Supplementary Data

Solvent-Free N-Arylation of Amines with Arylboronic Acids under Ball Milling Conditions

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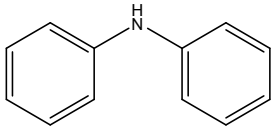
1. General methods

The arylboronic acids, amines, bases, and organo catalysts were all purchased from commercial sources. All reactions were carried out in an AGO-2 planetary-centrifugal mill (volume of one drum: 150 mL; diameter of stainless steel ball: 8 mm, 9mm and 4mm; weight of balls: 175 g) and monitored by TLC. The milling cycle was with a rotational speed of 1290 rpm for 15 min cooling down by water and followed by a 10 min pause for 6 times. Reaction products were purified by silica gel chromatography (300–400 mesh). ^{1}H-NMR and ^{13}C-NMR spectra were obtained on a Varian Mercury plus-400 spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C NMR spectroscopy) by using $CDCl_3$ as the solvent and TMS as the internal standard. Chemical shifts for ^{1}H and ^{13}C NMR were referred to internal TMS (0 ppm) and J-values were shown in Hz. Mass spectra were measured with MS instrument using ESI or EI ionization.

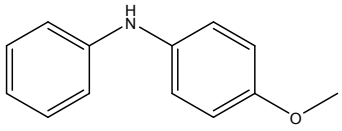
2. General Procedure for Preparation of 4-methyl-*N*-phenylaniline (3aa).

A mixture of substrate arylboronic acid (5 mmol), *p*-methylaniline (7 mmol), $Cu(OAc)_2 \cdot H_2O$ (1 equiv), K_2CO_3 (2.5 equiv) and silica gel (2.5 g) were added to the screw-capped stainless steel vial, along with nearly 50 big stainless steel balls (8 mm and 9mm) and 60 small steel balls (4 mm) up to 175g. Then, the vial was placed in the AGO-2 planetary-centrifugal mill, and the contents were ball milled. At the end of the experiment, all of the reaction mixture was scratched off the vessel, dissolved in ethyl ether, followed by washing with brine and the organic layers were dried over anhydrous sodium sulfate and concentrated in vacuo to give a residue, which was purified by column chromatography on silica gel (eluents: petroleum ether/ethyl acetate 30:1) to provide the desired product 3**aa**. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.20 (t, J = 7.6 Hz, 2H), 7.05 (d, J = 8.1 Hz, 2H), 7.01-6.94 (m, 4H), 6.85 (t, J = 7.3 Hz, 1H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): 143.7, 140.1, 130.6, 129.1, 120.1, 118.7, 116.7, 20.7.

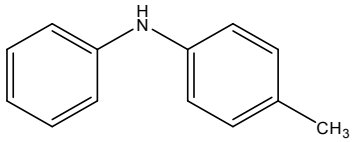
3. ^{1}H NMR, ^{13}C NMR and Mass Spectral data



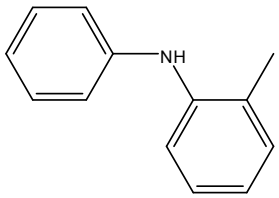
Diphenylamine (3aa). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 （t, *J* = 7.0 Hz, 4H ）, 7.07 (d, *J* = 7.7 Hz, 4H), 6.92 (t, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 142.9, 129.1, 120.8, 117.6; MS (ESI): m/z = 168 (M^+ - H).



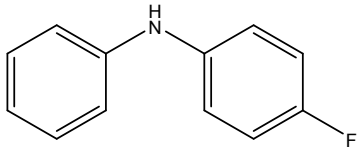
4-methoxy-*N*-phenylaniline (3ab)[1]. ^{1}H NMR (400 MHz, $CDCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 6.88 (d, *J* = 7.9 Hz, 2H), 6.86 – 6.77 (m, 3H),5.49 (br s, 1H, NH), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.2, 145.0, 135.7, 129.2, 122.1, 119.5, 115.7, 114.6, 55.7; MS (ESI): m/z = 199 (M^+ + H).



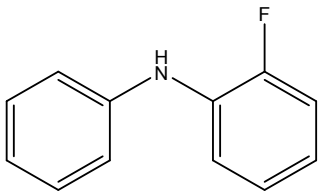
4-methyl-*N*-phenylaniline (3ac). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (t, *J* = 7.6 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 7.01-6.94 (m, 4H), 5.56 (br s, 1H, NH) , 6.85 (t, *J* = 7.3 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 143.7, 140.1, 130.6, 129.1, 120.1, 118.7, 116.7, 20.7; MS (ESI): m/z = 184 (M^+ + H).



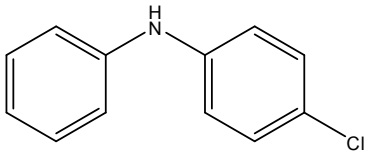
2-methyl-*N*-phenylaniline (3ad)[1]. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 – 7.20 (m, 3H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 7.1 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 4.3 Hz, 1H), 6.88 (t, *J* = 6.5 Hz, 1H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 141.0, 130.7, 129.1, 128.2, 126. 6, 121.9, 120.3, 118.8, 117.2, 18.0; MS (ESI): m/z = 184 (M^+ + H).



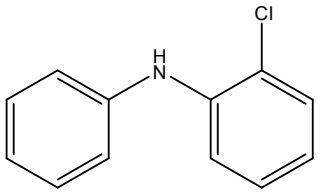
4-fluoro-*N*-phenylaniline (3ae). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (t, *J* = 7.9 Hz, 2H), δ 7.06 – 7.00 (m, 2H), 6.99 – 6.92 (m, 4H), 6.88 (t, *J* = 7.3 Hz, 1H), 5.56 (br s, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 157.8, 143.8, 138.8, 129.2, 120.5, 116.8, 115.8; MS (ESI): m/z = 188 (M^+ + H).



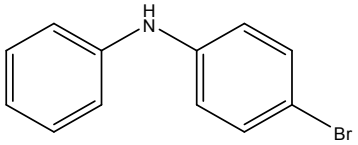
2-fluoro-*N*-phenylaniline (3af). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.24 (m, 3H), 7.09 (dd, *J* = 8.4, 0.9 Hz, 2H), 7.07 – 6.92 (m, 3H), 6.86 – 6.75 (m, 1H), 5.76 (br s, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 152.8, 141.9, 131.6, 129.2, 124.1, 121.7, 120.4, 118.6, 117.1, 115.3; MS (ESI): m/z = 188 (M^+ + H).



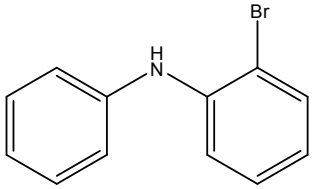
4-chloro-*N*-phenylaniline (3ag). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 (t, *J* = 7.6 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.5 Hz, 2H), 6.97 (d, *J* = 8.7 Hz, 2H), 6.94 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 142.5, 141.7, 129.2, 129.1, 125.3, 121.4, 118.7, 118.0; MS (ESI): m/z = 204 (M^+ + H).



2-chloro-*N*-phenylaniline (3ah). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.25 (m, 4H), 7.14 (d, *J* = 8.3 Hz, 2H), 7.09 (t, *J* = 8.2 Hz, 1H), 7.02 (t, *J* = 7.3 Hz, 1H), 6.78 (dd, *J* = 10.9, 4.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 141.4, 140.2, 129.6, 129.3, 127.3, 122.5, 121.4, 120.2, 120.1, 115.5; MS (ESI): m/z = 204 (M^+ + H).

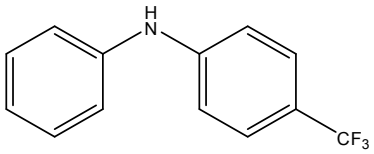


4-bromo-*N*-phenylaniline (3ai). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.8 Hz, 2H), 7.25 (t, *J* = 8.3 Hz, 2H), 7.03 (d, *J* = 8.2 Hz, 2H), 6.95 (t, *J* = 5.6 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 142.3, 132.0, 129.3, 121.5, 118.2, 112.5; MS (ESI): m/z =248, 250 (M^+).

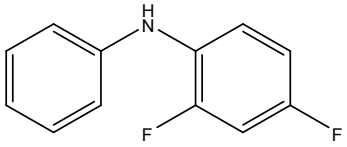


2-bromo-*N*-phenylaniline (3aj). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.22 (m, 1H), 7.18 – 7.10 (m, 3H), 7.02 (t, *J* = 7.3 Hz, 1H), 6.72 (dd, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): ^{13}C NMR

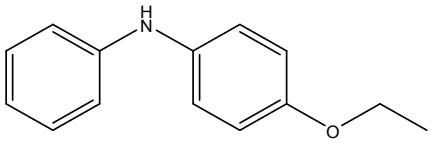
(101 MHz, $CDCl_3$) δ 141.4, 141.3, 132.8, 129.3, 127.9, 122.6, 120.8, 120.2, 115.7, 112.1. MS (ESI): m/z = 248, 250 (M^+).



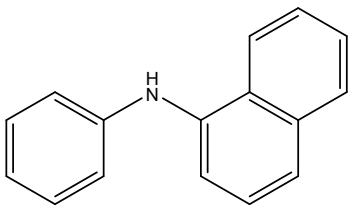
***N*-phenyl-4-(trifluoromethyl)aniline (3ak)**[2]. ^1H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.31 (t, *J* =7.9 Hz, 1H), 7.16 – 7.11 (m, 2H), 7.07 – 6.99 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 146.6, 141.0, 129.4, 126.5, 122.8, 119.9, 115.2; MS (ESI): m/z = 236 (M^+ - H).



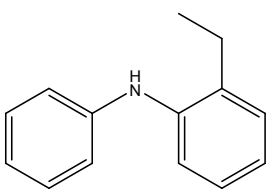
2,4-difluoro-*N*-phenylaniline (3al)[3]. ^1H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.20 (m, 3H), 6.99 (dd, *J* = 8.6, 1.0 Hz, 2H), 6.94 (tt, *J* = 7.3, 1.1, 1H), 6.86 (ddd, *J* = 11.1, 8.4, 2.8 Hz, 1H), 6.77 (dddd, *J* = 9.0, 8.0, 2.8, 1.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 156.1, 153.7, 142.5, 129.3, 127.5, 121.3, 119.3, 117.5, 110.7, 104.1; MS (ESI): m/z = 204 (M^+ - H).



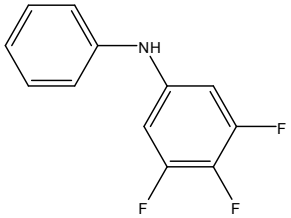
4-ethoxy-*N*-phenylaniline (3am). ^1H NMR (400 MHz, $CDCl_3$): δ 7.19 (t, *J* = 7.6 Hz, 2H), 7.05 (s, br, 2H), 6.89 (s, br, 2H). 6.86 – 6.80 (m, 3H), 4.00 (d, *J* = 6.6 Hz, 2H), 1.41 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.4, 145.0, 135.5, 129.1, 122.0, 119.4, 115.5, 115.2, 63.8, 15.0; MS (ESI): m/z = 214 (M^+ + H).



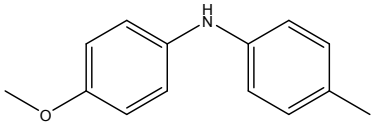
***N*-phenylnaphthalen-1-amine (3an)**[4]. ^1H NMR (400 MHz, $CDCl_3$): δ 8.03 – 7.98 (m, 1H), 7.87 – 7.82 (m, 1H), 7.55 (d, *J* = 7.0 Hz, 1H), 7.47 (tt, *J* = 6.8, 5.2 Hz, 2H), 7.41 – 7.34 (m, 2H), 7.24 (t, *J* =7.9 Hz, 2H), 6.98 (d, *J* = 7.6 Hz, 2H), 6.90 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 144.7, 138.7, 134.6, 129.2, 128.4, 127.7, 125.9, 125.5, 122.9, 121.7, 120.4, 117.3, 115.9; MS (ESI): m/z = 220 (M^+ + H).



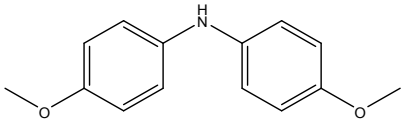
2-ethyl-*N*-phenylaniline (3ao). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 – 7.22 (m, 4H), 7.16 (td, *J* = 7.7, 1.6 Hz, 1H), 7.02 (td, *J* = 7.4, 1.3 Hz, 1H), 6.95 (dd, *J* = 8.6, 1.0 Hz, 2H), 6.90 (tt, *J* = 7.3 Hz, *J* = 1.1 Hz, 1H), 5.44 (br s, 1H, NH), 2.65 (q, *J* = 7.5 Hz, 2H), 1.28 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.3, 140.3, 134.6, 129.1, 128.8, 126.5, 122.5, 120.0, 120.0, 116.92, 24.38, 14.00.



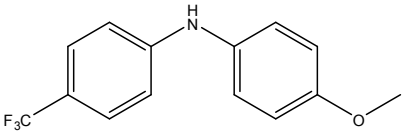
3,4,5-trifluoro-*N*-phenylaniline (3ap)[5]. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (t, *J* = 7.7 Hz, 2H), 7.04 (d, *J* = 7.7 Hz, 2H), 7.03–6.97 (m, 1H), 6.58 (dd, *J* = 9.9, 5.8 Hz, 2H), 5.61 (br s, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6 , 141.1, 139.6, 133.9 , 129.5, 122.8, 119.5, 100.4; MS (ESI): m/z = 223 (M^+ + H).



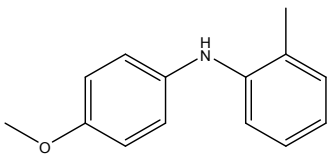
4-methoxy-*N*-p-tolylaniline (3bb). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12 – 6.91 (m, 4H), 6.92–6.69 (m, 4H), 3.78 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.60, 142.3, 136.5, 129.6, 129.2, 121.0, 116.50, 114.6, 55.6, 20.7; MS (ESI): m/z = 212 (M^+ - H).



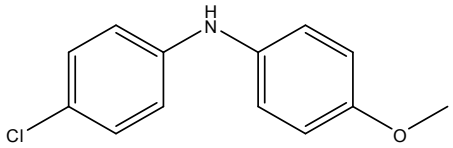
bis(4-methoxyphenyl)amine (3cb)[6]. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.75-7.10 (s, br, 4H), 6.80 (d, *J* = 7.9 Hz, 4H), 3.77 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.0, 119.4, 114.6, 55.6; MS (ESI): m/z = 230 (M^+ + H).



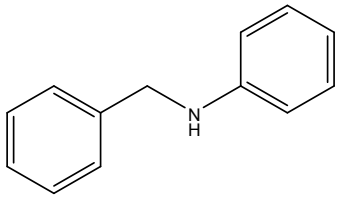
4-methoxy-*N*-(4-(trifluoromethyl)phenyl)aniline (3db)[7]. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 8.6 Hz, 2H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 148.5, 133.6, 126.5, 124.1, 120.2, 114.7, 113.7, 55.6; MS (ESI): m/z = 266 (M^+ - H).



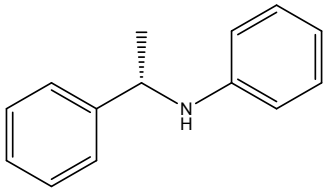
***N*-(4-methoxyphenyl)-2-methylaniline (3eb)**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.13 (d, *J* = 7.3 Hz, 1H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.92-7.02 (m, *J* = 7.1 Hz, 3H), 6.84 (d, *J* = 8.8 Hz, 1H), 6.79 (t, *J* = 7.4 Hz, 1H), 3.79 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 155.2, 143.4, 136.4, 130.9, 127.0, 125.5, 122.3, 120.2, 115.4, 114.9, 55.9, 18.2; MS (ESI): m/z = 214 (M^+ + H).



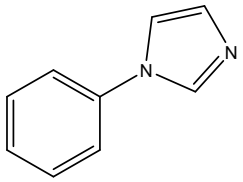
4-chloro-*N*-(4-methoxyphenyl)aniline (3fb)[8]. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.8 Hz, 1H), 7.02 (d, *J* = 8.7 Hz, 1H), 6.84 (d, *J* = 8.9 Hz, 1H), 6.79 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5, 143.8, 135.1, 129.0, 123.9, 122.5, 116.6, 114.7, 55.6.



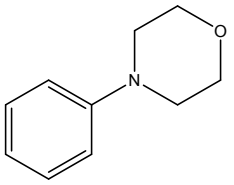
***N*-benzylaniline (3aq)**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 – 7.29 (m, 4H), 7.26 (dt, *J* = 5.3, 2.2 Hz, 1H), 7.16 (tt, *J* =7.9 Hz, *J* = 1.6 Hz, 2H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.63 (dd, *J* = 8.5, 0.9 Hz, 2H), 4.32 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 147.9, 139.2, 129.1, 128.5, 127.3, 127.1, 117.4, 112.7, 48.4.



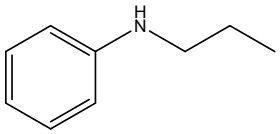
(S)-*N*-(1-phenylethyl)aniline (3ar)[9]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.2 Hz, 2H), 7.29 (dd, *J* = 8.2, 6.9 Hz, 2H), 7.20 (t, *J* = 7.1 Hz, 1H), 7.07 (dd, *J* = 8.5, 7.4 Hz, 2H), 6.62 (t, *J* = 7.3 Hz, 1H), 6.49 (d, *J* = 7.6 Hz, 2H), 4.47 (q, *J* = 6.7 Hz, 1H), 4.03 (br s, 1H, NH), 1.51 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 144.9, 128.9, 128.4, 126.6, 125.6, 117.0, 113.1, 53.4, 25.1.



1-phenyl-1*H*-imidazole (3as)[10]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.51 – 7.43 (m, 2H), 7.42 – 7.31 (m, 3H), 7.27 (s, 1H), 7.20 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.0, 135.3, 130.1, 129.6, 127.2, 121.2, 118.1.



4-phenylmorpholine (3at)[2]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.05-6.76 (t, 3H), 3.86 (m, *J* = 4.5, 4H), 3.16 (t, *J* = 4.8, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 129.0, 120.0, 115.6, 67.0, 49.5.



***N*-propylaniline (3au)**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (dd, *J* = 8.4, 7.4 Hz, 2H), 6.67 (t, *J* = 7.3 Hz, 1H), 6.59 (d, *J* = 7.6 Hz, 2H), 3.89 (br s, NH), 3.07 (t, *J* = 7.1 Hz, 2H), 1.70 – 1.57 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 129.0, 116.9, 112.5, 45.8, 22.8, 11.8.

