Catalyst Free and High Selective Electrophilic Mono-fluorination of Acetoacetamides: Facile and Efficient Preparation of 2-Fluoro Acetoacetamides in PEG-400

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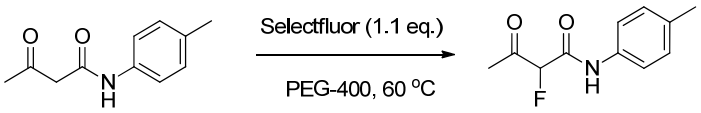
I. General Remarks:

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance/400 (^{1}H: 400 MHz, ^{13}C: 100 MHz at 25 ºC) and TMS as internal standard. Data are represented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = doublet, dd = double doublet, t = triplet, q = quartet, h = hepetet, m = multiplet), coupling constants in Hertz (Hz). The mass spectra (EI) were measured on a Waters LC-MS system. Elemental analysis was performed on a Perkin-Elmer-2400 CHN elemental analyzer, the C, H and N analysis were repeated twice. Melting points were measured on a YuHua X-5 melting point measurement instrument from YuHua Instrument Co., Ltd. Microwave irradiation experiments were carried out using the XH-100 B optimizer microwave (an internal thermometer can be insert into the reaction system to control the temperature) from XiangHu from Xiang Hu Technology Co., Ltd, and the power range was up to 800 W. IR spectra were recorded on a Bio-Rad FTS-40 FT-IR spectrophotometer as KBr pellets. All reactions were monitored by TLC with GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.

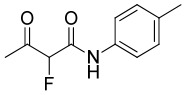
II. Typical Procedure:

(I) For 2 (2j as an example):



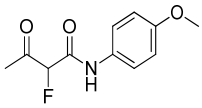
The mixture of 3-oxo-*N*-*p*-tolylbutanamide **1j** (191 mg, 1.0 mmol), selectfluor (390 mg, 1.1 mmol) was well stirred for 8 h in PEG-400 (3.0 mL) at 60 °C oil bath, then to the mixture was added water (10 mL), extracted with diethyl ether (15 mL×4). The solvent was removed under reduced pressure, and the residue was purified by a short flash silica gel column chromatography to give compound **2j** (199 mg, 95%) (Eluent: petroleum ether/ethyl acetate = 5/1).

III. Analytical data of compounds **2**



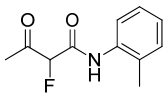
2-fluoro-3-oxo-*N*-(*p*-tolyl)butanamide (**2j**)

Light yellow solid; mp: 55-57 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (s, 1H), 7.40 (d, J = 8 Hz, 2H), 7.12 (d, J = 8 Hz, 2H), 5.40 (d, J = 49.2 Hz, 1H), 2.47-2.40 (m, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.58 (d, J = 19.2 Hz), 161.21 (d, J = 19.2 Hz), 135.15, 133.70, 129.62, 120.35, 92.94 (d, J = 200.8 Hz), 26.62, 20.91; MS: calcd *m/z* 209.1, found 210.1 $[(M+1)]^{+}$; IR (KBr, neat): *v* 3338, 1735, 1683, 1541, 1516, 1095, 811; Anal. Calcd for $C_{11}H_{12}FNO_2$: C, 63.15; H, 5.78; N, 6.69. Found: C, 63.22; H, 5.70; N, 6.74.



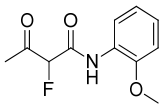
2-fluoro-*N*-(4-methoxyphenyl)-3-oxobutanamide (2k)

Yellow solid; mp: 57-59 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (s, 1H), 7.36 (d, J = 9.2 Hz, 2H), 6.77 (d, J = 8.8 Hz, 2H), 5.35 (d, J = 49.2 Hz, 1H), 3.69 (s, 3H), 2.40-2.33 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.68 (d, J = 20 Hz), 161.25 (d, J = 19.6 Hz), 157.05, 129.29, 122.16, 114.14, 92.92 (d, J = 200.5 Hz), 55.37, 26.49; MS: calcd *m/z* 225.1, found 226.1 $[(M+1)]^{+}$; IR (KBr, neat): *v* 3353, 1730, 1672, 1604, 1560, 1412, 824; Anal. Calcd for $C_{11}H_{12}FNO_3$: C, 58.66; H, 5.37; N, 6.22. Found: C, 58.58; H, 5.42; N, 6.26.



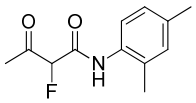
2-fluoro-3-oxo-*N*-(*o*-tolyl)butanamide (2l)

Yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (s, 1H), 7.60 (d, J = 8 Hz, 1H), 7.11 (m, 2H), 7.06 (d, J = 8 Hz, 1H), 5.35 (d, J = 48.8 Hz, 1H), 2.39-2.31 (m, 3H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.87 (d, J = 20.3 Hz),161.43 (d, J = 19.7 Hz), 133.83, 130.52, 130.08, 126.56, 126.08, 123.22, 92.76 (d, J = 199.7 Hz), 26.34, 17.25; MS: calcd *m/z* 209.1, found 210.1 $[(M+1)]^{+}$; IR (KBr, neat): *v* 3324, 1739, 1685, 1538, 1459, 1360, 754; Anal. Calcd for $C_{11}H_{12}FNO_2$: C, 63.15; H, 5.78; N, 6.69. Found: C, 63.28; H, 5.84; N, 6.77.



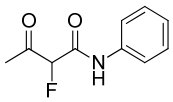
2-fluoro-*N*-(2-methoxyphenyl)-3-oxobutanamide (2m)

Yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.64 (s, 1H), 8.26 (d, J = 8 Hz, 1H), 7.06 (t, J = 7.6 Hz, 1H), 6.92 (t, J = 7.6 Hz, 1H), 6.86 (d, J = 8 Hz, 1H), 5.37 (d, J = 49.2 Hz, 1H), 3.84 (s, 3H), 2.39-2.46 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.06 (d, J = 19.9 Hz), 160.83 (d, J = 19.1 Hz), 148.27, 125.96, 124.98, 120.89, 119.82, 110.15, 92.92 (d, J = 201.3 Hz), 55.71, 26.45; MS: calcd *m/z* 225.1, found 226.1 $[(M+1)]^{+}$; IR (KBr, neat): *v* 3410, 1740, 1695, 1549, 1463, 1255, 1118, 752; Anal. Calcd for $C_{11}H_{12}FNO_3$: C, 58.66; H, 5.37; N, 6.22. Found: C, 58.74; H, 5.39; N, 6.30.



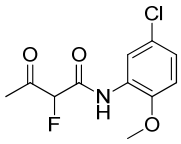
_N_-(2,4-dimethylphenyl)-2-fluoro-3-oxobutanamide (2n)

White solid; mp: 53-54 °C ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (s, 1H), 7.56 (d, J = 8.8 Hz, 1H), 7.00 (d, J = 5.6 Hz, 2H), 5.42 (d, J = 49.2 Hz, 1H), 2.49-2.42 (m, 3H), 2.28 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.67 (d, J = 20.2 Hz), 161.40 (d, J = 19.6 Hz), 136.08, 131.41, 131.31, 129.94, 127.42, 123.27, 93.03 (d, J = 200.5 Hz), 26.67, 20.92, 17.44; MS: calcd m/z 223.1, found 224.1 $[(M+1)]^+$; IR (KBr, neat): ν 3307, 1598, 1540, 1506, 1140, 1115, 875, 808; Anal. Calcd for $C_{12}H_{14}FNO_2$: C, 64.56; H, 6.32; N, 6.27. Found: C, 64.69; H, 6.38; N, 6.20.



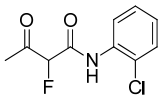
2-fluoro-3-oxo-_N_-phenylbutanamide (2o)

Yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (s, 1H), 7.53 (d, J = 8 Hz, 2H), 7.34 (t, J = 8 Hz, 2H),7.17 (t, J = 7.2 Hz, 1H), 5.41 (d, J = 49.2 Hz, 1H), 2.49-2.42 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.38 (d, J = 19.7 Hz) 161.28 (d, J = 16.0, Hz), 136.27, 129.25, 125.54, 120.32, 92.94 (d, J = 201.2 Hz), 26.71; MS: calcd m/z 195.1, found 196.1 $[(M+1)]^+$; IR (KBr, neat): ν 3330, 1738, 1684, 1601, 1541, 1446, 755, 692; Anal. Calcd for $C_{10}H_{10}FNO_2$: C, 61.53; H, 5.16; N, 7.18. Found: C, 61.43; H, 5.20; N, 7.15.



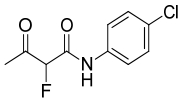
_N_-(5-chloro-2-methoxyphenyl)-2-fluoro-3-oxobutanamide (2p)

Yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (s, 1H), 8.31 (d, J = 2.4 Hz, 1H), 7.01 (dd, J_1 = 8.8 Hz, J_2 = 2 Hz, 1H), 6.76 (d, J = 8.8 Hz, 1H), 5.39 (d, J = 49.2 Hz, 1H), 3.85 (s, 3H), 2.48-2.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.95 (d, J = 20.0 Hz), 161.00 (d, J = 19.4 Hz), 146.83, 126.80, 125.90, 124.50, 119.79, 110.99, 92.82 (d, J = 201.6 Hz), 56.11, 26.62; MS: calcd m/z 259.0, found 260.0 $[(M+1)]^+$; IR (KBr, neat): ν 3408, 1740, 1698, 1598, 1533, 1485, 1254, 804, 645; Anal. Calcd for $C_{11}H_{11}ClFNO_3$: C, 50.88; H, 4.27; N, 5.39. Found: C, 50.97; H, 4.22; N, 5.40.



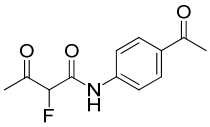
_N_-(2-chlorophenyl)-2-fluoro-3-oxobutanamide (2q)

Yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (s, 1H), 8.27 (d, J = 8.4 Hz, 1H), 7.38 (d, J = 8.4 Hz, 1H), 7.27 (t, J = 7.6 Hz, 1H), 7.10 (t, J = 7.2 Hz, 1H), 5.44 (d, J = 48.8 Hz, 1H), 2.43-2.51 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.10 (d, J = 20.3 Hz), 161.14 (d, J = 19.6 Hz), 133.01, 129.24, 127.73, 125.82, 123.65, 121.71, 92.70 (d, J = 201.1 Hz), 26.50; MS: calcd m/z 229.0, found 230.0 $[(M+1)]^+$; IR (KBr, neat): ν 3401, 1699, 1596, 1548, 1444, 1361, 753; Anal. Calcd for $C_{10}H_9ClFNO_2$: C, 52.30; H, 3.95; N, 6.10. Found: C, 52.35; H, 4.00; N, 6.12.



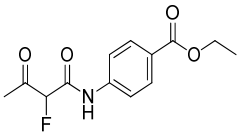
***N*-(4-chlorophenyl)-2-fluoro-3-oxobutanamide (2r)**

Yellow solid; mp: 56-57 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (s, 1H), 7.40 (d, J = 8.8 Hz, 2H), 7.18 (d, J = 8.8 Hz, 2H), 5.34 (d, J = 49.2 Hz, 1H), 2.39-2.32 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.56 (d, J = 20 Hz), 161.37 (d, J = 19.8 Hz), 134.83, 130.44, 129.08, 121.60, 92.79(d, J = 200.8 Hz), 26.55; MS: calcd *m/z* 229.0, found 230.0 $[(M+1)]^+$; IR (KBr, neat): ν 3325, 1738, 1683, 1670, 1558, 1493, 820; Anal. Calcd for $C_{10}H_9ClFNO_2$: C, 52.30; H, 3.95; N, 6.10. Found: C, 52.33; H, 3.89; N, 6.15.



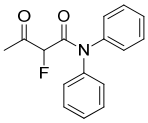
***N*-(4-acetylphenyl)-2-fluoro-3-oxobutanamide (2s)**

White solid; mp: 106-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54 (s, 1H), 7.94-7.92 (dd, J_1 = 8.8 Hz, J_2 = 2.4 Hz, 2H), 7.68-7.66 (d, J = 8.8 Hz, 2H), 5.51-5.39 (d, J = 48.8 Hz, 1H), 2.57 (s, 3H), 2.49-2.43 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.39 (d, J = 20.0 Hz), 197.18, 161.55 (d, J = 19.7 Hz), 140.64, 133.79, 129.80, 119.61, 92.81 (d, J = 201.2 Hz), 26.79, 26.58; IR (KBr, neat): ν 3324, 1736, 1690, 1674, 1556, 1493, 824; Anal. Calcd for $C_{12}H_{12}FNO_3$: C, 60.76; H, 5.10; N, 5.90. Found: C, 60.75; H, 5.05; N, 5.88.



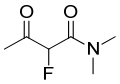
Ethyl 4-(2-fluoro-3-oxobutanamido)benzoate (2t)

Colourless liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (s, 1H), 8.03-8.01 (d, J = 8.8 Hz, 2H), 7.68-7.66 (d, J = 8.8 Hz, 2H), 5.54-5.42 (d, J = 48.8 Hz, 1H), 4.39-4.34 (q, J = 7.2 Hz, 2H), 2.50-2.45 (m, 3H), 1.41-1.38 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.45 (d, J = 19.5 Hz), 166.01, 161.53 (d, J = 19.7 Hz), 140.39, 130.80, 126.99, 119.46, 92.82 (d, J = 201.3 Hz), 61.11, 26.73, 14.32; IR (KBr, neat): ν 3326, 1748, 1693, 1530, 1526, 1428, 834; Anal. Calcd for $C_{13}H_{14}FNO_4$: C, 58.42; H, 5.28; N, 5.24. Found: C, 58.45; H, 5.36; N, 5.19.



2-fluoro-3-oxo-*N*,*N*-diphenylbutanamide (2g)

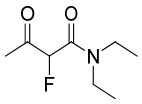
Yellow solid; mp: 97-98 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38-7.26 (m, 10H), 5.27 (d, J = 49.6 Hz, 1H), 2.30 (d, J = 4.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.22 (d, J = 26.1 Hz), 164.48 (d, J = 21.1 Hz), 141.62, 141.02, 130.07, 129.25, 129.00, 128.85, 127.10, 126.17, 89.91 (d, J = 191.4 Hz), 26.71; MS: calcd *m/z* 271.1, found 272.1 $[(M+1)]^+$; IR (KBr, neat): ν 1693, 1492, 1354, 1094, 700; Anal. Calcd for $C_{16}H_{14}FNO_2$: C, 70.84; H, 5.20; N, 5.16. Found: C, 70.80; H, 5.26; N, 5.20.



2-fluoro-*N*,*N*-dimethyl-3-oxobutanamide (2h)

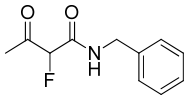
Light yellow liquid; 1H NMR (400 MHz, CDCl3): δ 5.50 (d, J = 49.6 Hz, 1H), 3.04 (s, 3H), 2.92 (s, 3H), 2.24 (d, J = 4 Hz); 13C NMR (100 MHz, CDCl3): δ 201.73 (d, J = 24.1 Hz), 163.49 (d, J = 20.4 Hz), 90.99 (d, J = 192.9 Hz), 36.75, 36.71, 35.70, 25.86; MS: calcd m/z 147.1, found

148.1[(M+1)]+; IR (KBr, neat): v 2962, 2876, 1718, 1653, 1261, 1110, 803; Anal. Calcd for $C_6H_{10}FNO_2$: C, 48.97; H, 6.85; N, 9.52. Found: C, 49.01; H, 6.86; N, 9.55.



