Iron Catalyzed Transfer Hydrogenation of Imine Assisted by Iron-based Lewis Acid

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Supporting Information

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I. General information.

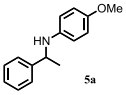
^{1}H and **^{13}C NMR** spectra were recorded on a Bruker AFC 300 (300 MHz) or AMX500 (500 MHz) spectrometer. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: ^{1}H (chloroform δ 7.26), ^{13}C (chloroform δ 77.00). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constants (Hz) and integration. For thin layer chromatography (**TLC**), Merck pre-coated TLC plates (Merck 60 F254) were used, and compounds were visualized with a UV light at 254nm.

II. General procedure for transfer hydrogenation of imine

In a nitrogen-filled glovebox, a 4 mL oven-dried sample vial was charged with imine (0.4 mmol), **2** (10.2 mg, 0.02 mmol), $Fe(acac)_3$ (14.1 mg, 0.04 mmol), 1.0 mL of isopropanol, and then sealed with a screw cap. The reaction mixture was taken outside the glovebox and heated to 110 ^{o}C for 48 h, after which the mixture was cooled to room temperature. All the volatiles were removed under vacuum. Purification by flash chromatography (Hexane : EA = 20 : 1) afforded the target products in pure form.

III. Characterization of compounds

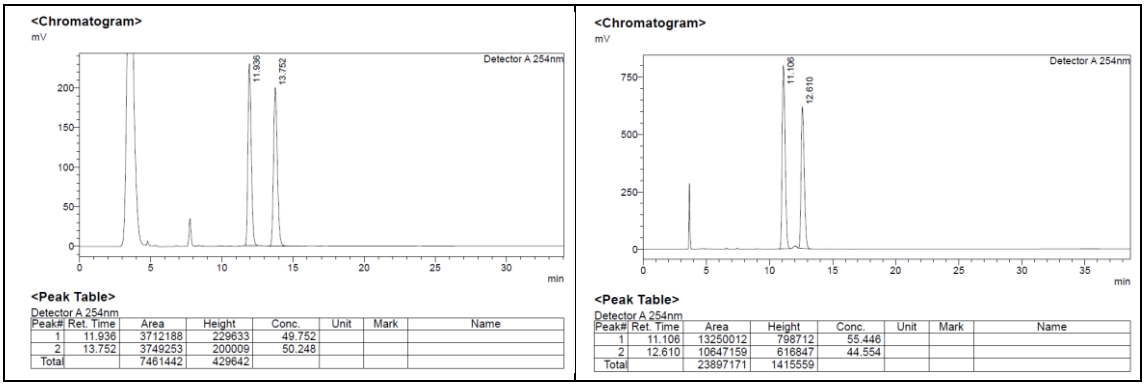
4-Methoxy-*N*-(1-phenylethyl)aniline[1] Yellow oil, 88.1 mg, 97% yield. **^{1}H NMR**



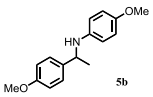
5a

(500 MHz, $CDCl_3$) δ 1.53 (d, *J* = 6.7 Hz, 3H), 3.72 (s, 3H), 4.45 (q, *J* = 6.7 Hz, 1H), 6.51 (d, *J* = 8.9 Hz, 2H), 6.73 (d, *J* = 8.9 Hz, 2H), 7.26 (t, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.40 (d, *J* = 7.2 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 25.1, 54.5, 55.8, 114.8, 114.9, 126.0, 126.9, 128.7, 141.5, 145.5, 152.1.

HPLC spectra of 5a



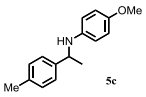
4-Methoxy-*N*-(1-(4-methoxyphenyl)ethyl)aniline[1] Yellow oil, 100.7 mg, 98% yield.



5b

^{1}H NMR (500 MHz, $CDCl_3$) δ 1.49 (d, *J* = 6.7 Hz, 3H), 3.71 (s, 3H), 3.80 (s, 3H), 4.39 (q, *J* = 6.7 Hz, 1H), 6.50 (d, *J* = 8.9, 2H), 6.71 (d, *J* = 8.9, 2H), 6.87 (d, *J* = 8.7, 2H), 7.29 (d, *J* = 8.7, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 25.1, 53.8, 55.3, 55.8, 114.1, 114.8, 114.9, 127.1, 137.6, 141.6, 152.1, 158.6.

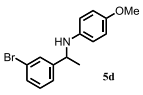
4-Methoxy-*N*-(1-*p*-tolylethyl)aniline[1] Yellow oil, 95.4 mg, 99% yield. **^{1}H NMR**



5c

(500 MHz, $CDCl_3$) δ 1.53 (d, *J* = 6.8 Hz, 3H), 2.37 (s, 3H), 3.73 (s, 3H), 4.43 (q, *J* = 6.7 Hz, 1H), 6.53 (d, *J* = 8.9 Hz, 2H), 6.74 (d, *J* = 8.9 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 21.2, 25.2, 54.1, 55.8, 114.7, 114.9, 125.9, 129.4, 136.4, 141.7, 142.5, 152.0.

4-Methoxy-*N*-(1-*m*-bromophenylethyl)aniline[1] Yellow oil, 53.7 mg, 44% yield. **^{1}H**

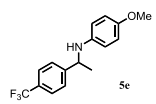


5d

NMR (500 MHz, $CDCl_3$) δ 1.49 (d, *J* = 6.7 Hz, 3H), 3.71 (s, 3H), 4.37 (q, *J* = 6.6 Hz, 1H), 6.46 (d, *J* = 8.6 Hz, 2H), 6.70 (d, *J* = 8.9 Hz, 2H), 7.18 (t, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.53 (s, 1H). **^{13}C**

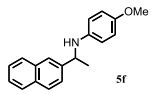
NMR (125 MHz, $CDCl_3$) δ 25.0, 54.1, 55.7, 114.7, 114.8, 122.8, 124.6, 129.0, 130.0, 130.2, 140.9, 148.0, 152.2.

4-Methoxy-*N*-{1-[4-(trifluoromethyl)phenyl]ethyl}aniline[1] Yellow oil, 64.9 mg, 44% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.51 (d, *J* = 6.8 Hz, 3H), 3.70 (s, 3H), 4.46 (q, *J* = 6.7 Hz, 1H), 6.43 (d, *J* = 8.9, 2H), 6.70 (d, *J* = 8.9, 2H), 7.49 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 25.1, 54.4, 55.8, 114.9, 115.0, 124.4 (q, J_{CF} = 270.2 Hz), 125.8(q, J_{CF} = 3.7 Hz), 126.4, 129.3 (q, J_{CF} = 32.1 Hz), 140.9, 149.7, 152.5. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -62.29 (3F).



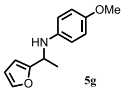
5e

4-Methoxy-*N*-(1-(naphthalen-2-yl)ethyl)aniline[1] Yellow oil, 109.7 mg, 99% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.60 (d, *J* = 6.8 Hz, 3H), 3.70 (s, 3H), 4.60 (q, *J* = 6.7 Hz, 1H), 6.56 (d, *J* = 8.9, 2H), 6.70 (d, *J* = 8.9, 2H), 7.44-7.54 (m, 3H), 7.81-7.85 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 25.1, 54.8, 55.8, 114.9, 115.0, 124.5, 124.6, 125.6, 126.1, 127.8, 128.0, 128.6, 132.9, 133.7, 141.4, 143.0, 152.2.



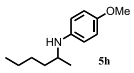
5f

***N*-[1-(furan-2-yl)ethyl]-N-(4-methoxyphenyl)amine**[2] Yellow oil, 81.6 mg, 94% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.54 (d, *J* = 6.7 Hz, 3H), 3.74 (s, 3H), 4.56 (q, *J* = 6.7 Hz, 1H), 6.15 (d, *J* = 3.2 Hz, 1H), 6.28-6.29 (m, 1H), 6.61 (d, *J* = 8.9 Hz, 2H), 6.77 (d, *J* = 8.9 Hz, 2H), 7.33-7.34 (m, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 21.0, 48.6, 55.8,, 105.2, 110.2, 114.9, 115.4, 141.2, 141.5, 152.6, 157.6.



5g

4-Methoxy-*N*-(hexan-2-yl)aniline[1] Colorless oil, 76.2 mg, 92% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.91 (t, *J* = 7.1 Hz, 3H), 1.16 (d, *J* = 6.3 Hz, 3H), 1.31-1.43 (m, 5H), 1.55-1.59 (m, 1H), 3.37 (m, 1H), 3.75 (s, 3H), 6.56 (d, *J* = 8.9, 2H), 6.79 (d, *J* = 8.9, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 14.2, 20.9, 22.9, 28.5, 37.0, 49.5, 55.8, 114.7, 115.0, 142.0, 151.8.



5h

4-Methoxy-*N*-(3-methylbutan-2-yl)aniline[3] Colorless oil, 74.9 mg, 97% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.91 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.9 Hz, 3H), 1.08 (d, *J* = 6.5 Hz, 3H), 1.84 (m, 1H), 3.26 (m, 1H), 3.75 (s, 3H), 6.56 (d, *J* = 8.9 Hz, 2H), 6.78 (d, *J* = 8.9 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 16.6, 17.4, 19.4, 32.2, 54.6, 55.9, 114.7, 115.0, 142.2, 151.8.

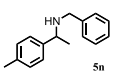
4-Methoxy-*N*-(1-*p*-tolylpropyl)aniline[4] Yellow oil, 101.0 mg, 99% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.4 Hz, 3H), 1.75-1.89 (m, 2H), 3.72 (s, 3H), 3.80 (s, 3H), 4.14 (t, *J* = 6.7 Hz, 1H), 6.52 (d, *J* = 8.9, 2H), 6.73 (d, *J* = 8.9, 2H), 6.89 (d, *J* = 8.7, 2H), 7.28 (d, *J* = 8.6, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 10.9, 31.8, 55.3, 55.8, 60.0, 113.9, 114.6, 114.8, 127.6, 136.2, 142.0, 151.9, 158.5.

***N*-(4-methoxyphenyl)-1, 1-diphenylmethylamine**[10] Yellow oil, 43.7 mg, 38% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 3.79 (s, 3H), 5.54 (s, 1H), 6.60 (d, *J* = 8.8, 2H), 6.81 (d, *J* = 8.8, 2H), 7.35 (t, *J* = 7.3, 2H), 7.42 (t, *J* = 7.9, 4H), 7.48 (d, *J* = 7.3, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 55.6, 63.8, 114.6, 114.7, 127.2, 127.4, 128.6, 141.6, 143.2, 152.1.

***N*-benzyl-α-methylbenzylamine**[5] Colorless oil, 81.9 mg, 97% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.44 (d, *J* = 6.6 Hz, 3H), 1.73 (br, 1H), 3.66 (d, *J* = 13.2 Hz, 1H), 3.73 (d, *J* = 13.2 Hz, 1H), 3.88 (q, *J* = 6.6 Hz, 1H), 7.30-7.44 (m, 10H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.6, 51.7, 57.6, 126.8, 126.9, 127.0, 128.2, 128.4, 128.6, 140.7, 145.6.

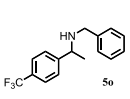
***N*-benzyl-N-(α-methyl-p-methoxybenzyl)amine**[6] Colorless oil, 92.5 mg, 96% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.36 (d, *J* = 6.6 Hz, 3H), 1.73 (br, 1H), 3.59 (d, *J* = 13.3 Hz, 1H), 3.66 (d, *J* = 13.3 Hz, 1H), 3.78 (q, *J* = 6.6 Hz, 1H), 3.82 (s, 3H), 6.90 (d, *J* = 8.7 Hz, 2H), 7.23-7.34 (m, 7H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.6, 51.7, 55.4, 56.9, 114.0, 127.0, 127.9, 128.3, 128.5, 137.7, 140.7, 158.7.

***N*-benzyl-1-(p-tolyl)ethanamine**[6] Colorless oil, 84.6 mg, 94% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.40 (d, *J* = 6.6 Hz, 3H), 1.70 (br, 1H), 2.39 (s, 3H), 3.62 (d, *J* = 13.1 Hz, 2H), 3.70 (d, *J* = 13.1 Hz, 2H), 3.83 (q, *J* = 6.6 Hz, 1H), 7.20-7.37 (m, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 21.2, 24.6, 51.7, 57.3, 126.7, 126.9, 128.3, 128.5, 129.3, 136.6, 140.8, 142.6.



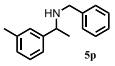
5n

***N*-benzyl-1-(4-(trifluoromethyl)phenyl)ethanamine**[6] Colorless oil, 79.6 mg, 67% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.37 (d, *J* = 6.7 Hz, 3H), 1.70 (br, 1H), 3.59 (d, *J* = 13.2 Hz, 1H), 3.65 (d, *J* = 13.2 Hz, 1H), 3.89 (q, *J* = 6.6 Hz, 1H), 7.24-7.34 (m, 5H), 7.50 (d, *J* = 8.2 Hz, 2H), 7.61 (d, *J* = 8.1 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.7, 51.8, 57.4, 124.4 (q, *J* = 270.3 Hz), 125.6 (q, *J* = 3.8 Hz), 127.1, 127.2, 128.2, 128.6, 129.4 (q, *J* = 32.1 Hz), 140.3, 149.9. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -62.25 (3F).



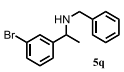
5o

1-(3-methylphenyl)-*N*-(phenylmethyl)ethanamine Colorless oil, 69.3 mg, 77% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.39 (d, *J* = 6.6 Hz, 3H), 1.70 (br, 1H), 2.37 (s, 3H), 3.61 (d, *J* = 13.2 Hz, 1H), 3.67 (d, *J* = 13.2 Hz, 1H), 3.79 (q, *J* = 6.6 Hz, 1H), 7.11-7.37 (m, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 21.6, 24.6, 51.8, 57.6, 123.9, 126.9, 127.5, 127.8, 128.2, 128.5, 138.1, 140.8, 145.7. **HRMS**: calcd. for $(M+H)^+$ m/z 226.1596, found $(M+H)^+$ m/z 226.1591.



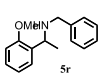
5p

1-(3-bromophenyl)-*N*-(phenylmethyl)ethanamine Colorless oil, 61.3 mg, 53% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.38 (d, *J* = 6.6 Hz, 3H), 1.62 (br, 1H), 3.62 (d, *J* = 13.2 Hz, 1H), 3.69 (d, *J* = 13.2 Hz, 1H), 3.80 (q, *J* = 6.6 Hz, 1H), 7.22-7.57 (m, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.6, 51.8, 57.2, 122.8, 125.5, 127.1, 128.2, 128.5, 129.9, 130.1, 130.2, 140.5, 148.3. **HRMS**: calcd. for $(M+H)^+$ m/z 290.0544, found $(M+H)^+$ m/z 290.0545.



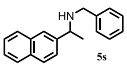
5q

***N*-benzyl-1-(2-Methoxyphenyl)ethanamine**[7] Colorless oil, 80.0 mg, 83% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.47 (d, J = 6.7 Hz, 3H), 1.99 (br, 1H), 3.68 (d, J = 12.9 Hz, 1H), 3.74 (d, J = 12.9 Hz, 1H), 3.87 (s, 1H), 4.25 (q, J = 6.7 Hz, 1H), 6.94-7.47 (m, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 22.2, 51.6, 52.3, 55.1, 110.4, 120.6, 126.6, 127.1, 127.5, 128.1, 128.2, 133.1, 140.8, 157.1.



5r

***N*-benzyl-1-(naphthalen-2-yl)ethanamine**[5] Colorless oil, 101.3 mg, 97% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.51 (d, *J* = 6.6 Hz, 3H), 1.81 (br, 1H), 3.69 (d, *J* = 13.2 Hz, 1H), 3.75 (d, *J* = 13.2 Hz, 1H), 4.04 (q, *J* = 6.6 Hz, 1H), 7.29-7.91 (m, 12H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.6, 51.8, 57.7, 125.0, 125.5, 125.6, 126.1, 127.0, 127.8, 127.9, 128.3, 128.4, 128.5, 133.0, 133.6, 140.7, 143.1.

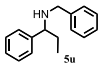


5s

1-(furan-2-yl)-*N*-(phenylmethyl)ethanamine[5] Colorless oil, 75.6 mg, 94% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.45 (d, *J* = 6.6 Hz, 3H), 1.76 (br, 1H), 3.68 (d, *J* = 13.1 Hz, 1H), 3.76 (d, *J* = 13.1 Hz, 1H), 3.91 (q, *J* = 6.6 Hz, 1H), 6.19 (d, *J* = 3.2 Hz, 1H), 6.34-6.35 (m, 1H), 7.25-7.39 (m, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 20.5, 50.6, 51.2, 105.5, 110.0, 127.0, 128.3, 128.5, 140.4, 141.5, 157.9.

5t

***N*-benzyl-α-ethylbenzylamine**[8] Colorless oil, 88.2 mg, 98% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.93 (t, J = 7.5 Hz, 3H), 1.75-1.90 (m, 3H), 3.65 (t, J = 7.6 Hz, 1H), 3.67 (d, J = 13.0 Hz, 1H), 3.77 (d, 13.2 Hz, 1H), 7.32-7.47 (m, 10H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 10.7, 31.0, 51.5, 64.1, 126.7, 126.8, 127.3, 128.0, 128.2, 140.7, 144.0.

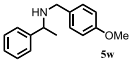


5u

***N*-benzyl-3-methylbutan-2-amine**[5] Colorless oil, 59.5 mg, 84% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.90 (d, *J* = 6.8 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 3H), 1.02 (d, *J* = 6.5 Hz, 3H), 1.41 (br, 1H), 1.71-1.78 (m, 1H), 2.51-2.56 (m, 1H), 3.72 (d, J = 13.1 Hz, 1H), 3.83 (d, J = 13.1 Hz, 1H), 7.24-7.27 (m, 1H), 7.31-7.35 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 16.1, 17.4, 19.4, 32.3, 51.7, 57.7, 126.9, 128.2, 128.4, 141.2.

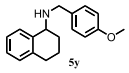
5v

(p-methoxybenzyl)(1-phenylethyl)amine[9] Colorless oil, 95.4 mg, 99% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.38 (d, *J* = 6.6 Hz, 3H), 1.76 (br, H), 3.55 (d, *J* = 12.9 Hz, 1H), 3.62 (d, *J* = 12.9 Hz, 1H), 3.81 (s, 3H), 3.82 (q, *J* = 6.6 Hz, 1H), 6.87 (d, *J* = 8.6, 2H), 7.22 (d, *J* = 8.5, 2H), 7.27-7.29 (m, 1H), 7.35-7.37 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 24.6, 51.1, 55.4, 57.5, 113.9, 126.8, 127.0, 128.6, 129.4, 132.9, 145.7, 158.7.



(Isobutyl)(1-phenylethyl)amine[7] Yellow oil, 53.1 mg, 75% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.88 (d, J = 6.7 Hz, 3H), 0.89 (d, J = 6.7 Hz, 3H), 1.36 (d, J = 6.6, 3H), 1.45 (br, 1H), 1.70 (m, 1H), 2.23 (dd, J = 11.4 Hz, J = 7.3 Hz, 1H), 2.35 (dd, J = 11.4 Hz, J = 7.3 Hz, 1H), 3.75 (q, J = 6.6 Hz, 1H), 7.22-7.26 (m, 1H), 7.30-7.34 (m, 4H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 20.6, 20.8, 24.5, 28.5, 55.8, 58.3, 126.5, 126.7, 128.3, 146.0.

***N*-[(4-methoxyphenyl)methyl]-1,2,3,4-tetrahydronaphthalen-1-amine** Yellow oil, 99.3 mg, 93% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.57 (br, 1H), 1.79 (m, 1H), 1.95 (m, 2H), 2.06 (m, 1H), 2.77 (m, 1H), 2.88 (m, 1H), 3.82-3.94 (m, 6H), 6.92 (d, *J* = 8.6 Hz, 2H), 7.12 (m, 1H), 7.17-7.19 (m, 2H), 7.35-7.40 (m, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 19.0, 28.1, 29.3, 50.5, 54.5, 55.2, 113.7, 125.6, 126.5, 128.7, 128.9, 129.2, 133.0, 137.4, 139.3, 158.5.



