, 6 H, 2 × CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 136.8, 133.0, 128.6, 128.0, 47.3, 35.4, 35.2, 33.4, 26.3, 0.7. IR ν (neat, cm^{-1}) 2955, 2928, 1685, 1447, 1215. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}IO$ 331.0553; found 331.0550.



4m 40%

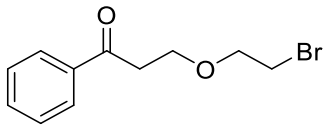
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1m** (70.4 mg, 0.40 mmol), **2** (52.1 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4m** (52.1 mg, 40%) (eluent:

PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2 H , Ar-H), 7.60 – 7.53 (m, 1 H , Ar-H), 7.50 – 7.43 (m, 2 H , Ar-H), 3.97 (s, 4 H, 2 × CH_2), 3.46 – 3.40 (m, 2 H, CH_2), 3.08 – 3.02 (m, 2 H, CH_2), 2.31 – 2.24 (m, 2 H, CH_2), 2.15 – 2.09 (m, 2H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.2, 136.7, 132.9, 128.5, 127.9, 110.0, 65.0, 40.9, 32.7, 31.2, 26.6. IR ν (neat, cm^{-1}) 2964, 2886, 1685, 1123, 1037. The spectra are matching with the known literature.[6]



5m 55%

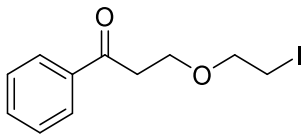
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1m** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5m** (81.1 mg, 55%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2 H , Ar-H), 7.56 (t, *J* = 7.40 Hz, 1 H , Ar-H), 7.46 (t, *J* = 7.63 Hz, 2 H , Ar-H), 3.96 (s, 4H, 2 × CH_2), 3.25 – 3.14 (m, 2 H, CH_2), 3.07 – 3.01 (m, 2 H, CH_2), 2.35 – 2.27 (m, 2 H, CH_2), 2.14 – 2.06 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.3, 136.8, 133.0, 128.5, 127.9, 110.8, 65.1, 42.5, 32.7, 30.7, -2.5. IR ν (neat, cm^{-1}) 2959, 2885, 1684, 1205, 1122. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}O_3I$ 361.02951; found 361.02951.



4n 61%

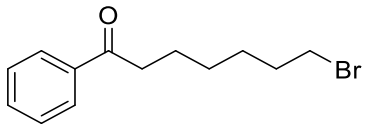
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1n** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4n** (62.0 mg, 61%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.93 (m, 2 H , Ar-H), 7.59 –

7.54 (m, 1 H , Ar-H), 7.49 – 7.43 (m, 2 H , Ar-H), 3.94 (t, J = 6.45 Hz, 2 H, CH_2), 3.80 (t, J = 6.16 Hz, 2 H, CH_2), 3.45 (t, J = 6.16 Hz, 2 H, CH_2), 3.27 (t, J = 6.45 Hz, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 136.7, 133.1, 128.5, 128.0, 70.9, 66.1, 38.5, 30.3. IR ν (neat, cm^{-1}) 2918, 2875, 1684, 1365, 1215, 1119. The spectra are matching with the known literature.[3]



5n 70%

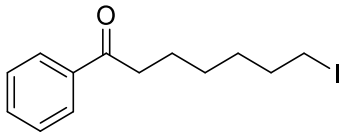
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1n** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5n** (86.1 mg, 70%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.94 (m, 2 H , Ar-H), 7.61 – 7.54 (m, 1 H , Ar-H), 7.49 – 7.43 (m, 2 H , Ar-H), 3.94 (t, *J* = 6.44 Hz, 2 H, CH_2), 3.75 (t, *J* = 6.79 Hz, 2 H, CH_2), 3.26 (dt, *J* = 11.70, 6.54 Hz, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 136.8, 133.2, 128.5, 128.0, 71.6, 65.9, 38.6, 2.9. IR ν (neat, cm^{-1}) 2908, 2877, 1682, 1364, 1214, 1102. The spectra are matching with the known literature.[3]



4o 84%

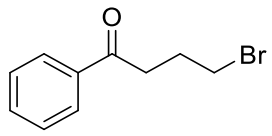
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1o** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4o** (90.3 mg, 84%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.92 (m, 2 H, Ar-H), 7.57 – 7.52 (m, 1 H, Ar-H), 7.48 – 7.42 (m, 2 H, Ar-H), 3.40 (t, *J* = 6.81 Hz, 2 H, CH_2), 2.97 (t, *J* = 7.30 Hz, 2 H, CH_2), 1.92 – 1.82 (m, 2 H, CH_2), 1.80 – 1.70 (m, 2 H, CH_2), 1.54

– 1.36 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.2, 137.0, 132.9, 128.5, 128.0, 38.3, 33.8, 32.5, 28.4, 28.0, 24.0. IR ν (neat, cm^{-1}) 2932, 2855, 1684, 1210. The spectra are matching with the known literature.[3]



5o 82%

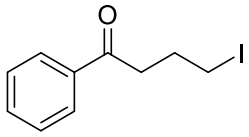
Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1o** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5o** (100.6 mg, 82%, 90% purity) (eluent: PE/EA = 10/1): white solid. mp: 46.0–46.9 ºC (DCM/hexane). The pure ^1H NMR was obtained after second flash chromatography on silica gel. ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2 H, Ar-H), 7.59 – 7.52 (m, 1 H, Ar-H), 7.46 (t, *J* = 7.64 Hz, 2 H, Ar-H), 3.20 (t, *J* = 7.00 Hz, 2 H, CH_2), 2.98 (t, *J* = 7.29 Hz, 2 H, CH_2), 1.88 – 1.80 (m, 2 H, CH_2), 1.80 – 1.71 (m, 2 H, CH_2), 1.51 – 1.36 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 137.1, 133.1, 128.7, 128.1, 38.5, 33.4, 30.4, 28.3, 24.1, 7.2. IR ν (neat, cm^{-1}) 2928, 2855, 1686, 1203. The spectra are matching with the known literature.[3]



4p 65%

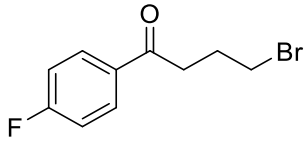
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1p** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4p** (57.6 mg, 65%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.95 (m, 2 H, Ar-H), 7.62 – 7.55 (m, 1 H, Ar-H), 7.52 – 7.45 (m, 2 H, Ar-H), 3.56 (t, *J* = 6.33 Hz, 2 H, CH_2), 3.19 (t, *J* = 7.01 Hz, 2 H, CH_2), 2.39 – 2.25 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ

198.8, 136.7, 133.2, 128.6, 128.0, 36.5, 33.6, 26.8. IR ν (neat, cm^{-1}) 2965, 2922, 1686, 1448, 1222. The spectra are matching with the known literature.[6]



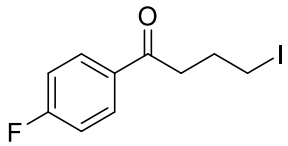
5p 50%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1p** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5p** (54.6 mg, 50%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.96 (m, 2 H, Ar-H), 7.57 (tt, 1H, Ar-H), 7.51 – 7.44 (m, 2 H, Ar-H), 3.33 (t, *J* = 6.62 Hz, 2 H, CH_2), 3.14 (t, *J* = 6.94 Hz, 2 H, CH_2), 2.32 – 2.22 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 136.6, 133.2, 128.6, 128.0, 38.9, 27.4, 6.9. IR ν (neat, cm^{-1}) 2933, 2857, 1681, 1447, 1205. The spectra are matching with the known literature.[12]



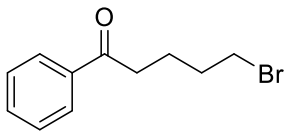
4q 74%

Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1q** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4q** (73.0 mg, 74%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.98 (m, 2 H, Ar-H), 7.18 – 7.10 (m, 2 H, Ar-H), 3.55 (t, *J* = 6.29 Hz, 2 H, Ar-H), 3.16 (t, *J* = 6.94 Hz, 2 H, CH_2), 2.37 – 2.25 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.2, 165.8 (d, *J* = 255.12 Hz), 133.1 (d, *J* = 3.38 Hz), 130.6 (d, *J* = 9.52 Hz), 115.7 (d, *J* = 21.92 Hz), 36.4, 33.6, 26.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.86. IR ν (neat, cm^{-1}) 2961, 2897, 1685, 1597, 1506, 1225, 1156. The spectra are matching with the known literature.[6]



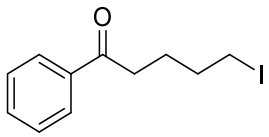
5q 56%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1q** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5q** (65.2 mg, 56%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.98 (m, 2 H, Ar-H), 7.20 – 7.11 (m, 2 H, Ar-H), 3.33 (t, *J* = 6.56 Hz, 2 H, CH_2), 3.12 (t, *J* = 6.92 Hz, 2 H, CH_2), 2.30 – 2.20 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.0, 165.8 (d, *J* = 255.08 Hz), 133.1 (d, *J* = 3.71 Hz), 130.6 (d, *J* = 8.99 Hz), 115.7 (d, *J* = 22.05 Hz), 38.8, 27.4, 6.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.85. IR ν (neat, cm^{-1}) 2932, 2865, 1681, 1597, 1504, 1225, 1156. The spectra are matching with the known literature.[12]



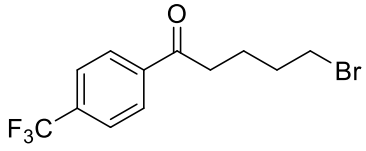
4r 77%

Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1r** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4r** (72.4 mg, 77%) (eluent: PE/EA = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2 H, Ar-H), 7.60 – 7.54 (m, 1 H, Ar-H), 7.50 – 7.44 (m, 3 H, Ar-H), 3.46 (t, *J* = 6.40 Hz, 2 H, CH_2), 3.02 (t, *J* = 6.77 Hz, 2 H, CH_2), 2.02 – 1.86 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.5, 136.8, 133.1, 128.6, 128.0, 37.4, 33.3, 32.2, 22.7. IR ν (neat, cm^{-1}) 2948, 2866, 1676, 1447. The spectra are matching with the known literature.[3]



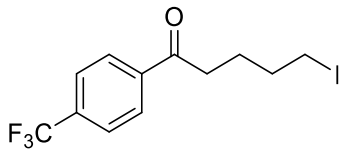
5r 82%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1r** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5r** (93.3 mg, 82%, 97% purity) (eluent: PE/EA = 10/1): oil. The pure ^1H NMR was obtained after preparative thin layer chromatography. ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2 H, Ar-H), 7.60 – 7.54 (m, 1 H, Ar-H), 7.51 – 7.43 (m, 2 H, Ar-H), 3.23 (t, *J* = 6.67 Hz, 2 H, CH_2), 3.01 (t, J = 6.84 Hz, 2 H, CH_2), 2.02 – 1.78 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.5, 136.8, 133.1, 128.6, 128.0, 37.2, 32.9, 25.0, 6.2. IR ν (neat, cm^{-1}) 2946, 2864, 1675, 1446. The spectra are matching with the known literature.[3]



4s 84%

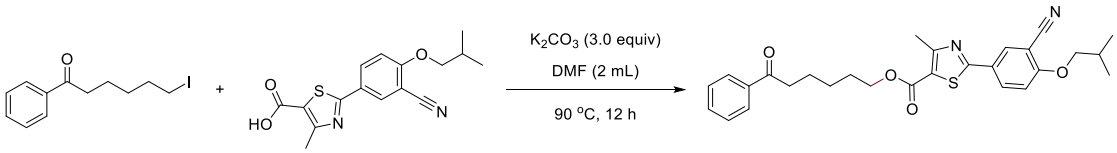
Following Typical Procedure 1, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1s** (70.4 mg, 0.40 mmol), **2** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **4s** (109.1 mg, 84%) (eluent: PE/EA = 10/1): oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.10 – 8.02 (m, 2 H, Ar-H), 7.74 (d, *J* = 8.32 Hz, 2 H, Ar-H), 3.46 (t, *J* = 6.32 Hz, 2 H, CH_2), 3.05 (t, *J* = 6.82 Hz, 2 H, CH_2), 2.04 – 1.87 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.5, 134.5 (q, *J* = 32.91 Hz), 128.4, 125.8 (q, *J* = 3.73 Hz), 123.7 (q, *J* = 272.21 Hz), 37.8, 33.3, 32.1, 22.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.01. IR ν (neat, cm^{-1}) 2933, 2867, 1691, 1408, 1325, 1135, 1107. The spectra are matching with the known literature.[2]



5s 92%

Following Typical Procedure 2, the reaction of $Fe(OAc)_2$ (1.4 mg, 0.008 mmol), *t*-BuONa (2.3 mg, 0.024 mmol), **1s** (70.4 mg, 0.40 mmol), **3** (135.0 mg, 0.60 mmol), and $NaBrO_3$ (60 mg, 0.4 mmol) in MeCN (1.0 mL) afforded **5s** (137.4 mg, 92%, 88% purity) (eluent: PE/EA = 10/1): white solid. mp: 43.9-45.0 ºC (DCM/hexane). The pure ^1H NMR was obtained after second flash chromatography on silica gel. ^1H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.12 Hz, 2 H, Ar-H), 7.74 (d, J = 8.20 Hz, 2 H, Ar-H), 3.24 (t, J = 6.55 Hz, 2 H, CH2), 3.04 (t, J = 6.76 Hz, 2 H, CH_2), 1.99 – 1.84 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 139.3, 134.3 (q, J = 32.69 Hz), 128.3, 125.7 (q, J = 3.82 Hz), 125.5 (q, *J* = 272.21 Hz), 37.5, 32.7, 24.7, 6.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.01. IR ν (neat, cm^{-1}) 2938, 2865, 1693, 1409, 1327, 1125, 1065. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{12}IOF_3$ 356.9958; found 356.9957.

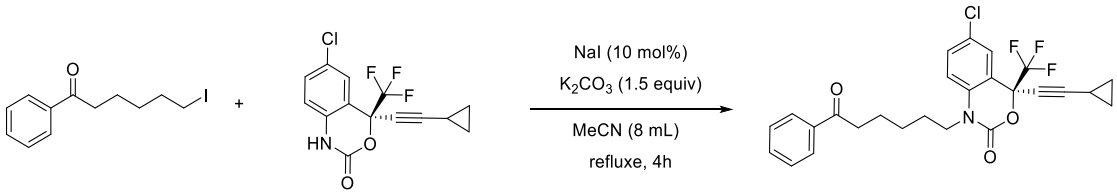
Synthetic potentials



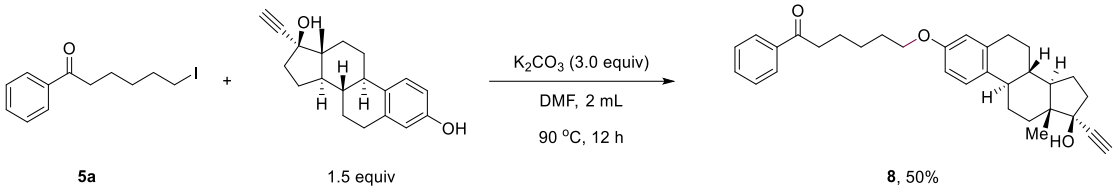
5a 1.5 equiv **6**, 70%

To a 25 mL vial were added the alkyl iodide **5b** (0.4 mmol, 1.0 equiv.), febuxostat (190 mg, 0.6 mmol, 1.5 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 3.0 equiv.), and anhydrous DMF (2.0 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. The reaction mixture was stirred at 90 ºC for 12 h. After completion of the reaction, water was then added and the aqueous layer was extracted with ethyl acetate (20 mL × 3). The organic layer was combined and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to afford the desired product **6** (137.4 mg, 70%) (eluent: PE/EA = 10/1): white solid. mp: 113.7–114.9 ºC (DCM/hexane).[9] ^1H NMR (400 MHz,

$CDCl_3$) δ 8.17 (d, *J* = 2.25 Hz, 1 H, Ar-H), 8.09 (dd, 1 H, Ar-H), 8.01 – 7.93 (m, 2 H, Ar-H), 7.61 – 7.51 (m, 1 H, Ar-H), 7.51 – 7.42 (m, 2 H, Ar-H), 7.01 (d, *J* = 8.90 Hz, 1 H, Ar-H), 4.32 (t, *J* = 6.51 Hz, 2 H, CH_2), 3.90 (d, *J* = 6.50 Hz, 2 H, CH_2), 3.03 (t, *J* = 7.28 Hz, 2 H, CH_2), 2.76 (s, 3 H, CH_3), 2.25 – 2.17 (m, 1 H, CH), 1.88 – 1.78 (m, 4 H, 2 × CH_2), 1.60 – 1.50 (m, 2 H, CH_2), 1.09 (d, *J* = 6.73 Hz, 6 H, 2 × CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 167.1, 162.4, 162.0, 161.1, 136.9, 133.0, 132.5, 132.0, 128.5, 127.9, 125.9, 121.8, 115.3, 112.5, 102.8, 75.6, 65.1, 38.3, 28.5, 28.1, 25.7, 23.8, 19.0, 17.4. IR ν (neat, cm^{-1}) 2958, 2230, 1713, 1693, 1605, 1327, 1264. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{28}H_{30}O_4N_2S$ 491.1999; found 491.1997.

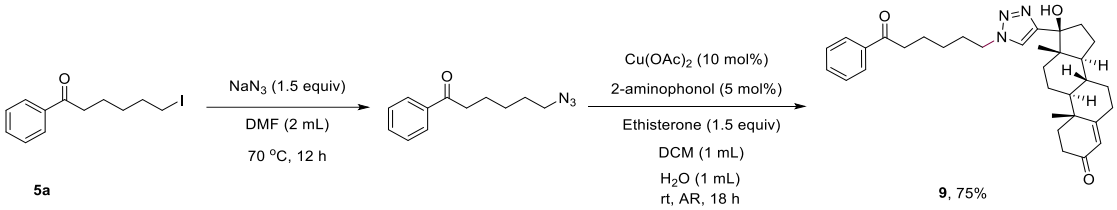


To a 50 mL vial were added the alkyl iodide **5b** (0.4 mmol, 1.0 equiv.), efavirenz (189 mg, 0.6 mmol, 1.5 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 3.0 equiv.), and anhydrous MeCN (8 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. The reaction mixture was refluxe for 4 h. After completion of the reaction, the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to afford the desired product **7** (121.5 mg, 62%) (eluent: PE/EA = 5/1): oil.[13] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2 H, Ar-H), 7.59 – 7.53 (m, 2 H, Ar-H), 7.49 – 7.44 (m, 2 H, Ar-H), 7.42 (dd, *J* = 8.80, 2.43 Hz, 1 H, Ar-H), 6.91 (d, *J* = 8.81 Hz, 1 H, Ar-H), 4.01 – 3.81 (m, 2 H, CH_2), 2.99 (t, *J* = 7.23 Hz, 2 H, CH_2), 1.84 – 1.66 (m, 4 H, 2 × CH_2), 1.52 – 1.43 (m, 2 H, CH_2), 1.42 – 1.35 (m, 1 H, CH), 0.96 – 0.88 (m, 2 H, CH_2), 0.87 – 0.81 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.0, 147.9, 136.9, 135.1, 133.0, 131.5, 128.6, 128.6, 128.2, 128.0, 122.2 (q, *J* = 287.83 Hz), 117.6, 115.0, 95.4, 66.3, 44.3, 38.2, 26.5, 26.2, 23.7, 8.7, 8.7, -0.7. IR ν (neat, cm^{-1}) 2936, 2865, 2251, 1737, 1685, 1495, 1194. HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{26}H_{23}O_3NClF_3$ 512.1211; found 512.1210.



5a 1.5 equiv **8**, 50%

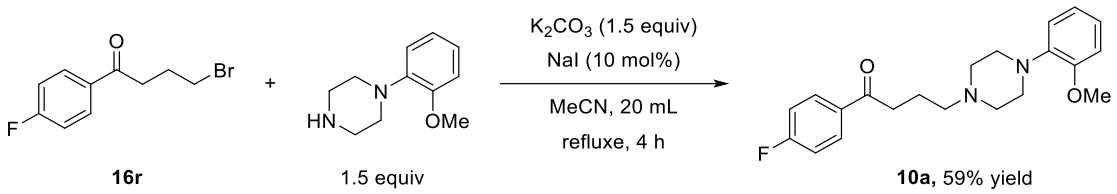
To a 25 mL vial were added the alkyl iodide **5b** (0.4 mmol, 1.0 equiv.), ethynyl estradiol (178 mg, 0.6 mmol, 1.5 equiv.), K_2CO_3 (166 mg, 1.2 mmol, 3.0 equiv.), and DMF (2.0 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. The reaction mixture was stirred at 90 °C for 12 h. After completion of the reaction, water was then added and the aqueous layer was extracted with ethyl acetate (20 mL × 3). The organic layer was separated and dried over anhydrous Na_2SO. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to afford the desired product **8** (92.9 mg, 50%) (eluent: PE/EA = 2/1): white solid. mp: 76.5–77.3 °C (DCM/hexane).[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2 H, Ar-H), 7.58 – 7.53 (m, 1 H, Ar-H), 7.50 – 7.43 (m, 2 H, Ar-H), 7.19 (d, *J* = 8.58 Hz, 1 H, Ar-H), 6.70 (dd, *J* = 8.54, 2.76 Hz, 1 H, Ar-H), 6.62 (d, *J* = 2.70 Hz, 1 H, Ar-H), 3.95 (t, *J* = 6.42 Hz, 2 H, CH_2), 3.00 (t, 2 H, CH_2), 2.87 – 2.81 (m, 2 H, CH_2), 2.60 (s, 1 H, CH), 2.40 – 2.30 (m, 2 H, CH_2), 2.26 – 2.18 (m, 1 H, CH), 2.07 – 1.98 (m, 1 H, CH), 1.97 – 1.68 (m, 10 H, 10 × CH_2), 1.63 – 1.52 (m, 2 H, CH_2), 1.51 – 1.33 (m, 4 H, 2 × CH_2), 0.88 (s, 3 H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 156.9, 137.9, 137.0, 132.9, 132.4, 128.6, 128.0, 126.3, 114.4, 112.0, 87.5, 79.9, 74.0, 67.6, 49.4, 47.1, 43.5, 39.4, 38.9, 38.4, 32.7, 29.8, 29.2, 27.2, 26.4, 25.9, 24.0, 22.8, 12.7. IR ν (neat, cm^{-1}) 3397, 3257, 2931, 2918, 1654, 1058. HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{32}H_{38}O_3$ 493.2713; found 493.2711.



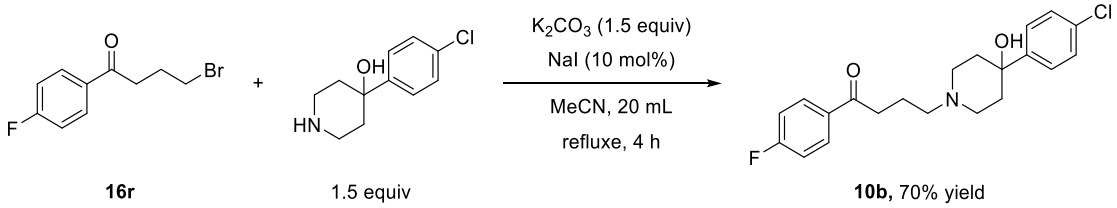
5a **9**, 75%

To a 25 mL vial were added the alkyl iodide **5b** (0.4 mmol, 1.0 equiv.), NaN_3 (39 mg, 0.6 mmol, 1.5 equiv.), and anhydrous DMF (2.0 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. The reaction mixture was stirred at 70 ºC for 12 h. After completion of the reaction, water was then added and the aqueous layer was extracted with ethyl acetate (20 mL × 3). The organic layer was separated and dried over anhydrous Na_2SO. The solvent was removed under reduced pressure and the residue was used without purified. To a 25 mL vial were added the alkyl azide, $Cu(OAc)_2$ (0.04 mmol, 10 mol%), 2-aminophenol (0.02 mmol, 5 mol %), and ethisterone (0.6 mmol, 1.5 equiv) were dissolved in a mixture of DCM (1 mL) and water (1 mL) under the atmosphere of nitrogen. The mixture was stirred at rt for 18 h. After completion, the reaction mixture was were dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel to afford the desired product **9** (159.1 mg, 75%) (eluent: DCM/MeOH = 40/1): yellow solid. mp: 85.0-85.8 ºC (DCM/hexane).[9,13] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.91 (m, 2 H, Ar-H), 7.60 – 7.53 (m, 1 H, Ar-H), 7.49 – 7.42 (m, 3 H, Ar-H), 5.68 (s, 1 H, Ar-H), 4.44 – 4.30 (m, 2 H, CH_2), 3.05 (s, 1 H, CH), 2.97 (t, *J* = 7.05 Hz, 2 H, CH_2), 2.43 – 2.32 (m, 3 H, CH_3), 2.29 – 2.21 (m, 2 H, CH_2), 2.12 (td, *J* = 14.39, 13.15, 3.58 Hz, 1 H, CH), 2.02 – 1.85 (m, 5 H, 5 × CH), 1.84 – 1.73 (m, 2 H, CH_2), 1.69 – 1.30 (m, 10 H, 10 × CH), 1.17 (s, 3 H, CH_3), 1.06 (s, 3 H, CH_3), 0.72 (td, *J* = 11.59, 4.47 Hz, 1 H, CH), 0.47 (td, *J* = 12.52, 4.25 Hz, 1 H, CH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.75, 199.48, 171.25, 153.40, 136.74, 133.08, 128.58, 127.90, 123.72, 121.07, 82.04, 53.14, 49.92, 48.83, 46.75, 38.49, 37.98, 37.72, 36.19, 35.50, 33.83, 32.75, 32.59, 31.48, 30.09, 26.03, 23.57, 23.18, 20.52, 17.32, 14.19. IR ν (neat, cm^{-1}) 3444, 2941, 2860, 2248, 1681, 1448, 1230. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{33}H_{43}O_3N_3$ 530.3377; found 530.3376.

Synthetic of Antipsychotics

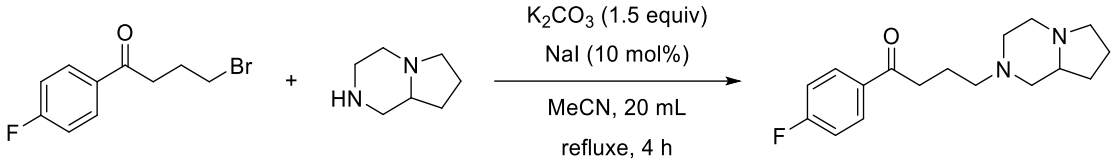


Typical Procedure 3: To a 50 mL vial were added the alkyl bromide **5a** (1 mmol, 1.0 equiv.), secondary amine (1.5 mmol, 1.5 equiv.), NaI (15 mg, 0.1 mmol, 0.1 equiv), K_2CO_3 (207 mg, 1.5 mmol, 1.5 equiv.), and anhydrous MeCN (20 mL) in an Ar glovebox. The vial was sealed and transferred out of glovebox. The reaction mixture was refluxe for 4 h. After completion of the reaction, the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to afford the desired product **10a** (210.2 mg, 59%) (eluent: PE/EA = 1/1): yellow oil.[12] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.96 (m, 2 H, Ar-H), 7.19 – 7.06 (m, 2 H, Ar-H), 7.05 – 6.94 (m, 1 H, Ar-H), 6.99 – 6.87 (m, 2 H, Ar-H), 6.92 – 6.79 (m, 1 H, Ar-H), 3.85 (s, 3 H, CH_3), 3.04 (s, 4 H, 2 × CH_2), 3.01 (t, J = 7.15 Hz, 2 H, CH_2), 2.65 (s, 4 H, 2 × CH_2), 2.49 (t, J = 7.14 Hz, 2 H, CH_2), 2.05 – 1.92 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 165.5 (d, J = 254.26 Hz), 152.2, 141.3, 133.5 (d, J = 2.98 Hz), 130.7, 122.8, 120.9, 118.1, 115.5 (d, J = 21.45 Hz), 111.1, 57.7, 55.3, 53.3, 50.5, 36.2, 21.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.24. IR ν (neat, cm^{-1}) 2924, 2812, 1682, 1596, 1501, 1239, 1156. The spectra are matching with the known literature.[15]



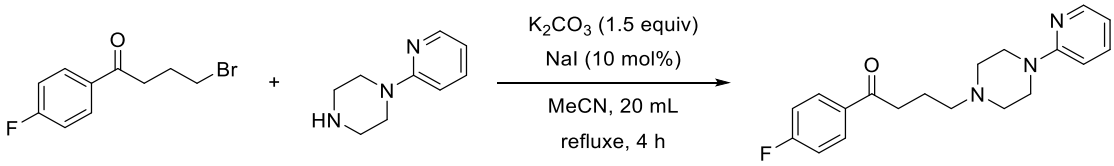
Following Typical Procedure 3, the reaction of alkyl bromide **5a** (1 mmol, 1.0 equiv.), 4-(4-chlorophenyl)piperidin-4-ol (366 mg, 1.5 mmol, 1.5 equiv.), NaI (15 mg, 0.1 mmol, 0.1 equiv), and K_2CO_3 (207 mg, 1.5 mmol, 1.5 equiv.) in MeCN (20.0 mL) afforded **10b** (258.8 mg, 70%) (eluent: DCM/MeOH = 10/1): yellow solid. mp: 152.1-153.4 °C (DCM/hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.95 (m, 2 H, Ar-H), 7.45 – 7.40

(m, 2 H, Ar-H), 7.33 – 7.29 (m, 2 H, Ar-H), 7.18 – 7.11 (m, 2 H, Ar-H), 3.12 (s, 2 H, CH_2), 3.09 (t, J = 6.75 Hz, 2 H, CH_2), 2.89 – 2.74 (m, 4 H, 2 × CH_2), 2.44 – 2.34 (m, 2 H, CH_2), 2.19 – 2.10 (m, 2 H, CH_2), 1.81 (d, J = 12.93 Hz, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 165.8 (d, J = 255.21 Hz), 145.7, 133.1, 130.7 (d, J = 9.39 Hz), 128.5, 126.0, 115.7 (d, J = 21.86 Hz), 70.3, 57.3, 49.1, 37.0, 36.0, 20.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.24. IR ν (neat, cm^{-1}) 3361, 2928, 2718, 1681, 1597, 1233, 1156. The spectra are matching with the known literature.[16]



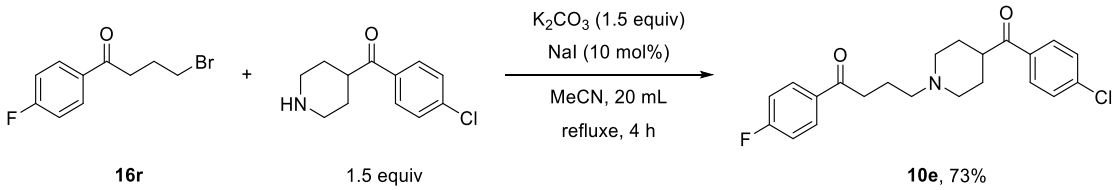
16r 1.5 equiv **10c**, 64% yield

Following Typical Procedure 3, the reaction of alkyl bromide **5a** (1 mmol, 1.0 equiv.), octahydropyrrolo[1,2-a]pyrazine (189 mg, 1.5 mmol, 1.5 equiv.), NaI (15 mg, 0.1 mmol, 0.1 equiv), and K_2CO_3 (207 mg, 1.5 mmol, 1.5 equiv.) in MeCN (20.0 mL) afforded **10c** (188.4 mg, 70%) (eluent: DCM/MeOH = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.95 (m, 2 H, Ar-H), 7.19 – 7.07 (m, 2 H, Ar-H), 3.14 – 3.03 (m, 1 H, Ar-H), 3.00 (q, J = 7.87, 7.10 Hz, 2 H, CH_2), 2.90 – 2.81 (m, 1 H, CH), 2.58 – 2.43 (m, 2 H, CH_2), 2.33 – 2.24 (m, 2 H, CH_2), 2.24 – 2.04 (m, 2 H, CH_2), 2.03 – 1.69 (m, 6 H, 3 × CH_2), 1.51 – 1.36 (m, 1 H, CH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.3, 165.5 (d, J = 254.32 Hz), 133.4 (d, J = 2.89 Hz), 130.6 (d, J = 8.95 Hz), 115.5 (d, J = 21.50 Hz), 62.4, 57.3 (d, J = 2.98 Hz), 53.0, 52.1, 51.1, 36.0, 27.3, 21.5, 21.1. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.24. IR ν (neat, cm^{-1}) 2936, 2798, 1686, 1597, 1229, 1156. The spectra are matching with the known literature.[17]



16r 1.5 equiv **10d**, 75%

Following Typical Procedure 3, the reaction of alkyl bromide **5a** (1 mmol, 1.0 equiv.), 1-(pyridin-2-yl)piperazine (245 mg, 1.5 mmol, 1.5 equiv.), NaI (15 mg, 0.1 mmol, 0.1 equiv.), and K_2CO_3 (207 mg, 1.5 mmol, 1.5 equiv.) in MeCN (20.0 mL) afforded **10d** (251.8 mg, 70%) (eluent: DCM/MeOH = 10/1): yellow solid. mp: 87.2-88.6 ºC (DCM/hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 – 8.17 (m, 1 H, Ar-H), 8.04 – 7.98 (m, 2 H, Ar-H), 7.47 (ddd, *J* = 8.94, 7.19, 1.98 Hz, 1 H, Ar-H), 7.16 – 7.09 (m, 2 H, Ar-H), 6.66 – 6.59 (m, 2 H, Ar-H), 3.49 (t, *J* = 5.10 Hz, 4 H, 2 × CH_2), 3.02 (t, *J* = 7.09 Hz, 2 H, CH_2), 2.55 (t, *J* = 5.08 Hz, 4 H, 2 × CH_2), 2.47 (t, *J* = 7.08 Hz, 2 H, CH_2), 2.04 – 1.95 (m, 2 H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.4, 165.6 (d, *J* = 254.28 Hz), 159.5, 147.9, 137.4, 133.6, 130.6 (d, *J* = 9.42 Hz), 115.6 (d, *J* = 21.91 Hz), 113.2, 107.0, 57.7, 52.9, 45.1, 36.1, 21.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.47. IR ν (neat, cm^{-1}) 2951, 2806, 2772, 1682, 1595, 1314. The spectra are matching with the known literature.[17]



16r 1.5 equiv **10e**, 73%

Following Typical Procedure 3, the reaction of alkyl bromide **5a** (1 mmol, 1.0 equiv.), (4-chlorophenyl)(piperidin-4-yl)methanone (334 mg, 1.5 mmol, 1.5 equiv.), NaI (15 mg, 0.1 mmol, 0.1 equiv), and K_2CO_3 (207 mg, 1.5 mmol, 1.5 equiv.) in MeCN (20.0 mL) afforded **10e** (252.8 mg, 70%) (eluent: DCM/MeOH = 10/1): oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.99 (m, 2 H, Ar-H), 7.90 – 7.83 (m, 2 H, Ar-H), 7.48 – 7.40 (m, 2 H, Ar-H), 7.18 – 7.10 (m, 2 H, Ar-H), 3.23 – 3.13 (m, 1 H, CH), 3.04 – 2.95 (m, 4 H, 2 × CH_2), 2.46 (t, *J* = 7.03 Hz, 2 H, CH_2), 2.14 (t, *J* = 10.61 Hz, 2 H, CH_2), 2.01 – 1.92 (m, 2 H, CH_2), 1.89 – 1.72 (m, 4 H, 2 × CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.4, 198.5, 165.6 (d, *J* = 254.31 Hz), 139.4, 134.3, 133.5, 130.7 (d, *J* = 8.91 Hz), 129.6, 129.0, 115.6 (d, *J* = 21.39 Hz), 57.6, 52.9, 43.4, 36.0, 28.4, 21.4. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.50. IR ν (neat, cm^{-1}) 2932, 2818, 2933, 1689, 1596, 1497, 1238. The spectra are matching with the known literature.[17]

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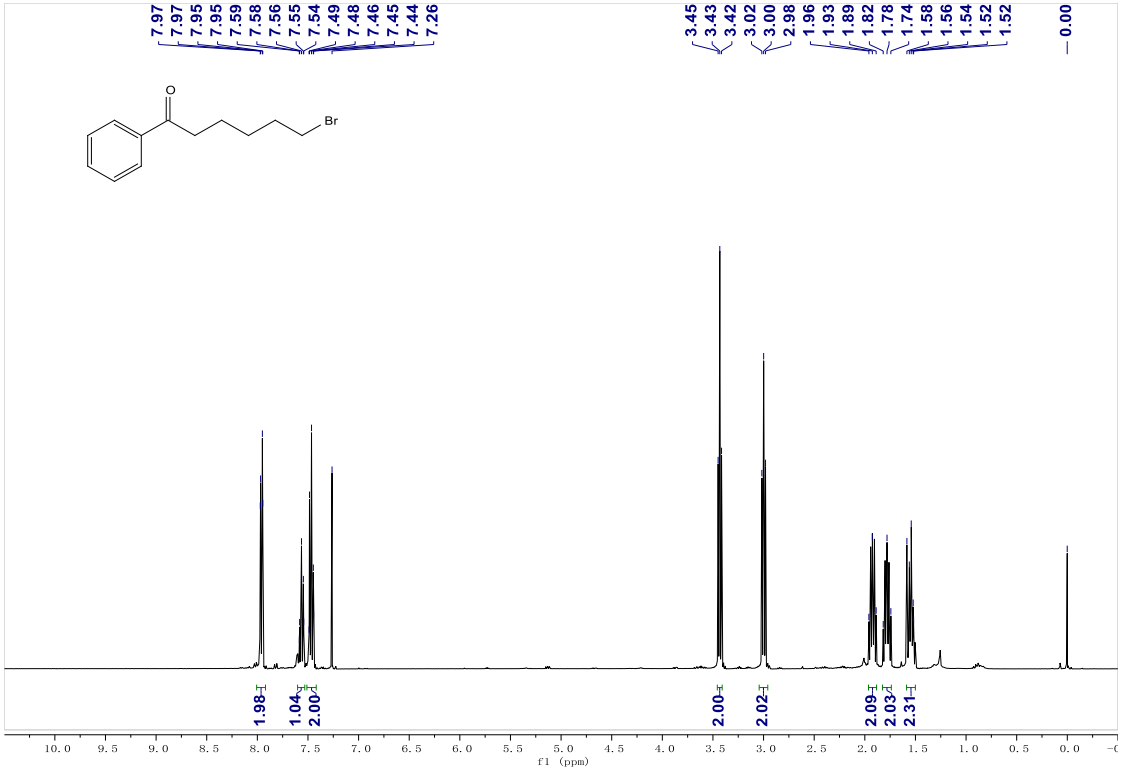
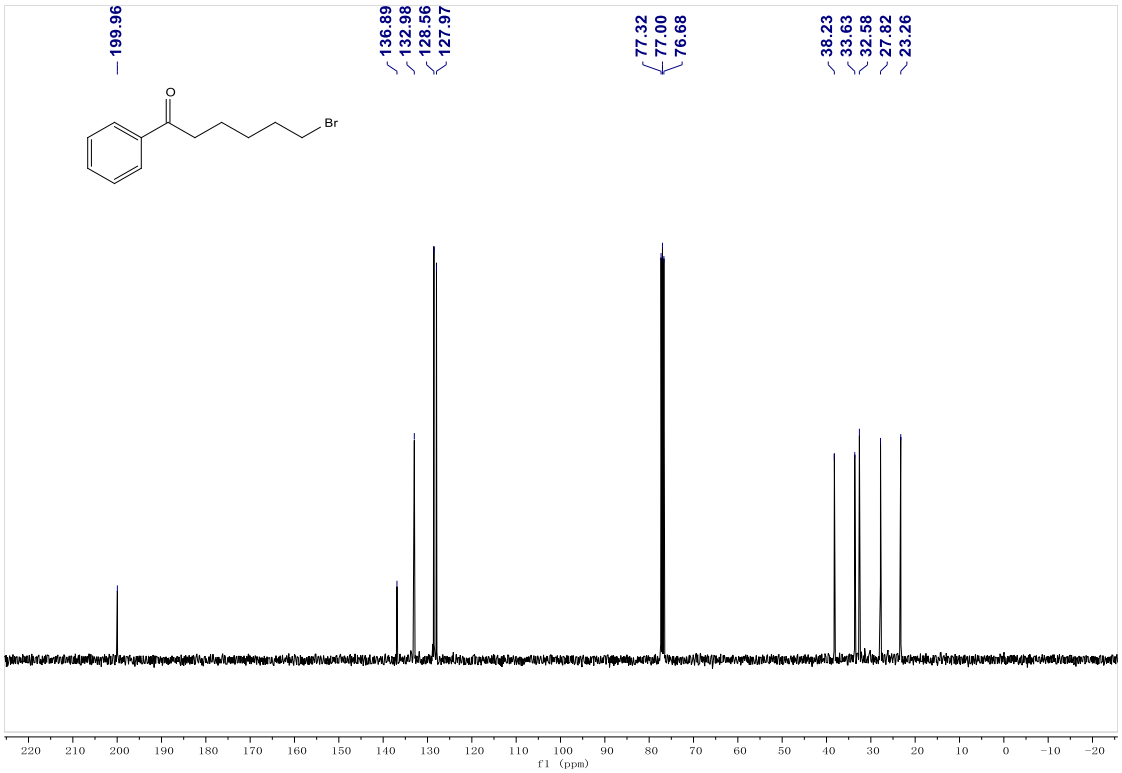
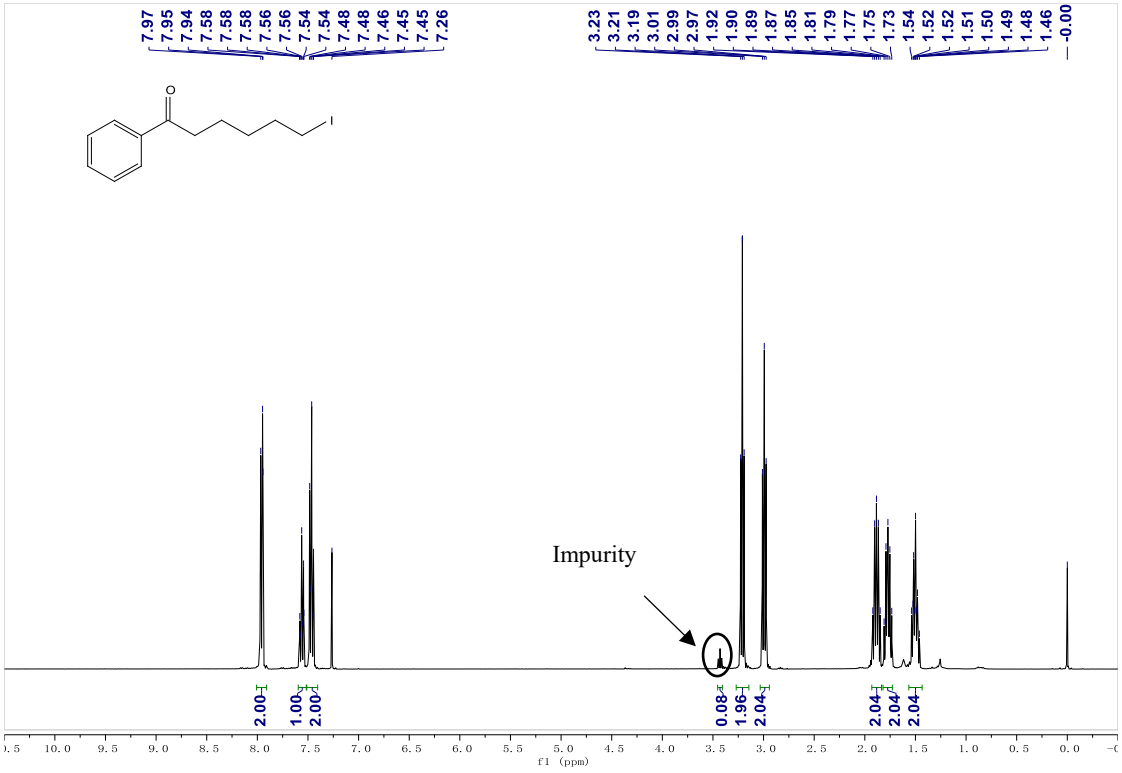
^{1}H NMR (400 MHz, $CDCl_3$) of **4a**



