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Surpporting Information

Silver-initiated Radical Ring Expansion/Fluorination of Ethynyl cyclobutanols: Efficient Synthesis of Monofluoroethenyl Cyclopentanones

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Contents

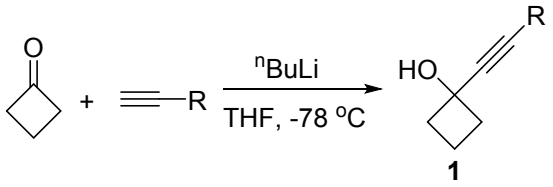
1. General information

All manipulations were carried out under argon using standard Schlenk techniques. All glassware was oven or flame dried immediately prior to use. All solvents were purified and dried according to standard methods prior to use, unless stated otherwise. All reagents were obtained from commercial sources and used without further purification. Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). Silica gel 60 (200 - 300 mesh) was used for column chromatography. ^{1}H NMR spectra were obtained at

400 MHz and recorded relative to the tetramethylsilane signal (0 ppm) or residual proton-solvent. ^{13}C NMR spectra were obtained at 100 MHz, and chemical shifts were recorded relative to the solvent resonance ($CDCl_3$, 77.0 ppm). Data for ^1H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, br = broad singlet, coupling constant (s) in Hz, integration).

2. Experimental Details

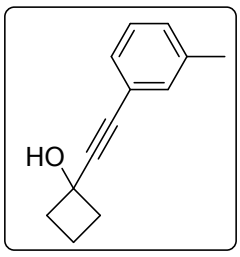
(a) General preparation and characterization of start materials



1

The compound **1** was synthesized followed a modified procedure from the literature.[1] In a 50 mL flashe, a solution of alkyne (3.78 mmol) in anhydrous THF (10 mL) was cooled to -78℃, 2.3 mL of n-butyllithium solution (1.6 M in THF) was added dropwise. After 30 min, a solution of cyclobutanone (0.28 mL, 3.78 mmol) in anhydrous THF was added added. The resulting reaction mixture was allowed to warm to room temperature. After 2 h, the mixture was quenched with water, then extracted with 3*20 mL of EtOAc. The combined organic phased were washed with NH_4Cl solution and brine. The organic phase was dried over $MgSO_4$, the filtrate was concentrated in vacuo to afford yellow oil, which was purified by chromatography (EA/PE=1/10) to afford the product (yield: 60-94 %).

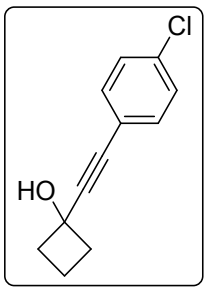
Characterization of start materials:



1-(m-tolylethynyl)cyclobutan-1-ol (1b)

Yield: 89%; Yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.25 (s, 1H), 7.23 (d, J = 8.1 Hz, 1H), 7.18 (t, J = 7.5 Hz, 1H), 7.10 (d, J = 7.5 Hz, 1H), 2.51 (dt, J = 15.8, 6.3 Hz, 2H), 2.34 (t, J = 9.3 Hz, 2H), 2.30 (s, 3H), 1.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$)

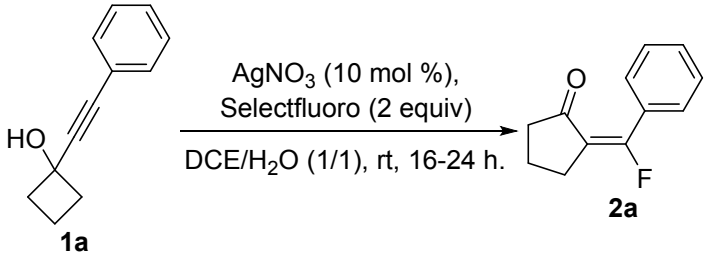
δ 137.94, 132.27, 129.19, 128.72, 128.17, 122.49, 92.16, 83.58, 68.31, 38.64, 21.20, 12.98; HRMS (EI$^+$, 70 eV): $C_{13}H_{14}O$ [M]$^+$: calcd. 186.1045, found 186.1047.



1-((4-chlorophenyl)ethynyl)cyclobutan-1-ol (1f)

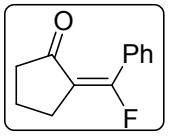
Yield: 90%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.19 (m, 4H), 2.53-2.47 (m, 2H), 2.36-2.29 (m, 2H), 1.89-1.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.07, 131.54, 129.78, 129.50, 128.58, 124.42, 93.71, 82.09, 68.22, 38.52, 12.97; HRMS (EI$^+$, 70 eV): $C_{12}H_{11}ClO$ [M]$^+$: calcd. 206.0498, found 206.0510.

(b) General procedure for silver-catalyzed ring-opening and fluorination.



In a vial, **1a** (52 mg, 0.3 mmol), selecfluoro (212 mg, 0.6 mmol) and silver nitrate (5.0 mg, 0.03 mmol) was dissolved in DEC/H_2O (1 mL /1 mL). The resulting mixture was stirred under N_2 atmosphere at room temperature for 16 h, the organic phase was purified by column chromatography on silica gel with a gradient eluent of petroleum ether and ethyl acetate to afford the fluorine product 2a (yield, 85%).

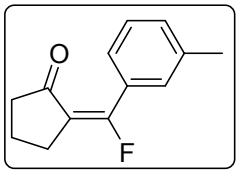
The characterization of products



(E)-2-(fluoro(phenyl)methylene)cyclopentan-1-one (2a)

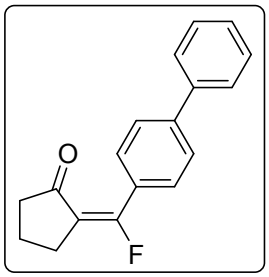
Yield: 85%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.46-7.40 (m, 3H), 2.94 (td, *J* = 7.3, 3.3 Hz, 2H), 2.43 (td, *J* = 7.9, 1.2 Hz, 2H),

1.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7 (d, *J* = 14.4 Hz), 162.4 (d, *J* = 270.7 Hz), 131.1, 130.0, 129.7, 128.7 (d, *J* = 7.0 Hz), 127.8, 117.3 (d, *J* = 19.4 Hz), 40.7 (d, *J* = 4.3 Hz), 27.6 (d, *J* = 3.9 Hz), 19.4; ^{19}F (376 MHz, $CDCl_3$) δ -76.9; HRMS (EI^+, 70 eV): $C_{12}H_{11}FO$ $[M]^+$: calcd. 190.0794, found 190.0795.



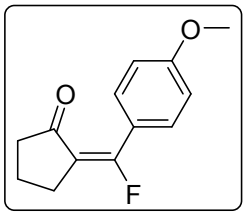
(E)-2-(fluoro(m-tolyl)methylene)cyclopentan-1-one (2b)

Yield: 80%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.58 (m, 2H), 7.33-7.26 (m, 2H), 2.93 (td, *J* = 7.3, 3.3 Hz, 2H), 2.43 (td, *J* = 7.9, 1.3 Hz, 2H), 2.39 (s, 3H), 1.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7 (d, *J* = 14.3 Hz), 162.7 (d, *J* = 270.6 Hz), 161.3, 137.4, 131.9, 129.9, 129.7, 129.1 (d, *J* = 6.7 Hz), 127.7, 126.0 (d, *J* = 7.0 Hz), 117.1 (d, *J* = 19.5 Hz), 40.7 (d, *J* = 4.3 Hz), 27.6 (d, *J* = 3.9 Hz), 21.3, 19.4; ^{19}F (376 MHz, $CDCl_3$) δ -76.3; HRMS (EI^+, 70 eV): $C_{13}H_{13}FO$ $[M]^+$: calcd. 204.0950, found 204.0952;



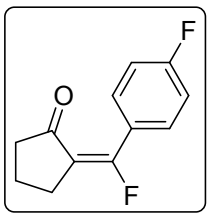
(E)-2-([1,1'-biphenyl]-4-ylfluoromethylene)cyclopentan-1-one (2c)

Yield: 74%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.64 (t, *J* = 8.1 Hz, 4H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.38 (dd, *J* = 8.4, 6.3 Hz, 1H), 2.97 (td, *J* = 7.3, 3.3 Hz, 2H), 2.46 (td, *J* = 7.9, 1.3 Hz, 2H), 2.01 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.9 (d, *J* = 14.3 Hz), 160.9 (d, *J* = 214.3 Hz), 143.8, 140.2, 129.1 (d, *J* = 7.3 Hz), 128.8, 127.8, 127.2, 126.5, 117.5, 40.8 (d, *J* = 4.3 Hz), 27.8 (d, *J* = 4.1 Hz), 19.4; ^{19}F (376 MHz, $CDCl_3$) δ -77.8; HRMS (EI^+, 70 eV): $C_{18}H_{15}FO$ $[M]^+$: calcd. 266.1107, found 266.1108;



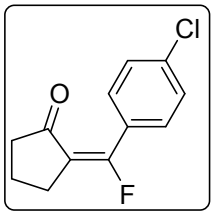
(E)-2-(fluoro(4-methoxyphenyl)methylene)cyclopentan-1-one (2d)

Yield: 83%; Colorless solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 2.92 (td, *J* = 7.2, 3.1 Hz, 2H), 2.43 (t, *J* = 7.9 Hz, 2H), 1.97 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8 (d, *J* = 17.7 Hz), 163.8 (d, *J* = 220.1 Hz), 161.7, 161.2, 130.5 (d, *J* = 7.7 Hz), 122.2, 115.8, 113.2, 55.3, 40.9 (d, *J* = 4.2 Hz), 27.7 (d, *J* = 4.3 Hz), 19.5; ^{19}F (376 MHz, $CDCl_3$) δ -77.3; HRMS (EI^+, 70 eV): $C_{13}H_{13}FO_2$ $[M]^+$: calcd. 220.0900, found 220.0902;



(E)-2-(fluoro(4-fluorophenyl)methylene)cyclopentan-1-one (2e)

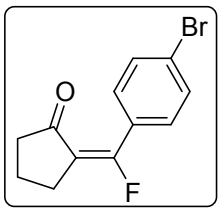
Yield: 77%; Colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.80 (m, 2H), 7.12-7.07 (m, 2H), 2.93 (td, *J* = 7.3, 3.3 Hz, 2H), 2.43 (td, *J* = 7.9, 1.3 Hz, 2H), 1.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.77 (d, *J* = 14.3 Hz), 174.58, 165.37, 162.79 (d, *J* = 18.0 Hz), 160.03, 131.10 (dd, *J* = 9.1, 7.2 Hz), 125.78, 117.21 (d, *J* = 19.5 Hz), 115.13, 114.91, 40.77 (d, *J* = 4.6 Hz), 27.65 (d, *J* = 4.4 Hz), 19.39; ^{19}F (376 MHz, $CDCl_3$) δ -77.0, -107.7 (m); HRMS (EI^+, 70 eV): $C_{12}H_{10}F_2O$ $[M]^+$: calcd. 208.0700, found 208.0703;



(E)-2-((4-chlorophenyl)fluoromethylene)cyclopentan-1-one (2f)

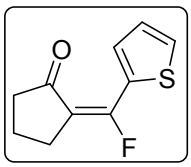
Yield: 67%; Colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 2.91 (td, *J* = 7.2, 3.3 Hz, 2H), 2.42 (t, *J* = 7.8 Hz, 2H), 1.97

(m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.71 (d, *J* = 14.4 Hz), 162.43 (d, *J* = 270.7 Hz), 131.14, 130.0, 129.7, 128.76 (d, *J* = 7.0 Hz), 127.80, 117.31 (d, *J* = 19.4 Hz), 40.74 (d, *J* = 4.3 Hz), 27.63 (d, *J* = 3.9 Hz), 19.49; ^{19}F (376 MHz, $CDCl_3$) δ -78.4; HRMS (EI^+, 70 eV): $C_{12}H_{10}ClFO$ $[M]^+$: calcd. 224.0404, found 224.0408;



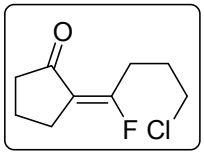
(E)-2-((4-bromophenyl)fluoromethylene)cyclopentan-1-one (2g)

Yield: 60%; Colorless oil ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 6.2 Hz, 2H), 7.44 (d, *J* = 7.7 Hz, 2H), 2.95 (td, *J* = 7.3, 3.3 Hz, 1H), 2.45 (t, *J* = 7.6 Hz, 1H), 2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.89, 162.76 (d, *J* = 262.5 Hz), 131.37, 128.99 (d, *J* = 7.1 Hz), 128.07, 117.67, 41.03 (d, *J* = 4.6 Hz), 27.90 (d, *J* = 4.1 Hz), 19.68; ^{19}F (376 MHz, $CDCl_3$) δ -77.4; HRMS (EI^+, 70 eV): $C_{12}H_{10}BrFO$ $[M]^+$: calcd. 267.9899, found 267.9901.



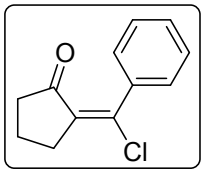
(E)-2-(fluoro(thiophen-2-yl)methylene)cyclopentan-1-one (2i)

Yield: 60%; White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 3.9 Hz, 1H), 7.53 (ddd, J = 4.5, 3.1, 1.1 Hz, 1H), 7.12 (ddd, J = 5.1, 3.9, 2.0 Hz, 1H), 2.94 (td, J = 7.3, 3.1 Hz, 2H), 2.48 (td, J = 7.9, 1.3 Hz, 2H), 2.00 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.09 (d, J = 14.7 Hz), 158.45 (d, J = 256 Hz), 132.40, 131.12 (d, J = 8.9 Hz), 130.53, 127.07, 115.56 (d, J = 21.0 Hz), 40.67 (d, J = 3.8 Hz), 27.75 (d, J = 4.6 Hz), 19.78; ^{19}F (376 MHz, $CDCl_3$) δ -81.40; HRMS (EI^+, 70 eV): $C_{10}H_9FOS$ $[M]^+$: calcd. 196.0358, found 196.0356.



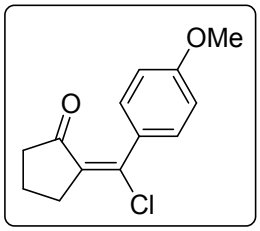
(E)-2-(4-chloro-1-fluorobutylidene)cyclopentan-1-one (2j)

Yield: 58%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.52 (t, *J* = 6.8 Hz, 2H), 2.96 (t, *J* = 7.4 Hz, 1H), 2.90 (t, *J* = 7.4 Hz, 1H), 2.69 (t, *J* = 6.5 Hz, 2H), 2.31 (t, *J* = 7.8 Hz, 2H), 2.03-1.96 (m, 2H), 1.91-1.83 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.23, 168.87, 116.49, 43.89, 40.26 (d, J = 4.9 Hz), 29.19, 26.56, 26.34, 25.80, 19.36. ^{19}F (376 MHz, $CDCl_3$) δ -78.5; HRMS (EI^+, 70 eV): $C_9H_{12}ClFO$ $[M]^+$: calcd. 190.0561, found 190.0563;



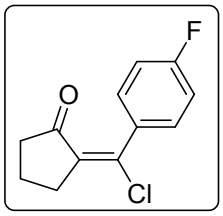
(E)-2-(chloro(phenyl)methylene)cyclopentan-1-one (3a)

Yield: 70%; Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (s, 5H), 2.98 (t, J = 7.3 Hz, 2H), 2.45 (t, J = 7.8 Hz, 2H), 2.00 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.80, 143.09, 136.60, 134.17, 129.69, 128.95, 127.78, 41.16, 32.66, 18.74; HRMS (EI^+, 70 eV): $C_{12}H_{11}ClO$ $[M]^+$: calcd. 206.0498, found 206.0499.



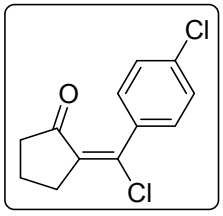
(E)-2-(chloro(4-methoxyphenyl)methylene)cyclopentan-1-one (3b)

Yield: 66%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 3.84 (s, 3H), 2.97 (t, J = 7.3 Hz, 2H), 2.46 (t, J = 7.9 Hz, 2H), 1.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.55, 163.31, 156.08, 131.50, 129.93, 121.91, 113.66, 55.47, 33.04, 32.09, 16.28; HRMS (EI^+, 70 eV): $C_{13}H_{13}ClO_2$ $[M]^+$: calcd. 236.0604, found 235.0607.



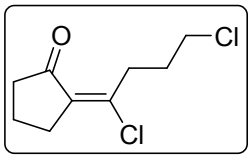
(E)-2-(chloro(4-fluorophenyl)methylene)cyclopentan-1-one (3c)

Yield: 60%; White solid;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (dd, J = 8.5, 5.4 Hz, 2H), 7.05 (t, J = 8.6 Hz, 2H), 2.97 (t, J = 7.3 Hz, 2H), 2.46 (t, J = 7.9 Hz, 2H), 2.00 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.83, 164.54, 162.05, 141.95, 134.37, 132.48, 131.21 (d, J = 8.6 Hz), 114.97, 114.75, 41.18, 32.73, 18.68; HRMS (EI^+, 70 eV): $C_{12}H_{10}ClFO$ $[M]^+$: calcd. 224.0404, found 224.0404.



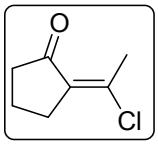
(E)-2-(chloro(4-chlorophenyl)methylene)cyclopentan-1-one (3d)

Yield: 71%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (s, 4H), 2.94 (t, J = 7.4 Hz, 2H), 2.43 (t, J = 7.9 Hz, 2H), 1.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.79, 135.68, 134.78, 130.46, 128.05, 41.15, 32.70, 18.68; HRMS (EI^+, 70 eV): $C_{12}H_{10}Cl_2O$ $[M]^+$: calcd. 240.0109, found 240.0108.



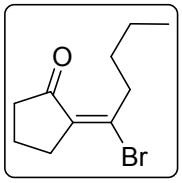
(E)-2-(1,4-dichlorobutylidene)cyclopentan-1-one (3e)

Yield: 47%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.56 (t, J = 6.9 Hz, 2H), 3.18 (t, J = 7.4 Hz, 2H), 2.79 (t, J = 7.3 Hz, 2H), 2.45 (t, J = 7.9 Hz, 2H), 2.06 (dt, J = 14.1, 7.0 Hz, 2H), 1.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.18, 147.94, 134.11, 43.81, 41.24, 32.57, 31.54, 30.96, 18.72; HRMS (EI^+, 70 eV): $C_9H_{12}Cl_2O$ $[M]^+$: calcd. 206.0265, found 206.0268.



(E)-2-(1-chloroethylidene)cyclopentan-1-one (3f)

Yield: 49%; Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.56 (t, J = 6.3 Hz, 2H), 2.72 (t, J = 7.1 Hz, 2H), 2.09 (m, 2H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.13, 120.33, 90.60, 43.96, 42.19, 26.47, 4.07; HRMS (EI^+, 70 eV): C_7H_9ClO $[M]^+$: calcd. 144.0342, found 144.0344.



