Electronic Supplementary Material (ESI)
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Supporting Information

Heterogeneous Cobalt Catalysts for the Reductive Amination with H_2: General Synthesis of Secondary and Tertiary Amines

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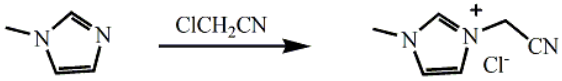
Experimental section

General Methods and Reagents

All reagents were purchased from Aladdin Reagent Company, Sigma-Aldrich Company and Alfa-Aesar Company and used without further purification. ^{1}H-NMR spectra were measured with a Bruker AVANCE 400D spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as internal reference. X-ray photoelectron spectroscopy (XPS) data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300 W AlKa radiations. X-ray diffraction (XRD) patterns were collected on the Bruker D8 Advance powder diffractometer using Ni-filtered Cu Kα radiation source at 40 Kv and 20 mA, from 5°C to 80°C with a scan rate of 0.5 °C/min. The base pressure was about $3x10^{-9}$ mbar. SEM images were performed on a HITACHI S-4800 field-emission scanning electron microscope and TEM images were obtained using a JEOL JEM-2010 (200 kV) TEM instrument. BET surface areas were measured at the temperature of liquid nitrogen using a Micromeritics ASAP2010 analyzer. The samples were degassed at 150 °C to vacuum of 10^{-3} Torr before analysis. The amount of Co was measured using a Jarrell-Ash 1100 ICP-AES spectrometer (Inductively Coupled Plasma-Atomic Emission Spectrometry).

The synthesis of [MCNIm]Cl

The ionic liquid [MCNIm]Cl was synthesized as following:



A mixture of 1-methylimidazole (8.21 g, 100 mmol) and $ClCH_2CN$ (9.06 g, 120 mmol) was stirred at room temperature for 24 hours, the solid could be formed in the process of reaction. The formed solid was then washed with diethyl ether (3*50 mL) and dried under vacuum for 24 hours. Finally, the [MCNIm]Cl was synthesized with 96% yield (15.1 g).

Procedure for the preparation of the Co@NC catalyst

The tyipcal procedure for the preparation of the catalysts is described as follows: A mixture of $Co(OAc)_2.4H_2O$ (0.249 g, 1.0 mmol) and [MCNIm]Cl (0.772 g, 3.0 mmol) in methanol was stirred for 30 minutes at room temperature. Then, the activated carbon powder(0.75 g) was added and the whole reaction mixture was stirred at 50 °C for 5 hours. The reaction mixture was cooled to room temperature and methanol was removed slowely under vacuum. The remaining solid sample obtained was dried at 60 °C for 12 hours. The dried sample was grinded to a powder. Then, the grinded powder was pyrolyzed at 600 °C - 800 °C for 2-3 hours under nitrogen atmosphere. The yield of Co@NC (800-2h) catalyst was 0.985g after pyrolysis at 800 °C for 2 hours. Yields of other catalysts were listed in Table S1. The Co/C(800-2h) catalyst was synthesized without adding [MCNIm]Cl with the same others conditions as above. The IL/C(800-2h) catalyst was synthesized without adding $Co(OAc)_2.4H_2O$ with similar conditions. The Co-IL/C catalyst was synthesized without pyrolysis.

ICP-AES analysis of Co@NC (800-2h): Co = 5.95

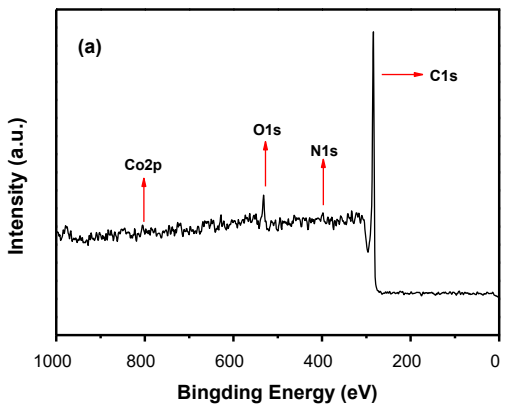
XPS data of Co@NC (800-2h) (Atom%): C = 92.47, N = 2.86, O = 4.28, Co = 0.39

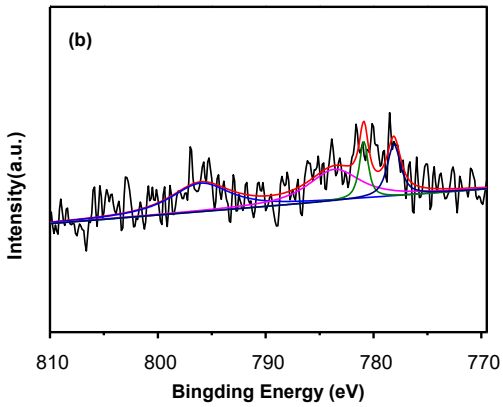
Table S1 the pyrolysis condition of the Co@NC catalyst

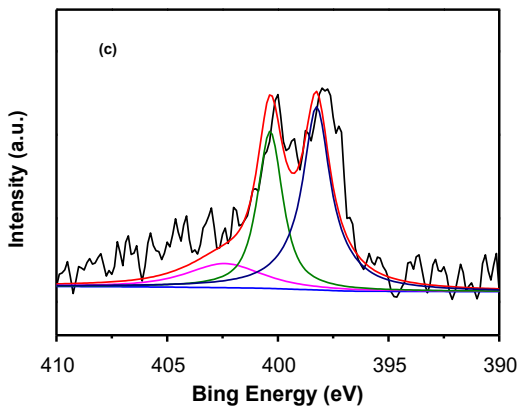
Entry	Catalyst	pyrolysis temperature (^{o}C)	pyrolysis time (h)	Yield of the Co catalysts (g)	Co content (Wt %)
1	Co@NC (600-2h)	600	2	0.993	5.93
2	Co@NC (700-2h)	700	2	0.985	5.98
3	Co@NC (800-2h)	800	2	0.984	5.95
4	Co@NC (800-3h)	800	3	0.977	6.03
5 [a]	Co/C(800-2h)	800	2	0.902	6.53
6 [b]	IL/C(800-2h)	800	2	0.934	--
7 [c]	Co-IL/C	no	no	1.885	0.312

[a] without adding [MCNIm]Cl. [b] without adding $Co(OAc)_2.4H_2O$. [c] without pyrolyzing.

Characterization of the Co@NC (800-2h) catalyst







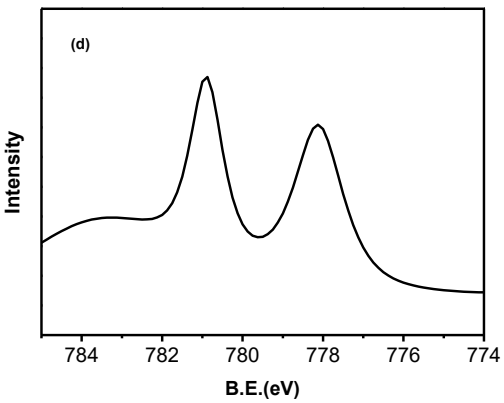


Figure S1 (a) XPS survey spectrum for Co@NC (800-2h). High-resolution XPS survey spectra of (b) Co 2p:780.91 eV is CoO, 778.12 eV is Co and (c) N 1s for Co@NC (800-2h):398.3 eV is pyridine-type nitrogen, 400.4 eV is pyrrole-type nitrogen, 402.4 eV is carbon nitrogen. (d) A close look at the XPS spectrum of Co 2p in Co@NC (800-2h).

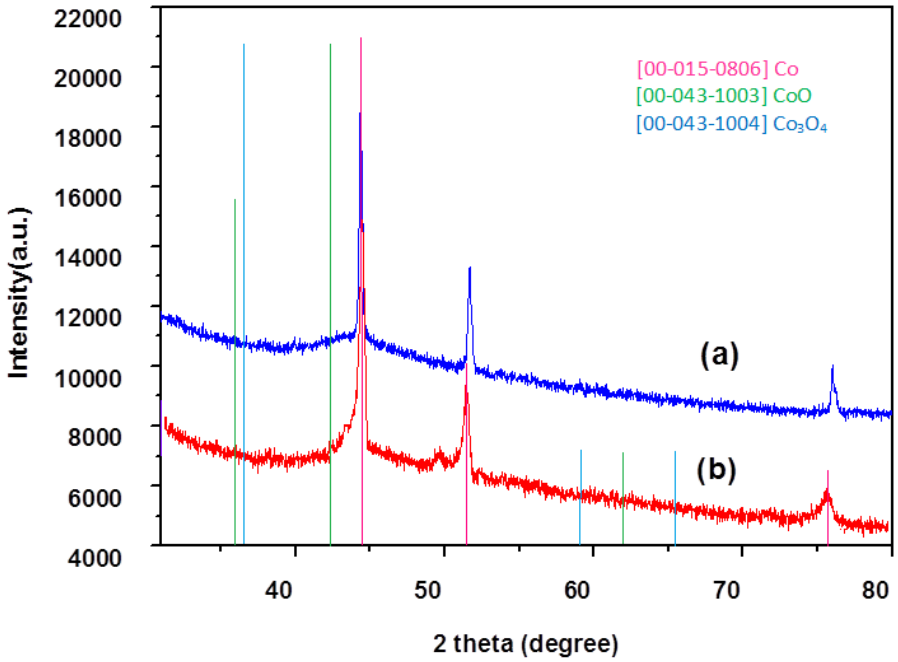


Figure S2 The peak locations of catalyst comparing with standard of Co/CoO/Co_3O_4 :(a) Co@NC(800-2h), (b) recycled Co@NC (800).

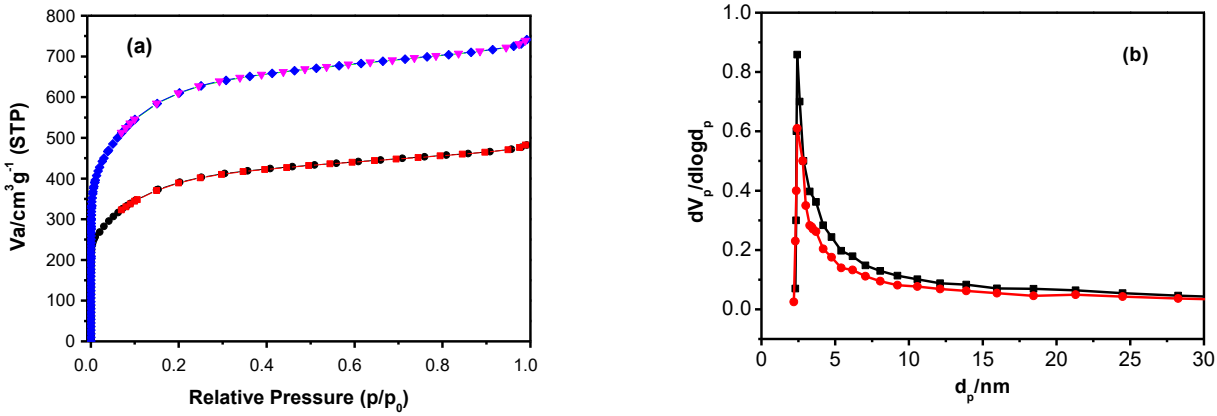


Figure S3 (a) Nitrogen adsorption-desorption isotherms at 77 K of AC (top):ADS(◆) and DES(▼); Co@NC (800-2h) (bottom):ADS(■) and DES(●) . (b) pore size distribution curves of AC (■) and Co@NC (800-2h) (●)

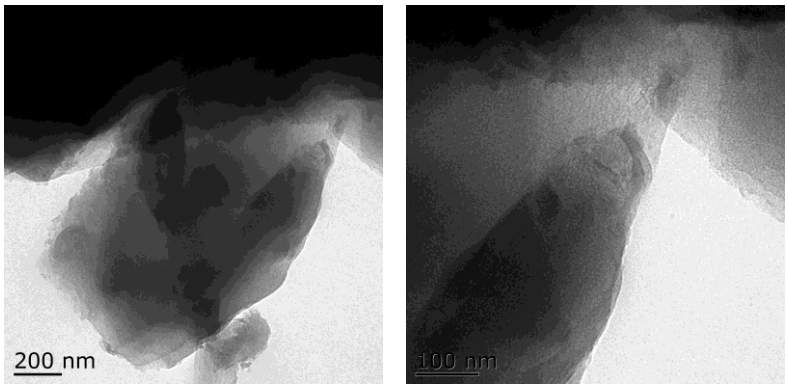


Figure S4 TEM images for Co@NC (800-2h).

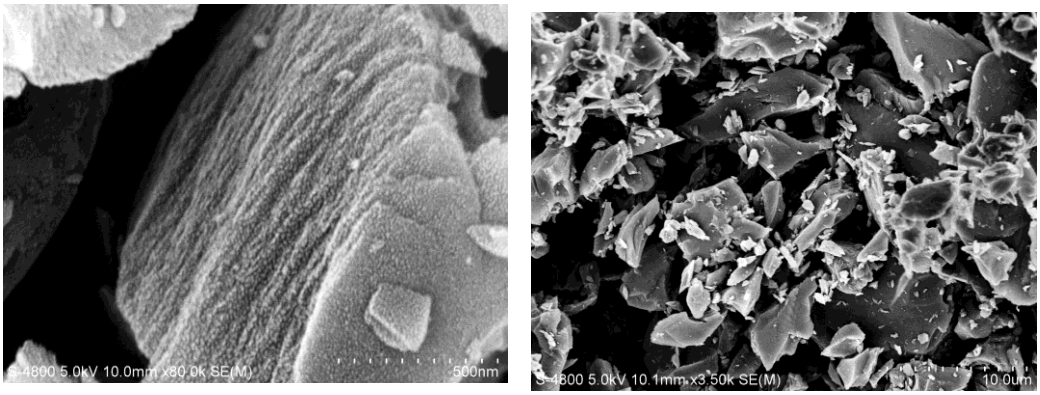
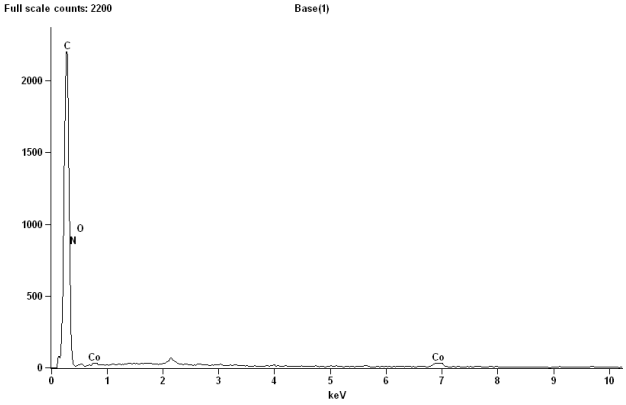


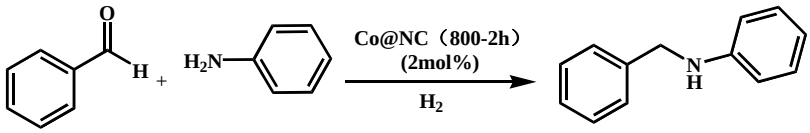
Figure S5 SEM images for Co@NC (800-2h)



Element Line	Weight %	Atom %
C K	85.84	89.05
N K	11.05	9.83
O K	0.80	0.63
CoK	2.3	0.49

Figure S6 EDX analysis for the Co@NC (800-2h)

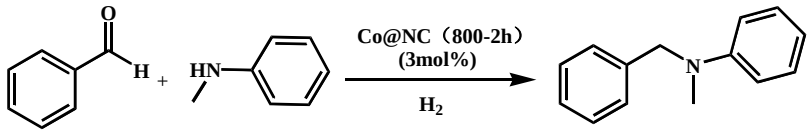
Table S2 The optimization of reaction conditions for the reductive amination of benzaldehyde with aniline [a]



Entry	Solvent	Temp (℃)	Time (h)	H_2 pressure (bar)	Conversion [b] (%)	Yield [b] (%)
1	acetonitrile	120	18	10	85	82
2	1,4-dioxane	120	18	10	83	81
3	o-xylene	120	18	10	93	90
4	toluene	120	18	10	100	98
5	toluene	110	18	10	100	98
6	toluene	90	18	10	87	74
7	toluene	110	14	10	99	63
8	toluene	110	18	5	90	46

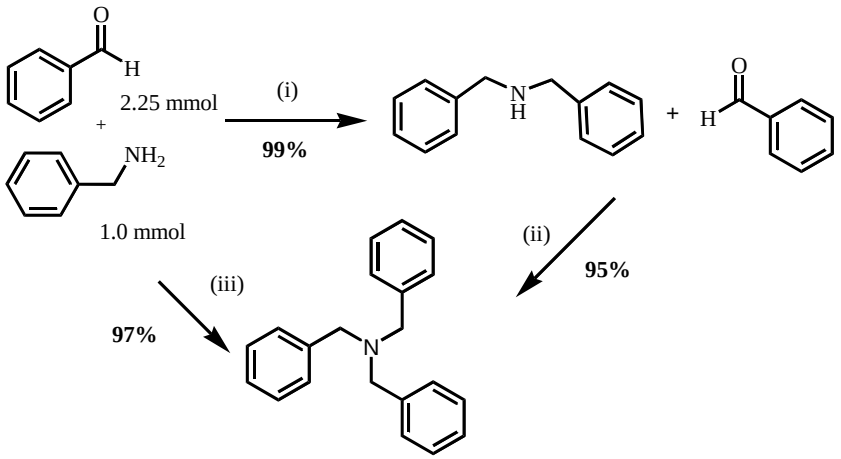
[a] Reaction conditions: 1.0 mmol aniline, 1.5 mmol benzaldehyde; Co 2.0 mol% added; solvent (2.0 mL). [b] Determined by GC. In case of lower yields, the imine was detected as the by-product.

Table S3 The optimization of reaction conditions for the reductive amination of benzaldehyde with N-methylaniline [a]



Entry	Solvent	Temp (℃)	Time (h)	H_2 pressure (bar)	Conversion [b] (%)	Yield [b] (%)
1	acetonitrile	140	18	30	80	80
2	1,4-dioxane	140	18	30	84	84
3	o-xylene	140	18	30	95	95
4	toluene	140	18	30	98	98
5	toluene	130	18	30	92	92
6	toluene	130	24	30	93	93
7	toluene	140	18	20	65	65
8	toluene	140	18	10	17	17

[a] Reaction conditions: 1.0 mmol N-methylaniline, 1.5 mmol benzaldehyde; Co 3.0 mol% added; solvent (2.0 mL). [b] Determined by GC.



Scheme S1 Control reactions for the reductive amination of benzaldehyde with benzylamine
Reaction conditions for (i): Co@NC(800-2h), 3.0 mol% 110 ^{o}C, 10 bar H_2, 18h, (ii): 140 ^{o}C, 30 bar H_2, 18h, (iii): Co@NC(800-2h), 3.0 mol% 140 ^{o}C, 30 bar H_2, 18h.

Reaction (i): Reactions were performed in a 30 mL stainless steel autoclave equipped with a stirring bar. Benzylamine (1.0 mmol), benzaldehyde (2.25 mmol), toluene (2.0 mL) and 3.0 mol % Co@NC(800-2h) were placed into the autoclave. Then, the autoclave was purged with hydrogen three times, and pressurized to 10 bar hydrogen and heated at 110 ℃ for 18 h. After the autoclave was cooled to room temperature, the sample was analyzed by GC. **Reaction (ii):** Then, the reactor was purged with hydrogen and pressurized to 30 bar hydrogen and heated at 140 ℃ for another 18 h. After the autoclave was cooled to room temperature, the sample was analyzed by GC

Reaction (iii): Benzylamine (1.0 mmol), benzaldehyde (2.25 mmol), toluene (2.0 mL) and 3.0 mol % Co@NC(800-2h) were added to the autoclave. Then, the autoclave was purged with hydrogen three times and pressurized to 30 bar hydrogen and heated to 140 ℃ for 18 h. After the autoclave was cooled to room temperature, the sample was analyzed by GC.

Typical procedure for the reductive amination of benzaldehyde and aniline over Co@NC(800-2h) catalyst (Table 1)

Reactions were performed in a 30 mL stainless steel autoclave equipped with a stirring bar. Aniline (1.0 mmol), benzaldehyde (1.5 mmol), toluene (2 mL) and Co catalyst containing Co 2.0 mol % were added into the autoclave. Then, the autoclave was purged with hydrogen three times, and pressurized to 10 bar hydrogen and heated to 110 ℃ for 18 h. After the autoclave was cooled to room temperature, the sample was analyzed by GC

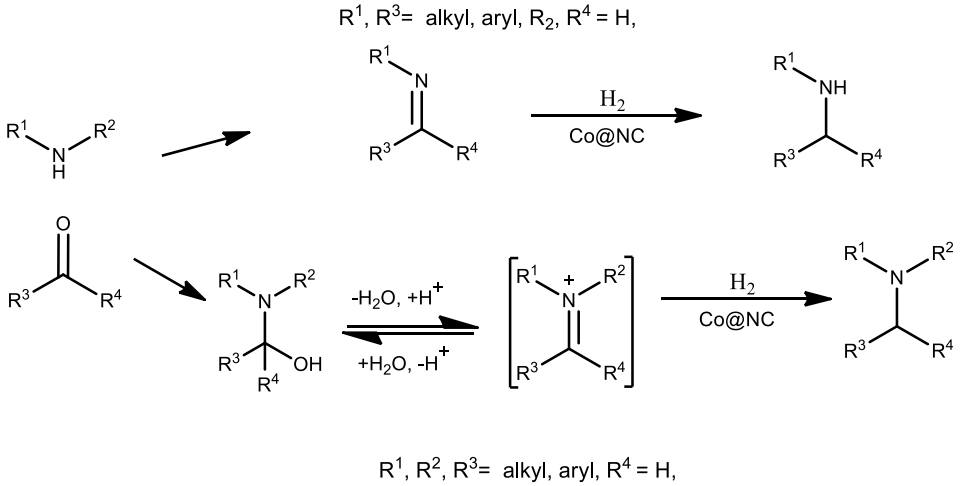
General procedure for the reductive amination of aldehydes and ketones with primary amines (Table 2 and Table S2)

Reactions were performed in a 30 mL stainless steel autoclave equipped with a stirring bar. Amine (1.0 mmol), aldehyde/ketone (1.5 mmol), toluene (2.0 mL) and 2.0 mol % Co@NC(800-2h)

were added into the autoclave. Then, the autoclave was purged with hydrogen three times, and pressurized to 10 bar hydrogen and heated to 110 ℃ for 18 h. After the autoclave was cooled to room temperature, the solution was filtered and concentrated, and the product was isolated by chromatography on a silica gel column with hexane and ethyl acetate.