IV. References

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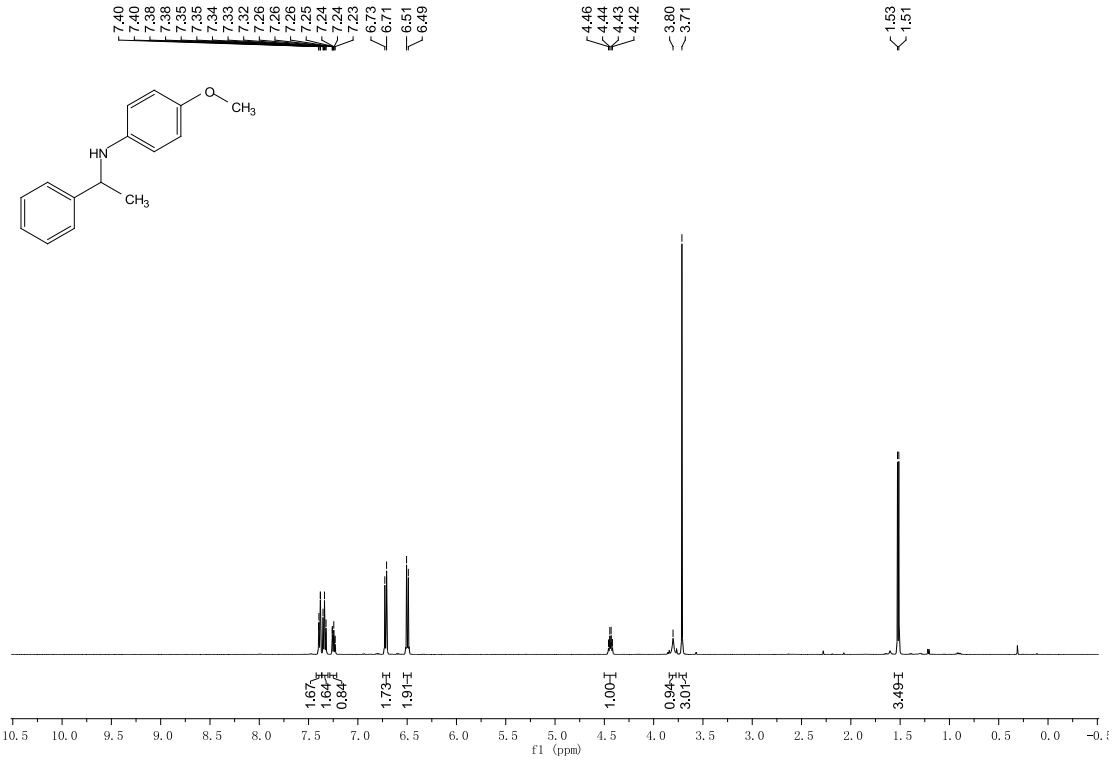
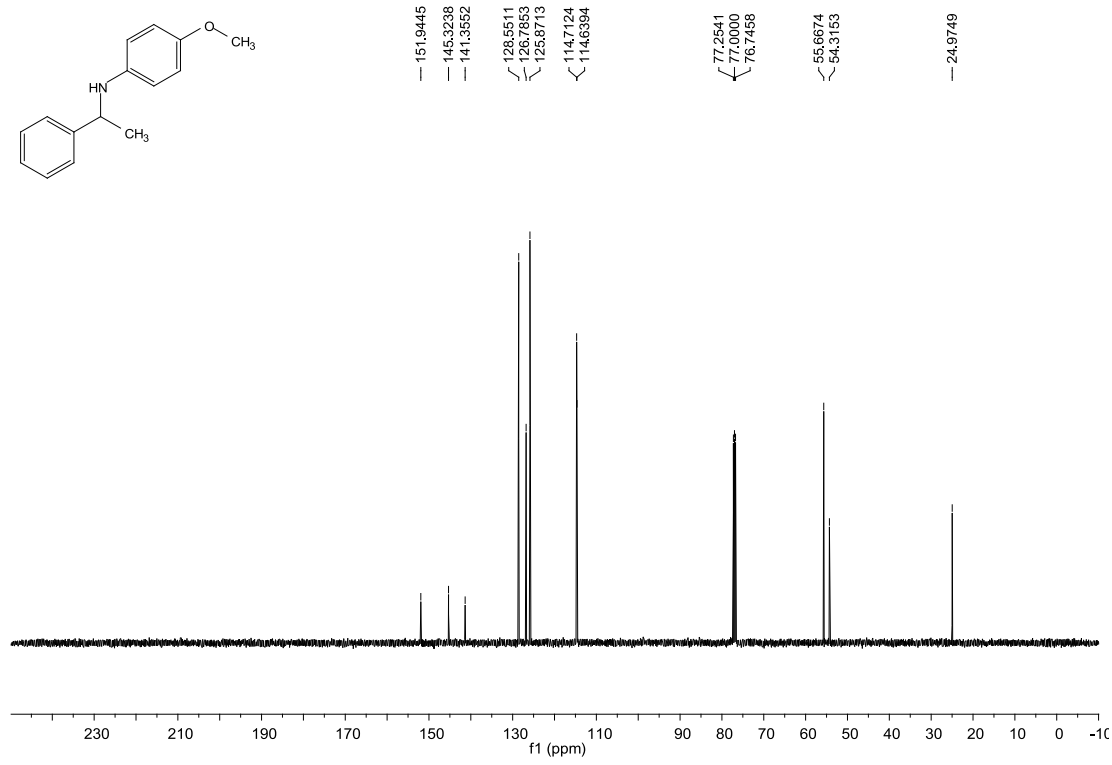
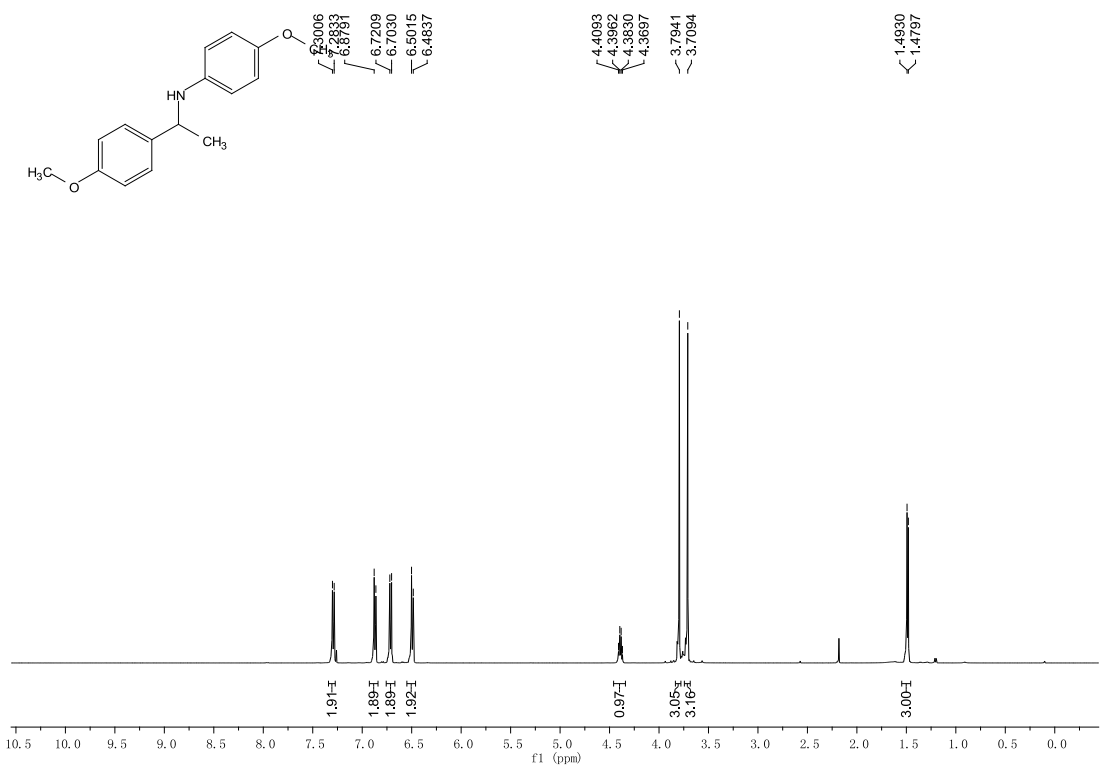
V. NMR Spectra