(E)-2-(1-bromopentylidene)cyclopentan-1-one (3g)

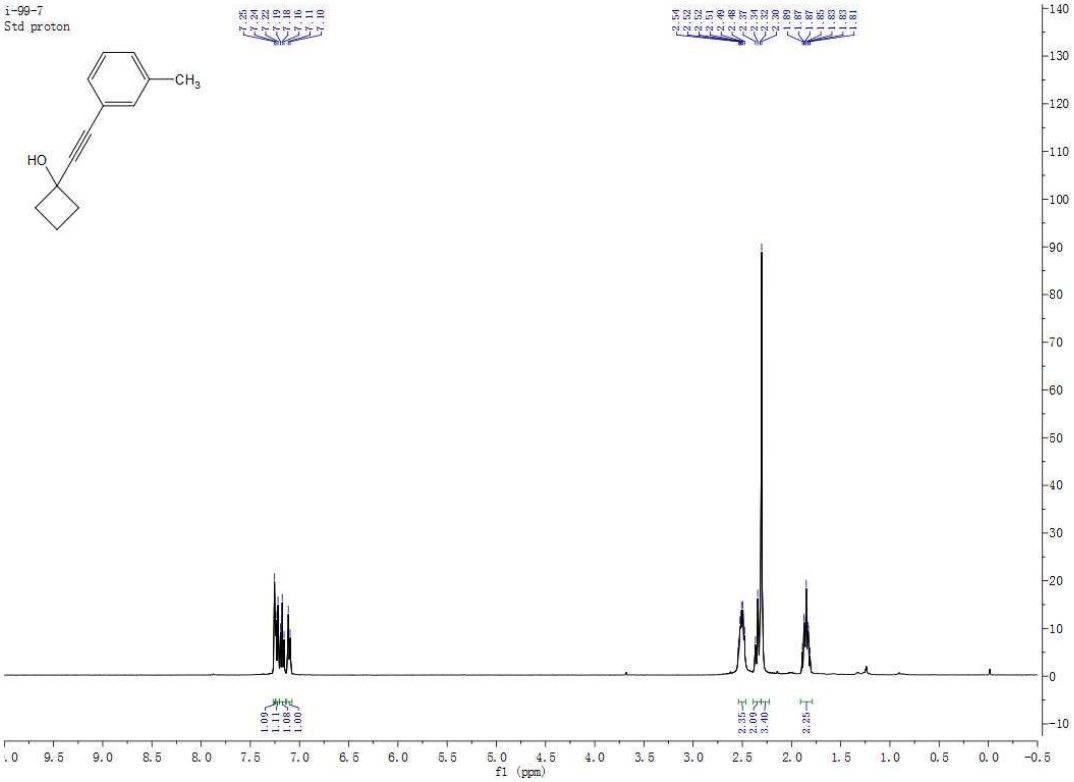
Yield: 40%; Yellow oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 2.73 (t, J = 7.2 Hz, 2H), 2.58 (t, J = 7.5 Hz, 2H), 2.42 (t, J = 7.9 Hz, 2H), 1.87 (m, 2H), 1.61 (dd, J = 15.3, 8.0 Hz, 2H), 1.36 (dq, J = 14.8, 7.3 Hz, 2H), 0.93 (t, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.66, 132.89, 131.61, 41.21, 40.42, 31.55, 29.77, 21.97, 18.78, 13.89; HRMS (EI^+, 70 eV): $C_{10}H_{15}BrO$ $[M]^+$: calcd. 230.0306, found 230.0305.

(c) Reference:

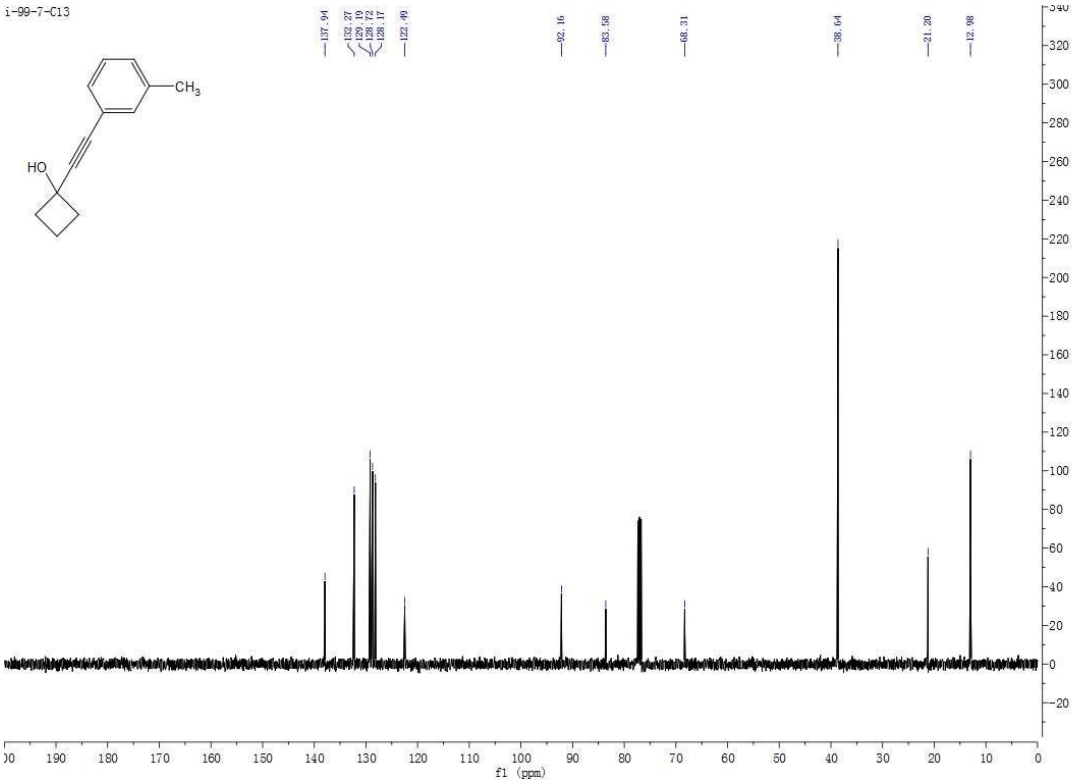
1. Jones, C.; Nguyen, Q.; Driver, T. G., *Angew. Chem. Inter. Ed*. **2014**, *53*, 785.

