Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting information

A metal-free protocol for the preparation of amines using ammonia borane under mild conditions

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1. Materials and methods

Aldehydes and amines were obtained commercially from various chemical companies. Ammonia borane (No. B5082-5G) was purchased from TCI Chemicals. Unless otherwise stated, all reagents were used directly without purification. GC and GC-MS analysis were recorded on Agilent 6890N instrument. GC conversion and yields were determined by GC-FID, HP6890 chromatograph with FID detector, column HP 530 m x 250 mm x 0.25 μm. NMR spectra are recorded using Bruker 300 Fourier, Bruker AV 300 and Bruker AV 400 spectrometers. Chemical shifts are reported in ppm relative to the deuterated solvent. Coupling constants are expressed in Hertz (Hz). The following abbreviations are used: s = singlet, bs = broad singlet d = doublet, t = triplet and m = multiple. The residual solvent signals were used as references for ^1H and ^{13}C NMR spectra ($CDCl_3$: δH = 7.26 ppm, δC = 77.16 ppm).

2. General procedure for the reductive amination of amines and aldehyde

2.1. One-pot direct reductive amination reaction:

The magnetic stirring bar, amine (0.5 mmol), aldehyde (0.6 mmol) and 2 mL trifluoroethanol were transferred to 10 mL glass tube, then, ammonia borane (0.75 mmol) was added to the tube. The reaction mixture was stirred at room temperature under open flask condition. Upon completion of the reaction, as revealed by TLC, the resulting mixture was concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired amine. Following procedure is applied for determining the conversion and yield by GC. After completion of the reaction, mesitylene (60 mg) as the standard was added to the reaction tube and the reaction products were diluted with ethyl acetate followed by filtration using plug of silica. Then the filtrate containing products were analysed by GC or GC-MS.

2.2. One-pot step wise reductive amination reaction

The magnetic stirring bar, amine (0.5 mmol), aldehyde (0.6 mmol) and 2 mL trifluoroethanol were transferred to 10 mL glass tube. The reaction mixture was stirred for 12-16 h at room temperature under open flask condition, then ammonia borane (0.75 mmol) was added to the reaction tube and continued the reaction for another 8-12 h. Upon completion of the reaction, the resulting mixture was concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

2.3. Methylation of anilines

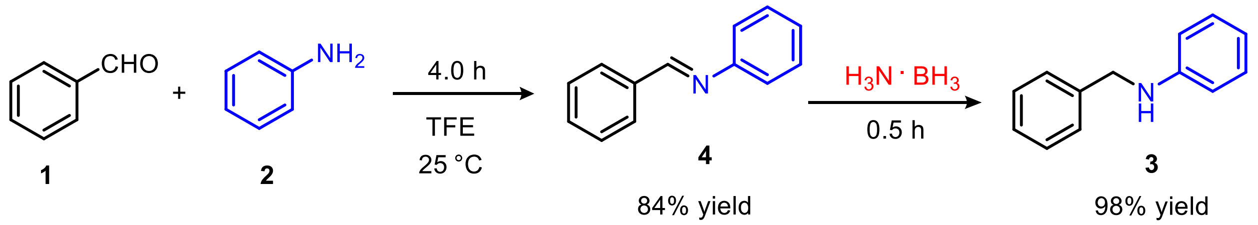
The magnetic stirring bar, amine (0.5 mmol), 37 w% aqueous formaldehyde (2.0 mmol) and 2 mL trifluoroethanol were transferred to 10 mL glass tube, then ammonia borane (1 mmol) was added to the tube. The reaction mixture was stirred for about 24 h at room temperature under open flask condition. Upon completion of the reaction, the resulting mixture was concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

2.4. Reductive amination of amines and aldehydes in gram-scale.

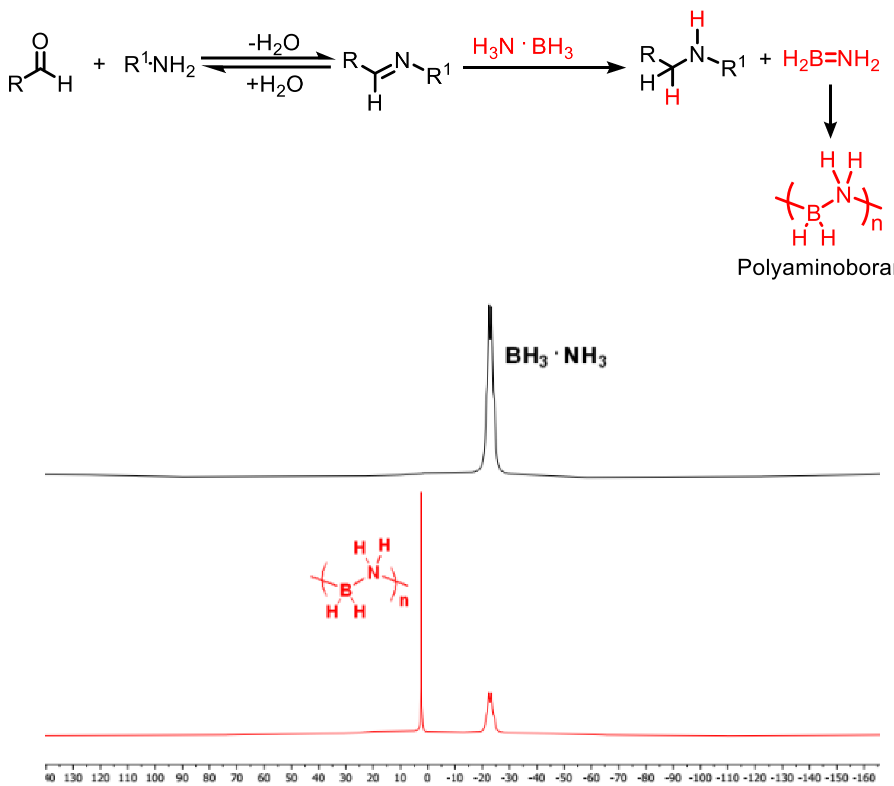
(a) The protocol for 15, 60, 61, 63, 64: the magnetic stirring bar, amine (7.2 mmol), aldehyde (6 mmol) and 20 mL trifluoroethanol were transferred to 50 mL flask. The reaction mixture was stirred for 16 h at room temperature under open flask condition, then ammonia borane (9.0 mmol) was added to the reaction mixture and continue to react for another 8 h. Upon completion of the reaction, the resulting mixture was concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

(b) The methylation of anilines to produce 62 in gram-scale*:* the magnetic stirring bar, amine (6 mmol), 37 w% aqueous formaldehyde (12 mmol) and 20 mL trifluoroethanol were transferred to 50 mL flask, then, ammonia borane (12 mmol) was added to the flask. The reaction mixture was stirred for about 24 h at room temperature under open flask condition. Upon completion of the reaction, the resulting mixture was concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

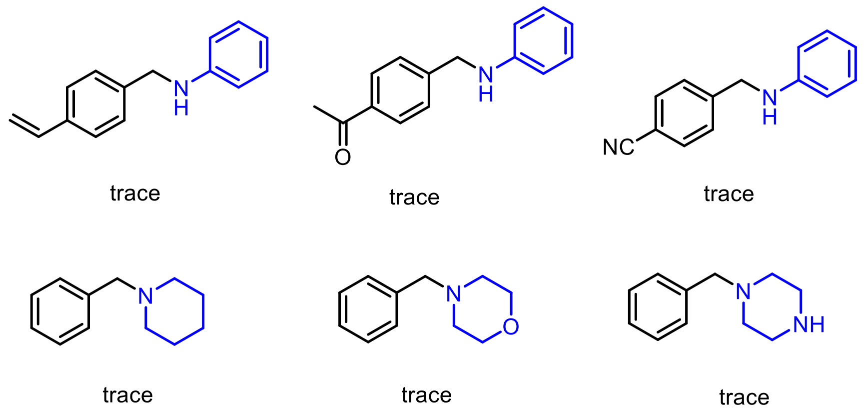
3. The scope, limitations and control experiments



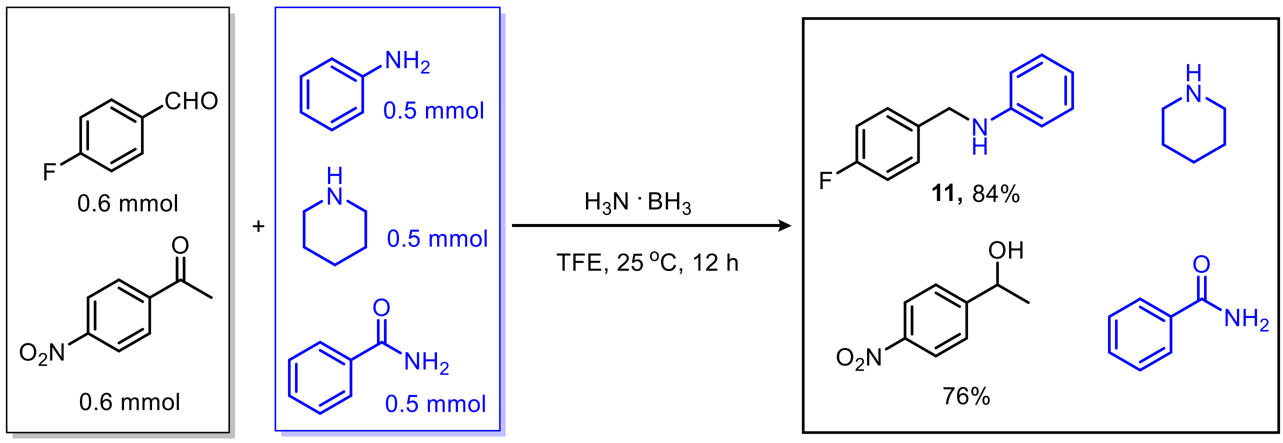
Scheme S1. Control experiment to understand the mechanism. Reaction conditions: benzaldehyde (0.6 mmol), aniline (0.5 mmol), TFE (2 mL), 25 °C, open flask. After 4 h, add ammonia borane (0.75 mmol), the product yield was determined based aniline by GC using mesitylene as a standard.



Scheme S2. [11]B NMR of $H_3N{\cdot}BH_3$ and the reaction mixture of benzaldehyde and aniline



Scheme S3. Scope and limitations.

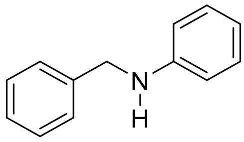


Scheme S4. Control experiments.

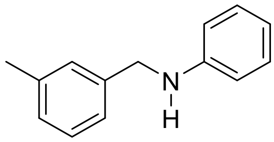
4. Characterization data of products

NMR data

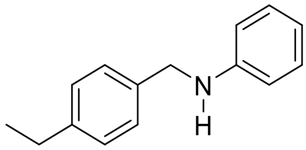
Note: For some compounds intensity of -NH peak is very low and hence not properly visible in ^{1}H-NMR.



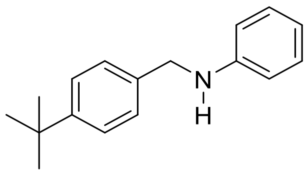
(3): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.49 – 7.29 (m, 5H), 7.29 – 7.19 (m, 2H), 6.79 (tt, *J* = 7.4, 1.1 Hz, 1H), 6.74 – 6.65 (m, 2H), 4.39 (s, 2H), 4.29 – 3.97 (m, 1H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.16, 139.48, 129.39, 128.75, 127.66, 127.36, 117.76, 113.04, 48.49.



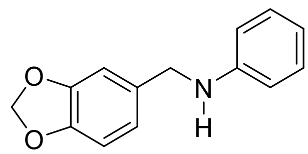
(5): ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.16 (t, *J* = 7.4 Hz, 1H), 7.13 – 7.06 (m, 4H), 7.01 (dtt, *J* = 7.4, 1.3, 0.7 Hz, 1H), 6.65 (tt, *J* = 7.4, 1.1 Hz, 1H), 6.60 – 6.53 (m, 2H), 4.21 (s, 2H), 2.27 (s, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 148.20, 139.37, 138.45, 129.39, 128.67, 128.48, 128.16, 124.77, 117.79, 113.10, 48.59, 21.56.



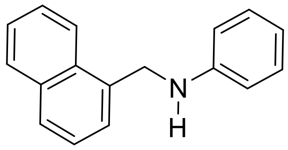
(6): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.35 – 7.28 (m, 2H), 7.24 – 7.16 (m, 4H), 6.78 – 6.71 (m, 1H), 6.70 – 6.64 (m, 2H), 4.31 (s, 2H), 2.67 (q, *J* = 7.6 Hz, 2H), 1.26 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.27, 143.47, 136.66, 129.38, 128.26, 127.78, 117.72, 113.05, 48.31, 28.66, 15.76.



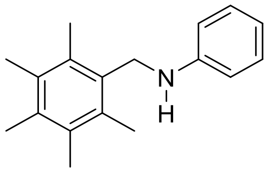
(7): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.45 – 7.30 (m, 4H), 7.21 (dd, *J* = 8.6, 7.4 Hz, 2H), 6.76 (t, *J* = 7.3 Hz, 1H), 6.70 (d, *J* = 8.7 Hz, 2H), 4.32 (s, 2H), 1.35 (s, 9H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.83, 146.65, 140.22, 134.23, 128.54, 128.31, 127.12, 121.41, 108.86, 108.19, 101.00, 53.00, 31.63, 30.26, 29.83.



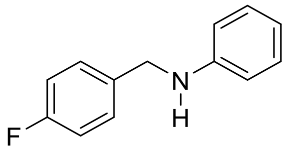
(8): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.14 – 7.05 (m, 2H), 6.81 – 6.72 (m, 2H), 6.71 – 6.62 (m, 2H), 6.61 – 6.53 (m, 2H), 5.86 (s, 2H), 4.16 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.04, 147.78, 146.93, 133.19, 129.41, 120.87, 118.10, 113.33, 108.44, 108.28, 101.14, 48.52.



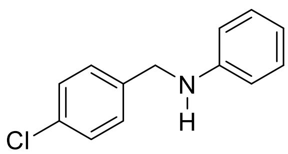
(9): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 8.12 – 8.04 (m, 1H), 7.94 – 7.86 (m, 1H), 7.82 (d, *J* = 8.3 Hz, 1H), 7.59 – 7.50 (m, 3H), 7.44 (dd, *J* = 8.2, 7.0 Hz, 1H), 7.25 – 7.18 (m, 2H), 6.82 – 6.69 (m, 3H), 4.76 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 134.02, 131.70, 129.48, 128.91, 128.39, 126.50, 126.34, 125.99, 125.68, 123.71, 118.04, 113.19, 46.75.



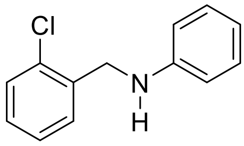
(10): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.26 – 7.17 (m, 2H), 6.76 (t, *J* = 7.3 Hz, 1H), 6.71 (d, *J* = 7.4 Hz, 2H), 4.25 (s, 2H), 2.30 (s, 6H), 2.27 (s, 3H), 2.25 (s, 6H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 164.15, 148.34, 134.88, 133.33, 133.06, 132.38, 129.40, 120.86, 117.48, 112.71, 43.73, 17.27, 17.10, 16.89, 16.51, 16.21.



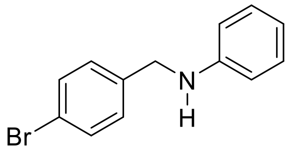
(11): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.41 – 7.30 (m, 2H), 7.25 – 7.17 (m, 2H), 7.12 – 6.99 (m, 2H), 6.82 – 6.71 (m, 1H), 6.69 – 6.60 (m, 2H), 4.32 (d, *J* = 1.1 Hz, 2H), 4.09 (s, 1H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 163.79, 160.54, 147.99, 135.18 (d, *J* = 3.2 Hz), 129.41, 129.13 (d, *J* = 8.0 Hz), 117.91, 115.55 (d, *J* = 21.4 Hz), 113.04.



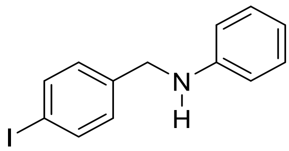
(12): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.22 (s, 4H), 7.13 – 7.05 (m, 2H), 6.65 (tt, *J* = 7.6, 1.1 Hz, 1H), 6.56 – 6.49 (m, 2H), 4.22 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.81, 138.02, 133.02, 129.44, 128.88, 128.86, 118.05, 113.12, 47.81.



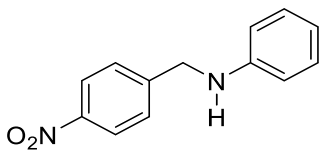
(13): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.39 – 7.26 (m, 2H), 7.17 – 7.04 (m, 4H), 6.65 (tt, *J* = 7.5, 1.1 Hz, 1H), 6.60 – 6.52 (m, 2H), 4.36 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*) δ** 147.71, 136.68, 133.40, 129.66, 129.43, 129.21, 128.54, 127.08, 118.04, 113.21, 46.11.



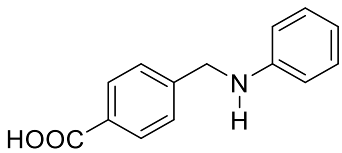
(14): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 8.4 Hz, 2H), 7.32 – 7.26 (m, 2H), 7.22 (dd, *J* = 8.6, 7.2 Hz, 2H), 6.78 (t, *J* = 7.3 Hz, 1H), 6.66 (d, *J* = 7.6 Hz, 2H), 4.33 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.83, 138.60, 131.80, 129.41, 129.17, 121.03, 117.98, 113.05, 47.77.



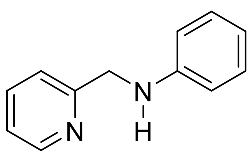
(15): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.72 – 7.63 (m, 2H), 7.24 – 7.16 (m, 2H), 7.16 – 7.11 (m, 2H), 6.76 (tt, *J* = 7.5, 1.1 Hz, 1H), 6.67 – 6.58 (m, 2H), 4.30 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.81, 139.30, 137.75, 129.44, 129.40, 117.96, 113.07, 113.03, 92.51, 47.84.



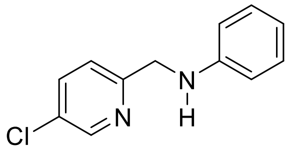
(16): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.24 – 8.12 (m, 2H), 7.60 – 7.46 (m, 2H), 7.24 – 7.11 (m, 2H), 6.82 – 6.70 (m, 1H), 6.65 – 6.53 (m, 2H), 4.48 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.55, 147.32, 147.29, 129.51, 127.84, 124.00, 118.41, 113.11, 47.78.



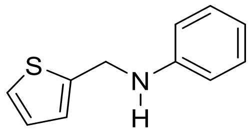
(17): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.7 Hz, 2H), 7.25 – 7.15 (m, 2H), 6.76 (tt, *J* = 7.4, 1.1 Hz, 2H), 6.69 – 6.60 (m, 1H), 4.47 (s, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 171.53, 146.92, 145.46, 130.75, 129.52, 128.43, 127.64, 118.88, 113.80, 48.66.



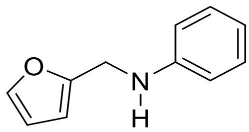
(18): ^{1}**H NMR (400 MHz, Chloroform-*d*)** δ 8.48 (d, *J* = 39.5 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.20 – 7.13 (m, 1H), 7.13 – 7.04 (m, 2H), 6.65 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.57 – 6.49 (m, 2H), 4.26 (s, 2H). ^{13}**C NMR (101 MHz, Chloroform-*d*)** δ 149.05, 148.58, 147.68, 135.29, 129.40, 123.68, 118.07, 113.00, 45.79.



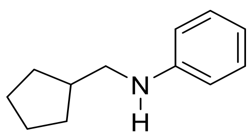
(19): ^{1}**H NMR (400 MHz, Chloroform-*d*)** δ 8.54 (d, *J* = 0.7 Hz, 1H), 7.61 (dd, *J* = 8.3, 2.5 Hz, 1H), 7.34 – 7.28 (m, 1H), 7.23 – 7.13 (m, 2H), 6.74 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.70 – 6.62 (m, 2H), 4.45 (s, 2H). ^{13}**C NMR (101 MHz, Chloroform-*d*)** δ 157.01, 148.19, 147.53, 136.56, 130.52, 129.44, 122.47, 118.15, 113.30, 49.04.



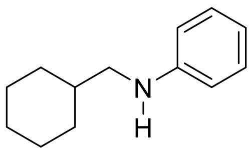
(20): ^{1}**H NMR (300 MHz, Chloroform-*d*)** δ 7.24 – 7.22 (m, 1H), 7.22 – 7.17 (m, 3H), 7.05 – 7.00 (m, 1H), 6.97 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.82 – 6.74 (m, 1H), 6.74 – 6.67 (m, 2H), 4.52 (s, 2H). ^{13}**C NMR (75 MHz, Chloroform-*d*)** δ 163.98, 147.31, 129.44, 127.02, 125.39, 124.84, 118.59, 113.64, 43.83.



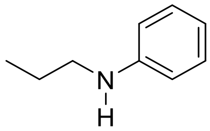
(21): ^{1}**H NMR (400 MHz, Chloroform-*d*)** δ 7.39 (dd, *J* = 1.9, 0.9 Hz, 1H), 7.25 – 7.17 (m, 2H), 6.77 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.73 – 6.67 (m, 2H), 6.34 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.28 – 6.20 (m, 1H), 4.34 (s, 2H). ^{13}**C NMR (101 MHz, Chloroform-*d*)** δ 152.81, 147.67, 142.05, 129.36, 118.21, 113.34, 110.46, 107.14, 41.59.



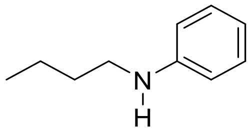
(22): ^{1}**H NMR (400 MHz, Chloroform-*d*)** δ 7.24 – 7.14 (m, 2H), 6.71 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.68 – 6.59 (m, 2H), 3.05 (d, *J* = 7.2 Hz, 2H), 2.24 – 2.12 (m, 1H), 1.91 – 1.78 (m, 2H), 1.72 – 1.53 (m, 4H), 1.36 – 1.22 (m, 2H). ^{13}**C NMR (101 MHz, Chloroform-*d*)** δ 148.69, 129.33, 117.22, 112.84, 49.63, 39.59, 30.81, 25.42.



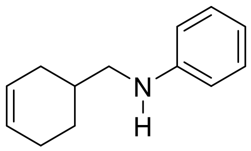
(23): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.25 – 7.12 (m, 2H), 6.69 (tt, *J* = 7.4, 1.1 Hz, 1H), 6.66 – 6.56 (m, 2H), 2.97 (d, *J* = 6.7 Hz, 2H), 1.90 – 1.67 (m, 5H), 1.65 – 1.48 (m, 1H), 1.42 – 1.11 (m, 3H), 1.11 – 0.87 (m, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.68, 129.34, 117.07, 112.80, 77.58, 77.16, 76.74, 50.77, 37.67, 31.44, 26.71, 26.11.



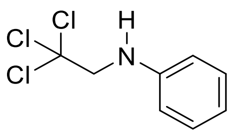
(24): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.23 – 7.10 (m, 2H), 6.74 – 6.63 (m, 1H), 6.59 (dtt, *J* = 7.5, 2.1, 1.2 Hz, 2H), 3.58 (s, 1H), 3.06 (tt, *J* = 7.2, 1.4 Hz, 2H), 1.69 – 1.55 (m, 2H), 1.05 – 0.93 (m, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.63, 129.31, 117.16, 112.79, 45.89, 22.83, 11.74.



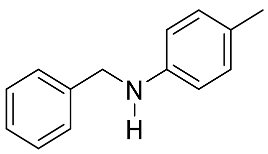
(25): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.25 – 7.14 (m, 2H), 6.77 – 6.66 (m, 1H), 6.66 – 6.58 (m, 2H), 3.13 (dd, *J* = 7.4, 6.7 Hz, 2H), 1.69 – 1.55 (m, 2H), 1.53 – 1.39 (m, 2H), 1.04 – 0.93 (m, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 148.69, 129.34, 129.21, 117.19, 112.81, 43.80, 31.82, 20.44, 14.04.



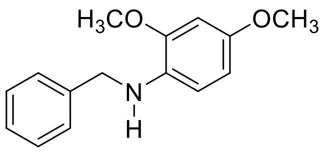
(26): ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.15 (m, 2H), 6.71 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.68 – 6.60 (m, 2H), 5.75 – 5.65 (m, 2H), 3.10 – 3.04 (m, 2H), 2.27 – 2.16 (m, 1H), 2.15 – 2.05 (m, 2H), 1.98 – 1.74 (m, 3H), 1.42 – 1.29 (m, 1H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 148.29, 129.04, 126.99, 125.66, 116.88, 112.50, 49.51, 33.27, 29.53, 26.57, 24.53.



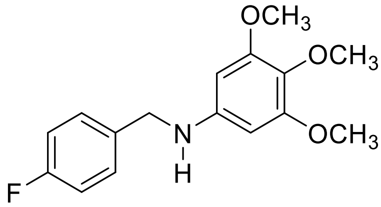
(27): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.18 (m, 2H), 6.86 – 6.78 (m, 2H), 6.78 (s, 1H), 4.42 (s, 1H), 4.15 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 146.24, 129.52, 119.18, 113.50, 100.29, 63.16.



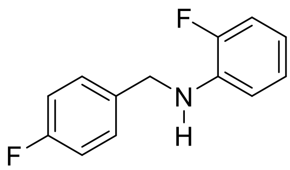
(28): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.32 – 7.09 (m, 5H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.47 (d, *J* = 8.6 Hz, 2H), 4.21 (s, 2H), 2.15 (s, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 145.98, 139.74, 129.88, 128.73, 127.64, 127.29, 126.94, 113.17, 48.80, 20.53.



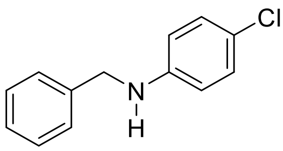
(29): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.49 – 7.36 (m, 4H), 7.36 – 7.25 (m, 1H), 6.57 (d, J = 8.5 Hz, 1H), 6.53 (d, J = 2.6 Hz, 1H), 6.43 (dd, J = 8.5, 2.6 Hz, 1H), 4.36 (s, 2H), 3.88 (s, 3H), 3.80 (s, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 152.13, 148.03, 139.88, 132.53, 128.63, 127.67, 127.17, 110.54, 103.80, 99.27, 55.87, 55.55, 48.94.



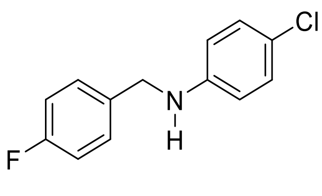
(30): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.40 – 7.28 (m, 2H), 7.03 (t, J = 8.8 Hz, 2H), 5.86 (s, 2H), 4.26 (d, J = 1.0 Hz, 2H), 3.77 (s, 6H), 3.76 (s, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 163.79, 160.54, 154.04, 144.79, 135.08, 135.04, 130.41, 129.26, 129.15, 115.70, 115.42, 90.64, 61.17, 55.99, 48.27.



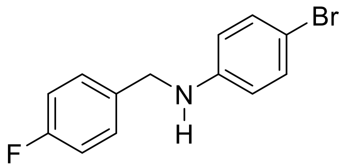
(31): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.35 – 7.28 (m, 2H), 7.15 – 7.09 (m, 2H), 7.07 – 6.99 (m, 2H), 6.57 – 6.51 (m, 2H), 4.28 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 163.87, 160.62, 146.41, 134.62 (d, J = 3.3 Hz), 129.21 (d, J = 5.5 Hz), 129.06, 122.58, 115.67 (d, J = 21.3 Hz), 114.20, 47.86.



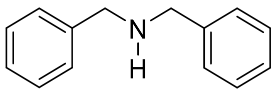
(32): **^{1}H NMR (300 MHz, Chloroform-*d*)** δ 7.29 – 7.15 (m, 5H), 7.02 (d, J = 9.0 Hz, 2H), 6.46 (d, J = 8.9 Hz, 2H, 4.21 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 146.64, 138.97, 129.21, 128.83, 127.57, 127.52, 122.35, 114.15, 48.53.



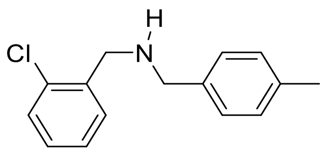
(33): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.29 (m, 2H), 7.10 – 6.92 (m, 4H), 6.72 – 6.60 (m, 2H), 4.35 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 163.88, 160.63, 153.26, 150.10, 136.42 (d, *J* = 11.7 Hz), 134.69 (d, *J* = 3.1 Hz), 129.07 (d, *J* = 8.1 Hz), 124.72 (d, *J* = 3.5 Hz), 117.22 (d, *J* = 7.1 Hz), 115.66 (d, *J* = 21.5 Hz), 114.59 (d, *J* = 18.3 Hz), 112.50 (d, *J* = 3.3 Hz), 47.32.



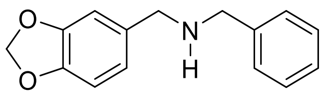
(34): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.38 – 7.30 (m, 2H), 7.05 (t, *J* = 8.7 Hz, 2H), 6.52 (d, *J* = 9.0 Hz, 2H), 6.56 – 6.49 (m, 2H), 4.30 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 163.88, 160.63, 146.80, 134.54 (d, *J* = 3.2 Hz), 132.12, 129.11 (d, *J* = 8.0 Hz), 115.68 (d, *J* = 21.3 Hz), 114.72, 109.64, 47.77.



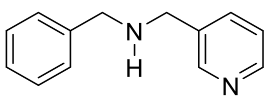
(35): ^{1}H NMR (300 MHz, Chloroform-d_3) δ 7.45 – 7.36 (m, 8H), 7.35 – 7.26 (m, 2H), 3.88 (s, 4H), 1.80 (s, 1H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 140.39, 128.54, 128.50, 128.44, 128.34, 128.32, 128.30, 128.26, 127.05, 53.24.



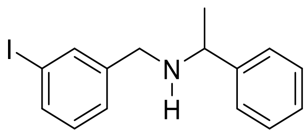
(36): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.41 – 7.26 (m, 2H), 7.21 – 7.11 (m, 4H), 7.10 – 7.03 (m, 2H), 3.82 (s, 2H), 3.69 (s, 2H), 2.26 (s, 3H).**^{13}C NMR (75 MHz, Chloroform-*d*)** δ 137.62, 137.02, 136.76, 133.92, 130.38, 129.64, 129.24, 128.45, 128.29, 126.90, 52.91, 50.76, 21.23.



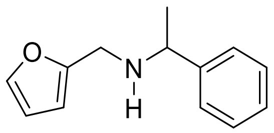
(37): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.02 – 6.95 (m, 4H), 6.94 – 6.87 (m, 1H), 6.56 – 6.48 (m, 1H), 6.48 – 6.37 (m, 2H), 5.59 (s, 2H), 3.44 (s, 2H), 3.37 (s, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.84, 146.65, 140.22, 134.23, 128.54, 128.31, 127.12, 121.41, 108.86, 108.30, 108.19, 101.11, 101.00, 53.01, 52.96.



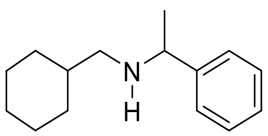
(38): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.38 (d, *J* = 23.8 Hz, 2H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.25 – 7.17 (m, 4H), 7.15 – 7.06 (m, 2H), 3.68 (s, 4H), 3.53 (s, 1H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 149.67, 148.47, 139.16, 136.18, 135.08, 128.55, 128.30, 127.31, 123.53, 52.93, 50.11.



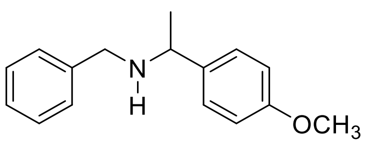
(39): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.62 – 7.54 (m, 1H), 7.54 – 7.44 (m, 1H), 7.29 – 7.25 (m, 4H), 7.22 – 7.10 (m, 2H), 6.95 (t, *J* = 7.7 Hz, 1H), 3.71 (q, *J* = 6.6 Hz, 1H), 3.58 – 3.39 (m, 2H), 1.29 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 145.39, 143.28, 137.17, 135.99, 130.17, 128.63, 127.46, 127.14, 126.76, 94.59, 57.70, 51.09, 24.61.



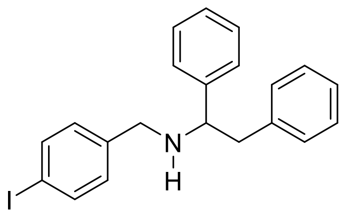
(40): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.20 – 7.13 (m, 5H), 7.13 – 7.04 (m, 1H), 6.12 (dd, *J* = 3.2, 1.9 Hz, 1H), 5.97 – 5.89 (m, 1H), 3.61 (q, *J* = 6.6 Hz, 1H), 3.54 – 3.35 (m, 2H), 1.19 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 154.13, 145.18, 141.84, 128.60, 127.14, 126.87, 110.16, 106.89, 57.18, 44.10, 24.37.



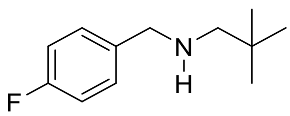
(41): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.36 – 7.27 (m, 4H), 7.26 – 7.16 (m, 1H), 3.71 (q, *J* = 6.6 Hz, 1H), 2.34 (dd, *J* = 11.5, 6.2 Hz, 1H), 2.23 (dd, *J* = 11.5, 7.1 Hz, 1H), 1.78 – 1.61 (m, 5H), 1.36 – 1.07 (m, 7H), 0.95 – 0.78 (m, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 146.22, 128.45, 126.83, 126.66, 58.54, 54.74, 38.31, 31.68, 31.55, 26.82, 26.23, 26.18, 24.66.



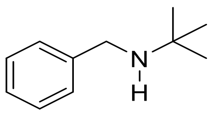
(42): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.14 (m, 8H), 6.80 (d, *J* = 8.7 Hz, 2H), 3.76 – 3.66 (m, 4H), 3.60 – 3.45 (m, 2H), 1.25 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 158.91, 139.17, 136.14, 128.55, 128.52, 128.04, 127.28, 127.05, 114.04, 56.99, 55.34, 51.25, 23.80.



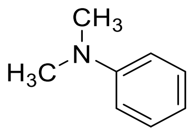
(43): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.63 – 7.55 (m, 2H), 7.40 – 7.35 (m, 4H), 7.33 – 7.23 (m, 4H), 7.18 – 7.09 (m, 2H), 6.92 – 6.83 (m, 2H), 3.87 (dd, *J* = 8.6, 5.6 Hz, 1H), 3.62 (d, *J* = 13.8 Hz, 1H), 3.43 (d, *J* = 13.9 Hz, 1H), 3.08 – 2.85 (m, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 138.77, 137.41, 130.12, 129.40, 128.59, 128.55, 127.49, 127.38, 126.58, 63.64, 50.73, 45.34.



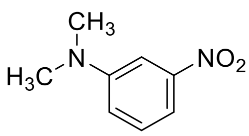
(44): ¹H NMR (300 MHz, Chloroform-*d*) δ 7.28 – 7.16 (m, 2H), 6.98 – 6.87 (m, 2H), 3.70 (d, *J* = 0.8 Hz, 2H), 2.26 (s, 2H), 0.84 (s, 9H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 163.60, 160.36, 136.66, 129.63, 129.52, 115.30, 115.02, 61.66, 54.00, 31.63, 27.92.



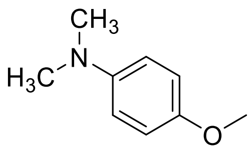
(45): ¹H NMR (300 MHz, Chloroform-*d*) δ 7.38 – 7.19 (m, 5H), 3.73 (d, *J* = 0.6 Hz, 2H), 1.18 (s, 9H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 141.64, 128.52, 128.36, 126.84, 50.79, 47.41, 29.29.



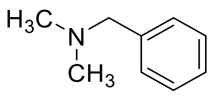
(46): ¹H NMR (300 MHz, Chloroform-*d*) δ 7.30 – 7.10 (m, 2H), 6.80 – 6.59 (m, 3H), 2.88 (s, 6H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 150.70, 129.12, 128.73, 122.34, 116.68, 112.71, 40.62.



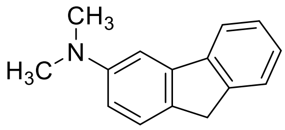
(47): ¹H NMR (300 MHz, Chloroform-*d*) δ 7.57 – 7.45 (m, 2H), 7.33 (t, *J* = 0.5 Hz, 1H), 7.00 – 6.90 (m, 1H), 3.03 (s, 6H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 150.93, 129.67, 117.74, 110.82, 106.28, 40.49.



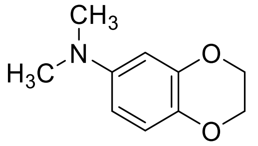
(48): ¹H NMR (300 MHz, Chloroform-*d*) δ 6.88 – 6.79 (m, 2H), 6.78 – 6.71 (m, 2H), 3.75 (s, 3H), 2.85 (s, 6H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 152.12, 145.89, 115.02, 114.75, 55.85, 41.93.



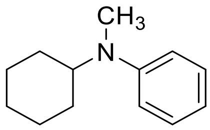
(49): ¹H NMR (300 MHz, Chloroform-*d*) δ 7.37 – 7.19 (m, 5H), 3.41 (s, 2H), 2.23 (s, 6H). **¹³C NMR (75 MHz, Chloroform-*d*)** δ 139.01, 129.22, 128.34, 127.14, 64.54, 45.49.



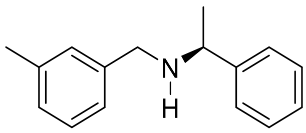
(50): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.71 – 7.61 (m, 2H), 7.48 (s, 1H), 7.37 – 7.28 (m, 1H), 7.24 – 7.15 (m, 1H), 7.08 (s, 1H), 6.91 (s, 1H), 3.86 (s, 2H), 3.04 (s, 6H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 145.13, 142.46, 126.75, 124.96, 124.82, 120.55, 118.67, 111.89, 109.63, 41.30, 37.24.



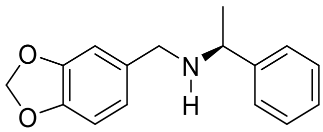
(51): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 6.73 – 6.66 (m, 1H), 6.28 – 6.22 (m, 2H), 4.18 – 4.14 (m, 2H), 4.14 – 4.09 (m, 2H), 2.77 (s, 6H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 146.34, 143.85, 135.79, 117.49, 107.36, 102.70, 64.87, 64.36, 41.72, 29.83.



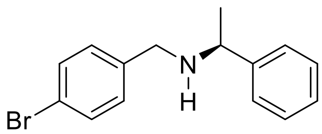
(52): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.18 (m, 2H), 6.81 – 6.75 (m, 2H), 6.67 (tt, *J* = 7.2, 1.1 Hz, 1H), 3.63 – 3.49 (m, 1H), 2.77 (s, 3H), 1.89 – 1.75 (m, 4H), 1.68 (dtt, *J* = 14.1, 3.1, 1.5 Hz, 1H), 1.48 – 1.26 (m, 4H), 1.20 – 1.05 (m, 1H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 150.32, 129.23, 116.35, 113.29, 58.27, 31.30, 30.20, 26.36, 26.10.



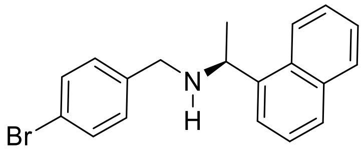
(54): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.32 – 7.21 (m, 4H), 7.21 – 7.06 (m, 2H), 7.06 – 6.91 (m, 3H), 3.73 (q, *J* = 6.6 Hz, 1H), 3.59 – 3.42 (m, 2H), 2.25 (d, *J* = 0.8 Hz, 3H), 1.28 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 145.65, 140.59, 138.05, 129.01, 128.56, 128.36, 127.70, 127.02, 126.81, 126.76, 125.29, 57.68, 51.77, 24.54, 21.49.



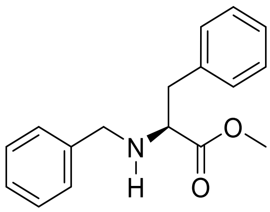
(55): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.39 – 7.32 (m, 4H), 7.32 – 7.21 (m, 1H), 6.82 (dq, *J* = 1.5, 0.5 Hz, 1H), 6.78 – 6.67 (m, 2H), 5.92 (s, 2H), 3.80 (q, *J* = 6.6 Hz, 1H), 3.63 – 3.45 (m, 2H), 1.37 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.73, 146.49, 145.66, 134.77, 128.57, 127.02, 126.78, 121.23, 108.80, 108.78, 108.13, 100.92, 57.43, 51.51, 24.61.



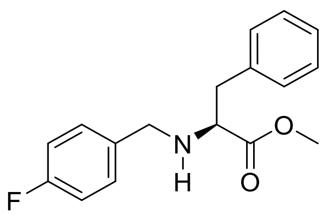
(56): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 8.4 Hz, 2H), 7.35 – 7.29 (m, 4H), 7.26 – 7.18 (m, 1H), 7.12 (d, *J* = 8.6 Hz, 2H), 3.74 (q, *J* = 6.6 Hz, 1H), 3.62 – 3.45 (m, 2H), 1.33 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 145.46, 139.80, 131.51, 129.96, 128.63, 127.14, 126.77, 120.68, 57.59, 51.04, 24.60.



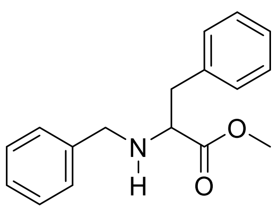
(57): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.15 (q, *J* = 3.7 Hz, 1H), 7.95 – 7.84 (m, 1H), 7.77 (t, *J* = 8.4 Hz, 2H), 7.58 – 7.38 (m, 5H), 7.24 – 7.13 (m, 2H), 4.68 (q, *J* = 6.6 Hz, 1H), 3.78 – 3.60 (m, 2H), 1.53 (dd, *J* = 6.7, 1.3 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 140.82, 139.76, 134.16, 131.55, 131.47, 130.07, 129.13, 127.49, 125.92, 125.88, 125.52, 123.06, 123.03, 120.80, 53.17, 51.24, 23.82.



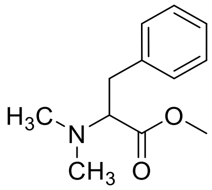
(58): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.10 – 6.95 (m, 8H), 6.94 – 6.87 (m, 2H), 3.57 (d, *J* = 13.2 Hz, 1H), 3.40 (d, *J* = 0.4 Hz, 4H), 3.30 (dd, *J* = 7.1, 6.6 Hz, 1H), 2.76 – 2.68 (m, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 175.16, 139.71, 137.44, 129.34, 128.50, 128.46, 128.24, 127.14, 126.80, 62.19, 52.12, 51.76, 39.86.



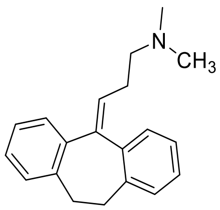
(59): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.16 – 7.03 (m, 3H), 7.02 – 6.91 (m, 4H), 6.83 – 6.69 (m, 2H), 3.59 (dq, *J* = 13.3, 0.9 Hz, 1H), 3.47 (s, 3H), 3.44 – 3.24 (m, 2H), 2.86 – 2.67 (m, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 175.15, 163.70, 160.45, 137.41, 135.42, 129.82, 129.72, 129.34, 128.53, 126.86, 115.36, 115.07, 62.06, 51.84, 51.36, 39.87.



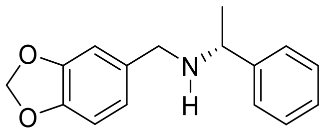
(60): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 77.32 – 7.19 (m, 8H), 7.19 – 7.13 (m, 2H), 3.81 (d, *J* = 13.2 Hz, 1H), 3.64 (s, 3H), 3.57 – 3.51 (m, 1H), 2.96 (d, *J* = 7.5 Hz, 2H). **^{13}C NMR (75 MHz, Chloroform-*d*)** 175.14, 139.69, 137.43, 129.39, 129.33, 128.49, 128.45, 128.23, 128.15, 127.13, 126.79, 62.23, 62.17, 52.11, 51.75, 39.91, 39.85.



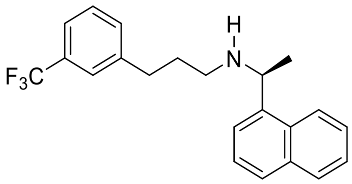
(61): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.32 – 7.13 (m, 5H), 3.59 (s, 3H), 3.42 (dd, *J* = 9.4, 5.8 Hz, 1H), 3.05 (dd, *J* = 13.4, 9.4 Hz, 1H), 2.93 (dd, *J* = 13.4, 5.8 Hz, 1H), 2.38 (s, 6H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 171.93, 138.21, 129.13, 128.45, 126.50, 69.70, 51.10, 41.95, 35.84.



(62): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.32 – 7.25 (m, 1H), 7.22 – 7.07 (m, 6H), 7.06 – 6.97 (m, 1H), 5.86 (t, *J* = 7.0 Hz, 1H), 3.64 – 2.57 (m, 4H), 2.40 – 2.23 (m, 4H), 2.14 (s, 6H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 143.56, 141.42, 140.23, 139.44, 137.18, 130.06, 129.49, 128.74, 128.35, 128.10, 127.48, 127.10, 126.07, 125.84, 77.58, 77.16, 76.74, 59.60, 45.48, 33.90, 32.15, 28.07.



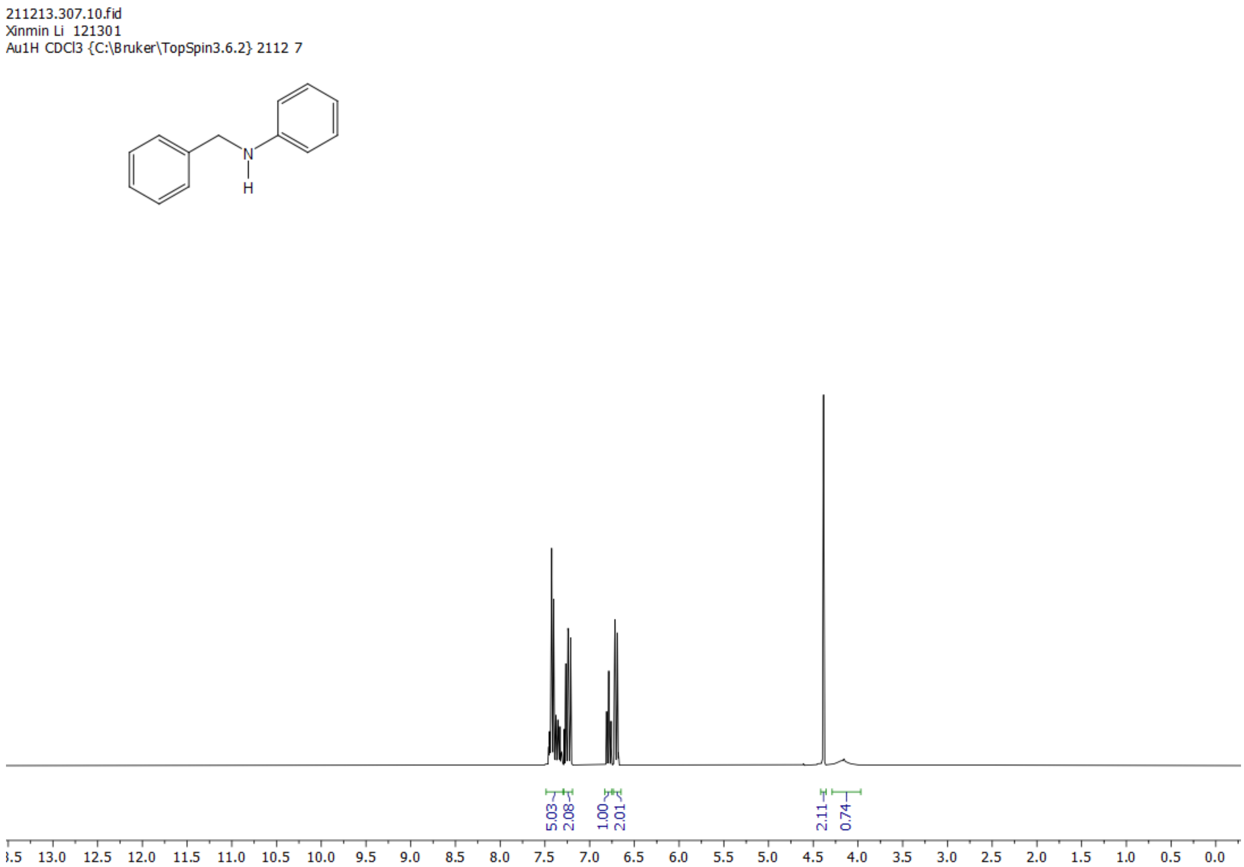
(63): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.32 (m, 4H), 7.32 – 7.23 (m, 1H), 6.86 – 6.80 (m, 1H), 6.79 – 6.68 (m, 2H), 5.93 (s, 2H), 3.81 (q, *J* = 6.6 Hz, 1H), 3.53 (q, 2H), 1.37 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 147.75, 146.51, 145.63, 134.74, 128.59, 127.05, 126.80, 121.27, 108.83, 108.15, 100.95, 57.45, 51.52, 24.60.

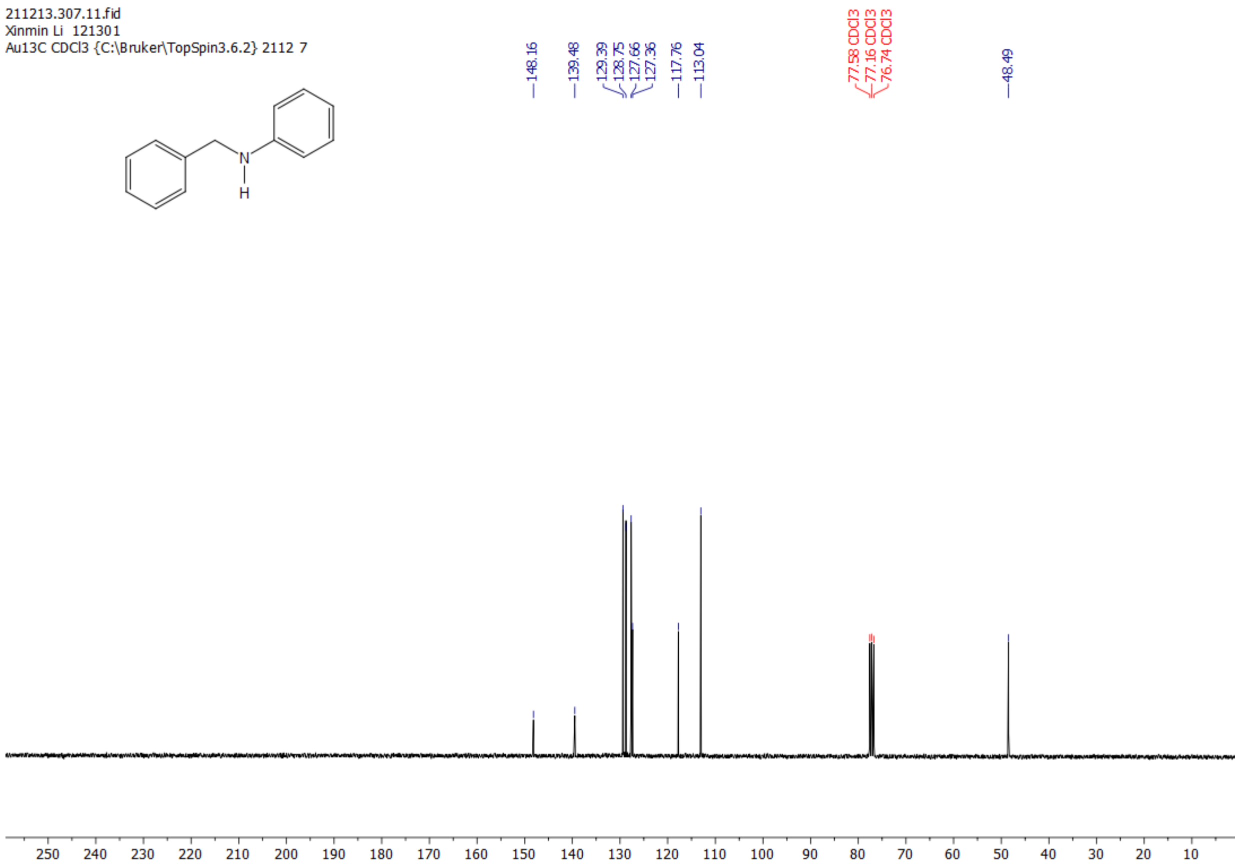


(64): ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.15 – 8.03 (m, 1H), 7.81 – 7.68 (m, 1H), 7.67 – 7.59 (m, 1H), 7.59 – 7.49 (m, 1H), 7.44 – 7.26 (m, 5H), 7.24 – 7.13 (m, 2H), 4.50 (q, *J* = 6.6 Hz, 1H), 2.68 – 2.39 (m, 4H), 1.81 – 1.62 (m, 2H), 1.38 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, Chloroform-*d*)** δ 143.22, 141.34, 134.11, 131.88 (d, *J* = 1.5 Hz), 131.43, 131.07 – 129.78 (m), 129.10, 128.77, 127.30, 125.84 (d, *J* = 4.5 Hz), 125.43, 125.15 (q, *J* = 3.8 Hz), 123.04, 122.78, 53.89, 47.38, 33.51, 31.99, 23.74.

