Palladium-catalyzed Suzuki cross-couplings of *N*'-mesyl arylhydrazines via C–N bond cleavage†

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Supporting Information

1. General procedures

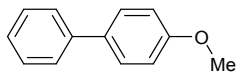
The solvents were distilled from standard drying agents. Unless otherwise stated, commercial reagents purchased from Alfa Aesar, Acros and Aldrich chemical companies were used without further purification. Purification of reaction products was carried out by flash chromatography using Qing Dao Sea Chemical Reagent silica gel (200–300 mesh). ^{1}H NMR spectra were recorded on a Bruker Avance III 400 (400 MHz) spectrometer and referenced internally to the residual proton resonance in $CDCl_3$ (δ = 7.26 ppm), or with tetramethylsilane (TMS, δ = 0.00 ppm) as the internal standard. Chemical shifts were reported as parts per million (ppm) in the δ scale downfield from TMS. Multiplicity is indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), dd (doublet of doublet), bs (broad singlet). ^{13}C NMR spectra were recorded on Bruker spectrometer with complete proton decoupling, and chemical shifts were reported in ppm from TMS with the solvent as the internal reference ($CDCl_3$, δ = 77.0 ppm). Analytical TLC was performed using EM separations percolated silica gel 0.2 mm layer UV 254 fluorescent sheets.

2 Typical procedure for Pd-catalyzed Suzuki cross-coupling of arylhydrazines with aryl boronic acids

A mixture of *N*'-mesyl arylhydrazines **1** (0.30 mmol), aryl boronic acid **2** (0.36 mmol, 1.2 equiv.), K_2CO_3 (0.6 mmol, 2.0 equiv.), and $PdCl_2(PPh_3)_2$ (0.015 mmol, 5 mol %) was stirred at 60 °C in MeOH (2.0 mL) for 4-8 h under air. After completion of the reaction (indicated by TLC), the mixture was quenched with saturated NaCl solution, extracted by EtOAc, and dried with Na_2SO_4. The crude product was purified by flash column chromatography to provide the corresponding product **3**.

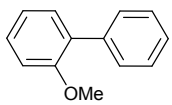
3. Characterization of the products

4-Methoxybiphenyl[1]



White solid; mp: 90-91 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57-7.52 (m, 4H), 7.42 (t, J = 7.6 Hz, 2H), 7.31 (t, J = 7.2 Hz, 1H), 6.99 (d, J = 7.2 Hz, 2H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.19, 140.85, 133.83, 128.69, 128.13, 126.72, 126.63, 114.23, 55.33.

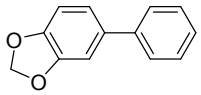
2-Methoxybiphenyl[2]



White solid; mp: 31-32 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, J = 7.2 Hz, 2H), 7.44 (t, J = 7.6 Hz, 2H), 7.37-7.33 (m, 3H), 7.06 (t, J = 7.2 Hz, 1H), 7.01 (d, J = 8.4 Hz, 1H), 3.83 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 156.50, 138.58, 130.91, 130.78, 129.56, 128.62, 127.98, 126.92,

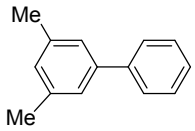
120.85, 111.29, 55.57.

5-phenylbenzo[d][1,3]dioxole[3]



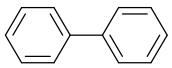
Yellow solid; mp: 81-83°C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, J = 7.2 Hz, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.34 (d, J = 7.2 Hz, 1H), 7.08 (d, J = 8.8 Hz, 2H), 6.90 (d, J = 8.0 Hz, 1H), 6.01 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 148.13, 147.08, 140.96, 135.65, 128.74, 126.94, 126.90, 120.64, 108.58, 107.70, 101.14.

3,5-Dimethylbiphenyl[4]



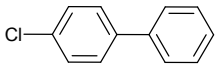
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (t, J = 8.0 Hz, 2H), 7.41 (t, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 1H), 7.21 (s, 2H), 6.99 (s, 1H), 2.37 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.53, 141.32, 138.26, 128.92, 128.66, 127.22, 127.10, 125.15, 21.43.

Biphenyl[1]



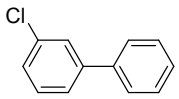
White solid; mp: 68-69 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, J = 7.2 Hz, 4H), 7.47 (t, J = 7.6 Hz, 4H), 7.37 (t, J = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.23, 128.72, 127.22, 127.14.

4-Chlorobiphenyl [5]



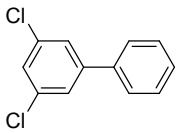
White solid; mp: 31-32 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (dd, J = 14.0, 8.0 Hz, 4H), 7.47-7.43 (m, 3H), 7.40-7.35 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.02, 139.70, 133.41, 128.93, 128.90, 128.41, 127.61, 127.01.

3-Chlorobiphenyl[6]



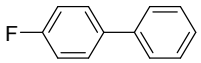
White solid; mp: 45-47 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 -7.57 (m, 3H), 7.48 - 7.34 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.11, 139.84, 134.68, 130.14, 129.99, 128.91, 127.88, 127.28, 127.13, 125.32.

3,5-Dichlorobiphenyl[7]



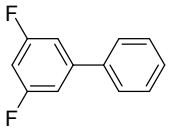
White solid; mp: 61-62 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, J = 7.6 Hz, 2H), 7.43 (dt, J = 8.8, 4.4 Hz, 5H), 7.34 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 148.13, 147.08, 140.96, 135.65, 128.74, 126.94, 126.90, 120.64, 108.58, 107.70, 101.14.

4-Fluorobiphenyl[8]



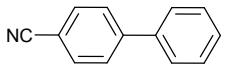
White solid; mp: 72-73 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57-7.55 (m, 4H), 7.46 (t, J = 7.2 Hz, 2H), 7.38 (d, J = 6.8 Hz, 1H), 7.15 (t, J = 8.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 162.69 (d, J = 247.45 Hz), 139.26, 136.36, 127.79, 127.71 (d, J = 8.08 Hz), 126.23, 126.00, 114.69 (d, J = 21.21 Hz).

3,5-Difluorobiphenyl[9]



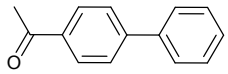
White solid; mp: 72-73 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, J = 6.8 Hz, 1H), 7.47-7.40 (m, 2H), 7.08 (dd, J = 16.4, 6.0 Hz, 3H), 6.87-6.78 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 164.66 (dd, J = 248.46 Hz), 144.61(t, J = 9.49 Hz), 138.93, 129.02, 128.46, 127.01, 110.07 (dd, J = 19.19 Hz), 102.73 (t, J = 25.25 Hz).

Biphenyl-4-carbonitrile[3]



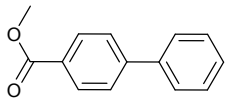
White solid; mp: 85-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (q, J = 8.4 Hz, 4H), 7.59 (d, J = 7.2 Hz, 2H), 7.49 (t, J = 7.6 Hz, 2H), 7.44 (d, J = 7.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 145.70, 139.20, 132.60, 129.12, 128.67, 127.74, 127.24, 118.93, 110.95.

1-(Biphenyl-4-yl)ethanone [3]



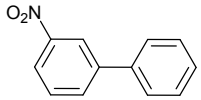
White solid; mp: 152-153 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 7.2 Hz, 2H), 7.47 (t, J = 7.2 Hz, 2H), 7.41 (d, J = 7.2 Hz, 1H), 2.64 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.85, 145.82, 139.89, 135.86, 128.97, 128.93, 128.25, 127.28, 127.25, 26.67.

Methyl biphenyl-4-carboxylate[3]



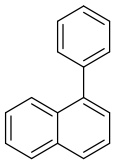
White solid; mp: 120-121 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 8.8 Hz, 2H), 7.65 (dd, J = 15.2, 7.2 Hz, 4H), 7.47 (t, J = 7.6 Hz, 2H), 7.40 (t, J = 7.2 Hz, 1H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.01, 145.66, 140.03, 130.10, 128.92, 128.13, 127.28, 127.05, 52.10.

3-Nitrobiphenyl [5]



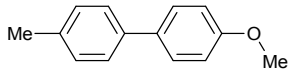
Yellow solid; mp: 59-60 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (s, 1H), 8.21 (d, J = 8.2 Hz, 1H), 7.92 (d, J = 7.2 Hz, 1H), 7.64-7.60 (m, 3H), 7.50 (t, J = 7.2 Hz, 2H), 7.44 (d, J = 7.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 148.75, 142.91, 138.69, 133.06, 129.72, 129.18, 128.56, 127.18, 122.05, 122.01.

1-Phenylnaphthalene[3]



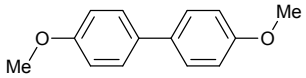
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, J = 9.2 Hz, 2H), 7.88 (d, J = 8.4 Hz, 1H), 7.55-7.50 (m, 6H), 7.47-7.45 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.84, 140.33, 133.87, 131.70, 130.13, 128.31, 127.68, 127.28, 126.97, 126.09, 126.07, 125.81, 125.42.

4-Methoxy-4'-methylbiphenyl[1]



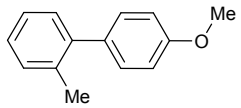
White solid; mp: 107-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, J = 8.4 Hz, 2H), 7.48 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H), 6.99 (d, J = 8.4 Hz, 2H), 3.87 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 158.94, 137.98, 136.35, 133.76, 129.43, 127.95, 126.58, 114.17, 55.34, 21.04.

4, 4'-Dimethoxybiphenyl[1]



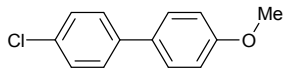
White solid; mp: 168-169 °C; ^{1}H NMR (400 MHz, CDCl3): δ 7.48 (d, J = 8.8 Hz, 4H), 6.96 (d, J = 8.8 Hz, 4H), 3.84 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 158.72, 133.51, 127.74, 114.18, 55.35.

4'-Methoxy-2-methylbiphenyl[2]



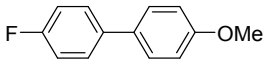
White solid; mp: 52-53 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.22 (m, 6H), 6.96-6.94 (m, 2H), 3.85 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 158.54, 141.58, 135.50, 134.41, 130.29, 130.26, 129.91, 126.97, 125.75, 113.51, 55.30, 20.54.

4-Chloro-4'-methoxybiphenyl[2]



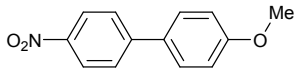
White solid; mp: 110-111 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49-7.46 (m, 4H), 7.38-7.36 (m, 2H), 6.98-6.96 (m, 2H), 3.85 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.36, 139.27, 132.66, 132.50, 128.81, 127.99, 127.91, 114.30, 55.35.

4-Fluoro-4'-methoxybiphenyl[8]



White solid; mp: 84-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.46 (m, 4H), 7.10 (t, J = 8.8 Hz, 2H), 6.98 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 163.35 (d, J = 246.34 Hz), 159.16, 137.02 (d, J = 3.03 Hz), 132.87, 128.26 (d, J = 8.10 Hz), 128.04, 115.64 (d, J = 21.21 Hz), 114.29, 55.35.

4-Methoxy-4'-nitrobiphenyl[1]



Yellow solid; mp: 104-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (d, J = 8.8 Hz, 2H), 7.70 (d, J = 8.8 Hz, 2H), 7.59 (d, J = 8.8 Hz, 2H), 7.03 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 160.49, 147.23, 146.59, 131.11, 128.58, 127.09, 124.16, 114.64, 55.44.

Reference

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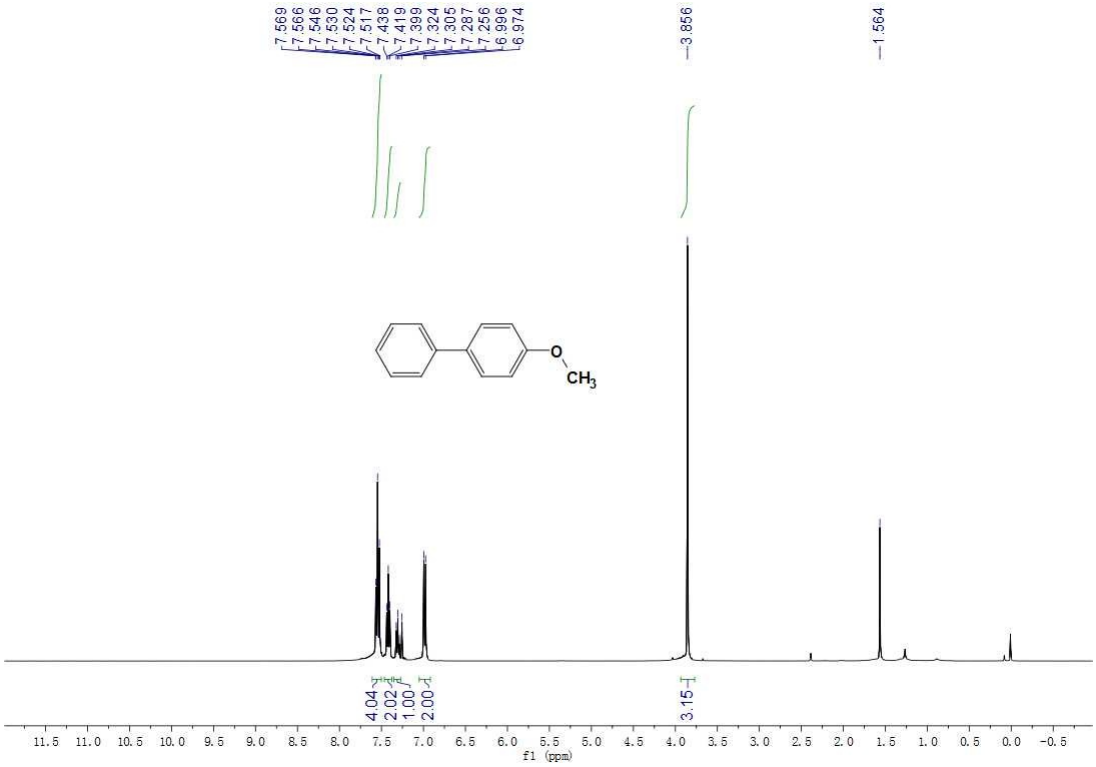
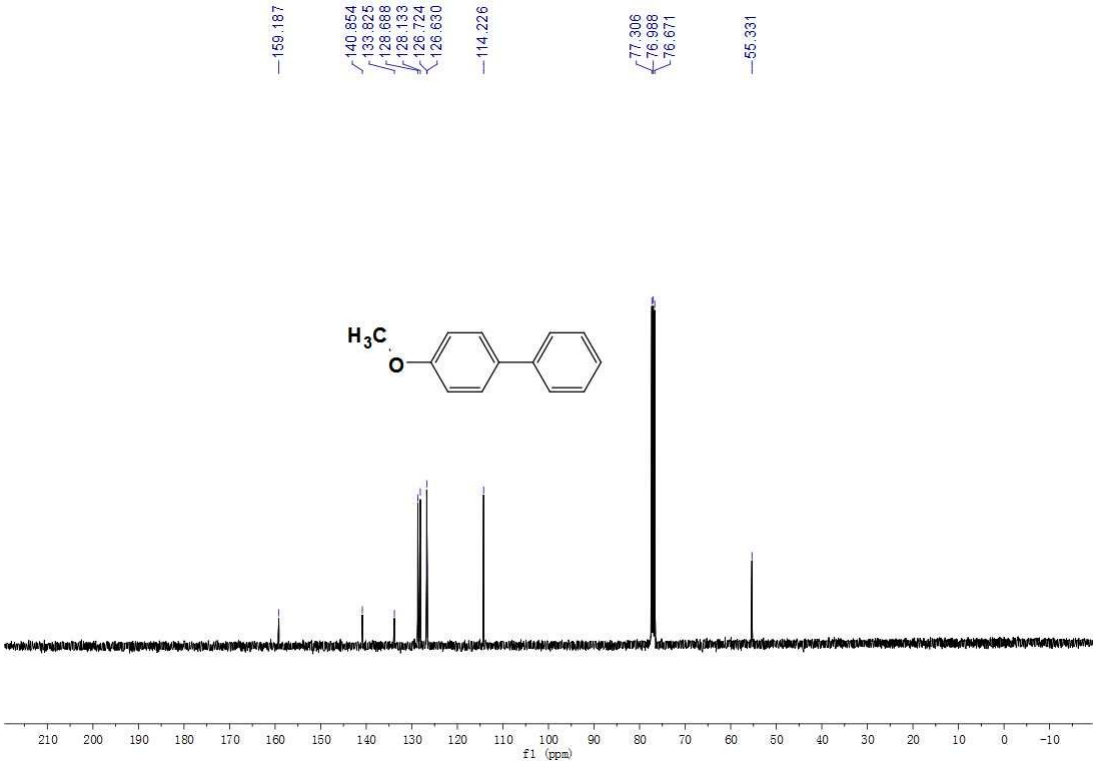
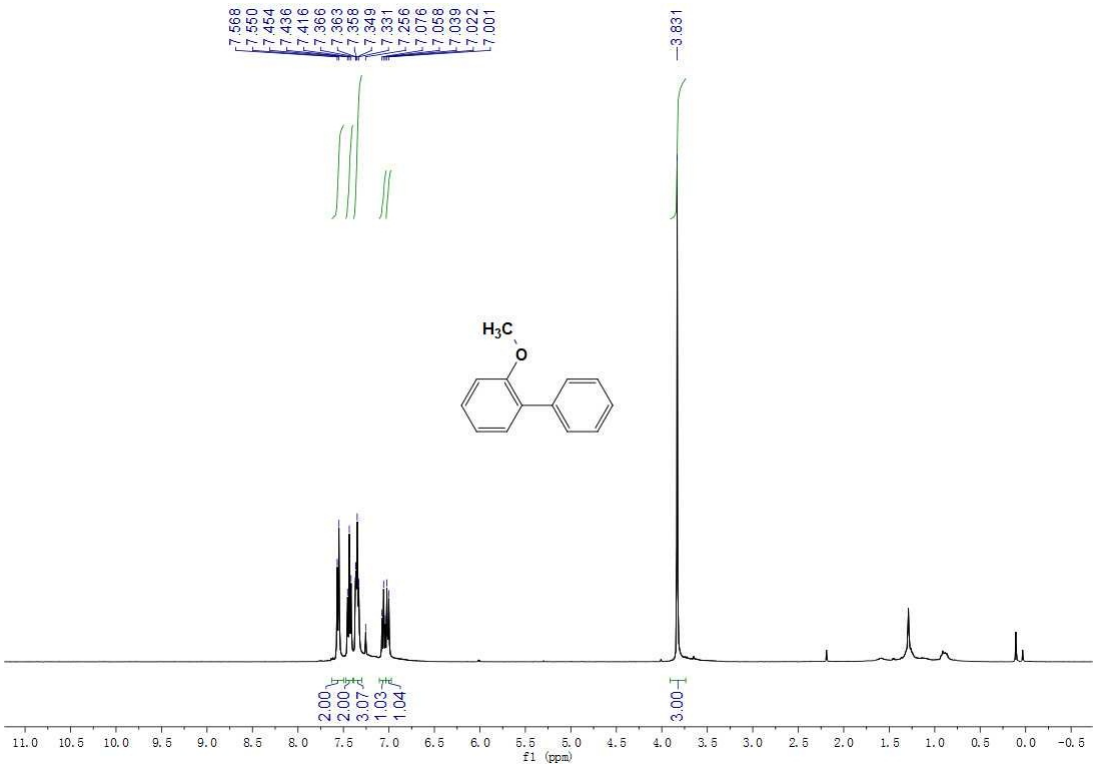
4. NMR spectra of the compounds



