Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Photoredox Aerobic Oxidation of Unreactive Amines Derivatives through LMCT Excitation of Copper Dichloride

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General information

Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) and ^{19}F NMR (377 MHz) data were recorded with $CDCl_3$ or DMSO-*d6* as solvent at room temperature unless specified otherwise. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. ^{1}H NMR spectra was recorded with tetramethylsilane (δ= 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.00 ppm) or DMSO-*d6* (δ = 39.50 ppm) as internal reference. IR and HRMS were performed by the State-authorized Analytical Center in Soochow University.

General procedures

C–H oxidation of tertiary amides

To a 25 mL Schlenk tube, tertiary amides (0.2 mmol), copper dichloride (0.01 mmol), H_2O (2.0 mmol) and NH_4Cl (0.04 mmol) were dissolved in $MeNO_2$ (2.0 mL). Under the irradiation of 38 W white LEDs, the reaction mixture was stirred at room temperature under oxygen atmosphere for 72 h. After the reaction, the system is quenched with sodium thiosulfate, then poured in brine solution (15 mL) and extracted with ethyl acetate (3 x 20 mL) and then dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (ethyl acetate/ petroleum ether ether) to afford desired products.

C–H oxidation of secondary amides

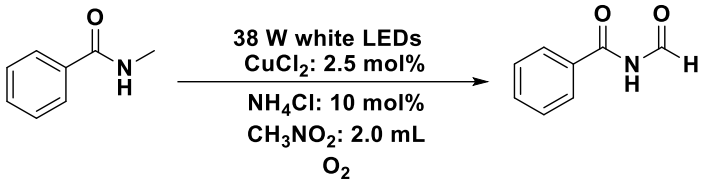
To a 25 mL Schlenk tube, secondary amides (0.2 mmol), copper dichloride (0.005 mmol) and NH_4Cl (0.02 mmol) were dissolved in $MeNO_2$ (2.0 mL). Under the irradiation of 38 W white LEDs, the reaction mixture was stirred at room temperature under oxygen atmosphere for 72 h. After the reaction, the reaction system is quenched with sodium thiosulfate, then poured in brine solution (15 mL) and extracted with ethyl acetate (3 x 20 mL) and then dried over $MgSO_4$. The solvent was removed under reduced pressure

and the residue was purified by silica gel column chromatography (ethyl acetate/ petroleum ether ether) to afford desired products.

N-Demethylation of tertiary amides

To a 25 mL Schlenk tube, amides (0.2 mmol), copper dichloride (0.01 mmol) and HCl (0.4 mmol, 37% in water) were dissolved in acetone (0.5 mL). Under the irradiation of 38 W white LEDs, the reaction mixture was stirred at room temperature under oxygen atmosphere for 72 h. After the reaction, the reaction system is quenched with sodium thiosulfate, then poured in brine solution (15 mL) and extracted with ethyl acetate (3 x 20 mL) and then dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (ethyl acetate/ petroleum ether ether) to afford desired products.

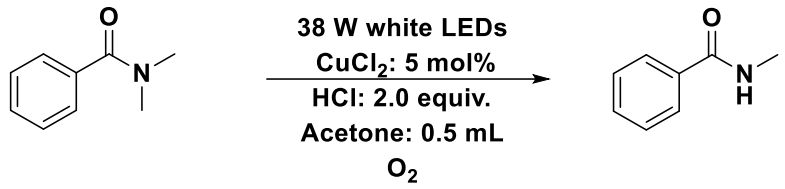
Table S1. Optimization for oxidation of secondary amides[a]



Entry	Variation from the "standard conditions" Catalyst	Yield (%)[b]
1	none	94
2	In the dark	N.D.
3	Et_3N instead of CH_3NO_2	N.D.
4	Benzene instead of CH_3NO_2	N.D.
5	MeCN instead of CH_3NO_2	62
6	EA instead of CH_3NO_2	15
7	DCM instead of CH_3NO_2	23
8	DMF instead of CH_3NO_2	N.D.
9	DMSO-*d6* instead of CH_3NO_2	N.D.
10	CH_3OH instead of CH_3NO_2	N.D.
11	THF instead of CH_3NO_2	N.D.
12	1,4-Dioxane instead of Acetone	N.D.
13	Acetone instead of Acetone	33
14	air instead of O_2	44
15	N_2 instead of O_2	N.D.

[a] Reaction conditions: amide (0.2 mmol), NH_4Cl (0.02 mmol, 10 mol%), $CuCl_2$ (0.005 mmol) in solvent (2.0 mL) irradiation with 38 W white light LEDs at ambient temperature for 72 h. [b] Isolated yields after column chromatography.

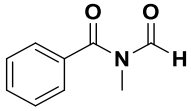
Table S2. Optimization for *N*-demethylation of tertiary amides[a]



Entry	Variation from the "standard conditions" Catalyst	Yield (%)[b]
1	none	90
2	In the dark	N.D.
3	MeCN instead of Acetone	49
4	Benzene instead of Acetone	57
5	EA instead of Acetone	41
6	CH_3NO_2 instead of Acetone	55
7	DMF instead of Acetone	< 5
8	DMSO-*d6* instead of Acetone	< 5
9	CH_3OH instead of Acetone	52
10	C_2H_5OH instead of Acetone	26
11	THF instead of Acetone	49
12	1,4-Dioxane instead of Acetone	39
13	2-Butanone instead of Acetone	62
14	air instead of O_2	80
15	N_2 instead of O_2	N.D.
16	NaCl instead of HCl	trace
17	LiCl instead of HCl	10
18	NH_4Cl instead of HCl	trace
19	Me_4NCl instead of HCl	N.D.
20	$MnCl_2$ instead of $CuCl_2$	N.D.
21	$CoCl_2$ instead of $CuCl_2$	37

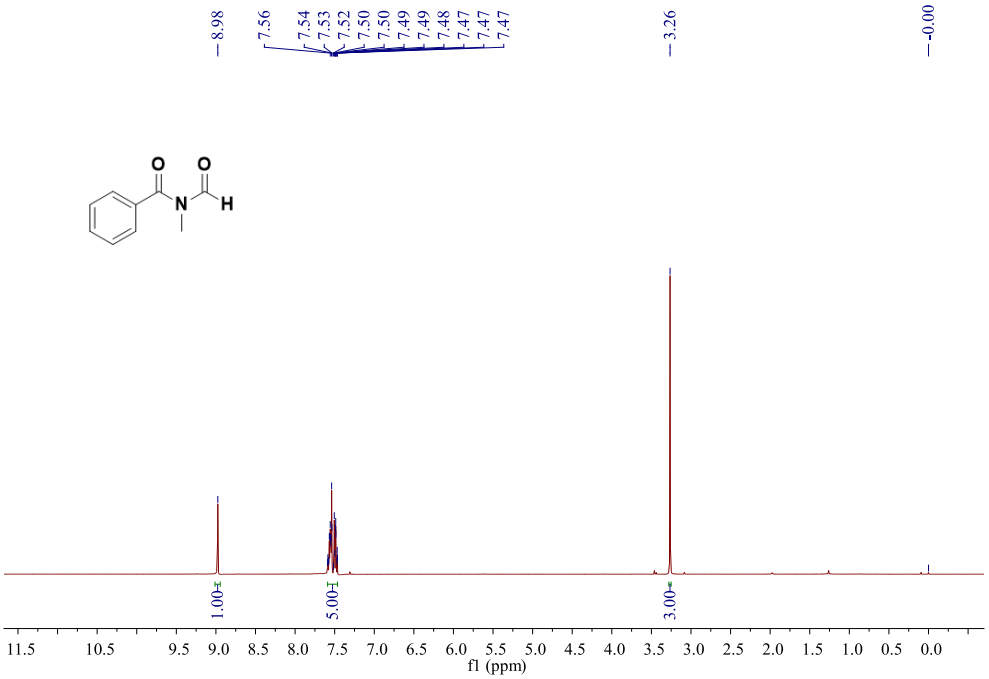
[a] Reaction conditions: amide (0.2 mmol), HCl (0.4 mmol, 2.0 equiv, 37% in water), $CuCl_2$ (0.01 mmol) in solvent (0.5 mL) irradiation with 38 W white light LEDs at ambient temperature for 72 h. [b] Isolated yields after column chromatography.

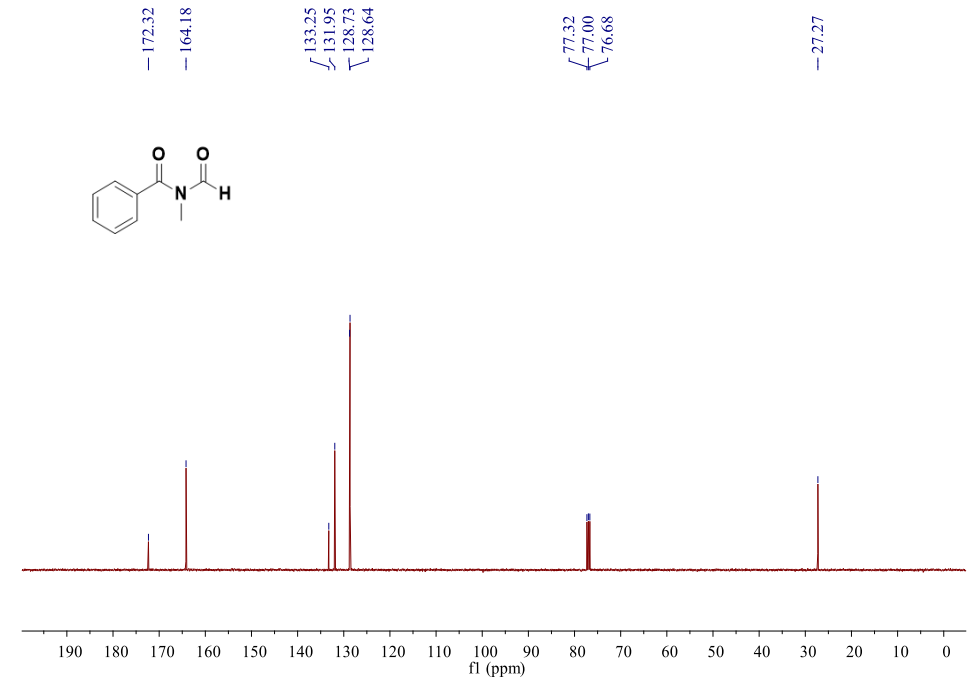
Spectral Data for Products

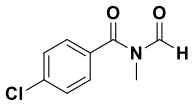


***N*-Formyl-*N*-Methylbenzamide (2a)**

petroleum ether / ethyl acetate = 5:1, white solid, 83% yield (27.1 mg). mp: 43 – 45℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.98 (s, 1H), 7.59 – 7.47 (m, 5H), 3.26 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.32, 164.18, 133.25, 131.95, 128.73, 128.64, 27.27. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_9NO_2$+Na^+: 186.0525, Found: 186.0525. **IR** (neat, cm^{-1}): υ 3049, 2956, 1717, 1652, 1529, 1340, 800, 729.

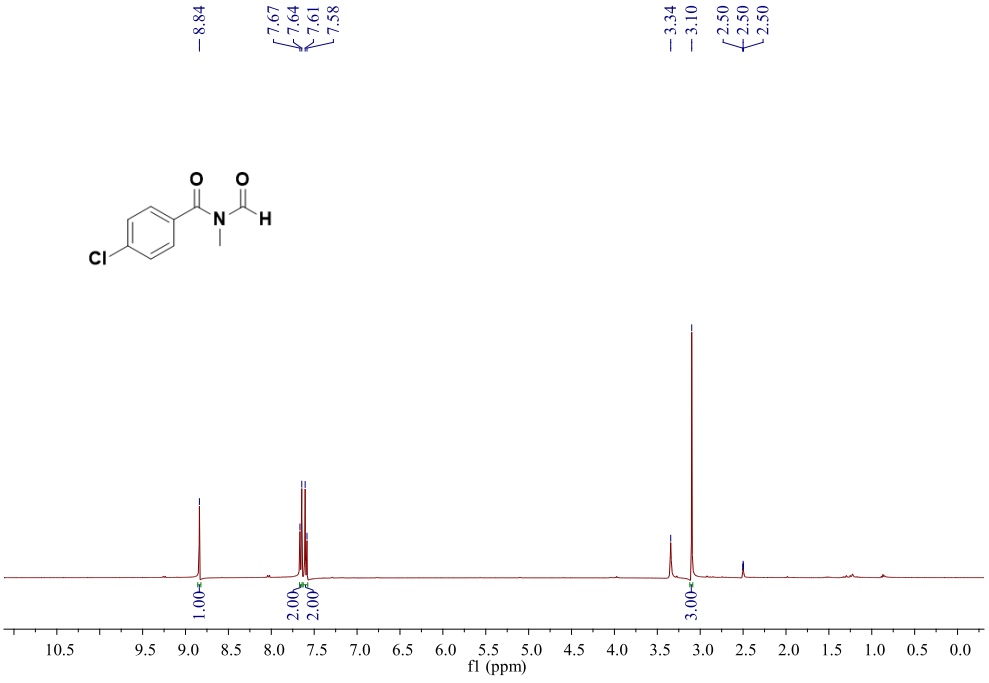


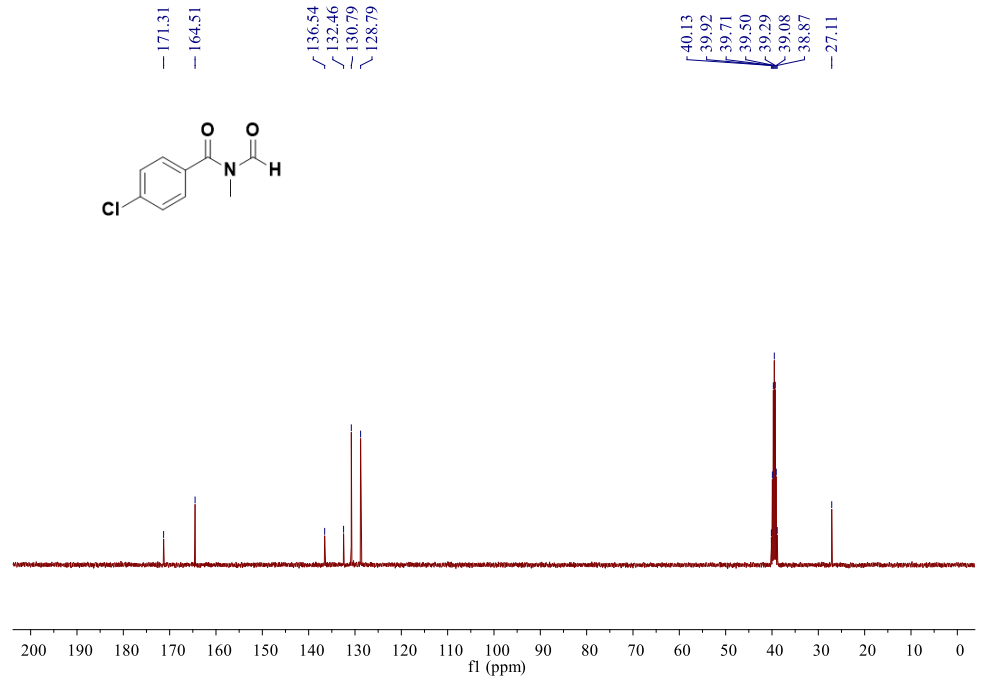


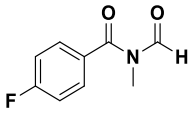


4-Chloro-*N*-formyl-*N*-Methylbenzamide (2b)

petroleum ether / ethyl acetate = 5:1, white solid, 68% yield (26.8 mg). mp: 178 – 180℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.84 (s, 1H), 7.66 (d, *J* = 8.6 Hz, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 3.10 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.31, 164.51, 136.54, 132.46, 130.79, 128.79, 27.11. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8{}^{35}ClNO_2$+Na^+: 220.0136, Found: 220.0133. Anal Calcd. For. $C_9H_8{}^{37}ClNO_2$+Na^+: 222.0106, Found: 222.0105. **IR** (neat, cm^{-1}): υ 3089, 2960, 1713, 1656, 1589, 1406, 1338, 917, 841.

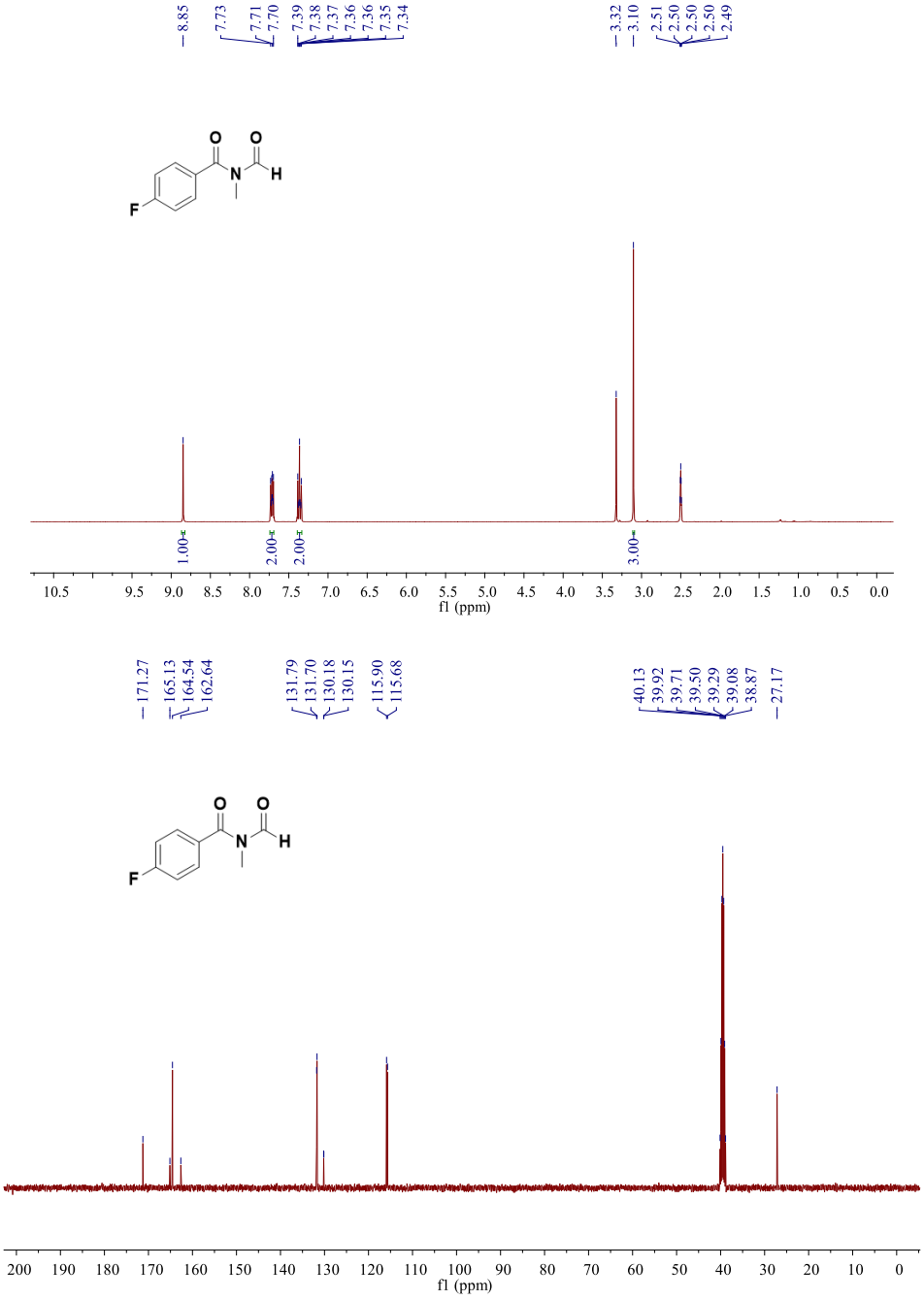


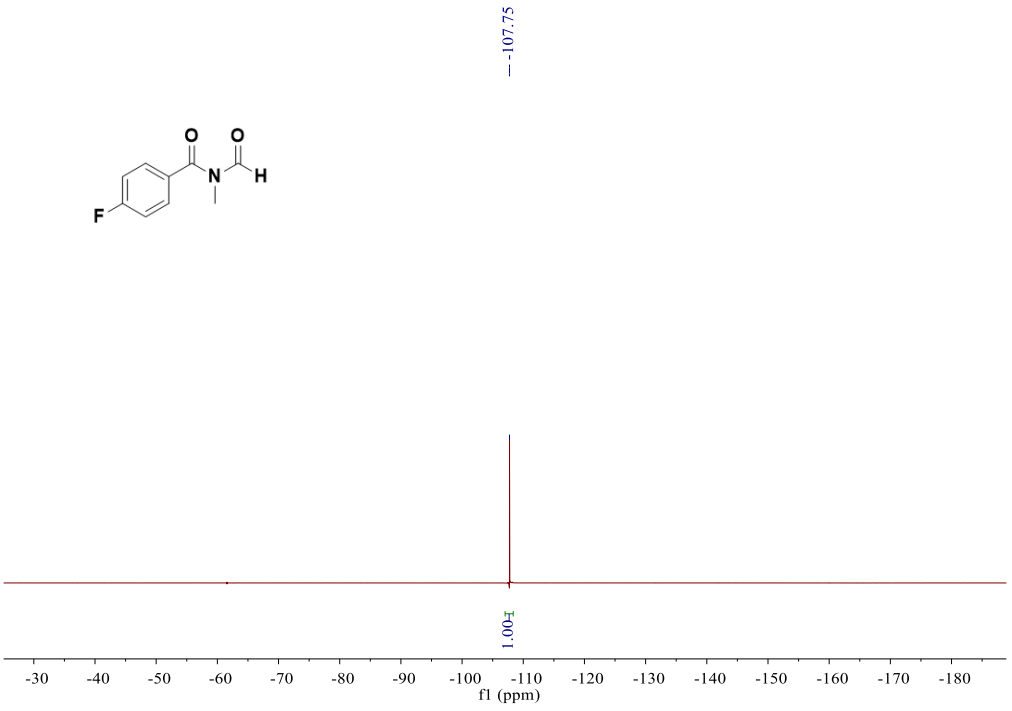




4-Fluoro-*N*-Formyl-*N*-Methylbenzamide (2c)

petroleum ether / ethyl acetate = 5:1, white solid, 58% yield (21.0 mg). mp: 69 – 71℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.85 (s, 1H), 7.73 – 7.70 (m, 2H), 7.39 – 7.34 (m, 2H), 3.10 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.27, 164.54, 163.9 (d, *J* = 249.0 Hz), 131.74 (d, *J* = 9.2 Hz), 130.17 (d, *J* = 3.0 Hz), 115.79 (d, *J* = 22.0 Hz), 27.17. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -107.75 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8FNO_2$+Na^+: 204.0431, Found: 204.0430. **IR** (neat, cm^{-1}): υ 3074, 2941, 1720, 1655, 1508, 1473, 1339, 823, 785.

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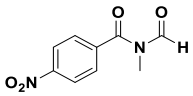
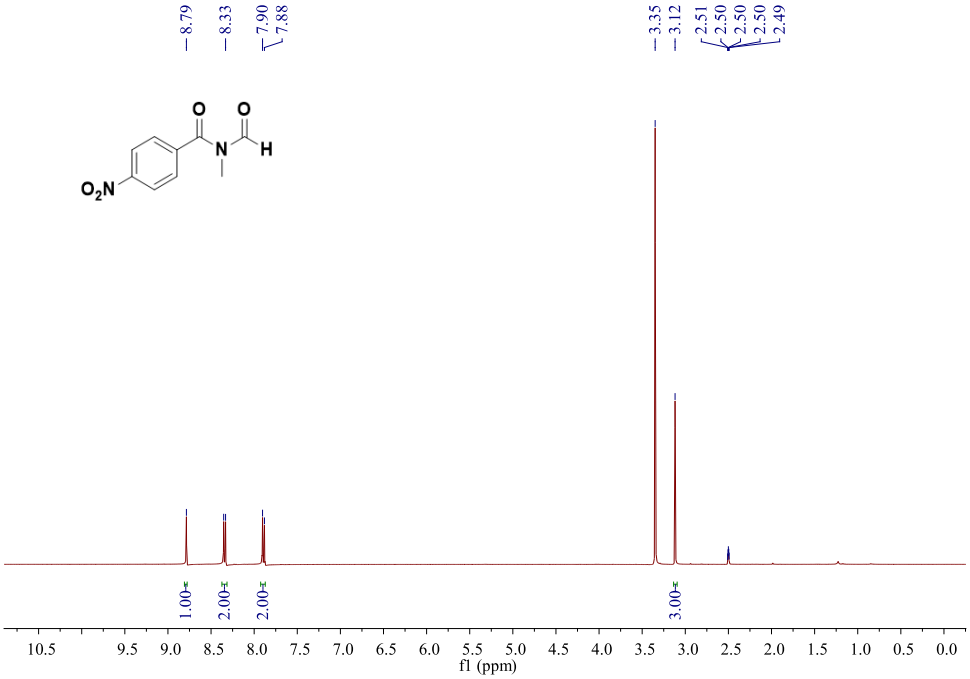
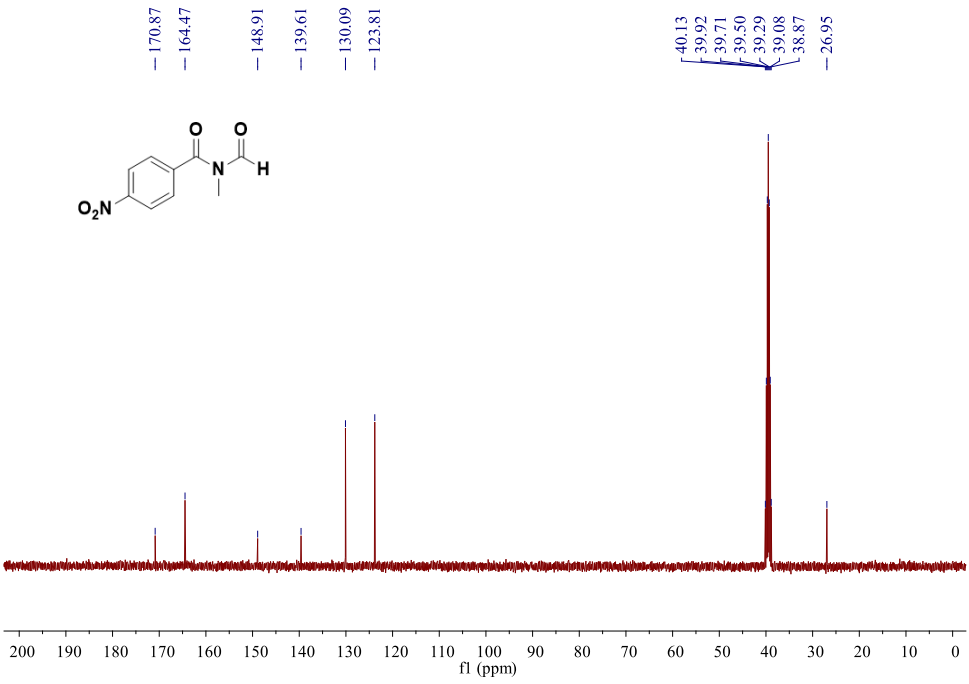
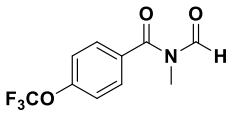


***N*-Formyl-*N*-Methyl-4-Nitrobenzamide (2d)**

petroleum ether / ethyl acetate = 5:1, white solid, 65% yield (27.0 mg). mp: 122 – 124℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.79 (s, 1H), 8.34 (d, *J* = 8.8 Hz, 2H), 7.89 (d, *J* = 8.8 Hz, 2H), 3.12 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 170.87, 164.47, 148.91, 139.61, 130.09, 123.81, 26.95. **HRMS** (EI-TOF): Anal Calcd. For. $C_9H_8N_2O_4$: 208.0484, Found: 208.0479. **IR** (neat, cm^{-1}): υ 2984, 2877, 1736, 1673, 1530, 1480, 1372, 847.

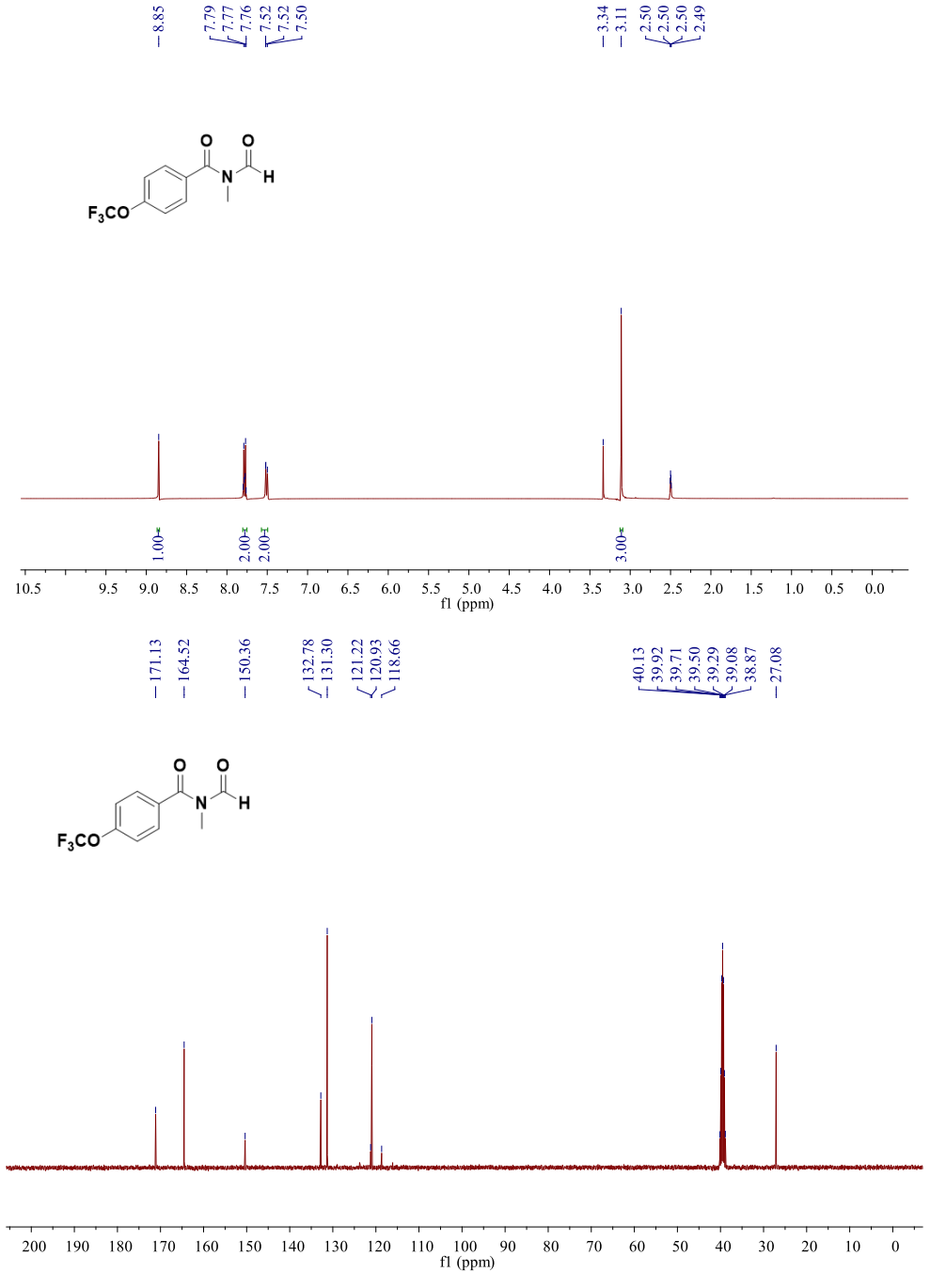


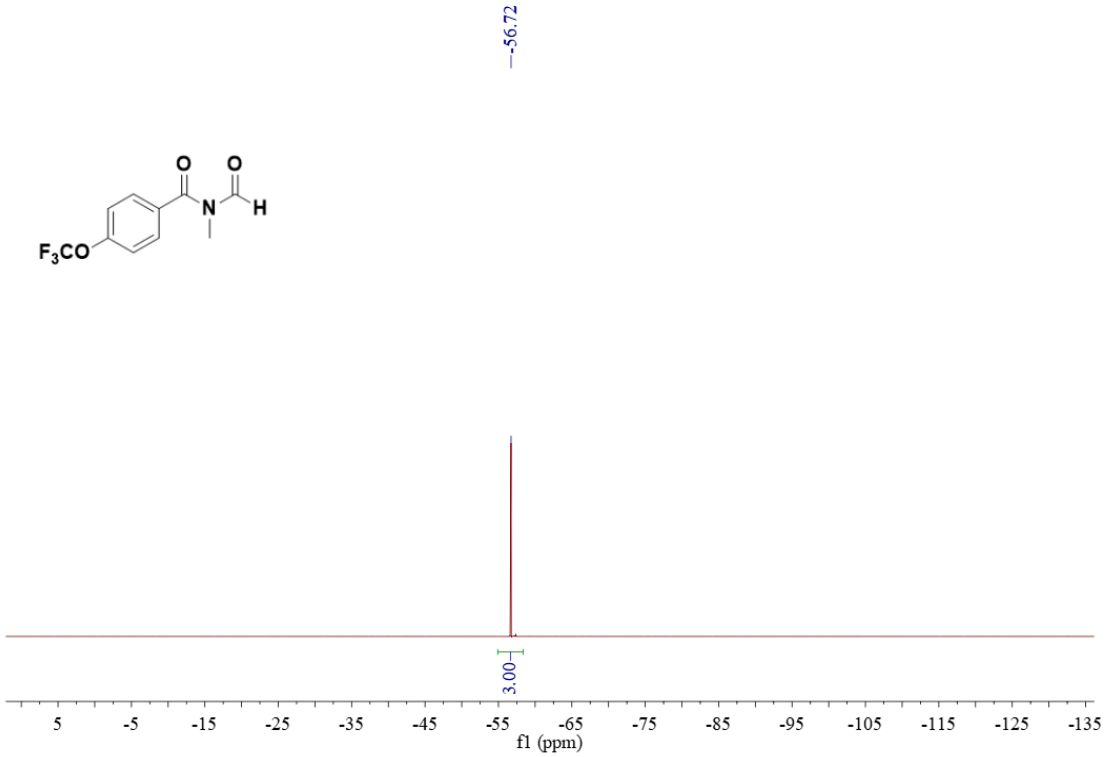




***N*-Formyl-*N*-Methyl-4-(trifluoromethoxy)benzamide (2e)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 63% yield (31.1 mg). mp: 99 – 100℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.85 (s, 1H), 7.79 – 7.76 (m, 2H), 7.52 – 7.50 (m, 2H), 3.11 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.13, 164.52, 150.36, 132.78, 131.30, 120.93, 119.94 (q, *J* = 256.0 Hz), 27.08. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.72 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_8F_3NO_3$+Na^+: 270.0348, Found: 270.0346. **IR** (neat, cm^{-1}): υ 3055, 2937, 1716, 1646, 1571, 1448, 1399, 1245, 864,792.

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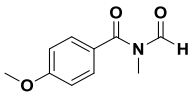
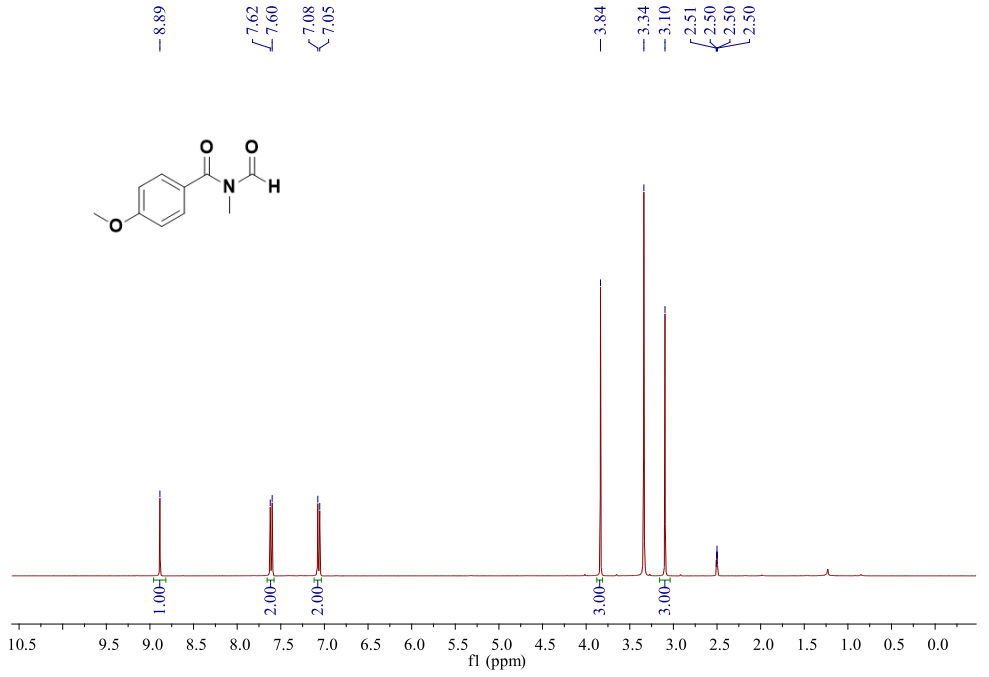
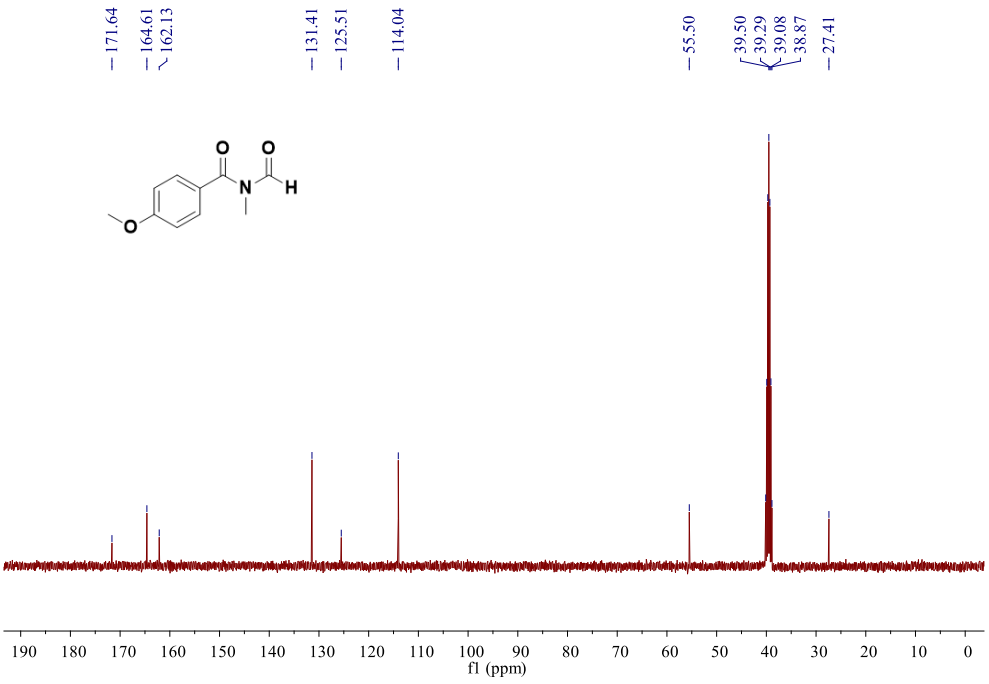
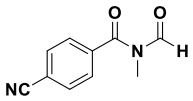


***N*-Formyl-4-Methoxy-*N*-methylbenzamide (2f)**

petroleum ether / ethyl acetate = 5:1, white solid, 44% yield (17.0 mg). mp: 77 – 79℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.89 (s, 1H), 7.61 (d, *J* = 8.8 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 3.10 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.64, 164.61, 162.13, 131.41, 125.51, 114.04, 55.50, 27.41. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{11}NO_3$+Na^+: 216.0631, Found: 216.0621. **IR** (neat, cm^{-1}): υ 3006, 2934, 1624, 1575, 1300, 1246, 840, 797.

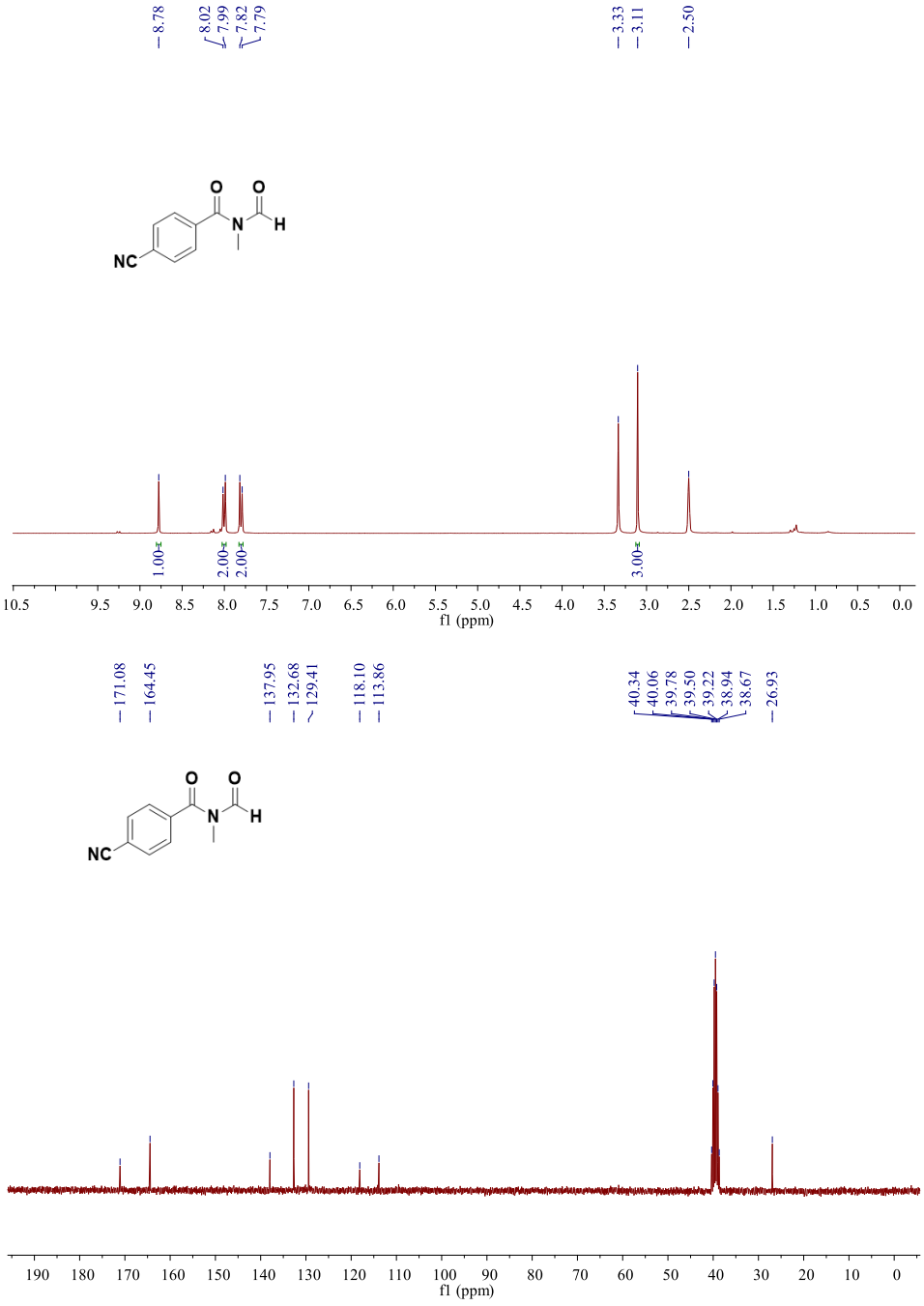


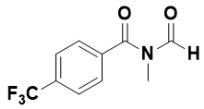




4-Cyano-*N*-formyl-N-methylbenzamide (2g)

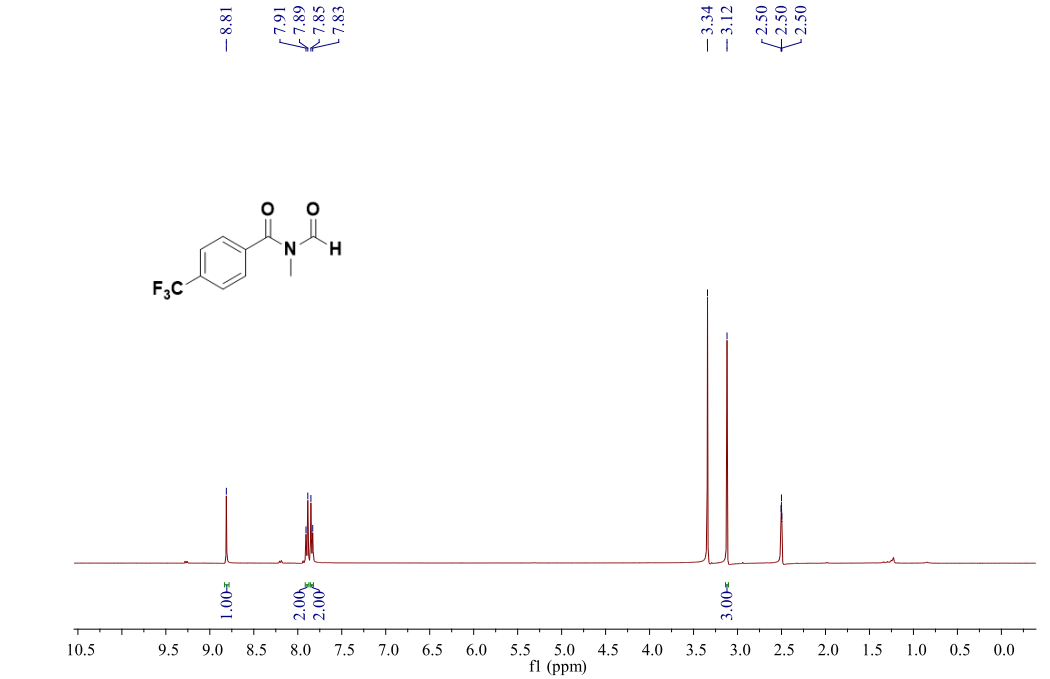
petroleum ether / ethyl acetate = 5:1, white solid, 78% yield (29.3 mg). mp: 120 – 121℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 8.78 (s, 1H), 8.00 (d, *J* = 8.2 Hz, 2H), 7.80 (d, *J* = 8.2 Hz, 2H), 3.11 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 171.08, 164.45, 137.95, 132.68, 129.41, 118.10, 113.86, 26.93. **HRMS** (EI-TOF): Anal Calcd. For. $C_{10}H_8N_2O_2$: 188.0586, Found: 188.0588. **IR** (neat, cm^{-1}): υ 3041, 2930, 2225, 1622, 1558, 1491, 1396, 857, 763.

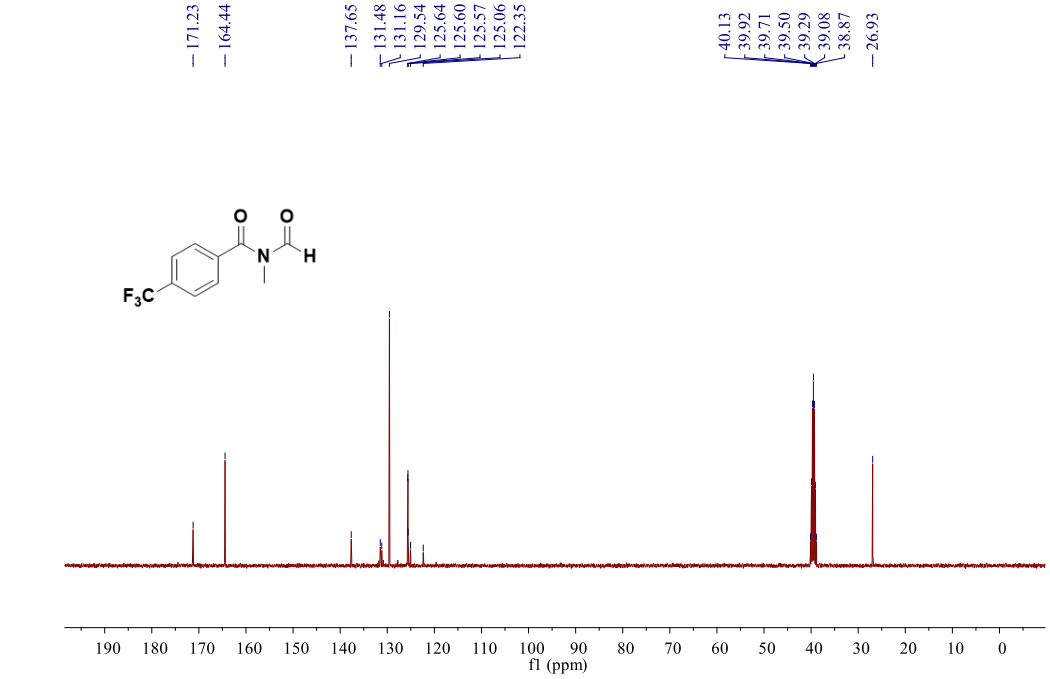


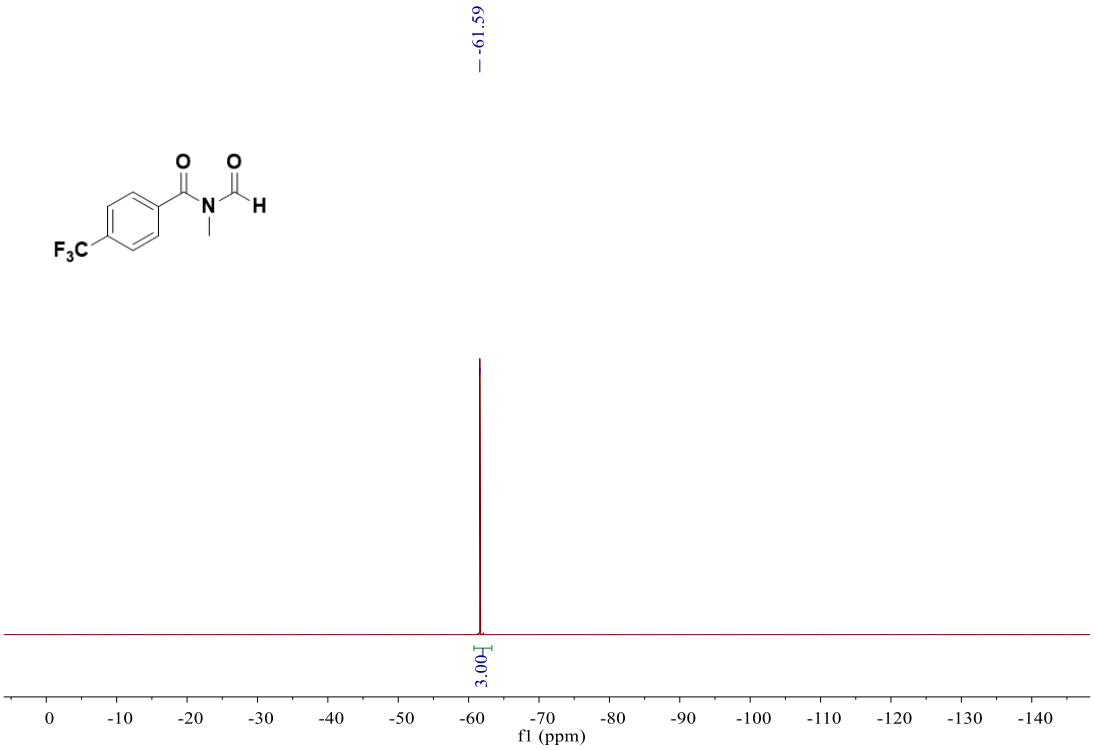


***N*-Formyl-*N*-Methyl-4-(trifluoromethyl)benzamide (2h)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 54% yield (25.0 mg). mp: 60 – 62℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.81 (s, 1H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.84 (d, *J* = 8.3 Hz, 2H), 3.12 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.23, 164.44, 137.65, 131.32 (q, *J* = 32.2 Hz), 129.54, 125.62 (q, *J* = 3.7 Hz), 123.71 (q, *J* = 272.5 Hz), 26.93. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.59 (s, 3F). **HRMS** (EI-TOF): Anal Calcd. For. $C_{10}H_8F_3NO_2$: 231.0507, Found: 231.0500. **IR** (neat, cm^{-1}): υ 3061, 2998, 1620, 1578, 1452, 1322, 861, 781.



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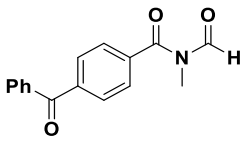
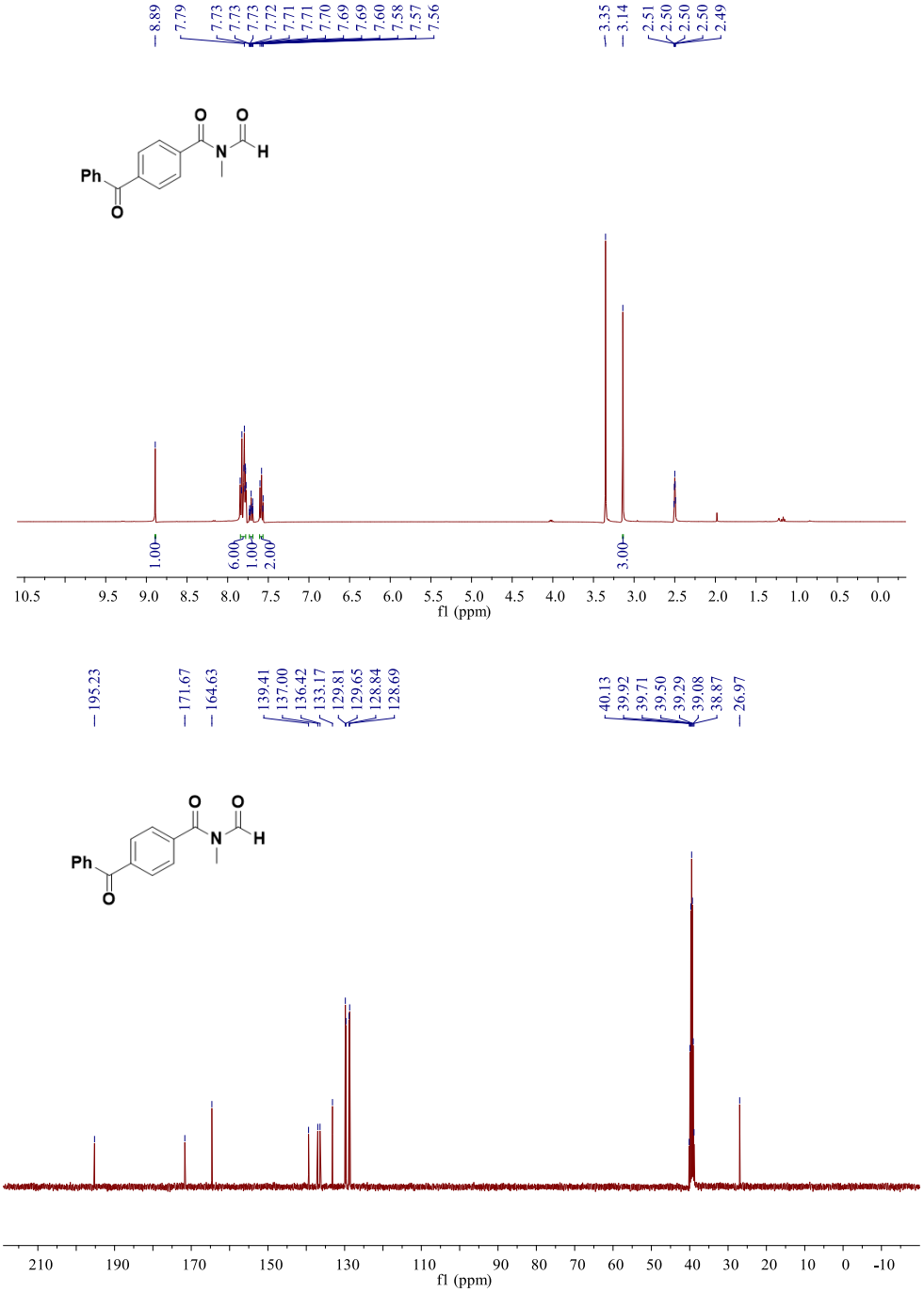
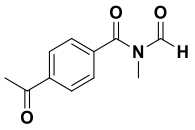


4-Benzoyl-*N*-Formyl-*N*-Methylbenzamide (2i)

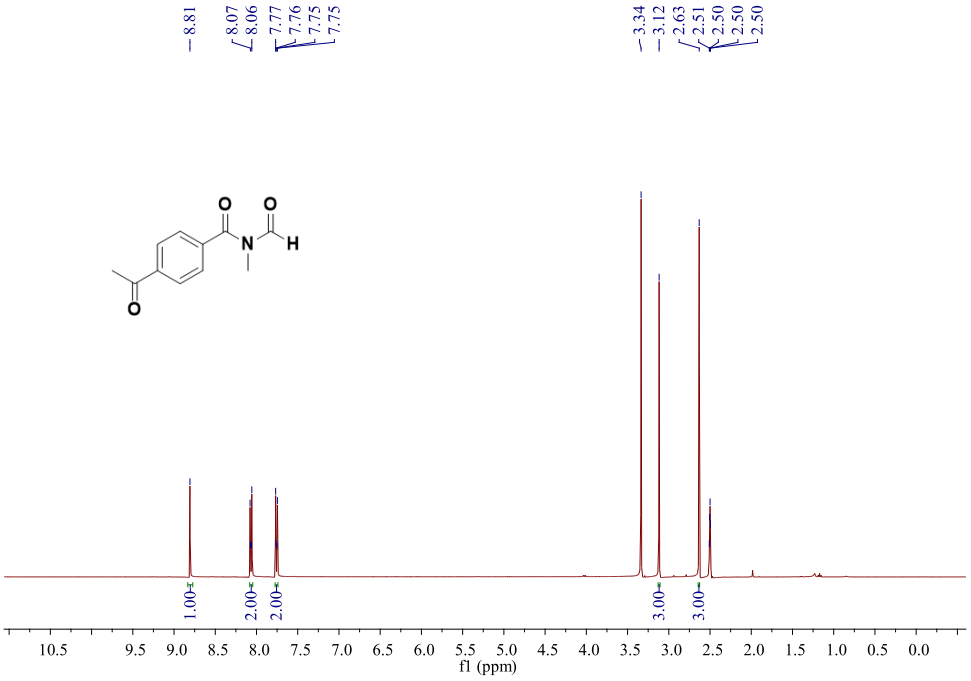
petroleum ether / ethyl acetate = 5:1, white solid, 80% yield (42.7 mg). mp: 111 – 113℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.89 (s, 1H), 7.85 – 7.77 (m, 6H), 7.73 – 7.69 (m, 1H), 7.60 – 7.56 (m, 2H), 3.14 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 195.23, 171.67, 164.63, 139.41, 137.00, 136.42, 133.17, 129.81, 129.65, 128.84, 128.69, 26.97. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{16}H_{13}NO_3$+Na^+: 290.0788, Found: 290.0771. **IR** (neat, cm^{-1}): υ 2944, 1724, 1648, 1593, 1403, 1338, 839,765.

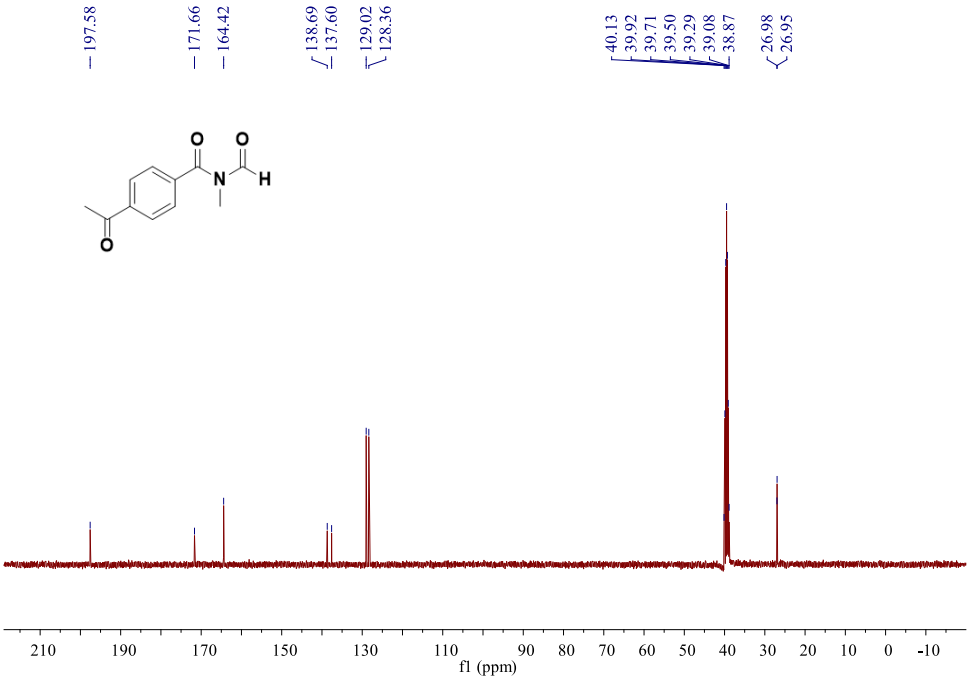


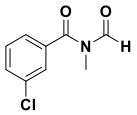


4-Acetyl-*N*-formyl-*N*-methylbenzamide (2j)

petroleum ether / ethyl acetate = 5:1, white solid, 57% yield (23.4 mg). mp: 63 – 65℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.81 (s, 1H), 8.08 – 8.06 (m, 2H), 7.77 – 7.75 (m, 2H), 3.12 (s, 3H), 2.63 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 197.58, 171.66, 164.42, 138.69, 137.60, 129.02, 128.36, 26.98, 26.95. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{11}NO_3$+Na^+: 228.0631, Found: 228.0624. **IR** (neat, cm^{-1}): υ 3091, 2941, 1722, 1671, 1525, 1470, 1348, 843, 778.

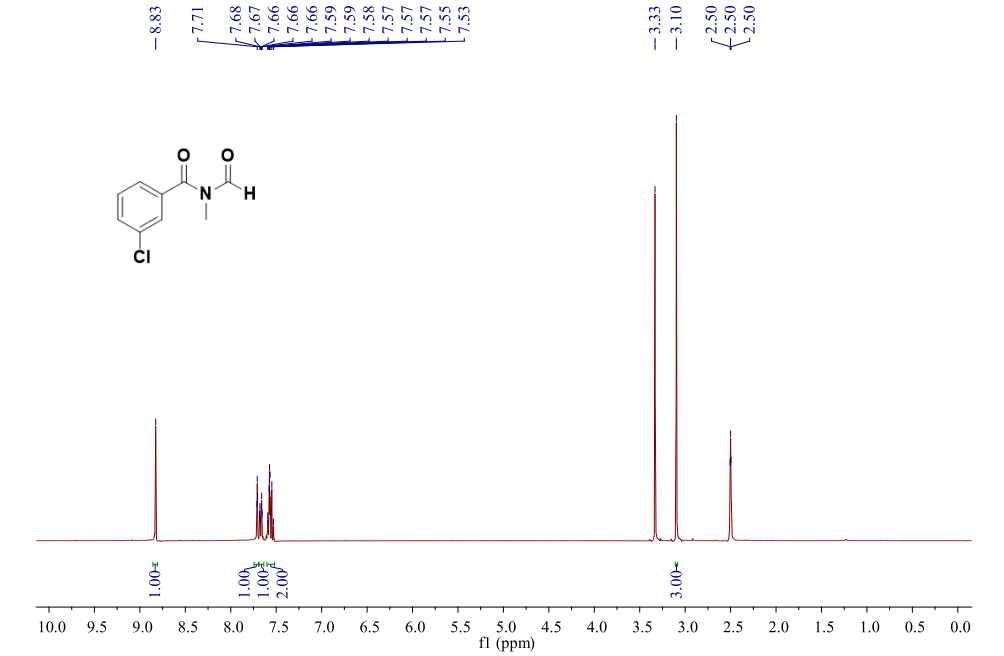


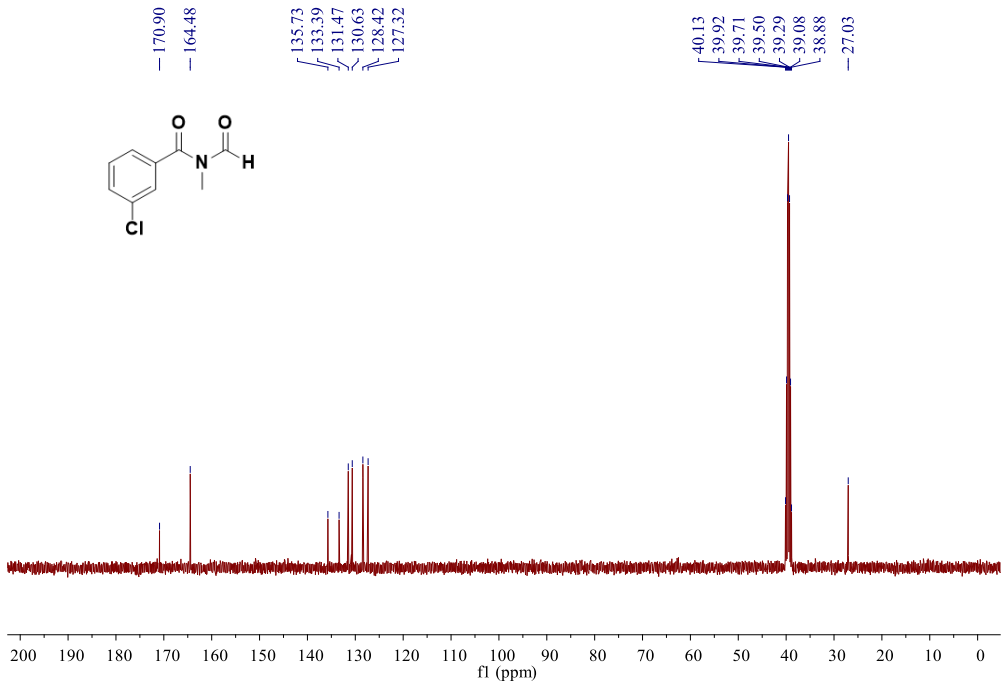


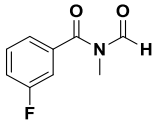


3-Chloro-*N*-Formyl-*N*-Methylbenzamide (2k)

petroleum ether / ethyl acetate = 5:1, white solid, 69% yield (27.2 mg). mp: 55 – 57℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.83 (s, 1H), 7.71 (t, *J* = 1.6 Hz, 1H), 7.68 – 7.66 (m, 1H), 7.59 – 7.53 (m, 2H), 3.10 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 170.90, 164.48, 135.73, 133.39, 131.47, 130.63, 128.42, 127.32, 27.03. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8{}^{35}ClNO_2$+Na^+: 220.0136, Found: 220.0137. Anal Calcd. For. $C_9H_8{}^{37}ClNO_2$+Na^+: 222.0106, Found: 222.0108. **IR** (neat, cm^{-1}): υ 3049, 2930, 2867, 1731, 1614, 1499, 1306, 922, 802, 769.

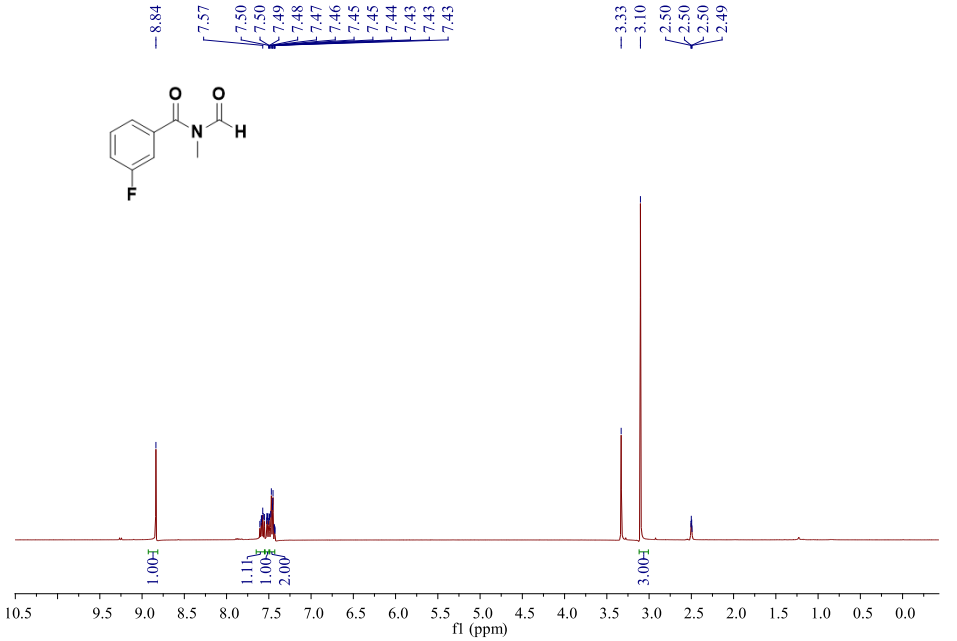


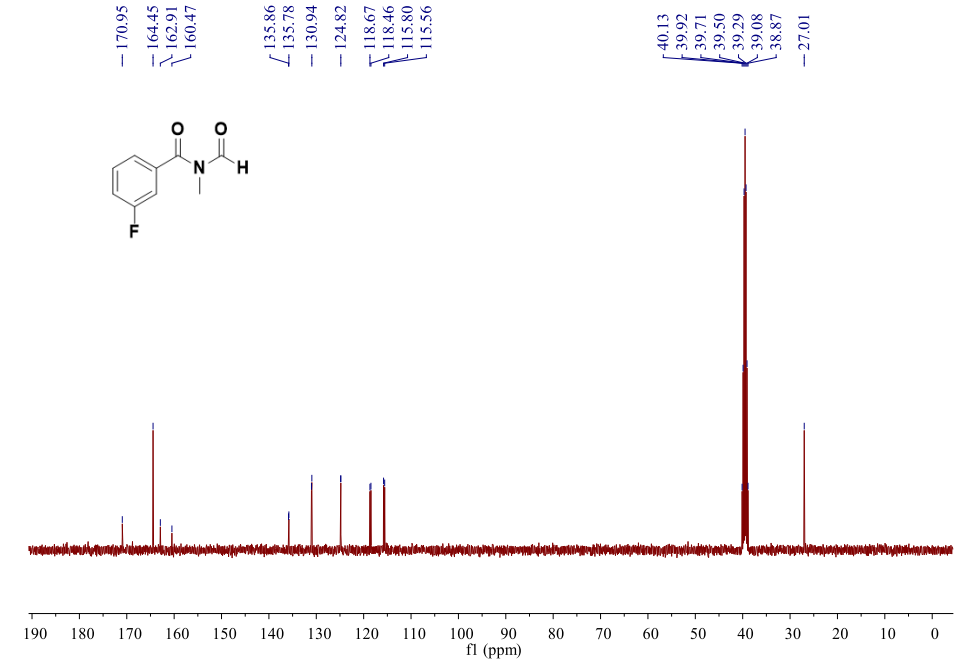


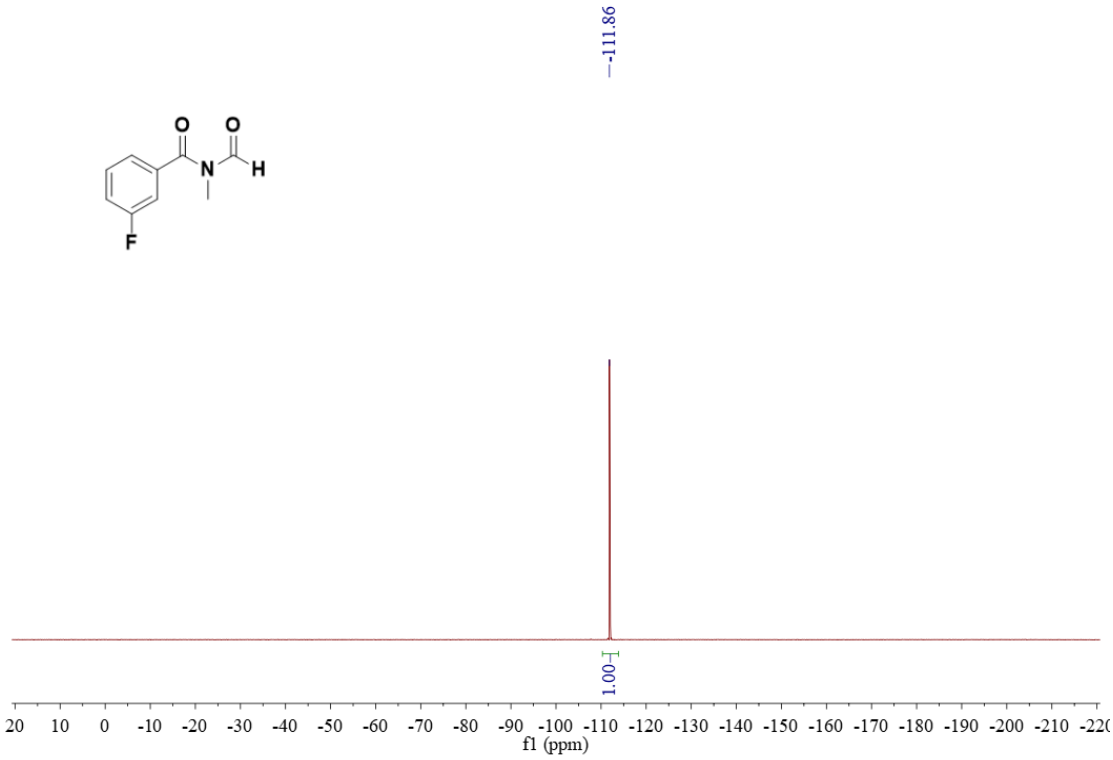


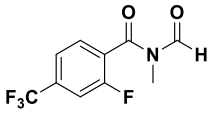
3-Fluoro-*N*-formyl-*N*-Methylbenzamide (2l)

petroleum ether / ethyl acetate = 5:1, yellow solid, 70% yield (25.3 mg). mp: 60 – 62℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.84 (s, 1H), 7.61 – 7.55 (m, 1H), 7.52 – 7.49 (m, 1H), 7.48 – 7.43 (m, 2H), 3.10 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 170.95, 164.45, 161.69 (d, *J* = 245.7 Hz), 135.82 (d, *J* = 7.4 Hz), 130.98 (d, *J* = 8.1 Hz), 124.84 (d, *J* = 3.0 Hz), 118.57 (d, *J* = 21.1 Hz), 115.68 (d, *J* = 23.8 Hz), 27.01. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -111.86 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8FNO_2$+Na^+: 204.0431, Found: 204.0429. **IR** (neat, cm^{-1}): υ 3073, 2950, 1720, 1656, 1508, 1416, 1340, 851, 785.