5. Characterization spectrum of products

NMR and HPLC spectra





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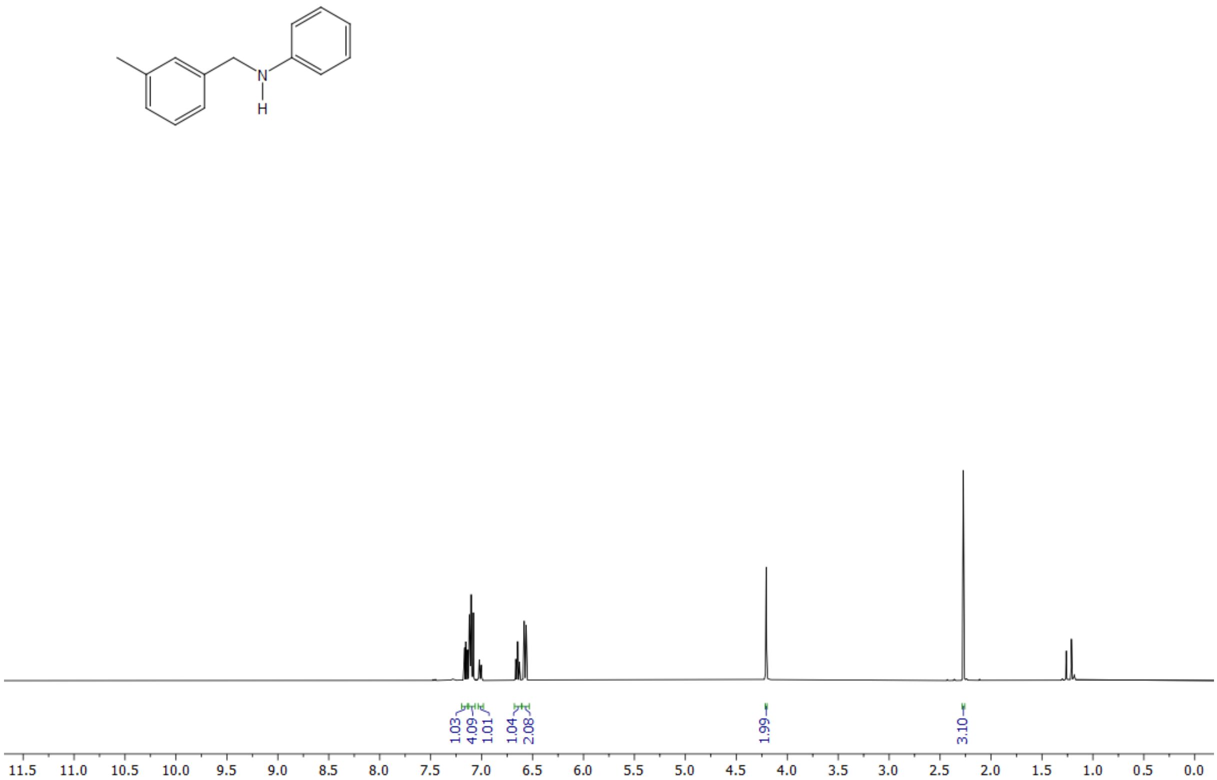

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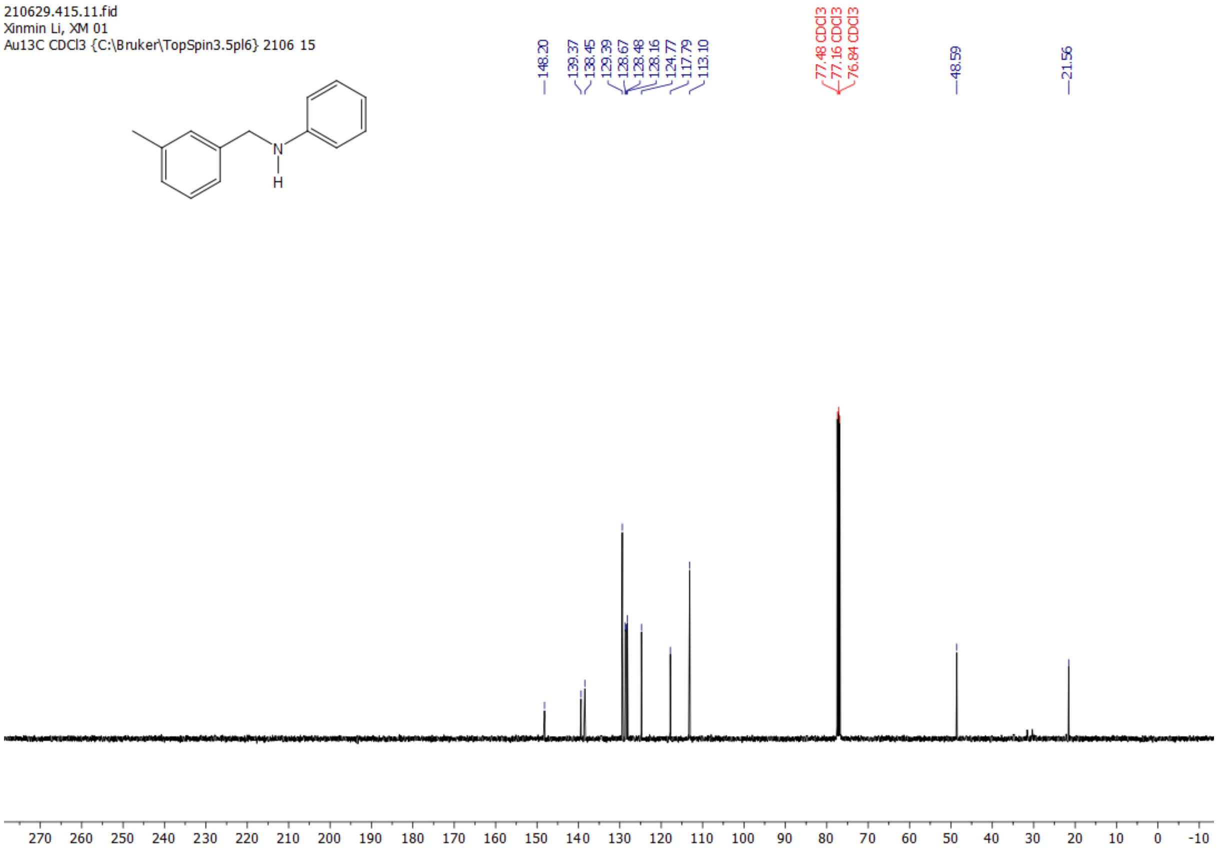

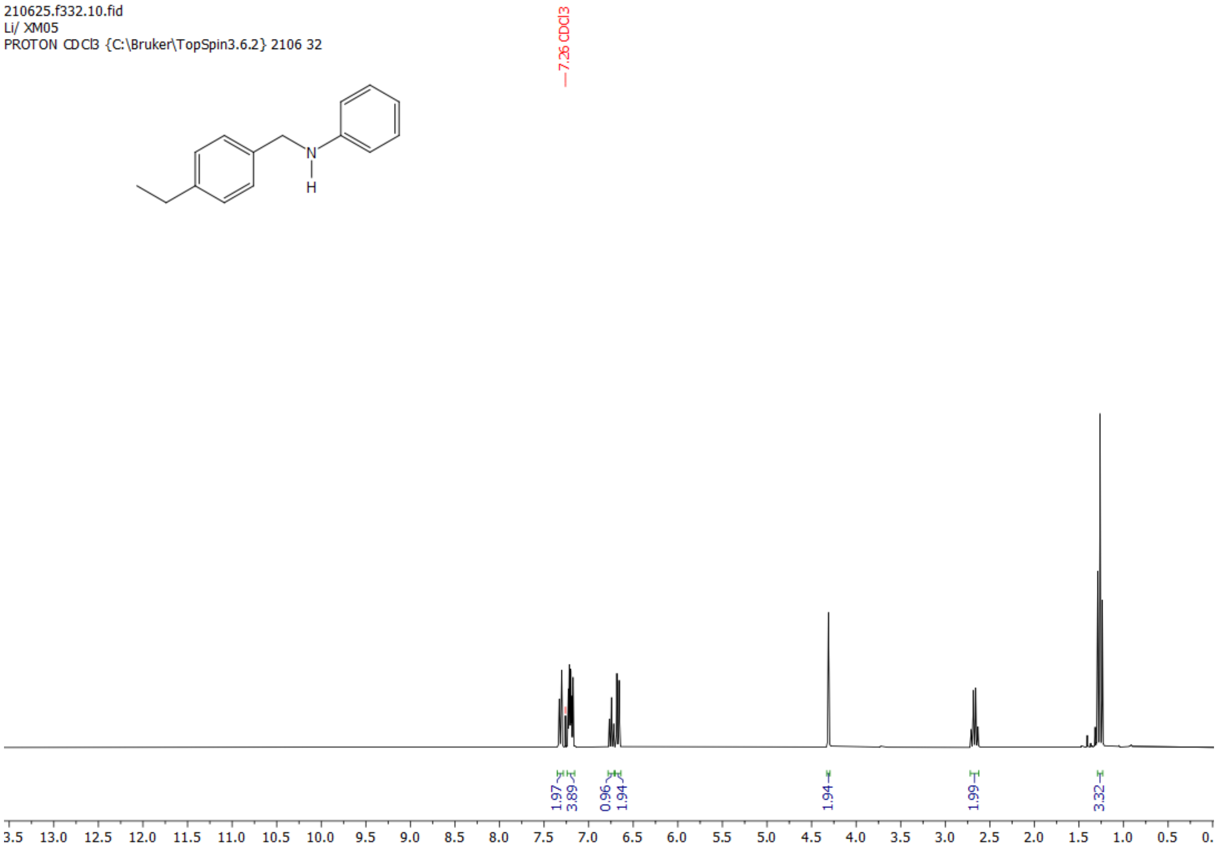
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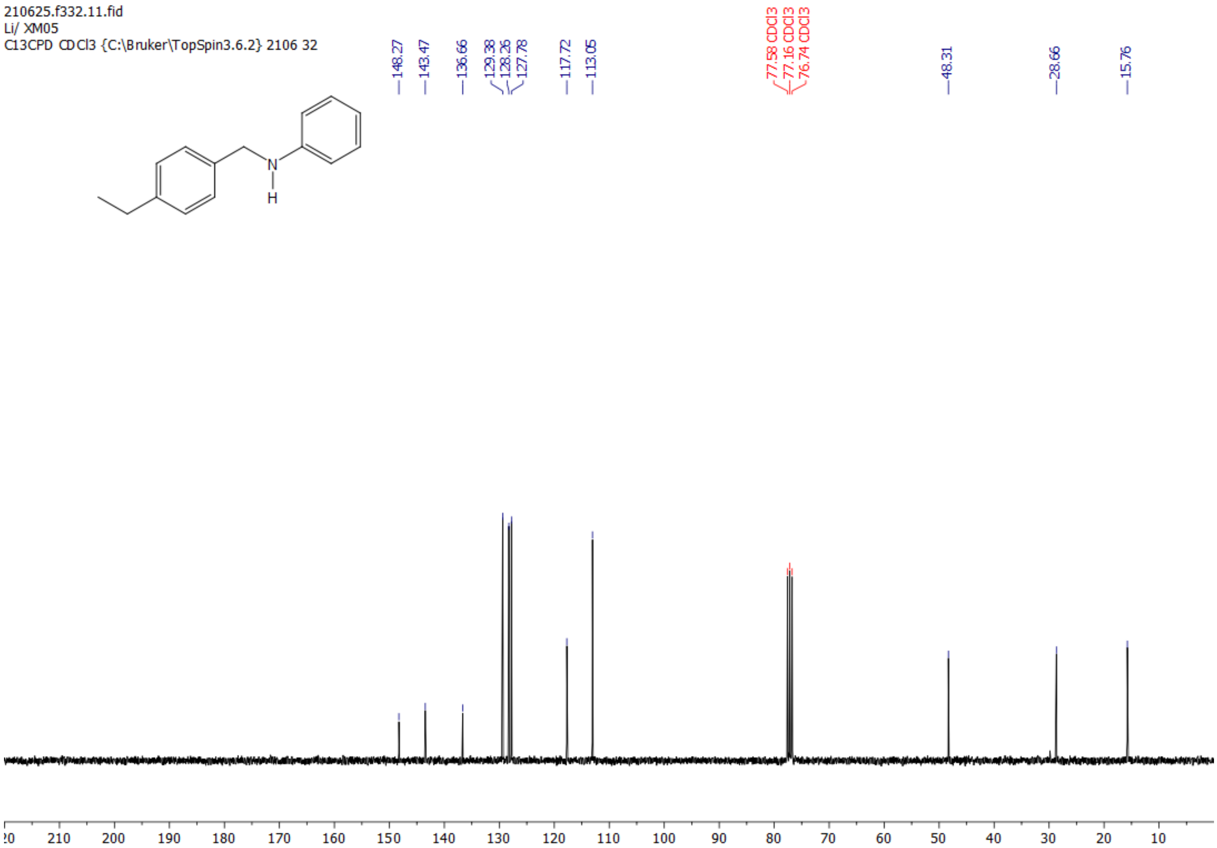
1.94
1.94
1.99
3.32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77.16 CDCl3
76.74 CDCl3
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N
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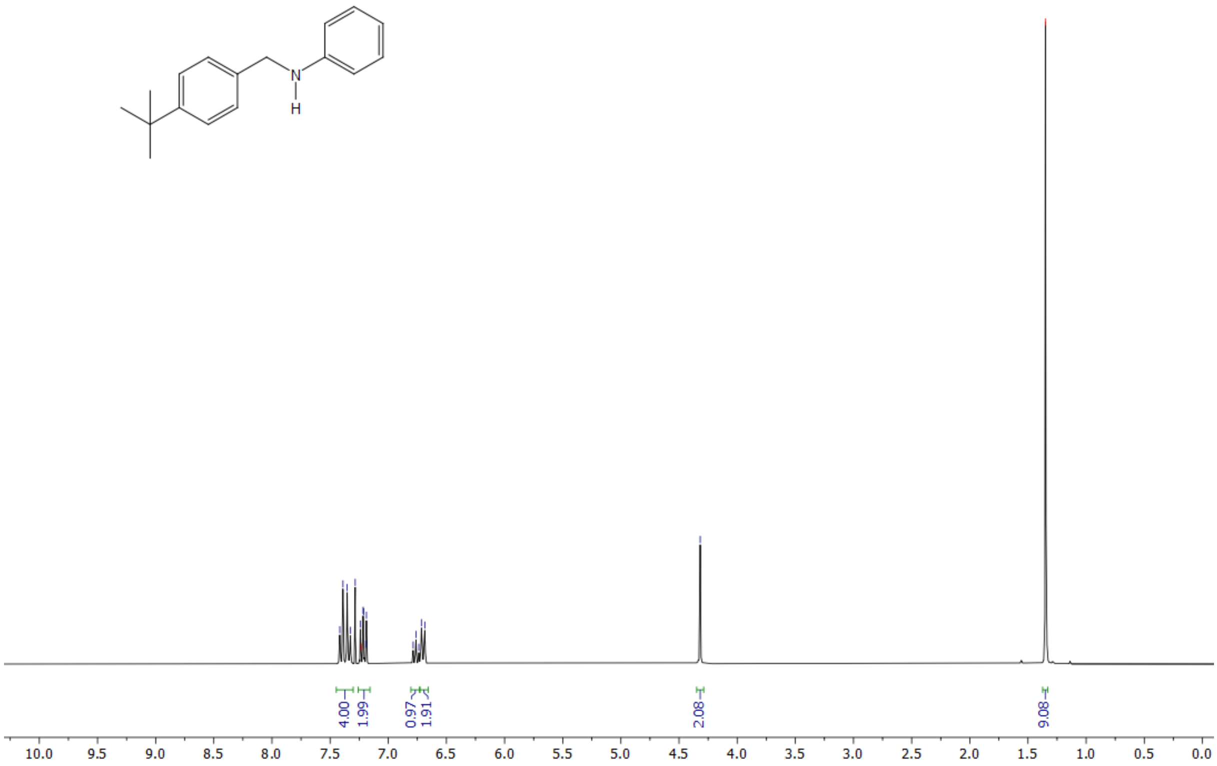

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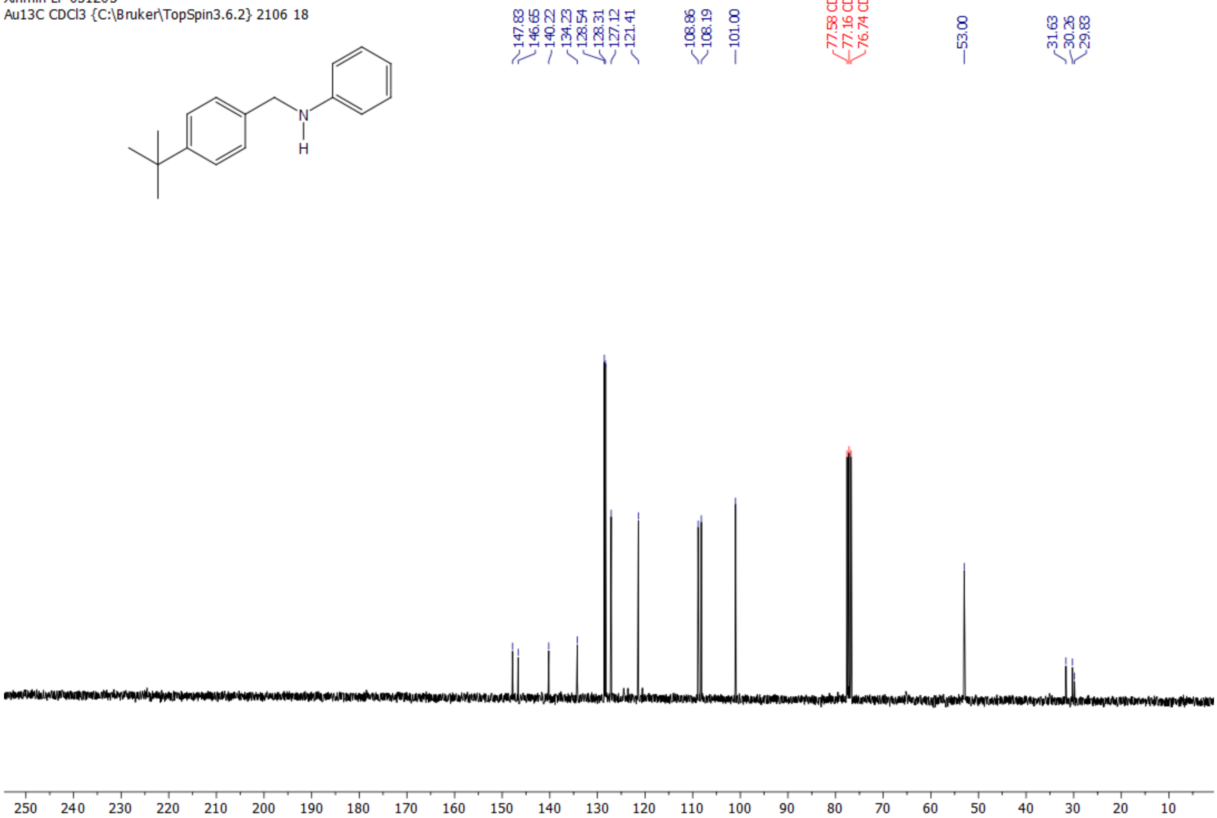

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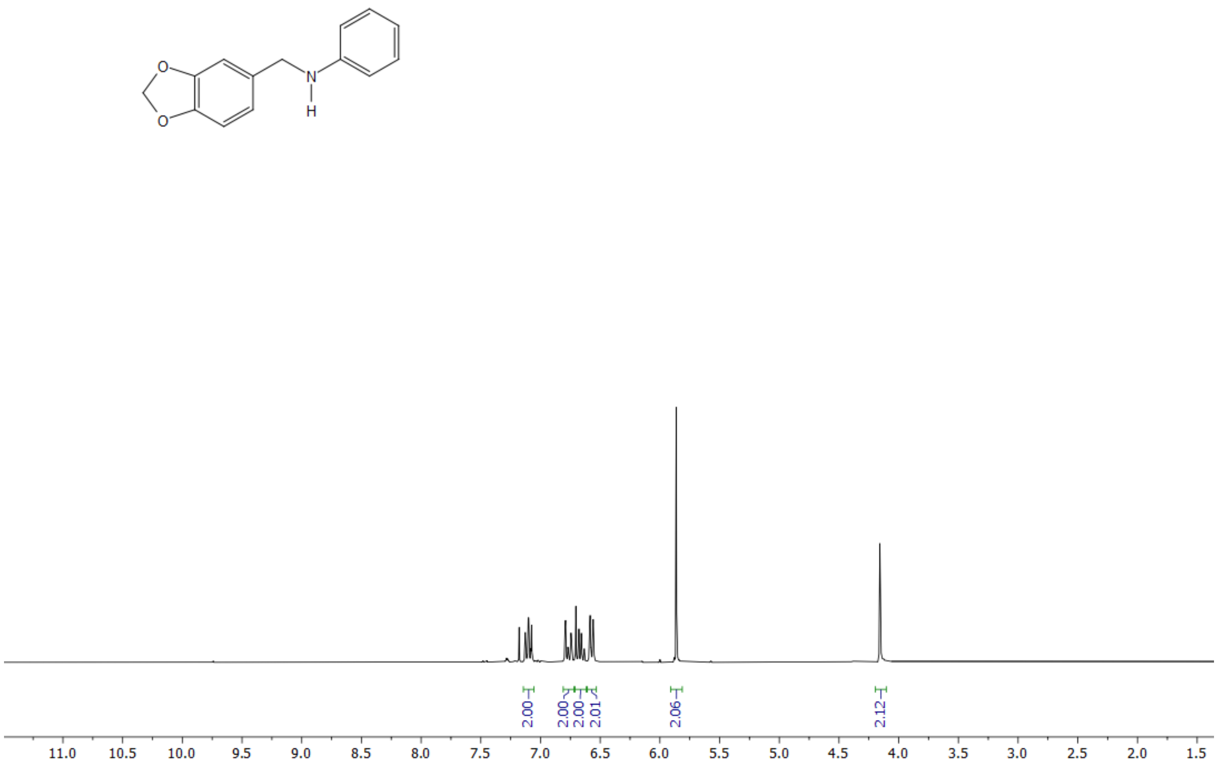

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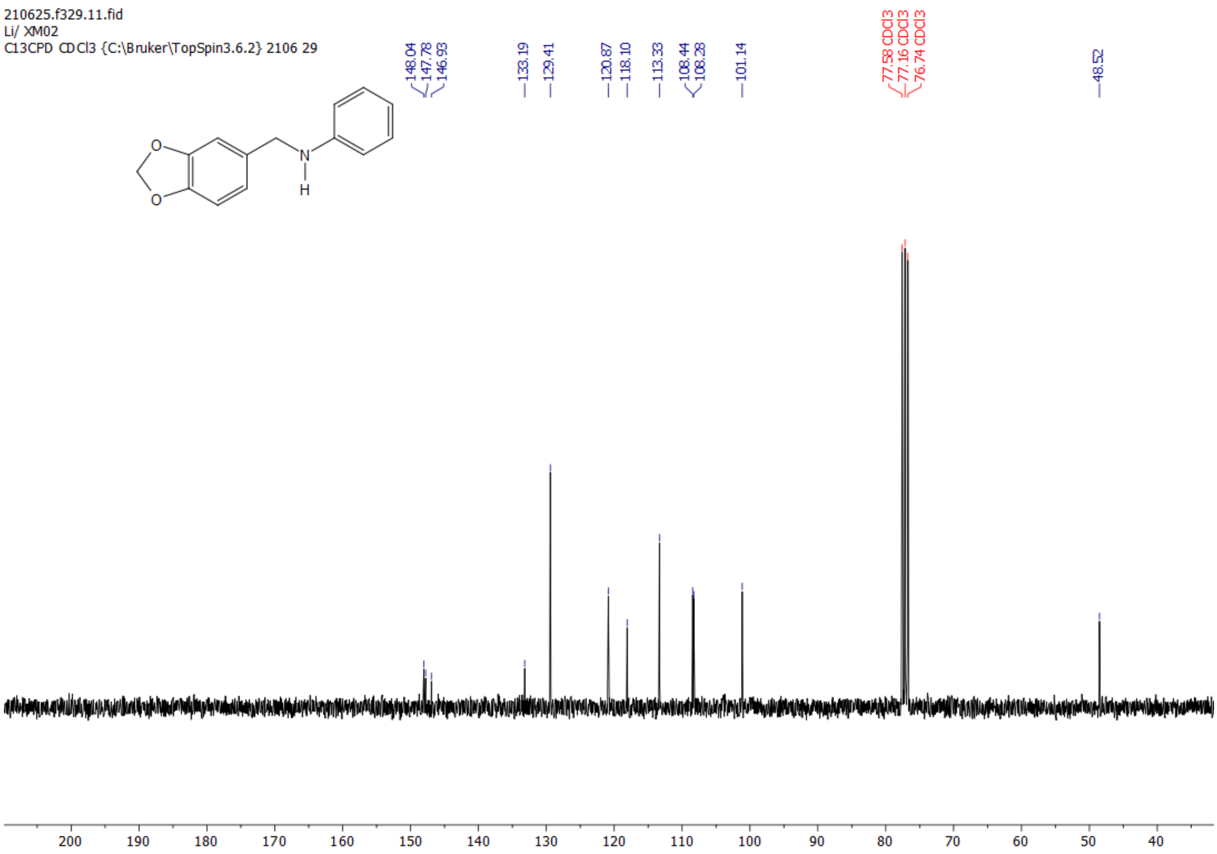

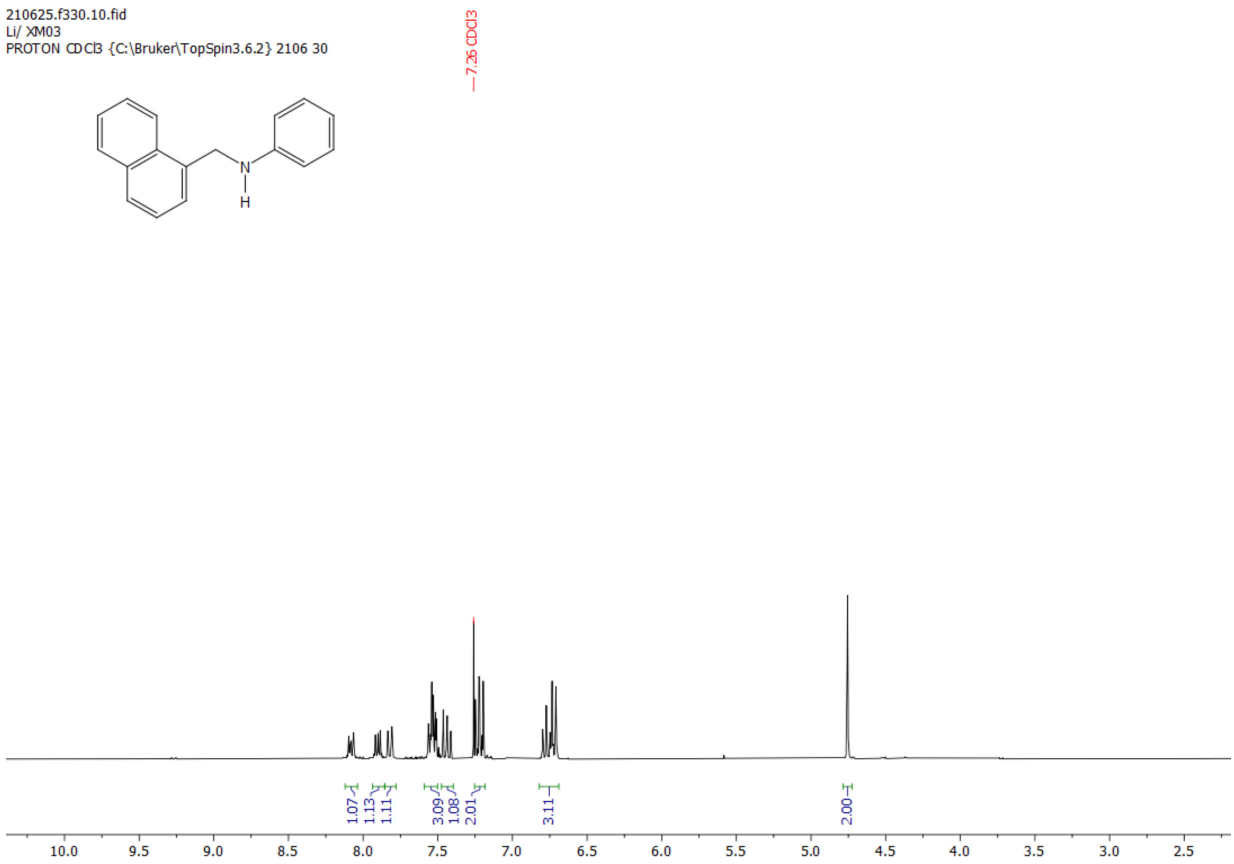
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3.11
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9.0
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4.5
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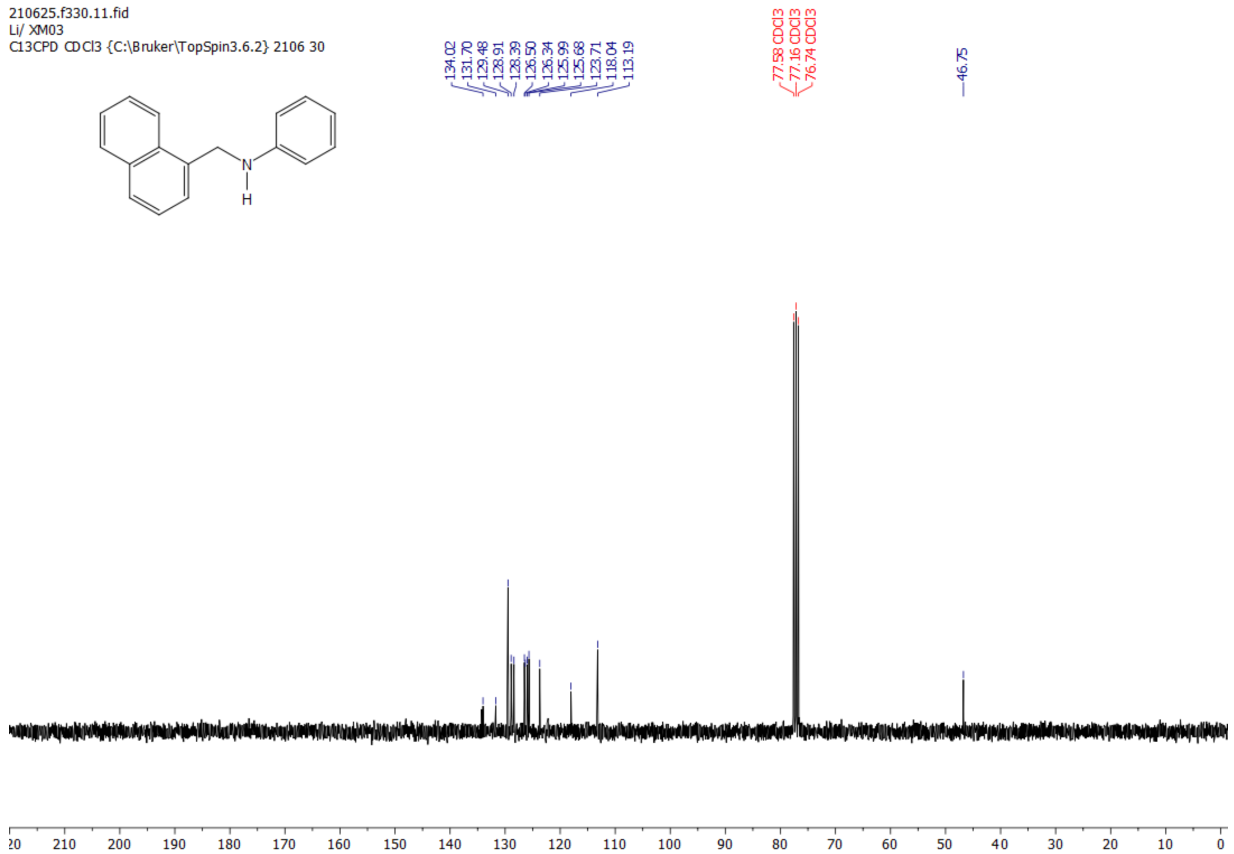
3.0
2.5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126.34
125.99
125.68
123.71
118.04
113.19
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
46.75
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
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10
0

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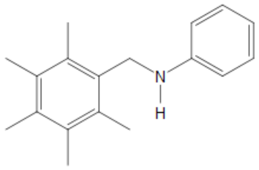

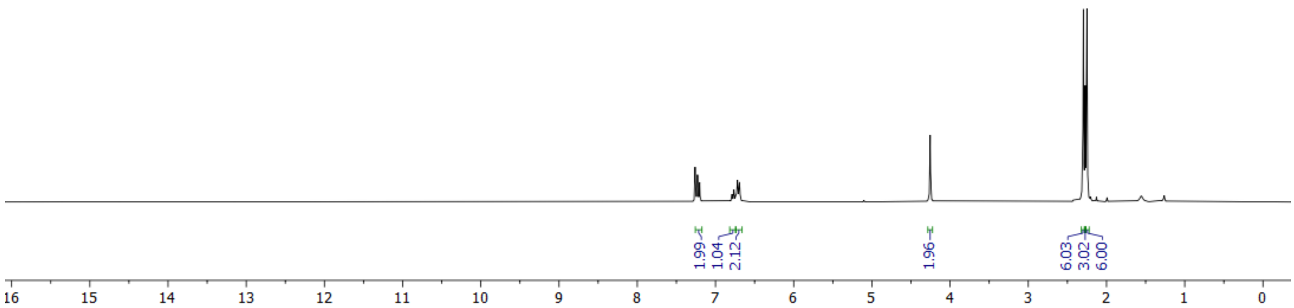

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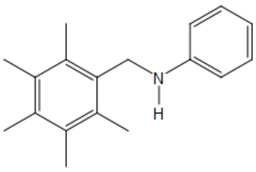

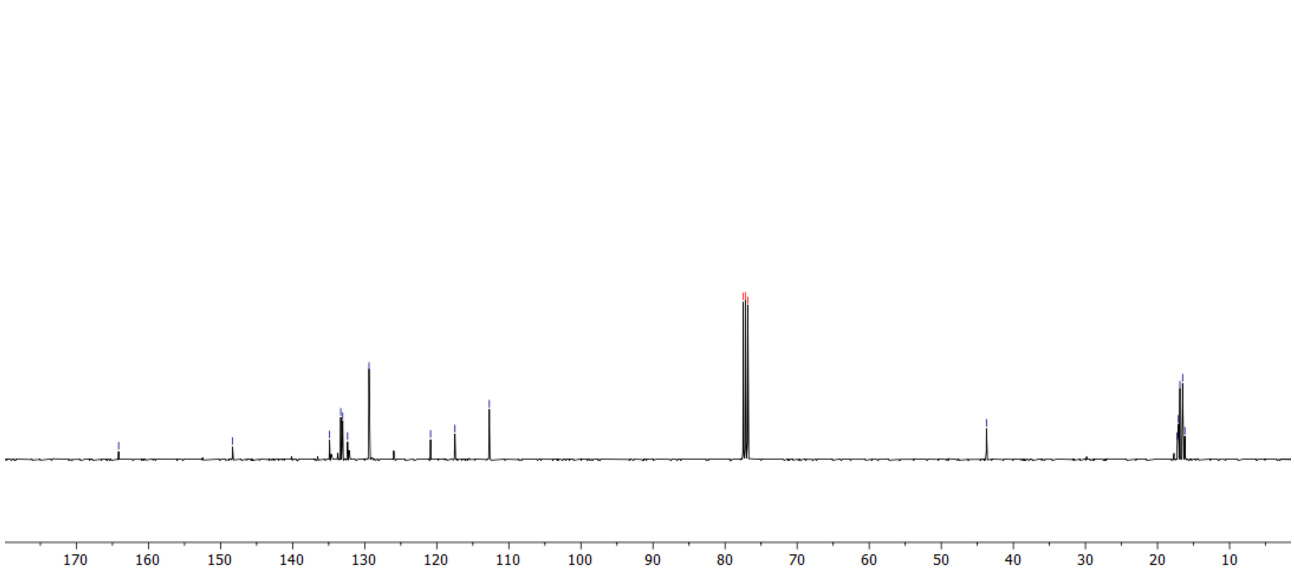

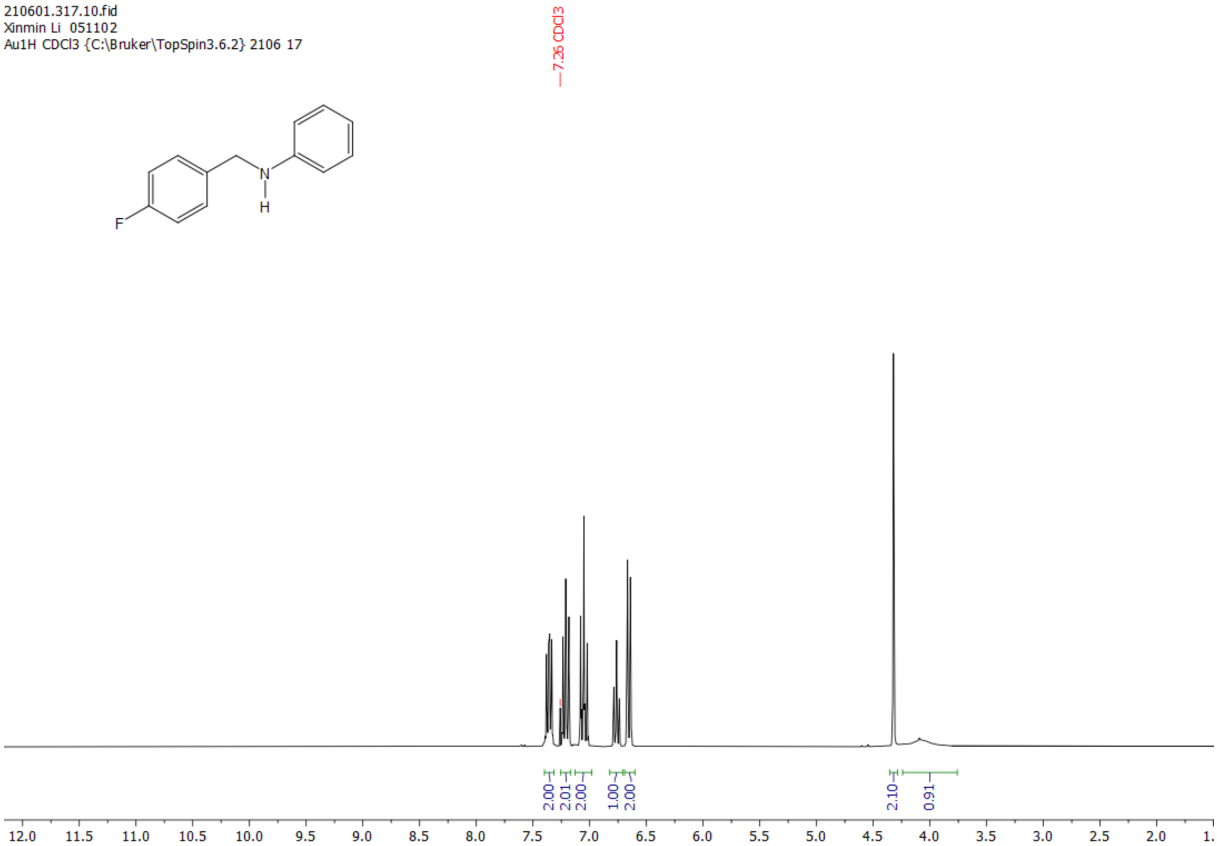
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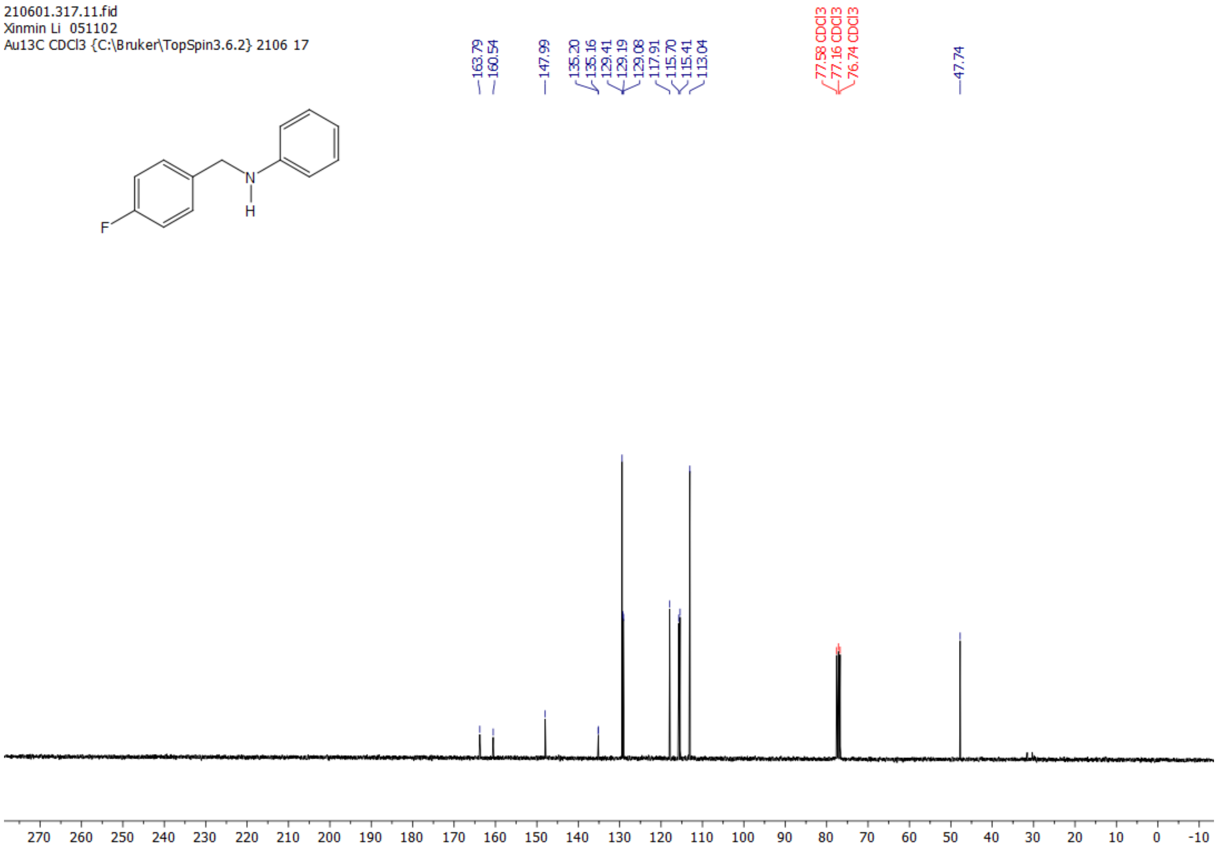
2.00
1.00
2.00
2.10
0.91
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.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129.41
129.19
129.08
117.91
115.70
115.41
113.04
77.58 CDCl3
77.16 CDCl3
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47.74
F
N
H
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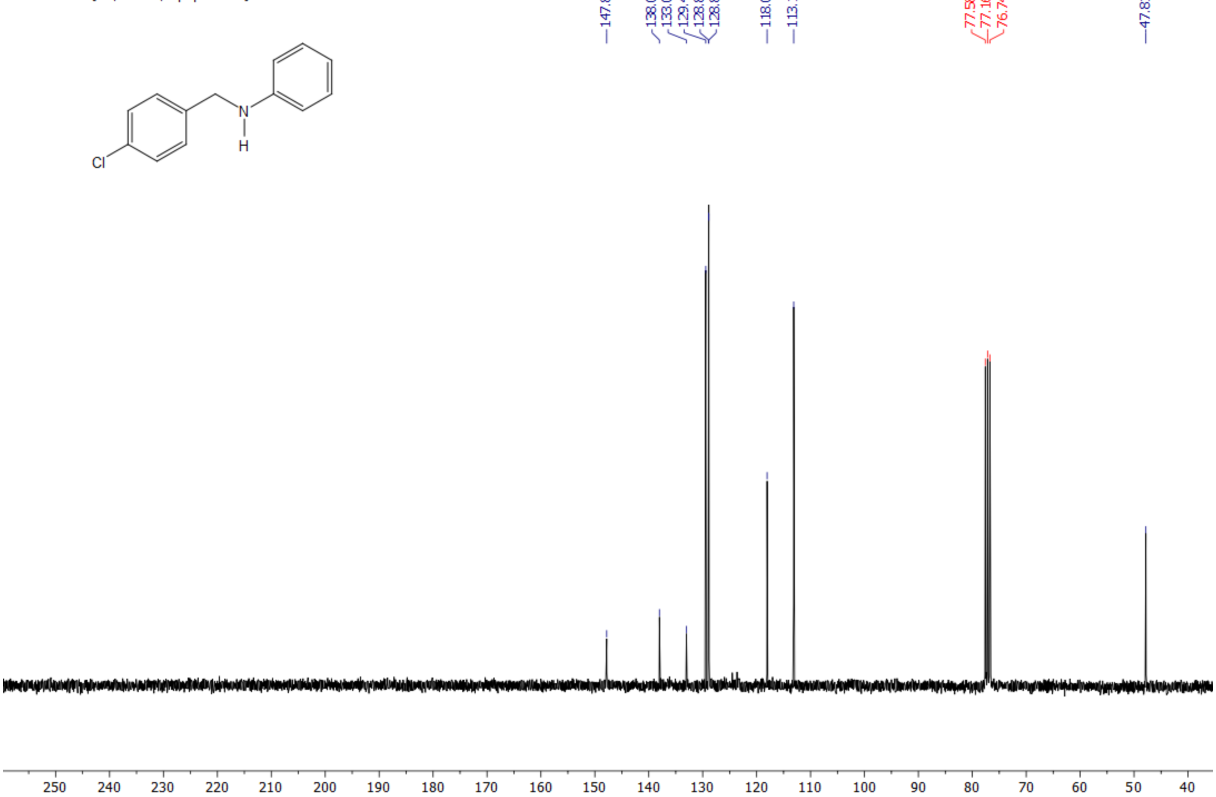

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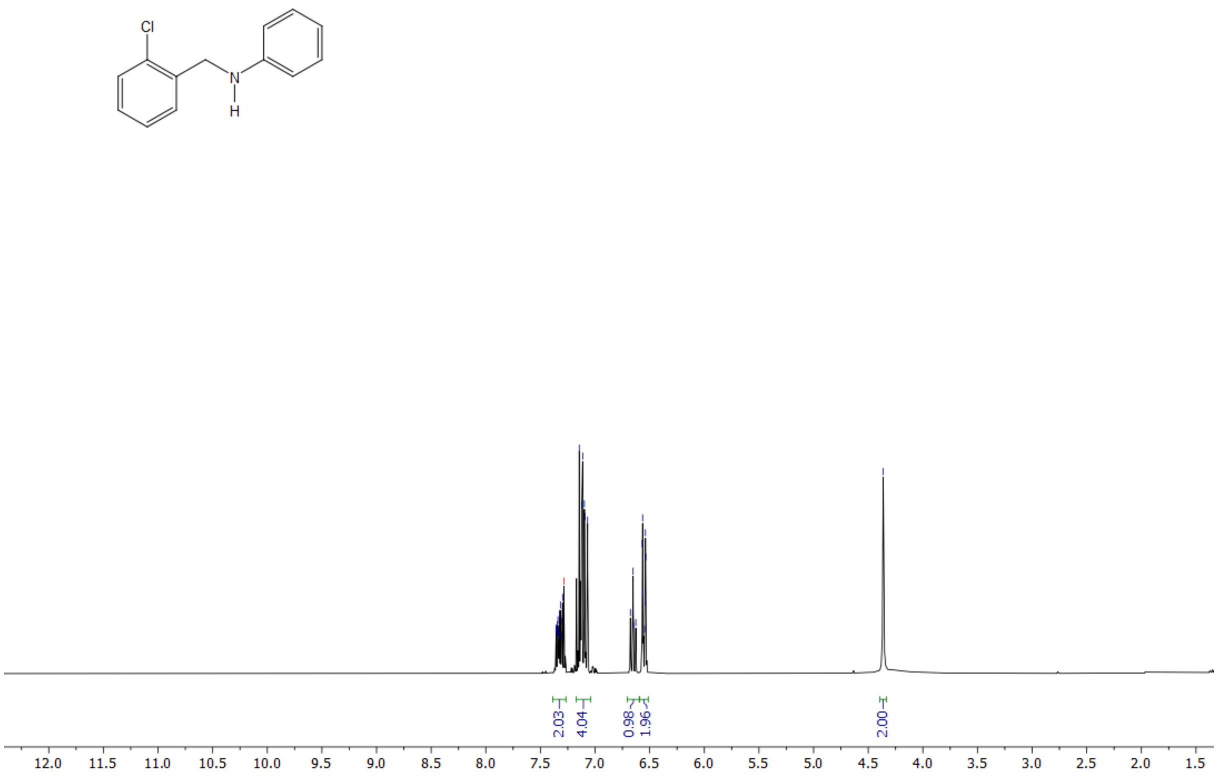

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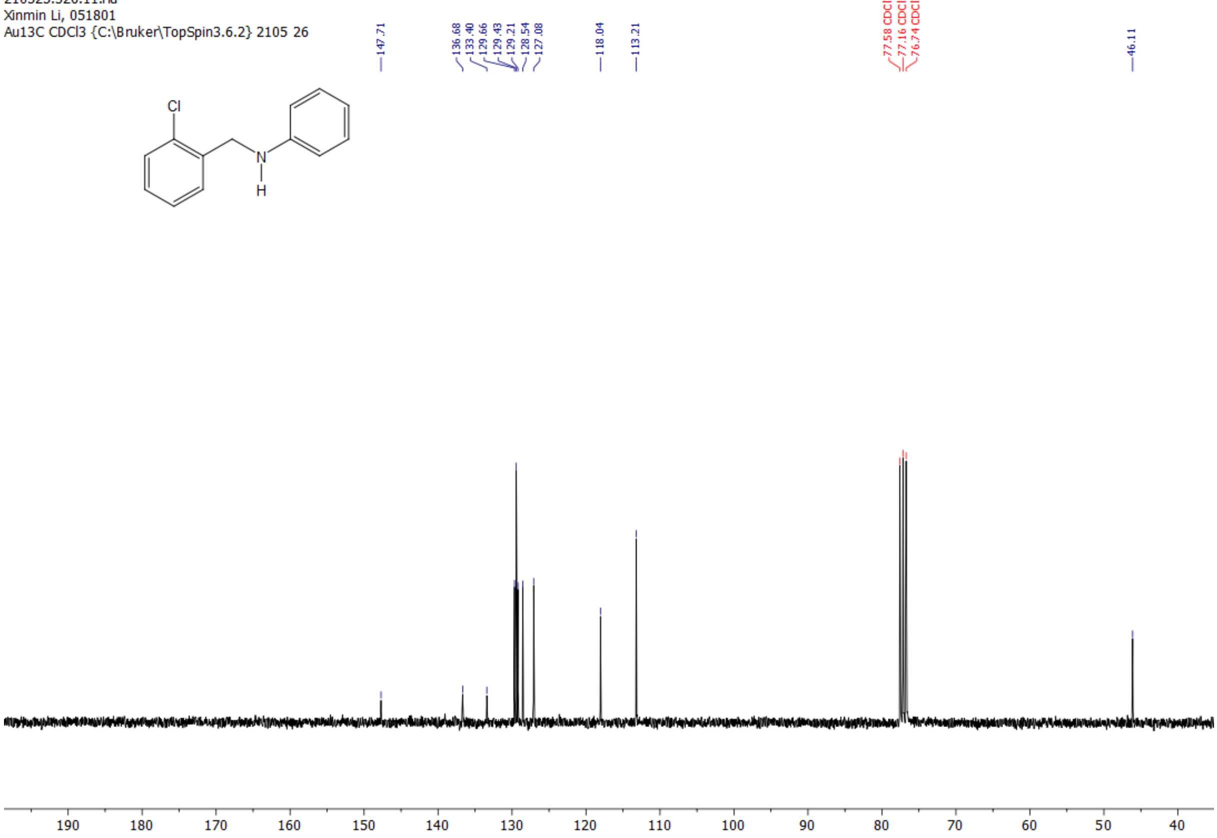

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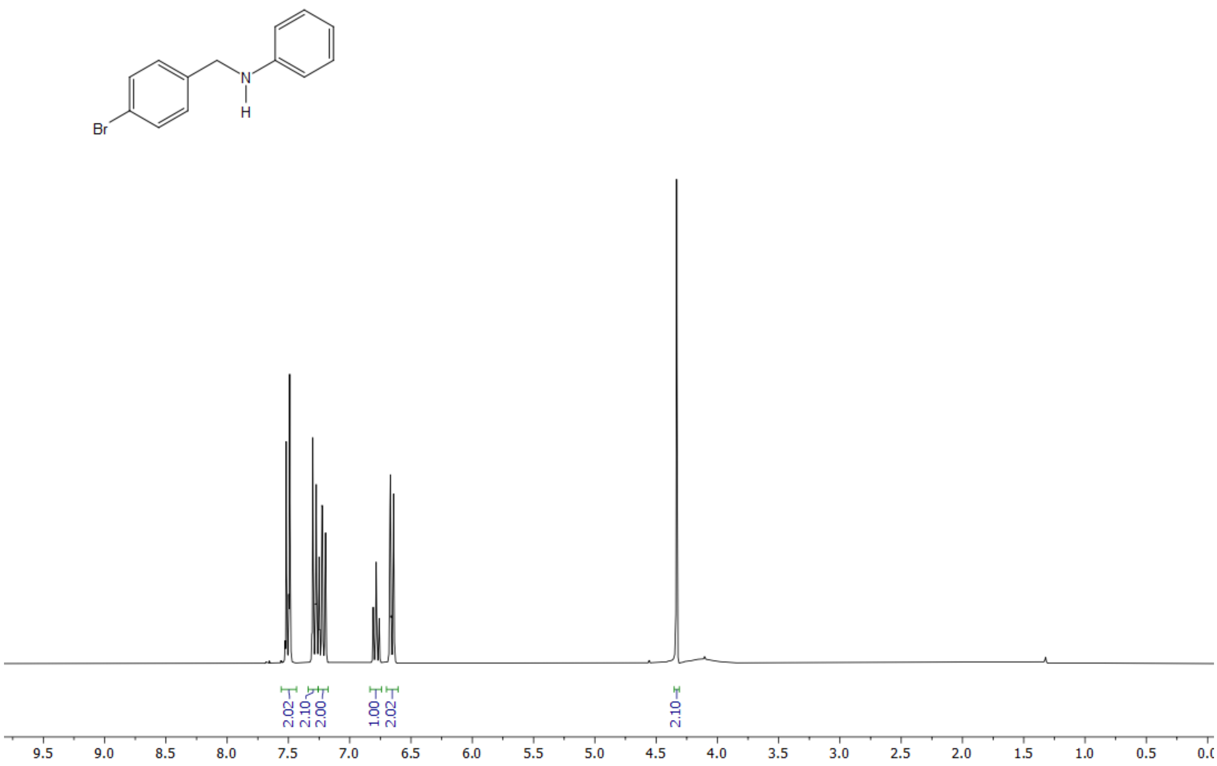

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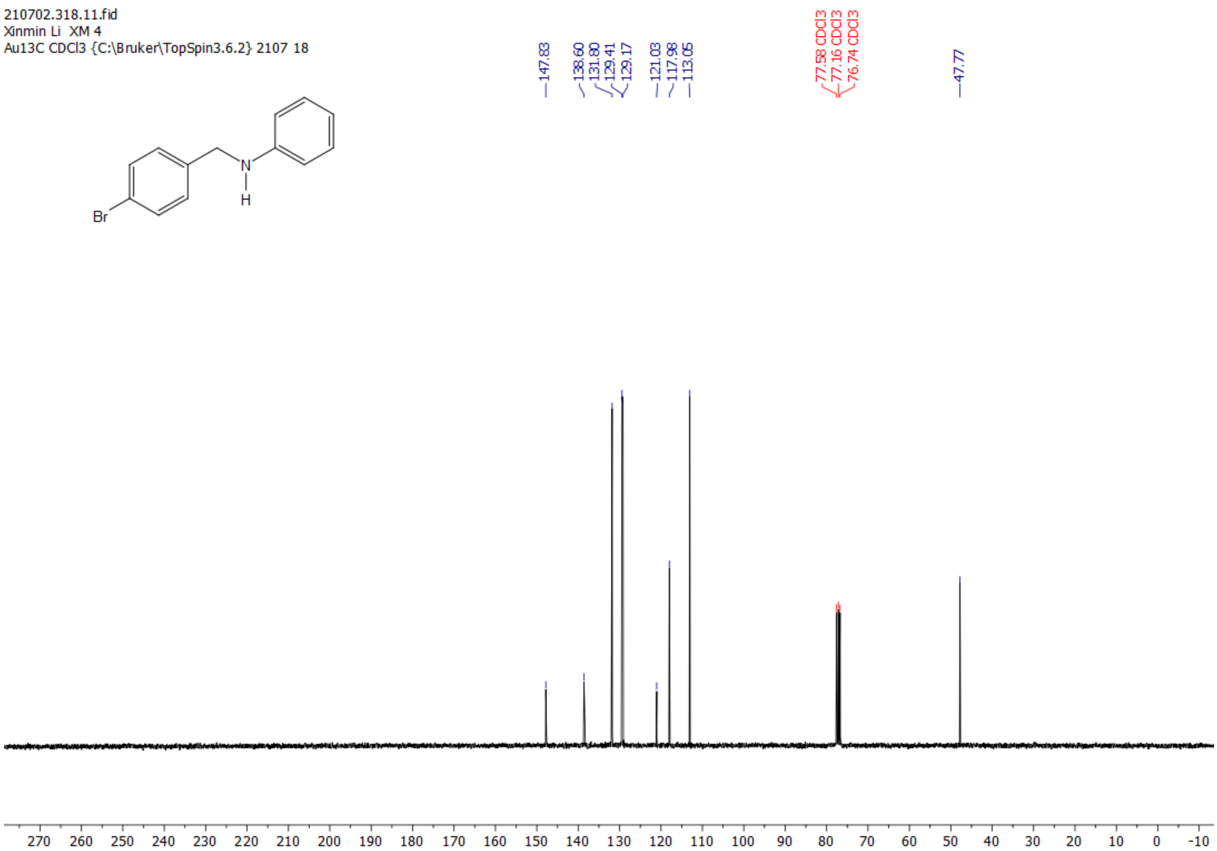