3. NMR and crystallography data

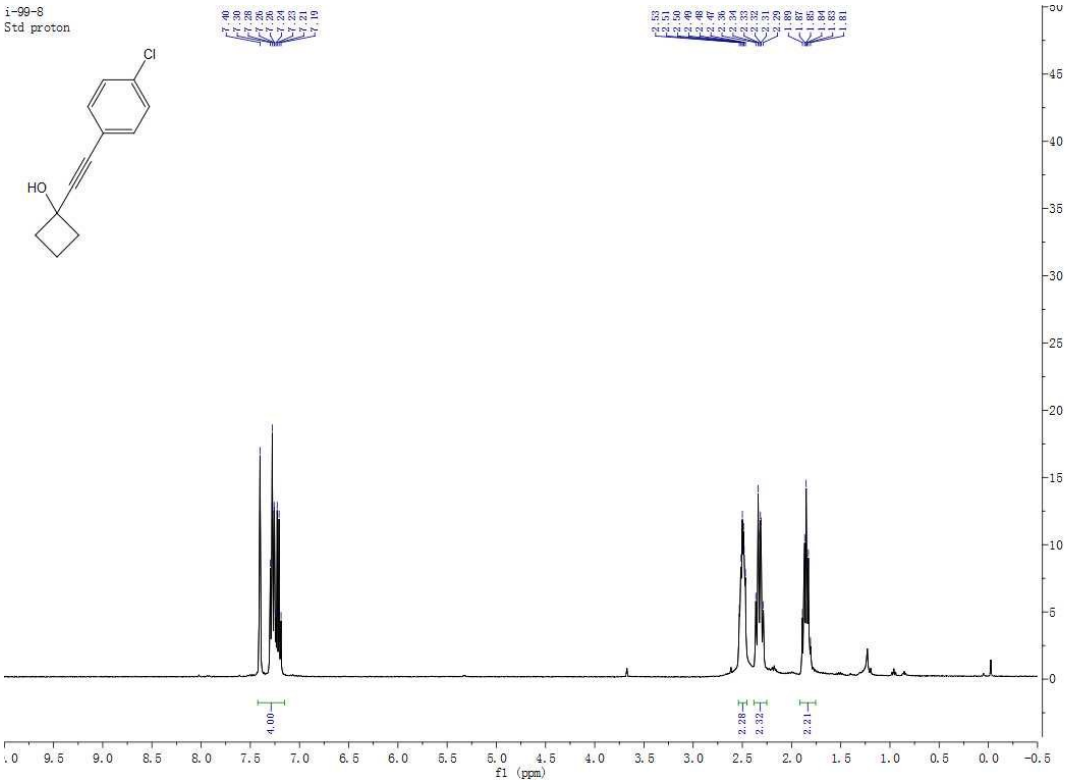
^{1}H-1b



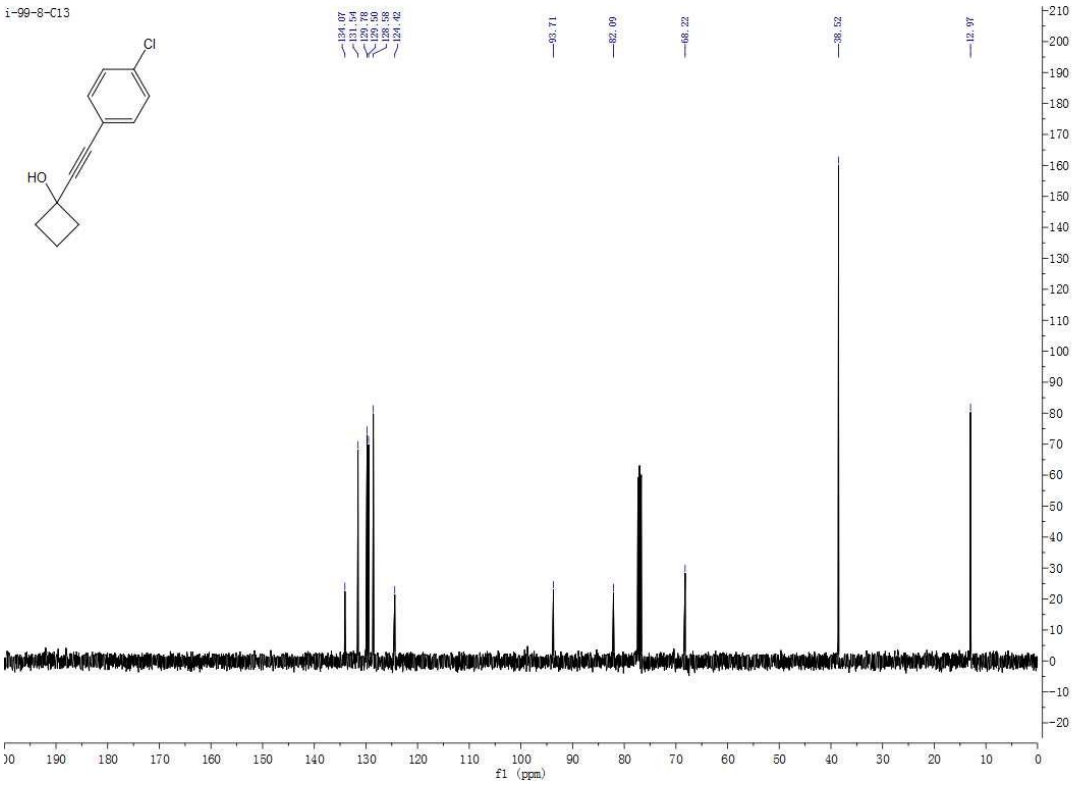
^{13}C-1b



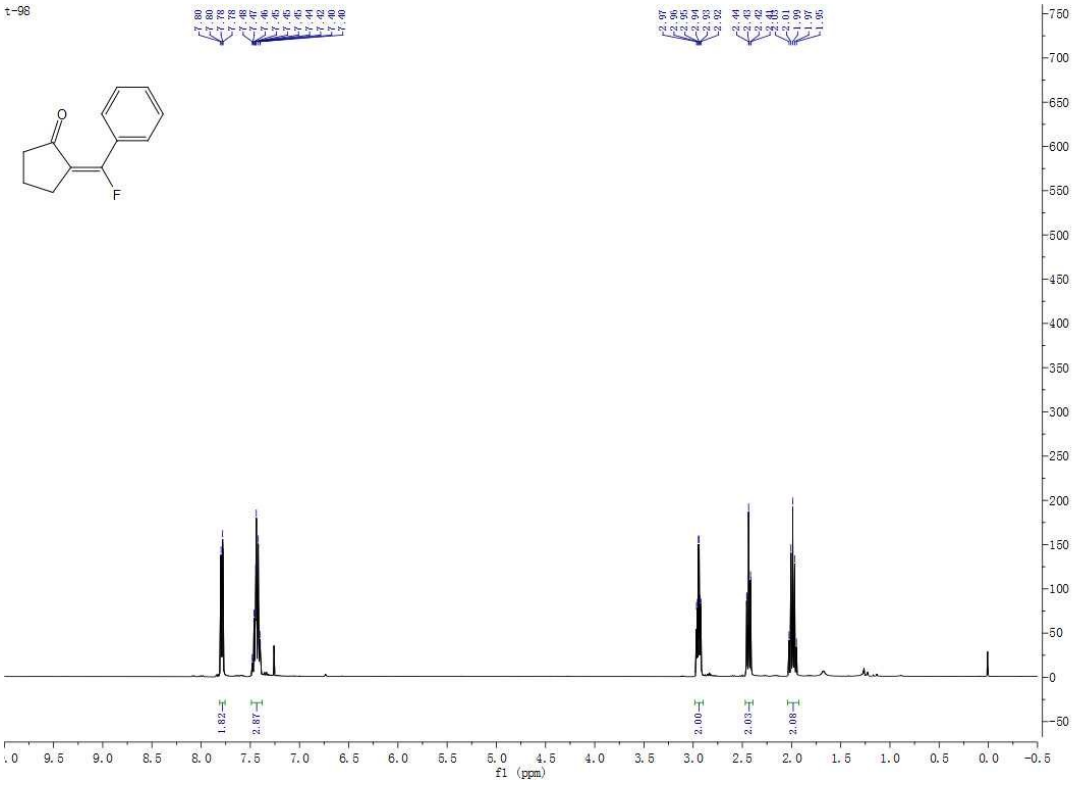
^{1}H-1f



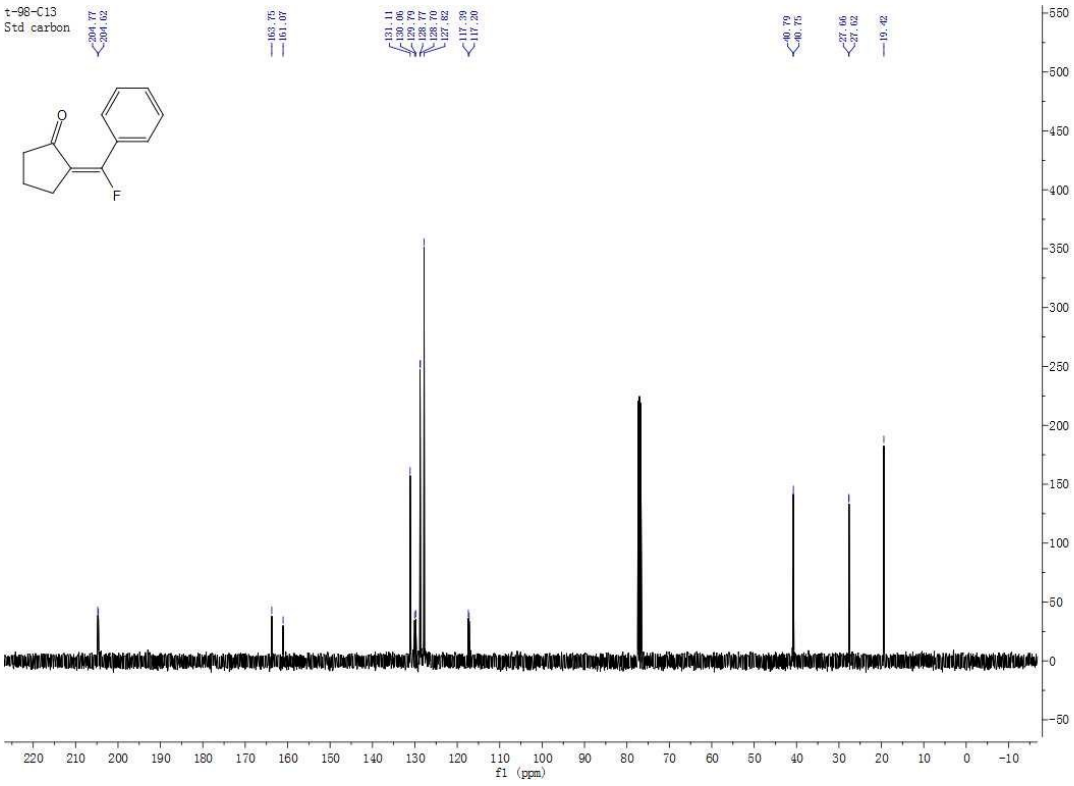
^{13}C-1f



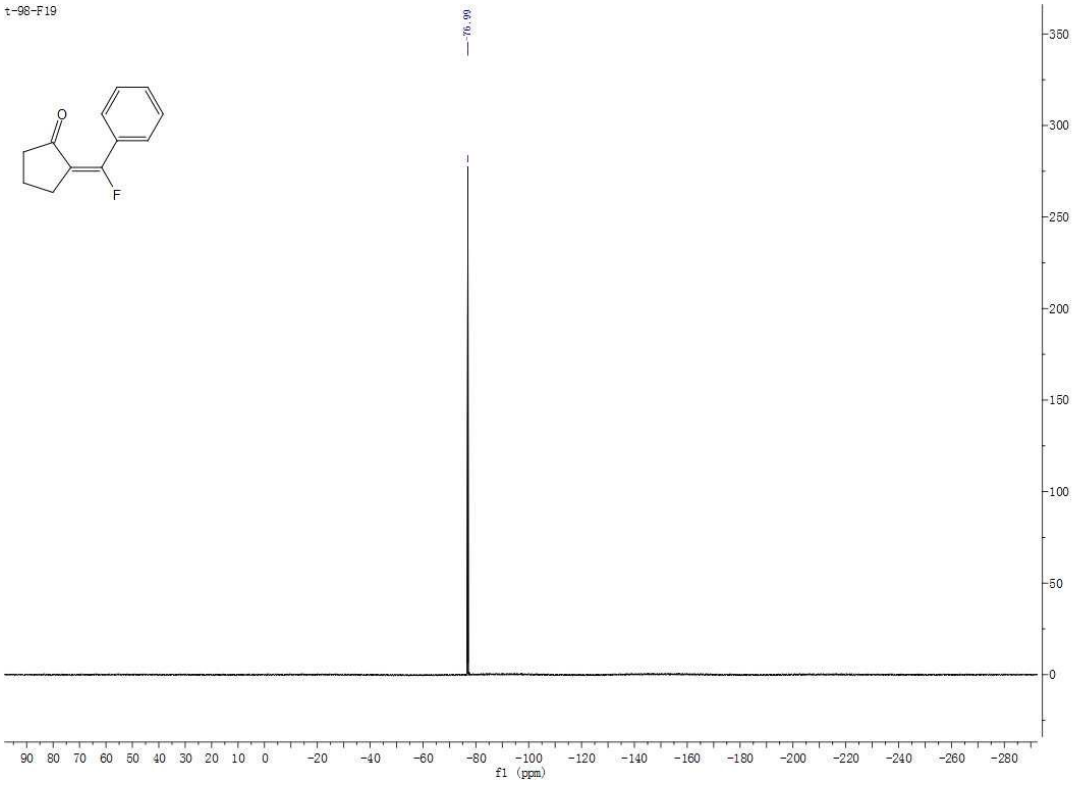
^{1}H-2a



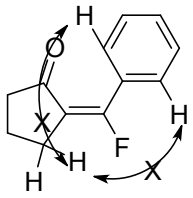
^{13}C-2a

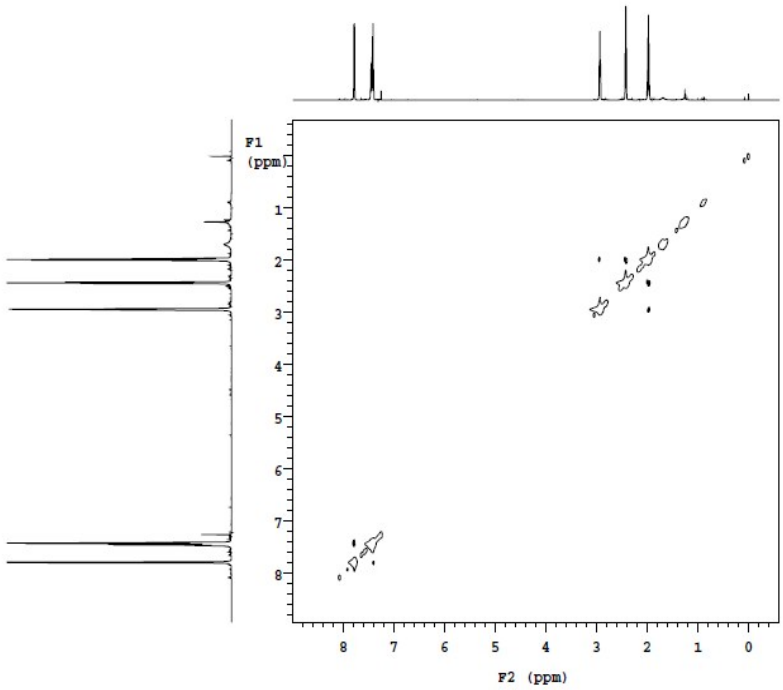


^{19}F-2a

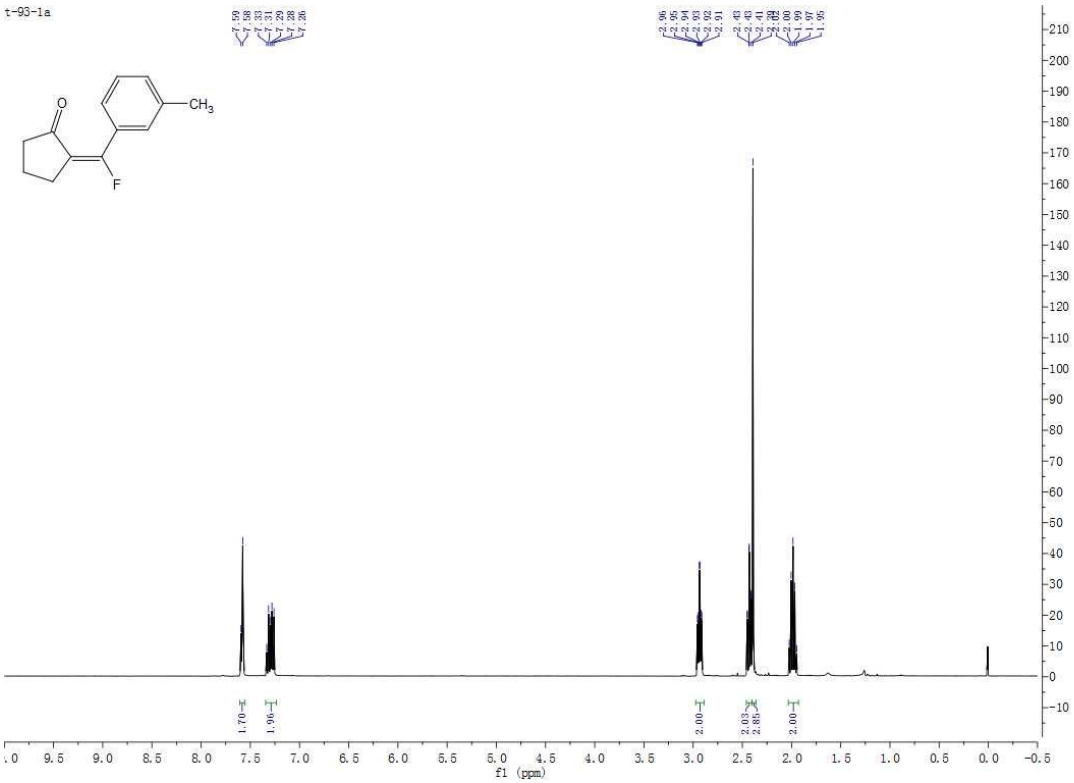

t-98-F19
-76.99
O
F
350
300
250
200
150
100
50
0
90 80 70 60 50 40 30 20 10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
f1 (ppm)

NOESY-2a

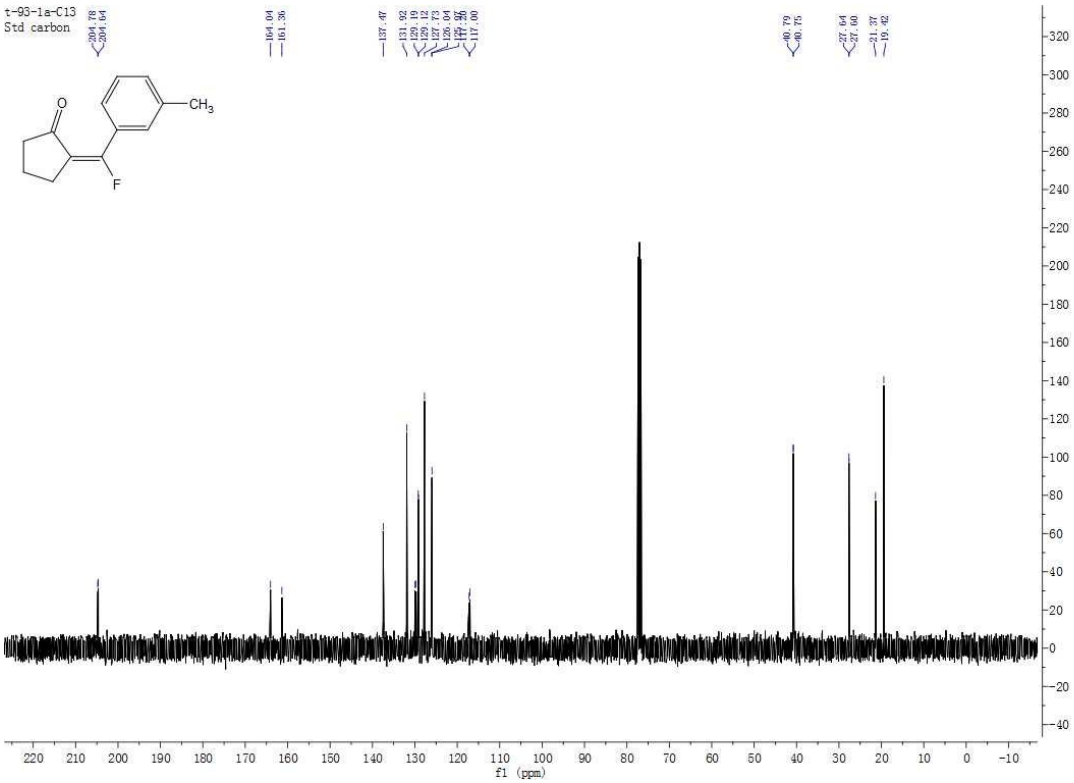

H
O
X
H
F
H
H
X


F1
(ppm)
1
2
3
4
5
6
7
8
8 7 6 5 4 3 2 1 0
F2 (ppm)

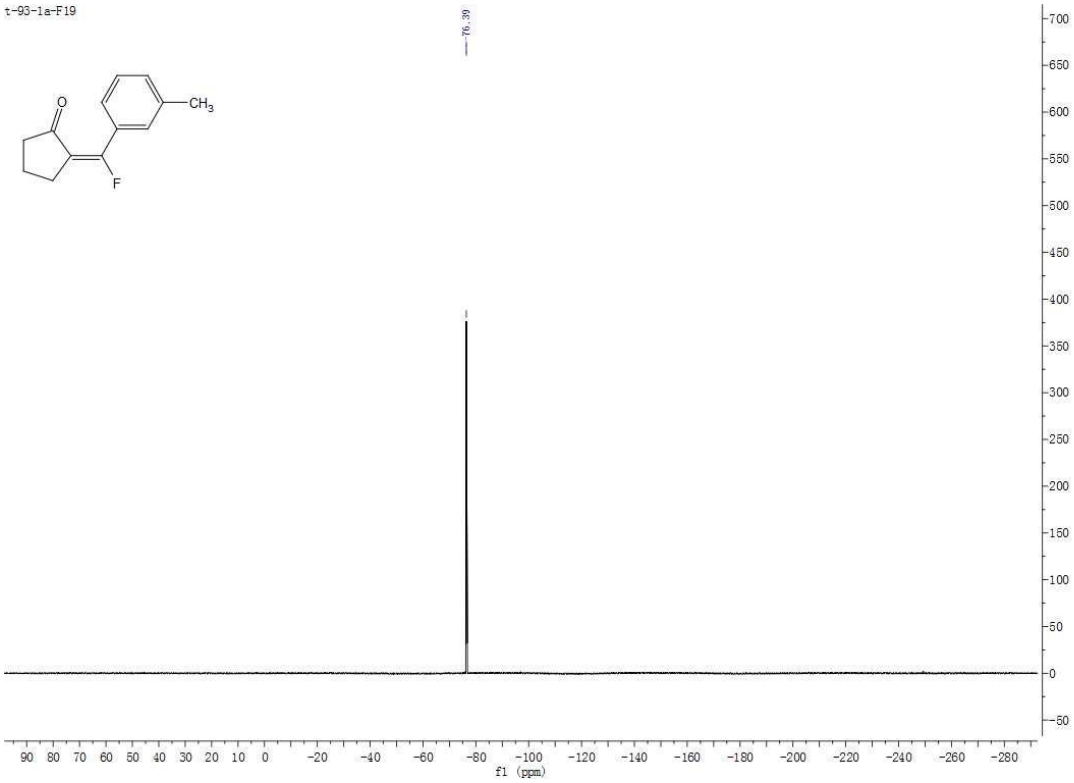
^{1}H-2b



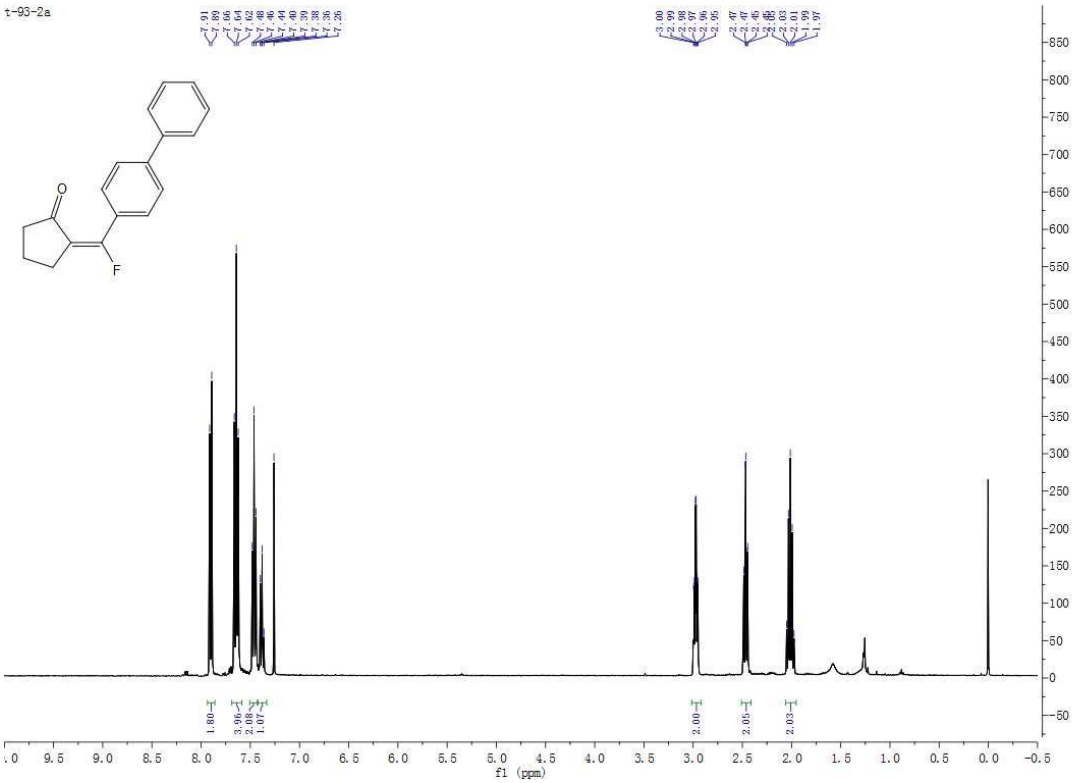
^{13}C-2b



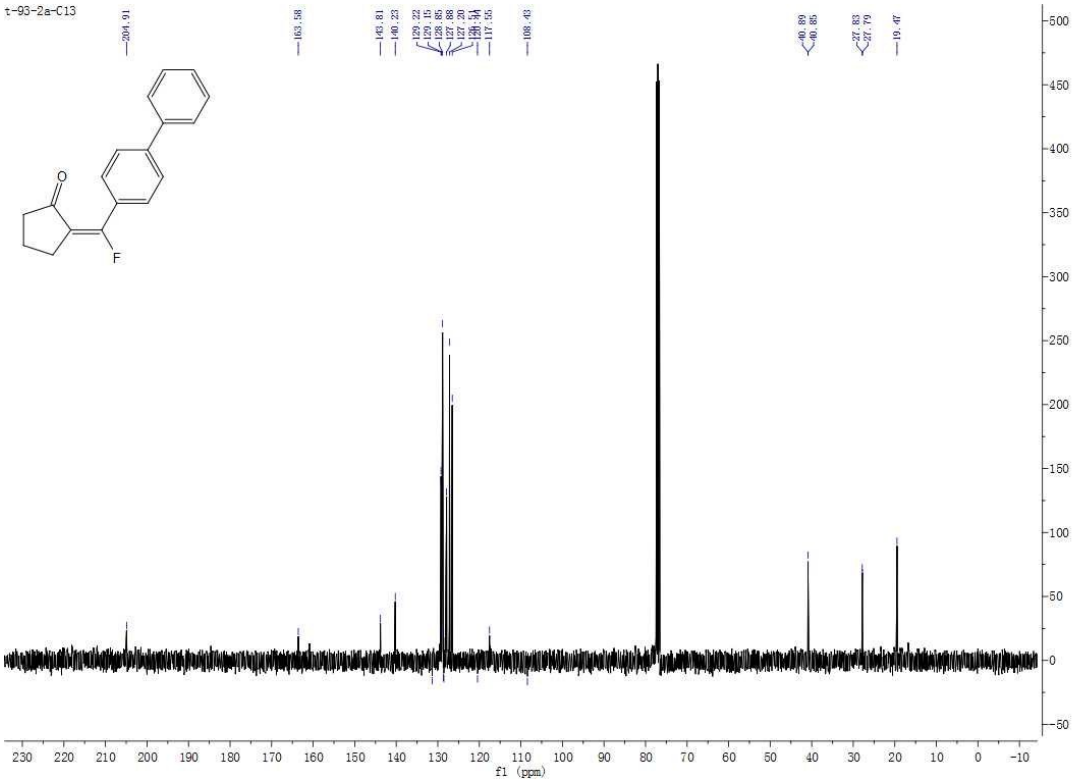
^{19}F-2b


t-93-1a-F19
-76.39
O
CH3
F
f1 (ppm)

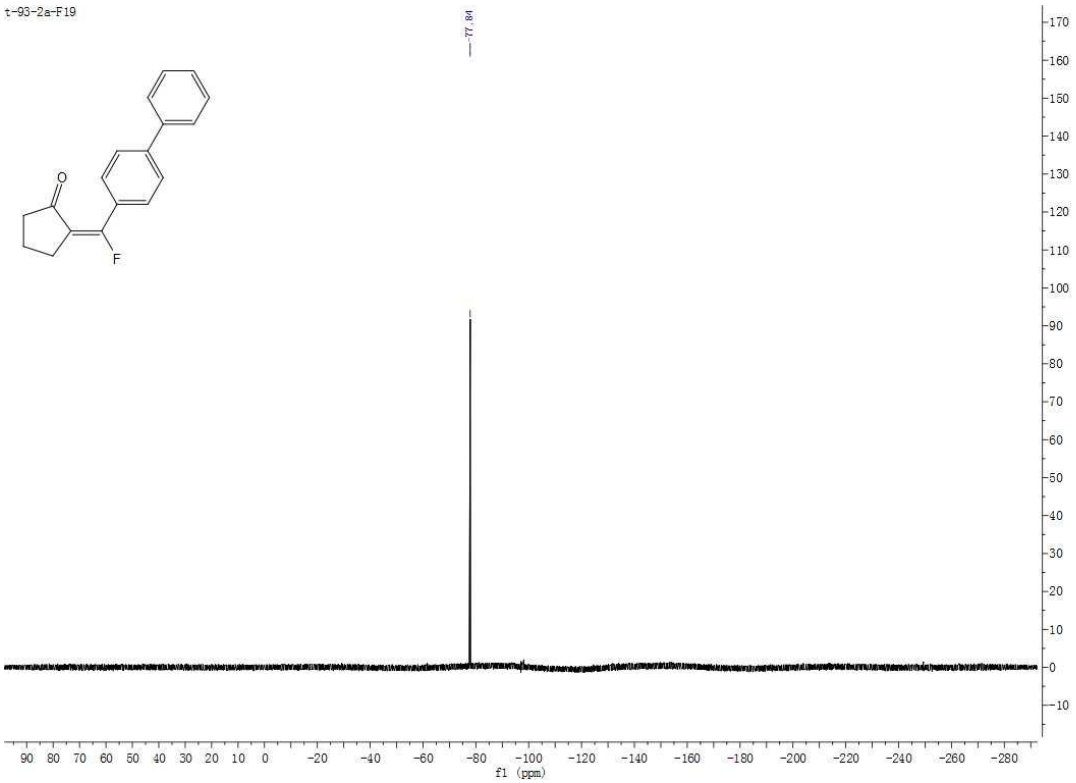
^{1}H-2c


t-93-2a
7.91
7.89
7.66
7.64
7.62
7.48
7.46
7.44
7.40
7.39
7.38
7.36
7.26
3.00
2.99
2.98
2.97
2.96
2.95
2.47
2.45
2.03
2.01
1.99
1.97
1.80
3.96
2.08
1.07
2.00
2.05
2.03
O
F
f1 (ppm)