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O
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1.89
1.89
1.92
0.97
3.05
3.16
3.00
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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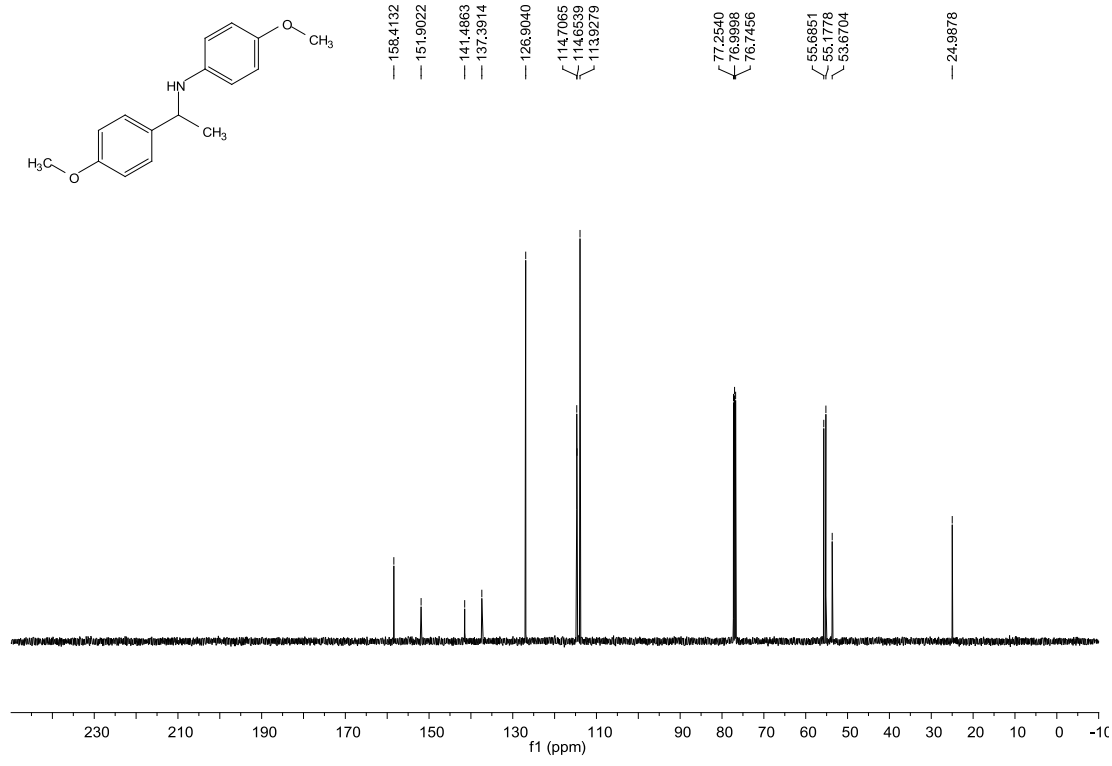
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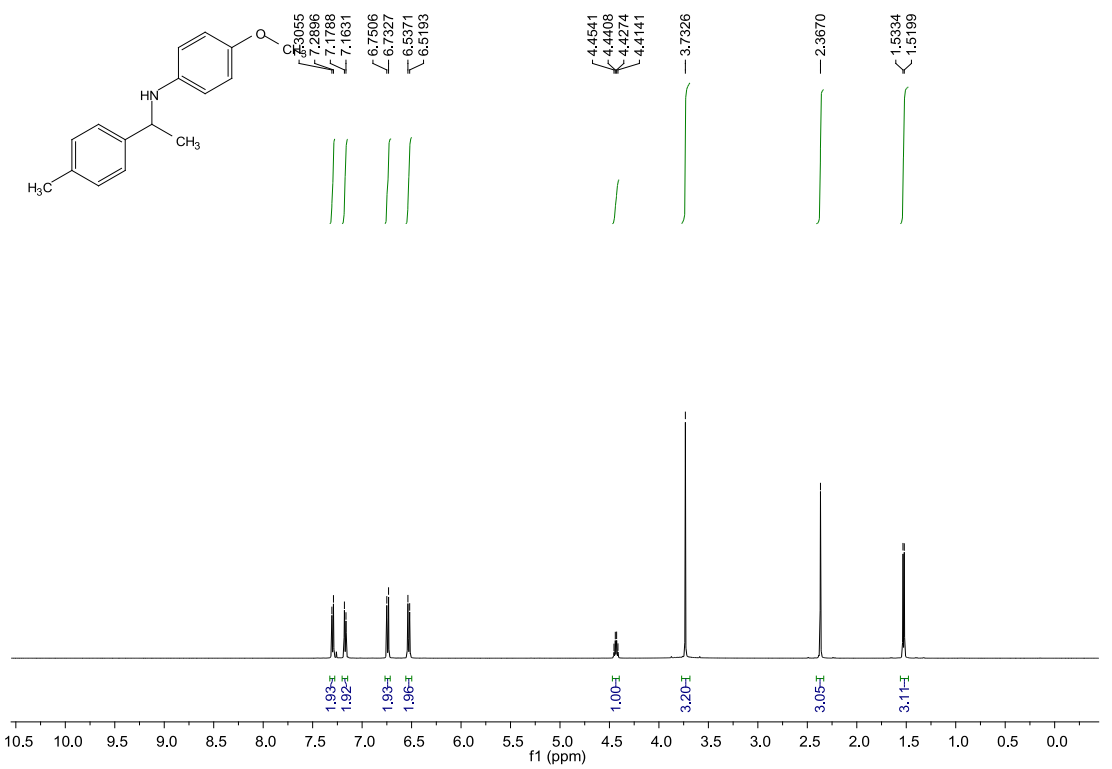
f1 (ppm)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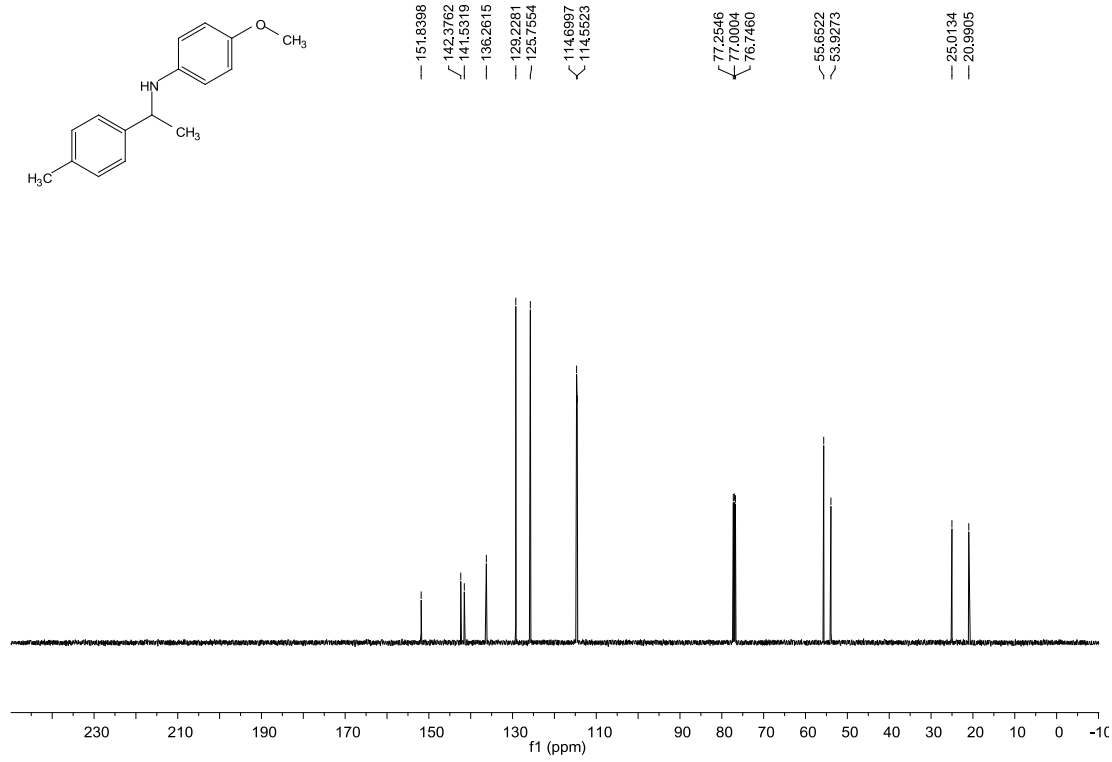
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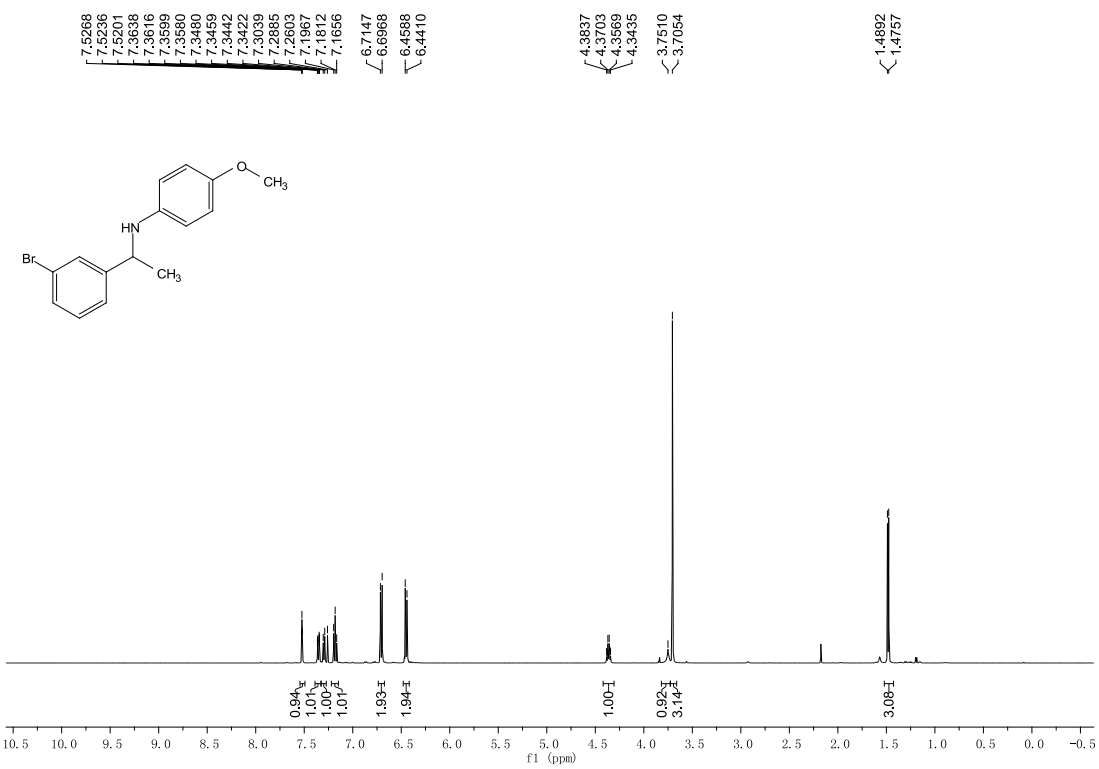
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3.08
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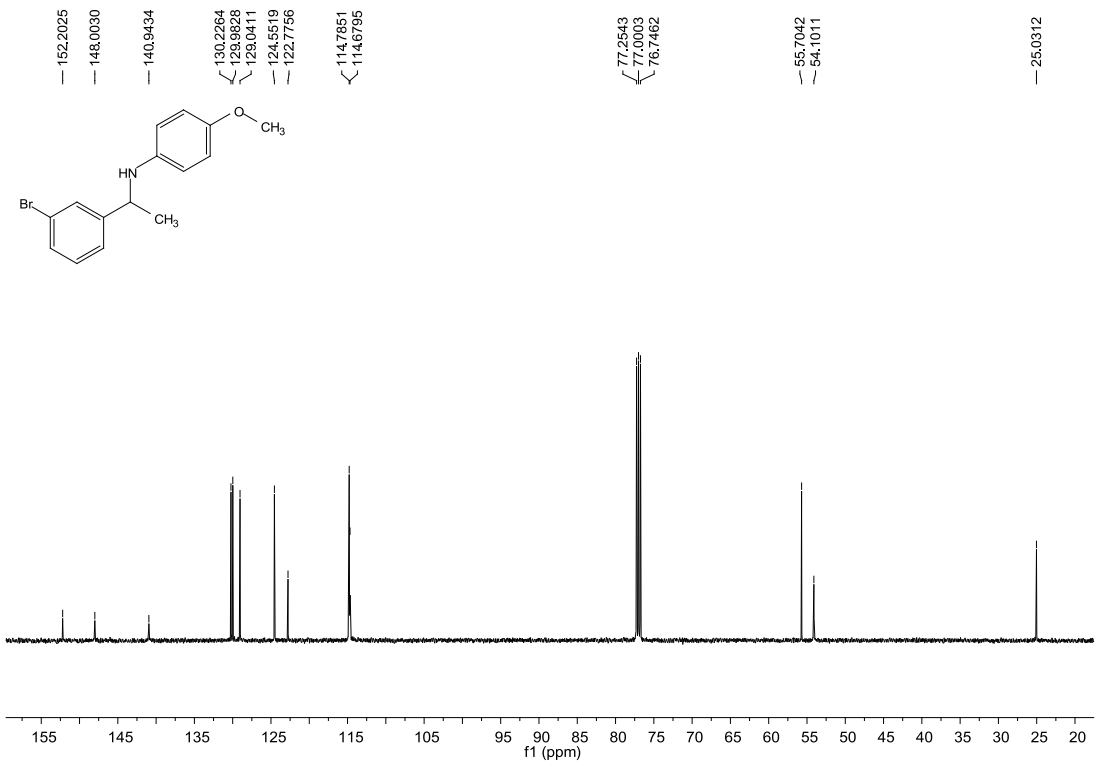
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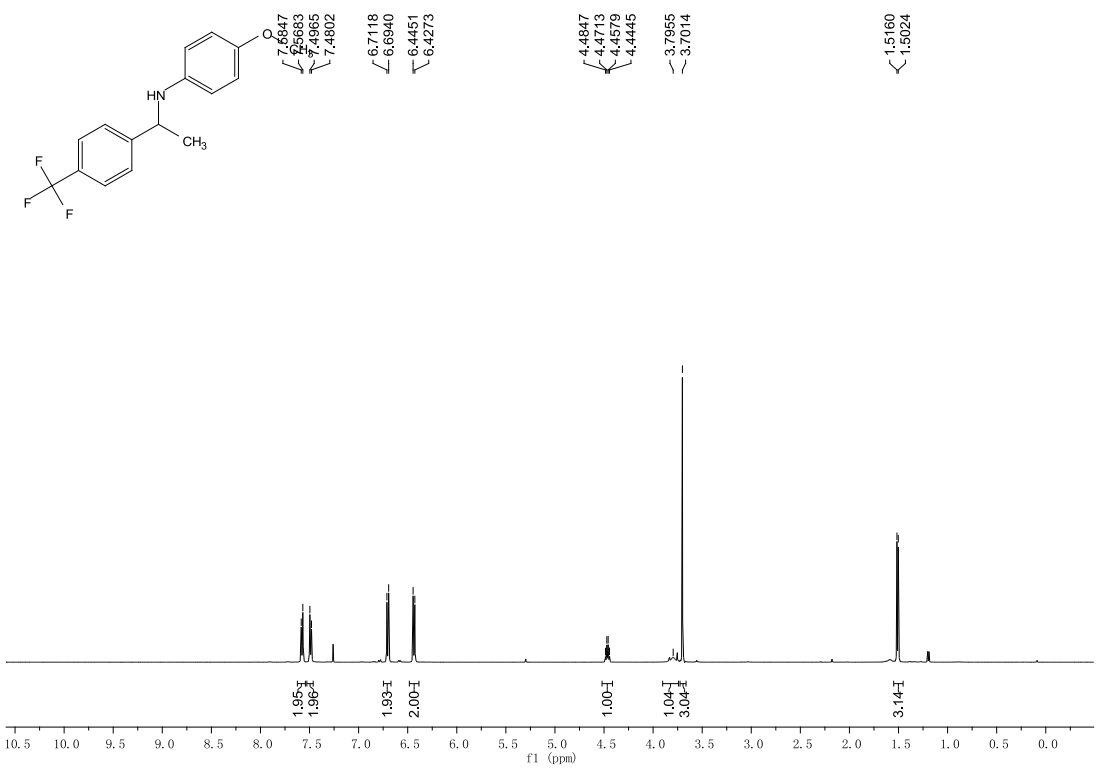
f1 (ppm)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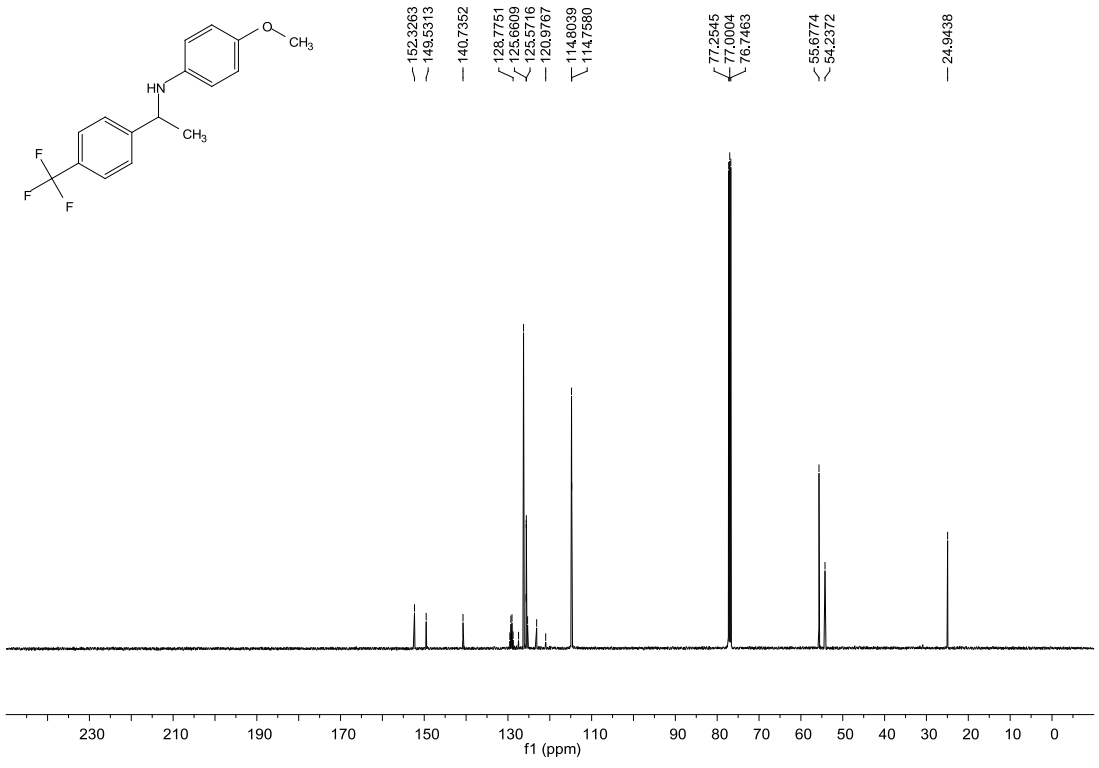
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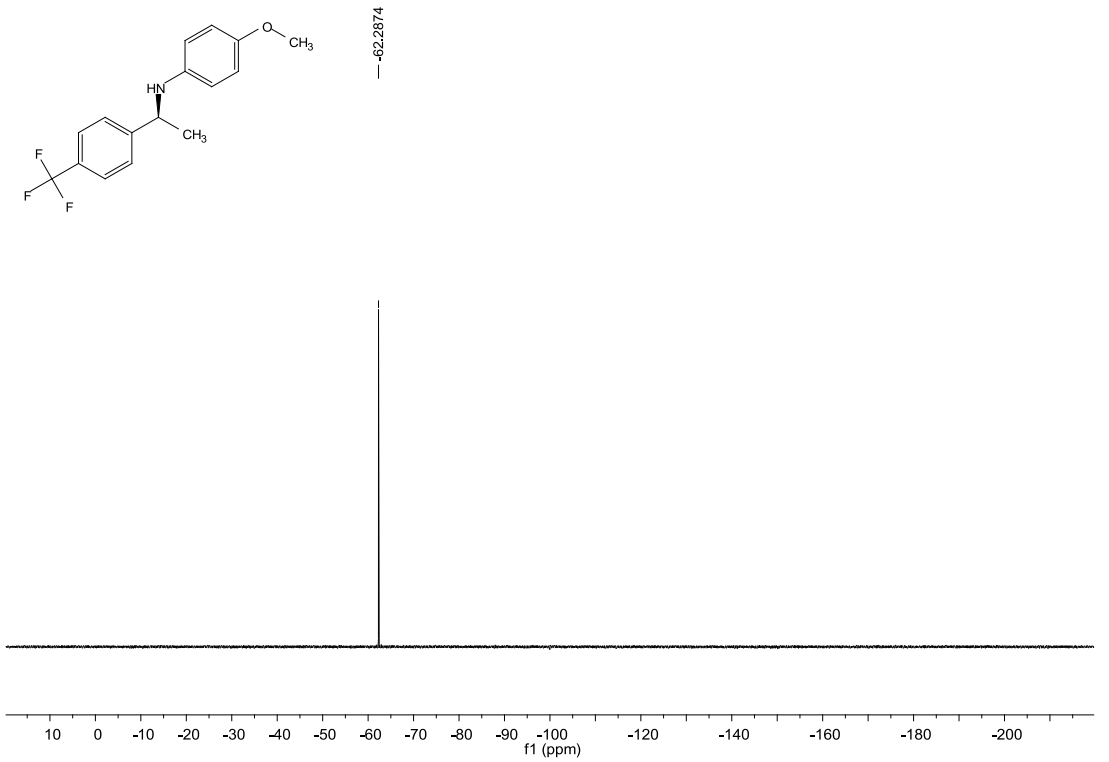
f1 (ppm)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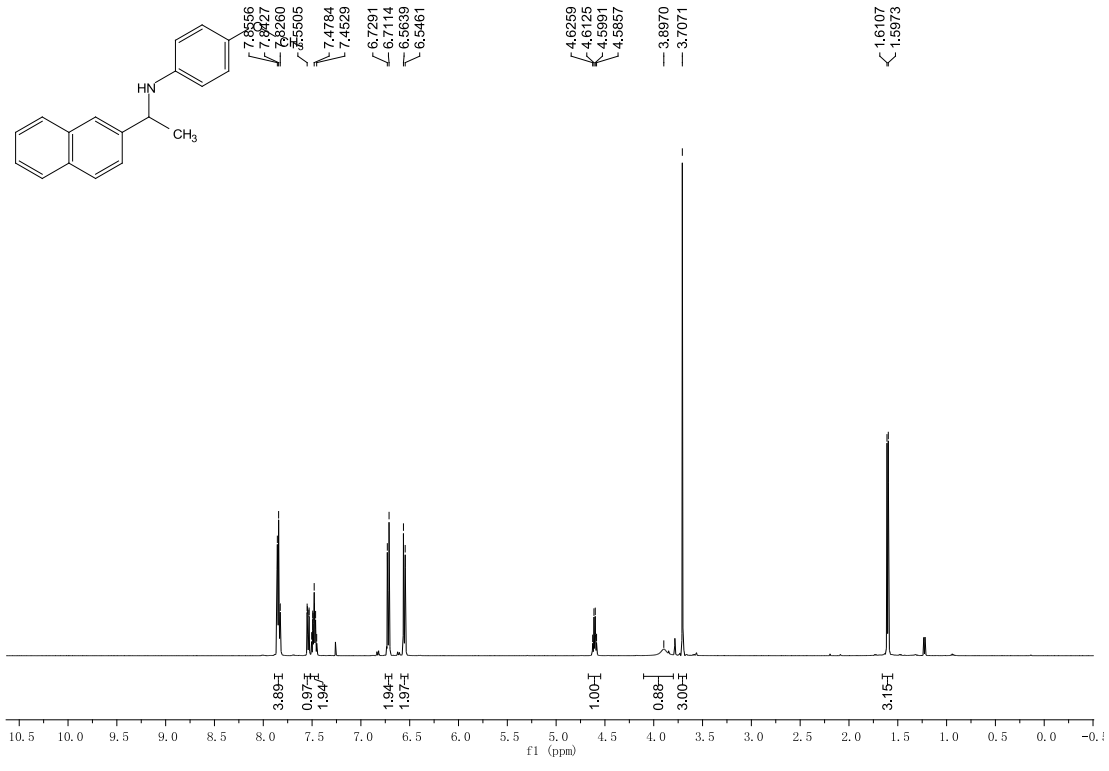
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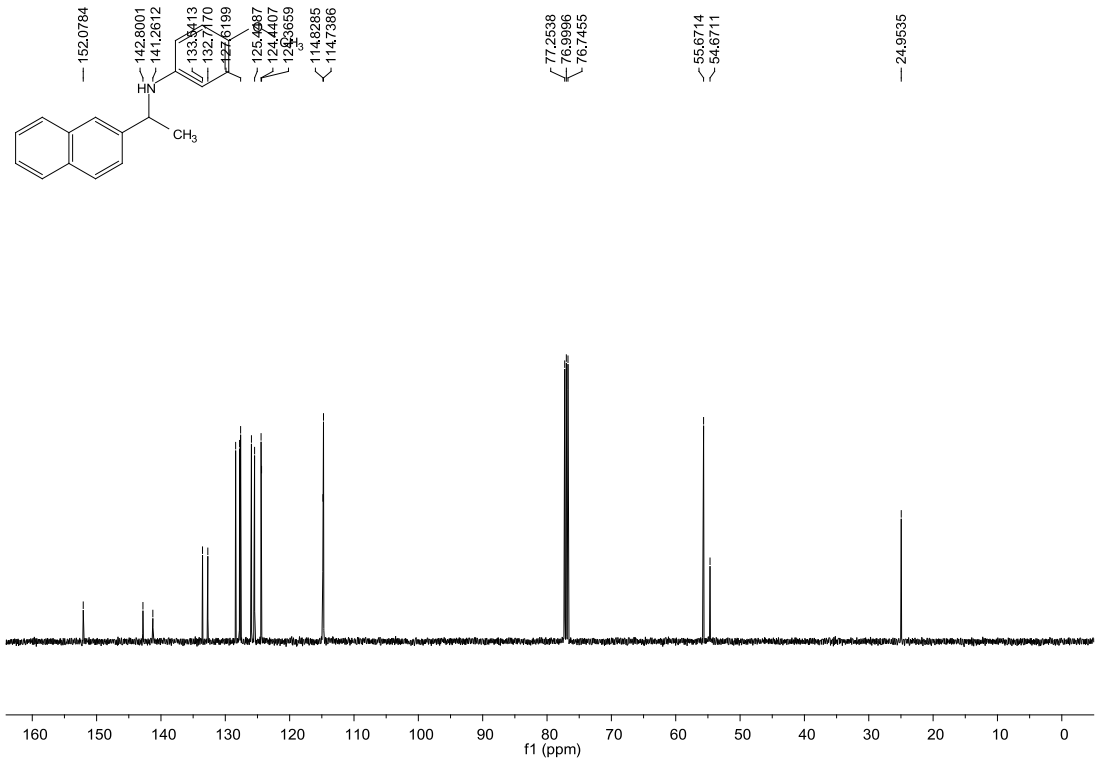