General procedure for the reductive amination of aldehydes and ketones with secondary amines (Table 3 and Table S3)

Reactions were performed in a 30 mL stainless steel autoclave equipped with a stirring bar. Amine (1.0 mmol), aldehyde/ketone (1.5 mmol), toluene (2.0 mL) and 3.0 mol % Co@NC(800-2h) were added into the autoclave. Then, the autoclave was purged with hydrogen three times, and pressurized to 30 bar hydrogen and heated to 140 ℃ for 18 h. After the autoclave was cooled to room temperature, the solution was filtered and concentrated, and the product was isolated by chromatography on a silica gel column with hexane and ethyl acetate.



Scheme S2 Proposed mechanism of the reductive amination of aldehydes and ketones with amines over Co@NC(800-2h).

General procedure for the synthesis of N-substituted isoindolinones (Scheme 2)

Amine (1.0 mmol), 2-carboxybenzaldehyde (1.0 mmol), toluene (2.0 mL) and 3.0 mol % Co@NC (800-2h) were added into the autoclave. Then, the autoclave was purged with hydrogen three times, and pressurized to 30 bar hydrogen and heated to 140 ℃ for 18 h. After the autoclave was cooled to room temperature, the solution was filtered and concentrated, and the product was isolated by chromatography on a silica gel column with hexane and ethyl acetate.

Recycling Procedure

The reductive amination of benzaldehyde with aniline

Initially, the stainless steel autoclave equipped with a stirring bar was added in aniline (0.093 g, 1.0 mmol), benzaldehyde (0.159 g, 1.5 mmol), toluene (2.0 mL) and Co@NC (800-2h) (20mg, 2 .0 mol% Co). Then, the autoclave was purged with hydrogen three times and pressurized to 10 bar hydrogen and heated at 110 ℃ for 18 h. After the autoclave was cooled to room temperature,

the reaction mixture was extracted with ethyl acetate (3 × 6 mL). The organic extract was dried with anhydrous $MgSO_4$ and analyzed by GC to determine the yield. The catalyst Co@NC (800-2h) was collected by centrifugation and washed with ethyl acetate. The recovered Co@NC (800-2h) was used again for the reaction under the same action conditions.

The reductive amination of benzaldehyde and N-methylaniline

The stainless steel autoclave equipped with a stirring bar was added in N-methylaniline (0.107 g, 1.0 mmol), benzaldehyde (0.159 g, 1.5 mmol), toluene (2.0 mL) and Co@NC (800-2h) (30 mg, 3 .0 mol% Co). Then the autoclave was purged with hydrogen three times and pressurized to 30 bar hydrogen and heated at 140 ℃ for 18 h. After the autoclave was cooled to room temperature, the reaction mixture was extracted with ethyl acetate (3 × 6 mL). The organic extract was dried with anhydrous $MgSO_4$ and analyzed by GC to determine the yield. The catalyst Co@NC (800-2h) was collected by centrifugation and washed with ethyl acetate. The recovered Co@NC (800-2h) was used again for the reaction under the same action conditions.

The product data.

Secondary amines:

N-benzylaniline (Table 2, entry 1)

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.36 (s, 2H), 6.67-7.77 (m, 3H), 7.21 (t, *J* = 8.4 Hz, 2H), 7.30-7.42 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ148.4, 139.8, 129.6, 128.9, 127.8, 127.6, 117.9, 113.1, 48.7; IR (neat): 3419, 3026, 2924, 2853, 1949, 1602, 1505, 1324, 1267, 989, 749.

N-(4-fluorobenzyl)aniline (Table 2, entry 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.81 (s, 2H), 7.23 (t, *J* = 8.3Hz, 3H), 7.48 (t, *J* = 6.8Hz, 2H), 7.55-7.58 (m, 1H), 7.72 (d, *J* = 8.5Hz, 2H), 7.91 (t, *J* = 7.4Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 163.7, 160.5, 148.0, 135.2, 129.4, 129.1, 129.0, 117.8, 115.6, 115.4, 112.9, 47.7; IR (neat): 3416, 1600, 1504, 1220.

N-(2-Chlorobenzyl)aniline (Table 2, entry 3)

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.50 (s, 2H), 6.68-6.75 (m, 2H), 7.21-7.27 (m, 5H), 7.42-7.57 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.8, 136.7, 133.2, 129.6, 129.4, 129.0, 128.4, 127.0, 117.8, 112.9,

45.9; IR (neat): 3425, 3018, 2914, 2833, 1864, 1600, 1502, 1453, 1321, 1177, 1094, 915, 815, 733, 698, 505.

N-(4-methoxybenzyl)aniline (Table 2, entry 4)

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.84 (s, 3H), 4.29 (s, 2H), 6.67-6.78 (m, 3H), 6.92 (d, *J* = 8.4Hz, 2H), 7.22 (t, *J* = 7.6Hz, 2H), 7.33 (d, *J* = 8.2Hz,2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 148.4, 131.6, 129.5, 129.1, 117.8, 114.3, 113.1, 55.5, 48.1; IR (neat): 3416, 3019, 2930, 2835, 1922, 1603, 1508, 1321, 1247, 1177, 1034, 824, 750, 692.

N-(3-methoxybenzyl)aniline (Table 2, entry 5)

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.83 (s, 3H), 4.34 (s, 2H), 6.69-6.86 (m, 4H), 6.99 (d, *J*=11.6 Hz, 2H), 7.20-7.29 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.9, 148.1, 141.2, 129.6, 129.2, 119.7, 117.6, 113.0, 112.8, 112.6, 55.2, 48.3; IR (neat): 3417, 3015, 2920, 2833, 1912, 1600, 1518, 1311, 1227, 1177, 1035, 826, 752, 694.

N-(4-methylbenzyl)aniline (Table 2, entry 6)

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.39 (s, 3H), 4.33 (s, 2H), 6.69-6.79 (m, 3H), 7.20-7.32 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 136.9, 136.5, 129.6, 129.3, 127.8, 117.7, 113.2, 48.3, 21.2; IR (neat): 3418, 2920, 1603, 1507, 1322, 1255, 1179, 749.

N-(2-methylbenzyl)aniline (Table 2, entry 7)

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.32 (s, 3H), 4.32 (s, 2H), 6.86 (s, 3H), 7.22 (t, *J* = 15.0 Hz, 5H), 7.41 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 148.3, 137.1, 136.3, 130.4, 129.3, 128.3, 127.4, 126.2, 117.5, 112.7, 46.4, 18.9; IR (neat): 3419, 2930, 1934, 1603, 1507, 1321, 1245, 1177, 749.

N-(4-Chlorophenyl)benzylamine (Table 2, entry 8)

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.29 (s, 2H), 6.54 (t, J = 8.8Hz, 2H), 7.10 (t, J = 8.8Hz, 2H), 7.25-7.29

(m, 1H), 7.32 (t, J = 8.6Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9, 139.2, 129.3, 128.9, 127.7, 127.5, 122.4, 114.3, 48.6; IR (neat): 3427, 3028, 2924, 2853, 1952, 1864, 1600, 1502, 1453, 1401, 1321, 1177, 1094, 915, 815, 733, 698, 505.

N-(4-cyanophenyl)benzylamine (Table 2, entry 9)

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.40 (s, 2H), 6.62 (d, *J*=8.6 Hz, 2H), 7.34-7.45 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.1, 137.8, 133.6, 128.8, 127.6, 127.2, 120.4, 112.3, 98.9, 47.4; IR (neat): 3357, 3018, 2914, 2843, 1942, 1864, 1600, 1502, 1443, 1407, 1325, 1177, 1084, 916, 817, 734, 698, 506.

N-(4-methylphenyl)benzylamine (Table 2, entry 10)

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.27 (s, 3H), 4.34 (s, 2H), 6.60 (d, *J* = 8.1 Hz, 2H), 7.02 (d, *J* = 7.8 Hz, 2H), 7.29 (d, *J* = 4.8 Hz, 1H), 7.35-7.41 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 139.9, 130.0, 128. 9, 127.8, 127.4, 127.0, 113.3, 48.9, 20.7; IR (neat): 3445, 3027, 2918, 2763, 1951, 1865, 1701, 1618, 1522, 1452, 1302, 1126, 807, 742, 697, 511.

N-(3-Methylphenyl)benzylamine (Table 2, entry 11)

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.18 (s, 3H), 4.23 (s, 2H), 6.50 (d, *J* = 36.2 Hz, 3H), 6.92 (d, *J* = 74.9 Hz, 1H), 7.20 (d, *J* = 34.3 Hz, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 139.5, 139.2, 129.4, 128.9, 119.0, 114.1, 110.3, 48.8, 21.7; IR (neat): 3446, 3029, 2915, 2767, 1950, 1815, 1704, 1615, 1523, 1452, 1302, 1126, 805, 742, 693, 511.

N-(4-methoxyphenyl)benzylamine (Table 2, entry 12)

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.84 (s, 3H), 4.29 (s, 2H), 6.67-6.78 (m, 3H), 6.92 (d, *J* = 8.4 Hz, 2H), 7.22 (t, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.5, 142.4, 139.7, 128.8, 127.8, 127.4, 115.1, 114.5, 56.0, 49.5; IR (neat): 3375, 2944, 1629, 1511, 1456,1236, 1033,

820.

N-(2-methoxyphenyl)benzylamine (Table 2, entry 13)

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.79 (s, 3H), 4.29 (s, 2H), 6.45-6.83 (m, 4H), 7.23 (d, *J* = 39.3Hz, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 139.6, 138.1, 128.6, 127.5, 127.1, 121.3, 116.6, 110.1, 109.4, 55.4, 48.0; IR (neat): 3376, 1607, 1512, 1403, 1239, 1177, 1076.

N-[2,6-Bis(1-methylethyl)phenyl]benzylamine (Table 2, entry 14)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.17 (d, *J* = 6.9Hz, 12H), 2.94-3.02 (m, 2H), 4.29 (s, 2H), 7.08-7.11 (m, 1H), 7.15 (d, *J* = 7.2Hz, 2H), 7.50 (d, *J* = 1.5Hz, 3H), 7.90-7.92 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.2, 140.1, 129.0, 128.1, 126.6, 122.7, 56.1, 29.2, 23.4; IR (neat): 3419, 3016, 2931, 2843, 1929, 1602, 1505, 1324, 1277,1092, 989, 814, 749, 692.

N-(2-pyridylmethyl)aniline (Table 2, entry 15)

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.52 (s, 2H), 6.68-6.76 (m, 3H), 7.18-7.26 (m, 3H), 7.41 (d, *J*=7.8Hz, 1H), 7.71 (t, *J*=6.2Hz, 1H), 8.61 (d, *J*=4.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 158.6, 149.1, 147.9, 136.7, 129.2, 122.1, 121.6, 117.5, 113.0, 49.2; IR (neat): 3416, 3028, 2972, 2843, 2788, 1566, 1542, 1028, 737, 700.

N-Isopropylaniline (Table 2, entry 16)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.27 (d, *J* = 6.3Hz, 6H), 3.65-3.72 (m, 1H), 6.65-6.75 (m, 3H), 7.22 (t, *J* = 7.5Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.4, 129.1, 116.7, 113.1, 43.9, 22.7. ; IR (neat): 3400, 3050, 3018, 2964, 2828, 2869, 1596, 1600, 1503, 1314, 1254, 1176, 745, 691.

N-Cyclopentylaniline (Table 2, entry 17)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.50-1.56 (m, 2H), 1.67-1.82 (m, 4H), 2.05-2.11 (m, 2H), 3.82-3.87 (m, 1H), 6.66 (d, *J* = 8Hz, 2H), 6.73 (t, *J* = 7.3Hz, 1H), 7.22 (t, *J* = 7.5Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$):

δ 148.4, 129.4, 117.1, 113.5, 54.8, 33.8, 24.4. ; IR (neat): 3341, 3047, 2843, 1922, 1731, 1601, 1503, 1447, 1321, 1257, 1176, 1117, 887, 759, 692.

N-Cyclohexylaniline (Table 2, entry 18)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.19-1.48 (m, 5H), 1.71-1.86 (m, 3H), 2.12-2.15 (m, 2H), 3.30-3.36 (m, 1H), 6,66-6.75 (m, 3H), 7.21-7.24 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.4, 129.2, 116.8, 113.1, 51.6, 33.5, 25.9, 25.0; IR (neat): 3399, 3050, 2929, 2853, 1912, 1731, 1601, 1502, 1449, 1320, 1255, 1177, 1147, 1117, 887, 749, 692.

N-Cyclohexylmethylaniline (Table 2, entry 19)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.01-1.09 (m, 2H), 1.23-1.34 (m, 3H), 1.62-1.90 (m, 6H), 3.02 (d, *J* = 6.7Hz, 2H), 6.66-6.76 (m, 3H), 7.21-7.25 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 148.0, 129.3, 116.9, 112.7, 50.7, 37.7, 31.4, 26.7, 26.1. ; IR (neat): 3408, 1600, 1505, 1470, 1447, 745, 691.

N-hexylaniline (Table 2, entry 20)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.89 (t, J = 7.0Hz, 3H), 1.30-1.33 (m, 4H), 1.37-1.43 (m, 2H),1.58-1.64 (m, 2H), 3.10 (t, J = 7.2Hz, 2H), 6.60 (d, J = 7.7Hz, 2H), 6.68 (m, J = 7.3Hz, 1H), 7.15-7.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.7, 129.3, 117.3, 113.0, 44.2, 31.8, 29.7, 27.0, 22.7, 14.7; IR (neat): 3412, 2956, 2928, 1603, 1507, 1321, 1259, 748, 692.

N-butylbenzylamine (Table 2, entry 21)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.91 (t, J= 7.4Hz, 3H), 1.32-1.39 (m, 2H), 1.47-1.51 (m, 2H), 2.63 (t, J = 7.3Hz, 2H), 3.79 (s, 2H), 7.22-7.26 (m, 1H), 7.31 (t, J = 8.6Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.9, 128.6, 128.3, 127.0, 54.4, 49.5, 32.6, 20.8, 14.4; IR (neat): 3322, 3067, 2957, 2871, 1870, 1719, 1646, 1495, 1028, 732, 466.

N-Phenylbenzylimine

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28-7.31 (m, 3H), 7.46 (t, *J* = 7.6Hz, 2H), 7.53-7.55 (m, 3H), 7.96-7.99 (m, 2H), 8.52 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.3, 152.1, 136.2, 131.4, 129.2, 128.8, 128.7, 126.0, 120.9; IR (neat): 3067, 2891, 1627, 1579, 1486, 1452, 1194, 907, 756, 695, 520.

Tertiary amines:

N-Benzyl-N-methylaniline (Table 3, entry 1)

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.40 (s, 3H), 4.33 (s, 2H), 6.69-6.79 (m, 3H), 7.20-7.32 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2, 139.4, 129.6, 128.9, 127.3, 126.9, 116.8, 112.6, 56.9, 38.7; IR (neat): 1599, 1506, 1452, 1355, 1214, 1118, 749, 729, 692.

4-fluoro-N-methyl-N-phenylbenzylamine (Table 3, entry 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.07 (s, 3H), 4.57 (s, 2H), 6.81-6.85 (m, 3H), 7.08 (t, *J* = 8.7Hz, 2H), 7.26-7.33 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.3, 150.2, 134.6, 131.7, 130.1, 128.0, 117.4, 114.8, 56.7, 39.2; IR (neat): 3067, 3017, 2893, 2817, 1597, 1507, 1424, 1371, 1292, 1213, 1114, 1036, 950, 749, 692.

4-Chloro-N-methyl-N-phenylbenzylamine (Table 3, entry 3)

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.07 (s, 3H), 4.55 (s, 2H), 6.80-6.82 (m, 3H), 7.24 (d, *J* = 8.35Hz, 2H), 7.28-7.31 (m, 2H), 7.35 (d, *J* = 8.5Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9, 137.7, 132.9, 129.5, 129.1, 128.4, 117.2, 112.8, 56.5, 38.8; IR (neat): 3061, 3027, 2896, 2817, 1599, 1575, 1505, 1448, 1427, 1406, 1371, 1347, 1292, 1251, 1213, 1114, 1094, 1034, 1013, 950, 927, 804, 749, 692.

2-Chloro-N-methyl-N-phenylbenzylamine (Table 3, entry 4)

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.16 (s, 3H), 4.68 (s, 2H), 6.76-6.82 (m, 3H), 7.26 (d, *J* = 6.5Hz, 3H),

7.28-7.31 (m, 2H), 7.45-7.49 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.1, 136.8, 134.0, 130.7, 129.5, 128.7, 118.0, 112.5, 56.8, 38.8; IR (neat): 3067, 3017, 2895, 2815, 1589, 1565, 1501, 1449, 1427, 1371, 1347, 1292, 1213, 1114, 1084, 1033, 950, 804, 748, 692.

2-bromo-N-methyl-N-phenylbenzylamine (Table 3, entry 5)

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.15 (s, 3H), 4.61 (s, 2H), 6.74-6.80 (m, 3H), 7.16-7.23 (m, 2H), 7.26-7.29 (m, 3H), 7.63 (d, *J* = 7.9Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.1, 137.3, 132.7, 129.2, 128.3, 127.8, 127.4, 122.6, 116.6, 111.9, 57.3, 38.6; IR (neat): 2920, 1599, 1502, 1440, 1025, 747, 687.

N-methyl-N-phenyl-4-methylaniline (Table 3, entry 6)

^{1}H NMR (500 MHz, $CDCl_3$): δ 2.41 (s, 3H), 3.08 (s, 3H), 4.57 (s, 2H), 6.79 (t, *J* = 7.3Hz, 1H), 6.84 (d, *J* = 8.2Hz, 2H), 7.21 (t, *J* = 8.7Hz, 4H), 7.28-7.32 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 150.2, 137.5, 136.4, 128.9, 128.3, 117.4, 112.0, 57.6, 39.1, 20.6; IR (neat): 3062, 2973, 2861, 1597, 1504, 1451, 1393, 1356, 1245, 1198, 1180, 1126, 1074, 987, 908, 862, 799, 747, 727, 693.

N-methyl-N-phenyl-2-methylaniline (Table 3, entry 7)

^{1}H NMR (500 MHz, $CDCl_3$): δ 2.38 (s, 3H), 3.09 (s, 3H), 4.53 (s, 2H), 6.77-6.80 (m, 3H), 7.20 (d, *J* = 3Hz, 2H), 7.24-7.31 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.8, 141.0, 138.2, 129.5, 129.0, 128.3, 127.5, 126.3, 117.5, 112.9, 56.8, 39.4, 19.6; IR (neat): 3065, 2976, 2871, 1599, 1504, 1441, 1391, 1356, 1198, 1074, 985, 906, 863, 795, 748, 722, 694.

N-methyl-N-phenyl-3-phenylpropylamine (Table 3, entry 8)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.99-2.05 (m, 2H), 2.75 (t, *J* = 7.8Hz, 2H), 3.01 (s, 3H), 3.44 (t, *J* = 7.5 Hz, 2H), 6.78 (t, *J* = 8.3 Hz, 3H), 7.28-7.33 (m, 5H), 7.38 (t, *J* = 7.5Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 150.3, 141.5, 129.5, 129.0, 126.6, 118.2, 113.1, 54.2, 39.6, 35.7, 29.4; IR (neat): 3065, 3022,

2931, 2871, 1599, 1504, 1453, 1394, 1357, 1271, 1198, 1125, 1071, 1030, 987, 908, 889, 862, 799, 746, 727, 694, 505.

N-methyl-N-pentylbenzenamine (Table 3, entry 9)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95-0.98 (m, 4H), 1.35-1.42 (m, 3H), 1.61-1.67 (m, 2H), 2.98 (s, 3H), 3.36 (t, *J* = 7.6Hz, 2H), 6.72-6.78 (m, 3H), 7.29-7.31 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.7, 129.6, 116.0, 112.1, 52.3, 39.1, 29.6, 26.4, 22.3, 14.4; IR (neat): 3072, 2956, 2928, 1603, 1507, 1321, 1259, 748, 692.

N-methyl-N-phenyl-Cyclohexanemethylamine (Table 3, entry 10)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.01 (t, *J* = 11Hz, 2H), 1.23-1.32 (m, 3H), 1.74-1.81 (m, 6H), 3.01 (s, 3H), 1.81 (d, *J* = 6.7Hz, 2H), 6.71-6.75 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.1, 129.5, 118.0, 114.5, 59.3, 40.1, 37.2, 34.0, 26.2, 24.6; IR (neat): 3058, 2966, 1600, 1505, 1470, 1447, 745, 691.

N-benzyl-N-ethylaniline (Table 3, entry 11)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.29 (t, *J* = 7.1Hz, 3H), 3.54-3.58 (m, 2H), 4.61 (s, 2H), 6.75-6.81 (m, 3H), 7.26-7.29 (m, 2H), 7.31-7.34 (m, 3H), 7.38-7.42 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.42, 139.22, 129.19, 128.50, 126.69, 126.49, 115.96, 112.07, 53.85, 45.08, 12.09; IR (neat): 3061, 3027, 2971, 2927, 2871, 1599, 1504, 1451, 1393, 1356, 1272, 1245, 1198, 1180, 1126, 1074, 1037, 987, 908, 879, 862, 799, 747, 727, 693.

Tribenzylamine (Table 3, entry 12)

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.57 (s, 6H)， 7.26 (t, *J* = 41.2 Hz,15H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.8, 128.9, 128.6,127.1, 58.2; IR (neat): 3102, 3082, 3061, 3025, 2932, 2880, 2836, 2798, 2749, 2714, 1601, 1492, 1449, 1365, 1307, 1245, 1205, 1119, 1070, 1027, 988, 971, 903, 879, 824, 740,

694, 618, 591, 491, 463, 418.

