Highly Dispersed Ruthenium Capsulated in UiO-66-NH_2 for Borrowing Hydrogen Mediated N-Alkylation Reactions

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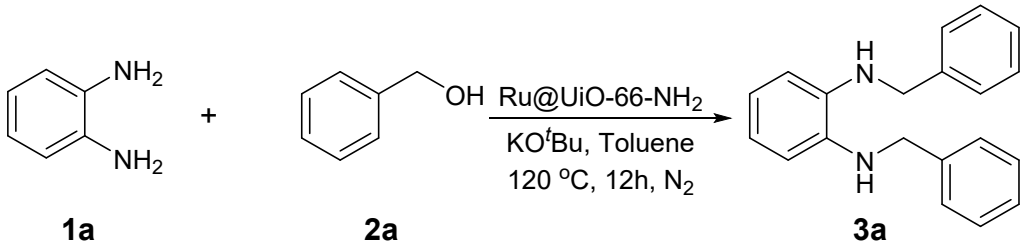
1. General Methods and materials:

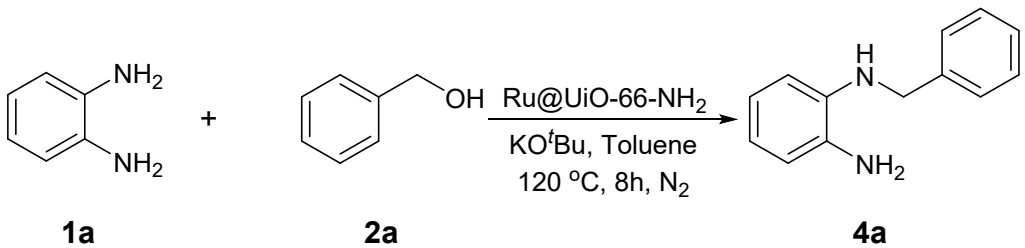
All the reactions dealing with air and/or moisture-sensitive reactions were carried out underan atmosphere of nitrogen using oven/flame-dried glassware and standard syringe/septa techniques. Unless otherwise noted, all commercial reagents and solvents were obtained from the commercial provider and used without further purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on Varian 400 or 101 MHz spectrometers. Chemical shifts were reported relative to internal tetramethylsilane (δ = 0.00 ppm) or $CDCL_3$ (δ = 7.26 ppm) for ^{1}H NMR and $CDCL_3$ (δ = 77.0 ppm) for ^{13}C NMR. Flash column chromatography was performed on 230-430 mesh silica gel. Analytical thin layer chromatography was performed with precoated glass baked plates (250μ) and visualized by fluorescence and by charring after treatment with potassium permanganate stain. TEM was recorded on a transmission electron microscope (JEM-2100, JEOL, Japan), operating at 200 kV. TEM-EDS was performed on an Avio 200 Transmission Electron Microscope. XPS data were recorded with electron energy analyzer (ESCALAB 250Xi, Thermo Fisher Co, USA). Inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis for Ru loading amount was determined by a Jarrell-Ash 1100 ICP-AES spectrometer.

2. Catalytic reactions



1a **2a** **3a**

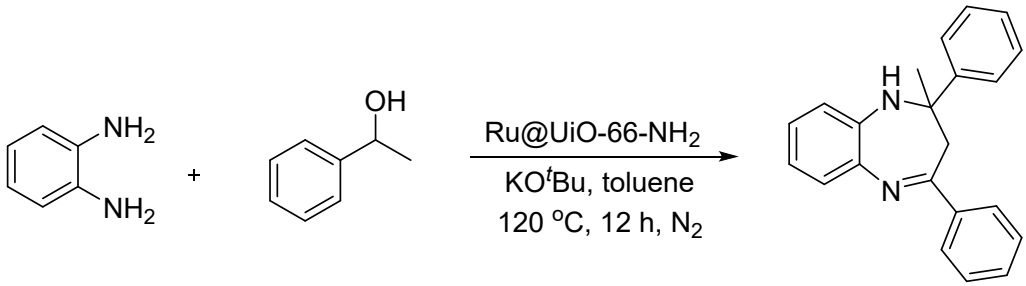
The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and Benzyl alcohol **2a** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.



1a **2a** **4a**

The mixture of 1,2-Phenylenediamine **1a** (4 mmol) and Benzyl alcohol **2a** (1 mmol), potassium tert-

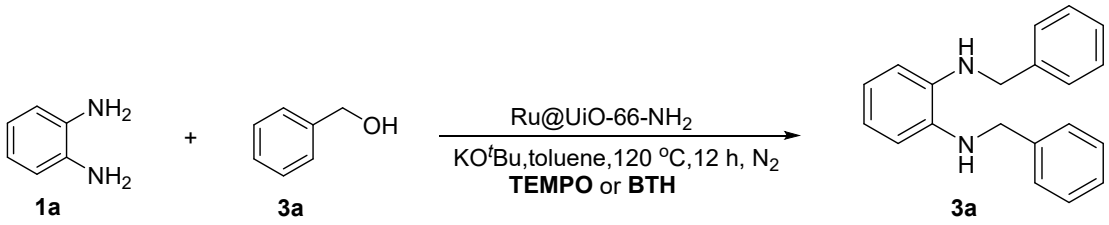
butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 8 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.



The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and 1-phenylethanol (2 mmol), potassium tert-butoxide (1 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

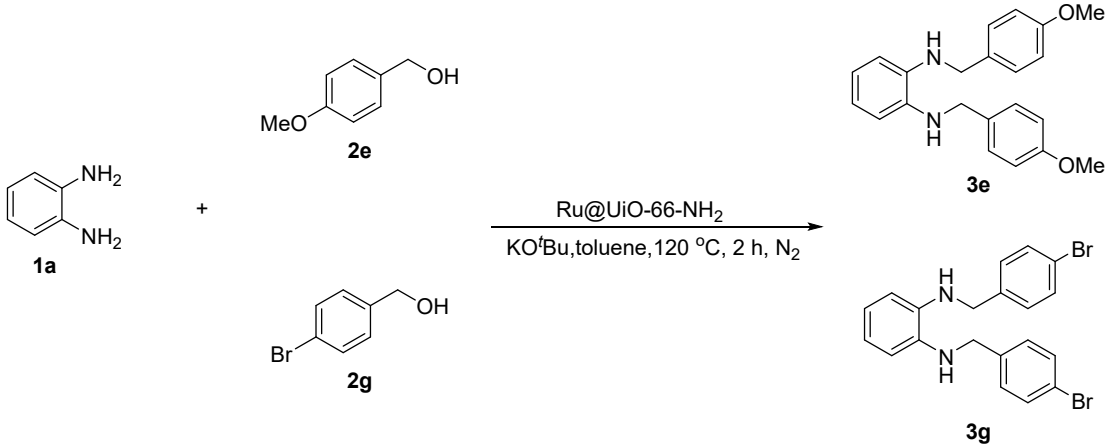
3. Mechanism study

3.1 Radical trapping experiment



The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and Benzyl alcohol **2a** (2 mmol), potassium tert-butoxide (0.5 mmol), TEMPO or BTH (1 mmol) and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

3.2 The effect of different substituents



The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and 4-methoxybenzyl alcohol **2e** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 2 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and 4-bromobenzyl alcohol **2g** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120

°C for 2 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

3.3 Control experiments

1. The mixture of benzyl alcohol **2a** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

2. The mixture of 1,2-Phenylenediamine **1a** (1 mmol) and benzaldehyde **2a'** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

3. The mixture of N^1, N^2-(1,2-phenylene)-bis(1-phenylmethanimine) **6a** (1 mmol) and benzyl alcohol **2a** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

4. The mixture of 2-(benzylideneamino)-aniline **5a** (1 mmol) and benzyl alcohol **2a** (2 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

5. The mixture of 2-(benzylideneamino)-aniline **5a** (4 mmol) and benzyl alcohol **2a** (1 mmol), potassium tert-butoxide (0.5 mmol), and Ru@UiO-66-NH_2 were reacted in toluene (3 mL) at 120 °C for 12 h under N_2. After the temperature was cooled to room temperature, the resulting solution was further purified by column chromatography.

4. Reusability of the catalyst

To a 25 mL reaction tube was added the catalyst Ru@UiO-66-NH_2 (30 mg), KO^{t}Bu (0.5 mmol), 1a (1 mmol), 2a (2 mmol). The mixture was heated at 120 °C for 12 h under N_2. After the reaction mixture was cooled to room temperature, the catalyst was separated by centrifugation with ethyl acetate, washed with carrene, dried in a vacuum, and reused for the next time.

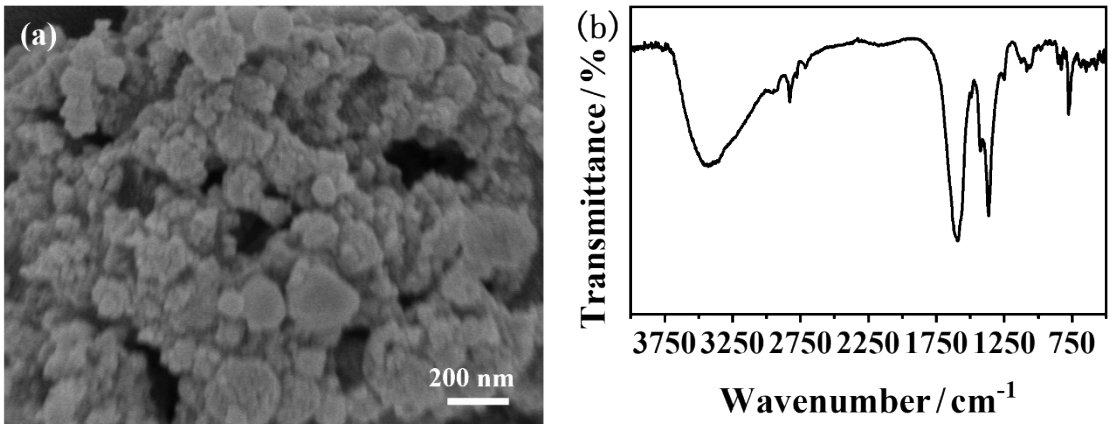
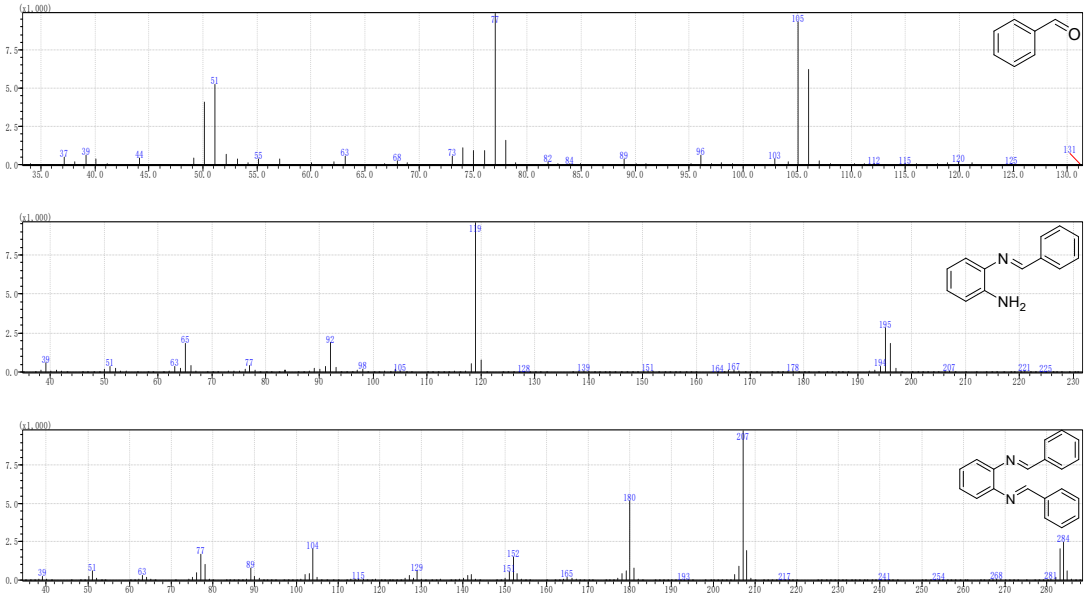


Fig. S1 (a) SEM image and (b) FT-IR spectra of recycled Ru@UiO-66-NH_2 with KO^{t}Bu.

5. GC-MS

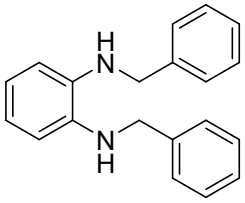
The mixture of 1,2-Phenylenediamine 1a (1 mmol) and Benzyl alcohol 2a (2 mmol), Potassium tert-butoxide (0.5 mmol), Ru@UiO-66-NH_2 (30 mg) were combined in toluene (2 mL) at 120 °C for 0.5 h under N_2. The reaction mixture was cooled to room temperature, filtered with diatomite,

diluted with methanol and analyzed by GC-MS.



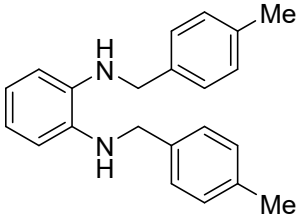
6. Analytical data of the obtained compounds

(1) *N*^{1},*N*^{2}-dibenzylbenzene-1,2-diamine(3a)[1]



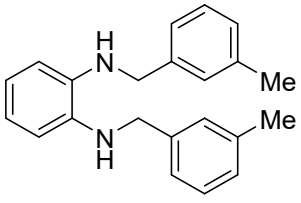
Yellow oil (239.0 mg, 0.83 mmol, 83%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.47 – 7.38 (m, 8H), 7.37 – 7.31 (m, 2H), 6.87 (dd, J = 5.8, 3.5 Hz, 2H), 6.79 (dd, J = 5.8, 3.5 Hz, 2H), 4.37 (s, 4H), 3.70 (s, 2H).^{13}C NMR (101 MHz, $CDCL_3$) δ 139.59, 137.31, 128.80, 128.01, 127.44, 119.62, 112.15, 48.96.

(2) *N*^{1},*N*^{2}-bis(4-methylbenzyl)benzene-1,2-diamine(3b)[1]



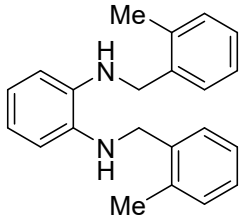
Yellow oil (265.4 mg, 0.84 mmol, 84%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.47 – 7.38 (m, 8H), 7.37 – 7.31 (m, 2H), 6.87 (dd, *J* = 5.8, 3.5 Hz, 2H), 6.79 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.37 (s, 4H), 3.70 (s, 2H).^{13}C NMR (101 MHz, $CDCL_3$) δ 139.59, 137.31, 128.80, 128.01, 127.44, 119.62, 112.15, 48.96.

(3) *N*^{1},*N*^{2}-bis(3-methylbenzyl)benzene-1,2-diamine(3c)[2]



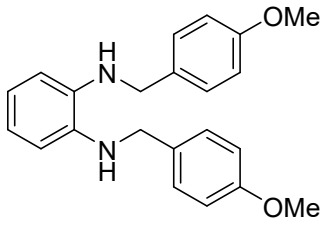
Yellow oil (224.3 mg, 0.71 mmol, 71%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.30 – 7.21 (m, 6H), 7.16 – 7.11 (m, 2H), 6.85 (dd, *J* = 5.8, 3.5 Hz, 2H), 6.77 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.31 (s, 4H), 3.66 (s, 2H), 2.40 (s, 6H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 139.49, 138.42, 137.35, 128.84, 128.68, 128.21, 125.10, 119.51, 111.94, 48.97, 21.64.

(4) *N*^{1},*N*^{2}-bis(2-methylbenzyl)benzene-1,2-diamine(3d)[2]



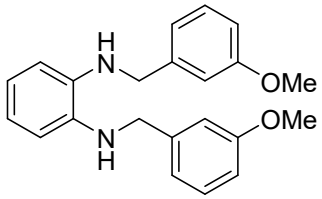
Yellow oil (214.9 mg, 0.68 mmol, 68%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.37 – 7.31 (m, 2H), 7.26 – 7.17 (m, 6H), 6.90 – 6.85 (m, 2H), 6.78 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.28 (s, 4H), 3.47 (s, 2H), 2.40 (s, 6H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 137.38, 137.16, 136.82, 130.57, 128.73, 127.63, 126.30, 119.49, 111.77, 46.93, 19.14.

(5) *N*^{1},*N*^{2}-bis(4-methoxybenzyl)benzene-1,2-diamine(3e)[1]



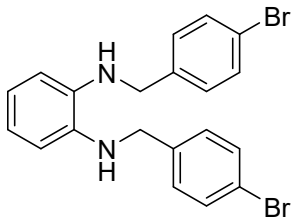
Yellow oil (285.3mg, 0.82 mmol, 82%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.33 (d, *J* = 8.7 Hz, 4H), 6.95 – 6.88 (m, 4H), 6.84 (dd, *J* = 5.7, 3.4 Hz, 2H), 6.76 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.26 (s, 4H), 3.83 (s, 6H), 3.58 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 158.97, 137.28, 131.53, 129.30, 119.46, 114.10, 111.88, 55.44, 48.36.

(6) *N*^{1},*N*^{2}-bis(3-methoxybenzyl)benzene-1,2-diamine(3f)



Yellow solid (261 mg, 0.75 mmol, 75%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.34 – 7.28 (m, 2H), 7.06 – 6.97 (m, 4H), 6.90 – 6.80 (m, 4H), 6.75 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.33 (s, 4H), 3.83 (s, 6H), 3.72 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 159.97, 141.28, 137.24, 129.80, 120.24, 119.59, 113.59, 112.71, 112.08, 55.35, 48.91.

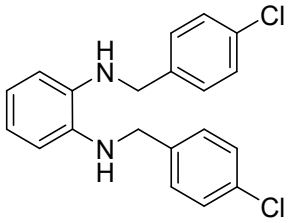
(7) *N*^{1},*N*^{2}-bis(4-bromobenzyl)benzene-1,2-diamine(3g)[3]



Yellow oil (325.6 mg, 0.73 mmol, 73%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.49 – 7.43 (m, 4H), 7.28

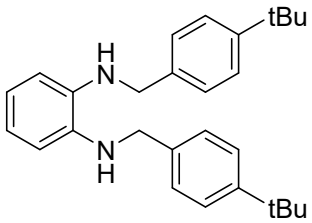
– 7.23 (m, 4H), 6.79 (dd, *J* = 5.8, 3.4 Hz, 2H), 6.66 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.27 (s, 4H), 3.65 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 138.49, 136.94, 131.81, 129.52, 121.15, 119.83, 112.37, 48.24.

(8) *N*^{1},*N*^{2}-bis(4-chlorobenzyl)benzene-1,2-diamine(3h)[3]



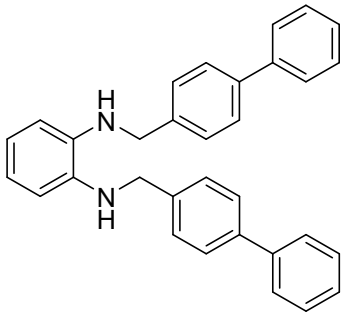
Yellow oil (267.7 mg, 0.75 mmol, 75%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.32 (s, 8H), 6.81 (dd, *J* = 5.8, 3.4 Hz, 2H), 6.67 (dd, *J* = 5.8, 3.5 Hz, 2H), 4.30 (s, 4H), 3.66 (s, 2H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 137.95, 136.95, 133.07, 129.20, 128.88, 119.79, 112.27, 48.18.

(9) *N*^{1},*N*^{2}-bis(4-(tert-butyl)benzyl)benzene-1,2-diamine(3i)



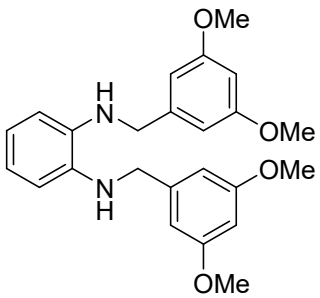
Yellow solid (348 mg, 0.87 mmol, 87%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.46 – 7.43 (m, 4H), 7.39 (d, *J* = 8.5 Hz, 4H), 6.91 – 6.85 (m, 2H), 6.84 – 6.79 (m, 2H), 4.33 (s, 4H), 3.65 (s, 2H), 1.39 (s, 18H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 150.40, 137.39, 136.53, 127.98, 125.72, 119.50, 111.88, 48.64, 34.71, 31.60.

(10) *N*^{1},*N*^{2}-bis([1,1'-biphenyl]-4-ylmethyl)benzene-1,2-diamine(3j)



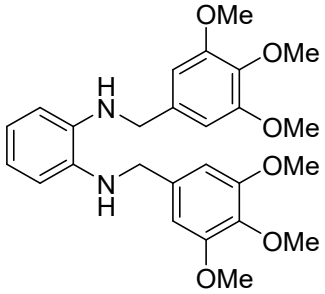
Yellow solid (277.2 mg, 0.63 mmol. 63%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.60 – 7.55 (m, 8H), 7.49 – 7.41 (m, 8H), 7.37 – 7.31 (m, 2H), 6.87 – 6.79 (m, 2H), 6.78 – 6.72 (m, 2H), 4.37 (s, 4H), 3.71 (s, 2H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 140.95, 140.36, 138.55, 137.23, 128.90, 128.40, 127.49, 127.38, 127.20, 119.61, 112.12, 48.60.

(11) *N*^{1},*N*^{2}-bis(3,5-dimethoxybenzyl)benzene-1,2-diamine(3k)



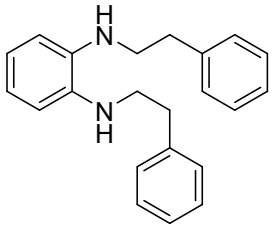
Yellow solid (350.9 mg, 0.86 mmol, 86%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 6.79 (dd, *J* = 5.8, 3.4 Hz, 2H), 6.70 (dd, *J* = 5.8, 3.5 Hz, 2H), 6.56 (d, *J* = 2.3 Hz, 4H), 6.38 (t, *J* = 2.3 Hz, 2H), 4.25 (s, 4H), 3.77 (s, 12H).^{13}C NMR (101 MHz, $CDCL_3$) δ 161.09, 142.09, 137.18, 119.61, 112.13, 105.81, 99.12, 55.44, 49.11.

(12) *N*^{1},*N*^{2}-bis(3,4,5-trimethoxybenzyl)benzene-1,2-diamine(3l)



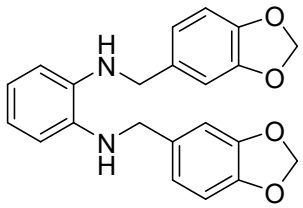
Yellow solid (248.0 mg, 0.53 mmol, 53%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 6.82 (dd, *J* = 5.8, 3.4 Hz, 2H), 6.73 (dd, *J* = 5.8, 3.5 Hz, 2H), 6.62 (s, 4H), 4.23 (s, 4H), 3.83 (d, *J* = 3.3 Hz, 18H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 153.48, 137.19, 137.14, 135.22, 119.81, 112.30, 104.88, 61.01, 56.21, 49.54.

(13) *N*^{1},*N*^{2}-diphenethylbenzene-1,2-diamine(3m)[4]



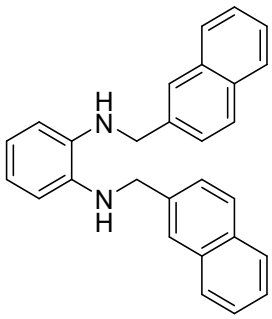
Yellow oil (212.2 mg, 0.68 mmol. 68%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.38 – 7.32 (m, 4H), 7.27 (td, *J* = 6.9, 1.6 Hz, 6H), 6.82 (dd, *J* = 5.8, 3.5 Hz, 2H), 6.72 (dd, *J* = 5.8, 3.5 Hz, 2H), 3.35 (t, *J* = 7.0 Hz, 4H), 2.95 (t, *J* = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 139.67, 137.34, 128.94, 128.74, 126.57, 119.42, 112.00, 45.63, 35.99.

(14) *N*^{1},*N*^{2}-bis(benzo[d][1,3]dioxol-5-ylmethyl)benzene-1,2-diamine(3n)



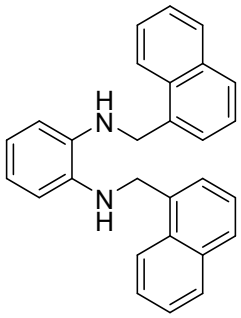
Yellow solid (302.4 mg, 0.80 mmol. 80%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 6.89 (d, *J* = 1.7 Hz, 2H), 6.86 – 6.76 (m, 6H), 6.71 (dd, *J* = 5.7, 3.4 Hz, 2H), 5.94 (s, 4H), 4.21 (s, 4H), 3.56 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 147.96, 146.89, 137.12, 133.39, 121.07, 119.58, 112.11, 108.52, 108.39, 101.10, 48.74.

(15) *N*^{1},*N*^{2}-bis(naphthalen-2-ylmethyl)benzene-1,2-diamine(3o)[5]



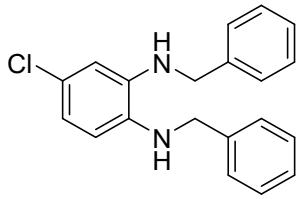
Yellow solid (287.1 mg, 0.74 mmol. 74%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.86 – 7.79 (m, 8H), 7.55 – 7.52 (m, 2H), 7.50 – 7.45 (m, 4H), 6.85 – 6.77 (m, 4H), 4.50 (s, 4H), 3.79 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 137.35, 137.05, 133.60, 132.91, 128.46, 127.90, 127.83, 126.38, 126.26, 126.23, 125.86, 119.69, 112.34, 49.15.

(16) *N*^{1},*N*^{2}-bis(naphthalen-1-ylmethyl)benzene-1,2-diamine(3p)



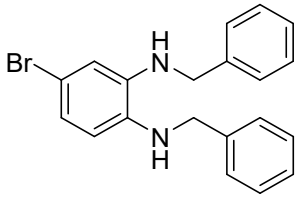
Yellow solid (248.3 mg, 0.64 mmol. 64%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 8.10 – 8.03 (m, 2H), 7.89 – 7.83 (m, 2H), 7.77 (d, *J* = 8.2 Hz, 2H), 7.50 – 7.43 (m, 6H), 7.39 (dd, *J* = 8.2, 7.0 Hz, 2H), 6.87 (d, *J* = 2.7 Hz, 4H), 4.71 (s, 4H), 3.69 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 137.33, 134.53, 133.91, 131.83, 128.80, 128.23, 126.33, 126.26, 125.86, 125.59, 123.77, 119.66, 112.08, 46.86.

(17) *N*^{1},*N*^{2}-dibenzyl-4-chlorobenzene-1,2-diamine(3q)[6]



Yellow oil (258.0 mg, 0.80 mmol, 80%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.41 – 7.35 (m, 8H), 7.34 – 7.29 (m, 2H), 6.73 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.68 (d, *J* = 2.3 Hz, 1H), 6.60 (d, *J* = 8.3 Hz, 1H), 4.28 (s, 4H), 3.68 (s, 1H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 139.02, 138.76, 138.64, 135.36, 128.86, 128.80, 128.02, 127.93, 127.65, 127.54, 124.89, 118.57, 113.02, 111.76, 48.94, 48.71.

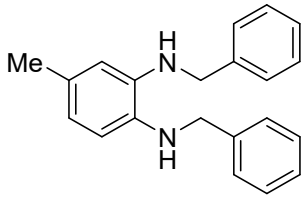
(18) *N*^{1},*N*^{2}-dibenzyl-4-bromobenzene-1,2-diamine(3r)



Yellow oil (238.5 mg, 0.65 mmol, 65%). ^{1}H NMR (400 MHz, $CDCL_3$) δ 7.36 (ddd, *J* = 7.9, 3.8, 1.9 Hz, 8H), 7.33 – 7.28 (m, 2H), 6.87 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.81 (d, *J* = 2.2 Hz, 1H), 6.54 (d, *J* = 8.3 Hz, 1H), 4.27 (d, *J* = 3.7 Hz, 4H), 3.64 (s, 2H). ^{13}C NMR (101 MHz, $CDCL_3$) δ 138.97, 138.76,

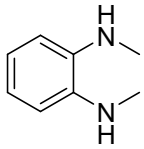
138.73, 135.96, 128.86, 128.80, 128.07, 127.88, 127.66, 127.53, 121.69, 114.51, 113.35, 112.13, 48.81, 48.76.