4. References

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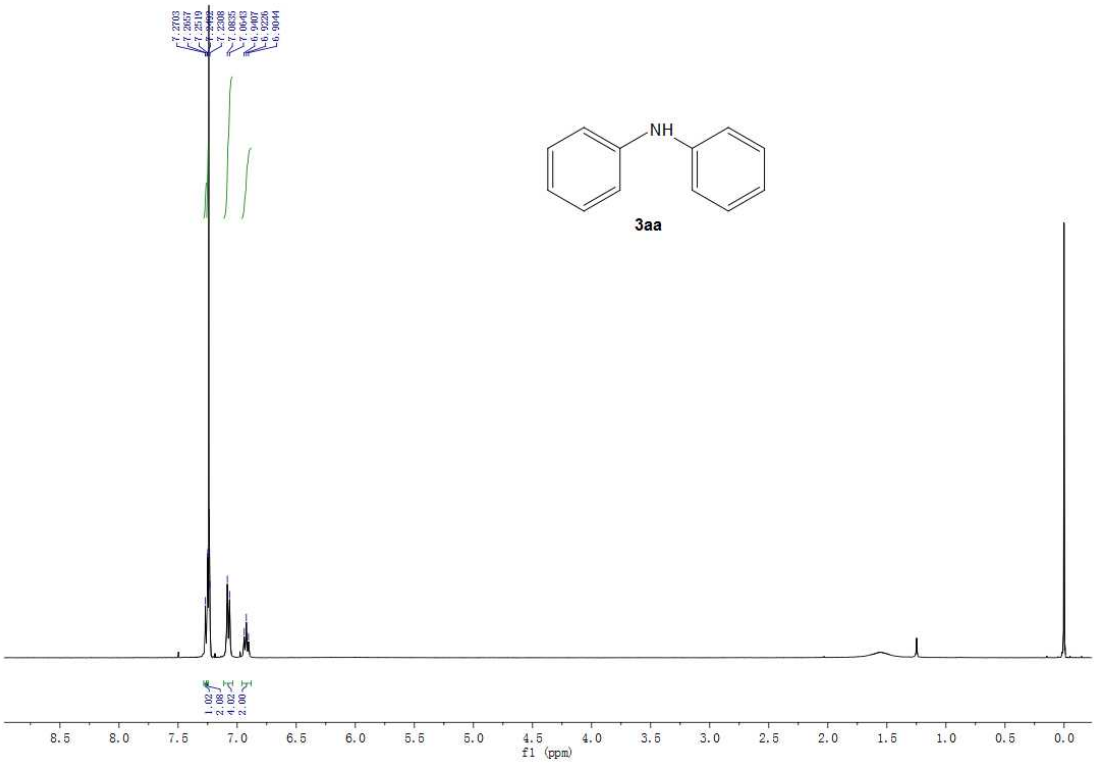
5. Copies of ^{1}H and ^{13}C NMR Spectra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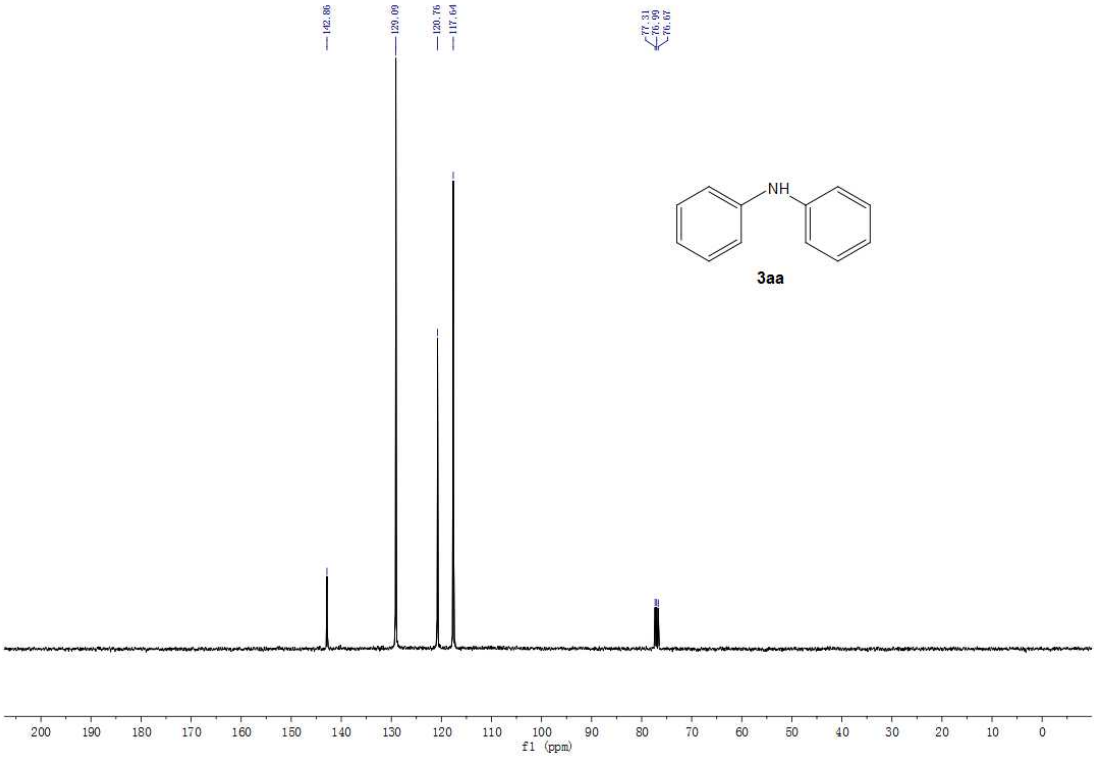
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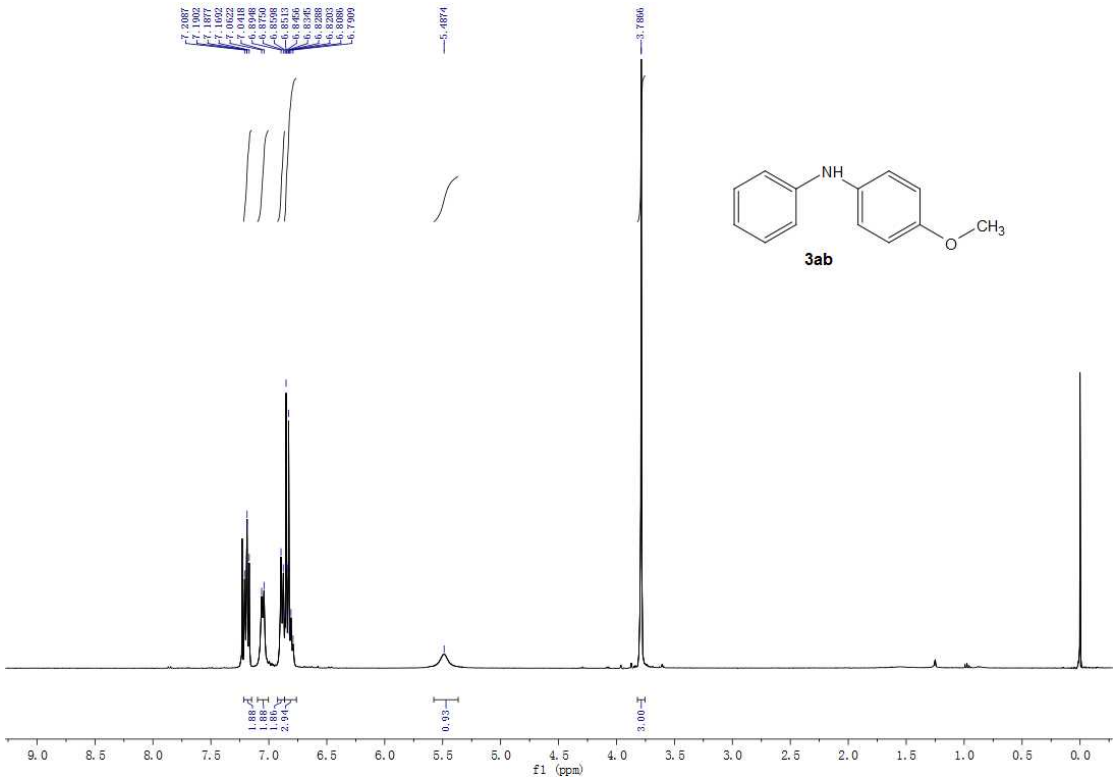
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6.8513
6.8456
6.8295
6.8288
6.8203
6.8086
6.7909
5.4874
3.7866
NH
O
CH3
3ab
1.88
1.88
1.86
2.94
0.93
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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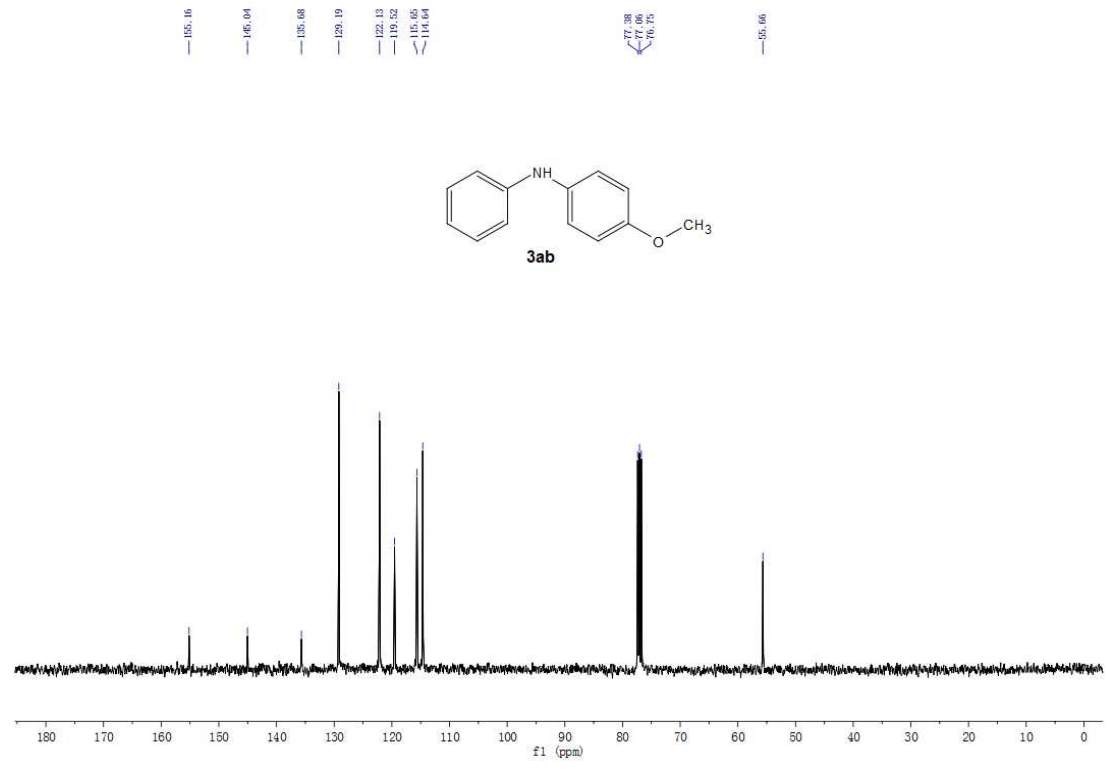
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O