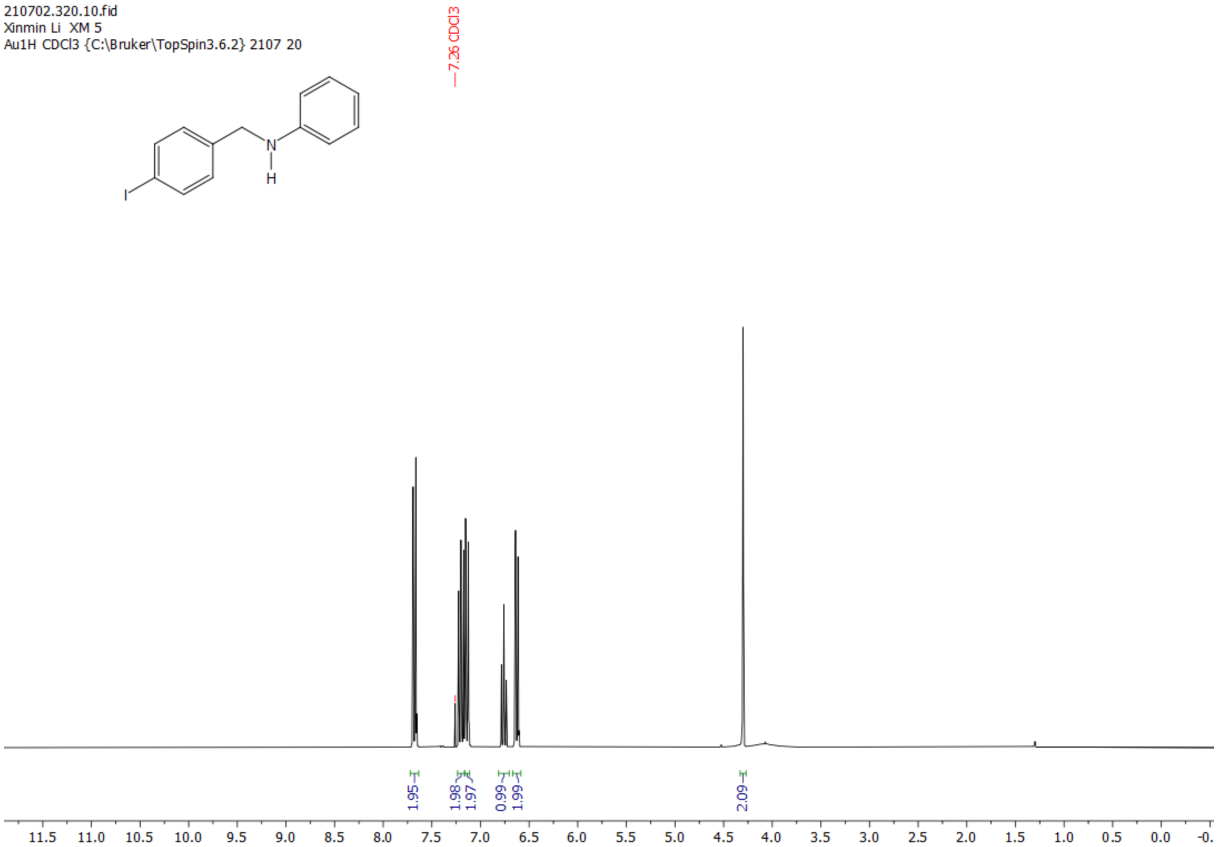
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H
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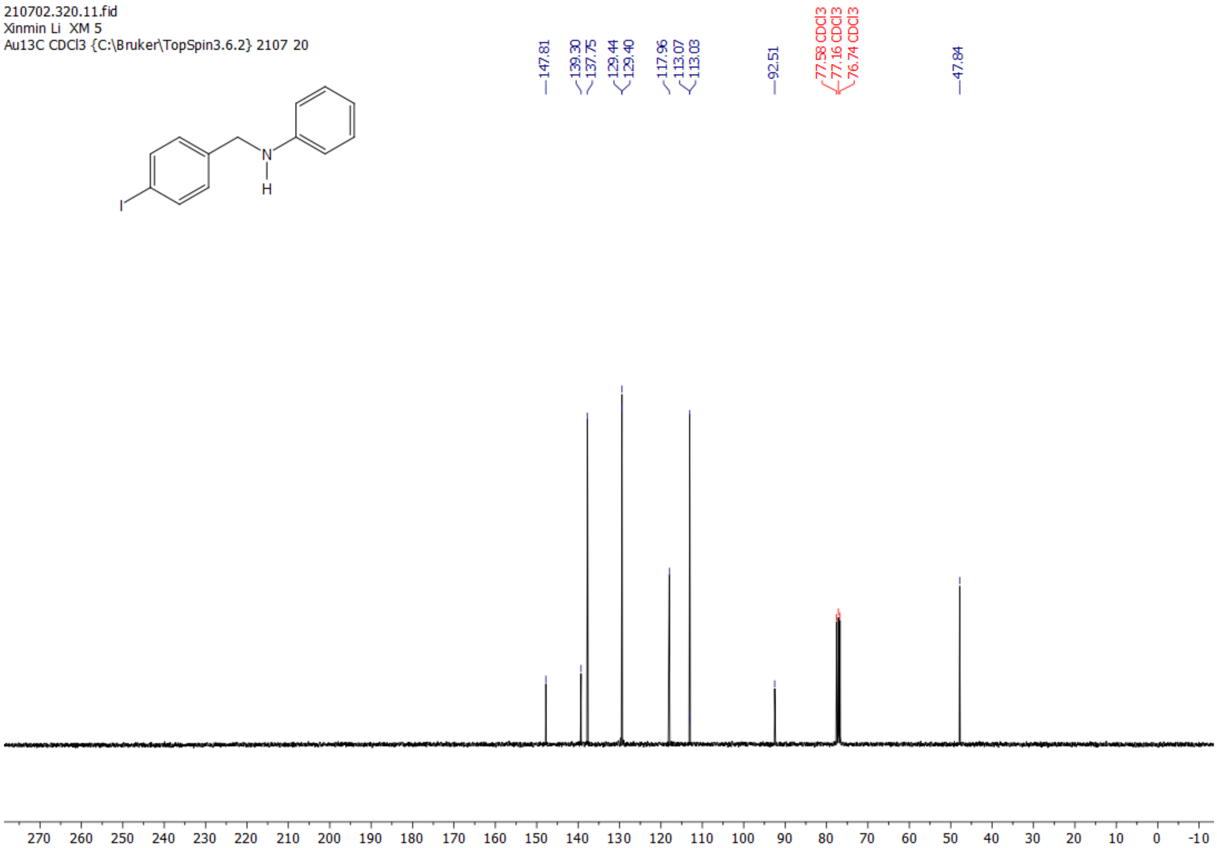
1.98
1.97
0.99
1.99
2.09
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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Xinmin Li XM 5
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113.07
113.03
92.51
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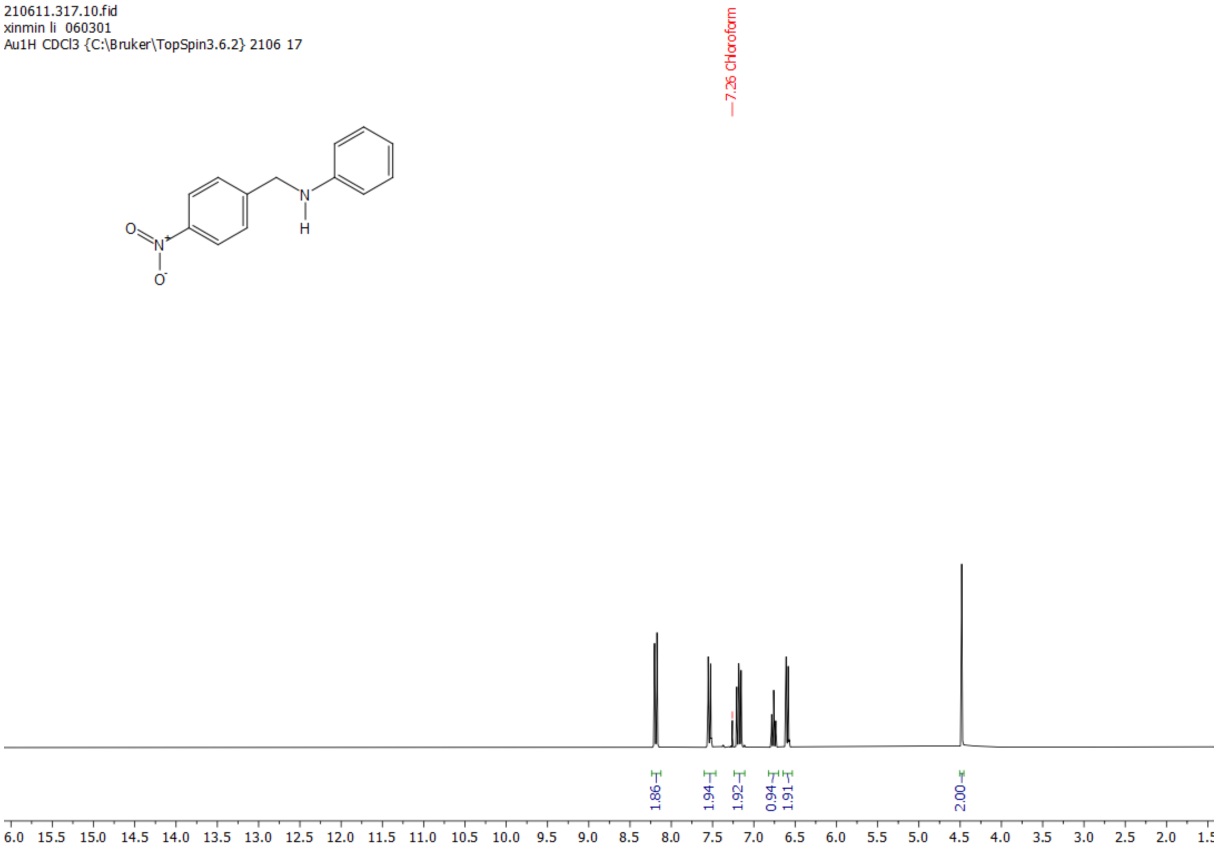
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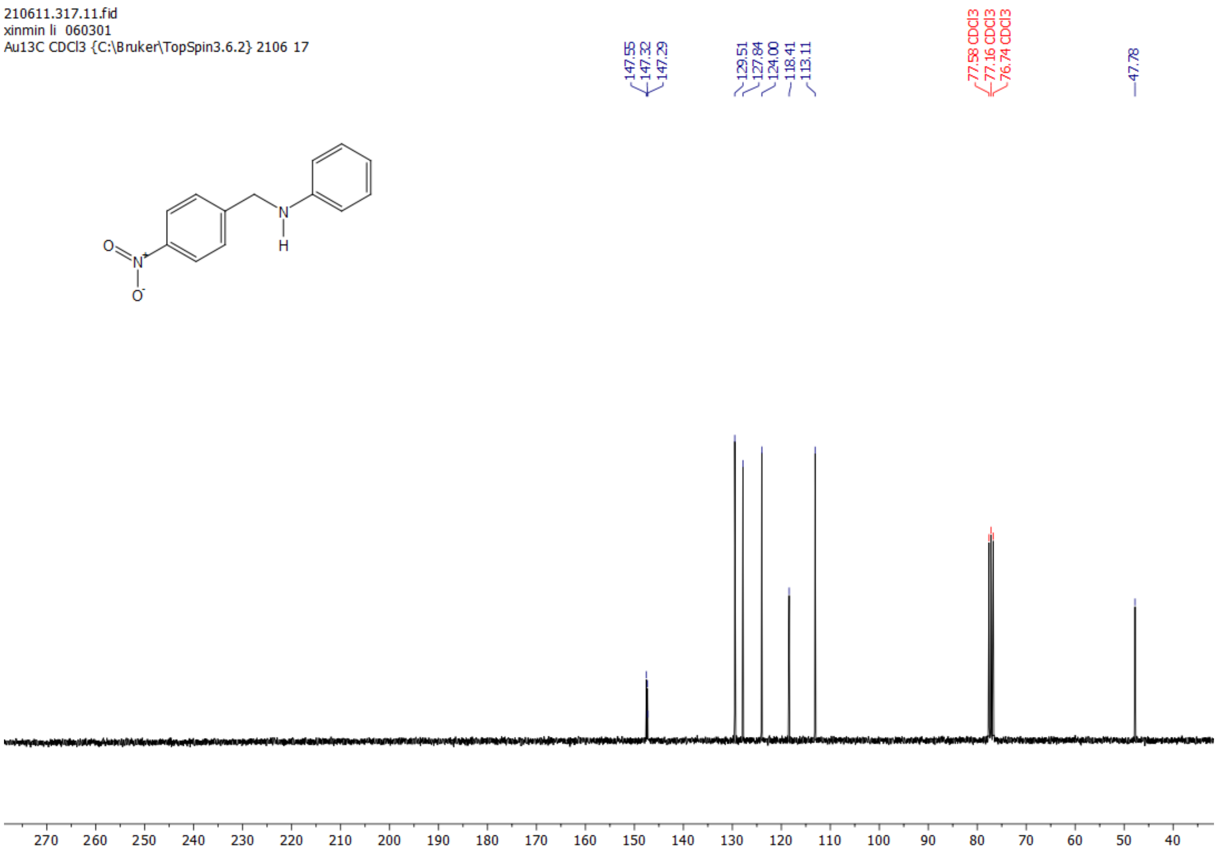
1.91
2.00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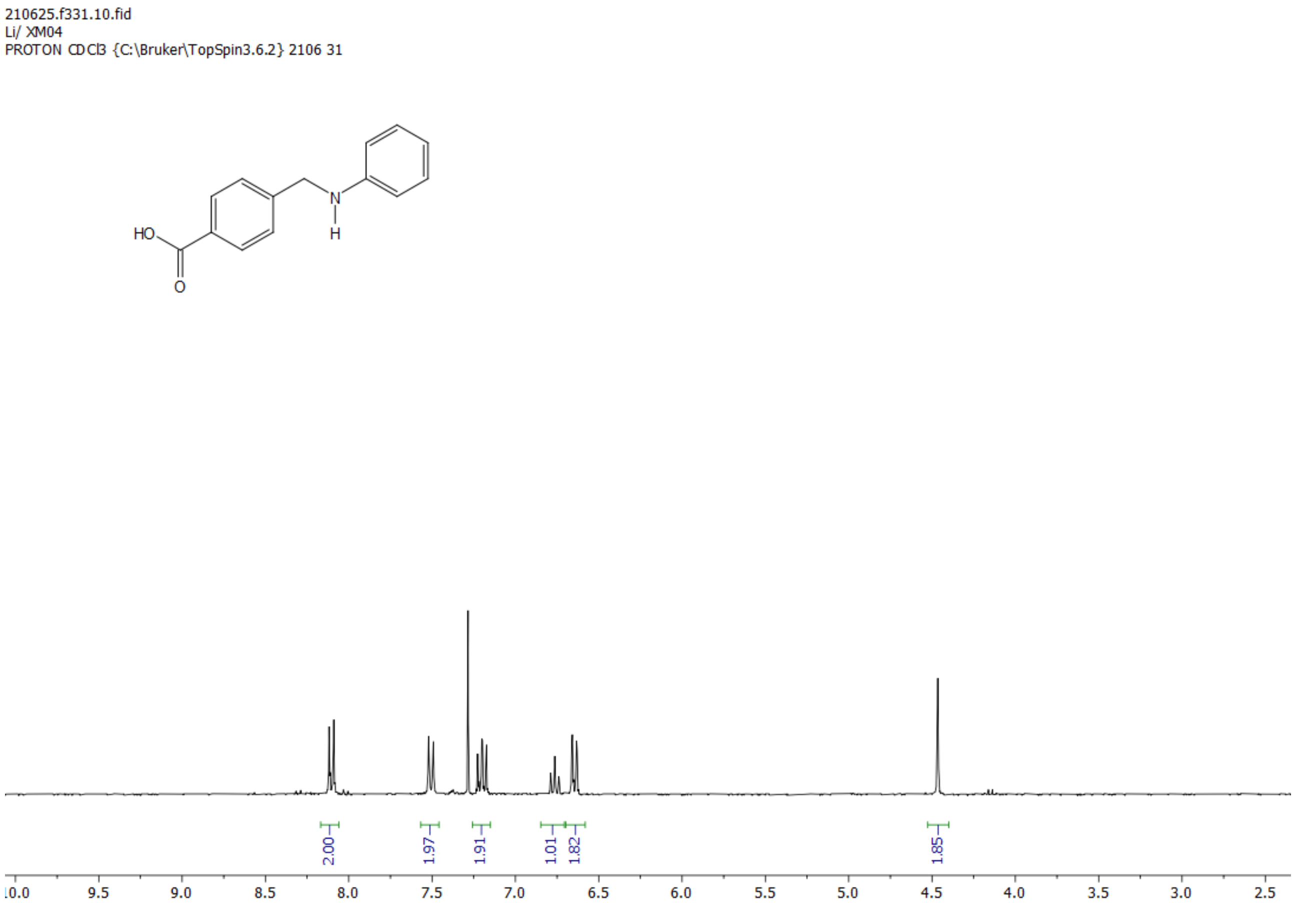
47.78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1.85
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9.0
8.5
8.0
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6.5
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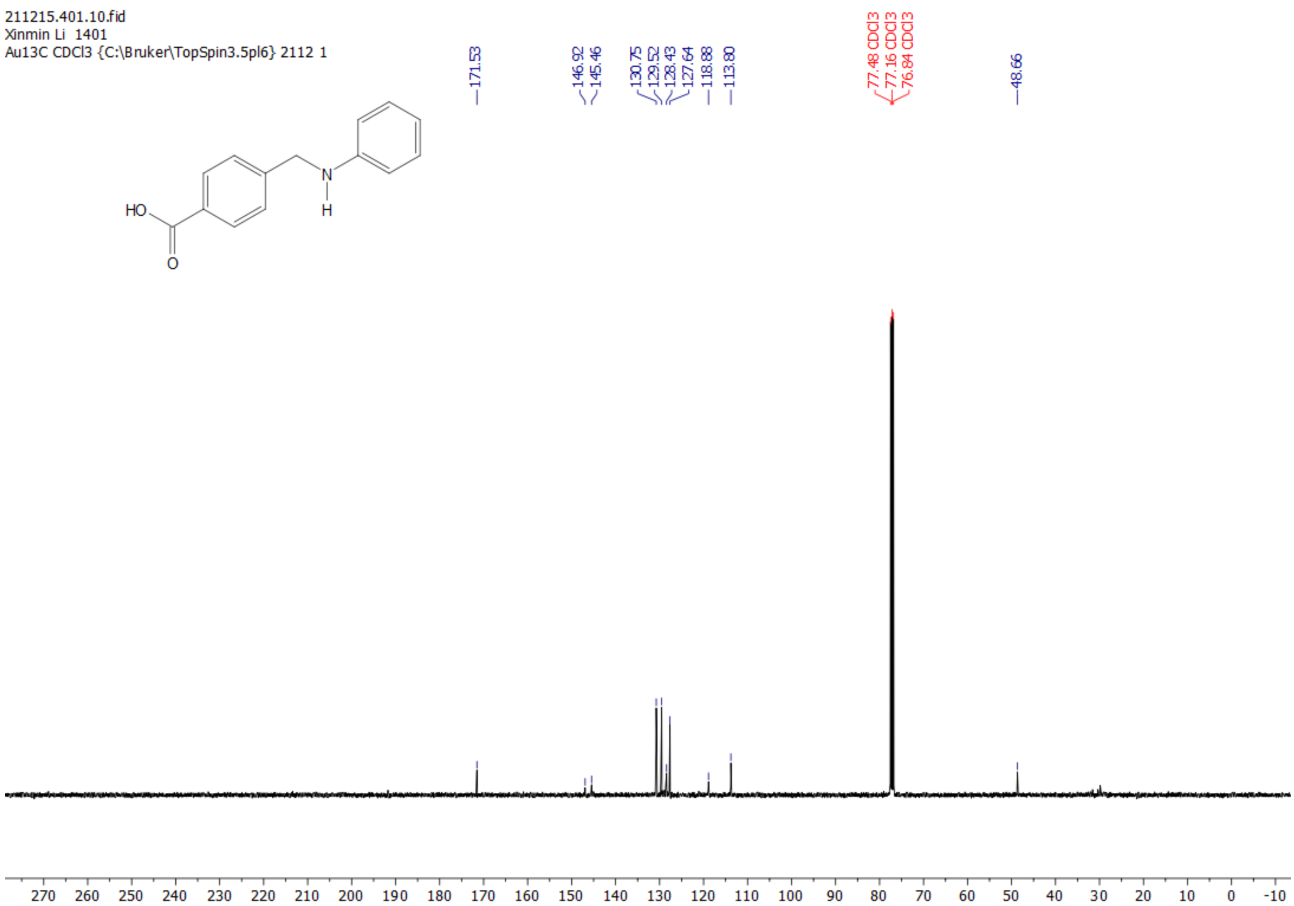
5.5
5.0
4.5
4.0
3.5
3.0
2.5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Xinmin Li 1401
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2112 1
171.53
146.92
145.46
130.75
129.52
128.43
127.64
118.88
113.80
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
48.66
HO
O
N
H
270
260
250
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

210701.404.10.fid
Xinmin Li, XM 1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2107 4

210701.404.11.fid
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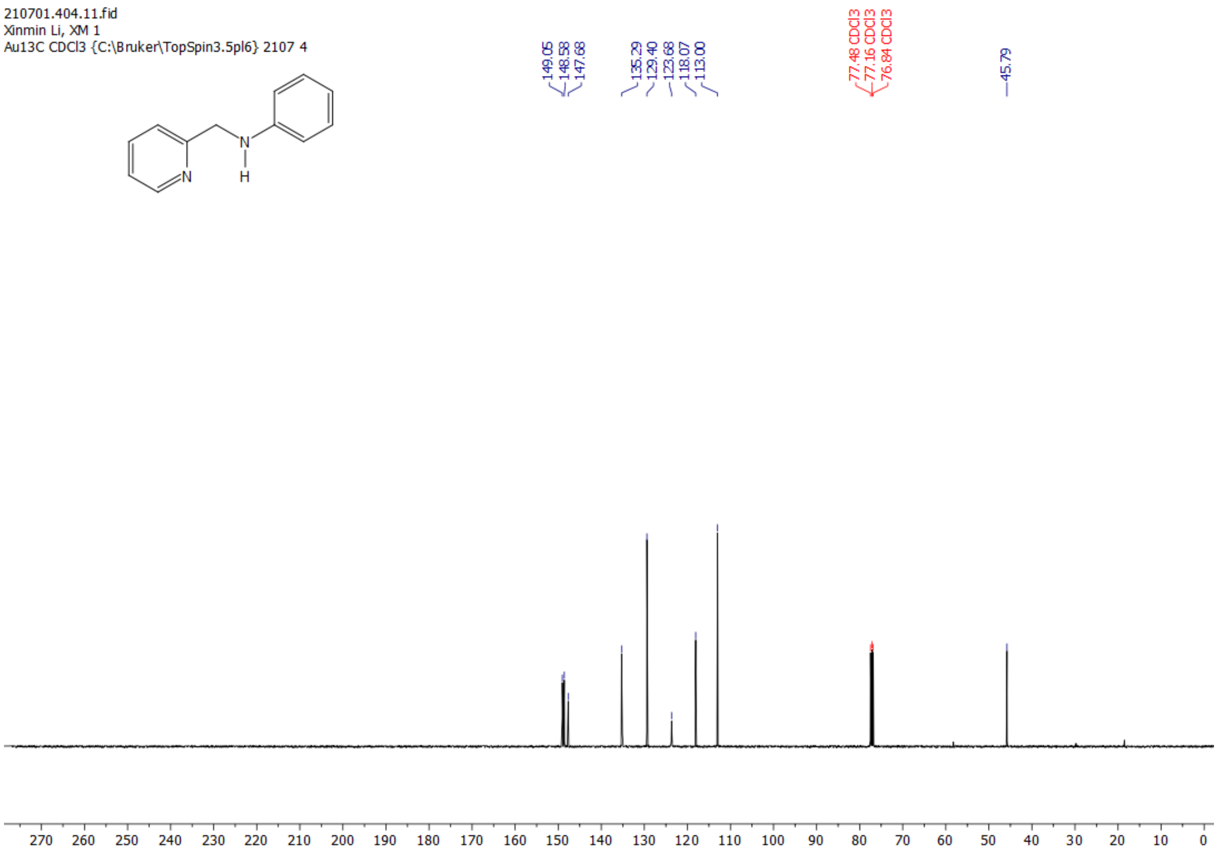
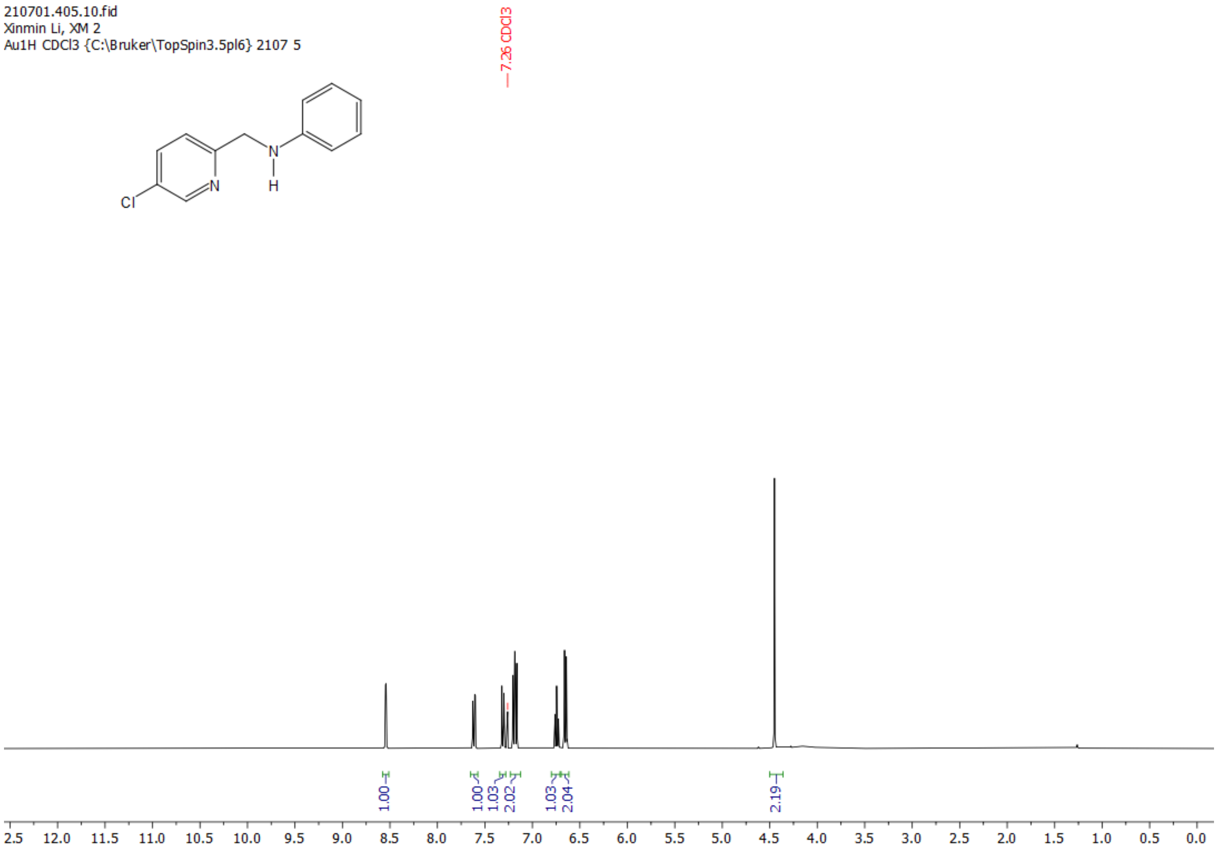
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2107 4

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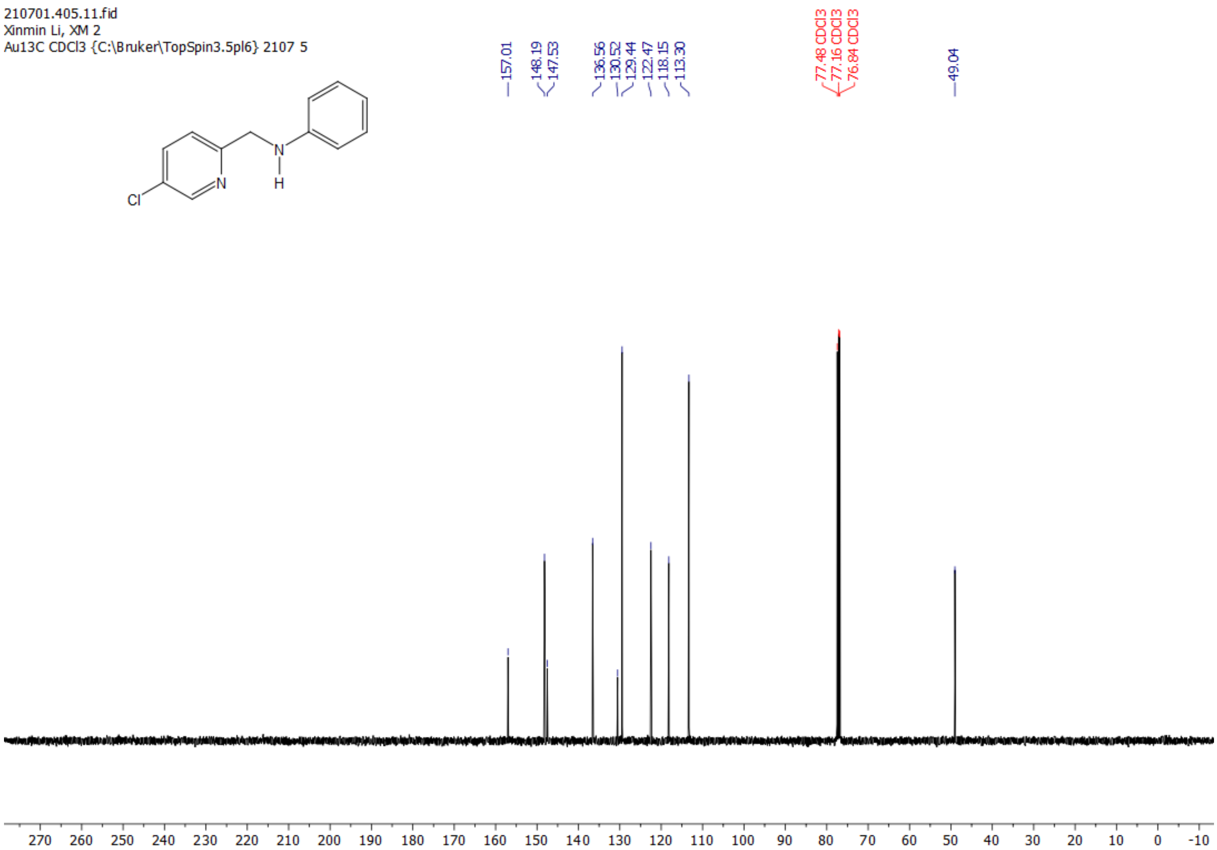
Xinmin Li, XM 2
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2107 5
Cl
N
N
H
7.26 CDCl3
1.00
1.00
1.03
2.02
1.03
2.04
2.19
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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Xinmin Li, XM 2
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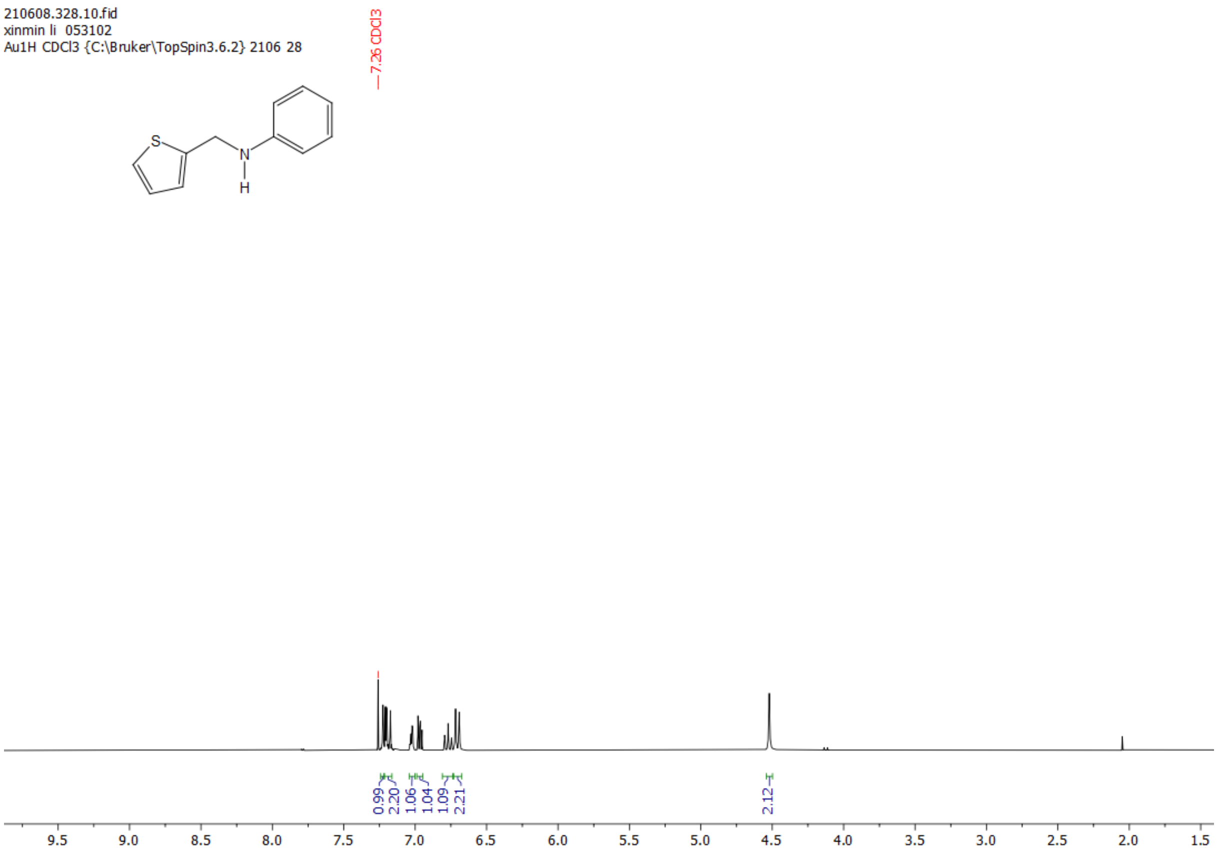
Cl
N
N
H
157.01
148.19
147.53
136.56
130.52
129.44
122.47
118.15
113.30
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
49.04
270 260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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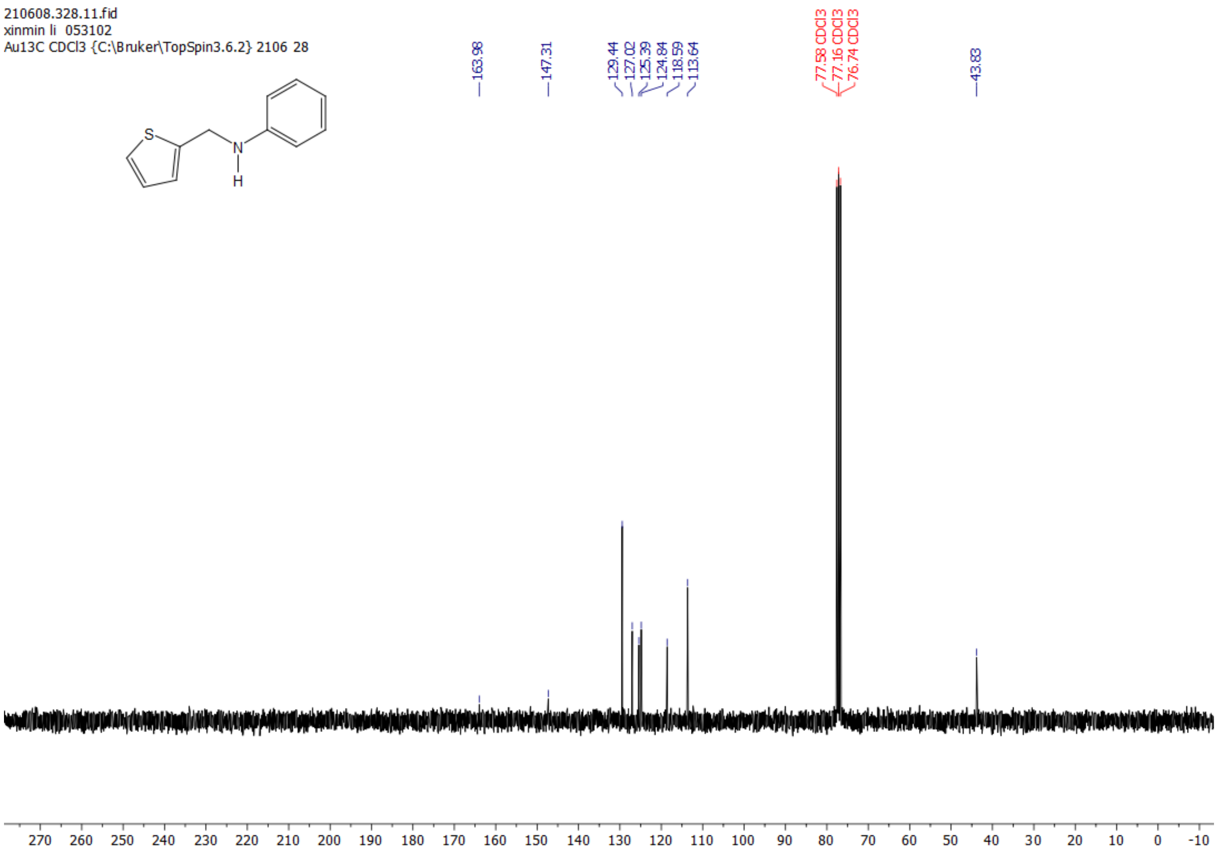
xinmin li 053102
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 28
7.26 CDCl3
S
N
H
0.99
2.20
1.06
1.04
1.09
2.21
2.12
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5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Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 28
163.98
147.31
129.44
127.02
125.39
124.84
118.59
113.64
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
43.83
S
N
H
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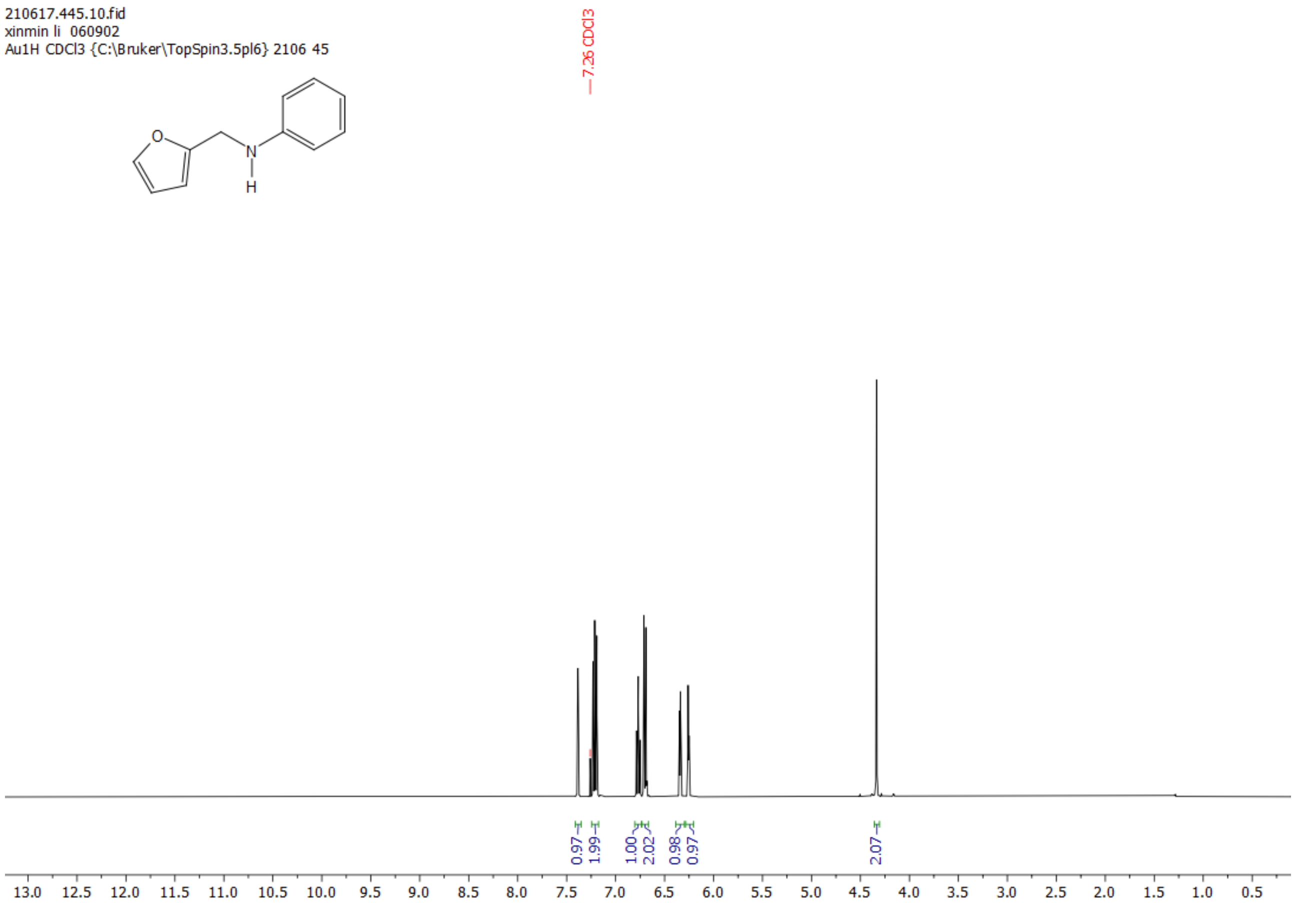
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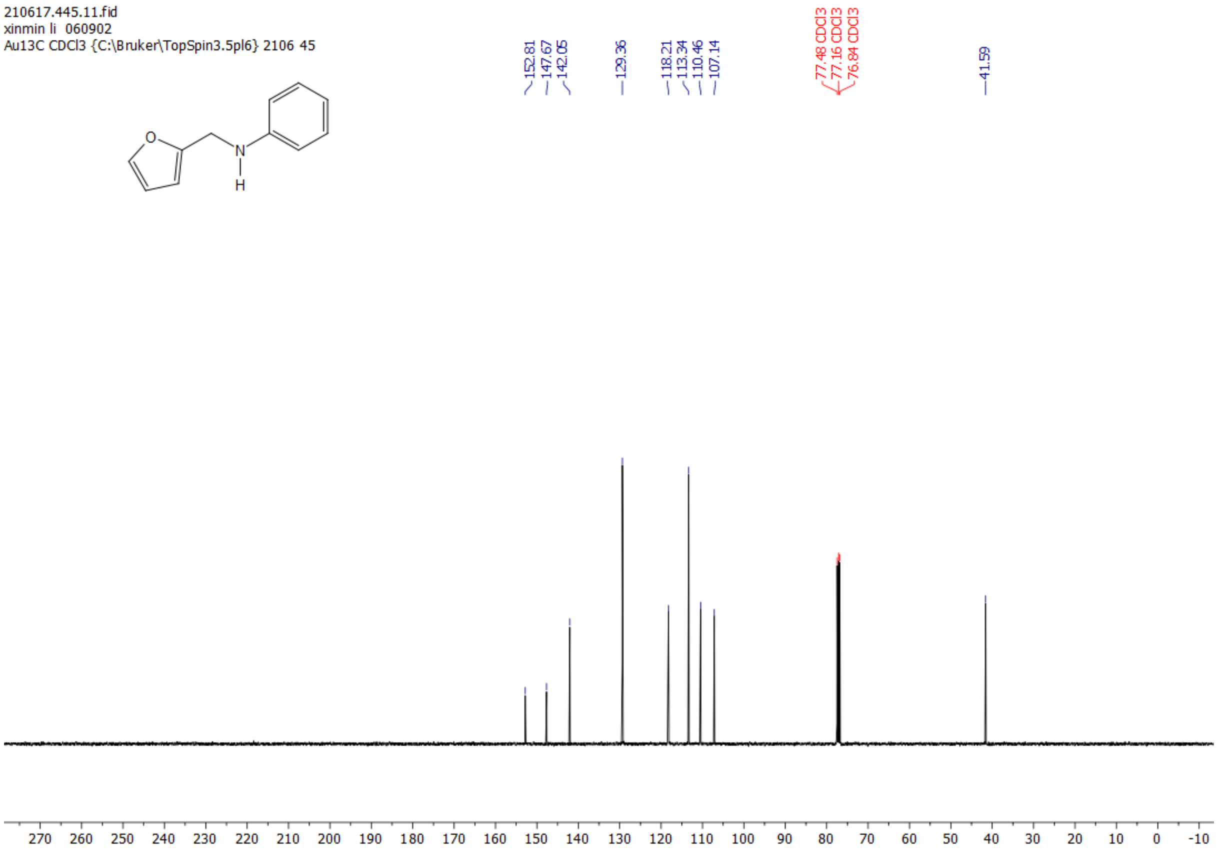
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 45
7.26 CDCl3
0.97
1.99
1.00
2.02
0.98
0.97
2.07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113.34
110.46
107.14
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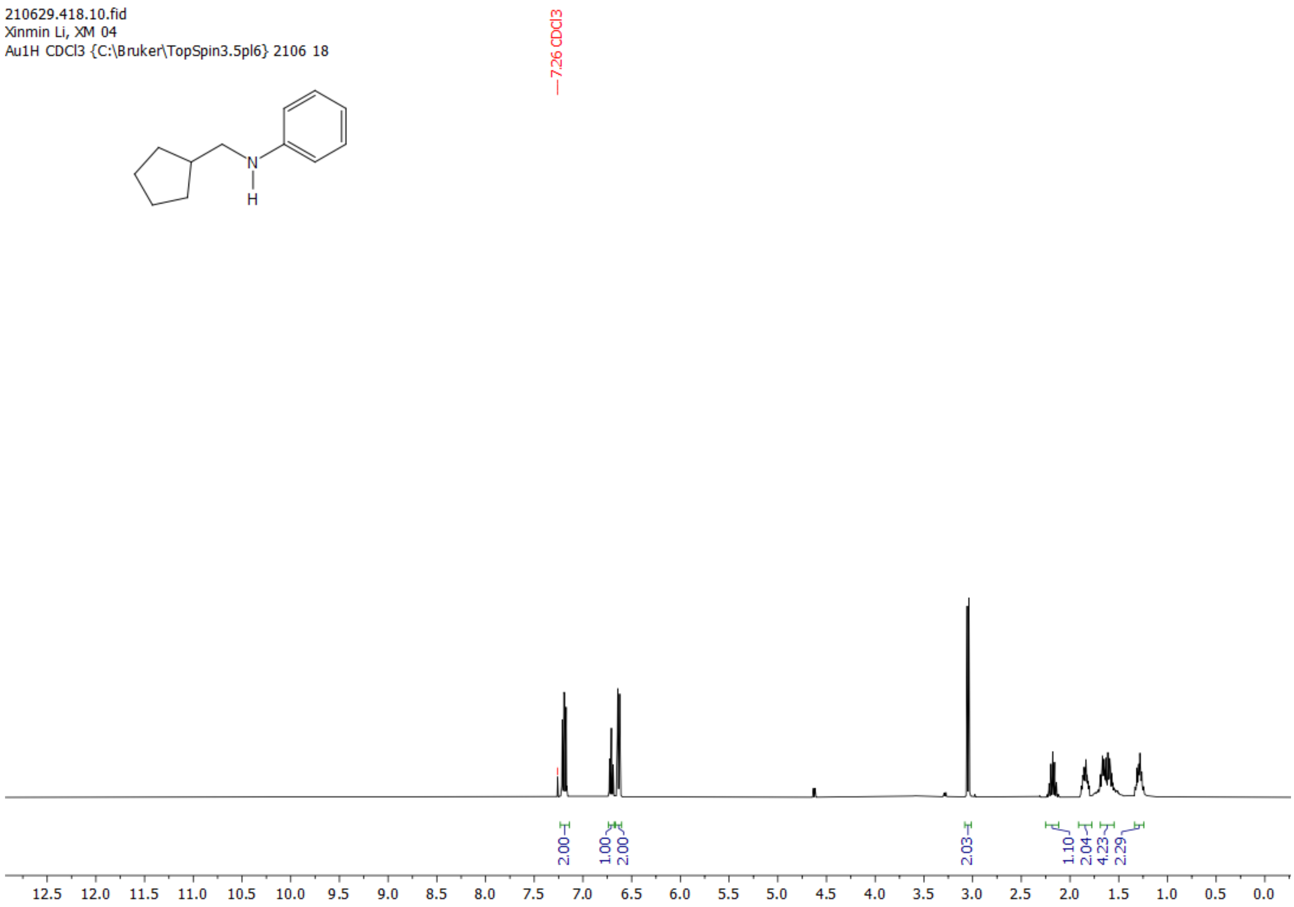
77.16 CDCl3
76.84 CDCl3
41.59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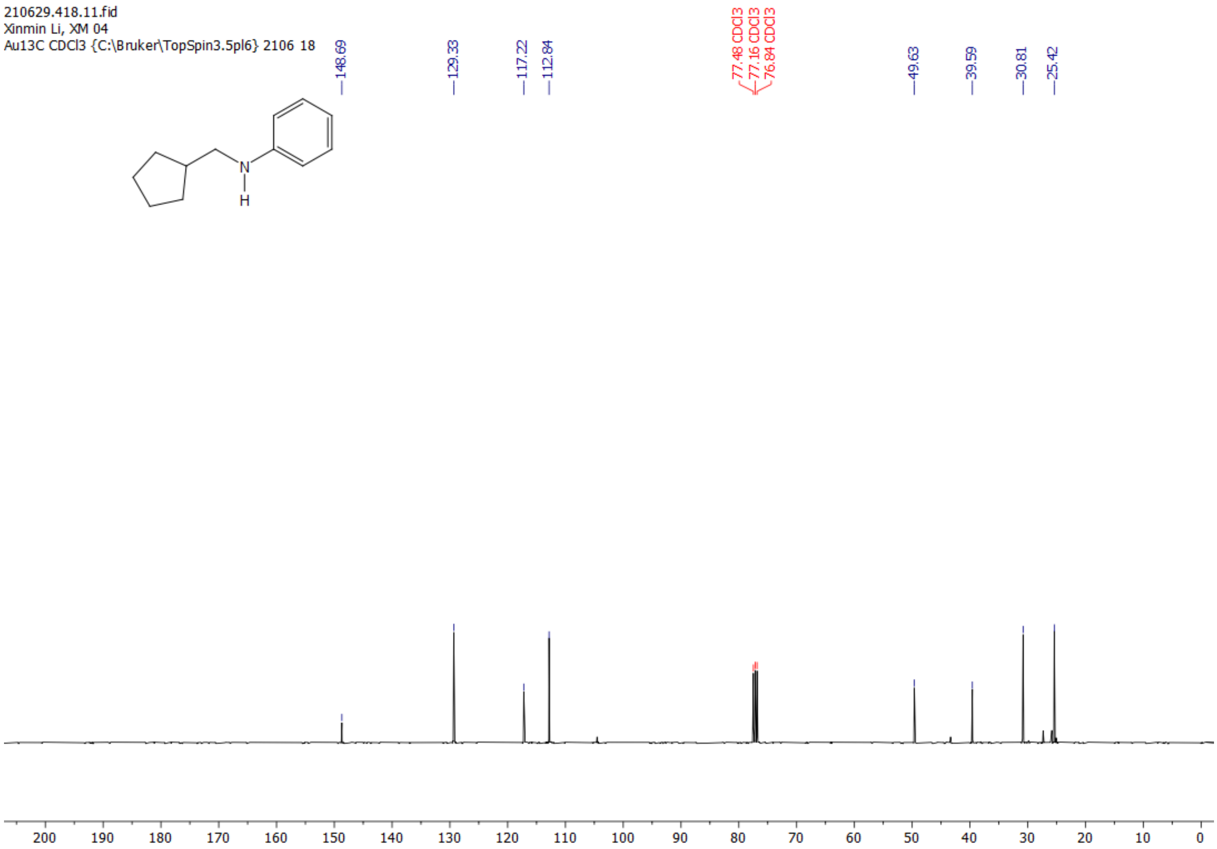
Xinmin Li, XM 04
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 18
7.26 CDCl3
N
H
2.00
1.00
2.00
2.03
1.10
2.04
4.23
2.29
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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Xinmin Li, XM 04
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 18
148.69
129.33
117.22
112.84
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
49.63
39.59
30.81
25.42
N
H
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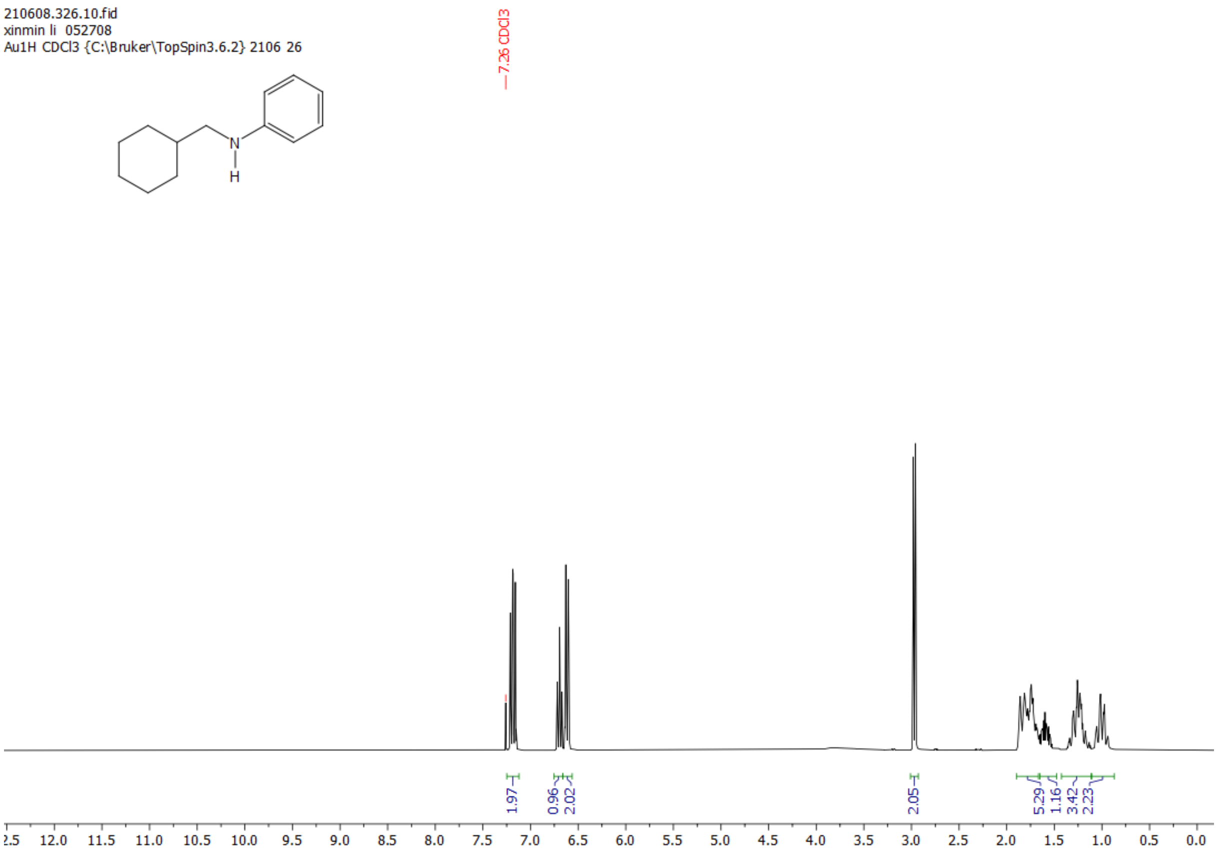
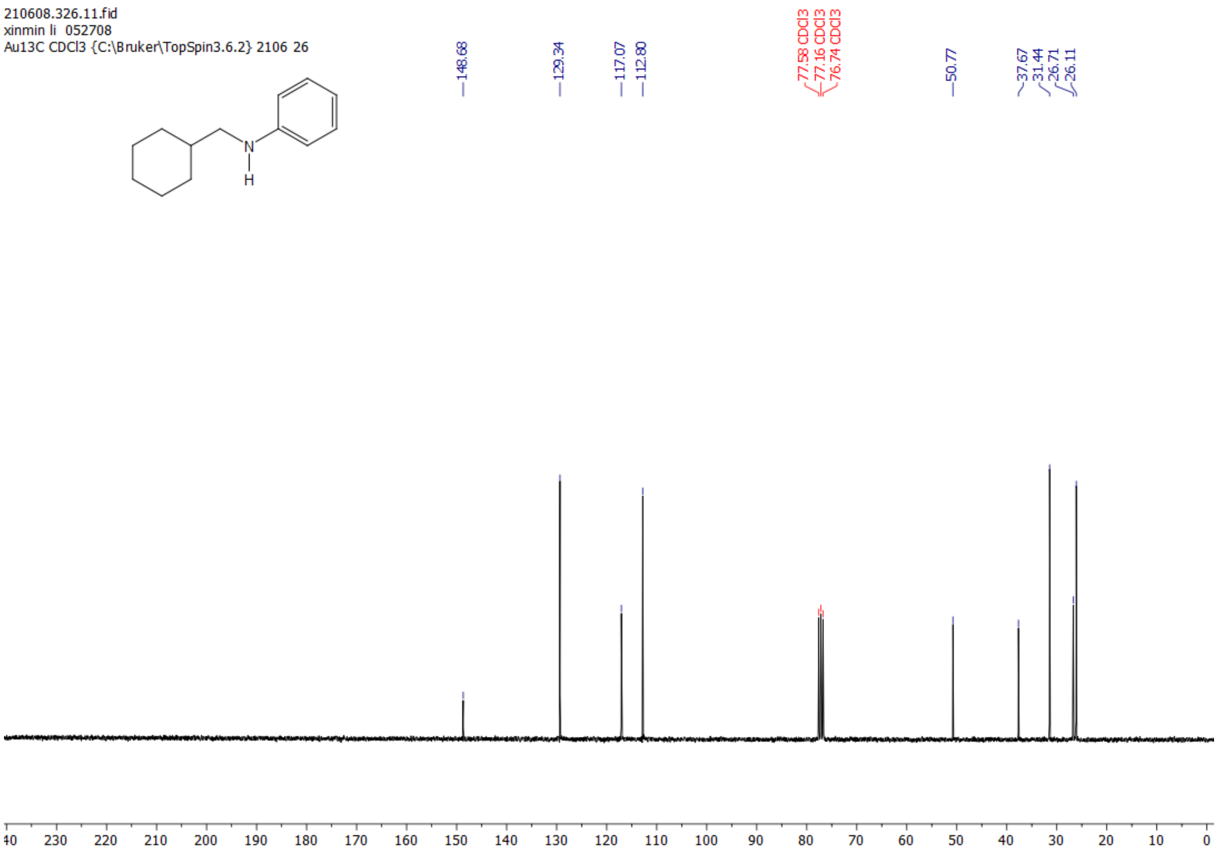
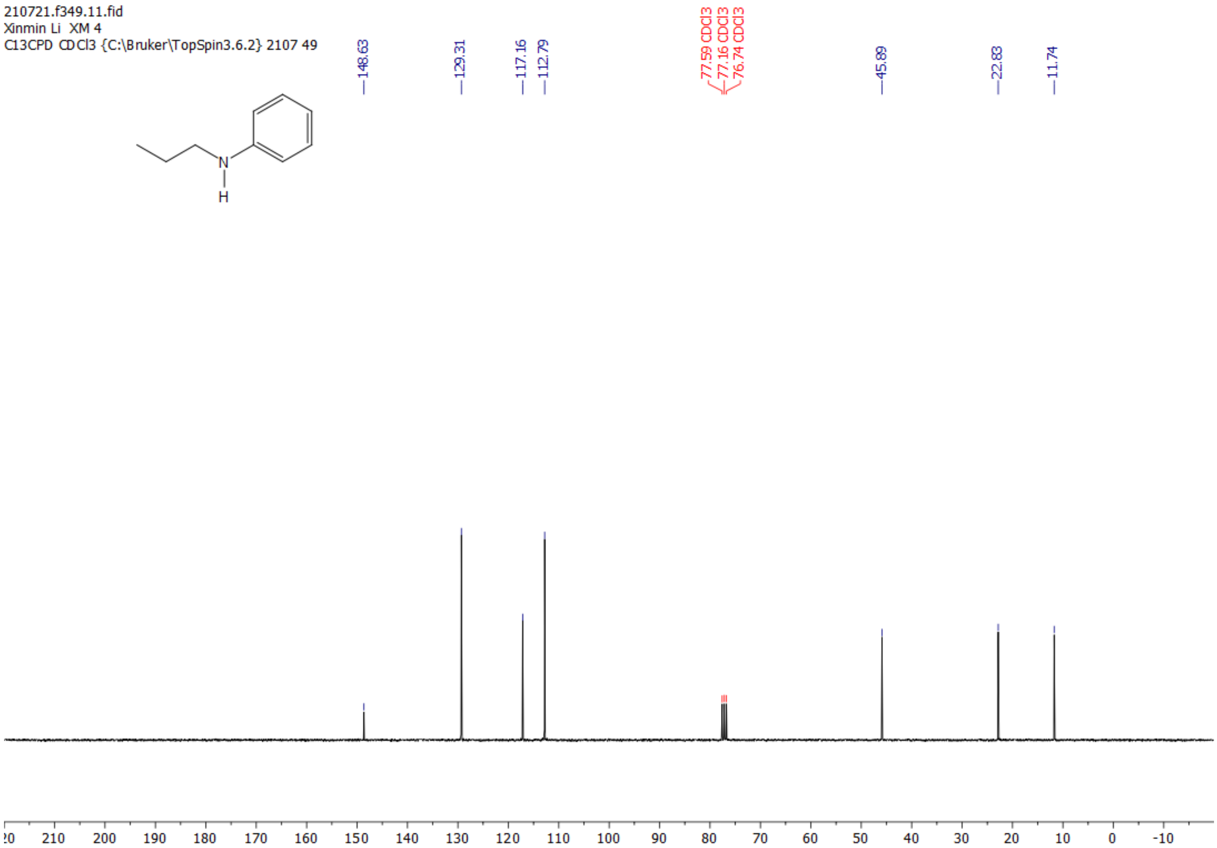
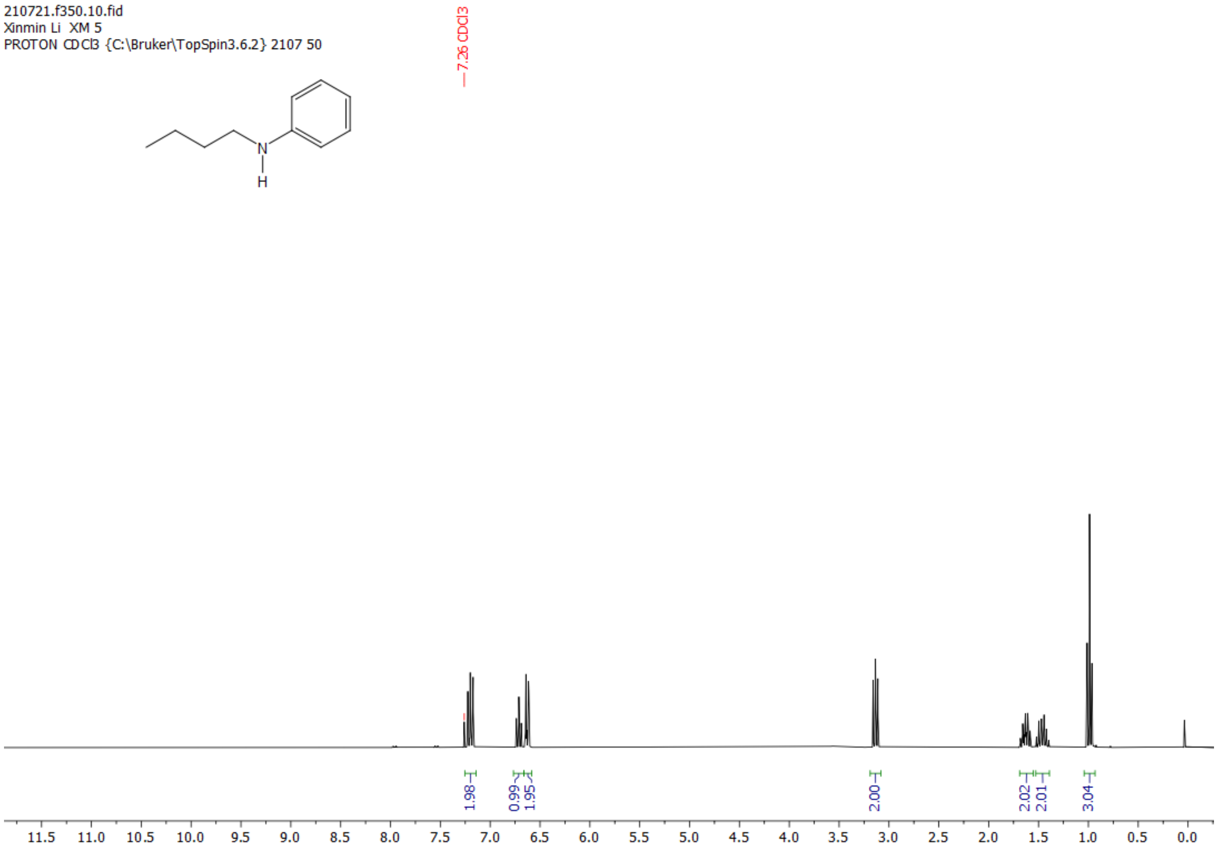

