

Supporting Information

Silver-Catalyzed Ring-Opening Trifluoromethylthiolation/ Difluoromethylthiolation of Cycloalkanols with PhSO₂SCF₂H or PhSO₂SCF₃

Bin Xu,^a Decai Wang,^a Yonghong Hu^{*a} and Qilong Shen^{*b}

^aBiotechnology and Pharmaceutical Engineering, Nanjing Tech University, Nanjing,
Jiangsu Province, 210009, P. R. China

^bKey Laboratory of Organofluorine Chemistry, Center for Excellence in Molecular
Synthesis, Shanghai Institute of Organic Chemistry, University of Chinese Academy
Sciences, Chinese Academy of Sciences, 345 Lingling Road, Shanghai 200032, P. R.
China

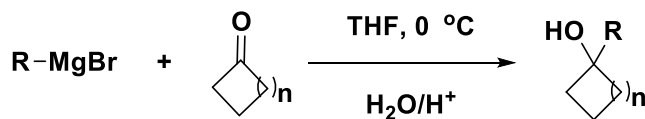
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General information. All reactions were maintained under an argon atmosphere unless otherwise stated. All solvents were purified by standard method. ^1H , ^{13}C and ^{19}F NMR spectra were acquired on 400 MHz, 125 MHz, 100 MHz, 375 MHz spectrometer (400 MHz for ^1H ; 100 MHz or 125 MHz for ^{13}C ; 375 MHz for ^{19}F). ^1H NMR and ^{13}C NMR chemical shifts were determined relative to internal standard TMS at δ 0.0 ppm and ^{19}F NMR chemical shifts were determined relative to CFCl_3 as inter standard. Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. All reactions were monitored by TLC or ^{19}F NMR. Flash column chromatograph was carried out using 300-400 mesh silica gel at medium pressure.

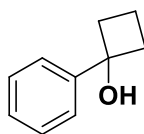
Materials. Cyclic alcohols (**3a**, **3c**, **3f**) were received from commercial sources. Solvents were freshly dried and degassed according to the purification handbook *Purification of Laboratory Chemicals* before using.

General procedure A for the preparation of cycloalkanols



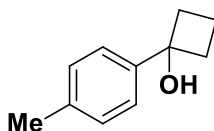
Aryl magnesium bromide (2.0 equiv., 1.0 M in Et₂O) in diethyl ether was added drop wise over 30 min at 0 °C to a solution of cyclic ketones (10 mmol, 1.0 equiv) in THF. The mixture was warmed to room temperature and was stirred overnight. Then the mixture was quenched with acidic water solution. The precipitate was removed by filtration. The filtrate was extracted with diethyl ether (30.0 mL $\times$ 3), washed with water and dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was then purified by column chromatography (eluent: petroleum ether: ethyl acetate = 10:1) to afford the cycloalkanols **1a-e**, **3j**, **3l**, **3n**, **3o** and **3p**.

1-Phenylcyclobutanol **1a**¹



The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-phenylcyclobutanol **1a** (1.1 g, 80%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.55 – 7.46 (m, 2 H), 7.45 – 7.35 (m, 2 H), 7.30 (m, 1 H), 2.69 – 2.46 (m, 2 H), 2.46 – 2.29 (m, 2 H), 2.14 – 1.93 (m, 1 H), 1.79 – 1.60 (m, 1 H); ¹³C NMR (101 MHz, CDCl₃) δ 146.34, 128.46, 127.24, 125.05, 77.01, 36.84, 13.07 ppm.

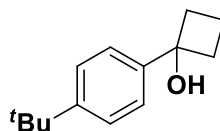
1-(*p*-Tolyl)cyclobutanol **1b**



The general procedure **A** conducted with *p*-tolylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(*p*-tolyl)cyclobutanol **1b** (1.2 g, 72%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.56 – 7.35 (m, 2 H), 7.30 – 7.13

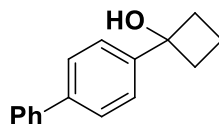
(m, 2 H), 2.81 – 2.47 (m, 2 H), 2.46 – 2.23 (m, 5 H), 2.14 – 1.84 (m, 1 H), 1.83 – 1.54 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 143.39, 136.85, 129.13, 125.13, 76.87, 36.83, 21.13, 13.06 ppm.

1-(4-(*tert*-Butyl)phenyl)cyclobutanol **1c**



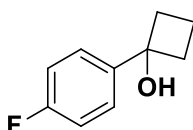
The general procedure **A** conducted with (4-(*tert*-butyl)phenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(4-(*tert*-butyl)phenyl)cyclobutanol **1c** (1.4 g, 70%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.63 – 7.29 (m, 4 H), 2.78 – 2.46 (m, 2 H), 2.48 – 2.26 (m, 2 H), 2.21 – 1.83 (m, 2 H), 1.89 – 1.45 (m, 1 H), 1.32 (d, J = 15.8 Hz, 9 H); ^{13}C NMR (101 MHz, CDCl_3) δ 150.20, 143.28, 125.37, 124.75, 76.87, 36.79, 34.51, 31.38, 13.02 ppm.

1-([1,1'-Biphenyl]-4-yl)cyclobutanol **1d**



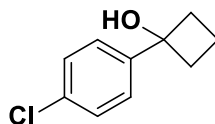
The general procedure **A** conducted with [1,1'-biphenyl]-4-ylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-([1,1'-biphenyl]-4-yl)cyclobutanol **1d** (1.5 g, 67%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.68 – 7.53 (m, 6 H), 7.52 – 7.42 (m, 2 H), 7.42 – 7.32 (m, 1 H), 2.72 – 2.53 (m, 2 H), 2.54 – 2.33 (m, 2 H), 2.15 (d, J = 1.9 Hz, 1 H), 2.12 – 1.98 (m, 1 H), 1.86 – 1.66 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 145.32, 140.84, 140.21, 128.82, 127.34, 127.23, 127.14, 125.50, 76.92, 36.99, 13.07 ppm.

1-(4-Fluorophenyl)cyclobutanol **1e**



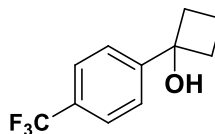
The general procedure **A** conducted with (4-fluorophenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(4-fluorophenyl)cyclobutanol **1e** (0.9 g, 56%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.70 – 7.34 (m, 2 H), 7.03 (m, 2 H), 2.64 – 2.46 (m, 2 H), 2.42 – 2.25 (m, 2 H), 2.17 – 1.80 (m, 1 H), 1.80 – 1.39 (m, 1 H); ^{19}F NMR (376 MHz, CDCl_3) δ -115.68 – -115.76 (m, 1 F); ^{13}C NMR (101 MHz, CDCl_3) δ 161.98 (d, J = 245.5 Hz), 142.10 (d, J = 3.1 Hz), 126.84 (d, J = 8.1 Hz), 115.12 (d, J = 21.2 Hz), 76.61, 37.00, 12.91 ppm.

1-(4-Chlorophenyl)cyclobutanol **1f**



The general procedure **A** conducted with (4-chlorophenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(4-chlorophenyl)cyclobutanol **1f** (0.9 g, 50%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.37 (m, 2 H), 7.35 – 7.27 (m, 2 H), 2.63 (s, 1 H), 2.56 – 2.42 (m, 2 H), 2.39 – 2.26 (m, 2 H), 2.11 – 1.89 (m, 1 H), 1.76 – 1.59 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 144.71, 132.99, 128.51, 126.56, 76.62, 36.95, 12.95 ppm.

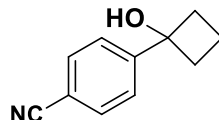
1-(4-(Trifluoromethyl)phenyl)cyclobutanol **1g**



The general procedure **A** conducted with (4-(trifluoromethyl)phenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(4-(trifluoromethyl)phenyl)cyclobutanol **1g** (1.3 g, 62%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 8.09 – 7.18 (m, 4 H), 2.73 – 2.46 (m, 2 H), 2.46 – 2.28 (m, 2 H), 2.19 – 1.88 (m, 1 H), 1.86 – 1.62 (m, 1 H); ^{19}F NMR (376 MHz, CDCl_3) δ -62.51 (s); ^{13}C NMR (101 MHz,

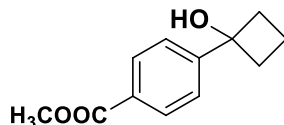
CDCl₃) δ 150.20, 129.40 (q, J = 33.3 Hz), 125.38 (q, J = 4.0 Hz), 125.31, 124.21 (q, J = 272.7 Hz), 76.70, 37.13, 12.97 ppm.

4-(1-Hydroxycyclobutyl)benzonitrile **1h**



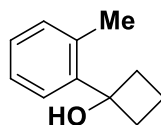
The general procedure **A** conducted with (4-cyanophenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 4-(1-hydroxycyclobutyl)benzonitrile **1h** (1.0 g, 60%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.57-7.60 (m, 4 H), 2.64 – 2.39 (m, 2 H), 2.36 (dt, J = 9.1, 8.2 Hz, 2 H), 2.20 – 1.88 (m, 1 H), 1.88 – 1.60 (m, 1 H); ¹³C NMR (101 MHz, CDCl₃) δ 151.94, 132.24, 125.75, 118.93, 110.53, 76.50, 37.23, 13.02 ppm.

Methyl 4-(1-hydroxycyclobutyl)benzoate **1i**



The general procedure **A** conducted with (4-(methoxycarbonyl)phenyl)magnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave methyl 4-(1-hydroxycyclobutyl)benzoate **1i** (1.7 g, 82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, J = 8.0 Hz, 2 H), 7.54 (d, J = 8.3 Hz, 2 H), 3.89 (s, 3 H), 2.70 (s, 1 H), 2.49 – 2.56 (m, 2 H), 2.47 – 2.29 (m, 2 H), 2.26 – 1.93 (m, 1 H), 1.84 – 1.62 (m, 1 H); ¹³C NMR (101 MHz, CDCl₃) δ 167.05, 151.51, 129.76, 128.81, 124.93, 76.77, 52.13, 37.09, 13.05 ppm.

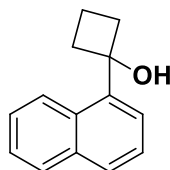
1-(*o*-Tolyl)cyclobutanol **1j**



The general procedure **A** conducted with *o*-tolylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave

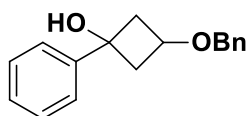
1-(*o*-tolyl)cyclobutanol **1j** (0.9 g, 55%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.36 – 7.27 (m, 1 H), 7.23 – 7.11 (m, 3 H), 2.96 – 2.59 (m, 2 H), 2.51 – 2.31 (m, 5 H), 2.30 – 2.05 (m, 1 H), 1.93 (s, 1 H), 1.84 – 1.47 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 142.97, 137.11, 131.76, 127.74, 125.52, 125.15, 79.08, 36.02, 20.20, 14.73.

1-(Naphthalen-1-yl)cyclobutanol **1k**



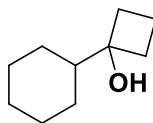
The general procedure **A** conducted with naphthalen-1-ylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-(naphthalen-1-yl)cyclobutanol **1k** (0.8 g, 41%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 8.38 – 8.25 (m, 1 H), 8.00 – 7.84 (m, 1 H), 7.81 (d, J = 8.2 Hz, 1 H), 7.62 – 7.37 (m, 4 H), 2.97 – 2.82 (m, 2 H), 2.73 – 2.54 (m, 2 H), 2.32 (s, 1 H), 2.16 (m, 1 H), 1.83 – 1.53 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 140.40, 134.78, 130.82, 128.96, 128.75, 126.28, 125.76, 125.65, 124.84, 122.91, 78.53, 36.78, 14.54 ppm.

3-(Benzyloxy)-1-phenylcyclobutanol **1l**



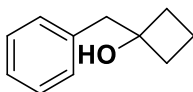
The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), 3-(benzyloxy)cyclobutanone (1.76g, 10.0 mmol) in THF (20.0 mL) gave 3-(benzyloxy)-1-phenylcyclobutanol **1l** (1.0 g, 40%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.43 (m, 2 H), 7.42 – 7.33 (m, 5 H), 7.34 – 7.26 (m, 2 H), 4.47 (s, 2 H), 3.86 (quint, J = 6.7 Hz, 1H), 2.92-2.98 (m, 2 H), 2.45-2.51 (m, 2 H); ^{13}C NMR (101 MHz, CDCl_3) δ 145.47, 138.05, 128.59, 128.50, 127.94, 127.80, 127.46, 127.30, 125.24, 124.84, 70.56, 70.24, 65.39, 45.37 ppm.

1-Cyclohexylcyclobutanol **1m**



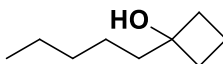
The general procedure **A** conducted with cyclohexylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-cyclohexylcyclobutanol **1m** (1.3 g, 85%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 2.21 – 2.06 (m, 2 H), 1.97 – 1.62 (m, 8 H), 1.57 – 1.43 (m, 1 H), 1.31-1.38 (m, 1 H), 1.29 – 0.95 (m, 5 H); ^{13}C NMR (101 MHz, CDCl_3) δ 78.03, 45.55, 33.82, 26.50, 26.47, 25.62, 12.40 ppm.

1-Benzylcyclobutanol **1n**



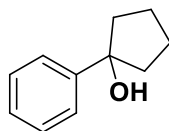
The general procedure **A** conducted with benzylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-benzylcyclobutanol **1n** (1.1 g, 70%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.06 (m, 5 H), 2.92 (s, 2 H), 2.36 – 2.08 (m, 2 H), 1.98-2.06 (m, 2 H), 1.93 – 1.71 (m, 1 H), 1.72 – 1.49 (m, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 137.64, 130.15, 128.36, 126.57, 75.07, 45.55, 35.41, 12.24 ppm.

1-Pentylcyclobutanol **1o**



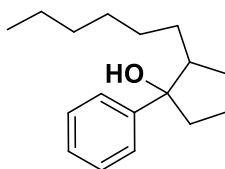
The general procedure **A** conducted with pentylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclobutanone (0.700 g, 10.0 mmol) in THF (20.0 mL) gave 1-pentylcyclobutanol **1o** (0.9 g, 63%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 2.12 – 1.88 (m, 5 H), 1.81 – 1.64 (m, 1 H), 1.63 – 1.46 (m, 2 H), 1.40 – 1.21 (m, 6 H), 0.87 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 75.40, 39.54, 35.88, 32.26, 23.08, 22.70, 14.05, 12.14 ppm.

1-Phenylcyclopentanol **3j**²



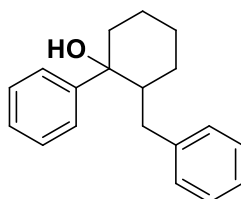
The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cyclopentanone (0.840 g, 10.0 mmol) in THF (20.0 mL) gave 1-phenylcyclopentanol **3j** (0.6 g, 40%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 30:1). ^1H NMR (400 MHz, CDCl_3) δ 7.63 – 7.42 (m, 2 H), 7.39 – 7.30 (m, 2 H), 7.22-7.26 (m, 1 H), 2.14 – 1.89 (m, 6 H), 1.80-1.85 (m, 2 H), 1.64 (s, 1 H); ^{13}C NMR (101 MHz, CDCl_3) δ 147.08, 128.24, 126.83, 125.11, 83.51, 41.86, 23.88 ppm.

2-Hexyl-1-phenylcyclopentanol **3l**



The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), 2-hexylcyclopentanone (1.68 g, 10.0 mmol) in THF (20.0 mL) gave 2-hexyl-1-phenylcyclopentanol **3l** (1.8 g, 75%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 40:1). ^1H NMR (400 MHz, CDCl_3) δ 7.51 – 7.39 (m, 2 H), 7.33 (t, J = 7.6 Hz, 2 H), 7.27 – 7.15 (m, 1 H), 2.17 – 1.99 (m, 2 H), 2.00 – 1.84 (m, 2 H), 1.85 – 1.69 (m, 1 H), 1.67 – 1.50 (m, 1 H), 1.41 – 1.06 (m, 10 H), 0.92 – 0.73 (m, 4 H); ^{13}C NMR (101 MHz, CDCl_3) δ 146.23, 128.09, 126.39, 125.03, 84.20, 50.94, 43.68, 31.81, 29.77, 29.59, 28.63, 28.06, 22.65, 21.76, 14.11 ppm. MS (EI): m/z (%) 133 (100). HRMS (EI) for $\text{C}_{17}\text{H}_{26}\text{O}$ Calcd: 246.1984; Found: 246.1992. IR (KBr): ν_{max} = 2950, 2856, 1718, 1445 cm^{-1} .

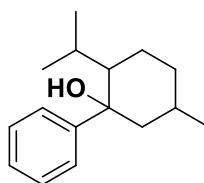
2-Benzyl-1-phenylcyclohexanol **3n**



The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0

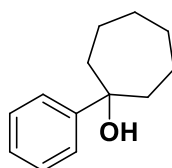
mmol, 1.00 N), 2-benzylcyclohexanone (1.88 g, 10.0 mmol) in THF (20.0 mL) gave 2-benzyl-1-phenylcyclohexanol **3n** (2.0 g, 78%) as a white solid (M.P.: 93.2-94.9 °C) (eluent: petroleum ether: ethyl acetate = 40:1). ¹H NMR (400 MHz, CDCl₃) δ 7.55 (d, *J* = 7.7 Hz, 2 H), 7.40 (t, *J* = 7.8 Hz, 2 H), 7.33 – 7.23 (m, 1 H), 7.23 – 7.16 (m, 2 H), 7.16 – 7.09 (m, 1 H), 6.95 (d, *J* = 7.1 Hz, 2 H), 2.42-2.46 (m, 1 H), 2.11-2.17 (m, 1 H), 2.08 – 1.93 (m, 1 H), 1.90 – 1.69 (m, 4 H), 1.68 – 1.56 (m, 2 H), 1.48 – 1.34 (m, 1 H), 1.22-1.30 (m, 1 H); ¹³C NMR (101 MHz, CDCl₃) δ 148.24, 141.43, 129.08, 128.33, 128.11, 126.50, 125.64, 124.74, 76.41, 47.79, 41.76, 36.51, 26.74, 25.94, 22.12 ppm. MS (EI): *m/z* (%) 120 (100). HRMS (EI) for C₁₉H₂₂O Calcd: 266.1671; Found: 266.1669. IR (KBr): ν_{max} = 2931, 2854, 1493, 1444 cm⁻¹.

2-Isopropyl-5-methyl-1-phenylcyclohexanol **3o**



The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), 2-isopropyl-5-methylcyclohexanone (1.54 g, 10.0 mmol) in THF (20.0 mL) gave 2-isopropyl-5-methyl-1-phenylcyclohexanol **3o** (1.3 g, 56%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.51 (m, 2 H), 7.33-7.37 (m, 2 H), 7.29 – 7.17 (m, 1 H), 1.81-1.90 (m, 2 H), 1.75 – 1.58 (m, 5 H), 1.56 – 1.37 (m, 2 H), 1.17 – 0.95 (m, 1 H), 0.97 – 0.87 (m, 3 H), 0.88 – 0.79 (m, 3 H), 0.79 – 0.69 (m, 3 H); ¹³C NMR (101 MHz, CDCl₃) δ 148.68, 128.13, 126.16, 124.66, 78.45, 51.67, 49.98, 35.25, 28.56, 26.75, 23.78, 22.28, 21.37, 18.44 ppm.

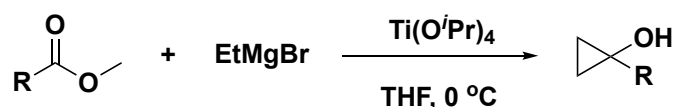
1-Phenylcycloheptanol **3p**³



The general procedure **A** conducted with phenylmagnesium bromide (20.0 mL, 20.0 mmol, 1.00 N), cycloheptanone (1.12 g, 10.0 mmol) in THF (20.0 mL) gave

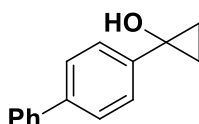
1-phenylcycloheptanol **3p** (1.3 g, 67%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (300 MHz, CDCl_3) δ 7.50 (d, $J = 7.2$ Hz, 2 H), 7.33 (t, $J = 7.4$ Hz, 2 H), 7.31 – 6.93 (m, 1 H), 2.41 – 1.31 (m, 12 H); ^{13}C NMR (101 MHz, CDCl_3) δ 150.78, 128.19, 126.54, 124.50, 76.86, 43.25, 29.16, 22.60 ppm.

General procedure B for the preparation of cyclopropanols



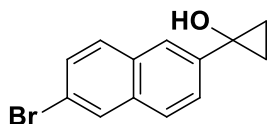
Ethyl magnesium bromide (10.0 mL, 2.0 equiv., 1.0 M in Et₂O) in THF was added dropwise over 30 min at 0 °C to a solution of ester (5.0 mmol, 1.0 equiv.) and titanium isopropoxide (1.6 g, 1.1 equiv.) in THF (20.0 mL). The mixture was warmed to room temperature and was stirred overnight. The mixture was then quenched with water. The precipitate was removed by filtration (30.0 mL × 3), washed with water and dried over anhydrous Na₂SO₄, filtered and concentrated. The residue was then purified by column chromatography to afford the cyclopropanols **3b**, **3d**, **3g**, **3h** and **3i** (eluent: petroleum ether: ethyl acetate = 10:1).

1-([1,1'-Biphenyl]-4-yl)cyclopropanol **3b**



The general procedure **B** conducted with ethylmagnesium bromide (10 mL, 10.0 mmol, 1.00 N), titanium isopropoxide (1.6 g, 5.5 mmol) and methyl [1,1'-biphenyl]-4-carboxylate (1.1 g, 5.0 mmol) in THF (20.0 mL) gave 1-([1,1'-biphenyl]-4-yl)cyclopropanol **3b** (0.6 g, 54%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.55-7.60 (m, 4 H), 7.51 – 7.39 (m, 2 H), 7.40 – 7.30 (m, 3 H), 2.52 (s, 1 H), 1.29-1.32 (m, 2 H), 1.19 – 1.01 (m, 2 H); ¹³C NMR (101 MHz, CDCl₃) δ 143.46, 140.84, 139.37, 128.81, 127.24, 127.12, 127.04, 124.87, 56.54, 18.06 ppm.

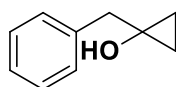
1-(6-Bromonaphthalen-2-yl)cyclopropanol **3d**



The general procedure **B** conducted with ethylmagnesium bromide (10 mL, 10.0 mmol, 1.00 N), titanium isopropoxide (1.6 g, 5.5 mmol) and methyl 6-bromo-2-naphthoate (1.3 g, 5.0 mmol) in THF (20.0 mL) gave

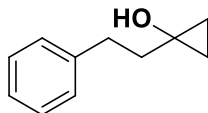
1-(6-bromonaphthalen-2-yl)cyclopropanol **3d** (0.7 g, 48%) as a white solid (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.94 (s, 1 H), 7.78 (s, 1 H), 7.73 – 7.57 (m, 2 H), 7.56 – 7.45 (m, 1 H), 7.24-7.27 (m, 1 H), 2.60 (s, 1 H), 1.33 (q, J = 5.3 Hz, 2 H), 1.13 (q, J = 5.2 Hz, 2 H); ^{13}C NMR (101 MHz, CDCl_3) δ 142.31, 133.19, 131.68, 129.61, 129.59, 129.43, 127.29, 123.87, 122.88, 119.41, 56.74, 18.06 ppm.

1-Benzylcyclopropanol **3g**⁴



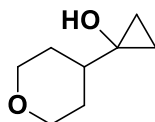
The general procedure **B** conducted with ethylmagnesium bromide (10 ml, 10.0 mmol, 1.00 N), titanium isopropoxide (1.6 g, 5.5 mmol) and methyl methyl 2-phenylacetate (0.8 g, 5.0 mmol) in THF (20.0 mL) gave 1-benzylcyclopropanol **3g** (0.5 g, 72%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.75 – 7.10 (m, 5 H), 2.87 (s, 2 H), 1.96 (s, 1 H), 0.80 (q, J = 5.2 Hz, 2 H), 0.63 (q, J = 5.0 Hz, 2 H); ^{13}C NMR (101 MHz, CDCl_3) δ 138.70, 129.51, 128.54, 126.65, 56.12, 44.13, 13.24 ppm.

1-Phenethylcyclopropanol **3h**⁵



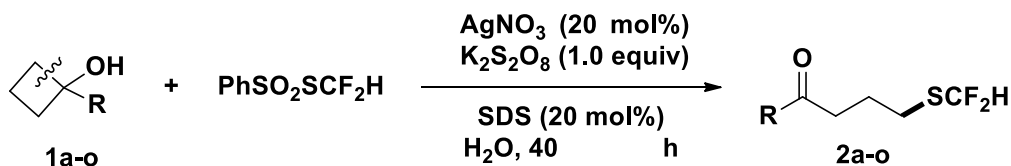
The general procedure **B** conducted with ethylmagnesium bromide (10 ml, 10.0 mmol, 1.00 N), titanium isopropoxide (1.6 g, 5.5 mmol) and methyl methyl 3-phenylpropanoate (0.8 g, 5.0 mmol) in THF (20.0 mL) gave 1-phenethylcyclopropanol **3h** (0.6 g, 75%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 7.84 – 6.99 (m, 5 H), 2.86-2.93 (m, 2 H), 1.87-1.93 (m, 2 H), 0.78-0.82 (m, 2 H), 0.47-0.50 (m, 2 H); ^{13}C NMR (101 MHz, CDCl_3) δ 142.41, 142.38, 128.51, 125.91, 55.66, 40.53, 32.58, 13.59 ppm.

1-(Tetrahydro-2 H-pyran-4-yl)cyclopropanol **3i**⁶



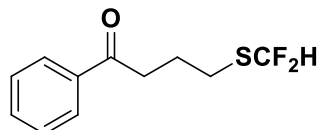
The general procedure **B** conducted with ethylmagnesium bromide (10 ml, 10.0 mmol, 1.00 N), titanium isopropoxide (1.6 g, 5.5 mmol) and methyl methyl tetrahydro-2 H-pyran-4-carboxylate (0.8 g, 5.0 mmol) in THF (20.0 mL) gave 1-(tetrahydro-2 H-pyran-4-yl)cyclopropanol **3i** (0.5 g, 70%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 30:1). ¹H NMR (400 MHz, CDCl₃) δ 4.75 (s, 1 H), 4.27 – 3.73 (m, 2 H), 3.25-3.32 (m, 2 H), 1.99 – 1.33 (m, 4 H), 1.37 – 0.96 (m, 1 H), 0.91 – 0.48 (m, 2 H), 0.52 – 0.14 (m, 2 H); ¹³C NMR (101 MHz, CDCl₃) δ 68.02, 58.19, 42.07, 28.68, 12.06 ppm.

General procedure for silver-catalyzed ring-opening difluoromethylation of cyclobutanol



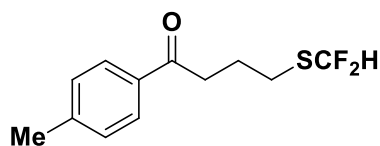
Cyclobutanol (0.300 mmol), AgNO₃ (10.2 mg, 6.00×10⁻² mmol), sodium dodecyl sulfate (SDS) (18.0 mg, 6.00×10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol), H₂O (0.5 mL) were placed into an oven-dried Schlenk tube that was equipped with a stirring bar under an atmosphere of argon. S-(Difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol) was added. The tube was quickly sealed with a rubber stopper. The reaction was stirred at 40 °C for 12 h and was then allowed to cool to room temperature. The mixture was extracted with diethyl ether (20 ml × 3), The organic layer was combined, dried over anhydrous Na₂SO₄ and concentrated under vacuum. The residue was purified by flash column chromatography (eluent: petroleum ether: ethyl acetate = 100:1). The yields of the products were calculated based on cyclobutanol.

4-((Difluoromethyl)thio)-1-phenylbutan-1-one 2a



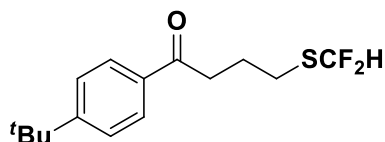
The general procedure conducted with 1-phenylcyclobutanol (44.5 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 4-((difluoromethyl)thio)-1-phenylbutan-1-one **2a** (57 mg, 82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 7.8 Hz, 2 H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2 H), δ 6.83 (t, *J* = 56.3 Hz, 1H), 3.14 (t, *J* = 7.0 Hz, 2 H), 2.93 (t, *J* = 7.1 Hz, 2 H), 2.14 (quint, *J* = 7.0 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.50 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 198.91, 136.74, 133.16, 128.62, 127.96, 120.64 (t, *J* = 272.7 Hz), 36.63, 26.82, 24.38 ppm. MS (EI): *m/z* (%) 120 (100). HRMS (EI) for C₁₁H₁₂F₂OS Calcd: 230.0577; Found: 230.0582. IR (KBr): ν_{max} = 1685, 1597 cm⁻¹.

4-((Difluoromethyl)thio)-1-(*p*-tolyl)butan-1-one **2b**



The general procedure conducted with 1-(*p*-tolyl)cyclobutanol (48.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 4-((difluoromethyl)thio)-1-(*p*-tolyl)butan-1-one **2b** (56.4 mg, 77%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 8.1 Hz, 2 H), 7.24 (d, *J* = 8.0 Hz, 2 H), 6.80 (t, *J* = 56.3 Hz, 1H), 3.09 (t, *J* = 7.0 Hz, 2 H), 2.91 (t, *J* = 7.1 Hz, 2 H), 2.39 (s, 3H), 2.10 (quint, *J* = 7.1 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.50 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 198.56, 143.97, 134.29, 129.29, 128.09, 120.67 (t, *J* = 272.6 Hz), 36.52, 26.88 (t, *J* = 3.1 Hz), 24.48, 21.59 ppm. MS (EI): *m/z* (%) 119 (100). HRMS (EI) for C₁₂H₁₄F₂OS Calcd: 244.0733; Found: 244.0731. IR (KBr): ν_{max} = 1682, 1573 cm⁻¹.

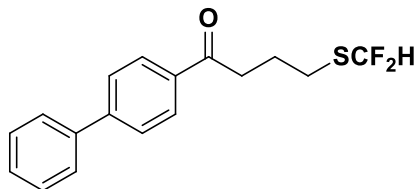
1-(4-(*tert*-Butyl)phenyl)-4-((difluoromethyl)thio)butan-1-one **2c**



The general procedure conducted with 1-(4-(*tert*-butyl)phenyl)cyclobutanol (61.3 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-(4-(*tert*-butyl)phenyl)-4-((difluoromethyl)thio)butan-1-one **2c** (54 mg, 63%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.4 Hz, 2 H), 7.50 (d, *J* = 8.4 Hz, 2 H), 6.84 (t, *J* = 56.3 Hz, 1H), 3.14 (t, *J* = 7.0 Hz, 2 H), 2.95 (t, *J* = 7.1 Hz, 2 H), 2.15 (quint, *J* = 7.0 Hz, 2 H), 1.36 (s, 9H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.48 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 198.58, 156.93, 134.19, 127.95, 125.56, 120.67 (t, *J* = 272.7 Hz), 36.53,

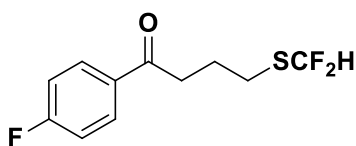
35.09, 31.04, 26.89, 24.48 ppm. MS (EI): m/z (%) 161 (100). HRMS (EI) for $C_{15}H_{20}F_2OS$ Calcd: 286.1203; Found: 286.1198. IR (KBr): $\nu_{\max}$ = 1682, 1605 cm^{-1} .

1-([1,1'-Biphenyl]-4-yl)-4-((difluoromethyl)thio)butan-1-one 2d



The general procedure conducted with 1-([1,1'-biphenyl]-4-yl)cyclobutanol (67.3 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-([1,1'-biphenyl]-4-yl)-4-((difluoromethyl)thio)butan-1-one **2d** (62.5 mg, 68%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ^1H NMR (400 MHz, CDCl_3) δ 8.03 (d, J = 8.2 Hz, 2 H), 7.68 (d, J = 8.2 Hz, 2 H), 7.62 (d, J = 7.4 Hz, 2 H), 7.46 (t, J = 7.4 Hz, 2 H), 7.39 (t, J = 7.2 Hz, 1 H), 6.82 (t, J = 56.3 Hz, 1 H), 3.15 (t, J = 6.9 Hz, 2 H), 2.94 (t, J = 7.1 Hz, 2 H), 2.15 (quint, J = 7.0 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.45 (d, J = 56.3 Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 198.53, 145.85, 139.78, 135.39, 128.94, 128.58, 128.25, 127.26, 127.24, 120.66 (t, J = 272.7 Hz), 36.68, 26.85, 24.44 ppm. MS (EI): m/z (%) 196 (100). HRMS (EI) for $C_{17}H_{16}F_2OS$ Calcd: 306.0890; Found: 306.0887. IR (KBr): $\nu_{\max}$ = 1677, 1603 cm^{-1} .

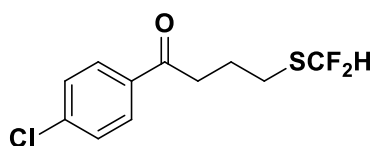
4-((Difluoromethyl)thio)-1-(4-fluorophenyl)butan-1-one 2e



The general procedure conducted with 1-(4-fluorophenyl)cyclobutanol (49.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 4-((difluoromethyl)thio)-1-(4-fluorophenyl)butan-1-one **2e** (60 mg, 80%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (dd, J = 8.6, 5.5 Hz, 2 H), 7.13 (t, J = 8.5 Hz, 2 H), 6.82 (t, J = 56.2 Hz,

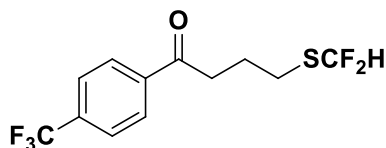
1H), 3.10 (t, $J = 7.0$ Hz, 2 H), 2.92 (t, $J = 7.1$ Hz, 2 H), 2.12 (quint, $J = 7.0$ Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.50 (d, $J = 56.2$ Hz), -105.10 (tt, $J = 8.4, 5.4$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 197.28, 165.77 (d, $J = 254.9$ Hz), 133.17 (d, $J = 3.0$ Hz), 130.59 (d, $J = 9.3$ Hz), 120.61 (t, $J = 272.7$ Hz), 115.70 (d, $J = 21.9$ Hz), 36.50, 26.74 (t, $J = 3.1$ Hz), 24.32 ppm. MS (EI): m/z (%) 138 (100). HRMS (EI) for $\text{C}_{11}\text{H}_{11}\text{F}_3\text{OS}$ Calcd: 248.0483; Found: 248.0487. IR (KBr): $\nu_{\text{max}} = 1685, 1598\text{ cm}^{-1}$.

1-(4-Chlorophenyl)-4-((difluoromethyl)thio)butan-1-one **2f**



The general procedure conducted with 1-(4-chlorophenyl)cyclobutanol (54.8 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-(4-chlorophenyl)-4-((difluoromethyl)thio)butan-1-one **2f** (44.5 mg, 56%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.6$ Hz, 2 H), 7.43 (d, $J = 8.6$ Hz, 2 H), 6.82 (t, $J = 56.2$ Hz, 1H), 3.10 (t, $J = 7.0$ Hz, 2 H), 2.92 (t, $J = 7.1$ Hz, 2 H), 2.12 (quint, $J = 7.0$ Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.48 (d, $J = 56.2$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 197.65, 139.61, 135.03, 129.38, 128.93, 120.58 (t, $J = 272.8$ Hz), 36.56, 26.70 (t, $J = 3.2$ Hz), 24.27 ppm. MS (EI): m/z (%) 154 (100). HRMS (EI) for $\text{C}_{11}\text{H}_{11}\text{ClF}_2\text{OS}$ Calcd: 264.0187; Found: 264.0181. IR (KBr): $\nu_{\text{max}} = 1685, 1590\text{ cm}^{-1}$.

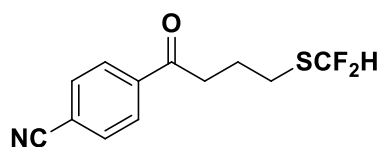
4-((Difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)butan-1-one **2g**



The general procedure conducted with 1-(4-(trifluoromethyl)phenyl)cyclobutanol (64.8 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave

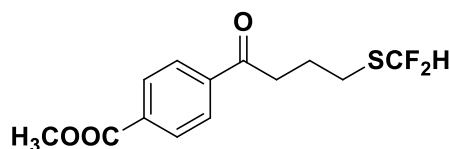
4-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)butan-1-one **2g** (53.7 mg, 60%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ^1H NMR (400 MHz, CDCl_3) δ 8.06 (d, J = 8.1 Hz, 2 H), 7.73 (d, J = 8.2 Hz, 2 H), 6.82 (t, J = 56.2 Hz, 1H), 3.16 (t, J = 6.9 Hz, 2 H), 2.94 (t, J = 7.1 Hz, 2 H), 2.15 (quint, J = 7.0 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -63.21 (s), -92.53 (d, J = 56.2 Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 197.97, 139.38, 134.53 (q, J = 32.3 Hz), 128.34, 125.75 (q, J = 3.7 Hz), 123.59 (q, J = 274.7 Hz), 120.58 (t, J = 273.7 Hz), 36.91, 26.64 (t, J = 3.2 Hz), 24.19 ppm. MS (EI): m/z (%) 188 (100). HRMS (EI) for $\text{C}_{12}\text{H}_{11}\text{F}_5\text{OS}$ Calcd: 298.0451; Found: 298.0457. IR (KBr): ν_{max} = 1693, 1582 cm^{-1} .

4-(4-((Difluoromethyl)thio)butanoyl)benzonitrile **2h**



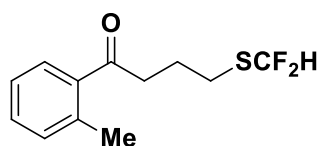
The general procedure conducted with 4-(1-hydroxycyclobutyl)benzonitrile (52 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 4-(4-((difluoromethyl)thio)butanoyl)benzonitrile **2h** (62.8 mg, 82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, J = 8.7 Hz, 2 H), 7.76 (d, J = 8.7 Hz, 2 H), 6.81 (t, J = 56.2 Hz, 1H), 3.14 (t, J = 6.9 Hz, 2 H), 2.93 (t, J = 7.0 Hz, 2 H), 2.13 (quint, J = 7.0 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.49 (d, J = 56.2 Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 197.62, 139.64, 132.59, 128.43, 119.89 (dd, J = 406.5, 139.4 Hz), 117.90, 116.51, 36.92, 26.56 (t, J = 3.1 Hz), 24.10 ppm. MS (EI): m/z (%) 145 (100). HRMS (EI) for $\text{C}_{12}\text{H}_{11}\text{F}_2\text{NOS}$ Calcd: 255.0529; Found: 255.0534. IR (KBr): ν_{max} = 2232, 1689 cm^{-1} .

Methyl 4-(4-((difluoromethyl)thio)butanoyl)benzoate **2i**



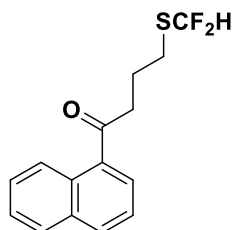
The general procedure conducted with methyl 4-(1-hydroxycyclobutyl)benzoate (62 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave methyl 4-(4-((difluoromethyl)thio)butanoyl)benzoate **2i** (77.8 mg, 90%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 40:1). ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.1 Hz, 2 H), 7.97 (d, *J* = 8.0 Hz, 2 H), 6.80 (t, *J* = 56.2 Hz, 1H), 3.91 (s, 3H), 3.13 (t, *J* = 6.9 Hz, 2 H), 2.91 (t, *J* = 7.0 Hz, 2 H), 2.30 – 1.93 (m, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.53 (d, *J* = 56.2 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 198.41, 166.13, 139.83, 133.92, 129.83, 127.85, 120.56 (t, *J* = 272.8 Hz), 52.43, 36.96 (s), 26.63 (t, *J* = 3.1 Hz), 24.19 ppm. MS (EI): *m/z* (%) 178 (100). HRMS (EI) for C₁₃H₁₄F₂O₃S Calcd: 288.0632; Found: 288.0634. IR (KBr): ν_{max} = 1724, 1689 cm⁻¹.

4-((Difluoromethyl)thio)-1-(*o*-tolyl)butan-1-one **2j**



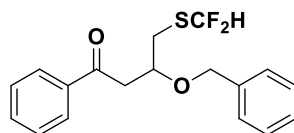
The general procedure conducted with 1-(*o*-tolyl)cyclobutanol (48.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 4-((difluoromethyl)thio)-1-(*o*-tolyl)butan-1-one **2j** (63 mg, 86%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 7.7 Hz, 1 H), 7.36 (t, *J* = 7.5 Hz, 1 H), 7.25 (t, *J* = 8.4 Hz, 2 H), 6.80 (t, *J* = 56.3 Hz, 1 H), 3.04 (t, *J* = 7.0 Hz, 2 H), 2.90 (t, *J* = 7.1 Hz, 2 H), 2.48 (s, 3 H), 2.09 (quint, *J* = 7.1 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.57 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 202.91, 138.10, 137.66, 132.01, 131.37, 128.39, 125.71, 120.60 (t, *J* = 272.7 Hz), 39.51, 26.76, 24.52, 21.28 ppm. MS (EI): *m/z* (%) 119 (100). HRMS (EI) for C₁₂H₁₄F₂OS Calcd: 244.0733; Found: 244.0743. IR (KBr): ν_{max} = 2923, 1684, 1601, 1570 cm⁻¹.

4-((Difluoromethyl)thio)-1-(naphthalen-1-yl)butan-1-one **2k**



The general procedure conducted with 1-(naphthalen-1-yl)cyclobutan-1-ol (59.5 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 4-((difluoromethyl)thio)-1-(naphthalen-1-yl)butan-1-one **2k** (79.0 mg, 95%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 8.60 (d, *J* = 8.5 Hz, 1 H), 7.99 (d, *J* = 8.2 Hz, 1 H), 7.88 (d, *J* = 7.4 Hz, 2 H), 7.70 – 7.39 (m, 3 H), 6.83 (t, *J* = 56.3 Hz, 1 H), 3.21 (t, *J* = 7.0 Hz, 2 H), 2.97 (t, *J* = 7.1 Hz, 2 H), 2.19 (quint, *J* = 7.1 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.50 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 203.16, 135.71, 134.02, 132.82, 130.14, 128.50, 128.04, 127.61, 126.54, 125.73, 124.40, 120.69 (t, *J* = 272.7 Hz), 40.14, 26.86, 24.85 ppm. MS (EI): *m/z* (%) 127 (100). HRMS (EI) for C₁₅H₁₄F₂OS Calcd: 280.0733; Found: 280.0731. IR (KBr): ν_{max} = 1678, 1593 cm⁻¹.

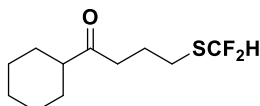
3-(Benzyloxy)-4-((difluoromethyl)thio)-1-phenylbutan-1-one **2l**



The general procedure conducted with 3-(benzyloxy)-1-phenylcyclobutan-1-ol (76.3 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 3-(benzyloxy)-4-((difluoromethyl)thio)-1-phenylbutan-1-one **2l** (50.5 mg, 50%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 40:1). ¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 7.5 Hz, 2 H), 7.57 (t, *J* = 7.2 Hz, 1 H), 7.46 (t, *J* = 7.4 Hz, 2 H), 7.27 (m, 5 H), 6.89 (t, *J* = 57.0 Hz, 1 H), 4.61 (m, 2 H), 4.44 – 4.30 (m, 1 H), 3.22 (m, 4 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.68 (d, *J* = 56.9 Hz); ¹³C NMR (101 MHz,

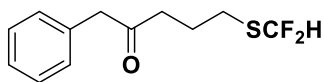
CDCl₃) δ 198.07, 137.98, 137.13, 133.67, 128.95, 128.71, 128.42, 128.20, 128.16, 120.80 (t, J = 272.9 Hz), 75.14, 72.67, 42.67, 31.86 ppm. MS (DART): m/z (%) 337 (100). HRMS (DART) for C₁₈H₁₉F₂O₂S⁺[M+H]⁺ Calcd: 337.1068; Found: 337.1068. IR (KBr): $\nu_{\max}$ = 1678, 1593 cm⁻¹.

1-Cyclohexyl-4-((difluoromethyl)thio)butan-1-one **2m**



The general procedure conducted with 1-cyclohexylcyclobutan-1-ol (46.3 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 $\times$ 10⁻² mmol), SDS (18.0 mg, 6.00 $\times$ 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-cyclohexyl-4-((difluoromethyl)thio)butan-1-one **2m** (65.2 mg, 92%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ¹H NMR (400 MHz, CDCl₃) δ 6.79 (t, J = 56.3 Hz, 1 H), 2.81 (t, J = 7.1 Hz, 2 H), 2.58 (t, J = 6.9 Hz, 2 H), 2.32 (m, 1 H), 1.92 (quint, J = 6.9 Hz, 2 H), 1.80 (m, 4 H), 1.74 – 1.58 (m, 1 H), 1.46 – 1.08 (m, 5 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.63 (d, J = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 212.87, 120.60 (t, J = 272.6 Hz), 50.86, 38.47, 28.44, 26.72 (t, J = 3.1 Hz), 25.78, 25.60, 23.82 ppm. MS (EI): m/z (%) 83 (100). HRMS (EI) for C₁₁H₁₈F₂OS Calcd: 236.1046; Found: 236.1051. IR (KBr): $\nu_{\max}$ = 1705 cm⁻¹.

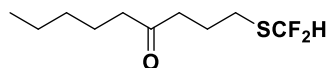
5-((Difluoromethyl)thio)-1-phenylpentan-2-one **2n**



The general procedure conducted with 3-(benzyloxy)-1-phenylcyclobutan-1-ol (48.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 $\times$ 10⁻² mmol), SDS (18.0 mg, 6.00 $\times$ 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 5-((difluoromethyl)thio)-1-phenylpentan-2-one **2n** (62.3 mg, 85%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 7.33 (t, J = 7.3 Hz, 2 H), 7.29 – 7.24 (m, 1 H), 7.19 (d, J = 6.9 Hz, 2 H), 6.74 (t, J = 56.3 Hz, 1 H), 3.68 (s, 2 H), 2.75 (t, J = 7.2 Hz, 2 H), 2.59 (t, J = 6.9 Hz, 2 H), 1.90 (quint, J =

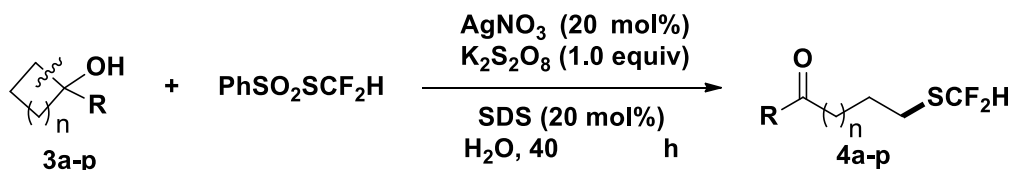
7.0 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.56 (d, $J = 56.3$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 207.13, 133.98, 129.35, 128.77, 127.10, 120.59 (t, $J = 272.7$ Hz), 50.21, 39.87, 26.52 (t, $J = 3.2$ Hz), 23.93 ppm. MS (EI): m/z (%) 89 (100). HRMS (EI) for $\text{C}_{12}\text{H}_{14}\text{F}_2\text{OS}$ Calcd: 244.0733; Found: 244.0723. IR (KBr): $\nu_{\text{max}} = 1713\text{ cm}^{-1}$.

1-((Difluoromethyl)thio)nonan-4-one 2o



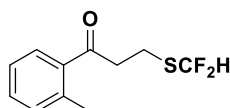
The general procedure conducted with 1-pentylcyclobutan-1-ol (42.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-((difluoromethyl)thio)nonan-4-one **2o** (61.2 mg, 91%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ^1H NMR (400 MHz, CDCl_3) δ 6.77 (t, $J = 56.3$ Hz, 1 H), 2.80 (t, $J = 7.2$ Hz, 2 H), 2.53 (t, $J = 7.0$ Hz, 2 H), 2.37 (t, $J = 7.5$ Hz, 2 H), 1.91 (quint, $J = 7.1$ Hz, 2 H), 1.60 – 1.46 (m, 2 H), 1.45 – 1.11 (m, 4 H), 0.86 (t, $J = 7.0$ Hz, 3 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.65 (d, $J = 56.3$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 210.03, 120.57 (t, $J = 272.6$ Hz), 42.87, 40.59, 31.34, 26.64 (t, $J = 3.1$ Hz), 23.91, 23.48, 22.39, 13.85 ppm. MS (EI): m/z (%) 43 (100). HRMS (EI) for $\text{C}_{10}\text{H}_{18}\text{F}_2\text{OS}$ Calcd: 224.1046; Found: 224.1039. IR (KBr): $\nu_{\text{max}} = 1695\text{ cm}^{-1}$.

General procedure for silver-catalyzed ring-opening difluoromethylation of cycloalkanol.



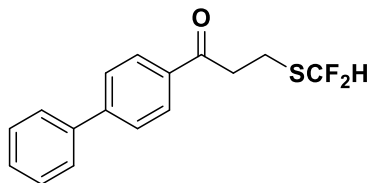
Cycloalkanol (0.300 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), sodium dodecyl sulfate (SDS) (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol), H₂O (0.5 mL) were placed into an oven-dried Schlenk tube that was equipped with a stirring bar under an atmosphere of argon. S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol) was added. The tube was quickly sealed with a rubber stopper. The reaction was stirred at 40 °C for 12 h and was then allowed to cool to room temperature. The mixture was extracted with diethyl ether (20 ml × 3), The organic layer was combined, dried over anhydrous Na₂SO₄ and concentrated under vacuum. The residue was purified by flash column chromatography (eluent: petroleum ether: ethyl acetate = 100:1). The yields of the products were calculated based on cyclic-alcohols.

3-((Difluoromethyl)thio)-1-(*o*-tolyl)propan-1-one 4a



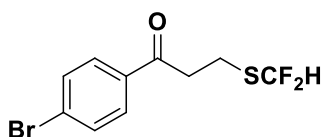
The general procedure conducted with 1-(*o*-tolyl)cyclopropanol (44.5 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 3-((difluoromethyl)thio)-1-(*o*-tolyl)propan-1-one **4a** (48.4 mg, 70%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 9.6 Hz, 2 H), 7.38 (m, 2 H), 6.87 (t, *J* = 56.2 Hz, 1H), 3.40 (t, *J* = 6.9 Hz, 2 H), 3.19 (t, *J* = 6.8 Hz, 2 H), 2.42 (s, 3 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.77 (d, *J* = 56.1 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 197.66, 138.53, 136.33, 134.22, 128.56, 128.49, 125.20, 120.80 (t, *J* = 272.8 Hz), 39.79, 21.32, 21.25 (t, *J* = 3.5 Hz) ppm. MS (EI): *m/z* (%) 119 (100). HRMS (EI) for C₁₁H₁₂F₂OS Calcd: 230.0577; Found: 230.0584. IR (KBr): ν_{max} = 1682, 1604, 1586 cm⁻¹.

1-([1,1'-Biphenyl]-4-yl)-3-((difluoromethyl)thio)propan-1-one **4b**



The general procedure conducted with 1-([1,1'-biphenyl]-4-yl)cyclopropanol (63.0 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-([1,1'-biphenyl]-4-yl)-3-((difluoromethyl)thio)propan-1-one **4b** (70.1 mg, 80%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.5 Hz, 2 H), 7.70 (d, *J* = 8.6 Hz, 2 H), 7.64 (d, *J* = 8.7 Hz, 2 H), 7.48 (t, *J* = 7.9 Hz, 2 H), 7.42 (t, *J* = 7.3 Hz, 1 H), 6.89 (t, *J* = 56.1 Hz, 1 H), 3.45 (t, *J* = 6.8 Hz, 2 H), 3.22 (t, *J* = 6.8 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.69 (d, *J* = 56.1 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 197.06, 146.13, 139.69, 134.99, 128.97, 128.60, 128.33, 127.32, 127.25, 120.83 (t, *J* = 272.8 Hz), 39.79, 21.27 (t, *J* = 3.6 Hz) ppm. MS (EI): *m/z* (%) 181 (100). HRMS (EI) for C₁₆H₁₄F₂OS Calcd: 292.0733; Found: 292.0724. IR (KBr): ν_{max} = 1677, 1603 cm⁻¹.

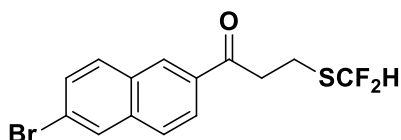
1-(4-Bromophenyl)-3-((difluoromethyl)thio)propan-1-one **4c**



The general procedure conducted with 1-(4-bromophenyl)cyclopropanol (0.3 mmol, 63.9 mg), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-(4-bromophenyl)-3-((difluoromethyl)thio)propan-1-one **4c** (51.3 mg, 58%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, *J* = 10.6 Hz, 2 H), 7.60 (d, *J* = 8.6 Hz, 2 H), 6.84 (t, *J* = 56.0 Hz, 1H), 3.36 (t, *J* = 6.8 Hz, 2 H), 3.16 (t, *J* = 6.8 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ

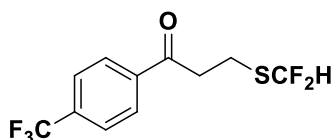
-92.77 (d, $J = 56.0$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 196.47, 135.00, 132.03, 129.48, 128.71, 120.70 (t, $J = 273.0$ Hz), 39.77, 21.02 (t, $J = 3.6$ Hz) ppm. MS (EI): m/z (%) 183 (100). HRMS (EI) for $\text{C}_{10}\text{H}_9\text{BrF}_2\text{OS}$ Calcd: 293.9526; Found: 293.9527. IR (KBr): $\nu_{\text{max}} = 1685, 1587\text{ cm}^{-1}$.

1-(6-Bromonaphthalen-2-yl)-3-((difluoromethyl)thio)propan-1-one 4d



The general procedure conducted with 1-(6-bromonaphthalen-2-yl)cyclopropanol (78.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave (6-bromonaphthalen-2-yl)-3-((difluoromethyl)thio)propan-1-one **4d** (41.4 mg, 40%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ^1H NMR (400 MHz, CDCl_3) δ 8.38 (s, 1H), 8.09 – 7.92 (m, 2 H), 7.78 (t, $J = 8.5$ Hz, 2 H), 7.60 (dd, $J = 8.7, 1.8$ Hz, 1 H), 6.88 (t, $J = 56.1$ Hz, 1 H), 3.50 (t, $J = 6.9$ Hz, 2 H), 3.23 (t, $J = 6.8$ Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.66 (d, $J = 56.0$ Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 197.04, 136.55, 133.91, 131.04, 130.85, 130.42, 129.94, 129.56, 127.66, 124.71, 123.08, 120.80 (t, $J = 273.7$ Hz), 39.88, 21.24 (t, $J = 3.6$ Hz) ppm. MS (EI): m/z (%) 126 (100). HRMS (EI) for $\text{C}_{14}\text{H}_{11}\text{BrF}_2\text{OS}$ Calcd: 343.9682; Found: 343.9678. IR (KBr): $\nu_{\text{max}} = 1682, 1618\text{ cm}^{-1}$.

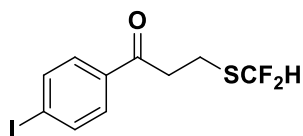
3-((Difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)propan-1-one 4e



The general procedure conducted with 1-(4-(trifluoromethyl)phenyl)cyclopropanol (60.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 3-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)propan-1-one **4e** (69.9 mg,

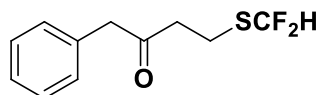
82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ^1H NMR (400 MHz, CDCl_3) δ 8.06 (d, J = 8.2 Hz, 2 H), 7.74 (d, J = 8.2 Hz, 2 H), 6.87 (t, J = 56.0 Hz, 1H), 3.44 (t, J = 6.8 Hz, 2 H), 3.20 (t, J = 6.7 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -63.26 (s, 3 F), -92.81 (d, J = 56.0 Hz, 2 F); ^{13}C NMR (101 MHz, CDCl_3) δ 196.56, 138.89, 134.72 (q, J = 32.7 Hz), 128.32, 123.47 (q, J = 273.7 Hz), 125.78 (q, J = 3.7 Hz), 120.68 (t, J = 273.7 Hz), 40.14, 20.88 (t, J = 3.7 Hz) ppm. MS (EI): m/z (%) 173 (100). HRMS (EI) for $\text{C}_{11}\text{H}_9\text{F}_5\text{OS}$ Calcd: 284.0294; Found: 284.0289. IR (KBr): ν_{max} = 1693 cm^{-1} .

3-((Difluoromethyl)thio)-1-(4-iodophenyl)propan-1-one 4f



The general procedure conducted with 1-(4-iodophenyl)cyclopropanol (78.0 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 3-((difluoromethyl)thio)-1-(4-iodophenyl)propan-1-one **4f** (67.7 mg, 66%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ^1H NMR (400 MHz, CDCl_3) δ 7.82 (d, J = 8.5 Hz, 2 H), 7.64 (d, J = 8.6 Hz, 2 H), 6.84 (t, J = 56.0 Hz, 1H), 3.35 (t, J = 6.8 Hz, 2 H), 3.16 (t, J = 6.8 Hz, 2 H); ^{19}F NMR (376 MHz, CDCl_3) δ -92.76 (d, J = 56.0 Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 196.79, 138.03, 135.52, 129.33, 120.70 (t, J = 273.0 Hz), 101.53, 39.70, 21.01 ppm. MS (EI): m/z (%) 231 (100). HRMS (EI) for $\text{C}_{10}\text{H}_9\text{F}_2\text{IOS}$ Calcd: 341.9387; Found: 341.9389. IR (KBr): ν_{max} = 1682, 1578 cm^{-1} .

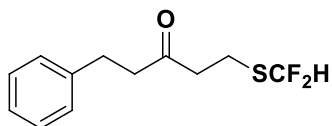
4-((Difluoromethyl)thio)-1-phenylbutan-2-one 4g



The general procedure conducted with 1-benzylcyclopropanol (44.5 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol)

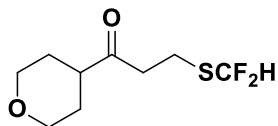
in H₂O (0.5 mL) gave 4-((difluoromethyl)thio)-1-phenylbutan-2-one **4g** (49.1 mg, 71%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.34 (t, *J* = 7.2 Hz, 2 H), 7.28 (dd, *J* = 9.4, 6.5 Hz, 1 H), 7.23 – 7.16 (m, 2 H), 6.78 (t, *J* = 56.2 Hz, 1H), 3.71 (s, 2 H), 2.97 (t, *J* = 6.7 Hz, 2 H), 2.87 (t, *J* = 6.5 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.84 (d, *J* = 56.2 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 205.73, 133.52, 129.37, 128.82, 127.23, 120.67 (t, *J* = 272.8 Hz), 50.21, 42.67, 20.79 (d, *J* = 3.5 Hz) ppm. MS (EI): *m/z* (%) 91 (100). HRMS (EI) for C₁₁H₁₂F₂OS Calcd: 230.0577; Found: 230.0585. IR (KBr): ν_{max} = 1716 cm⁻¹.

1-((Difluoromethyl)thio)-5-phenylpentan-3-one **4h**



The general procedure conducted with 1-phenethylcyclopropanol (48.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-((difluoromethyl)thio)-5-phenylpentan-3-one **4h** (60.0 mg, 82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.29 (m, 2 H), 7.23 (m, 3 H), 6.84 (t, *J* = 56.1 Hz, 1 H), 3.03 (t, *J* = 6.8 Hz, 2 H), 2.96 (t, *J* = 7.6 Hz, 2 H), 2.85 (t, *J* = 6.8 Hz, 2 H), 2.80 (t, *J* = 7.5 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.84 (d, *J* = 56.1 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 209.96, 143.35, 131.22, 130.96, 128.90, 123.42 (t, *J* = 272.8 Hz), 47.12, 46.34, 32.27, 23.44 (t, *J* = 3.5 Hz) ppm. MS (EI): *m/z* (%) 91 (100). HRMS (EI) for C₁₂H₁₄F₂OS Calcd: 244.0733; Found: 244.0730. IR (KBr): ν_{max} = 1715, 1603 cm⁻¹.

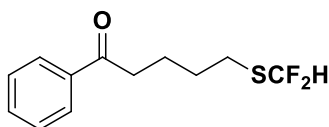
3-((Difluoromethyl)thio)-1-(tetrahydro-2 H-pyran-4-yl)propan-1-one **4i**



The general procedure conducted with 1-(tetrahydro-2 H-pyran-4-yl)cyclopropanol (42.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol),

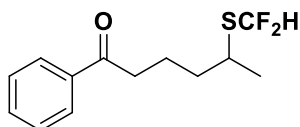
K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 3-((difluoromethyl)thio)-1-(tetrahydro-2*H*-pyran-4-yl)propan-1-one **4i** (43.0 mg, 64%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ¹H NMR (400 MHz, CDCl₃) δ 6.82 (t, *J* = 56.1 Hz, 1H), 4.25 – 3.88 (m, 2 H), 3.43 (td, *J* = 11.4, 2.5 Hz, 2 H), 3.02 (t, *J* = 6.6 Hz, 2 H), 2.89 (t, *J* = 6.6 Hz, 2 H), 2.76 – 2.39 (m, 1 H), 1.99 – 1.49 (m, 4 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.67 (d, *J* = 56.1 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 209.30, 120.73 (t, *J* = 272.8 Hz), 67.10, 47.55, 41.21, 27.87, 20.75 (t, *J* = 3.5 Hz) ppm. MS (EI): *m/z* (%) 91 (100). HRMS (EI) for C₉H₁₄F₂O₂S Calcd: 224.0683; Found: 224.0688. IR (KBr): ν_{max} = 1717 cm⁻¹.

5-((Difluoromethyl)thio)-1-phenylpentan-1-one **4j**



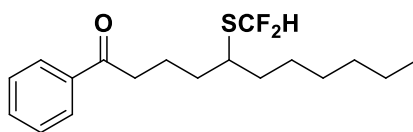
The general procedure conducted with 1-phenylcyclopentanol (48.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 5-((difluoromethyl)thio)-1-phenylpentan-1-one **4j** (49.1 mg, 67%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 8.02 – 7.88 (m, 2 H), 7.62 – 7.49 (m, 1 H), 7.44 (dd, *J* = 10.4, 4.7 Hz, 2 H), 6.79 (t, *J* = 56.3 Hz, 1 H), 2.99 (t, *J* = 7.0 Hz, 2 H), 2.83 (t, *J* = 7.2 Hz, 2 H), 1.94 – 1.80 (m, 2 H), 1.74 (ddt, *J* = 8.7, 6.9, 4.3 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.76 (d, *J* = 56.3 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 199.55, 136.82, 133.04, 128.58, 127.96, 120.65 (t, *J* = 272.5 Hz), 37.73, 29.76, 26.96 (t, *J* = 3.2 Hz), 23.11 ppm. MS (EI): *m/z* (%) 105 (100). HRMS (EI) for C₁₂H₁₄F₂OS Calcd: 244.0733; Found: 244.0736. IR (KBr): ν_{max} = 1685, 1597 cm⁻¹.

5-((Difluoromethyl)thio)-1-phenylhexan-1-one **4k**



The general procedure conducted with 2-methyl-1-phenylcyclopentanol (52.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 5-((difluoromethyl)thio)-1-phenylhexan-1-one **4k** (54.2 mg, 70%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.94 (dd, *J* = 8.3, 1.2 Hz, 2 H), 7.66 – 7.48 (m, 1 H), 7.44 (dd, *J* = 10.5, 4.7 Hz, 2 H), 6.84 (t, *J* = 56.5 Hz, 1 H), 3.29 (hept, *J* = 6.8 Hz, 1 H), 3.12 – 2.80 (m, 2 H), 2.09 – 1.78 (m, 2 H), 1.69 (dt, *J* = 10.3, 6.3 Hz, 2 H), 1.41 (d, *J* = 6.9 Hz, 3 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -85.74 – -97.61 (m); ¹³C NMR (101 MHz, CDCl₃) δ 199.63, 136.85, 133.03, 128.58, 127.96, 120.89 (t, *J* = 271.9 Hz), 38.49, 37.90, 36.93, 22.75, 21.31 ppm. MS (EI): *m/z* (%) 105 (100). HRMS (EI) for C₁₃H₁₆F₂OS Calcd: 258.0890; Found: 258.0885. IR (KBr): *v*_{max} = 1685, 1597 cm⁻¹.

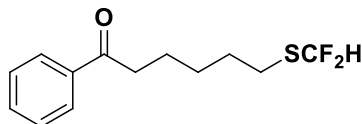
5-((Difluoromethyl)thio)-1-phenylundecan-1-one **4l**



The general procedure conducted with 2-hexyl-1-phenylcyclopentanol (73.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 5-((difluoromethyl)thio)-1-phenylundecan-1-one **4l** (59.1 mg, 60%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 7.3 Hz, 2 H), 7.55 (t, *J* = 7.4 Hz, 1 H), 7.45 (t, *J* = 7.5 Hz, 2 H), 6.83 (t, *J* = 56.6 Hz, 1 H), 3.23 – 3.06 (m, 1 H), 2.98 (t, *J* = 7.2 Hz, 2 H), 1.88 (m, 2 H), 1.80 – 1.52 (m, 4 H), 1.51 – 1.34 (m, 2 H), 1.27 (m, 6H), 0.86 (t, *J* = 6.5 Hz, 3 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -91.00 (dd, *J* = 56.6, 5.5 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 199.75, 136.88, 133.00, 128.57, 127.97, 120.95 (t, *J* = 271.9 Hz), 43.93, 38.03, 35.55, 35.21, 31.65, 28.99, 26.47, 22.56, 21.09, 14.03 ppm. MS (EI): *m/z* (%) 105 (100). HRMS

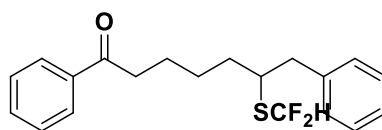
(EI) for $C_{18}H_{26}F_2OS$ Calcd: 328.1672; Found: 328.1680. IR (KBr): $\nu_{\max} = 1686, 1597\text{ cm}^{-1}$.