(19) *N*^{1},*N*^{2}-dibenzyl-4-methylbenzene-1,2-diamine(3s)[6]



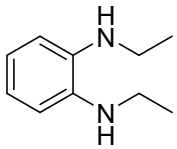
Yellow oil (244.6 mg, 0.81 mmol, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.34 (m, 10H), 6.76 – 6.67 (m, 2H), 6.66 (d, *J* = 1.8 Hz, 1H), 4.38 (d, *J* = 8.9 Hz, 4H), 3.66 (s, 1H), 2.37 (s, 3H).^{13}C NMR (101 MHz, $CDCL_3$) δ 139.82, 139.72, 137.87, 134.62, 129.38, 128.85, 128.80, 128.11, 128.05, 127.48, 127.42, 119.46, 112.84, 112.77, 49.30, 48.95, 21.40.

(20) *N*^{1},*N*^{2}-dimethylbenzene-1,2-diamine (3t) [1]



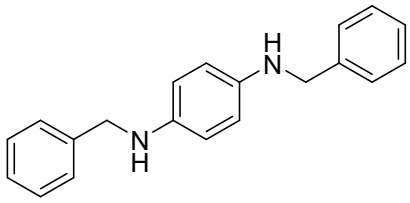
Black solid (34.1 mg, 0.25 mmol. 25%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (ddd, *J* = 5.8, 3.5, 1.2 Hz, 2H), 6.69 (ddd, *J* = 5.6, 3.5, 1.1 Hz, 2H), 2.86 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.44, 119.17, 110.58, 31.15.

(21) *N*^{1},*N*^{2}-diethylbenzene-1,2-diamine (3u)[1]



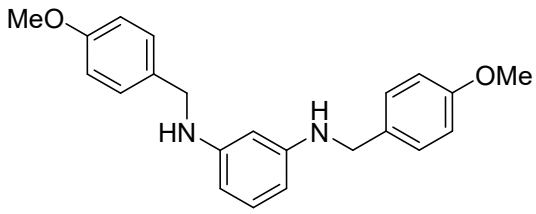
Black solid (52.5 mg, 0.32 mmol. 32%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (dd, *J* = 5.8, 3.4 Hz, 2H), 6.68 (dd, *J* = 5.8, 3.5 Hz, 2H), 3.17 – 3.12 (m, 4H), 1.31 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.44, 119.11, 111.47, 38.92, 15.16.

(22) *N*^{1},*N*^{4}-dibenzylbenzene-1,4-diamine (3v) [1]



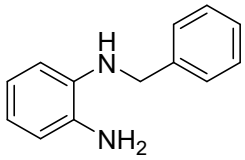
Yellow solid (252.6 mg, 0.89 mmol. 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.32 (m, 8H), 7.31 – 7.25 (m, 2H), 6.59 (d, *J* = 0.9 Hz, 4H), 4.27 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.88, 140.09, 128.67, 127.74, 127.21, 114.80, 49.63.

(23) *N*^{1},*N*^{3}-bis(4-methoxybenzyl)benzene-1,3-diamine (3w) [1]



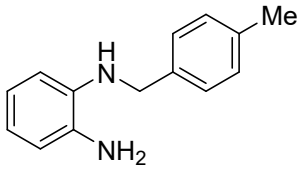
Yellow solid (289.2 mg, 0.83 mmol. 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 4H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 8.7 Hz, 4H), 6.06 (dd, *J* = 8.0, 2.2 Hz, 2H), 5.92 (t, *J* = 2.2 Hz, 1H), 4.20 (s, 4H), 3.80 (s, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 158.88, 149.53, 131.77, 130.11, 128.93, 114.08, 103.11, 97.30, 55.40, 47.91.

(24) *N*^{1}-benzylbenzene-1,2-diamine (4a)[7]



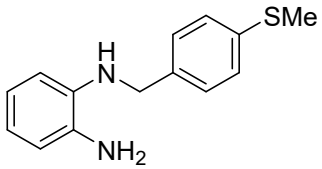
Brown oil (180.2 mg, 0.91 mmol, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.30 (m, 4H), 7.29 – 7.24 (m, 1H), 6.79 (ddd, *J* = 7.7, 6.0, 3.0 Hz, 1H), 6.73 – 6.56 (m, 3H), 4.27 (s, 2H), 3.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.52, 137.75, 134.28, 128.68, 127.87, 127.33, 120.80, 118.93, 116.63, 112.13, 48.72.

(25) *N*^{1}-(4-methylbenzyl)benzene-1,2-diamine (4b)[7]



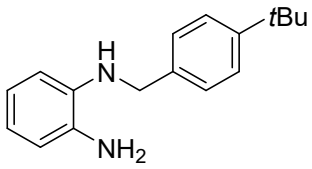
Brown oil (195.0 mg, 0.92mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 6.83 – 6.75 (m, 1H), 6.69 (tdd, *J* = 7.8, 7.3, 1.9 Hz, 3H), 4.25 (s, 2H), 3.37 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.84, 136.93, 136.45, 134.23, 129.33, 127.83, 120.76, 118.81, 116.53, 112.04, 48.46, 21.16.

(26) *N*^{1}-(4-(methylthio)benzyl)benzene-1,2-diamine (4c)



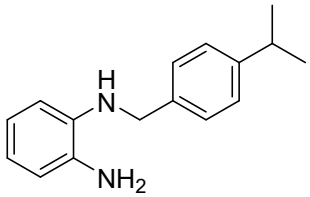
Brown oil (178.1 mg, 0.73 mmol, 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 6.79 (td, *J* = 7.4, 1.9 Hz, 1H), 6.72 (ddd, *J* = 13.4, 7.5, 1.7 Hz, 2H), 6.65 (dd, *J* = 7.8, 1.4 Hz, 1H), 4.27 (s, 2H), 3.37 (s, 3H), 2.48 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 137.50, 137.26, 136.35, 134.24, 128.33, 127.04, 120.77, 119.02, 116.65, 112.20, 48.23, 16.08.

(27) *N*^{1}-(4-(*tert*-butyl)benzyl)benzene-1,2-diamine (4d)



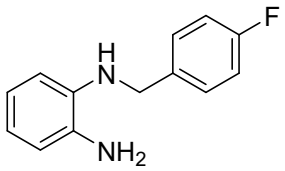
Brown oil (238.8 mg, 0.94 mmol, 94%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 6.80 (ddd, *J* = 8.6, 6.5, 2.1 Hz, 1H), 6.73 – 6.66 (m, 3H), 4.26 (s, 2H), 3.41 (s, 3H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.30, 137.88, 136.44, 134.25, 127.70, 125.57, 120.76, 118.83, 116.53, 112.01, 48.40, 34.56, 31.45.

(28) *N*^{1}-(4-isopropylbenzyl)benzene-1,2-diamine (4e)



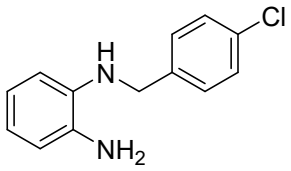
Brown oil (220.8 mg, 0.92 mmol, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.22 – 7.17 (m, 2H), 6.79 (ddd, *J* = 8.5, 5.6, 3.2 Hz, 1H), 6.68 (dd, *J* = 5.5, 2.1 Hz, 3H), 4.24 (s, 2H), 3.35 (s, 3H), 2.89 (p, *J* = 6.9 Hz, 1H), 1.24 (d, *J* = 6.9 Hz, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 148.06, 137.87, 136.85, 134.29, 128.00, 126.74, 120.77, 118.86, 116.55, 112.05, 48.52, 33.91, 24.13.

(29) *N*^{1}-(4-fluorobenzyl)benzene-1,2-diamine (4f)[7]



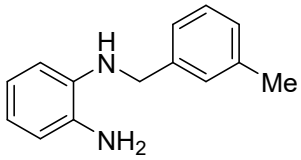
Yellow oil (179.3 mg, 0.83 mmol, 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 2H), 7.04 – 6.99 (m, 2H), 6.79 (td, *J* = 7.4, 2.0 Hz, 1H), 6.76 – 6.66 (m, 2H), 6.63 (dd, *J* = 7.8, 1.3 Hz, 1H), 4.27 (s, 2H), 3.44 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 162.11 (d, *J* = 245.0 Hz), 137.54, 135.14 (d, *J* = 3.1 Hz), 134.25, 129.31 (d, *J* = 7.9 Hz), 120.79, 119.06, 116.70, 115.43 (d, *J* = 21.3 Hz), 112.13, 47.96.

(30) *N*^{1}-(4-chlorobenzyl)benzene-1,2-diamine (4g)[8]



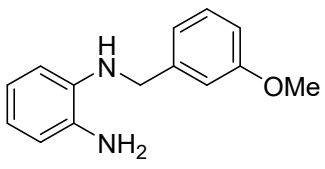
Brown oil (177.2 mg, 0.85 mmol, 85%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (s, 4H), 6.74 – 6.45 (m, 4H), 4.17 (s, 2H), 3.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.05, 137.47, 134.25, 132.95, 129.03, 128.77, 120.84, 119.13, 116.78, 112.17, 47.94.

(31) *N*^{1}-(3-methylbenzyl)benzene-1,2-diamine (4h)[7]



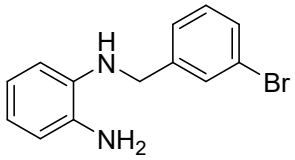
Brown oil (188.7 mg, 0.89 mmol, 89%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.15 (m, 3H), 7.08 (d, *J* = 7.3 Hz, 1H), 6.79 (ddd, *J* = 7.1, 5.9, 2.7 Hz, 1H), 6.73 – 6.63 (m, 3H), 4.23 (s, 2H), 2.34 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 139.47, 138.32, 137.87, 134.26, 128.66, 128.60, 128.11, 124.96, 120.81, 118.87, 116.59, 112.07, 48.77, 21.51.

(32) *N*^{1}-(3-methoxybenzyl)benzene-1,2-diamine (4i)[9]



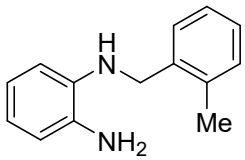
Brown oil (205.2 mg, 0.90 mmol, 90%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.18 (m, 1H), 6.92 (dd, *J* = 7.6, 1.6 Hz, 2H), 6.81 – 6.72 (m, 2H), 6.63 (tt, *J* = 8.5, 6.7 Hz, 3H), 4.19 (s, 2H), 3.71 (s, 3H), 3.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.04, 141.39, 137.78, 134.39, 129.77, 120.80, 120.17, 118.97, 116.67, 113.58, 112.73, 112.17, 55.33, 48.72.

(33) *N*^{1}-(3-bromobenzyl)benzene-1,2-diamine (4j)[9]



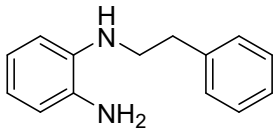
Brown oil (223.5 mg, 0.81 mmol, 81%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.15 (d, *J* = 7.7 Hz, 1H), 7.05 (t, *J* = 7.8 Hz, 1H), 6.67 (dt, *J* = 8.1, 4.3 Hz, 1H), 6.58 (d, *J* = 4.5 Hz, 2H), 6.47 (d, *J* = 7.7 Hz, 1H), 4.11 (s, 2H), 3.39 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 142.10, 137.43, 134.32, 130.69, 130.40, 130.26, 126.28, 122.81, 120.88, 119.21, 116.85, 112.20, 48.07.

(34) *N*^{1}-(2-methylbenzyl)benzene-1,2-diamine (4k)[7]



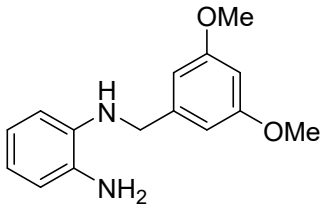
Brown oil (182.3 mg, 0.86 mmol, 86%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 7.0 Hz, 1H), 7.18 – 7.10 (m, 3H), 6.82 – 6.74 (m, 1H), 6.68 – 6.60 (m, 3H), 4.18 (s, 2H), 3.27 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.98, 137.35, 136.66, 134.36, 130.57, 128.61, 127.59, 126.33, 120.86, 118.90, 116.63, 111.97, 46.74, 19.10.

(35) *N*^{1}-phenethylbenzene-1,2-diamine (4l)[10]



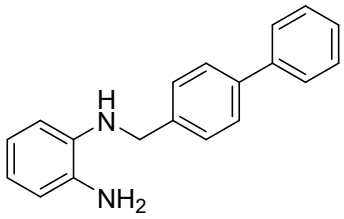
Brown oil (186.6 mg, 0.88 mmol, 88%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.4 Hz, 2H), 7.21 (d, *J* = 7.3 Hz, 3H), 6.84 – 6.77 (m, 1H), 6.70 – 6.59 (m, 3H), 3.34 (t, *J* = 7.1 Hz, 2H), 3.23 (s, 3H), 2.91 (t, *J* = 7.1 Hz, 2H).^{13}C NMR (101 MHz, $CDCl_3$) δ 139.62, 137.64, 134.47, 128.88, 128.71, 126.52, 120.78, 118.82, 116.64, 112.06, 45.50, 35.89.

(36) *N*^{1}-(3,5-dimethoxybenzyl)benzene-1,2-diamine (4m)



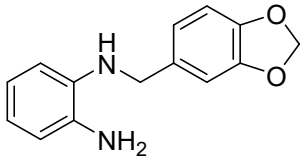
Brown oil (221.9 mg, 0.86 mmol, 86%).^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 (td, *J* = 7.1, 2.6 Hz, 1H), 6.71 – 6.65 (m, 2H), 6.63 (d, *J* = 7.9 Hz, 1H), 6.55 (d, *J* = 2.3 Hz, 2H), 6.37 (t, *J* = 2.4 Hz, 1H), 4.21 (s, 2H), 3.75 (s, 6H), 3.40 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 161.07, 142.02, 137.68, 134.22, 120.78, 118.93, 116.61, 112.14, 105.73, 99.16, 55.36, 48.91.

(37) *N*^{1}-([1,1'-biphenyl]-4-ylmethyl)benzene-1,2-diamine (4n)[11]



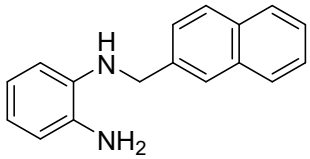
Brown oil (230.2 mg, 0.84 mmol, 84%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.56 (m, 4H), 7.48 – 7.41 (m, 4H), 7.37 – 7.32 (m, 1H), 6.84 – 6.78 (m, 1H), 6.77 – 6.66 (m, 3H), 4.36 (s, 2H), 3.41 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 140.90, 140.28, 138.54, 137.74, 134.23, 128.79, 128.20, 127.37, 127.27, 127.09, 120.80, 118.92, 116.63, 112.10, 48.37.

(38) *N*^{1}-(benzo[*d*][1,3]dioxol-5-ylmethyl)benzene-1,2-diamine (4o)



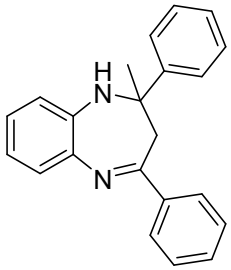
Brown oil (210.5 mg, 0.87 mmol, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (d, *J* = 1.7 Hz, 1H), 6.83 – 6.73 (m, 3H), 6.70 – 6.65 (m, 2H), 6.64 – 6.61 (m, 1H), 5.89 (s, 2H), 4.17 (s, 2H), 3.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.93, 146.81, 137.65, 134.30, 133.45, 120.93, 120.75, 118.95, 116.61, 112.11, 108.39, 108.34, 101.05, 48.50.

(39) *N*^{1}-(naphthalen-2-ylmethyl)benzene-1,2-diamine (4p)



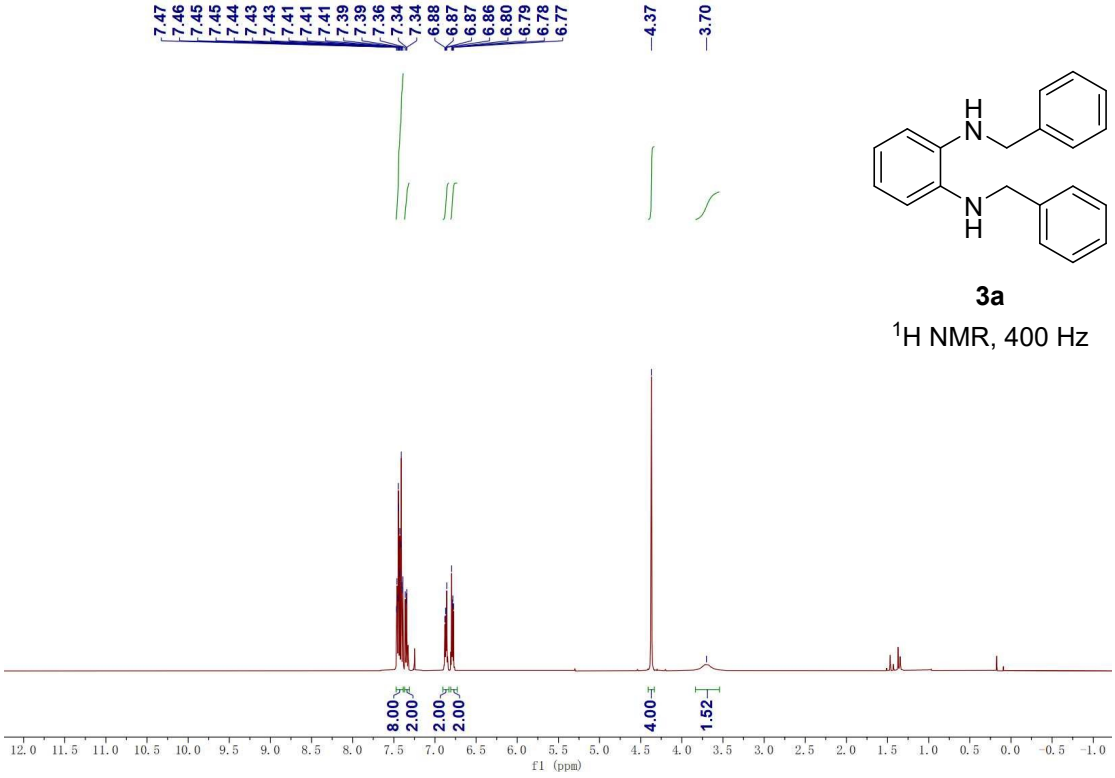
Brown oil (188.5 mg, 0.76 mmol, 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.78 (m, 4H), 7.50 – 7.42 (m, 3H), 6.79 (ddd, *J* = 8.1, 6.7, 2.0 Hz, 1H), 6.75 – 6.67 (m, 3H), 4.45 (s, 2H), 3.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.77, 136.98, 134.26, 133.55, 132.85, 128.36, 127.82, 127.75, 126.23, 126.18, 126.09, 125.78, 120.85, 118.97, 116.69, 112.22, 48.87.

(40) 2-methyl-2,4-diphenyl-2,3-dihydro-1*H*-benzo[b][1,4]diazepine



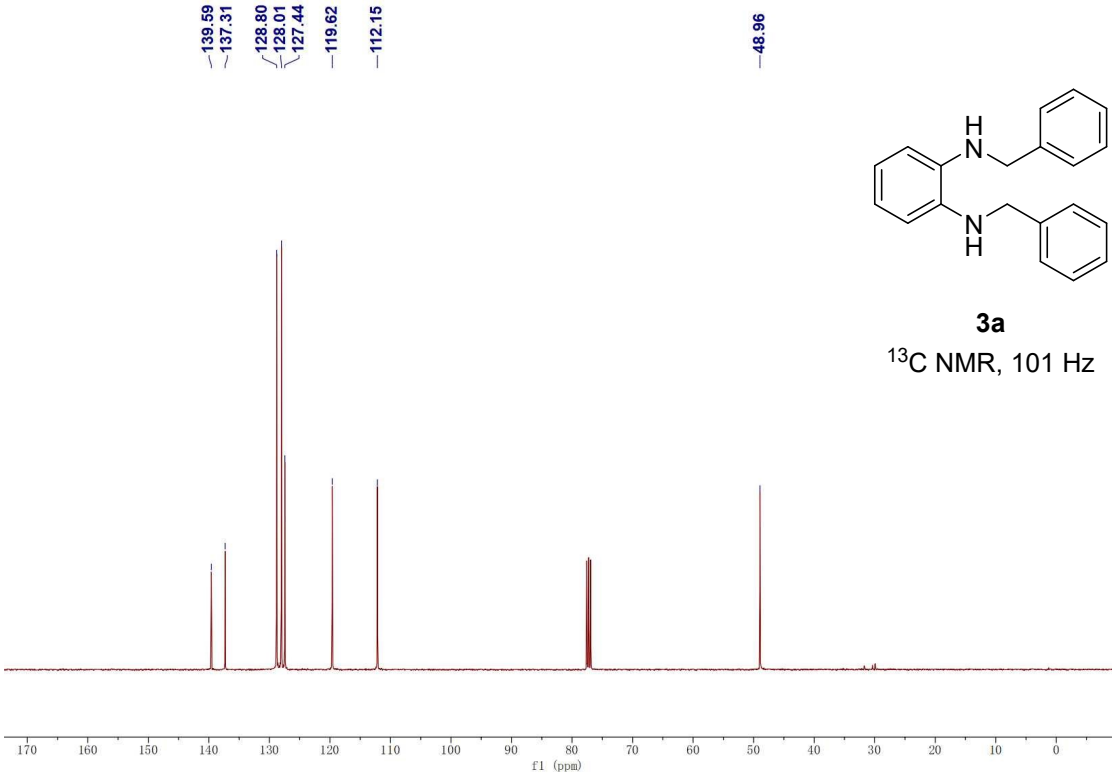
Yellow solid (224.9 mg, 0.72 mmol, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (tt, *J* = 7.4, 1.6 Hz, 4H), 7.32 – 7.15 (m, 7H), 7.06 (ddd, *J* = 8.2, 7.2, 1.9 Hz, 2H), 6.86 – 6.81 (m, 1H), 3.14 (d, *J* = 13.2 Hz, 1H), 2.97 (d, *J* = 13.2 Hz, 1H), 1.76 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.84, 147.71, 140.17, 139.67, 138.18, 129.86, 128.72, 128.68, 128.42, 128.14, 127.18, 126.46, 125.54, 121.77, 121.53, 73.83, 43.18, 29.98.

