ASIAN JOURNAL
OF ORGANIC CHEMISTRY

Supporting Information

Carbonylation of Csp^3–H Bonds through Oxidative Wittig-Type Reaction: An Unprecedented Version of Wittig Reaction

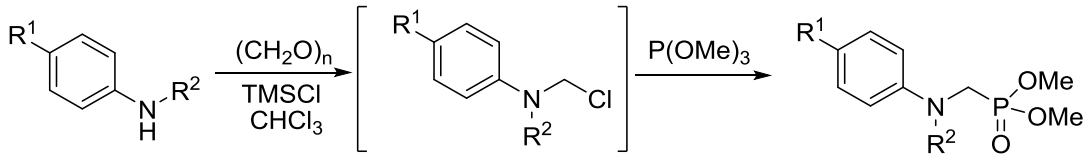
Yingzu Zhu, Shiwei Lü, Xingge Ma, Liang Zhang, Liangliang Luo, and Xiaodong Jia*[a]

ajoc_201600055_sm_miscellaneous_information.pdf

General:

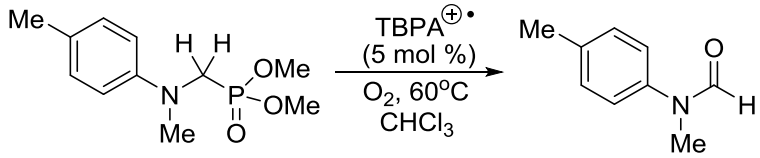
Solvents (CH_2Cl_2, $CHCl_3$, $ClCH_2CH_2Cl$, MeOPh and MeCN) are anhydrous. $TBPA^{+\cdot}$ was purchased from commercial source and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^1H NMR and ^{13}C NMR (400 MHz, 600 MHz and 100 MHz, 150 MHz respectively) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard and spin-spin coupling constants (J) are given in Hz. EI-MS spectra were measured by direct inlet at 70eV.

Synthesis of Starting Materials



A solution of *N*-methylaniline (10 mmol), paraformaldehyde (10mmol) and TMSCl (30 mmol) in 10 mL $CHCl_3$ was refluxed for 6 hours. After completion monitored by TLC, the solvent was removed under reduced pressure, and then $P(OMe)_3$ (5 mmol), triethylamine (5 mmol) and toluene (5 mL) was added. After the reaction solution was refluxed for additional 6 hours, the product was separated by silica gel column chromatography eluted with petroleum ether/acetone (v/v 5:1) to afford the pure compound.

General Experimental Procedure



A solution of **1** (1mmol) in $CHCl_3$ (5ml) was mixed fully and flushed with O_2, then $TBPA^{+\cdot}$ (5 mol %) was added dropwise under oxygen atmosphere. The reaction solution was stirred under 60°C. After completion monitored by TLC (by UV visualization), the reaction was quenched by addition of saturated Na_2CO_3 in MeOH (10 ml) solution. The mixture was poured into a separator funnel with the addition of excess $CHCl_3$ (10 ml), and then the crude organic solution was extracted three times with water to remove inorganic salts. The organic phase was then dried over anhydrous magnesium sulfate, filtered, and the solvent was removed under reduced pressure. The products were separated by silica gel column chromatography eluted with petroleum ether/acetone (v/v 10:1) to afford the products.

Analytical data for compounds

***N*-Methyl-*N*-(p-tolyl)formamide (2a)**

^1H NMR (600 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 3.28 (s, 2H), 2.35 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.3, 139.7, 136.4, 130.1, 122.5, 32.2, 20.8; **EI-MS***m/z* (relative intensity, %): 149 (91.0%), 120 (100%), 108 (43.0%), 91 (26.8%).

***N*-(4-Methoxyphenyl)-*N*-methylformamide (2b)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 3.80 (s, 3H), 3.25 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.4, 158.3, 135.2, 124.6, 114.7, 55.5, 32.7;**EI-MS***m/z* (relative intensity, %): 165 (100%), 136 (15.4%), 124 (55.6%), 108 (12.0%), 94 (25.5%).

***N*-(4-ethoxyphenyl)-*N*-methylformamide (2c)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (s, 1H), 7.07 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.03 (q, *J* = 7.0 Hz, 2H), 3.26 (s, 3H), 1.41 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.4, 157.6, 135.0, 124.6, 115.2, 63.8, 32.6, 14.7;**EI-MS***m/z* (relative intensity, %): 179 (100%), 150 (13.0%), 138 (21.4%), 122 (84.6%), 110 (52.9%), 94 (28.9%).

***N*-Methyl-*N*-phenylformamide (2d)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.47 (s, 1H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 2H), 3.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.3, 142.2, 129.6, 126.4, 122.4, 32.0; **EI-MS***m/z* (relative intensity, %): 135 (91.4%), 120 (12.3%), 106 (100%), 94 (34.9%), 77 (44.3%), 44 (61.8%).

***N*-(4-Fluorophenyl)-*N*-methylformamide (2e)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.18 – 7.06 (m, 4H), 3.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.2, 161.0 (d, J_{CF}= 246.7 Hz), 138.3 (d, J_{CF} = 3.0 Hz), 124.6 (d, J_{CF} = 8.4 Hz), 116.5 (d, J_{CF}= 22.8 Hz), 32.5; **EI-MS***m/z* (relative intensity, %): 153 (66.5%), 124 (100%), 112 (34.6%), 97 (36.3%), 83 (31.1%).

***N*-(4-Chlorophenyl)-*N*-methylformamide (2f)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.44 (s, 1H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 8.4 Hz, 2H), 3.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.0, 140.7, 132.0, 129.7, 129.1 (minor rotamor), 124.6 (minor rotamor), 123.5, 32.0, 30.9 (minor rotamor); **EI-MS***m/z* (relative intensity, %): 171 (23.3%), 169 (77.5%), 142 (30.9%), 140 (100%), 130 (17.9%), 128 (59.4%).

***N*-(4-Bromophenyl)-*N*-methylformamide (2g)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.05 (d, *J* = 8.7 Hz, 2H), 3.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 161.9, 141.2, 132.7, 132.0 (minor rotamor), 124.8 (minor rotamor), 123.8, 119.7, 32.0;**EI-MS***m/z* (relative intensity, %): 215 (84.2%), 213 (86.2%), 186 (89.0%), 184 (100%), 174 (65.9%), 172 (72.1%), 105 (42.0).

***N*-(2,4-Dimethylphenyl)-*N*-methylformamide (2h)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.09 (s, 1H), 7.04 (d, *J* = 7.9 Hz, 1H), 6.98 (d, *J* = 7.9 Hz, 1H), 3.16 (s, 3H), 2.33 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.1, 138.3, 138.1, 135.1, 132.0, 127.7, 127.6, 33.06, 20.91, 17.53; **EI-MS***m/z* (relative intensity, %): 163 (100%), 146 (66.2%), 134 (46.6%), 122 (31.6%), 120 (46.9%), 107 (18.4%), 91 (24.9%), 44 (39.3%).

***N*-Methyl-*N*-(m-tolyl)formamide (2i)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 6.97 (m, 2H), 3.30 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, two rotamers) δ 162.3, 142.2, 139. 7, 132.5, 130.1, 129.4, 127.2, 125.6, 123.1, 122.6, 119.5, 32.1, 29.7, 21.4; **EI-MS***m/z* (relative intensity, %): 149 (100%), 120 (75.3%), 108 (35.8%), 91 (34.9%), 80 (28.0%).

***N*-(2-Fluoro-4-methylphenyl)-*N*-methylformamide (2k)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.09 – 7.02 (m, 1H), 7.01 – 6.94 (m, 2H), 3.23 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.9, 157.2 (d, J_{CF} = 249.1 Hz), 139.8 (d, J_{CF} = 7.5 Hz), 127.1 (d, J_{CF} = 1.4 Hz), 125.4 (d, J_{CF} = 3.4 Hz), 117.4, 117.3, 32.8, 21.0; **EI-MS***m/z* (relative intensity, %): 167 (55.1%), 138 (100%), 126 (13.6%), 109 (11.6%), 91 (18.3%).

***N*-Ethyl-*N*-(*p*-tolyl)formamide (2l)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.20 (d, *J* = 7.7 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 3.82 (q, *J* = 7.1 Hz, 2H), 2.36 (s, 3H), 1.14 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, two rotamers) δ 162.0, 138.2, 136.8, 132.5, 130.1, 129.9, 126.0, 125.6, 124.5, 40.1, 20.9, 13.0; **EI-MS***m/z* (relative intensity, %): 163 (93.8%), 148 (9.6%), 135 (32.5%), 120 (100%), 91 (42.7%).

***N*-Ethyl-*N*-phenylformamide (2m)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.41 (t, *J* = 7.3 Hz, 2H), 7.30 (t, *J* = 7.3 Hz, 1H), 7.17 (d, *J* = 7.6 Hz, 2H), 3.86 (q, *J* = 6.9 Hz, 2H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.0, 140.8, 129.6, 126.8, 124.3, 40.1, 13.0; **EI-MS***m/z* (relative intensity, %): 149 (100%), 121 (64.4%), 106 (99.9%), 97 (41.3%), 57 (68.7%).

***N*-Propyl-*N*-(*p*-tolyl)formamide (2n)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (s, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 3.79 – 3.72 (m, 2H), 2.37 (s, 3H), 1.61 – 1.49 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.4, 138.4, 136.8, 130.1, 124.4, 46.6, 20.9, 20.8, 11.2; **EI-MS***m/z* (relative intensity, %): 177 (43.7%), 148 (21.3%), 135 (49.5%), 120 (100%), 91 (28.3%).

***N*-Butyl-*N*-(*p*-tolyl)formamide (2o)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.2 Hz, 2H), 3.79 (t, *J* = 7.2 Hz, 2H), 2.36 (s, 3H), 1.55 – 1.39 (m, 2H), 1.35 – 1.24 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.3, 138.5, 136.8, 130.1, 124.4, 44.8, 29.6, 20.9, 20.0, 13.7; **EI-MS***m/z* (relative intensity, %): 191 (39.5%), 148 (17.1%), 135 (36.9%), 120 (100%), 91 (23.9%).

***N*-Hexyl-*N*-(*p*-tolyl)formamide (2p)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.04 (d, *J* = 8.3 Hz, 2H), 3.79 – 3.74 (t, *J* = 7.2 Hz, 2H), 2.36 (s, 3H), 1.54 – 1.46 (m, 2H), 1.31 – 1.19 (m, 6H), 0.84 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.3, 138.5, 136.7, 130.1, 124.4, 45.0, 31.4, 27.5, 26.4, 22.5, 20.9, 13.9; **EI-MS***m/z* (relative intensity, %): 219 (38.4%), 148 (17.2%), 135 (47.0%), 120 (100%), 91 (23.6%).

***N*-Isobutyl-*N*-(*p*-tolyl)formamide (2r)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.20 (d, *J* = 7.7 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 3.63 (d, *J* = 7.5 Hz, 2H), 2.35 (s, 3H), 1.81 (m, 1H), 0.87 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.8, 138.6, 136.7, 130.2, 124.3, 51.7, 26.6, 20.9, 20.0; **EI-MS***m/z* (relative intensity, %): 191 (20.2%), 148 (29.6%), 135 (64.6%), 120 (100%), 91 (23.0%).

***N*-(Cyclopropylmethyl)-*N*-(*p*-tolyl)formamide (2t)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.21 (d, *J* = 8.3 Hz, 2H), 7.11 (d, *J* = 8.2 Hz, 2H), 3.65 (d, *J* = 7.1 Hz, 2H), 2.38 (s, 3H), 1.07 – 0.85 (m, 1H), 0.51 – 0.37 (m, 2H), 0.27 – 0.15 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.4, 138.7, 137.0, 130.1, 125.1, 49.6, 20.9, 9.8, 3.8; **EI-MS***m/z* (relative intensity, %): 189 (26.9%), 160 (21.8%), 120 (100%), 107 (84.0%), 91 (34.0%), 55 (32.9%).

***N*-(cyclohexylmethyl)-*N*-(*p*-tolyl)formamide (2u)**

^{1}H NMR (600 MHz, cdcl$_3$) δ 8.35 (s, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.05 (d, *J* = 8.3 Hz, 2H), 3.65 (d, *J* = 7.4 Hz, 2H), 2.36 (s, 3H), 1.74 – 1.57 (m, 6H), 1.57 – 1.48 (m, 1H), 1.17 – 1.06 (m, 2H), 0.99 – 0.91 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.8, 138.9, 136.6, 130.1, 124.2, 50.7, 35.9, 30.7, 26.3, 25.7, 20.9; **EI-MS***m/z* (relative intensity, %): 231 (24.3%), 148 (15.8%), 135 (100%), 120 (88.2%), 107 (14.4%), 91 (19.1%).

***N*-Benzyl-*N*-(*p*-tolyl)formamide (2v)**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (s, 1H), 7.29 – 7.25 (m, 2H), 7.22 (t, *J* = 7.4 Hz, 3H), 7.13 (d, *J* =

7.7 Hz, 2H), 6.97 (d, *J* = 7.9 Hz, 2H), 4.96 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.4, 138.4, 136.9, 136.7, 130.1, 128.5, 127.9, 127.4, 124.3, 49.0, 20.9; **EI-MS** *m/z* (relative intensity, %): 225 (91.3%), 196 (12.1%), 135 (4.0%), 120 (5.9%), 91 (100%).

***N*-Phenethyl-*N*-(*p*-tolyl)formamide (2w)**

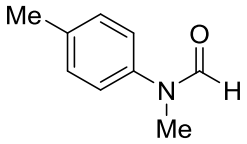
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.27 (t, *J* = 7.4 Hz, 2H), 7.19 (m, 5H), 6.98 (d, *J* = 8.0 Hz, 2H), 4.02 (t, *J* = 7.8 Hz, 2H), 2.86 (t, *J* = 7.7 Hz, 2H), 2.37 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.3, 138.4, 138.3, 136.9, 130.2, 128.8, 128.5, 126.5, 124.4, 46.8, 33.8, 20.9; **EI-MS** *m/z* (relative intensity, %): 239 (7.6%), 148 (26.1%), 135 (100%), 120 (93.5%), 91 (33.1%).