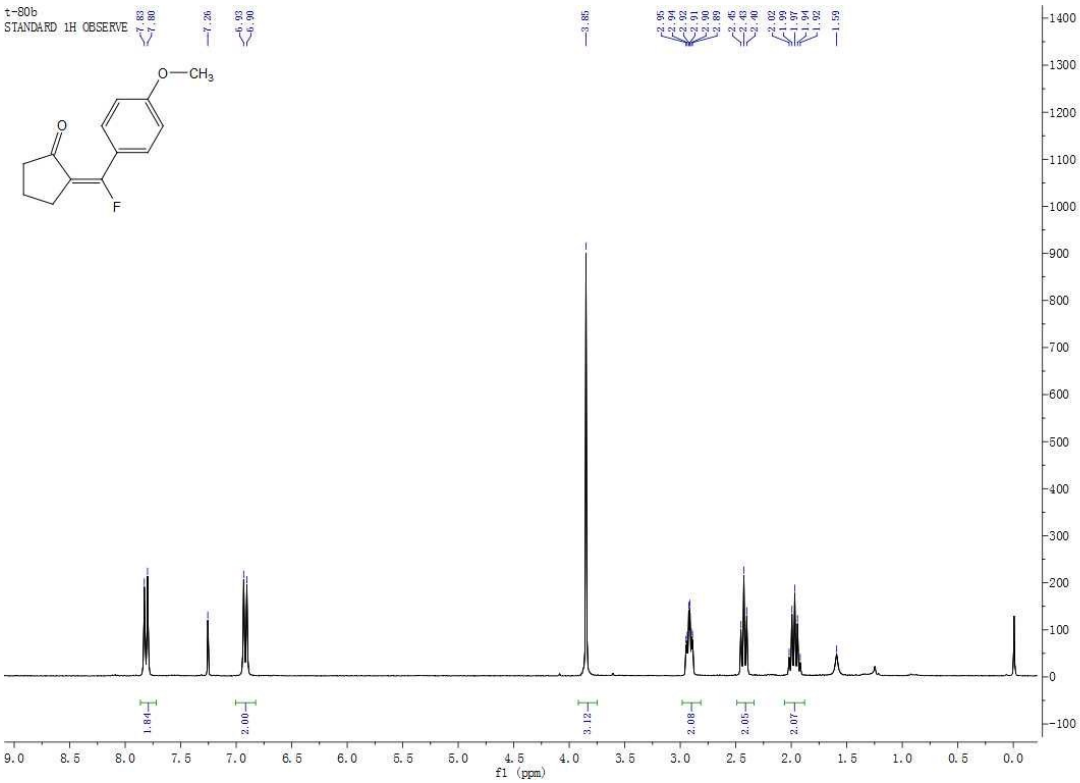
^{13}C-2c


t-93-2a-C13
204.91
163.58
143.81
140.23
129.22
129.15
128.85
127.88
127.20
117.55
108.43
40.89
40.85
27.83
27.79
19.47
O
F
f1 (ppm)

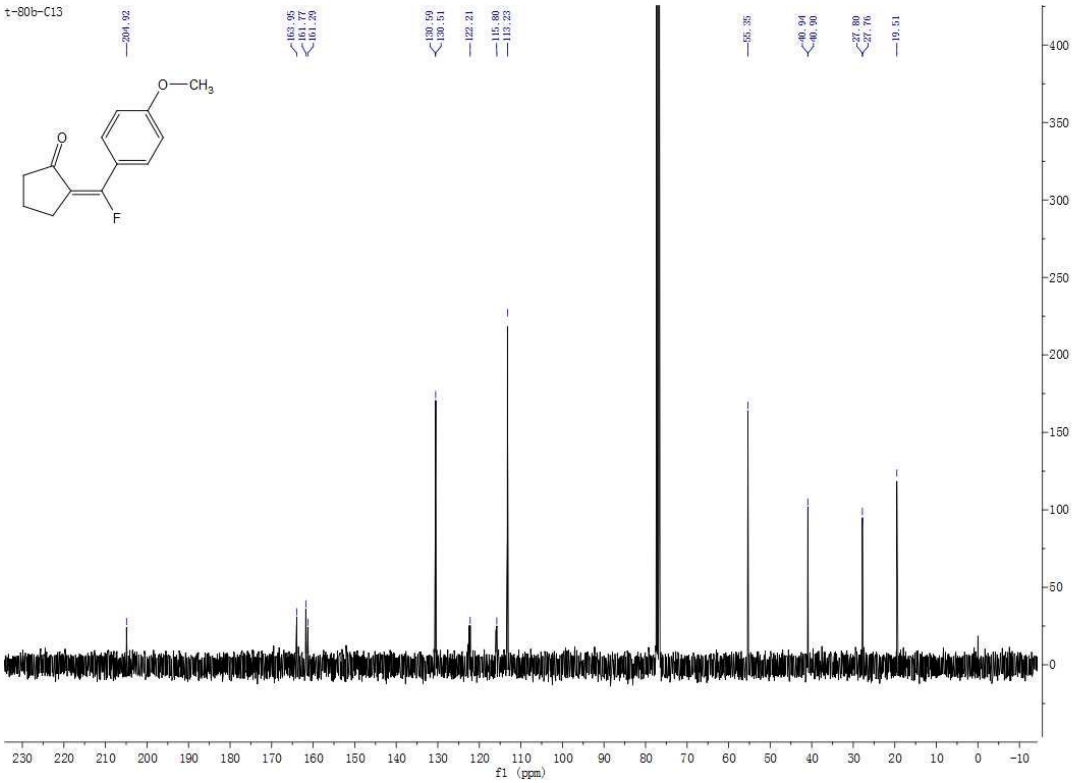
^{19}F-2c


t-93-2a-F19
77.84
O
F
f1 (ppm)

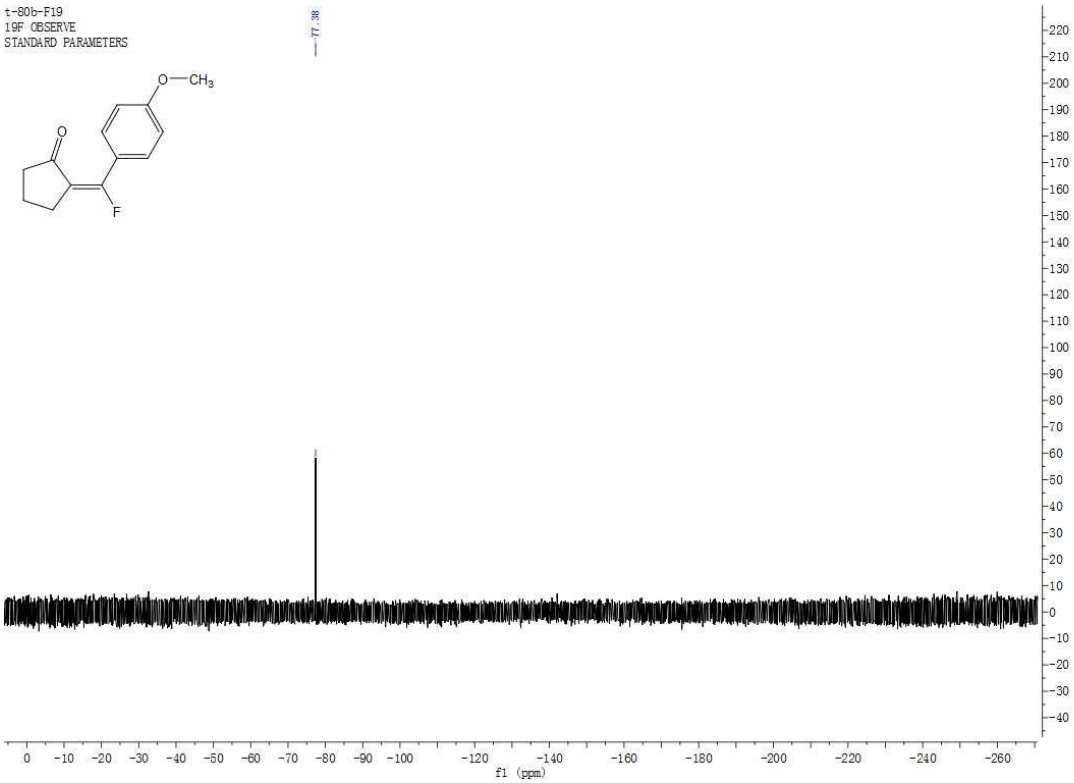
^{1}H-2d



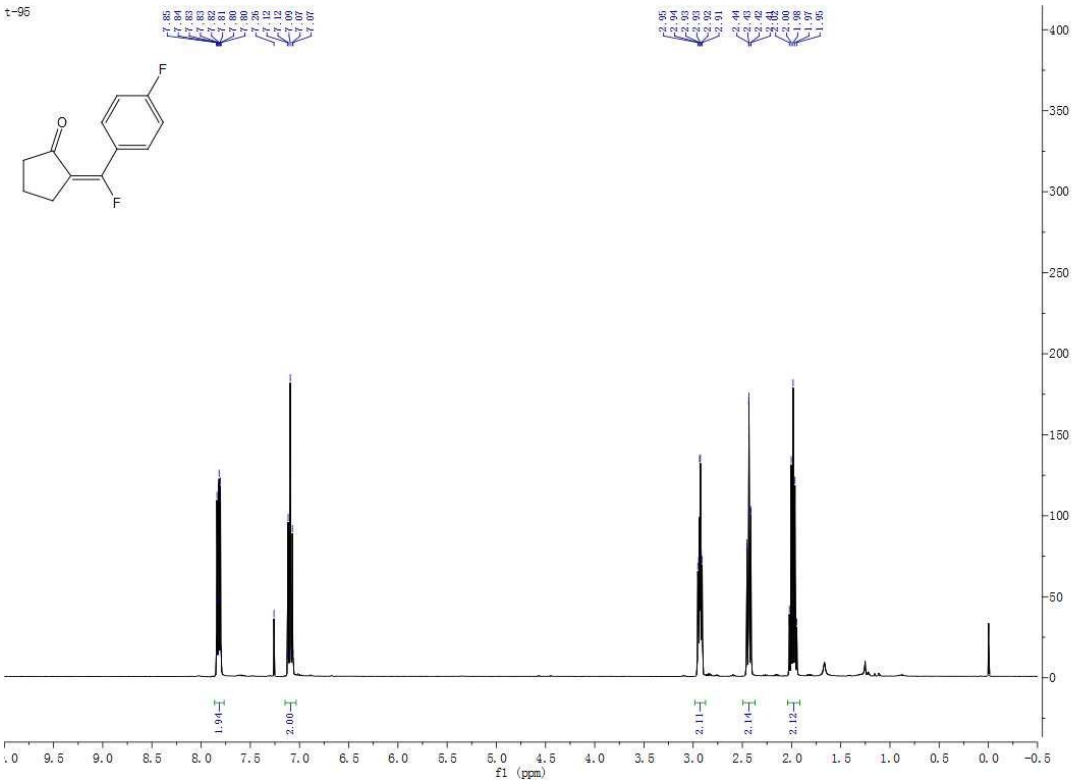
^{13}C-2d



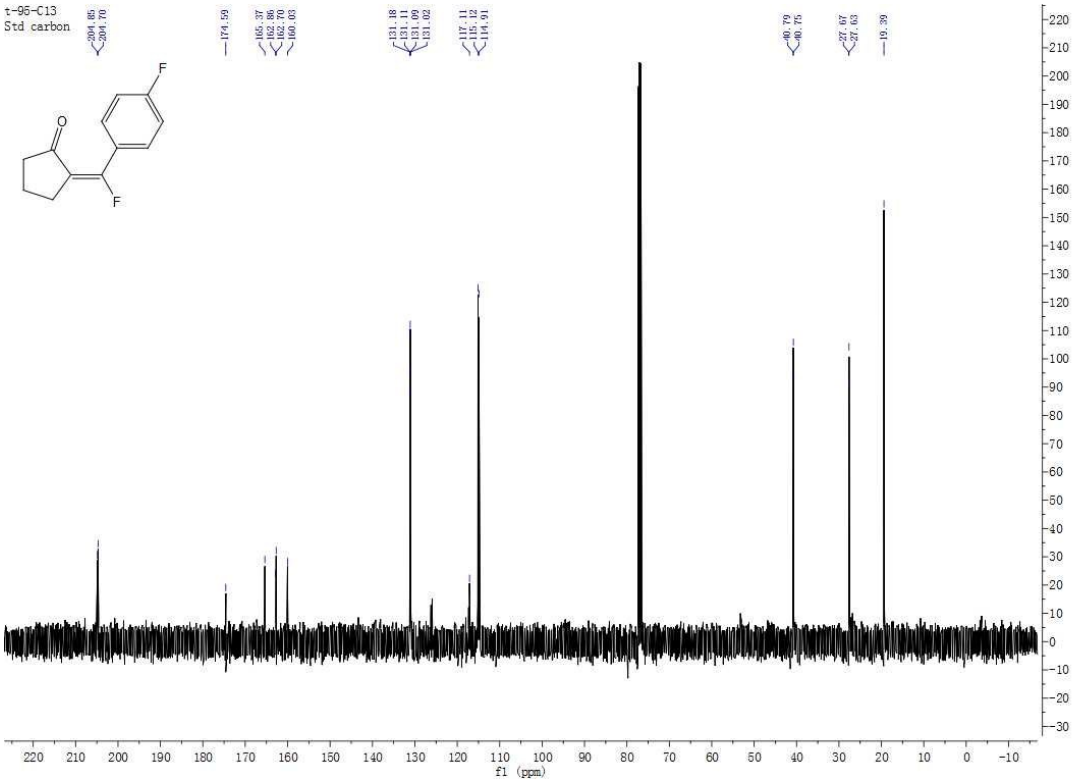
^{19}F-2d


t-80b-F19
19F OBSERVE
STANDARD PARAMETERS
-77.38
O—CH3
O
F
f1 (ppm)

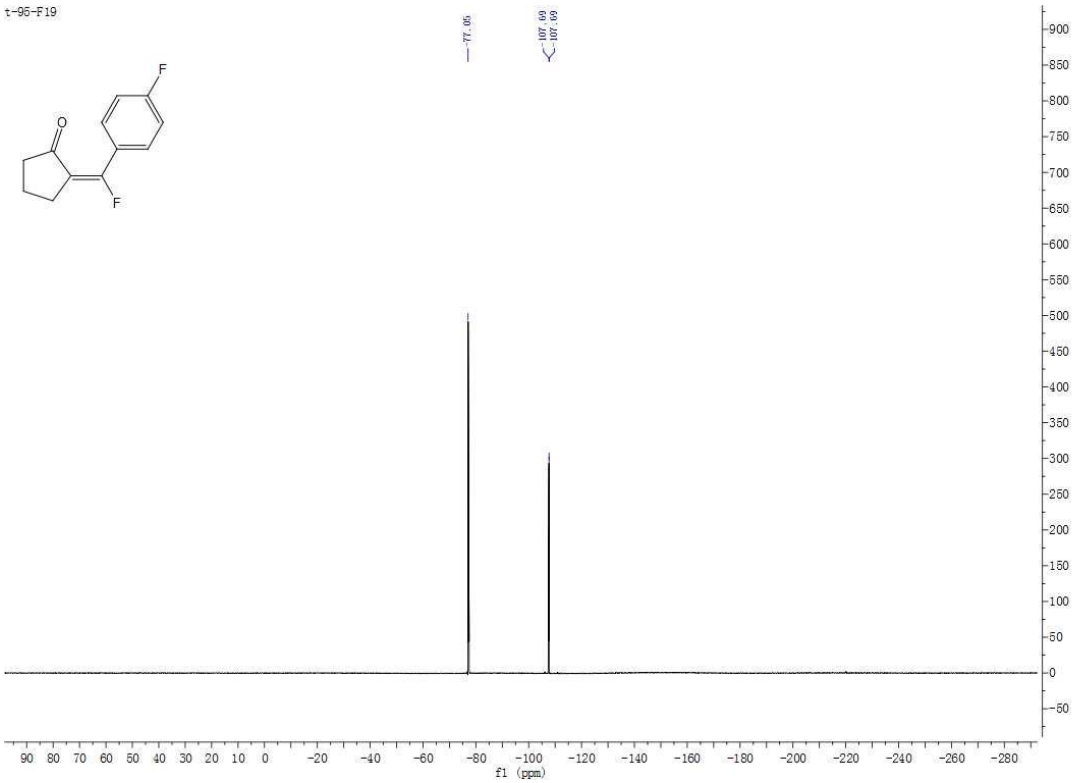
^{1}H-2e


t-95
F
O
F
1.94
2.00
2.11
2.14
2.12
f1 (ppm)

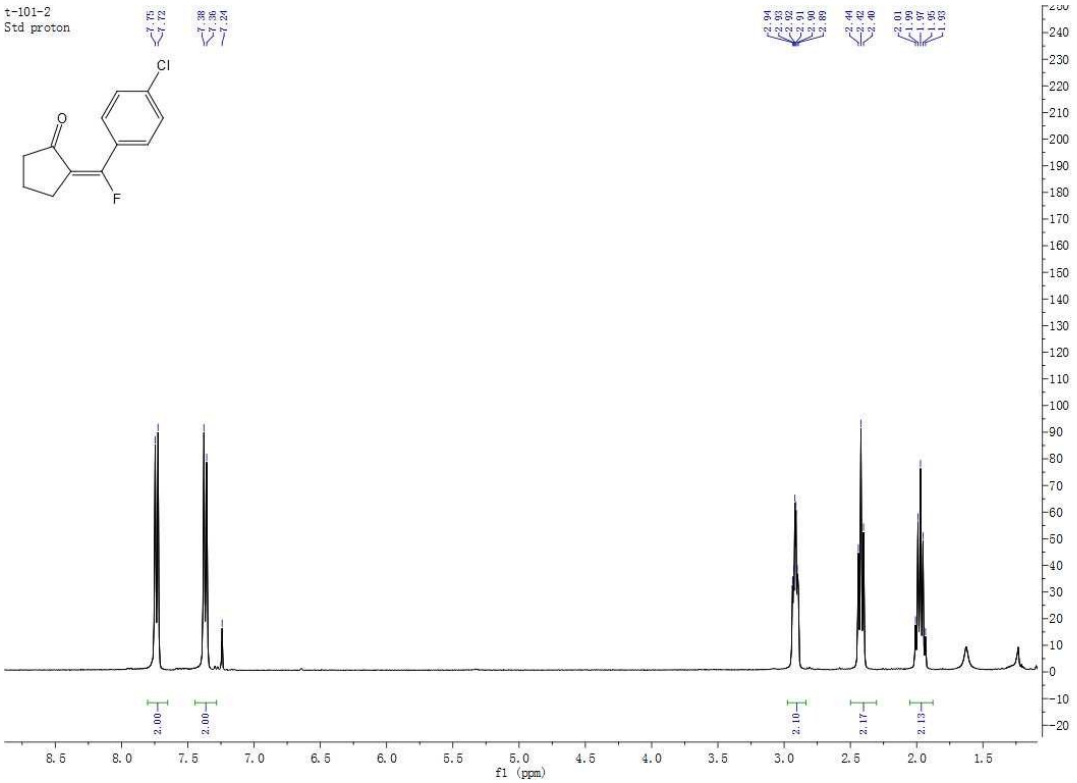
^{13}C-2e


t-96-C13
Std carbon
204.85
204.70
174.59
165.37
162.86
162.70
160.03
131.18
131.11
131.09
131.02
117.11
115.12
114.91
40.79
40.75
27.67
27.63
19.39
F
O
F
f1 (ppm)