7. NMR spectra of obtained compounds



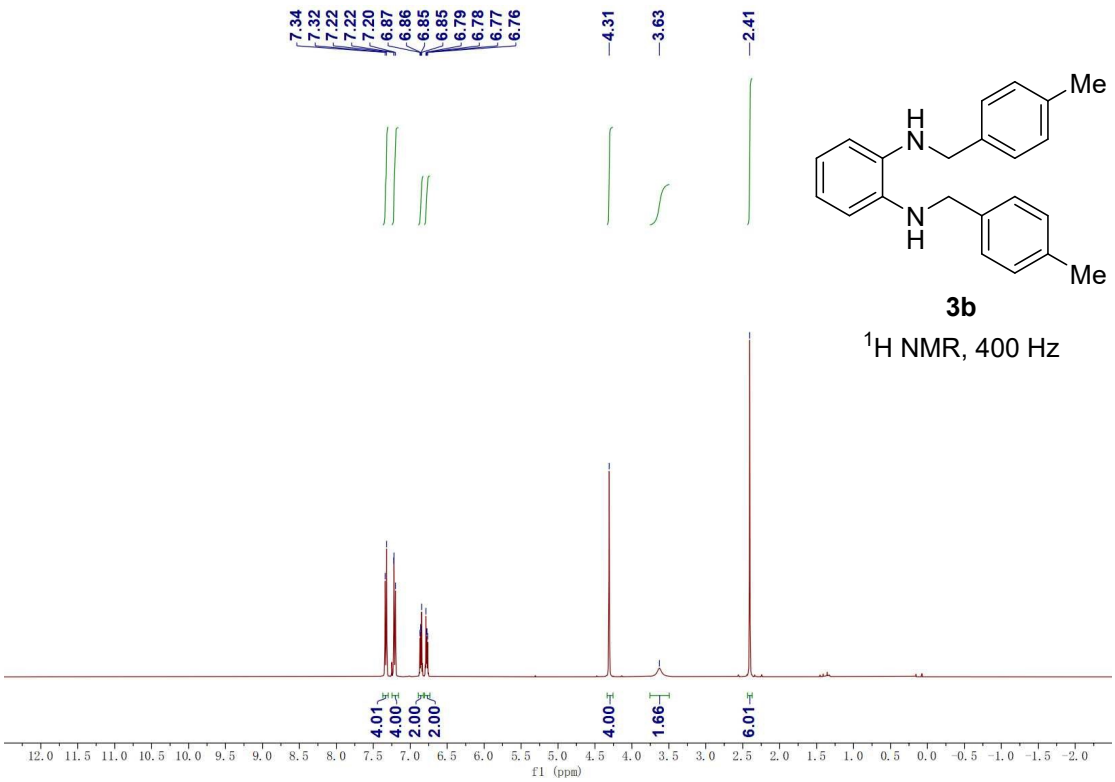
3a

^{1}H NMR, 400 Hz



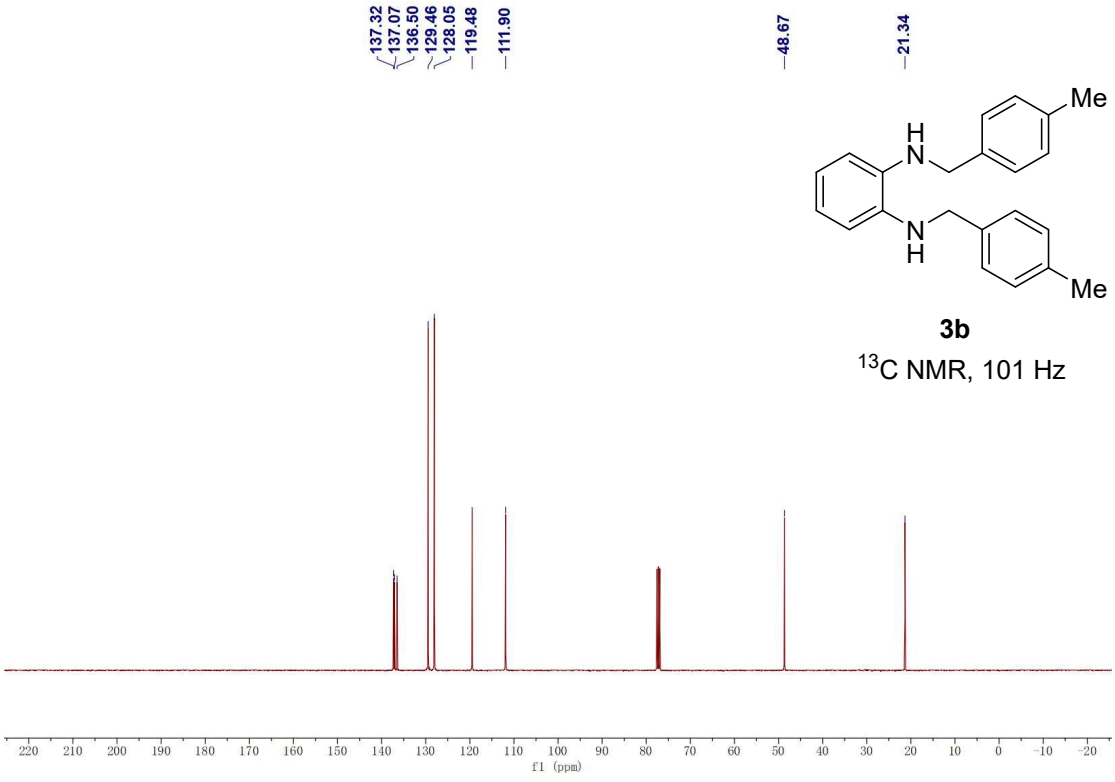
3a

^{13}C NMR, 101 Hz



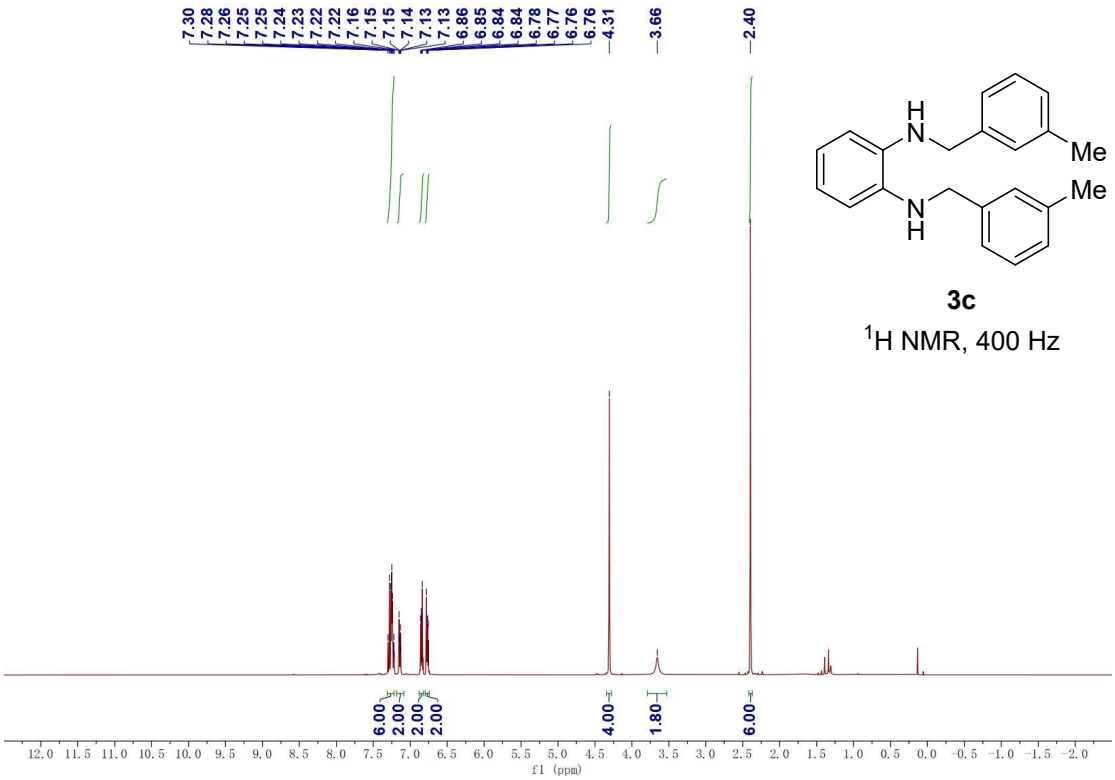
3b

^{1}H NMR, 400 Hz



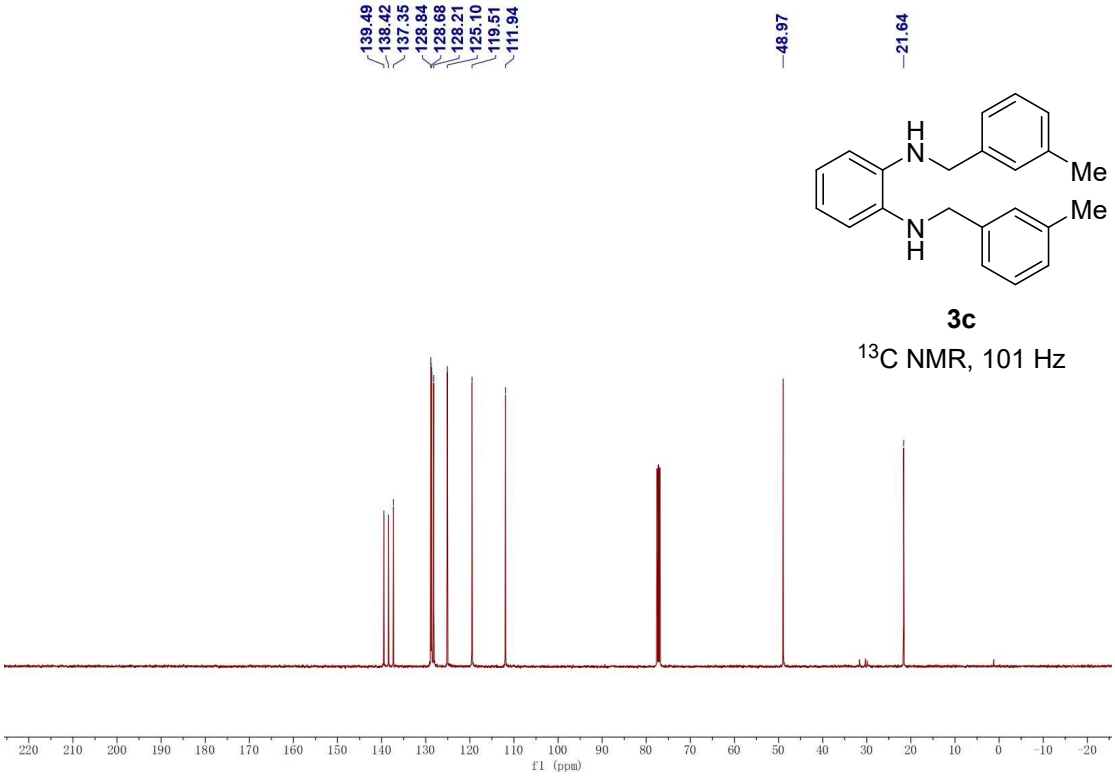
3b

^{13}C NMR, 101 Hz



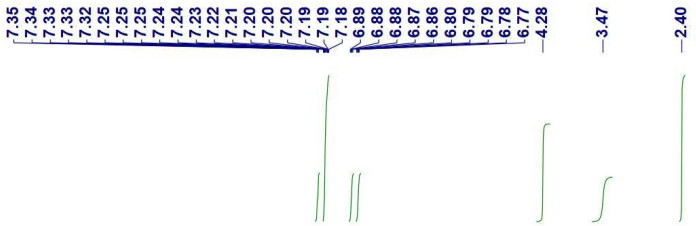
3c

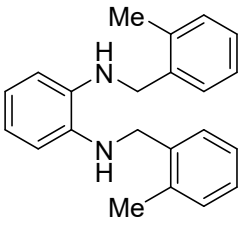
^{1}H NMR, 400 Hz



3c

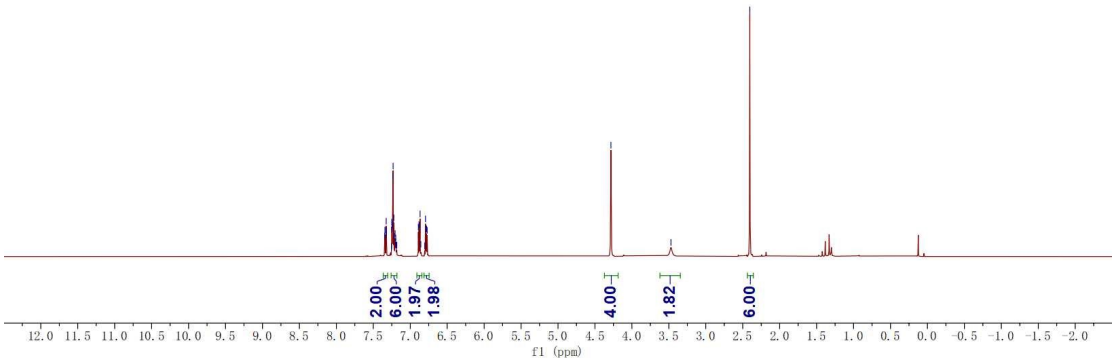
^{13}C NMR, 101 Hz

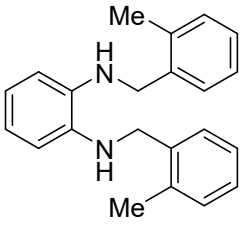




3d

^{1}H NMR, 400 Hz





3d

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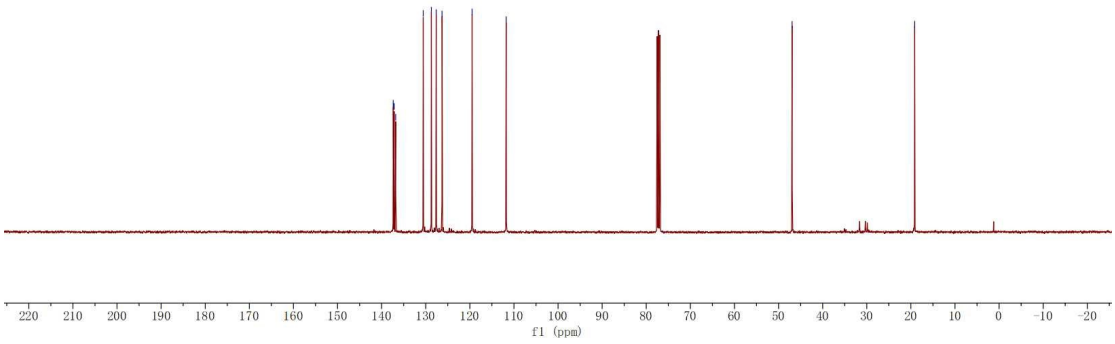
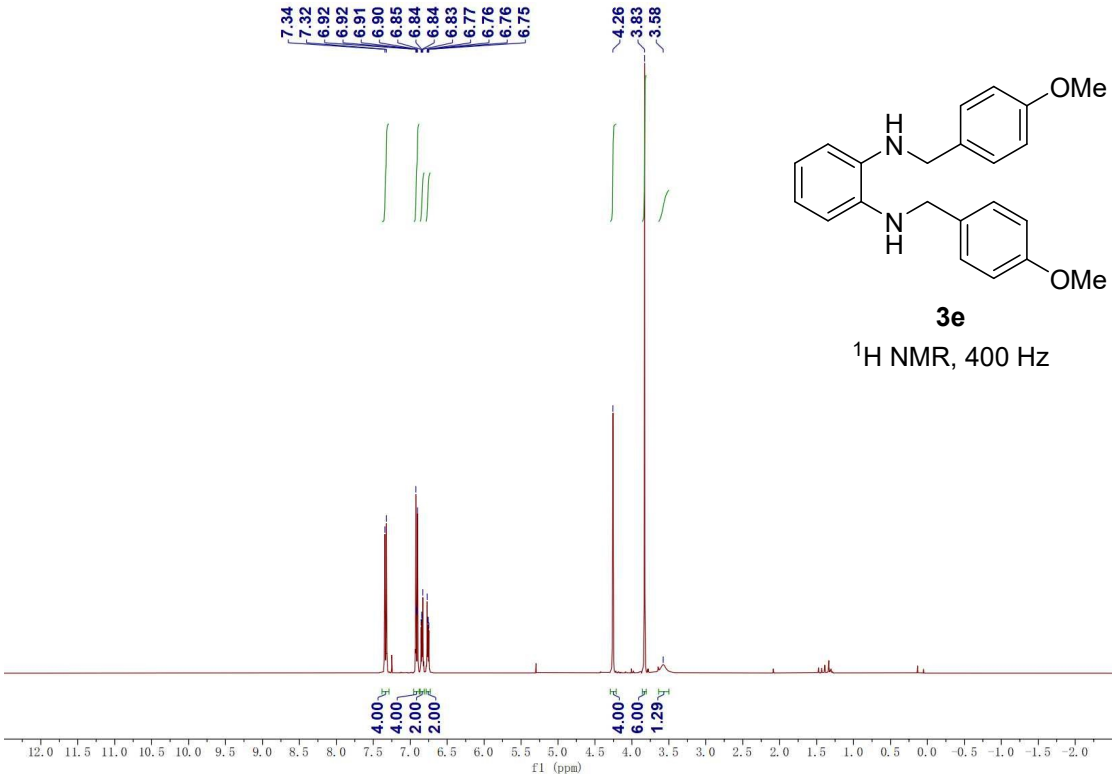
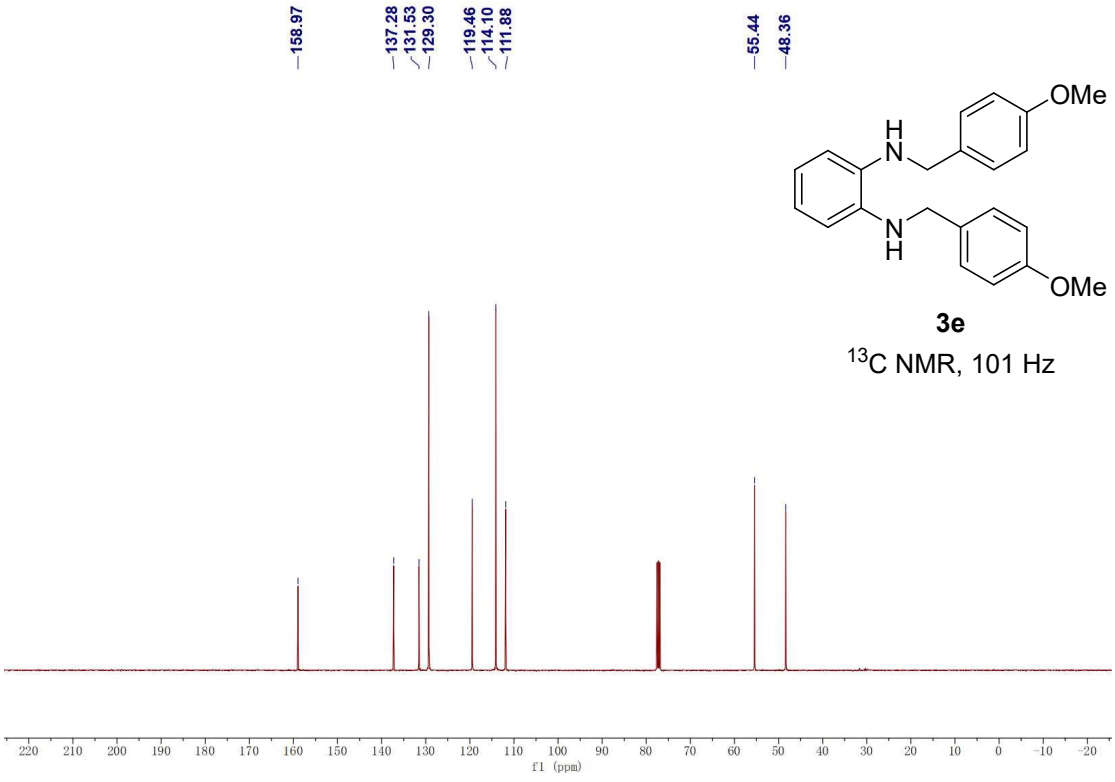