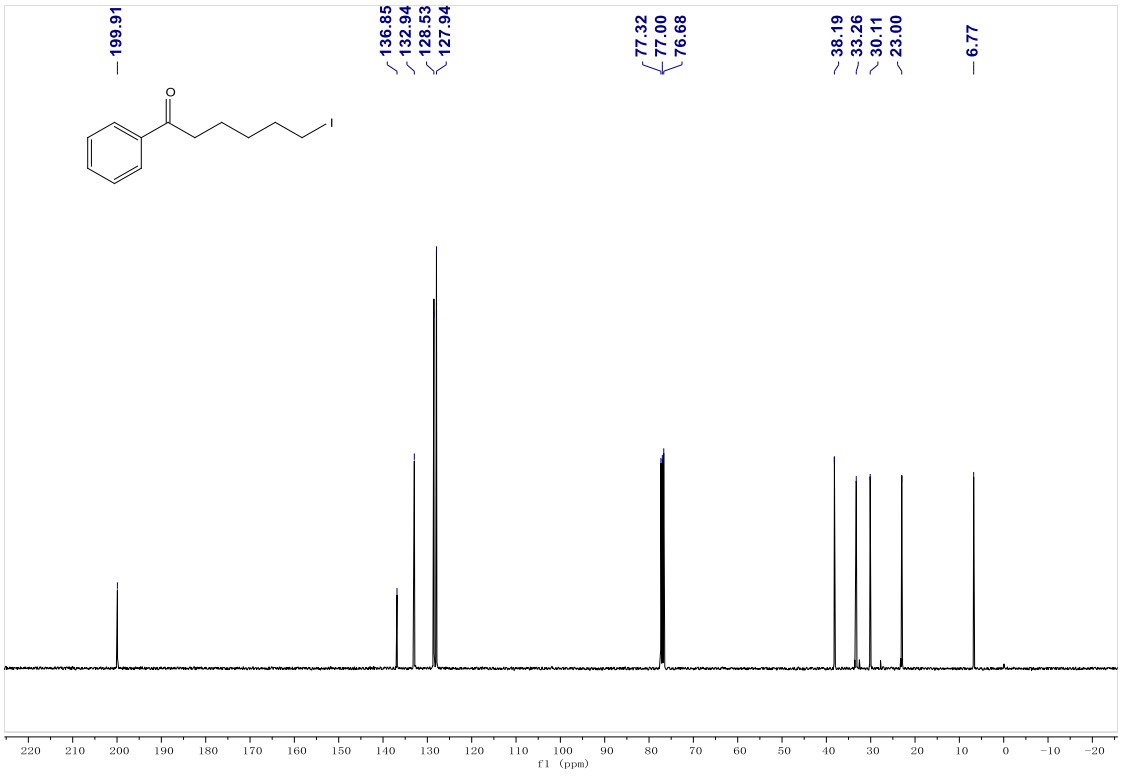
^{13}C NMR (101 MHz, $CDCl_3$) of **4a**



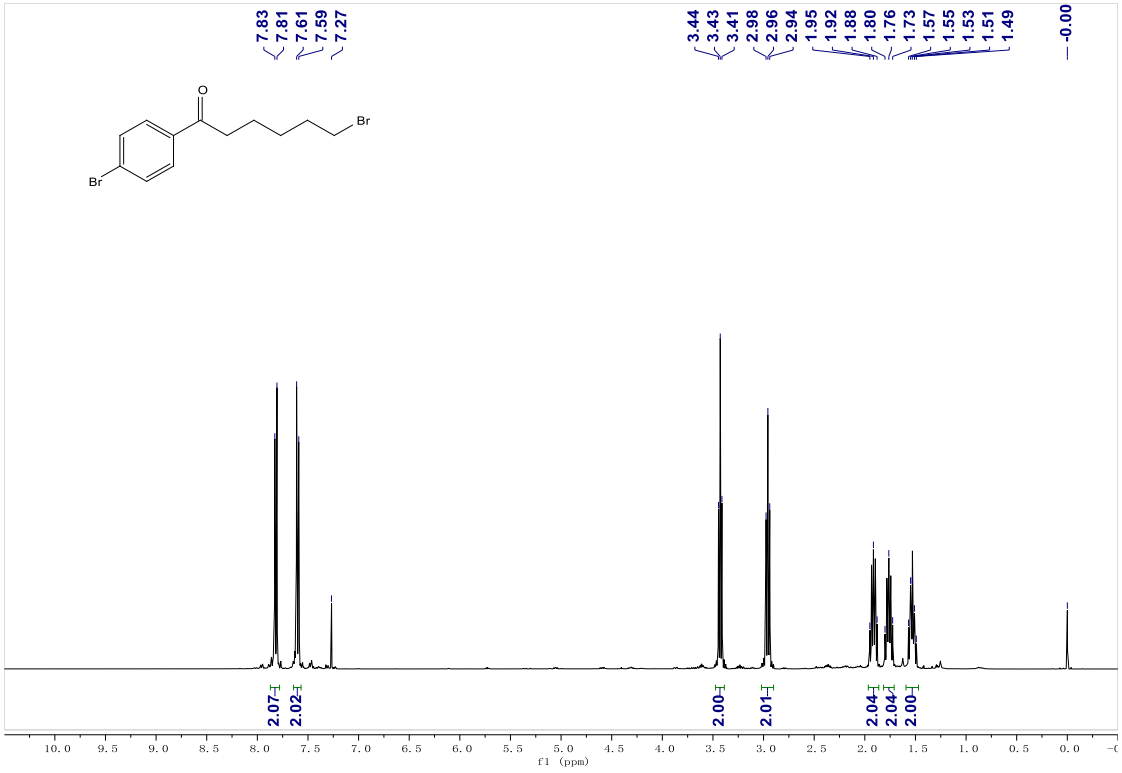
^{1}H NMR (400 MHz, $CDCl_3$) of **5a**



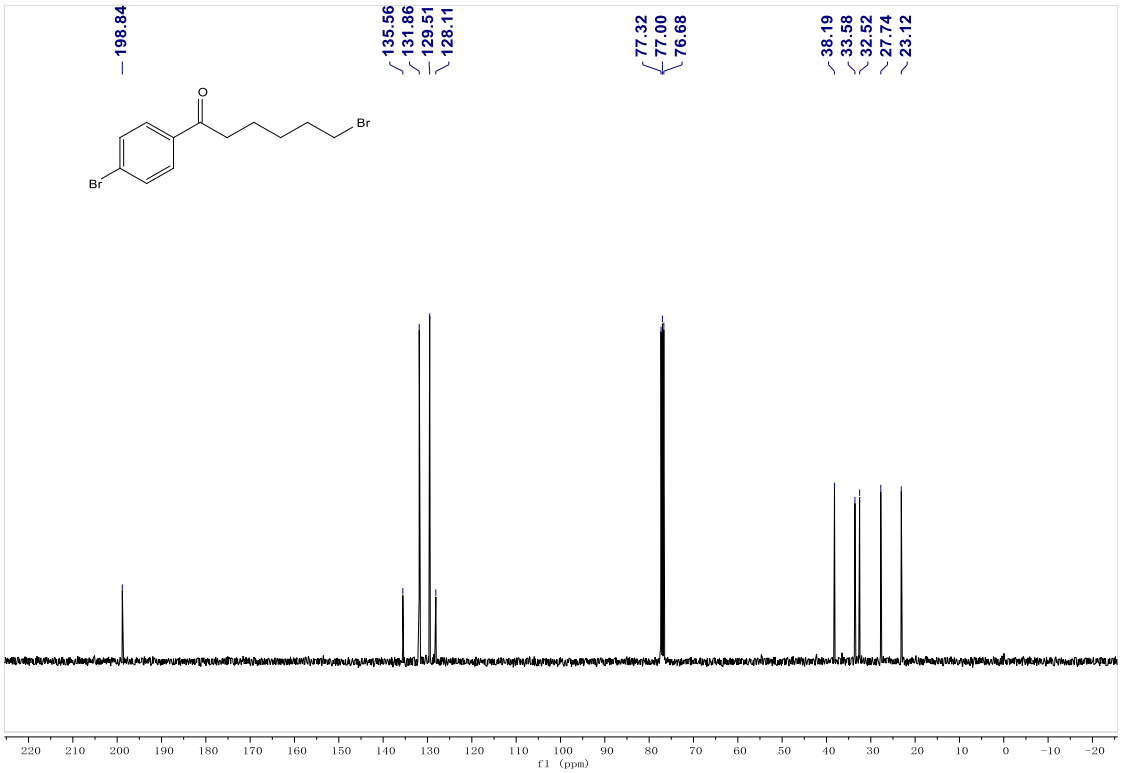
^{13}C NMR (101 MHz, $CDCl_3$) of **5a**



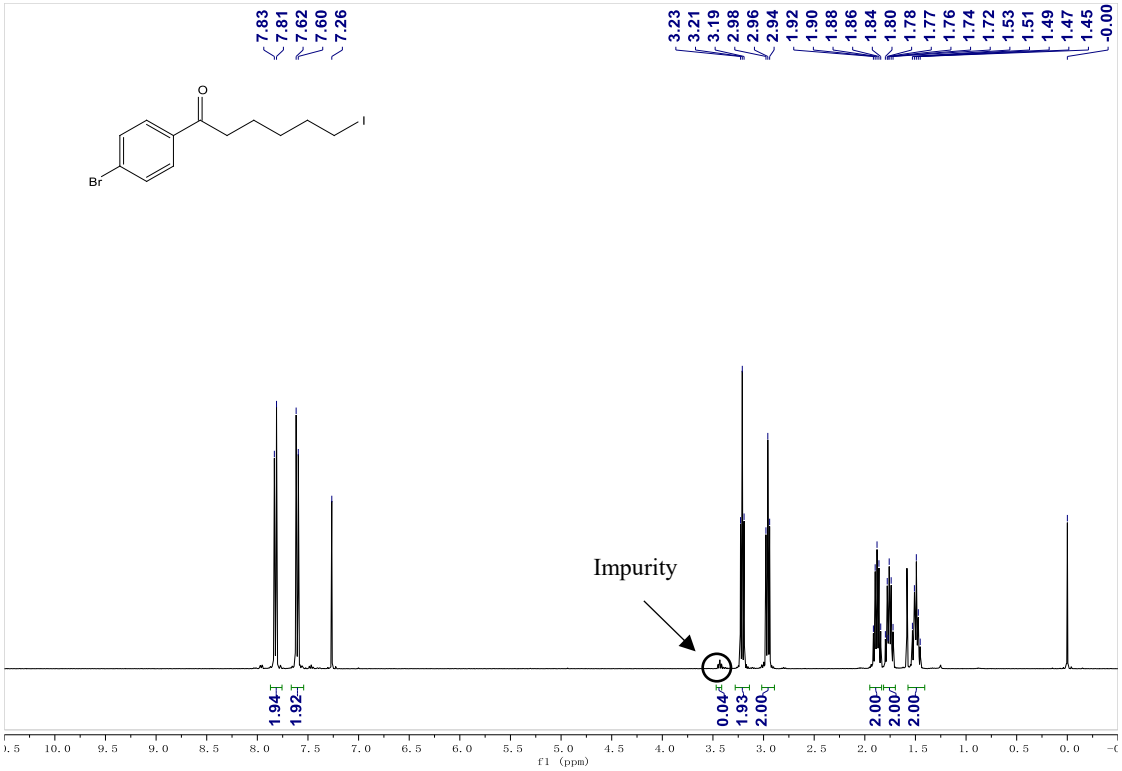
^{1}H NMR (400 MHz, $CDCl_3$) of **4b**



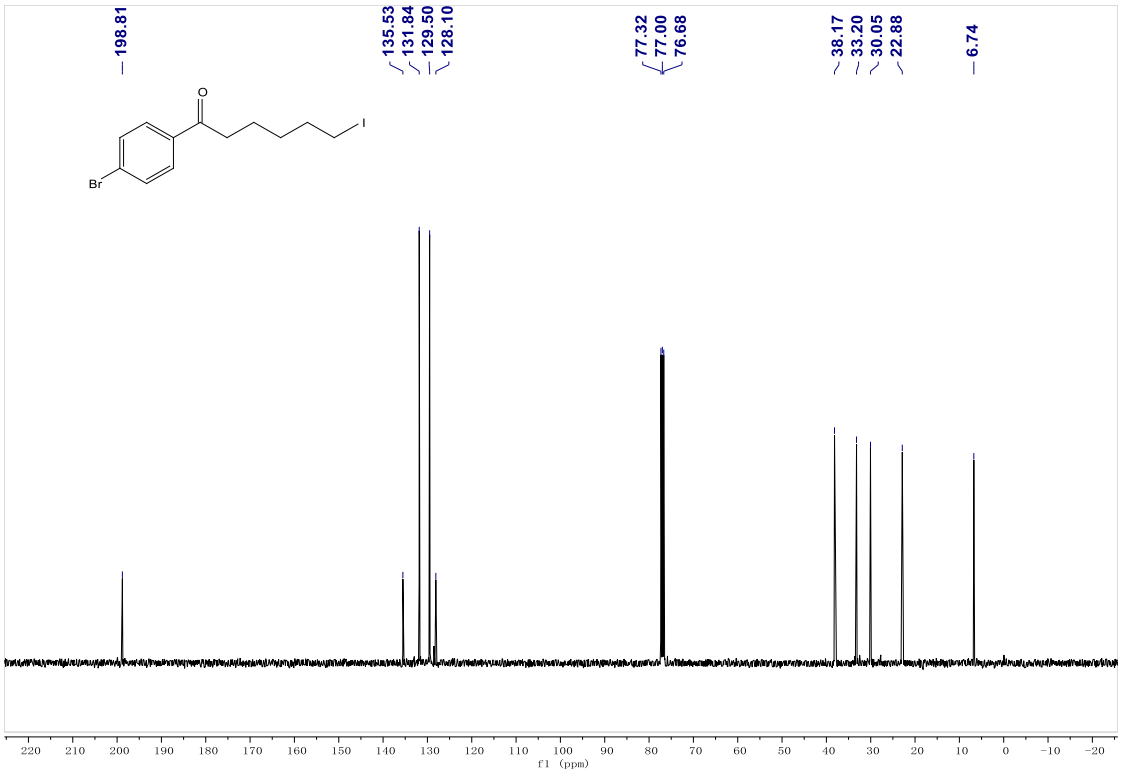
^{13}C NMR (101 MHz, $CDCl_3$) of **4b**



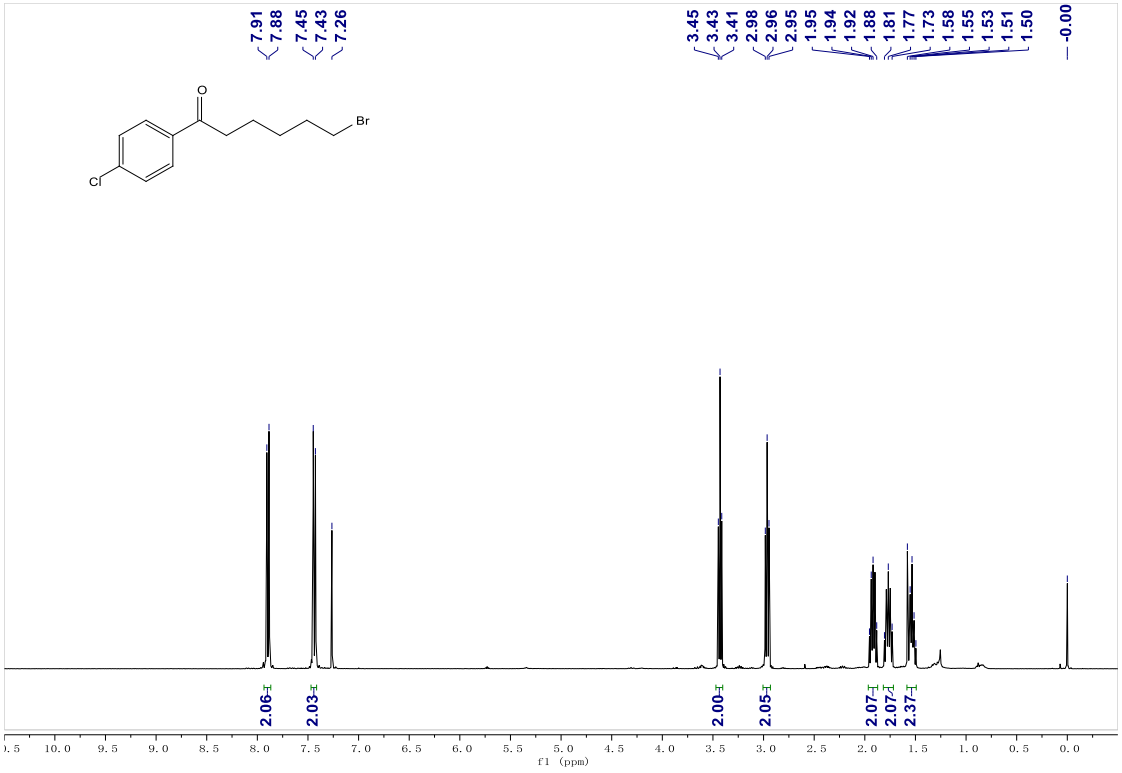
^{1}H NMR (400 MHz, $CDCl_3$) of **5b**



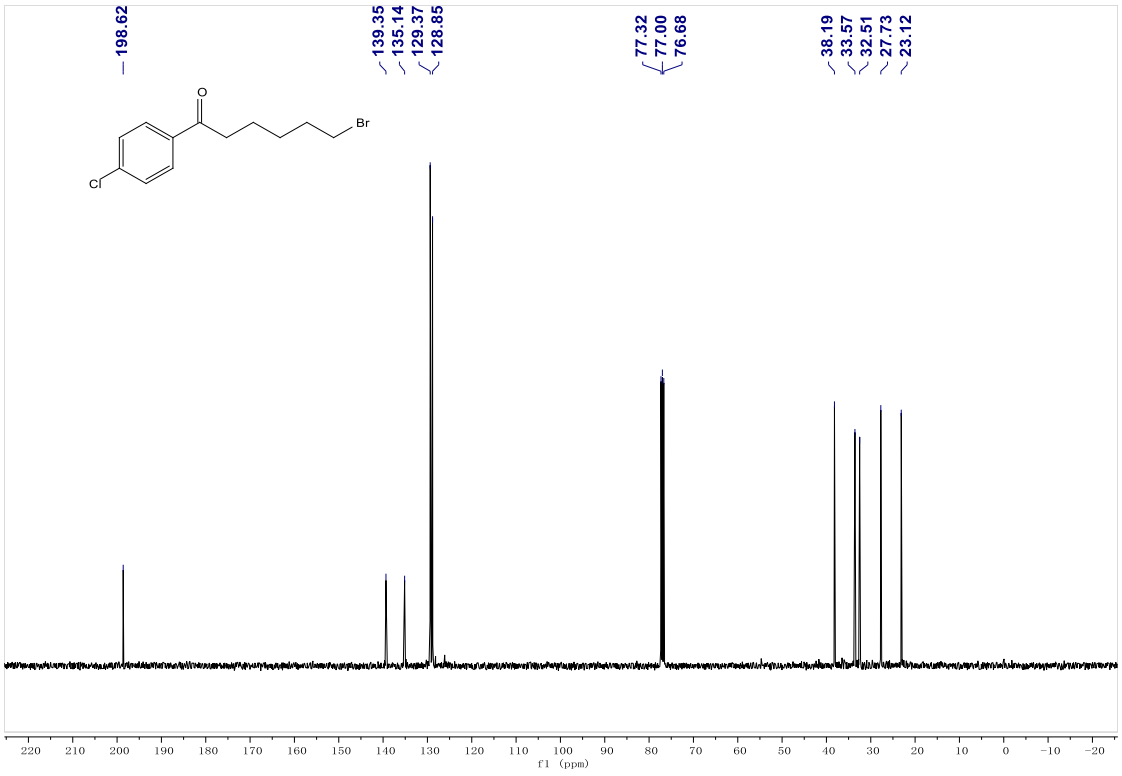
^{13}C NMR (101 MHz, $CDCl_3$) of **5b**



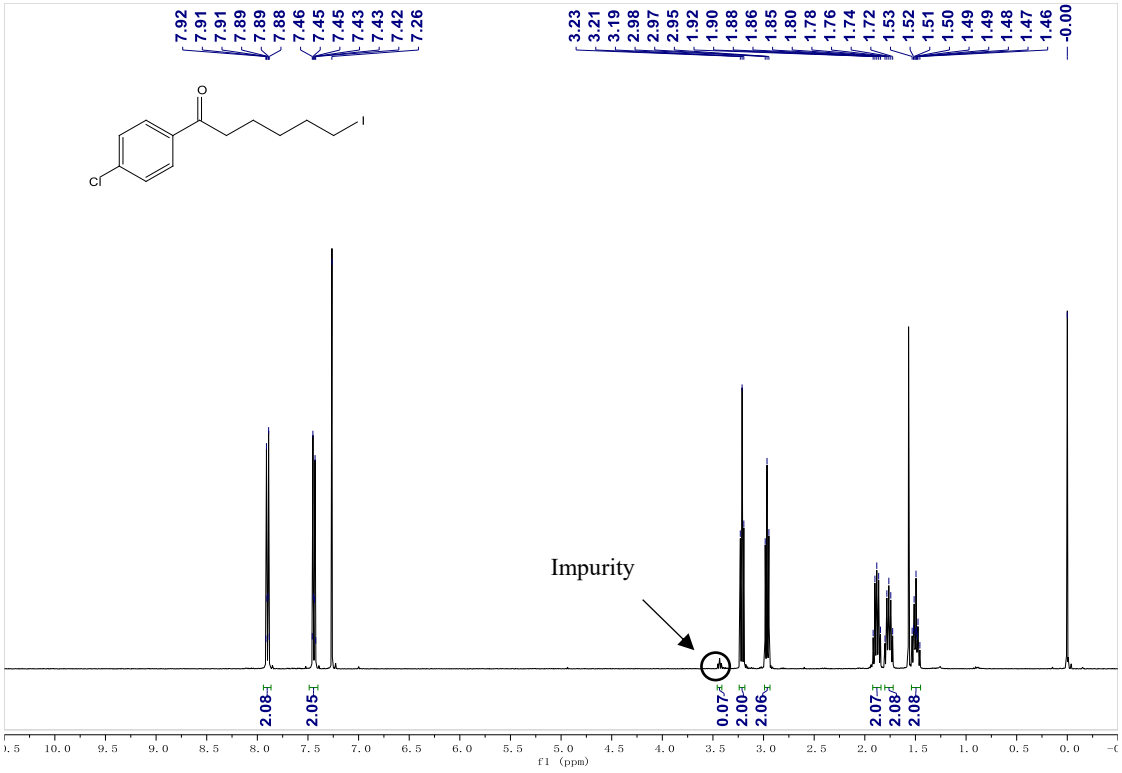
^{1}H NMR (400 MHz, $CDCl_3$) of **4c**



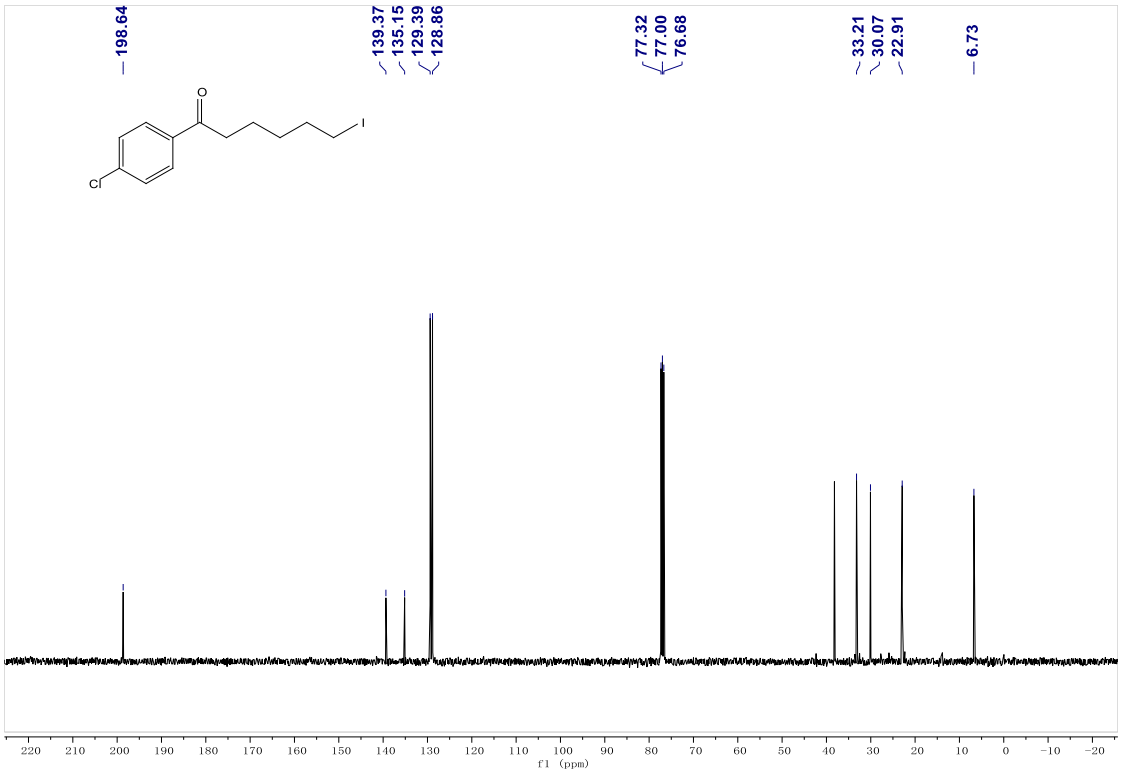
^{13}C NMR (101 MHz, $CDCl_3$) of **4c**



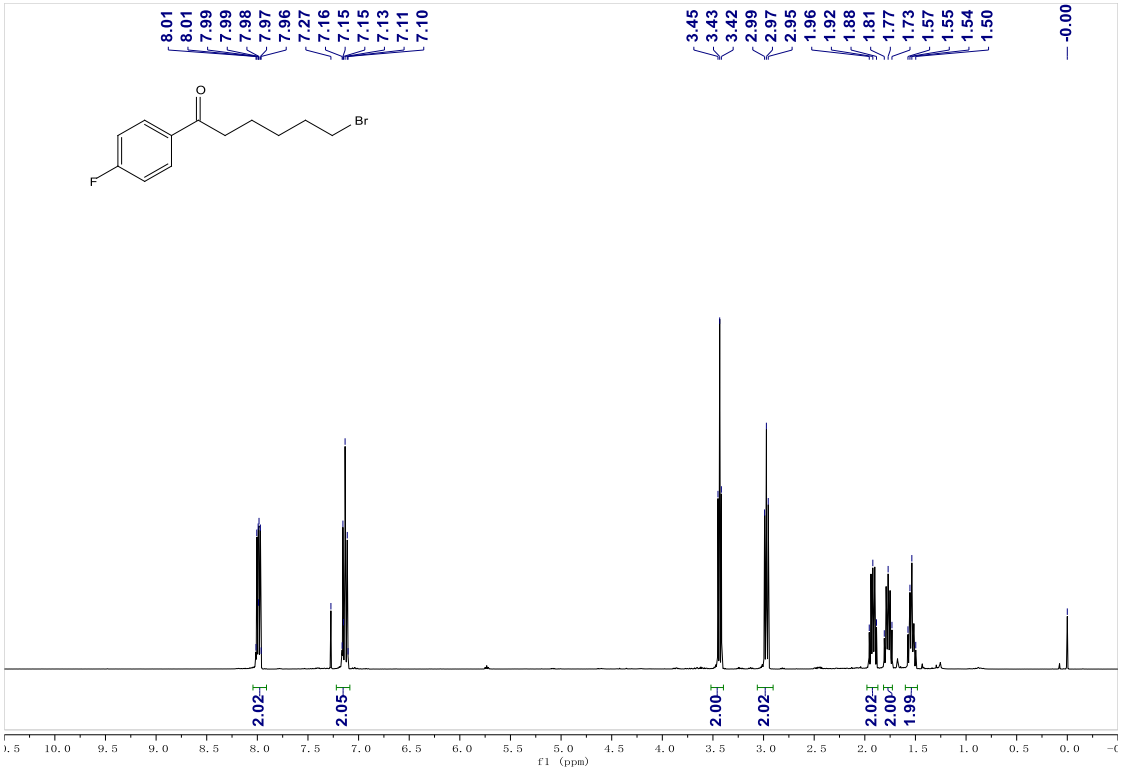
^{1}H NMR (400 MHz, $CDCl_3$) of **5c**



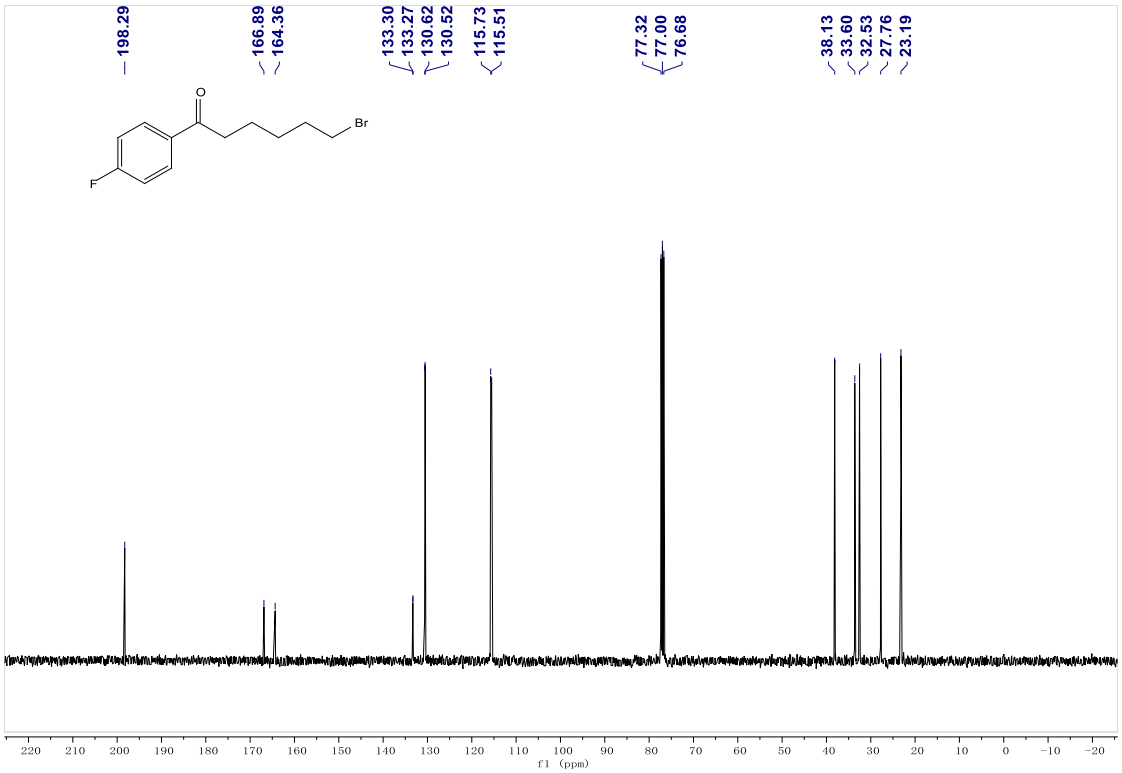
^{13}C NMR (101 MHz, $CDCl_3$) of **5c**



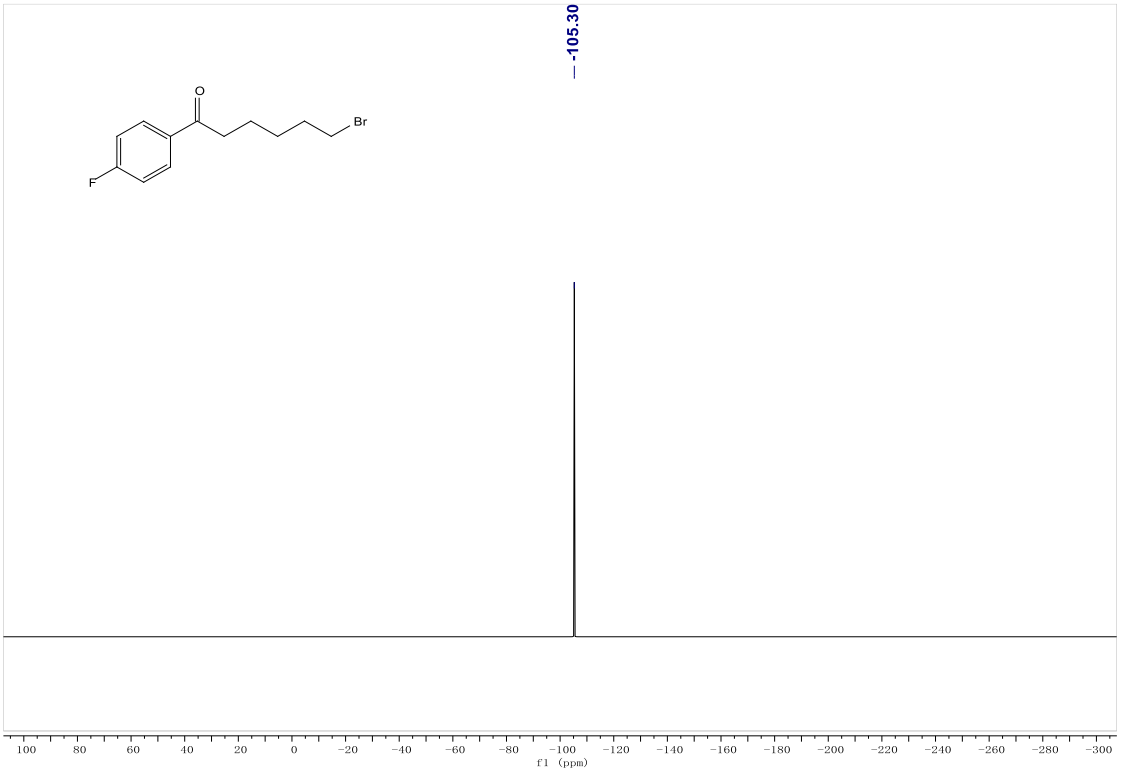
^{1}H NMR (400 MHz, $CDCl_3$) of **4d**



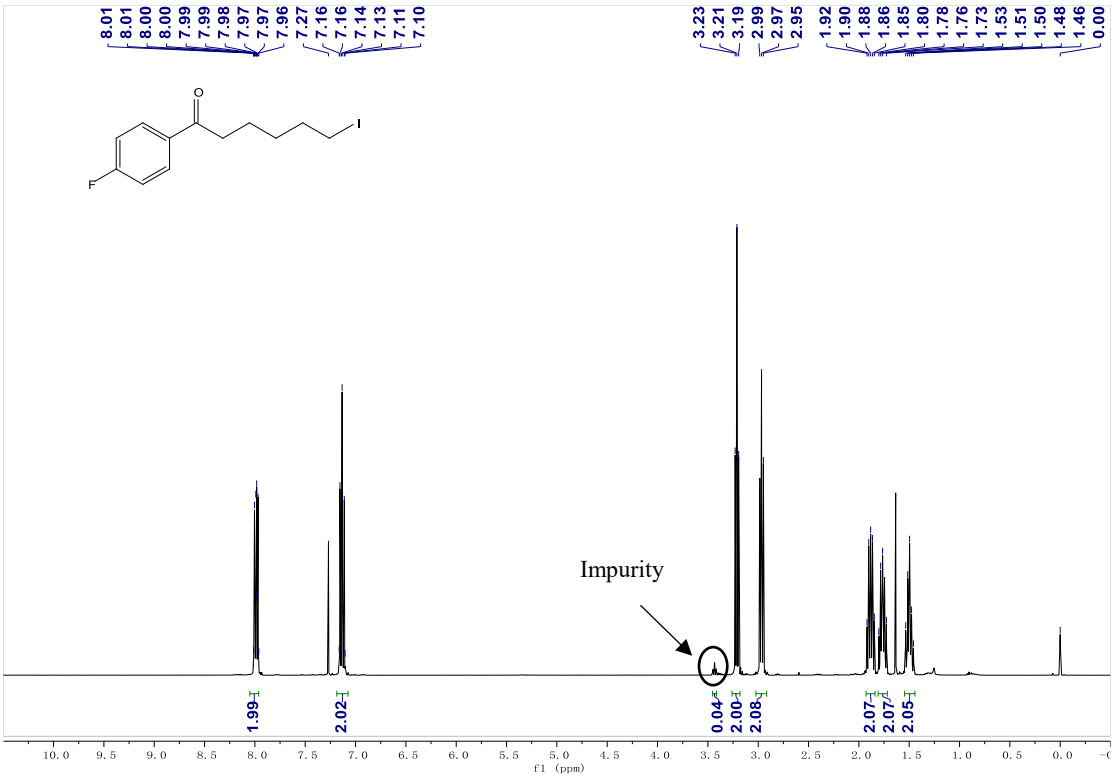
^{13}C NMR (101 MHz, $CDCl_3$) of **4d**



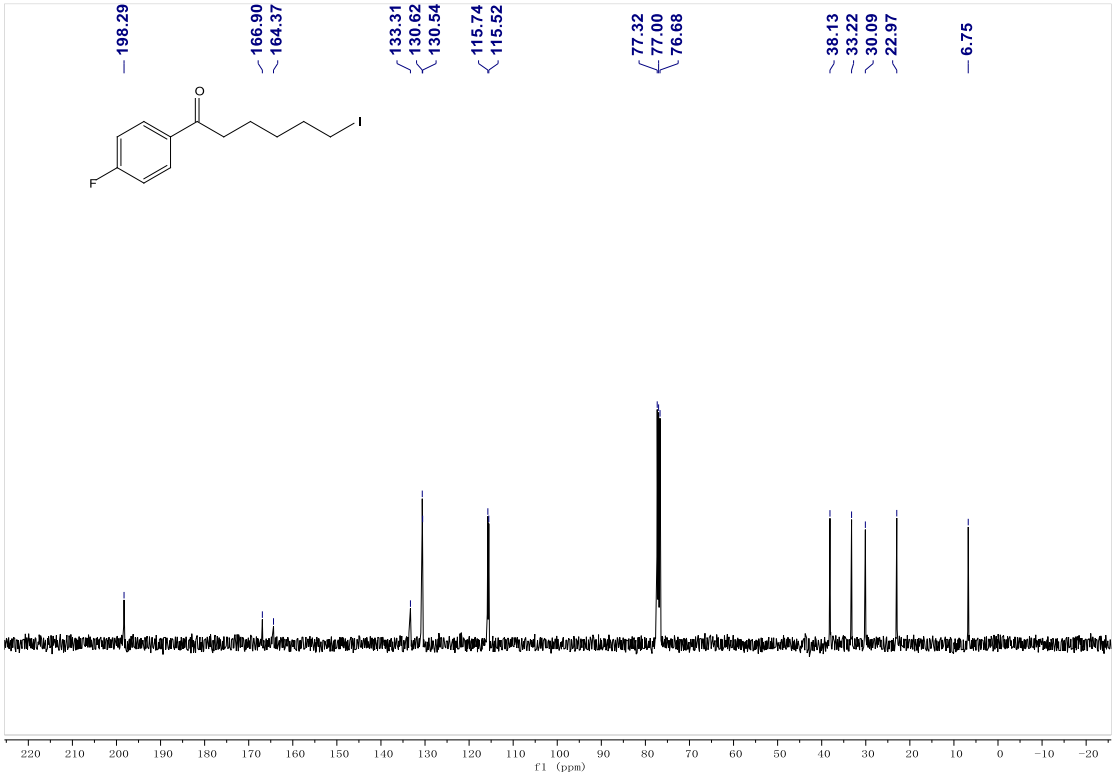
^{19}F NMR (376 MHz, $CDCl_3$) of **4d**