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7.550
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7.366
7.363
7.358
7.349
7.331
7.256
7.076
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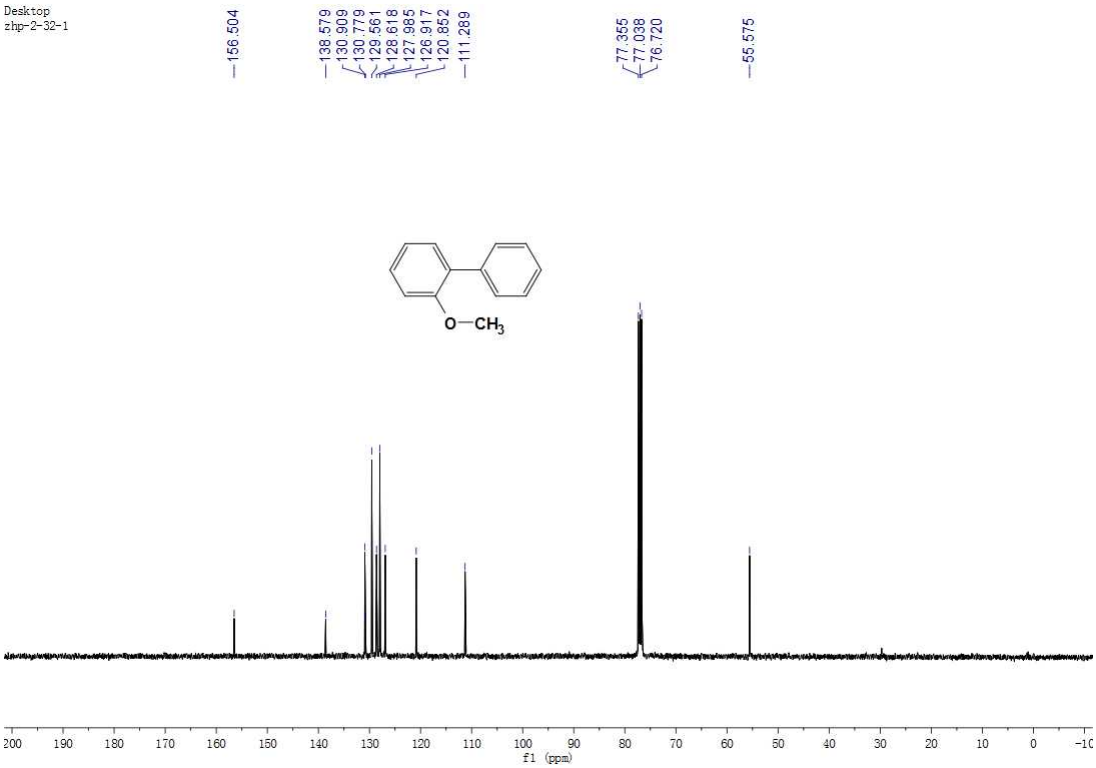
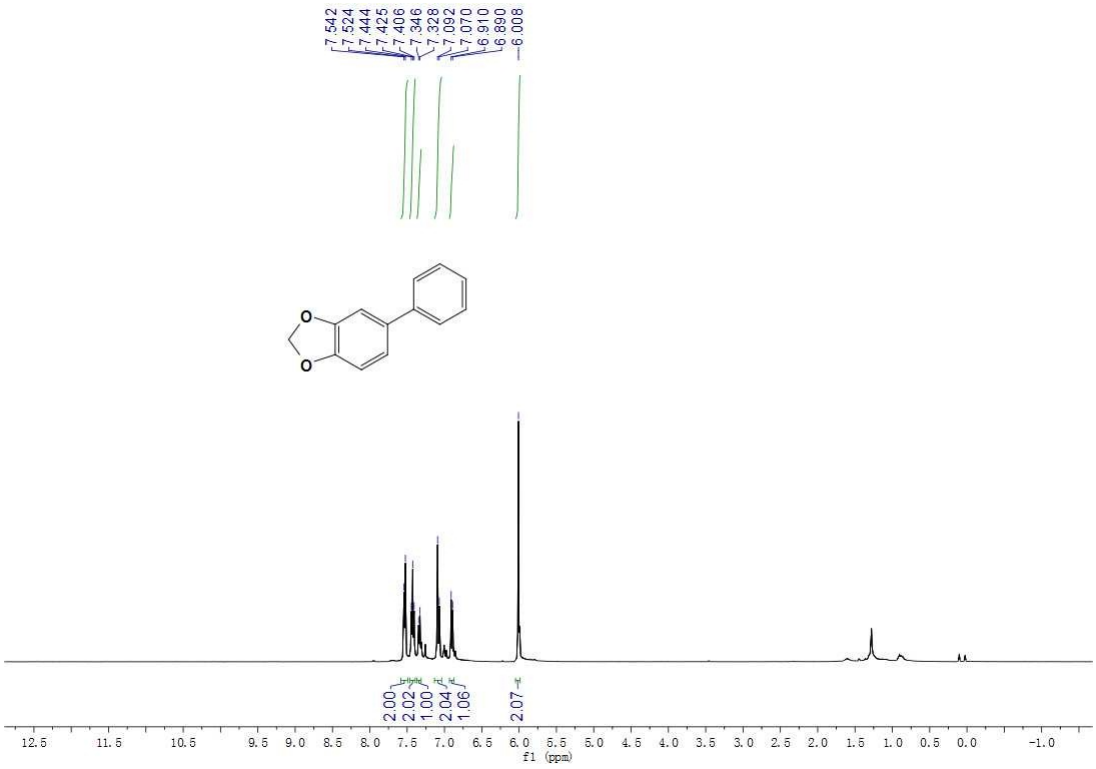
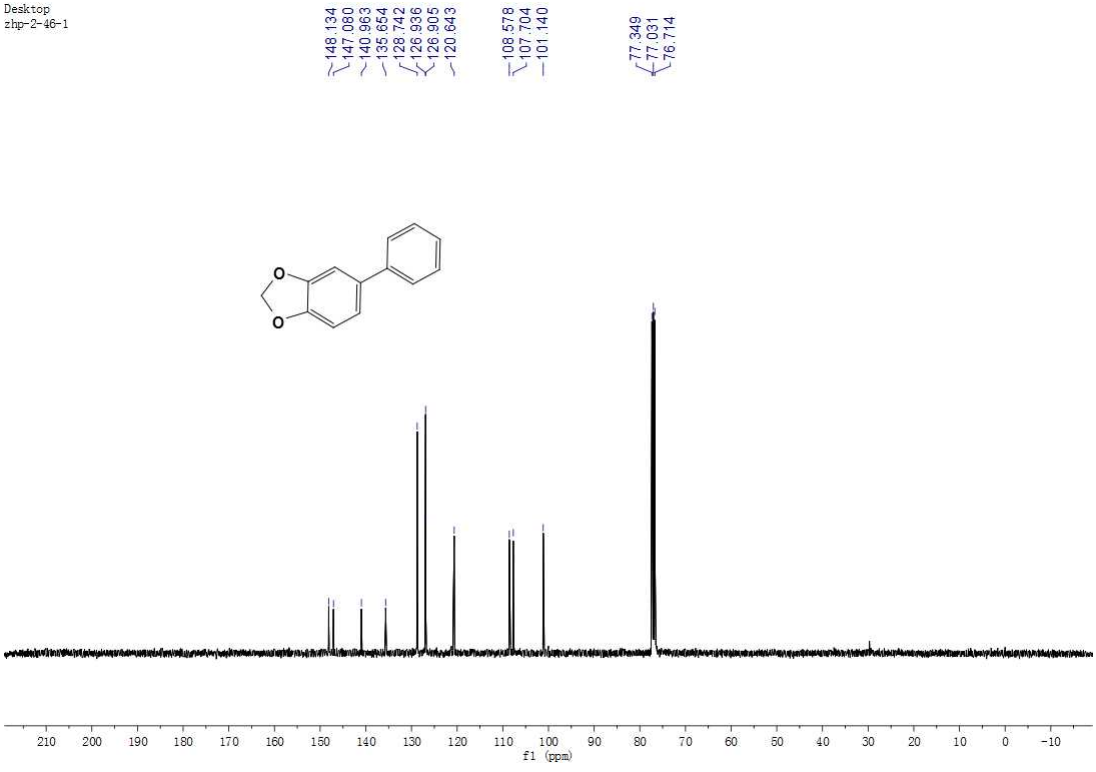
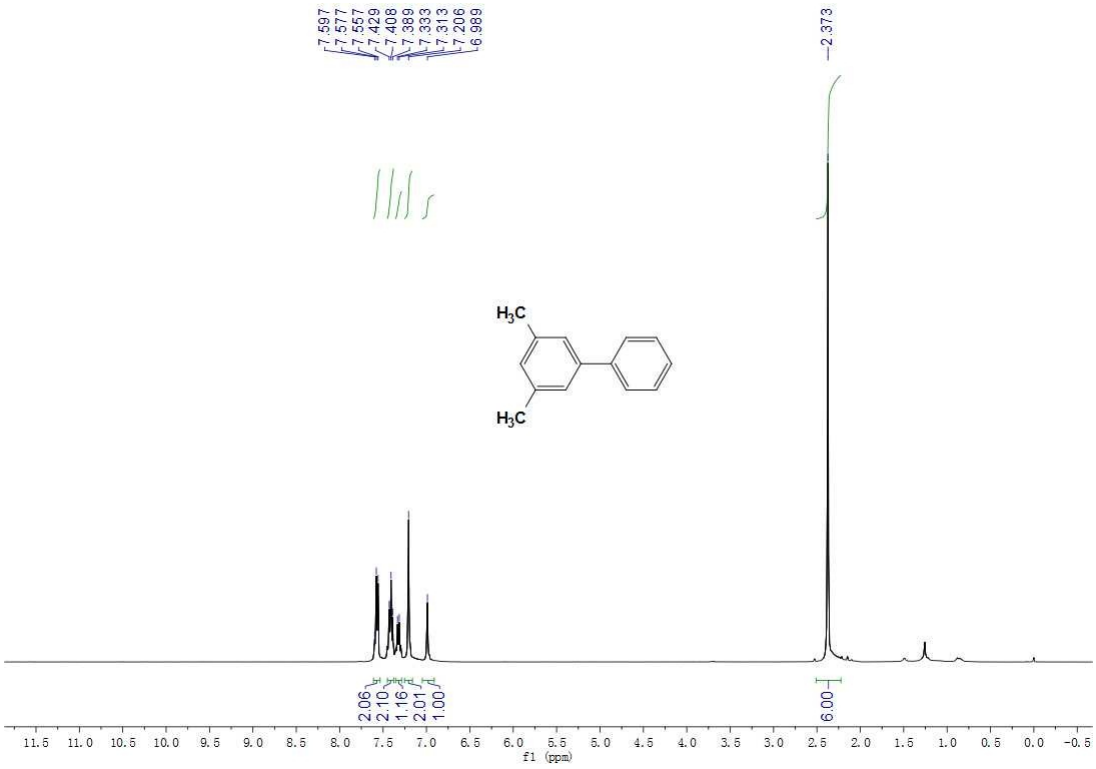
7.039
7.022
7.001
3.831
H3C
O
2.00
2.00
3.07
1.03
1.04
3.00
f1 (ppm)


Desktop
zhp-2-32-1
156.504
138.579
130.909
130.779
129.561
128.618
127.985
126.917
120.852
111.289
77.355
77.038
76.720
55.575
O—CH3
f1 (ppm)