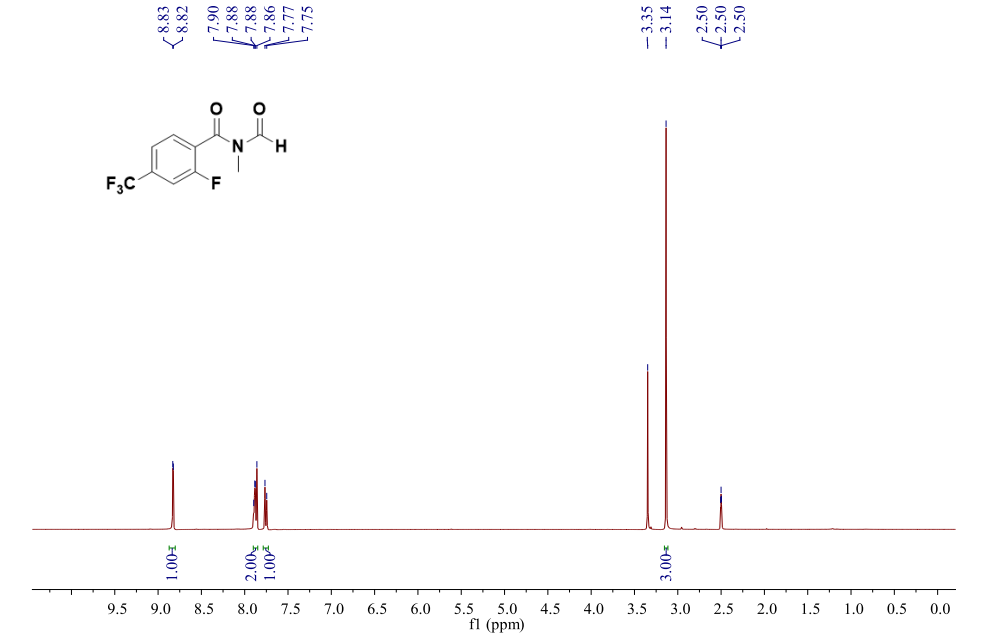


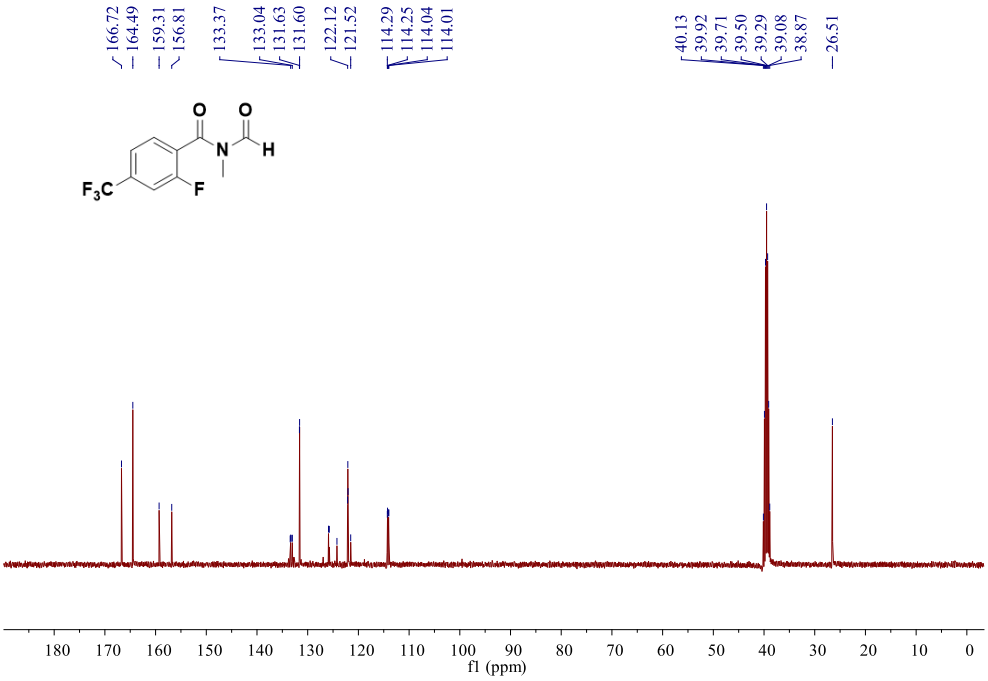

-111.86
O
O
N
H
F
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
f1 (ppm)

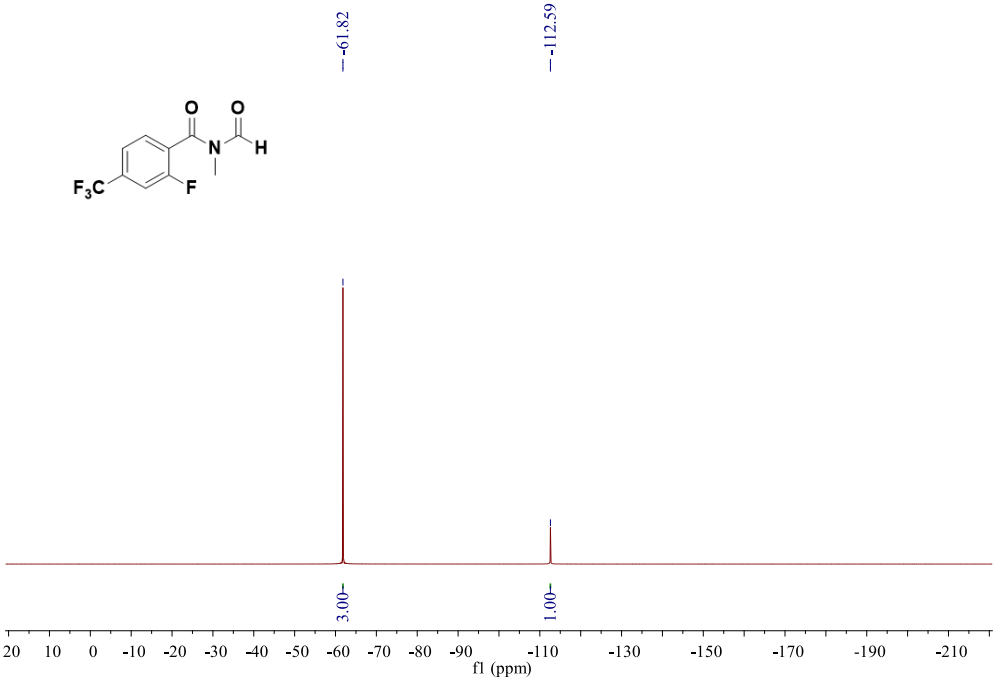


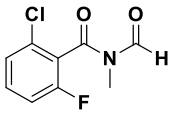
2-Fluoro-*N*-Formyl-*N*-Methyl-4-(trifluoromethyl)Benzamide (2m)

petroleum ether / ethyl acetate = 5:1, white solid, 80% yield (39.9 mg). mp: 60 – 62℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.83 (d, *J* = 2.6 Hz, 1H), 7.90 – 7.86 (m, 2H), 7.77 – 7.75 (m, 1H), 3.14 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.72, 164.49, 158.06 (d, *J* = 250.7 Hz), 133.24 (m), 131.61 (d, *J* = 2.9 Hz), 125.82 (d, *J* = 15.8 Hz), 122.88 (d, *J* = 271.0 Hz), 122.08 (m), 114.15 (m), 26.51. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.82 (s, 3F), -112.59 (s, 1F). **HRMS** (EI-TOF): Anal Calcd. For. $C_{10}H_7F_4NO_2$: 249.0413, Found: 249.0420. **IR** (neat, cm^{-1}): υ 3056, 2939, 1720, 1642, 1580, 1491, 1327, 893, 767.



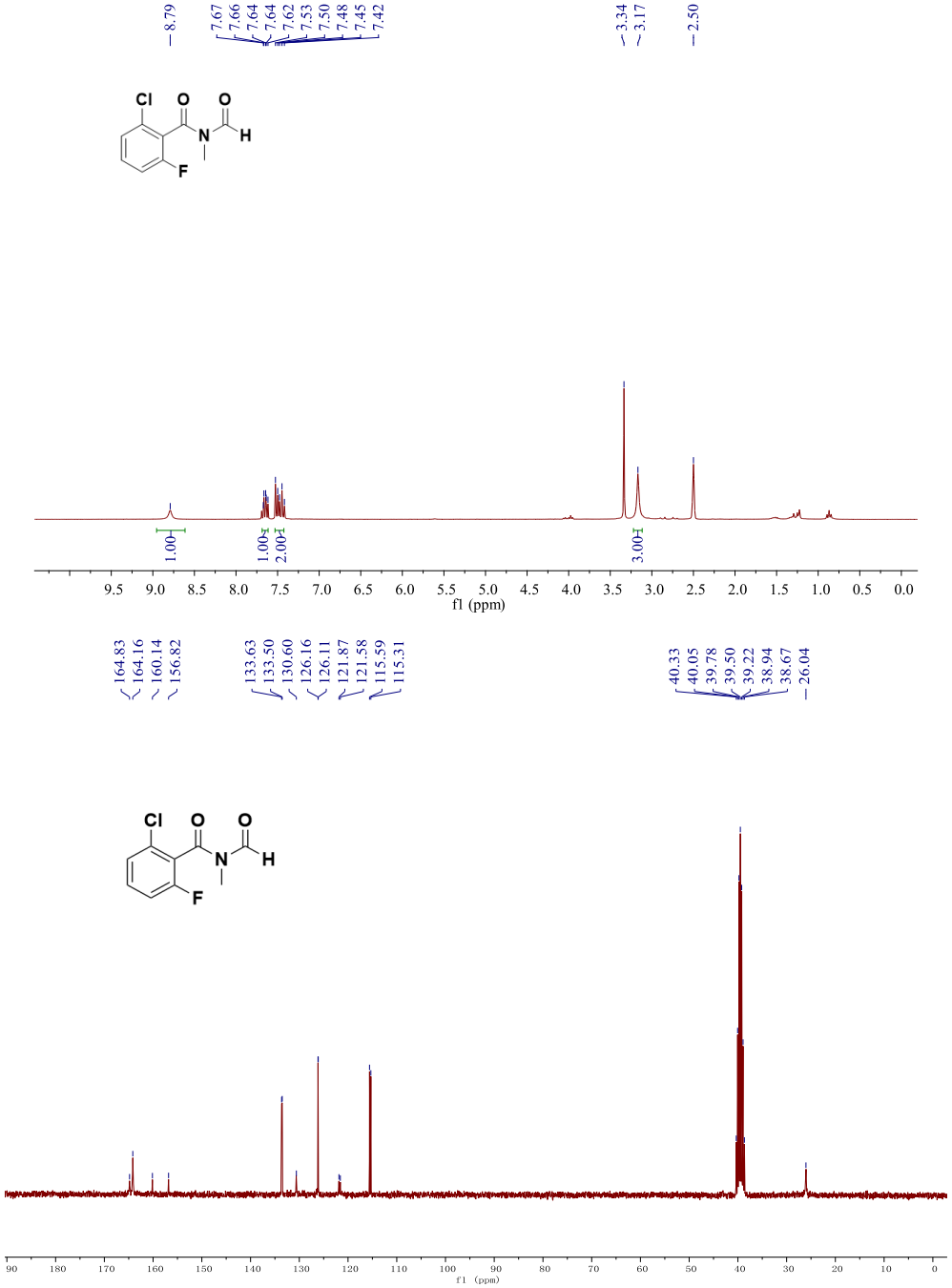


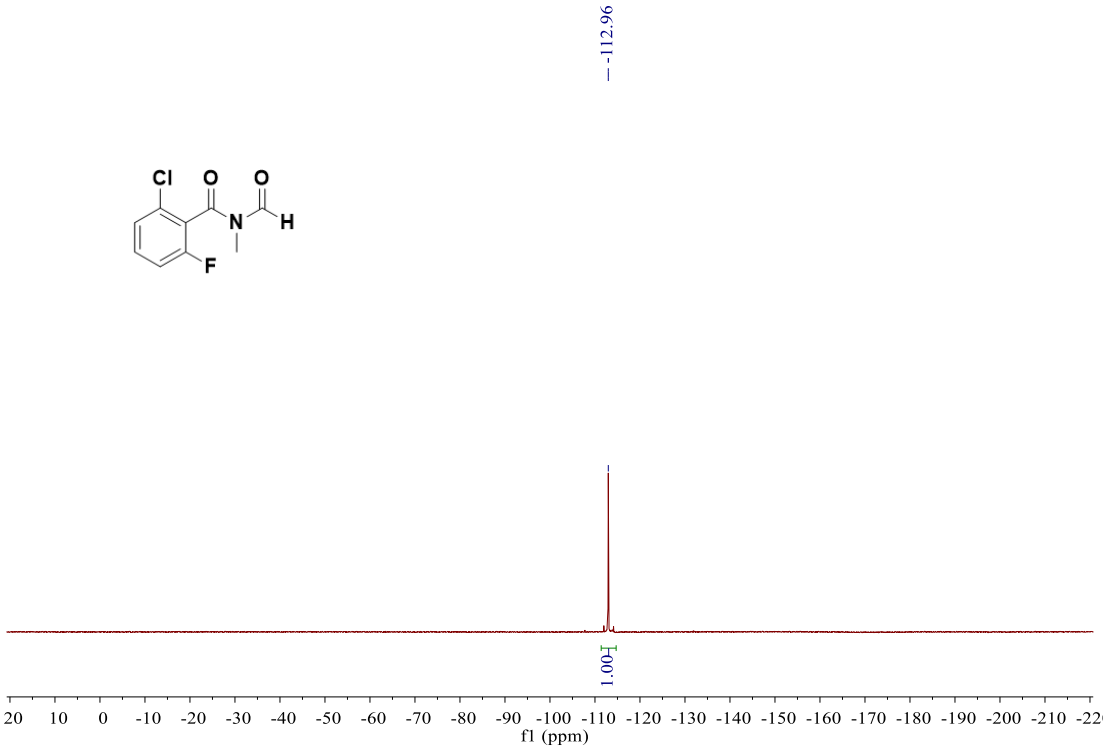

-61.82
-112.59
O
O
N
H
F3C
F
3.00
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)



2-Chloro-6-fluoro-*N*-formyl-*N*-methylbenzamide (2n)

petroleum ether / ethyl acetate = 5:1, white solid, 74% yield (31.8 mg). mp: 50 – 52℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 8.79 (s, 1H), 7.67 – 7.62 (m, 1H), 7.53 – 7.42 (m, 2H), 3.17 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 164.83, 164.16, 158.48 (d, *J* = 250.0 Hz), 133.56 (d, *J* = 9.4 Hz), 130.60, 126.14 (d, *J* = 3.3 Hz), 121.72 (d, *J* = 21.7 Hz), 115.45 (d, *J* = 20.7 Hz), 26.04. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -112.96 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_7{}^{35}ClFNO_2$+Na^+: 238.0042, Found: 238.0043. Anal Calcd. For. $C_9H_7{}^{37}ClFNO_2$+Na^+: 240.0012, Found: 240.0008. **IR** (neat, cm^{-1}): υ 3041, 2930, 1643, 1571, 1446, 1398, 792.

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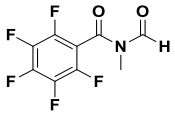
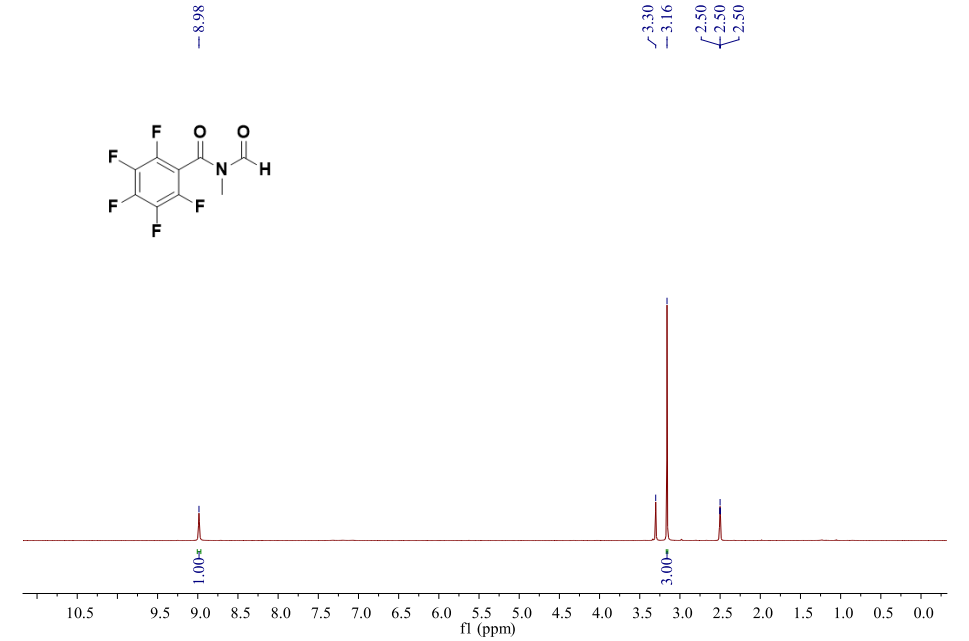
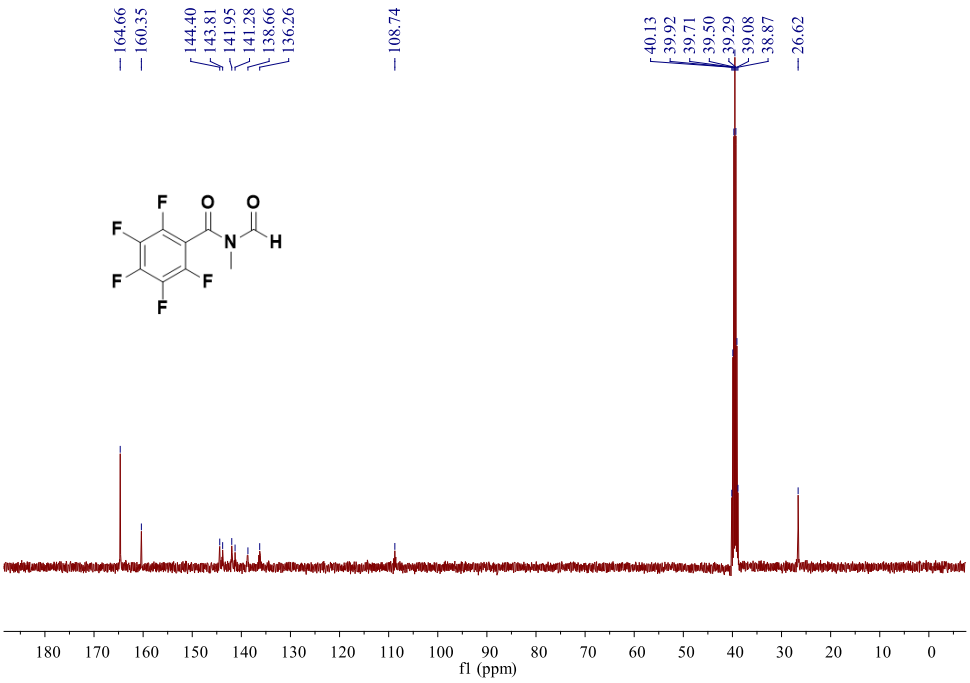
Cl
O
O
N
H
F
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
f1 (ppm)

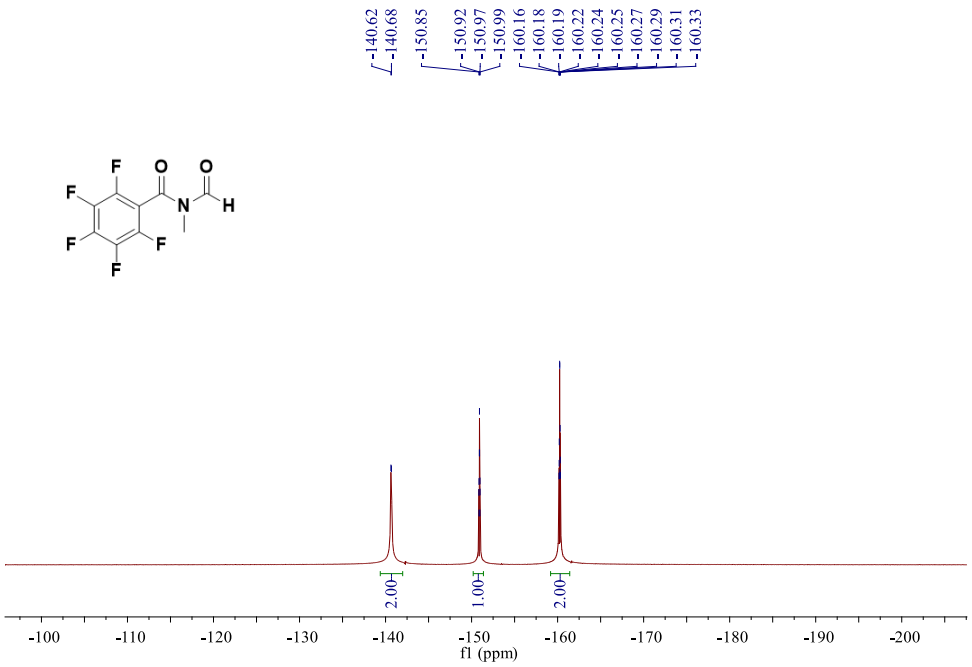


2,3,4,5,6-Pentafluoro-*N*-Formyl-*N*-Methylbenzamide (2o)

petroleum ether / ethyl acetate = 5:1, white solid, 51% yield (25.8 mg). mp: 80 – 82℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.98 (s, 1H), 3.16 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 164.66, 160.35, 143.2 (m), 142.55 (m), 137.46 (m), 108.74 (m), 26.62. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -140.65 (d, *J* = 23.7 Hz, 2F), -150.85 – -152.99 (m, 1F), -160.16 – -160.33 (m, 2F). **HRMS** (EI-TOF): Anal Calcd. For. $C_9H_4F_5NO_2$: 253.0162, Found: 253.0159. **IR** (neat, cm^{-1}): υ 2961, 1742, 1713, 1673, 1503, 1433, 1349.






-140.62
-140.68
-150.85
-150.92
-150.97
-150.99
-160.16
-160.18
-160.19
-160.22
-160.24
-160.25
-160.27
-160.29
-160.31
-160.33
F
O
O
N
H
F
F
F
F
2.00
1.00
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-130
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-150
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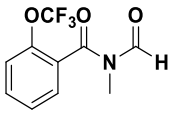
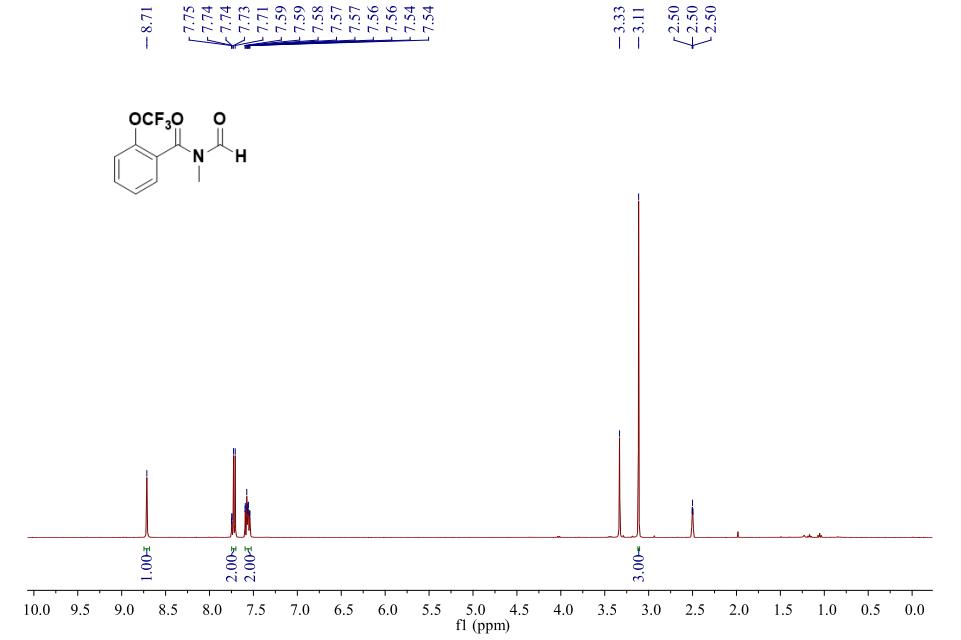
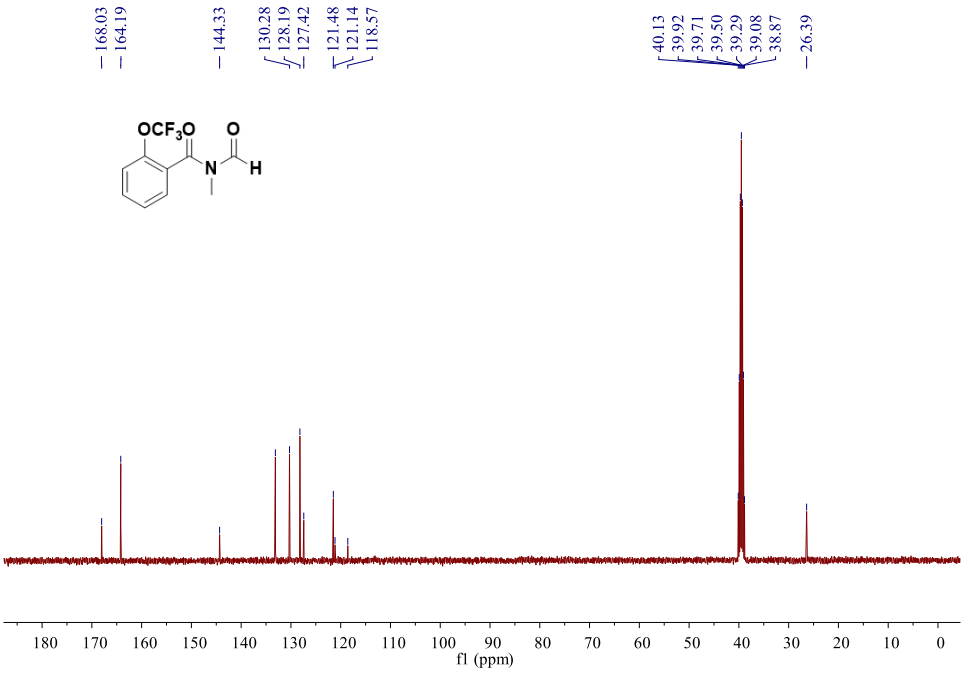
-170
-180
-190
-200
f1 (ppm)

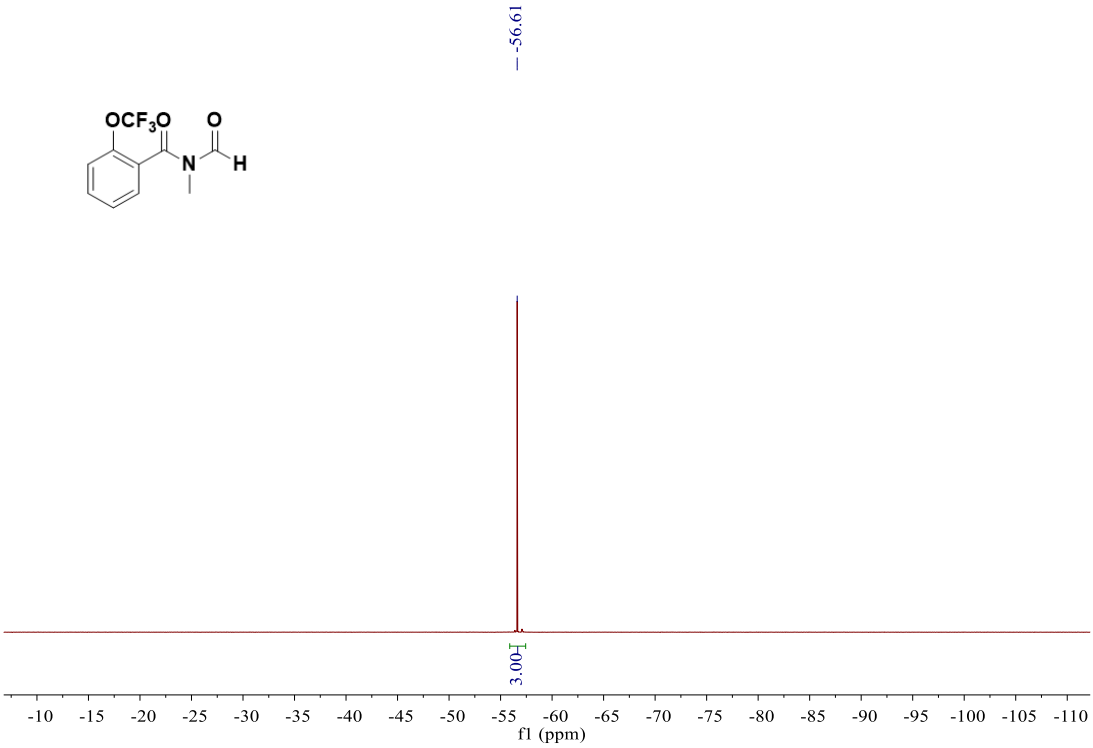


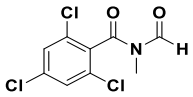
***N*-Formyl-*N*-Methyl-2-(trifluoromethoxy)benzamide (2p)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 60% yield (29.7 mg). mp: 65 – 67℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.71 (s, 1H), 7.75 – 7.71 (m, 2H), 7.59 – 7.54 (m, 2H), 3.11 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 168.03, 164.19, 144.33, 133.12, 130.28, 128.19, 127.42, 121.48, 119.86 (q, *J* = 258.2 Hz), 26.39. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.61 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_8F_3NO_3$+Na^+: 270.0348, Found: 270.0344. **IR** (neat, cm^{-1}): υ 2953, 2850, 1731, 1657, 1485, 1350, 765.



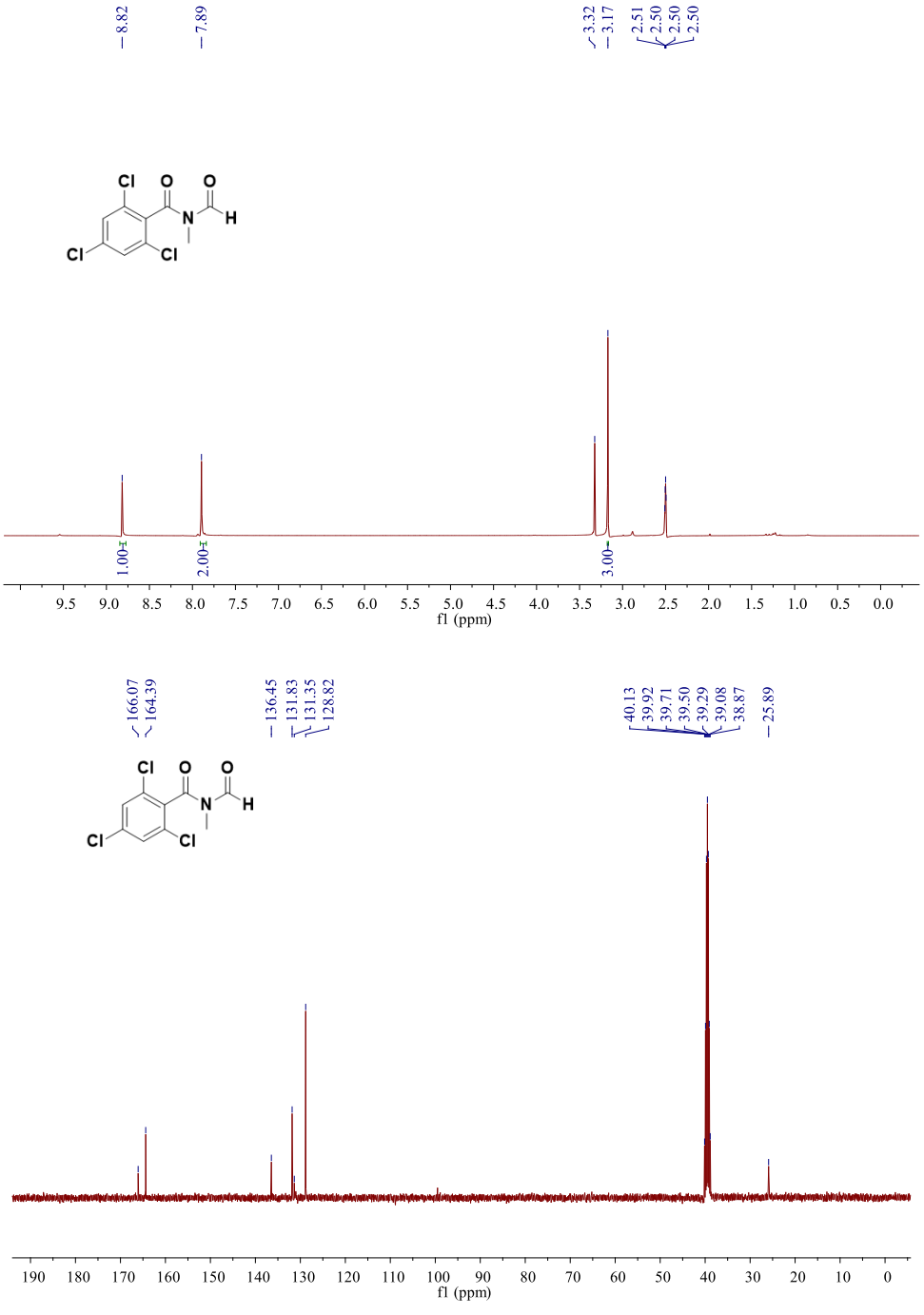


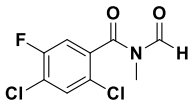

-56.61
OCF3O
O
N
H
3.00
-10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110
f1 (ppm)



2,4,6-Trichloro-*N*-Formyl-*N*-Methylbenzamide (2q)

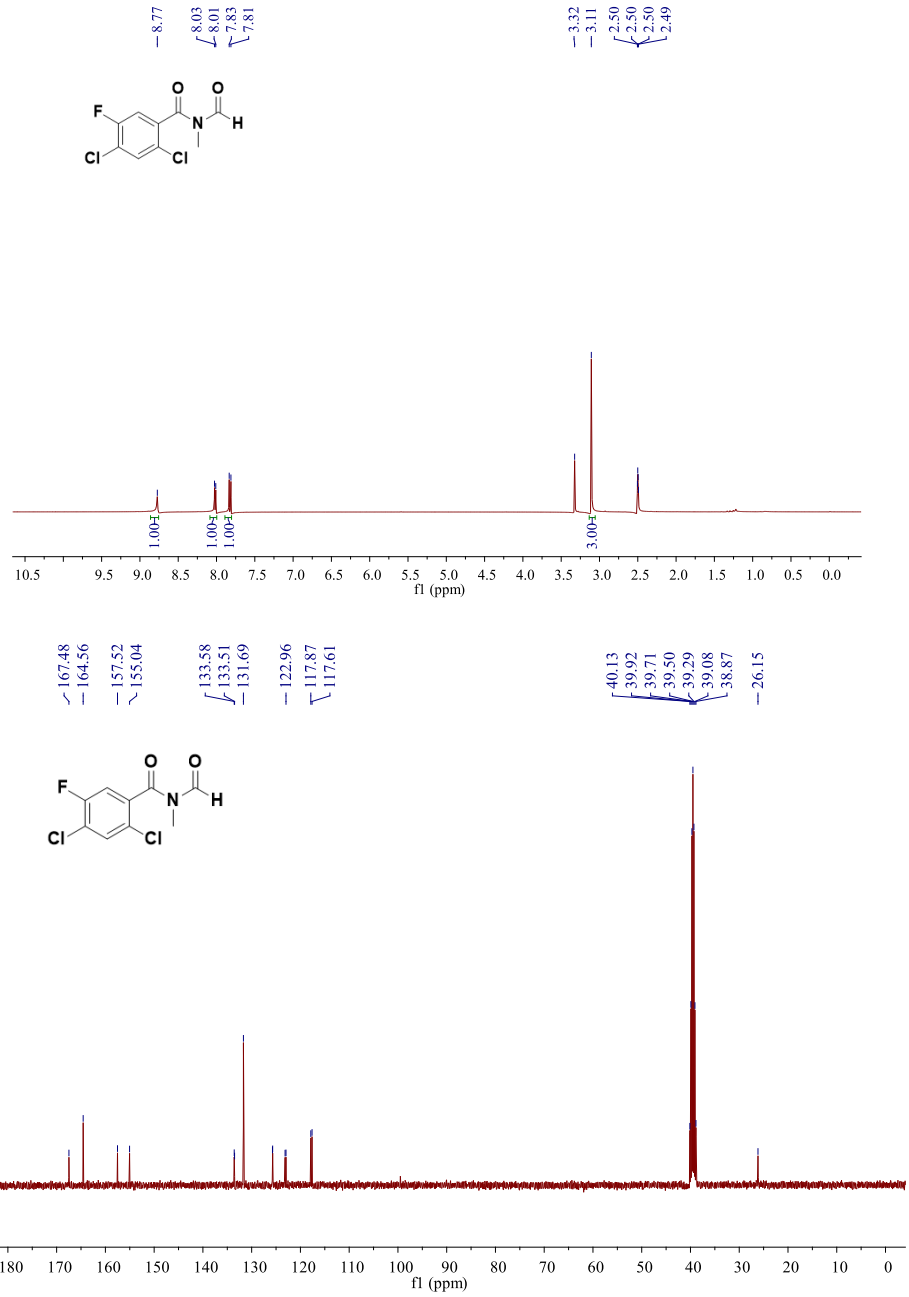
petroleum ether / ethyl acetate = 5:1, white solid, 57% yield (30.2 mg). mp: 65 – 67℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.82 (s, 1H), 7.89 (s, 2H), 3.17 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.07, 164.39, 136.45, 131.83, 131.35, 128.82, 25.89. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6{}^{35}Cl_3NO_2$+Na^+: 287.9356, Found: 287.9425. Anal Calcd. For. $C_9H_6{}^{35,35,37}Cl_3NO_2$+$Na^+$: 289.9327, Found: 289.9319. **IR** (neat, cm^{-1}): υ 3066, 2932, 1715, 1637, 1573, 1428, 1370, 886.

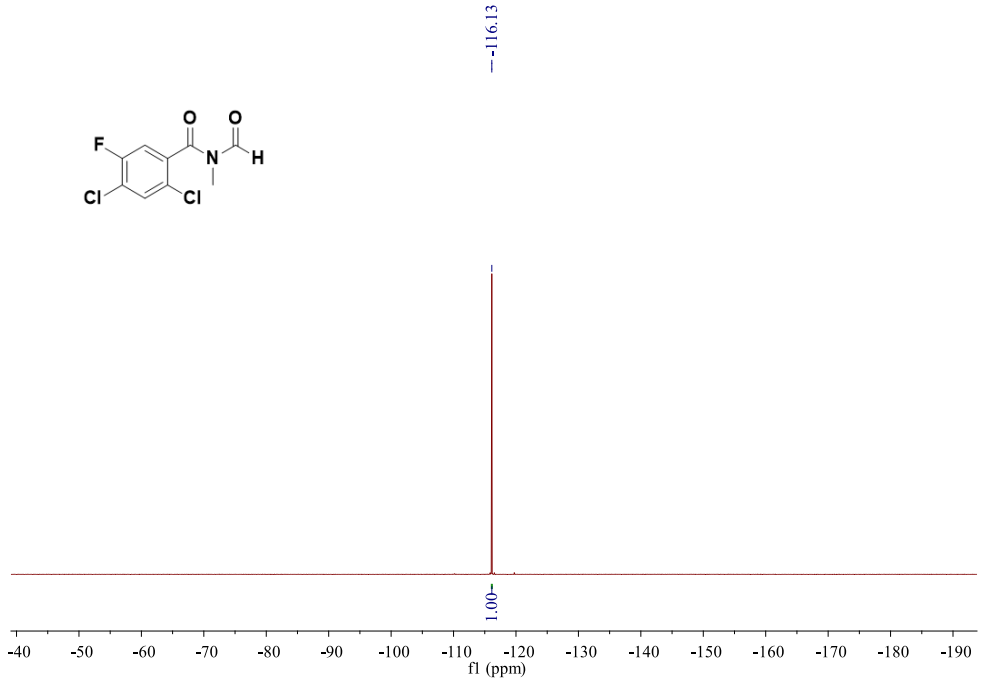




2,4-Dichloro-5-Fluoro-*N*-Formyl-*N*-Methylbenzamide (2r)

petroleum ether / ethyl acetate = 5:1, white solid, 71% yield (49.8 mg). mp: 55 – 57℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.77 (s, 1H), 8.02 (d, *J* = 6.5 Hz, 1H), 7.82 (d, *J* = 9.0 Hz, 1H), 3.11 (s, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 167.48, 164.56, 156.28 (d, *J* = 249.0 Hz), 133.54 (d, *J* = 7.2 Hz), 131.69, 125.71 (d, *J* = 3.8 Hz), 123.05 (d, *J* = 19.0 Hz), 117.74 (d, *J* = 25.5 Hz), 26.15. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -116.13 (s, 1F). **HRMS** (EI-TOF): Anal Calcd. For. $C_9H_6Cl_2FNO_2$: 248.9760, Found: 248.9753. Anal Calcd. For. $C_9H_6{}^{35}Cl^{37}ClFNO_2$: 250.9730, Found: 250.9733. Anal Calcd. For. $C_9H_6{}^{37}Cl_2FNO_2$: 252.9701, Found: 252.9735. **IR** (neat, cm^{-1}): υ 3070, 1730, 1682, 1511, 1475, 1384, 876.




-116.13
F
O
O
N
H
Cl
Cl
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-50
-60
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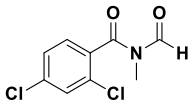
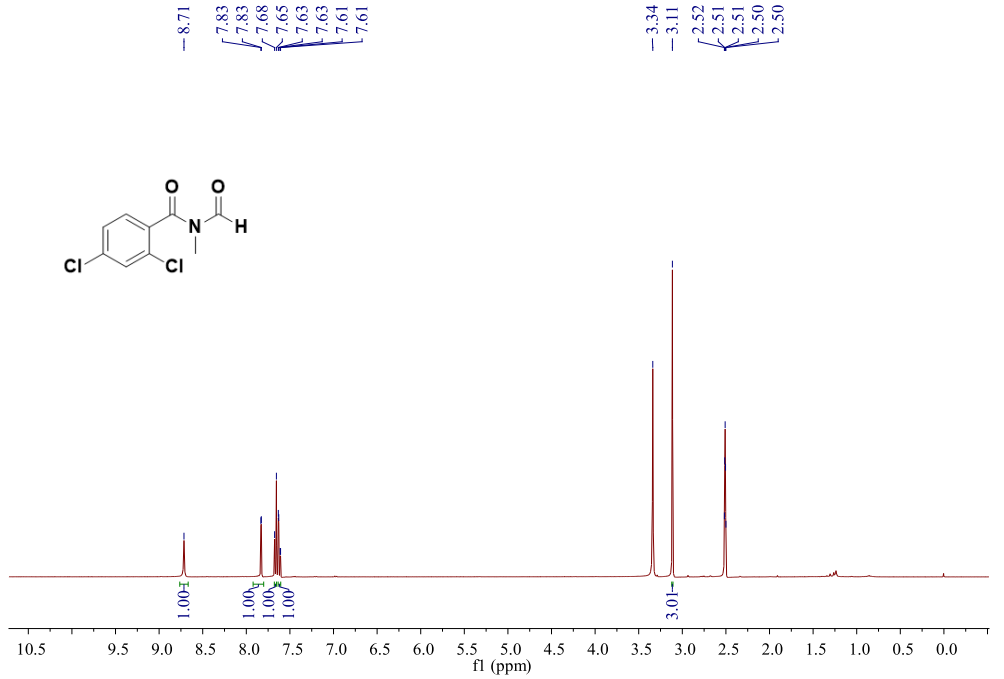
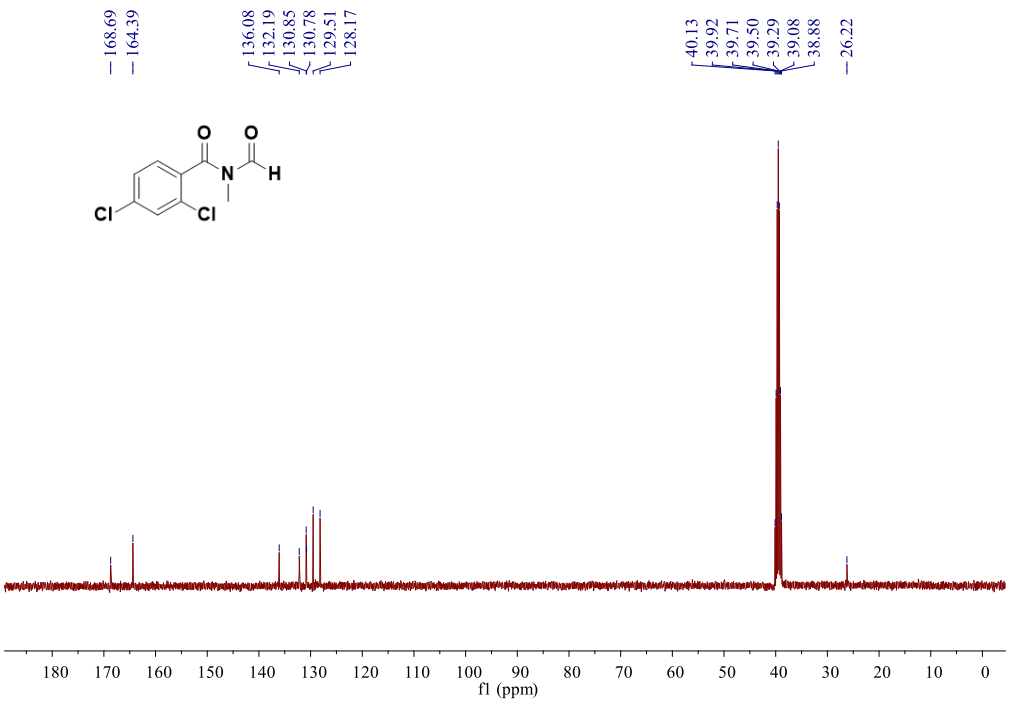
-150
-160
-170
-180
-190
f1 (ppm)

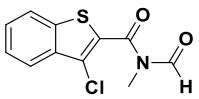


2,4-Dichloro-*N*-Formyl-*N*-Methylbenzamide (2s)

petroleum ether / ethyl acetate = 5:1, white solid, 62% yield (28.6 mg). mp: 148 – 150℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.71 (s, 1H), 7.83 (d, *J* = 1.9 Hz, 1H), 7.67 (d, *J* = 8.3 Hz, 1H), 7.62 (dd, *J* = 8.3, 1.9 Hz, 1H), 3.11 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 168.69, 164.39, 136.08, 132.19, 130.85, 130.78, 129.51, 128.17, 26.22. **HRMS** (EI-TOF): Anal Calcd. For. $C_9H_7Cl_2NO_2$: 230.9854, Found: 230.9860. **IR** (neat, cm^{-1}): υ 3019, 2934, 1718, 1630, 1587, 1458, 1372, 868, 797.

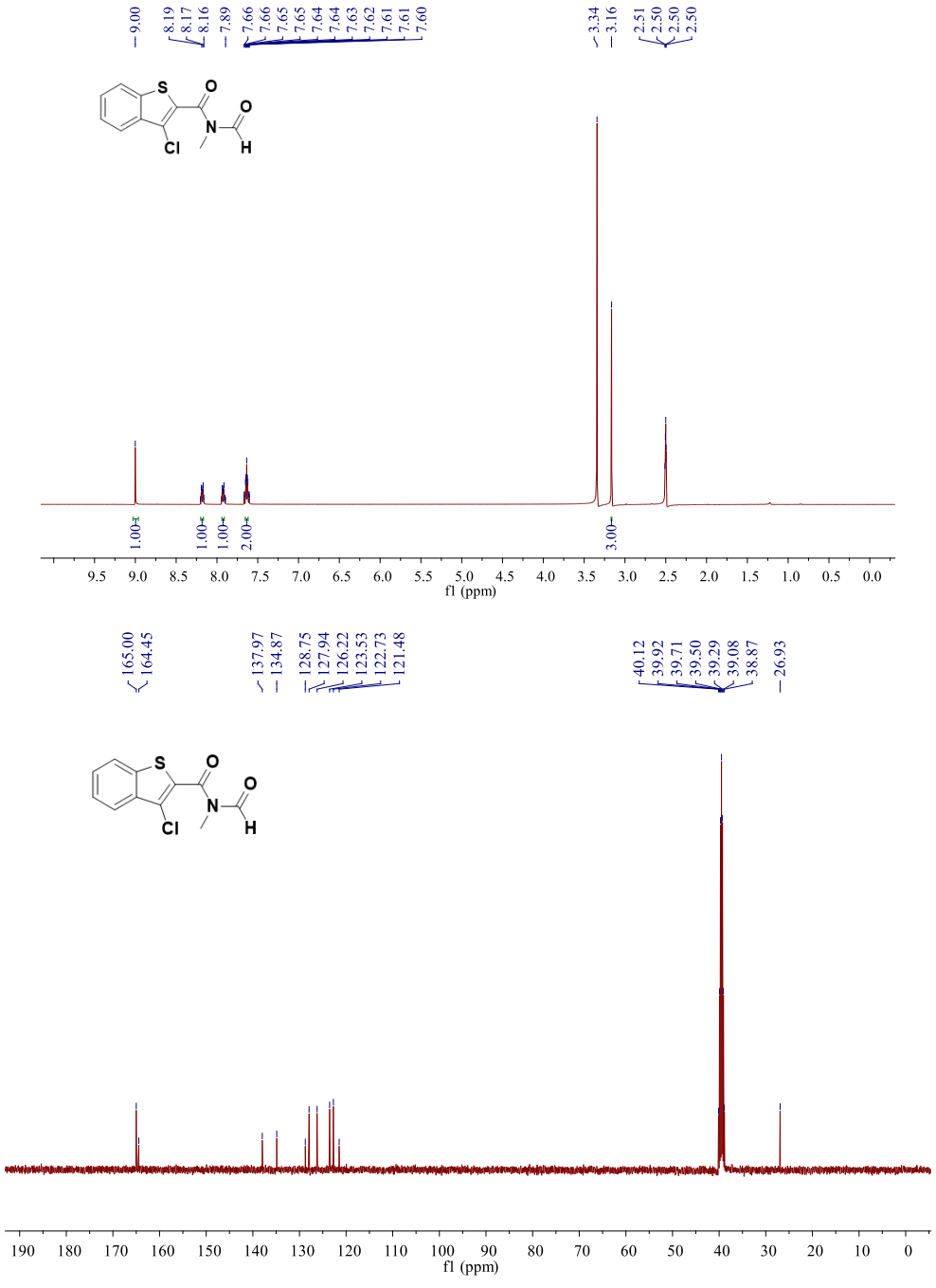


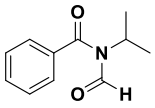




3-Chloro-*N*-Formyl-*N*-Methylbenzo[b]thiophene-2-Carboxamide (2t)

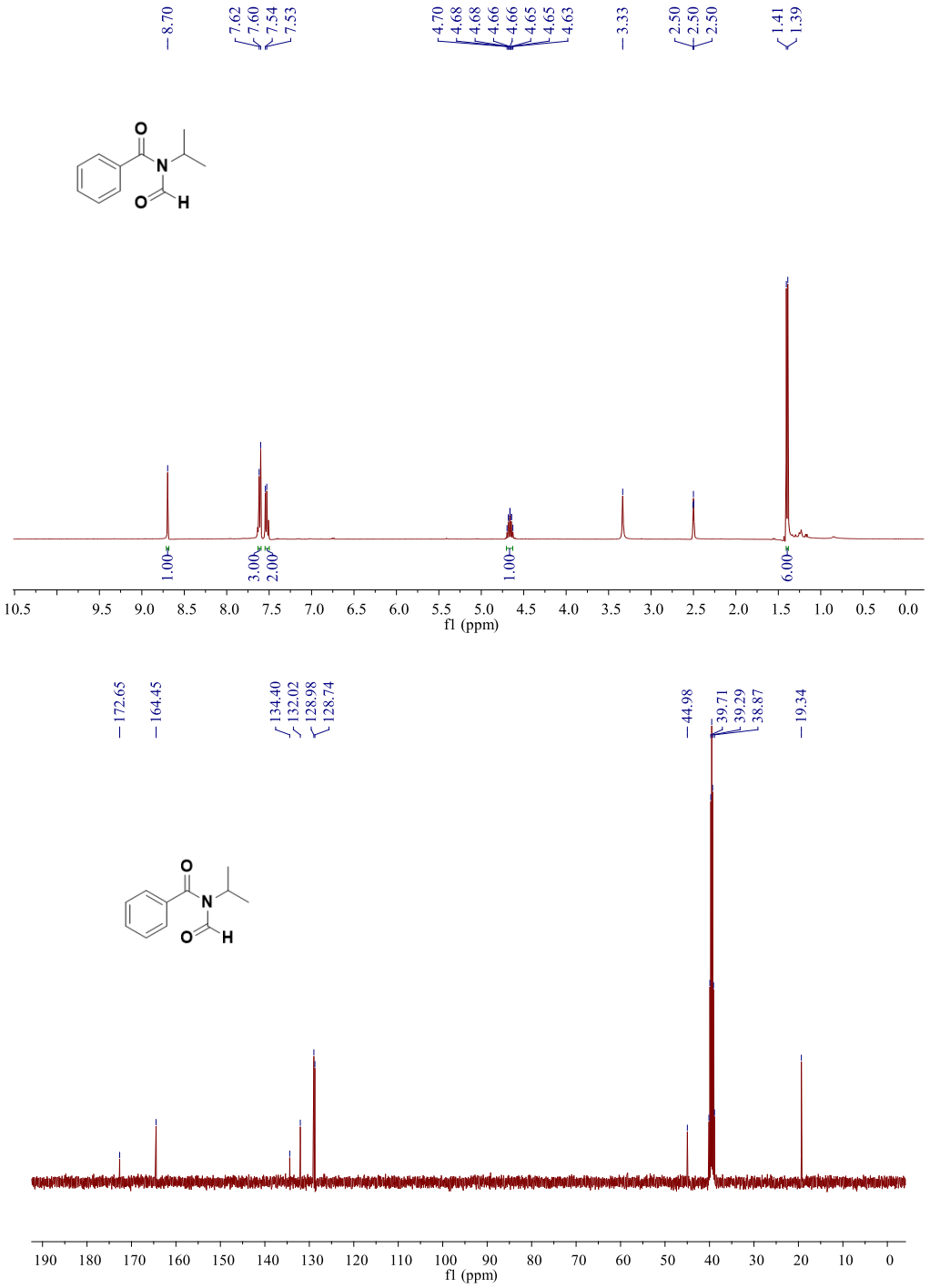
petroleum ether / ethyl acetate = 5:1, white solid, 38% yield (19.2 mg). mp: 57 – 59℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.00 (s, 1H), 8.20 – 8.16 (m, 1H), 7.94 – 7.89 (m, 1H), 7.67 – 7.60 (m, 2H), 3.16 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.00, 164.45, 137.97, 134.87, 128.75, 127.94, 126.22, 123.53, 122.73, 121.48, 26.93. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_8{}^{35}ClNO_2S+Na^+$: 275.9856, Found: 275.9851. Anal Calcd. For. $C_{11}H_8{}^{37}ClNO_2S+Na^+$: 277.9827, Found: 277.9808. **IR** (neat, cm^{-1}): υ 3079, 2951, 1659, 1591, 1453, 1305, 730.

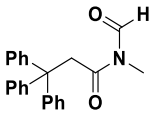




***N*-Formyl-*N*-Isopropylbenzamide (2u)**

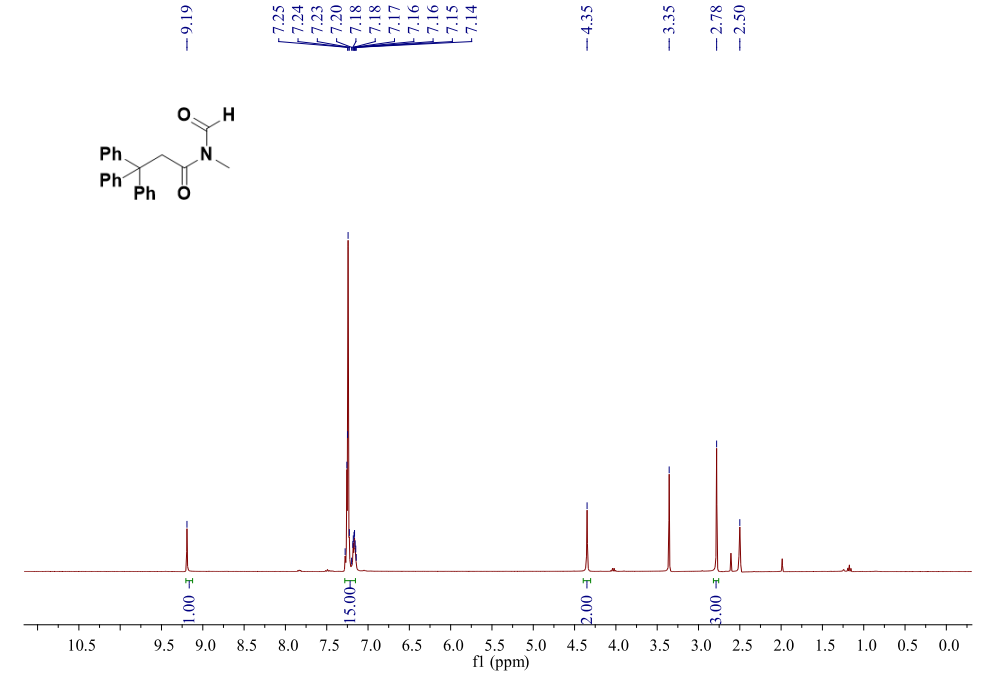
petroleum ether / ethyl acetate = 5:1, white solid, 51% yield (19.5 mg). mp: 67 – 69℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.70 (s, 1H), 7.64 – 7.60 (m, 3H), 7.54 – 7.51 (m, 2H), 4.70 – 4.63 (m, 1H), 1.40 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 172.65, 164.45, 134.40, 132.02, 128.98, 128.74, 44.98, 19.34. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{13}NO_2$+Na^+: 214.0838, Found: 214.0824. **IR** (neat, cm^{-1}): υ 3071, 2937, 1719, 1661, 1528, 1407, 1298, 758, 692.

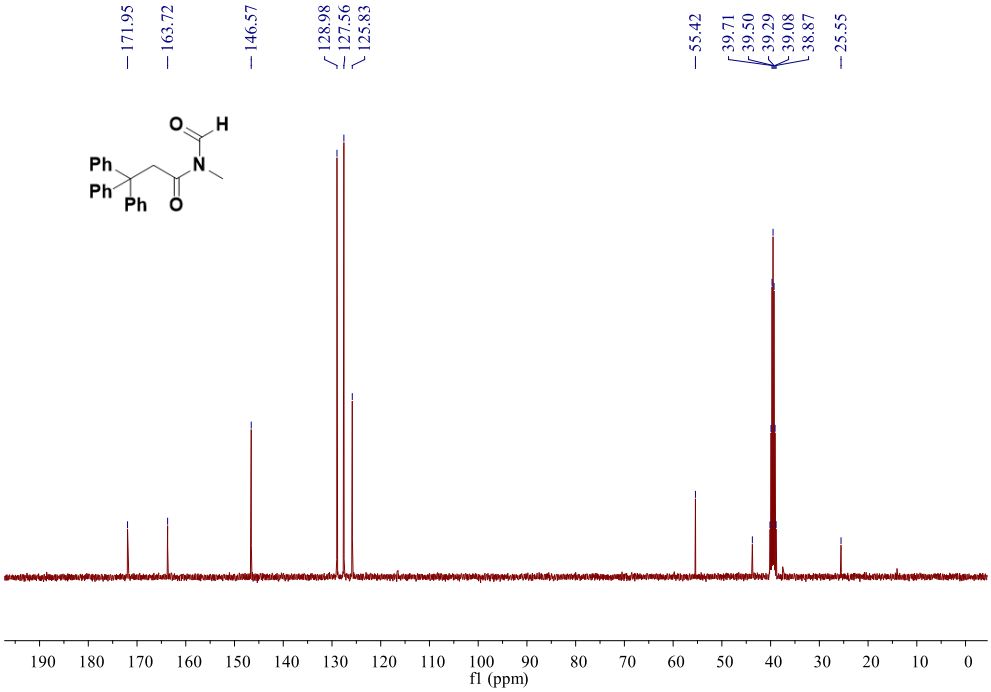


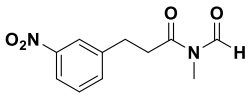


***N*-Formyl-*N*-Methyl-3,3,3-Triphenylpropanamide (2v)**

petroleum ether / ethyl acetate = 5:1, white solid, 77% yield (52.8 mg). mp: 86 – 88℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.19 (s, 1H), 7.25 – 7.14 (m, 15H), 4.35 (s, 2H), 2.78 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.95, 163.72, 146.57, 128.98, 127.56, 125.83, 55.42, 43.74, 25.55. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{23}H_{21}NO_2$+Na^+: 338.1465, Found: 338.1469. **IR** (neat, cm^{-1}): υ 3058, 2935, 1732, 1671, 1569, 1446, 1361, 744, 690.

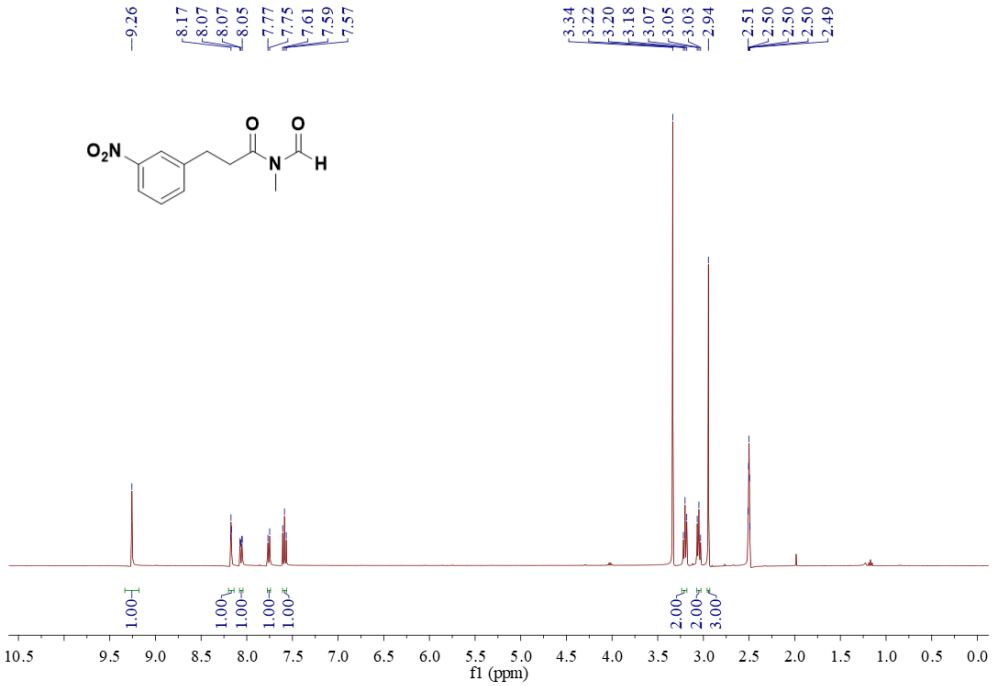


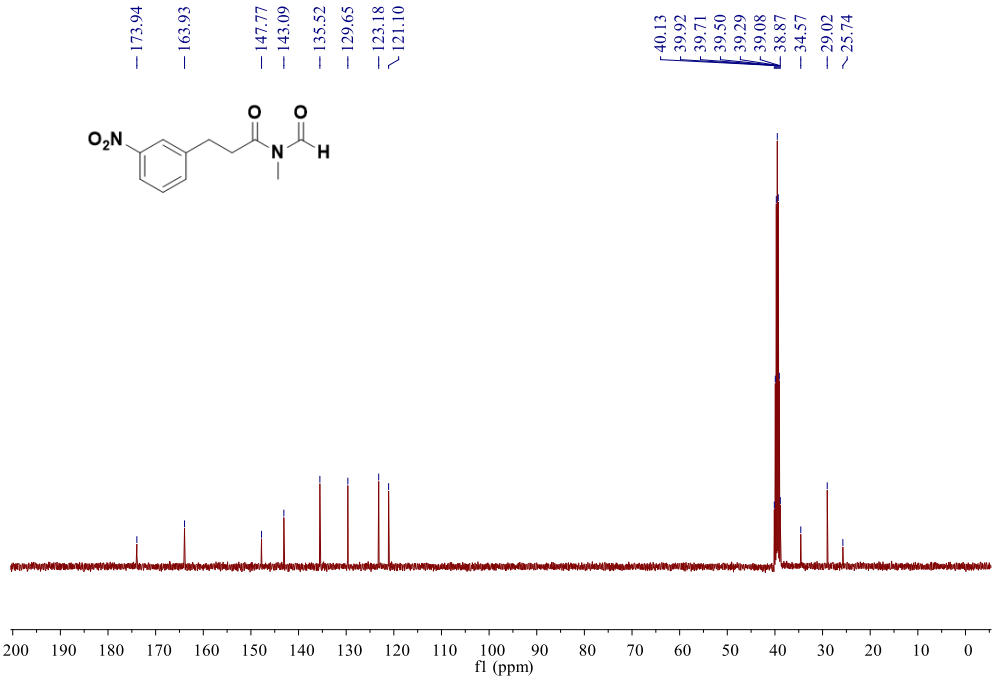


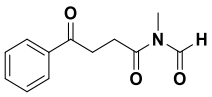


***N*-Formyl-*N*-Methyl-3-(3-Nitrophenyl)propenamide (2w)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 55% yield (26.0 mg). mp: 49 – 51℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.26 (s, 1H), 8.17 (t, *J* = 1.8 Hz, 1H), 8.07 – 8.05 (m, 1H), 7.76 (d, *J* = 7.7 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 1H), 3.20 (t, *J* = 7.4 Hz, 2H), 3.05 (t, *J* = 7.4 Hz, 2H), 2.94 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 173.94, 163.93, 147.77, 143.09, 135.52, 129.65, 123.18, 121.10, 34.57, 29.02, 25.74. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{12}N_2O_4$+Na^+: 259.0689, Found: 259.0688. **IR** (neat, cm^{-1}): υ 3091, 0949, 1737, 1674, 1522, 1417, 1345, 889, 780.