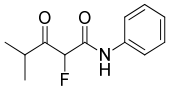
_N,N_-diethyl-2-fluoro-3-oxobutanamide (2i)

Light yellow solid; mp: 92-94 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.32 (d, J = 50.0 Hz, 1H), 3.35-3.17 (m, 4H), 2.17-2.16 (m, 3H), 1.06 (t, J = 6.8 Hz, 3H), 0.98 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.87 (t, J = 23.9 Hz), 162.98 (t, J = 20.1 Hz), 91.33 (d, J = 193.6 Hz), 41.59, 40.52, 14.00, 12.30; MS: calcd *m/z* 175.1, found 176.1 $[(M+1)]^+$; IR (KBr, neat): v 2978, 2940, 1740, 1642, 1464, 1360, 1084; Anal. Calcd for $C_8H_{14}FNO_2$: C, 54.84; H, 8.05; N, 7.99. Found: C, 54.80; H, 8.11; N, 8.02.



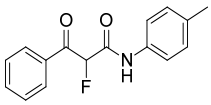
_N_-benzyl-2-fluoro-3-oxobutanamide (2u)

Colourless liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.24 (m, 5H), 6.87 (s, 1H), 5.31 (d, J = 49.2 Hz, 1H), 4.51-4.40 (m, 2H), 2.45-2.37 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.33 (d, J = 19.4 Hz), 163.32 (d, J = 19.8 Hz), 137.06, 128.85, 127.87, 127.79, 92.96 (d, J = 199.6 Hz), 43.37, 26.62; MS: calcd *m/z* 209.1, found 210.1 $[(M+1)]^+$; IR (KBr, neat): v 3338, 1736, 1684, 1539, 1455, 1360, 1093, 700; Anal. Calcd for $C_{11}H_{12}FNO_2$: C, 63.15; H, 5.78; N, 6.69. Found: C, 63.20; H, 5.84; N, 6.72.



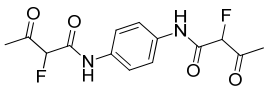
2-fluoro-4-methyl-3-oxo-_N_-phenylpentanamide (2v)

Light yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (s, 1H), 7.53 (d, J = 7.6 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 7.16 (t, J = 7.6 Hz, 1H), 5.53 (d, J = 49.2 Hz, 1H), 3.26 (h, J = 6.8 Hz, 1H), 1.20 (d, J = 7.2 Hz, 3H), 1.13 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.86 (d, J = 18.6 Hz), 161.56 (d, J = 19.7 Hz), 136.40, 129.22, 125.41, 120.27, 91.52 (d, J = 200.7 Hz), 37.36, 18.42, 17.23; MS: calcd *m/z* 223.1, found 224.1 $[(M+1)]^+$; IR (KBr, neat): v 3323, 2976, 1733, 1684, 1541, 1446, 755, 692; Anal. Calcd for $C_{12}H_{14}FNO_2$: C, 64.56; H, 6.32; N, 6.27. Found: C, 64.62; H, 6.28; N, 6.24.



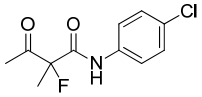
2-fluoro-3-oxo-3-phenyl-_N_-(_p_-tolyl)propanamide (2w)

Yellow solid; mp: 91-93 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (s, 1H), 8.14 (d, J = 7.6 Hz, 2H), 7.59 (t, J = 7.6 Hz, 1H), 7.46 (t, J = 8 Hz, 2H), 7.42 (d, J = 8 Hz, 2H), 7.09 (d, J = 8.4 Hz, 2H), 6.25 (d, J = 48.8 Hz, 1H),2.33-2.29 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.59 (d, J = 17.7 Hz),161.88 (d, J = 19.3 Hz), 135.01, 134.65, 133.83, 133.62, 129.92, 129.56, 128.74, 120.27, 90.45 (d, J = 198.6 Hz), 20.88; MS: calcd *m/z* 271.1, found 272.1 $[(M+1)]^+$; IR (KBr, neat): v 3346, 1698, 1657, 1530, 1107, 731, 684; Anal. Calcd for $C_{16}H_{14}FNO_2$: C, 70.84; H, 5.20; N, 5.16. Found: C, 70.82; H, 5.24; N, 5.11.



***N,N'*-(1,4-phenylene)bis(2-fluoro-3-oxobutanamide) (2x)**

Light yellow solid; mp: 185-187 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.50 (s, 2H), 7.66 (d, J = 12.8 Hz, 4H), 5.69 (d, J = 48.4 Hz, 2H), 2.48-2.32 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.43 (d, J = 20.9 Hz), 162.15 (d, J = 21.0 Hz), 134.12, 120.70, 92.92 (d, J = 193.6 Hz), 26.45; MS: calcd *m/z* 312.1, found 313.1 $[(M+1)]^+$; IR (KBr, neat): v 3323, 1732, 1683, 1568, 1515, 1407, 1095, 824; Anal. Calcd for $C_{14}H_{14}F_2N_2O_4$: C, 53.85; H, 4.52; N, 8.97. Found: C, 53.93; H, 4.56; N, 9.01.

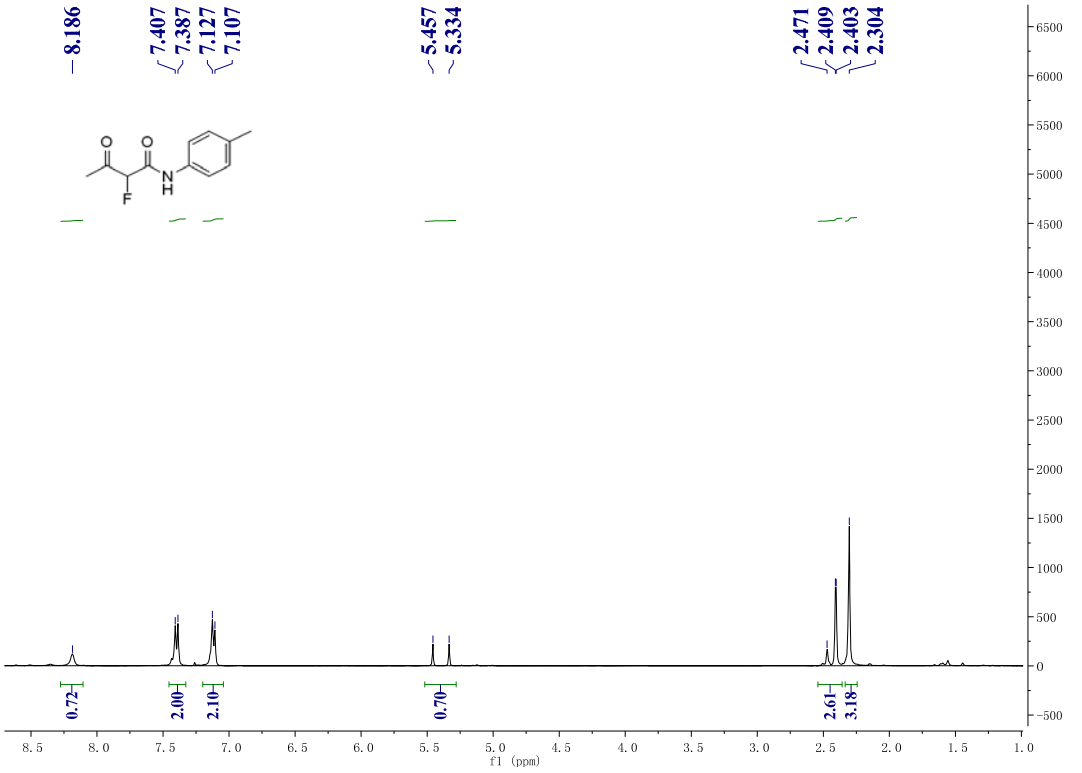


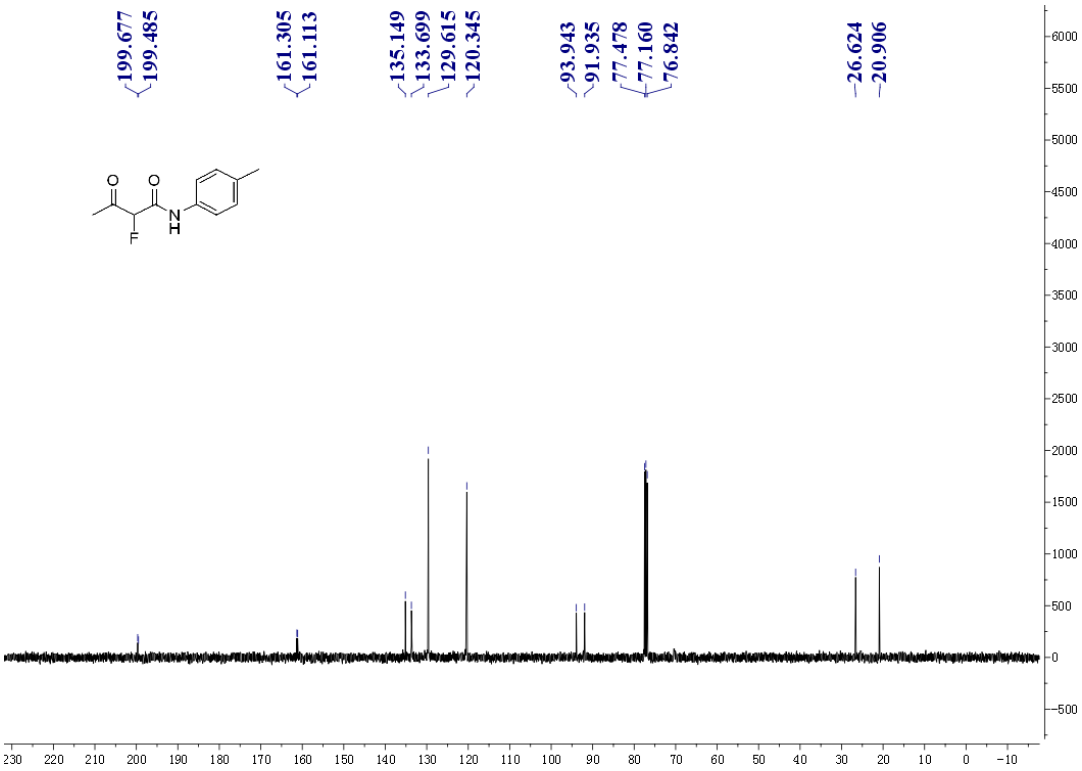
***N*-(4-chlorophenyl)-2-fluoro-2-methyl-3-oxobutanamide (2y)**

Light yellow solid; mp: 105-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36 (s, 1H), 7.53 (d, J = 8.8 Hz, 2H), 7.30 (d, J = 8.8 Hz, 2H), 2.36 (d, J = 3.2 Hz, 3H), 1.81 (d, J = 22.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.09 (d, J = 24.5 Hz), 164.91 (d, J = 21.0 Hz), 135.15, 130.29, 129.13, 121.47, 99.56 (d, J = 196.4 Hz), 25.20, 20.84 (d, J = 22.7 Hz); MS: calcd *m/z* 243.0, found 244.0 $[(M+1)]^+$; IR (KBr, neat): v 3330, 1739, 1683, 1653, 1541, 1495, 1403, 833; Anal. Calcd for $C_{11}H_{11}ClFNO_2$: C, 54.22; H, 4.55; N, 5.75. Found: C, 54.22; H, 4.49; N, 5.77.

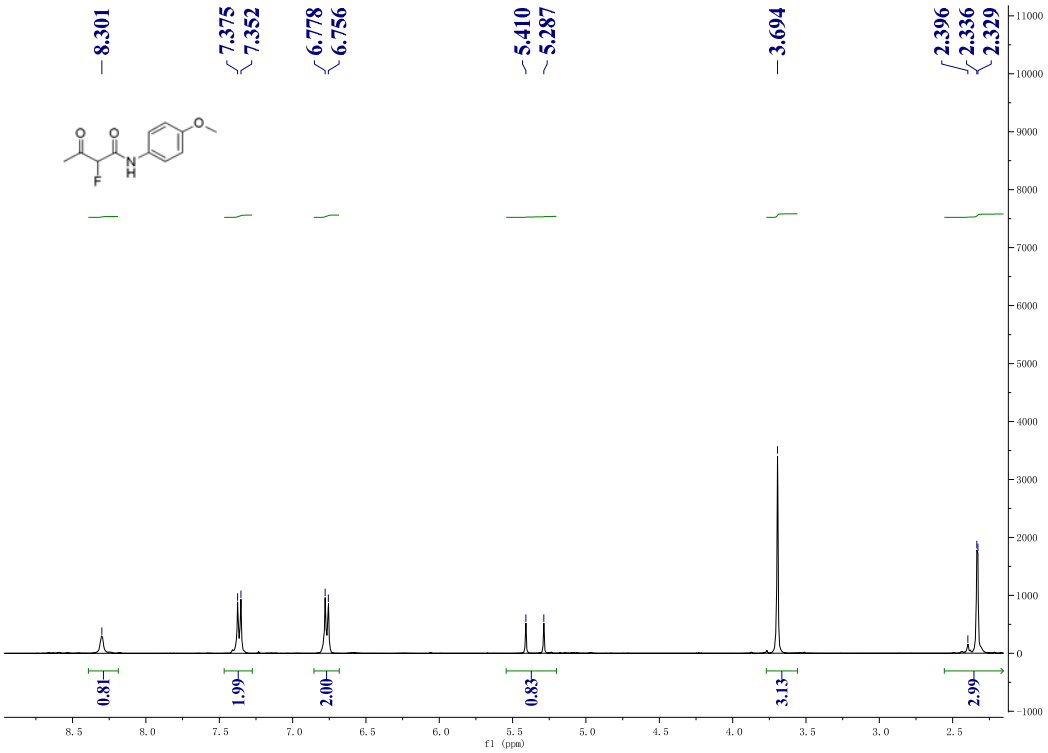
IV. ^{1}H NMR and ^{13}C NMR spectra copies