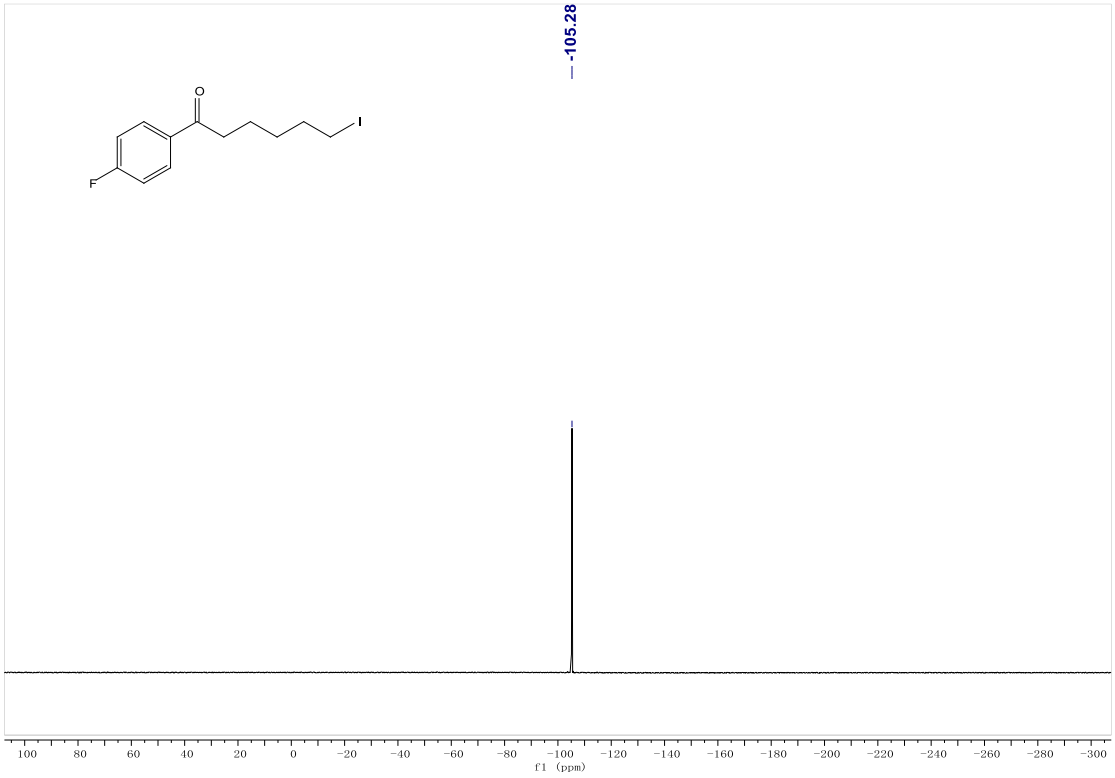
^{1}H NMR (400 MHz, $CDCl_3$) of **5d**



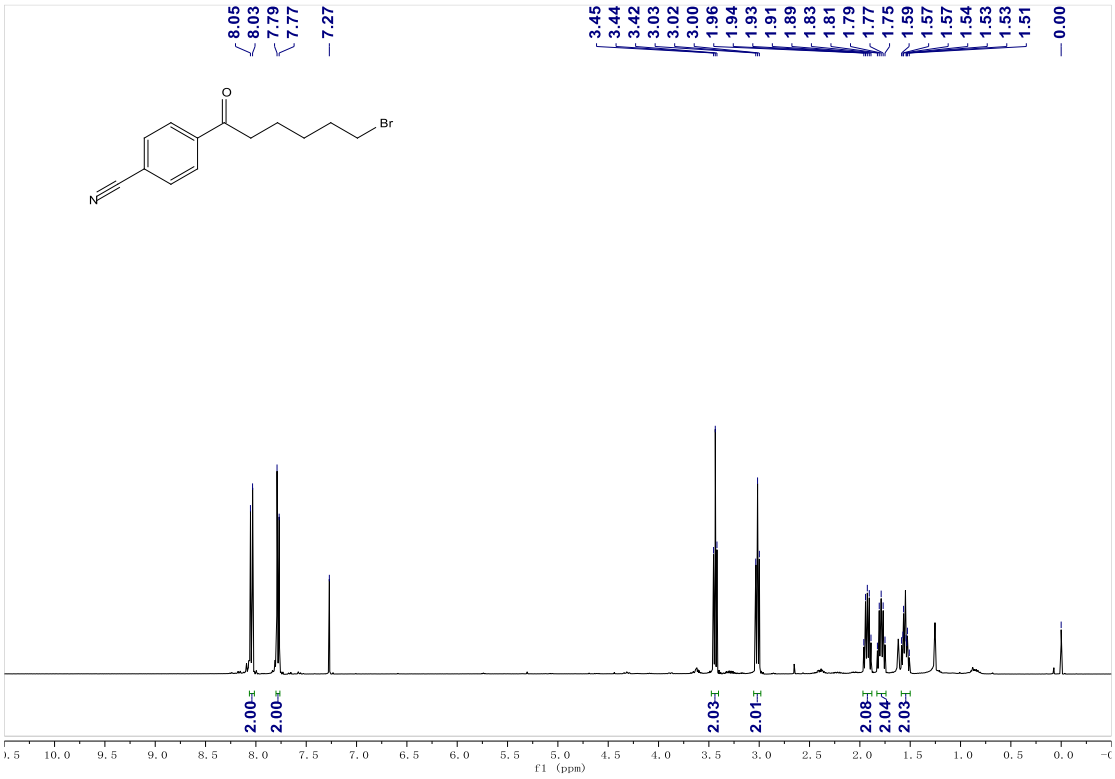
^{13}C NMR (101 MHz, $CDCl_3$) of **5d**



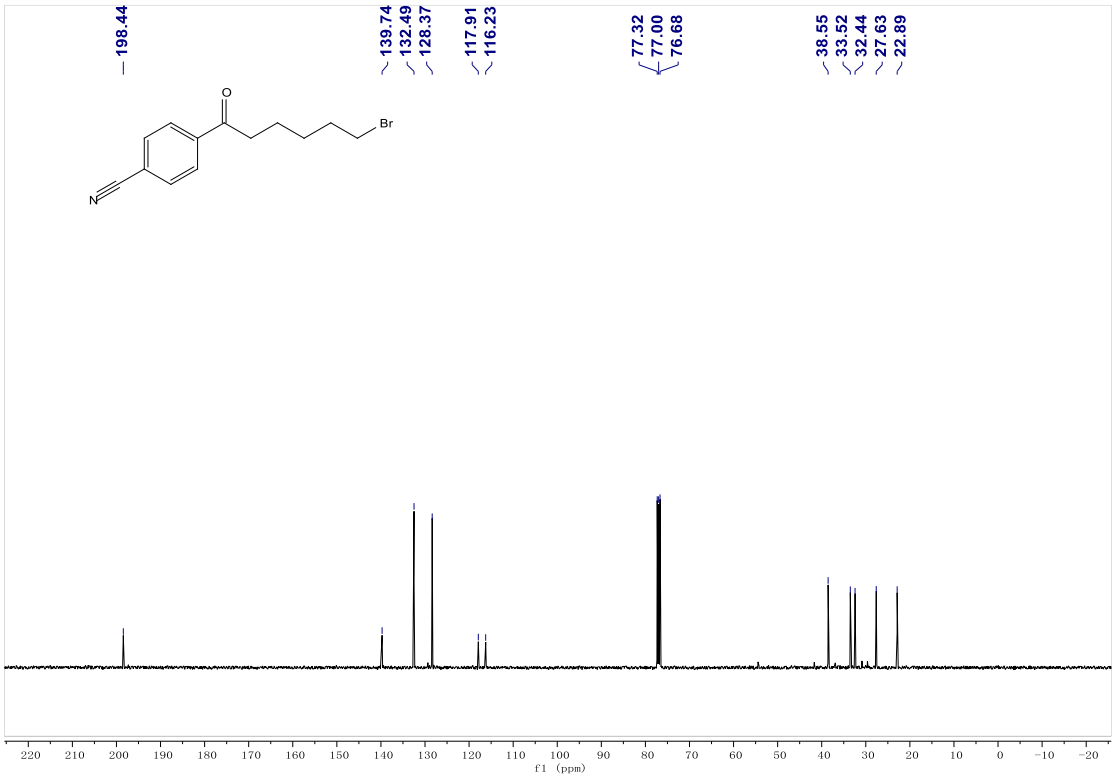
^{19}F NMR (376 MHz, $CDCl_3$) of **5d**



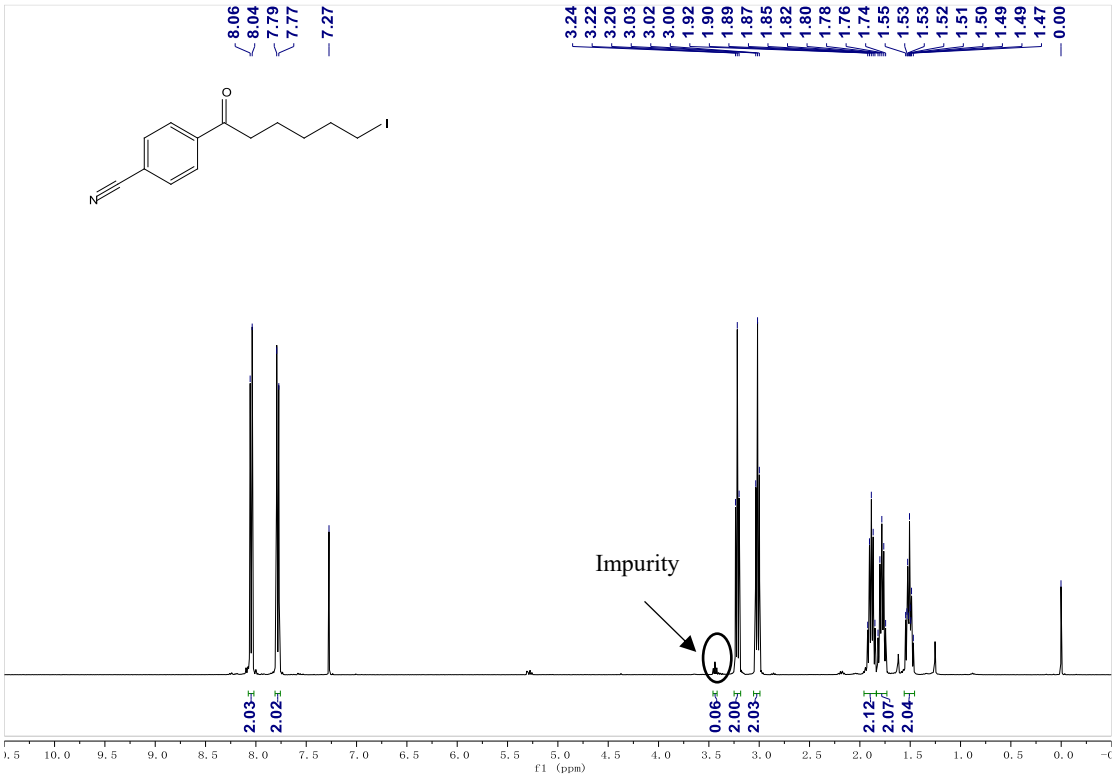
^{1}H NMR (400 MHz, $CDCl_3$) of **4e**



^{13}C NMR (101 MHz, $CDCl_3$) of **4e**



^{1}H NMR (400 MHz, $CDCl_3$) of **5e**



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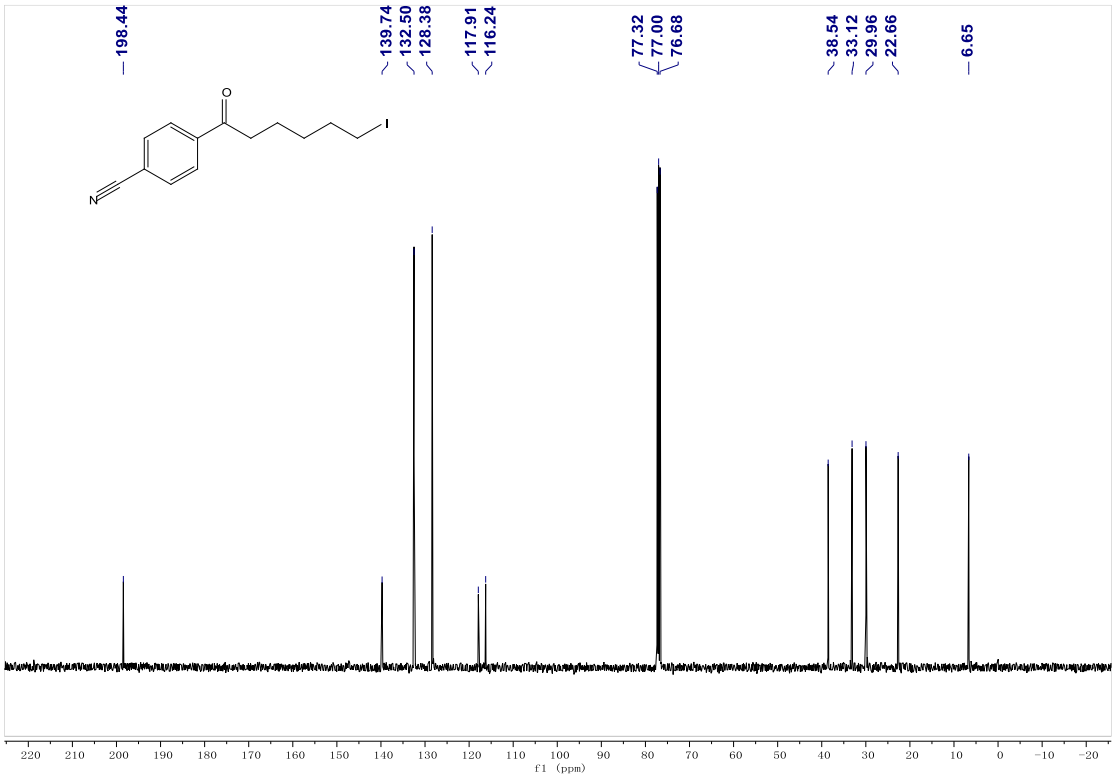

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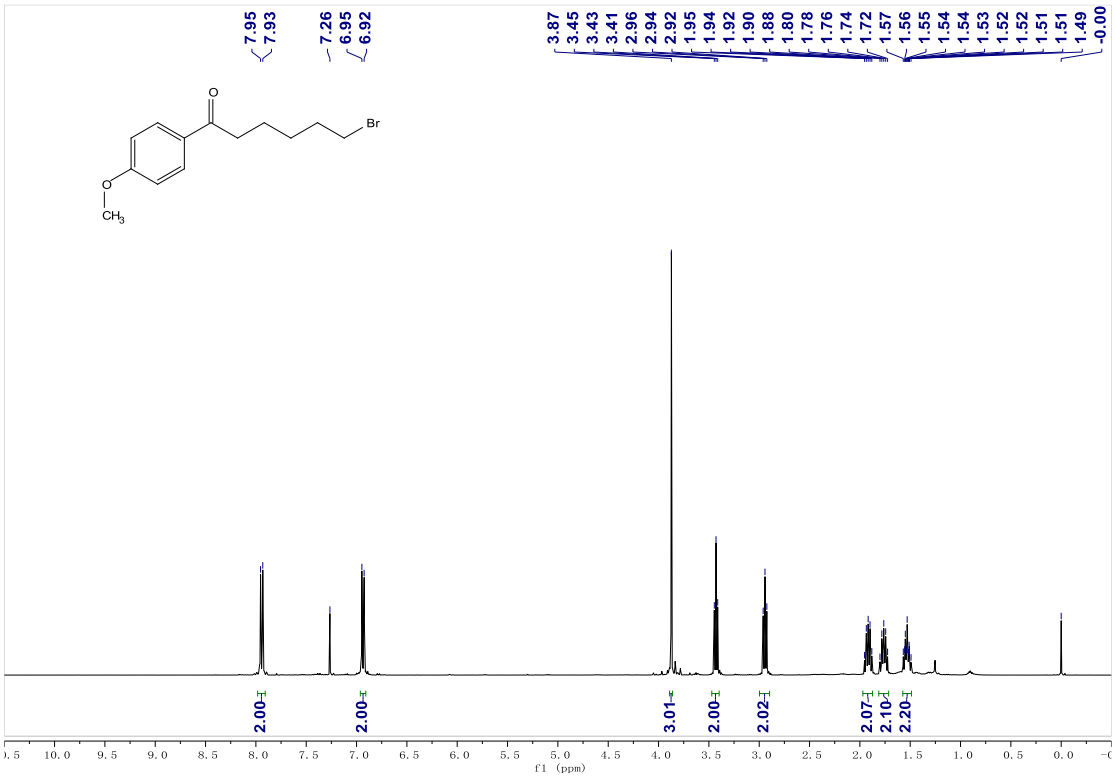
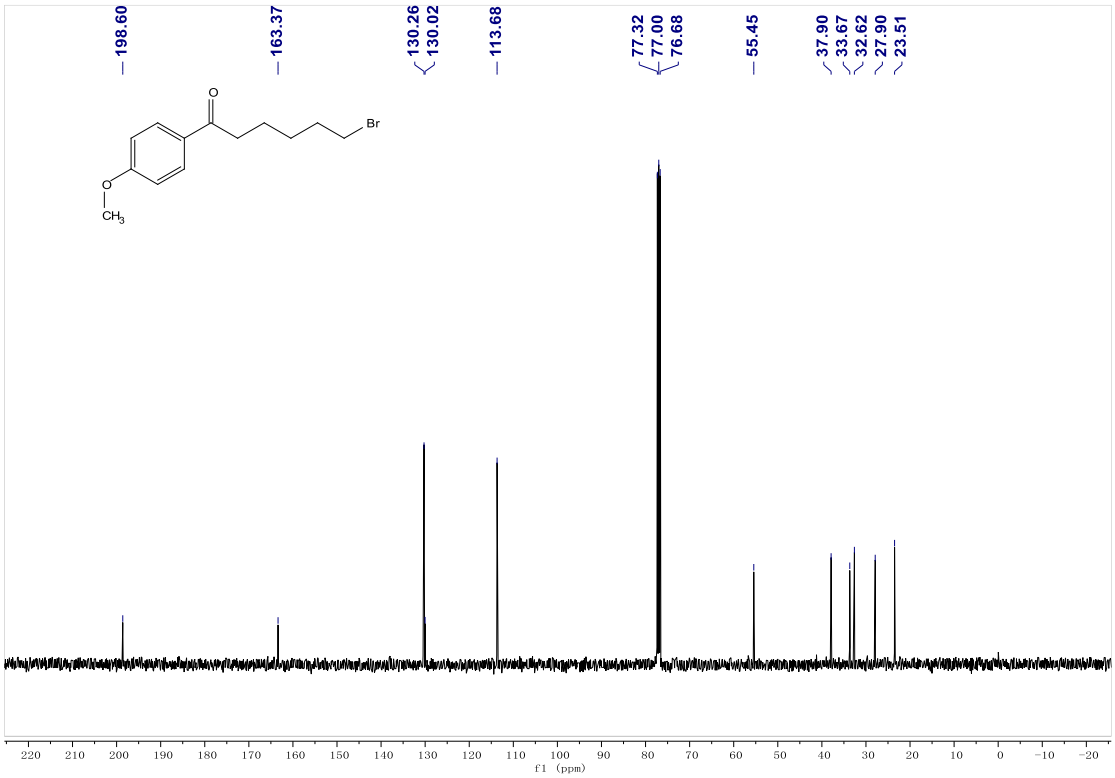
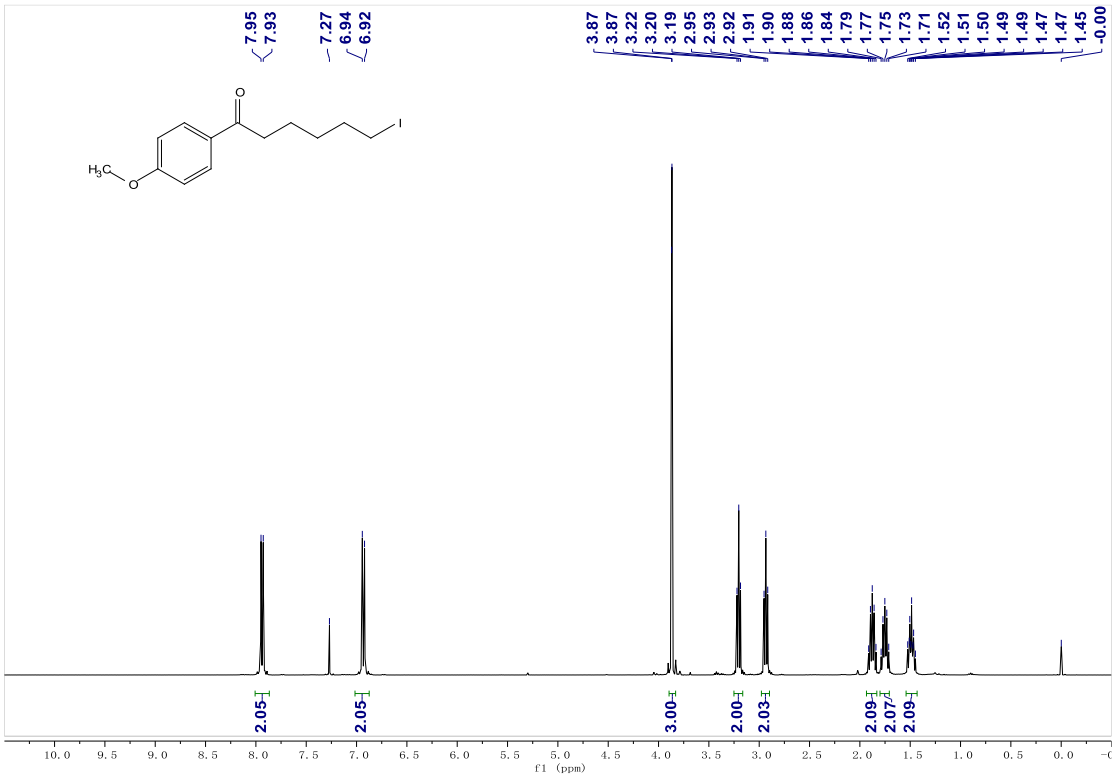


^{13}C NMR (101 MHz, $CDCl_3$) of **4f**



^{1}H NMR (400 MHz, $CDCl_3$) of **5f**



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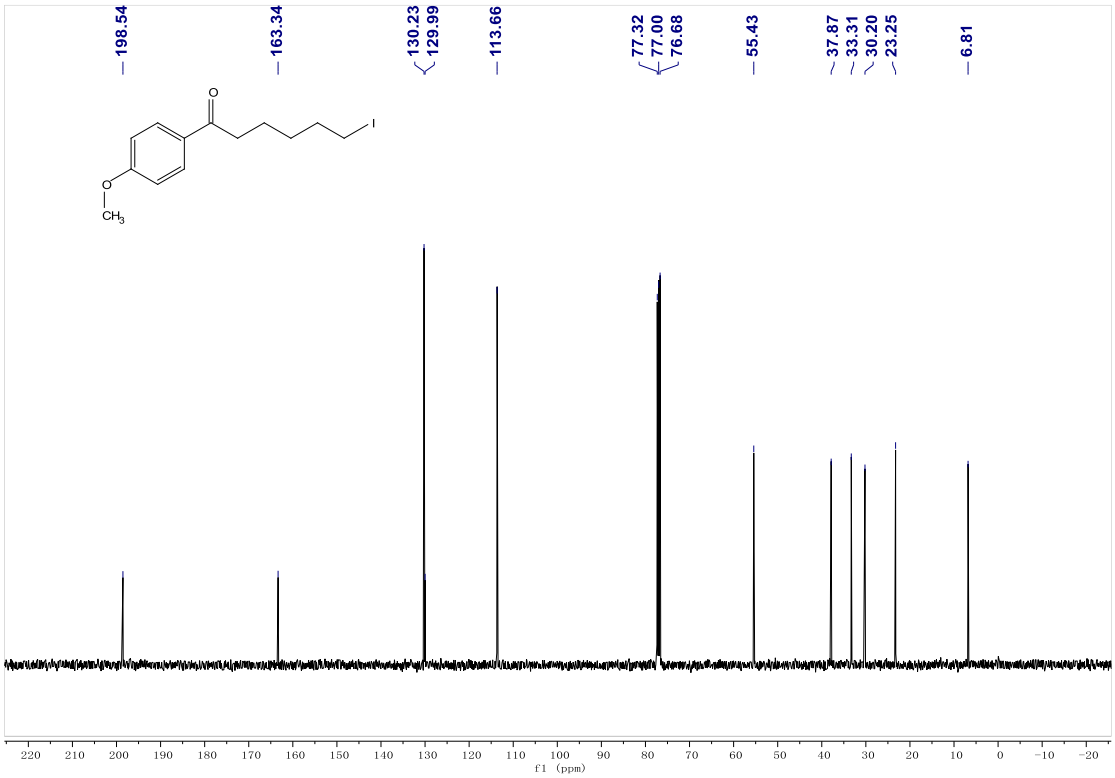
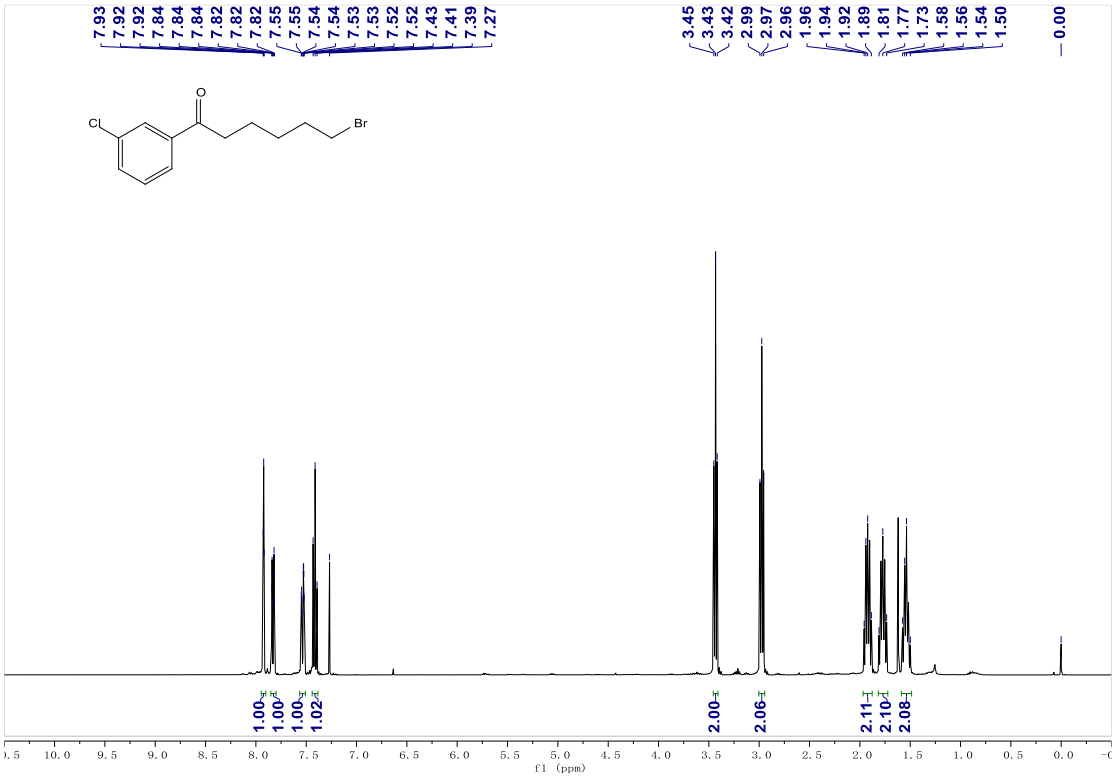
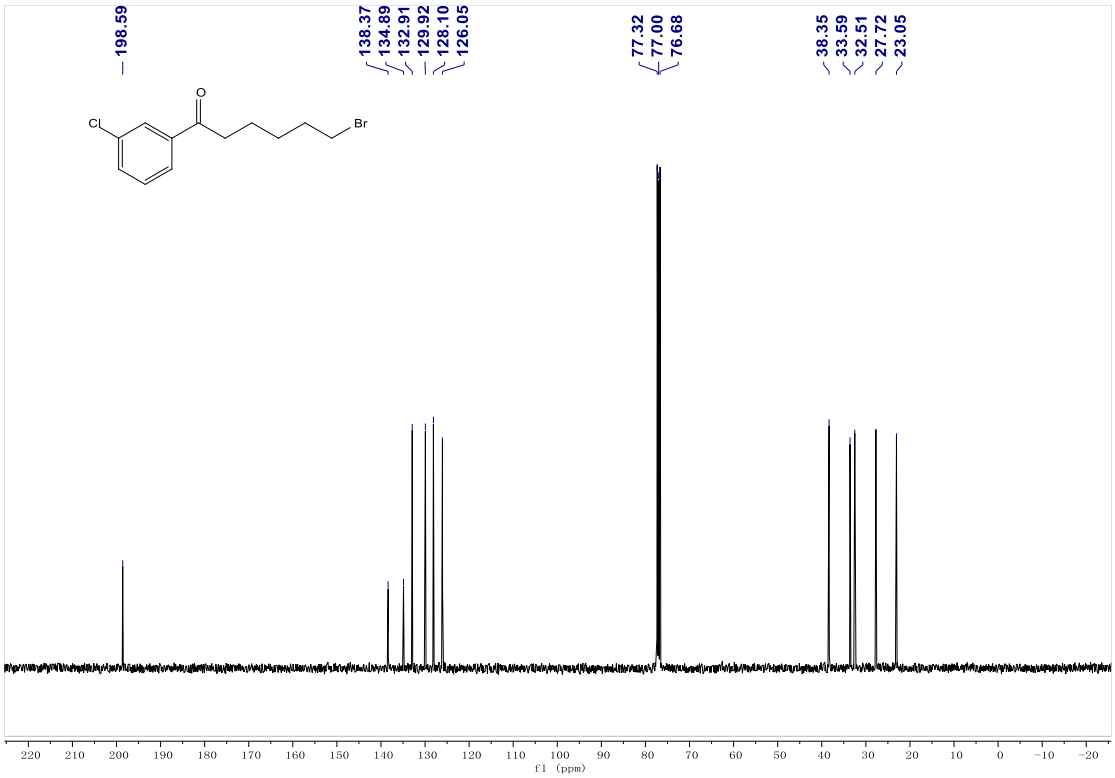


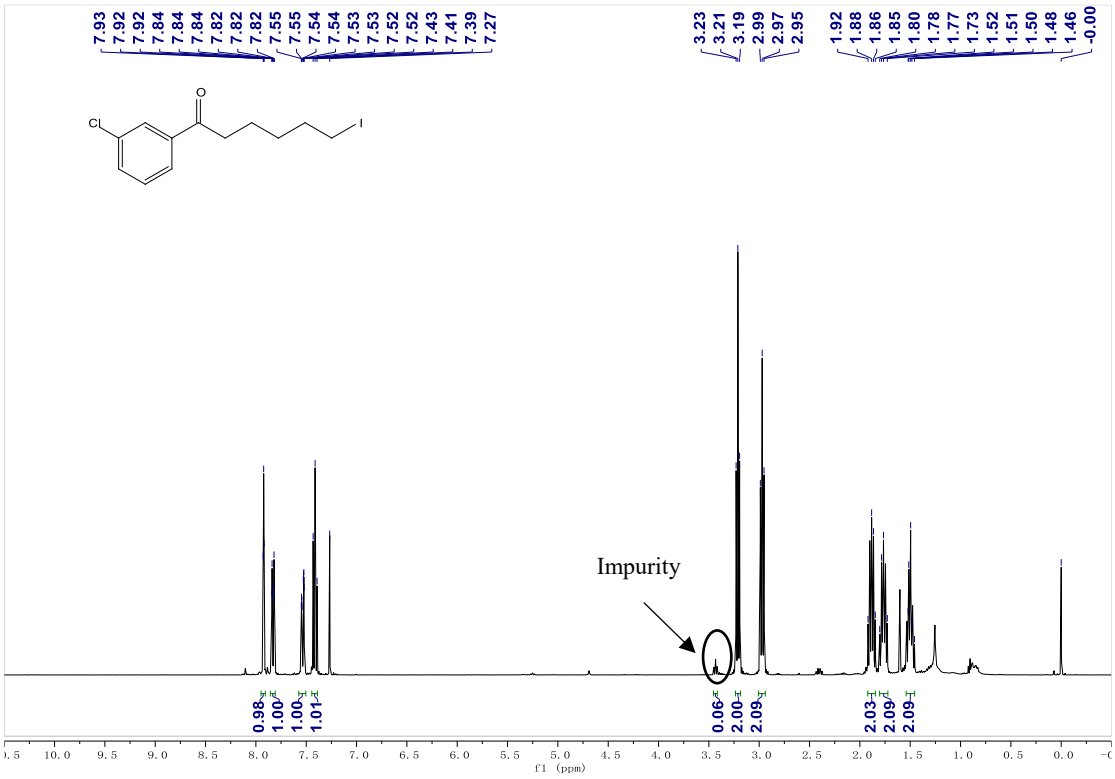
^{1}H NMR (400 MHz, $CDCl_3$) of **4g**



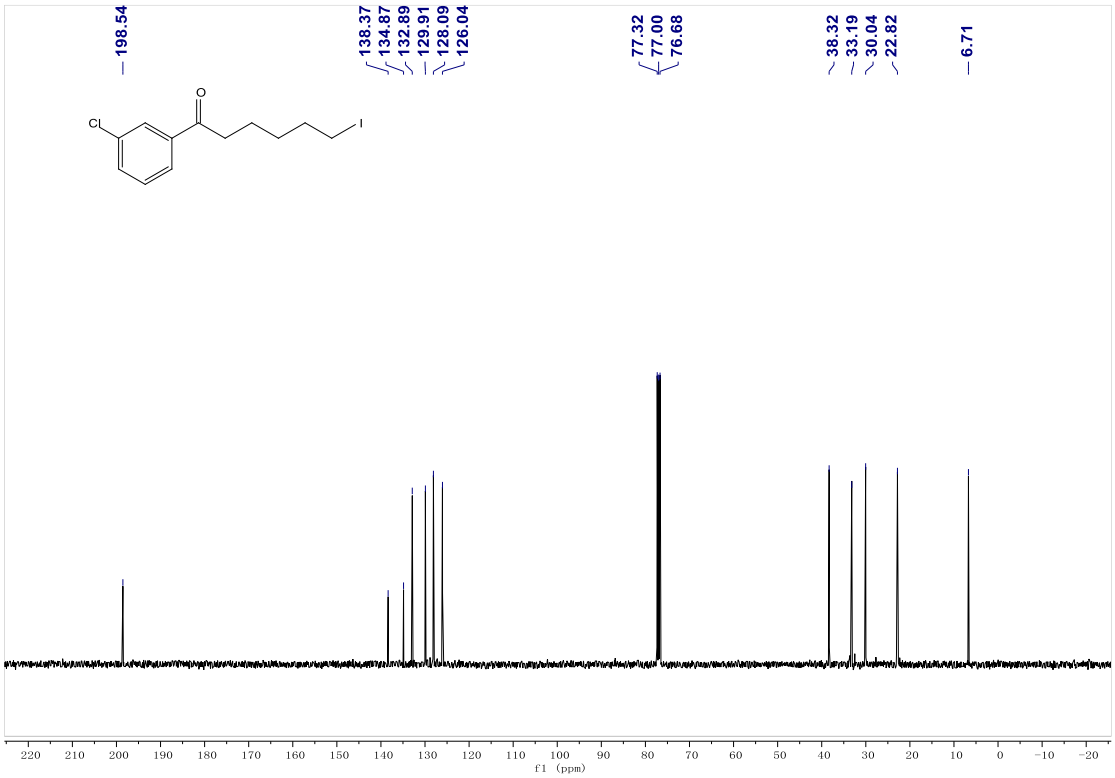
^{13}C NMR (101 MHz, $CDCl_3$) of **4g**



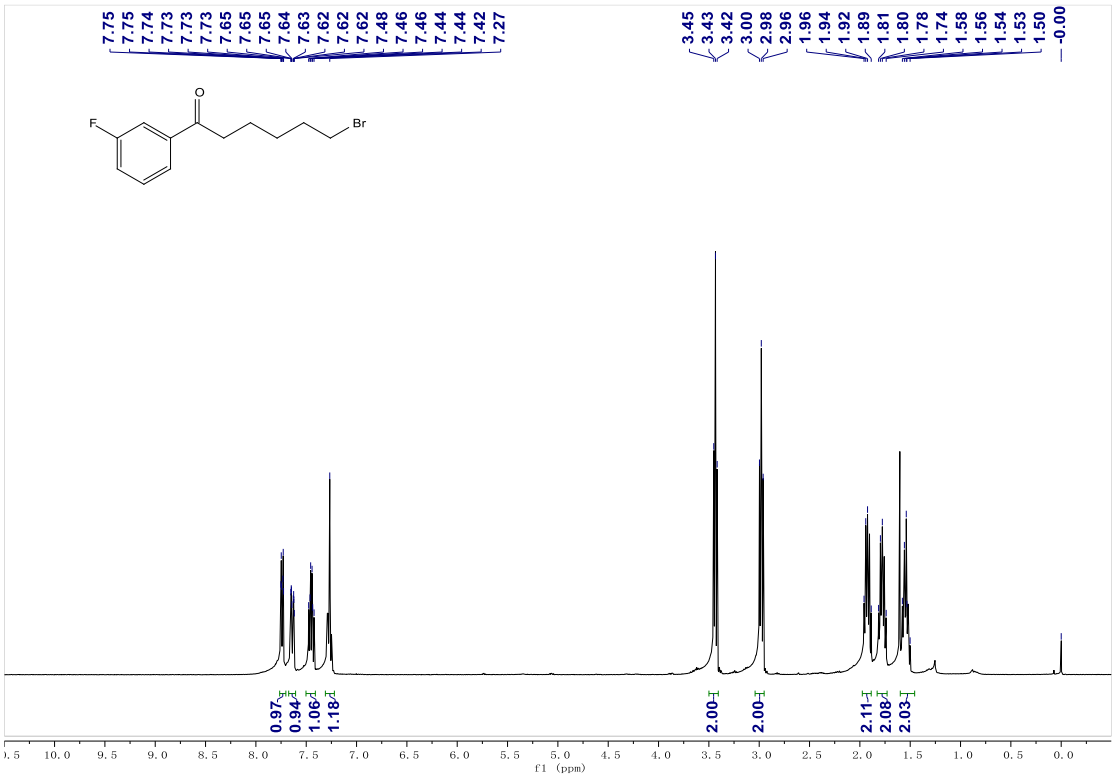
^{1}H NMR (400 MHz, $CDCl_3$) of **5g**



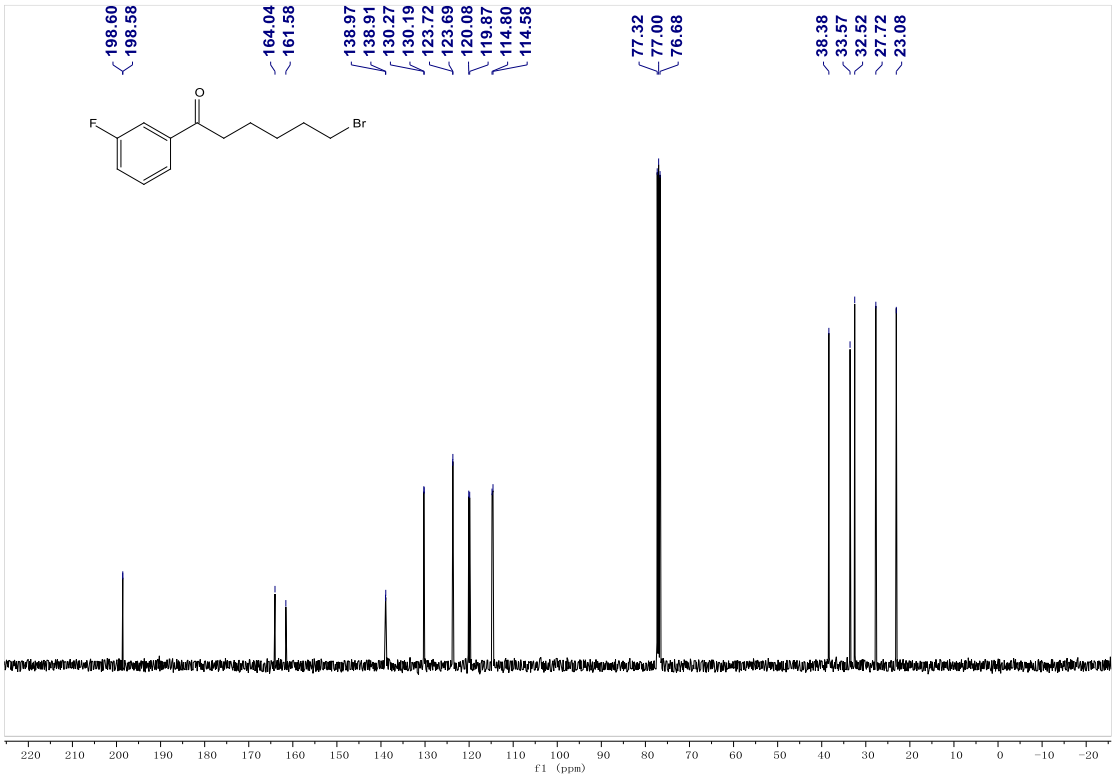
^{13}C NMR (101 MHz, $CDCl_3$) of **5g**