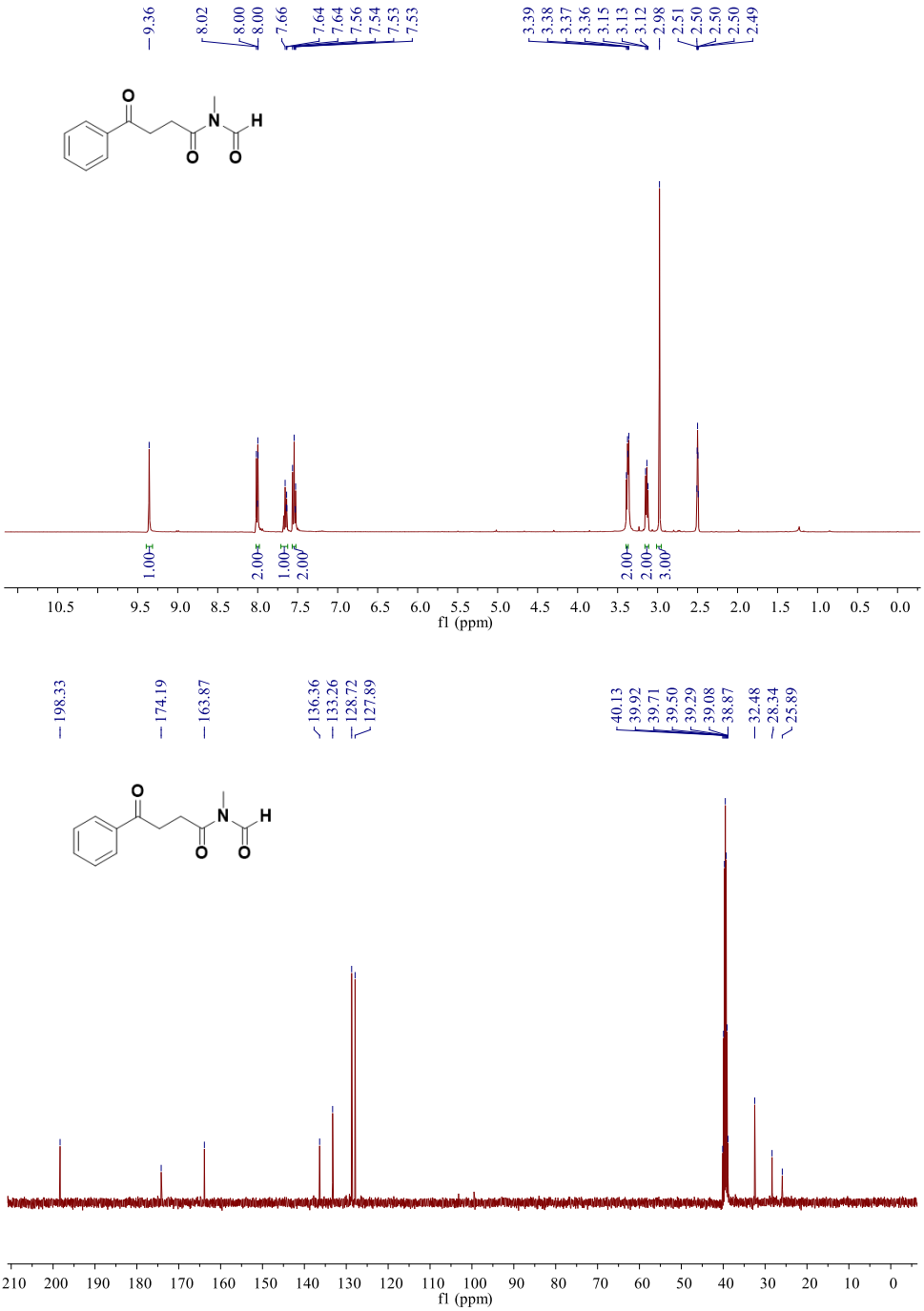


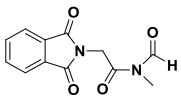




***N*-Formyl-*N*-Methyl-4-Oxo-4-Phenylbutanamide (2x)**

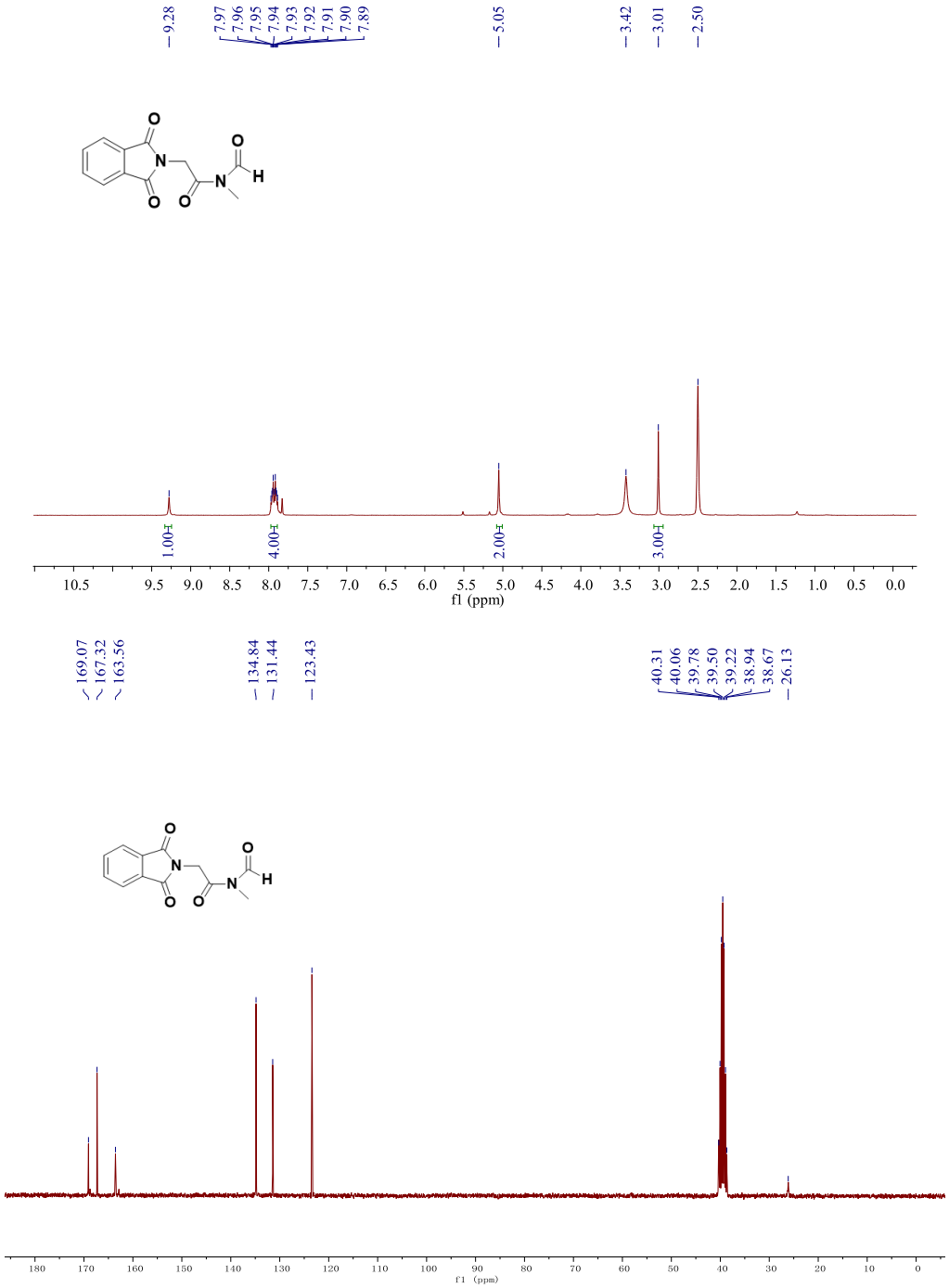
petroleum ether / ethyl acetate = 5:1, white solid, 54% yield (23.7 mg). mp: 76 – 78℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.36 (s, 1H), 8.02 – 8.00 (m, 2H), 7.66 – 7.64 (m, 1H), 7.56 – 7.53 (m, 2H), 3.39 – 3.37 (m, 2H), 3.15 – 3.12 (m, 2H), 2.98 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 198.33, 174.19, 163.87, 136.36, 133.26, 128.72, 127.89, 32.48, 28.34, 25.89. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{12}H_{13}NO_3$+Na^+: 242.0788, Found: 242.0794. **IR** (neat, cm^{-1}): υ 3062, 1716, 1682, 1581, 1451, 1333, 800, 747.

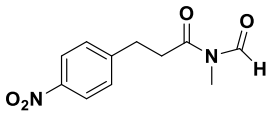




2-(1,3-dioxoisoindolin-2-yl)-*N*-Formyl-*N*-Methylacetamide (2y)

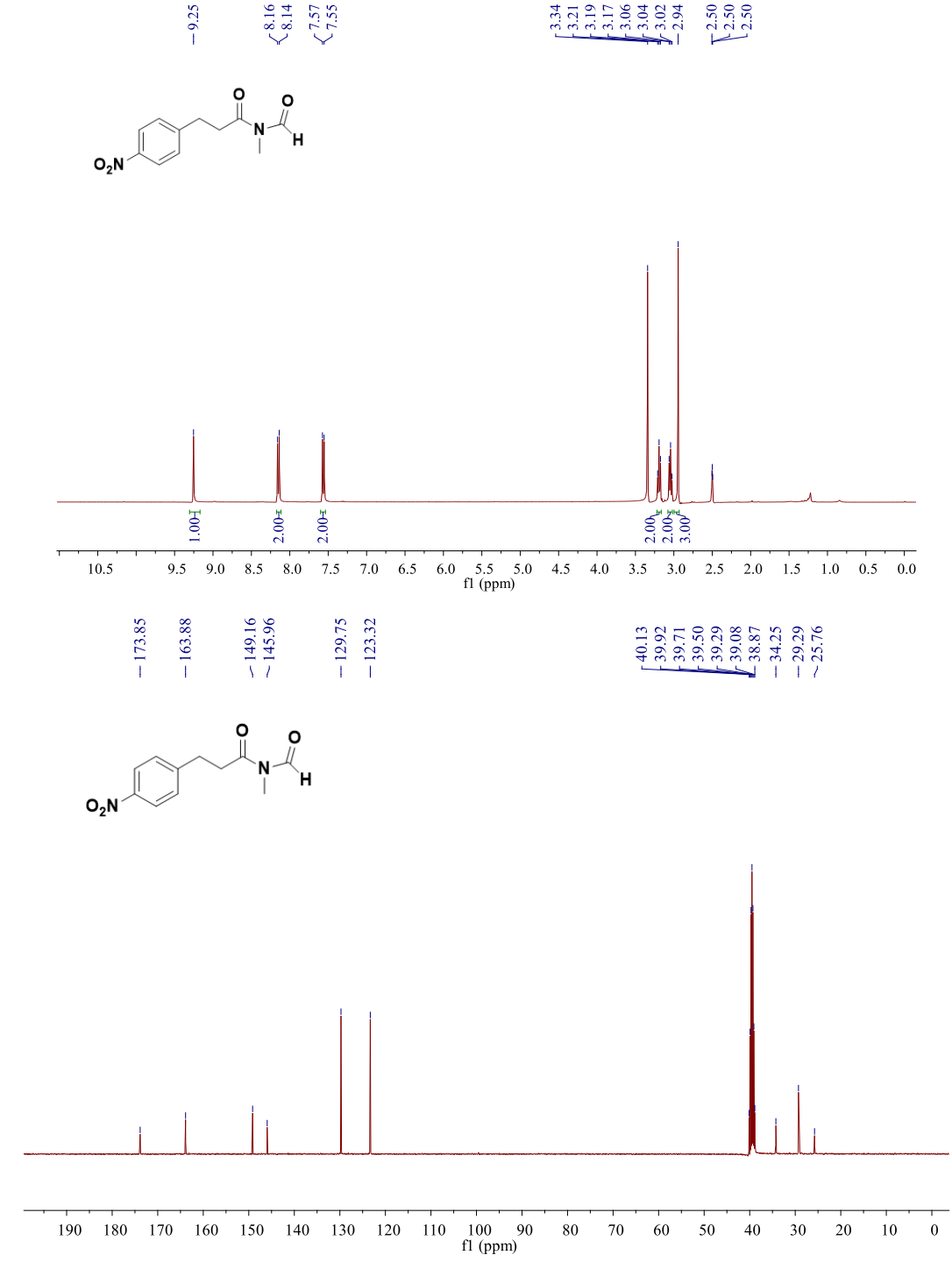
petroleum ether / ethyl acetate = 5:1, white solid, 71% yield (34.9 mg). mp: 162 – 164℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 9.28 (s, 1H), 8.09 – 7.85 (m, 4H), 5.05 (s, 2H), 3.01 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 169.07, 167.32, 163.56, 134.84, 131.44, 123.43, 26.13. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{12}H_{10}N_2O_4$+Na^+: 269.0540, Found: 269.0540. **IR** (neat, cm^{-1}): υ 2946, 2852, 1707, 1671, 1396, 832, 714.

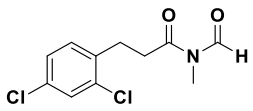




***N*-Formyl-*N*-Methyl-3-(4-nitrophenyl)propenamide (2z)**

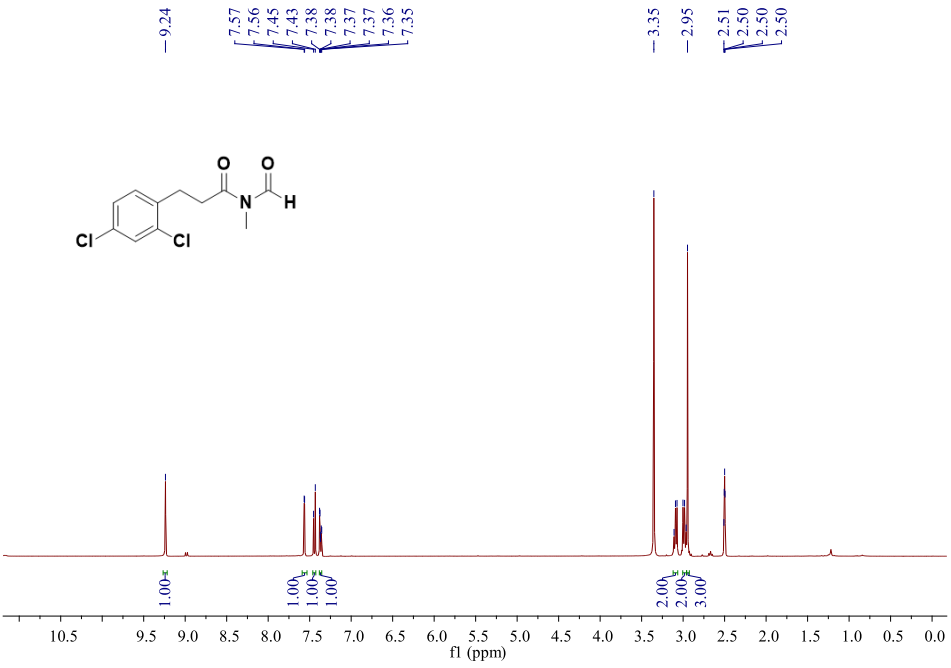
petroleum ether / ethyl acetate = 5:1, yellow solid, 70% yield (33.1 mg). mp: 70 – 72℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.25 (s, 1H), 8.15 (d, *J* = 8.6 Hz, 2H), 7.56 (d, *J* = 8.6 Hz, 2H), 3.19 (t, *J* = 7.4 Hz, 2H), 3.04 (t, *J* = 7.4 Hz, 2H), 2.94 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 173.85, 163.88, 149.16, 145.96, 129.75, 123.32, 34.25, 29.29, 25.76. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{12}N_2O_4$+Na^+: 259.0689, Found: 259.0693. **IR** (neat, cm^{-1}): υ 3069, 2912, 1715, 1686, 1511, 1450, 1344, 798.

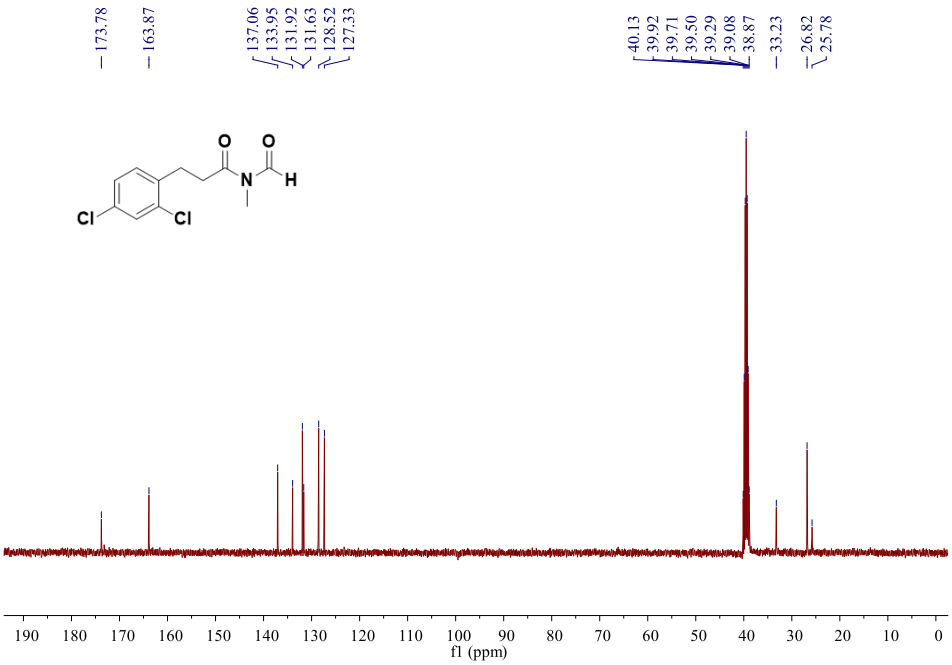


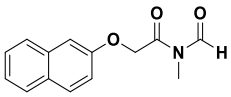


3-(2,4-Dichlorophenyl)-*N*-Formyl-*N*-Methylpropanamide (2aa)

petroleum ether / ethyl acetate = 5:1, colorless oil, 72% yield (37.3 mg). **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.24 (s, 1H), 7.56 (d, *J* = 2.1 Hz, 1H), 7.44 (d, *J* = 8.3 Hz, 1H), 7.38 – 7.35 (m, 1H), 3.11 – 3.07 (m, 2H), 3.00 – 2.96 (m, 2H), 2.95 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 173.78, 163.87, 137.06, 133.95, 131.92, 131.63, 128.52, 127.33, 33.23, 26.82, 25.78. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{11}{}^{35}Cl_2NO_2$+$Na^+$: 282.0059, Found: 282.0059. Anal Calcd. For. $C_{11}H_{11}{}^{35,37}Cl_2NO_2$+$Na^+$: 284.0030, Found: 284.0015. **IR** (neat, cm^{-1}): υ 2934, 1735, 1641, 1587, 1472, 1344, 865, 748.

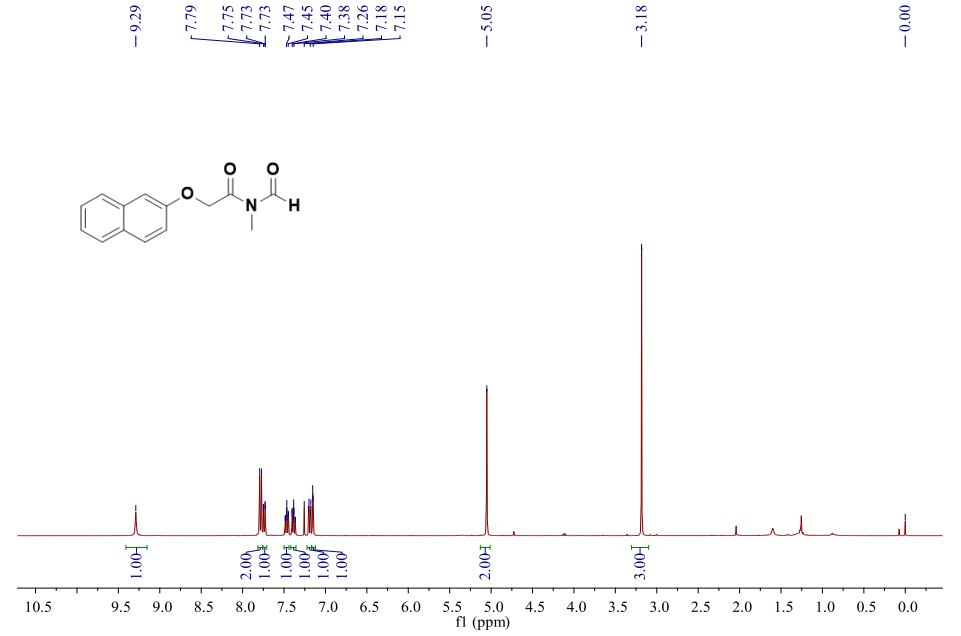


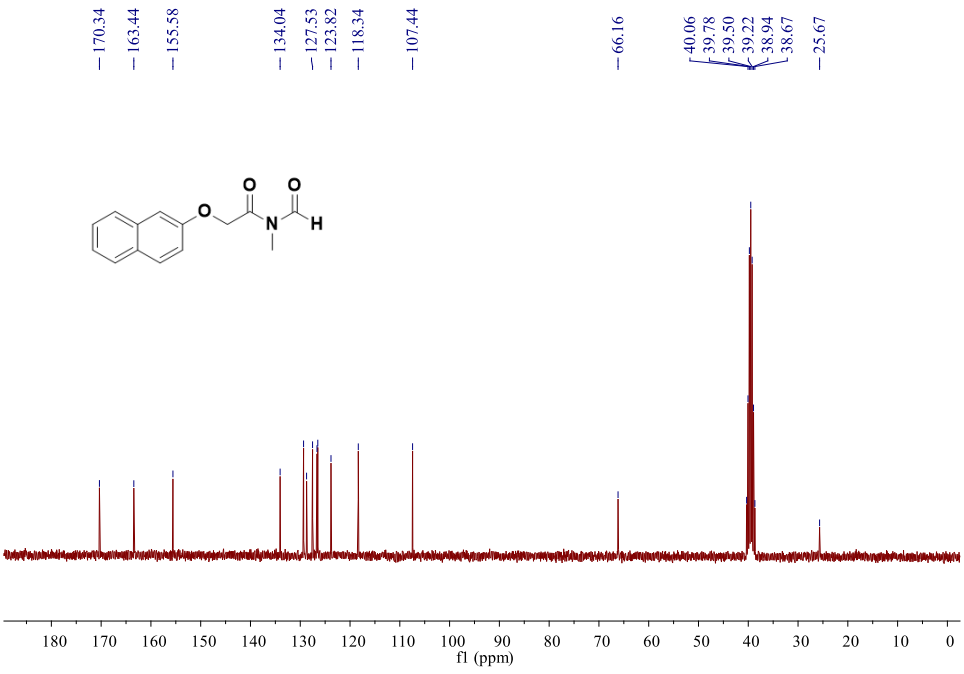


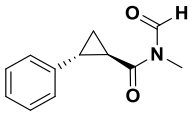


***N*-Formyl-*N*-Methyl-2-(naphthalen-2-yloxy)acetamide (2ab)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 35% yield (17.0 mg). mp: 79 – 80℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.29 (s, 1H), 7.78 (d, *J* = 8.7 Hz, 2H), 7.74 (dd, *J* = 8.3, 1.1 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.40 – 7.36 (m, 1H), 7.21 – 7.18 (m, 1H), 7.15 (d, *J* = 2.7 Hz, 1H), 5.05 (s, 2H), 3.18 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 170.34, 163.44, 155.58, 134.04, 129.33, 128.73, 127.53, 126.64, 126.48, 123.82, 118.34, 107.44, 66.16, 25.67. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{14}H_{13}NO_3$+Na^+: 266.0788, Found: 266.0781. **IR** (neat, cm^{-1}): υ 3057, 2919, 1729, 1680, 1509, 1469, 1253, 810, 744.

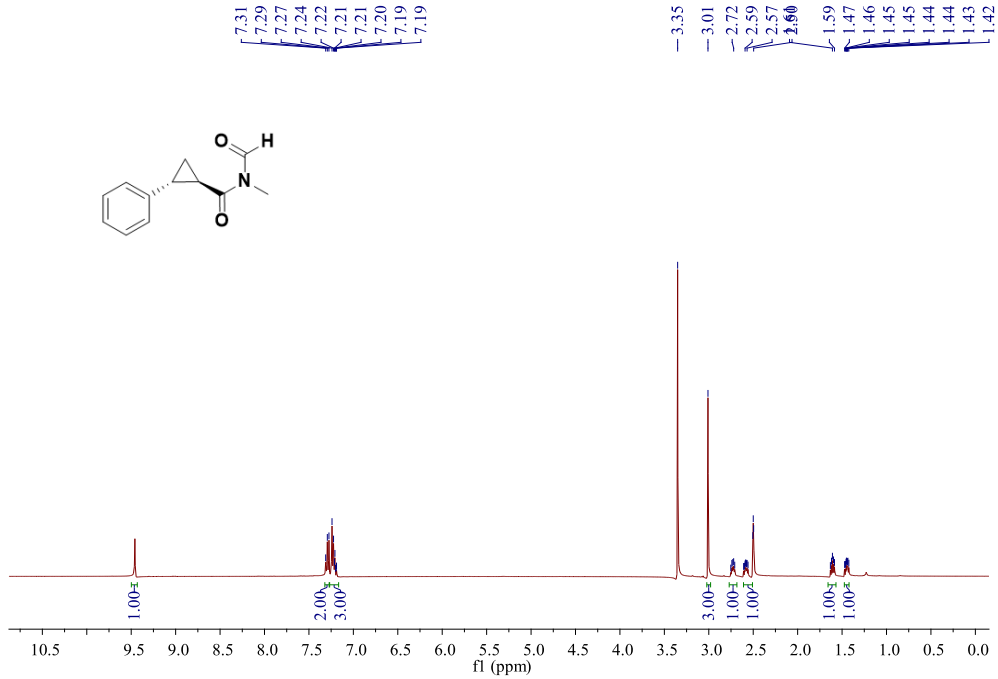


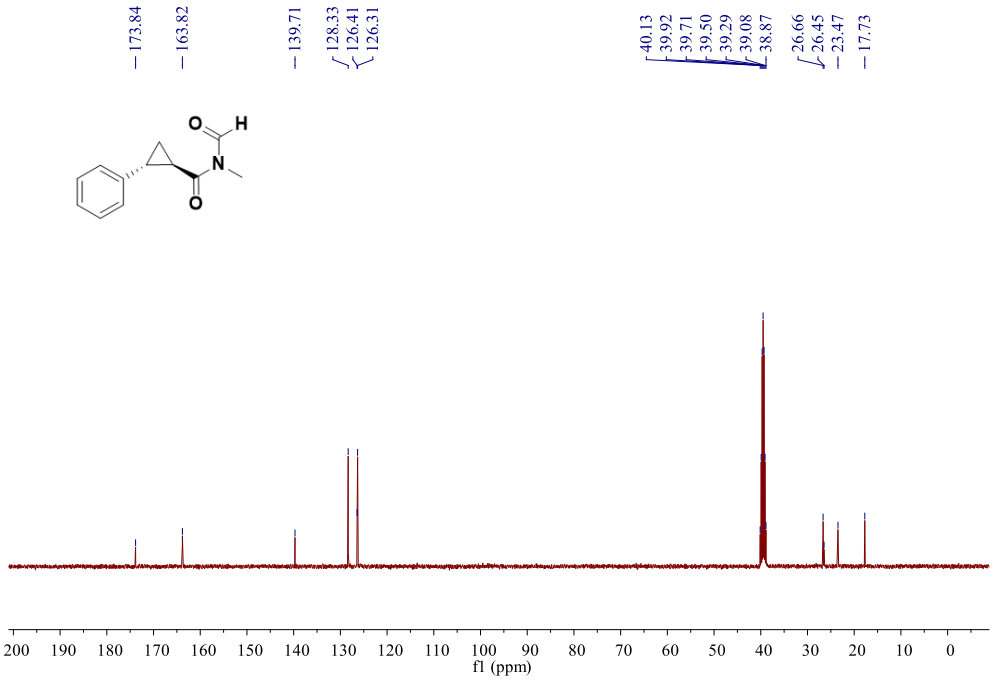


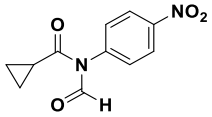


(1R,2R)-*N*-Formyl-*N*-Methyl-2-Phenylcyclopropane-1-Carboxamide (2ac)

petroleum ether / ethyl acetate = 5:1, white solid, 61% yield (24.8 mg). mp: 55 –57°C. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.45 (d, *J* = 9.8 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.24 – 7.19 (m, 3H), 3.01 (s, 3H), 2.74 – 2.71 (m, 1H), 2.59 – 2.56 (m, 1H), 1.63 – 1.59 (m, 1H), 1.47 – 1.42 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 173.84, 163.82, 139.71, 128.33, 126.41, 126.31, 26.66, 26.45, 23.47, 17.73. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{12}H_{13}NO_2$+Na^+: 226.0838, Found: 226.0842. **IR** (neat, cm^{-1}): υ 3062, 2927, 1771, 1626, 1496, 1319, 720, 699.

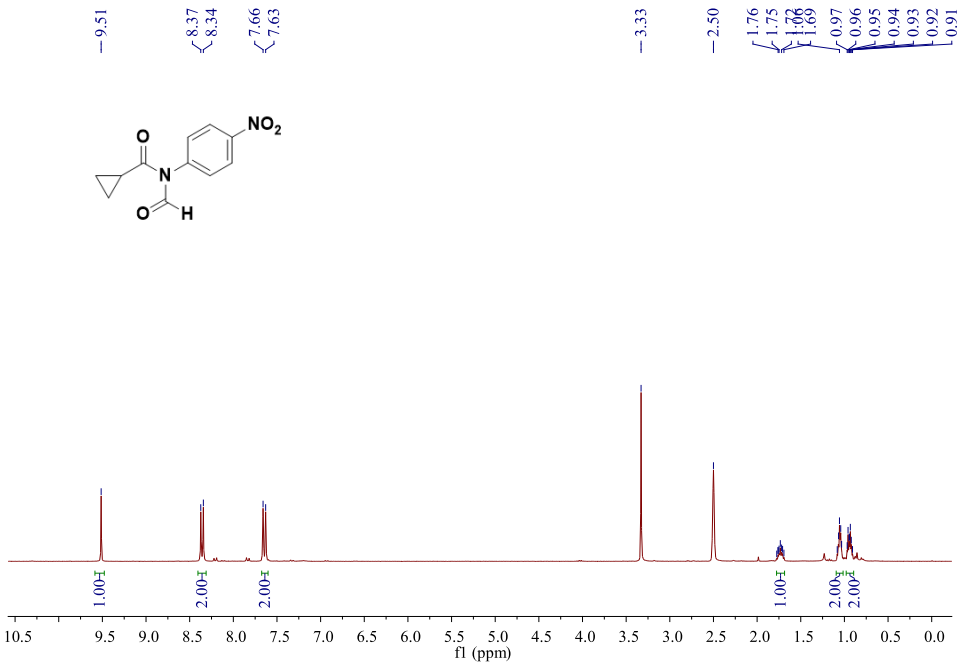


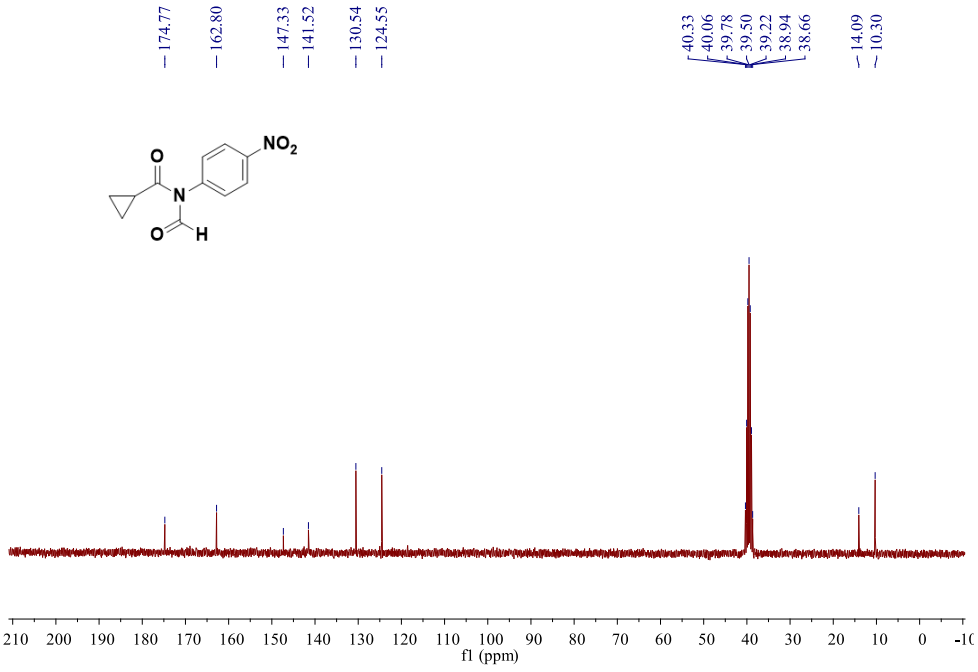


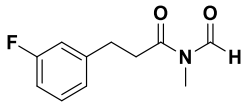


***N*-Formyl-*N*-(4-nitrophenyl)cyclopropanecarboxamide (2ad)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 63% yield (29.5 mg). mp: 76 – 77℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 9.51 (s, 1H), 8.36 (d, *J* = 8.9 Hz, 2H), 7.64 (d, *J* = 8.9 Hz, 2H), 1.77 – 1.69 (m, 1H), 1.08 – 1.03 (m, 2H), 0.97 – 0.91 (m, 2H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 174.77, 162.80, 147.33, 141.52, 130.54, 124.55, 14.09, 10.30. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{10}N_2O_4$+Na^+: 257.0533, Found: 257.0524. **IR** (neat, cm^{-1}): υ 3085, 2942, 1730, 1654, 1557, 1447, 1358, 855, 799.

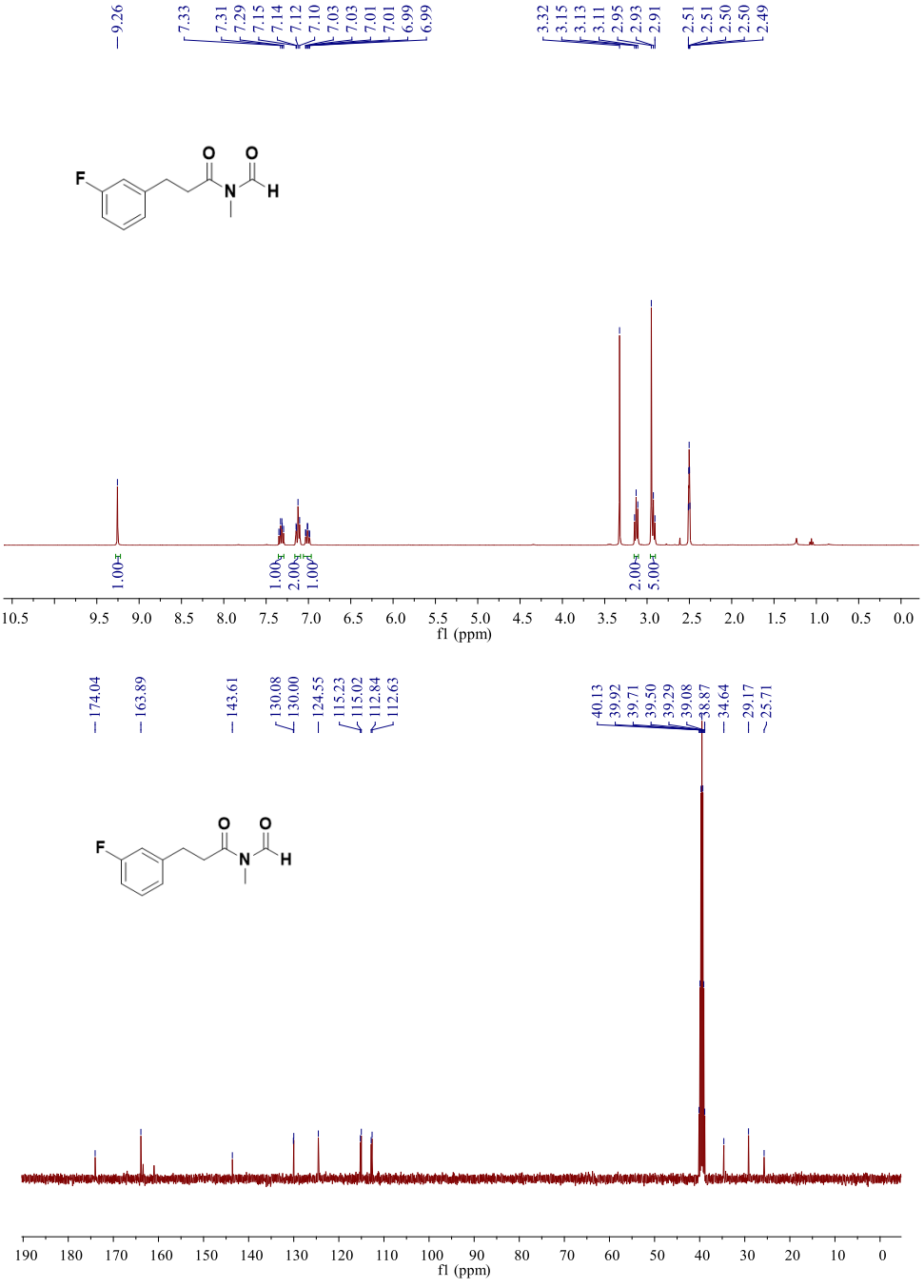


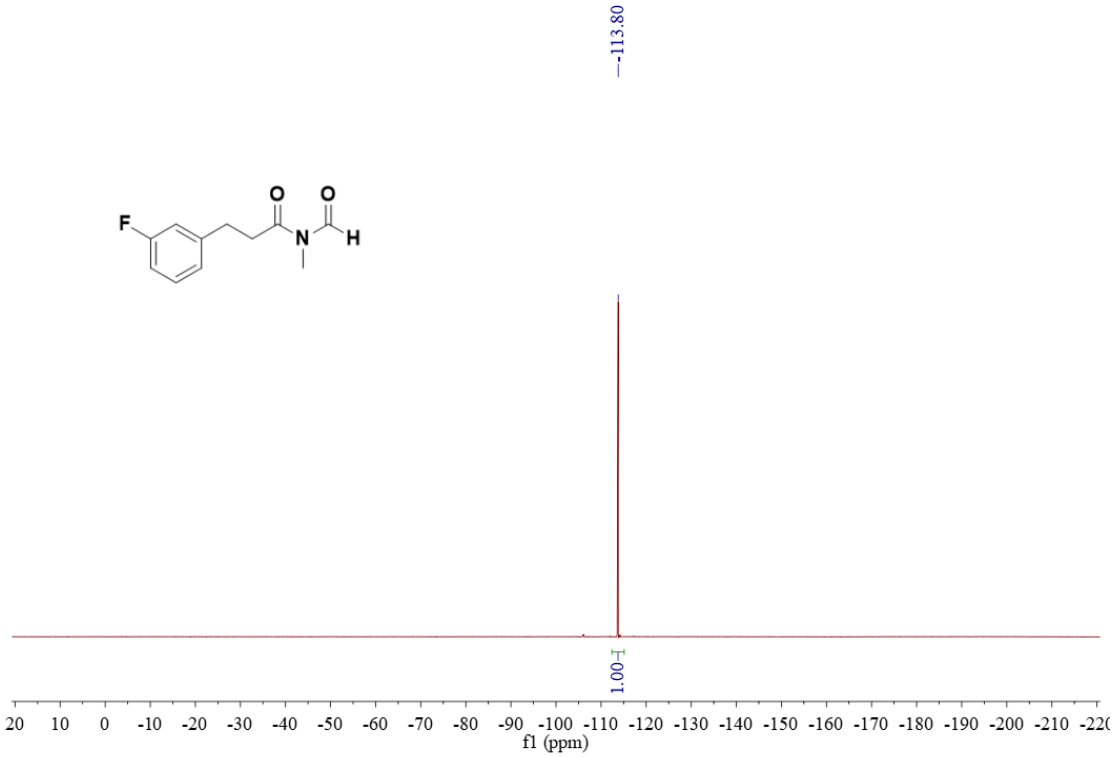


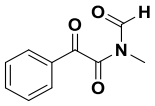


3-(3-Fluorophenyl)-*N*-Formyl-N-Methylpropanamide (2ae)

petroleum ether / ethyl acetate = 5:1, yellow oil, 50% yield (20.9 mg). **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.26 (s, 1H), 7.35 – 7.29 (m, 1H), 7.15 – 7.10 (m, 2H), 7.03 – 6.99 (m, 1H), 3.13 (t, *J* = 7.6 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.95 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 174.04, 163.89, 162.18 (d, *J* = 241.1 Hz), 143.57 (d, *J* = 7.4 Hz), 130.04 (d, *J* = 8.5 Hz), 124.56 (d, *J* = 2.2 Hz), 115.12 (d, *J* = 21.1 Hz), 112.73 (d, *J* = 20.9 Hz), 34.64, 29.17, 25.71. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -113.80 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{12}FNO_2$+Na^+: 232.0744, Found: 232.0722. **IR** (neat, cm^{-1}): υ 3068, 2955, 1729, 1666, 1509, 1420, 1332, 893, 778.

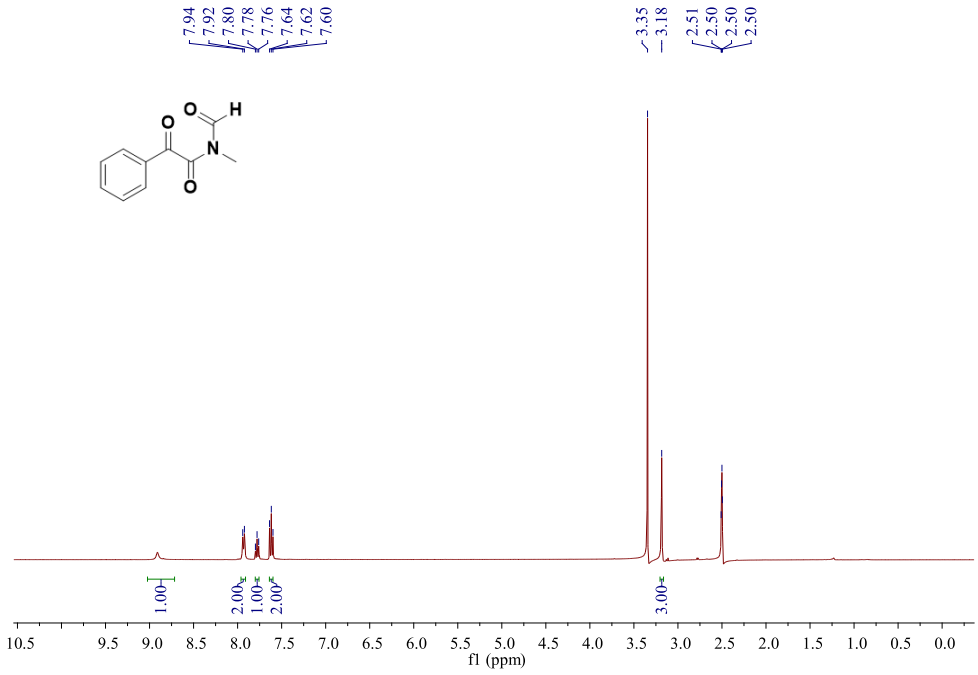


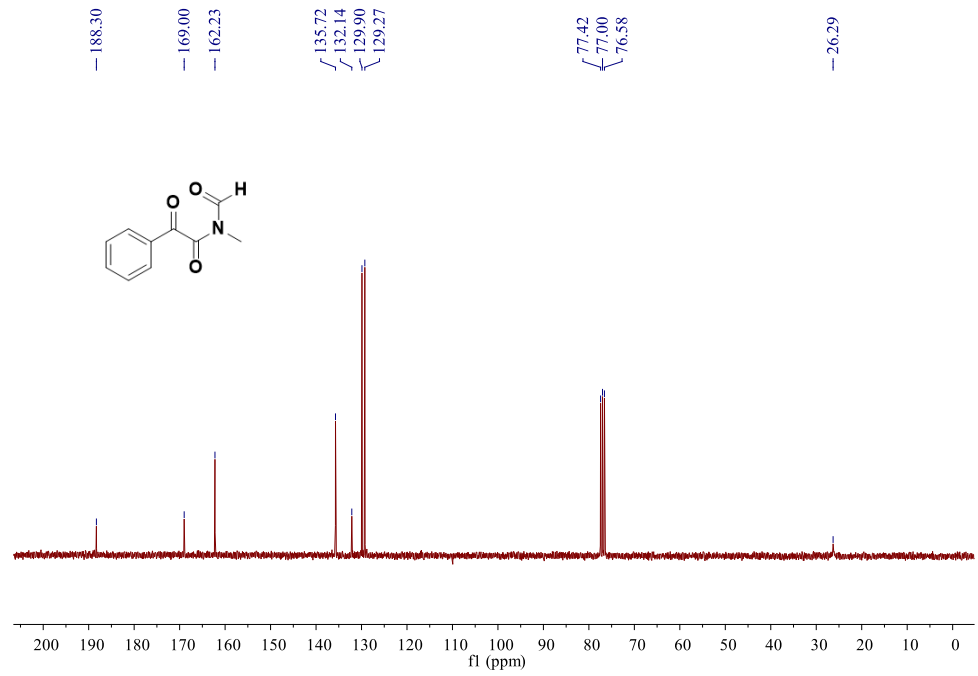

-113.80
F
O
O
N
H
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22(
f1 (ppm)

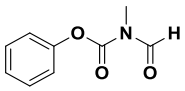


***N*-Formyl-*N*-Methyl-2-Oxo-2-Phenylacetamide (2af)**

petroleum ether / ethyl acetate = 5:1, yellow oil, 63% yield (24.1 mg). **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.91 (s, 1H), 7.93 (d, *J* = 7.6 Hz, 2H), 7.80 – 7.76 (m, 1H), 7.64 – 7.60 (m, 2H), 3.18 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 188.30, 169.00, 162.23, 135.72, 132.14, 129.90, 129.27, 26.29. **HRMS** (EI-TOF): Anal Calcd. For. $C_{10}H_9NO_3$-CO: 163.0633, Found: 163.0634. **IR** (neat, cm^{-1}): υ 3097, 2932, 1729, 1666, 1597, 1474, 1385, 723, 684.

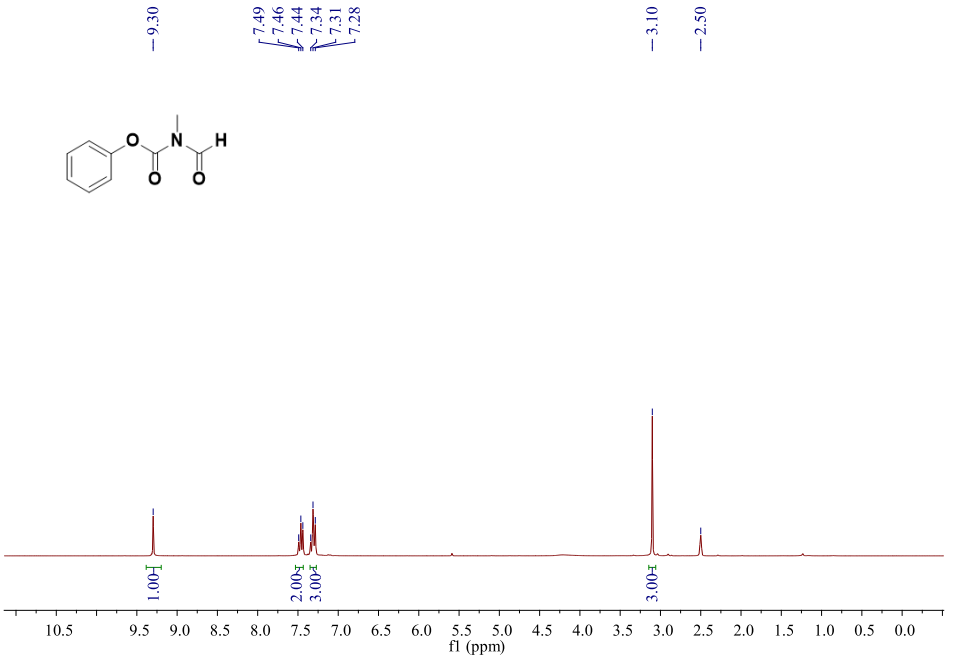


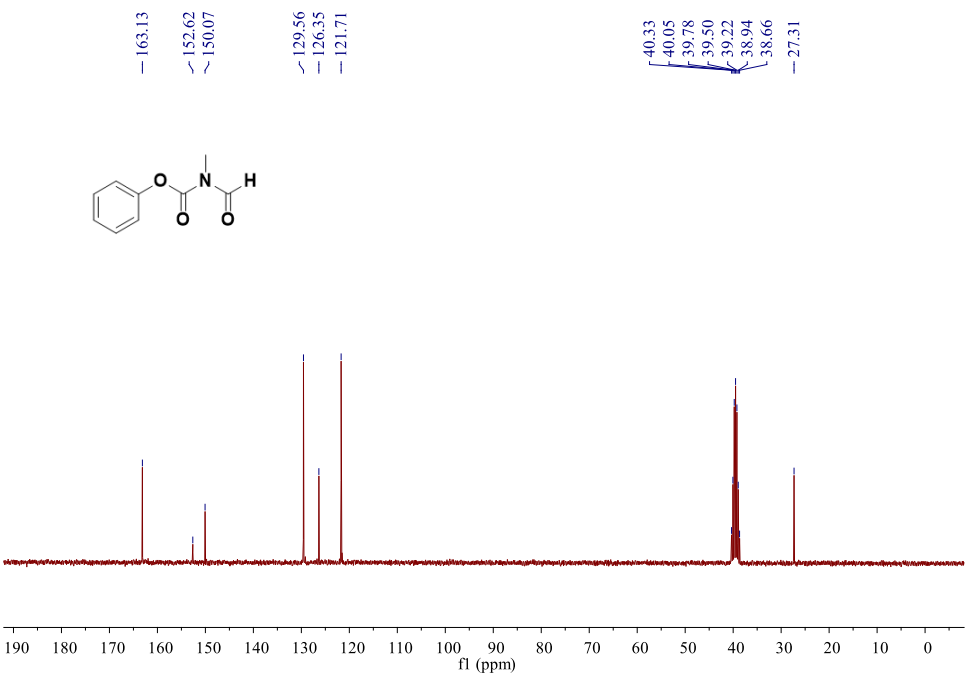


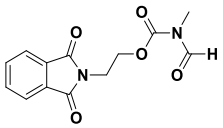


Phenyl Formyl(methyl)carbamate (2ag)

petroleum ether / ethyl acetate = 5:1, white solid, 48% yield (17.2 mg). mp: 38 – 40℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 9.30 (s, 1H), 7.49 – 7.44 (m, 2H), 7.34 – 7.28 (m, 3H), 3.10 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 163.13, 152.62, 150.07, 129.56, 126.35, 121.71, 27.31. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8FNO_3S+Na^+$: 202.0475, Found: 202.0473. **IR** (neat, cm^{-1}): υ 3396, 2953, 2826, 1759, 1685, 1590, 1491, 1291, 862, 736.

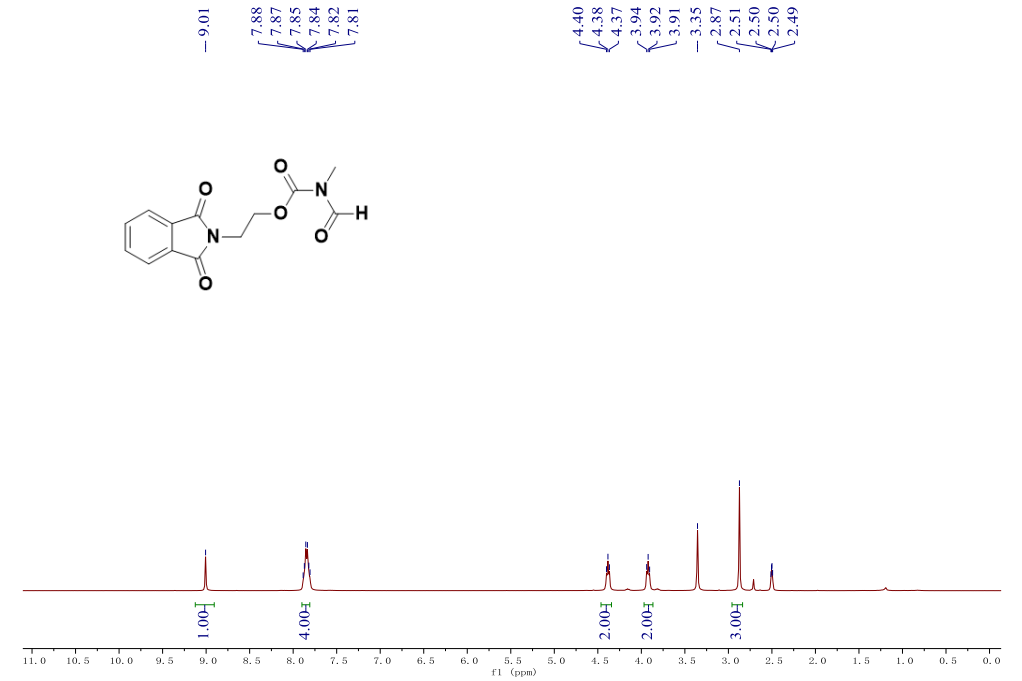


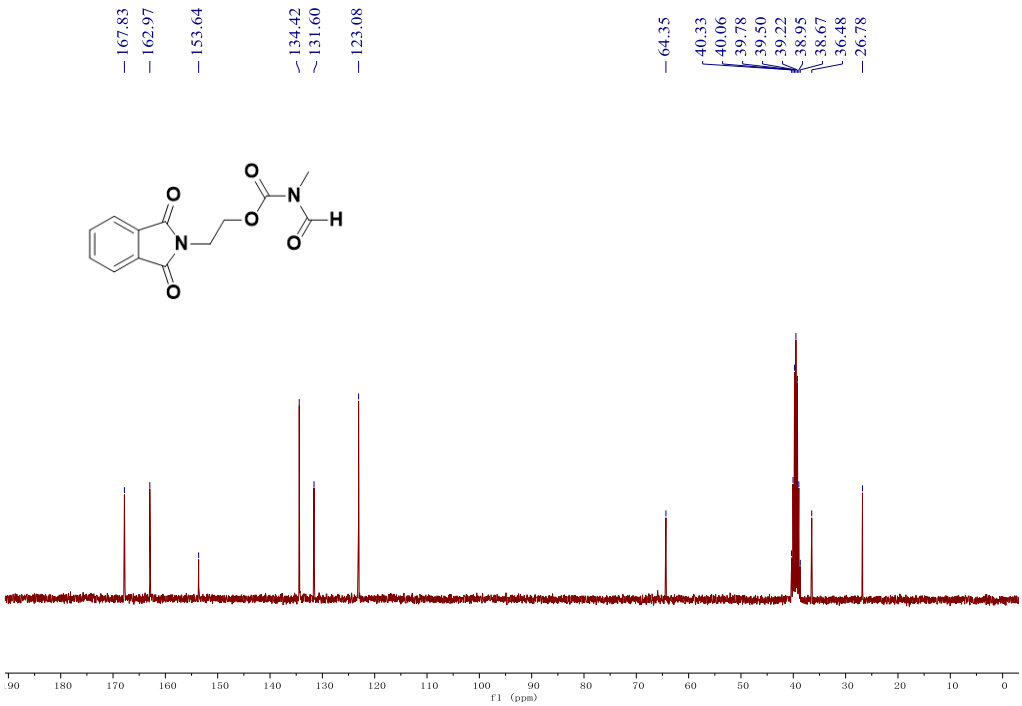


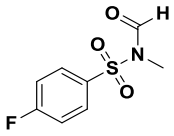


2-(1,3-dioxoisoindolin-2-yl)ethyl formyl(methyl)carbamate (2ah)

petroleum ether / ethyl acetate = 5:1, white solid, 64% yield (35.3 mg). mp: 116 – 118℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 9.01 (s, 1H), 7.88 – 7.81 (m, 4H), 4.38 (t, *J* = 5.1 Hz, 2H), 3.92 (t, *J* = 5.1 Hz, 2H), 2.87 (s, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 167.83, 162.97, 153.64, 134.42, 131.60, 123.08, 64.35, 36.48, 26.78. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{13}H_{12}N_2O_5$+Na^+: 299.0638, Found: 299.0647. **IR** (neat, cm^{-1}): υ 2979, 1771, 1650, 1497, 1392, 1262, 791.

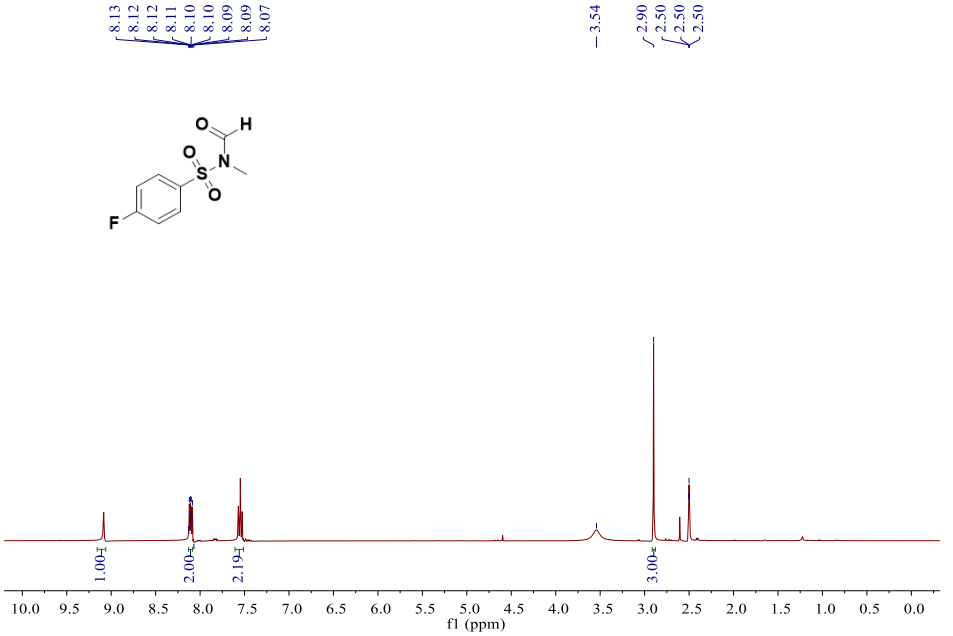


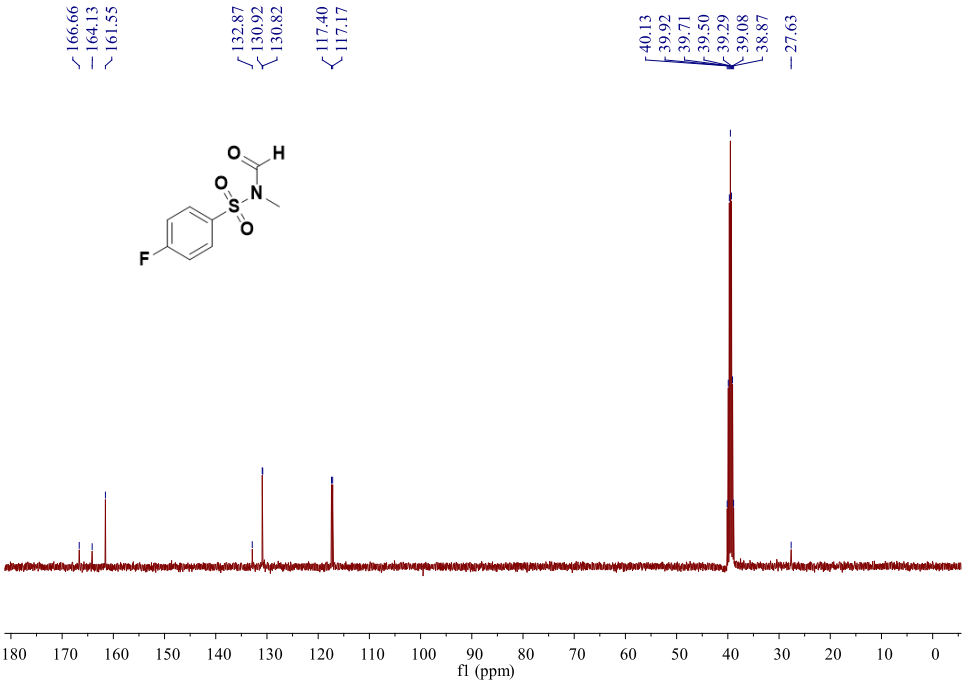


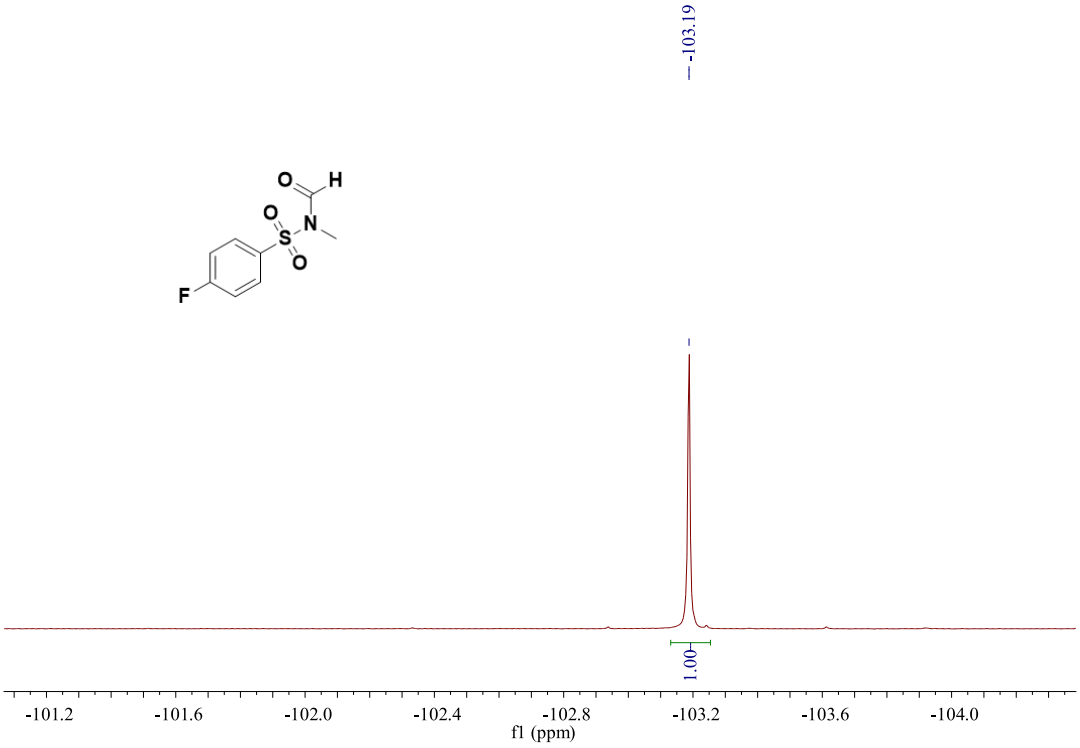


***N*-((4-fluorophenyl)sulfonyl)-*N*-Methylformamide (2ai)**

petroleum ether / ethyl acetate = 5:1, white solid, 74% yield (36.4 mg). mp: 88 – 90℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.07 (d, *J* = 12.2 Hz, 1H), 8.13 – 8.09 (m, 2H), 7.57 – 7.49 (m, 2H), 2.90 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.40 (d, *J* = 254.3 Hz), 161.55, 132.85 (d, *J* = 3.0 Hz), 130.87 (d, *J* = 10.1 Hz), 117.28 (d, *J* = 23.1 Hz), 27.63. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -103.19 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8FNO_3S+Na^+$: 240.0101, Found: 240.0101. **IR** (neat, cm^{-1}): υ 3103, 1687, 1588, 1493, 1351, 836, 779.



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O
H
O
N
S
O
F
1.00
-101.2
-101.6
-102.0
-102.4
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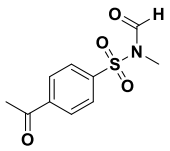
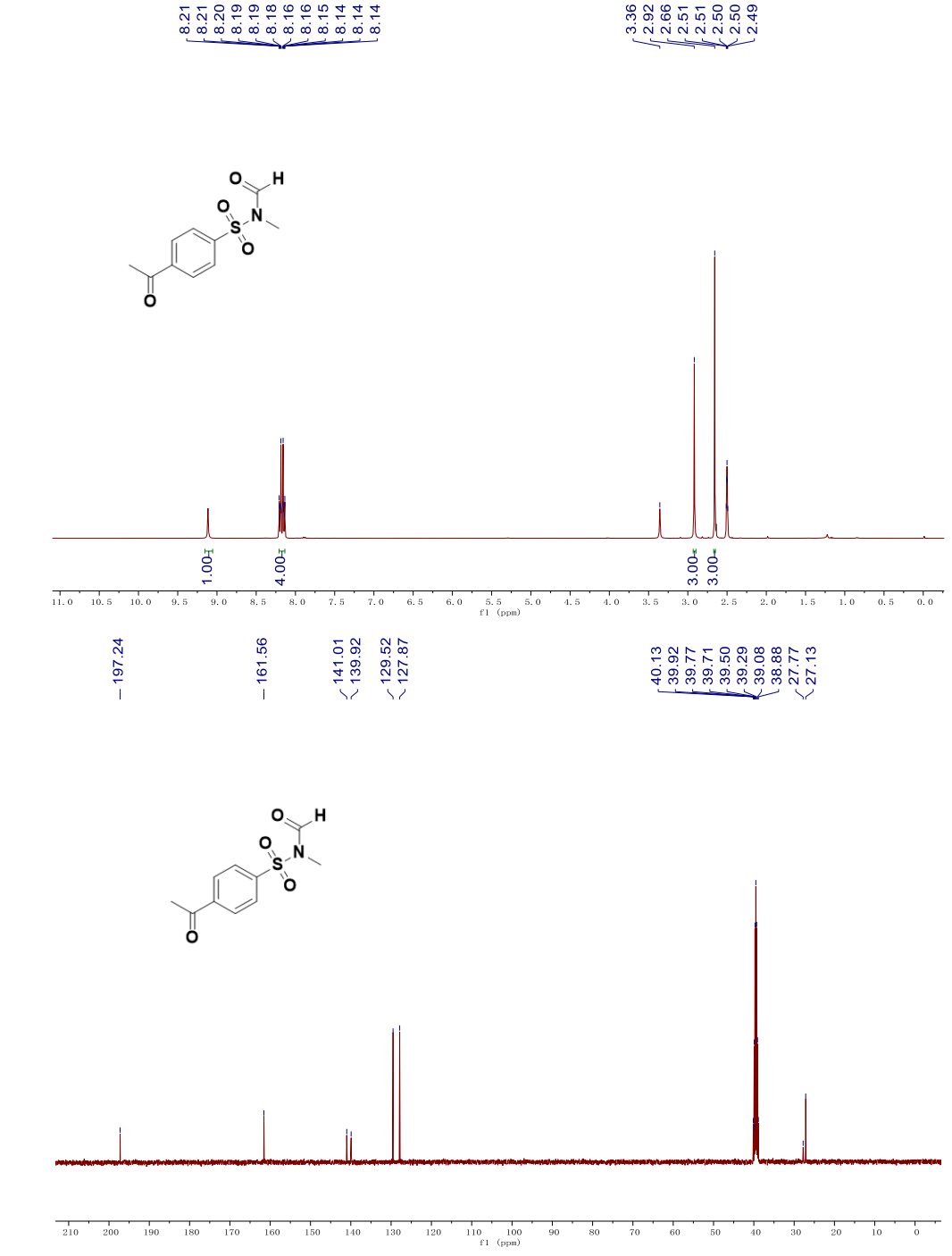
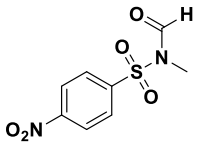
-103.2
-103.6
-104.0
f1 (ppm)



***N*-((4-Acetylphenyl)sulfonyl)-*N*-Methylformamide (2aj)**

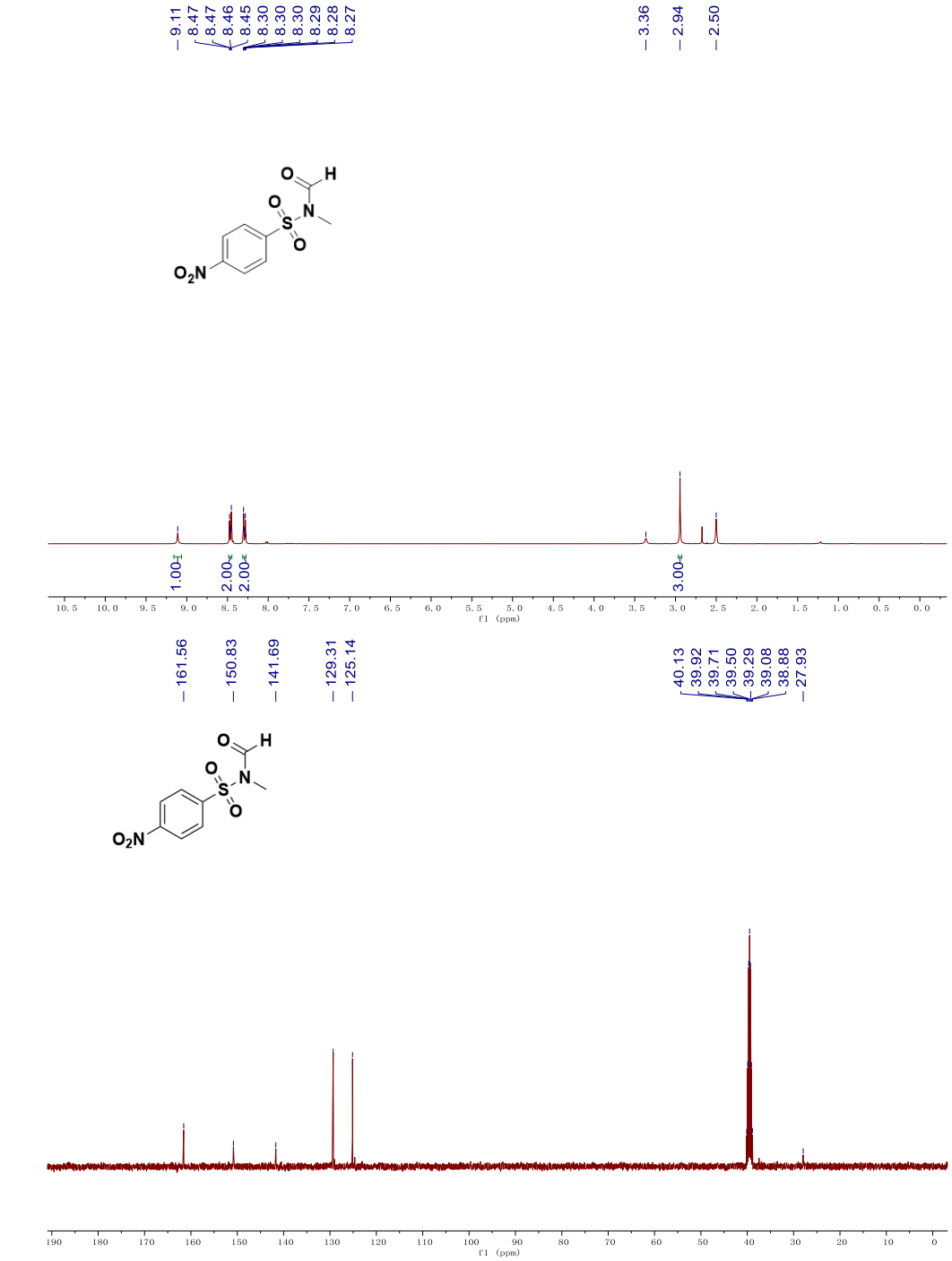
petroleum ether / ethyl acetate = 5:1, white solid, 36% yield (17.4 mg). mp: 90 – 92℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.11 (s, 1H), 8.21 – 8.14 (m, 4H), 2.92 (s, 3H), 2.66 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 197.24, 161.56, 141.01, 139.92, 129.52, 127.87, 27.77, 27.13. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8N_2O_5S+Na^+$: 264.0301, Found: 264.0292. **IR** (neat, cm^{-1}): υ 3098, 2852, 1771, 1694, 1596, 1417, 1362, 836, 771.

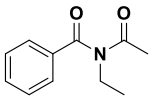




***N*-Methyl-*N*-((4-Nitrophenyl)sulfonyl)formamide (2ak)**

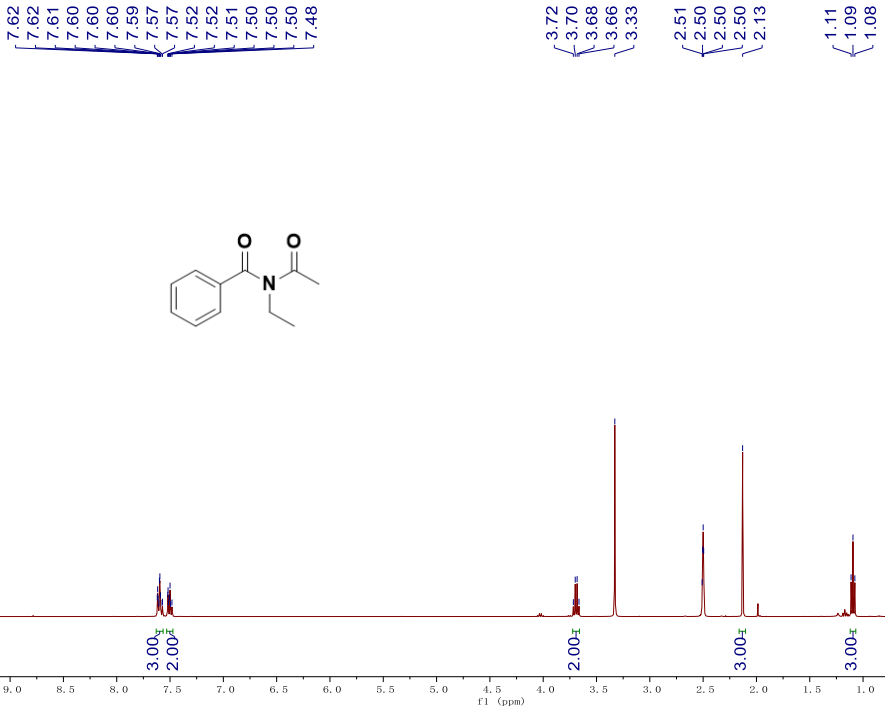
petroleum ether / ethyl acetate = 5:1, white solid, 50% yield (32.6 mg). mp: 100 – 102℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.11 (s, 1H), 8.47 – 8.45 (m, 2H), 8.30 – 8.27 (m, 2H), 2.94 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 161.56, 150.83, 141.69, 129.31, 125.14, 27.93. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8N_2O_5S+Na^+$: 267.0046, Found: 267.0045. **IR** (neat, cm^{-1}): υ 3105, 2959, 1774, 1606, 1530, 1458, 1349, 854, 755.