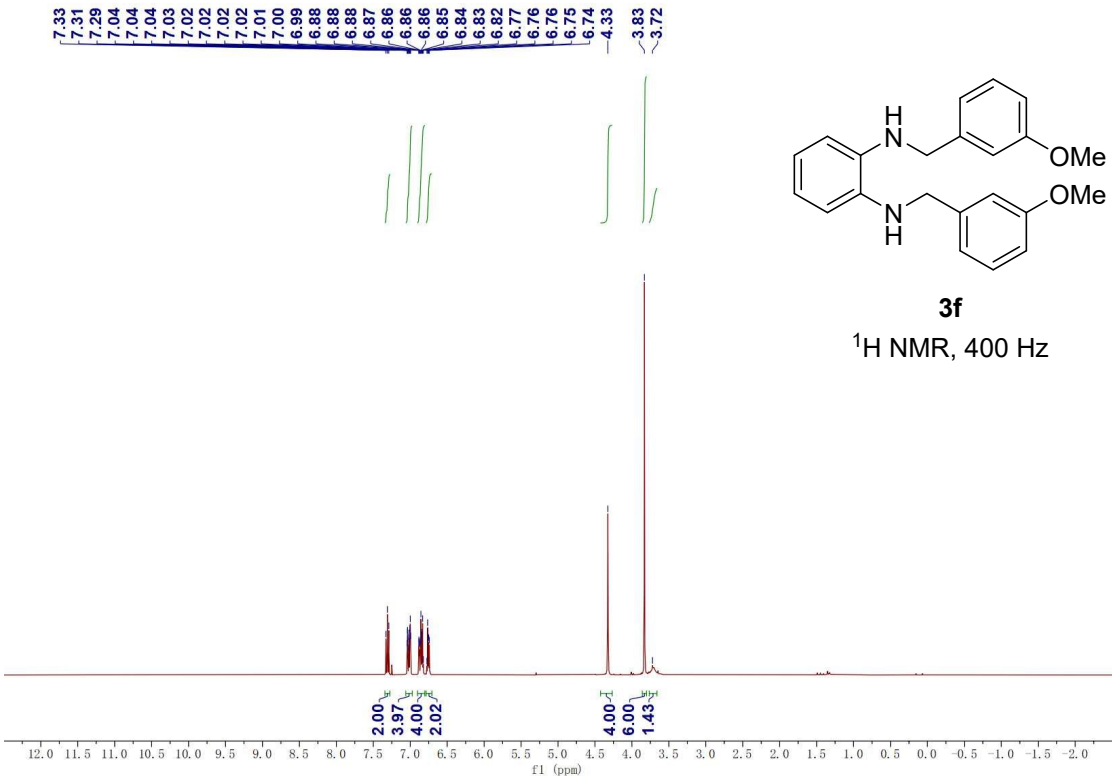
3e

^{1}H NMR, 400 Hz



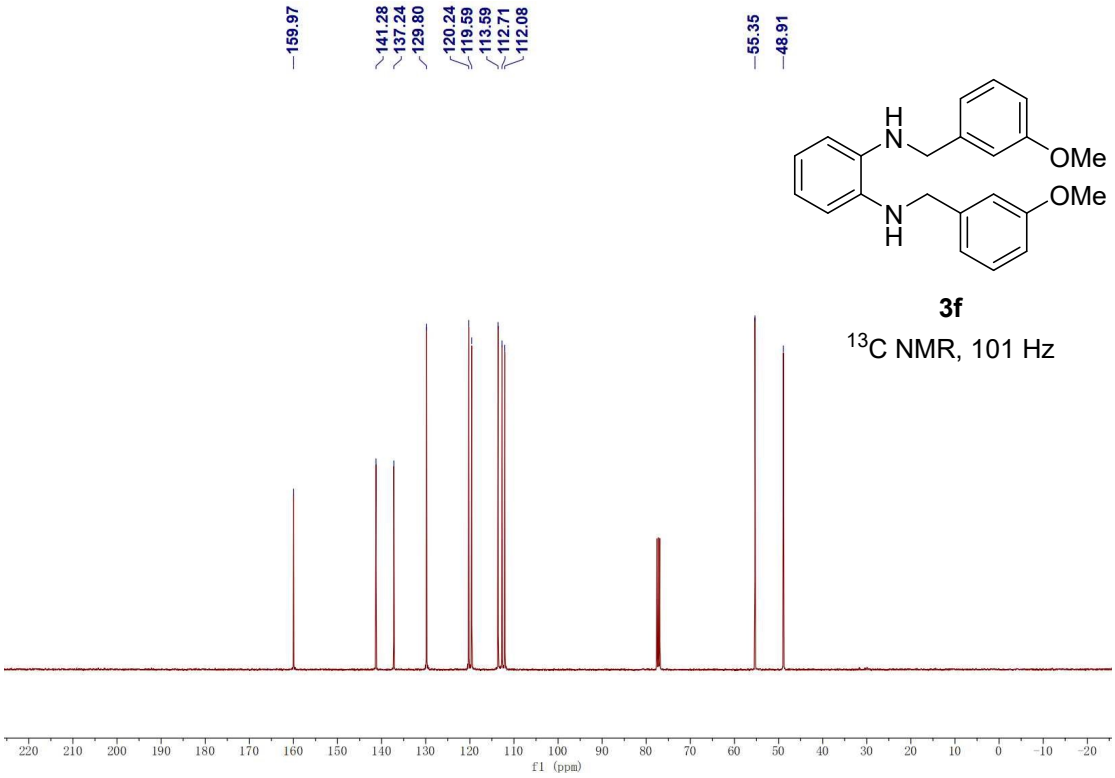
3e

^{13}C NMR, 101 Hz



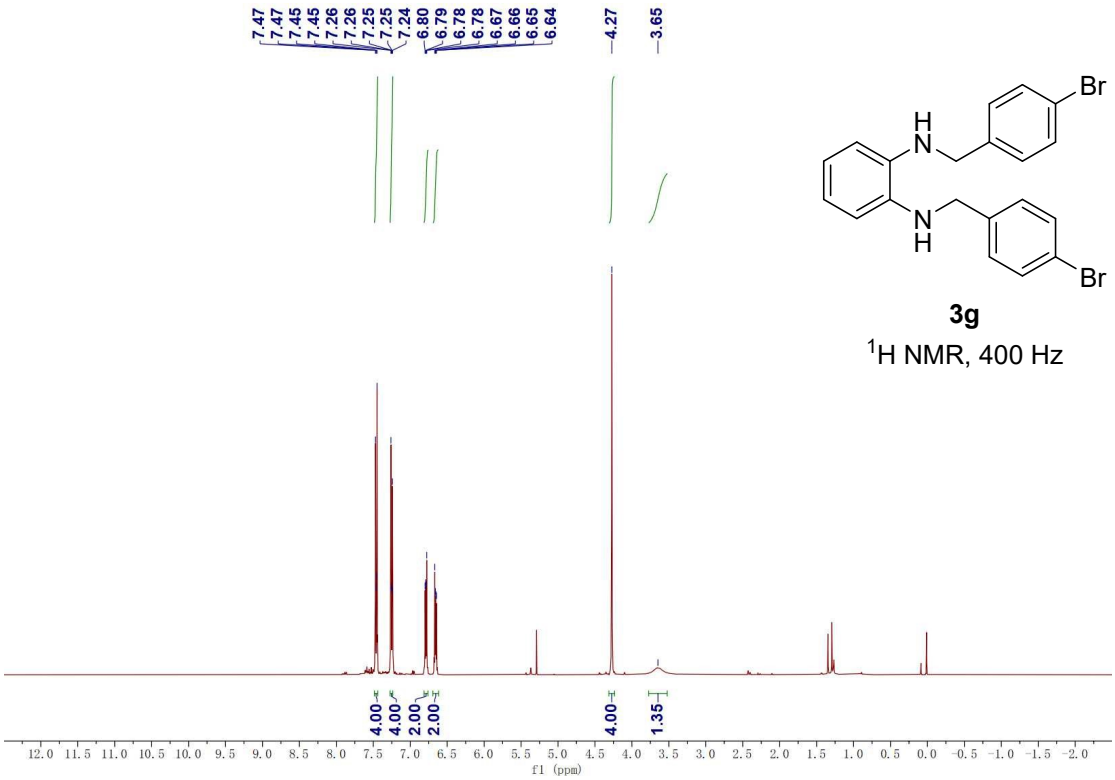
3f

^{1}H NMR, 400 Hz



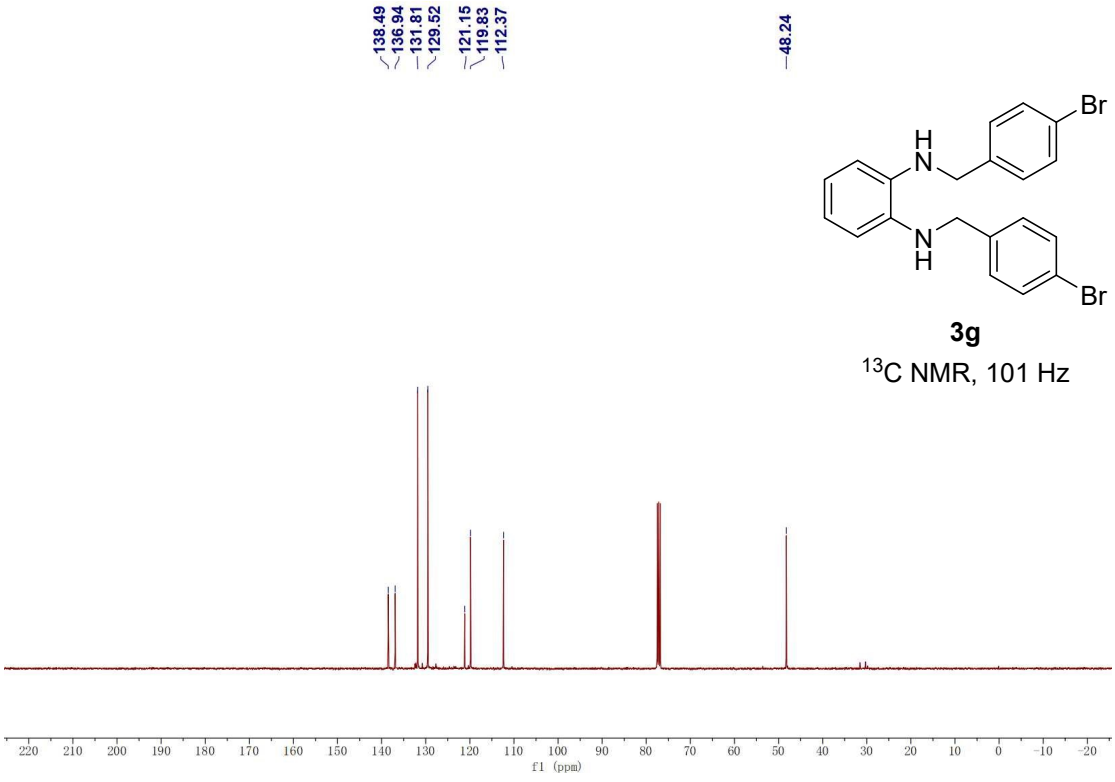
3f

^{13}C NMR, 101 Hz



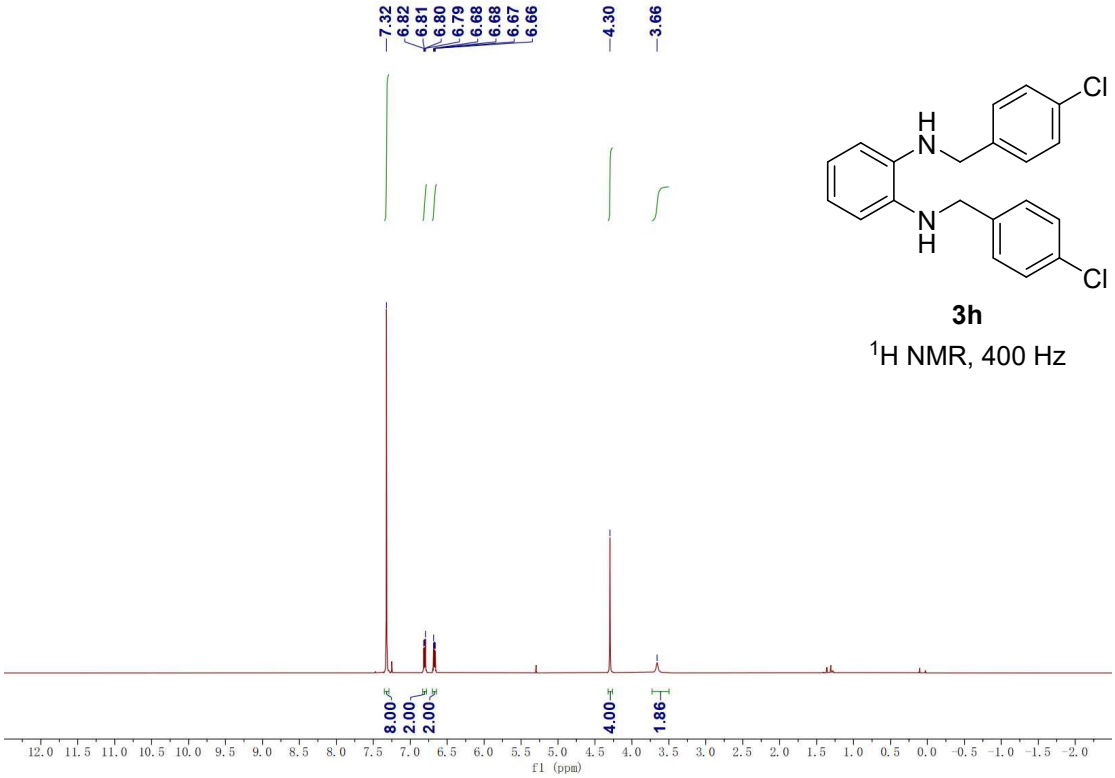
3g

^{1}H NMR, 400 Hz



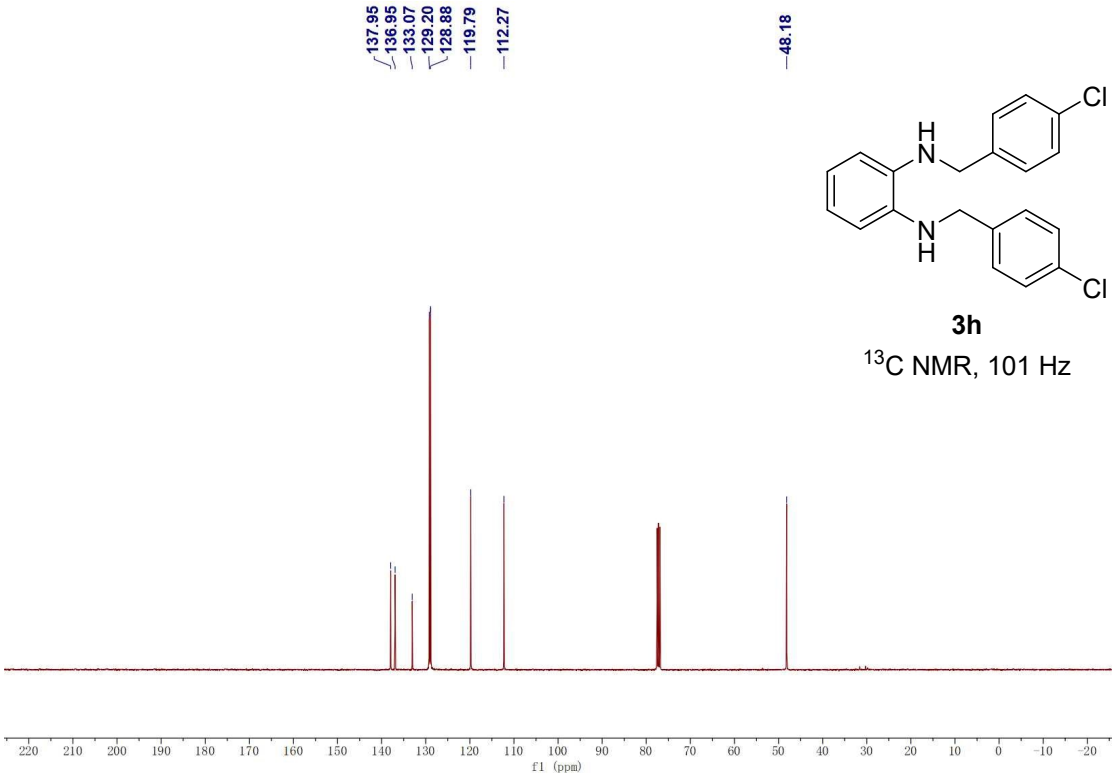
3g

^{13}C NMR, 101 Hz



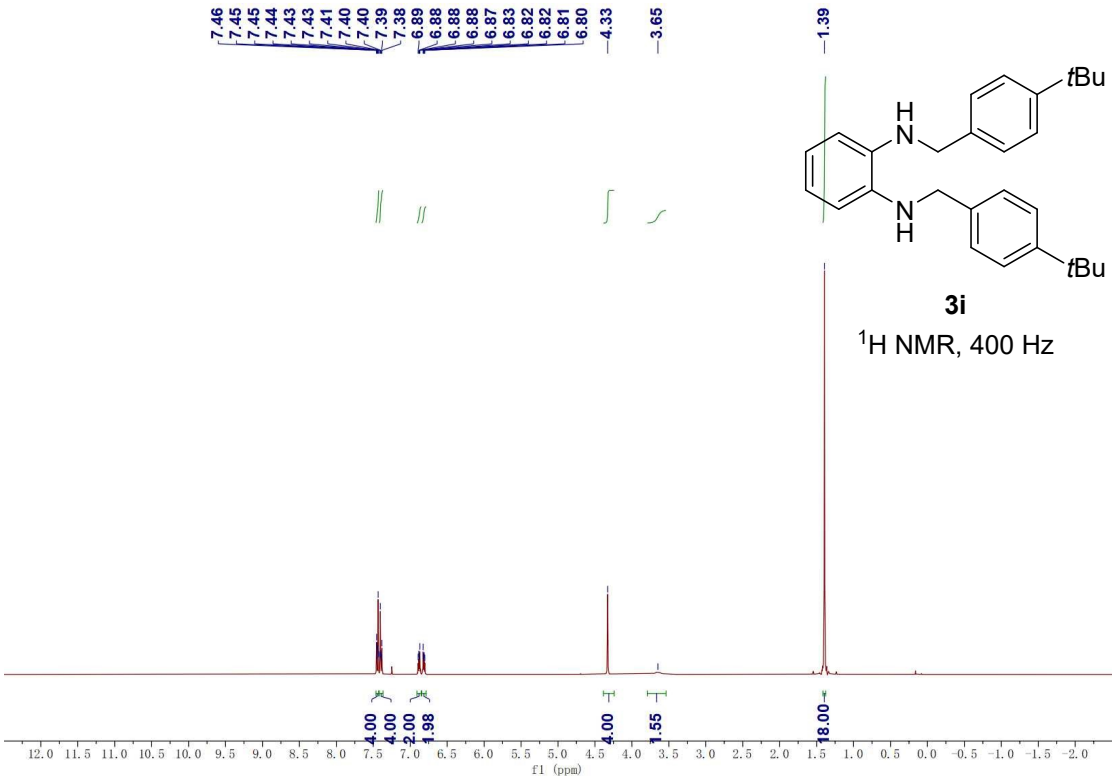
3h

^{1}H NMR, 400 Hz



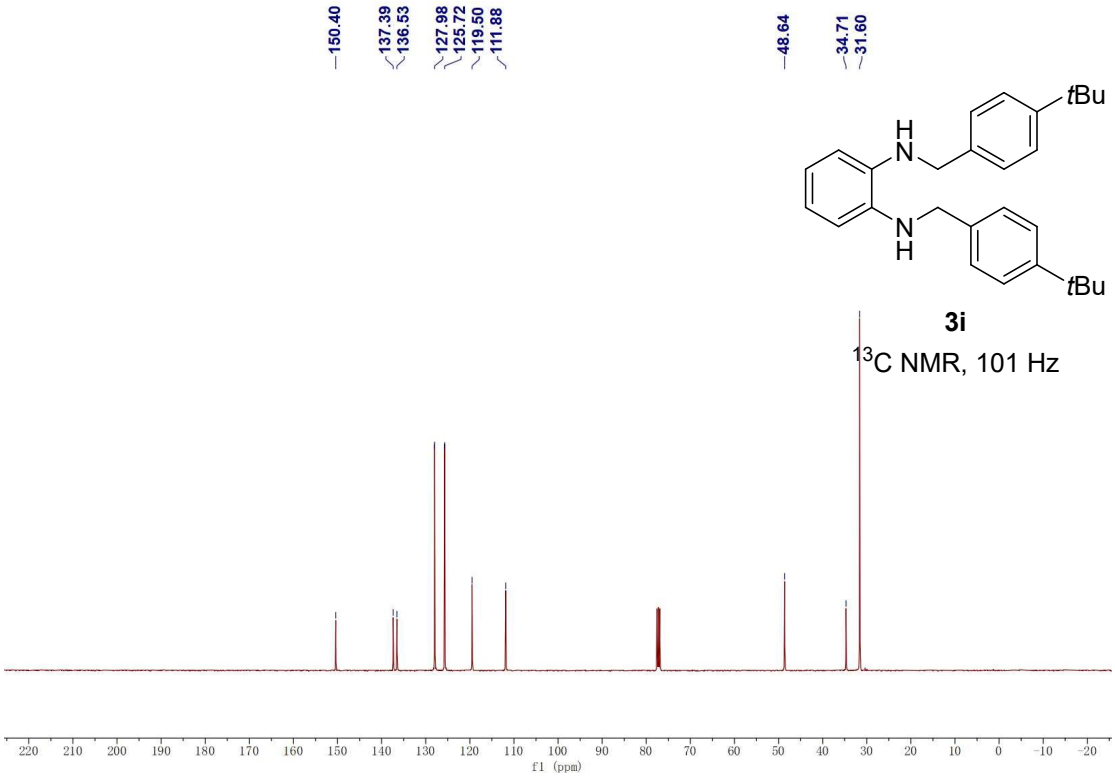
3h

^{13}C NMR, 101 Hz



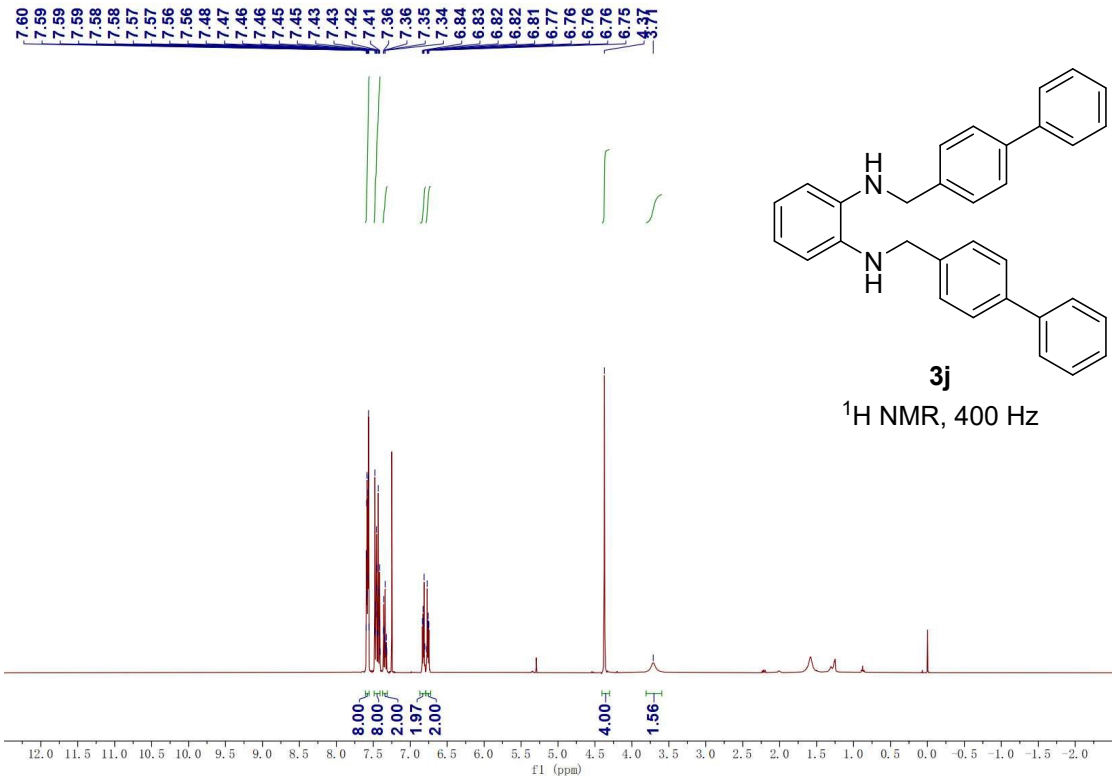
3i

^{1}H NMR, 400 Hz



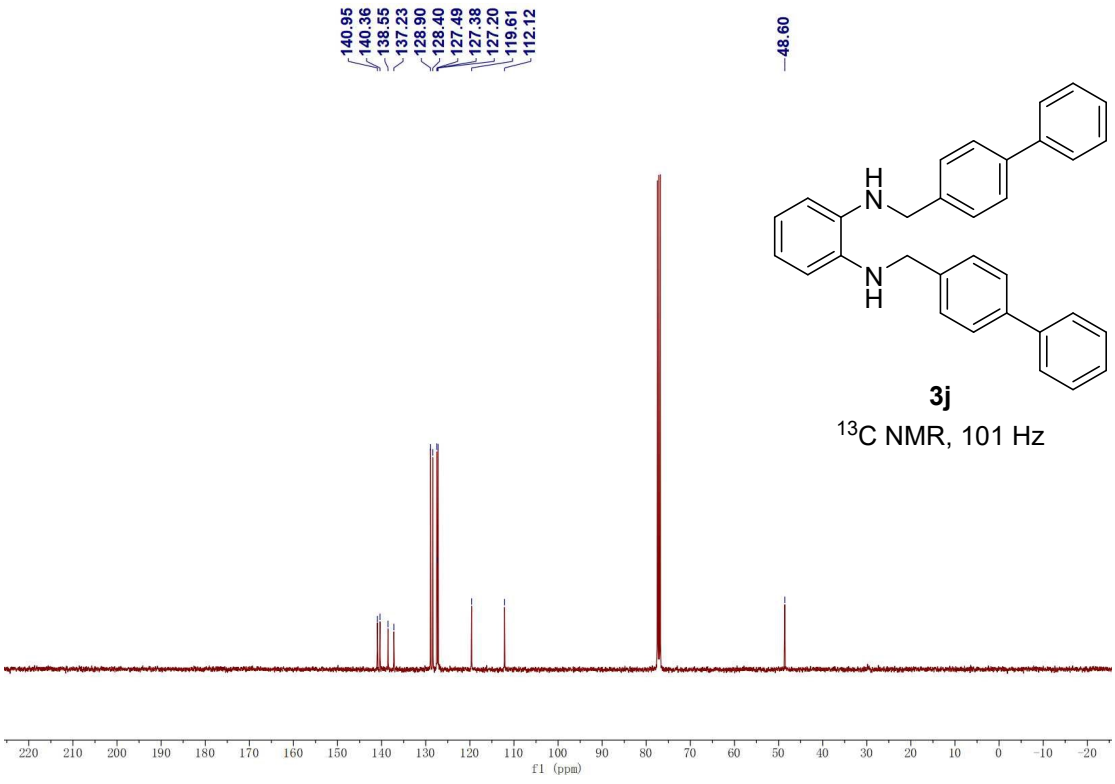
3i

^{13}C NMR, 101 Hz



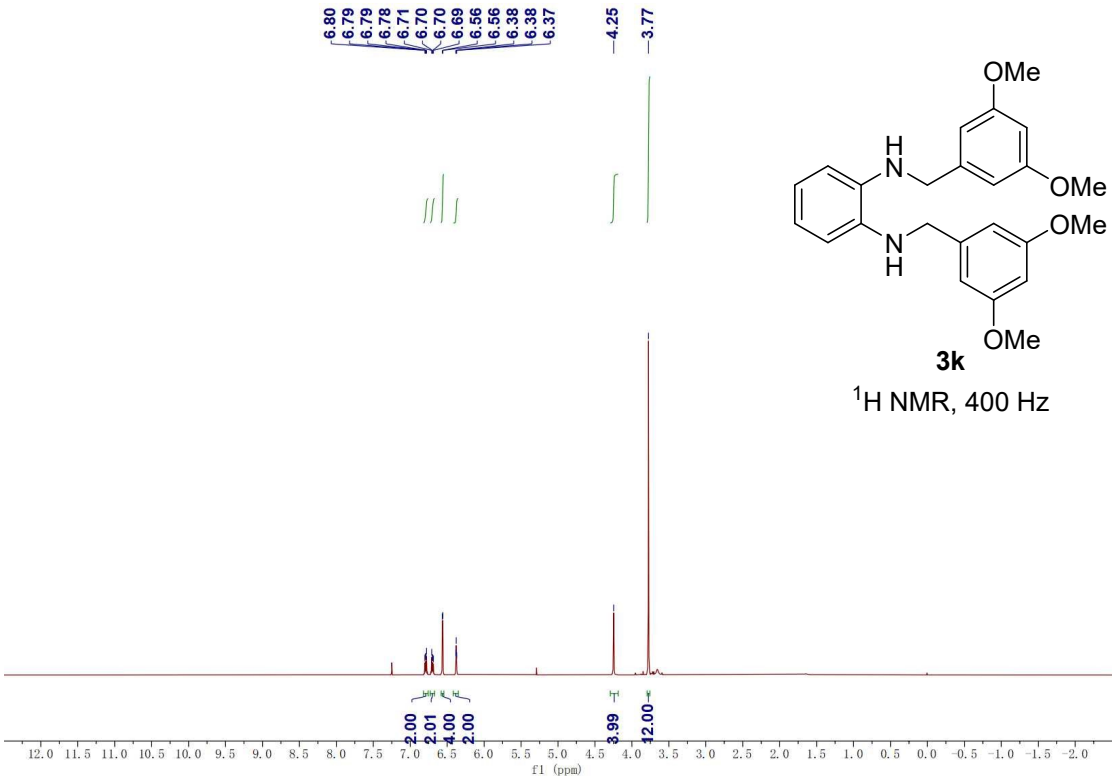
3j

^{1}H NMR, 400 Hz



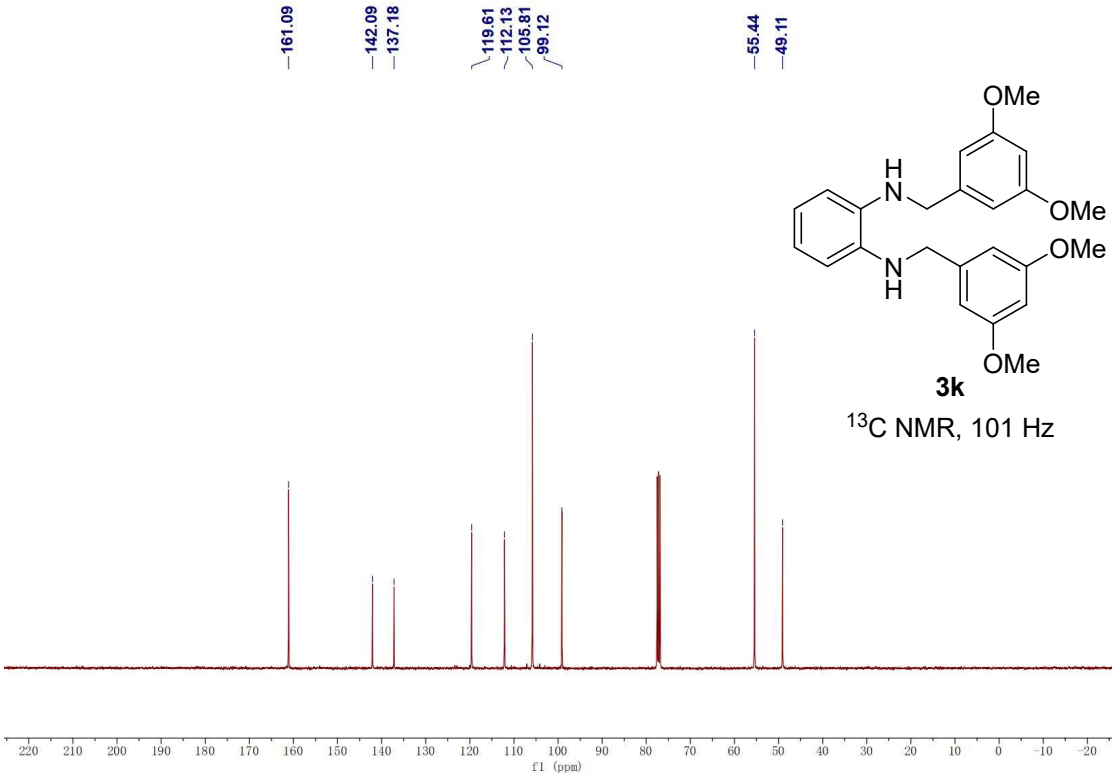
3j

^{13}C NMR, 101 Hz



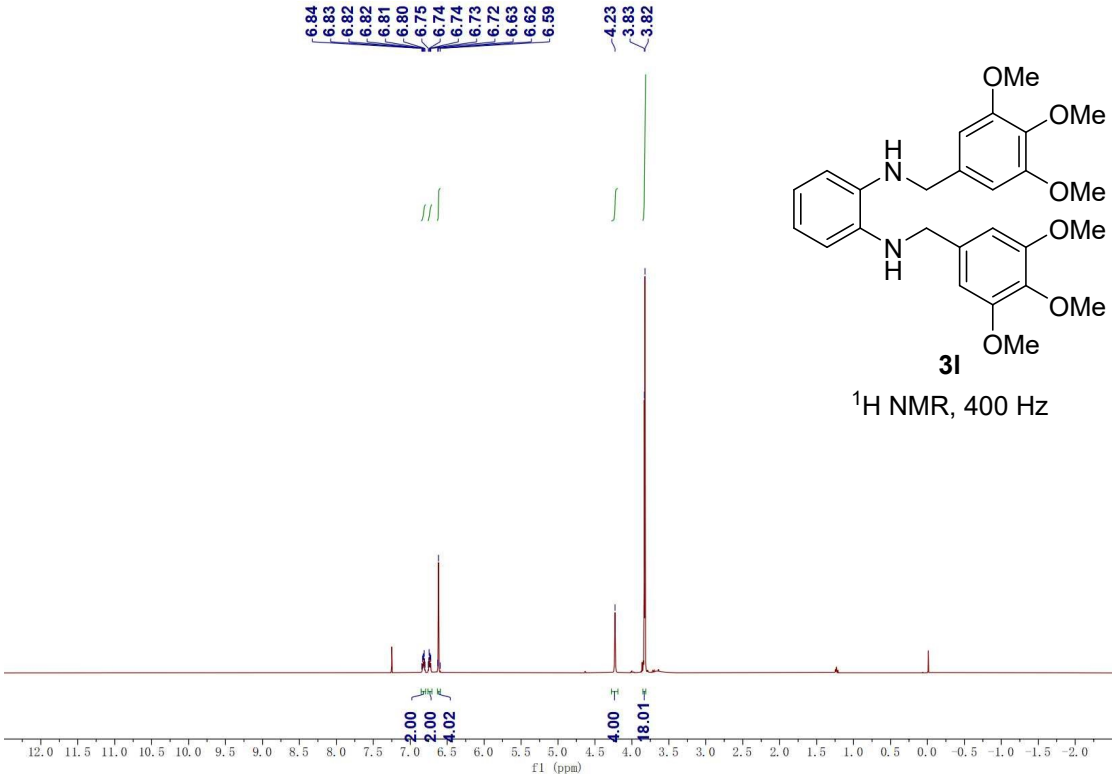
3k

^{1}H NMR, 400 Hz



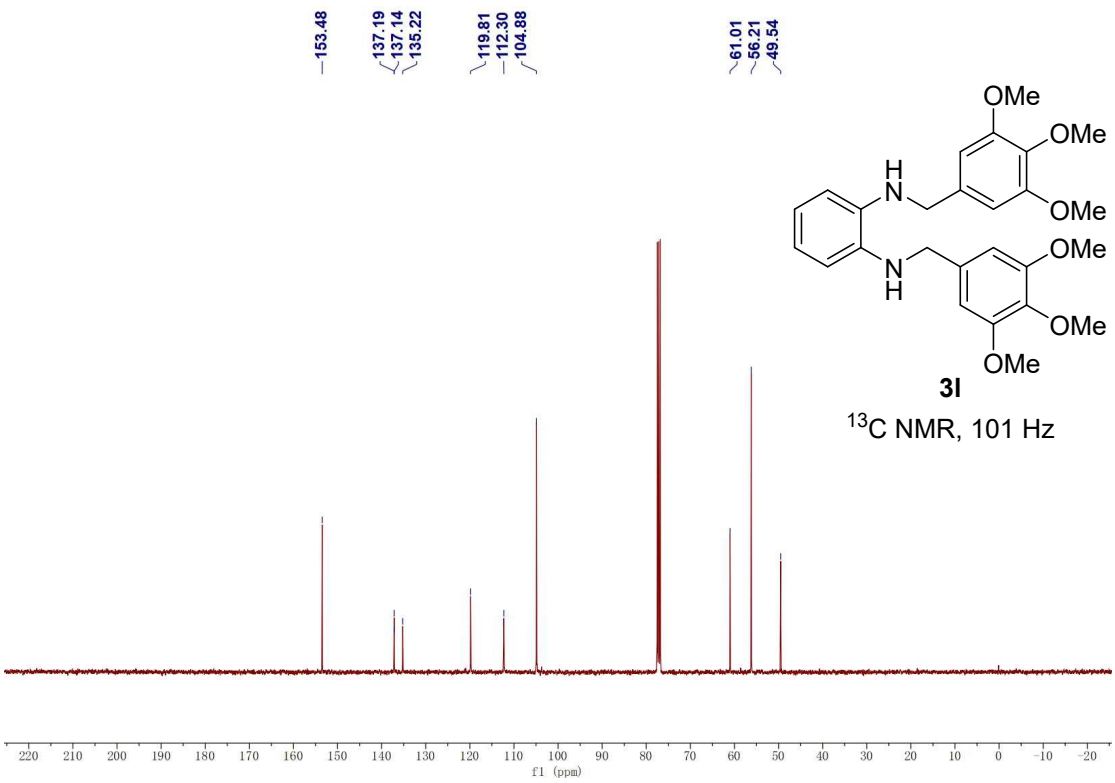
3k

^{13}C NMR, 101 Hz



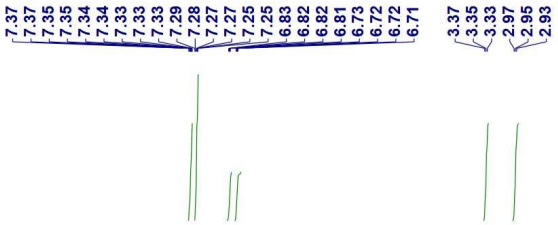
3l

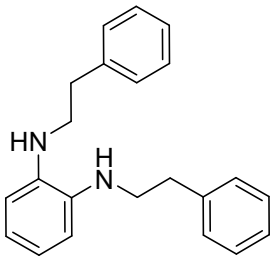
^{1}H NMR, 400 Hz



3l

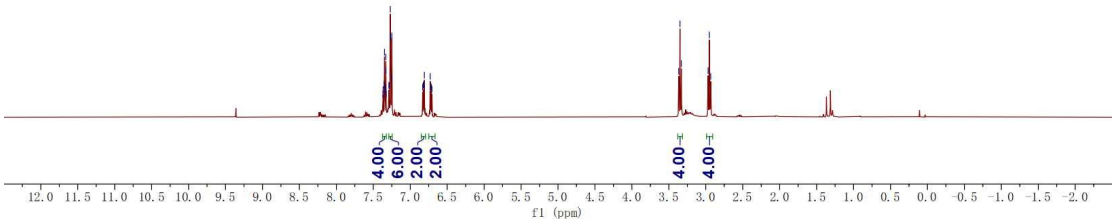
^{13}C NMR, 101 Hz

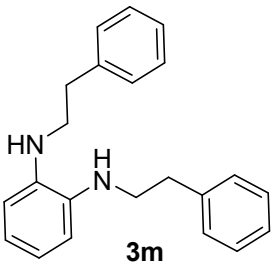




3m

^{1}H NMR, 400 Hz





3m

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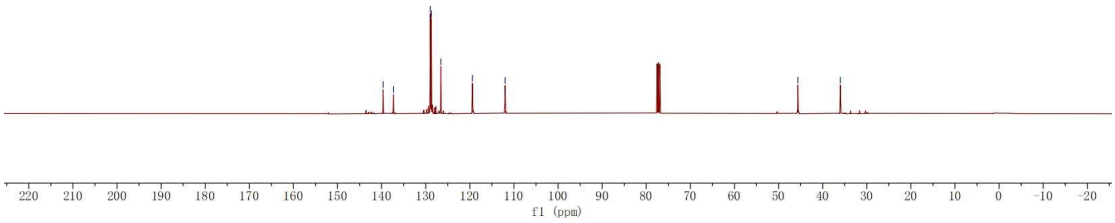