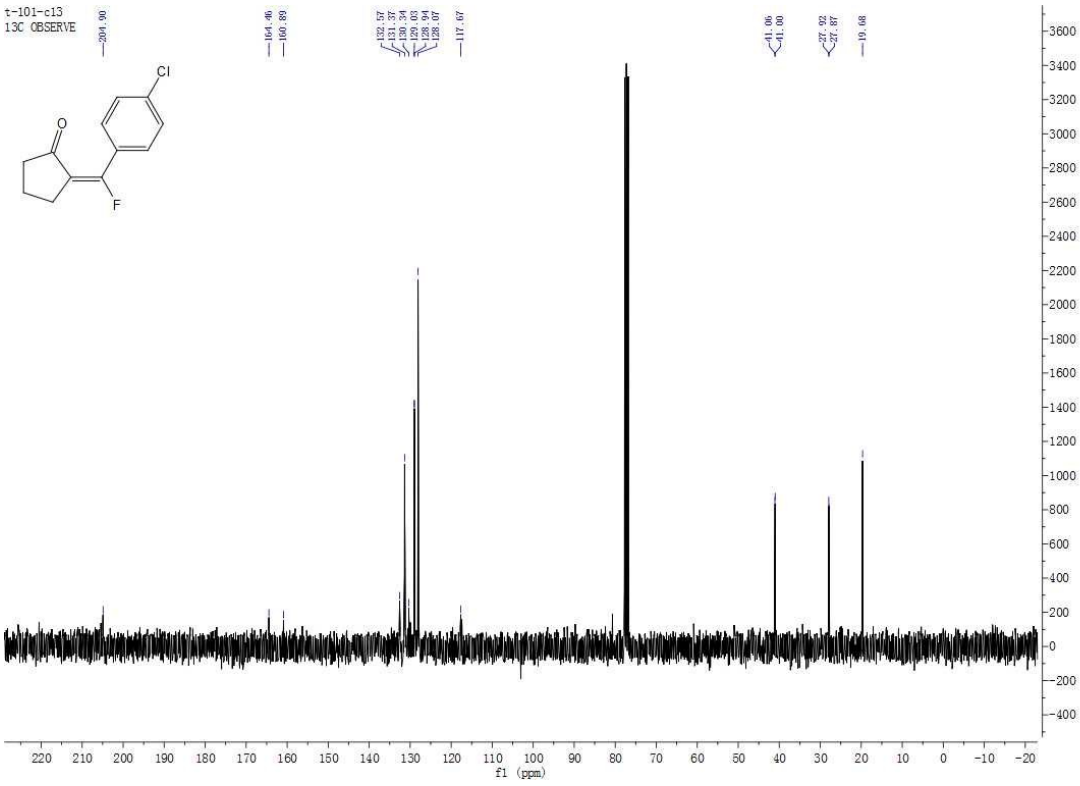
^{19}F-2e


t-96-F19
77.05
107.69
107.69
F
O
F
f1 (ppm)

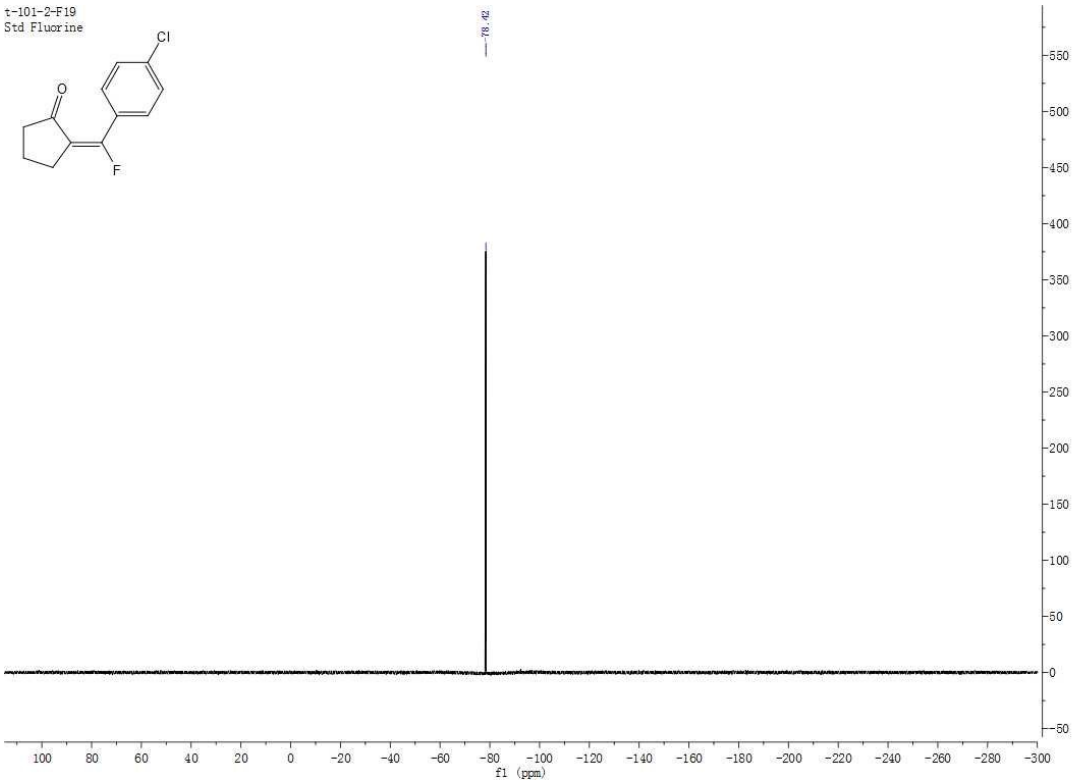
^{1}H-2f



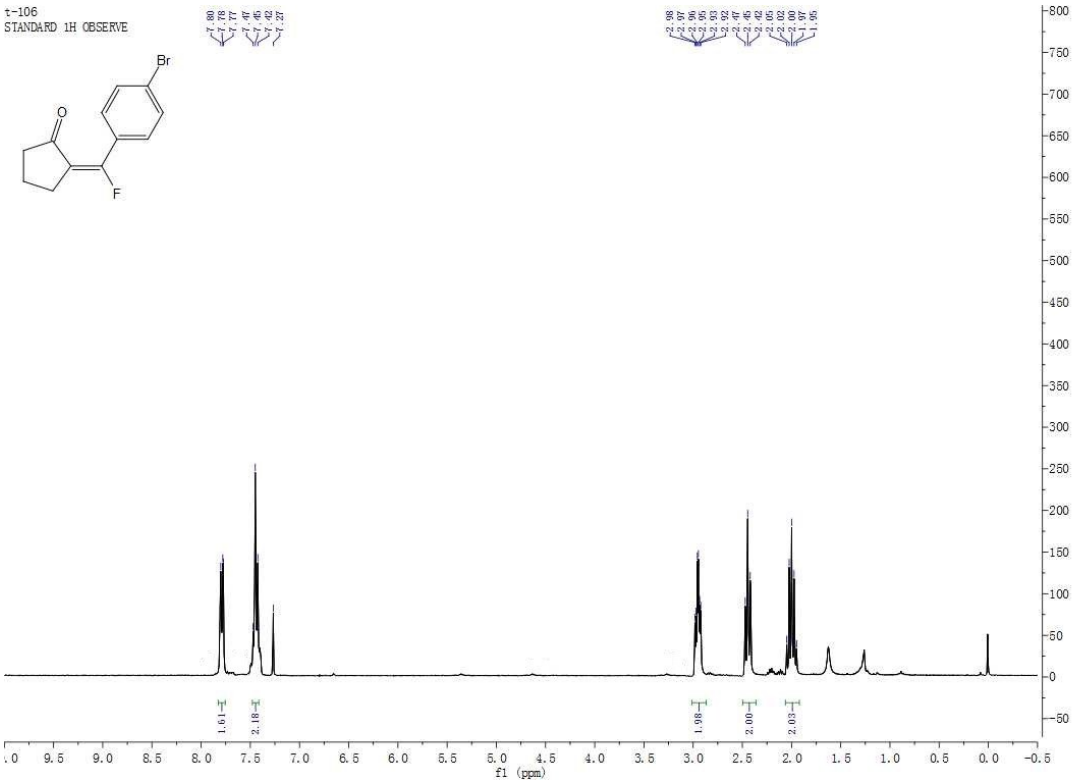
^{13}C-2f



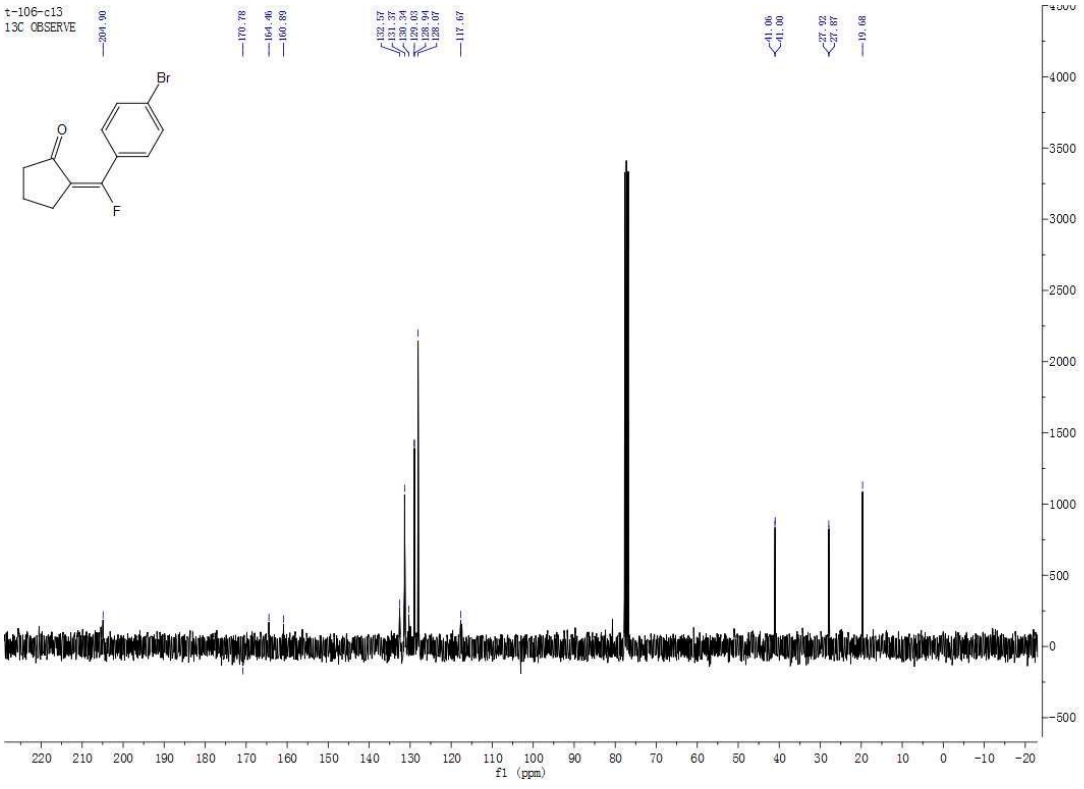
^{19}F-2f


t-101-2-F19
Std Fluorine
Cl
O
F
-78.42
f1 (ppm)

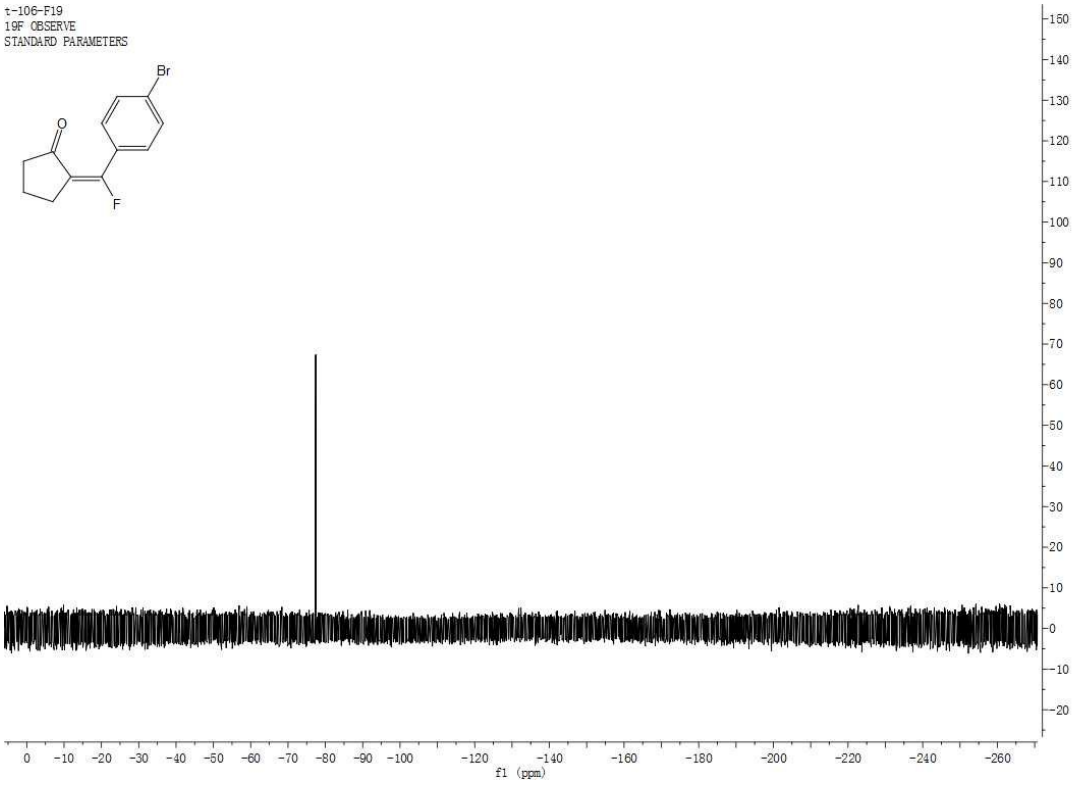
^{1}H-2g


t-106
STANDARD 1H OBSERVE
Br
O
F
f1 (ppm)

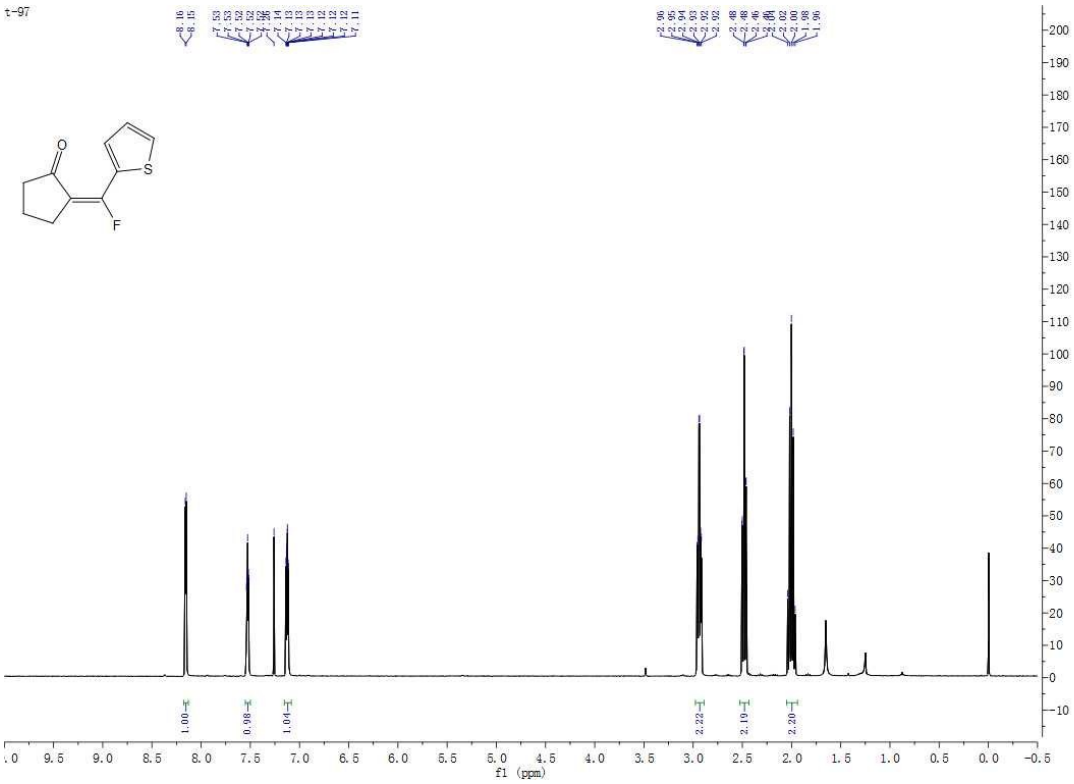
^{13}C-2g


t-106-c13
13C OBSERVE
204.90
170.78
164.46
160.89
122.57
131.37
130.34
129.03
128.94
128.07
117.67
41.06
41.00
27.92
27.87
19.68
Br
O
F
f1 (ppm)