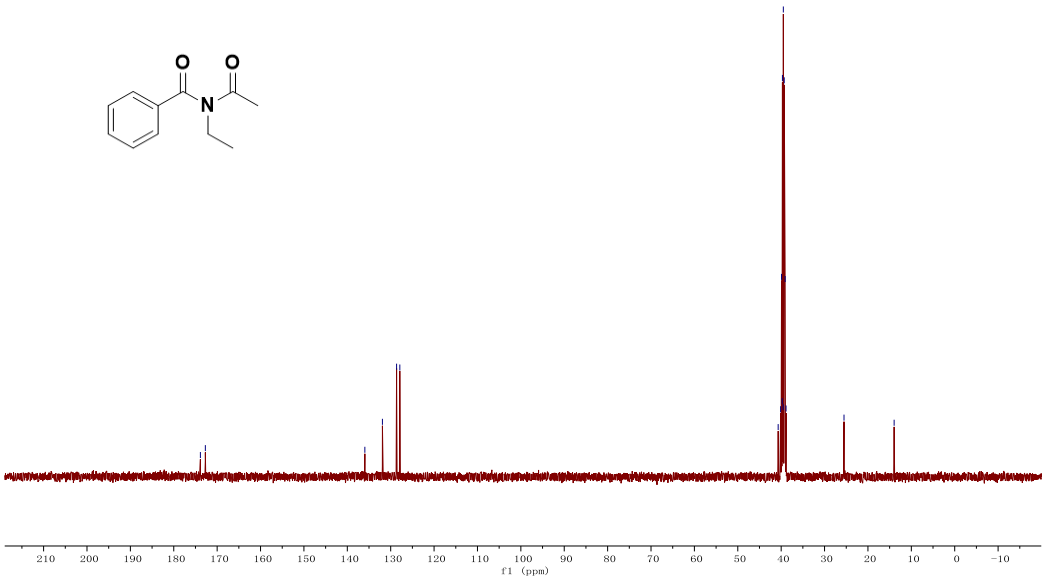


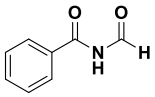


***N*-acetyl-*N*-ethylbenzamide (2al)**

petroleum ether / ethyl acetate = 5:1, yellow oil, 35% yield (13.5 mg). **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 7.62 – 7.57 (m, 3H), 7.52 – 7.48 (m, 2H), 3.69 (q, *J* = 7.0 Hz, 2H), 2.13 (s, 3H), 1.09 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 173.86, 172.72, 135.95, 131.91, 128.64, 127.90, 40.69, 25.52, 13.96. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{13}NO_2$+Na^+: 214.0838, Found: 214.0840. **IR** (neat, cm^{-1}): υ 3211, 2984, 1736, 1673, 1530, 1480, 1234, 847, 711.

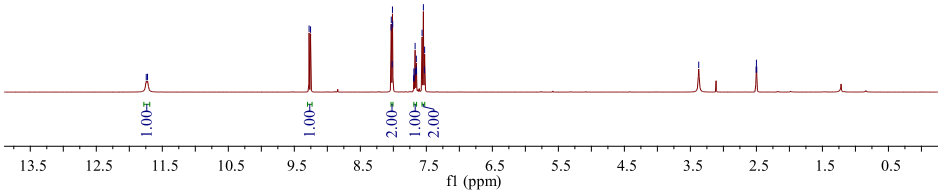


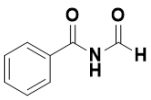


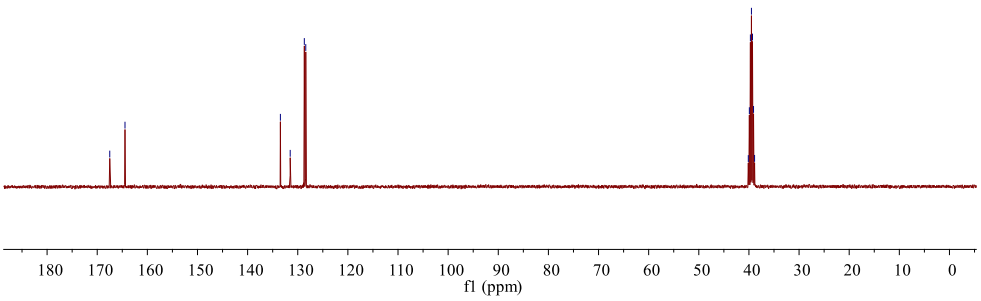


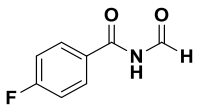
***N*-Formylbenzamide (2ba)**

petroleum ether / ethyl acetate = 5:1, white solid, 94% yield (28.0 mg). mp: 106 – 108℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.73 (d, *J* = 7.7 Hz, 1H), 9.27 (d, *J* = 8.9 Hz, 1H), 8.03 – 8.01 (m, 2H), 7.69 – 7.65 (m, 1H), 7.56 – 7.53 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 167.50, 164.46, 133.46, 131.50, 128.69, 128.40. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_7NO_2$+Na^+: 172.0369, Found: 172.0378. **IR** (neat, cm^{-1}): υ 3265, 2923, 1720, 1684, 1457, 1363, 885, 697.



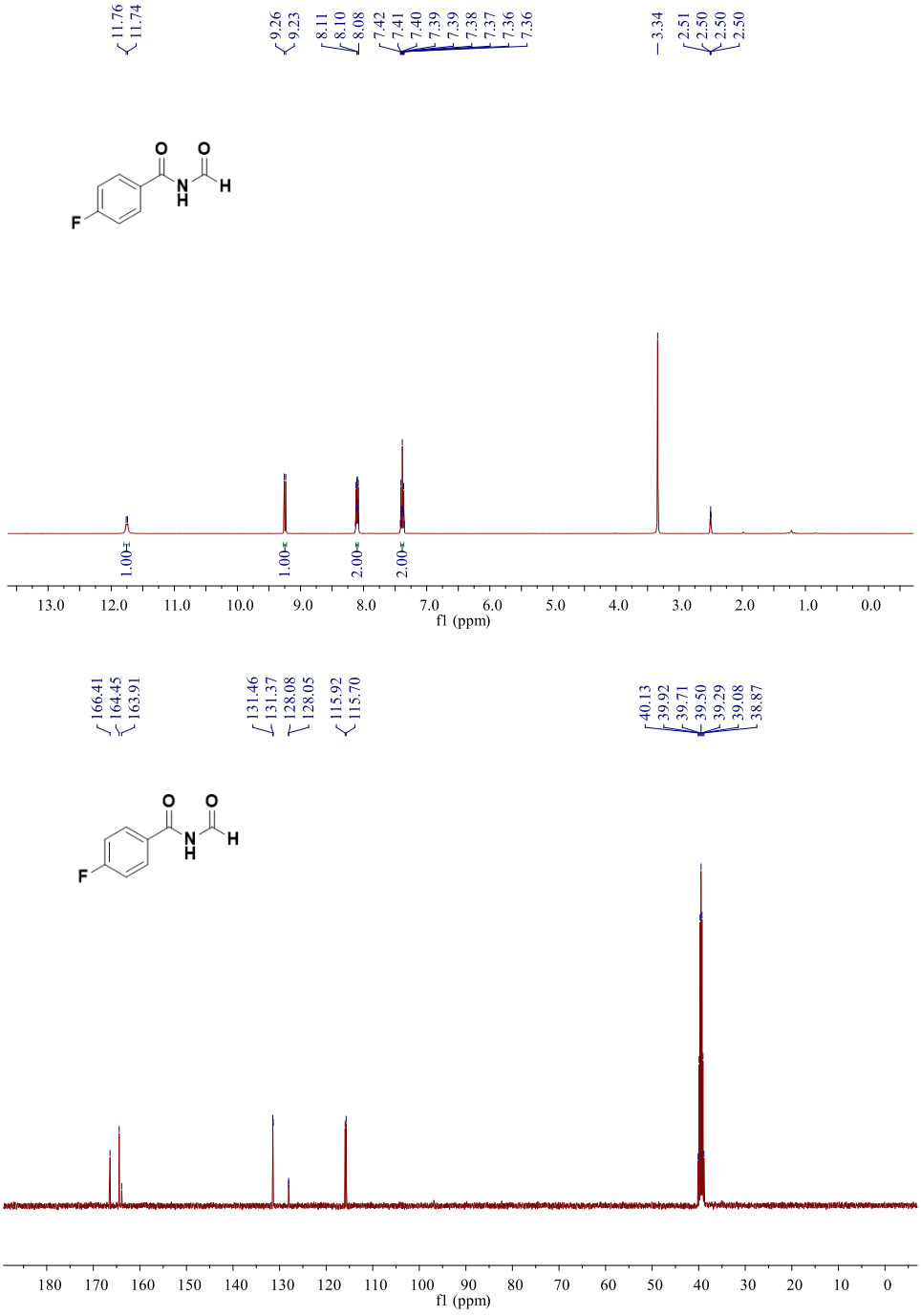


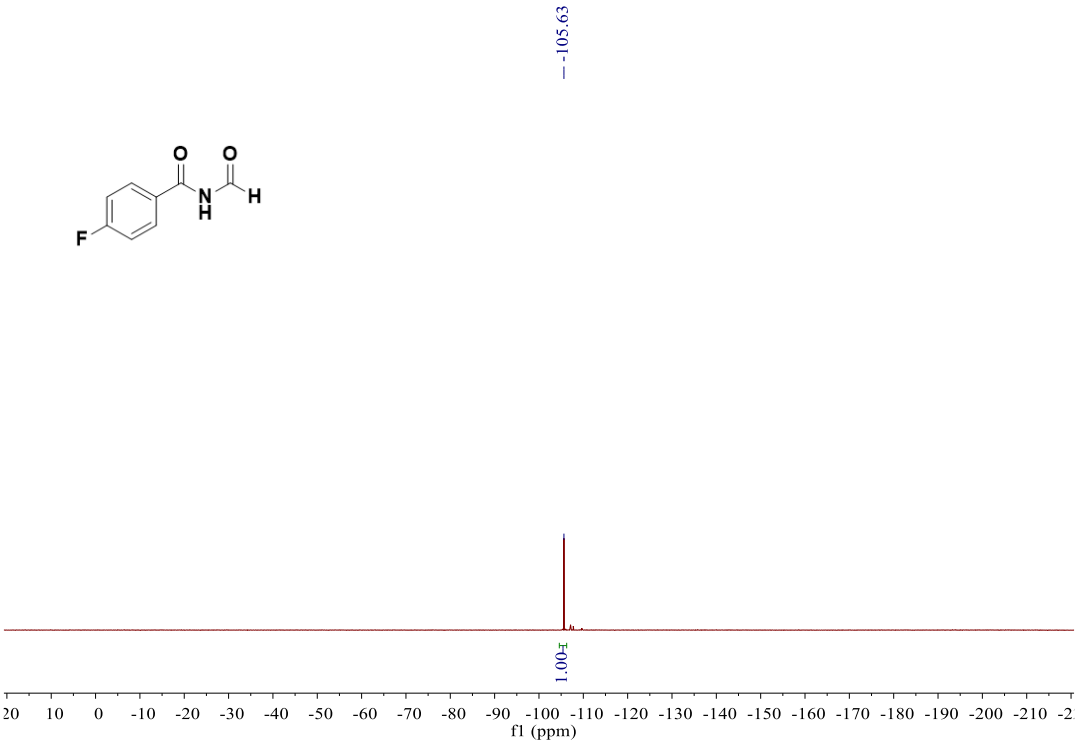




4-Fluoro-*N*-Formylbenzamide (2bb)

petroleum ether / ethyl acetate = 5:1, white solid, 78% yield (26.1 mg). mp: 161 – 163℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.75 (d, *J* = 8.2 Hz, 1H), 9.25 (d, *J* = 8.9 Hz, 1H), 8.13 – 8.08 (m, 2H), 7.42 – 7.36 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.41, 164.45, 163.91, 131.41 (d, *J* = 9.6 Hz), 128.07 (d, *J* = 2.8 Hz), 115.81 (d, *J* = 22.1 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -105.63 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8FNO_2$+Na^+: 190.0275, Found: 190.0274. **IR** (neat, cm^{-1}): υ 3336, 3078, 2947, 1637, 1547, 1409, 1328, 892, 791.

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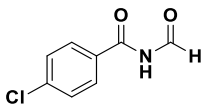
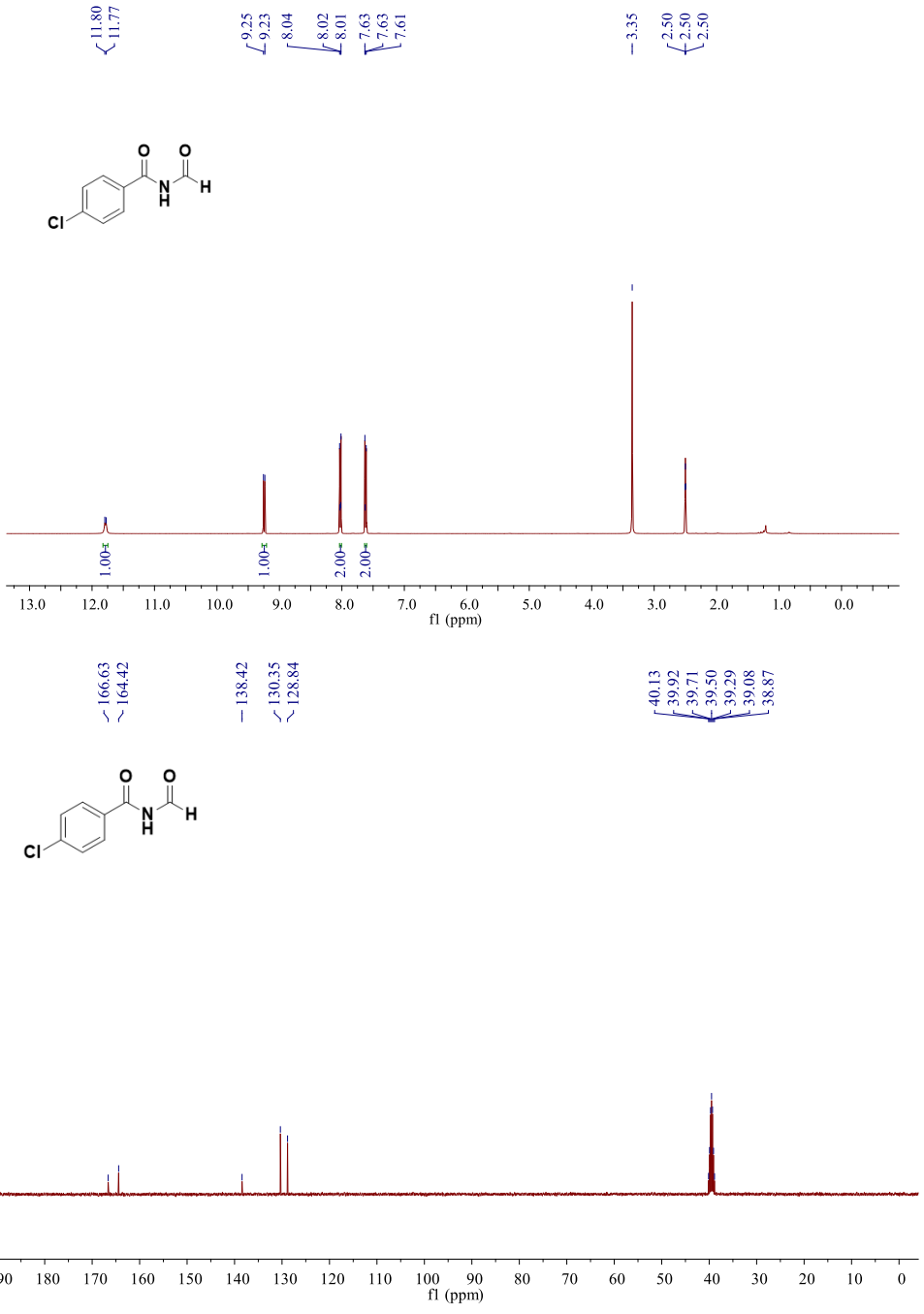
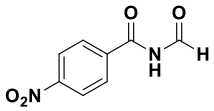
O
O
N
H
H
F
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2
f1 (ppm)



4-Chloro-*N*-Formylbenzamide (2bc)

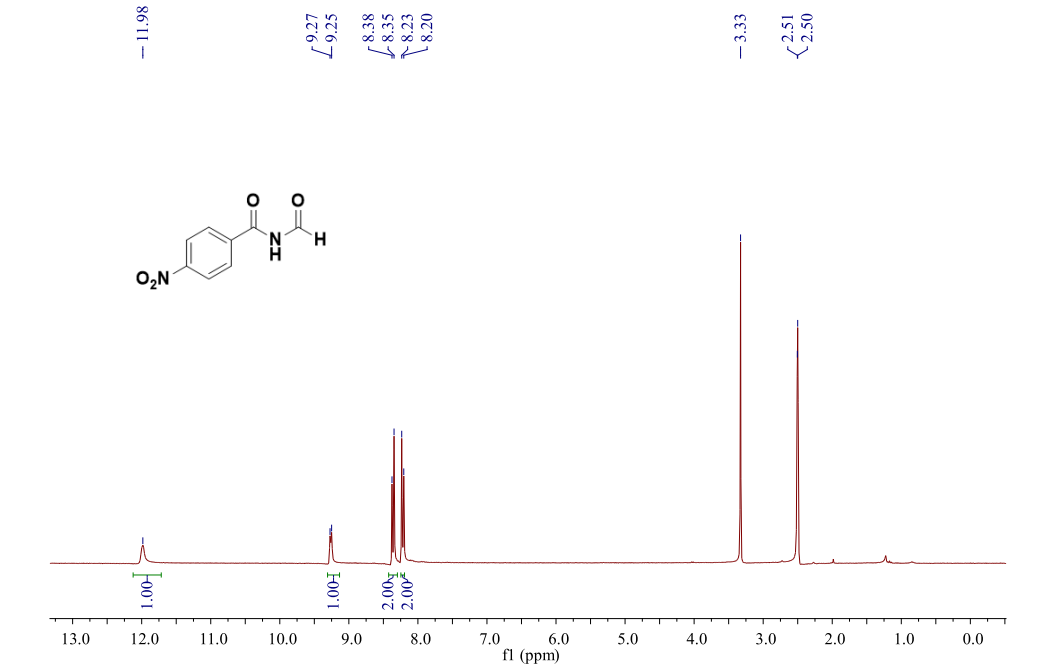
petroleum ether / ethyl acetate = 5:1, white solid, 51% yield (18.7 mg). mp: 183 – 185℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.78 (d, *J* = 8.8 Hz, 1H), 9.24 (d, *J* = 8.8 Hz, 1H), 8.04 – 8.01 (m, 2H), 7.63 – 7.61 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.63, 164.42, 138.42, 130.35, 128.84, 128.84. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6{}^{35}ClNO_2$+Na^+: 205.9979, Found: 206.0012. Anal Calcd. For. $C_8H_6{}^{37}ClNO_2$+Na^+: 207.9950, Found: 207.9954. **IR** (neat, cm^{-1}): υ 3331, 3076, 2938, 1634, 1551, 1454, 1343, 839, 750.

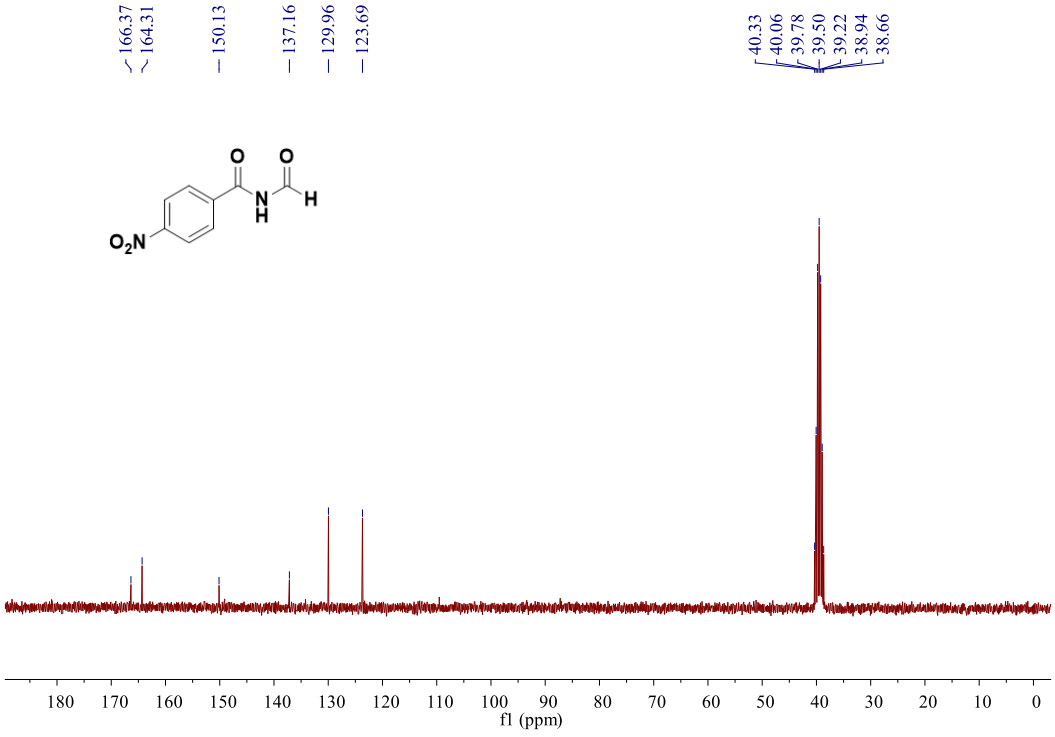


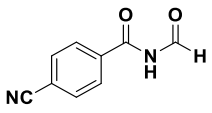


***N*-Formyl-4-Nitrobenzamide (2bd)**

petroleum ether / ethyl acetate = 4:1, white solid, 50% yield (19.4 mg). mp: 206 – 208℃.**^{1}H NMR** (300 MHz, DMSO-*d6*) δ 11.98 (s, 1H), 9.26 (d, *J* = 6.9 Hz, 1H), 8.36 (d, *J* = 8.9 Hz, 2H), 8.22 (d, *J* = 8.9 Hz, 2H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 166.37, 164.31, 150.13, 137.16, 129.96, 123.69. **HRMS** (EI-TOF): Anal Calcd. For. $C_8H_6N_2O_4$: 194.0328, Found: 194.0325. **IR** (neat, cm^{-1}): υ 3328, 3051, 2950, 1643, 1549, 1490, 1348, 824, 770.

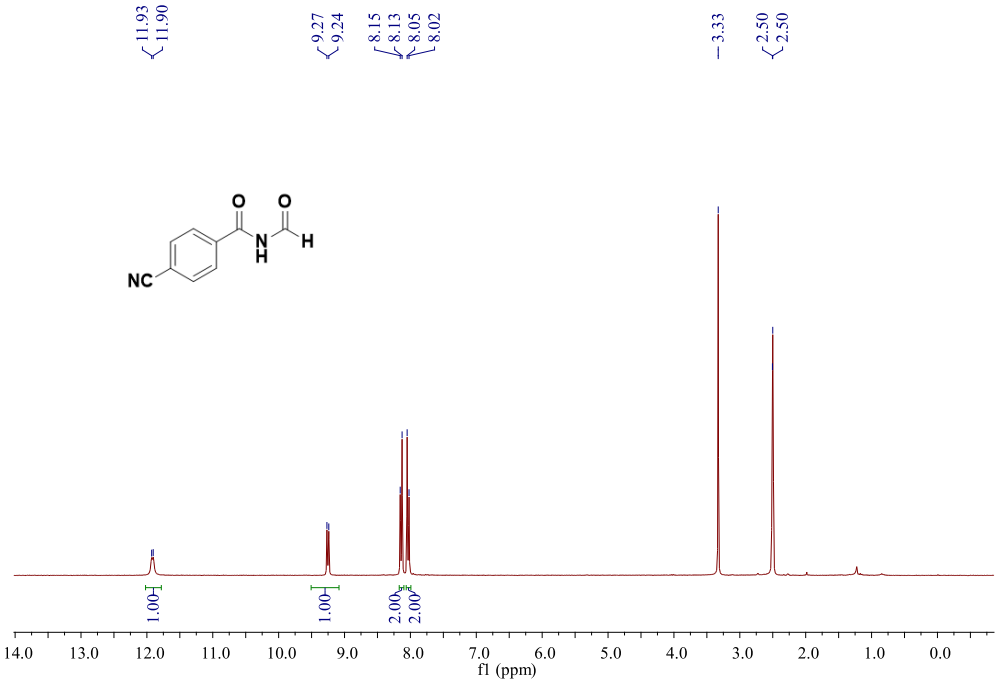


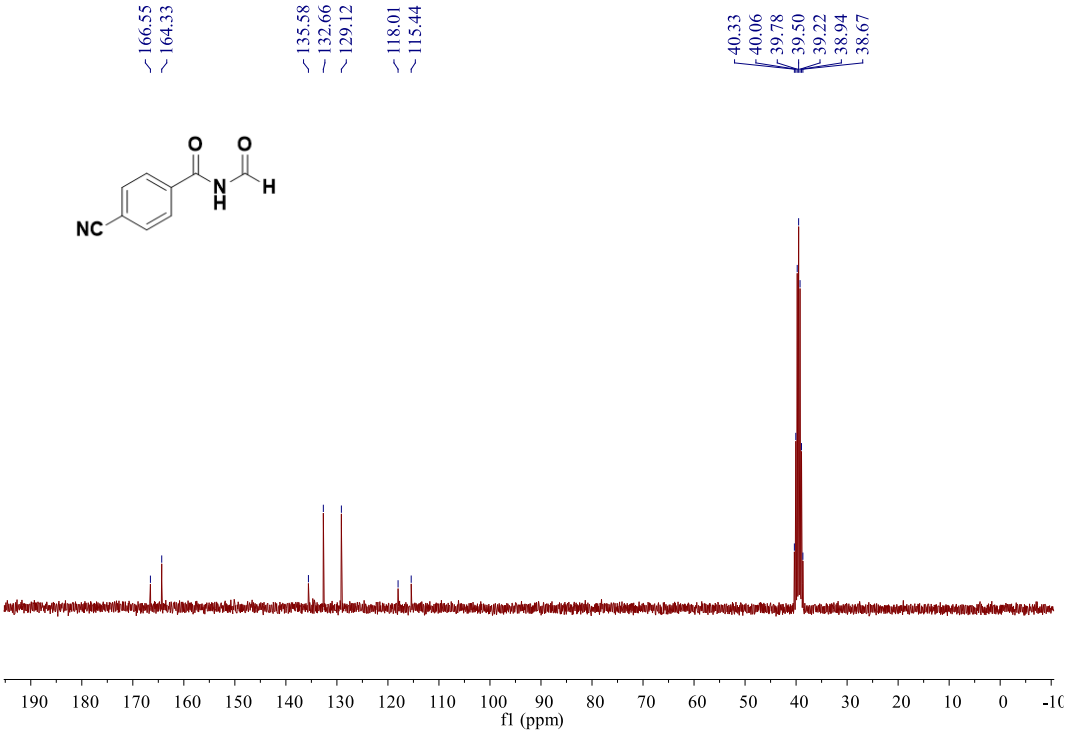


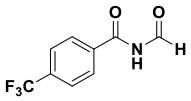


4-Cyano-*N*-Formylbenzamide (2be)

petroleum ether / ethyl acetate = 4:1, white solid, 82% yield (28.5 mg). mp: 78 –80°C. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 11.92 (d, *J* = 7.1 Hz, 1H), 9.25 (d, *J* = 8.6 Hz, 1H), 8.14 (d, *J* = 8.6 Hz, 2H), 8.04 (d, *J* = 8.6 Hz, 2H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 166.55, 164.33, 135.58, 132.66, 129.12, 118.01, 115.44. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6N_2O_2$ +Na^+: 197.0321, Found: 197.0318. **IR** (neat, cm^{-1}): υ 3309, 3054, 2960, 2280, 1724, 1631, 1550, 1447, 1361, 803, 770.

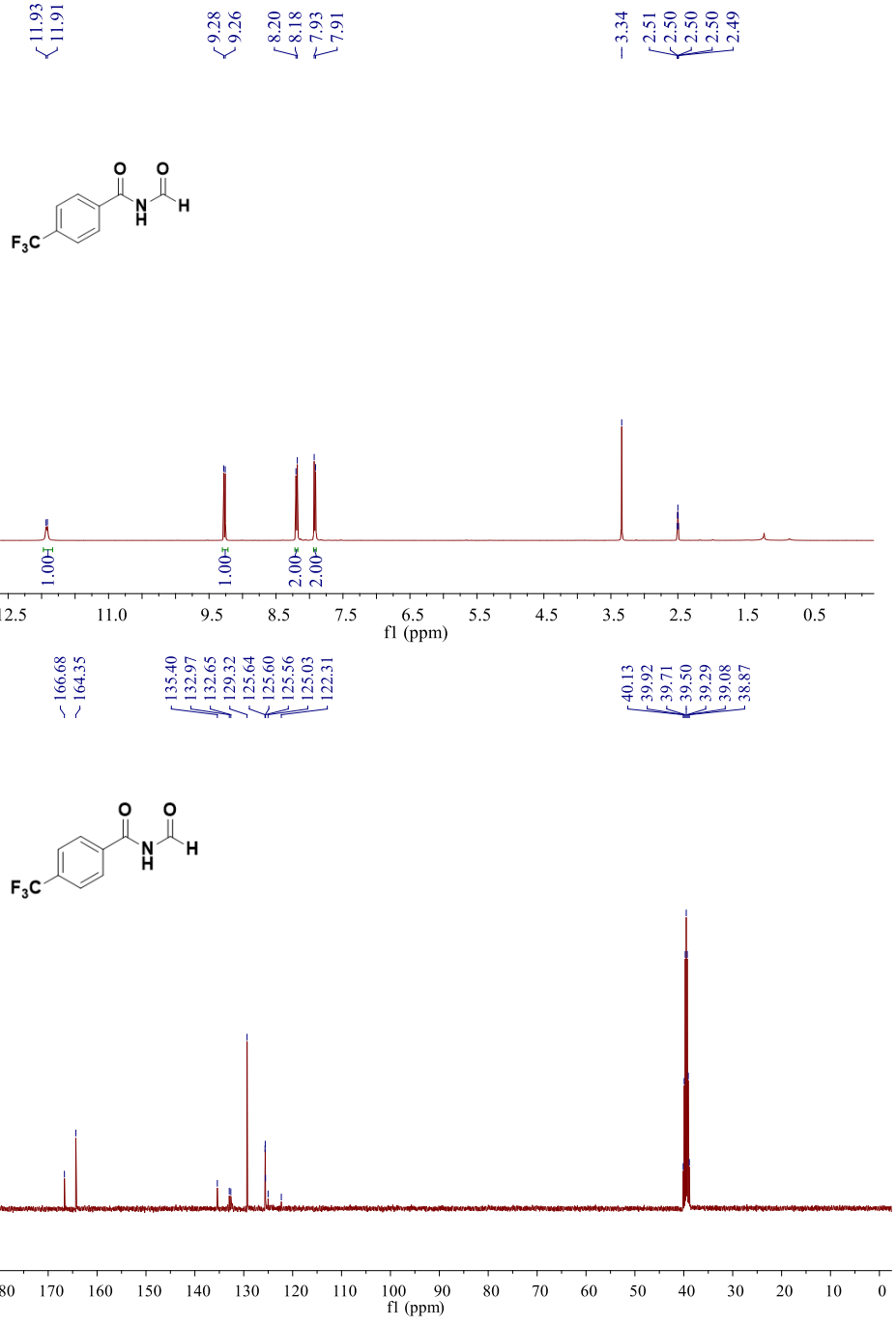


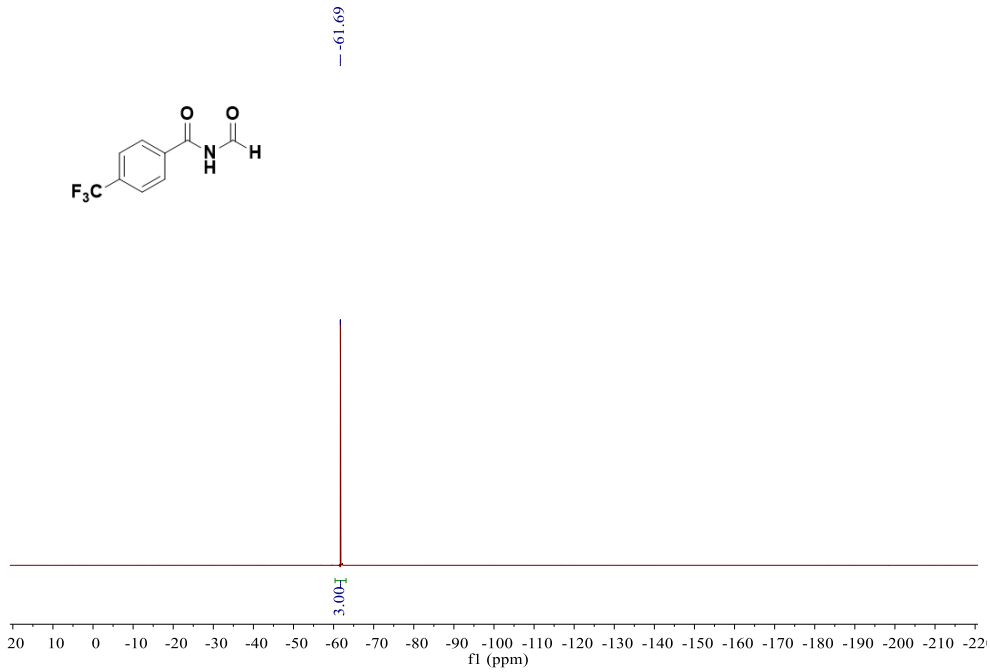




***N*-Formyl-4-(trifluoromethyl)Benzamide (2bf)**

petroleum ether / ethyl acetate = 5:1, white solid, 52% yield (22.6mg). mp: 125 – 127℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.92 (d, *J* = 8.3 Hz, 1H), 9.27 (d, *J* = 8.8 Hz, 1H), 8.19 (d, *J* = 8.3 Hz, 2H), 7.92 (d, *J* = 8.3 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.68, 164.35, 135.40, 132.81 (q, *J* = 32.0 Hz), 129.32, 125.62 (q, *J* = 40.0 Hz), 123.67 (d, *J* = 272.8 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.69 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_2$+Na^+: 240.0243, Found: 240.0248. **IR** (neat, cm^{-1}): υ 3271, 2963, 1731, 1681, 1503, 815, 797.

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H
H
F3C
3.00
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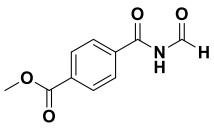
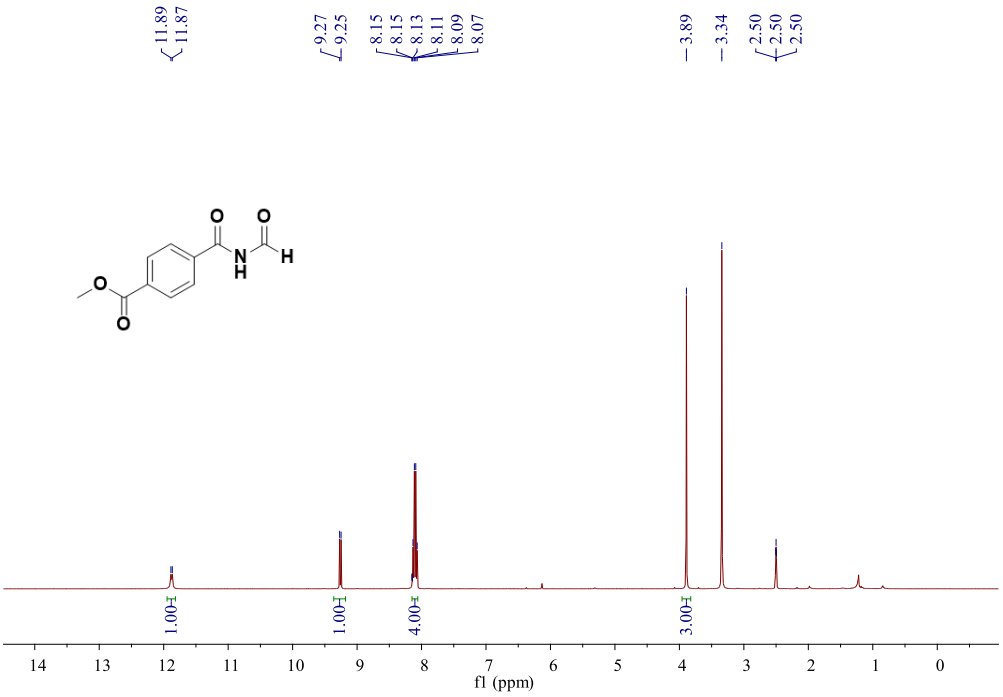
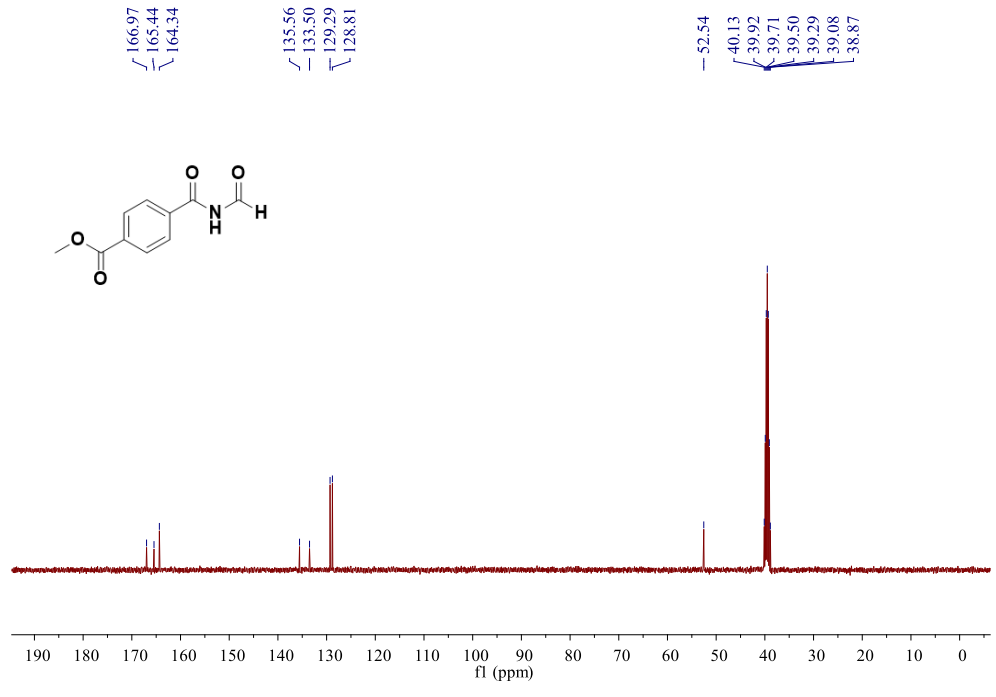
f1 (ppm)

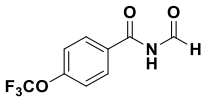


Methyl 4-(formylcarbamoyl)Benzoate (2bg)

petroleum ether / ethyl acetate = 5:1, yellow solid, 49% yield (20.3 mg). mp: 185 – 186℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.88 (d, *J* = 8.8 Hz, 1H), 9.26 (d, *J* = 8.8 Hz, 1H), 8.10 (q, *J* = 8.6 Hz, 4H), 3.89 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.97, 165.44, 164.34, 135.56, 133.50, 129.29, 128.81, 52.54. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_9NO_4$ +Na^+: 230.0424, Found: 230.0414. **IR** (neat, cm^{-1}): υ 3349, 2959, 2851, 1714, 1675, 1515, 1435, 1377, 1277, 826, 795.

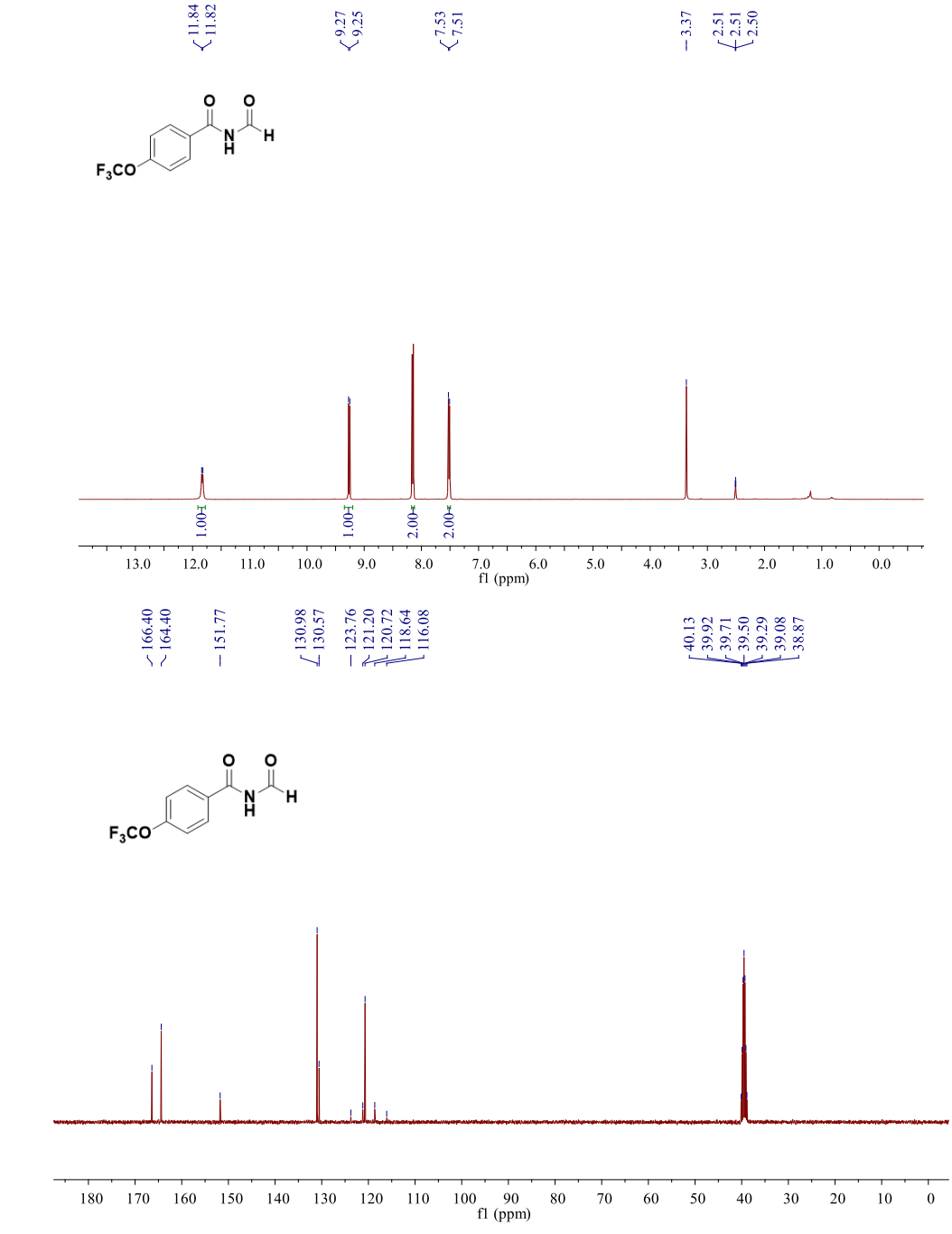


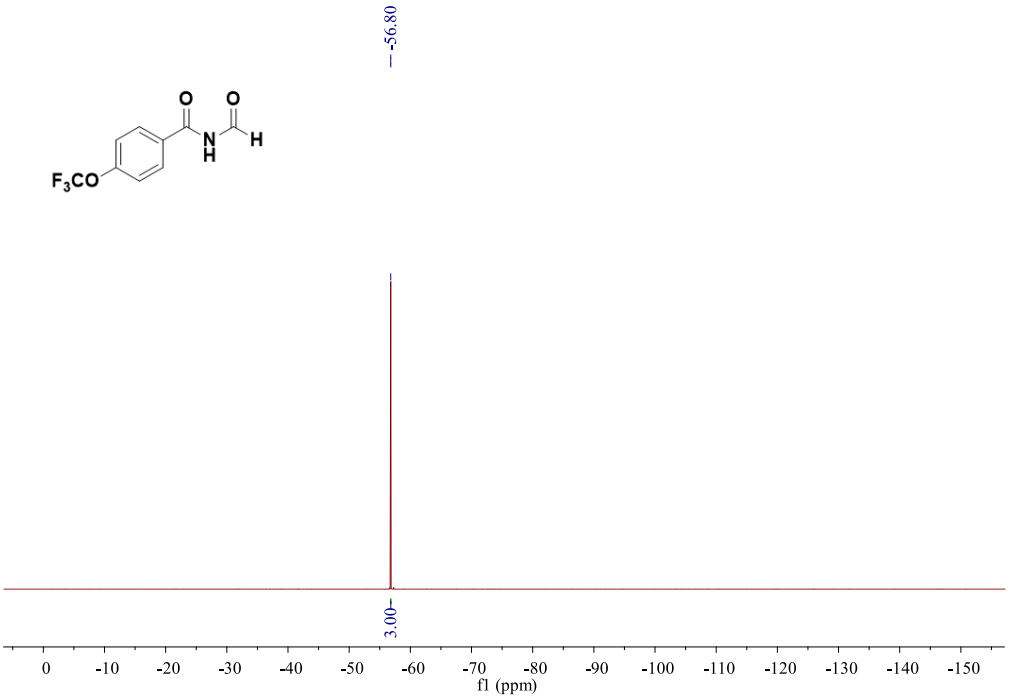




***N*-Formyl-4-(trifluoromethoxy)Benzamide (2bh)**

petroleum ether / ethyl acetate = 5:1, white solid, 81% yield (37.8 mg). mp: 101 – 103℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.83 (d, *J* = 8.8 Hz, 1H), 9.26 (d, *J* = 8.8 Hz, 1H), 8.16 – 8.13 (m, 2H), 7.53 – 7.51 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.40, 164.40, 151.77, 130.98, 130.57, 120.72, 119.92 (q, *J* = 256.0 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.80 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_3$ +Na^+: 256.0192, Found: 256.0188. **IR** (neat, cm^{-1}): υ 3259, 2853, 1722, 1681, 1518, 1456, 1219, 809.

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O
O
N
H
H
F3CO
3.00
0
-10
-20
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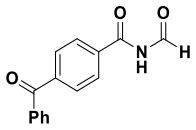
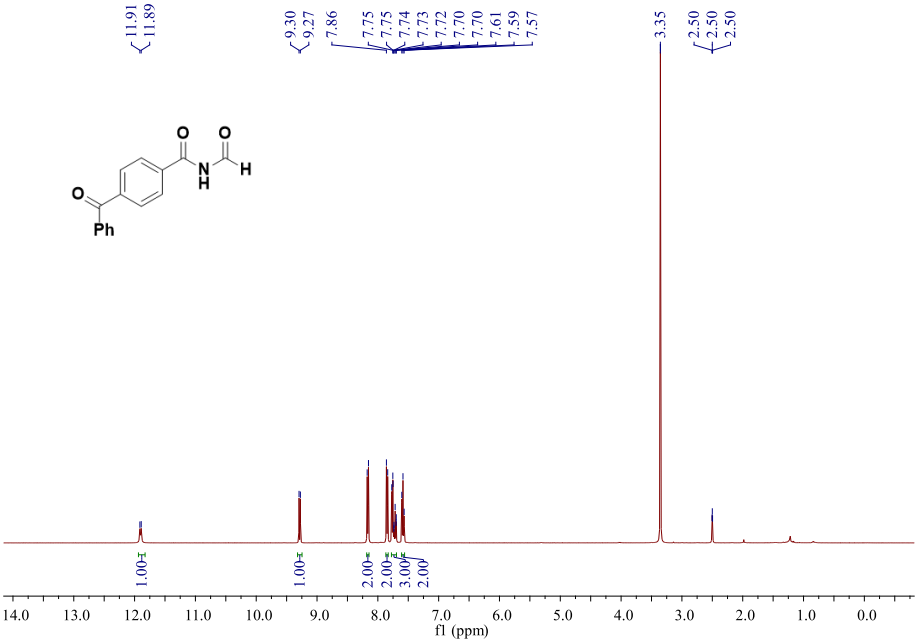
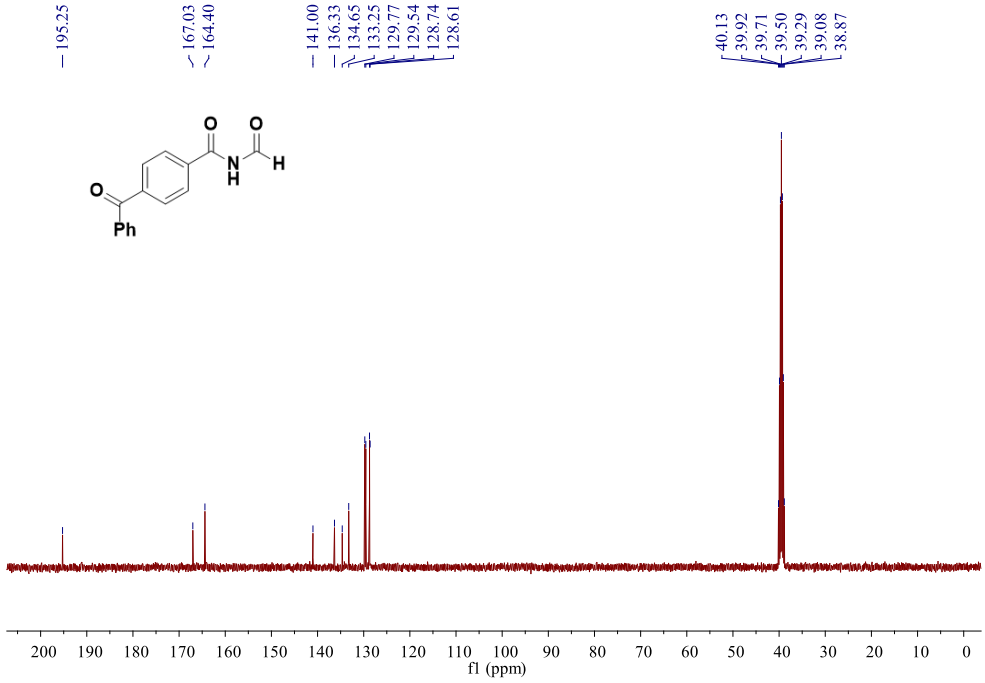
-140
-150
f1 (ppm)

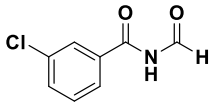


4-Benzoyl-*N*-FormylBenzamide (2bi)

petroleum ether / ethyl acetate = 5:1, white solid, 40% yield (20.2 mg). mp: 96 – 98℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.90 (d, *J* = 8.8 Hz, 1H), 9.28 (d, *J* = 8.8 Hz, 1H), 8.16 (d, *J* = 8.5 Hz, 2H), 7.85 (d, *J* = 8.5 Hz, 2H), 7.77 – 7.70 (m, 3H), 7.59 (t, *J* = 7.6 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 195.25, 167.03, 164.40, 141.00, 136.33, 134.65, 133.25, 129.77, 129.54, 128.74, 128.61. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{15}H_{11}NO_3$ +Na^+: 276.0631, Found: 276.0615. **IR** (neat, cm^{-1}): υ 3332, 0934, 1660, 1553, 1447, 1315, 839, 766.

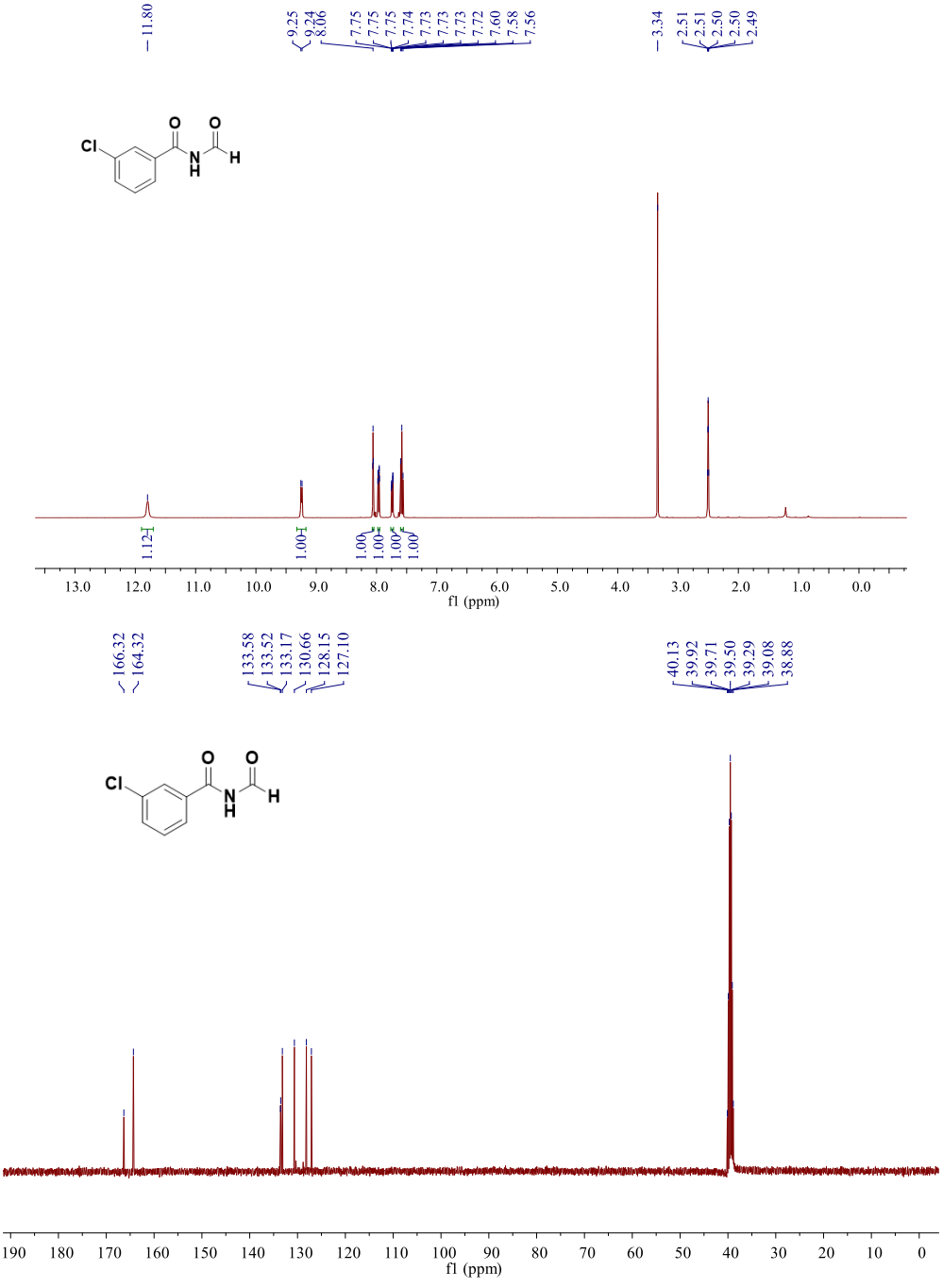


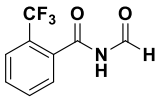




3-Chloro-*N*-Formylbenzamide (2bj)

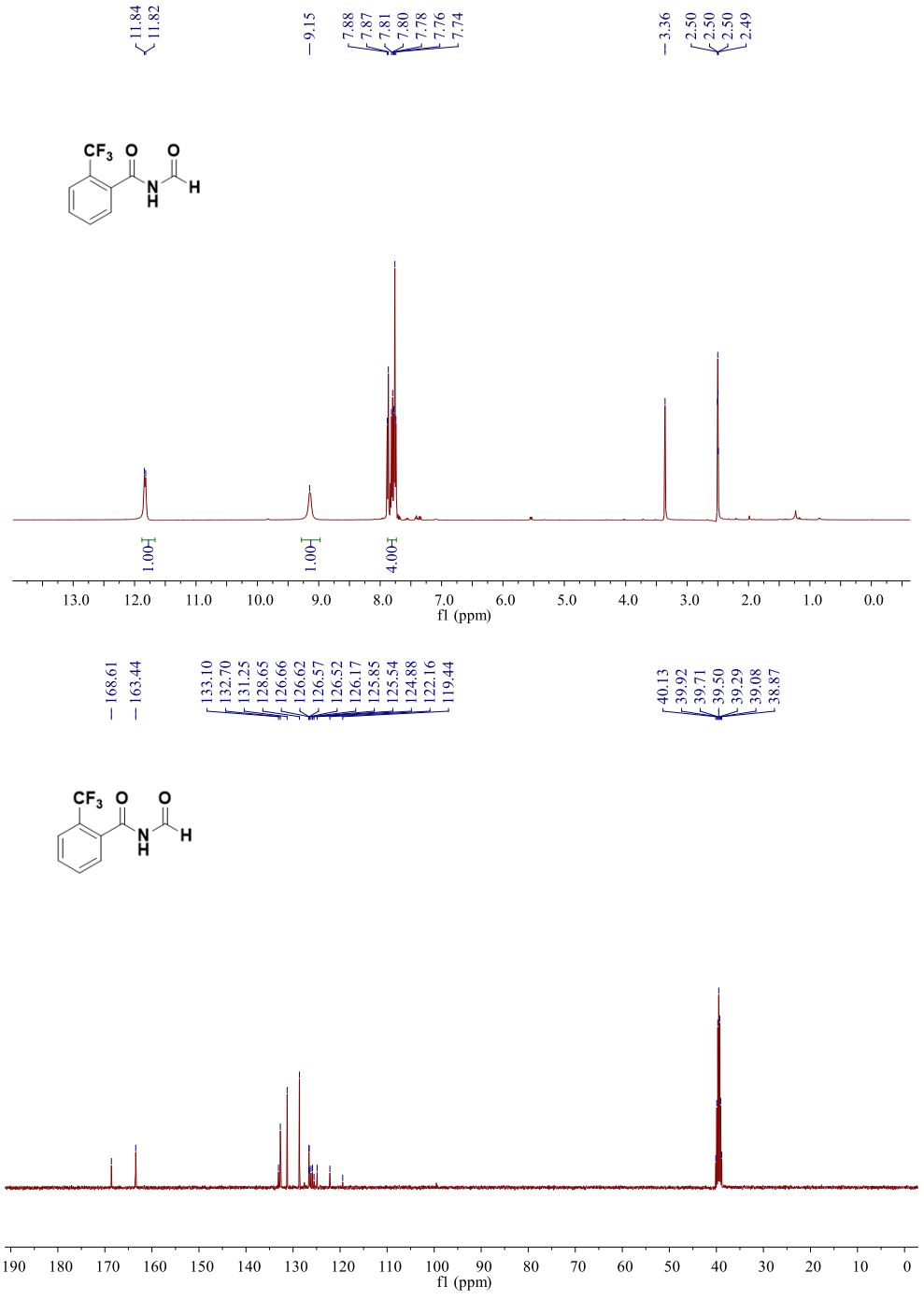
petroleum ether / ethyl acetate = 5:1, white solid, 78% yield (36.6 mg). mp: 135 – 137℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.80 (s, 1H), 9.24 (d, *J* = 7.8 Hz, 1H), 8.06 (t, *J* = 1.9 Hz, 1H), 7.98 – 7.95 (m, 1H), 7.75 – 7.72 (m, 1H), 7.58 (t, *J* = 7.8 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.32, 164.32, 133.58, 133.52, 133.17, 130.66, 128.15, 127.10. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6{}^{35}ClNO_2$+Na^+: 205.9979, Found: 205.9985. Anal Calcd. For. $C_8H_6{}^{35}ClNO_2$+Na^+: 205.9950, Found: 205.9932. **IR** (neat, cm^{-1}): υ 3077, 2921, 1726, 1674, 1588, 1459, 1364, 887, 762.

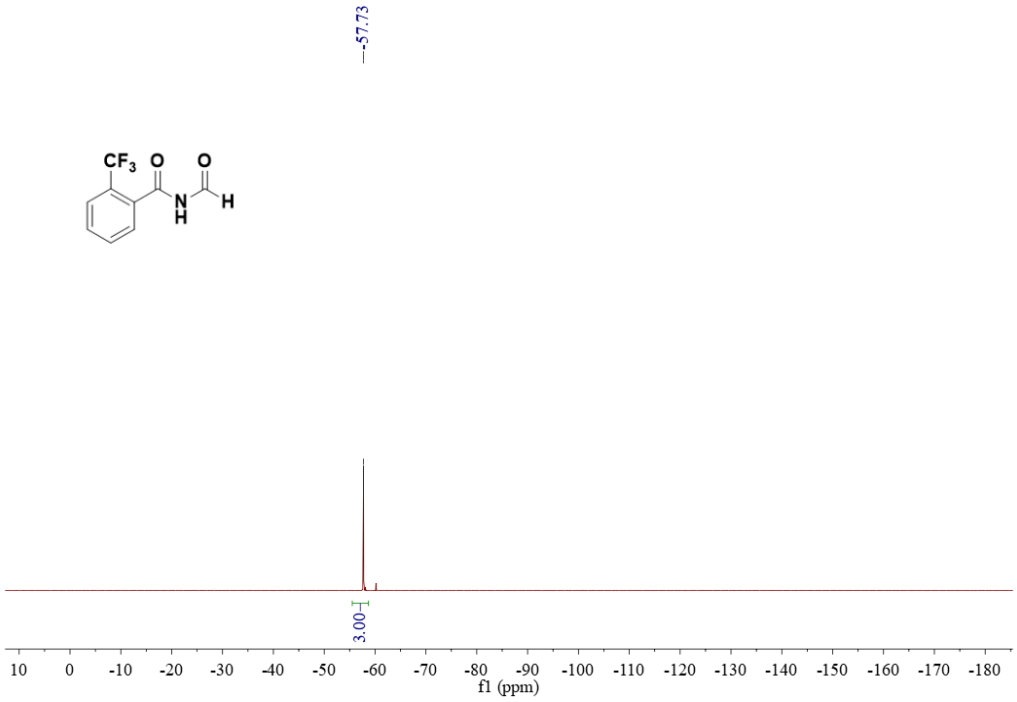




***N*-Formyl-2-(trifluoromethyl)Benzamide (2bk)**

petroleum ether / ethyl acetate = 5:1, white solid, 62% yield (26.9 mg). mp: 107 – 109℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.83 (d, *J* = 8.5 Hz, 1H), 9.15 (s, 1H), 7.88 – 7.74 (m, 4H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 168.61, 163.44, 133.10, 132.70, 131.25, 128.65, 126.59 (q, *J* = 4.8 Hz), 126.01 (q, *J* = 31.7 Hz, 1H), 123.52 (q, *J* = 273.6 Hz, 1H). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -57.73 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_2$+Na^+: 240.0423, Found: 240.0234. **IR** (neat, cm^{-1}): υ 3269, 2925, 1729, 1682, 1587, 1465, 1363, 757.

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O
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H
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0
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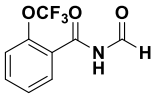
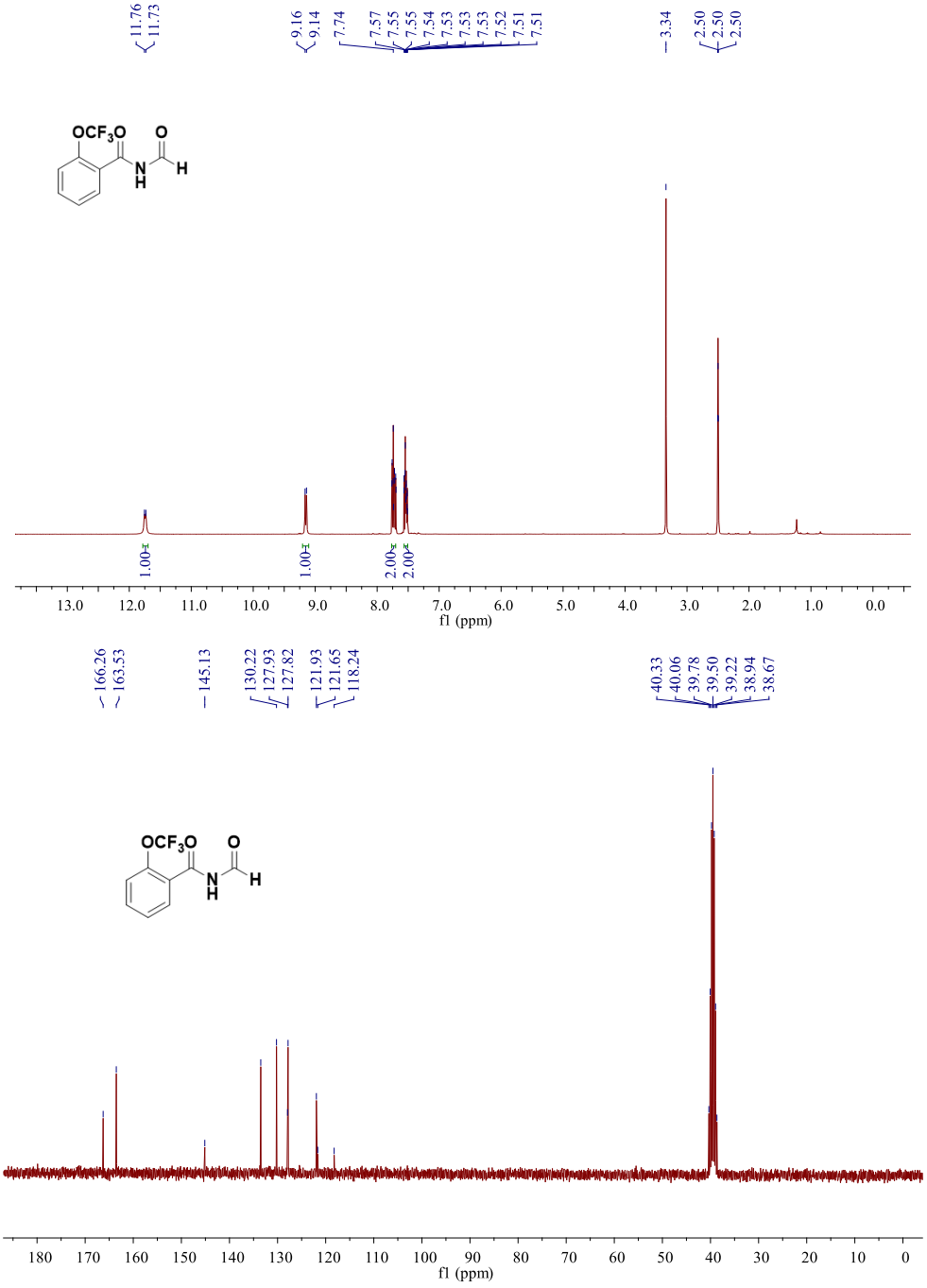
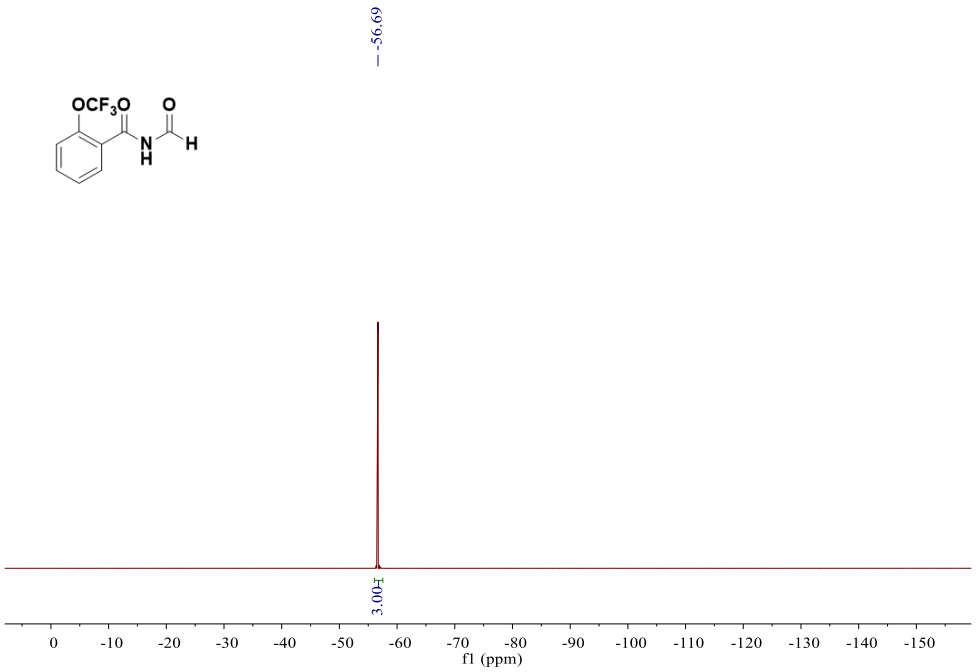
f1 (ppm)



***N*-Formyl-2-(trifluoromethoxy)Benzamide (2bl)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 65% yield (30.3 mg). mp: 95 – 97℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.74 (d, *J* = 9.1 Hz, 1H), 9.15 (d, *J* = 9.1 Hz, 1H), 7.76 – 7.70 (m, 2H), 7.57 – 7.51 (m, 2H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 166.26, 163.53, 145.13, 133.49, 130.22, 127.93, 127.82, 121.93, 119.94 (q, *J* = 257.4 Hz, 1H). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.69 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_3$ +Na^+: 256.0192, Found: 256.0196. **IR** (neat, cm^{-1}): υ 3285, 3073, 2981, 1648, 1552, 1444, 1320, 1248, 746.