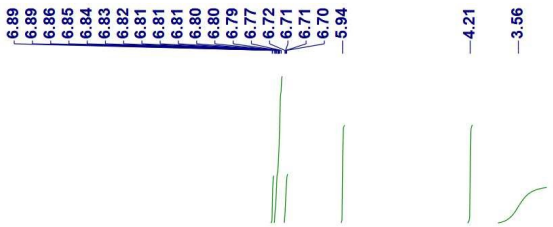

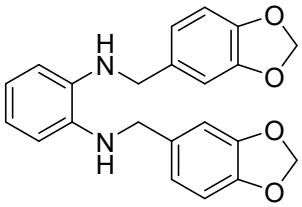

3n

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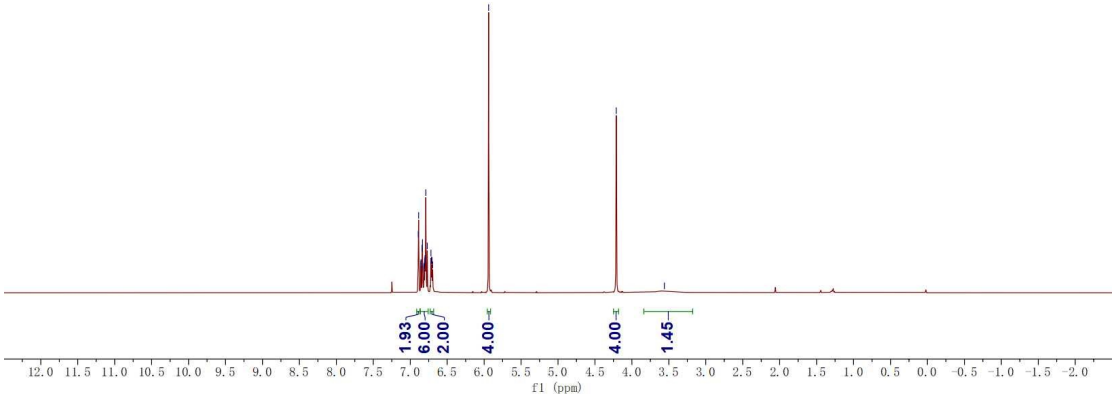
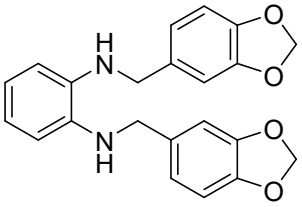
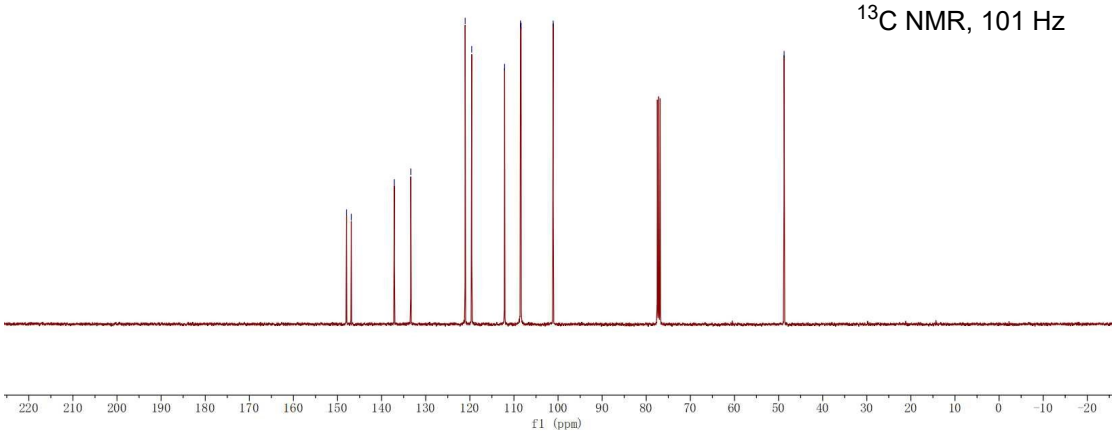
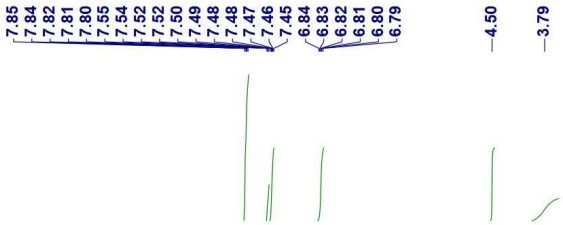


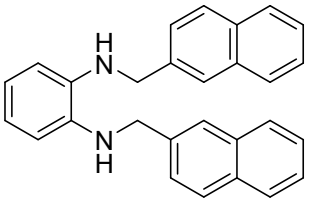


3n

^{13}C NMR, 101 Hz







3o

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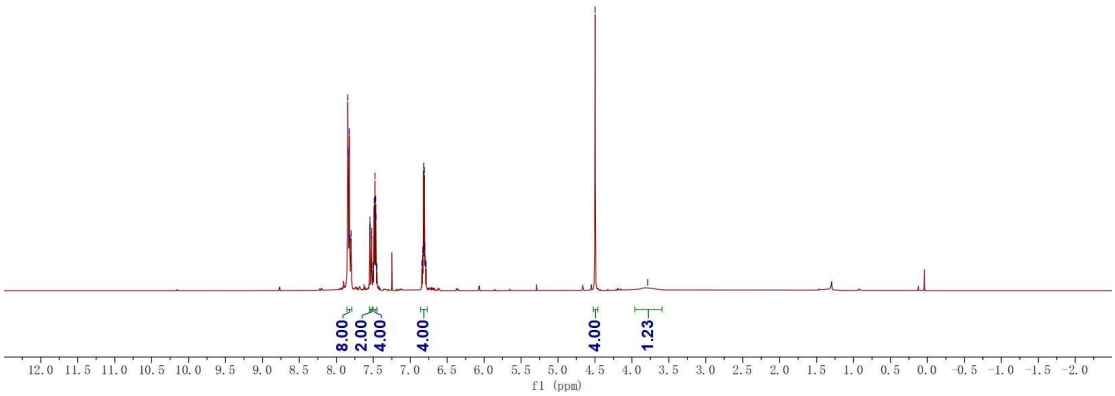

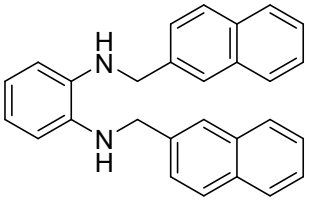

3o

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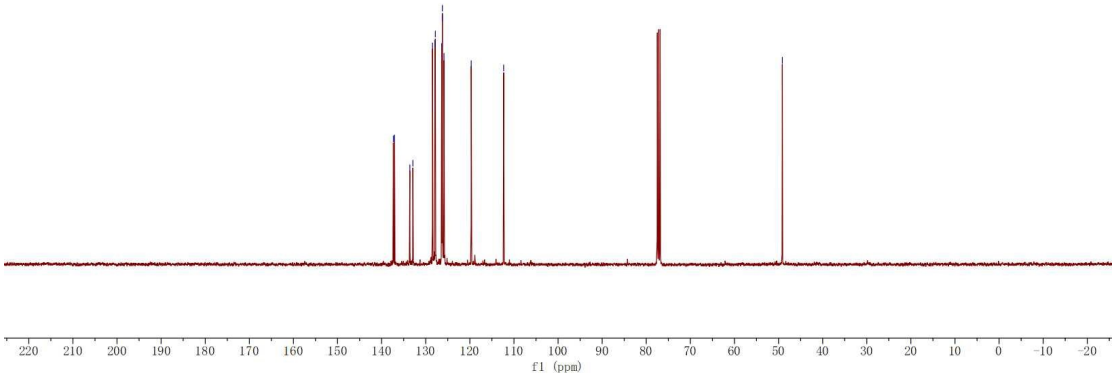
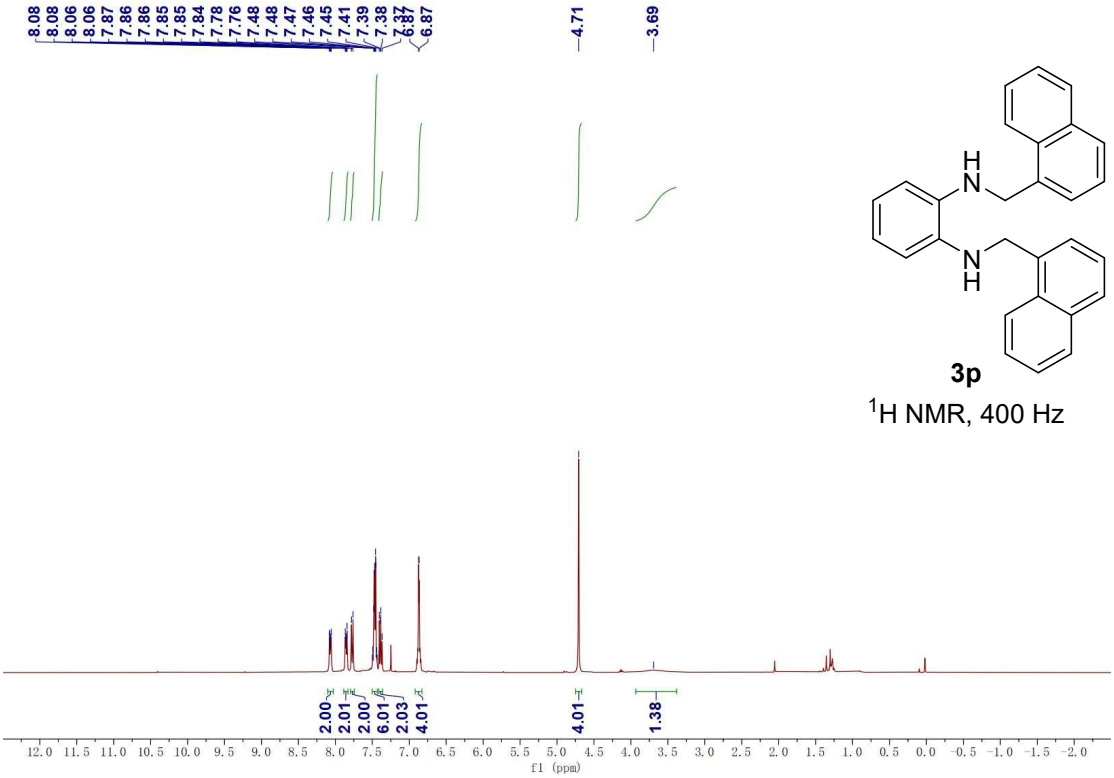
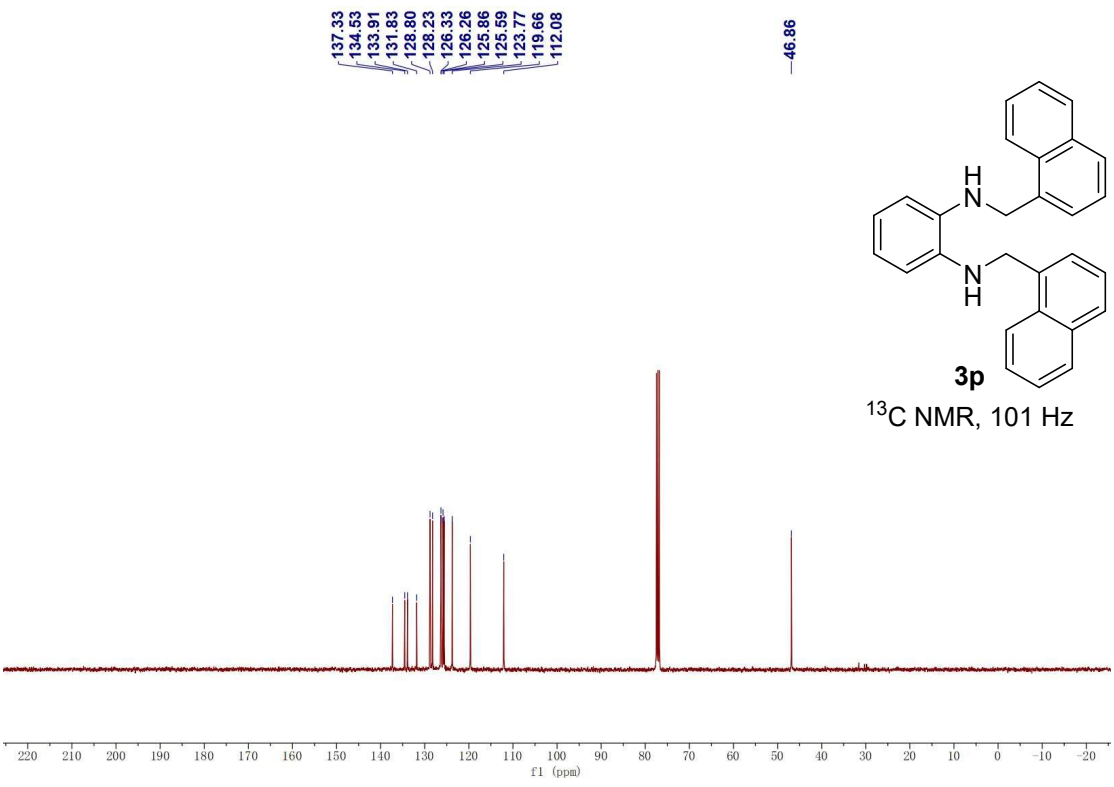




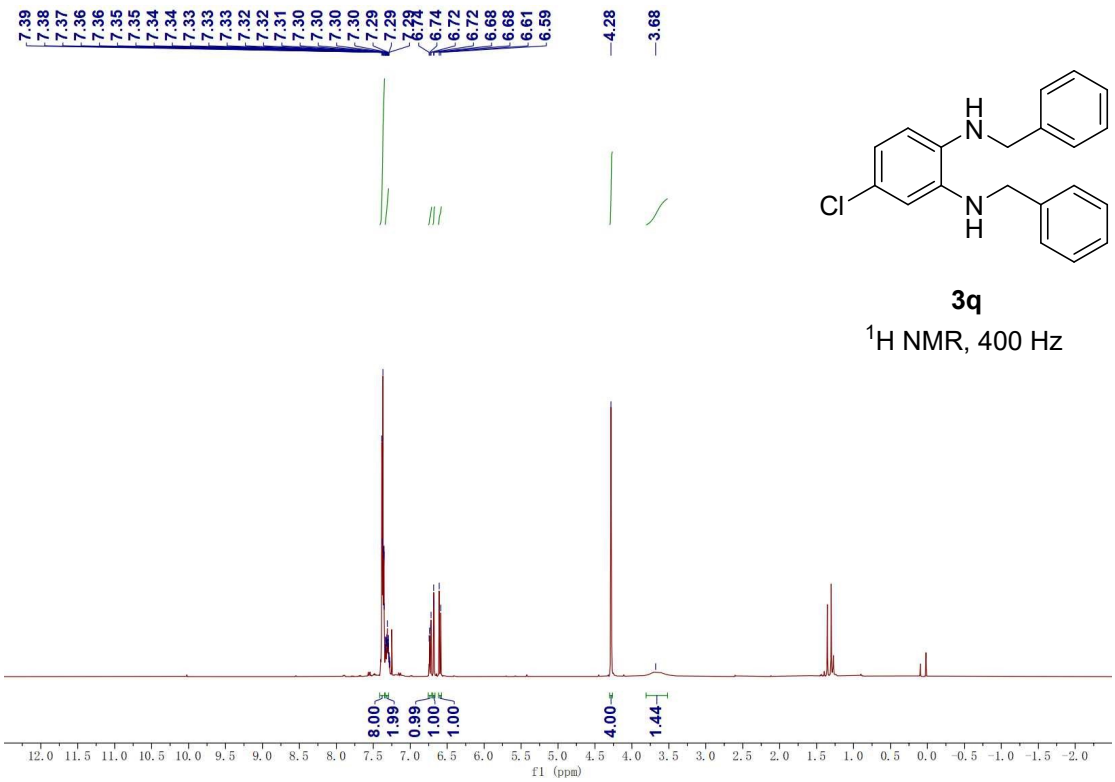
3p

^{1}H NMR, 400 Hz



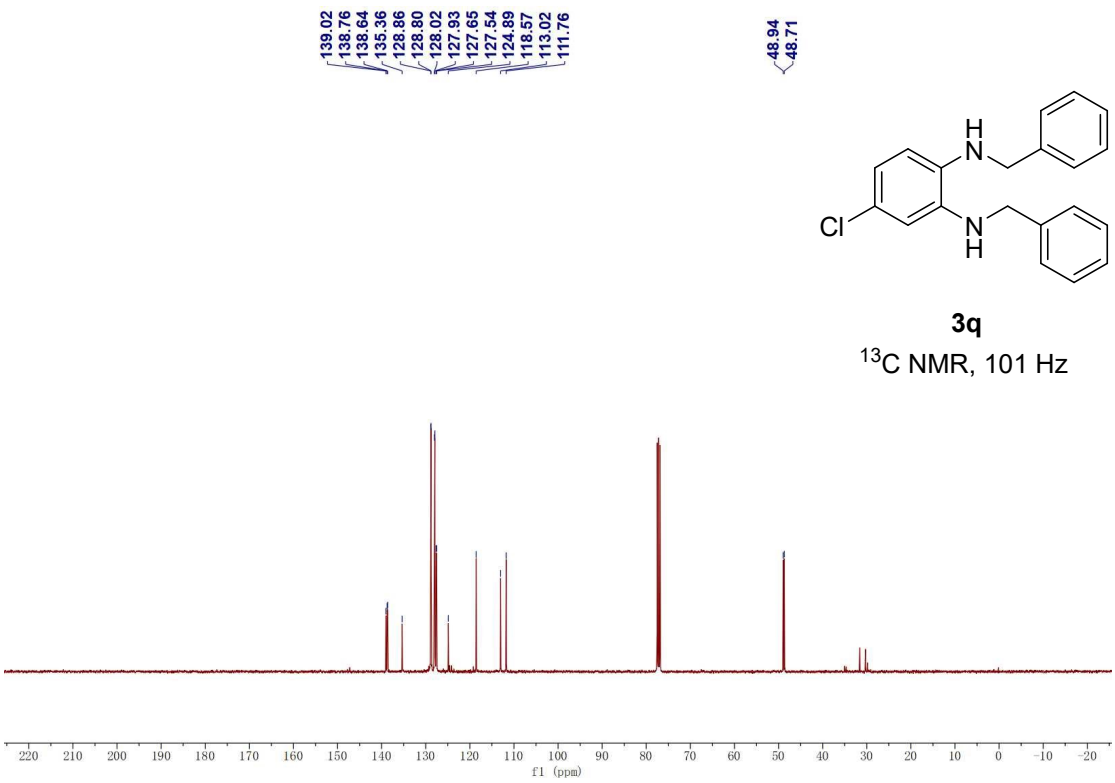
3p

^{13}C NMR, 101 Hz



3q

^{1}H NMR, 400 Hz



3q

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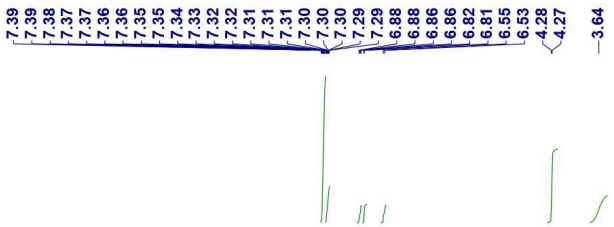
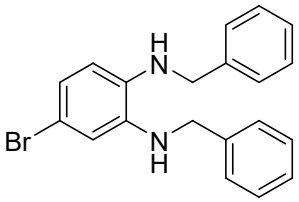
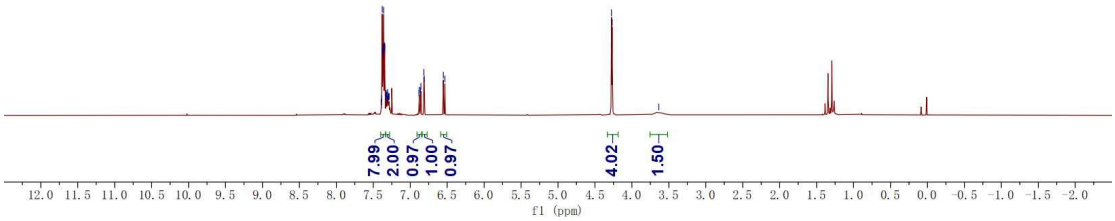
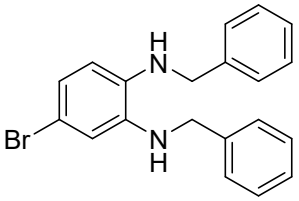