210608.326.10.fid
xinmin li 052708
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 26
7.26 CDCl3
N
H
1.97
0.96
2.02
2.05
5.29
1.16
3.42
2.23


210608.326.11.fid
xinmin li 052708
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 26
148.68
129.34
117.07
112.80
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
50.77
37.67
31.44
26.71
26.11
N
H

210721.f349.10.fid
Xinmin Li XM 4
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 49

210721.f349.11.fid
Xinmin Li XM 4
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 49

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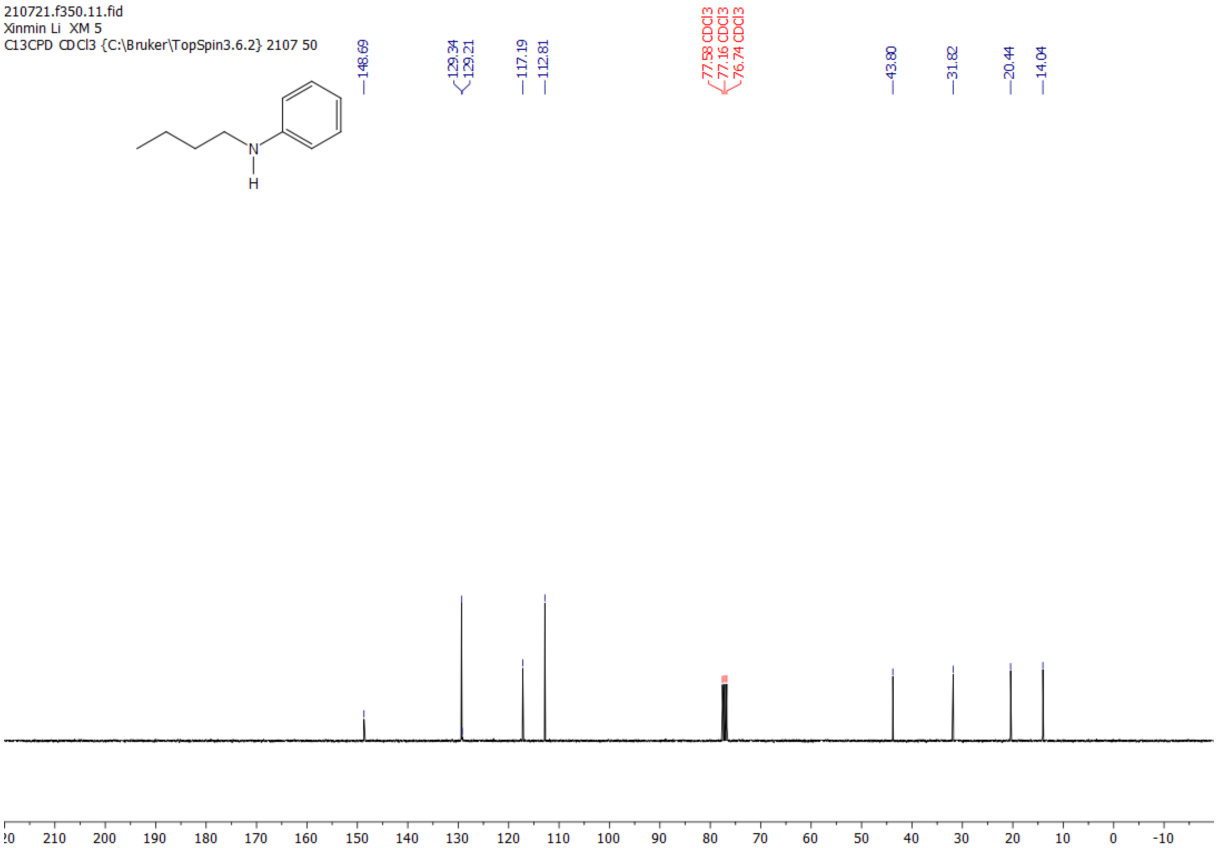
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 50
7.26 CDCl3
N
H
1.98
0.99
1.95
2.00
2.02
2.01
3.04
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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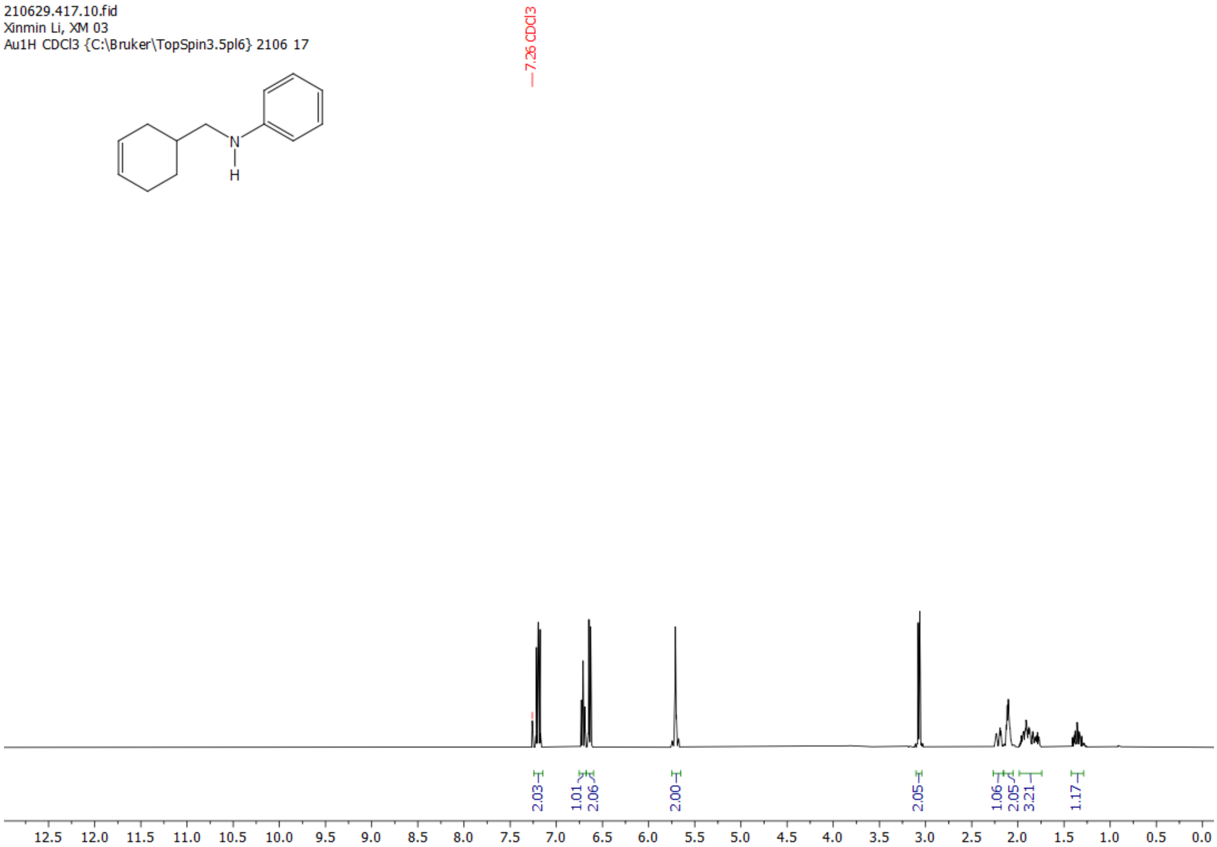
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 50
148.69
129.34
129.21
117.19
112.81
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
43.80
31.82
20.44
14.04
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

210629.417.10.fid
Xinmin Li, XM 03
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 17



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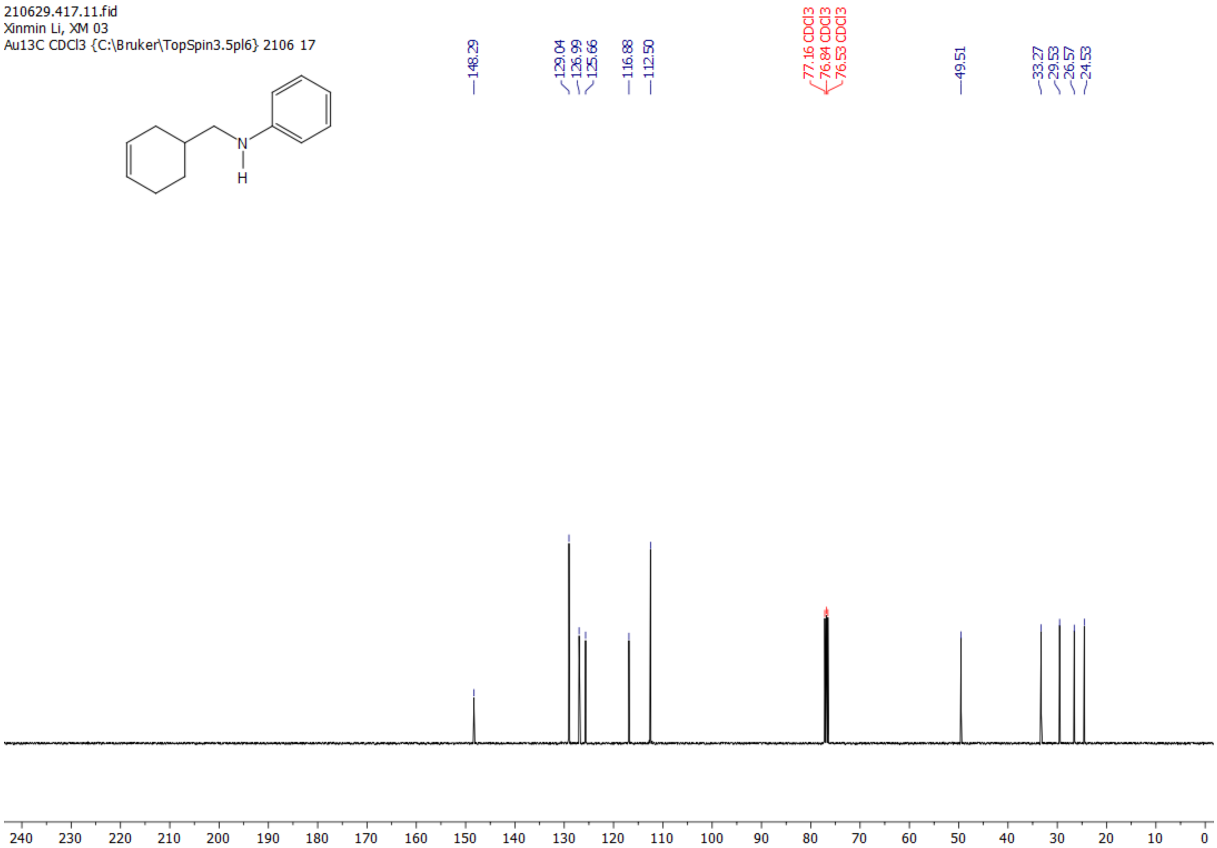
Xinmin Li, XM 03
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2106 17



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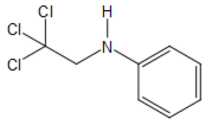
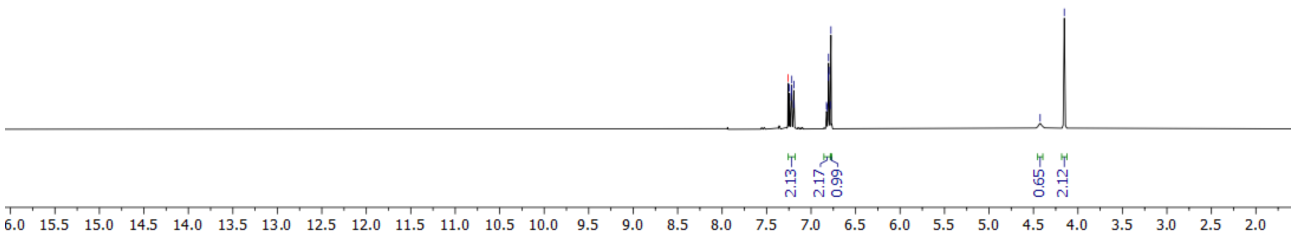
Xinmin Li, XM 435
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 36





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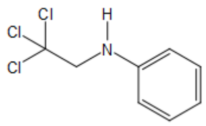
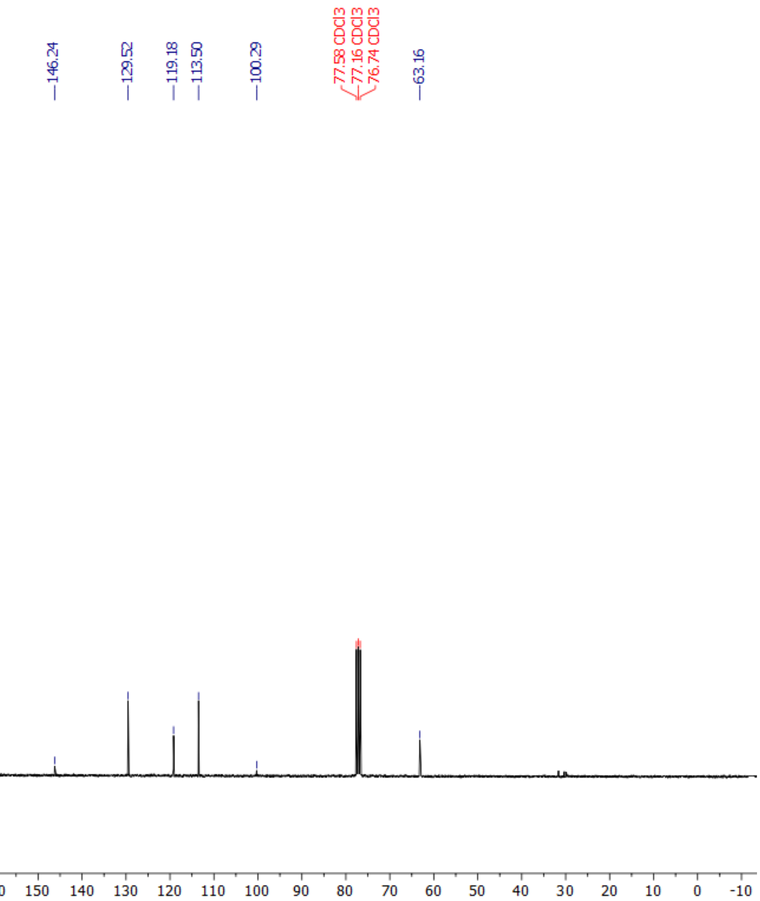
Xinmin Li, 030402
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 52





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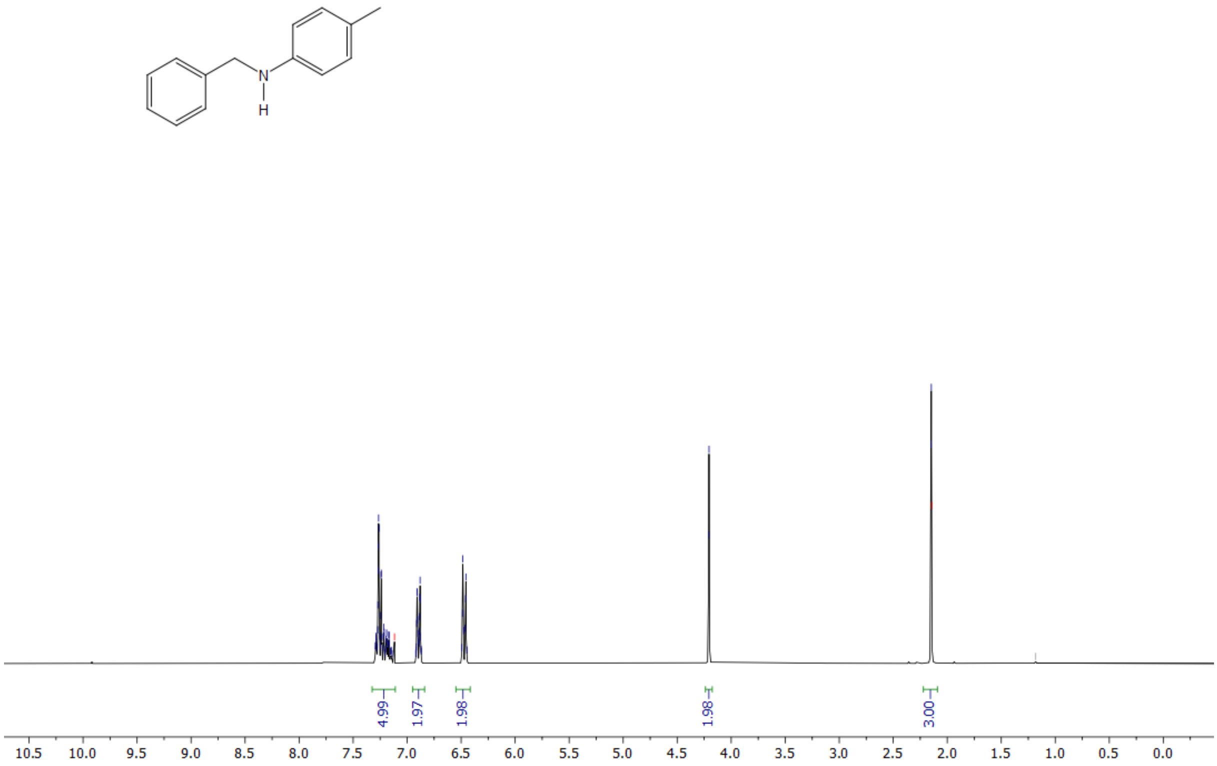
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 15



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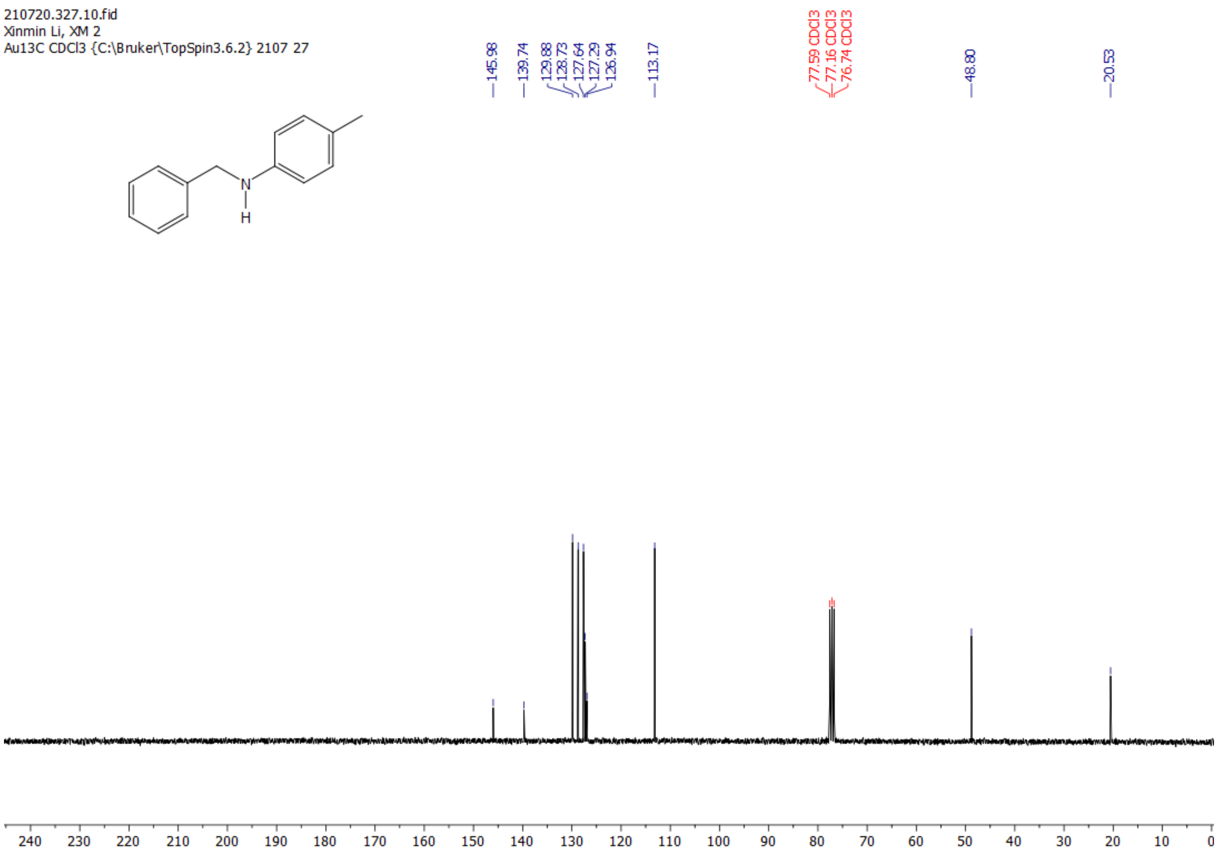
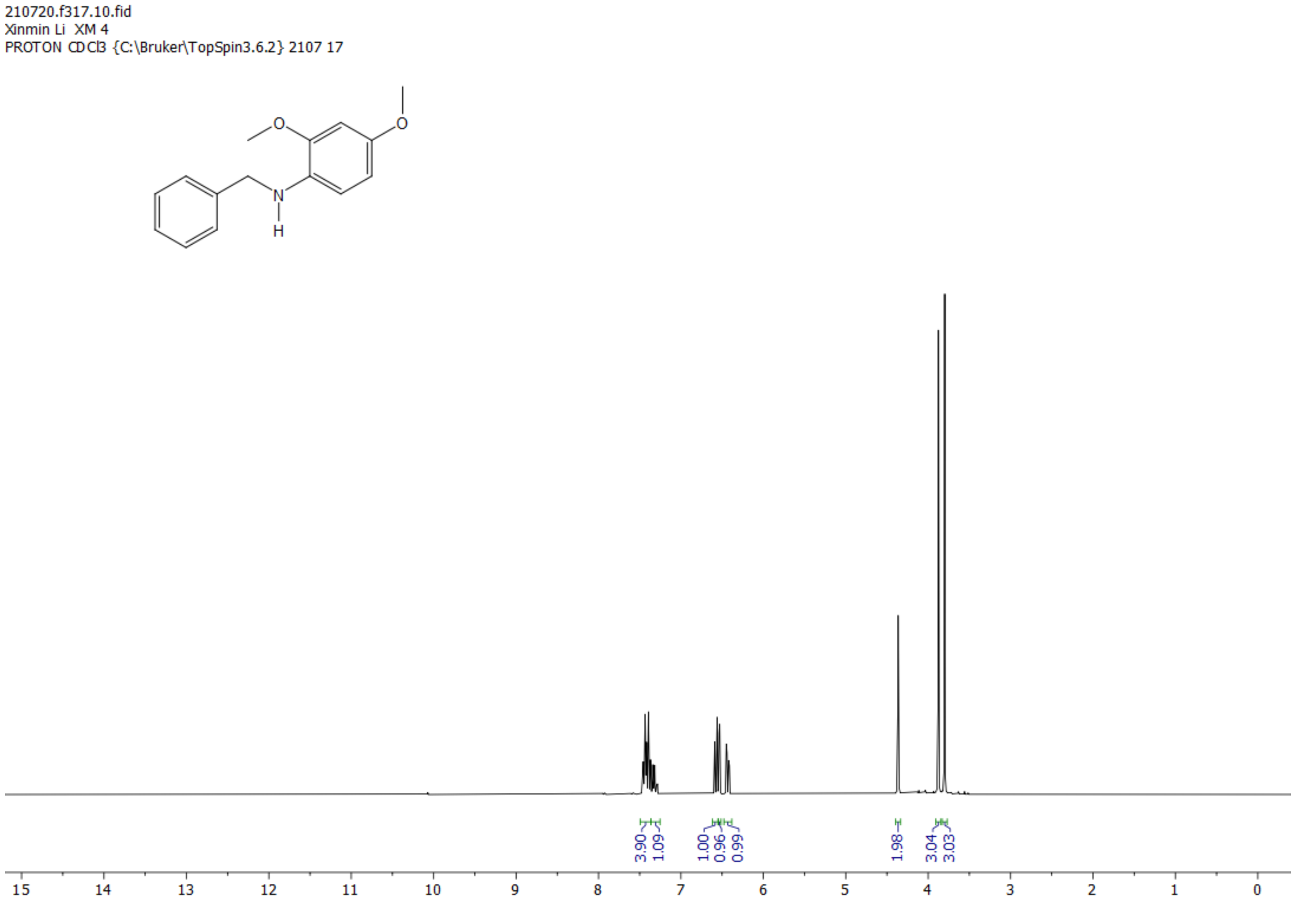
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 27

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0.96
0.99
1.98
3.04
3.03
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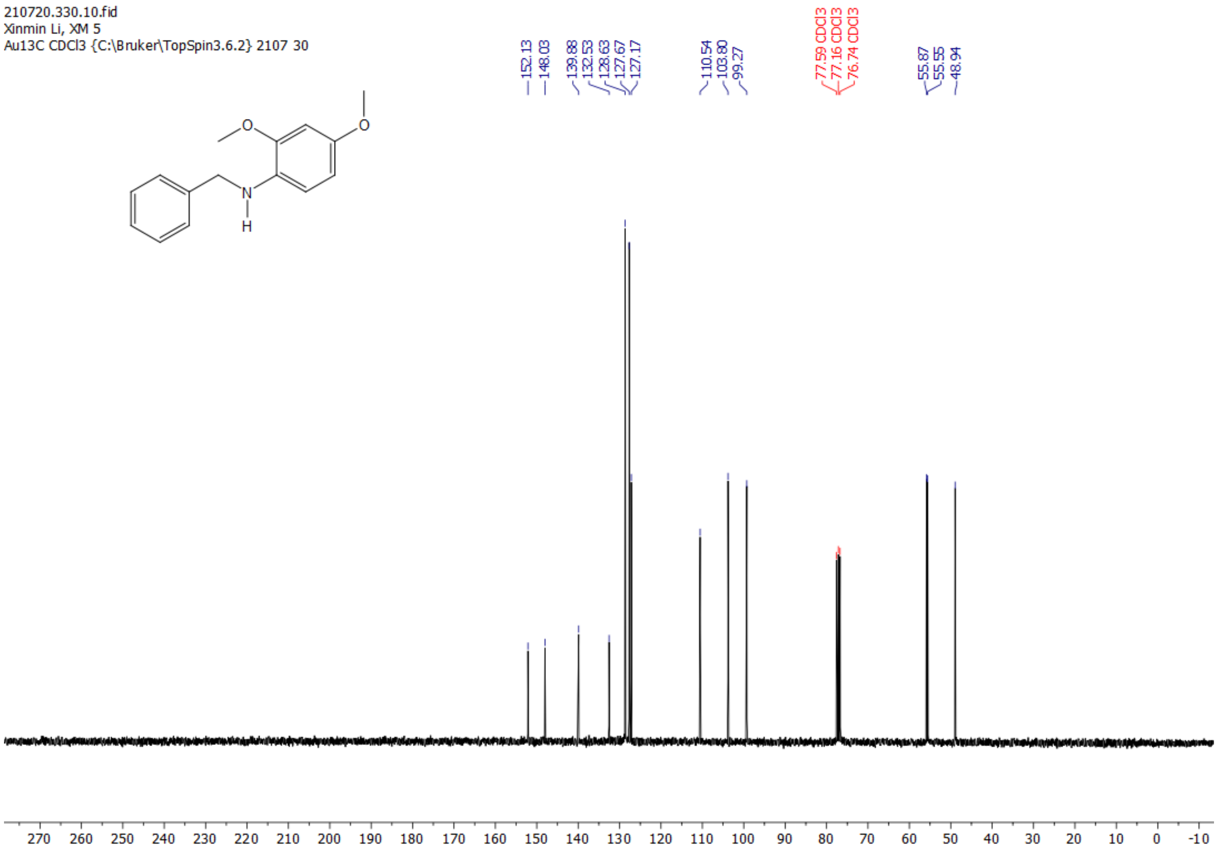
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152.13
148.03
139.88
132.53
128.63
127.67
127.17
110.54
103.80
99.27
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
55.87
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48.94
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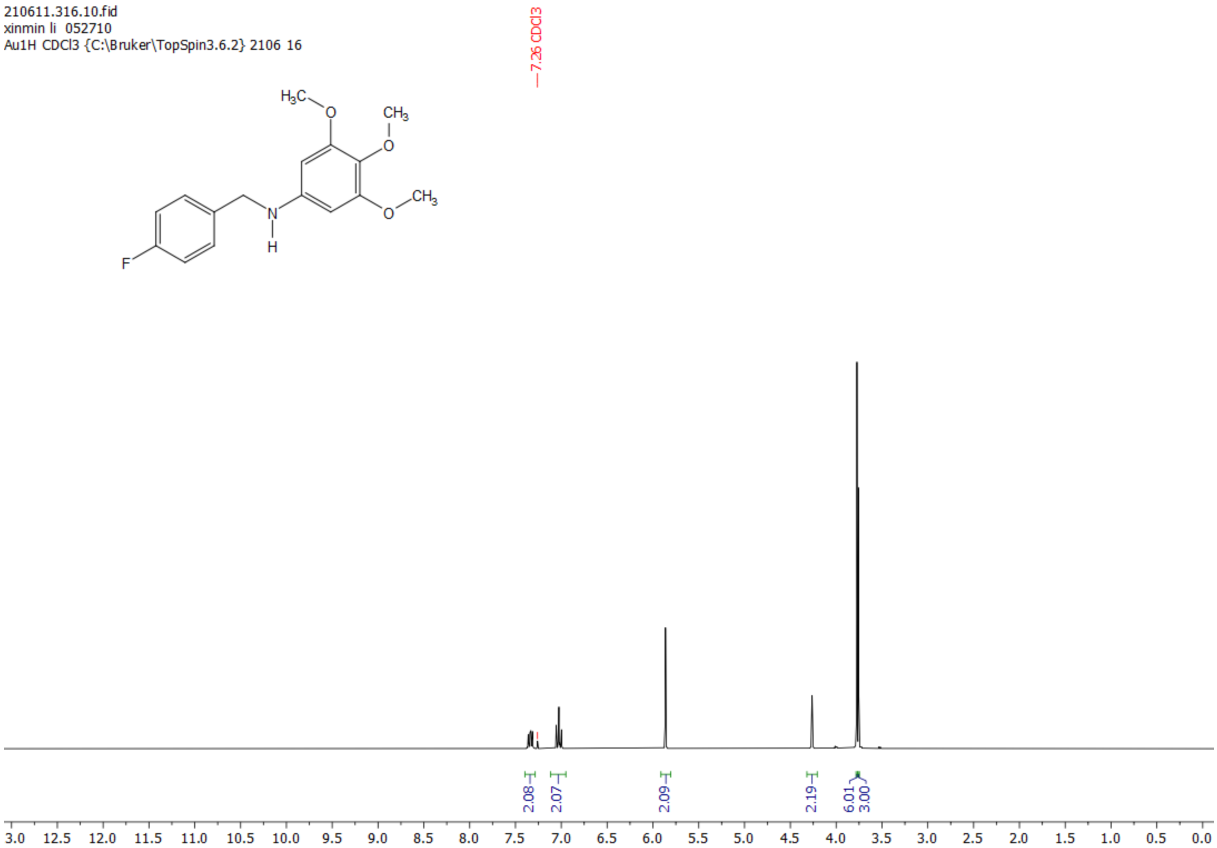
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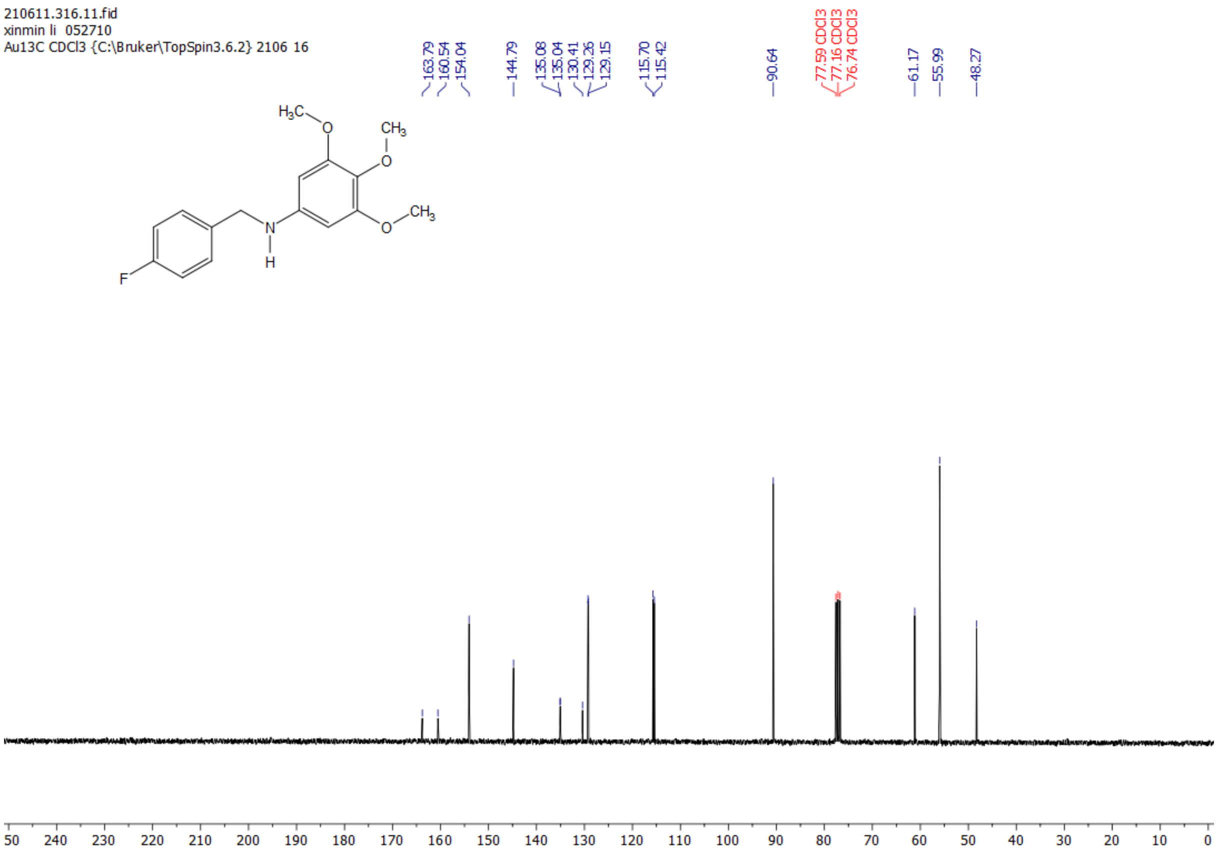
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 16
7.26 CDCl3
2.08
2.07
2.09
2.19
6.01
3.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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xinmin li 052710
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163.79
160.54
154.04
144.79
135.08
135.04
130.41
129.26
129.15
115.70
115.42
90.64
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
61.17
55.99
48.27
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

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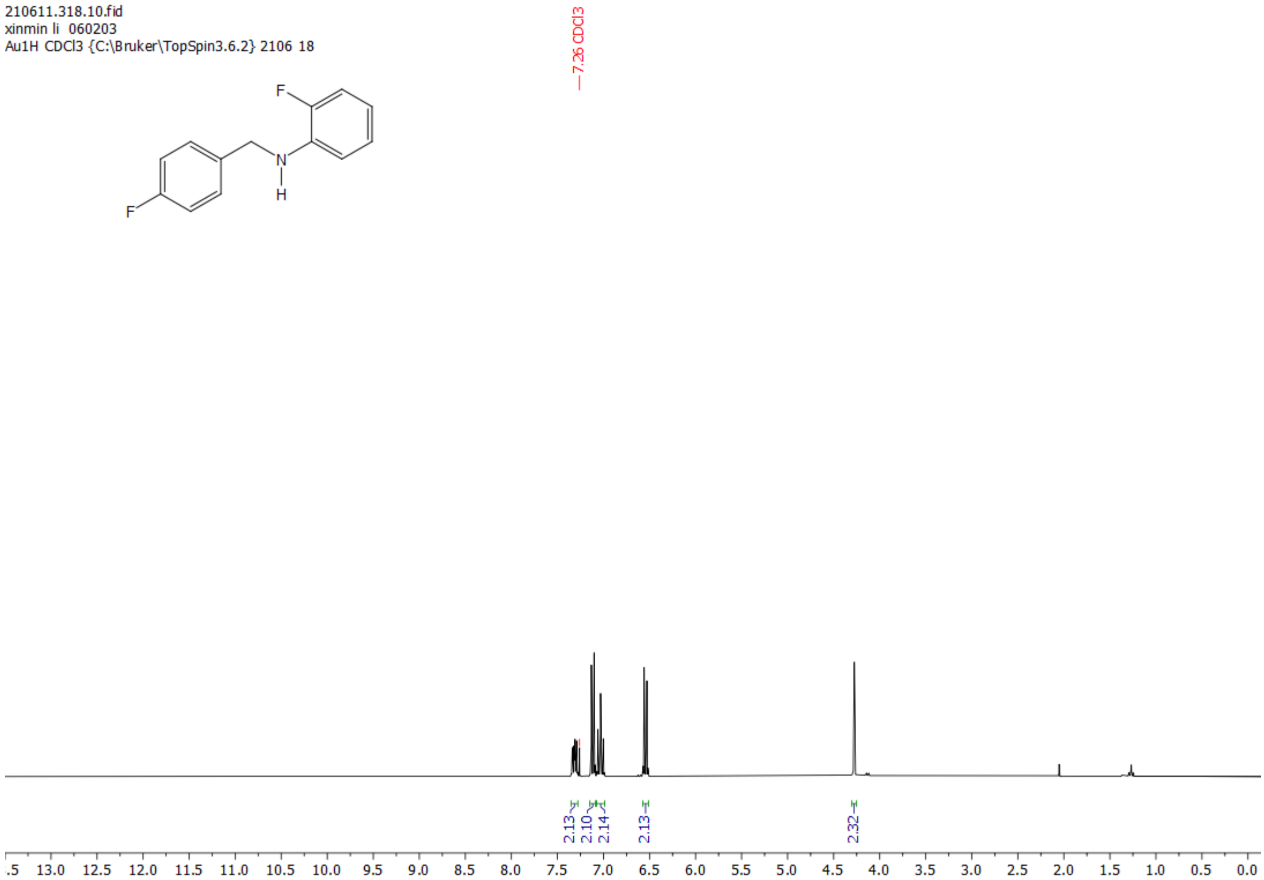
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 18



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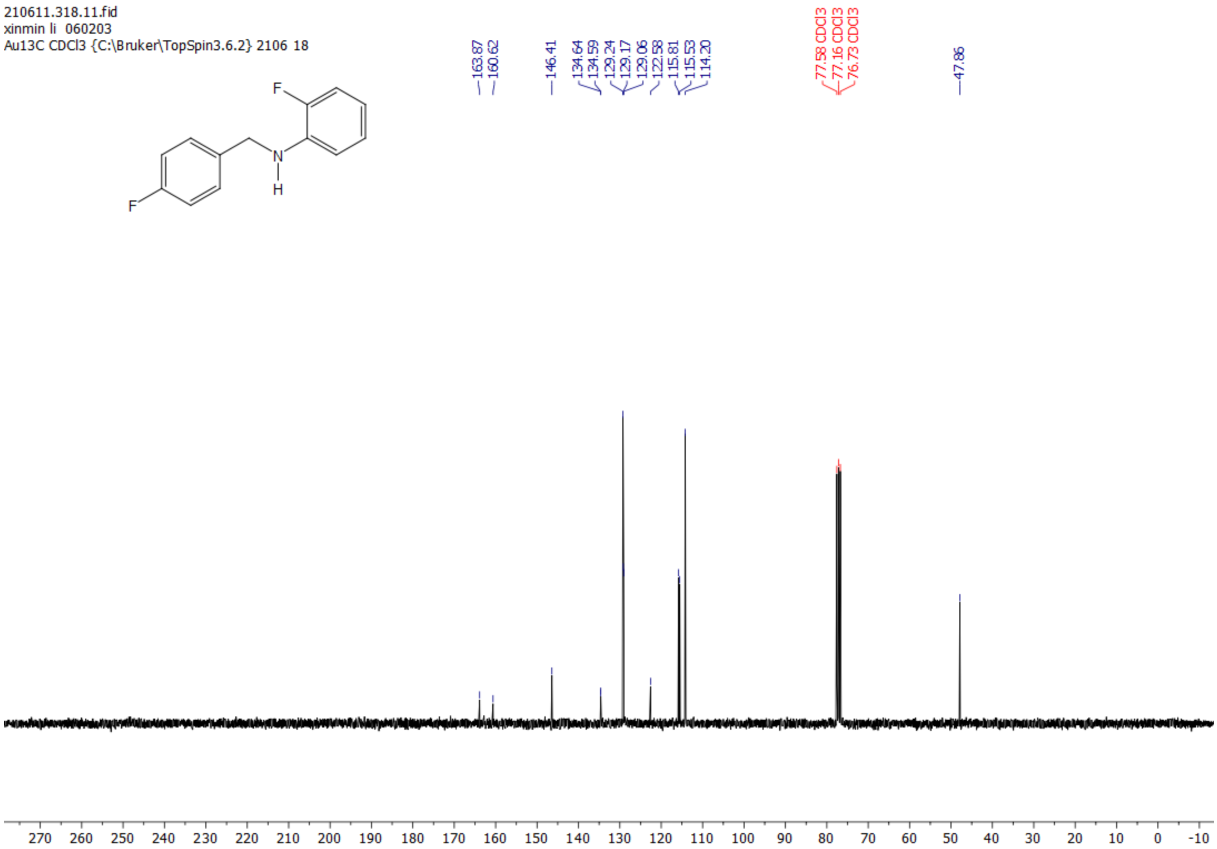
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 18



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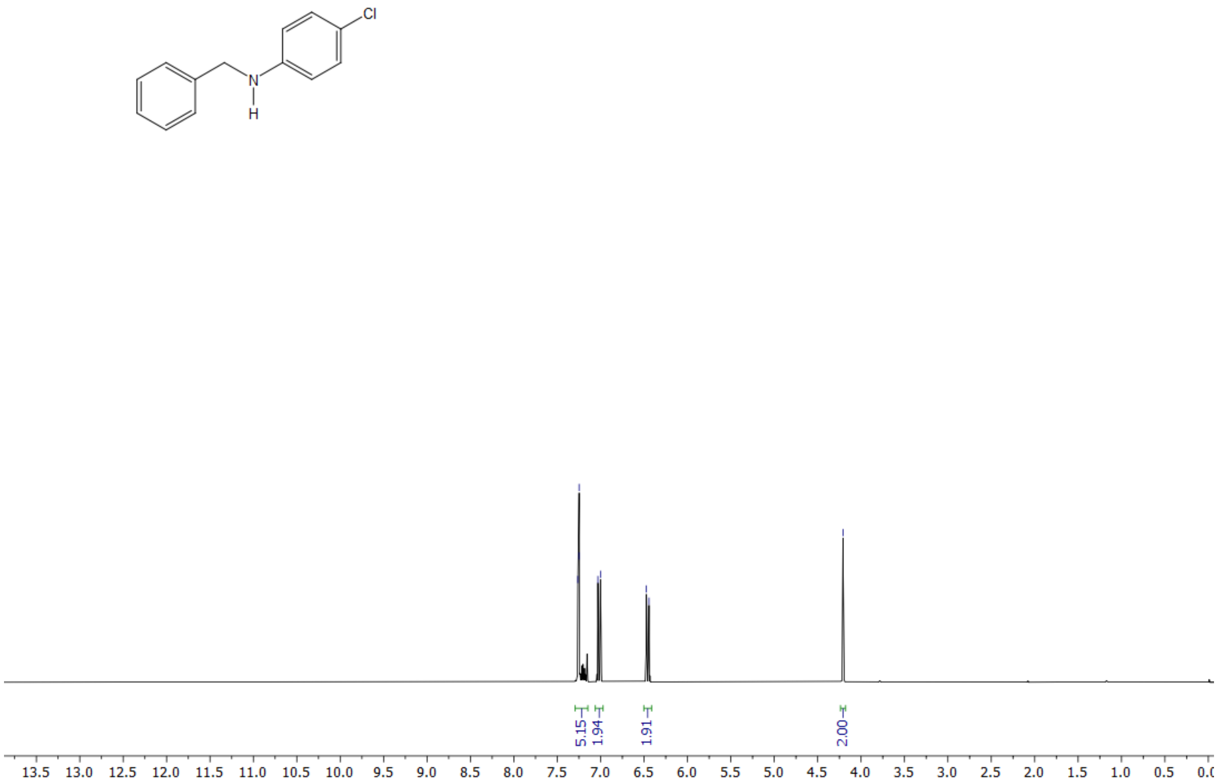