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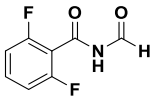
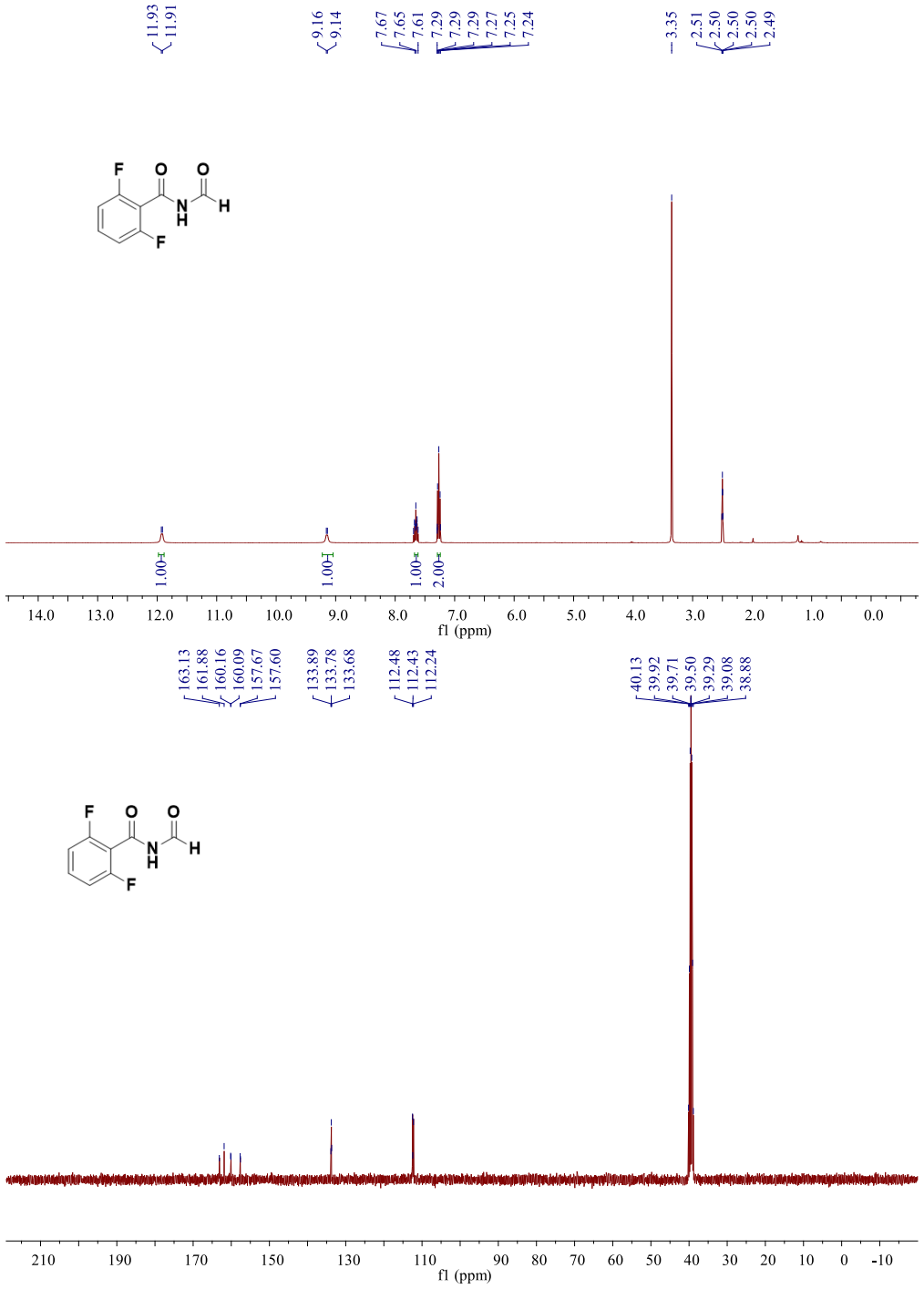
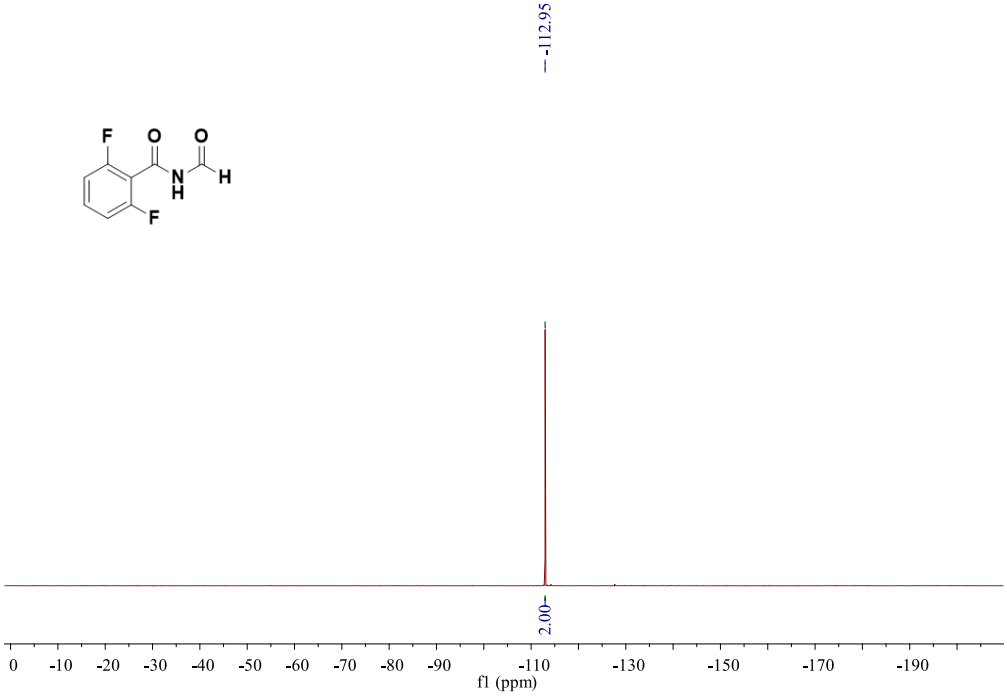
f1 (ppm)



2,6-Difluoro-*N*-Formylbenzamide (2bm)

petroleum ether / ethyl acetate = 5:1, white solid, 68% yield (25.2 mg). mp: 140 – 142℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.92 (d, *J* = 7.4 Hz, 1H), 9.15 (d, *J* = 7.4 Hz, 1H), 7.67 – 7.61 (m, 1H), 7.29 – 7.24 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 163.13, 161.88, 160.13 (d, *J* = 7.0 Hz), 157.63 (d, *J* = 7.0 Hz), 133.78 (t, *J* = 10.4 Hz), 112.4 (m). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -112.95 (s, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_5F_2NO_2$+Na^+: 208.0181, Found: 208.0188. **IR** (neat, cm^{-1}): υ 3277, 2945, 1722, 1668, 847, 775.

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O
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H
F
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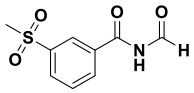
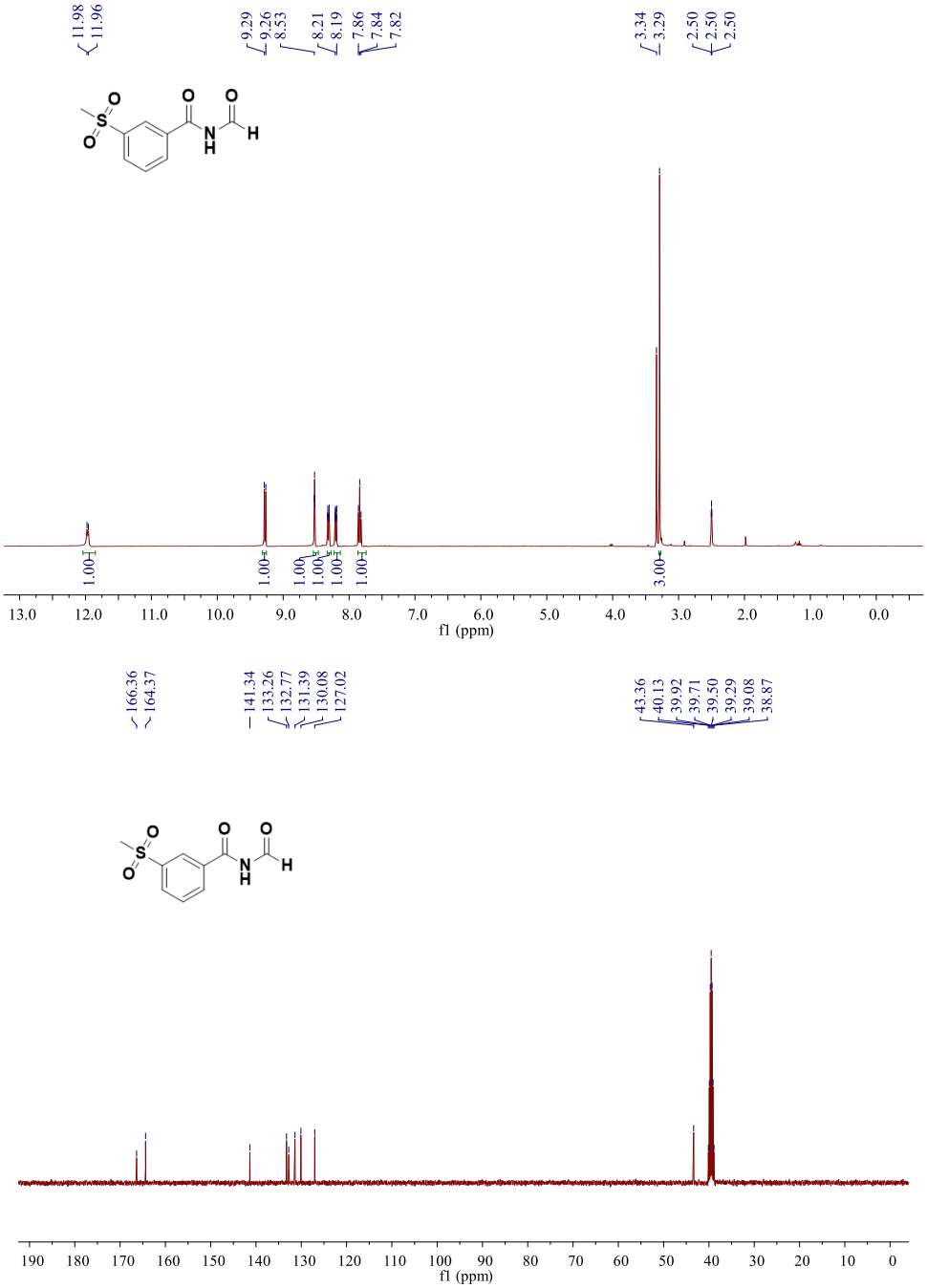
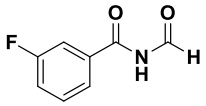
f1 (ppm)



***N*-Formyl-3-(methylsulfonyl)Benzamide (2bn)**

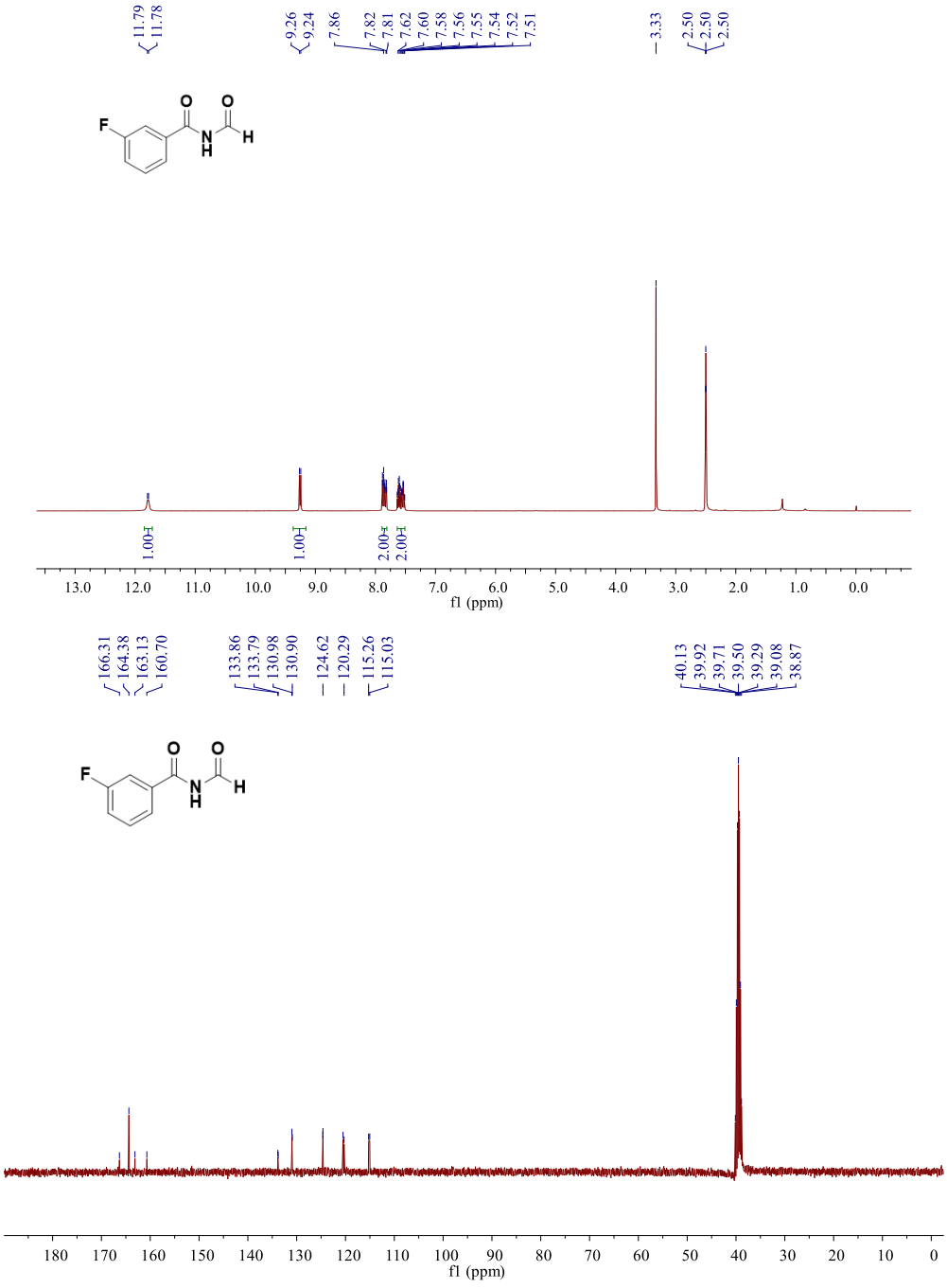
petroleum ether / ethyl acetate = 1:1, white solid, 47% yield (21.3 mg). mp: 45 – 46℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.97 (d, *J* = 8.8 Hz, 1H), 9.27 (d, *J* = 8.8 Hz, 1H), 8.52 (t, *J* = 1.6 Hz, 1H), 8.33 – 8.30 (m, 1H), 8.21 – 8.19 (m, 1H), 7.84 (t, *J* = 7.8 Hz, 1H), 3.29 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.36, 164.37, 141.34, 133.26, 132.77, 131.39, 130.08, 127.02, 43.36. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_9NO_4S+Na^+$: 250.0144, Found: 250.0145. **IR** (neat, cm^{-1}): υ 3200, 2927, 2854, 1730, 1667, 1523, 907, 865, 778.

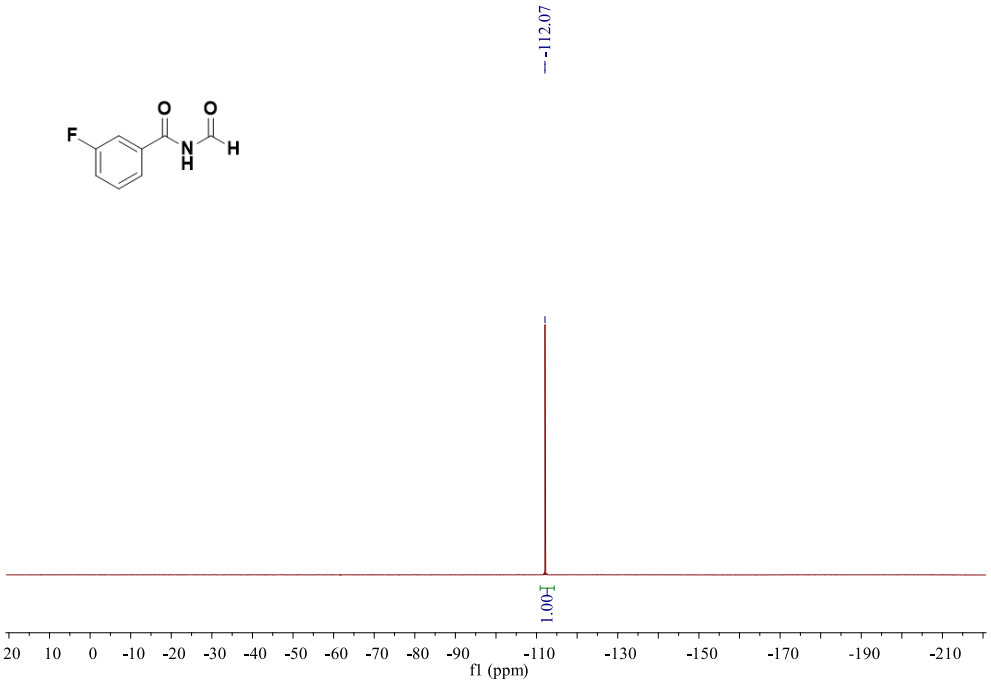




3-Fluoro-*N*-Formylbenzamide (2bo)

petroleum ether / ethyl acetate = 5:1, yellow solid, 62% yield (20.7 mg). mp: 144 – 146℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.79 (d, *J* = 7.0 Hz, 1H), 9.25 (d, *J* = 8.7 Hz, 1H), 7.86 – 7.81 (m, 2H), 7.64 – 7.51 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.31, 164.38, 161.91 (d, *J* = 245.0 Hz), 133.82 (d, *J* = 7.1 Hz), 130.94 (d, *J* = 8.0 Hz), 124.64 (d, *J* = 2.9 Hz), 120.40 (d, *J* = 21.2 Hz), 115.14 (d, *J* = 23.4 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -112.07 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6FNO_2$+Na^+: 190.0275, Found: 190.0276. **IR** (neat, cm^{-1}): υ 3336, 2948, 1686, 1548, 1440, 1327, 892, 791.

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F
O
N
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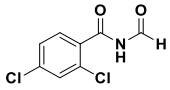
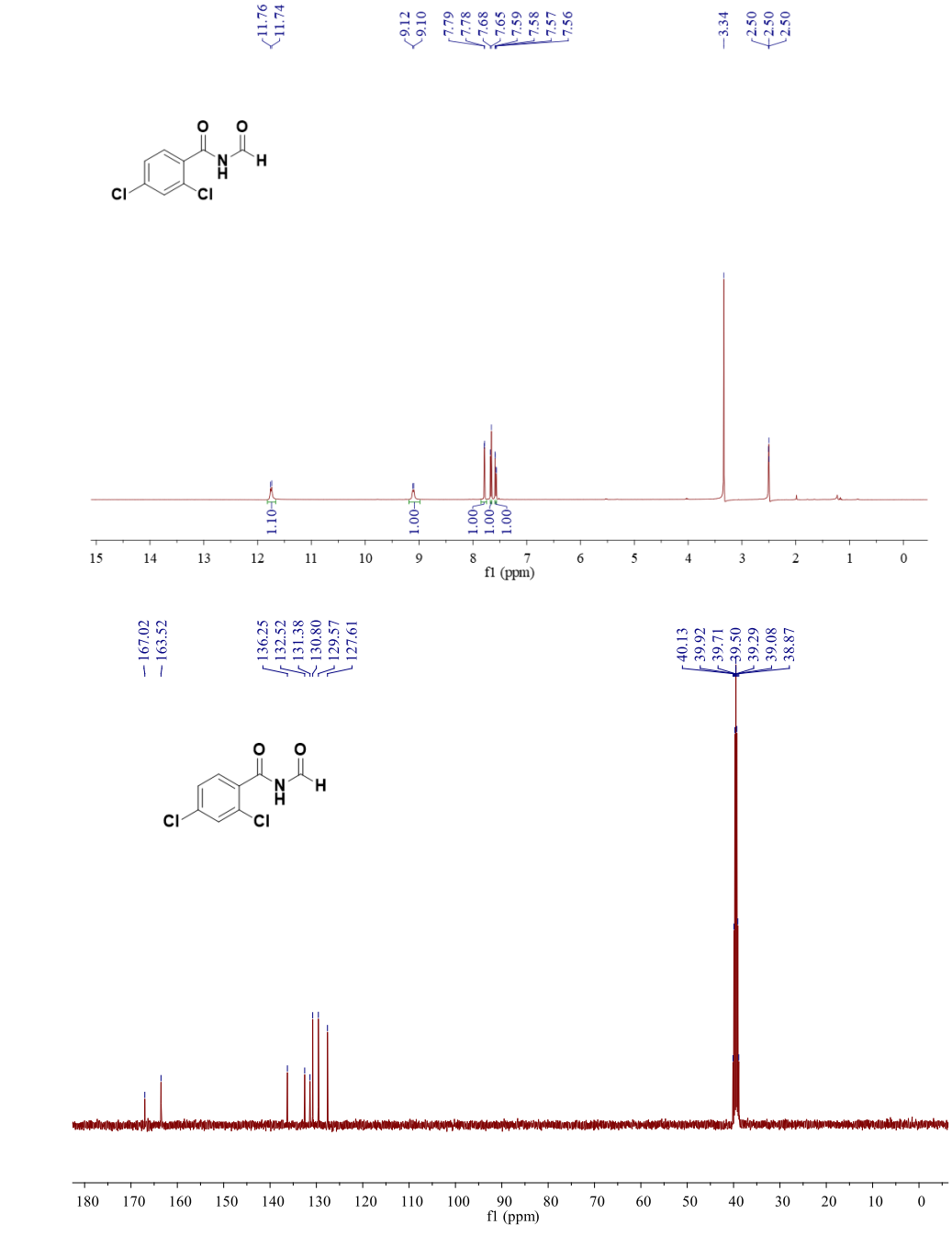
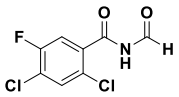
f1 (ppm)



2,4-Dichloro-*N*-Formylbenzamide (2bp)

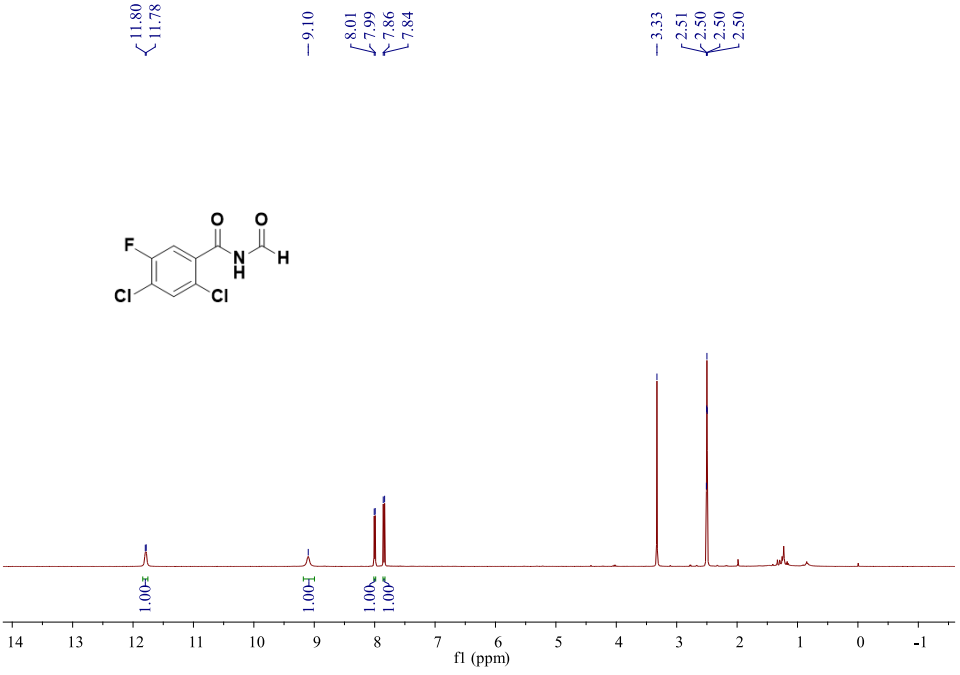
petroleum ether / ethyl acetate = 5:1, white solid, 67% yield (29.1 mg). mp: 150 – 152℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.75 (d, *J* = 8.7 Hz, 1H), 9.11 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 2.0 Hz, 1H), 7.67 (d, *J* = 8.3 Hz, 1H), 7.58 (dd, *J* = 8.3, 2.0 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 167.02, 163.52, 136.25, 132.52, 131.38, 130.80, 129.57, 127.61. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_5{}^{35}Cl_2NO_2$+Na^+: 239.9590, Found: 239.9600. Anal Calcd. For. $C_8H_5{}^{35,37}Cl_2NO_2$+Na^+: 241.9560, Found: 241.9588. **IR** (neat, cm^{-1}): υ 3335, 3076, 2871, 1781, 1696, 1599, 1404, 1374, 838, 781.

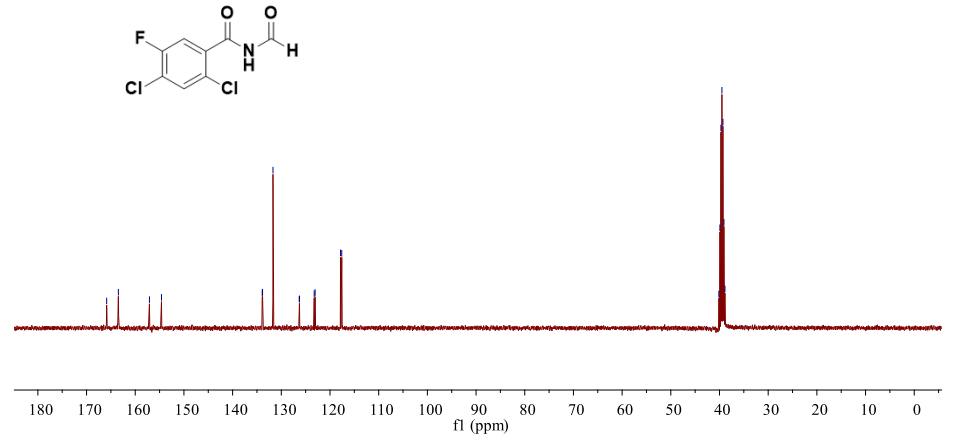


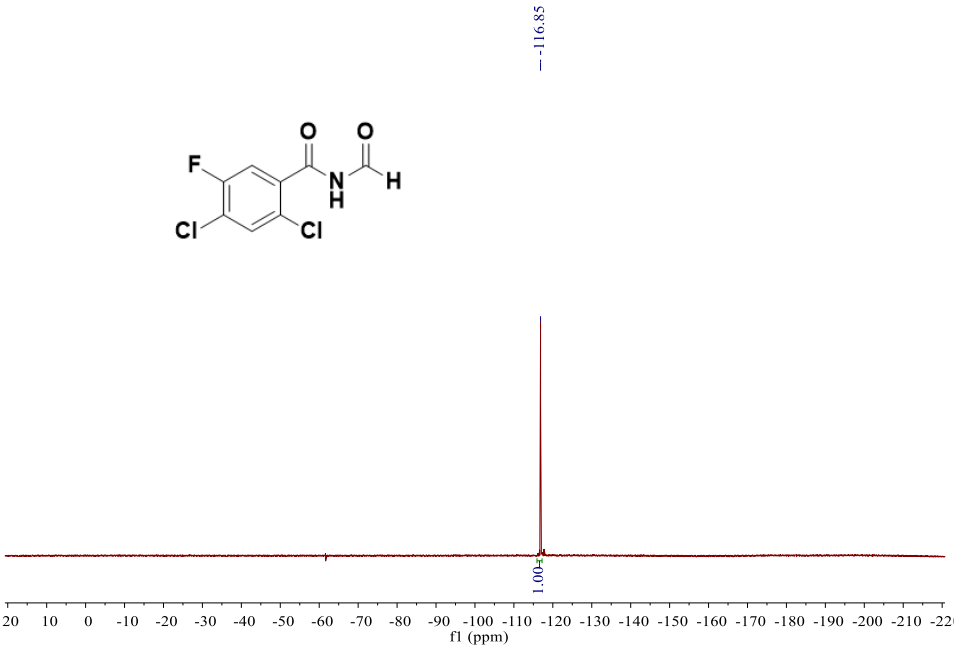


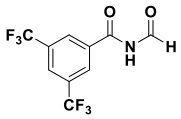
2,4-Dichloro-6-fluoro-*N*-formylbenzamide (2bq)

petroleum ether / ethyl acetate = 5:1, yellow solid, 58% yield (27.3 mg). mp: 110 – 112℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.79 (d, *J* = 6.2 Hz, 1H), 9.10 (s, 1H), 8.00 (d, *J* = 6.5 Hz, 1H), 7.85 (d, *J* = 9.1 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.87, 163.47, 155.85 (d, *J* = 248.8 Hz), 133.88 (d, *J* = 6.6 Hz), 131.71, 126.29 (d, *J* = 3.7 Hz), 123.15 (d, *J* = 18.9 Hz), 117.71 (d, *J* = 24.9 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -116.85 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_4{}^{35}Cl_2FNO_2$+Na^+: 257.9495, Found: 257.9487. Anal Calcd. For. $C_8H_4{}^{35,37}Cl_2FNO_2$+$Na^+$: 259.9466, Found: 259.9493. **IR** (neat, cm^{-1}): υ 3295, 2936, 1637, 1552, 1470, 1313, 886.



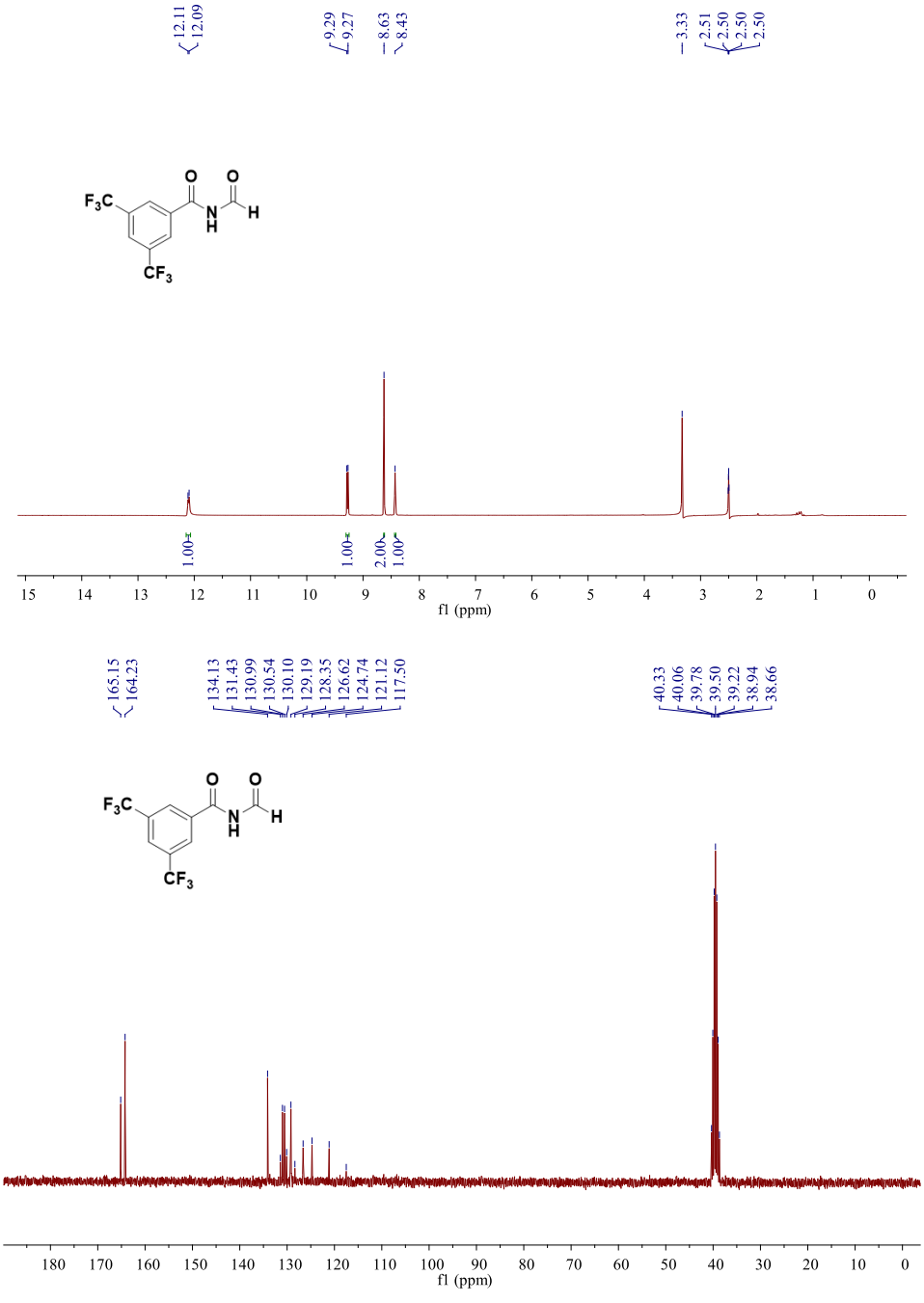


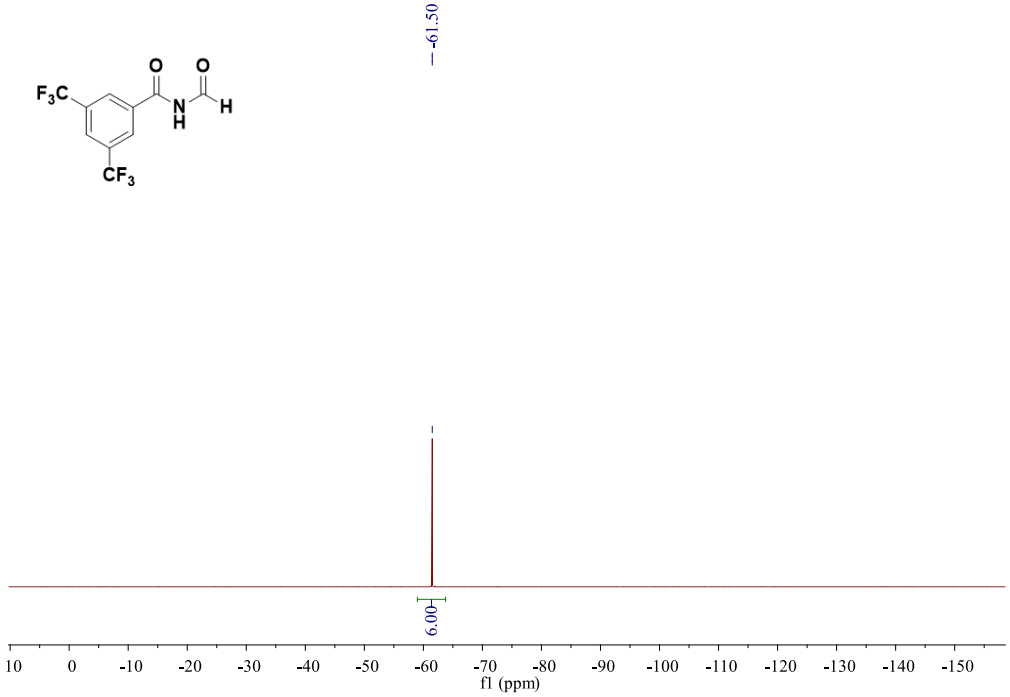

-116.85
F
O
O
N
H
H
Cl
Cl
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)



***N*-Formyl-3,5-Bis(trifluoromethyl)benzamide (2br)**

petroleum ether / ethyl acetate = 5:1, light yellow solid, 62% yield (35.3 mg). mp: 49 – 51℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 12.10 (d, *J* = 7.9 Hz, 1H), 9.28 (d, *J* = 8.4 Hz, 1H), 8.63 (s, 2H), 8.43 (s, 1H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 165.15, 164.23, 134.13, 130.76 (q, *J* = 33.6 Hz), 129.19 (q, *J* = 3.0 Hz), 126.62 (m), 122.93 (q, *J* = 273.0 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.50 (s, 6F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_5F_6NO_2$+Na^+: 308.0117, Found: 308.0117. **IR** (neat, cm^{-1}): υ 3335, 3075, 2871, 1701, 1635, 1570, 1486, 1322, 878.




F3C
O
N
H
O
H
CF3
-61.50
6.00
10
0
-10
-20
-30
-40
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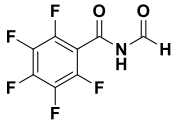
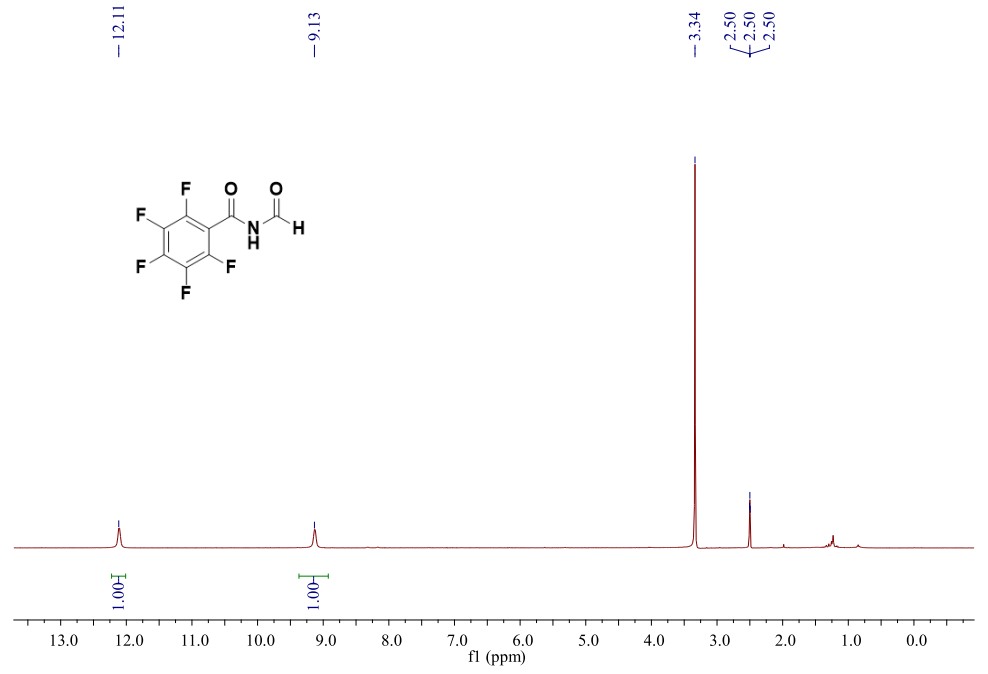
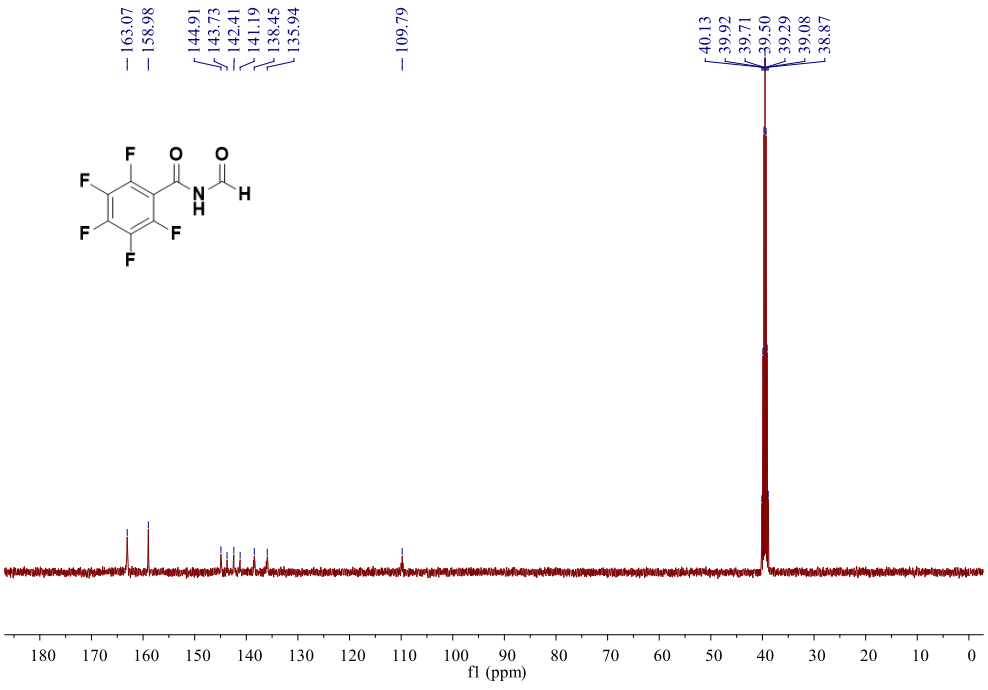
-110
-120
-130
-140
-150
f1 (ppm)

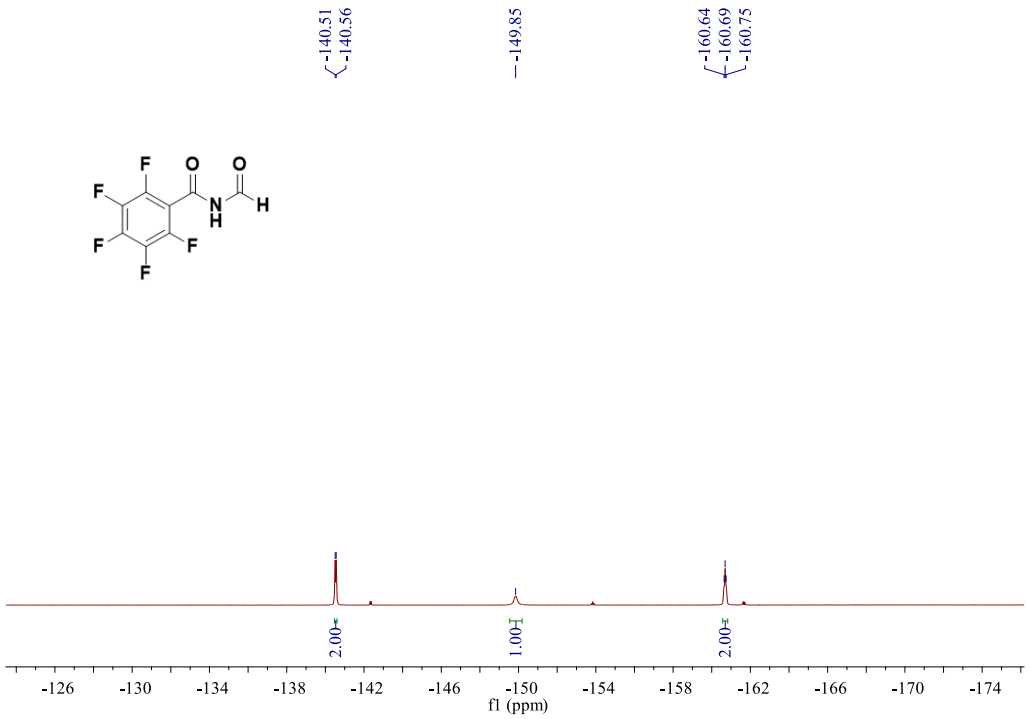


2,3,4,5,6-Pentafluoro-*N*-Formylbenzamide (2bs)

petroleum ether / ethyl acetate = 5:1, yellow oil, 63% yield (30.1 mg). **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 12.11 (s, 1H), 9.13 (s, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 163.07, 158.98, 146.66 (m), 142.46 (m), 137.20 (m), 109.79 (m). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -140.53 (d, *J* = 19.4 Hz, 2F), -149.85 (s, 1F), -160.69 (t, *J* = 19.4 Hz, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_2F_5NO_2$+Na^+: 261.9898, Found: 261.9878. **IR** (neat, cm^{-1}): υ 3396, 3242, 2960, 2850, 1729, 1673, 1503, 1379.






-140.51
-140.56
-149.85
-160.64
-160.69
-160.75
F
O
O
F
N
H
H
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F
F
2.00
1.00
2.00
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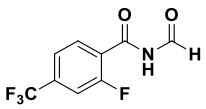
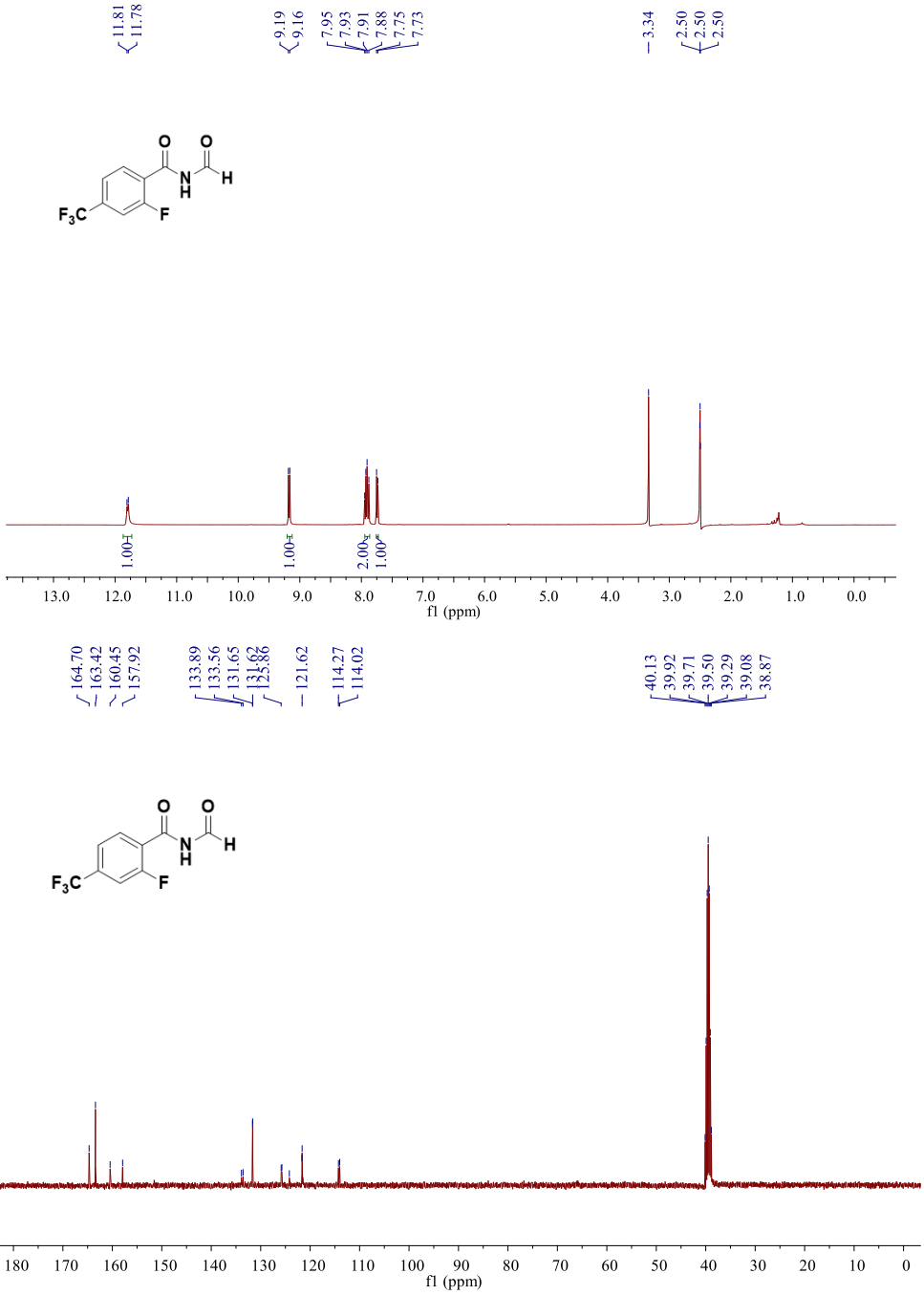
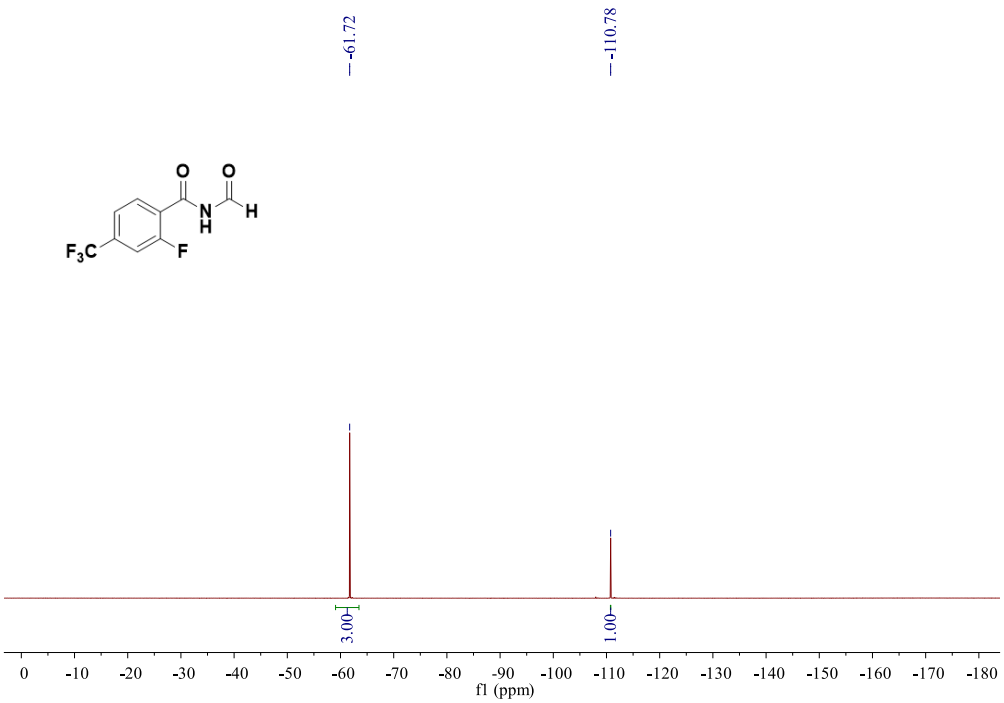
-158
-162
-166
-170
-174
f1 (ppm)



2-Fluoro-*N*-formyl-4-(trifluoromethyl)benzamide (2bt)

petroleum ether / ethyl acetate = 5:1, yellow solid, 67% yield (31.5 mg). mp: 108 – 110℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.80 (d, *J* = 8.6 Hz, 1H), 9.17 (d, *J* = 9.0 Hz, 1H), 7.92 (dd, *J* = 17.5, 9.4 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 164.70, 163.42, 159.19 (d, *J* = 254.6 Hz,), 133.73 (q, *J* = 33.1 Hz), 131.64 (d, *J* = 2.5 Hz), 125.79 (d, *J* = 15.0 Hz), 124.21 (d, *J* = 3.0 Hz), 121.64 (t, *J* = 4.0 Hz), 114.15 (m). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.72 (s, 3F), -110.78 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_5F_4NO_2$+Na^+: 258.0149, Found: 258.0190. **IR** (neat, cm^{-1}): υ 3265, 3056, 2851, 1642, 1507, 1409, 878, 779.




-61.72
-110.78
O
O
N
H
H
F3C
F
3.00
1.00
0
-10
-20
-30
-40
-50
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-90
-100
-110
-120
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-140
-150
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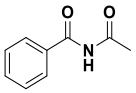
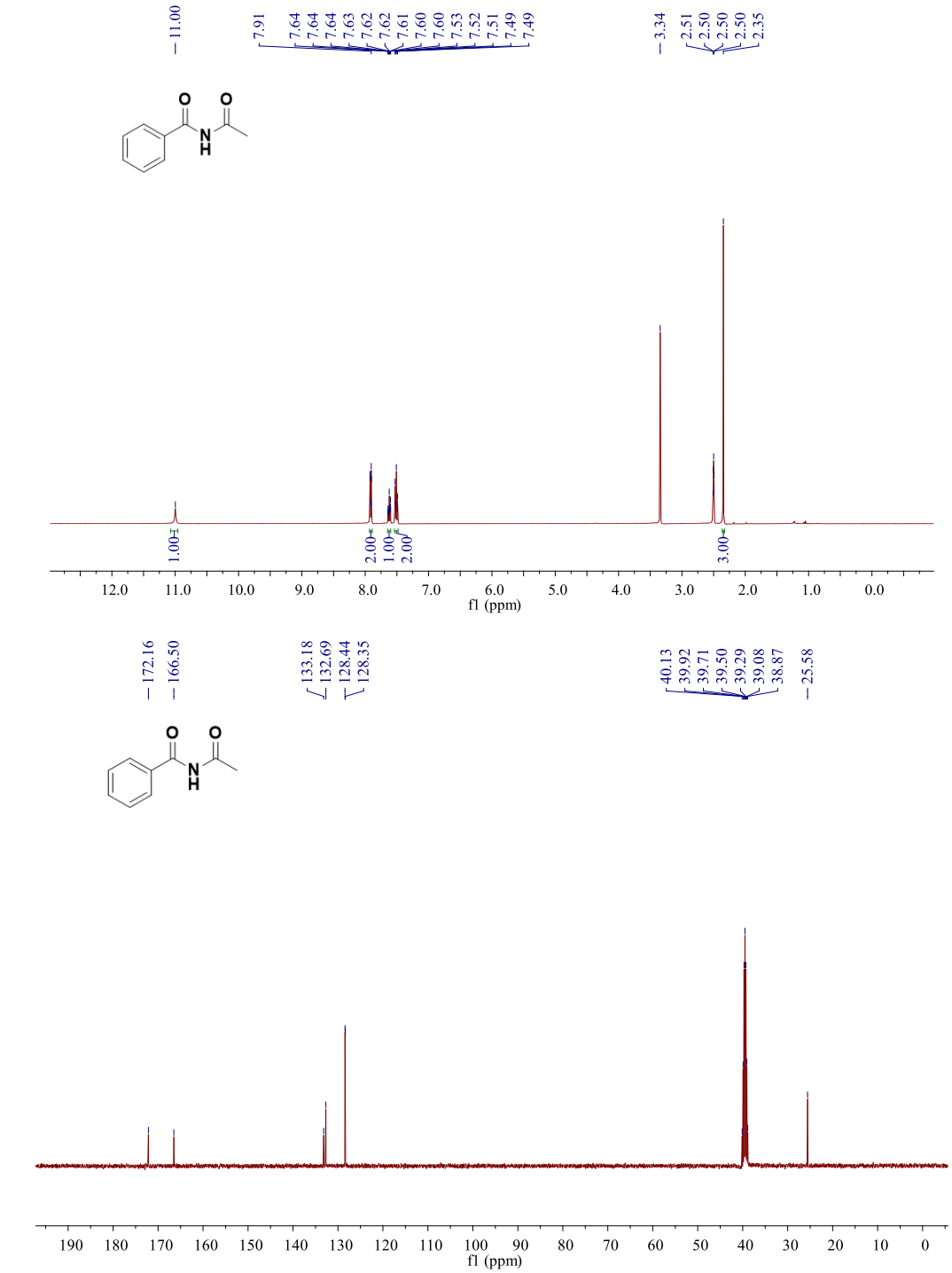
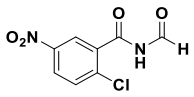
-170
-180
f1 (ppm)



***N*-Acetylbenzamide (2bu)**

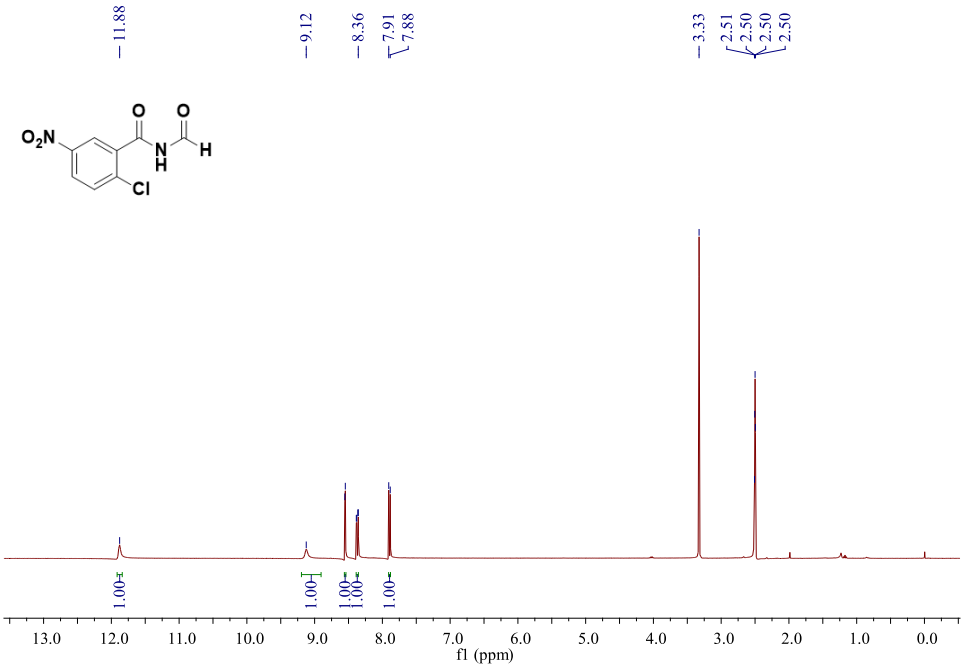
petroleum ether / ethyl acetate = 5:1, white solid, 68% yield (22.2 mg). mp: 107 – 109℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.00 (s, 1H), 7.93 – 7.90 (m, 2H), 7.64 – 7.60 (m, 1H), 7.53 – 7.49 (m, 2H), 2.35 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 172.16, 166.50, 133.18, 132.69, 128.44, 128.35, 25.58. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_9NO_2$ +Na^+: 186.0525, Found: 186.0526. **IR** (neat, cm^{-1}): υ 3300, 2984, 1736, 1568, 1466, 1330, 760, 634.

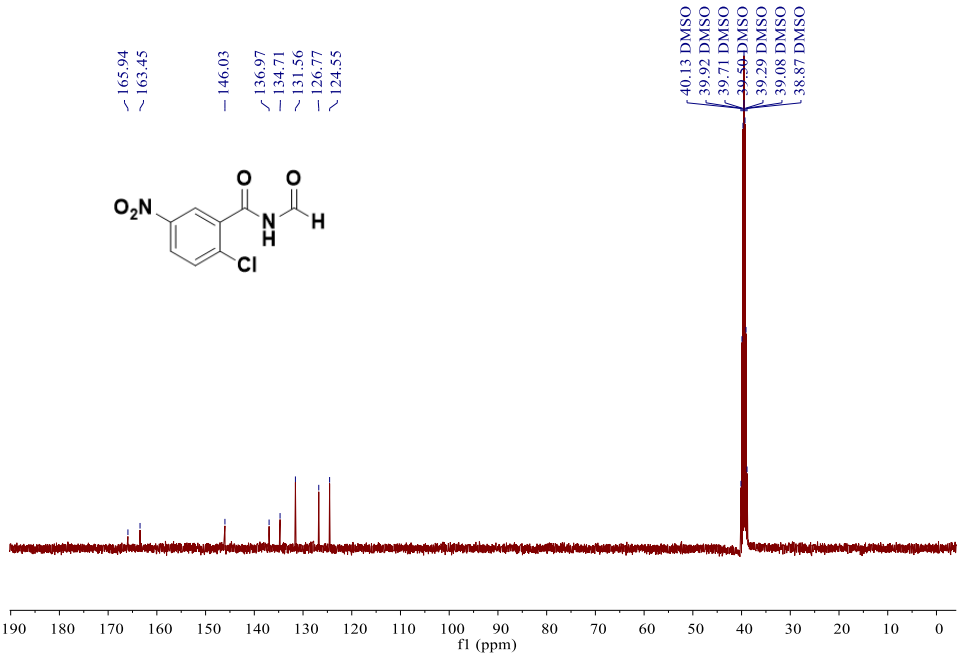


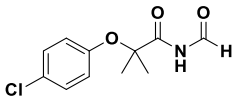


2-Chloro-*N*-Formyl-5-Nitrobenzamide (2bv)

petroleum ether / ethyl acetate = 2:1, white solid, 41% yield (18.7 mg). mp: 53 – 55℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.88 (s, 1H), 9.12 (s, 1H), 8.55 (d, *J* = 2.8 Hz, 1H), 8.37 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.90 (d, *J* = 8.8 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.94, 163.45, 146.03, 136.97, 134.71, 131.56, 126.77, 124.55. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_5{}^{35}ClN_2O_4$+Na^+: 250.9830, Found: 250.9835. Anal Calcd. For. $C_8H_5{}^{37}ClN_2O_4$+Na^+: 252.9801, Found: 252.9781. **IR** (neat, cm^{-1}): υ 3334, 3090, 2935, 1702, 1549, 1403, 1323, 863, 781.

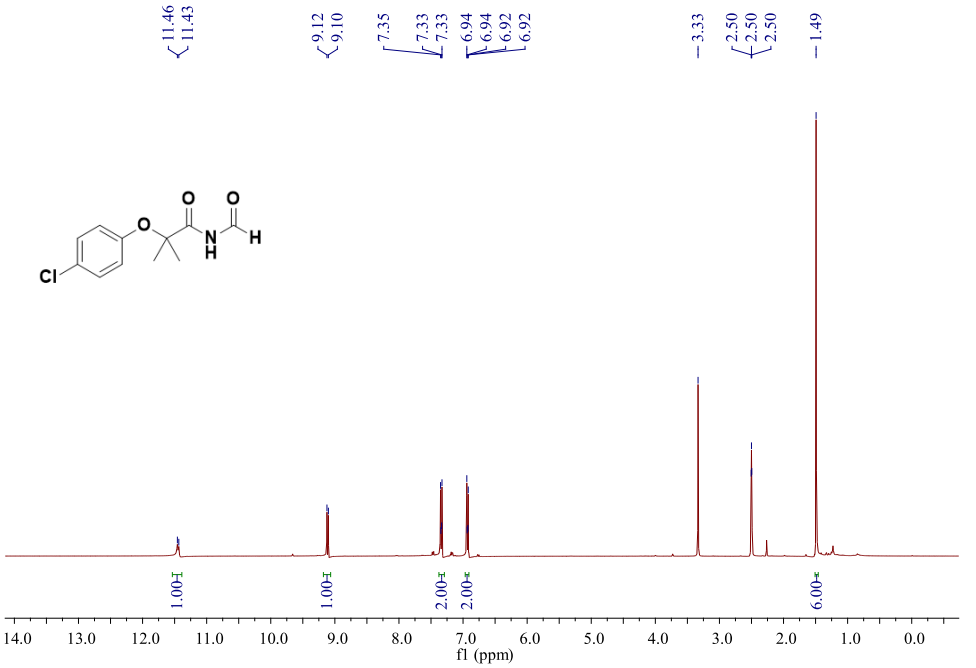


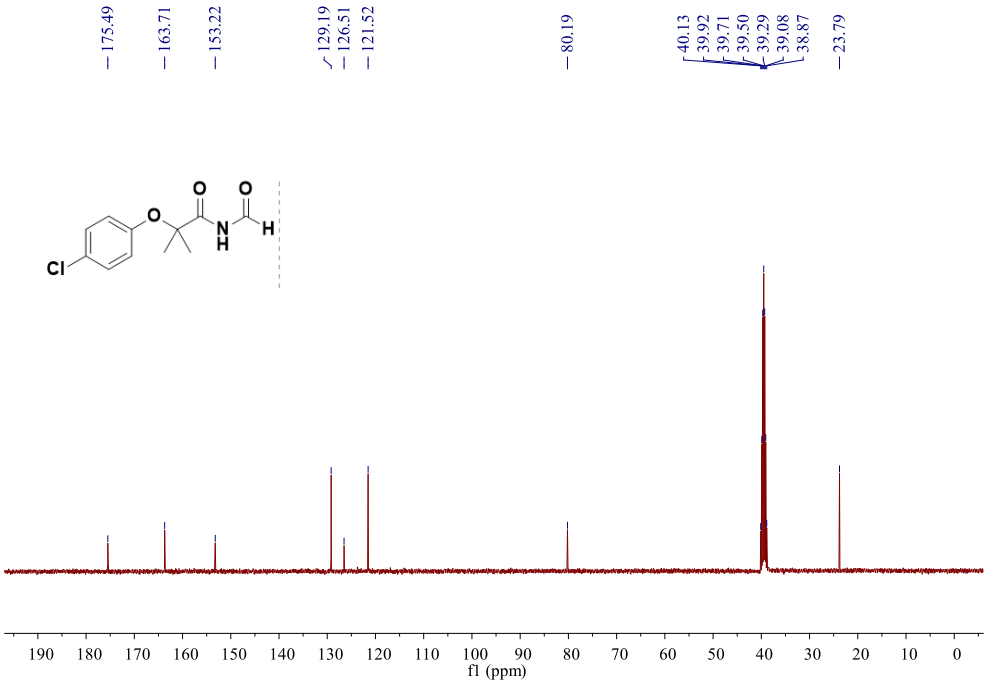


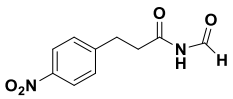


2-(4-chlorophenoxy)-*N*-Formyl-2-Methylpropanamide (2bw)

petroleum ether / ethyl acetate = 5:1, yellow solid, 46% yield (22.2 mg). mp: 45 – 47℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.45 (d, *J* = 9.0 Hz, 1H), 9.11 (d, *J* = 9.0 Hz, 1H), 7.35 – 7.33 (m, 2H), 6.94 – 6.92 (m, 2H), 1.49 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 175.49, 163.71, 153.22, 129.19, 126.51, 121.52, 80.19, 23.79. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{12}{}^{35}ClNO_3$+$Na^+$: 264.0398, Found: 264.0397. Anal Calcd. For. $C_{11}H_{12}{}^{37}ClNO_3$+$Na^+$: 266.0368, Found: 266.0366. **IR** (neat, cm^{-1}): υ 3069, 2917, 1741, 1698, 1594, 1450, 1382, 840, 790.

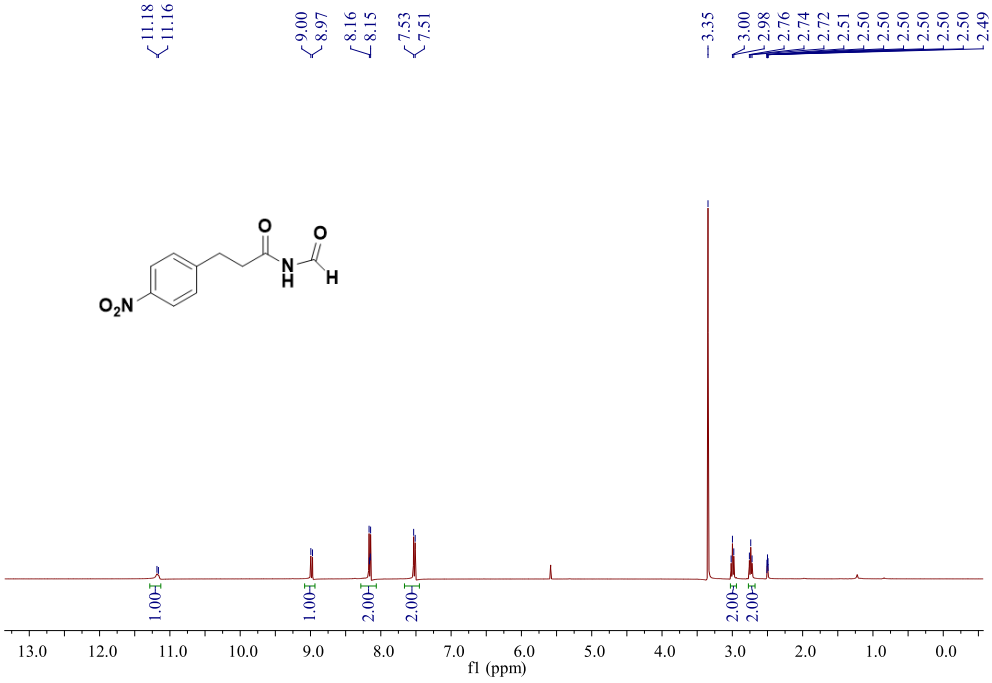


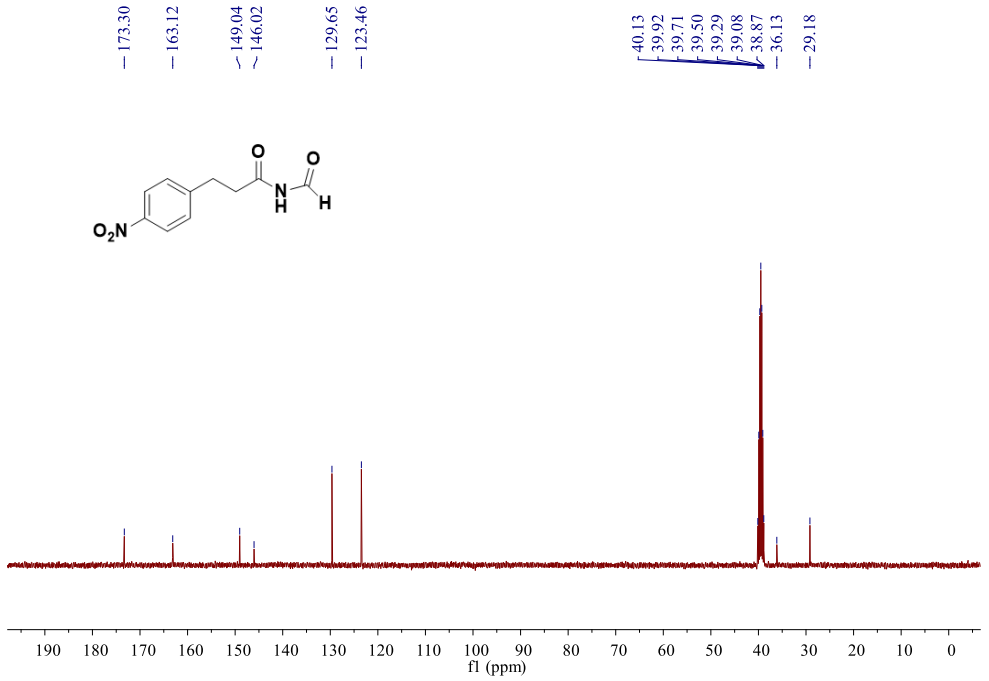


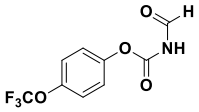


***N*-Formyl-3-(4-nitrophenyl)Propenamide (2bx)**

petroleum ether / ethyl acetate = 2:1, yellow solid, 39% yield (17.3 mg). mp: 112 – 114℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.17 (d, *J* = 8.5 Hz, 1H), 8.98 (d, *J* = 9.5 Hz, 1H), 8.17 – 8.15 (m, 2H), 7.53 – 7.51 (m, 2H), 3.00 (t, *J* = 7.5 Hz, 2H), 2.74 (t, *J* = 7.5 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 173.30, 163.12, 149.04, 146.02, 129.65, 123.46, 36.13, 29.18. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{10}N_2O_4$+Na^+: 245.0533, Found: 245.0537. **IR** (neat, cm^{-1}): υ 3290, 3104, 2939, 1635, 1559, 1452, 1343, 855, 747.