Compound **2j**





Compound **2k**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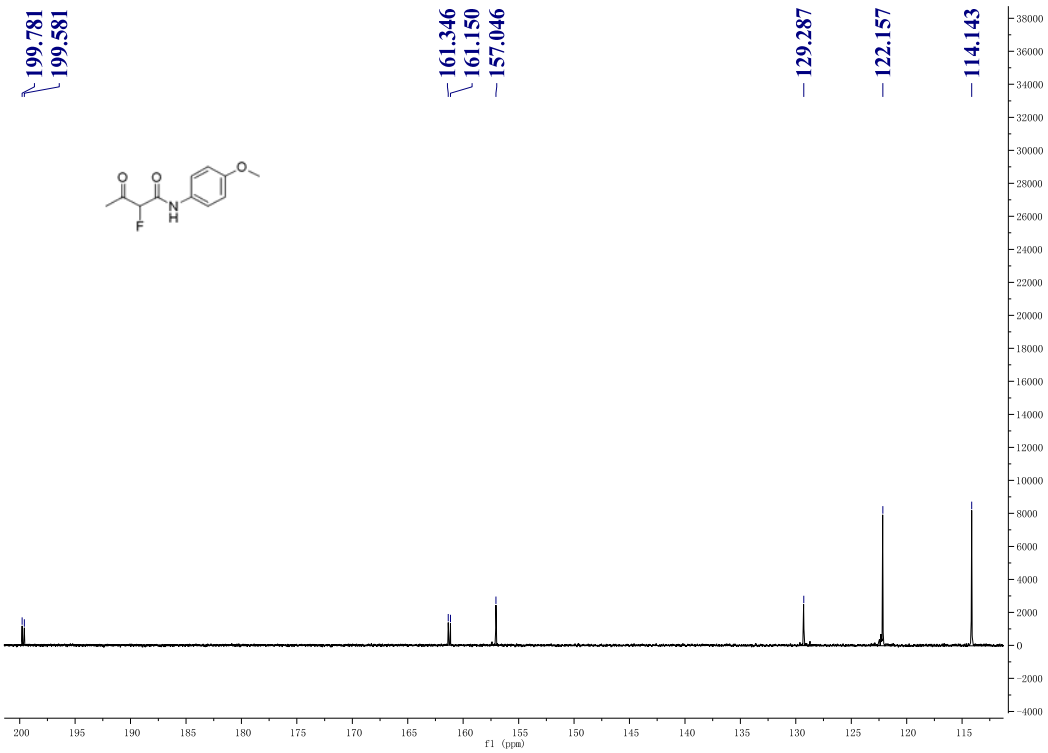
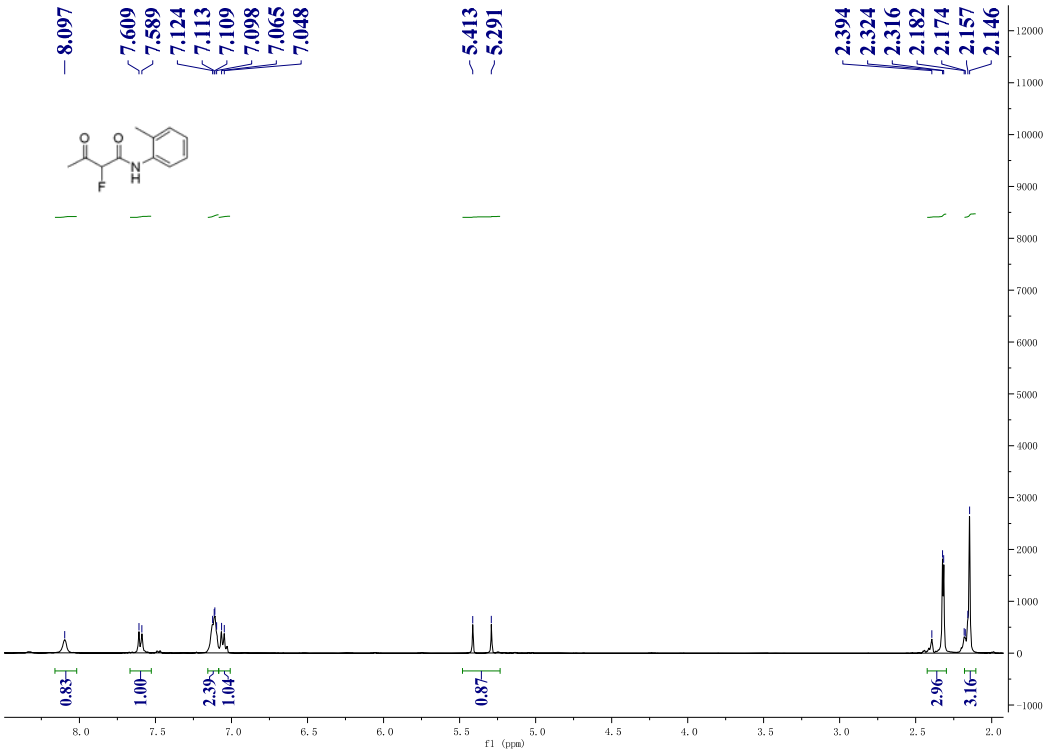
7.375
7.352
6.778
6.756
5.410
5.287
3.694
2.396
2.336
2.329
0.81
1.99
2.00
0.83
3.13
2.99
f1 (ppm)


199.781
199.581
161.346
161.150
157.046
129.287
122.157
114.143
f1 (ppm)

Compound **2l**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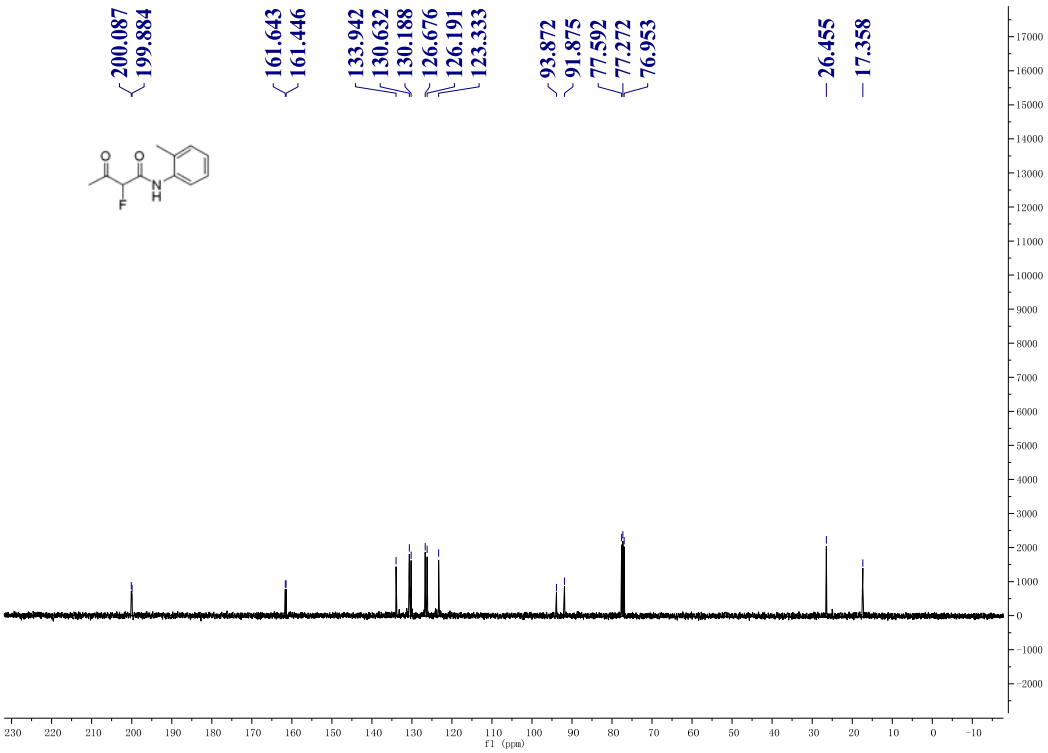
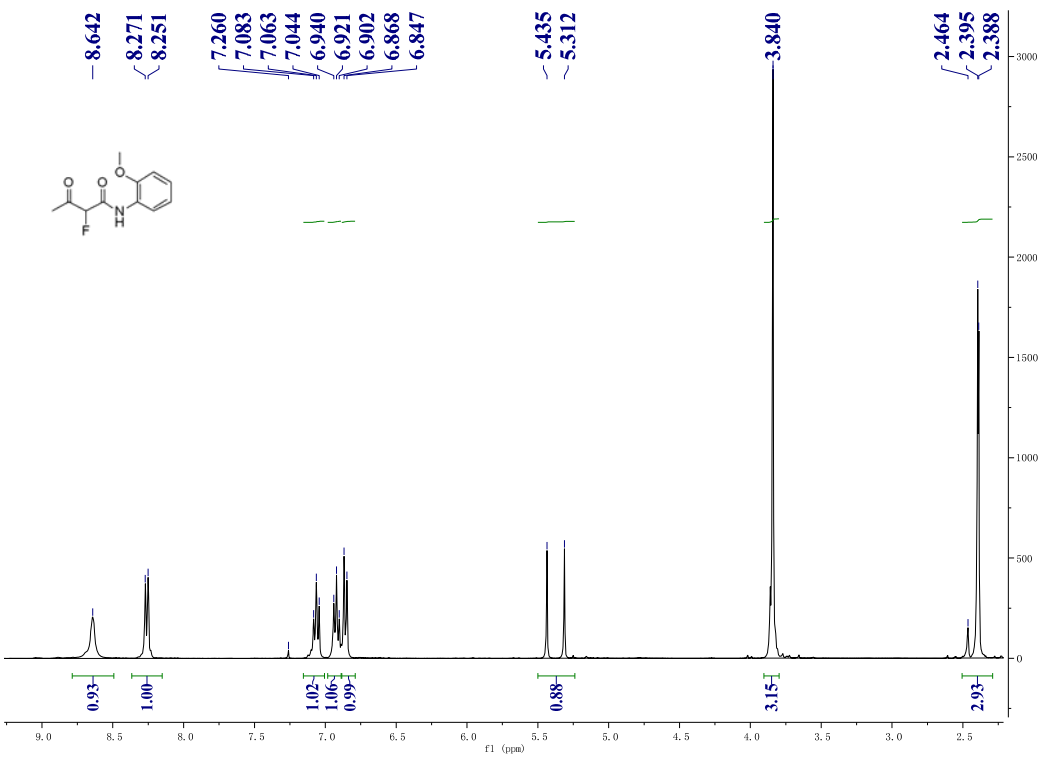
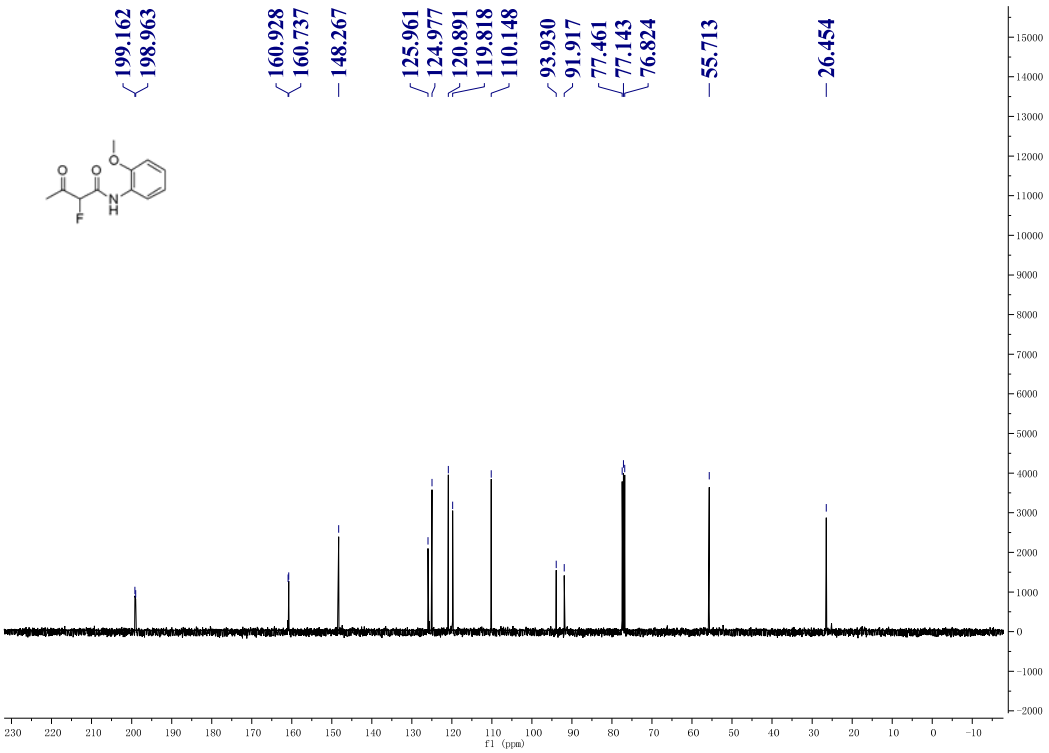
7.609
7.589
7.124
7.113
7.109
7.098
7.065
7.048
5.413
5.291
2.394
2.324
2.316
2.182
2.174
2.157
2.146
0.83
1.00
2.39
1.04
0.87
2.96
3.16
f1 (ppm)

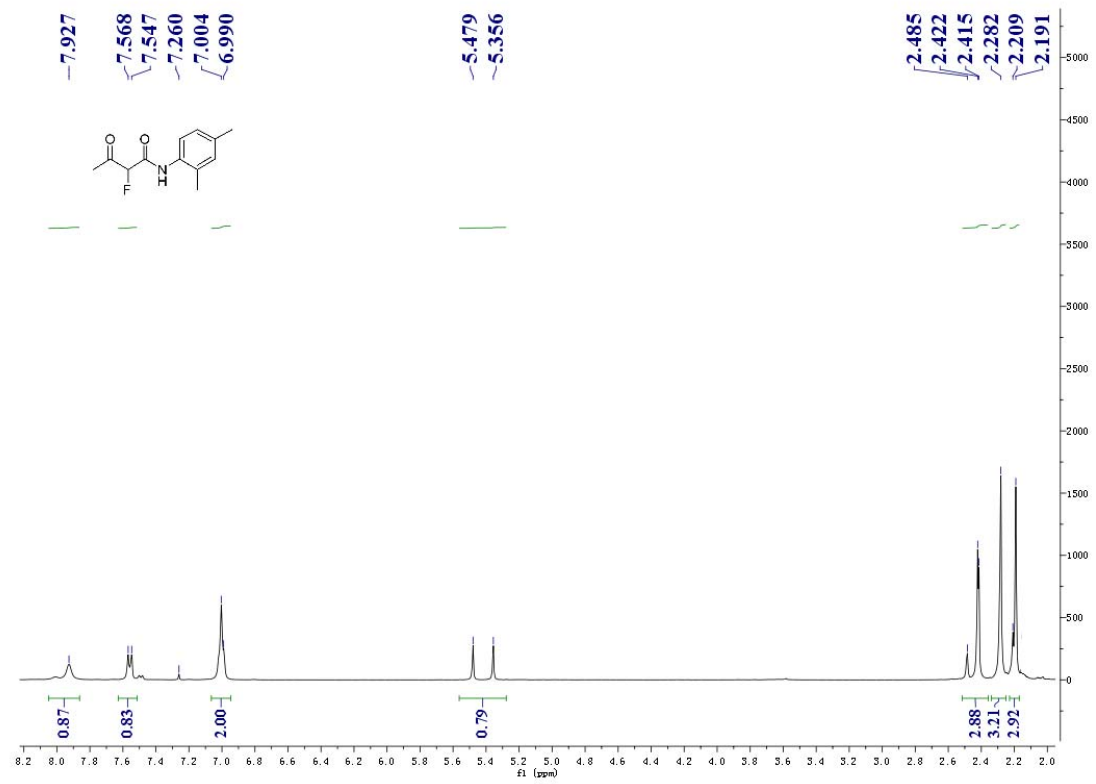

200.087
199.884
161.643
161.446
133.942
130.632
130.188
126.676
126.191
123.333
93.872
91.875
77.592
77.272
76.953
26.455
17.358
f1 (ppm)

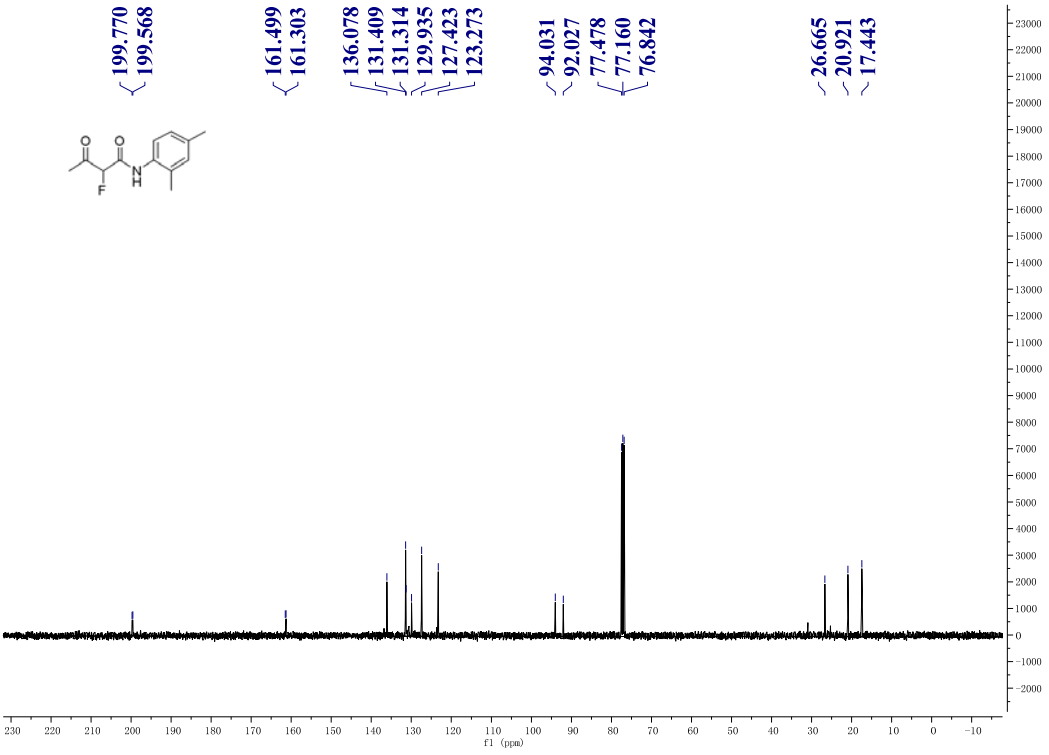
Compound **2m**


8.642
8.271
8.251
7.260
7.083
7.063
7.044
6.940
6.921
6.902
6.868
6.847
5.435
5.312
3.840
2.464
2.395
2.388
0.93
1.00
1.02
1.06
0.99
0.88
3.15
2.93
f1 (ppm)