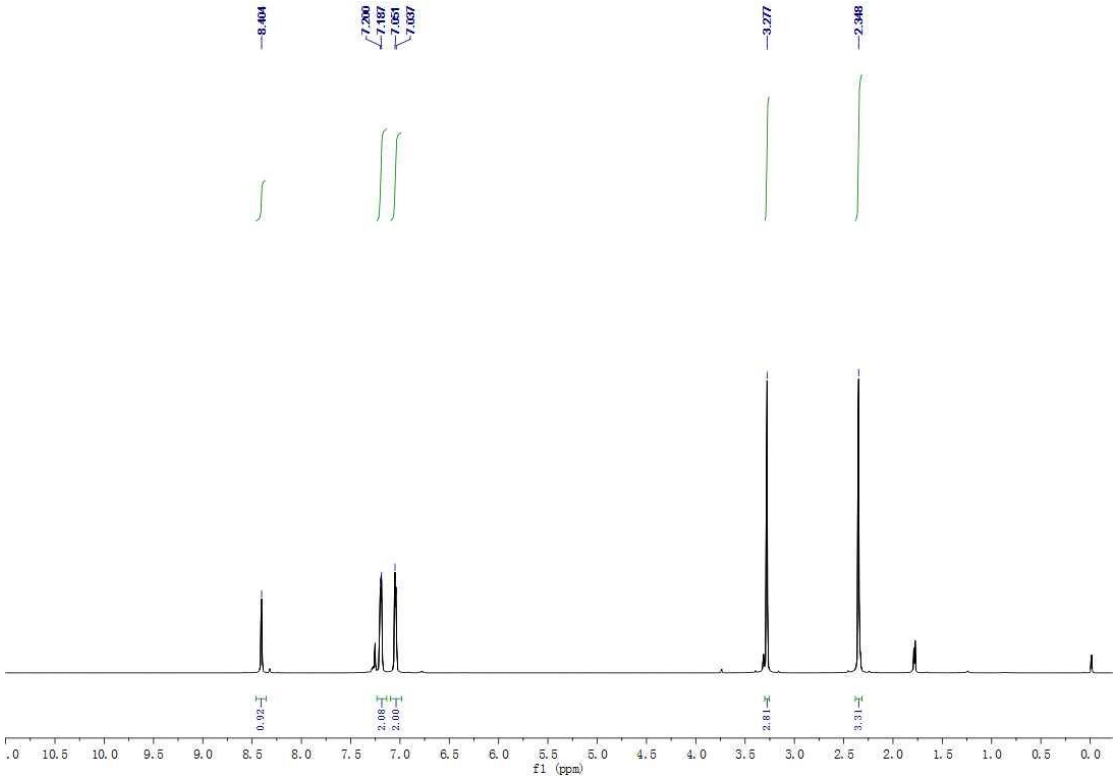
3,4-Dihydroquinoline-1(2*H*)-carbaldehyde (2x)

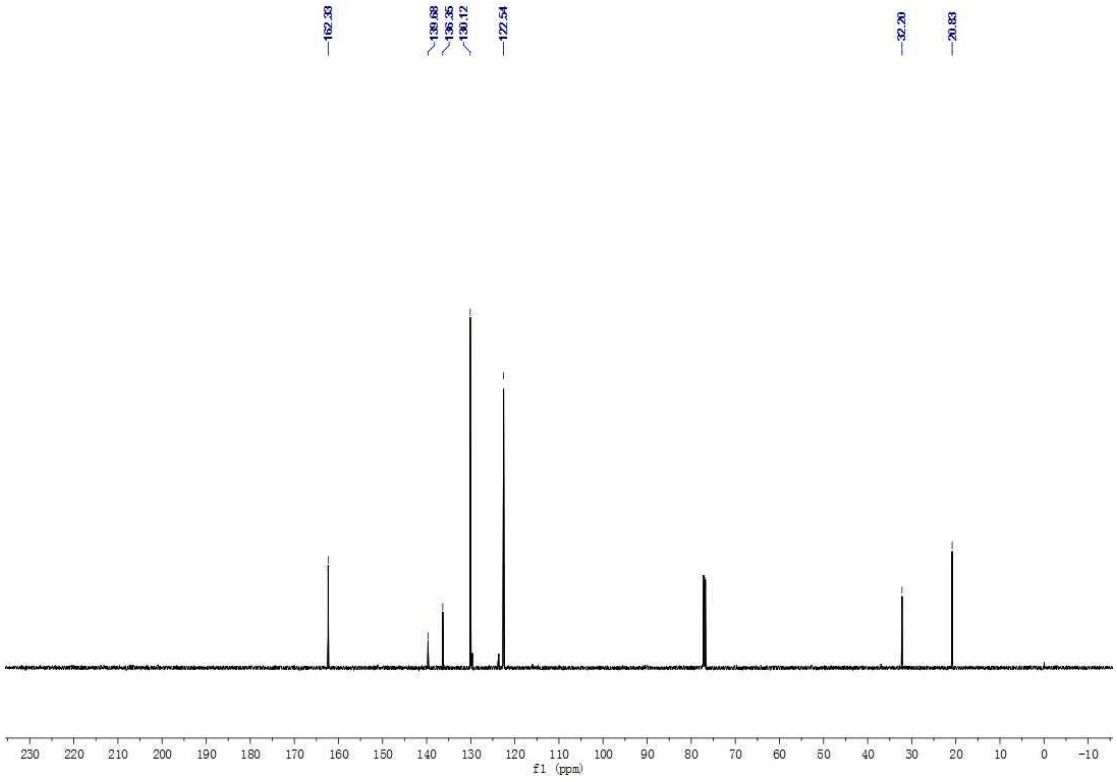
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.79 (s, 1H), 7.23 – 7.13 (m, 3H), 7.13 – 7.08 (m, 1H), 3.82 – 3.80 (t, *J* = 6.0 Hz,2H), 2.81 (t, *J* = 6.4 Hz, 2H), 1.99 – 1.93 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 161.1, 137.3, 129.6, 128.9, 127.1, 124.6, 117.0, 40.3, 27.1, 22.3; **EI-MS** *m/z* (relative intensity, %): 161 (82.9%), 132 (100%), 117 (25.0%), 91 (8.4%), 77 (12.6%).

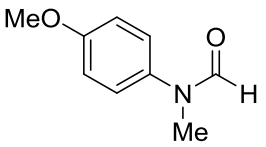
^{1}H and ^{13}C spectra

2a:

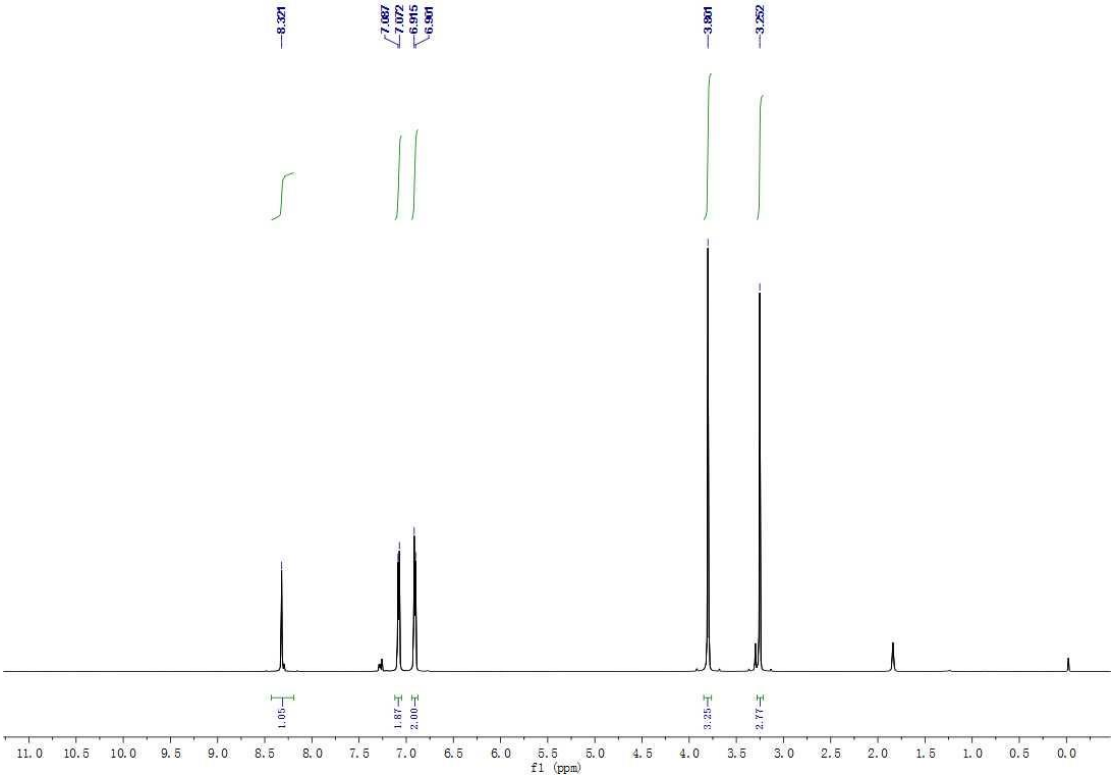


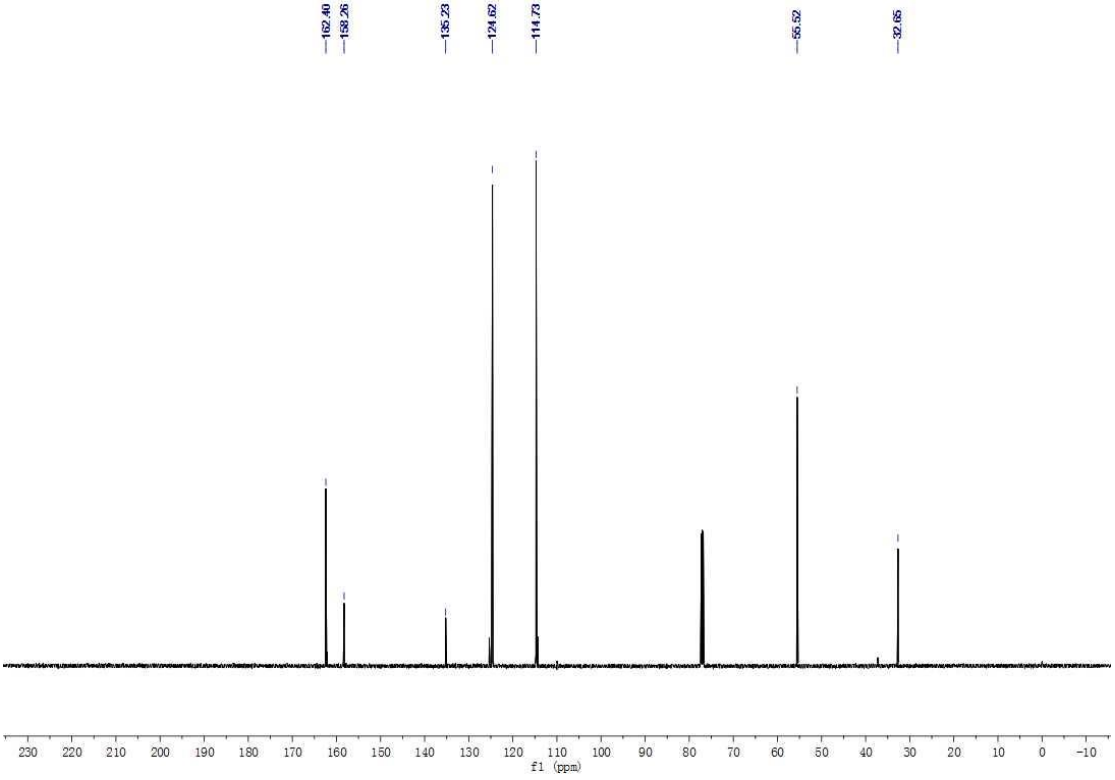




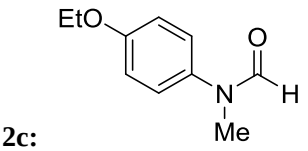

MeO
O
N
H
Me

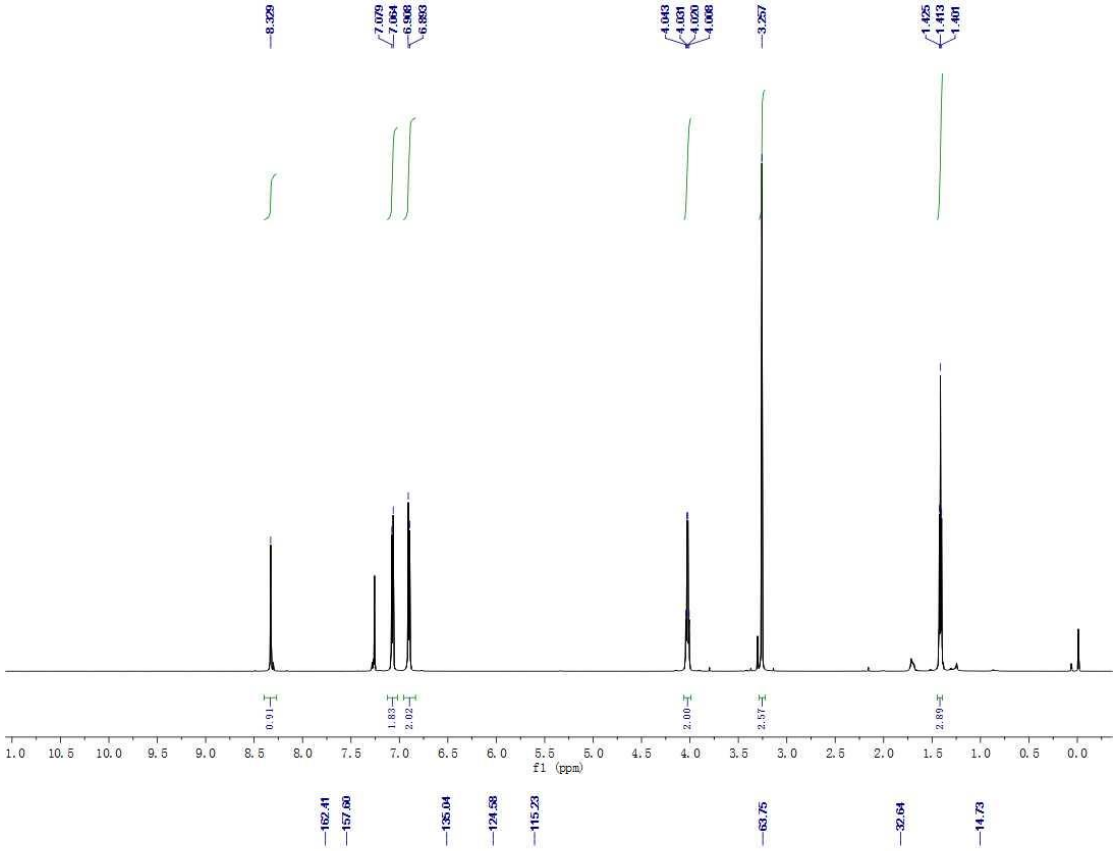
2b:

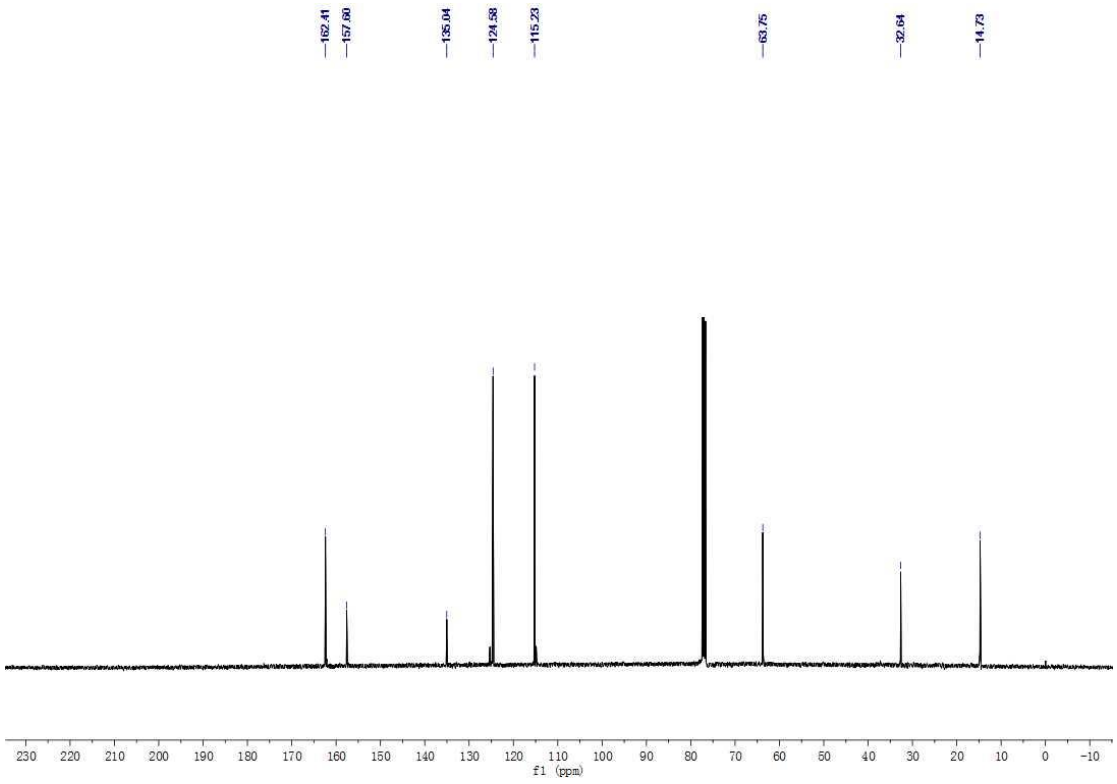

8.321
7.087
7.072
6.915
6.901
3.801
3.252
1.05
1.87
2.00
3.25
2.77
f1 (ppm)


162.40
158.26
135.23
124.62
114.73
55.52
32.65
f1 (ppm)

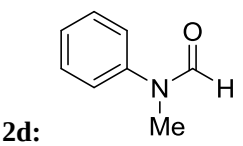
2c:

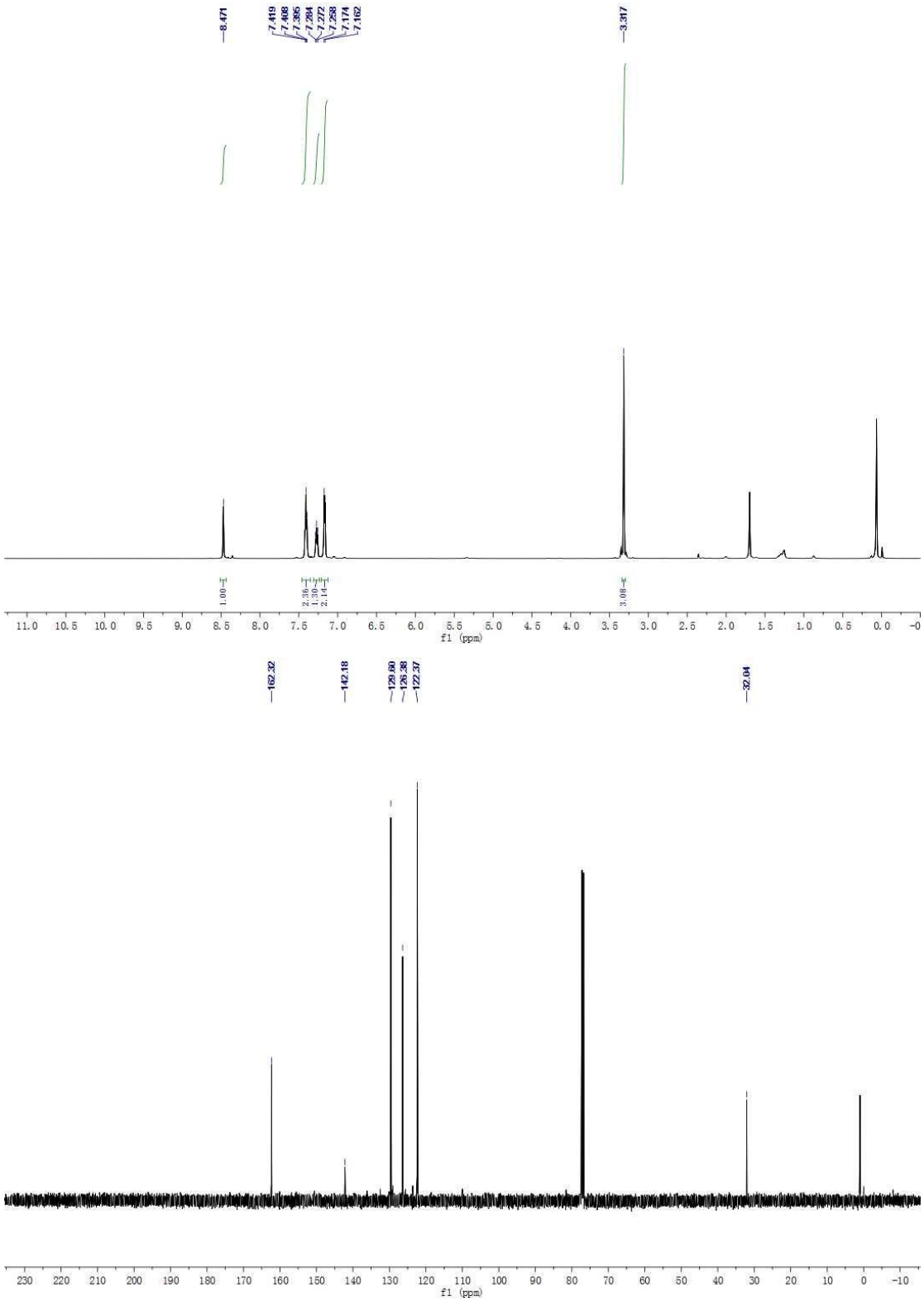

EtO
O
N
H
Me

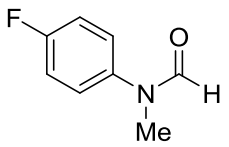

8.329
7.079
7.064
6.908
6.883
4.043
4.031
4.020
4.008
3.257
1.425
1.413
1.401
0.91
1.83
2.02
2.00
2.57
2.89
f1 (ppm)


162.41
157.60
135.04
124.58
115.23
63.75
32.64
14.73
f1 (ppm)

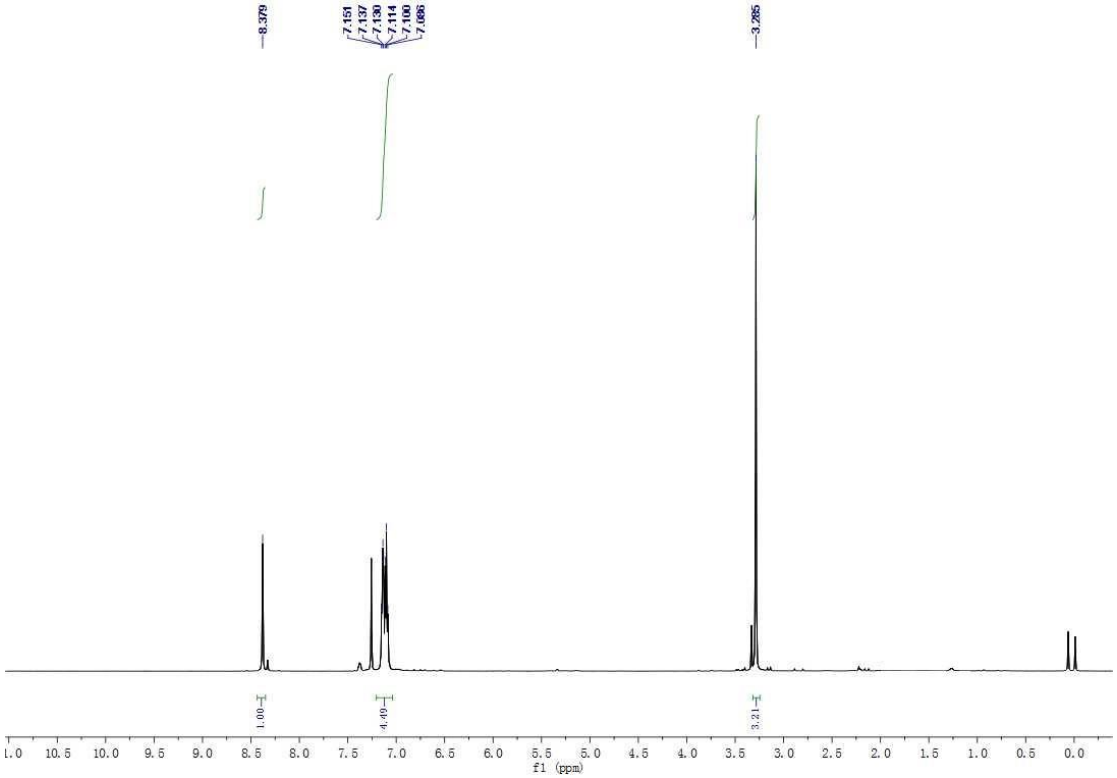
2d:

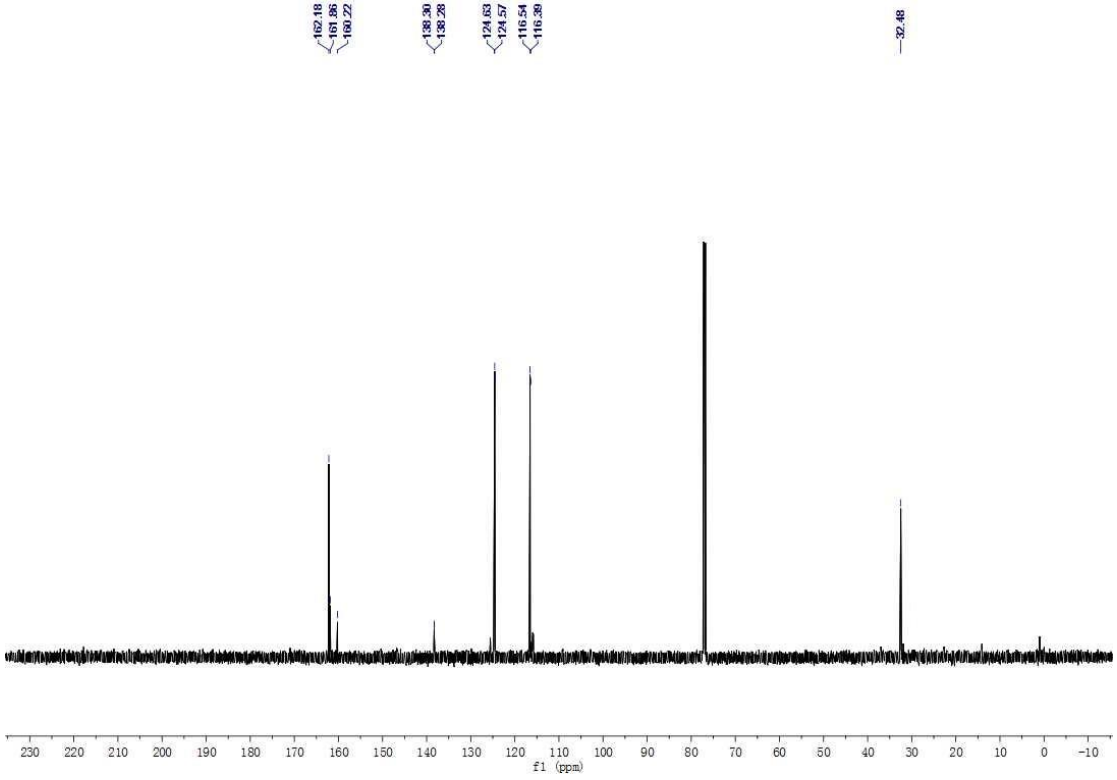

O
N
H
Me

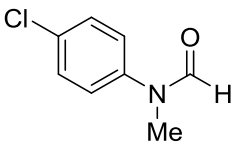

8.471
7.419
7.408
7.395
7.294
7.272
7.258
7.174
7.162
3.317
1.00
2.36
1.39
2.14
3.08
f1 (ppm)
162.32
142.18
129.60
126.38
122.37
32.04
f1 (ppm)


F
O
N
H
Me

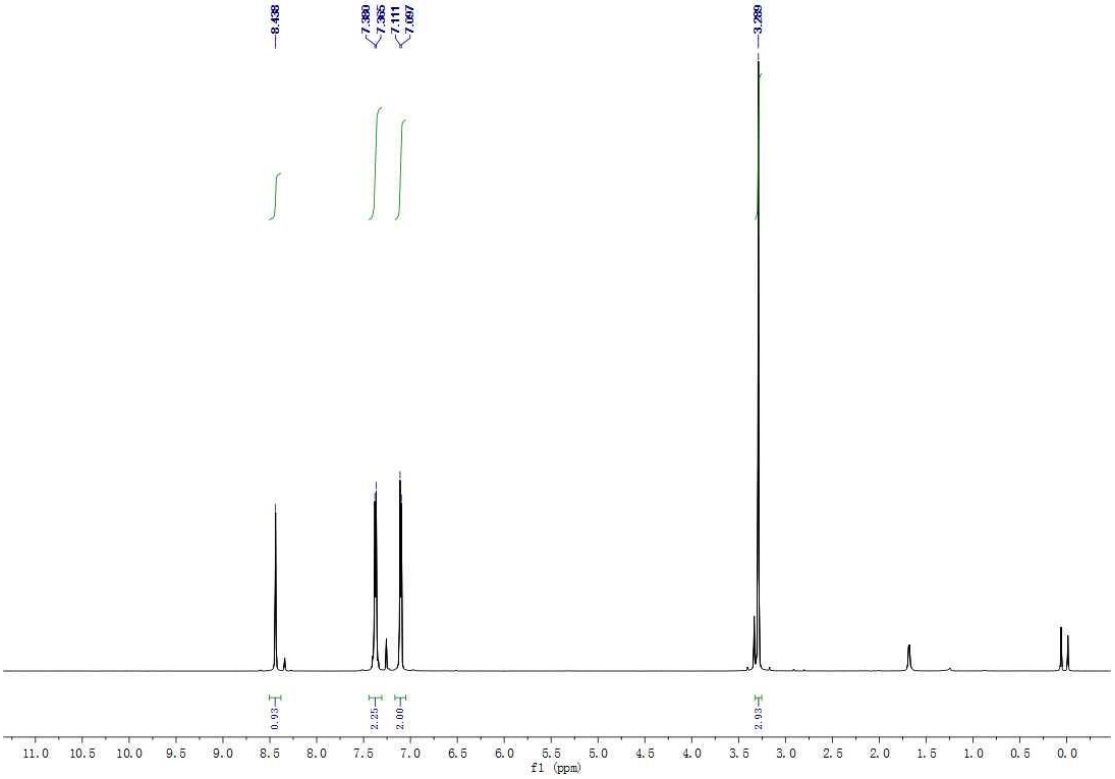
2e:

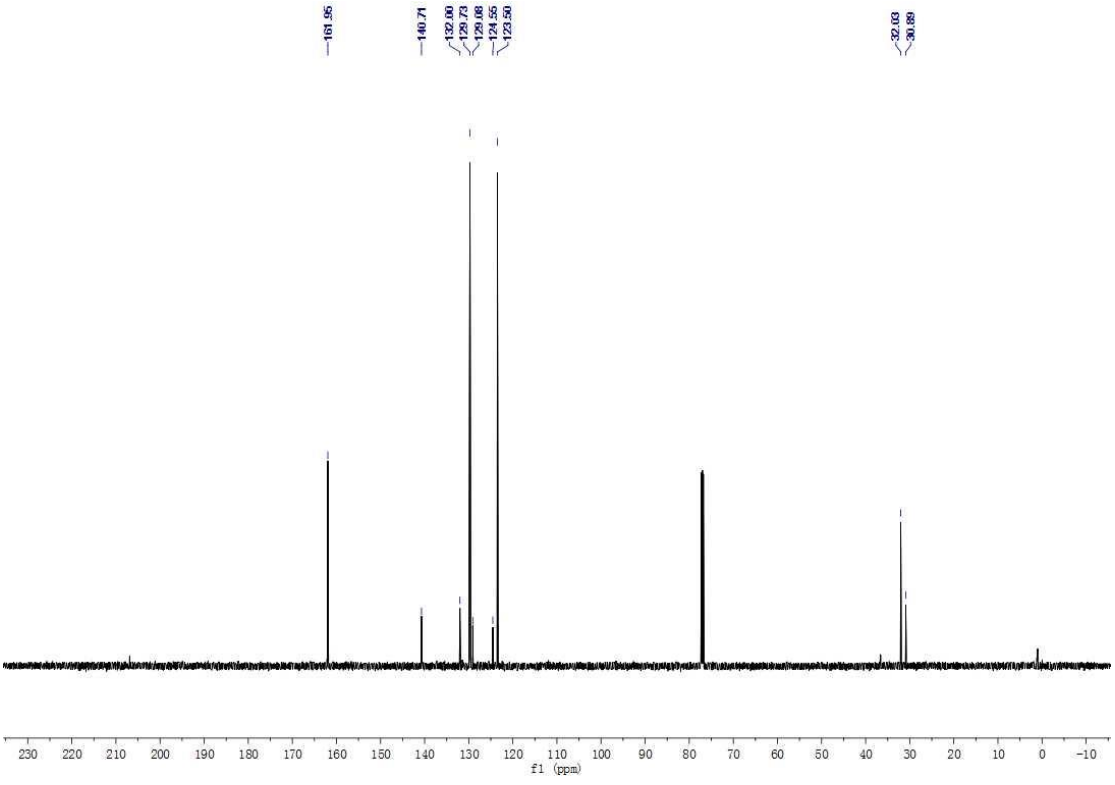

8.379
7.151
7.137
7.130
7.114
7.100
7.086
3.285
1.00
4.49
3.21
f1 (ppm)

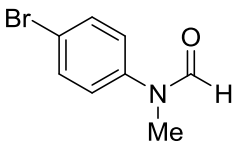

162.18
161.86
160.22
138.30
138.28
124.63
124.57
116.54
116.39
32.48
f1 (ppm)