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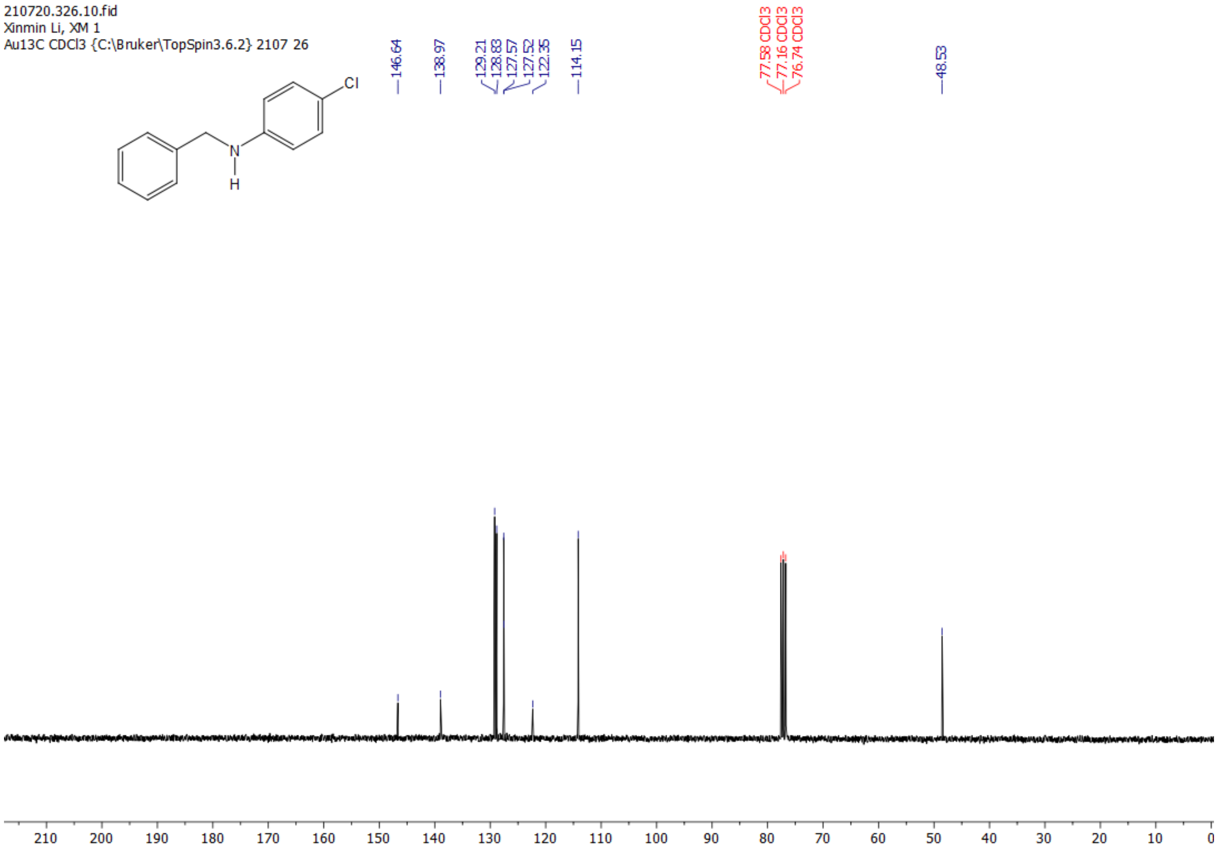

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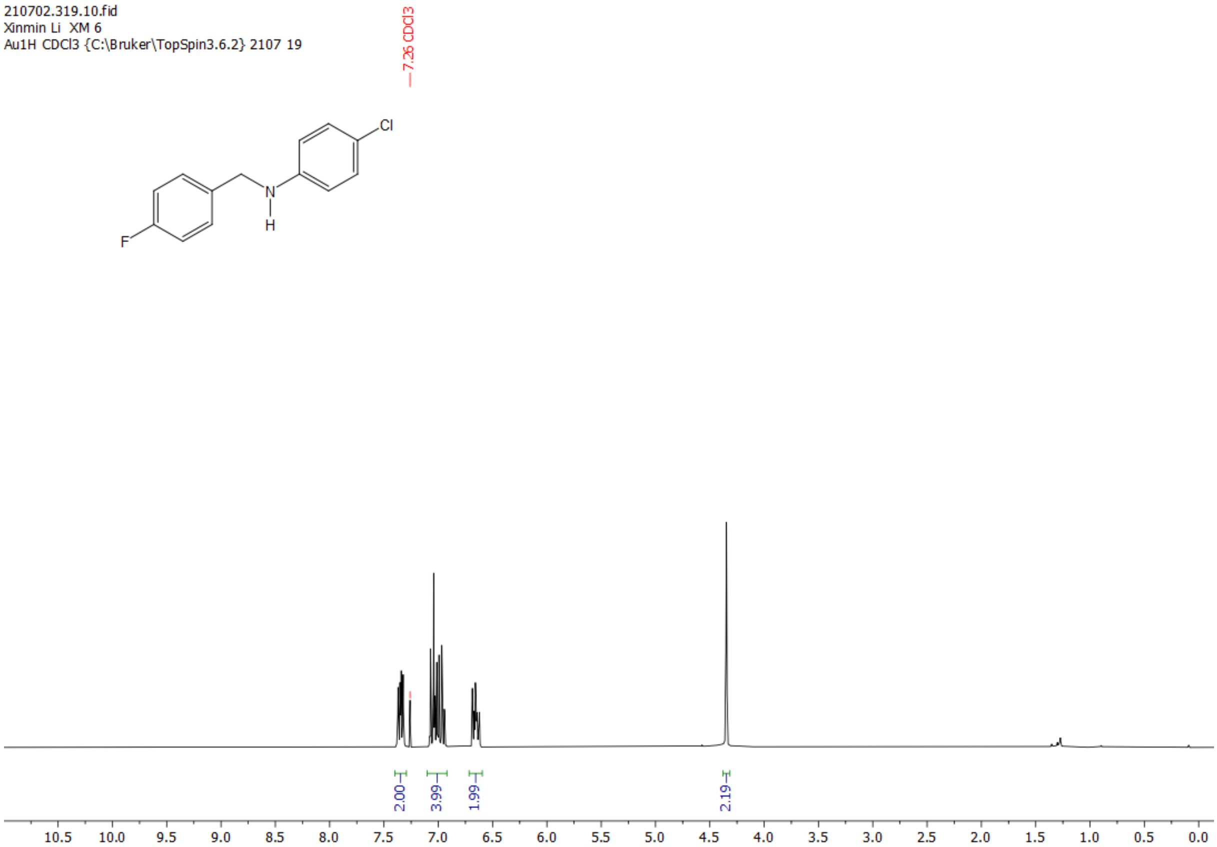

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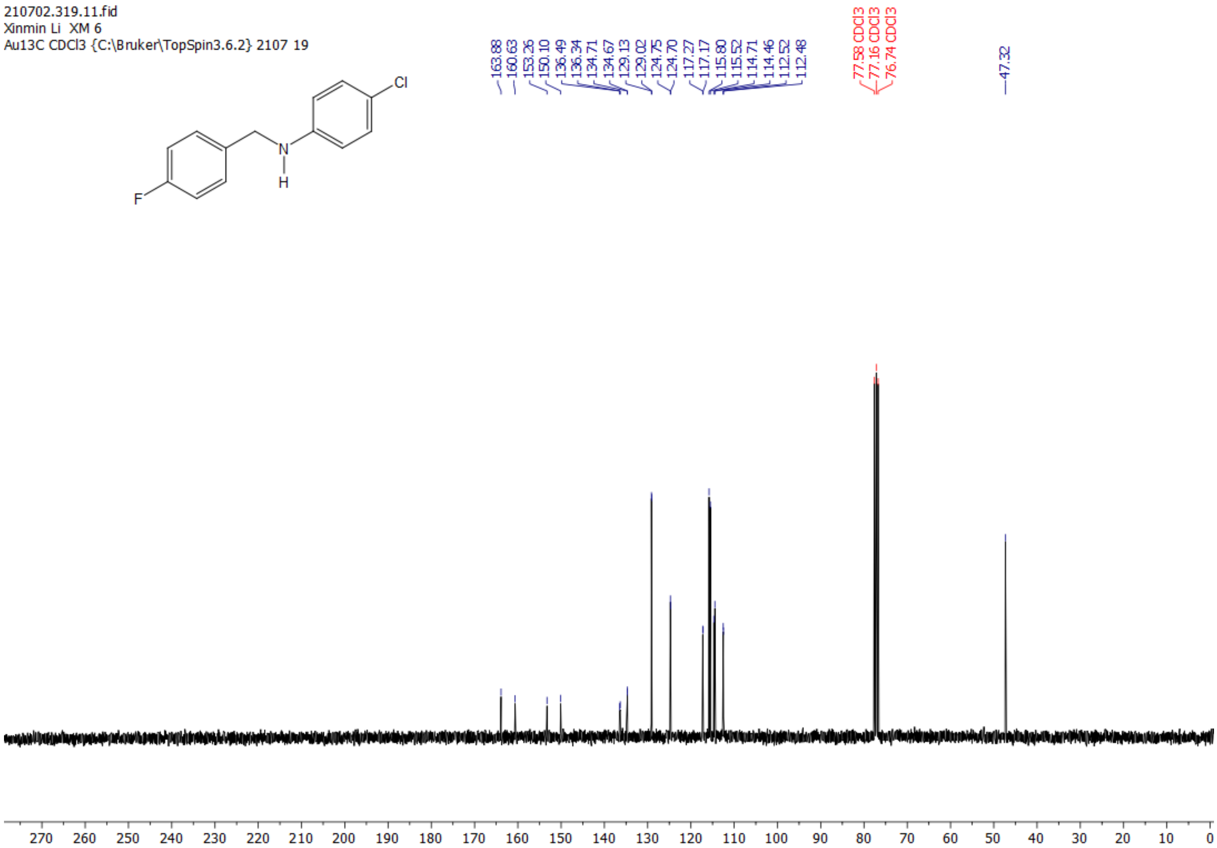
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 19



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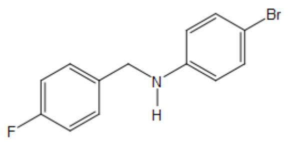

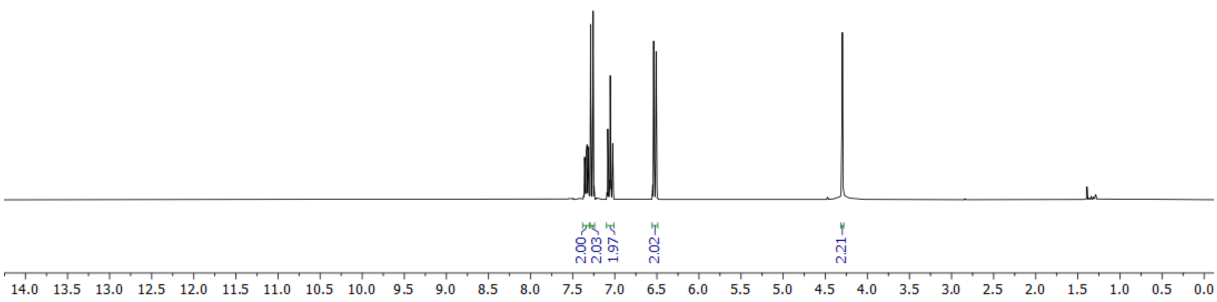

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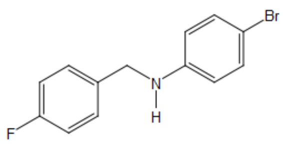
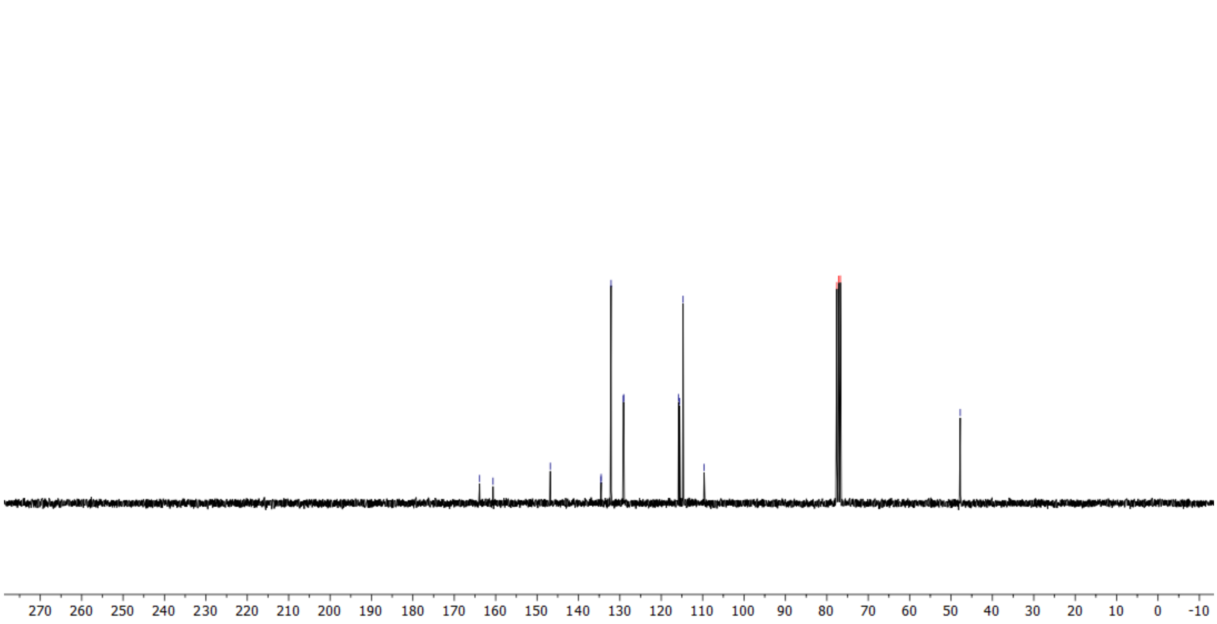
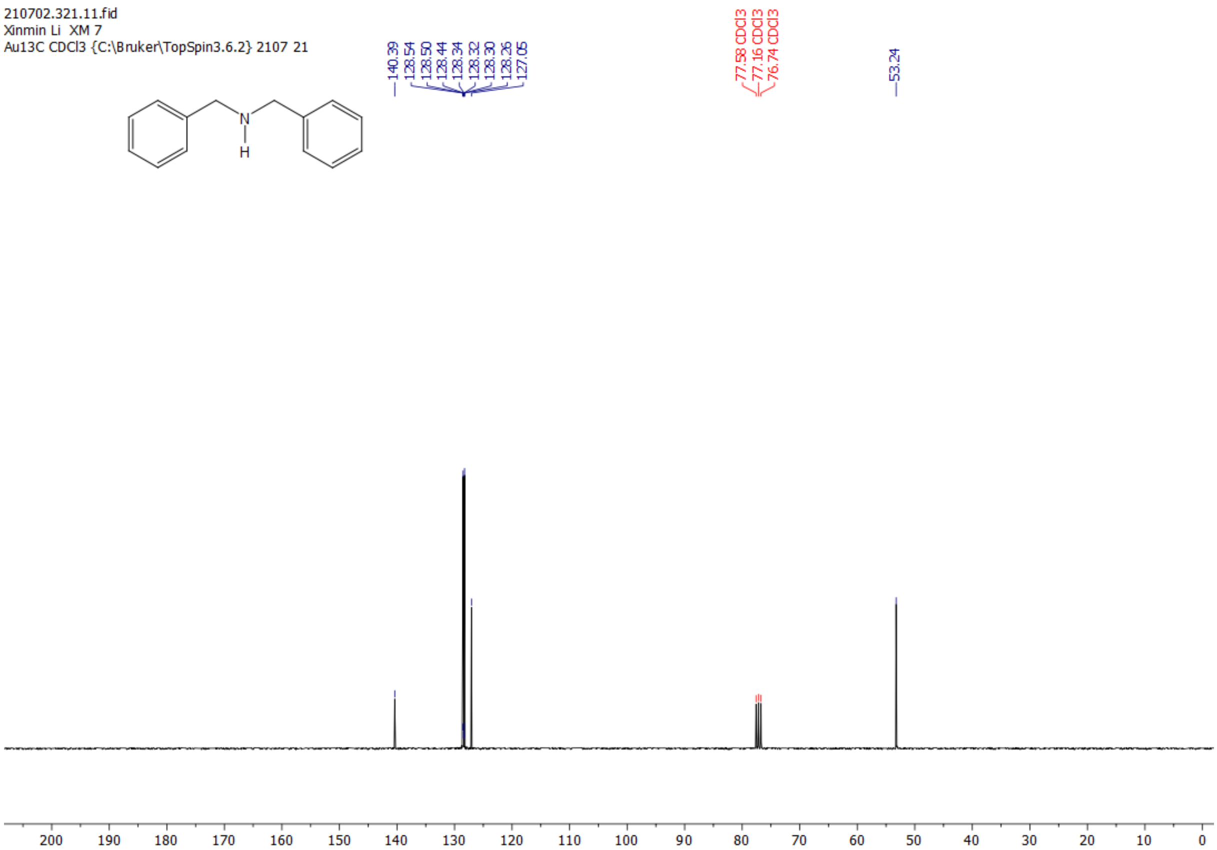
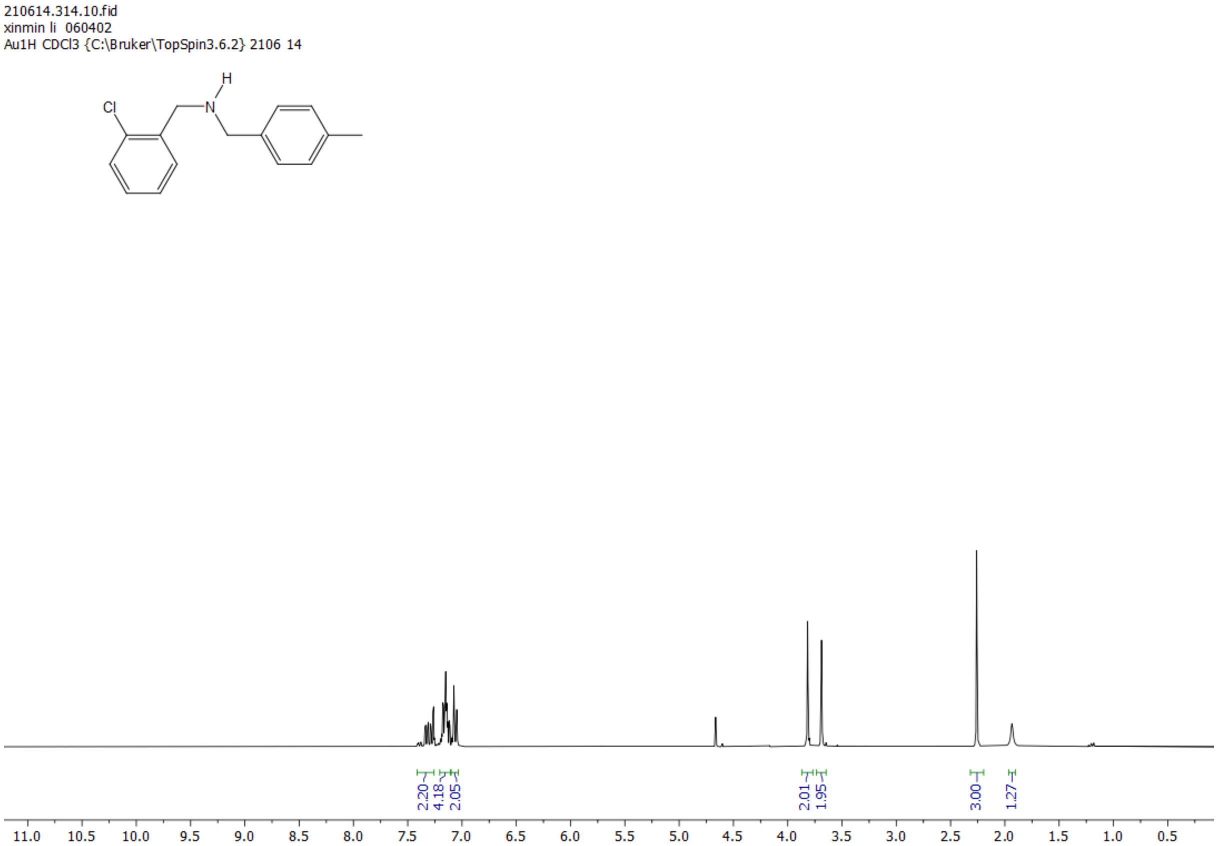




210702.321.10.fid
Xinmin Li XM 7
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 21

210702.321.11.fid
Xinmin Li XM 7
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 21

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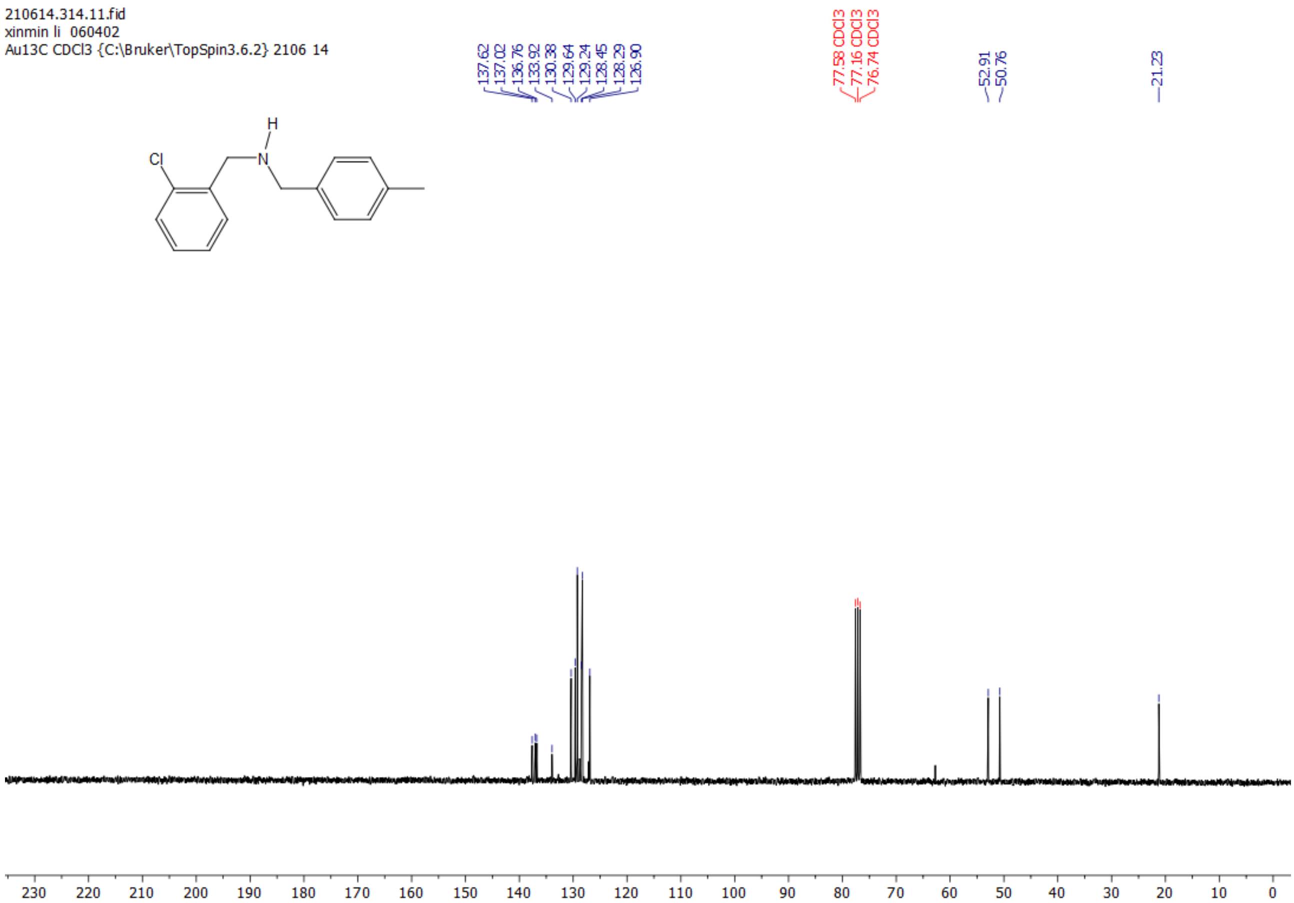
xinmin li 060402
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 14
Cl
H
N
2.20
4.18
2.05
2.01
1.95
3.00
1.27
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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xinmin li 060402
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 14
137.62
137.02
136.76
133.92
130.38
129.64
129.24
128.45
128.29
126.90
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
52.91
50.76
21.23
Cl
H
N
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

210601.318.10.fid
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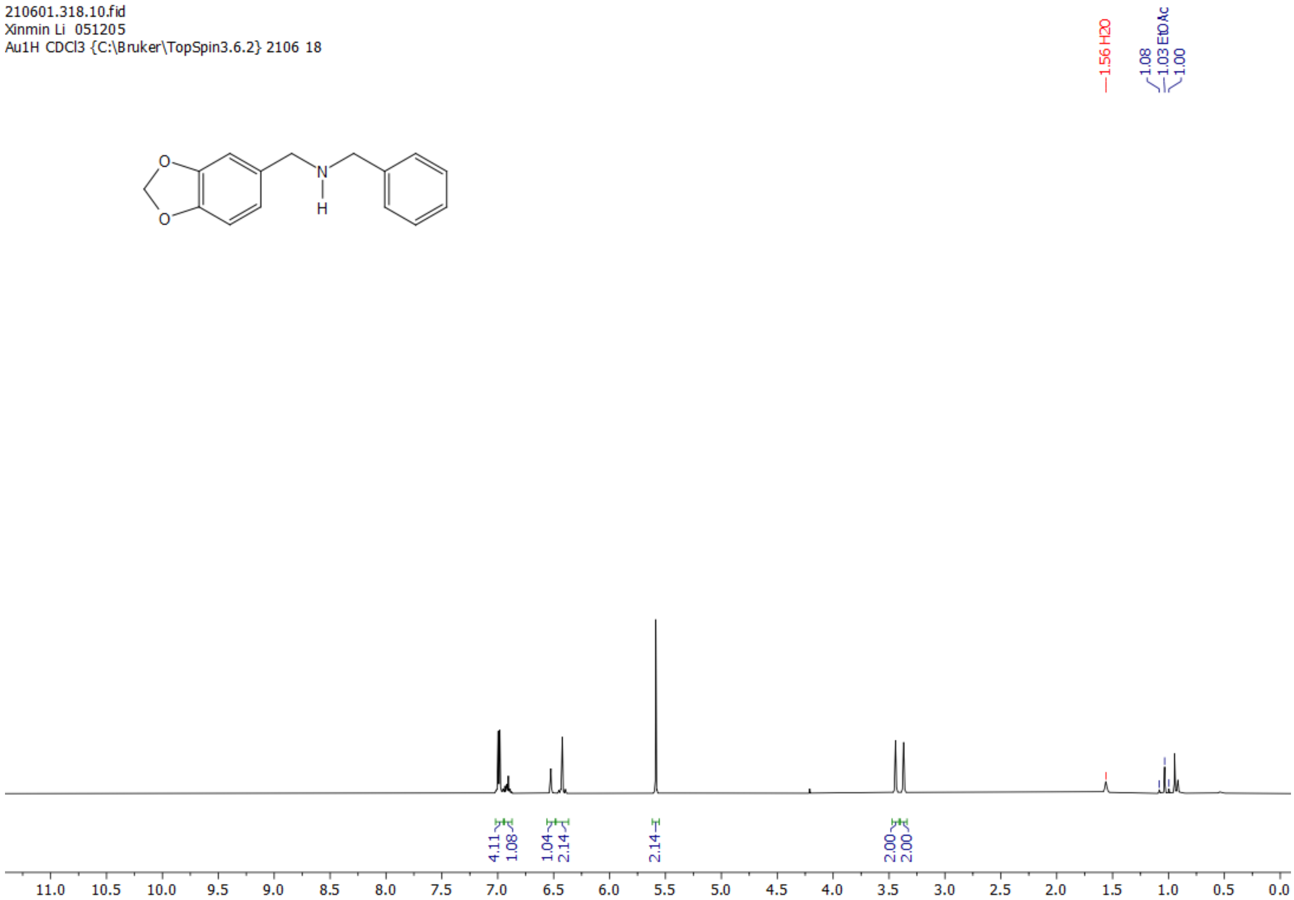
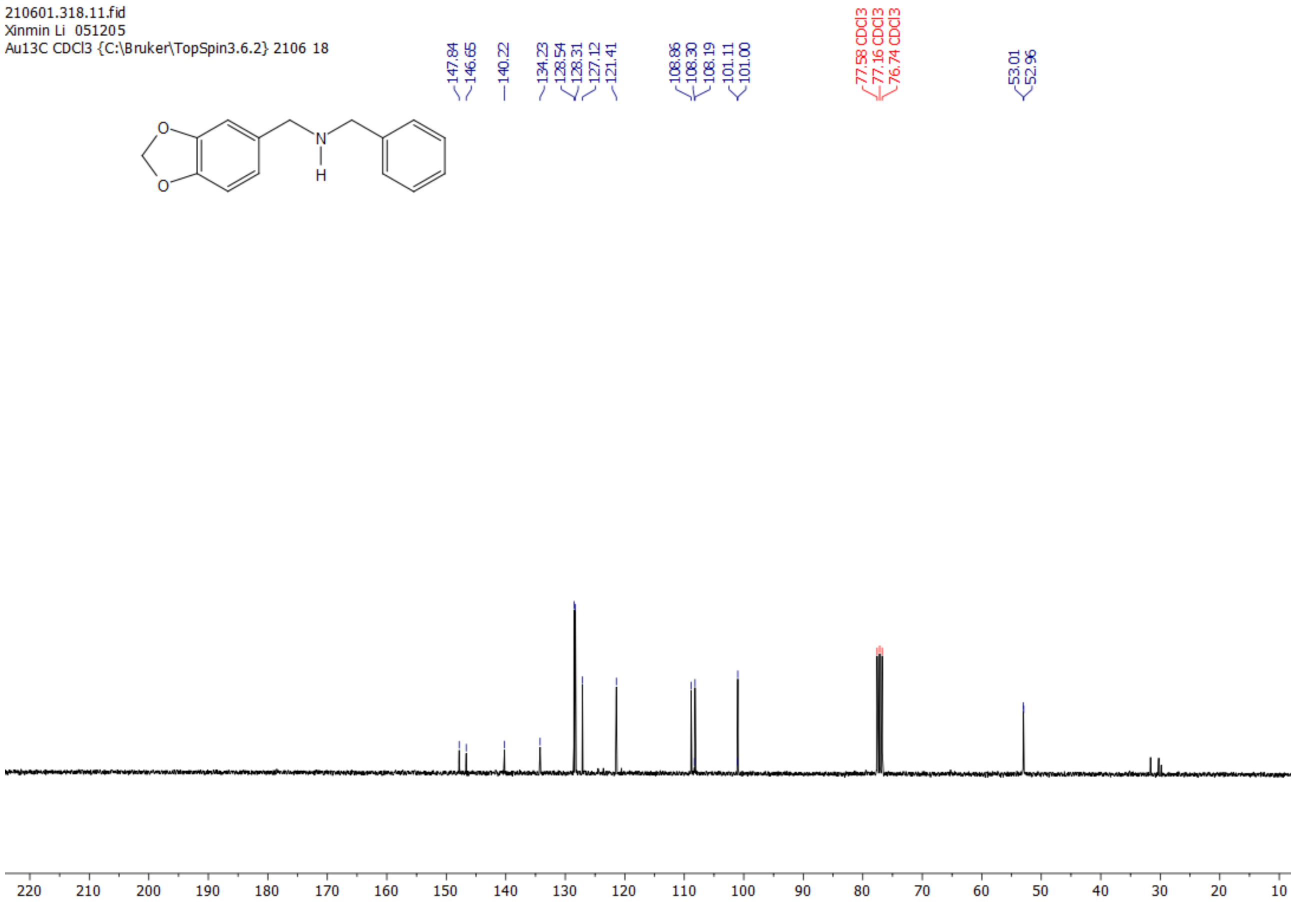
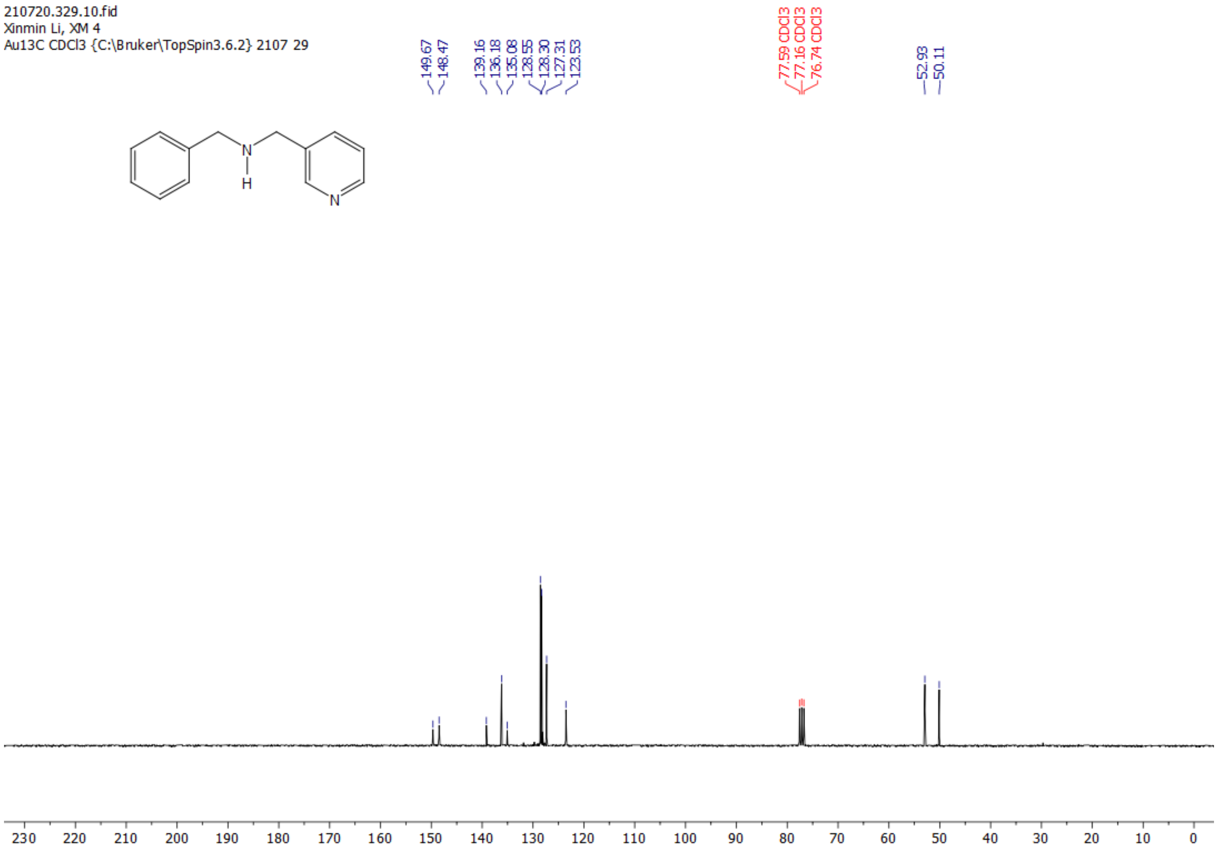


210601.318.11.fid
Xinmin Li 051205
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 18

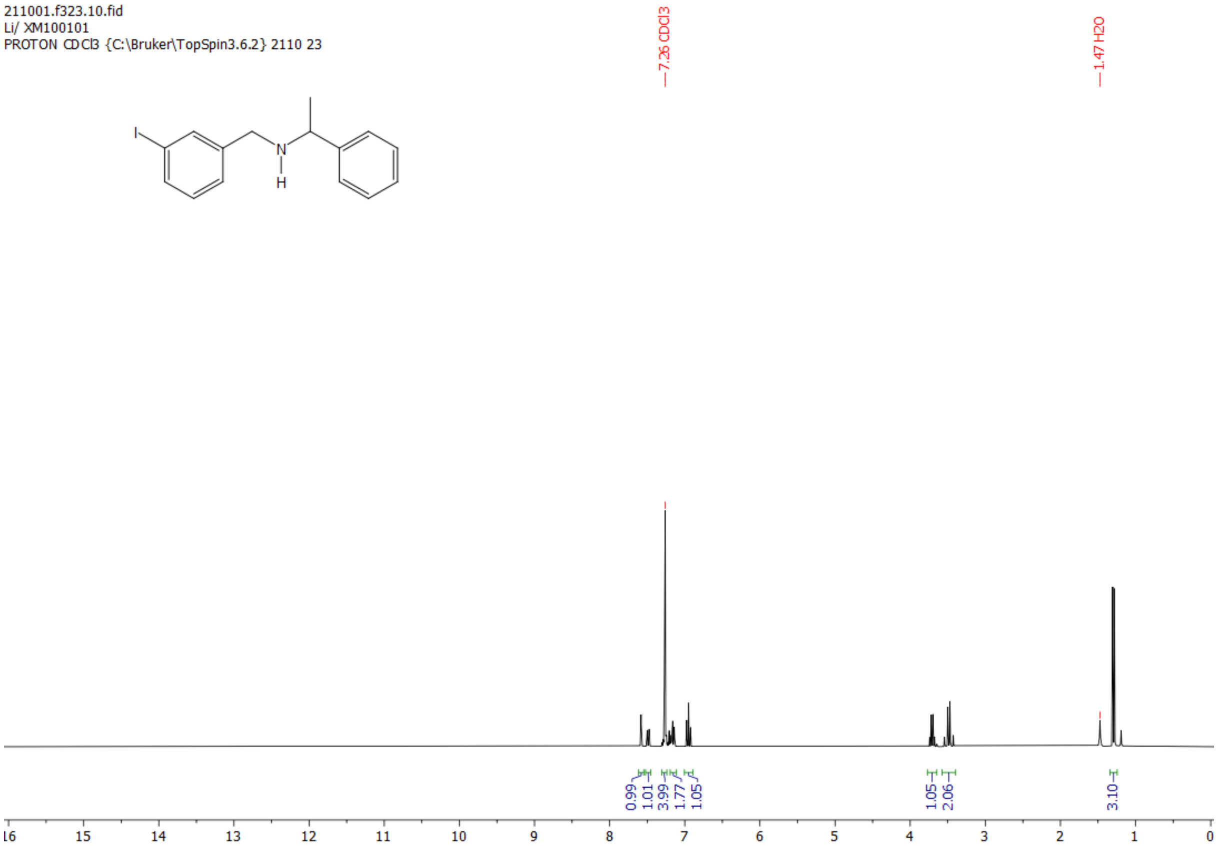


210720.f318.10.fid
Xinmin Li XM 5
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 18

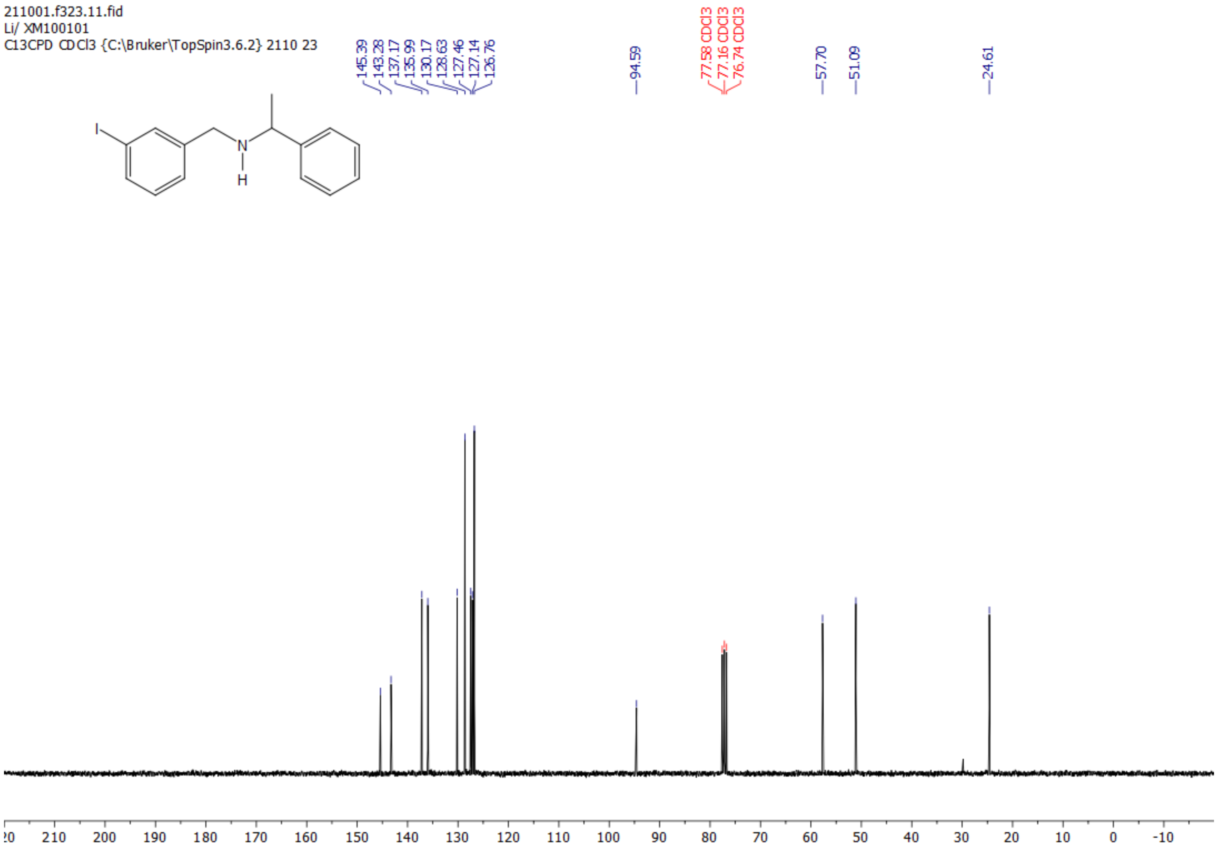
210720.329.10.fid
Xinmin Li, XM 4
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 29



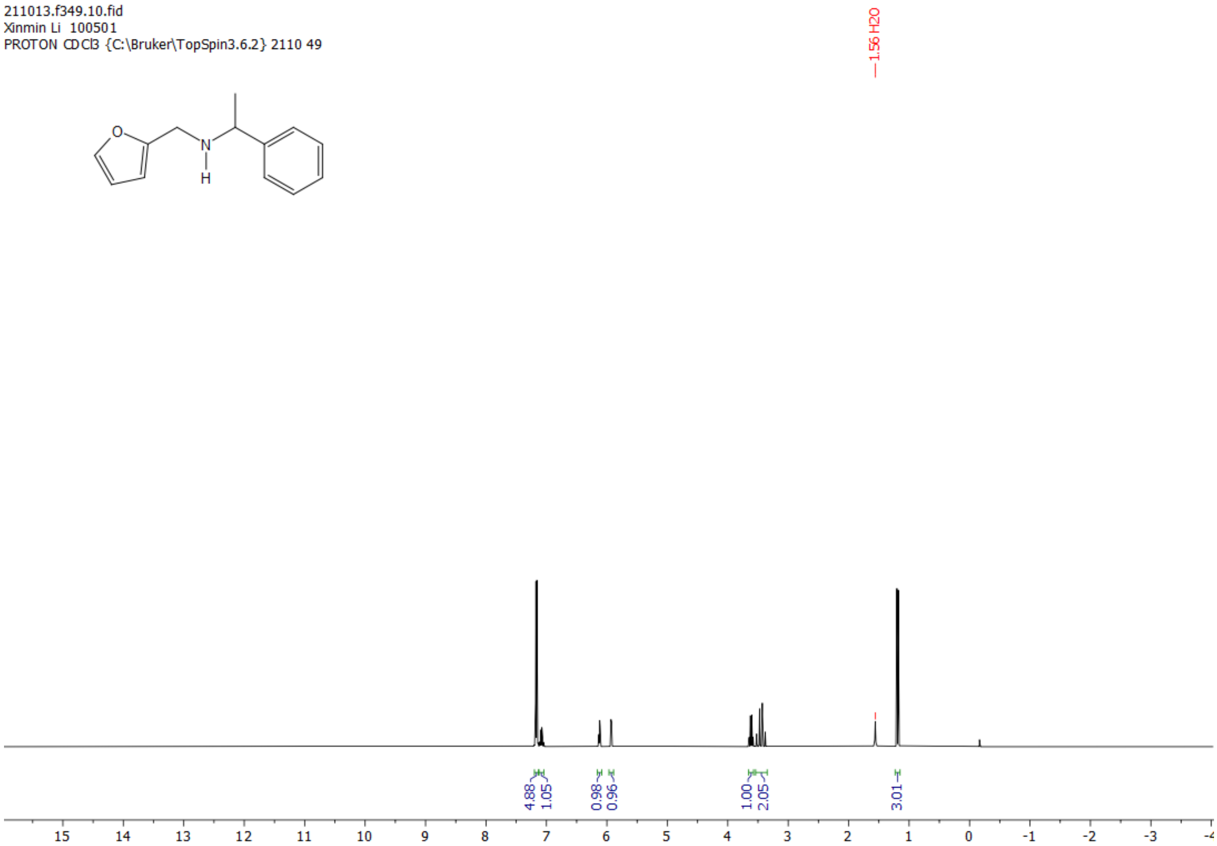
211001.f323.10.fid
Li/ XM100101
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 23



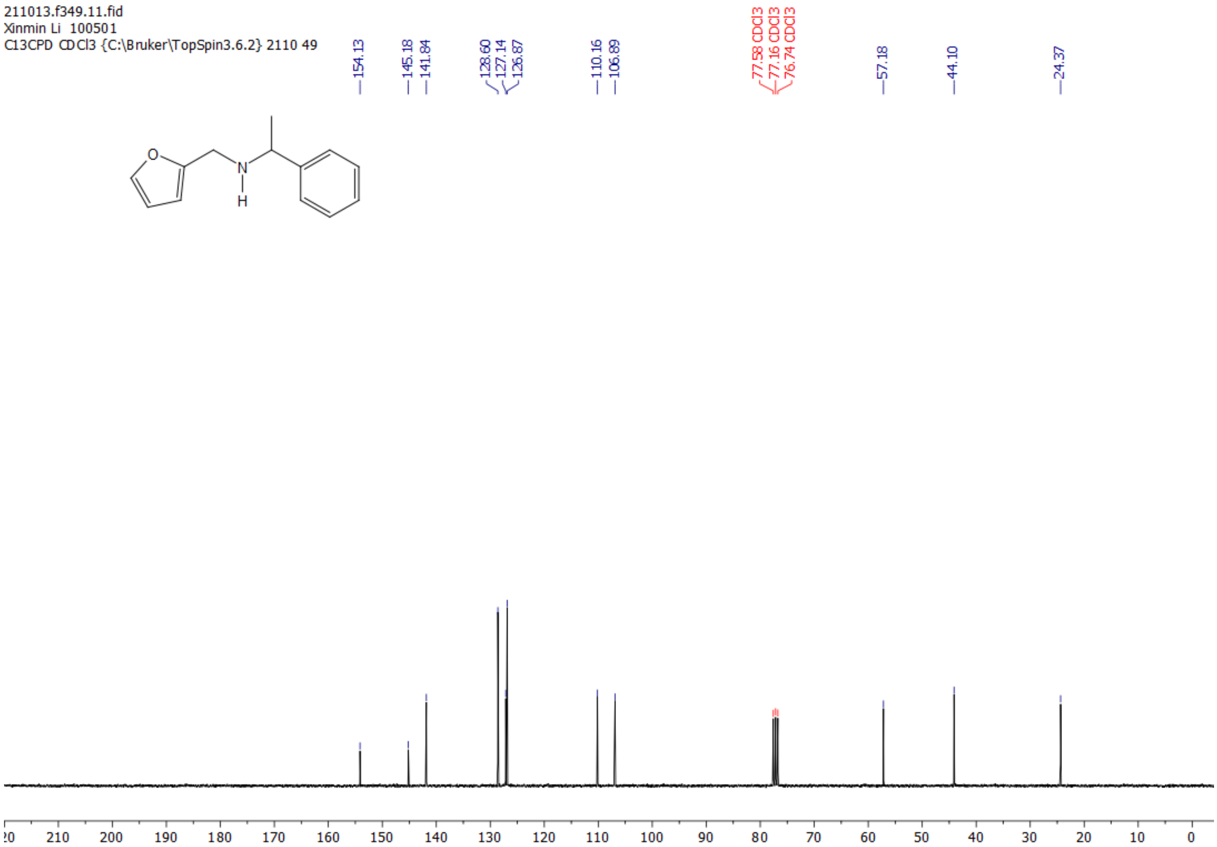
211001.f323.11.fid
Li/ XM100101
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 23



211013.f349.10.fid
Xinmin Li 100501
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 49

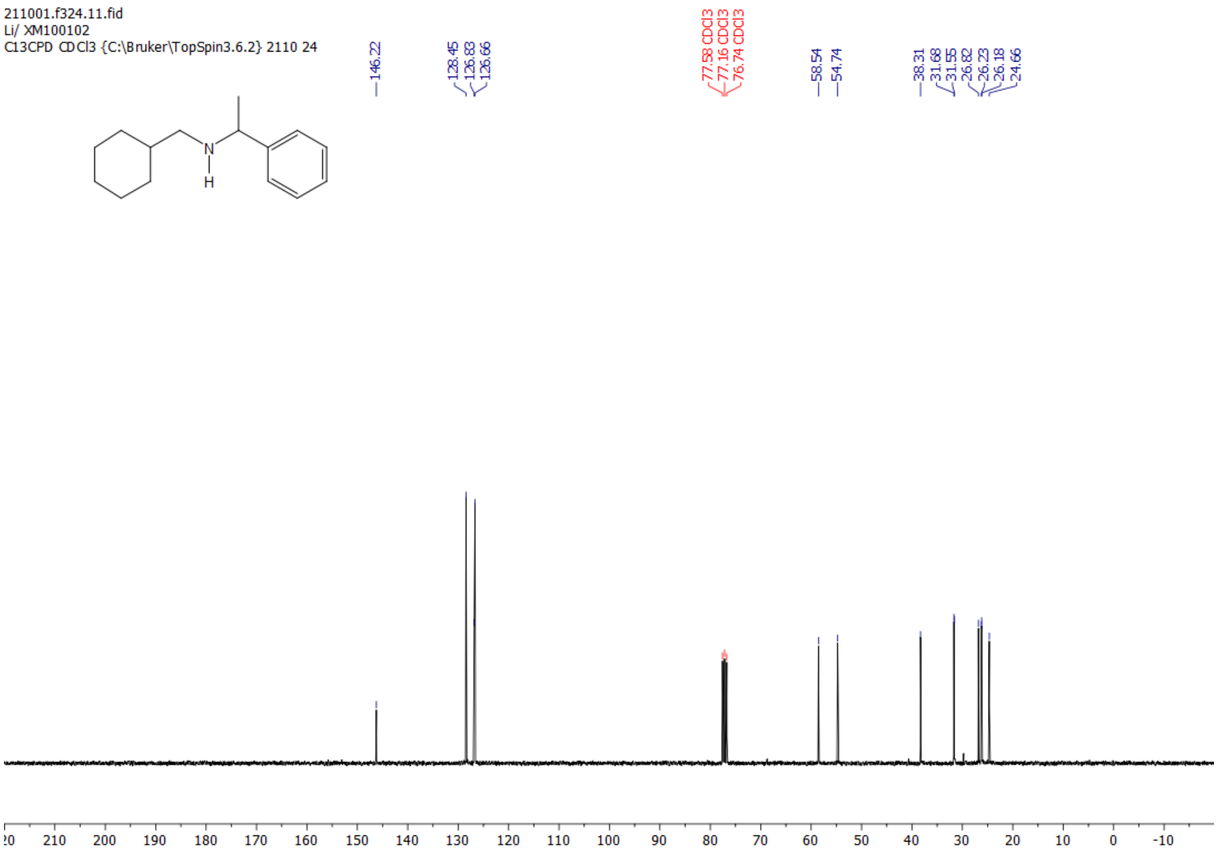


211013.f349.11.fid
Xinmin Li 100501
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 49

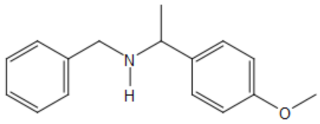


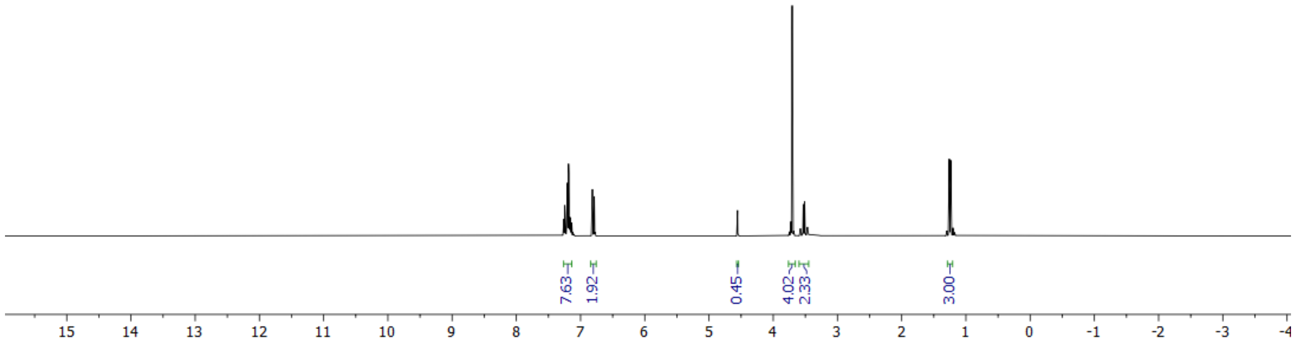
211001.f324.10.fid
Li/ XM100102
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 24

211001.f324.11.fid
Li/ XM100102
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 24

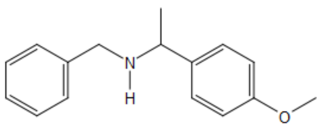


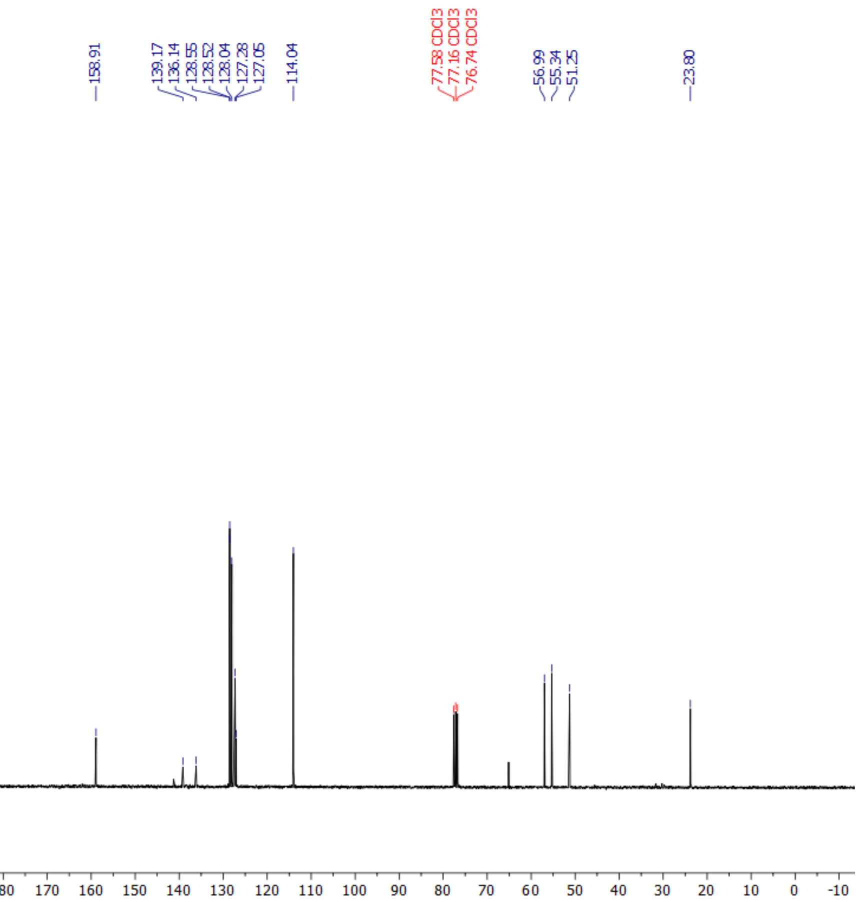
211210.323.10.fid
Xinmin Li 1209-02
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 23