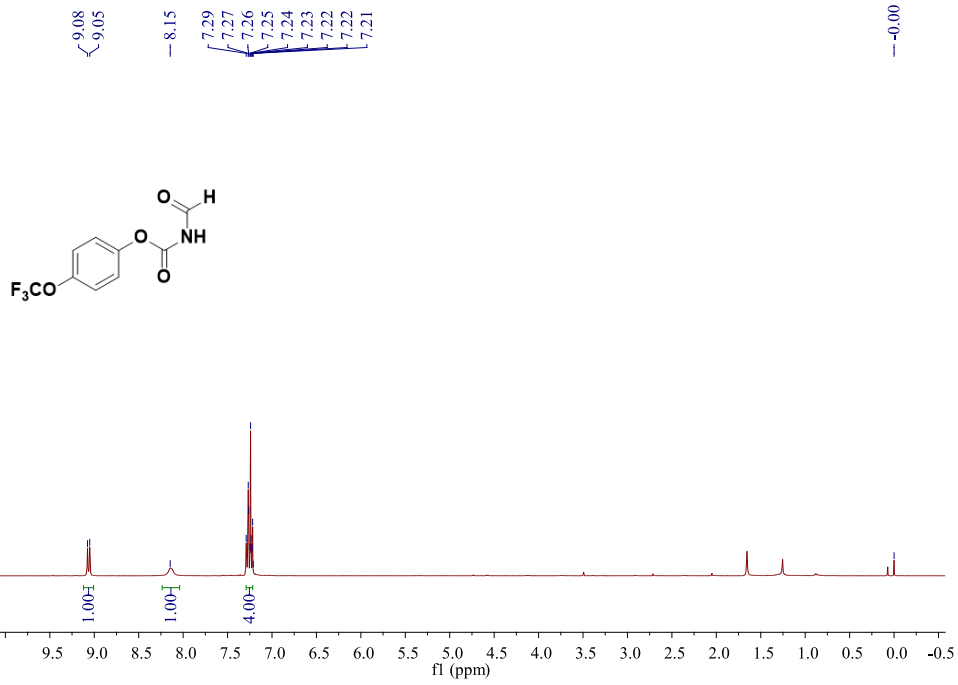


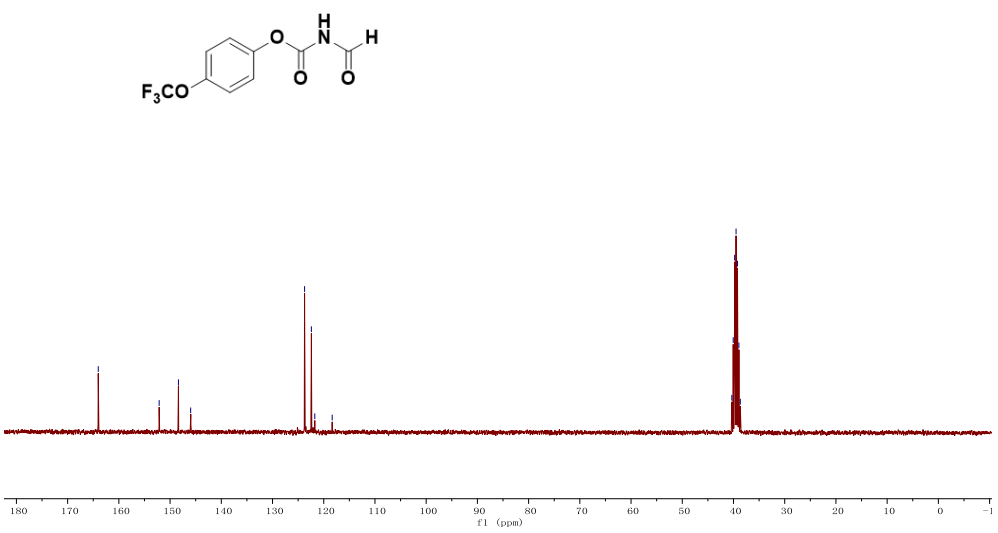


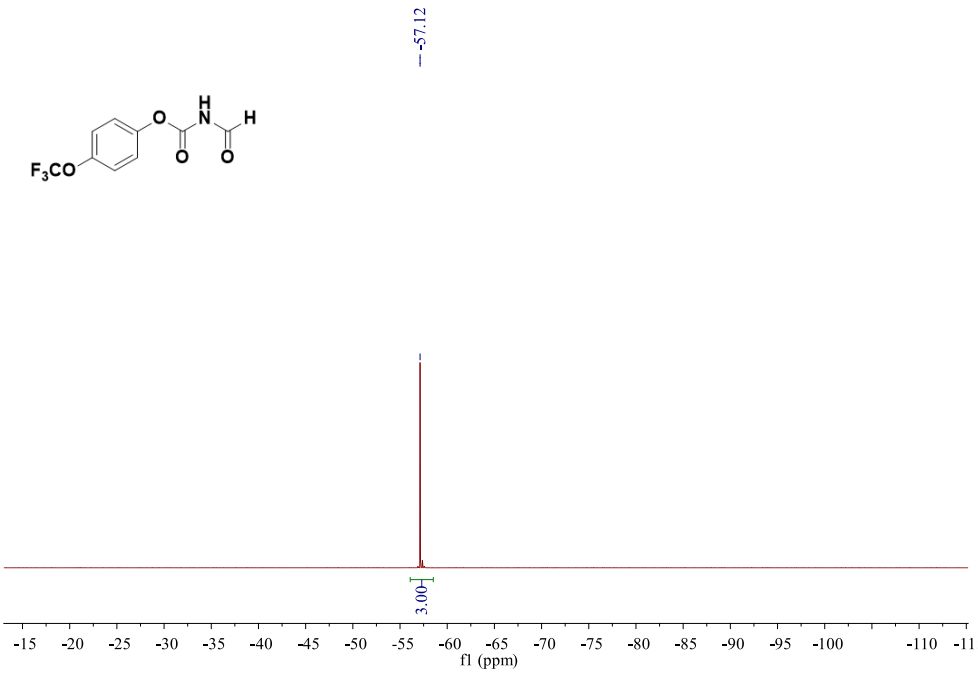


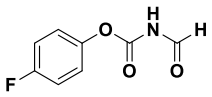
4-(trifluoromethoxy)Phenyl-Formylcarbamate (2by)

petroleum ether / ethyl acetate = 5:1, yellow solid, 66% yield (32.9 mg). mp: 56 – 58℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.06 (d, *J* = 10.0 Hz, 1H), 8.15 (s, 1H), 7.29 – 7.21 (m, 4H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 164.02, 152.13, 148.36, 145.98, 123.74, 122.41, 120.05 (q, *J* = 256.3 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -57.12 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_4$+Na^+: 272.0141, Found: 272.0144. **IR** (neat, cm^{-1}): υ 3295, 2945, 1781, 1693, 1599, 1489, 1343, 1261, 836, 796.



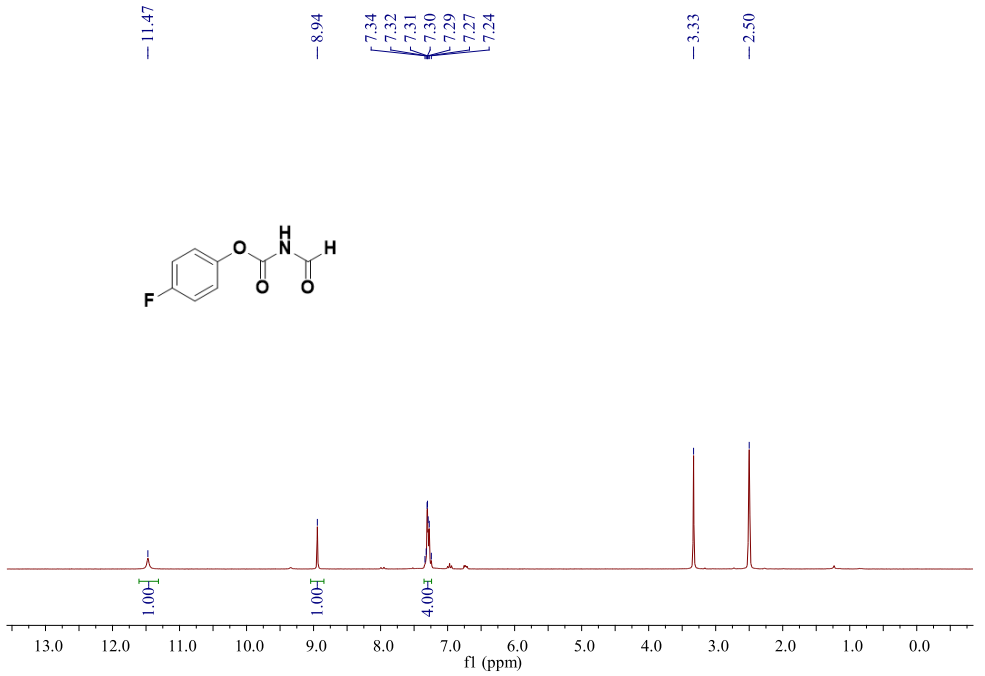


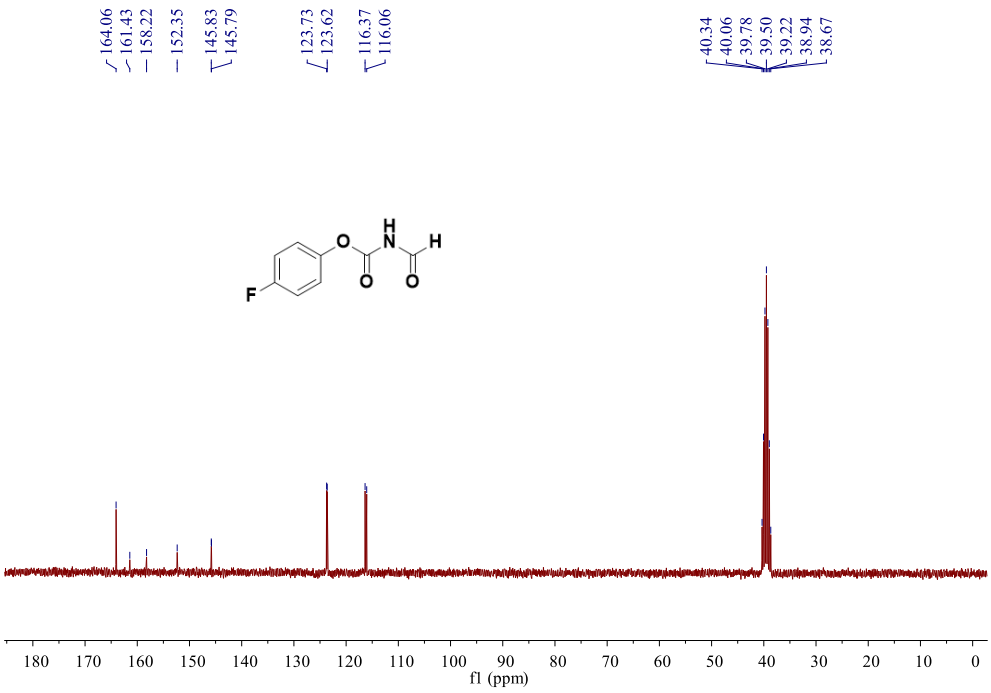

-57.12
3.00
-15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -110 -11
f1 (ppm)

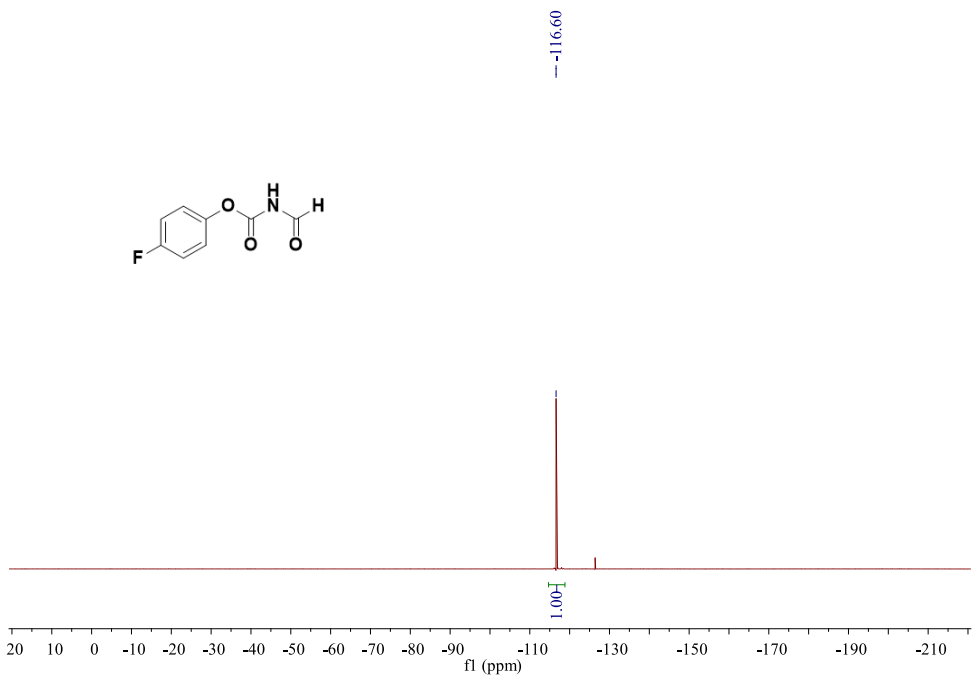


4-Fluorophenyl Formylcarbamate (2bz)

petroleum ether / ethyl acetate = 5:1, yellow solid, 74% yield (26.7 mg). mp: 61 – 63℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 11.47 (s, 1H), 8.94 (s, 1H), 7.34 – 7.24 (m, 4H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 164.06, 161.43, 155.29 (d, *J* = 442.8 Hz), 145.81 (d, *J* = 2.7 Hz), 123.68 (d, *J* = 8.8 Hz,), 116.21 (d, *J* = 23.6 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -116.60 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6FNO_3$+Na^+: 206.0224, Found: 206.0201. **IR** (neat, cm^{-1}): υ 3391, 2916, 2848, 1769, 1600, 1496, 1380, 1212, 825, 757.



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F
O
H
N
H
O
O
1.00
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10
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-10
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-130
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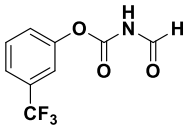
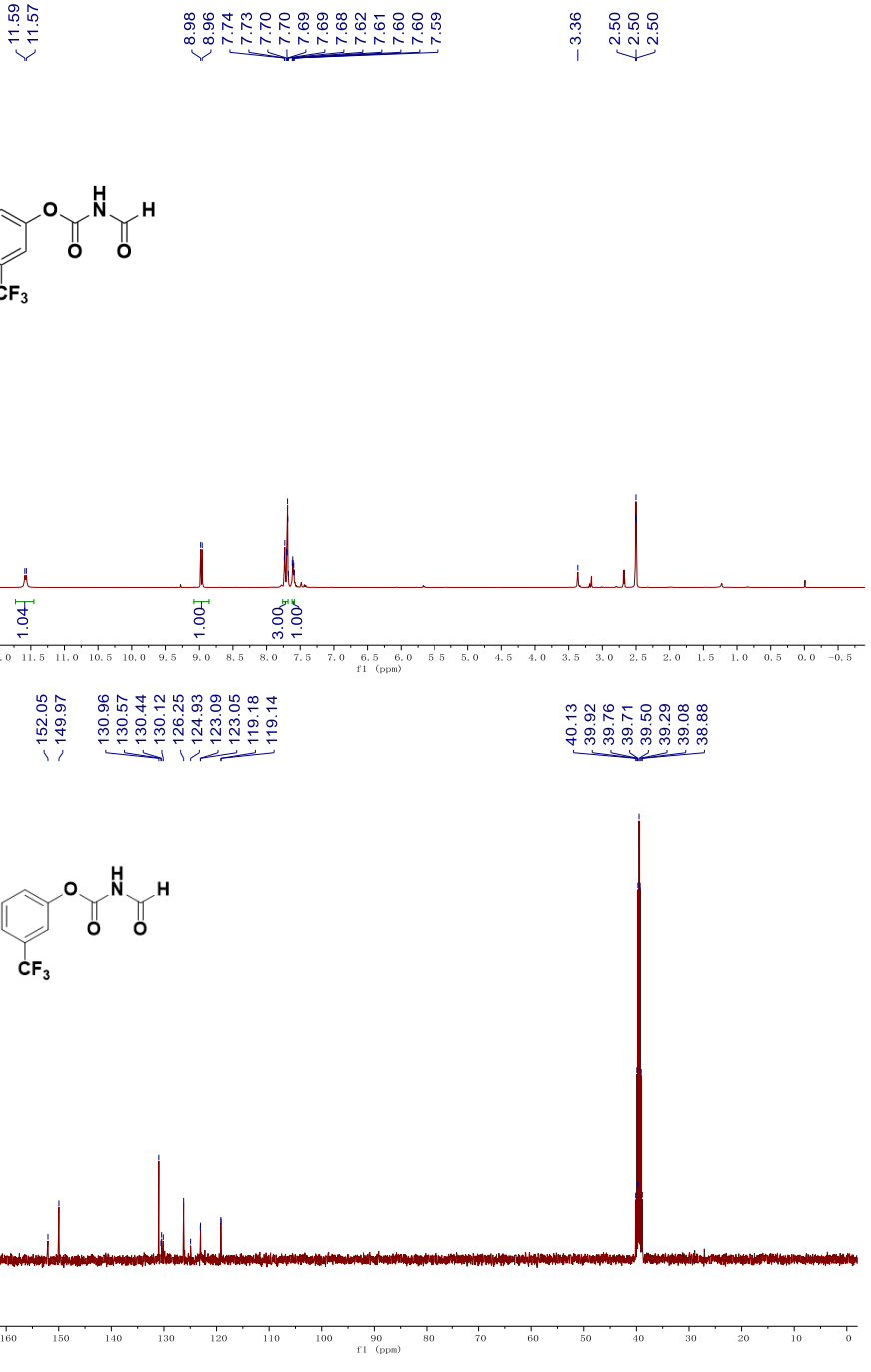
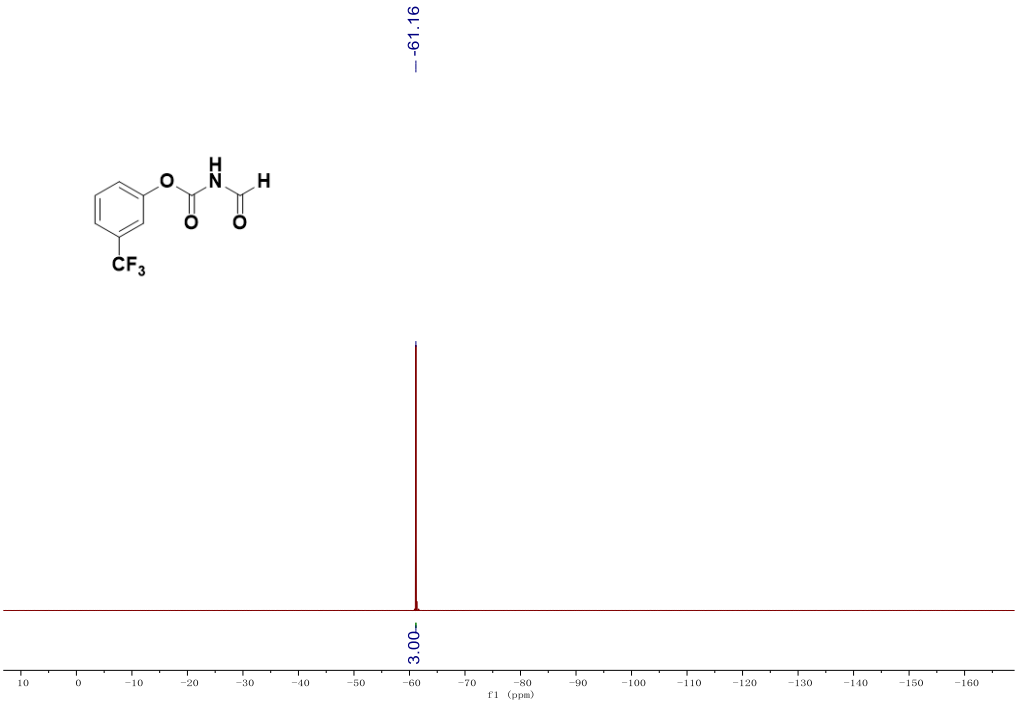
f1 (ppm)



3-(Trifluoromethyl)phenyl Formylcarbamate (2ca)

petroleum ether / ethyl acetate = 5:1, yellow solid, 70% yield (31.7 mg). mp: 95 – 97℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 11.58 (d, *J* = 9.3 Hz, 1H), 8.97 (d, *J* = 9.3 Hz, 1H), 7.74 – 7.68 (m, 3H), 7.62 – 7.59 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 164.04, 152.05, 149.97, 130.96, 130.28 (q, *J* = 32.1 Hz), 126.25, 123.58 (q, *J* = 270.8 Hz), 123.07 (d, *J* = 4.0 Hz), 119.16 (d, *J* = 4.0 Hz). **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.16 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_6F_3NO_3$ +Na^+: 256.0192, Found: 256.0192. **IR** (neat, cm^{-1}): υ 3332, 2959, 1779, 1695, 1483, 1253, 914, 807, 759.

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H
N
H
O
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−130
−140
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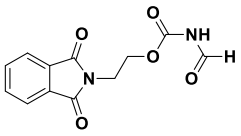
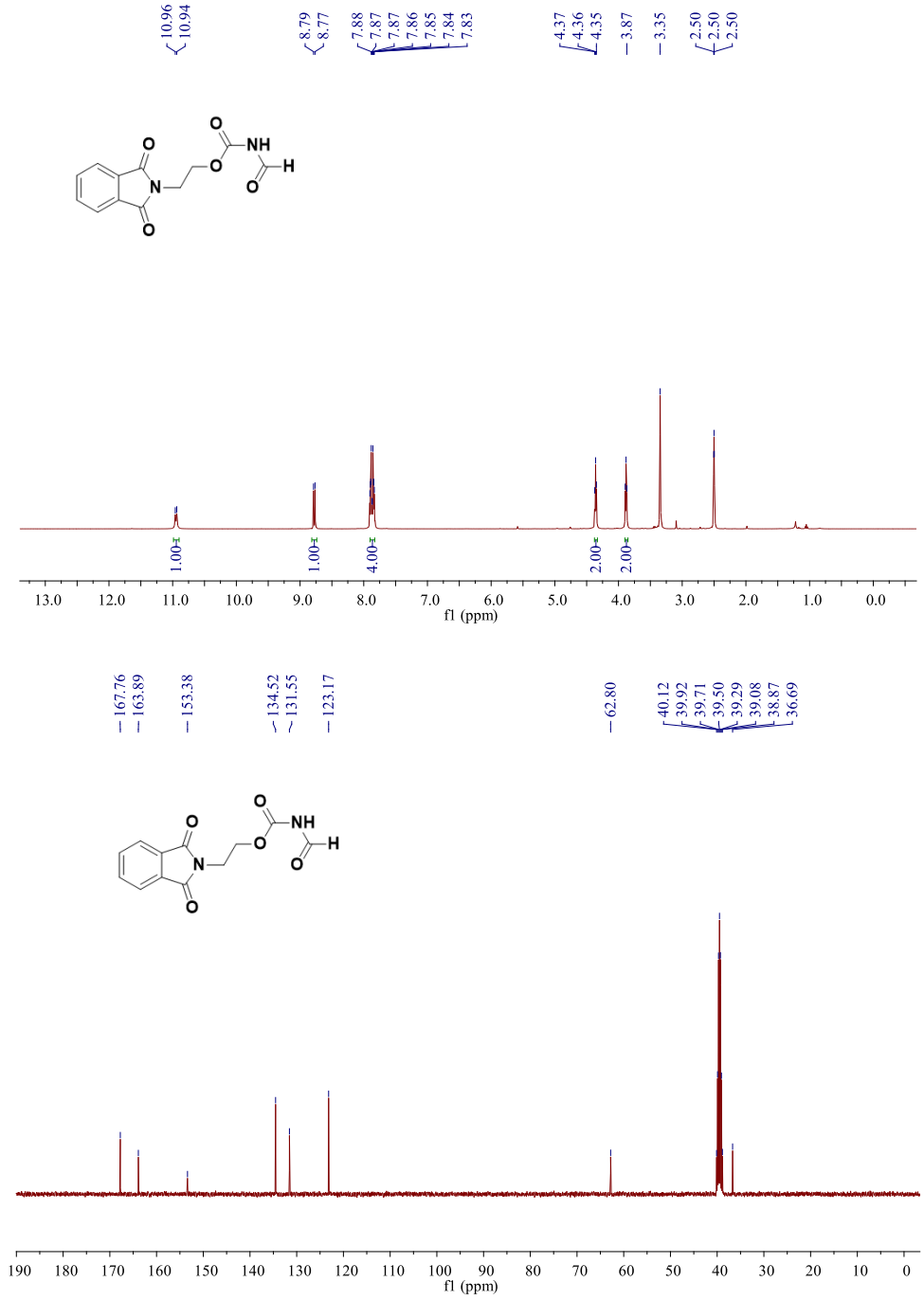
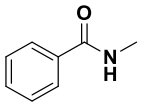
f1 (ppm)



2-(1,3-dioxoisoindolin-2-yl)Ethyl Formylcarbamate (2cb)

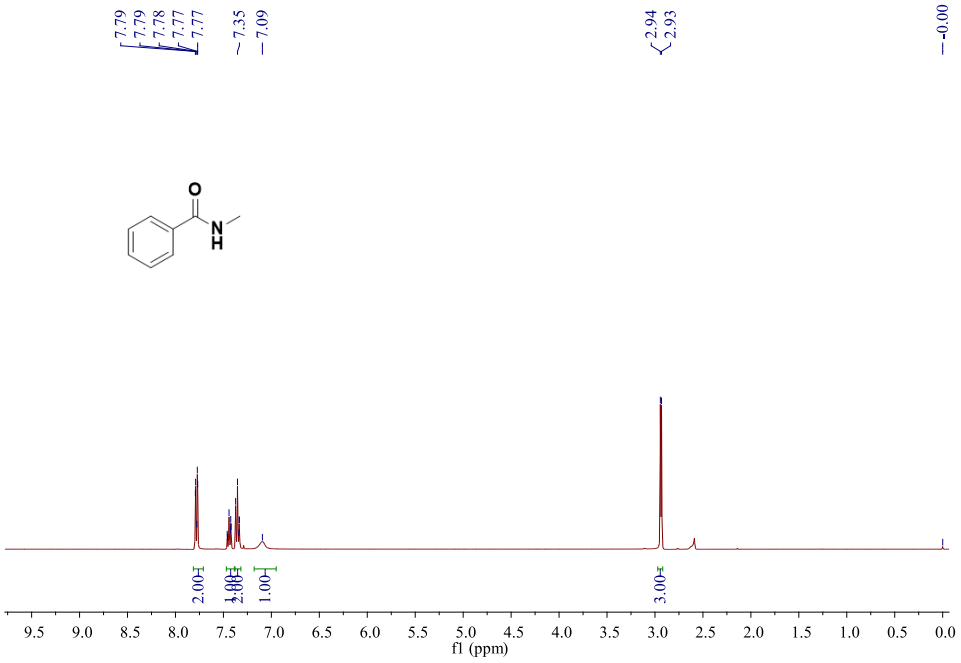
petroleum ether / ethyl acetate = 2:1, white solid, 62% yield (32.5 mg). mp: 87 – 89℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 10.95 (d, *J* = 9.5 Hz, 1H), 8.78 (d, *J* = 9.5 Hz, 1H), 7.88 – 7.83 (m, 4H), 4.36 (t, *J* = 5.3 Hz, 2H), 3.88 (t, *J* = 5.3 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 167.76, 163.89, 153.38, 134.52, 131.55, 123.17, 62.80, 36.69. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{12}H_{10}N_2O_5$+Na^+: 285.0482, Found: 285.0484. **IR** (neat, cm^{-1}): υ 3386, 3270, 1761, 1689, 1358, 1192, 825, 715.

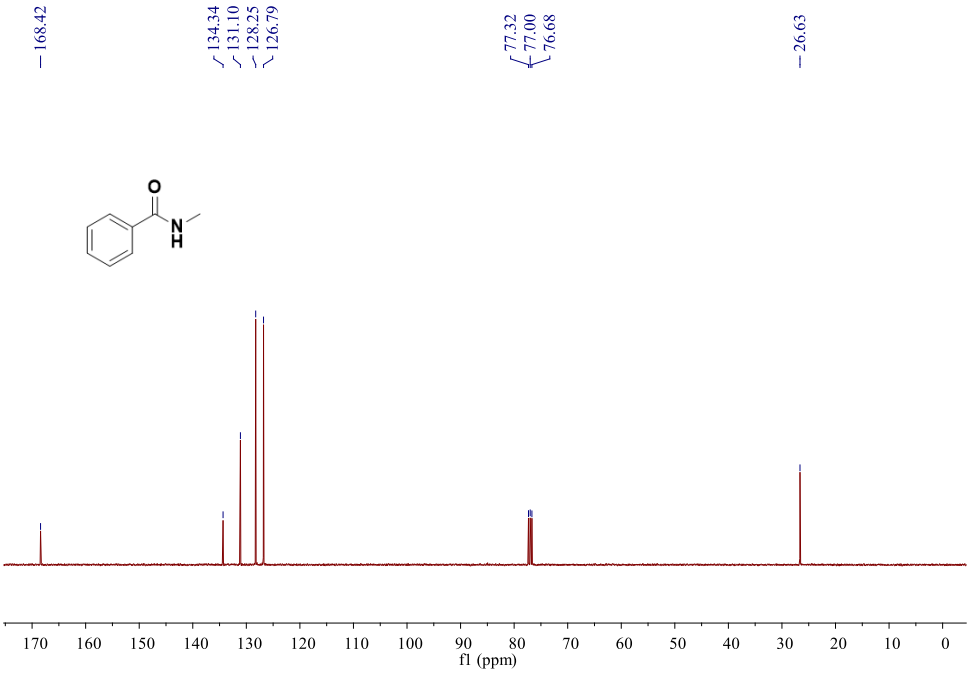


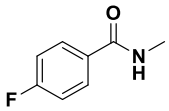


***N*-methylbenzamide (3a)**

petroleum ether / ethyl acetate = 2:1, white solid, 90% yield (23.7 mg). mp: 70 –72°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 – 7.77 (m, 2H), 7.46 – 7.42 (m, 1H), 7.37 – 7.33 (m, 2H), 7.09 (s, 1H), 2.94 (d, *J* = 4.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.42, 134.34, 131.10, 128.25, 126.79, 26.63. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_9NO+Na^+$: 136.0576, Found: 136.0580. **IR** (neat, cm^{-1}): υ 3282, 2940, 1642, 1594, 1489, 1300, 746, 696.

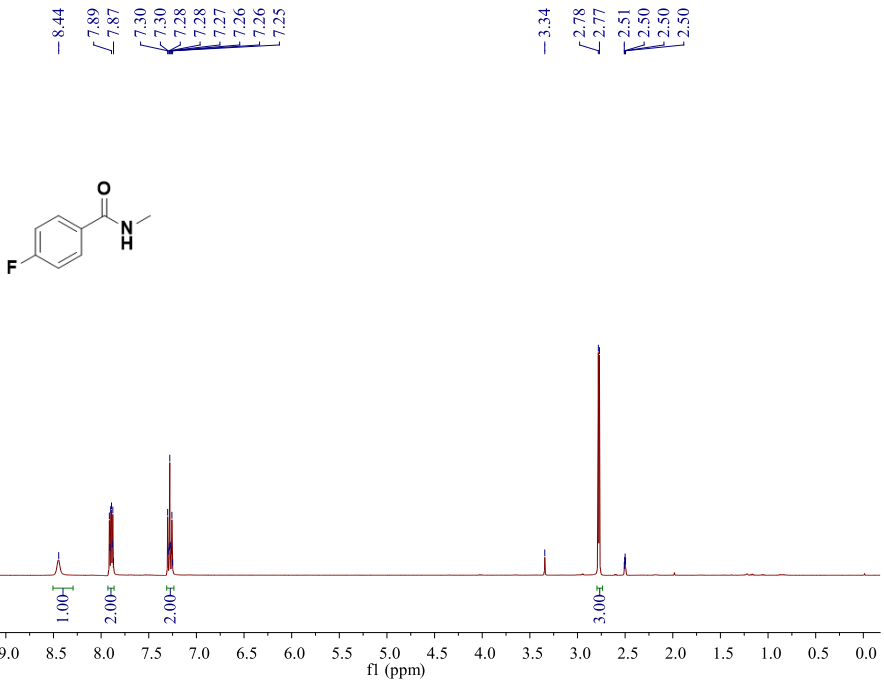


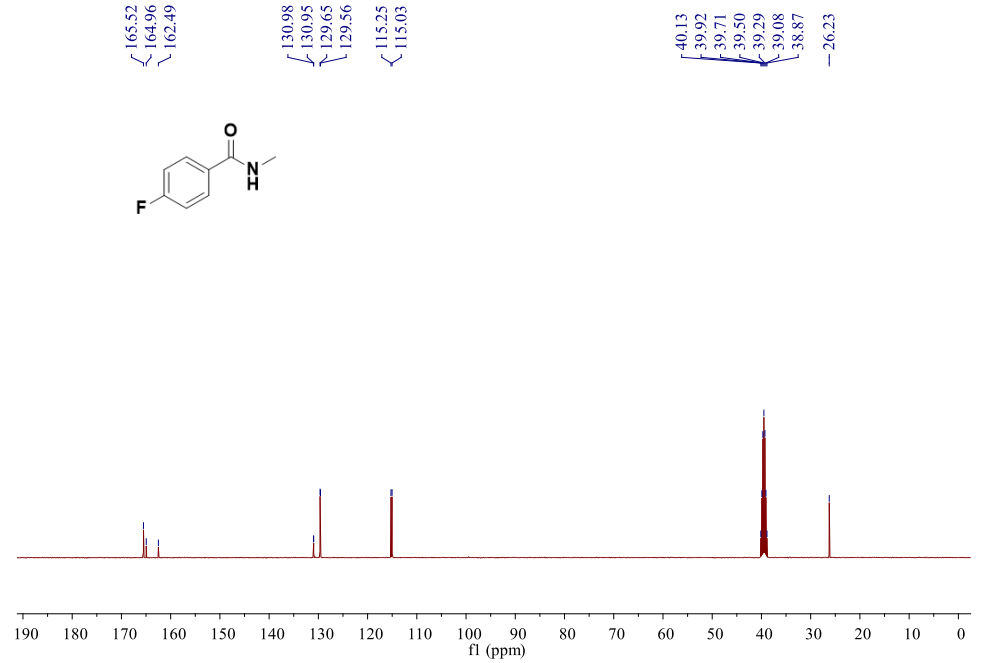


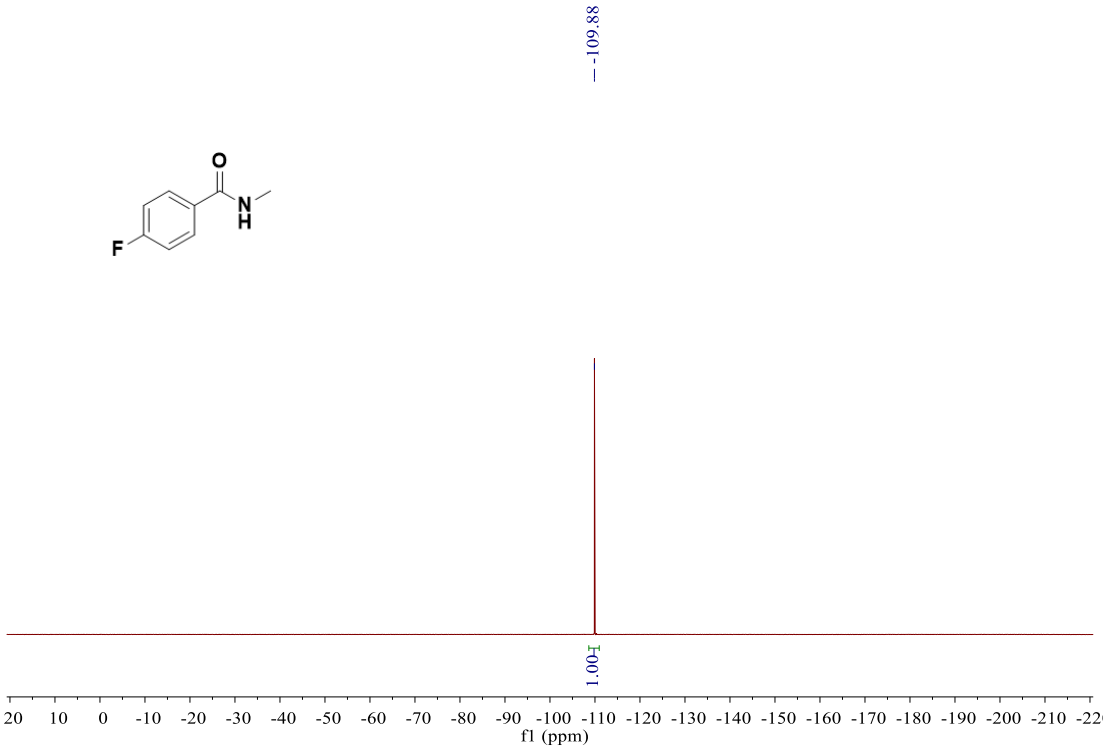


4-Fluoro-*N*-methylbenzamide (3b)

petroleum ether / ethyl acetate = 2:1, white solid, 66% yield (20.2 mg). mp: 128 – 130℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.44 (s, 1H), 7.91 – 7.87 (m, 2H), 7.30 – 7.25 (m, 2H), 2.78 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.52, 163.72 (d, *J* = 248.0 Hz), 130.97 (d, *J* = 2.9 Hz, 1H), 129.61 (d, *J* = 8.9 Hz, 4H), 115.14 (d, *J* = 21.7 Hz, 5H), 26.23. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -109.88 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8FNO+Na^+$: 176.0482, Found: 176.0489. **IR** (neat, cm^{-1}): υ 3346, 1634, 1589, 1436, 1319, 845, 793.



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O
N
H
F
1.00
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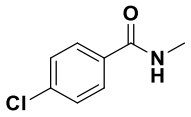
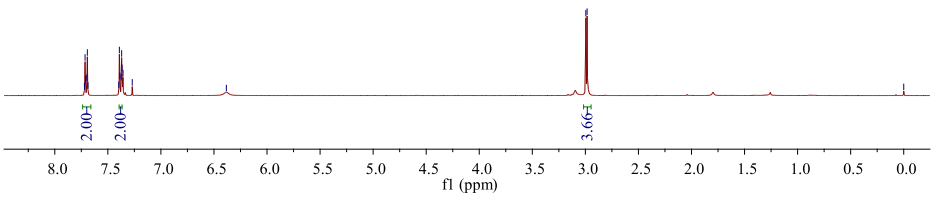
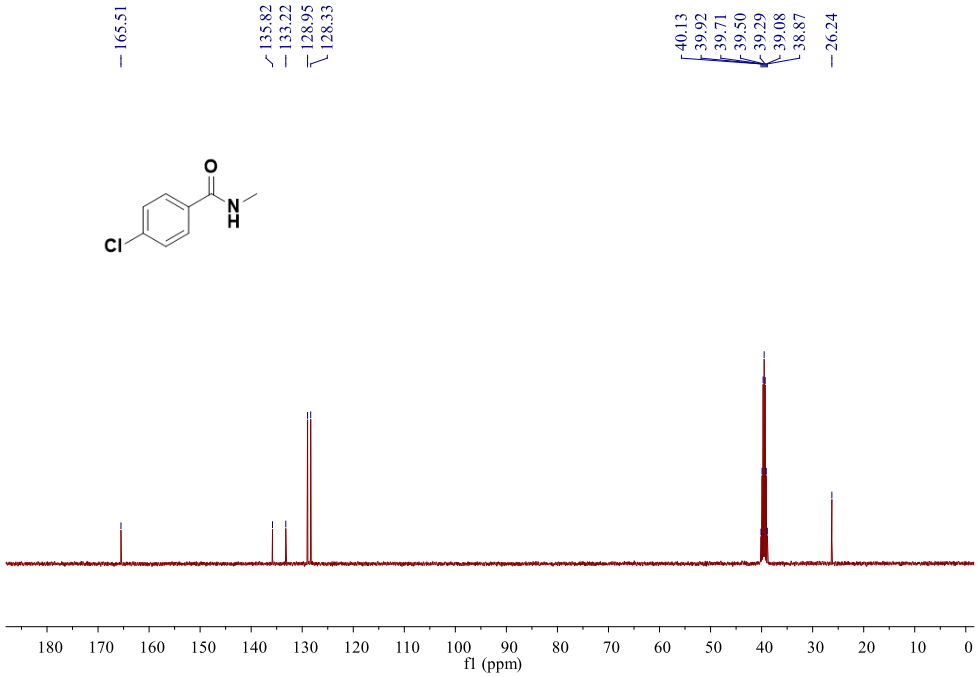
f1 (ppm)

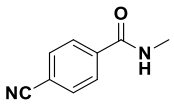


4-Chloro-*N*-methylbenzamide (3c)

petroleum ether / ethyl acetate = 2:1, white solid, 86% yield (29.1 mg). mp: 159 – 161℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.69 (m, 2H), 7.49 – 7.36 (m, 2H), 6.38 (s, 1H), 2.99 (d, *J* = 4.8 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.51, 135.82, 133.22, 128.95, 128.33, 26.24. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8{}^{35}ClNO+Na^+$: 192.0187, Found: 192.0185; Anal Calcd. For. $C_8H_8{}^{37}ClNO+Na^+$: 194.0157, Found: 194.0194. **IR** (neat, cm^{-1}): υ 3343, 1634, 1601, 1545, 1487, 841, 794.

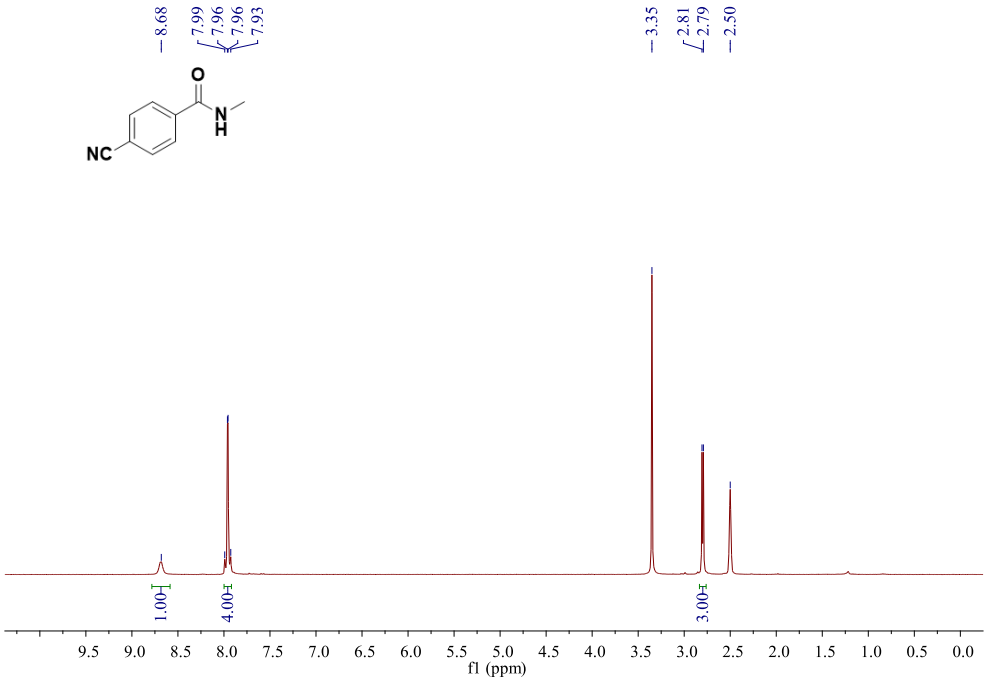


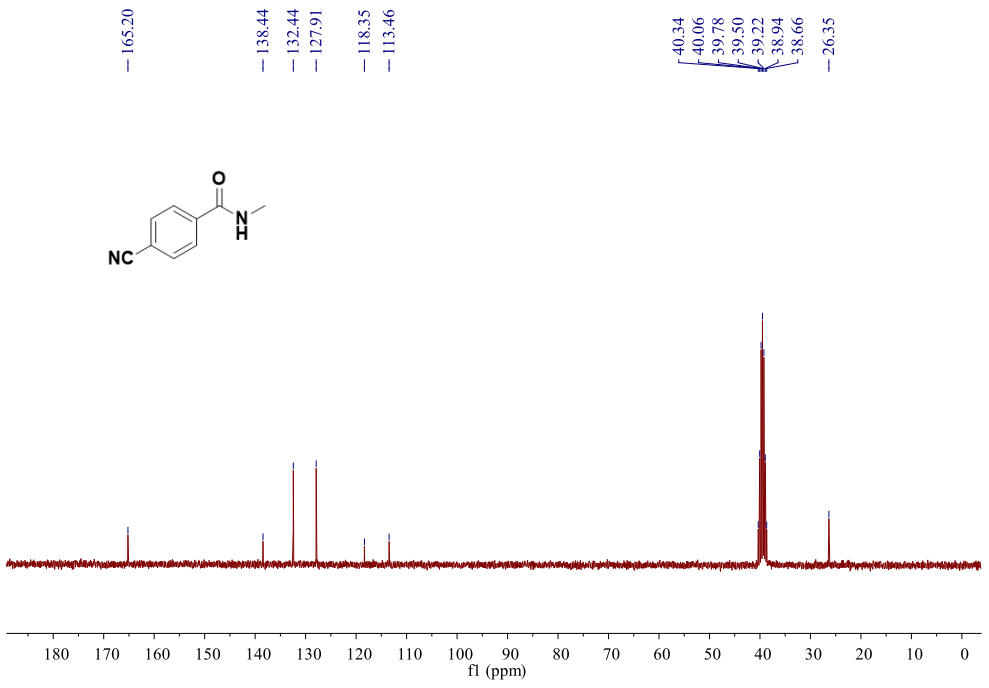


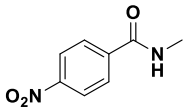


4-Cyano-*N*-methylbenzamide (3d)

petroleum ether / ethyl acetate = 1:1, white solid, 57% yield (18.2 mg). mp: 203 – 205℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 8.68 (s, 1H), 7.99 – 7.93 (m, 4H), 2.80 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 165.20, 138.44, 132.44, 127.91, 118.35, 113.46, 26.35. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8N_2O+Na^+$: 183.0529, Found: 183.0525. **IR** (neat, cm^{-1}): υ 3336, 2943, 2227, 1722, 1638, 1550, 1498, 1349, 854, 760.

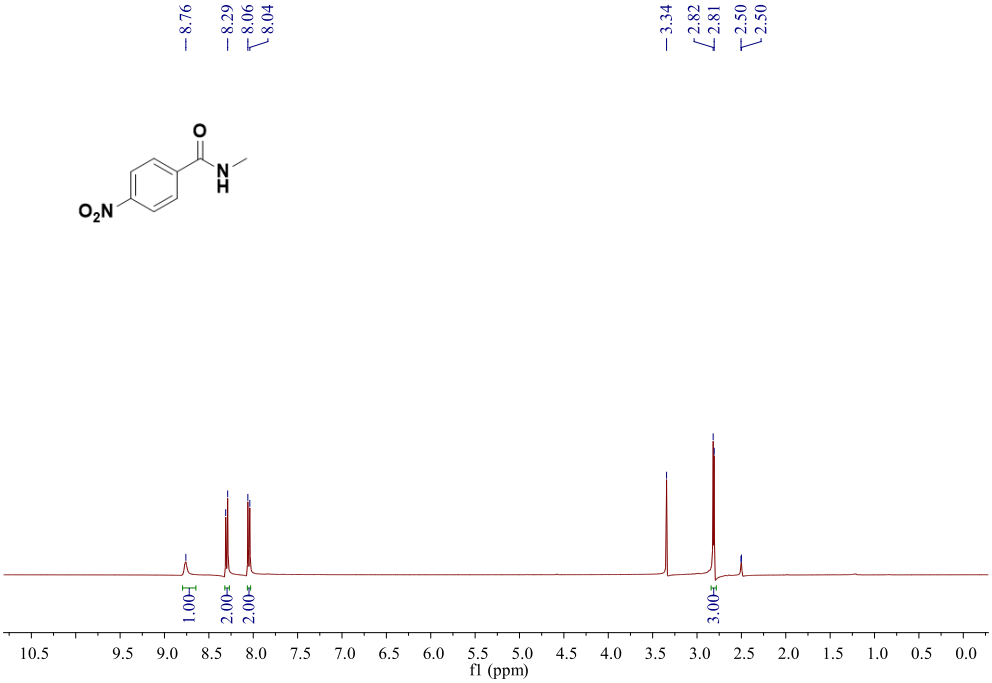


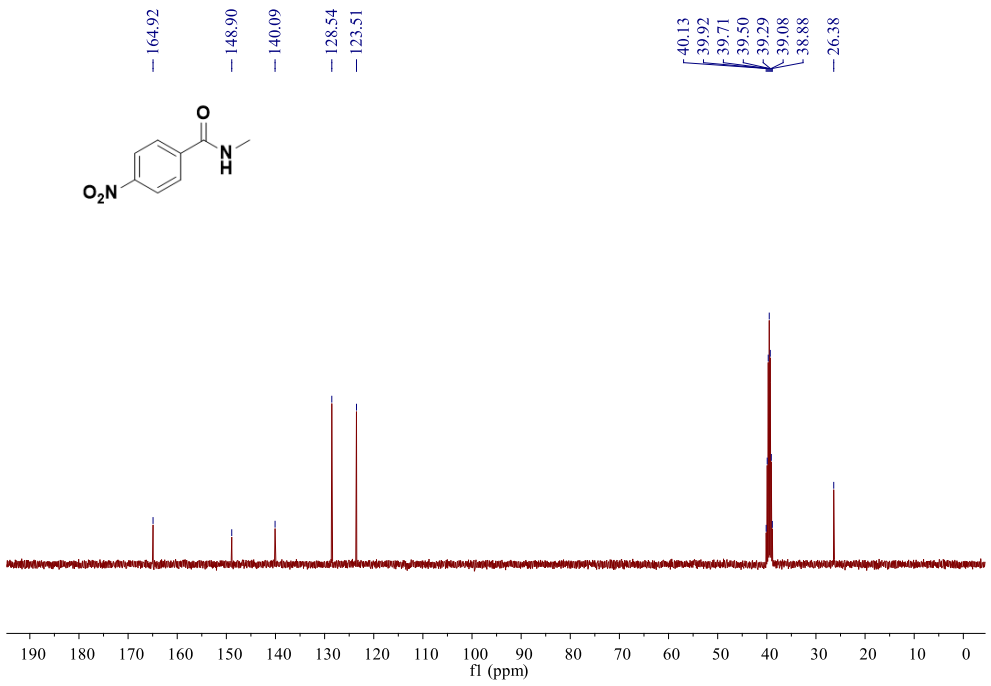


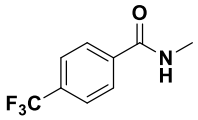


***N*-Methyl-4-nitrobenzamide (3e)**

petroleum ether / ethyl acetate = 1:1, yellow solid, 79% yield (28.4 mg). mp: 218 – 220℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.76 (s, 1H), 8.30 (d, *J* = 8.8 Hz, 2H), 8.05 (d, *J* = 8.8 Hz, 2H), 2.81 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 164.92, 148.90, 140.09, 128.54, 123.51, 26.38. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8N_2O_3$+Na^+: 203.0427, Found: 203.0425. **IR** (neat, cm^{-1}): υ 3330, 2946, 1645, 1597, 1489, 1347, 824, 780.

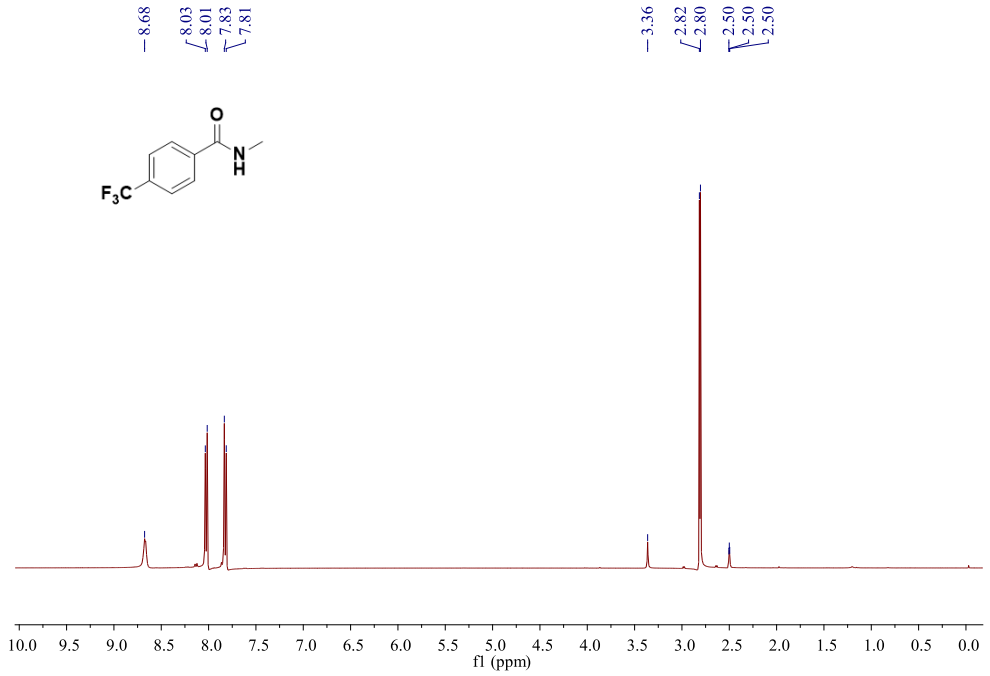


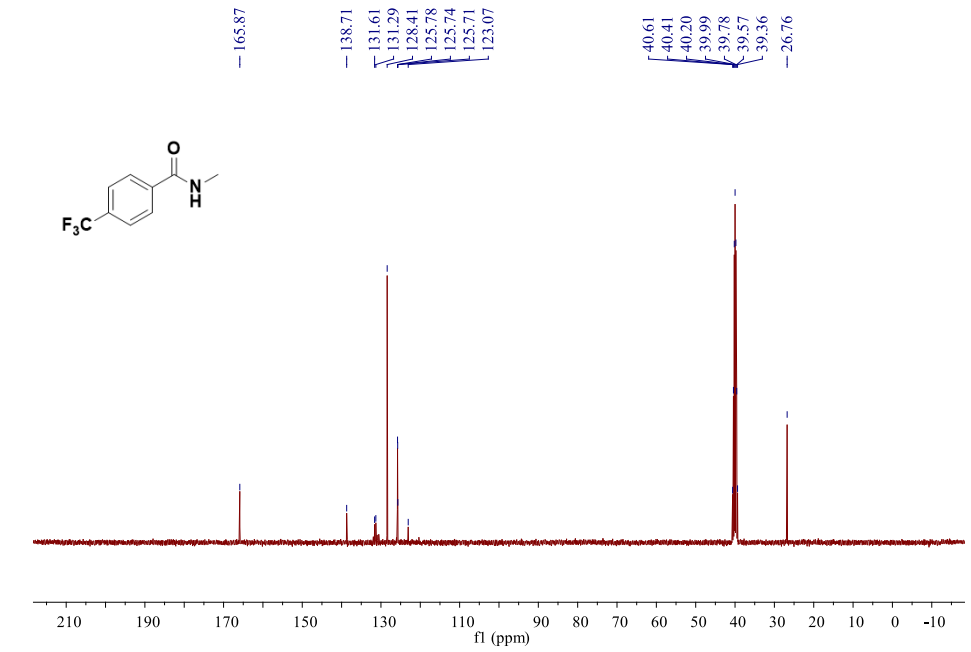


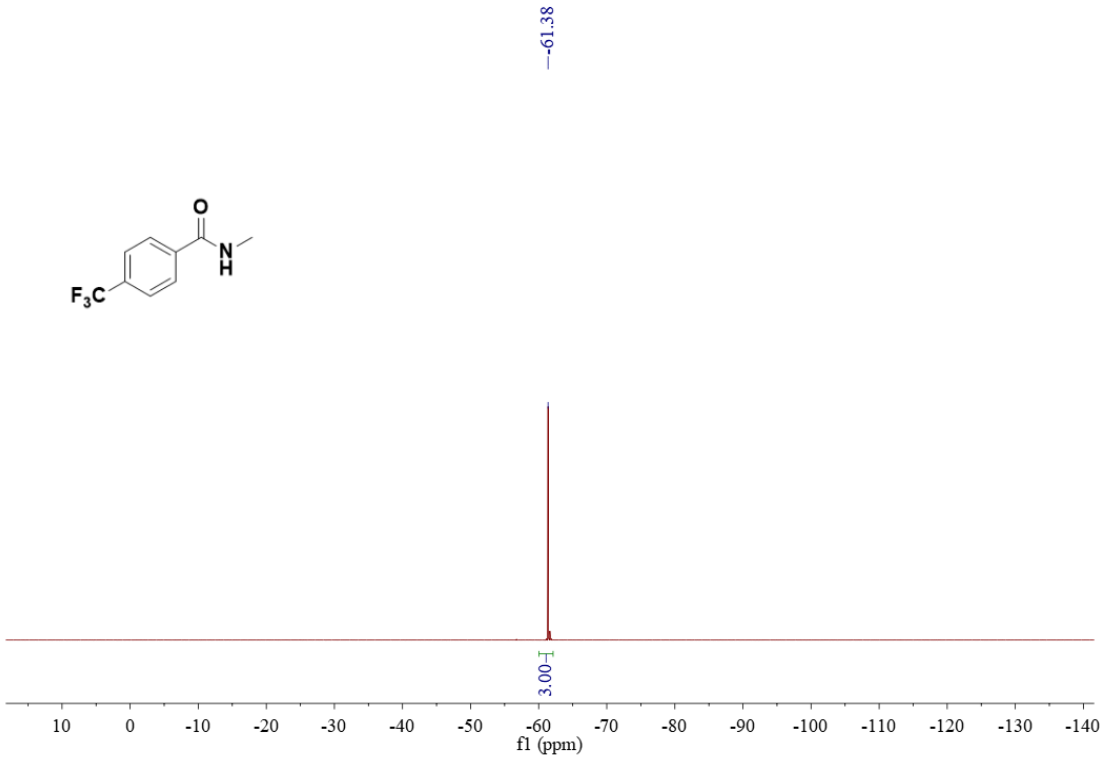


***N*-Methyl-4-(trifluoromethyl)benzamide (3f)**

petroleum ether / ethyl acetate = 2:1, white solid, 73% yield (29.7 mg). mp: 157-159℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.68 (s, 1H), 8.02 (d, *J* = 8.2 Hz, 2H), 7.82 (d, *J* = 8.2 Hz, 2H), 2.81 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.87, 138.71, 131.45 (q, *J* = 31.7 Hz), 128.41, 125.76 (q, *J* = 3.8 Hz), 123.07, 26.76. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.38 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8F_3NO$ $+H^+$: 204.0631, Found: 204.0627. **IR** (neat, cm^{-1}): υ 3334, 3082, 1635, 1556, 1495, 1360, 846, 776.



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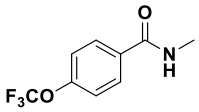
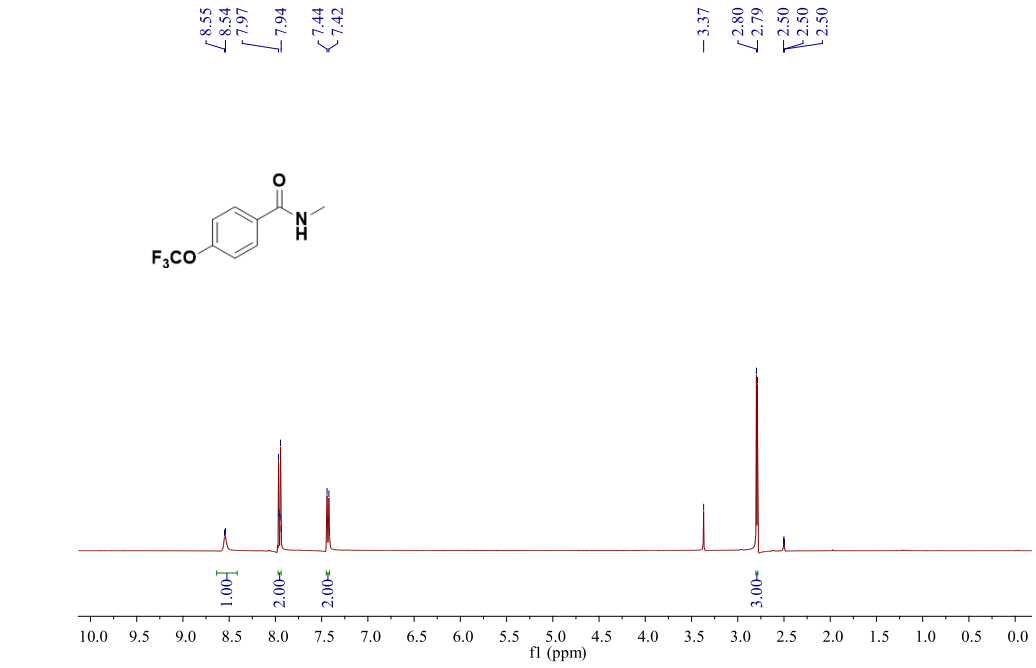
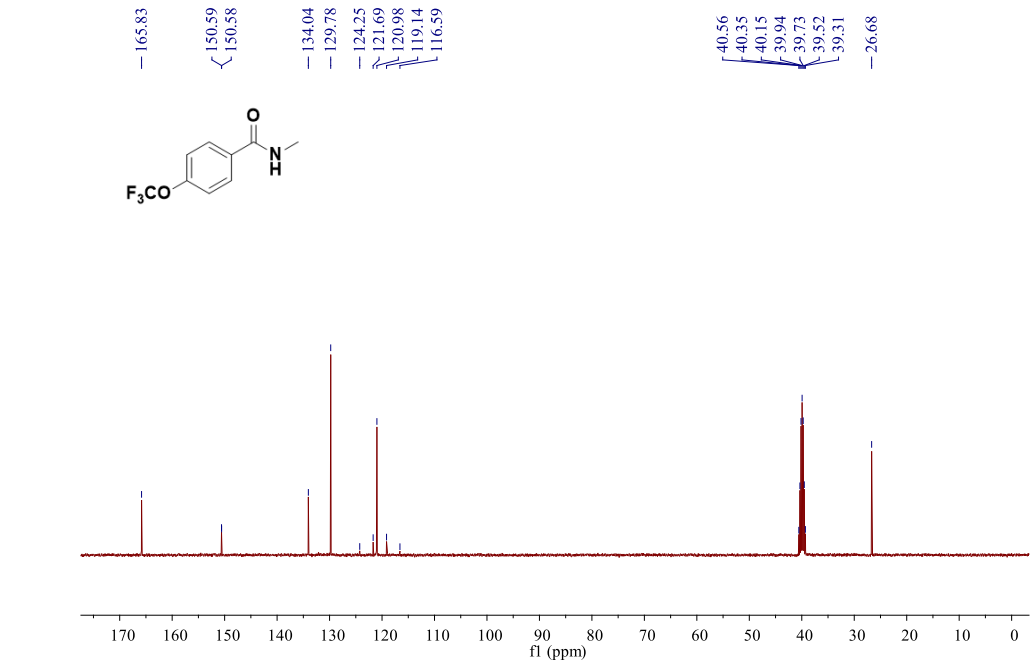
f1 (ppm)

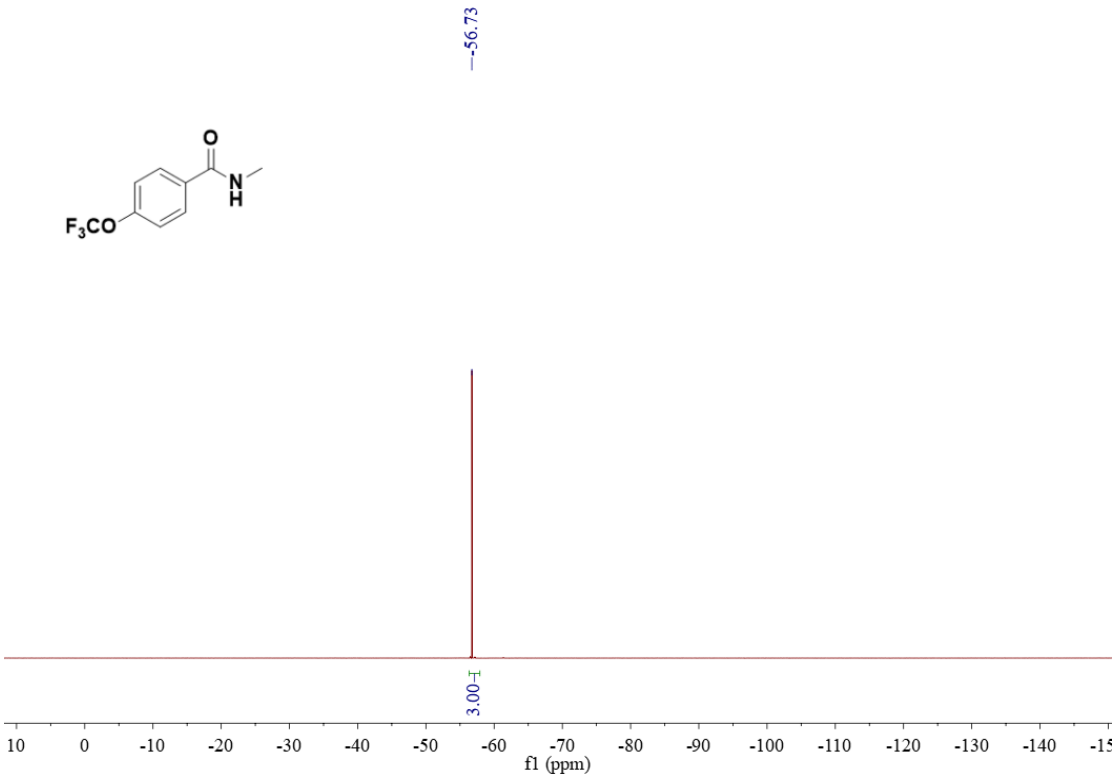


***N*-Methyl-4-(trifluoromethoxy)benzamide (3g)**

petroleum ether / ethyl acetate = 2:1, white solid, 66% yield (28.9 mg). mp: 99 – 101℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.55 (d, *J* = 4.1 Hz, 1H), 7.97 – 7.94 (m, 2H), 7.44 – 7.42 (m, 2H), 2.79 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.83, 150.59, 134.04, 129.78, 120.98, 120.42 (q, *J* = 255 Hz), 26.68. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.73 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8F_3NO_2$ +Na^+: 242.0399, Found: 242.0385. **IR** (neat, cm^{-1}): υ 3326, 3050, 1636, 1586, 1410, 1352, 1207, 840, 762.



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O
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H
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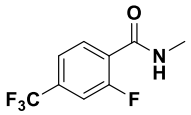
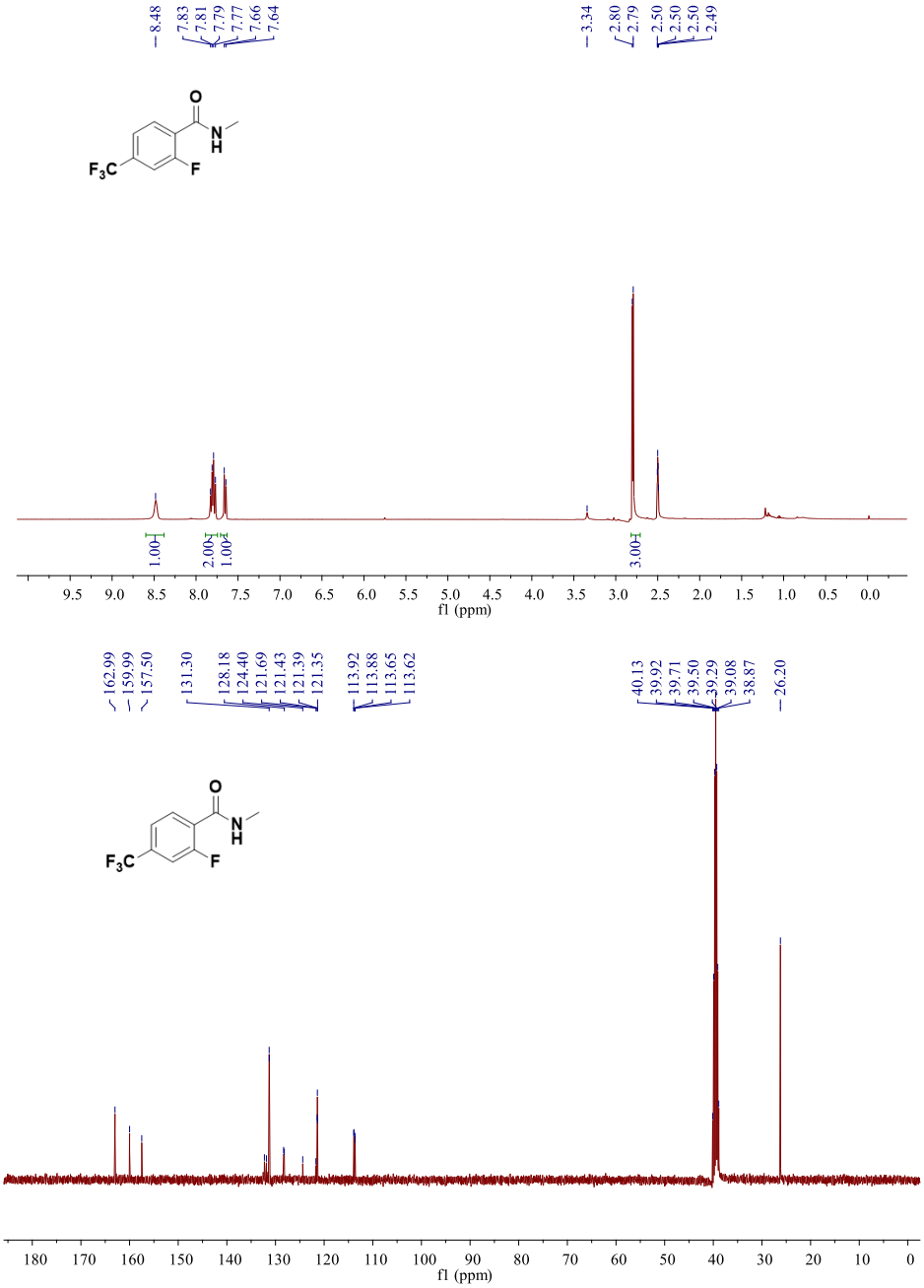
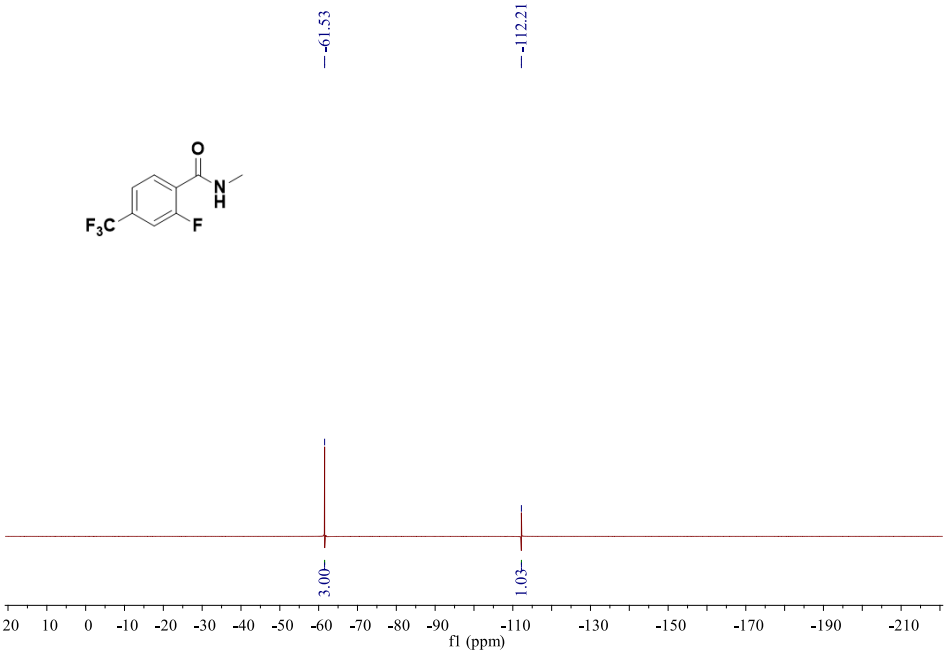
f1 (ppm)

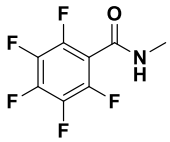


2-Fluoro-*N*-Methyl-4-(trifluoromethyl)benzamide (3h)

petroleum ether / ethyl acetate = 2:1, white solid, 50% yield (22.1 mg). mp: 90 – 91℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.48 (s, 1H), 7.83 –7.77 (m, 2H), 7.66 –7.64 (m, 1H), 2.80 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 162.99, 158.74 (d, *J* = 251.2 Hz), 132.07 (dd, *J* = 41.5, 9.0 Hz), 131.28 (d, *J* = 3.6 Hz), 128.26 (d, *J* = 15.4 Hz), 123.04 (dd, *J* = 272.8, 3.0 Hz), 113.77 (dd, *J* = 26.3, 3.8 Hz), 26.20. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -61.53 (s, 3F), -112.21 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_7F_4NO$ $+Na^+$: 244.0356, Found: 244.0363. **IR** (neat, cm^{-1}): υ 3262, 2932, 1635, 1523, 1412, 1331, 879, 778.