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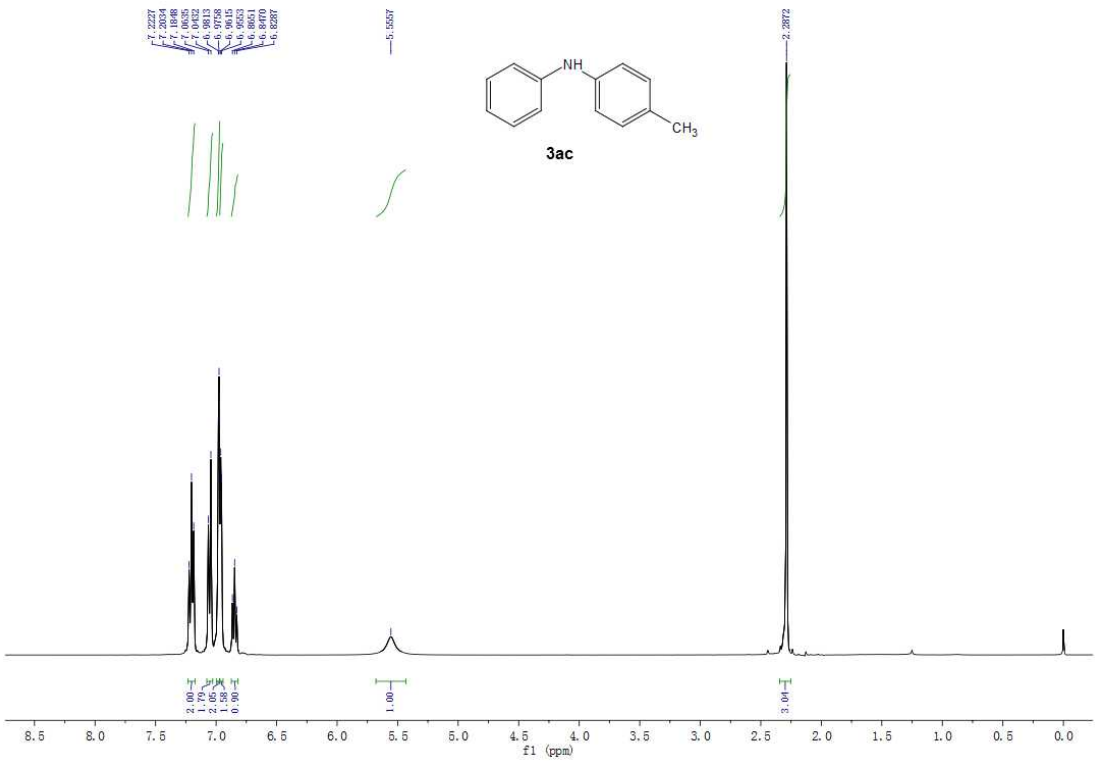
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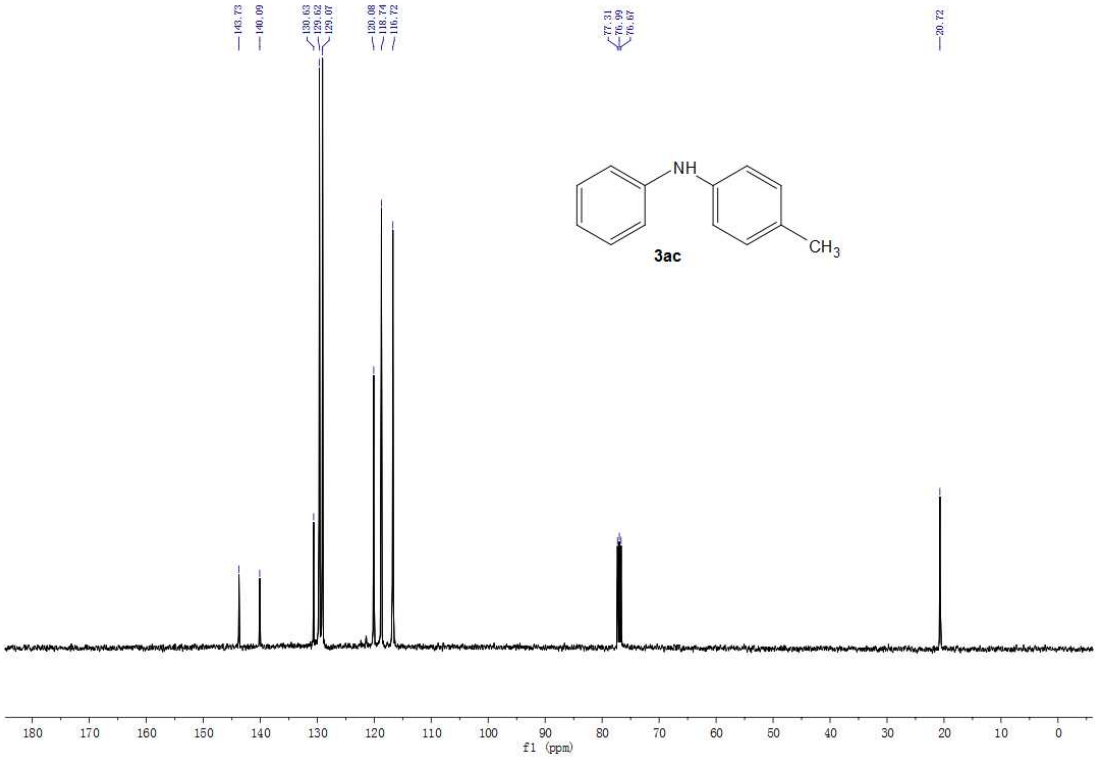
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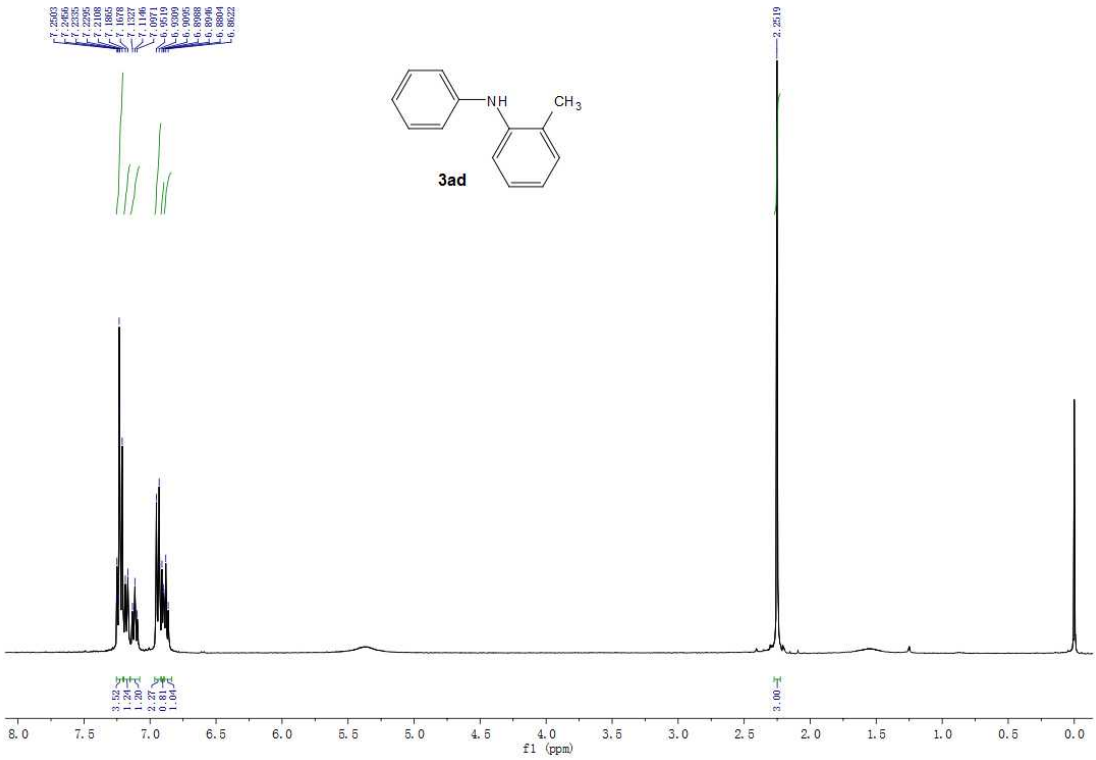
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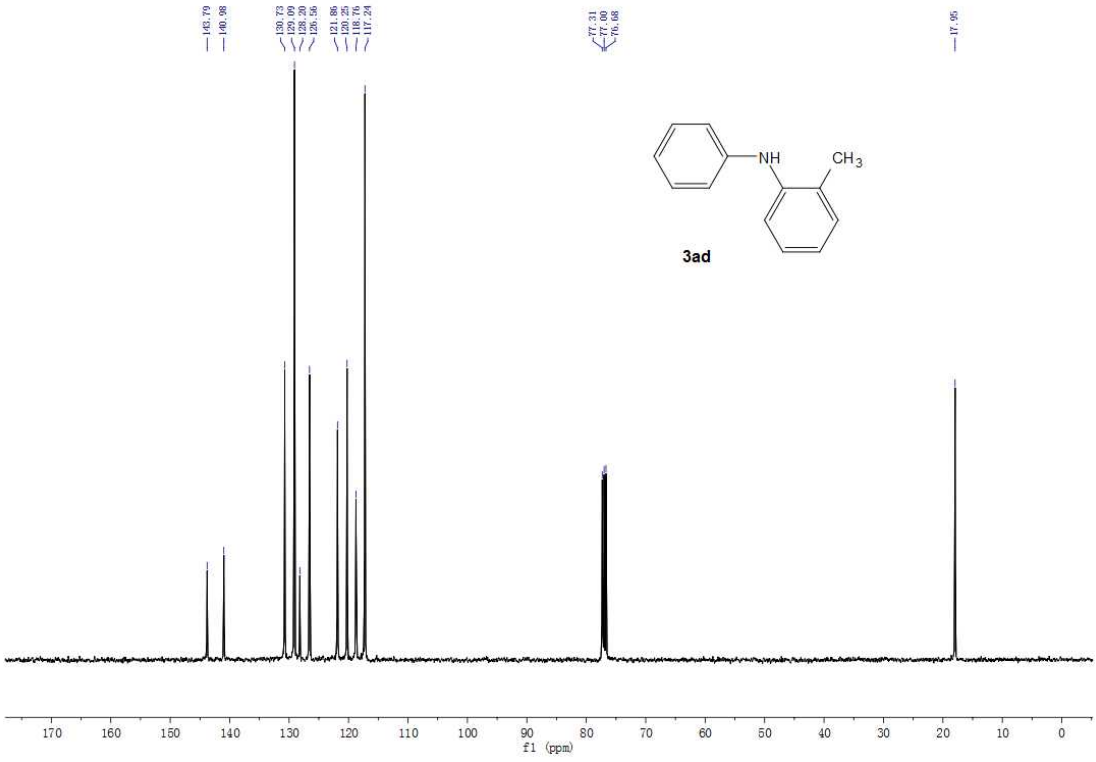
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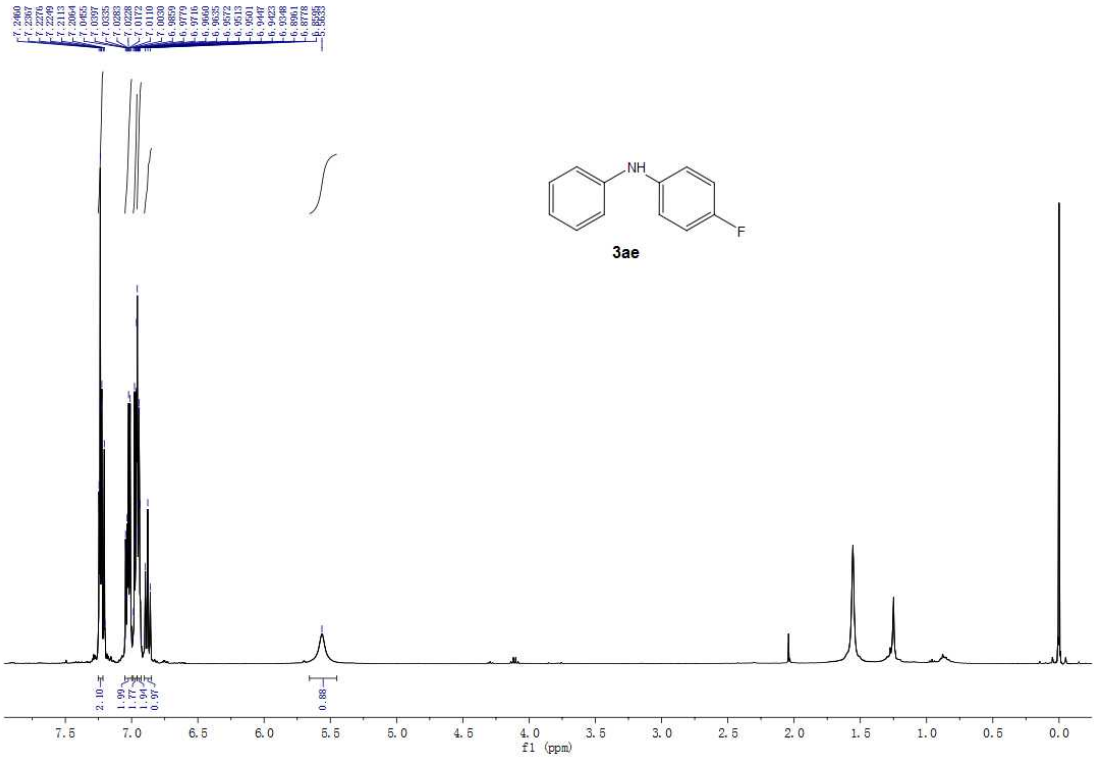
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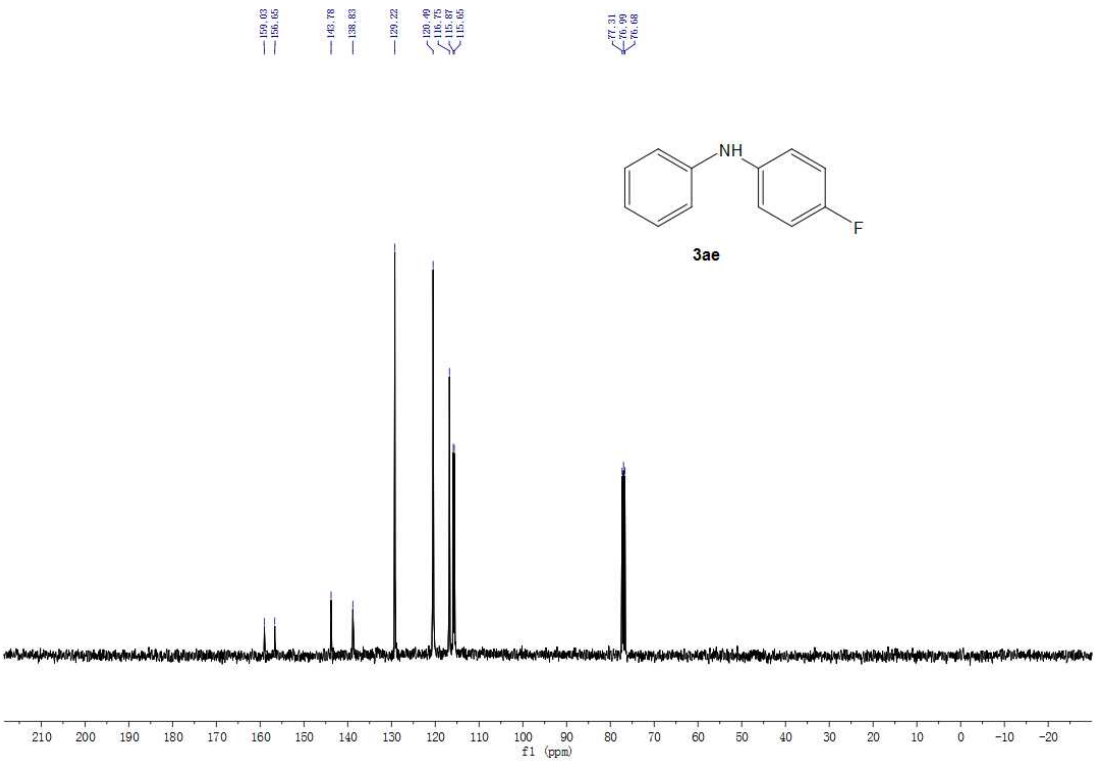
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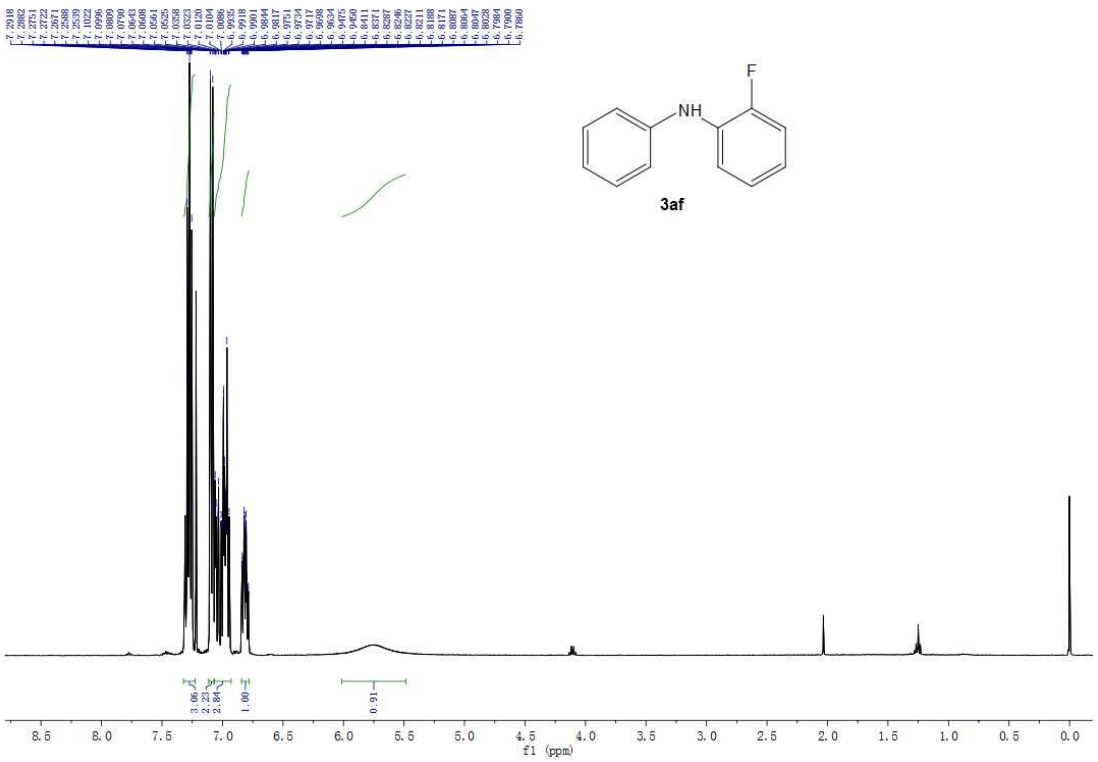
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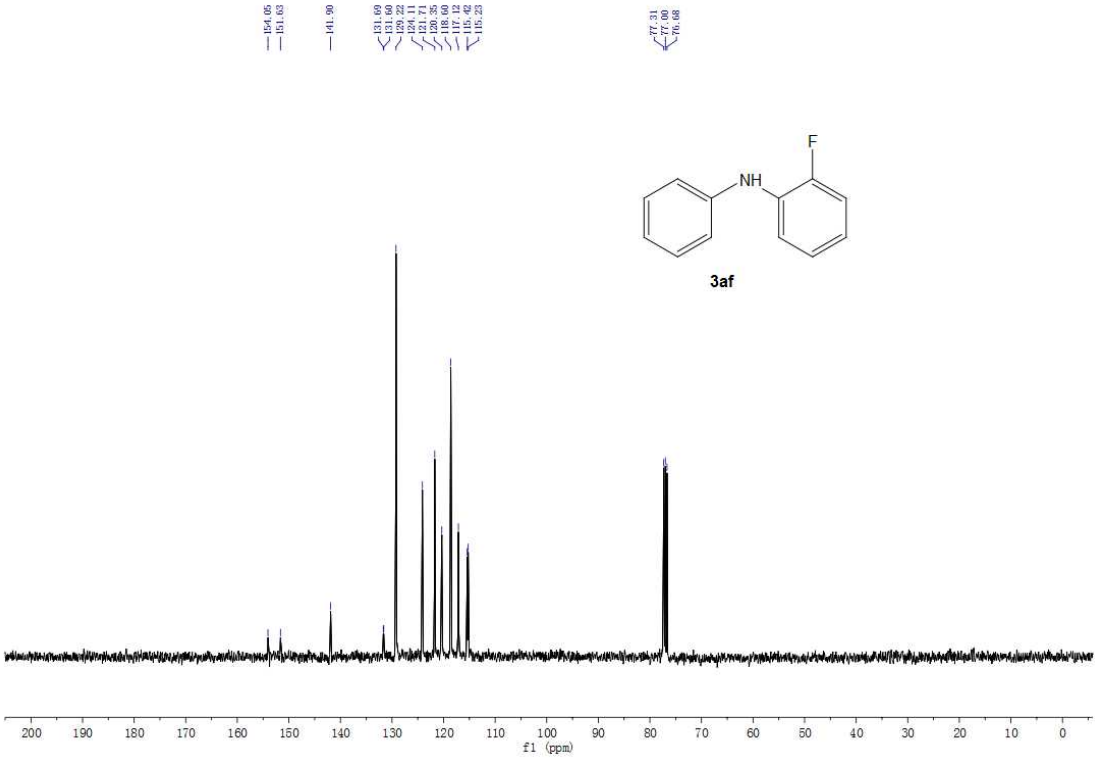