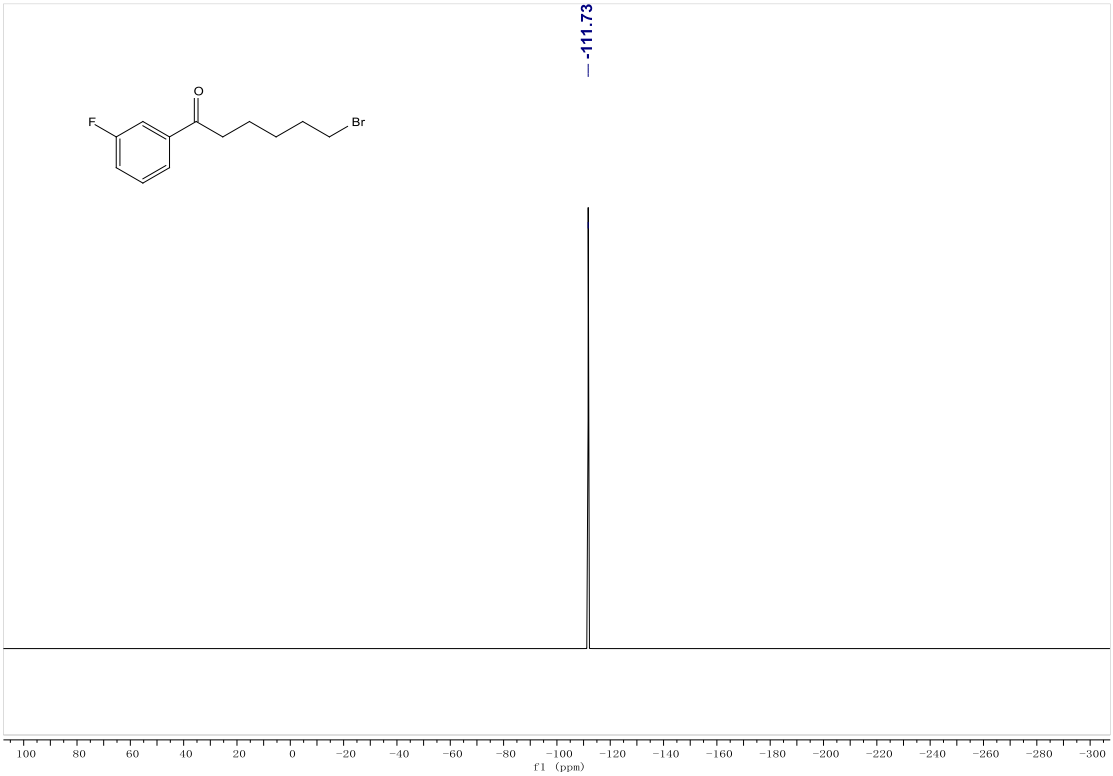
^{1}H NMR (400 MHz, $CDCl_3$) of **4h**



^{13}C NMR (101 MHz, $CDCl_3$) of **4h**



^{19}F NMR (376 MHz, $CDCl_3$) of **4h**



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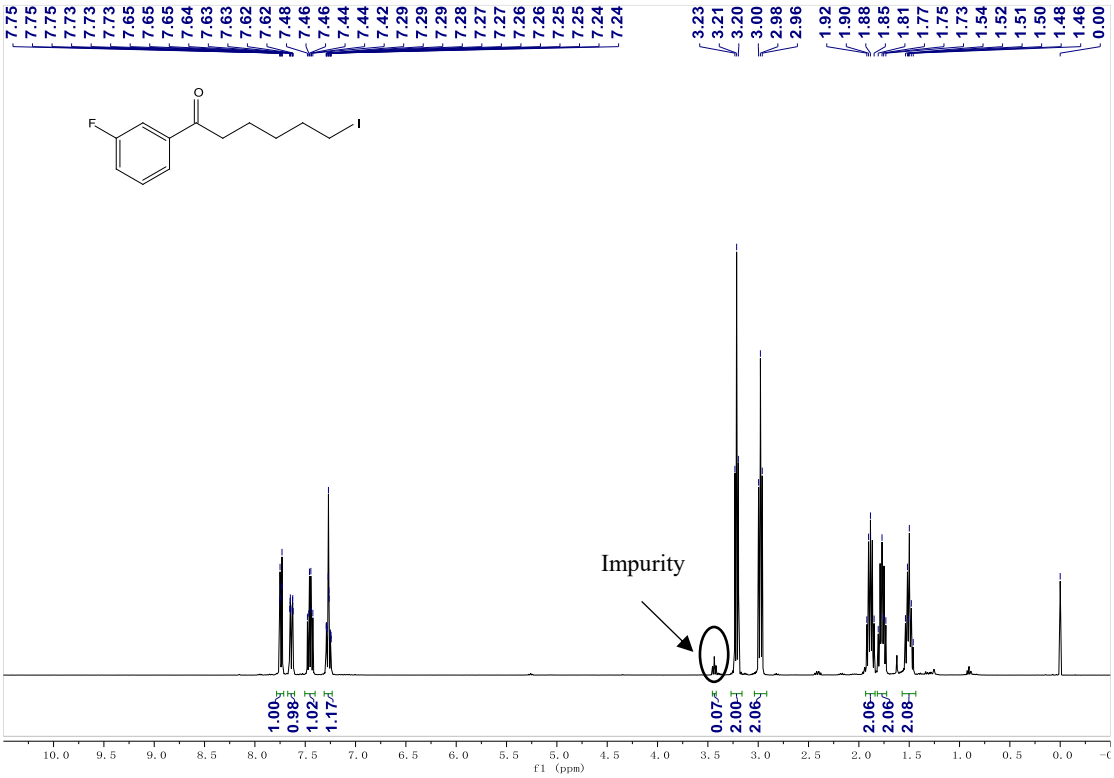
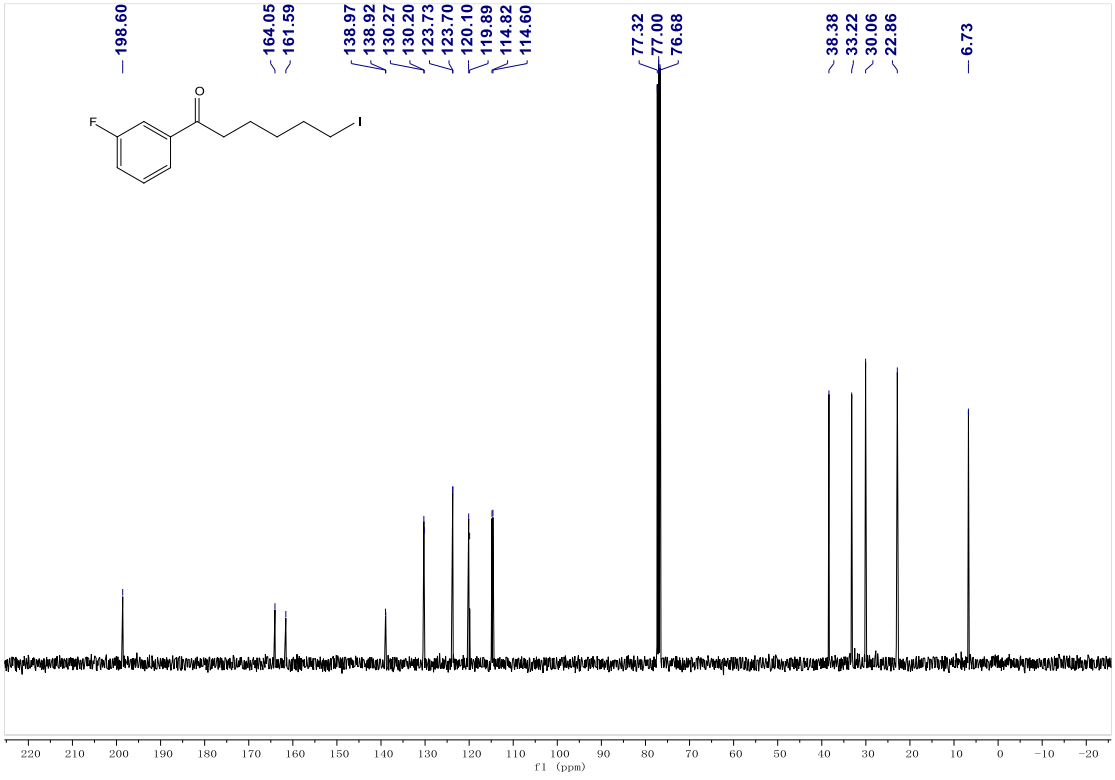
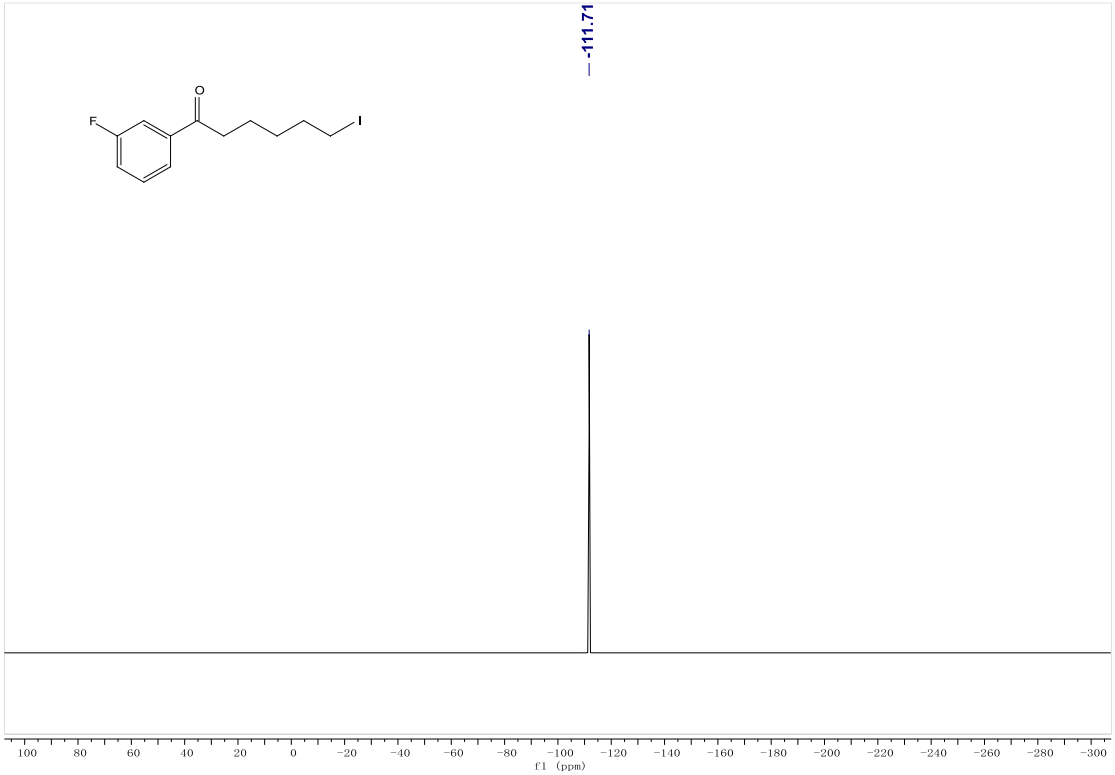


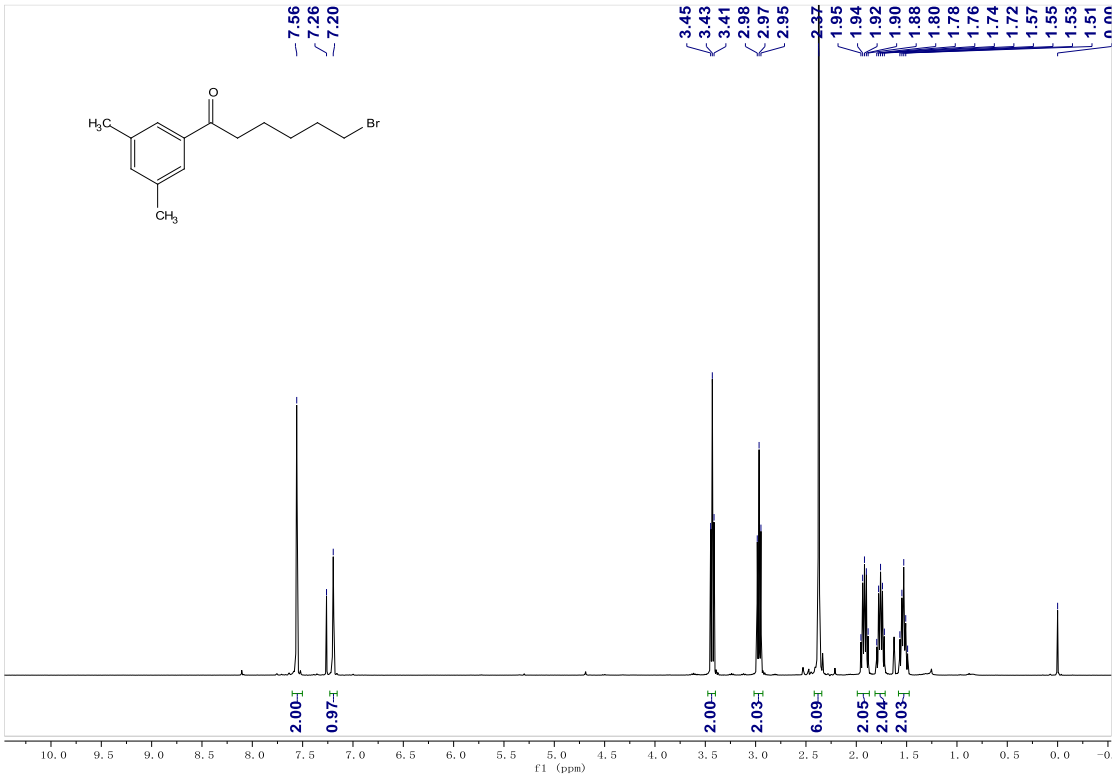
^{13}C NMR (101 MHz, $CDCl_3$) of **5h**



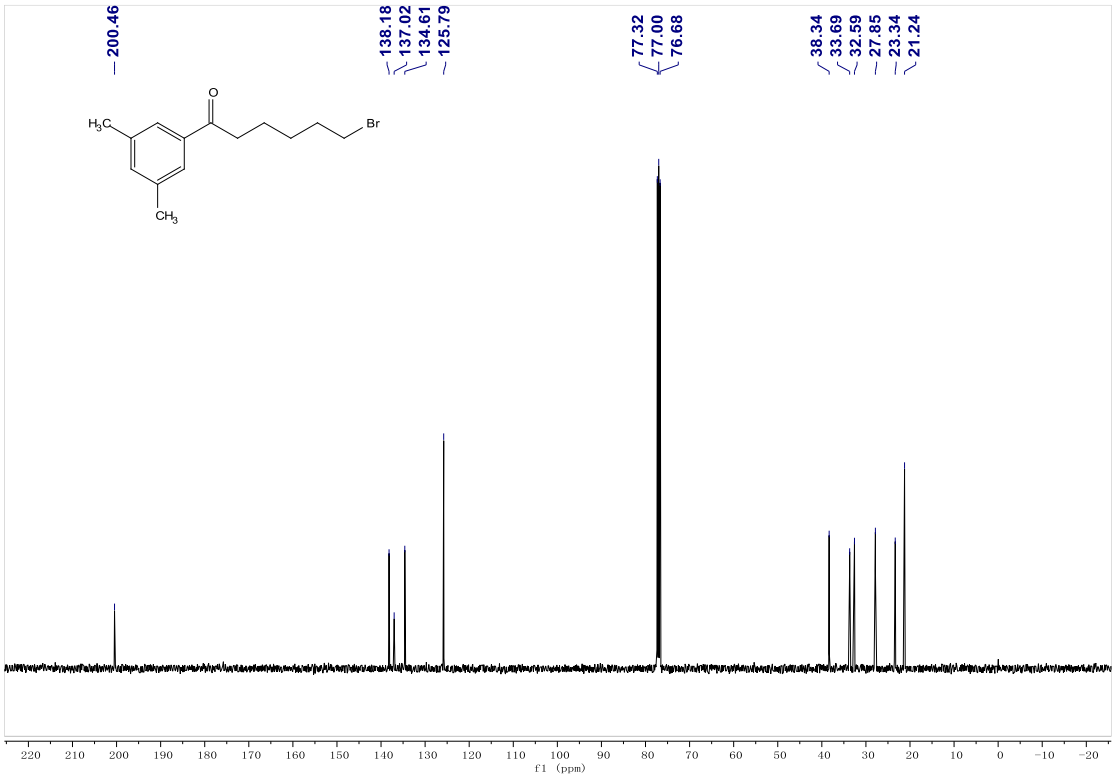
^{19}F NMR (376 MHz, $CDCl_3$) of **5h**



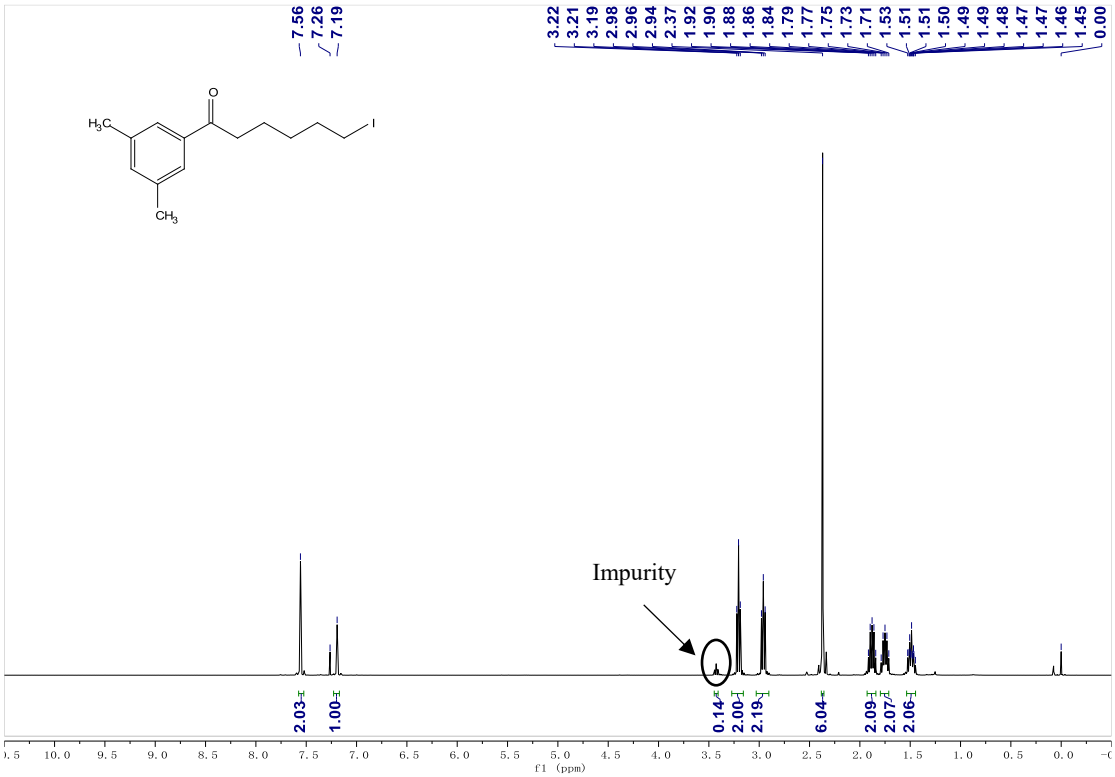
^{1}H NMR (400 MHz, $CDCl_3$) of **4i**



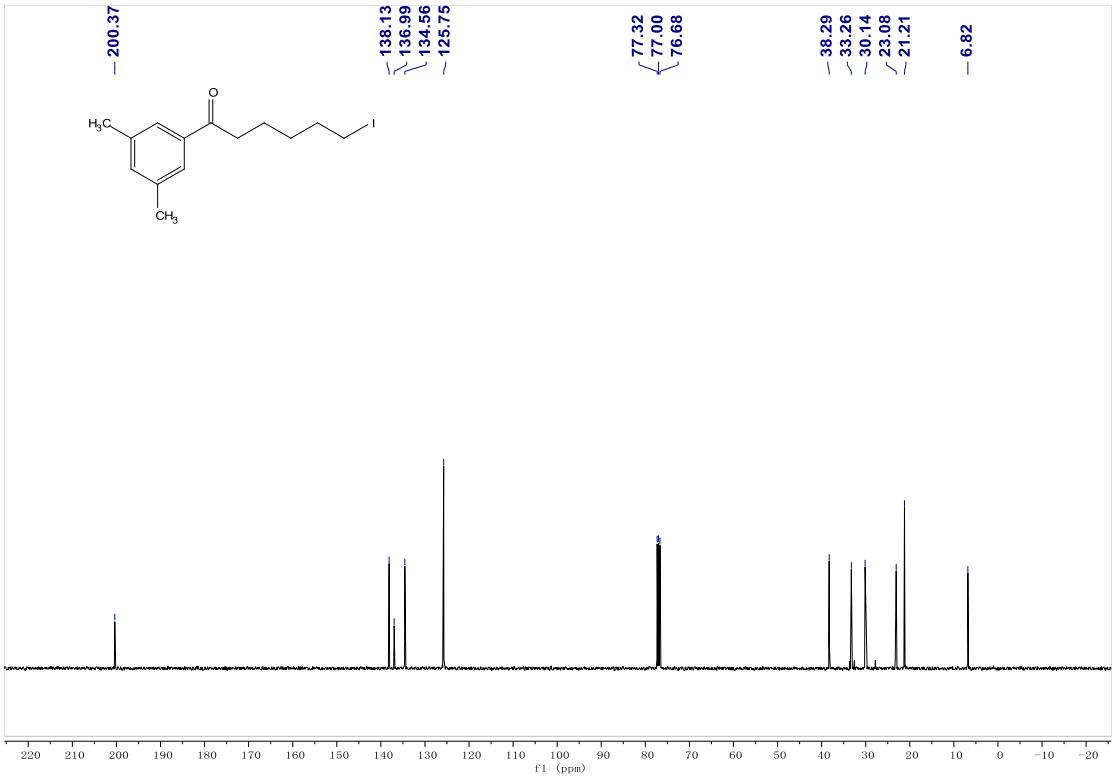
^{13}C NMR (101 MHz, $CDCl_3$) of **4i**



^{1}H NMR (400 MHz, $CDCl_3$) of **5i**



^{13}C NMR (101 MHz, $CDCl_3$) of **5i**



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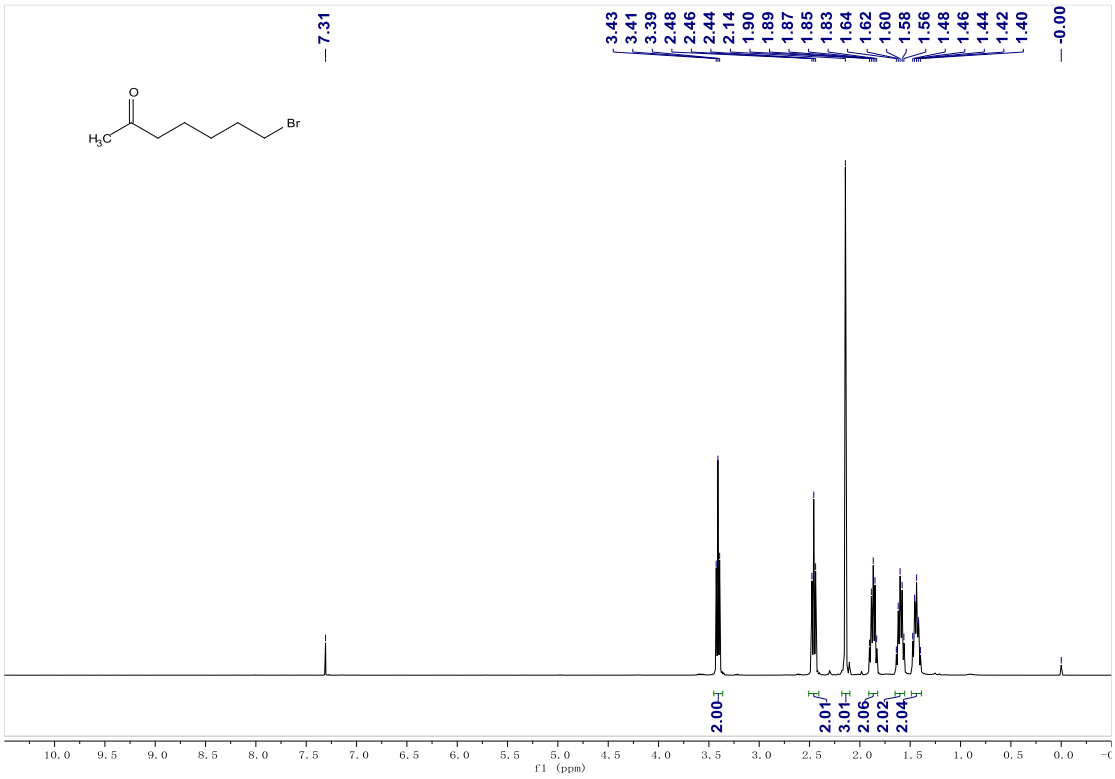

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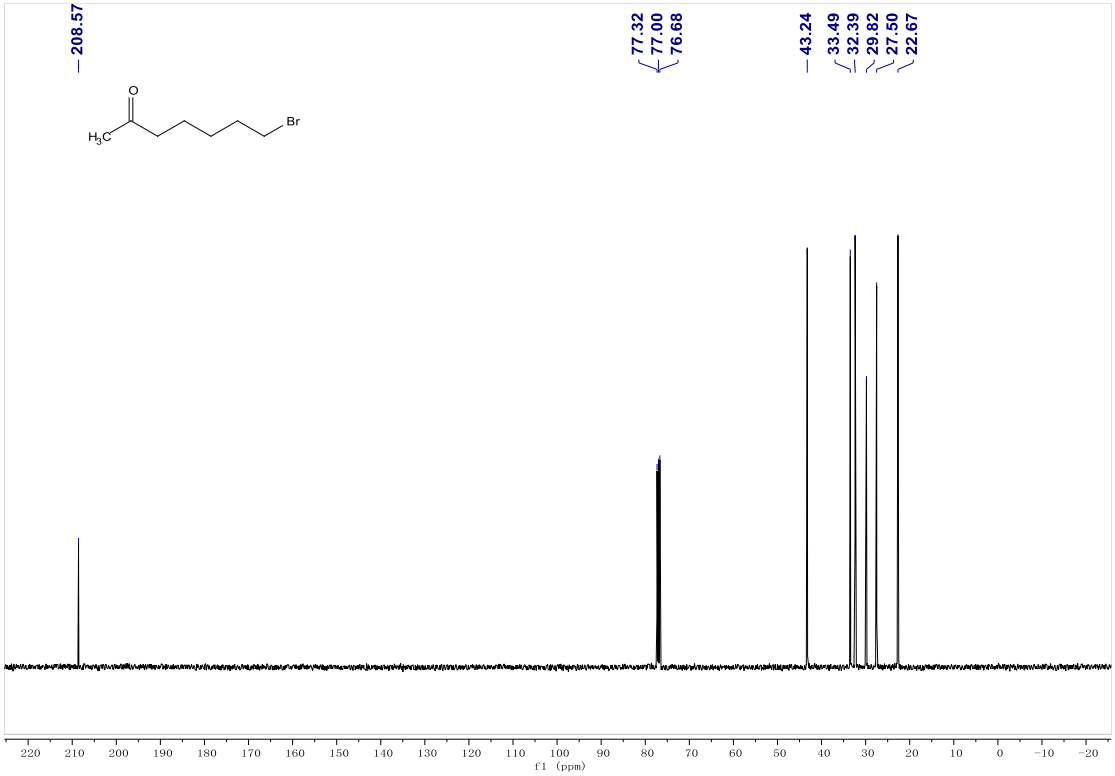

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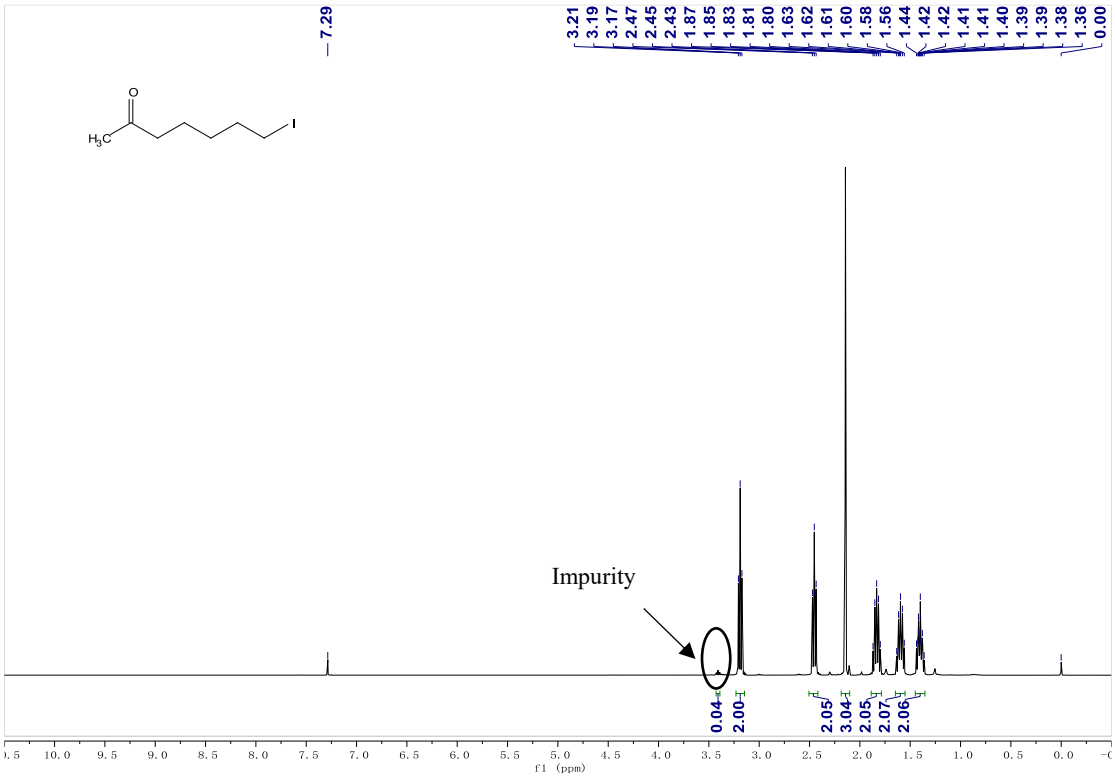

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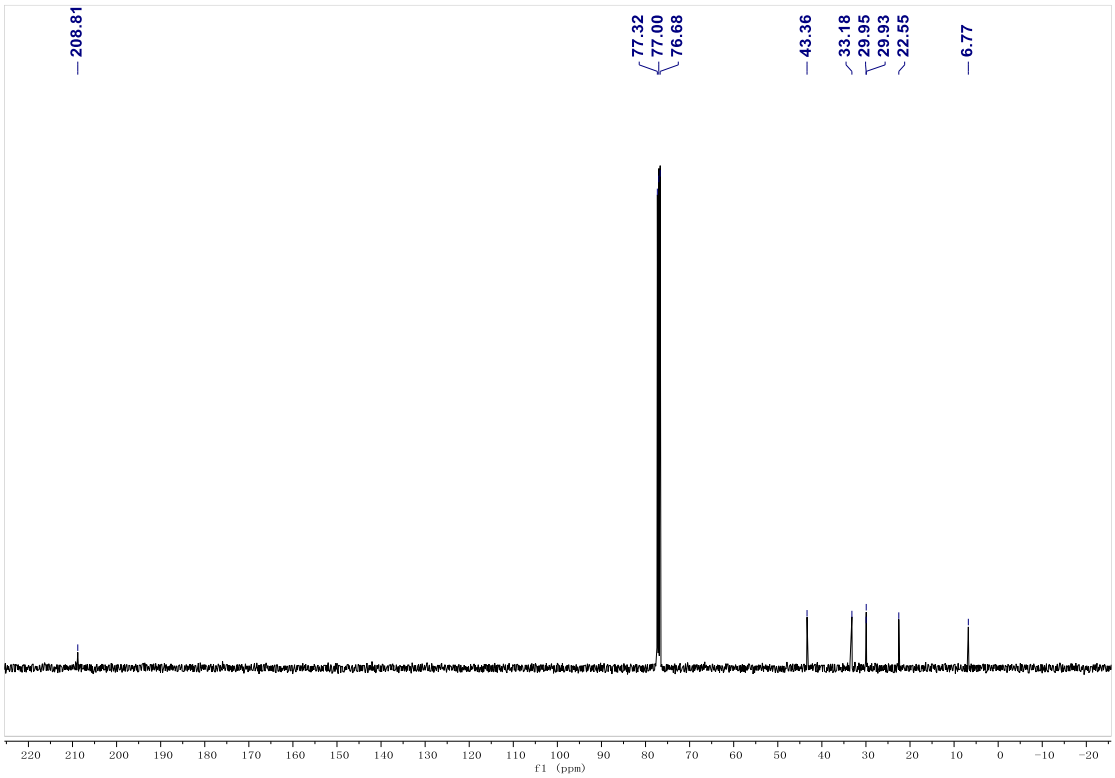

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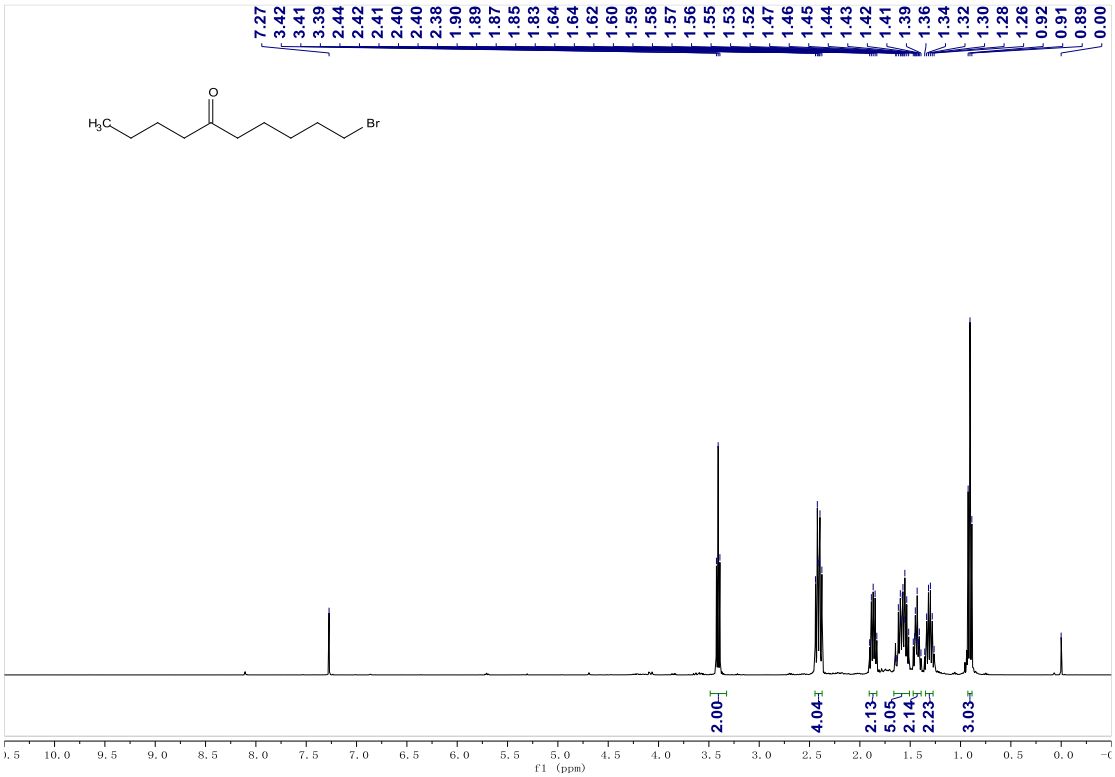
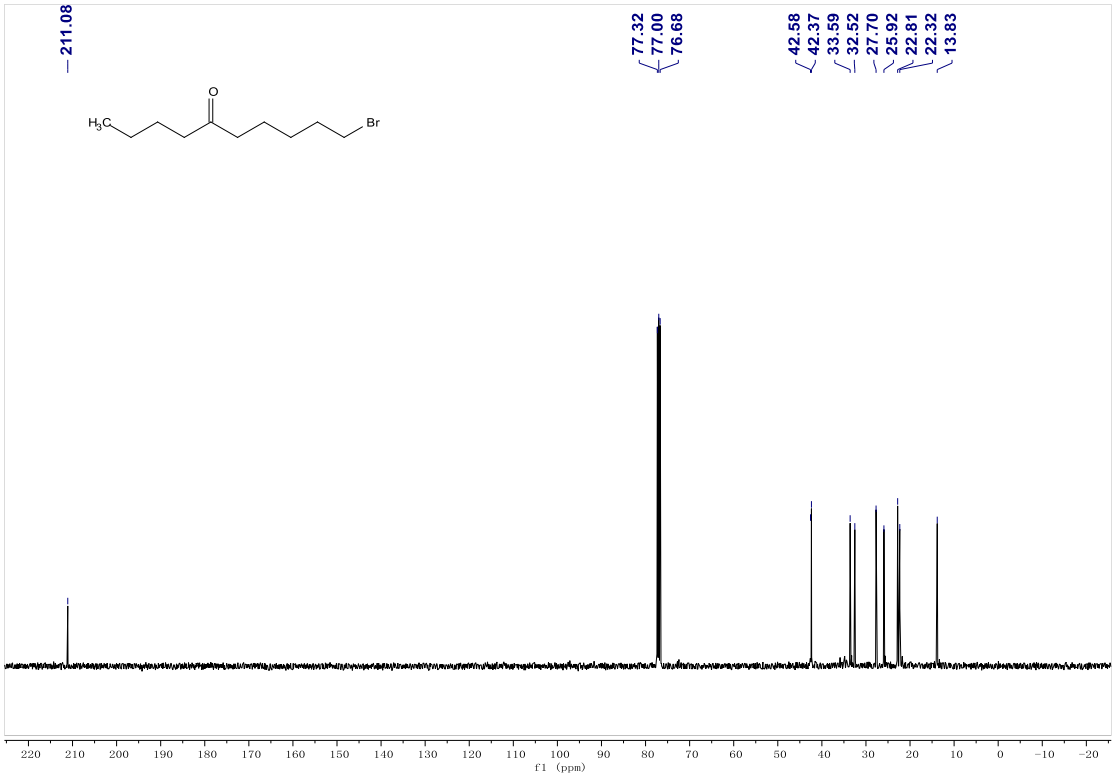


^{13}C NMR (101 MHz, $CDCl_3$) of **4k**



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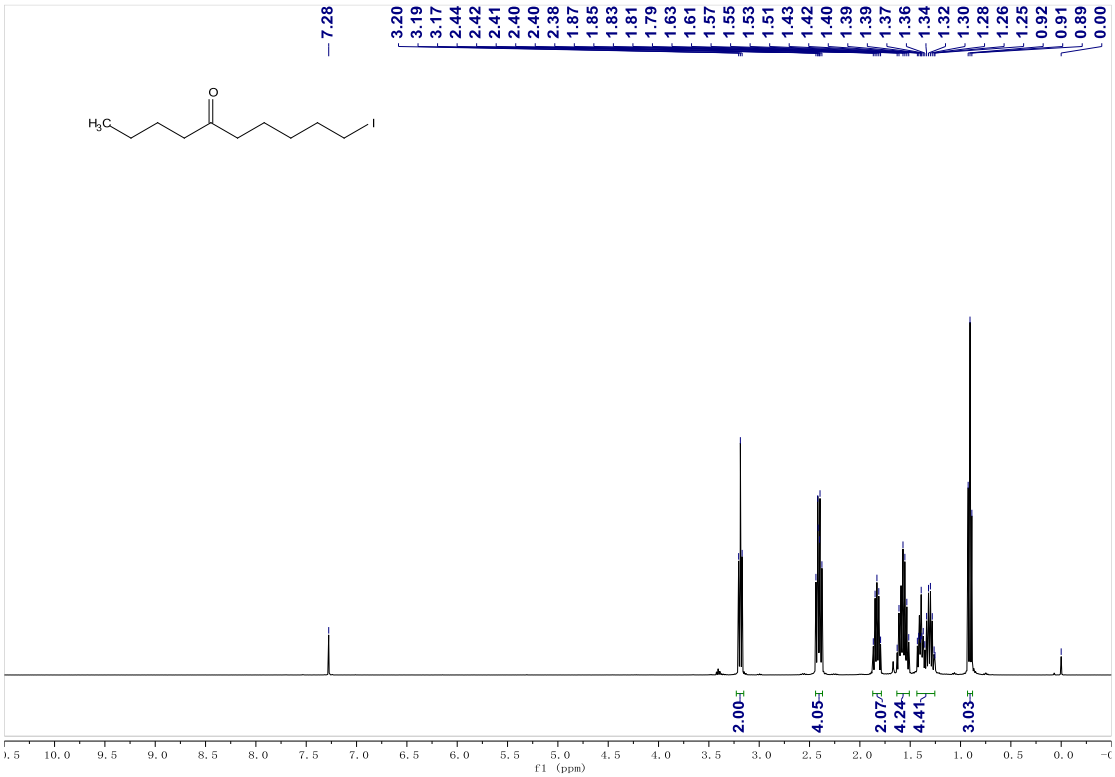