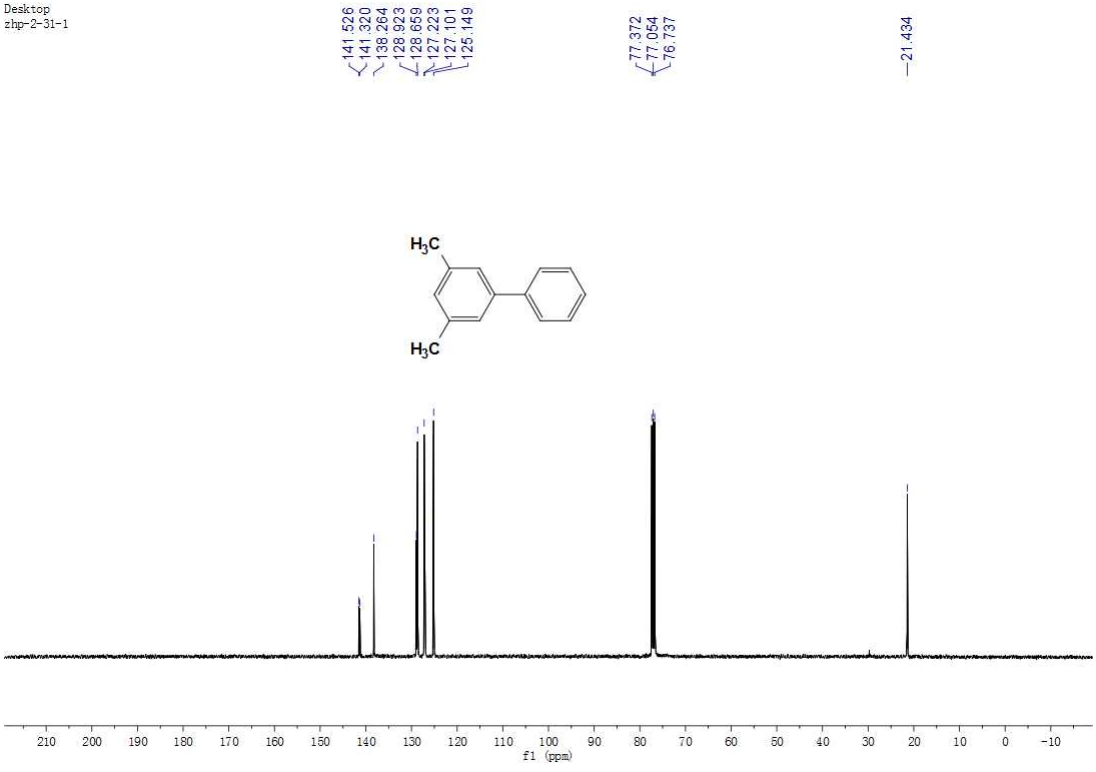


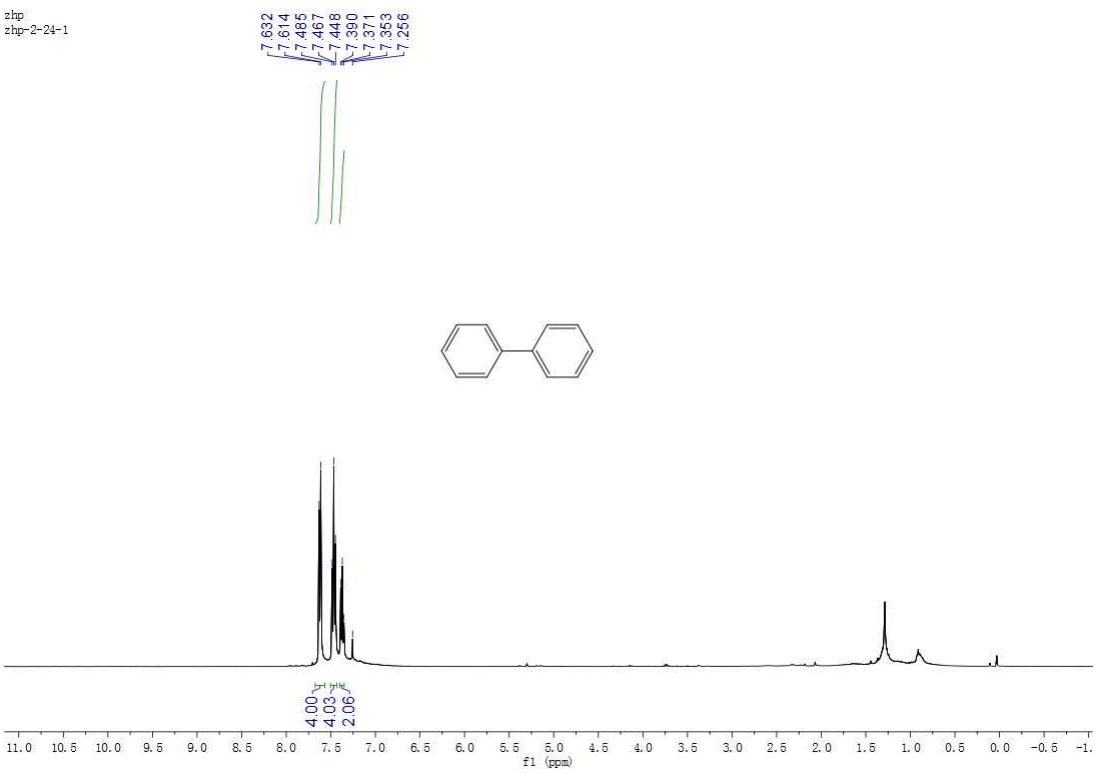
Desktop
zhp-2-46-1

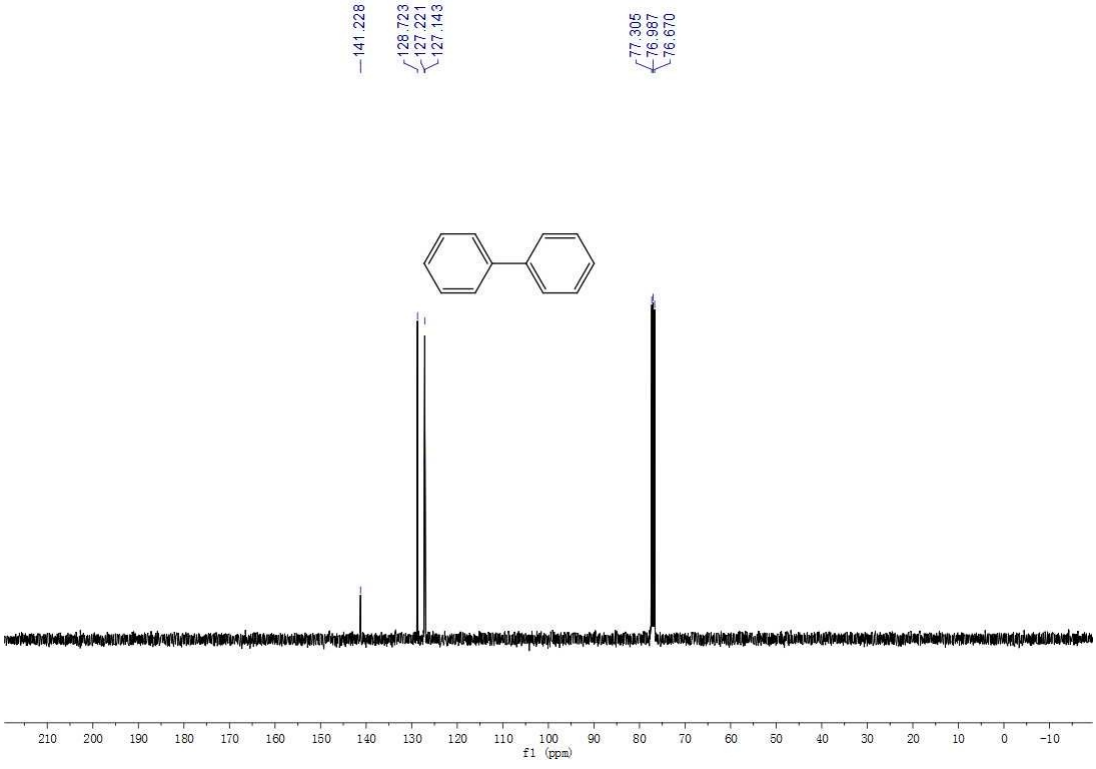


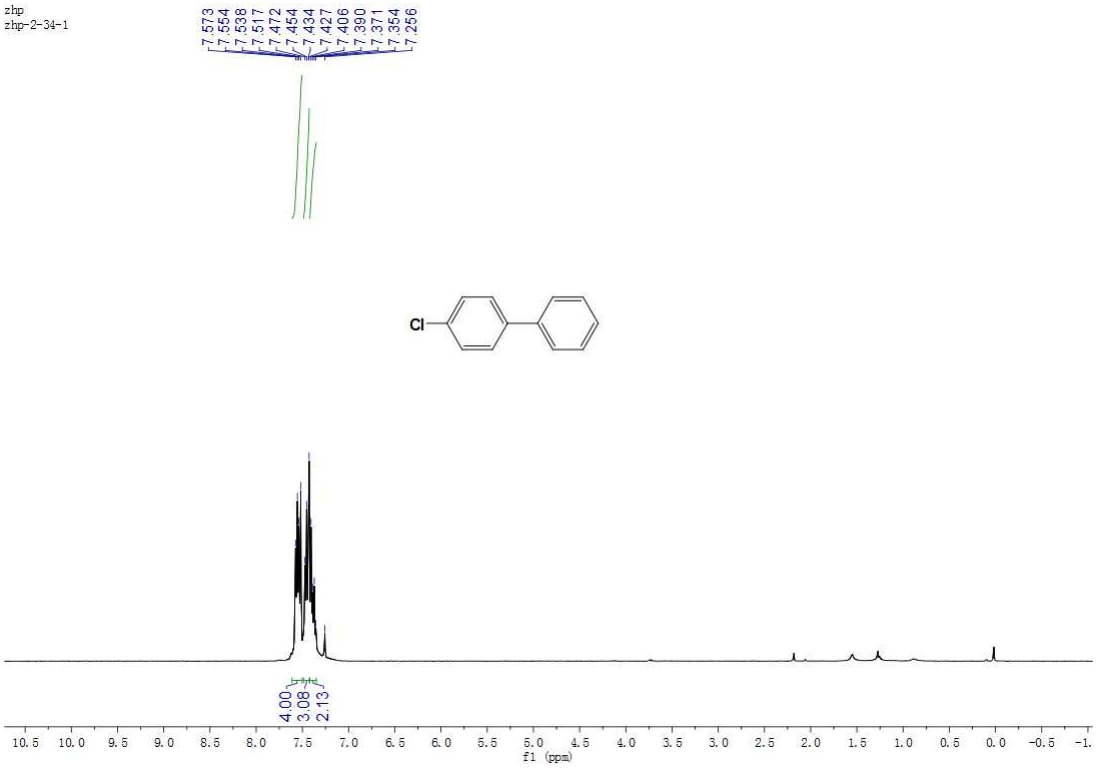


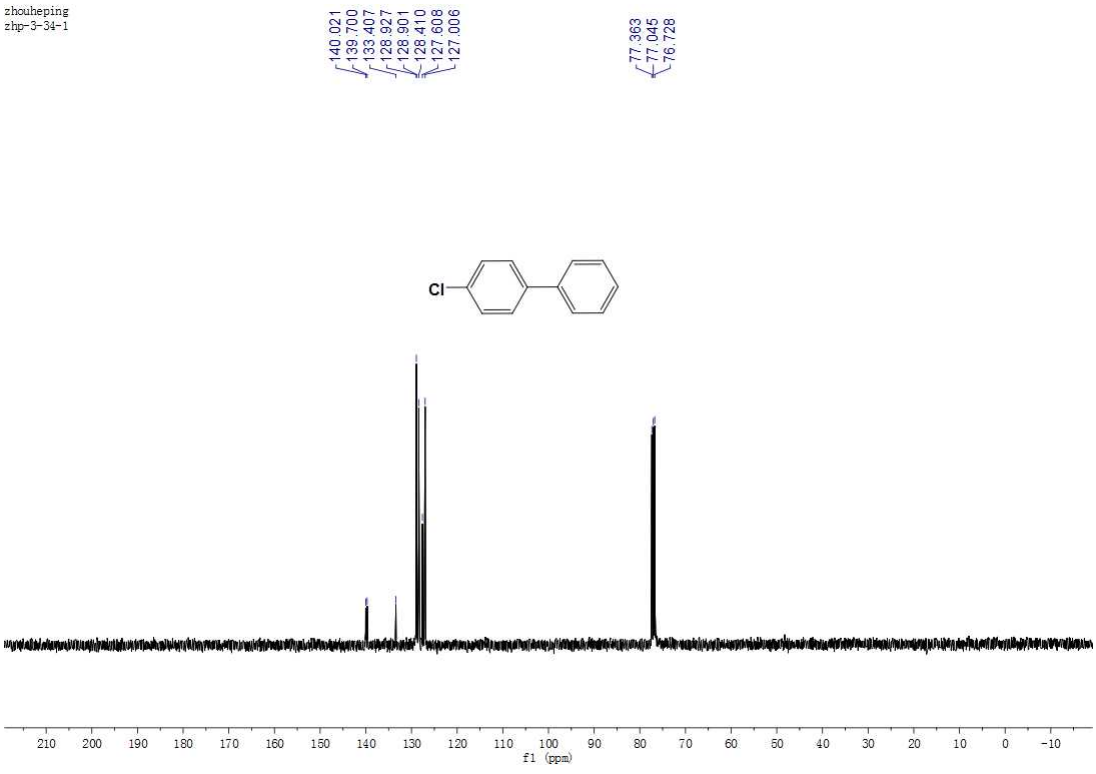
Desktop
zhp-2-31-1

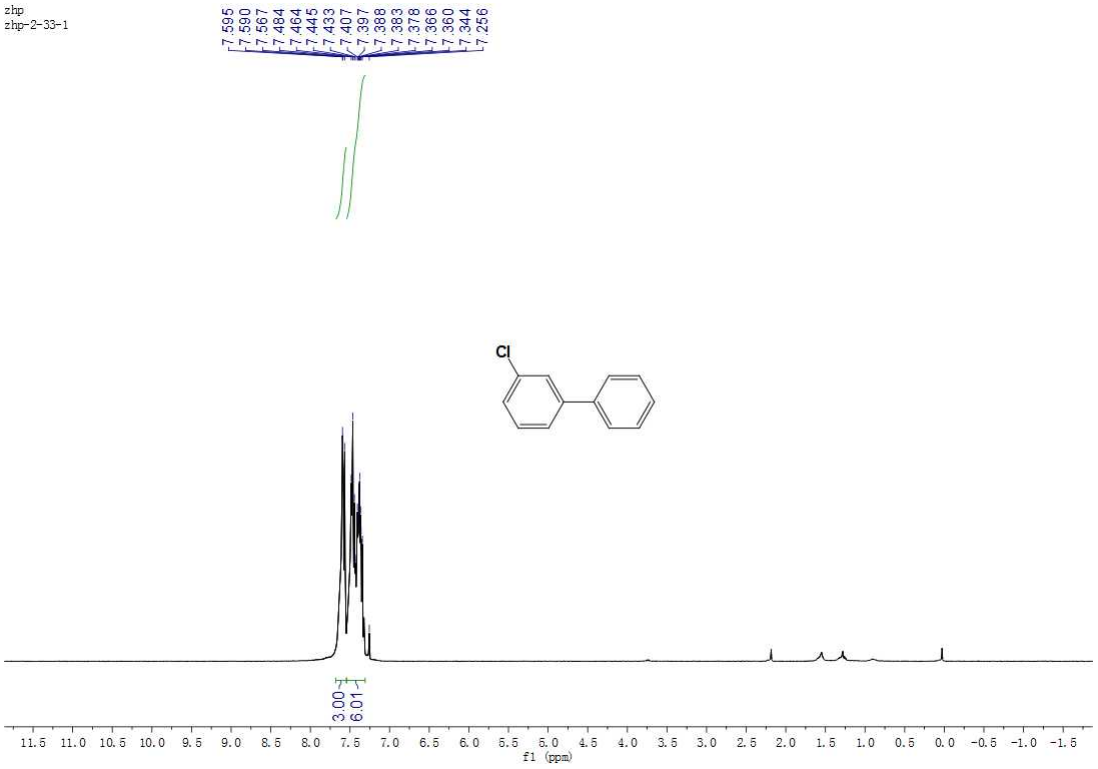


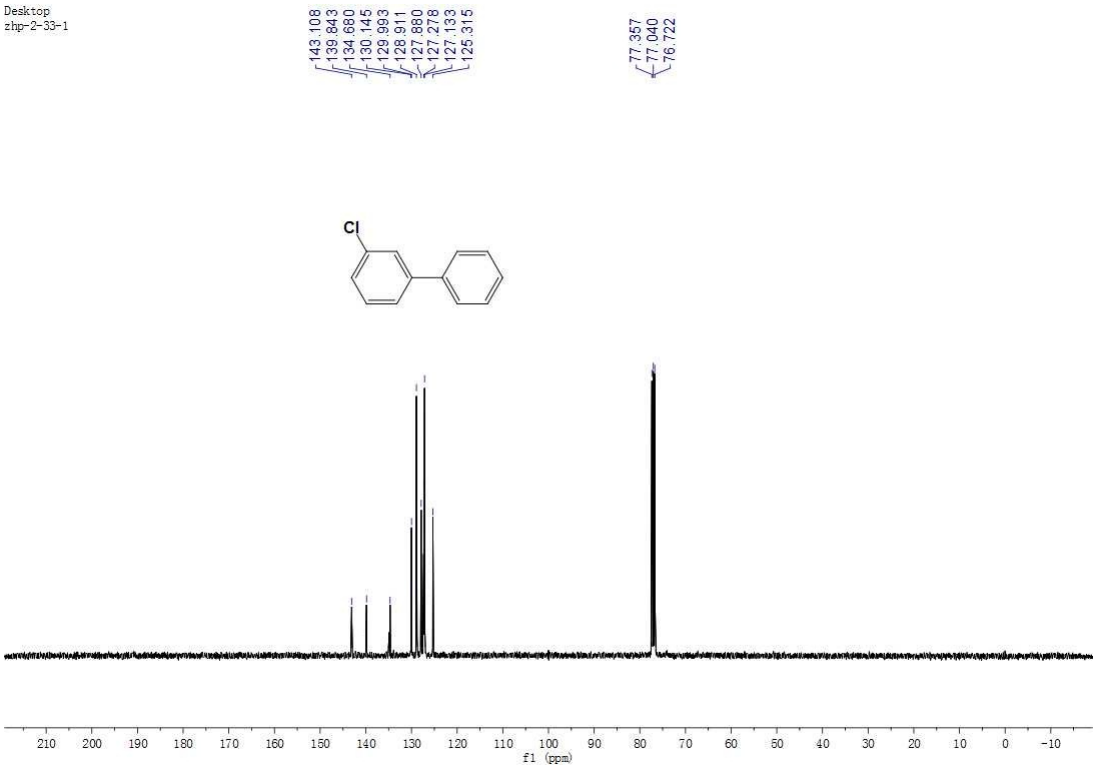

zhp
zhp-2-24-1
7.632
7.614
7.485
7.467
7.448
7.390
7.371
7.353
7.256
4.00
4.03
2.06
f1 (ppm)