3r

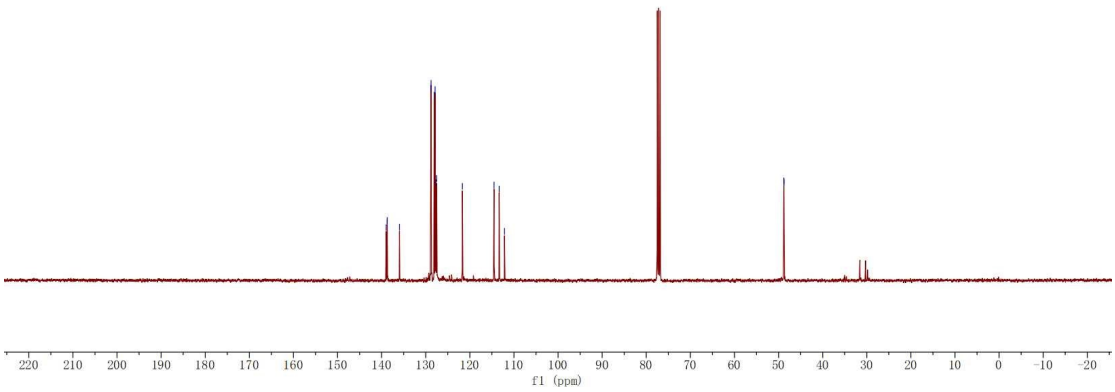
^{1}H NMR, 400 Hz

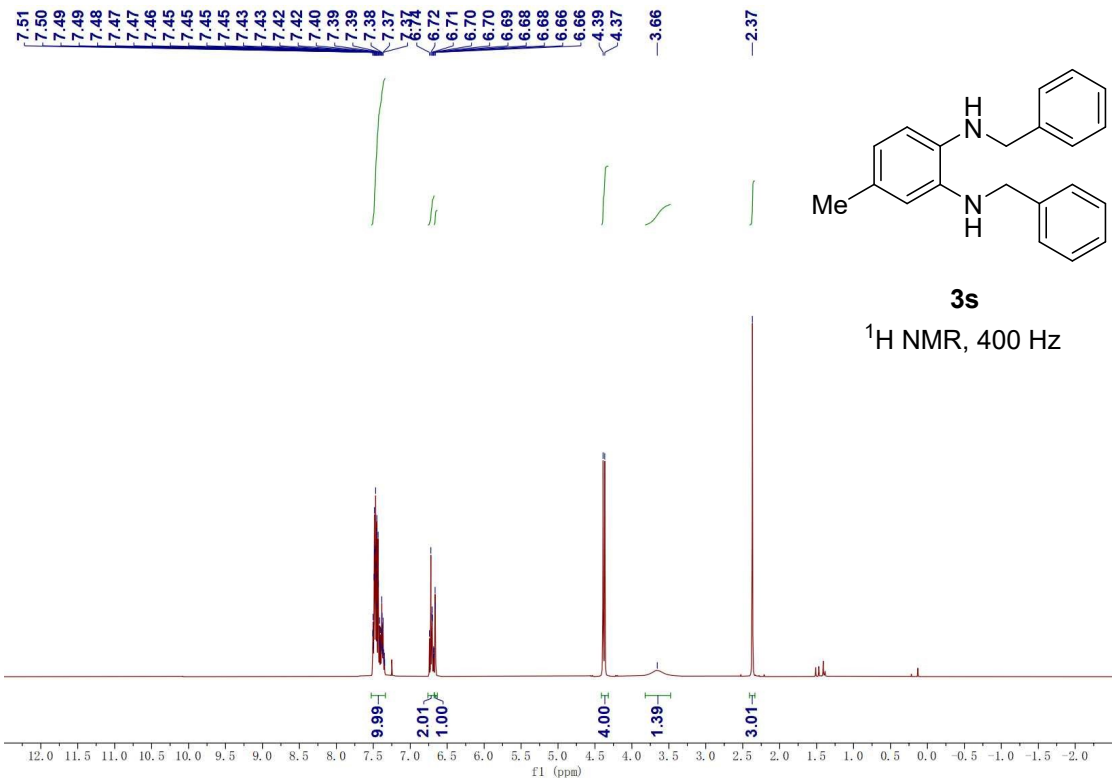




3r

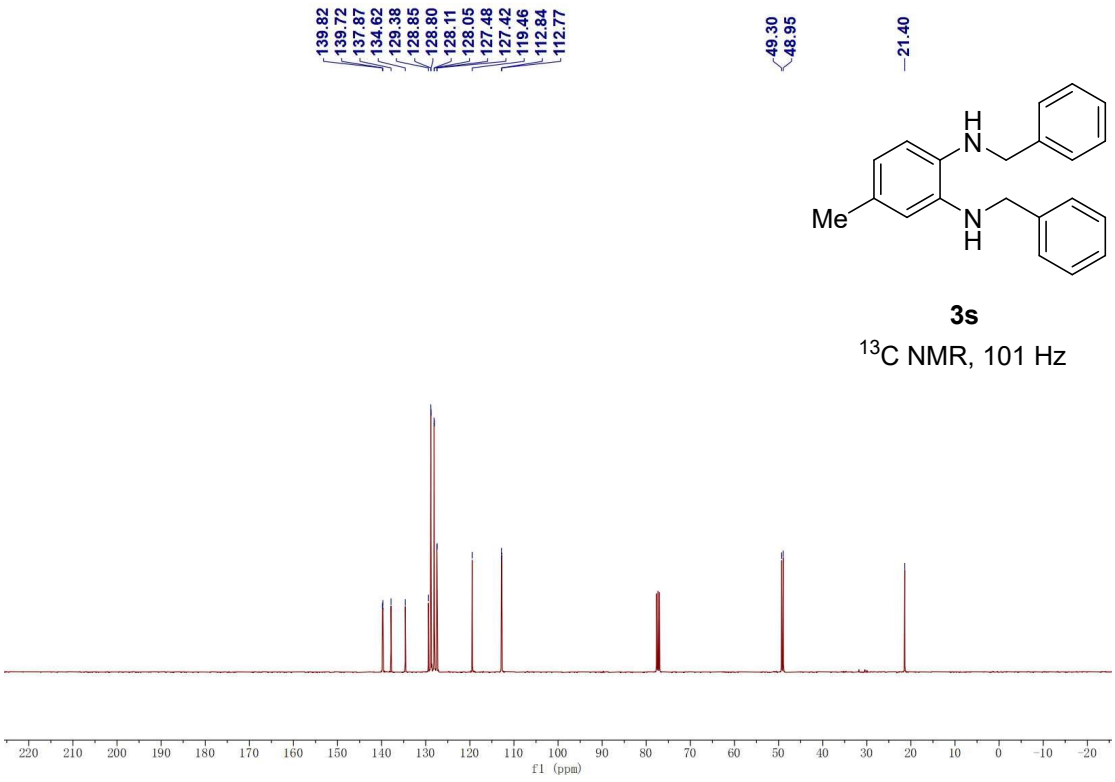
^{13}C NMR, 101 Hz





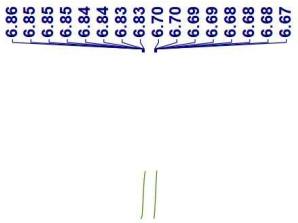
3s

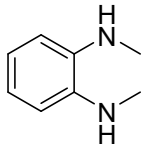
^{1}H NMR, 400 Hz



3s

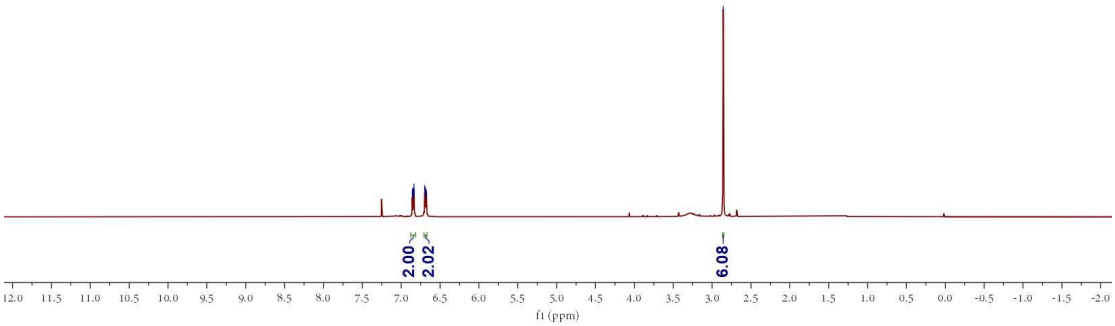
^{13}C NMR, 101 Hz

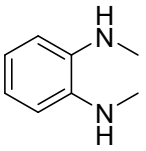




3t

^{1}H NMR, 400 Hz





3t

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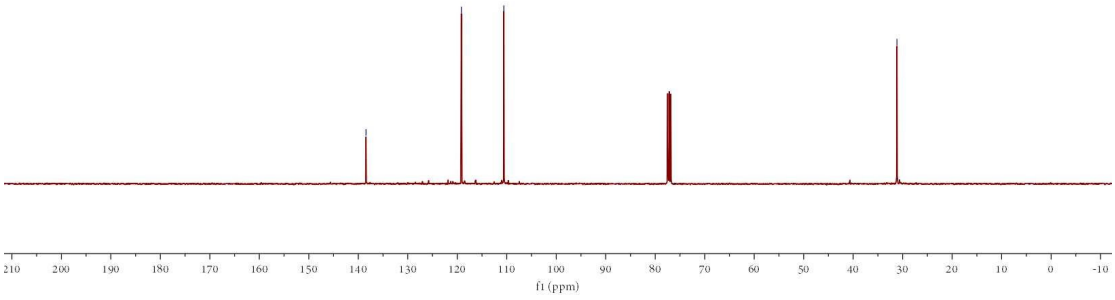
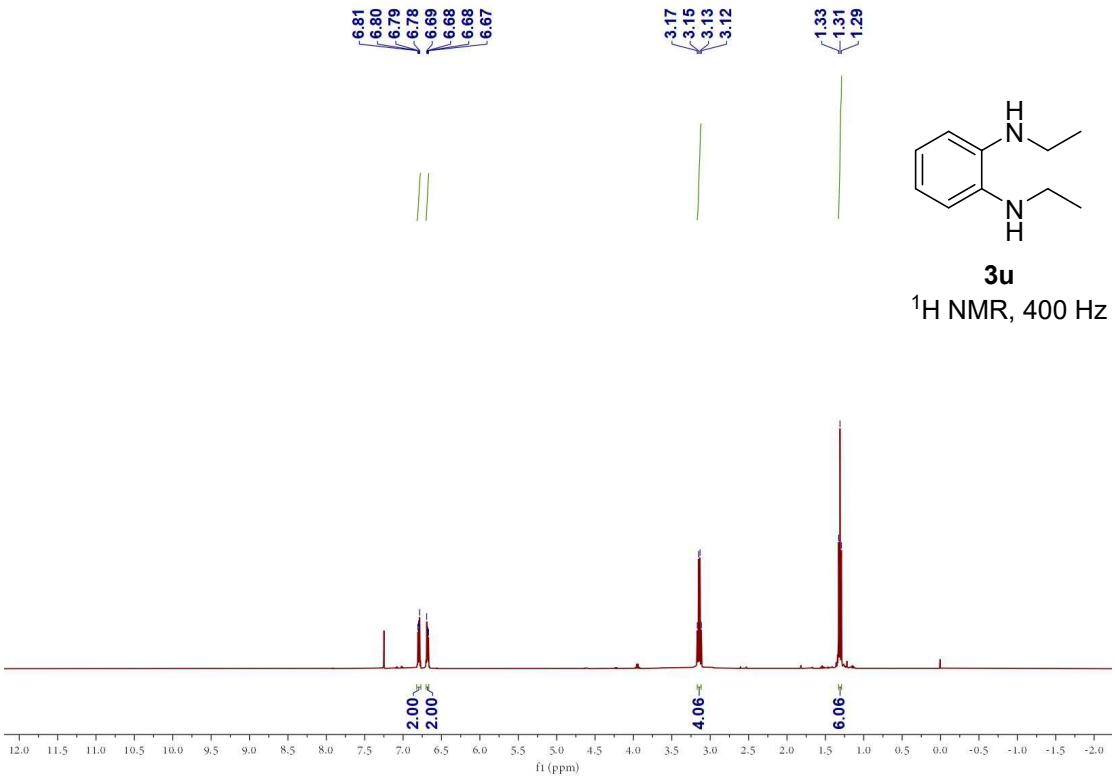
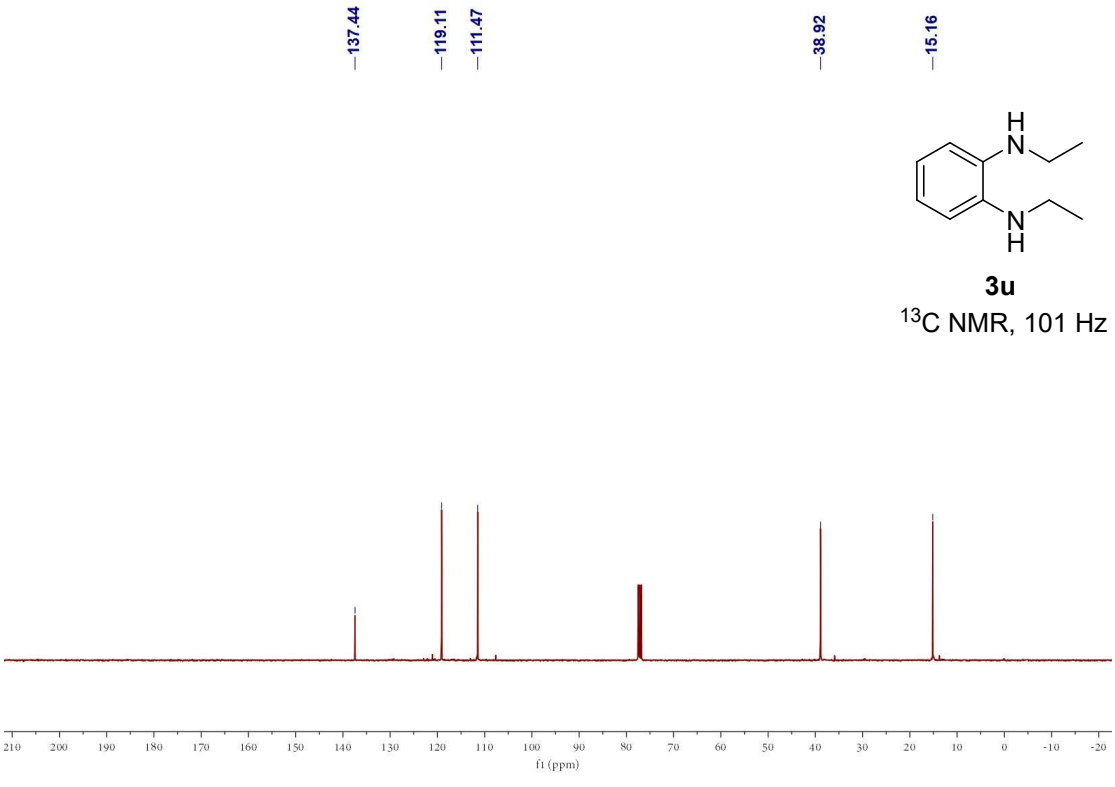




3u

^{1}H NMR, 400 Hz



3u

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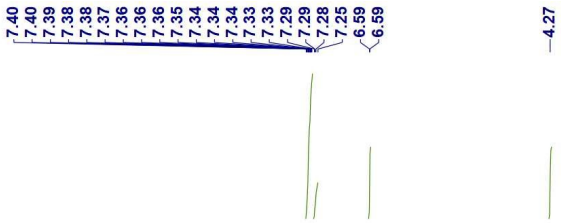
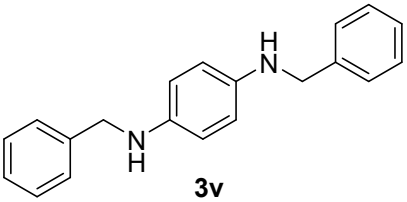
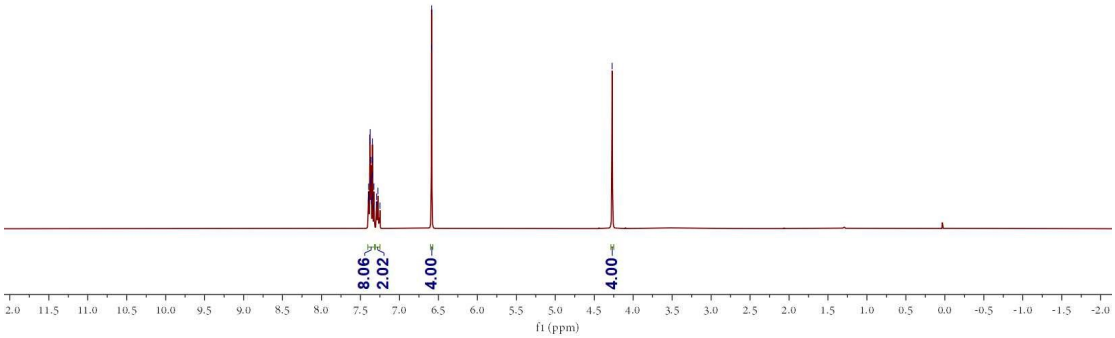
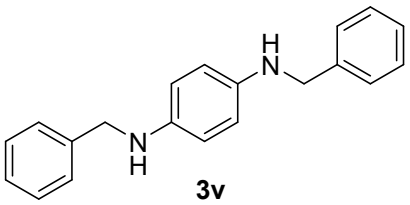