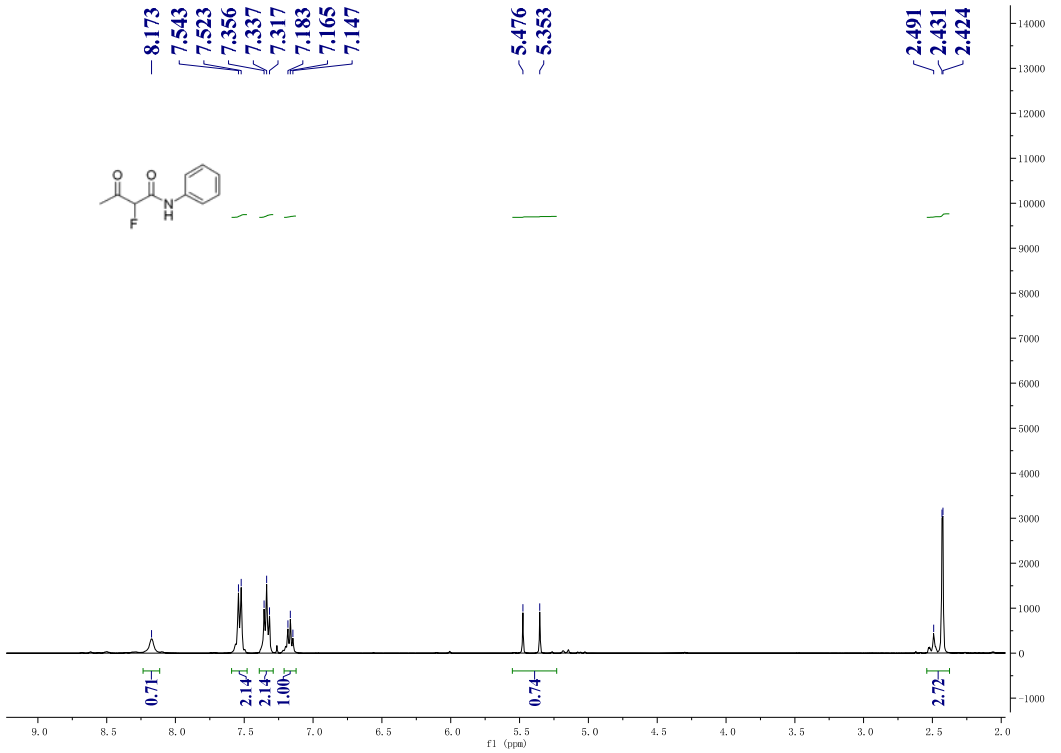

199.162
198.963
160.928
160.737
148.267
125.961
124.977
120.891
119.818
110.148
93.930
91.917
77.461
77.143
76.824
55.713
26.454
f1 (ppm)

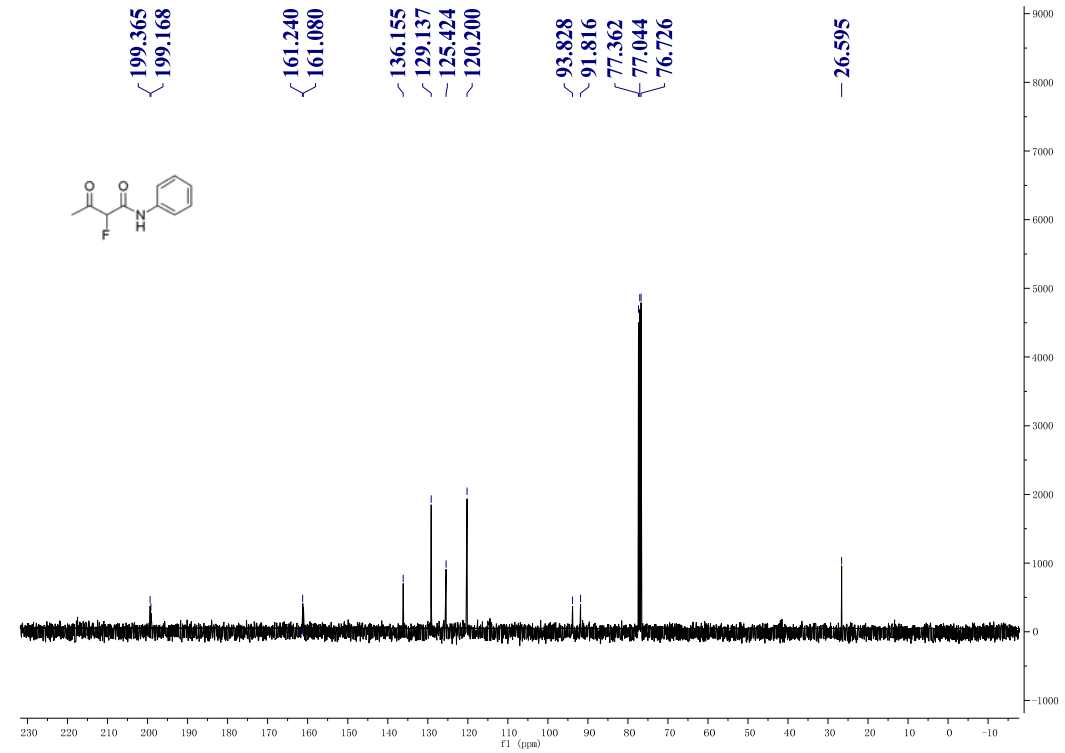
Compound **2n**


7.927
7.568
7.547
7.260
7.004
6.990
5.479
5.356
2.485
2.422
2.415
2.282
2.209
2.191
0.87
0.83
2.00
0.79
2.88
3.21
2.92
f1 (ppm)

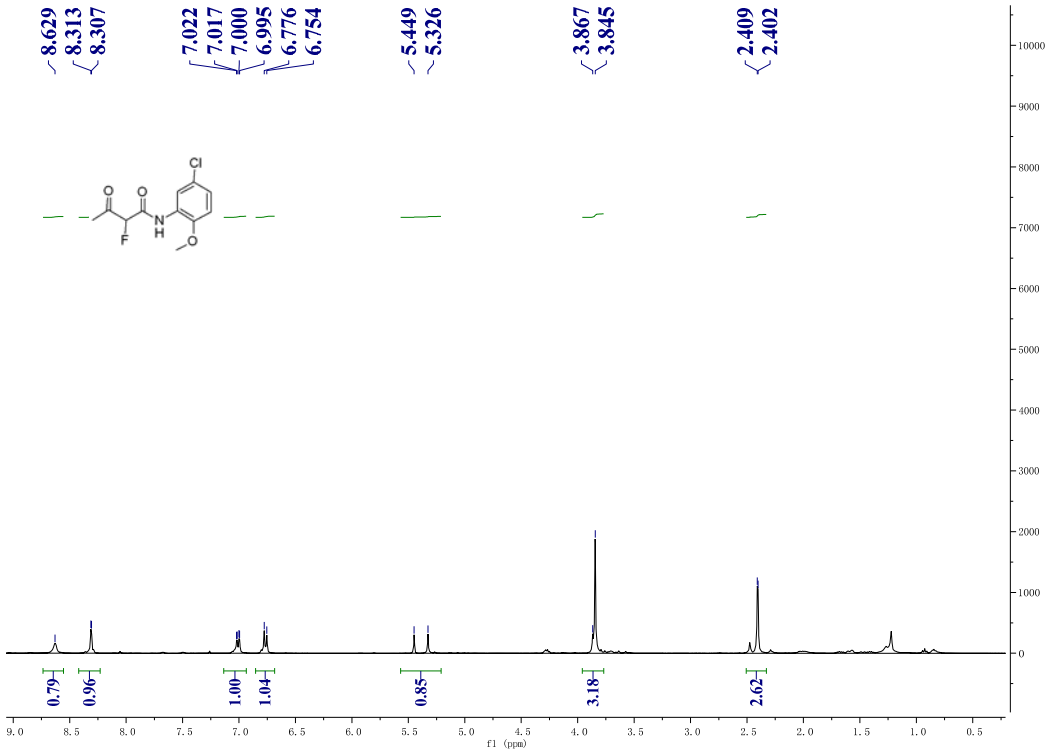

199.770
199.568
161.499
161.303
136.078
131.409
131.314
129.935
127.423
123.273
94.031
92.027
77.478
77.160
76.842
26.665
20.921
17.443
f1 (ppm)

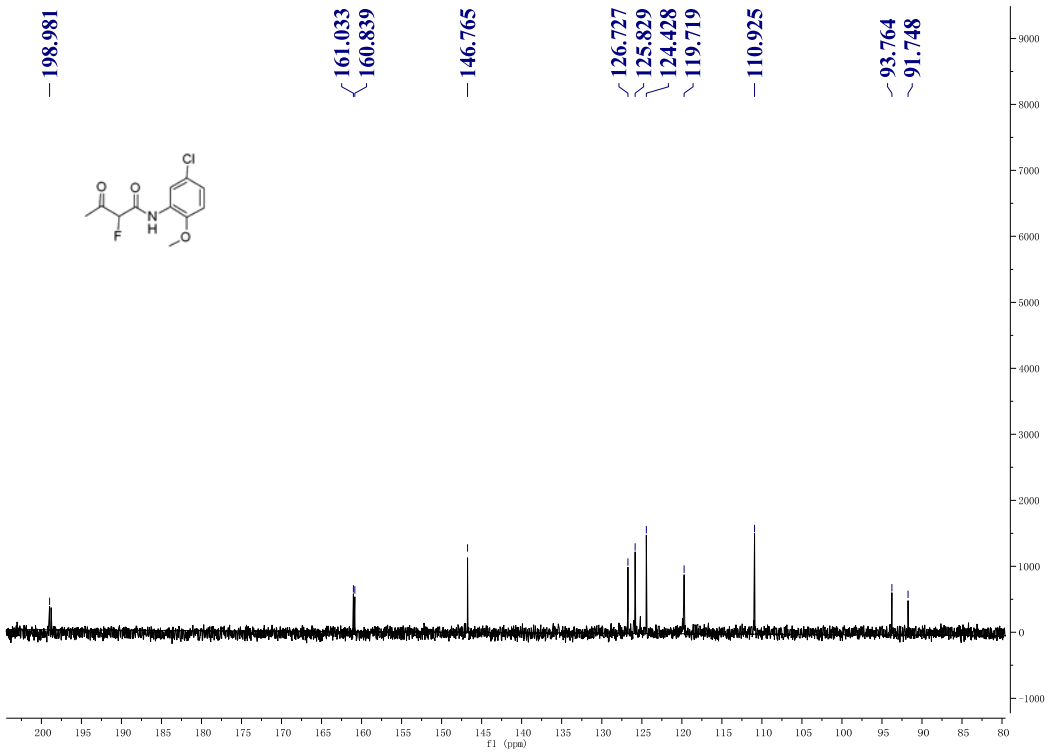
Compound **2o**


8.173
7.543
7.523
7.356
7.337
7.317
7.183
7.165
7.147
5.476
5.353
2.491
2.431
2.424
0.71
2.14
2.14
1.00
0.74
2.72
f1 (ppm)

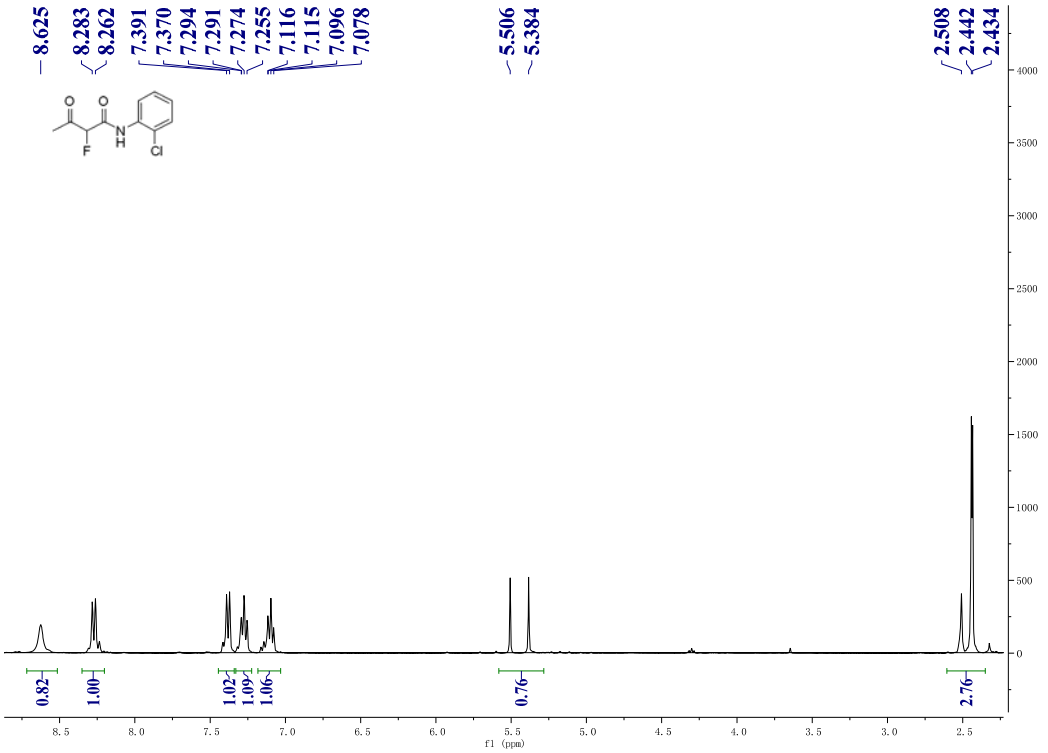

199.365
199.168
161.240
161.080
136.155
129.137
125.424
120.200
93.828
91.816
77.362
77.044
76.726
26.595
f1 (ppm)