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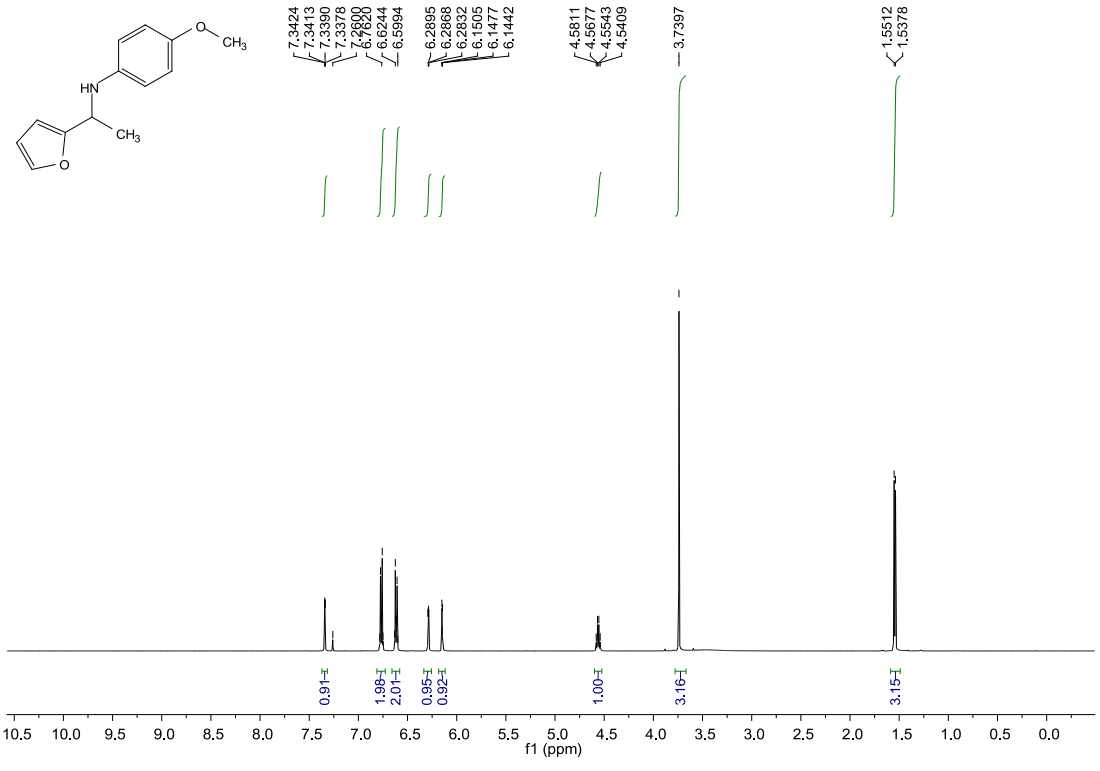
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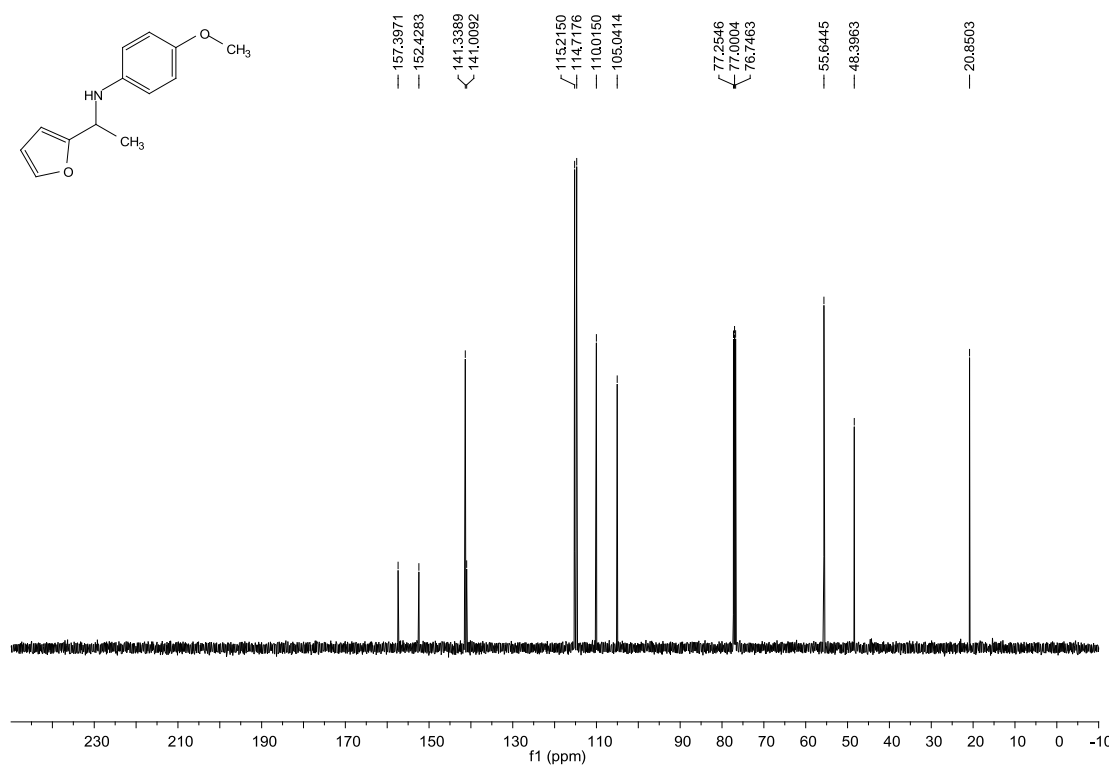
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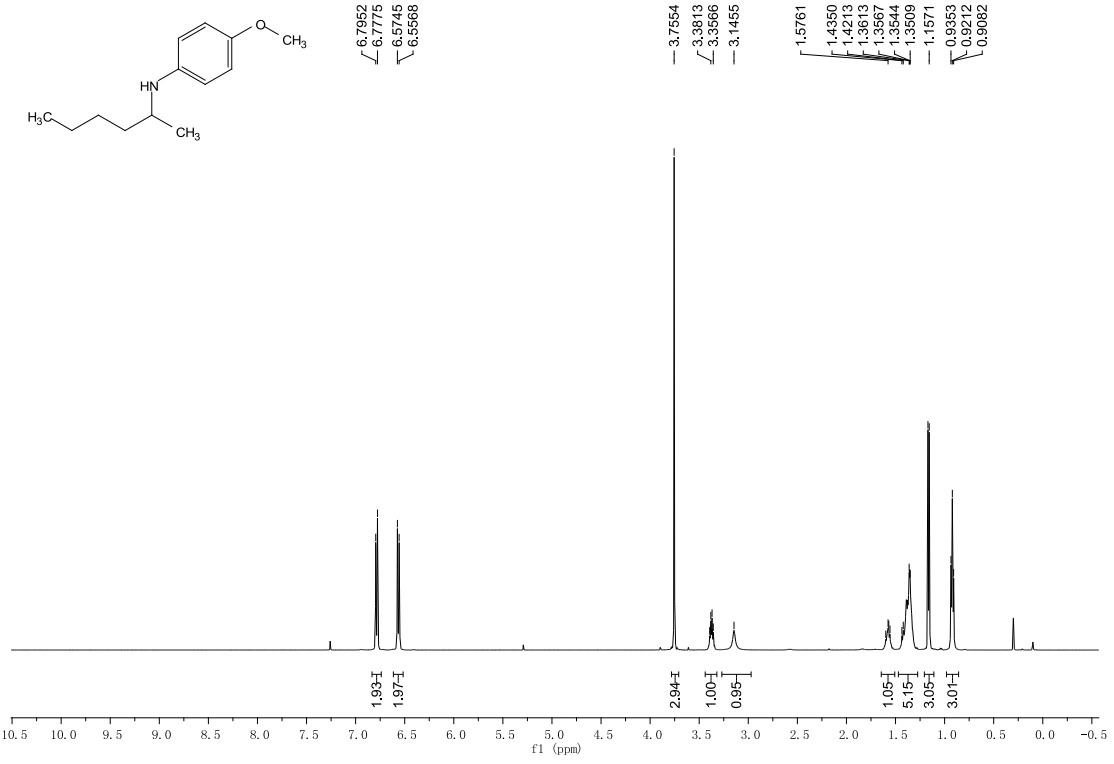
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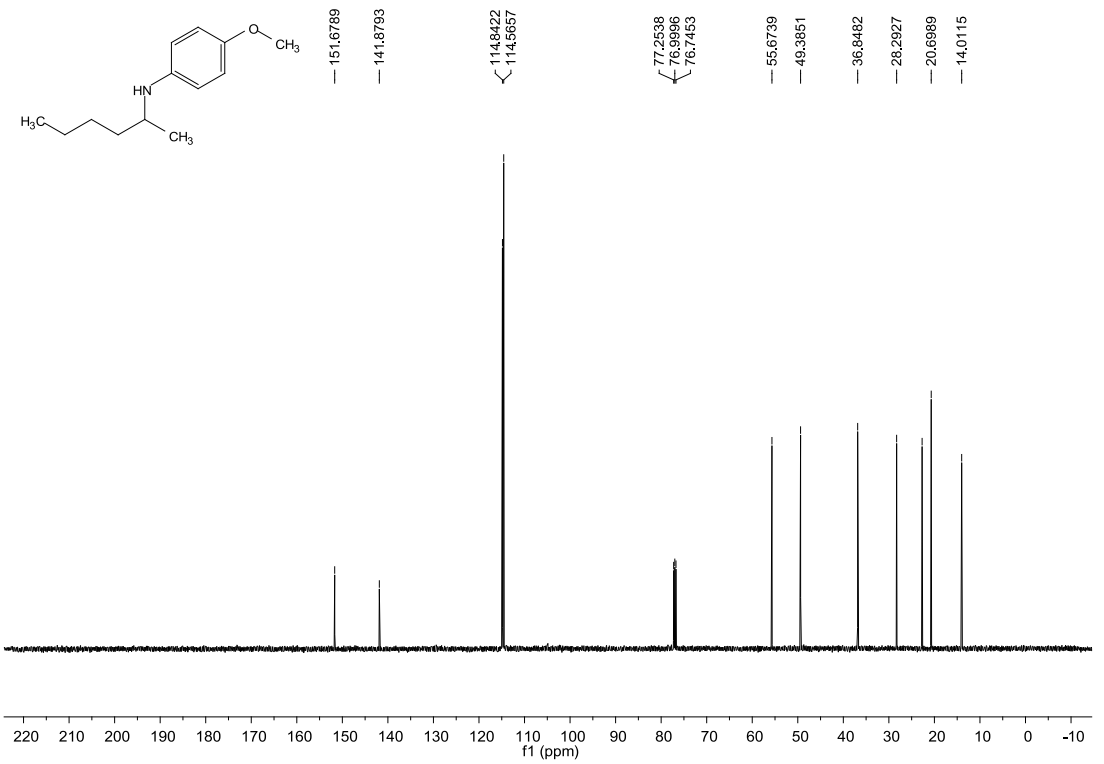
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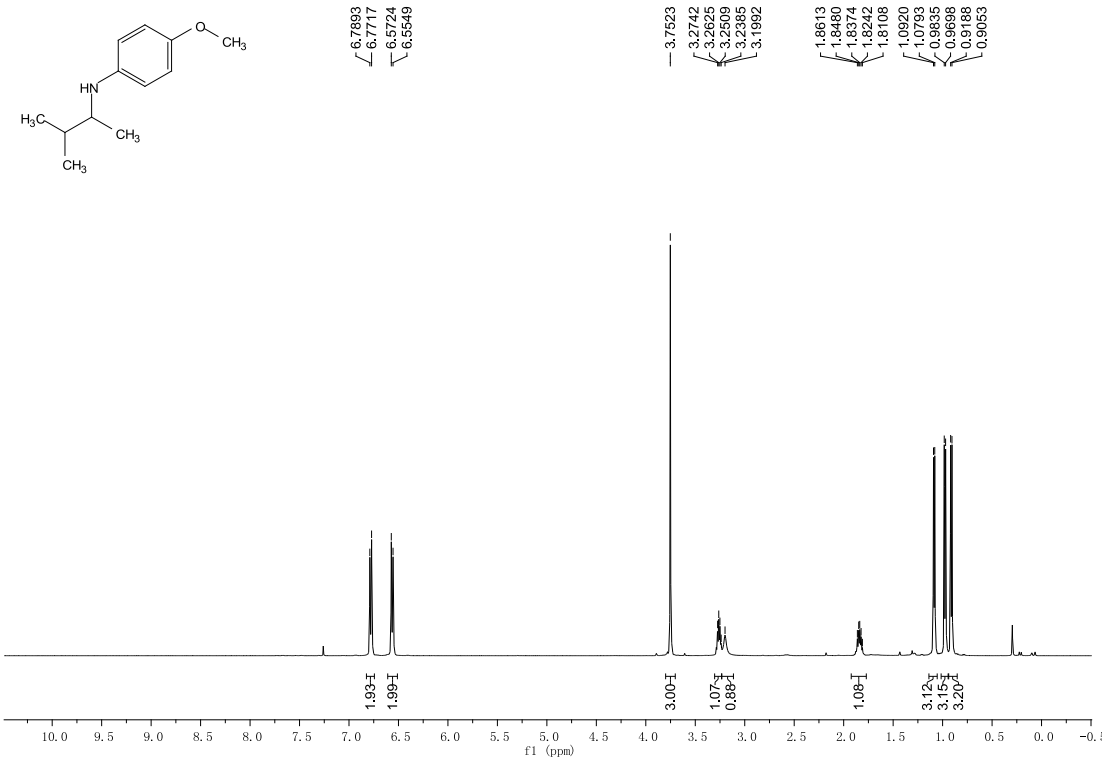
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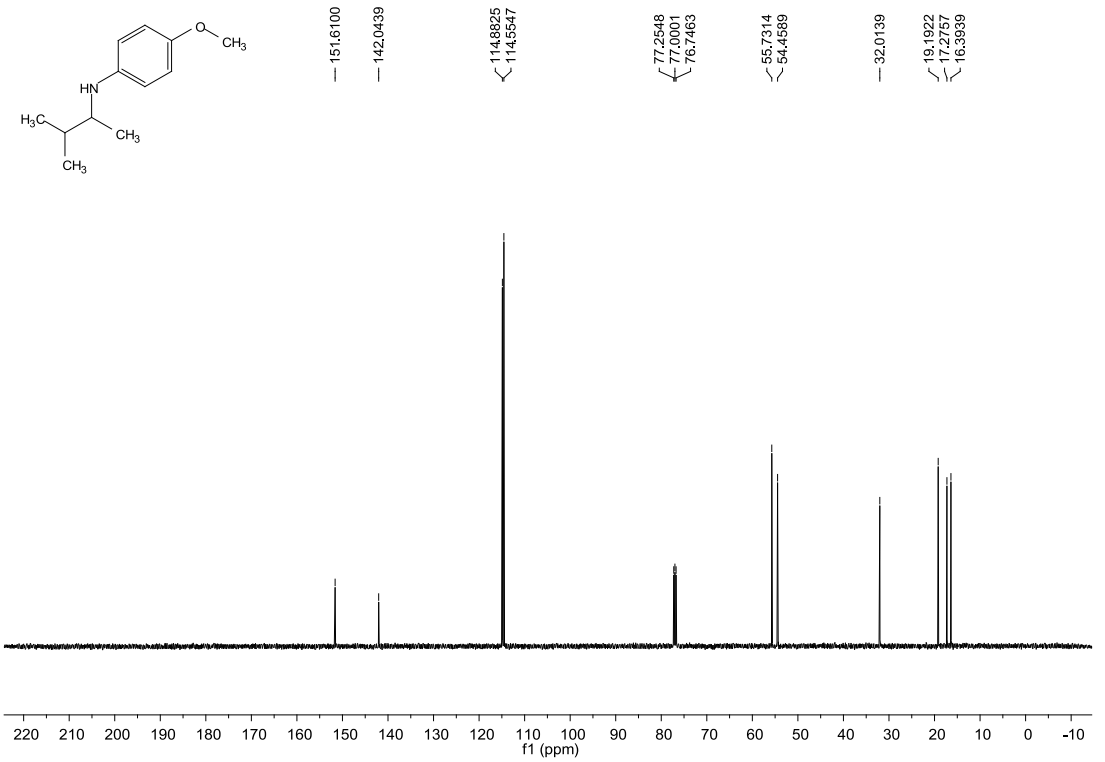
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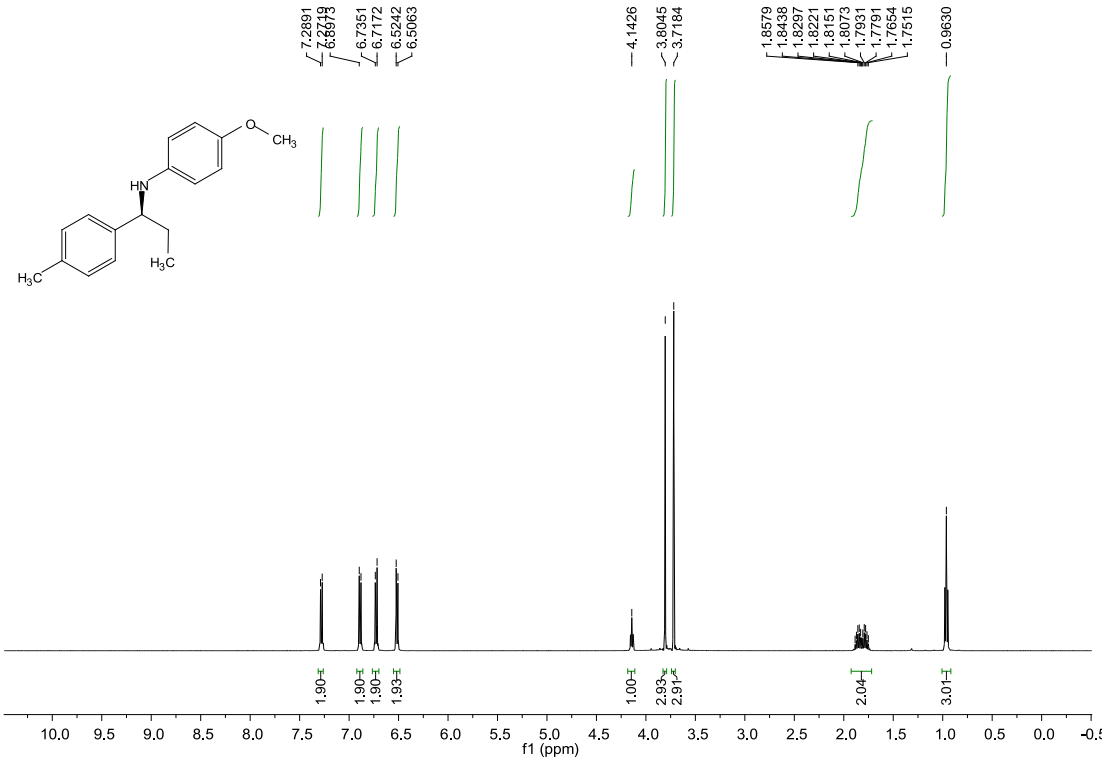
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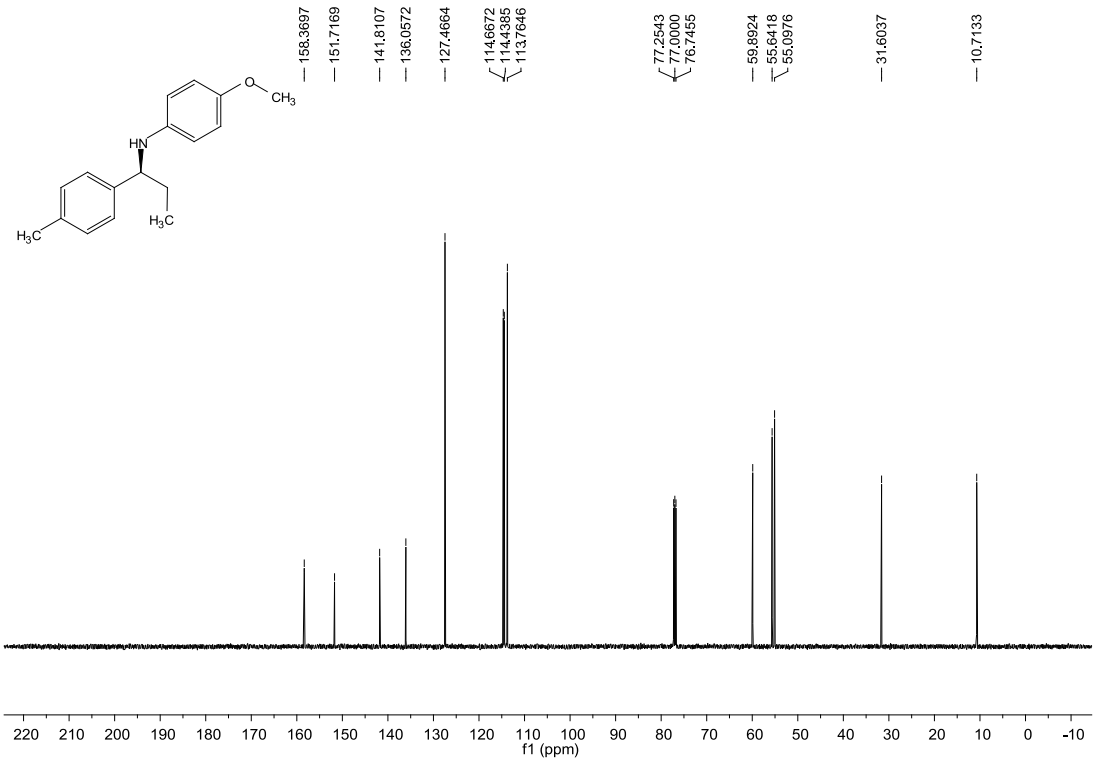
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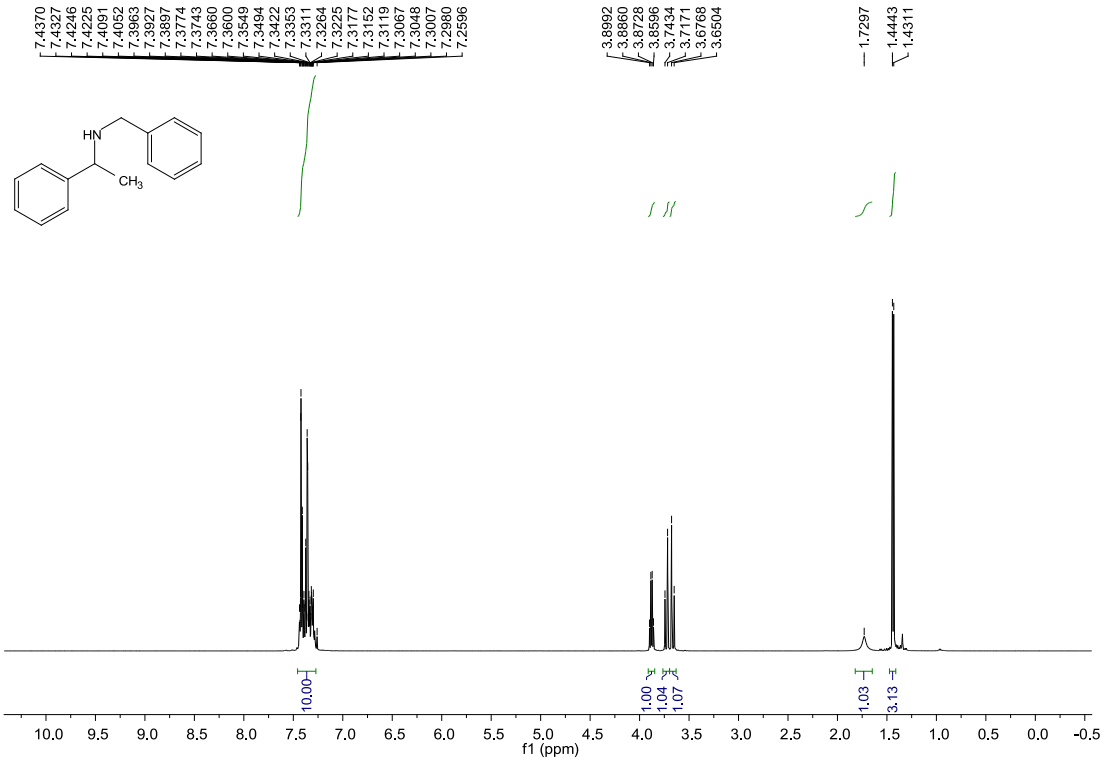
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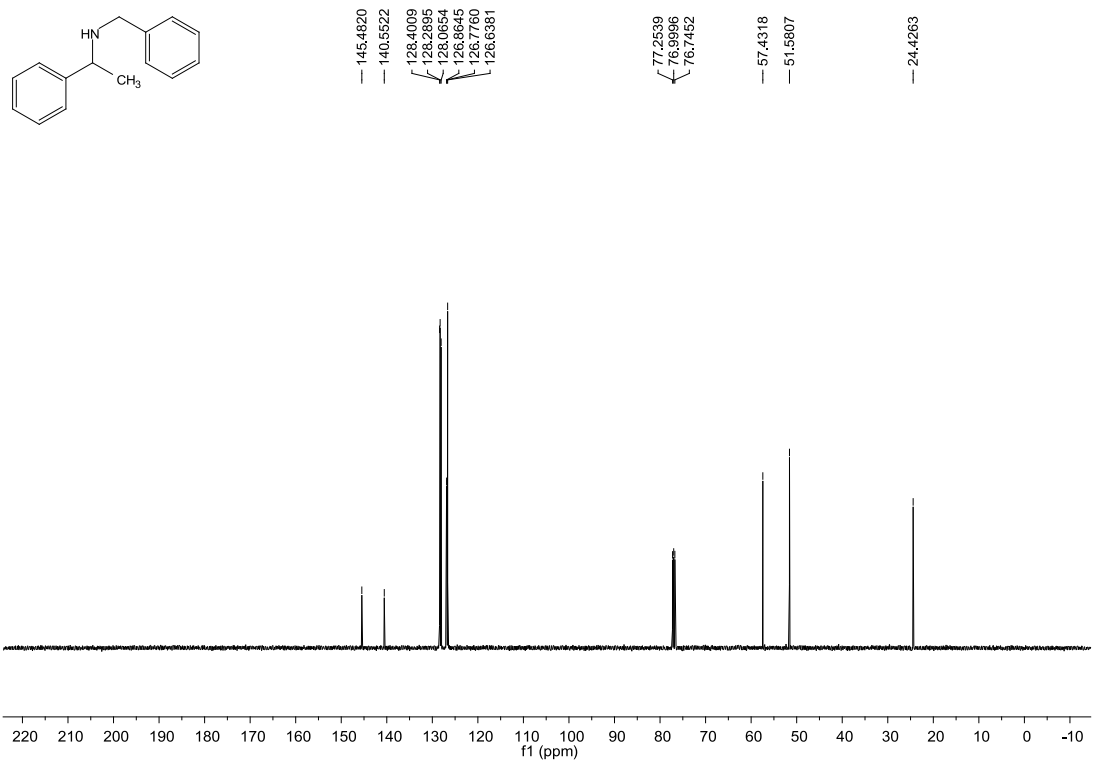
3.13
f1 (ppm)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CH3
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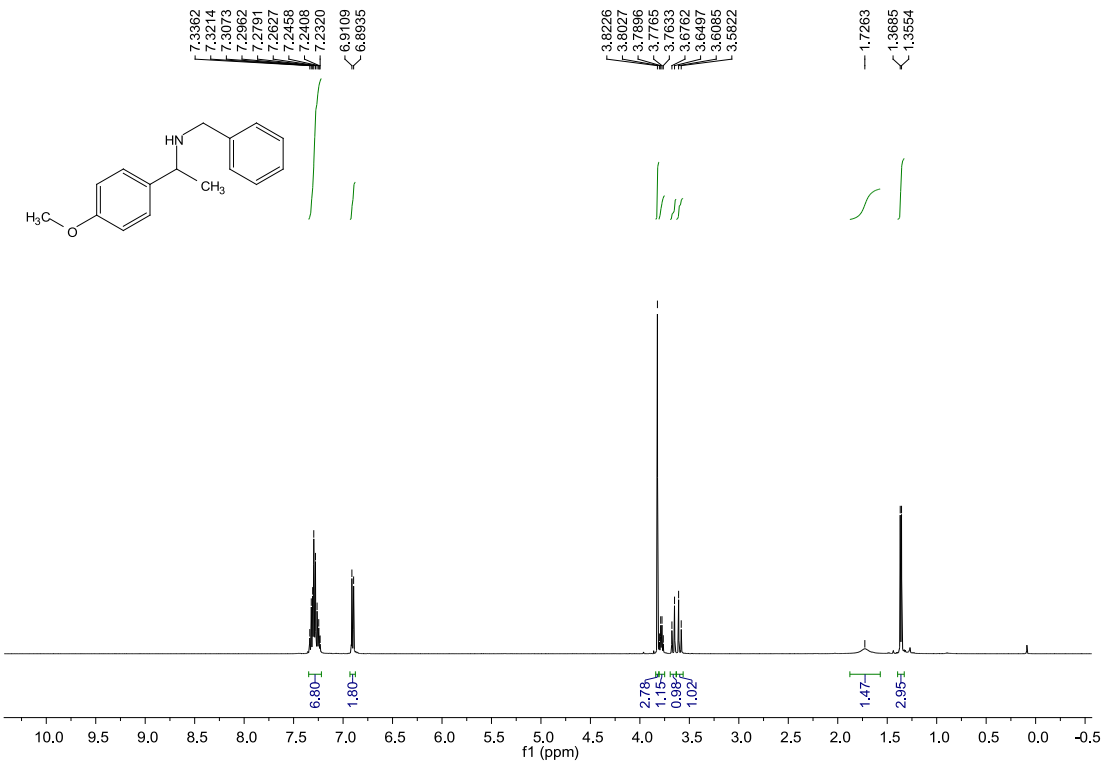
140.5522
128.4009
128.2895
128.0654
126.8645
126.7760
126.6381
77.2539
76.9996
76.7452
57.4318
51.5807
24.4263