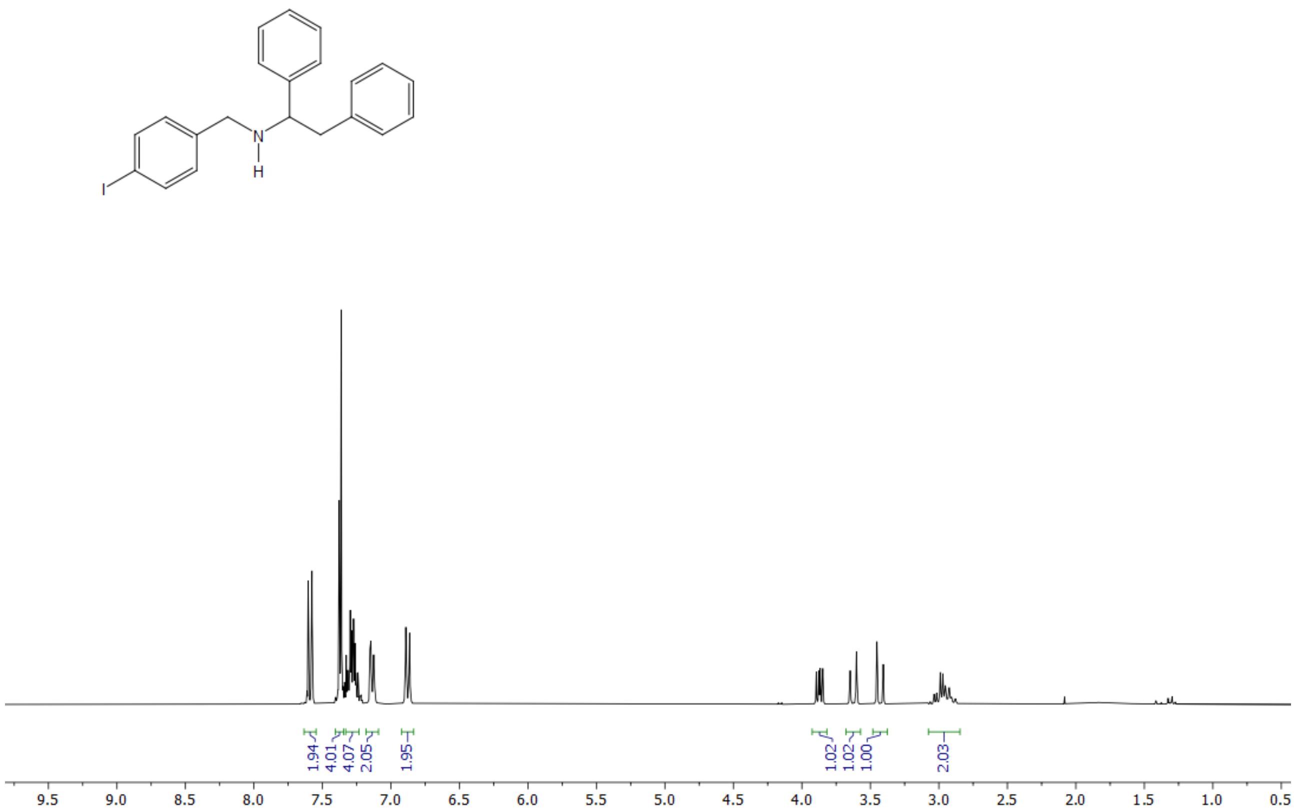


211210.323.11.fid
Xinmin Li 1209-02
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 23

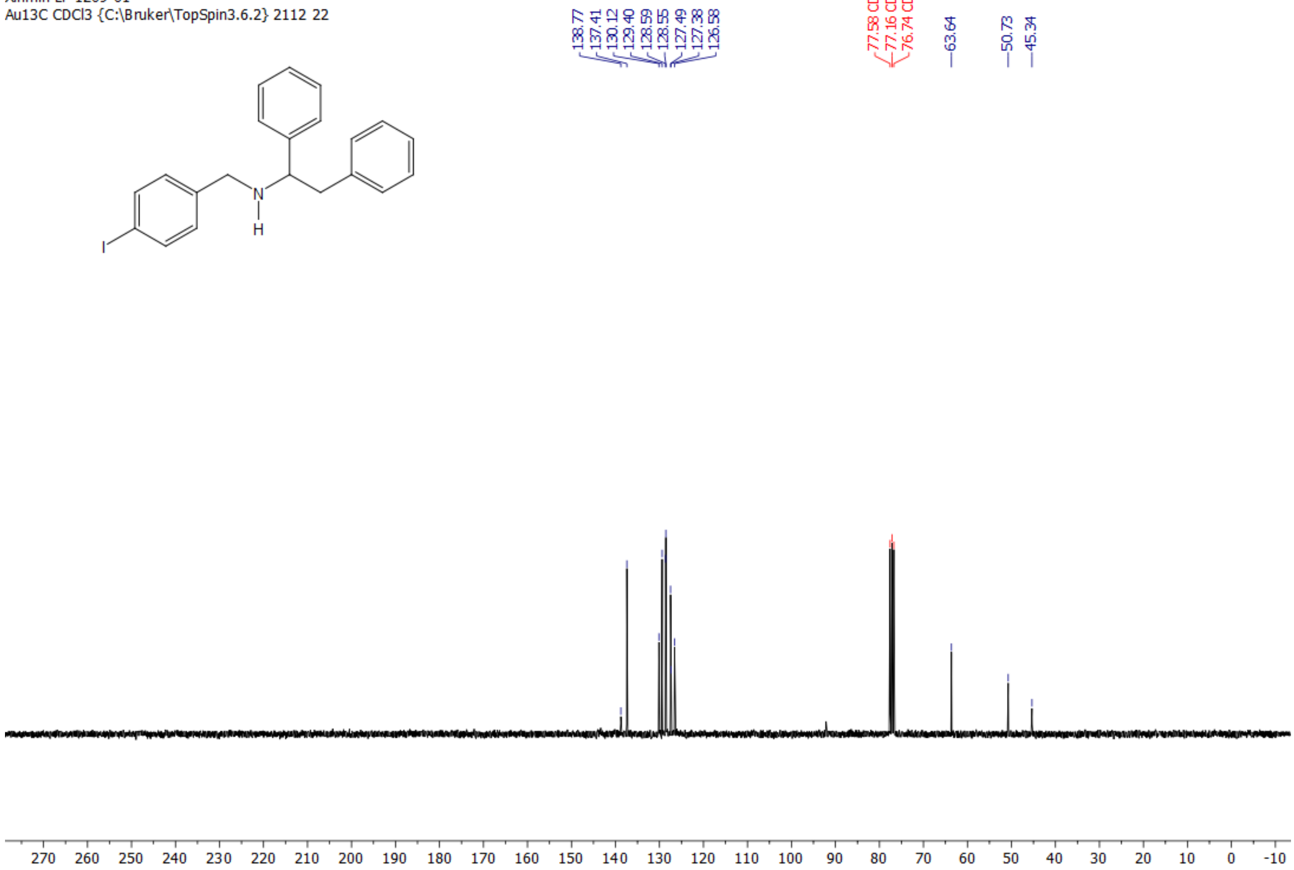


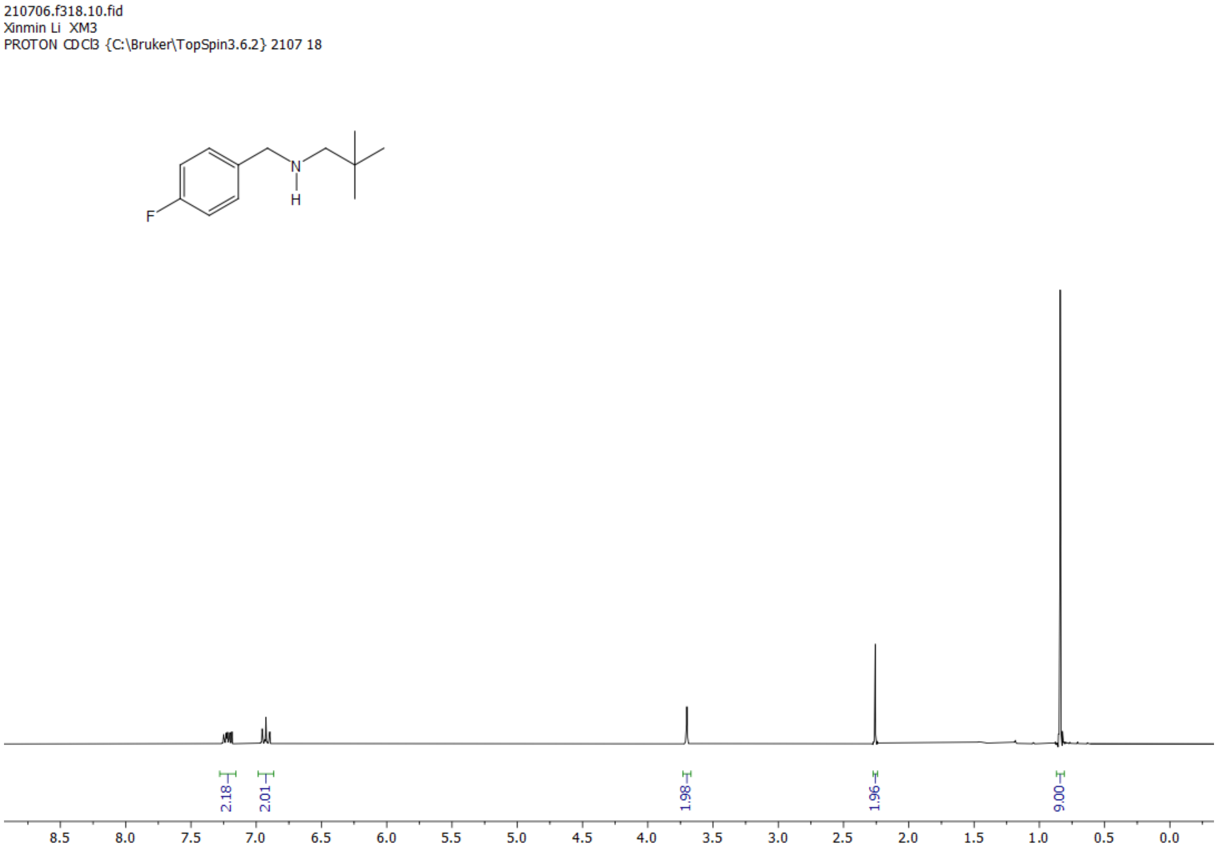


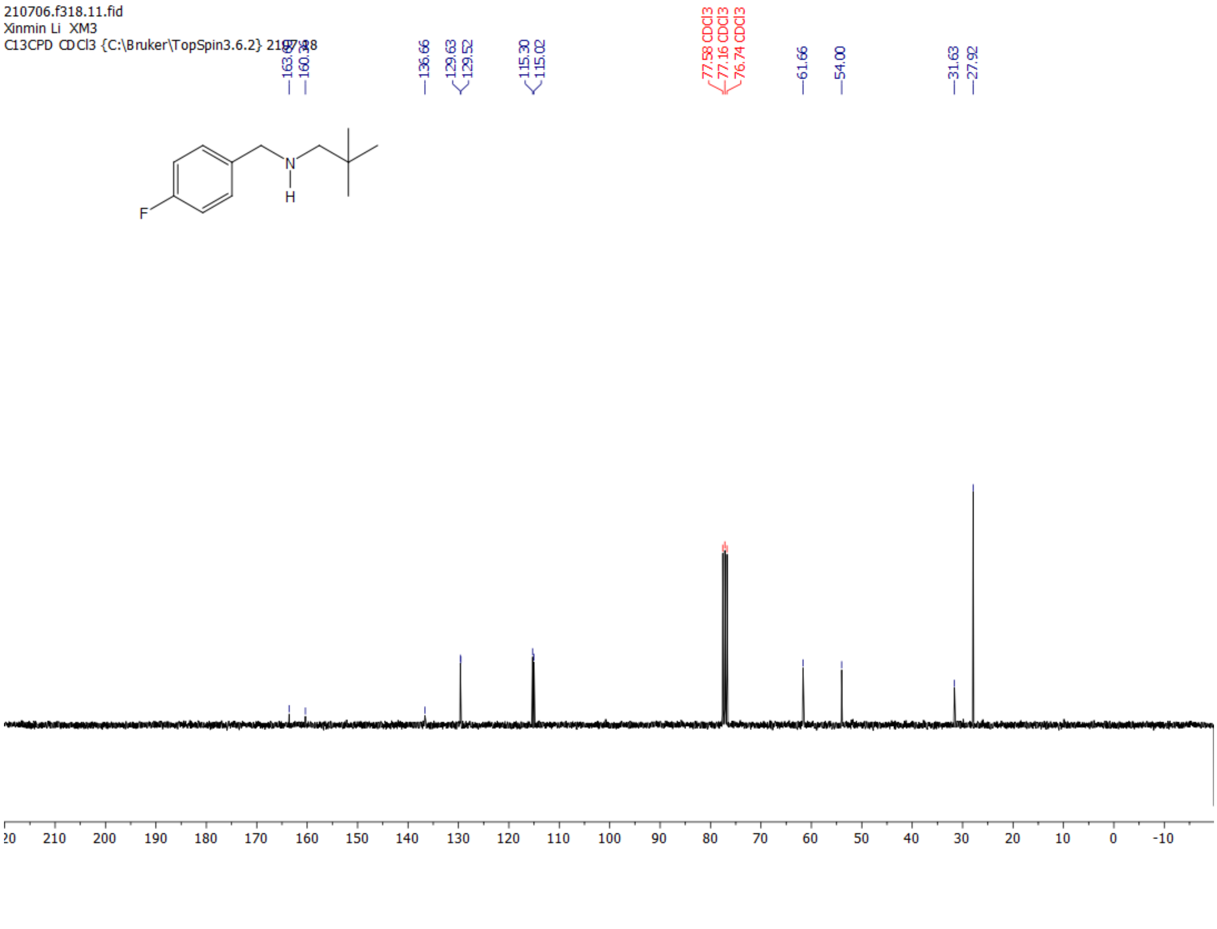
211210.322.10.fid
Xinmin Li 1209-01
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 22



211210.322.11.fid
Xinmin Li 1209-01
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 22

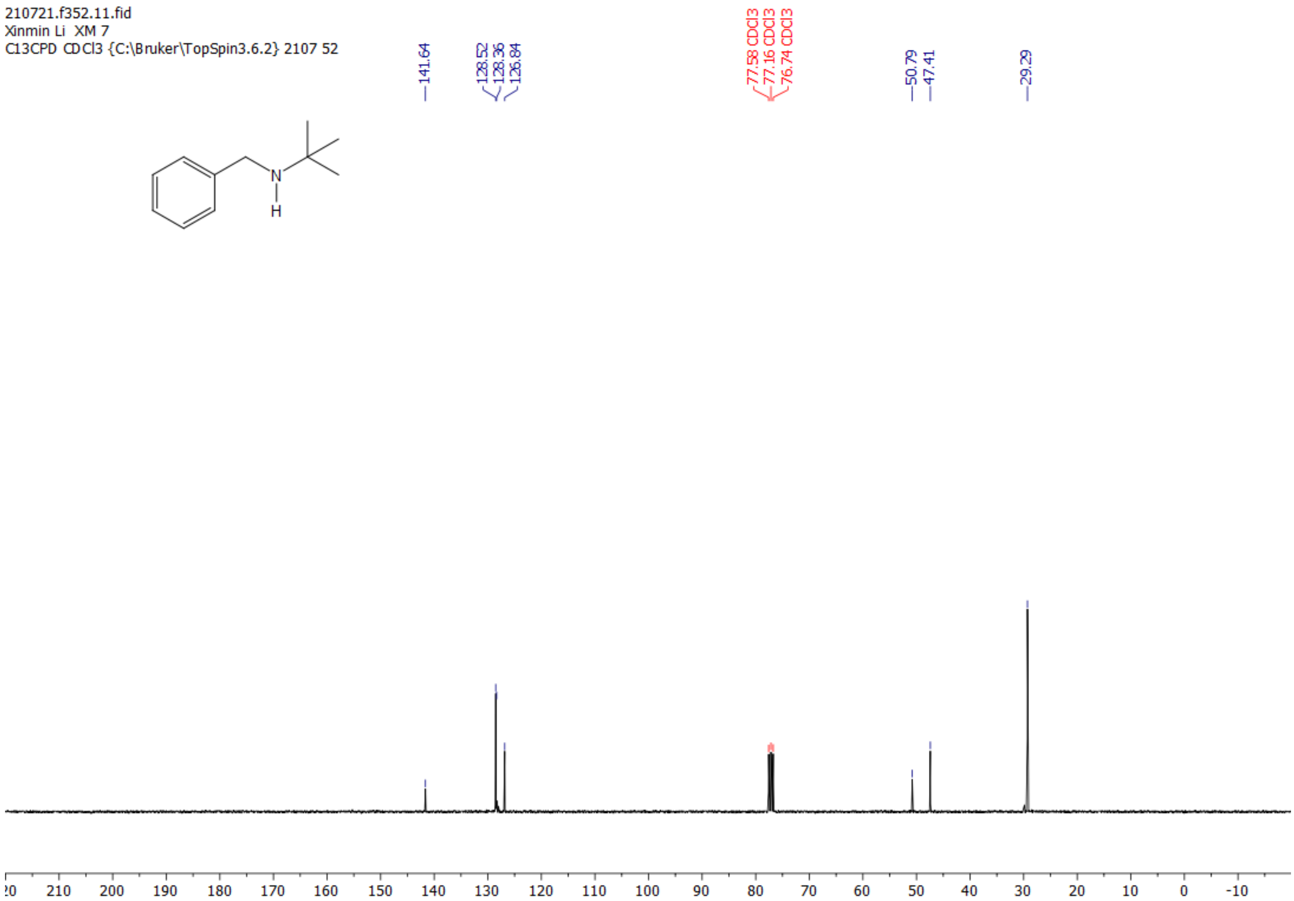



210706.f318.10.fid
Xinmin Li XM3
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 18
F
N
H
2.18
2.01
1.98
1.96
9.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

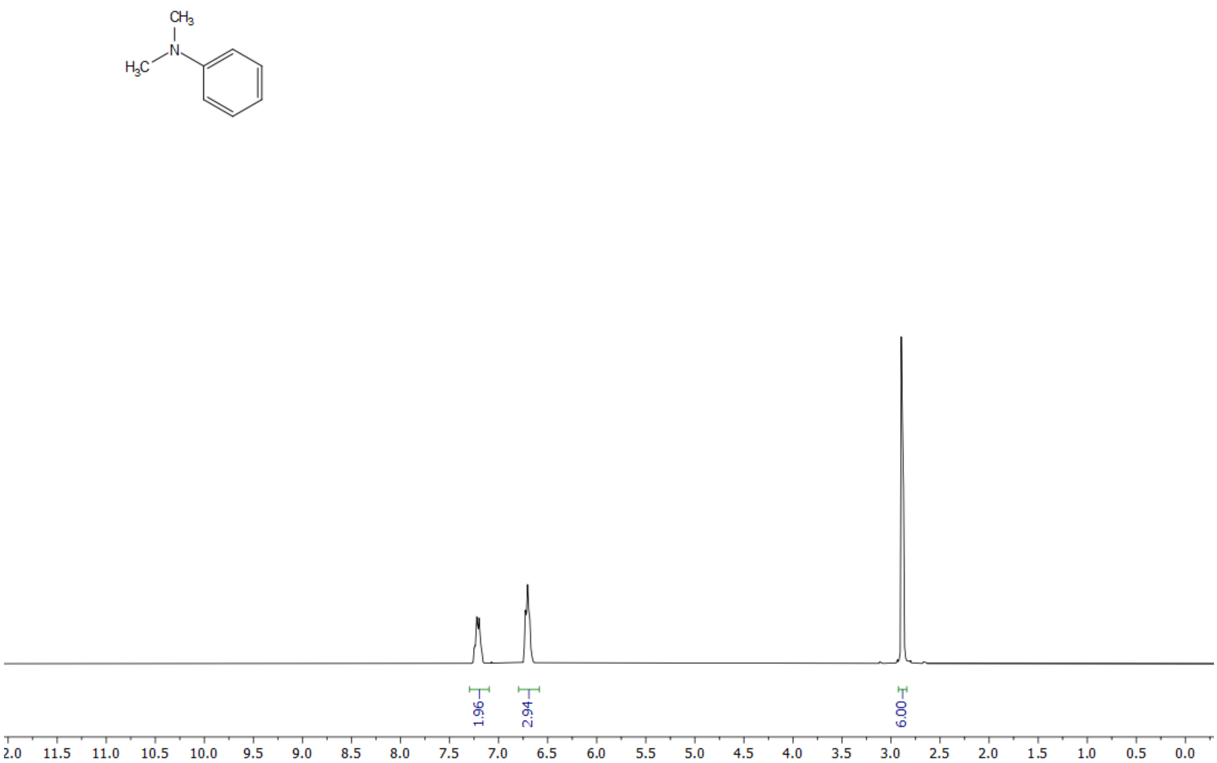

210706.f318.11.fid
Xinmin Li XM3
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 18
136.66
129.63
129.52
115.30
115.02
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
61.66
54.00
31.63
27.92
F
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

210721.f352.10.fid
Xinmin Li XM 7
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 52

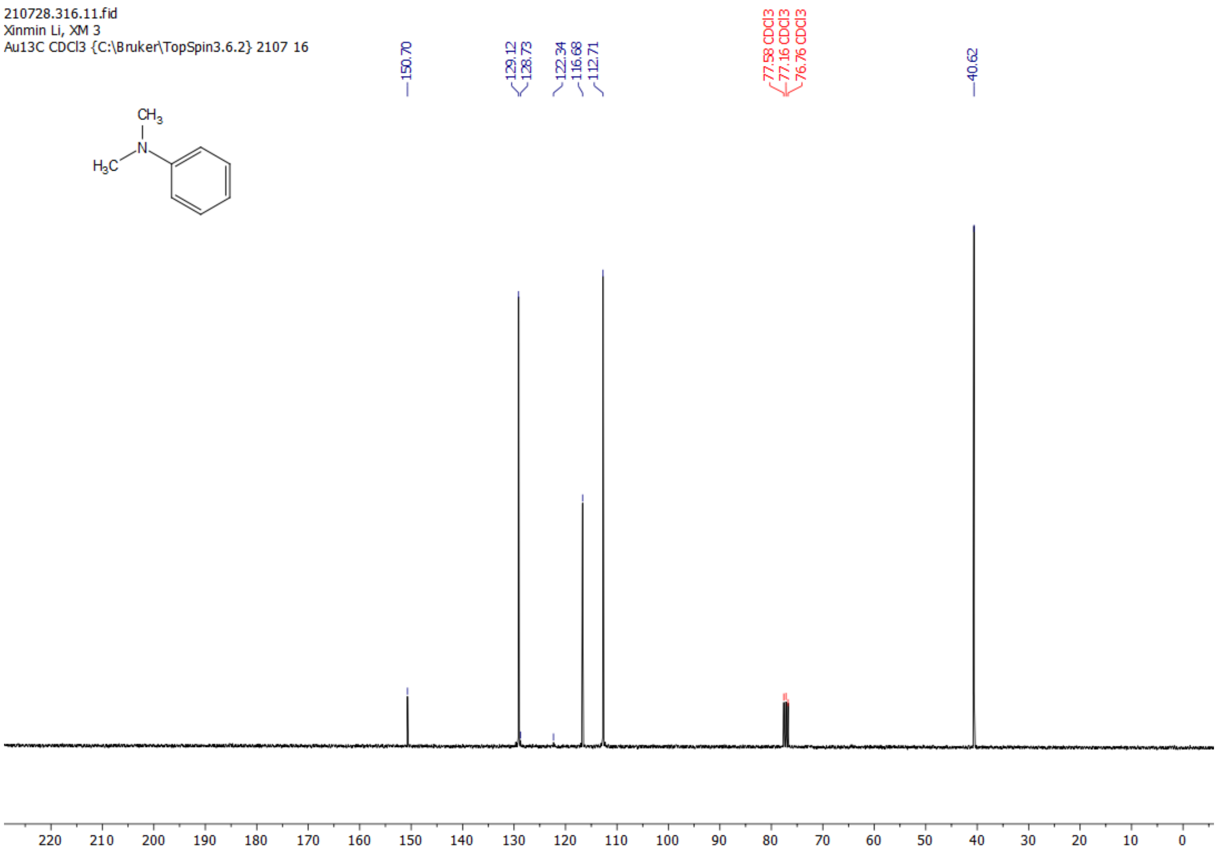
210721.f352.11.fid
Xinmin Li XM 7
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 52

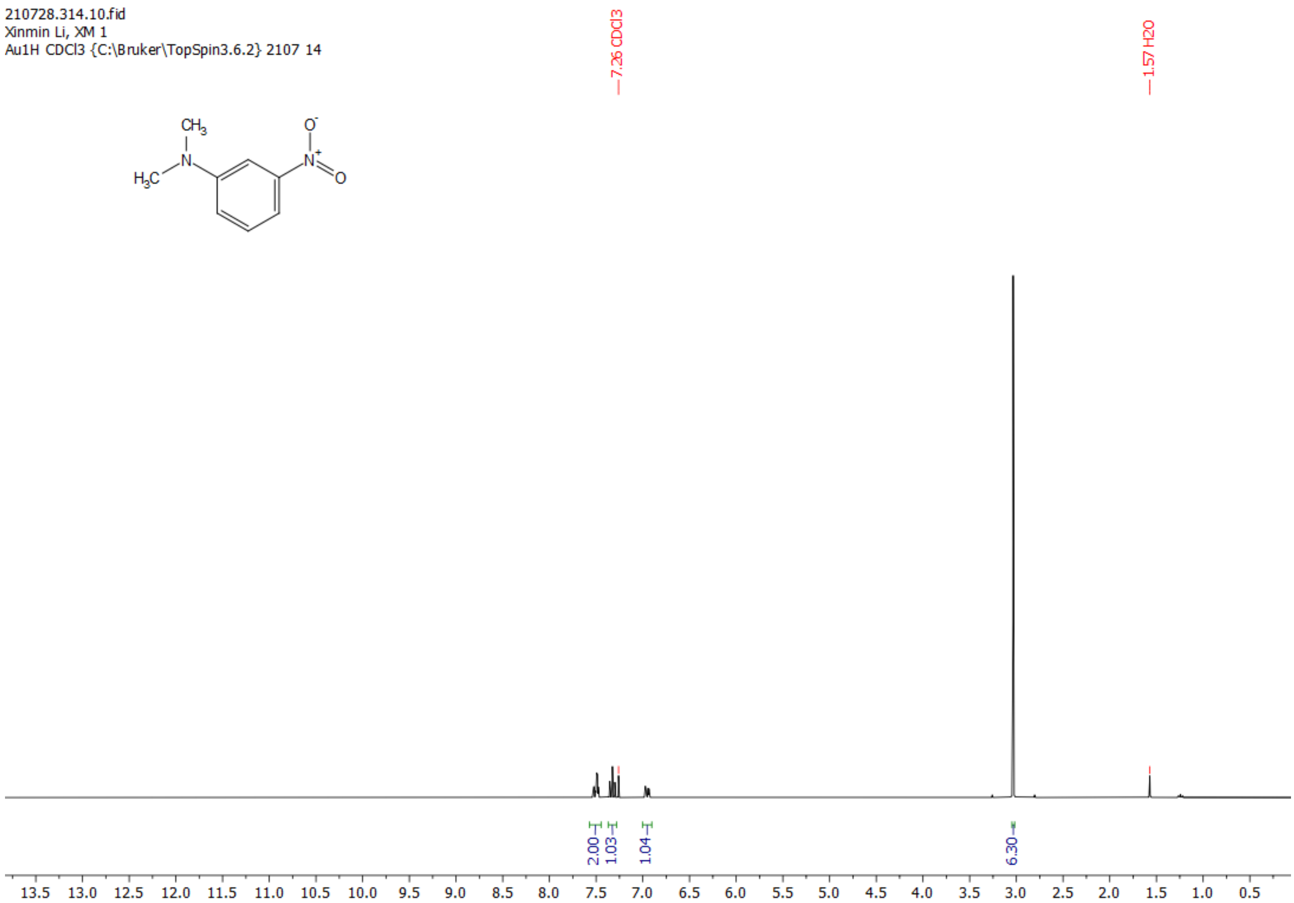


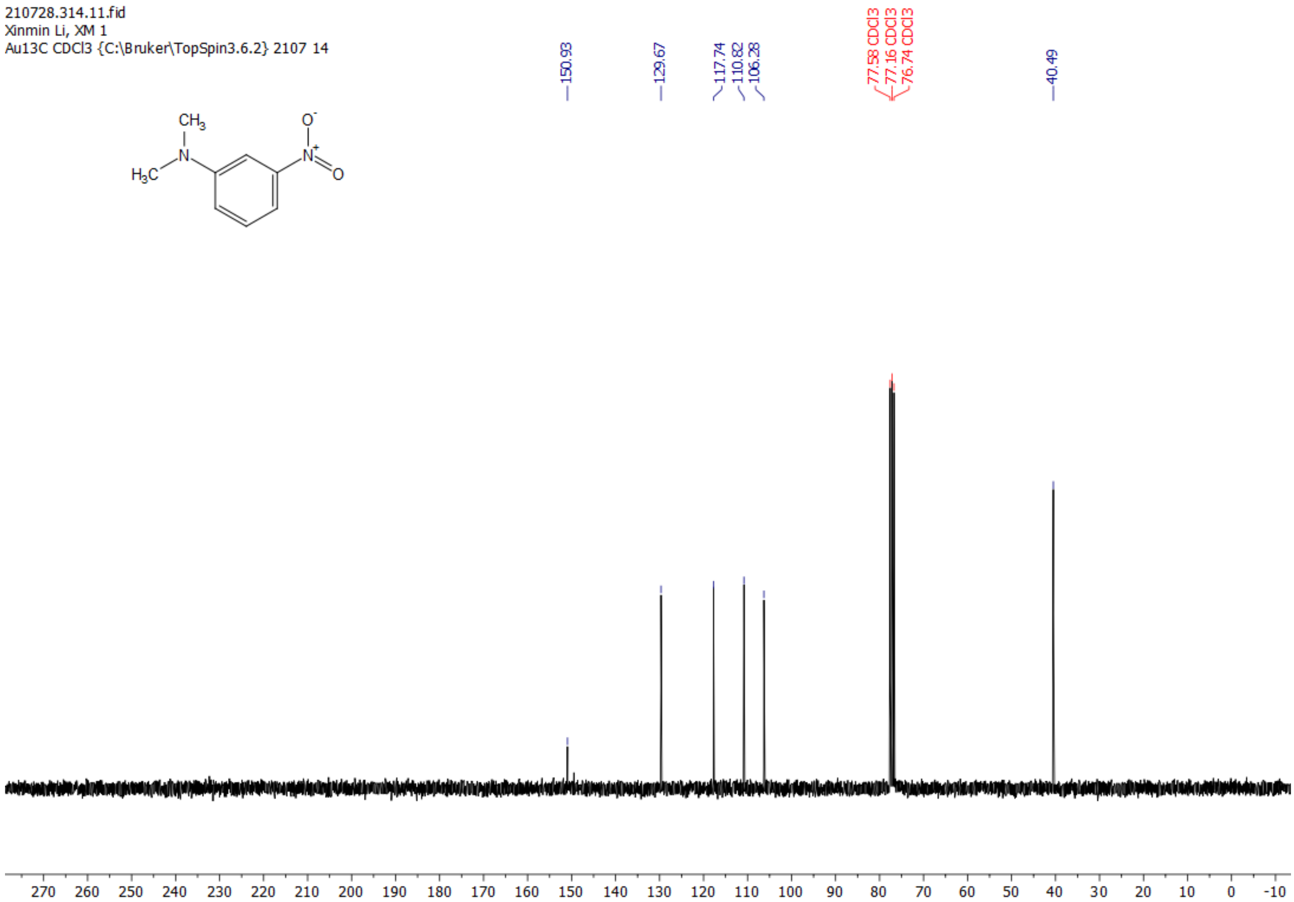
210728.316.10.fid
Xinmin Li, XM 3
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 16



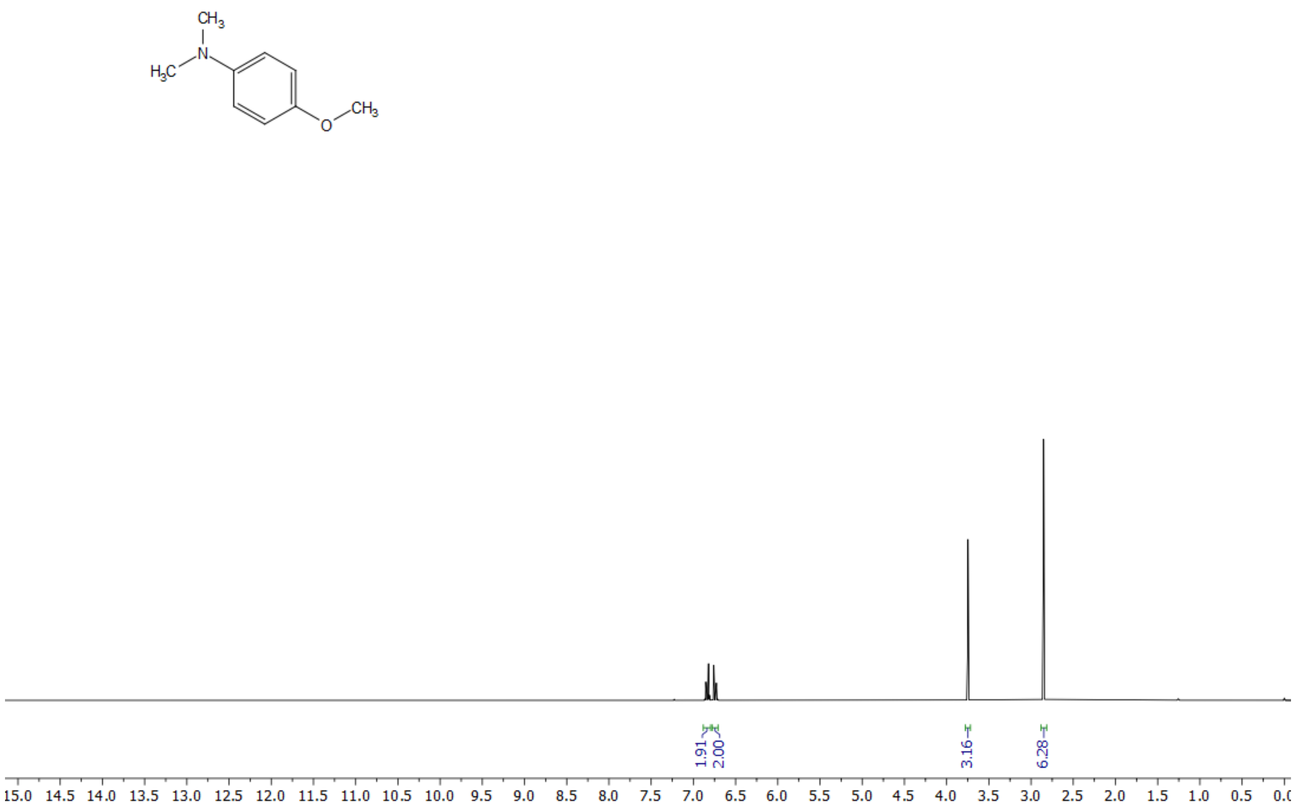
210728.316.11.fid
Xinmin Li, XM 3
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 16



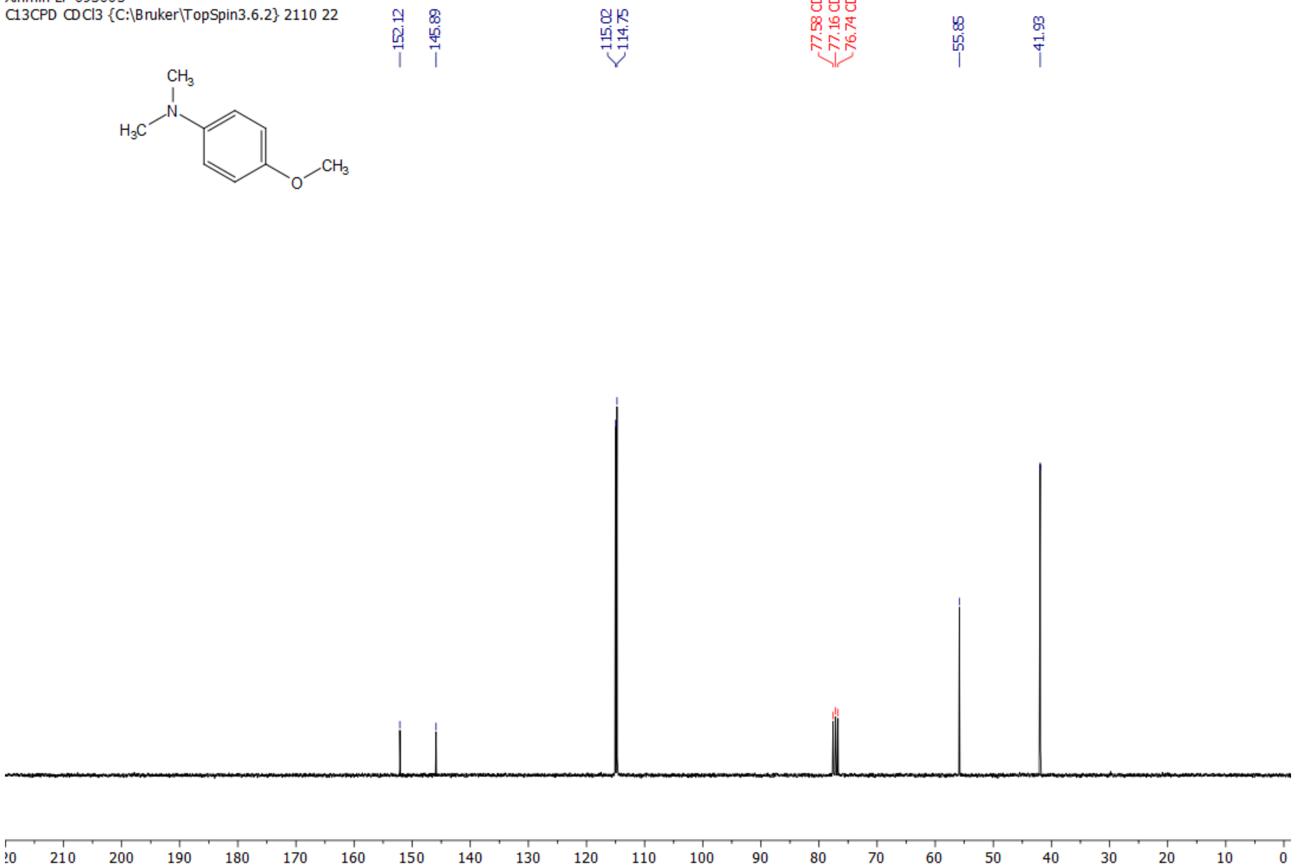

210728.314.10.fid
Xinmin Li, XM 1
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14
7.26 CDCl3
1.57 H2O
2.00
1.03
1.04
6.30

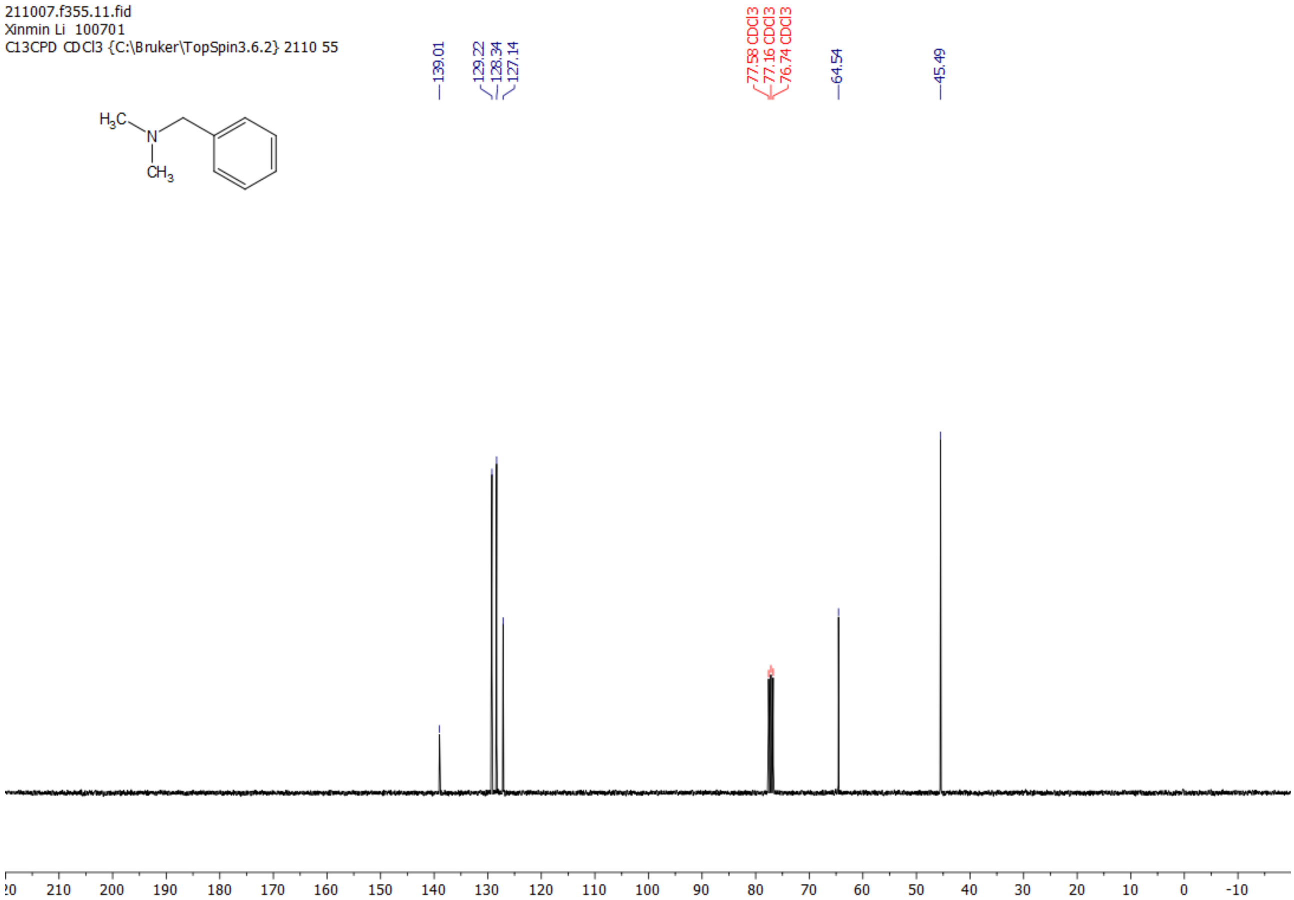

210728.314.11.fid
Xinmin Li, XM 1
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14
150.93
129.67
117.74
110.82
106.28
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
40.49

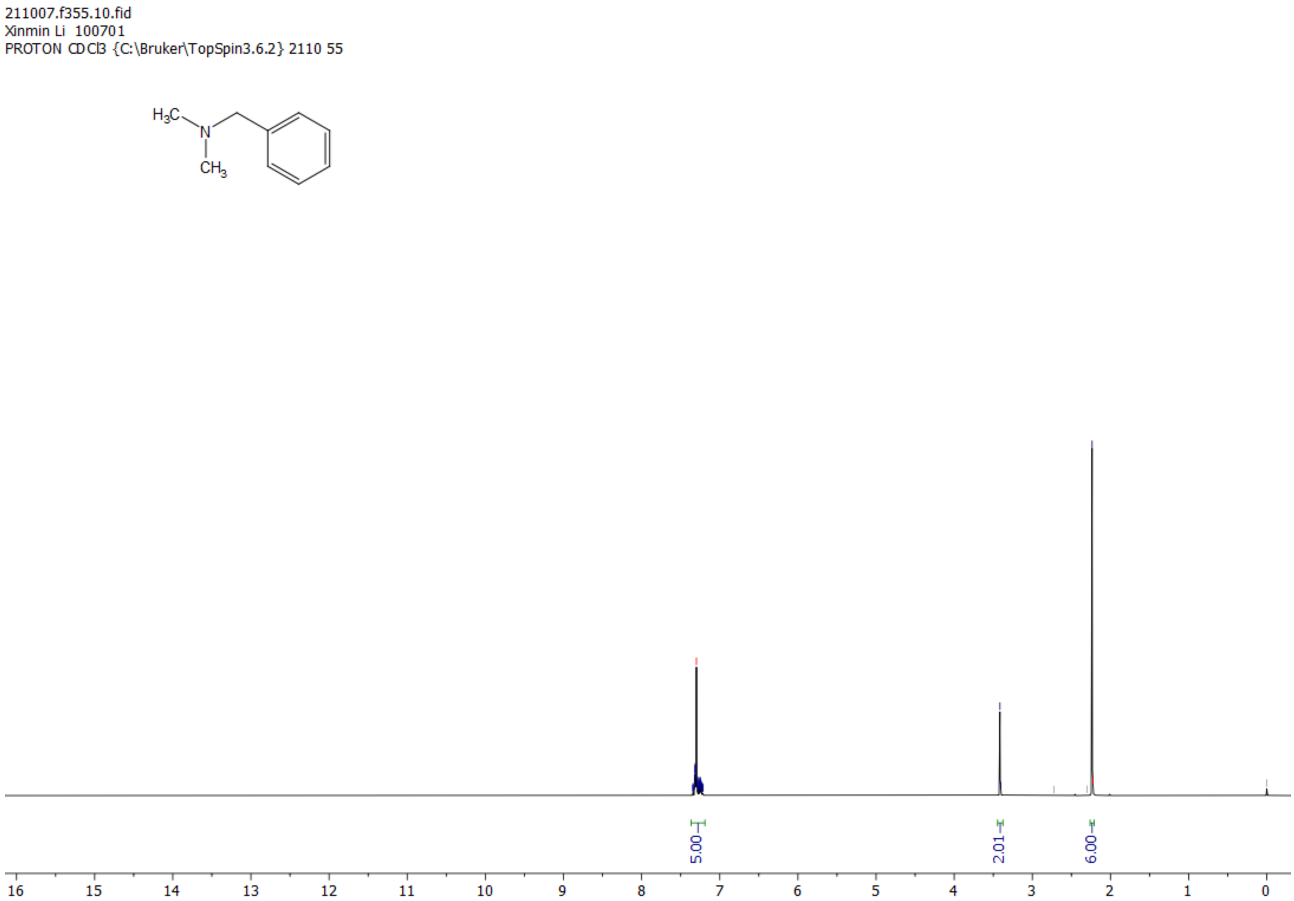
211004.f322.10.fid
Xinmin Li 093003
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 22



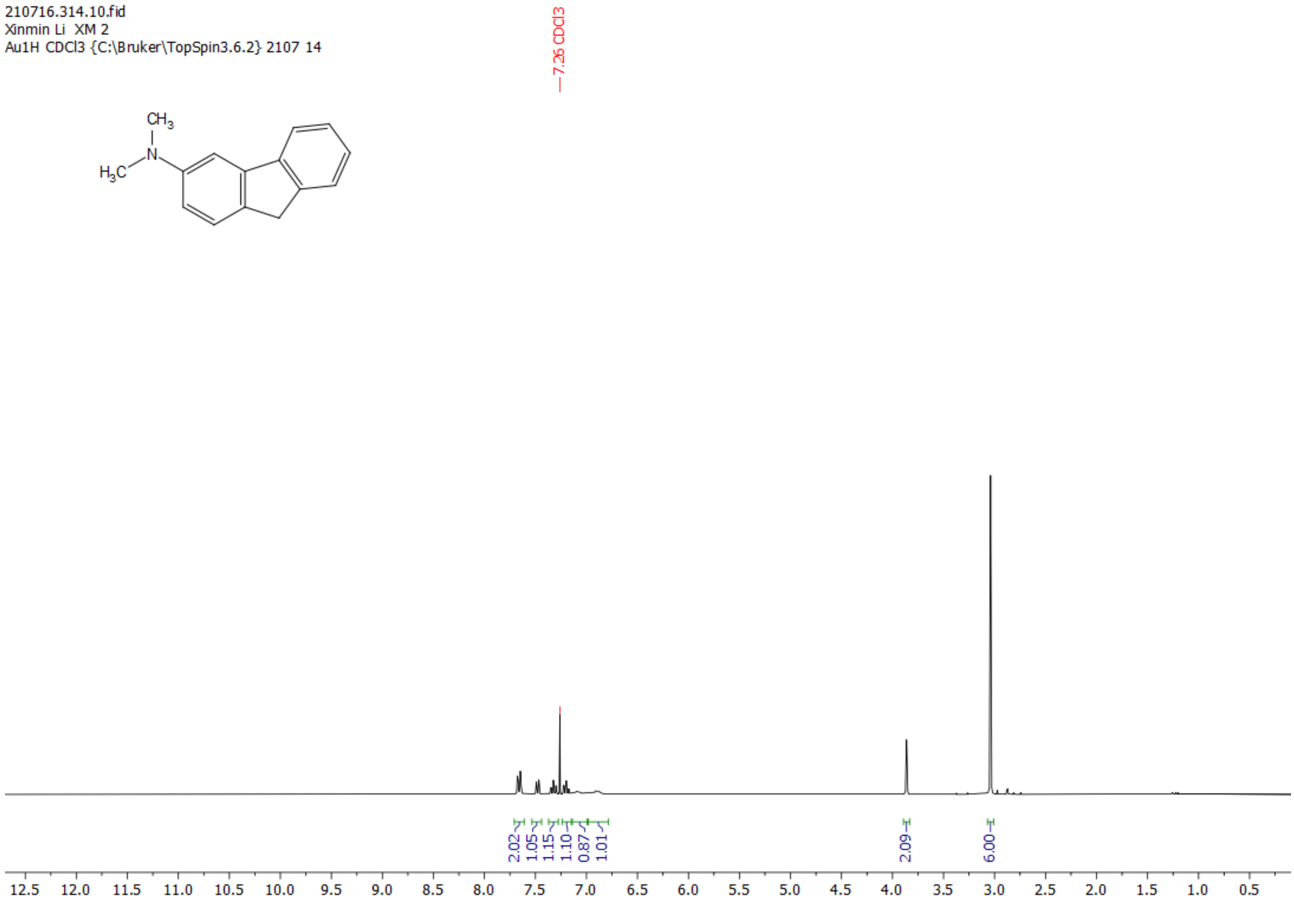
211004.f322.11.fid
Xinmin Li 093003
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 22



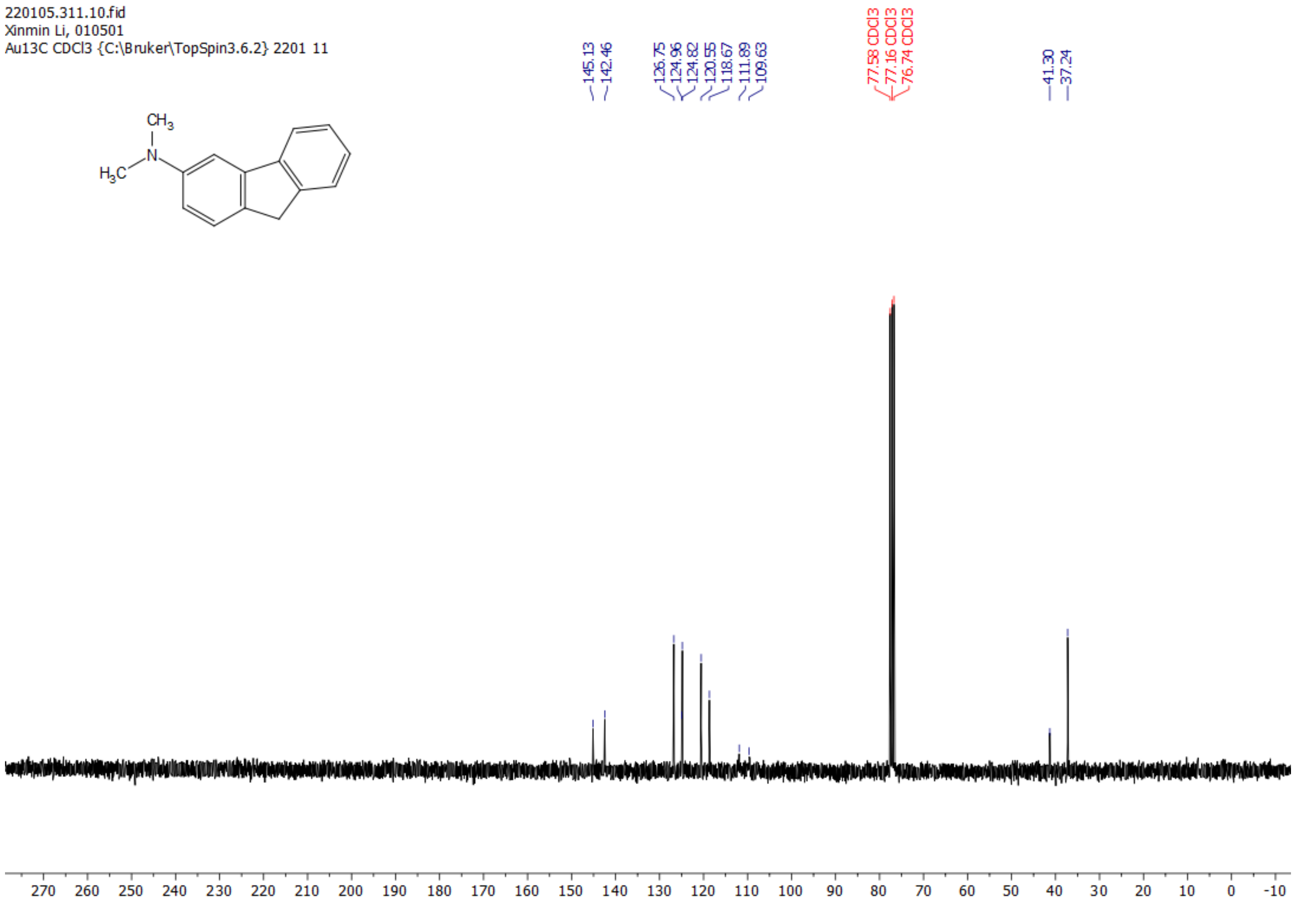

211007.f355.11.fid
Xinmin Li 100701
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 55
139.01
129.22
128.34
127.14
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
64.54
45.49


211007.f355.10.fid
Xinmin Li 100701
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 55
5.00
2.01
6.00

210716.314.10.fid
Xinmin Li XM 2
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 14

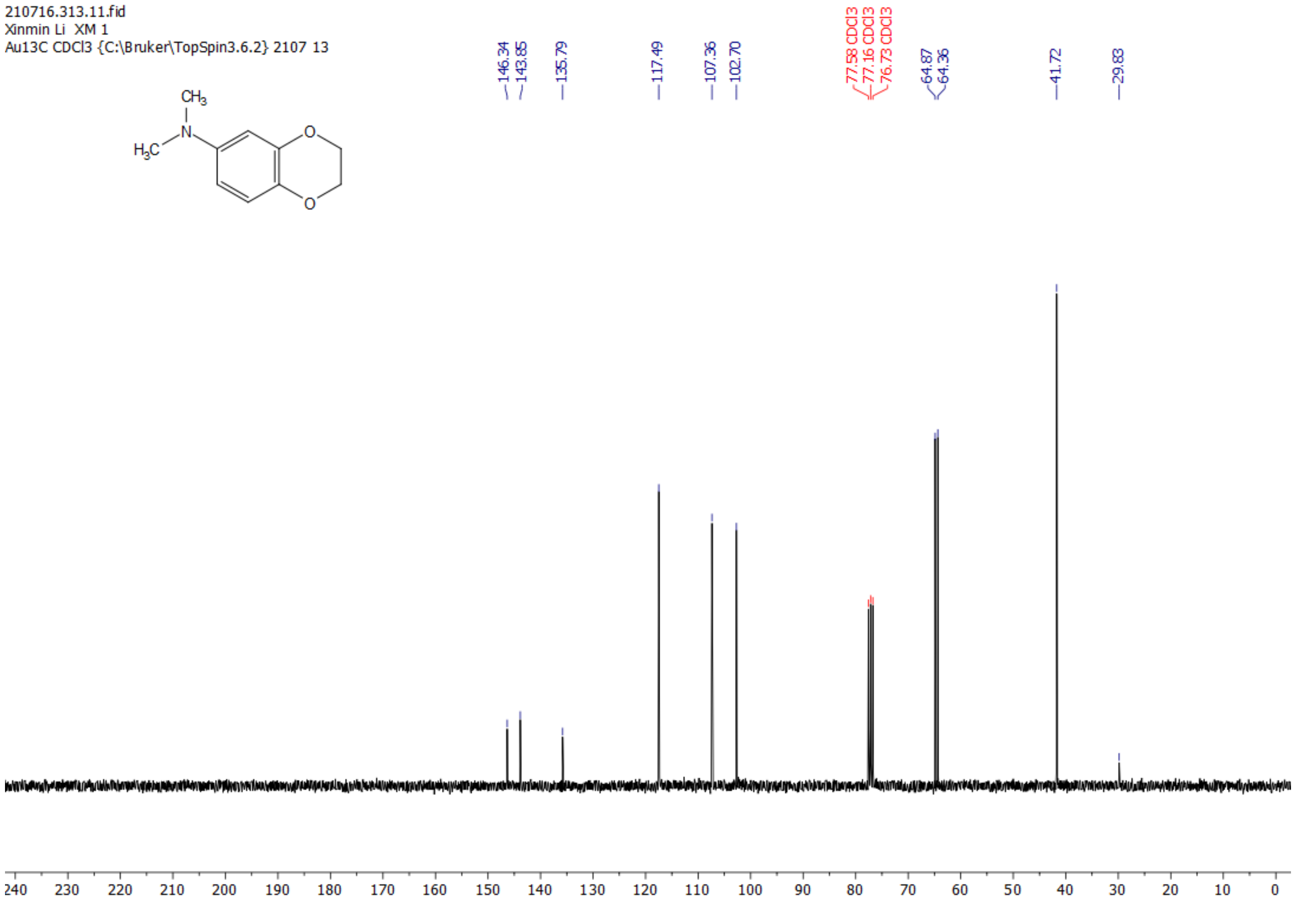


220105.311.10.fid
Xinmin Li, 010501
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2201 11