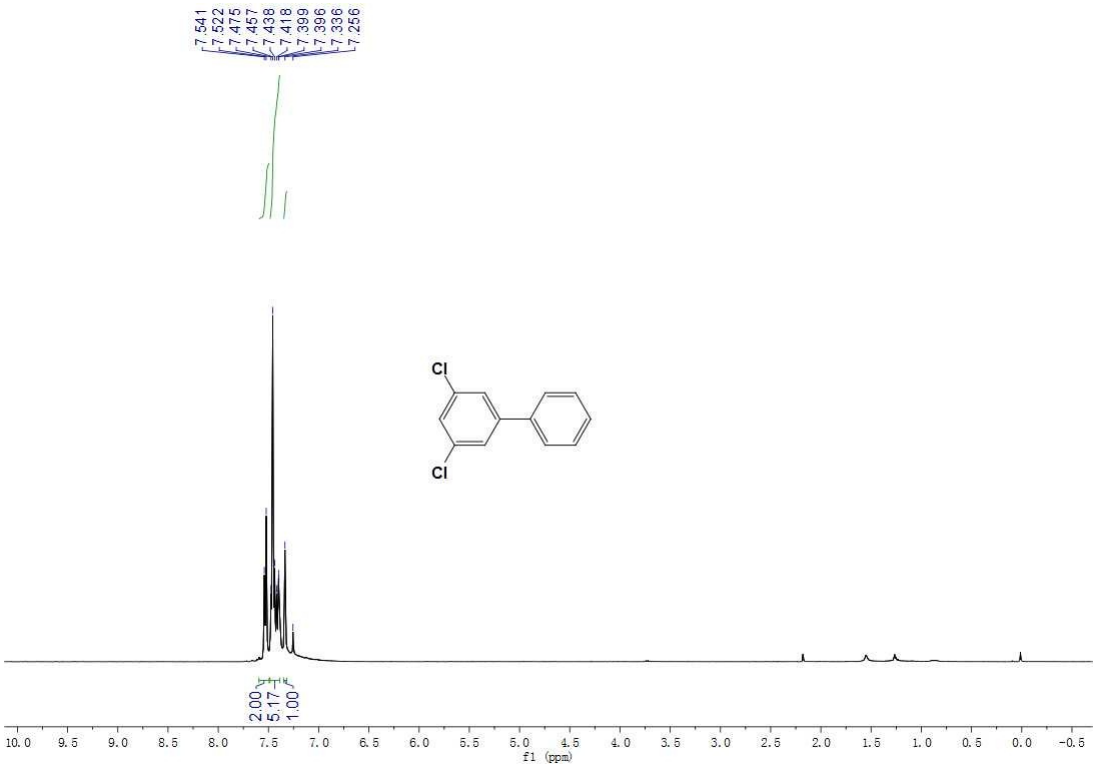

141.228
128.723
127.221
127.143
77.305
76.987
76.670
f1 (ppm)


zhp
zhp-2-34-1
7.573
7.554
7.538
7.517
7.472
7.454
7.434
7.427
7.406
7.390
7.371
7.354
7.256
Cl
4.00
3.08
2.13
f1 (ppm)

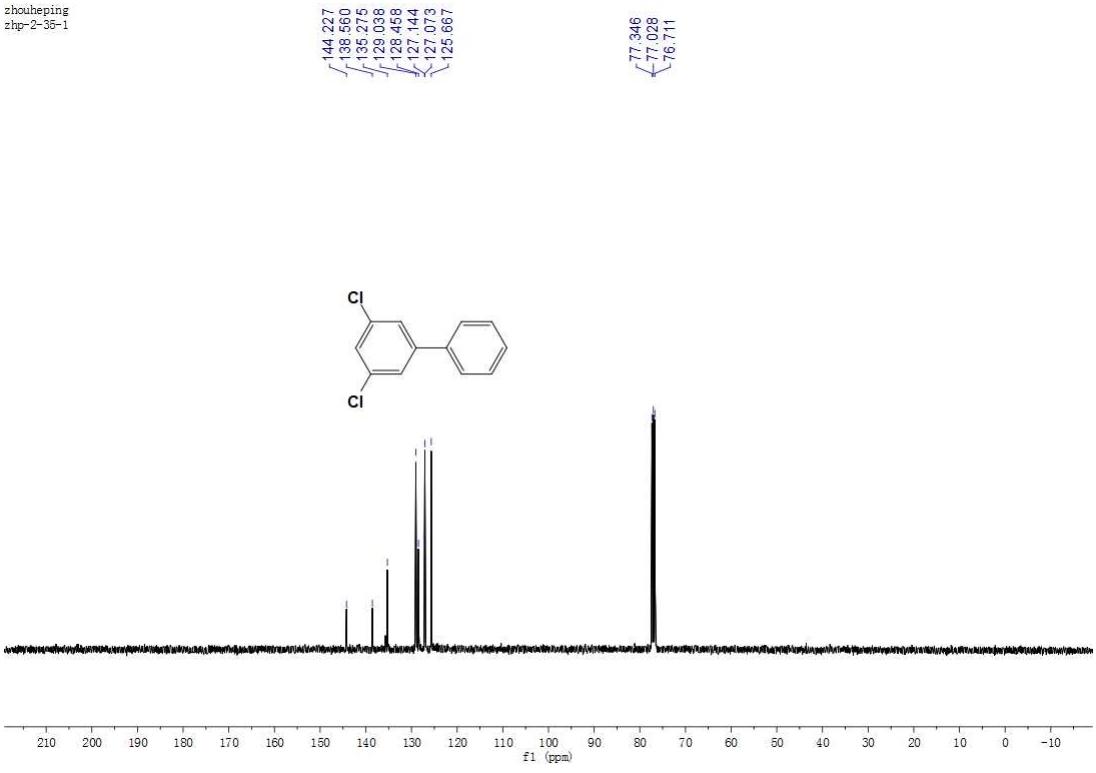

zhouheping
zhp-3-34-1
140.021
139.700
133.407
128.927
128.901
128.410
127.608
127.006
77.363
77.045
76.728
Cl
f1 (ppm)

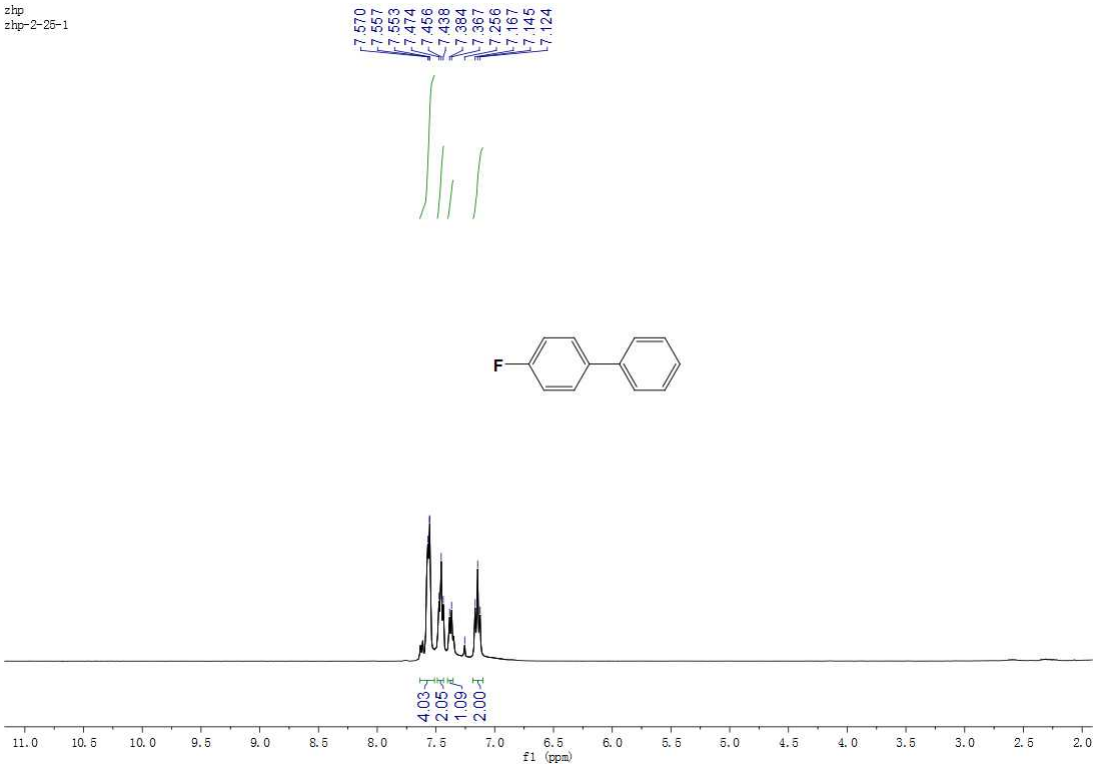

zhp
zhp-2-33-1
7.595
7.590
7.567
7.484
7.464
7.445
7.433
7.407
7.397
7.388
7.383
7.378
7.366
7.360
7.344
7.256
Cl
3.00
6.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

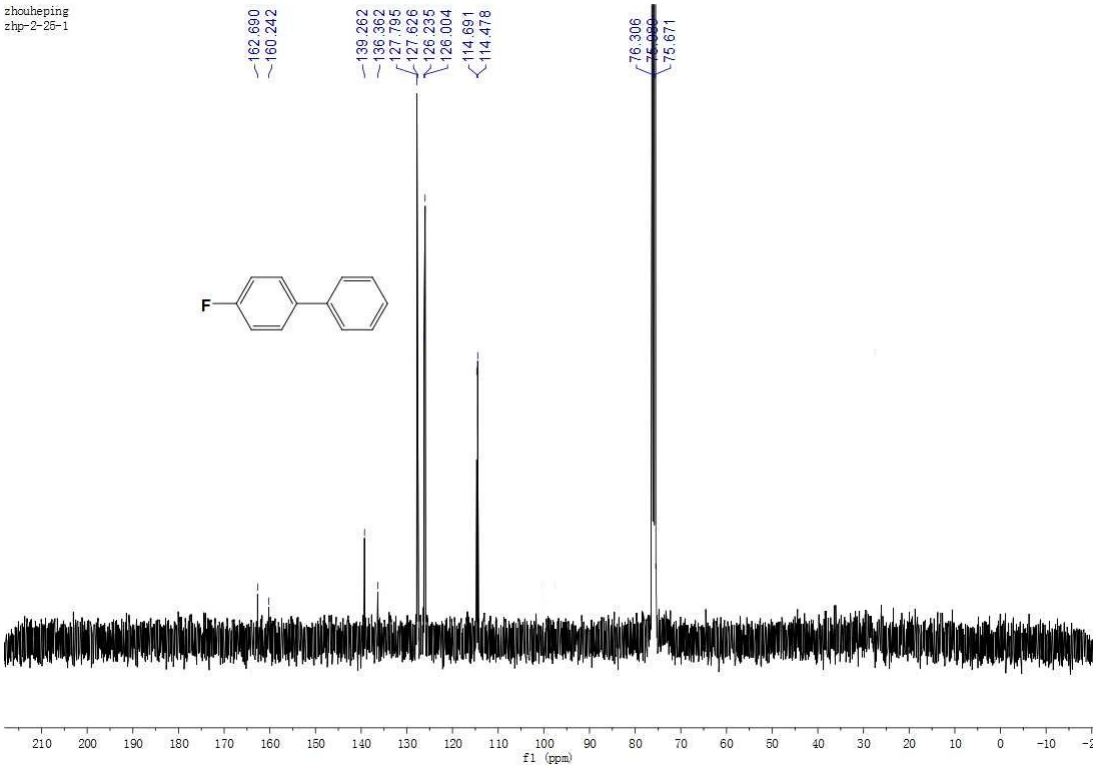

Desktop
zhp-2-33-1
143.108
139.843
134.680
130.145
129.993
128.911
127.880
127.278
127.133
125.315
77.357
77.040
76.722
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