6-((Difluoromethyl)thio)-1-phenylhexan-1-one 4m



The general procedure conducted with 1-phenylcyclohexanol (52.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 6-((difluoromethyl)thio)-1-phenylhexan-1-one **4m** (54.2 mg, 70%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). 1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, $J = 7.6$ Hz, 2 H), 7.56 (t, $J = 7.4$ Hz, 1 H), 7.46 (t, $J = 7.6$ Hz, 2 H), 6.80 (t, $J = 56.4$ Hz, 1 H), 2.98 (t, $J = 7.3$ Hz, 2 H), 2.81 (t, $J = 7.4$ Hz, 2 H), 2.01 – 1.62 (m, 4 H), 1.60 – 1.30 (m, 2 H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -92.72 (d, $J = 56.4$ Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.97, 136.97, 132.95, 128.56, 127.98, 120.71 (t, $J = 272.4$ Hz), 38.20, 29.98, 28.28, 26.99 (t, $J = 3.0$ Hz), 23.56 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $C_{13}H_{16}F_2OS$ Calcd: 258.0890; Found: 258.0898. IR (KBr): $\nu_{\max} = 1685, 1597\text{ cm}^{-1}$.

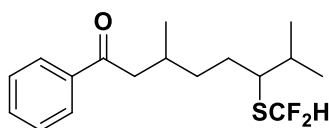
6-((Difluoromethyl)thio)-1,7-diphenylheptan-1-one 4n



The general procedure conducted with 2-benzyl-1-phenylcyclohexanol (79.9 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 6-((difluoromethyl)thio)-1,7-diphenylheptan-1-one **4n** (63.8 mg, 61%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). 1H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.89 (m, 2 H), 7.55 (t, $J = 7.4$ Hz, 1 H), 7.46 (d, $J = 7.8$ Hz, 2 H), 7.30 (t, $J = 7.2$ Hz, 2 H), 7.26 – 7.14 (m, 3 H), 6.63 (t, $J = 56.8$ Hz, 1 H), 3.44 – 3.26 (m, 1 H), 3.02 (dd, J

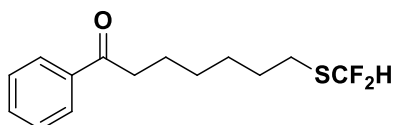
= 13.9, 6.7 Hz, 2 H), 2.93 (t, J = 7.2 Hz, 2 H), 1.87 – 1.43 (m, 6 H); ^{19}F NMR (376 MHz, CDCl_3) δ -91.46 (qd, J = 244.7, 56.8 Hz); ^{13}C NMR (101 MHz, CDCl_3) δ 200.01, 138.47, 136.94, 132.94, 129.36, 128.55, 128.42, 127.99, 126.69, 123.96 – 117.46 (m), 45.10, 43.03, 38.20, 34.14, 26.17, 23.67 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $\text{C}_{20}\text{H}_{21}\text{FOS}[\text{M}-\text{HF}]^+$ Calcd: 328.1297; Found: 328.1298. IR (KBr): ν_{max} = 1686, 1597 cm^{-1} .

6-((Difluoromethyl)thio)-3,7-dimethyl-1-phenyloctan-1-one **4o**



The general procedure conducted with 2-isopropyl-5-methyl-1-phenylcyclohexanol (69.7 mg, 0.300 mmol), S-(difluoromethyl)benzenesulfonylthionate (134 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 6-((difluoromethyl)thio)-3,7-dimethyl-1-phenyloctan-1-one **4o** (1:1 d.r., 64.1 mg, 68%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, J = 7.4 Hz, 2 H), 7.77 – 7.50 (m, 1 H), 7.44 (dd, J = 11.2, 4.0 Hz, 2 H), 6.79 (td, J = 56.8, 5.1 Hz, 1 H), 3.10 – 2.85 (m, 2 H), 2.79 (dd, J = 16.1, 7.8 Hz, 1H), 2.17 (td, J = 13.2, 6.5 Hz, 1 H), 1.96 (d, J = 5.6 Hz, 1 H), 1.81 – 1.39 (m, 4 H), 1.11 – 0.81 (m, 9 H); ^{19}F NMR (376 MHz, CDCl_3) δ -82.32 – -96.21 (m); ^{13}C NMR (101 MHz, CDCl_3) δ 200.22, 136.98, 132.90, 128.53, 127.98, 120.73 (tt, J = 272.2 Hz, 272.2 Hz), 50.59 (d, J = 4.9 Hz), 45.46, 45.23, 34.07, 31.20 (dd, J = 135.0, 42.2 Hz), 24.26 (dd, J = 943.7, 14.2 Hz), 19.30 (d, J = 4.0 Hz), 17.89, 17.62 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $\text{C}_{17}\text{H}_{24}\text{F}_2\text{OS}$ Calcd: 314.1516; Found: 314.1519. IR (KBr): ν_{max} = 1677 cm^{-1} .

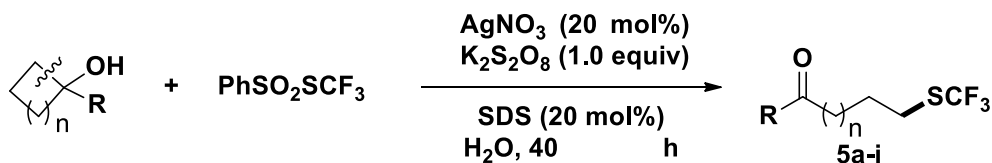
7-((Difluoromethyl)thio)-1-phenylheptan-1-one **4p**



The general procedure conducted with 1-phenylcycloheptanol (57.1 mg, 0.300 mmol),

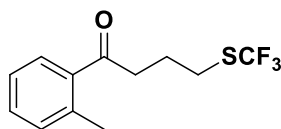
S-(difluoromethyl)benzenesulfonothionate (134 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 7-((difluoromethyl)thio)-1-phenylheptan-1-one **4p** (44.1 mg, 54%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 7.2 Hz, 2 H), 7.56 (dd, *J* = 10.6, 4.1 Hz, 1 H), 7.45 (t, *J* = 7.5 Hz, 2 H), 6.79 (t, *J* = 56.4 Hz, 1 H), 2.97 (t, *J* = 7.3 Hz, 2 H), 2.79 (t, *J* = 7.4 Hz, 2 H), 1.90 – 1.56 (m, 4 H), 1.55 – 1.24 (m, 4 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.75 (d, *J* = 56.4 Hz); ¹³C NMR (101 MHz, CDCl₃) δ 200.22, 136.98, 132.90, 128.53, 127.98, 120.73 (t, *J* = 272.2 Hz), 38.34, 29.90, 28.68, 28.47, 27.09, 24.01 ppm. MS (EI): *m/z* (%) 105 (100). HRMS (EI) for C₁₄H₁₈F₂OS Calcd: 272.1046; Found: 272.1052. IR (KBr): ν_{max} = 1685, 1597 cm⁻¹.

General procedure for silver-catalyzed ring-opening trifluoromethylation of cycloalkanol



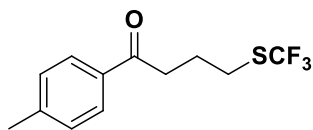
Cycloalkanol (0.300 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), sodium dodecyl sulfate (SDS) (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol), H₂O (0.5 mL) were placed into an oven-dried Schlenk tube that was equipped with a stirring bar under an atmosphere of argon. S-(Trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol) was added. The tube was quickly sealed with a rubber stopper. The reaction was stirred at 40 °C for 12 h and was then allowed to cool to room temperature. The mixture was extracted with diethyl ether (20 ml × 3), The organic layer was combined, dried over anhydrous Na₂SO₄ and concentrated under vacuum. The residue was purified by flash column chromatography (eluent: petroleum ether: ethyl acetate = 100:1). The yields of the products were calculated based on cyclobutanol.

1-(*o*-Tolyl)-4-((trifluoromethyl)thio)butan-1-one **5a**



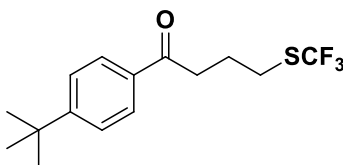
The general procedure conducted with 1-(*o*-tolyl)cyclobutanol (48.7 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-(*o*-tolyl)-4-((trifluoromethyl)thio)butan-1-one **5a** (70.8 mg, 90%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 7.7 Hz, 1 H), 7.37 (t, *J* = 7.5 Hz, 1 H), 7.32 – 7.18 (m, 2 H), 3.02 (m, 4 H), 2.48 (s, 3 H), 2.11 (quint, *J* = 7.0 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -41.01 (s); ¹³C NMR (101 MHz, CDCl₃) δ 202.48, 138.20, 137.42, 132.06, 131.49, 130.99 (q, *J* = 307.0 Hz), 128.41, 125.74, 39.16, 29.37 (d, *J* = 1.9 Hz), 23.86, 21.34 ppm. MS (EI): *m/z* (%) 119 (100). HRMS (EI) for C₁₂H₁₃F₃OS Calcd: 262.0639; Found: 262.0640. IR (KBr): ν_{max} = 1685, 1601 cm⁻¹.

1-(*p*-Tolyl)-4-((trifluoromethyl)thio)butan-1-one **5b**



The general procedure conducted with 1-(*p*-tolyl)cyclobutanol (48.7 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-(*p*-tolyl)-4-((trifluoromethyl)thio)butan-1-one **5b** (64.5 mg, 82%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 80:1). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 8.1 Hz, 2 H), 7.25 (d, *J* = 7.8 Hz, 2 H), 3.10 (t, *J* = 6.9 Hz, 2 H), 3.00 (t, *J* = 7.2 Hz, 2 H), 2.40 (s, 3 H), 2.13 (quint, *J* = 7.0 Hz, 2 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -40.99 (s); ¹³C NMR (101 MHz, CDCl₃) δ 197.75, 143.61, 133.70, 130.54 (q, *J* = 307.0 Hz), 128.84, 127.58, 35.75, 28.96 (d, *J* = 2.0 Hz), 23.32, 21.11 ppm. MS (EI): *m/z* (%) 119 (100). HRMS (EI) for C₁₂H₁₃F₃OS Calcd: 262.0639; Found: 262.0638. IR (KBr): ν_{max} = 1677, 1605 cm⁻¹.

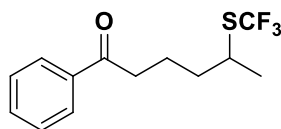
1-(4-(*tert*-Butyl)phenyl)-4-((trifluoromethyl)thio)butan-1-one **5c**



The general procedure conducted with 1-(4-(*tert*-butyl)phenyl)cyclobutanol (61.3 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), AgNO₃ (10.2 mg, 6.00 × 10⁻² mmol), SDS (18.0 mg, 6.00 × 10⁻² mmol), K₂S₂O₈ (81.0 mg, 0.300 mmol) in H₂O (0.5 mL) gave 1-(4-(*tert*-butyl)phenyl)-4-((trifluoromethyl)thio)butan-1-one **5c** (80.4 mg, 88%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 8.3 Hz, 2 H), 7.47 (d, *J* = 8.3 Hz, 2 H), 3.11 (t, *J* = 6.9 Hz, 2 H), 3.00 (t, *J* = 7.2 Hz, 2 H), 2.14 (quint, *J* = 7.0 Hz, 2 H), 1.33 (s, 9H); ¹⁹F NMR (376 MHz, CDCl₃) δ -40.97 (s); ¹³C NMR (101 MHz, CDCl₃) δ 198.23, 157.04, 134.06, 131.02 (q, *J* = 308.0 Hz), 127.92, 125.58, 36.23, 35.10, 31.03, 29.44 (d, *J* = 1.9 Hz), 23.79 ppm. MS (EI): *m/z* (%) 161 (100). HRMS (EI) for C₁₅H₁₉F₃OS Calcd: 304.1109;

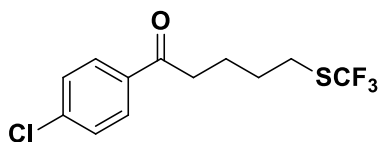
Found: 304.1102. IR (KBr): $\nu_{\max}$ = 1683, 1605 cm^{-1} .

1-Phenyl-5-((trifluoromethyl)thio)hexan-1-one 5d



The general procedure conducted with 2-methyl-1-phenylcyclopentanol (52.9 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-phenyl-5-((trifluoromethyl)thio)hexan-1-one **5d** (64.7 mg, 78%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ^1H NMR (400 MHz, CDCl_3) δ 7.93 (dd, J = 5.2, 3.3 Hz, 2 H), 7.69 – 7.47 (m, 1 H), 7.50 – 7.38 (m, 2 H), 3.63 – 3.16 (m, 1 H), 2.99 (dd, J = 10.8, 3.8 Hz, 2 H), 2.01 – 1.78 (m, 2 H), 1.79 – 1.62 (m, 2 H), 1.43 (dd, J = 6.4, 5.9 Hz, 3 H); ^{19}F NMR (376 MHz, CDCl_3) δ -39.14 (s); ^{13}C NMR (101 MHz, CDCl_3) δ 199.43, 136.80, 133.08, 131.14 (q, J = 308.0 Hz), 128.60, 127.95, 41.04, 37.80, 36.33, 22.18, 21.15 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $\text{C}_{13}\text{H}_{15}\text{F}_3\text{OS}$ Calcd: 276.0796; Found: 276.0806. IR (KBr): $\nu_{\max}$ = 1686, 1597 cm^{-1} .

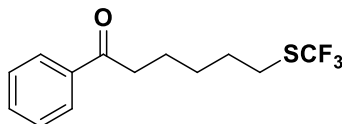
1-(4-Chlorophenyl)-5-((trifluoromethyl)thio)pentan-1-one 5e



The general procedure conducted with 1-(4-chlorophenyl)cyclopentanol (59.0 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), AgNO_3 (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $\text{K}_2\text{S}_2\text{O}_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-(4-chlorophenyl)-5-((trifluoromethyl)thio)pentan-1-one **5e** (55.2 mg, 62%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, J = 8.5 Hz, 2 H), 7.42 (d, J = 8.5 Hz, 2 H), 2.94 (m, 4 H), 2.15 – 1.62 (m, 4 H); ^{19}F NMR (376 MHz, CDCl_3) δ -41.16 (s); ^{13}C NMR (101 MHz, CDCl_3) δ 197.57, 139.08, 134.61, 130.59 (q, J = 307.0 Hz), 128.88, 128.45, 37.13, 29.18 (d, J

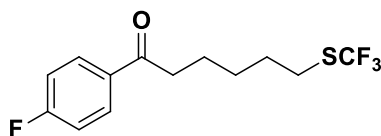
= 1.9 Hz), 28.57, 22.31 ppm. MS (EI): m/z (%) 139 (100). HRMS (EI) for $C_{12}H_{12}ClF_3OS$ Calcd: 296.0249; Found: 296.0252. IR (KBr): $\nu_{\max}$ = 1686, 1590 cm^{-1} .

1-Phenyl-6-((trifluoromethyl)thio)hexan-1-one **5f**



The general procedure conducted with 1-phenylcyclohexanol (52.9 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-phenyl-6-((trifluoromethyl)thio)hexan-1-one **5f** (73.8 mg, 89%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). 1H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.3 Hz, 2 H), 7.54 (t, J = 7.4 Hz, 1 H), 7.44 (t, J = 7.6 Hz, 2 H), 2.97 (t, J = 7.2 Hz, 2 H), 2.88 (t, J = 7.4 Hz, 2 H), 1.88 – 1.65 (m, 4 H), 1.55 – 1.33 (m, 2 H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -41.19 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.84, 136.90, 132.98, 131.13 (q, J = 308.0 Hz), 128.56, 127.96, 38.12, 29.65 (d, J = 1.9 Hz), 29.31, 28.10, 23.45 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $C_{13}H_{15}F_3OS$ Calcd: 276.0796; Found: 276.0794. IR (KBr): $\nu_{\max}$ = 1686, 1597 cm^{-1} .

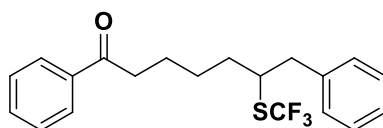
1-(4-Fluorophenyl)-6-((trifluoromethyl)thio)hexan-1-one **5g**



The general procedure conducted with 1-(4-fluorophenyl)cyclohexanol (58.3 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-(4-fluorophenyl)-6-((trifluoromethyl)thio)hexan-1-one **5g** (69.7 mg, 79%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). 1H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, J = 8.4, 5.7 Hz, 2 H), 7.10 (t, J = 8.6 Hz, 2 H), 2.93 (t, J = 7.2 Hz, 2 H), 2.87 (t, J = 7.4 Hz, 2 H), 1.73 (m, 4 H), 1.57 – 1.29 (m, 2 H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -41.21, -105.46 (tt, J = 8.4, 5.5 Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ

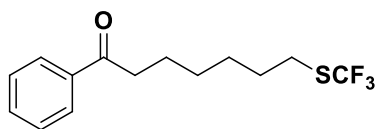
198.16, 166.92, 164.39, 133.31 (d, $J = 3.1$ Hz), 131.11 (q, $J = 307.0$ Hz), 123.09 (dd, $J = 9.3$ Hz, 21.9 Hz), 38.03, 29.62 (d, $J = 1.9$ Hz), 29.29, 28.04, 23.39 ppm. MS (EI): m/z (%) 123 (100). HRMS (EI) for $C_{13}H_{14}F_4OS$ Calcd: 294.0701; Found: 294.0695. IR (KBr): $\nu_{\max} = 1686, 1599\text{ cm}^{-1}$.

1,7-Diphenyl-6-((trifluoromethyl)thio)heptan-1-one 5h



The general procedure conducted with 2-benzyl-1-phenylcyclohexanol (79.9 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1,7-diphenyl-6-((trifluoromethyl)thio)heptan-1-one **5h** (61.6 mg, 56%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). 1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, $J = 7.9$ Hz, 2 H), 7.55 (t, $J = 7.4$ Hz, 1 H), 7.44 (t, $J = 7.6$ Hz, 2 H), 7.29 (t, $J = 7.2$ Hz, 2 H), 7.26 – 7.20 (m, 1 H), 7.17 (d, $J = 7.7$ Hz, 2 H), 3.50 – 3.30 (m, 1 H), 3.09 (dd, $J = 14.0, 5.7$ Hz, 1 H), 2.99 – 2.84 (m, 2 H), 1.59 (m, 7 H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -39.02 (s); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.90, 137.85, 136.90, 132.96, 131.18 (q, $J = 308.0$ Hz), 129.30, 128.55, 128.48, 127.97, 126.81, 47.38, 42.17, 38.11, 33.31, 25.97, 23.57 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for $C_{20}H_{21}F_3OS$ Calcd: 366.1265; Found: 366.1271. IR (KBr): $\nu_{\max} = 1686, 1597\text{ cm}^{-1}$.

1-Phenyl-7-((trifluoromethyl)thio)heptan-1-one 5i



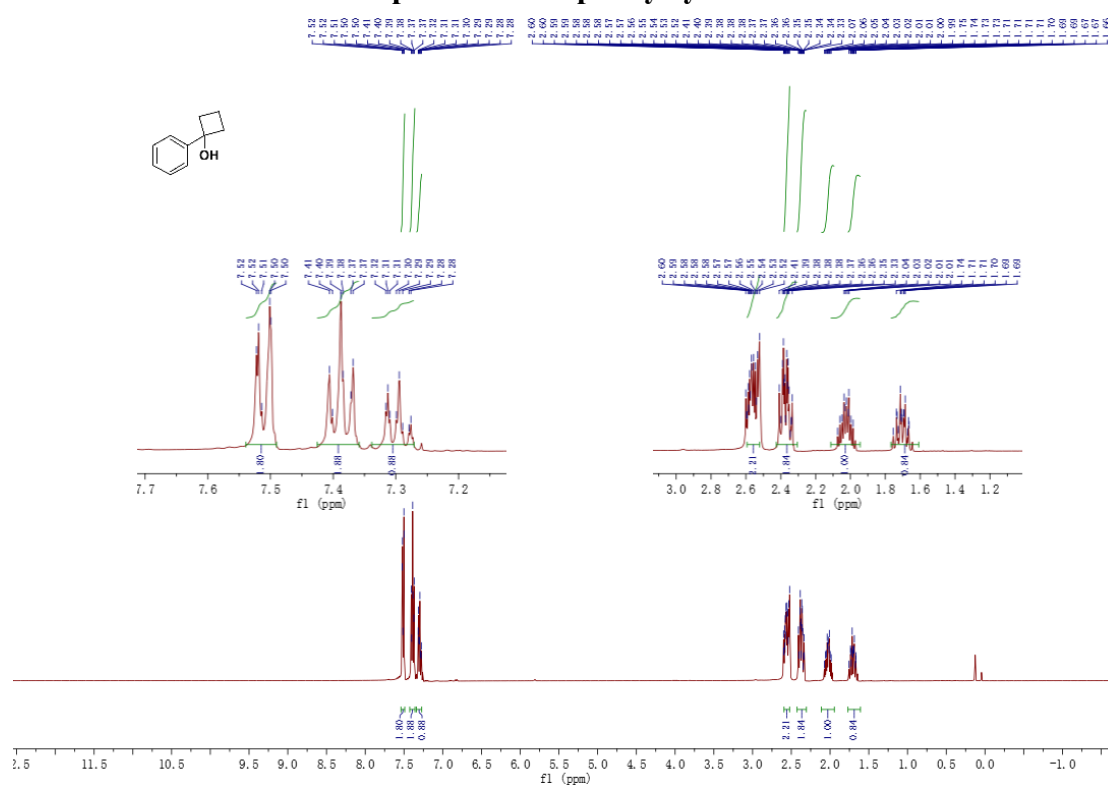
The general procedure conducted with 1-phenylcycloheptanol (57.1 mg, 0.300 mmol), S-(trifluoromethyl)benzenesulfonylthionate (145 mg, 0.600 mmol), $AgNO_3$ (10.2 mg, 6.00×10^{-2} mmol), SDS (18.0 mg, 6.00×10^{-2} mmol), $K_2S_2O_8$ (81.0 mg, 0.300 mmol) in H_2O (0.5 mL) gave 1-phenyl-7-((trifluoromethyl)thio)heptan-1-one **5i** (65.3 mg, 75%) as a colorless oil (eluent: petroleum ether: ethyl acetate = 100:1). 1H NMR (400

MHz, CDCl₃) δ 8.06 – 7.84 (m, 2 H), 7.54 (t, J = 7.4 Hz, 1 H), 7.44 (t, J = 7.6 Hz, 2 H), 2.96 (t, J = 7.3 Hz, 2 H), 2.86 (t, J = 7.4 Hz, 2 H), 1.72 (m, 4 H), 1.57 – 1.28 (m, 4 H); ¹⁹F NMR (376 MHz, CDCl₃) δ -41.22 (s); ¹³C NMR (101 MHz, CDCl₃) δ 199.66, 136.52, 132.43, 130.68 (q, J = 306.0 Hz), 128.07, 127.50, 37.81, 29.27 (d, J = 1.9 Hz), 28.74, 28.14, 27.83, 23.48 ppm. MS (EI): m/z (%) 105 (100). HRMS (EI) for C₁₄H₁₇F₃OS Calcd: 290.0952; Found: 290.0956. IR (KBr): ν_{max} = 2859, 1686 cm⁻¹.

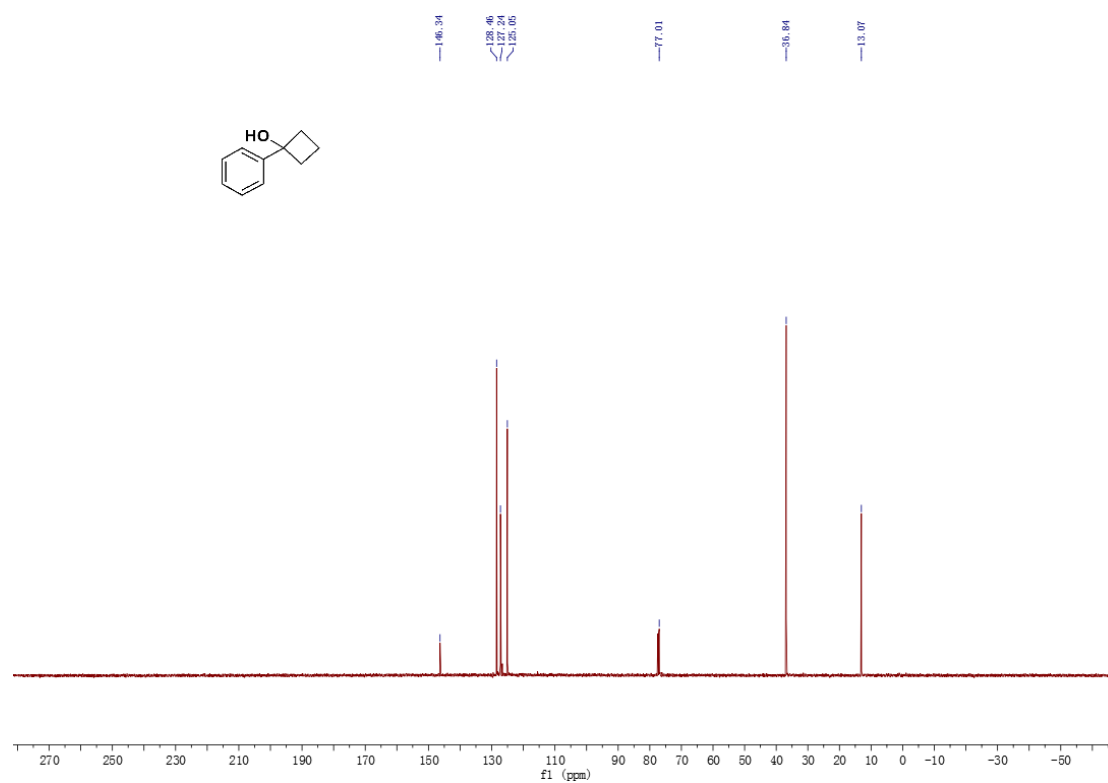
References

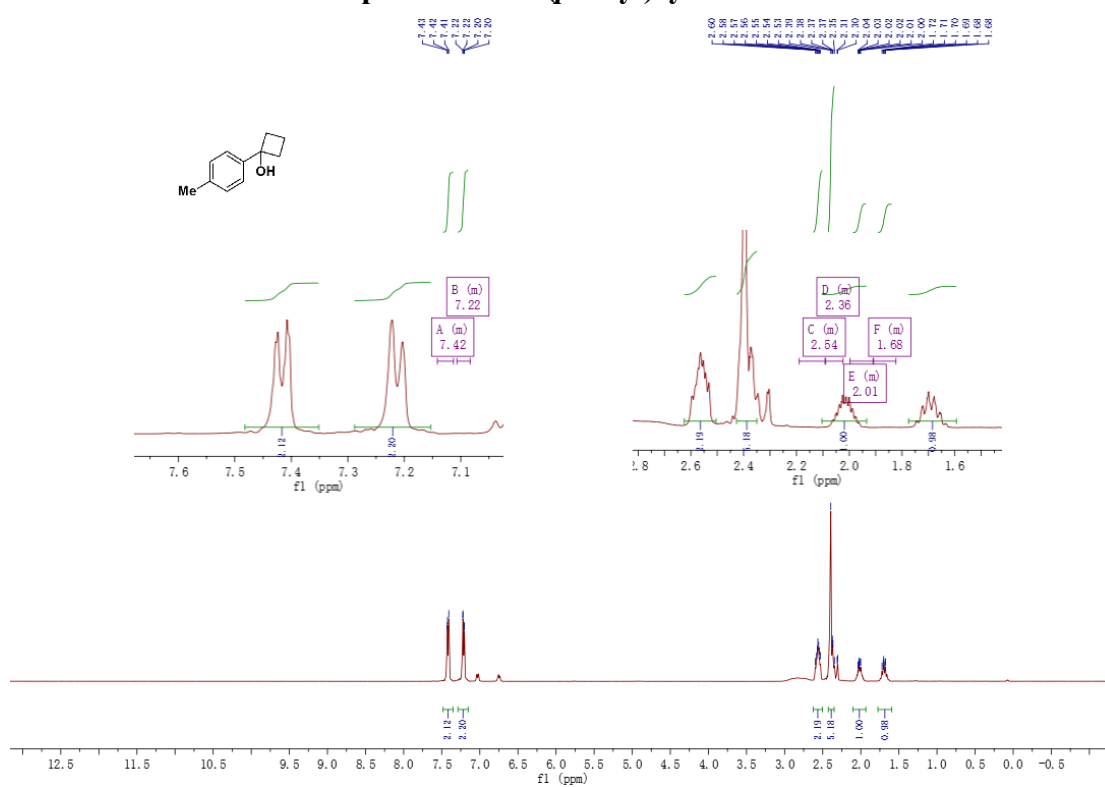
- [1] Zhao, H.-J.; Fan, X.-F.; Yu, J.-J.; and Zhu, C. *J. Am. Chem. Soc.* **2015**, *137*, 3490.
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¹H NMR spectrum of 1-phenylcyclobutanol 1a

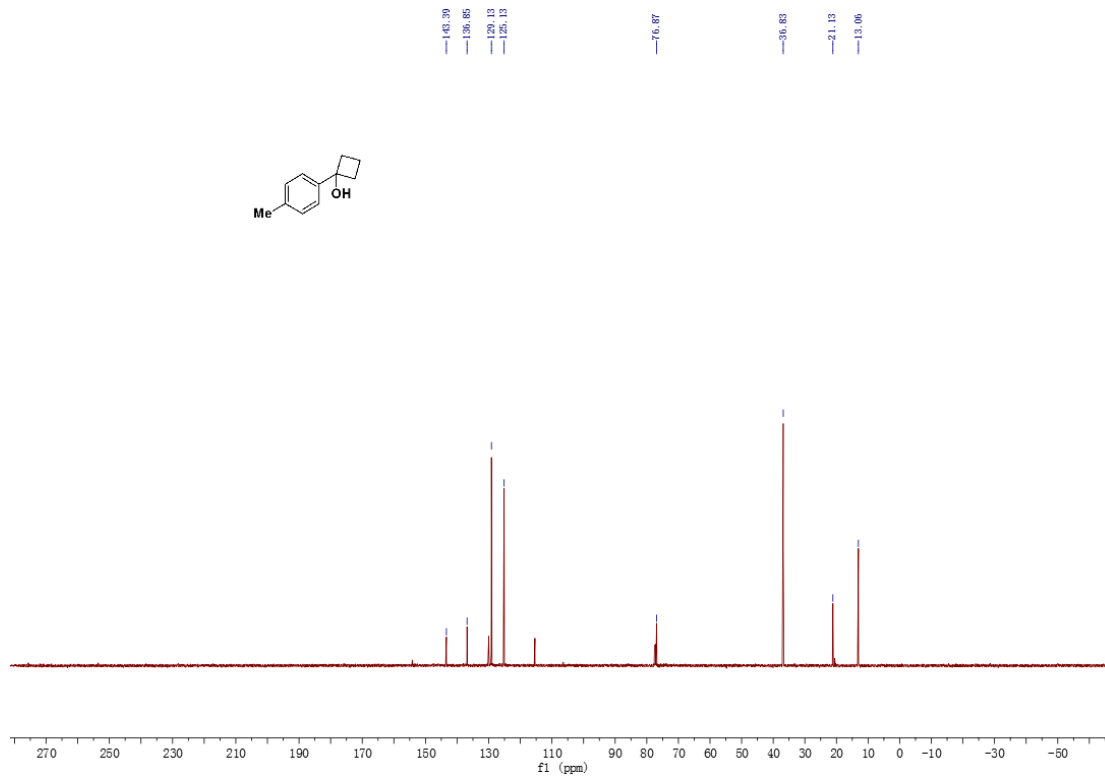


¹³C NMR spectrum of 1-phenylcyclobutanol 1a

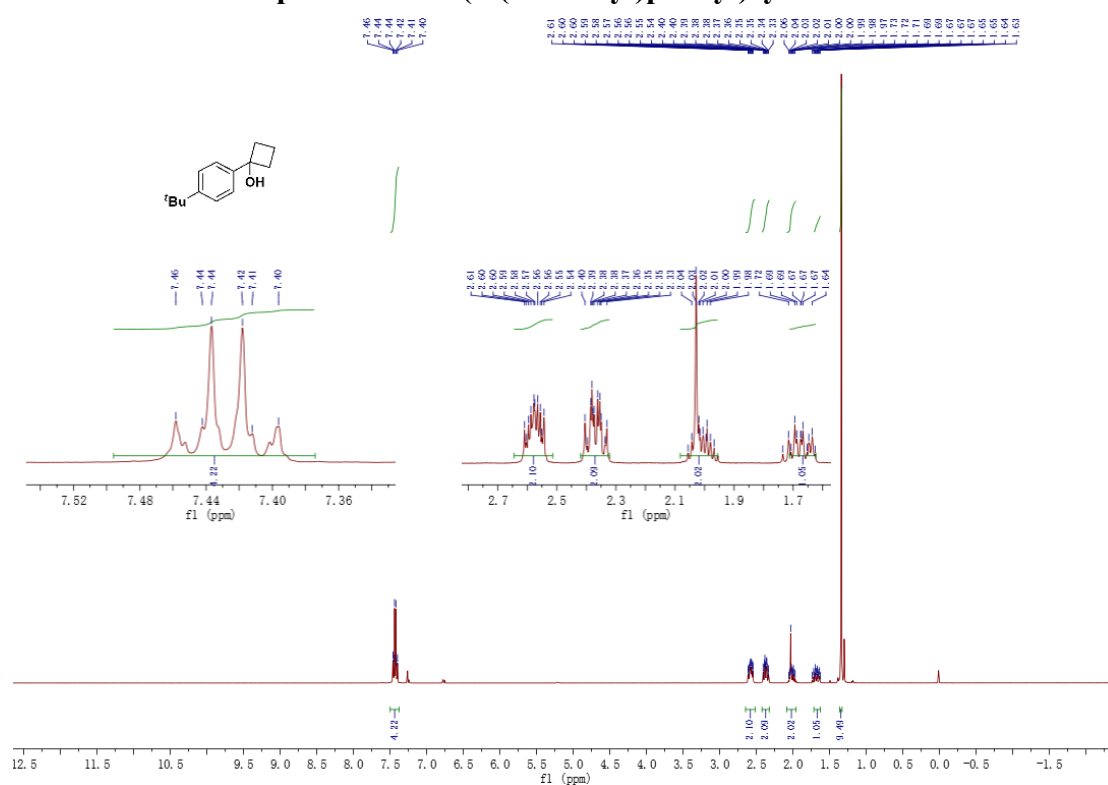


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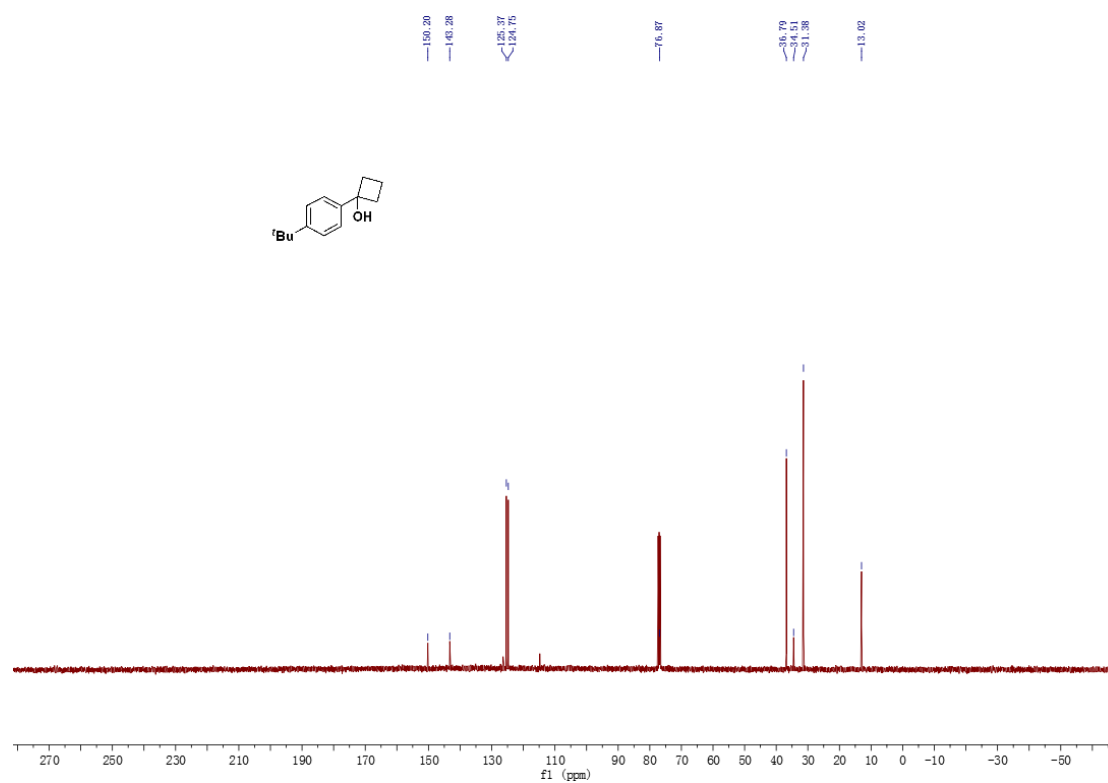
CC1=CC=C(C=C1)C2(O)C3C(C2)C4C3C4



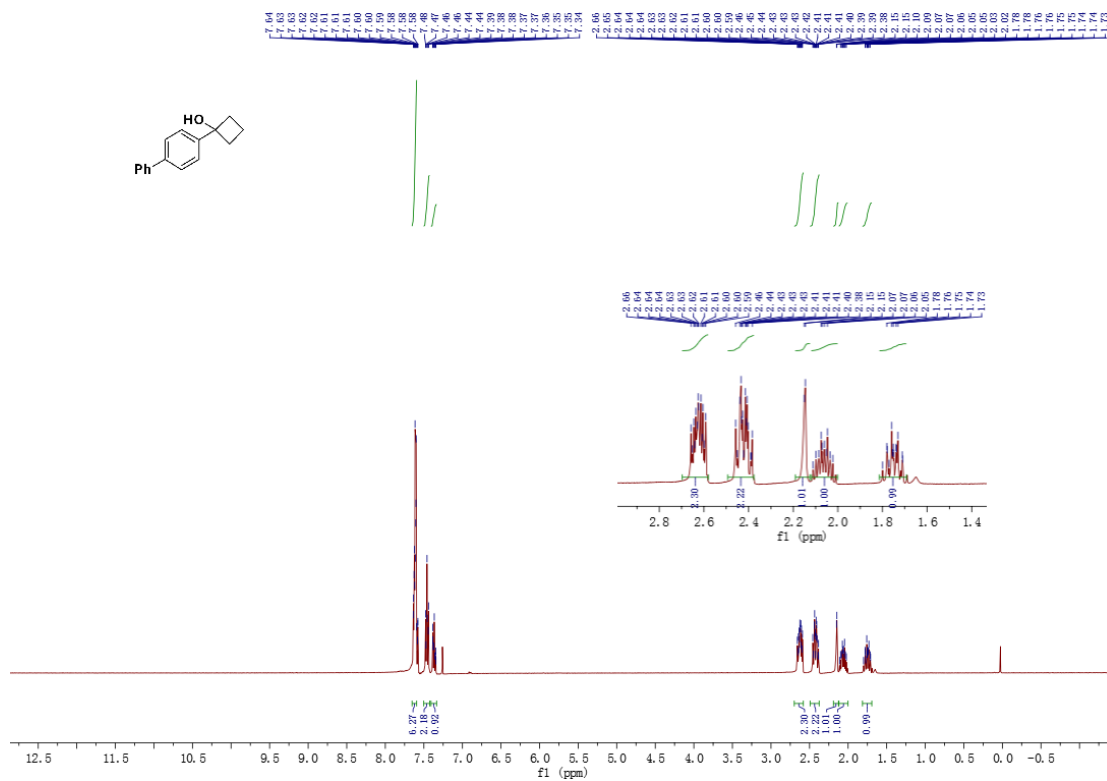
¹H NMR spectrum of 1-(4-(*tert*-butyl)phenyl)cyclobutan-1-ol 1c



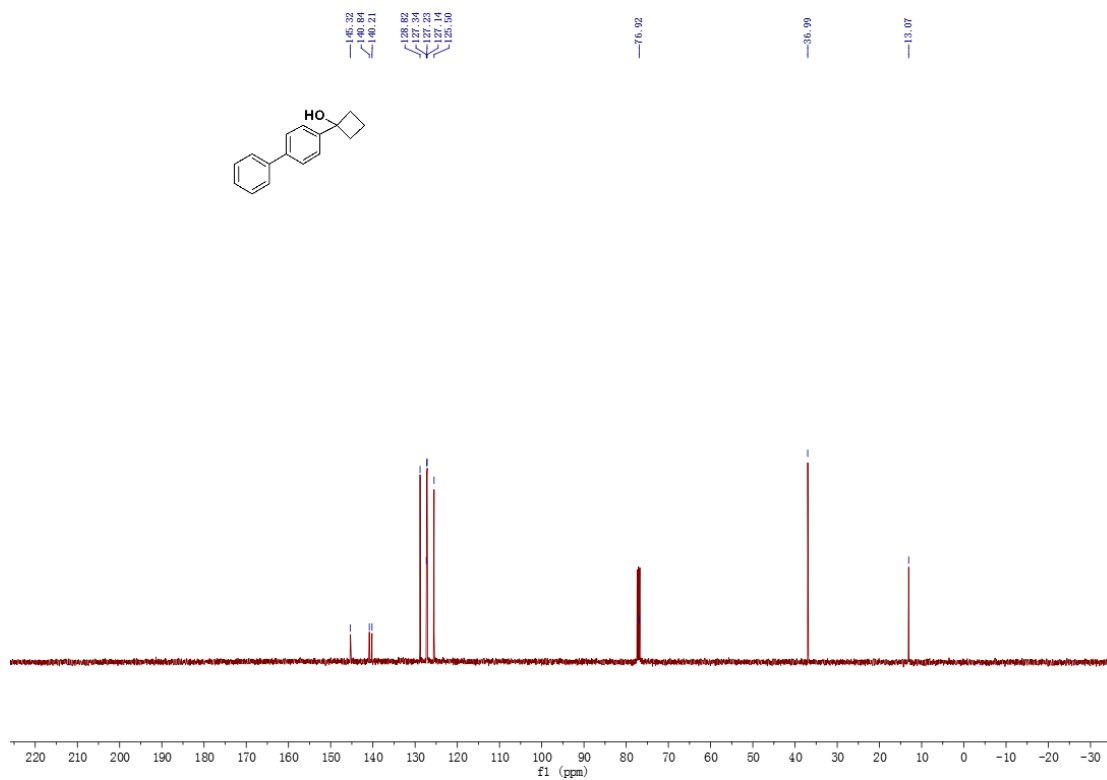
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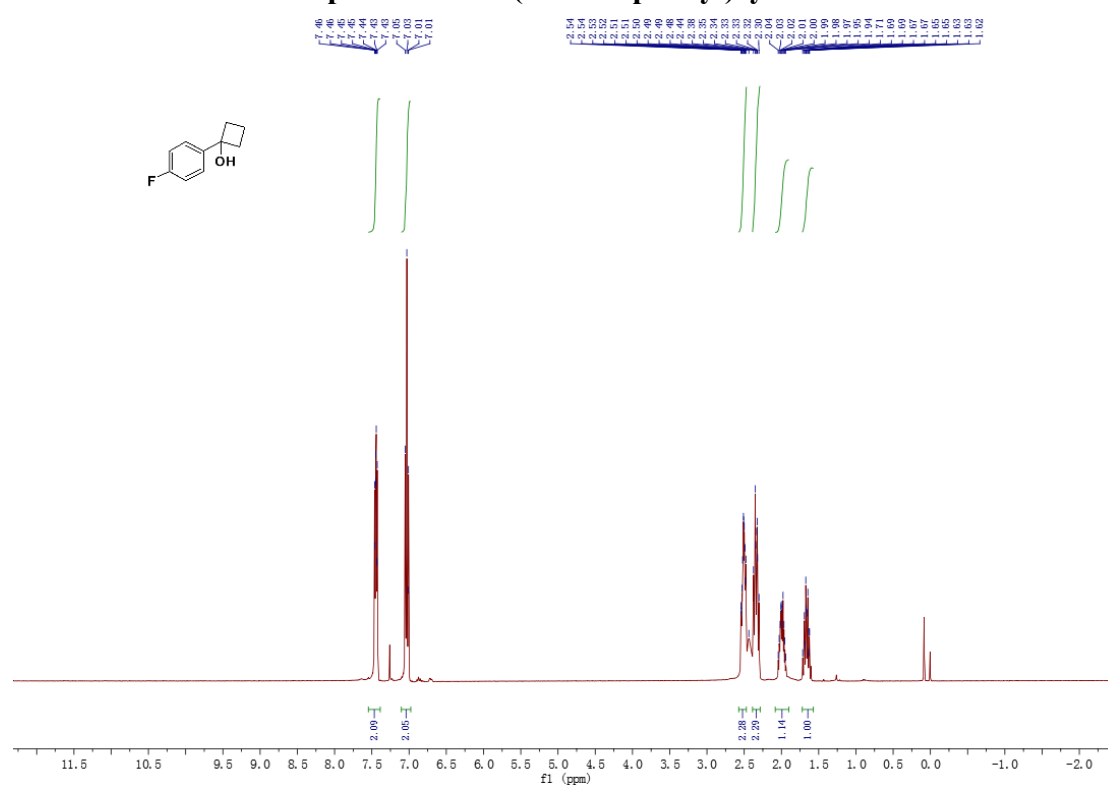
**^1H NMR spectrum of
1-([1,1'-biphenyl]-4-yl)cyclobutan-1-ol 1d**



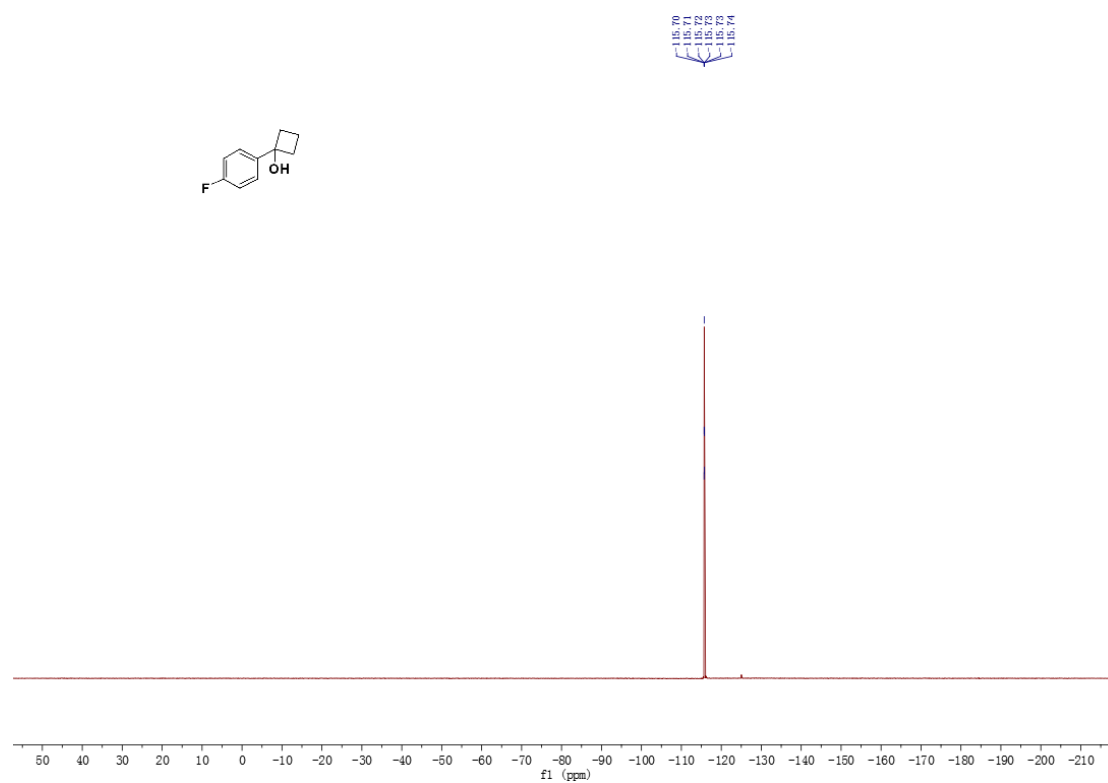
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1-([1,1'-biphenyl]-4-yl)cyclobutan-1-ol 1d**



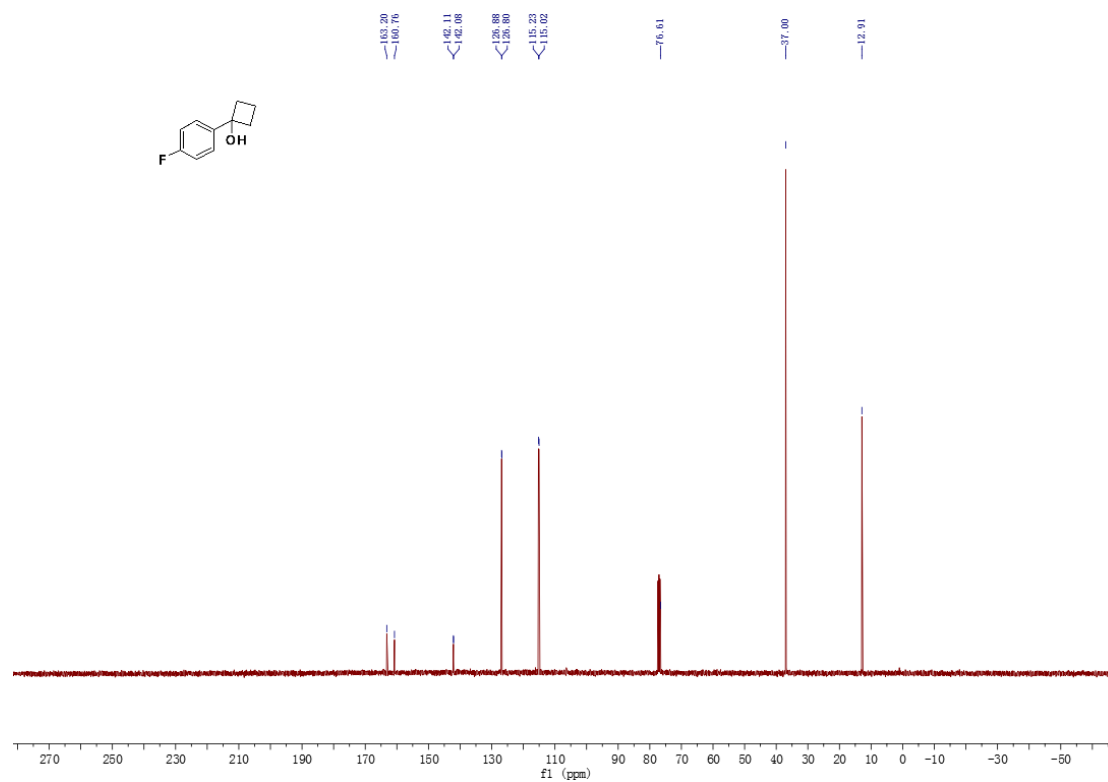
¹H NMR spectrum of 1-(4-fluorophenyl)cyclobutanol 1e



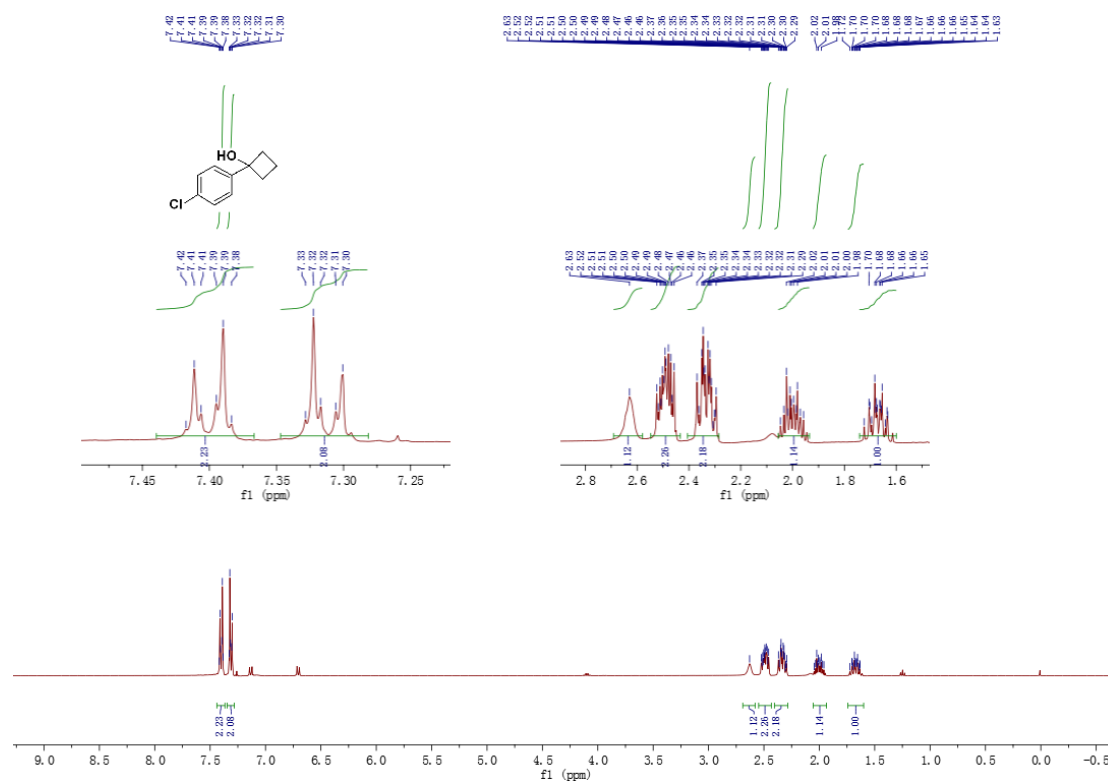
¹⁹F NMR spectrum of 1-(4-fluorophenyl)cyclobutanol 1e



^{13}C NMR spectrum of 1-(4-fluorophenyl)cyclobutanol 1e



^1H NMR spectrum of 1-(4-chlorophenyl)cyclobutanol 1f

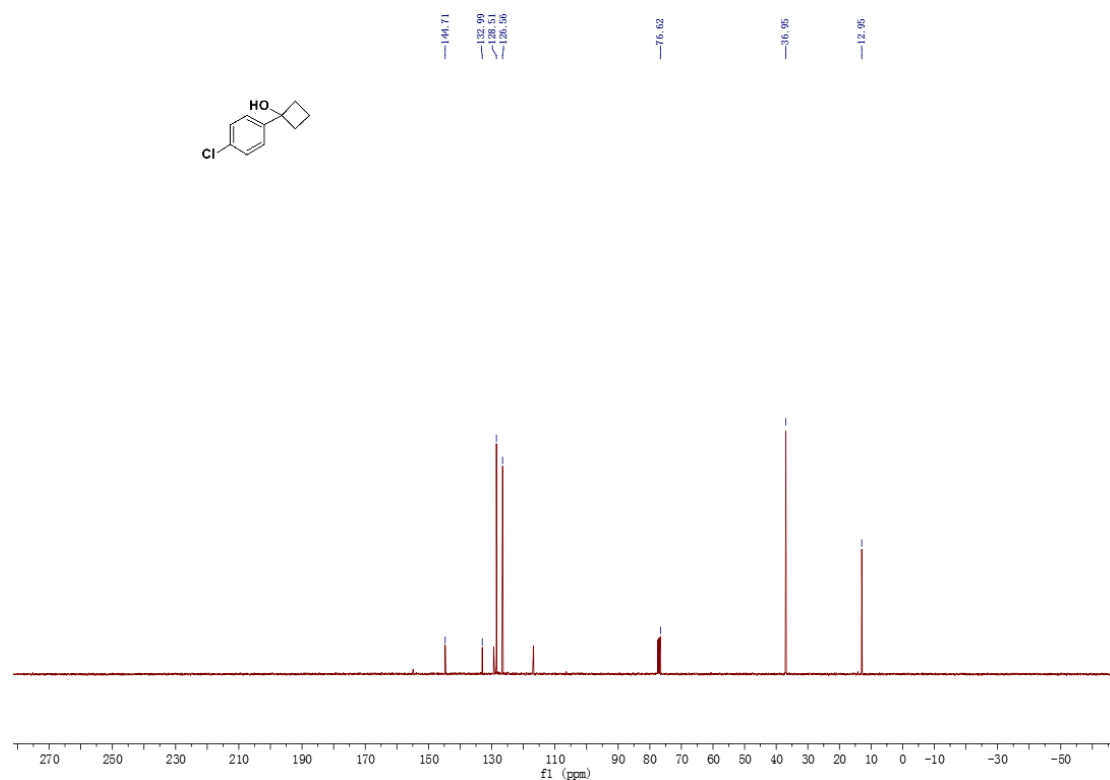


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—128.51
—126.56

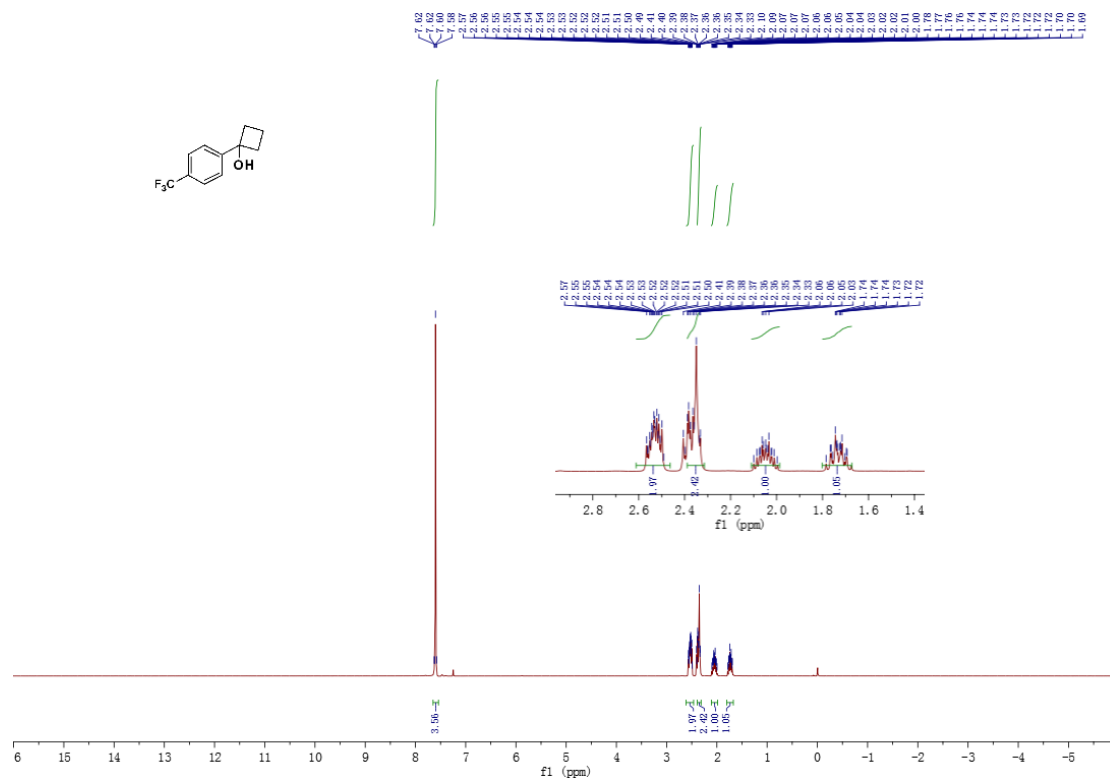
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—36.95

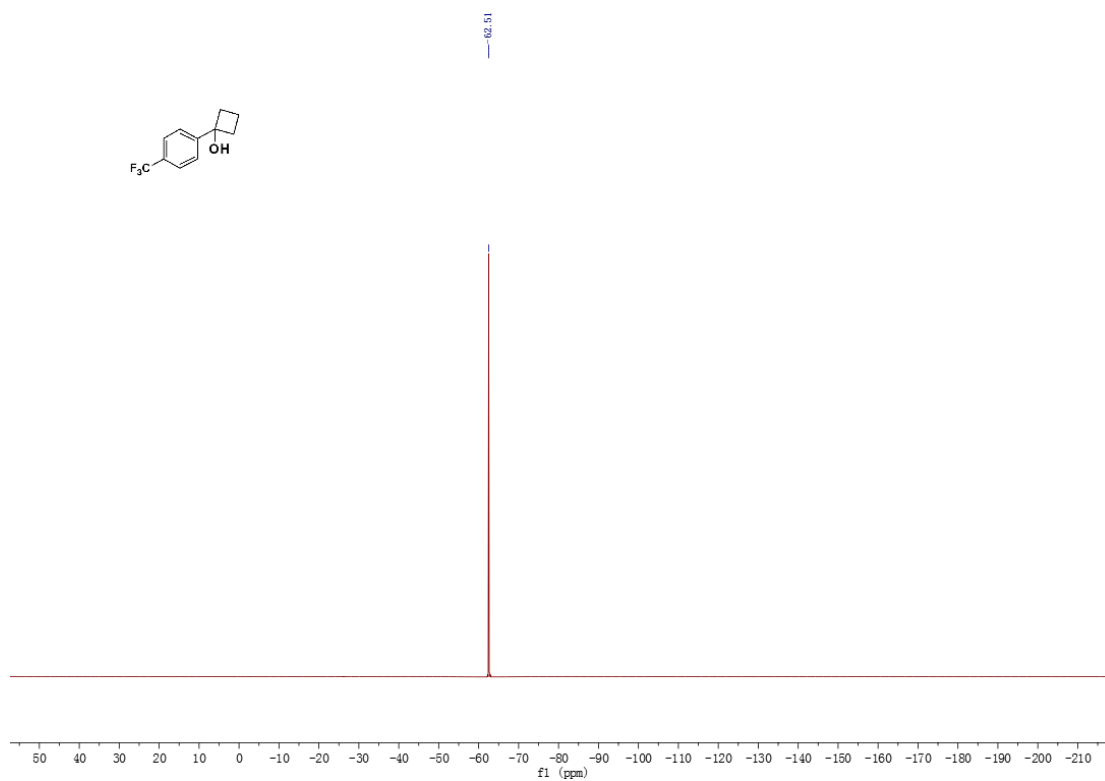
—12.95



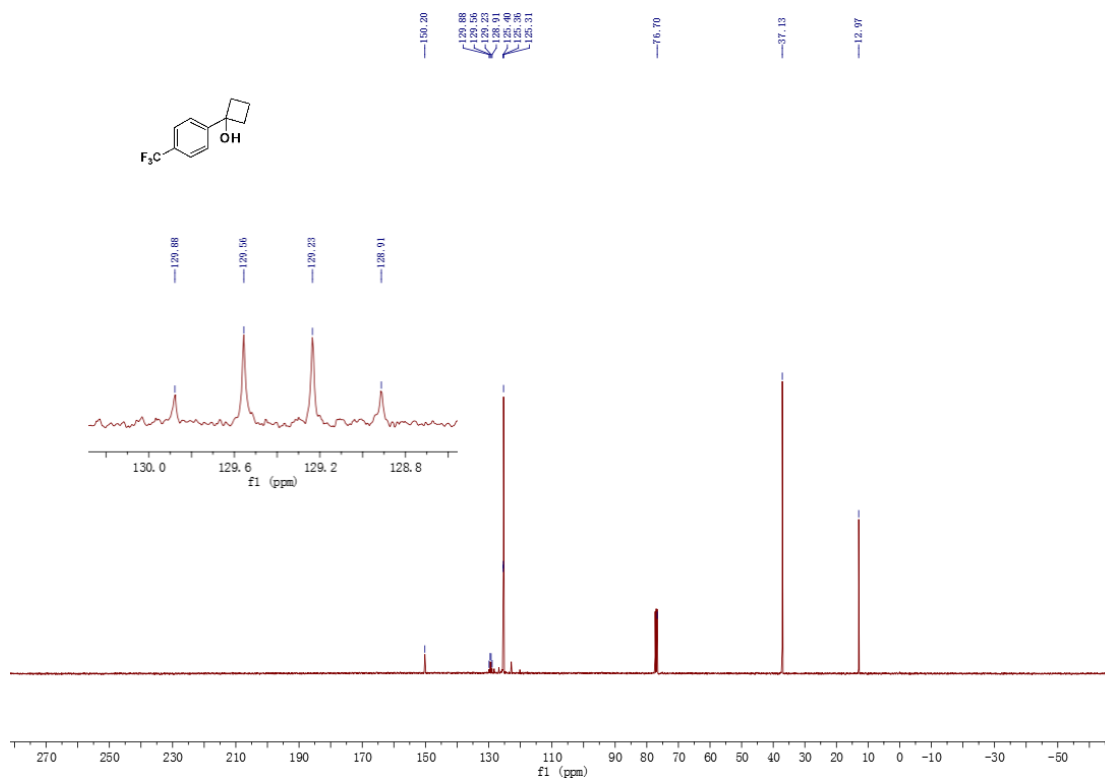
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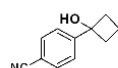
**^{19}F NMR spectrum of
1-(4-(trifluoromethyl)phenyl)cyclobutanol 1g**