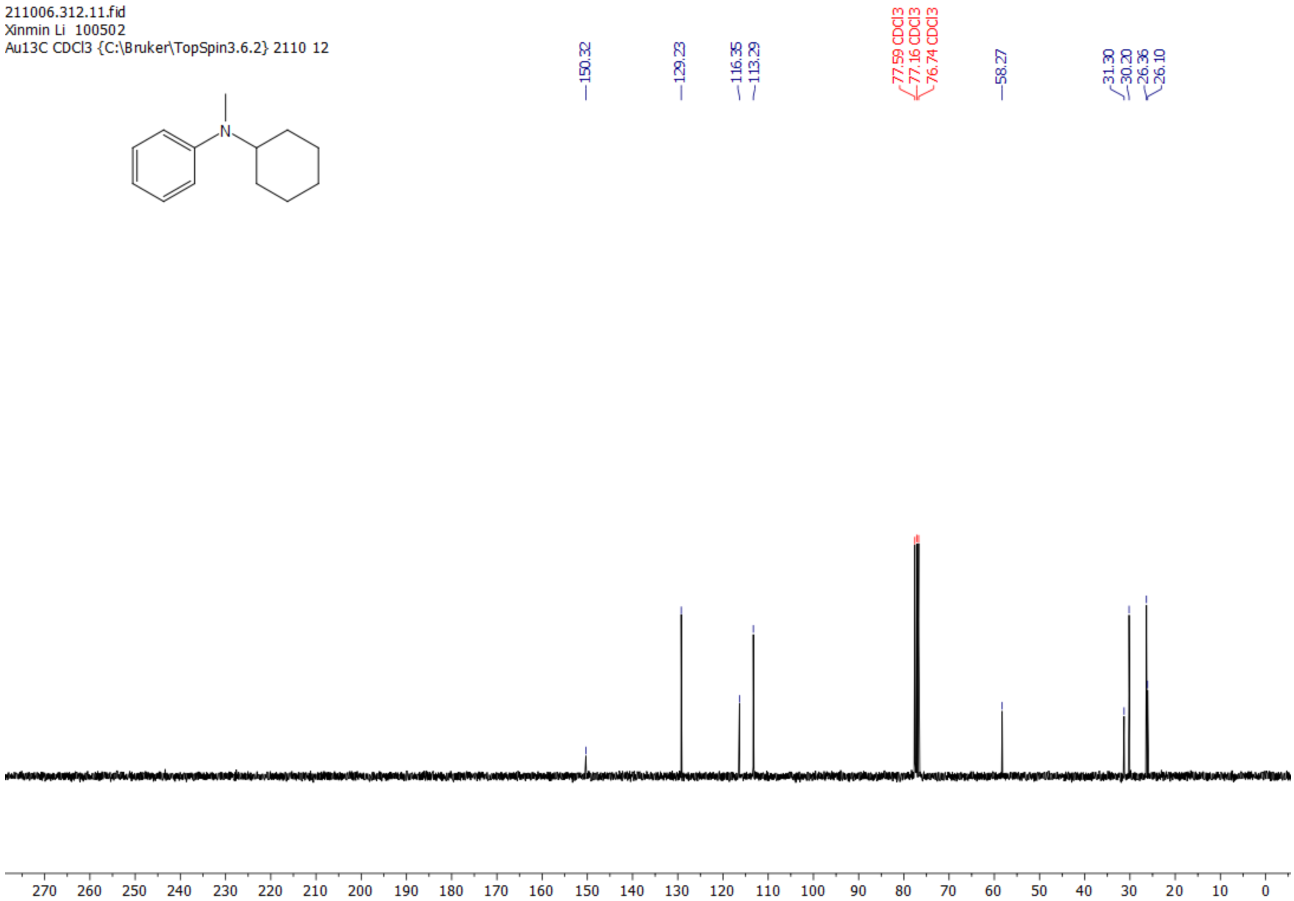
210716.313.10.fid
Xinmin Li XM 1
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 13

210716.313.11.fid
Xinmin Li XM 1
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 13

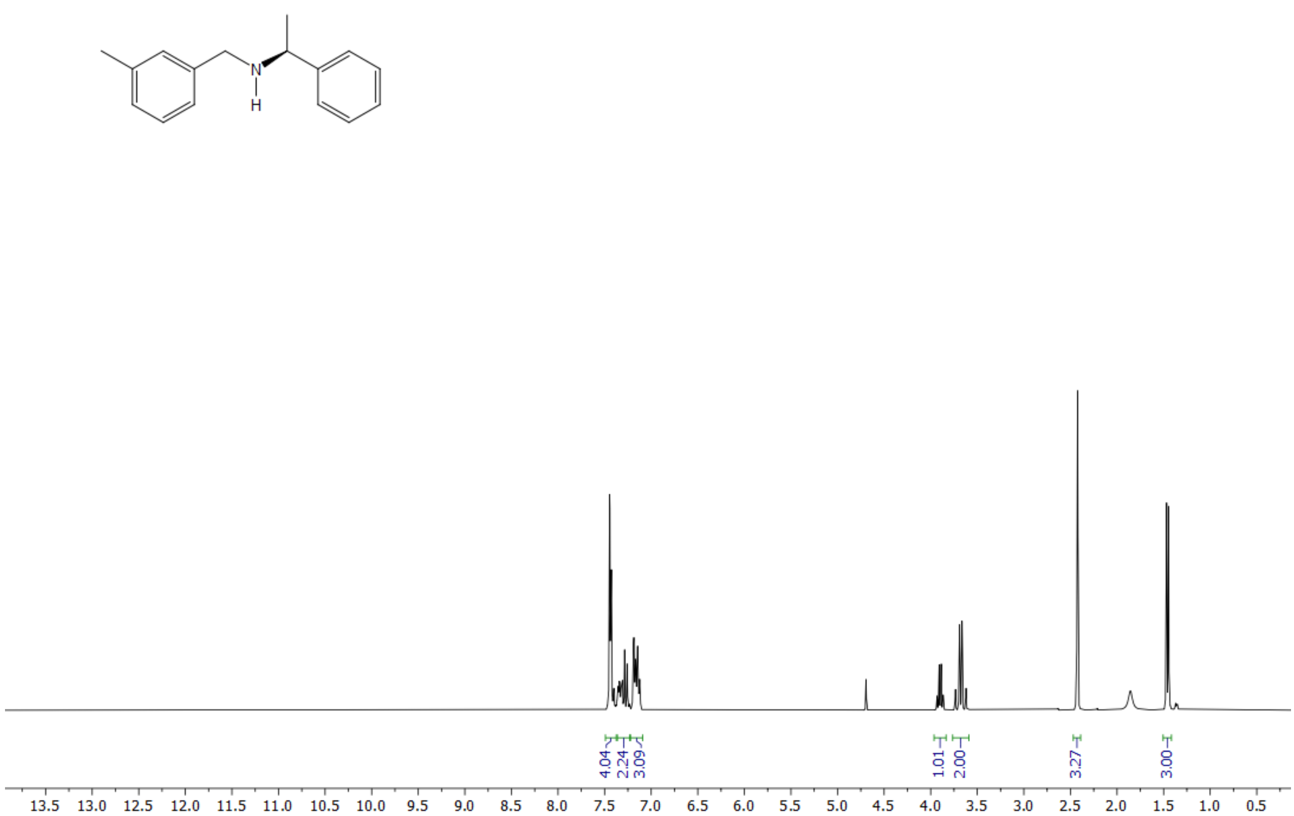


211006.312.10.fid
Xinmin Li 100502
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 12

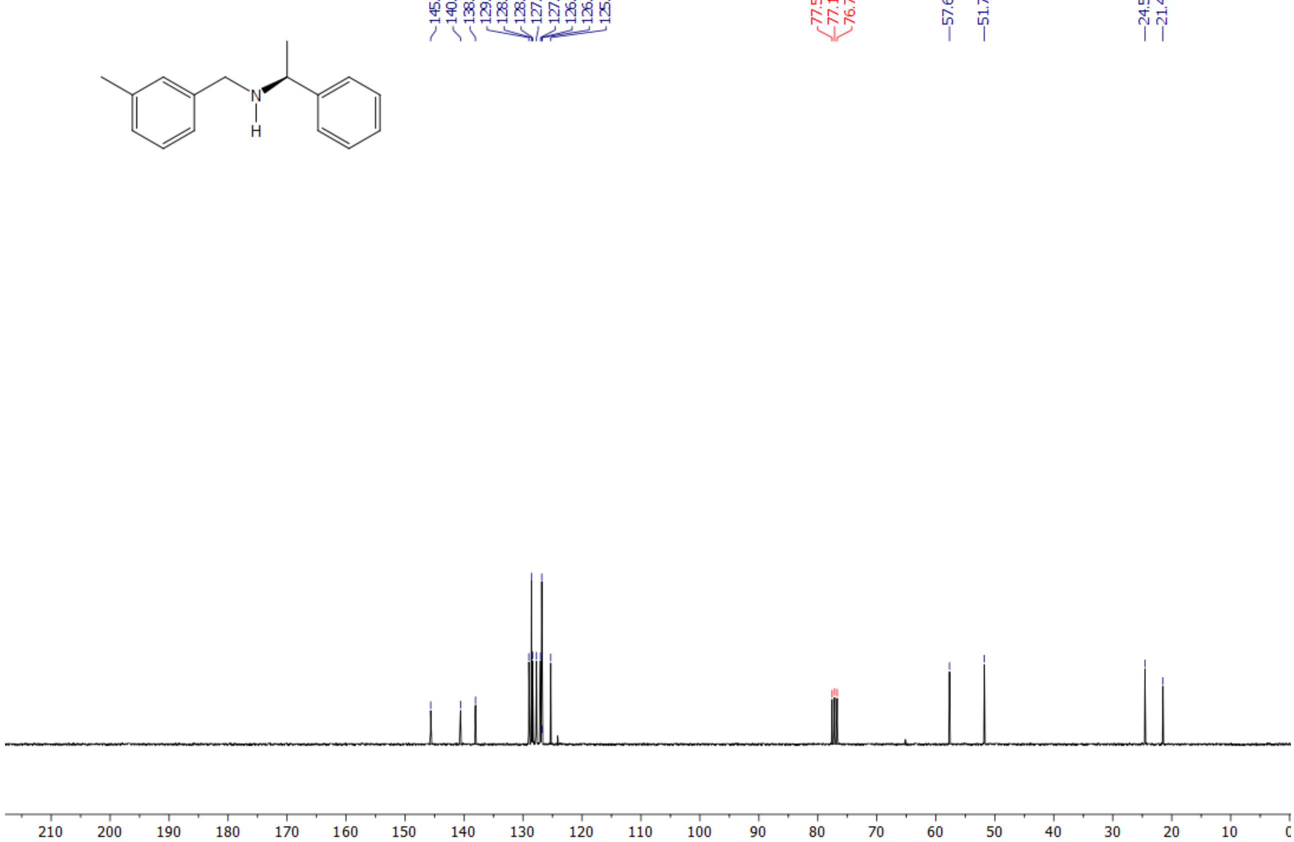
211006.312.11.fid
Xinmin Li 100502
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 12



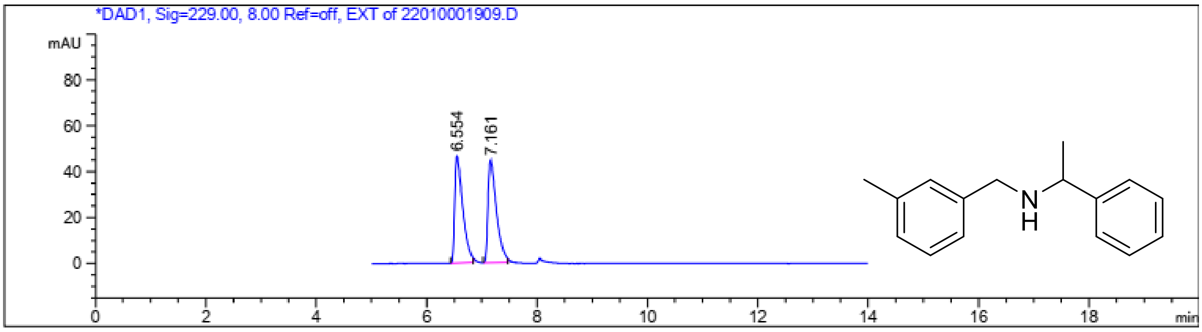
210706.f317.10.fid
Xinmin Li XM2
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 17



210706.f317.11.fid
Xinmin Li XM2
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 17

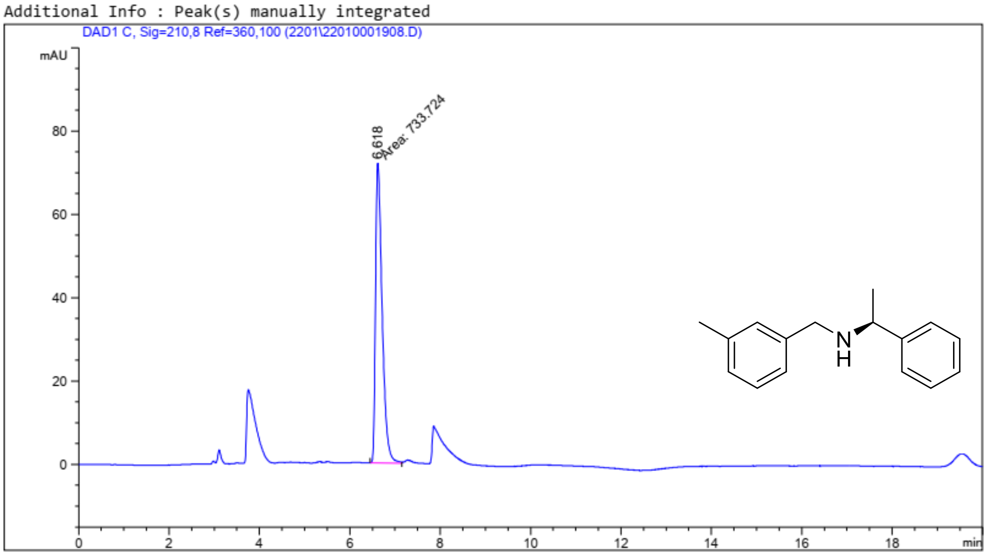


```
---------------------------------------------------------------------
Acq. Operator    : Analytik                          Seq. Line :   6
Acq. Instrument : LC5                                 Location : Vial 41
Injection Date  : 1/19/2022 4:57:10 PM                     Inj :   1
                                                    Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\OJ-H.M
Last changed     : 1/19/2022 3:32:16 PM by Analytik
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\OJ-H.M
Last changed     : 1/21/2022 12:26:13 PM by Analytik
                   (modified after loading)
Method Info      : OJ-H , Hept./EtOH 98:2 , 1ml/min

Additional Info : Peak(s) manually integrated
```



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.554	BV	0.1510	3663.30371	353.17343	49.7071
2	7.161	VB	0.1620	3706.47656	337.93845	50.2929

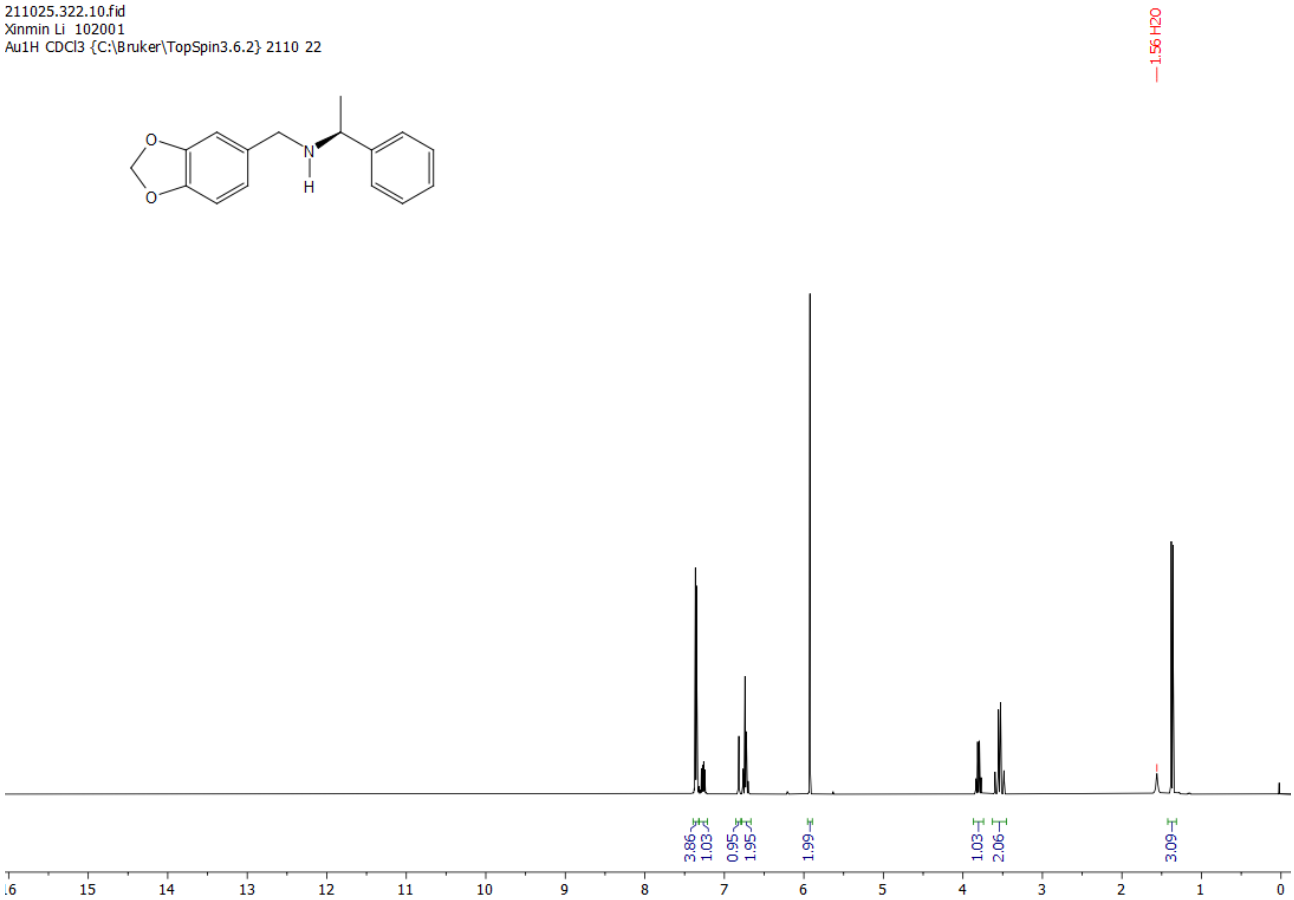
Additional Info : Peak(s) manually integrated



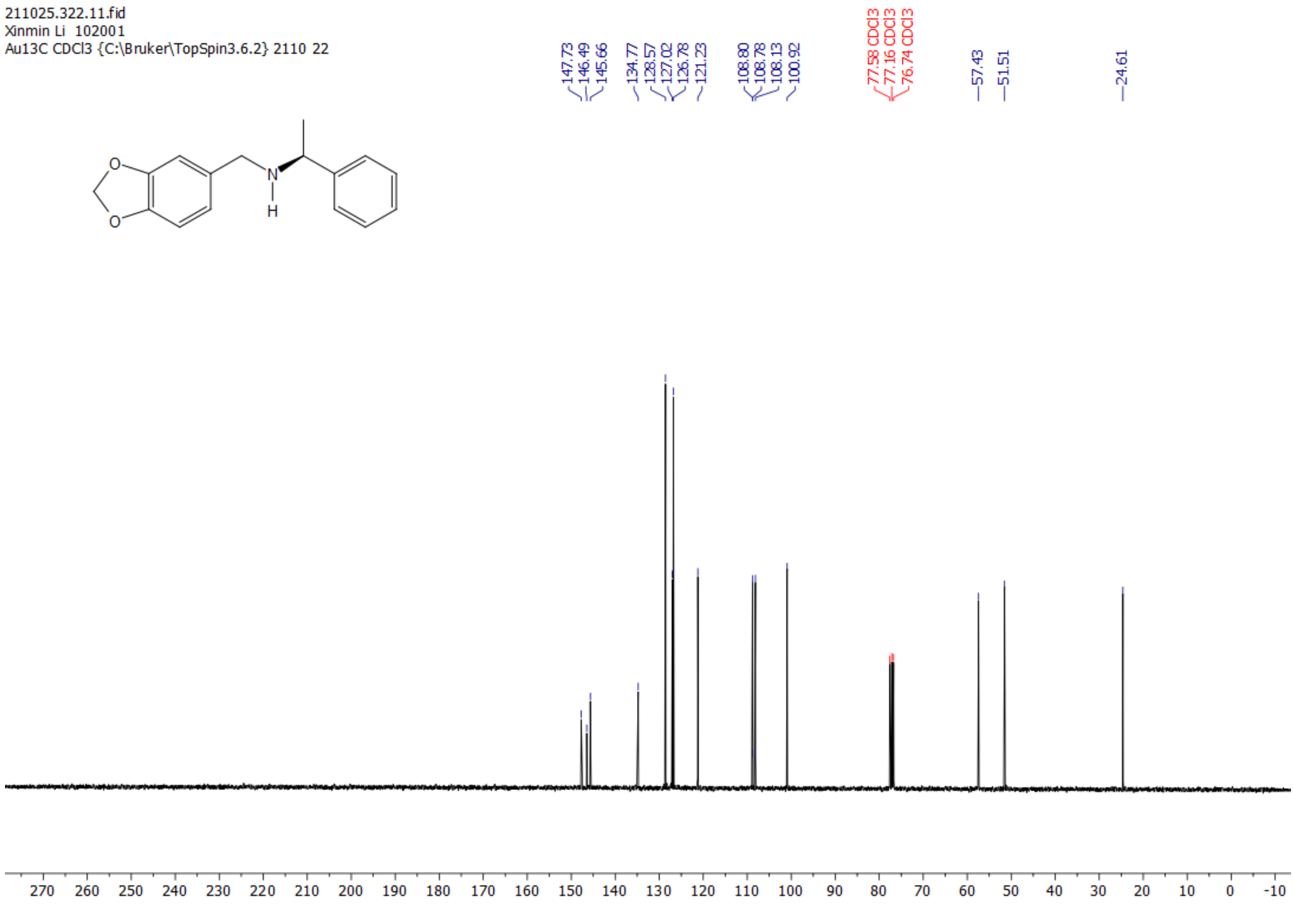
Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.618	MF	0.1697	733.72369	72.06634	100.0000

211025.322.10.fid
Xinmin Li 102001
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 22

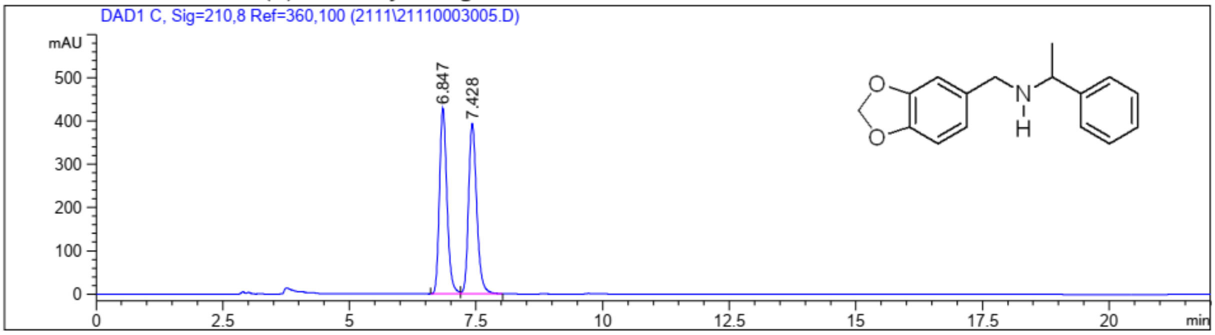


211025.322.11.fid
Xinmin Li 102001
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 22



```
Acq. Operator   : Analytik                         Seq. Line :   4
Acq. Instrument : LC5                               Location : Vial 1
Injection Date  : 11/30/2021 12:32:58 PM                 Inj :   1
                                                  Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 11/30/2021 1:06:19 PM by Analytik
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 12/1/2021 11:08:07 AM by Analytik
                  (modified after loading)
Method Info     : Amylose2 , Hept./EtOH 98:2 , 0.6ml/min
```

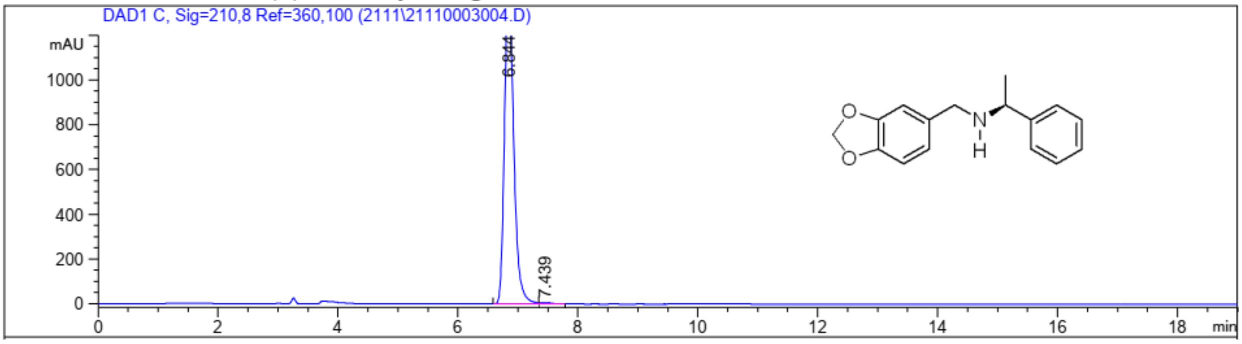
Additional Info : Peak(s) manually integrated



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.847	BV	0.1582	4415.57666	428.74023	49.7592
2	7.428	VB	0.1743	4458.31836	393.17505	50.2408

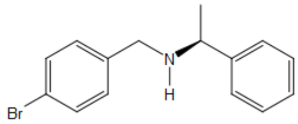
Additional Info : Peak(s) manually integrated

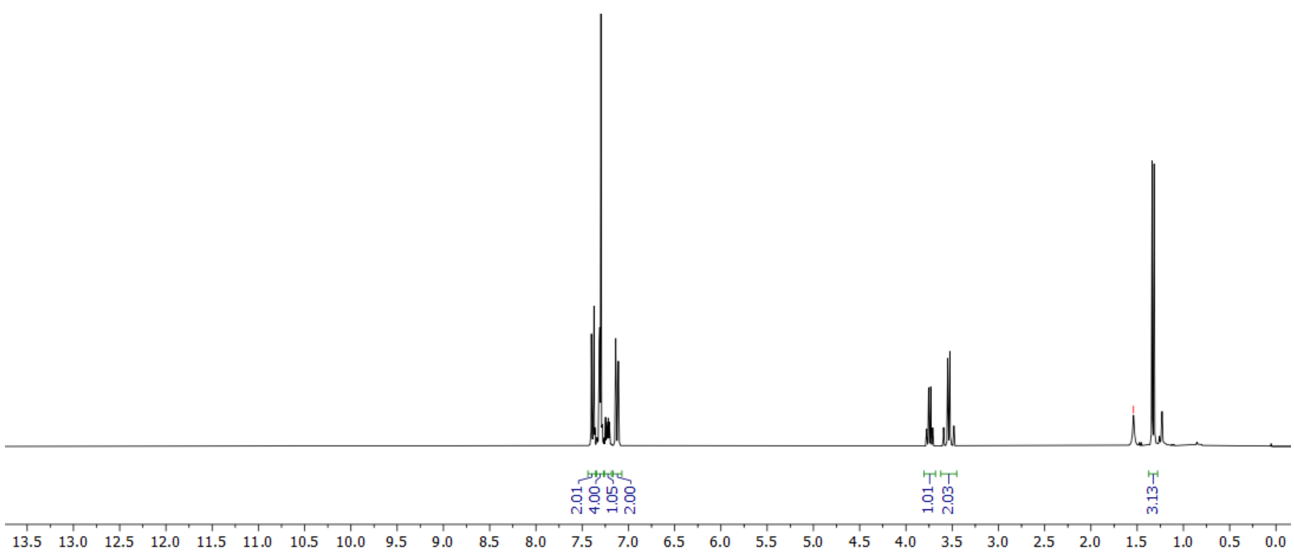


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

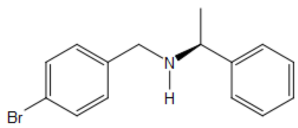
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.844	VV	0.1684	1.52210e4	1404.42493	99.4342
2	7.439	VB	0.1899	86.60313	6.65014	0.5658

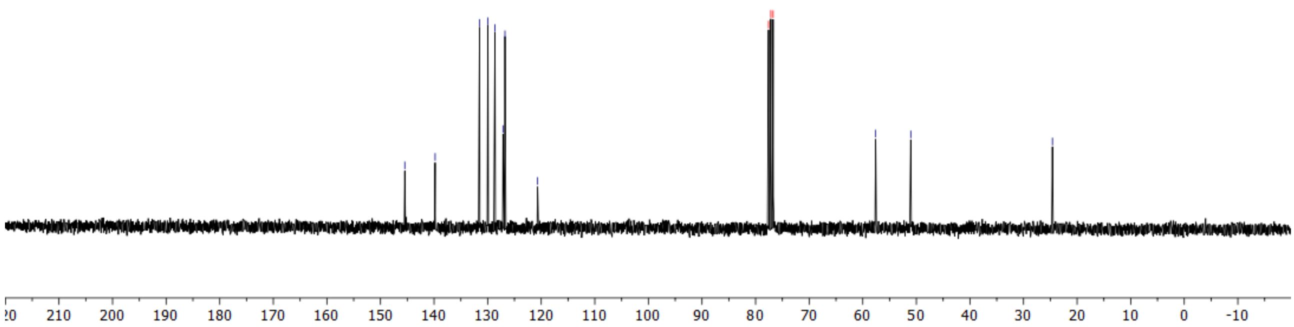
211208.f362.10.fid
Xinmin Li 120801
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 2



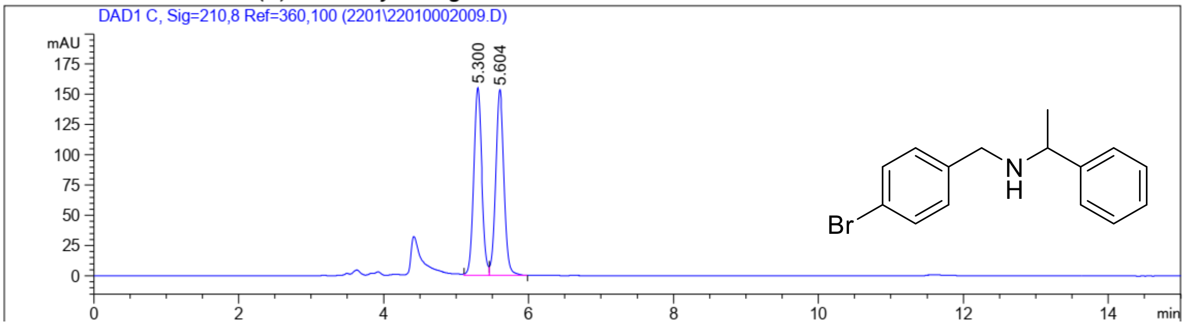


211208.f362.11.fid
Xinmin Li 120801
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 2





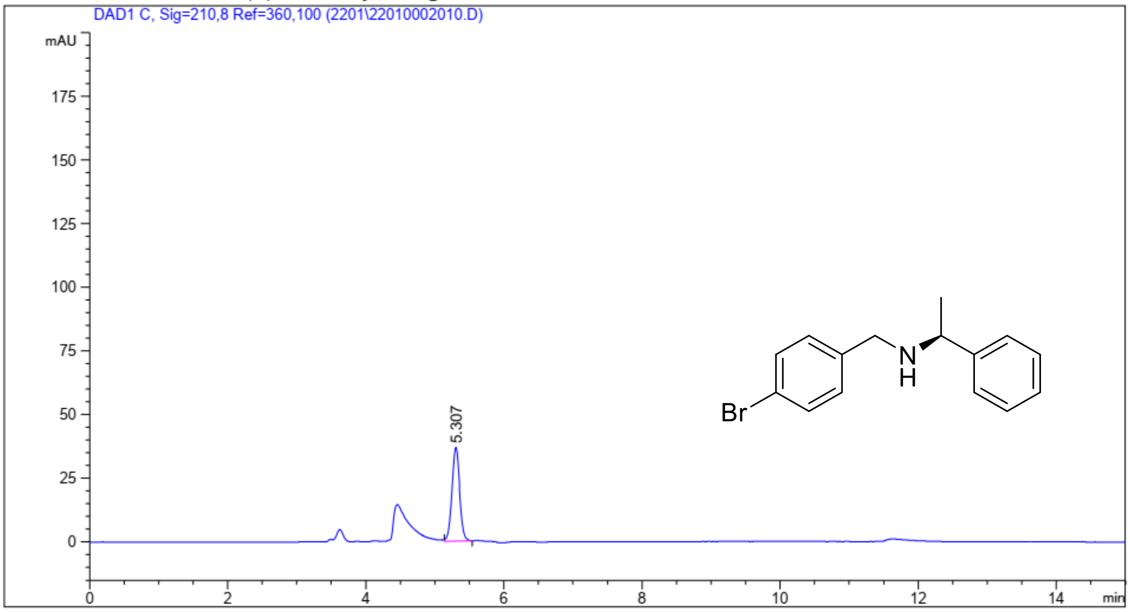
```
Acq. Operator   : Analytik                        Seq. Line :   4
Acq. Instrument : LC5                              Location : Vial 71
Injection Date  : 1/20/2022 7:16:40 PM                  Inj :   2
                                                 Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\AMYLOSE_2.M
Last changed    : 1/20/2022 3:48:53 PM by Analytik
Analysis Method : C:\CHEM32\1\METHODS\AMYLOSE_2   FL0.3.M
Last changed    : 1/21/2022 11:55:18 AM by Analytik
                  (modified after loading)
Method Info     : Amylose_2 , Hept./EtOH 98:2 , 0.3 ml-min

Additional Info : Peak(s) manually integrated
```



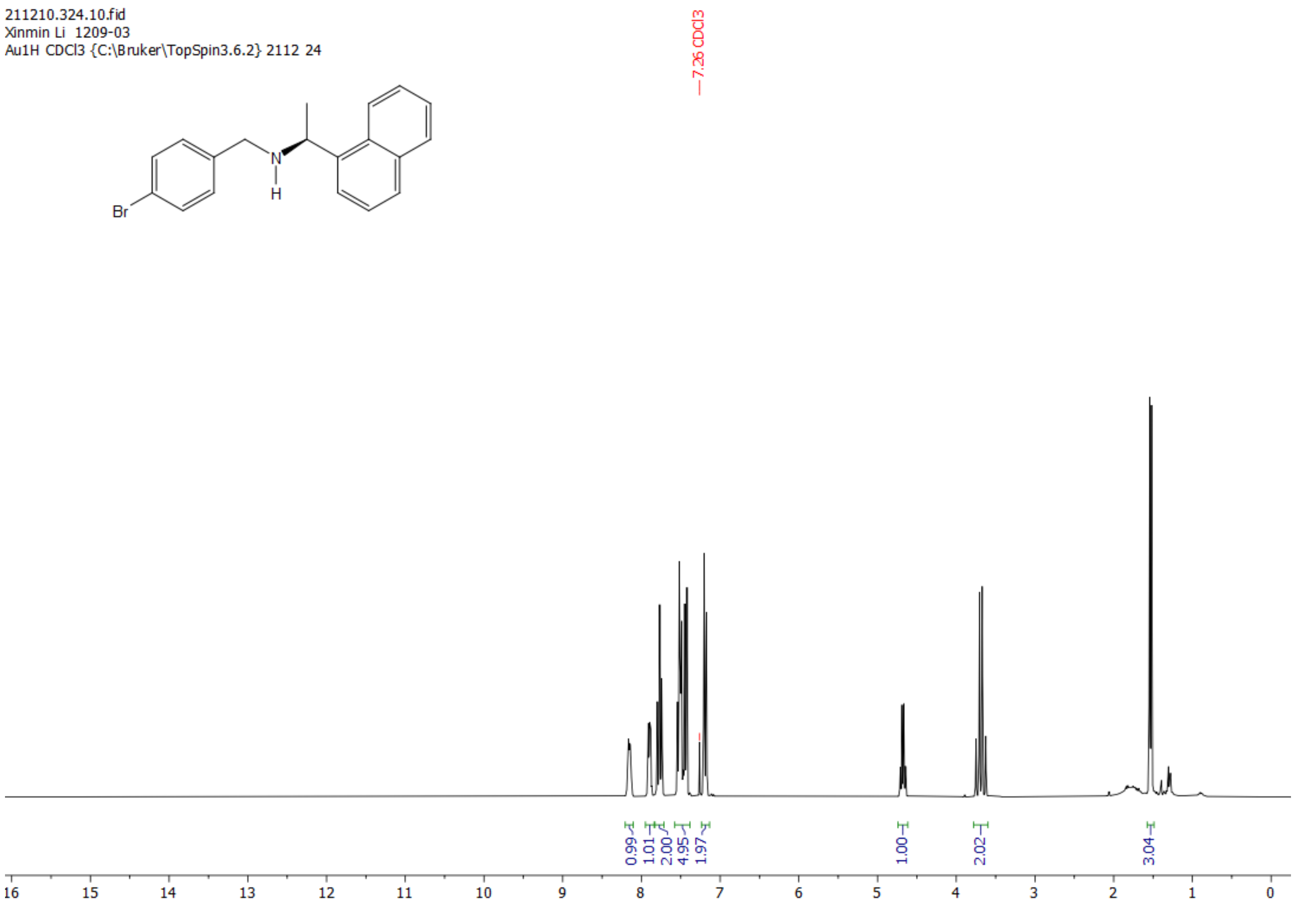
Signal 1: DAD1 C, Sig=210,8 Ref=360,100

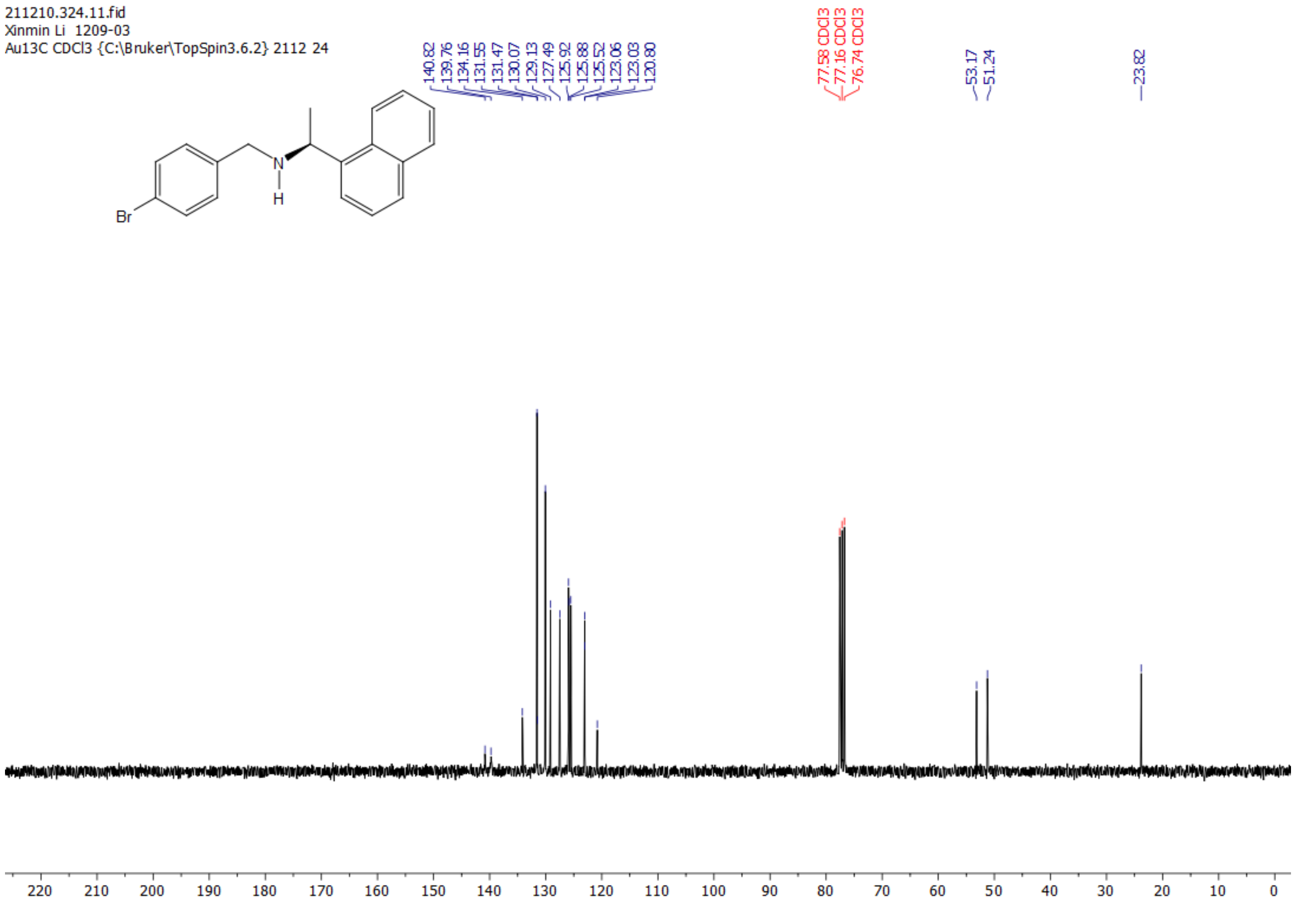
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.300	BV	0.1207	1207.48206	155.35339	49.4251
2	5.604	VB	0.1257	1235.57373	153.82600	50.5749

Additional Info : Peak(s) manually integrated

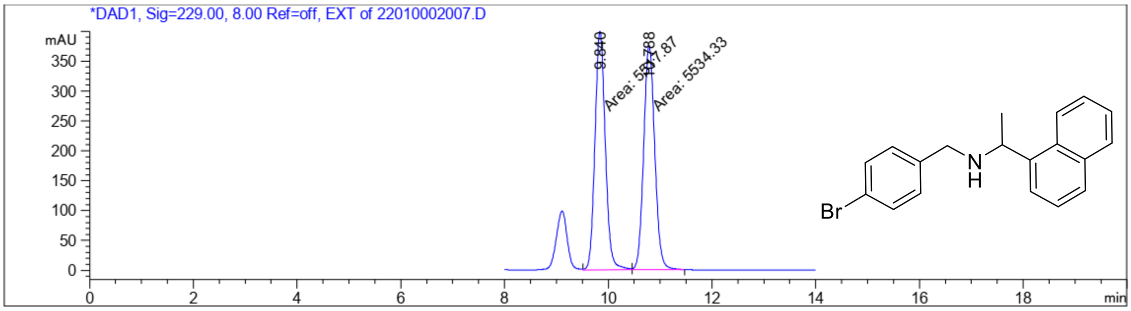


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.307	BB	0.1188	285.78754	36.73170	100.0000


211210.324.10.fid
Xinmin Li 1209-03
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 24
7.26 CDCl3
Br
N
H
0.99
1.01
2.00
4.95
1.97
1.00
2.02
3.04
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0

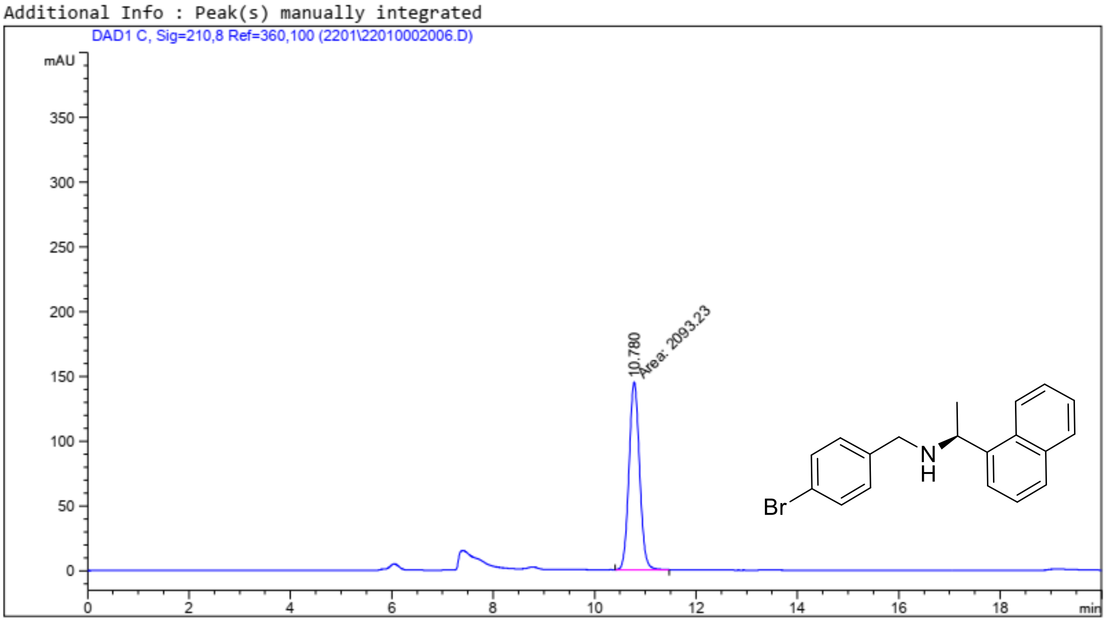

211210.324.11.fid
Xinmin Li 1209-03
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 24
140.82
139.76
134.16
131.55
131.47
130.07
129.13
127.49
125.92
125.88
125.52
123.06
123.03
120.80
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
53.17
51.24
23.82
Br
N
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

```
-----------------------------------------------------------------------
Acq. Operator   : Analytik                        Seq. Line :   3
Acq. Instrument : LC5                              Location : Vial 61
Injection Date  : 1/20/2022 5:54:25 PM                  Inj :   1
                                                 Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\AMYLOSE_2   FL0.3.M
Last changed    : 1/20/2022 3:49:38 PM by Analytik
Analysis Method : C:\CHEM32\1\METHODS\AMYLOSE_2   FL0.3.M
Last changed    : 1/21/2022 12:02:34 PM by Analytik
                  (modified after loading)
Method Info     : Amylose_2 , Hept./EtOH 98:2 , 0.3 ml-min

Additional Info : Peak(s) manually integrated
```

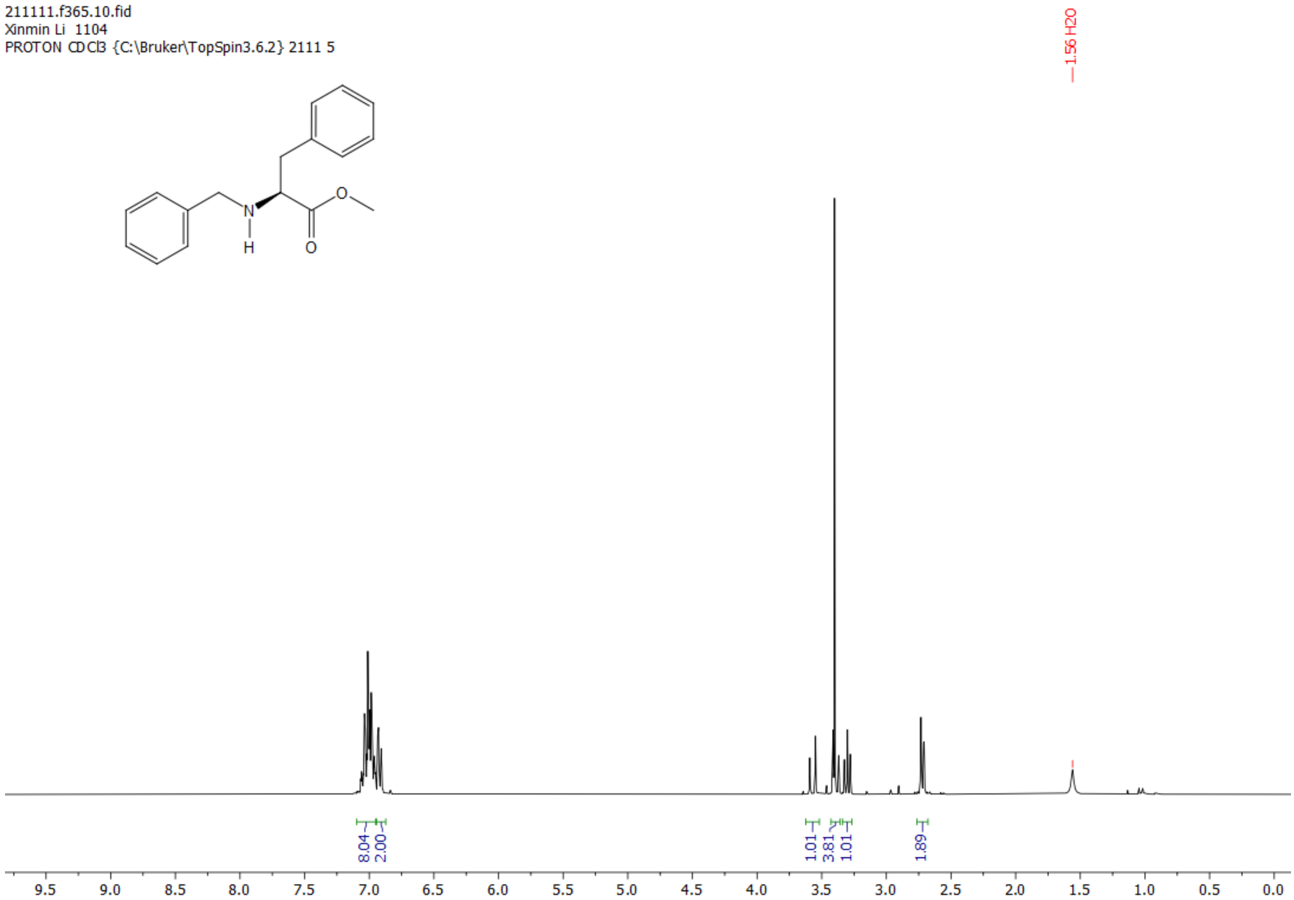


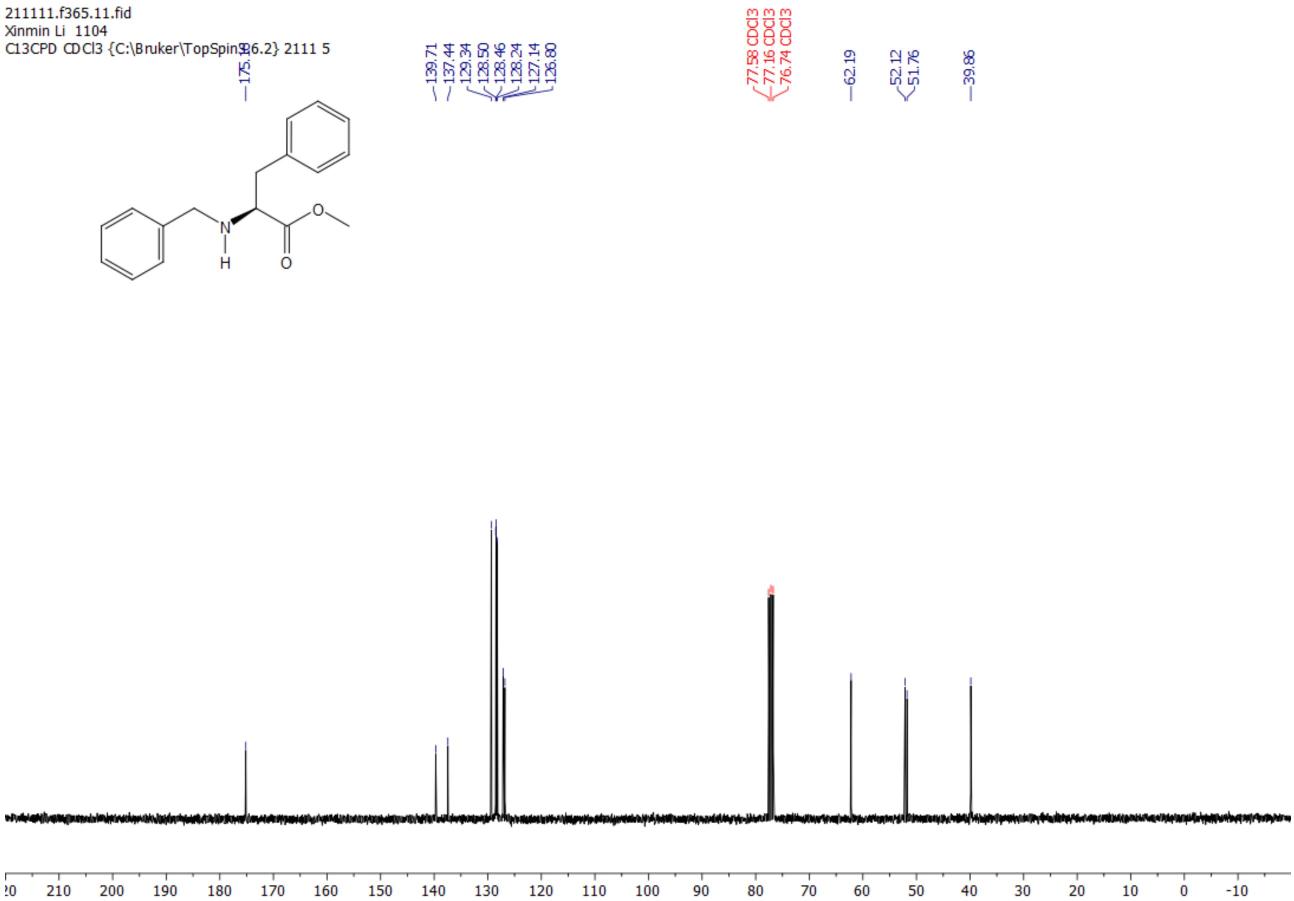
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.840	VV	0.2109	5130.98633	376.68033	49.8735
2	10.788	VB	0.2264	5157.00488	353.04102	50.1265