N,N-Dibutylbenzylamine (Table 3, entry 13)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.93 (t, *J* = 7.35Hz, 6H), 1.31-1.37 (m, 4H), 1.48-1.54 (m, 4H), 2.47 (t, *J* = 7.1 Hz, 4H), 3.62 (s, 2H), 7.27 (t, *J* = 7.1Hz, 1H), 7.33-7.40 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ140.5, 128.8, 128.2, 126.6, 58.7, 53.8, 29.4, 20.7, 14.1; IR (neat): 1492, 1451, 1365, 741, 696.

N-Benzylmorpholine (Table 3, entry 14)

^{1}H NMR (500 MHz, $CDCl_3$): δ 2.49 (t, *J* = 4.5Hz, 4H), 3.55 (s, 2H), 3.75 (t, *J* = 4.7Hz, 4H), 7.28-7.31 (m, 1H), 7.35-7.39 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.5, 129.4, 128.1, 126.9, 66.3, 64.9, 60.4; IR (neat): 3034, 2986, 2631, 1717, 1377, 1245, 1045, 939, 750, 716, 699, 609.

N-benzylpyrrolidine (Table 3, entry 15)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.86 (s, 4H), 2.62 (s, 4H), 3.72 (s, 2H), 7.30 (d, *J* = 5.1Hz, 1H), 7.35-7.41 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.5, 128.9, 128.2, 126.8, 60.7, 54.3, 23.6; IR (neat): 3054, 2965, 2784, 1454, 1348, 1125.

Tributylamine (Table 3, entry 16)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95 (t, *J* = 7.3Hz, 9H), 1.30-1.37 (m, 6H), 1.42-1.48 (m, 6H), 2.43 (t, *J* = 7.7Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 54.0, 29.5, 20.8, 13.3; IR (neat):2967, 2873, 2798, 1468, 1377, 1307, 1183, 1086, 996, 901, 785, 733.

N-methyl-N-isopropylaniline (Table 3, entry 17)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.23 (d, *J* = 6.6Hz, 6H),2.80 (s, 3H), 4.11-4.19 (m, 1H), 6.76 (t, *J* = 7.3Hz, 1H), 6.87 (d, *J* = 8.2Hz, 2H), 7.27-7.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4, 129.3, 116.6, 113.6, 49.1, 30.0, 19.5; IR (neat): 2917, 2849, 1713, 1673, 1598, 1503, 1450, 1287, 1232, 1111, 749, 698.

***N*-cyclopentyl-*N*-methyl-Benzenamine (Table 3, entry 18)**

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.63-1.71 (m, 4H), 1.75-1.79 (m, 2H), 1.89-1.96 (m, 2H), 2.87 (s, 3H), 4.15-4.22 (m, 1H), 6.80 (t, *J* = 7.3Hz, 1H), 6.93 (d, J = 8.2Hz, 2H), 7.27-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.2, 130.1, 117.0, 113.6, 61.2, 38.7, 29.8, 20.4; IR (neat): 2946, 2861, 1597, 1504.

N-methyl-N-cyclohexylaniline (Table 3, entry 19)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.19-1.24 (m, 1H), 1.40-1.57 (m, 4H), 1.75-1.93 (m, 5H), 2.86 (s, 3H), 3.61-3.67 (m, 1H), 6.75-6.87 (m, 3H) 7.28-7.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3, 129.2, 116.3, 113.3, 58.2, 30.2, 30.1, 26.3, 26.1; IR (neat): 2946, 2861, 1597, 1504.

4-*tert*-butyl-*N*-methyl-*N*-phenylCyclohexylamine (Table 3, entry 20)

^{1}H NMR (500 MHz, $CDCl_3$): δ 0.93 (s, 13H), 1.51-1.59 (m, 5H), 2.84 (s, 1H), 2.89 (s, 3H), 7.01 (d, *J* = 8Hz, 2H), 7.28-7.32 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.4, 130.1, 117.4, 115.8, 58.2, 48.1, 30.6, 27.2, 21.0; IR (neat): 2956, 2867, 1597, 1504,1409, 889, 783, 633, 507 .

***N*-methyl-*N*-phenyl-2-Pyridinemethanamine (Table 3, entry 21)**

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.14 (s, 3H), 4.82 (s, 2H), 6.58-6.89 (m, 3H), 7.09-7.29 (m, 4H), 7.63-8.02 (m, 1H), 8.67 (t, *J* = 29.7Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.0, 149.3, 148.5, 137.1, 130.2, 123.8, 122.3, 117.2, 112.4, 59.2, 39.5; IR (neat): 3056, 3018, 2952, 2833, 2768, 1566, 1542, 1028, 737, 700.

N-substituted isoindolinones:

N-Phenylisoindolin-1-one (Scheme 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.91 (s, 2H), 7.23 (t, *J* = 7.4Hz, 1H), 7.48 (t, *J* = 8.1Hz, 2H), 7.56 (d, *J* =

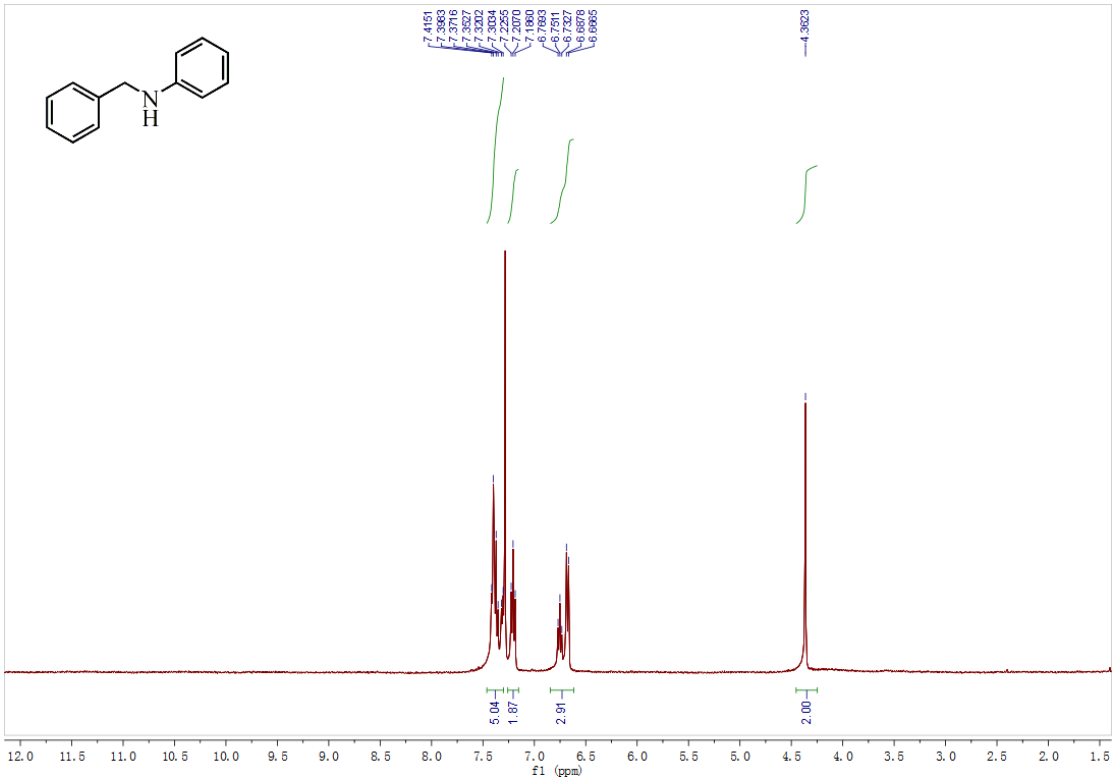
7.7Hz, 2H), 7.63 (d, *J* = 7.2Hz, 1H), 7.92 (d, *J*= 8.2Hz, 2H), 7.98 (d, *J* = 7.5Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.0, 140.5, 139.9, 133.6, 132.4, 129.5, 128.7, 124.8, 124.5, 123.0, 119.8, 51.1; IR (neat): 3026, 2922, 2851, 1686, 1595, 1501, 1464, 1440, 732.

N-(4-methylphenyl)isoindolin-1-one (Scheme 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ 2.40 (s, 3H), 4.97 (s, 2H), 7.23 (t, *J* = 7.5Hz, 3H), 7.37 (t, *J* = 5.5Hz, 3H), 7.88 (d, *J* = 8.9Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ167.7, 140.5, 137.3, 134.5, 133.7, 132.3, 130.0, 128.7, 124.4, 122.9, 119.9, 51.2, 21.2; IR (neat): 2921, 1683, 1513, 1447, 1390, 1305, 1159.

N-(4-chlorophenyl)isoindolin-1-one (Scheme 2)

