Supporting information

Stabilizing Pd Nanoparticles via N,O-Chelation in a MOF for General Reductive Amination

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S1. Experimental Section

1.1 Materials

Salicylaldehyde was purchased from Tianjin Guang fu Fine Chemical Research Institute; Zirconium tetrachloride (Shanghai Macklin); 2-Aminoterephthalic acid (Shanghai Macklin); *N*,*N*-Dimethylformamide (Tianjin Fuyu Fine Chemical Co., Ltd.); Palladium acetate (Beijing J&K Scientific Ltd.). Alcohol and nitroarene substrates such as 4-fluorobenzaldehyde, 2-methoxynitrobenzene, 4-fluoronitrobenzene, and p-methoxynitro benzene were used as received. All other reagents, including ethyl acetate, petroleum ether, ethanol, etc., were obtained from various commercial sources and could be used without further purification.

1.2 Characterization

The morphology and EDX elemental mapping of the samples were characterized using a Zeiss SUPRA 55 Sapphire field emission scanning electron microscope (FESEM, Germany) at an accelerating voltage of 5 kV. Nitrogen adsorption measurements were performed on a Micromeritics AsAp2020 analyzer (USA), with Brunauer-Emmett-Teller (BET) specific surface area and Barrett-Joyner-Halenda (BJH) pore size analyses carried out automatically. The BJH analysis was based on the desorption branch of the isotherm. X-ray diffraction (XRD) patterns were collected on a Rigaku Miniflex600 X-ray powder diffractometer (Japan) using Cu Kα radiation (λ = 1.5406 Å). Infrared spectra were acquired using a PerkinElmer Model 1600 Fourier-transform infrared spectrometer (FT-IR, USA). X-ray photoelectron spectroscopy (XPS) data were obtained on a VG Multi Lab 2000 system (USA) equipped with a monochromatic Al Kα X-ray source. Transmission electron microscopy (TEM) images were recorded on a JEOL JEM-2010 electron microscope (Japan) at an accelerating voltage of 200 kV. The Pd loading was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) using a Prodigy 7 instrument from Leeman Labs Inc. (USA). Gas chromatography (GC) yields were measured on a Varian GC 3900 gas

chromatograph (USA) equipped with an FID detector. ^{1}H NMR spectra were recorded at 300.1 MHz and 400.1 MHz on Bruker AVANCE 300 and 400 spectrometers, respectively, using $CDCl_3$ (7.26 ppm) as the internal standard. ^{13}C NMR spectra were obtained at 75 MHz or 100 MHz, referenced to the solvent signal (central peak of $CDCl_3$ at 77.16 ppm). Chemical shifts and coupling constants (J) are reported in ppm and Hz, respectively. Peak multiplicities are denoted as follows: s (singlet), d (doublet), t (triplet), m (multiplet).

Supplementary Figures

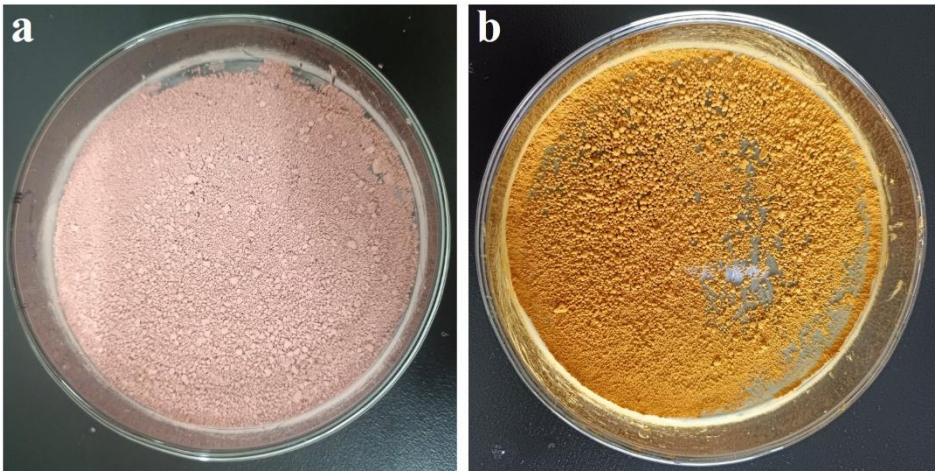


Figure S1 Comparative diagrams of (a) UiO-66-NH_2 and (b) UiO-66-NO samples.

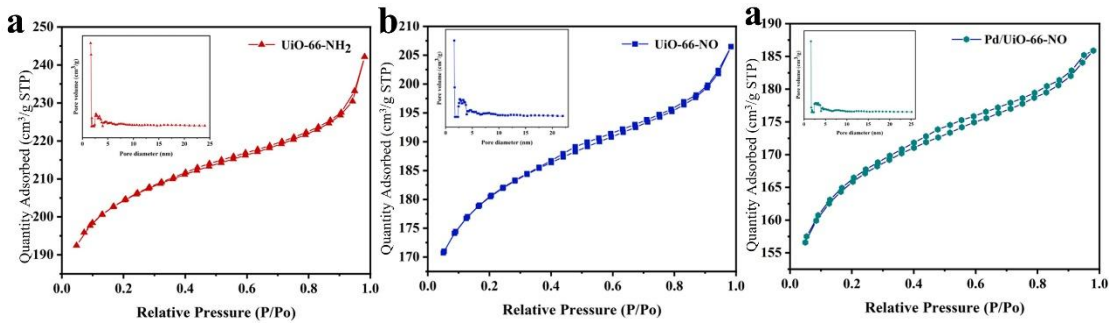


Figure S2. (a) UiO-66-NH_2, (b) UiO-66-NO, and (c) Pd/UiO-66-NO nitrogen adsorption-desorption isotherms and pore size distribution plots.

Sample	Surface area (m^2/g)	Mean Pore Diameter (nm)	Pore volume (cm^3/g)
UiO-66-NH_2	629	3.095	0.324
UiO-66-NO	563	3.090	0.281
Pd/UiO-66-NO	518	3.089	0.257

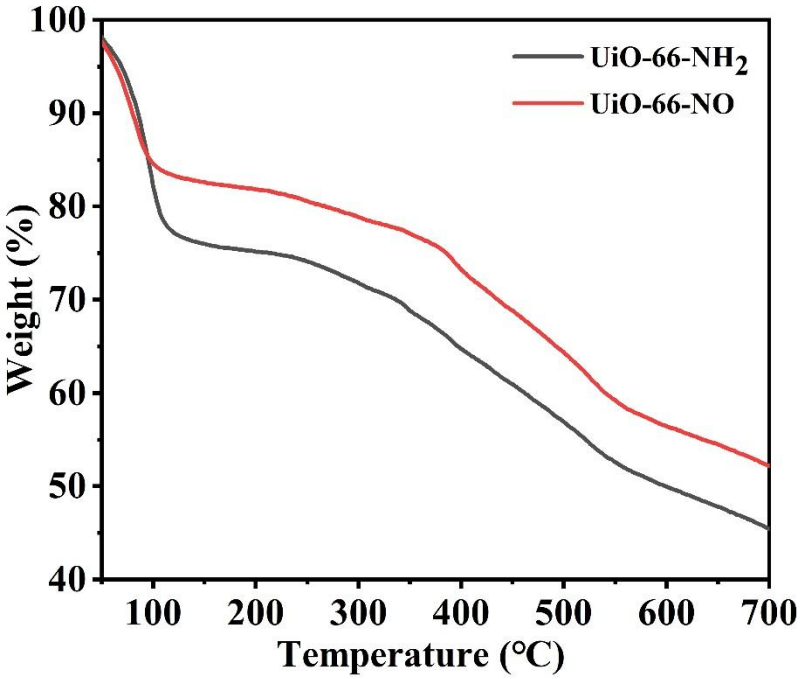


Figure S3. TG curves of UiO-66-NH_2 and UiO-66-NO.

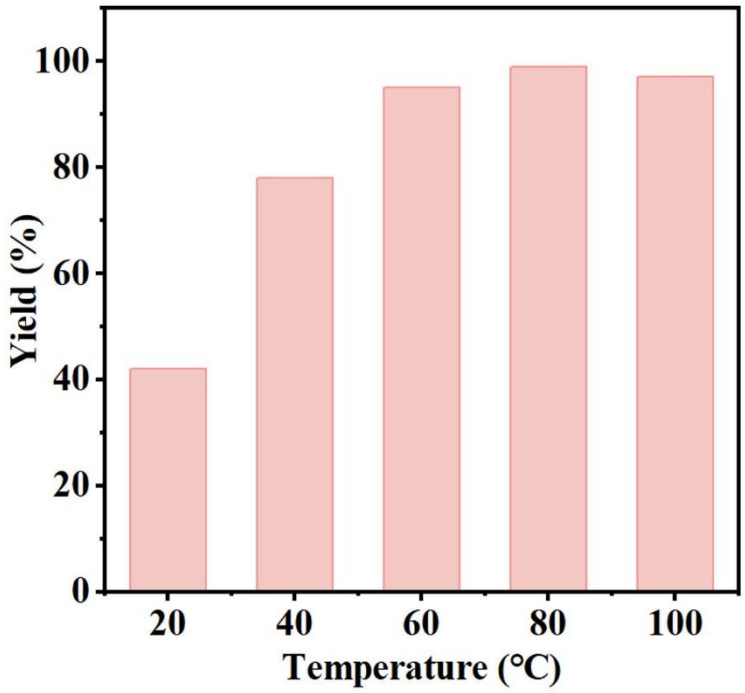


Figure S4. Effect of temperature on the reductive amination of aniline with aldehyde.

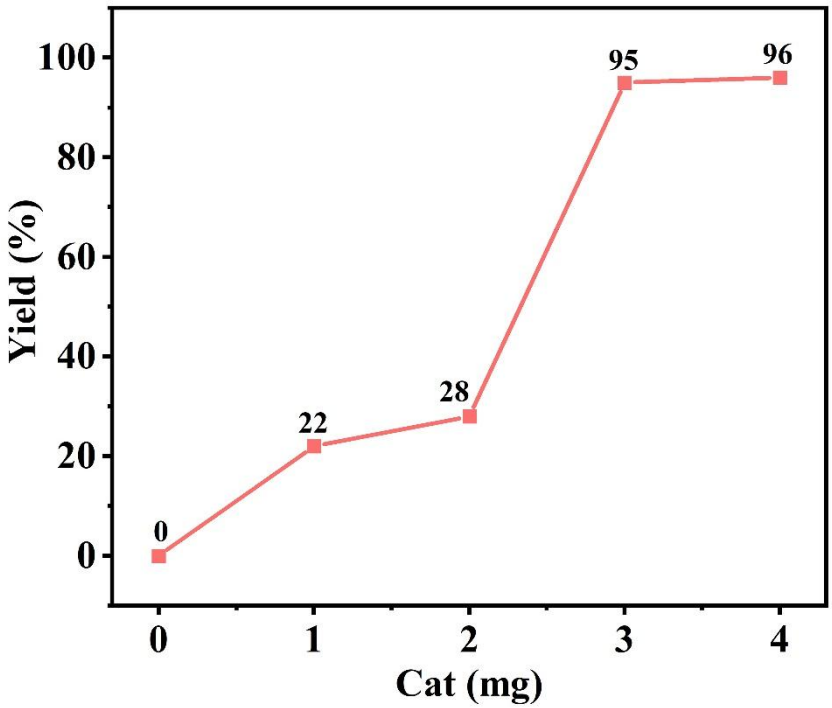


Figure S5. Effect of catalyst dosage on the reductive amination of aniline with aldehyde.

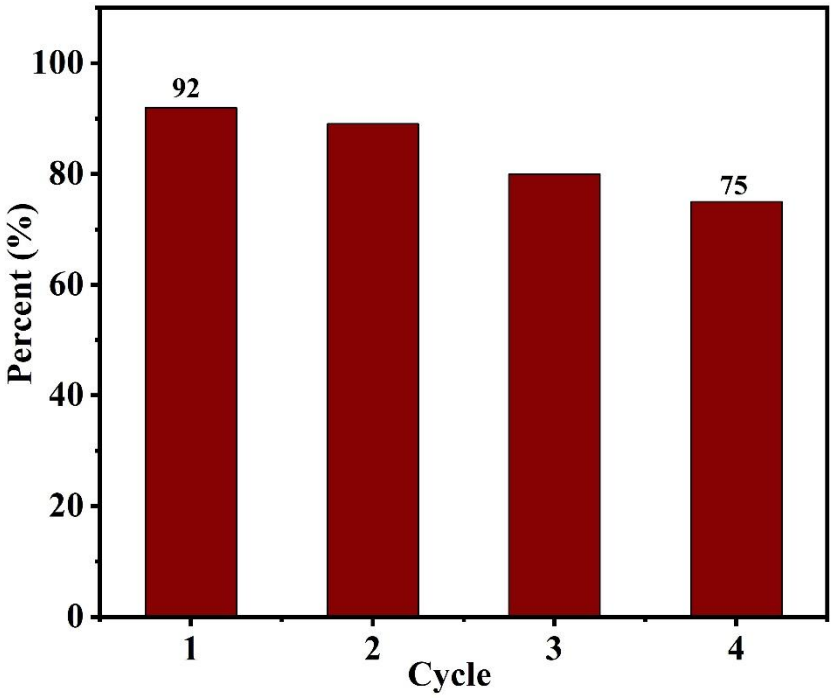


Figure S6. Recyclability of Pd/UiO-66-NO.

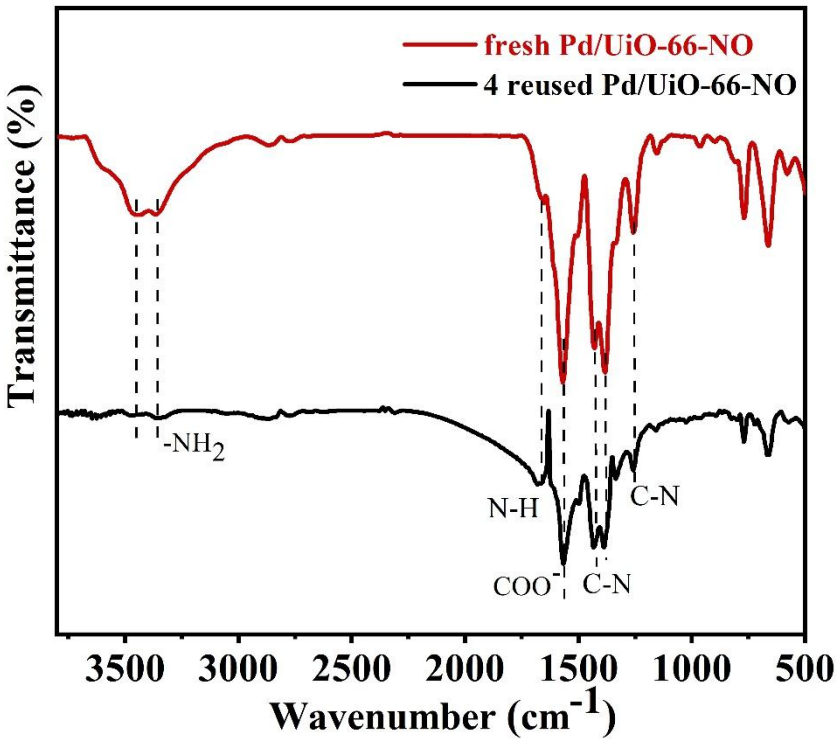
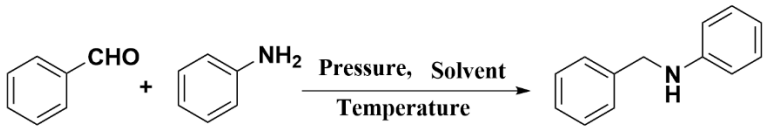


Figure S7. FT-IR spectra of Pd/UiO-66-NO after four reuses.

Supplementary Tables

Table S1. Effect of solvent and pressure on the reaction of aniline with aldehydes [a]



Entry	Solvent	Pressure (M pa)	Temperature (℃)	Yield [b] (%)
1	Toluene	3	60	95
2	DMSO	3	60	2
3	Methanol	3	60	81
4	H_2O	3	60	68
5	Acetonitrile	3	60	72
6	Ethyl acetate	3	60	83
7	Dichloromethane	3	60	35
8	THF	3	60	Trace
9	N-hexane	3	60	55
10	DMF	3	60	44
11	Toluene	1	60	55
12	Toluene	2	60	75

[a] Reaction conditions: 0.25 mmol benzaldehyde, 0.35 mmol aniline, 2 mL solvent, and 0.24 mol% [Pd] (Pd: substrate, mol%), reacted at 60 °C under 3 MPa H_2 for 3 h. [b] Product yield was determined by gas chromatography.

S2. NMR Data of the Products

N-benzylaniline (1): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (m, 4H), 7.18 (d, *J* = 3.7 Hz, 1H), 7.07 (t, *J* = 7.1 Hz, 2H), 6.62 (t, *J* = 7.2 Hz, 1H), 6.53 (d, *J* = 7.9 Hz, 2H), 4.21 (s, 2H), 3.91 (s, 1H).

N-(4-methoxybenzyl)aniline (2): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.5 Hz, 2H), 7.12 – 7.03 (m, 2H), 6.82 – 6.73 (m, 2H), 6.62 (t, *J* = 7.3 Hz, 1H), 6.53 (d, *J* = 7.8 Hz, 2H), 4.14 (s, 2H), 3.87 (s, 1H), 3.69 (s, 3H).

N-benzyl-4-methylaniline (3): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.12 (m, 5H), 6.90 (d, *J* = 8.1 Hz, 2H), 6.50 – 6.45 (m, 2H), 4.21 (s, 2H), 3.83 (s, 1H), 2.15 (s, 3H).

N-benzyl-3,5-dimethylaniline (4): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.21 (m, 4H), 7.20 – 7.11 (m, 1H), 6.30 (s, 1H), 6.20 (s, 2H), 4.20 (s, 2H), 3.85 (s, 1H), 2.14 (s, 6H).

N-(3-fluorobenzyl)-3,5-dimethylaniline (5): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.16 (m, 1H), 7.09 – 6.96 (m, 2H), 6.87 (m, 1H), 6.33 (s, 1H), 6.18 (s, 2H), 4.23 (s, 2H), 4.01 (s, 1H), 2.14 (s, 6H).

4-methoxy-N-(4-methoxybenzyl) aniline (6): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.81 – 6.74 (m, 2H), 6.71 – 6.64 (m, 2H), 6.53 – 6.47 (m, 2H), 4.09 (s, 2H), 3.69 (s, 3H), 3.63 (s, 3H), 3.46 (s, 1H).

N-(2-fluorobenzyl) aniline (7): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 1H), 7.18 – 7.03 (m, 3H), 7.02 - 6.91 (m, 2H), 6.67 – 6.59 (m, 1H), 6.55 (d, *J* = 8.0 Hz, 2H), 4.30 (s, 2H), 3.95 (s, 1H).

N-(4-methoxybenzyl)-4-methylaniline (8): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 – 7.12 (m, 2H), 6.89 (d, *J* = 9.1 Hz, 2H), 6.81 – 6.73 (m, 2H), 6.51 – 6.41 (m, 2H), 4.13 (s, 2H), 3.70 (s, 3H), 2.15 (s, 3H).

N-benzyl-3-methylaniline (9): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (m, 4H), 7.21 – 7.15 (m, 1H), 7.00 – 6.95 (m, 1H), 6.47 (d, *J* = 7.4 Hz, 1H), 6.41 – 6.34 (m, 2H), 4.22 (s, 2H), 3.89 (s, 1H), 2.18 (s, 3H).

N-benzyl-2-methoxyaniline (10): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.14 (m, 5H), 6.77 – 6.67 (m, 2H), 6.58 (m, 1H), 6.50 (m, 1H), 4.55 (s, 1H), 4.25 (s, 2H), 3.74 (s, 3H).

N-benzyl-4- (trifluoromethoxy) aniline (11): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.13 (m, 5H), 6.95 (d, *J* = 7.7 Hz, 2H), 6.59 – 6.40 (m, 2H), 4.23 (s, 2H).

N-benzyl-4-fluoroaniline (12): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.15 (m, 5H), 6.83 – 6.75 (m, 2H), 6.49 – 6.43 (m, 2H), 4.19 (s, 2H).

N-benzyl-3-methoxyaniline (13): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.09 (m, 5H), 6.97 (t, *J* = 8.1 Hz, 1H), 6.23 – 6.04 (m, 3H), 4.20 (s, 2H), 3.95 (s, 1H), 3.63 (s, 3H).

N-(4-methoxybenzyl)-3,5-dimethylaniline (14): ^{1}H NMR (400 MHz, $CDCl_3$ δ 7.18 (d, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 9.1 Hz, 2H), 6.29 (s, 1H), 6.18 (s, 2H), 4.12 (s, 2H), 3.69 (s, 3H), 2.14 (s, 6H).