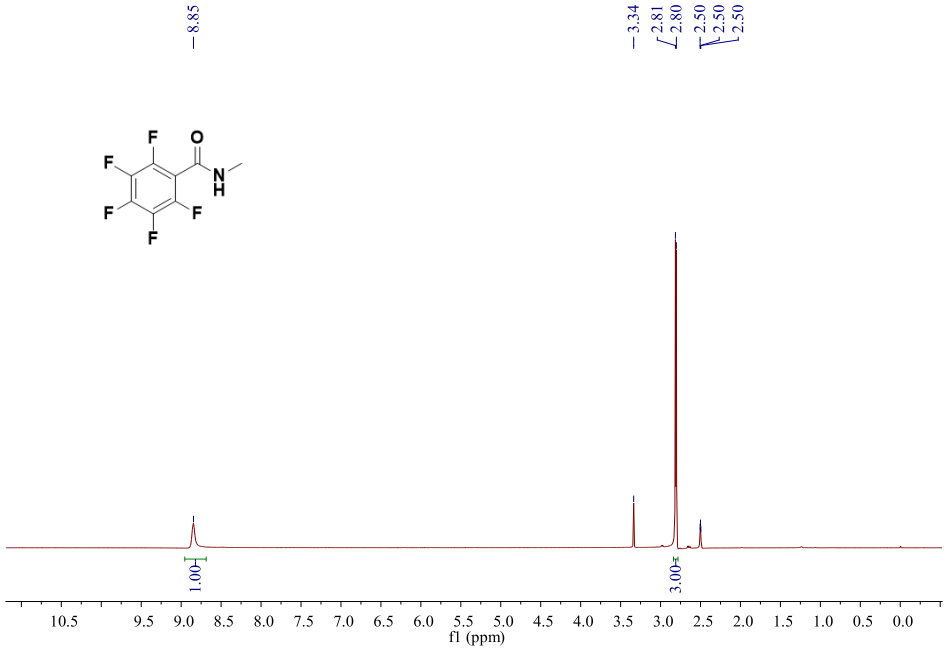


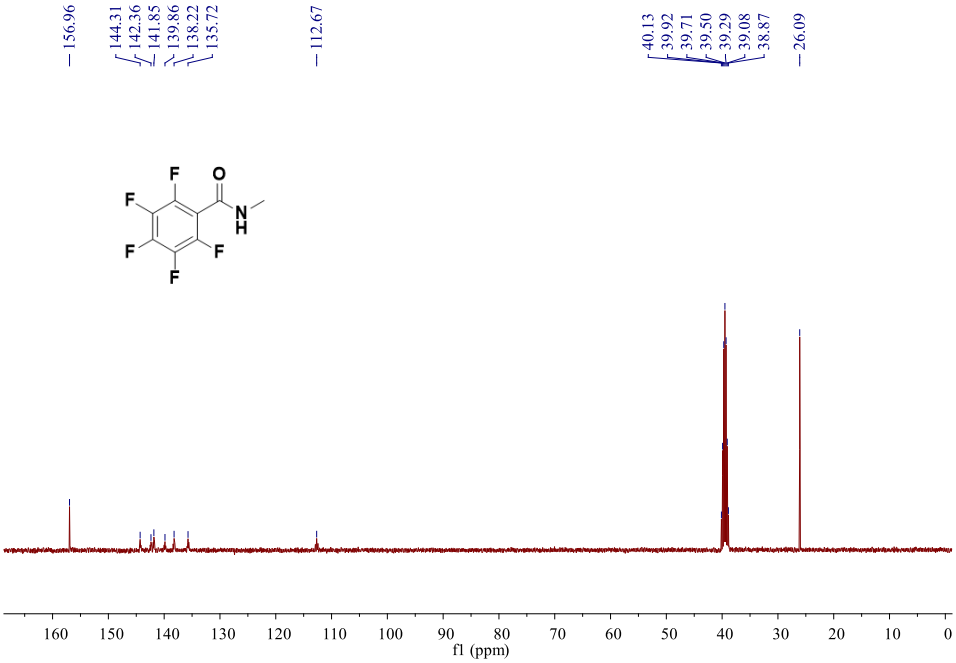

-61.53
-112.21
O
N
H
F3C
F
3.00
1.03
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

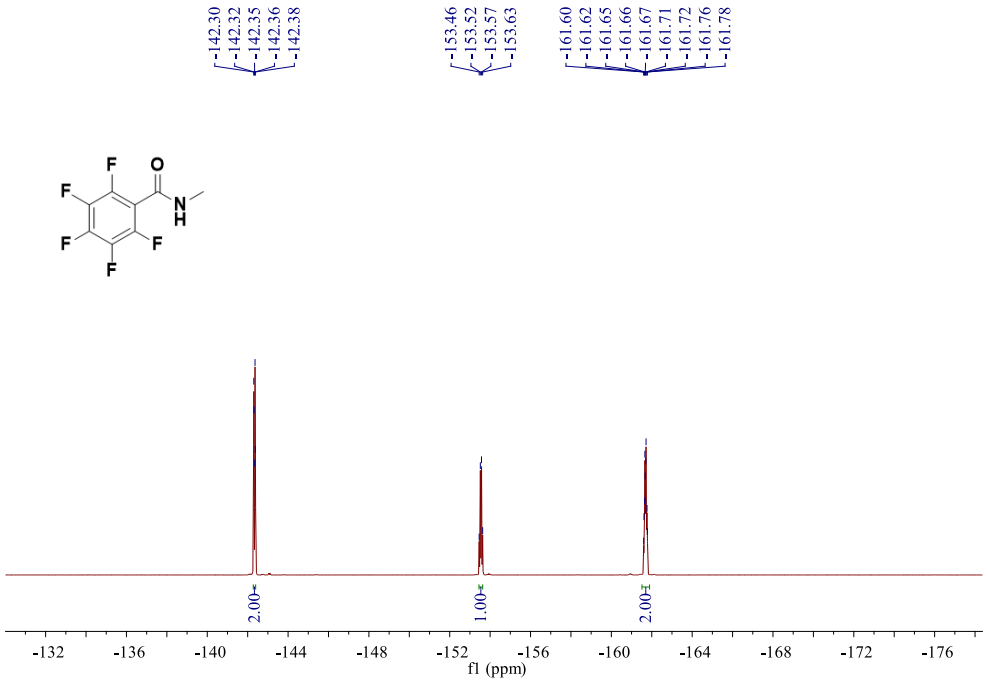


2,3,4,5,6-pentafluoro-*N*-methylbenzamide (3i)

petroleum ether / ethyl acetate = 2:1, white solid, 44% yield (19.8 mg). mp: 98 – 100℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.85 (s, 1H), 2.81 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 156.96, 143.08 (m), 141.11 (m), 136.97 (m), 112.67 (m), 26.09. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -141.30 – -143.38 (m, 2F), -153.54 (q, *J* = 21.8 Hz, 1F), -160.60 – -162.78 (m, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_4F_5NO+Na^+$: 248.0105, Found: 248.0106. **IR** (neat, cm^{-1}): υ 3269, 1687, 1651, 1570, 1496, 1322, 1032, 988.






-142.30
-142.32
-142.35
-142.36
-142.38
-153.46
-153.52
-153.57
-153.63
-161.60
-161.62
-161.65
-161.66
-161.67
-161.71
-161.72
-161.76
-161.78
F
O
N
H
F
F
F
F
2.00
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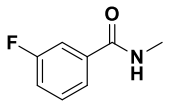
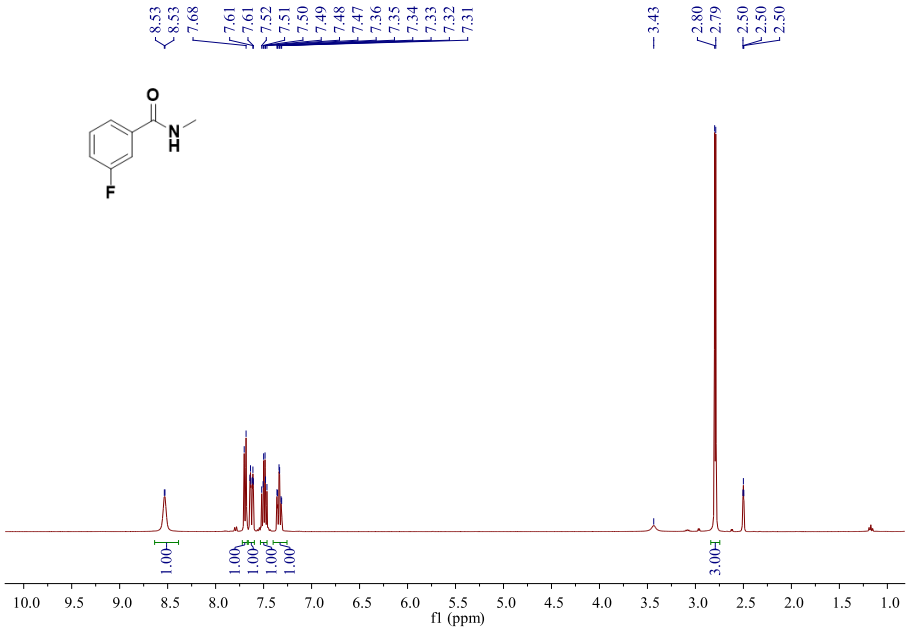
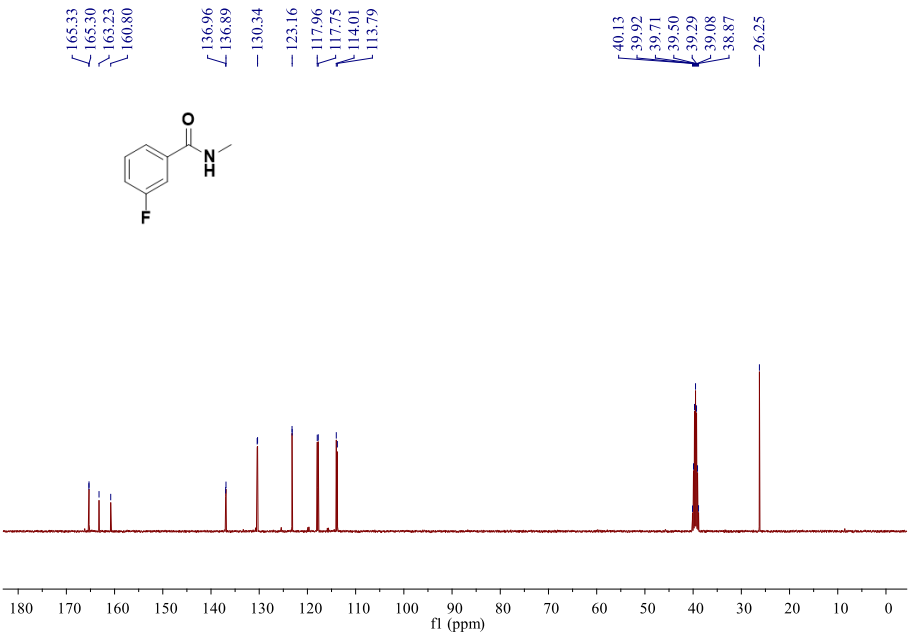
-168
-172
-176
f1 (ppm)

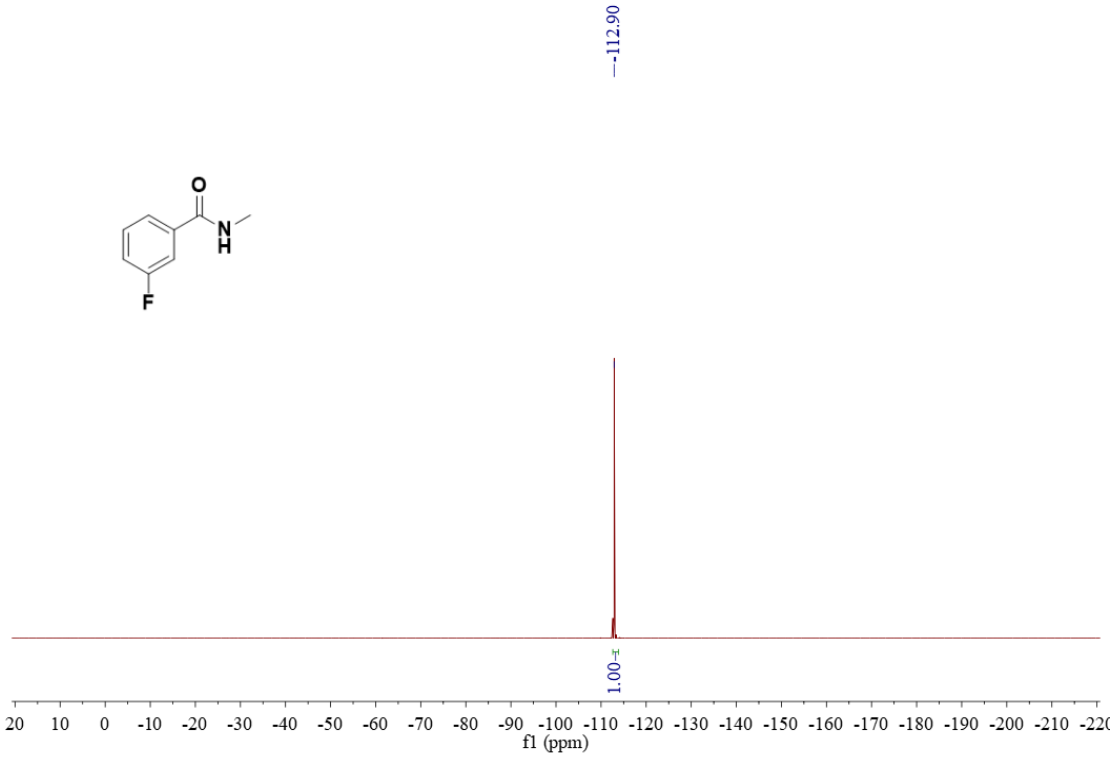


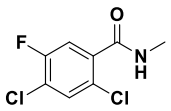
3-Fluoro-*N*-methylbenzamide (3j)

petroleum ether / ethyl acetate = 2:1, yellow solid, 74% yield (22.6 mg). mp: 87 – 88℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.53 (d, *J* = 2.6 Hz, 1H), 7.70 – 7.68 (m, 1H), 7.64 – 7.61 (m, 1H), 7.52 – 7.47 (m, 1H), 7.36 – 7.31 (m, 1H), 2.79 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.31 (d, *J* = 2.5 Hz), 162.01 (d, *J* = 244.1 Hz), 136.93 (d, *J* = 6.8 Hz), 130.38 (d, *J* = 8.0 Hz), 123.18 (d, *J* = 2.8 Hz), 117.86 (d, *J* = 21.1 Hz), 113.90 (d, *J* = 22.7 Hz), 26.25. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -112.90 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_8FNO+Na^+$: 176.0482, Found: 176.0478. **IR** (neat, cm^{-1}): υ 3335, 2947, 1634, 1552, 1488, 1301, 892, 802, 792.



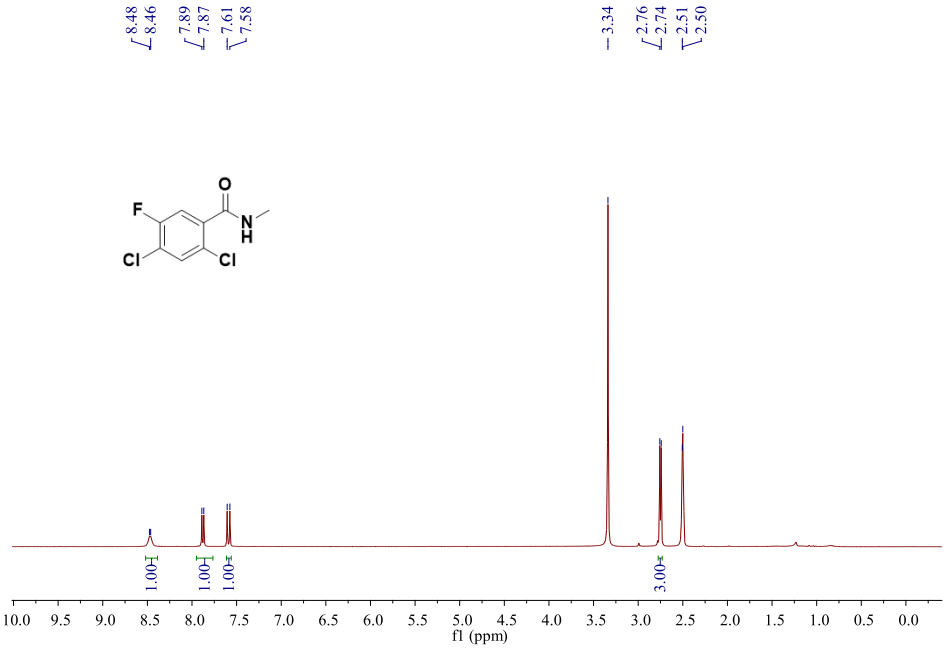


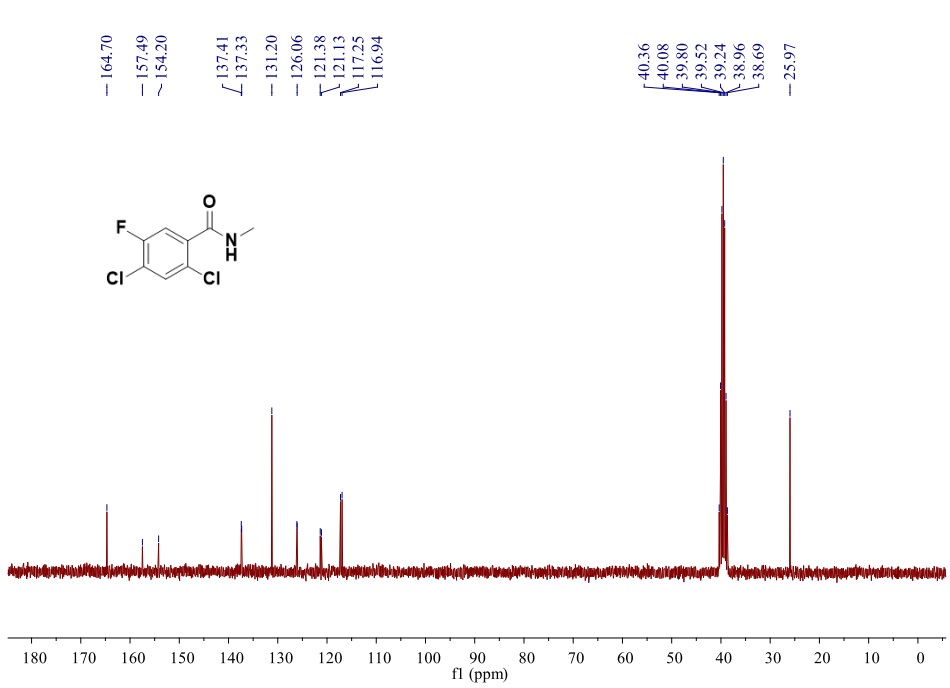

-112.90
O
N
H
F
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22(
f1 (ppm)

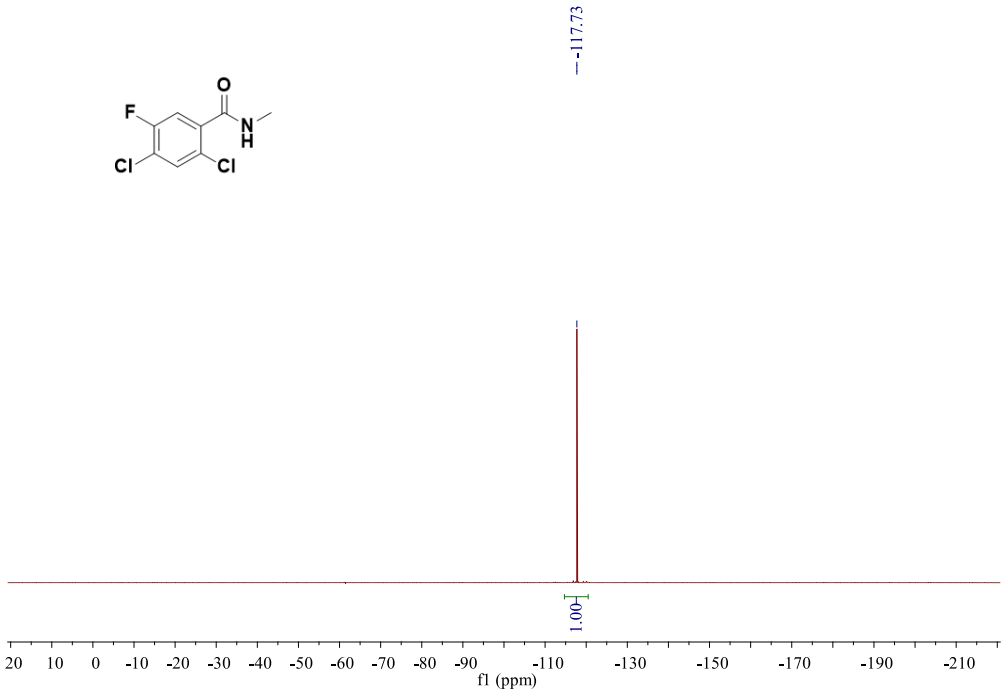


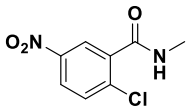
2,4,6-trichloro-*N*-methylbenzamide (3k)

petroleum ether / ethyl acetate = 2:1, white solid, 61% yield (27.0 mg). mp: 130 – 132℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 8.47 (d, *J* = 4.2 Hz, 1H), 7.88 (d, *J* = 6.6 Hz, 1H), 7.59 (d, *J* = 9.1 Hz, 1H), 2.75 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 164.70, 155.84 (d, *J* = 248.3 Hz), 137.37 (d, *J* = 6.3 Hz), 131.20, 126.09 (d, *J* = 3.7 Hz), 121.25 (d, *J* = 18.8 Hz), 117.10 (d, *J* = 23.8 Hz), 25.97. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -117.73 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6{}^{35}Cl_2FNO$ +Na^+: 243.9703, Found: 243.9700. Anal Calcd. For. $C_8H_6{}^{35,37}Cl_2FNO$ +Na^+: 245.9673, Found: 245.9671. **IR** (neat, cm^{-1}): υ 3295, 2854, 1752, 1645, 1553, 1469, 1309, 950, 886.



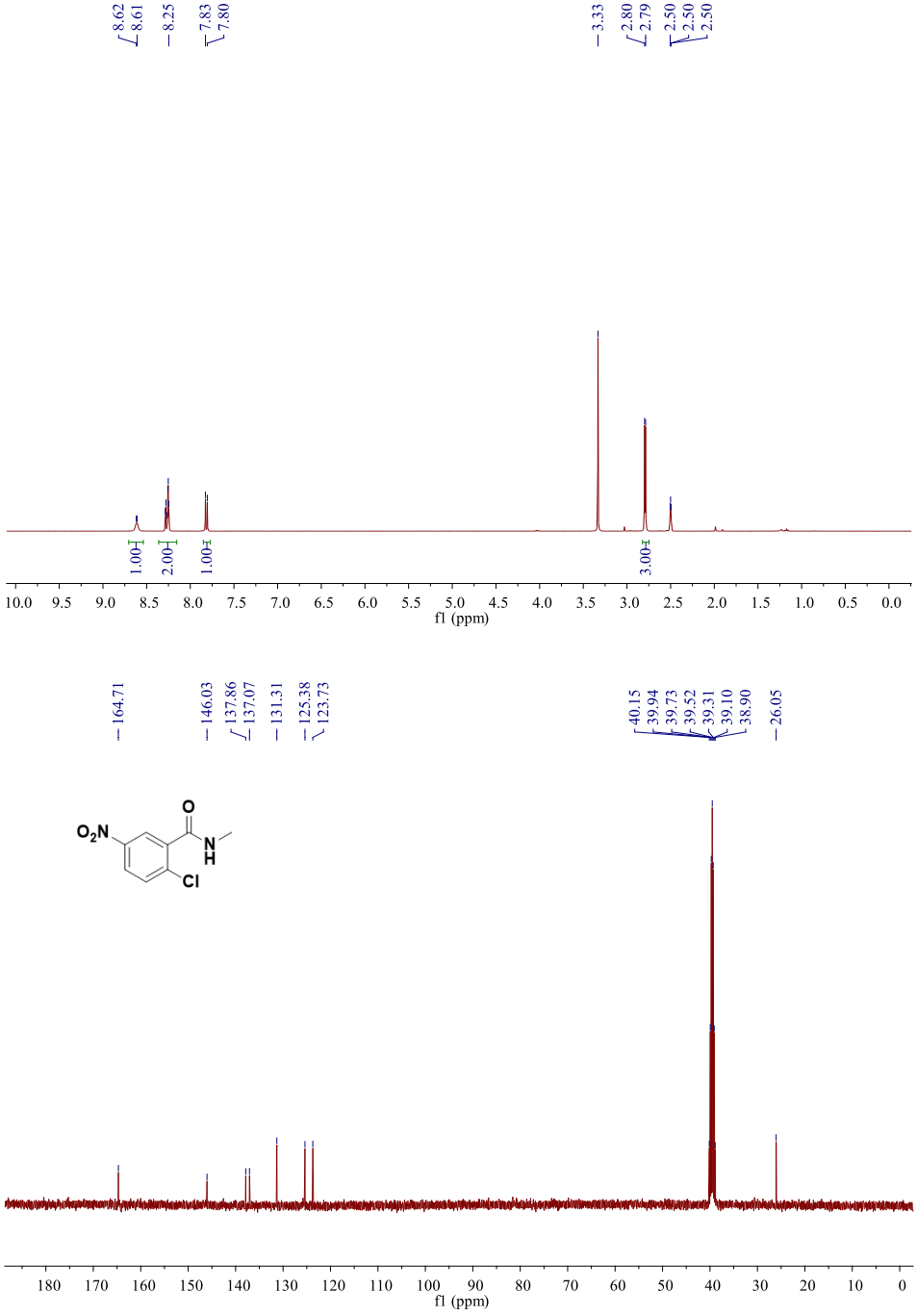


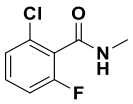

-117.73
F
O
N
H
Cl
Cl
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)



2-Chloro-*N*-methyl-5-nitrobenzamide (3l)

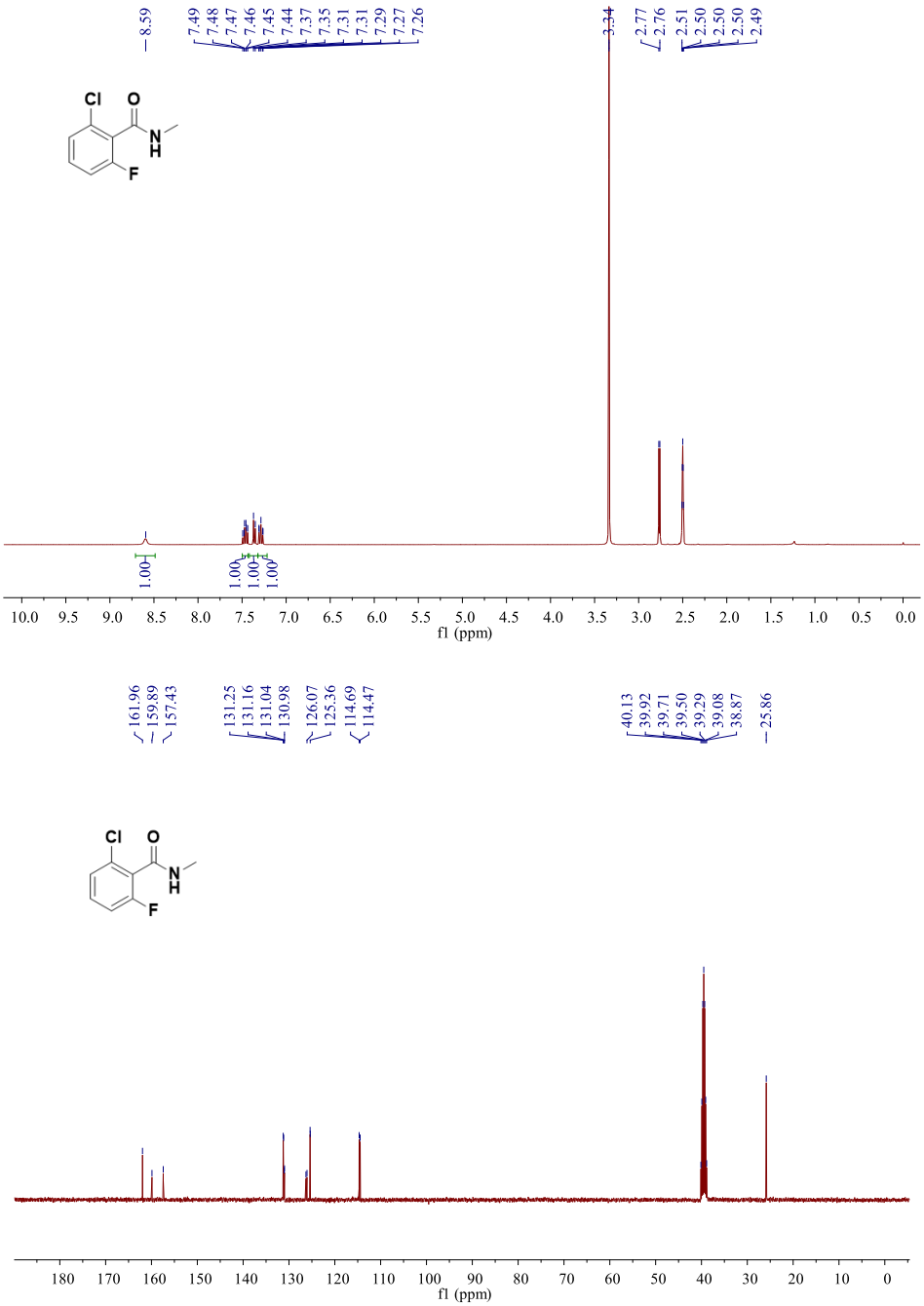
petroleum ether / ethyl acetate = 1:1, white solid, 52% yield (22.3 mg). mp: 171 – 172℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.61 (d, *J* = 3.7 Hz, 1H), 8.29 – 8.25 (m, 2H), 7.83 – 7.80 (m, 1H), 2.79 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 164.71, 146.03, 137.86, 137.07, 131.31, 125.38, 123.73, 26.05. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_7{}^{35}ClN_2O_3$ +Na^+: 237.0037, Found: 237.0038. Anal Calcd. For. $C_8H_7{}^{37}ClN_2O_3$ +Na^+: 239.0008, Found: 239.0030. **IR** (neat, cm^{-1}): υ 3276, 1656, 1557, 1464, 1352, 910, 844, 787.

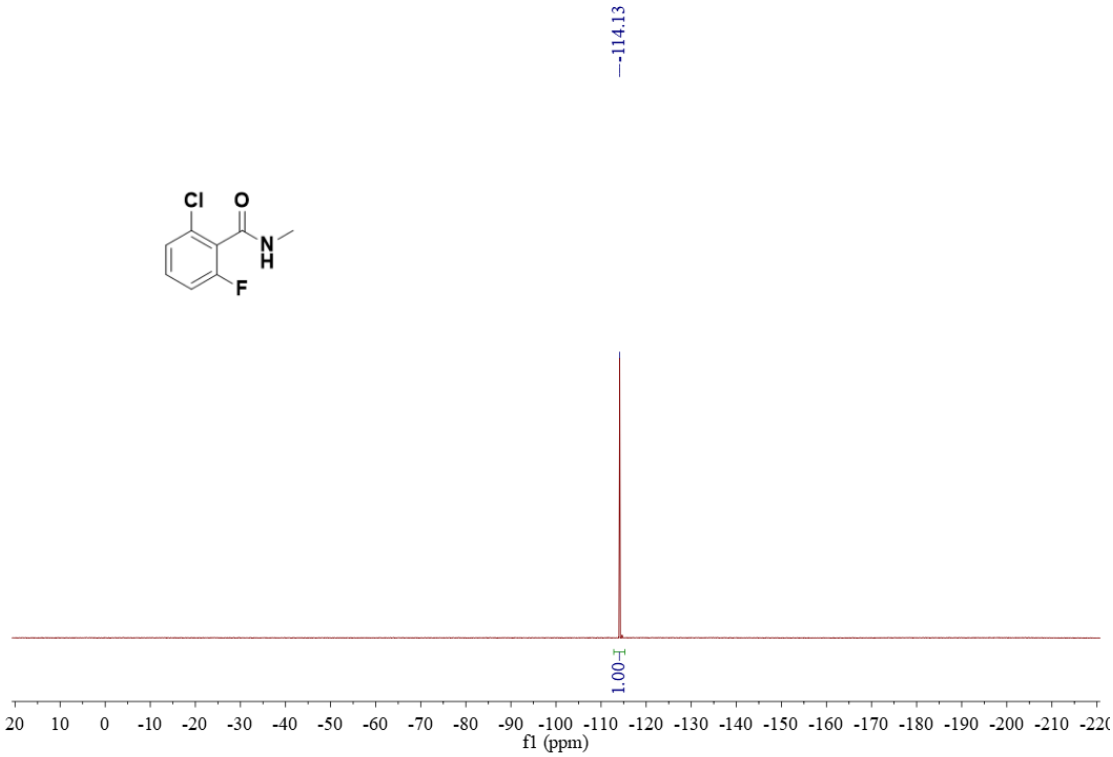




2-Chloro-6-fluoro-*N*-methylbenzamide (3m)

petroleum ether / ethyl acetate = 2:1, white solid, 48% yield (18.0 mg). mp: 135 – 137℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.59 (s, 1H), 7.49 – 7.44 (m, 1H), 7.37 – 7.35 (m, 1H), 7.31 – 7.26 (m, 1H), 2.77 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ161.96, 158.66 (d, *J* = 247.7 Hz), 131.21 (d, *J* = 9.2 Hz), 131.01 (d, *J* = 6.3 Hz), 126.18 (d, *J* = 23.3 Hz), 125.38 (d, *J* = 3.3 Hz), 114.58 (d, *J* = 21.9 Hz), 25.86. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -114.13 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_7{}^{35}ClFNO$ +Na^+: 210.0092, Found: 210.0096. Anal Calcd. For. $C_8H_7{}^{37}ClFNO$ +Na^+: 212.0063, Found: 212.0055. **IR** (neat, cm^{-1}): υ 3279, 2944, 1645, 1547, 1488, 1310, 816, 766.




-114.13
Cl
O
N
H
F
1.00
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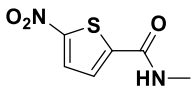
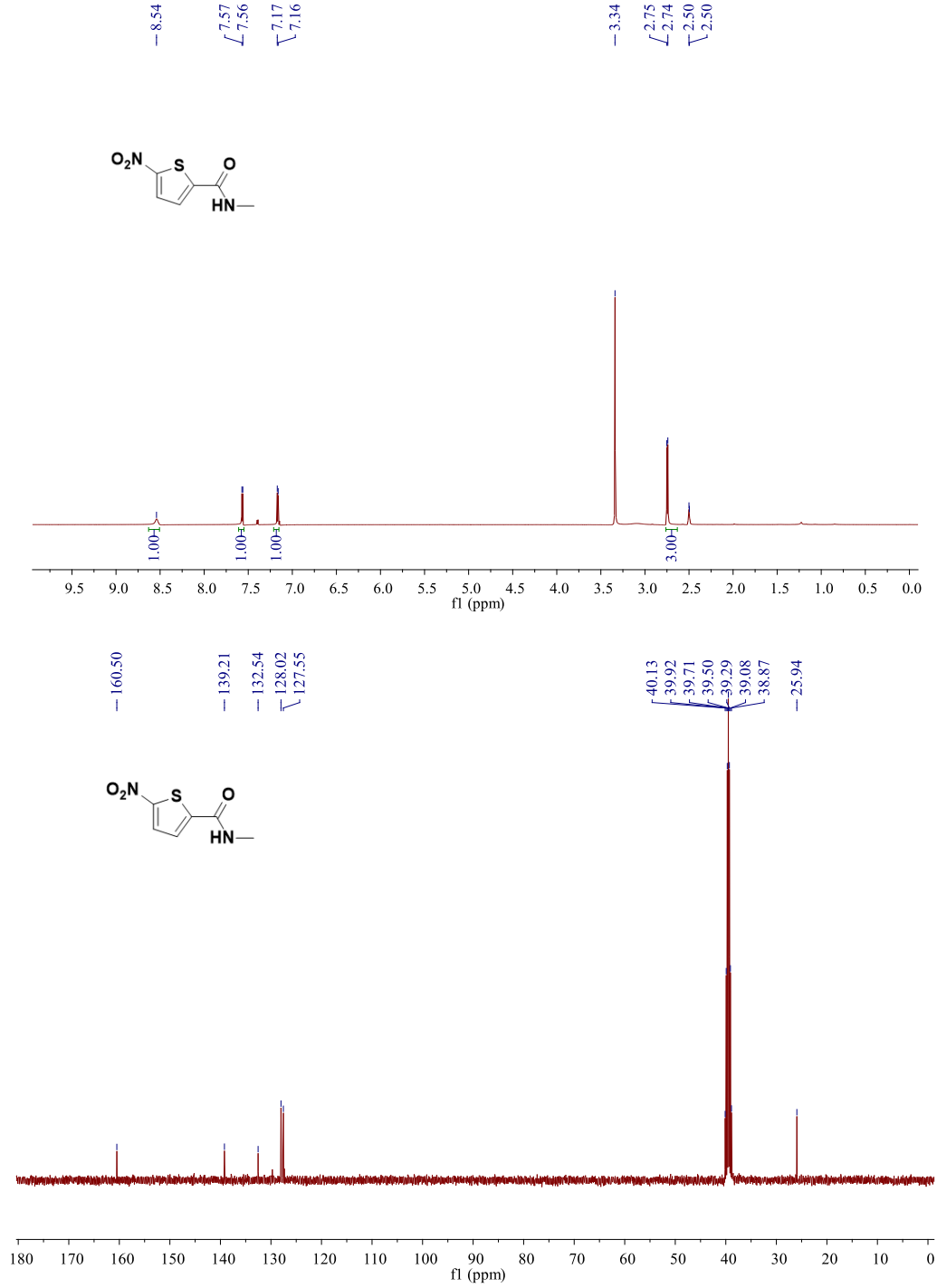
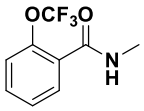
f1 (ppm)



***N*-Methyl-5-nitrothiophene-2-carboxamide (3n)**

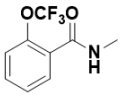
petroleum ether / ethyl acetate = 2:1, yellow solid, 41% yield (15.3 mg). mp: 214 – 215℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.54 (s, 1H), 7.57 (d, *J* = 4.0 Hz, 1H), 7.17 (d, *J* = 4.0 Hz, 1H), 2.75 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 160.50, 139.21, 132.54, 128.02, 127.55, 25.94. **HRMS** (ESI-TOF): Anal Calcd. For. $C_6H_6N_2O_3S$ +Na^+: 208.9991, Found: 208.9987. **IR** (neat, cm^{-1}): υ 3335, 3083, 1734, 1632, 1558, 1495, 1304, 831, 744.

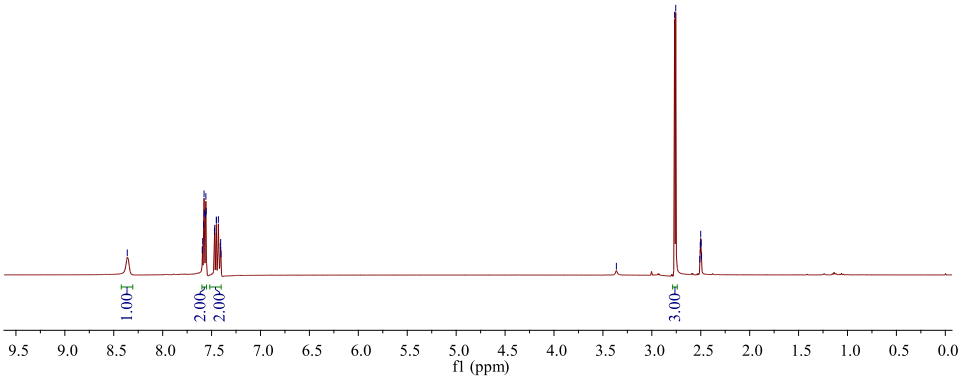


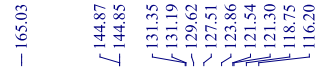


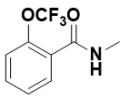
***N*-Methyl-2-(trifluoromethoxy)benzamide (3o)**

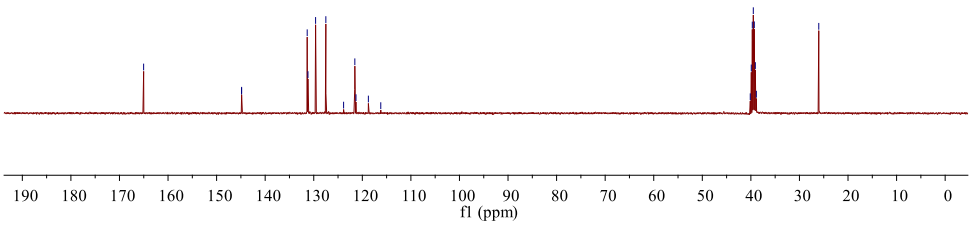
petroleum ether / ethyl acetate = 2:1, white solid, 53% yield (23.2 mg). mp: 63 – 65℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.36 (s, 1H), 7.60 – 7.56 (m, 2H), 7.47 – 7.40 (m, 2H), 2.76 (d, *J* = 4.6 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.03, 144.86 (q, *J* = 1.7 Hz), 131.35, 131.19, 129.62, 127.51, 121.54, 120.03 (q, *J* = 256.8 Hz), 26.02. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -56.51 (s, 3F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_8F_3NO_2$ +Na^+: 242.0399, Found: 242.0401. **IR** (neat, cm^{-1}): υ 3282, 2950, 1646, 1593, 1444, 1321, 1250, 765, 698.

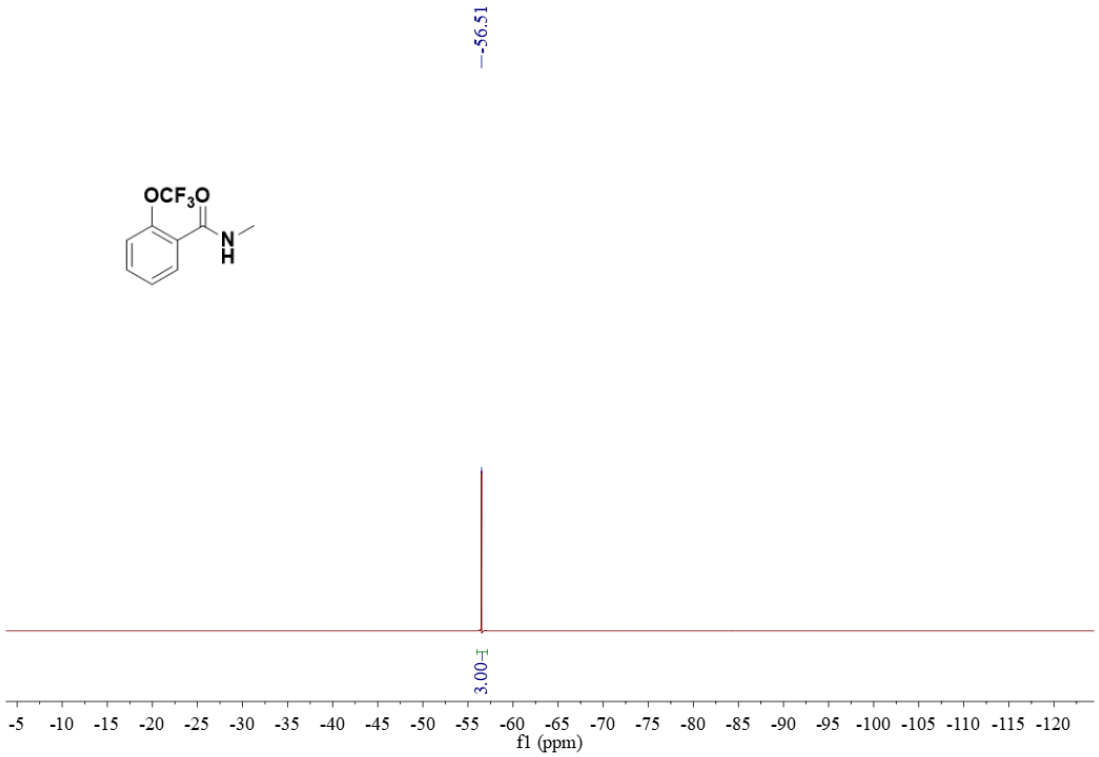


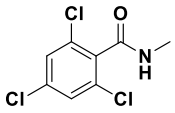






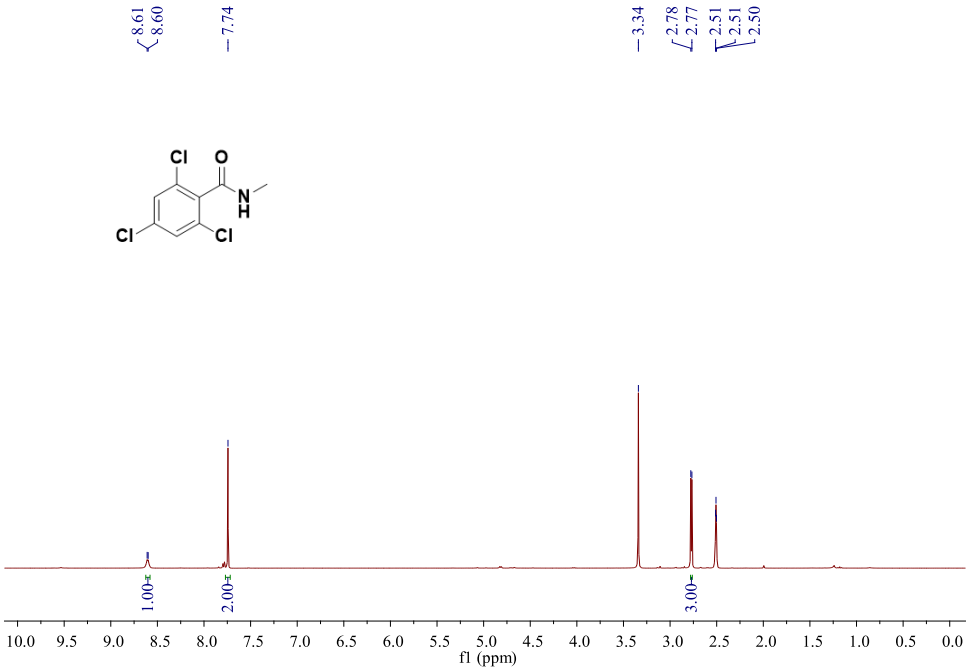


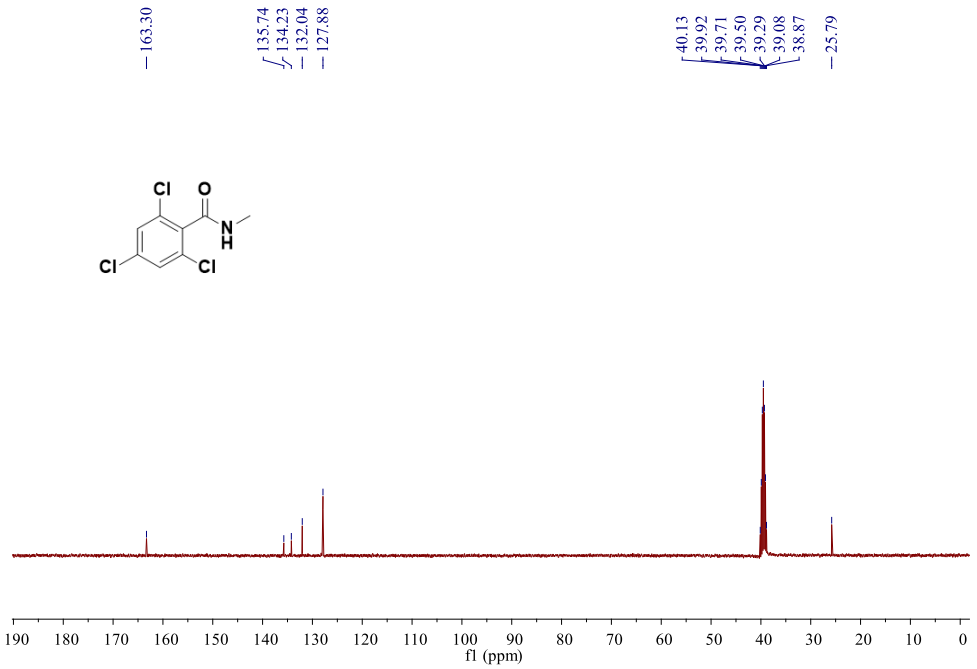

-56.51
OCF3O
N
H
3.00
-5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120
f1 (ppm)

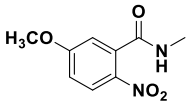


2,4,6-trichloro-*N*-methylbenzamide (3p)

petroleum ether / ethyl acetate = 2:1, white solid, 59% yield (28.0 mg). mp: 184 – 185℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.60 (d, *J* = 4.4 Hz, 1H), 7.74 (s, 2H), 2.77 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 163.30, 135.74, 134.23, 132.04, 127.88, 25.79. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_6{}^{35}Cl_3NO$ +Na^+: 259.09407, Found: 259.9410. Anal Calcd. For. $C_8H_6{}^{35,35,37}Cl_3NO$ +Na^+: 261.9378, Found: 261.9356. Anal Calcd. For. $C_8H_6{}^{35,37,37}Cl_3NO$ +Na^+: 263.9348, Found: 263.9298. **IR** (neat, cm^{-1}): υ 3280, 2943, 1646, 1546, 1488, 1309, 920, 849.

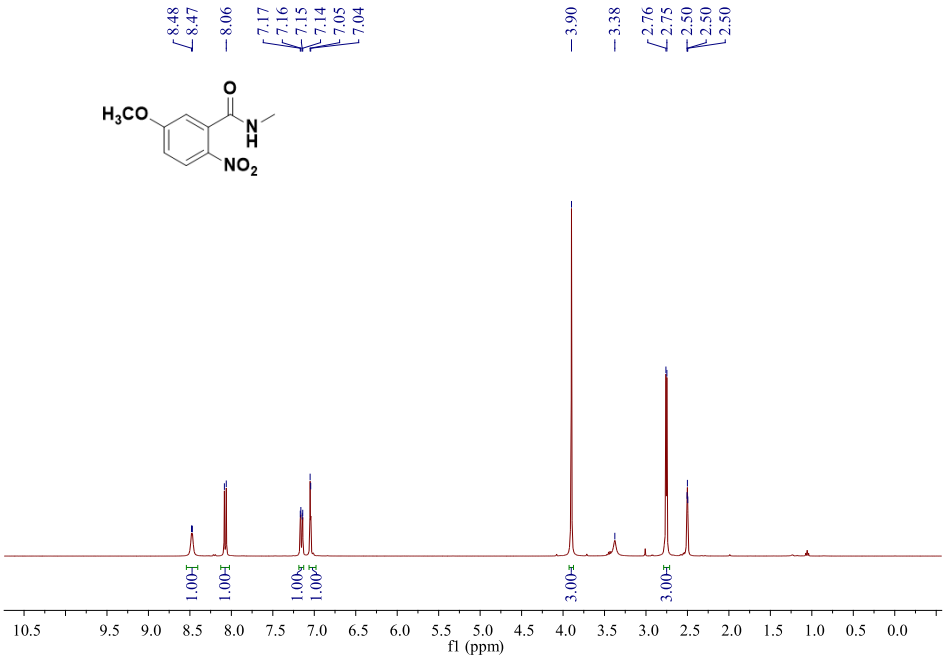


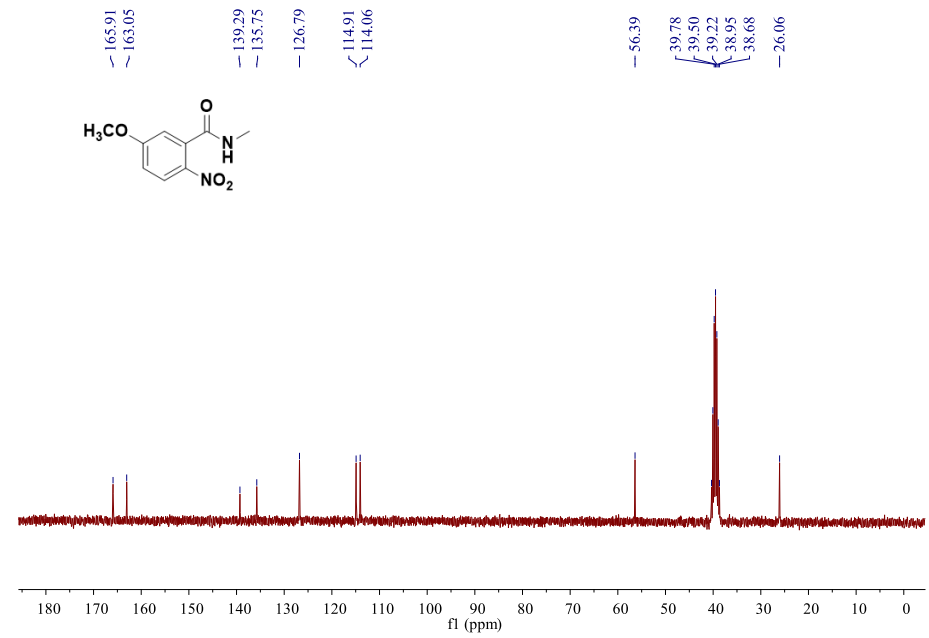


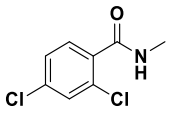


5-Methoxy-*N*-methyl-2-nitrobenzamide (3q)

petroleum ether / ethyl acetate = 1:1, yellow solid, 51% yield (21.4 mg). mp: 159 – 161℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.48 (d, *J* = 4.2 Hz, 1H), 8.07 (d, *J* = 9.1 Hz, 1H), 7.15 (dd, *J* = 9.1, 2.8 Hz, 1H), 7.05 (d, *J* = 2.8 Hz, 1H), 3.90 (s, 3H), 2.75 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 165.91, 163.05, 139.29, 135.75, 126.79, 114.91, 114.06, 56.39, 26.06. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_{10}N_2O_4$ +Na^+: 233.0533, Found: 233.0540. **IR** (neat, cm^{-1}): υ 3270, 2943, 1637, 1563, 1509, 1405, 1323, 888, 830, 796.

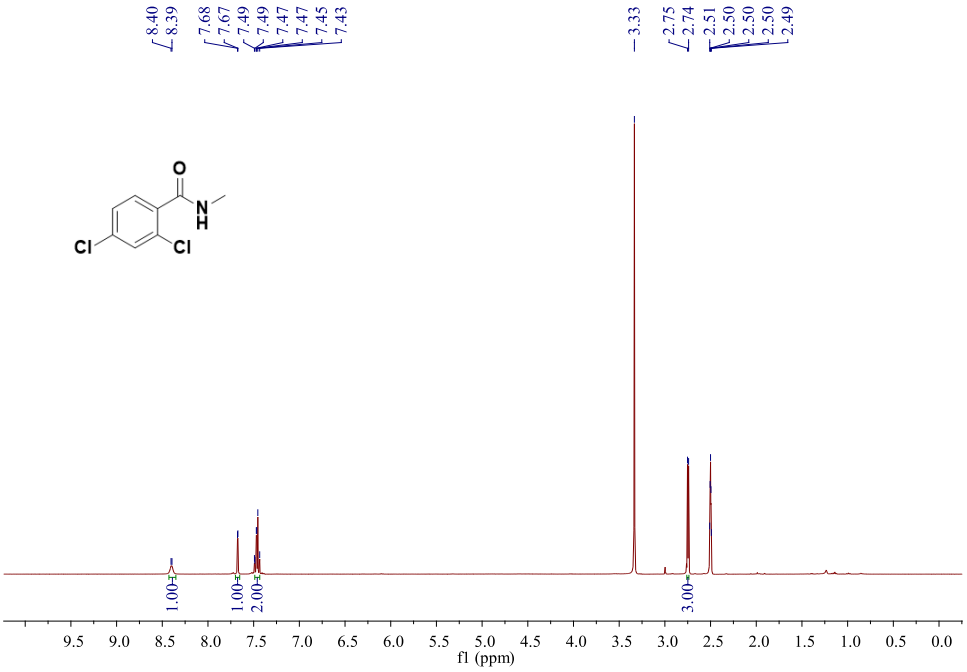


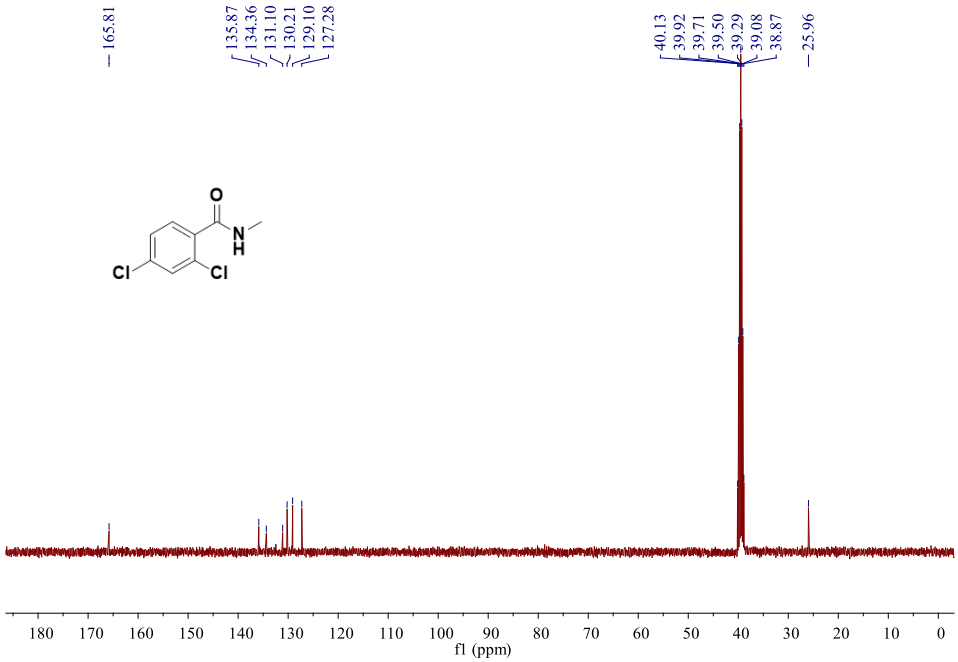


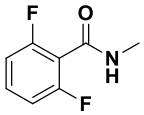


2,4-Dichloro-*N*-methylbenzamide (3r)

petroleum ether / ethyl acetate = 2:1, white solid, 52% yield (21.1 mg). mp: 125 – 127℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.40 (d, *J* = 4.1 Hz, 1H), 7.67 (d, *J* = 1.8 Hz, 1H), 7.49 – 7.43 (m, 2H), 2.75 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.81, 135.87, 134.36, 131.10, 130.21, 129.10, 127.28, 25.96. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_7{}^{35}Cl_2NO+Na^+$: 225.9797, Found: 225.9800. Anal Calcd. For. $C_8H_7{}^{37,35}Cl_2NO+Na^+$: 227.9767, Found: 227.9760. Anal Calcd. For. $C_8H_7{}^{35}Cl_2NO+Na^+$: 229.9738, Found: 229.9731. **IR** (neat, cm^{-1}): υ 3282, 2981, 1646, 1592, 1444, 1321, 881, 828, 788.

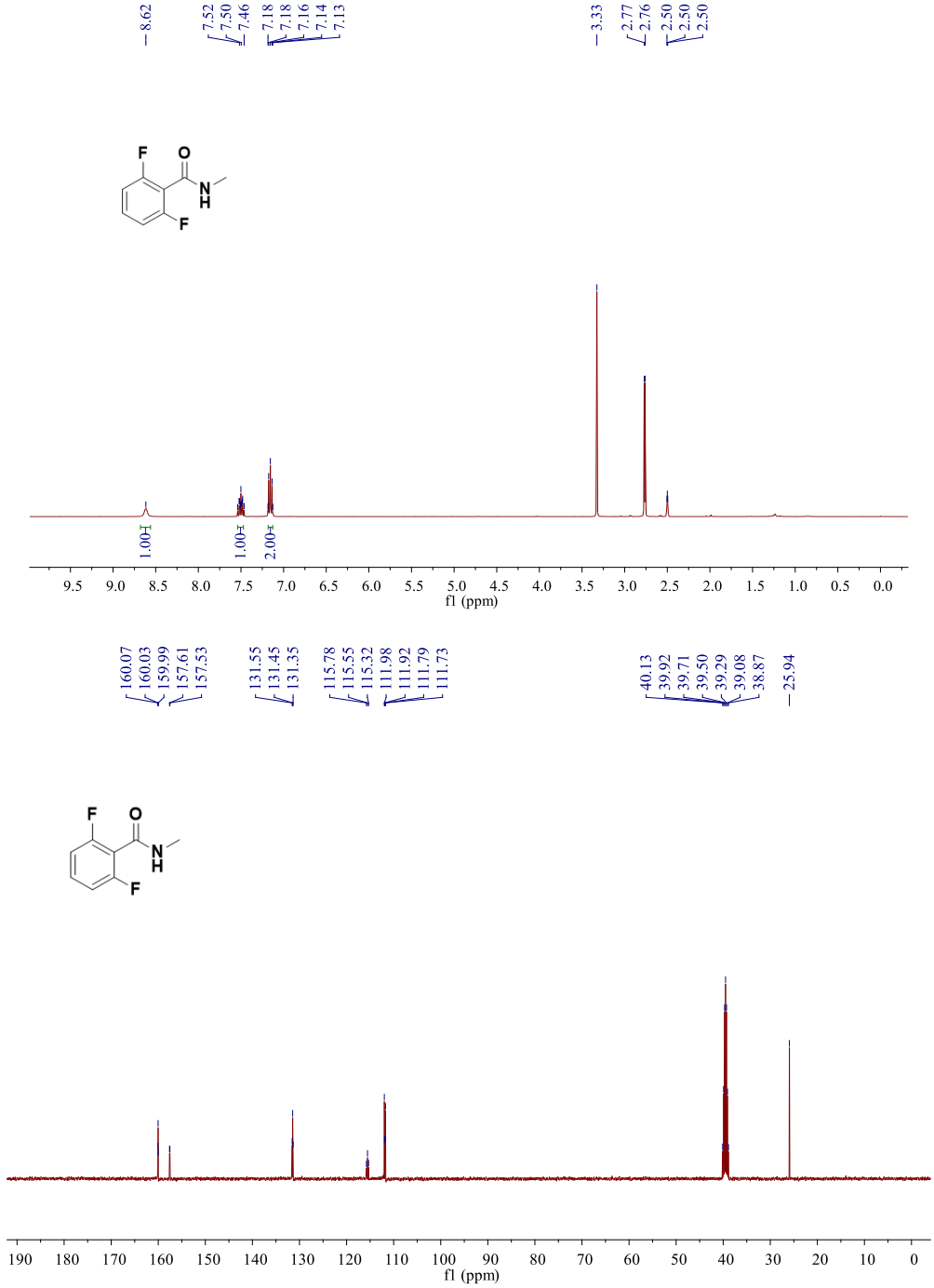


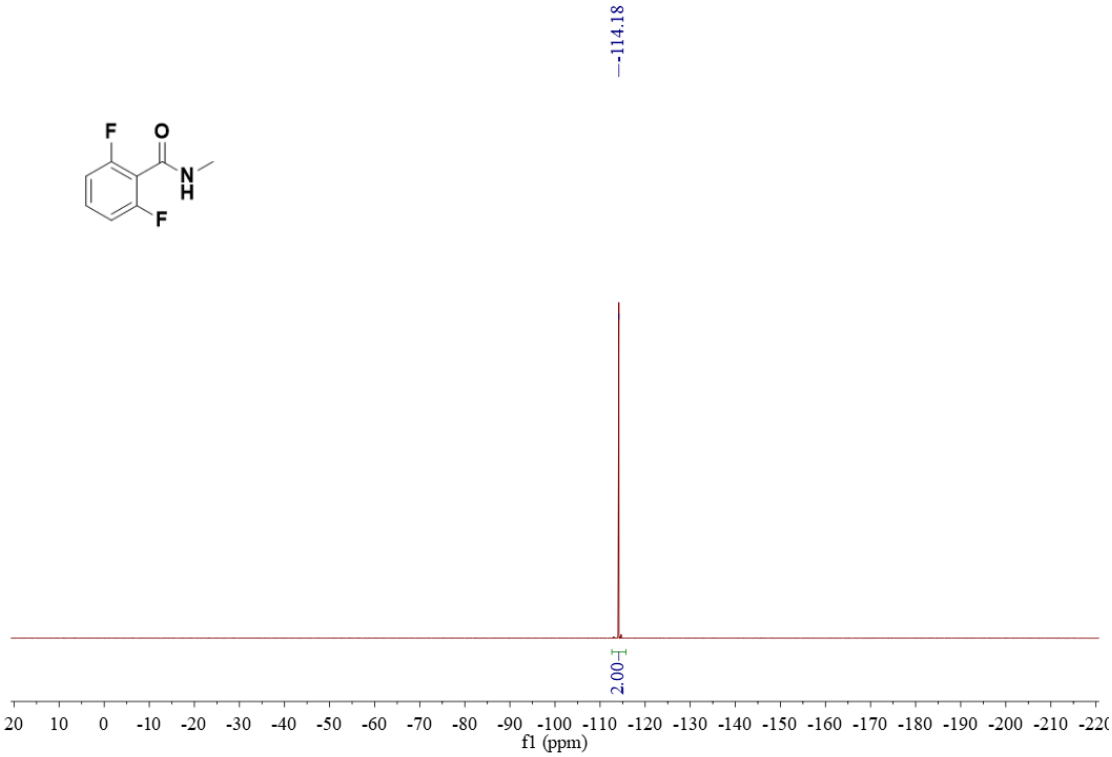


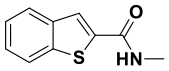


2,6-Difluoro-*N*-methylbenzamide (3s)

petroleum ether / ethyl acetate = 2:1, yellow solid, 49% yield (16.8 mg). mp: 123 – 125℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.62 (s, 1H), 7.52 – 7.46 (m, 1H), 7.18 – 7.13 (m, 2H), 2.77 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 160.03 (t, *J* = 4.1 Hz), 157.57 (d, *J* = 8.1 Hz), 131.45 (t, *J* = 10.0 Hz), 115.55 (t, *J* = 23.2 Hz), 111.86 (dd, *J* = 13.0, 6.0 Hz), 25.94. **^{19}F NMR** (377 MHz, DMSO-*d6*) δ -114.18 (s, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_7F_2NO$ +Na^+: 194.0388, Found: 194.0380. **IR** (neat, cm^{-1}): υ 3280, 2881, 1646, 1593, 1488, 1325, 1299, 845, 766.

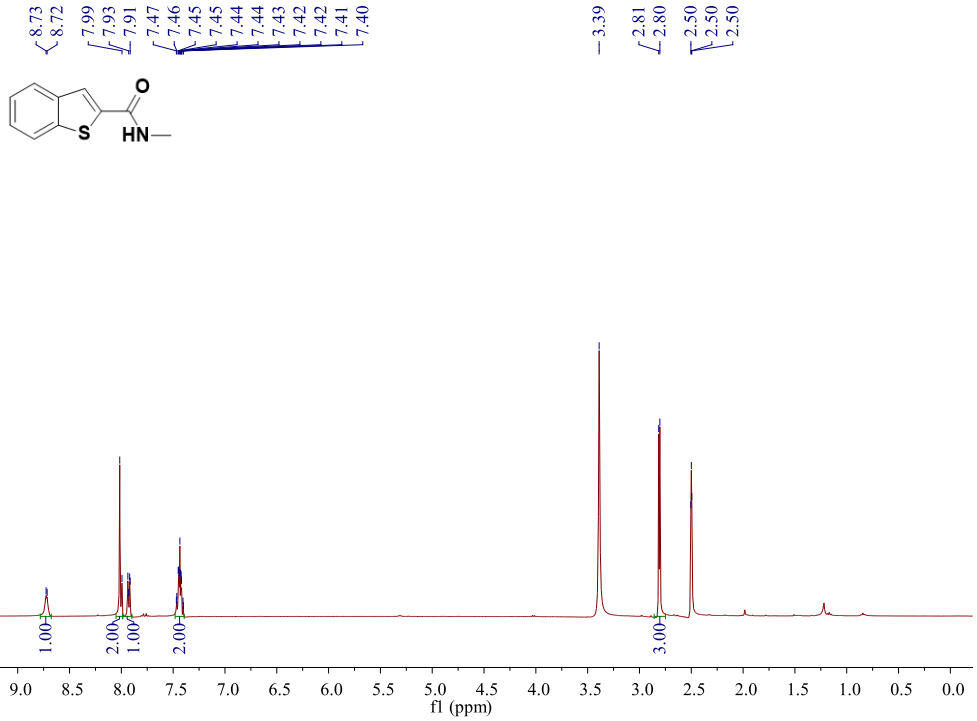


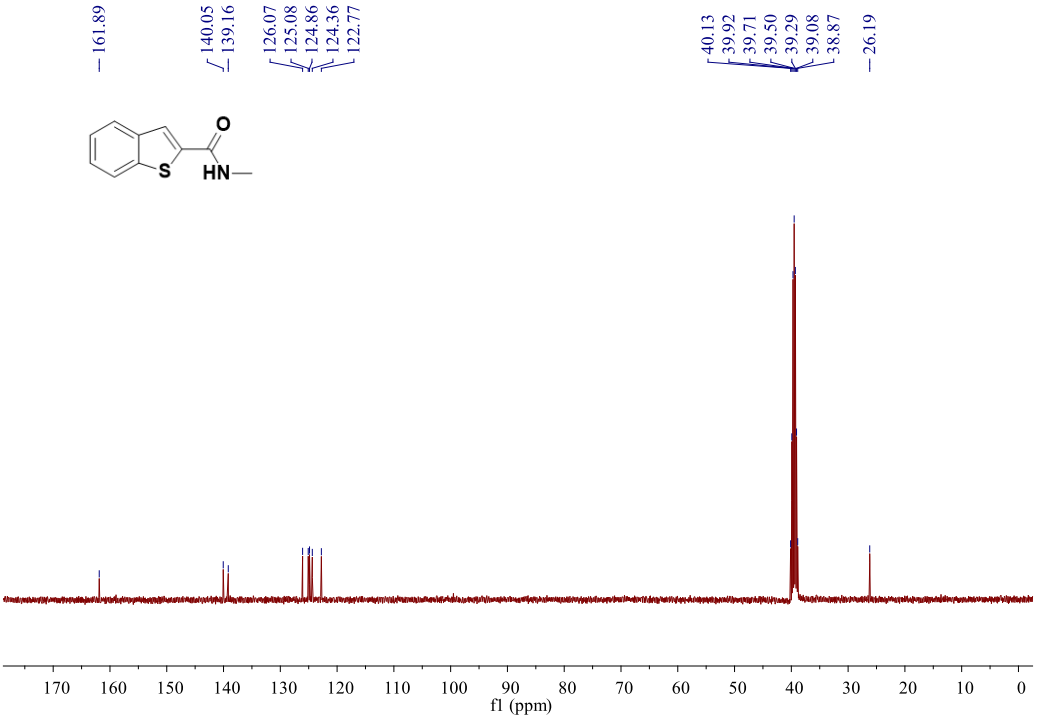

F
O
N
H
F
-114.18
2.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
f1 (ppm)

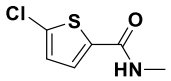


***N*-Methylbenzothiophene-2-carboxamide (3t)**

petroleum ether / ethyl acetate = 2:1, white solid, 51% yield (19.5 mg). mp: 200 – 201℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.72 (d, *J* = 4.1 Hz, 1H), 8.02 – 7.99 (m, 2H), 7.94 – 7.91 (m, 1H), 7.47 – 7.40 (m, 2H), 2.81 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 161.89, 140.05, 139.16, 126.07, 125.08, 124.86, 124.36, 122.77, 26.19. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_9NOS+Na^+$: 214.0297, Found: 214.0309. **IR** (neat, cm^{-1}): υ 3314, 3060, 1626, 1557, 1495, 1300, 757.