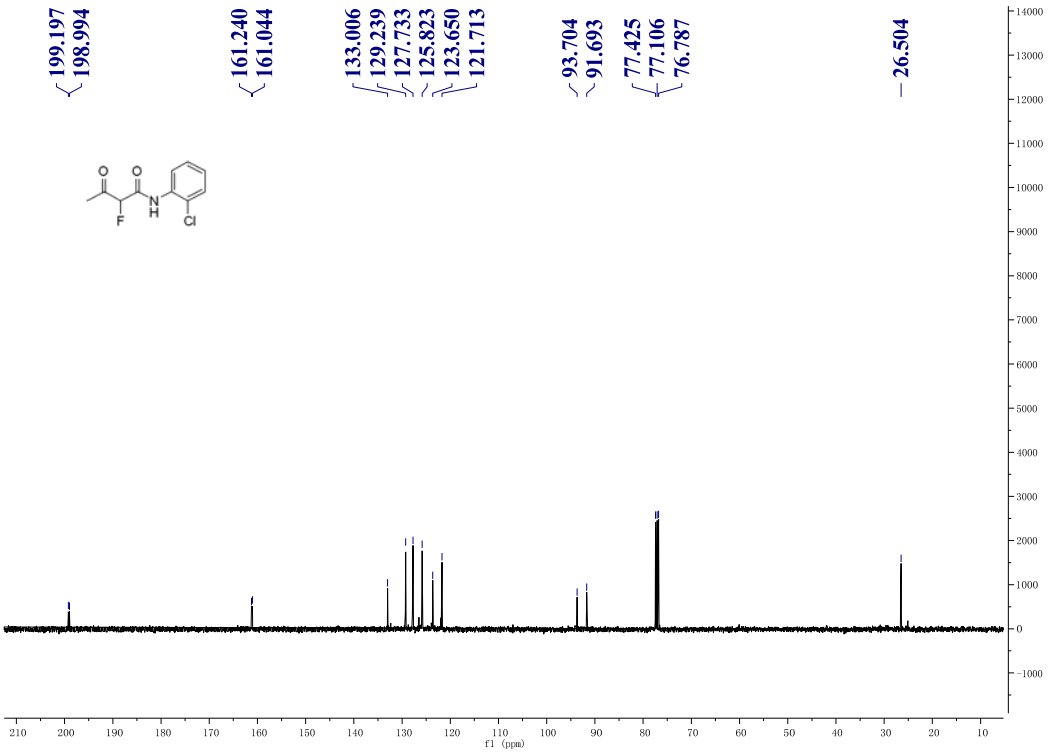
Compound **2p**


8.629
8.313
8.307
7.022
7.017
7.000
6.995
6.776
6.754
5.449
5.326
3.867
3.845
2.409
2.402
0.79
0.96
1.00
1.04
0.85
3.18
2.62
f1 (ppm)

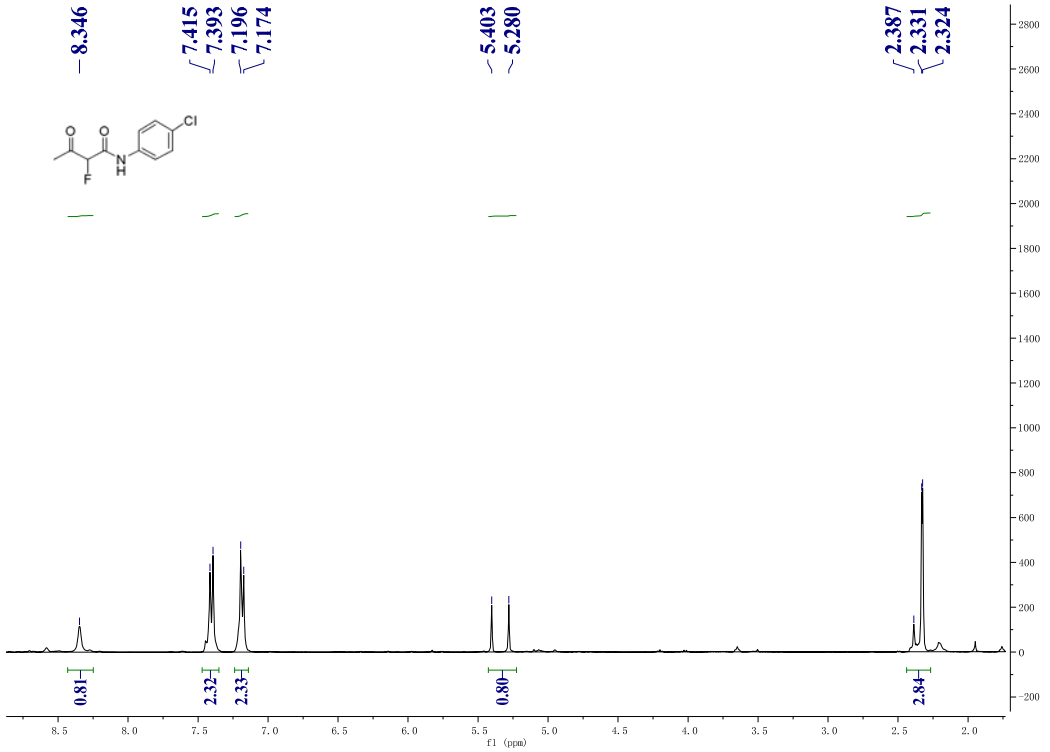

198.981
161.033
160.839
146.765
126.727
125.829
124.428
119.719
110.925
93.764
91.748
f1 (ppm)

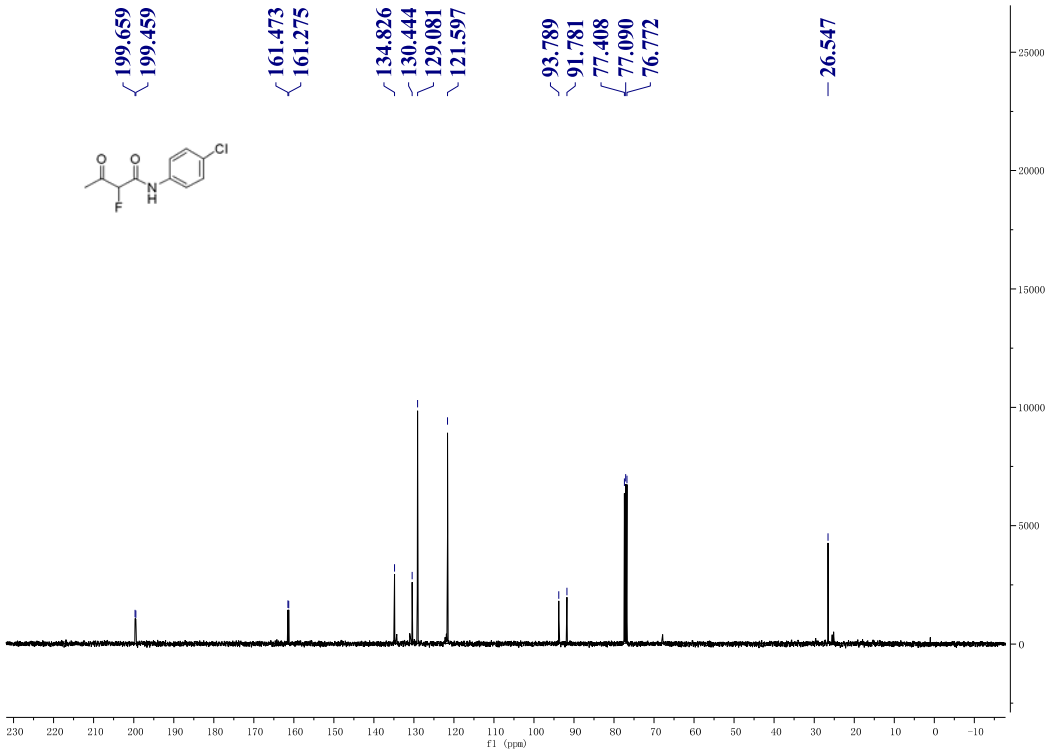
Compound **2q**


8.625
8.283
8.262
7.391
7.370
7.294
7.291
7.274
7.255
7.116
7.115
7.096
7.078
5.506
5.384
2.508
2.442
2.434
0.82
1.00
1.02
1.09
1.06
0.76
2.76
f1 (ppm)

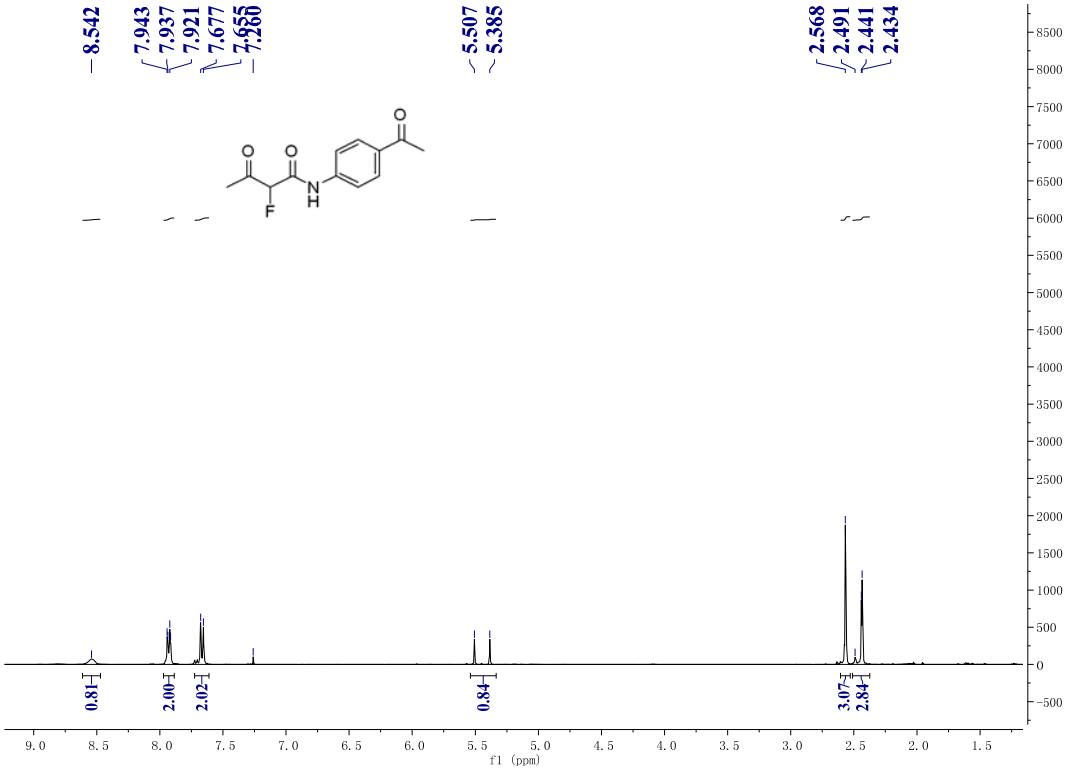

199.197
198.994
161.240
161.044
133.006
129.239
127.733
125.823
123.650
121.713
93.704
91.693
77.425
77.106
76.787
26.504
f1 (ppm)

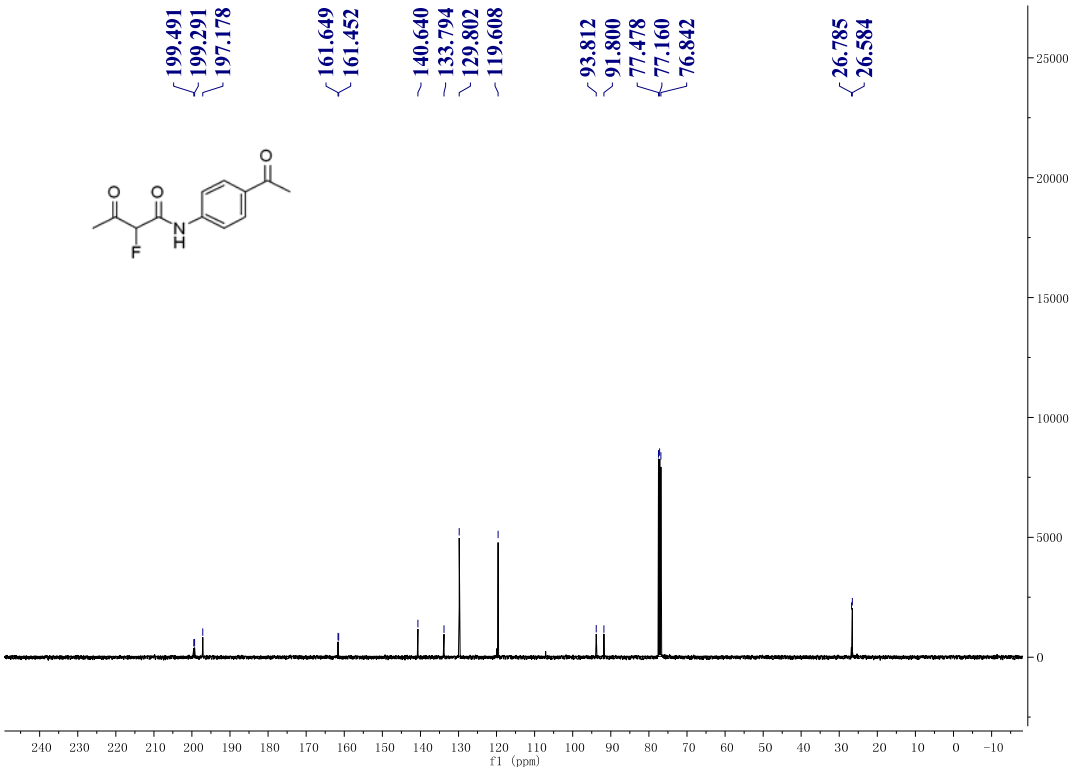
Compound **2r**


8.346
7.415
7.393
7.196
7.174
5.403
5.280
2.387
2.331
2.324
0.81
2.32
2.33
0.80
2.84
f1 (ppm)


199.659
199.459
161.473
161.275
134.826
130.444
129.081
121.597
93.789
91.781
77.408
77.090
76.772
26.547
f1 (ppm)

Compound **2s**


8.542
7.943
7.937
7.921
7.677
7.655
7.260
5.507
5.385
2.568
2.491
2.441
2.434
0.81
2.00
2.02
0.84
3.07
2.84
f1 (ppm)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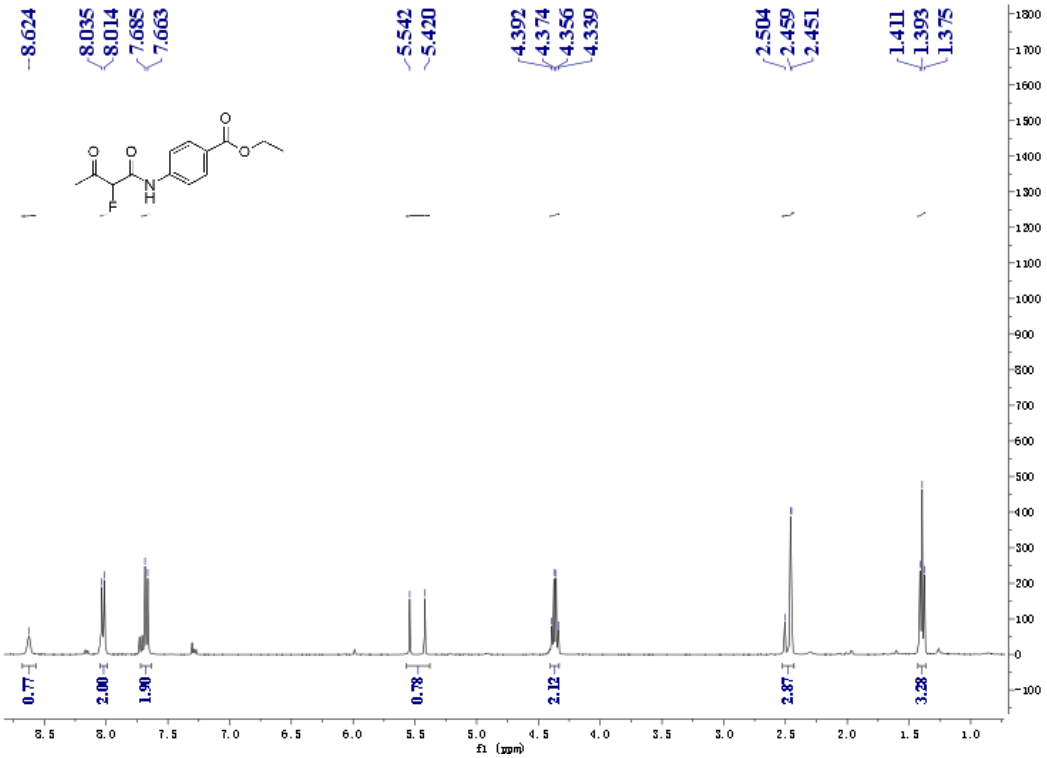
199.291
197.178
161.649
161.452
140.640
133.794
129.802
119.608
93.812
91.800
77.478
77.160
76.842
26.785
26.584
f1 (ppm)