3-methoxy-N-(4-methoxybenzyl) aniline (15): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.6 Hz, 2H), 6.99 – 6.94 (m, 1H), 6.77 (d, *J* = 8.7 Hz, 2H), 6.20 – 6.13 (m, 2H), 6.09 (t, *J* = 2.3 Hz, 1H), 4.12 (s, 2H), 3.92 (s, 1H), 3.68 (s, 3H), 3.63 (s, 3H).

N-(4-methoxybenzyl)-3-methylaniline (16): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.96 (t, *J* = 7.7 Hz, 1H), 6.77 (d, *J* = 8.5 Hz, 2H), 6.45 (d, *J* = 7.4 Hz, 1H), 6.35 (d, *J* = 10.5 Hz, 2H), 4.12 (s, 2H), 3.88 (s, 1H), 3.71 – 3.67 (m, 3H), 2.17 (s, 3H).

N-(4-methoxybenzyl)-4-(trifluoromethoxy) aniline (17): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.5 Hz, 2H), 6.92 (d, *J* = 8.6 Hz, 2H), 6.79 (d, *J* = 8.5 Hz, 2H), 6.47 (d, *J* = 8.9 Hz, 2H), 4.12 (s, 2H), 3.92 (s, 1H), 3.70 (s, 3H).

4-fluoro-N-(4-methoxybenzyl) aniline (18): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.79 (m, 4H), 6.49 – 6.43 (m, 2H), 4.11 (s, 2H), 3.70 (s, 3H).

3,5-dimethyl-N-(3-methylbenzyl) aniline (19): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 – 7.05 (m, 3H), 7.00 (d, *J* = 7.4 Hz, 1H), 6.31 (s, 1H), 6.21 (s, 2H), 4.16 (s, 2H), 3.80 (s, 1H), 2.26 (s, 3H), 2.15 (s, 6H).

3-methyl-N-(3-methylbenzyl) aniline (20): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.03 (m, 4H), 7.02 – 6.95 (m, 2H), 6.46 (d, *J* = 7.4 Hz, 1H), 6.41 – 6.34 (m, 2H), 4.18 (s, 2H), 3.93 (s, 1H), 2.26 (s, 3H), 2.19 (s, 3H).

3-methyl-N-(2-methylbenzyl) aniline (21): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 7.5 Hz, 1H), 7.12 – 7.04 (m, 3H), 7.00 – 6.94 (m, 1H), 6.45 (d, J = 7.4 Hz, 1H), 6.38 – 6.31 (m, 2H), 4.14 (s, 2H), 3.67 (s, 1H), 2.26 (s, 3H), 2.18 (s, 3H).

2-methoxy-N-(4-methoxybenzyl) aniline (22): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.3 Hz, 2H), 6.81 – 6.73 (m, 3H), 6.72 – 6.68 (m, 1H), 6.62 – 6.52 (m, 2H), 4.50 (s, 1H), 4.19 (s, 2H), 3.75 (s, 3H), 3.72 (s, 3H).

N-(2-methylbenzyl)-4-(trifluoromethoxy) aniline (23): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 6.8 Hz, 1H), 7.10 (d, *J* = 10.4 Hz, 3H), 6.94 (d, *J* = 8.3 Hz, 2H), 6.46 (d, *J* = 8.8 Hz, 2H), 4.14 (s, 2H), 3.84 (s, 1H), 2.26 (s, 3H).

3-methoxy-N-(2-methylbenzyl) aniline (24): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 6.5 Hz, 1H), 7.15 – 7.06 (m, 3H), 7.04 – 6.98 (m, 1H), 6.20 (m, 2.4 Hz, 2H), 6.12 (t, *J* = 2.2 Hz, 1H), 4.17 (s, 2H), 3.84 (s, 1H), 3.68 (s, 3H), 2.28 (s, 3H).

N-(2-fluorobenzyl)-2-methoxyaniline (25): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 1H), 7.17 – 7.11 (m, 1H), 7.02 – 6.94 (m, 2H), 6.73 (m, 7.6, 6.5 Hz, 2H), 6.60 (m, 1.4 Hz, 1H), 6.51 (m, 1H), 4.56 (s, 1H), 4.34 (s, 2H), 3.76 (s, 3H).

N-(2-fluorobenzyl)-3,5-dimethylaniline (26): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (t, *J* = 7.4 Hz, 1H), 7.12 (m, 1H), 7.02 – 6.89 (m, 2H), 6.30 (s, 1H), 6.19 (s, 2H), 4.26 (s, 2H), 3.70 (s, 1H), 2.13 (s, 6H).

N-(2-fluorobenzyl)-3-methylaniline (27): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 1H), 7.17 – 7.10 (m, 1H), 7.01 – 6.92 (m, 3H), 6.46 (d, J = 8.5 Hz, 1H), 6.36 (d, J = 10.0 Hz, 2H), 4.28 (s, 2H), 3.86 (s, 1H), 2.17 (s, 3H).

N-(2-fluorobenzyl)-4-(trifluoromethoxy) aniline (28): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (t, *J* = 7.9 Hz, 1H), 7.17 – 7.09 (m, 1H), 7.01 – 6.86 (m, 4H), 6.47 (d, *J* = 1.9 Hz, 2H), 4.25 (s, 2H), 3.98 (s, 1H).

N-(2-fluorobenzyl)-4-methylaniline (29): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 6.59 (m, 6H), 6.57 – 6.17 (m, 2H), 4.26 (d, *J* = 9.9 Hz, 2H), 3.65 (s, 1H), 2.12 (d, *J* = 10.3 Hz, 3H).

N-(furan-2-ylmethyl) aniline (30): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 1.0 Hz, 1H), 7.13 – 7.08 (m, 2H), 6.66 (t, *J* = 7.3 Hz, 1H), 6.59 (d, *J* = 7.8 Hz, 2H), 6.26 – 6.22 (m, 1H), 6.15 (d, *J* = 3.1 Hz, 1H), 4.23 (s, 2H), 3.93 (s, 1H).

N-(4-chlorobenzyl)-4-methylaniline (31): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 1.3 Hz, 4H), 6.89 (d, J = 6.8 Hz, 2H), 6.48 – 6.38 (m, 2H), 4.18 (s, 2H), 3.85 (s, 1H), 2.14 (s, 3H).

N-(4-chlorobenzyl)-4-(trifluoromethoxy) aniline (32): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.16 (m, 4H), 6.94 (d, *J* = 8.6 Hz, 2H), 6.50 – 6.43 (m, 2H), 4.20 (s, 2H), 4.12 (s, 1H).

N-(4-chlorobenzyl)-3,5-dimethylaniline (33): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (s, 4H), 6.31 (s, 1H), 6.16 (s, 2H), 4.18 (s, 2H), 3.80 (s, 1H), 2.13 (s, 6H).

N-(furan-2-ylmethyl)-3,5-dimethylaniline (34): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 1.7 Hz, 1H), 6.33 (s, 1H), 6.24 (s, 3H), 6.14 (d, J = 3.2 Hz, 1H), 4.21 (s, 2H), 3.81 (s, 1H), 2.16 (s, 6H).

N-(4-chlorobenzyl)-4-fluoroaniline (35): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.17 (m, 4H), 6.82 – 6.75 (m, 2H), 6.48 – 6.41 (m, 2H), 4.17 (s, 2H), 3.83 (s, 1H).

N-(4-chlorobenzyl)-4-methoxyaniline (36): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (s, 4H), 6.71 – 6.67 (m, 2H), 6.52 – 6.48 (m, 2H), 4.18 (s, 2H), 3.66 (s, 3H).

N-(4-fluorobenzyl)-4-methylaniline (37): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (m, 2H), 6.94 – 6.86 (m, 4H), 6.44 (d, *J* = 8.4 Hz, 2H), 4.15 (s, 2H), 3.64 (s, 1H), 2.14 (s, 3H).

N-(4-fluorobenzyl)-4-methoxyaniline (38): ^{1}H NMR ($CDCl_3$) δ 7.21 (m, 5.5 Hz, 2H), 6.91 (t, *J* = 8.7 Hz, 2H), 6.70 – 6.64 (m, 2H), 6.50 – 6.45 (m, 2H), 4.13 (s, 2H), 3.62 (s, 3H), 3.42 (s, 1H).

4-fluoro-N-(4-fluorobenzyl) aniline (39): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (m, 2H), 6.92 (t, *J* = 8.6 Hz, 2H), 6.81 – 6.74 (m, 2H), 6.47 – 6.40 (m, 2H), 4.14 (s, 2H), 3.65 (s, 1H).

N-(4-chlorobenzyl)-3-methoxyaniline (40): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (s, 4H), 6.96 (t, *J* = 7.7 Hz, 1H), 6.46 (d, *J* = 7.4 Hz, 1H), 6.38 – 6.26 (m, 2H), 4.18 (s, 2H), 3.86 (s, 1H), 2.17 (s, 3H).

N-(4-fluorobenzyl)-4-(trifluoromethoxy) aniline (41): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (m, 2H), 6.94 (m, 4H), 6.50 – 6.44 (m, 2H), 4.18 (s, 2H), 4.07 – 3.57 (m, 1H).

N-(4-fluorobenzyl)-3-methoxyaniline (42): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 2H), 7.00 – 6.89 (m, 3H), 6.20 (dd, *J* = 7.9, 2.5 Hz, 1H), 6.14 (dd, *J* = 7.6, 1.8 Hz, 1H), 6.09 – 6.05 (m, 1H), 4.17 (s, 2H), 4.06 – 3.77 (m, 1H), 3.64 (s, 3H).

N-(4-fluorobenzyl)-2-methoxyaniline (43): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (m, 2H), 6.91 (t, *J* = 8.7 Hz, 2H), 6.76 – 6.66 (m, 2H), 6.62 – 6.56 (m, 1H), 6.49 – 6.43 (m, 1H), 4.52 (s, 1H), 4.19 (s, 2H), 3.74 (s, 3H).

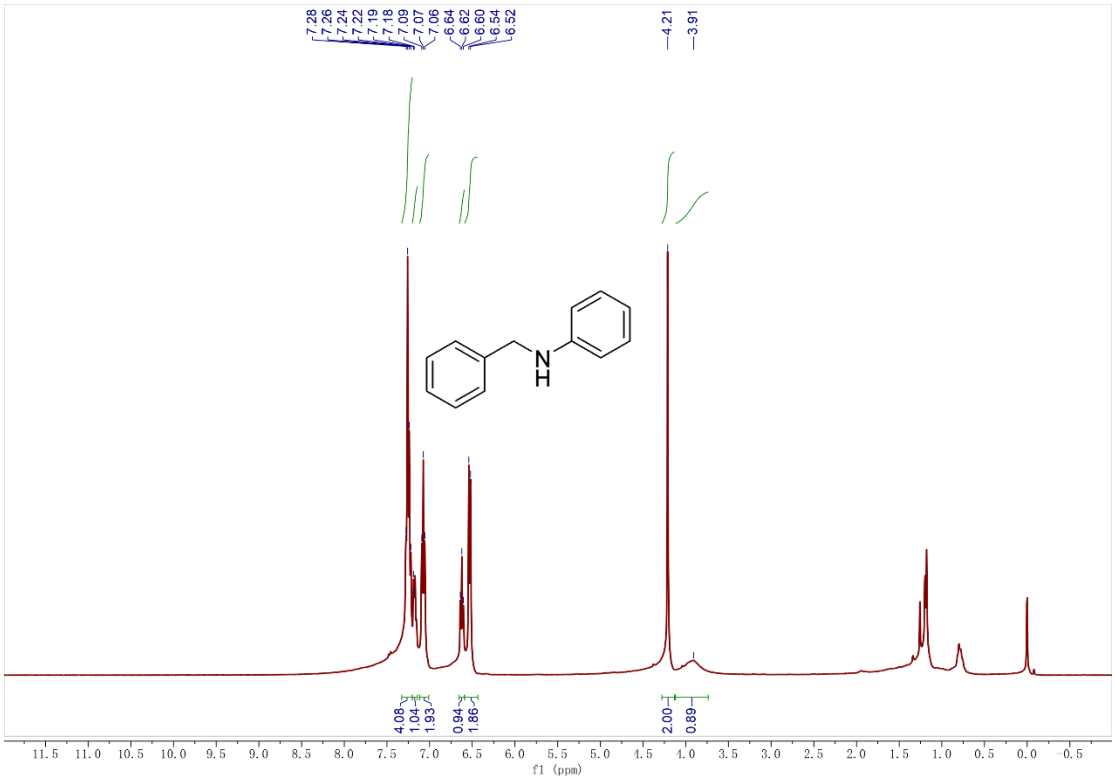
N-(4-fluorobenzyl)-3-methylaniline (44): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (m, 2H), 7.00 – 6.84 (m, 3H), 6.46 (d, *J* = 7.4 Hz, 1H), 6.38 – 6.27 (m, 2H), 4.16 (s, 2H), 3.77 (s, 1H), 2.17 (s, 3H).

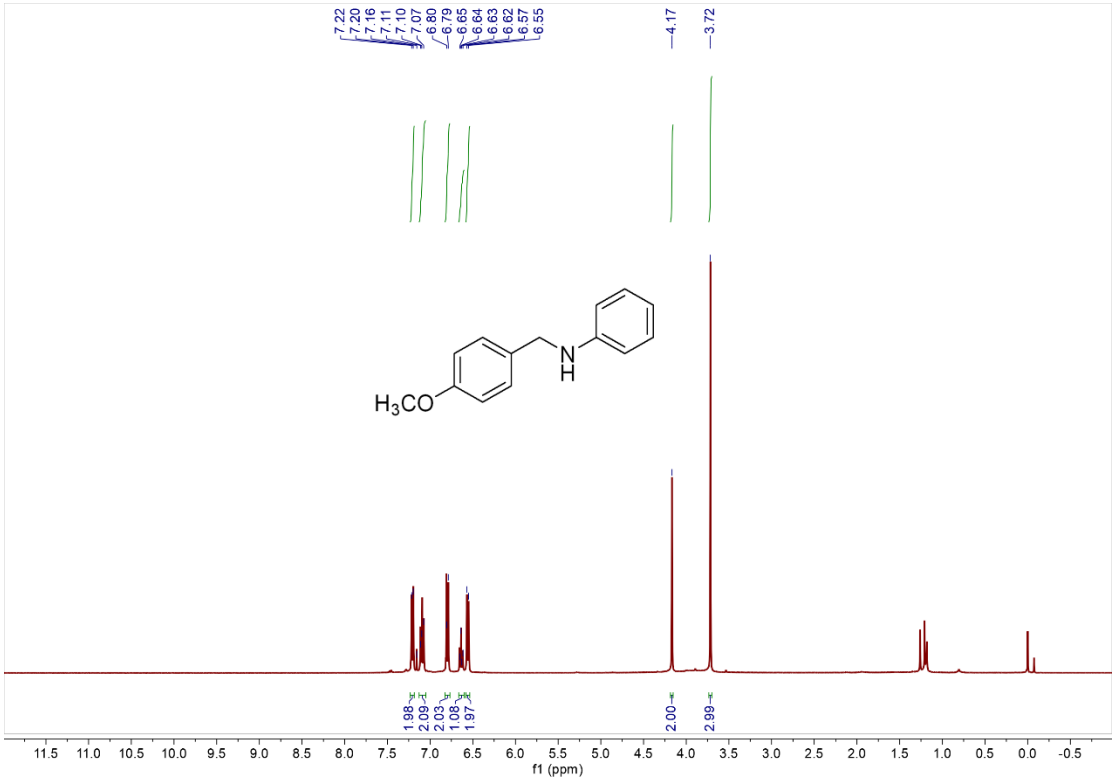
N-(4-fluorobenzyl)-3,5-dimethylaniline (45): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (m, 2H), 6.95 – 6.87 (m, 2H), 6.30 (s, 1H), 6.17 (s, 2H), 4.16 (s, 2H), 3.74 (s, 1H), 2.13 (s, 6H).

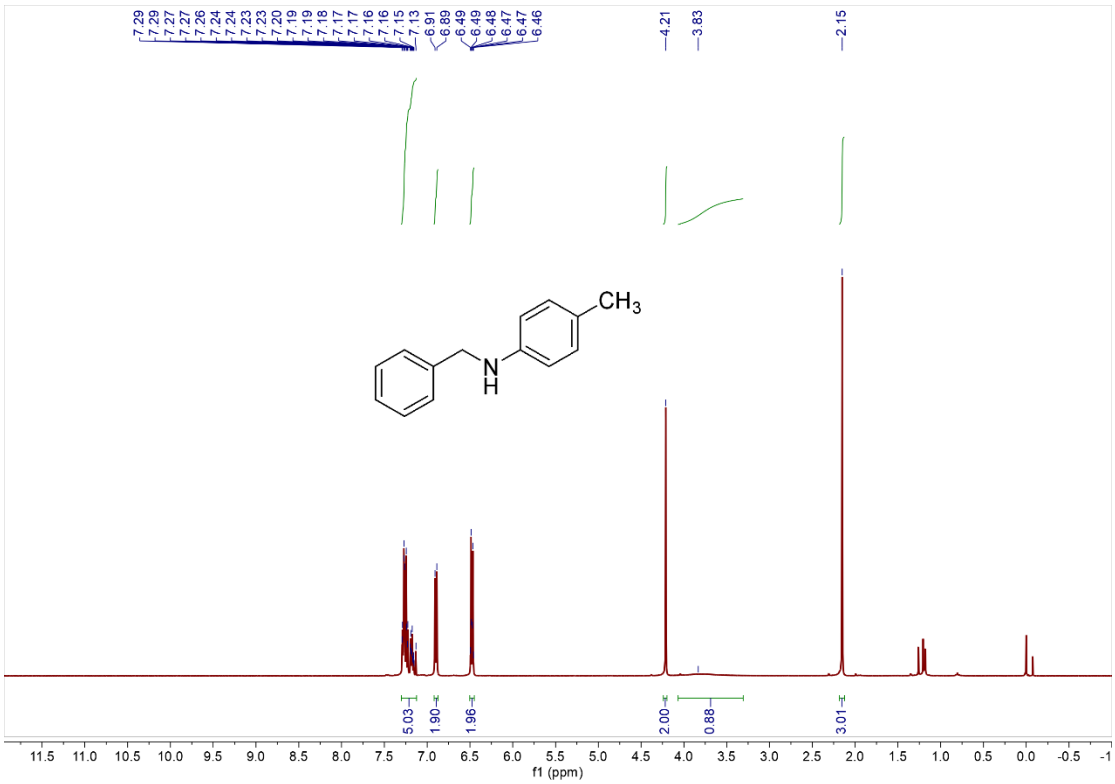
4-methoxy-N-(4-methylbenzyl) aniline (46): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 7.9 Hz, 2H), 7.04 (d, *J* = 7.9 Hz, 2H), 6.70 – 6.65 (m, 2H), 6.51 – 6.47 (m, 2H), 4.12 (s, 2H), 3.63 (s, 3H), 3.38 (s, 1H), 2.24 (s, 3H).

N-(4-methylbenzyl) aniline (47): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 7.8 Hz, 2H), 7.12 – 7.01 (m, 4H), 6.65 – 6.58 (m, 1H), 6.53 (d, *J* = 8.5 Hz, 2H), 4.17 (s, 2H), 3.86 (s, 1H), 2.25 (s, 3H).