3v

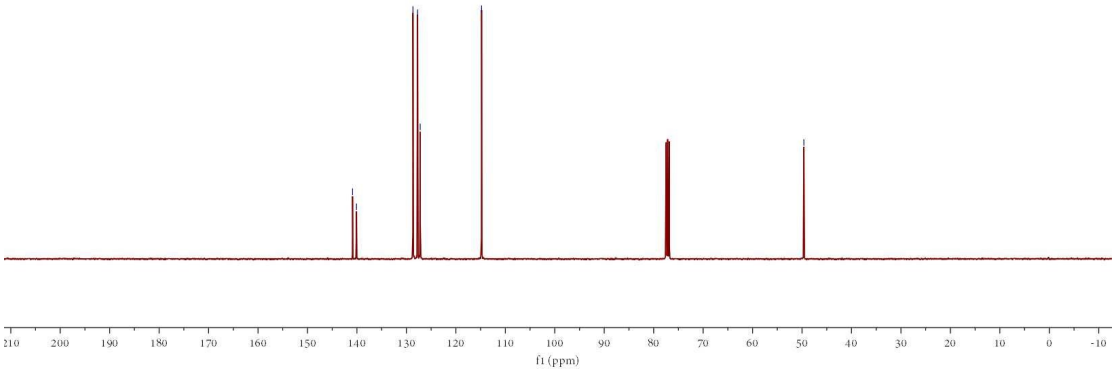
^{1}H NMR, 400 Hz

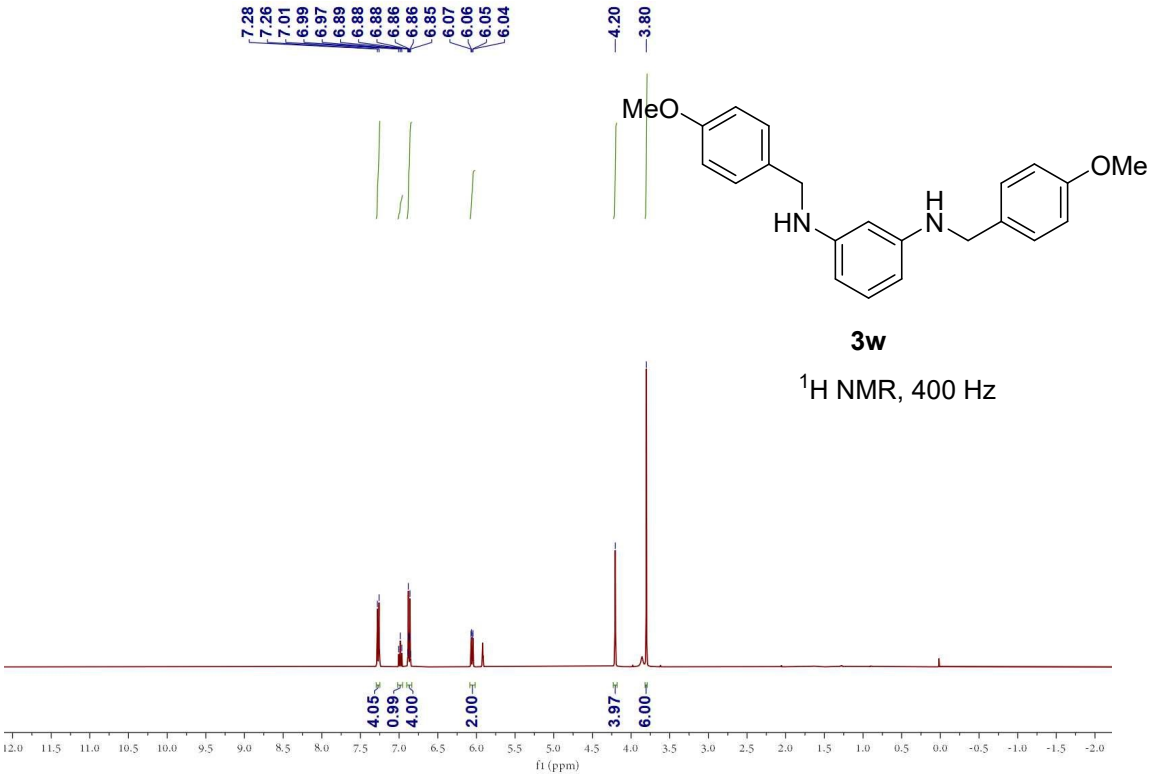




3v

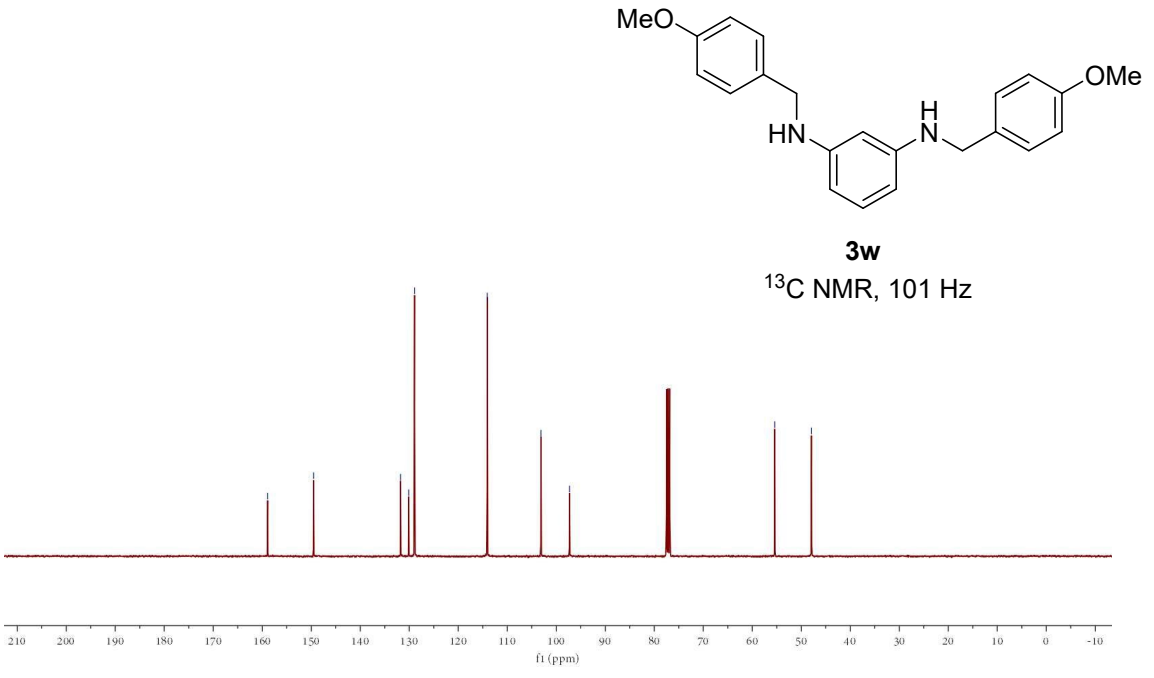
^{13}C NMR, 101 Hz





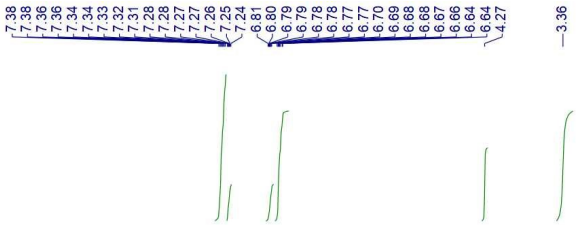
3w

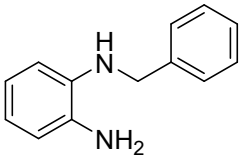
^{1}H NMR, 400 Hz



3w

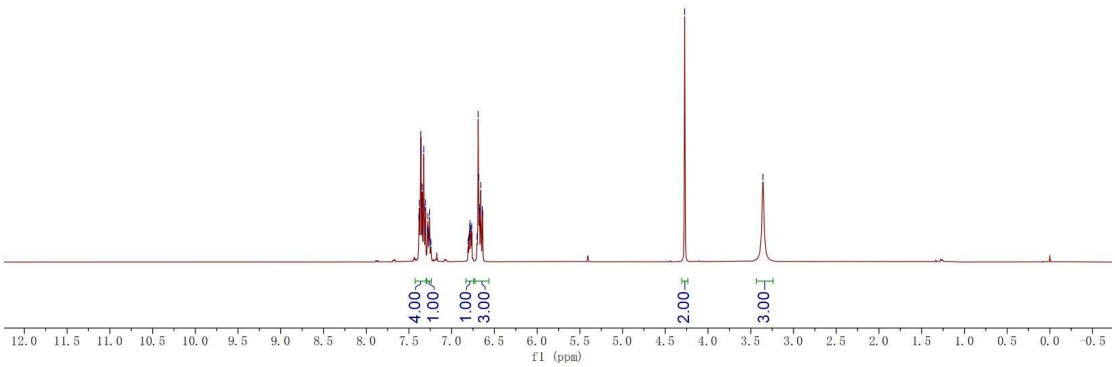
^{13}C NMR, 101 Hz

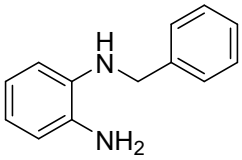




4a

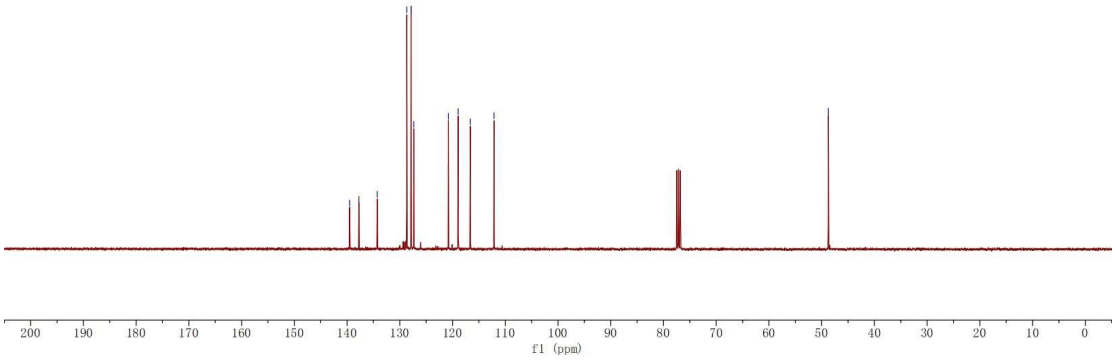
^{1}H NMR, 400 Hz

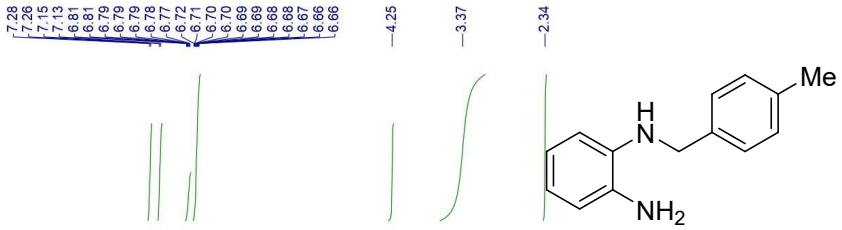




4a

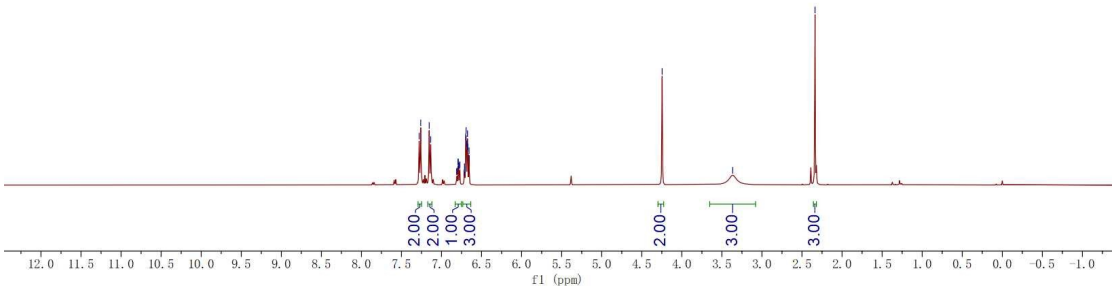
^{13}C NMR, 101 Hz

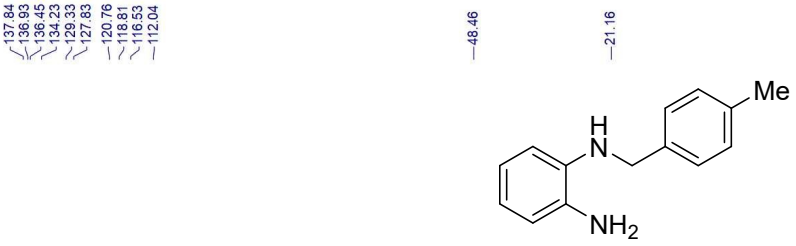




4b

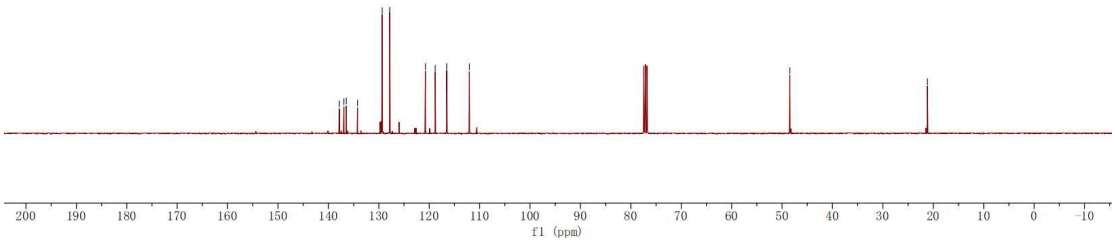
^{1}H NMR, 400 Hz

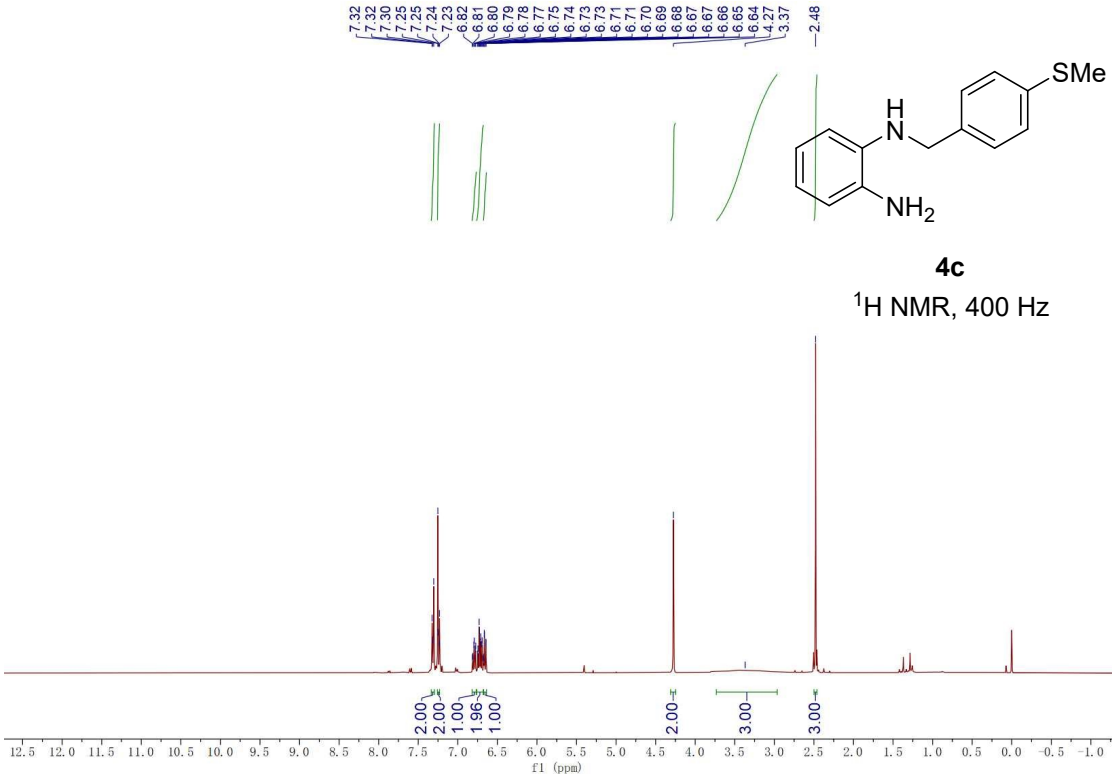




4b

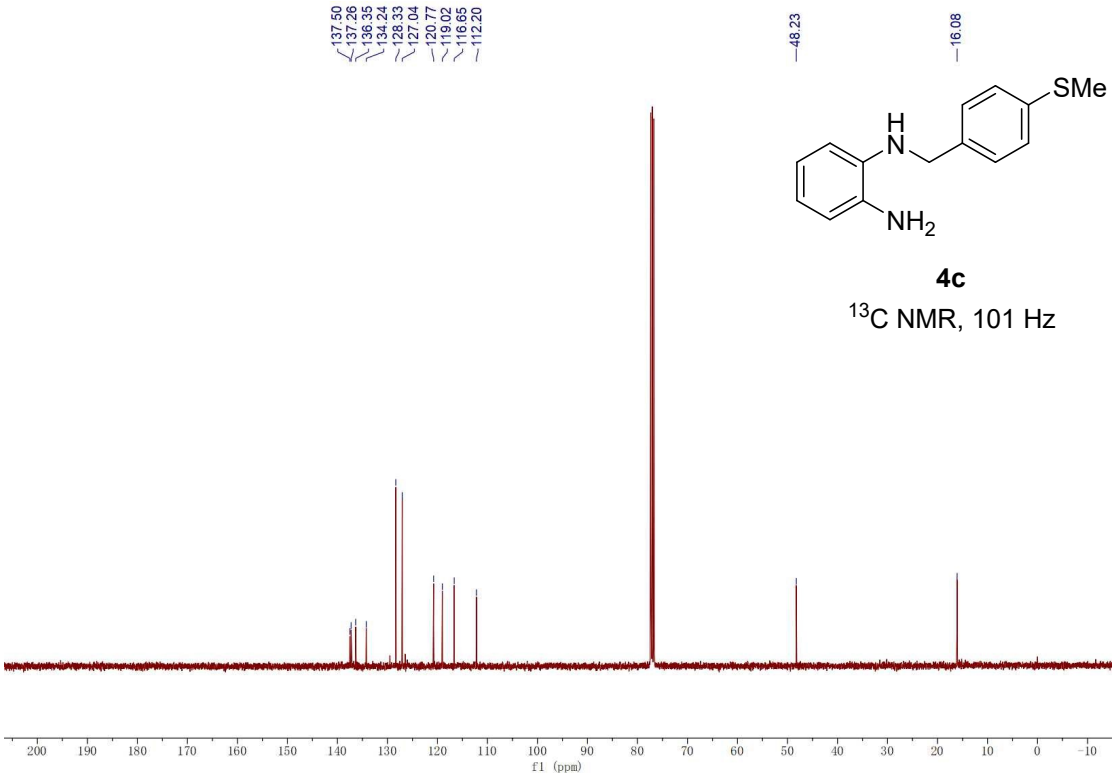
^{13}C NMR, 101 Hz





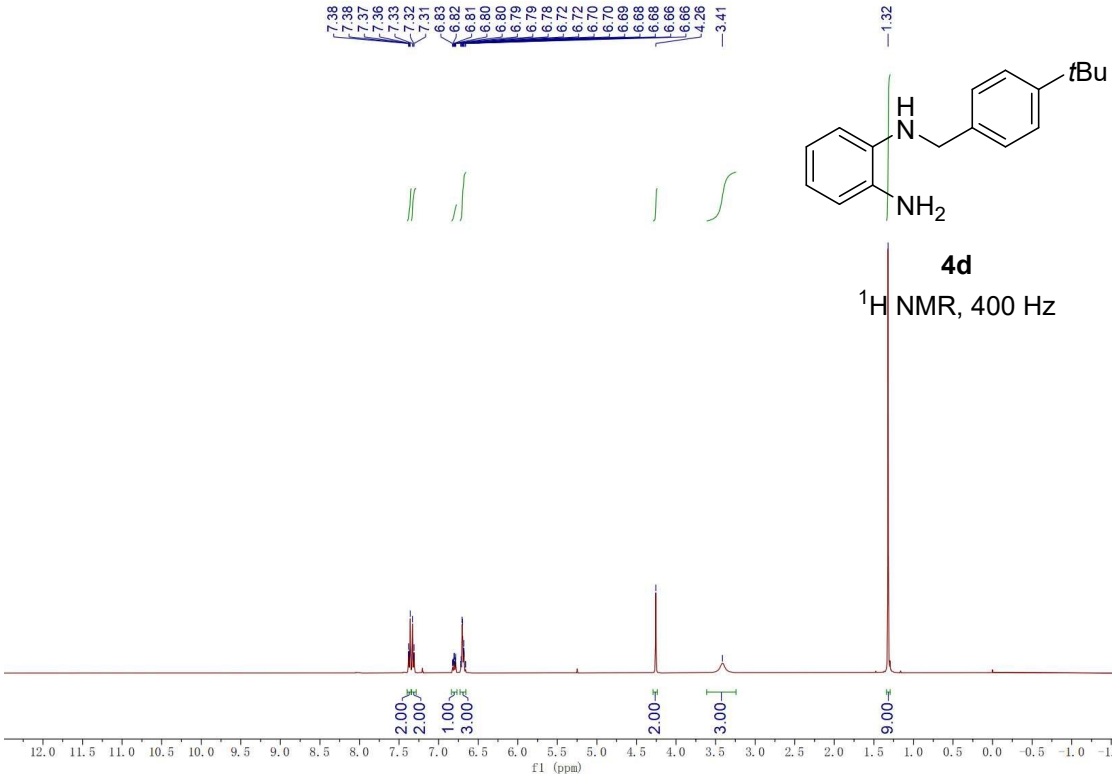
4c

^{1}H NMR, 400 Hz



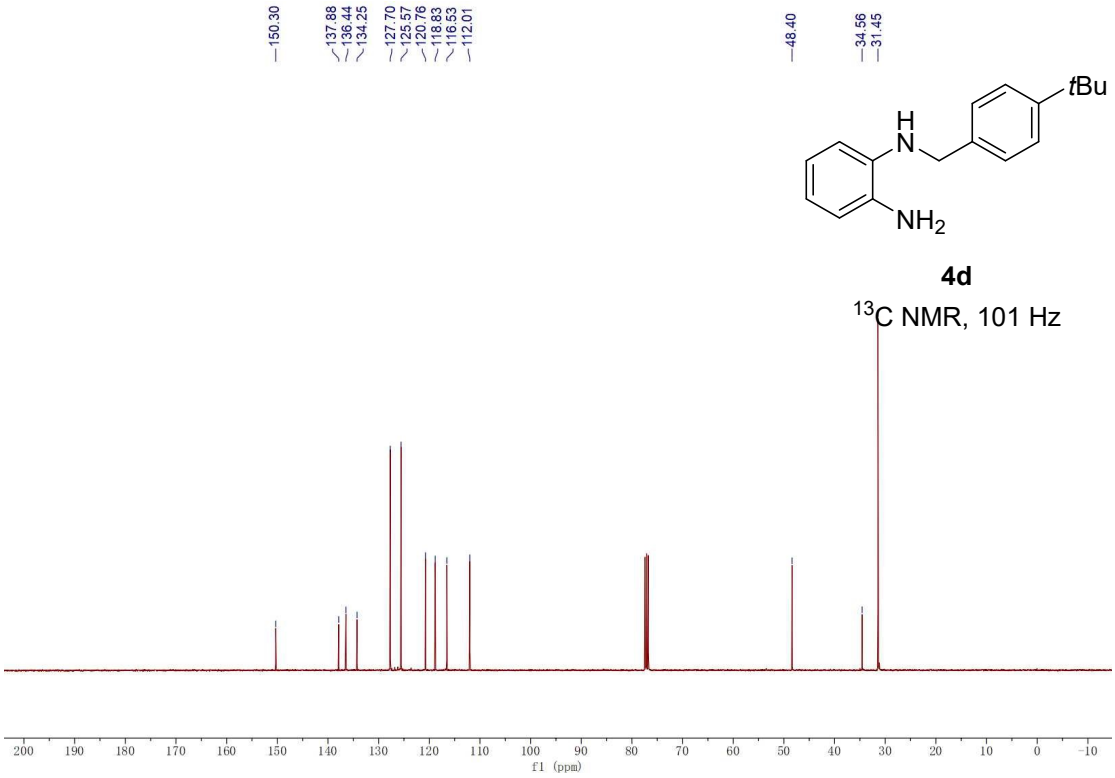
4c

^{13}C NMR, 101 Hz



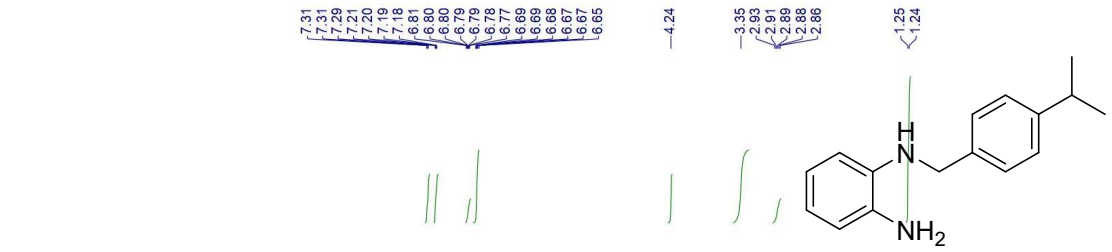
4d

^{1}H NMR, 400 Hz



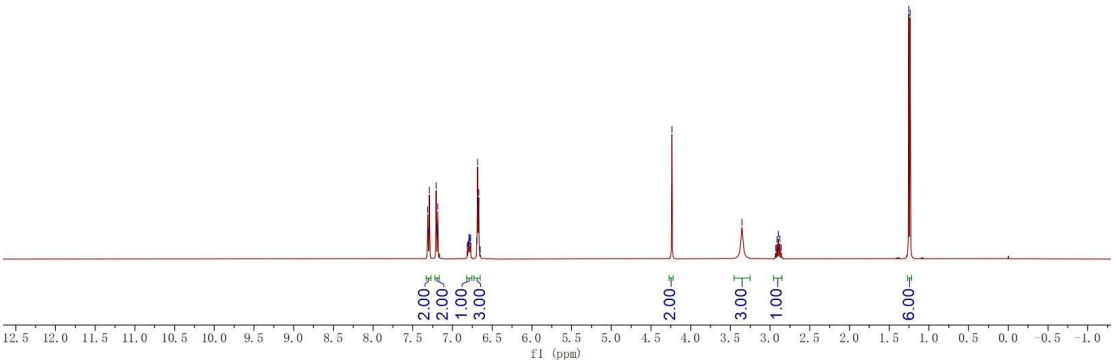
4d

^{13}C NMR, 101 Hz



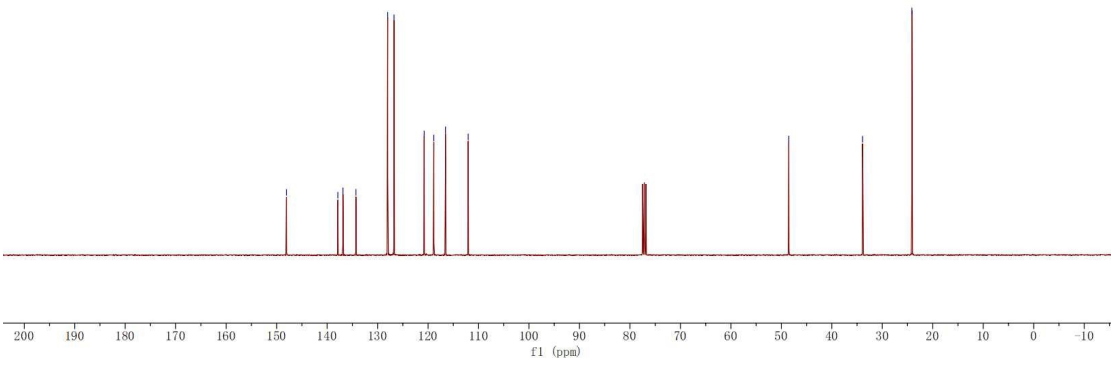
4e

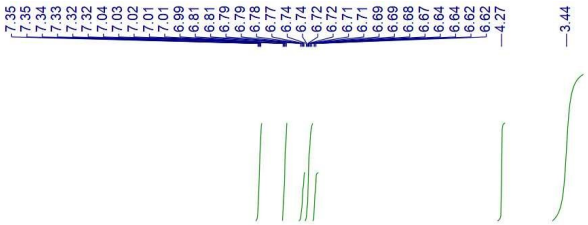
^{1}H NMR, 400 Hz

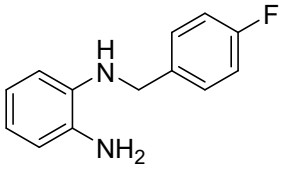


4e

^{13}C NMR, 101 Hz

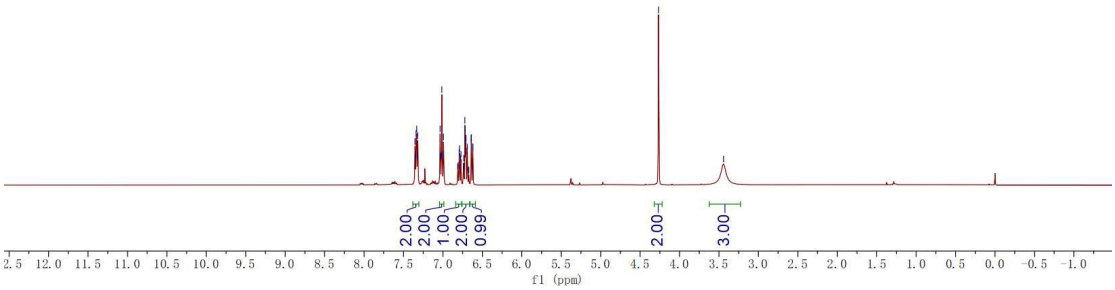


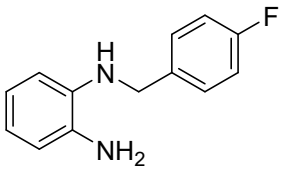




4f

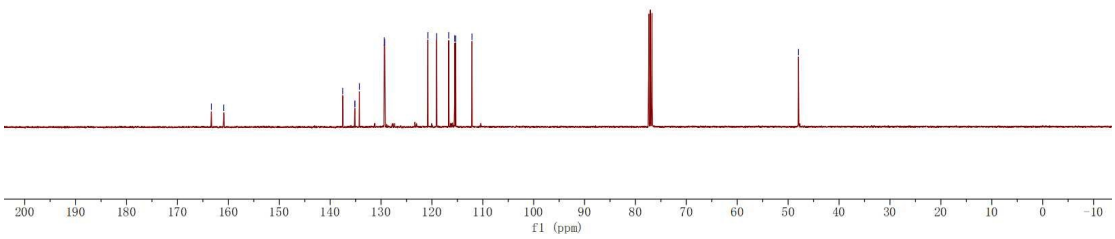
^{1}H NMR, 400 Hz

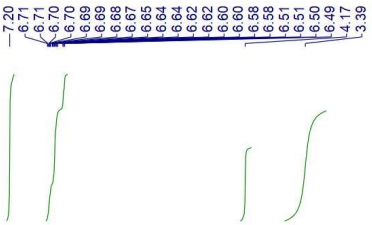


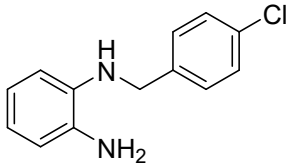


4f

^{13}C NMR, 101 Hz

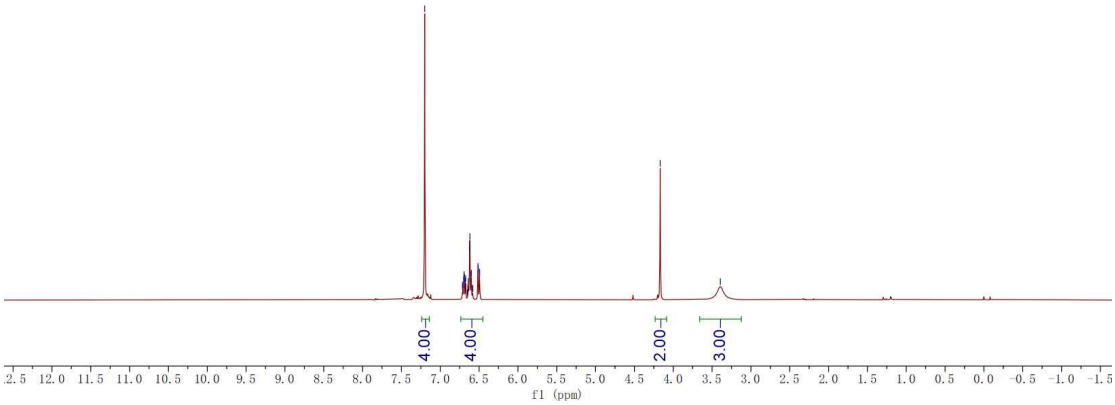


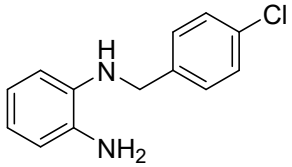




4g

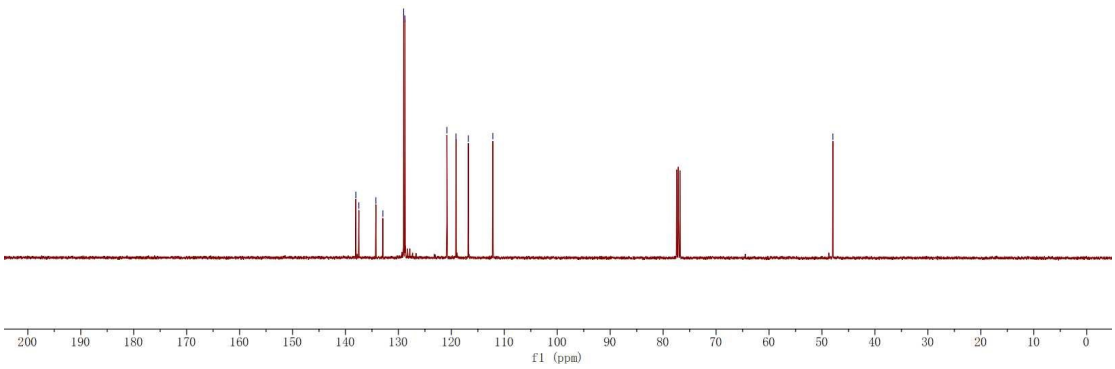
^{1}H NMR, 400 Hz

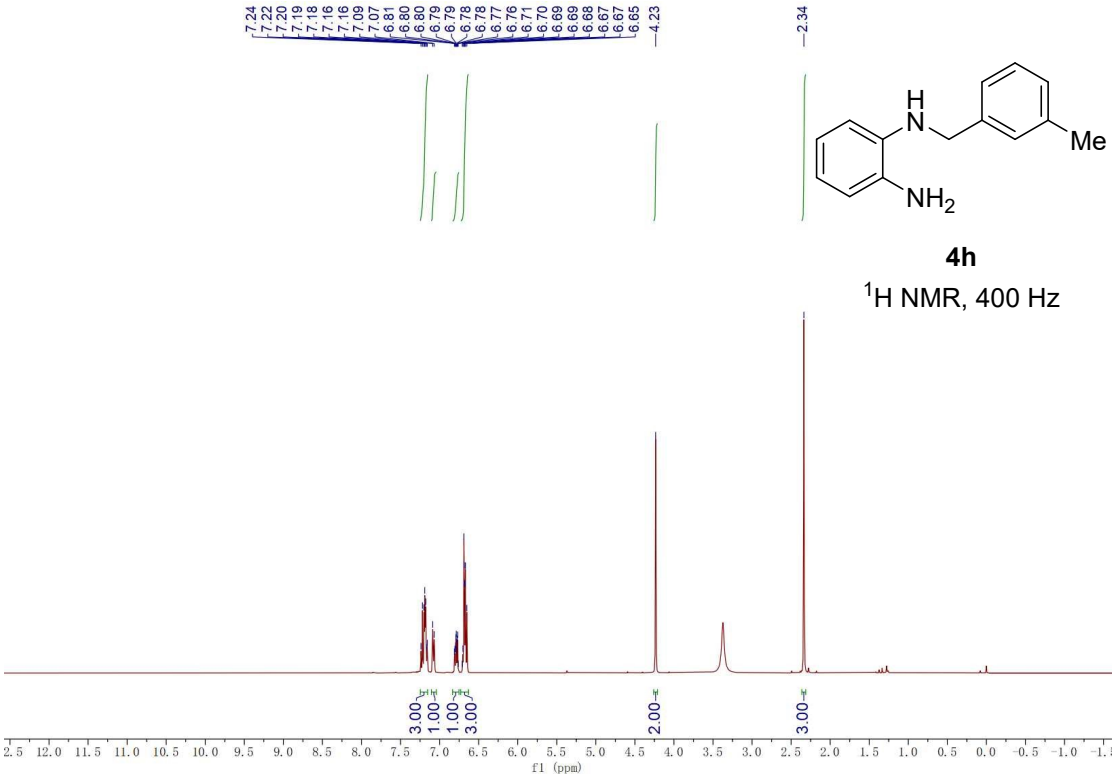




4g

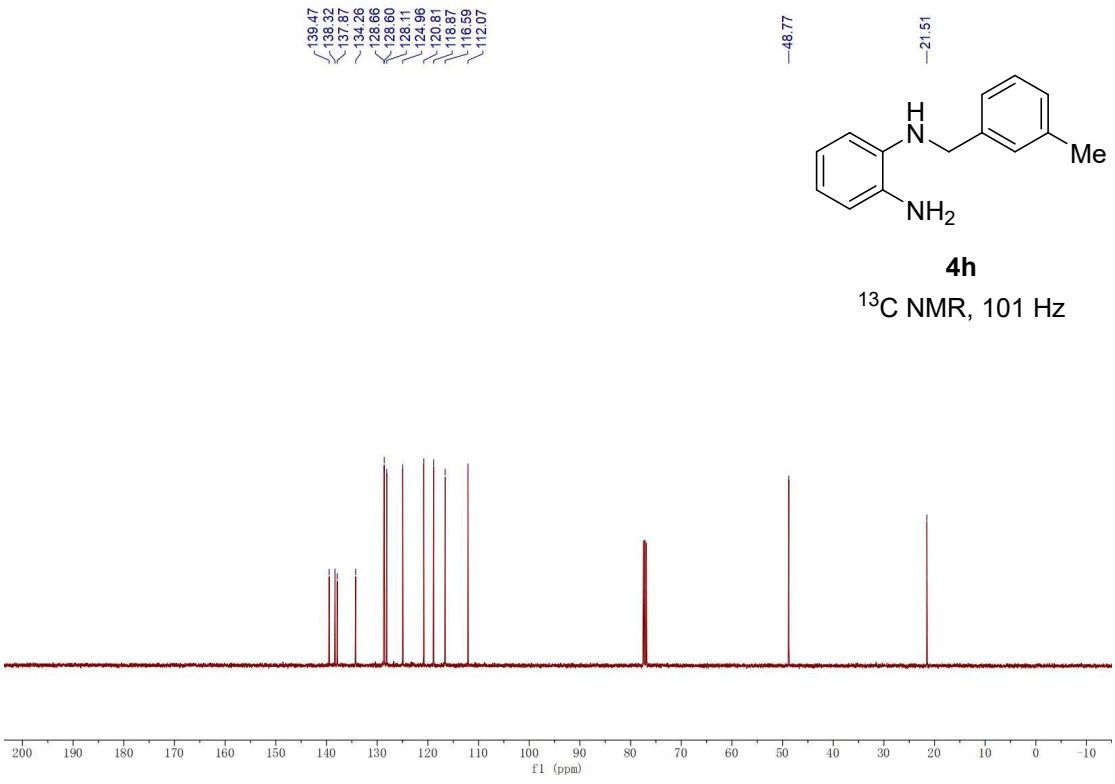
^{13}C NMR, 101 Hz





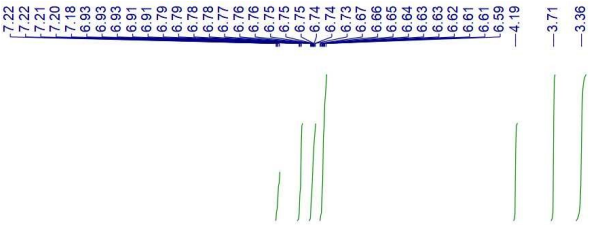
4h

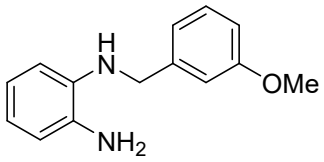
^{1}H NMR, 400 Hz



4h

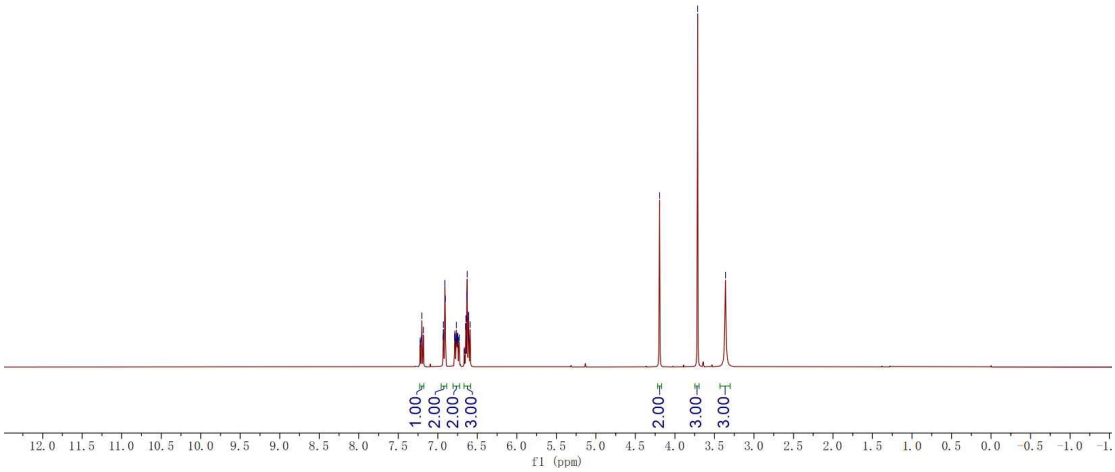
^{13}C NMR, 101 Hz

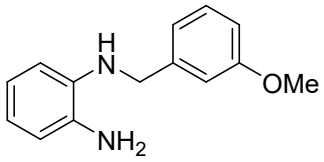




4i

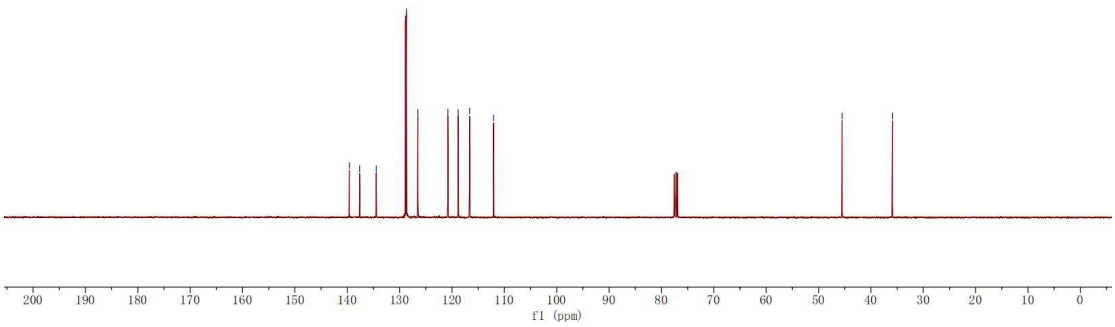
^{1}H NMR, 400 Hz

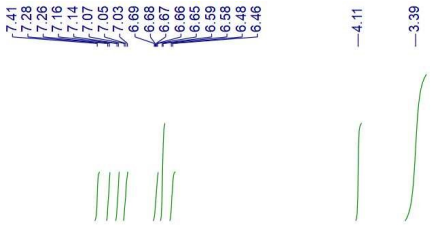


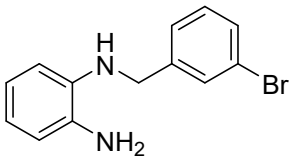


4i

^{1}H NMR, 400 Hz







4j

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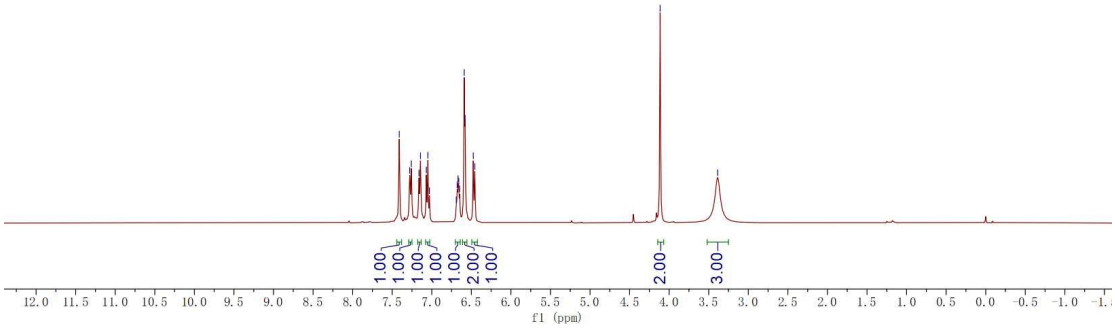
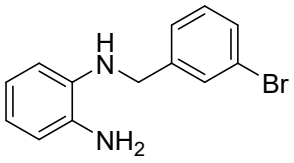
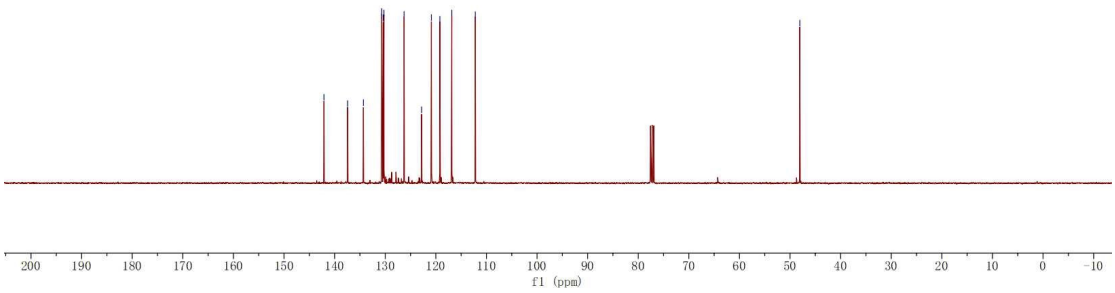
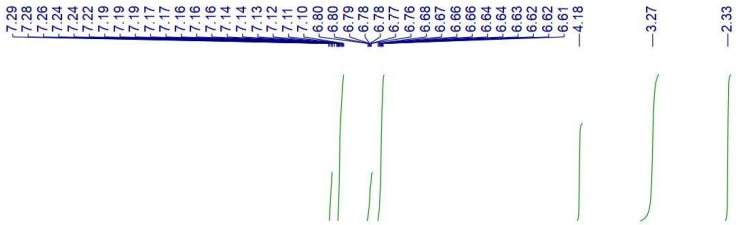