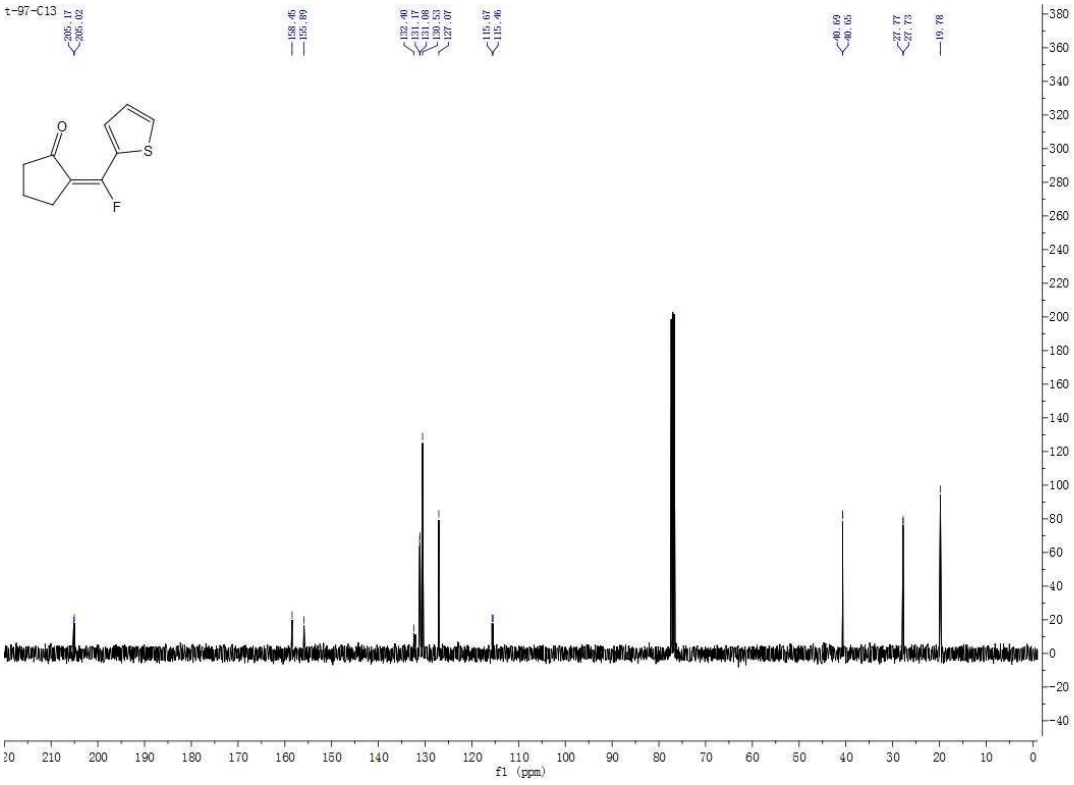
^{19}F-2g


t-106-F19
19F OBSERVE
STANDARD PARAMETERS
Br
O
F
f1 (ppm)

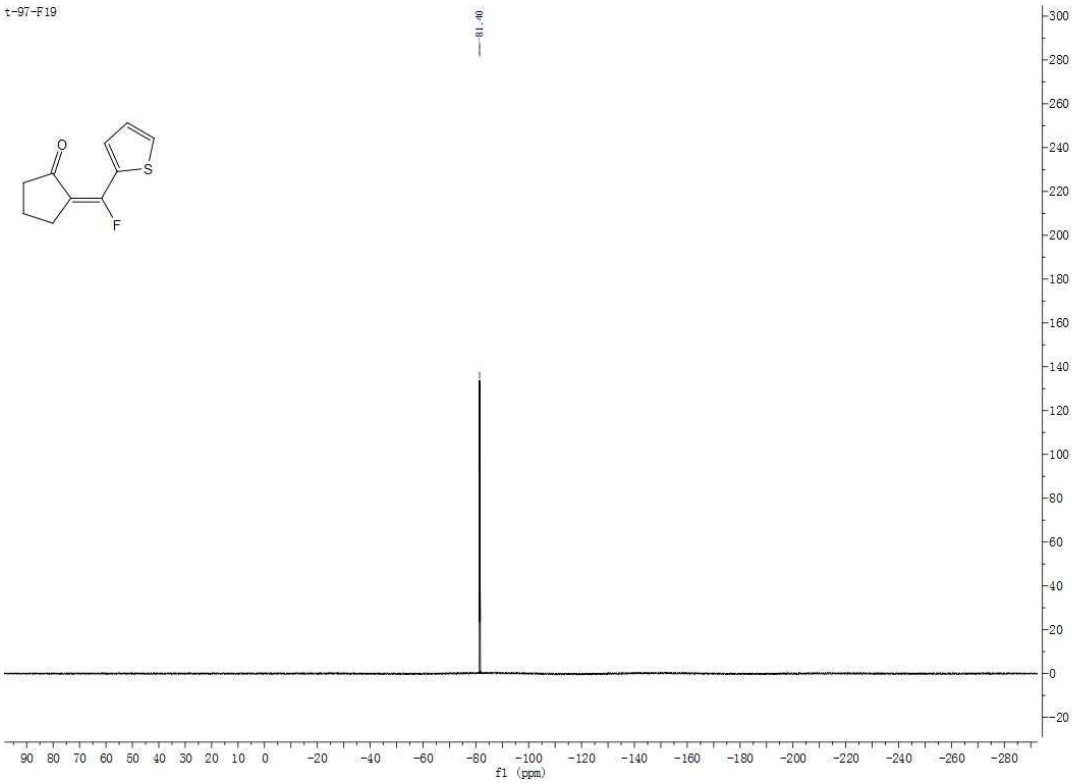
^{1}H-2i



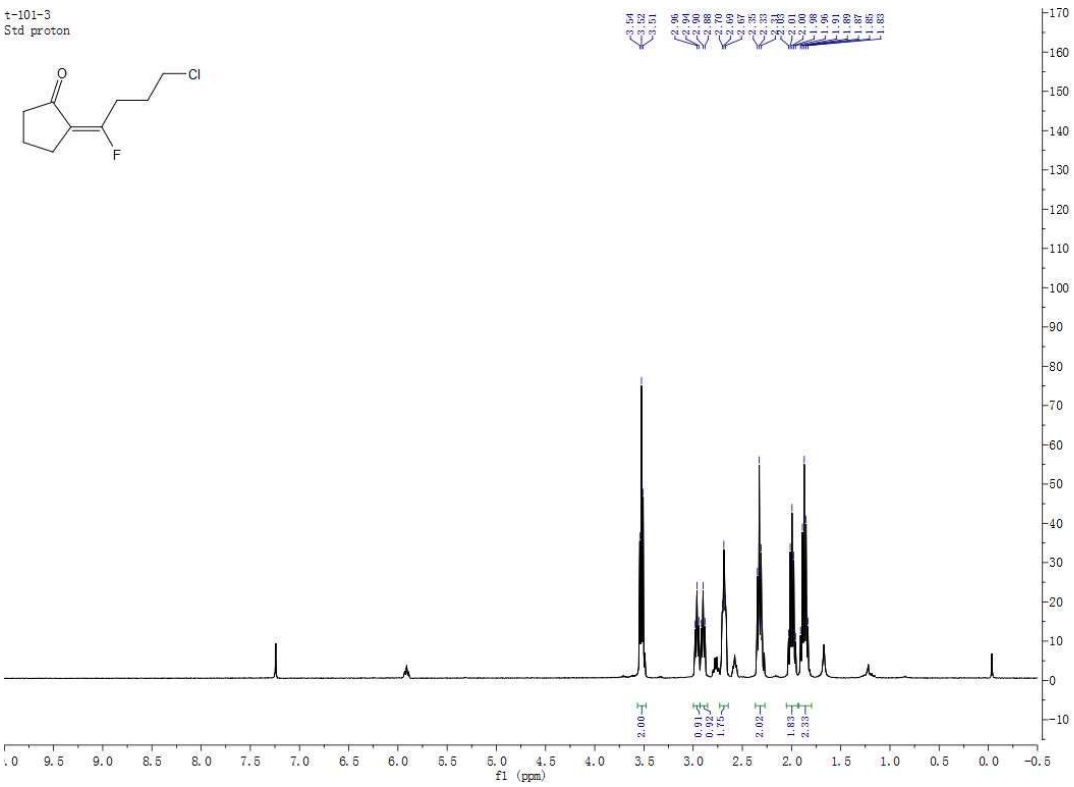
^{13}C-2i



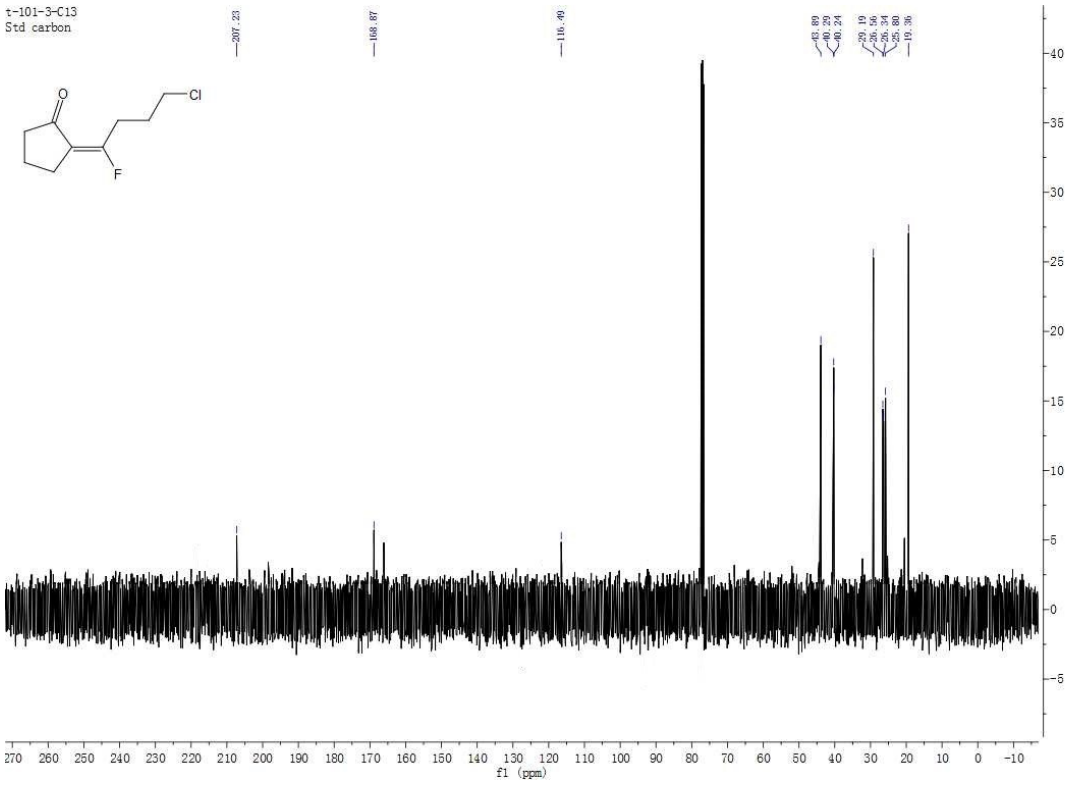
^{19}F-2i



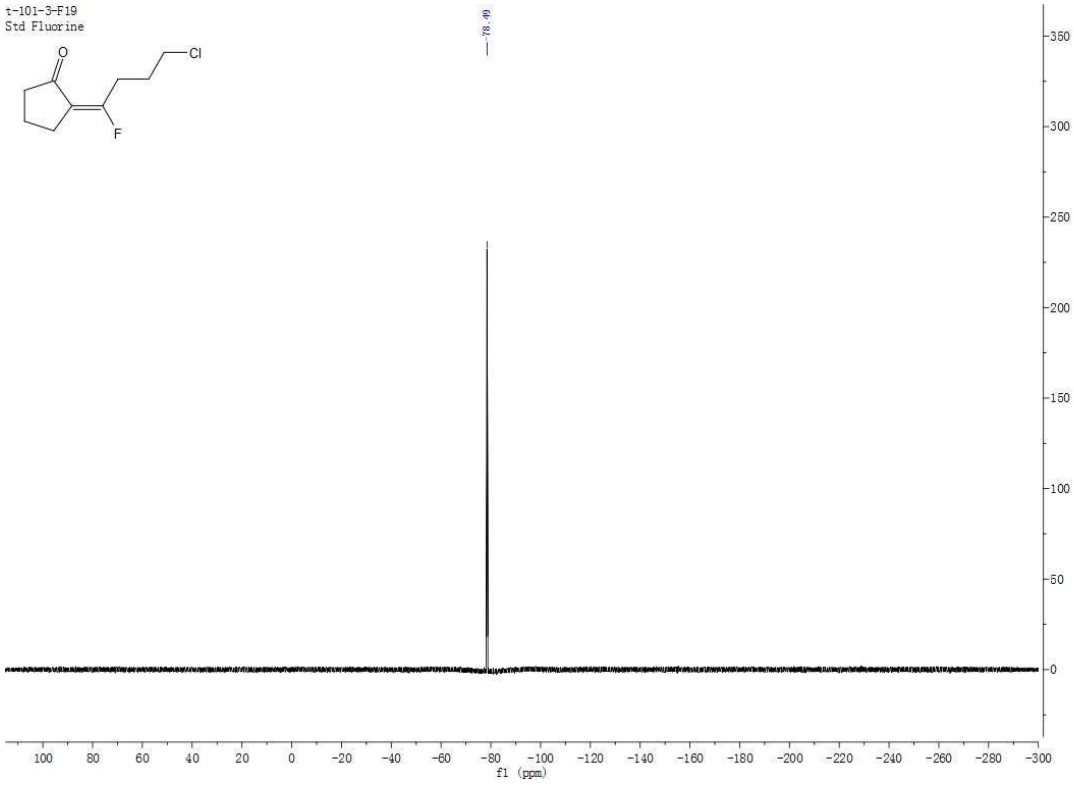
^{1}H-2j



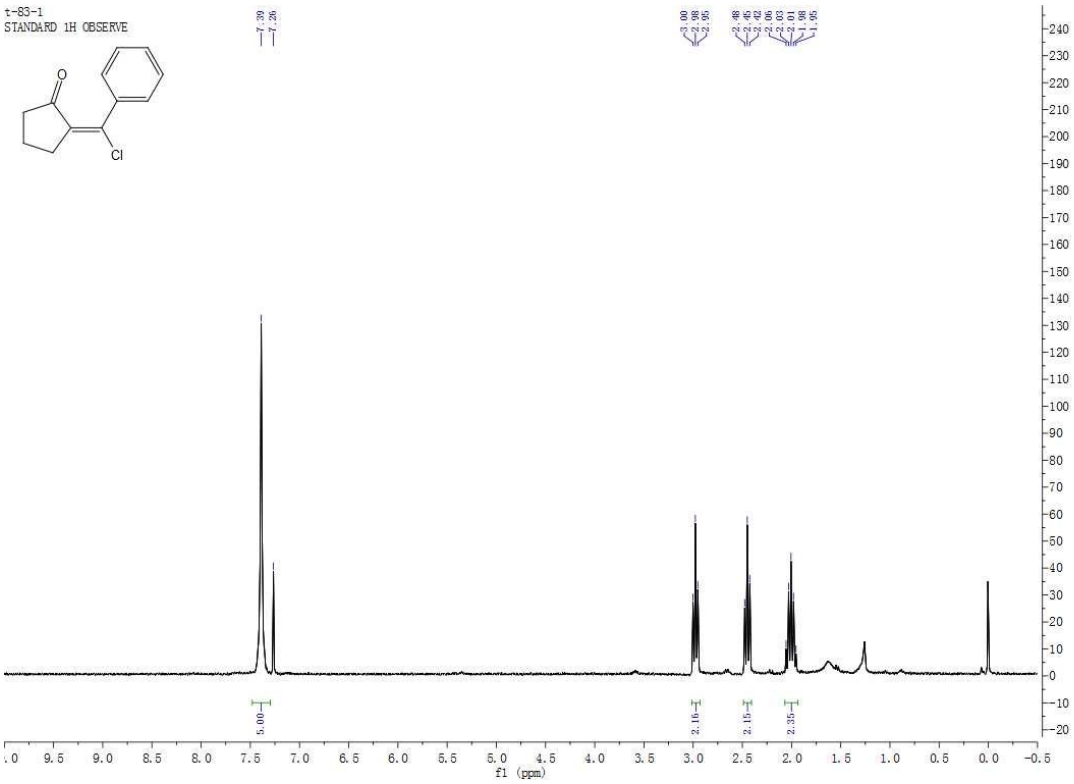
^{13}C-2j


t-101-3-C13
Std carbon
207.23
168.87
116.49
62.89
60.29
60.24
29.19
26.56
26.34
25.80
19.36
O
Cl
F
f1 (ppm)

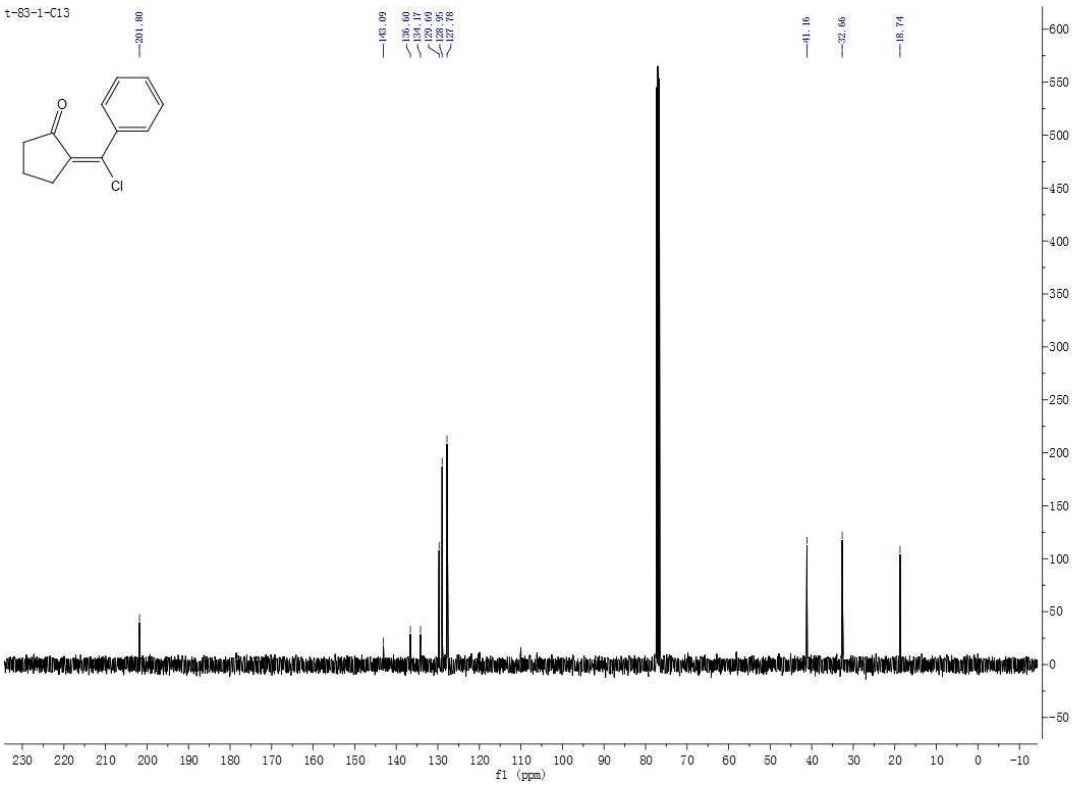
^{19}F-2j


t-101-3-F19
Std Fluorine
78.49
O
Cl
F
f1 (ppm)