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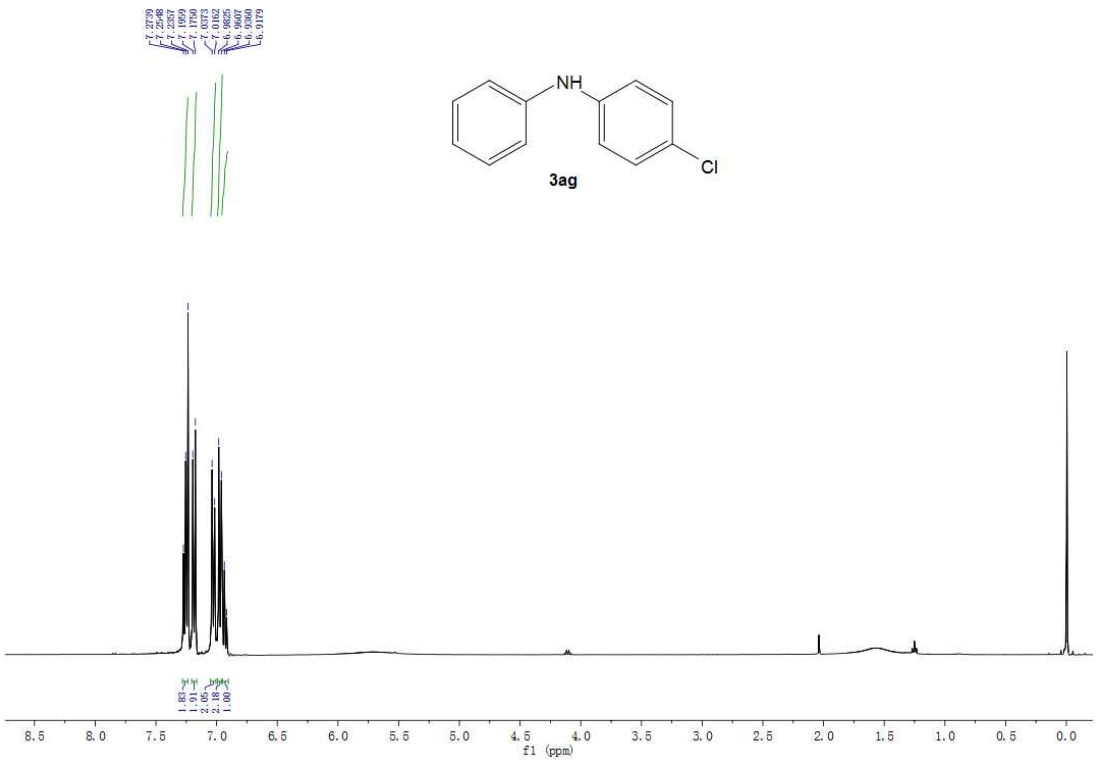
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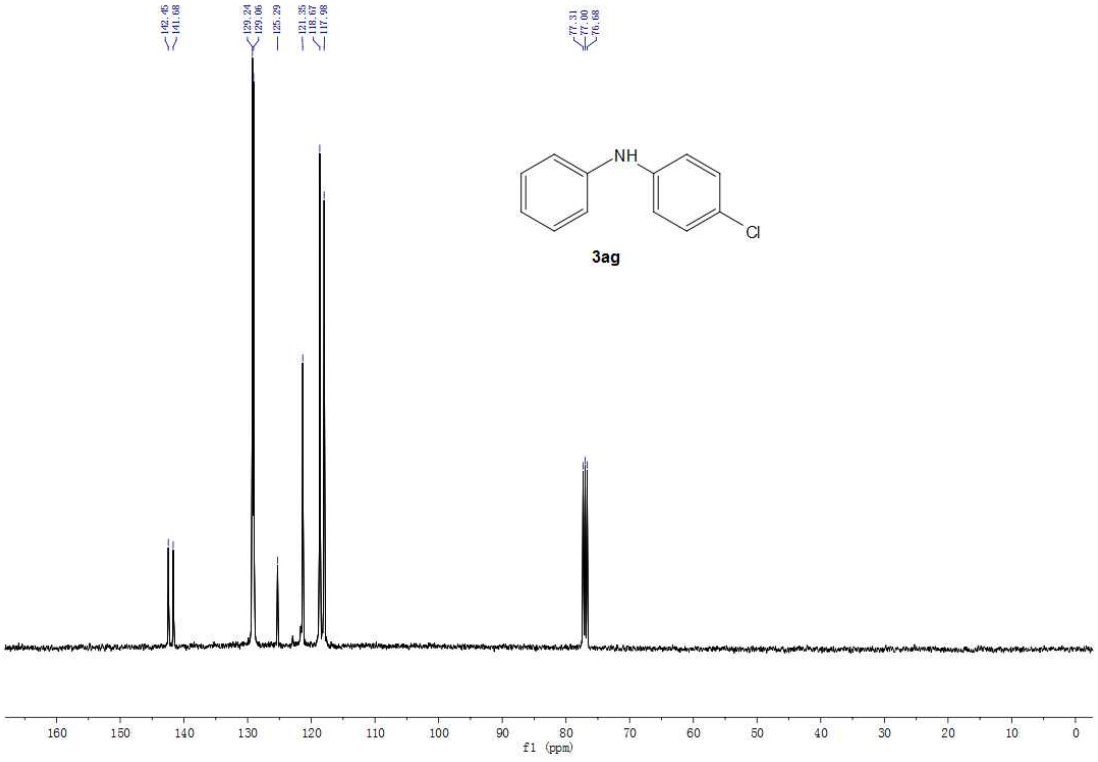
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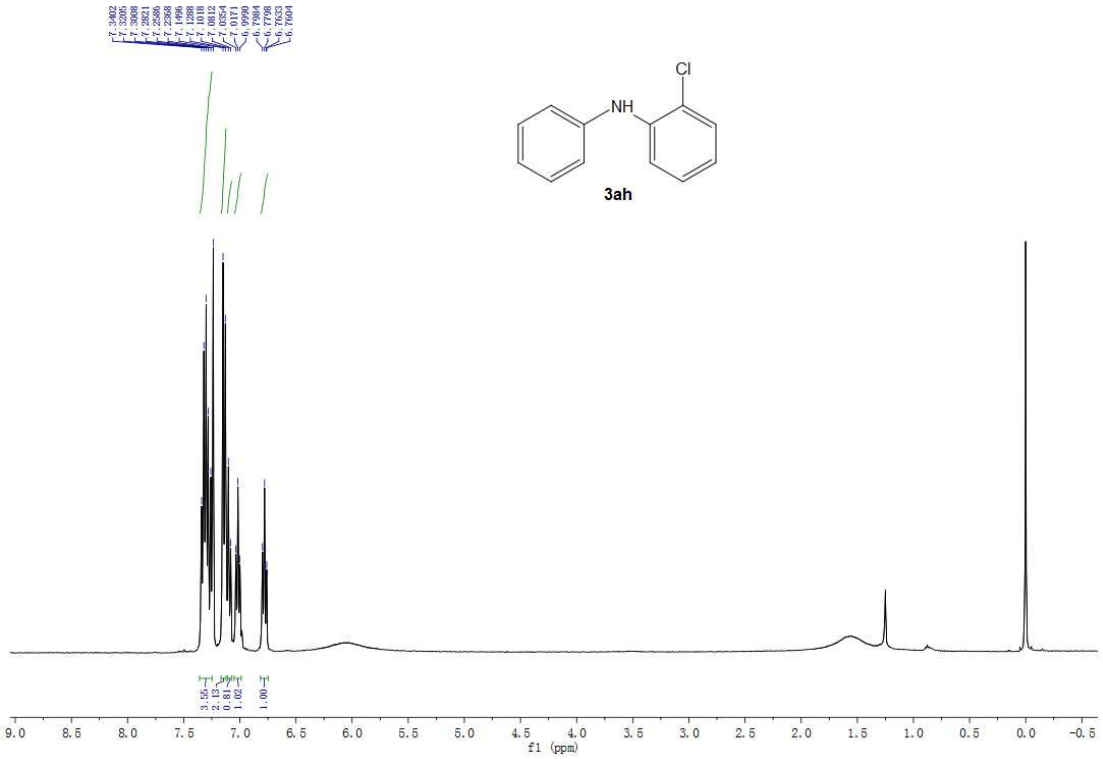
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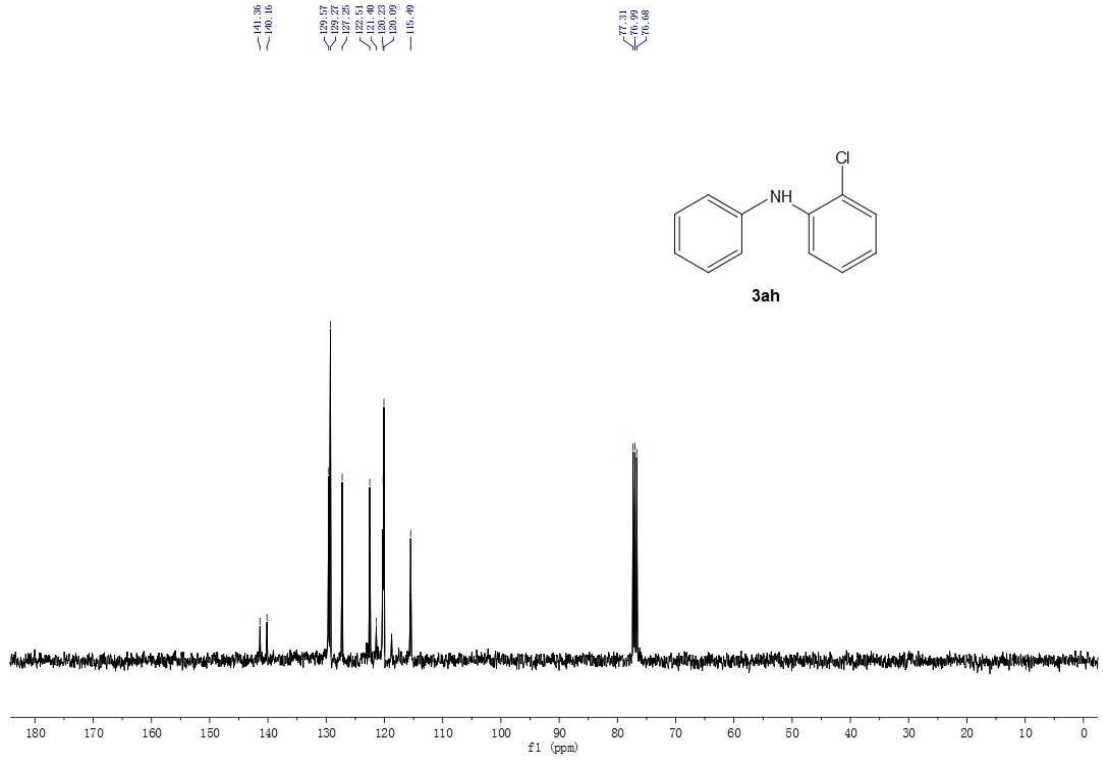
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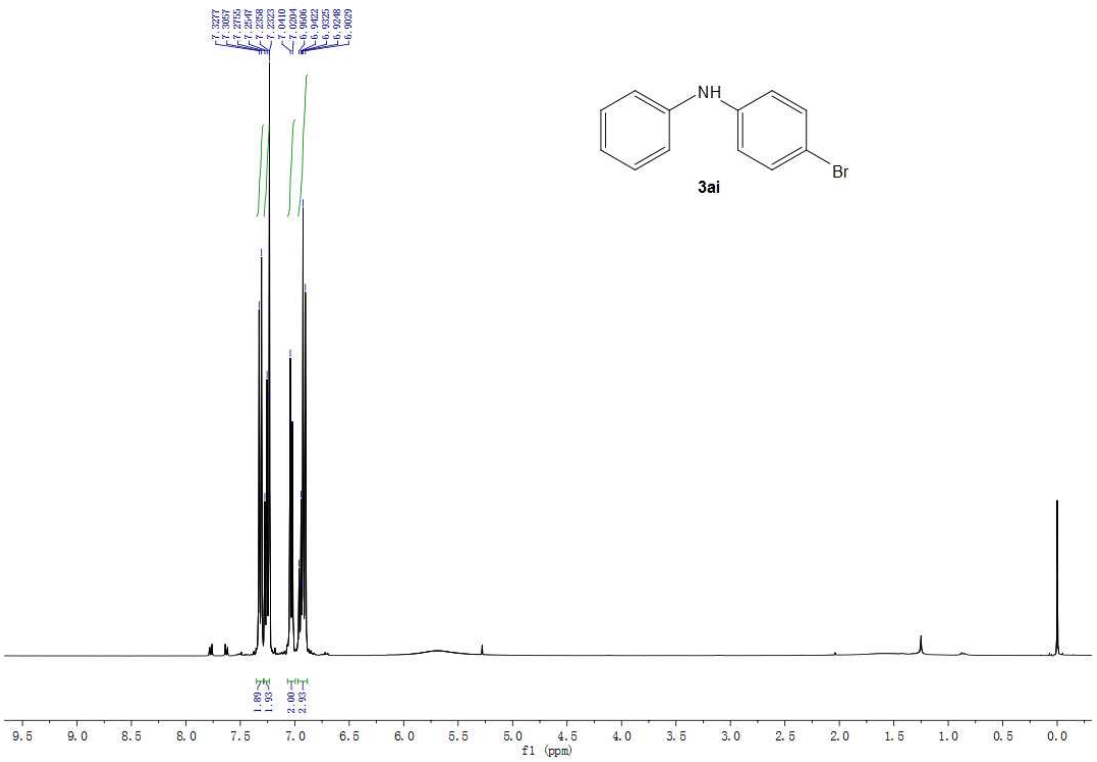
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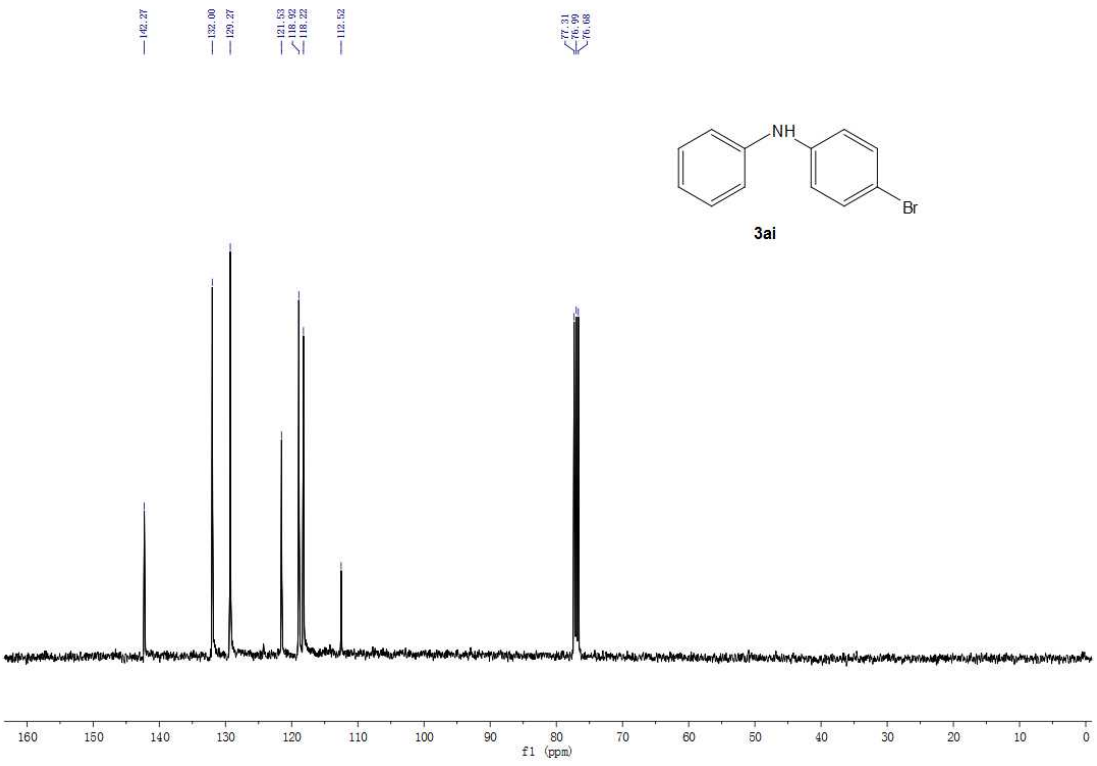
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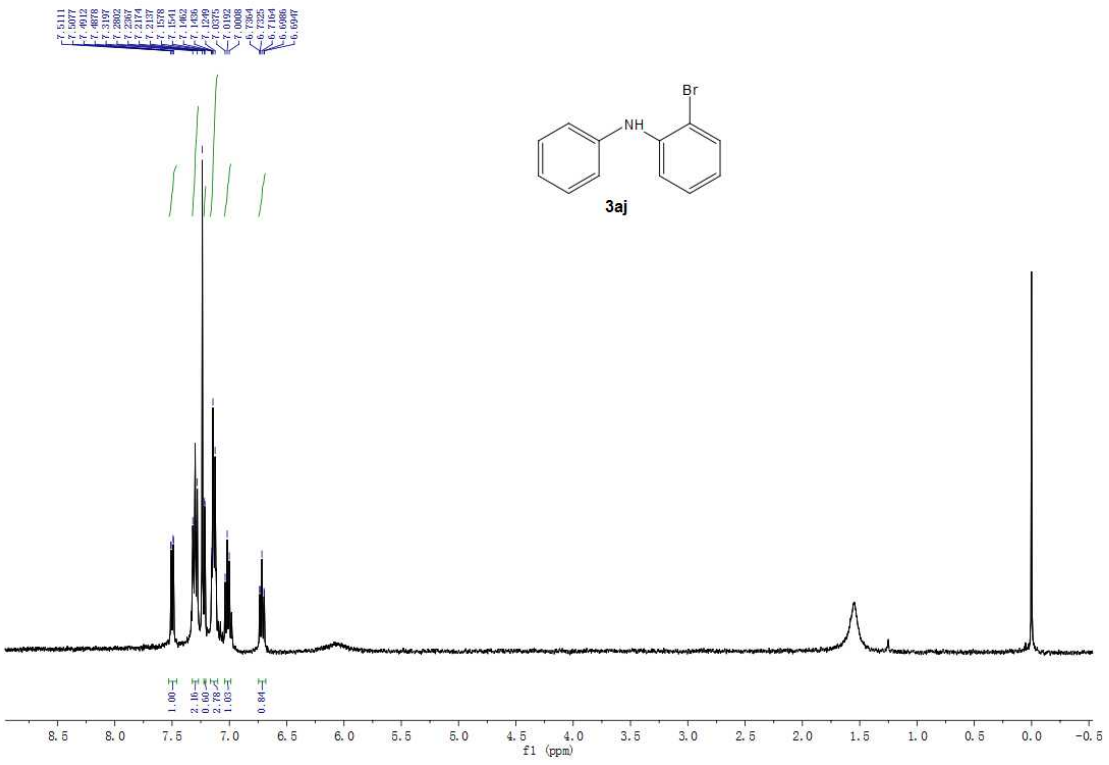
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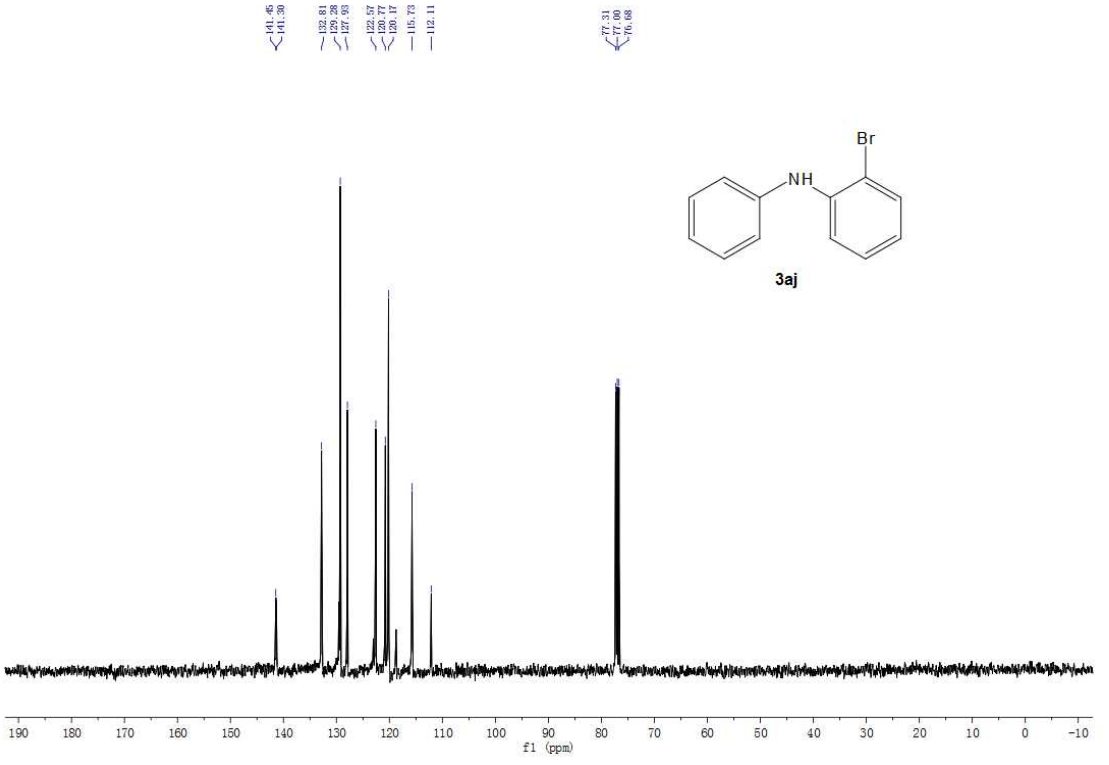