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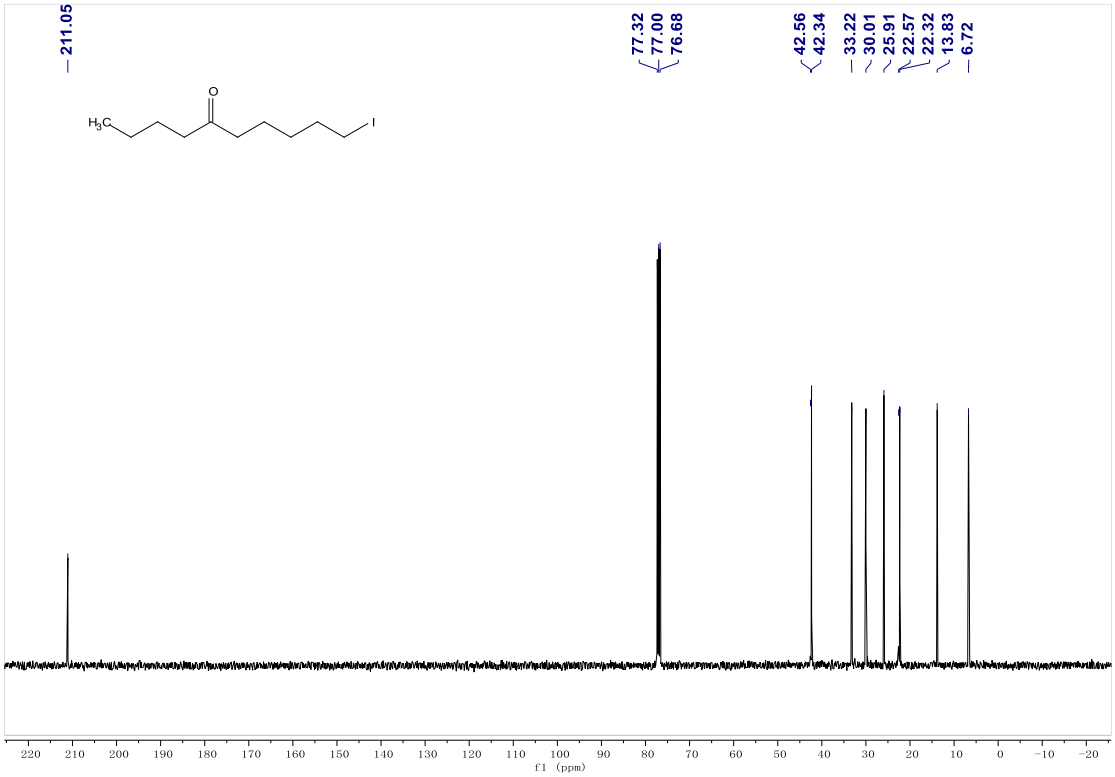
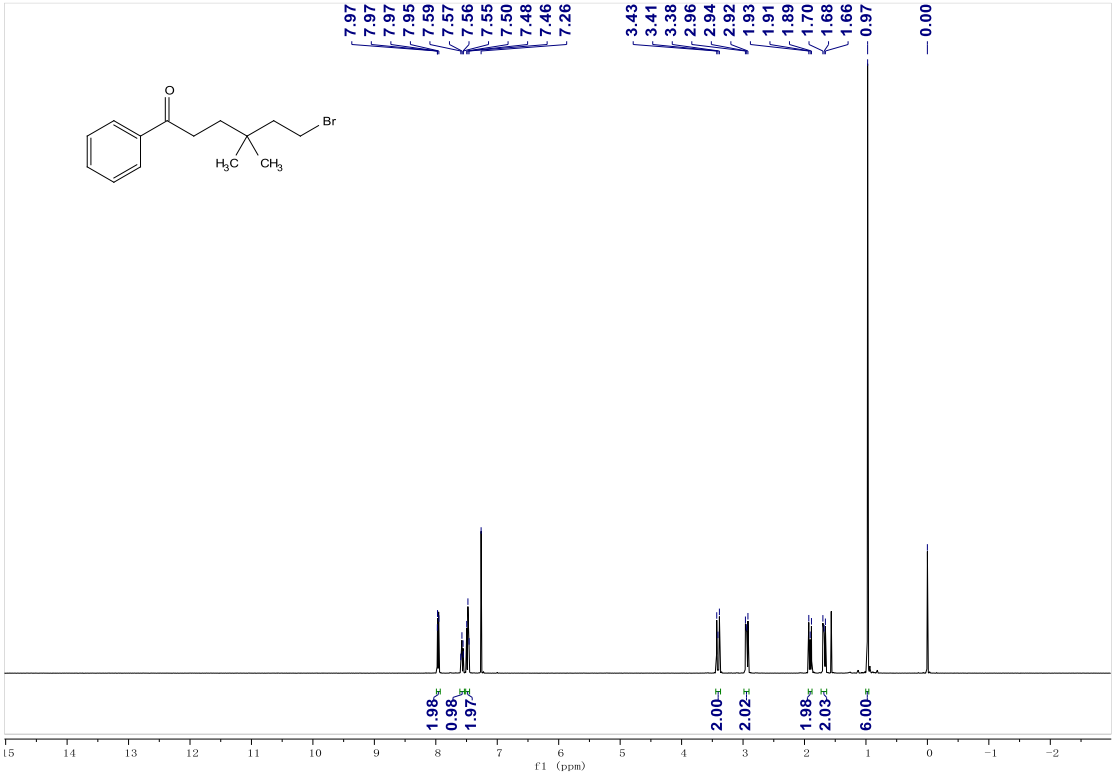
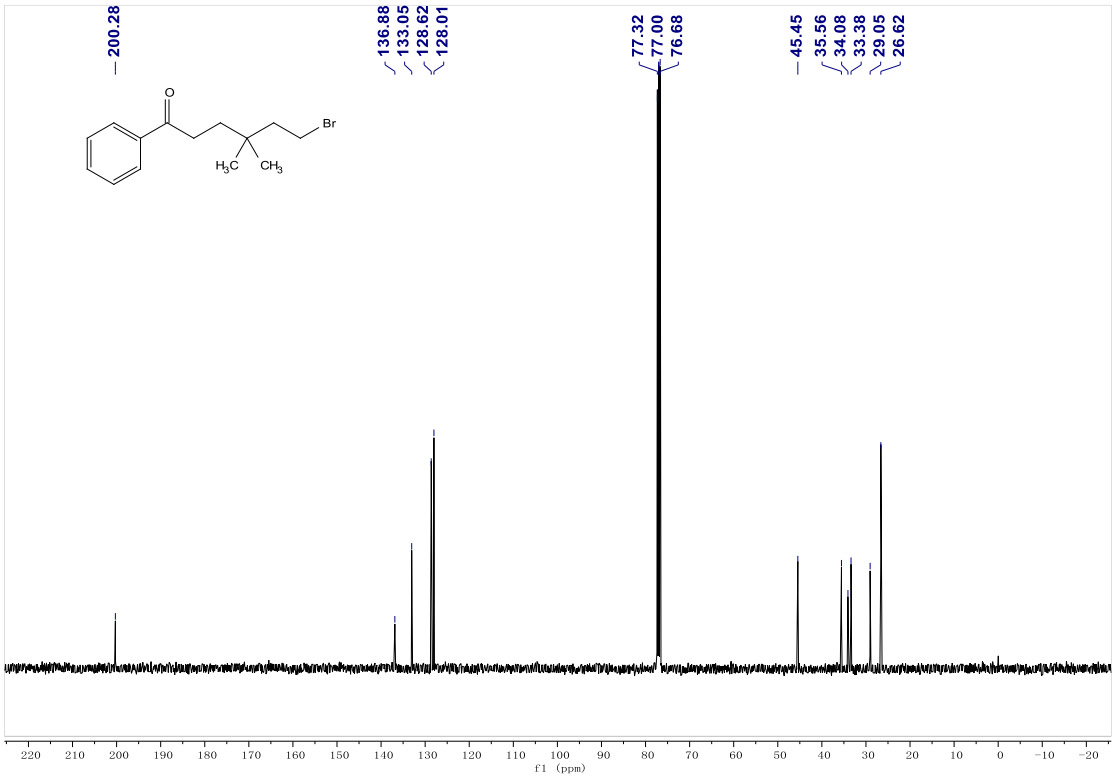


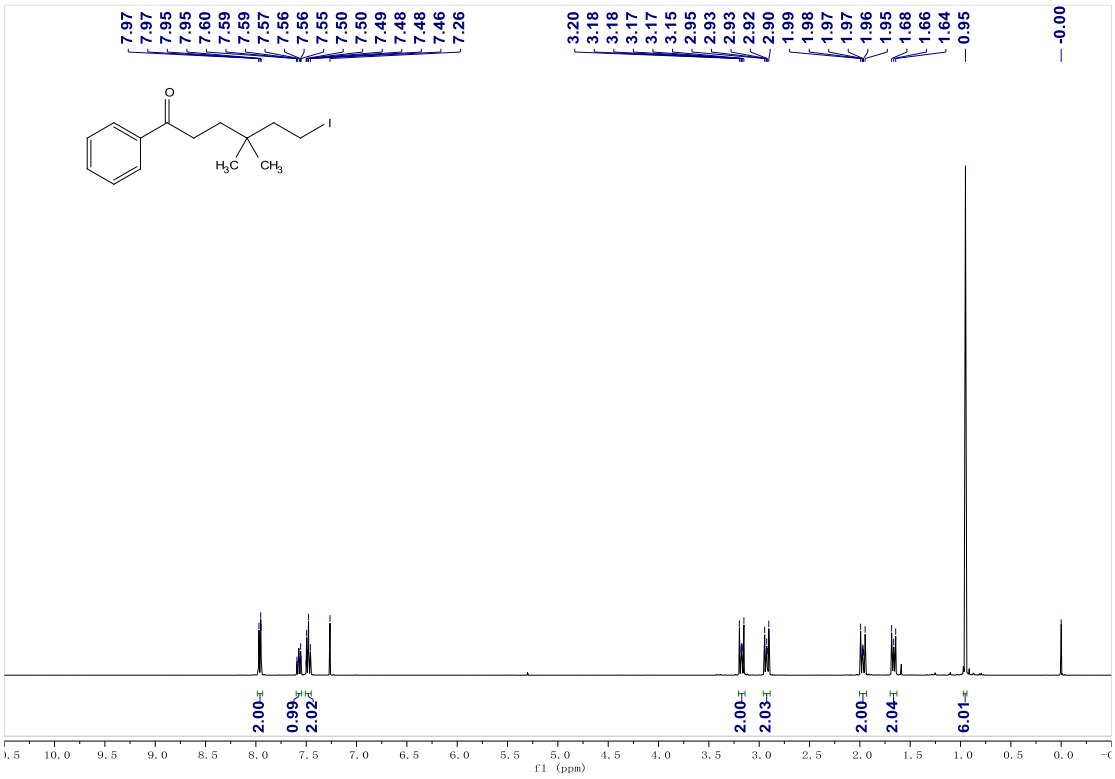
^{1}H NMR (400 MHz, $CDCl_3$) of **4l**



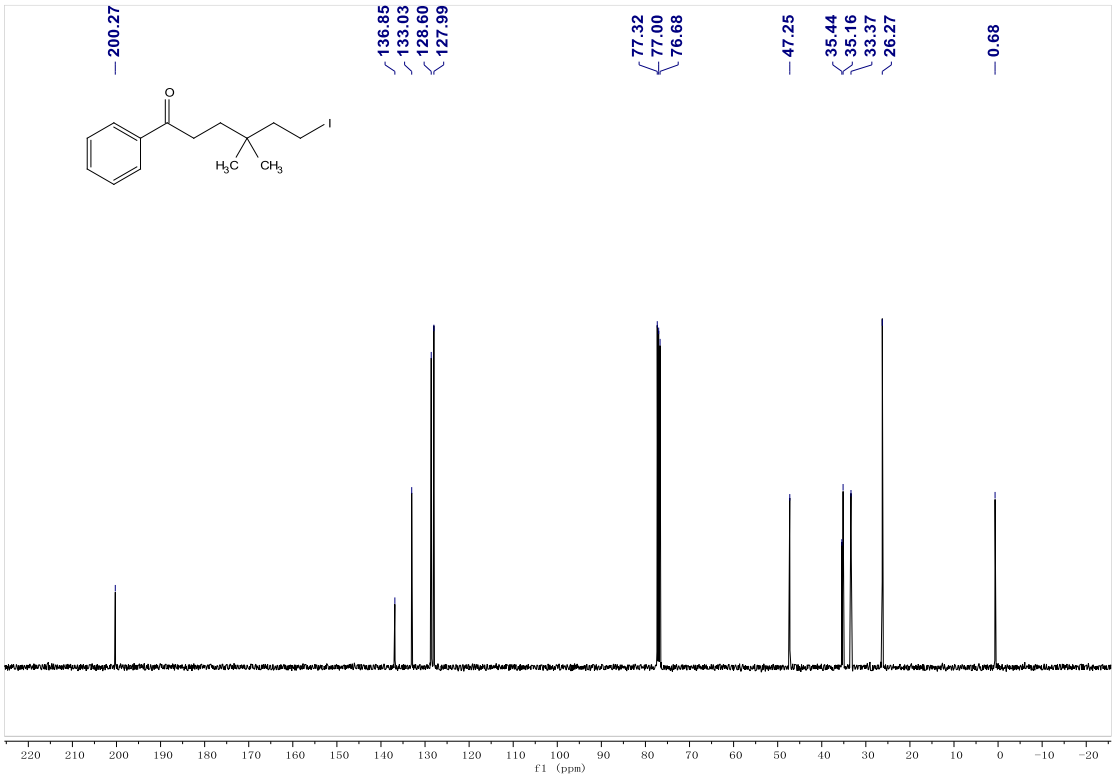
^{13}C NMR (101 MHz, $CDCl_3$) of **4l**



^{1}H NMR (400 MHz, $CDCl_3$) of **5l**



^{13}C NMR (101 MHz, $CDCl_3$) of **5l**



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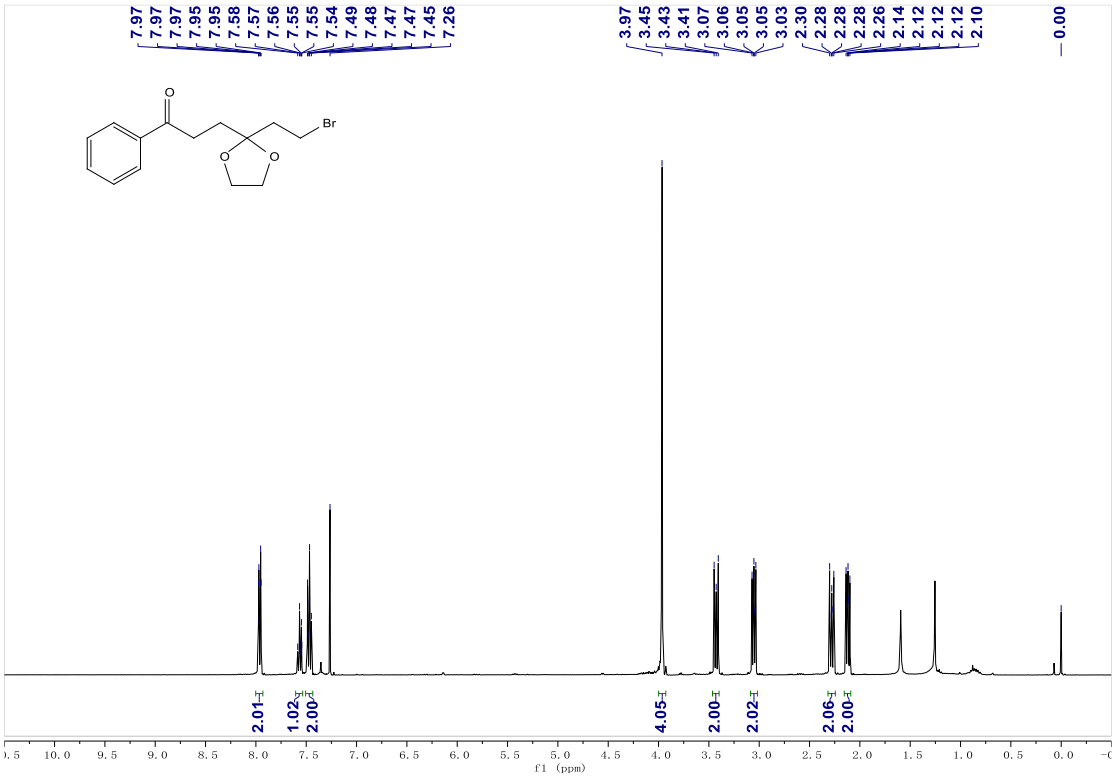

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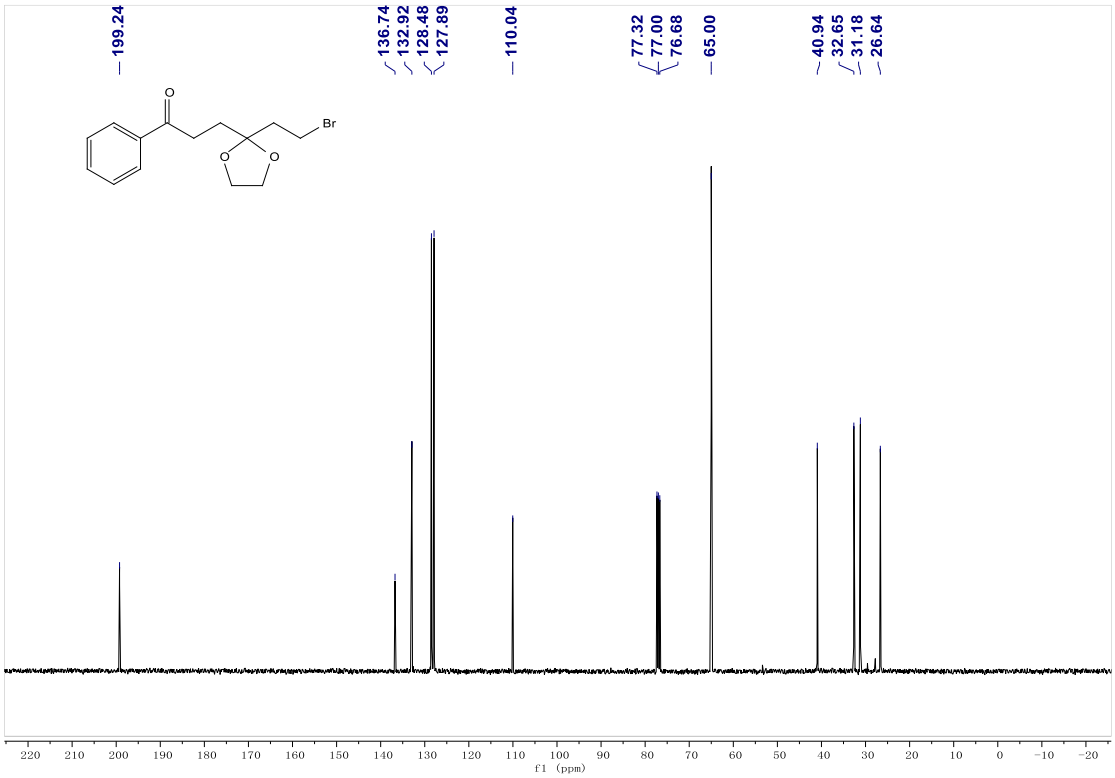
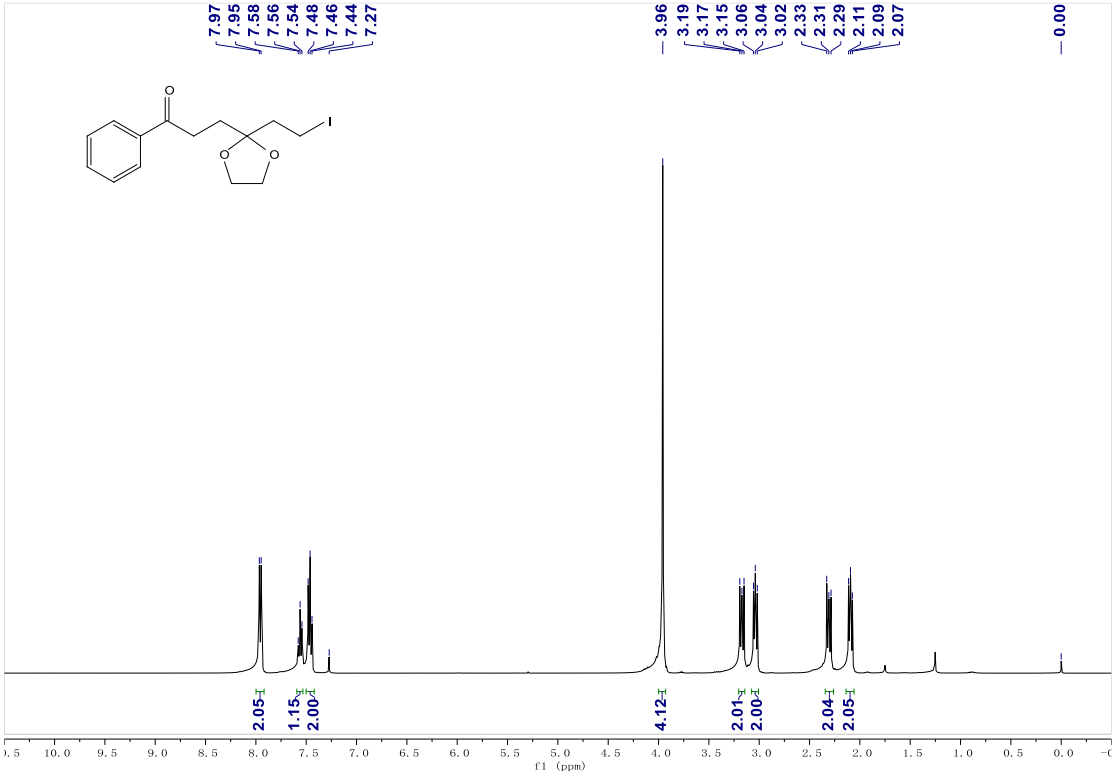
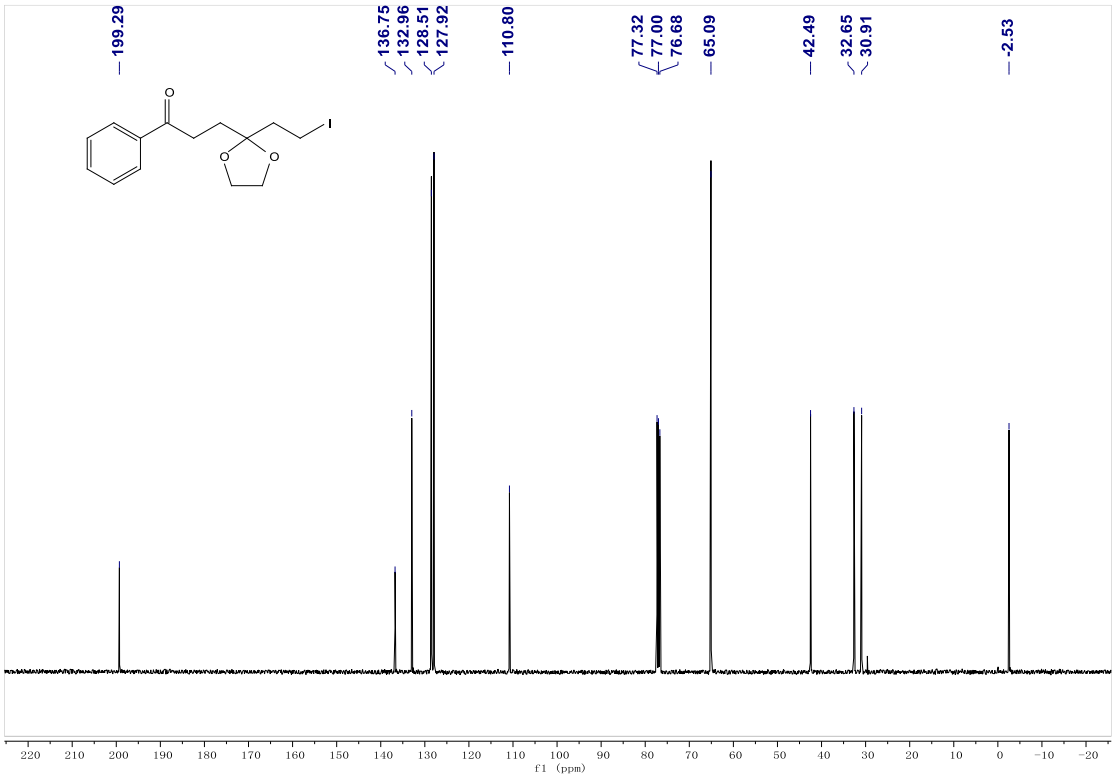


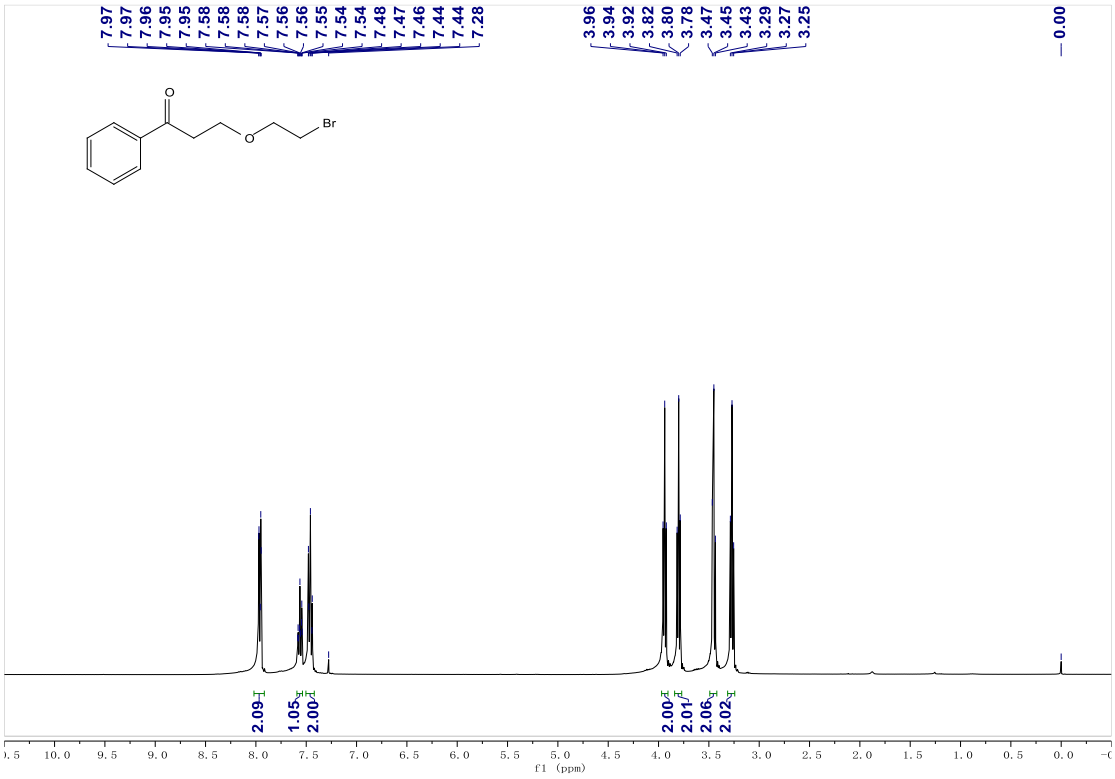
^{1}H NMR (400 MHz, $CDCl_3$) of **5m**



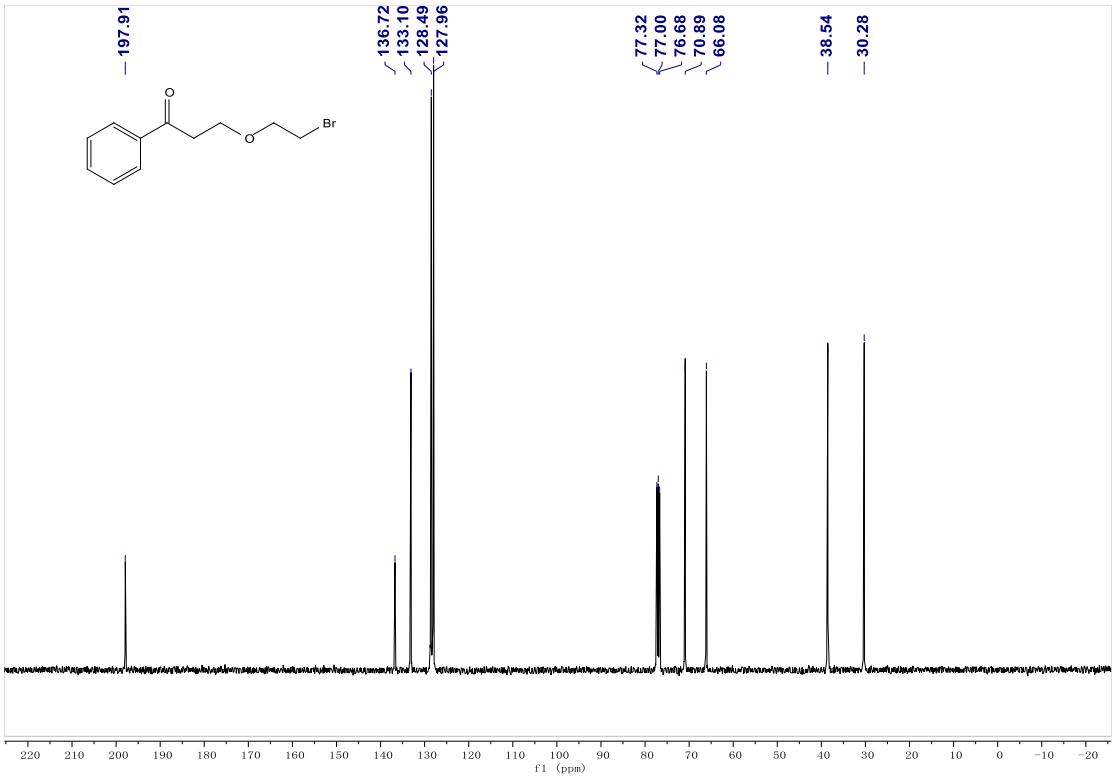
^{13}C NMR (101 MHz, $CDCl_3$) of **5m**



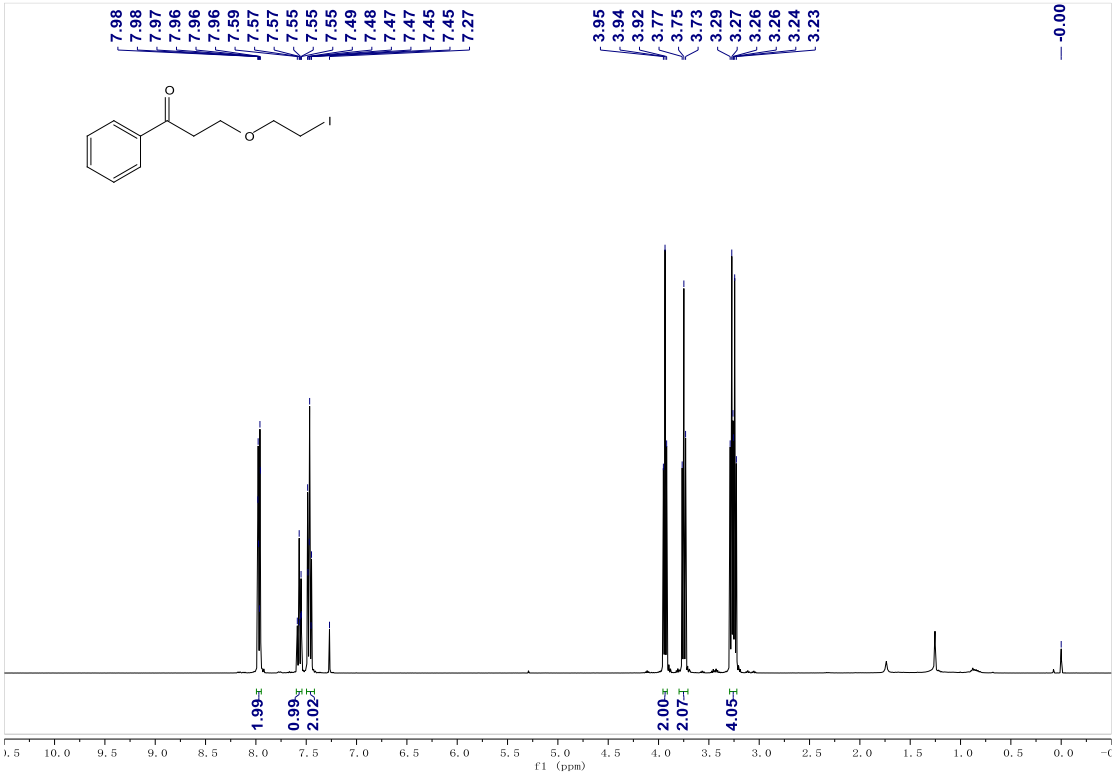
^{1}H NMR (400 MHz, $CDCl_3$) of **4n**



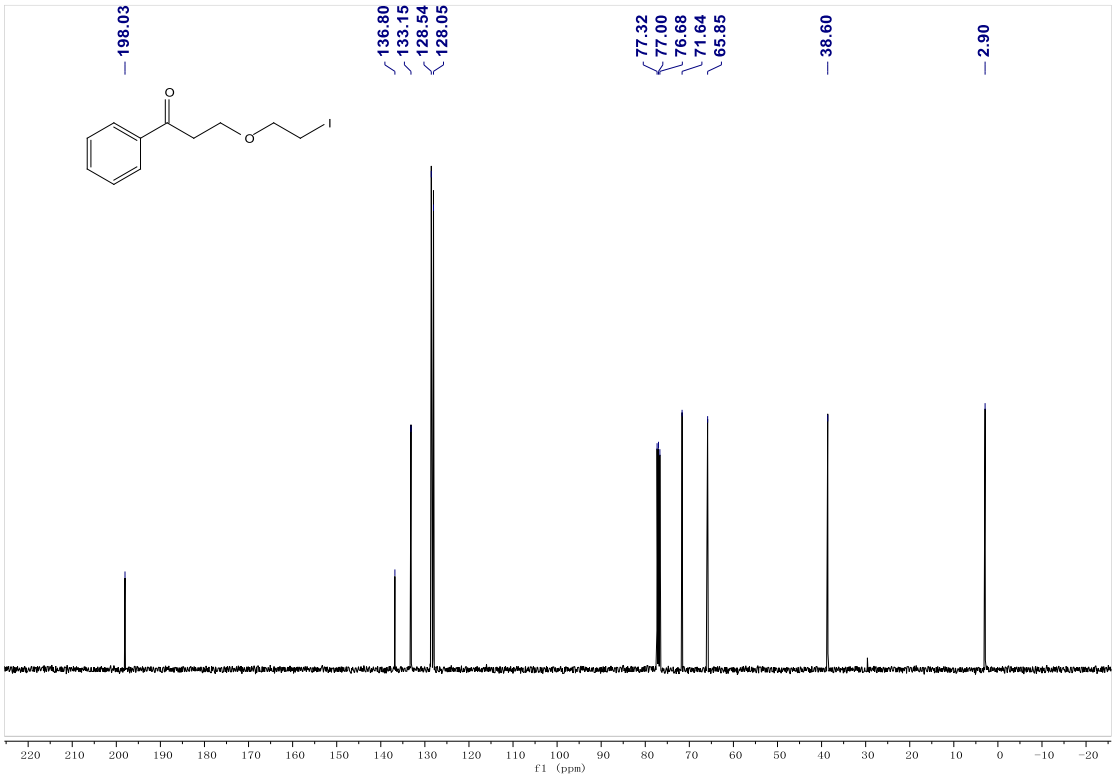
^{13}C NMR (101 MHz, $CDCl_3$) of **4n**



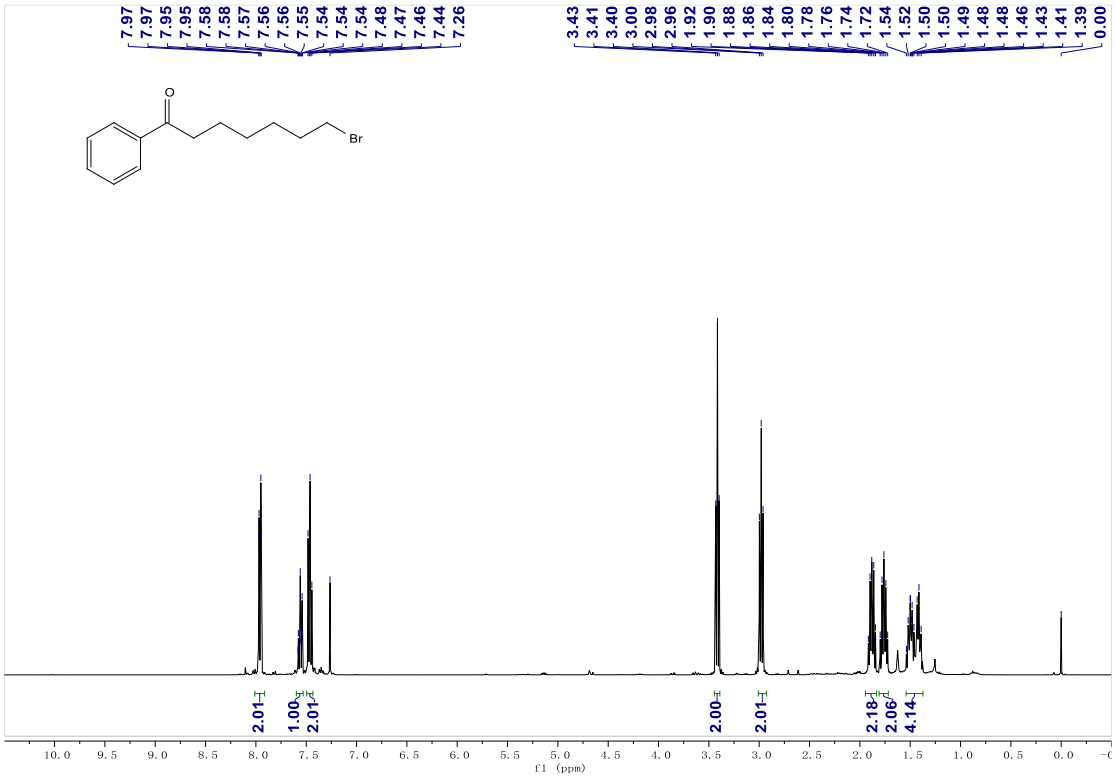
^{1}H NMR (400 MHz, $CDCl_3$) of **5n**



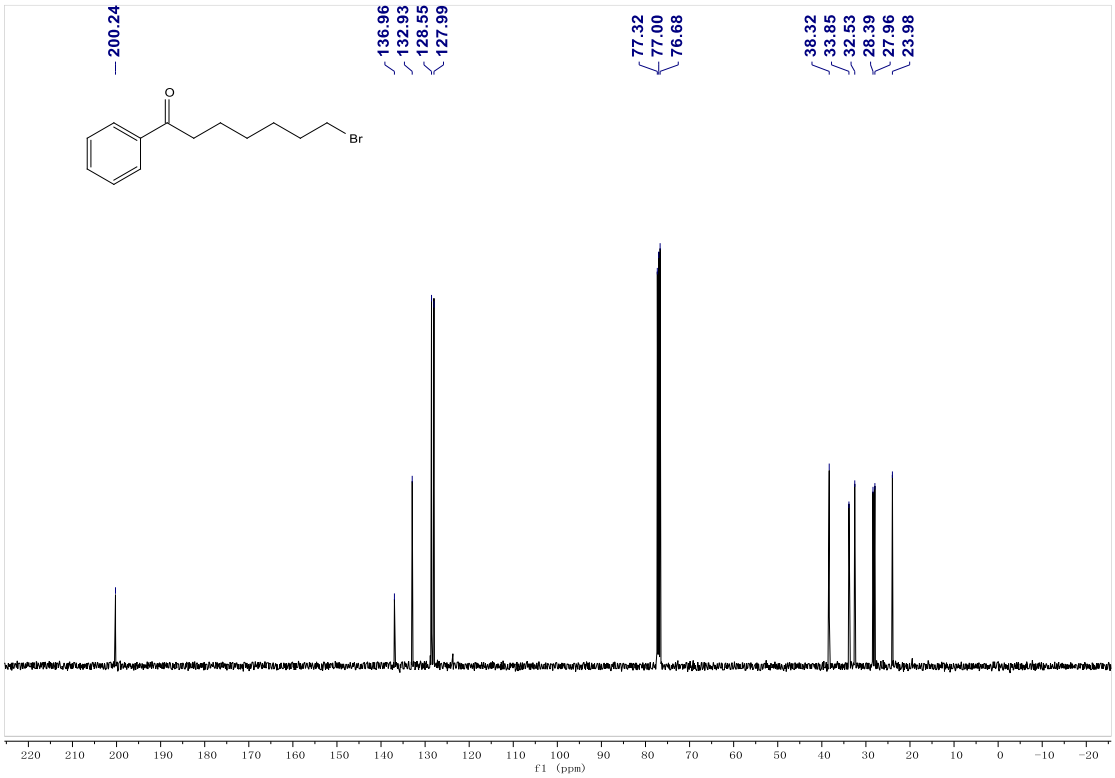
^{13}C NMR (101 MHz, $CDCl_3$) of **5n**