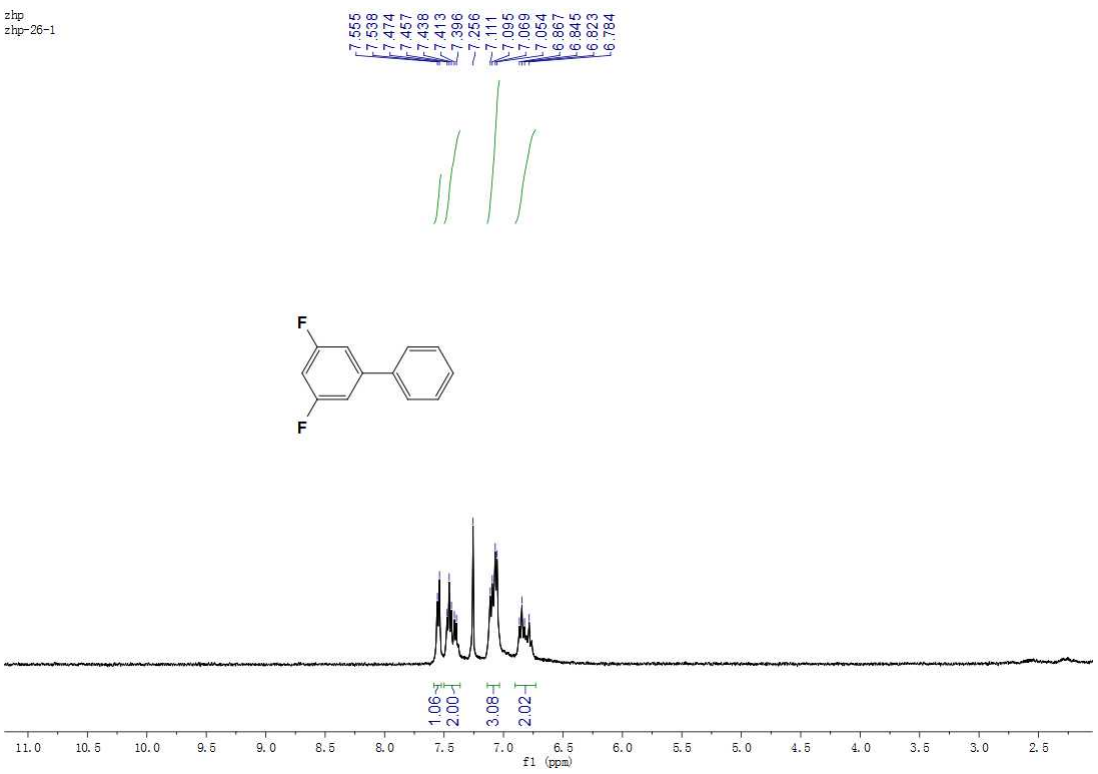


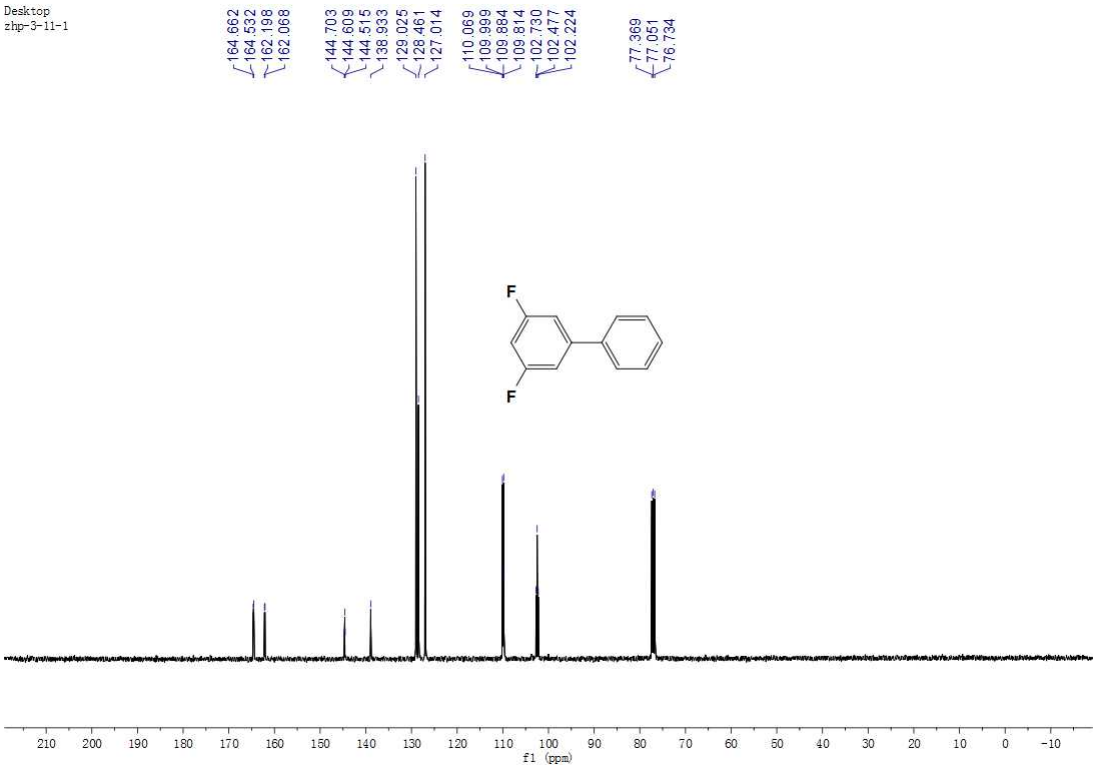
zhouheping
zhp-2-35-1

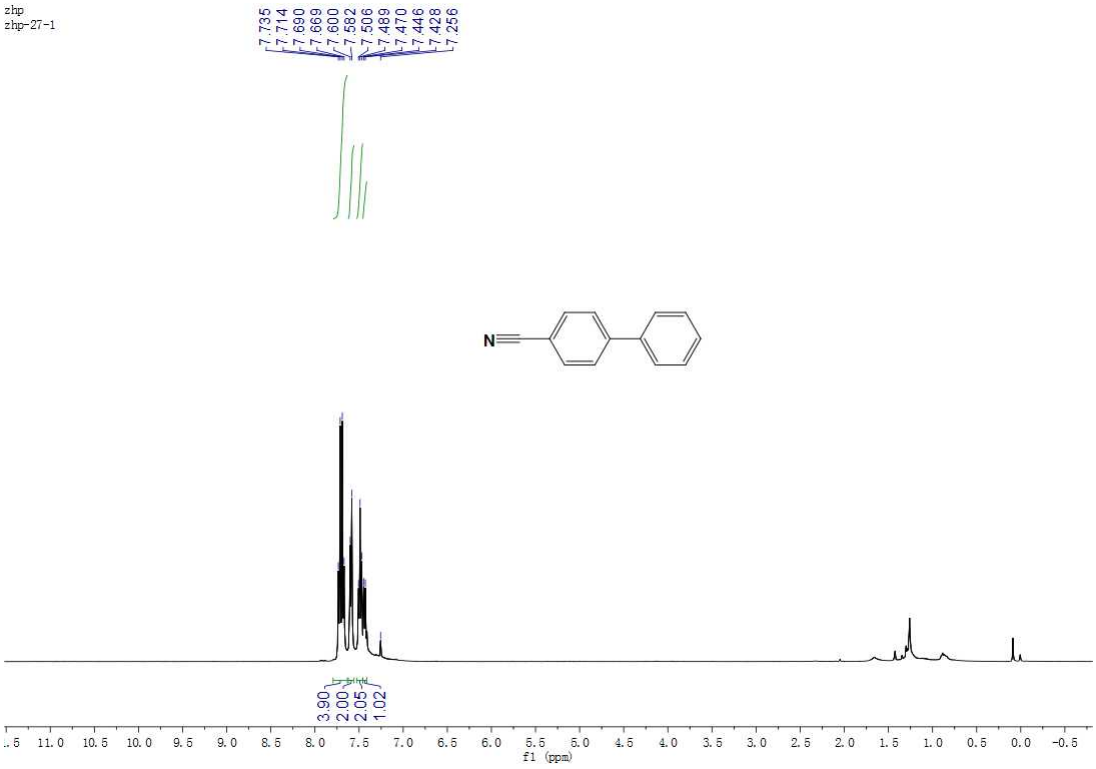


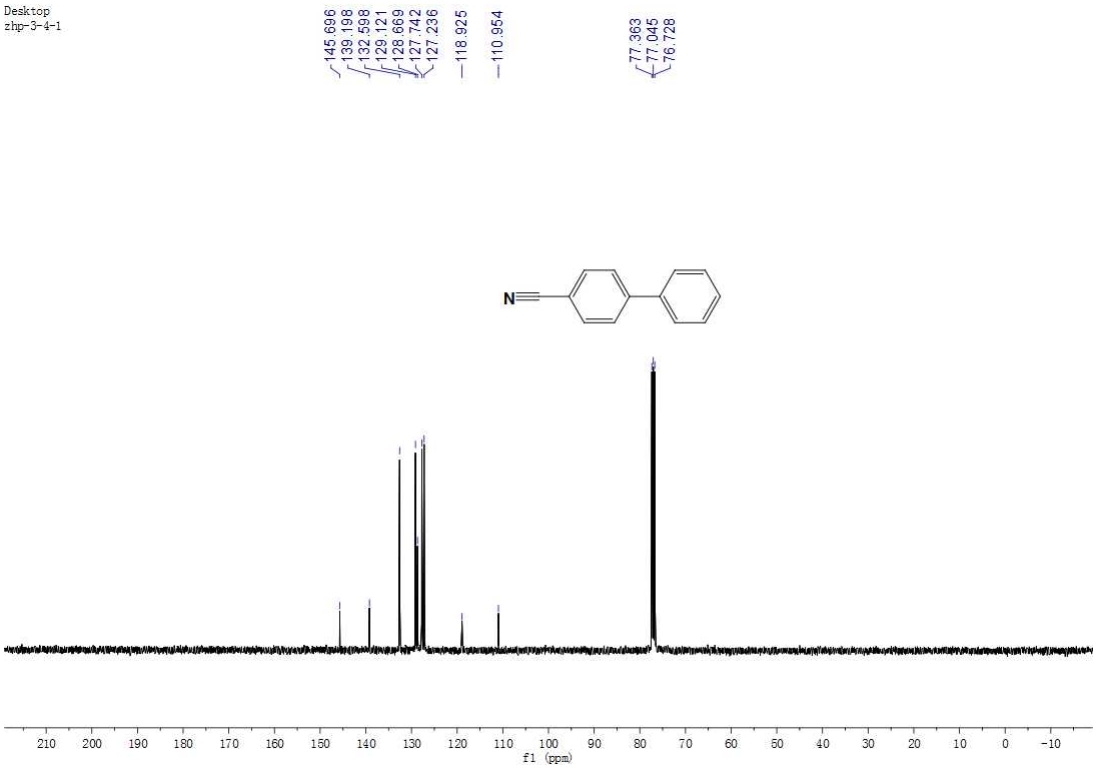

zhp
zhp-2-25-1
7.570
7.557
7.553
7.474
7.456
7.438
7.384
7.367
7.256
7.167
7.145
7.124
F
4.03
2.05
1.09
2.00
f1 (ppm)

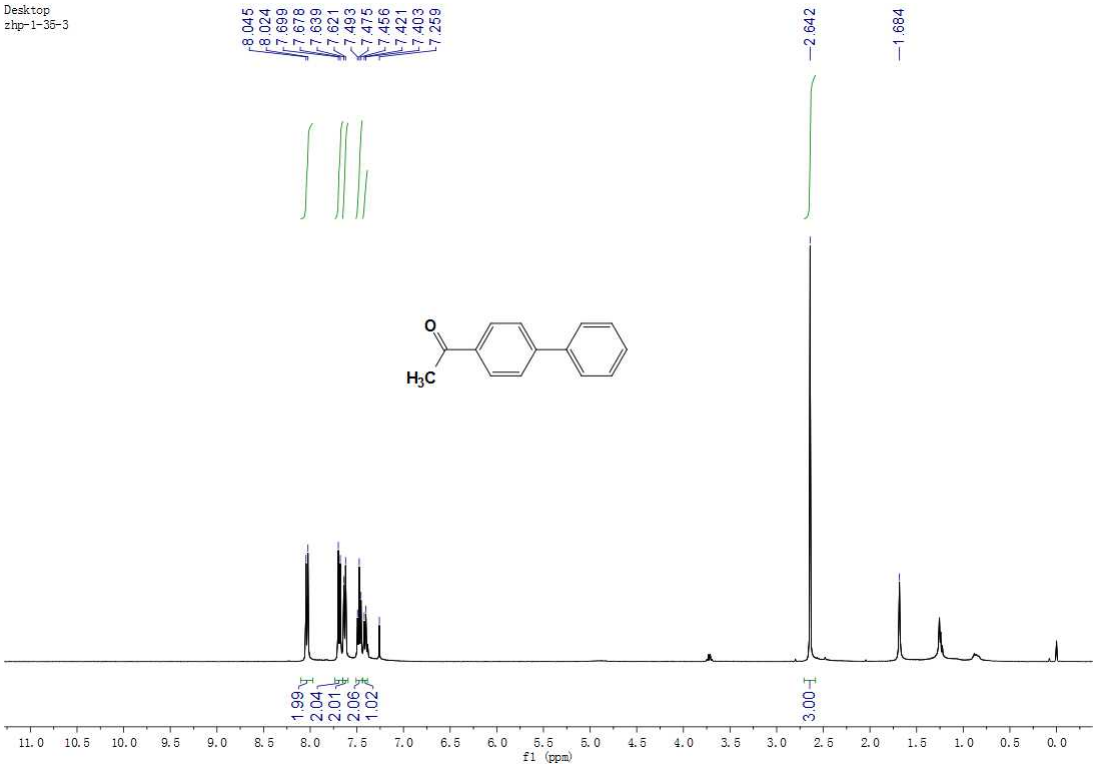

zhouheping
zhp-2-25-1
162.690
160.242
139.262
136.362
127.795
127.626
126.235
126.004
114.691
114.478
76.306
75.671
F
f1 (ppm)

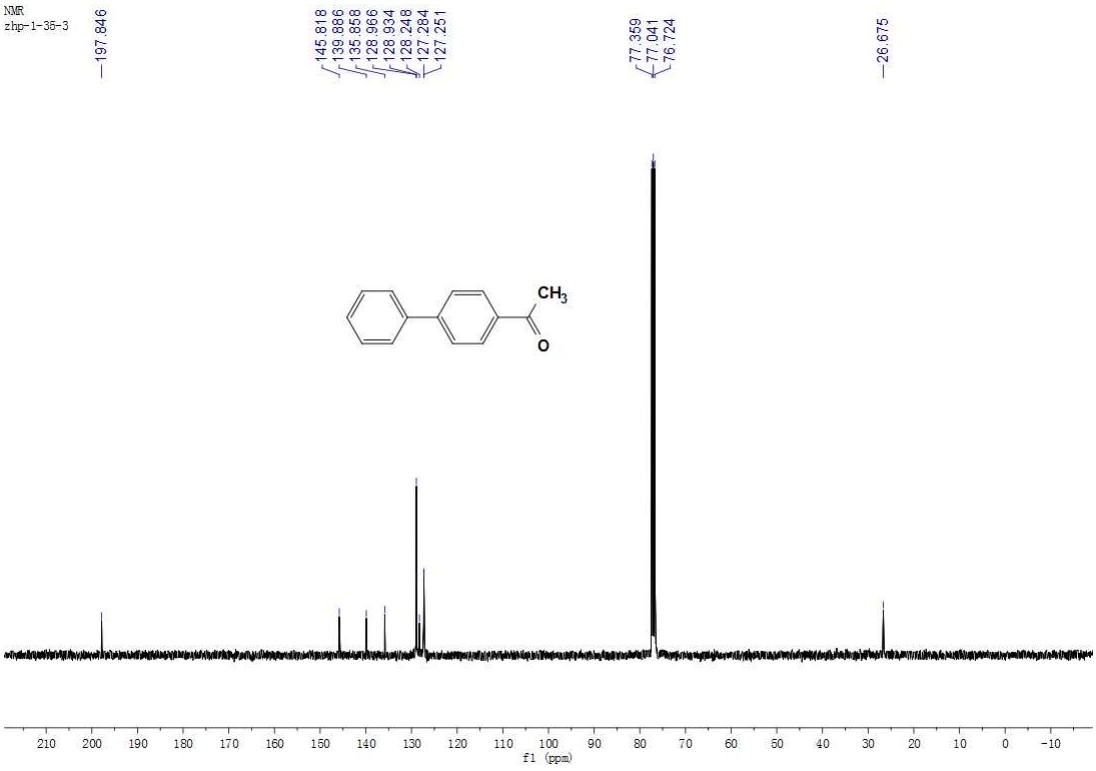

zhp
zhp-26-1
7.555
7.538
7.474
7.457
7.438
7.413
7.396
7.256
7.111
7.095
7.069
7.054
6.867
6.845
6.823
6.784
F
F
1.06
2.00
3.08
2.02
f1 (ppm)

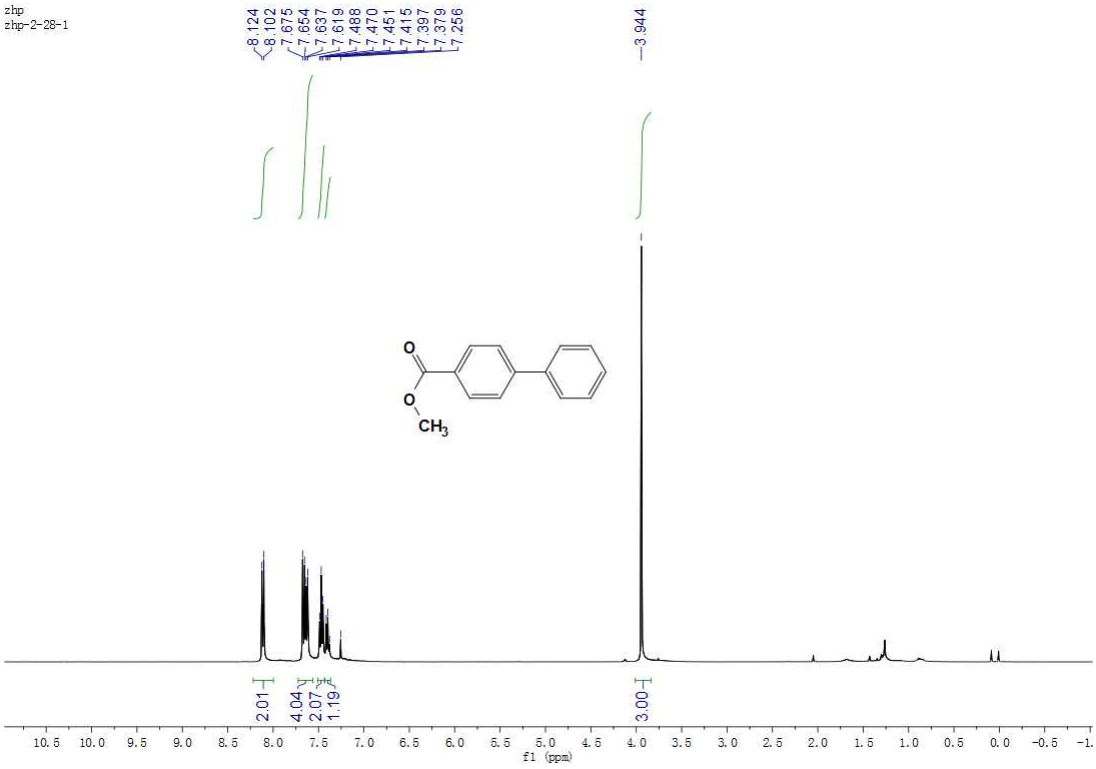

Desktop
zhp-3-11-1
164.662
164.532
162.198
162.068
144.703
144.609
144.515
138.933
129.025
128.461
127.014
110.069
109.999
109.884
109.814
102.730
102.477
102.224
77.369
77.051
76.734
F
F
f1 (ppm)