S3. NMR Image of the Products



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7.16
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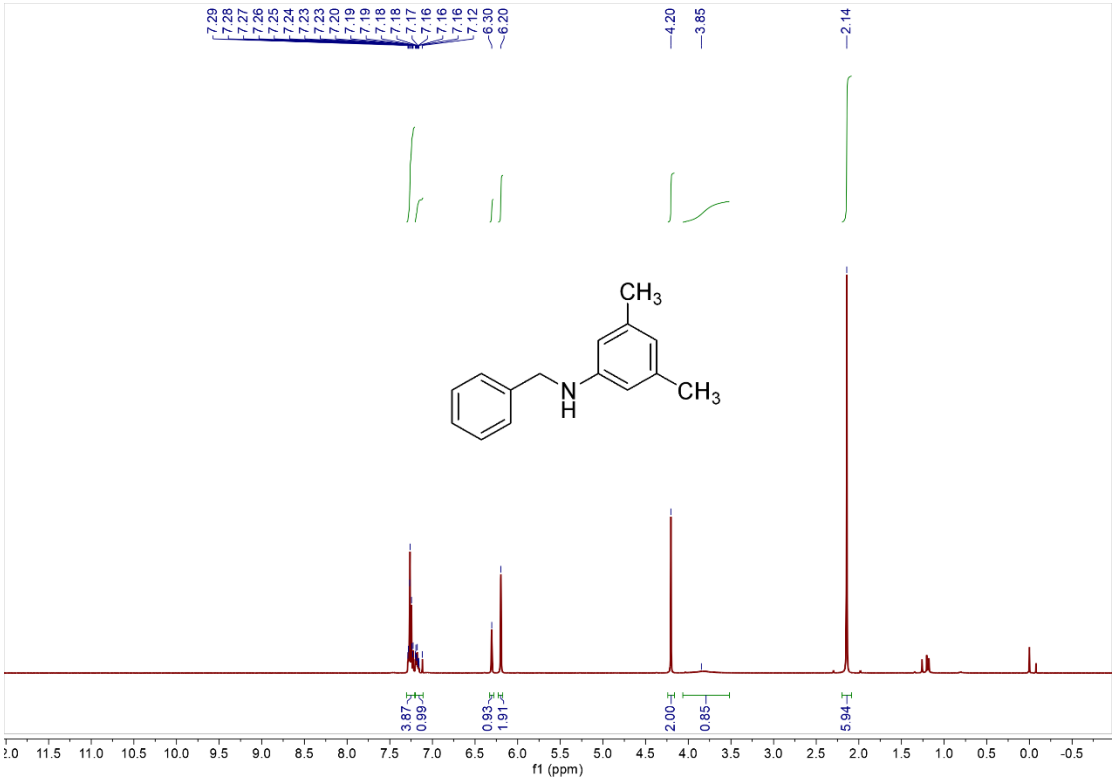
7.13
6.91
6.89
6.49
6.49
6.48
6.47
6.47
6.46
4.21
3.83
2.15
CH3
N
H
5.03
1.90
1.96
2.00
0.88
3.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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2.14
CH3
CH3
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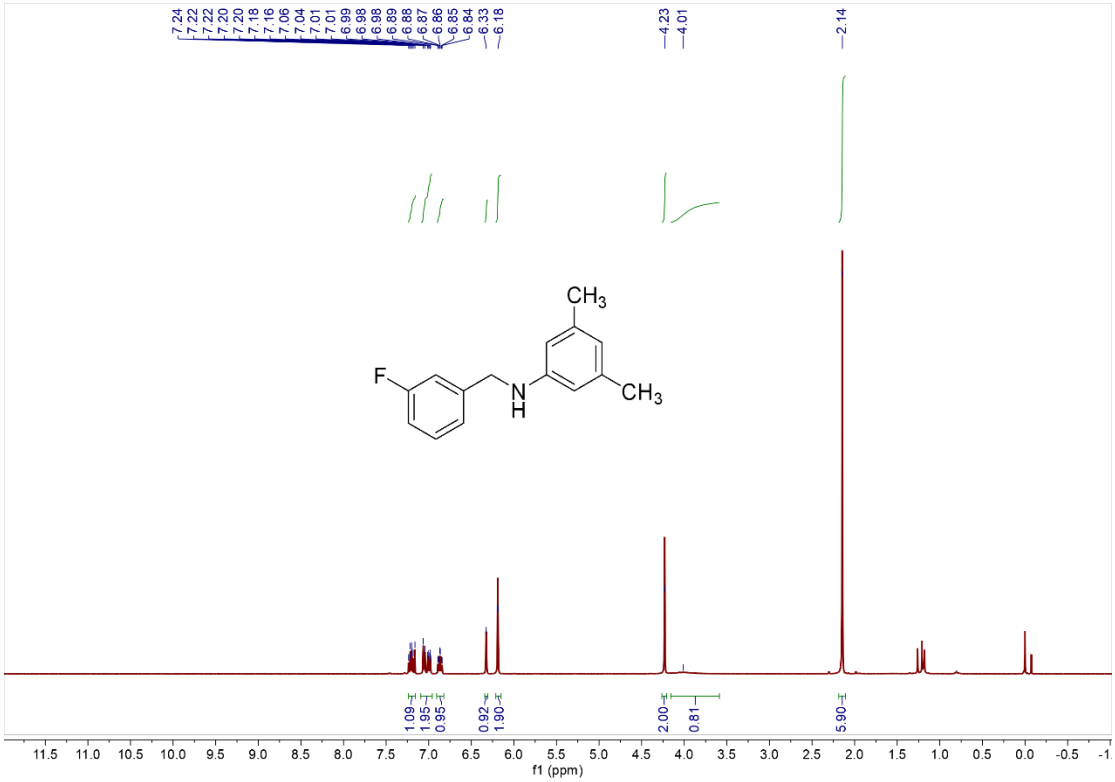
0.99
0.93
1.91
2.00
0.85
5.94
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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4.01
2.14
CH3
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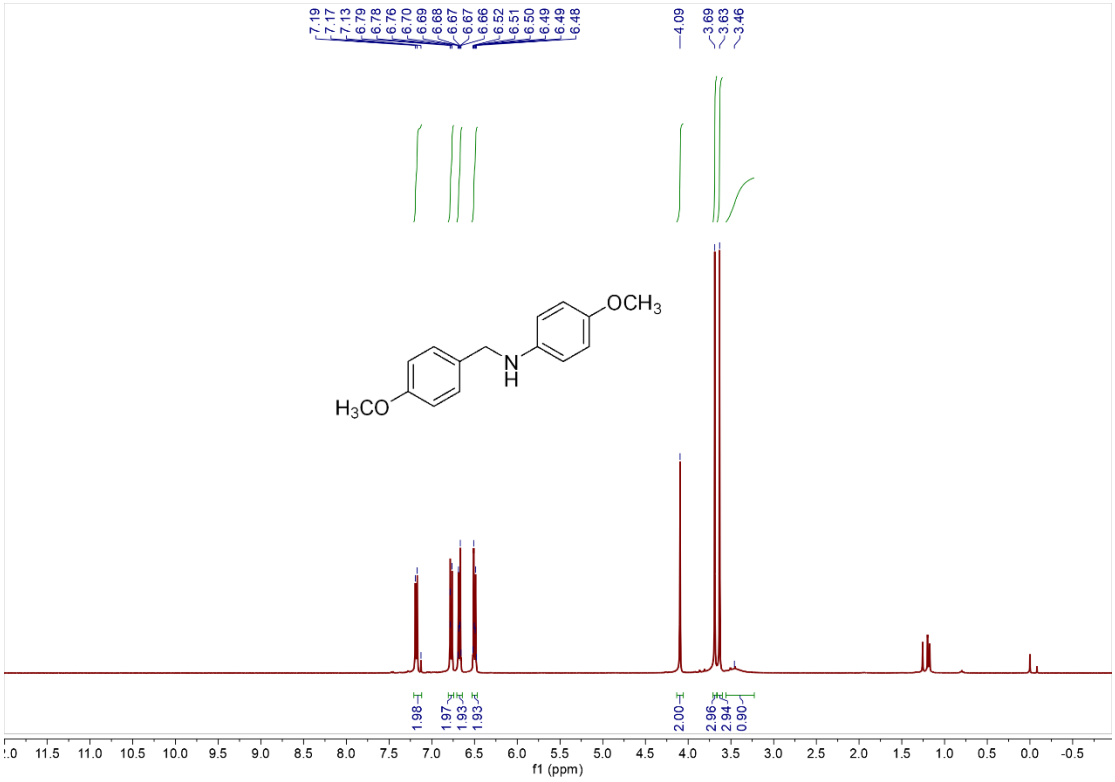
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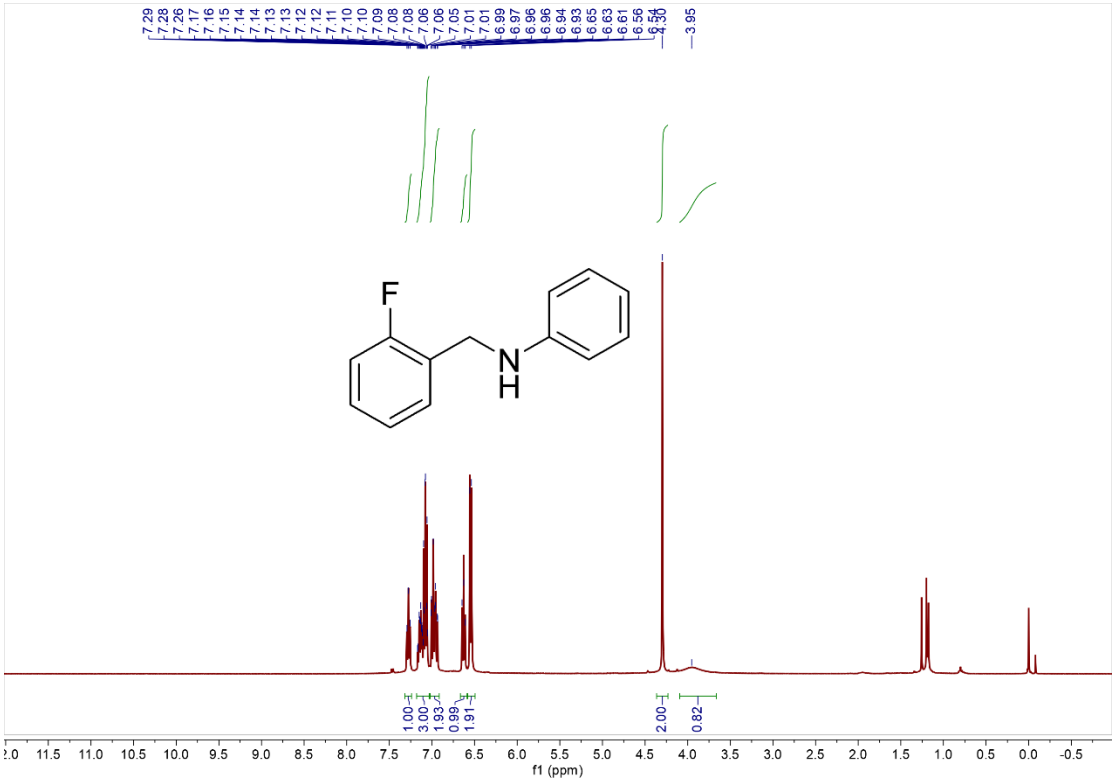
0.90
f1 (ppm)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6.61
6.56
6.54
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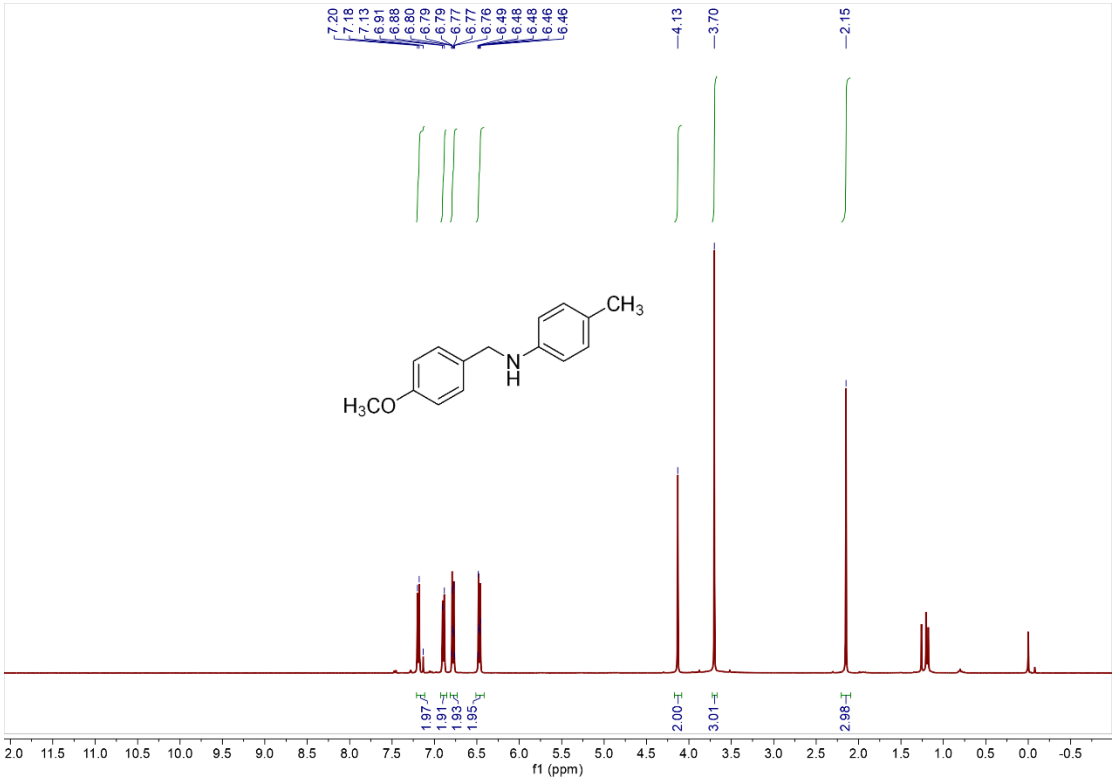
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6.77
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6.48
6.46
6.46
4.13
3.70
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H
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1.91
1.93
1.95
2.00
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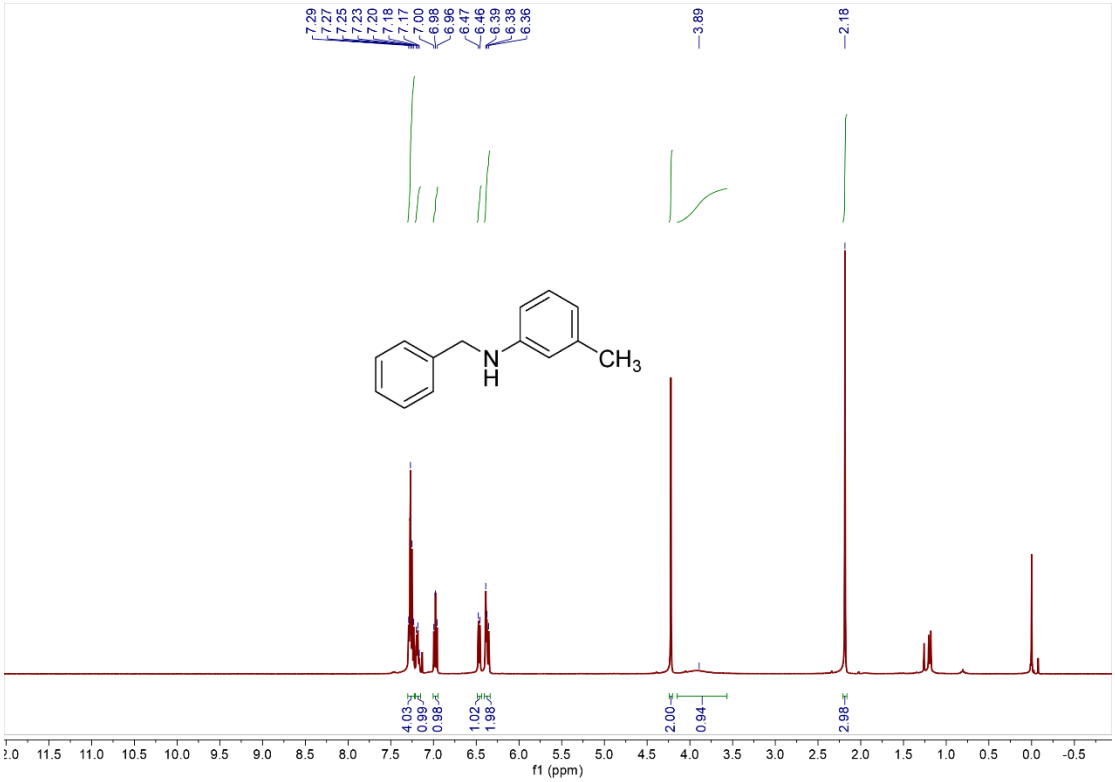
2.98
f1 (ppm)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6.36
3.89
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CH3
4.03
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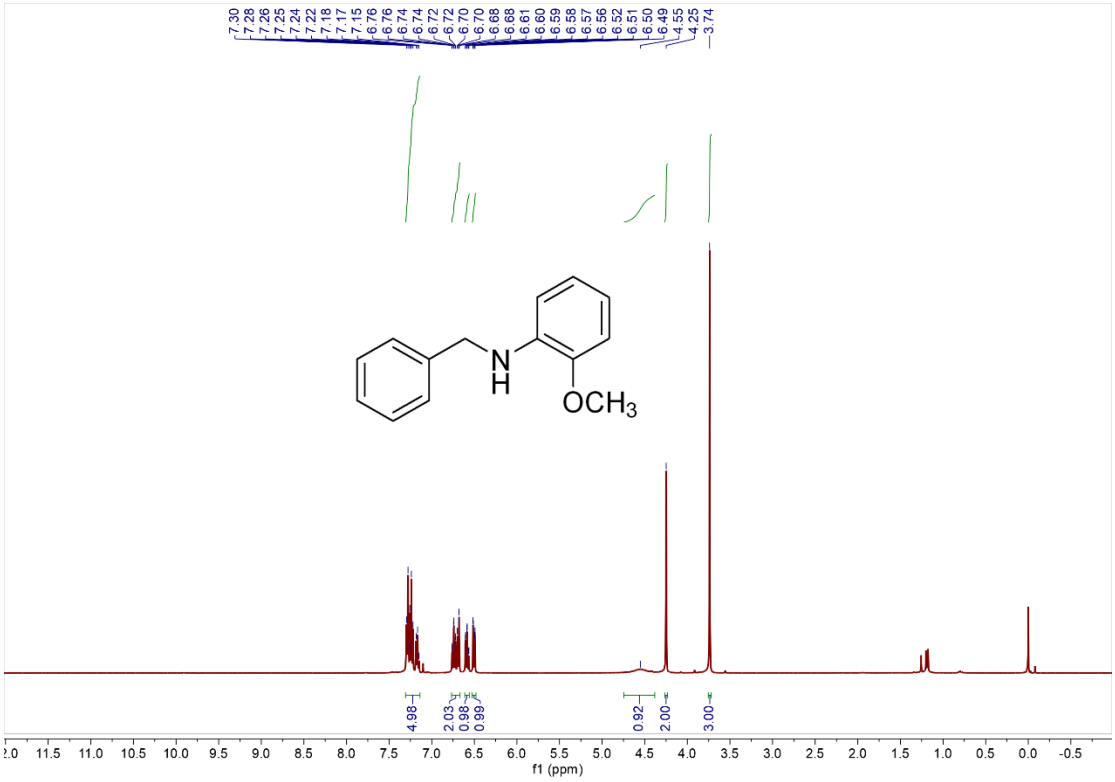
0.98
1.02
1.98
2.00
0.94
2.98
f1 (ppm)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6.52
6.51
6.50
6.49
4.55
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OCH3
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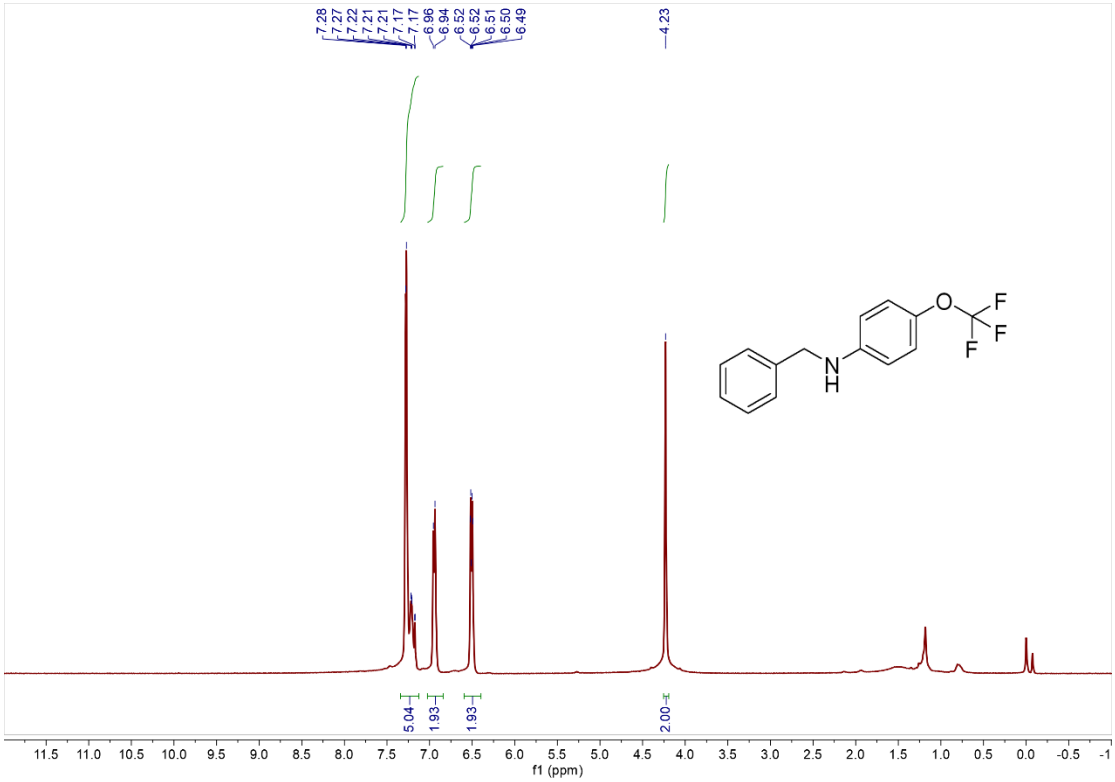
2.03
0.98
0.99
0.92
2.00