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.88 (s, 2H), 7.43 (t, *J* = 9.0Hz, 2H), 7.53-7.57 (m, 2H), 7.65 (t, *J* = 7.6Hz, 1H), 7.88 (d, *J* = 8.9Hz, 2H), 7.97 (d, *J* = 7.7Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ167.7, 139.9, 138.2, 133.1, 132.5, 129.6, 129.4, 128.6, 124.4, 122.8, 120.5, 50.7; IR (neat): 3056, 2952, 2871, 1685, 1599, 1503, 1466, 1441, 733.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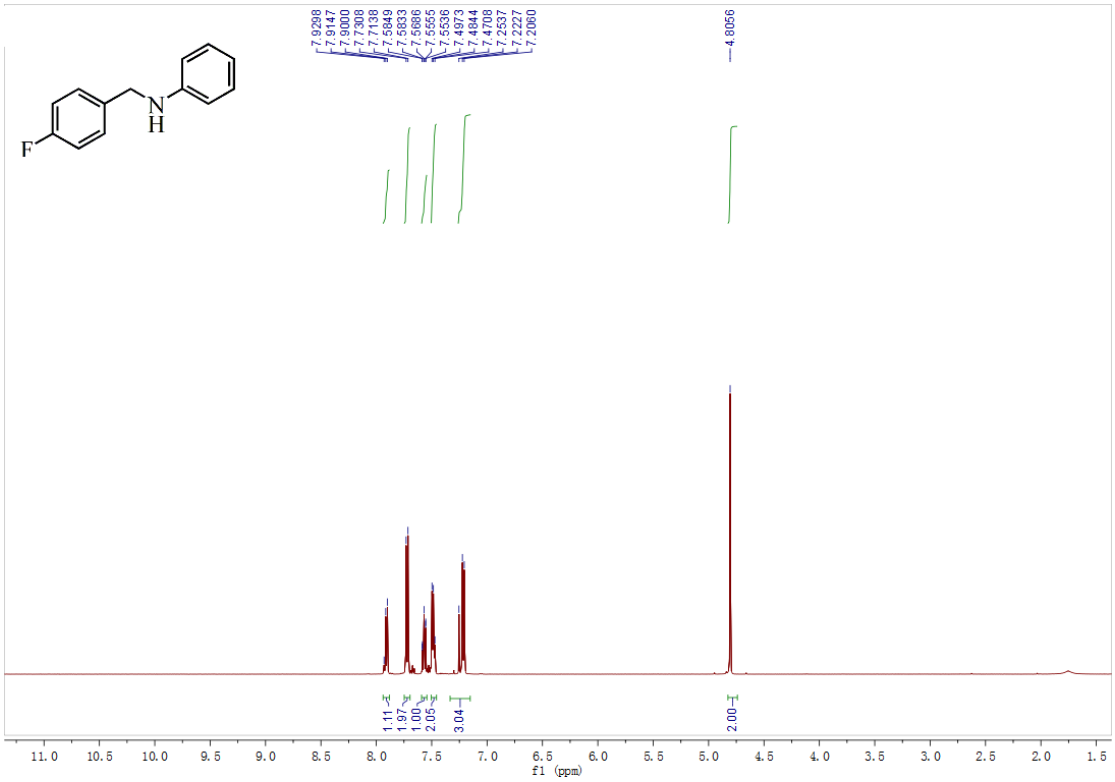
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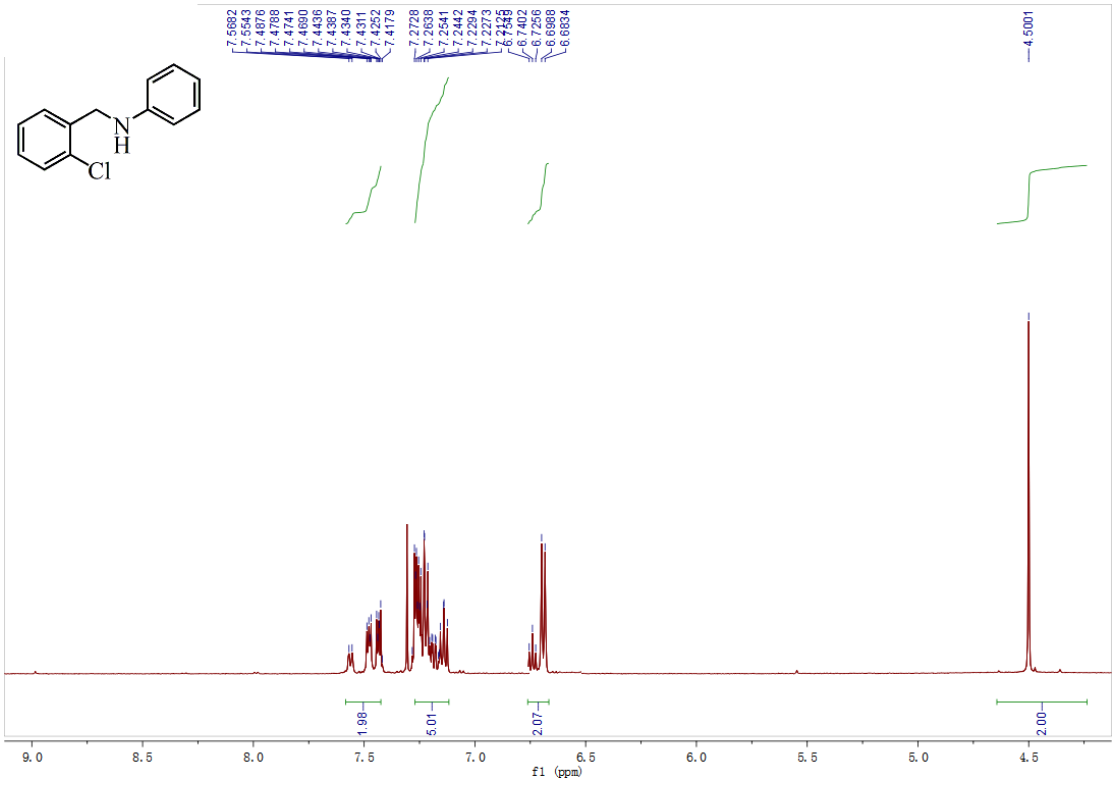
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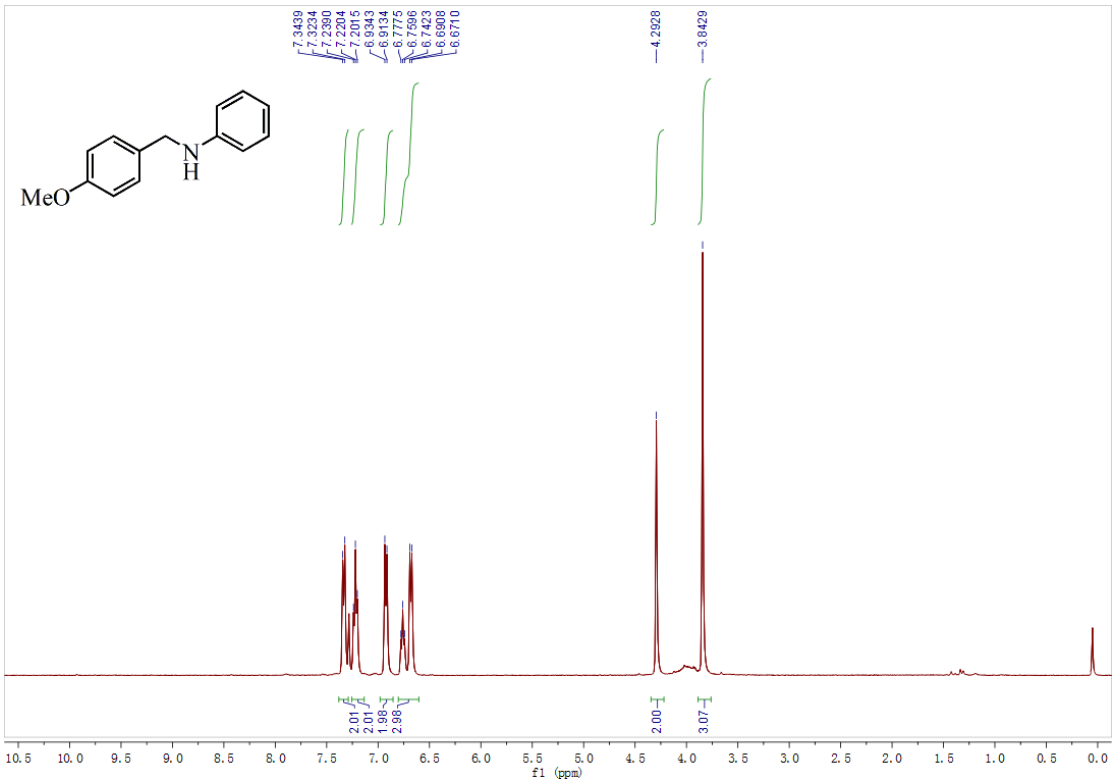
f1 (ppm)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2.98
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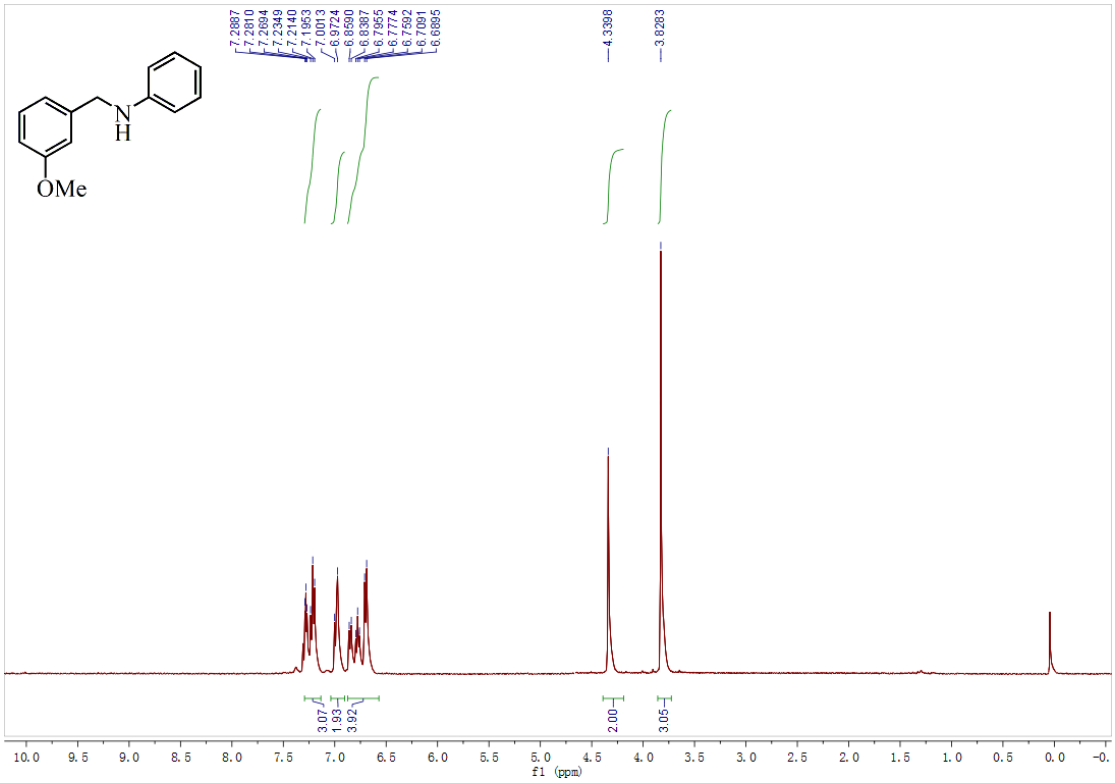
3.07
f1 (ppm)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OMe
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3.92
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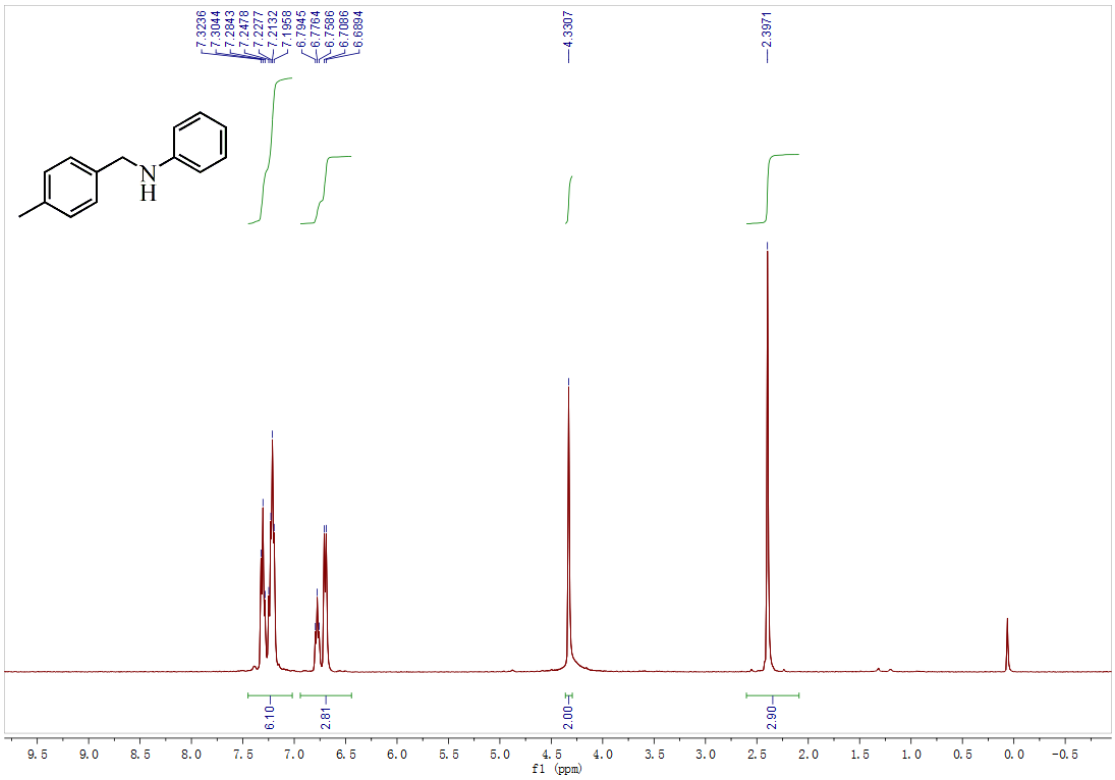
f1 (ppm)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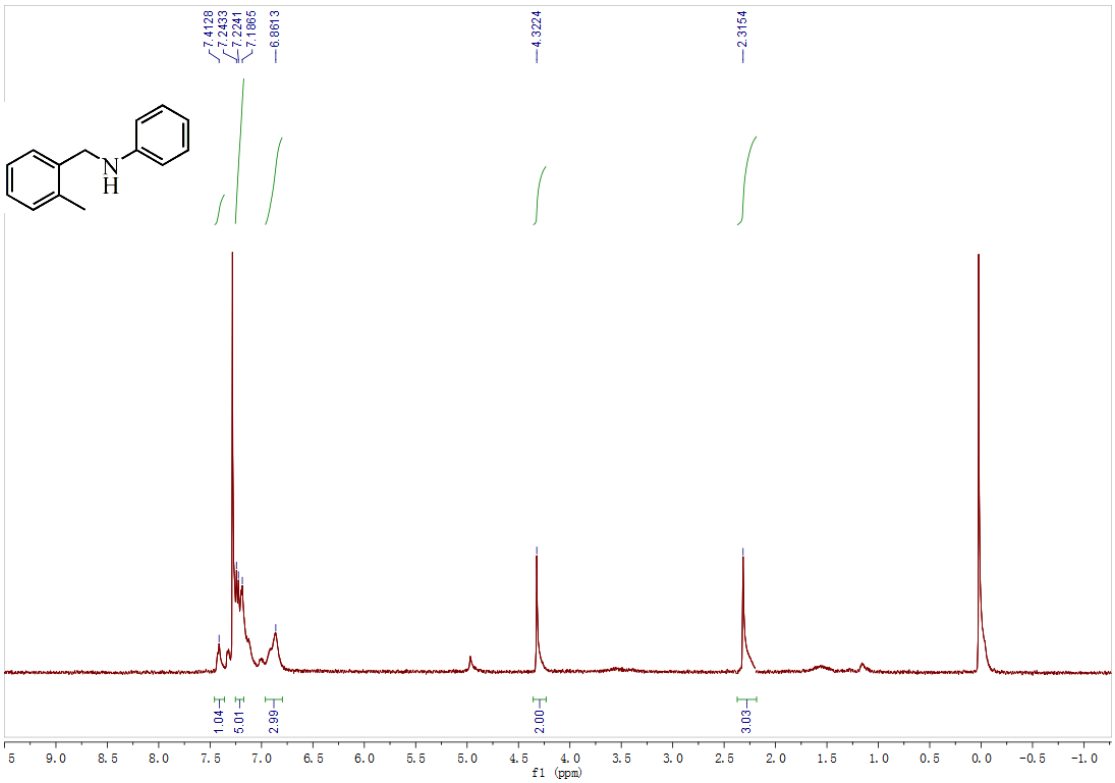
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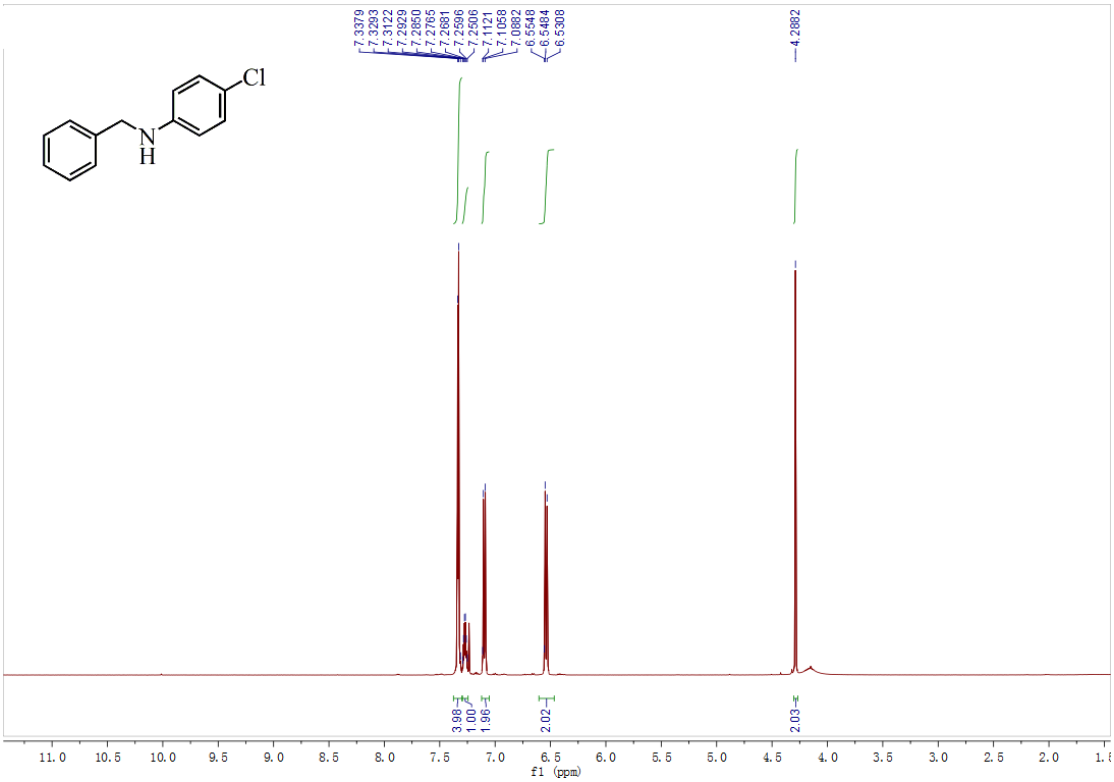
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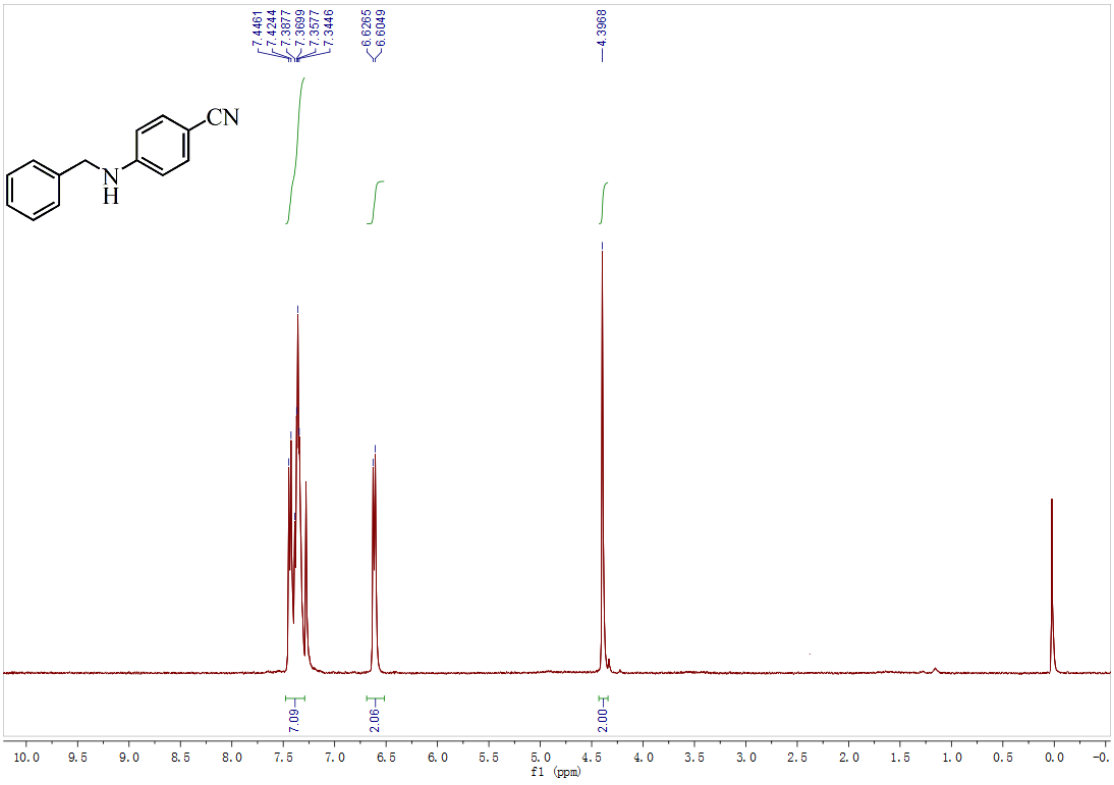
2.03
f1 (ppm)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H
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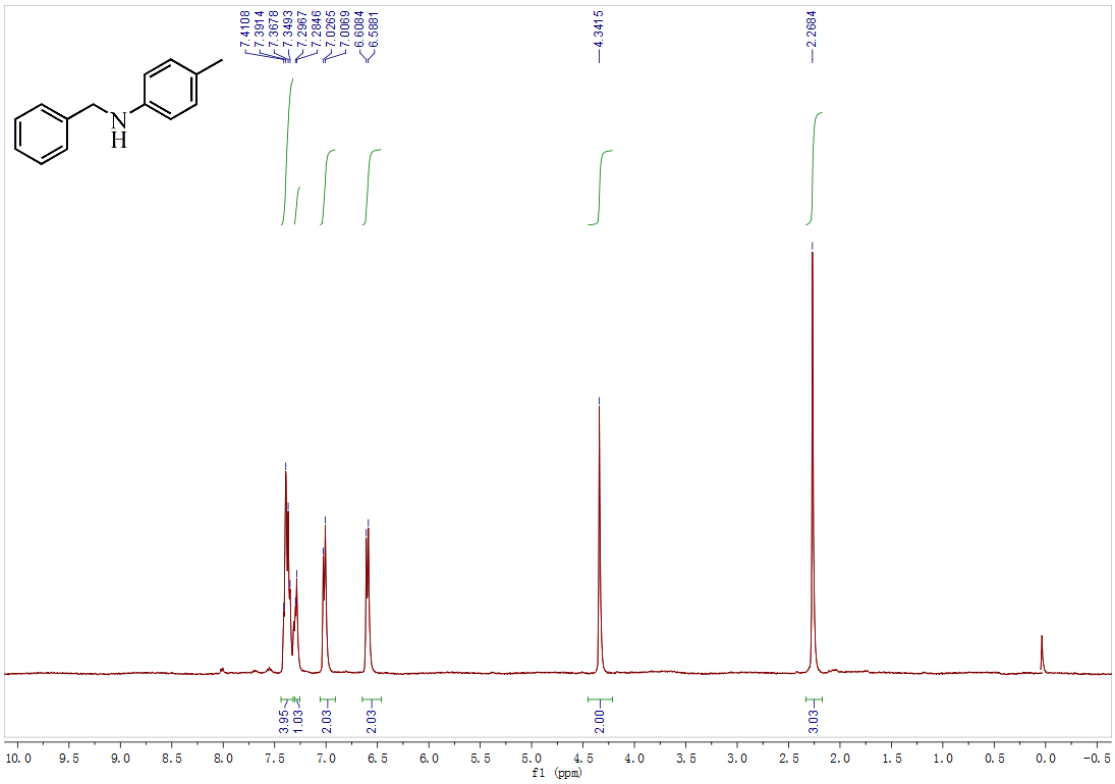
2.00
f1 (ppm)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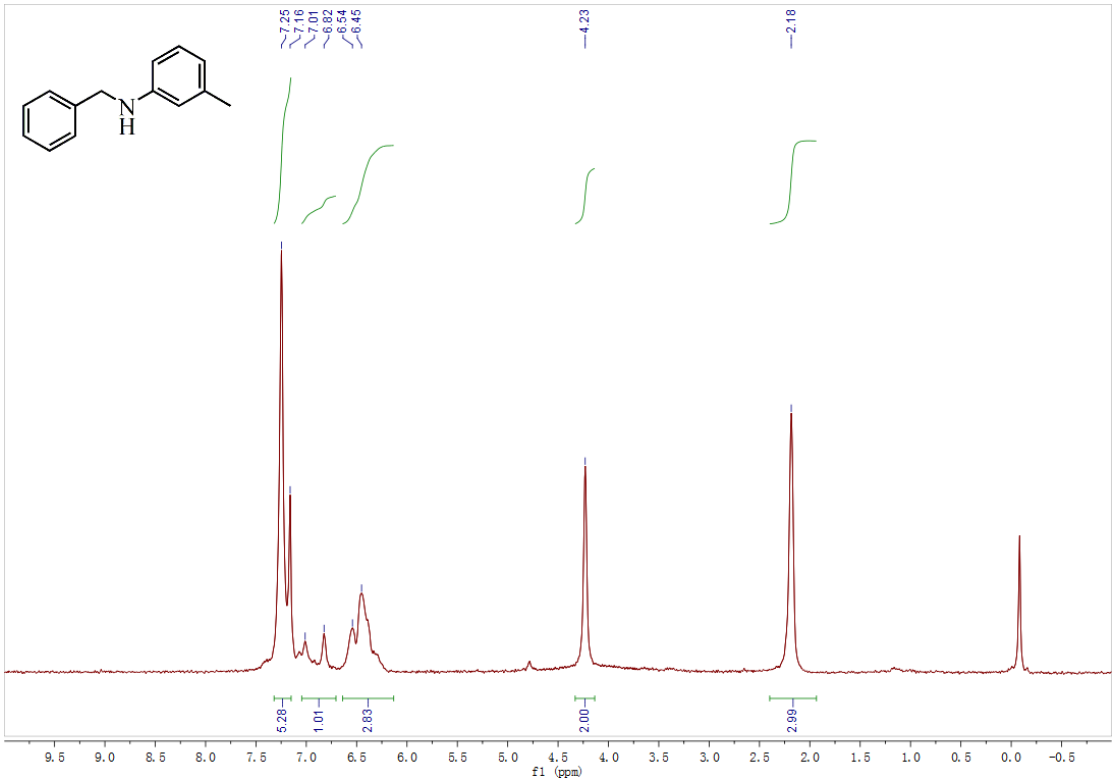
3.03