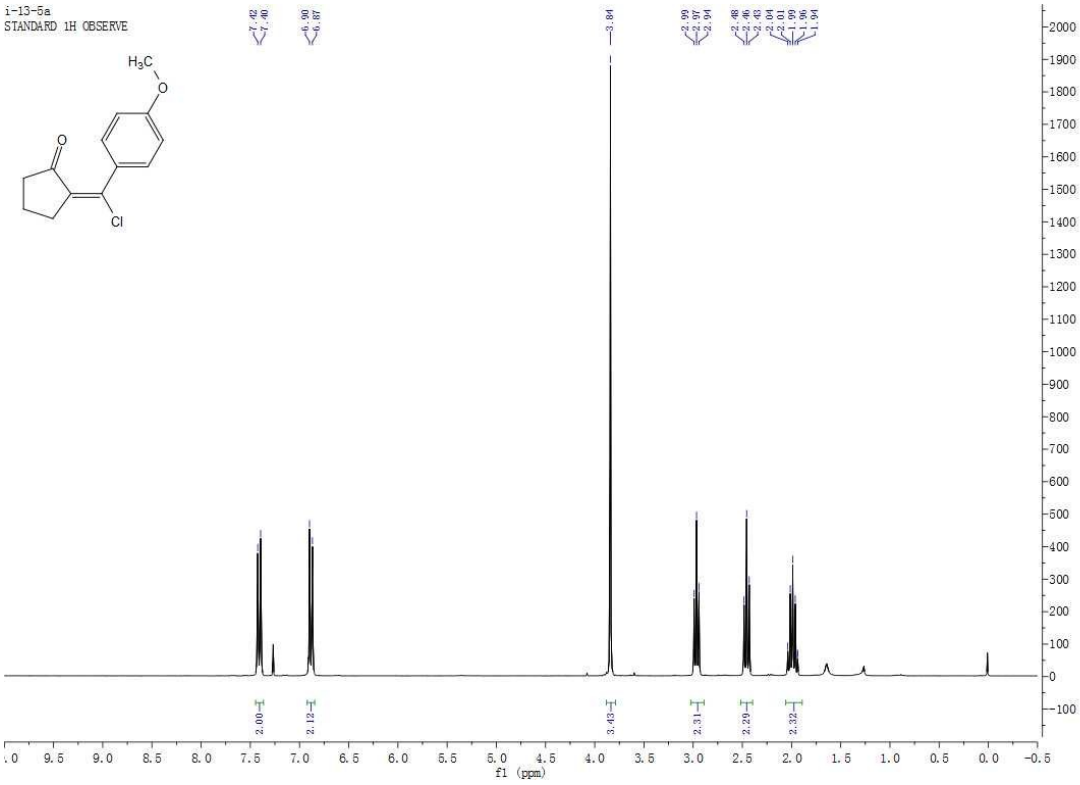
^{1}H-3a


t-83-1
STANDARD 1H OBSERVE
O
Cl
7.39
7.26
3.00
2.98
2.95
2.48
2.45
2.42
2.06
2.03
2.01
1.98
1.95
5.00
2.16
2.15
2.35
f1 (ppm)

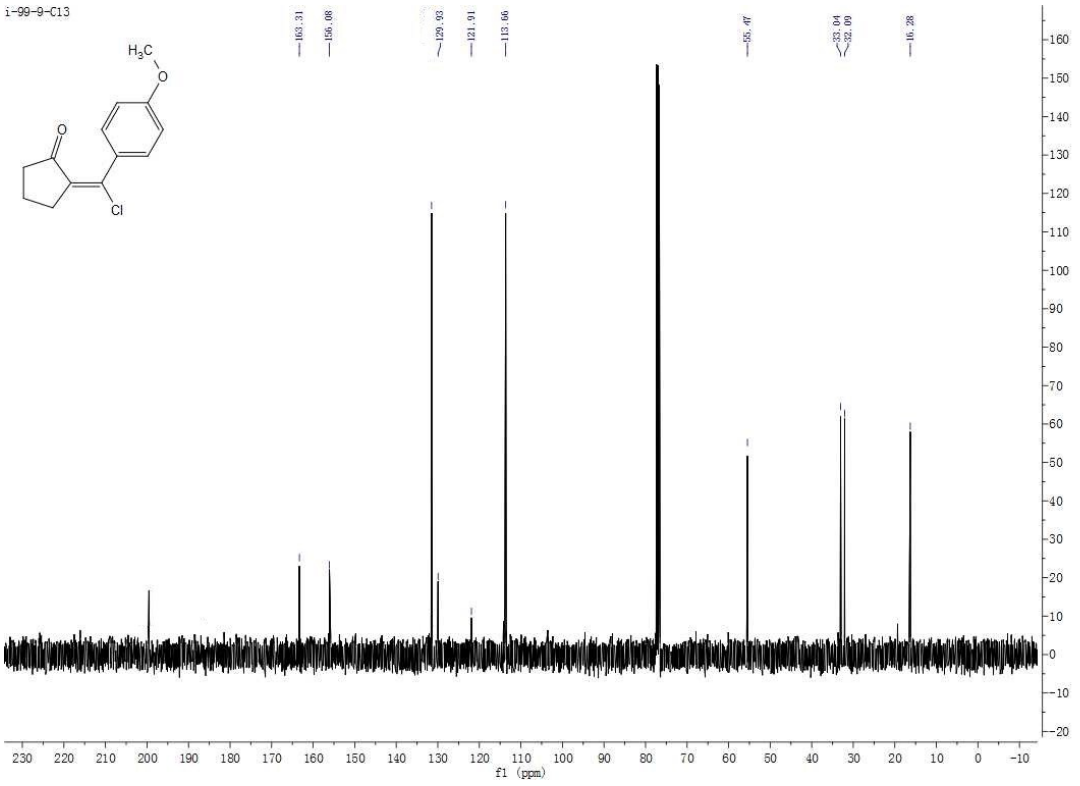
^{13}C-3a


t-83-1-C13
O
Cl
201.80
143.09
136.60
134.17
129.69
128.95
127.78
41.16
32.66
18.74
f1 (ppm)

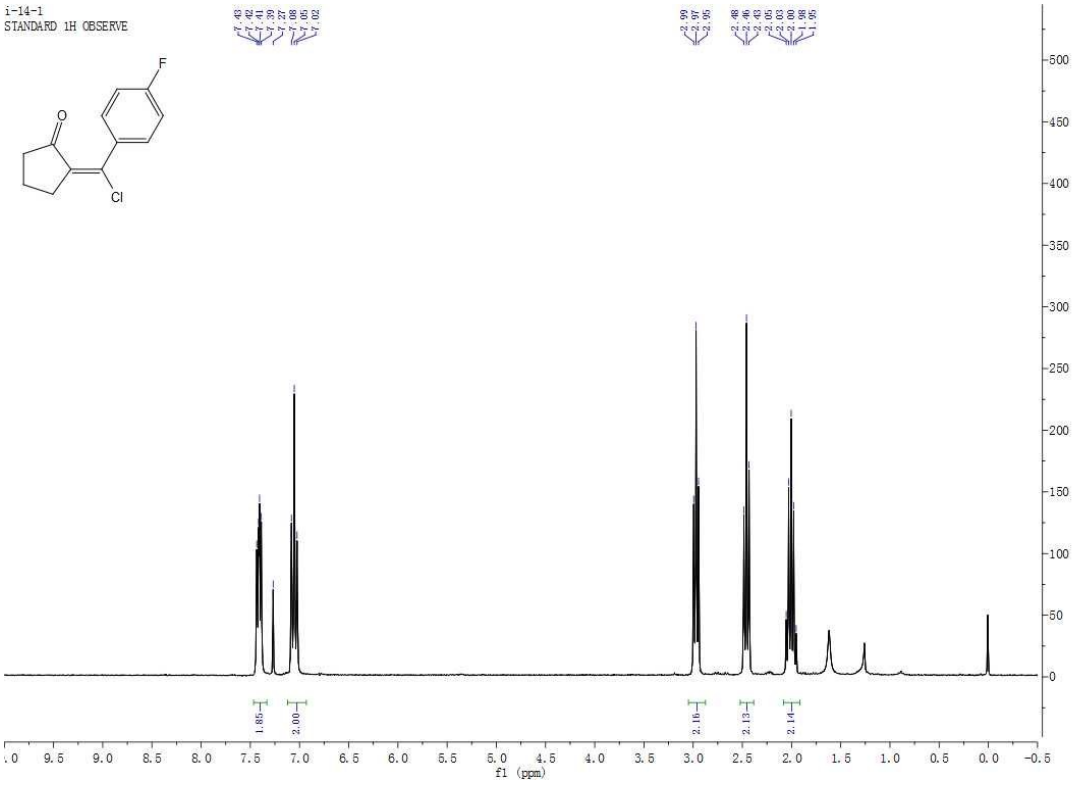
^{1}H-3b


i-13-5a
STANDARD 1H OBSERVE
H3C
O
O
Cl
7.42
7.40
6.90
6.87
3.84
2.99
2.97
2.94
2.48
2.46
2.45
2.04
2.01
1.99
1.96
1.94
2.00
2.12
3.43
2.31
2.29
2.32
f1 (ppm)

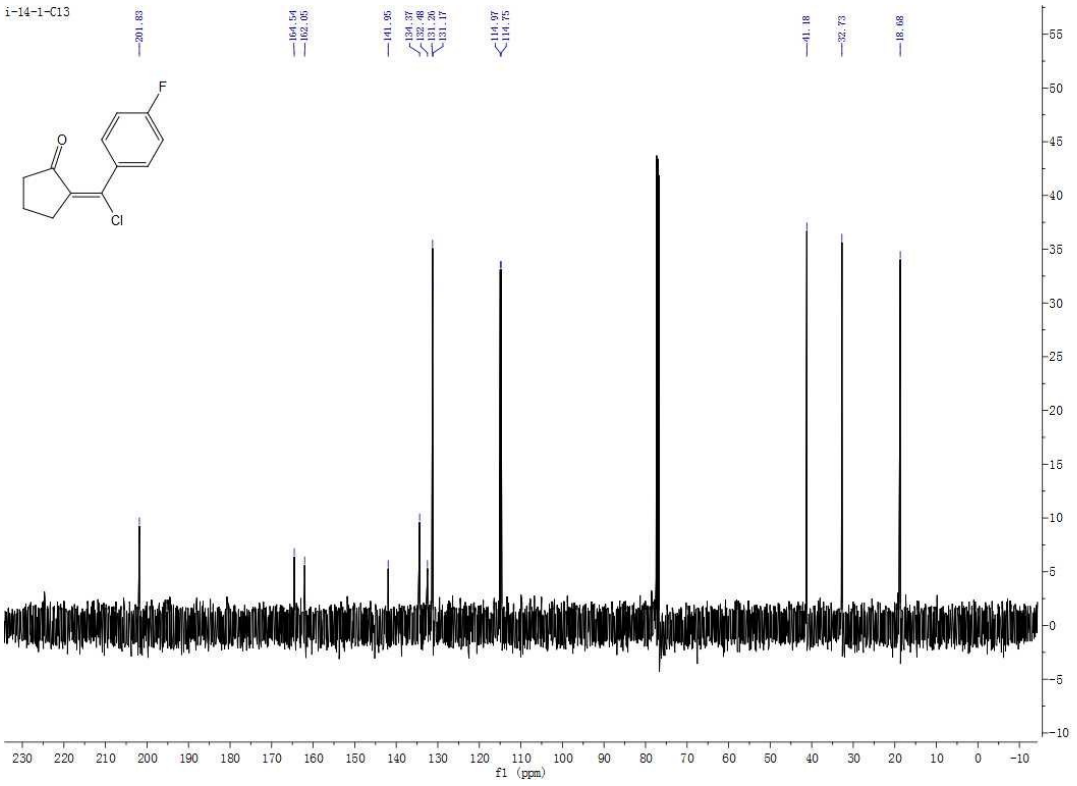
^{13}C-3b


i-99-9-C13
H3C
O
O
Cl
163.31
156.08
129.93
121.91
113.66
55.47
33.04
32.00
16.28
f1 (ppm)

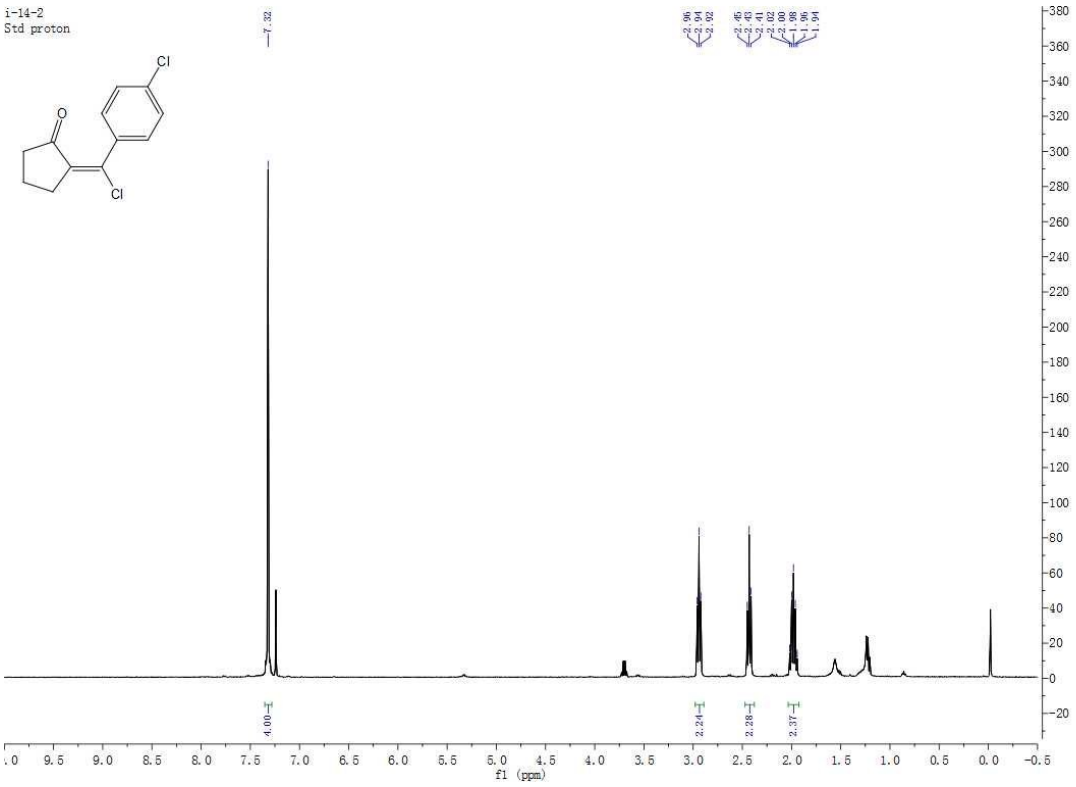
^{1}H-3c



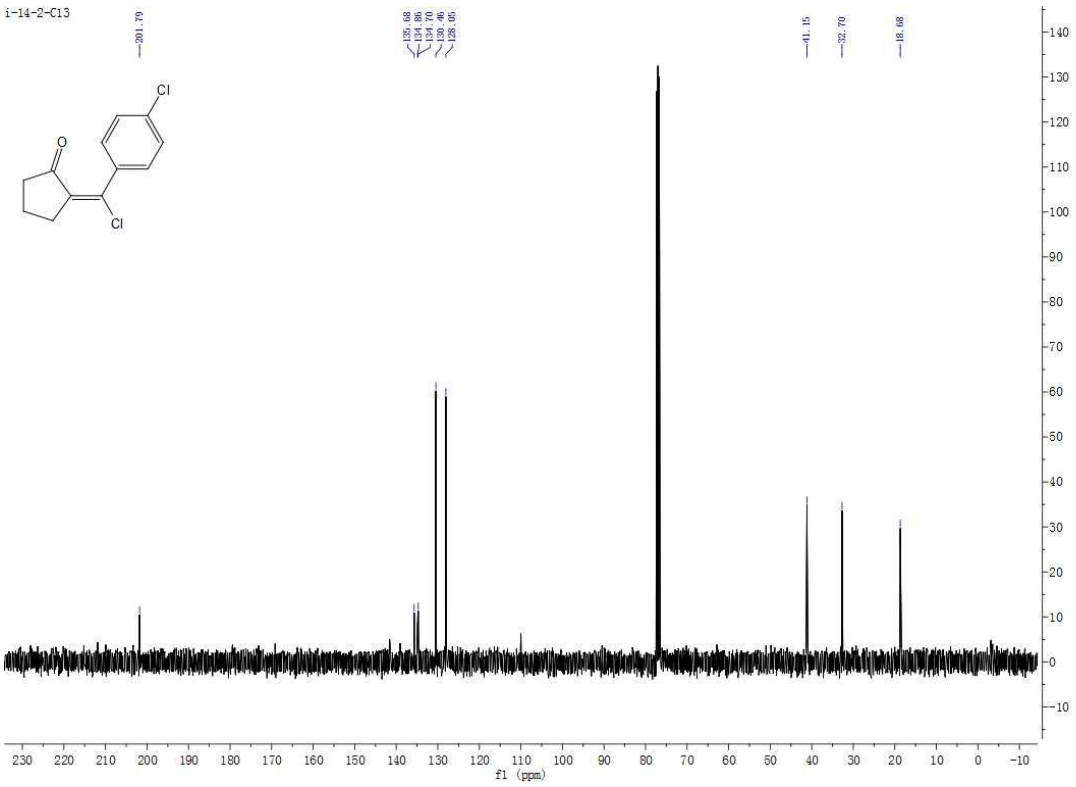
^{13}C-3c



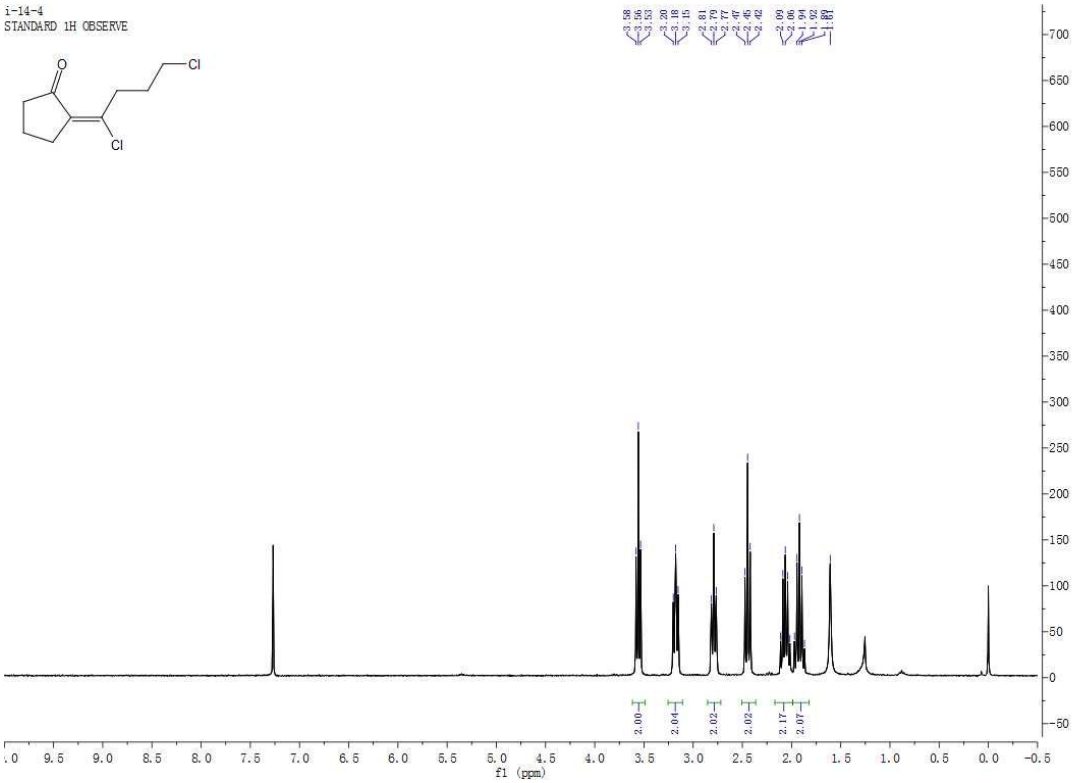
^{1}H-3d


i-14-2
Std proton
Cl
O
Cl
7.32
2.96
2.94
2.92
2.45
2.43
2.41
2.03
2.00
1.98
1.96
1.94
4.00
2.24
2.28
2.37
f1 (ppm)