3.00
f1 (ppm)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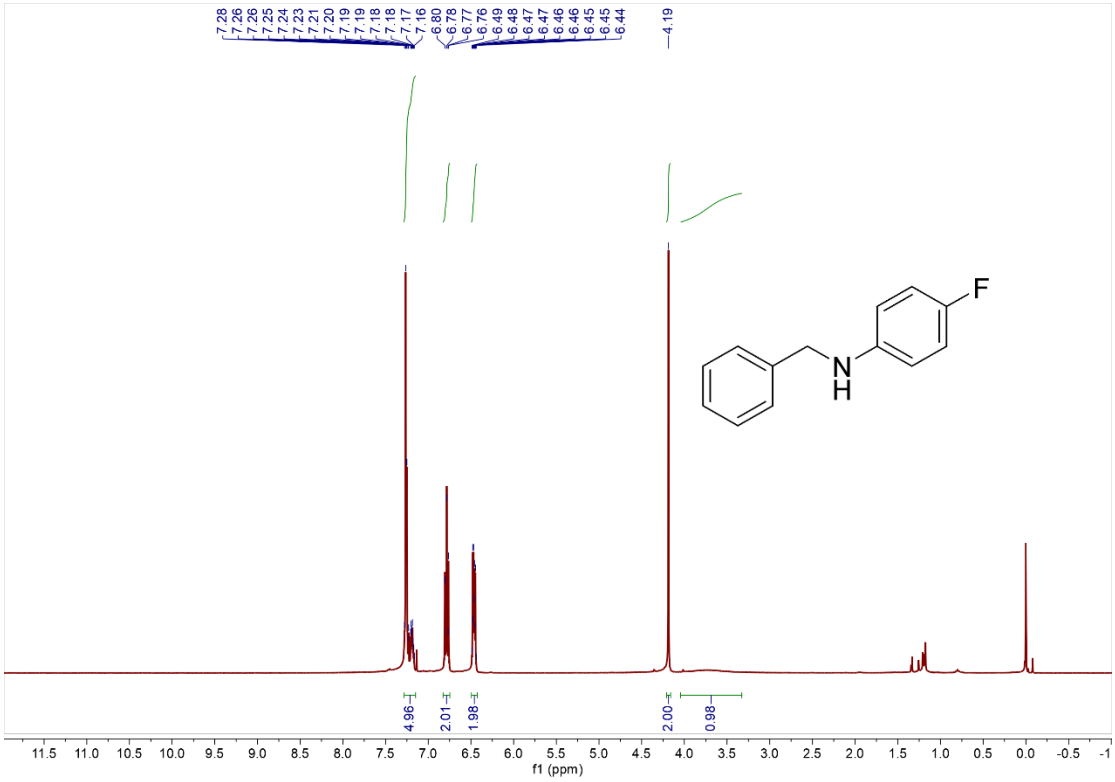
6.50
6.49
4.23
5.04
1.93
1.93
2.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)
O
F
F
F
N
H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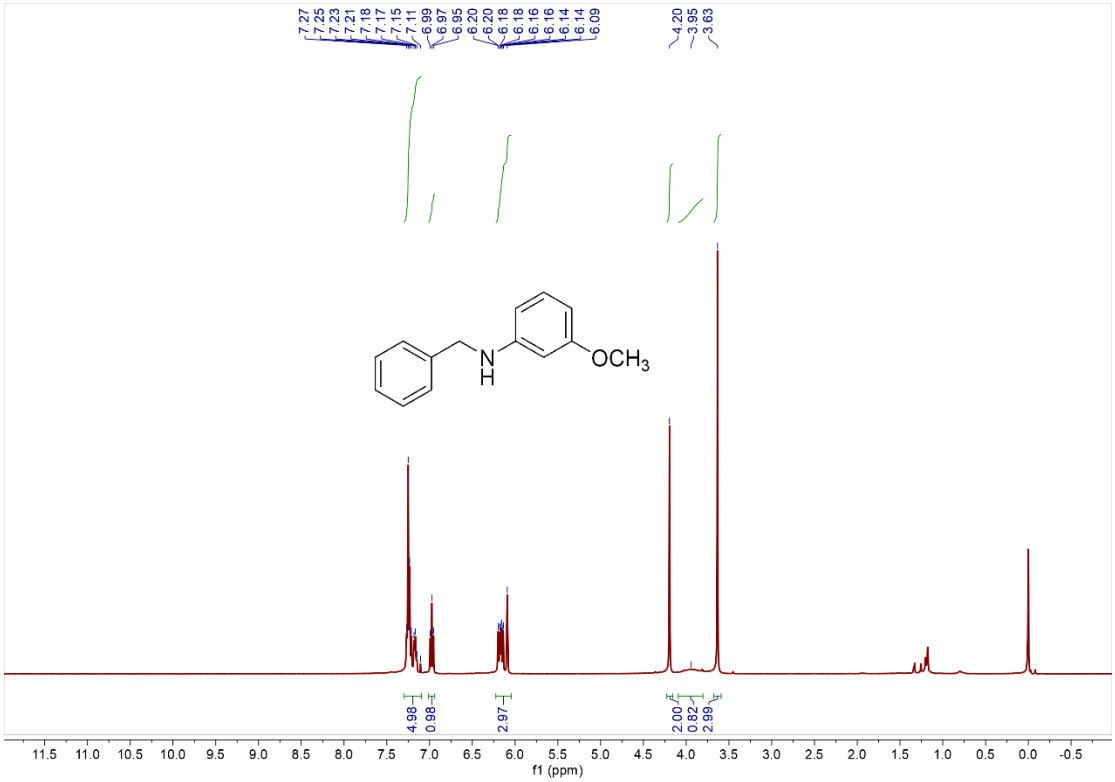
6.47
6.46
6.46
6.45
6.44
4.19
4.96
2.01
1.98
2.00
0.98
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)
F
N
H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6.97
6.95
6.20
6.18
6.16
6.14
6.09
4.20
3.95
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N
H
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4.98
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2.97
2.00
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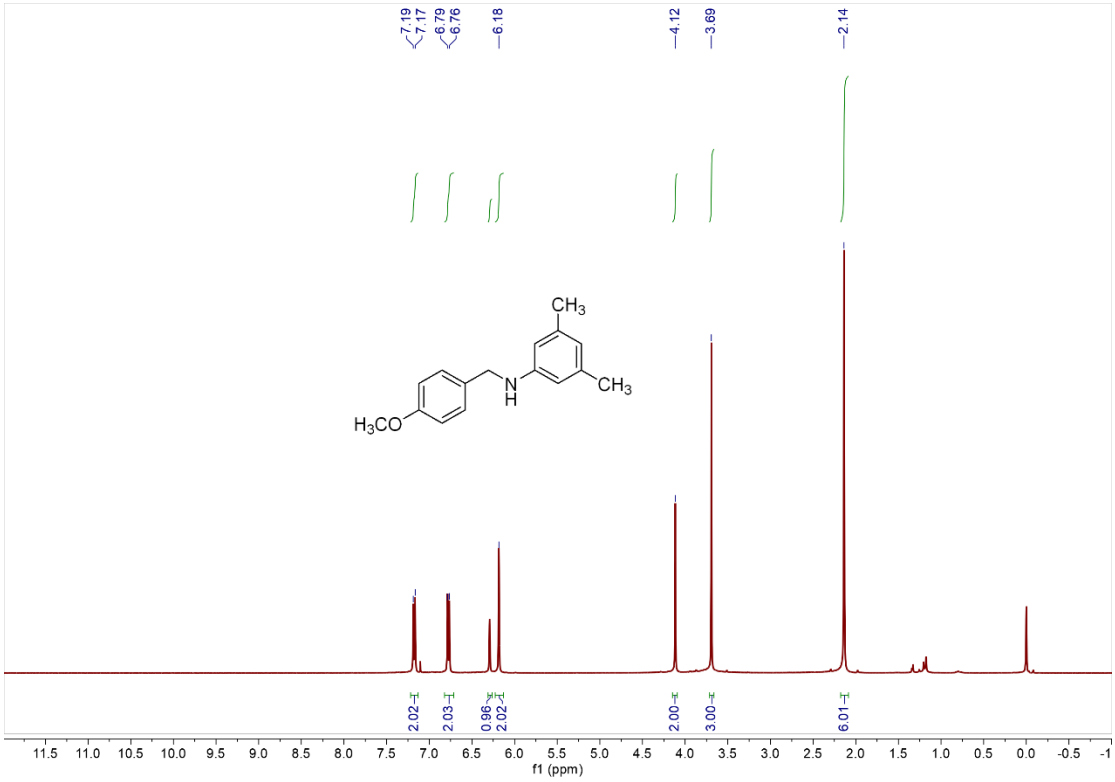
2.99
f1 (ppm)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CH3
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H
H3CO
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2.03
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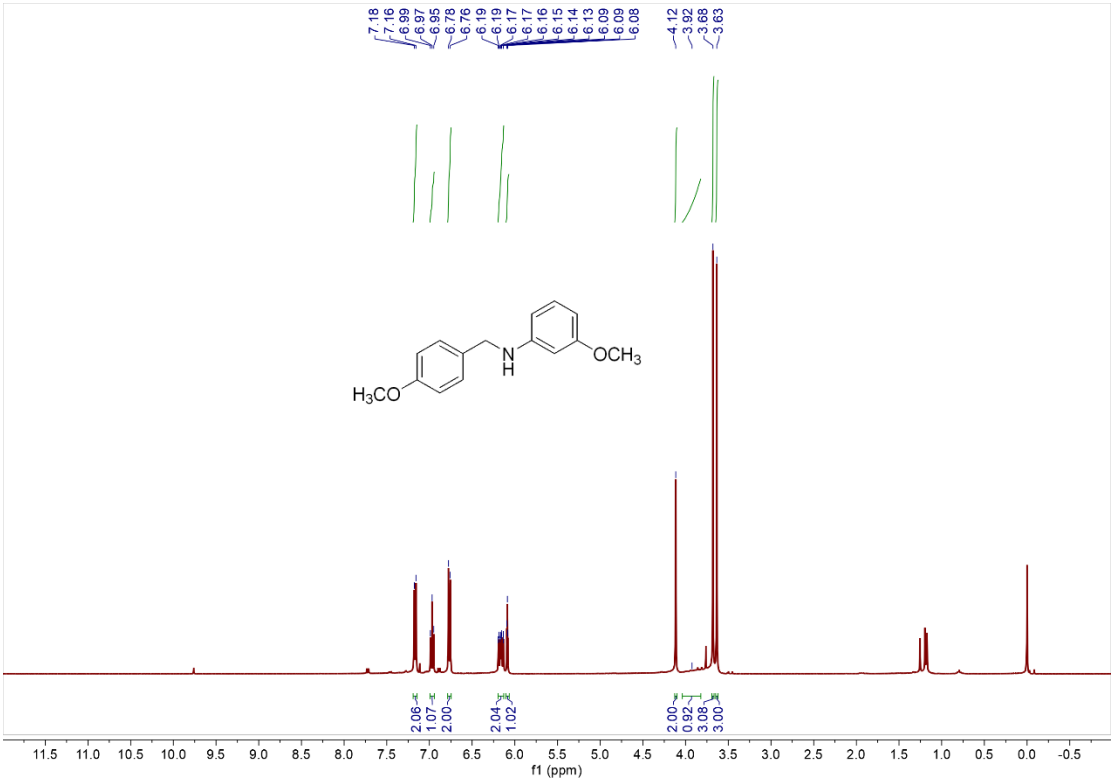
2.02
2.00
3.00
6.01
f1 (ppm)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6.13
6.09
6.08
4.12
3.92
3.68
3.63
N
H
OCH3
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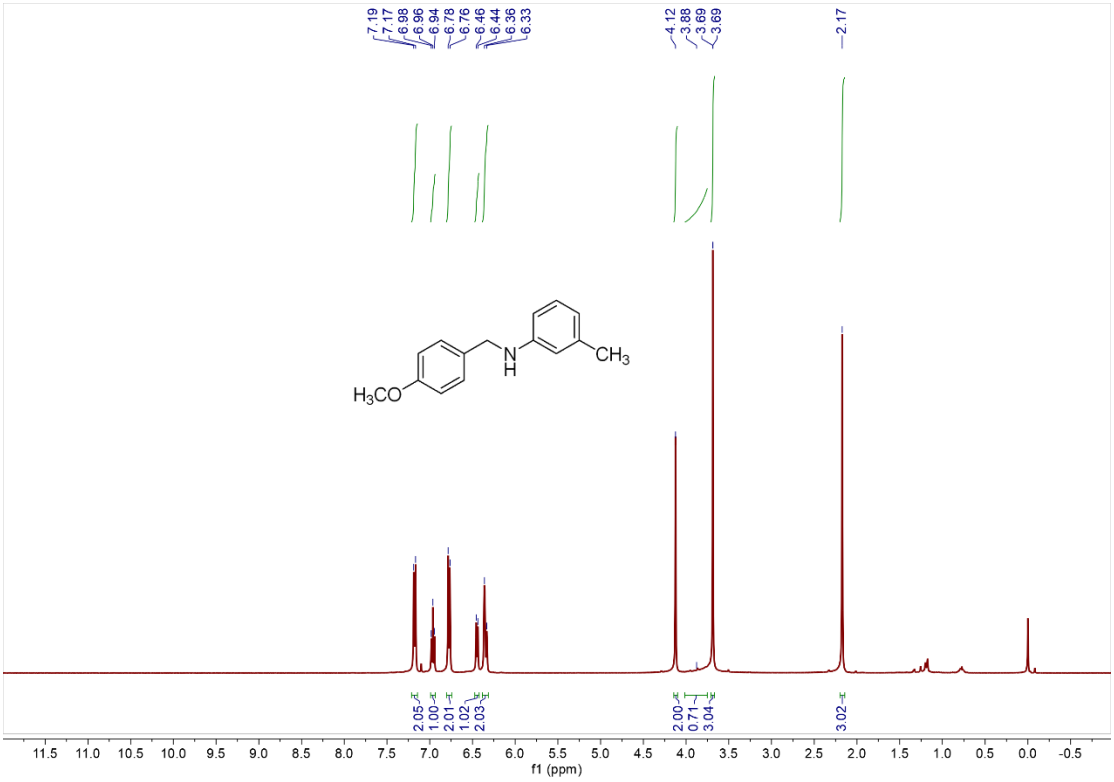
0.92
3.08
3.00
f1 (ppm)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3.69
3.69
2.17
N
H
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H3CO
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2.01
1.02
2.03
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0.71
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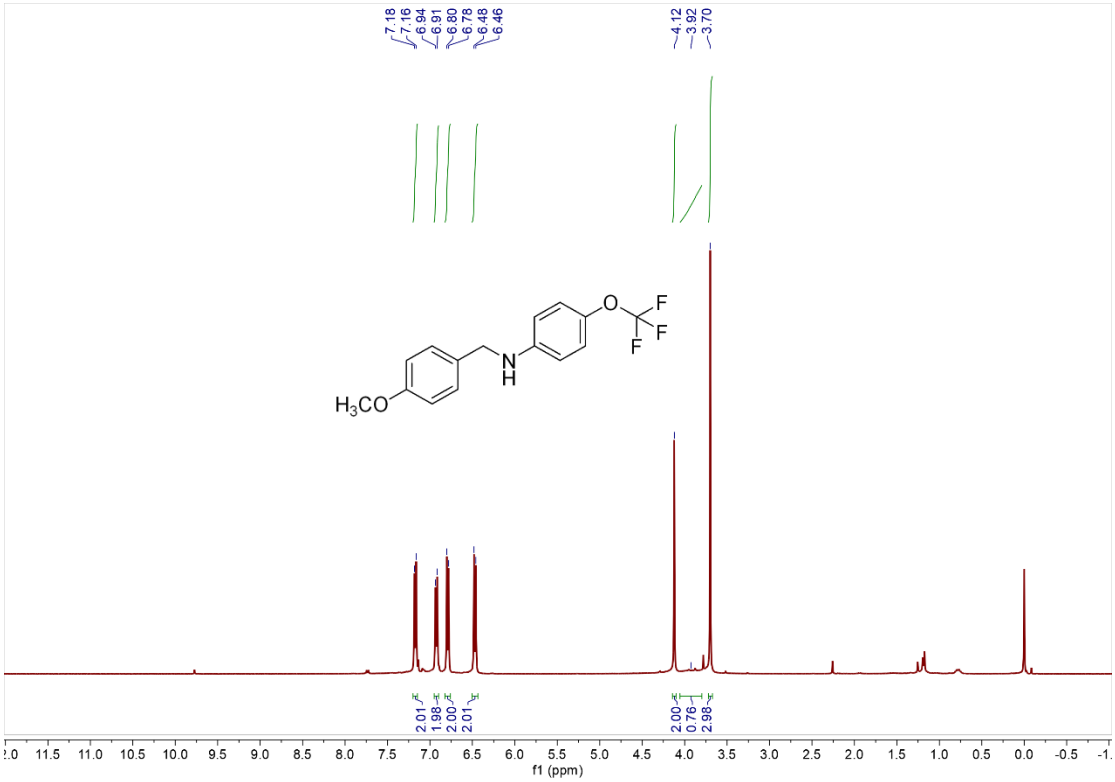
3.02
f1 (ppm)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4.12
3.92
3.70
O
F
F
F
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H
H3CO
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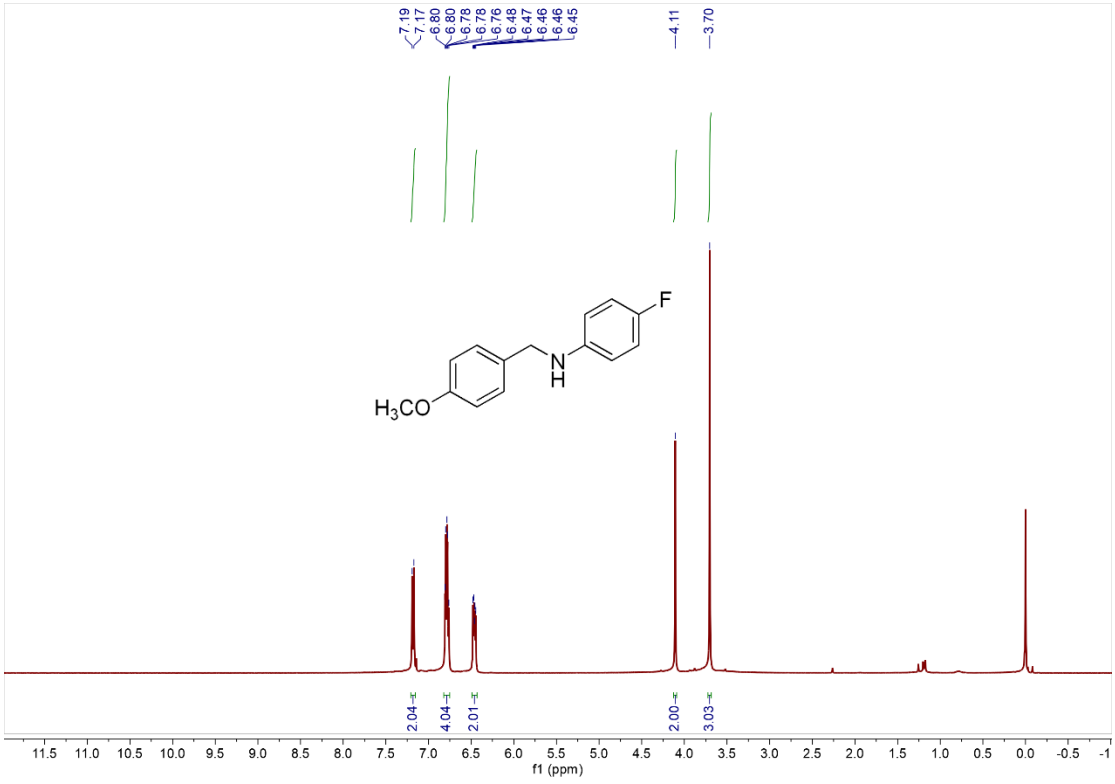