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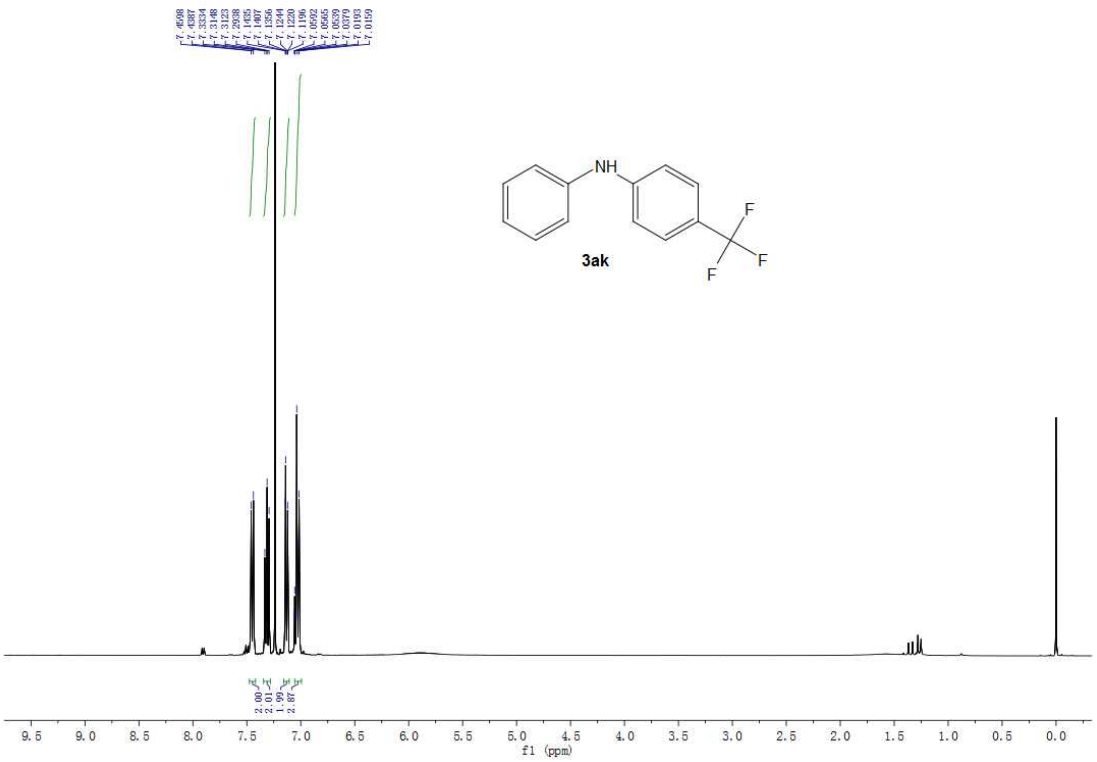
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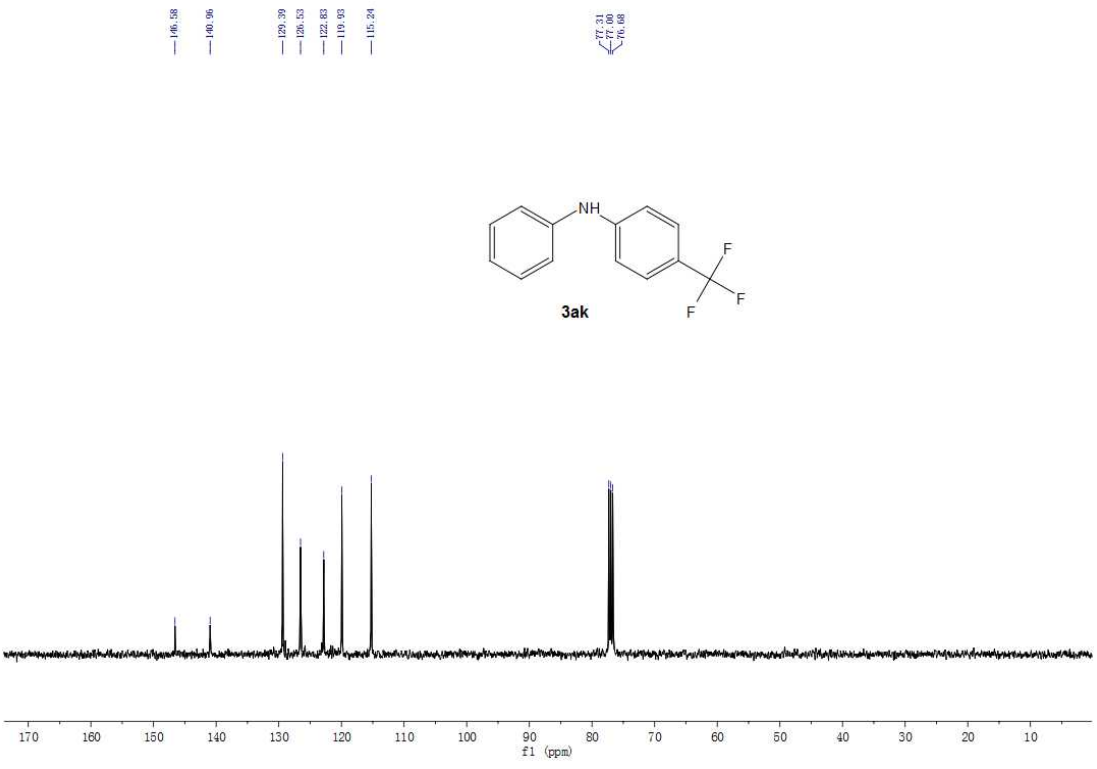
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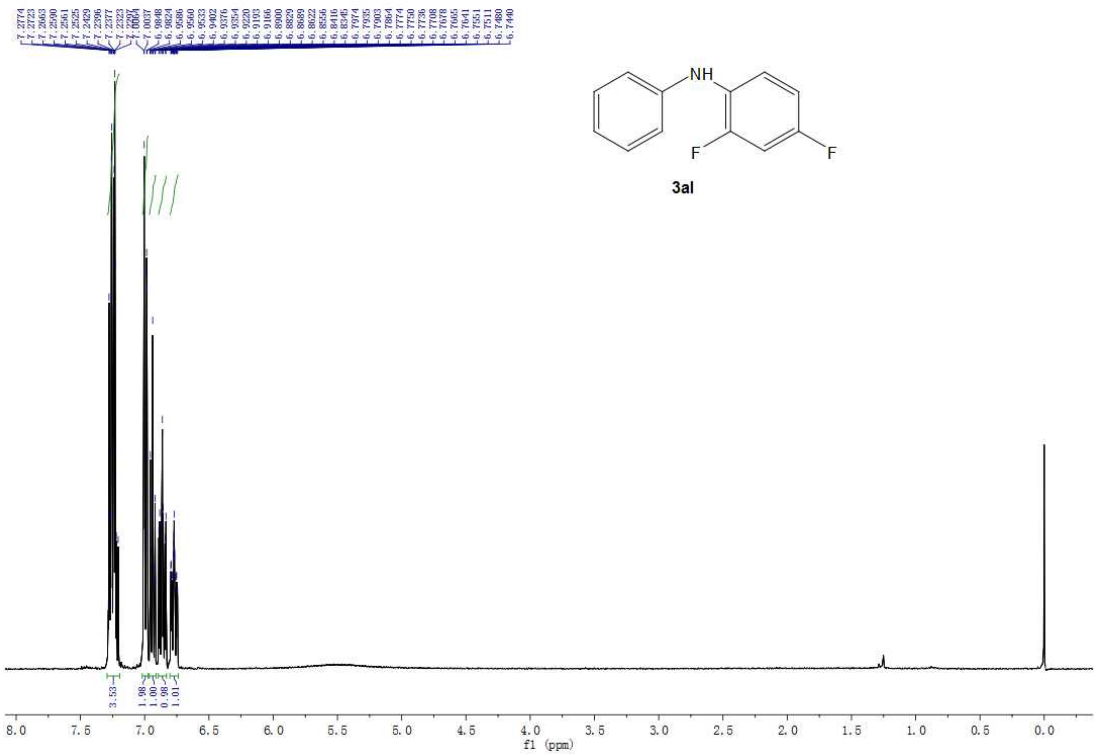
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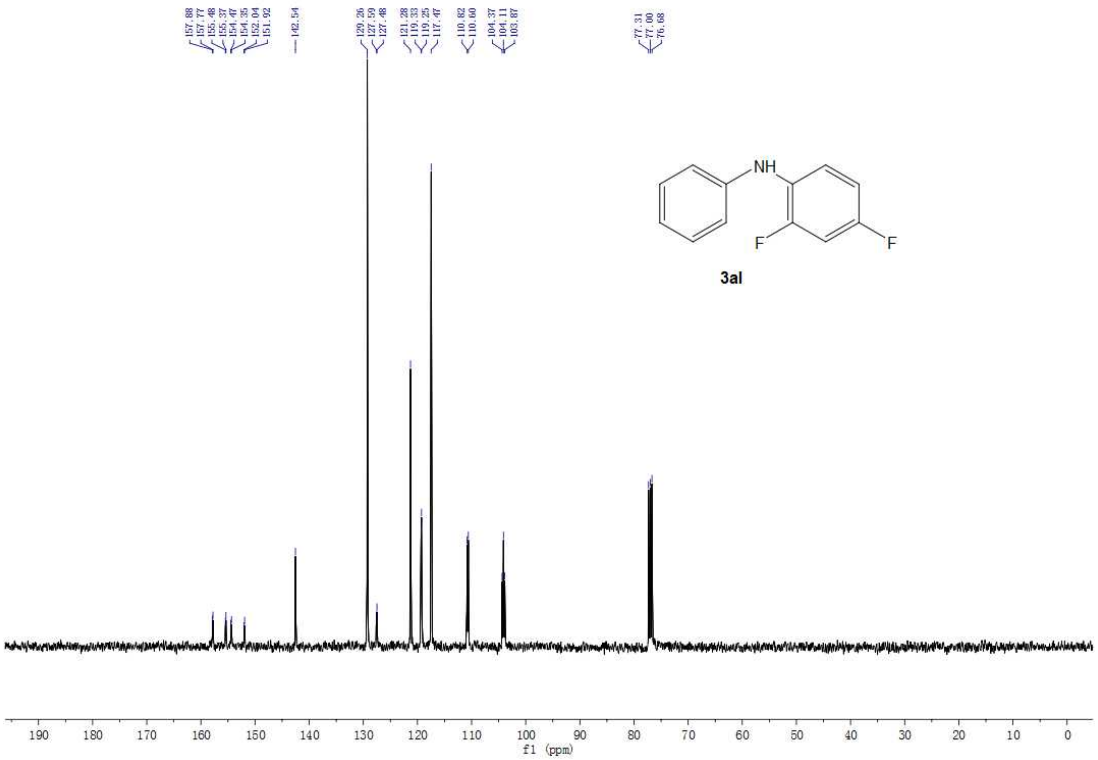
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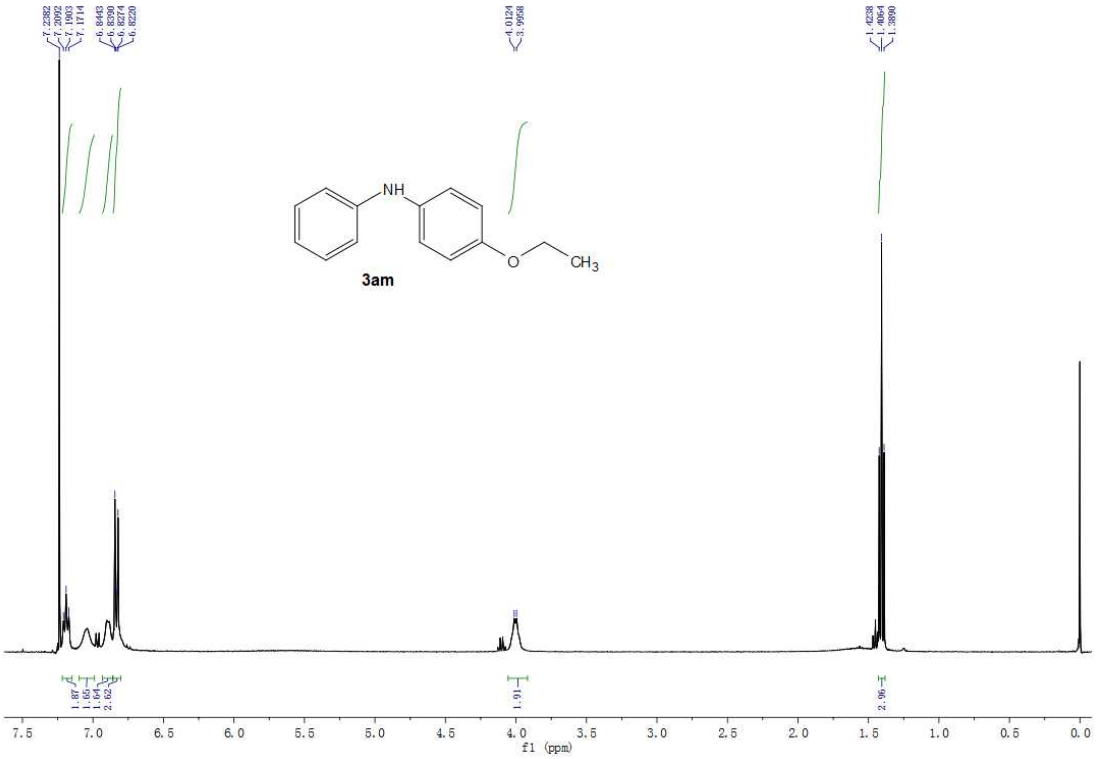
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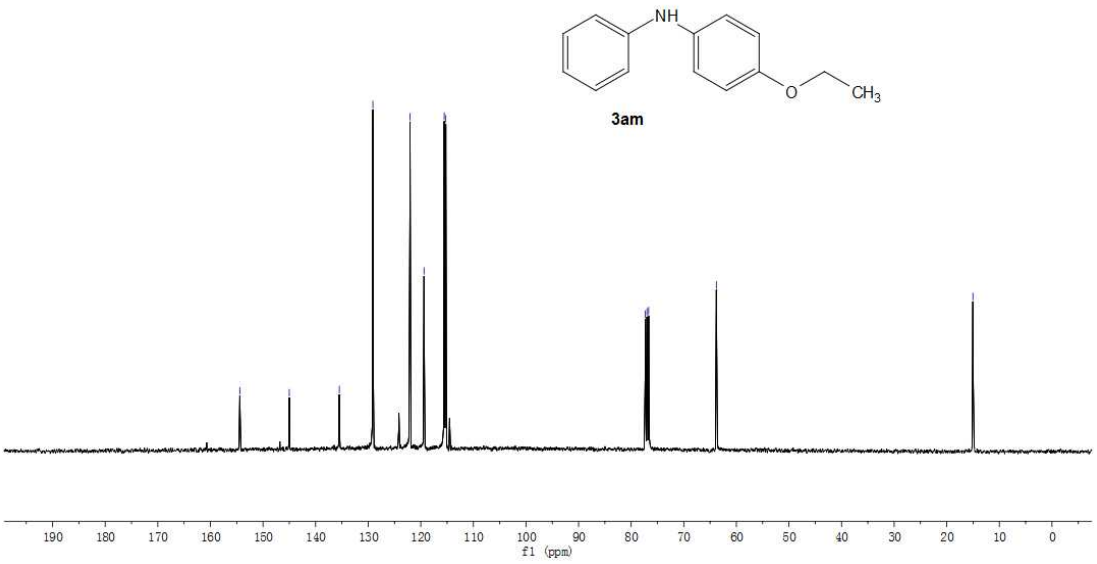
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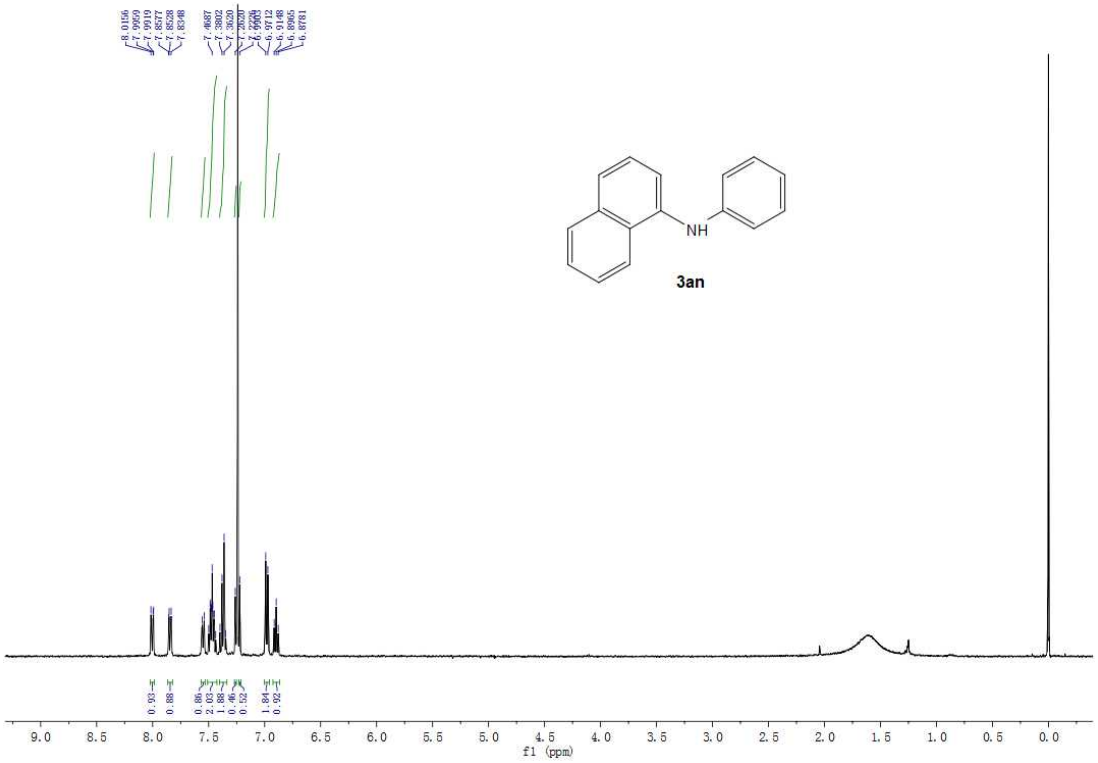