f1 (ppm)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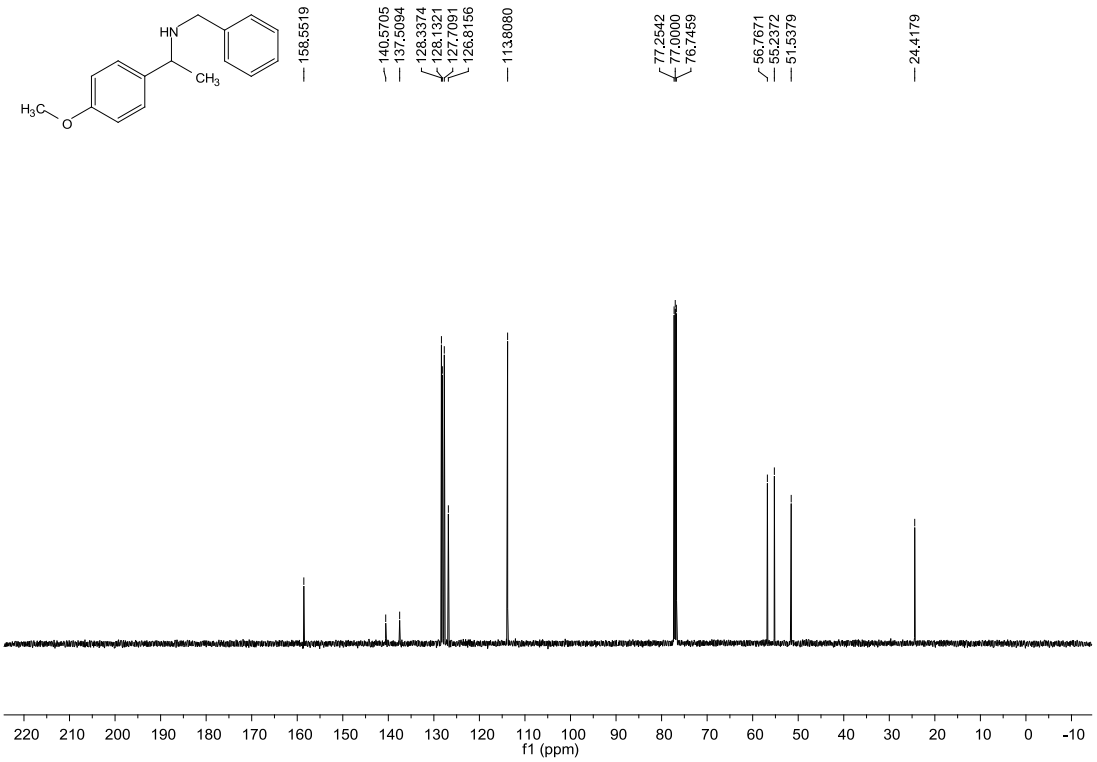
7.3073
7.2962
7.2791
7.2627
7.2458
7.2408
7.2320
6.9109
6.8935
3.8226
3.8027
3.7896
3.7765
3.7633
3.6762
3.6497
3.6085
3.5822
1.7263
1.3685
1.3554
HN
CH3
H3C
O
6.80
1.80
2.78
1.15
0.98
1.02
1.47
2.95
f1 (ppm)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CH3
H3C
O
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137.5094
128.3374
128.1321
127.7091
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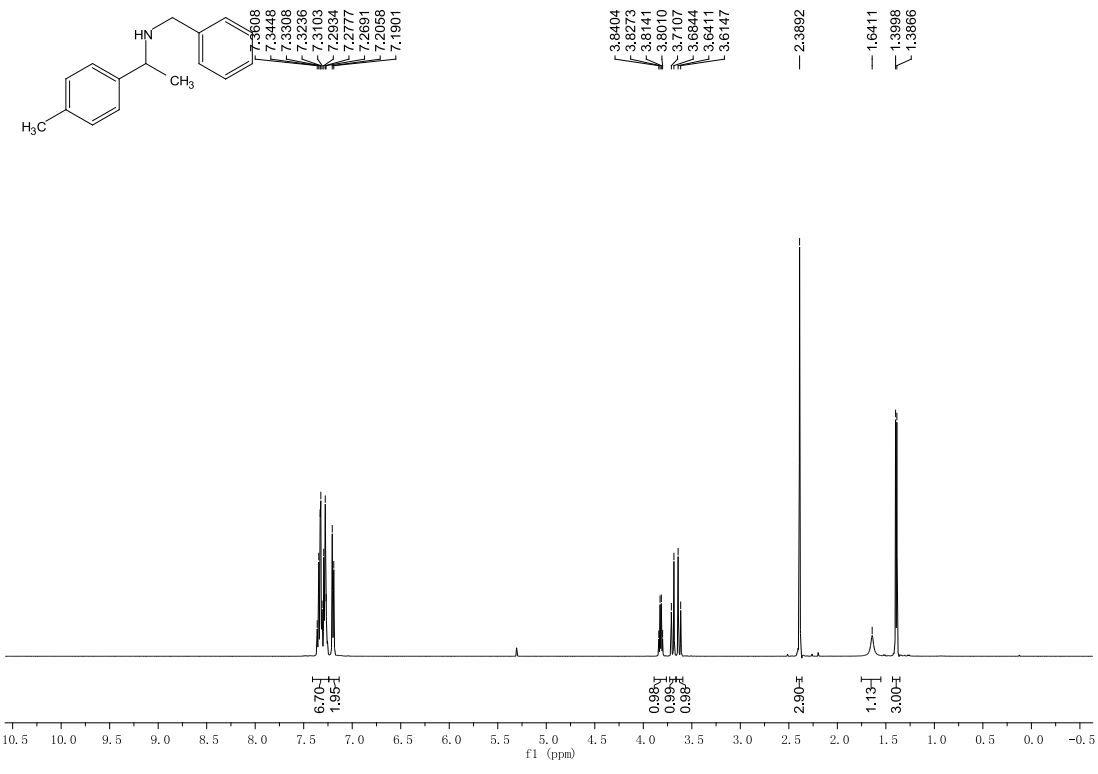
77.2542
77.0000
76.7459
56.7671
55.2372
51.5379
24.4179
f1 (ppm)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CH3
H3C
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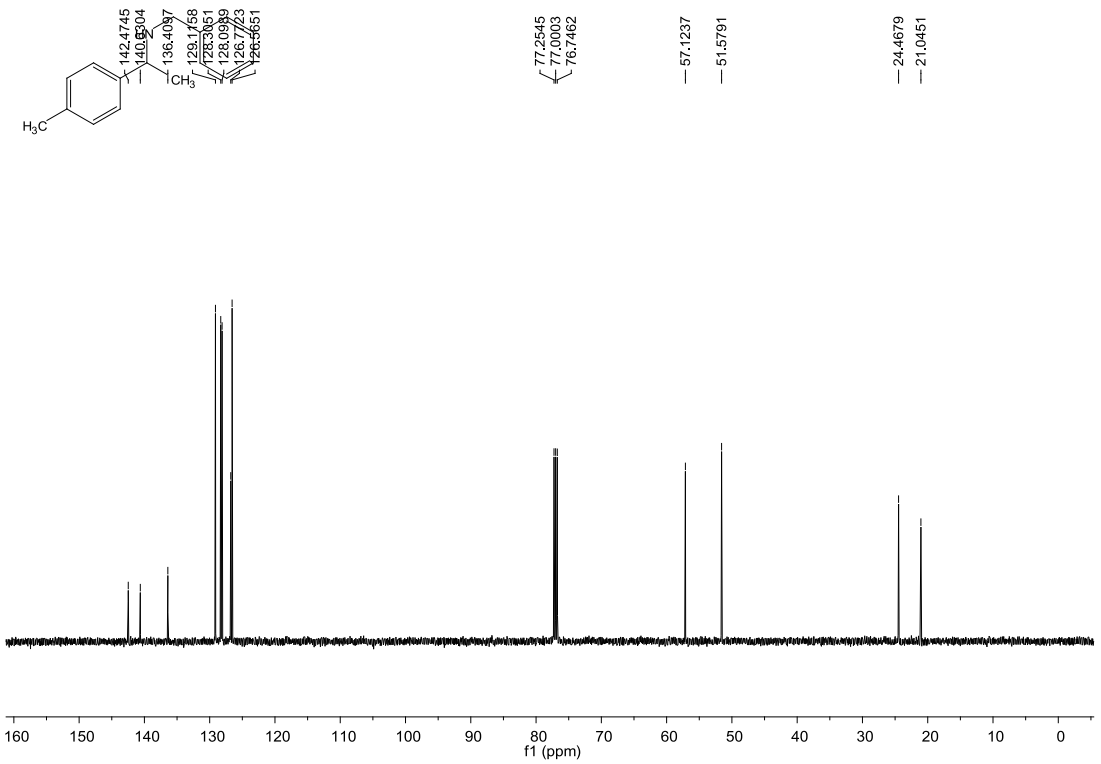
7.3308
7.3236
7.3103
7.2934
7.2777
7.2691
7.2058
7.1901
3.8404
3.8273
3.8141
3.8010
3.7107
3.6844
3.6411
3.6147
2.3892
1.6411
1.3998
1.3866
6.70
1.95
0.98
0.99
0.98
2.90
1.13
3.00
f1 (ppm)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126.7723
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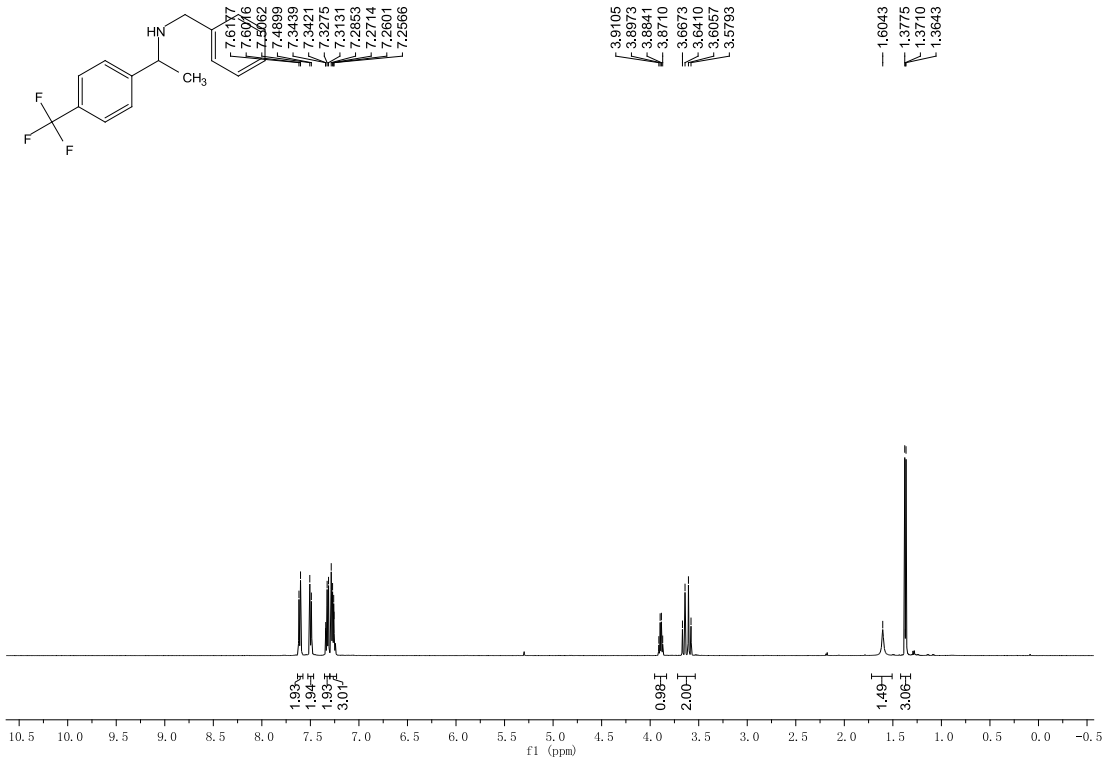
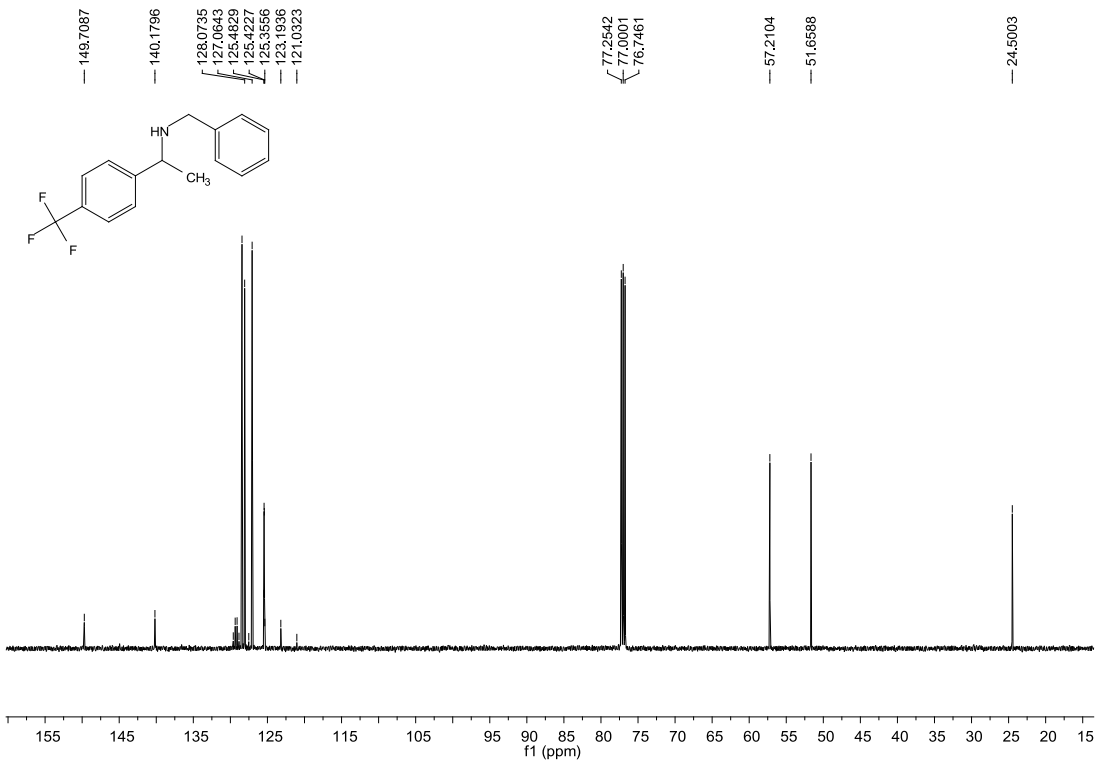
77.0003
76.7462
57.1237
51.5791
24.4679
21.0451
CH3
H3C
f1 (ppm)