1.98
2.00
2.01
2.00
0.78
2.98
f1 (ppm)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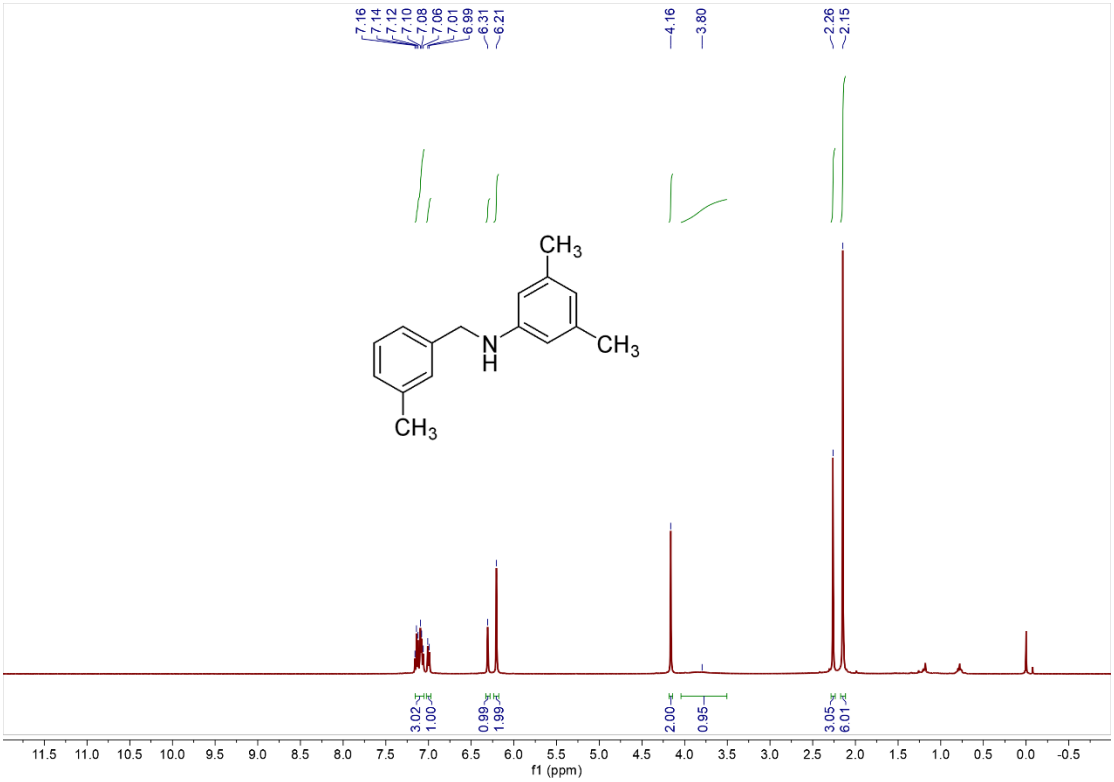
F
N
H
H3CO
2.04
4.04
2.01
2.00
3.03
f1 (ppm)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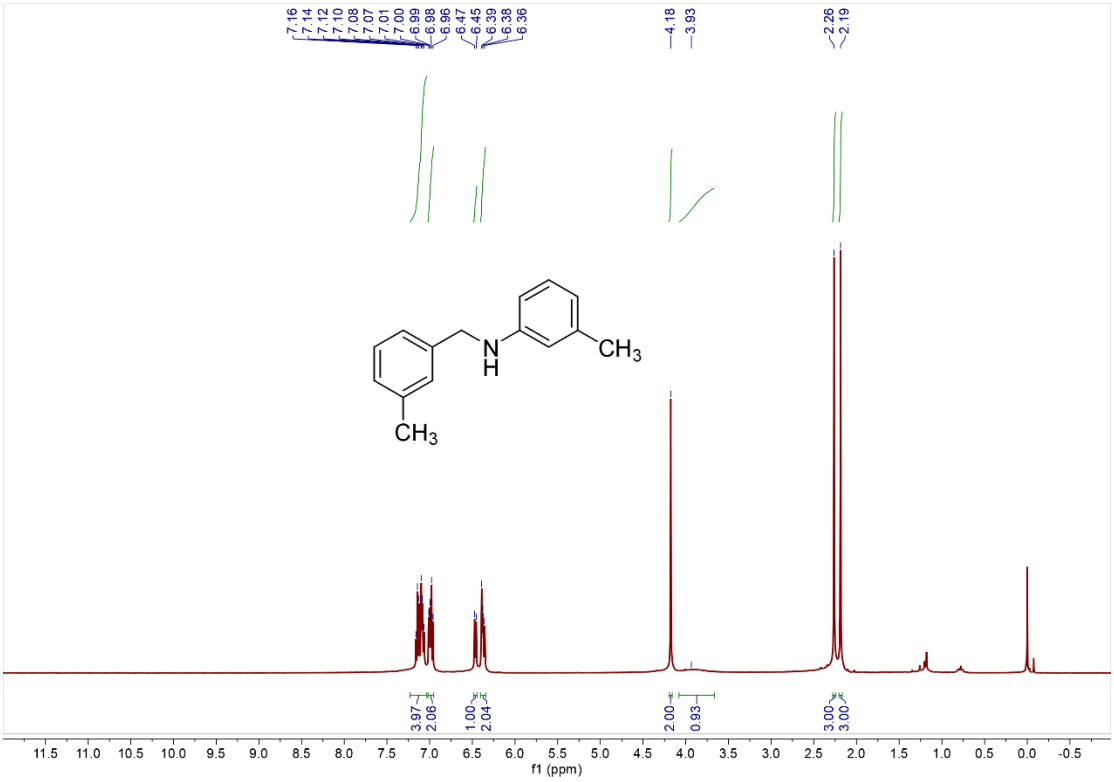
2.15
CH3
N
H
CH3
CH3
3.02
1.00
0.99
1.99
2.00
0.95
3.05
6.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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6.38
6.36
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3.93
2.26
2.19
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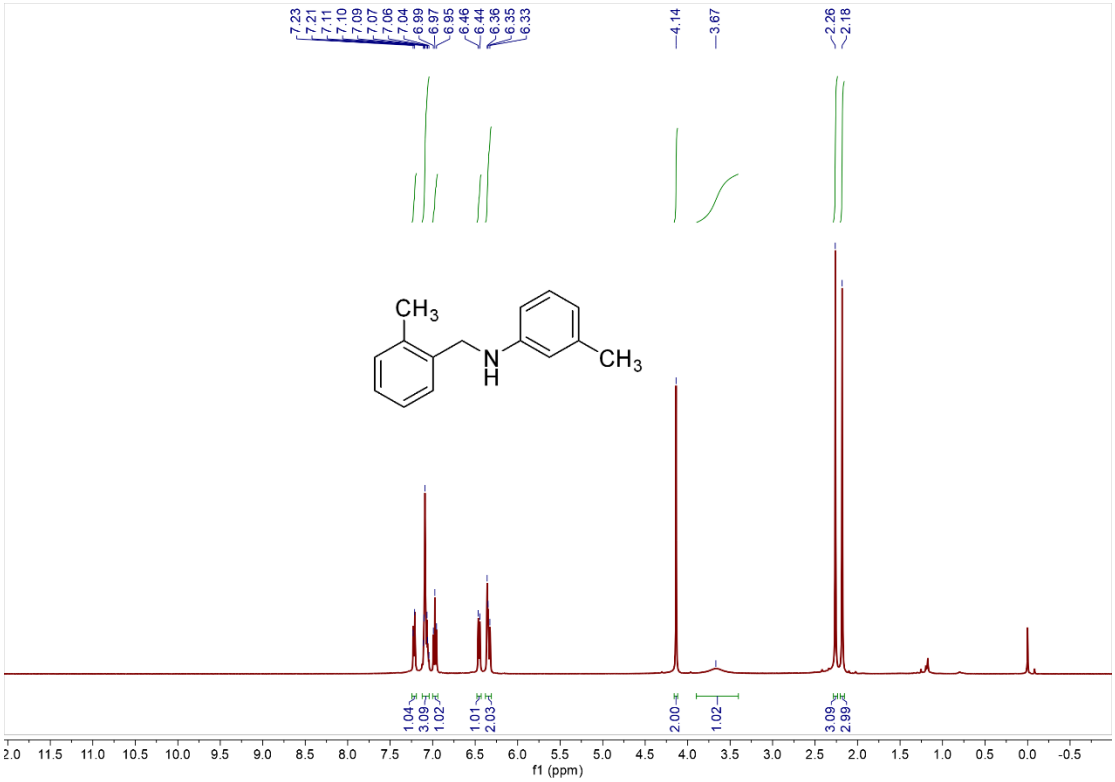
H
CH3
CH3
3.97
2.06
1.00
2.04
2.00
0.93
3.00
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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6.36
6.35
6.33
4.14
3.67
2.26
2.18
CH3
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1.01
2.03
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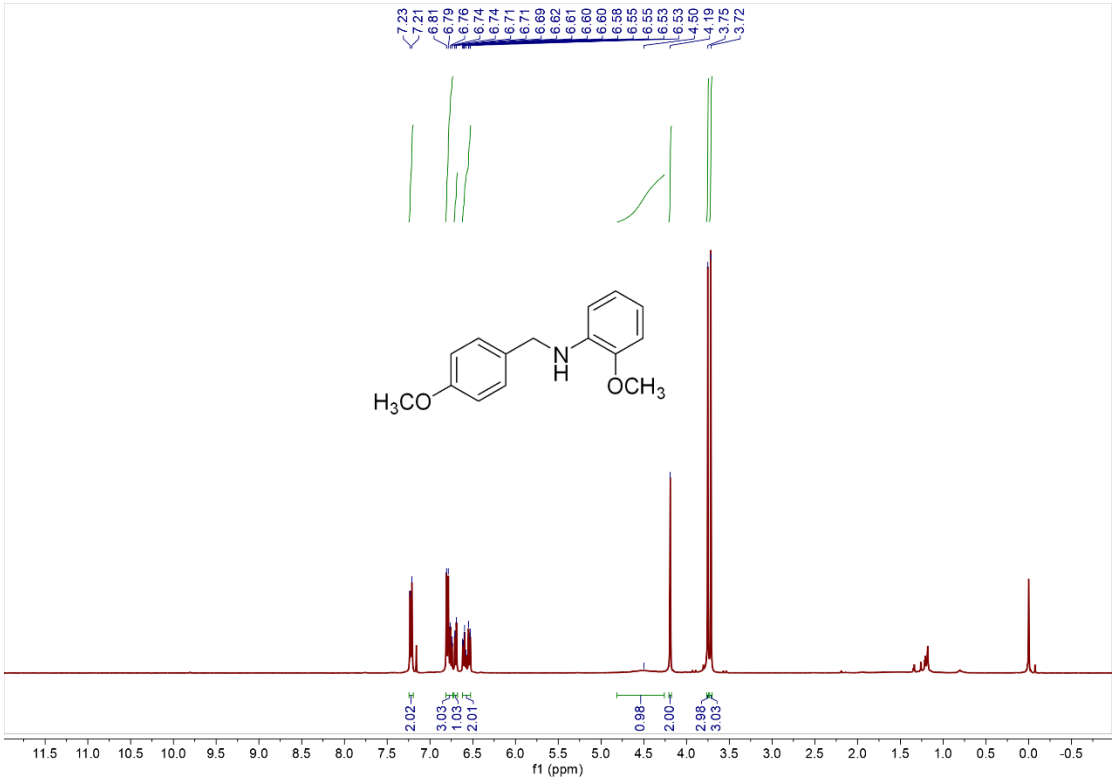
1.02
3.09
2.99
f1 (ppm)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6.53
4.50
4.19
3.75
3.72
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1.03
2.01
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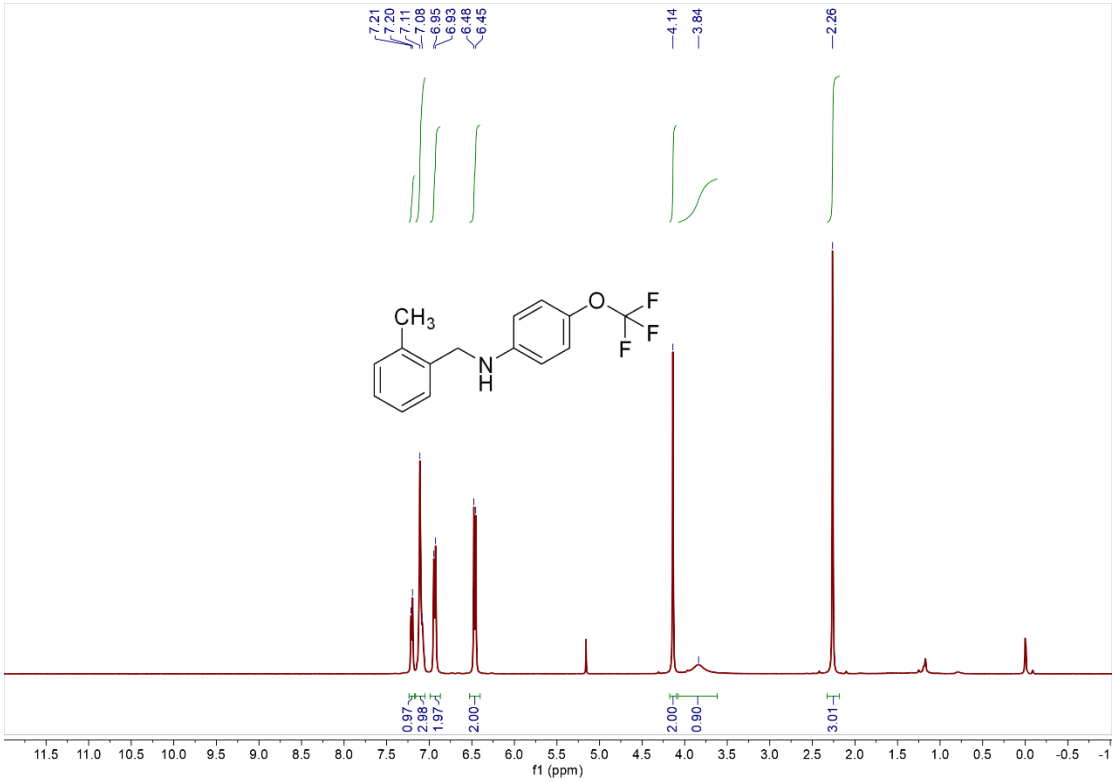
2.00
2.98
3.03
f1 (ppm)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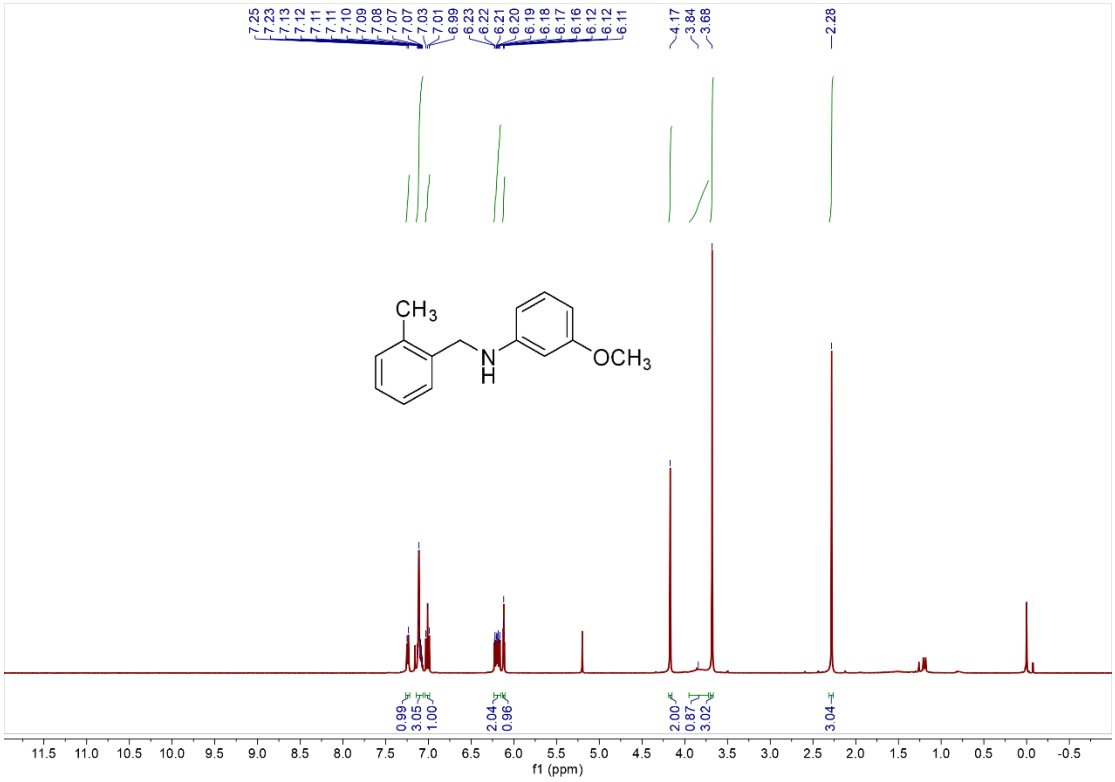
CH3
N
H
O
F
F
F
0.97
2.98
1.97
2.00
2.00
0.90
3.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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2.00
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3.02
3.04
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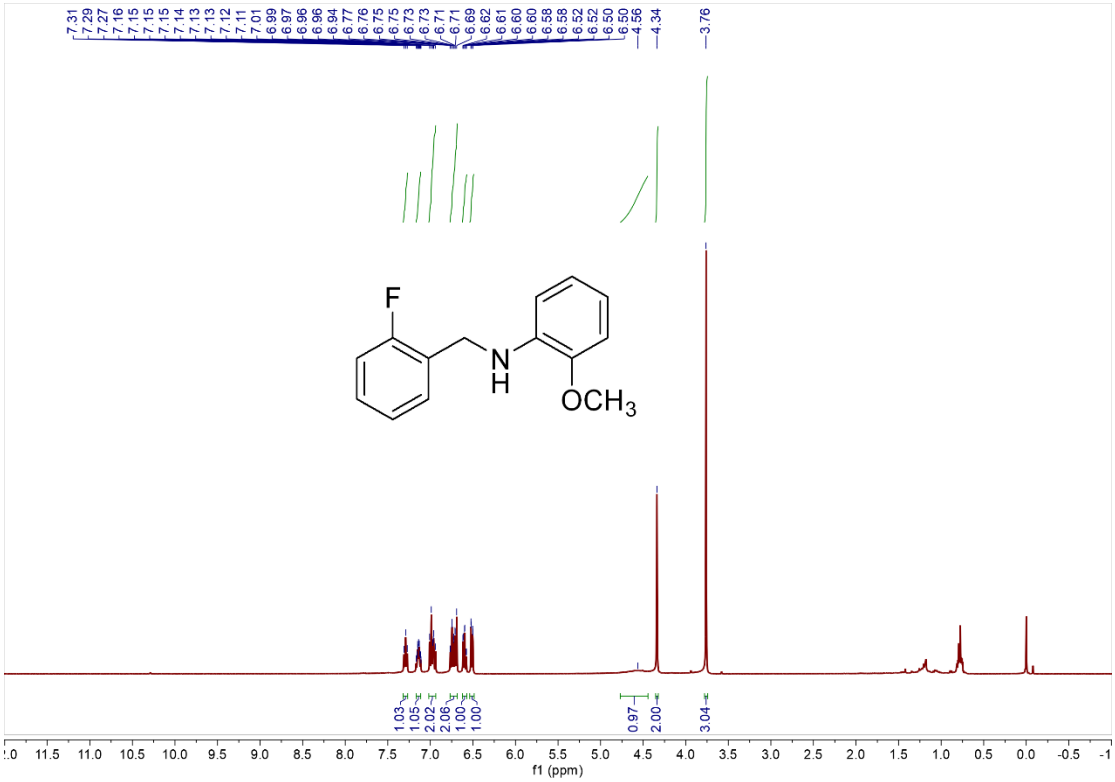
f1 (ppm)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4.56
4.34
3.76
F
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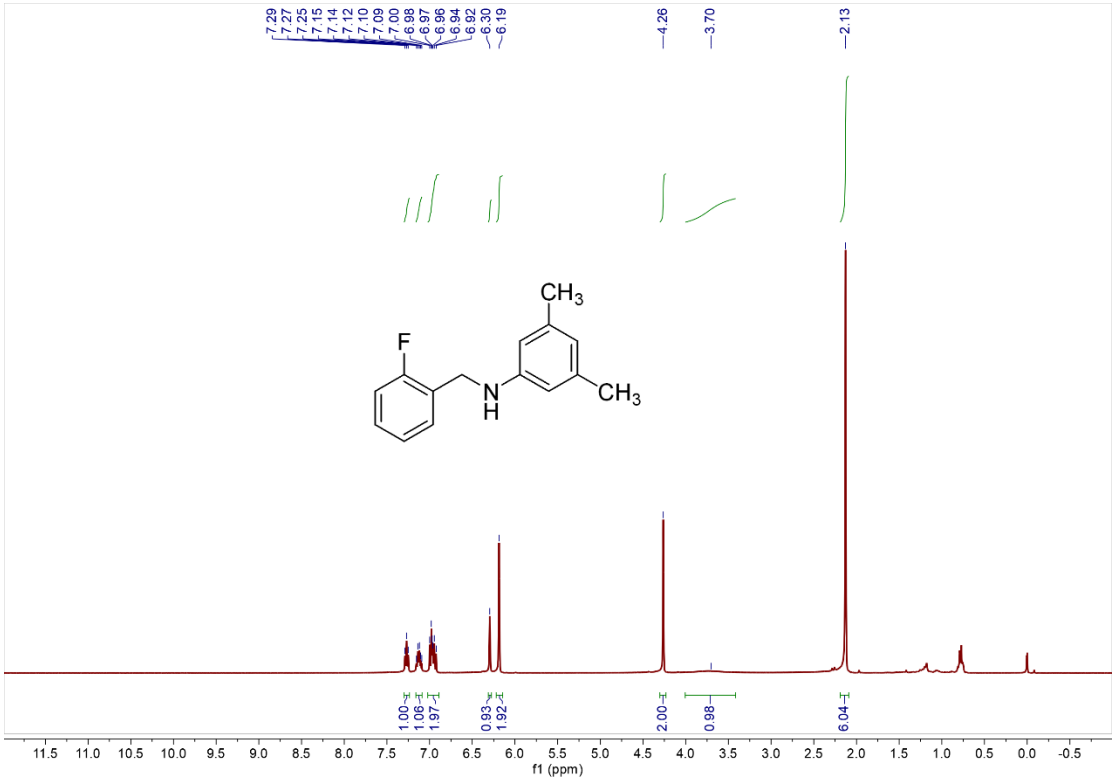
2.02
2.06
1.00
1.00
0.97
2.00
3.04