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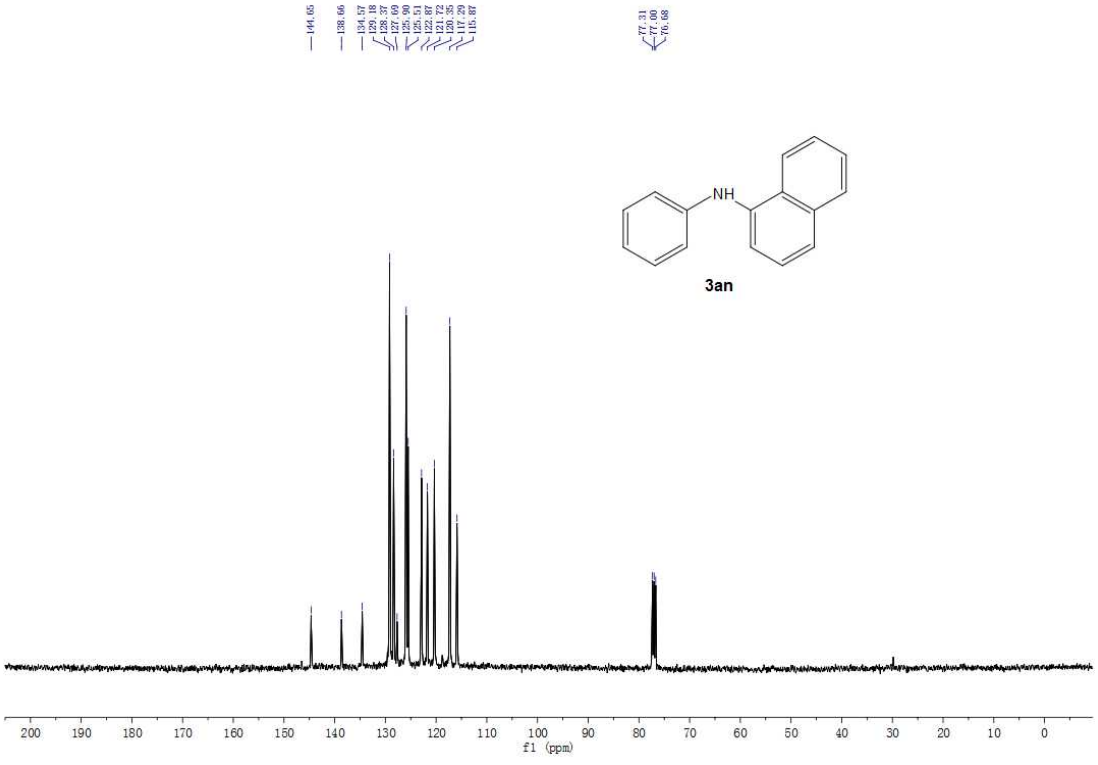
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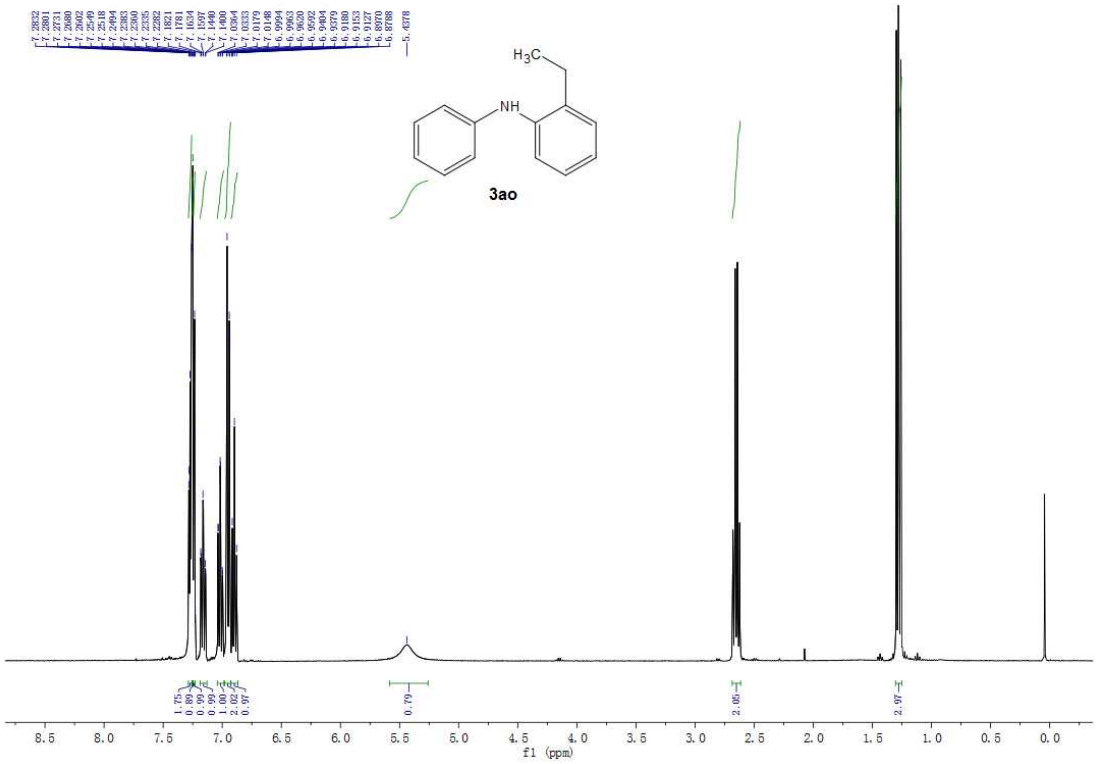
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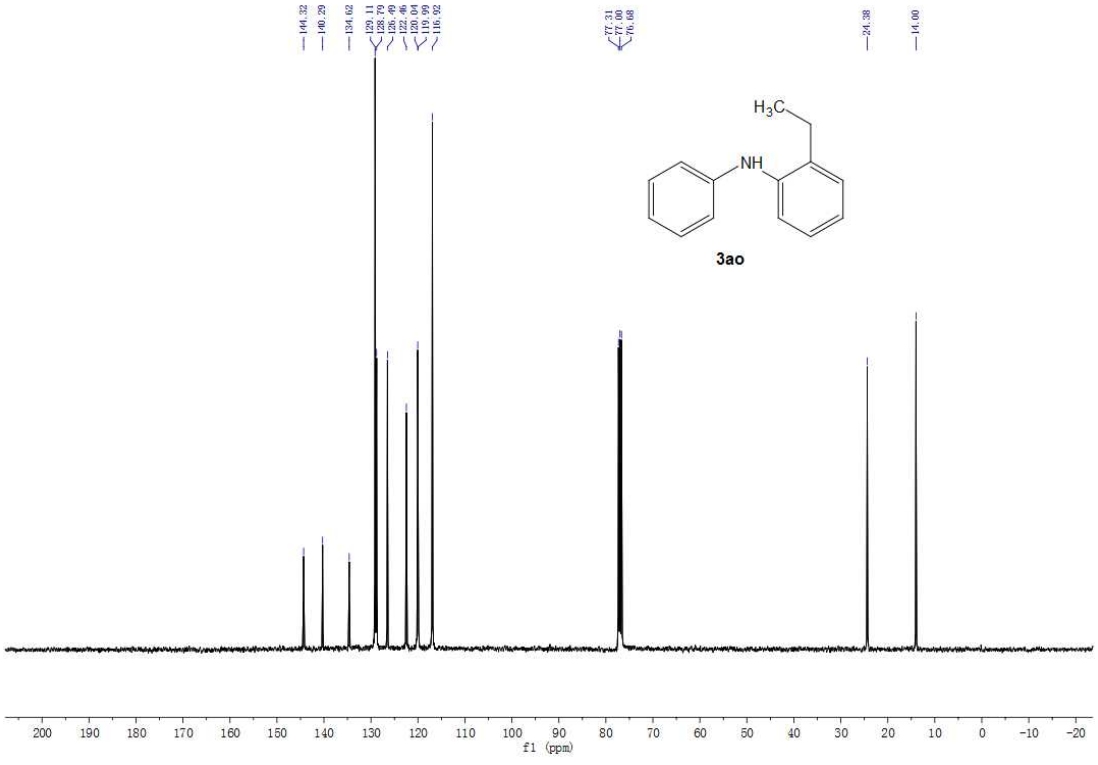
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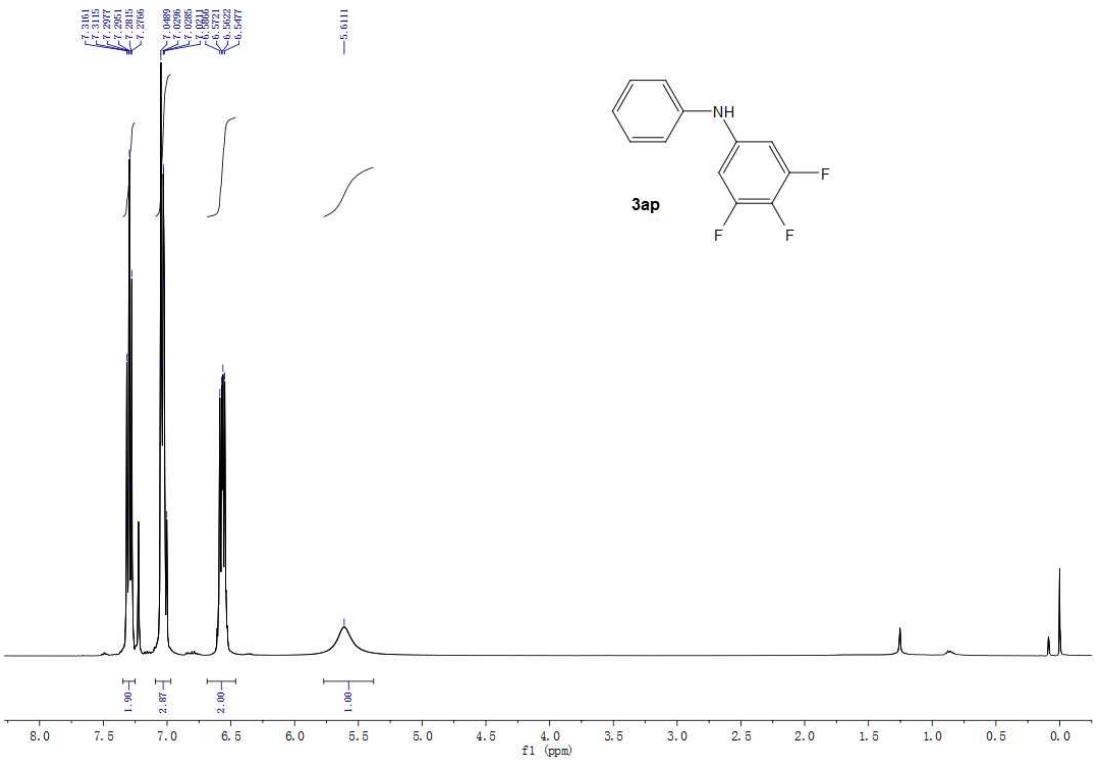
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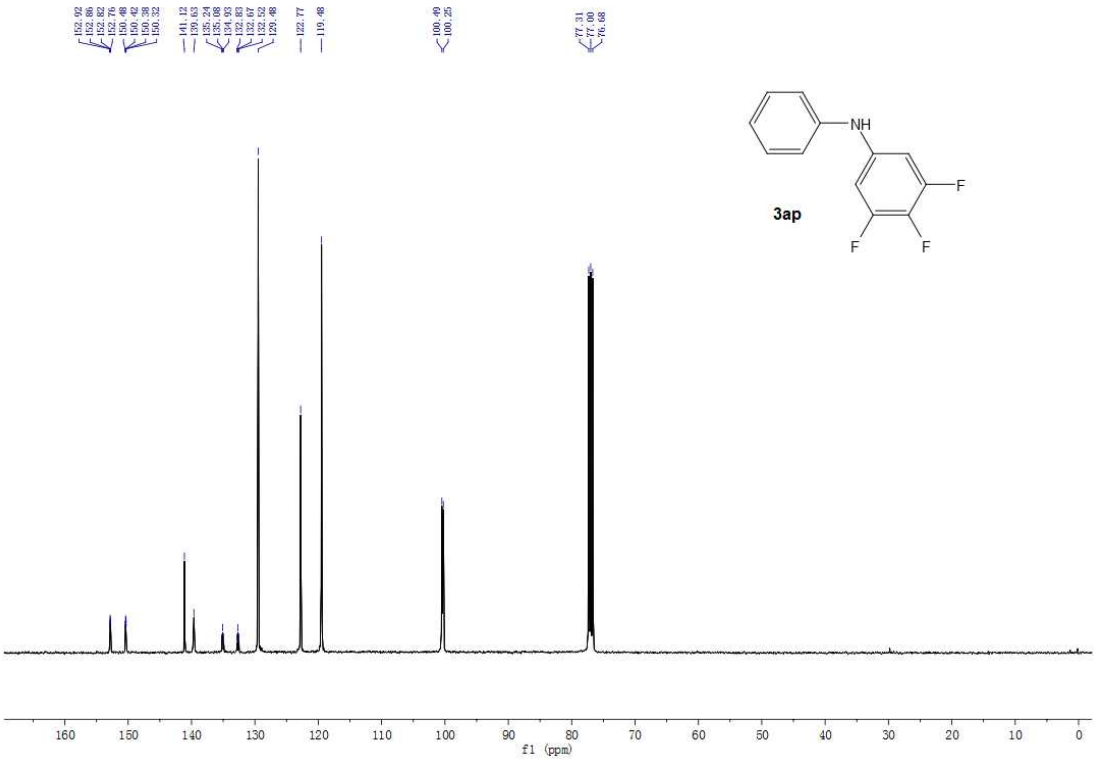
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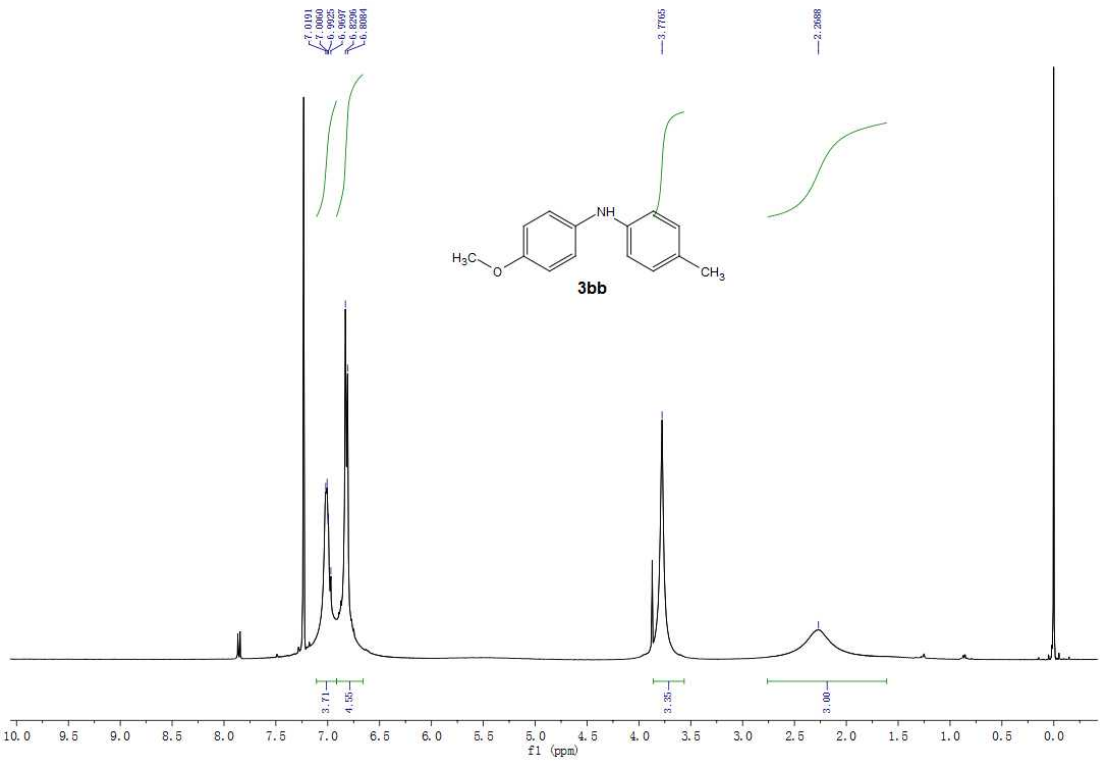
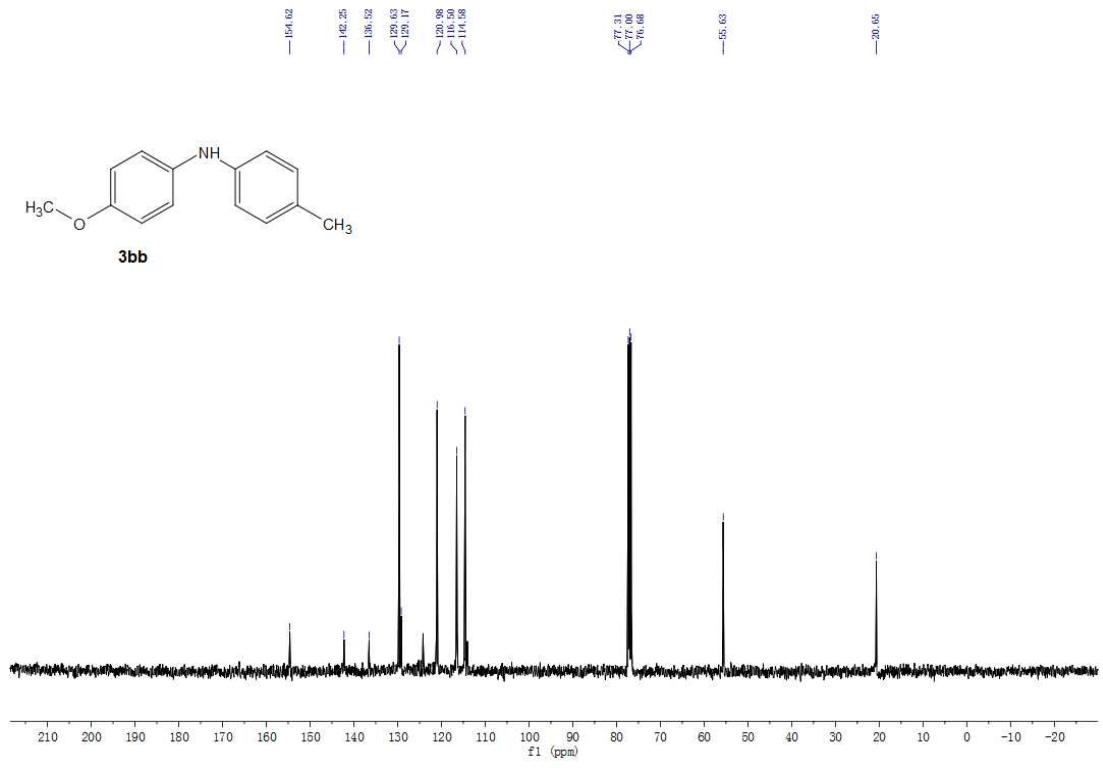
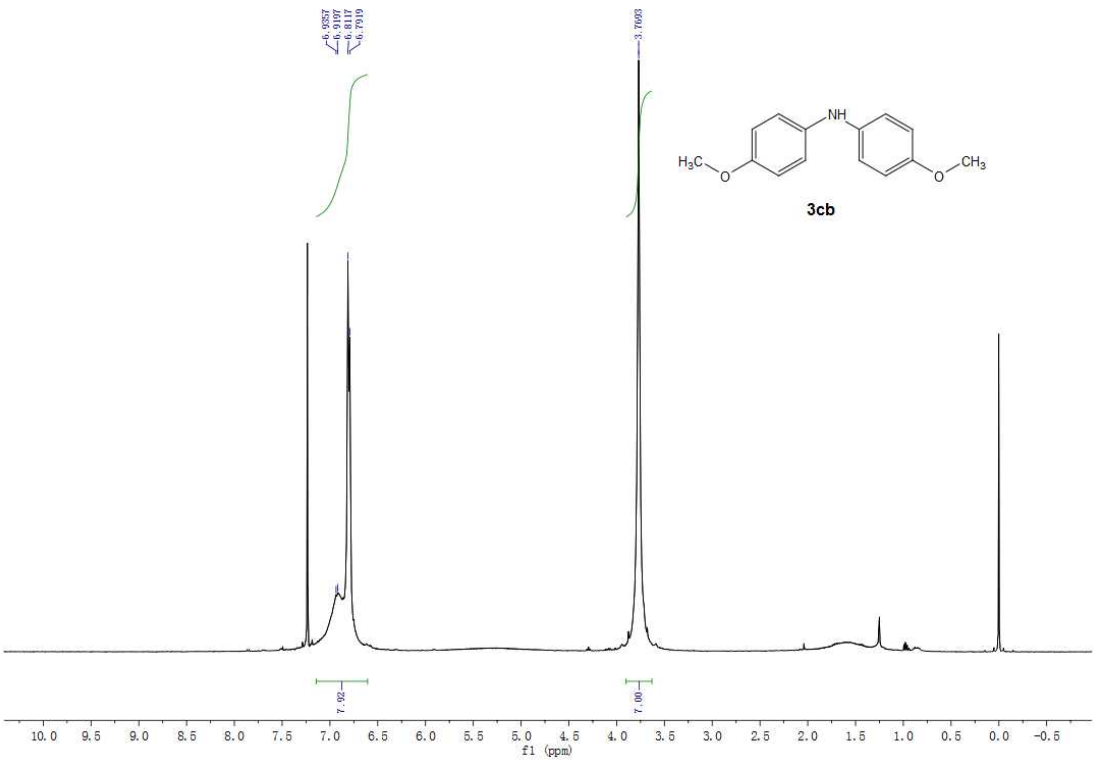
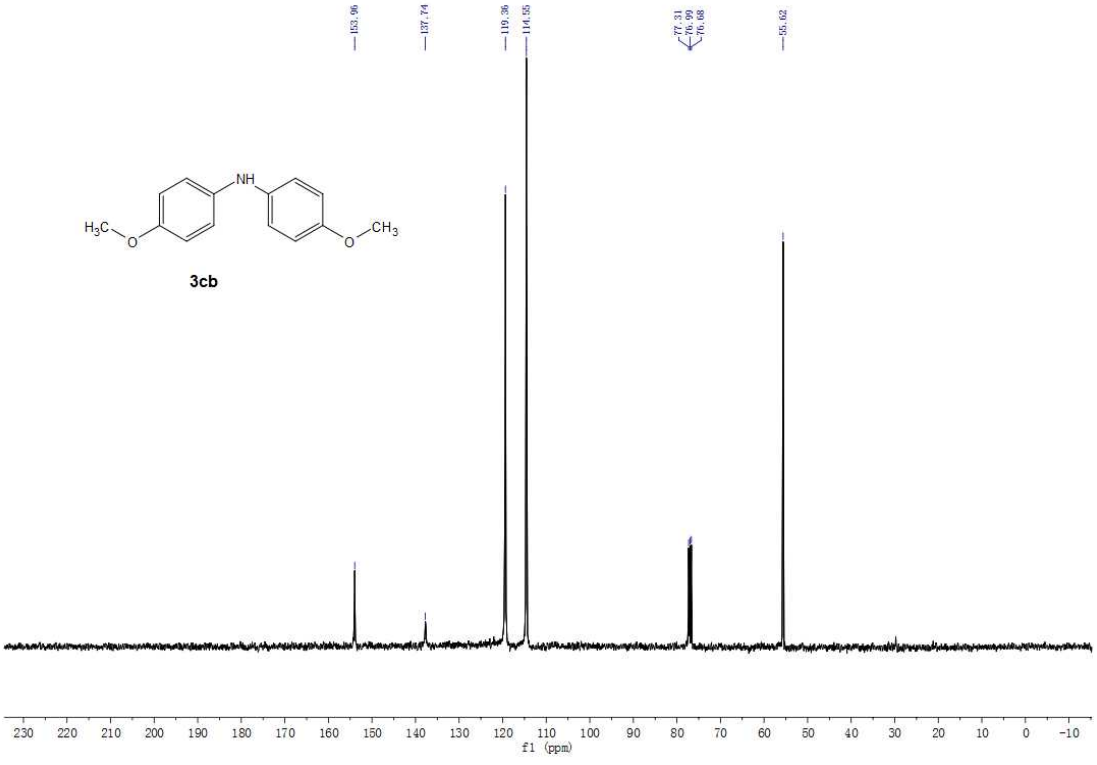
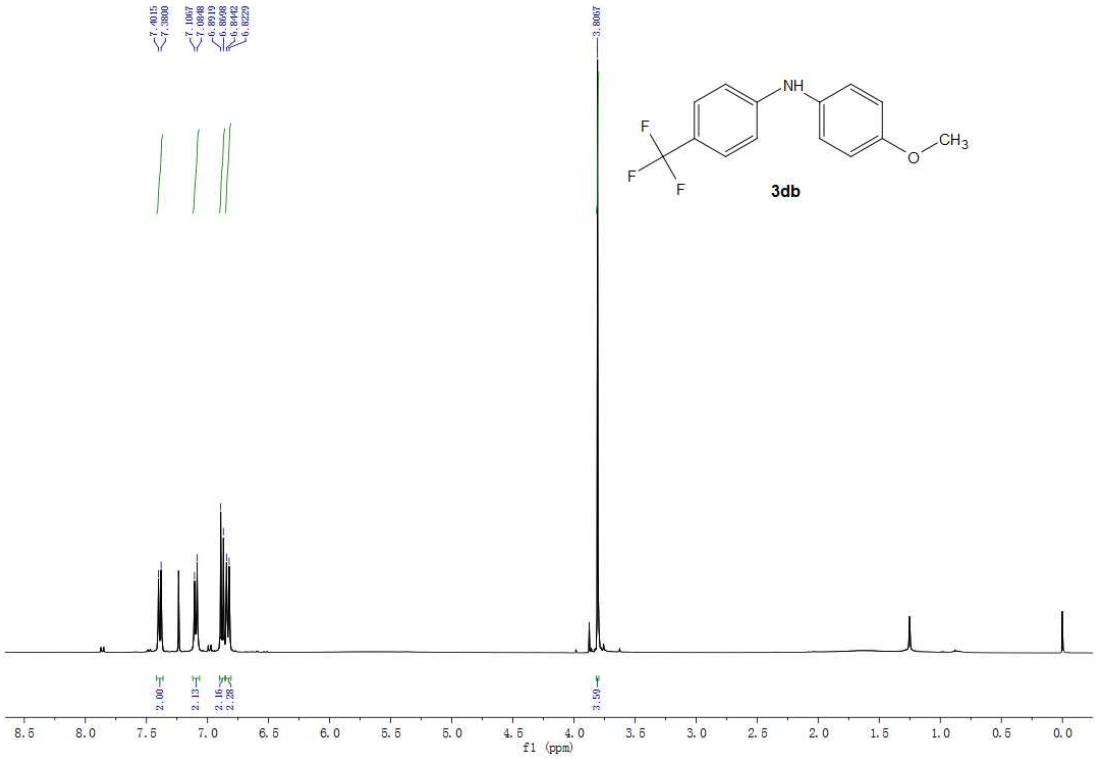
0
f1 (ppm)