Compound **2t**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8.035
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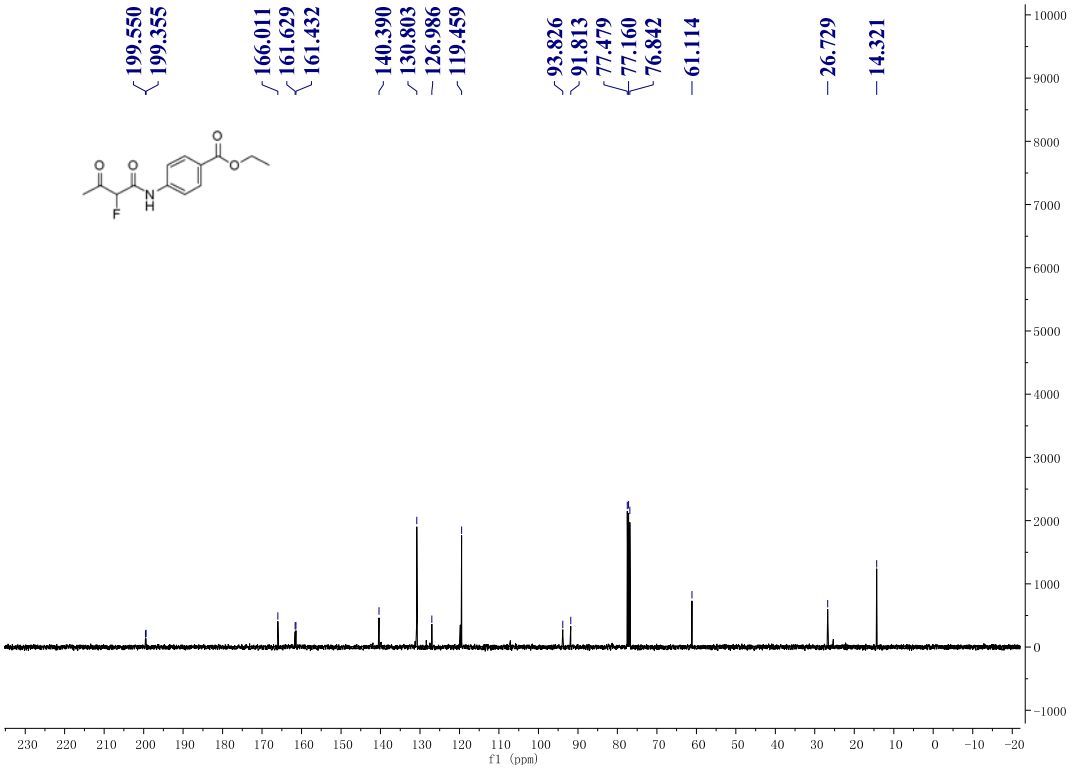
7.663
5.542
5.420
4.392
4.374
4.356
4.339
2.504
2.459
2.451
1.411
1.393
1.375
0.77
2.00
1.90
0.78
2.12
2.87
3.28
f1 (ppm)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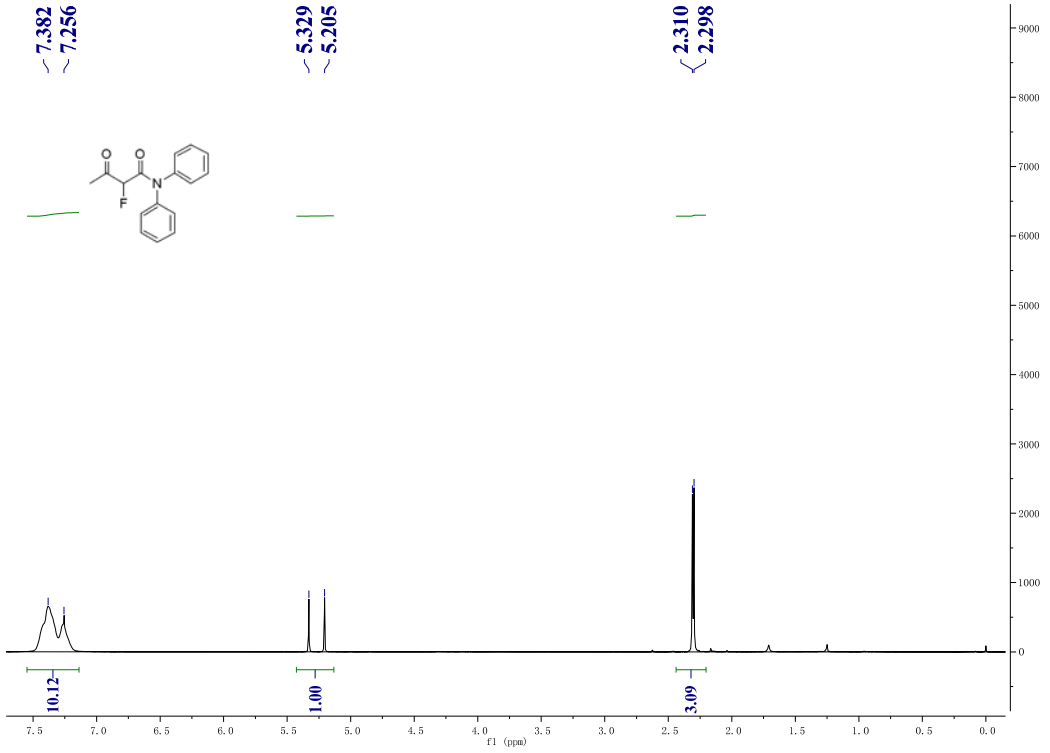
161.629
161.432
140.390
130.803
126.986
119.459
93.826
91.813
77.479
77.160
76.842
61.114
26.729
14.321
f1 (ppm)

Compound **2g**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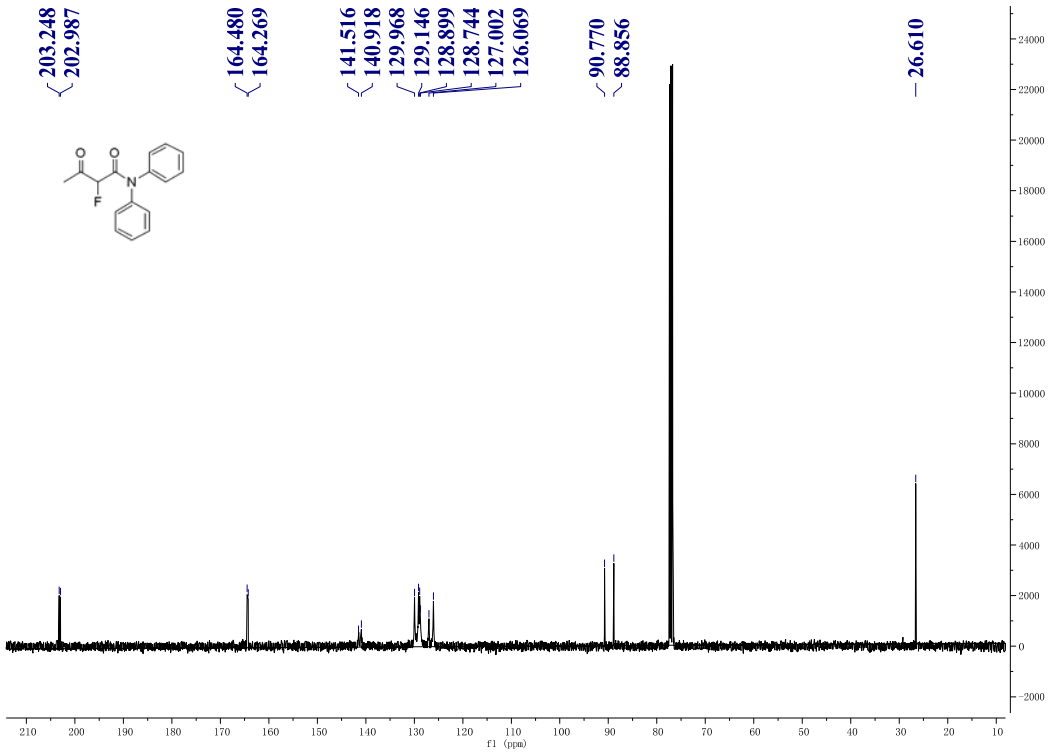
5.329
5.205
2.310
2.298
10.12
1.00
3.09
f1 (ppm)


203.248
202.987
164.480
164.269
141.516
140.918
129.968
129.146
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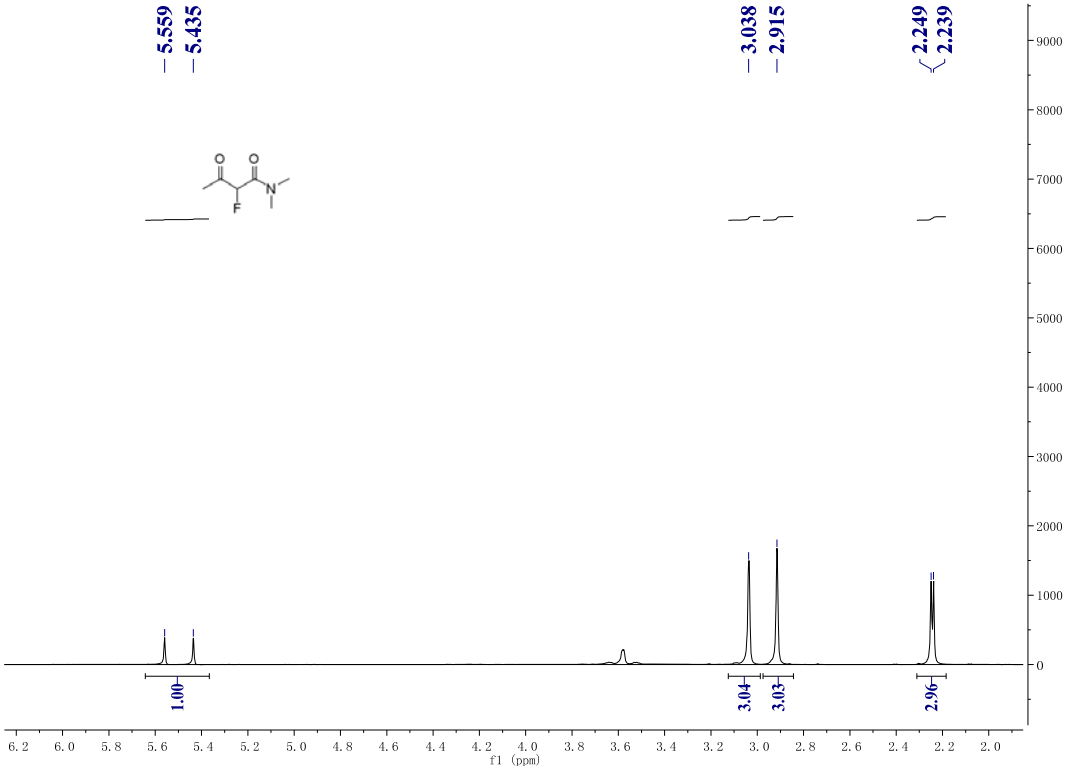
128.744
127.002
126.069
90.770
88.856
26.610
f1 (ppm)

Compound **2h**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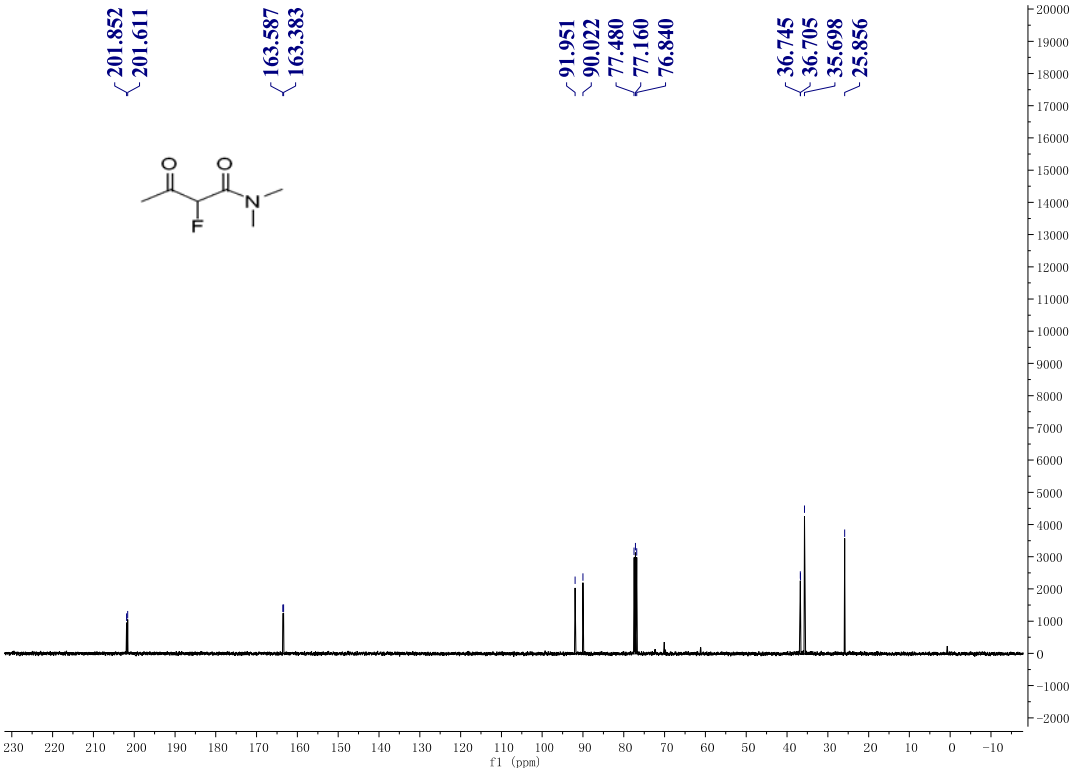
2.249
2.239
1.00
3.04
3.03
2.96
f1 (ppm)


201.852
201.611
163.587
163.383
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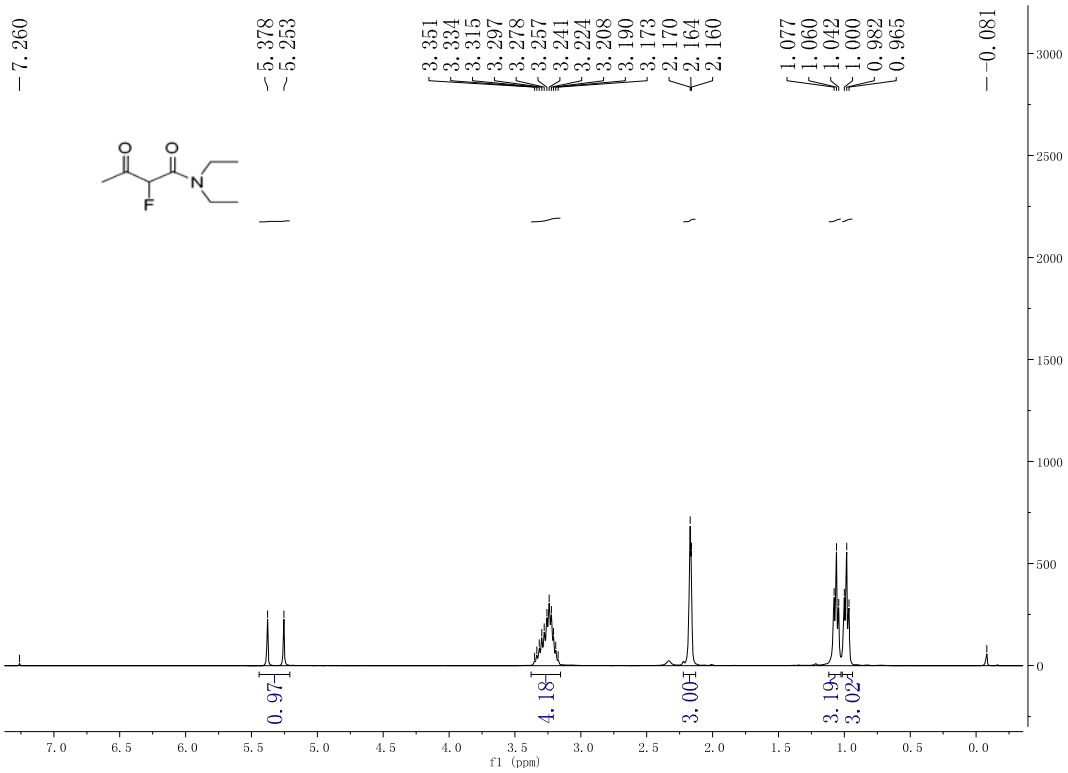
90.022
77.480
77.160
76.840
36.745
36.705
35.698
25.856
f1 (ppm)