f1 (ppm)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6.92
6.30
6.19
4.26
3.70
2.13
CH3
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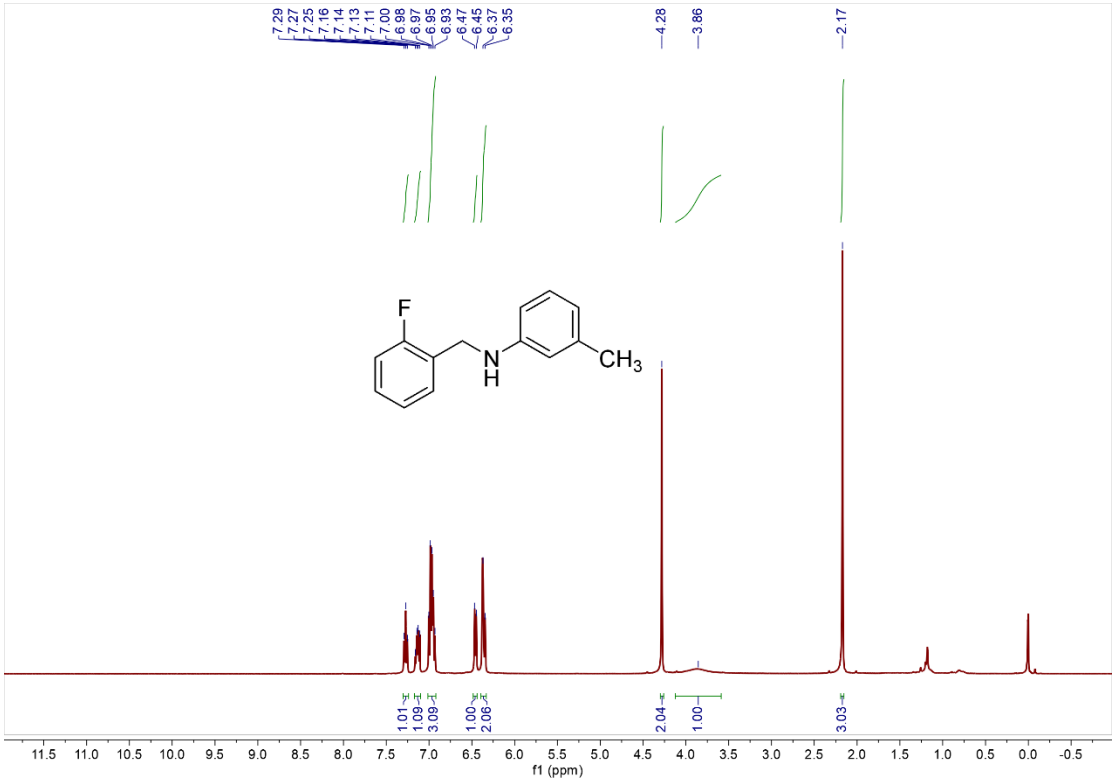
1.06
1.97
0.93
1.92
2.00
0.98
6.04
f1 (ppm)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6.47
6.45
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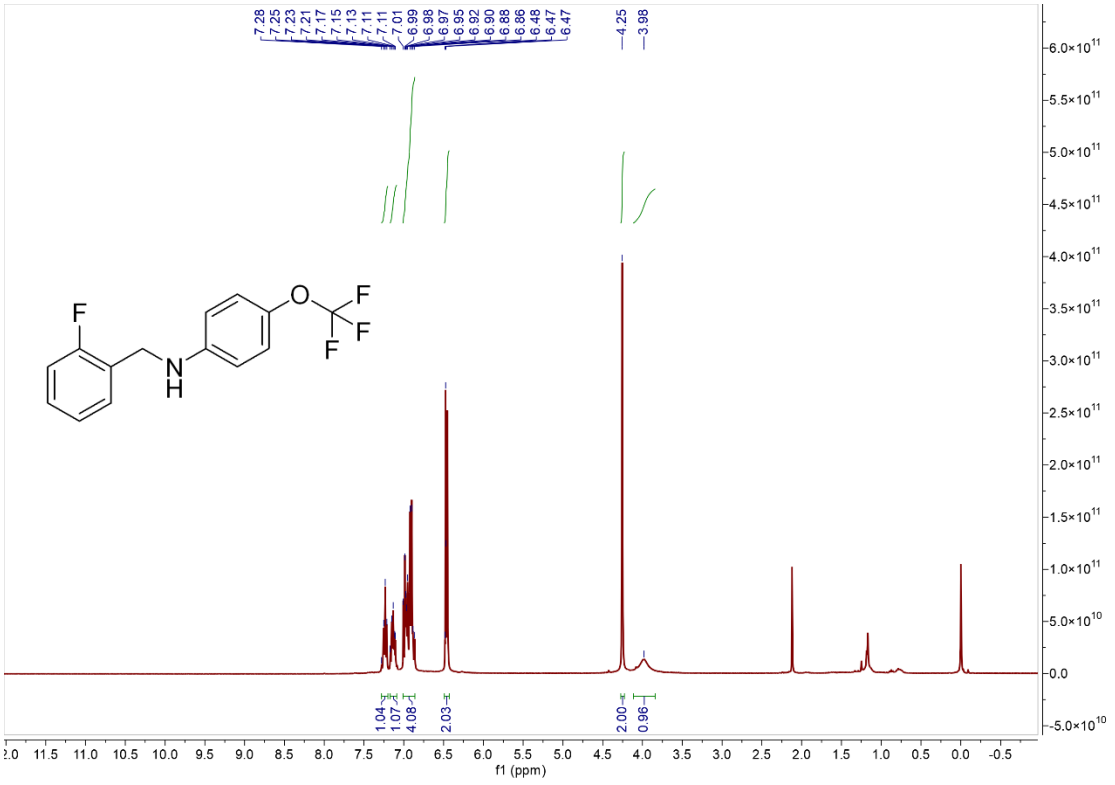
f1 (ppm)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6.92
6.90
6.88
6.86
6.48
6.47
6.47
4.25
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F
F
F
N
H
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1.07
4.08
2.03
2.00
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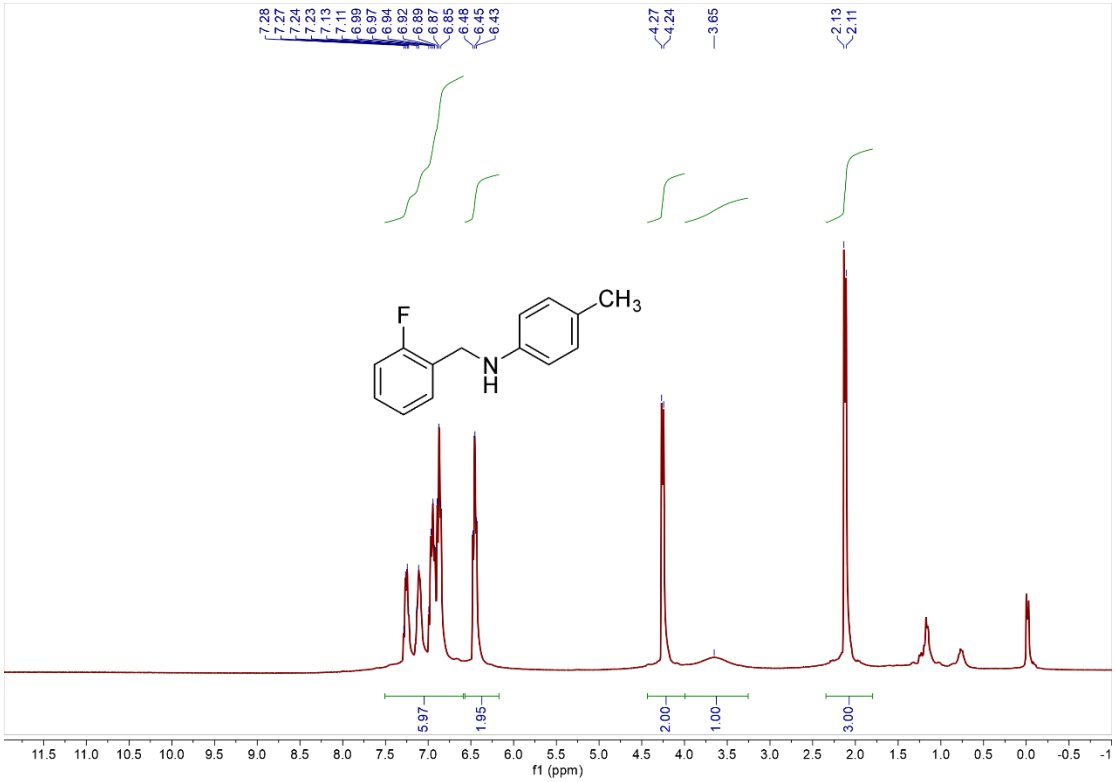
f1 (ppm)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6.45
6.43
4.27
4.24
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CH3
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H
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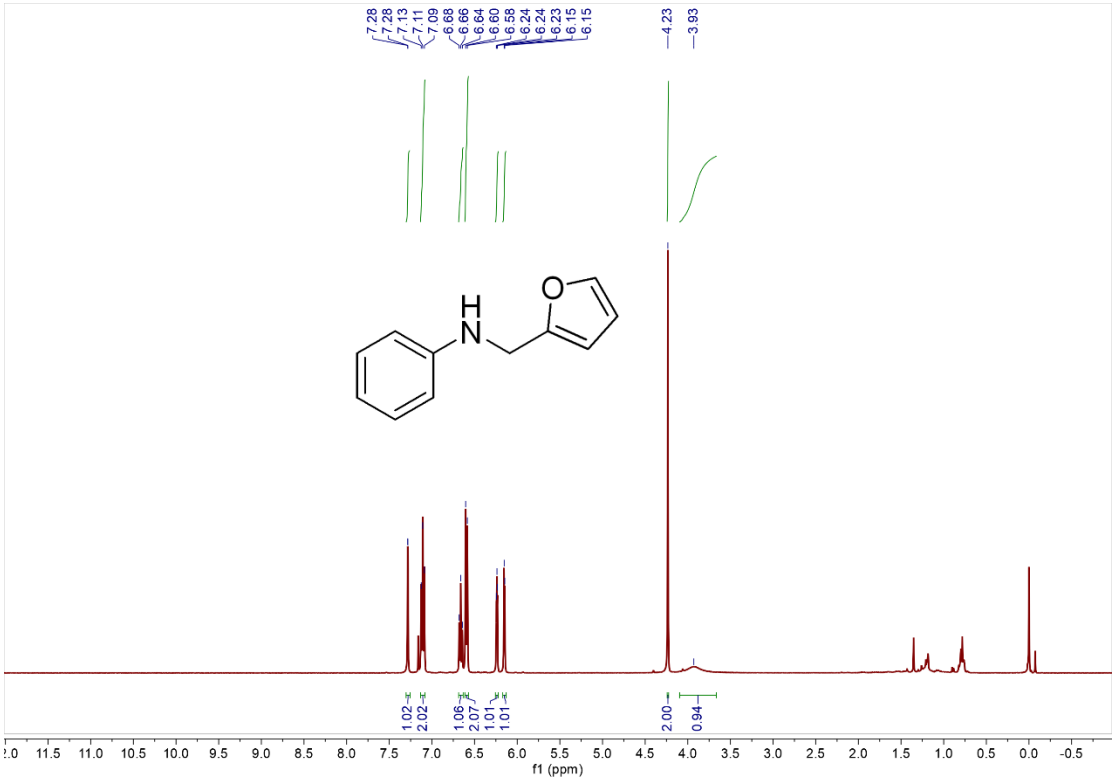
2.00
1.00
3.00
f1 (ppm)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6.23
6.15
6.15
4.23
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O
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2.02
1.06
2.07
1.01
1.01
2.00
0.94
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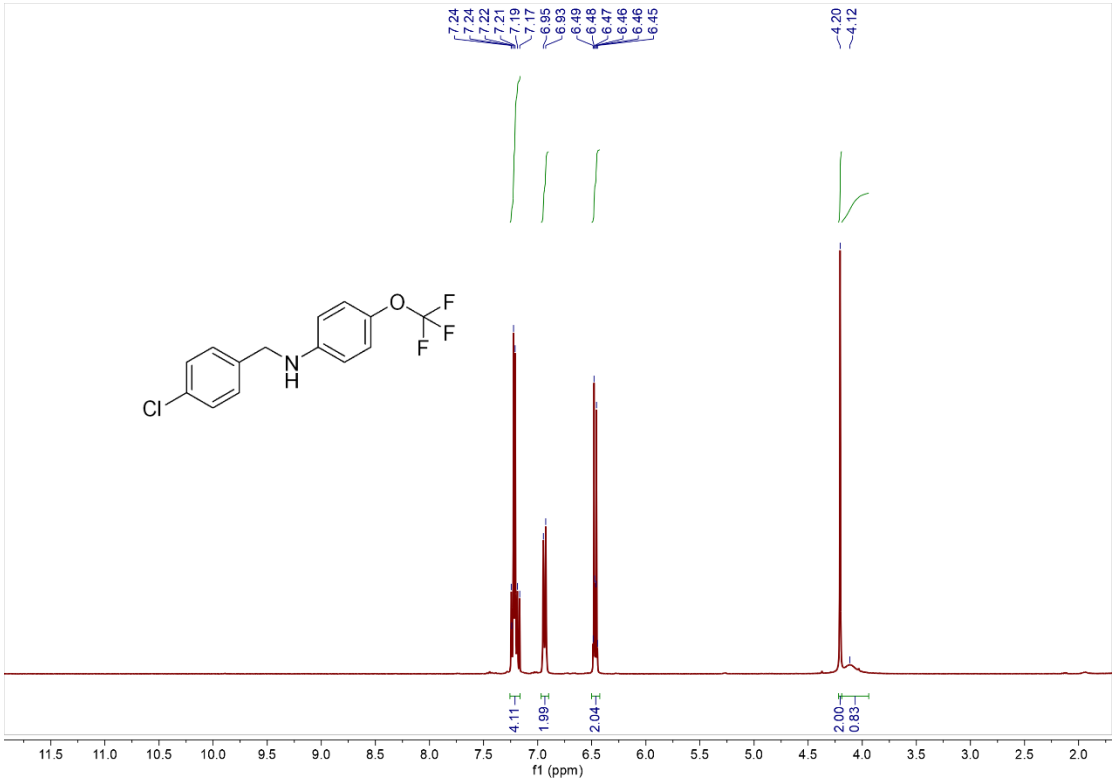
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6.47
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6.46
6.45
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F
F
F
N
H
Cl
4.11
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2.04
2.00
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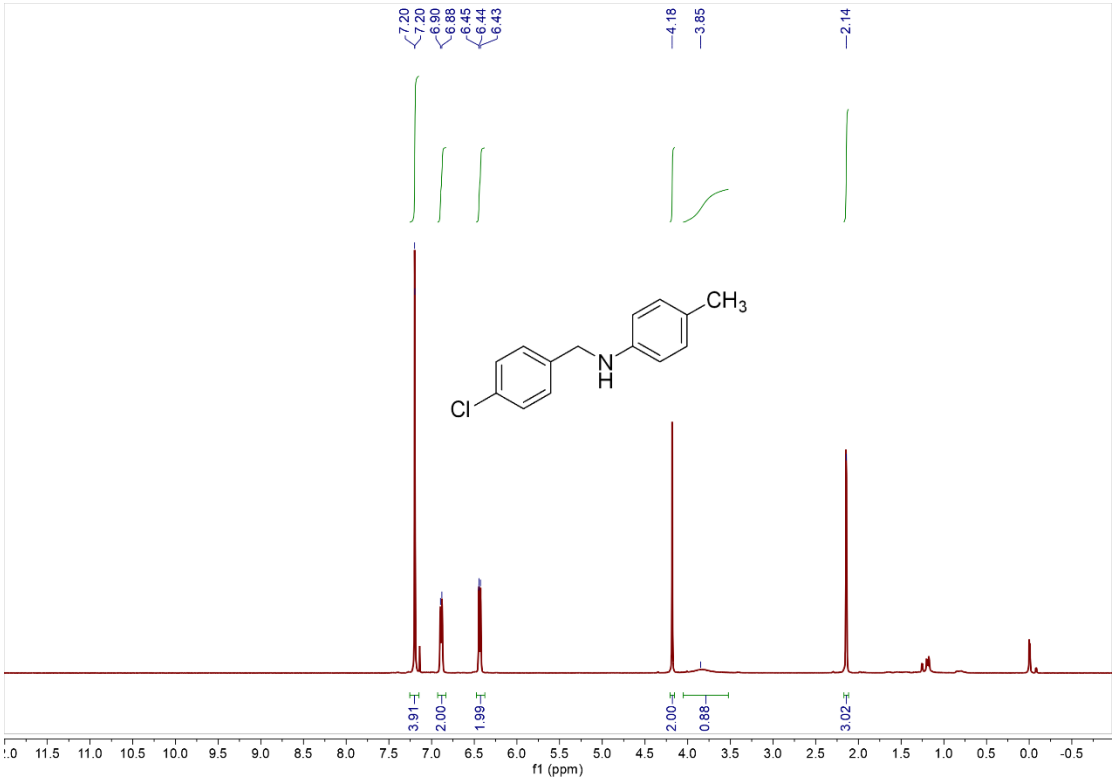
f1 (ppm)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Cl
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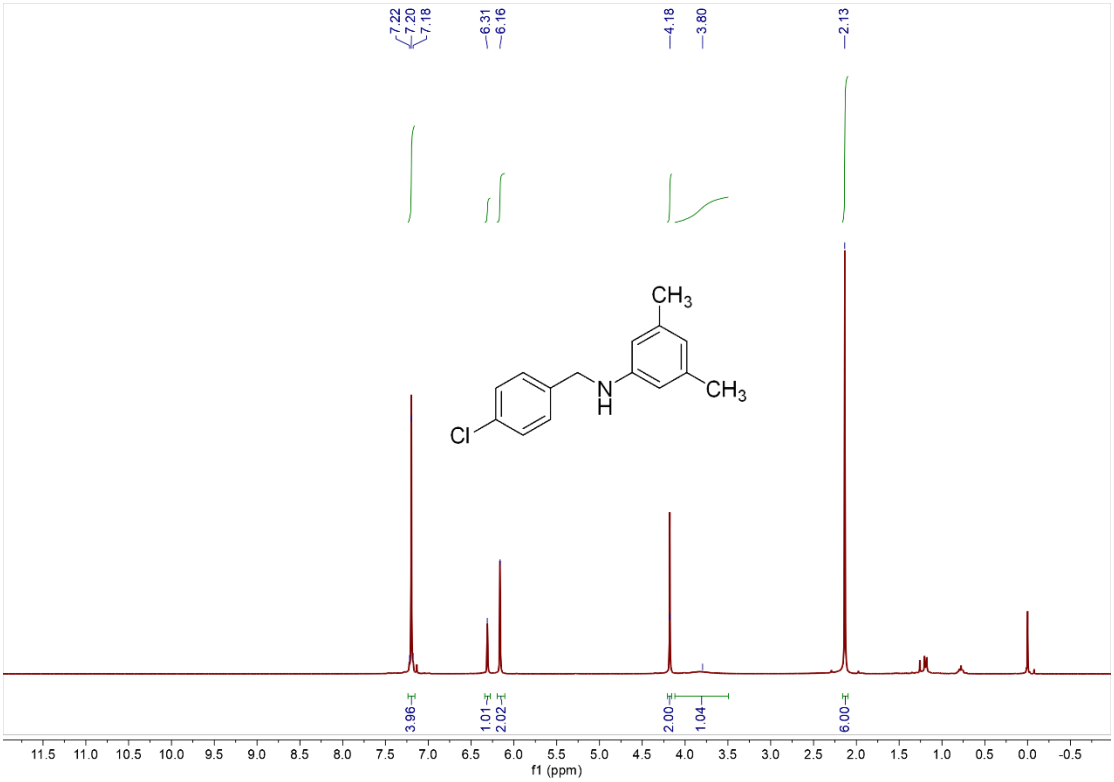
2.00
0.88
3.02
f1 (ppm)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CH3