7.0191
7.0060
6.9925
6.9801
6.8296
6.8084
3.7765
2.2688
NH
H3C
O
CH3
3bb
3.71
4.55
3.35
3.00
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)


154.62
142.25
136.52
129.63
129.17
120.98
116.50
114.58
77.31
77.00
76.68
55.63
20.65
NH
H3C
O
CH3
3bb
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


6.9957
6.9797
6.8117
6.7919
3.7693
NH
H3C
O
O
CH3
3cb
7.92
7.00
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
f1 (ppm)


153.96
137.74
119.36
114.55
77.31
76.99
76.68
55.62
NH
H3C
O
O
CH3
3cb
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)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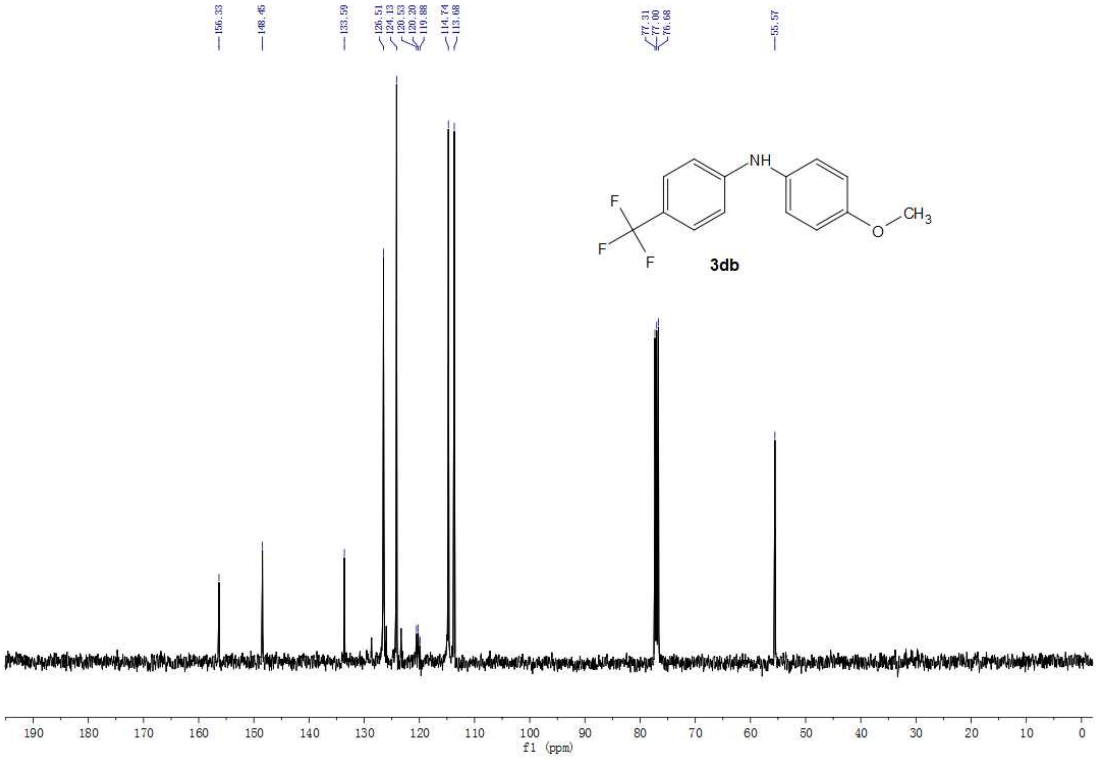
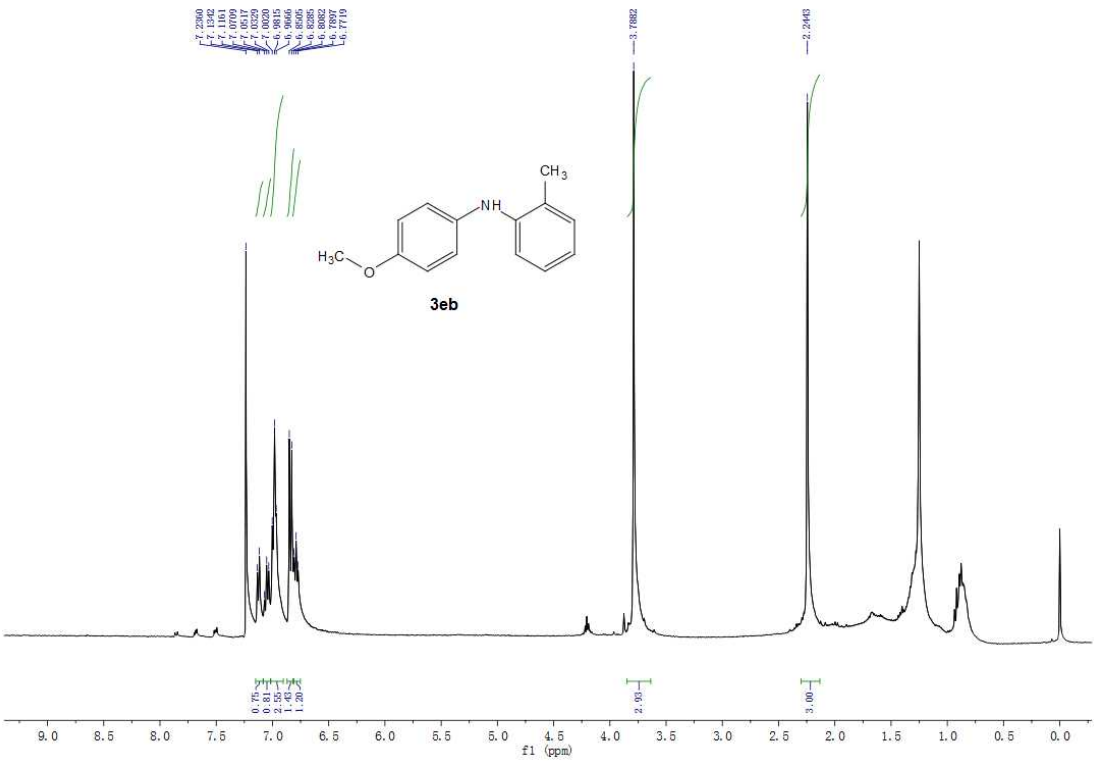
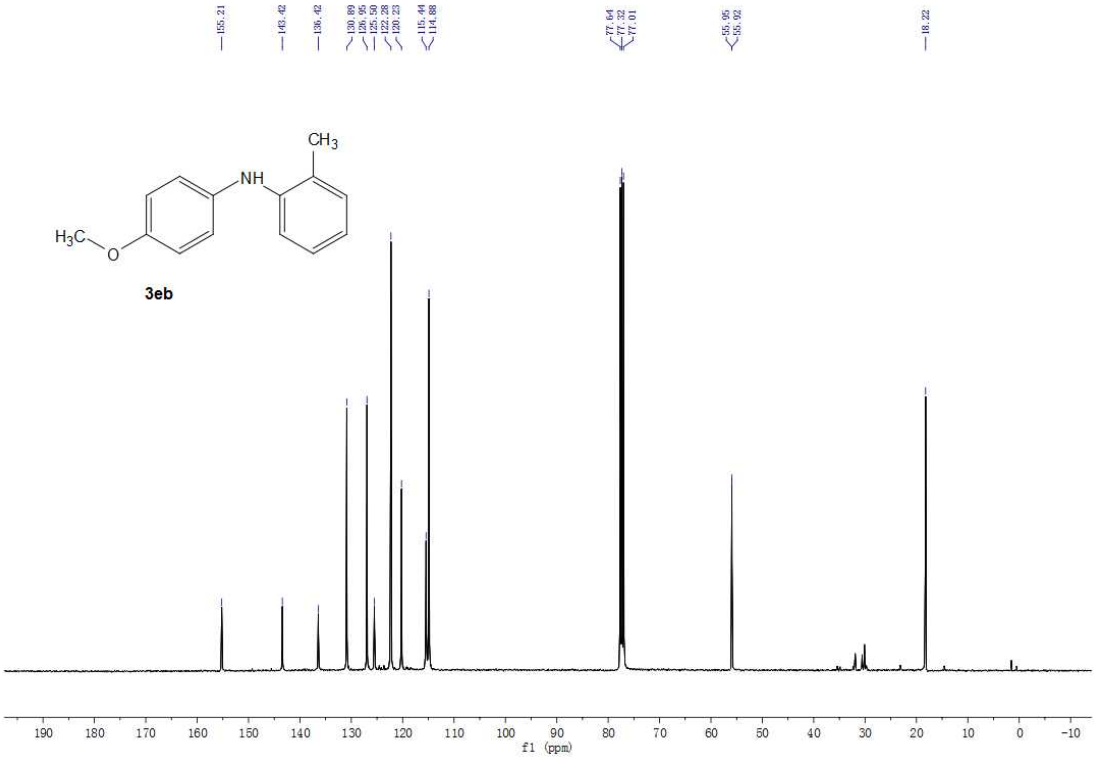
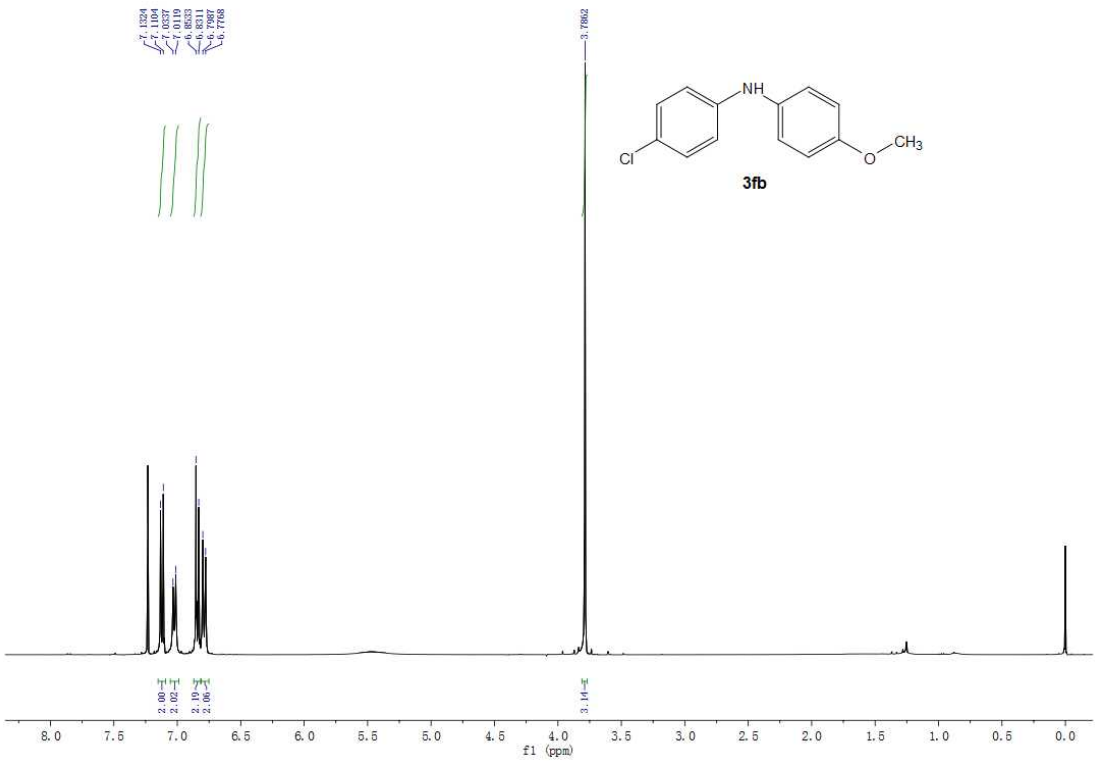
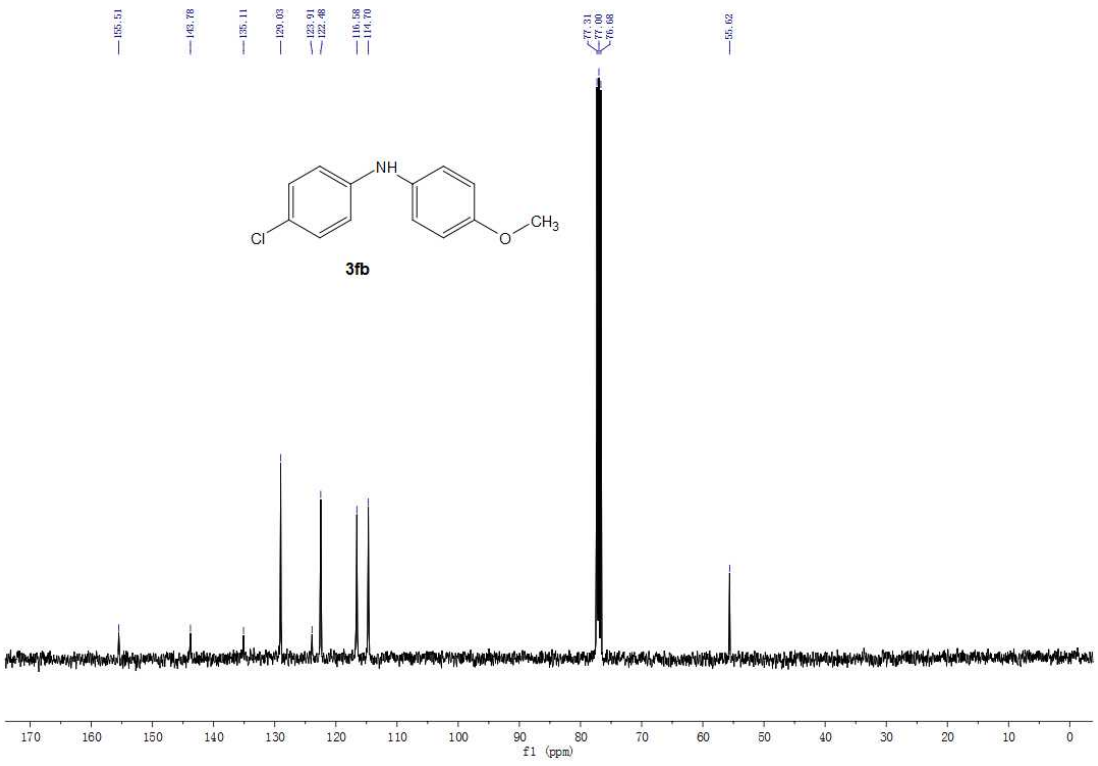
7.3899
7.1067
7.0946
7.0713
6.8698
6.8442
6.6229
3.8067
NH
F
F
F
O
CH3
3db
2.00
2.13
2.16
2.28
3.59
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

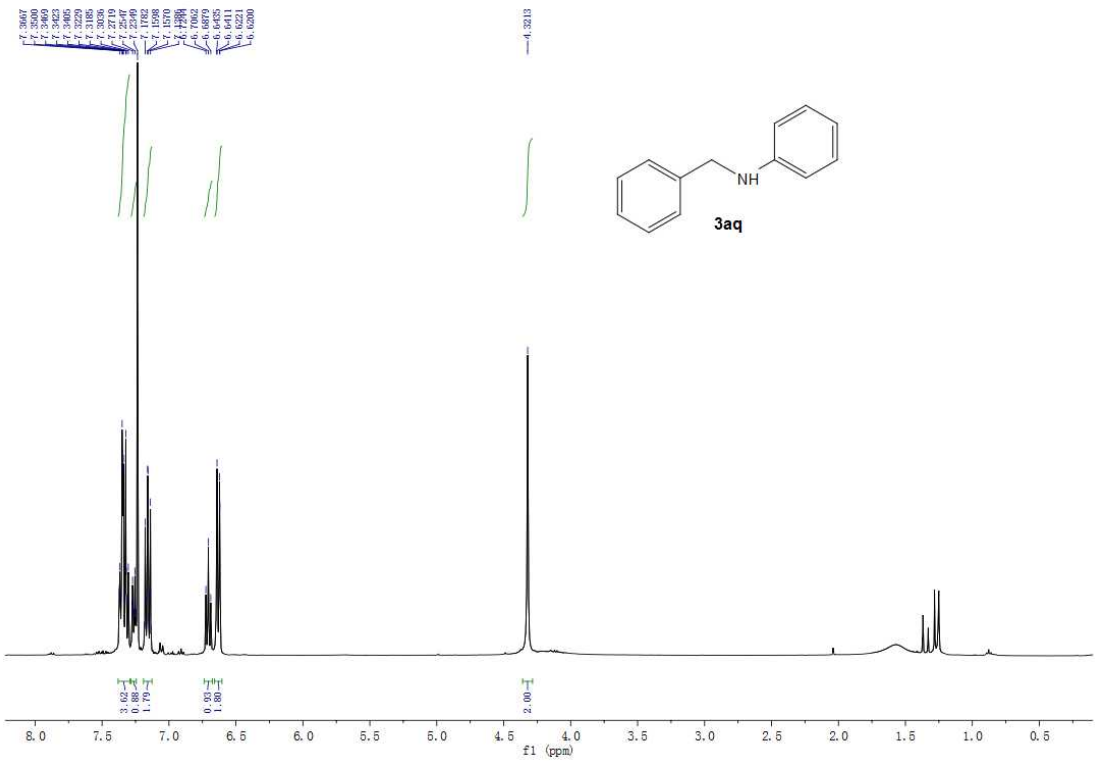

156.33
148.45
133.59
126.51
124.13
120.53
120.20
119.88
114.74
113.68
77.31
77.00
76.68
55.57
NH
F
F
F
O
CH3
3db
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

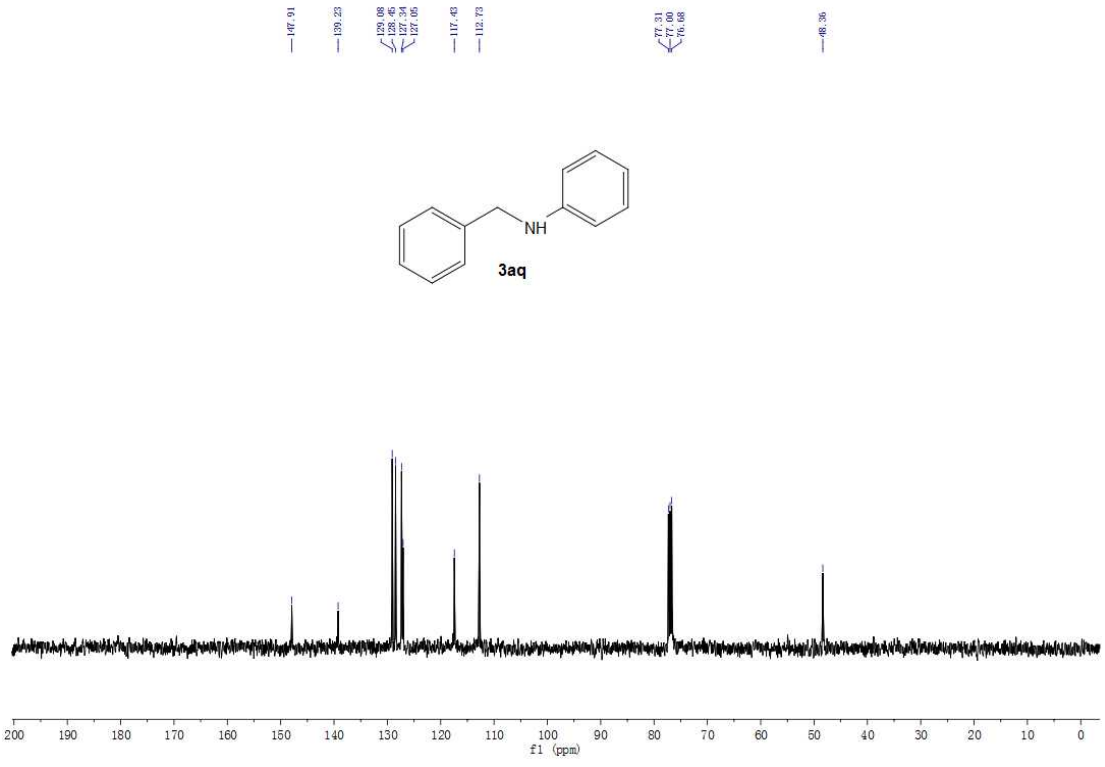

7.2360
7.1342
7.1161
7.0709
7.0517
7.0329
7.0020
6.9815
6.9666
6.8505
6.8355
6.8082
6.7897
6.7719
3.7882
2.2443
CH3
NH
H3C
O
3eb
0.75
0.81
2.55
1.43
1.20
2.93
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

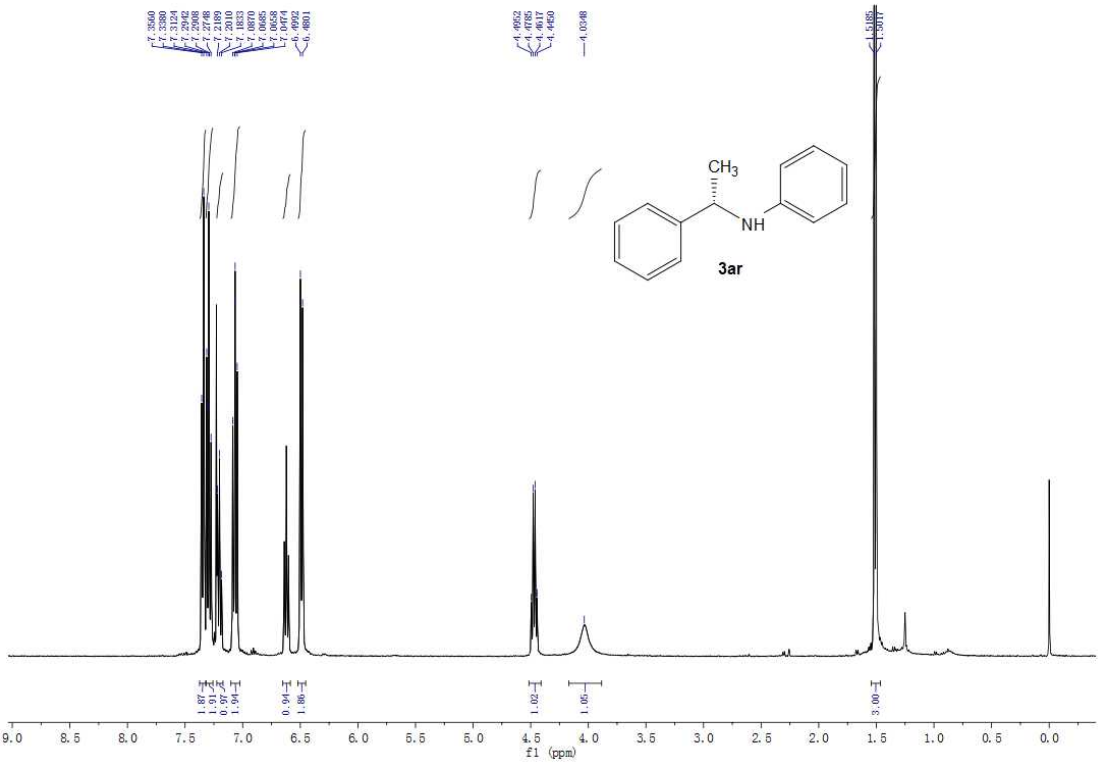

155.21
143.42
136.42
130.89
126.95
125.50
122.28
120.23
115.44
114.88
77.64
77.32
77.01
55.95
55.92
18.22
CH3
NH
H3C
O
3eb
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