Cl
O
N
H
Me

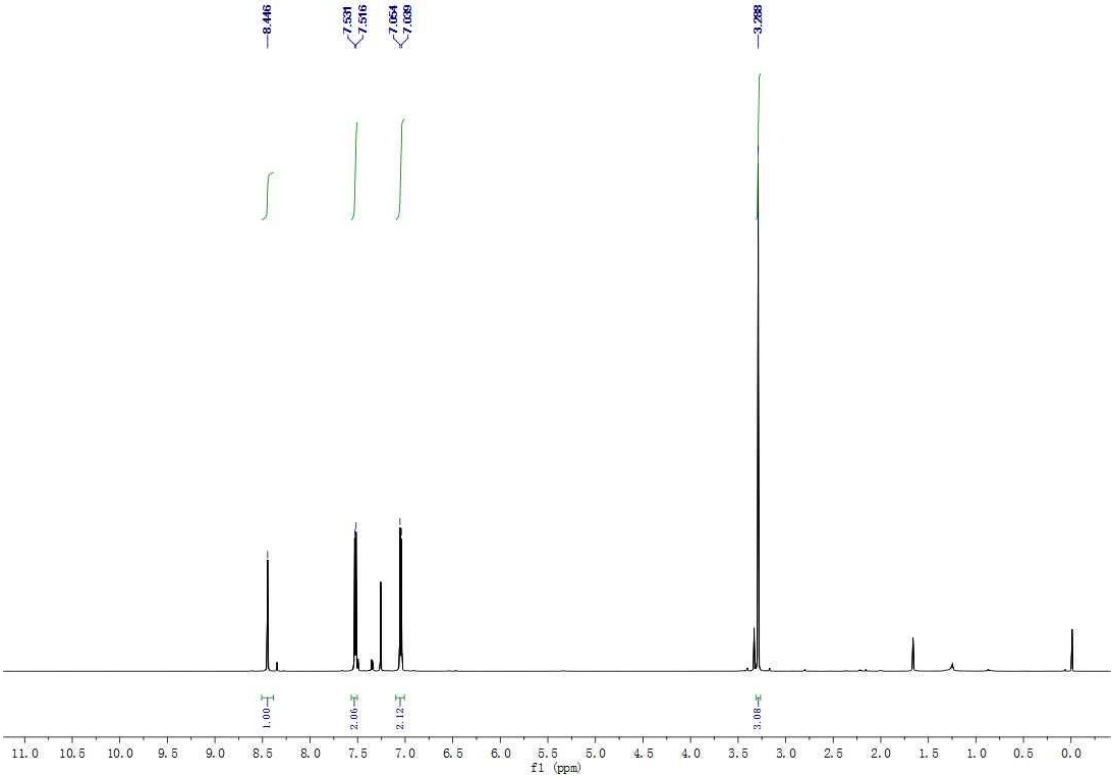
2f:

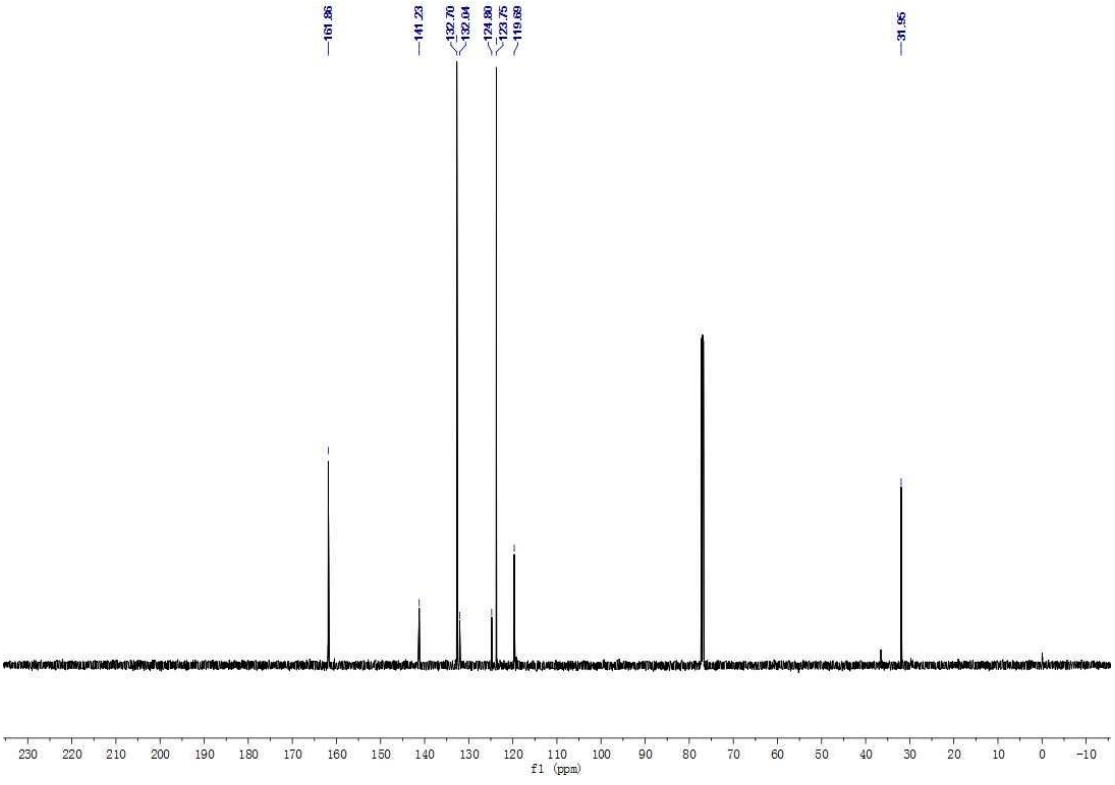

8.438
7.380
7.365
7.111
7.097
3.289
0.93
2.25
2.09
2.93
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.95
140.71
132.00
129.73
129.08
124.55
123.50
32.03
30.89
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

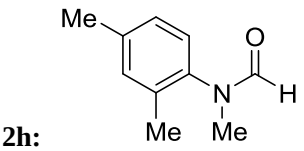

Br
O
N
H
Me

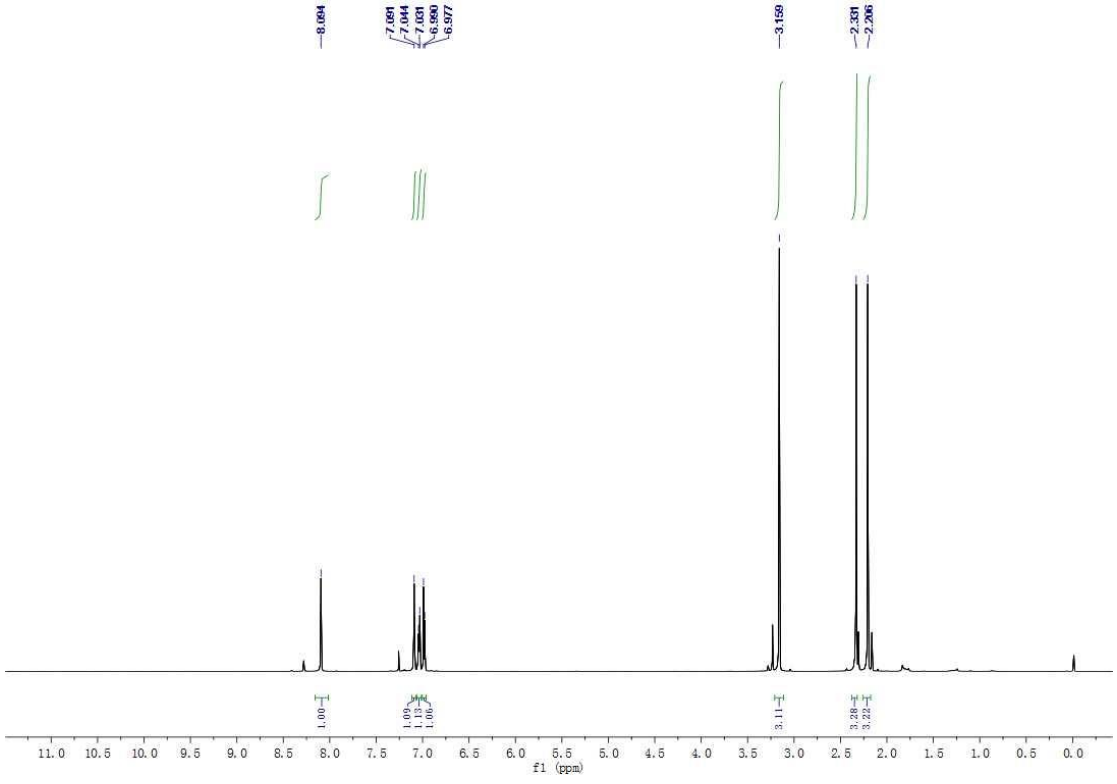
2g:

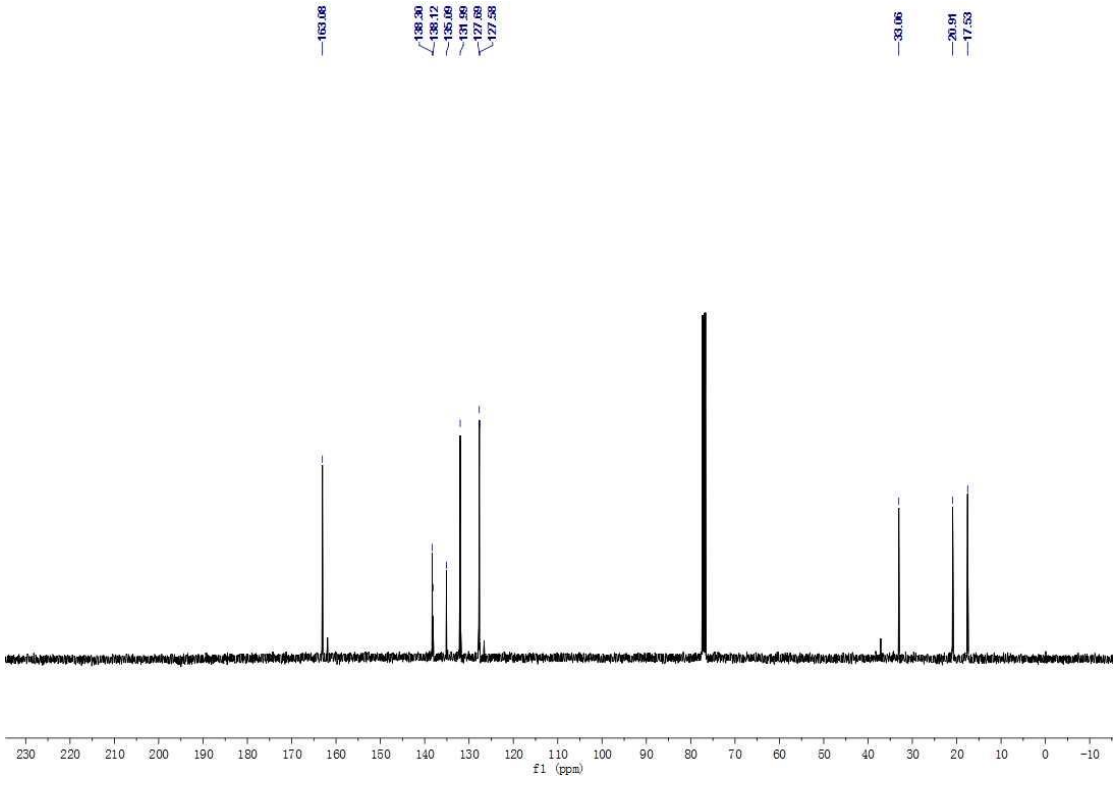

8.446
7.531
7.516
7.054
7.039
3.288
1.00
2.06
2.12
3.08
f1 (ppm)


161.86
141.23
132.70
132.04
124.90
123.75
119.69
31.95
f1 (ppm)

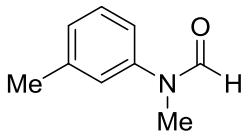
2h:

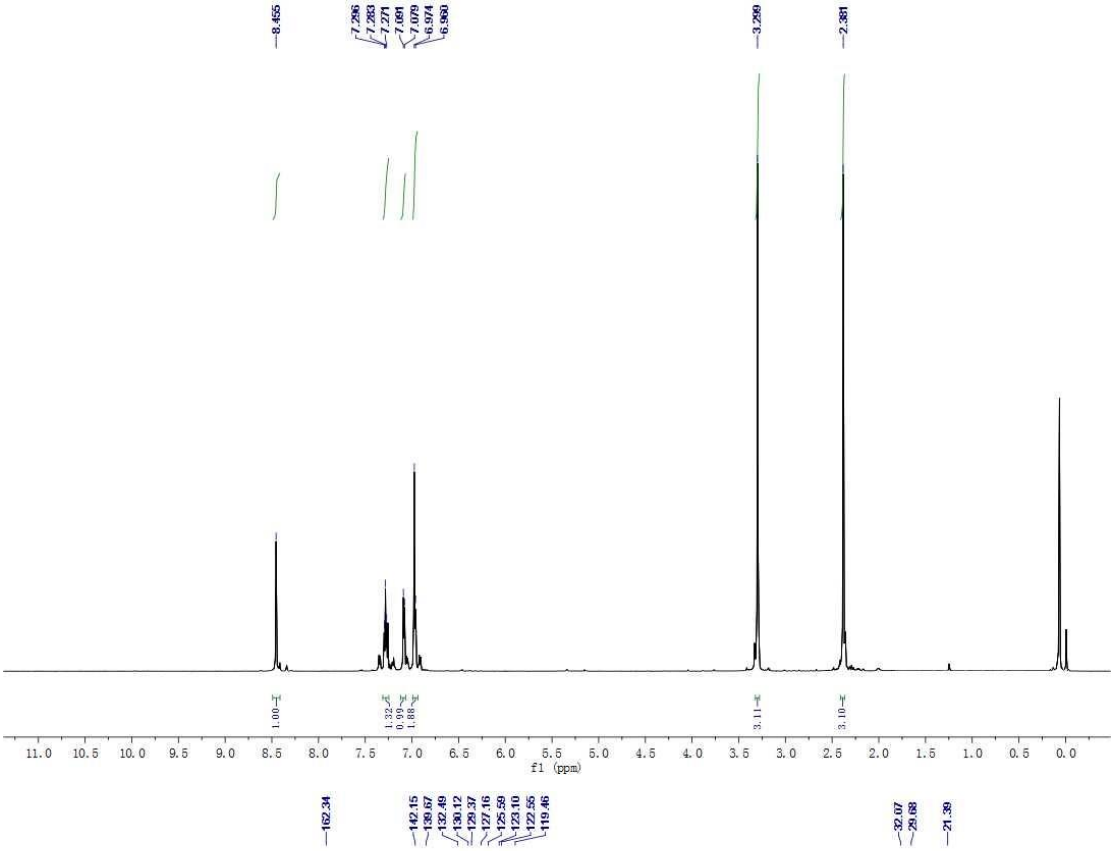

Me
O
N
H
Me
Me


8.004
7.061
7.044
7.031
6.990
6.977
3.159
2.331
2.206
1.00
1.09
1.11
1.00
3.11
3.28
3.22
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

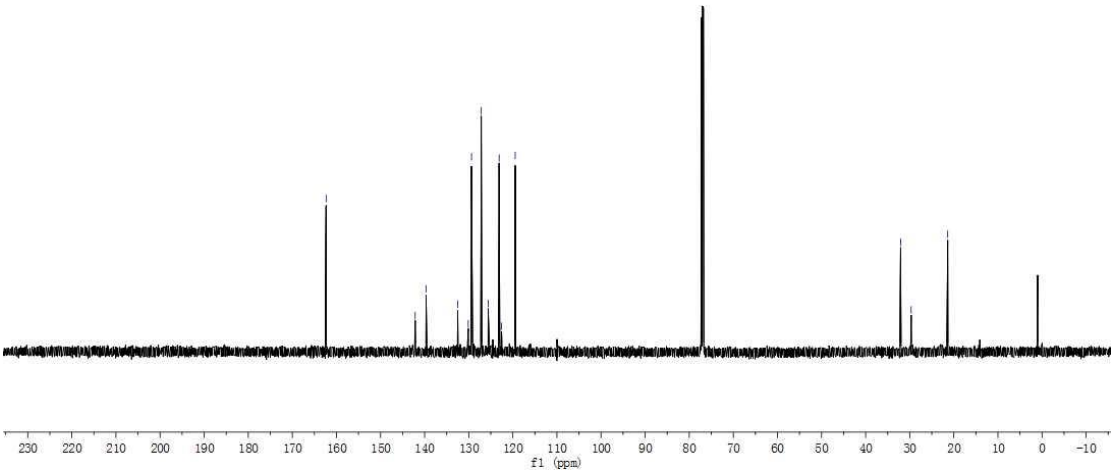

163.08
138.30
138.12
135.89
131.99
127.69
127.58
33.06
20.91
17.53
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2i:

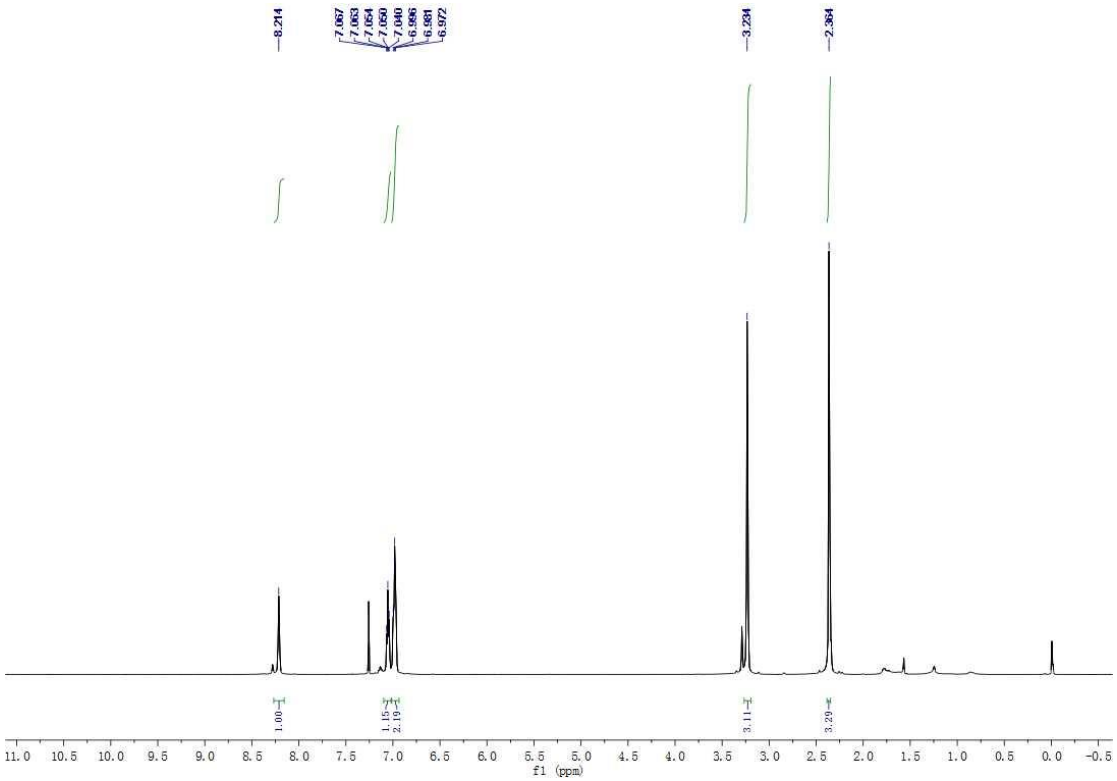

Me
N
O
H
Me

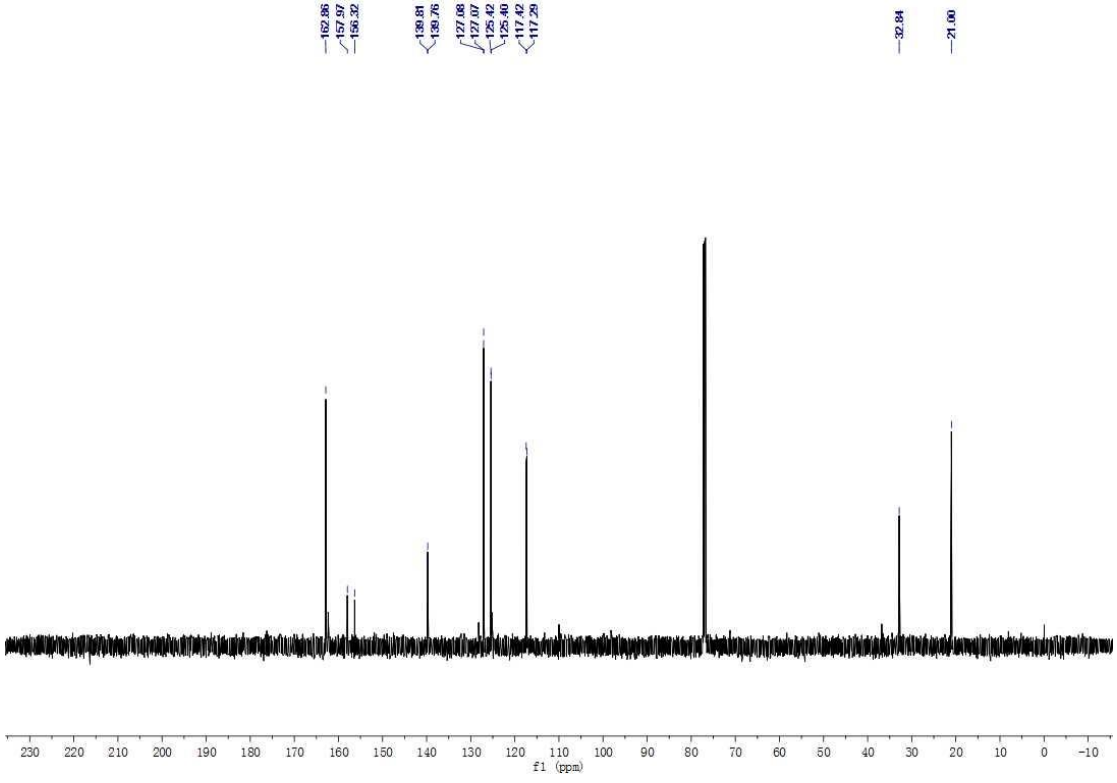

8.455
7.296
7.283
7.271
7.091
7.079
6.974
6.960
3.299
2.381
1.00
1.33
0.99
1.88
3.11
3.10
f1 (ppm)

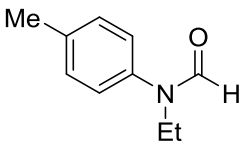
162.34
142.15
139.67
132.49
130.12
129.37
127.16
125.59
123.10
122.55
119.46
32.07
29.68
21.39


f1 (ppm)

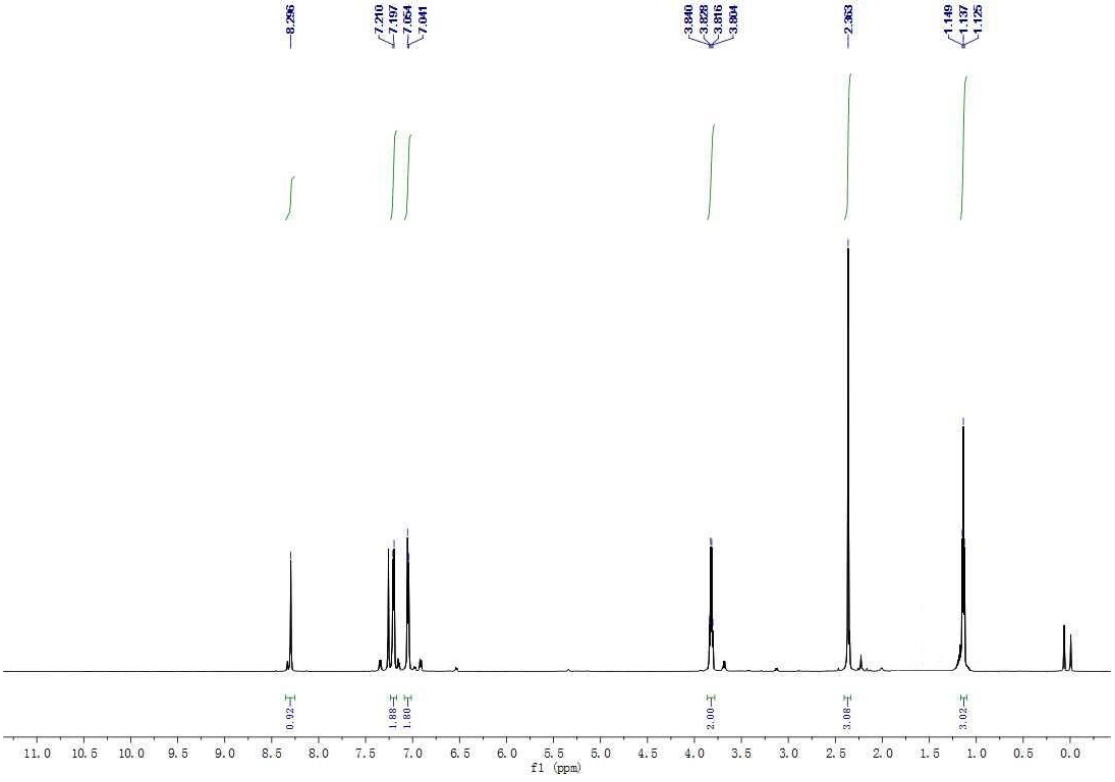
2k:

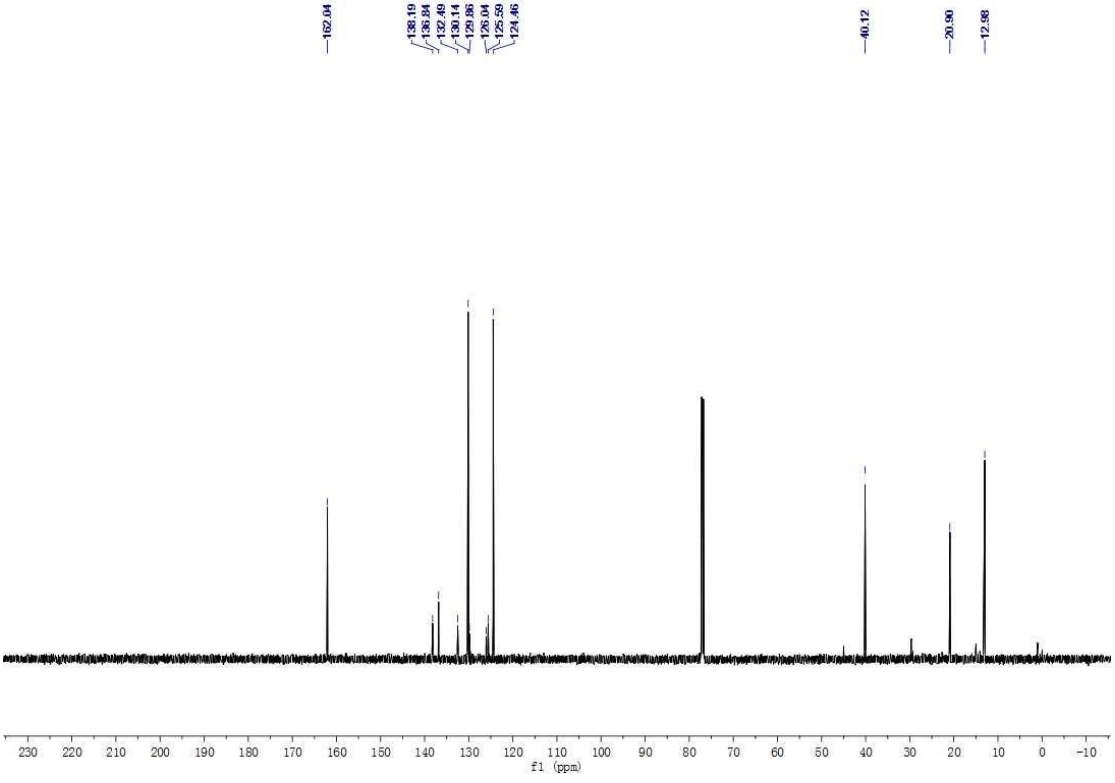

8.214
7.067
7.063
7.054
7.050
7.040
6.996
6.981
6.972
3.234
2.364
1.00
1.15
2.19
3.11
3.29
f1 (ppm)


162.86
157.97
156.32
140.81
139.76
127.08
127.07
125.42
125.40
117.42
117.29
32.84
21.00
f1 (ppm)

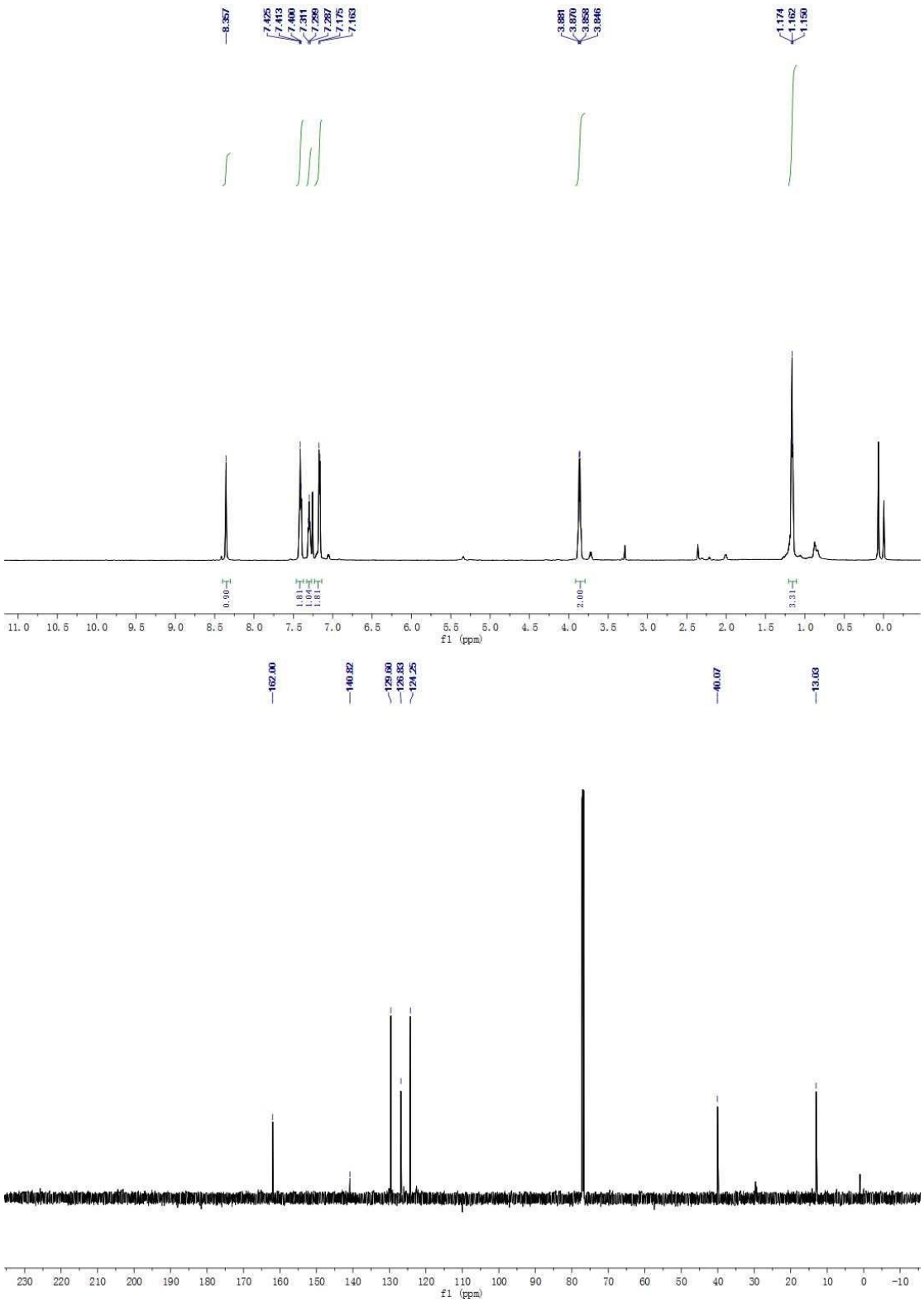

Me
O
N
H
Et

2l:

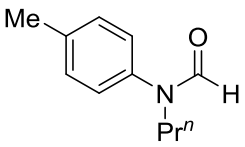

8.296
7.210
7.197
7.054
7.041
3.840
3.828
3.816
3.804
2.363
1.149
1.137
1.125
0.92
1.88
1.80
2.00
3.08
3.02
f1 (ppm)

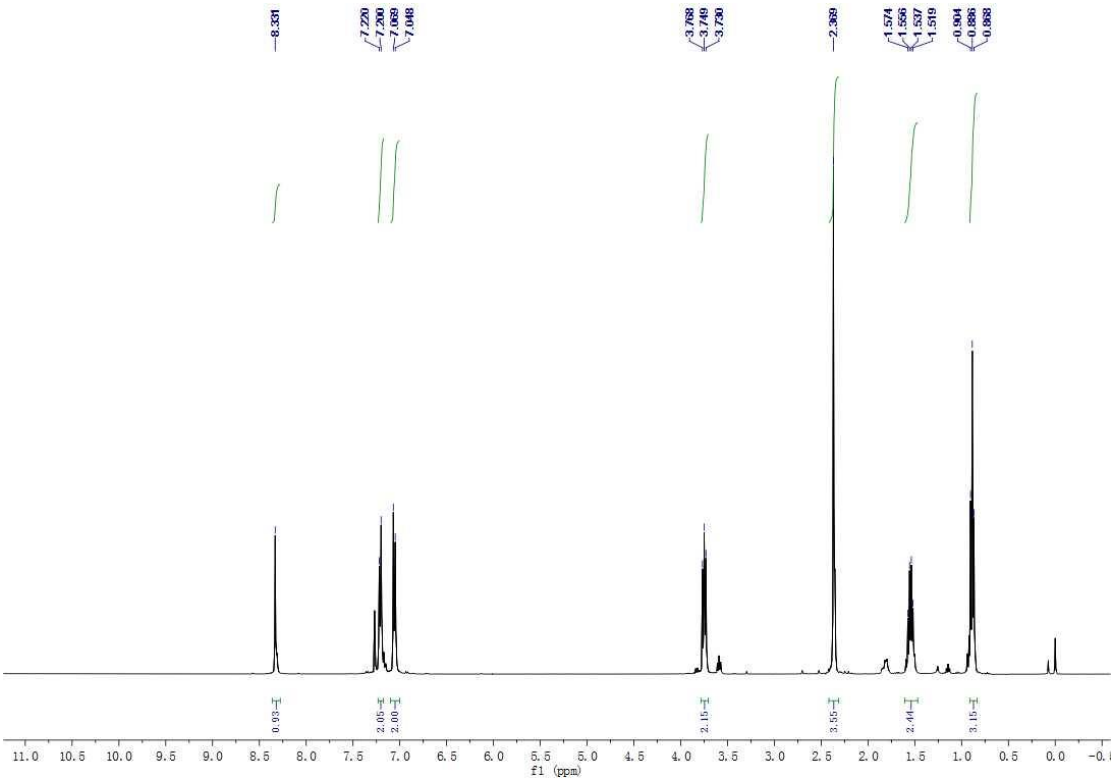

162.04
138.19
136.84
132.49
130.14
129.86
126.04
125.59
124.46
40.12
20.90
12.98
f1 (ppm)