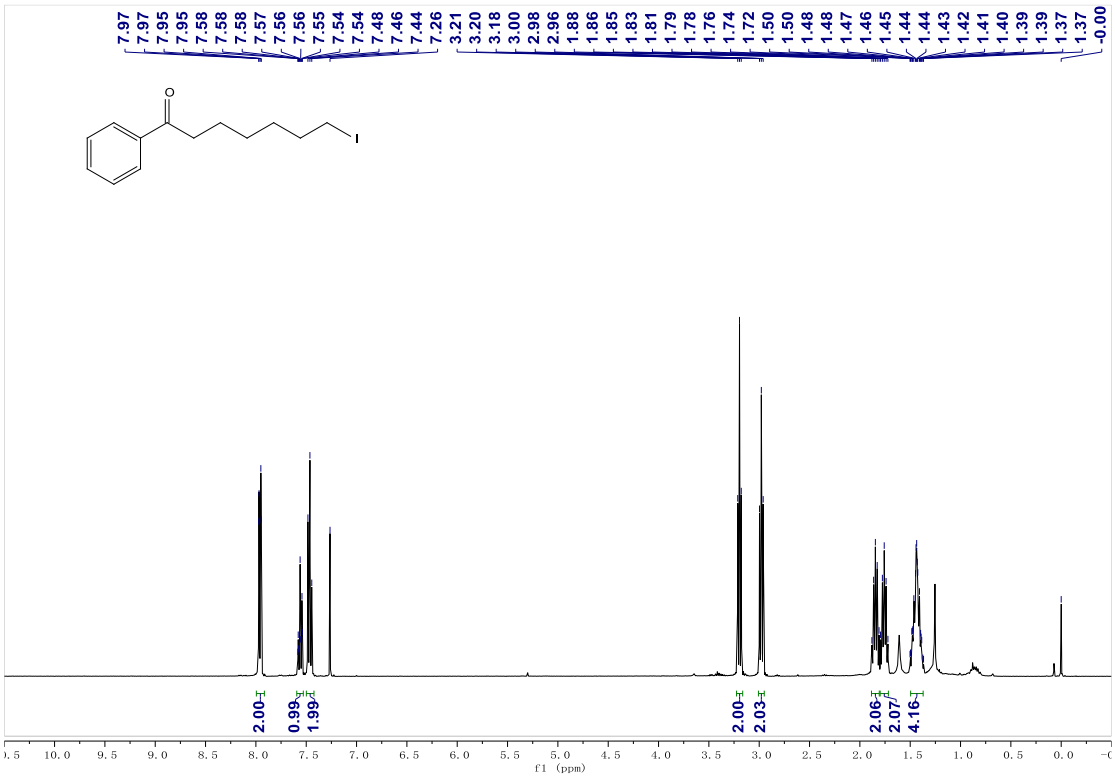
^{1}H NMR (400 MHz, $CDCl_3$) of **4o**



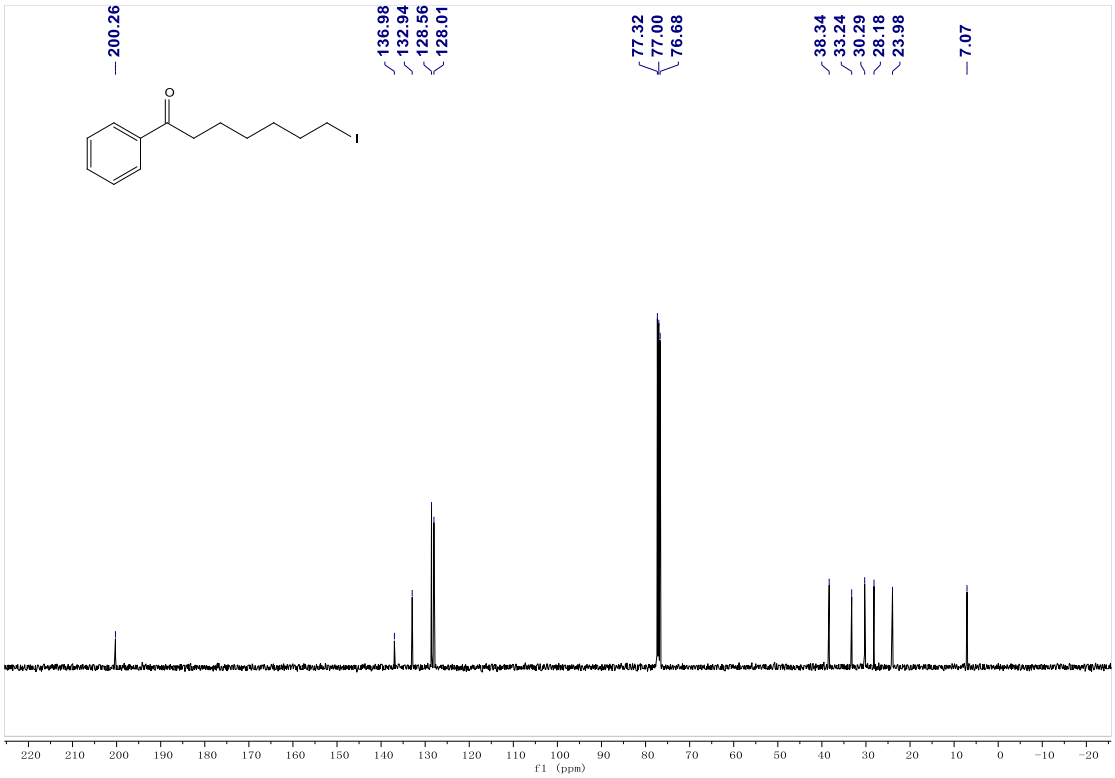
^{13}C NMR (101 MHz, $CDCl_3$) of **4o**



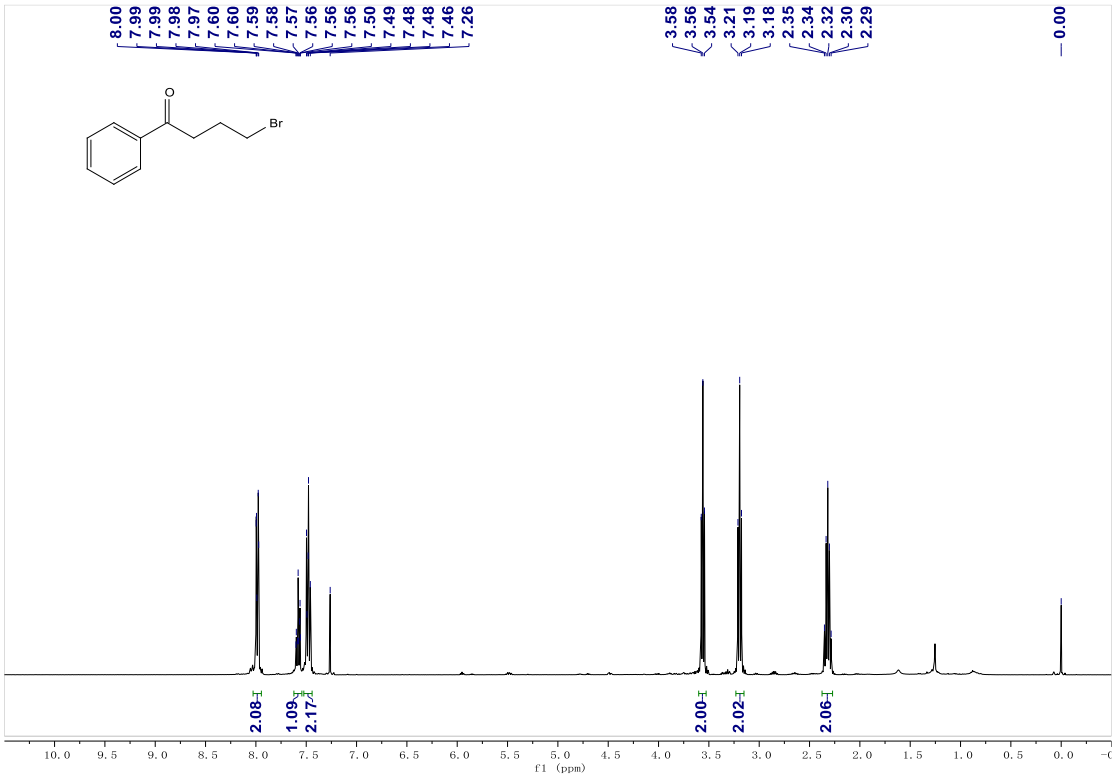
^{1}H NMR (400 MHz, $CDCl_3$) of **5o**



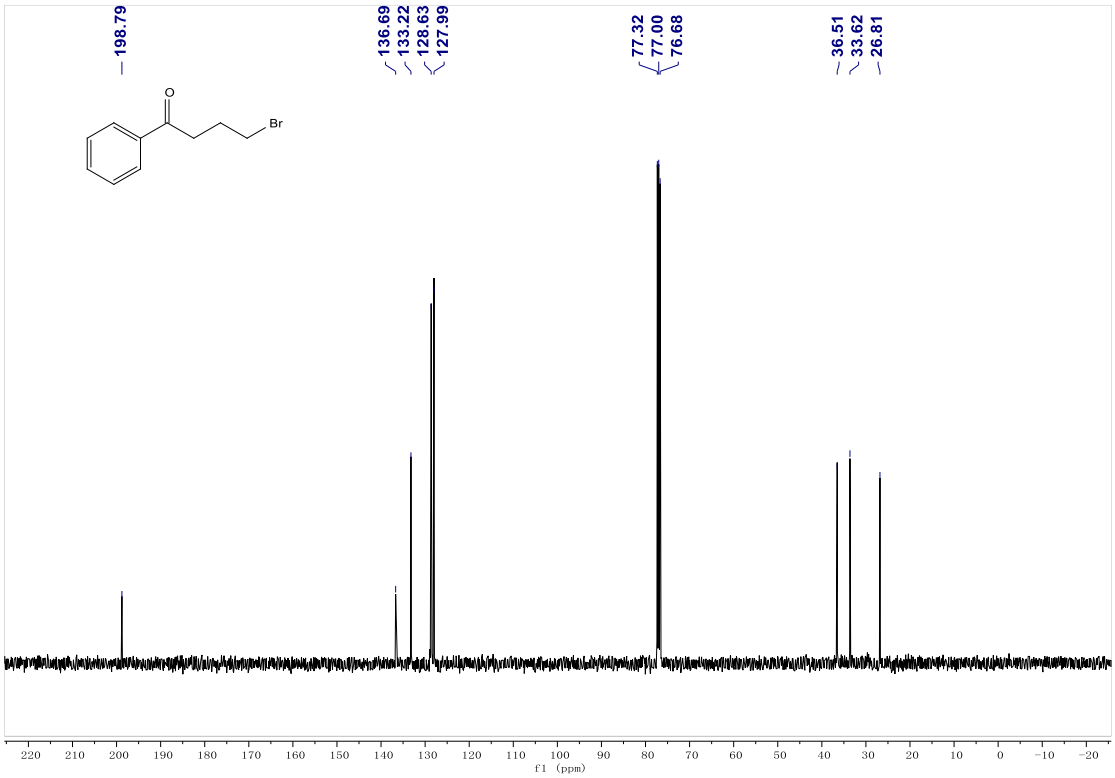
^{13}C NMR (101 MHz, $CDCl_3$) of **5o**



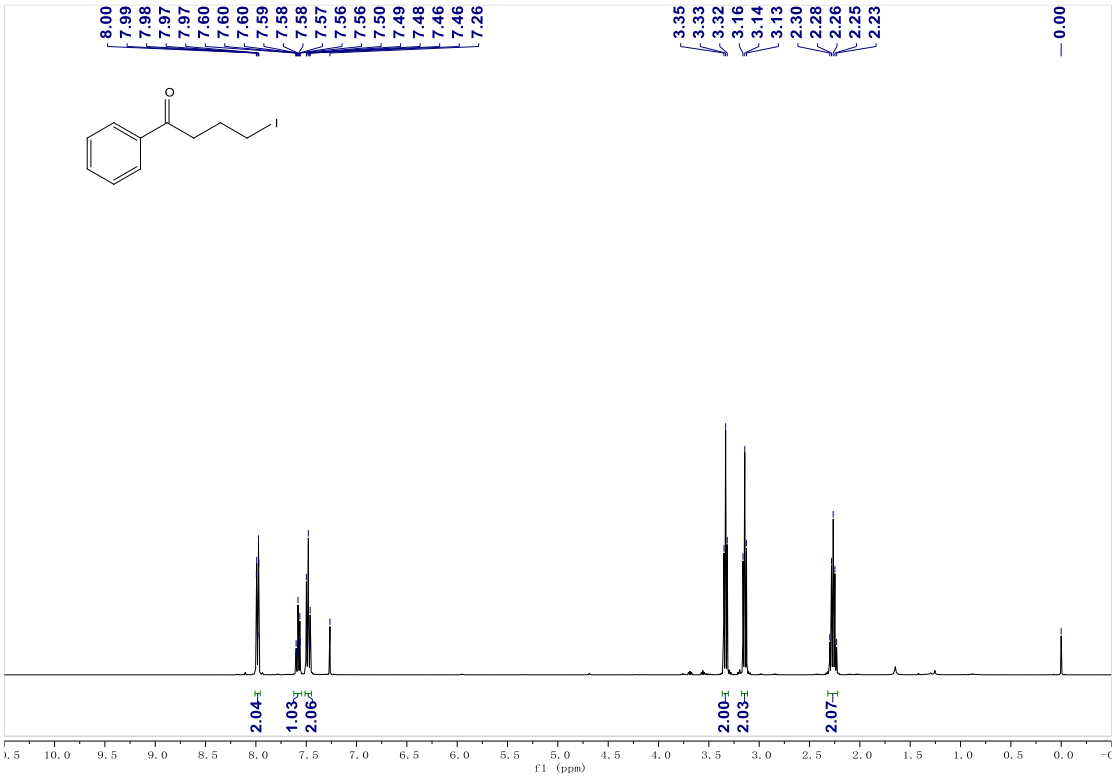
^{1}H NMR (400 MHz, $CDCl_3$) of **4p**



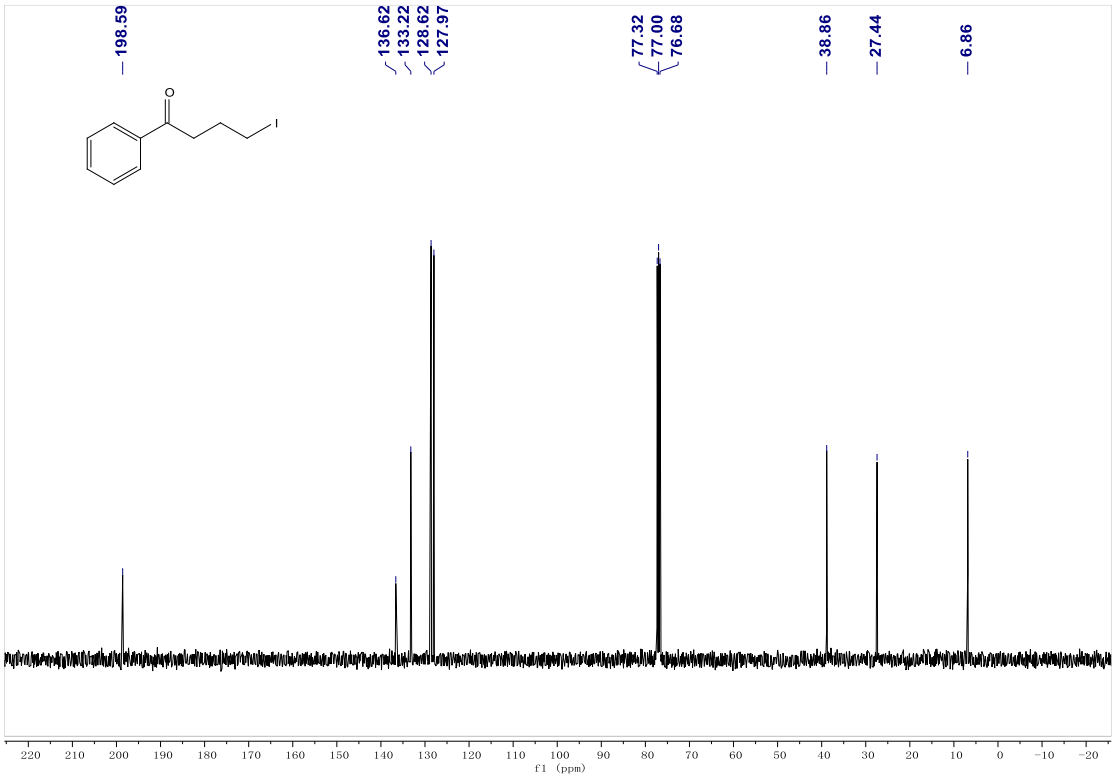
^{13}C NMR (101 MHz, $CDCl_3$) of **4p**



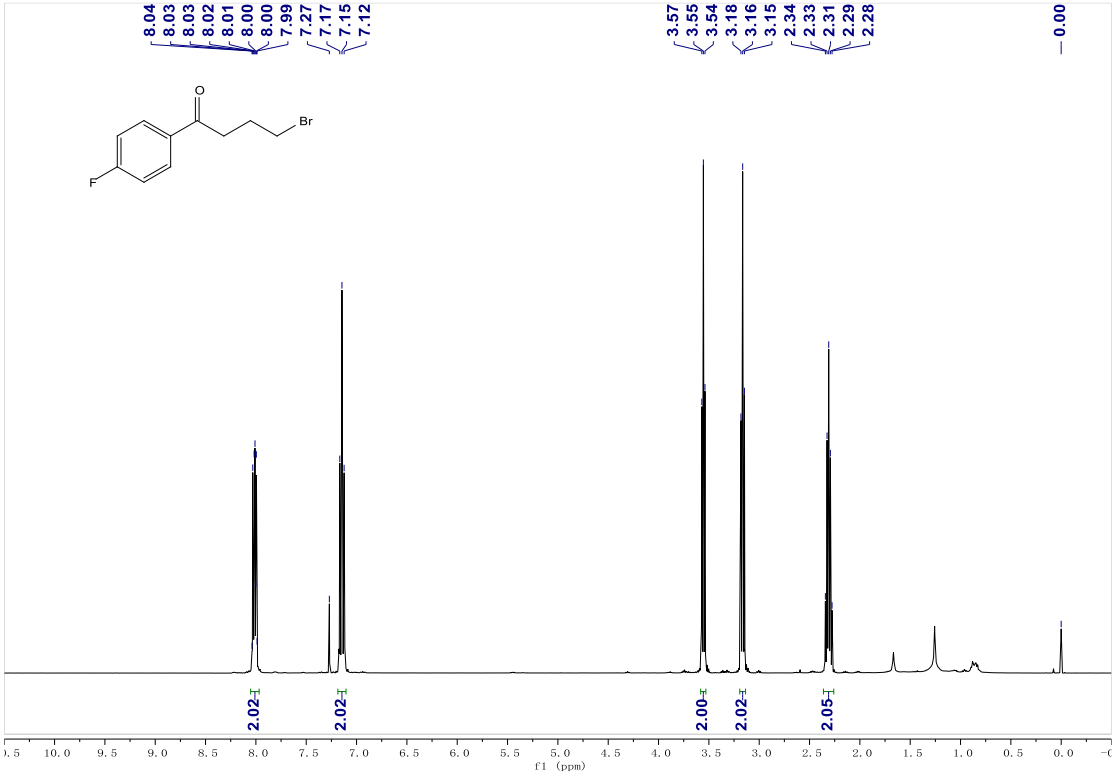
^{1}H NMR (400 MHz, $CDCl_3$) of **5p**



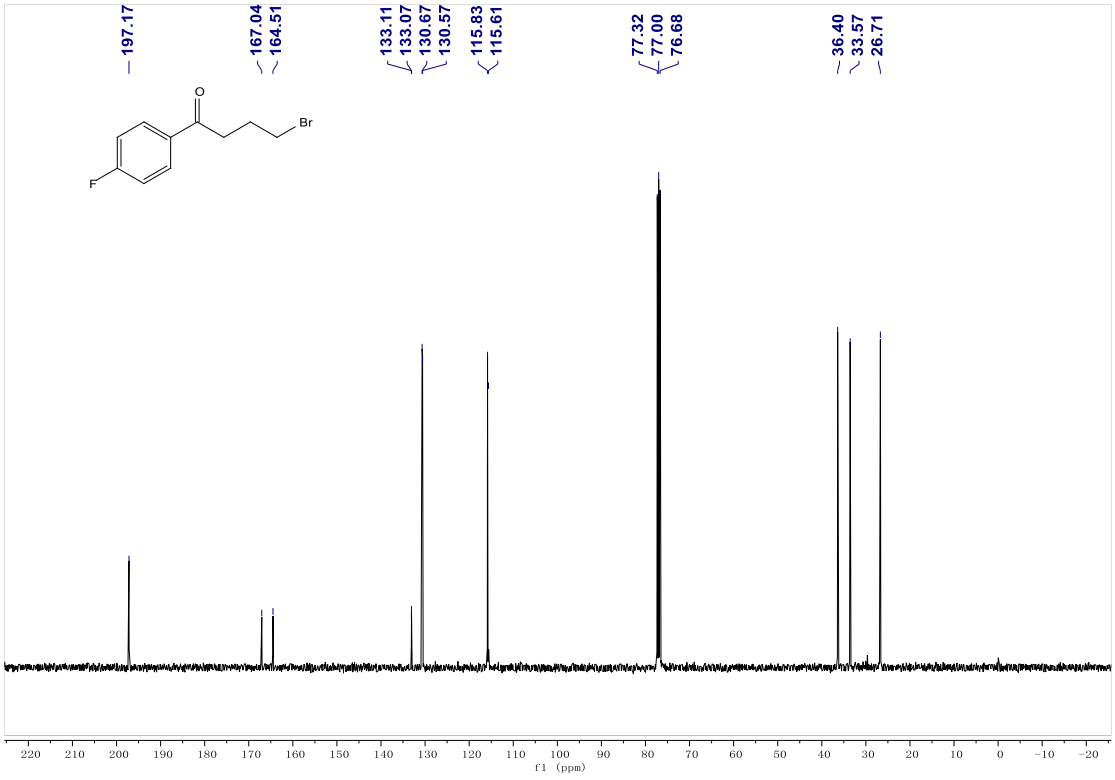
^{13}C NMR (101 MHz, $CDCl_3$) of **5p**



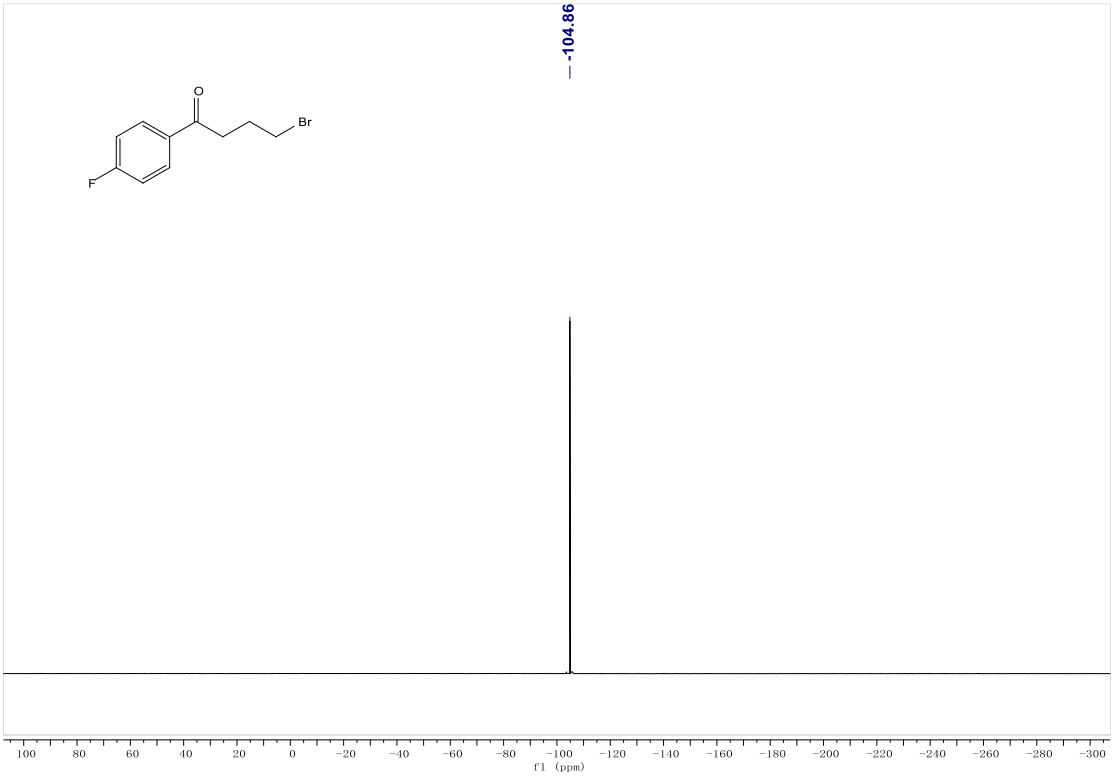
^{1}H NMR (400 MHz, $CDCl_3$) of **5q**



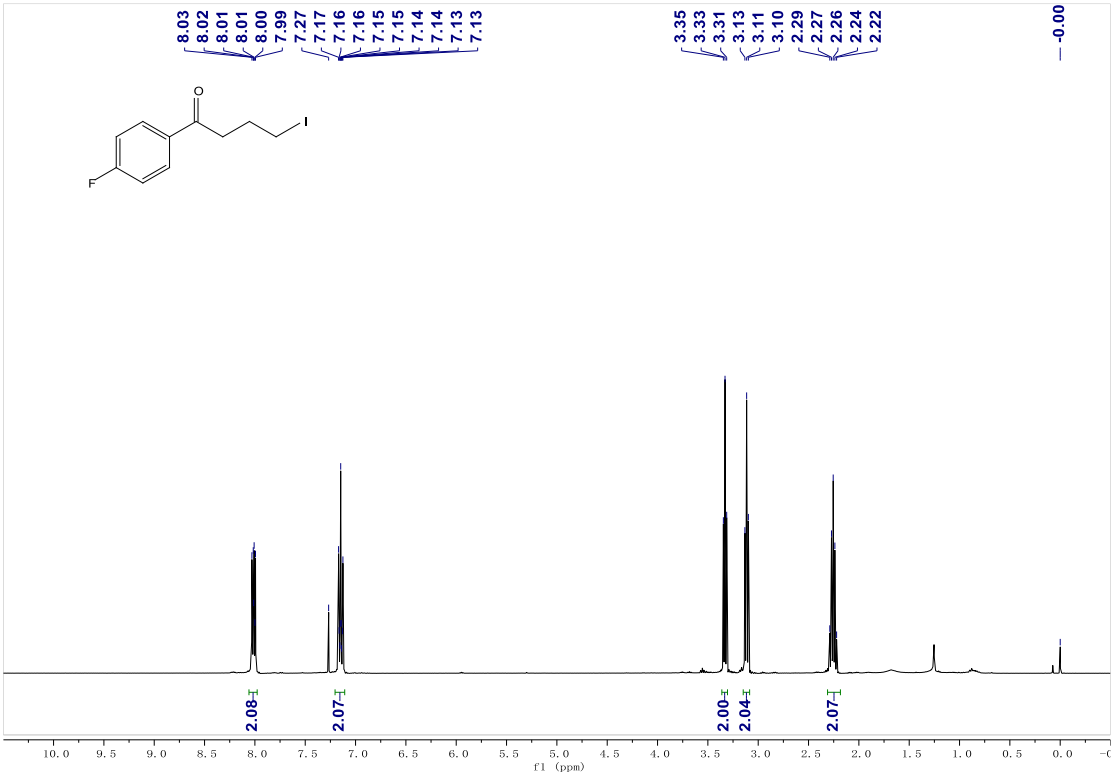
^{13}C NMR (101 MHz, $CDCl_3$) of **5q**



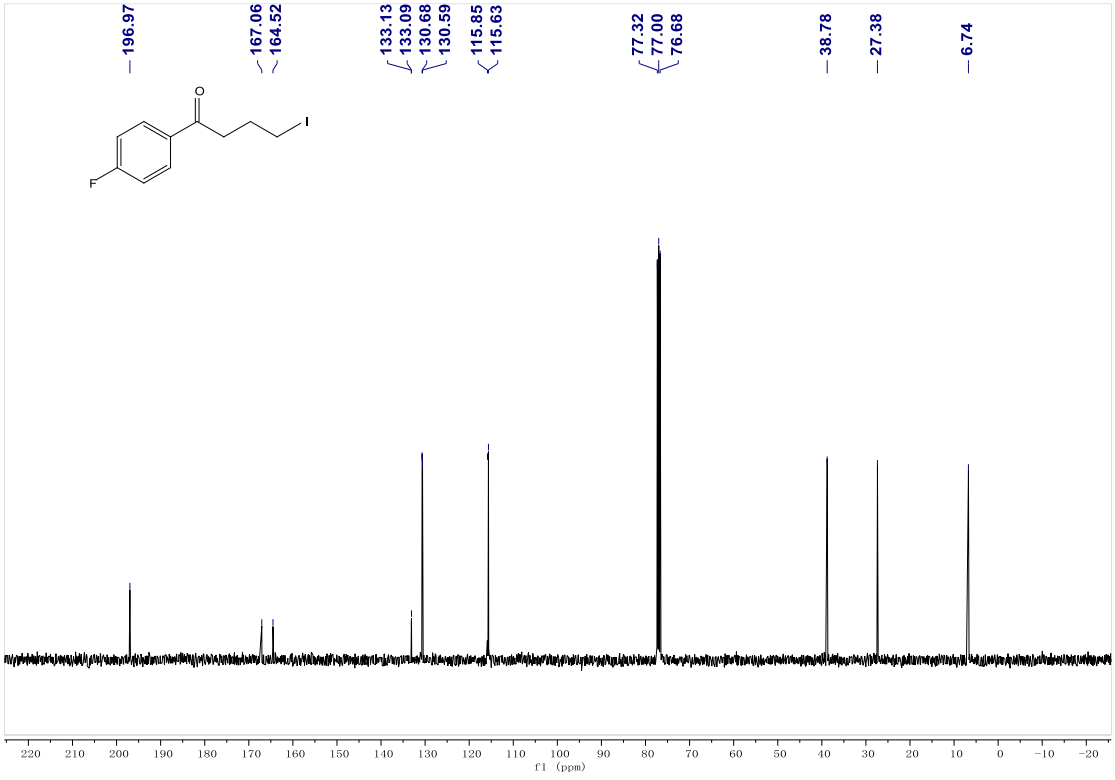
^{19}F NMR (376 MHz, $CDCl_3$) of **5q**



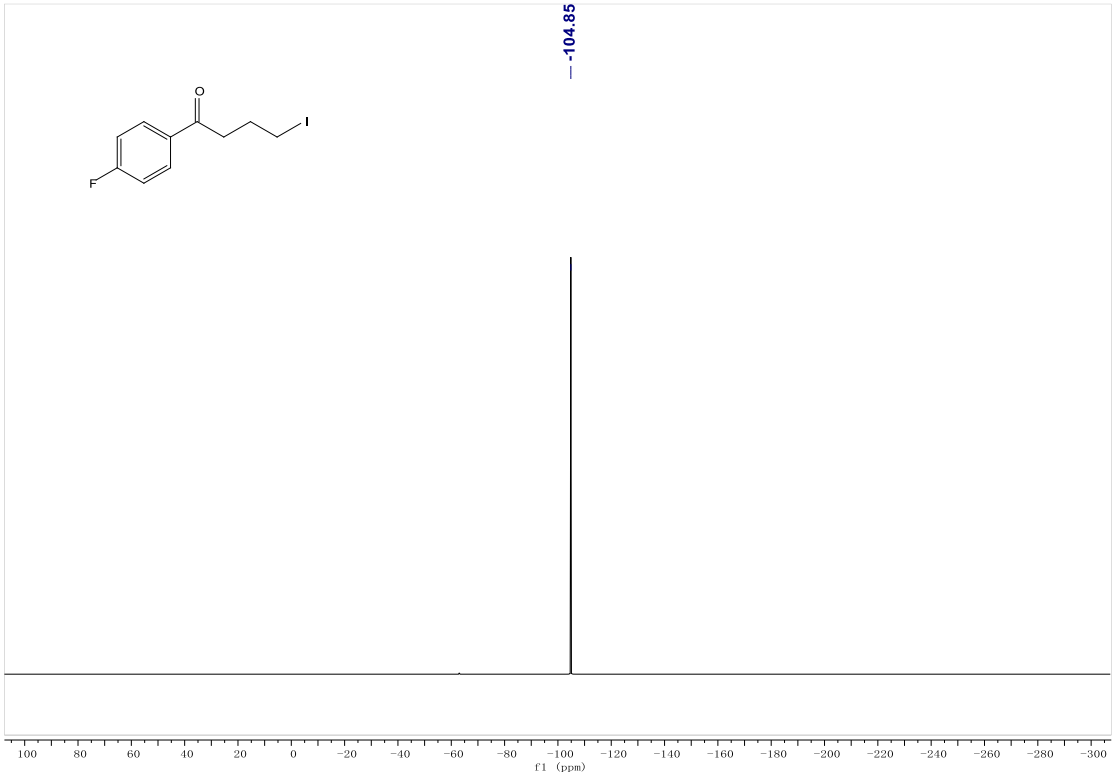
^{1}H NMR (400 MHz, $CDCl_3$) of **4q**



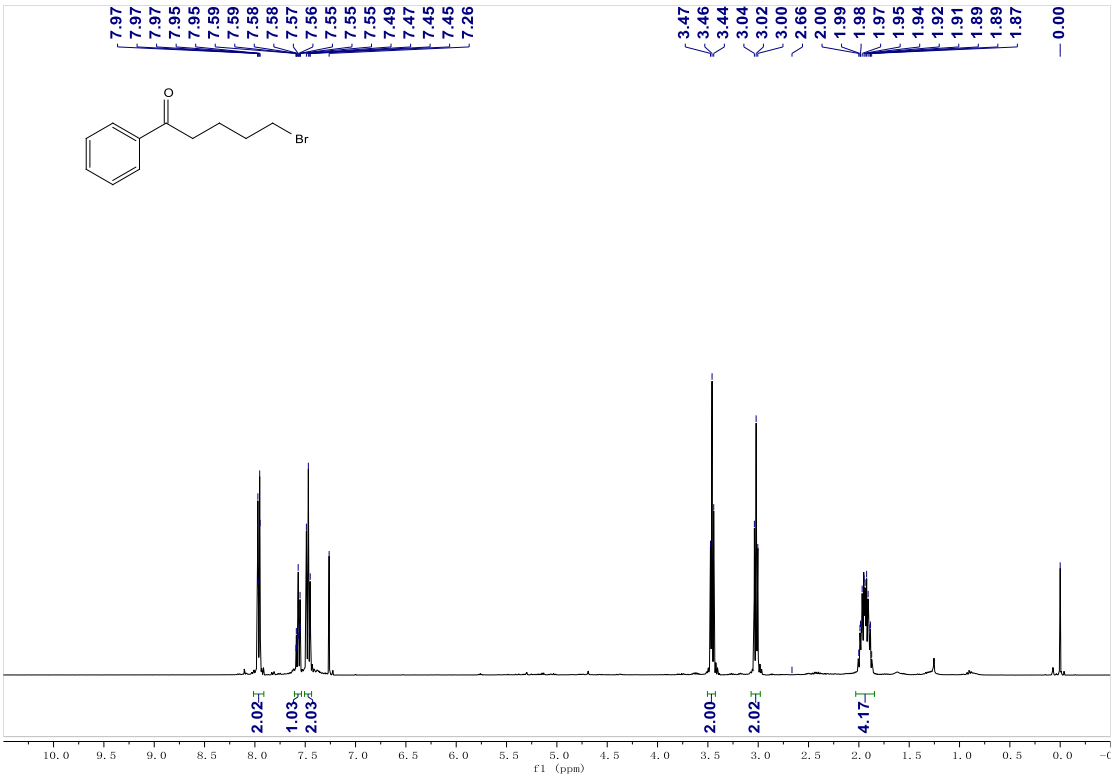
^{13}C NMR (101 MHz, $CDCl_3$) of **4q**



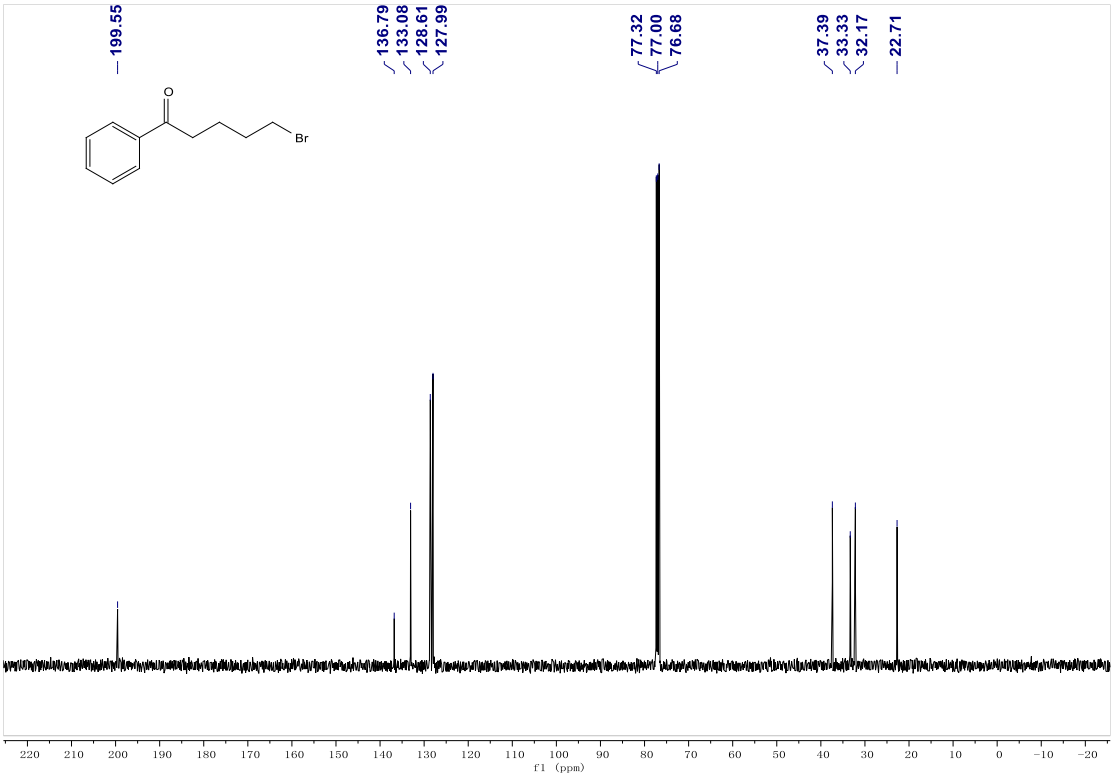
^{19}F NMR (376 MHz, $CDCl_3$) of **4q**



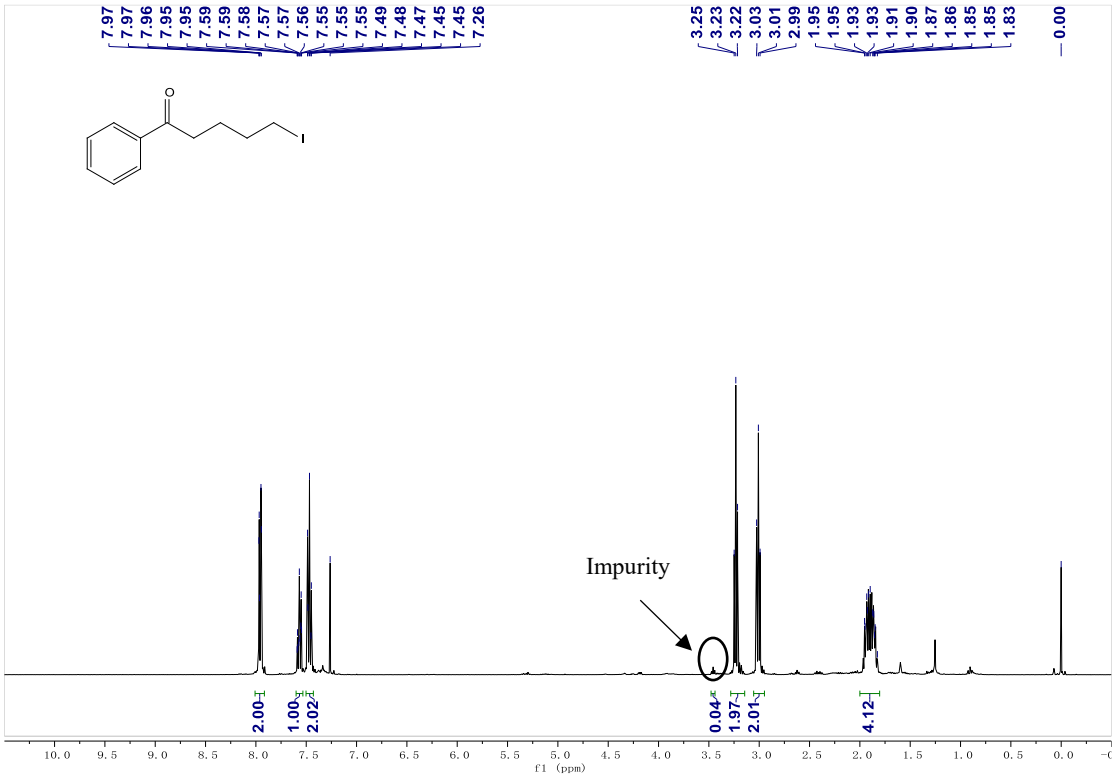
^{1}H NMR (400 MHz, $CDCl_3$) of **4r**



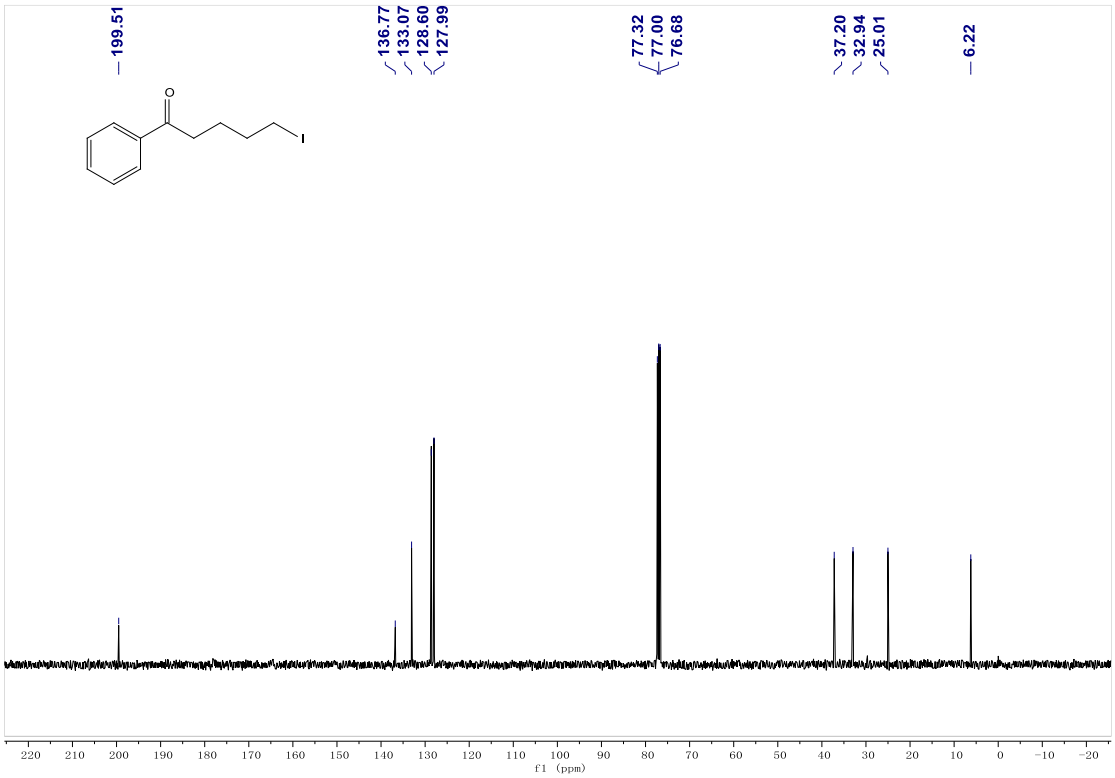
^{13}C NMR (101 MHz, $CDCl_3$) of **4r**