Compound **2i**


7.260
5.378
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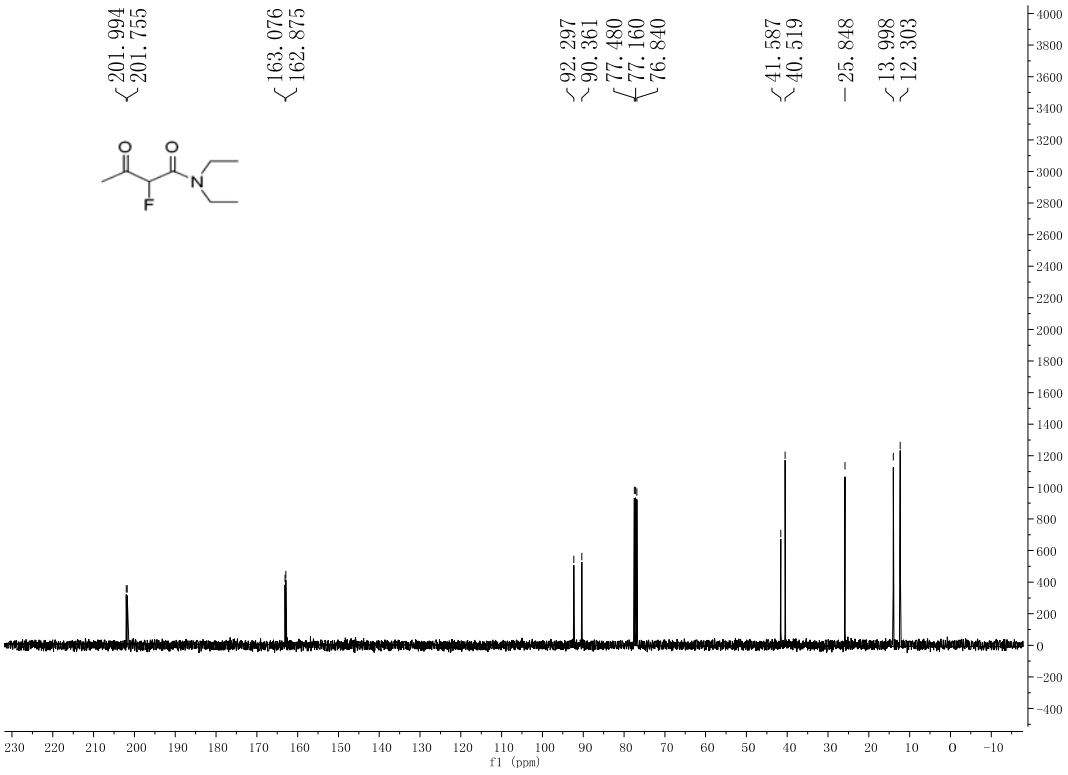
3.351
3.334
3.315
3.297
3.278
3.257
3.241
3.224
3.208
3.190
3.173
2.170
2.164
2.160
1.077
1.060
1.042
1.000
0.982
0.965
0.081
0.97
4.18
3.00
3.19
3.02
f1 (ppm)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162.875
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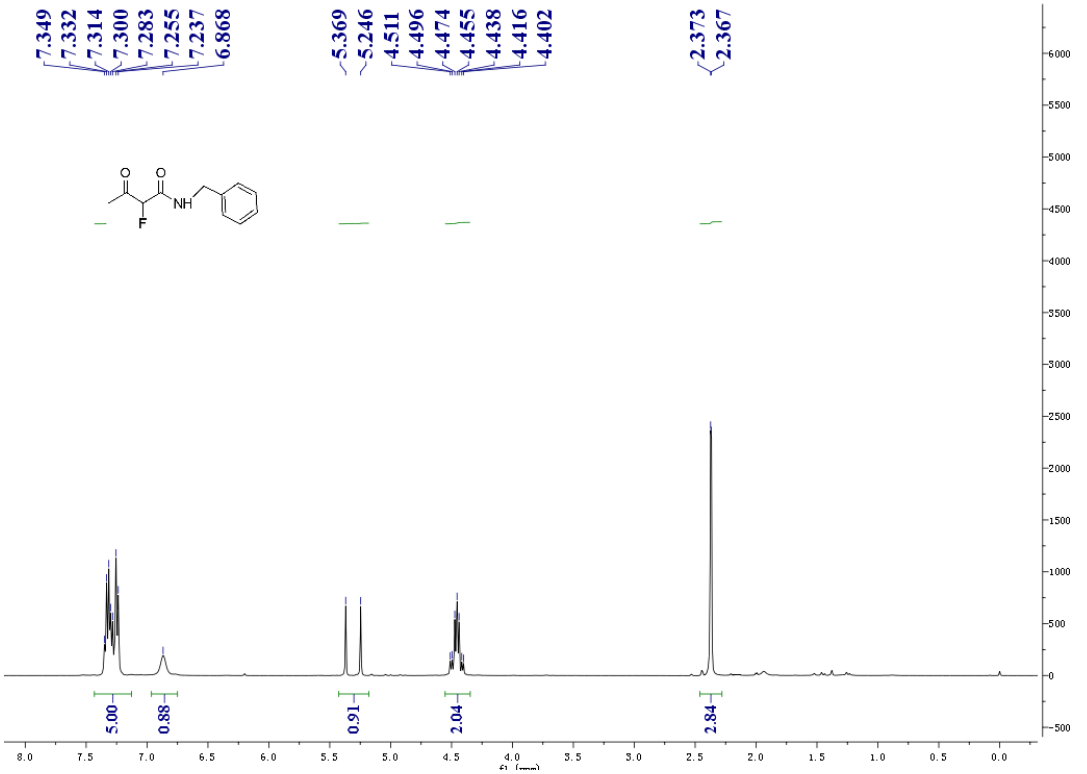
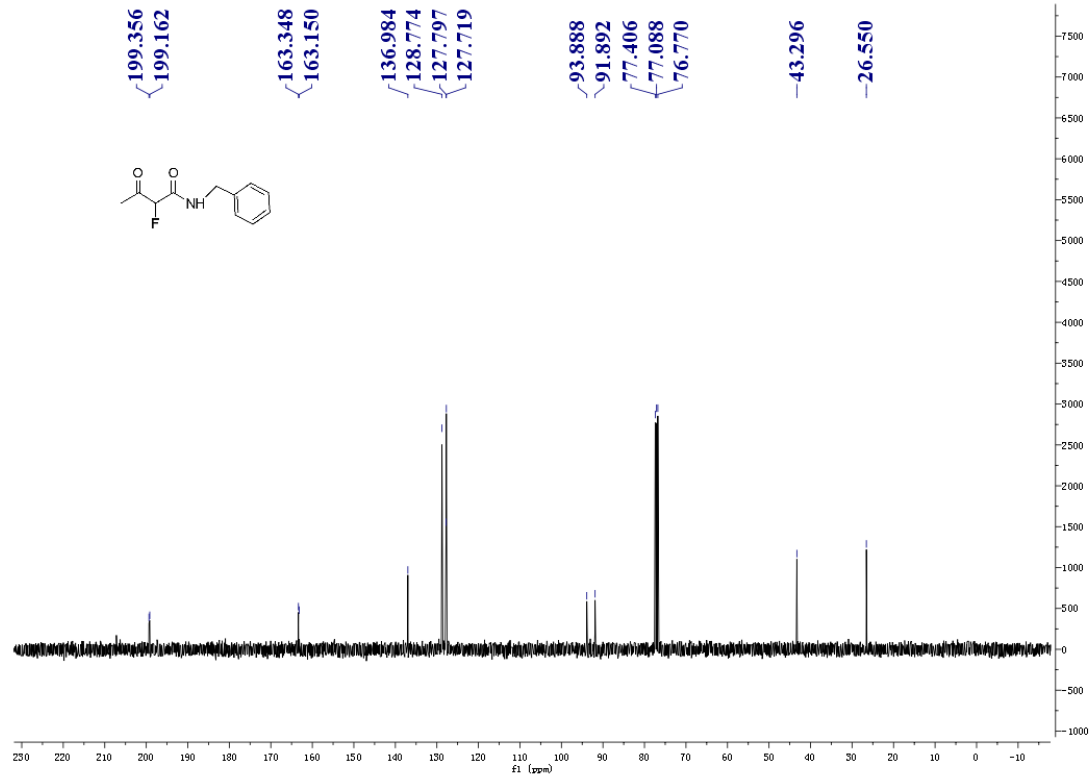
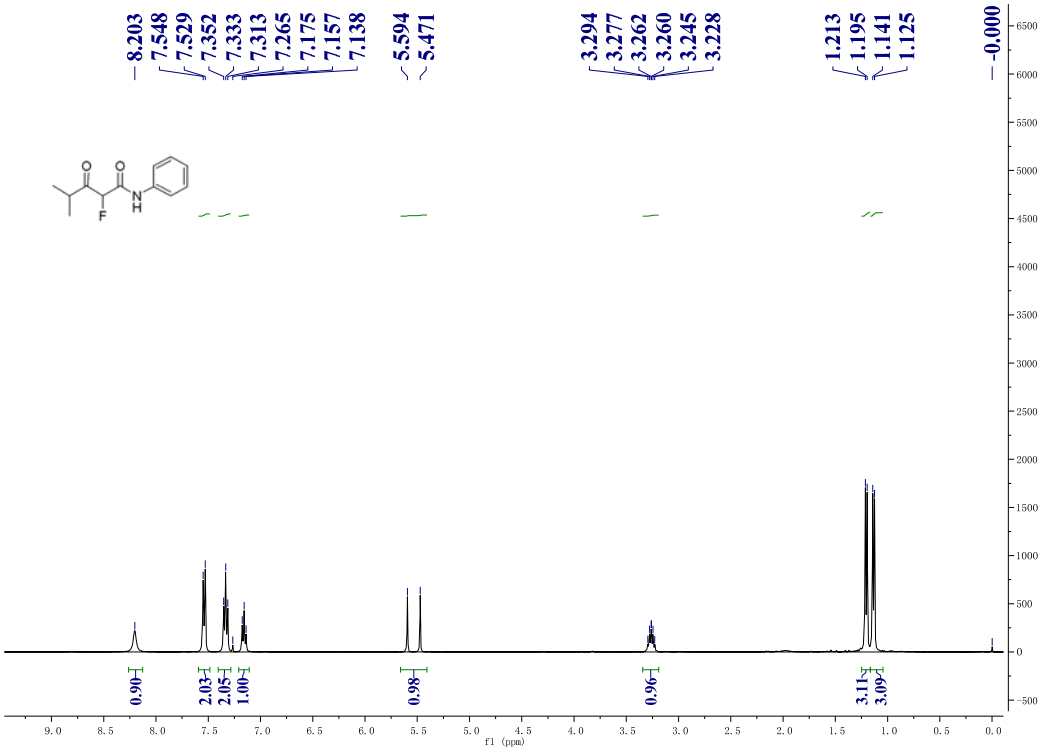
90.361
77.480
77.160
76.840
41.587
40.519
25.848
13.998
12.303
f1 (ppm)

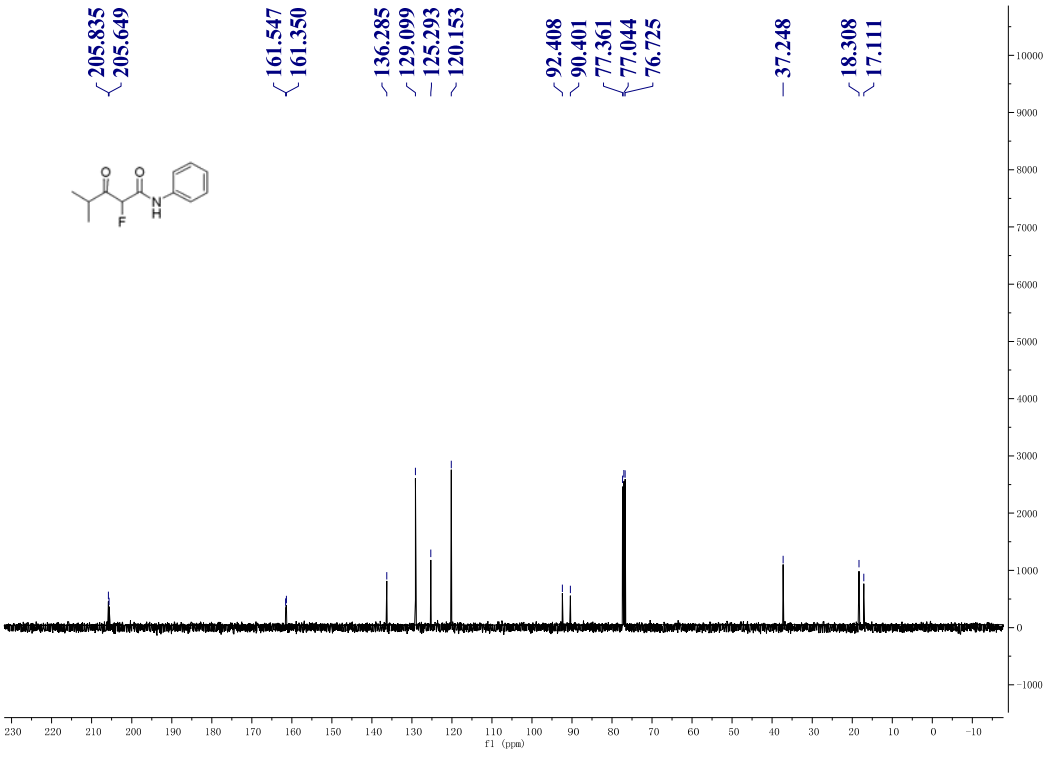
Compound **2u**


7.349
7.332
7.314
7.300
7.283
7.255
7.237
6.868
5.369
5.246
4.511
4.496
4.474
4.455
4.438
4.416
4.402
2.373
2.367
O
O
NH
F
5.00
0.88
0.91
2.04
2.84