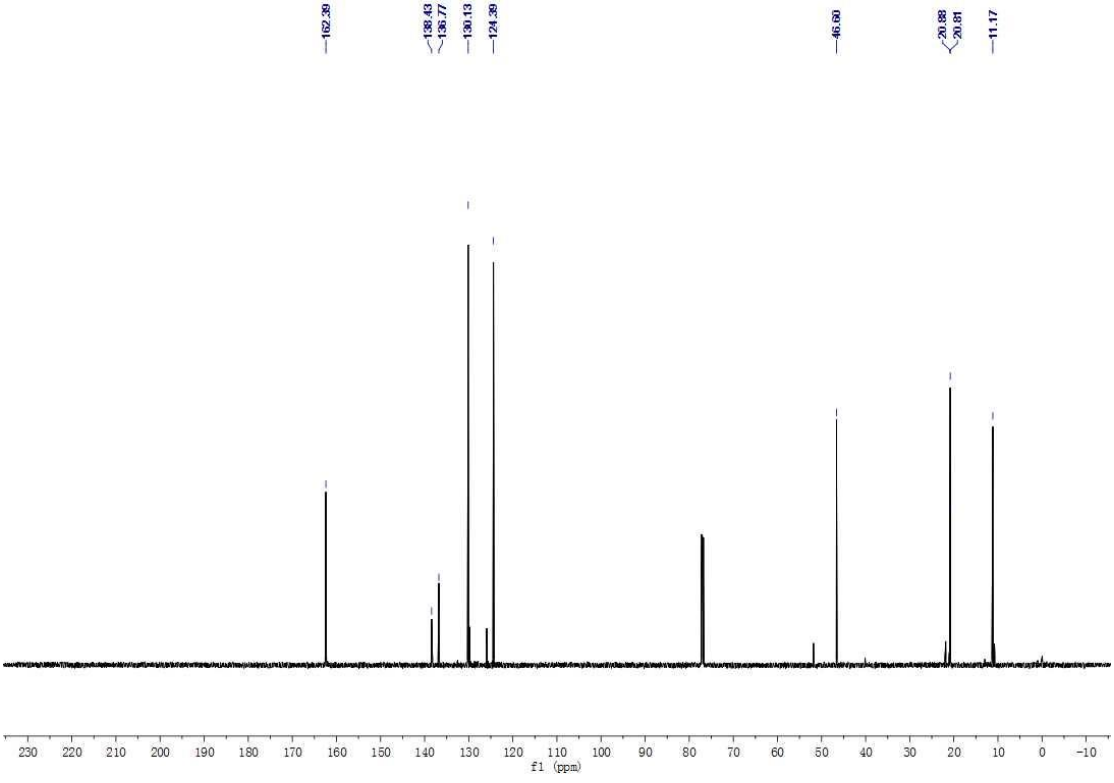
2m:


8.357
7.425
7.413
7.400
7.311
7.299
7.287
7.175
7.163
3.881
3.870
3.858
3.846
1.174
1.162
1.150
0.99
1.81
1.04
1.81
2.00
3.31
f1 (ppm)
162.00
140.82
129.60
126.83
124.25
40.07
13.03
f1 (ppm)

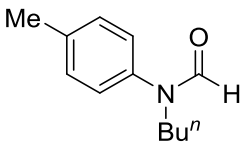
2n:

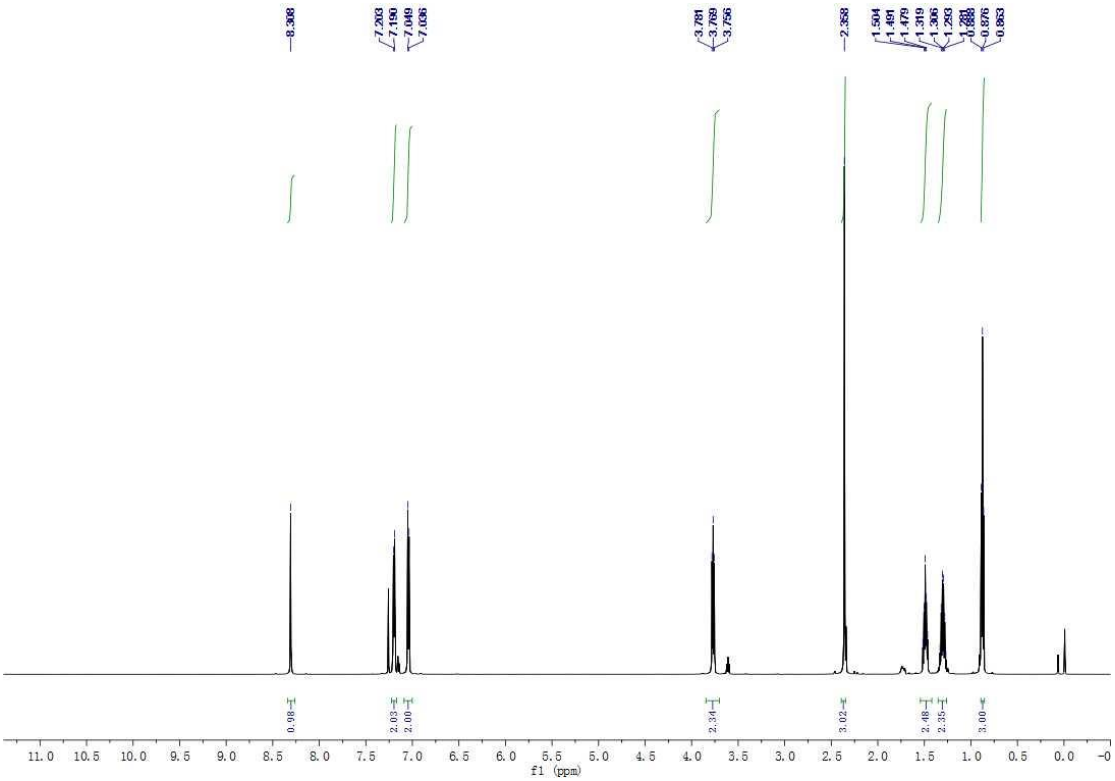

Me
O
N
H
Pr^n

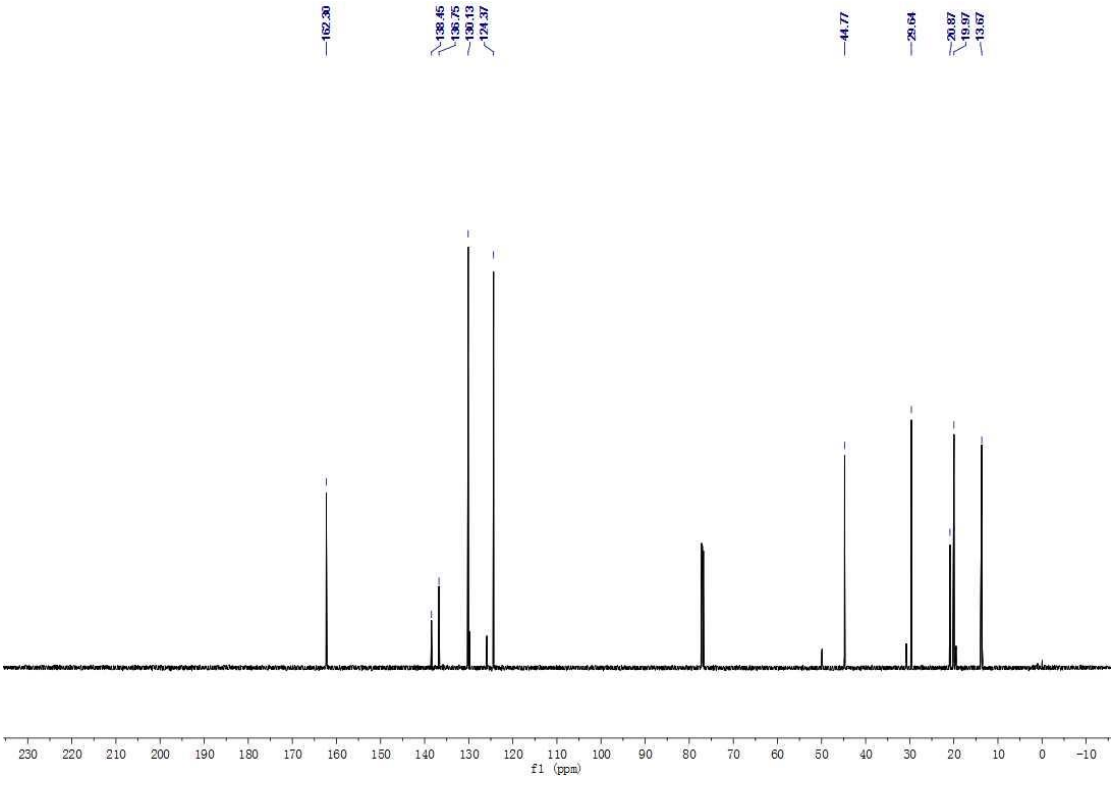

8.331
7.220
7.200
7.069
7.048
3.768
3.749
3.730
2.369
1.574
1.556
1.537
1.519
0.904
0.886
0.868
0.93
2.05
2.09
2.15
3.55
2.44
3.15
f1 (ppm)


162.39
138.43
136.77
130.13
124.39
46.60
20.88
20.81
11.17
f1 (ppm)

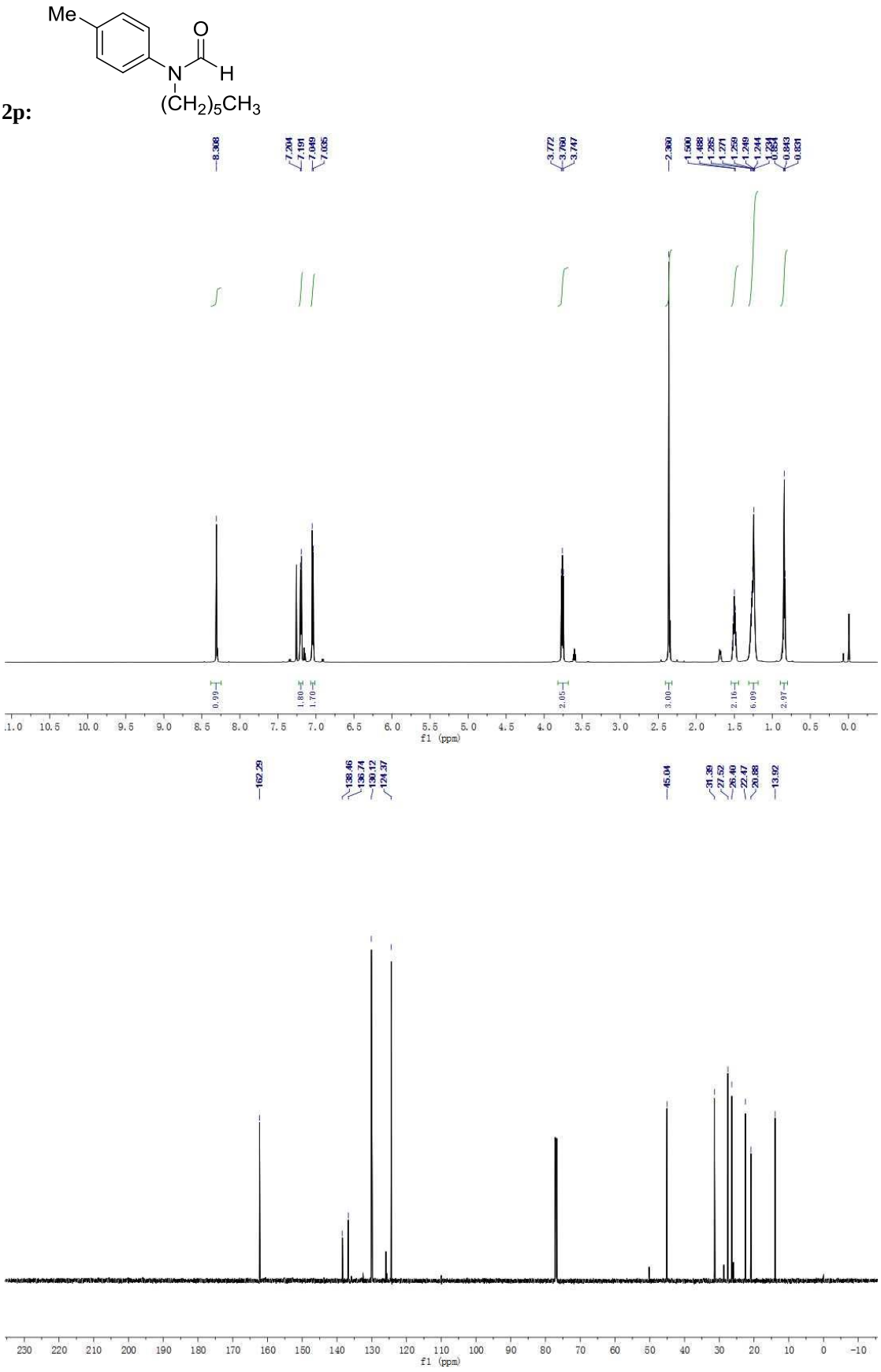
2o:


Me
O
N
H
Bu^n

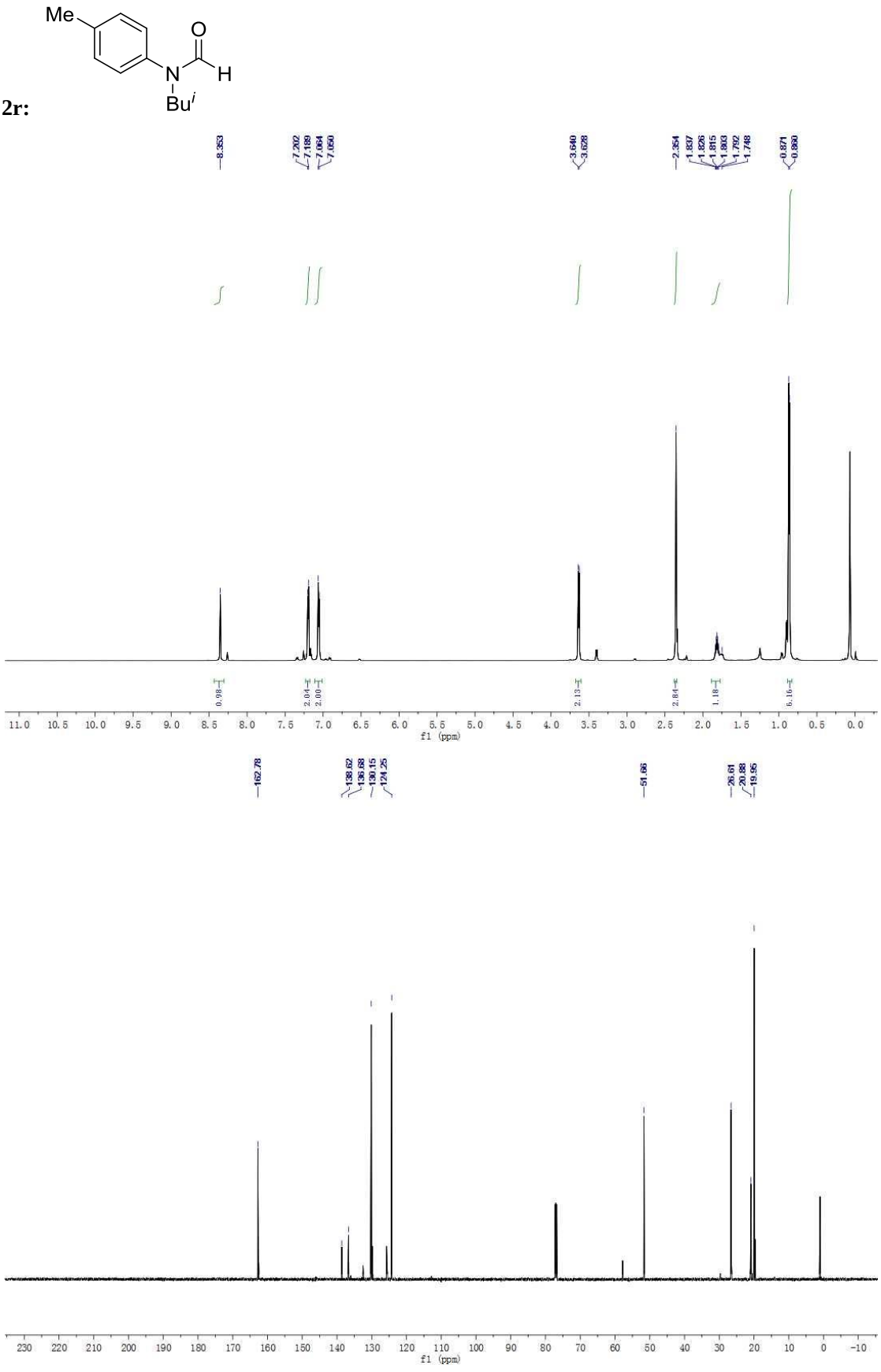

8.308
7.203
7.190
7.049
7.036
3.781
3.769
3.756
2.358
1.504
1.491
1.479
1.319
1.306
1.293
0.876
0.863
0.98
2.03
2.00
2.34
3.02
2.48
2.35
3.00
f1 (ppm)