**^{13}C NMR spectrum of
1-(4-(trifluoromethyl)phenyl)cyclobutanol 1g**



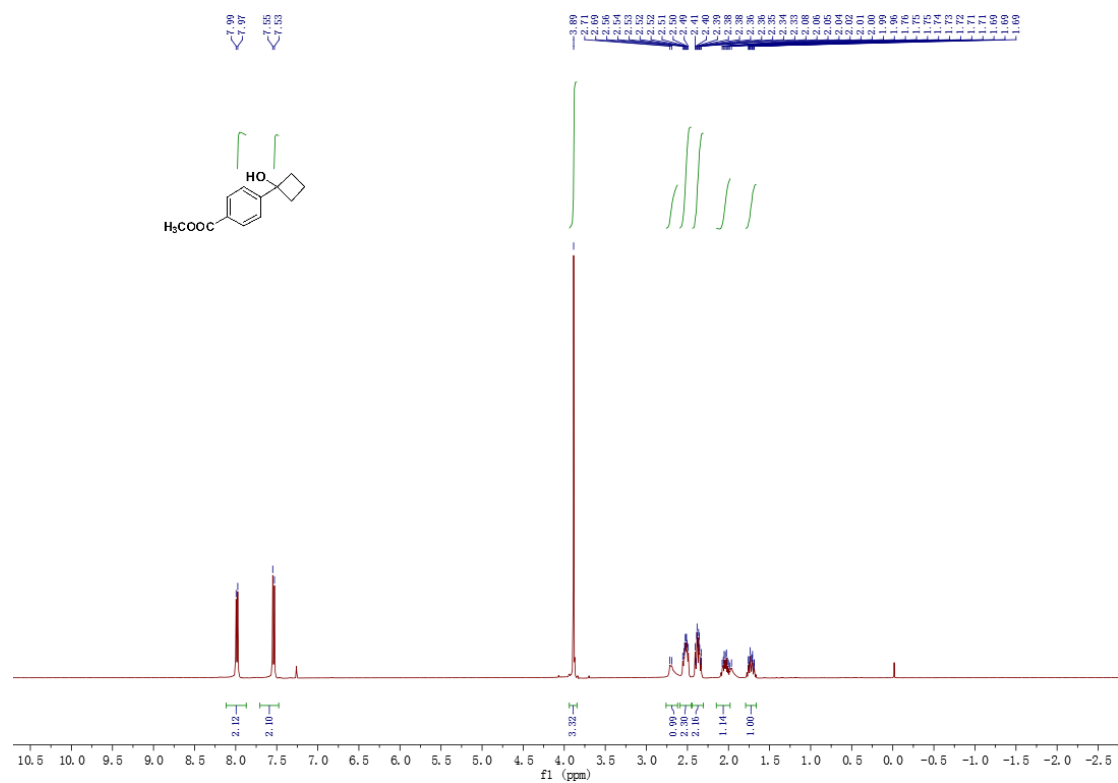
Chemical structure of 4-cyano-4'-(cyclobutyl)phenol (1): N#Cc1ccc(cc1C2CCC2)O



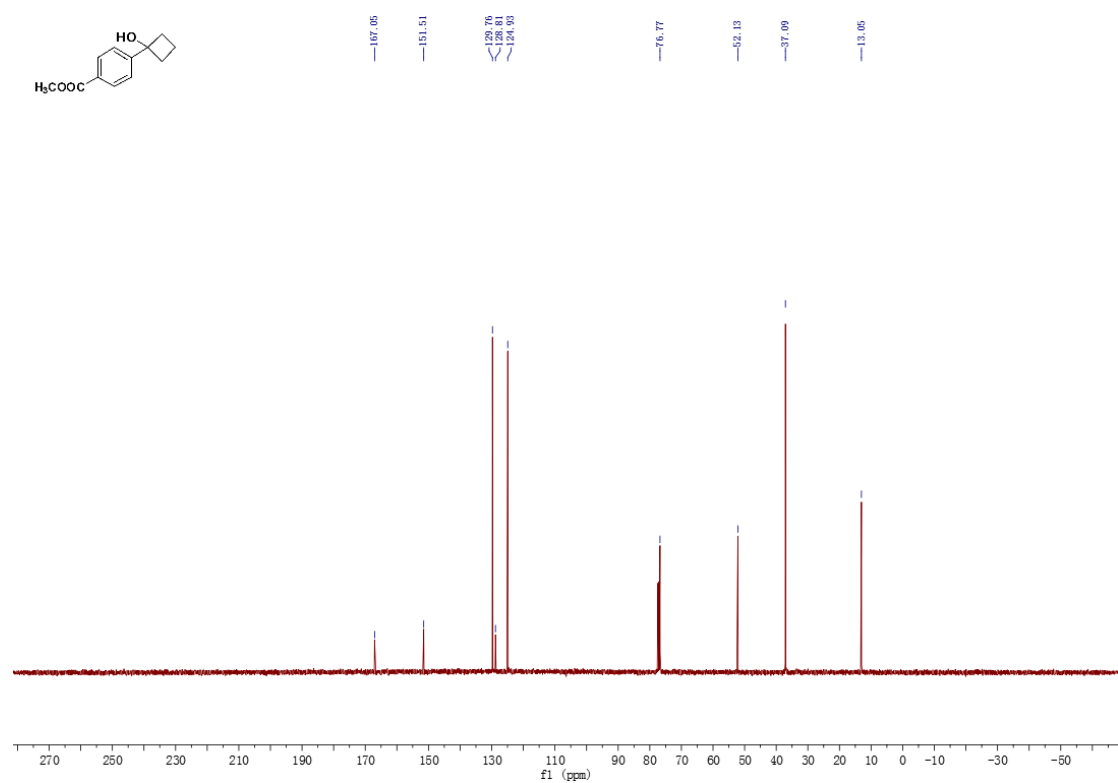
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—125.75
—118.93
—110.53
—76.50
—37.23
—13.02



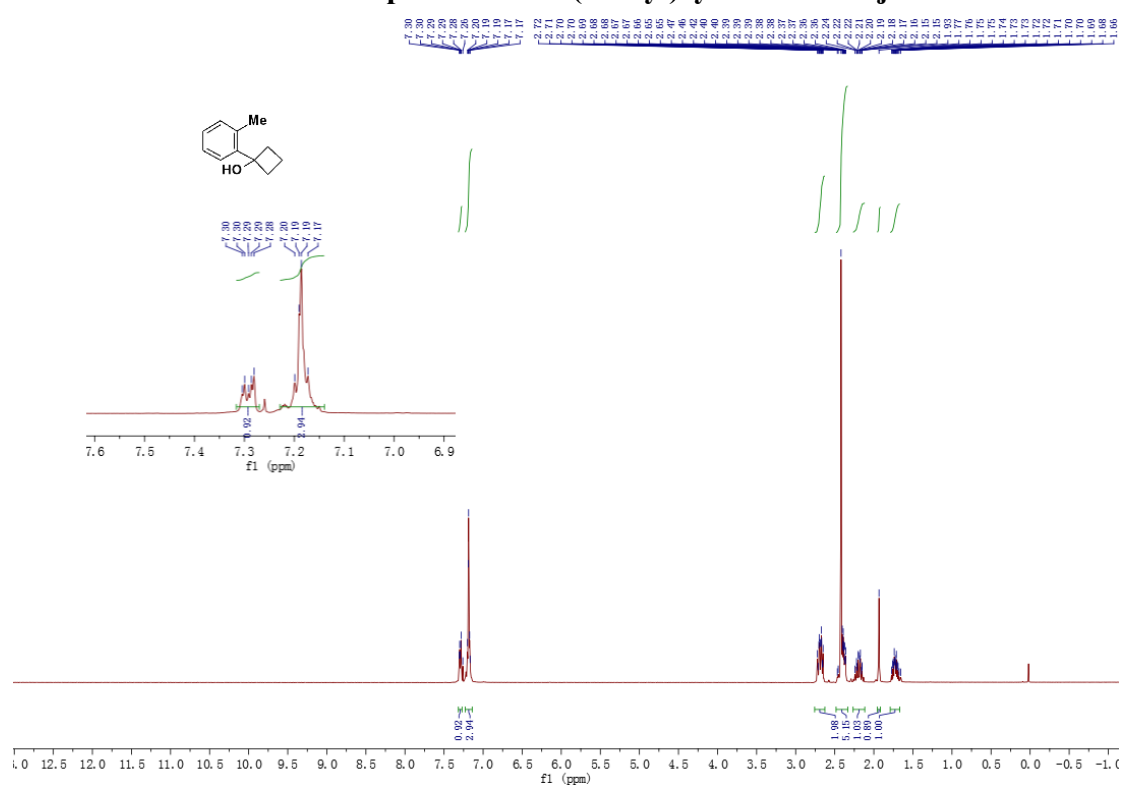
**¹H NMR spectrum of
methyl 4-(1-hydroxycyclobutyl)benzoate 1i**



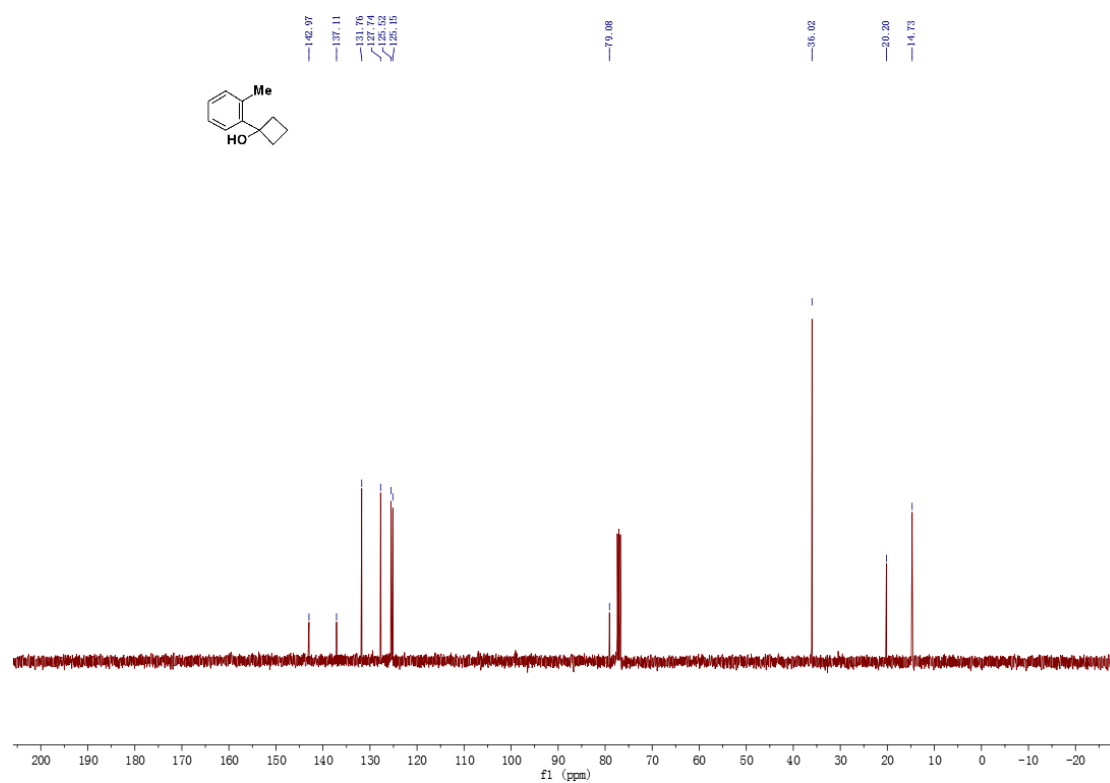
**¹³C NMR spectrum of
methyl 4-(1-hydroxycyclobutyl)benzoate 1i**



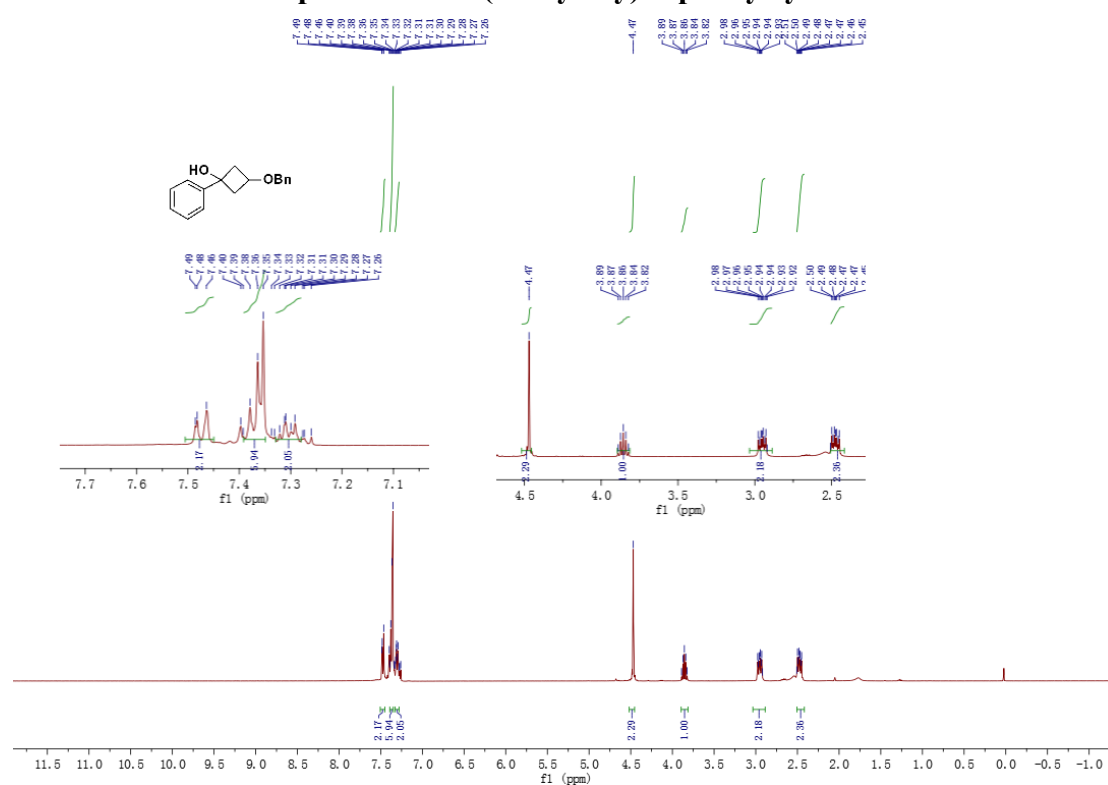
¹H NMR spectrum of 1-(*o*-tolyl)cyclobutanol 1j



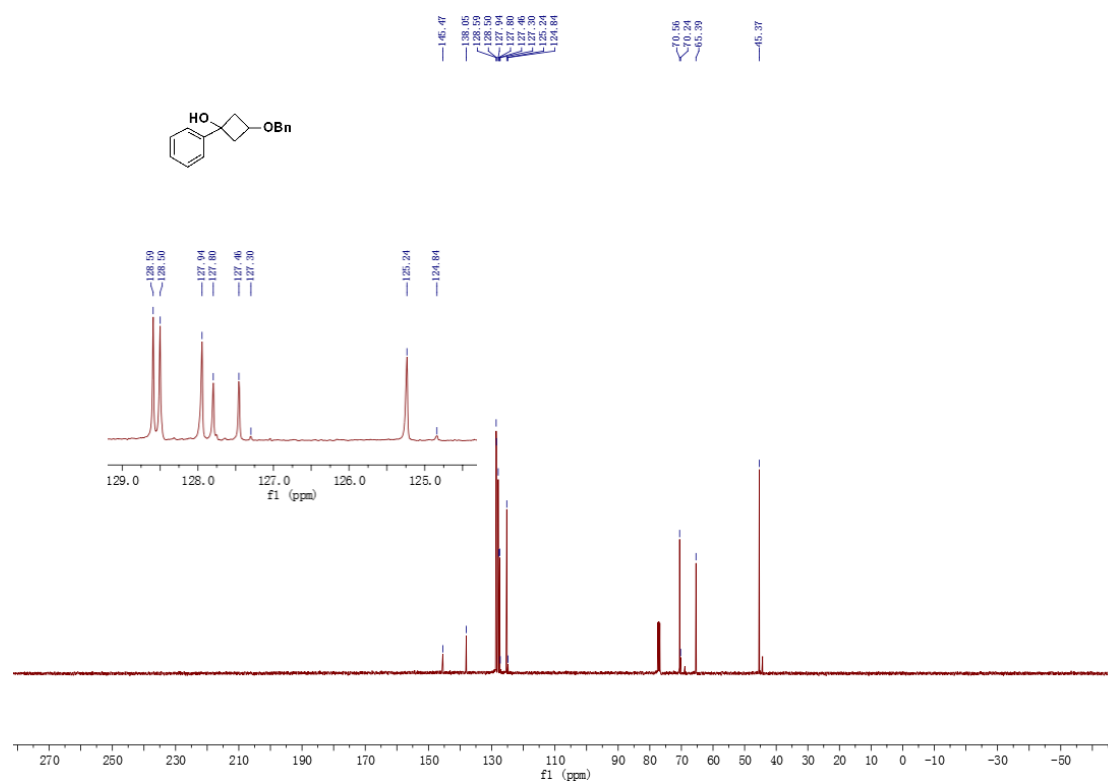
¹³C NMR spectrum of 1-(*o*-tolyl)cyclobutanol 1j



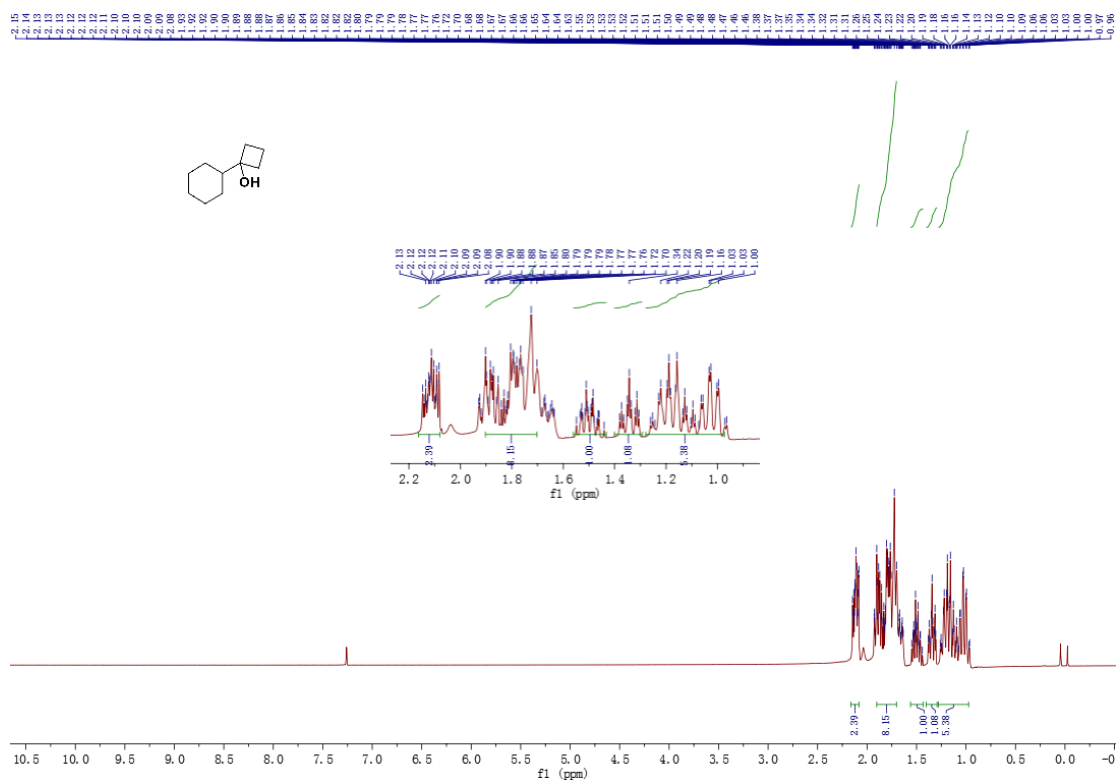
¹H NMR spectrum of 3-(benzyloxy)-1-phenylcyclobutanol 11



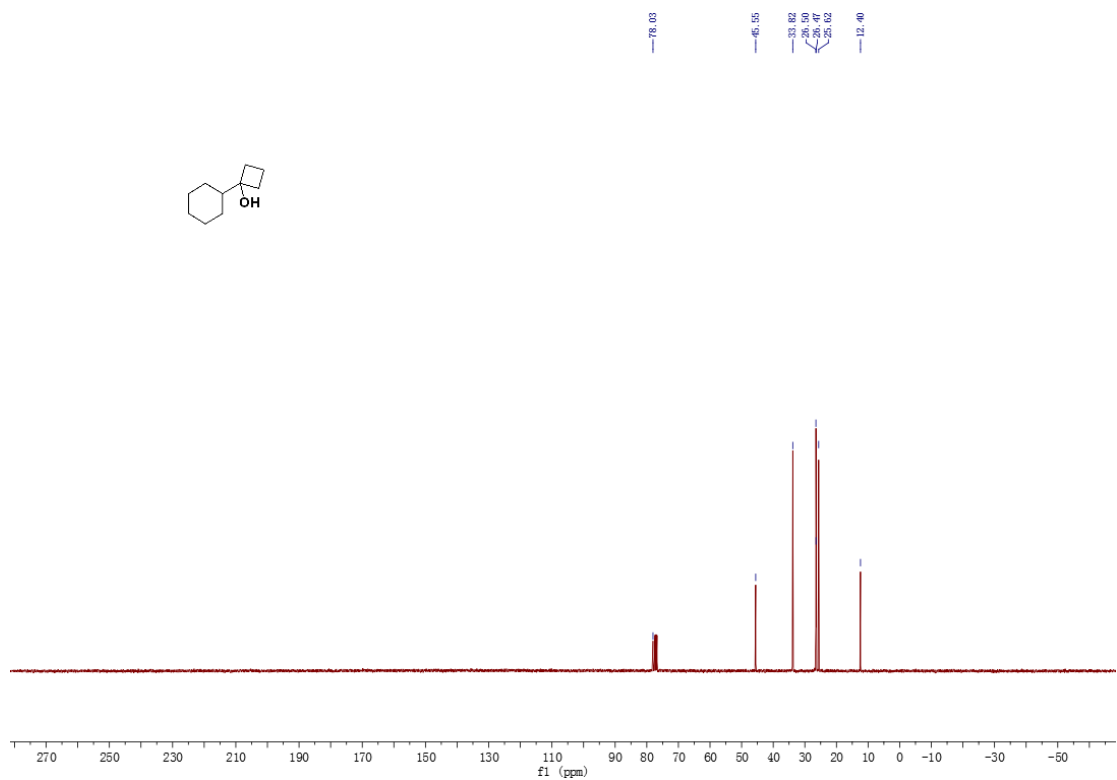
¹³C NMR spectrum of 3-(benzyloxy)-1-phenylcyclobutanol 11



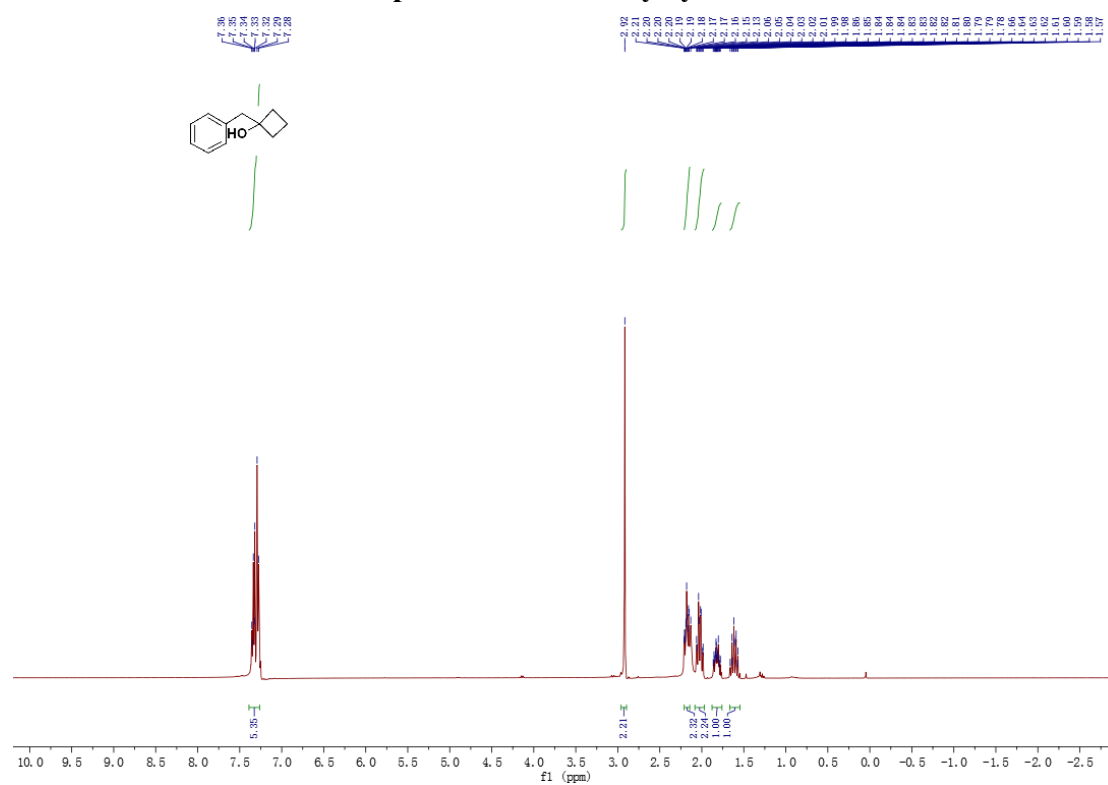
¹H NMR spectrum of 1-cyclohexylcyclobutanol 1m



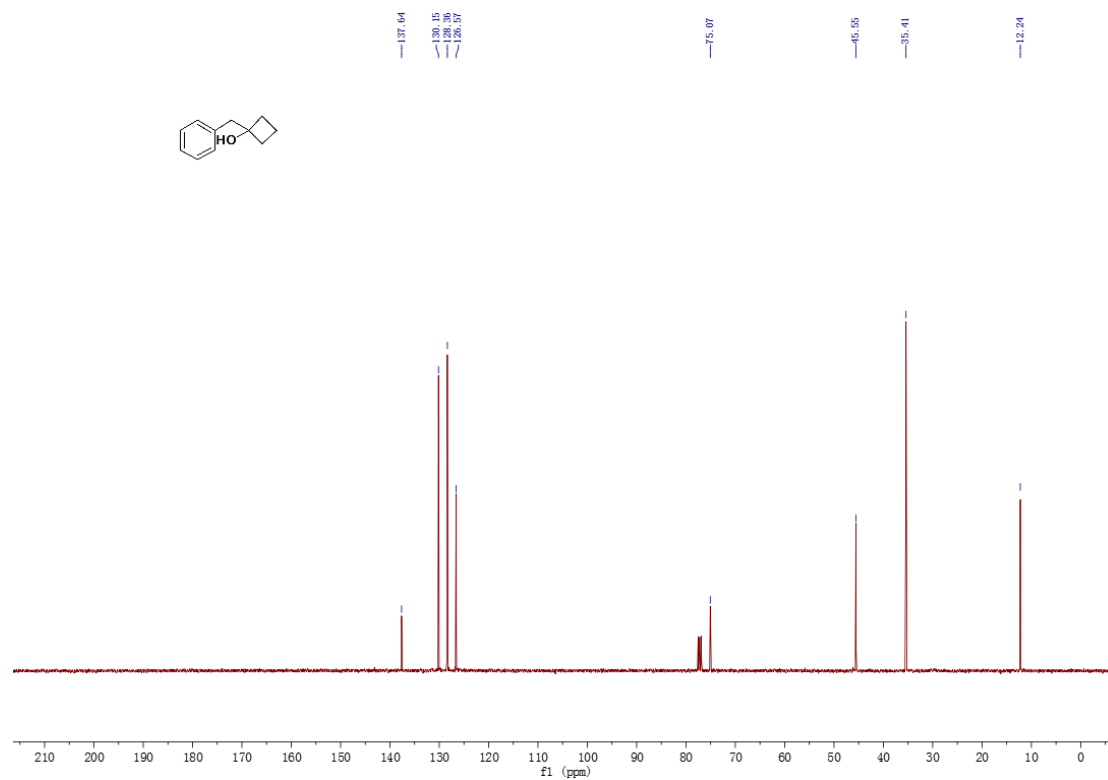
¹³C NMR spectrum of 1-cyclohexylcyclobutanol 1m



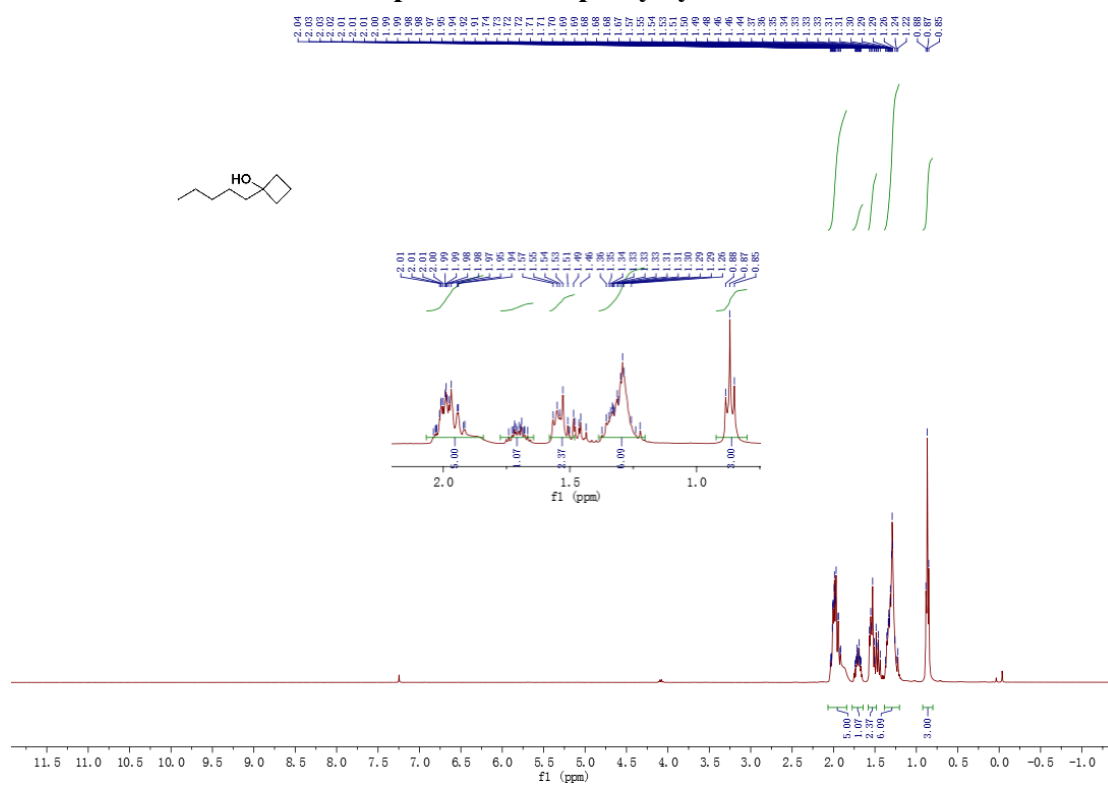
¹H NMR spectrum of 1-benzylcyclobutanol 1n



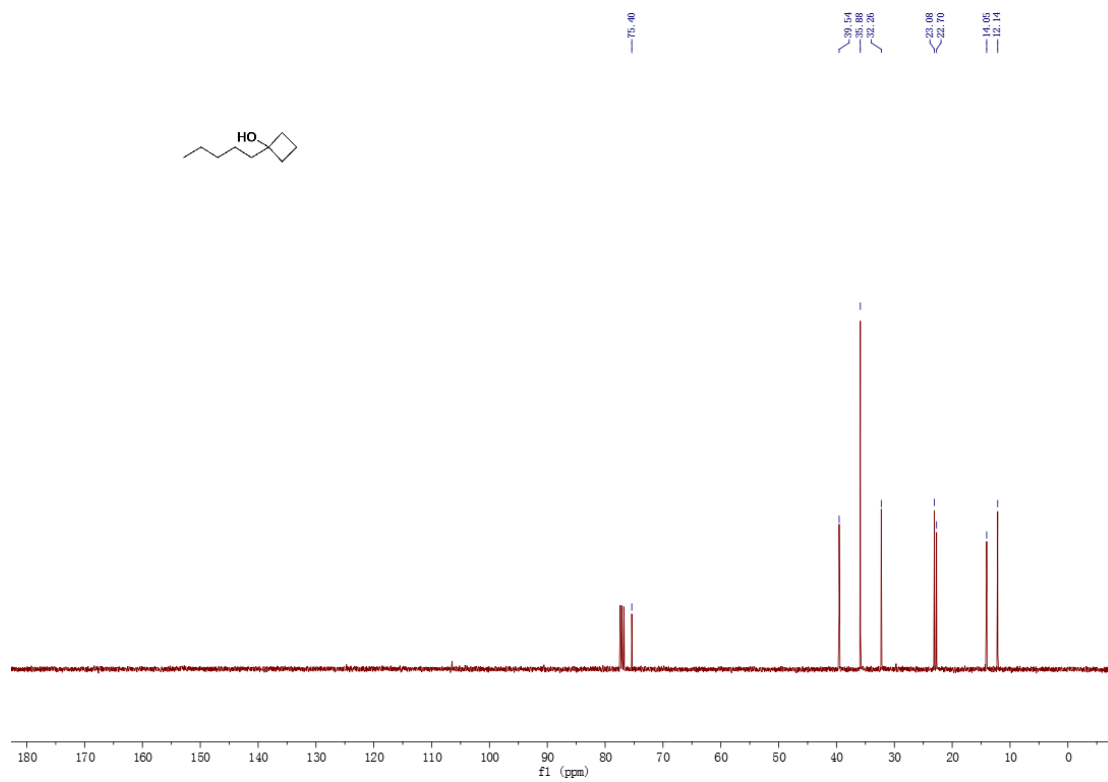
¹³C NMR spectrum of 1-benzylcyclobutanol 1n



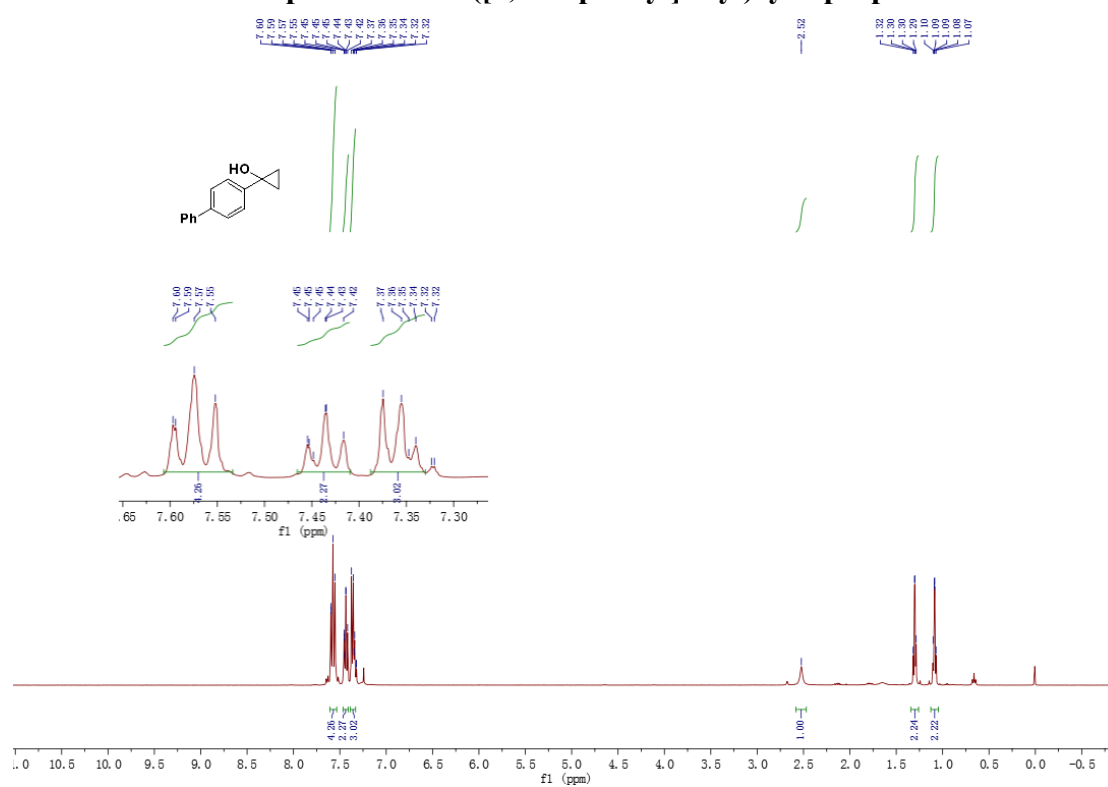
¹H NMR spectrum of 1-pentylcyclobutanol 1o



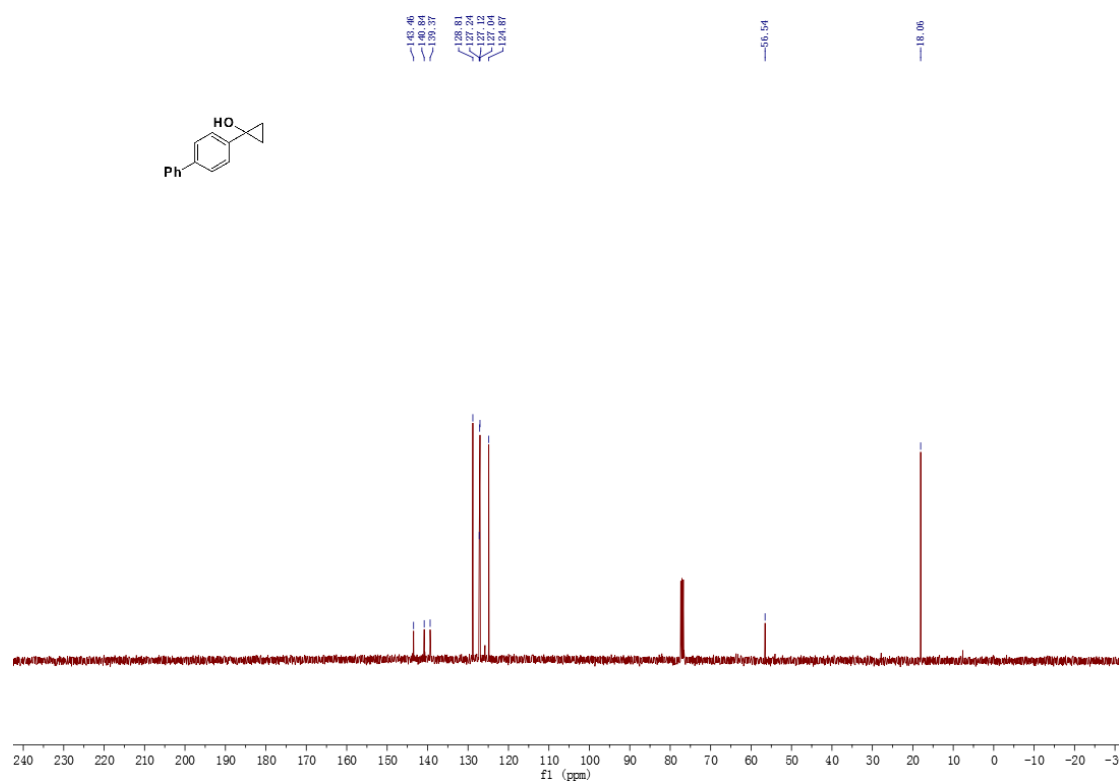
^{13}C NMR spectrum of 1-pentylcyclobutanol 1o



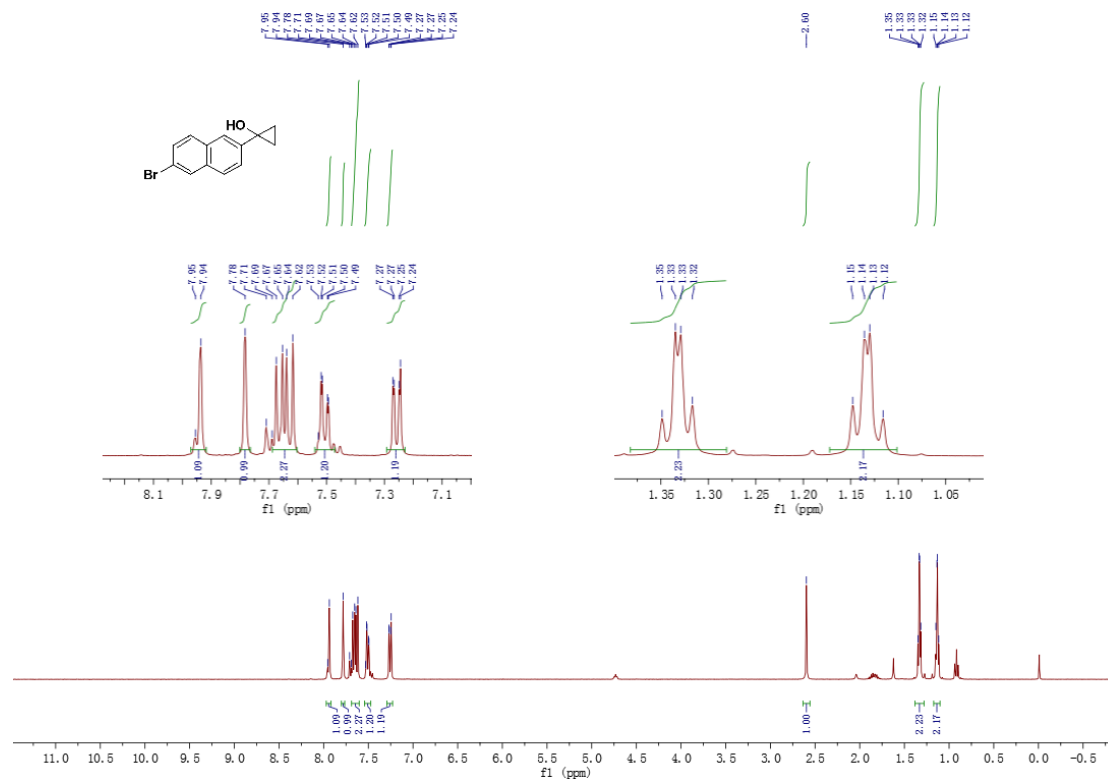
¹H NMR spectrum of 1-([1,1'-biphenyl]-4-yl)cyclopropanol 3b



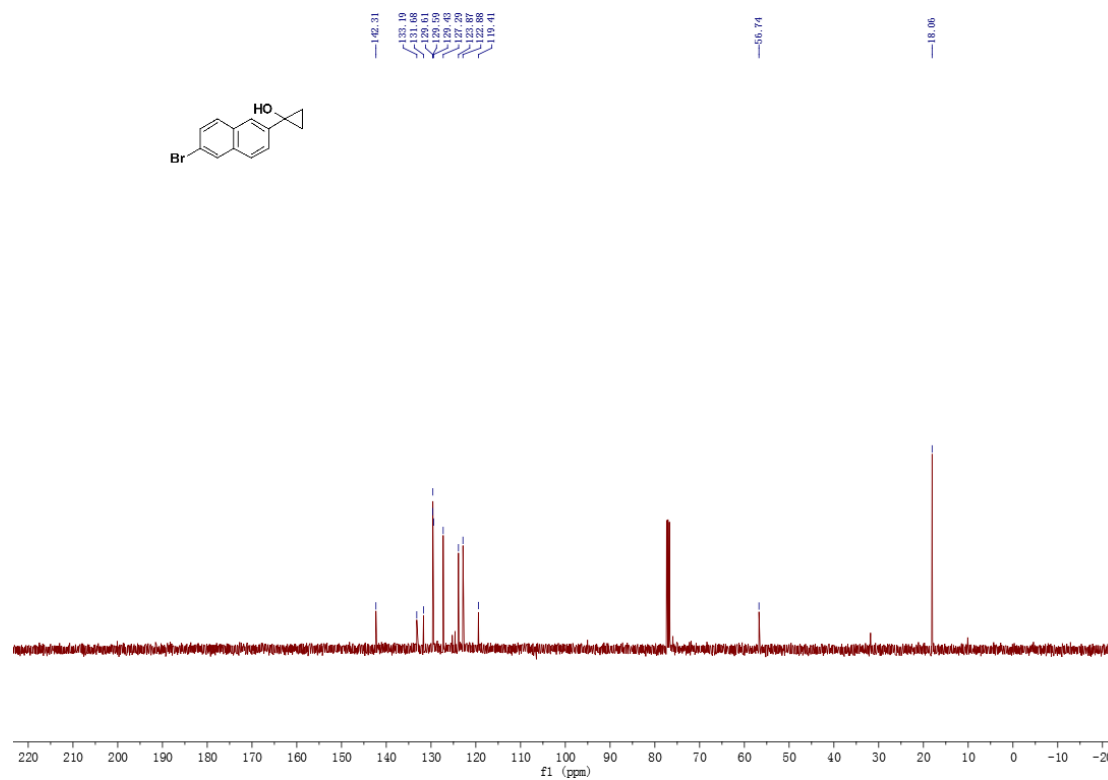
¹³C NMR spectrum of 1-([1,1'-biphenyl]-4-yl)cyclopropanol 3b



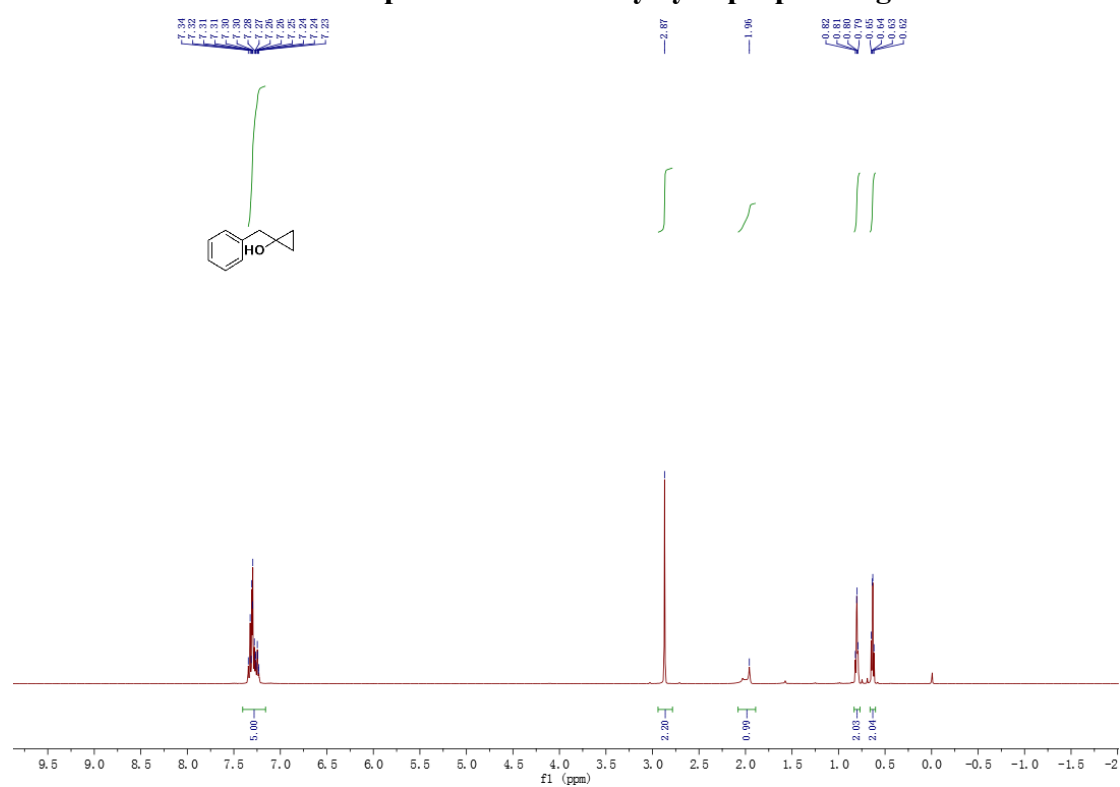
**^1H NMR spectrum of
1-(6-bromonaphthalen-2-yl)cyclopropanol 3d**



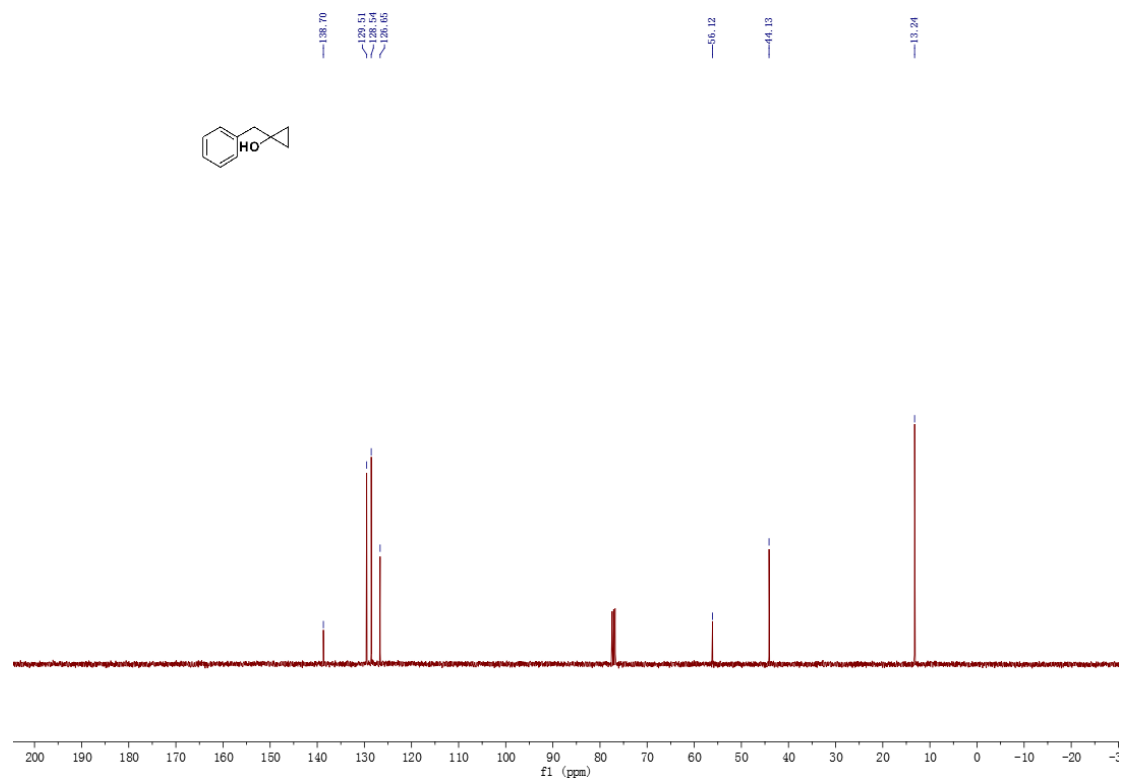
**^{13}C NMR spectrum of
1-(6-bromonaphthalen-2-yl)cyclopropanol 3d**



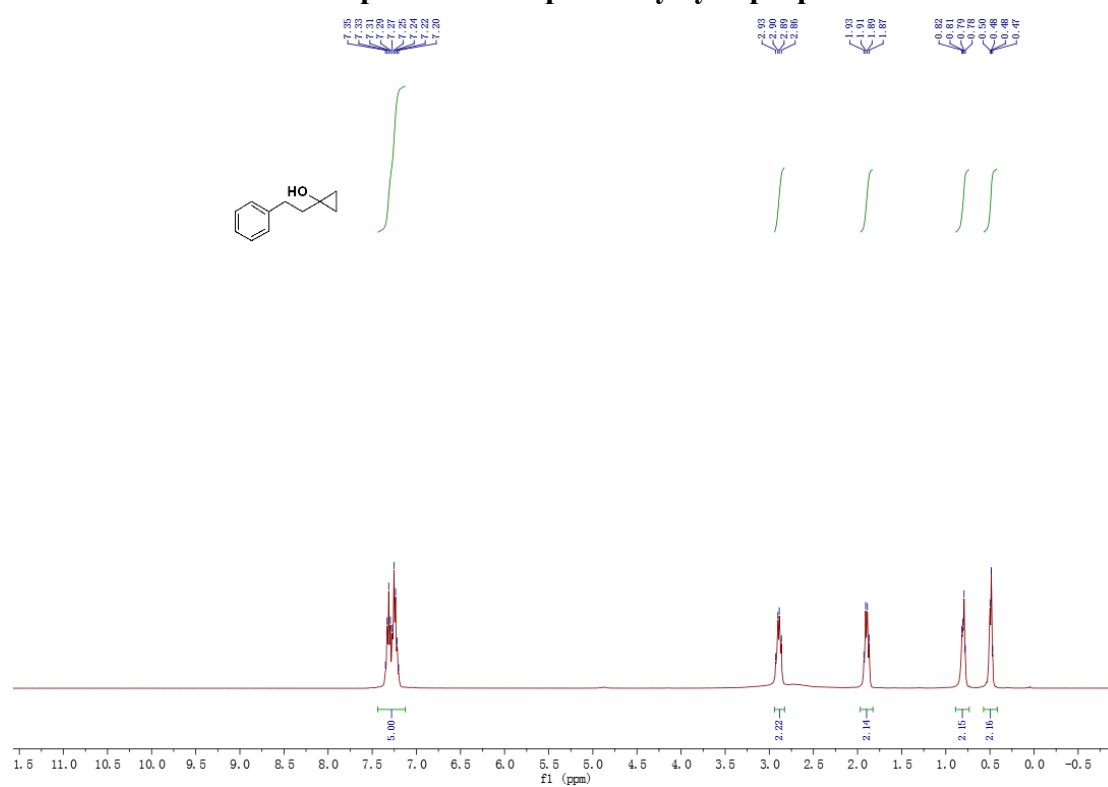
¹H NMR spectrum of 1-benzylcyclopropanol 3g



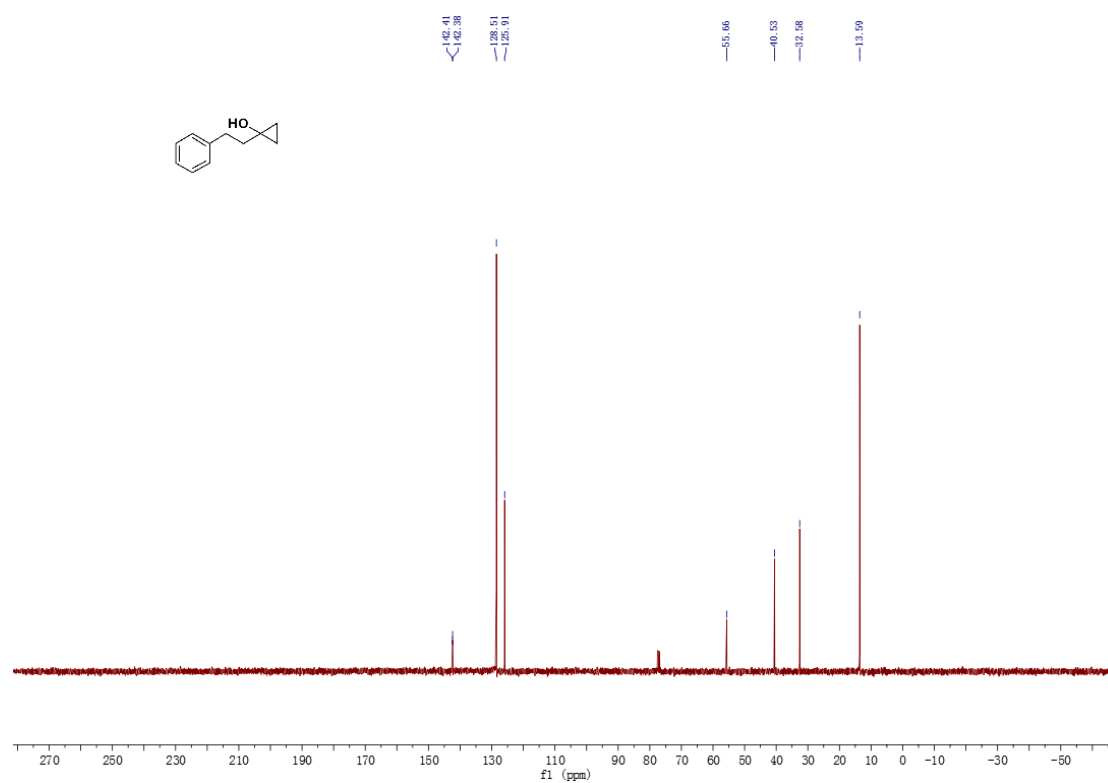
¹³C NMR spectrum of 1-benzylcyclopropanol 3g



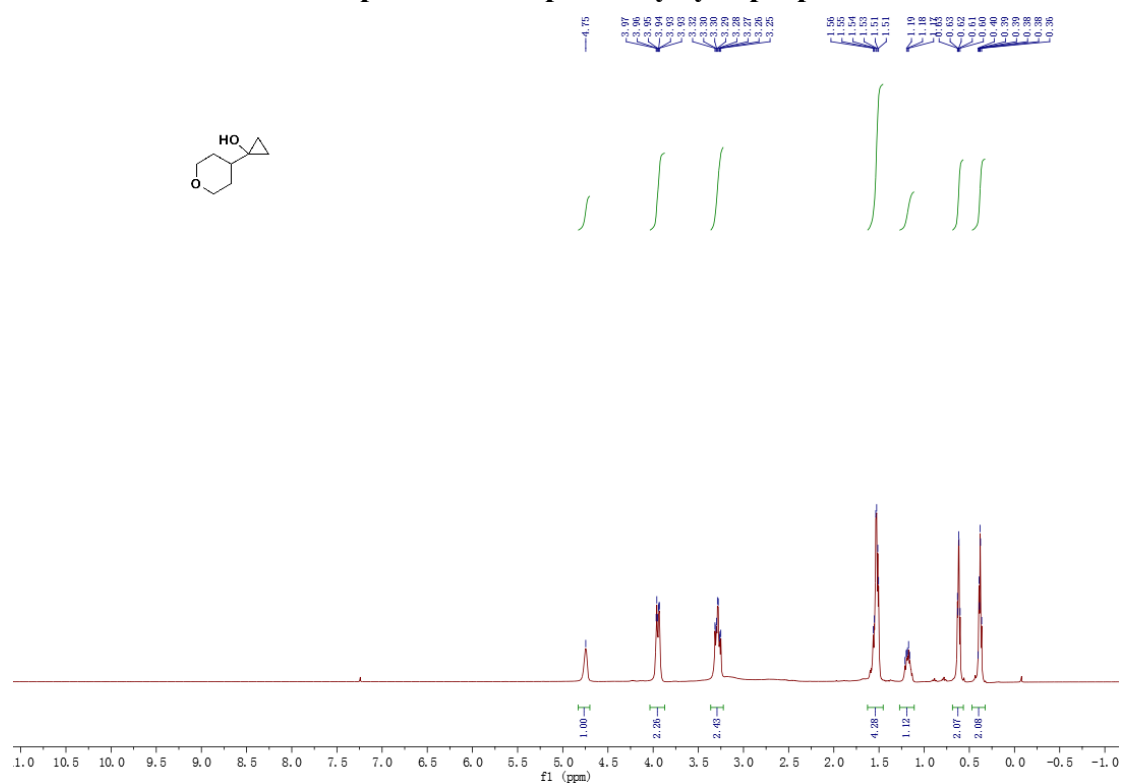
¹H NMR spectrum of 1-phenethylcyclopropanol 3h



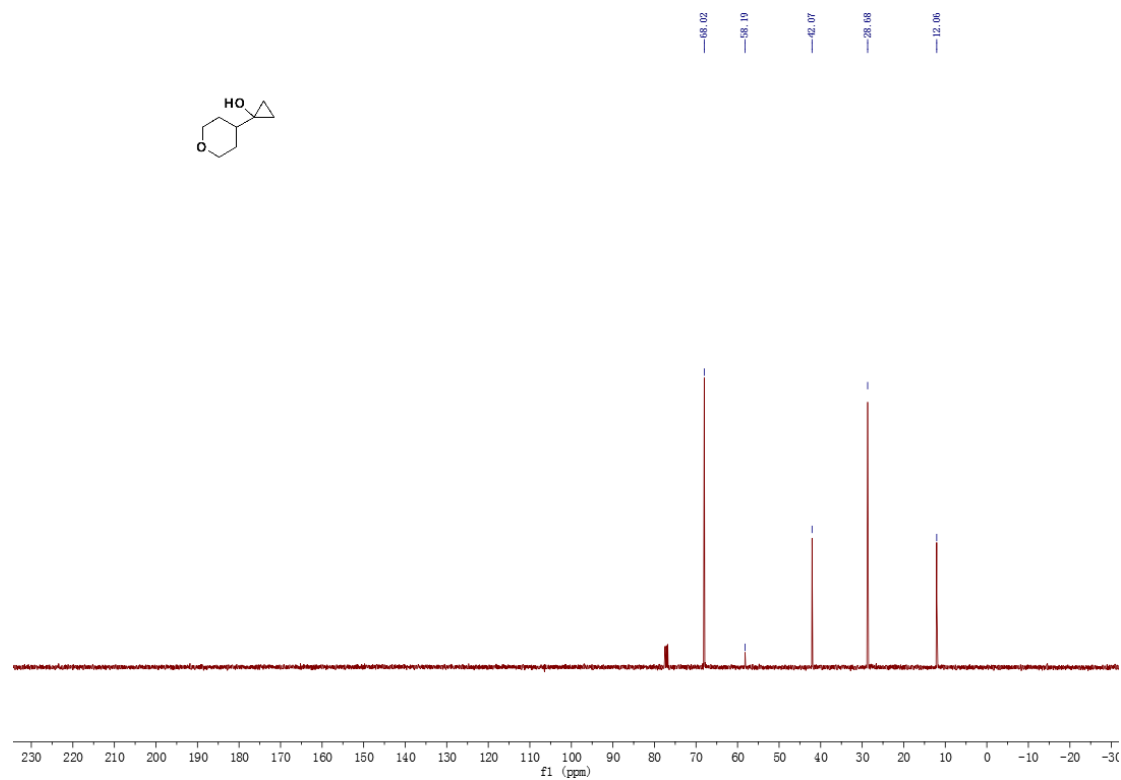
¹³C NMR spectrum of 1-phenethylcyclopropanol 3h



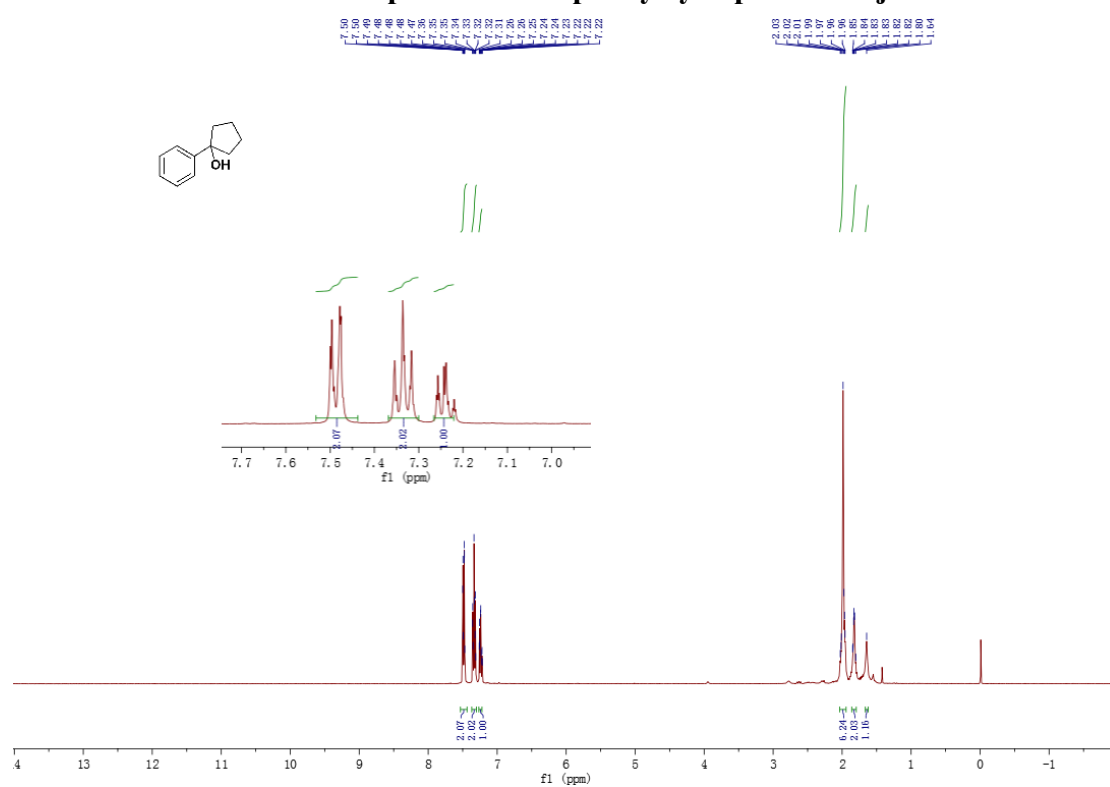
¹H NMR spectrum of 1-phenethylcyclopropanol 3h