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H
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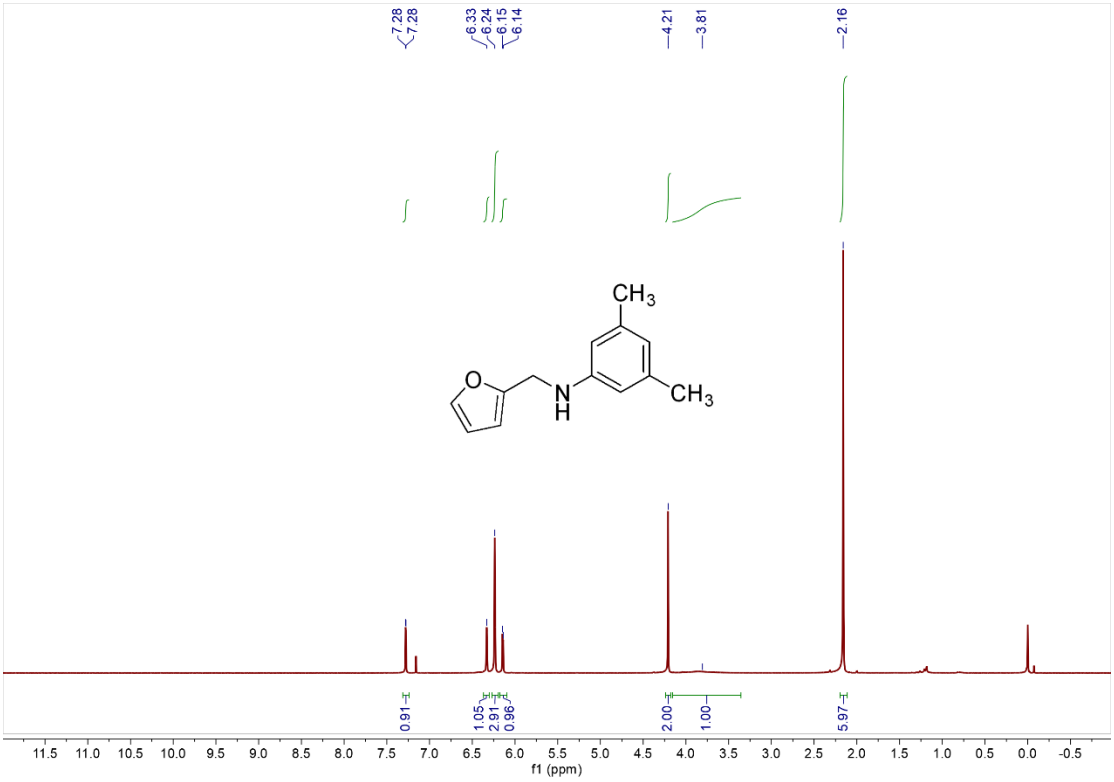
2.02
2.00
1.04
6.00
f1 (ppm)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CH3
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H
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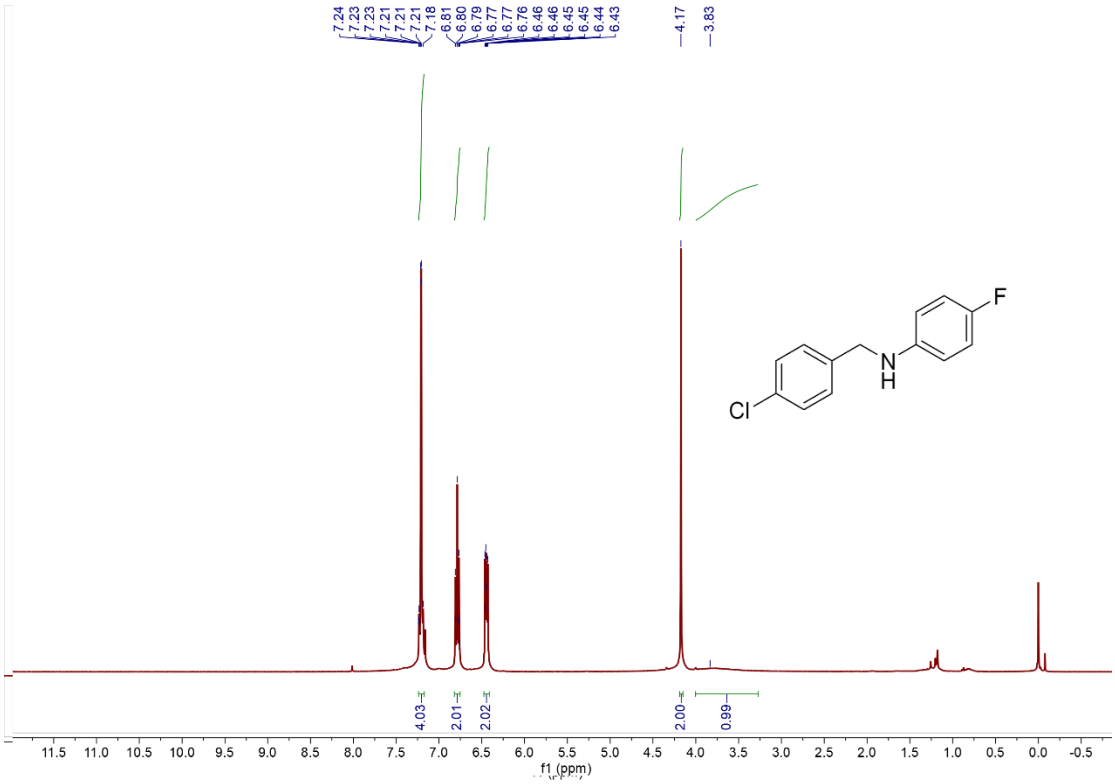
2.91
0.96
2.00
1.00
5.97
f1 (ppm)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6.46
6.45
6.45
6.44
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F
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H
Cl
4.03
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2.02
2.00
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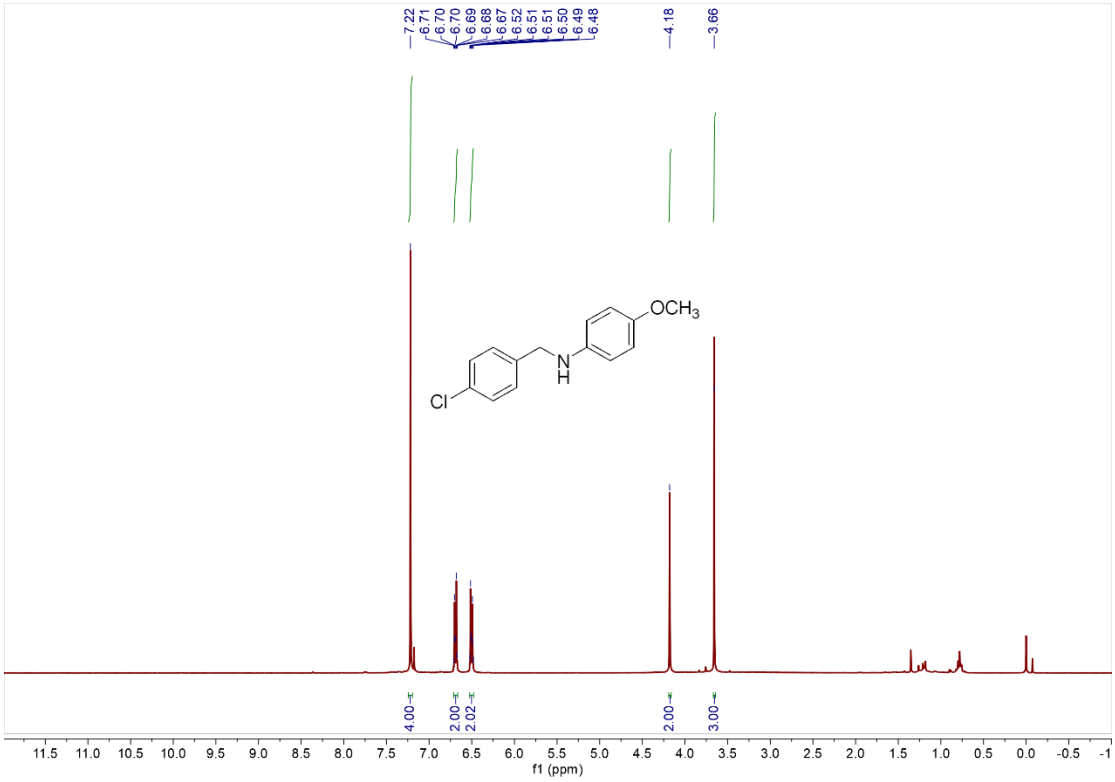
f1 (ppm)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6.49
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H
Cl
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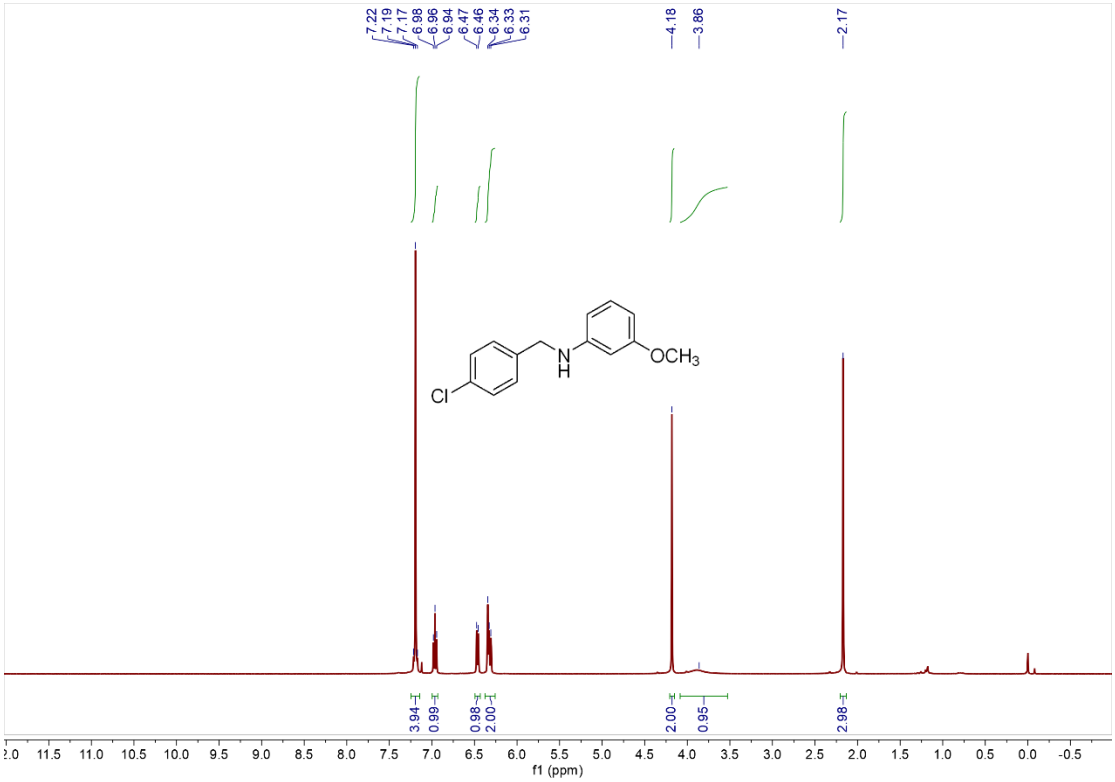
2.00
2.02
2.00
3.00
f1 (ppm)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6.31
4.18
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Cl
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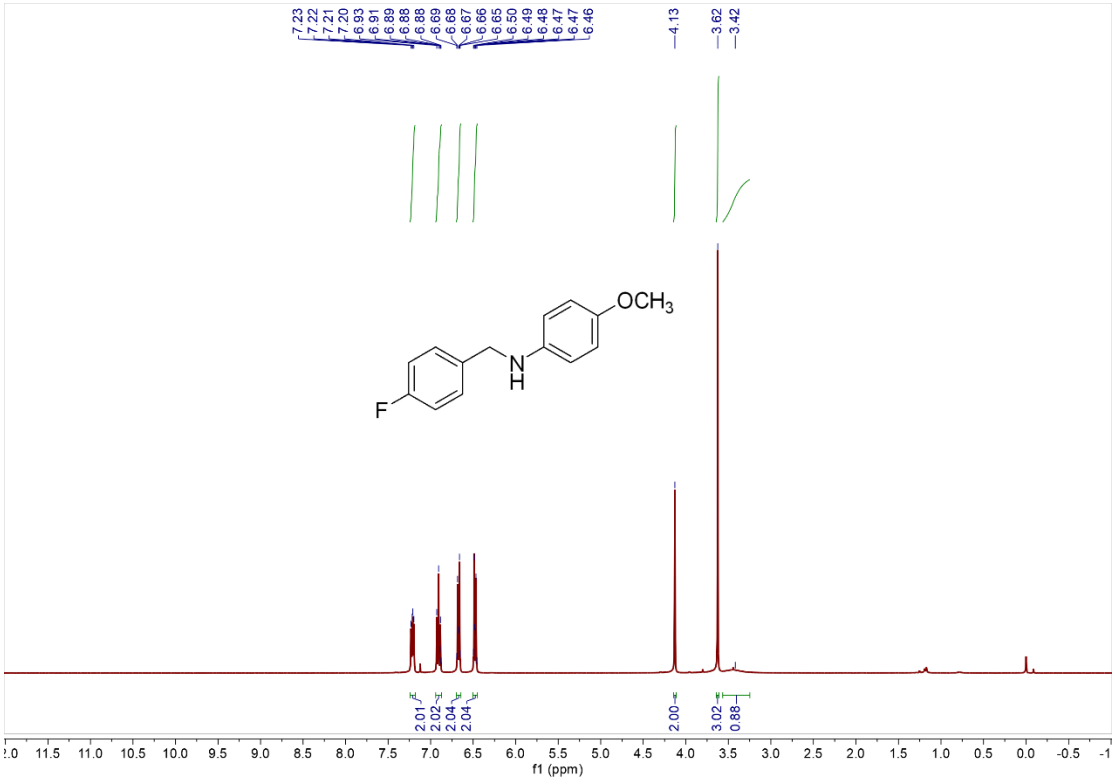
2.98
f1 (ppm)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6.47
6.47
6.46
4.13
3.62
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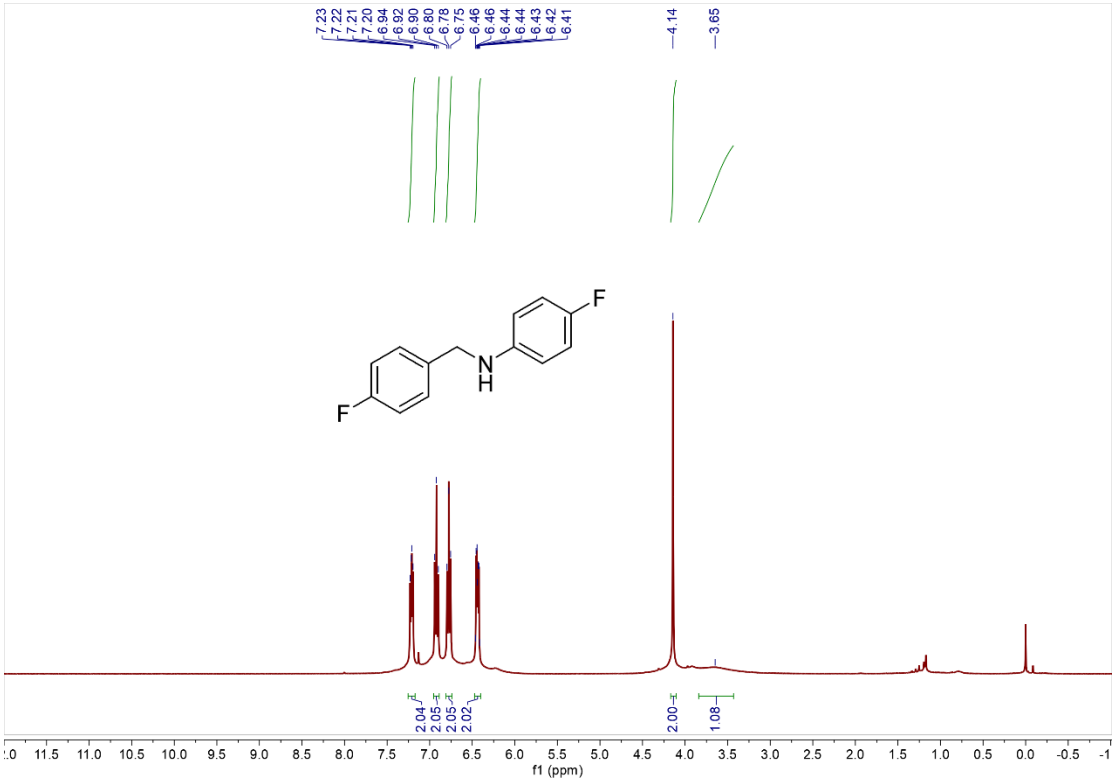
2.04
2.00
3.02
0.88
f1 (ppm)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6.43
6.42
6.41
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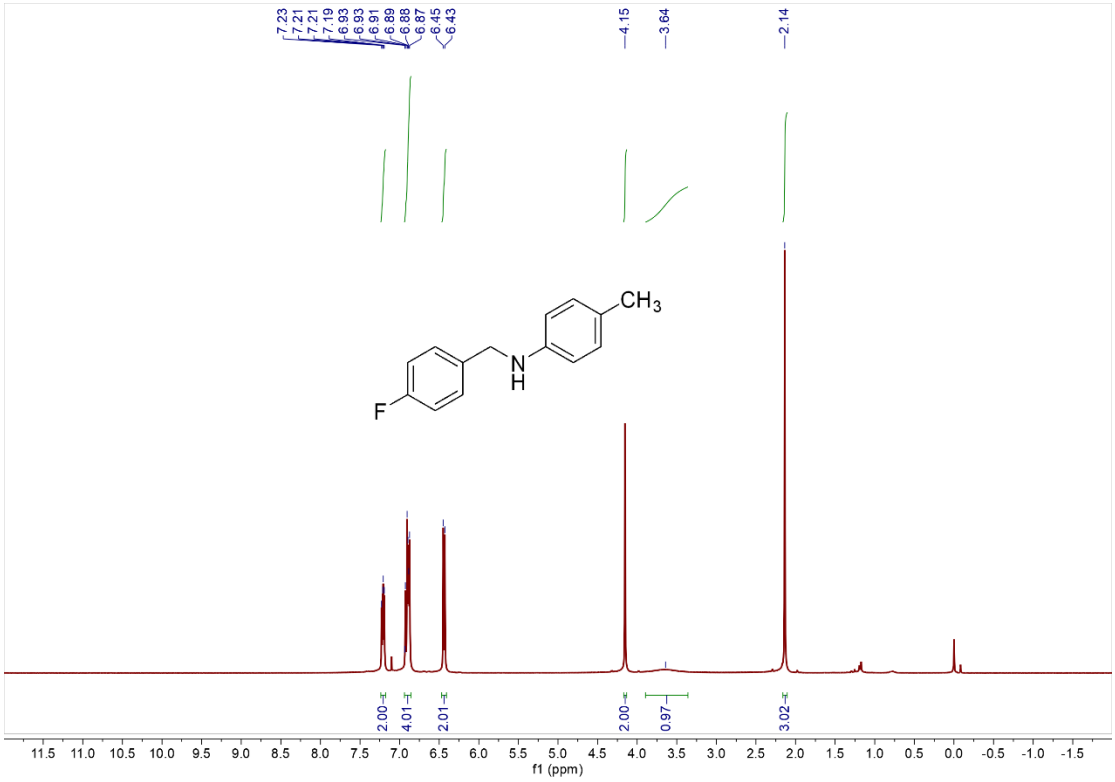
F
N
H
F
2.04
2.05
2.05
2.02
2.00
1.08
f1 (ppm)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H
F
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2.00
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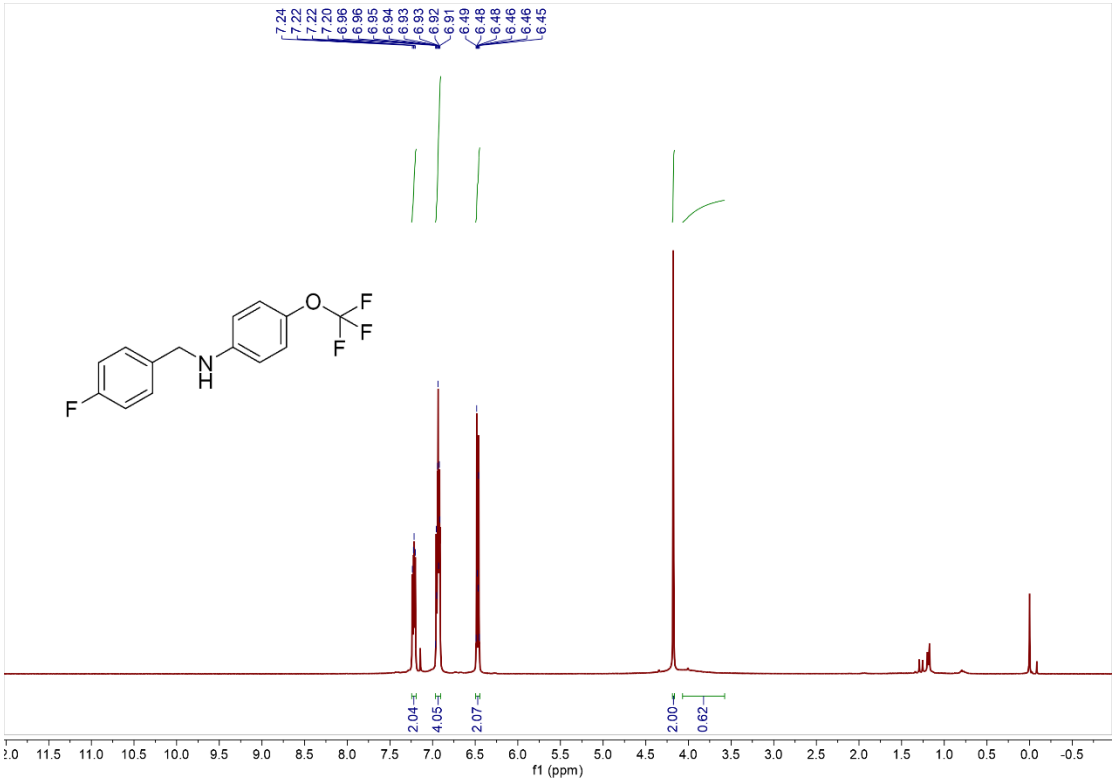
3.02