f1 (ppm)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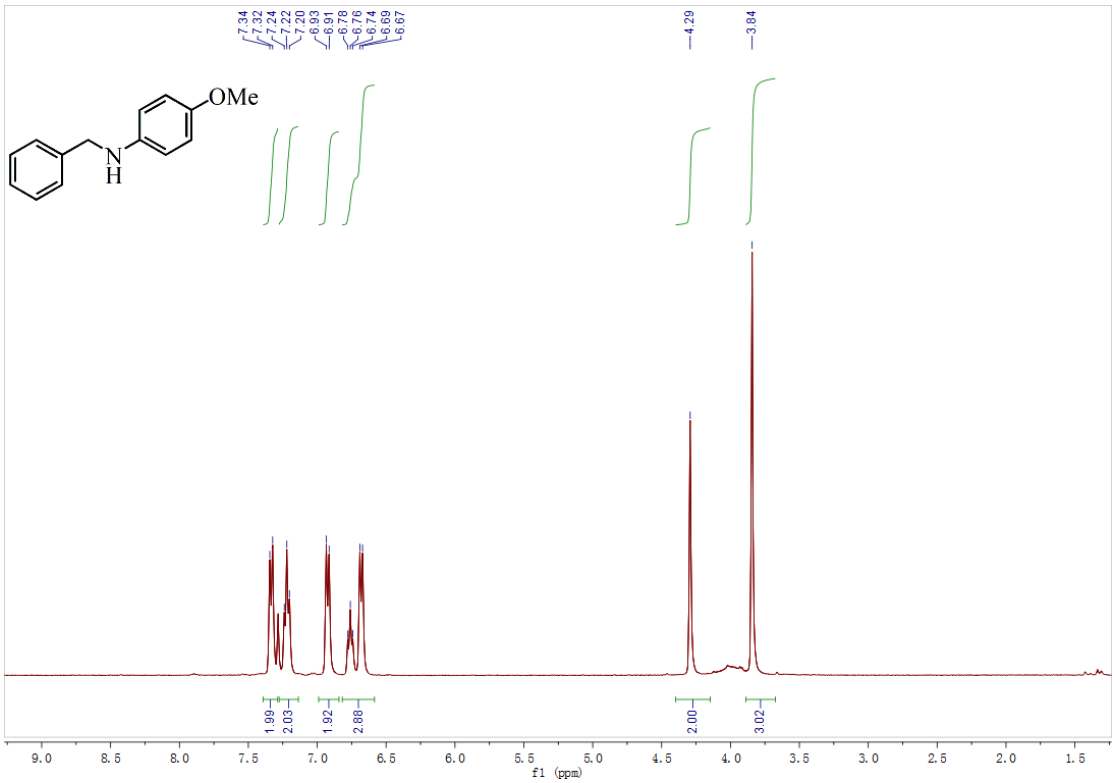
f1 (ppm)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4.29
3.84
OMe
N
H
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2.03
1.92
2.88
2.00
3.02
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8.5
8.0
7.5
7.0
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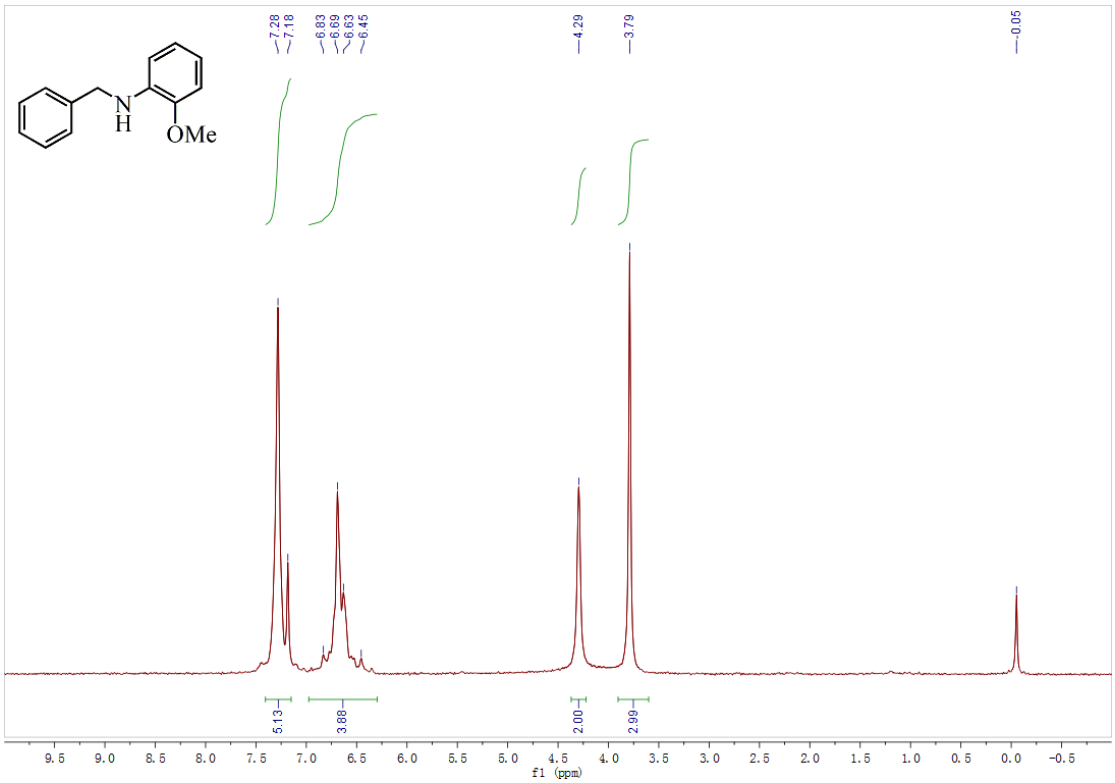
1.5
f1 (ppm)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H
OMe
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2.00
2.99
9.5
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8.5
8.0
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7.0
6.5
6.0
5.5
5.0
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0.5
0.0
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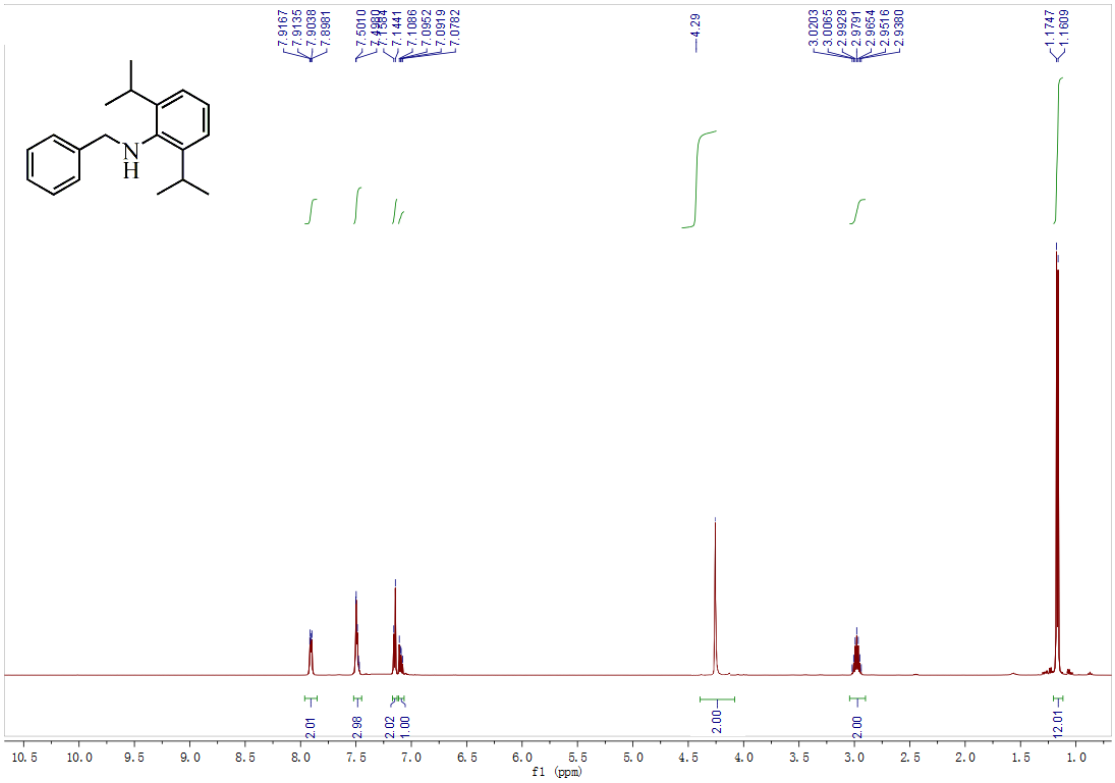
f1 (ppm)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4.29
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3.0065
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2.9516
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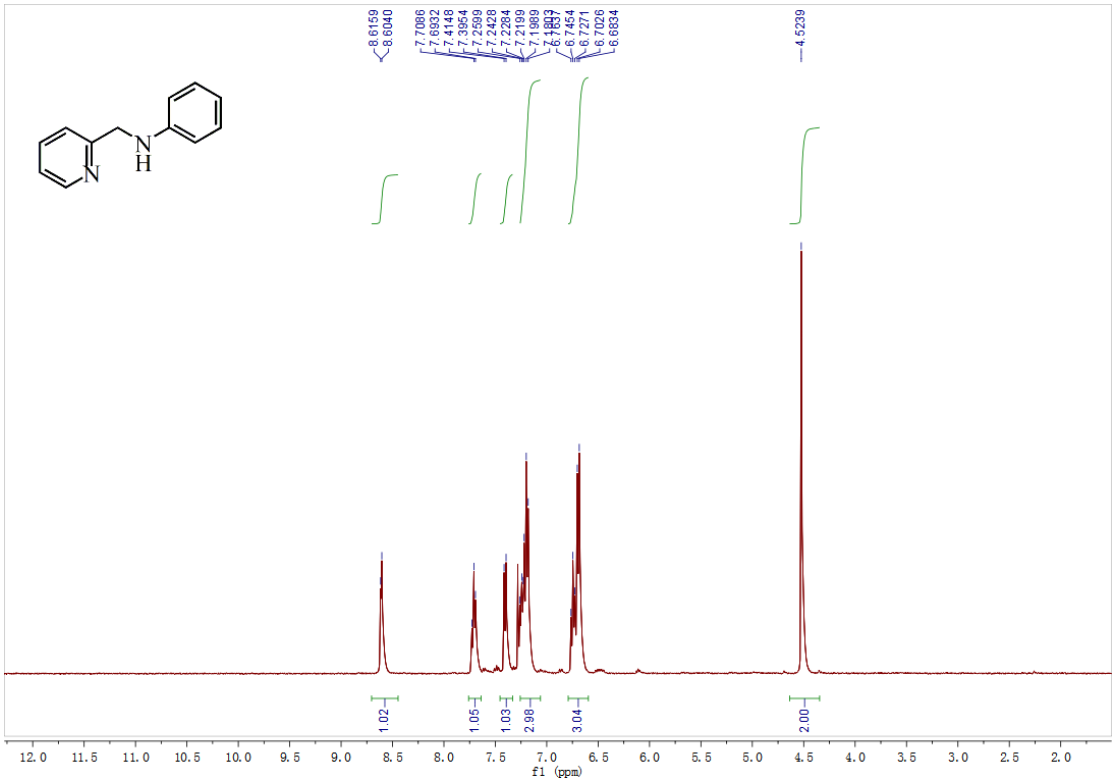
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2.98
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2.00
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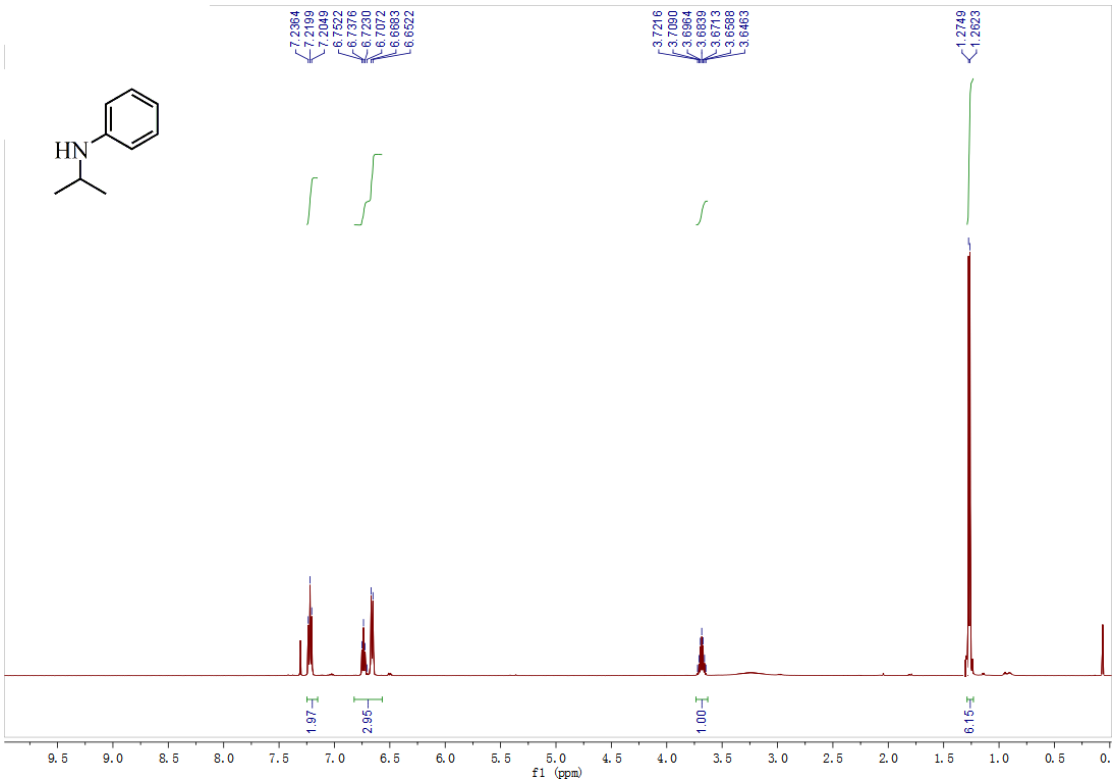
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2.95
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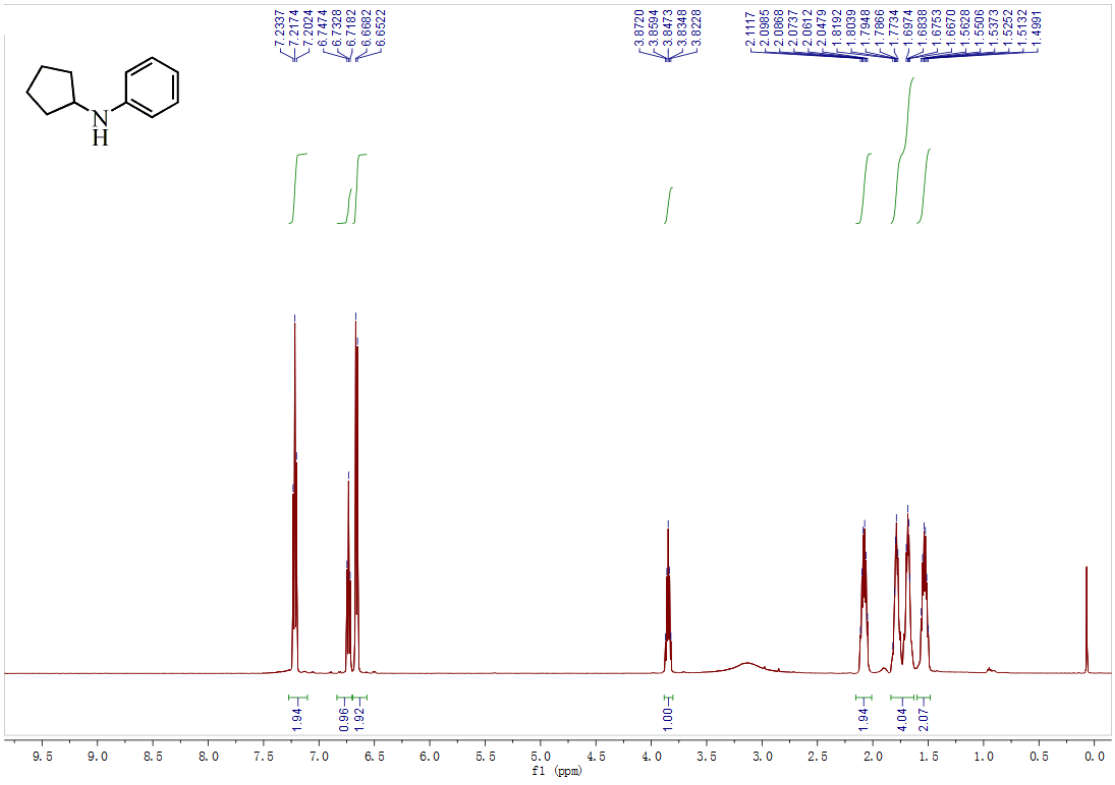
6.15
f1 (ppm)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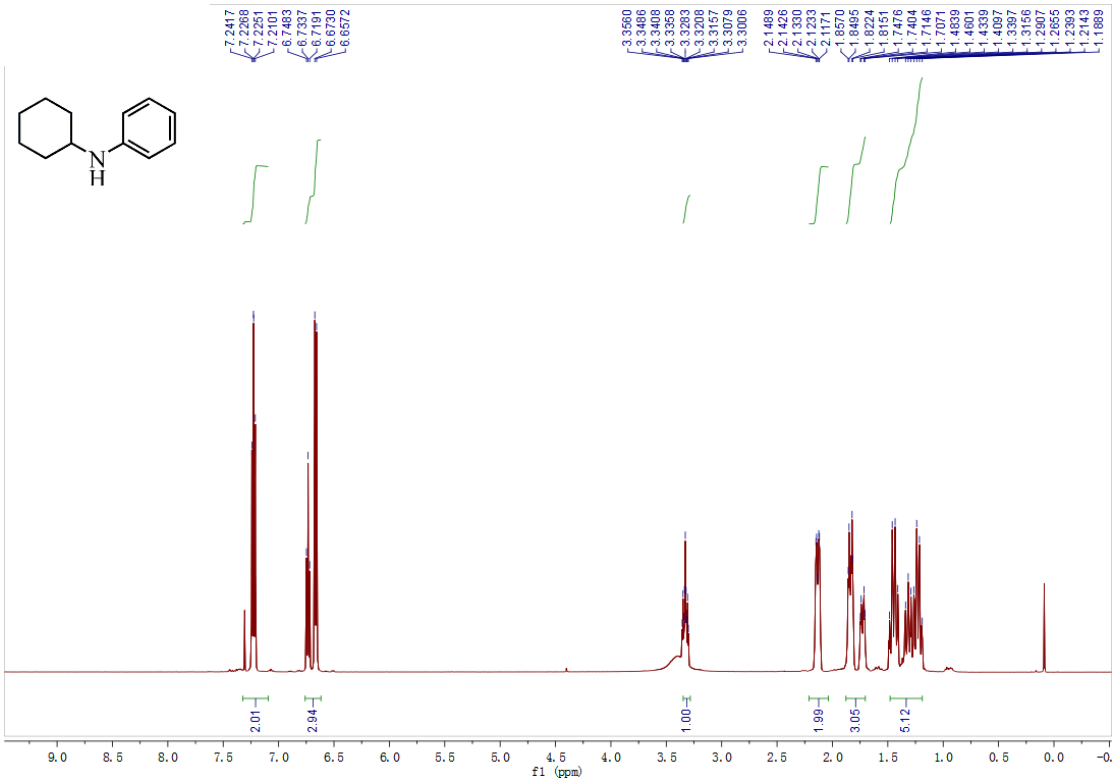