HN
CH3
F
F
F
7.6177
7.6062
7.4899
7.3439
7.3421
7.3275
7.3131
7.2853
7.2714
7.2601
7.2566
3.9105
3.8973
3.8841
3.8710
3.6673
3.6410
3.6057
3.5793
1.6043
1.3775
1.3710
1.3643
1.93
1.94
1.93
3.01
0.98
2.00
1.49
3.06
f1 (ppm)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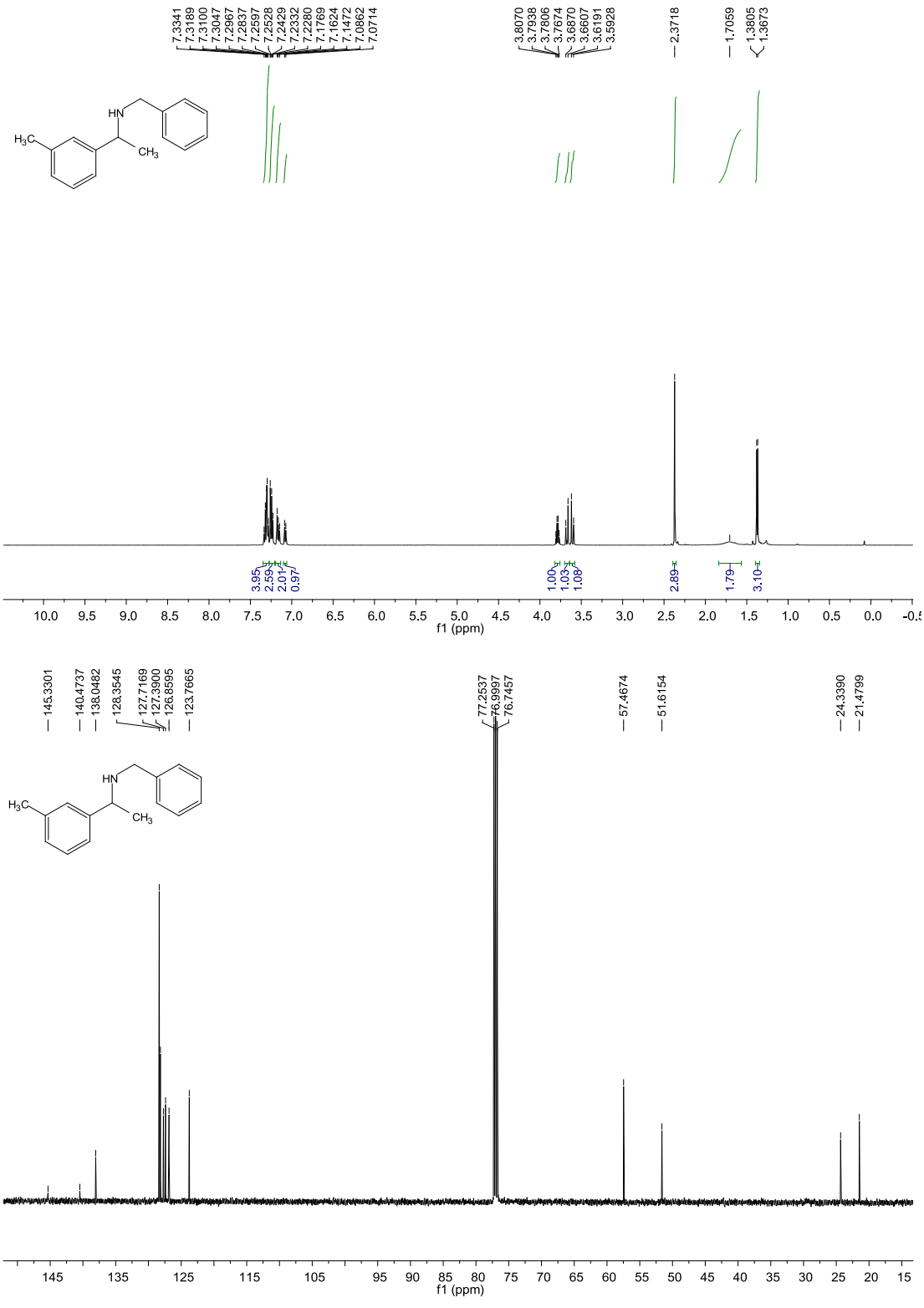
128.0735
127.0643
125.4829
125.4227
125.3556
123.1936
121.0323
77.2542
77.0001
76.7461
57.2104
51.6588
24.5003
HN
CH3
F
F
F
155 145 135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
f1 (ppm)