f1 (ppm)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6.46
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F
F
N
H
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2.07
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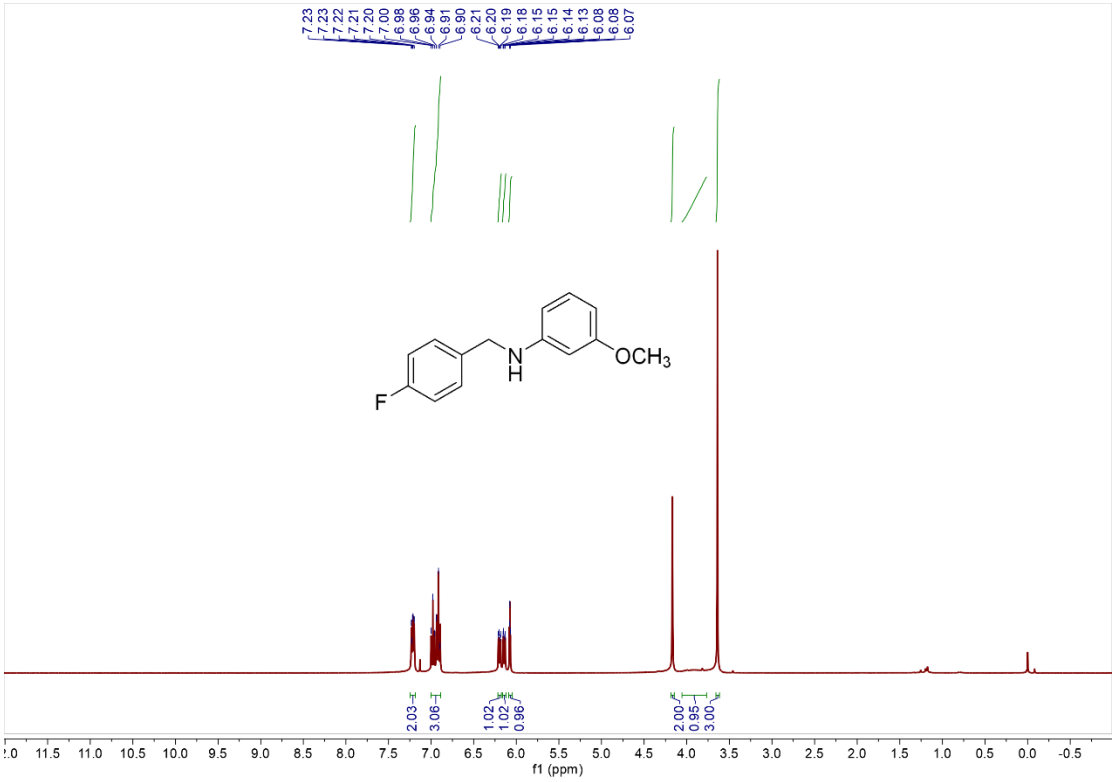
0.62
f1 (ppm)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6.90
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6.20
6.19
6.18
6.15
6.15
6.14
6.13
6.08
6.08
6.07
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F
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3.06
1.02
1.02
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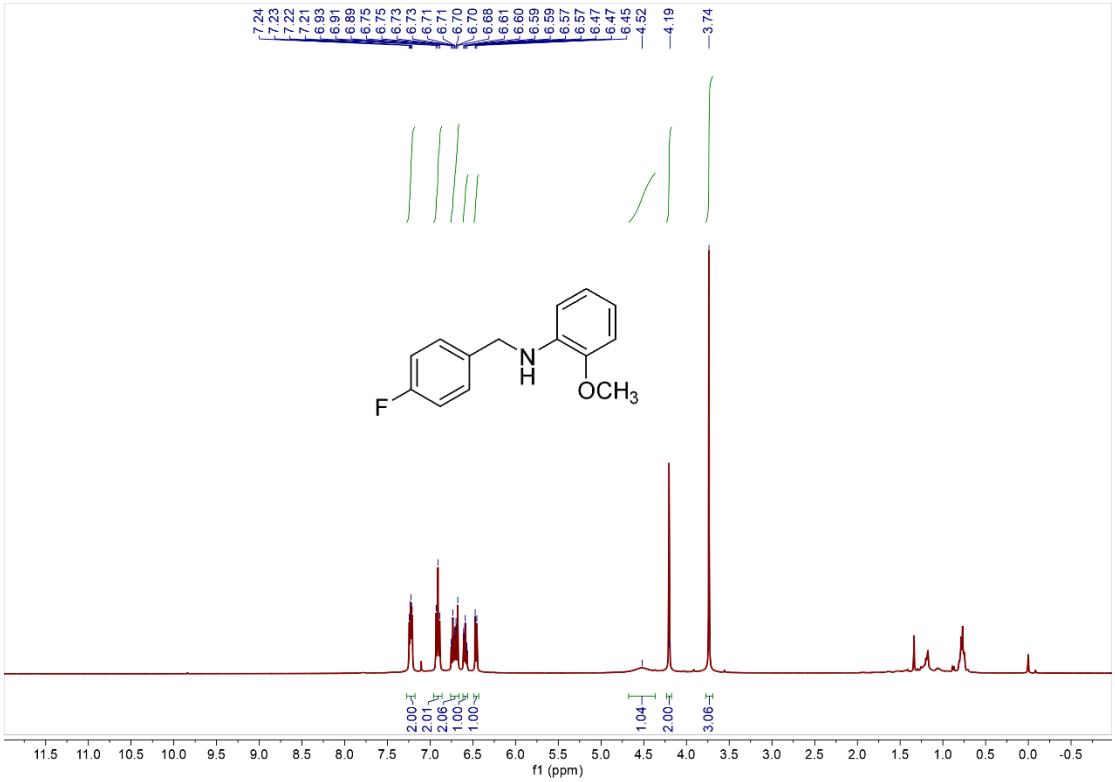
2.00
0.95
3.00
f1 (ppm)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6.45
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4.19
3.74
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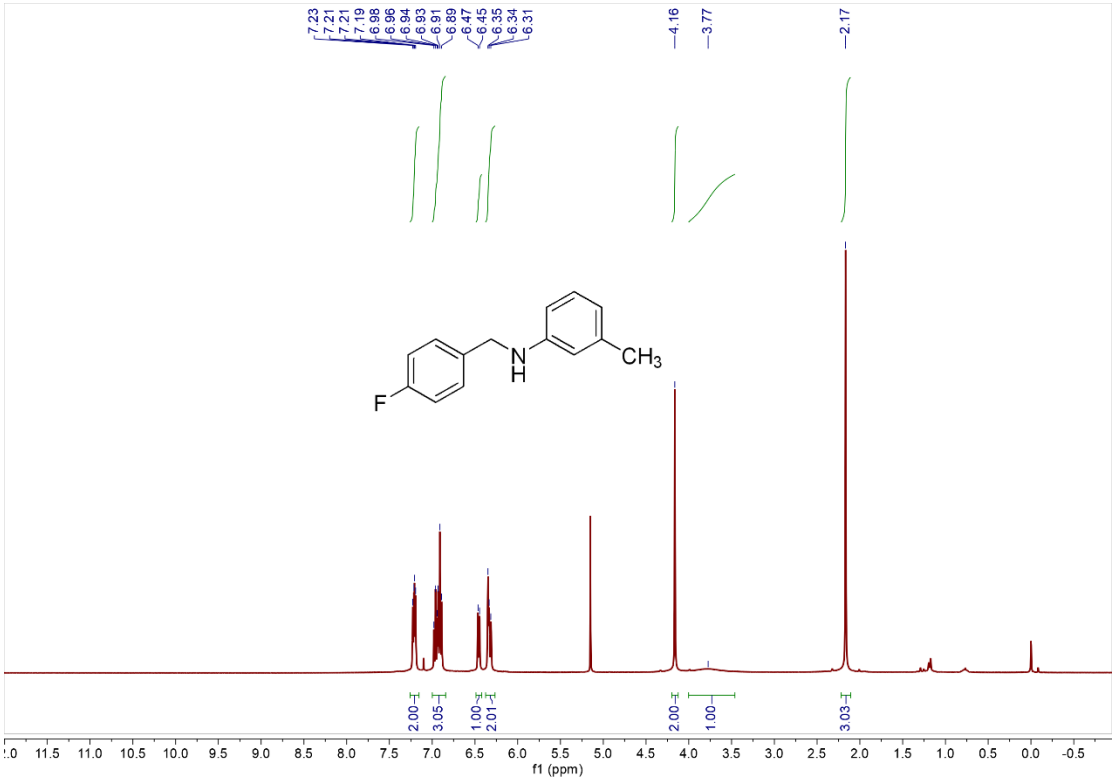
2.06
1.00
1.00
1.04
2.00
3.06
f1 (ppm)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6.34
6.31
4.16
3.77
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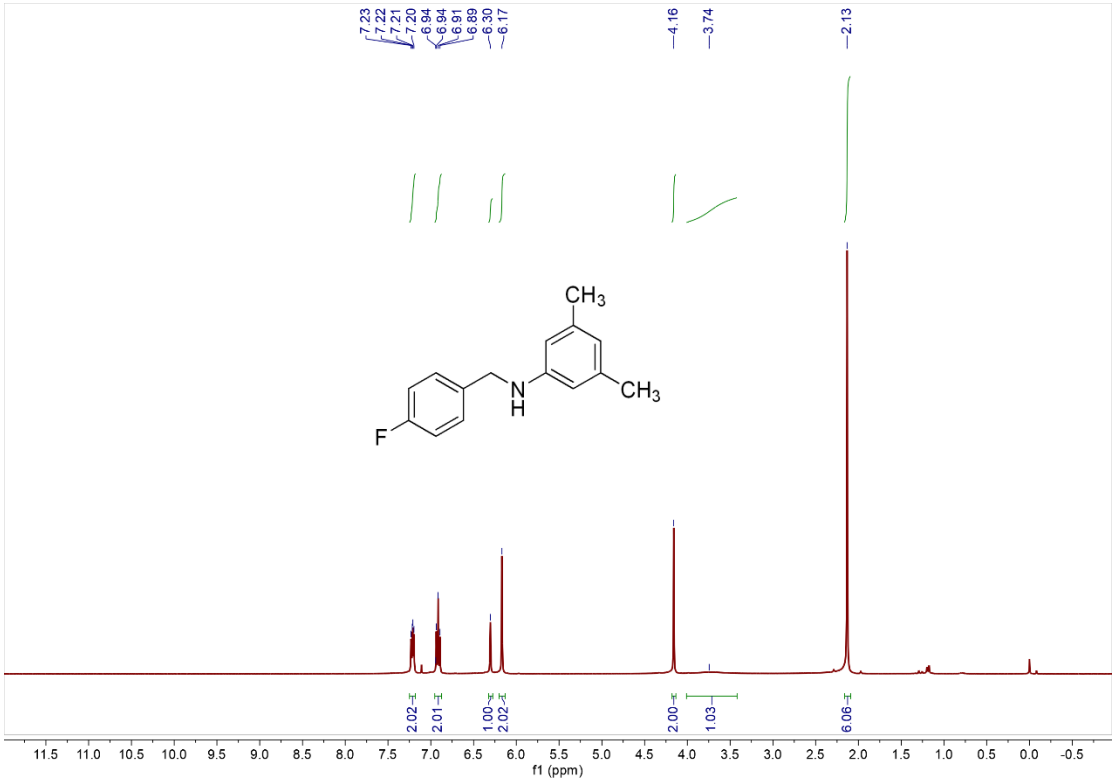
3.05
1.00
2.01
2.00
1.00
3.03
f1 (ppm)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2.13
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CH3
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H
F
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1.00
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2.00
1.03
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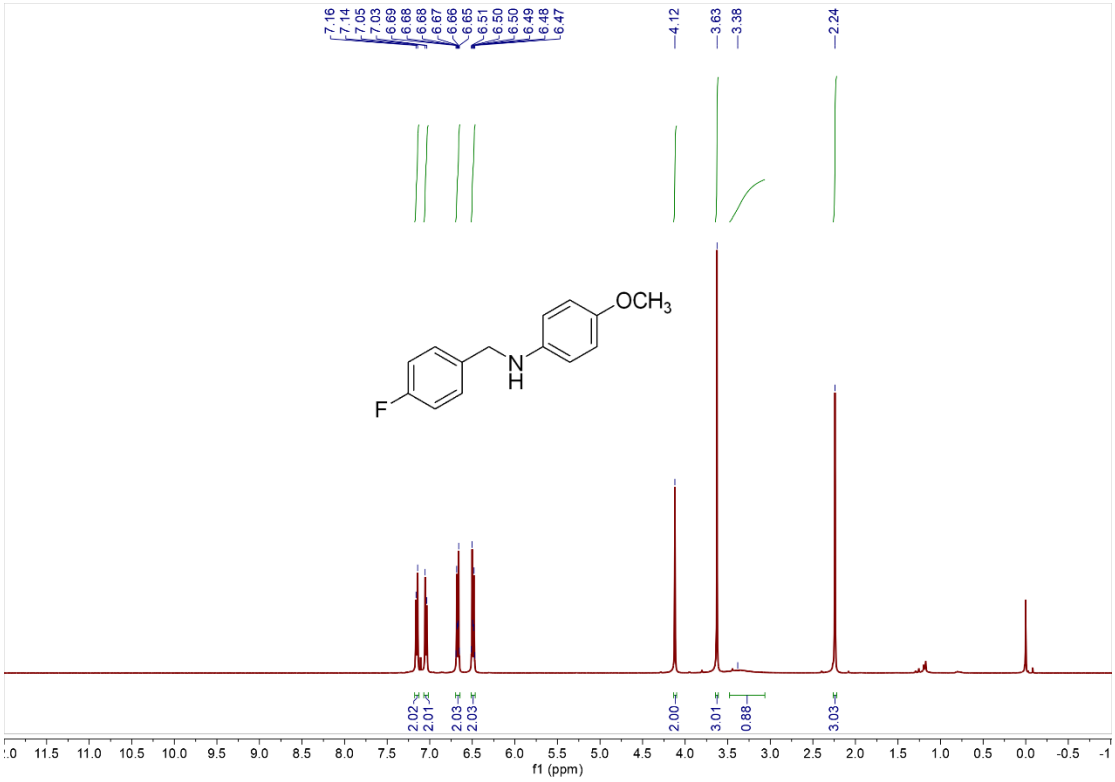
f1 (ppm)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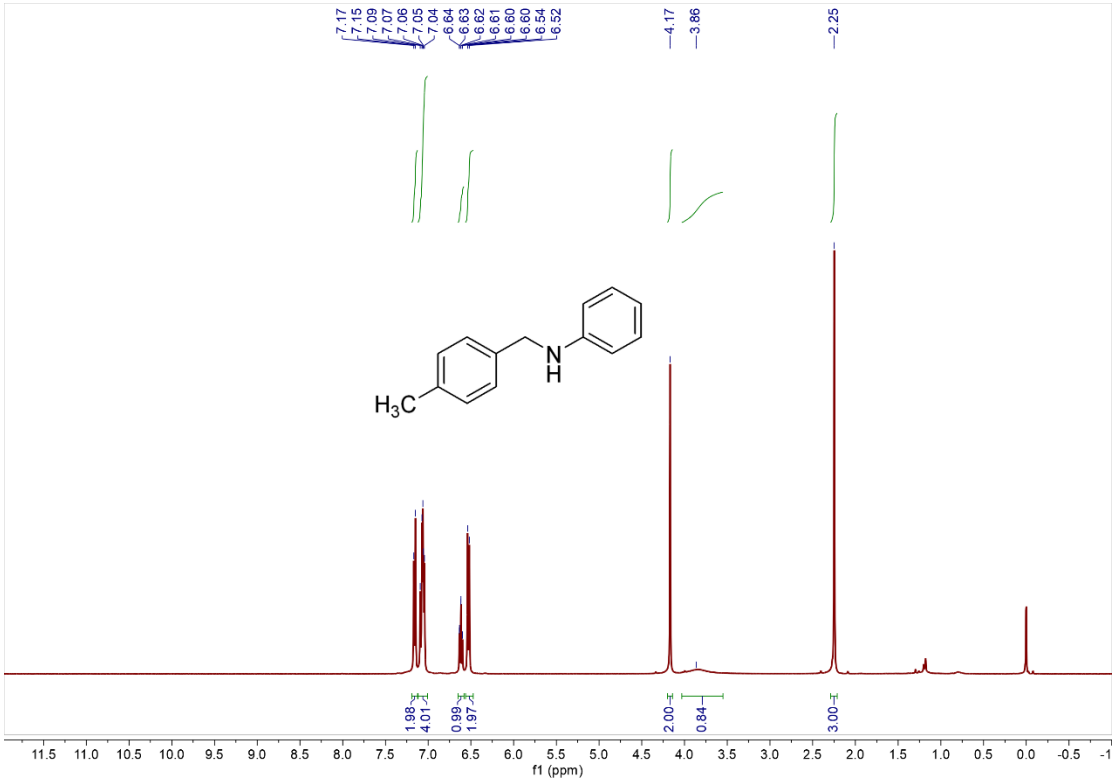
f1 (ppm)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6.54
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3.86
2.25
N
H
H3C
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4.01
0.99
1.97
2.00
0.84
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)