f1 (ppm)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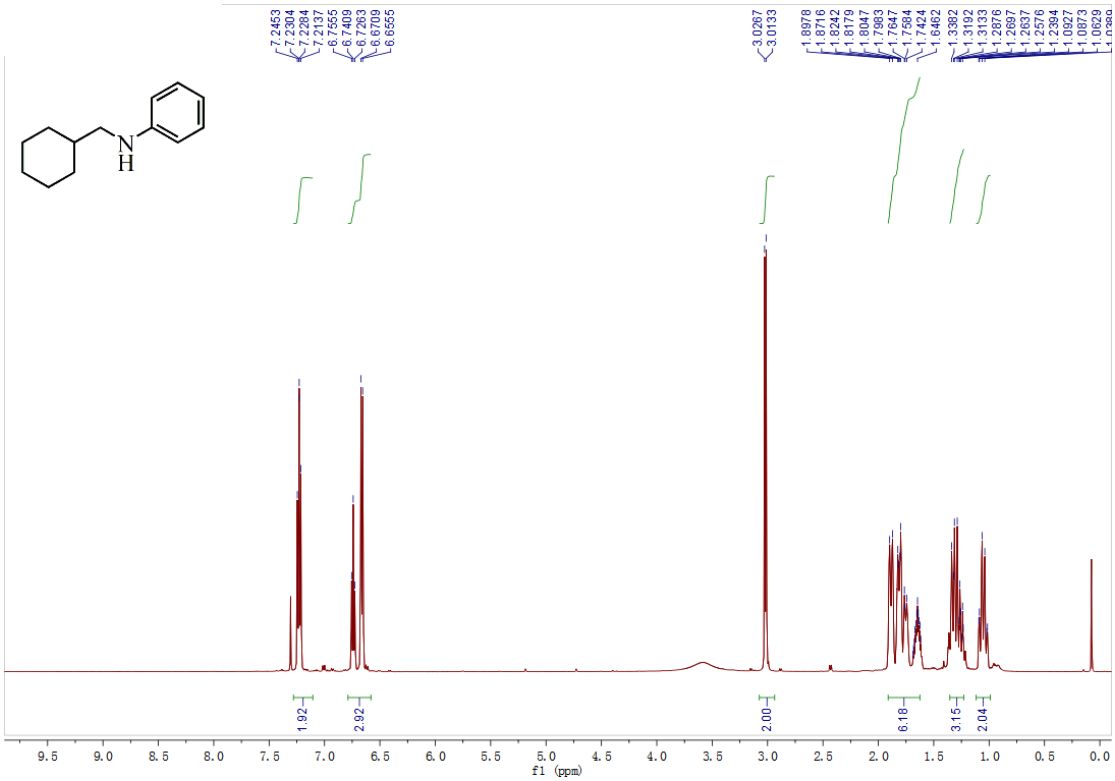
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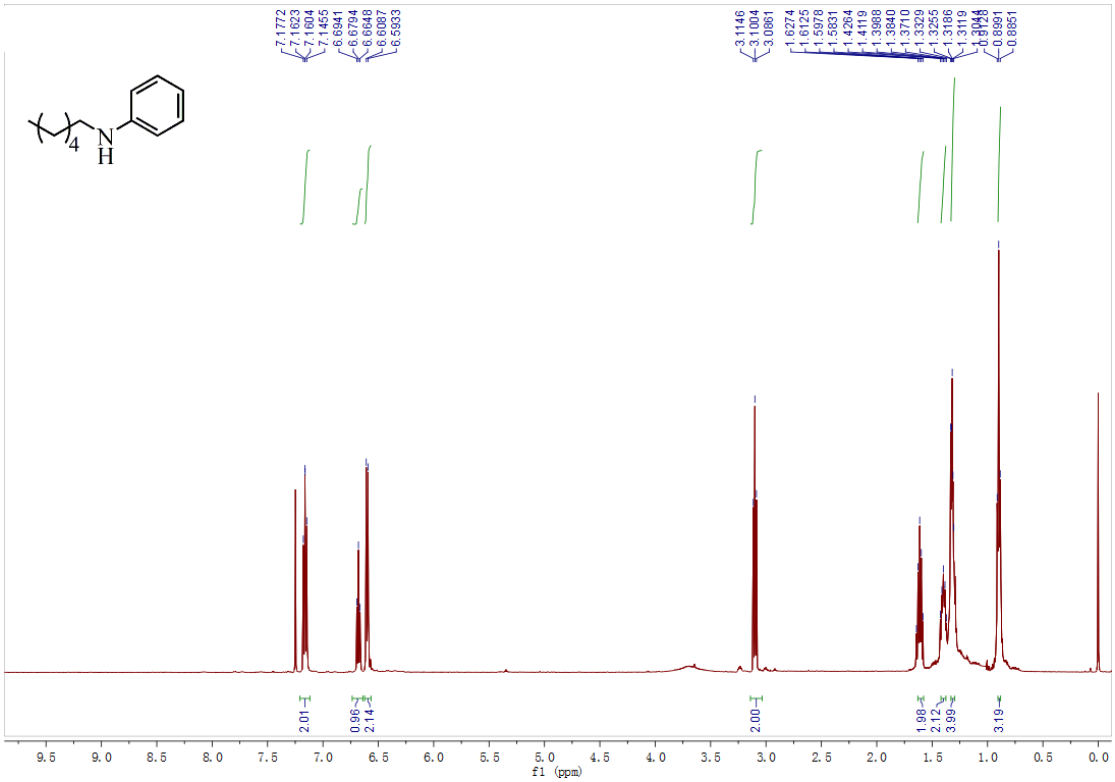
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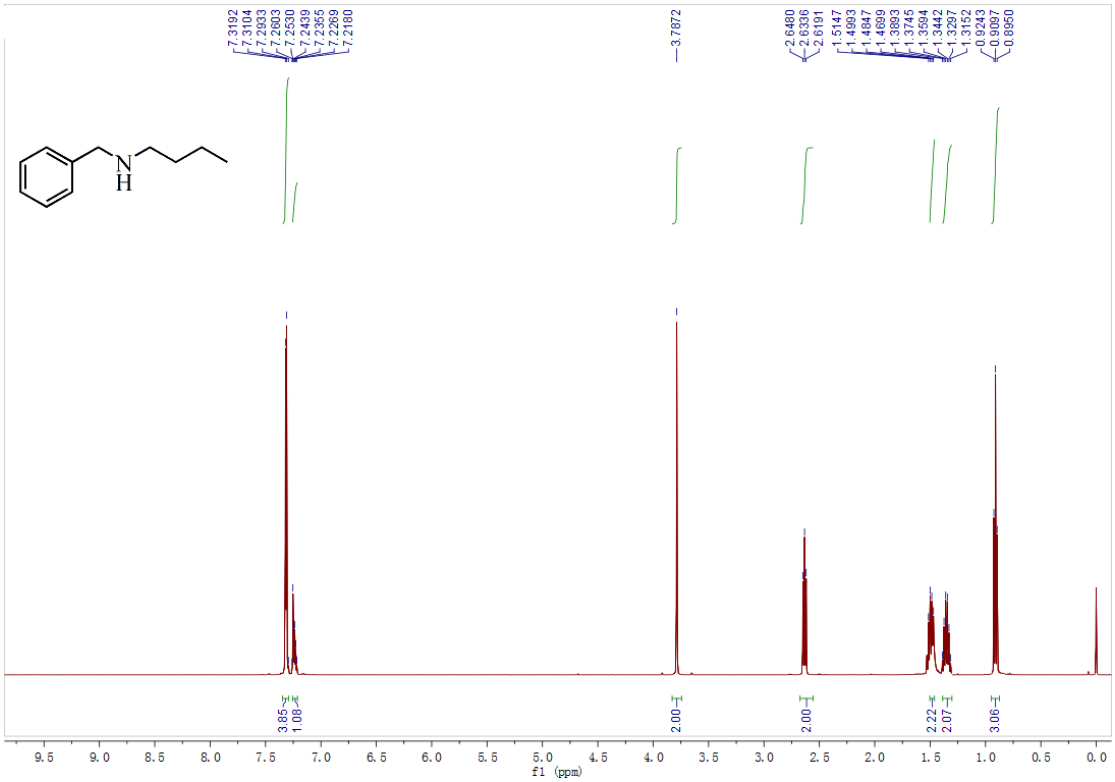
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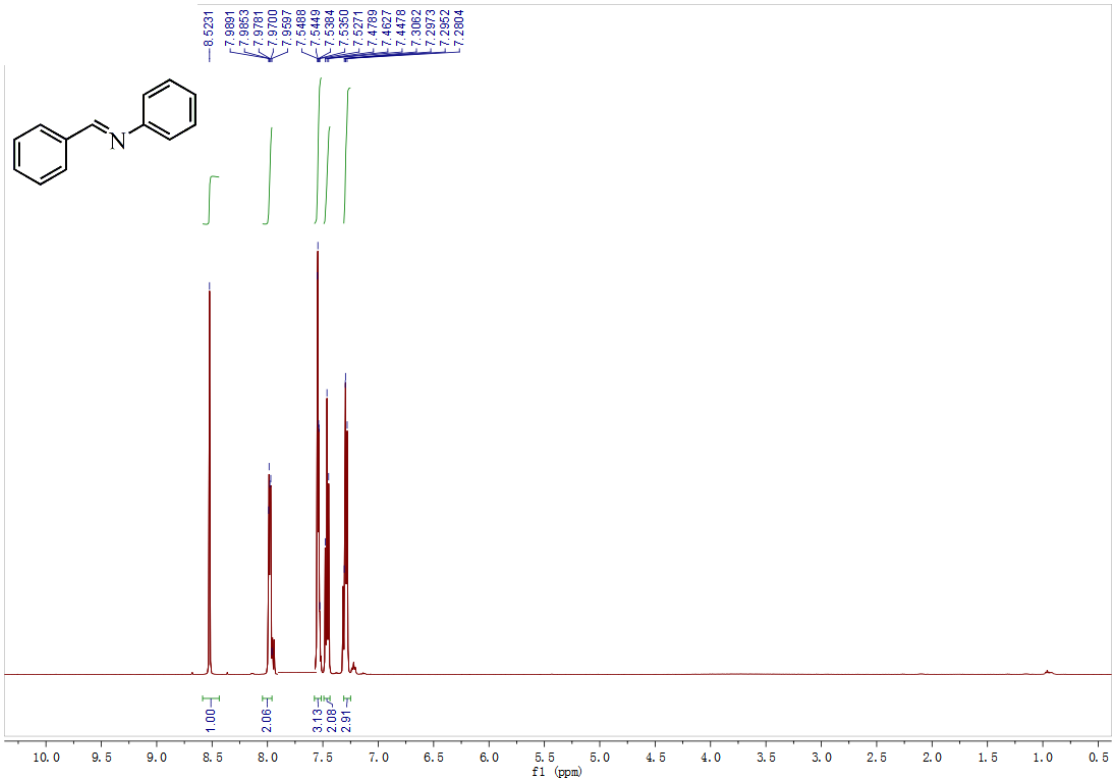
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2.91
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
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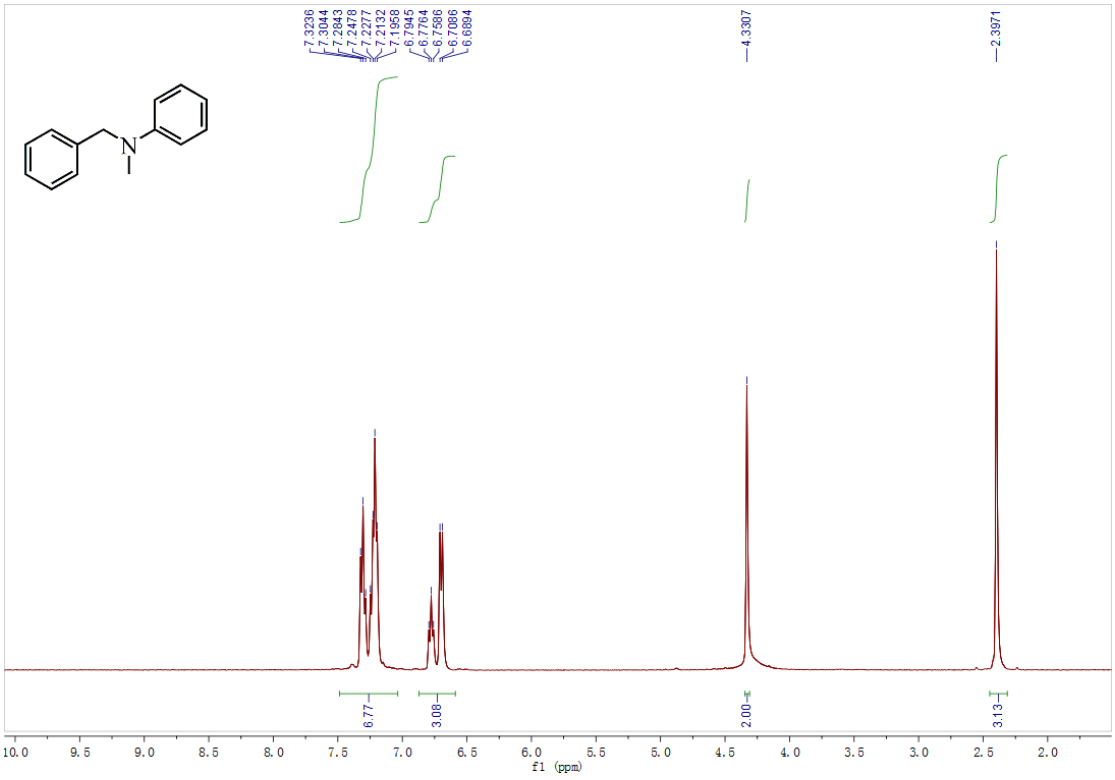
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2.00
3.13
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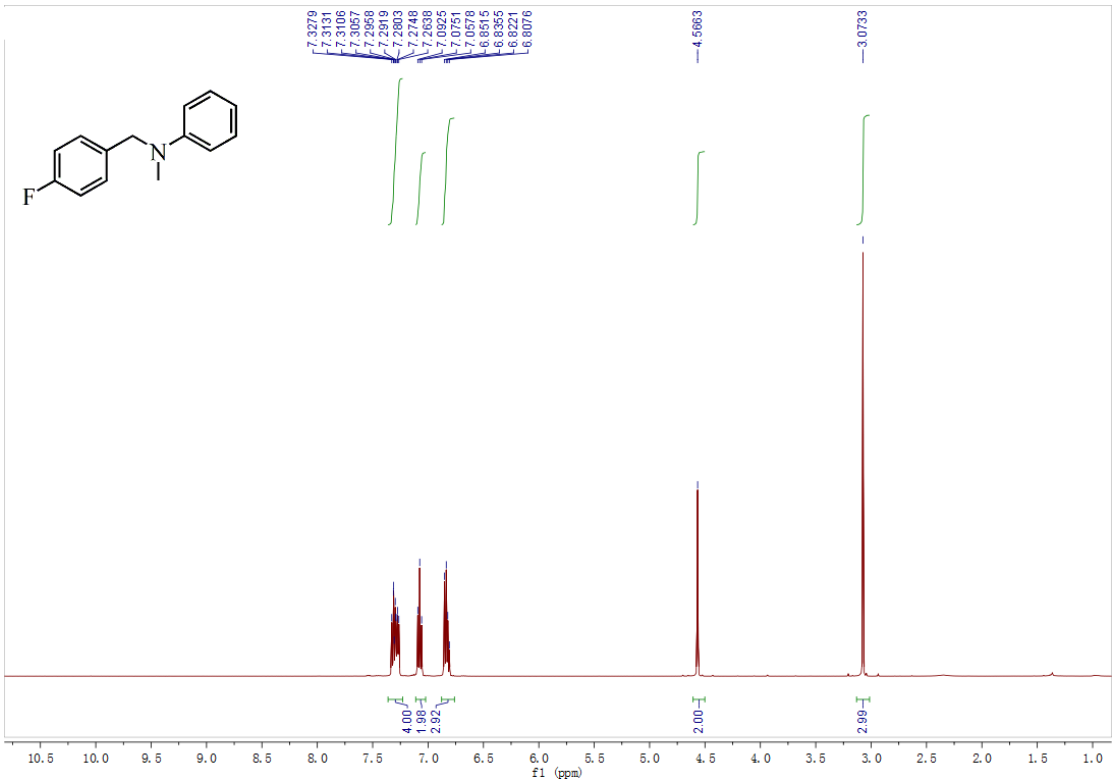
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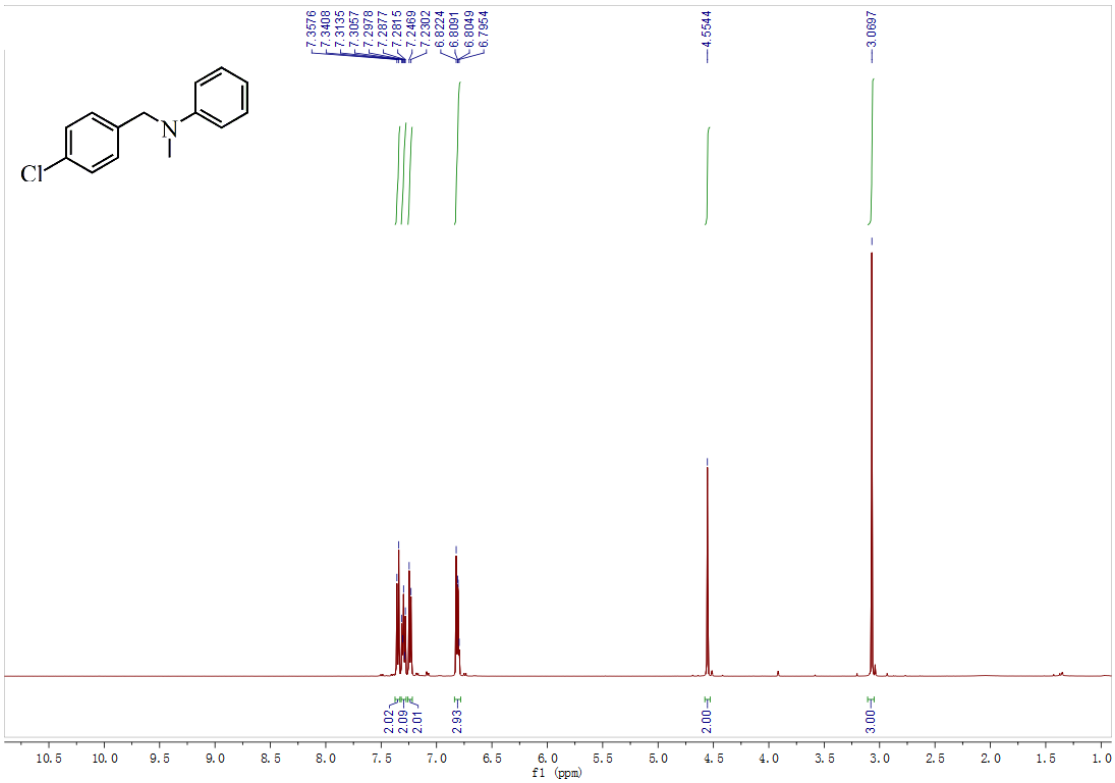
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2.93
2.00
3.00
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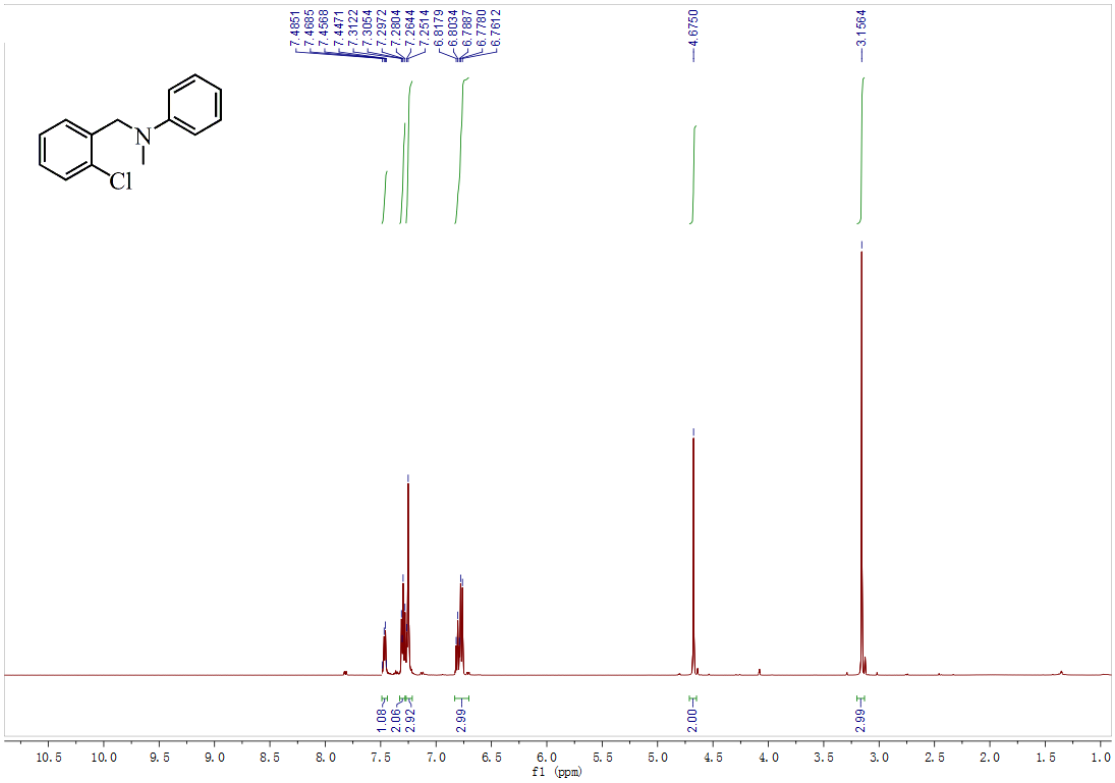
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2.00
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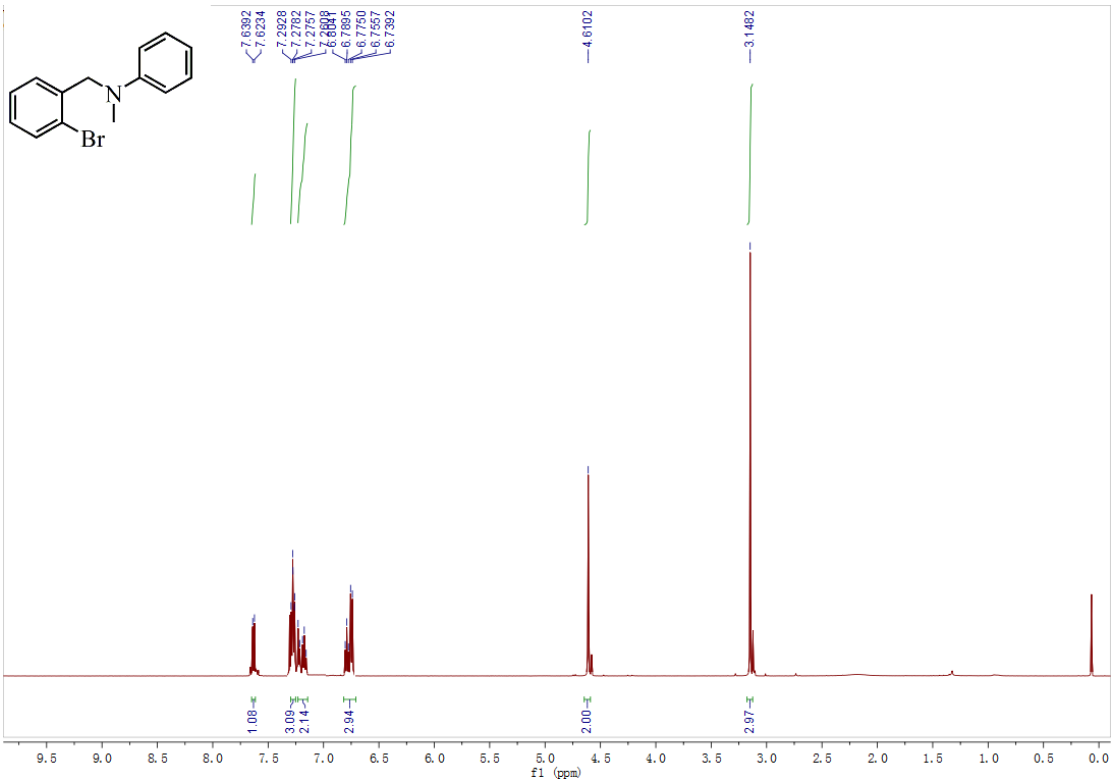
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)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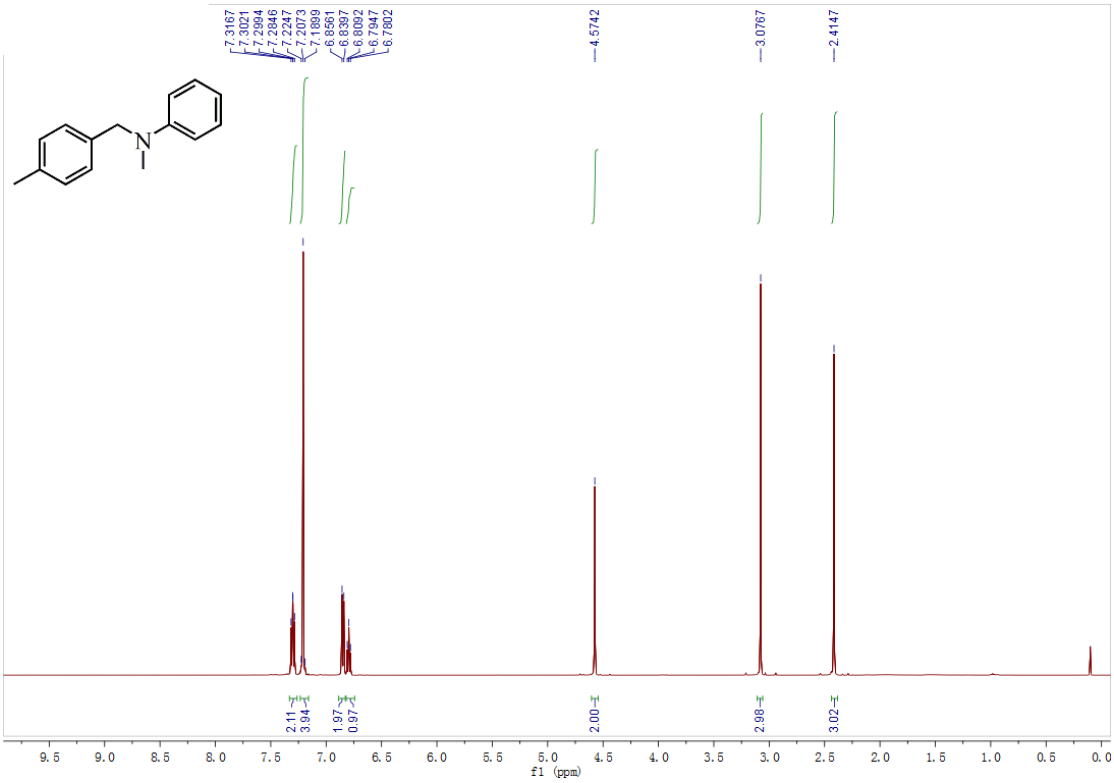
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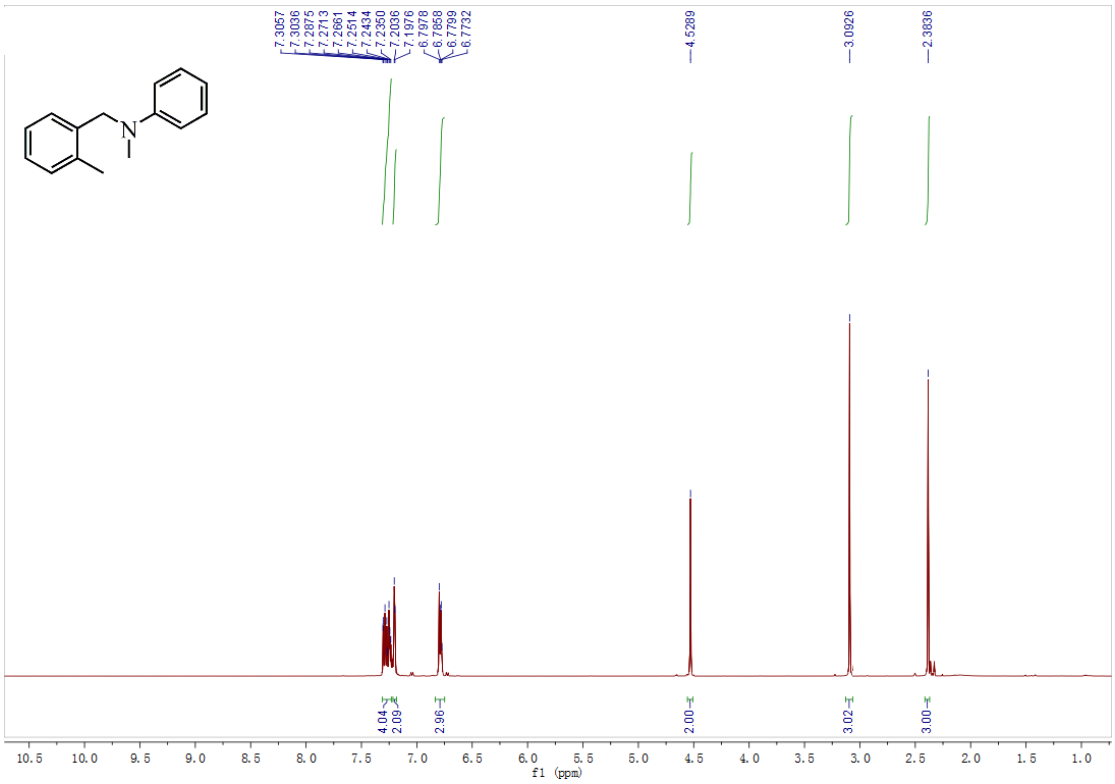
f1 (ppm)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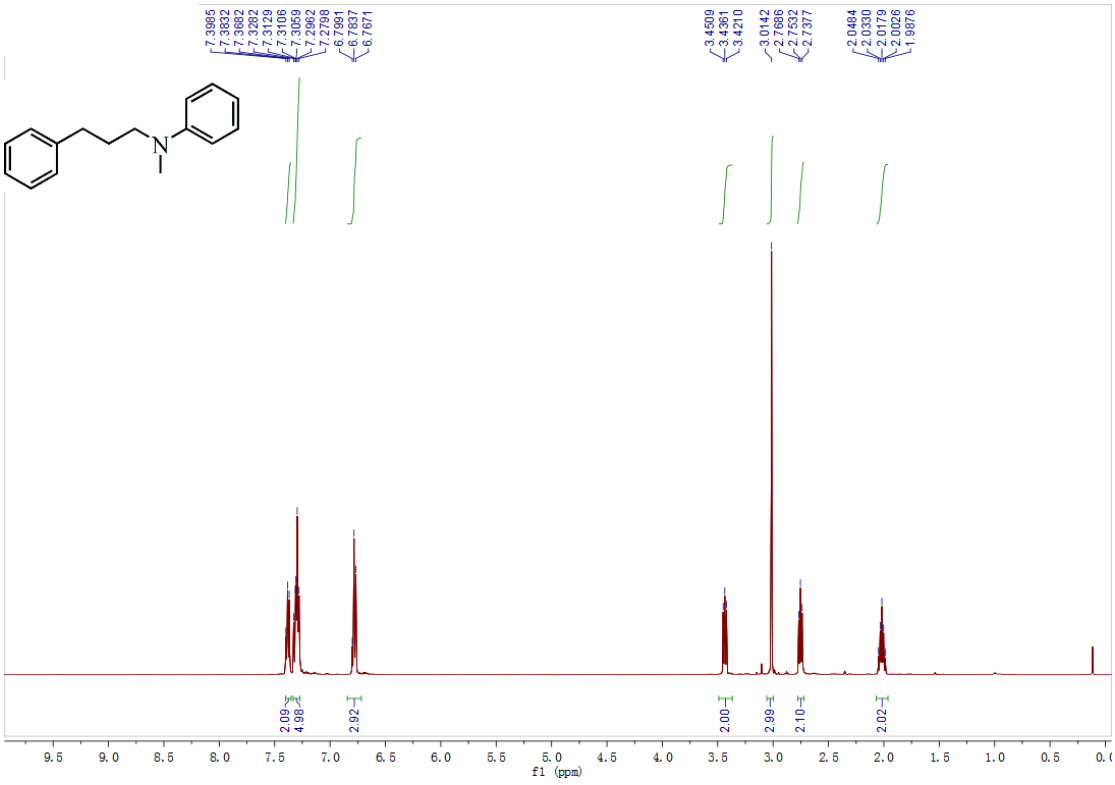
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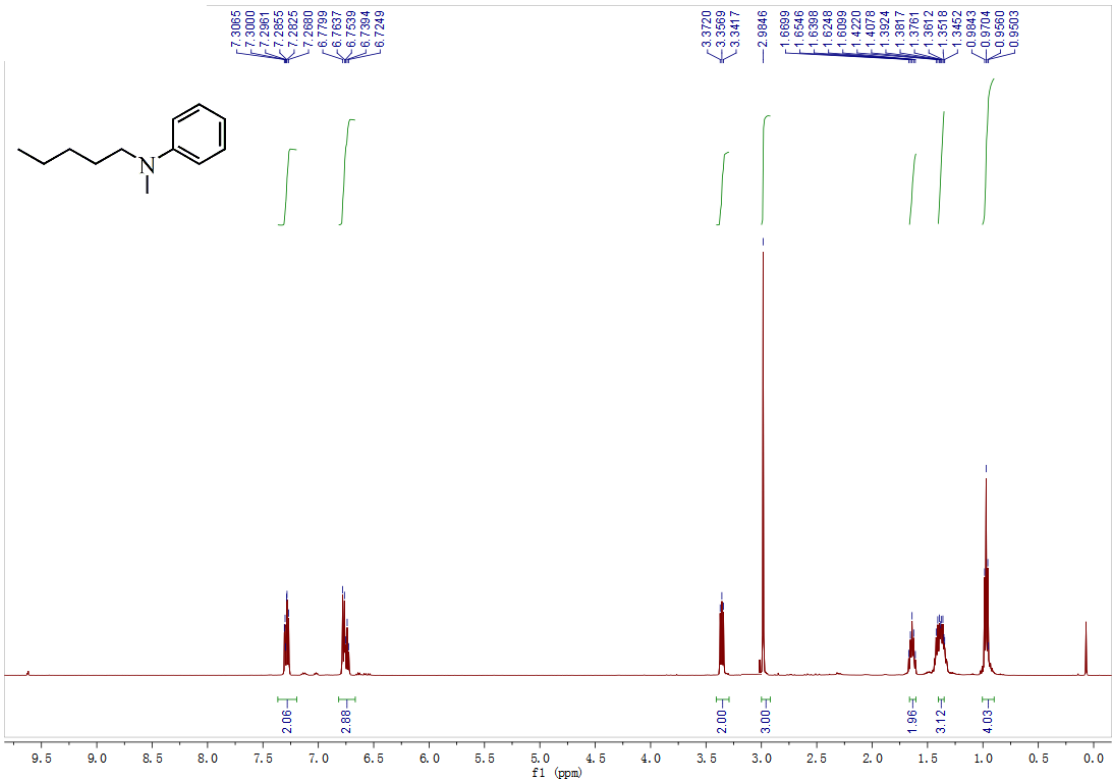