-62.2528
HN
CH3
F
F
F
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)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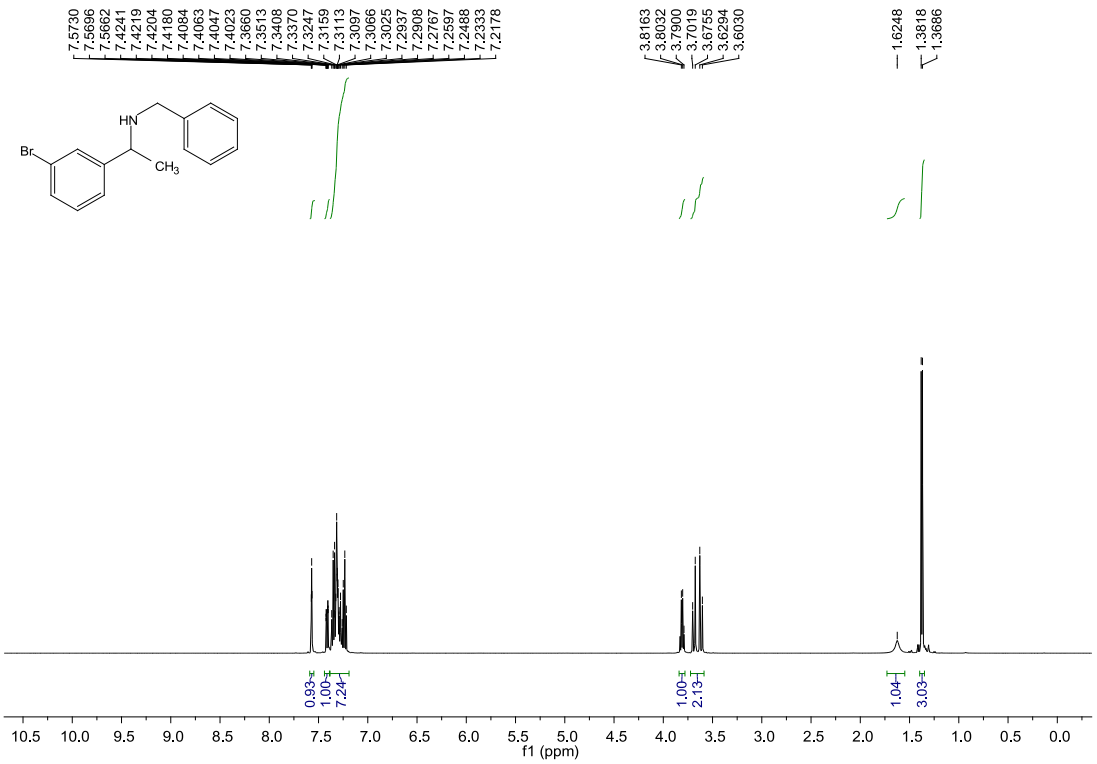
7.3100
7.3047
7.2967
7.2837
7.2597
7.2528
7.2429
7.2332
7.2280
7.1769
7.1624
7.1472
7.0862
7.0714
3.8070
3.7938
3.7806
3.7674
3.6870
3.6607
3.6191
3.5928
2.3718
1.7059
1.3805
1.3673
HN
H3C
CH3
3.95
2.59
2.01
0.97
1.00
1.03
1.08
2.89
1.79
3.10
f1 (ppm)
145.3301
140.4737
138.0482
128.3545
127.7169
127.3900
126.8595
123.7665
77.2537
76.9997
76.7457
57.4674
51.6154
24.3390
21.4799
f1 (ppm)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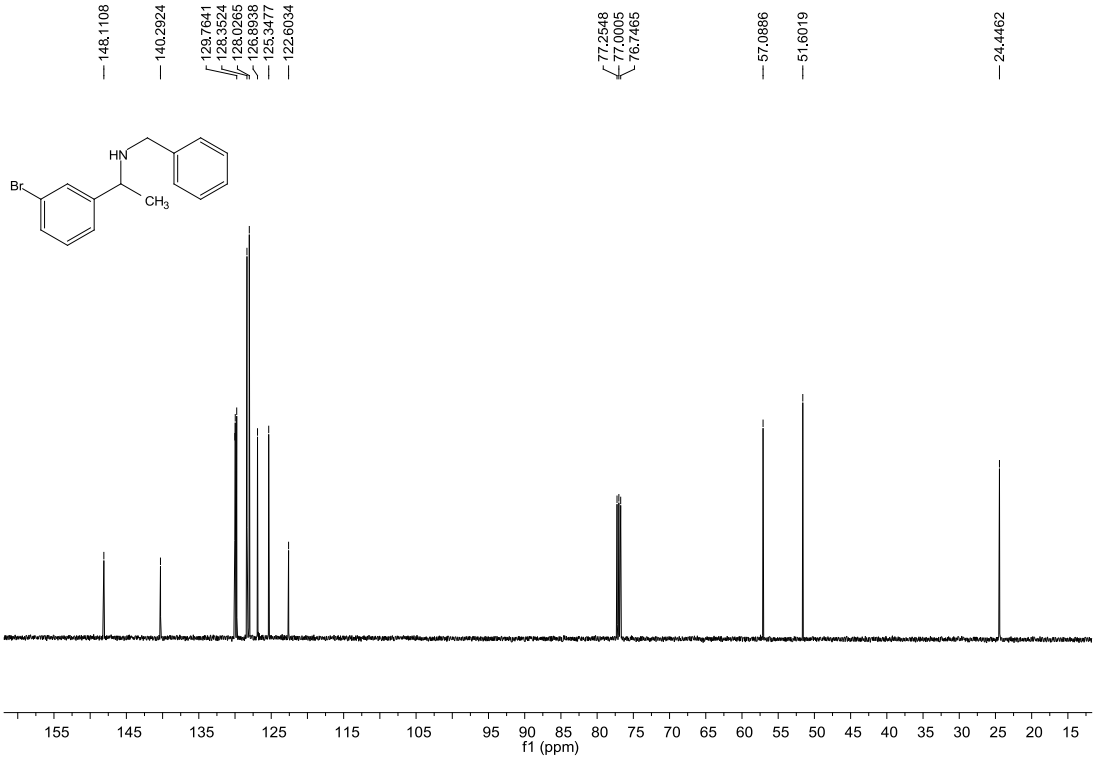
7.5696
7.5662
7.4241
7.4219
7.4204
7.4180
7.4084
7.4063
7.4047
7.4023
7.3660
7.3513
7.3408
7.3370
7.3247
7.3159
7.3113
7.3097
7.3066
7.3025
7.2937
7.2908
7.2767
7.2597
7.2488
7.2333
7.2178
3.8163
3.8032
3.7900
3.7019
3.6755
3.6294
3.6030
1.6248
1.3818
1.3686
HN
Br
CH3
0.93
1.00
7.24
1.00
2.13
1.04
3.03
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
f1 (ppm)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128.3524
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125.3477
122.6034
77.2548
77.0005
76.7465
57.0886
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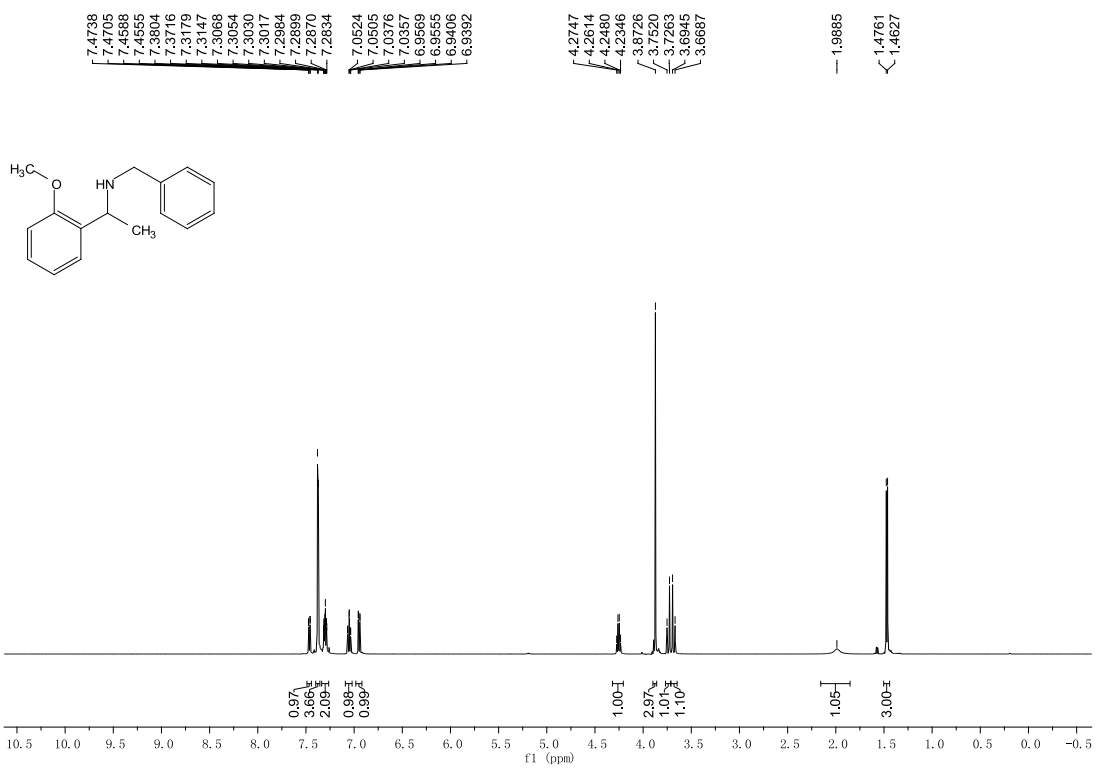
24.4462
HN
Br
CH3
155 145 135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
f1 (ppm)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7.4555
7.3804
7.3716
7.3179
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7.3068
7.3054
7.3030
7.3017
7.2984
7.2899
7.2870
7.2834
7.0524
7.0505
7.0376
7.0357
6.9569
6.9555
6.9406
6.9392
4.2747
4.2614
4.2480
4.2346
3.8726
3.7520
3.7263
3.6945
3.6687
1.9885
1.4761
1.4627
H3C
O
HN
CH3
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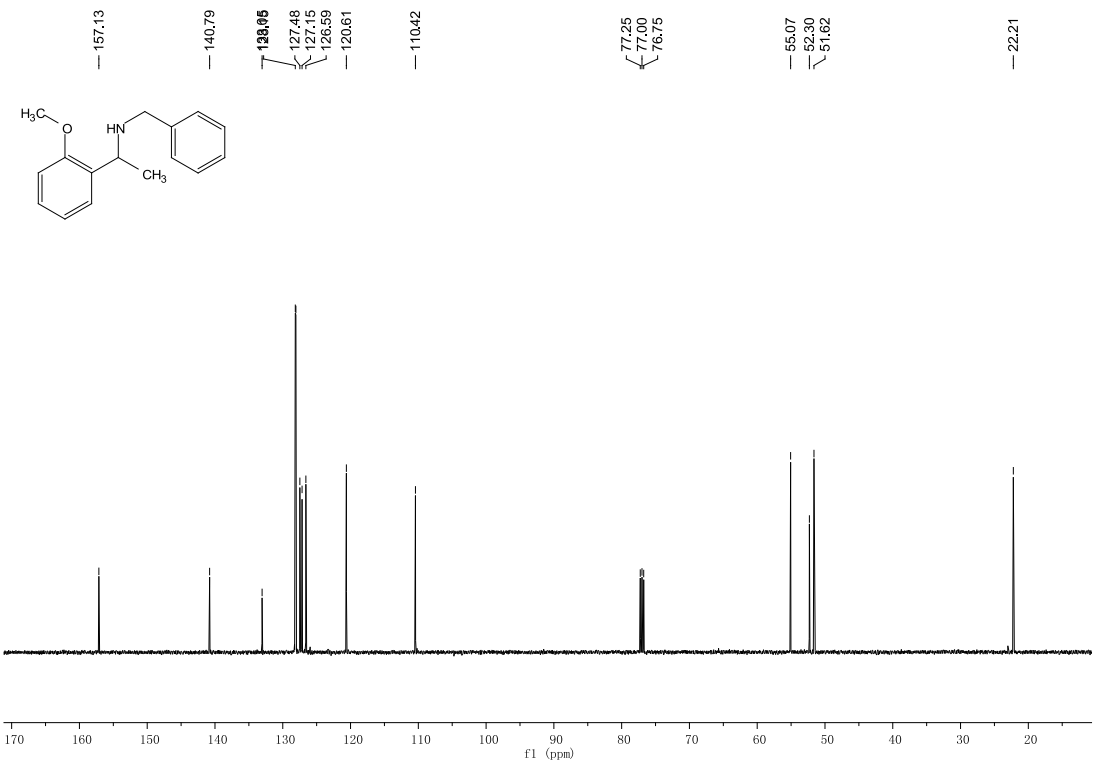
3.66
2.09
0.98
0.99
1.00
2.97
1.01
1.10
1.05
3.00
f1 (ppm)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126.59
120.61
110.42
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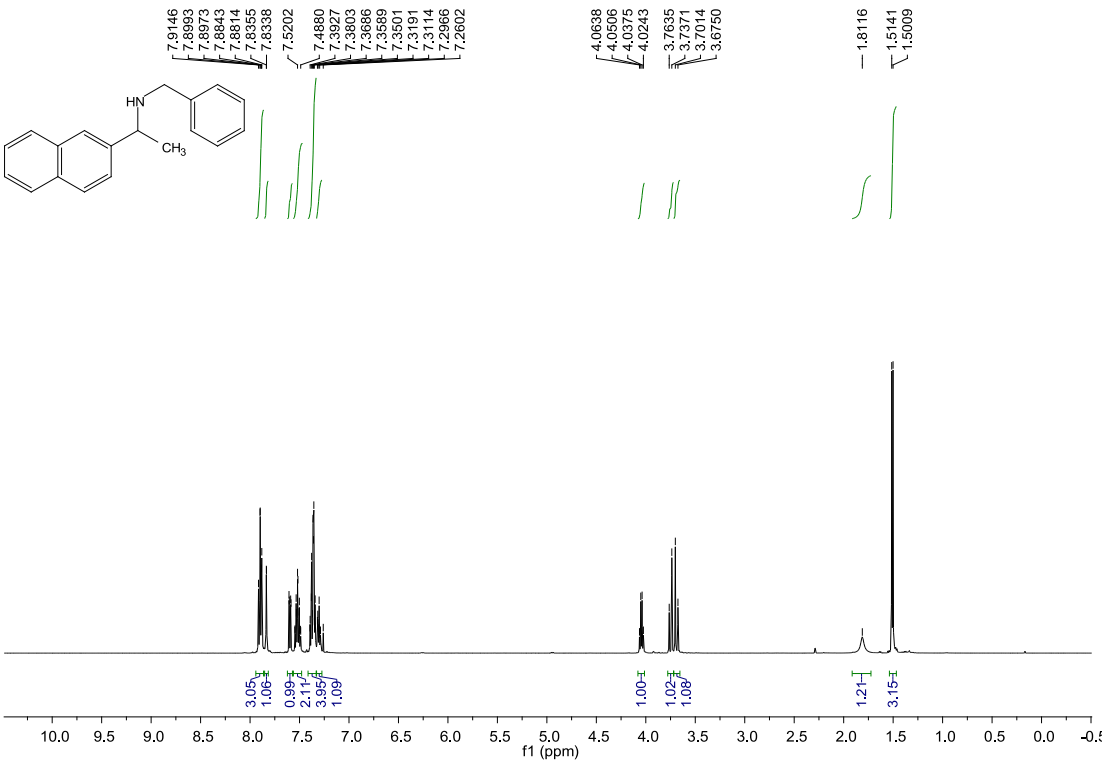
77.00
76.75
55.07
52.30
51.62
22.21
H3C
O
HN
CH3
f1 (ppm)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7.8338
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7.4880
7.3927
7.3803
7.3686
7.3589
7.3501
7.3191
7.3114
7.2966
7.2602
4.0638
4.0506
4.0375
4.0243
3.7635
3.7371
3.7014
3.6750
1.8116
1.5141
1.5009
HN
CH3
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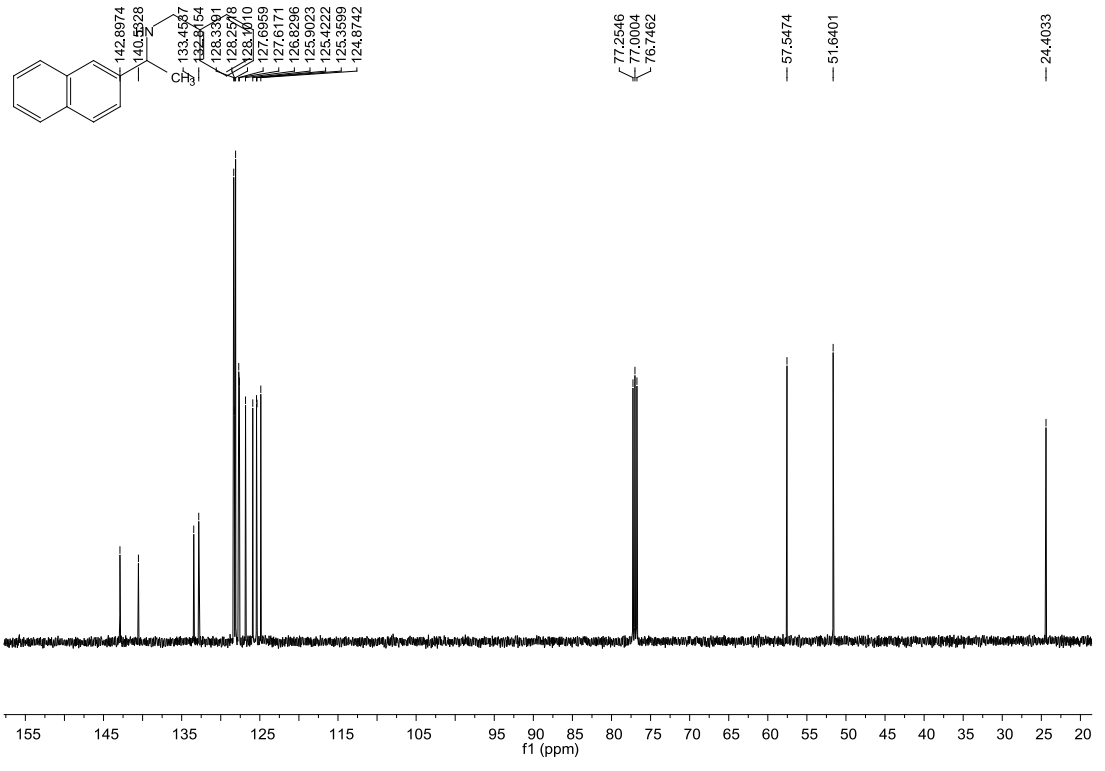
1.06
0.99
2.11
3.95
1.09
1.00
1.02
1.08
1.21
3.15
f1 (ppm)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125.4222
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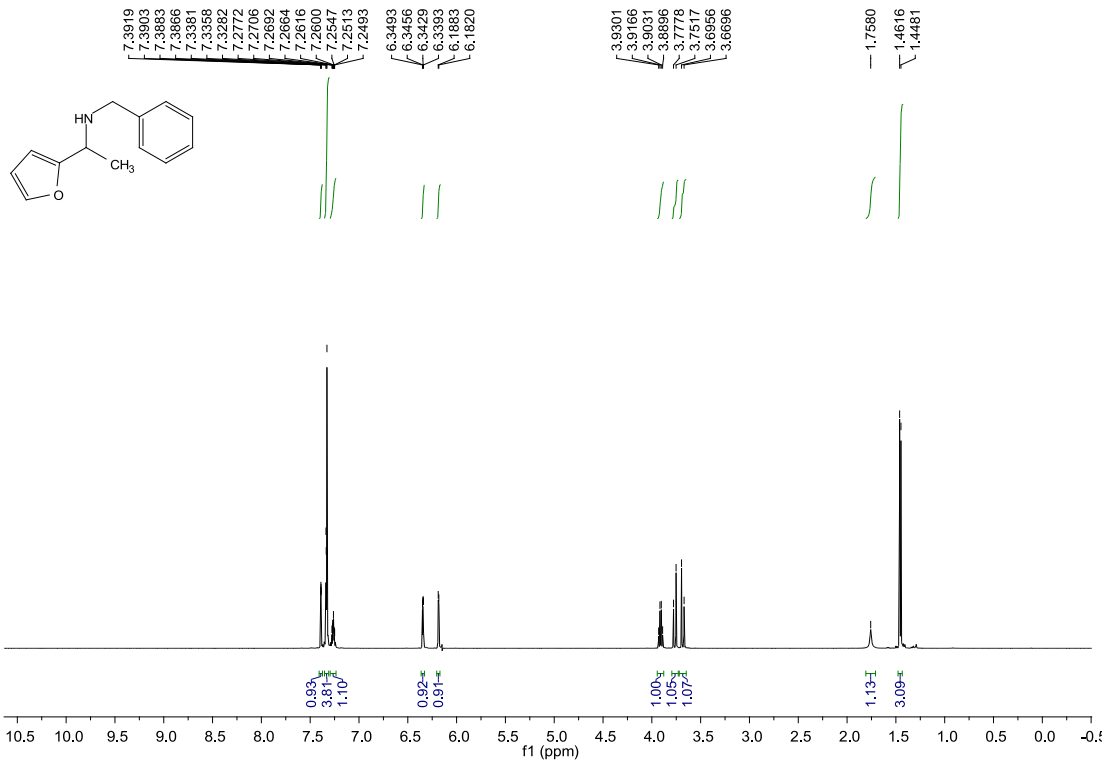
77.2546
77.0004
76.7462
57.5474
51.6401
24.4033
HN
CH3
f1 (ppm)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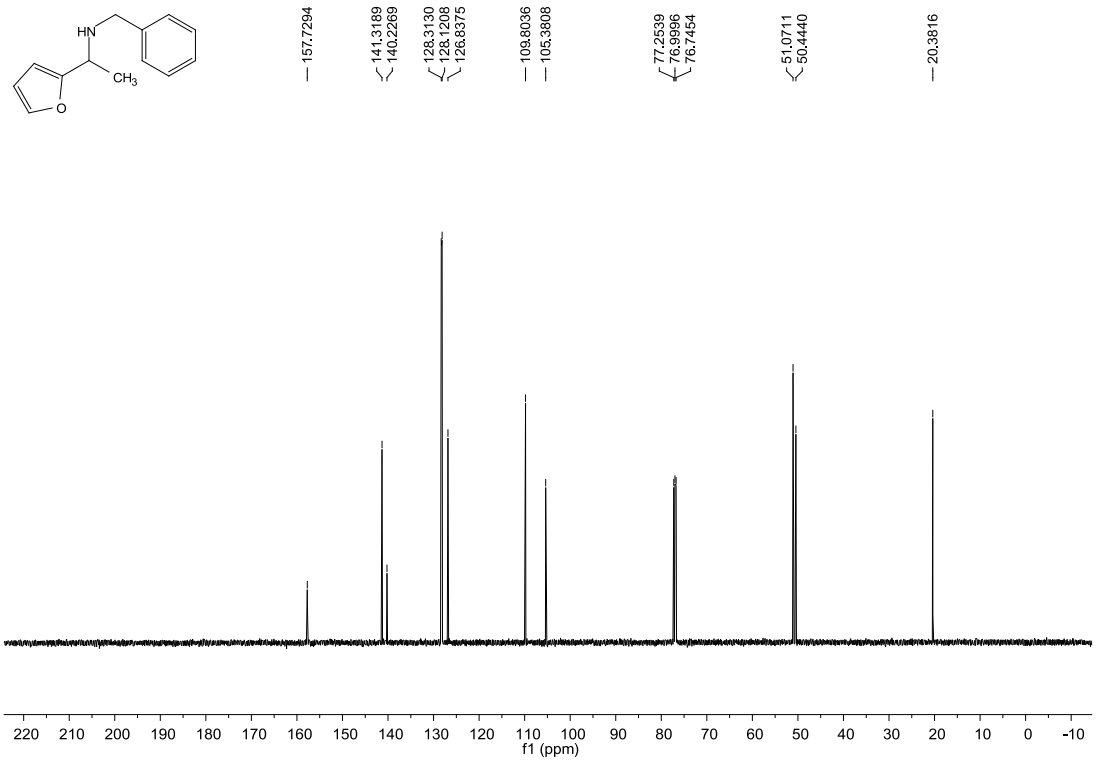
7.3883
7.3866
7.3381
7.3358
7.3282
7.2772
7.2706
7.2692
7.2664
7.2616
7.2600
7.2547
7.2513
7.2493
6.3493
6.3456
6.3429
6.3393
6.1883
6.1820
3.9301
3.9166
3.9031
3.8896
3.7778
3.7517
3.6956
3.6696
1.7580
1.4616
1.4481
HN
CH3
O
0.93
3.81
1.10
0.92
0.91
1.00
1.05
1.07
1.13
3.09
f1 (ppm)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76.9996
76.7454
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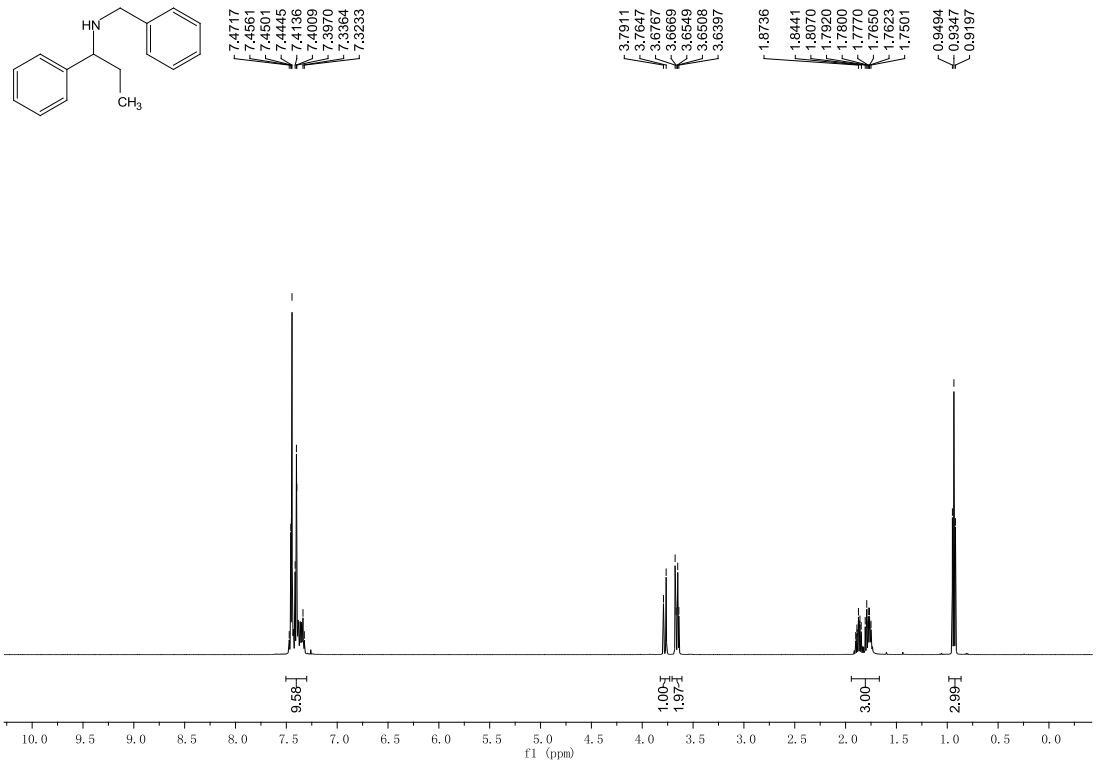
50.4440
20.3816
HN
CH3
O
f1 (ppm)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7.4009
7.3970
7.3364
7.3233
3.7911
3.7647
3.6767
3.6669
3.6549
3.6508
3.6397
1.8736
1.8441
1.8070
1.7920
1.7800
1.7770
1.7650
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0.9347
0.9197
9.58
1.00
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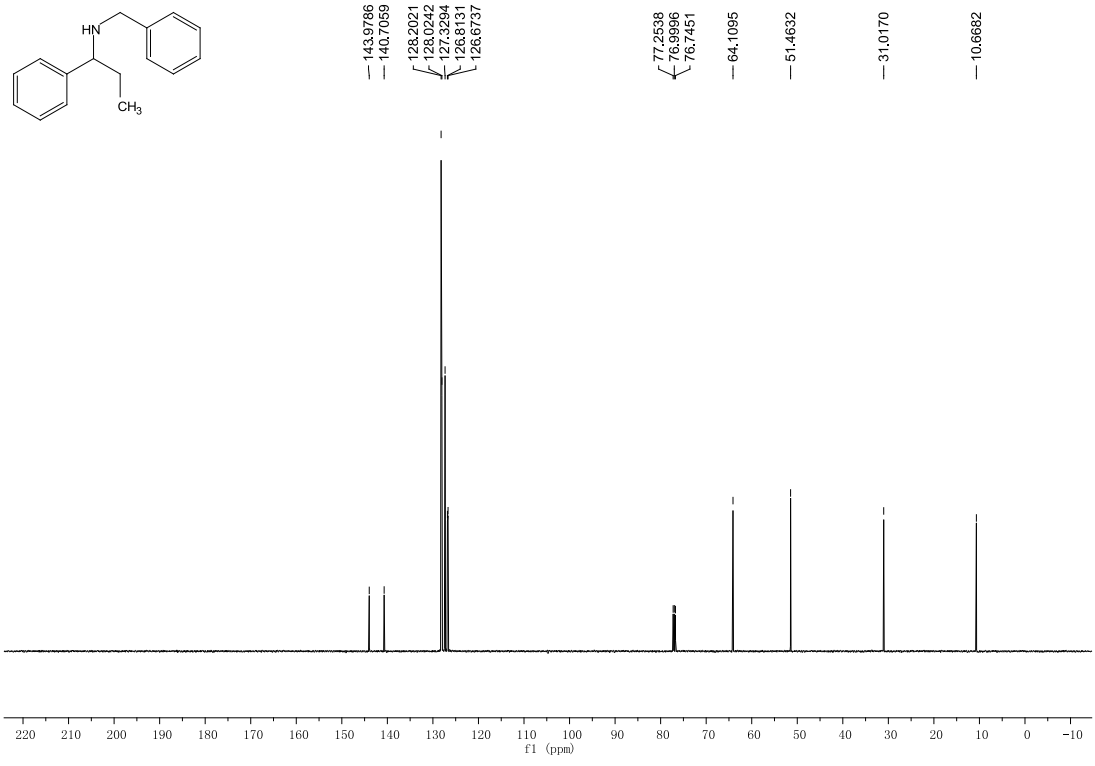
3.00
2.99
f1 (ppm)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128.0242
127.3294
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77.2538
76.9996
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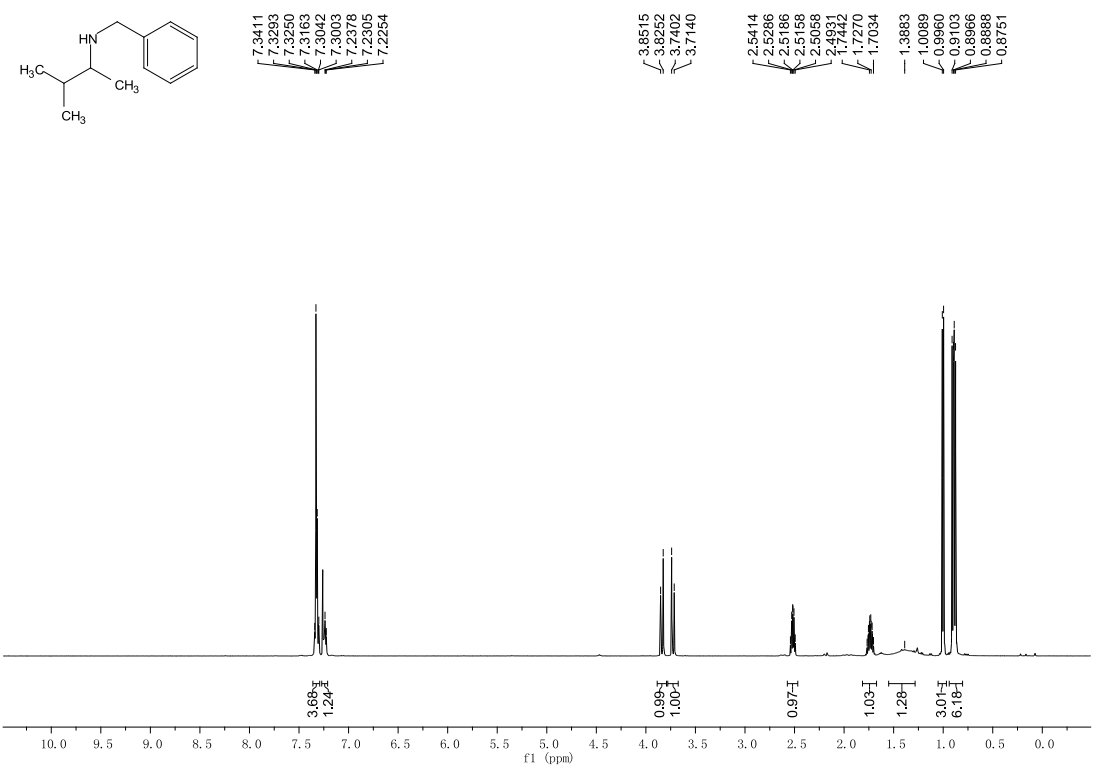
51.4632
31.0170
10.6682
f1 (ppm)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H3C
CH3
CH3
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7.3003
7.2378
7.2305
7.2254
3.8515
3.8252
3.7402
3.7140
2.5414
2.5286
2.5186
2.5158
2.5058
2.4931
1.7442
1.7270
1.7034
1.3883
1.0089
0.9960
0.9103
0.8966
0.8888
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1.24
0.99
1.00
0.97
1.03
1.28
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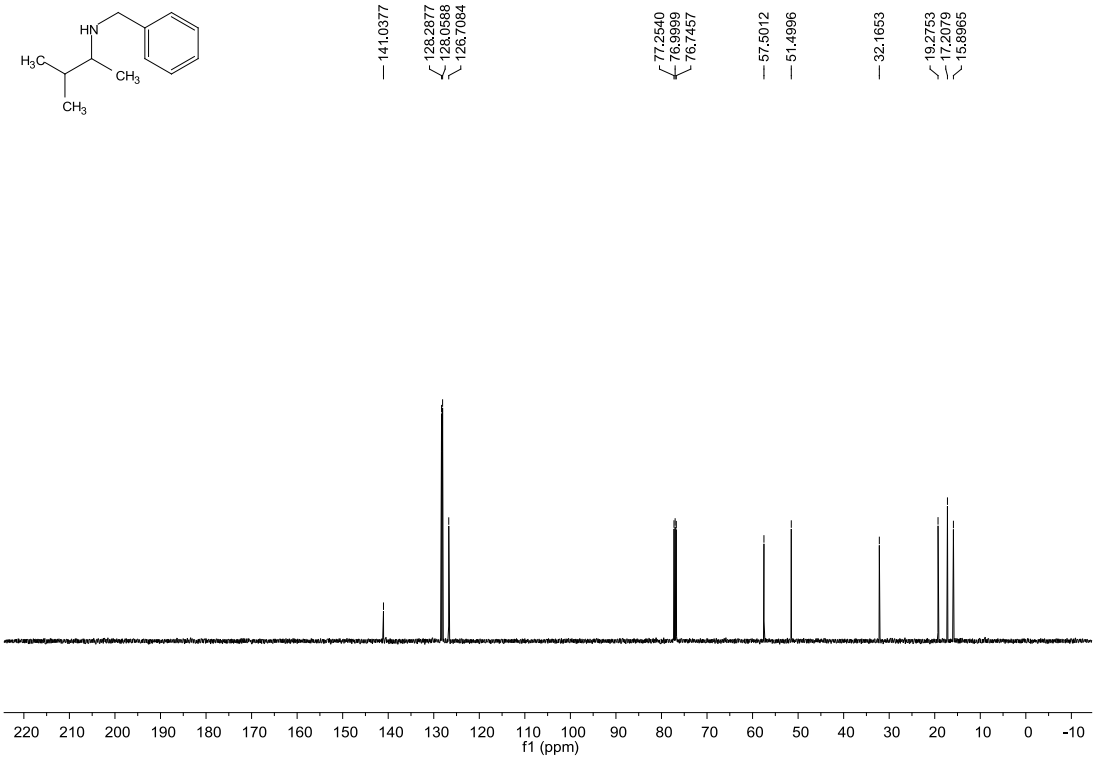
6.18
f1 (ppm)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H3C
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CH3
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126.7084
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76.7457
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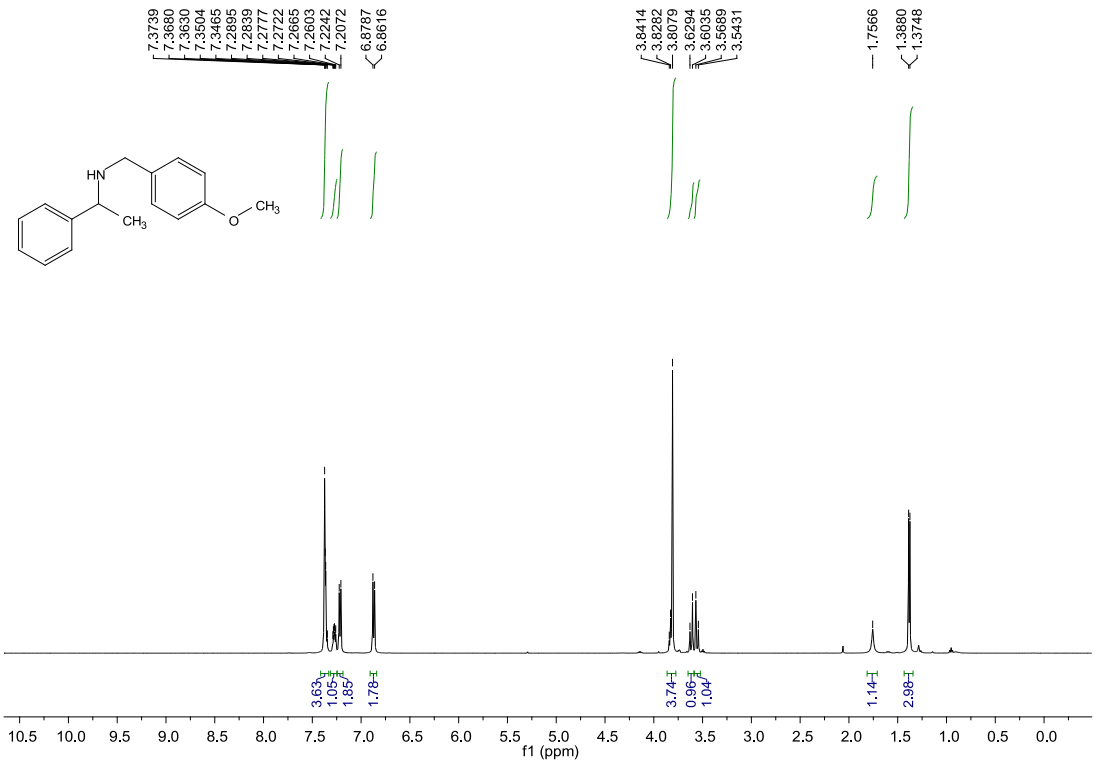
32.1653
19.2753
17.2079
15.8965
f1 (ppm)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7.3465
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7.2839
7.2777
7.2722
7.2665
7.2603
7.2242
7.2072
6.8787
6.8616
3.8414
3.8282
3.8079
3.6294
3.6035
3.5689
3.5431
1.7566
1.3880
1.3748
HN
CH3
CH3
O
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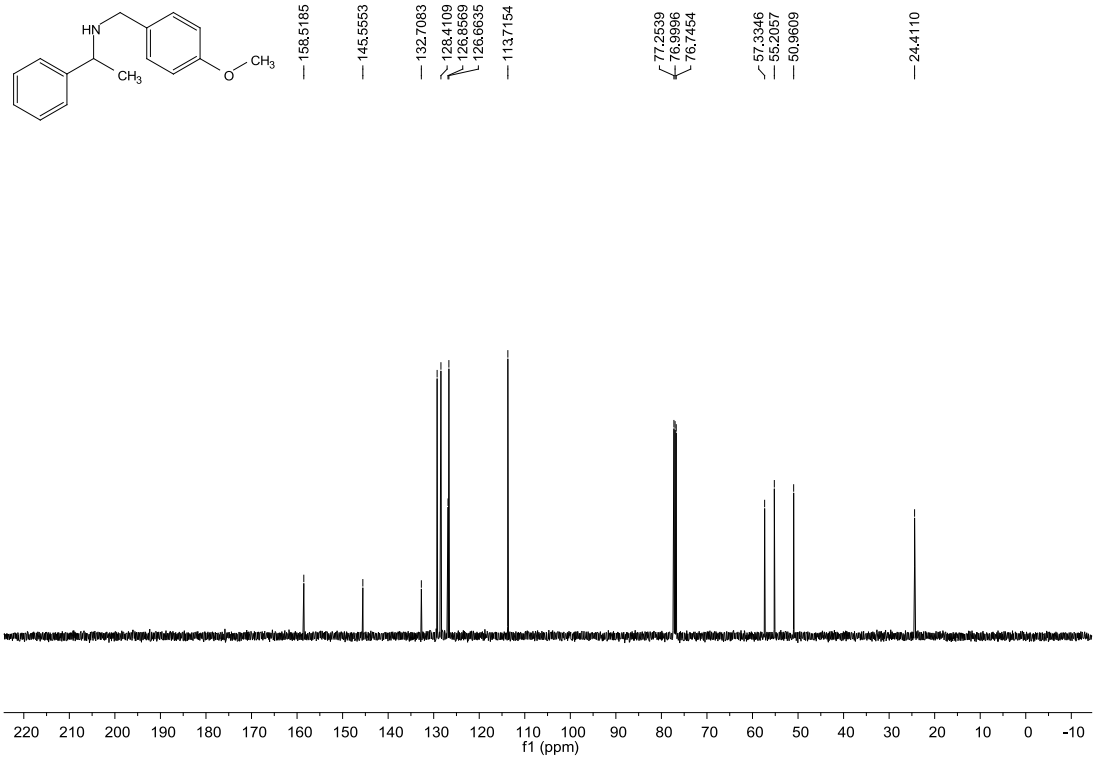
1.05
1.85
1.78
3.74
0.96
1.04
1.14
2.98
f1 (ppm)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128.4109
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113.7154
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57.3346
55.2057
50.9609
24.4110
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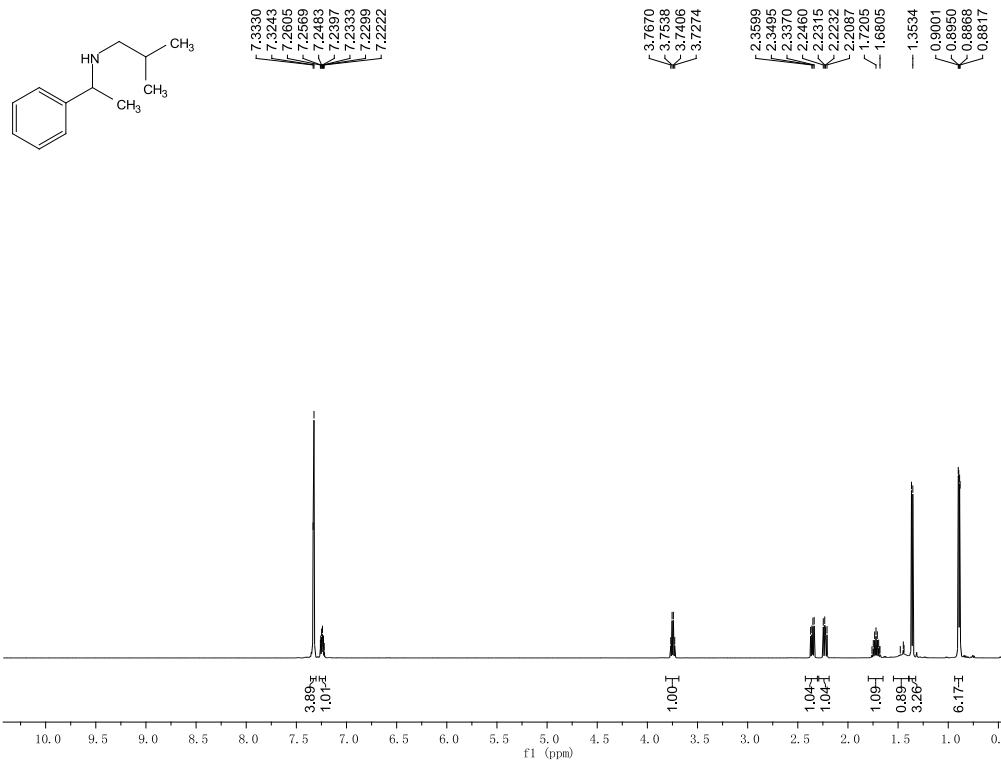
CH3
CH3
O
f1 (ppm)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CH3
CH3
CH3
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7.2569
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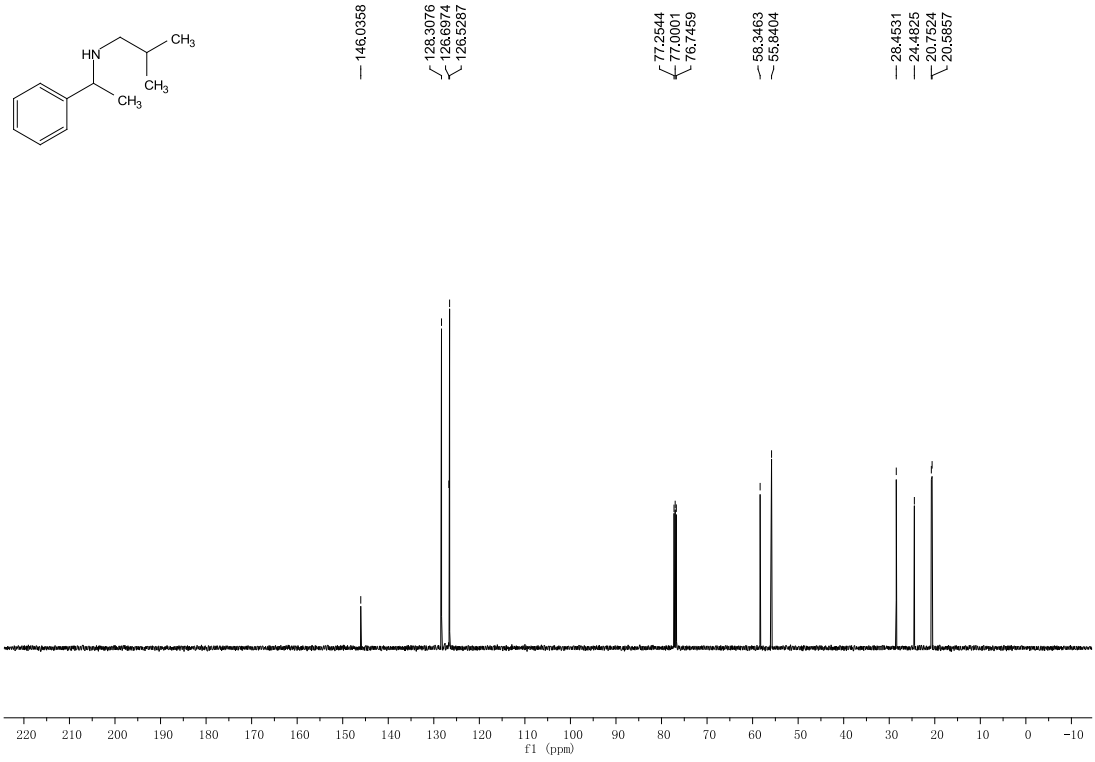
7.2397
7.2333
7.2299
7.2222
3.7670
3.7538
3.7406
3.7274
2.3599
2.3495
2.3370
2.2460
2.2315
2.2232
2.2087
1.7205
1.6805
1.3534
0.9001
0.8950
0.8868
0.8817
3.89
1.01
1.00
1.04
1.04
1.09
0.89
3.26
6.17
f1 (ppm)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CH3
CH3
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126.5287
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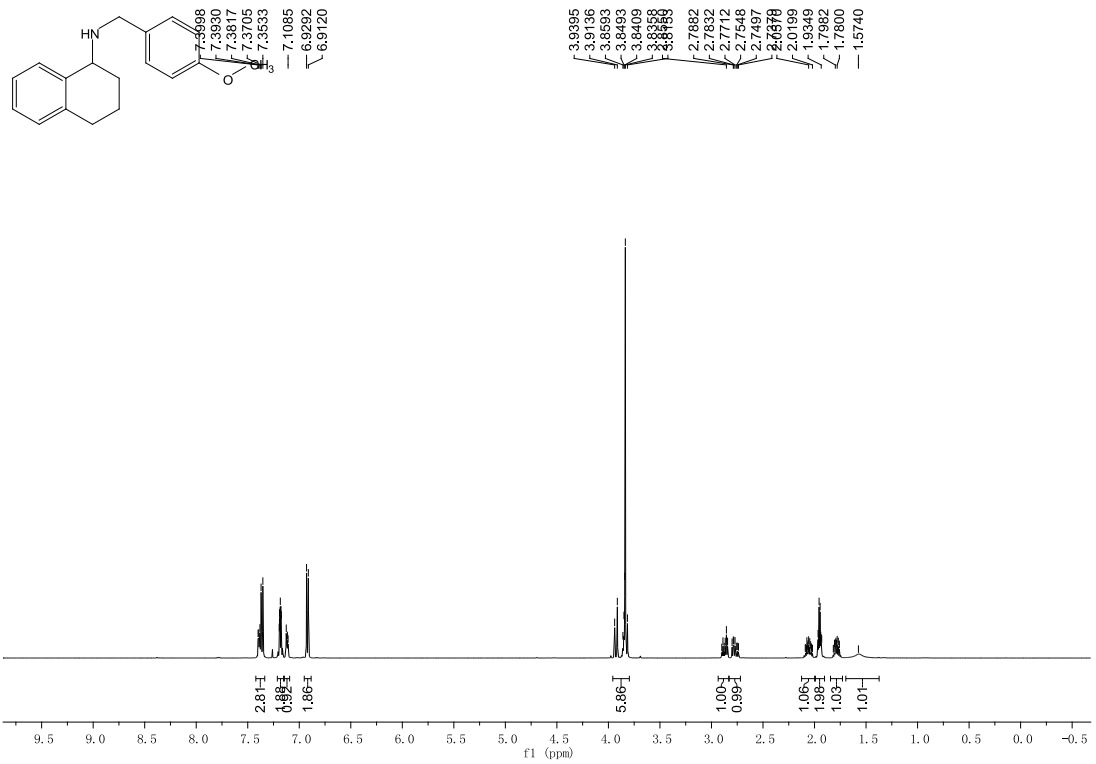
76.7459
58.3463
55.8404
28.4531
24.4825
20.7524
20.5857
f1 (ppm)