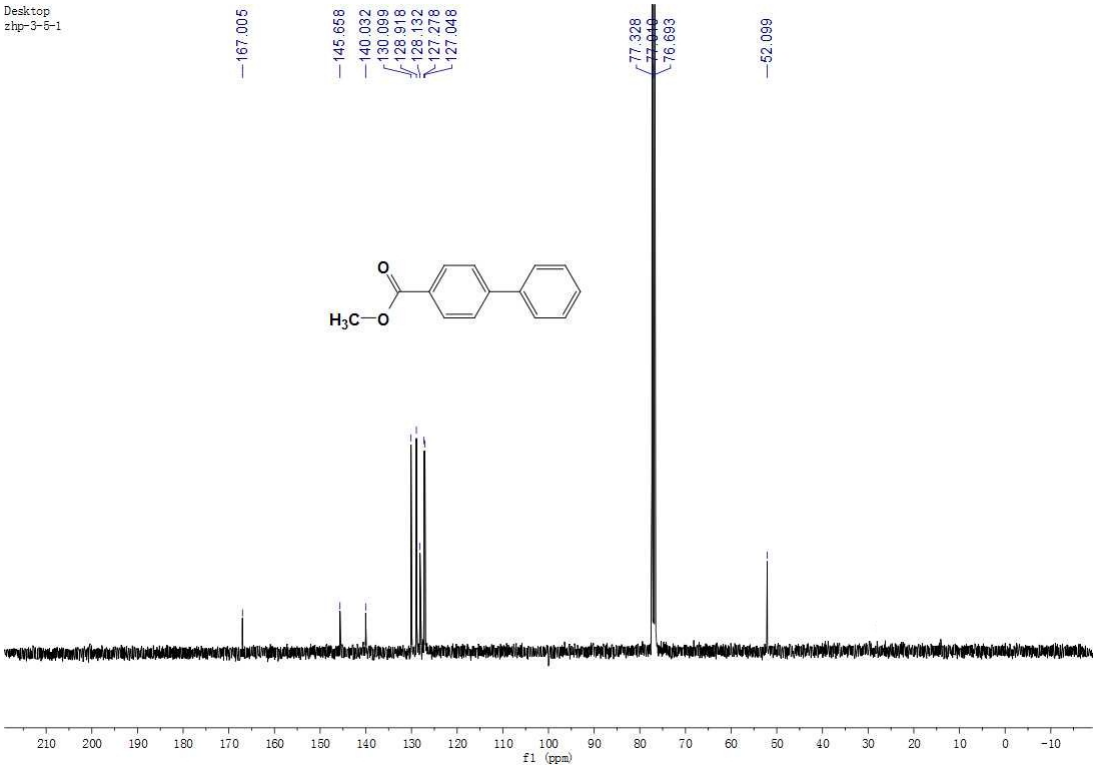

zhp
zhp-27-1
7.735
7.714
7.690
7.669
7.600
7.582
7.506
7.489
7.470
7.446
7.428
7.256
N
3.90
2.00
2.05
1.02
f1 (ppm)

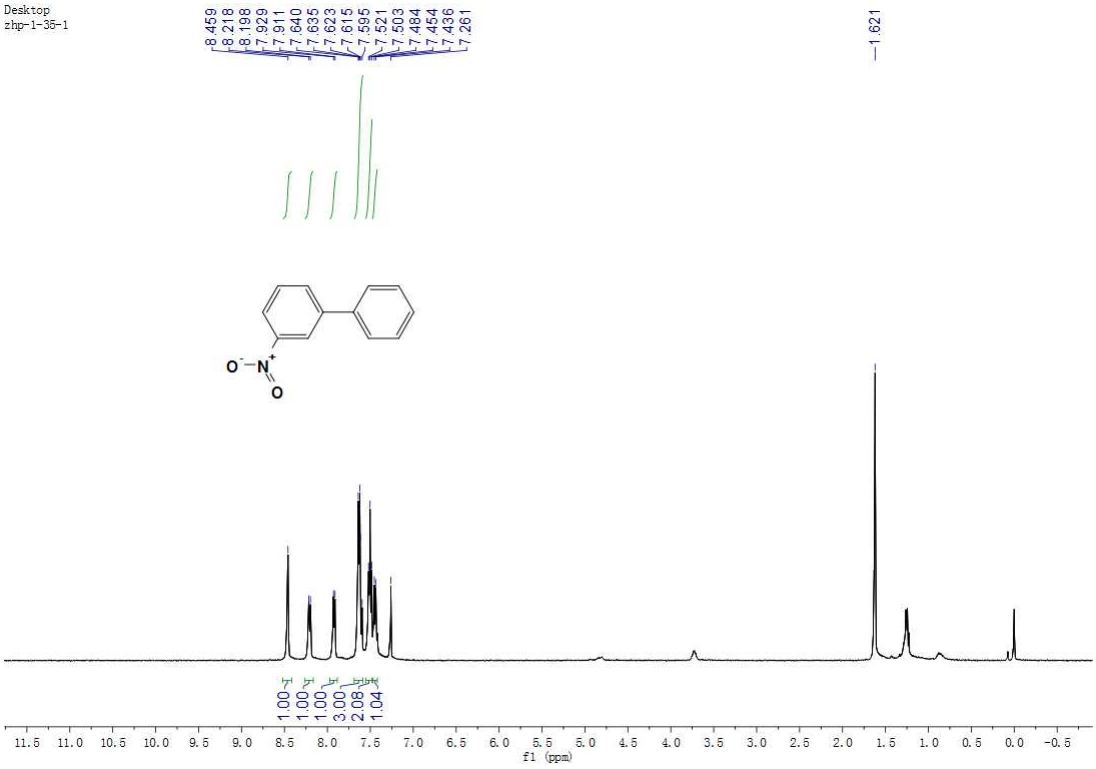

Desktop
zhp-3-4-1
145.696
139.198
132.598
129.121
128.669
127.742
127.236
118.925
110.954
77.363
77.045
76.728
N
f1 (ppm)

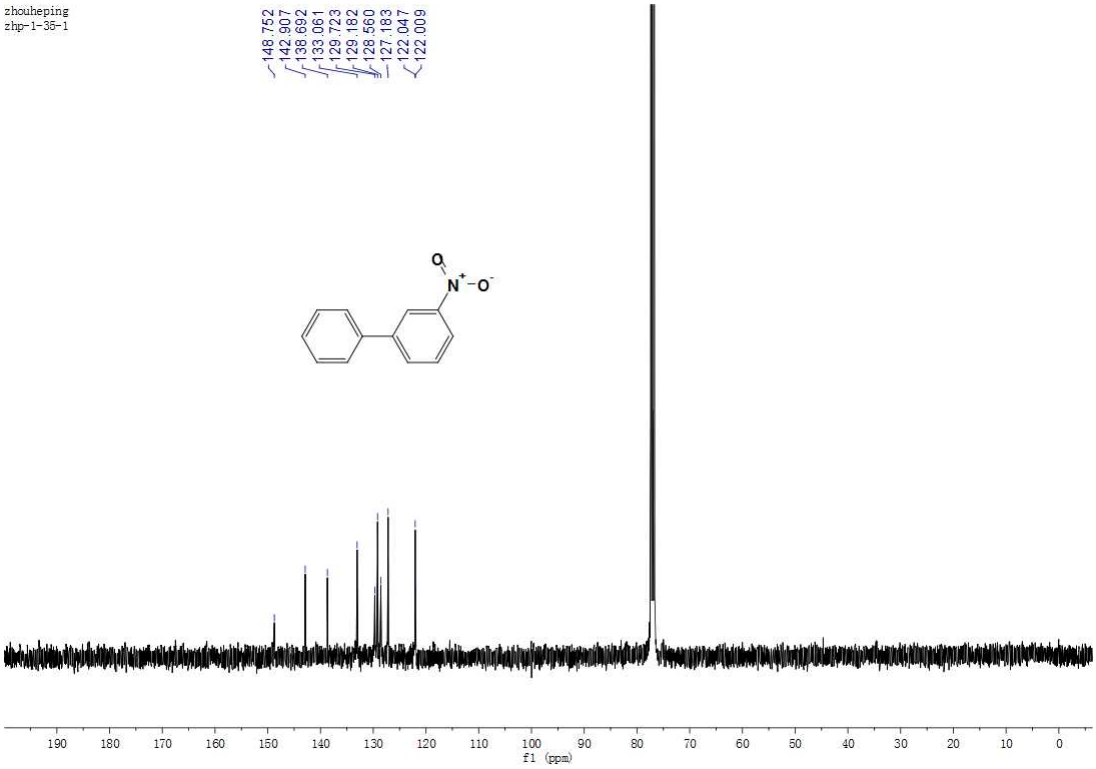

Desktop
zhp-1-35-3
8.045
8.024
7.699
7.678
7.639
7.621
7.493
7.475
7.456
7.421
7.403
7.259
2.642
1.684
O
H3C
1.99
2.04
2.01
2.06
1.02
3.00
f1 (ppm)

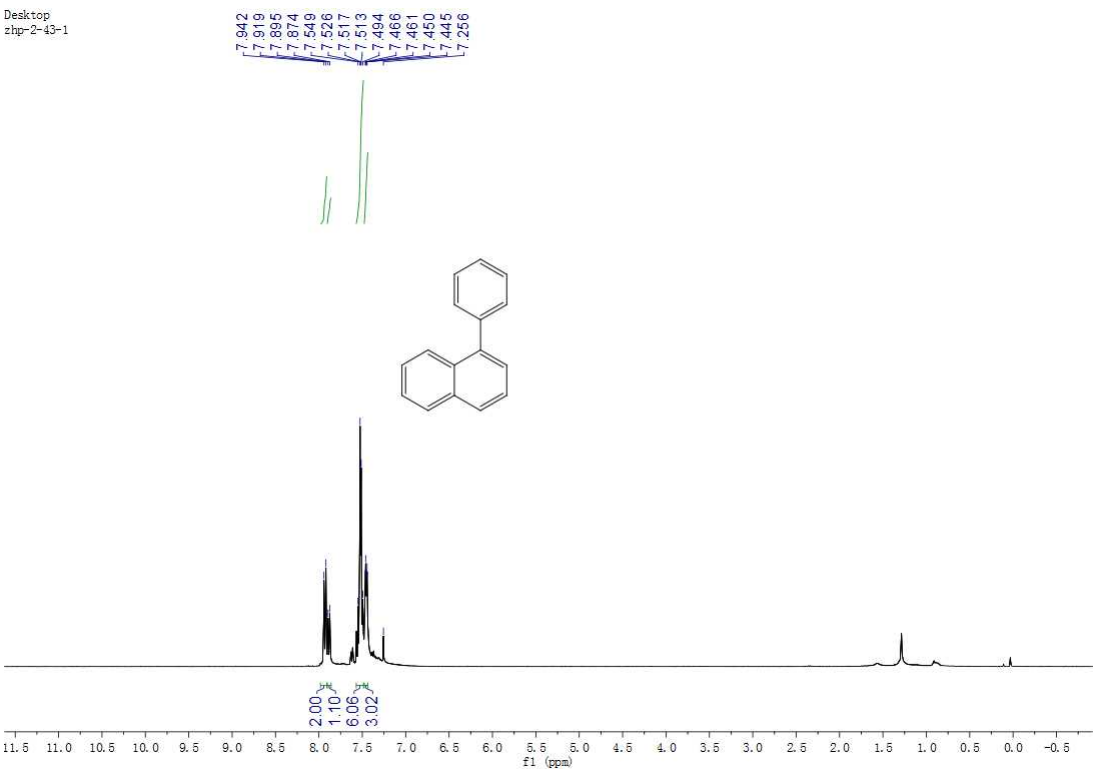

NMR
zhp-1-35-3
197.846
145.818
139.886
135.858
128.966
128.934
128.248
127.284
127.251
77.359
77.041
76.724
26.675
CH3
O
f1 (ppm)

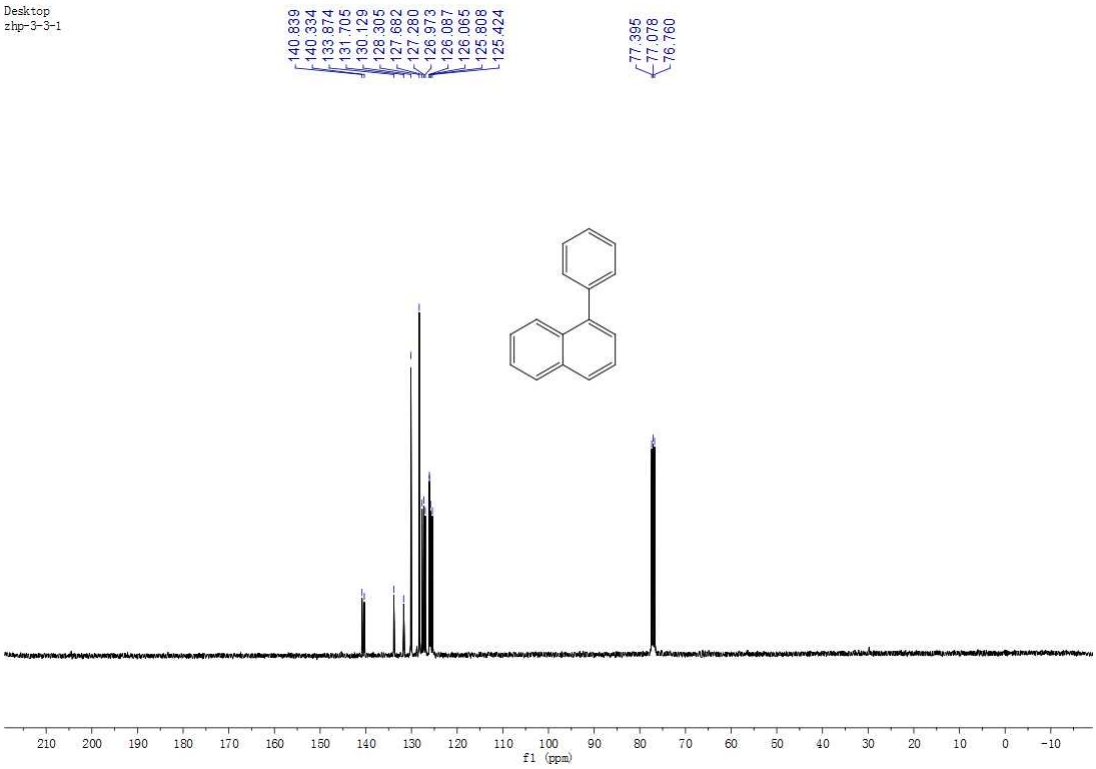

zhp
zhp-2-28-1
8.124
8.102
7.675
7.654
7.637
7.619
7.488
7.470
7.451
7.415
7.397
7.379
7.256
3.944
O
O
CH3
2.01
4.04
2.07
1.19
3.00
f1 (ppm)

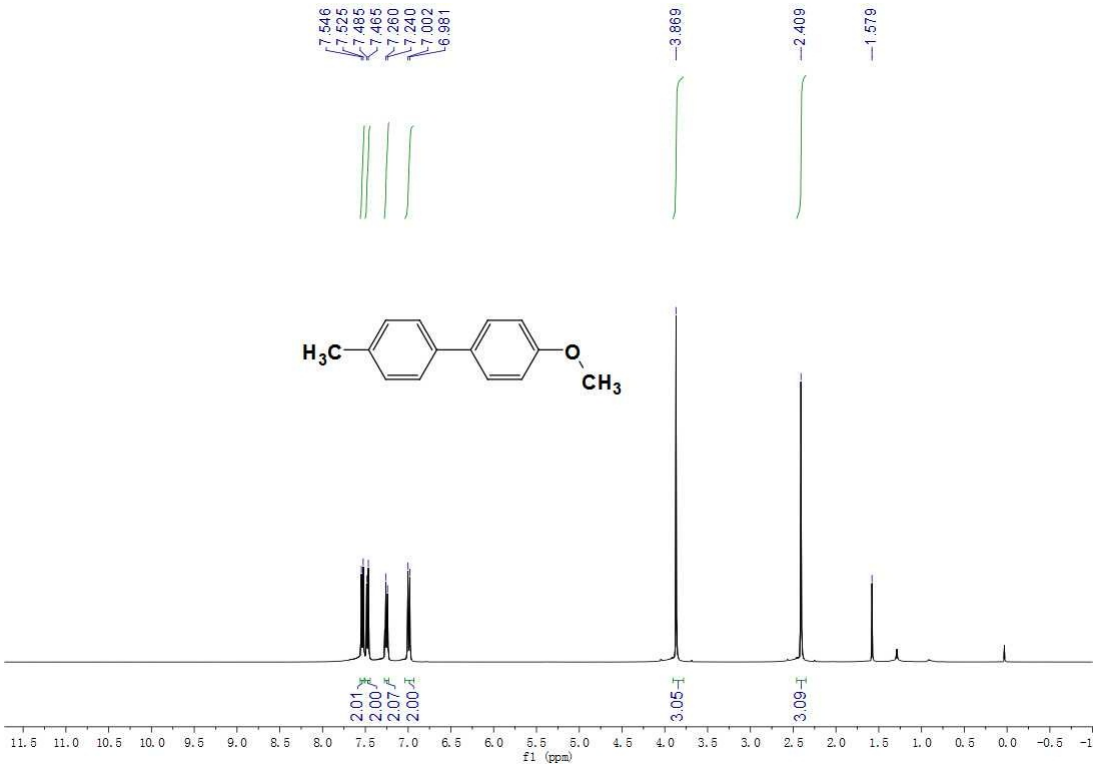

Desktop
zhp-3-5-1
167.005
145.658
140.032
130.099
128.918
128.132
127.278
127.048
77.328
77.010
76.693
52.099
O
H3C—O
f1 (ppm)