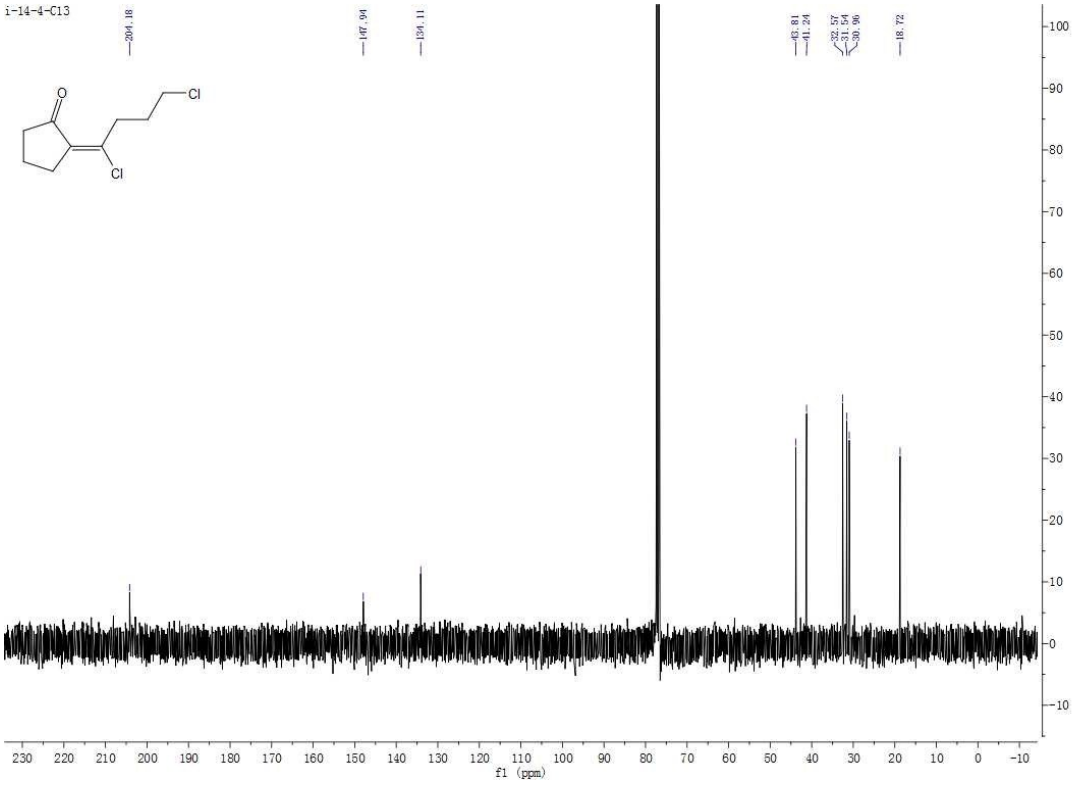
^{13}C-3d


i-14-2-C13
201.79
135.68
134.86
134.70
130.46
128.05
41.15
32.70
18.68
Cl
O
Cl
f1 (ppm)

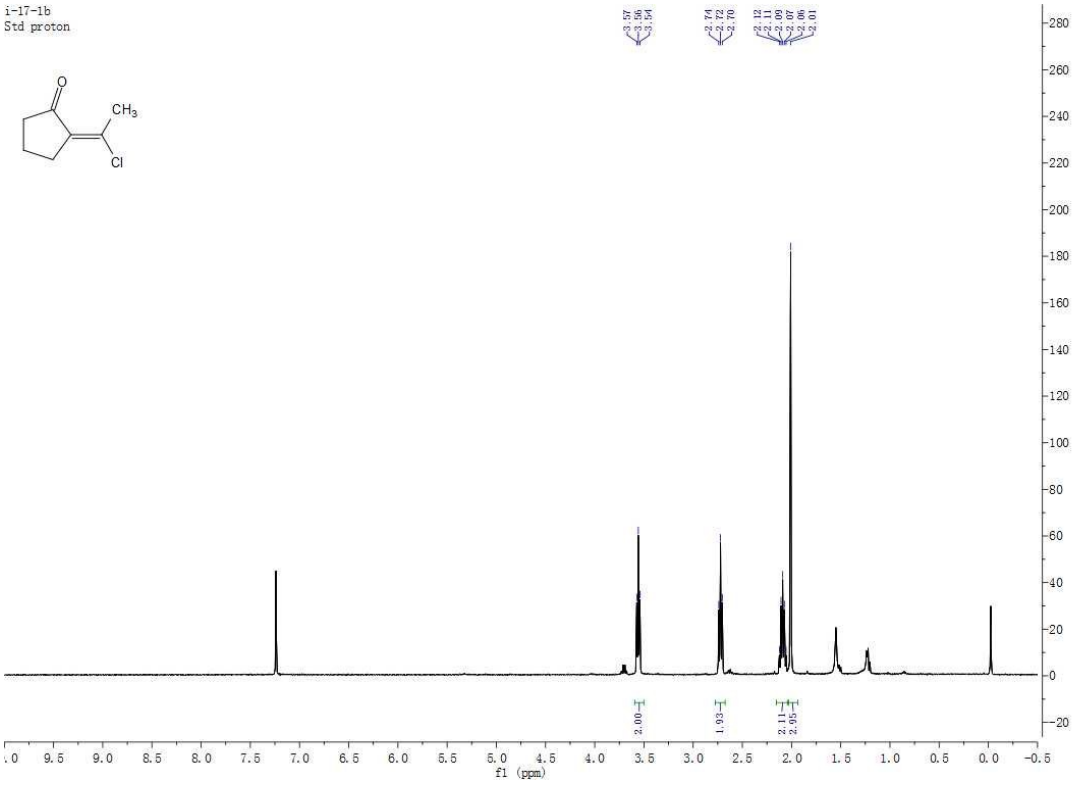
^{1}H-3e


i-14-4
STANDARD 1H OBSERVE
O
Cl
Cl
f1 (ppm)

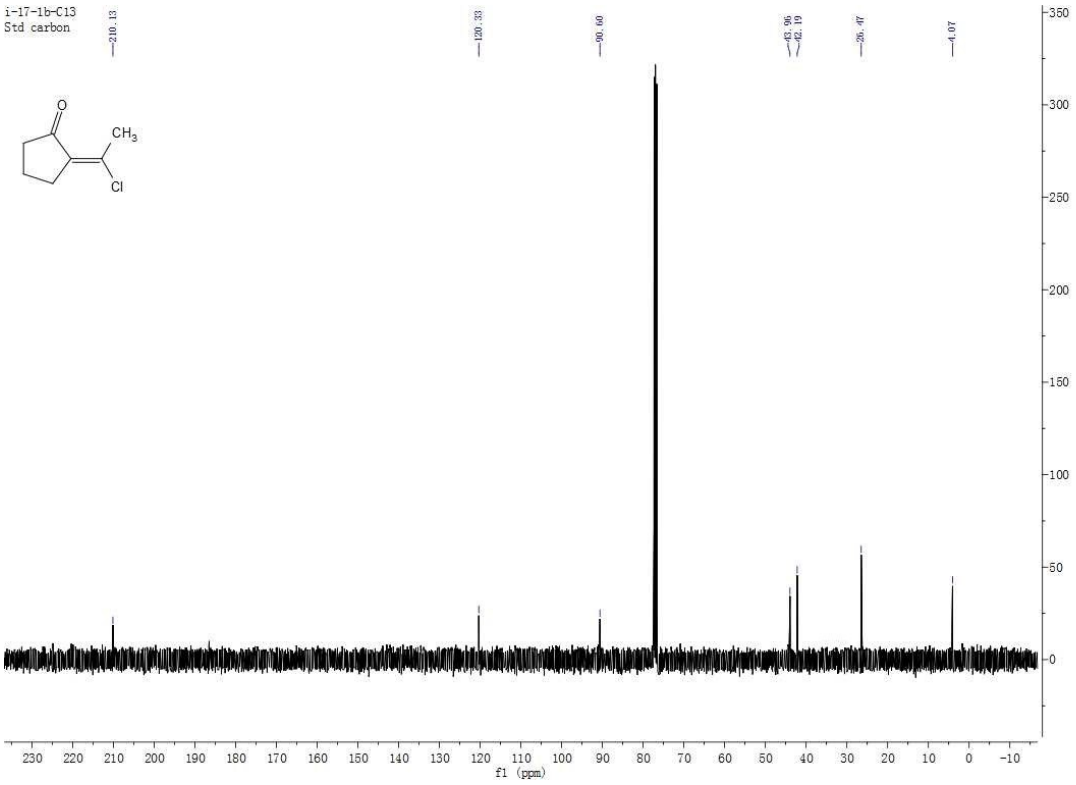
^{13}C-3e


i-14-4-C13
204.18
147.94
134.11
63.81
41.24
32.57
31.54
30.96
18.72
O
Cl
Cl
f1 (ppm)

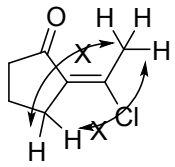
^{1}H-3f

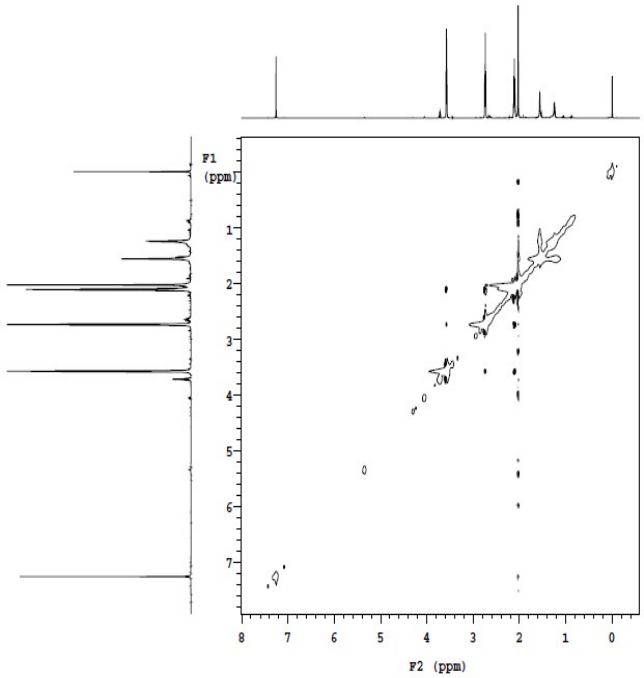


^{13}C-3f

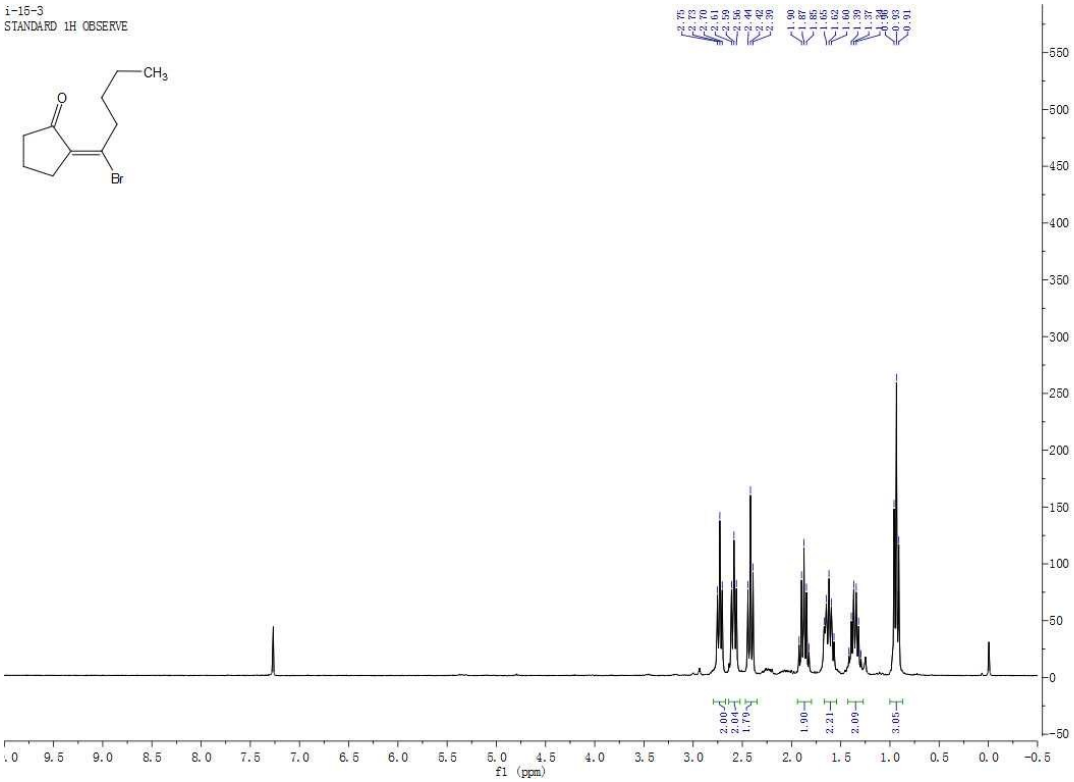


NOESY-3f

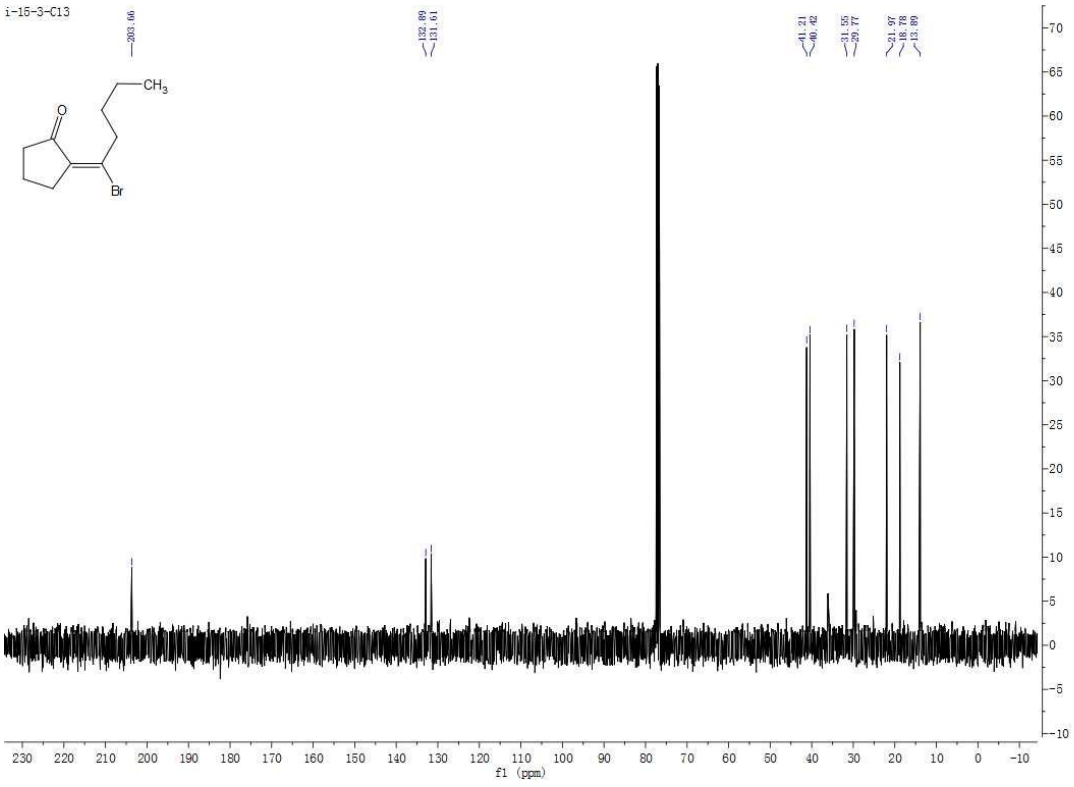

O
H
H
X
H
Cl
X
H
H


F1 (ppm)
F2 (ppm)

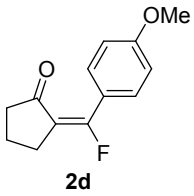
^{1}H-3g


i-15-3
STANDARD 1H OBSERVE
CH3
O
Br
f1 (ppm)

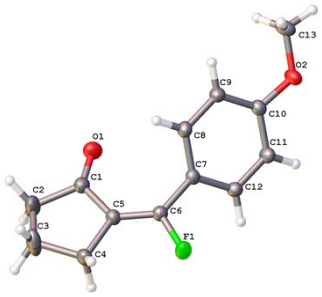
^{13}C-3g


i-16-3-C13
203.66
132.89
131.61
41.21
40.42
31.55
29.77
21.97
18.78
13.89
O
CH3
Br
f1 (ppm)

Crystallography data



2d



2d

Identification code	dm16644	
Empirical formula	C13 H13 F O2	
Formula weight	220.23	
Temperature	130 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 14.2255(14) Å	α= 90°.
	b = 6.0009(6) Å	β= 114.3980(10)°.
	c = 13.9246(13) Å	γ = 90°.
Volume	1082.53(18) $Å^3$	
Z	4	
Density (calculated)	1.351 Mg/m^3	
Absorption coefficient	0.101 mm^{-1}	
F(000)	464	
Crystal size	0.22 x 0.2 x 0.15 mm^3	
Theta range for data collection	2.936 to 30.588°.	
Index ranges	-20<=h<=20, -7<=k<=8, -19<=l<=19	
Reflections collected	10460	
Independent reflections	3317 [R(int) = 0.0210]	
Completeness to theta = 26.000°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.6860	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3317 / 0 / 146	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0552, wR2 = 0.1464	
R indices (all data)	R1 = 0.0660, wR2 = 0.1576	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.561 and -0.589 $e.Å^{-3}$	