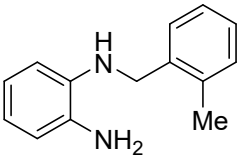


4j

^{13}C NMR, 101 Hz

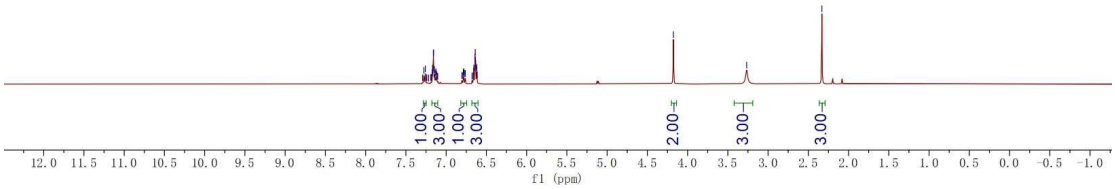


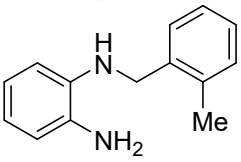




4k

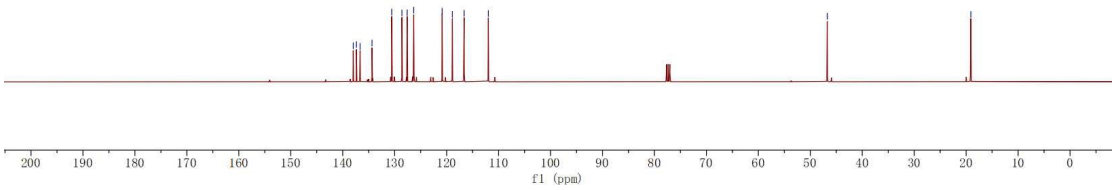
^{1}H NMR, 400 Hz

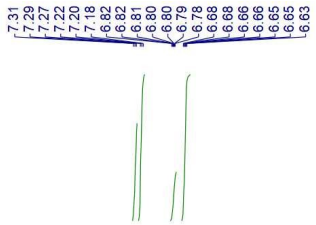


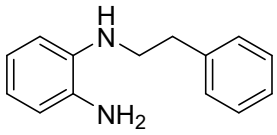


4k

^{13}C NMR, 101 Hz

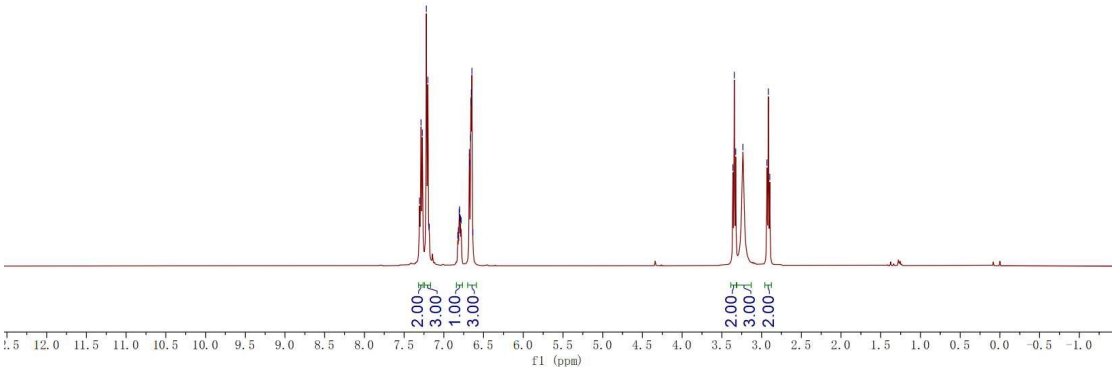


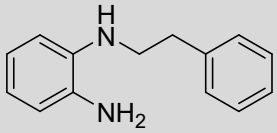




4l

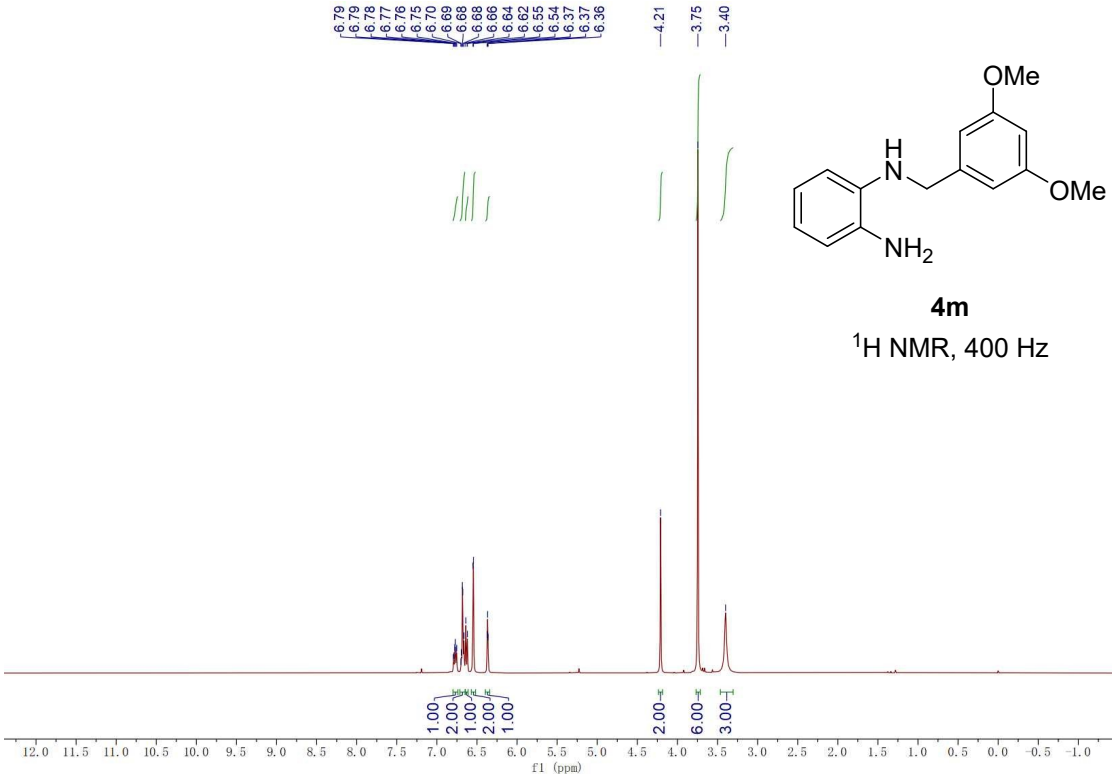
^{1}H NMR, 400 Hz





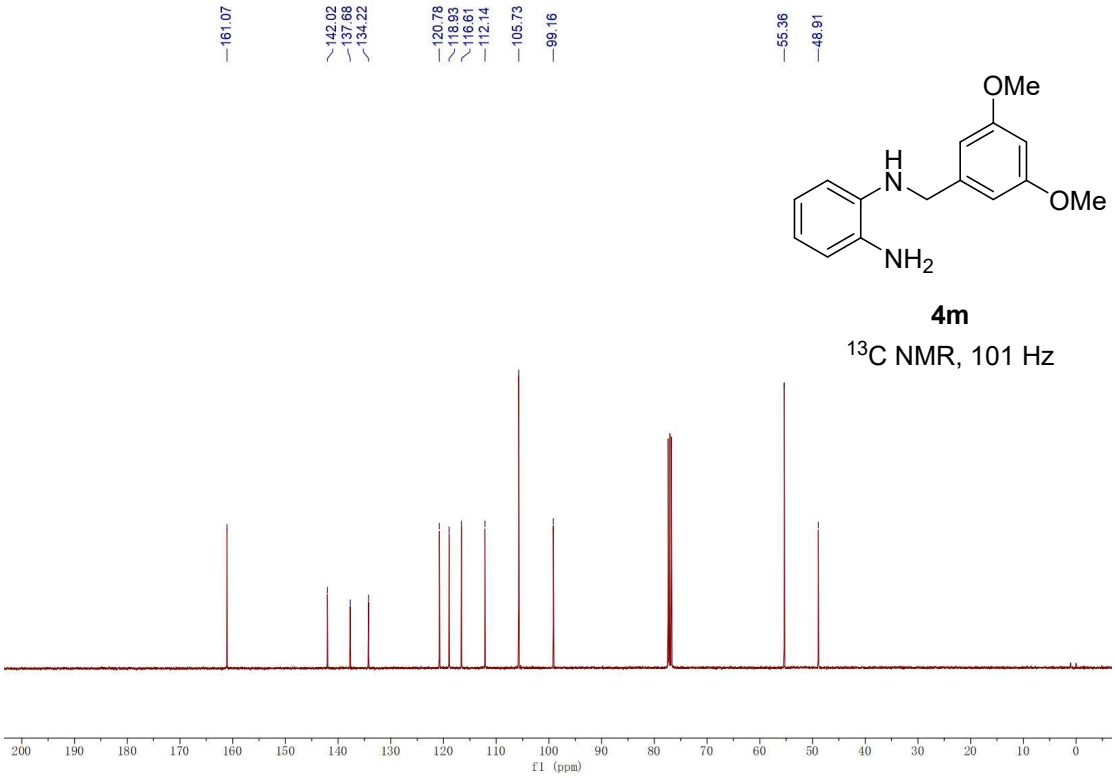
4l

^{13}C NMR, 101 Hz



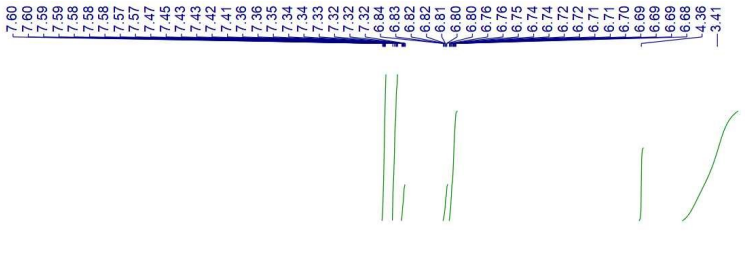
4m

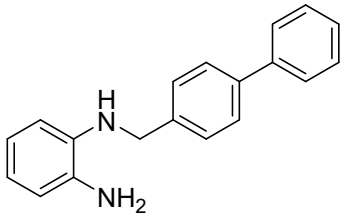
^{1}H NMR, 400 Hz



4m

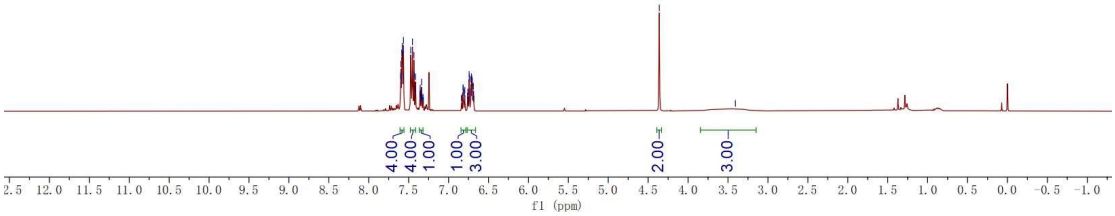
^{13}C NMR, 101 Hz

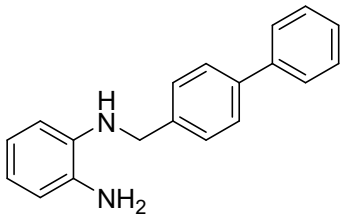




4n

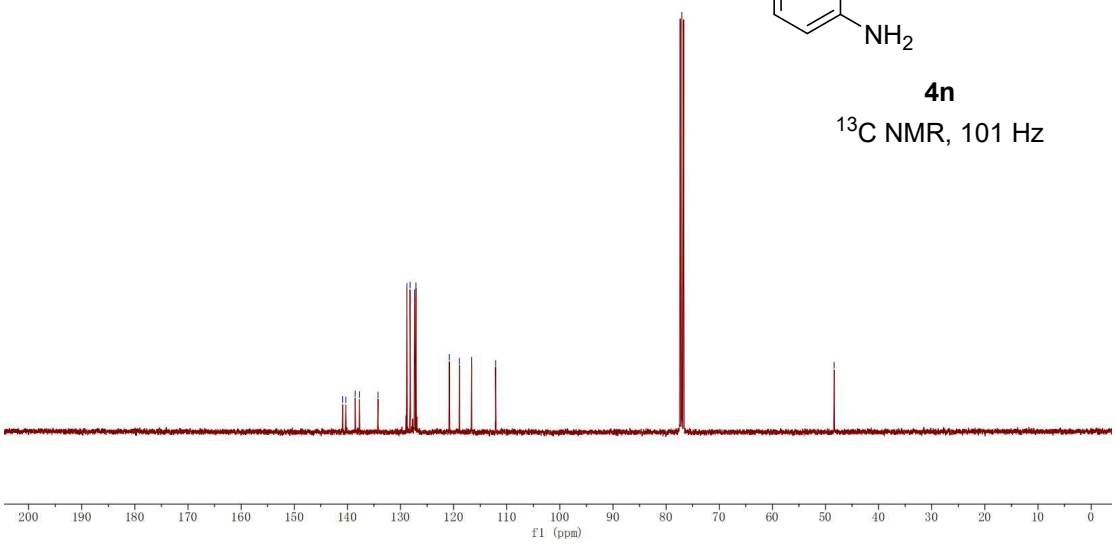
^{1}H NMR, 400 Hz

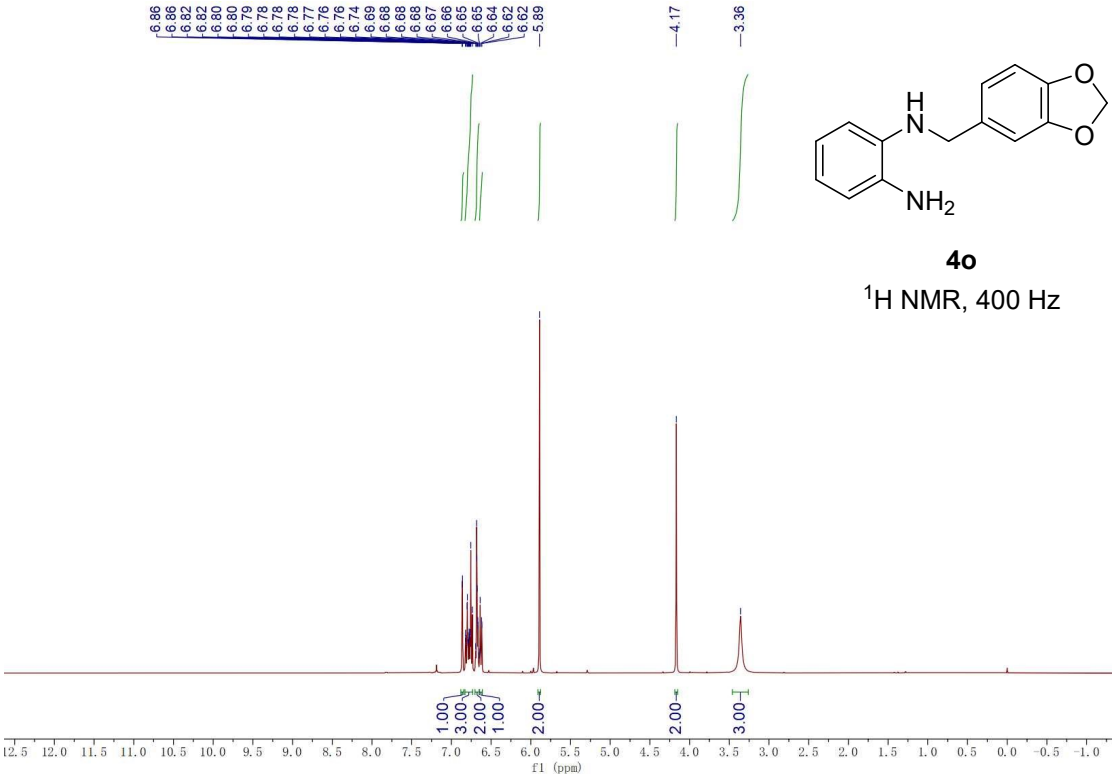




4n

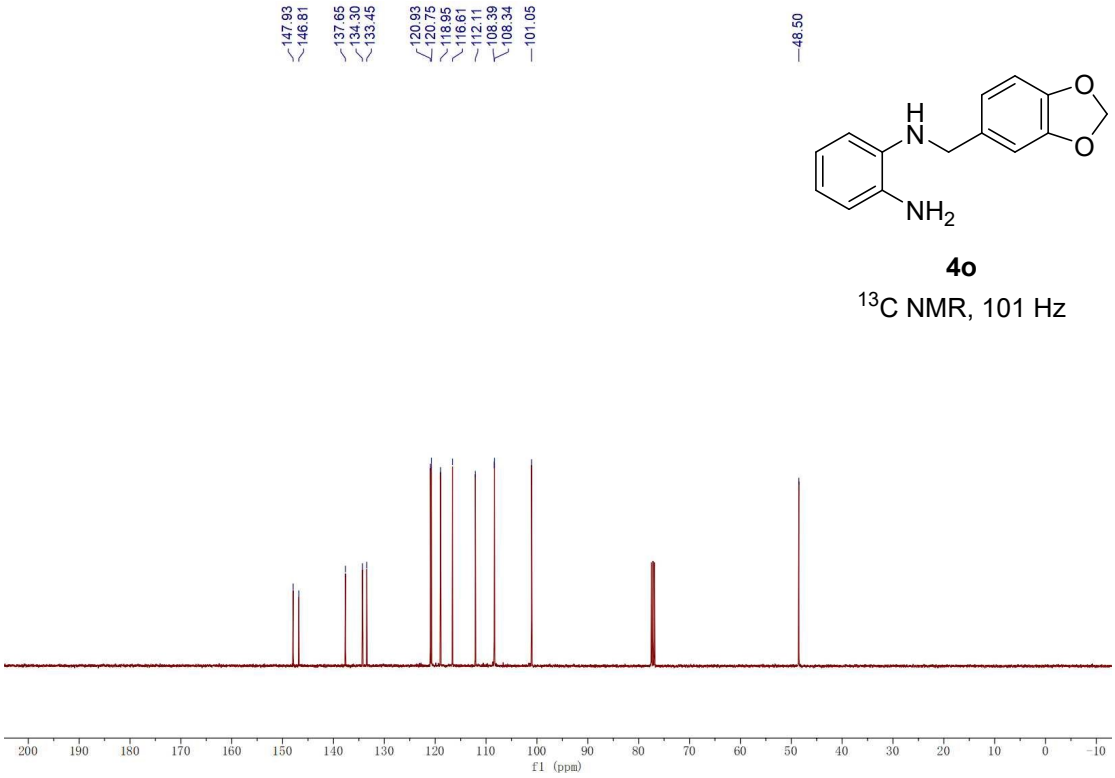
^{13}C NMR, 101 Hz





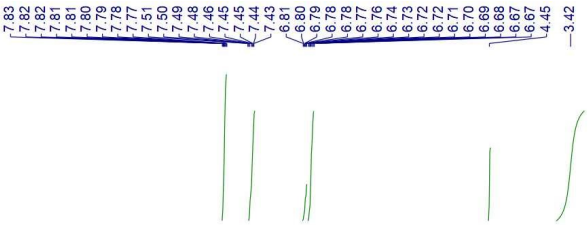
4o

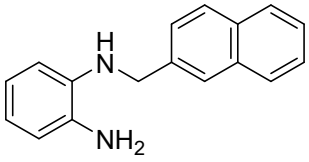
^{1}H NMR, 400 Hz



4o

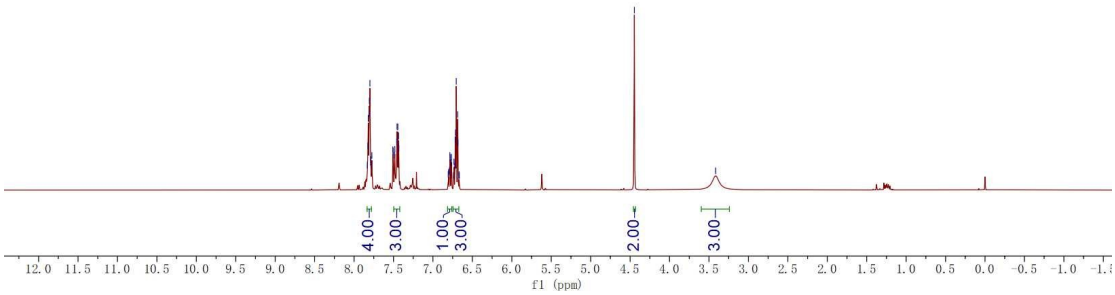
^{13}C NMR, 101 Hz

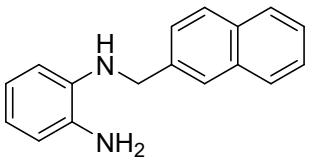




4p

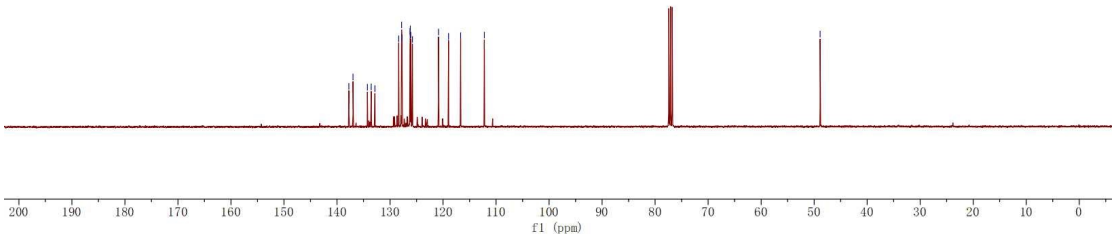
^{1}H NMR, 400 Hz

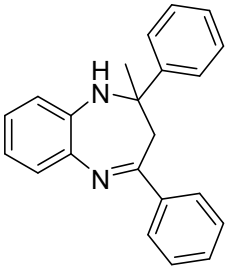




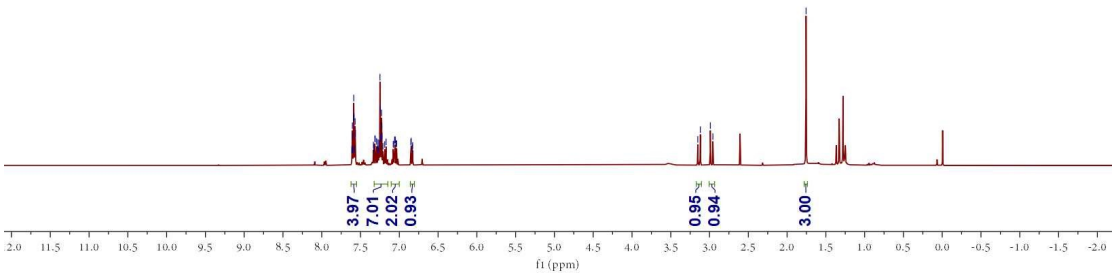
4p

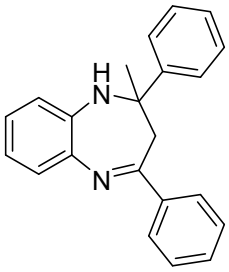
^{13}C NMR, 101 Hz





^{1}H NMR, 400 Hz





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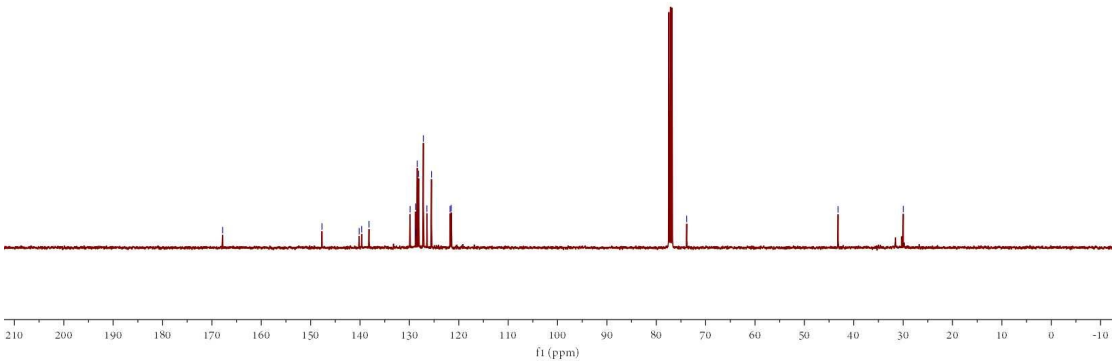

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