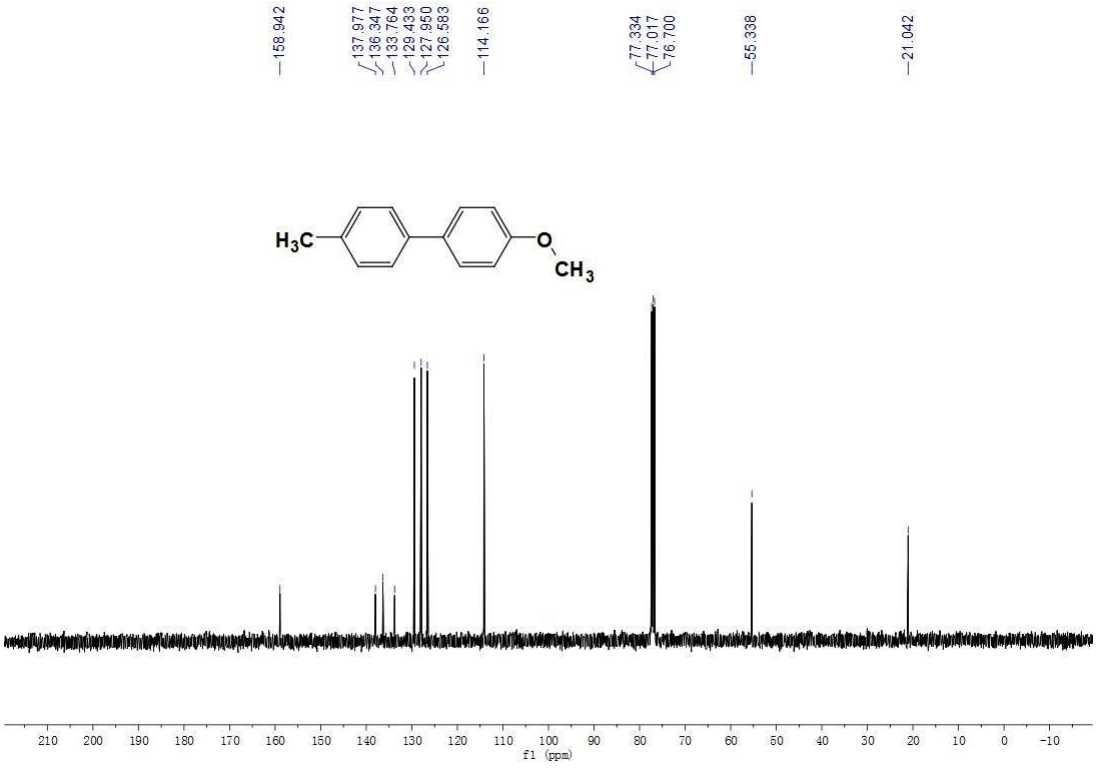

Desktop
zhp-1-35-1
8.459
8.218
8.198
7.929
7.911
7.640
7.635
7.623
7.615
7.595
7.521
7.503
7.484
7.454
7.436
7.261
1.621
1.00
1.00
1.00
3.00
2.08
1.04
f1 (ppm)

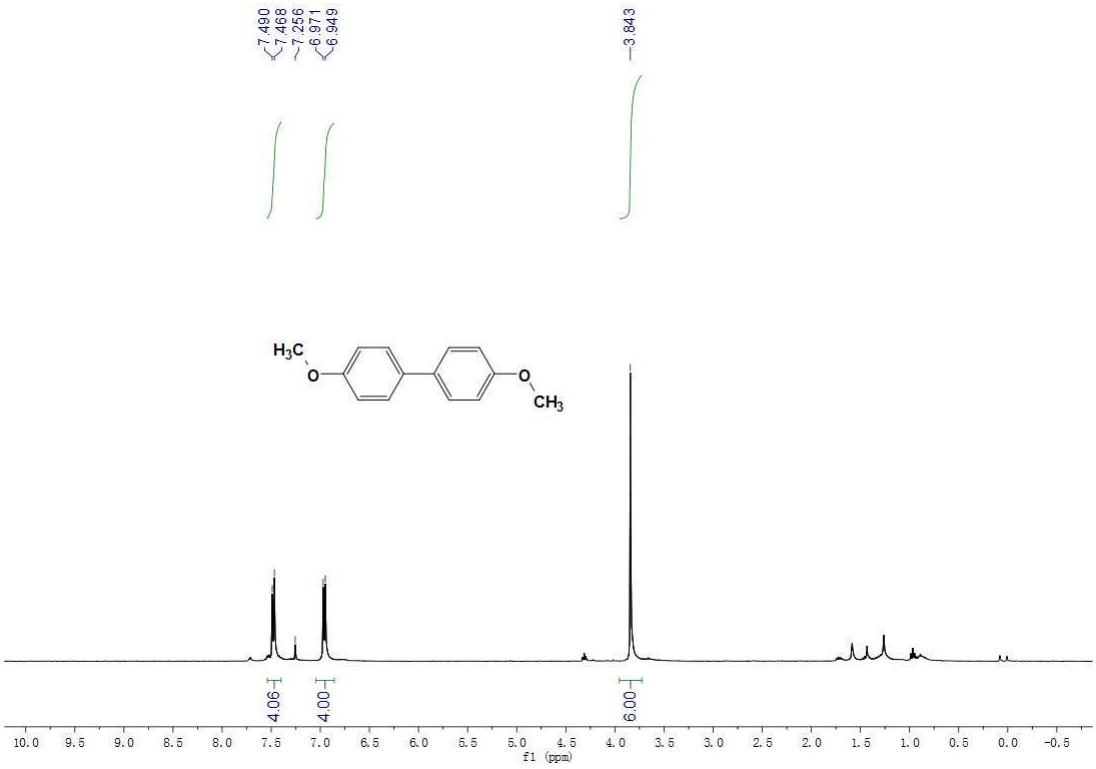

zhouheping
zhp-1-35-1
148.752
142.907
138.692
133.061
129.723
129.182
128.560
127.183
122.047
122.009
f1 (ppm)

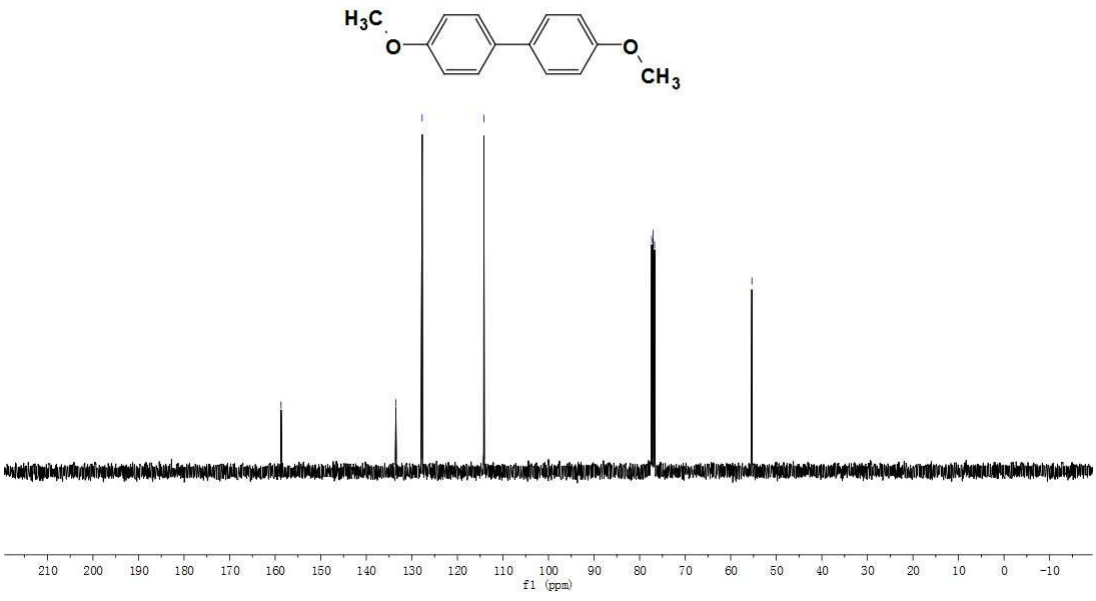

Desktop
zhp-2-43-1
7.942
7.919
7.895
7.874
7.549
7.526
7.517
7.513
7.494
7.466
7.461
7.450
7.445
7.256
2.00
1.10
6.06
3.02
f1 (ppm)

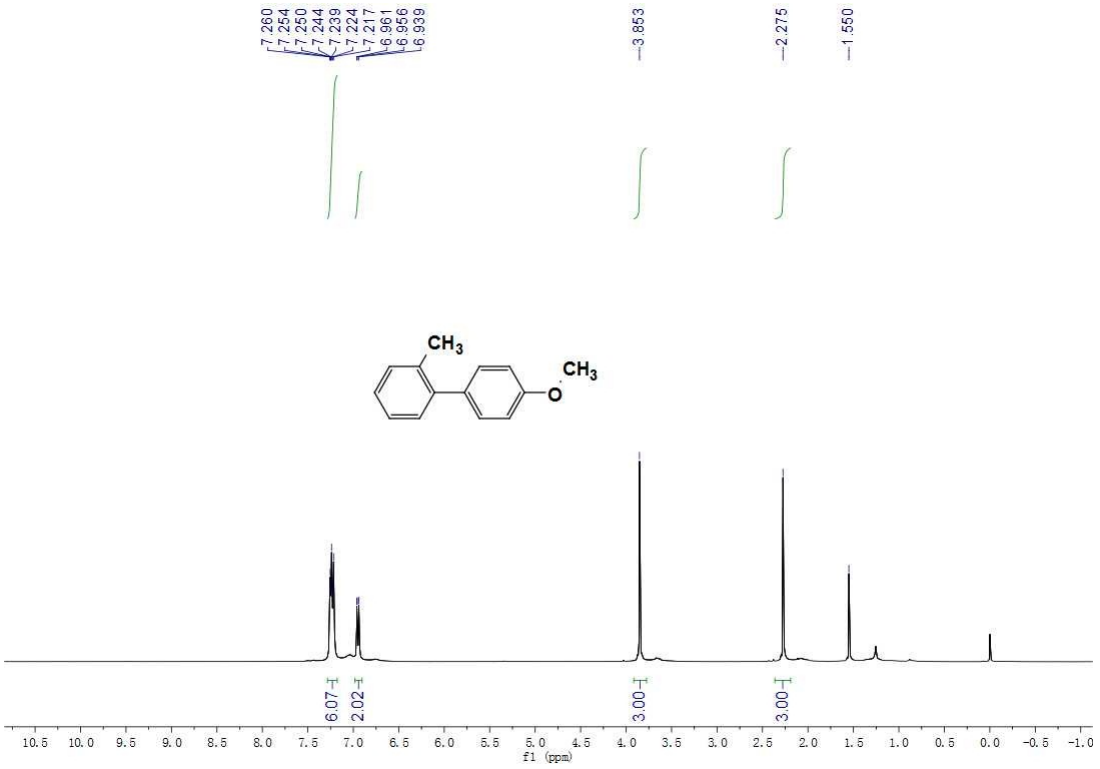

Desktop
zhp-3-3-1
140.839
140.334
133.874
131.705
130.129
128.305
127.682
127.280
126.973
126.087
126.065
125.808
125.424
77.395
77.078
76.760
f1 (ppm)

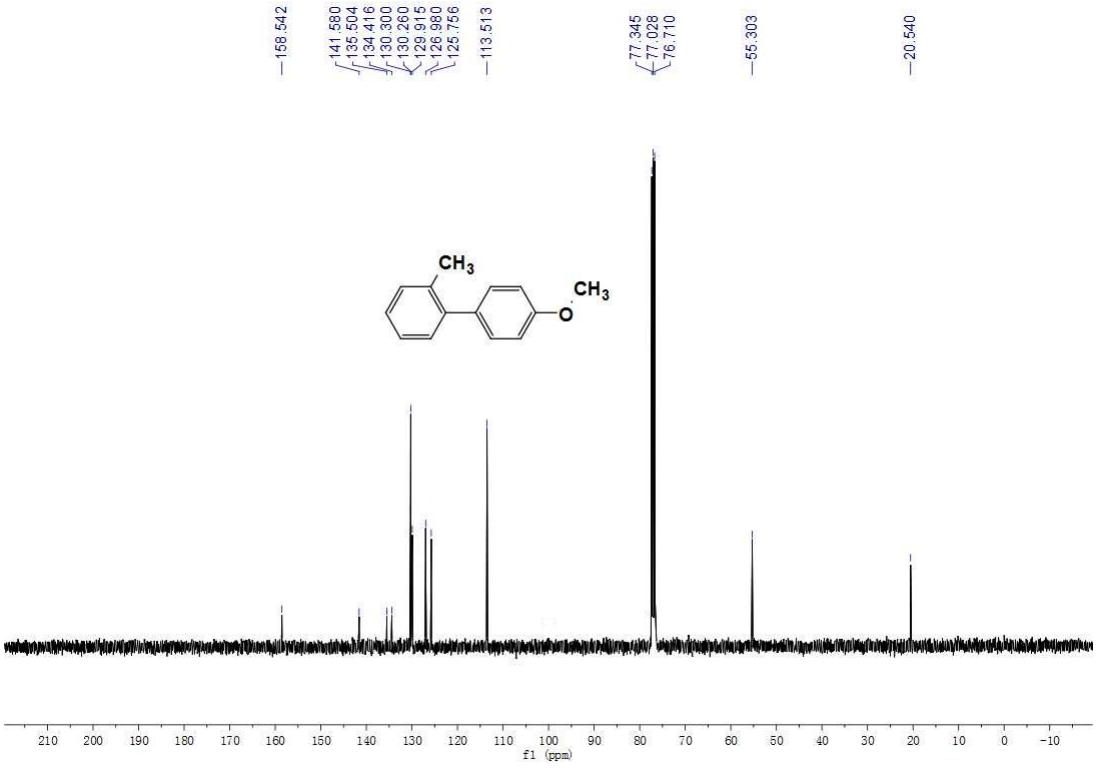

7.546
7.525
7.485
7.465
7.260
7.240
7.002
6.981
3.869
2.409
1.579
H3C
O
CH3
2.01
2.00
2.07
2.00
3.05
3.09
f1 (ppm)

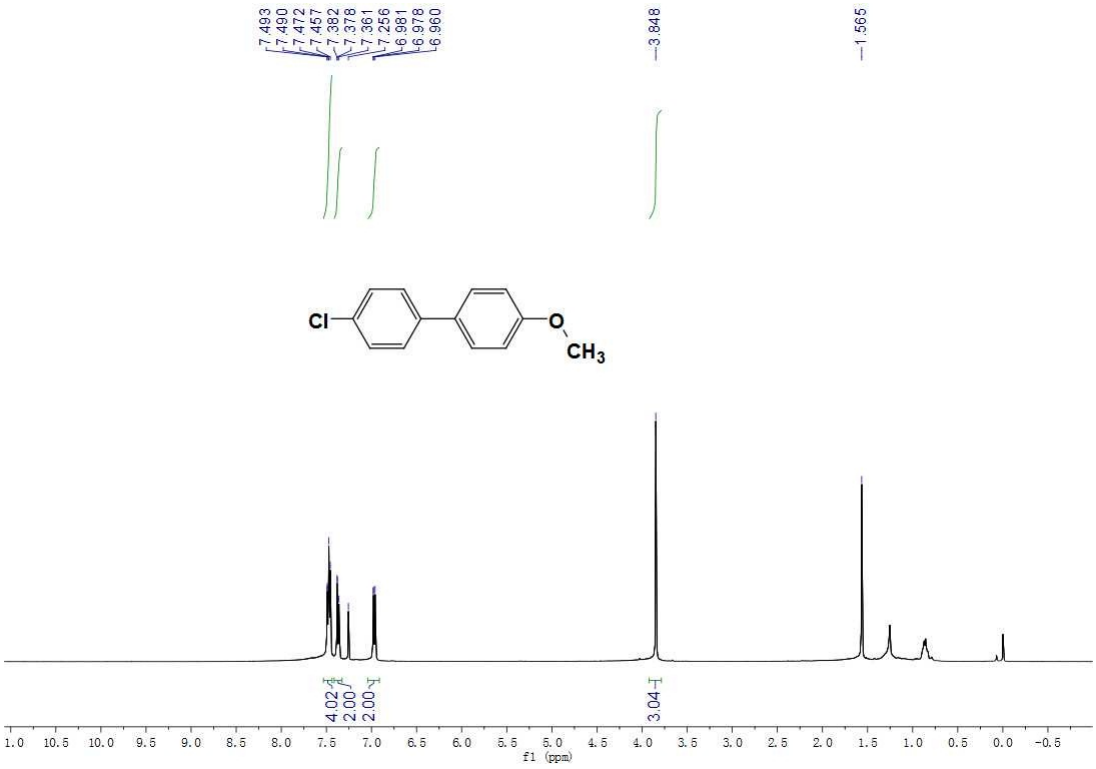

158.942
137.977
136.347
133.764
129.433
127.950
126.583
114.166
77.334
77.017
76.700
55.338
21.042
H3C
O
CH3
f1 (ppm)