Additional Info : Peak(s) manually integrated



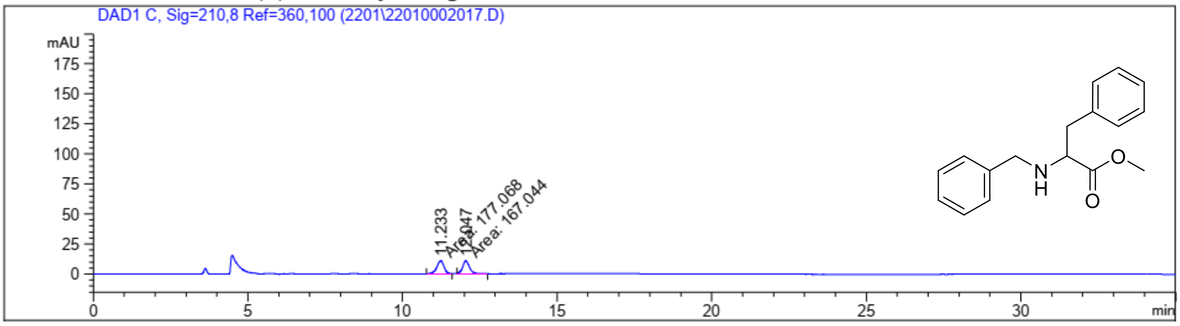
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.780	MM	0.2409	2093.22656	144.82303	100.0000


211111.f365.10.fid
Xinmin Li 1104
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 5
1.56 H2O
N
H
O
O
8.04
2.00
1.01
3.81
1.01
1.89
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


211111.f365.11.fid
Xinmin Li 1104
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 5
175.10
139.71
137.44
129.34
128.50
128.46
128.24
127.14
126.80
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
62.19
52.12
51.76
39.86
N
H
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

===

```
Acq. Operator    : Analytik                        Seq. Line :   8
Acq. Instrument  : LC5                              Location : Vial 51
Injection Date   : 1/21/2022 12:45:23 AM                 Inj :   2
                                                  Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\AMYLOSE_2.M
Last changed     : 1/20/2022 3:48:53 PM by Analytik
Analysis Method  : C:\CHEM32\1\METHODS\AMYLOSE_2.M
Last changed     : 1/25/2022 10:56:54 AM by Analytik
                   (modified after loading)
Method Info      : Amylose_2 , Hept./EtOH 98:2 , 0.3 ml/min

Additional Info  : Peak(s) manually integrated
```

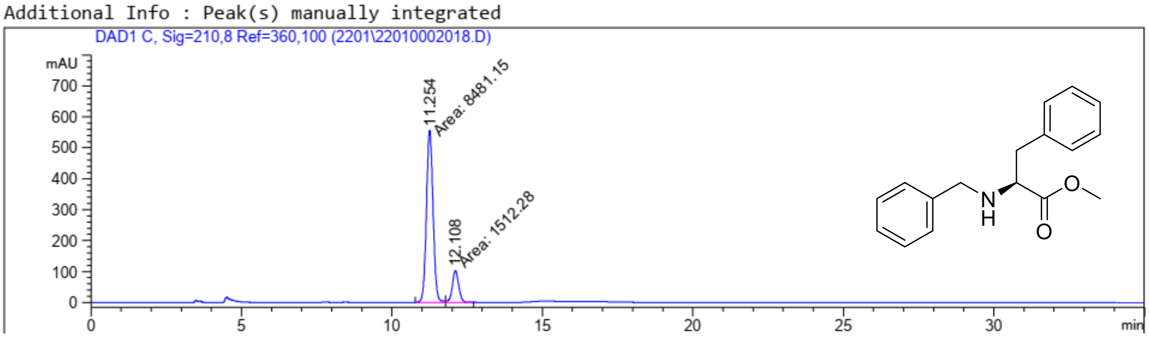


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.233	MF	0.2707	177.06828	10.90124	51.4566
2	12.047	FM	0.2583	167.04390	10.77900	48.5434

===

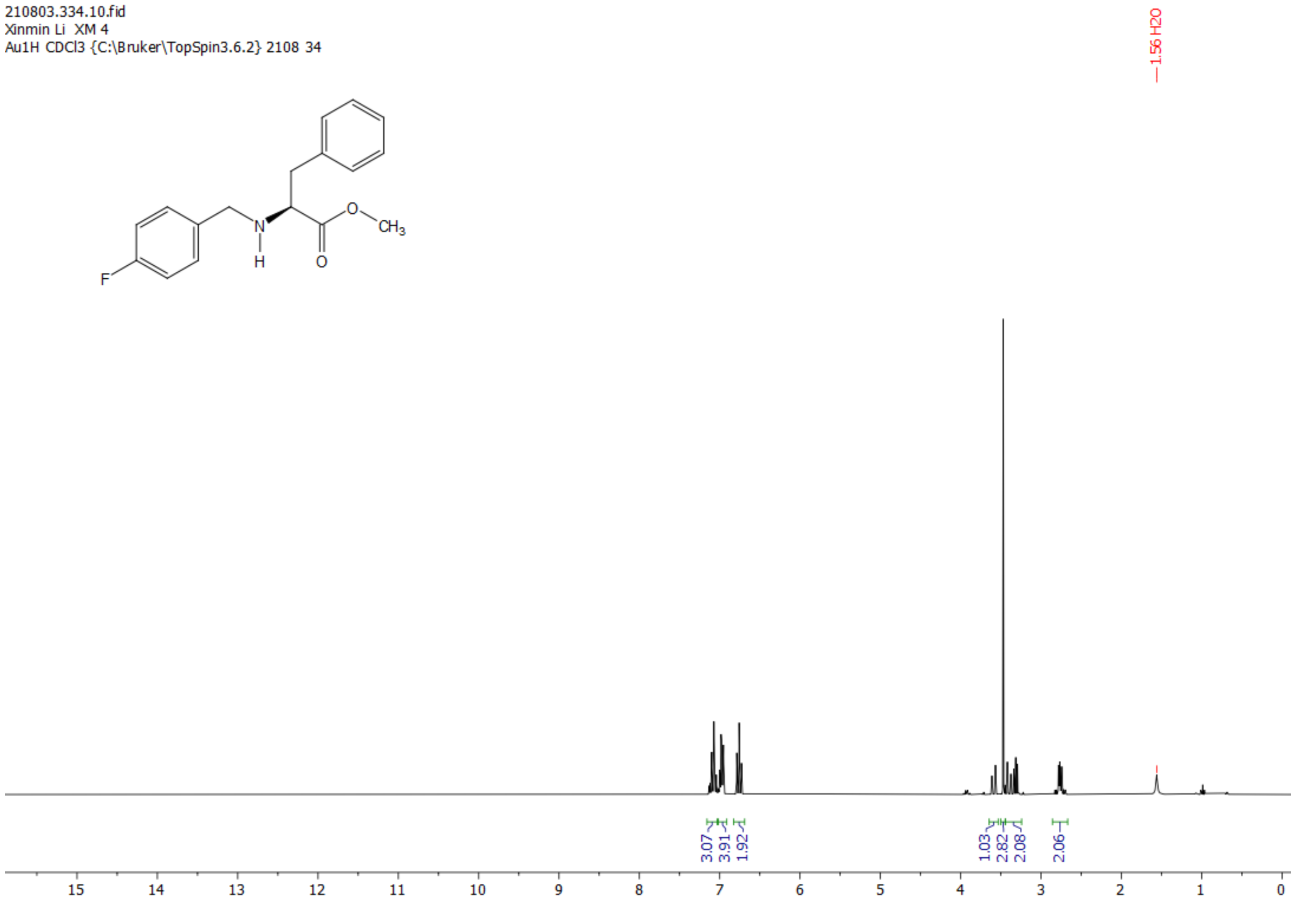
```
Acq. Operator    : Analytik                        Seq. Line :   9
Acq. Instrument  : LC5                              Location : Vial 52
Injection Date   : 1/21/2022 1:26:27 AM                  Inj :   1
                                                  Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\AMYLOSE_2.M
Last changed     : 1/20/2022 3:48:53 PM by Analytik
Analysis Method  : C:\CHEM32\1\METHODS\AMYLOSE_2.M
Last changed     : 1/25/2022 10:54:08 AM by Analytik
                   (modified after loading)
Method Info      : Amylose_2 , Hept./EtOH 98:2 , 0.3 ml/min

Additional Info  : Peak(s) manually integrated
```



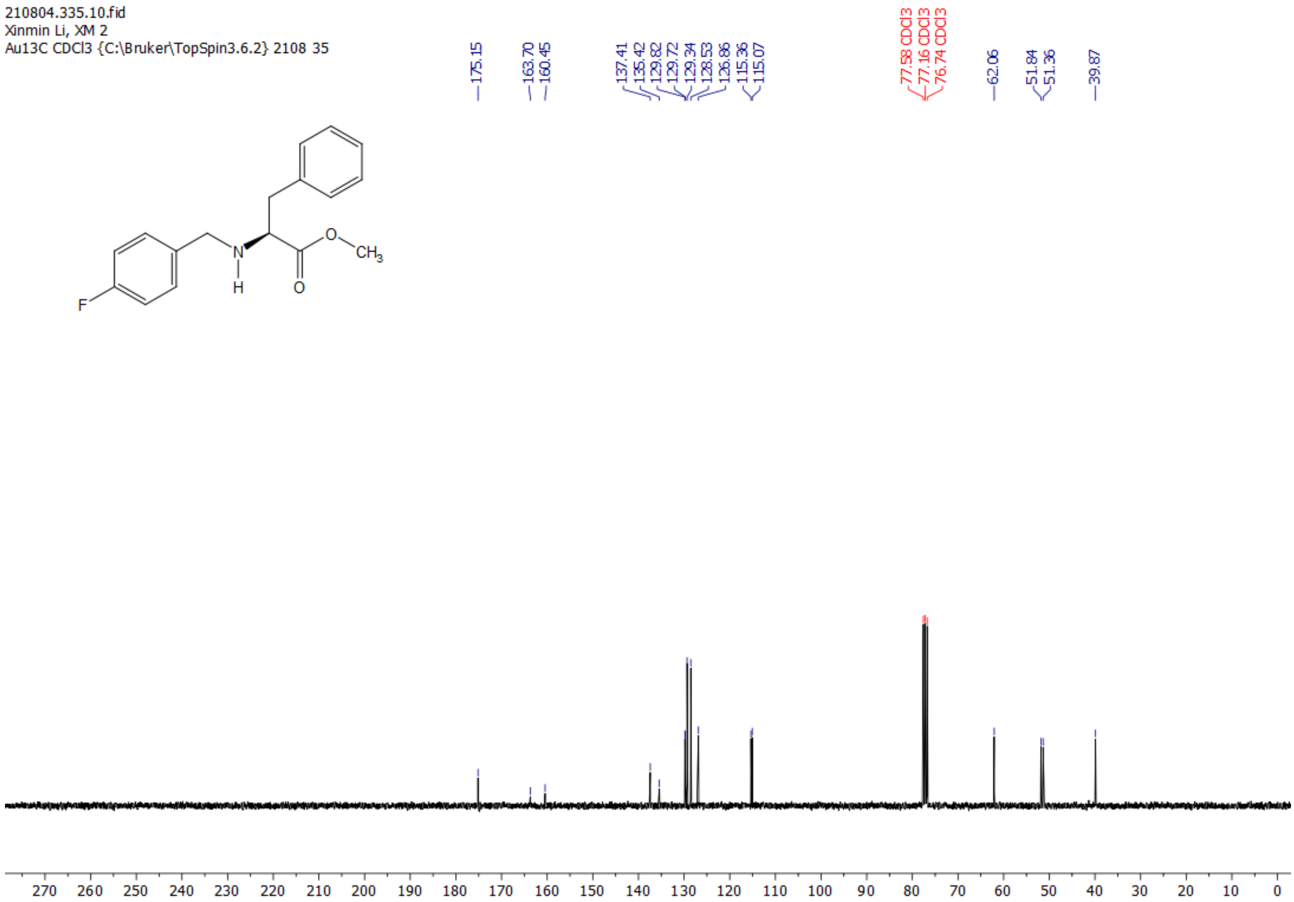
Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.254	MF	0.2542	8481.15234	556.14001	84.8673
2	12.108	FM	0.2464	1512.27881	102.27789	15.1327

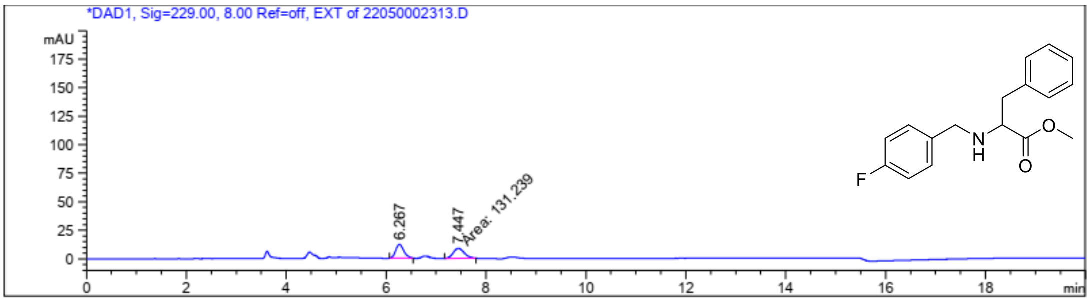
210803.334.10.fid
Xinmin Li XM 4
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 34



210804.335.10.fid
Xinmin Li, XM 2
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 35



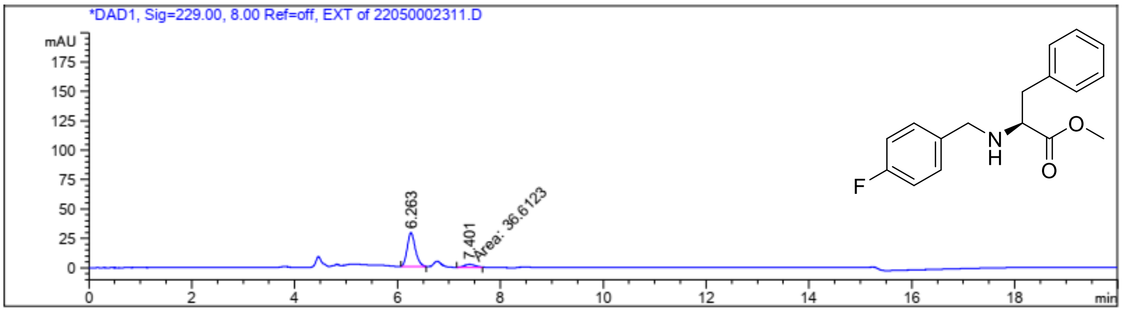
```
====================================================================
Acq. Operator   : Analytik                        Seq. Line :   4
Acq. Instrument : LC5                              Location : Vial 3
Injection Date  : 5/23/2022 5:19:49 PM                  Inj :   1
                                                 Inj Volume : 0.8 µl
Acq. Method     : C:\CHEM32\1\METHODS\AS-H.M
Last changed    : 5/23/2022 3:46:18 PM by Analytik
Analysis Method : C:\CHEM32\1\METHODS\AS-H.M
Last changed    : 5/24/2022 7:56:15 AM by Analytik
                  (modified after loading)
Method Info     : AS-H, Hept./EtOH 99:1,  0.5ml/min

Additional Info : Peak(s) manually integrated
```



Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.267	BB	0.1698	135.48434	12.17911	50.7959
2	7.447	MM	0.2511	131.23851	8.71192	49.2041

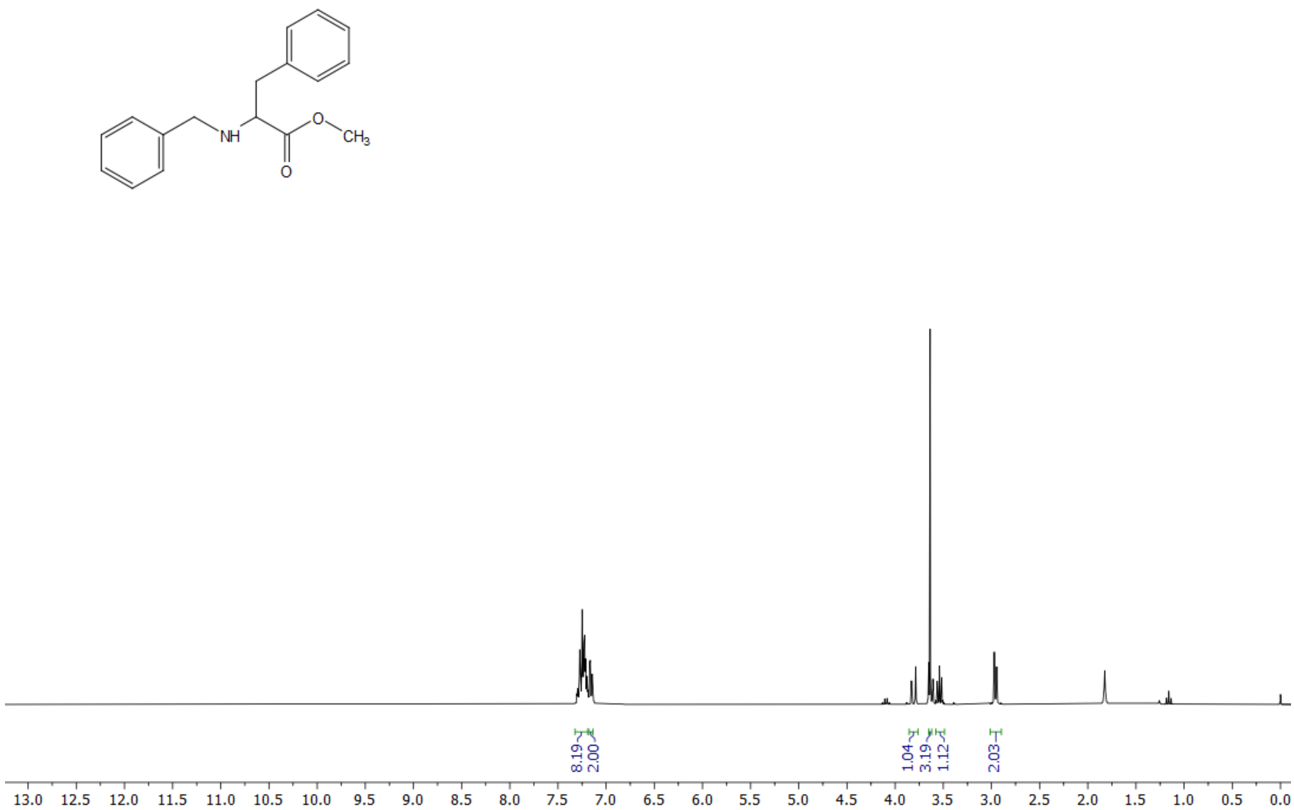
```
====================================================================
Acq. Operator   : Analytik                        Seq. Line :   2
Acq. Instrument : LC5                              Location : Vial 11
Injection Date  : 5/23/2022 4:17:40 PM                  Inj :   1
                                                 Inj Volume : 0.8 µl
Acq. Method     : C:\CHEM32\1\METHODS\AS-H.M
Last changed    : 5/23/2022 3:46:18 PM by Analytik
Analysis Method : C:\CHEM32\1\METHODS\AS-H.M
Last changed    : 5/24/2022 7:56:15 AM by Analytik
                  (modified after loading)
Method Info     : AS-H, Hept./EtOH 99:1,  0.5ml/min

Additional Info : Peak(s) manually integrated
```



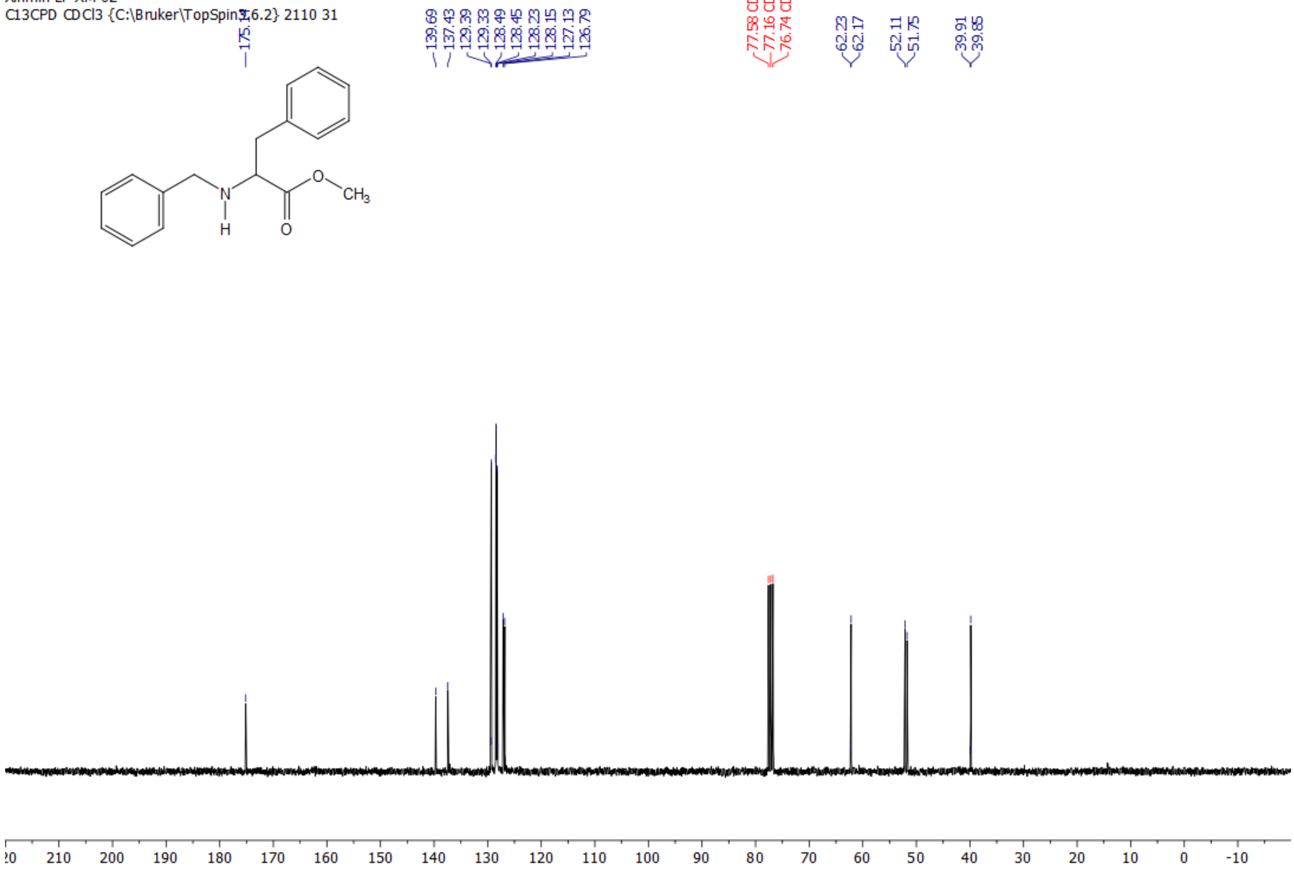
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.263	BB	0.1675	313.19147	28.65039	89.5335
2	7.401	MM	0.2417	36.61227	2.52463	10.4665

211015.f331.10.fid
Xinmin Li XM 02
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 31

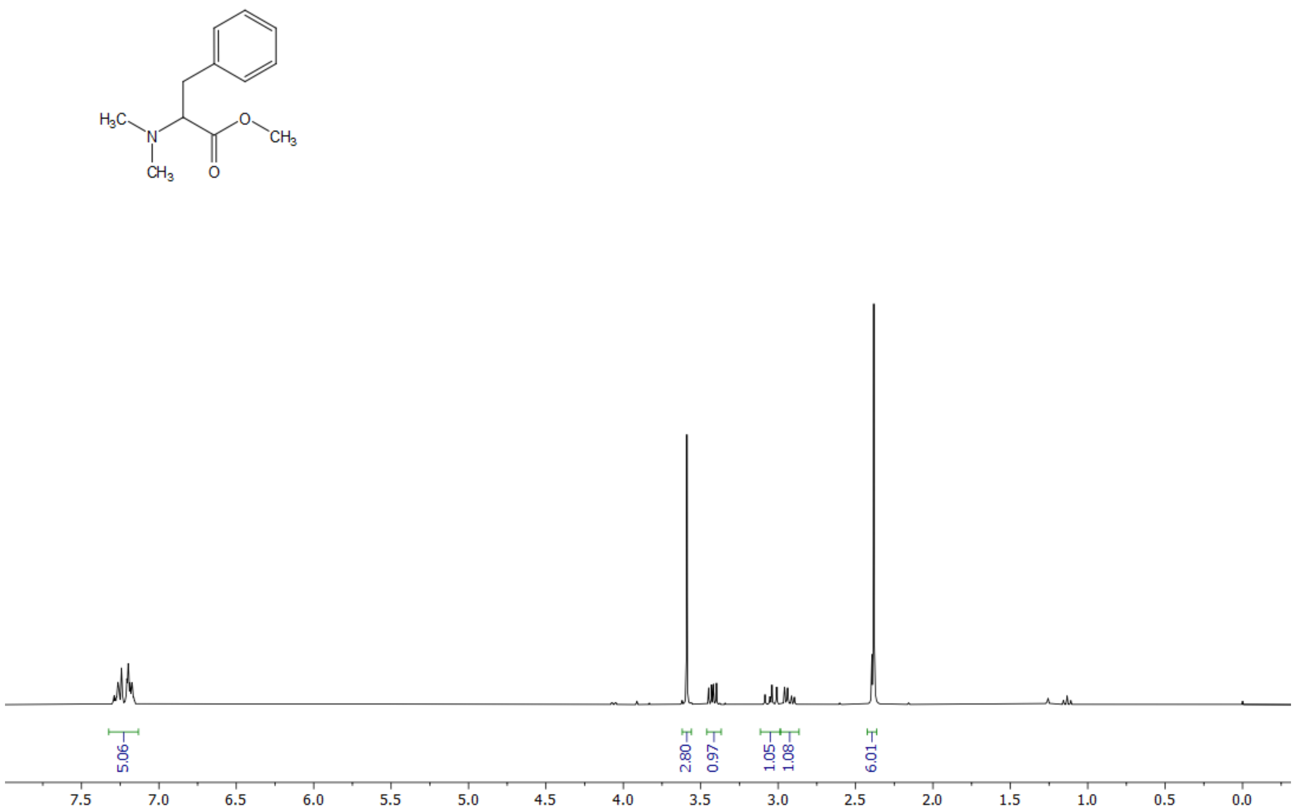


211015.f331.11.fid
Xinmin Li XM 02
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 31

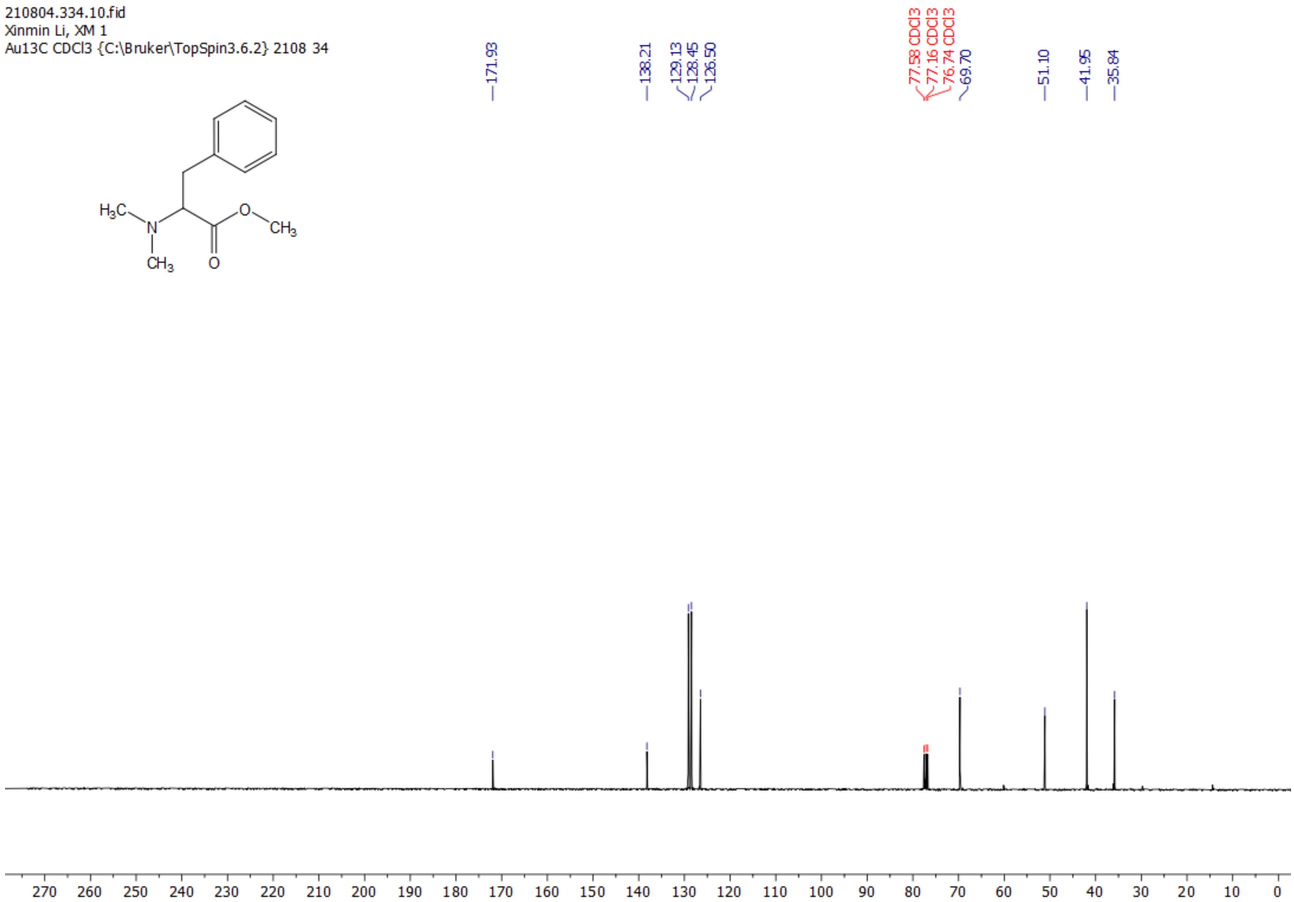


FF

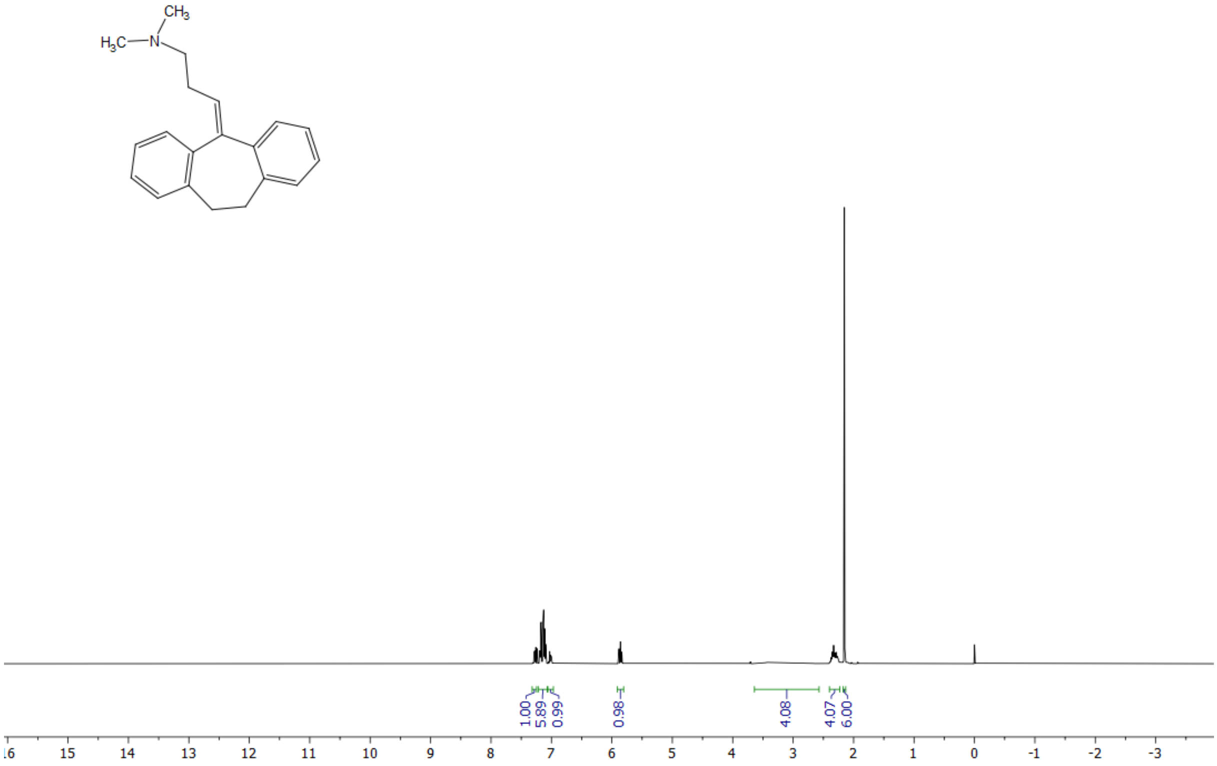
210803.333.10.fid
Xinmin Li XM 3
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 33



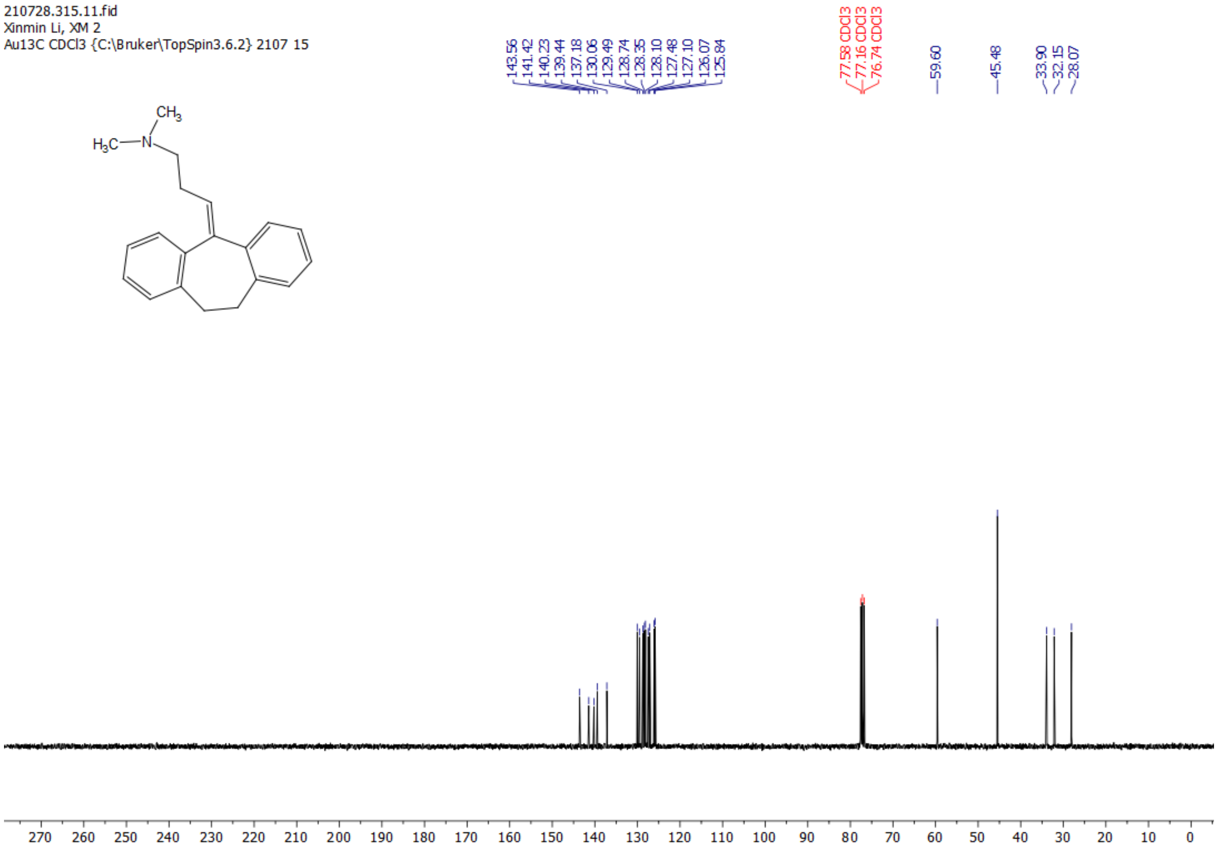
210804.334.10.fid
Xinmin Li, XM 1
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 34



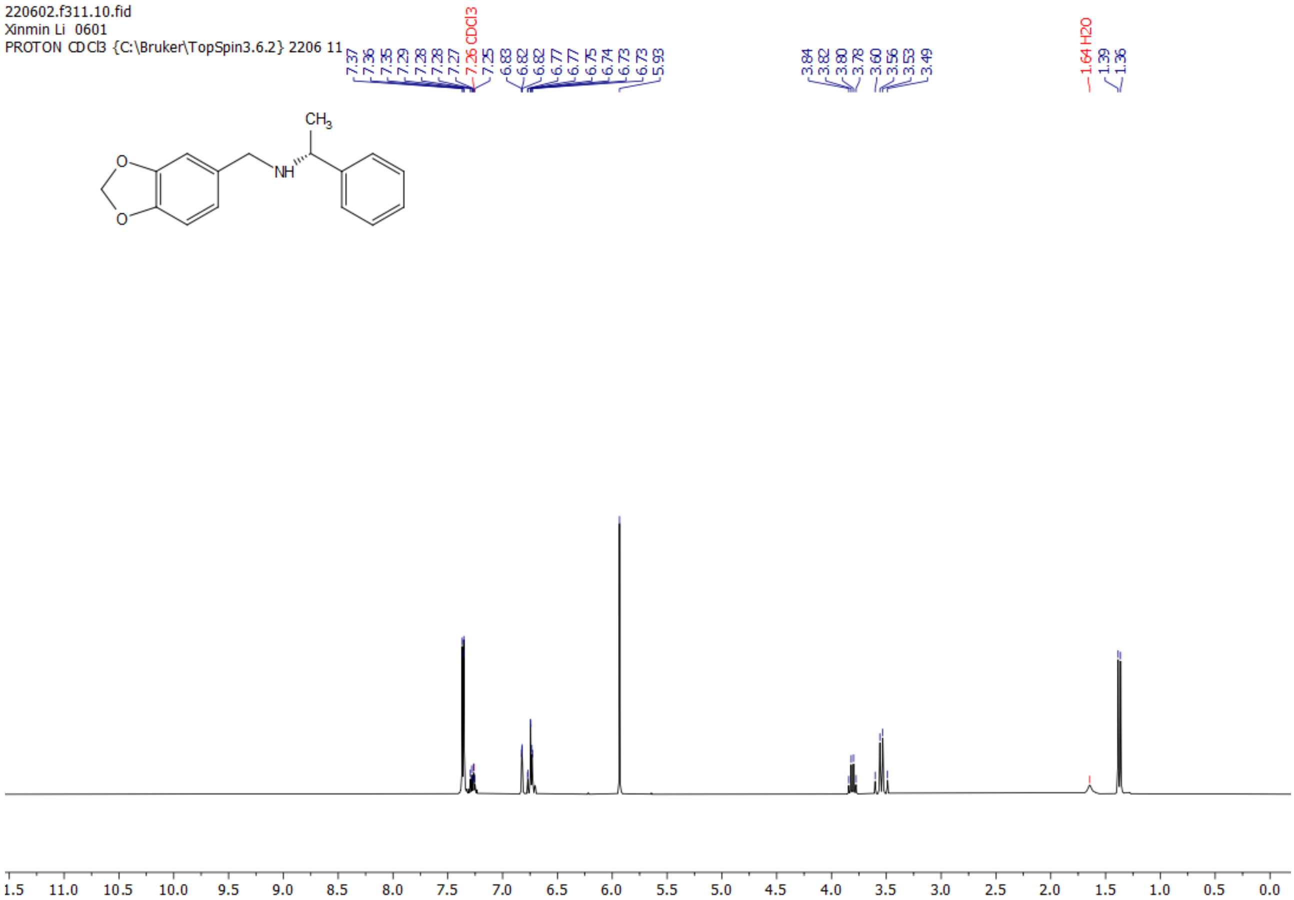
210728.315.10.fid
Xinmin Li, XM 2
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 15



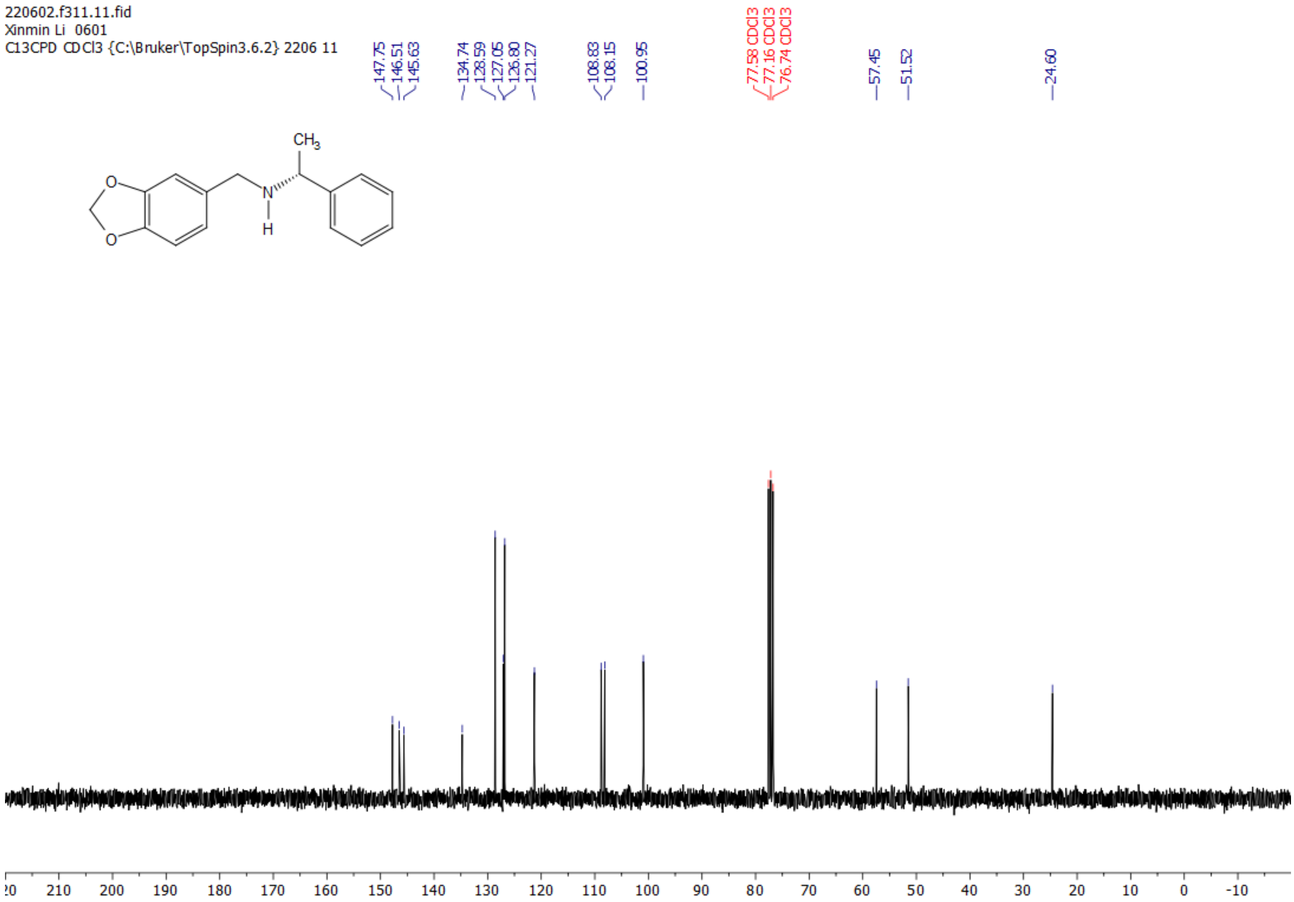
210728.315.11.fid
Xinmin Li, XM 2
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 15



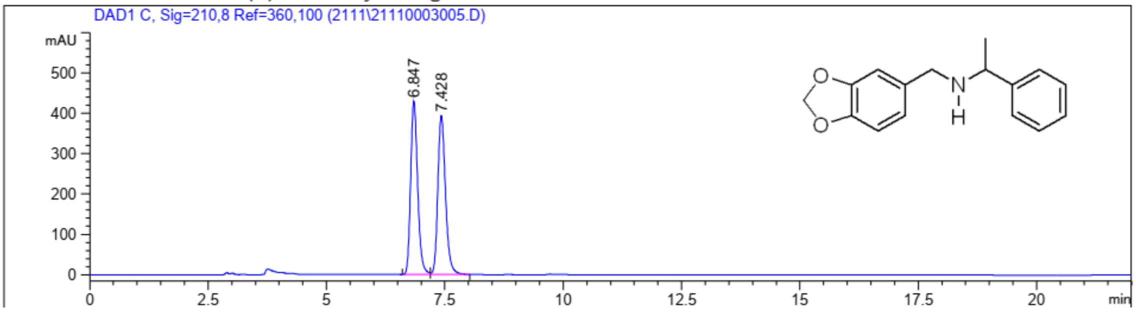
220602.f311.10.fid
Xinmin Li 0601
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 11



220602.f311.11.fid
Xinmin Li 0601
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 11

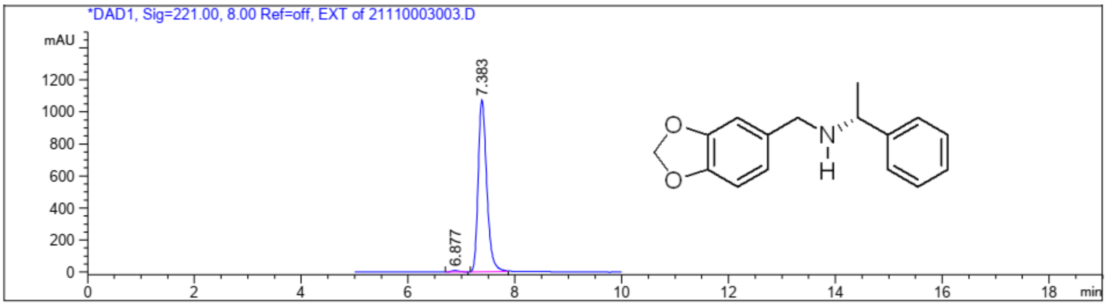


```
Acq. Operator   : Analytik                          Seq. Line :   4
Acq. Instrument : LC5                                Location : Vial 1
Injection Date  : 11/30/2021 12:32:58 PM                  Inj :   1
                                                   Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 11/30/2021 1:06:19 PM by Analytik
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 12/1/2021 11:08:07 AM by Analytik
                  (modified after loading)
Method Info     : Amylose2 , Hept./EtOH 98:2 , 0.6ml/min

Additional Info : Peak(s) manually integrated
```



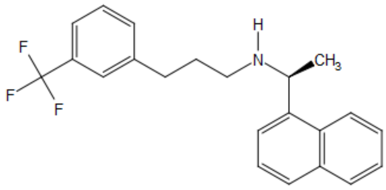
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.847	BV	0.1582	4415.57666	428.74023	49.7592
2	7.428	VB	0.1743	4458.31836	393.17505	50.2408

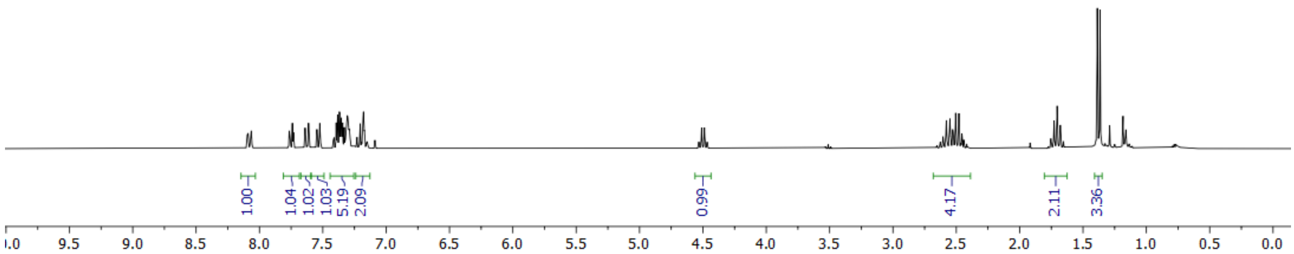
```
Acq. Operator   : Analytik                          Seq. Line :   2
Acq. Instrument : LC5                                Location : Vial 11
Injection Date  : 11/30/2021 11:20:50 AM                  Inj :   1
                                                   Inj Volume : 1.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 11/30/2021 10:44:28 AM by Analytik
Analysis Method : C:\CHEM32\1\METHODS\AMYLOSE2.M
Last changed    : 12/1/2021 11:20:30 AM by Analytik
                  (modified after loading)
Method Info     : Amylose2 , Hept./EtOH 98:2 , 0.6ml/min
```



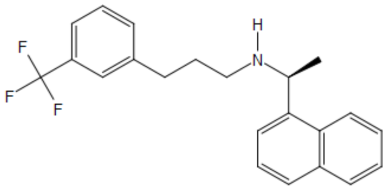
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.877	BV	0.1555	277.07733	27.04879	0.9377
2	7.383	VB	0.2081	2.92704e4	2244.93018	99.0623

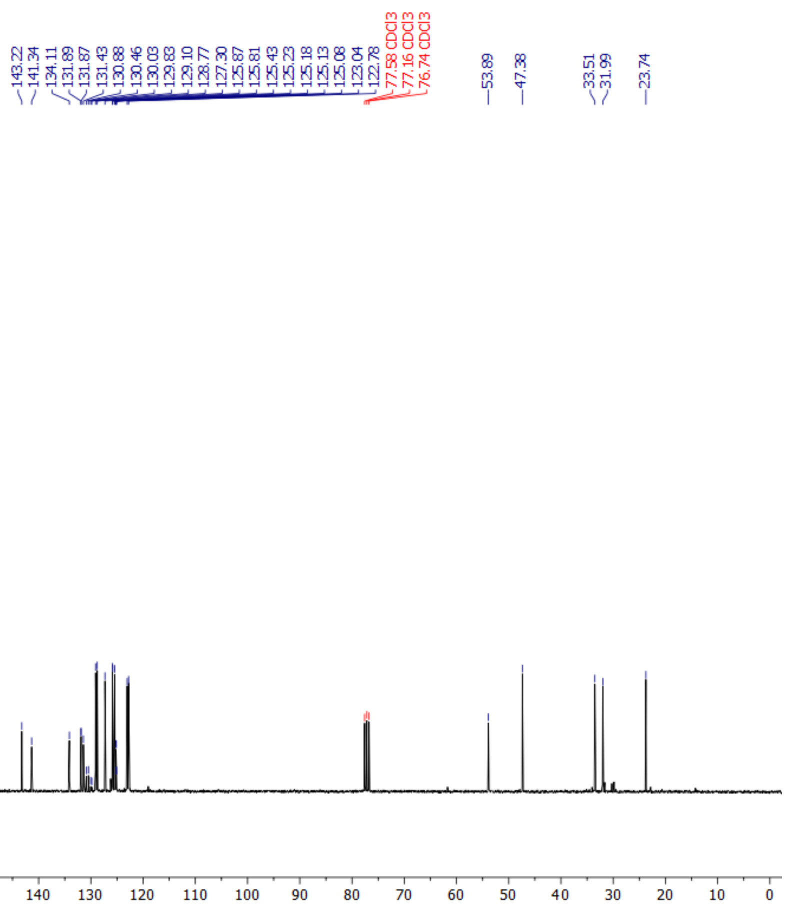
211210.325.10.fid
Xinmin Li 1209-04
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 25





211210.325.11.fid
Xinmin Li 1209-04
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 25

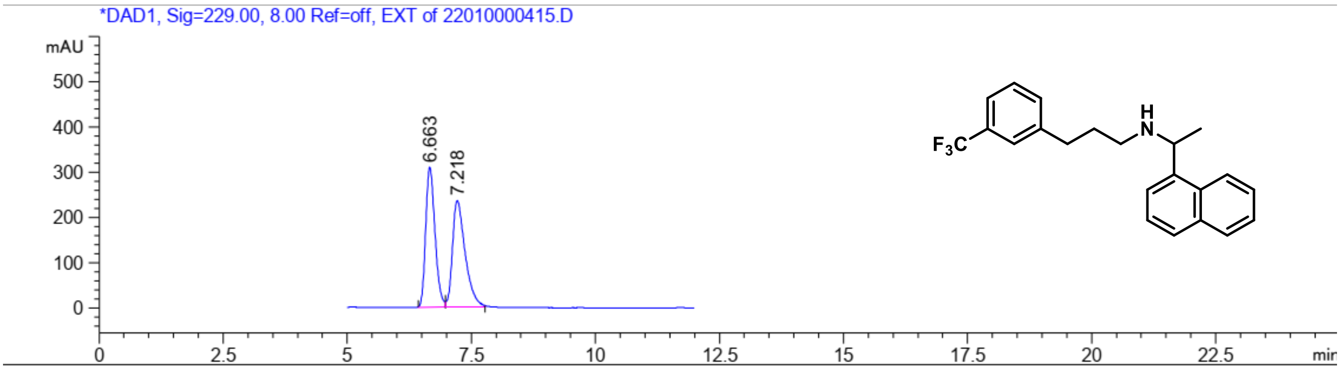




Acq. Operator : Analytik   Seq. Line : 4
Acq. Instrument : LC5   Location : Vial 2
Injection Date : 1/4/2022 5:16:17 PM   Inj : 2
Inj Volume : 1.0 µl

Acq. Method : C:\CHEM32\1\METHODS\CELLULOSE1.M
Last changed : 1/4/2022 3:58:07 PM by Analytik
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\CELLULOSE1.M
Last changed : 1/4/2022 3:44:53 PM by Analytik
(modified after loading)
Method Info : Cellulose1 , Hept./EtOH 98:2 , 0.6ml/min

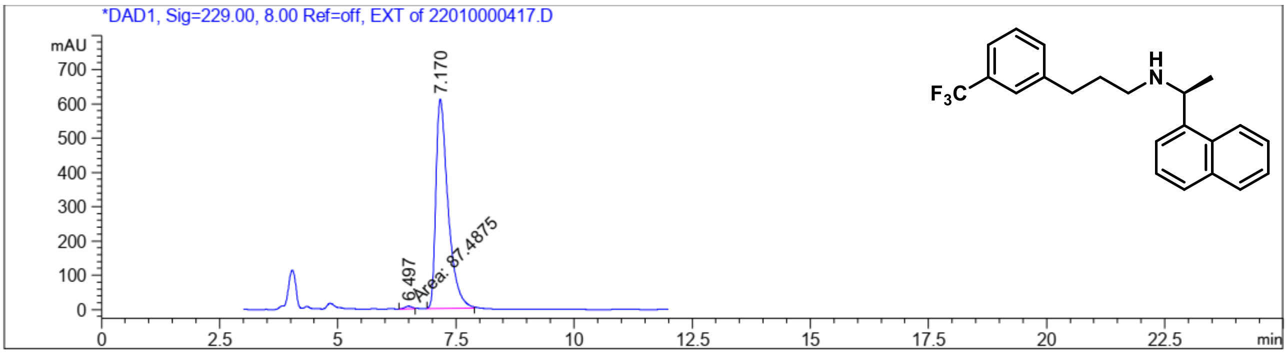
Additional Info : Peak(s) manually integrated



Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.663	BV	0.1983	4889.54883	379.48343	49.2292
2	7.218	VB	0.2648	5042.67383	284.53693	50.7708

Totals : 9932.22266 664.02036



Signal 2: DAD1, Sig=229.00, 8.00 Ref=off, EXT
Signal has been modified after loading from rawdata file!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.497	MF	0.1806	87.48751	8.07397	0.7925
2	7.170	BB	0.2690	1.09526e4	611.28619	99.2075

Totals : 1.10400e4 619.36016