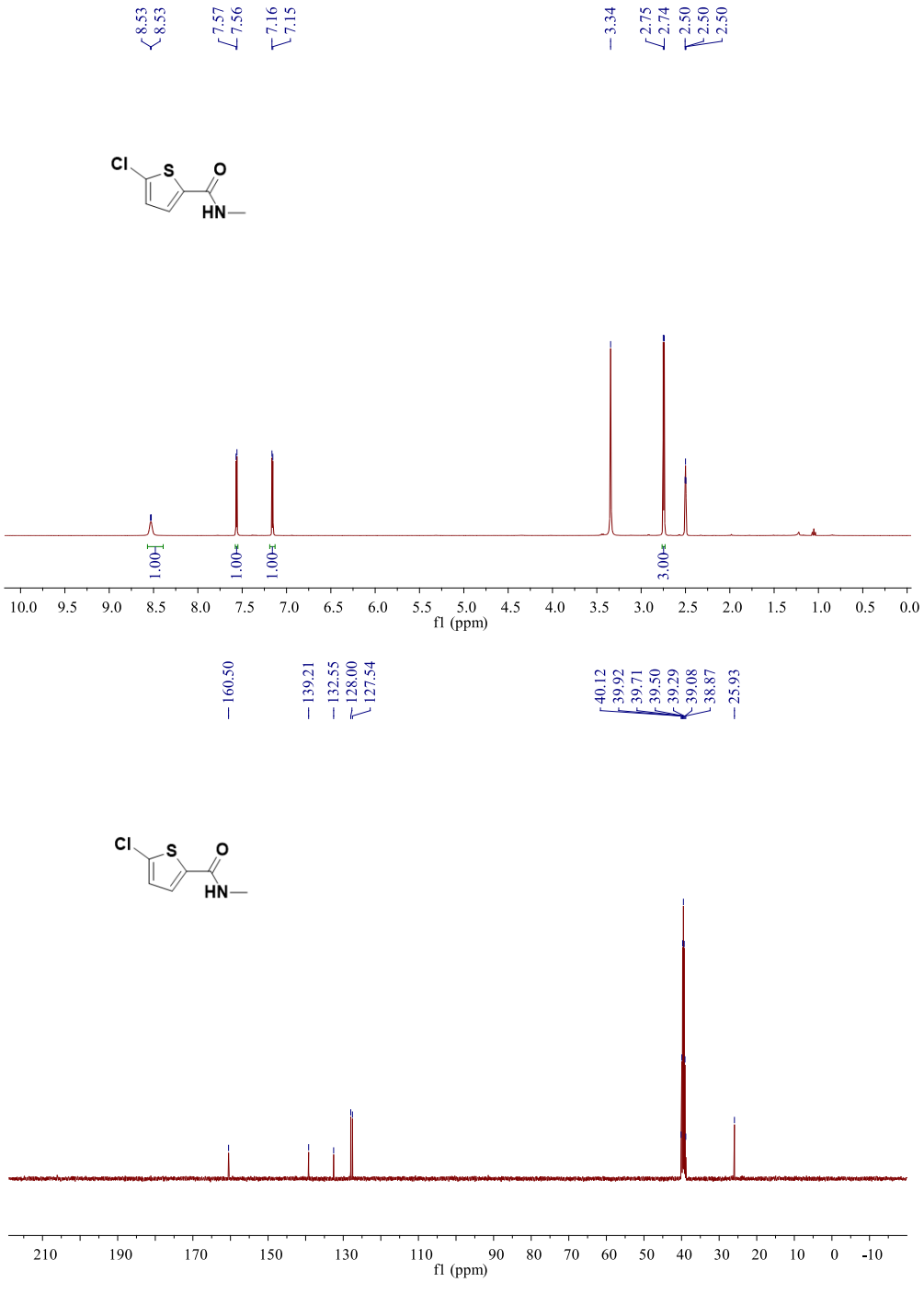


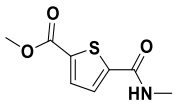




5-Chloro-*N*-methylthiophene-2-carboxamide (3u)

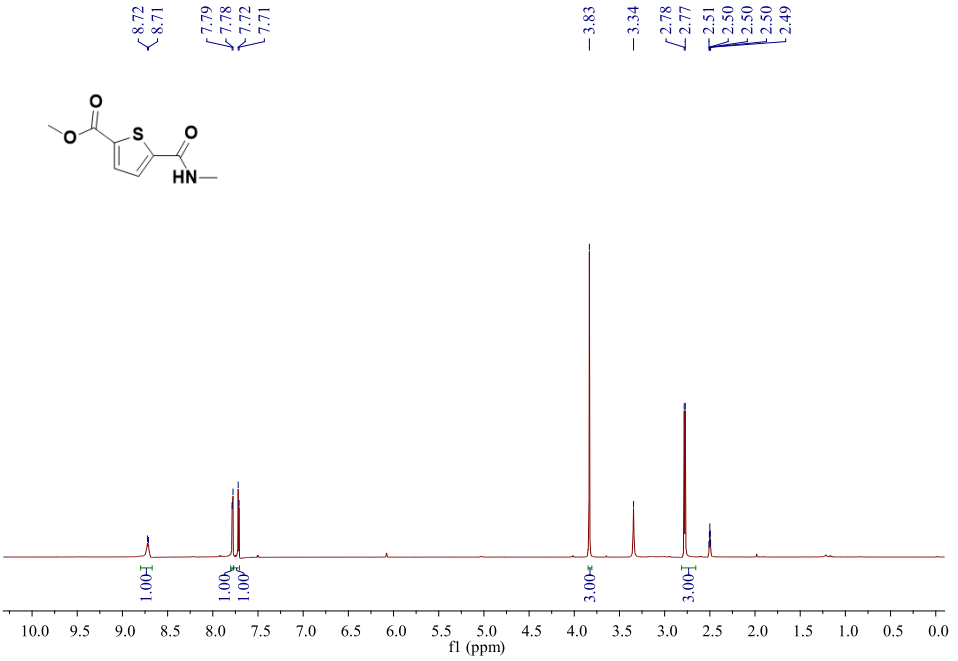
petroleum ether / ethyl acetate = 2:1, white solid, 70% yield (24.5 mg). mp: 170 – 172℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.53 (d, *J* = 3.7 Hz, 1H), 7.56 (d, *J* = 4.0 Hz, 1H), 7.16 (d, *J* = 4.0 Hz, 1H), 2.74 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 160.50, 139.21, 132.55, 128.00, 127.54, 25.93. **HRMS** (ESI-TOF): Anal Calcd. For. $C_6H_6{}^{35}ClNOS+Na^+$: 197.9751, Found: 197.9750. Anal Calcd. For. $C_6H_6{}^{37}ClNOS+Na^+$: 199.9721, Found: 199.9718. **IR** (neat, cm^{-1}): υ 3282, 2982, 1645, 1593, 1429, 1299, 812, 791.

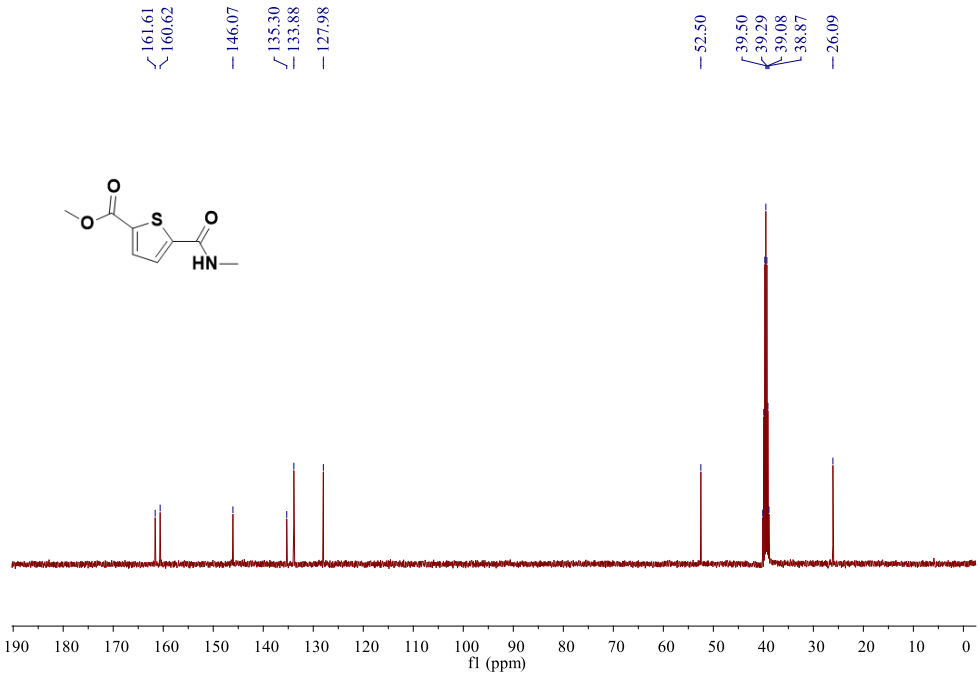


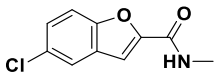


Methyl 5-(methylcarbamoyl)thiophene-2-carboxylate (3v)

petroleum ether / ethyl acetate = 2:1, white solid, 57% yield (22.7 mg). mp: 148 – 150℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.72 (d, *J* = 4.4 Hz, 1H), 7.78 (d, *J* = 4.0 Hz, 1H), 7.71 (d, *J* = 4.0 Hz, 1H), 3.83 (s, 1H), 2.78 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 161.61, 160.62, 146.07, 135.30, 133.88, 127.98, 52.50, 26.09. **HRMS** (ESI-TOF): Anal Calcd. For. $C_8H_9NO_3S$ +Na^+: 222.0195, Found: 222.0196. **IR** (neat, cm^{-1}): υ 3262, 2937, 1654, 1572, 1480, 1348, 1264, 820, 730.

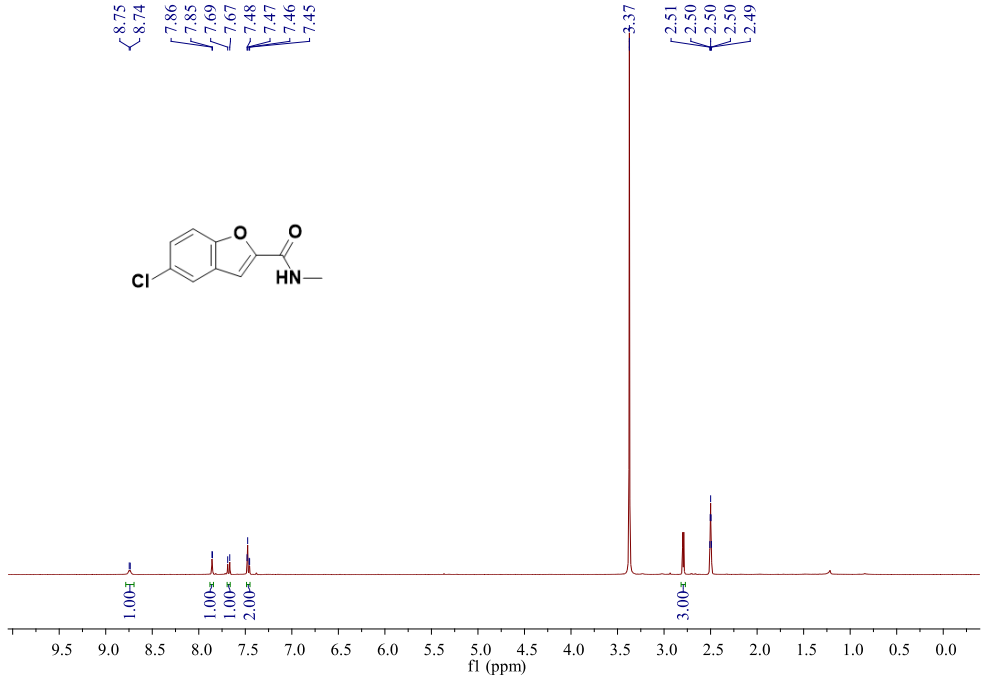


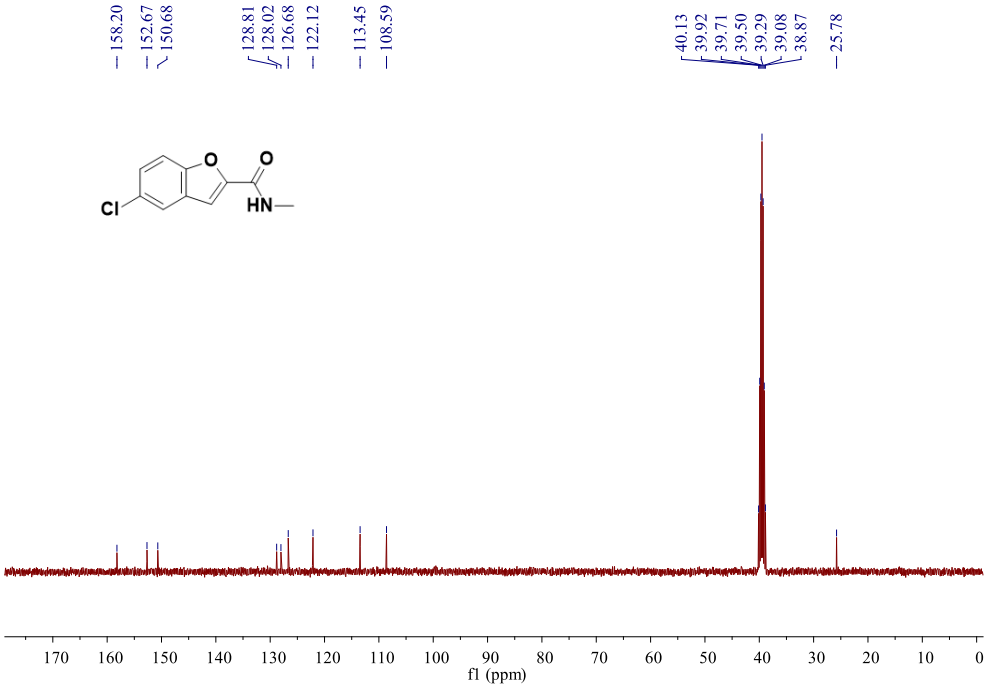


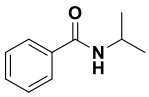


5-Chloro-*N*-methylbenzofuran-2-carboxamide (3w)

petroleum ether / ethyl acetate = 2:1, white solid, 43% yield (18.0 mg). mp: 160 – 162℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.74 (d, *J* = 4.4 Hz, 1H), 7.86 (d, *J* = 2.1 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.48 (d, *J* = 2.1 Hz, 1H), 2.88 – 2.72 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 158.20, 152.67, 150.68, 128.81, 128.02, 126.68, 122.12, 113.45, 108.59, 25.78. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_8{}^{35}ClNO_2$+Na^+: 232.0136, Found: 232.0140. Anal Calcd. For. $C_{10}H_8{}^{37}ClNO_2$+Na^+: 234.0106, Found: 234.0069. **IR** (neat, cm^{-1}): υ 3331, 2930, 1655, 1599, 1440, 1348, 880, 776.

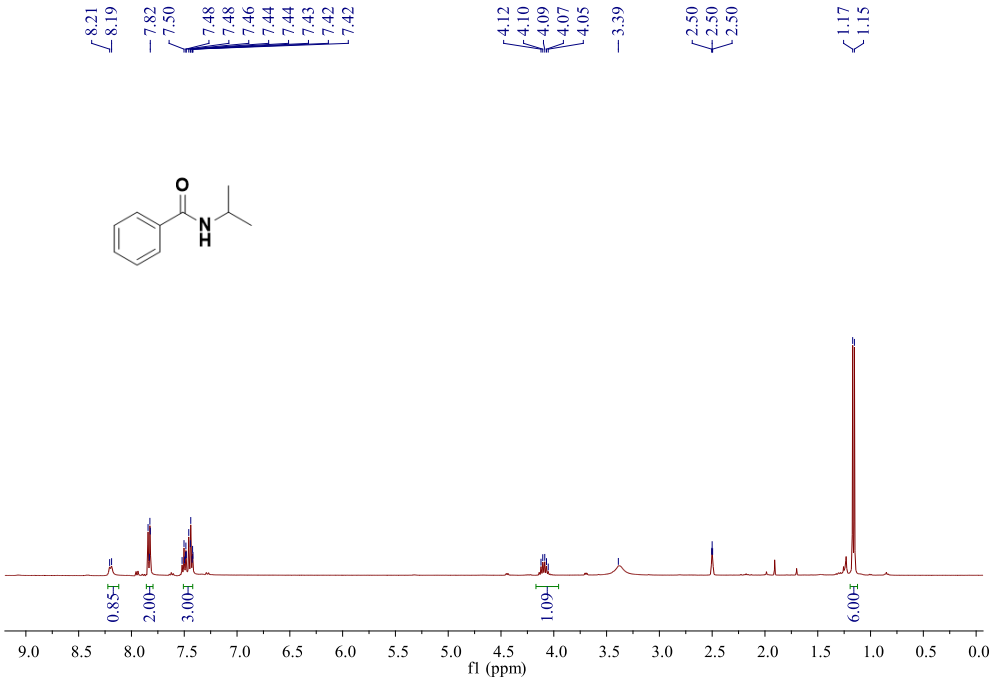


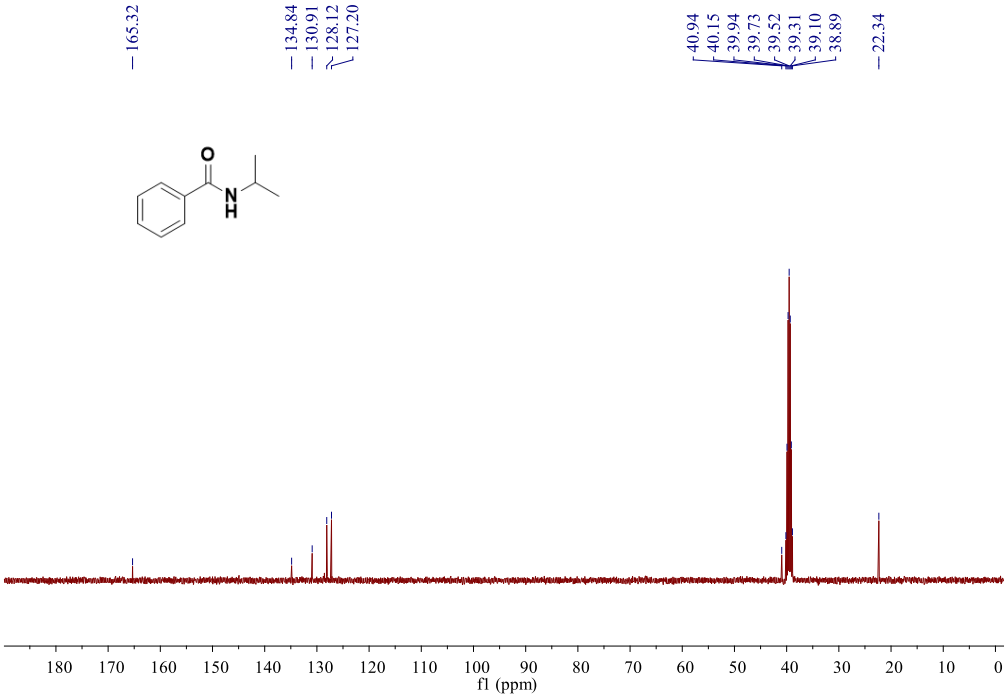


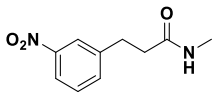


***N*-Isopropylbenzamide (3x)**

petroleum ether / ethyl acetate = 2:1, white solid, 49% yield (16.0 mg). mp: 84 – 86℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.20 (d, *J* = 7.3 Hz, 1H), 7.84 – 7.82 (m, 2H), 7.52 – 7.42 (m, 3H), 4.14 – 4.05 (m, 1H), 1.16 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.32, 134.84, 130.91, 128.12, 127.20, 40.94, 22.34. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{13}NO+Na^{+}$: 186.0889, Found: 186.0885. **IR** (neat, cm^{-1}): υ 3333, 2937, 1719, 1661, 1599, 1407, 758, 692.

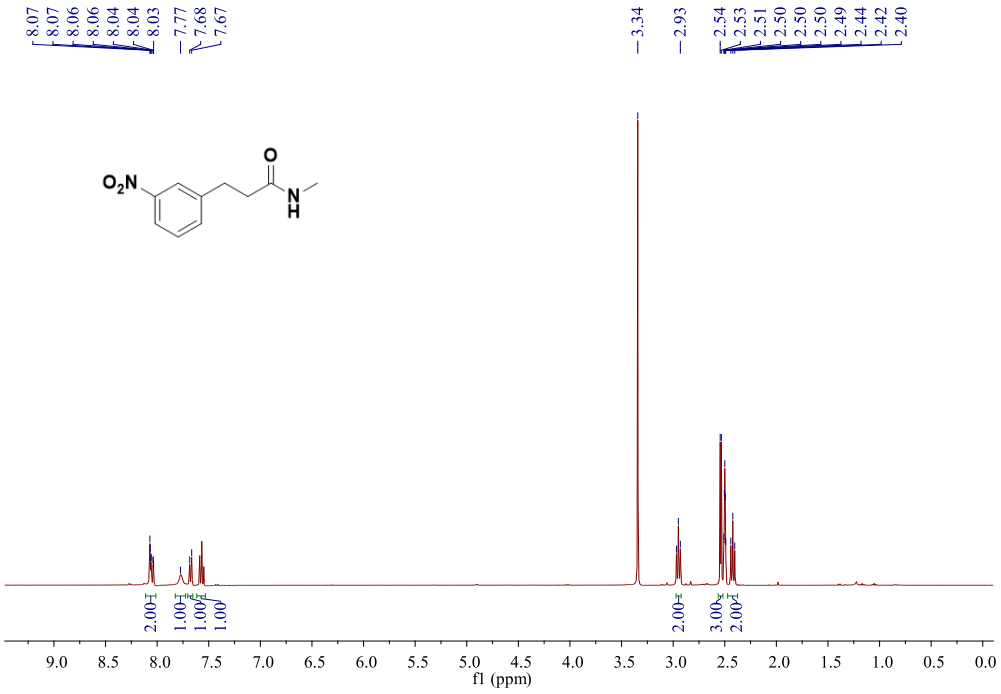


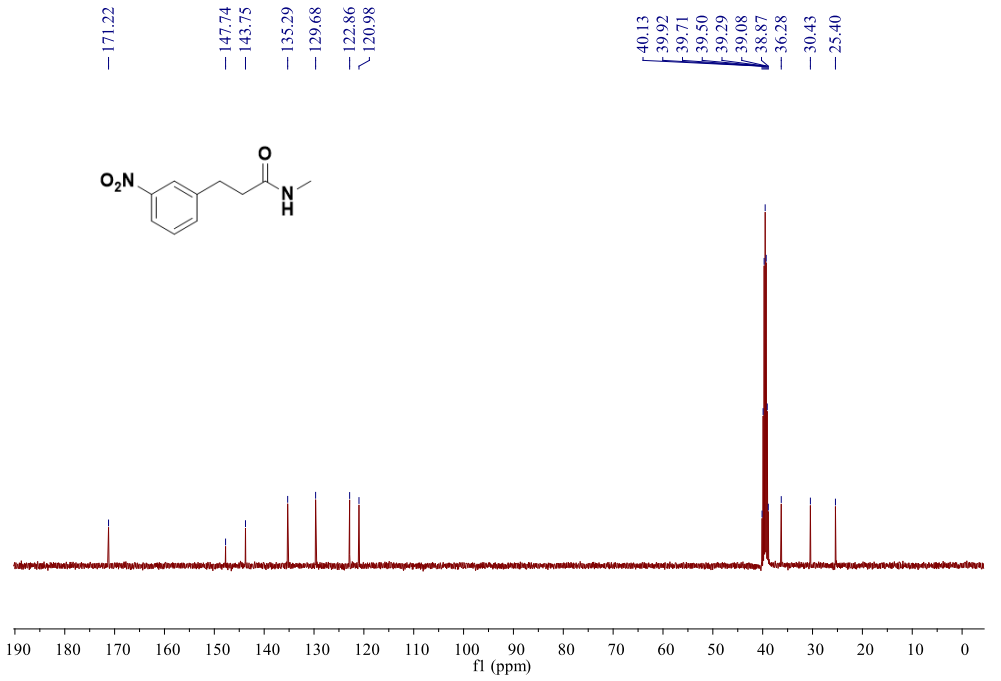


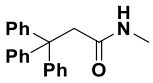


***N*-Methyl-3-(3-nitrophenyl)propenamide (3y)**

petroleum ether / ethyl acetate = 1:1, yellow solid, 42% yield (17.5 mg). mp: 84 – 85℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.07 – 8.03 (m, 2H), 7.77 (s, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 2.95 (t, *J* = 7.6 Hz, 2H), 2.54 (d, *J* = 4.6 Hz, 3H), 2.42 (t, *J* = 7.6 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.22, 147.74, 143.75, 135.29, 129.68, 122.86, 120.98, 36.28, 30.43, 25.40. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{12}N_2O_3$+Na^+: 231.0740, Found: 231.0747. **IR** (neat, cm^{-1}): υ 3262, 2932, 1635, 1572, 1348, 887, 730.

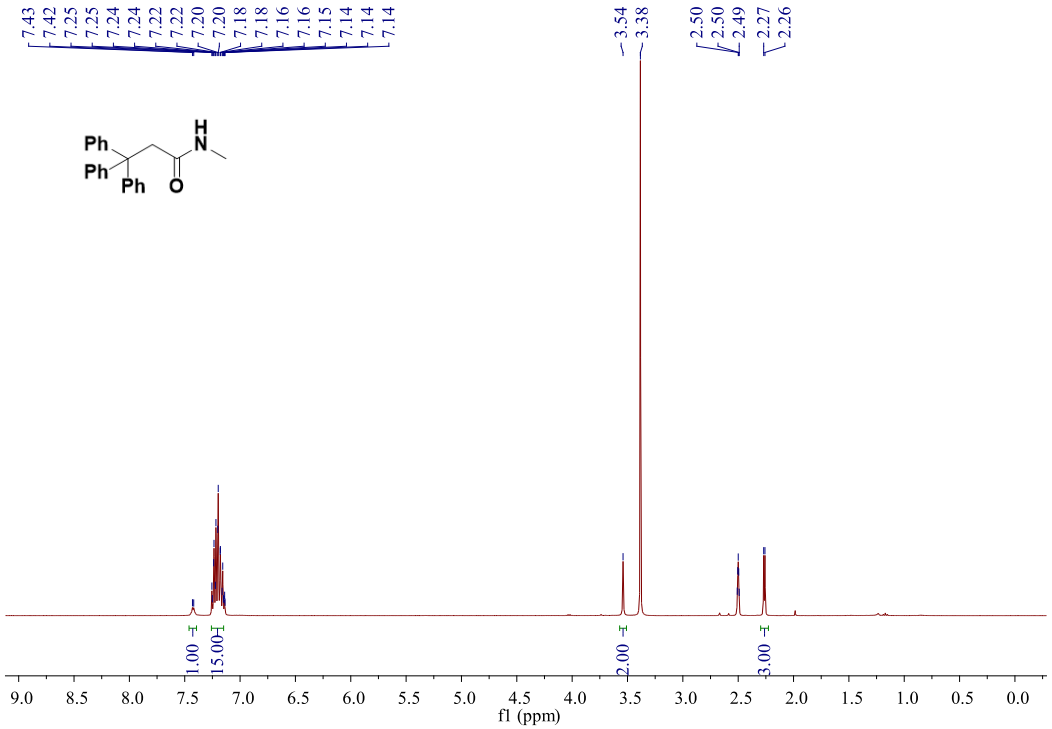


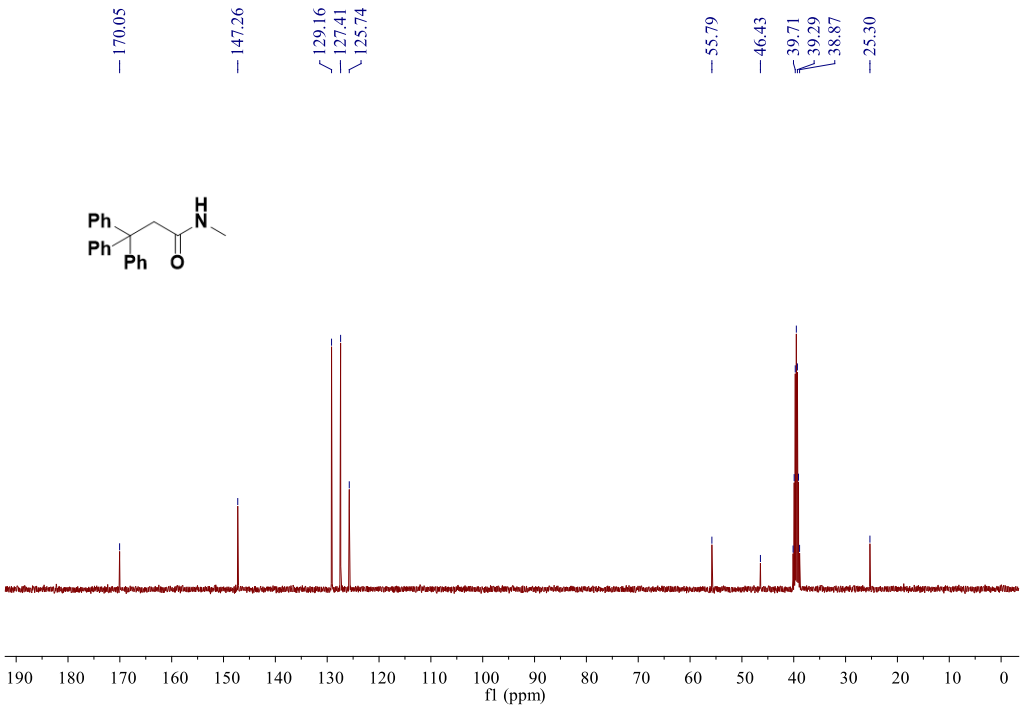


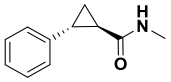


***N*-methyl-3,3,3-triphenylpropanamide (3z)**

petroleum ether / ethyl acetate = 2:1, white solid, 68% yield (42.9 mg). mp: 210 – 211℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 7.42 (d, *J* = 4.5 Hz, 1H), 7.25 – 7.14 (m, 15H), 3.54 (s, 2H), 2.26 (d, *J* = 4.6 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 170.05, 147.26, 129.16, 127.41, 125.74, 55.79, 46.43, 25.30. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{22}H_{21}NO+Na^{+}$: 338.1515, Found: 338.1523. **IR** (neat, cm^{-1}): υ 3262, 2935, 1657, 1594, 1407, 1363, 763, 695.

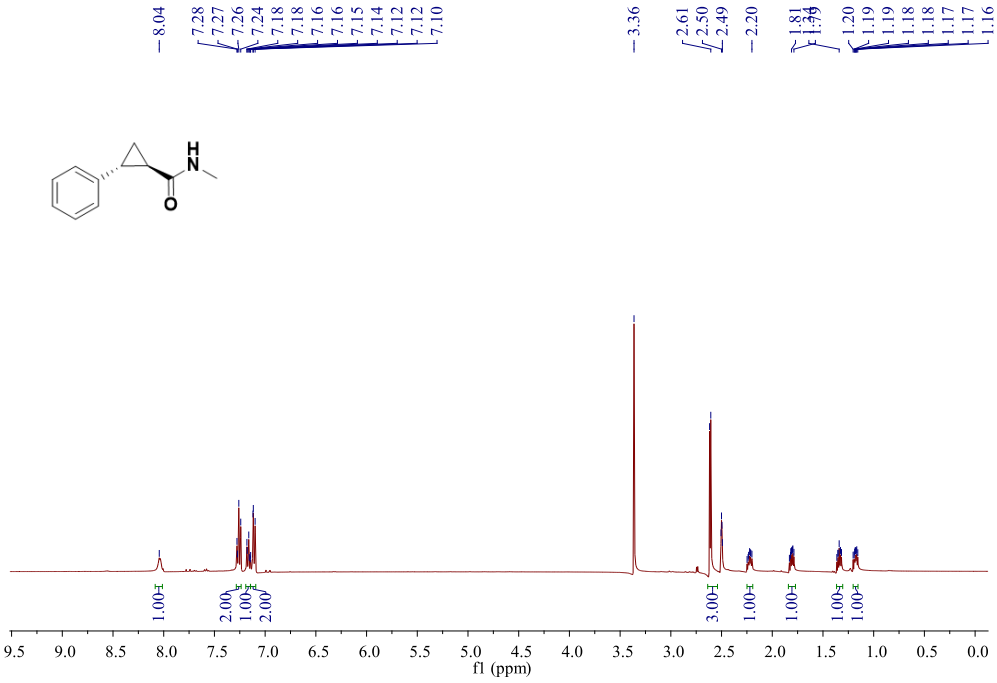


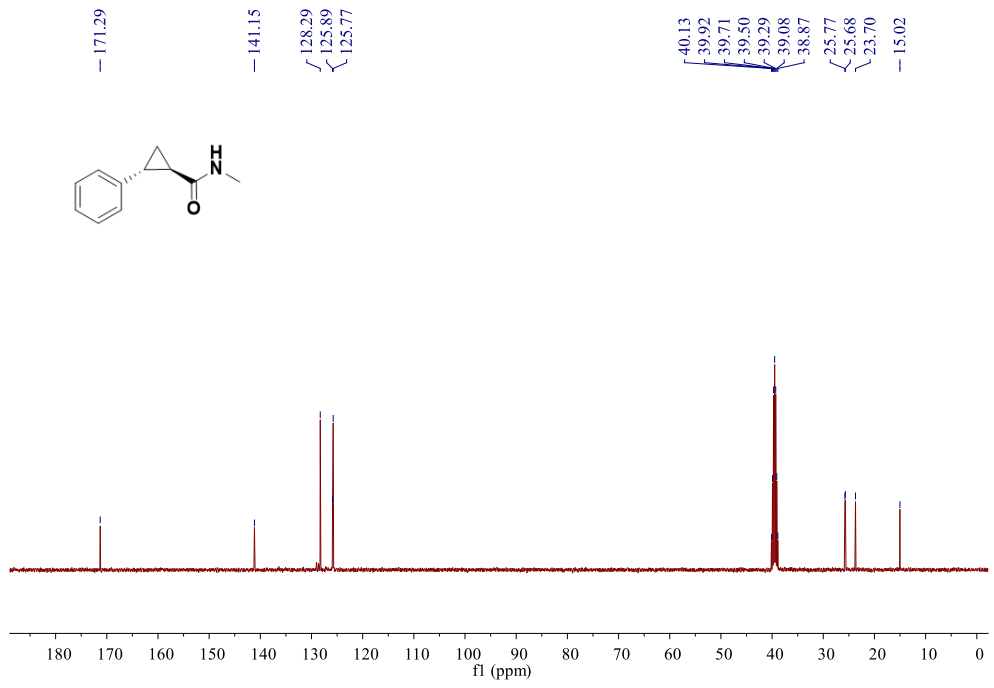


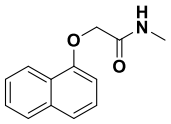


(1R,2R)-*N*-methyl-2-phenylcyclopropane-1-carboxamide (3aa)

petroleum ether / ethyl acetate = 5:1, yellow solid, 33% yield (11.6 mg). mp: 98 – 100℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.04 (s, 1H), 7.28 – 7.24 (m, 2H), 7.18 – 7.14 (m, 1H), 7.12 – 7.10 (m, 2H), 2.61 (d, *J* = 4.6 Hz, 3H), 2.25 – 2.20 (m, 1H), 1.83 – 1.79 (m, 1H), 1.36 – 1.32 (m, 1H), 1.20 – 1.16 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.29, 141.15, 128.29, 125.89, 125.77, 25.77, 25.68, 23.70, 15.02. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{11}H_{13}NO+Na^{+}$: 198.0889, Found: 198.0898. **IR** (neat, cm^{-1}): υ 3346, 1634, 1589, 1458, 958, 845, 764.

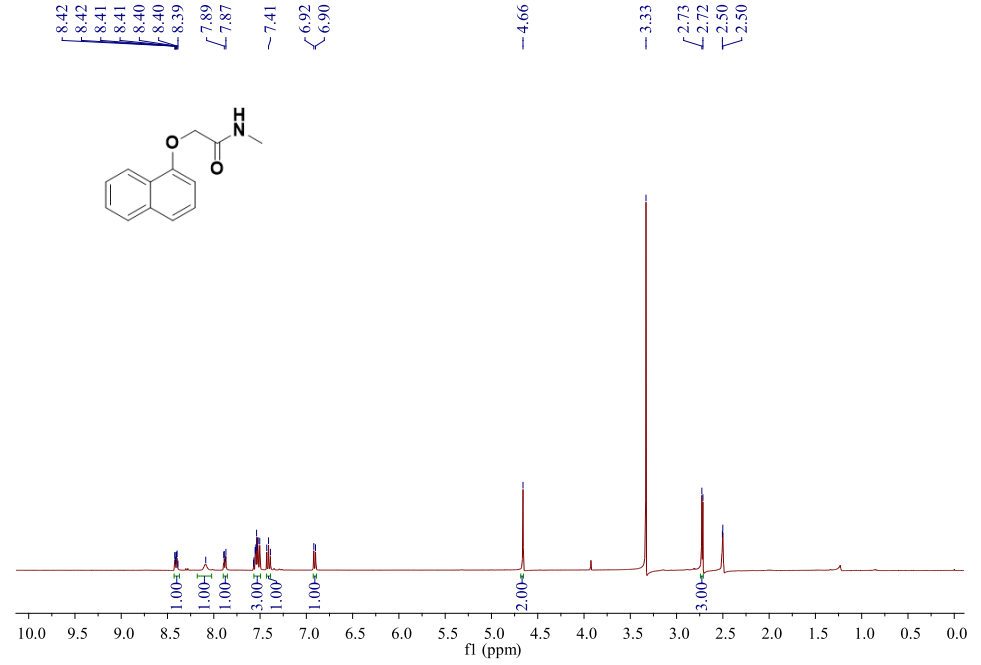


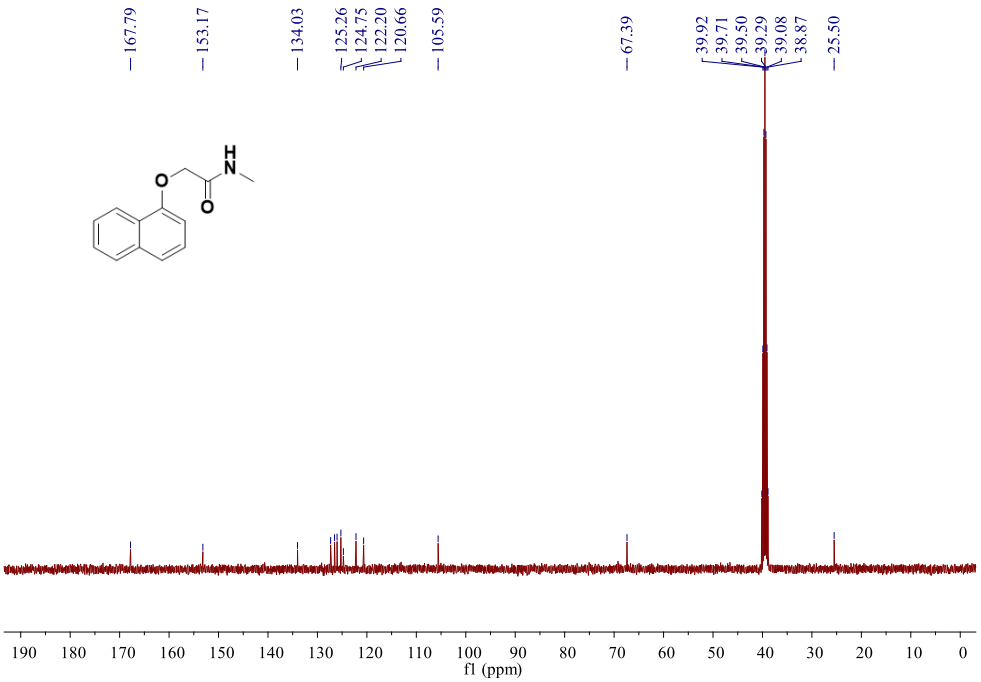


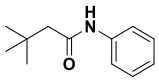


***N*-Methyl-2-(naphthalen-1-yloxy)acetamide (3ab)**

petroleum ether / ethyl acetate = 2:1, light yellow solid, 50% yield (21.5 mg). mp: 98 – 100℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 8.42 – 8.40 (m, 1H), 8.09 (s, 1H), 7.89 – 7.87 (m, 1H), 7.55 – 7.50 (m, 3H), 7.43 – 7.39 (m, 1H), 6.91 (d, *J* = 7.3 Hz, 1H), 4.66 (s, 2H), 2.72 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 167.79, 153.17, 134.03, 127.33, 126.54, 126.03, 125.26, 124.75, 122.20, 120.66, 105.59, 67.39, 25.50. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{13}H_{13}NO_2$ +Na^+: 238.0838, Found: 238.0841. **IR** (neat, cm^{-1}): υ 3262, 2937, 1655, 1572, 1457, 1348, 1264, 765, 682.

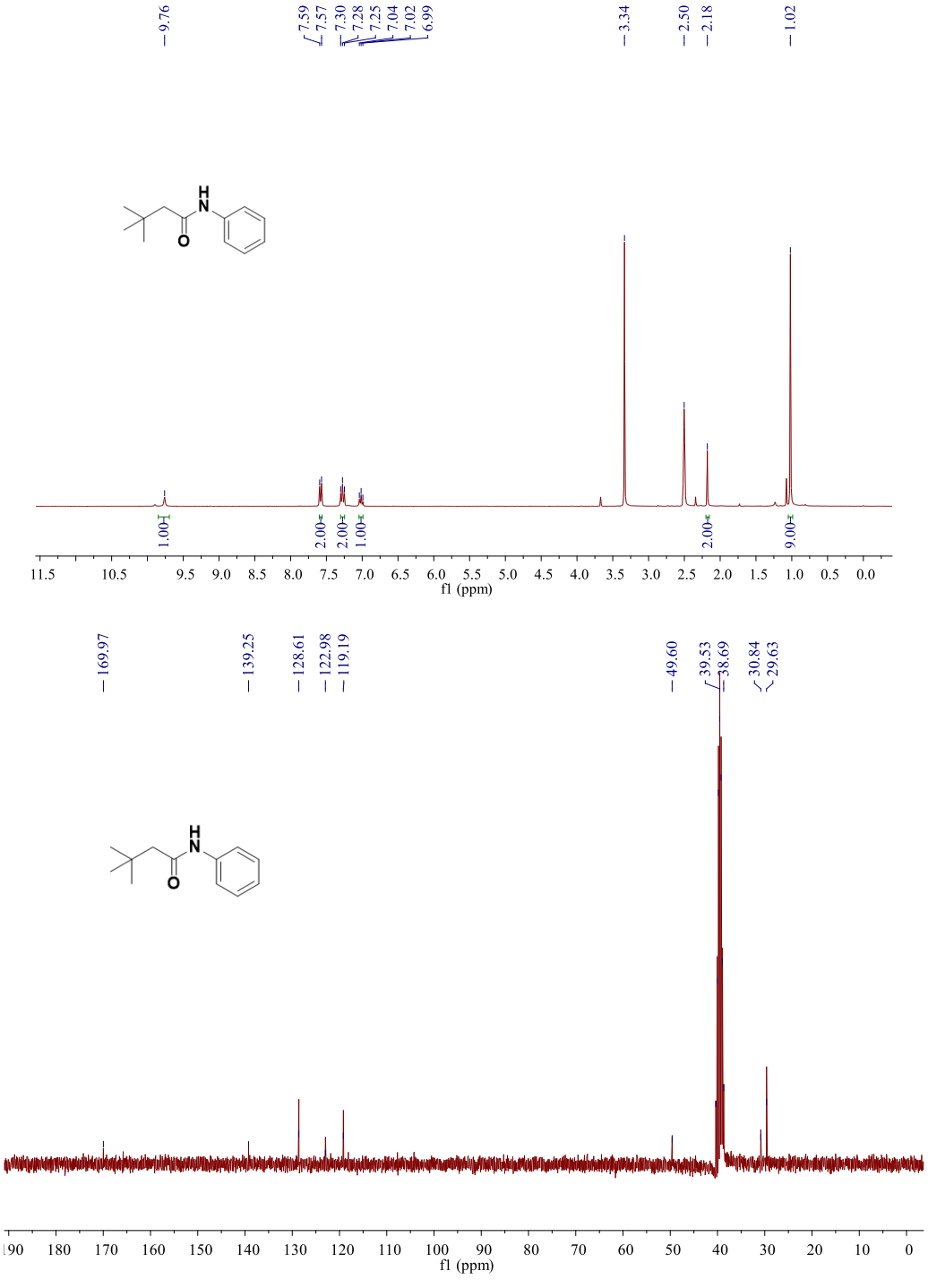


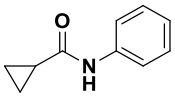




3,3-Dimethyl-*N*-phenylbutanamide (3ac)

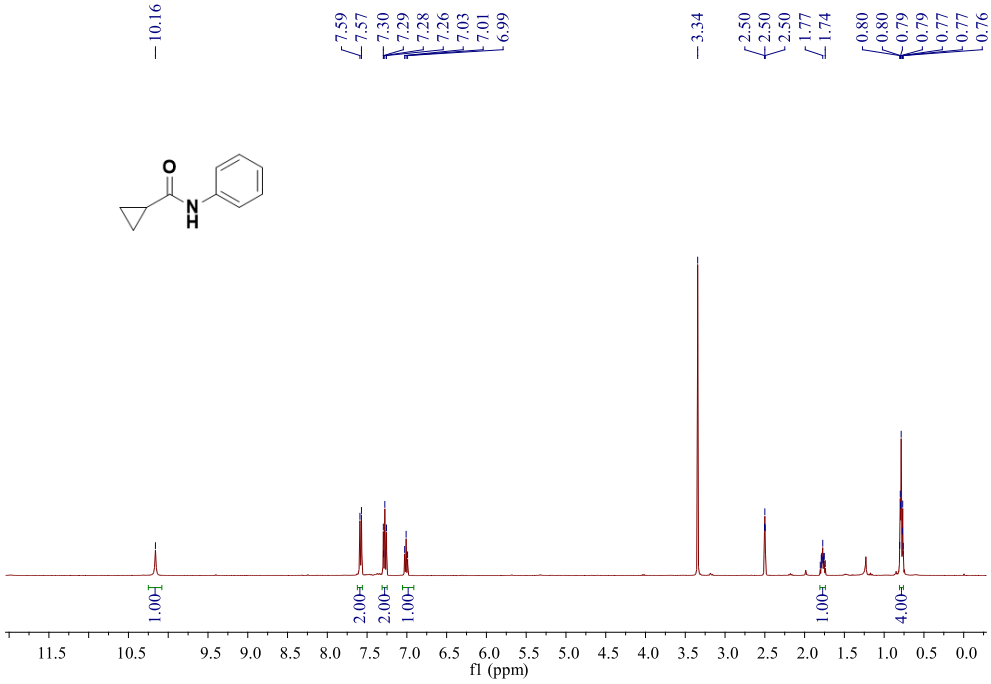
petroleum ether / ethyl acetate = 2:1, white solid, 57% yield (21.8 mg). mp: 130 – 131℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 9.76 (s, 1H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.28 (t, *J* = 8.0 Hz, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 2.18 (s, 2H), 1.02 (s, 9H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 169.97, 139.25, 128.61, 122.98, 119.19, 49.60, 30.84, 29.63. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{12}H_{17}NO$ +Na^{+}: 214.1202, Found: 214.1209. **IR** (neat, cm^{-1}): υ 3262, 2937, 1654, 1573, 1434, 1348, 730, 683.

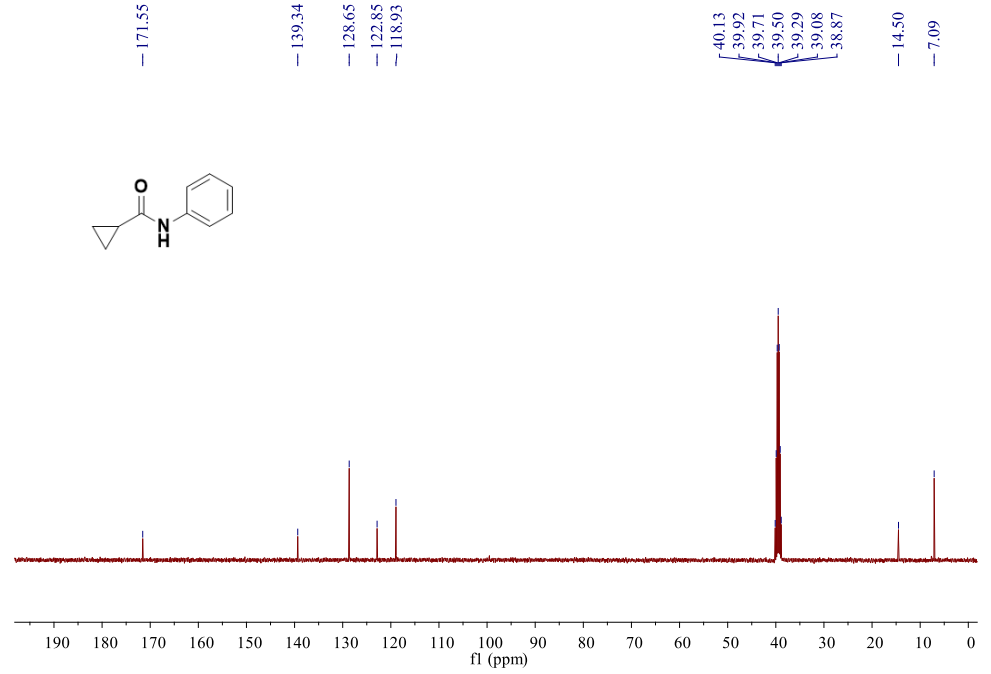


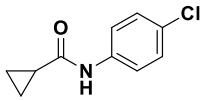


***N*-phenylcyclopropanecarboxamide (3ad)**

petroleum ether / ethyl acetate = 5:1, white solid, 80% yield (25.8 mg). mp: 109 – 111℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 10.16 (s, 1H), 7.58 (d, *J* = 7.6 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.01 (t, *J* = 7.6 Hz, 1H), 1.80 – 1.74 (m, 1H), 0.80 – 0.76 (m, 4H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.55, 139.34, 128.65, 122.85, 118.93, 14.50, 7.09. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{11}NO+Na^{+}$: 184.0733, Found: 184.0727. **IR** (neat, cm^{-1}): υ 3277, 2960, 1651, 1540, 1439, 1307, 754, 693.

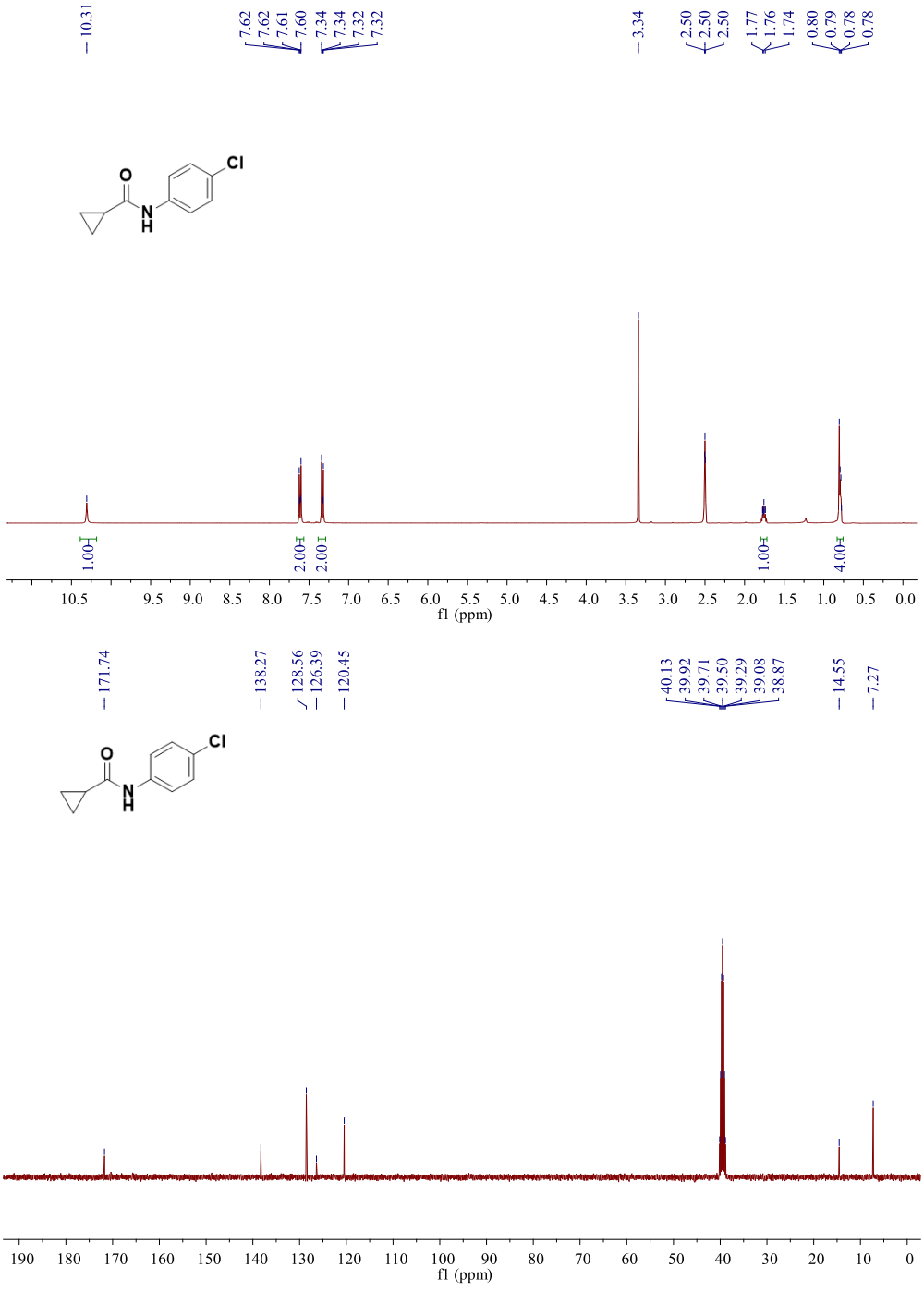


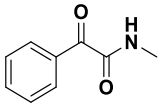




***N*-(4-nitrophenyl)cyclopropanecarboxamide (3ae)**

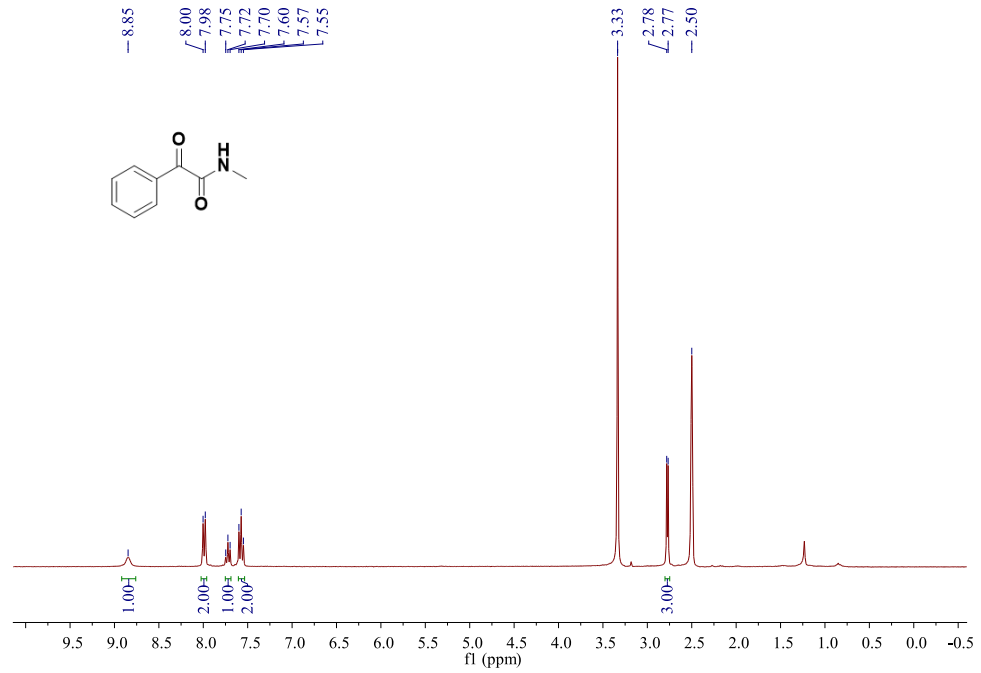
petroleum ether / ethyl acetate = 5:1, white solid, 68% yield (26.5 mg). mp: 164 – 165℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 10.31 (s, 1H), 7.62 – 7.60 (m, 2H), 7.34 – 7.32 (m, 2H), 1.77 – 1.74 (m, 1H), 0.80 – 0.78 (m, 4H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 171.74, 138.27, 128.56, 126.39, 120.45, 14.55, 7.27. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{10}H_{10}{}^{35}ClNO+Na^{+}$: 218.0343, Found: 218.0340. Anal Calcd. For. $C_{10}H_{10}{}^{37}ClNO+Na^{+}$: 220.0314, Found: 220.0307. **IR** (neat, cm^{-1}): υ 3281, 2920, 1653, 1593, 1445, 1390, 828, 782.

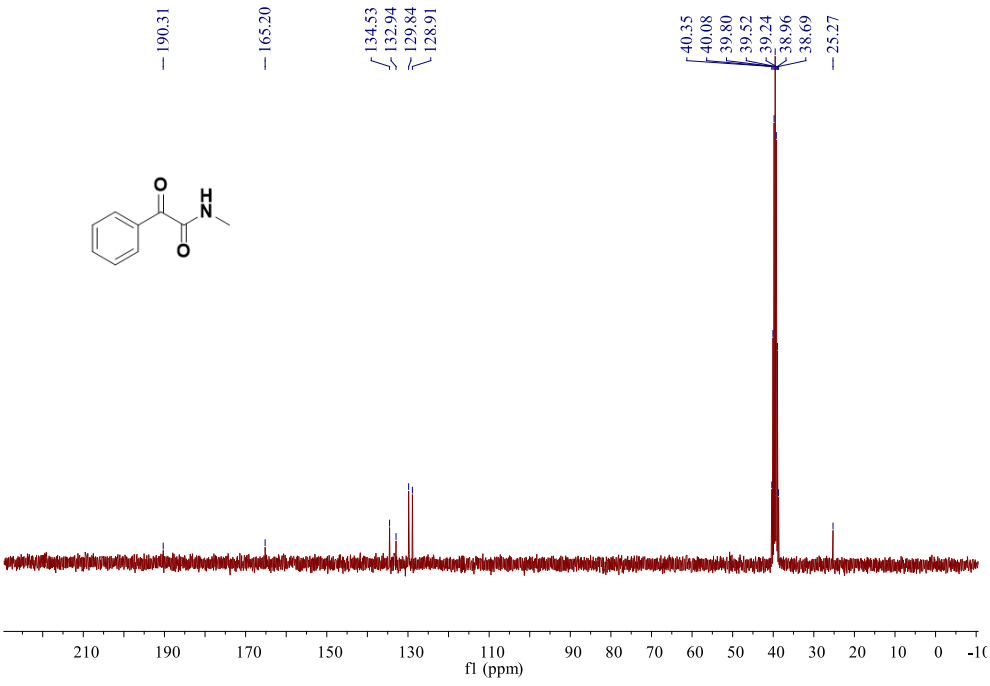


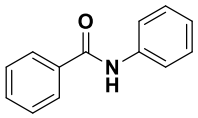


***N*-Methyl-2-oxo-2-phenylacetamide (3af)**

petroleum ether / ethyl acetate = 5:1, yellow solid, 41% yield (13.4 mg). mp: 66 – 68℃. **^{1}H NMR** (300 MHz, DMSO-*d6*) δ 8.85 (s, 1H), 7.99 (d, *J* = 7.5 Hz, 2H), 7.75 – 7.70 (m, 1H), 7.60 – 7.55 (m, 2H), 2.77 (d, *J* = 4.7 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO-*d6*) δ 190.31, 165.20, 134.53, 132.94, 129.84, 128.91, 25.27. **HRMS** (ESI-TOF): Anal Calcd. For. $C_9H_9NO_2$+Na^+: 186.0525, Found: 186.0520. **IR** (neat, cm^{-1}): υ 3327, 2932, 1686, 1594, 1447, 1308, 746, 685.

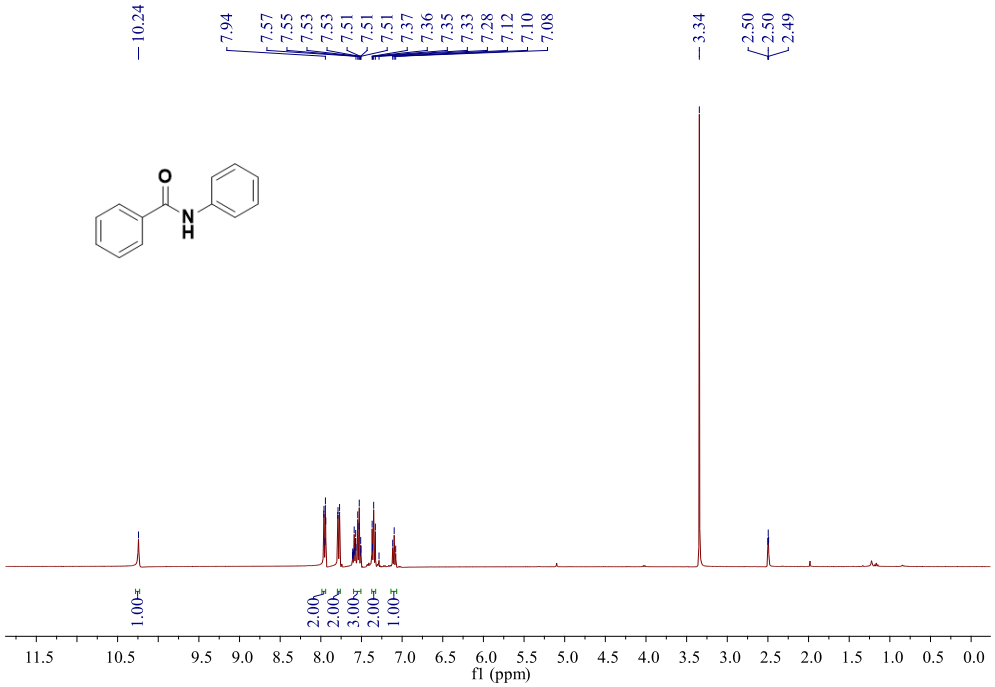


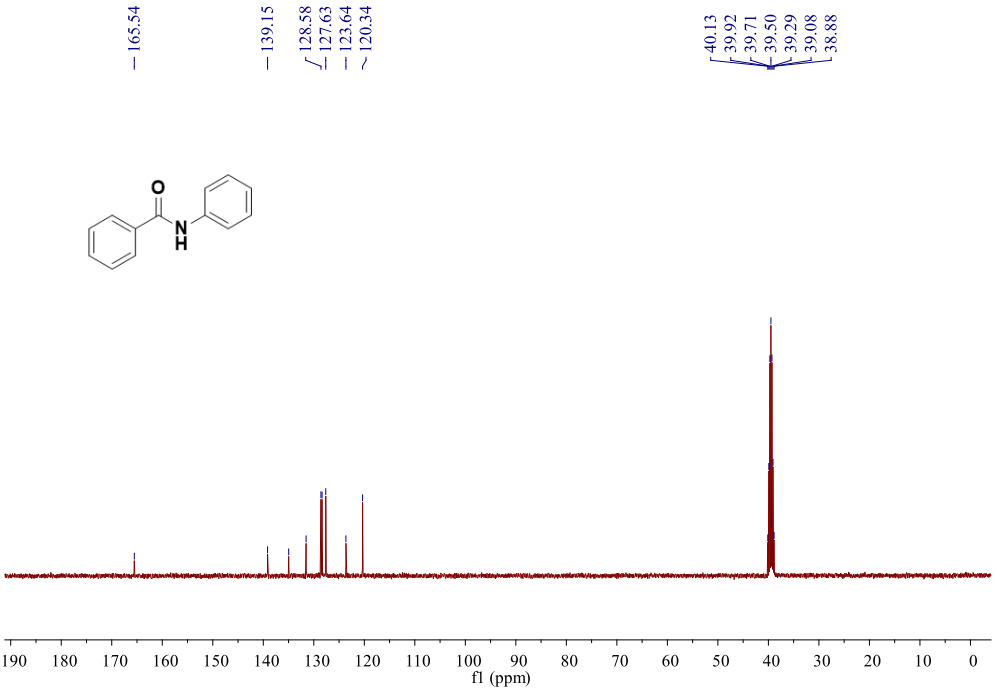


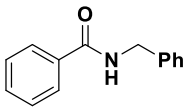


***N*-Phenylbenzamide (3ag)**

petroleum ether / ethyl acetate = 5:1, white solid, 47% yield (18.5 mg). mp: 162 – 163℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 10.24 (s, 1H), 7.96 – 7.94 (m, 2H), 7.79 – 7.77 (m, 2H), 7.61 – 7.51 (m, 3H), 7.37 – 7.33 (m, 2H), 7.10 (t, *J* = 7.4 Hz, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 165.54, 139.15, 134.97, 131.52, 128.58, 128.36, 127.63, 123.64, 120.34. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{13}H_{11}NO+Na^{+}$: 220.0733, Found: 220.0740. **IR** (neat, cm^{-1}): υ 3284, 3030, 1635, 1600, 1549, 1451, 1313, 724, 691.

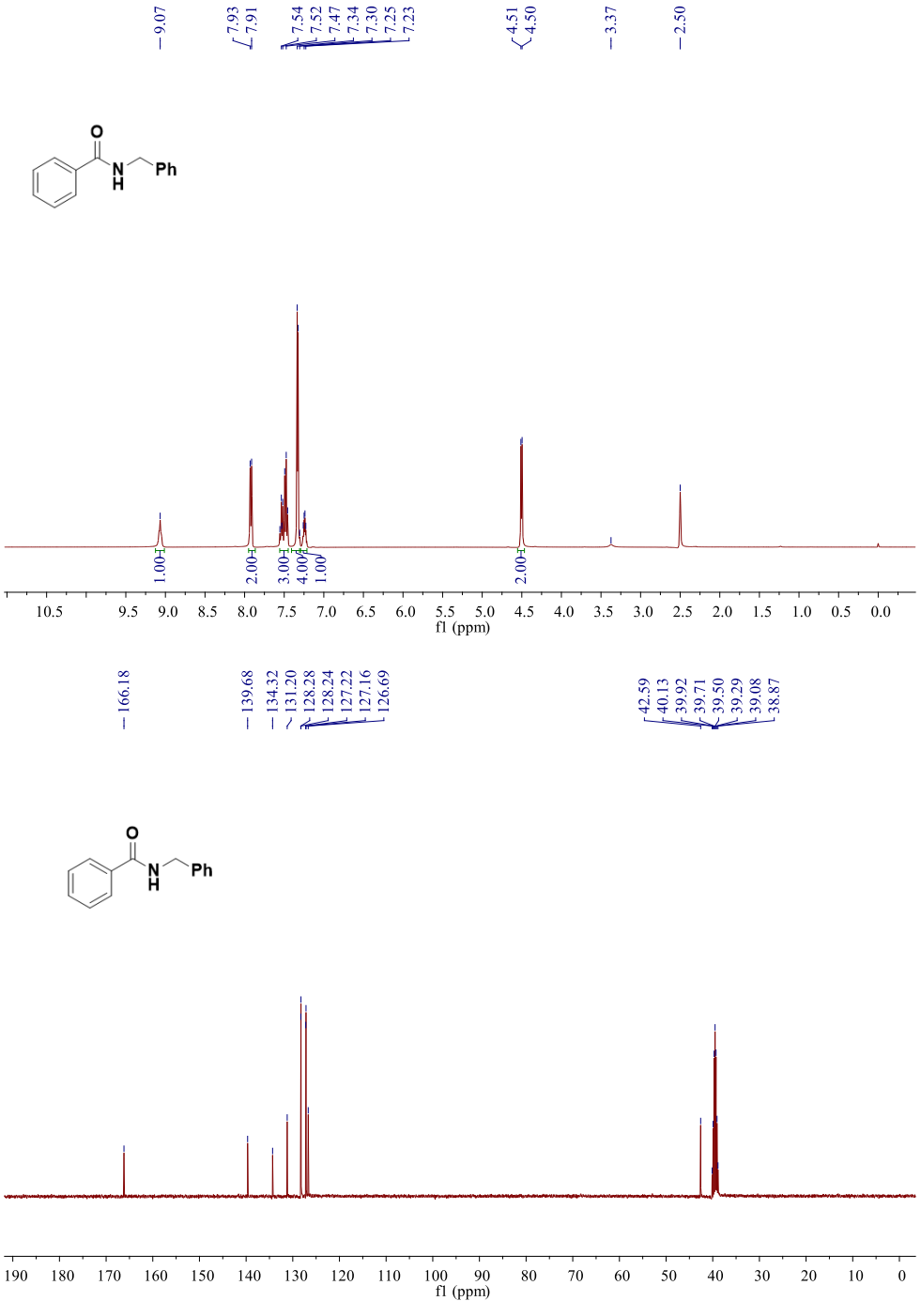


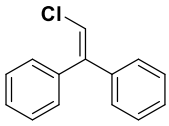




***N*-benzylbenzamide (3ah)**

petroleum ether / ethyl acetate = 2:1, white solid, 52% yield (21.9 mg). mp: 99 – 100℃. **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 9.07 (s, 1H), 7.92 (d, *J* = 7.5 Hz, 2H), 7.55 – 7.46 (m, 3H), 7.34 – 7.30 (m, 4H), 7.26 – 7.23 (m, 1H), 4.50 (d, *J* = 6.0 Hz, 2H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 166.18, 139.68, 134.32, 131.20, 128.28, 128.24, 127.22, 127.16, 126.69, 42.59. **HRMS** (ESI-TOF): Anal Calcd. For. $C_{14}H_{13}NO+Na^{+}$: 234.0889, Found: 234.0891. **IR** (neat, cm^{-1}): υ 3282, 2927, 1635, 1548, 1488, 793, 691.





(2-Chloroethene-1,1-diyl) dibenzene (4)

pure petroleum ether, colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37-7.17 (m, 10H), 6.56 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.82, 140.07, 137.53, 129.82, 128.38, 128.22, 128.16, 128.12, 128.03, 127.92, 127.67, 115.84. **HRMS** (EI-TOF): Anal Calcd. For. $C_{14}H_{11}Cl$: 214.0549, found: 214.0551; IR (neat, cm^{-1}): υ 3023, 1609, 1573, 1442, 896, 770, 694.