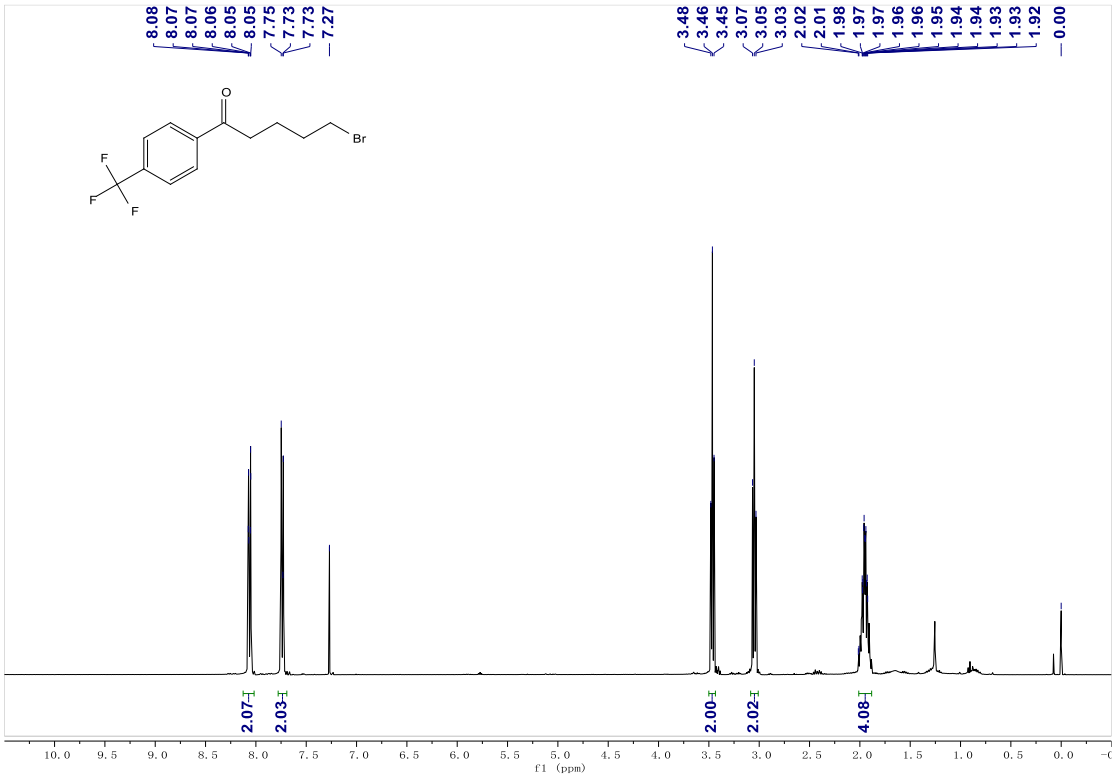
^{1}H NMR (400 MHz, $CDCl_3$) of **5r**



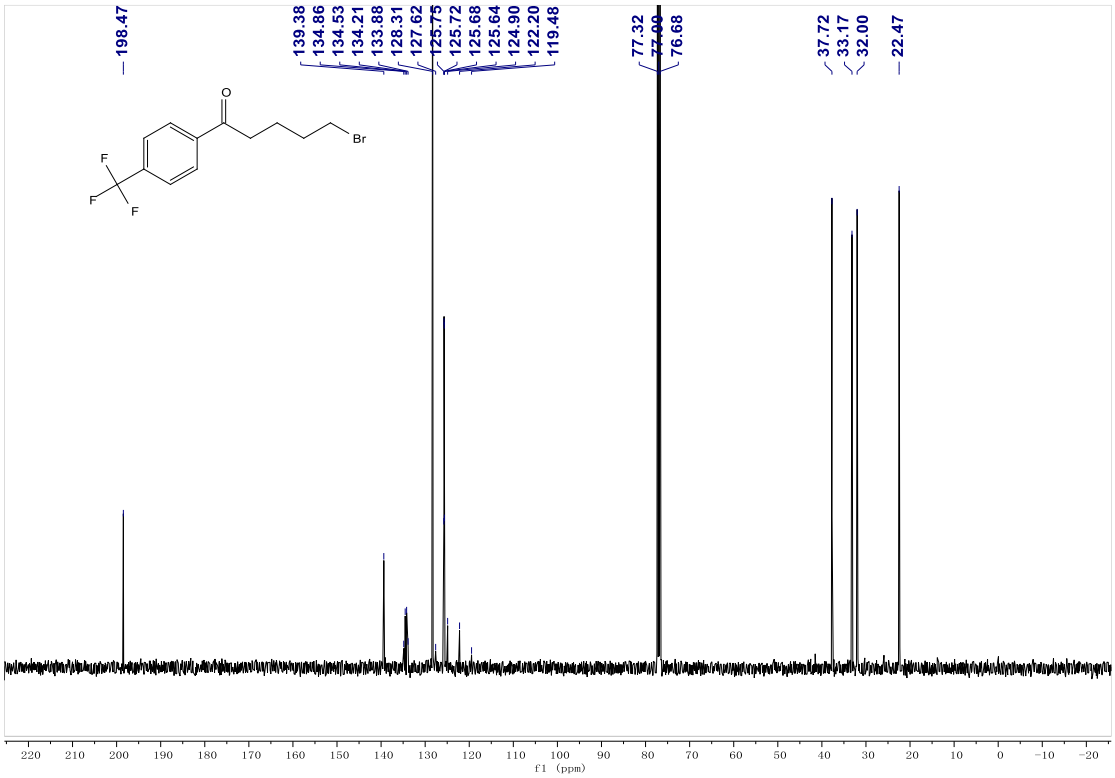
^{13}C NMR (101 MHz, $CDCl_3$) of **5r**



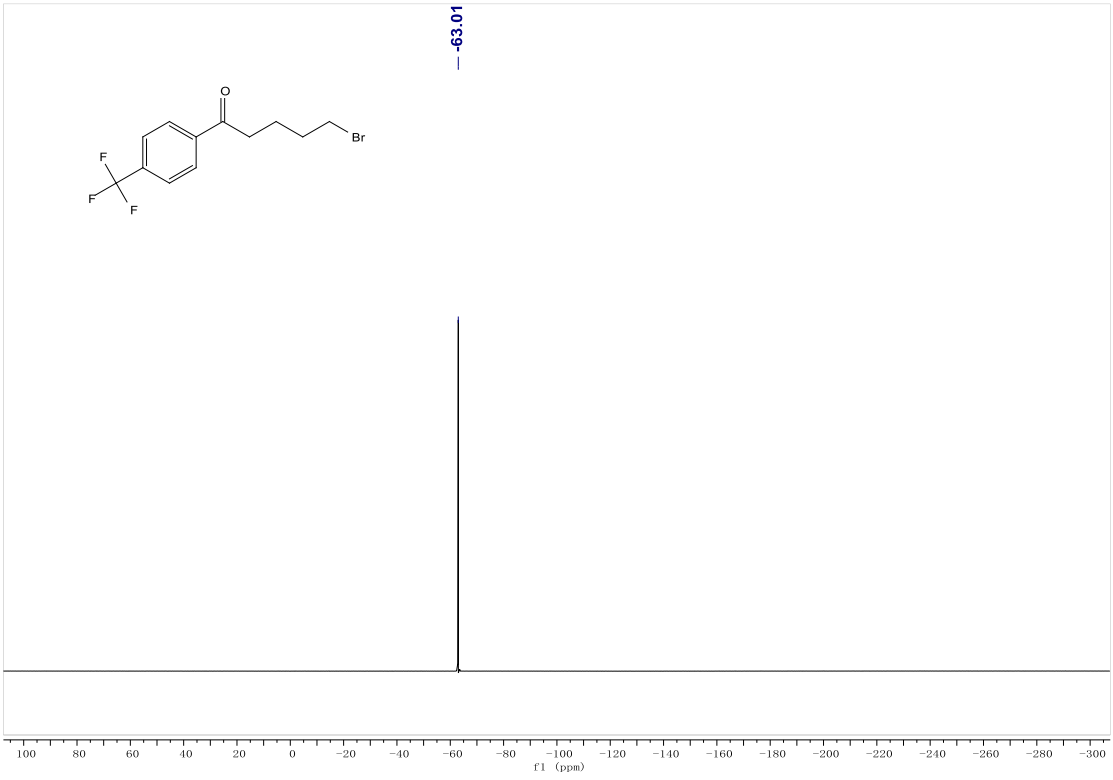
^{1}H NMR (400 MHz, $CDCl_3$) of **4s**



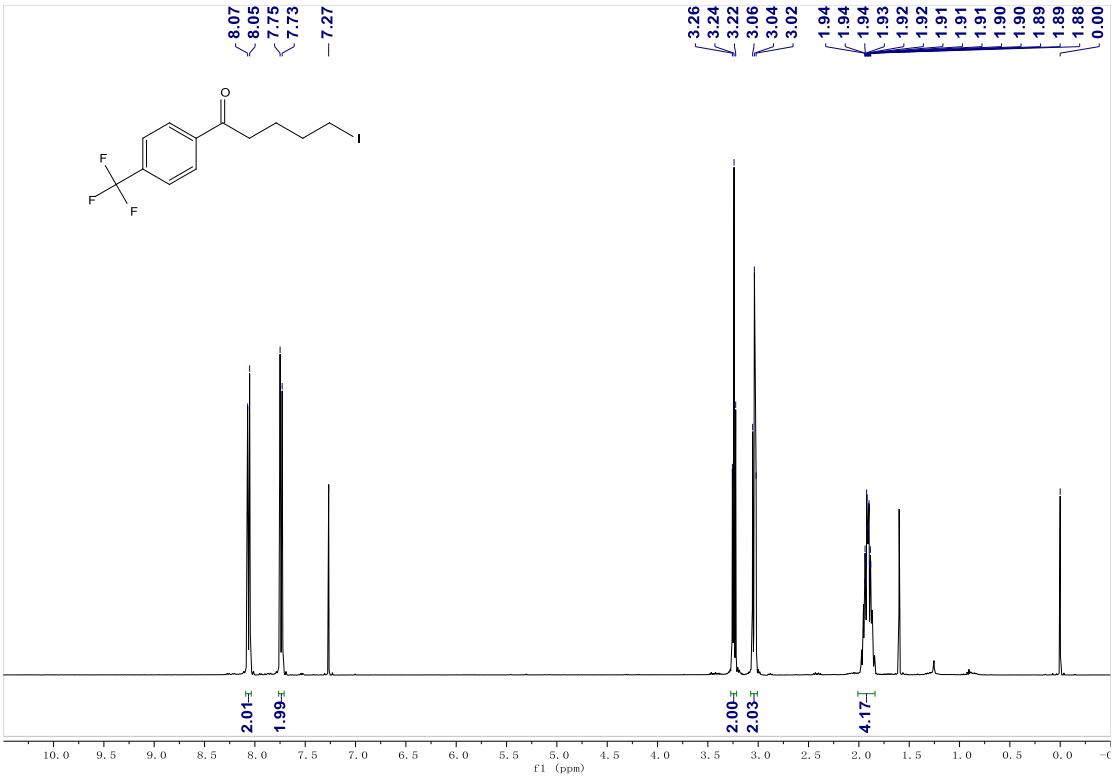
^{13}C NMR (101 MHz, $CDCl_3$) of **4s**



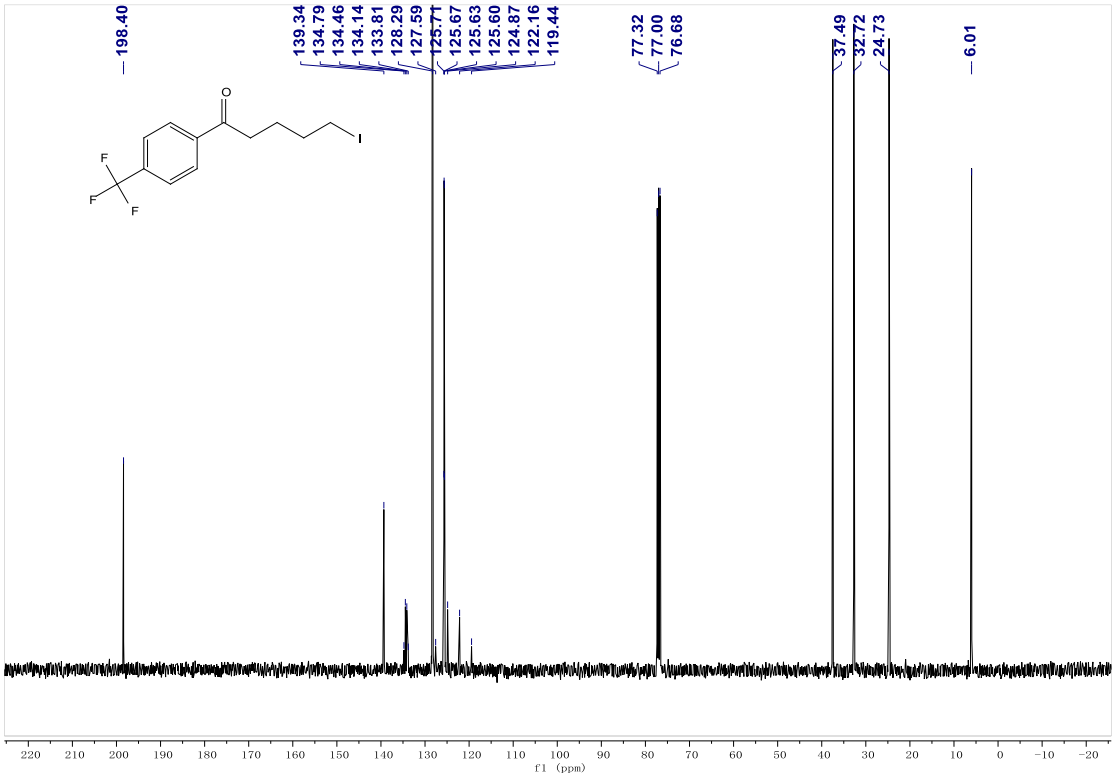
^{19}F NMR (376 MHz, $CDCl_3$) of **5s**



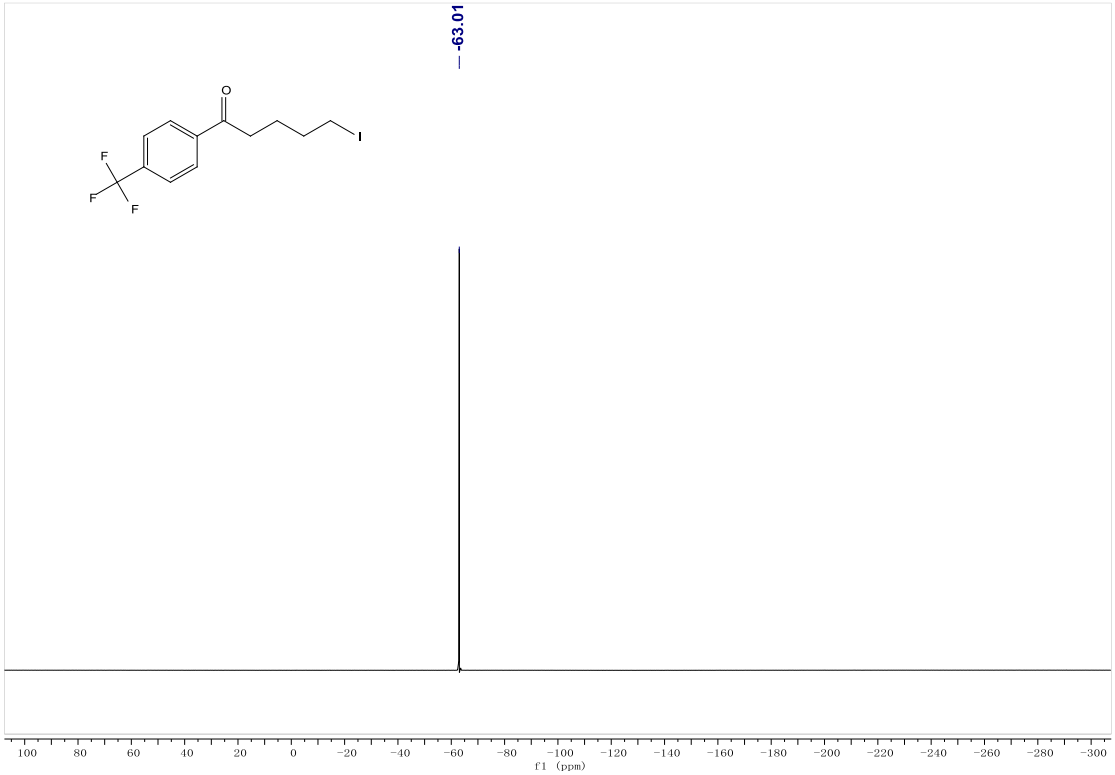
^{1}H NMR (400 MHz, $CDCl_3$) of **5s**



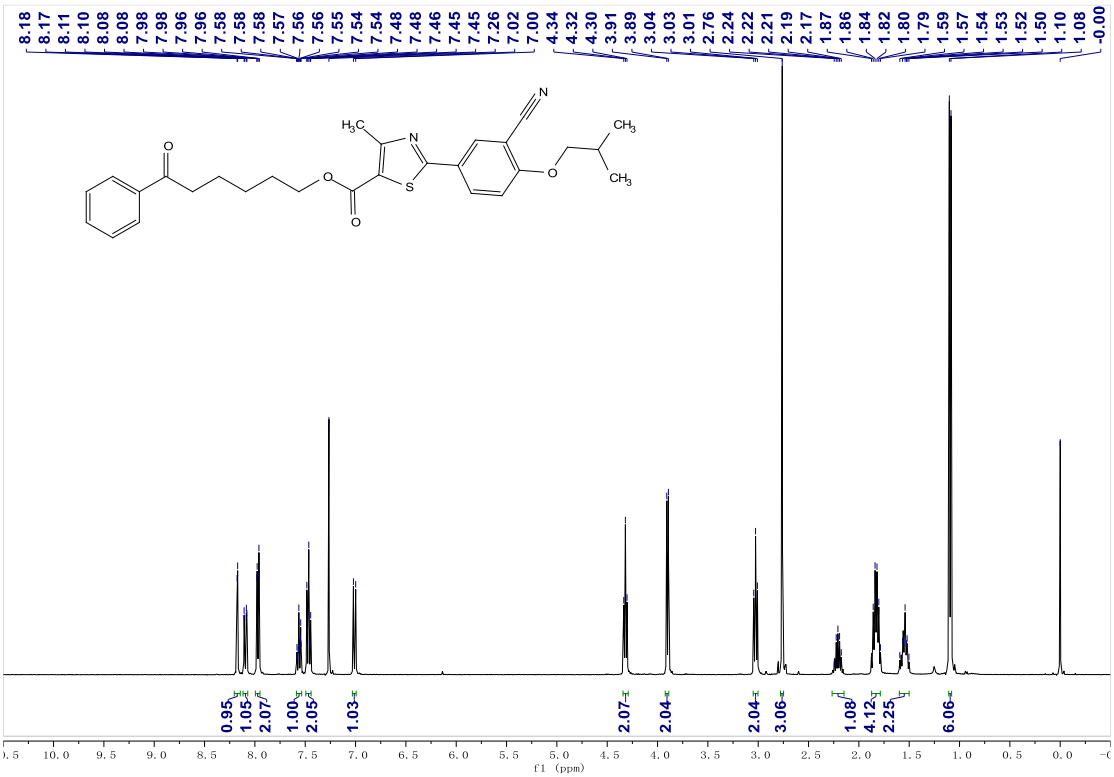
^{13}C NMR (101 MHz, $CDCl_3$) of **5s**



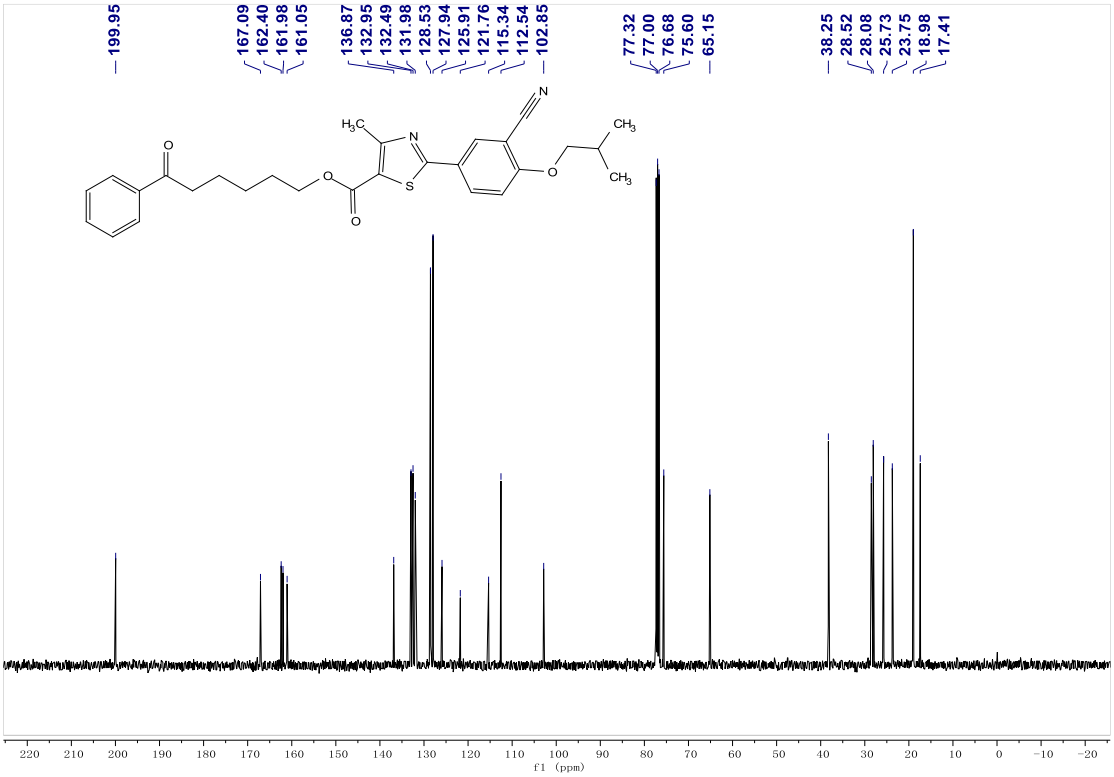
^{19}F NMR (376 MHz, $CDCl_3$) of **5s**