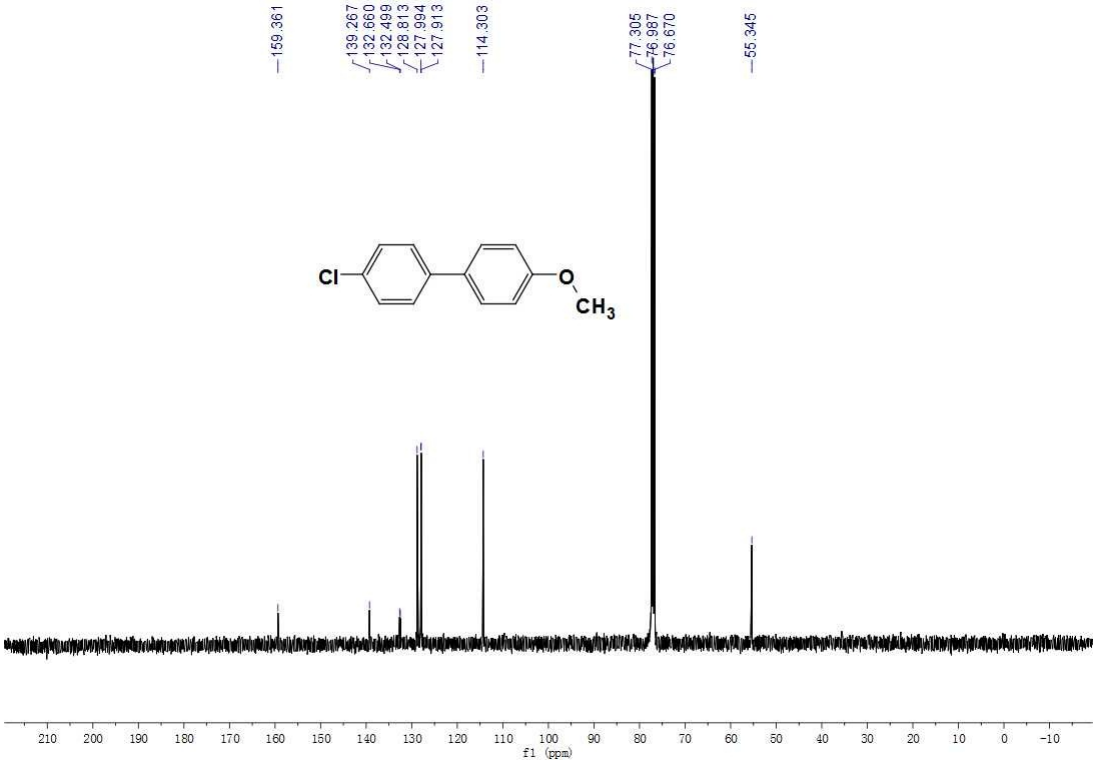

7.490
7.468
7.256
6.971
6.949
3.843
4.06
4.00
6.00
f1 (ppm)

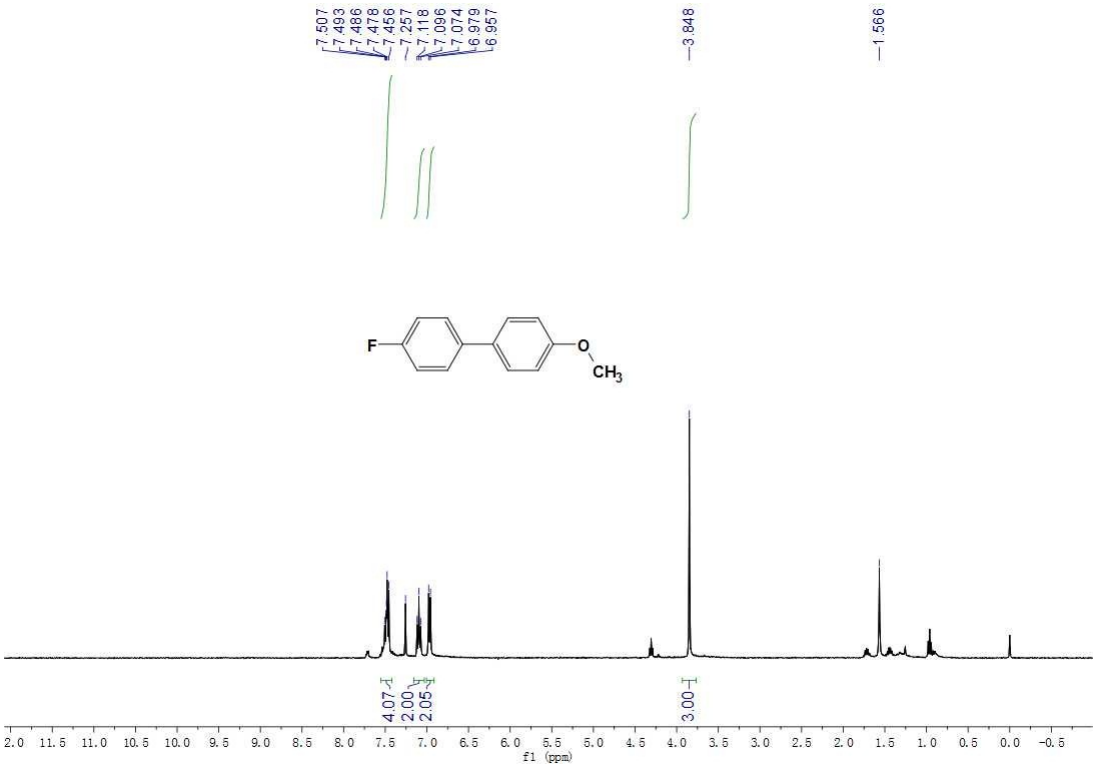

158.716
133.506
127.740
114.182
77.345
77.028
76.711
55.352
f1 (ppm)

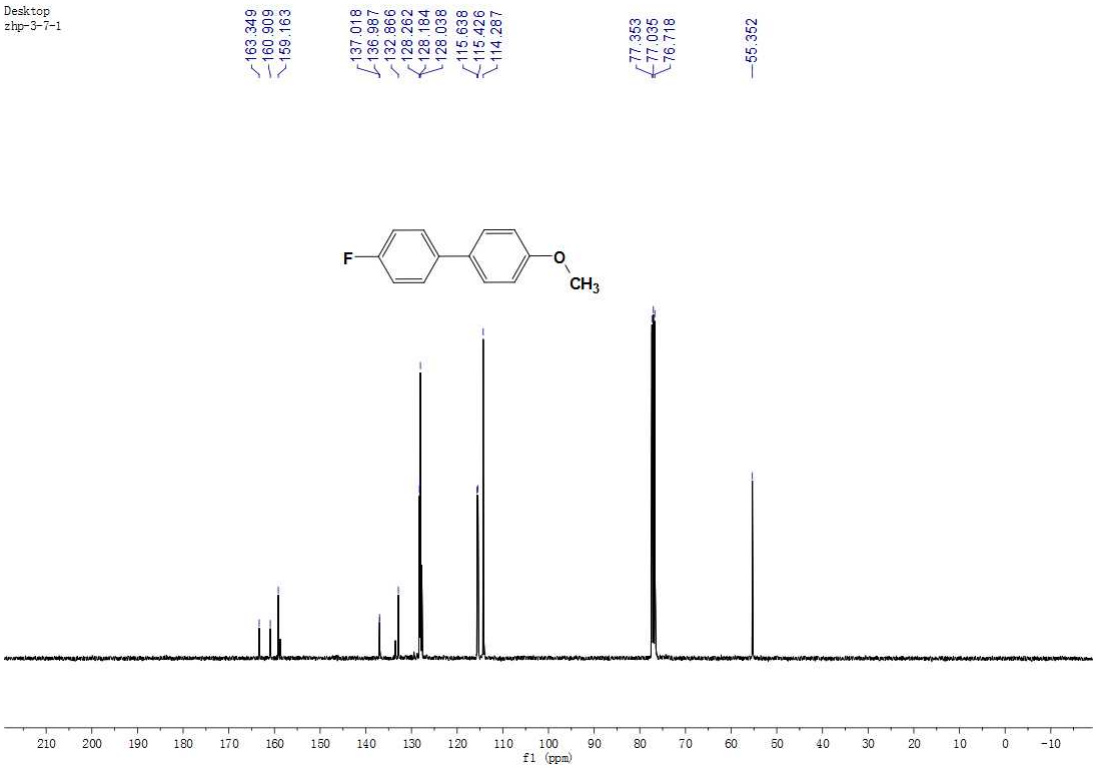

7.260
7.254
7.250
7.244
7.239
7.224
7.217
6.961
6.956
6.939
3.853
2.275
1.550
CH3
CH3
O
6.07
2.02
3.00
3.00
f1 (ppm)

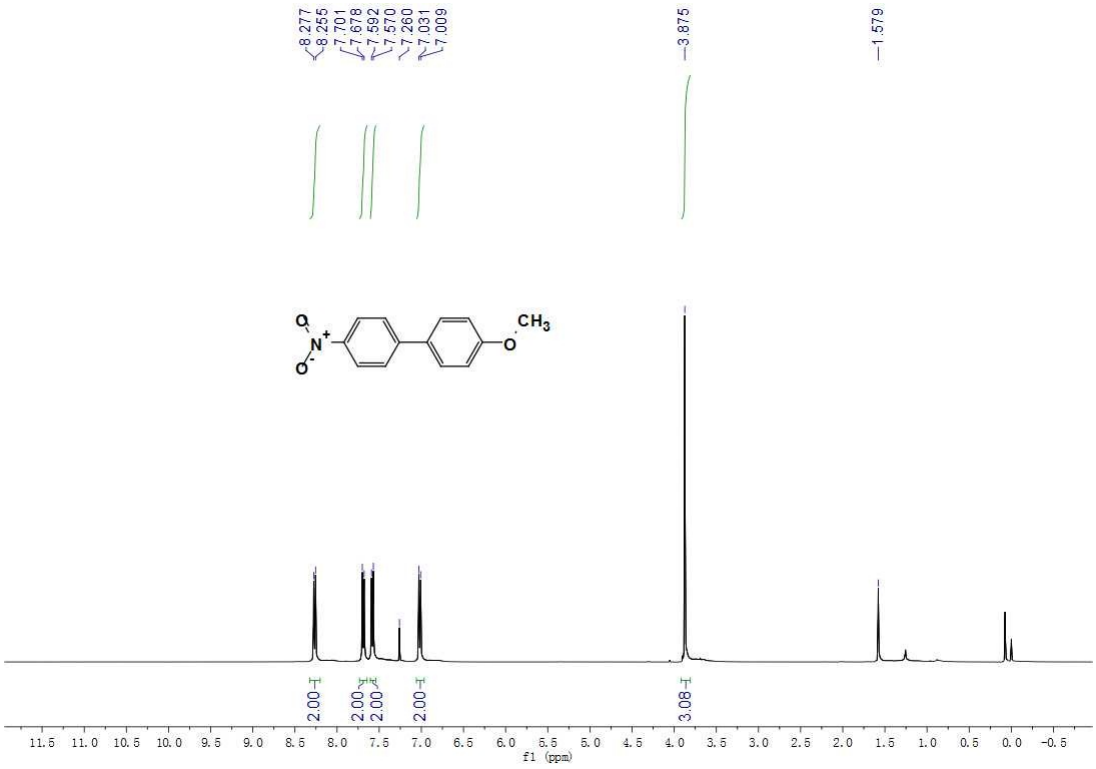

158.542
141.580
135.504
134.416
130.300
130.260
129.915
126.980
125.756
113.513
77.345
77.028
76.710
55.303
20.540
CH3
CH3
O
f1 (ppm)

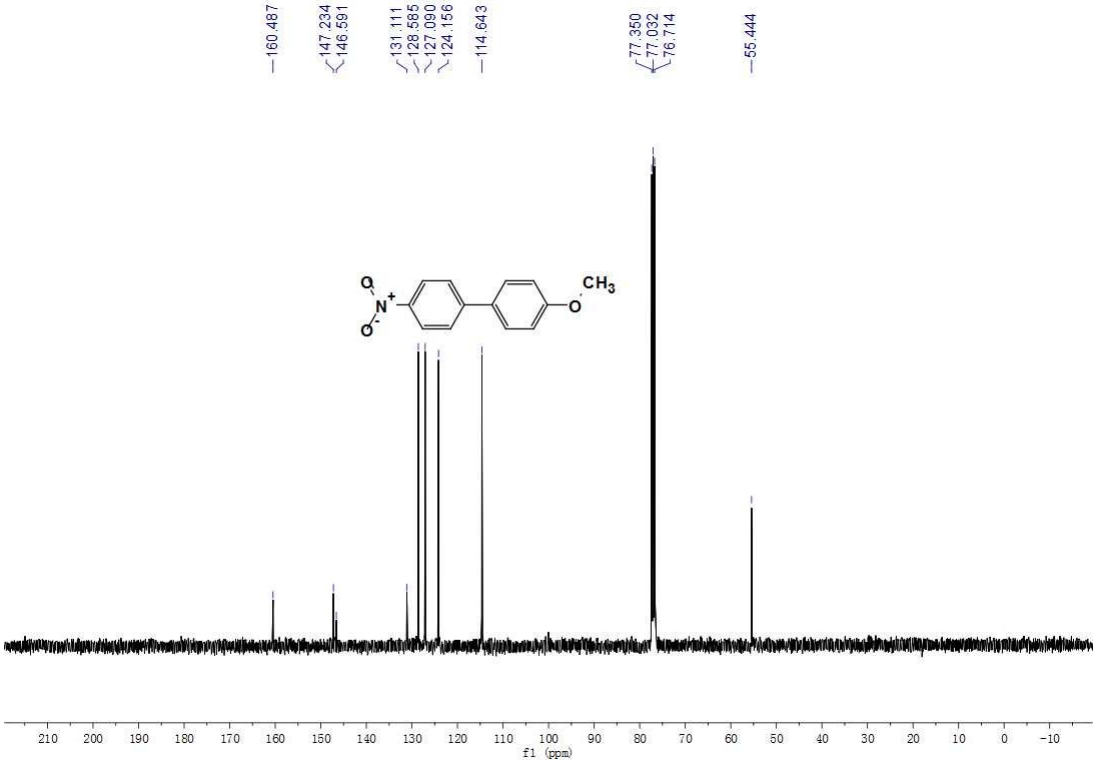

7.493
7.490
7.472
7.457
7.382
7.378
7.361
7.256
6.981
6.978
6.960
3.848
1.565
Cl
O
CH3
4.02
2.00
2.00
3.04
f1 (ppm)


159.361
139.267
132.660
132.499
128.813
127.994
127.913
114.303
77.305
76.987
76.670
55.345
Cl
O
CH3
f1 (ppm)


7.507
7.493
7.486
7.478
7.456
7.257
7.118
7.096
7.074
6.979
6.957
3.848
1.566
F
O
CH3
4.07
2.00
2.05
3.00
f1 (ppm)


Desktop
zhp-3-7-1
163.349
160.909
159.163
137.018
136.987
132.866
128.262
128.184
128.038
115.638
115.426
114.287
77.353
77.035
76.718
55.352
F
O
CH3
f1 (ppm)


8.277
8.255
7.701
7.678
7.592
7.570
7.260
7.031
7.009
3.875
1.579
2.00
2.00
2.00
2.00
3.08
f1 (ppm)


160.487
147.234
146.591
131.111
128.585
127.090
124.156
114.643
77.350
77.032
76.714
55.444
f1 (ppm)