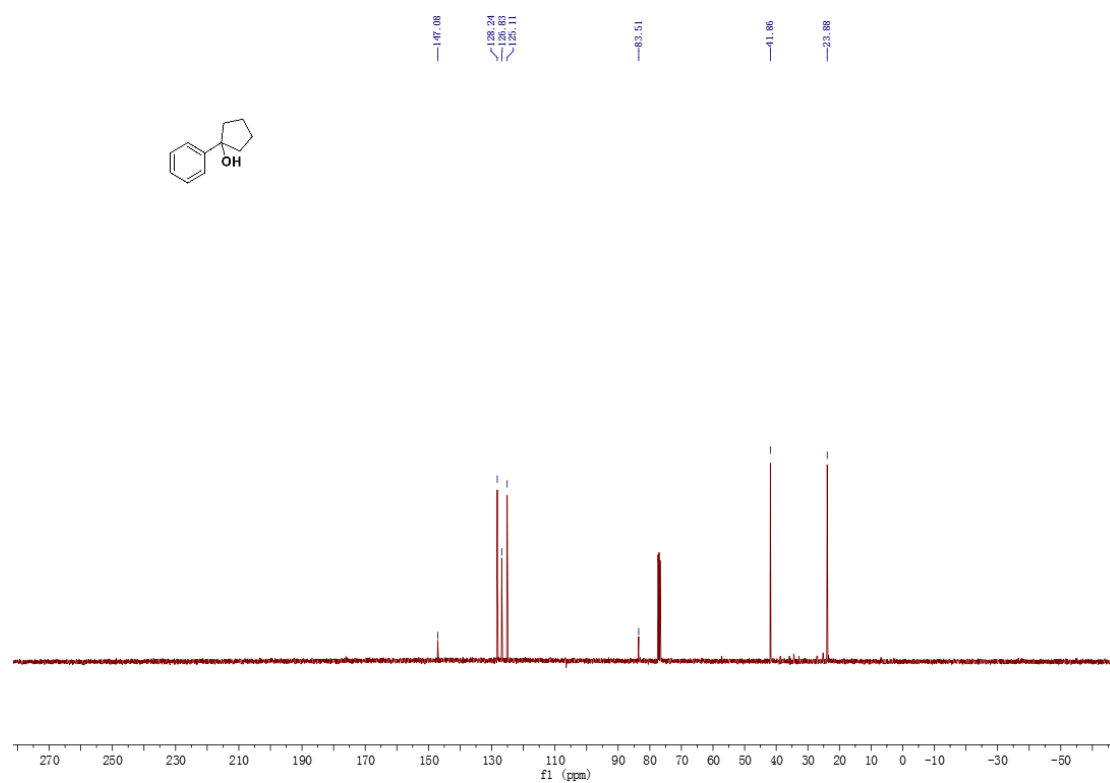
¹³C NMR spectrum of 1-(tetrahydro-2H-pyran-4-yl)cyclopropanol 3i

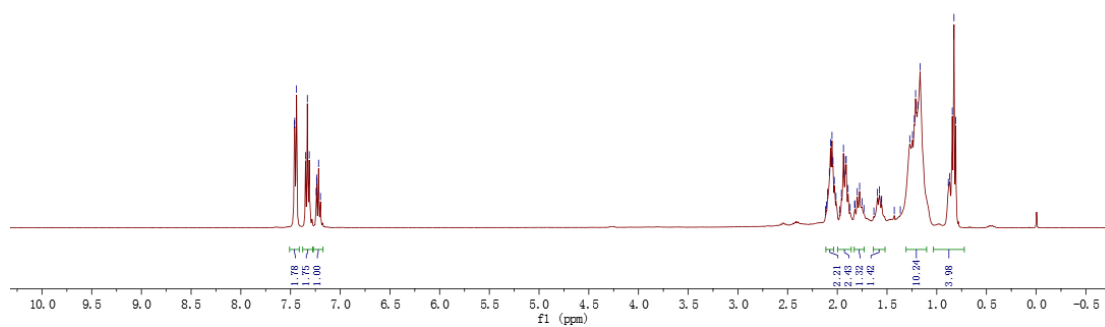


¹H NMR spectrum of 1-phenylcyclopentanol 3j

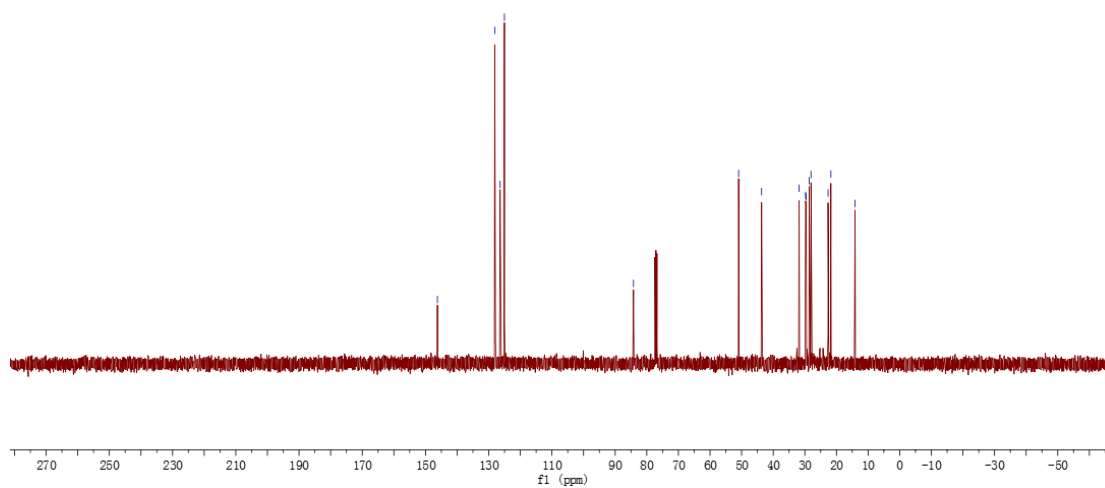


¹³C NMR spectrum of 1-phenylcyclopentanol 3j

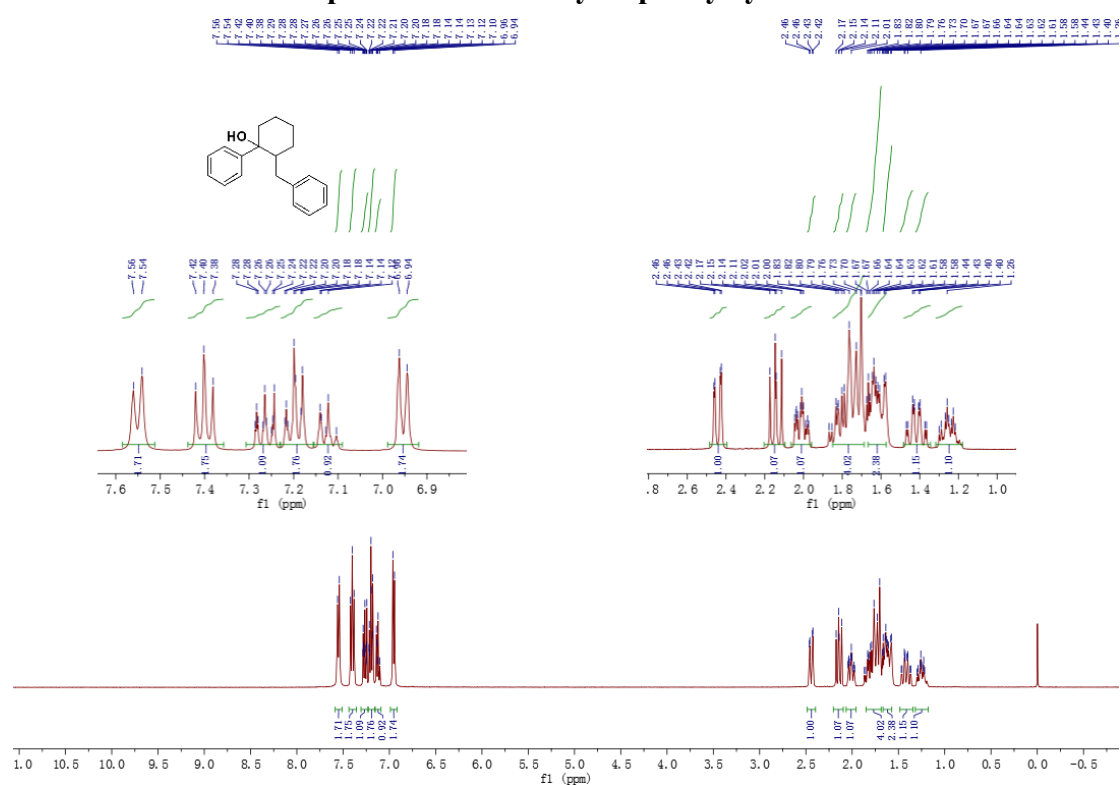




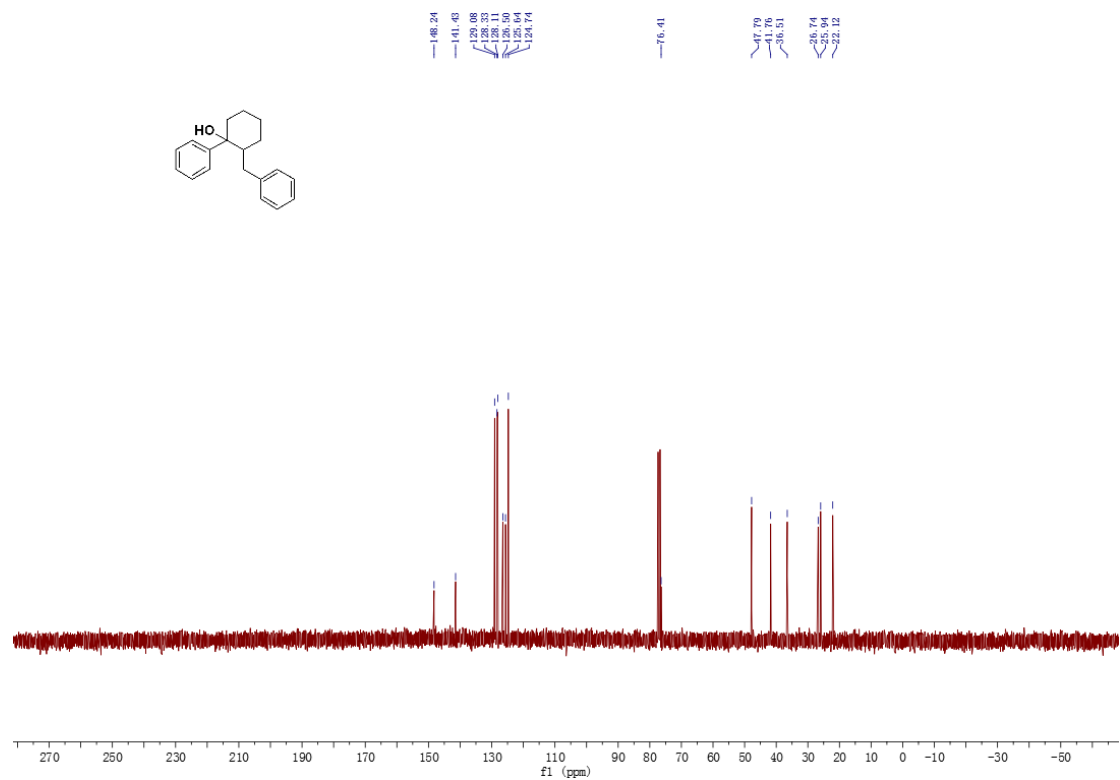
O=C1CCCC1(CO)Cc2ccccc2



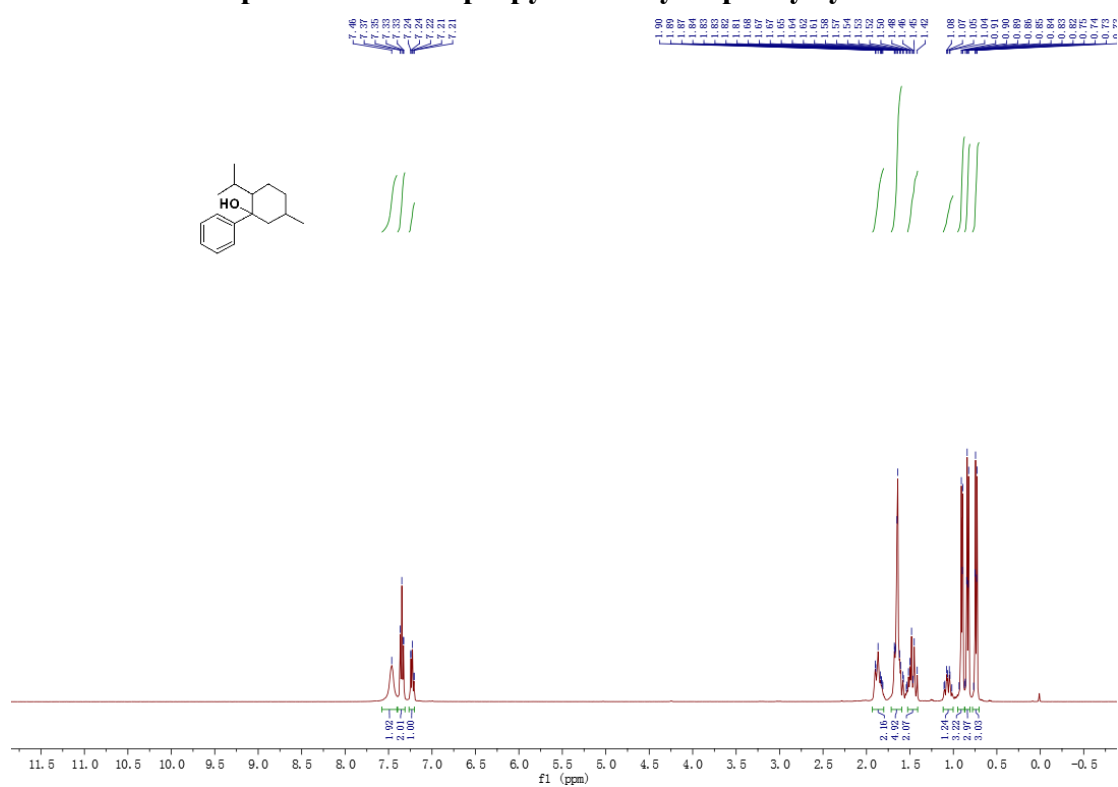
¹H NMR spectrum of 2-benzyl-1-phenylcyclohexanol 3n



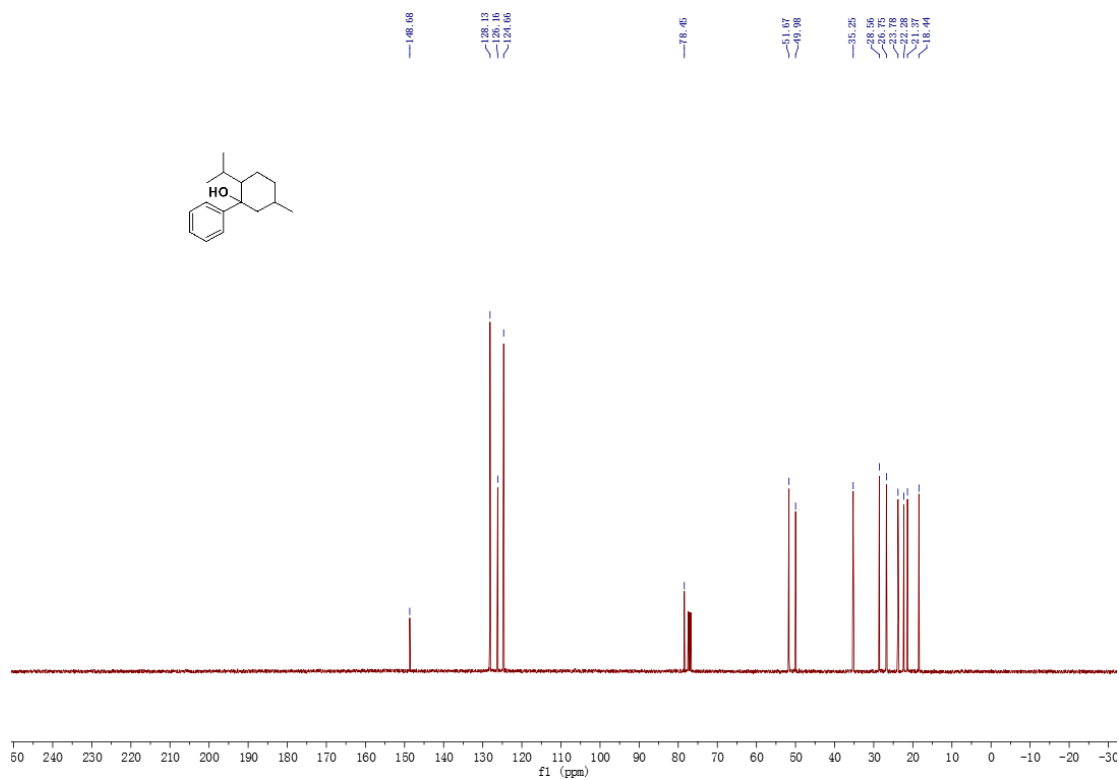
¹³C NMR spectrum of 2-benzyl-1-phenylcyclohexanol 3n



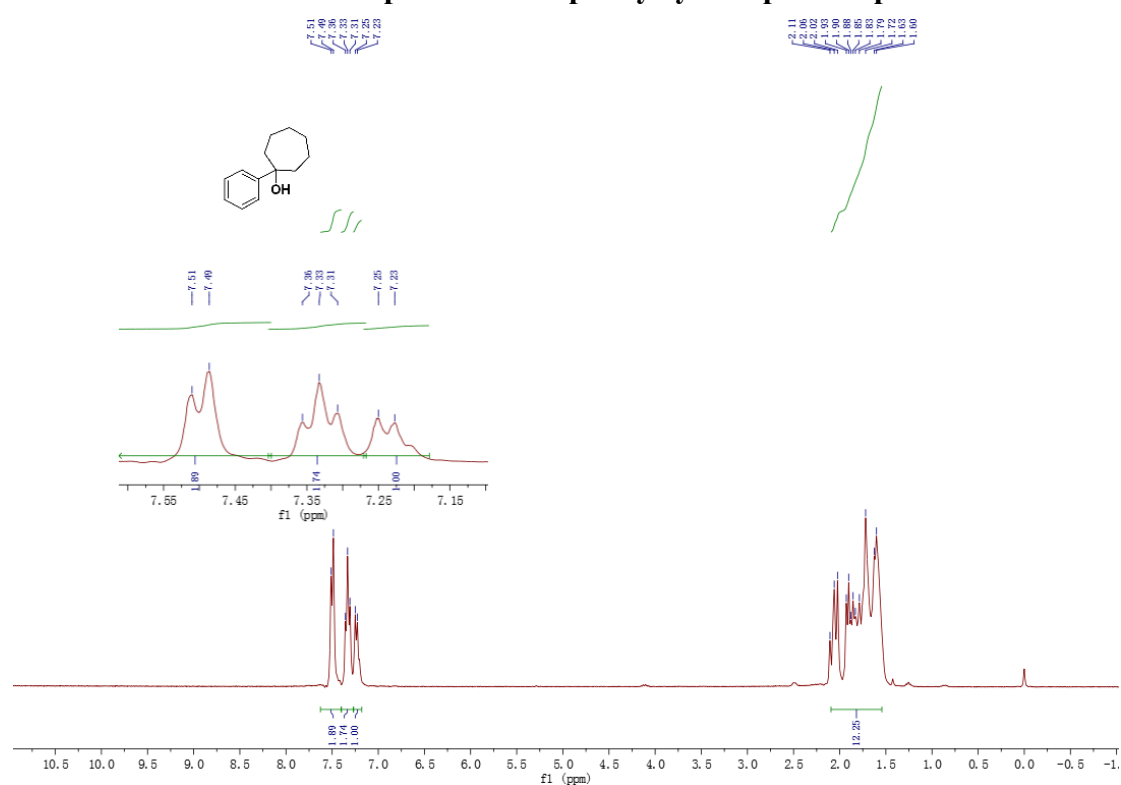
^1H NMR spectrum of 2-isopropyl-5-methyl-1-phenylcyclohexanol 3o



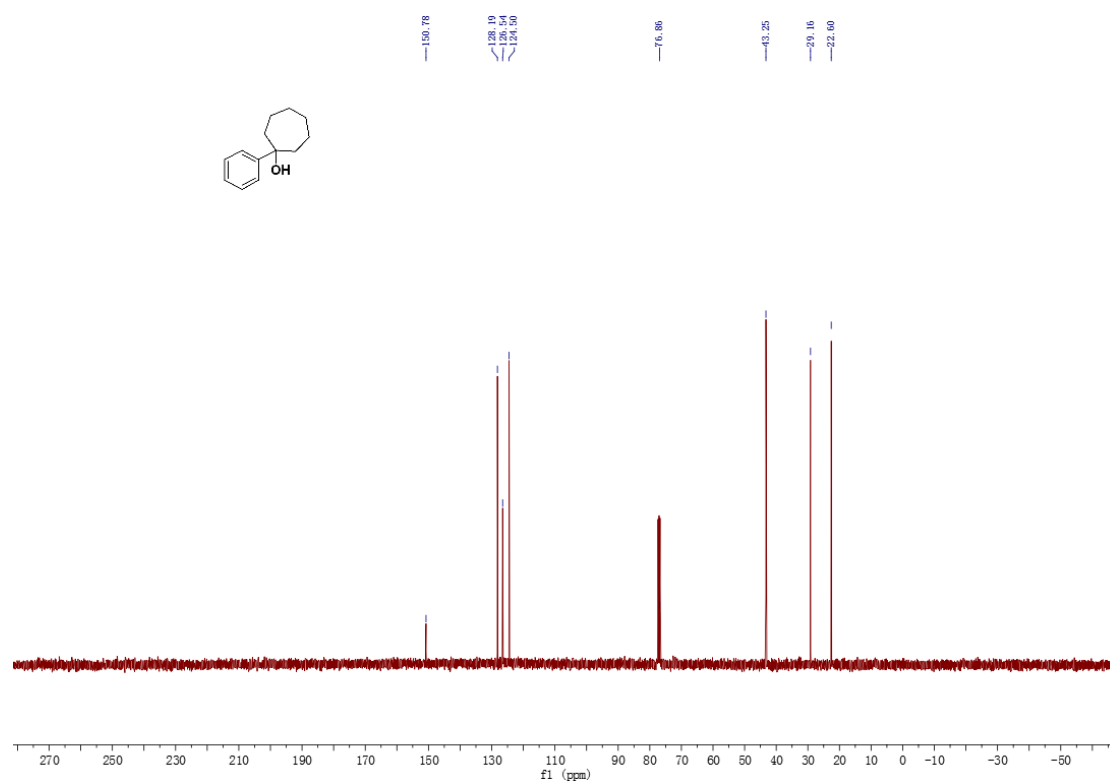
^{13}C NMR spectrum of 2-isopropyl-5-methyl-1-phenylcyclohexanol 3o



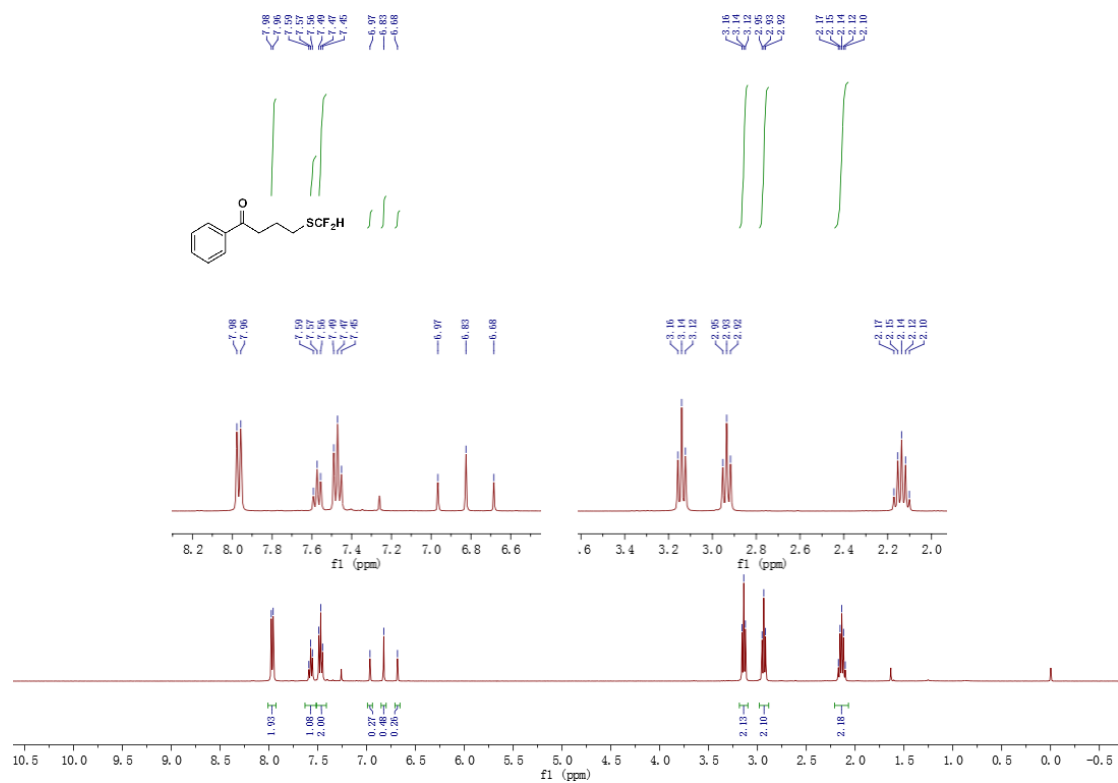
¹H NMR spectrum of 1-phenylcycloheptanol 3p



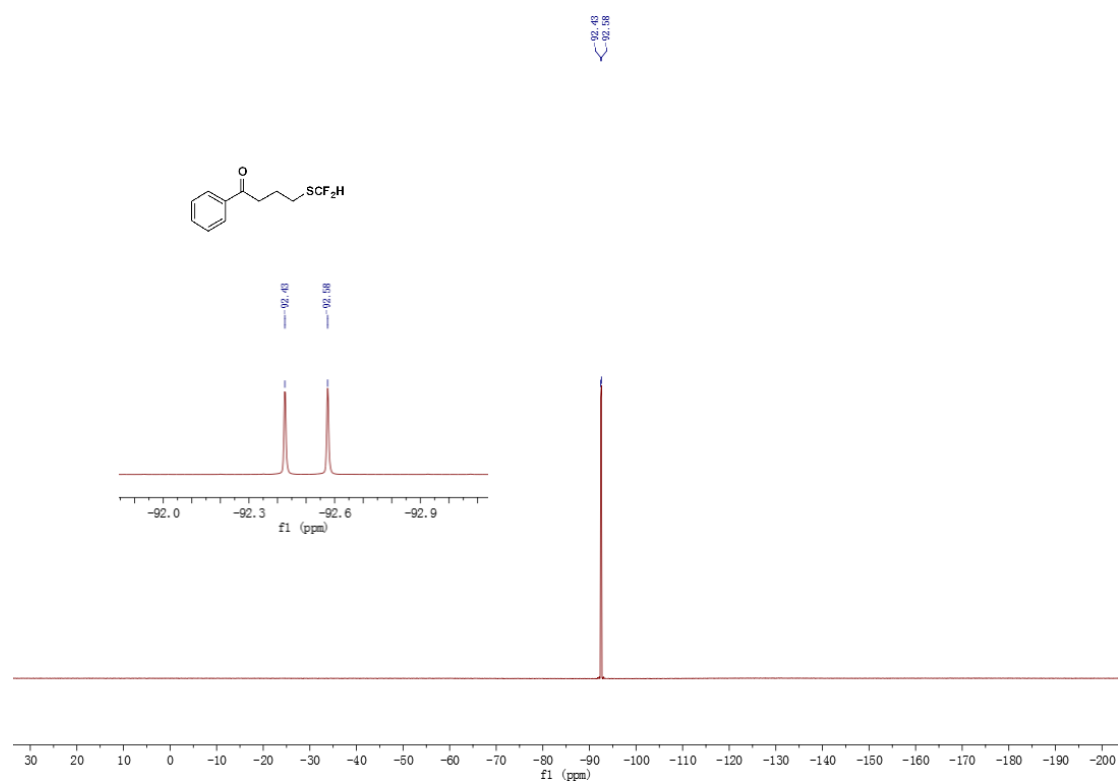
¹³C NMR spectrum of 1-phenylcycloheptanol 3p



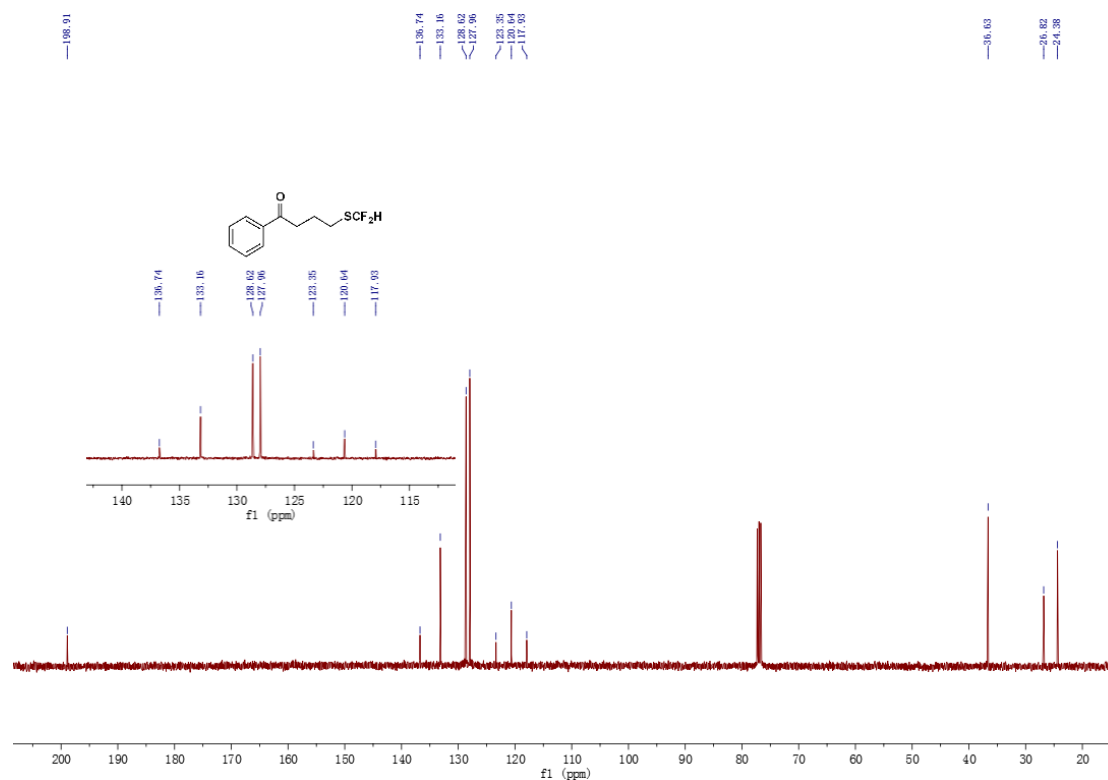
**¹H NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-1-one 2a**



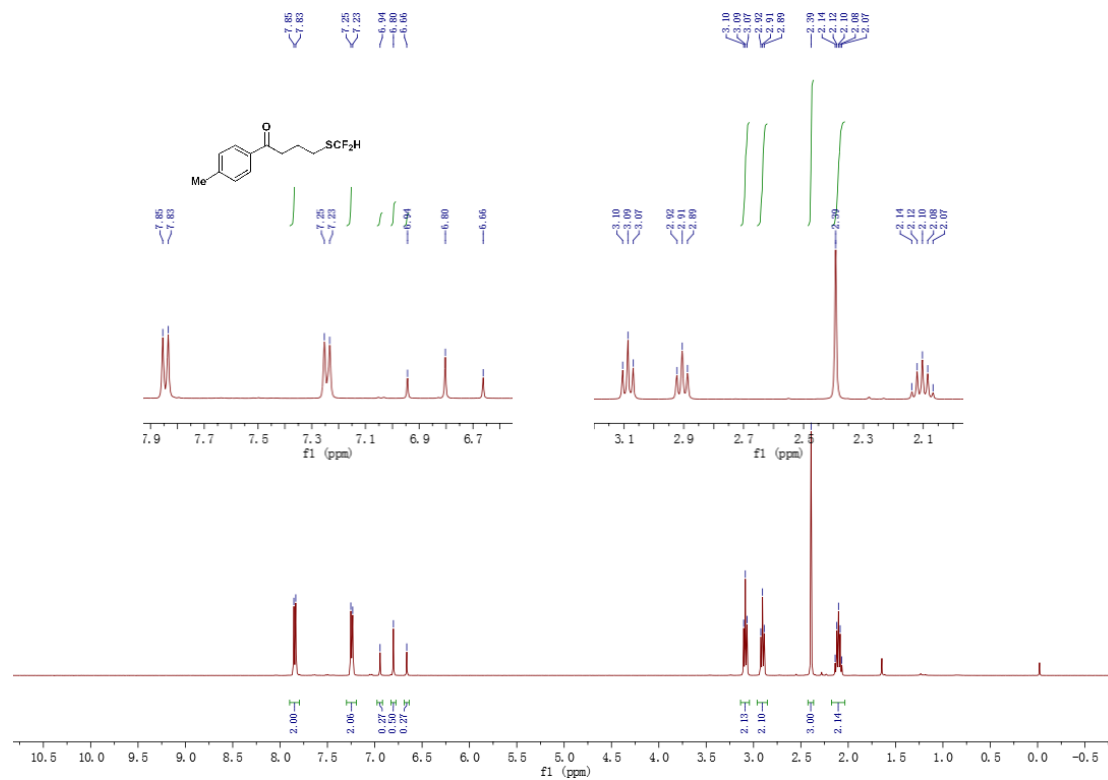
**¹⁹F NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-1-one 2a**



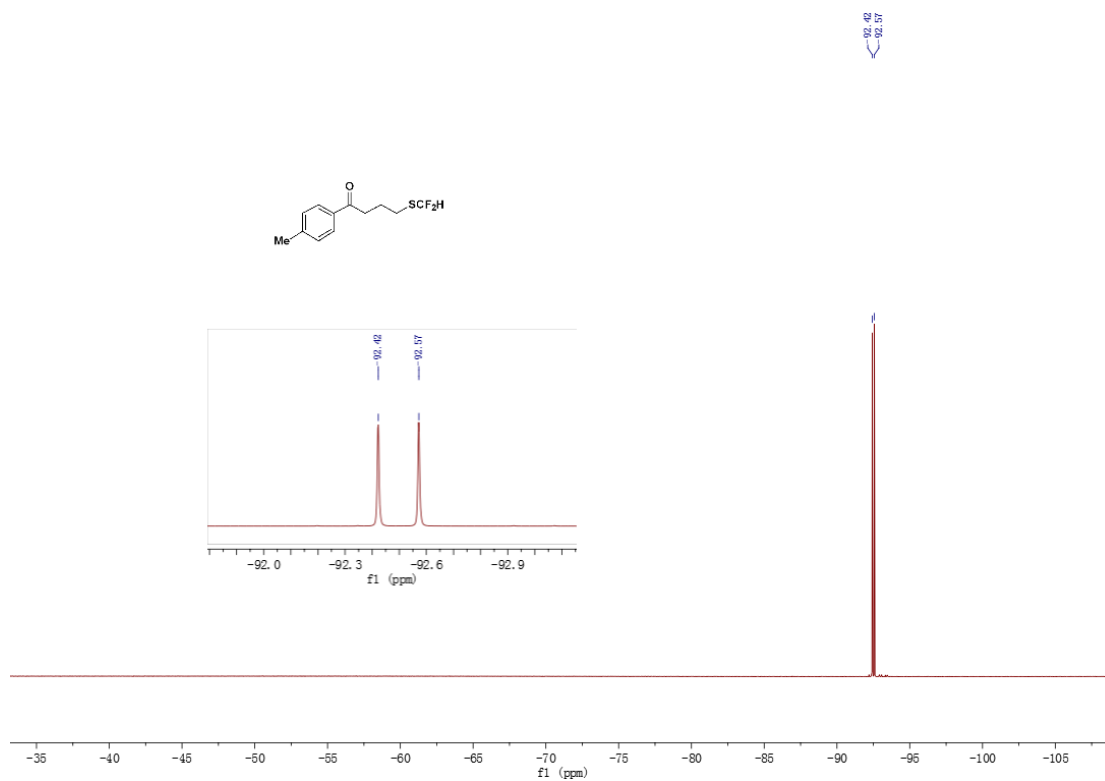
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-1-one 2a**



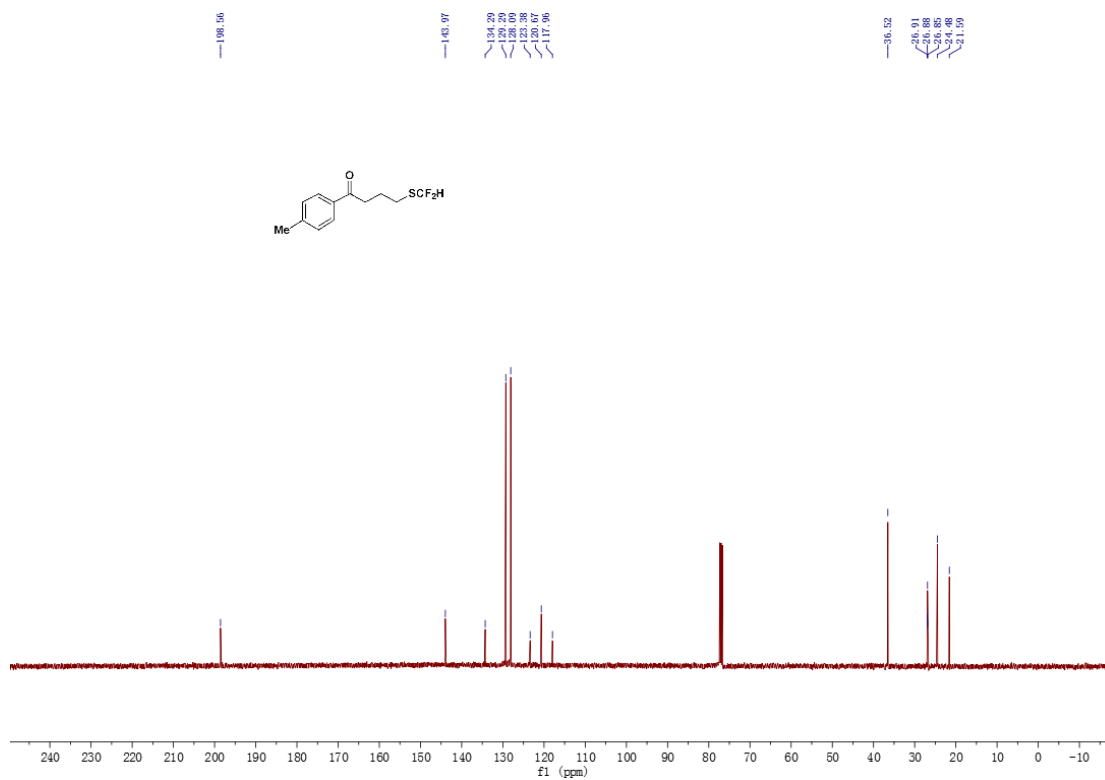
**^1H NMR spectrum of
4-((difluoromethyl)thio)-1-(*p*-tolyl)butan-1-one 2b**



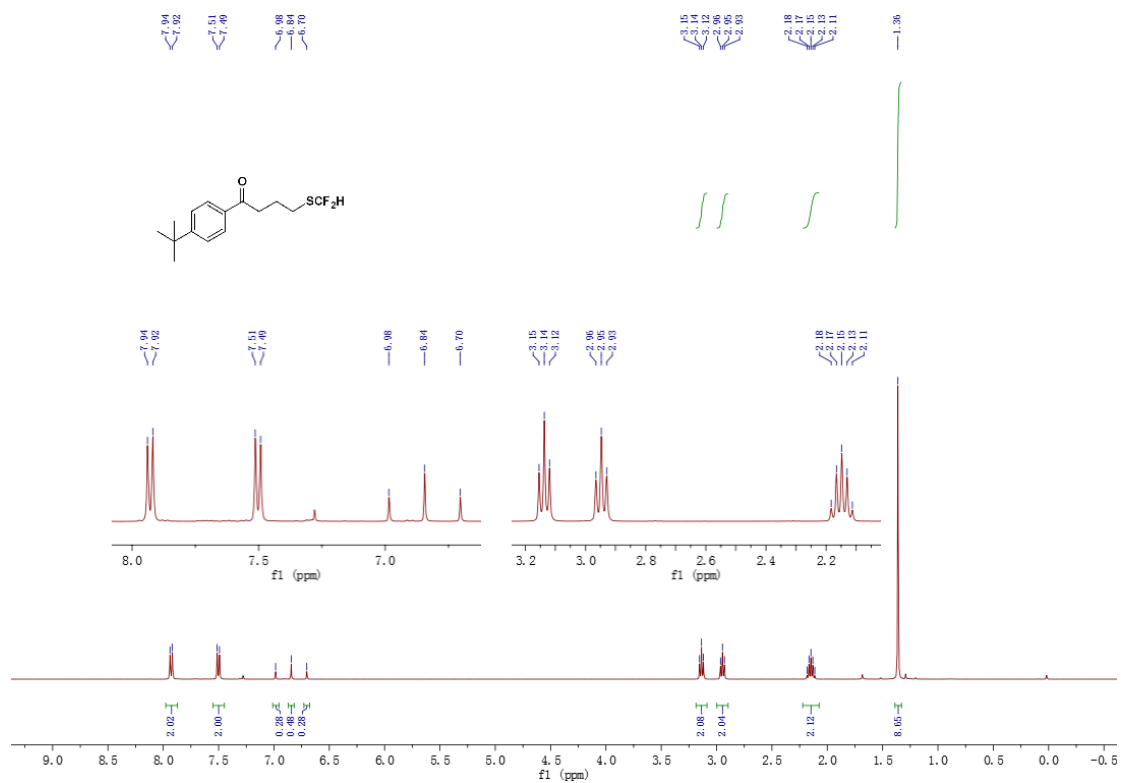
**^{19}F NMR spectrum of
4-((difluoromethyl)thio)-1-(*p*-tolyl)butan-1-one 2b**



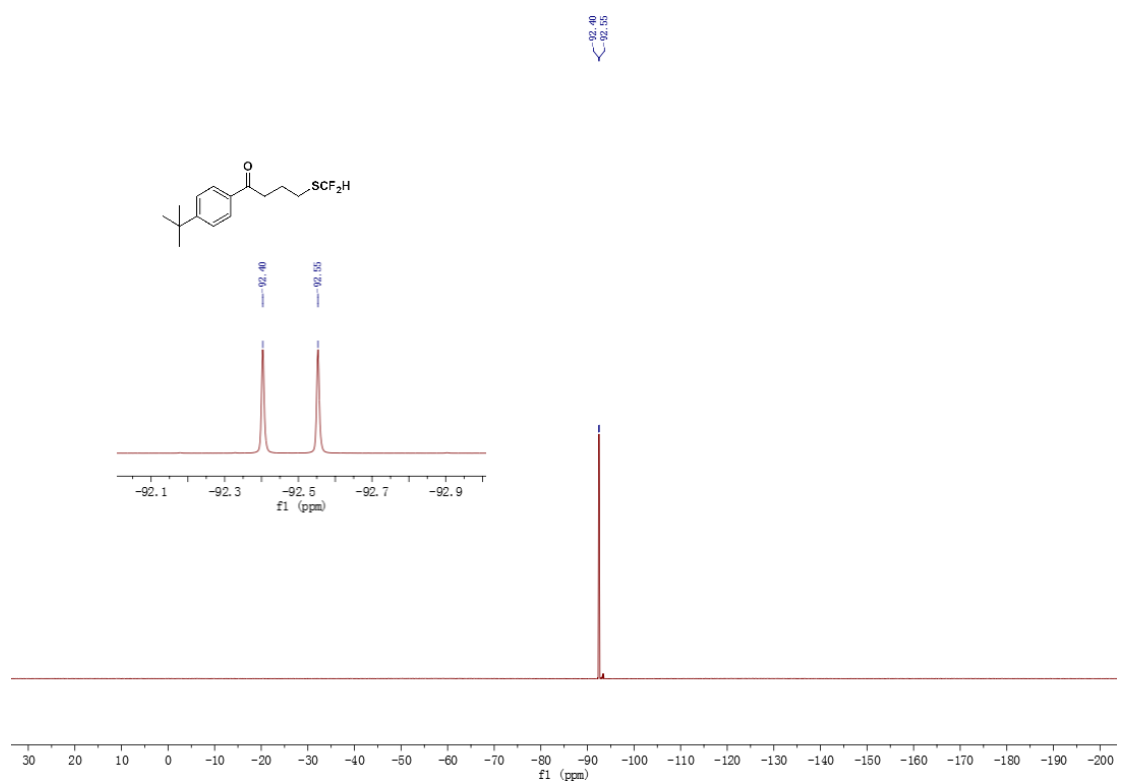
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-(*p*-tolyl)butan-1-one 2b**



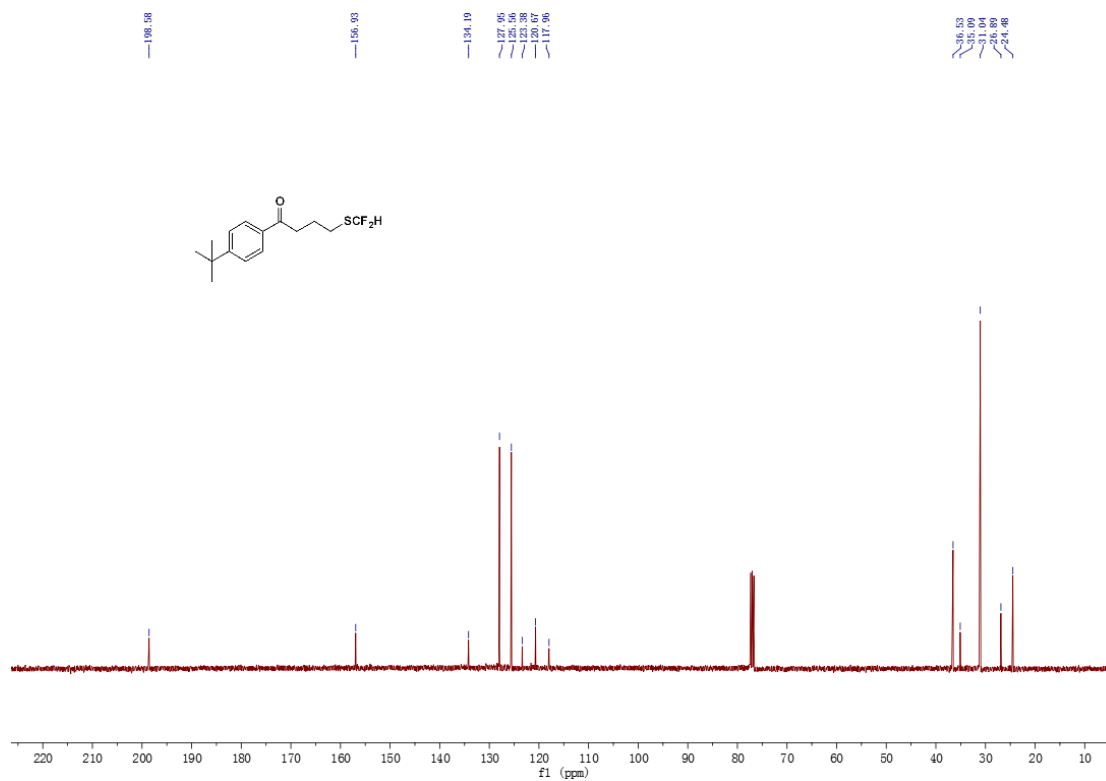
¹H NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((difluoromethyl)thio)butan-1-one 2c



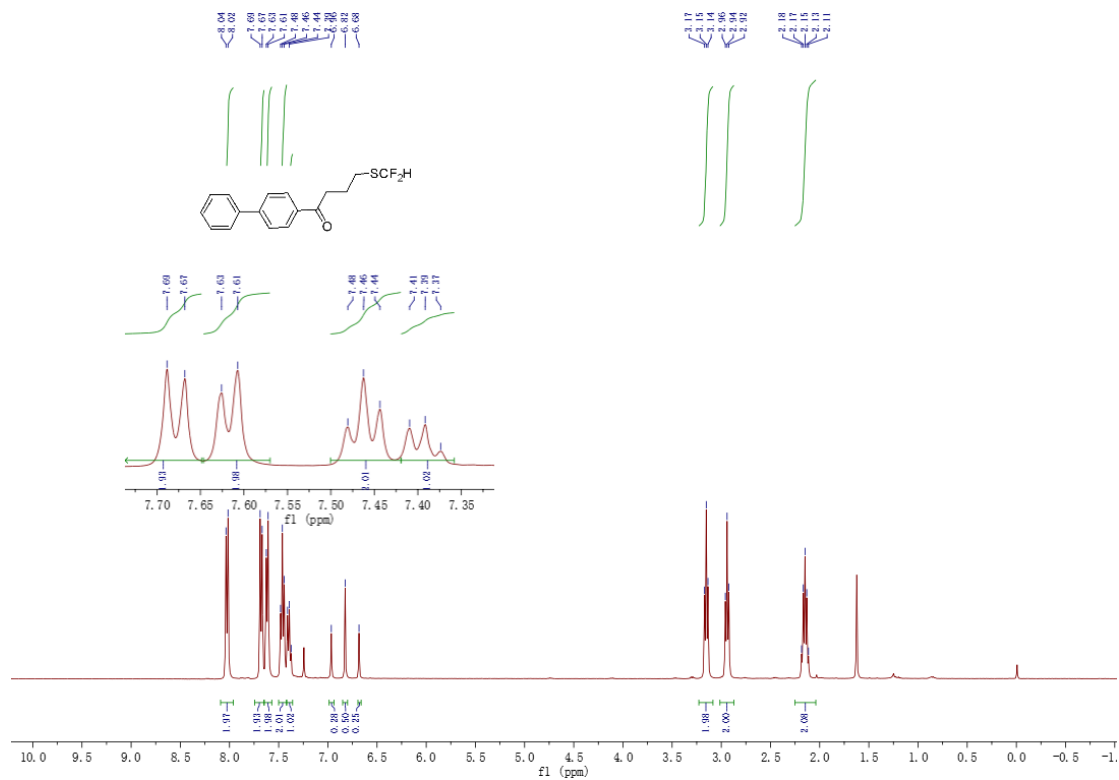
¹⁹F NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((difluoromethyl)thio)butan-1-one 2c



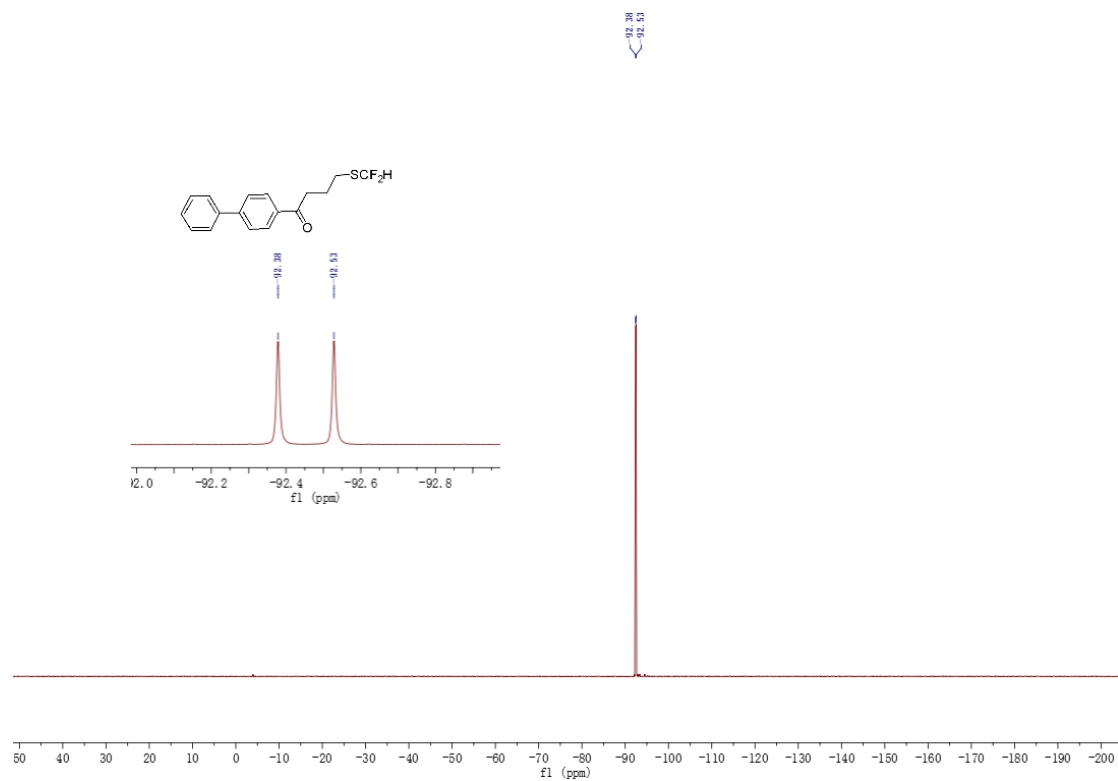
^{13}C NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((difluoromethyl)thio)butan-1-one 2c



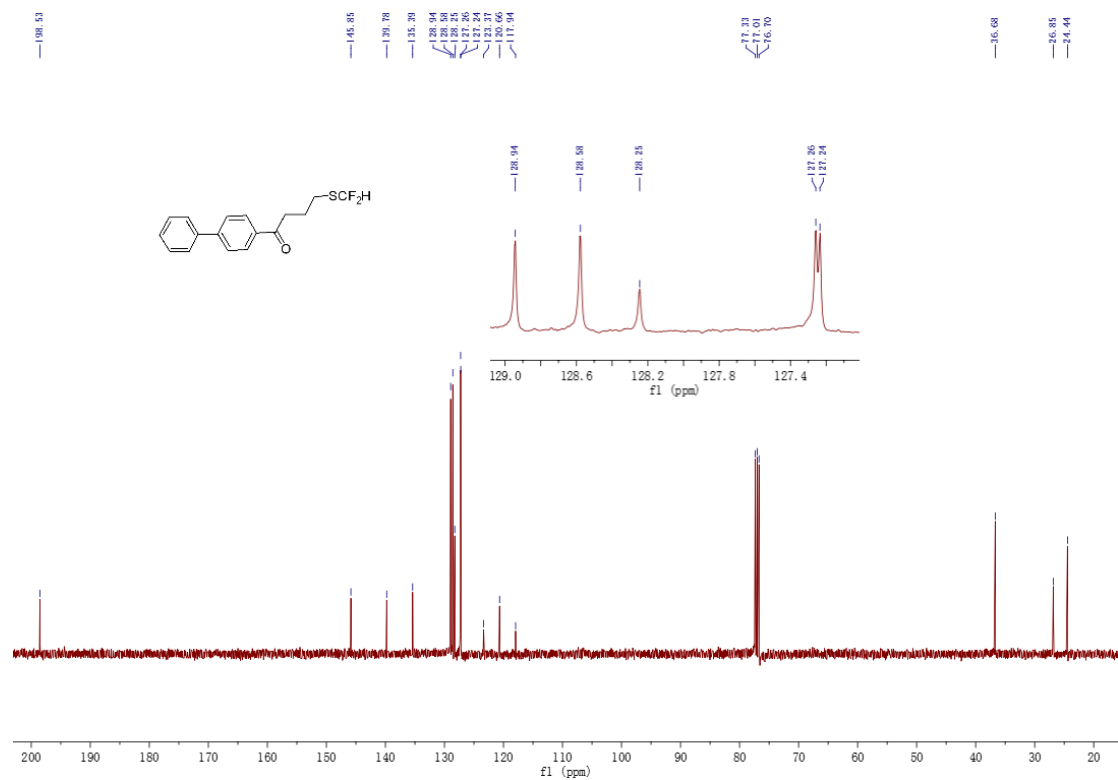
^1H NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-4-((difluoromethyl)thio)butan-1-one 2d



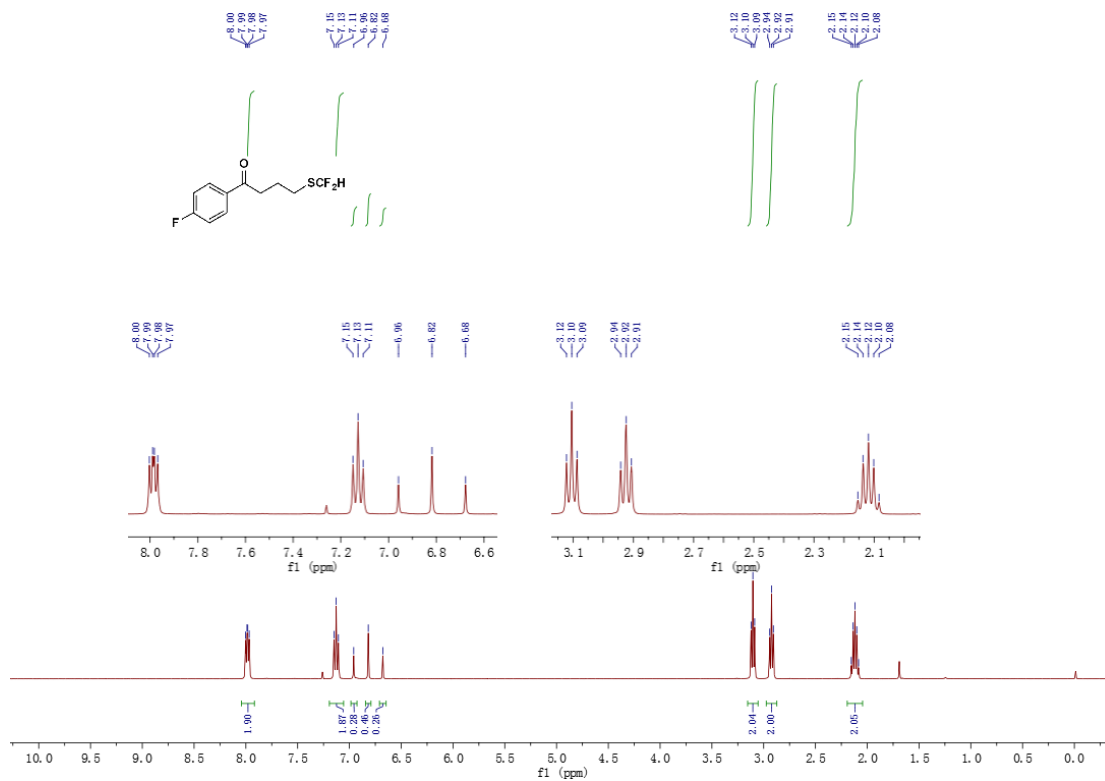
**^{19}F NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-4-((difluoromethyl)thio)butan-1-one 2d**



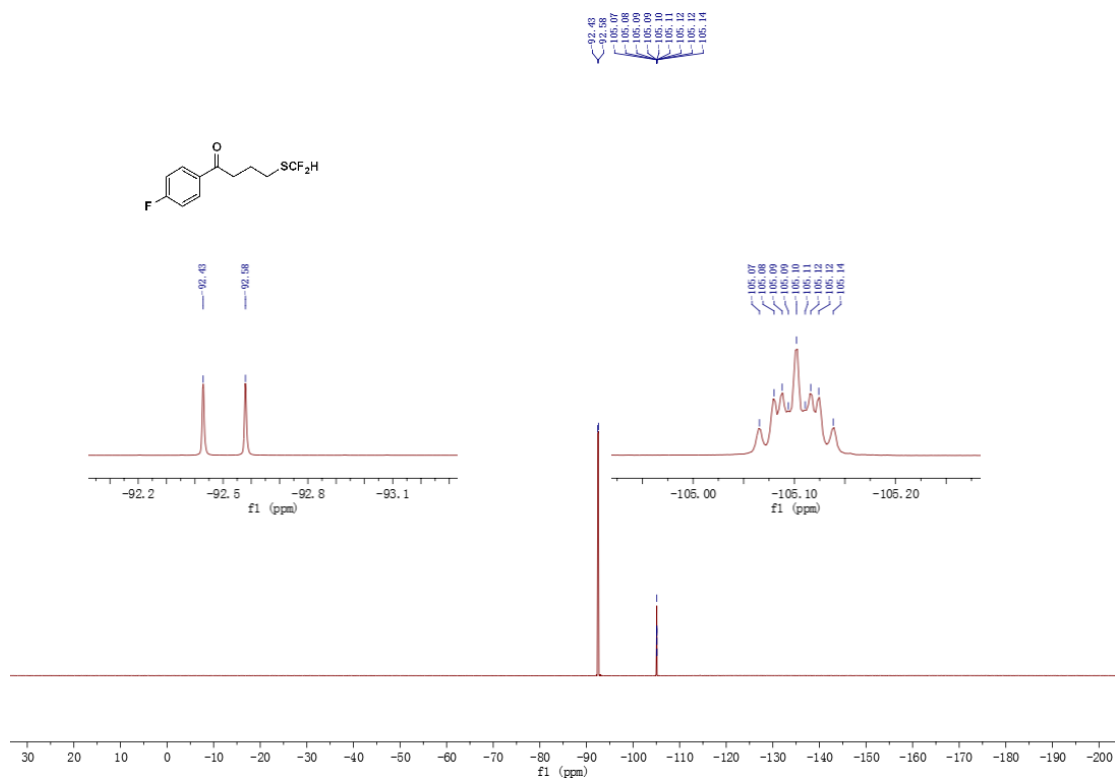
**^{13}C NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-4-((difluoromethyl)thio)butan-1-one 2d**



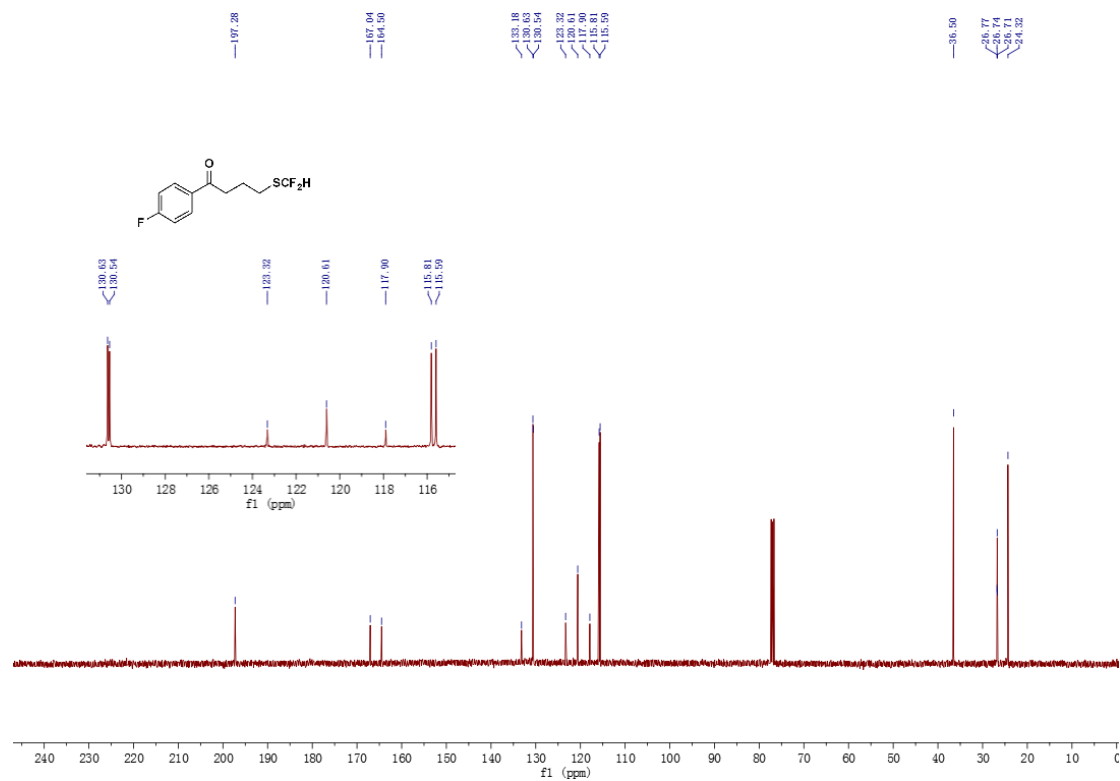
**¹H NMR spectrum of
4-((difluoromethyl)thio)-1-(4-fluorophenyl)butan-1-one 2e**



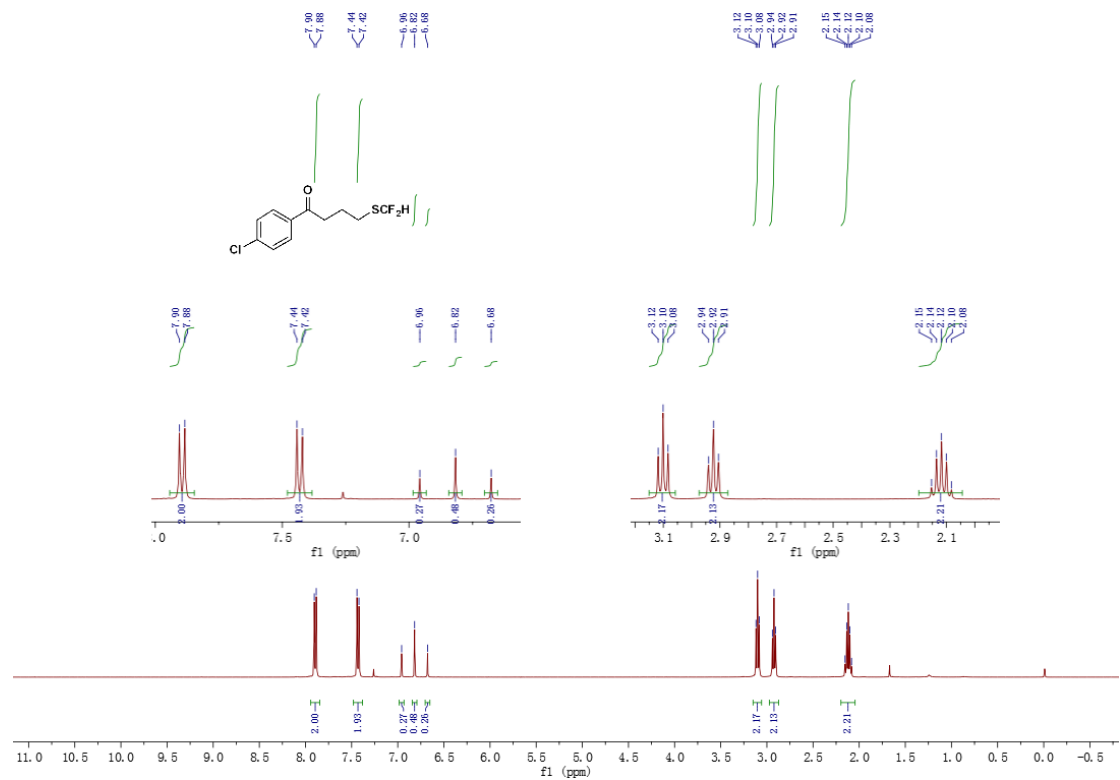
**¹⁹F NMR spectrum of
4-((difluoromethyl)thio)-1-(4-fluorophenyl)butan-1-one 2e**



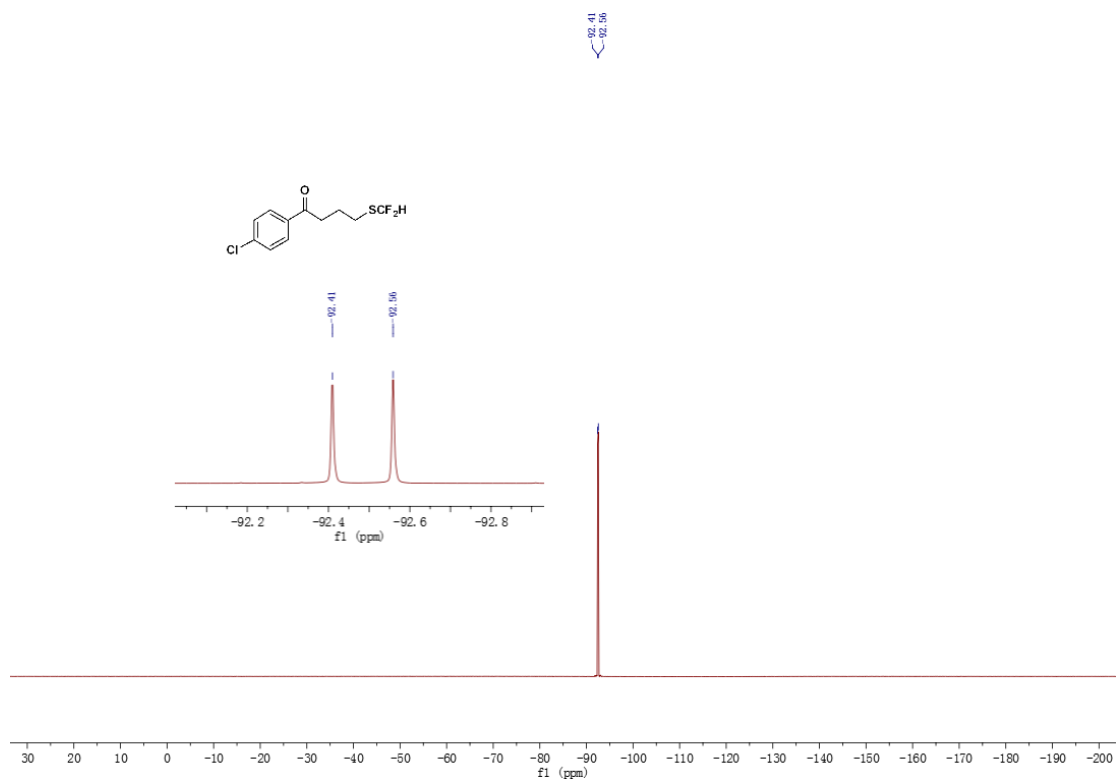
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-(4-fluorophenyl)butan-1-one 2e**



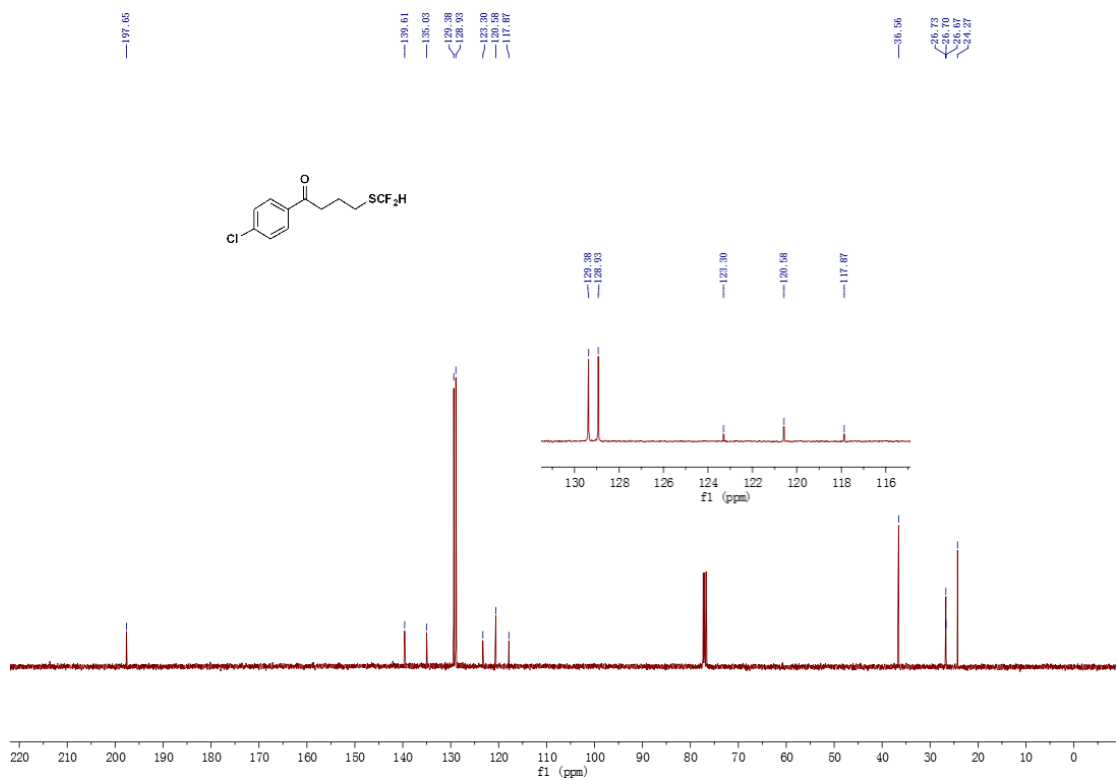
**^1H NMR spectrum of
1-(4-chlorophenyl)-4-((difluoromethyl)thio)butan-1-one 2f**



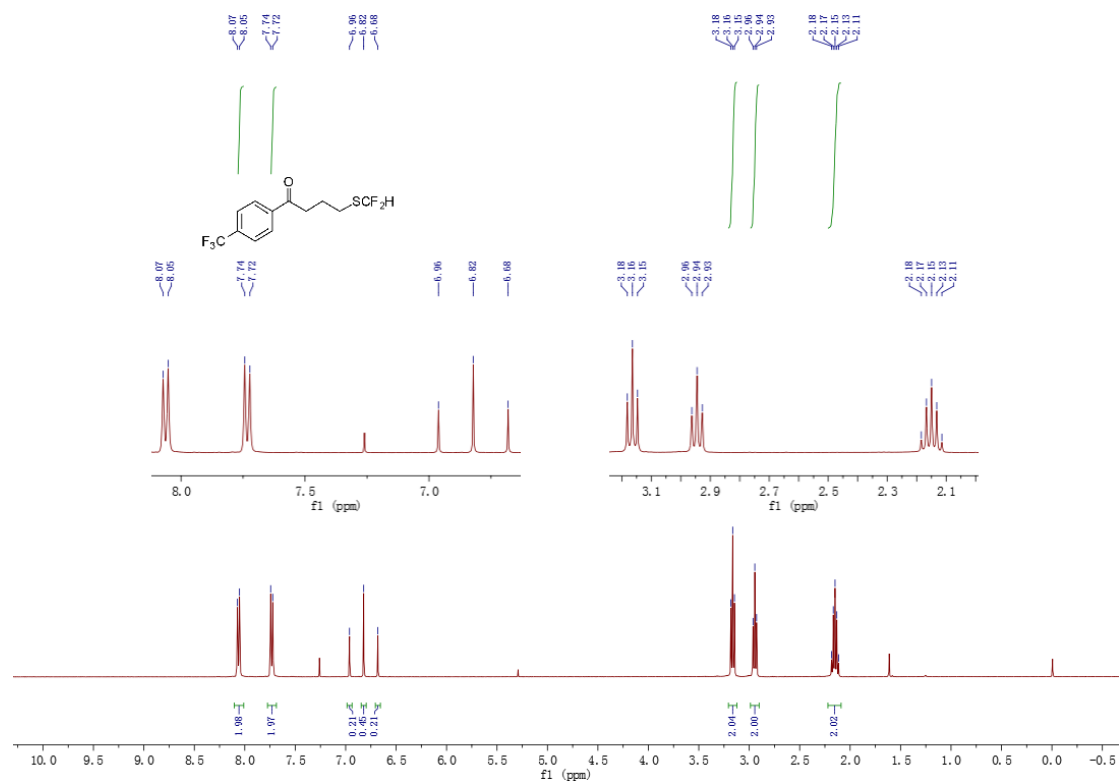
**^{19}F NMR spectrum of
1-(4-chlorophenyl)-4-((difluoromethyl)thio)butan-1-one 2f**



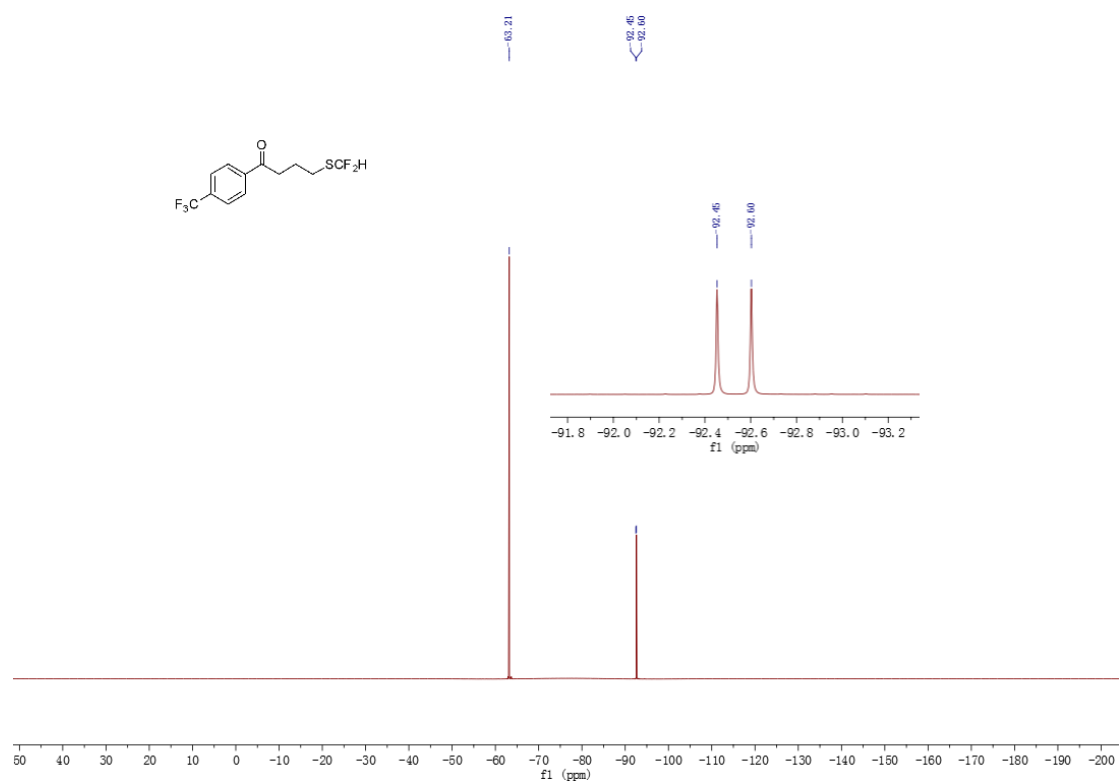
**^{13}C NMR spectrum of
1-(4-chlorophenyl)-4-((difluoromethyl)thio)butan-1-one 2f**



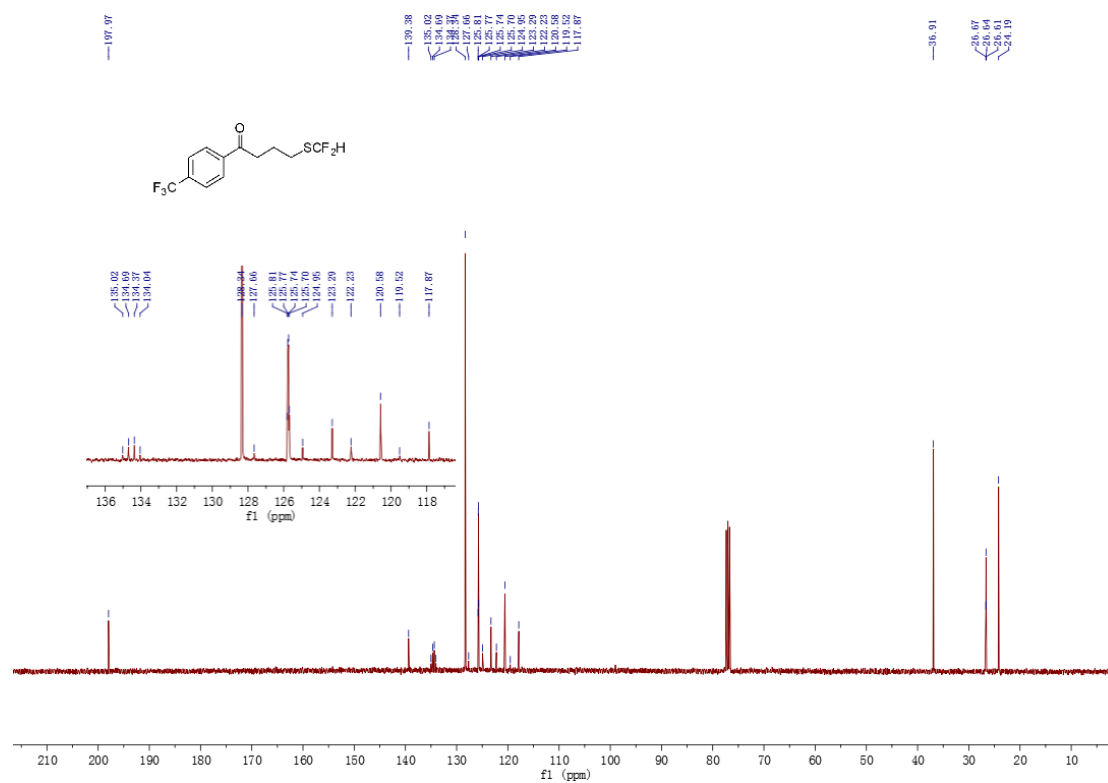
**¹H NMR spectrum of
4-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)butan-1-one 2g**



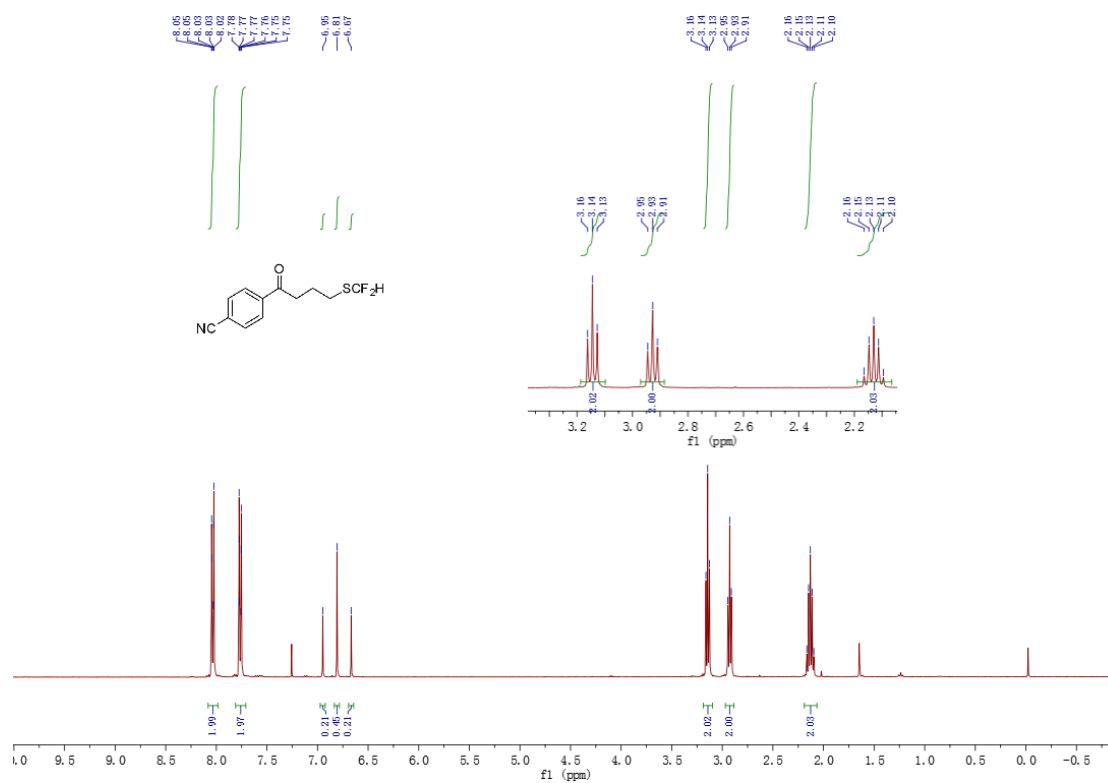
**¹⁹F NMR spectrum of
4-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)butan-1-one 2g**



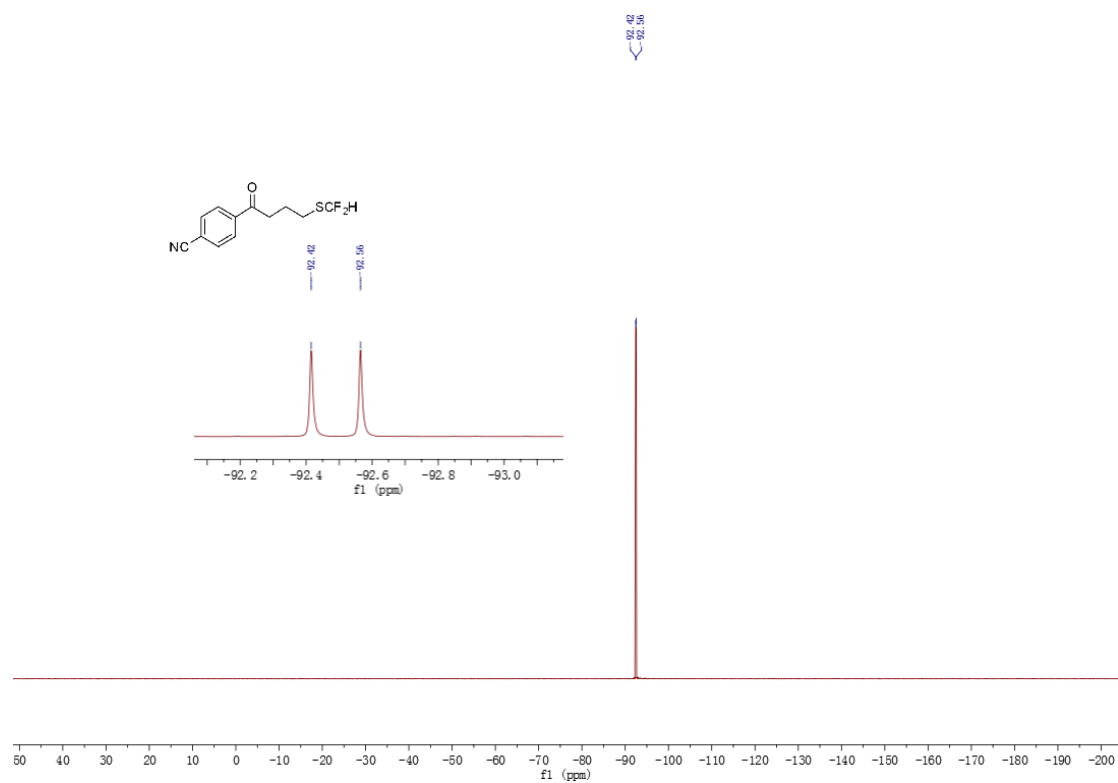
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)butan-1-one 2g**



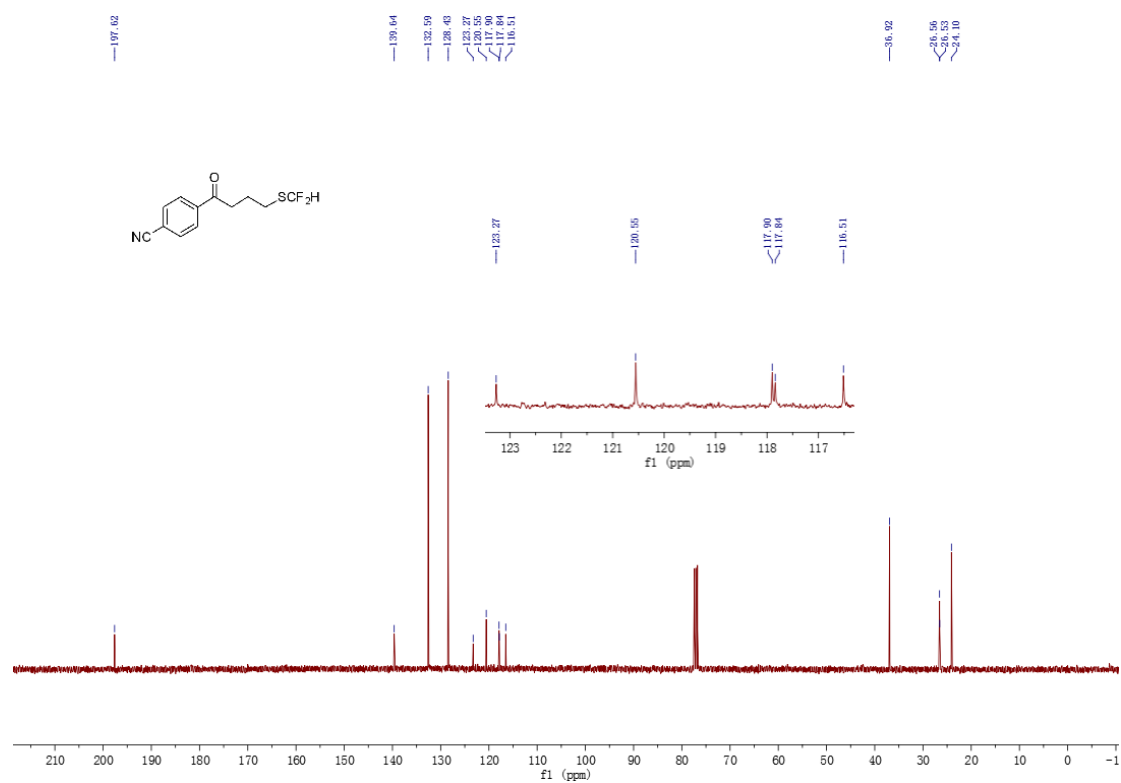
**^1H NMR spectrum of
4-(4-((difluoromethyl)thio)butanoyl)benzonitrile 2h**



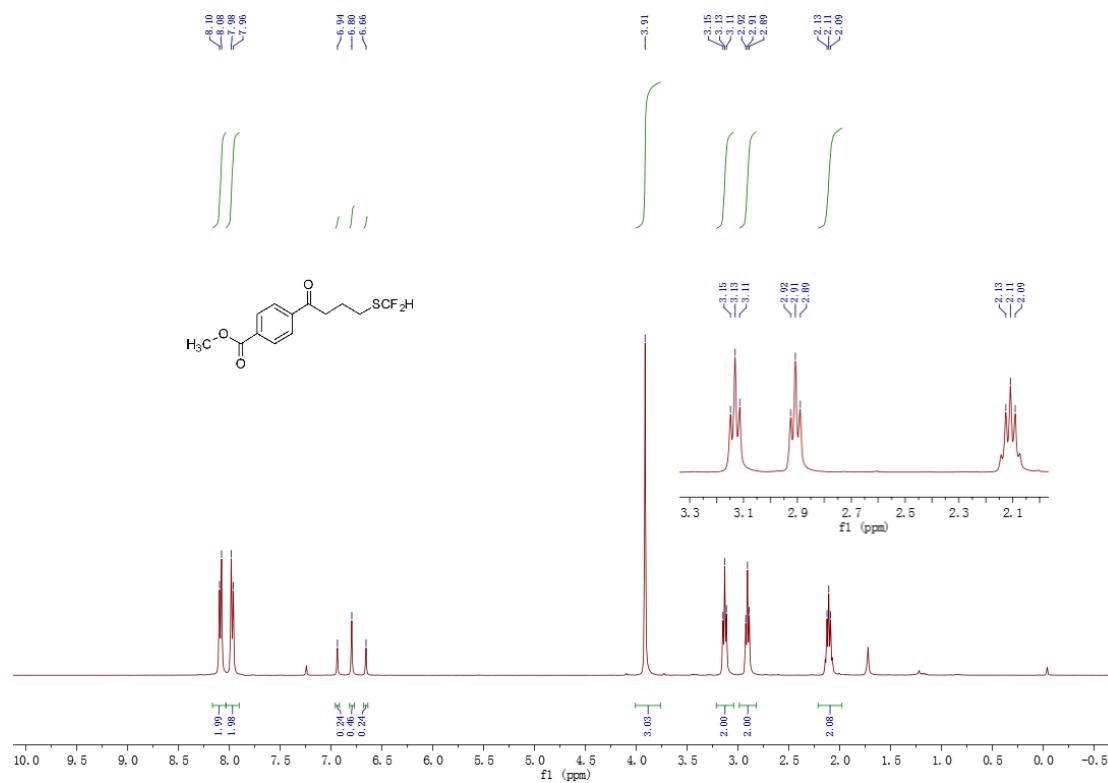
**^{19}F NMR spectrum of
4-(4-((difluoromethyl)thio)butanoyl)benzonitrile 2h**



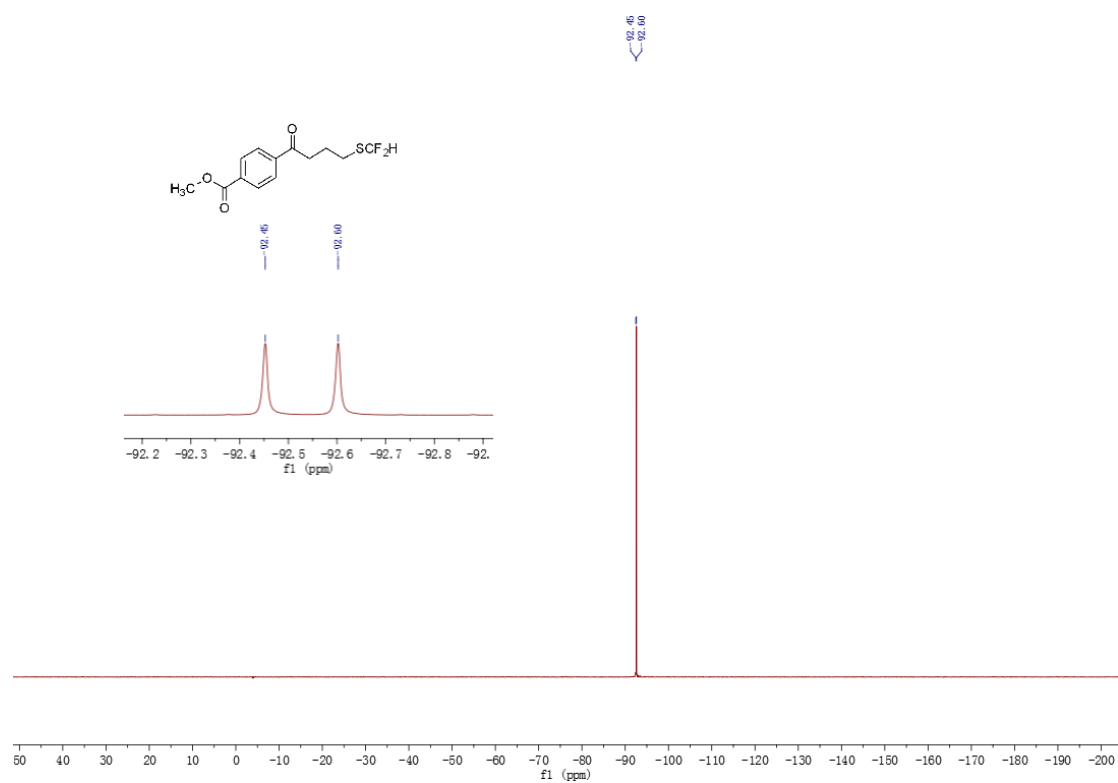
**^{13}C NMR spectrum of
4-(4-((difluoromethyl)thio)butanoyl)benzonitrile 2h**



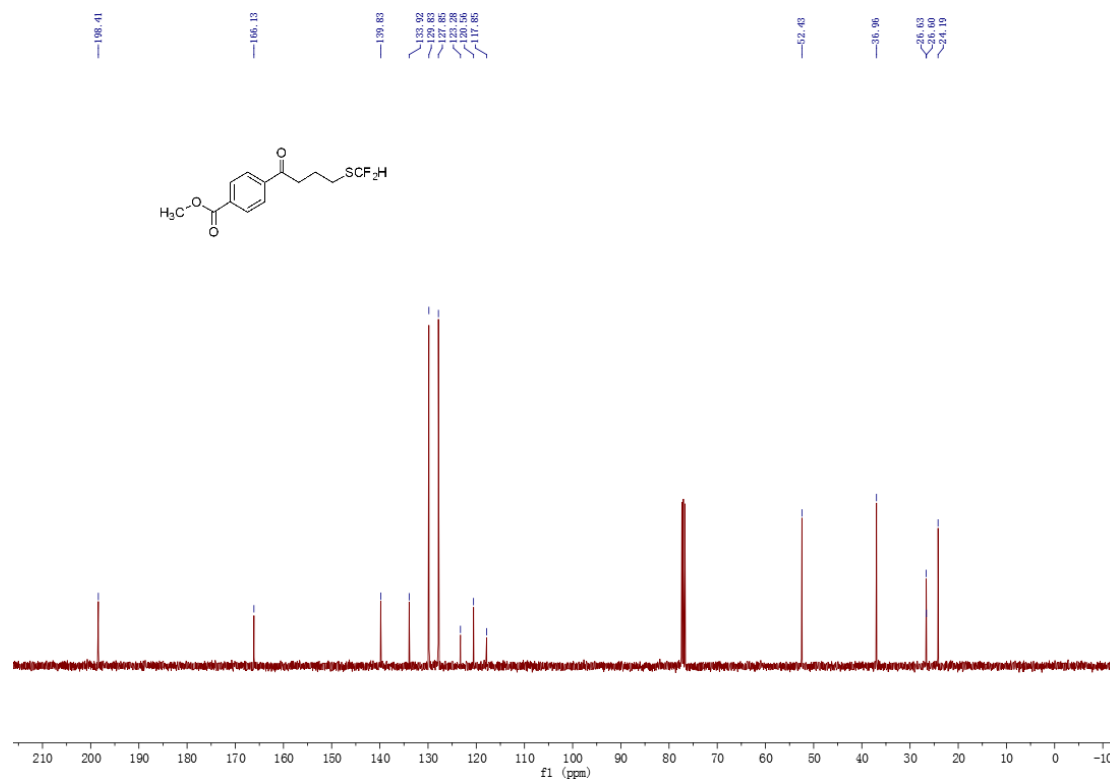
**¹H NMR spectrum of
methyl 4-(4-((difluoromethyl)thio)butanoyl)benzoate 2i**



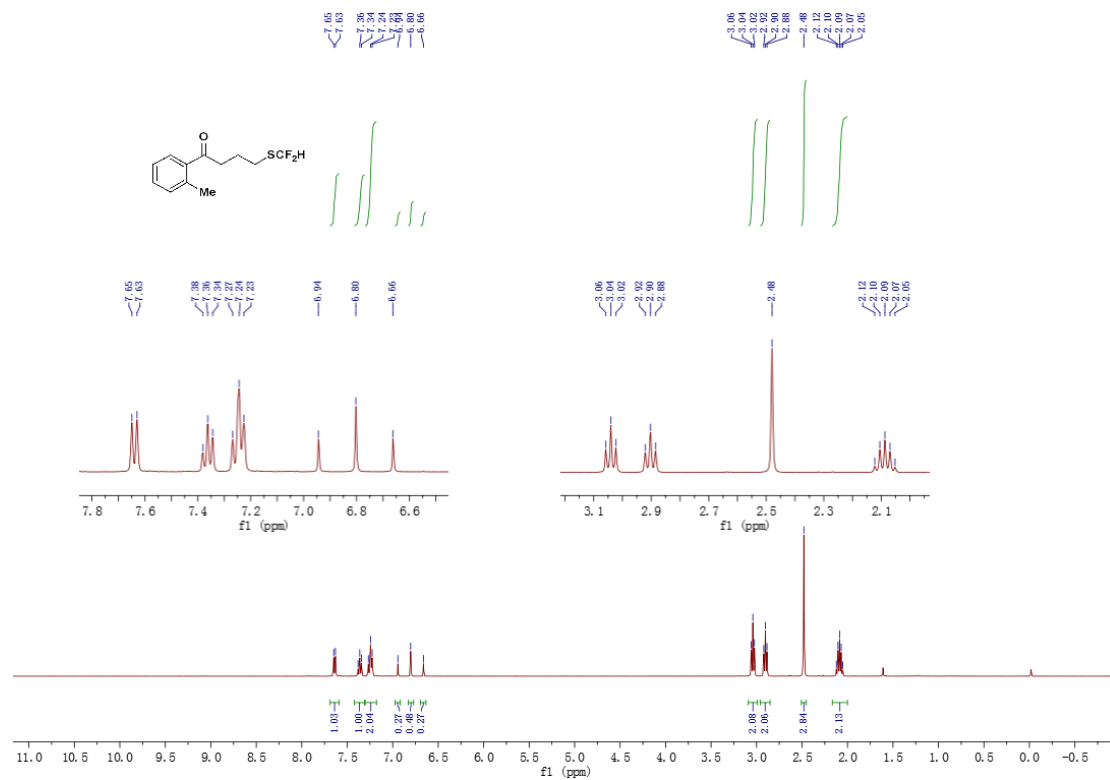
**¹⁹F NMR spectrum of
methyl 4-(4-((difluoromethyl)thio)butanoyl)benzoate 2i**



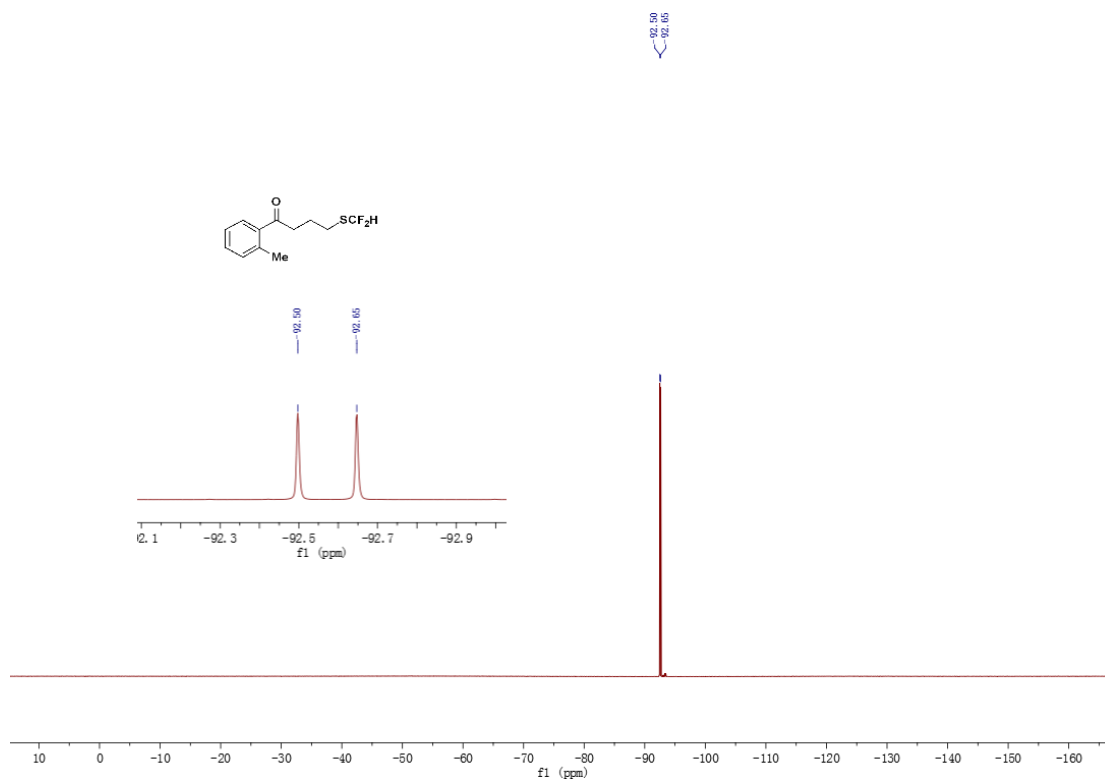
**^{13}C NMR spectrum of
methyl 4-(4-((difluoromethyl)thio)butanoyl)benzoate 2i**



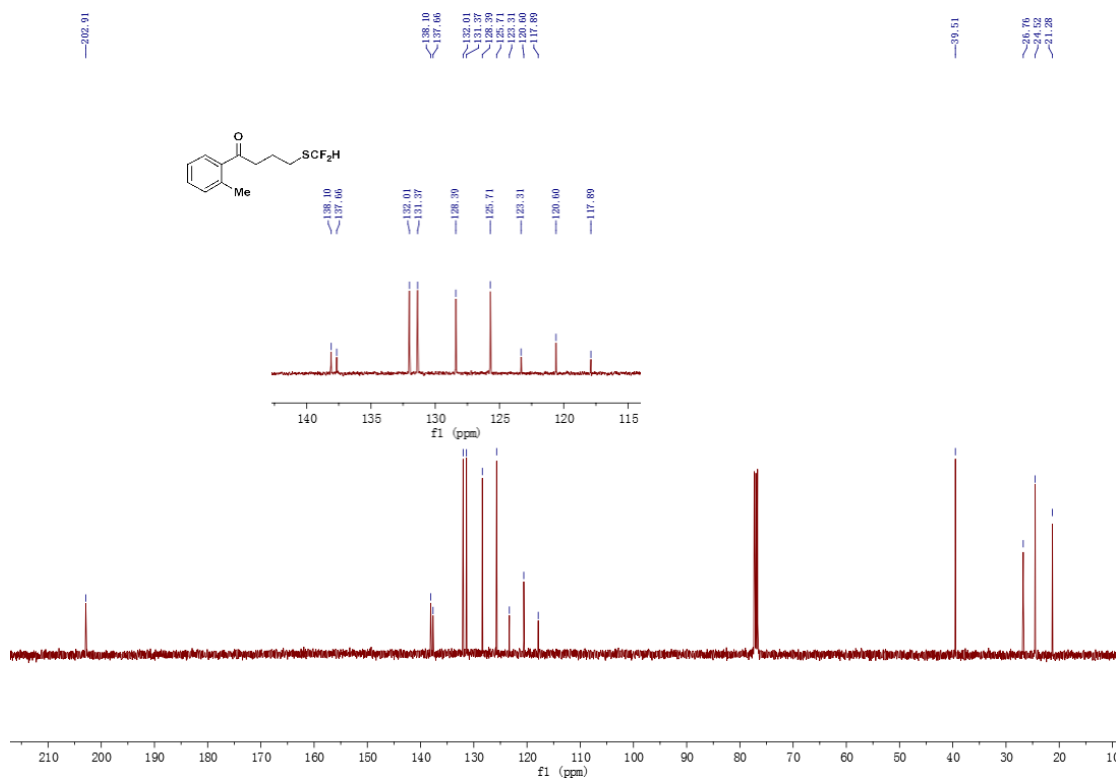
**^1H NMR spectrum of
4-((difluoromethyl)thio)-1-(*o*-tolyl)butan-1-one 2j**



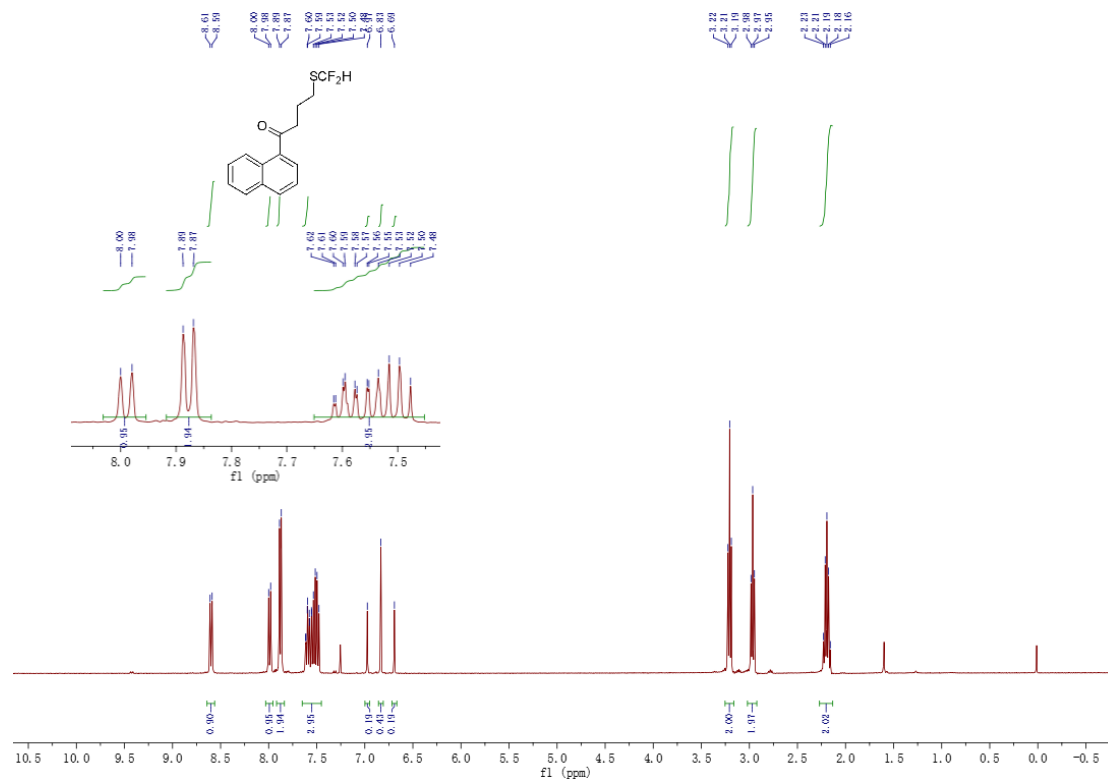
**^{19}F NMR spectrum of
4-((difluoromethyl)thio)-1-(*o*-tolyl)butan-1-one 2j**



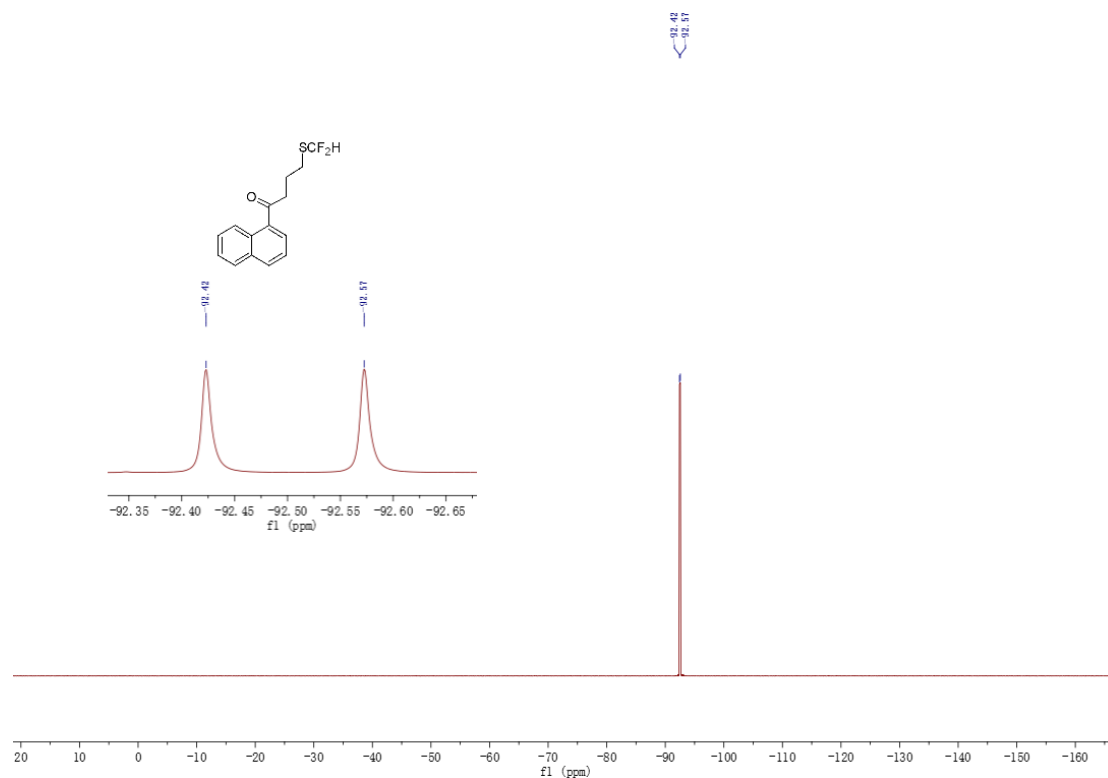
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-(*o*-tolyl)butan-1-one 2j**



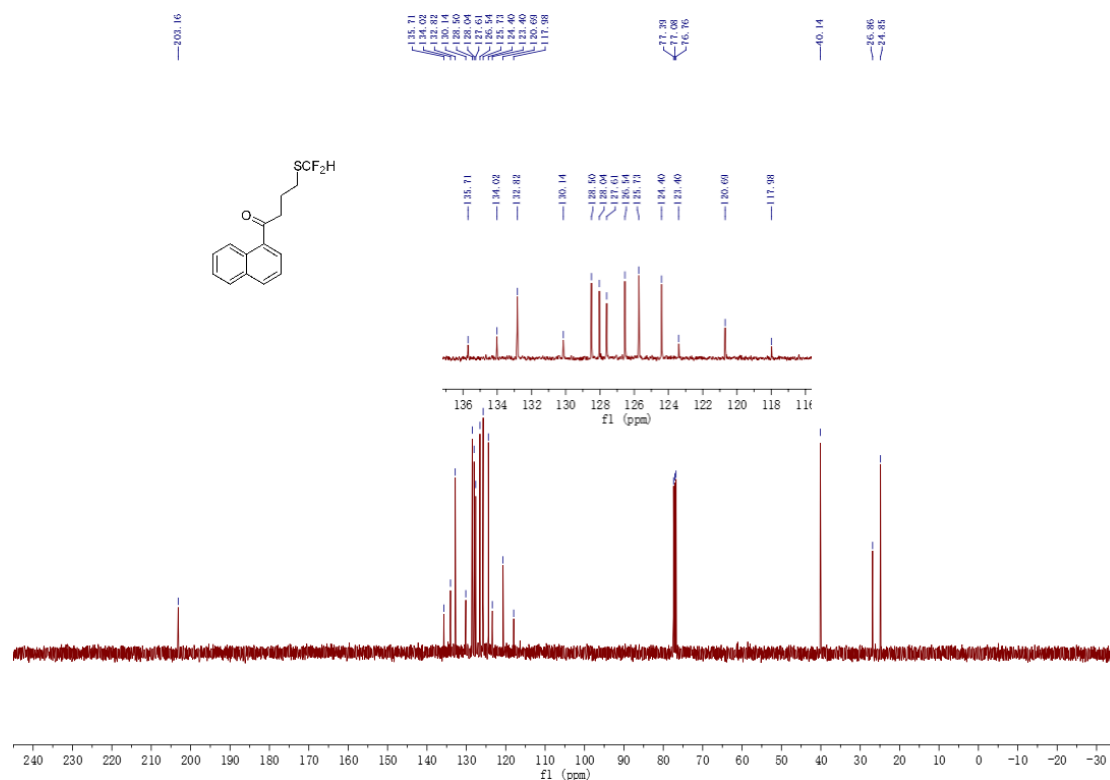
**¹H NMR spectrum of
4-((difluoromethyl)thio)-1-(naphthalen-1-yl)butan-1-one 2k**



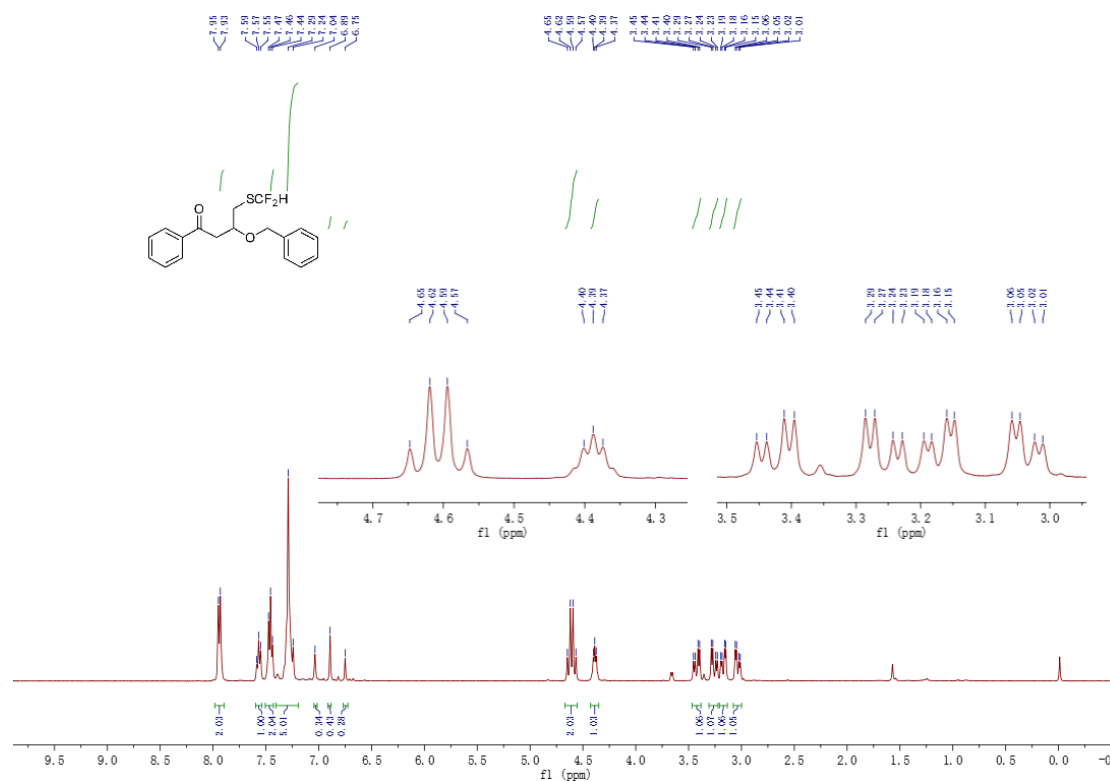
**¹⁹F NMR spectrum of
4-((difluoromethyl)thio)-1-(naphthalen-1-yl)butan-1-one 2k**



**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-(naphthalen-1-yl)butan-1-one 2k**



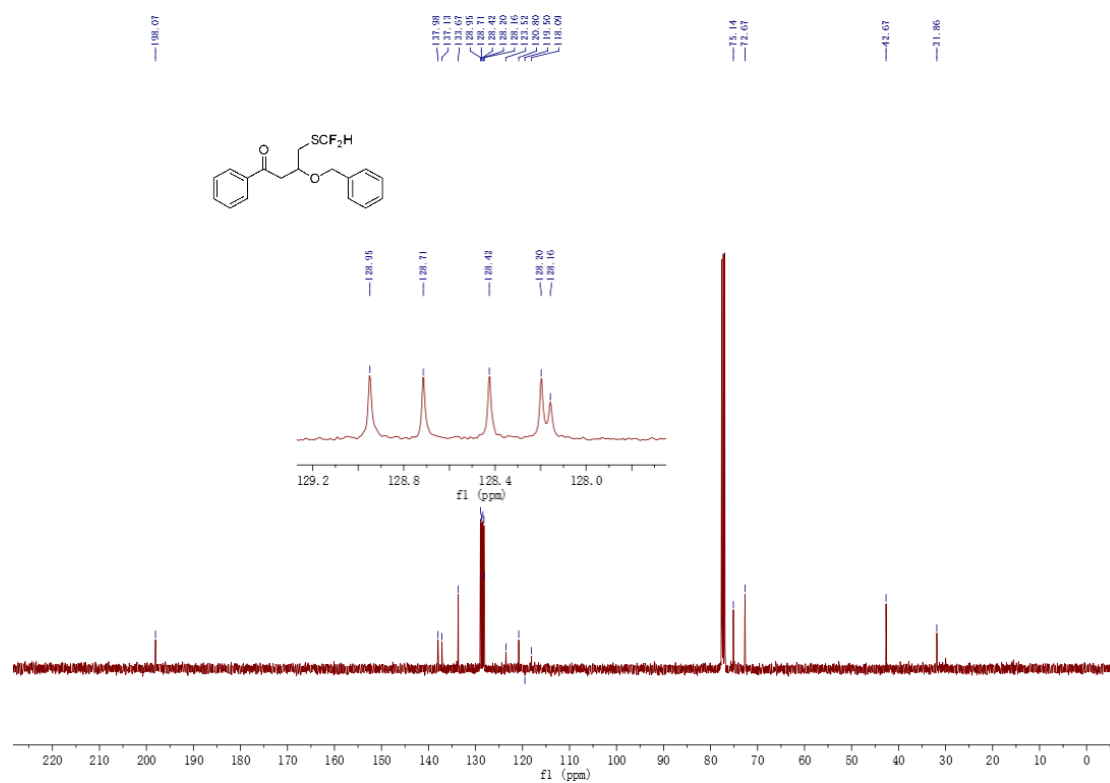
**^1H NMR spectrum of
3-(benzyloxy)-4-((difluoromethyl)thio)-1-phenylbutan-1-one 2l**



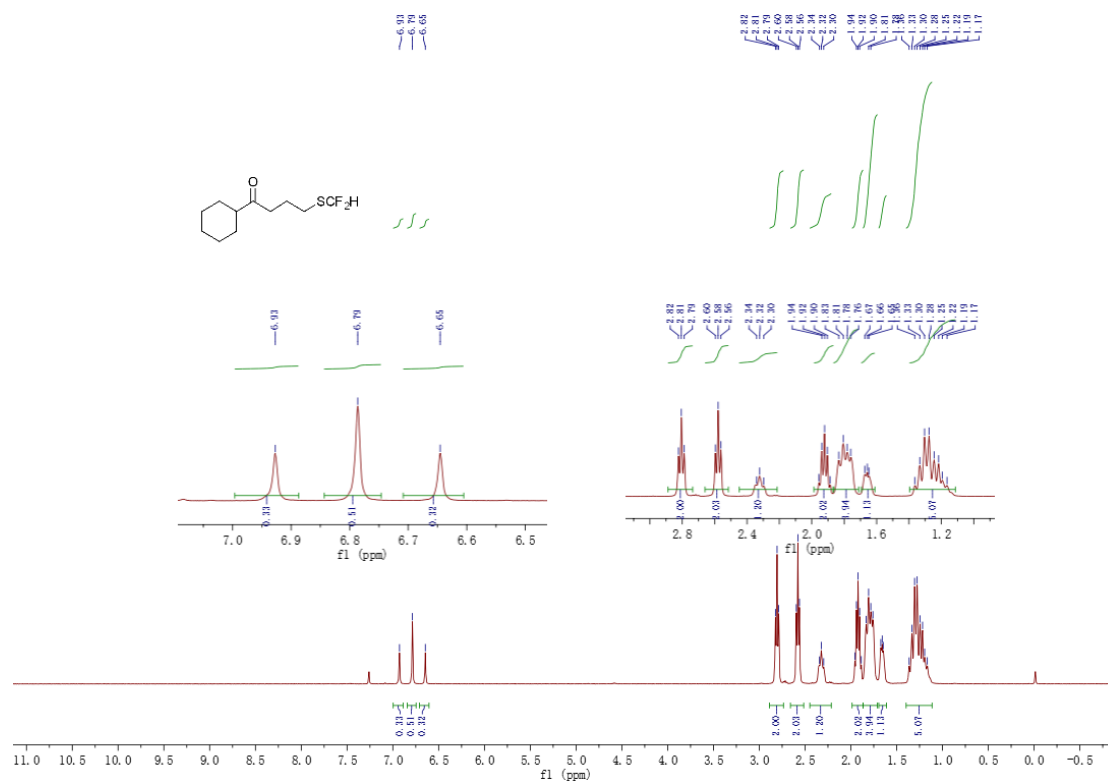
**^{19}F NMR spectrum of
3-(benzyloxy)-4-((difluoromethyl)thio)-1-phenylbutan-1-one 2l**



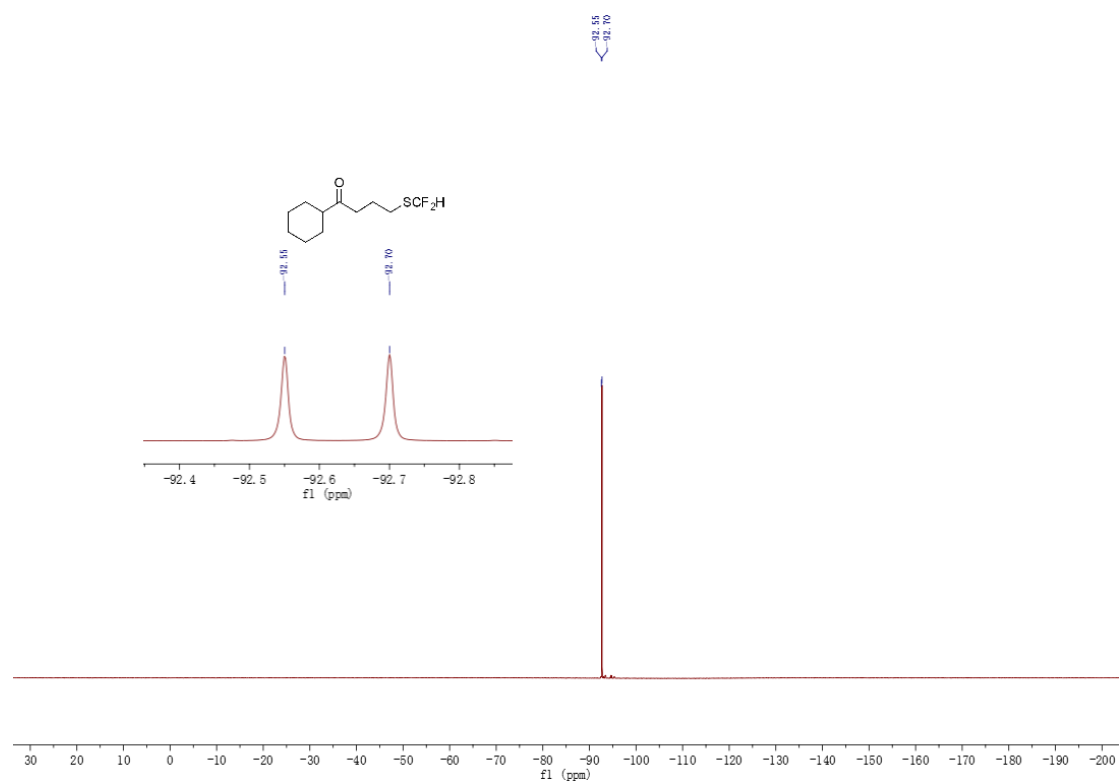
**^{13}C NMR spectrum of
3-(benzyloxy)-4-((difluoromethyl)thio)-1-phenylbutan-1-one 2l**



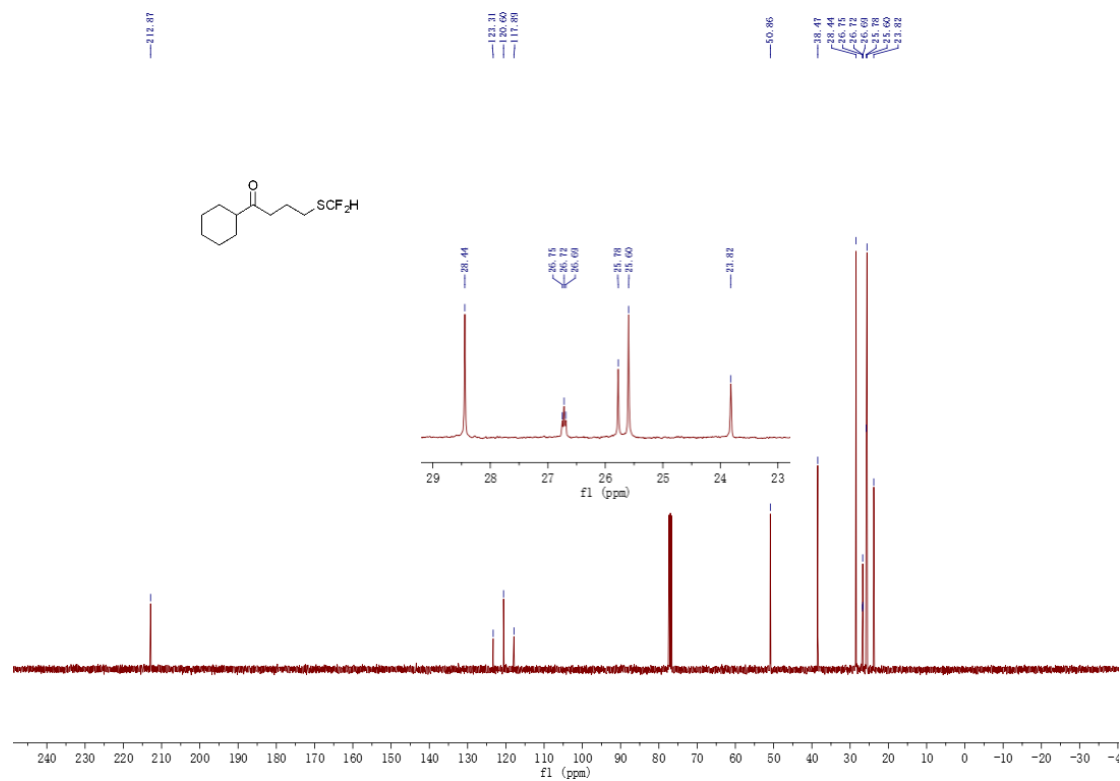
**^1H NMR spectrum of
1-cyclohexyl-4-((difluoromethyl)thio)butan-1-one 2m**



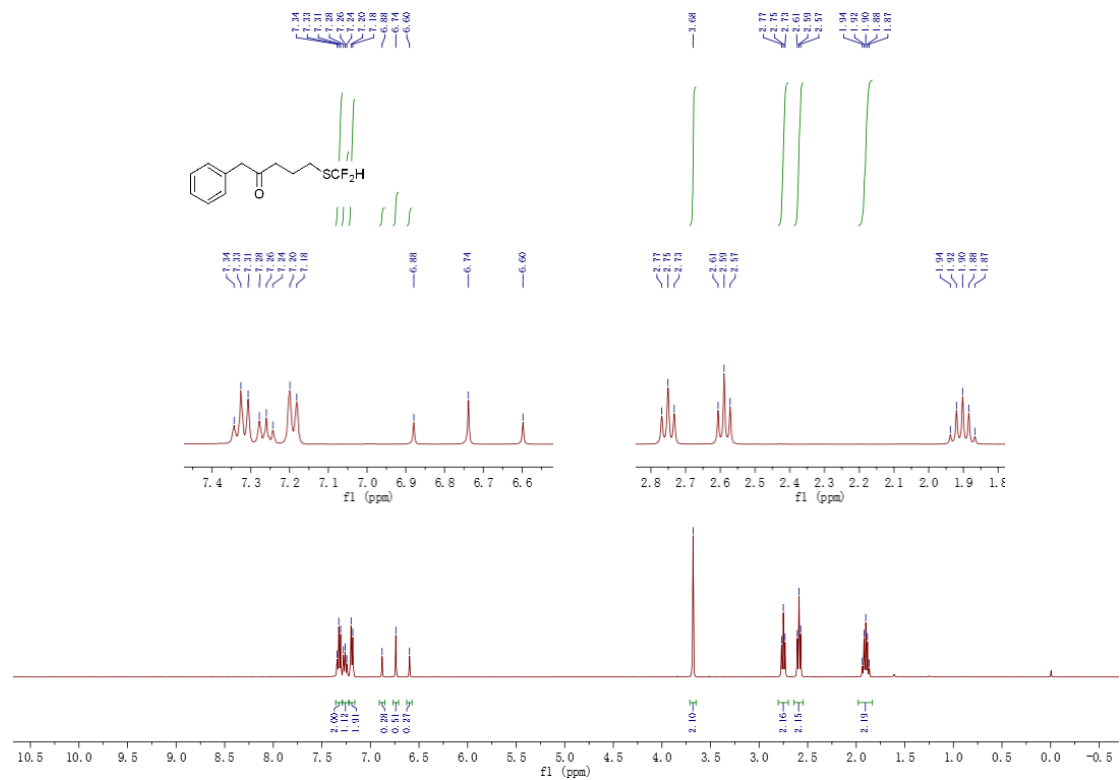
**^{19}F NMR spectrum of
1-cyclohexyl-4-((difluoromethyl)thio)butan-1-one 2m**



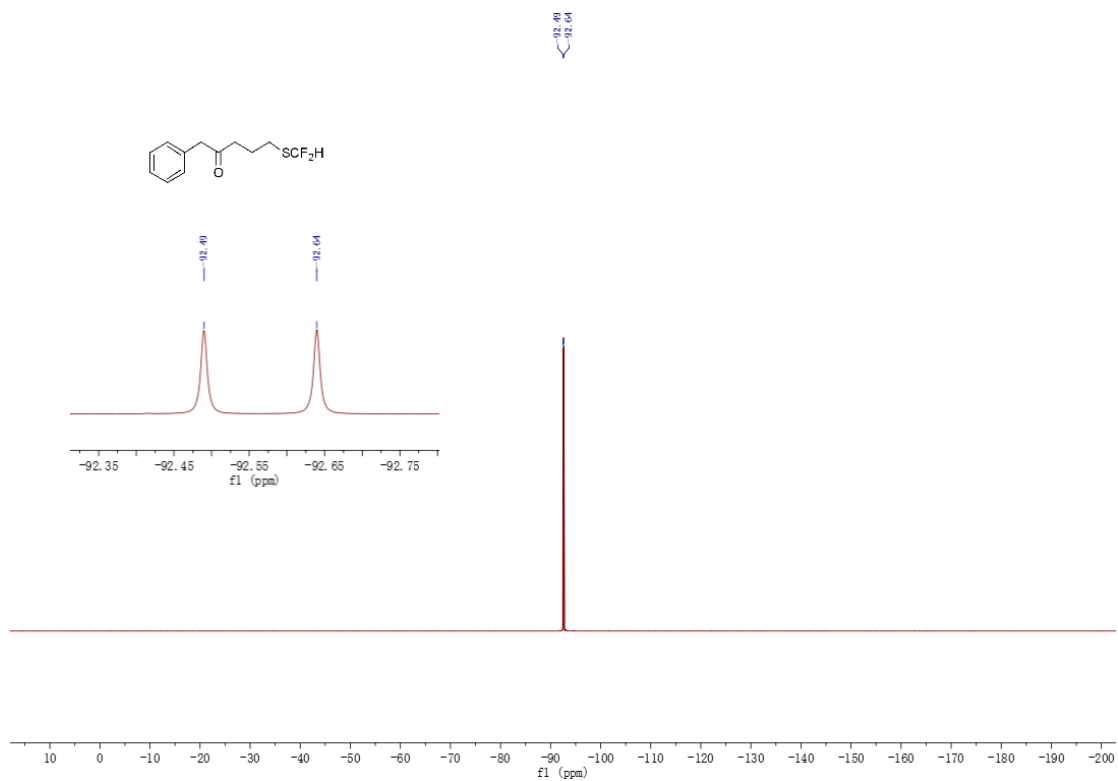
**^{13}C NMR spectrum of
1-cyclohexyl-4-((difluoromethyl)thio)butan-1-one 2m**



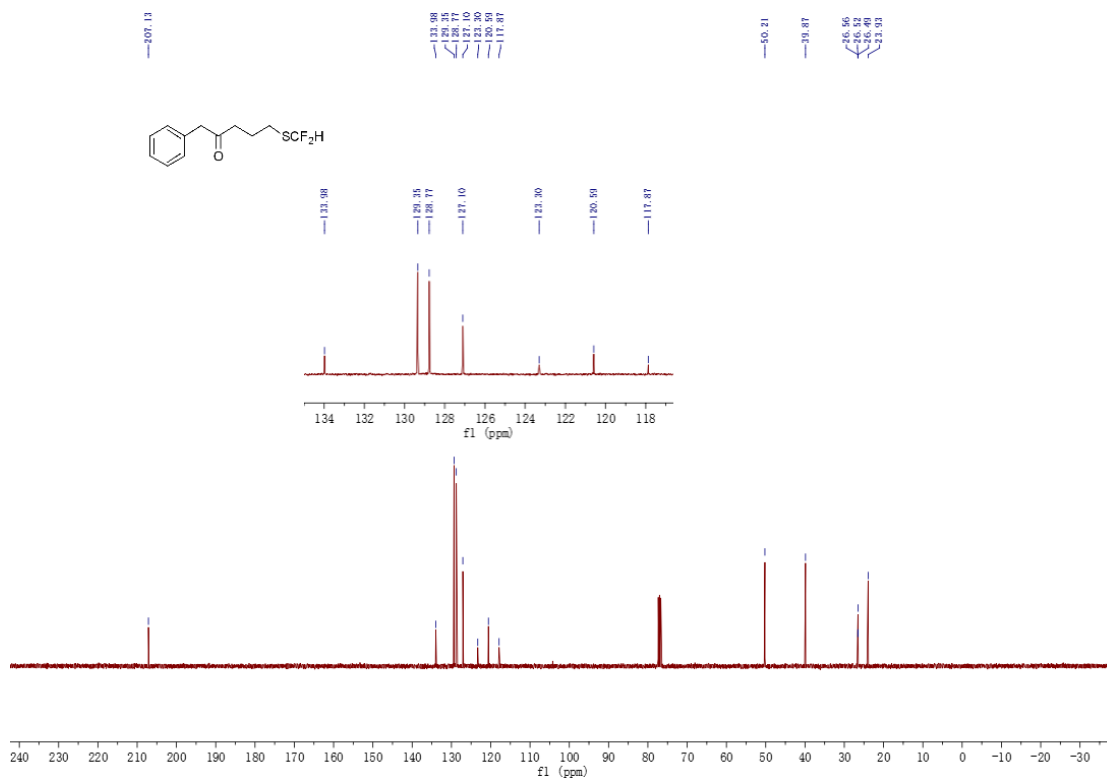
**^1H NMR spectrum of
5-((difluoromethyl)thio)-1-phenylpentan-2-one 2n**



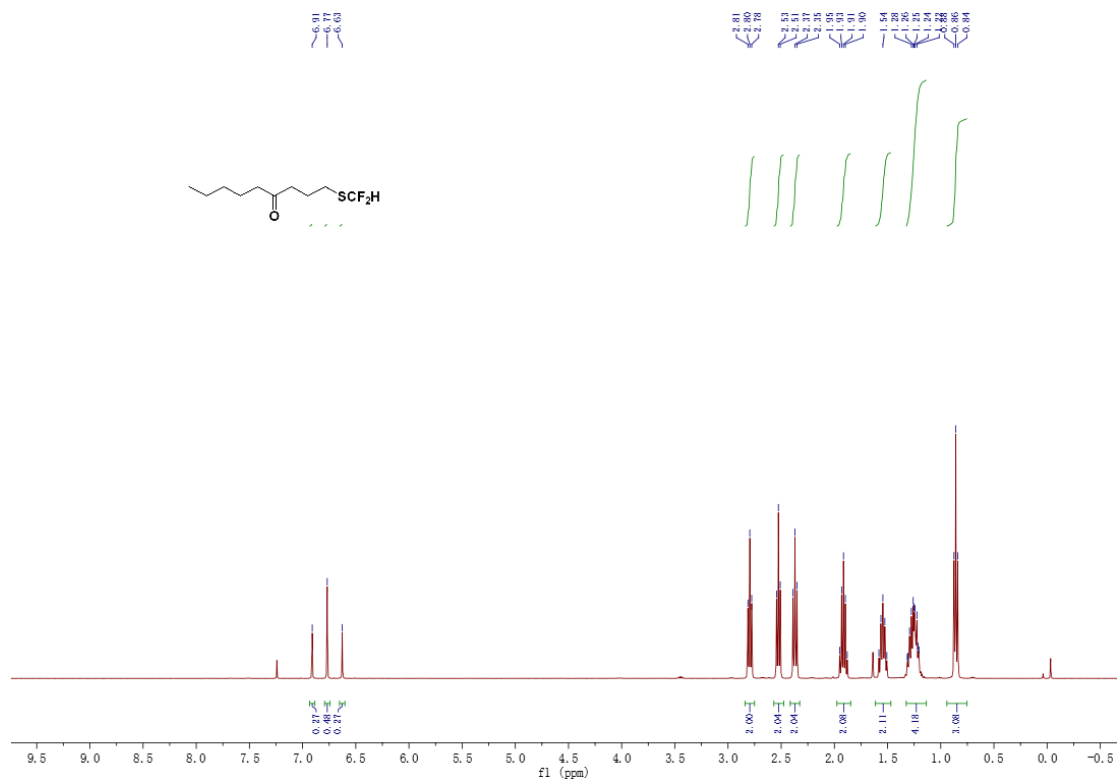
**^{19}F NMR spectrum of
5-((difluoromethyl)thio)-1-phenylpentan-2-one 2n**



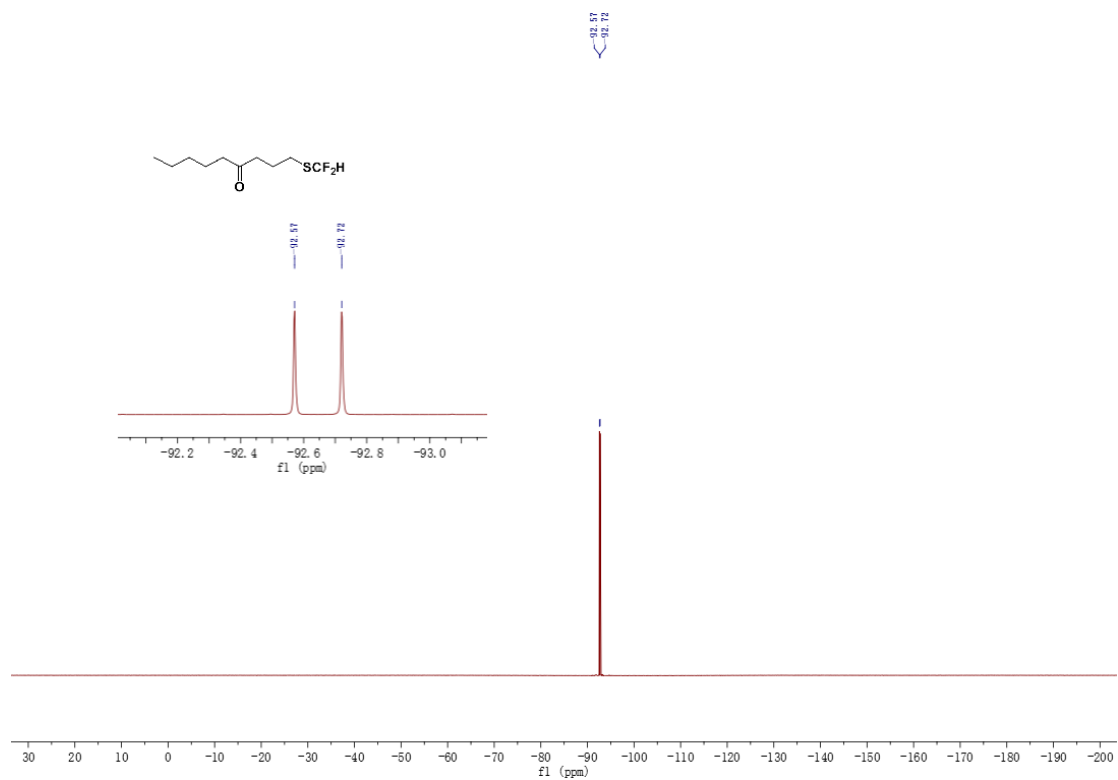
**^{13}C NMR spectrum of
5-((difluoromethyl)thio)-1-phenylpentan-2-one 2n**



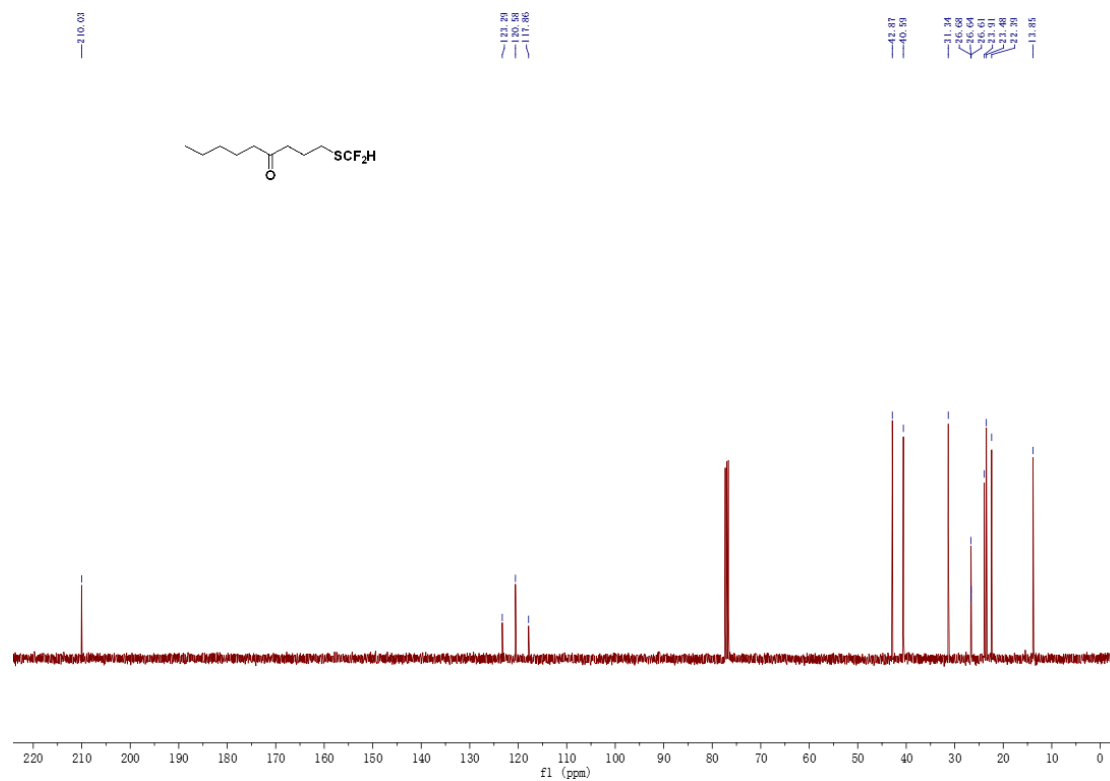
**^1H NMR spectrum of
1-((difluoromethyl)thio)nonan-4-one 2o**



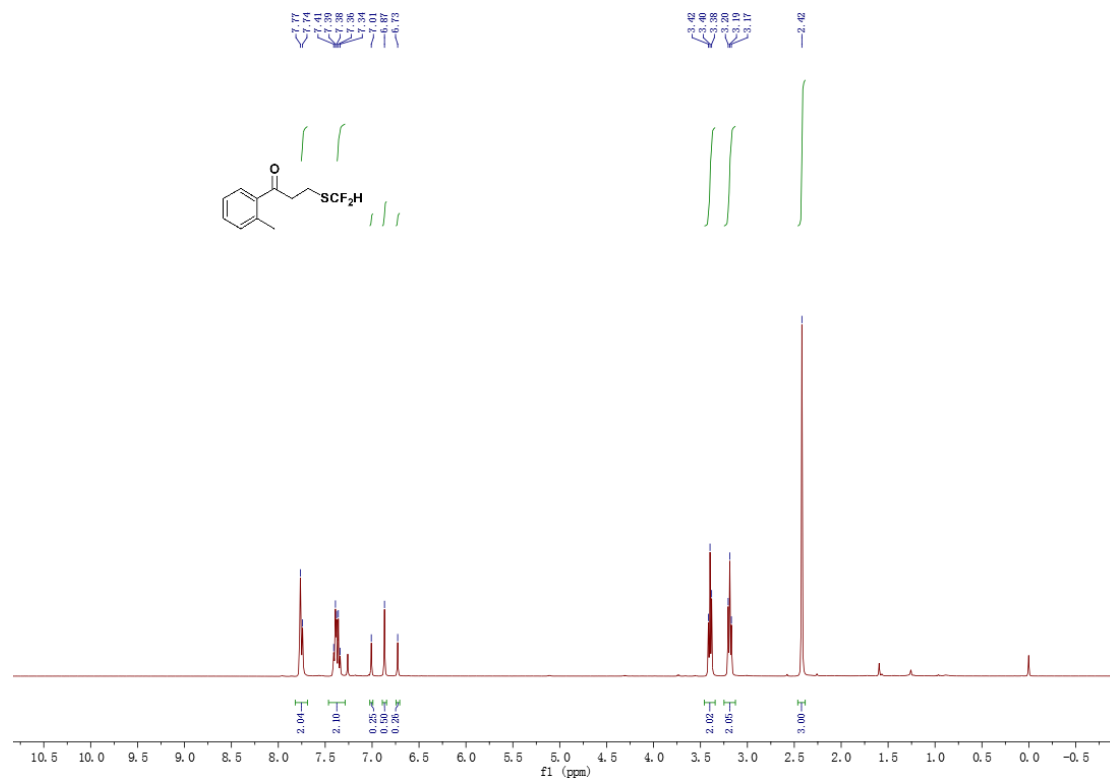
**^{19}F NMR spectrum of
1-((difluoromethyl)thio)nonan-4-one 2o**



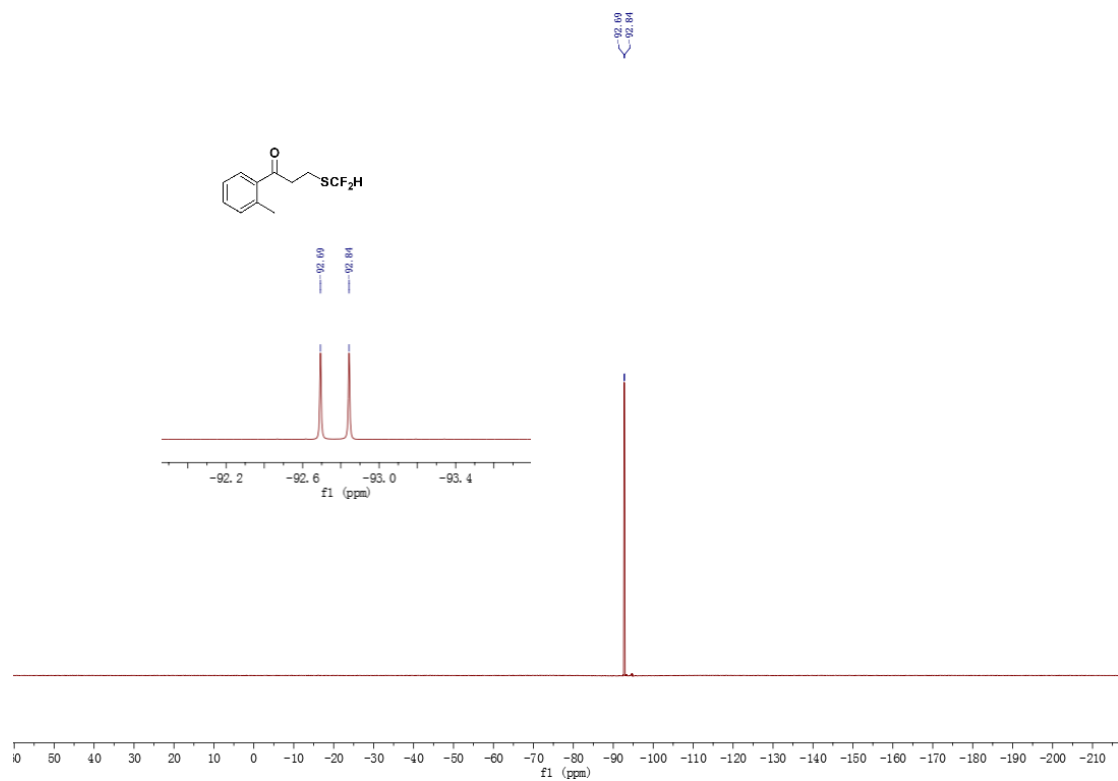
**^{13}C NMR spectrum of
1-((difluoromethyl)thio)nonan-4-one 2o**



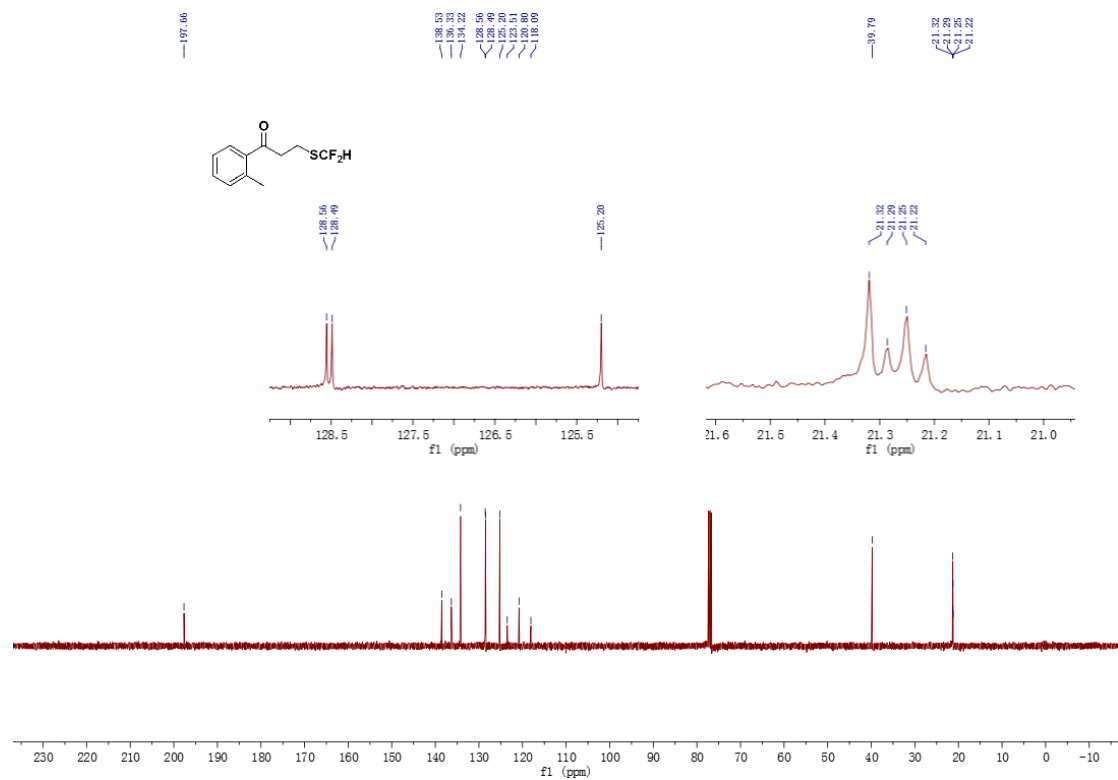
**^1H NMR spectrum of
3-((difluoromethyl)thio)-1-(*o*-tolyl)propan-1-one 4a**



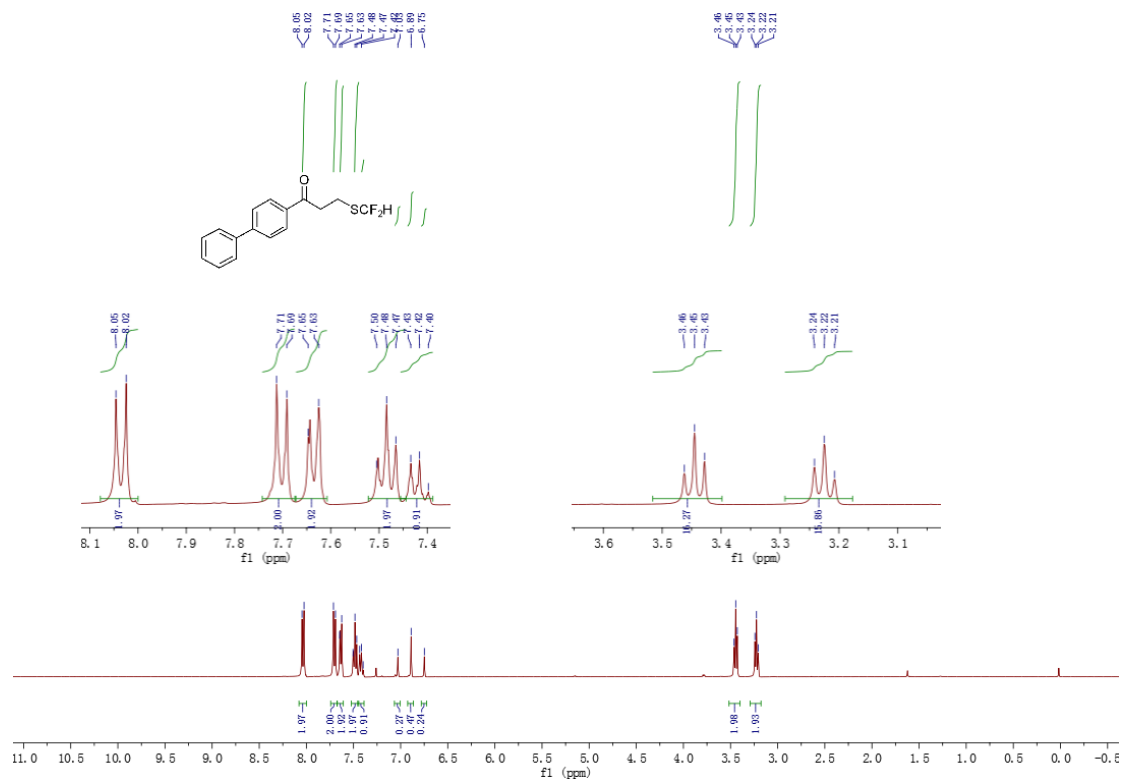
**^{19}F NMR spectrum of
3-((difluoromethyl)thio)-1-(*o*-tolyl)propan-1-one 4a**



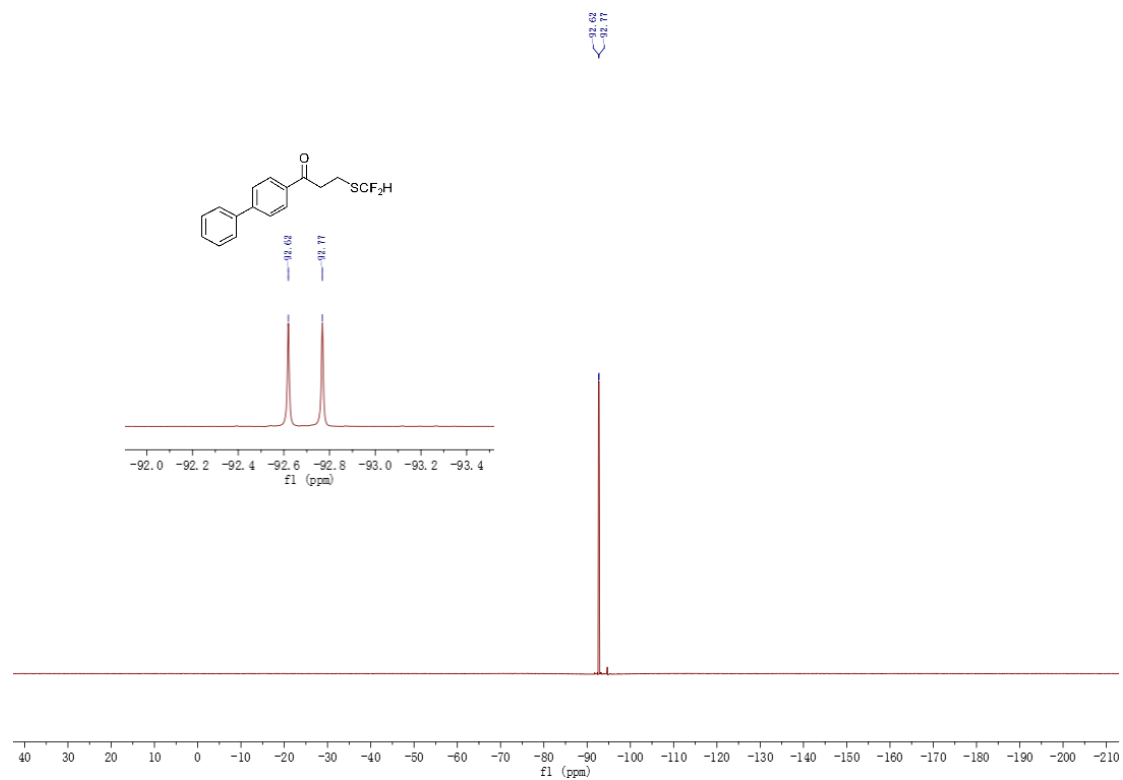
**^{13}C NMR spectrum of
3-((difluoromethyl)thio)-1-(*o*-tolyl)propan-1-one 4a**



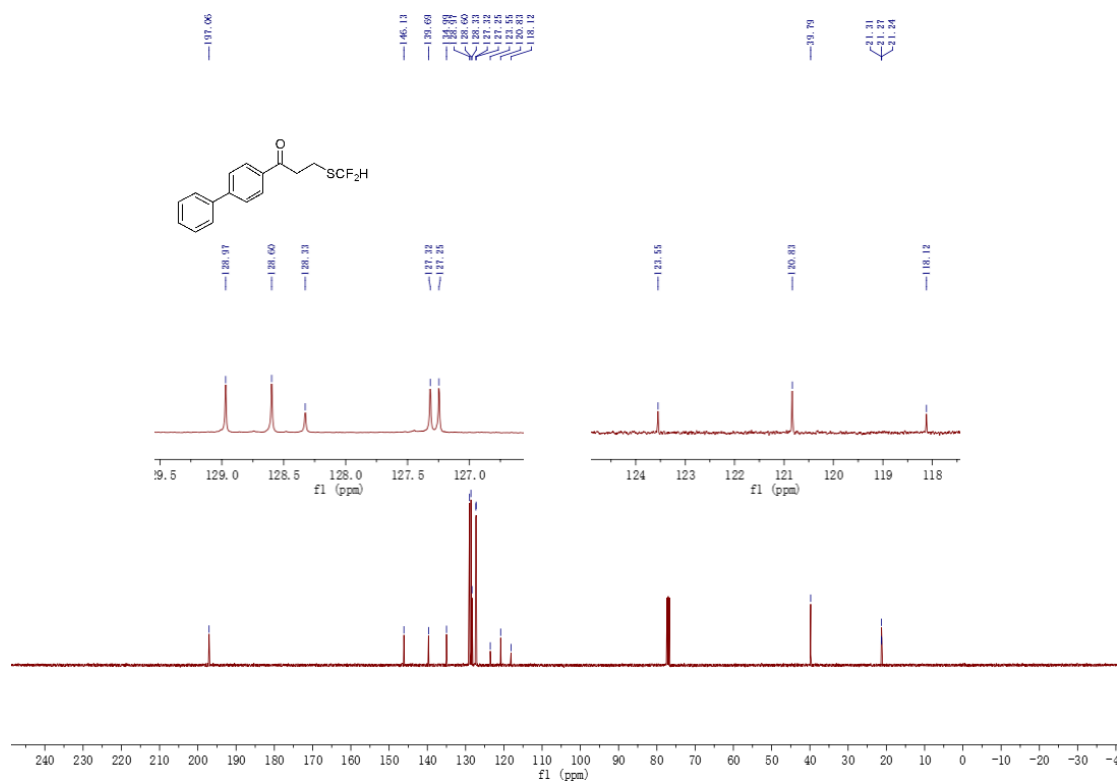
¹H NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-3-((difluoromethyl)thio)propan-1-one 4b



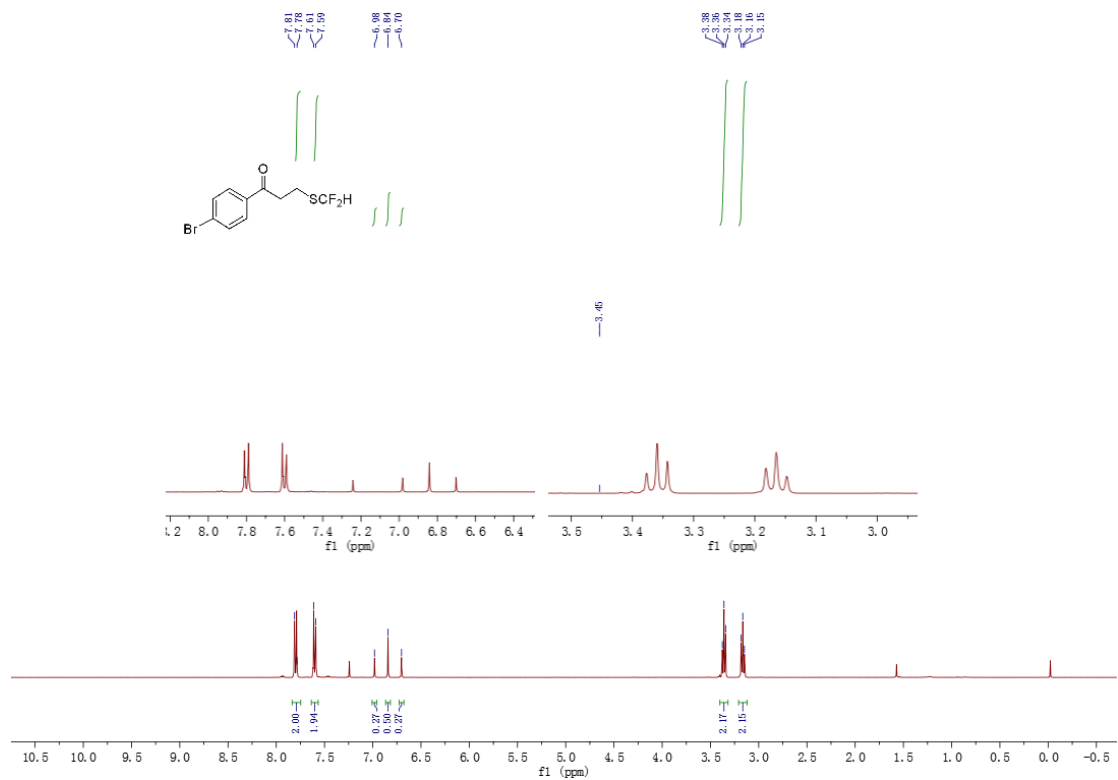
¹⁹F NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-3-((difluoromethyl)thio)propan-1-one 4b



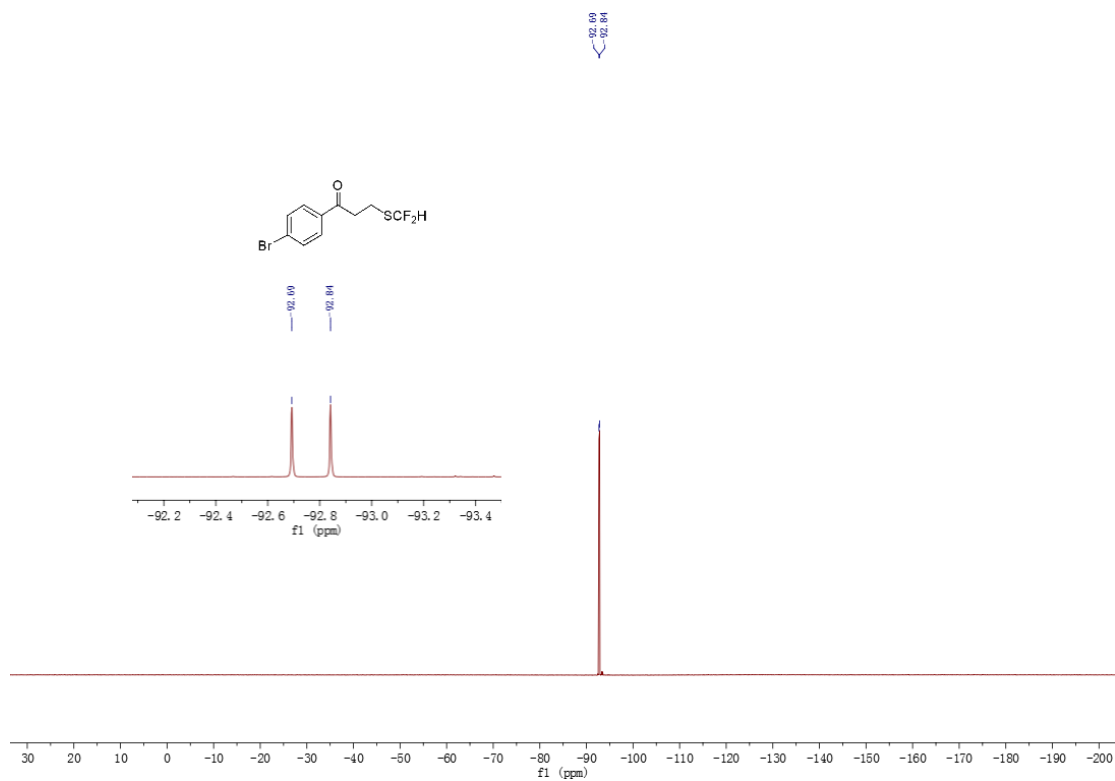
^{13}C NMR spectrum of
1-([1,1'-biphenyl]-4-yl)-3-((difluoromethyl)thio)propan-1-one 4b



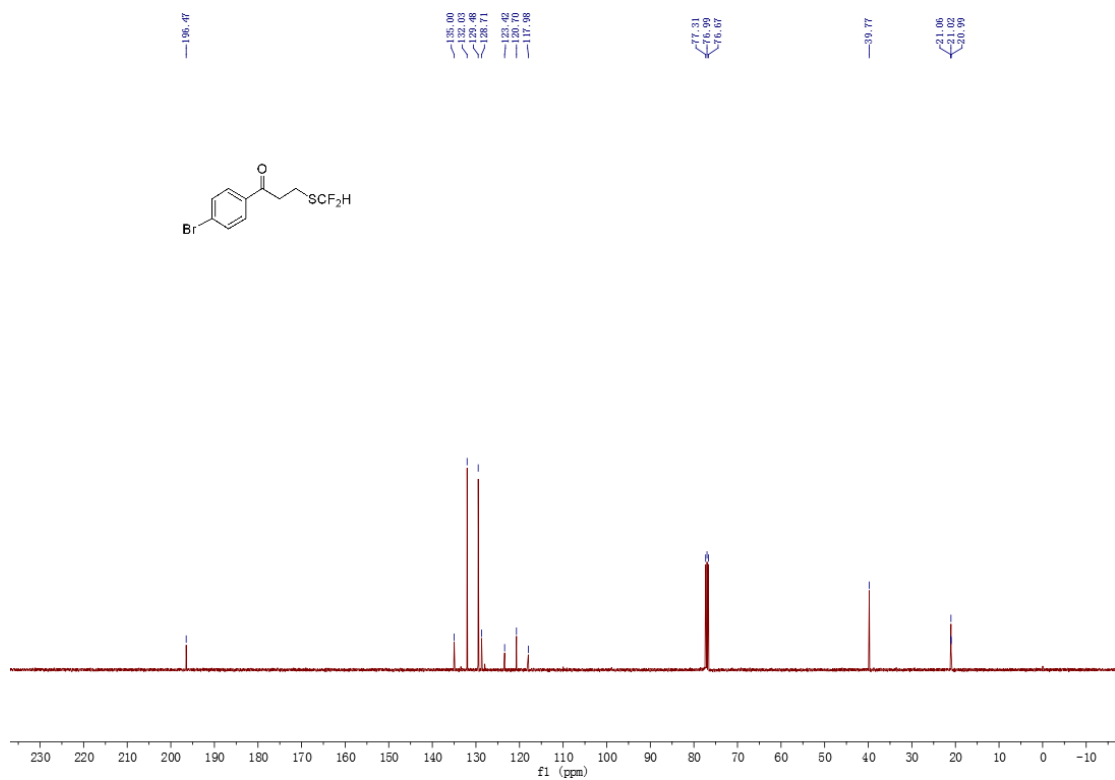
^1H NMR spectrum of
1-(4-bromophenyl)-3-((difluoromethyl)thio)propan-1-one 4c



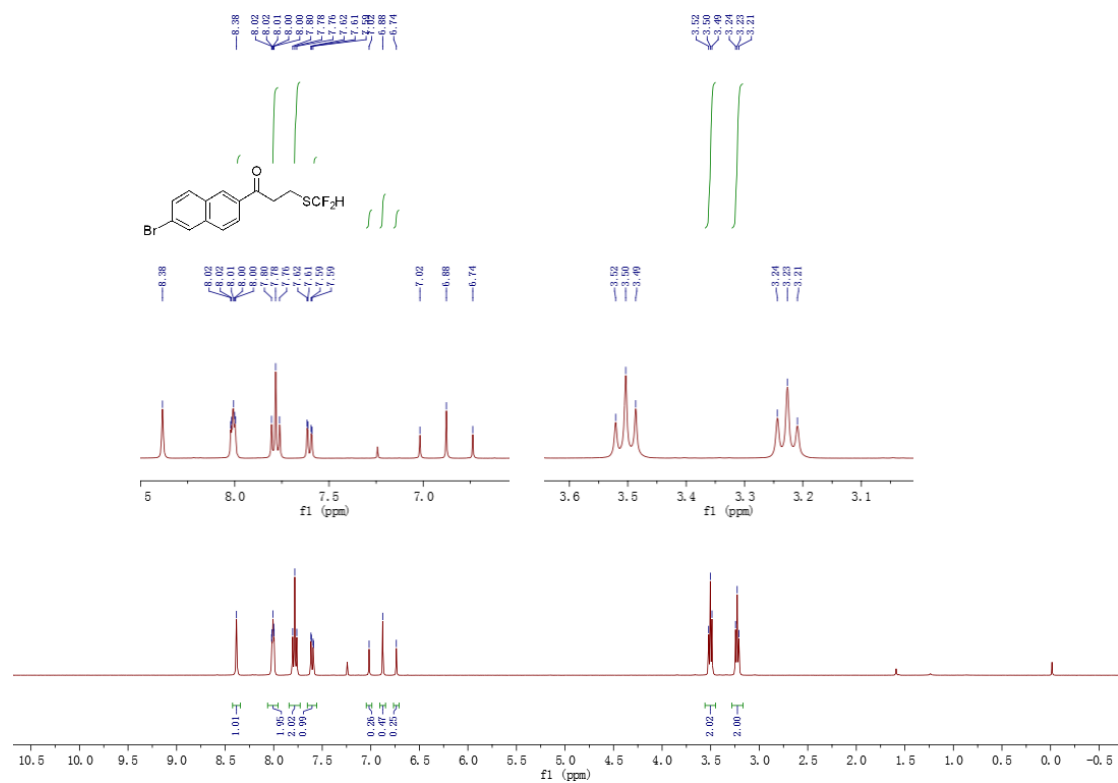
**^{19}F NMR spectrum of
1-(4-bromophenyl)-3-((difluoromethyl)thio)propan-1-one 4c**



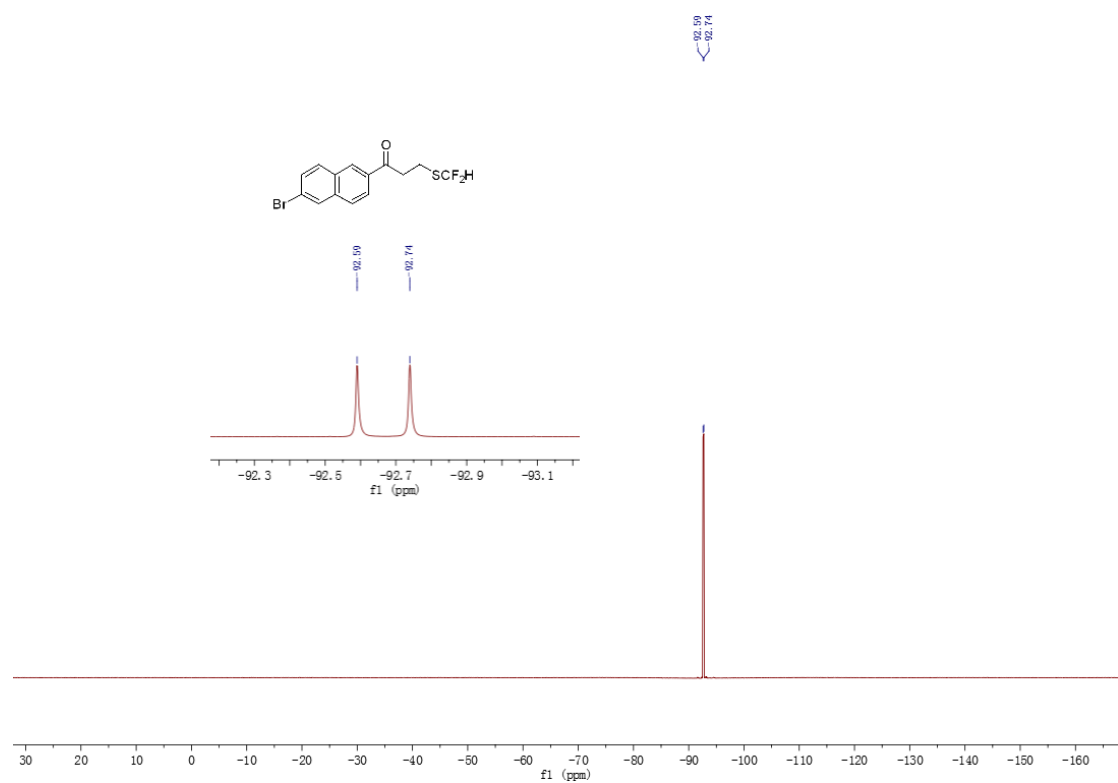
**^{13}C NMR spectrum of
1-(4-bromophenyl)-3-((difluoromethyl)thio)propan-1-one 4c**



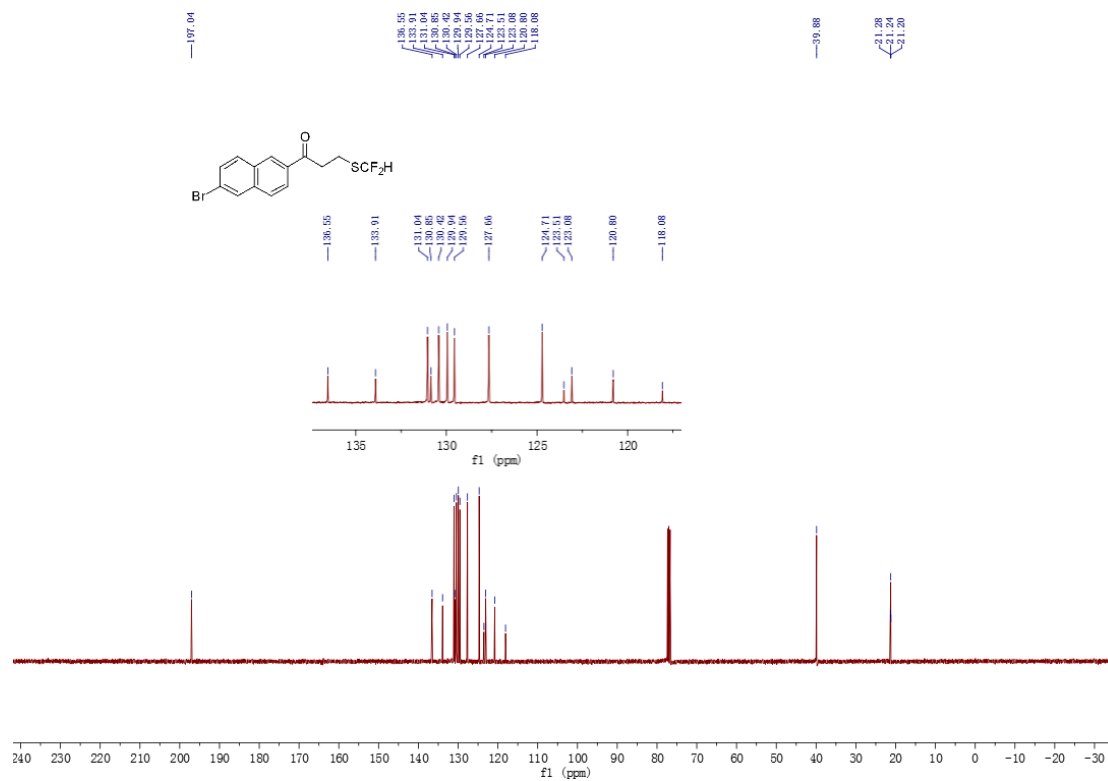
**¹H NMR spectrum of
1-(6-bromonaphthalen-2-yl)-3-((difluoromethyl)thio)propan-1-one 4d**



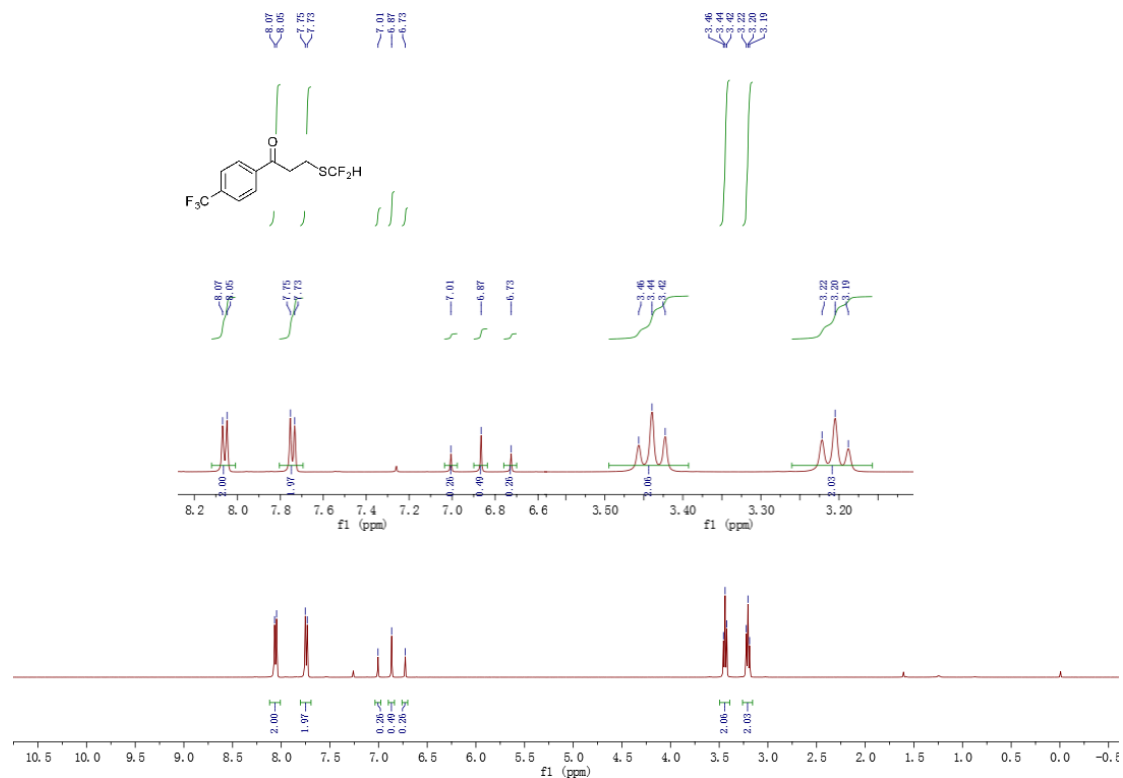
**¹⁹F NMR spectrum of
1-(6-bromonaphthalen-2-yl)-3-((difluoromethyl)thio)propan-1-one 4d**



**^{13}C NMR spectrum of
1-(6-bromonaphthalen-2-yl)-3-((difluoromethyl)thio)propan-1-one 4d**



**^1H NMR spectrum of
3-((difluoromethyl)thio)-1-(4-(trifluoromethyl)phenyl)propan-1-one 4e**



Chemical structure of 2-(4-(trifluoromethyl)phenyl)ethan-1-one and its ^{13}C NMR spectrum.

Chemical structure: CC(=O)c1ccc(C(F)(F)F)cc1

^{13}C NMR spectrum (CDCl₃) showing peaks at:

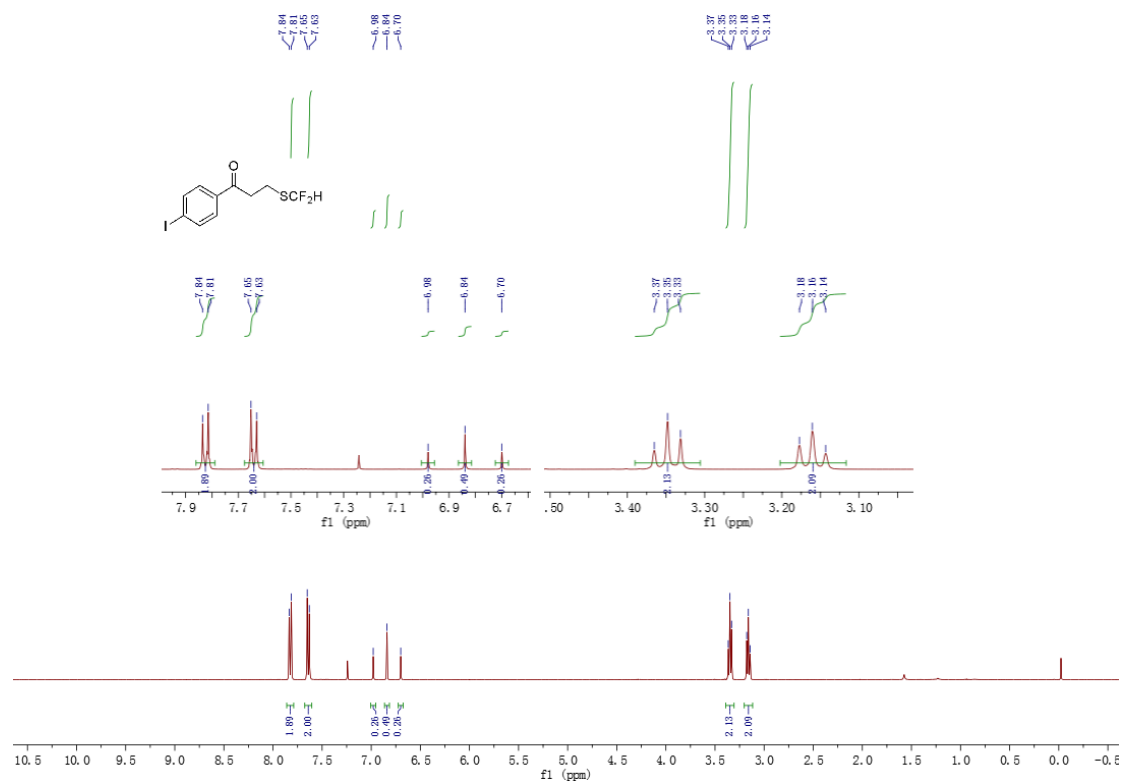
- 154.86 ppm (C=O)
- 132.73 ppm (Aromatic C)
- 132.88 ppm (Aromatic C)
- 63.26 ppm (CH₂)
- 92.86 ppm (CDCl₃ solvent)

Chemical structure: FC(F)(F)c1ccc(cc1)C(=O)CC(F)(F)F

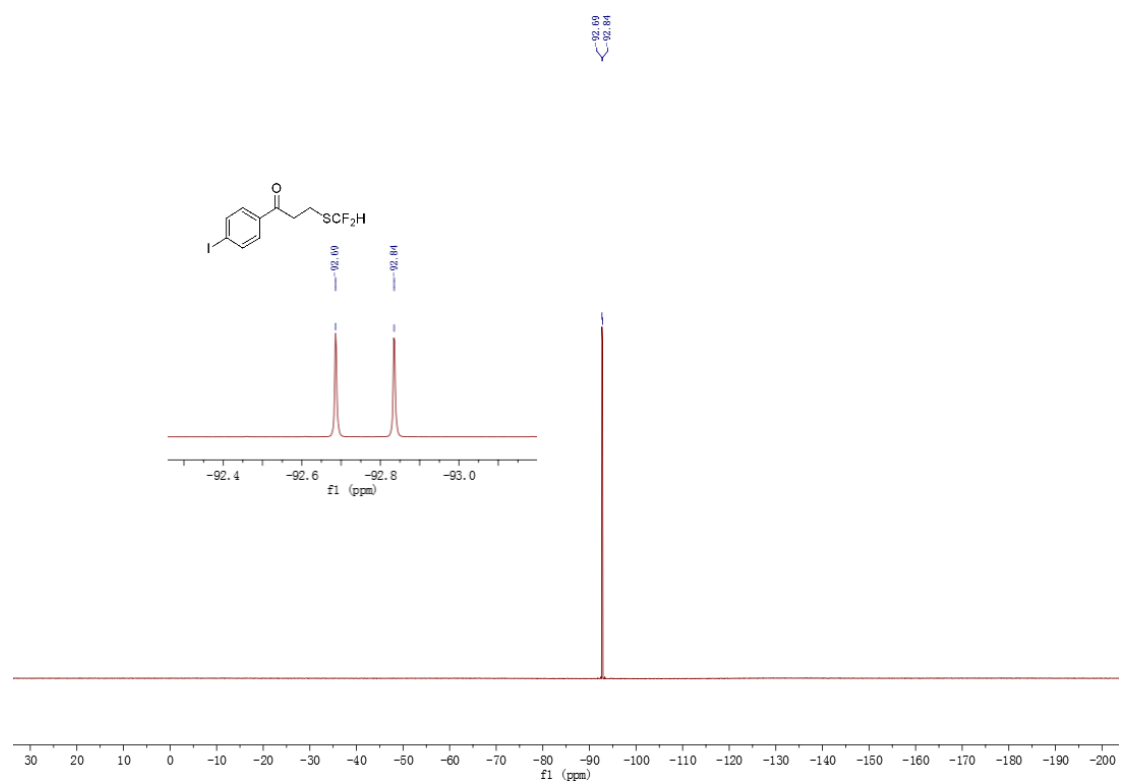
¹³C NMR spectrum (ppm):

- 135.21, 134.89, 134.56, 134.23 (aromatic carbons)
- 127.54, 125.83, 125.70, 125.72, 124.82 (aromatic carbons)
- 123.39, 122.11, 120.68 (aromatic carbons)
- 119.40, 117.96 (aromatic carbons)
- 19.56, 19.40, 19.24, 19.08, 18.92, 18.76, 18.60, 18.44, 18.28, 18.12, 17.96, 17.80, 17.64, 17.48, 17.32, 17.16, 17.00, 16.84, 16.68, 16.52, 16.36, 16.20, 16.04, 15.88, 15.72, 15.56, 15.40, 15.24, 15.08, 14.92, 14.76, 14.60, 14.44, 14.28, 14.12, 13.96, 13.80, 13.64, 13.48, 13.32, 13.16, 13.00, 12.84, 12.68, 12.52, 12.36, 12.20, 12.04, 11.88, 11.72, 11.56, 11.40, 11.24, 11.08, 10.92, 10.76, 10.60, 10.44, 10.28, 10.12, 9.96, 9.80, 9.64, 9.48, 9.32, 9.16, 9.00, 8.84, 8.68, 8.52, 8.36, 8.20, 8.04, 7.88, 7.72, 7.56, 7.40, 7.24, 7.08, 6.92, 6.76, 6.60, 6.44, 6.28, 6.12, 5.96, 5.80, 5.64, 5.48, 5.32, 5.16, 5.00, 4.84, 4.68, 4.52, 4.36, 4.20, 4.04, 3.88, 3.72, 3.56, 3.40, 3.24, 3.08, 2.92, 2.76, 2.60, 2.44, 2.28, 2.12, 1.96, 1.80, 1.64, 1.48, 1.32, 1.16, 1.00, 0.84, 0.68, 0.52, 0.36, 0.20, 0.04 (aliphatic carbons)

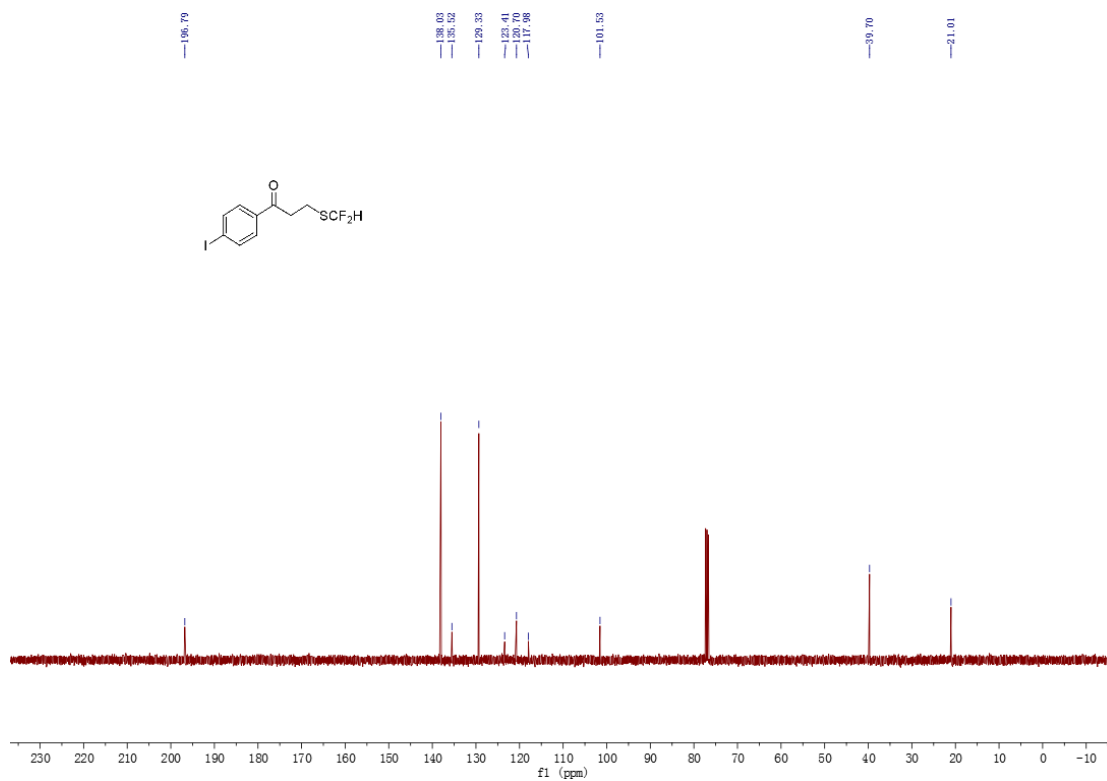
**¹H NMR spectrum of
3-((difluoromethyl)thio)-1-(4-iodophenyl)propan-1-one 4f**



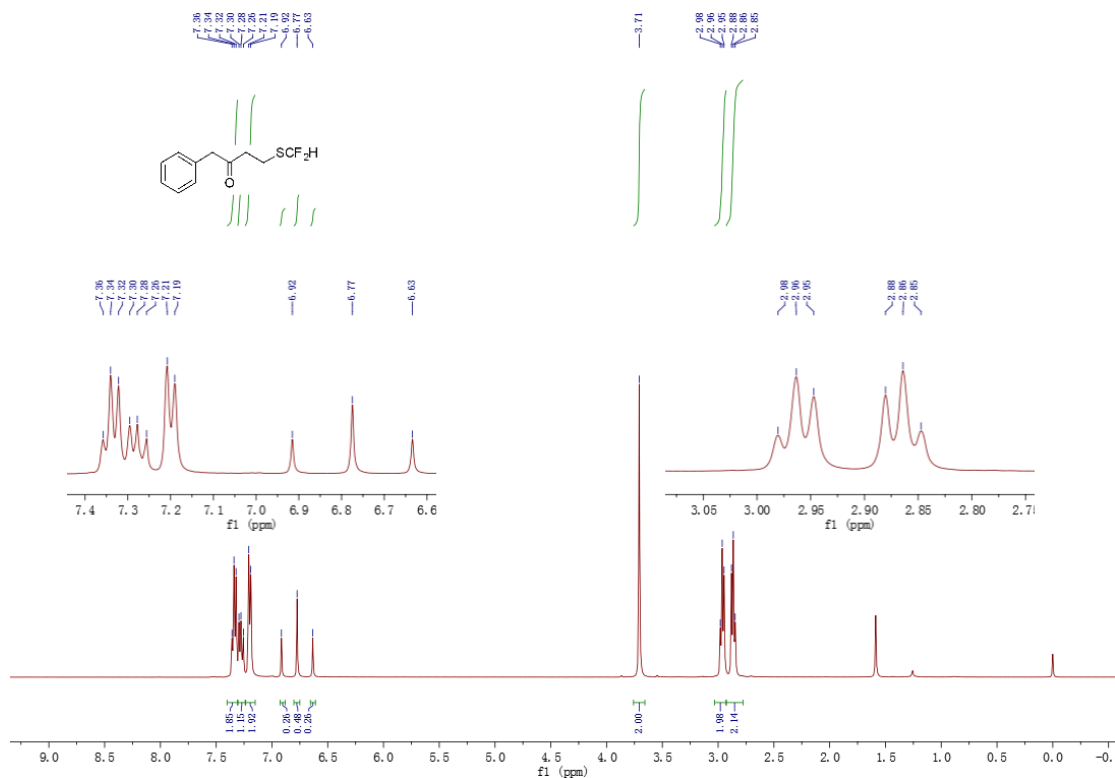
**¹⁹F NMR spectrum of
3-((difluoromethyl)thio)-1-(4-iodophenyl)propan-1-one 4f**



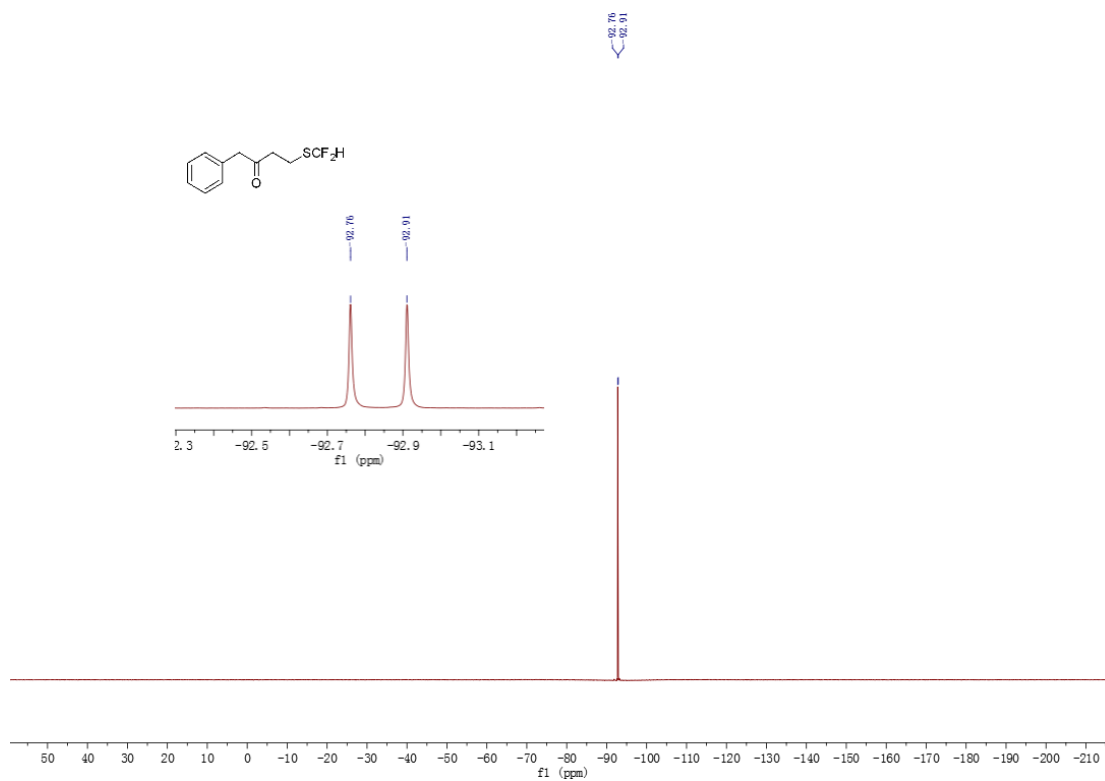
**^{13}C NMR spectrum of
3-((difluoromethyl)thio)-1-(4-iodophenyl)propan-1-one 4f**



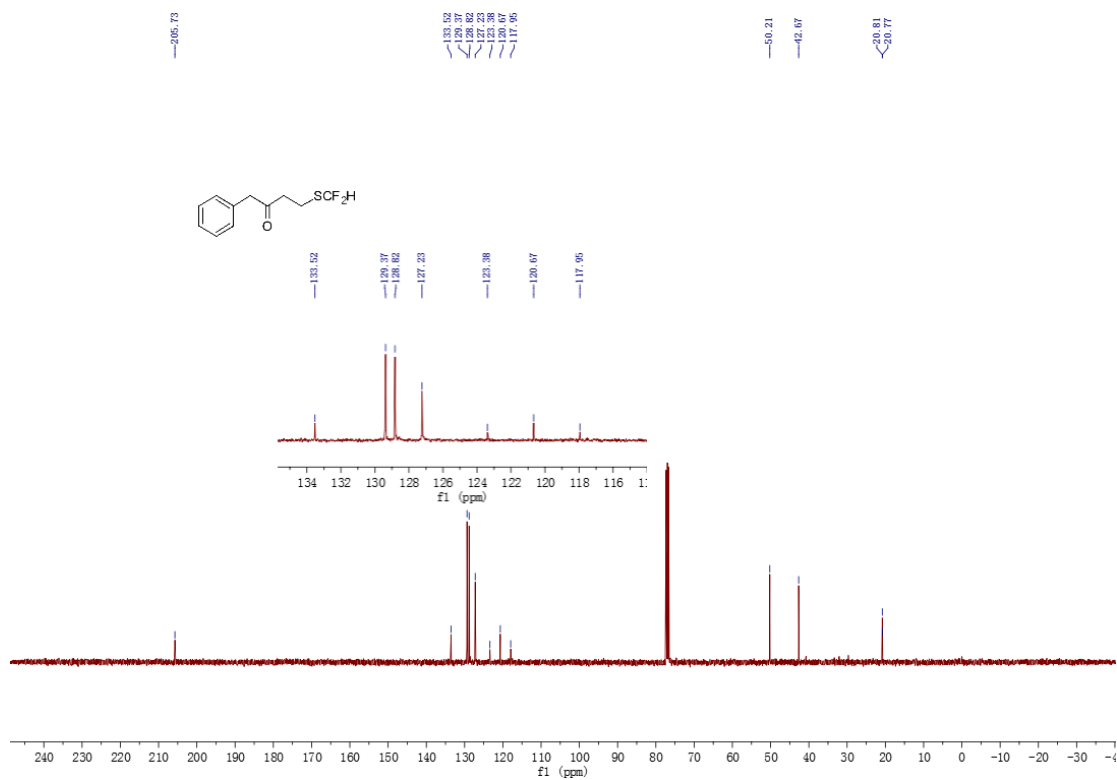
**^1H NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-2-one 4g**



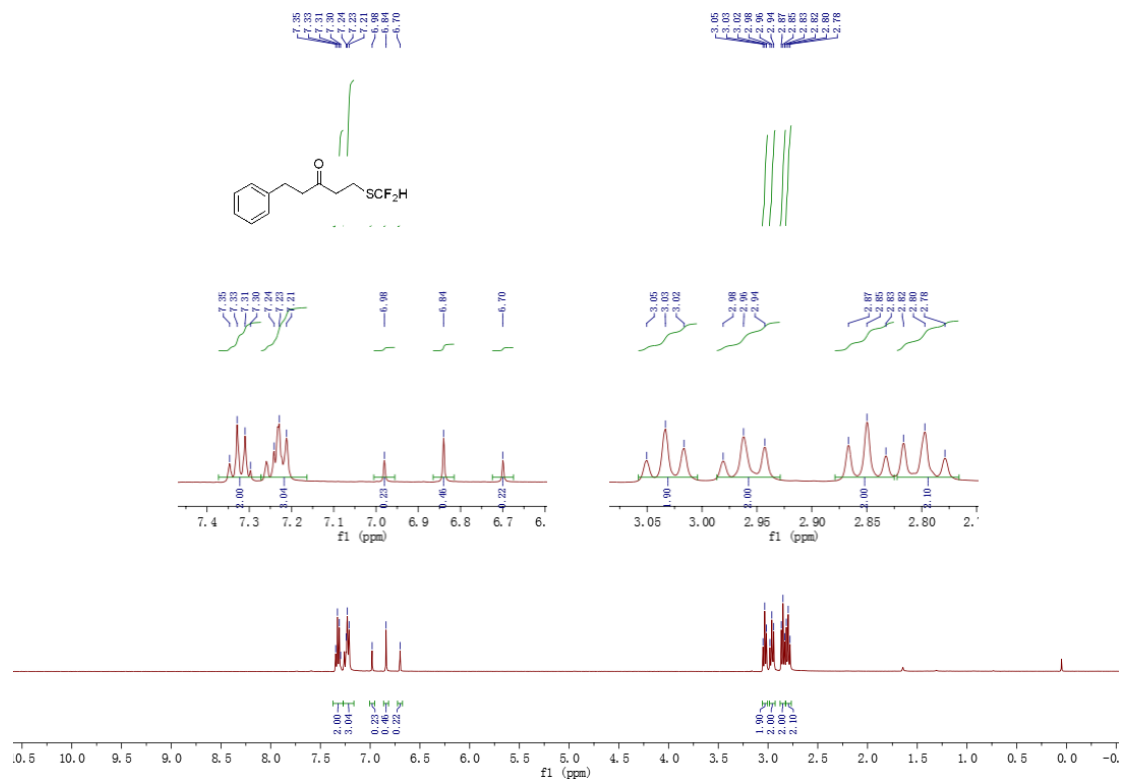
**^{19}F NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-2-one 4g**



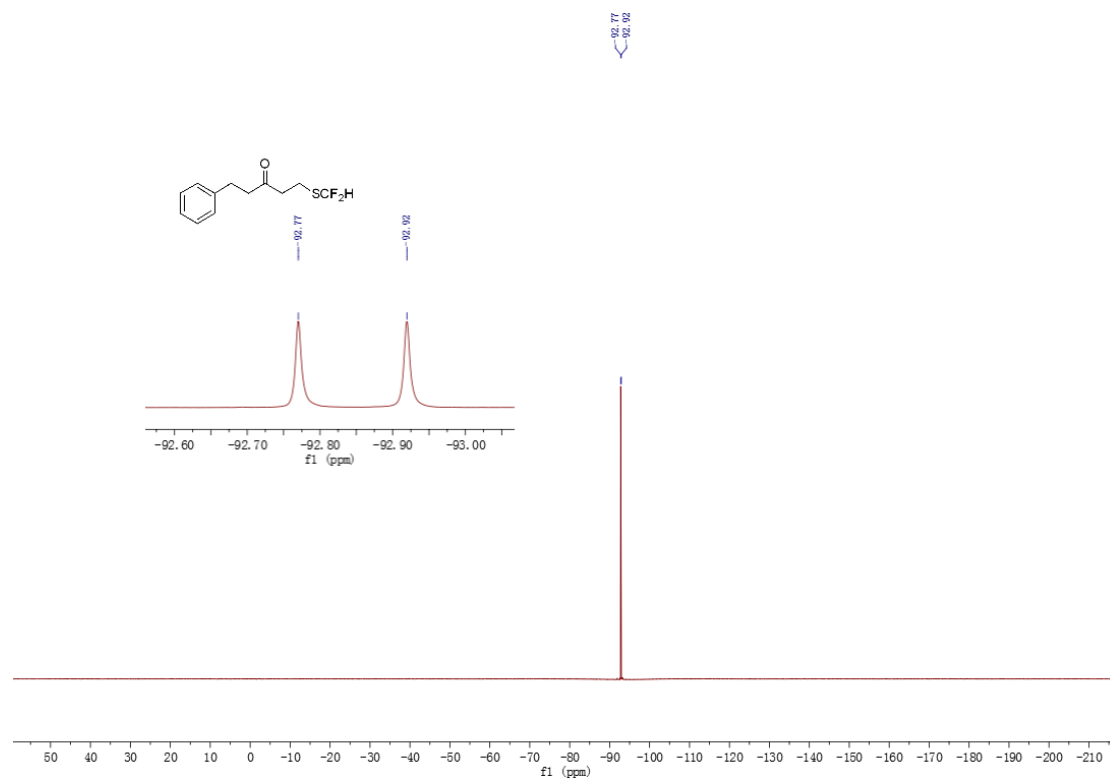
**^{13}C NMR spectrum of
4-((difluoromethyl)thio)-1-phenylbutan-2-one 4g**



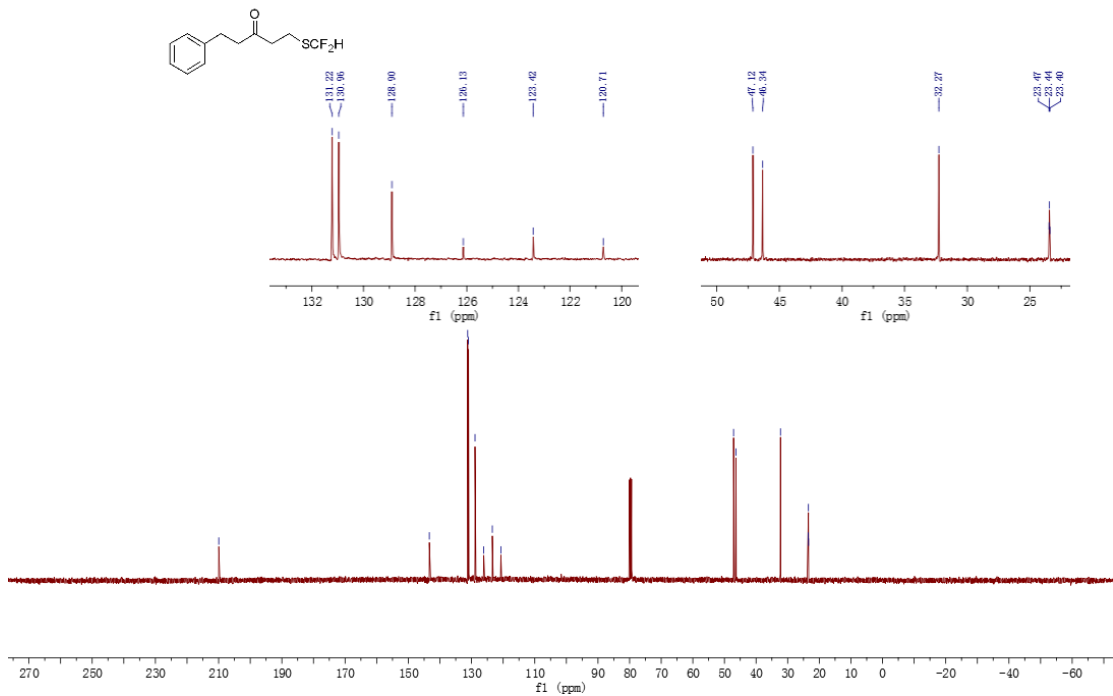
**^1H NMR spectrum of
1-((difluoromethyl)thio)-5-phenylpentan-3-one 4h**



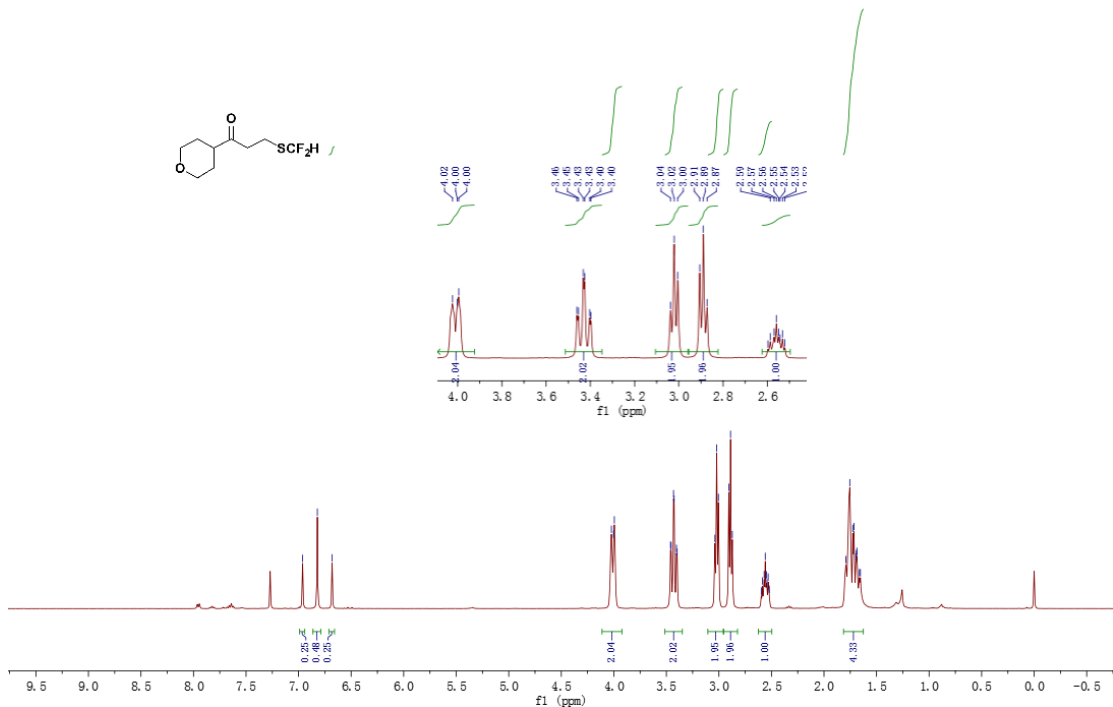
**^{19}F NMR spectrum of
1-((difluoromethyl)thio)-5-phenylpentan-3-one 4h**



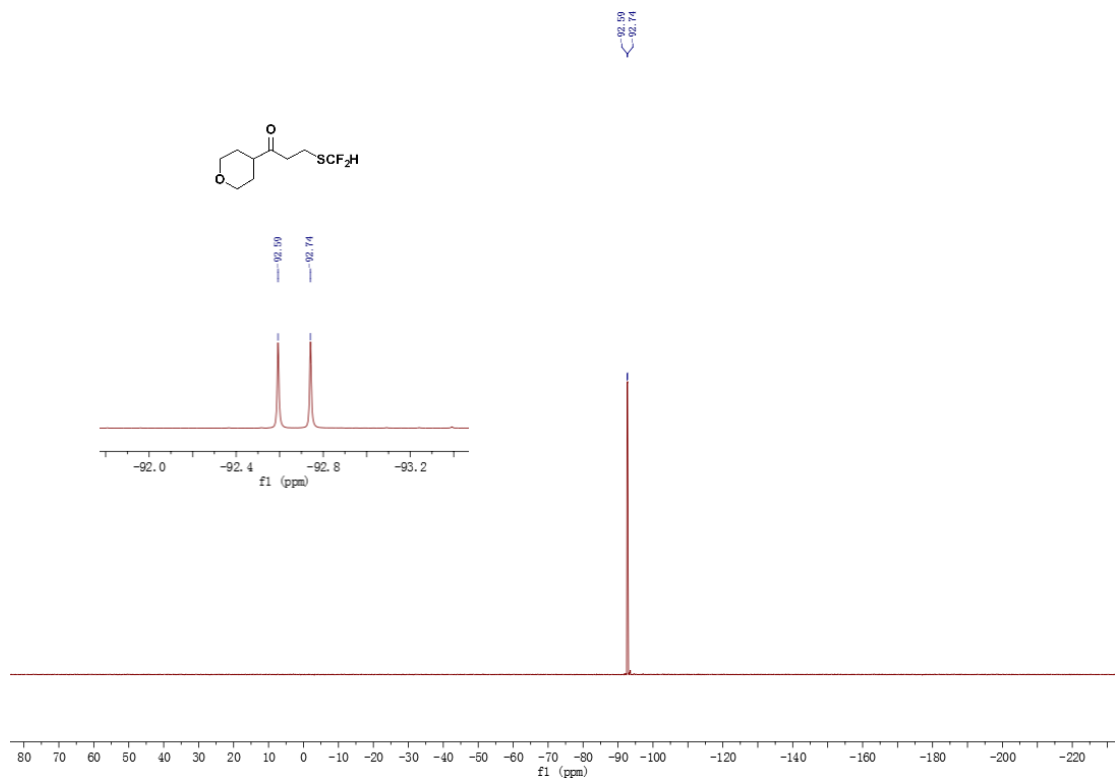
—209, 90
—143, 35
—131, 22
—130, 90
—128, 90
—126, 13
—123, 42
—120, 71
—47, 12
—46, 34
—32, 27
—23, 47
—23, 44
—23, 40



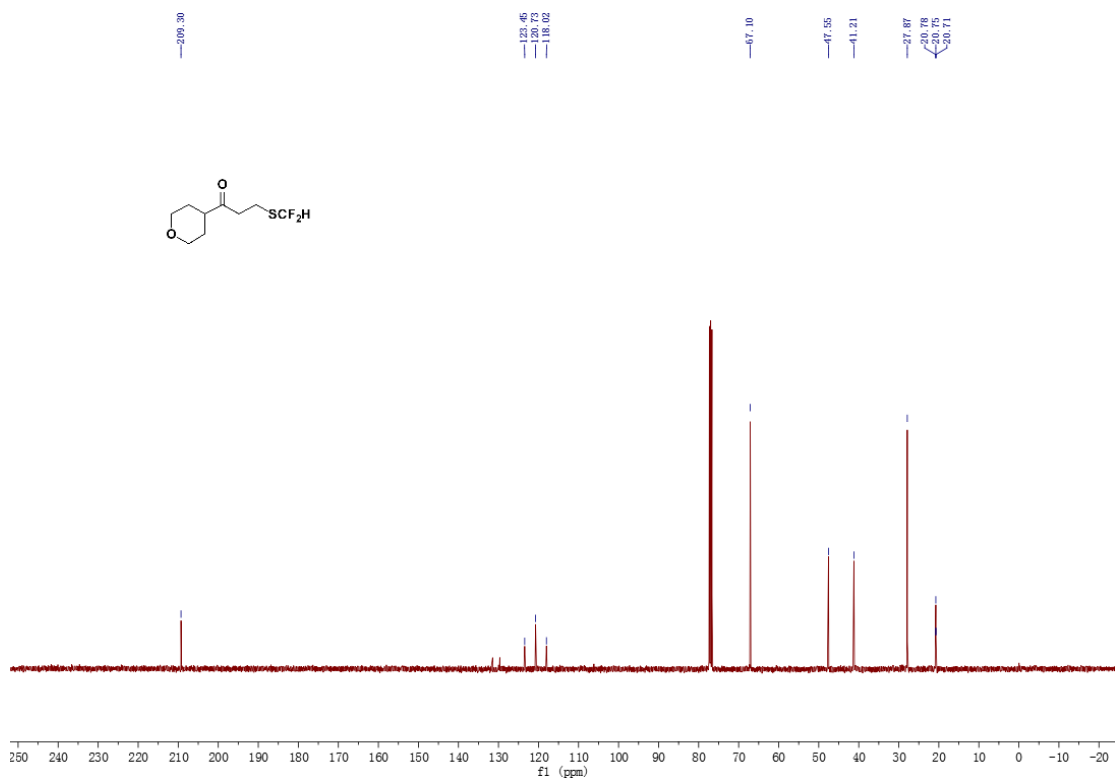
$\begin{array}{r} 6.96 \\ 6.82 \\ 6.68 \end{array}$

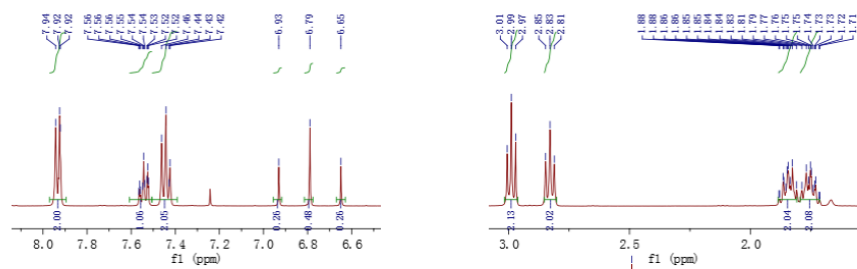
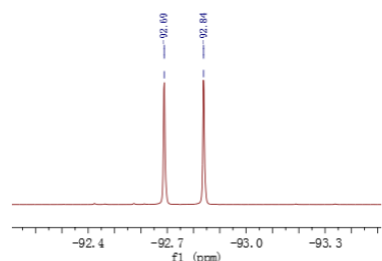


**^{19}F NMR spectrum of
3-((difluoromethyl)thio)-1-(tetrahydro-2 H-pyran-4-yl)propan-1-one 4i**

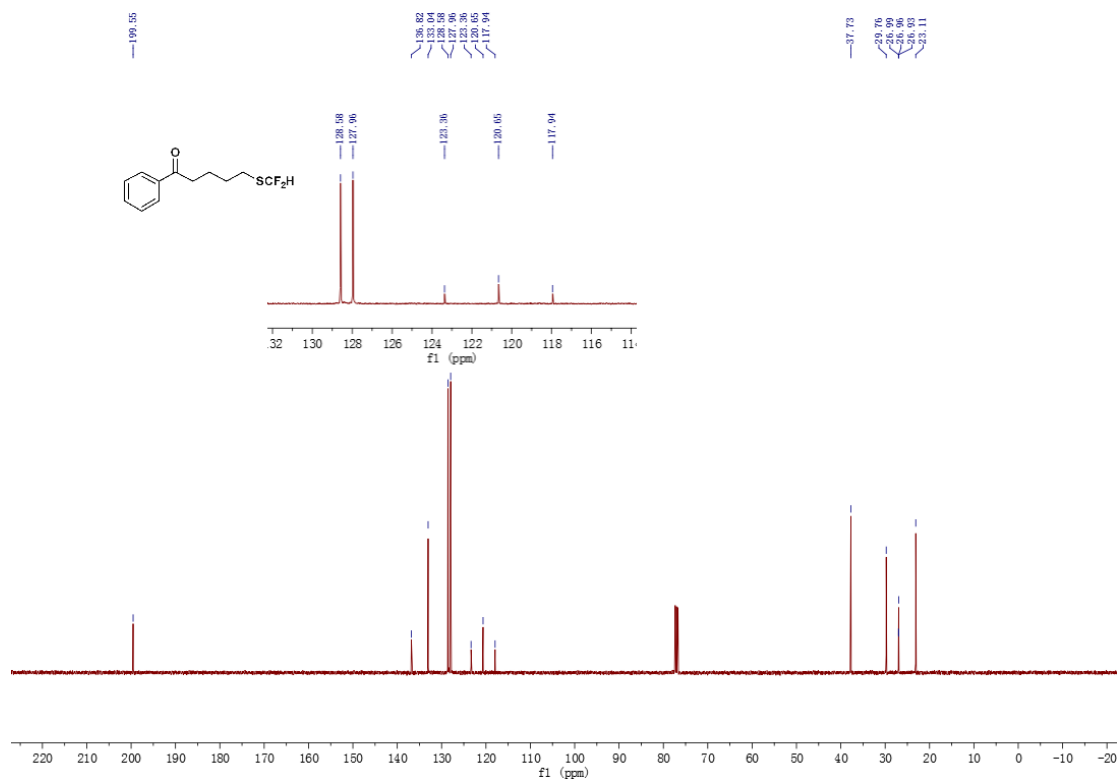


**^{13}C NMR spectrum of
3-((difluoromethyl)thio)-1-(tetrahydro-2 H-pyran-4-yl)propan-1-one 4i**

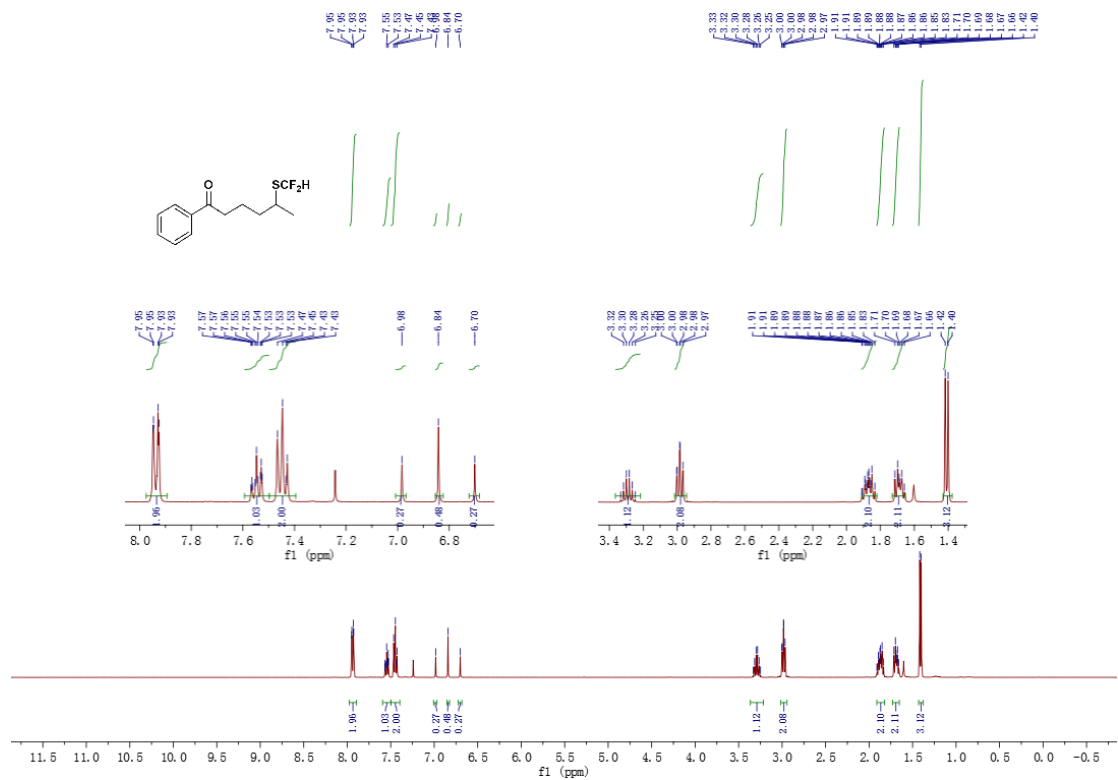


[illegible]O=C(CCCCCF)C1=CC=CC=C1

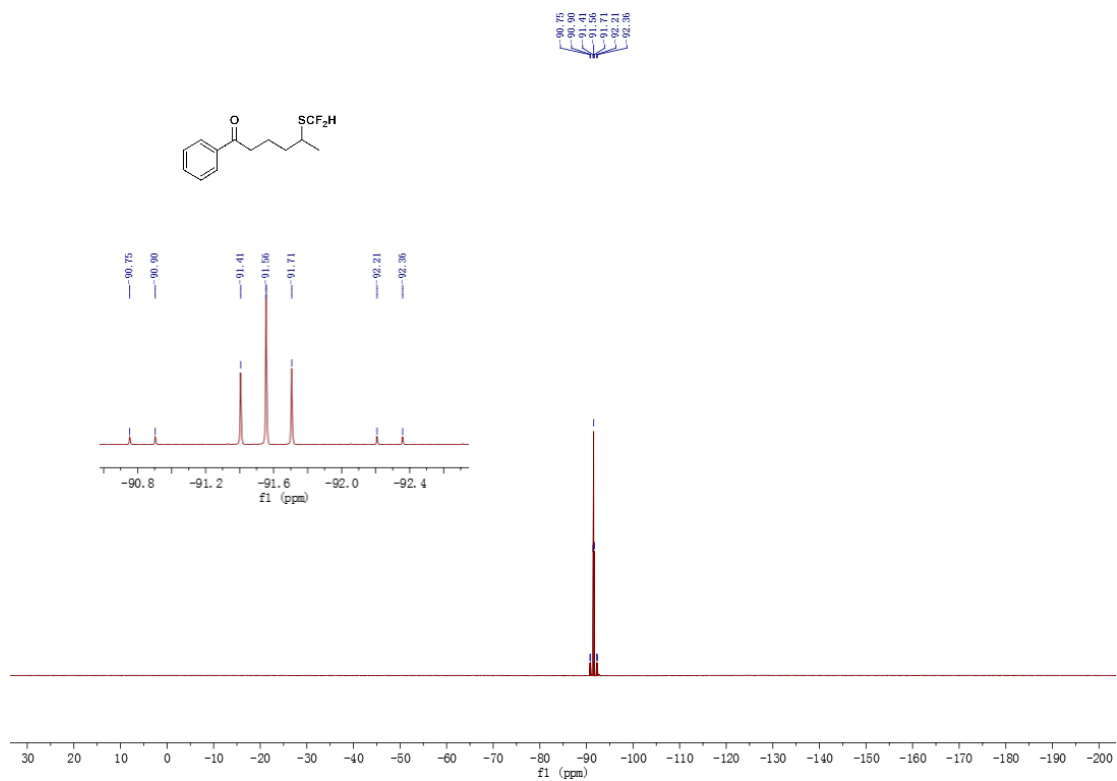
**^{13}C NMR spectrum of
5-((difluoromethyl)thio)-1-phenylpentan-1-one 4j**



**^1H NMR spectrum of
5-((difluoromethyl)thio)-1-phenylhexan-1-one 4k**



**^{19}F NMR spectrum of
5-((difluoromethyl)thio)-1-phenylhexan-1-one 4k**



**^{13}C NMR spectrum of
5-((difluoromethyl)thio)-1-phenylhexan-1-one 4k**

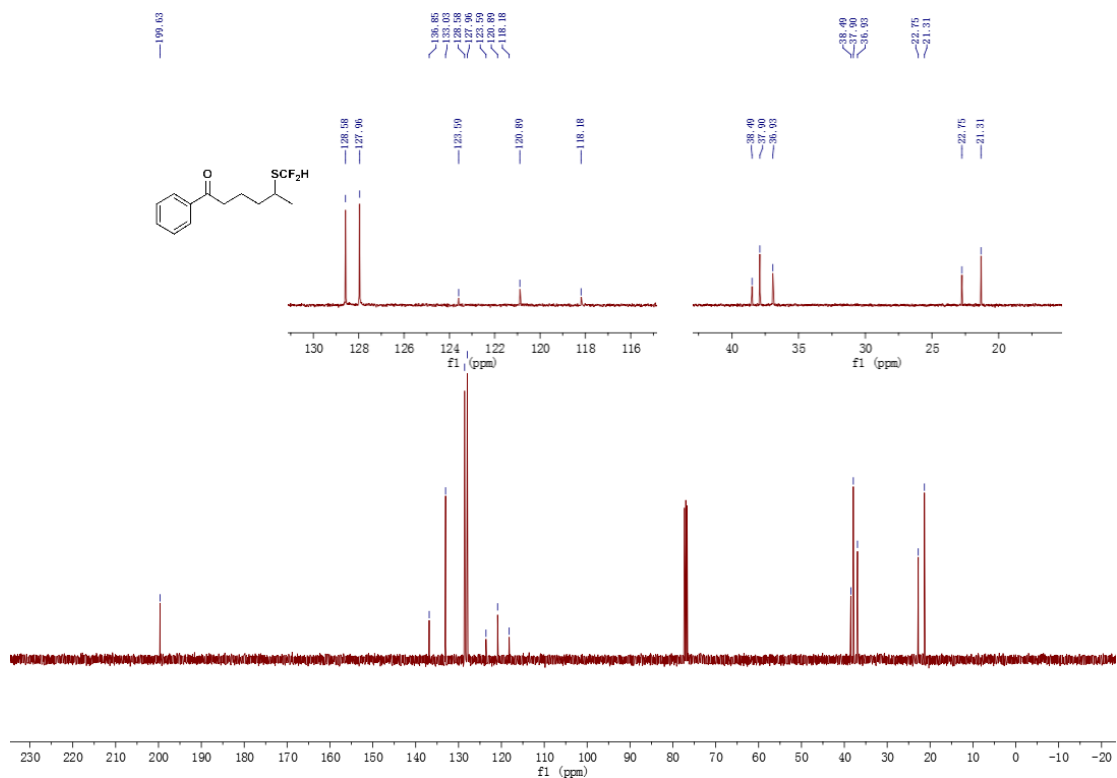
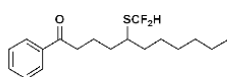
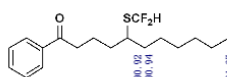


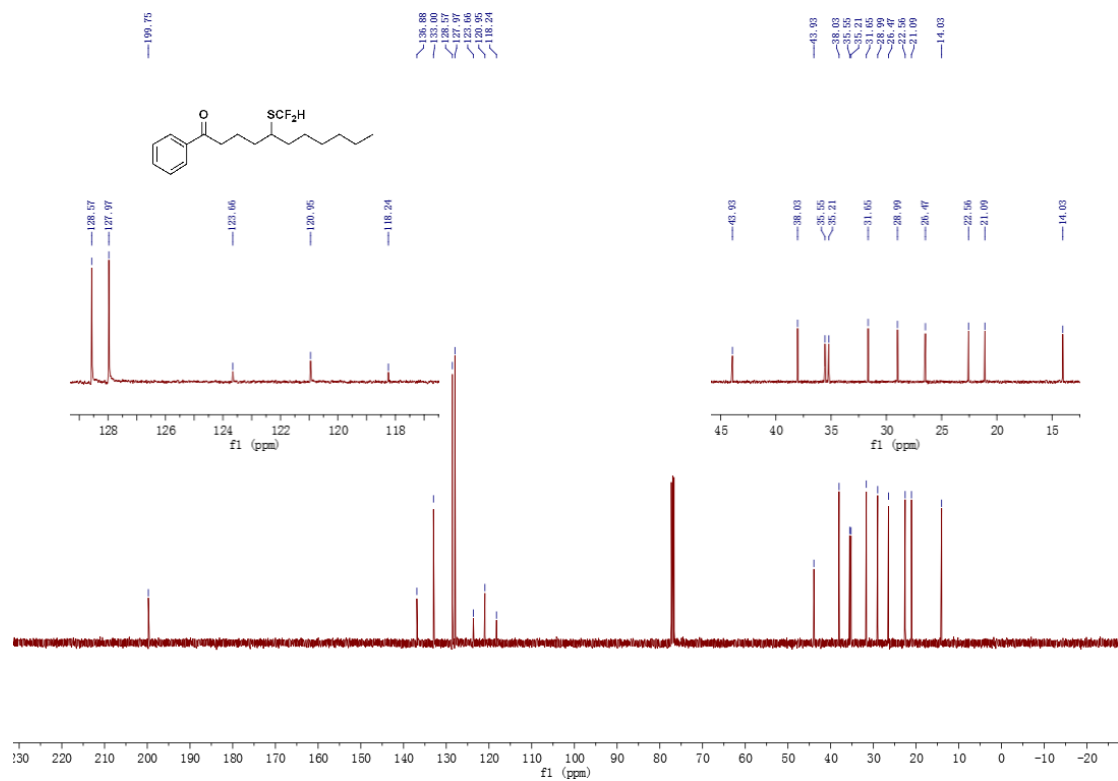
Figure 1 is a dendrogram illustrating the hierarchical clustering of 18 variables. The variables are grouped into three main clusters. The first cluster contains variables 1-10, the second contains 11-14, and the third contains 15-18. The dendrogram shows the distance between variables and the order in which they are merged.



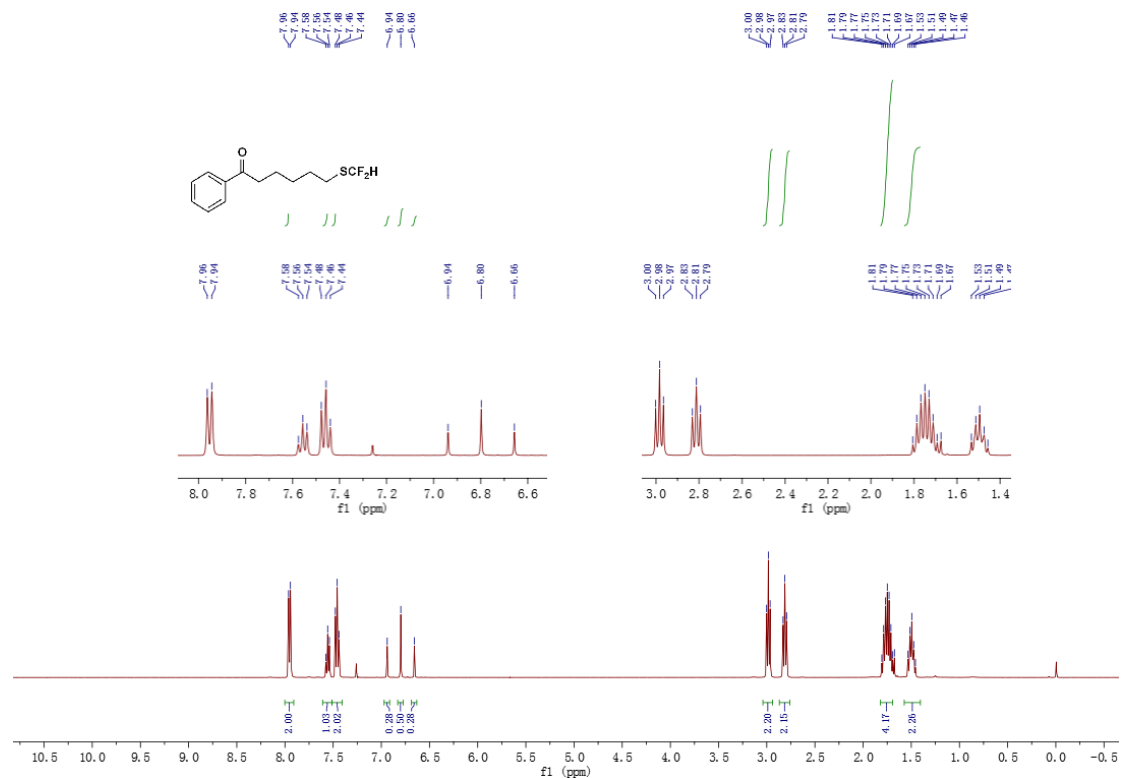
90.92
90.94
91.07
91.09



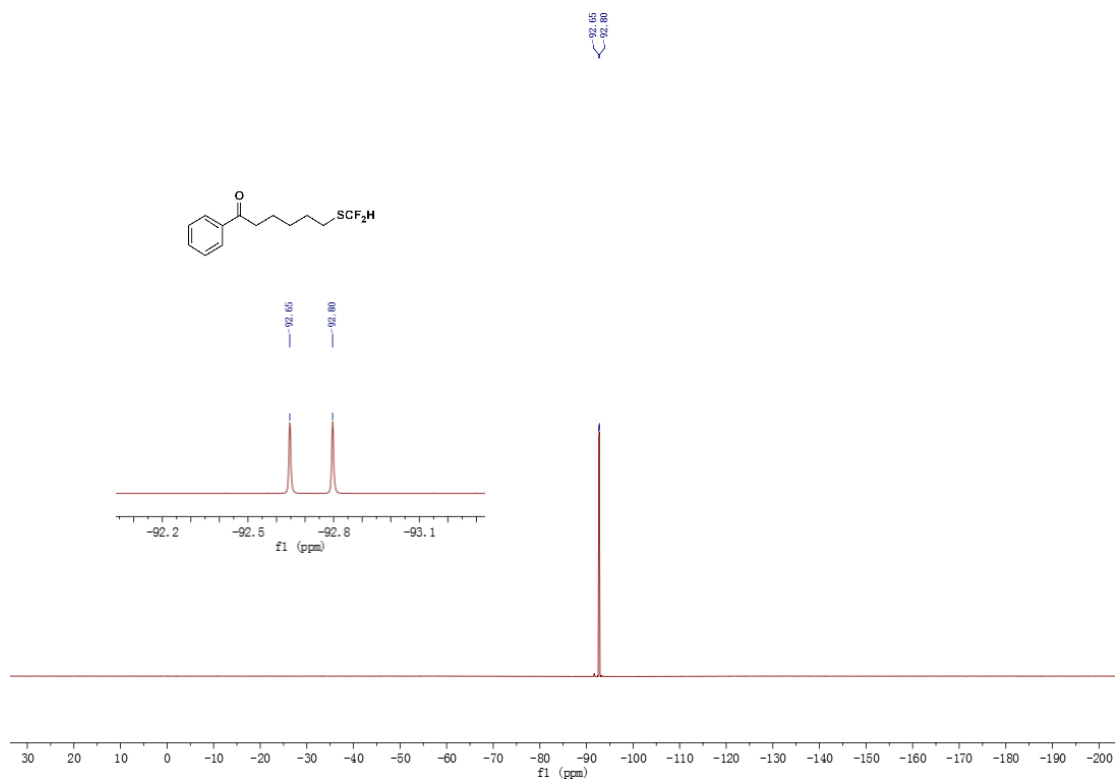
**^{13}C NMR spectrum of
5-((difluoromethyl)thio)-1-phenylundecan-1-one 4l**



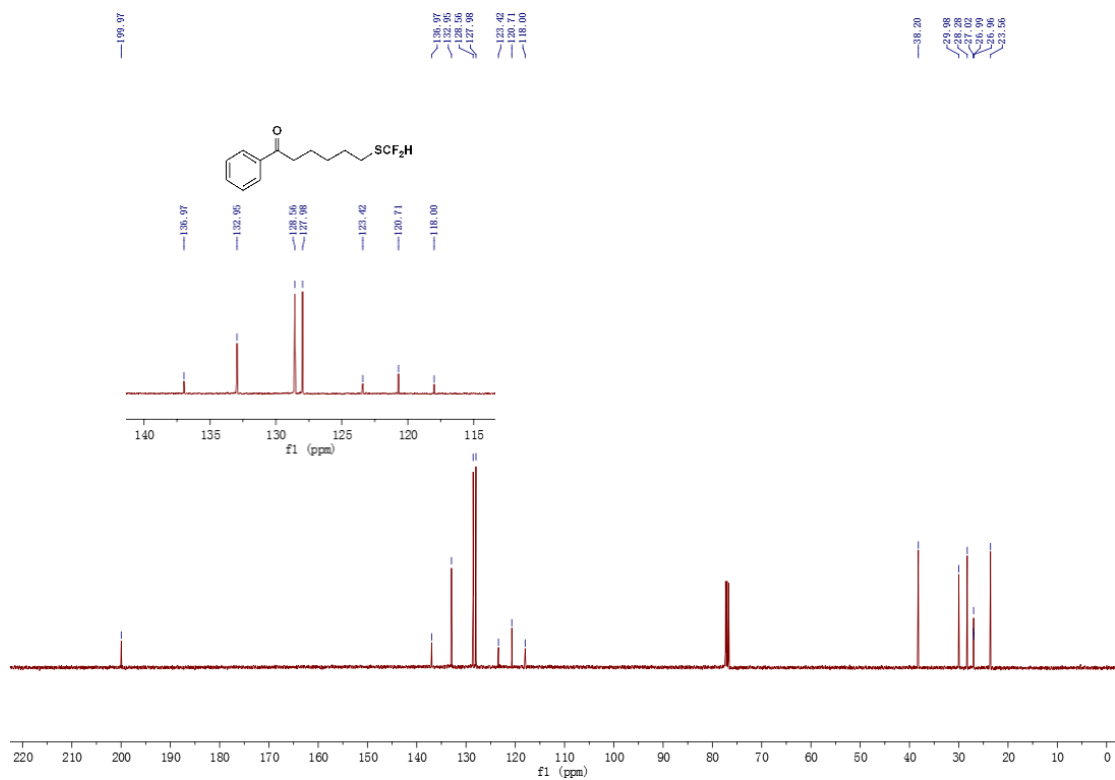
**^1H NMR spectrum of
6-((difluoromethyl)thio)-1-phenylhexan-1-one 4m**



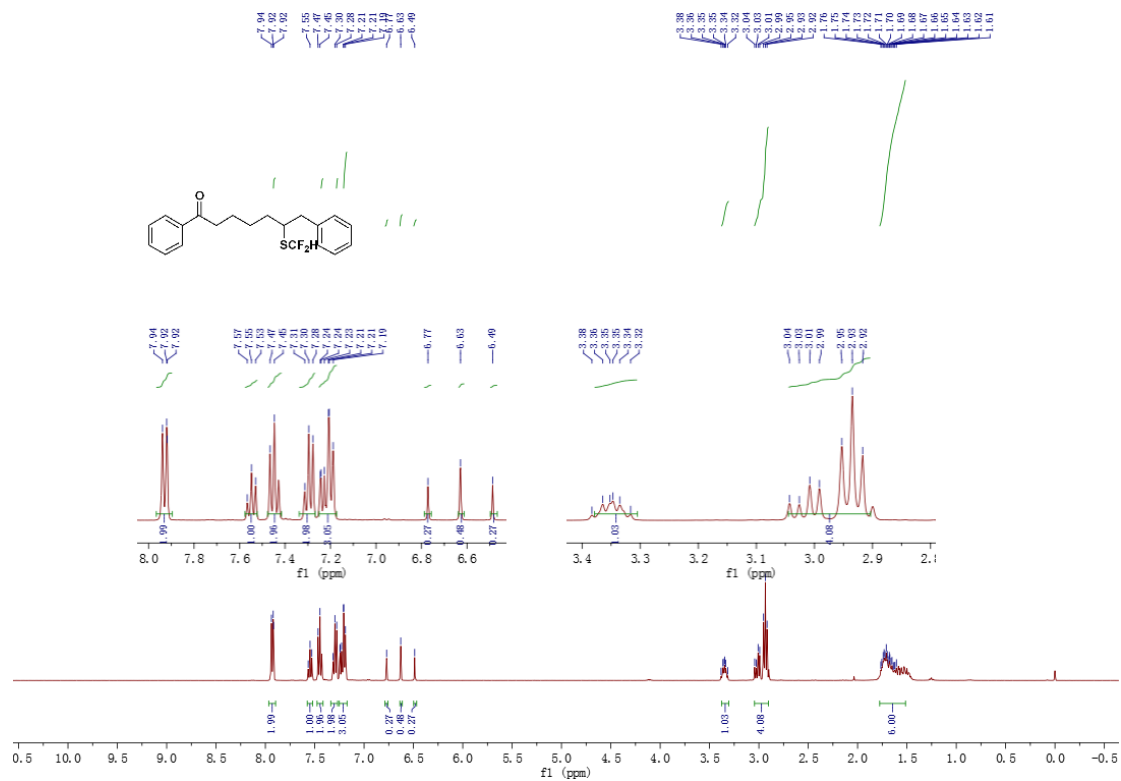
**^{19}F NMR spectrum of
6-((difluoromethyl)thio)-1-phenylhexan-1-one 4m**



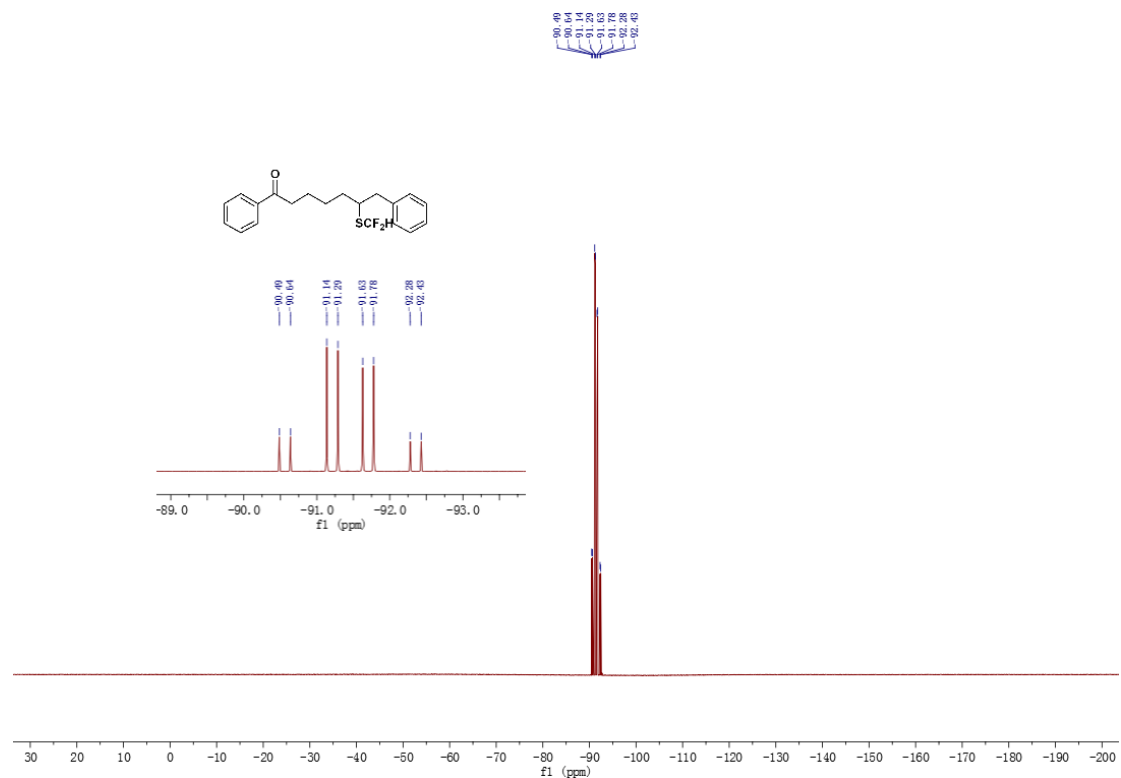
**^{13}C NMR spectrum of
6-((difluoromethyl)thio)-1-phenylhexan-1-one 4m**



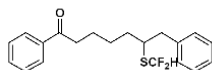
**¹H NMR spectrum of
6-((difluoromethyl)thio)-1,7-diphenylheptan-1-one 4n**



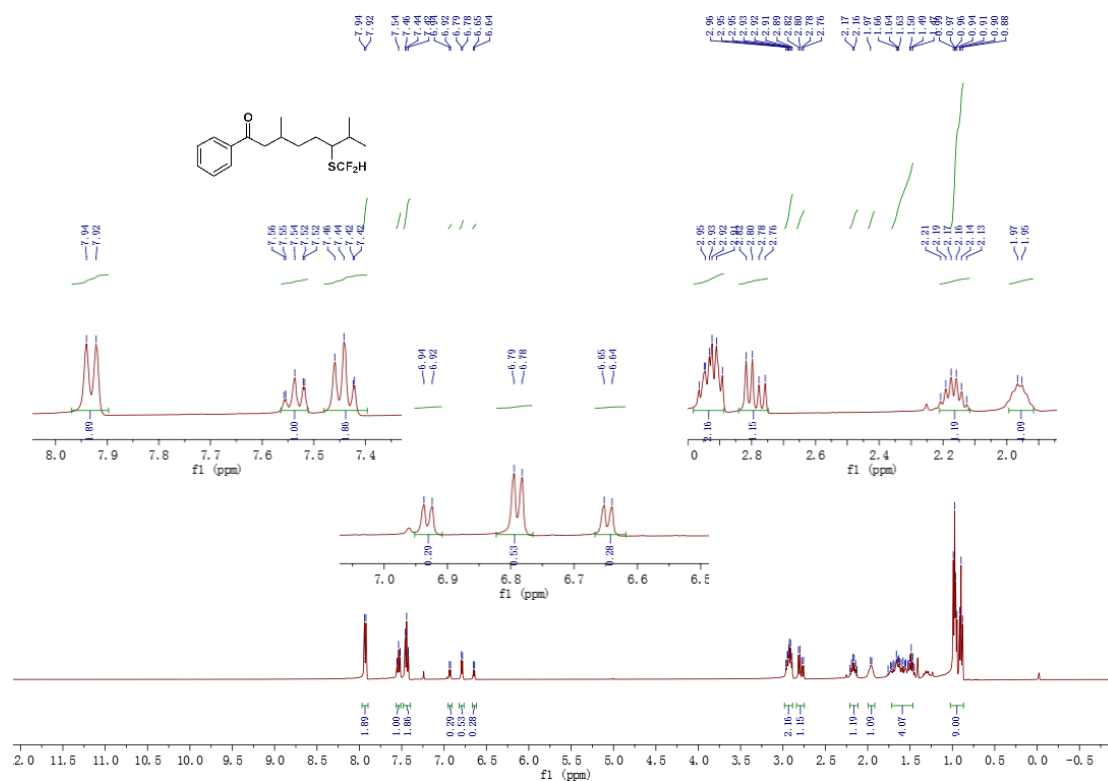
**¹⁹F NMR spectrum of
6-((difluoromethyl)thio)-1,7-diphenylheptan-1-one 4n**



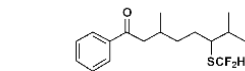
—200.01



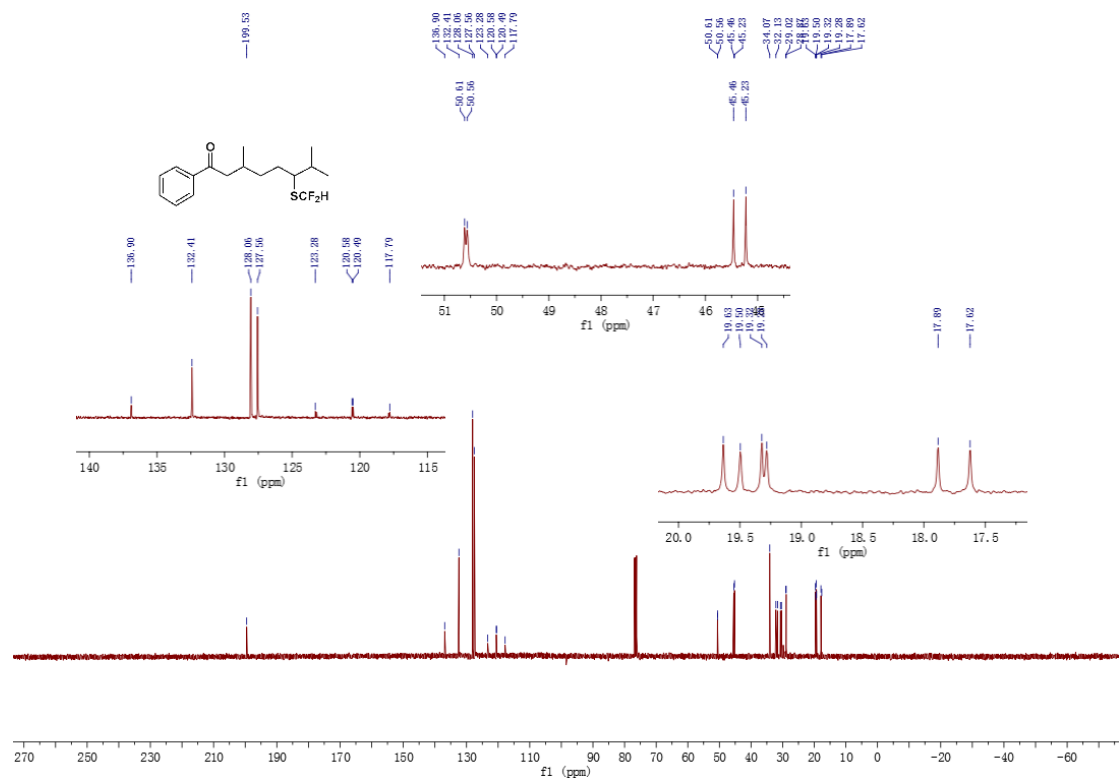
7.94

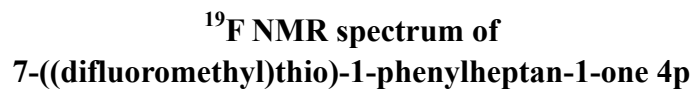


90.26
90.27
90.38
90.42
90.43
90.53

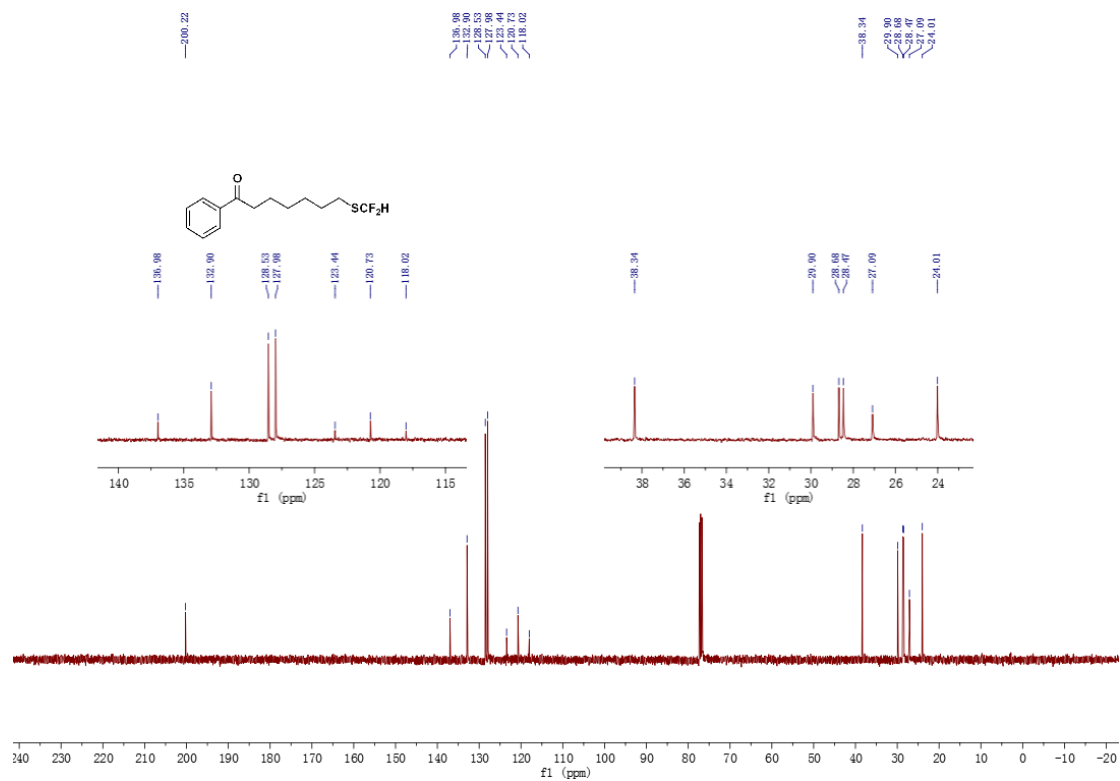


136.90	50.61
132.41	50.56
128.06	45.46
127.56	45.23
123.28	
120.58	
120.49	
117.79	
	50.61
	50.56
	45.46
	45.23
	34.07
	32.13
	29.02
	28.83
	19.50
	19.32
	19.28
	17.89
	17.62

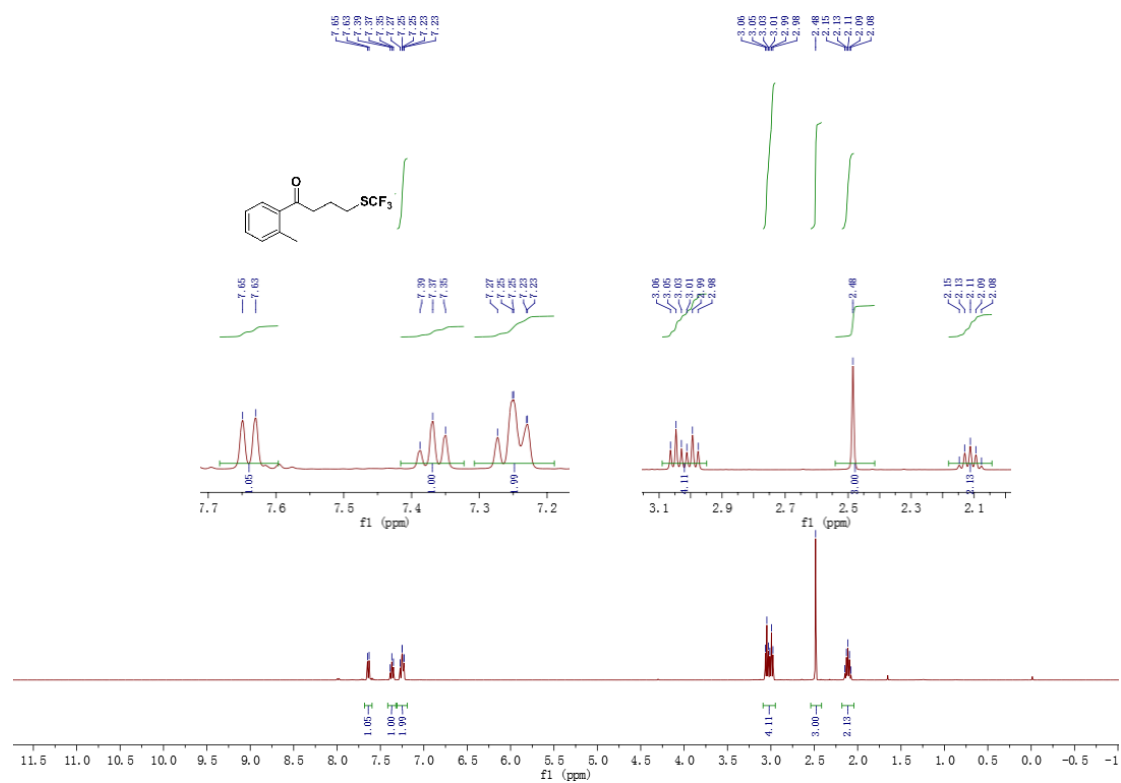


[illegible]

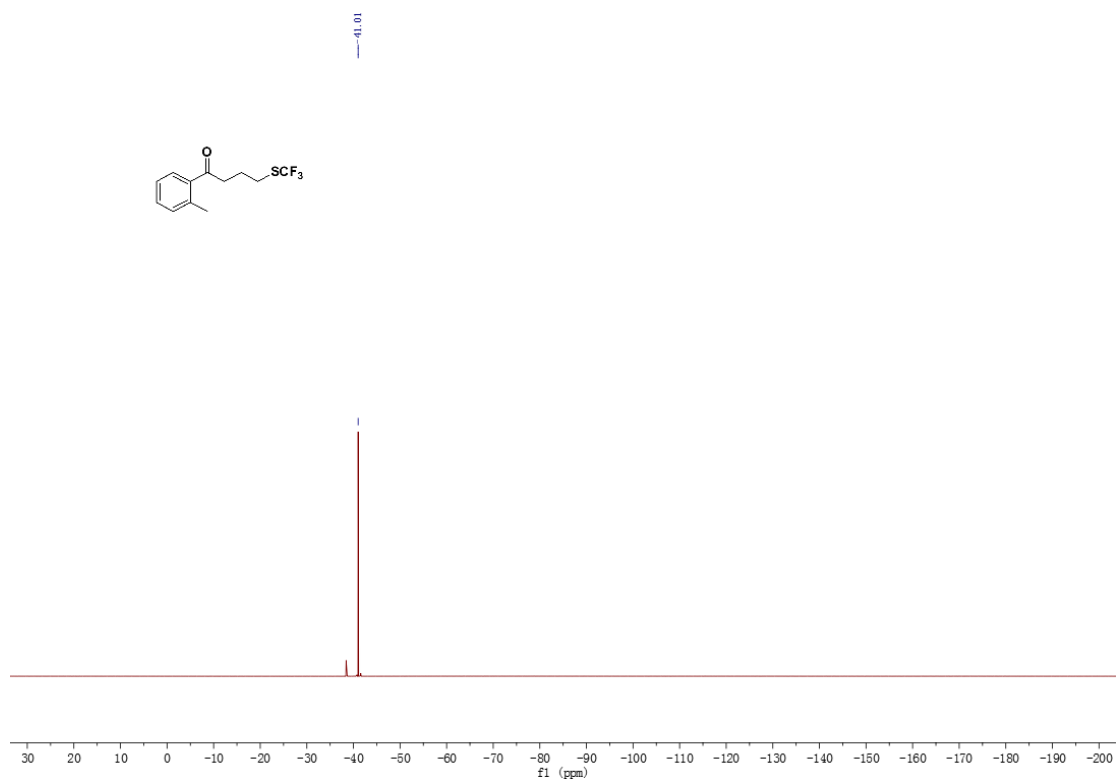
**^{13}C NMR spectrum of
7-((difluoromethyl)thio)-1-phenylheptan-1-one 4p**



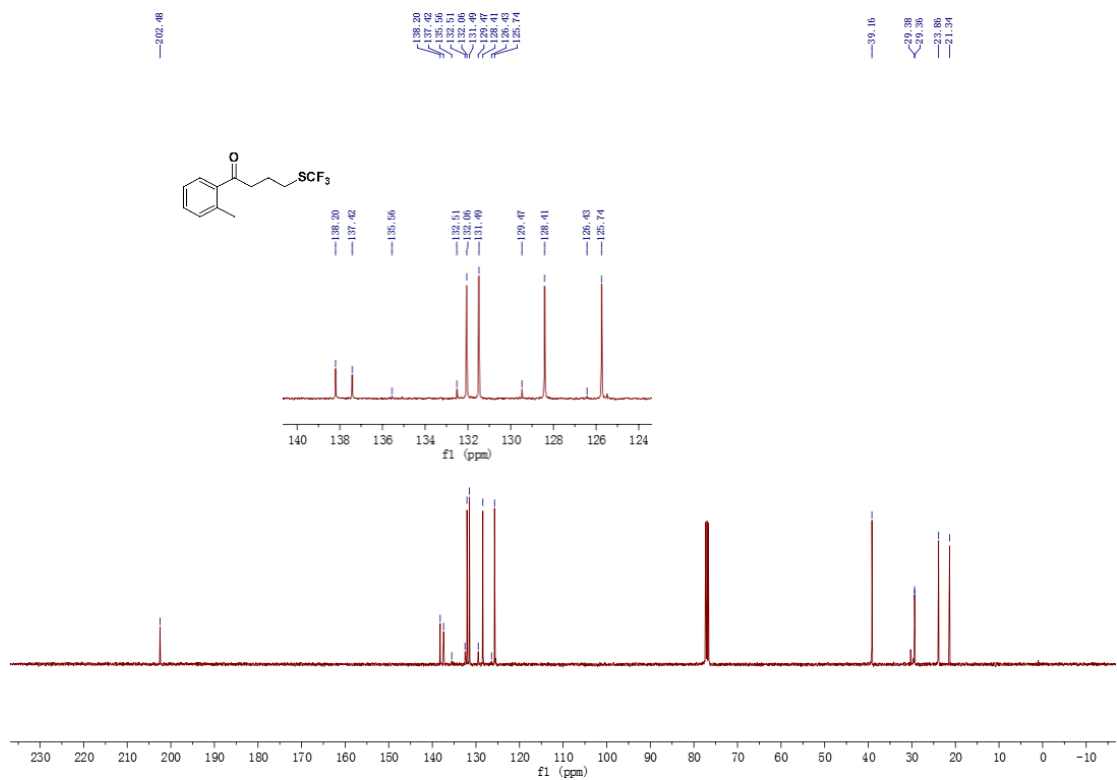
**^1H NMR spectrum of
1-(*o*-tolyl)-4-((trifluoromethyl)thio)butan-1-one 5a**



**^{19}F NMR spectrum of
1-(*o*-tolyl)-4-((trifluoromethyl)thio)butan-1-one 5a**



**^{13}C NMR spectrum of
1-(*o*-tolyl)-4-((trifluoromethyl)thio)butan-1-one 5a**

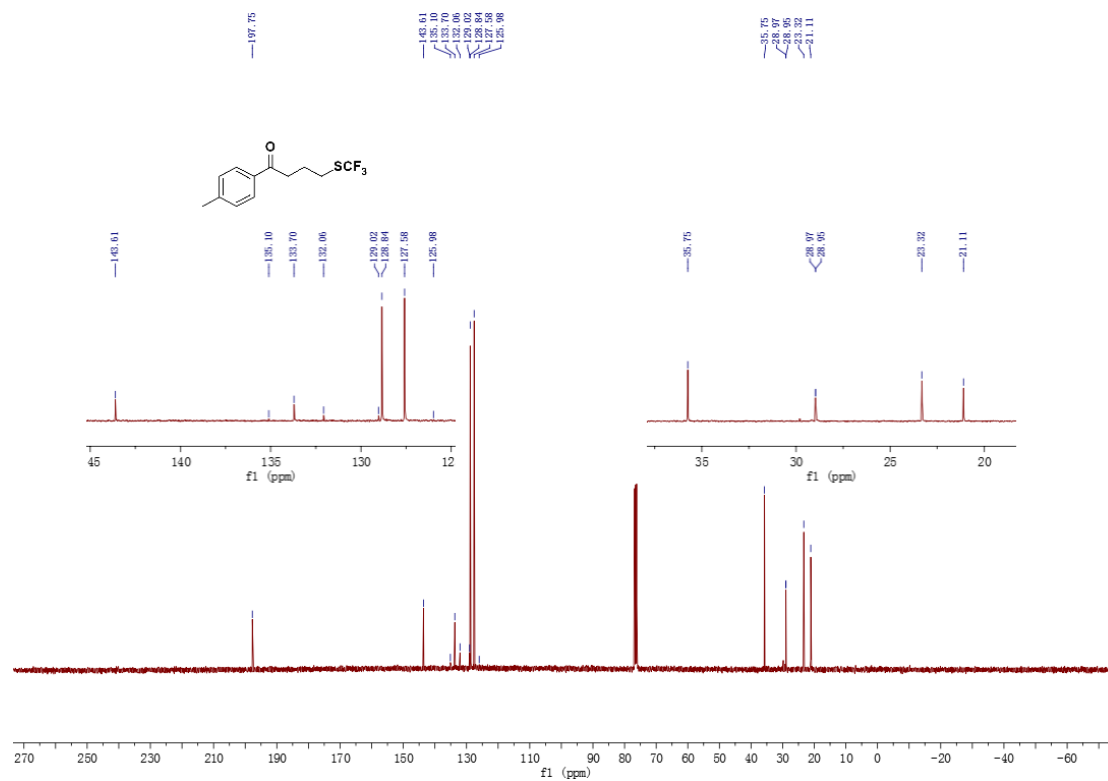


$$\begin{array}{r} 7.85 \\ - 7.83 \\ \hline \end{array} \qquad \begin{array}{r} 7.26 \\ - 7.24 \\ \hline \end{array}$$

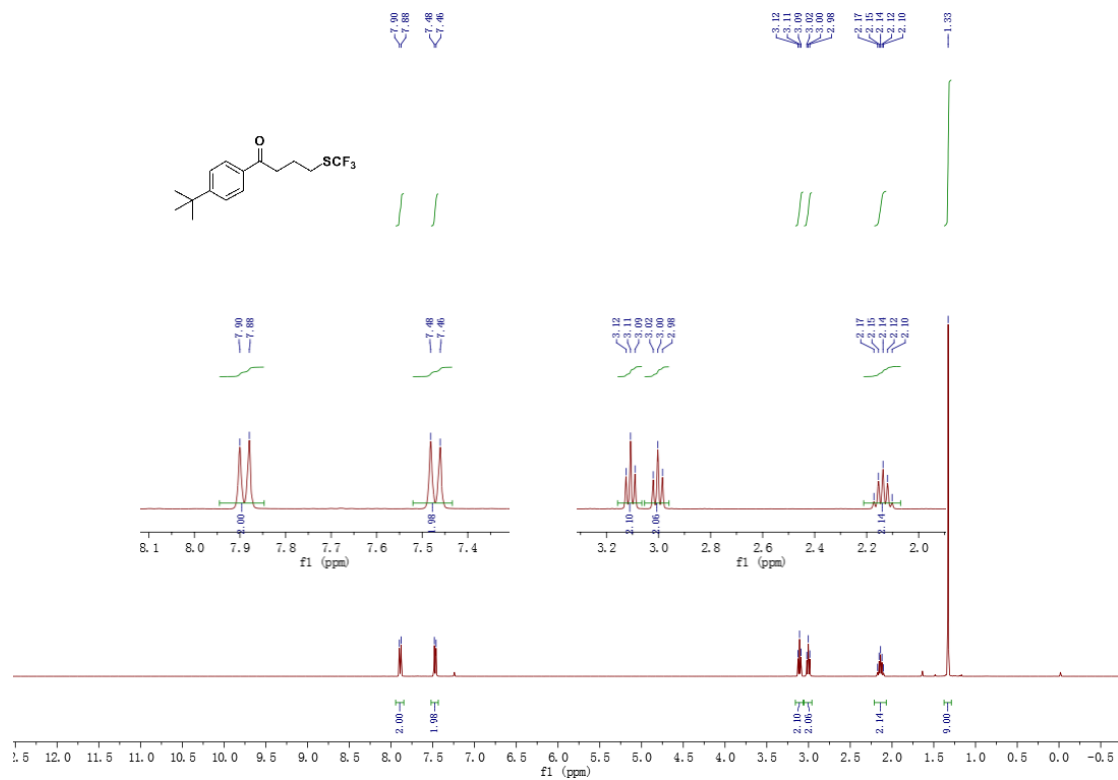

—40.99



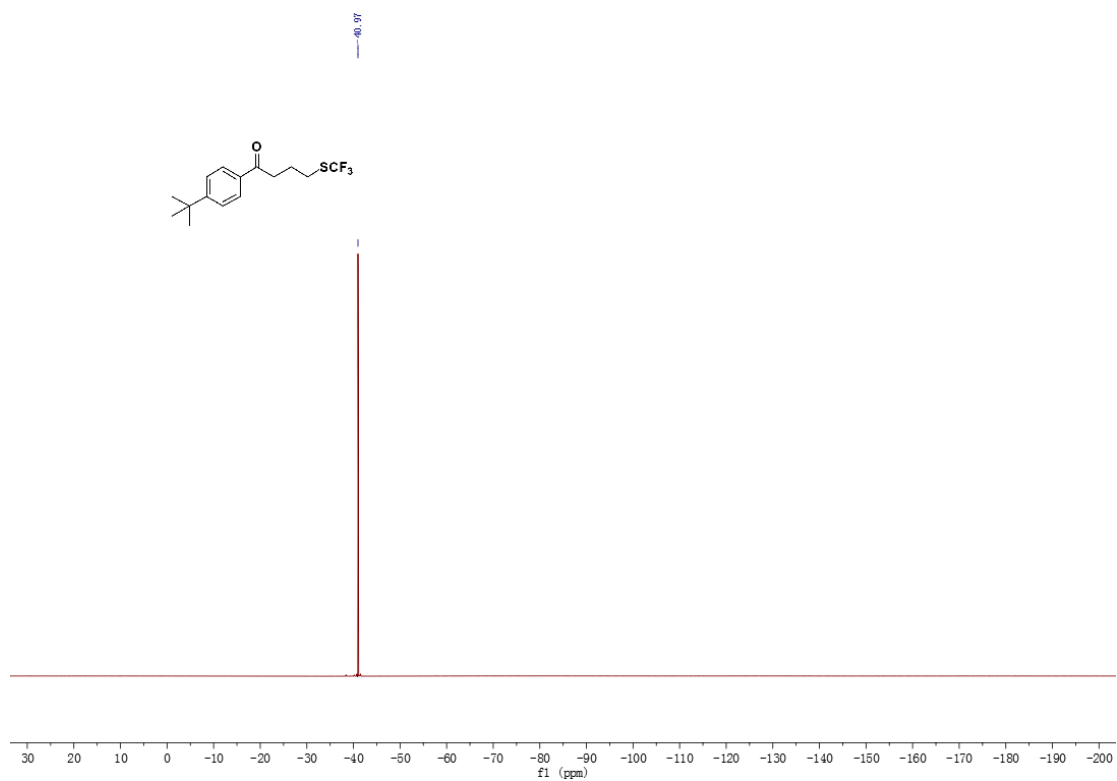
**^{13}C NMR spectrum of
1-(*p*-tolyl)-4-((trifluoromethyl)thio)butan-1-one 5b**



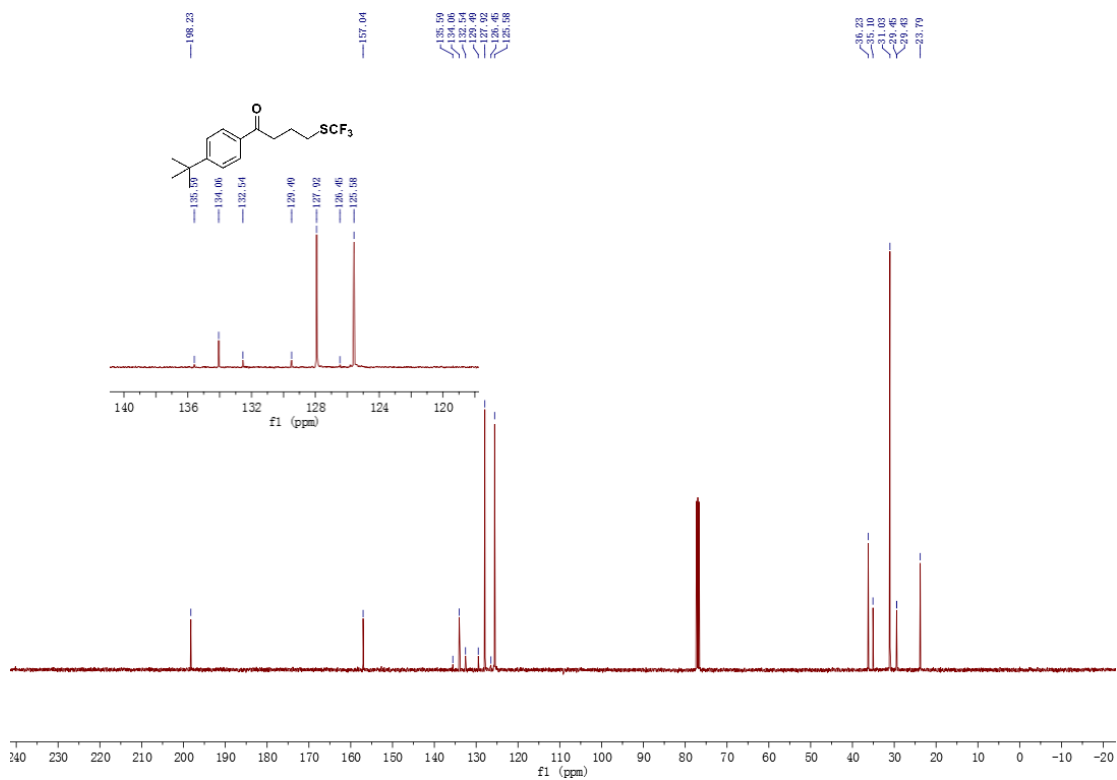
**^1H NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((trifluoromethyl)thio)butan-1-one 5c**



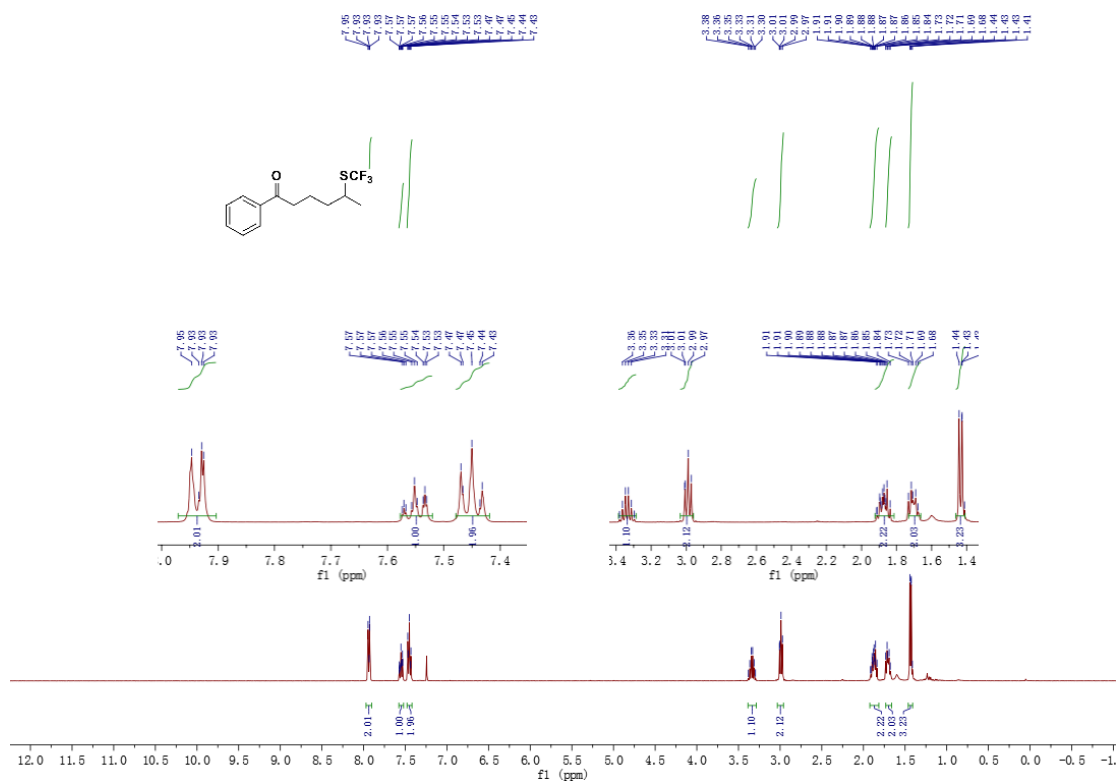
**^{19}F NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((trifluoromethyl)thio)butan-1-one 5c**



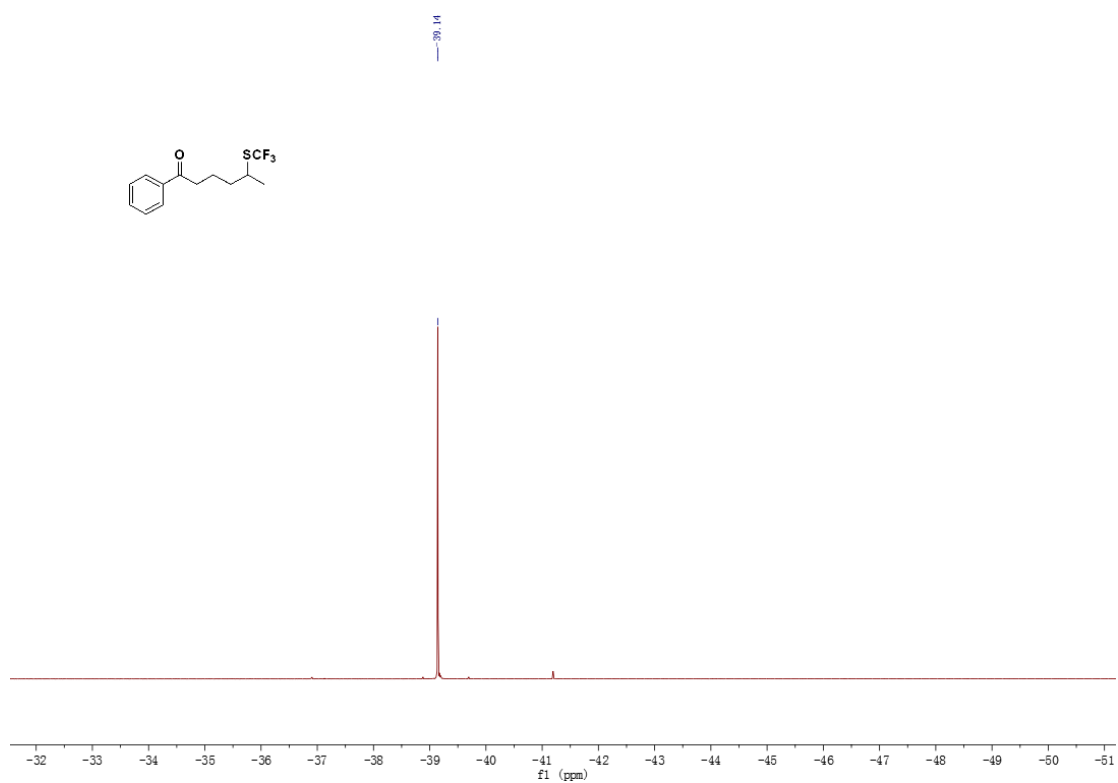
**^{13}C NMR spectrum of
1-(4-(*tert*-butyl)phenyl)-4-((trifluoromethyl)thio)butan-1-one 5c**



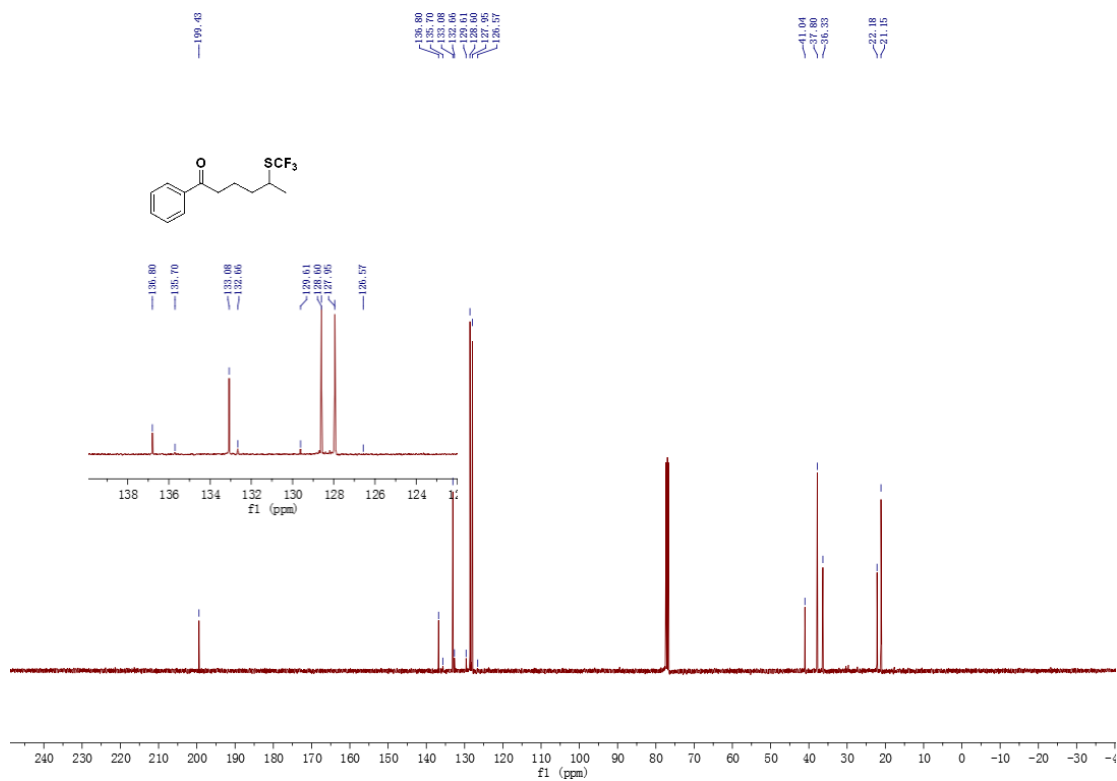
**^1H NMR spectrum of
1-phenyl-5-((trifluoromethyl)thio)hexan-1-one 5d**



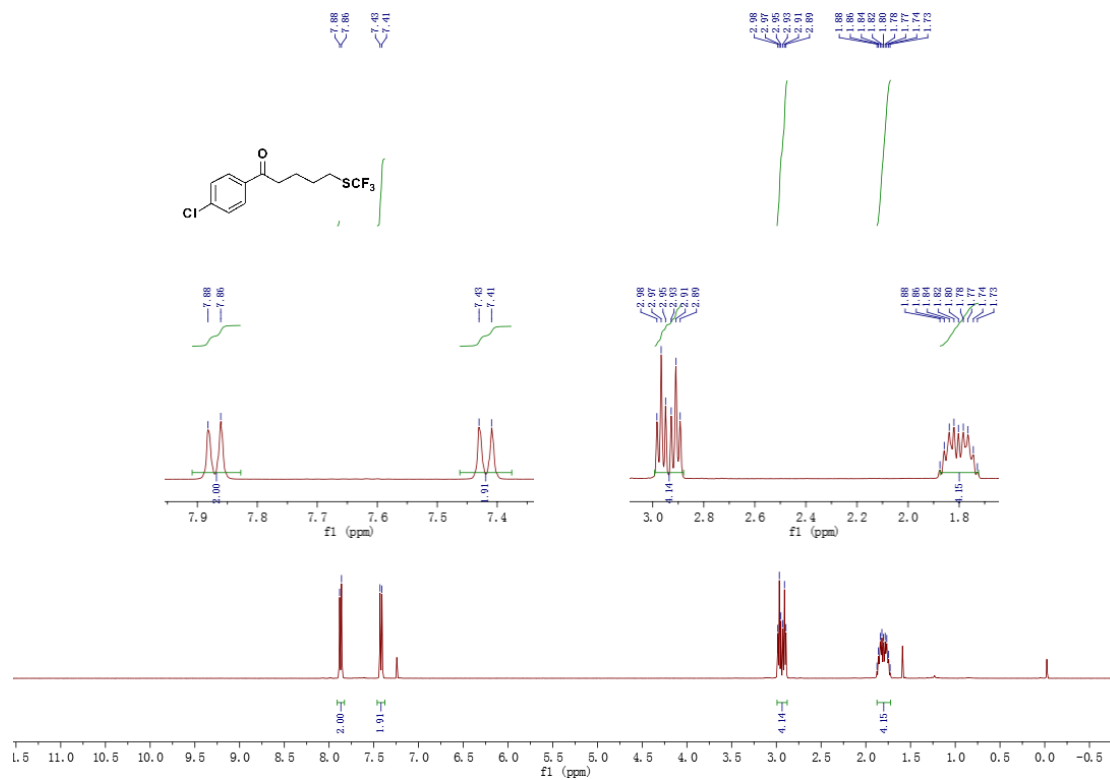
**^{19}F NMR spectrum of
1-phenyl-5-((trifluoromethyl)thio)hexan-1-one 5d**



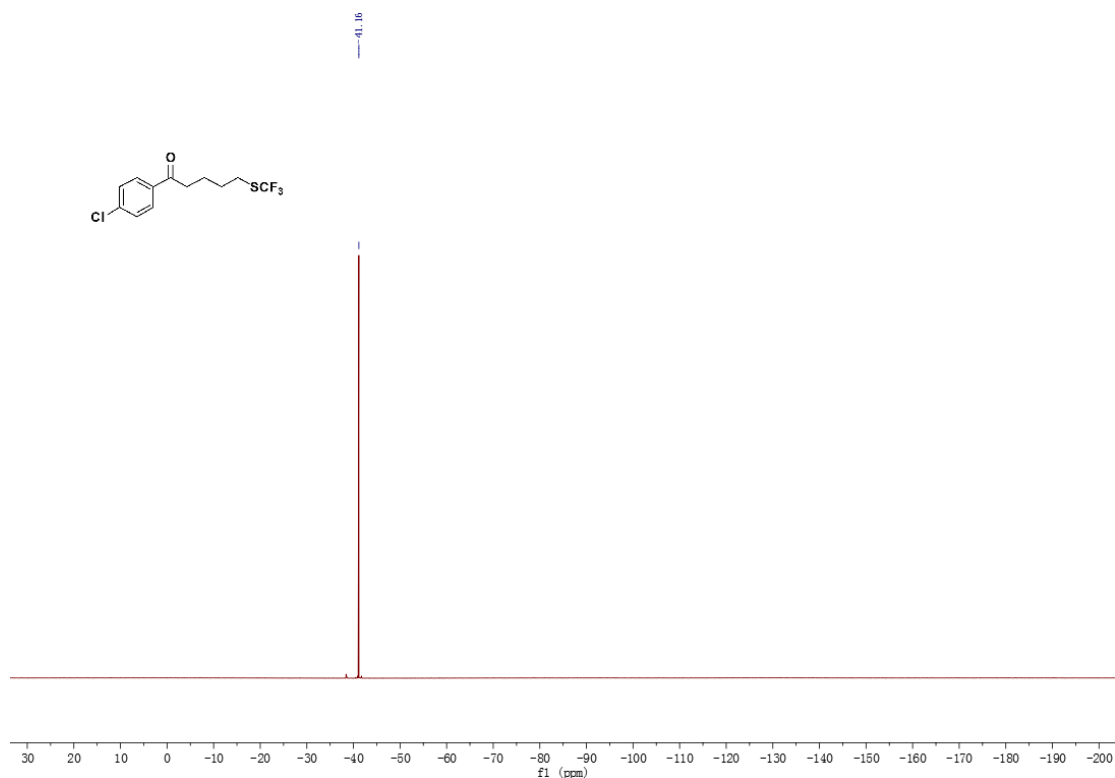
**^{13}C NMR spectrum of
1-phenyl-5-((trifluoromethyl)thio)hexan-1-one 5d**



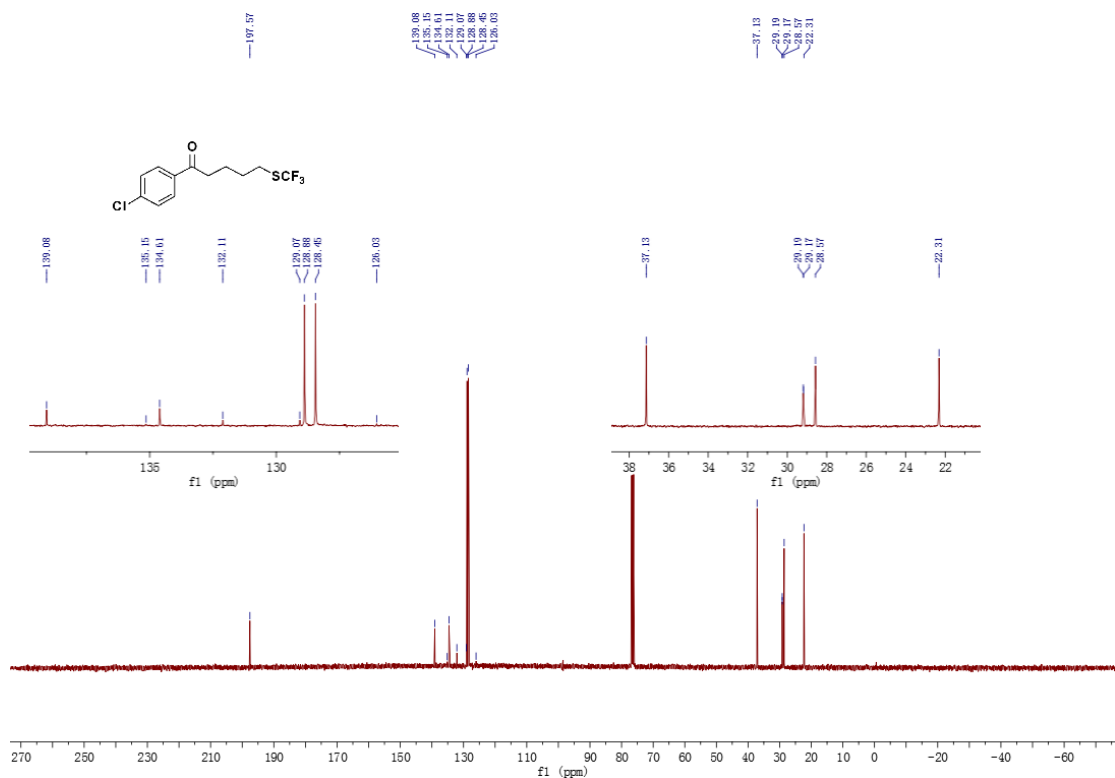
**^1H NMR spectrum of
1-(4-chlorophenyl)-5-((trifluoromethyl)thio)pentan-1-one 5e**



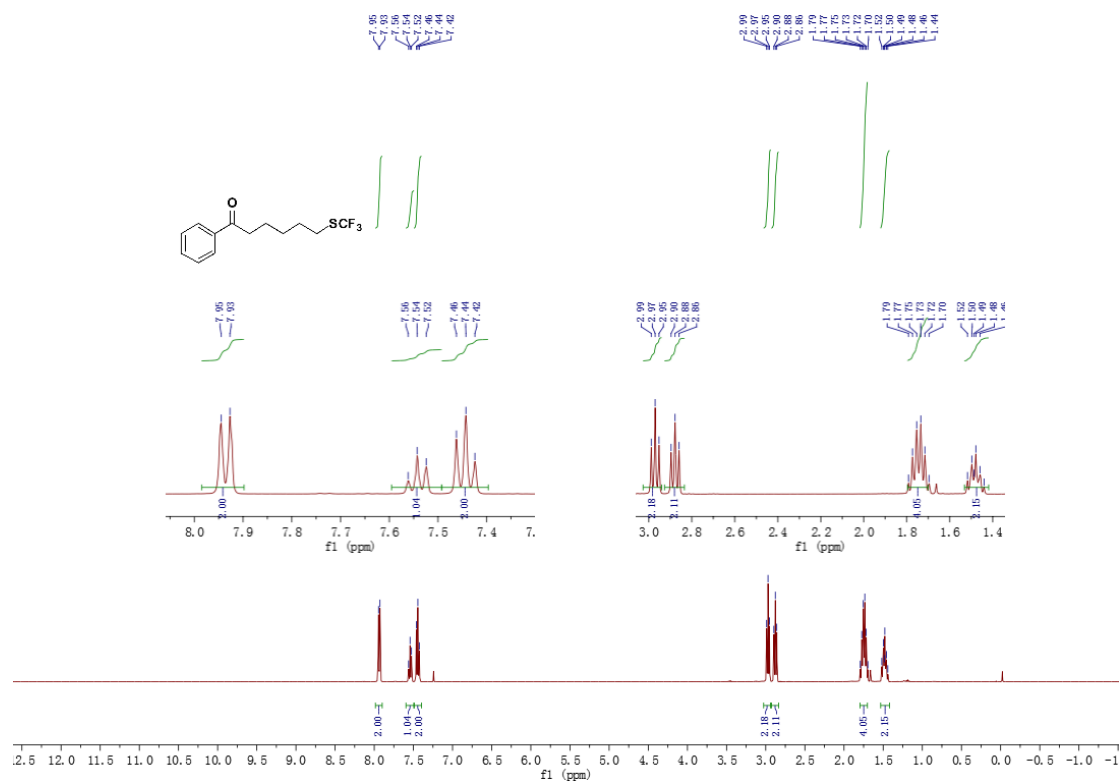
**^{19}F NMR spectrum of
1-(4-chlorophenyl)-5-((trifluoromethyl)thio)pentan-1-one 5e**



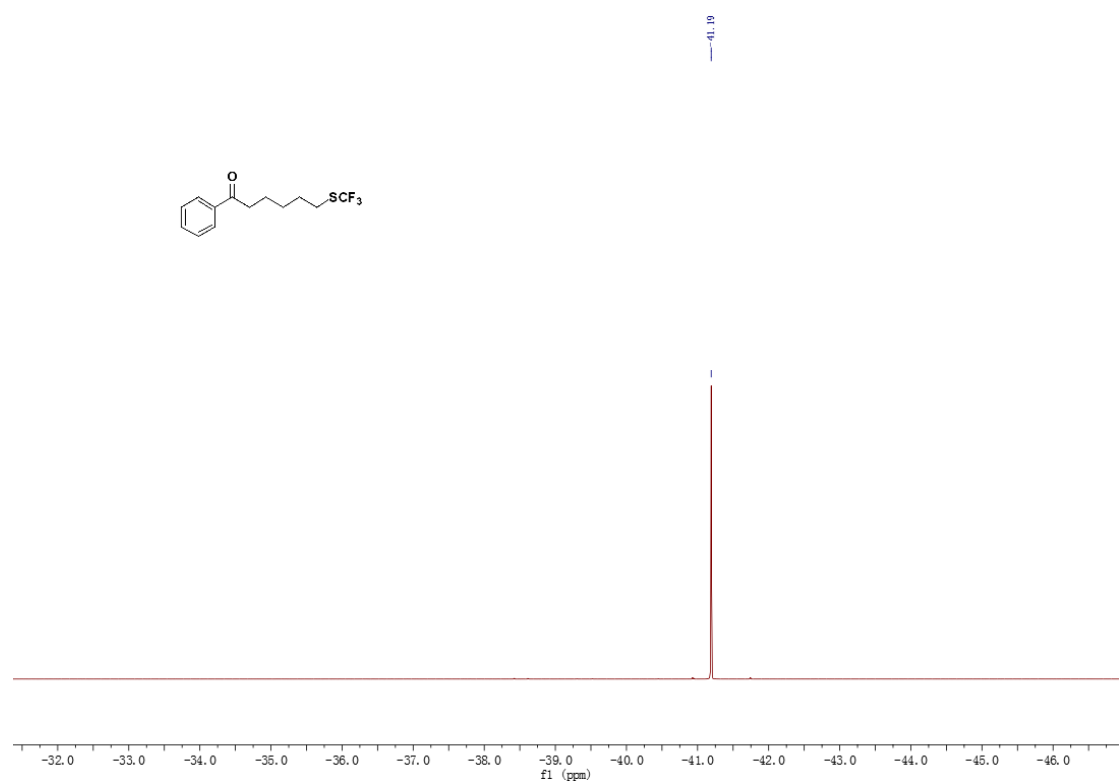
**^{13}C NMR spectrum of
1-(4-chlorophenyl)-5-((trifluoromethyl)thio)pentan-1-one 5e**



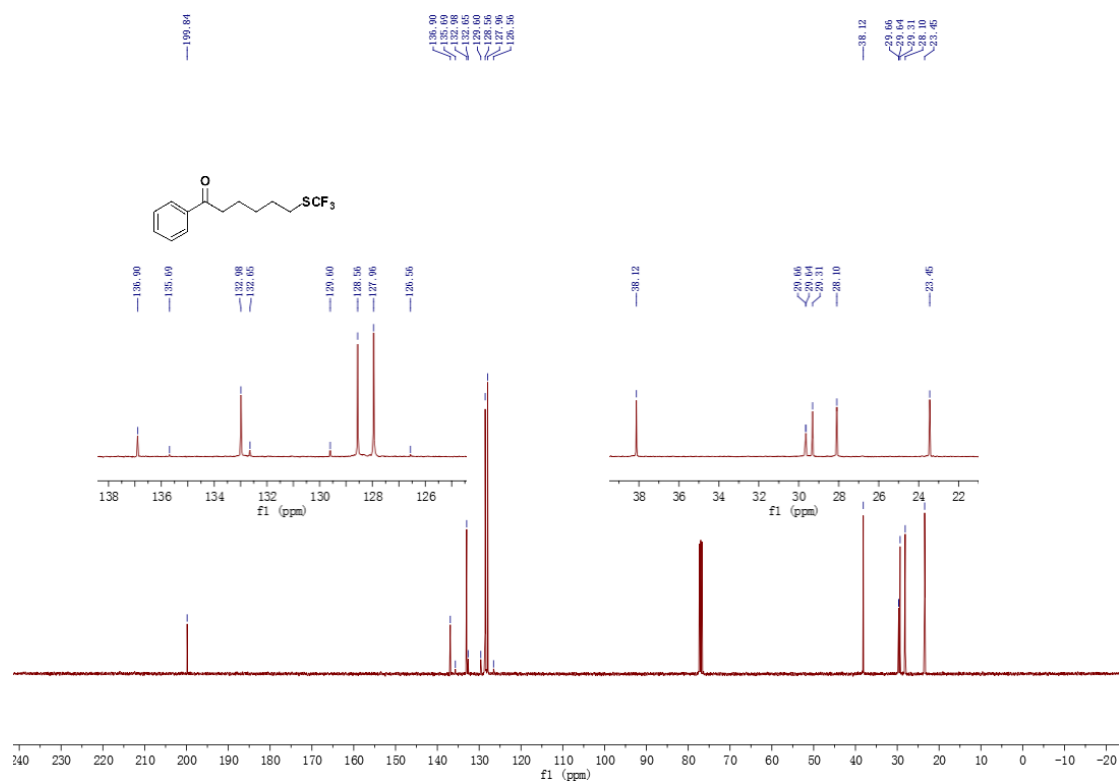
**¹H NMR spectrum of
1-phenyl-6-((trifluoromethyl)thio)hexan-1-one 5f**



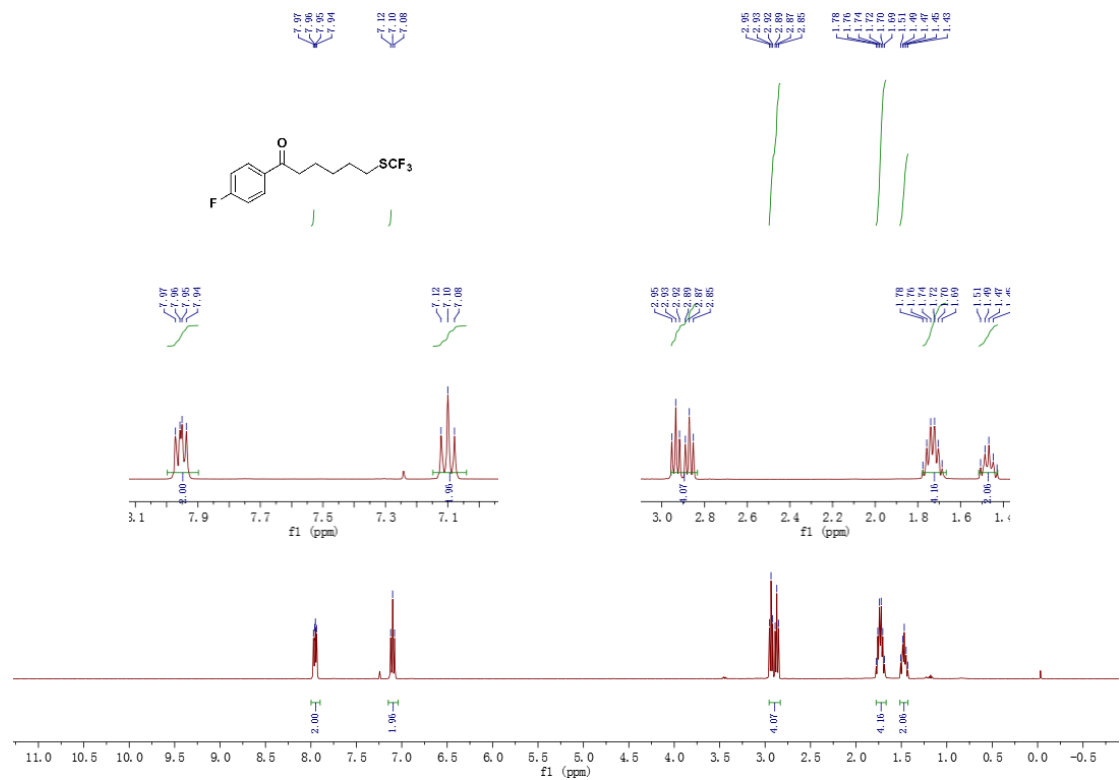
**¹⁹F NMR spectrum of
1-phenyl-6-((trifluoromethyl)thio)hexan-1-one 5f**



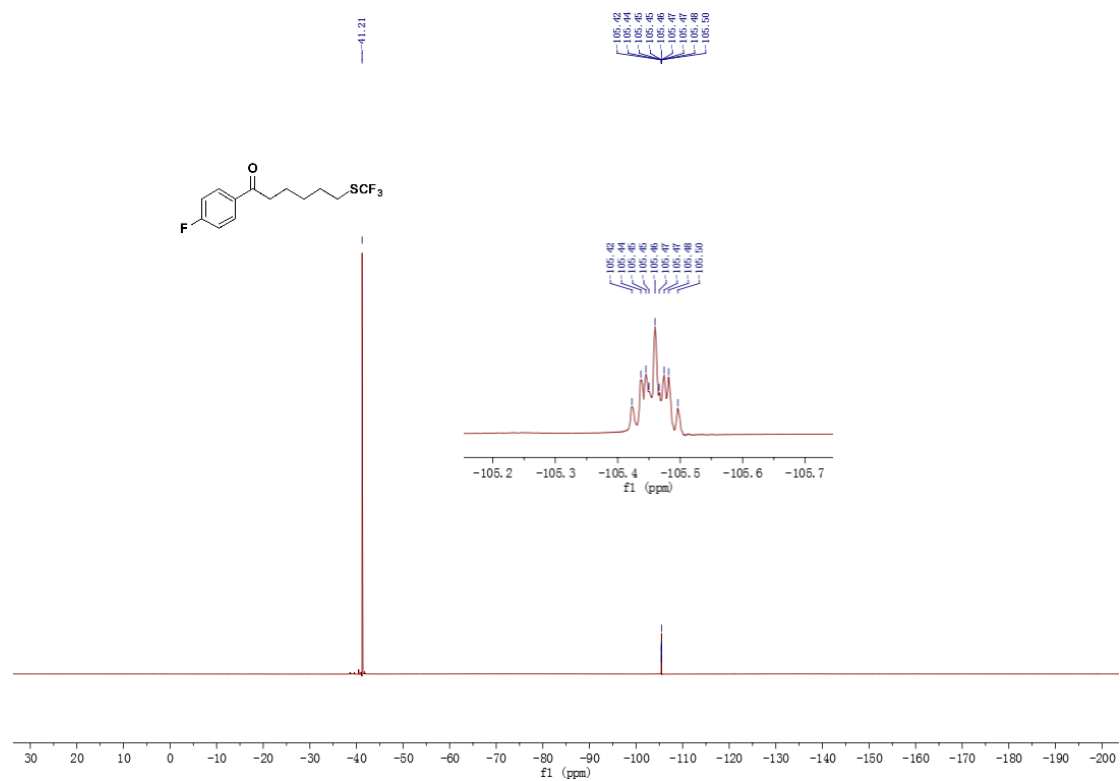
**^{13}C NMR spectrum of
1-phenyl-6-((trifluoromethyl)thio)hexan-1-one 5f**



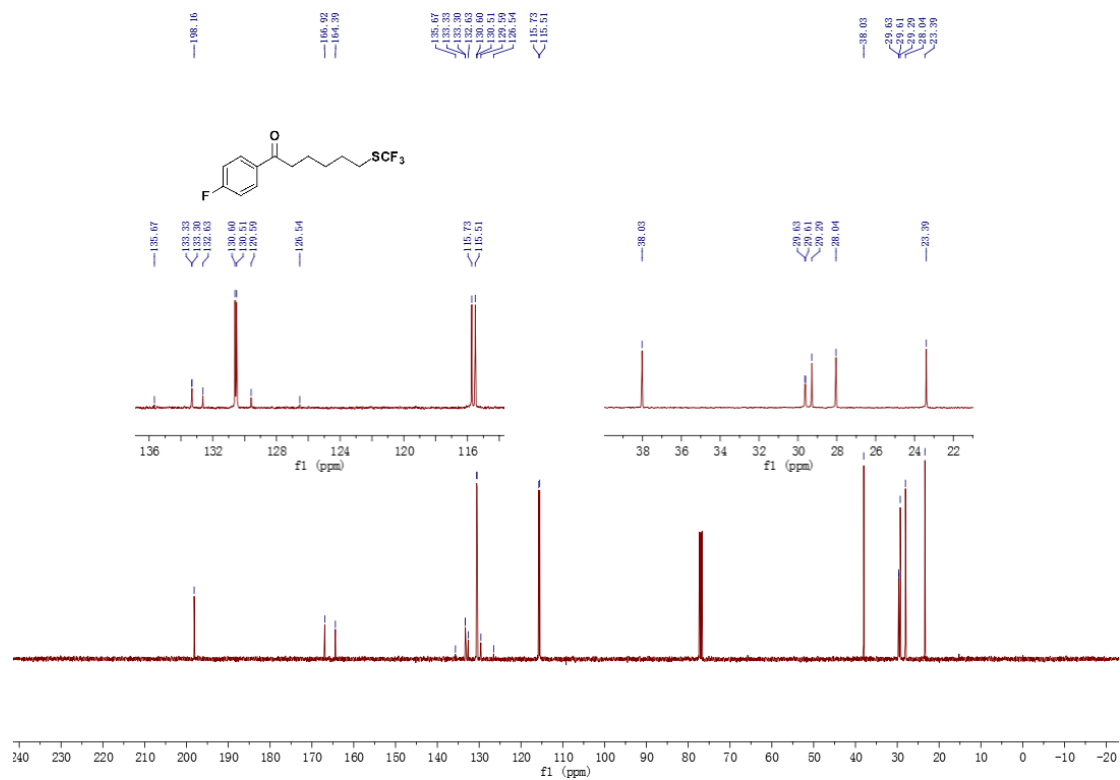
**^1H NMR spectrum of
1-(4-fluorophenyl)-6-((trifluoromethyl)thio)hexan-1-one 5g**



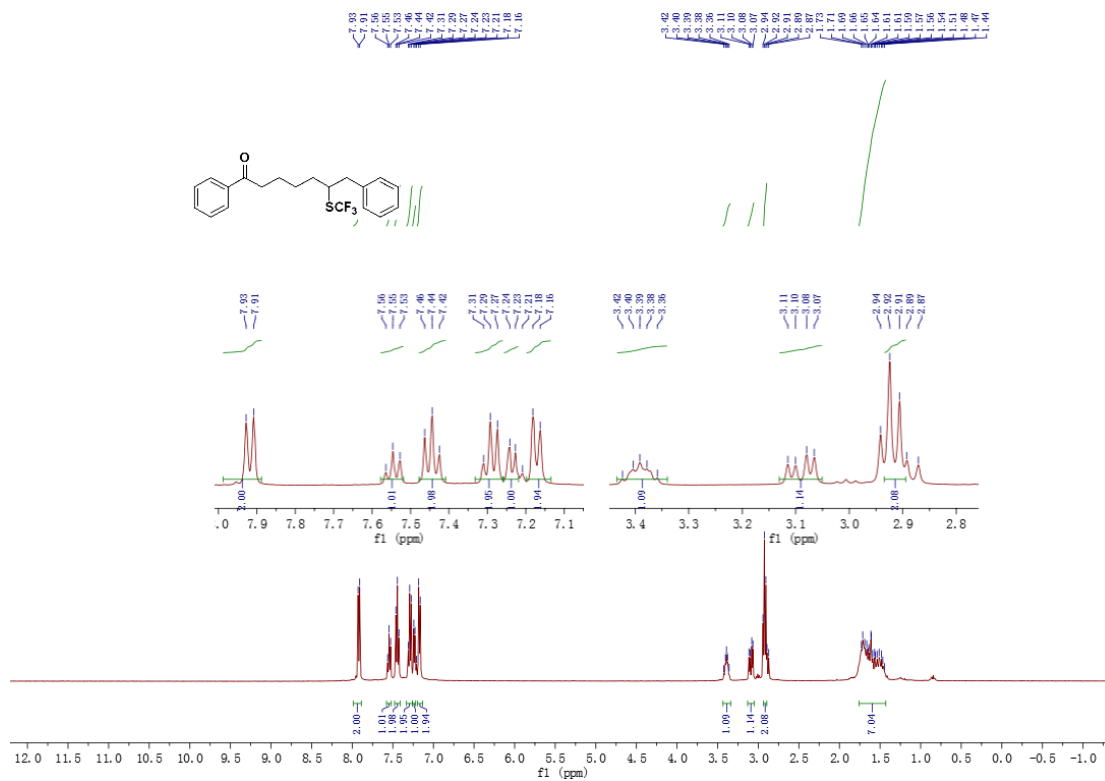
**^{19}F NMR spectrum of
1-(4-fluorophenyl)-6-((trifluoromethyl)thio)hexan-1-one 5g**



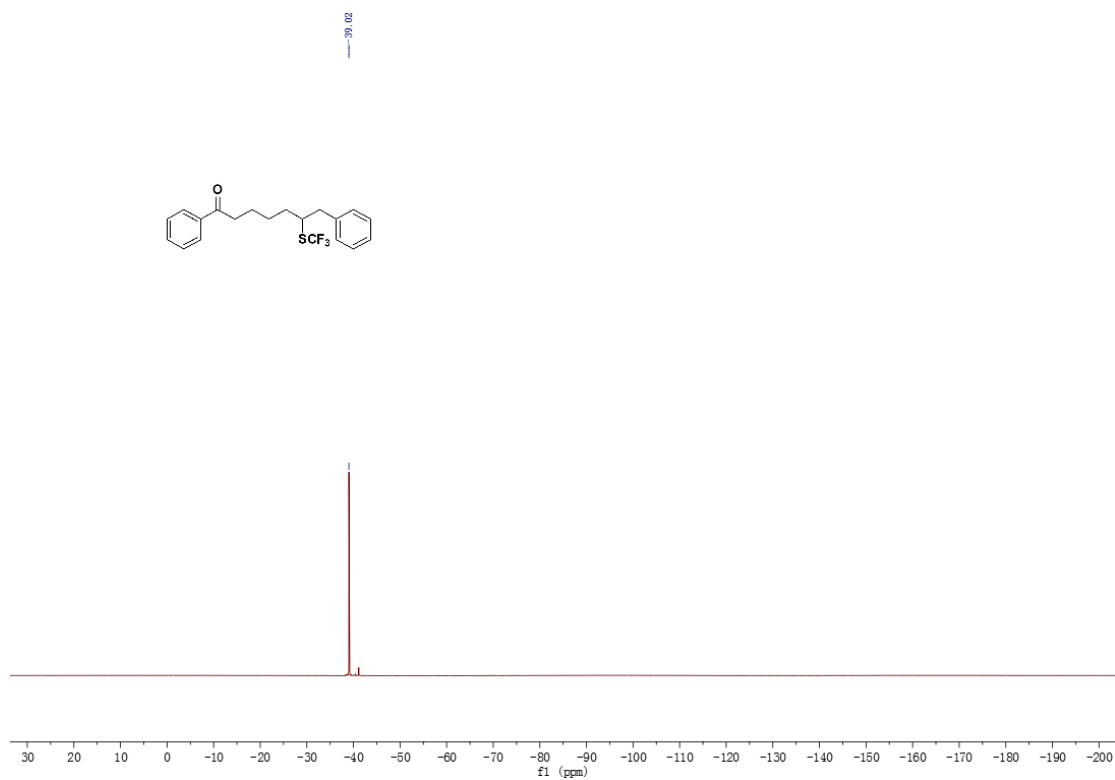
**^{13}C NMR spectrum of
1-(4-fluorophenyl)-6-((trifluoromethyl)thio)hexan-1-one 5g**



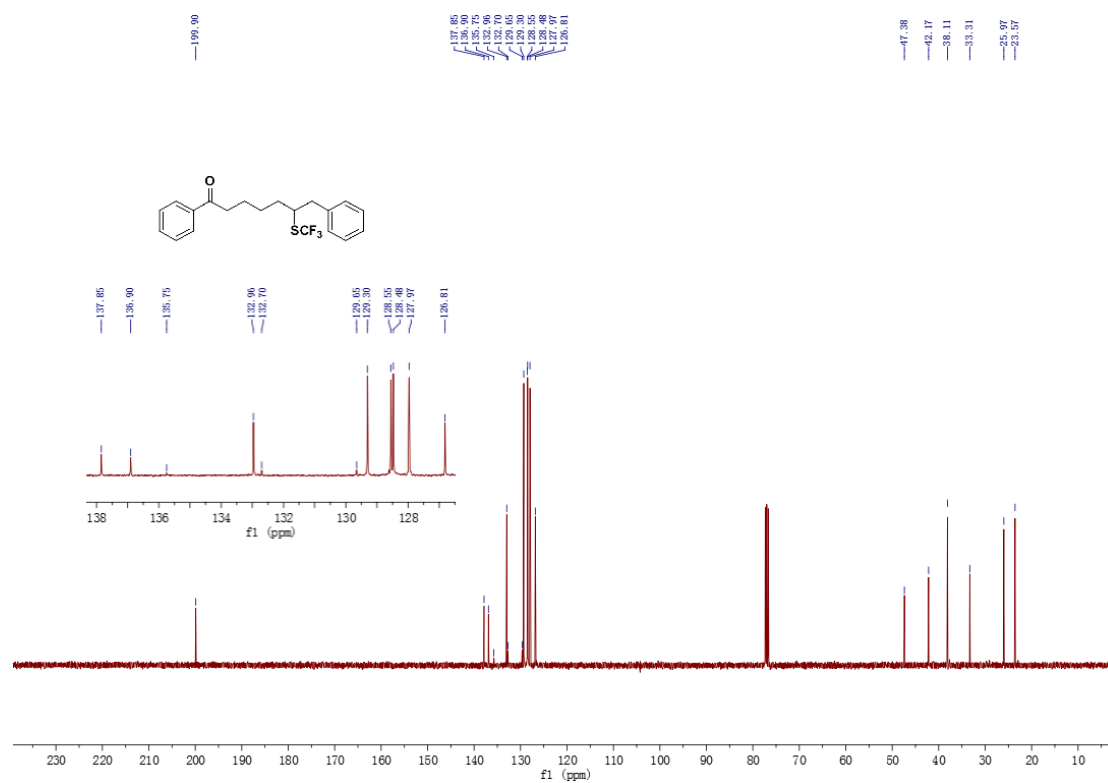
**^1H NMR spectrum of
1,7-diphenyl-6-((trifluoromethyl)thio)heptan-1-one 5h**



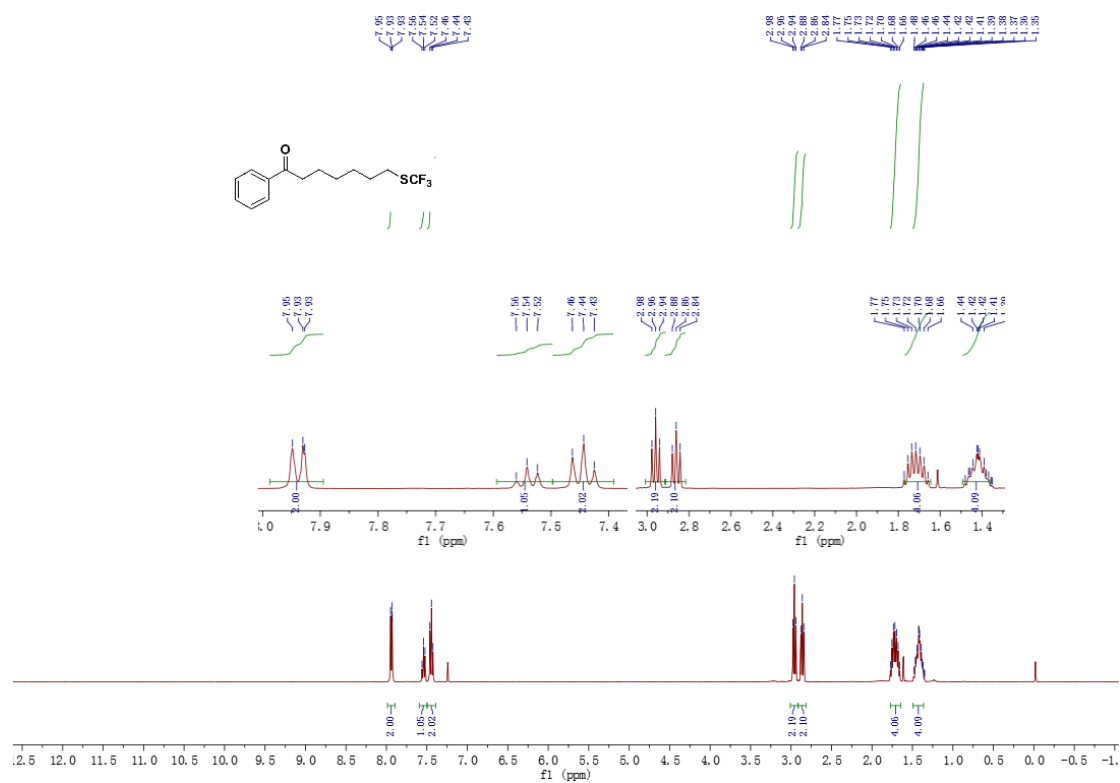
**^{19}F NMR spectrum of
1,7-diphenyl-6-((trifluoromethyl)thio)heptan-1-one 5h**



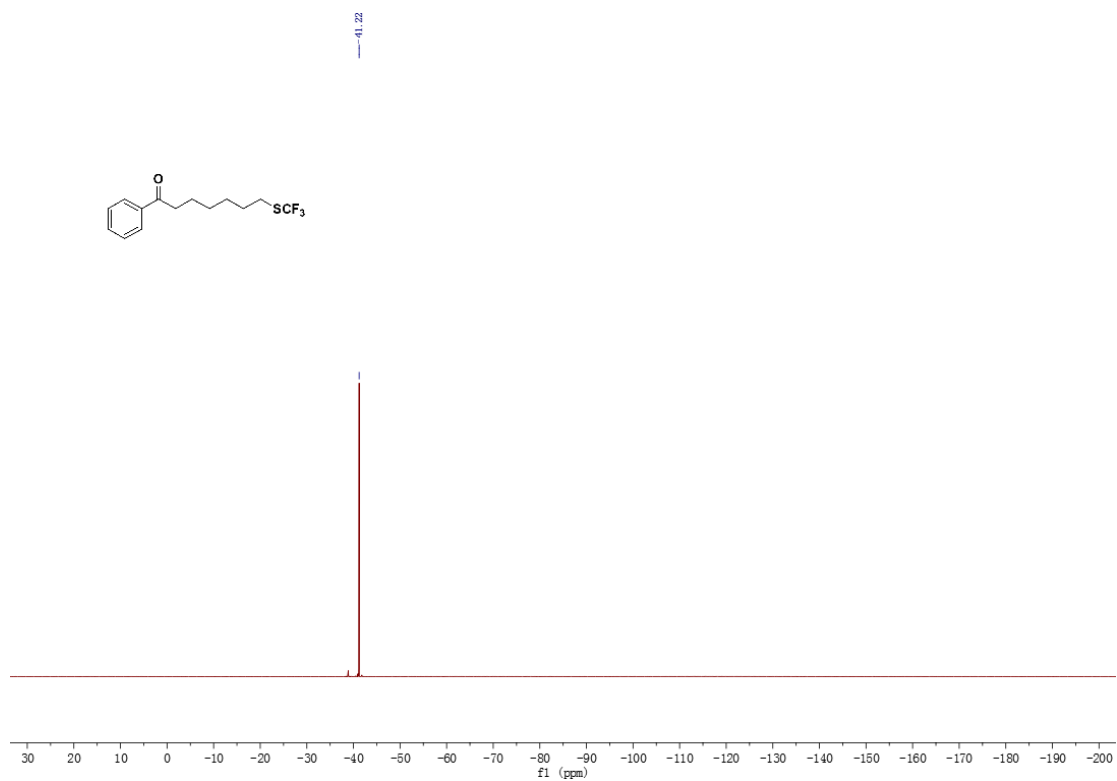
**^{13}C NMR spectrum of
1,7-diphenyl-6-((trifluoromethyl)thio)heptan-1-one 5h**



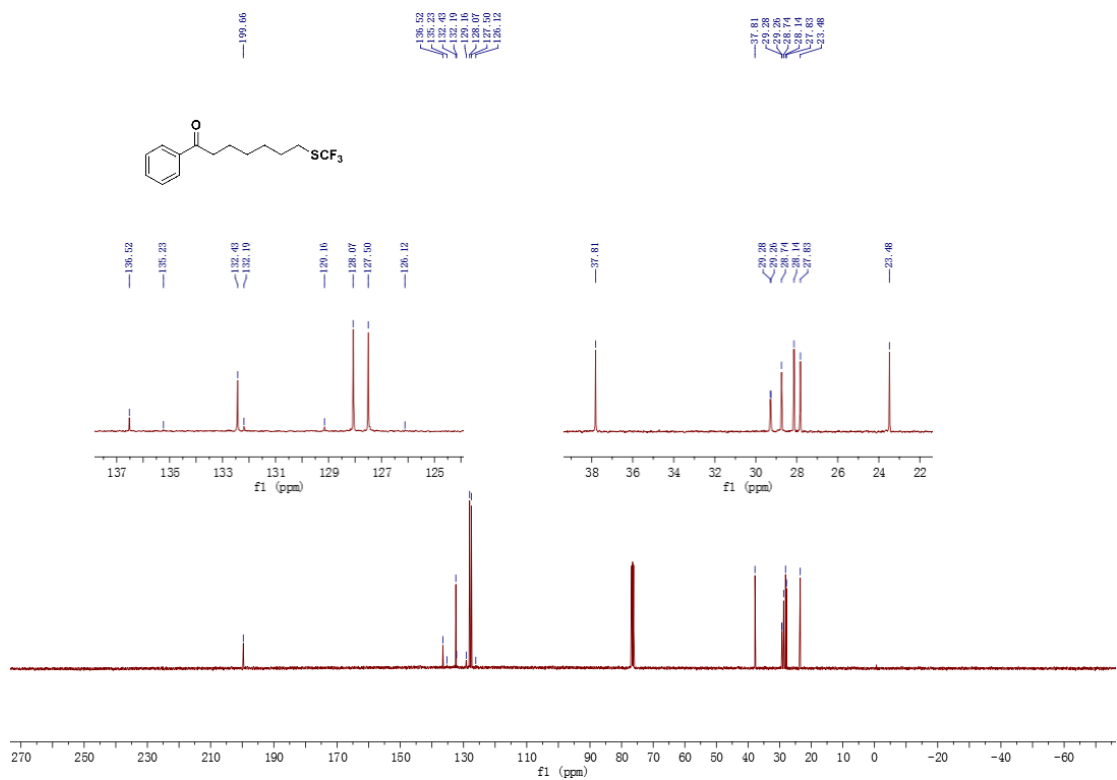
**^1H NMR spectrum of
1-phenyl-7-((trifluoromethyl)thio)heptan-1-one 5i**



**^{19}F NMR spectrum of
1-phenyl-7-((trifluoromethyl)thio)heptan-1-one 5i**

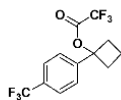


**^{13}C NMR spectrum of
1-phenyl-7-((trifluoromethyl)thio)heptan-1-one 5i**



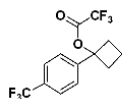
7.74
7.72
7.69
7.67

2.88
2.87
2.87
2.86
2.84
2.84
2.83
2.82
2.81
2.80
2.80
2.80
2.77
2.76
2.21
2.19
2.18
2.18
2.17
2.16
2.16
2.15
2.15
2.14
2.14
2.13
2.12
2.11
1.92
1.90
1.89
1.88
1.87
1.85
1.85
1.83
1.83



—62.91

—75.78



**^{13}C NMR spectrum of
1-(4-(trifluoromethyl)phenyl)cyclobutyl 2,2,2-trifluoroacetate**