f1 (ppm)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3.3569
3.3417
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1.6699
1.6546
1.6398
1.6248
1.6099
1.4220
1.4078
1.3924
1.3817
1.3761
1.3612
1.3518
1.3452
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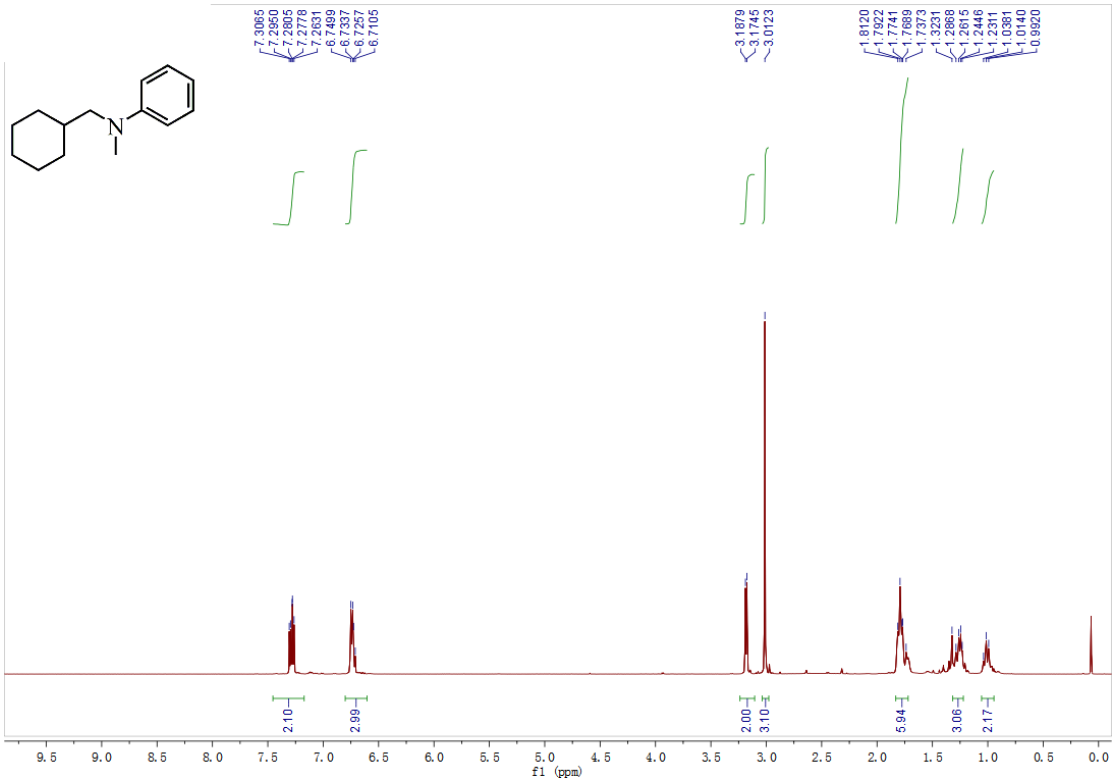
2.88
2.00
3.00
1.96
3.12
4.03
f1 (ppm)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3.0123
1.8120
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1.7741
1.7689
1.7373
1.3231
1.2868
1.2615
1.2446
1.2311
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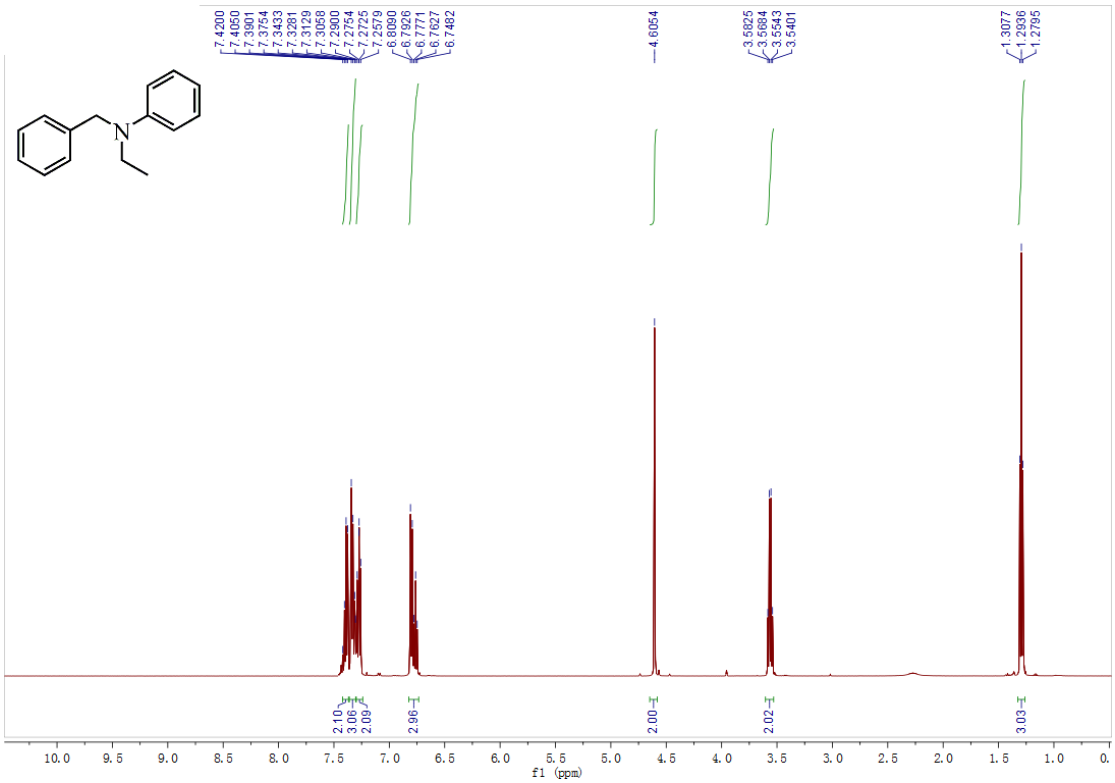
2.10
2.99
2.00
3.10
5.94
3.06
2.17
f1 (ppm)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6.7482
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3.5401
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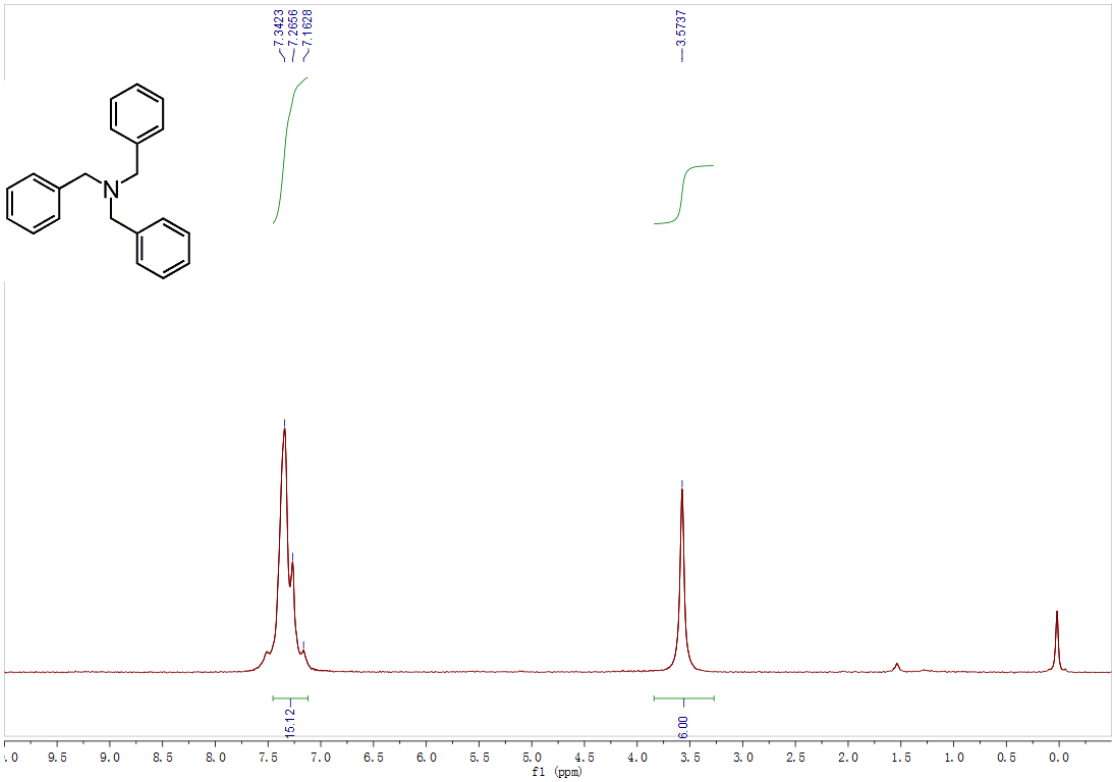
3.06
2.09
2.96
2.00
2.02
3.03
f1 (ppm)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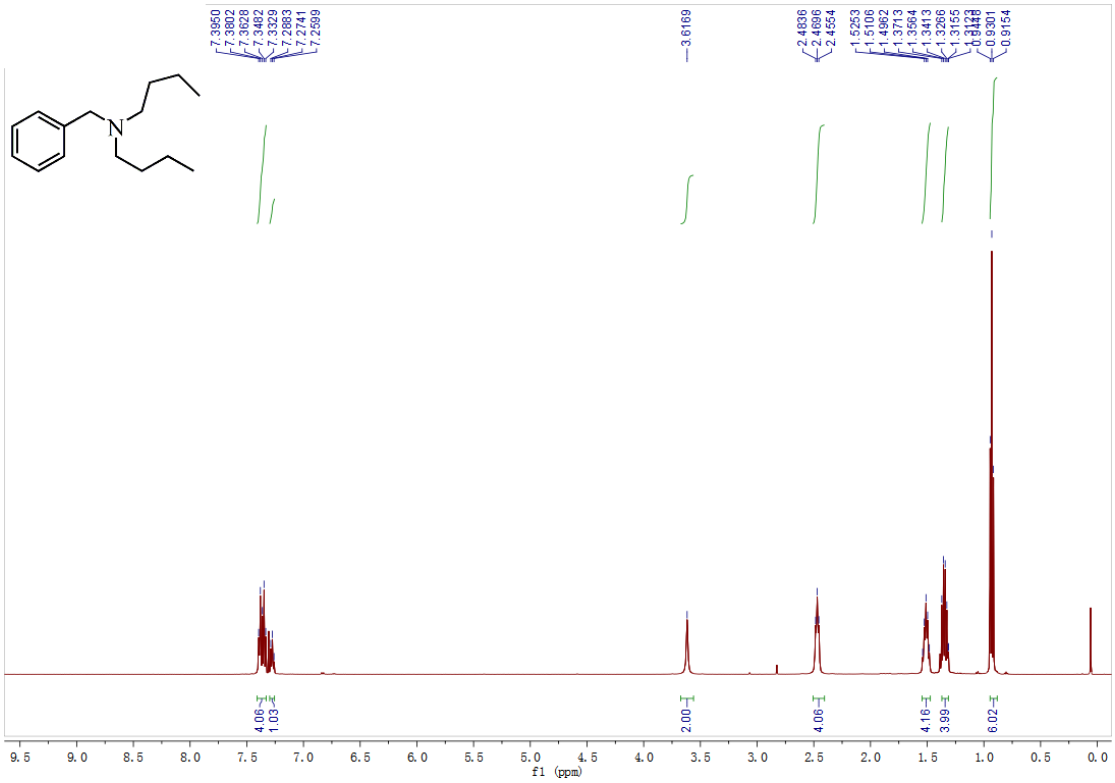
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2.4554
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1.5106
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1.4813
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1.3413
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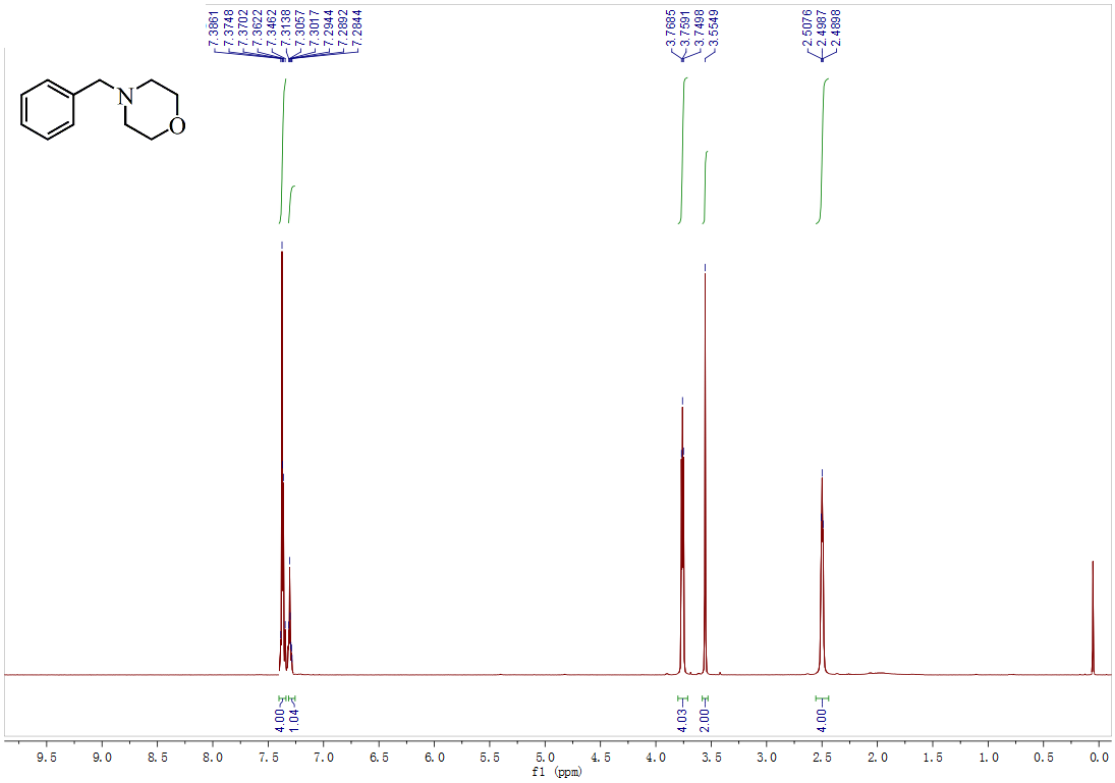
f1 (ppm)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3.7591
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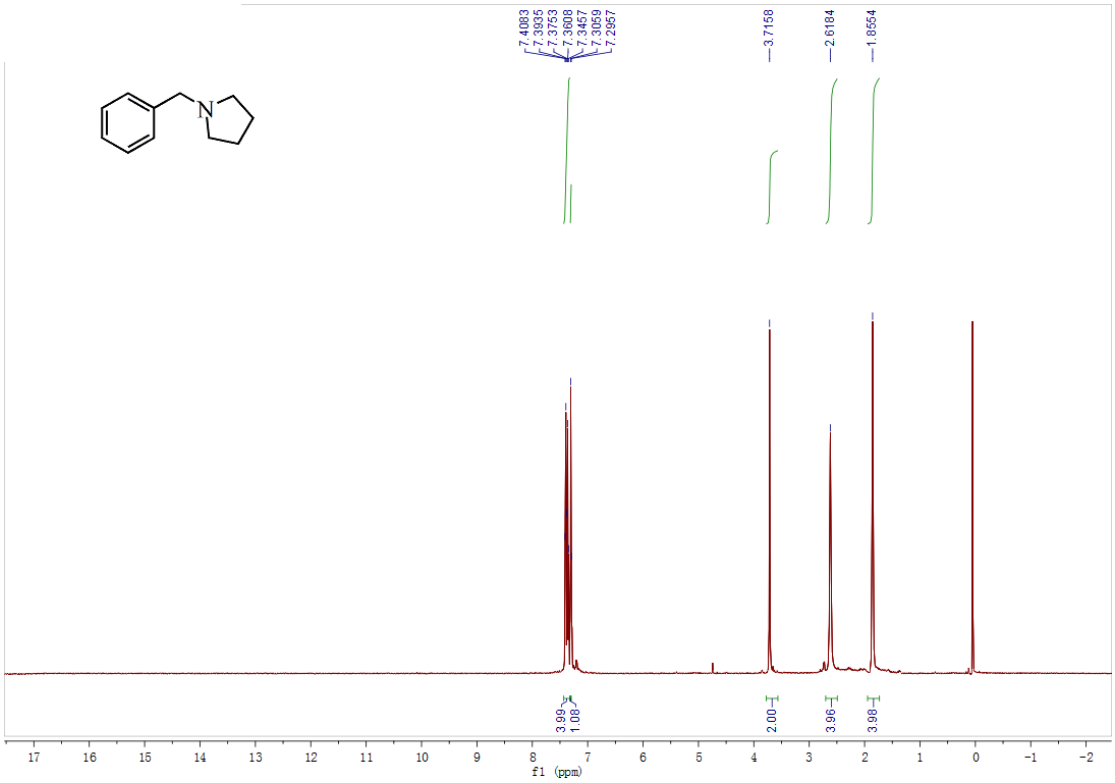
4.00
f1 (ppm)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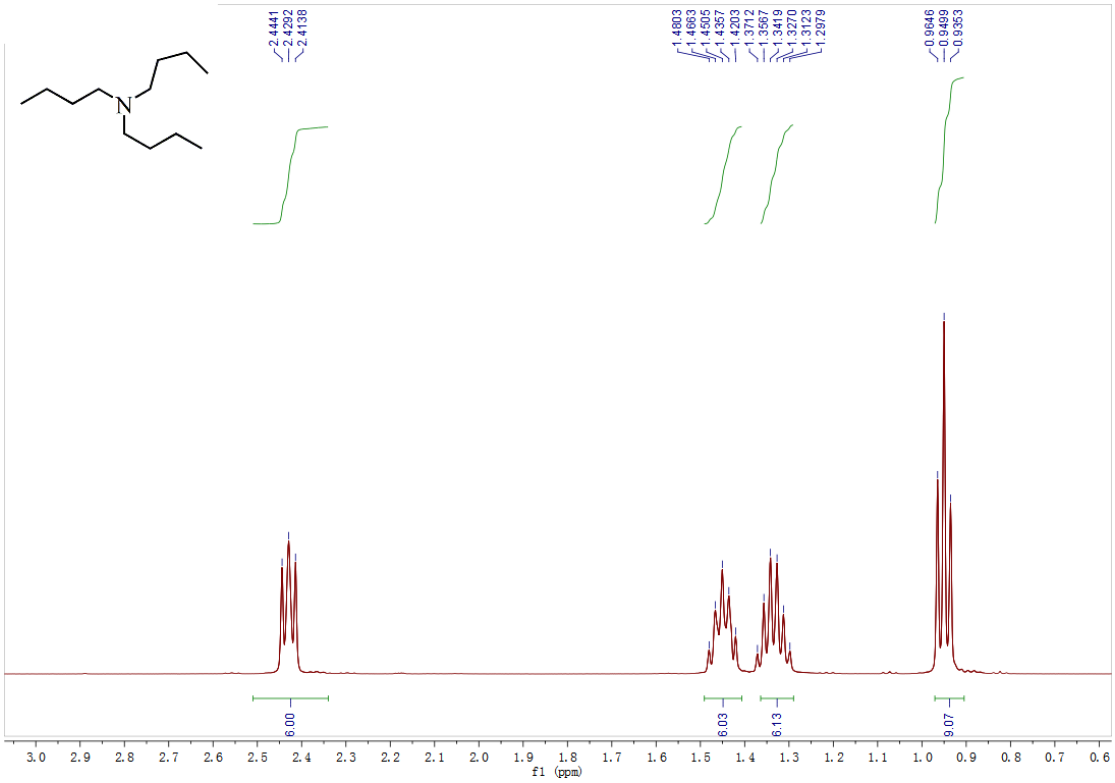
3.98