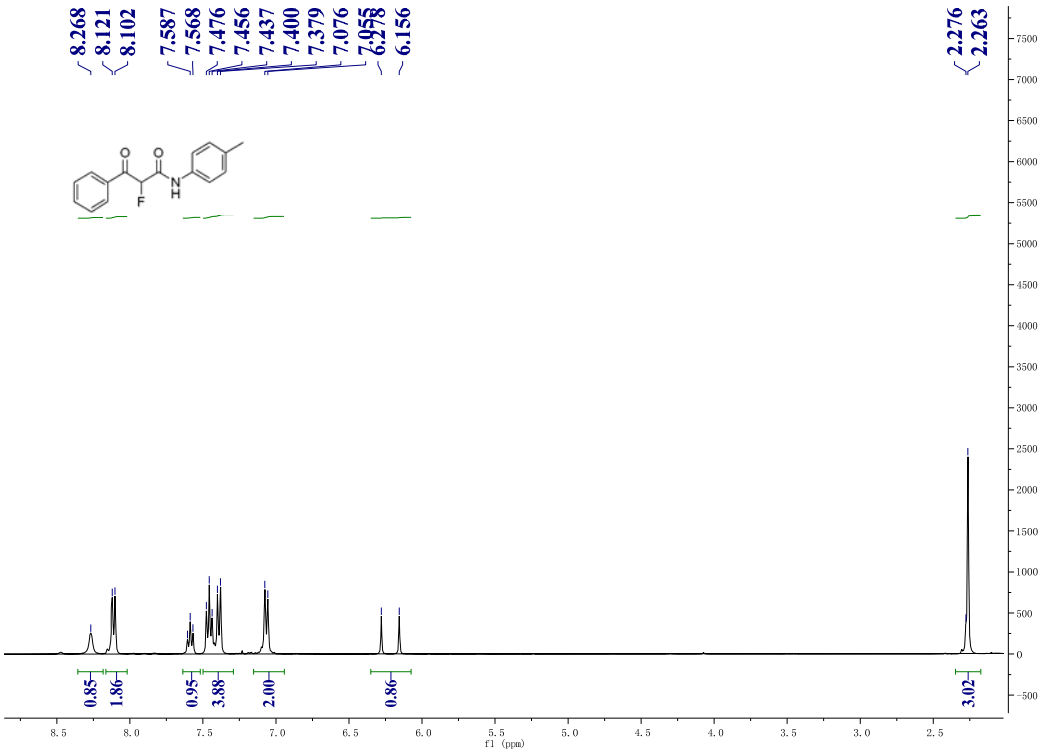

199.356
199.162
163.348
163.150
136.984
128.774
127.797
127.719
93.888
91.892
77.406
77.088
76.770
43.296
26.550
O
O
NH
F

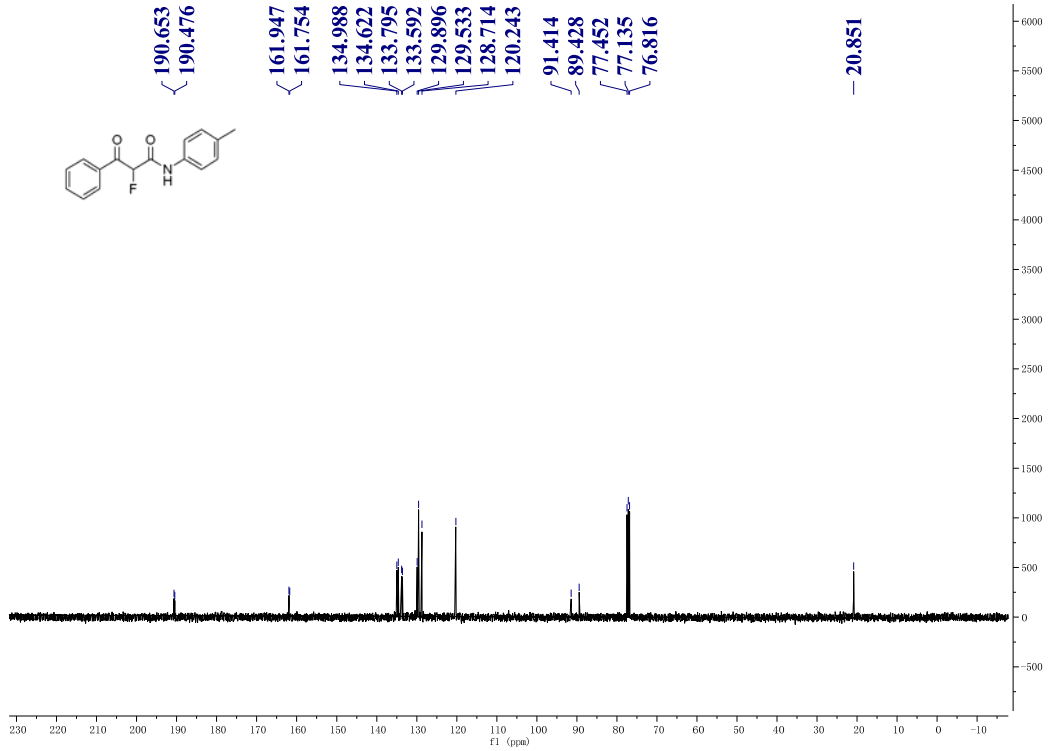
Compound **2v**


8.203
7.548
7.529
7.352
7.333
7.313
7.265
7.175
7.157
7.138
5.594
5.471
3.294
3.277
3.262
3.260
3.245
3.228
1.213
1.195
1.141
1.125
0.000
0.90
2.03
2.05
1.00
0.98
0.96
3.11
3.09
f1 (ppm)

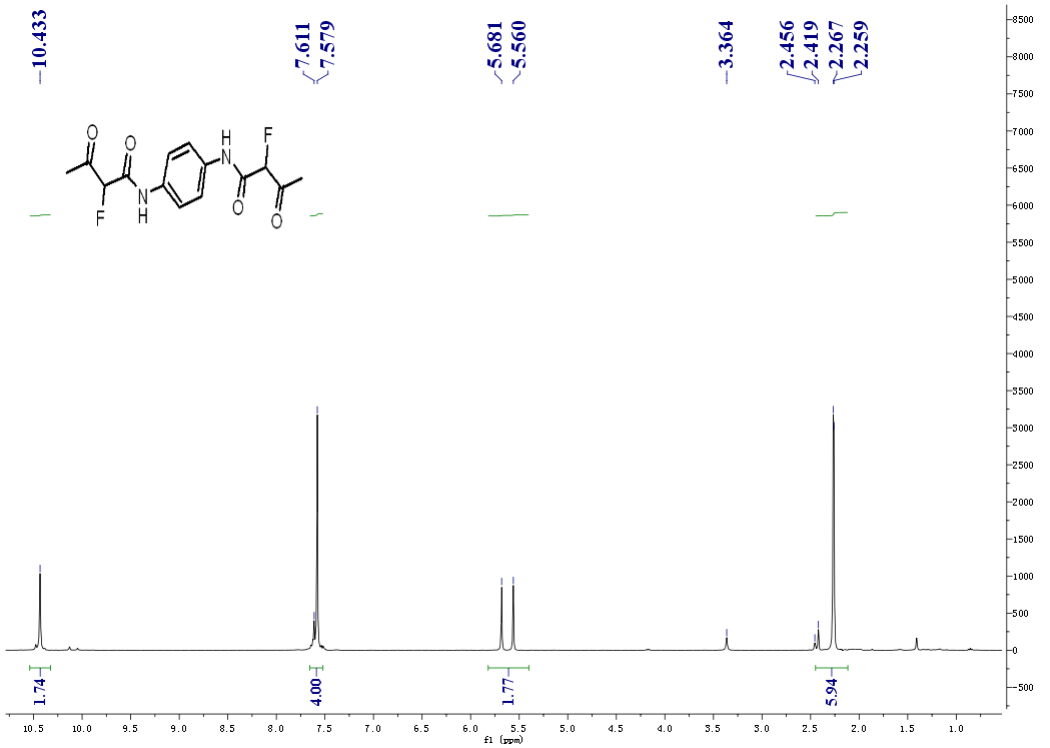

205.835
205.649
161.547
161.350
136.285
129.099
125.293
120.153
92.408
90.401
77.361
77.044
76.725
37.248
18.308
17.111
f1 (ppm)

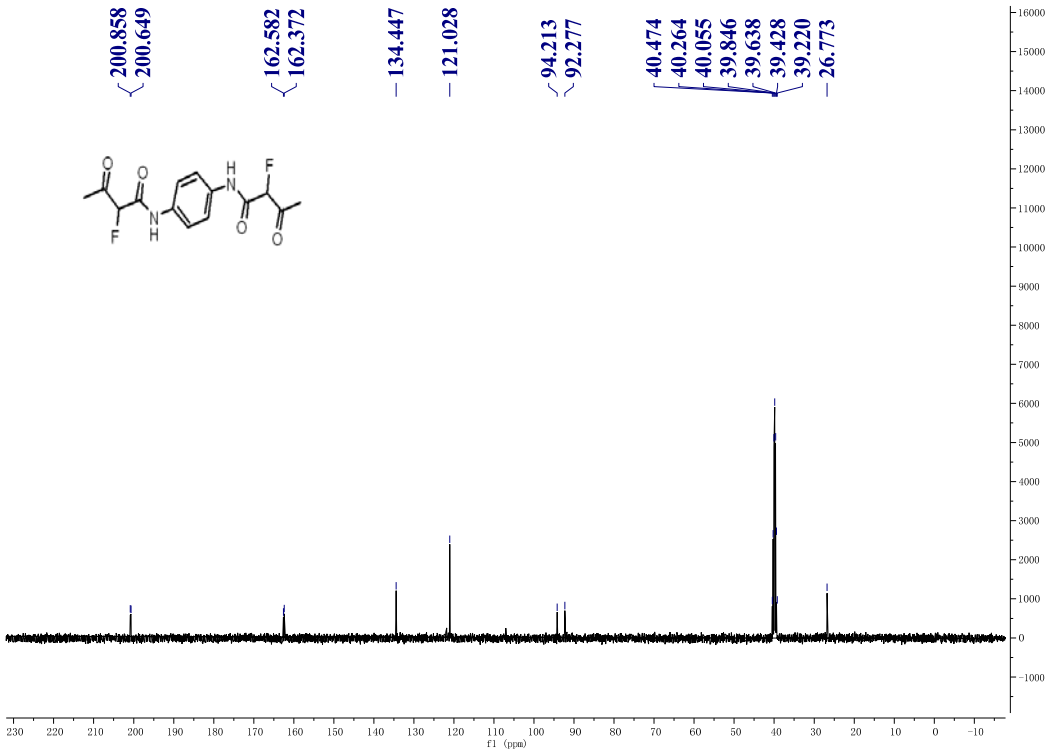
Compound **2w**


8.268
8.121
8.102
7.587
7.568
7.476
7.456
7.437
7.400
7.379
7.076
7.055
6.278
6.156
2.276
2.263
0.85
1.86
0.95
3.88
2.00
0.86
3.02
f1 (ppm)

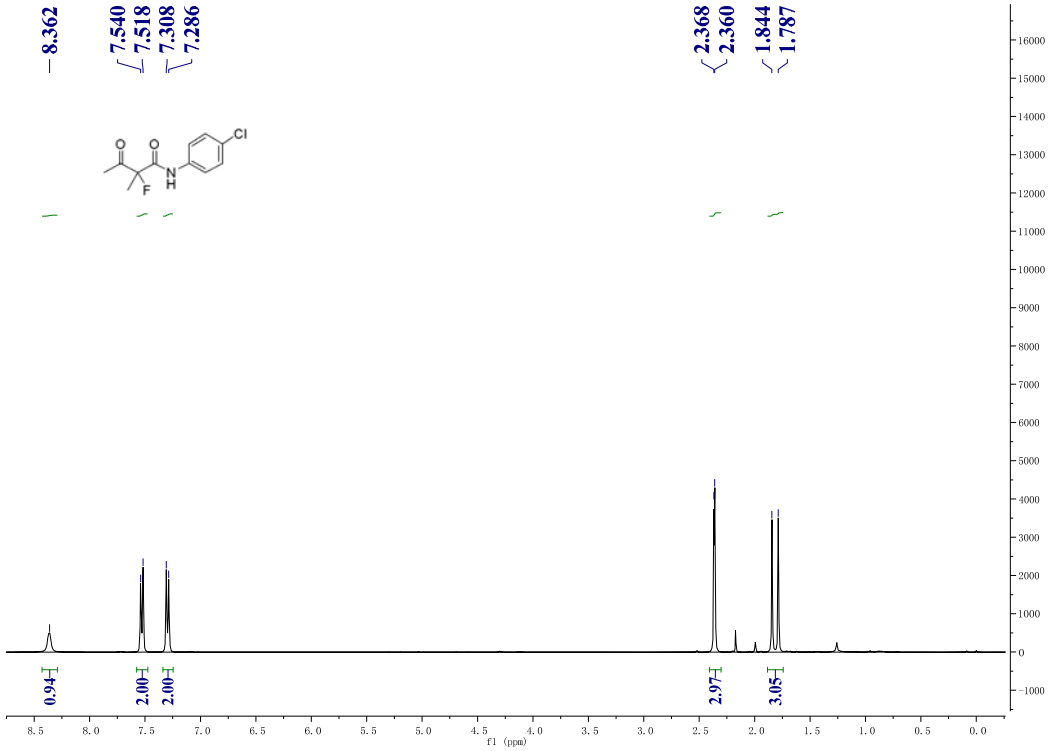

190.653
190.476
161.947
161.754
134.988
134.622
133.795
133.592
129.896
129.533
128.714
120.243
91.414
89.428
77.452
77.135
76.816
20.851
f1 (ppm)

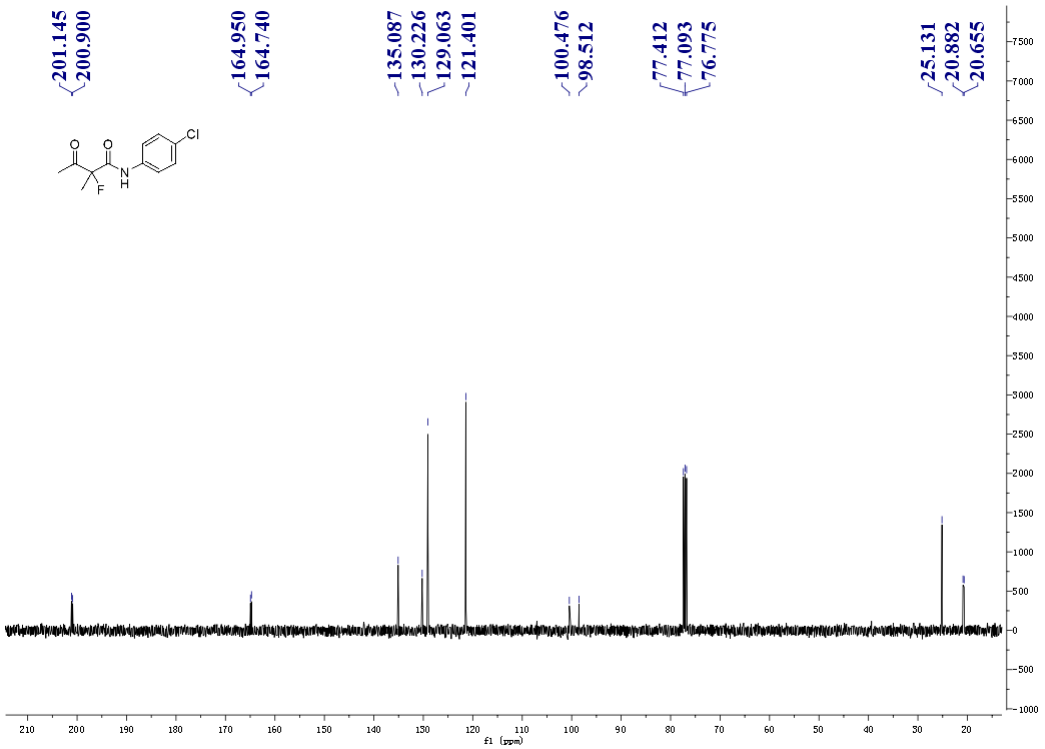
Compound **2x**


10.433
7.611
7.579
5.681
5.560
3.364
2.456
2.419
2.267
2.259
1.74
4.00
1.77
5.94
f1 (ppm)


200.858
200.649
162.582
162.372
134.447
121.028
94.213
92.277
40.474
40.264
40.055
39.846
39.638
39.428
39.220
26.773
f1 (ppm)

Compound **2y**


8.362
7.540
7.518
7.308
7.286
2.368
2.360
1.844
1.787
0.94
2.00
2.00
2.97
3.05
f1 (ppm)


201.145
200.900
164.950
164.740
135.087
130.226
129.063
121.401
100.476
98.512
77.412
77.093
76.775
25.131
20.882
20.655
f1 (ppm)