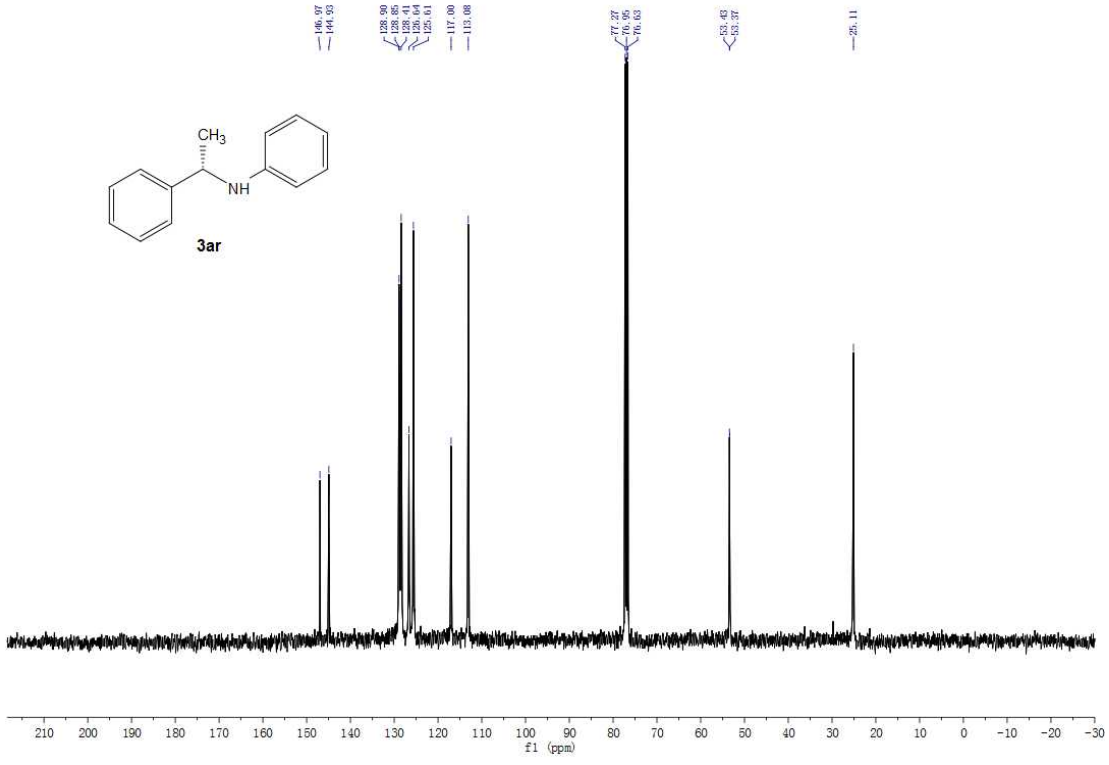

7.1324
7.1194
7.0337
7.0119
6.8533
6.8311
6.7987
6.7768
3.7862
NH
Cl
O
CH3
3fb
2.00
2.02
2.19
2.06
3.14
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

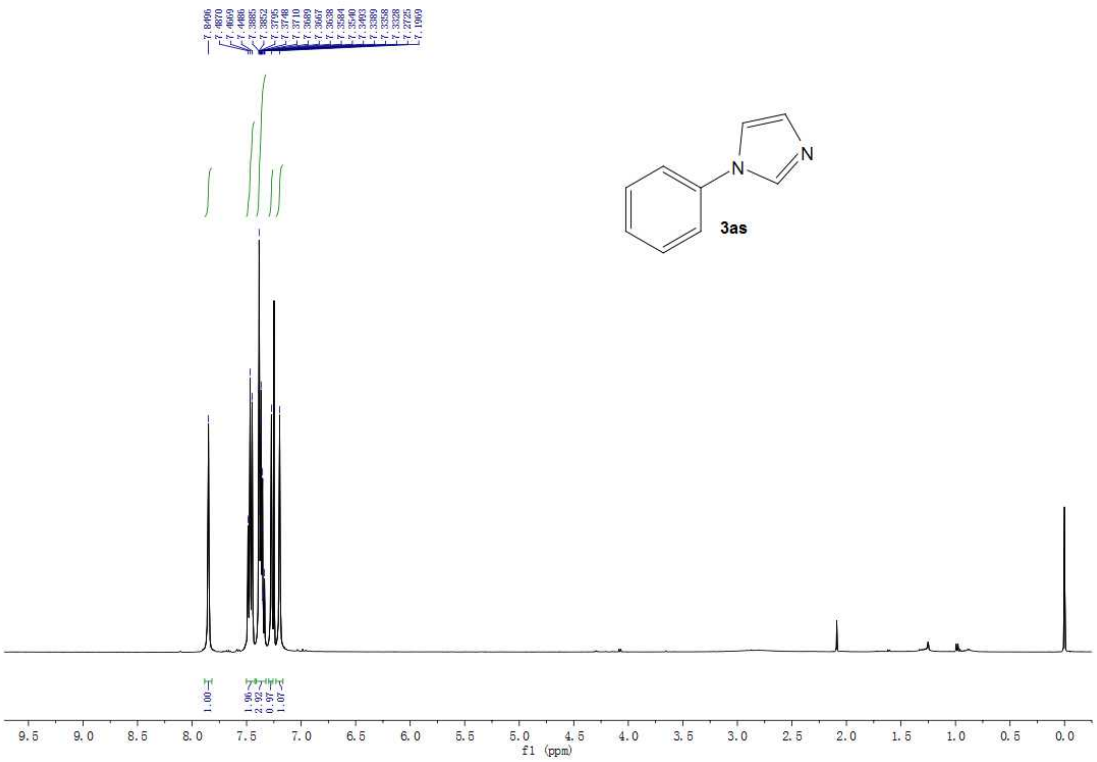

155.51
143.78
135.11
129.03
123.91
122.48
116.58
114.70
77.31
77.00
76.68
55.62
NH
Cl
O
CH3
3fb
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

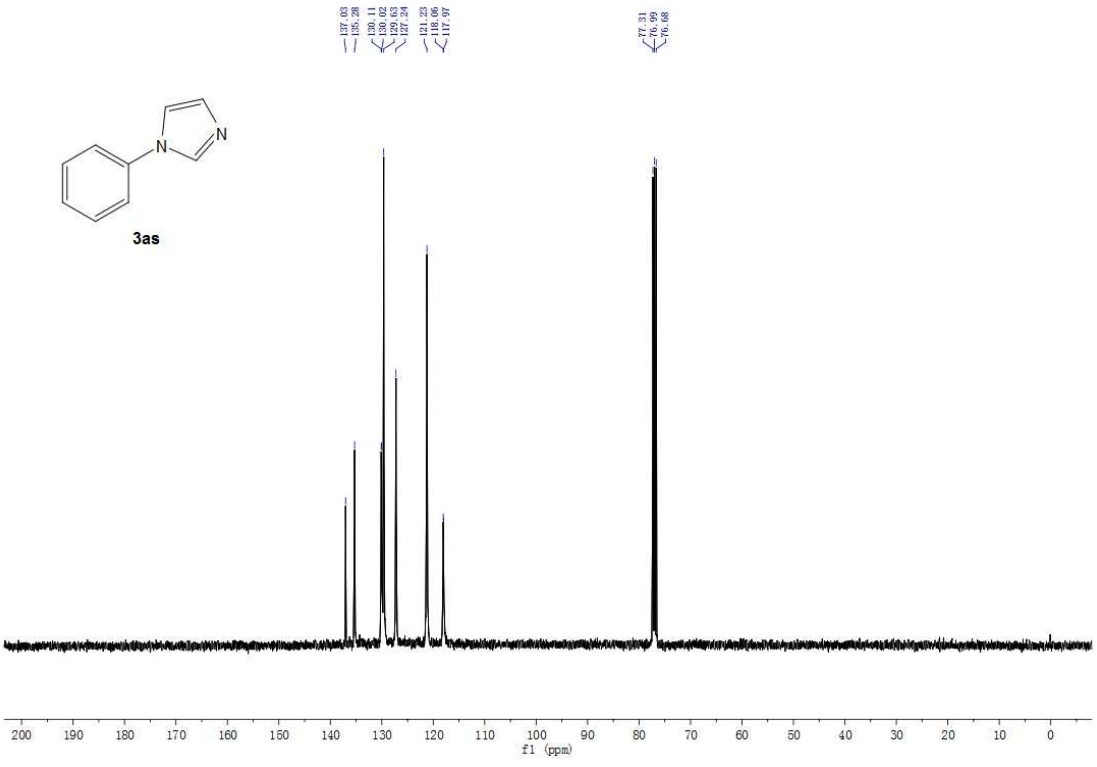

NH
3aq
f1 (ppm)

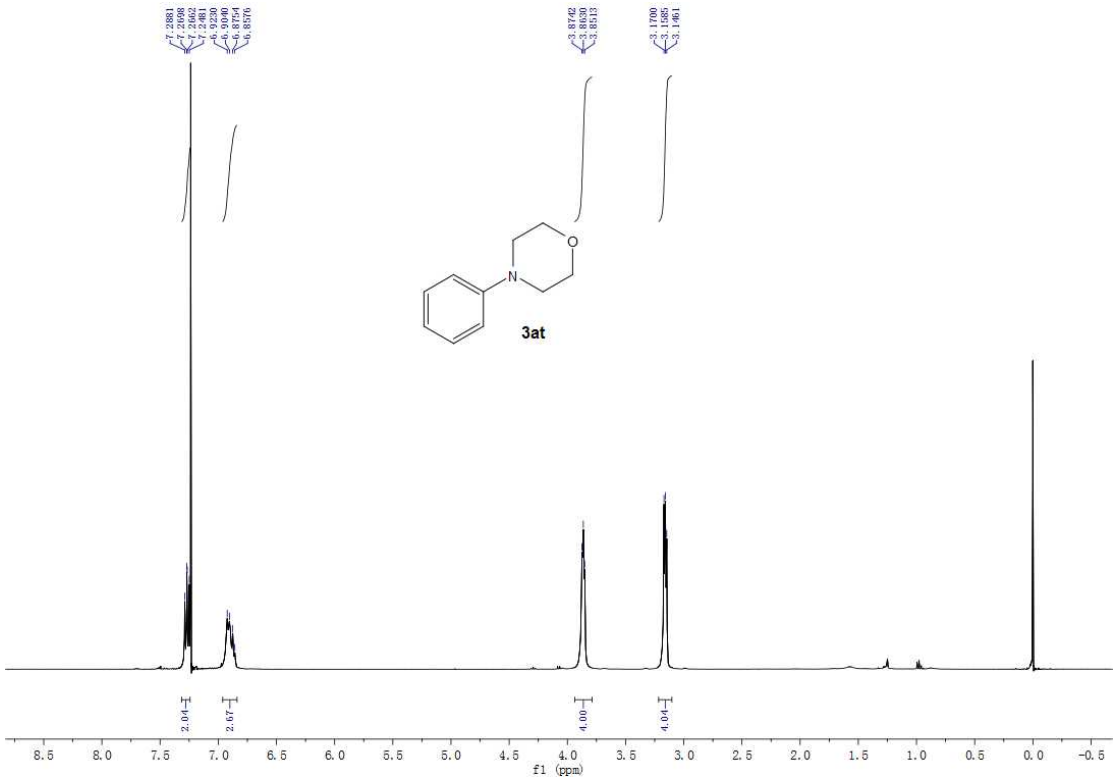

NH
3aq
f1 (ppm)


CH3
NH
3ar
f1 (ppm)


CH3
NH
3ar
f1 (ppm)


3as
f1 (ppm)


3as
f1 (ppm)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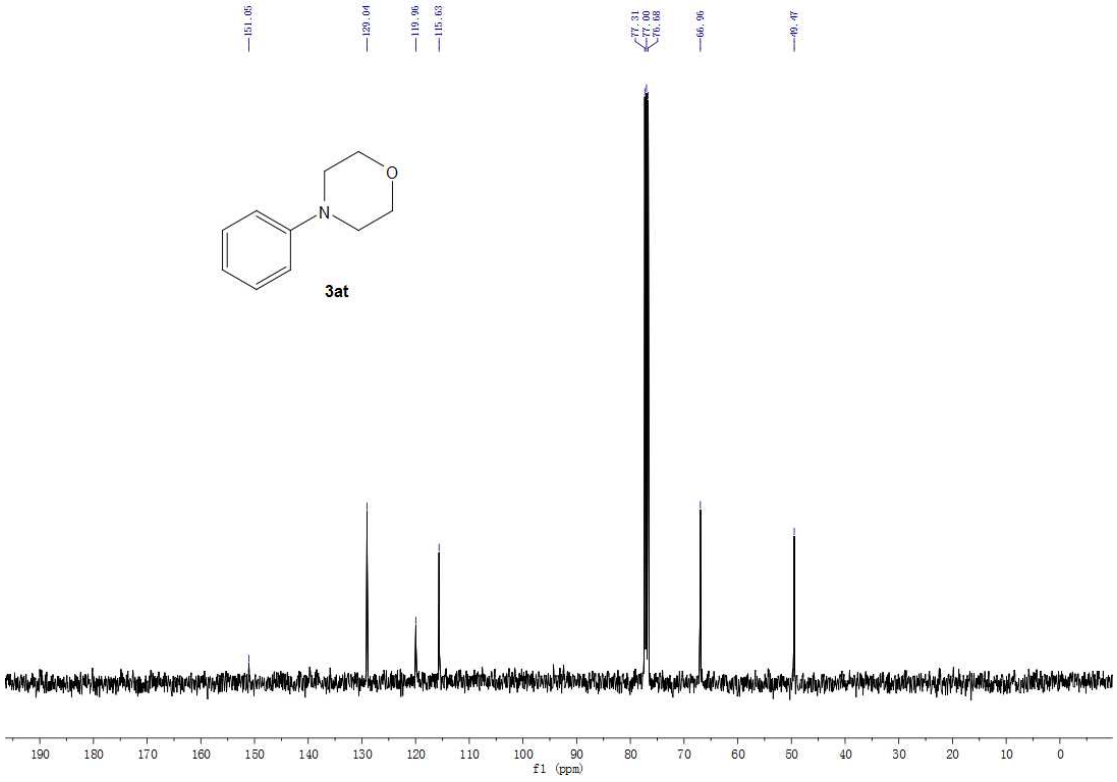
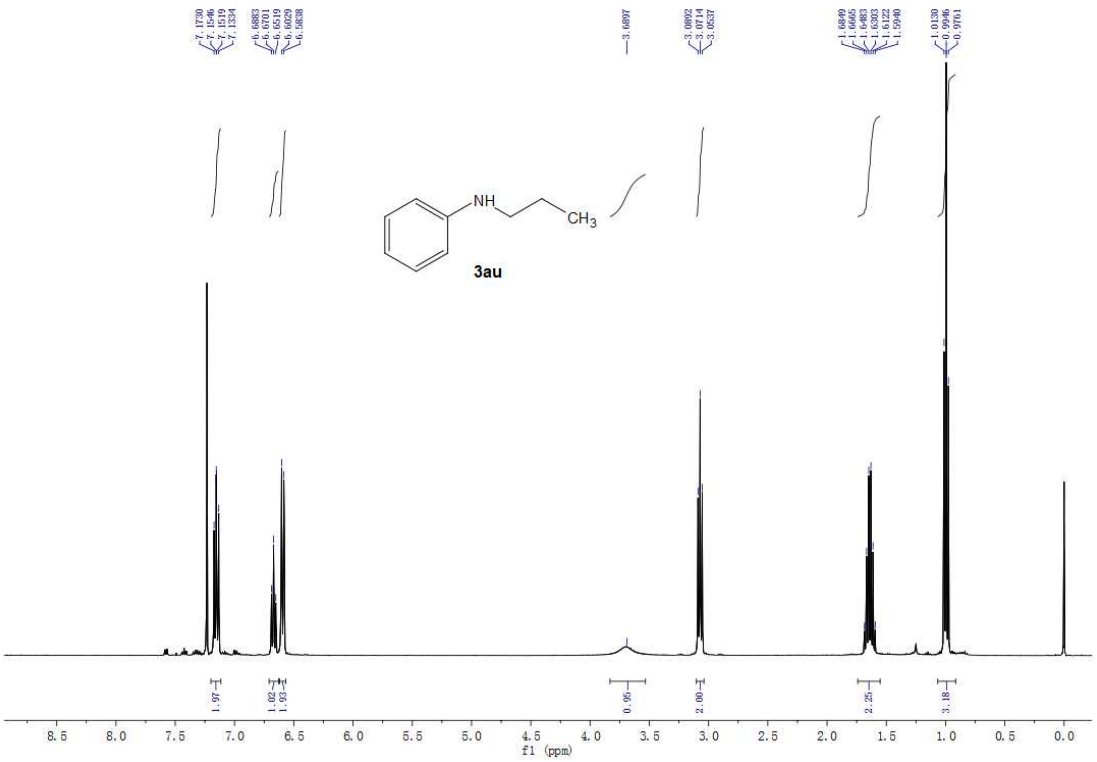
7.2662
7.2481
6.9230
6.9090
6.8954
6.8576
3.8742
3.8630
3.8513
3.1700
3.1585
3.1461
O
N
3at
2.04
2.67
4.00
4.04
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


151.05
129.04
119.96
115.63
77.31
77.00
76.68
66.96
49.47
O
N
3at
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.1546
7.1519
7.1334
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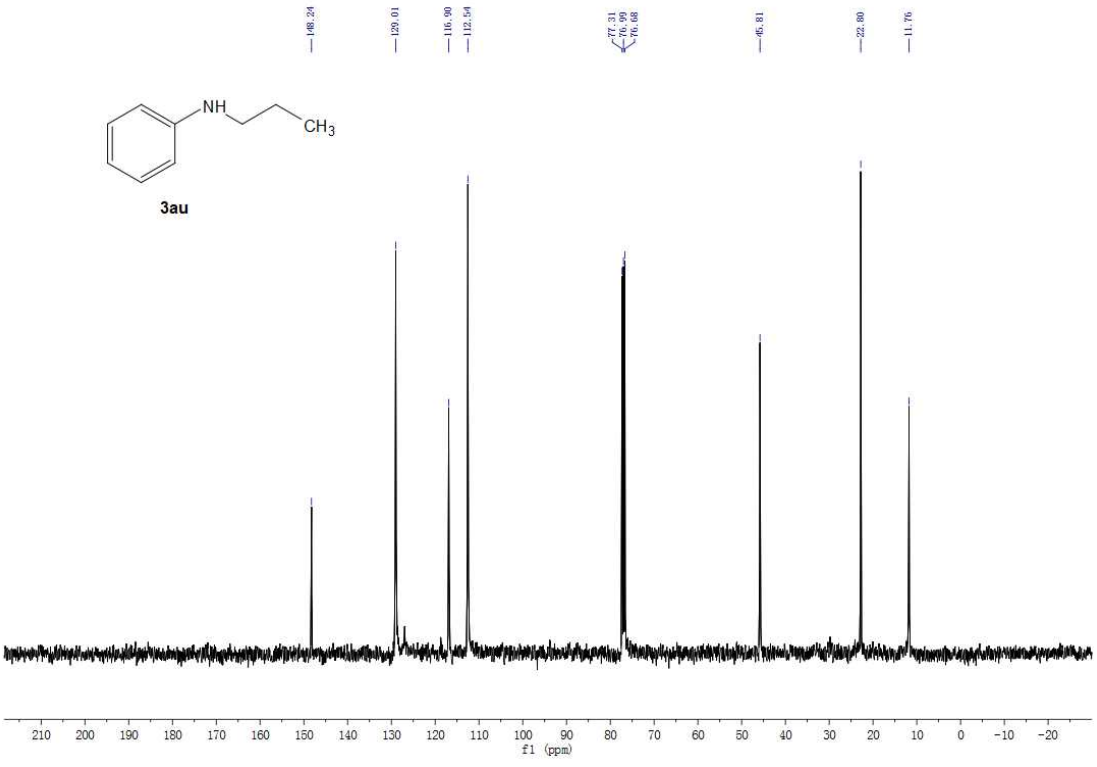
6.6701
6.6519
6.6029
6.5838
3.6897
3.0992
3.0714
3.0537
1.6849
1.6665
1.6483
1.6303
1.6122
1.5940
1.0130
0.9946
0.9761
NH
CH3
3au
1.97
1.02
1.93
0.95
2.00
2.25
3.18
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


148.24
129.01
116.90
112.54
77.31
76.99
76.68
45.81
22.80
11.76
NH
CH3
3au
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)