HN
O
CH3
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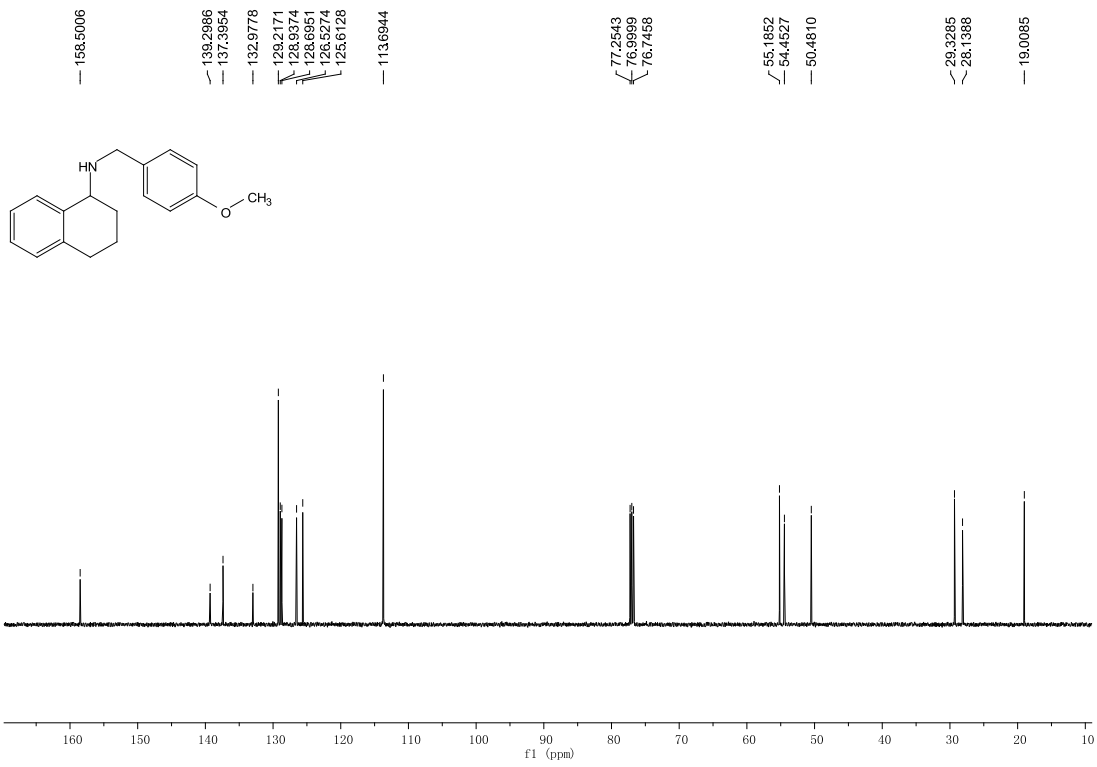
7.3705
7.3533
7.1085
6.9292
6.9120
3.9395
3.9136
3.8593
3.8493
3.8409
2.7882
2.7832
2.7712
2.7548
2.7497
2.0199
1.9349
1.7982
1.7800
1.5740
2.81
1.86
5.86
1.00
0.99
1.06
1.98
1.03
1.01
f1 (ppm)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125.6128
113.6944
77.2543
76.9999
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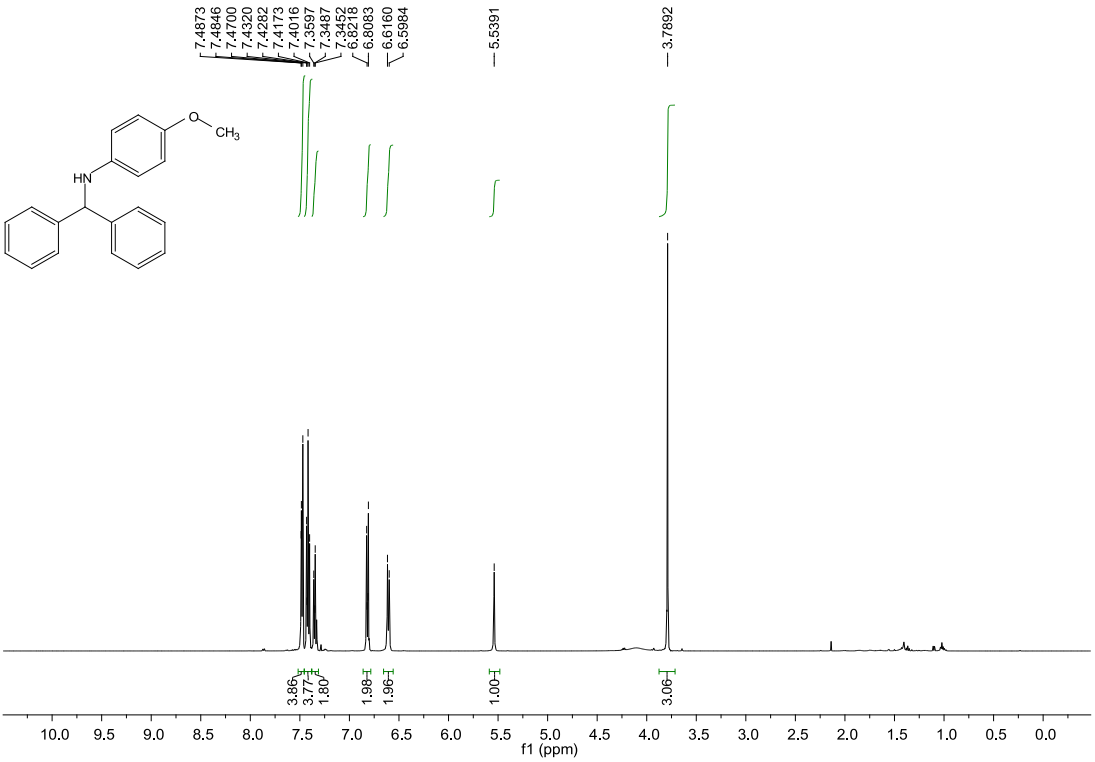
54.4527
50.4810
29.3285
28.1388
19.0085
HN
O
CH3
f1 (ppm)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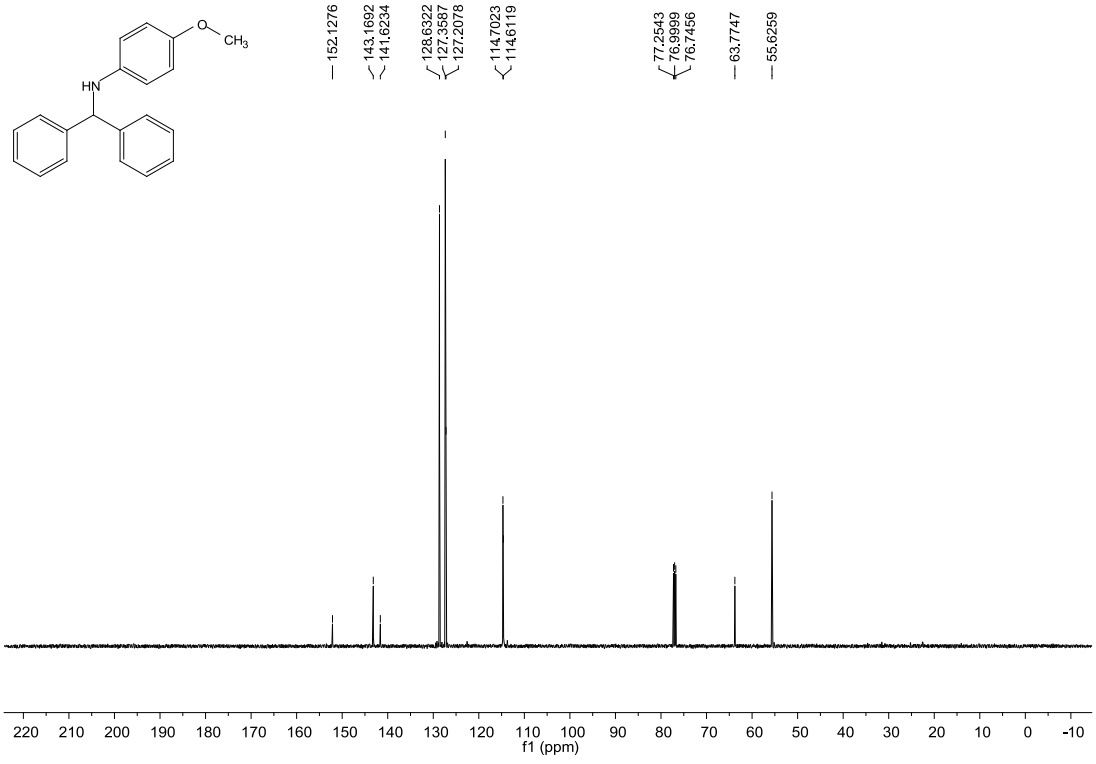
7.4320
7.4282
7.4173
7.4016
7.3597
7.3487
7.3452
6.8218
6.8083
6.6160
6.5984
5.5391
3.7892
O
CH3
HN
3.86
3.77
1.80
1.98
1.96
1.00
3.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


O
CH3
HN
152.1276
143.1692
141.6234
128.6322
127.3587
127.2078
114.7023
114.6119
77.2543
76.9999
76.7456
63.7747
55.6259
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)