f1 (ppm)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6.03
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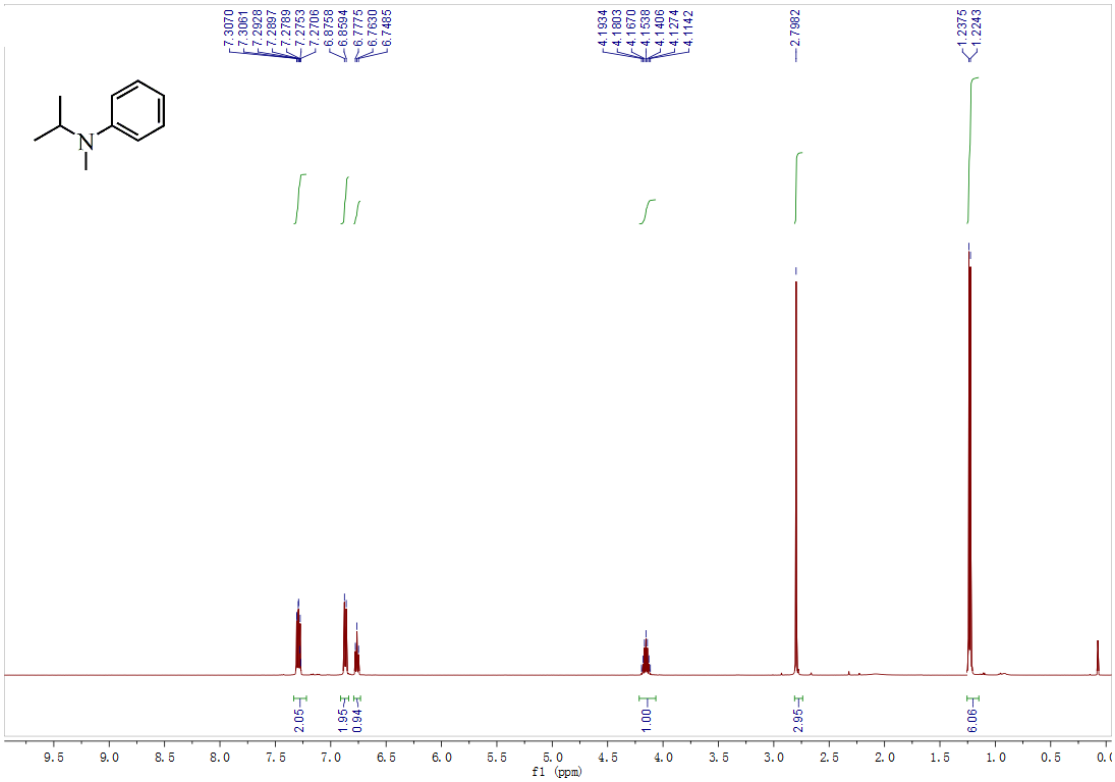
9.07
f1 (ppm)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4.1406
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0.94
1.00
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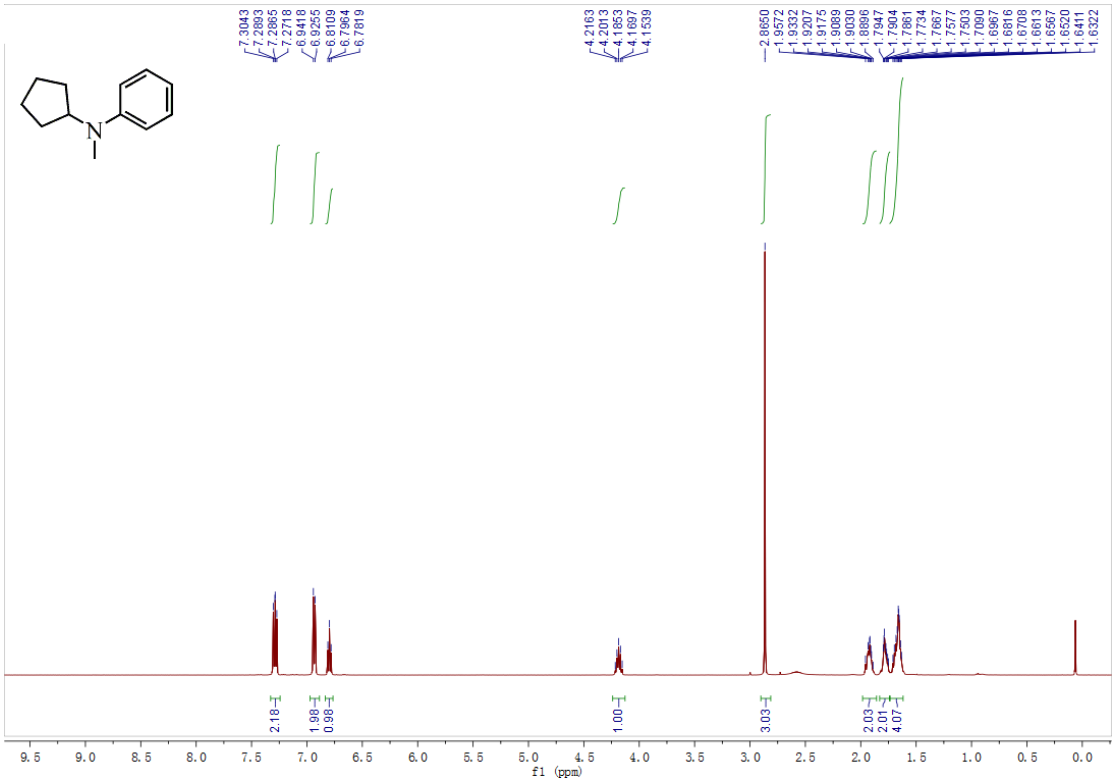
6.06
f1 (ppm)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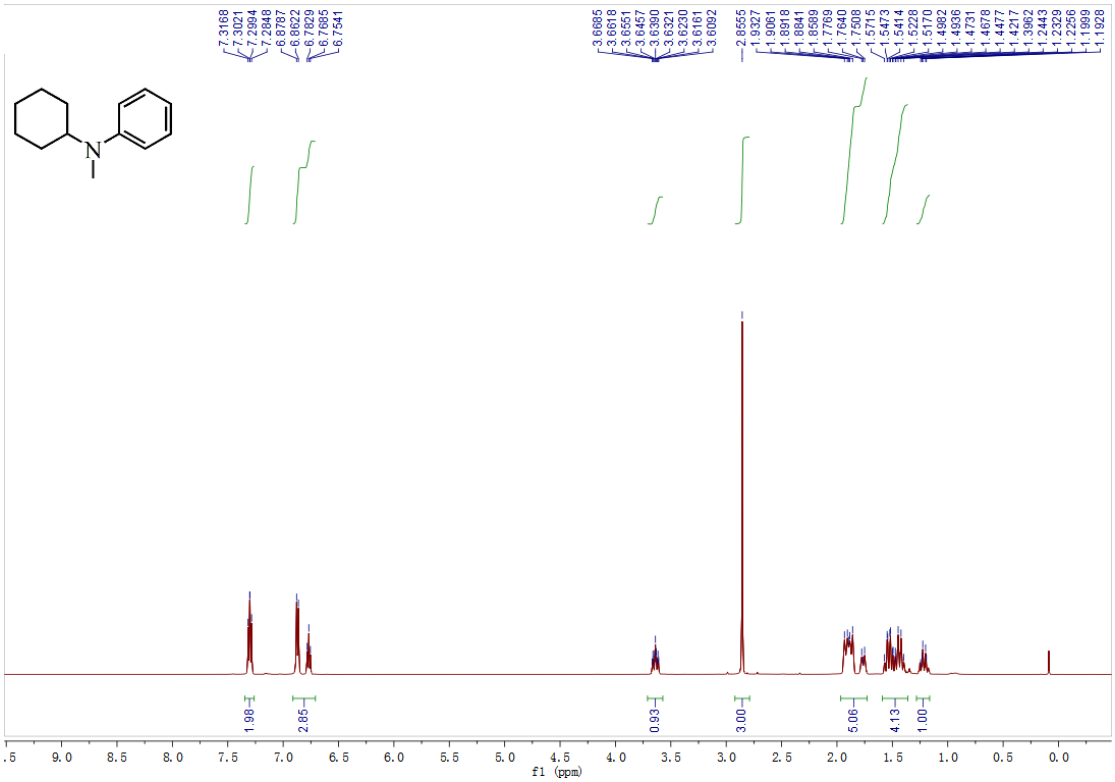
4.07
f1 (ppm)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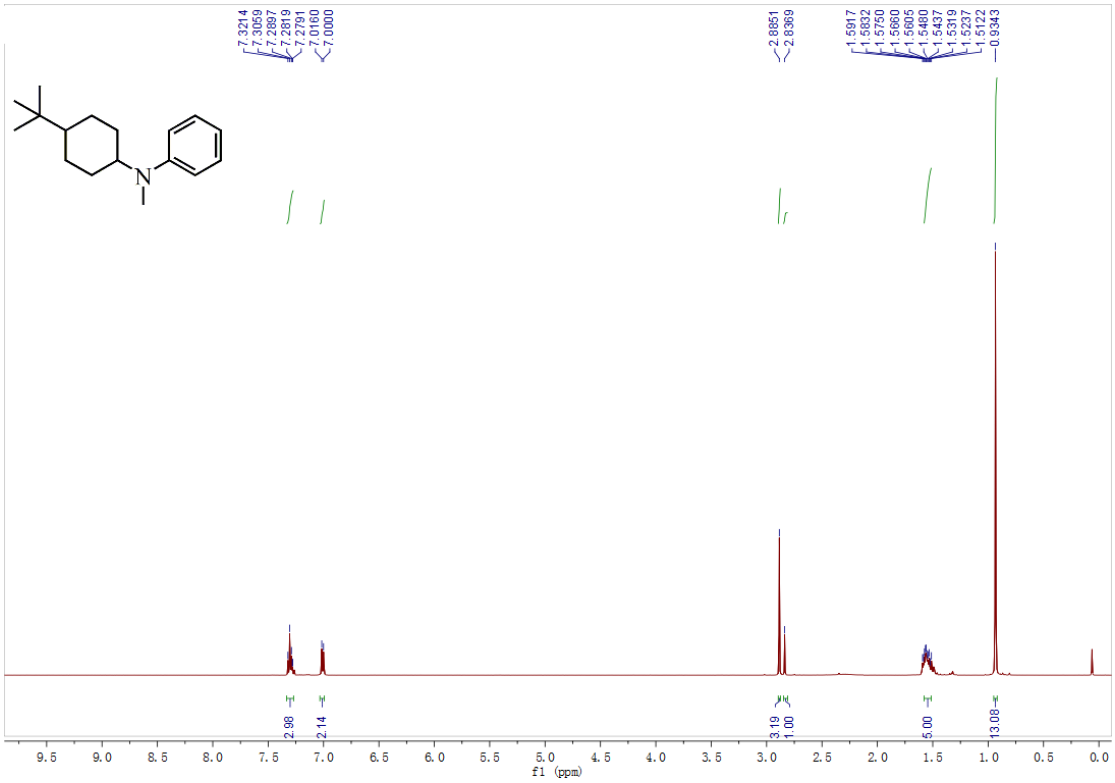
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13.08
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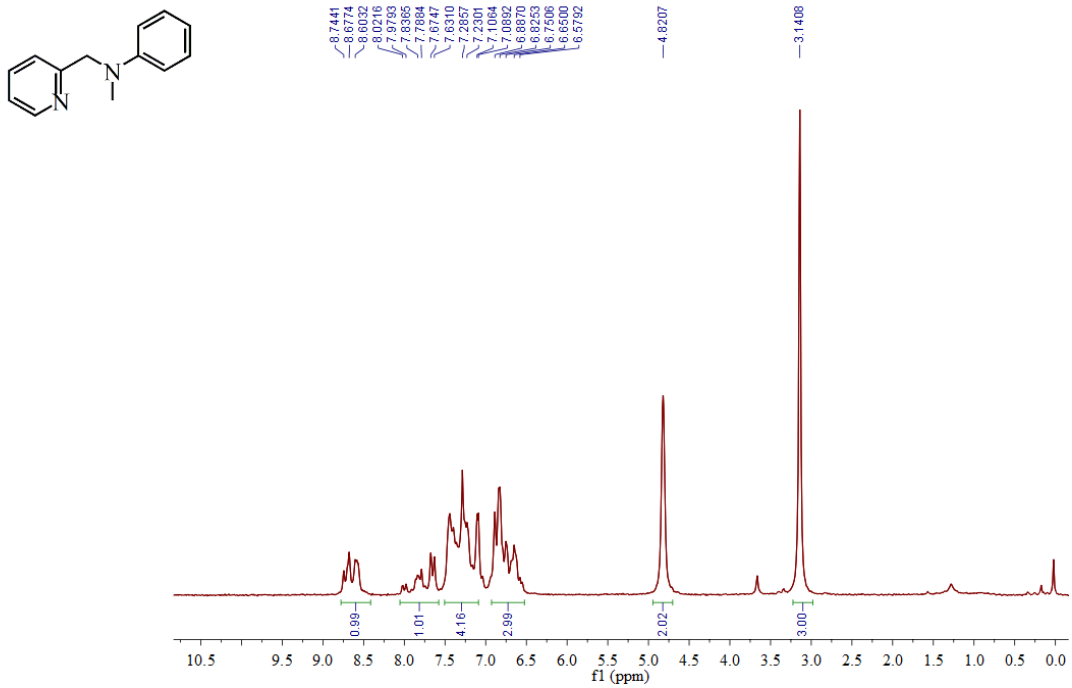
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2.02
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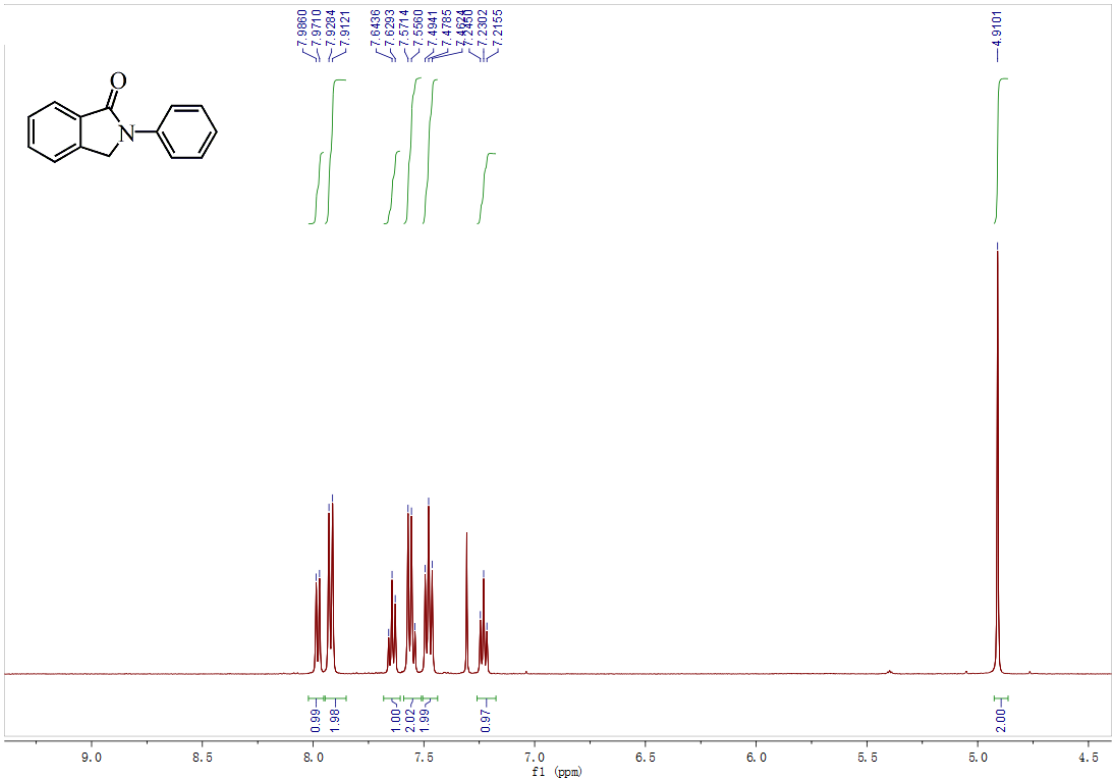
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1.98
1.00
2.02
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7.0
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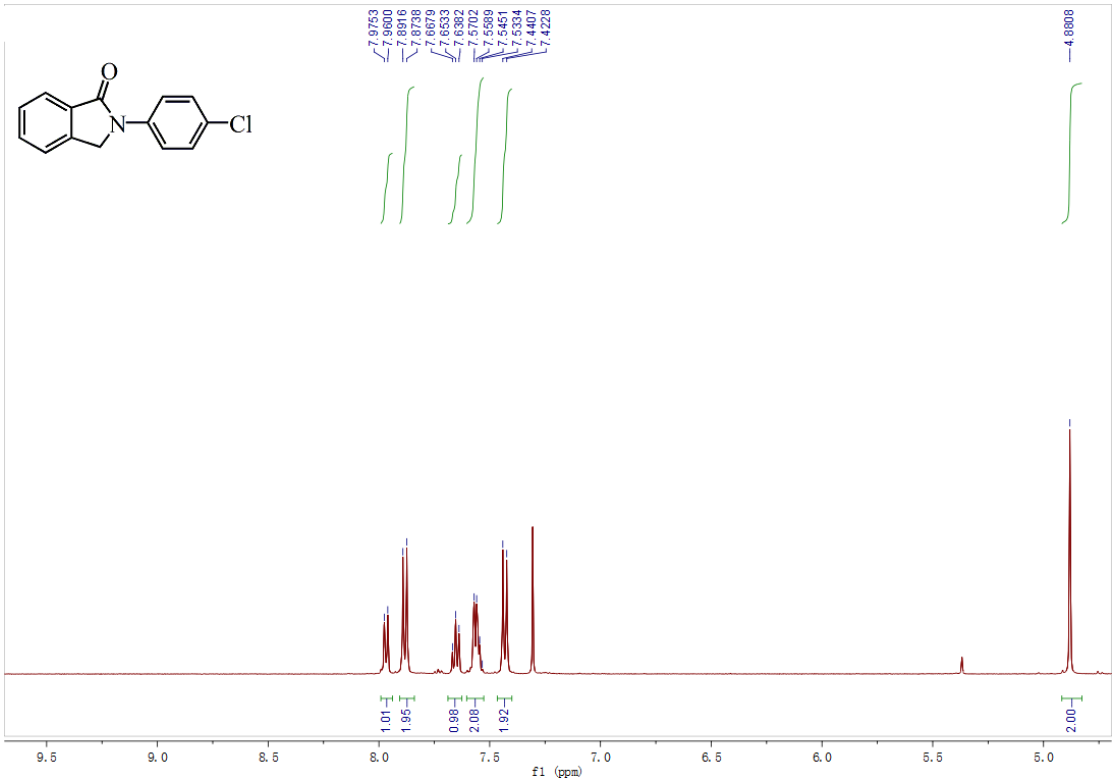
4.5
f1 (ppm)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7.4228
4.8808
1.01
1.95
0.98
2.08
1.92
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7.5
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6.0
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5.0
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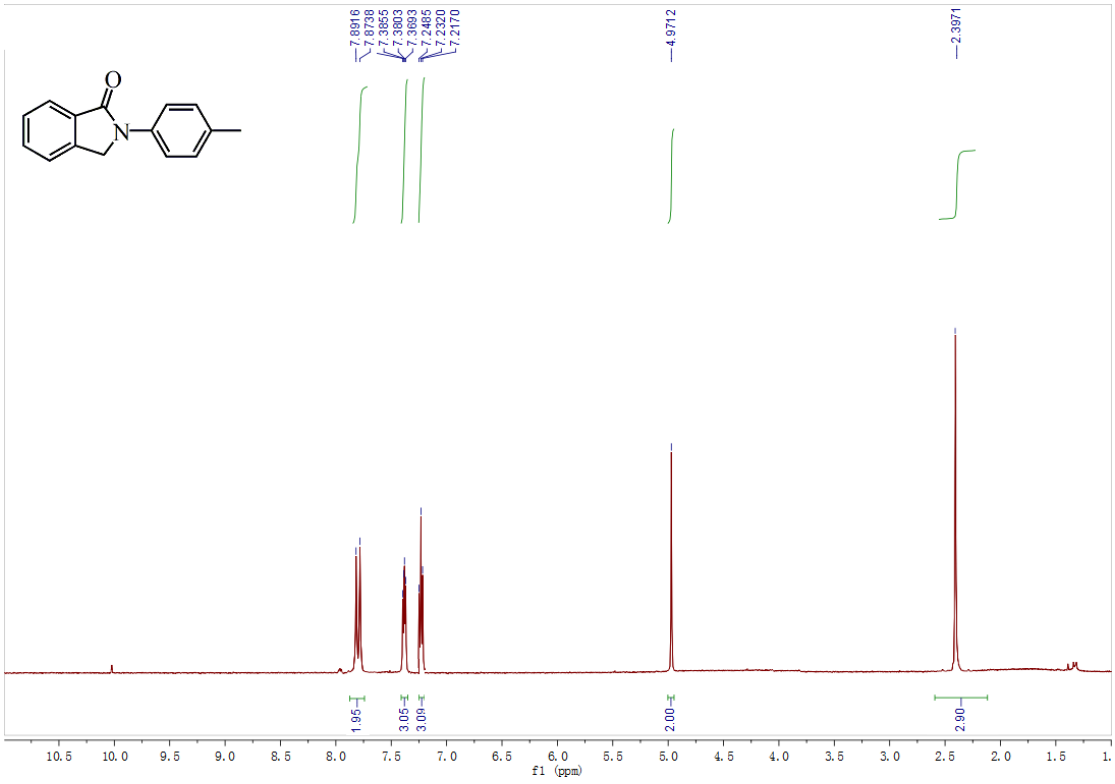
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3.05
3.00
2.00
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f1 (ppm)