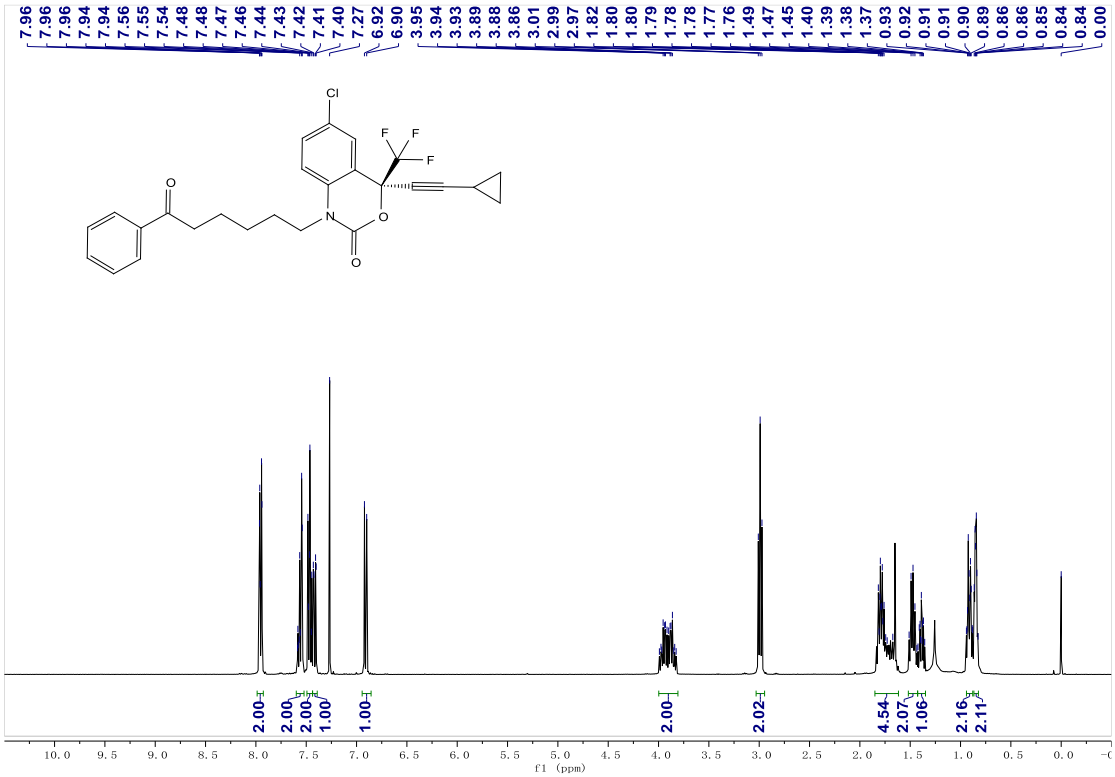
^{1}H NMR (400 MHz, $CDCl_3$) of **6**



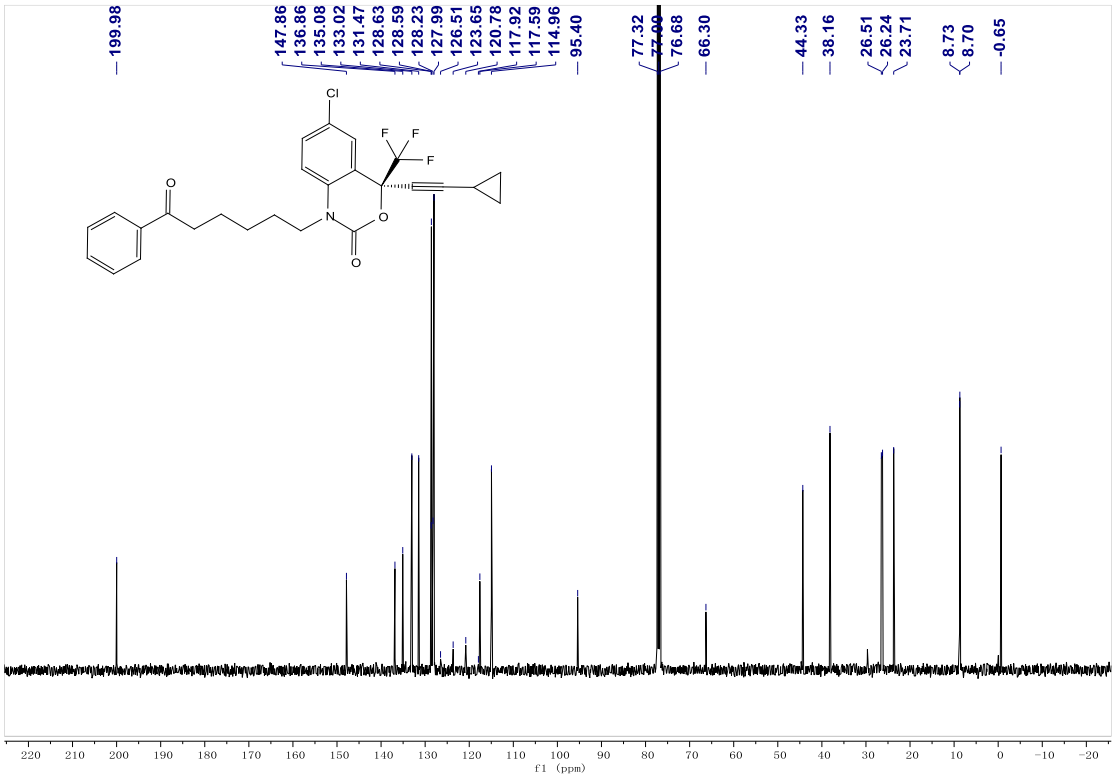
^{13}C NMR (101 MHz, $CDCl_3$) of **6**



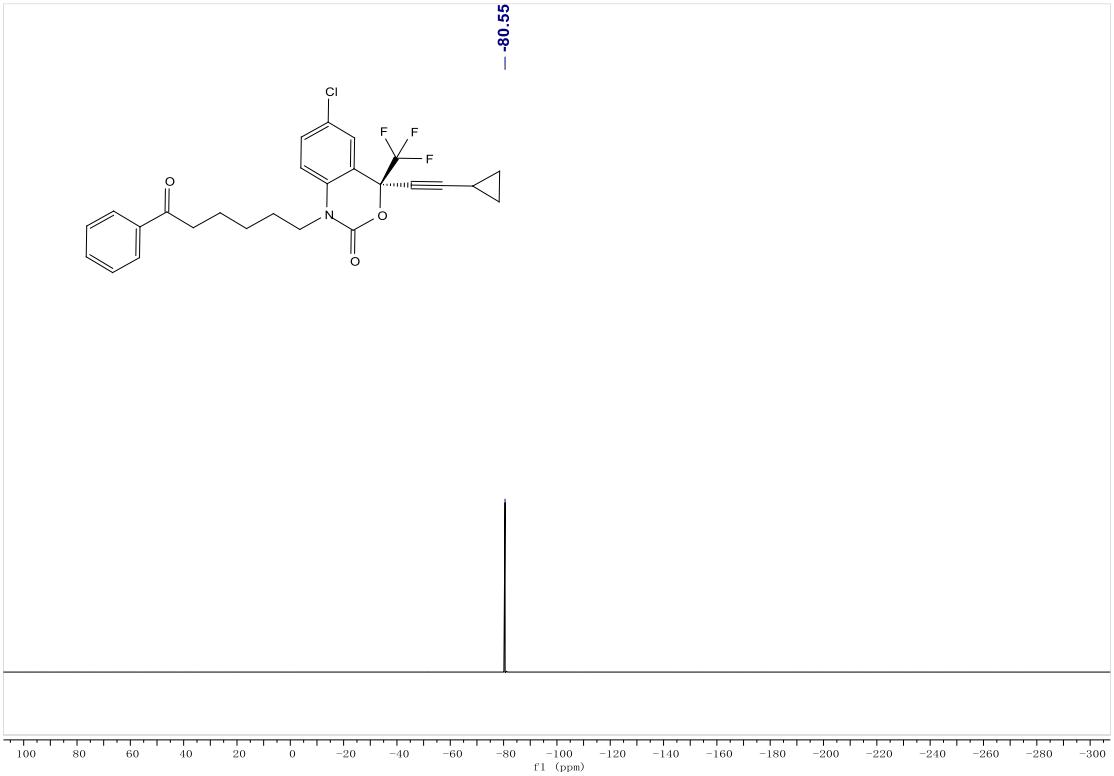
^{1}H NMR (400 MHz, $CDCl_3$) of **7**



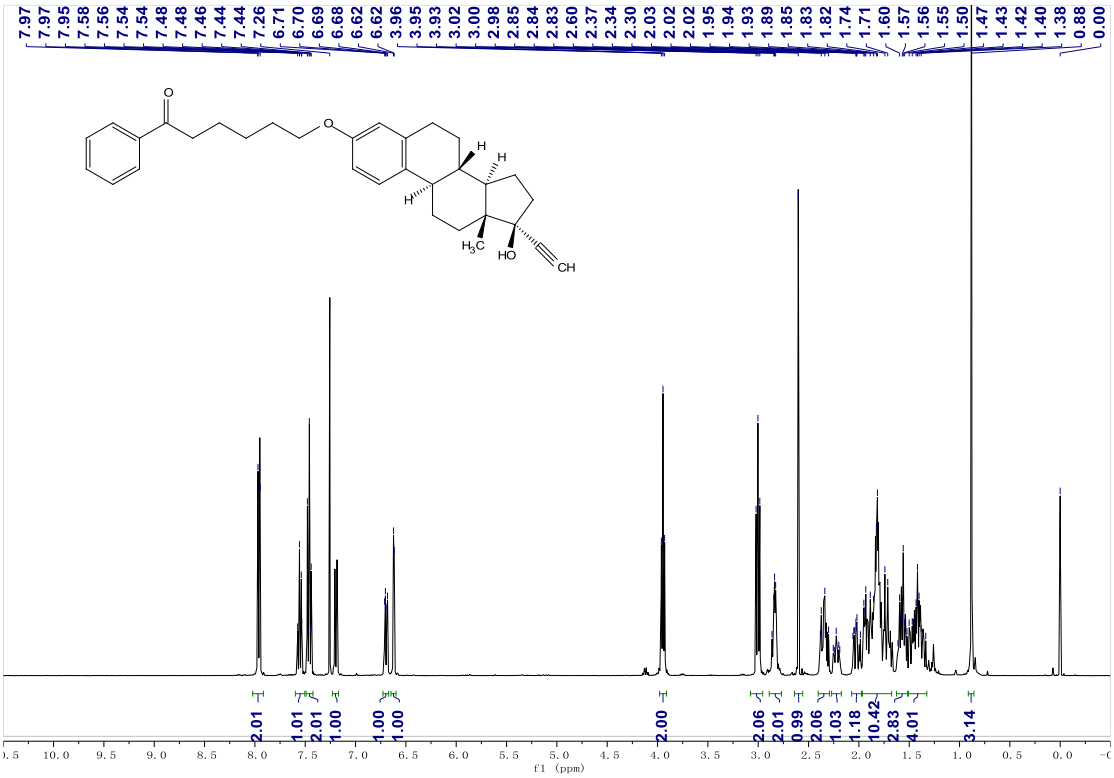
^{13}C NMR (101 MHz, $CDCl_3$) of **7**



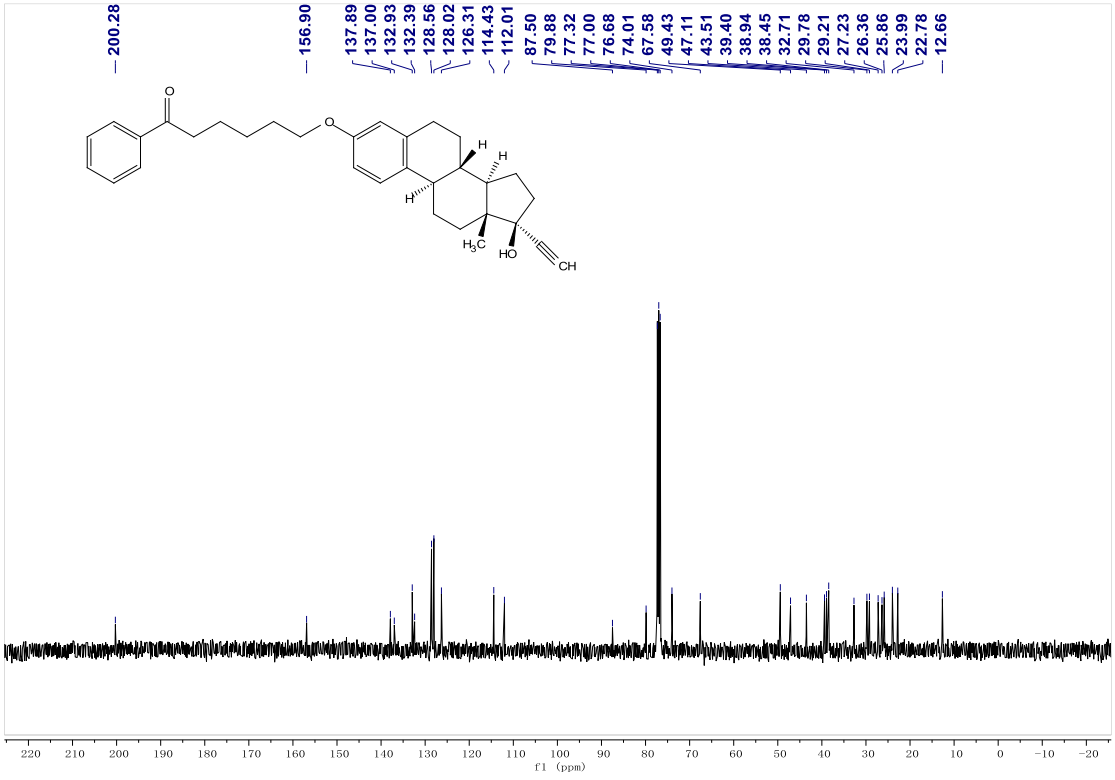
^{19}F NMR (376 MHz, $CDCl_3$) of **7**



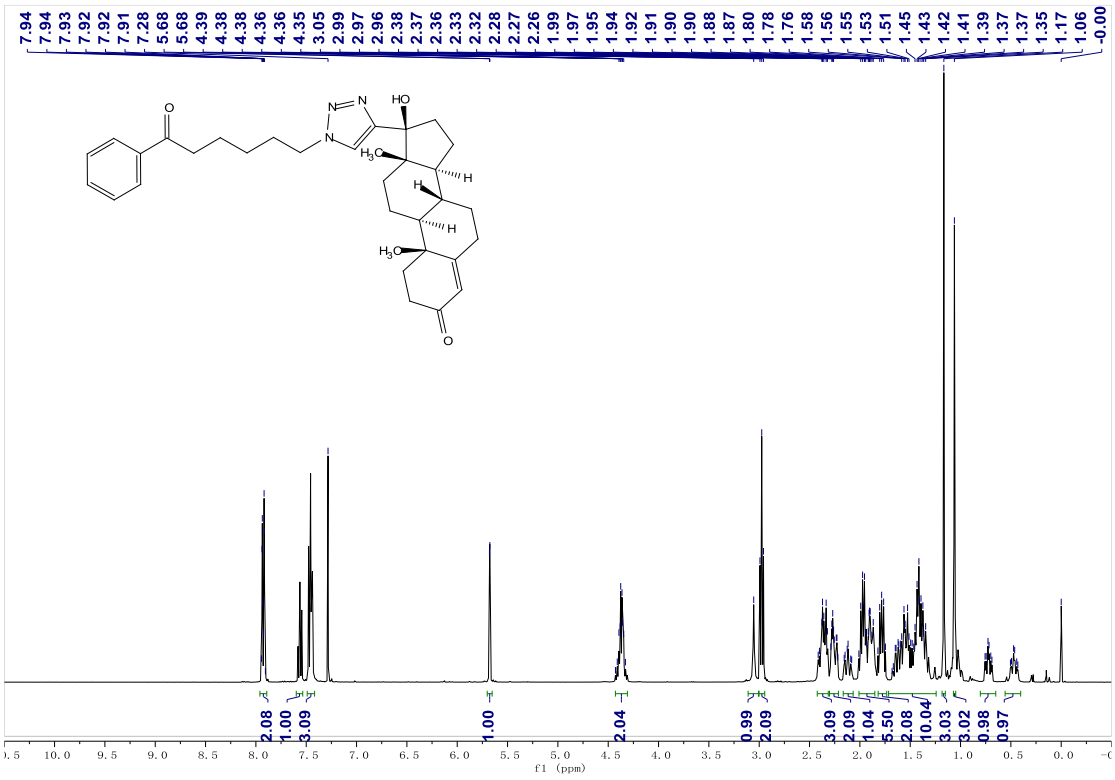
^{1}H NMR (400 MHz, $CDCl_3$) of **8**



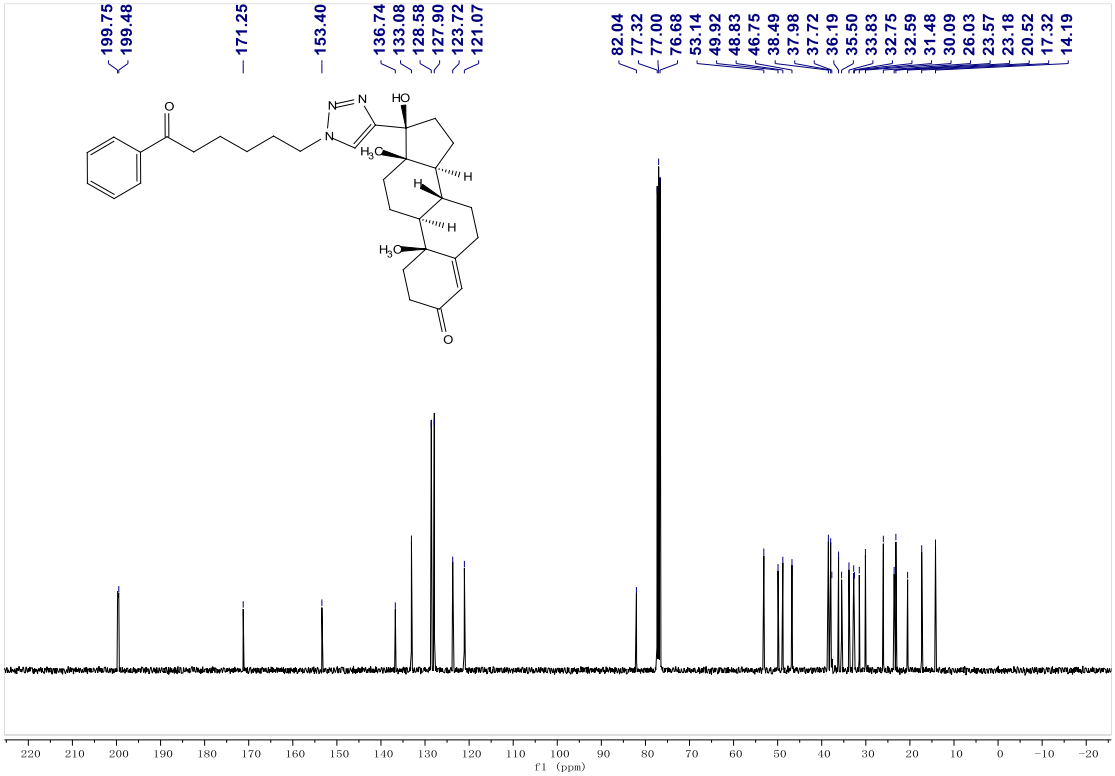
^{13}C NMR (101 MHz, $CDCl_3$) of **8**



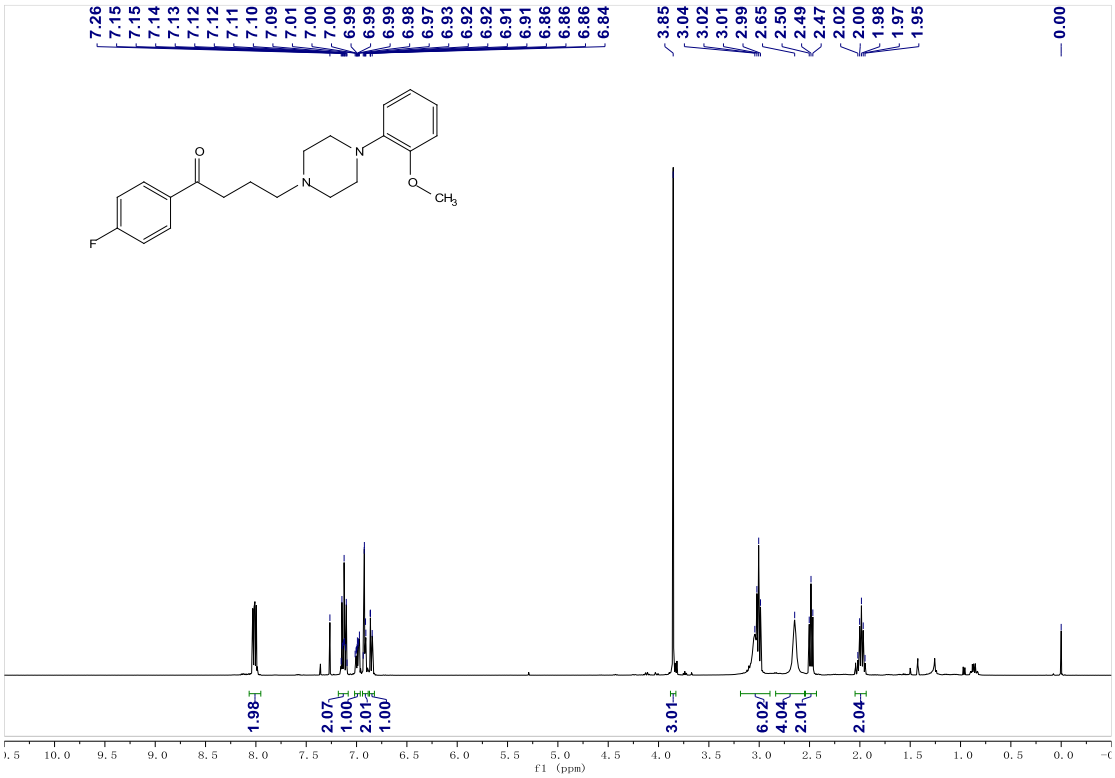
^{1}H NMR (400 MHz, $CDCl_3$) of **9**



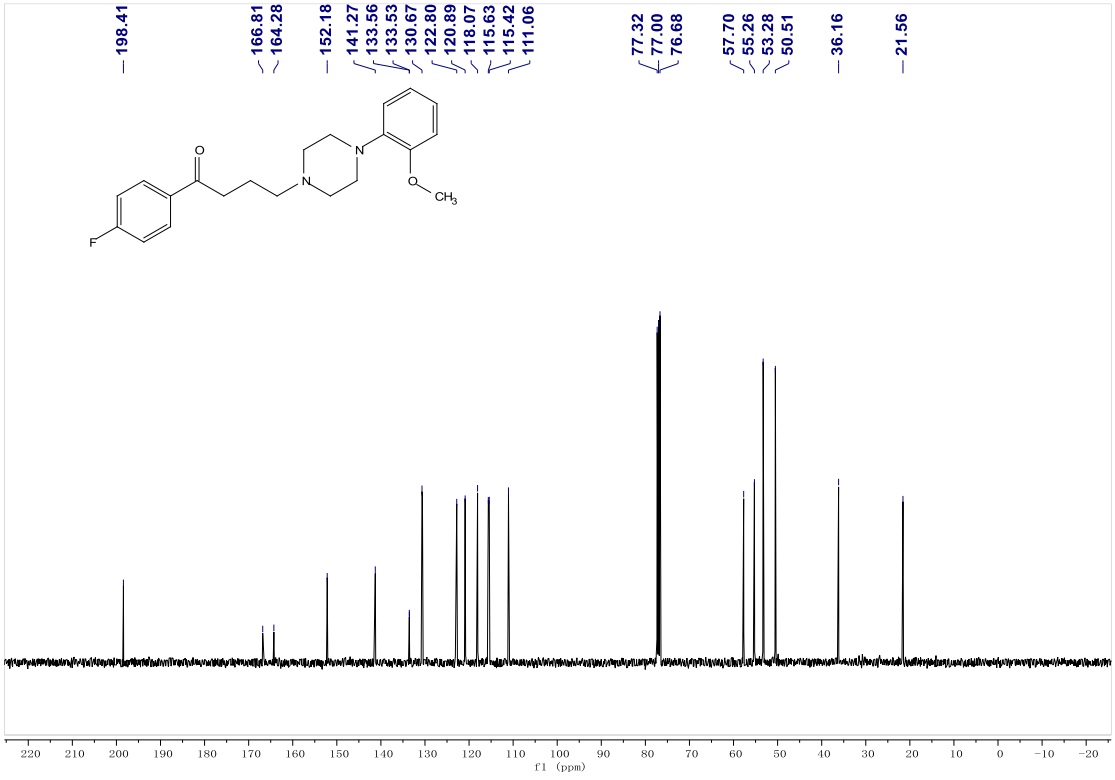
^{13}C NMR (101 MHz, $CDCl_3$) of **9**



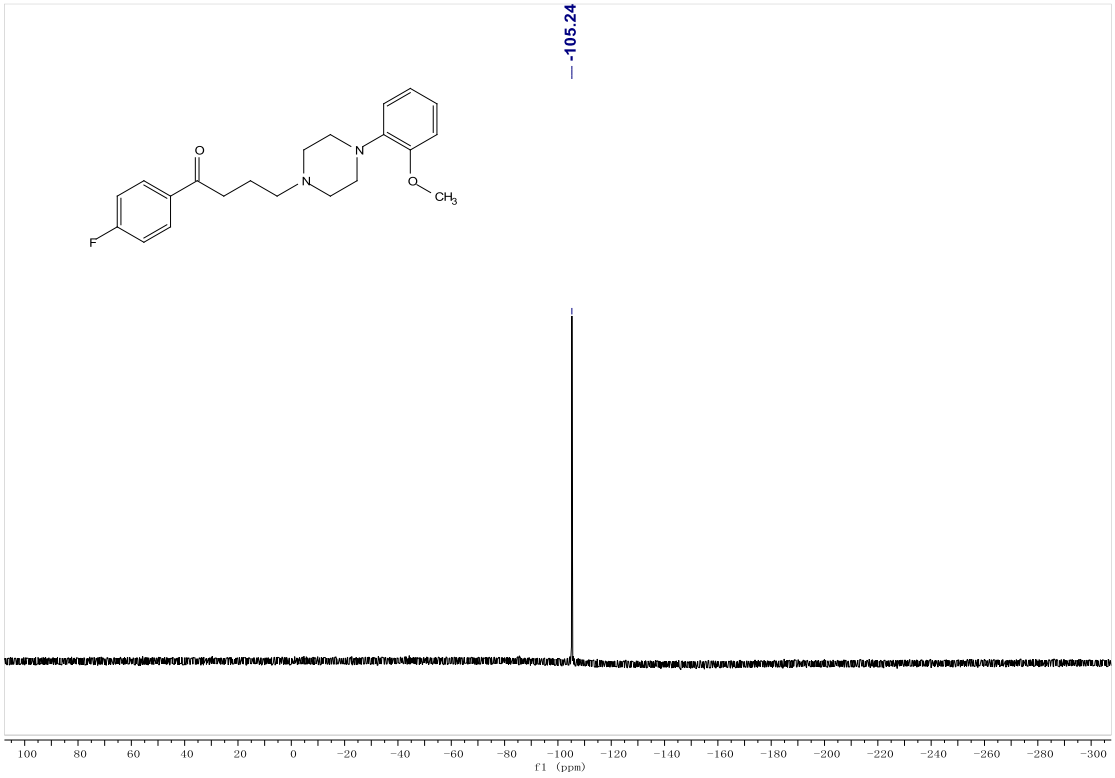
^{1}H NMR (400 MHz, $CDCl_3$) of **10a**



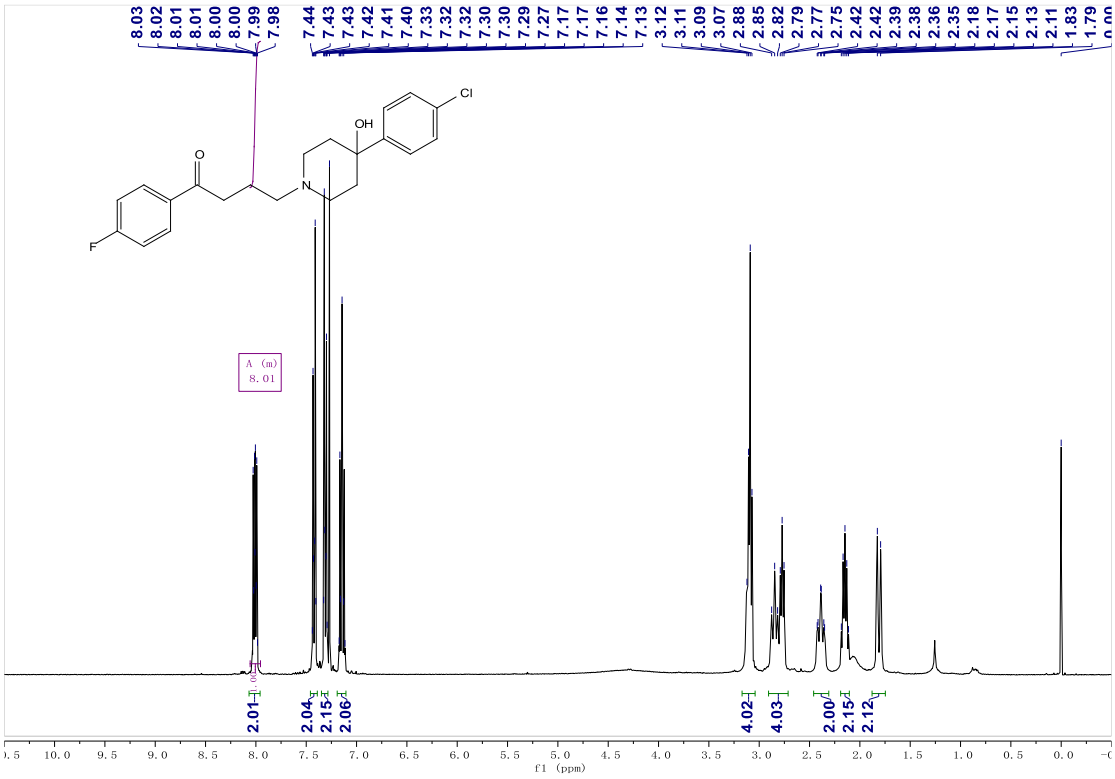
^{13}C NMR (101 MHz, $CDCl_3$) of **10a**



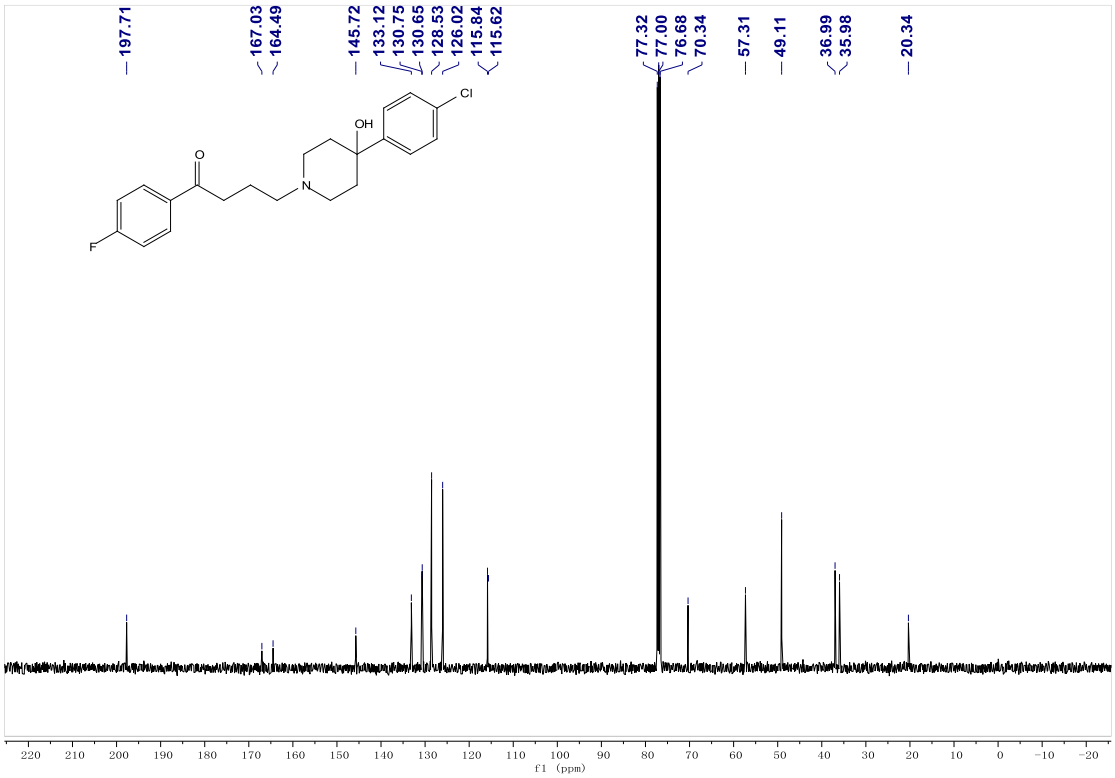
^{19}F NMR (376 MHz, $CDCl_3$) of **10a**



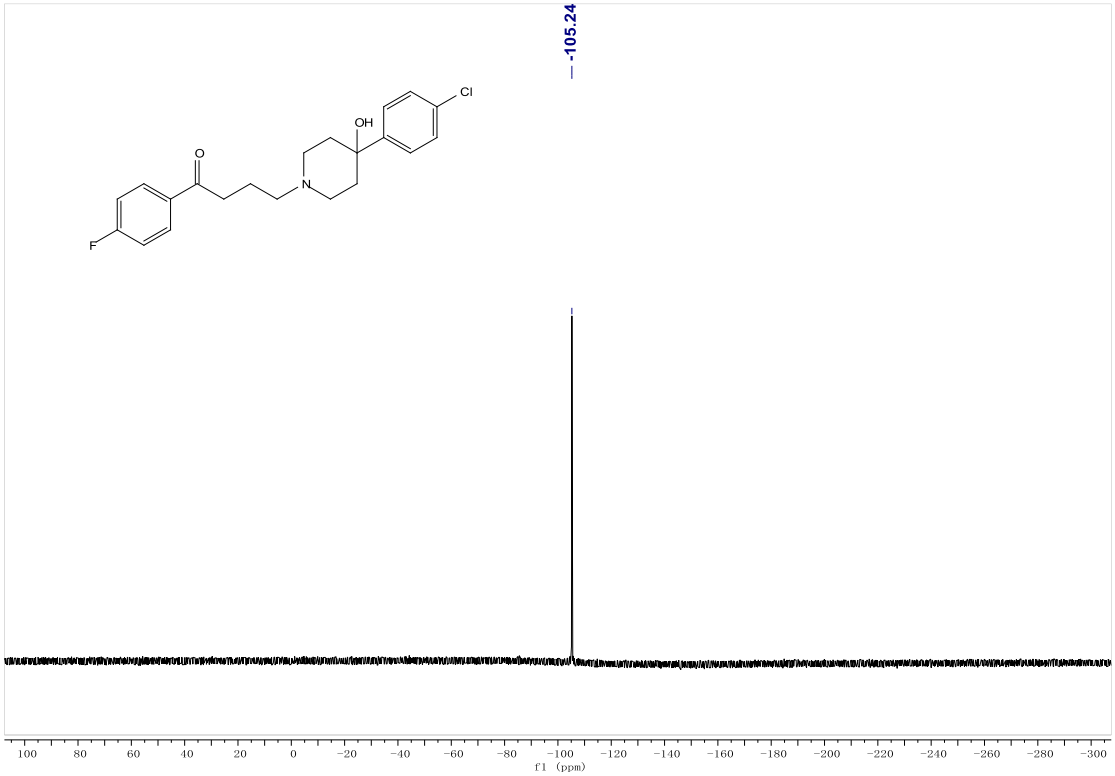
^{1}H NMR (400 MHz, $CDCl_3$) of **10b**



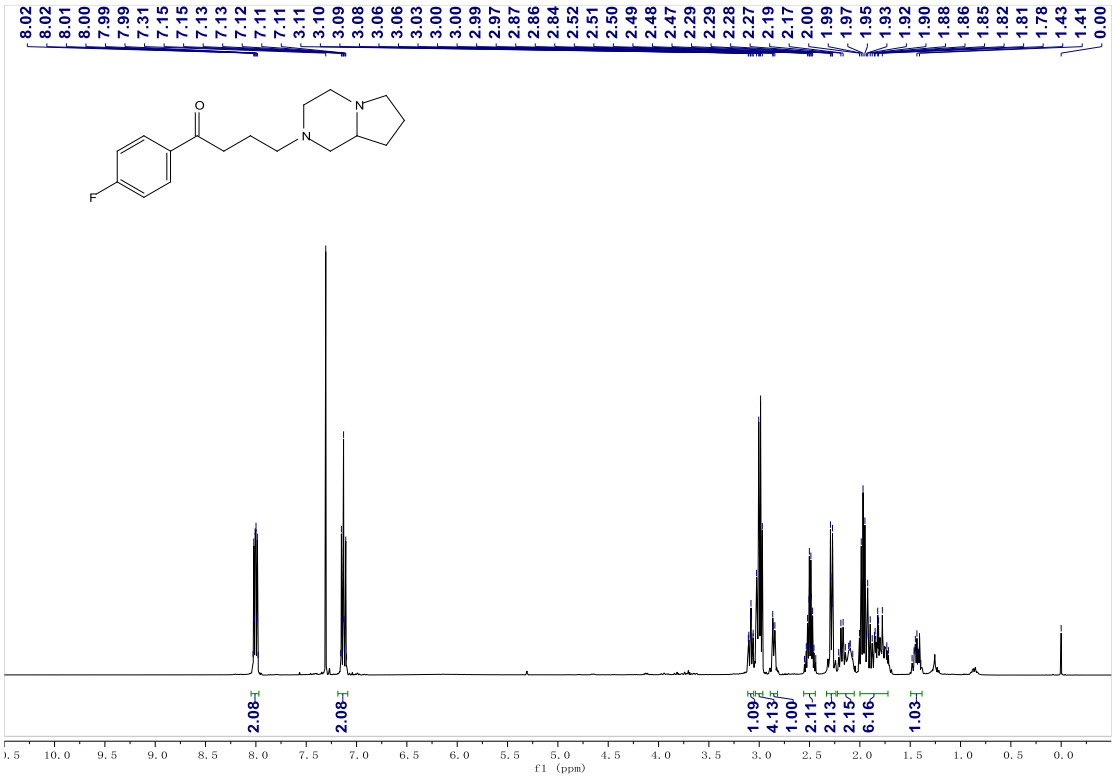
^{13}C NMR (101 MHz, $CDCl_3$) of **10b**



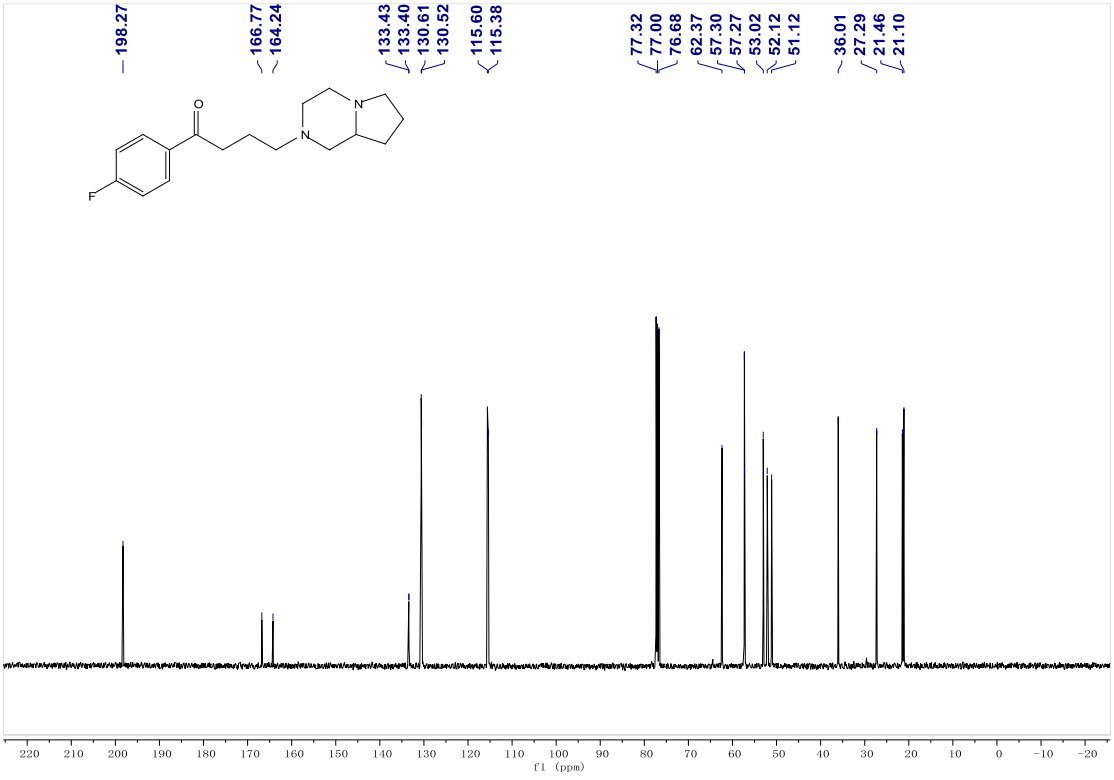
^{19}F NMR (376 MHz, $CDCl_3$) of **10b**



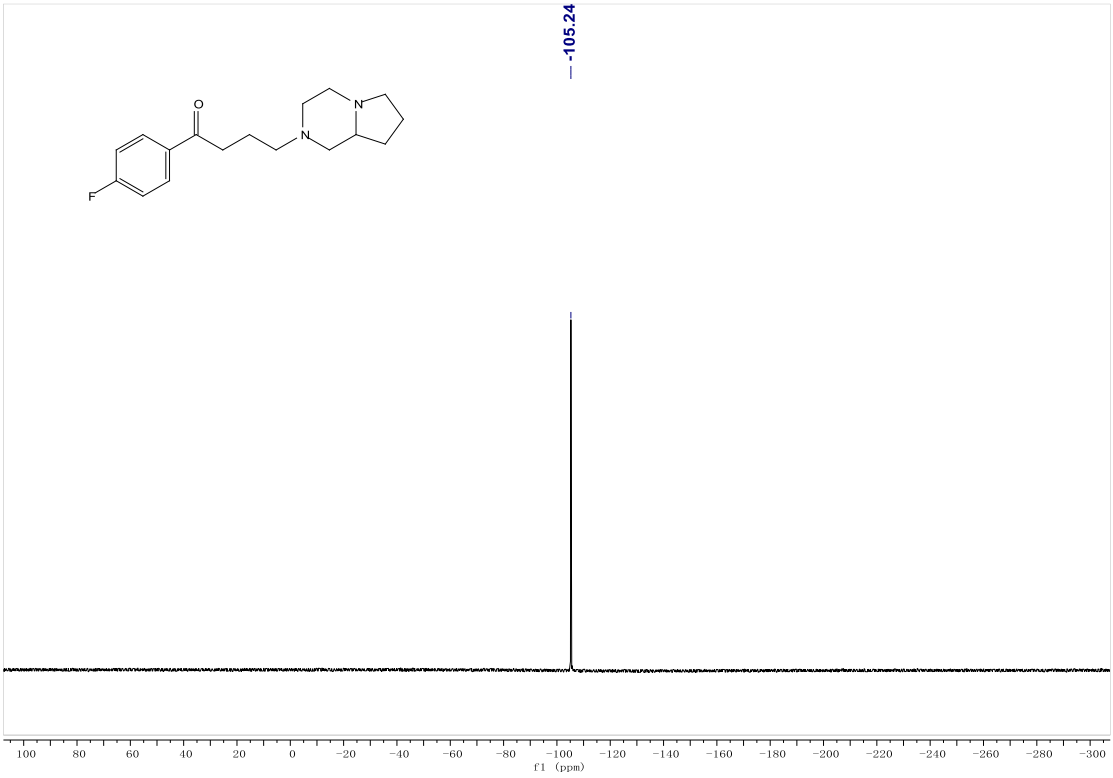
^{1}H NMR (400 MHz, $CDCl_3$) of **10c**



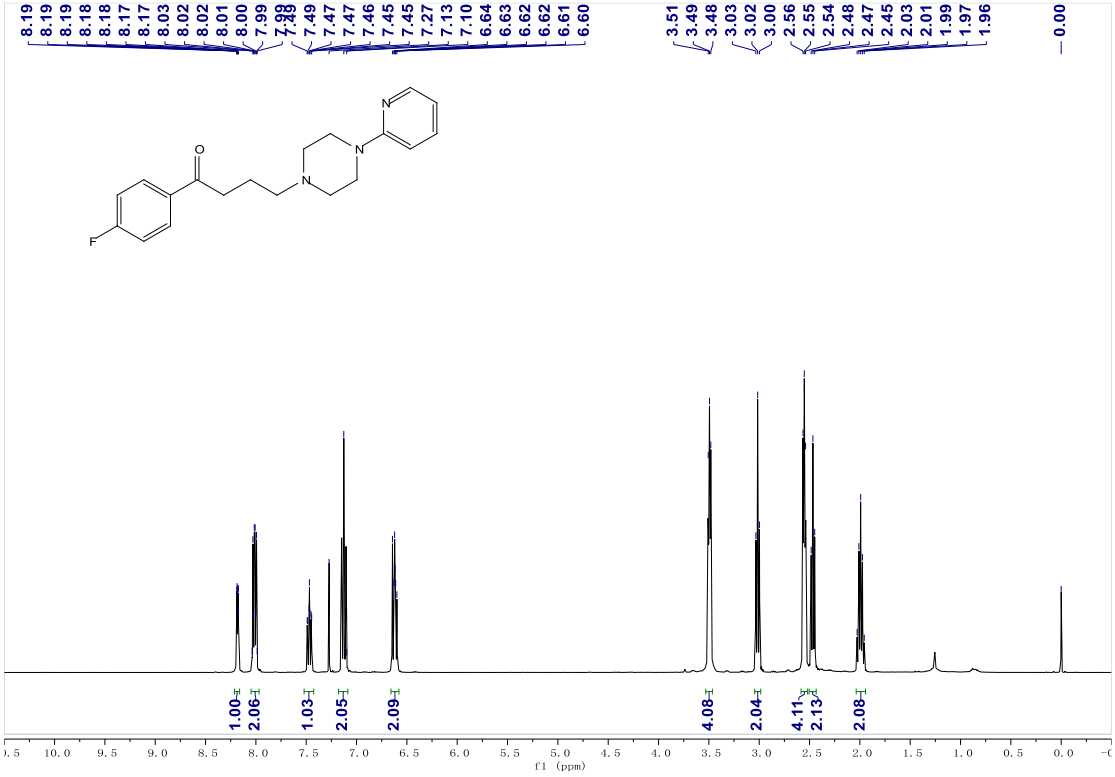
^{13}C NMR (101 MHz, $CDCl_3$) of **10c**



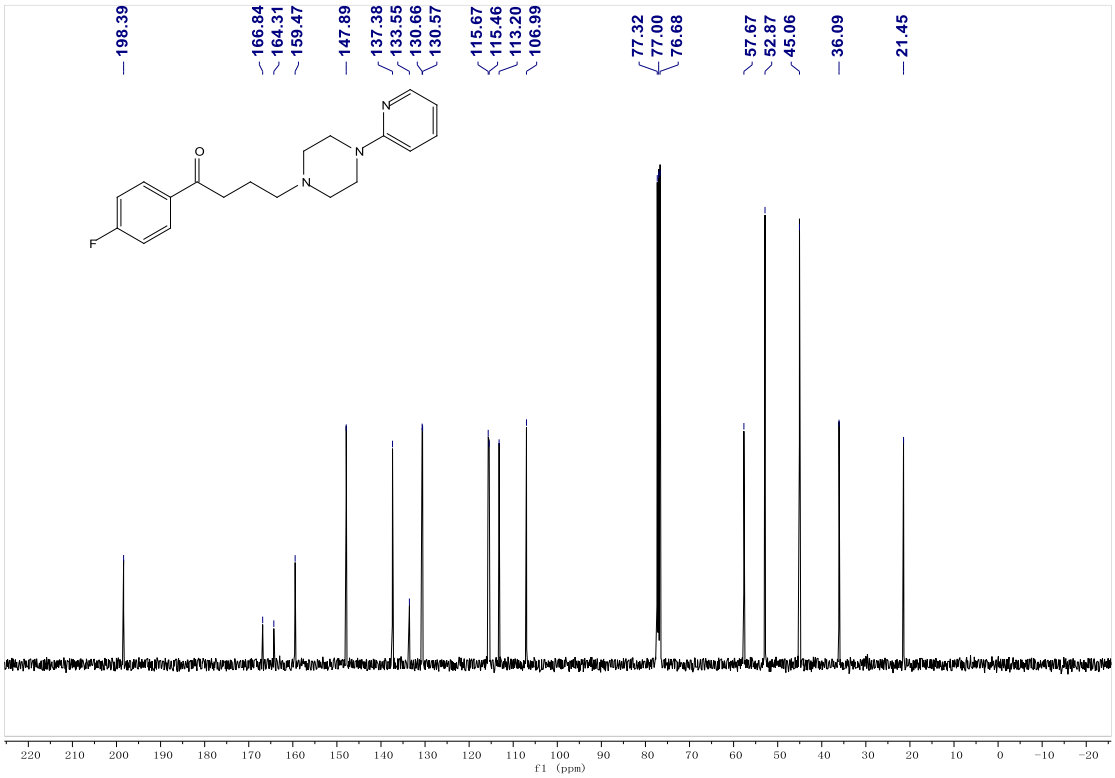
^{19}F NMR (376 MHz, $CDCl_3$) of **10c**



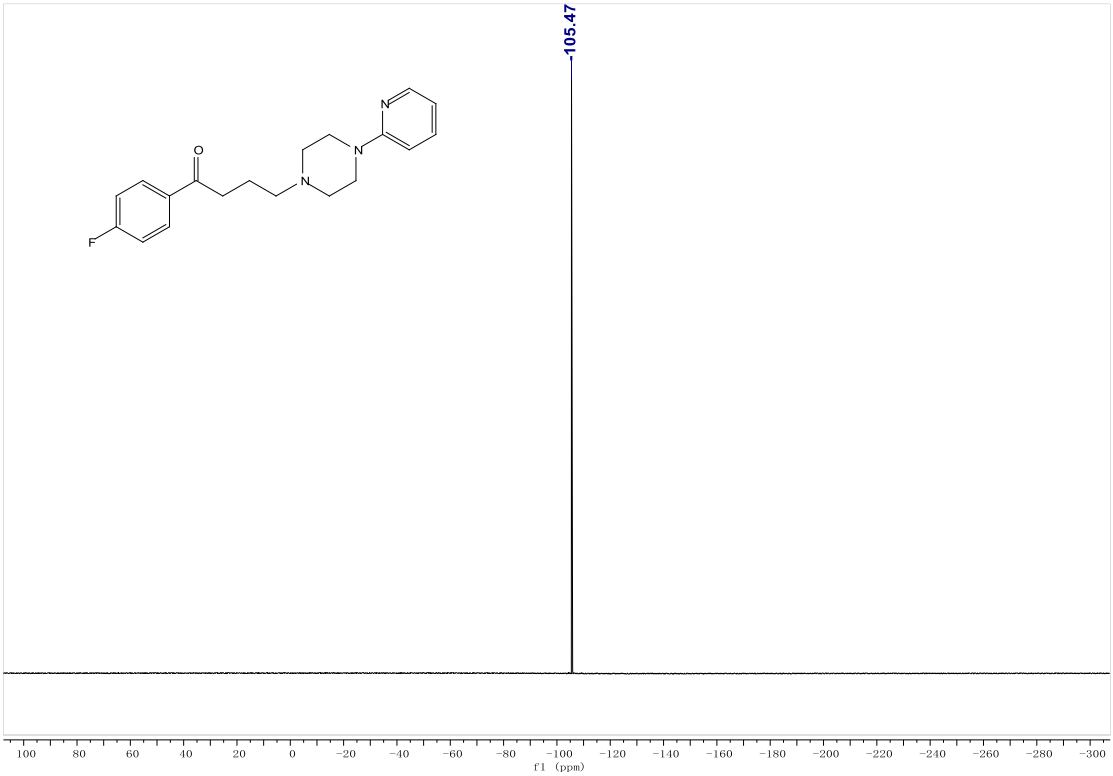
^{1}H NMR (400 MHz, $CDCl_3$) of **10d**



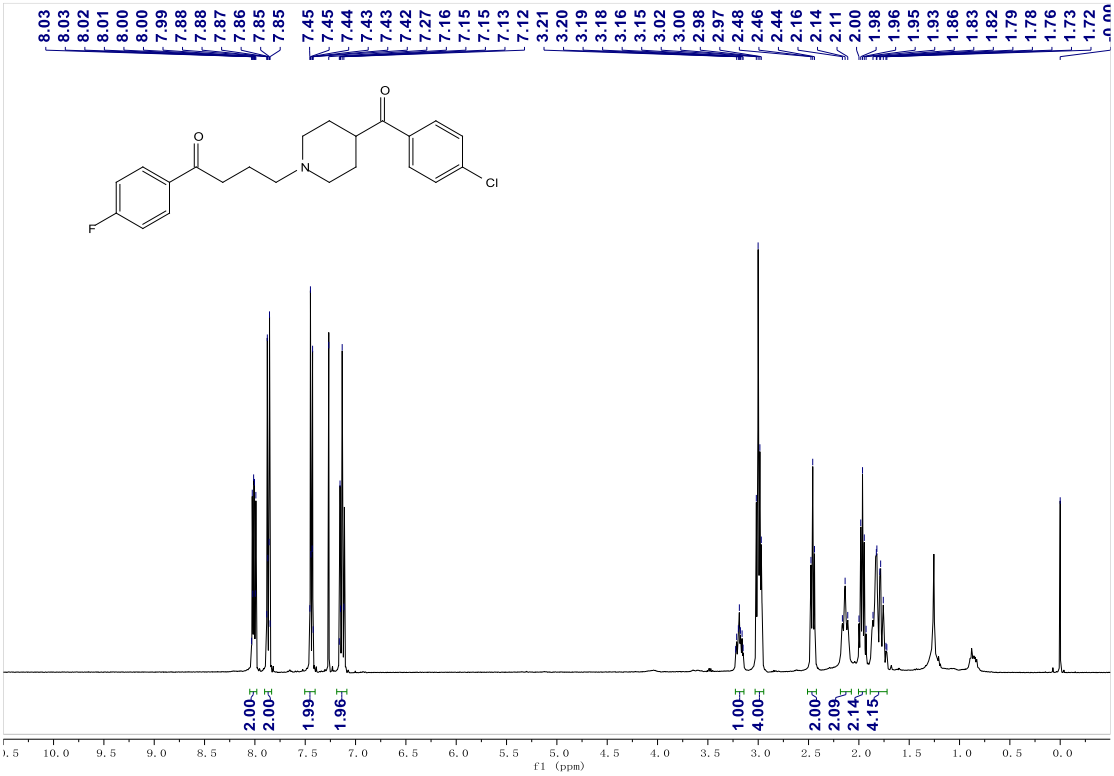
^{13}C NMR (101 MHz, $CDCl_3$) of **10d**



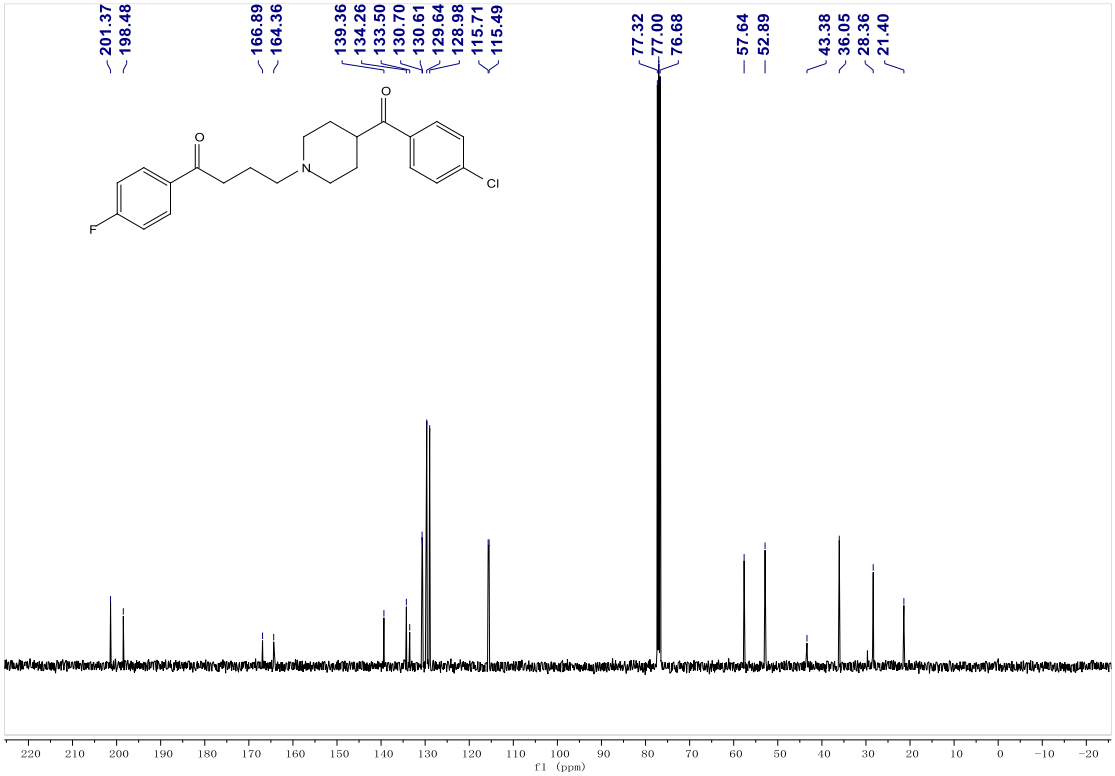
^{19}F NMR (376 MHz, $CDCl_3$) of **10d**



^{1}H NMR (400 MHz, $CDCl_3$) of **10e**



^{13}C NMR (101 MHz, $CDCl_3$) of **10e**



^{19}F NMR (376 MHz, $CDCl_3$) of **10e**