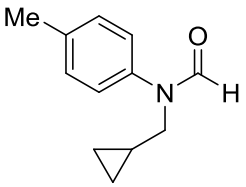

162.30
138.45
136.75
130.13
124.37
44.77
29.64
20.87
19.97
13.67
f1 (ppm)

2p:

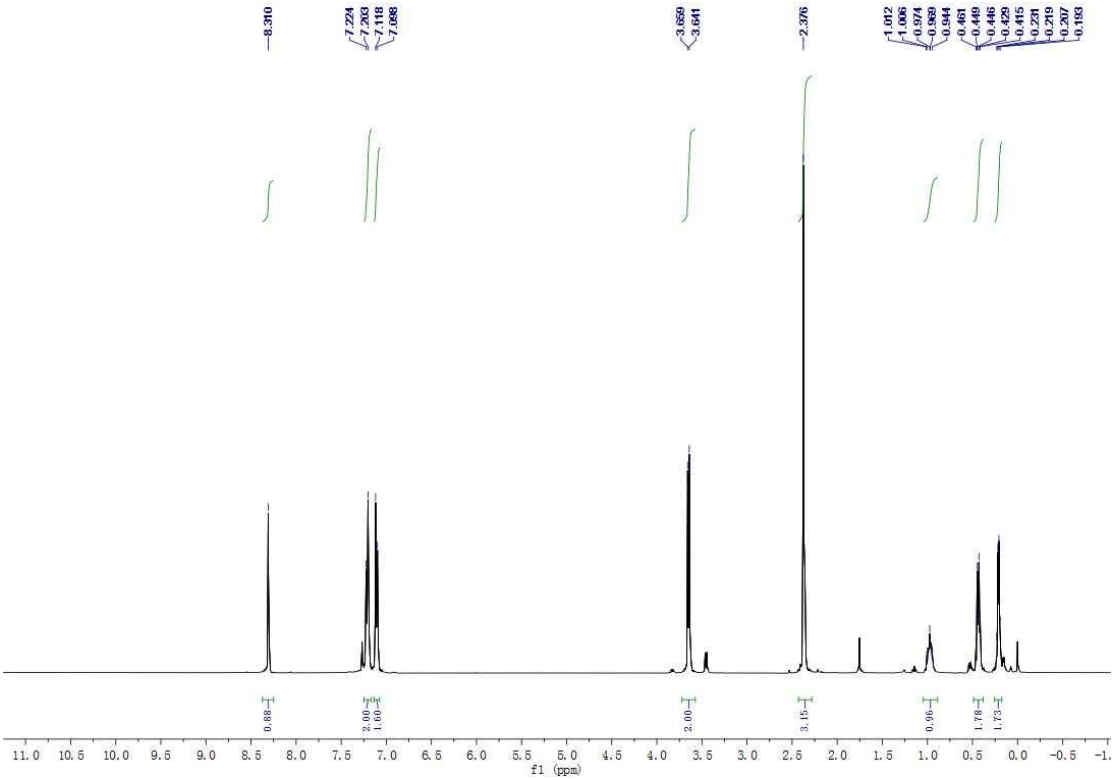

8.308
7.204
7.191
7.049
7.035
3.772
3.760
3.747
2.360
1.500
1.488
1.285
1.271
1.259
1.249
1.244
0.854
0.843
0.831
0.99
1.80
1.70
2.05
3.00
2.16
6.09
2.97
f1 (ppm)
162.29
138.46
136.74
130.12
124.37
45.04
31.39
27.52
26.40
22.47
20.88
13.92
f1 (ppm)

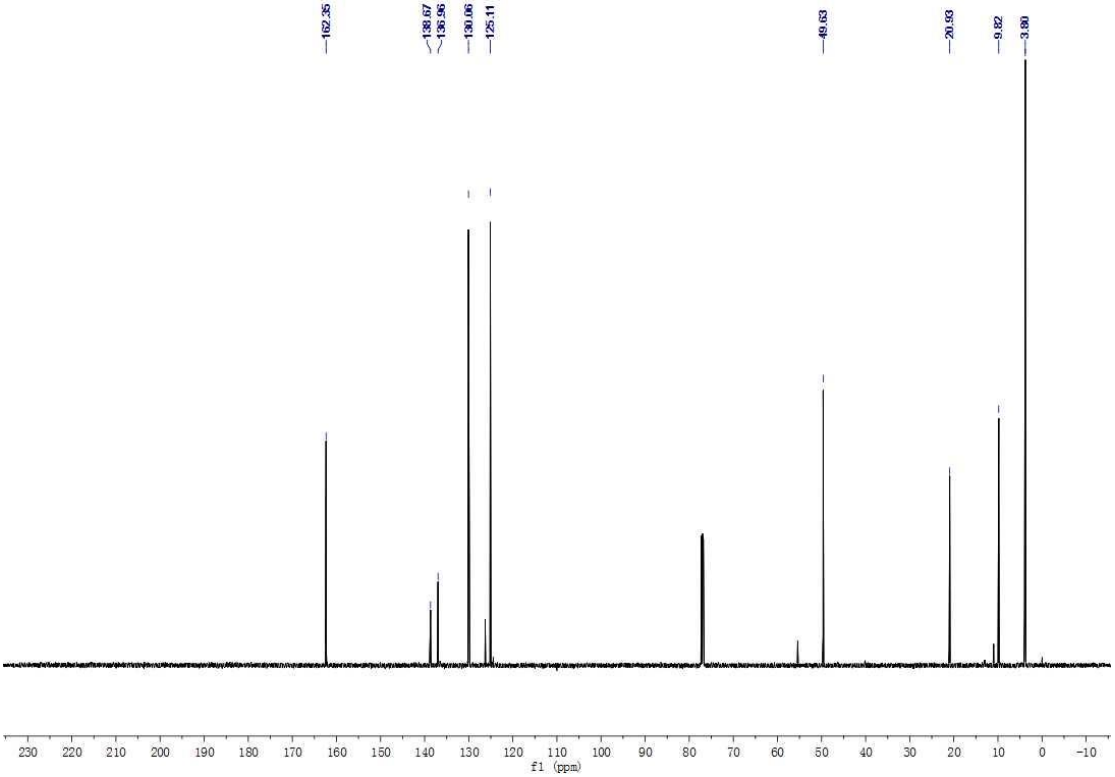
2r:

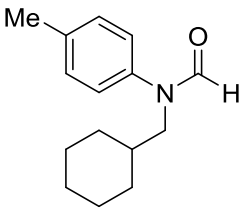

8.363
7.202
7.189
7.064
7.050
3.640
3.628
2.364
1.837
1.826
1.815
1.803
1.792
1.748
0.871
0.860
0.98
2.04
2.00
2.13
2.84
1.18
6.16
f1 (ppm)
162.78
138.62
136.68
130.15
124.25
51.66
26.61
20.88
19.95
f1 (ppm)


Me
O
N
H

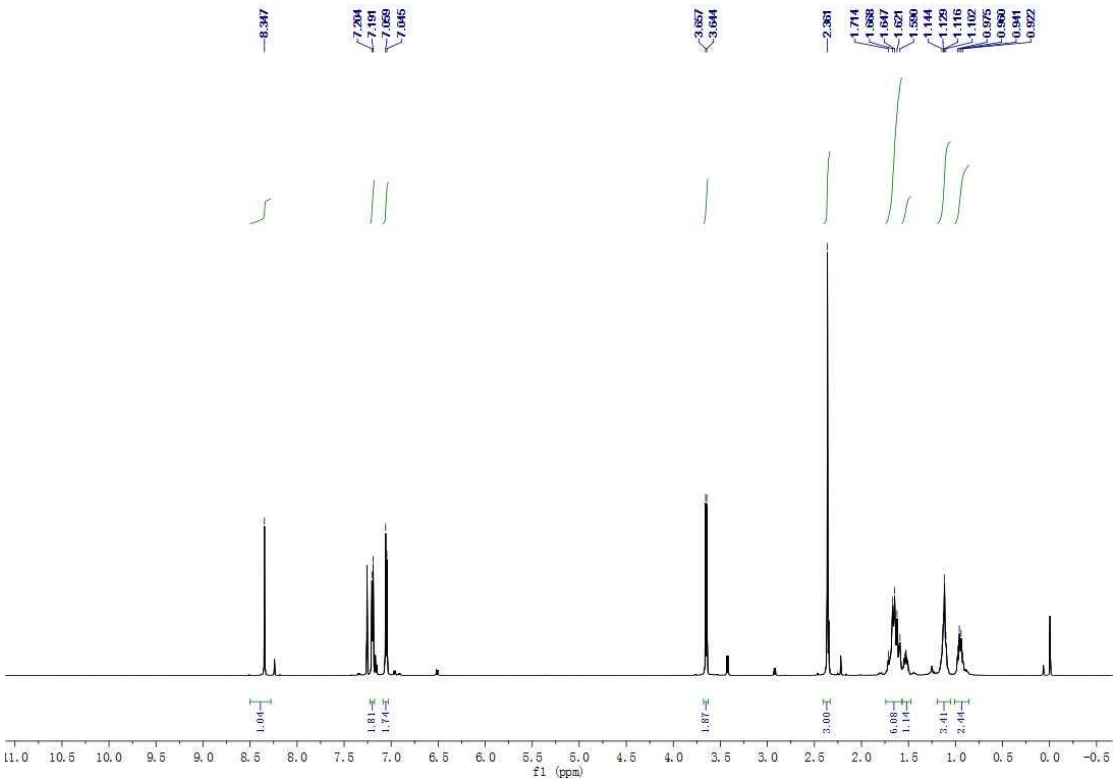
2t:

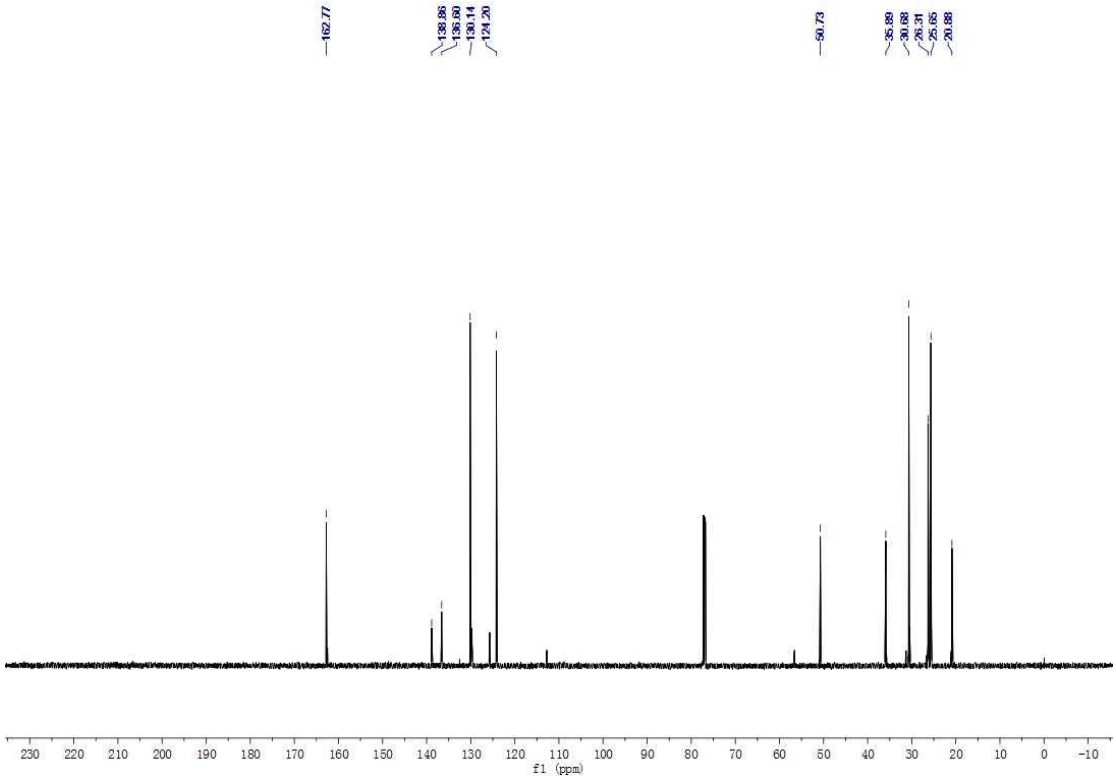

8.310
7.224
7.203
7.118
7.088
3.659
3.641
2.376
1.012
1.006
0.974
0.969
0.944
0.461
0.449
0.446
0.429
0.415
0.231
0.219
0.207
0.193
0.88
2.09
1.60
2.00
3.15
0.96
1.78
1.73
f1 (ppm)


162.35
138.67
136.96
130.06
125.11
49.63
20.93
9.82
3.80
f1 (ppm)

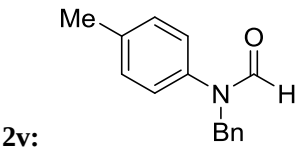

Me
O
N
H

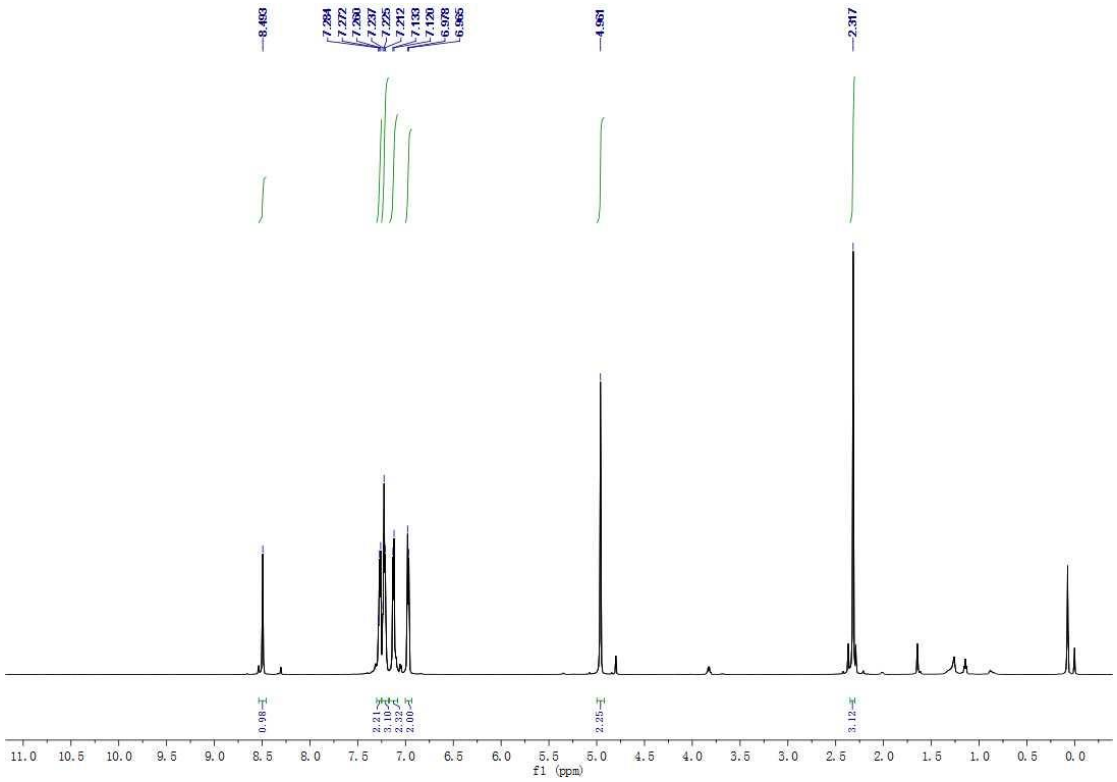
2u:

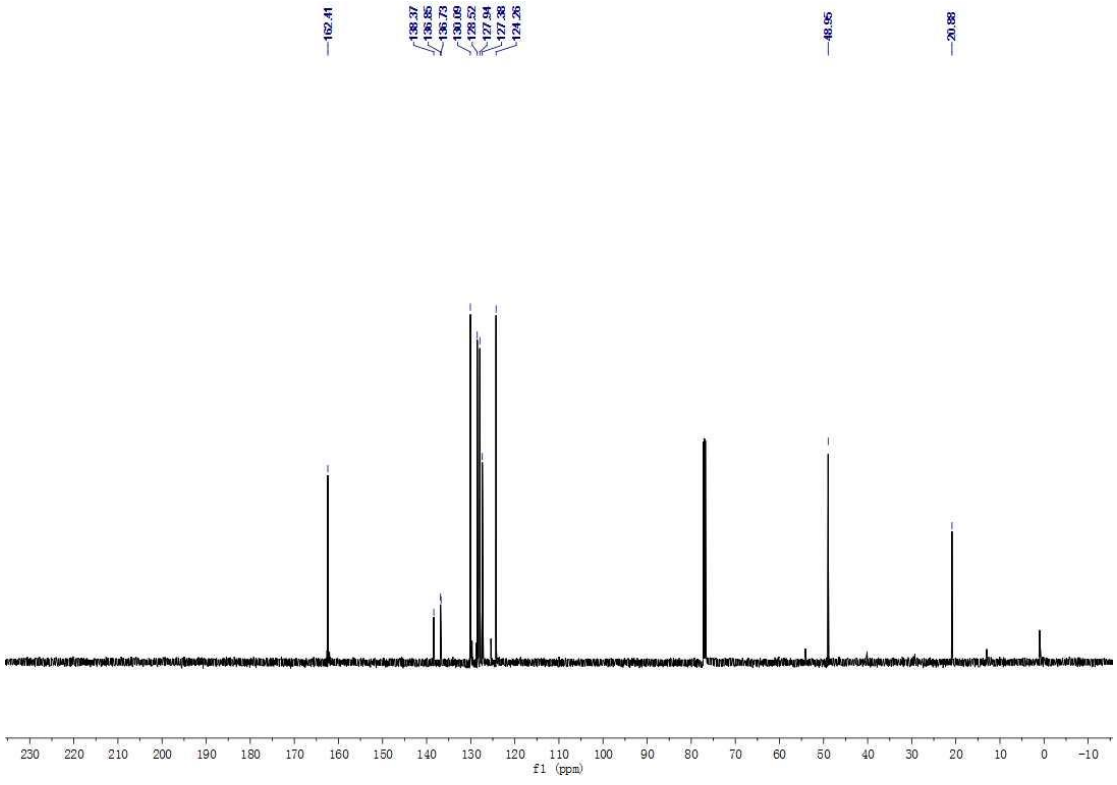

8.347
7.204
7.191
7.059
7.045
3.657
3.644
2.361
1.714
1.668
1.647
1.621
1.590
1.144
1.129
1.116
1.102
0.975
0.960
0.941
0.922
1.04
1.81
1.74
1.87
3.00
6.08
1.14
3.41
2.44
f1 (ppm)


162.77
138.86
136.60
130.14
124.20
50.73
35.89
30.88
26.31
25.65
20.88
f1 (ppm)

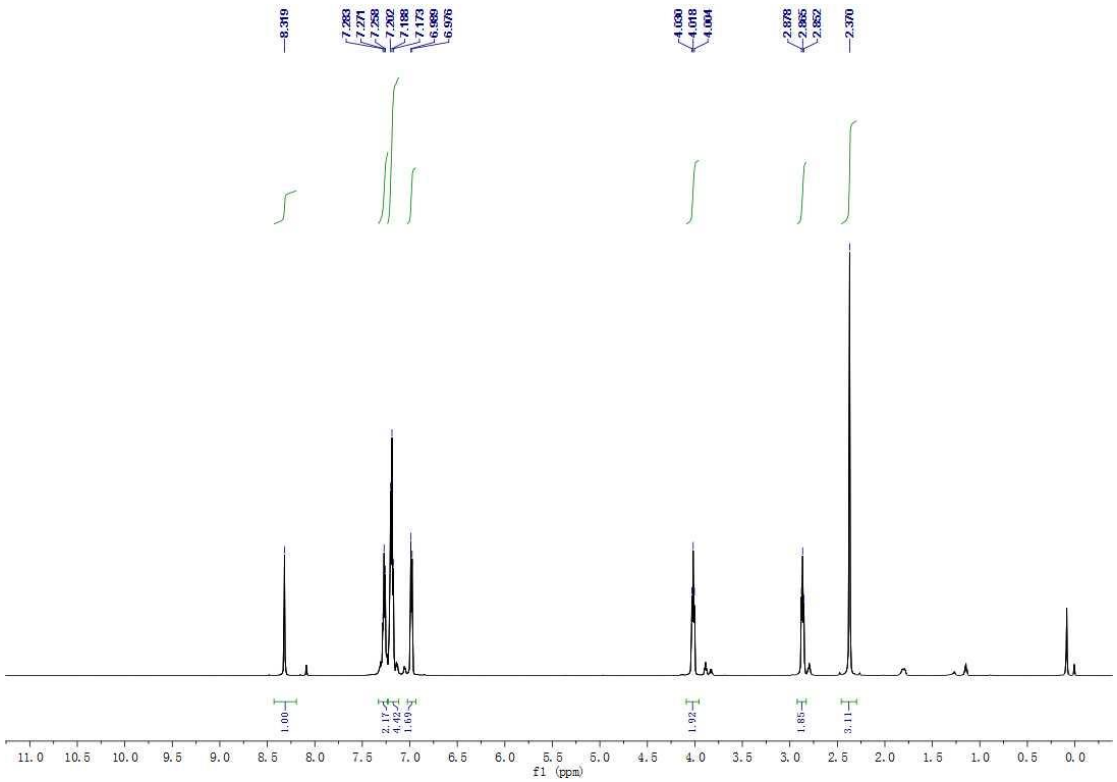
2v:

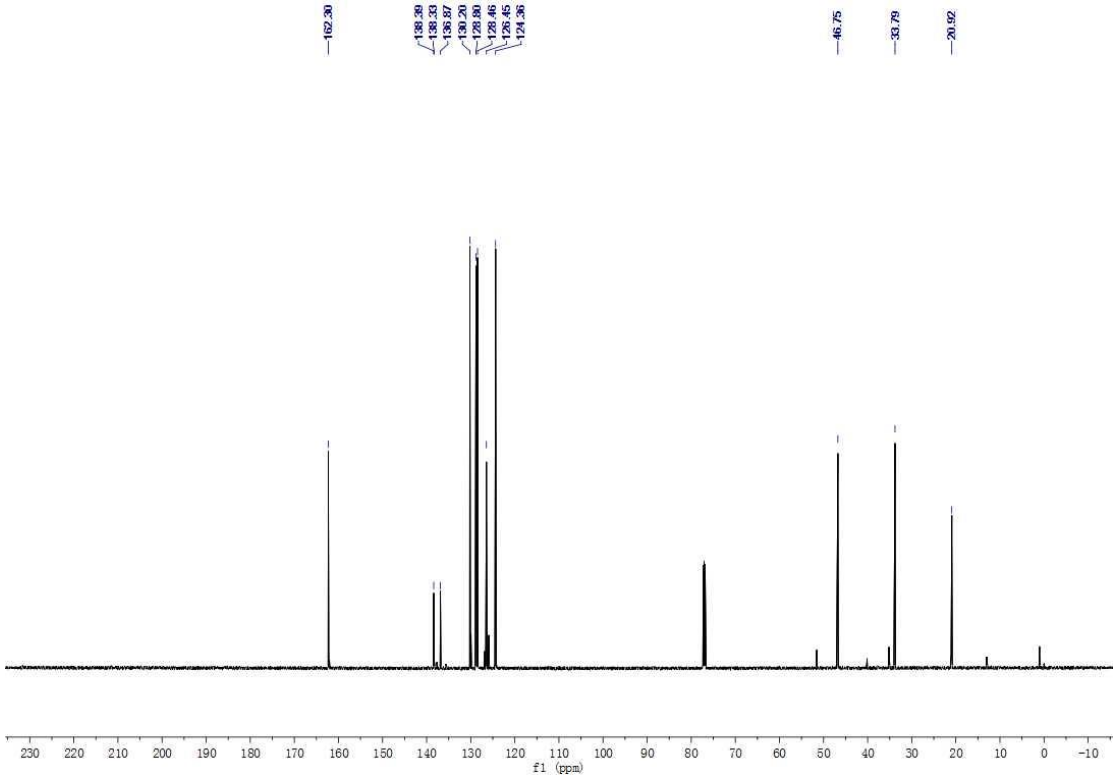

Me
O
N
H
Bn


8.493
7.284
7.272
7.260
7.237
7.225
7.212
7.133
7.120
6.978
6.965
4.961
2.317
0.98
2.21
3.10
2.32
2.00
2.25
3.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

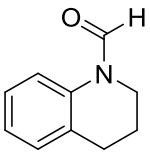

162.41
138.37
136.85
136.73
130.09
128.52
127.94
127.38
124.26
48.95
20.88
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

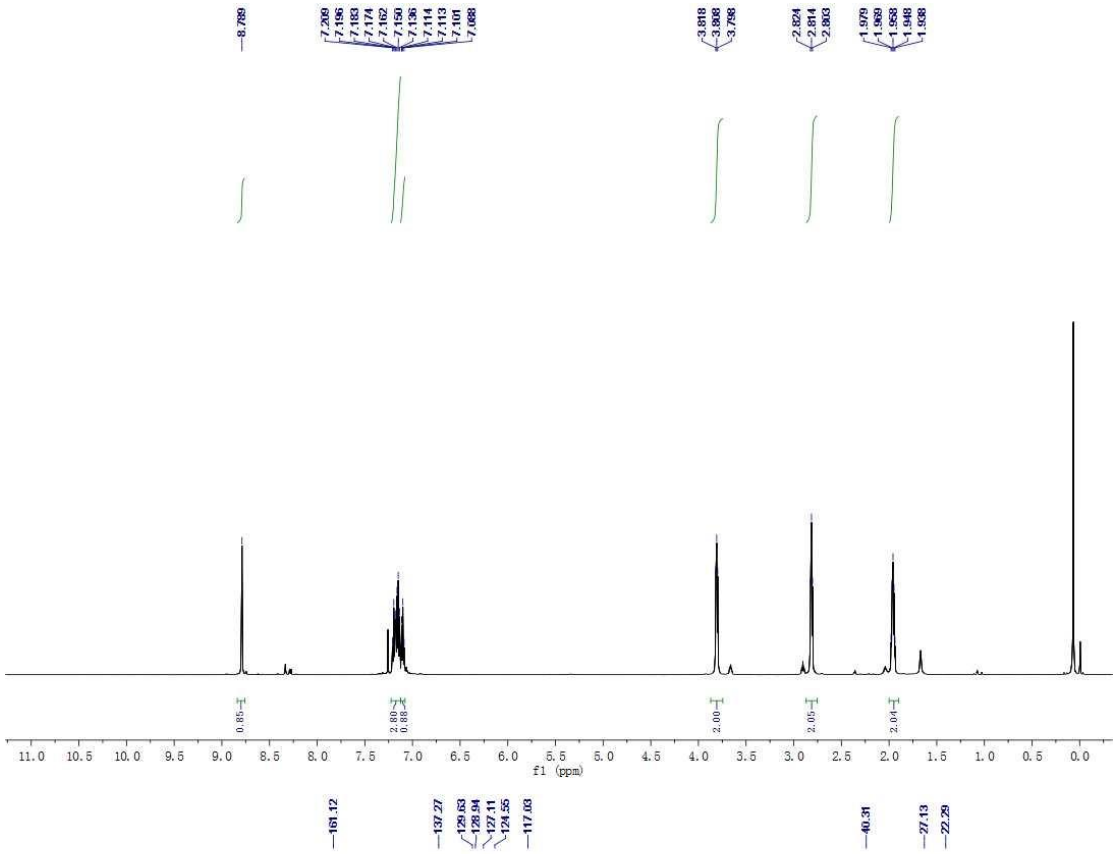
2w:

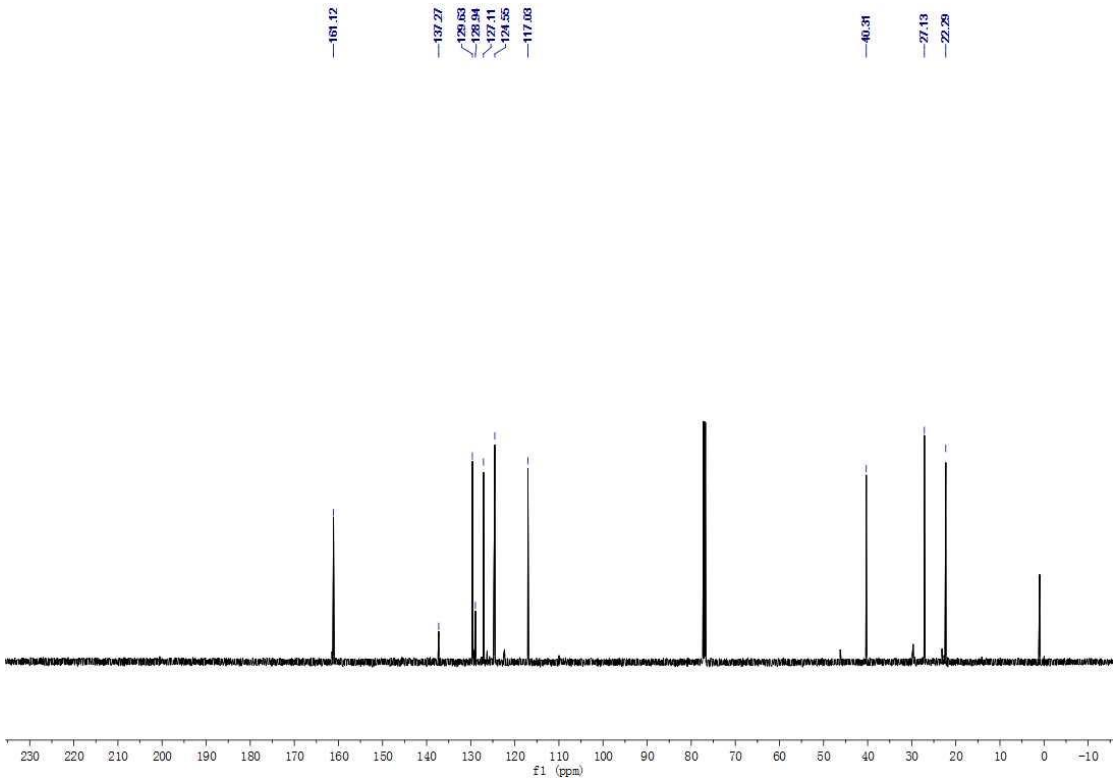

8.319
7.293
7.271
7.258
7.202
7.198
7.173
6.989
6.976
4.030
4.018
4.004
2.878
2.865
2.852
2.370
1.00
2.17
4.42
1.69
1.92
1.85
3.11
f1 (ppm)


162.30
139.30
138.33
136.57
130.20
128.80
128.46
126.45
124.36
46.75
33.79
20.92
f1 (ppm)

2x:




8.789
7.209
7.196
7.183
7.174
7.162
7.150
7.136
7.114
7.113
7.101
7.088
3.818
3.808
3.798
2.824
2.814
2.803
1.979
1.969
1.958
1.948
1.938
0.85
2.89
0.85
2.00
2.05
2.04
f1 (ppm)


161.12
137.27
129.63
128.94
127.11
124.55
117.03
40.31
27.13
22.29
f1 (ppm)

^{31}P NMR Spectra: