Supporting Information for

Room-Temperature Cu-Catalyzed *N*-Arylation of Aliphatic Amines in Neat Water

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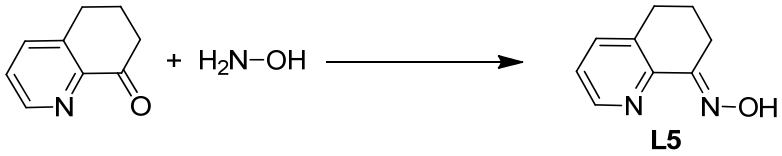
1. General Methods

Unless otherwise stated, all commercial reagents were used as purchased without further purification; all reactions were carried out in 10 mL sealed tubes, under a pure and dry Argon atmosphere. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 F254 plates. Column chromatography was performed with QDHY 60 A C.C silica gel (35-70 mm). All products were characterized by their NMR, LC/MS. NMR spectras were recorded at 25°C on a 400 MHz or 500 MHz spectrometer working at 400 MHz or 500 MHz for ^{1}H, at 100 MHz or 125 MHz for ^{13}C, respectively. The first order peak patterns are indicated as s (singlet), d (doublet), t (triplet), q (quadruplet). Complex nonfirst-order signals are indicated as m (multiplet).

2. Preparation of L1



L5

L1 was assembled according to the reported procedure (*J. Het. Chem.* **1978**, *15*, 249). 6,7-Dihydro-5*H*-quinolin-8-one (2.94 g, 20 mmol) was reacted with hydroxylamine hydrochloride (1.47 g, 21 mmol) to afford the title compound (**L1**) (3.07 g, 95%).

^{1}H NMR ($CDCl_3$): δ 10.03 (br, 1H), 8.46 (d, *J* = 3.6 Hz, 1H), 7.47 (d, *J* = 7.6, 1H), 7.18 (dd, *J*= 8.0, 4.8 Hz, 1H), 2.93 (t, *J* = 6.8 Hz, 2H), 2.81 (t, *J*= 6.0 Hz, 2H), 1.87-1.93 (m, 2H). ^{13}C NMR ($CDCl_3$): δ152.9, 148.8, 148.1, 136.8, 134.6, 123.5, 28.9, 23.8, 20.7.

3. General Procedure for the Coupling of Ar-I with Aliphatic Amines

A 10 mL sealed tube equipped with a Teflon valve was charged with a magnetic stir bar, CuI (9 mg, 5 mol%), **L1** (10 mg, 6 mol%), KOH (168 mg, 3.0 mmol) and solid aryl iodides (1.0 mmol). The tube was evacuated and backfilled with argon (this procedure was repeated three times). Under a counter flow of Argon, H_2O (1.5 mL), 1.0 mmol aryl iodides (if liquid), 1.5 mmol amines were added by syringe. The tube was sealed quickly. The reaction mixture was allowed to stir under argon at room temperature (25 ^{o}C) until completion. Then the mixture was extracted by diethyl ether (3×6 mL). The combined extracts were dried by anhydrous sodium sulfate, filtered and the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel with an eluent of petroleum ether and ethyl acetate.

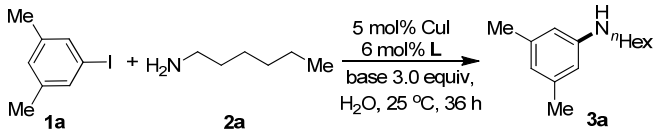
4. General Procedure for the Coupling of Ar-Br with Aliphatic Amines

A 10 mL sealed tube equipped with a Teflon valve was charged with a magnetic stir bar, CuI (9 mg, 5 mol%), **L1** (10 mg, 6 mol%), KOH (168 mg, 3.0 mmol) and solid aryl bromides (1.0 mmol). The tube was evacuated and backfilled with argon (this procedure was repeated three times). Under a counter flow of Argon, H_2O (1.5 mL), 1.0 mmol aryl bromides (if liquid), 1.5 mmol amines were added by syringe. The tube was sealed quickly. The reaction mixture was allowed to stir under argon at 65 ^{o}C until completion. The reaction mixture was cooled to room temperature, then the mixture was extracted by diethyl ether (3×6 mL). The combined extracts were dried by anhydrous sodium sulfate, filtered and the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel with an eluent of petroleum ether and ethyl acetate.

5. General Procedure for the Reuse of the CuI/L1 Catalyst System for the Coupling Reaction: A 10 mL sealed tube equipped with a Teflon valve was charged with a magnetic stir bar, CuI (9 mg, 5 mol%), **L1** (10 mg, 6 mol%), KOH (168 mg, 3.0 mmol). The tube was evacuated and backfilled with argon (this procedure was repeated three times). Under a counter flow of Argon, H_2O (1.5 mL), 1.0 mmol aryl iodides, 1.5 mmol amines were added by syringe. The tube was sealed quickly. The reaction mixture was allowed to stir under argon at room temperature (25 ^oC) until completion. Then the mixture was extracted by diethyl ether (3×6 mL). The combined extracts were dried by anhydrous sodium sulfate, filtered and the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel with an eluent of petroleum ether and ethyl acetate.

The residue mixture of CuBr/**L1** catalyst system was was evaporated under vacuo and subjected to a second run of the C-N coupling reaction by charging with the same substrates (aryl-I 1.0 mmol, amine 1.5 mmol, and KOH 2.0 mmol). The results were listed as follows:

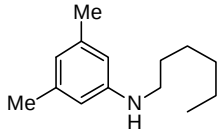
Table 1. Reuse of the CuI/L1 Catalyst System for the coupling of 1-iodo-3,5-dimethylbenzene with *n*-hexylamine.



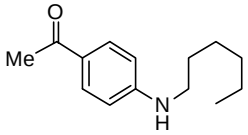
runs	yield (%)
1	91
2	86
3	60
4	43

6. Spectral and Analytic Data of 3a-4j: All compounds were identified by spectral comparison with literature data.

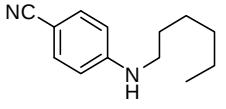
N-*n*-hexyl-3,5-dimethylbenzenamine (3a).[1] Pale yellow oil. ^1H NMR ($CDCl_3$): δ 6.5 (s, 1H), 6.38 (s, 2H), 3.56 (s, 1H), 3.19 (t, *J*= 7.2 Hz, 2H), 2.39 (s, 6H), 1.77-1.69 (m, 2H), 1.57-1.43 (m, 6H), 1.04 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 148.8, 138.9, 119.3, 110.9, 44.2, 31.8, 29.8, 27.0, 22.8, 21.6, 14.2.



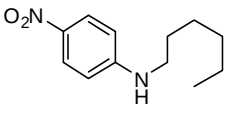
N-*n*-hexyl-4-acety-benzenamine (3b). [2] White solid. ^1H NMR ($CDCl_3$): δ 7.81 (d, *J*= 8.8 Hz, 2H), 6.55 (d, *J*= 8.8 Hz, 2H), 4.47 (br s, 1H), 3.15 (t, *J*= 7.6 Hz, 2 H), 2.49 (s, 3H), 1.67-1.59 (m, 2H), 1.42-1.27 (m, 6H), 0.91 (m, 3 H); ^{13}C NMR ($CDCl_3$): δ 196.3, 152.4, 130.8, 126.4, 111.3, 43.4, 31.6, 29.2, 26.7, 25.9, 22.6, 14.0.



N-*n*-hexyl-4-benzonitrile (3c). [3] Pale yellow oil. ^1H NMR ($CDCl_3$): δ 7.39 (d, *J*= 8.8 Hz, 2H), 6.55 (d, *J*= 8.4 Hz, 2H), 4.51 (s, 1H), 3.11 (t, *J*= 8.8 Hz, 2H), 1.66-1.59 (m, 2H), 1.43-1.25 (m, 6H), 0.88 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 151.3, 133.7, 120.5, 112.3, 98.5, 43.5, 31.5, 29.1, 26.7, 22.6, 14.0.

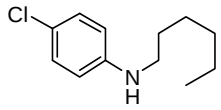


N-hexyl-4-nitroaniline (3d). [4] Pale yellow solid. ^1H NMR ($CDCl_3$): δ 8.09 (d, *J*= 9.2 Hz, 2H), 6.54 (d, *J*= 9.2 Hz, 2H), 3.22 (t, *J*= 7.2 Hz, 2H), 1.68 (t, *J*= 7.2 Hz, 2H), 1.43 (m, 7H), 0.92 (t, *J*= 6.8 Hz, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 153.3, 138.0, 126.4,

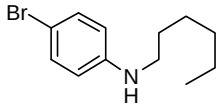


110.0, 43.6, 31.5, 29.1, 26.7, 22.6, 14.0.

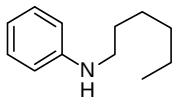
N-*n*-hexyl-4-chloroaniline (3e). [3] Pale purple oil. ^{1}H NMR ($CDCl_3$): δ 7.11 (dd, *J*= 6.8, 2.4 Hz, 2H), 6.51 (dd, *J*= 6.8, 2.0 Hz, 2H), 3.59 (s, 1H), 3.06 (t, *J*= 7.2 Hz, 2H), 1.65-1.58 (m, 2H), 1.45-1.30 (m, 6H), 0.92 (t, *J*= 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 147.1, 129.0, 121.6, 113.8, 44.2, 31.7, 29.4, 26.8, 22.7, 14.1.



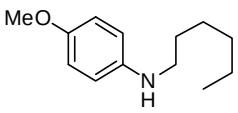
4-Bromo-N-hexylaniline (3f). [5] Pale yellow solid. ^{1}H NMR ($CDCl_3$): δ 7.25 (t, *J*= 8.4 Hz, 2H), 6.48 (d, *J*= 8.4 Hz, 2H), 3.74 (br, 1H), 3.07 (t, *J*= 7.2, Hz, 2H), 1.63 (m, 2H), 1.42 (m, 7H), 0.91 (t, *J*= 6.8, Hz, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 147.4, 131.9, 114.3, 108.7, 44.1, 31.6, 29.4, 26.8, 22.6, 14.0.



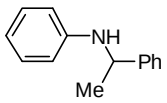
N-*n*-hexylbenzenamine (3g).[3] Pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 7.21-7.17 (m, 2H), 6.69 (t, *J*= 7.6 Hz, 1H), 6.63 (d, *J*= 7.6 Hz, 2H), 3.73 (s, 1H), 3.10 (t, *J*= 7.2 Hz, 2H), 1.67-1.60 (m, 2H), 1.46-1.29 (m, 6H), 0.91 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 148.3, 129.2, 117.3, 112.9, 44.2, 31.7, 29.5, 26.9, 22.7, 14.1.



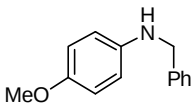
N-*n*-hexyl-4-methoxybenzenamine (3h).[3] Pale-yellow oil. ^{1}H NMR ($CDCl_3$): δ 6.82 (d, *J*=8.8 Hz, 2H), 6.62 (d, *J*= 8.8 Hz, 2H), 3.79 (s, 3H), 3.34 (s, 1H), 3.08 (t, *J*= 7.2 Hz, 2H), 1.63 (m, 2H), 1.47-1.35 (m, 6H), 0.94 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 152.1, 142.8, 114.9, 114.1, 55.8, 45.1, 31.7, 29.7, 26.9, 22.7, 14.1.



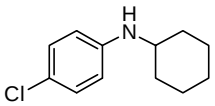
(S)-N-Phenyl-*α*-methylbenzylamine (3i). [1] Pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 7.49-7.41 (m, 4H), 7.32 (t, *J*= 7.2 Hz, 1H), 7.19 (t, *J*= 8.0 Hz, 2H) 6.76 (t, *J*= 7.6 Hz, 1H), 6.63 (d, *J*= 8.0 Hz, 2H), 4.58 (m, 1H), 4.19 (s, 1H), 1.62 (d, *J*= 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$) : δ 147.3, 145.3, 129.2, 128.8, 126.9, 126.9, 117.4, 113.5, 53.6, 25.1.



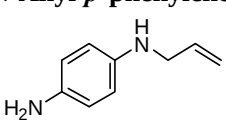
N-benzyl-4-methoxybenzenamine (3j). [6] White solid. ^{1}H NMR ($CDCl_3$): δ 7.43-7.33 (m, 5H), 6.84 (d, *J*= 8.8 Hz, 2H), 6.65 (d, *J*= 8.8 Hz, 2H), 4.33 (s, 2H), 3.79 (s, 3H); ^{13}C NMR ($CDCl_3$) : δ 152.3, 142.5, 139.8, 128.7, 127.7, 127.3, 115.0, 114.3, 55.7, 49.3.



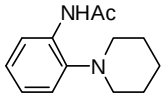
N-cyclohexyl-4-chloro-benzenamine (3k). [7] White solid. ^{1}H NMR ($CDCl_3$): δ 7.28-7.10 (m, 2H), 6.53 (m, 2H), 3.61 (br s, 1H), 3.27-3.19 (m, 1H), 2.08-2.05 (m, 2H), 1.82-1.77 (m, 2H), 1.71-1.66 (m, 1H), 1.45-1.24 (m, 5H); ^{13}C NMR ($CDCl_3$): δ 145.9, 129.1, 121.4, 114.3, 51.9, 33.3, 25.9, 24.9.



N-Allyl-*p*-phenylenediamine (3l). [8] Brown oil. ^{1}H NMR ($CDCl_3$): δ 6.66-6.55 (m, 4H), 6.01-5.92 (m, 1H), 5.24 (m, 1H), 5.13 (m, 1H), 3.72 (br s, 2H), 4.58 (br s, 3H); ^{13}C NMR ($CDCl_3$) : δ 140.9, 137.9, 135.8, 116.9, 116.3, 115.1, 47.9.

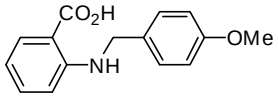


N-[2-(1-piperidinyl)phenyl]-acetamide (3m). [8] Pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.54 (br s, 1H),



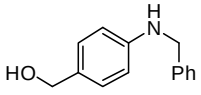
8.33 (d, *J*= 7.6 Hz, 1H), 7.14-7.02 (m, 3H), 2.79 (br s, 4H), 2.21 (s, 3H), 1.74 (br s, 4H), 1.62 (br s, 2H); ^{13}C NMR ($CDCl_3$): δ 168.0, 142.4, 133.5, 124.9, 123.5, 120.3, 119.3, 53.7, 26.9, 24.9, 24.0.

2-(4-methoxybenzylamino)benzoic acid (3n). [9] White solid. ^{1}H NMR ($CDCl_3$): δ 7.79 (d, *J*= 7.6 Hz,



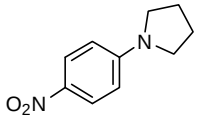
1H), 7.33-7.27 (m, 3H), 6.90 (d, *J*= 8.4 Hz, 2H), 6.69 (d, *J*= 8.4 Hz, 1H), 6.54 (t, *J*= 7.2 Hz, 1H), 4.36 (s, 2H), 3.73 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 158.8, 151.1, 134.8, 132.1, 131.5, 128.9, 114.9, 114.4, 112.1, 111.5, 55.5, 45.9.

(4-(Benzylamino)phenyl)methanol (3o). [10] Colorless oil. ^{1}H NMR ($CDCl_3$): δ 7.37 (m, 4H), 7.29 (m,



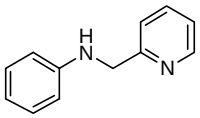
1H), 7.18 (d, *J*= 8.4 Hz, 2H), 4.53 (s, 2H), 4.33 (s, 2H); ^{13}C-NMR ($CDCl_3$): δ 147.7, 139.2, 130.1, 128.9, 128.7, 127.5, 127.3, 113.0, 65.4, 48.4.

4-pyrrolidinonitrobenzene (3p). [11] Yellow solid. ^{1}H NMR (*d*-DMSO): δ 8.02 (d, *J*= 9.6 Hz, 2H),



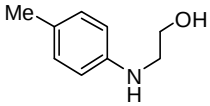
6.56 (d, *J*= 9.2 Hz, 2H), 3.34 (t, *J*= 5.6 Hz, 4H), 1.96 (t, *J*= 6.8 Hz, 4H); ^{13}C NMR (*d*-DMSO): δ 152.3, 135.7, 126.4, 111.3, 48.2, 25.4.

N-(Pyridin-2-ylmethyl)aniline (3q). [12] White solid. ^{1}H NMR ($CDCl_3$): δ 8.59 (d, *J*= 4.4 Hz, 1H),



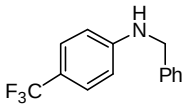
7.67 (t, *J*= 7.6 Hz, 1H), 7.35 (d, *J*= 8.0 Hz, 1H), 6.74 (t, *J*= 7.6 Hz, 1H), 6.68 (d, *J*= 8.0 Hz, 2H), 4.47 (s, 2H). ^{13}C-NMR ($CDCl_3$): δ 158.5, 149.1, 147.9, 136.7, 129.3, 122.1, 121.6, 117.6, 113.1, 49.3.

2-*p*-Tolylaminoethanol (3r). [10] Pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 7.03 (d, *J*= 8.0 Hz, 2H), 6.60 (d,



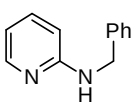
J= 8.4 Hz, 2H), 3.78 (t, *J*= 5.2 Hz, 2H), 3.43 (br s, 2H), 3.24 (t, *J*= 5.2 Hz, 2H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 145.8, 129.8, 127.3, 113.7, 61.1, 46.7, 20.4.

N-Benzyl-4-(trifluoromethyl)aniline (3s). [10] White solid. ^{1}H NMR ($CDCl_3$): δ 7.40 (d, *J*= 8.4 Hz, 2H),



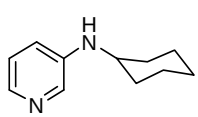
7.36 (m, 4H), 7.31 (m,1H), 6.65 (d, *J*= 8.4 Hz, 2H), 4.37 (s, 2H). ^{13}C-NMR ($CDCl_3$): δ 150.3, 138.3, 128.8, 127.6, 127.4, 126.6, 126.3, 123.6, 112.2, 47.9.

N-Benzylpyridin-2-amine (3t). [13] Pale yellow solid. ^{1}H NMR ($CDCl_3$): δ 8.11 (d, *J*= 4.4 Hz, 1H),

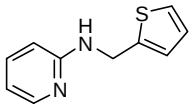


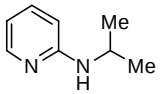
7.43 (m, 5H), 7.29 (m, 1H), 6.61 (dd, *J*= 6.8, 5.6 Hz, 1H), 6.39 (d, *J*= 8.4 Hz, 1H), 4.99 (br, 1H), 4.52 (d, *J*= 6.0 Hz, 2H). ^{13}C-NMR ($CDCl_3$): δ 158.5, 147.9, 139.1, 137.6, 128.7, 127.4, 127.3, 113.2, 106.9, 46.3.

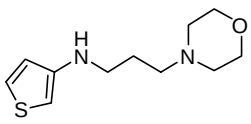
3-(Cyclohexylamino)pyridine (3u). [1] Whit solid. ^{1}H NMR ($CDCl_3$): δ 8.01 (br s, 1H), 7.91 (br s,

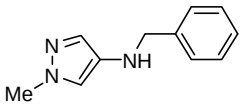


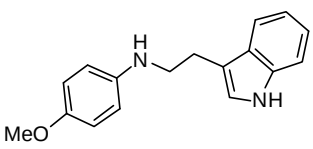
1H), 7.05 (br s, 1H), 6.84 (d, *J*= 7.6 Hz, 1H), 3.70 (br s, 1H), 3.28-3.21 (m, 1H), 2.05-2.02 (m, 2H), 1.68-1.63 (m, 1H), 1.43-1.26 (m, 5H); ^{13}C NMR ($CDCl_3$): δ 143.5, 137.9, 136.1, 123.8, 118.8, 51.4, 33.2, 25.8, 24.9.

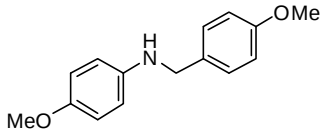
2-[N-(2-thiophenmethyl)]-pyridinamine (3v). [8] White solid. ^{1}H NMR ($CDCl_3$): δ 8.13 (m, 1H),  7.46-7.41 (m, 1H), 7.21 (dd, *J*= 5.2 1.2 Hz, 1H), 7.02 (m, 1H), 6.96 (m, 1H), 6.61 (dd, *J*= 6.0, 0.4 Hz, 1H), 6.44 (d, *J*= 8.0 Hz, 1H), 5.08 (br s, 1H), 4.71 (d, *J*= 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$): δ 158.1, 147.9, 142.6, 137.5, 126.8, 125.2, 124.6, 113.5, 107.4, 41.3.

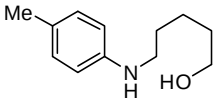
N-Isopropylpyridin-2-amine (3w). [14] Pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.07 (d, *J*= 4.4 Hz, 1H),  7.43 (m, 1H), 6.55 (dd, *J*= 6.8, 5.6 Hz, 1H), 6.37 (d, *J*= 8.4 Hz, 1H), 4.45 (br, 1H), 3.93 (m, 1H), 1.24 (d, *J*= 6.4 Hz, 6H). ^{13}C-NMR ($CDCl_3$): δ 158.1, 148.0, 137.5, 112.4, 106.8, 43.1, 23.0.

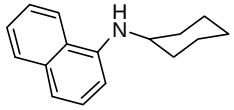
3-[N-(3-morpholinopropyl)]thiophenamine (3x). [8] Brown oil. ^{1}H NMR ($CDCl_3$): δ 7.15 (dd, *J*=4.8  2.8 Hz, 1H), 6.62 (dd, *J*=5.2 1.6 Hz, 1H), 5.95 (m, 1H), 3.73 (m, 4H), 3.16 (t, *J*=6.4 Hz, 2H), 2.51 (m, 6H), 1.87-1.81 (m, 2H); ^{13}C NMR ($CDCl_3$) : δ 148.9, 125.1, 119.9, 95.2, 66.9, 57.6, 53.7, 45.5, 25.5.

4-(N-benzyl)-1-methyl-1H- pyrazolamine (3y). [8] Brown oil. ^{1}H NMR ($CDCl_3$): δ 7.39-7.28 (m, 5H),  7.14 (s, 1H), 6.86 (s, 1H), 4.16 (s, 2H), 3.78 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 139.6, 134.1, 128.7, 128.5, 127.7, 127.2, 116.7, 52.2, 39.1.

N-(2-(1H-indol-3-yl)ethyl)-4-methoxyaniline (3z). [15] Brown solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ  8.13 (br., 1H), 7.69 (d, *J*= 8.0 Hz, 1H), 7.39 (d, *J*= 8.0 Hz, 1H), 7.29 (t, *J*= 7.5 Hz, 1H), 7.21 (t, *J*= 7.5 Hz, 1H), 7.05 (s, 1H), 6.86 (d, *J*= 8.5 Hz, 2H), 6.66 (d, *J*= 8.5 Hz, 2H), 3.81 (s, 3H), 3.53 (t, *J*= 5.6 Hz, 2H), 3.14 (t, *J*=6.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.2, 142.6, 136.5, 127.5, 122.2, 119.4, 118.9, 115.0, 114.5, 113.4, 111.3, 55.9, 45.1, 25.2.

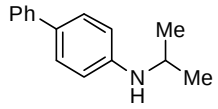
N-(4-methoxybenzyl)- 4-methoxy-benzenamine (4b). [16] White solid. ^{1}H NMR ($CDCl_3$): δ 7.31 (d,  *J*= 8.4 Hz, 2H), 6.89 (d, *J*= 8.4 Hz, 2H), 6.79 (d, *J*= 9.2 Hz, 2H), 6.65 (d, *J*= 9.2 Hz, 2H), 4.24 (s, 2H), 3.83 (s, 3H), 3.77 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 158.9, 152.5, 142.0, 131.4, 128.9, 114.9, 114.5, 114.0, 55.8, 55.3, 48.9.

5-[(4-methylphenyl)amino]-1-Pentanol (4c). [14] Yellow oil. ^{1}H NMR ($CDCl_3$): δ 7.01 (d, *J*= 8.0 Hz,  2H), 6.56 (d, *J*= 8.0 Hz, 2H), 3.61 (t, *J*= 6.8 Hz, 2H), 3.09 (t, *J*= 6.8 Hz, 2H), 2.92 (br s, 2H), 2.27 (s, 3H), 1.68-1.57 (m, 4H), 1.51-1.45 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 146.2, 129.8, 126.6, 113.2, 62.6, 44.4, 32.5, 29.4, 23.4, 20.4.

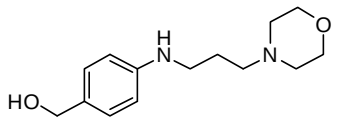
1-(N-cyclohexyl)-naphthalenamine (4d). [17] Colorless oil. ^{1}H NMR ($CDCl_3$): δ 7.87-7.83 (m, 2H),  7.52-7.45 (m, 2H), 7.38 (t, *J*= 8.0 Hz, 1H), 7.25 (d, *J*= 8.0 Hz, 1H), 6.71 (d, *J*= 7.6 Hz, 1H), 4.38 (br s, 1H), 3.57-3.49 (m, 1H), 2.22 (m, 2H), 2.19-1.85 (m, 2H), 1.78-1.73 (m, 1H), 1.56-1.41 (m, 5H); ^{13}C NMR ($CDCl_3$) : δ 142.3, 134.6,

128.7, 126.7, 125.6, 124.5, 123.5, 119.9, 116.8, 104.9, 51.9, 33.3, 26.1, 25.1.

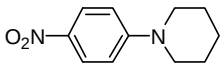
N-Isopropyl-[1,1'-biphenyl]-4-amine (4e). [18] Pale yellow solid. ^{1}H NMR ($CDCl_3$): δ 7.54 (d, *J*= 7.6 Hz, 2H), 7.44 (d, *J*= 8.8 Hz, 2H), 7.40 (t, *J*= 8.0 Hz, 2H), 7.26 (t, *J*= 8.0 Hz, 1H), 6.67 (d, *J*= 8.4 Hz, 2H), 3.72 (m, 1H), 1.25 (d, *J*= 6.4 Hz, 6H); ^{13}C-NMR ($CDCl_3$): δ 146.8, 141.3, 130.0, 128.6, 128.0, 126.3, 126.0, 113.6, 44.4, 23.0.



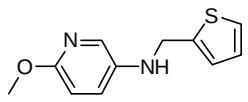
4-(3-morpholinopropylamino)-benzylnol (4f). [8] White solid. ^{1}H NMR (*d*-DMSO): δ 7.00 (d, *J*= 8.0 Hz, 2H), 6.49 (d, *J*= 8.4 Hz, 2H), 5.5 (br s, 1H), 4.77 (t, *J*= 5.2 Hz, 1H), 4.29 (d, *J*= 5.2 Hz, 2H), 3.58 (s, 4H), 3.03 (br s, 2H), 2.36 (s, 6H), 1.66 (t, *J*= 6.8 Hz, 2H); ^{13}C NMR (*d*-DMSO): δ 148.5, 129.8, 128.4, 112.1, 66.7, 63.6, 56.7, 53.9, 41.8, 26.1.



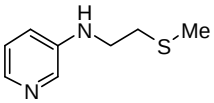
1-(4-Nitrophenyl)piperidine (4g). [11] Yellow solid. ^{1}H NMR ($CDCl_3$): δ 8.12 (d, *J*= 9.6 Hz, 2H), 6.83 (d, *J*= 9.6 Hz, 2H), 3.44 (s, 4H), 1.70 (s, 7H); ^{13}C-NMR ($CDCl_3$): δ 154.7, 137.8, 126.1, 112.7, 48.7, 25.2, 24.2.



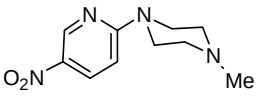
3-[N-(2-methylthiophen)]-6-methoxy-pyridinamine (4h. [8] White solid. ^{1}H NMR ($CDCl_3$): δ 7.66 (s, 1H), 7.22 (d, *J*= 5.2 Hz, 1H), 7.06-6.96 (m, 3H), 6.63 (d, *J*= 8.8 Hz, 1H), 4.48 (s, 2H), 3.88 (s, 3H), 3.74 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ 157.8, 142.5, 138.4, 130.9, 126.9, 126.3, 125.2, 124.8, 110.8, 53.4, 44.4.



3-[N-(2-(methylthio)ethyl)]-pyridineamine (4i). [8] Yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.04 (s, 1H), 7.94 (d, *J*= 3.6 Hz, 1H), 7.08-7.05 (m, 1H), 6.87 (d, *J*= 8.0 Hz, 1H), 4.28 (br s, 1H), 3.29 (t, *J*= 6.4 Hz, 2H), 2.73 (t, *J*= 6.4 Hz, 2H), 2.09 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 143.9, 138.8, 135.9, 123.8, 118.9, 41.5, 33.4, 15.0.



2-(N-Methylpiperazino)-5-nitropyridine (4j). [19] Yellow solid. ^{1}H NMR ($CDCl_3$): δ 9.03 (t, *J*= 2.4 Hz, 1H), 8.19 (m, 1H), 6.57 (dd, *J*= 9.6 0.8 Hz, 1H), 3.83 (d, *J*= 4.4 Hz, 4H), 2.57 (d, *J*= 4.0 Hz, 4H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 160.3, 146.4, 135.1, 133.0, 104.6, 54.5, 45.9, 44.7.



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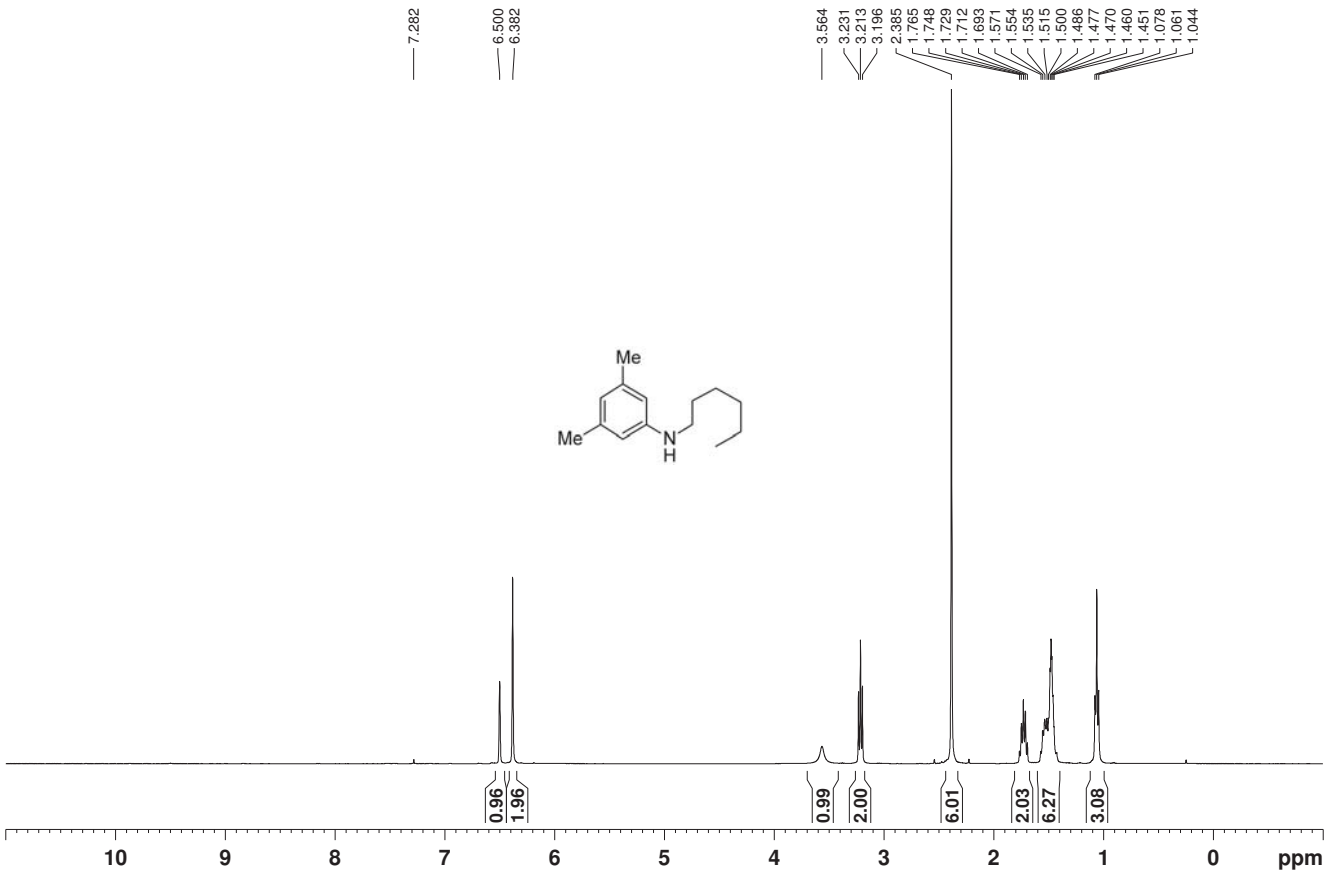
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1.486
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1.470
1.460
1.451
1.078
1.061
1.044
Me
Me
N
H
0.96
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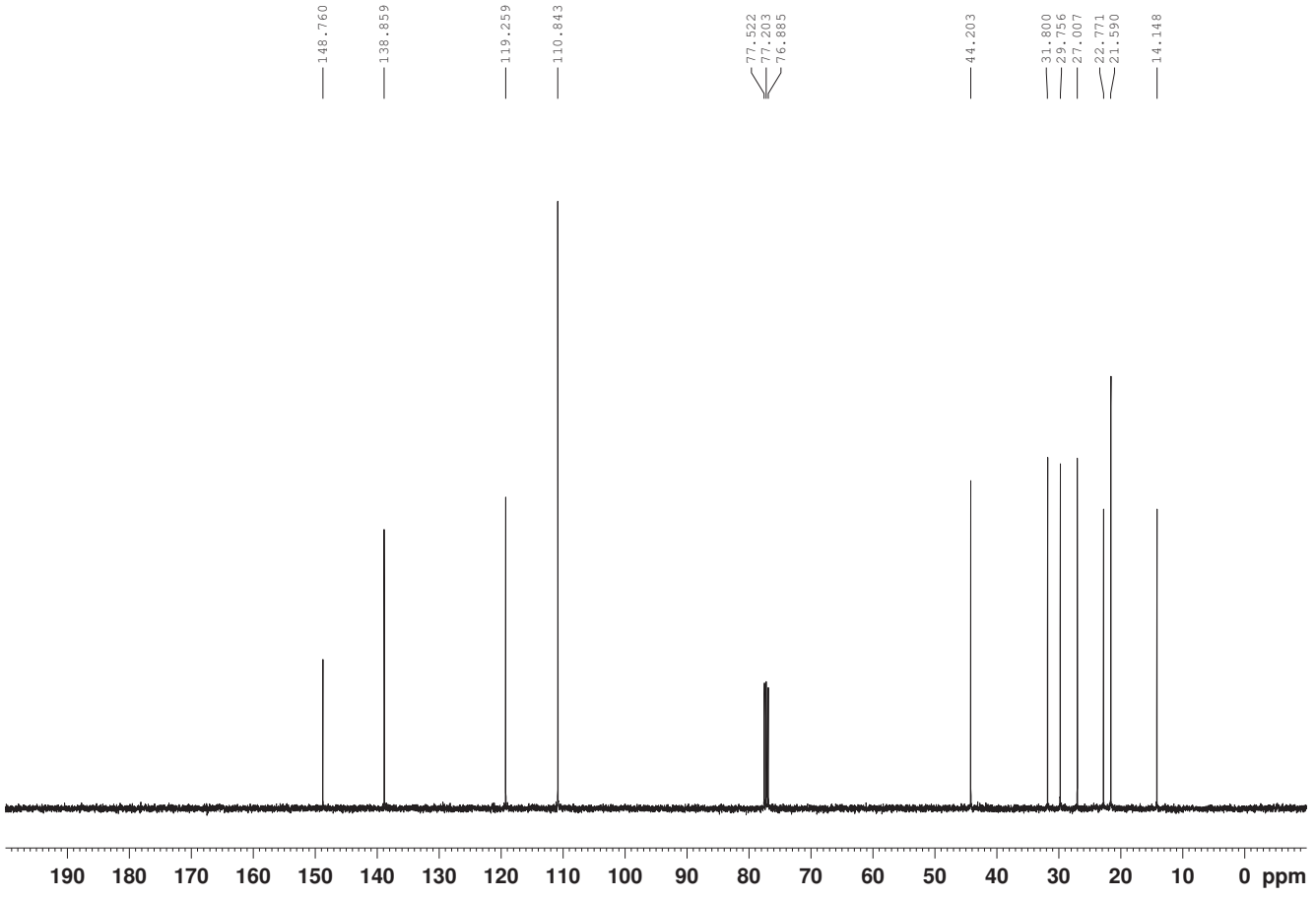
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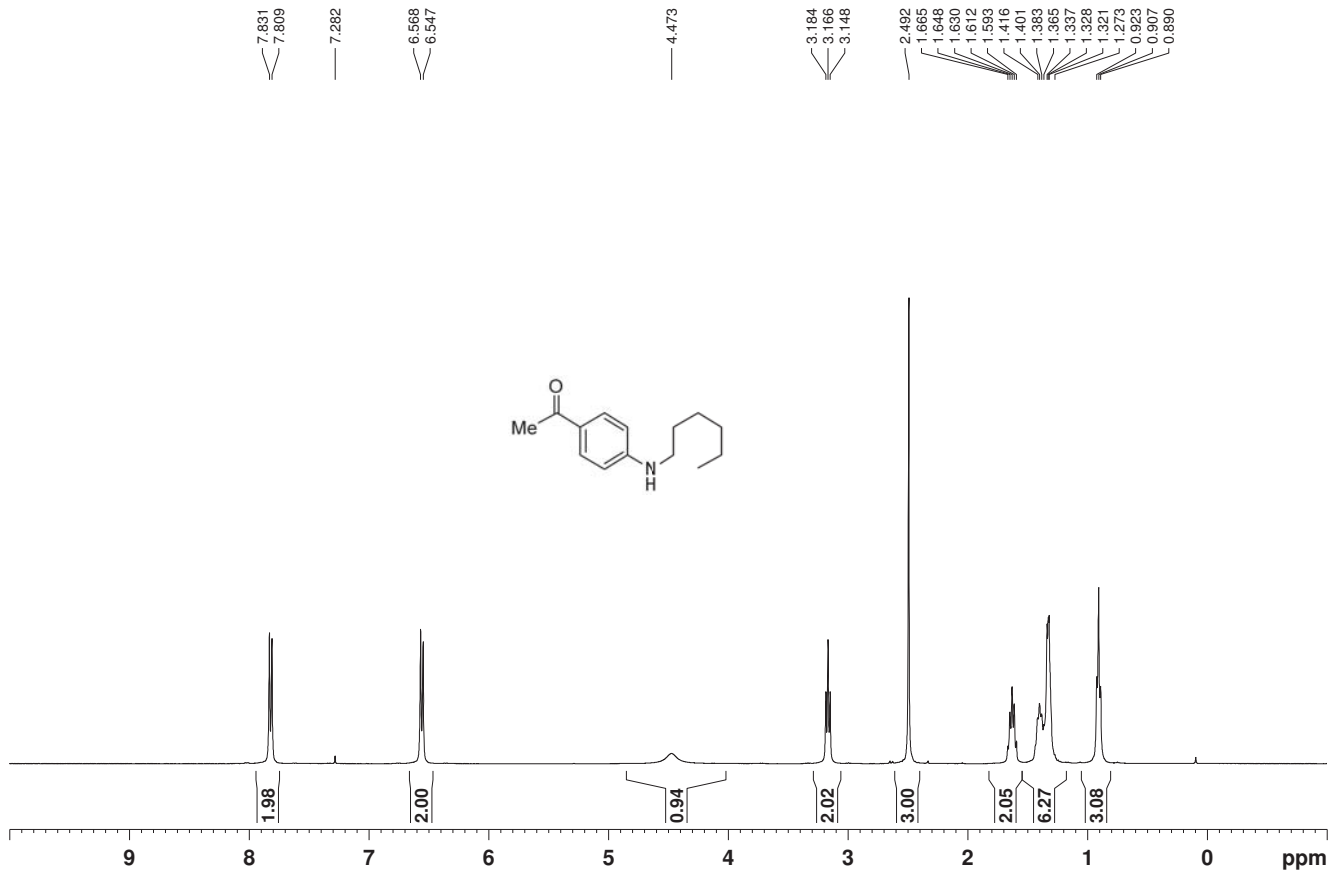
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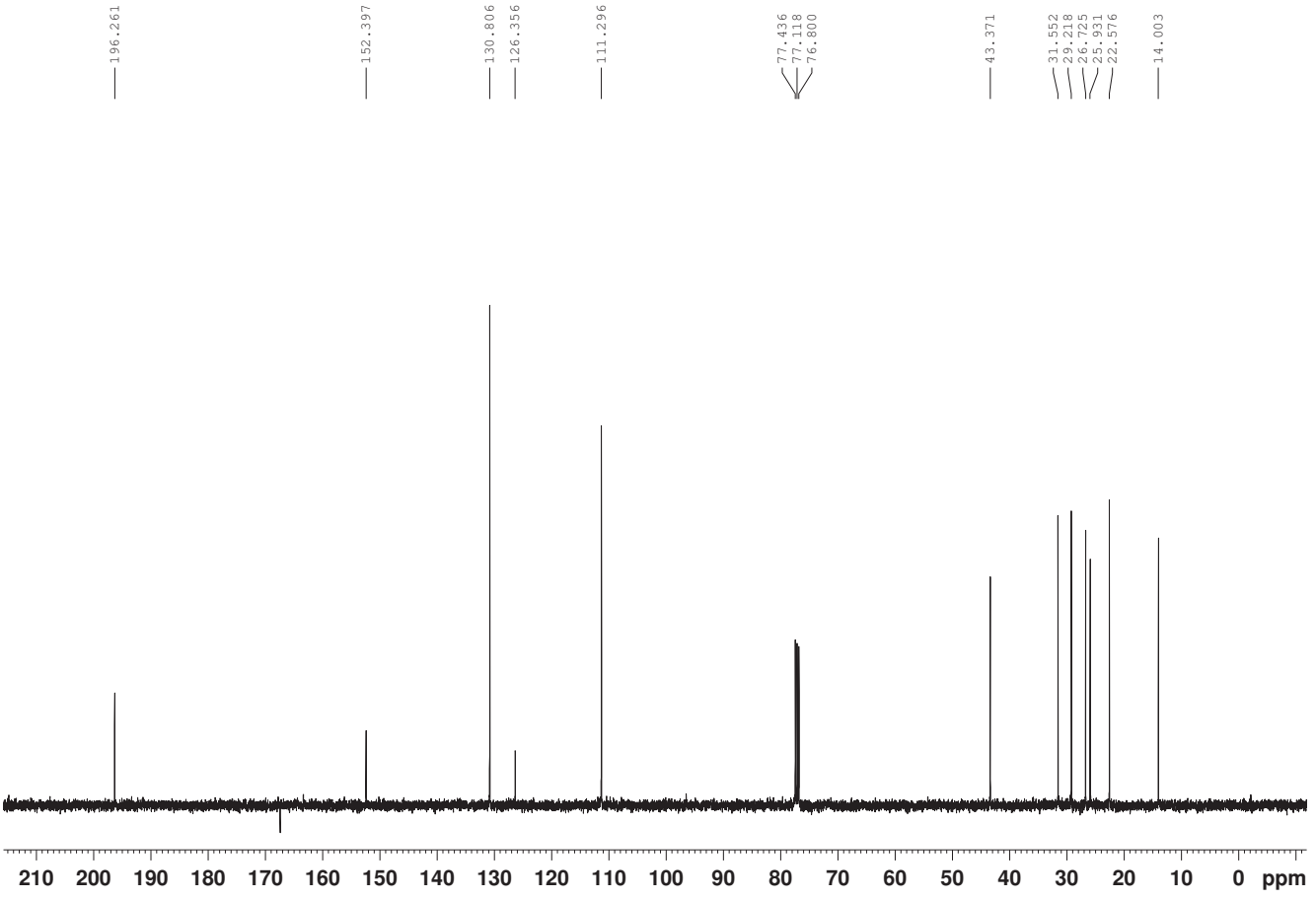
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200
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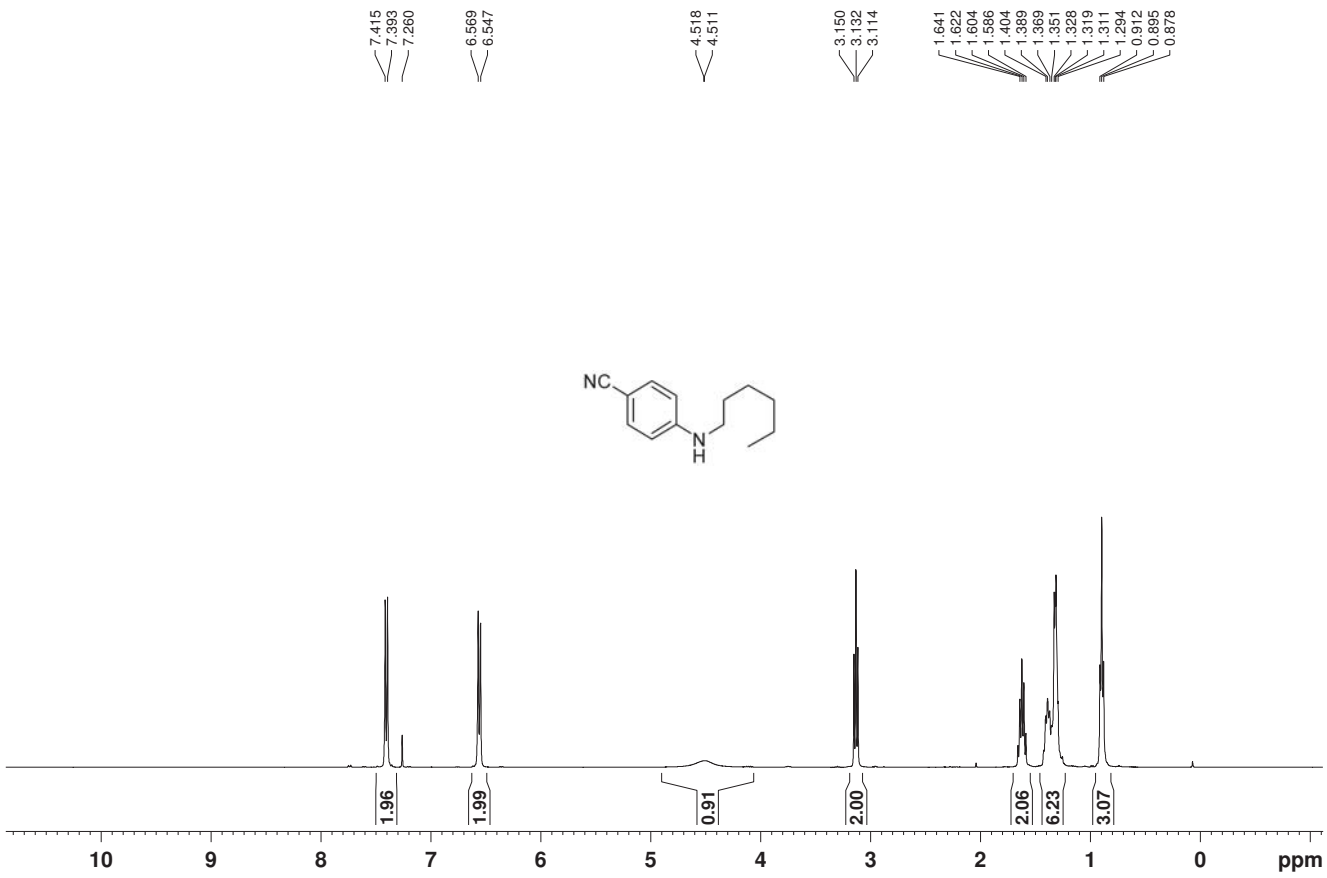
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N
H
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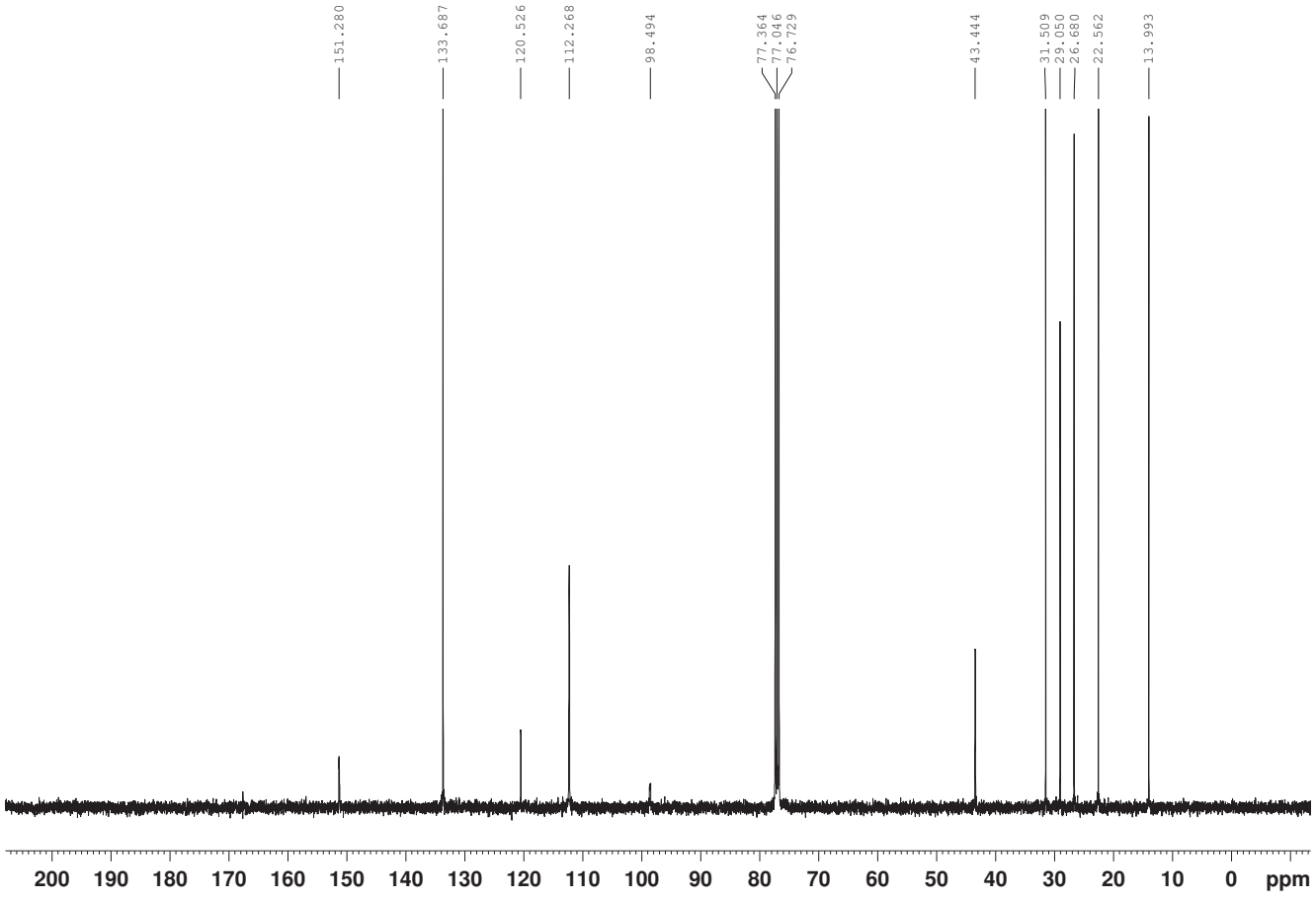
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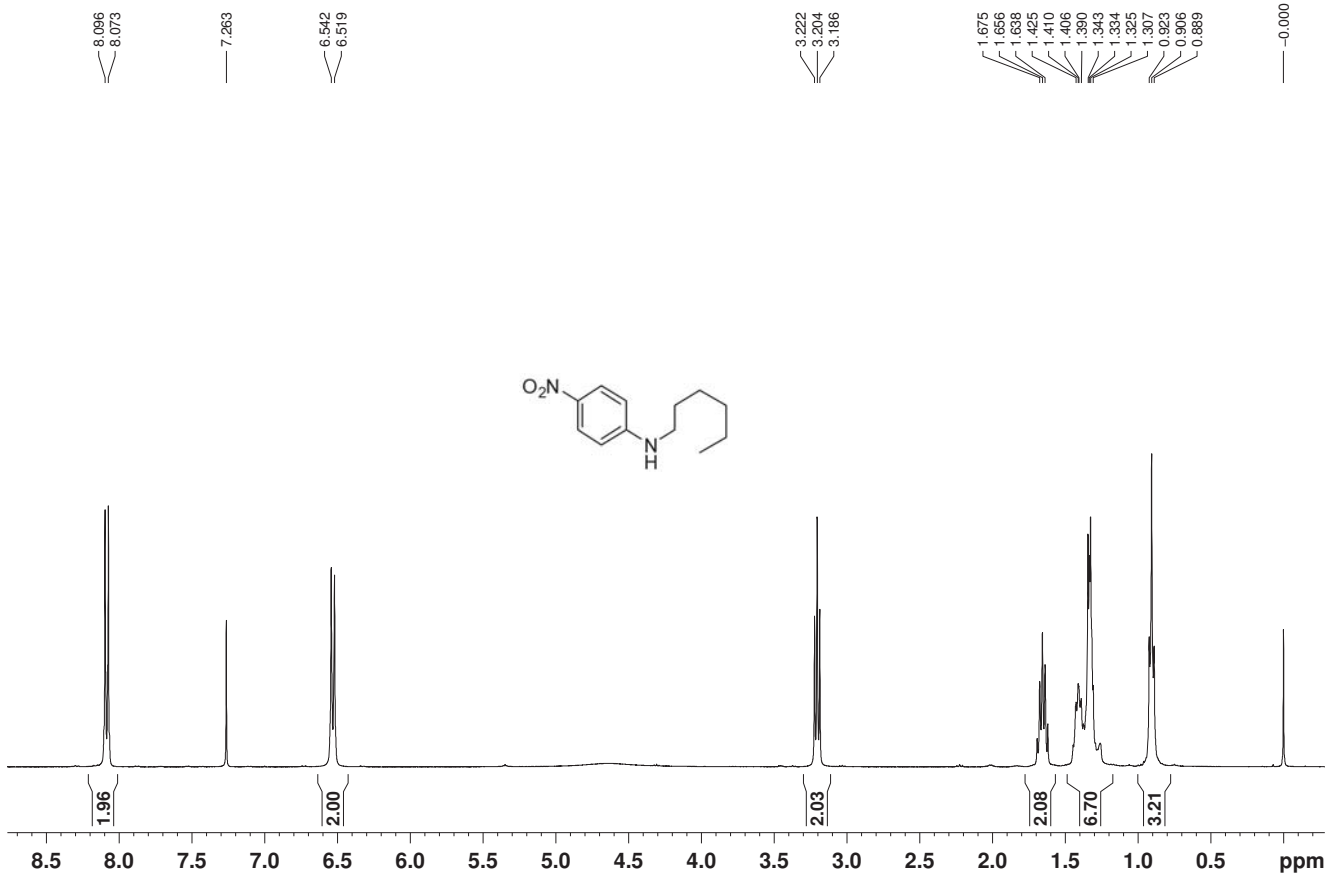
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0.889
0.000
O2N
N
H
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2.00
2.03
2.08
6.70
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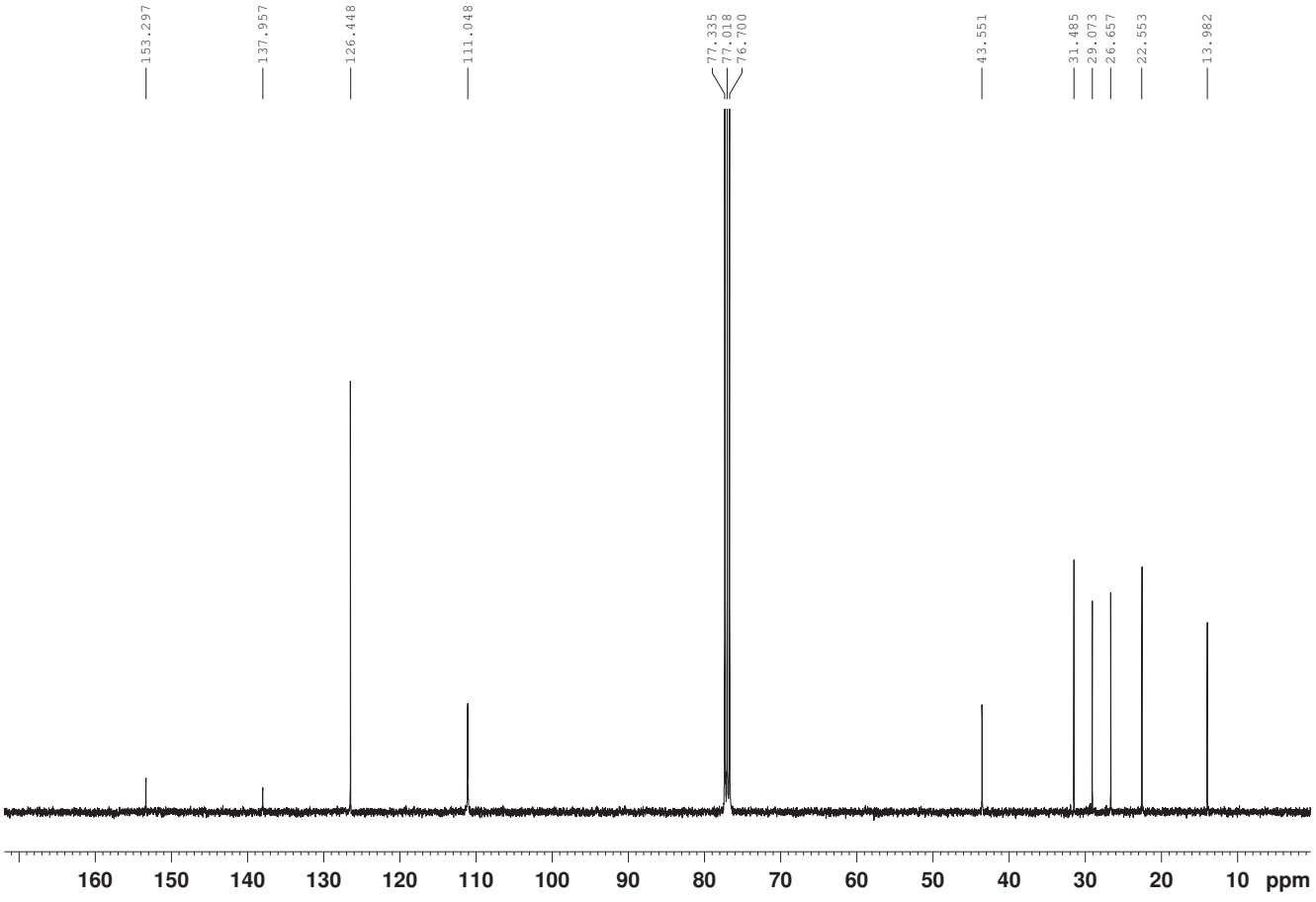
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77.335
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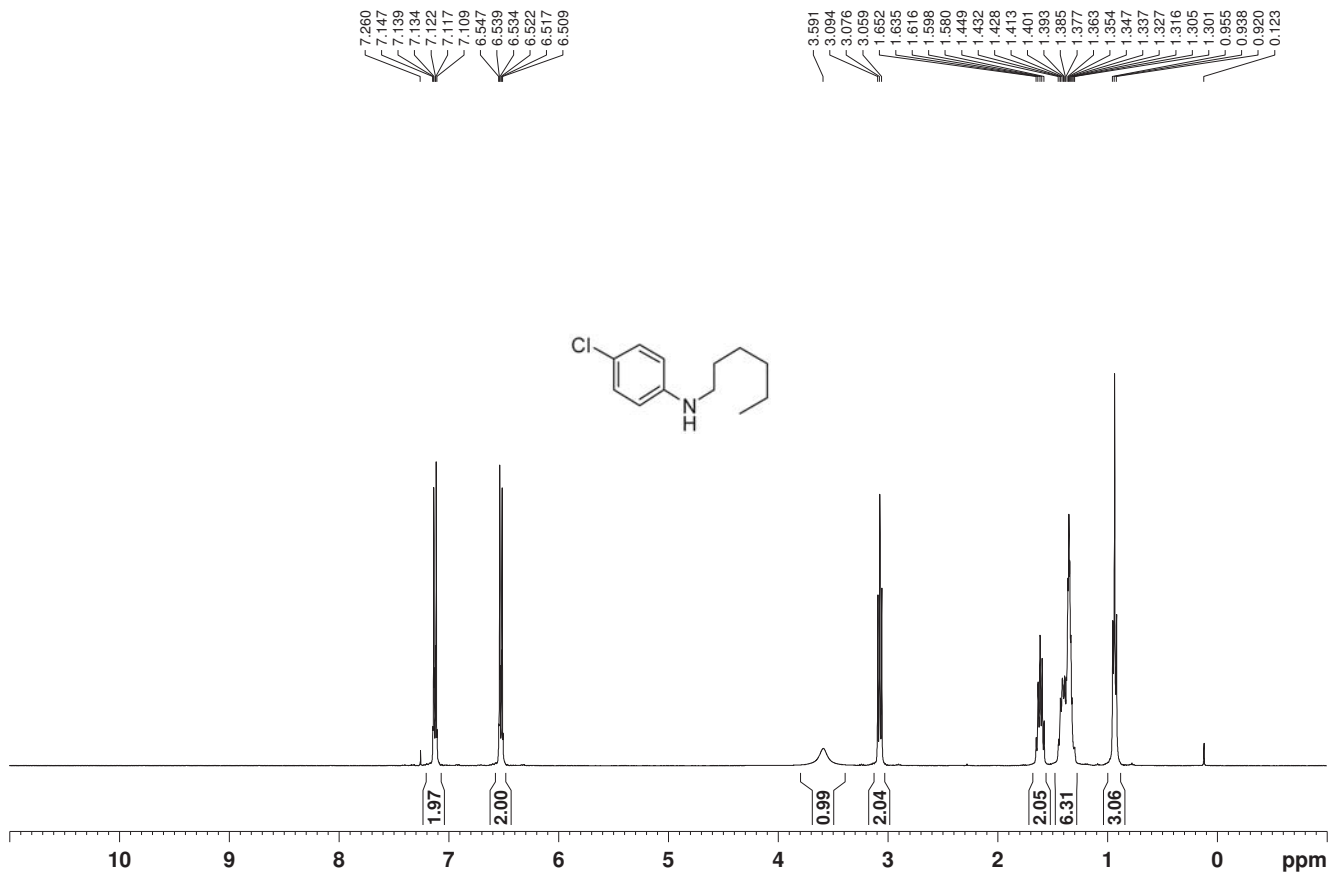
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3.094
3.076
3.059
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1.635
1.616
1.598
1.580
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1.428
1.413
1.401
1.393
1.385
1.377
1.363
1.354
1.347
1.337
1.327
1.316
1.305
1.301
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0.938
0.920
0.123
Cl
N
H
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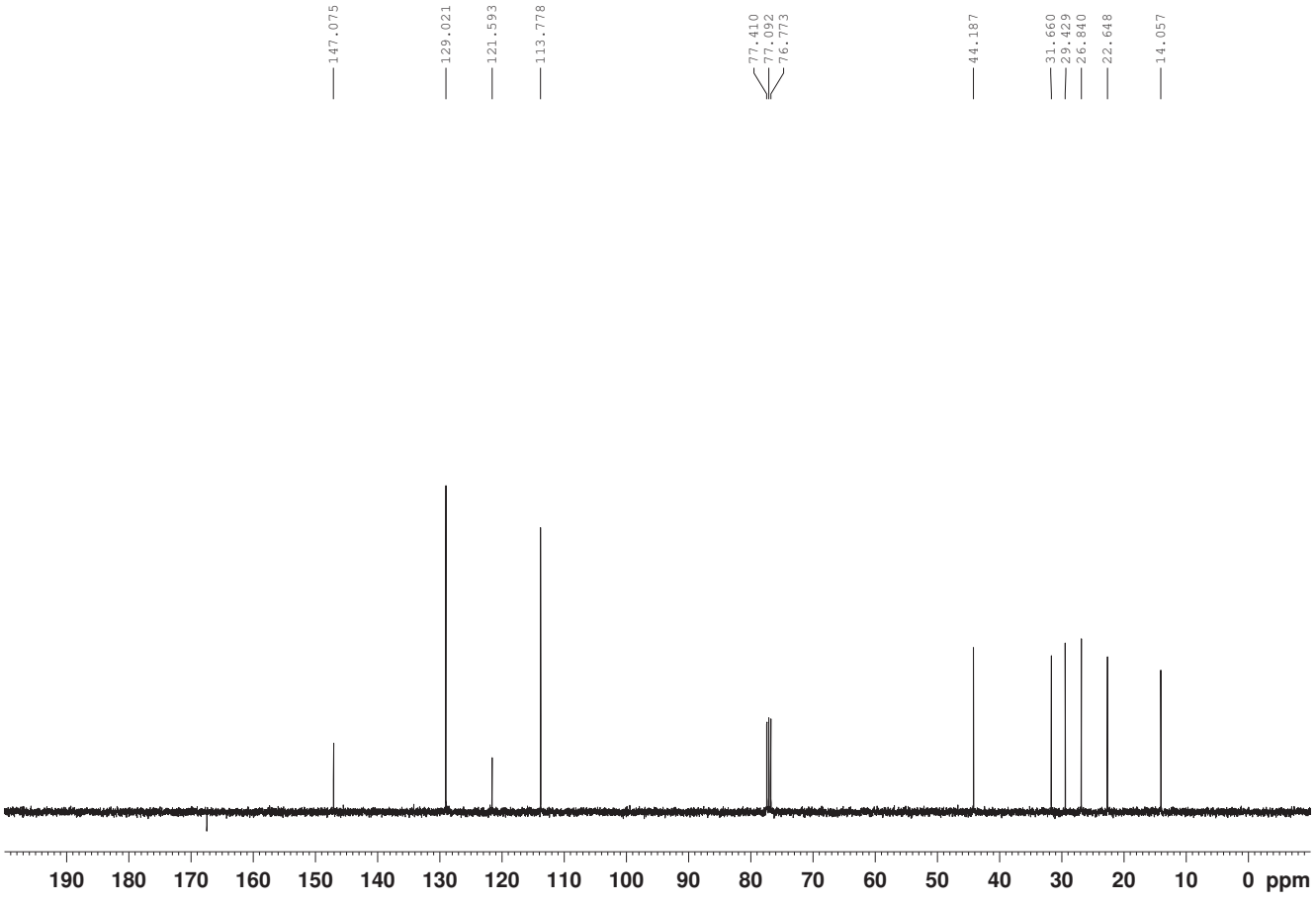
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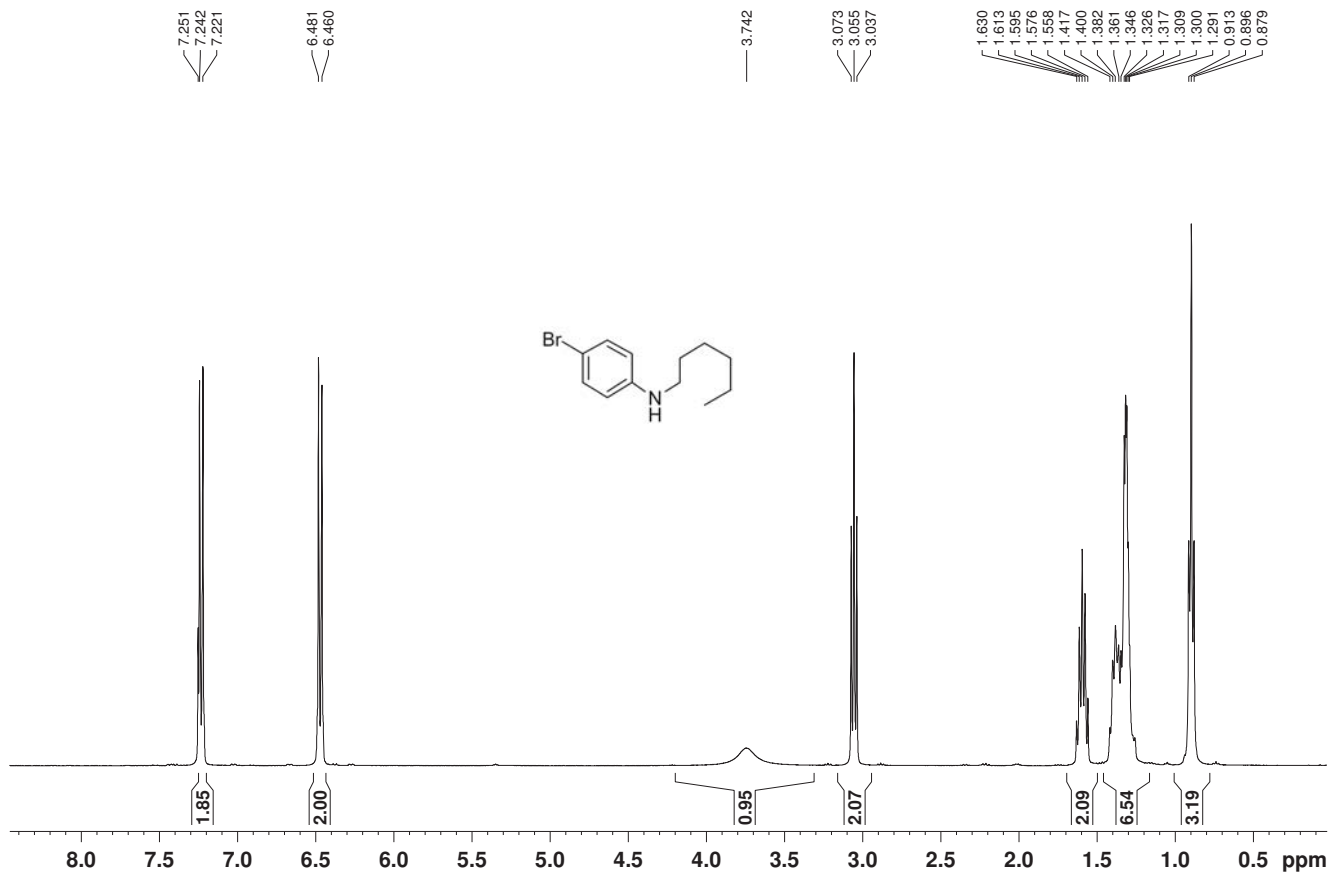
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3.055
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1.576
1.558
1.417
1.400
1.382
1.361
1.346
1.326
1.317
1.309
1.300
1.291
0.913
0.896
0.879
Br
N
H
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2.07
2.09
6.54
3.19
8.0
7.5
7.0
6.5
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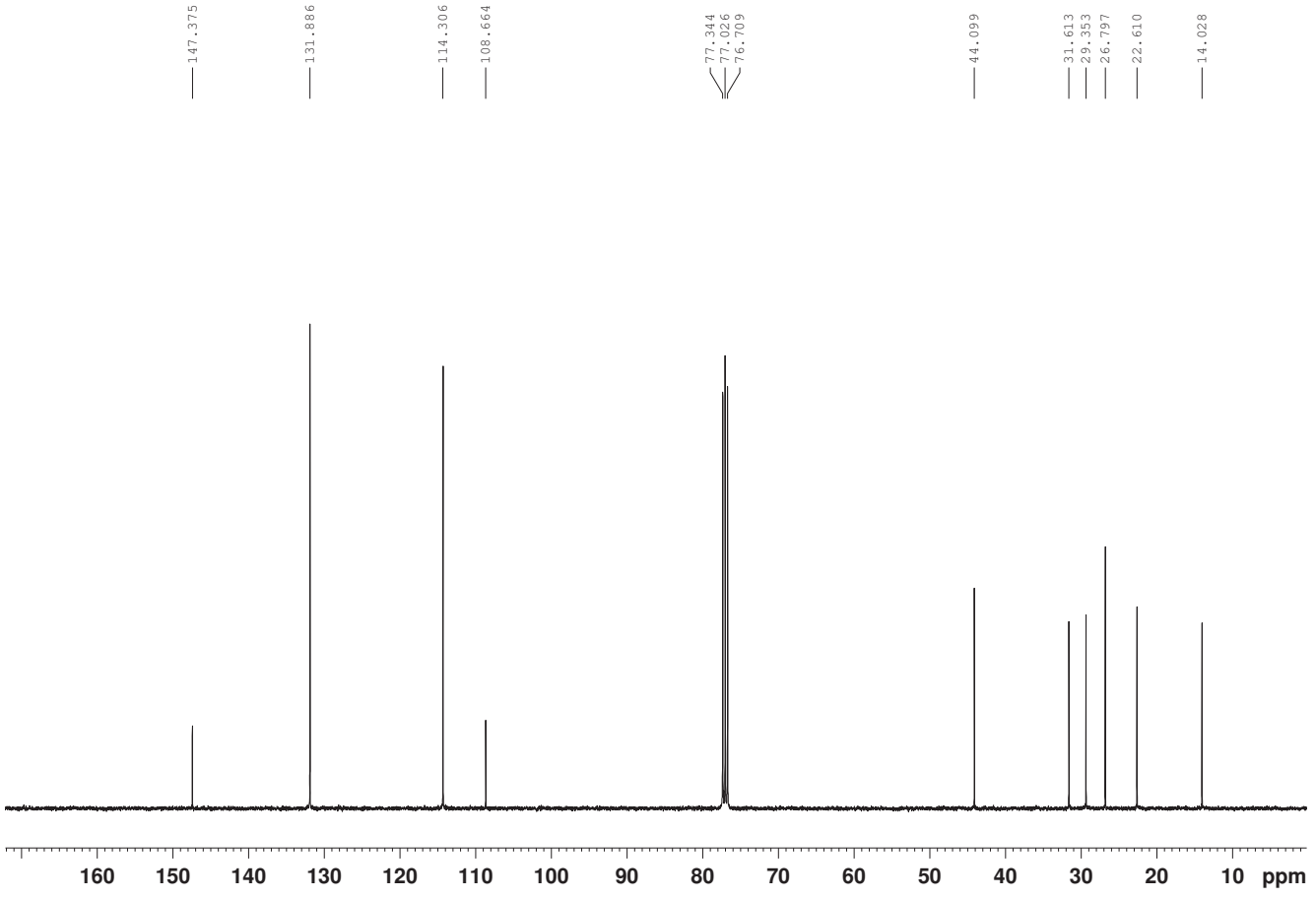
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150
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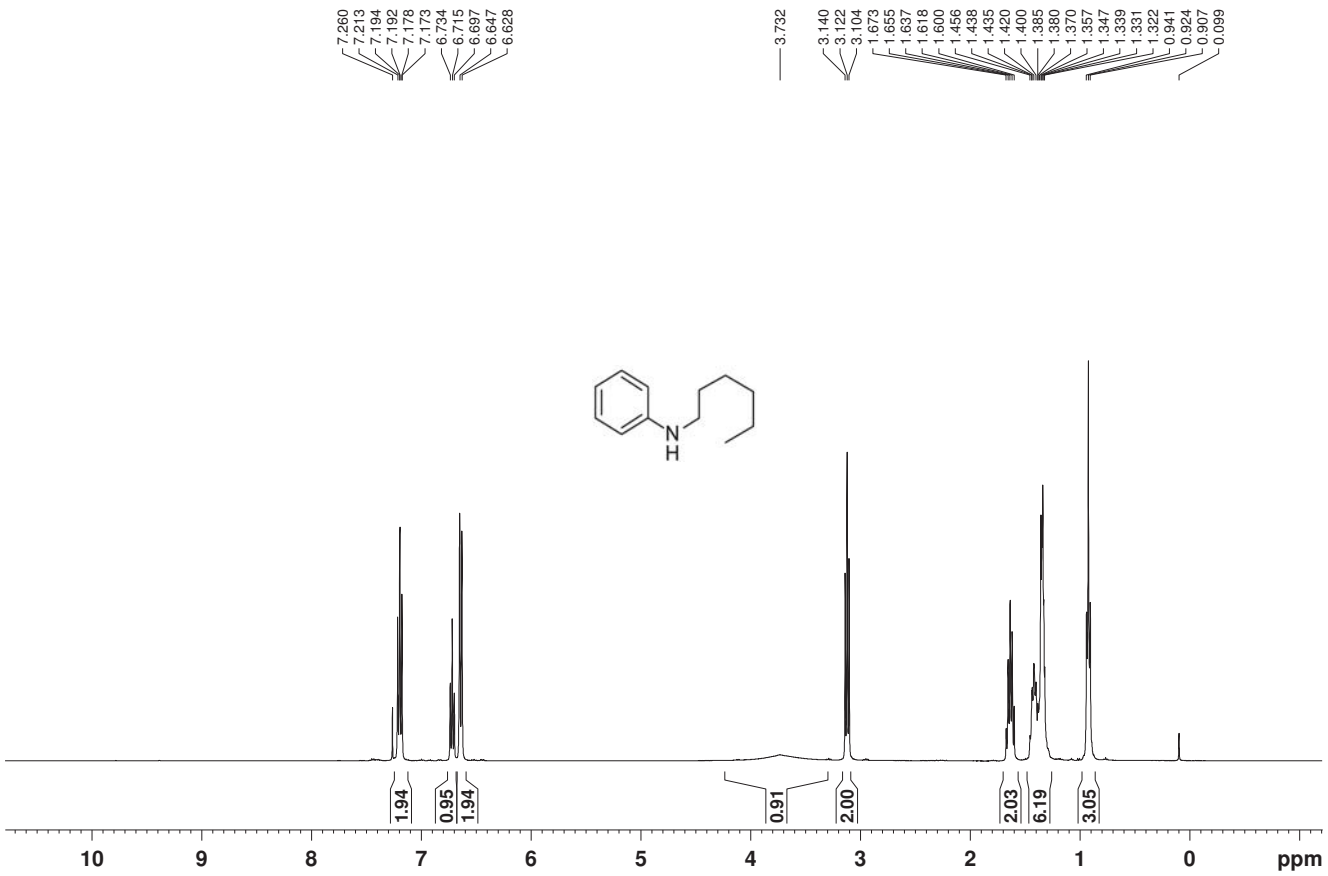
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6.715
6.697
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6.628
3.732
3.140
3.122
3.104
1.673
1.655
1.637
1.618
1.600
1.456
1.438
1.435
1.420
1.400
1.385
1.380
1.370
1.357
1.347
1.339
1.331
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0.907
0.099
N
H
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0.95
1.94
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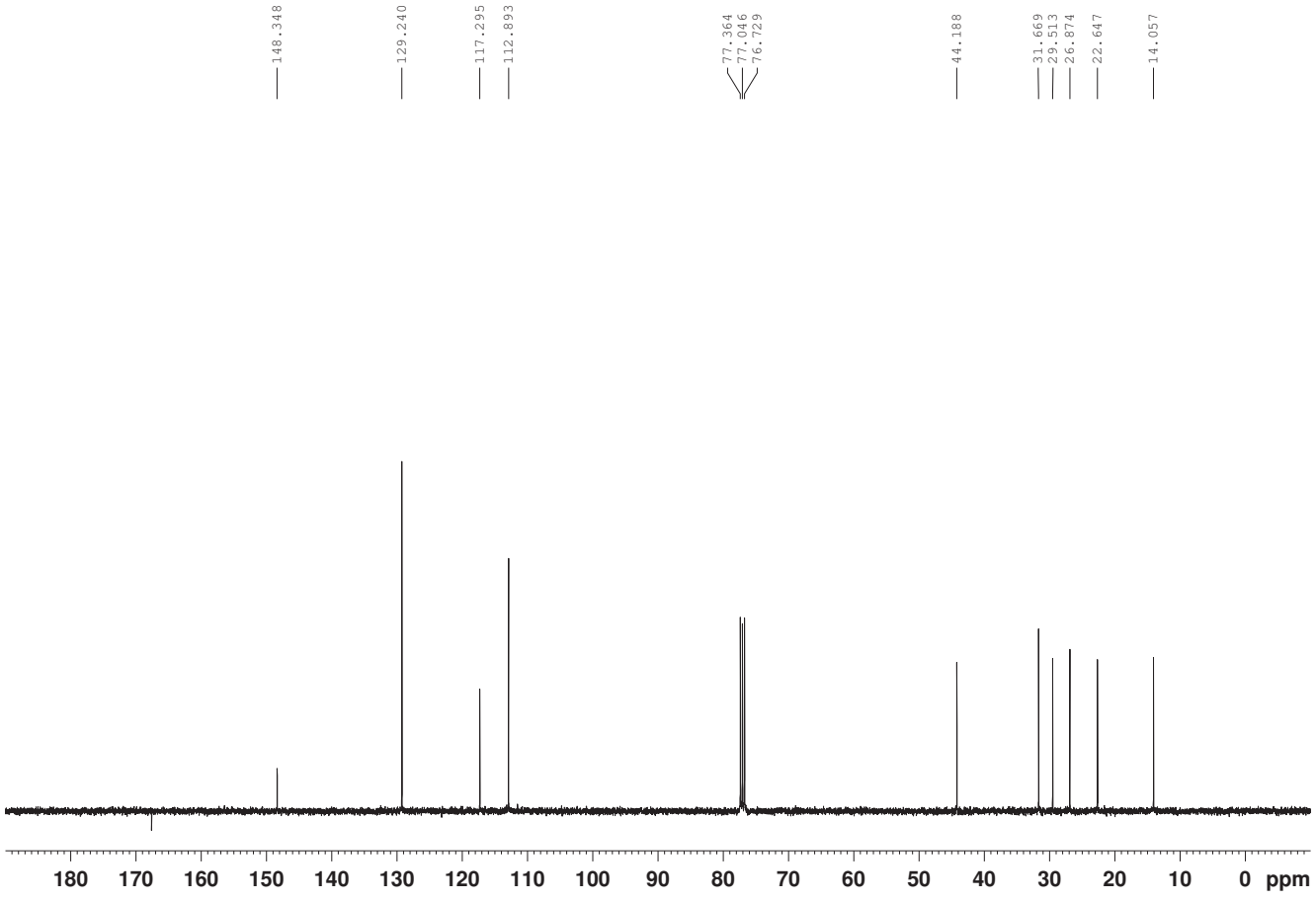
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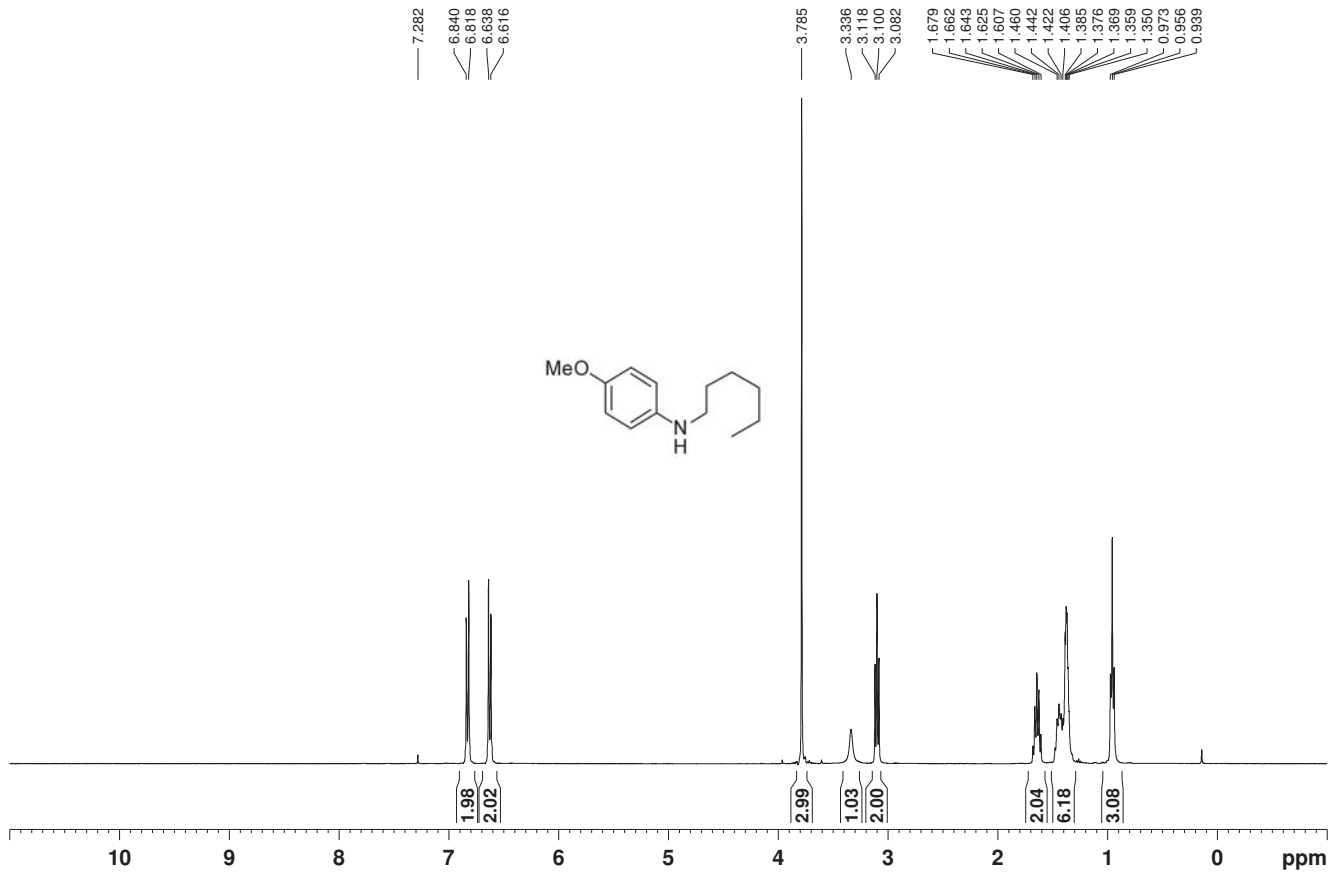
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6.638
6.616
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3.336
3.118
3.100
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1.679
1.662
1.643
1.625
1.607
1.460
1.442
1.422
1.406
1.385
1.376
1.369
1.359
1.350
0.973
0.956
0.939
MeO
N
H
1.98
2.02
2.99
1.03
2.00
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6.18
3.08
10
9
8
7
6
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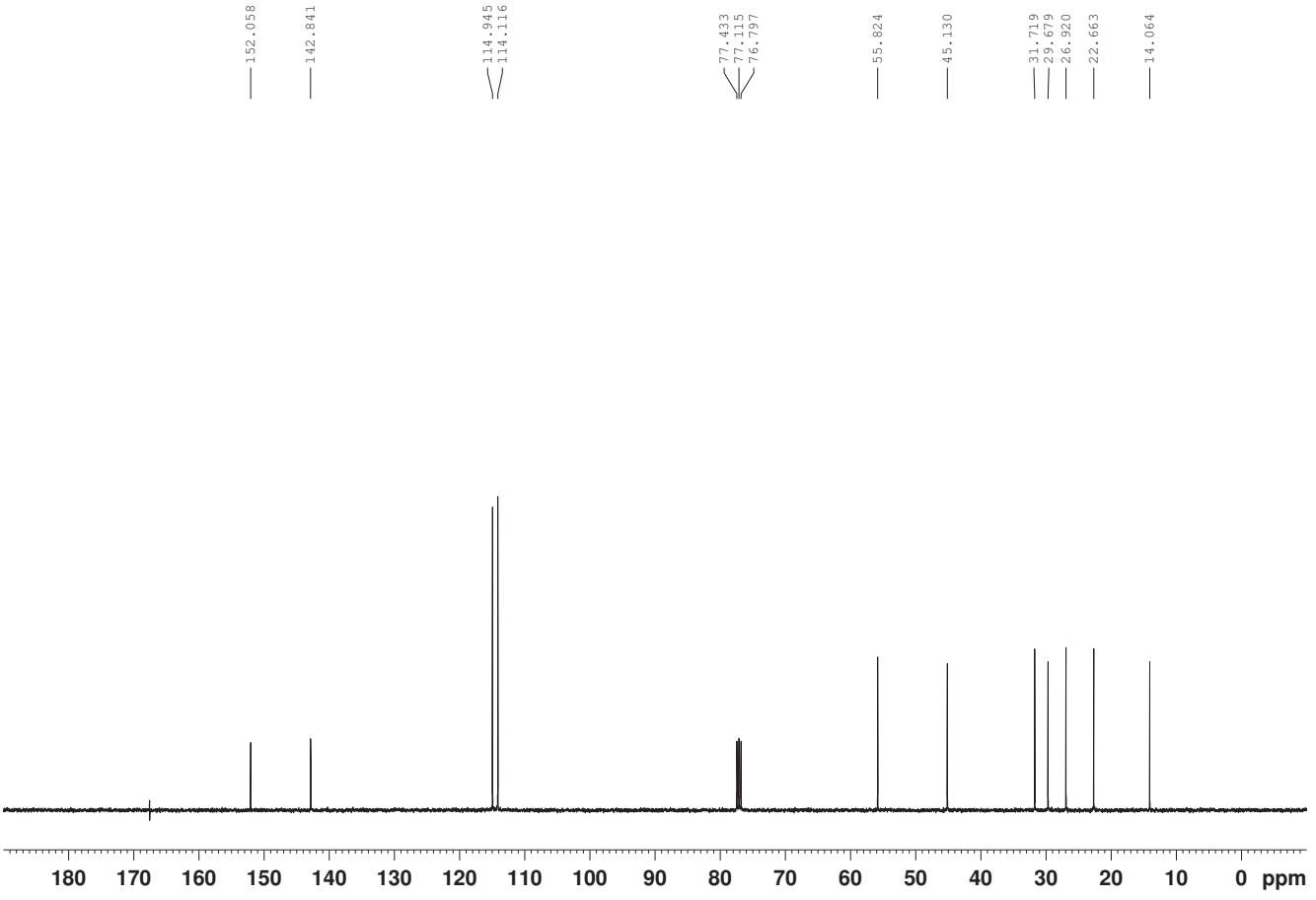
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140
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120
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100
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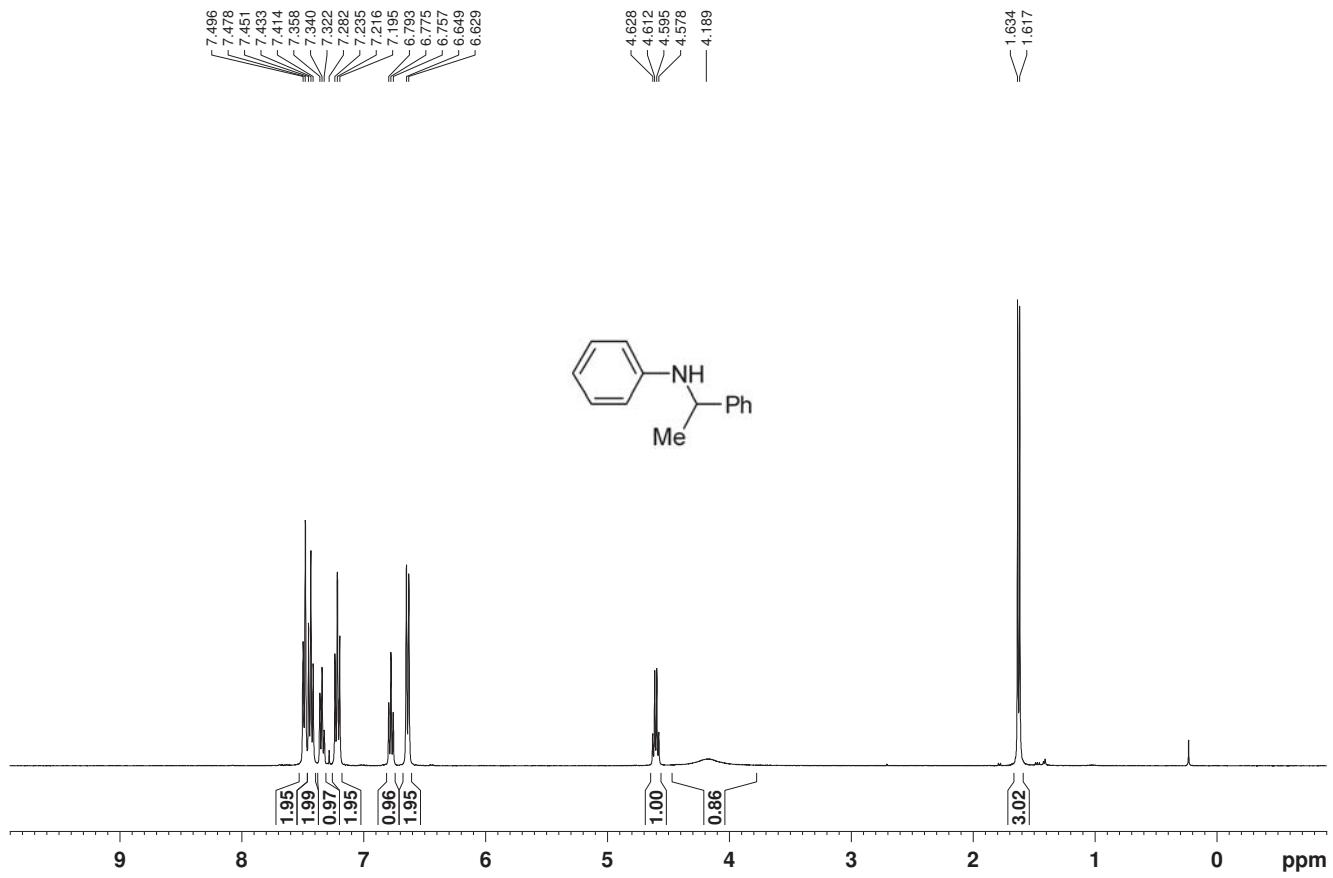
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7.340
7.322
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7.235
7.216
7.195
6.793
6.775
6.757
6.649
6.629
4.628
4.612
4.595
4.578
4.189
1.634
1.617
NH
Ph
Me
1.95
1.99
0.97
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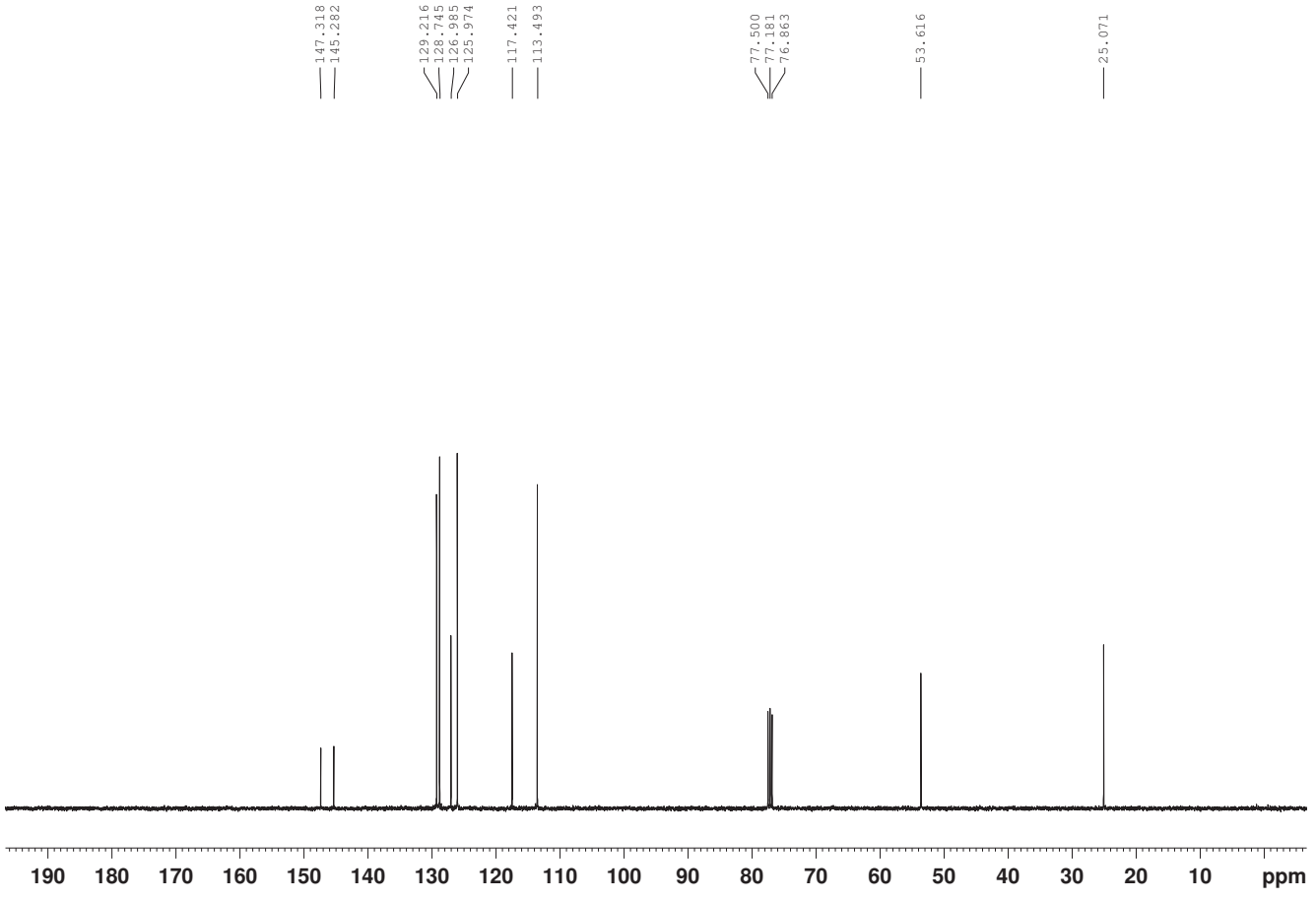
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113.493
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77.181
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25.071
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150
140
130
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70
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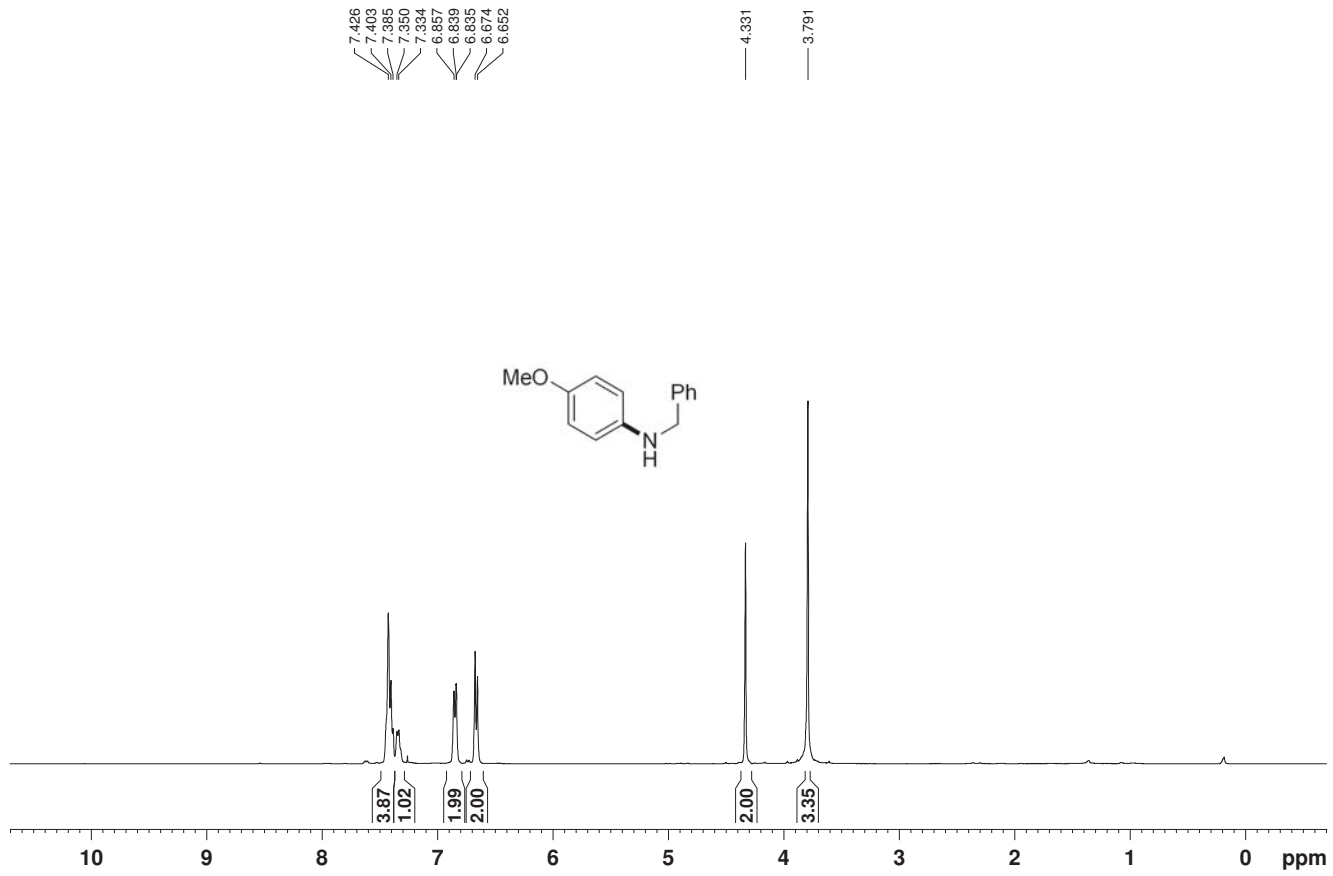
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6.835
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6.652
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MeO
Ph
N
H
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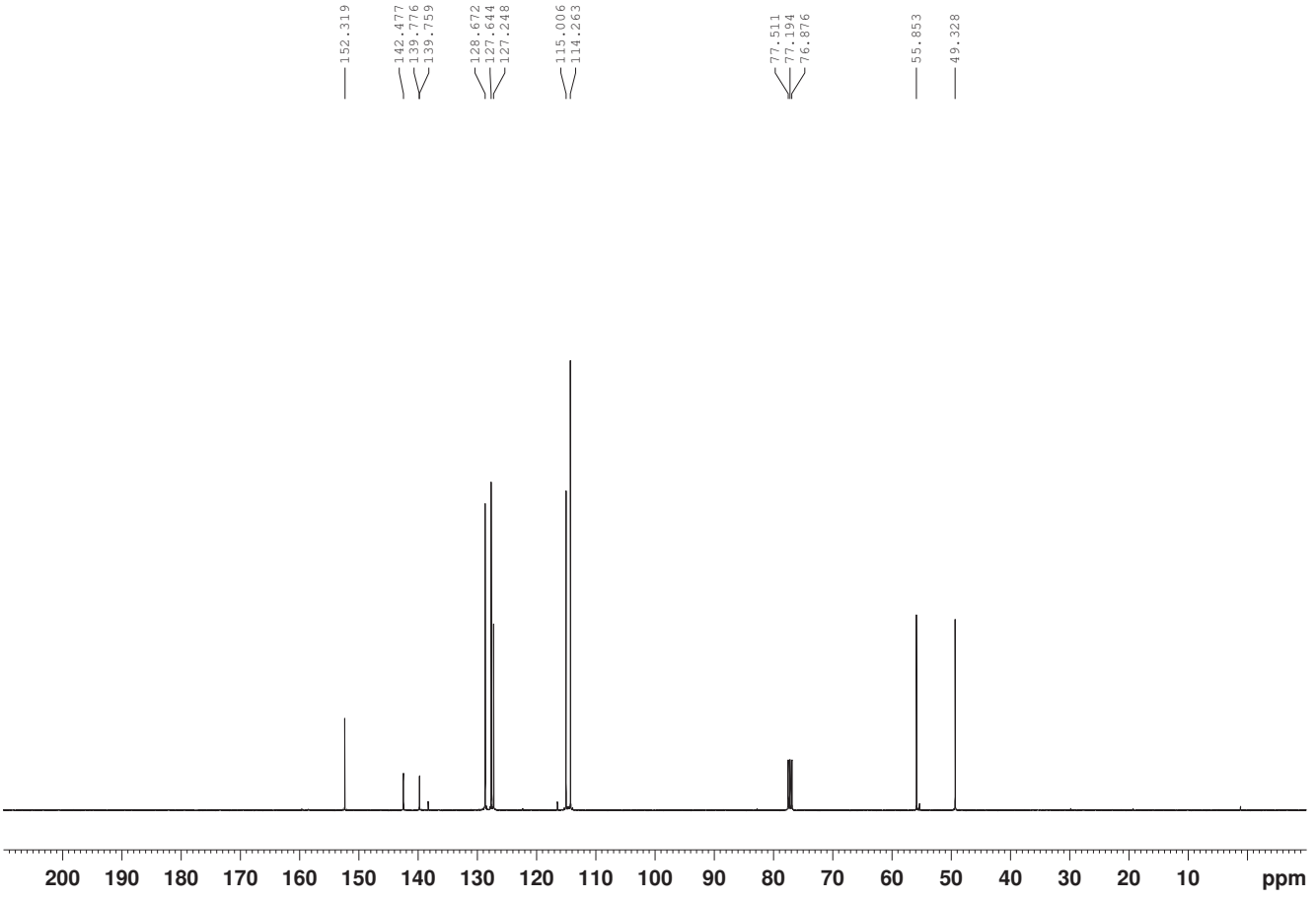
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49.328
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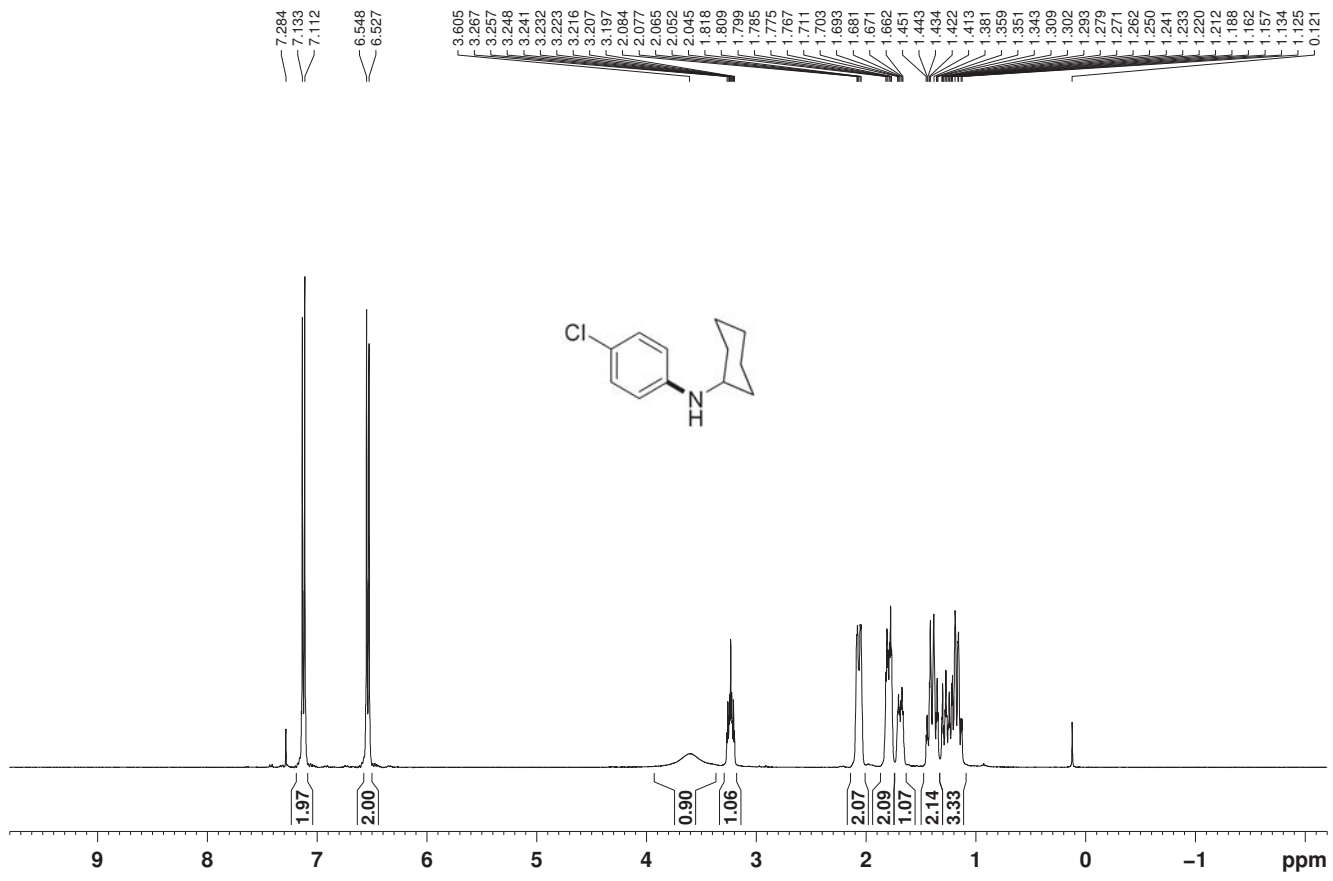
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3.216
3.207
3.197
2.084
2.077
2.065
2.052
2.045
1.818
1.809
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1.767
1.711
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1.681
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1.662
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1.443
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1.422
1.413
1.381
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1.351
1.343
1.309
1.302
1.293
1.279
1.271
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1.250
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1.233
1.220
1.212
1.188
1.162
1.157
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1.125
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Cl
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1.06
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2.14
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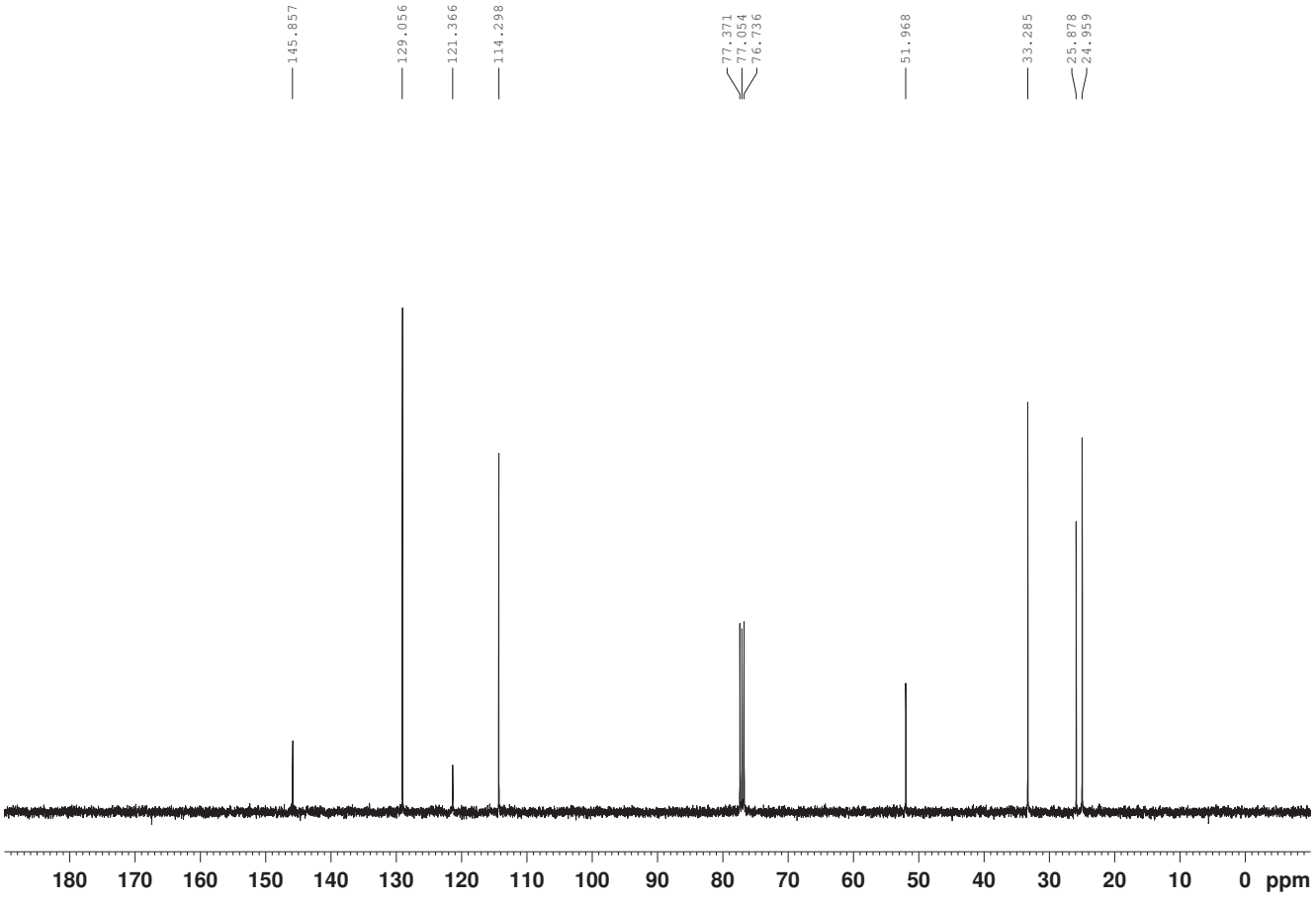
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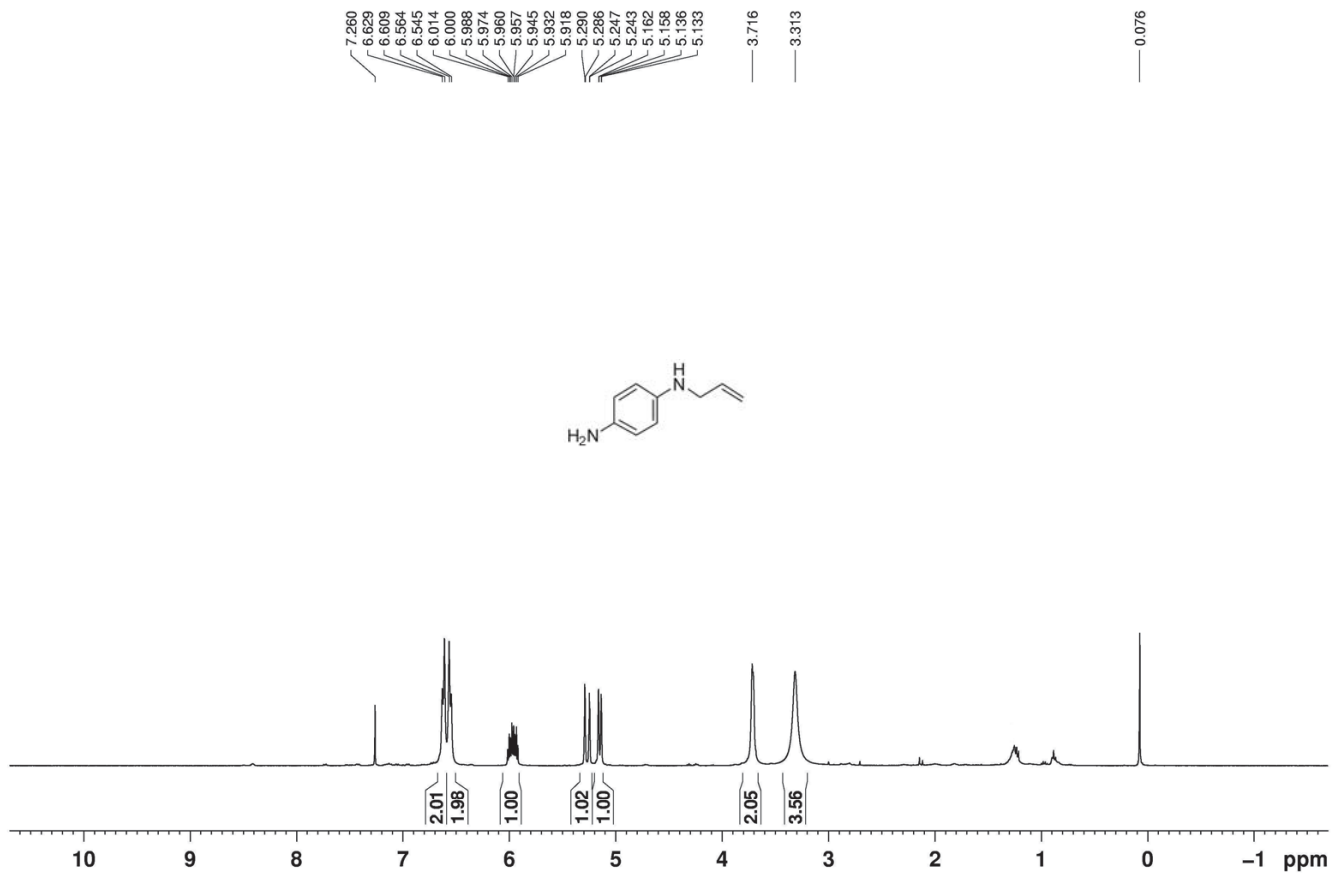
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5.945
5.932
5.918
5.290
5.286
5.247
5.243
5.162
5.158
5.136
5.133
3.716
3.313
0.076
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N
H2N
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1.98
1.00
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5
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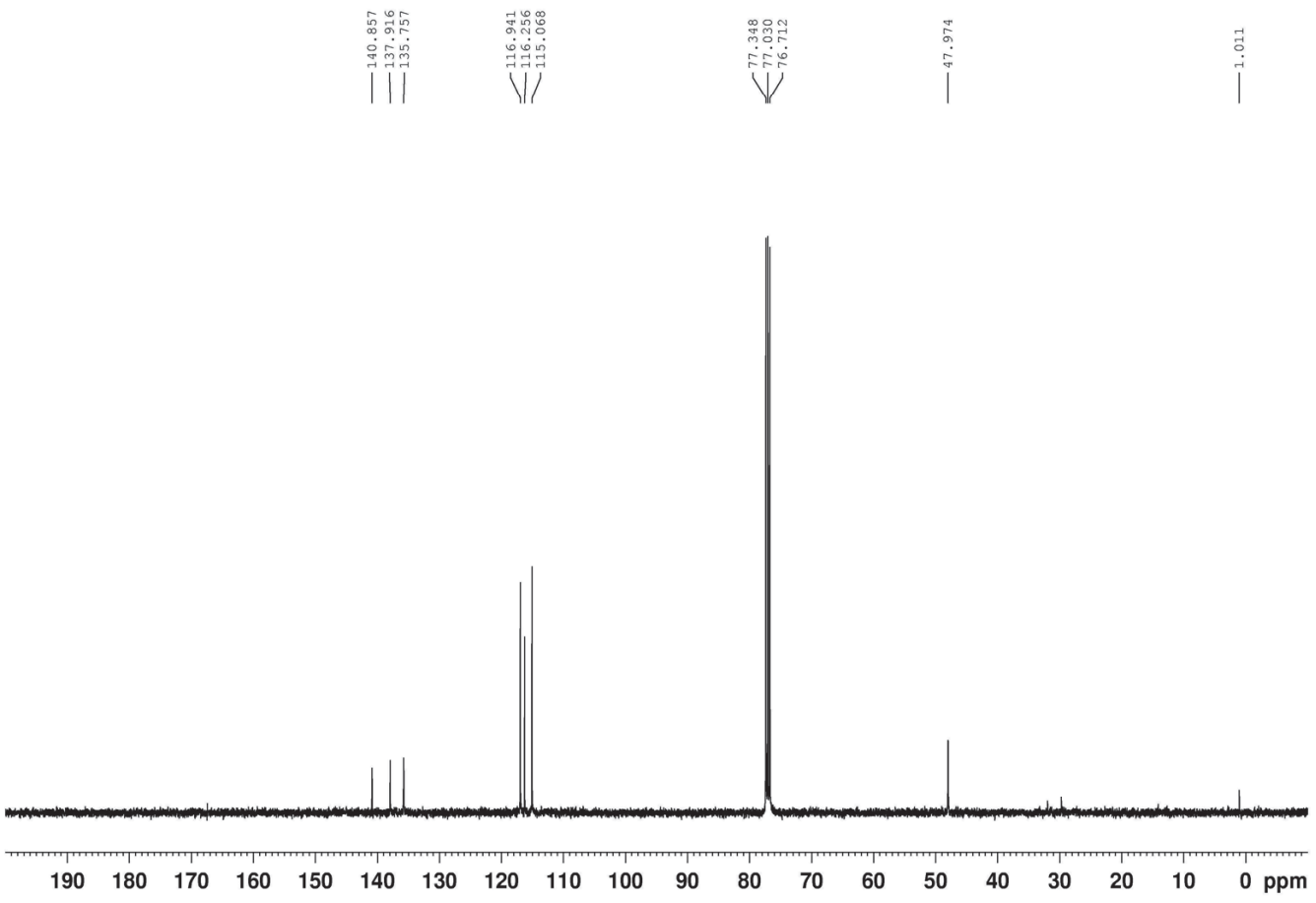
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76.712
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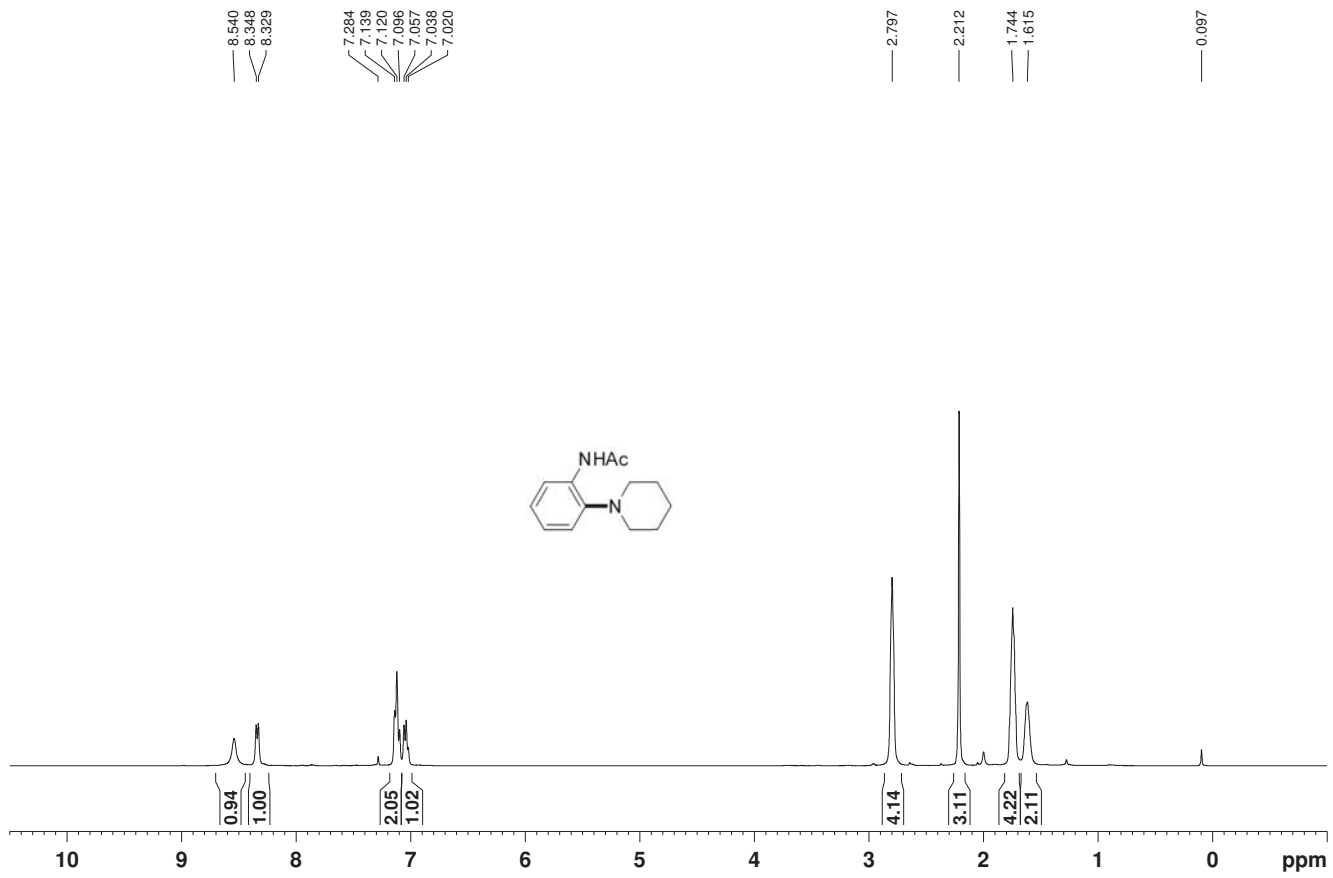
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7.057
7.038
7.020
2.797
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1.615
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NHAc
N
0.94
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2.05
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4.22
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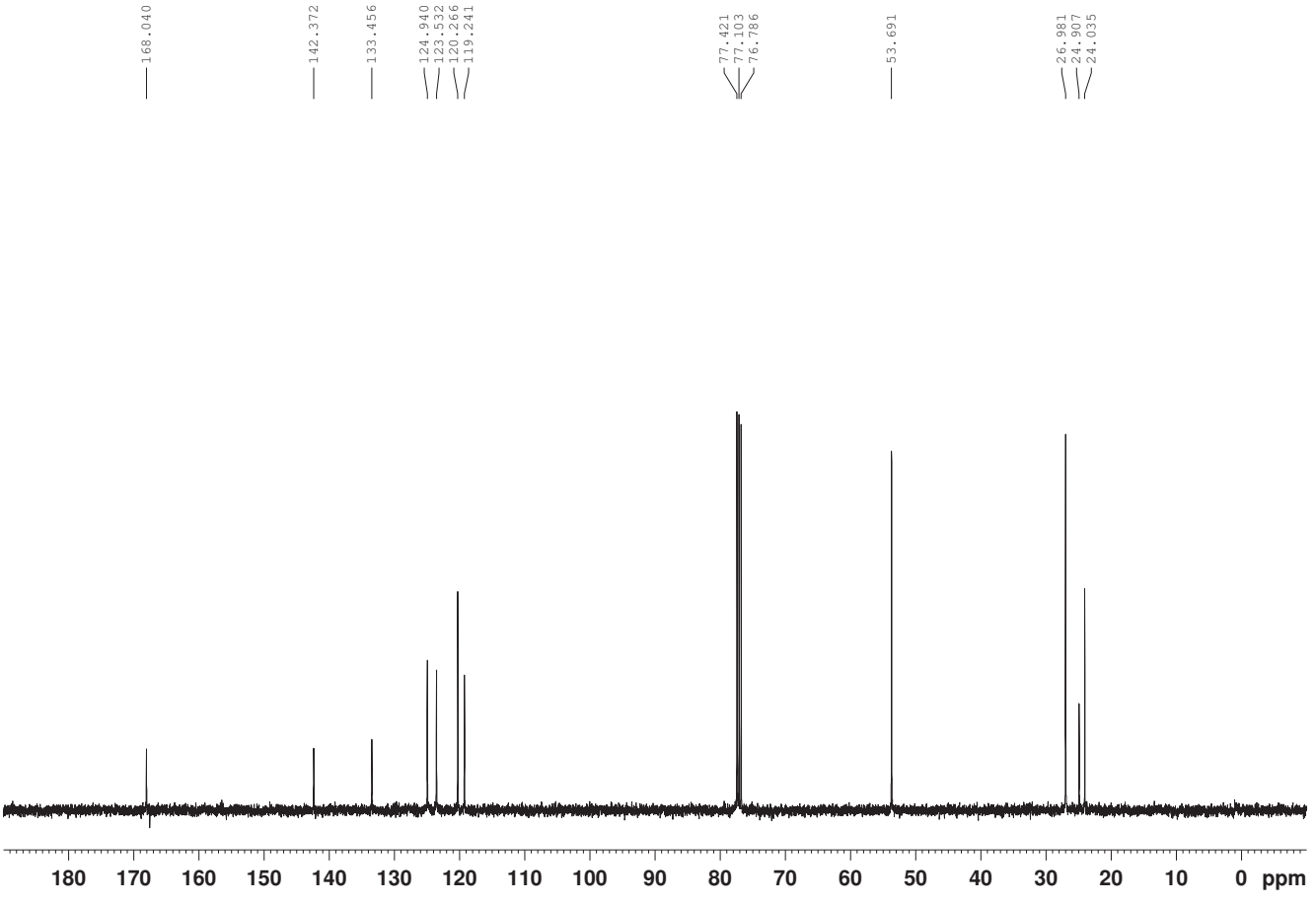
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77.421
77.103
76.786
53.691
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24.035
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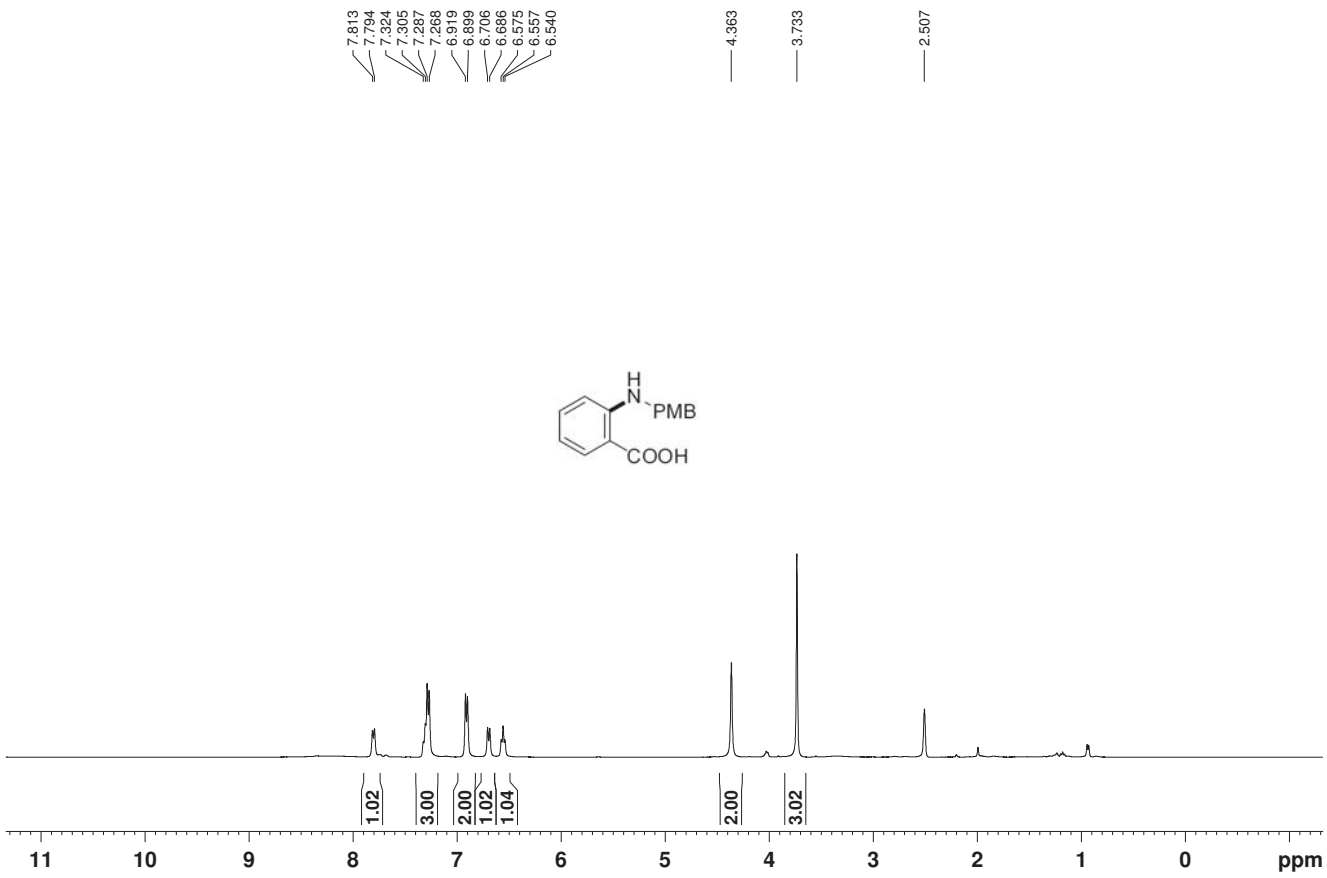
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6.899
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6.686
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6.540
4.363
3.733
2.507
H
N
PMB
COOH
1.02
3.00
2.00
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3.02
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10
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6
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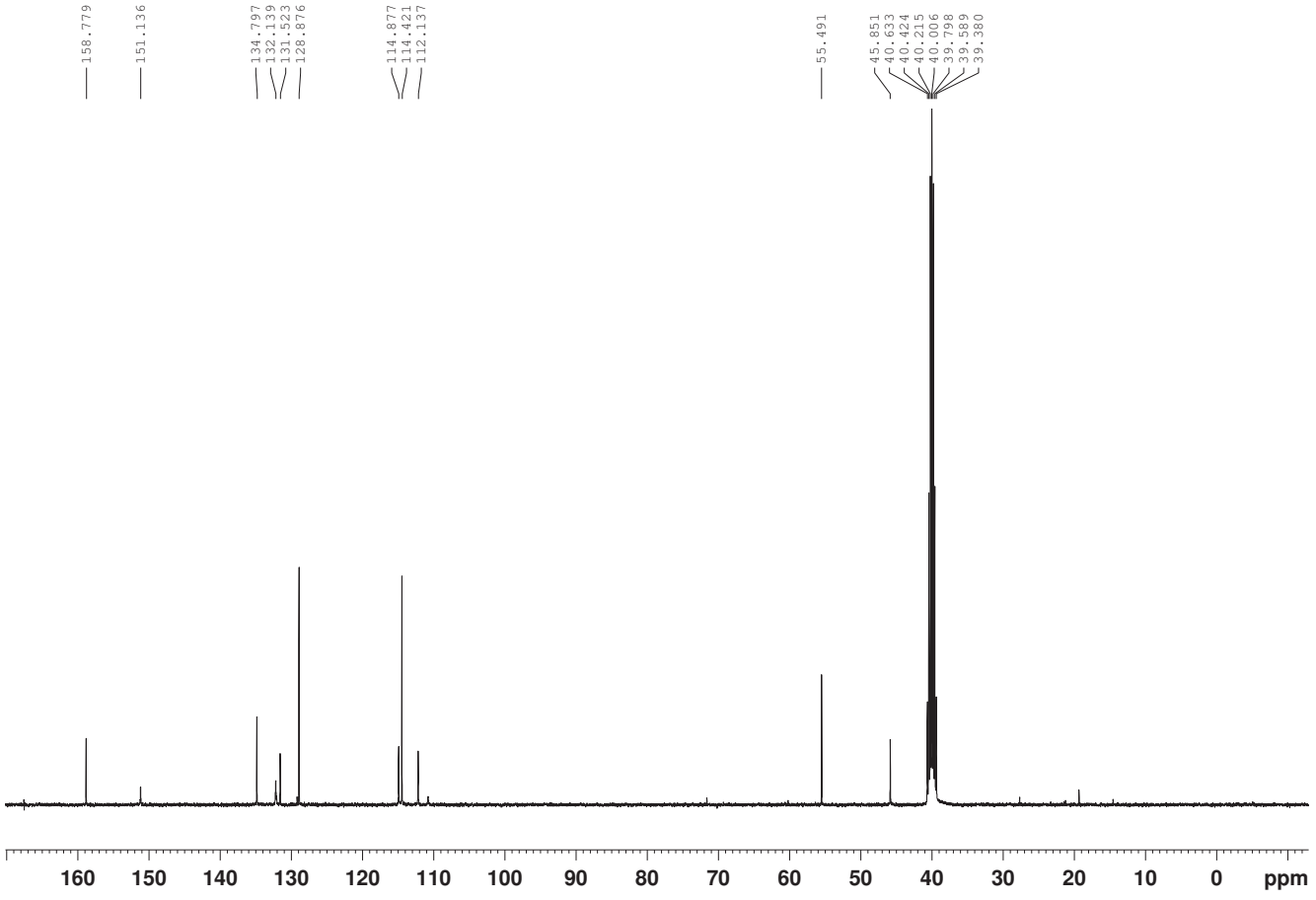
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40.006
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39.589
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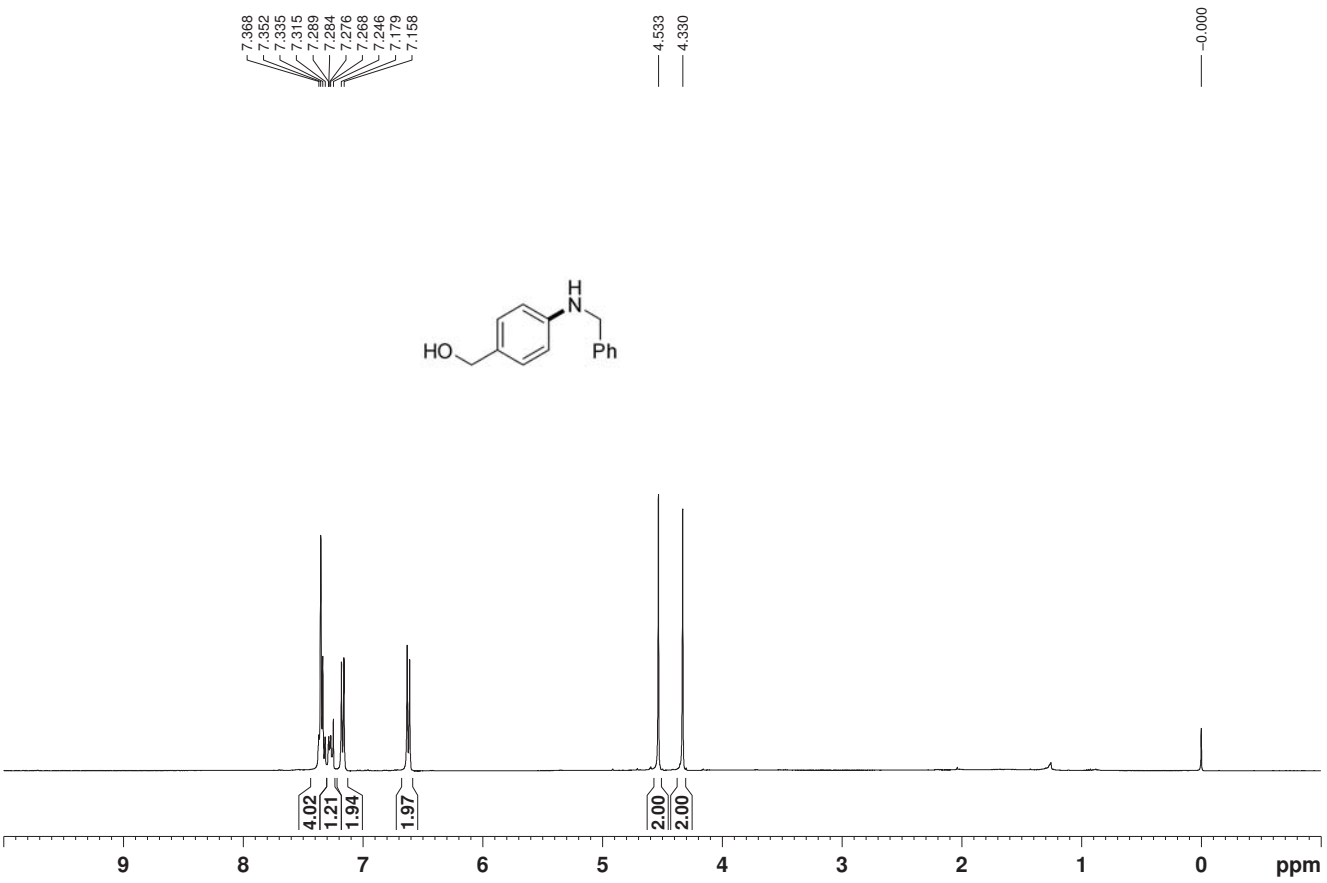
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7.179
7.158
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H
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Ph
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2.00
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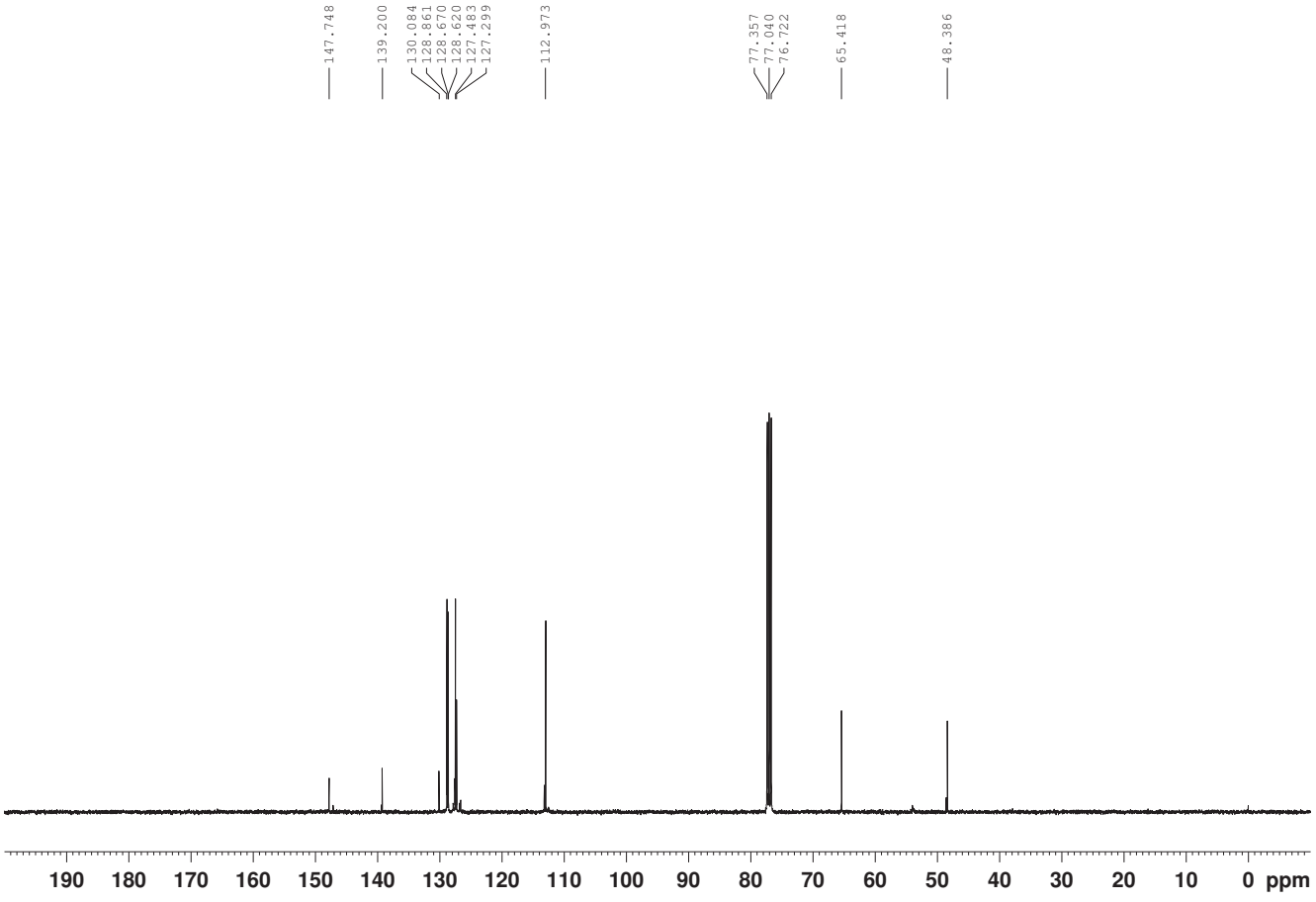
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127.299
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48.386
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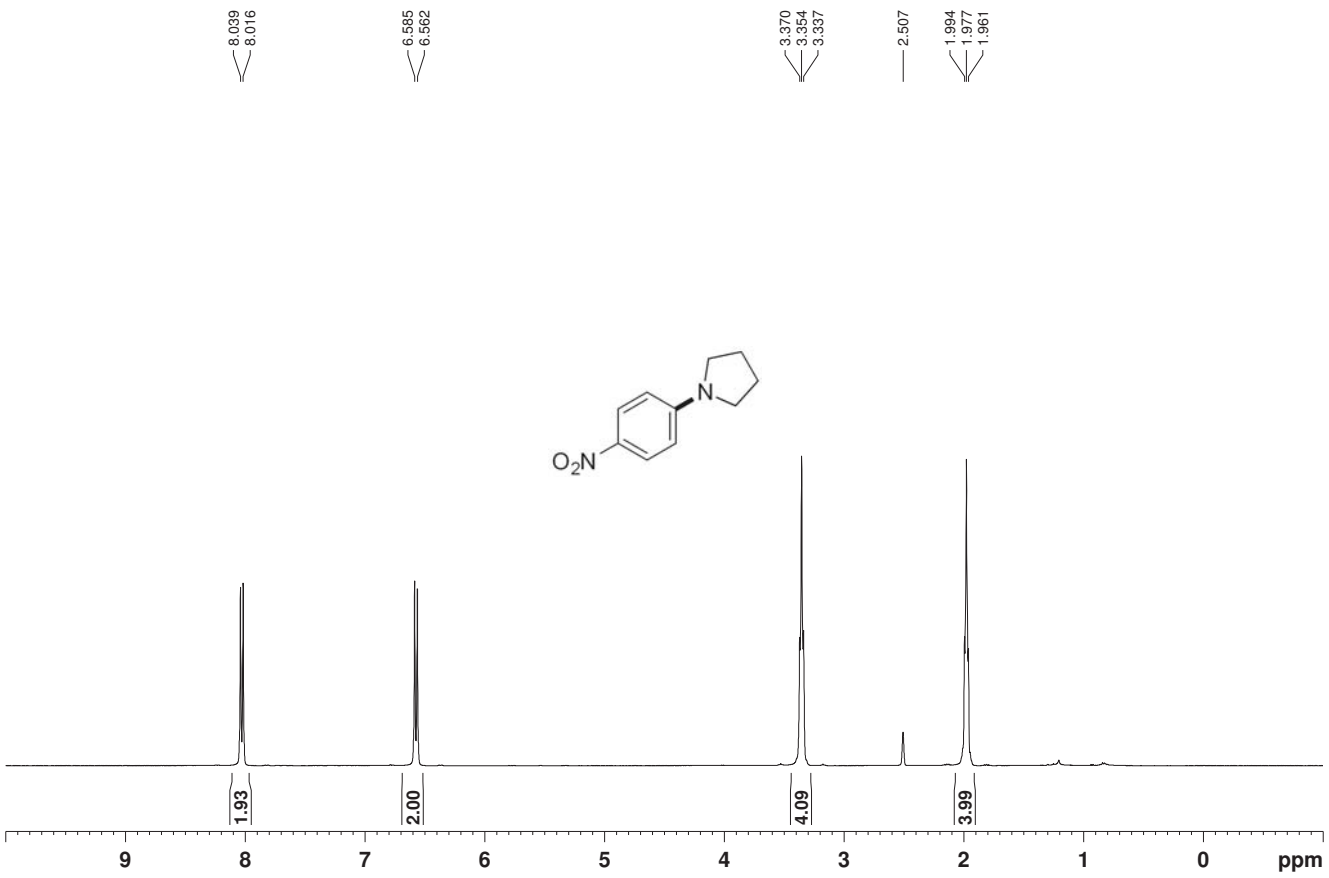
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6
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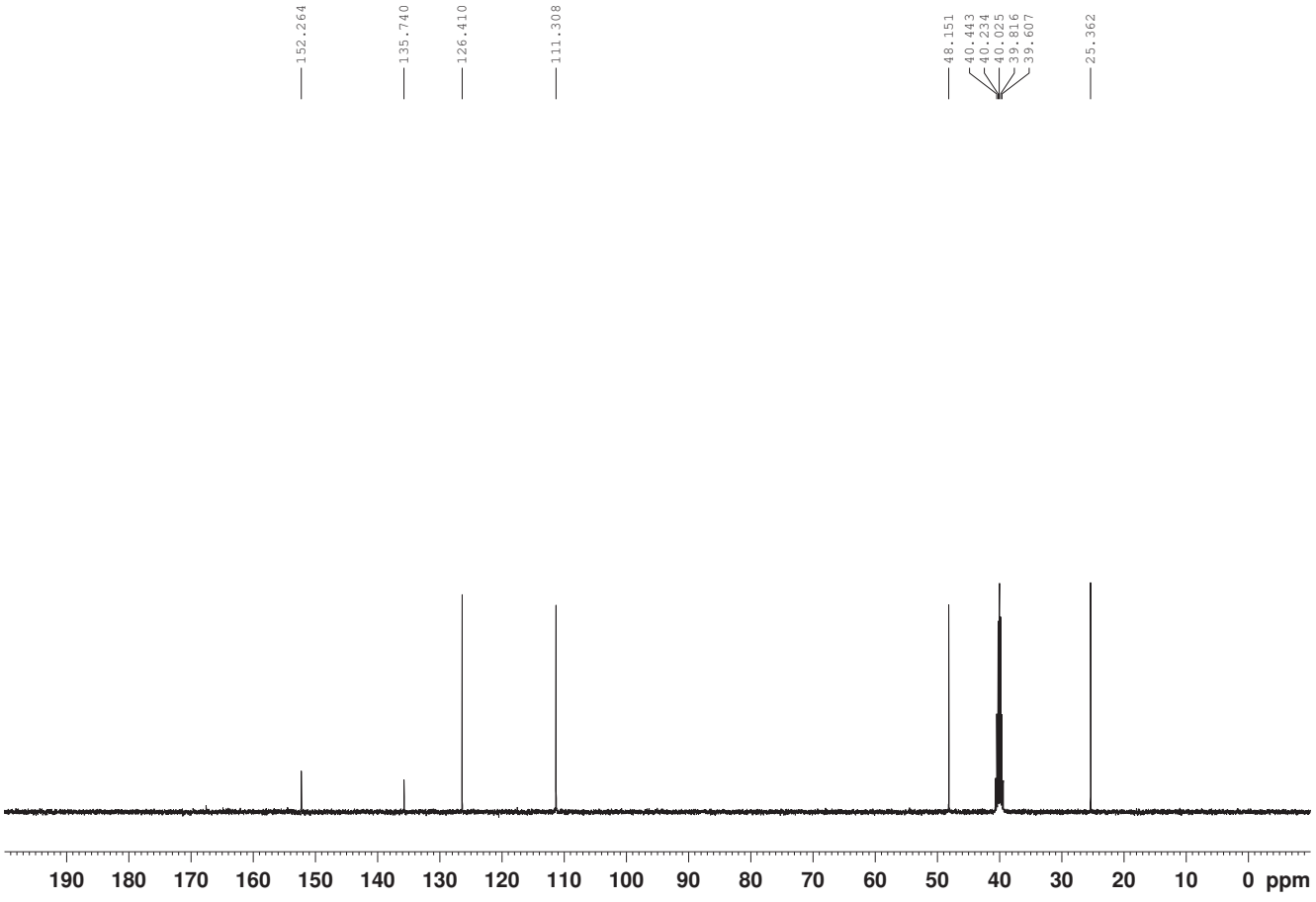
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40.025
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39.607
25.362
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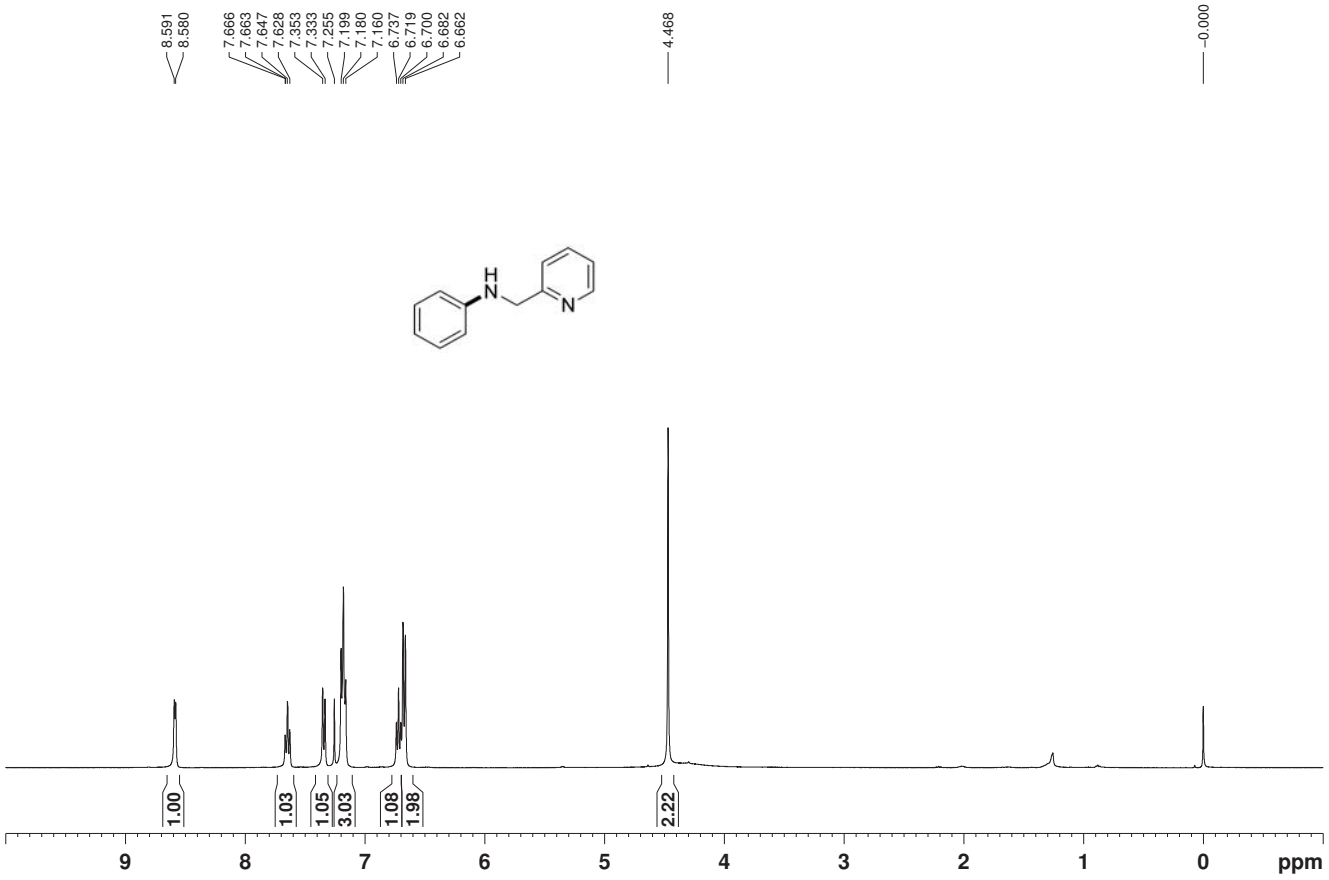
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7.180
7.160
6.737
6.719
6.700
6.682
6.662
4.468
0.000
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1.08
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6
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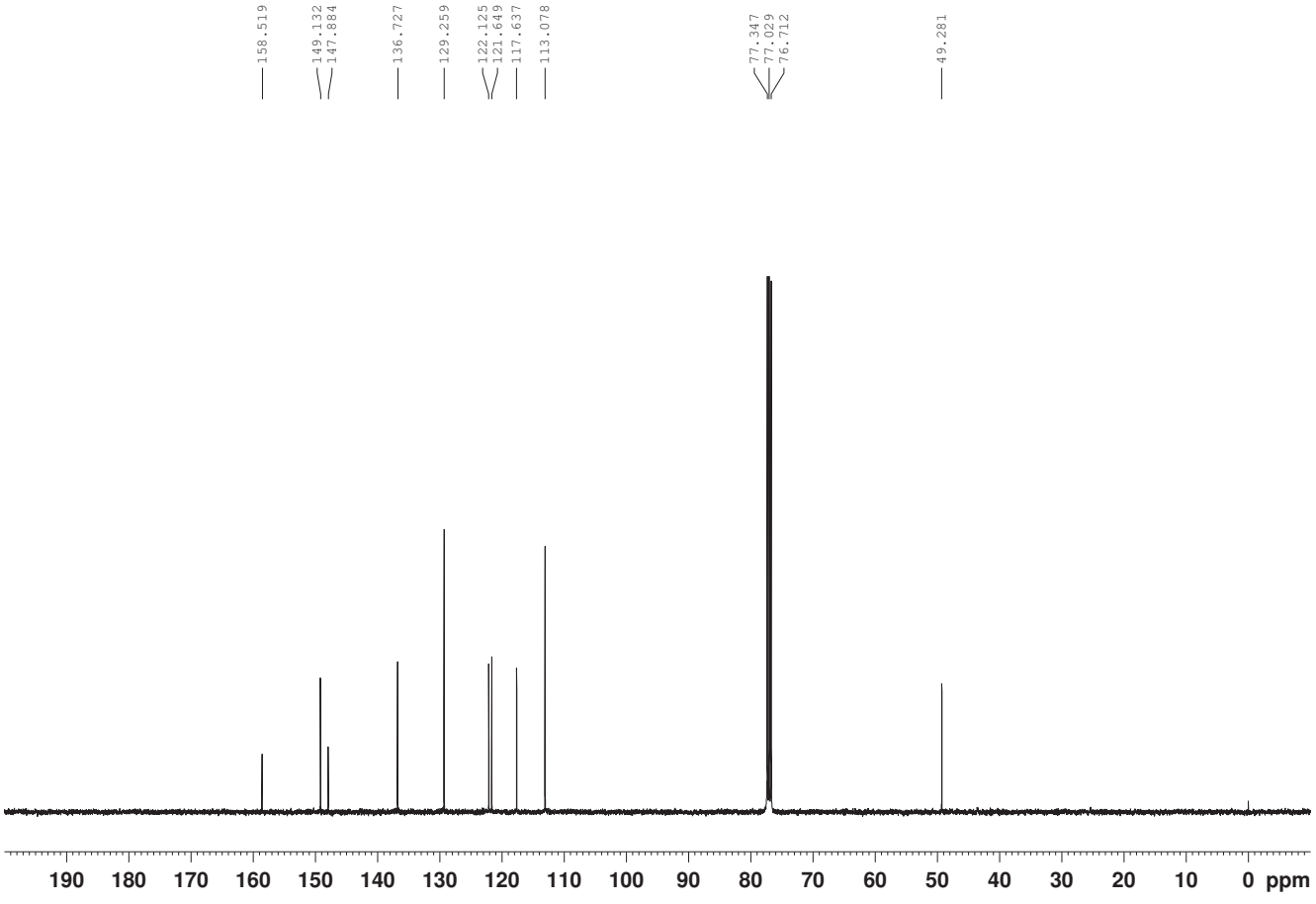
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121.649
117.637
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77.347
77.029
76.712
49.281
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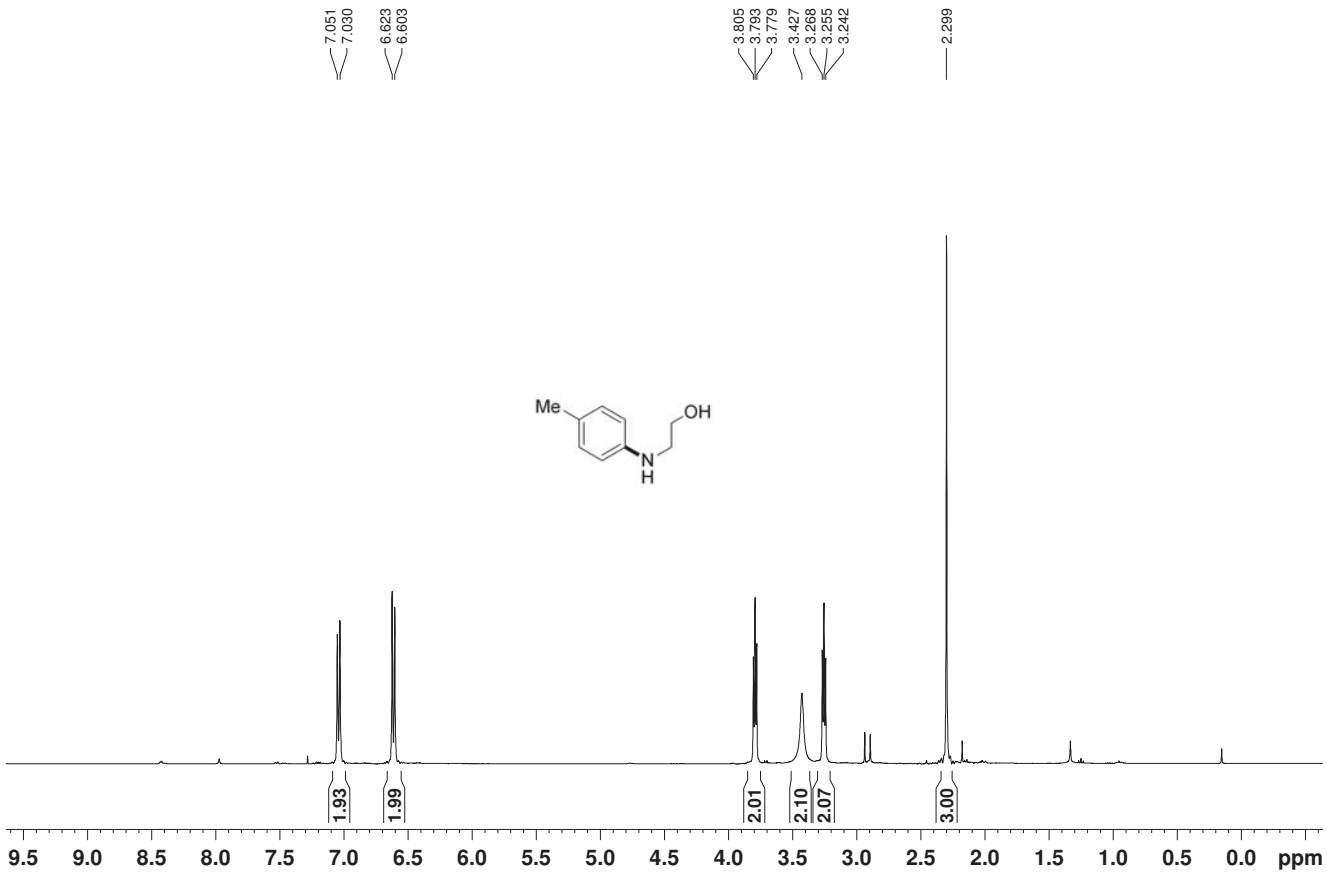
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Me
OH
N
H
1.93
1.99
2.01
2.10
2.07
3.00
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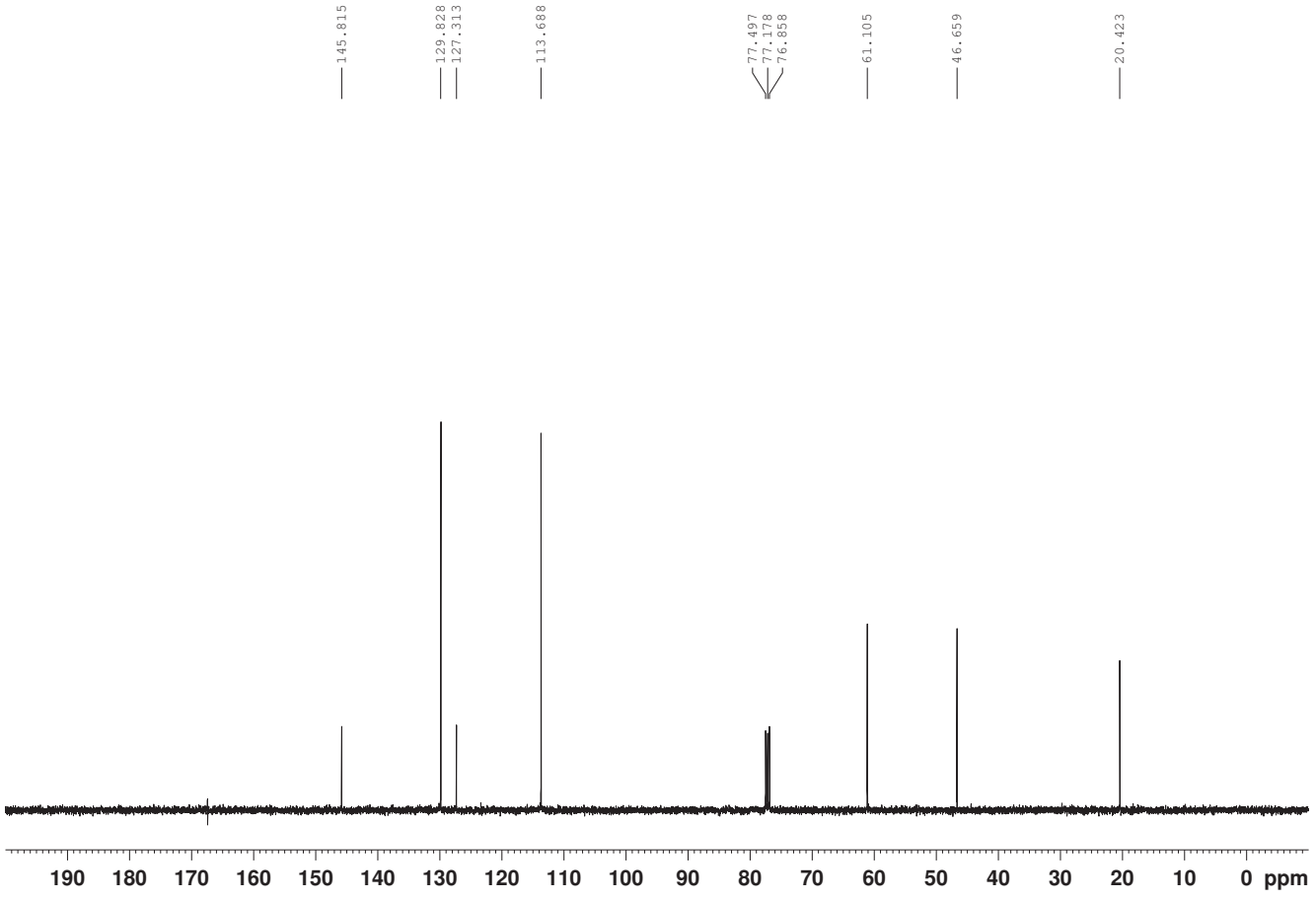
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129.828
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76.858
61.105
46.659
20.423
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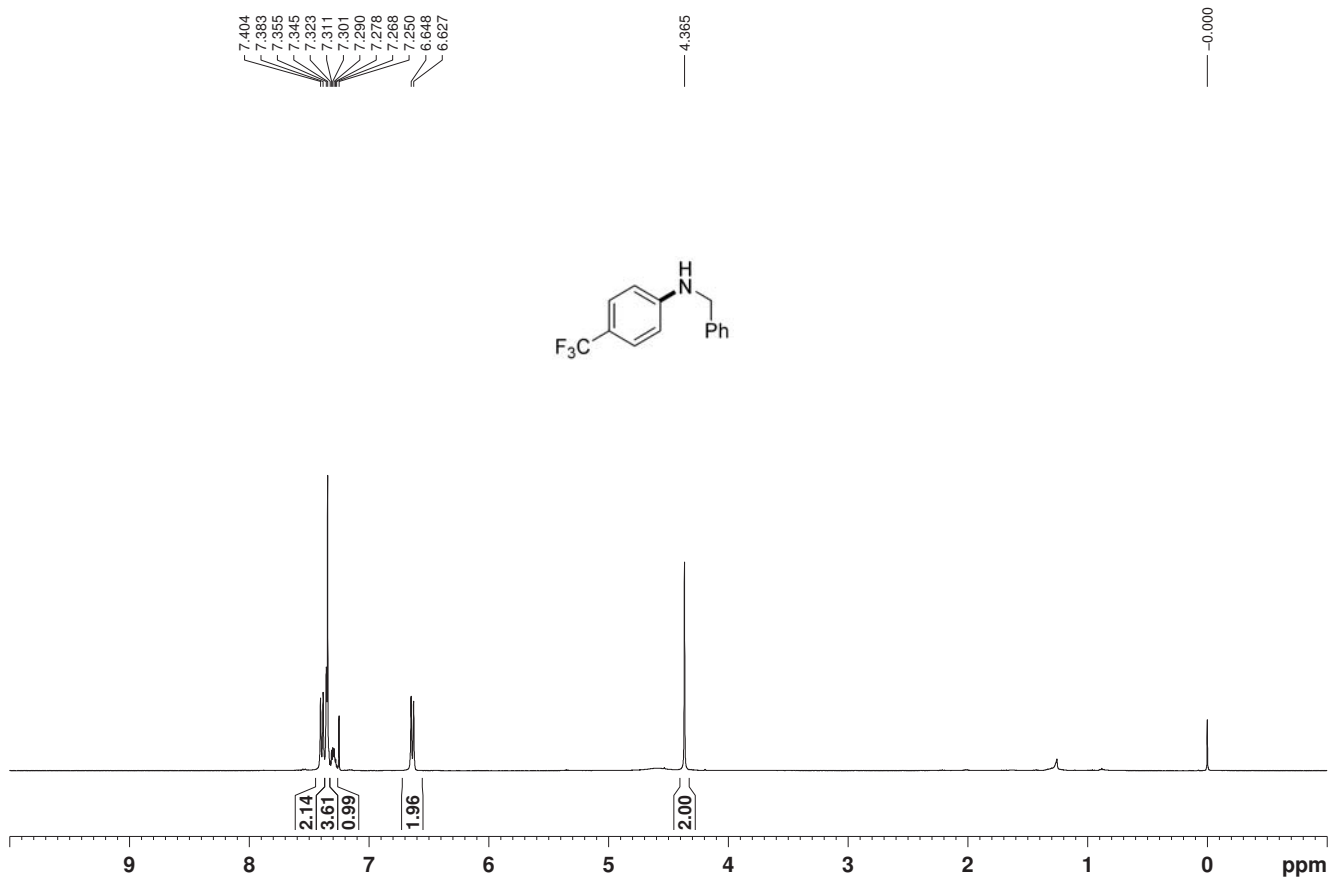
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7.290
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7.268
7.250
6.648
6.627
4.365
0.000
H
N
Ph
F3C
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6
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3
2
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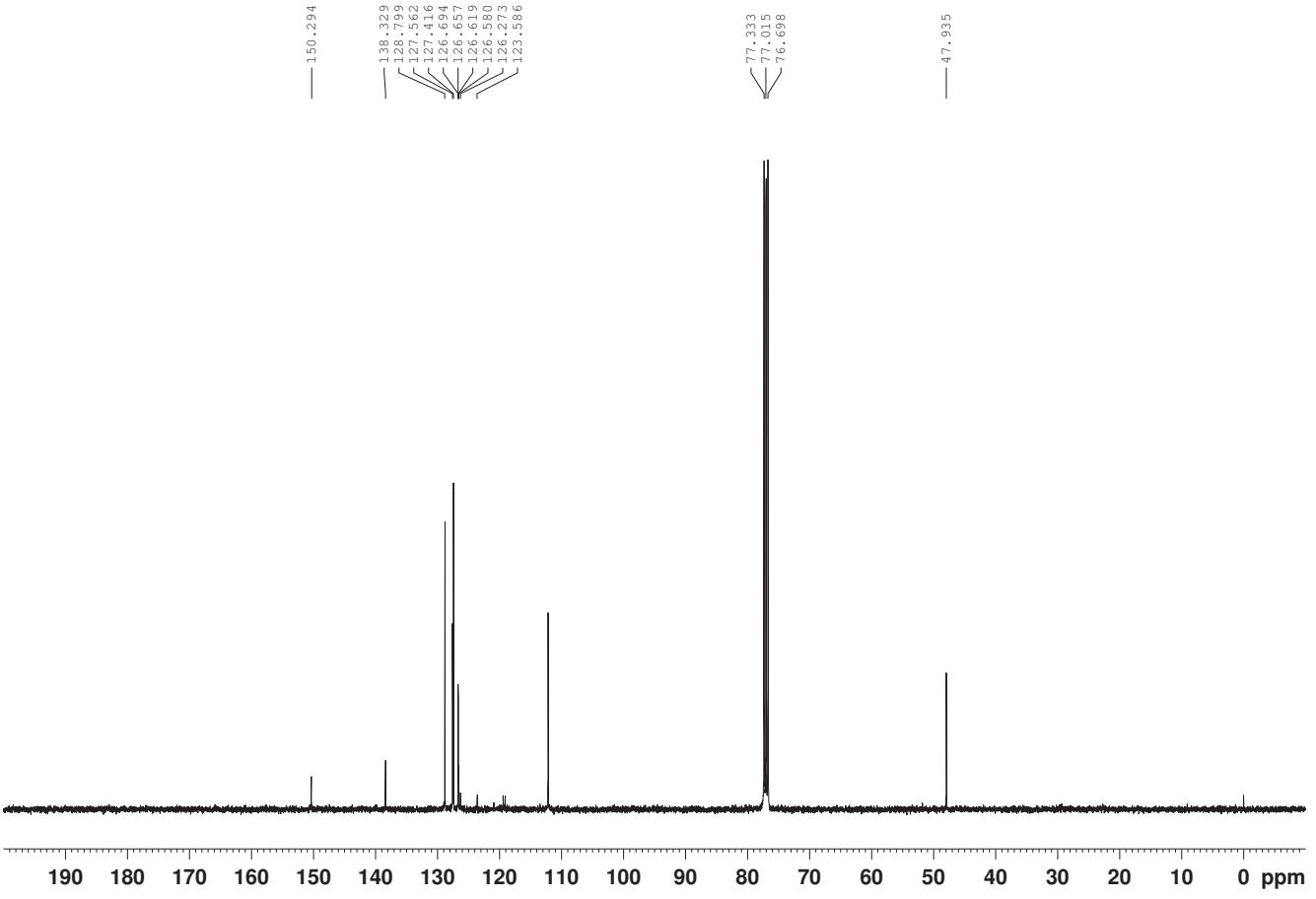
0
ppm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126.619
126.580
126.273
123.586
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77.015
76.698
47.935
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150
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130
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110
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60
50
40
30
20
10
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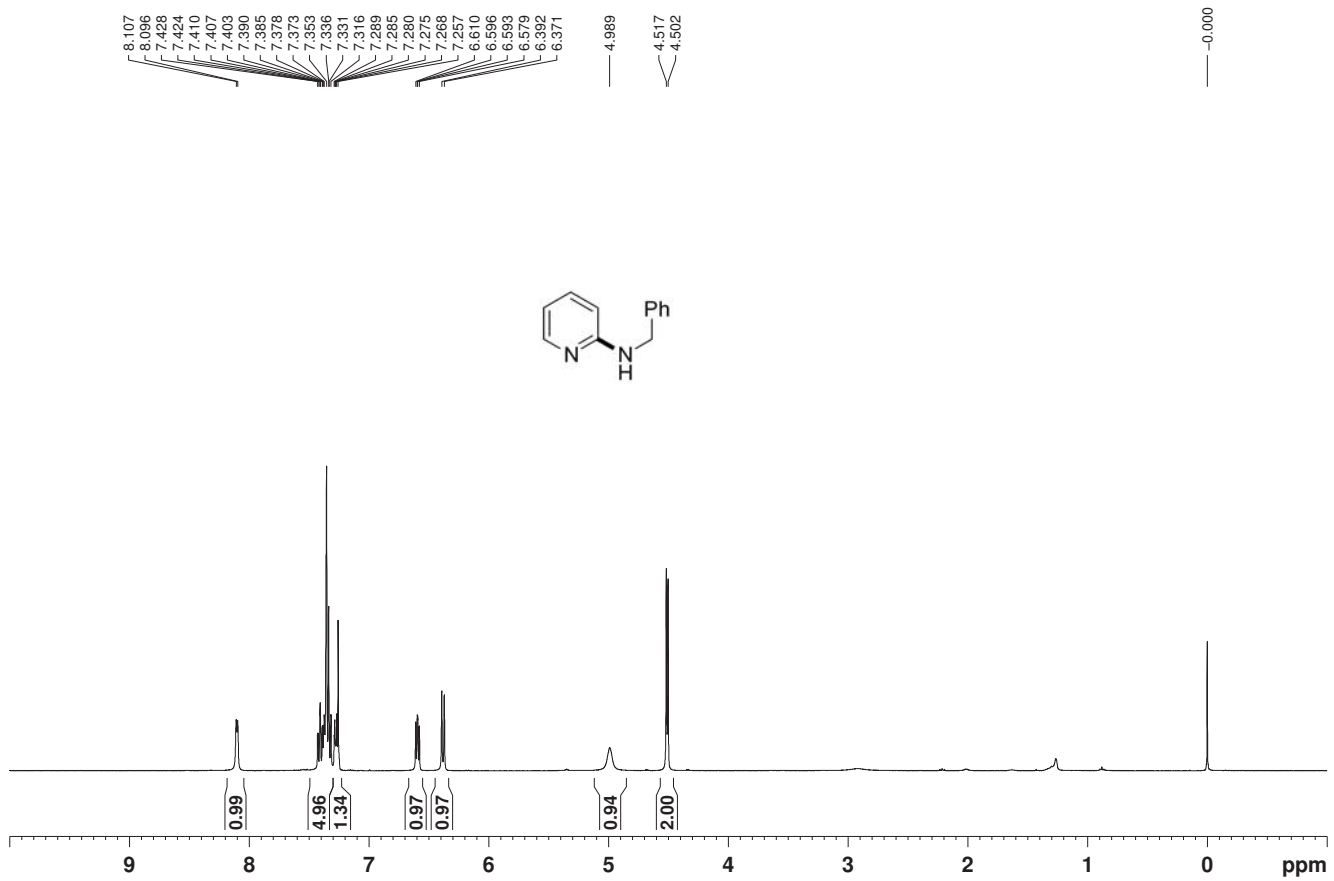
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7.373
7.353
7.336
7.331
7.316
7.289
7.285
7.280
7.275
7.268
7.257
6.610
6.596
6.593
6.579
6.392
6.371
4.989
4.517
4.502
0.000
Ph
N
H
0.99
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0.97
0.94
2.00
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6
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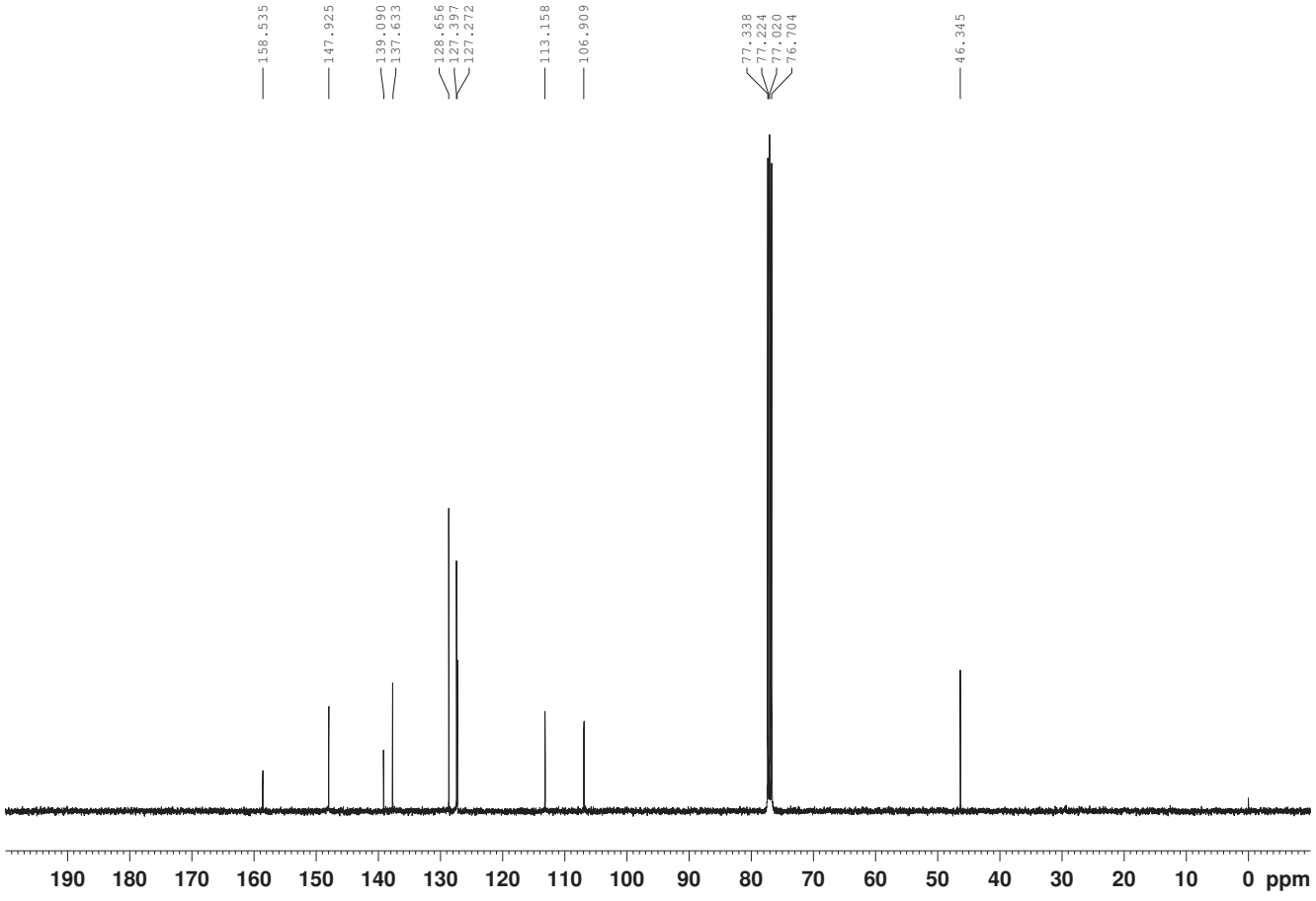
0
ppm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113.158
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77.020
76.704
46.345
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150
140
130
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110
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80
70
60
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40
30
20
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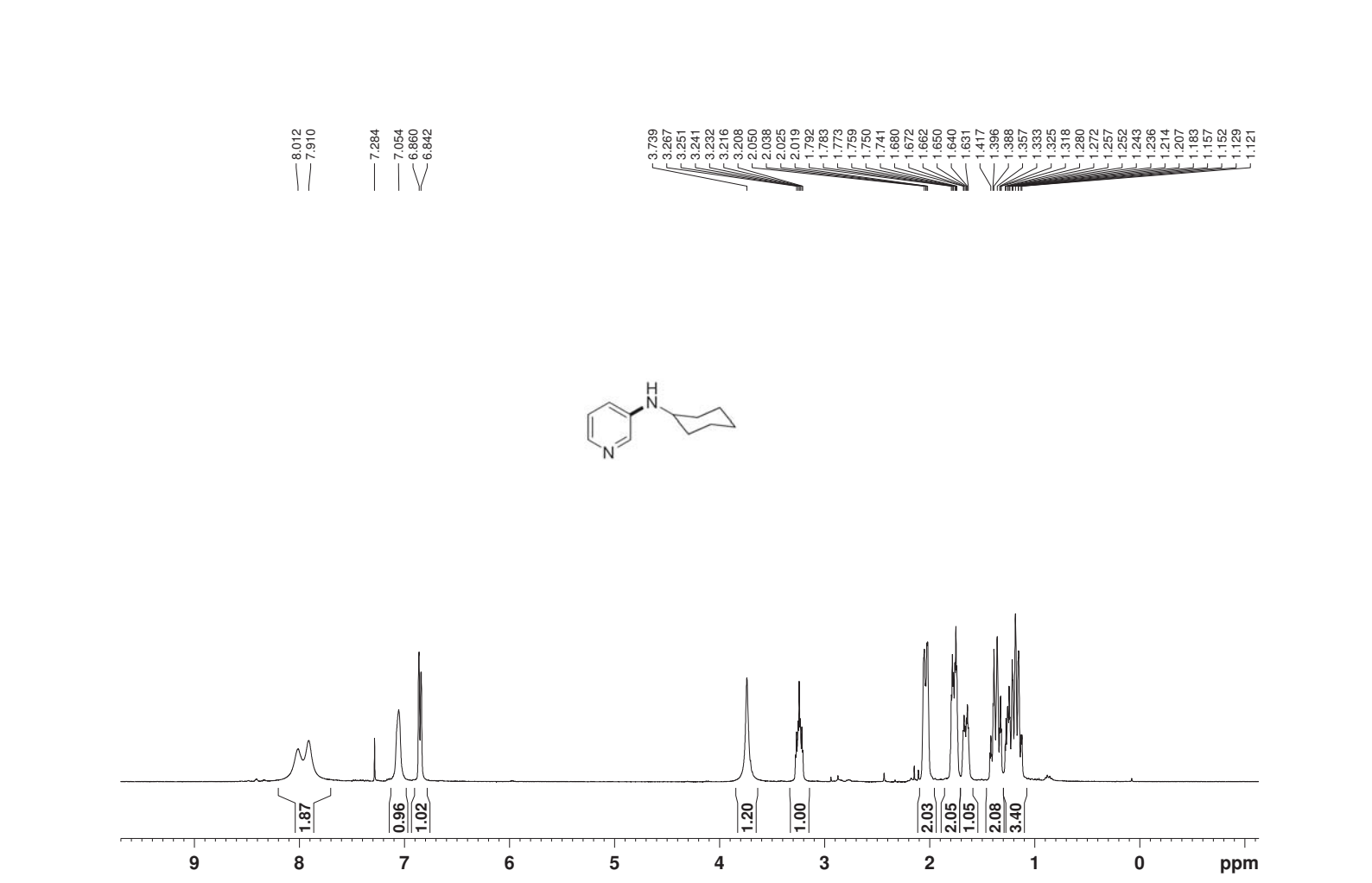
0 ppm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3.267
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3.241
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3.216
3.208
2.050
2.038
2.025
2.019
1.792
1.783
1.773
1.759
1.750
1.741
1.680
1.672
1.662
1.650
1.640
1.631
1.417
1.396
1.388
1.357
1.333
1.325
1.318
1.280
1.272
1.257
1.252
1.243
1.236
1.214
1.207
1.183
1.157
1.152
1.129
1.121
1.87
0.96
1.02
1.20
1.00
2.03
2.05
1.05
2.08
3.40
9
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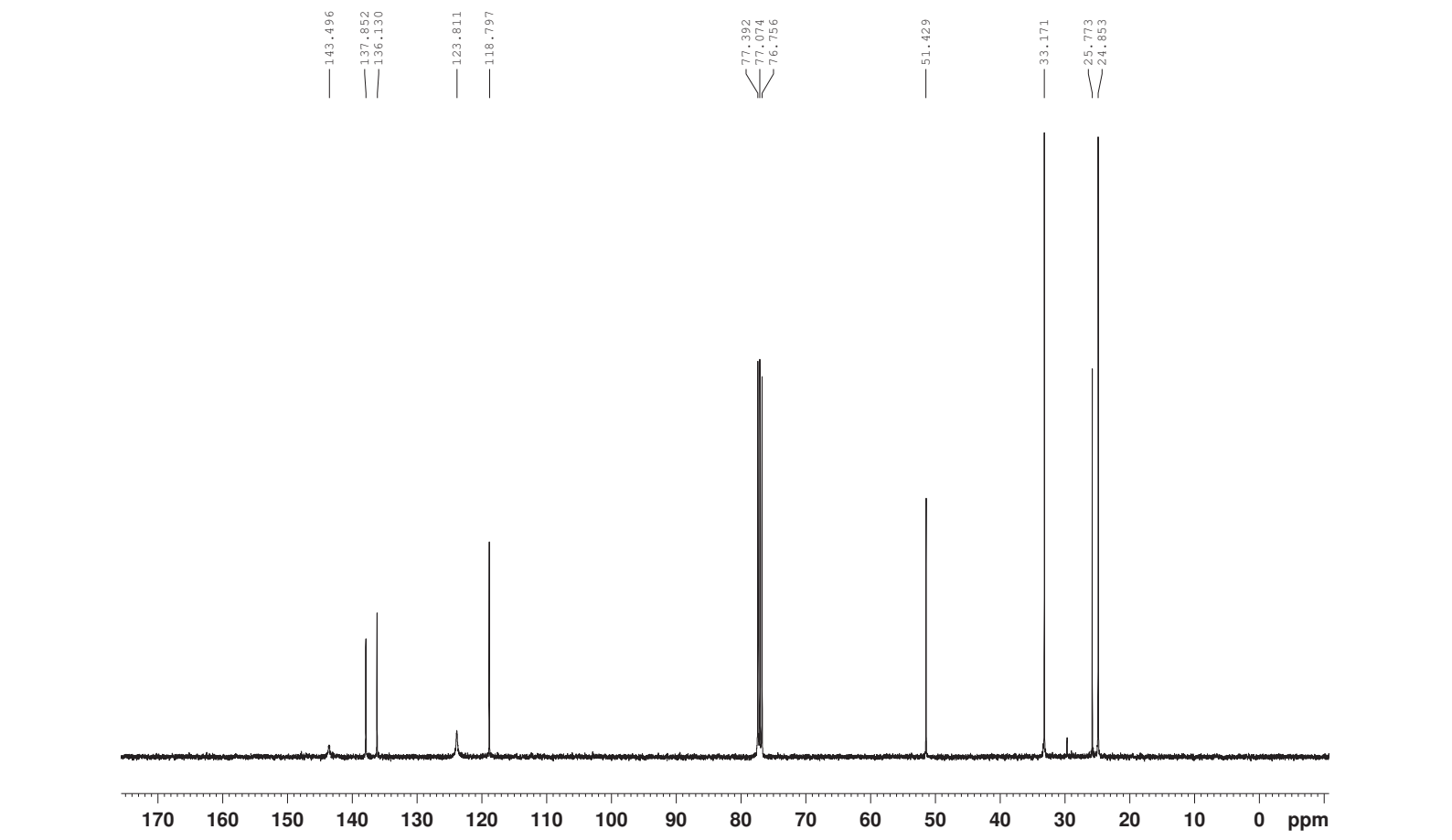
7
6
5
4
3
2
1
0
ppm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77.074
76.756
51.429
33.171
25.773
24.853
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.139
8.129
8.127
7.455
7.451
7.437
7.434
7.431
7.417
7.412
7.283
7.225
7.223
7.213
7.210
7.034
7.031
7.025
7.023
6.983
6.974
6.971
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6.631
6.628
6.615
6.614
6.458
6.437
5.074
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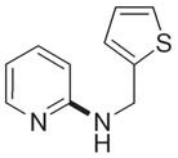
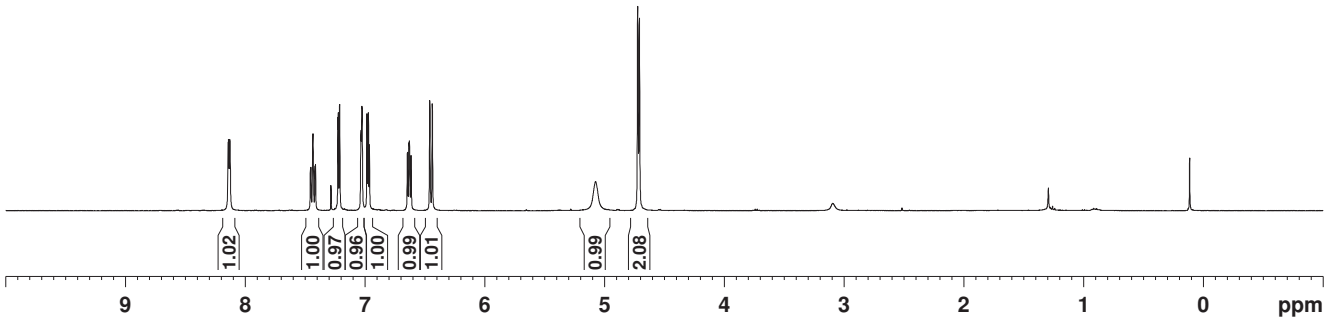
4.708


S
N
N
H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1.00
0.97
0.96
1.00
0.99
1.01
0.99
2.08
9
8
7
6
5
4
3
2
1
0
ppm


158.111
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137.529
126.830
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107.403
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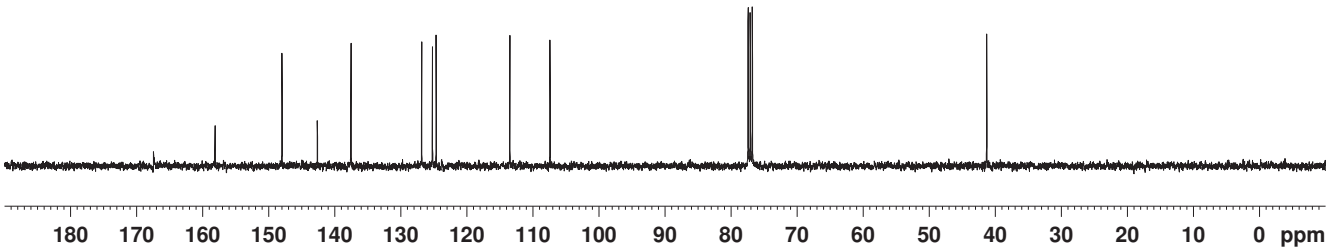
77.090
76.773
41.283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170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
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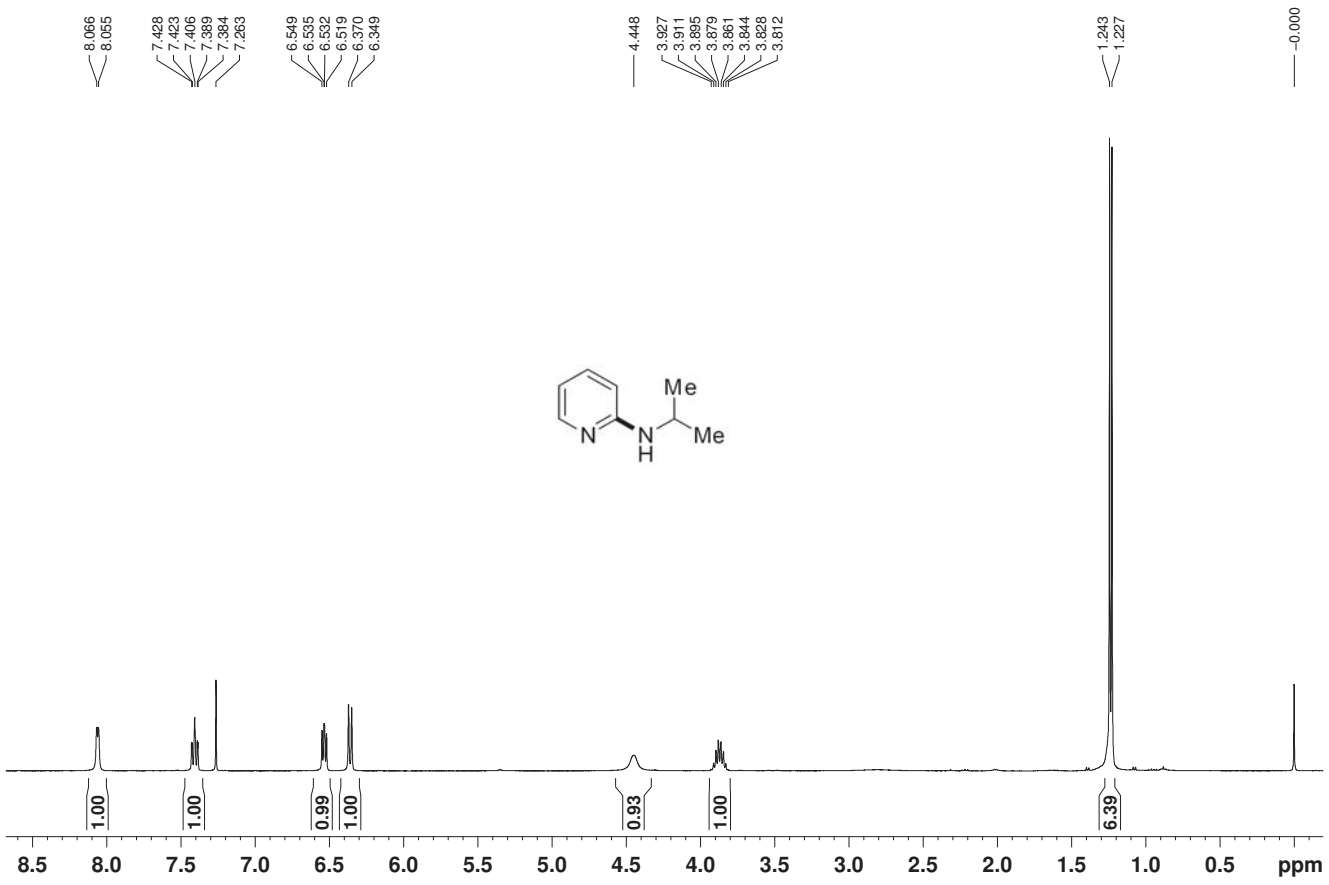
0
ppm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7.428
7.423
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7.389
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7.263
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6.535
6.532
6.519
6.370
6.349
4.448
3.927
3.911
3.895
3.879
3.861
3.844
3.828
3.812
1.243
1.227
0.000
Me
N
N
H
Me
1.00
1.00
0.99
1.00
0.93
1.00
6.39
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
3.5
3.0
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2.0
1.5
1.0
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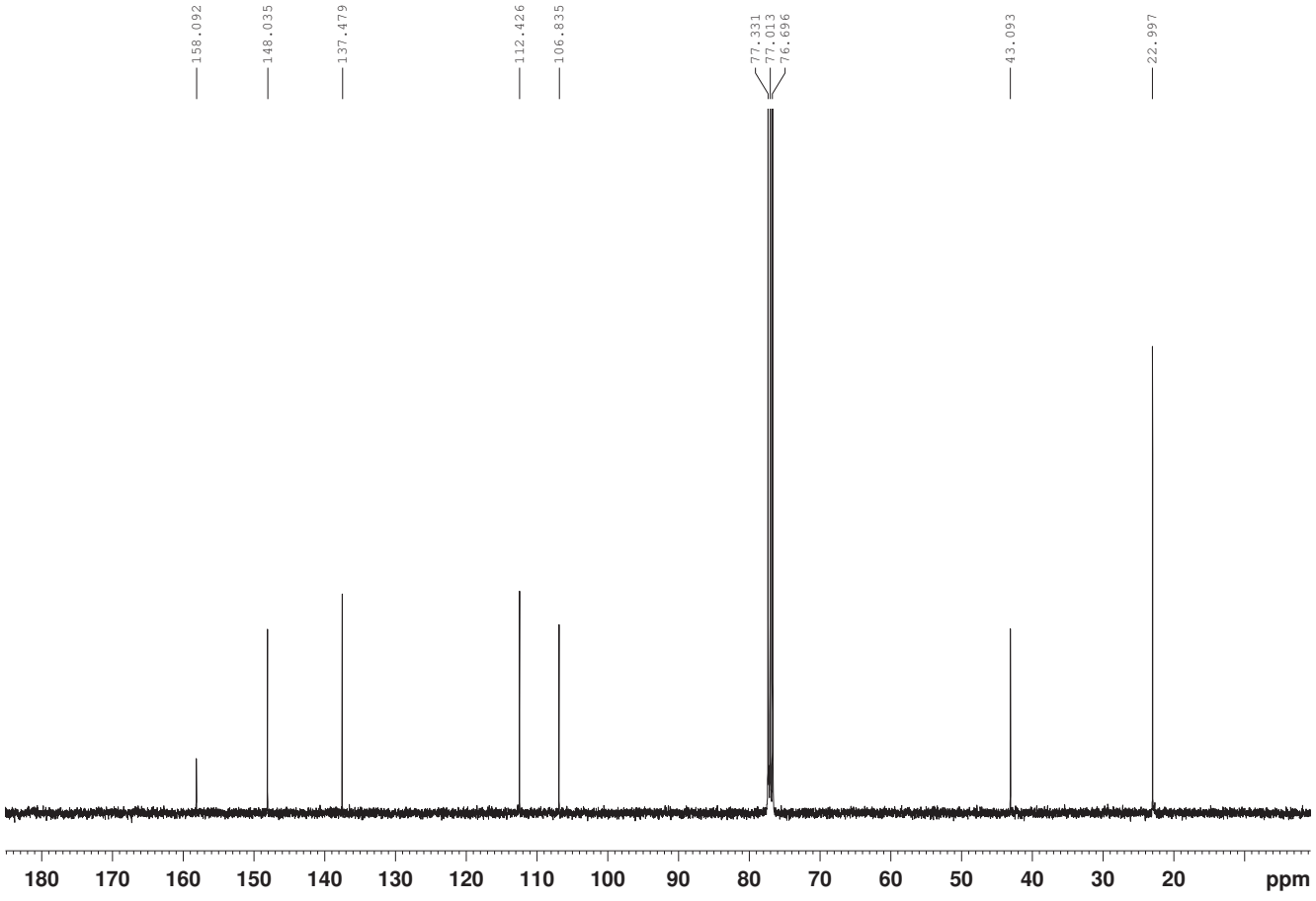
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76.696
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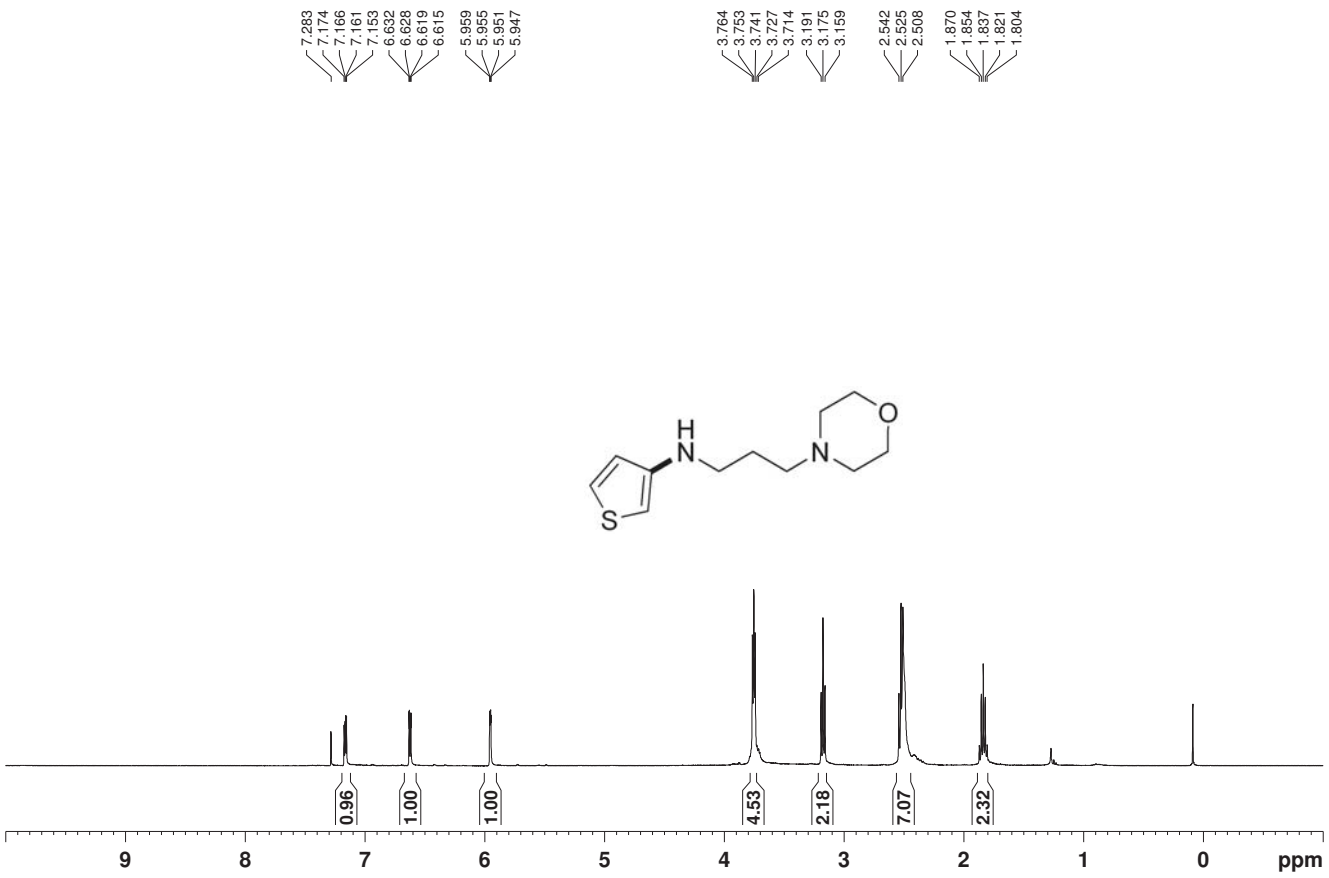
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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6.628
6.619
6.615
5.959
5.955
5.951
5.947
3.764
3.753
3.741
3.727
3.714
3.191
3.175
3.159
2.542
2.525
2.508
1.870
1.854
1.837
1.821
1.804
H
N
O
N
S
0.96
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7.07
2.32
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8
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6
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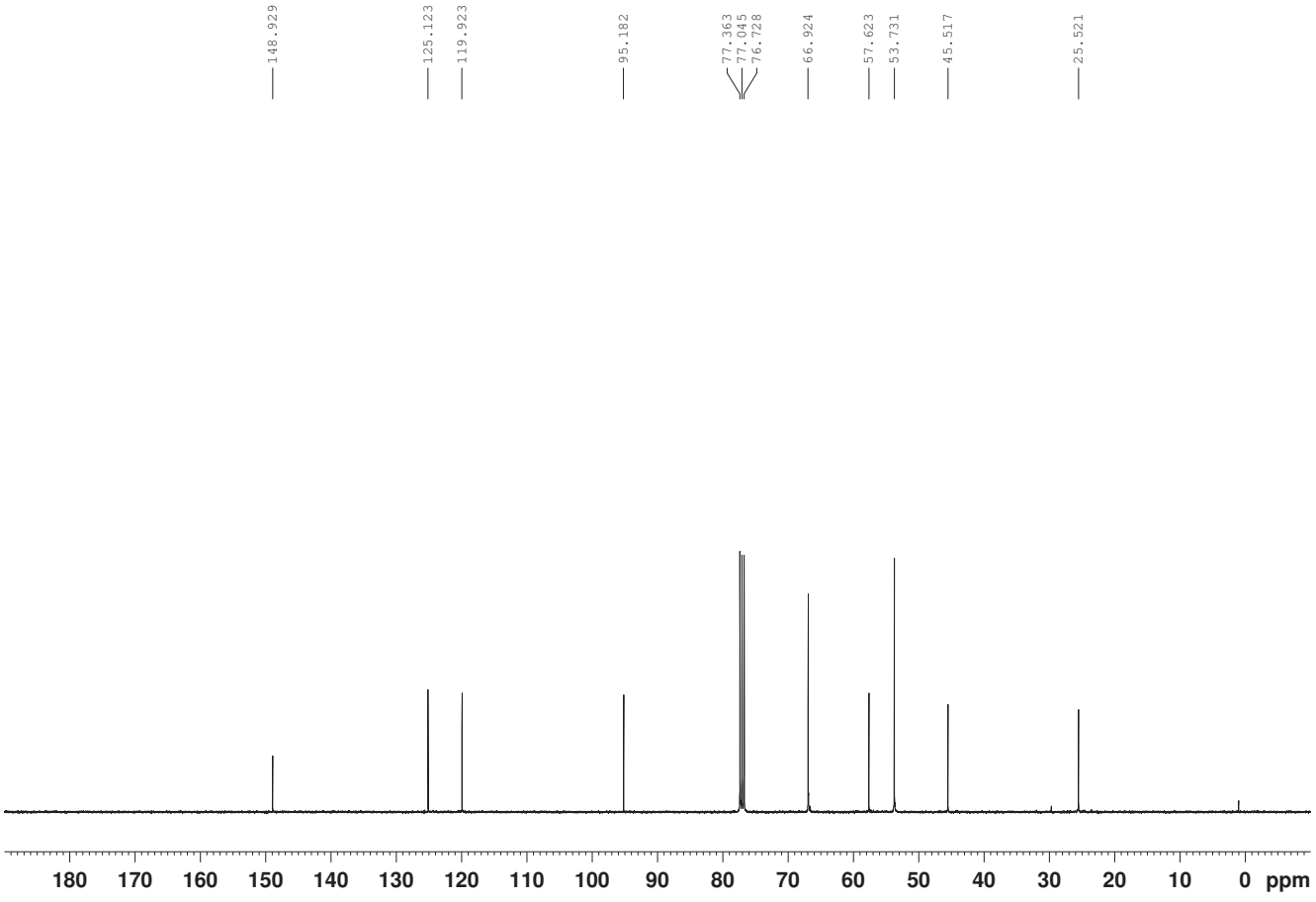
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77.045
76.728
66.924
57.623
53.731
45.517
25.521
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150
140
130
120
110
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60
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40
30
20
10
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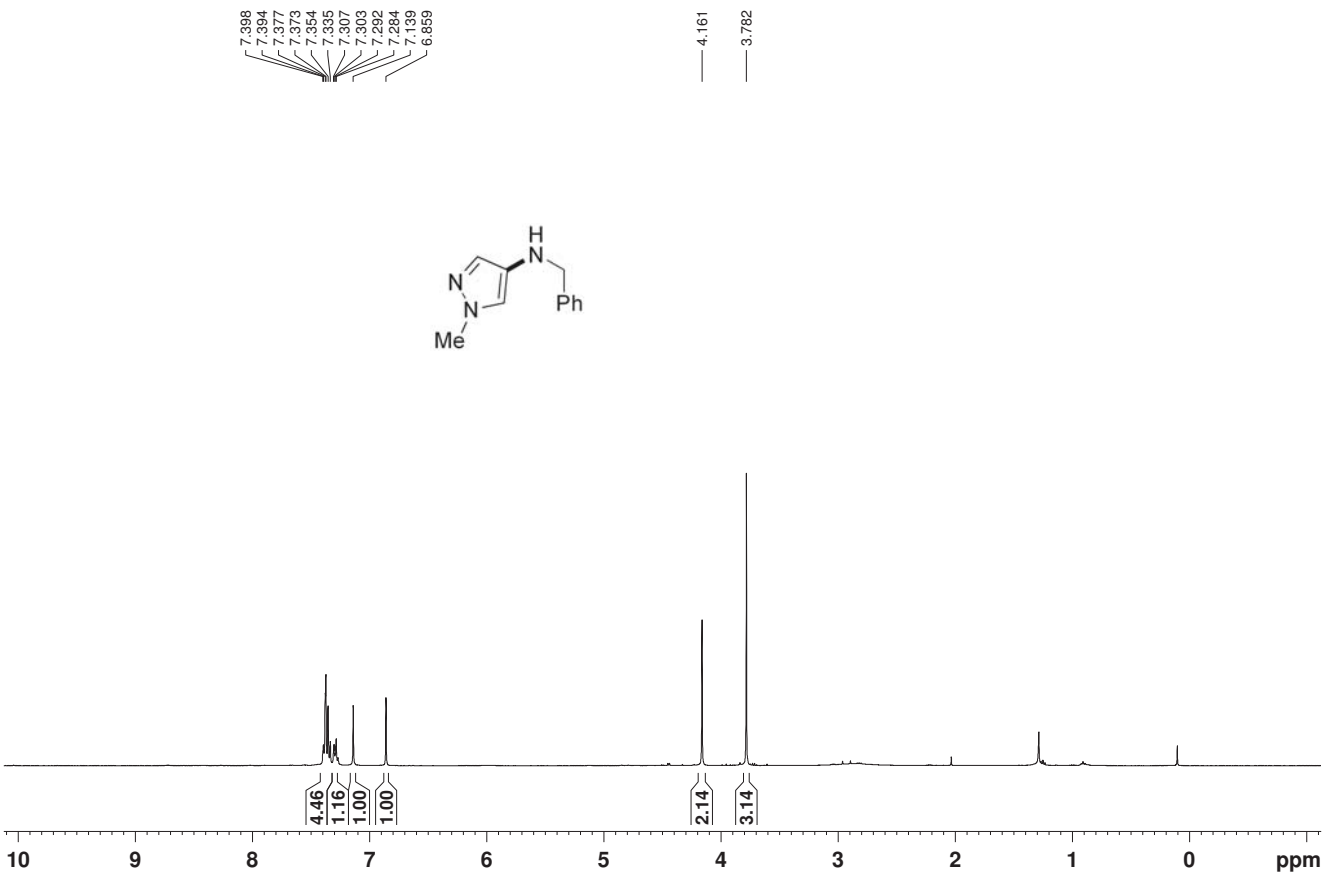
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7.335
7.307
7.303
7.292
7.284
7.139
6.859
4.161
3.782
H
N
N
N
Me
Ph
4.46
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1.00
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3.14
10
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6
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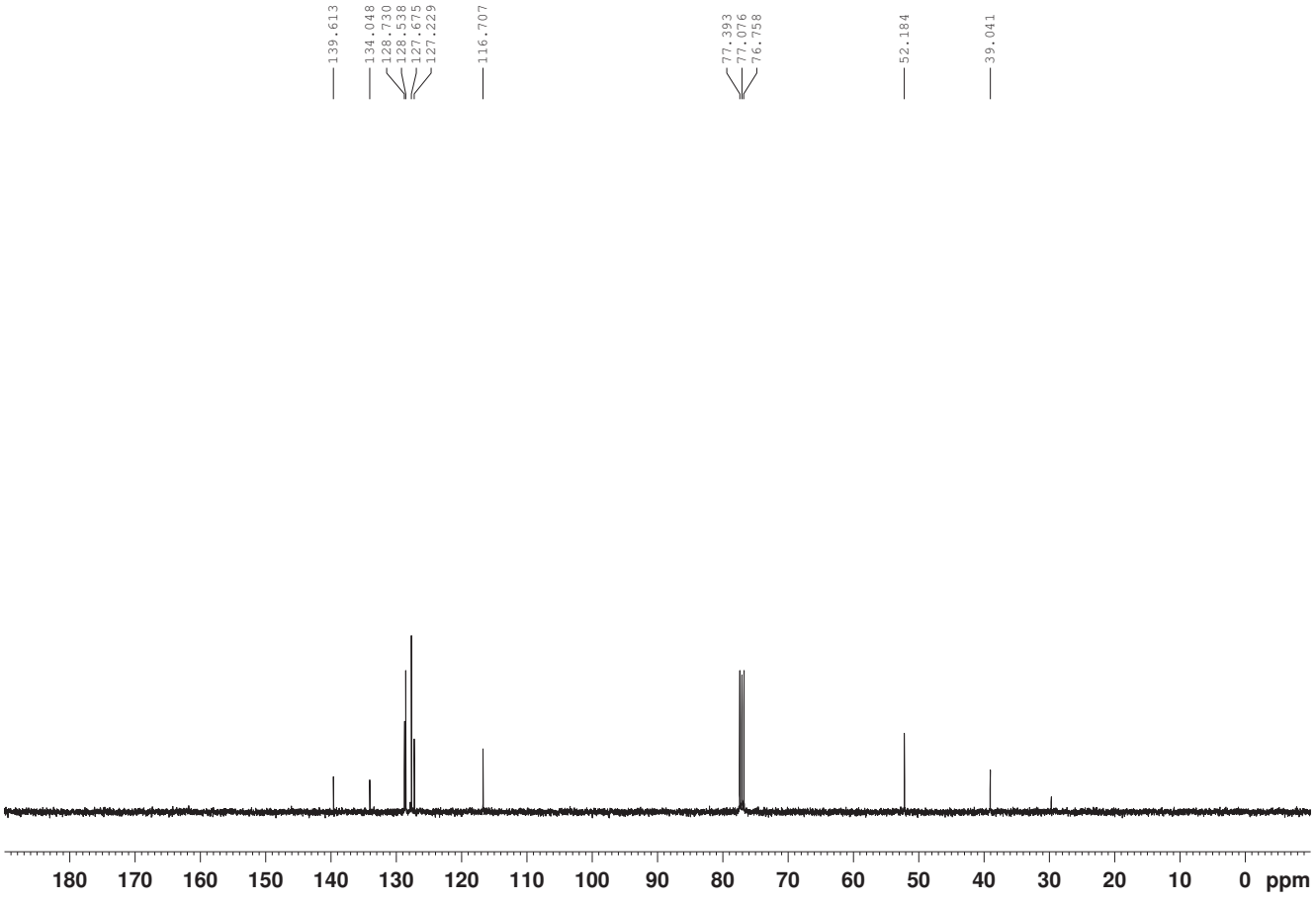
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127.229
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77.076
76.758
52.184
39.041
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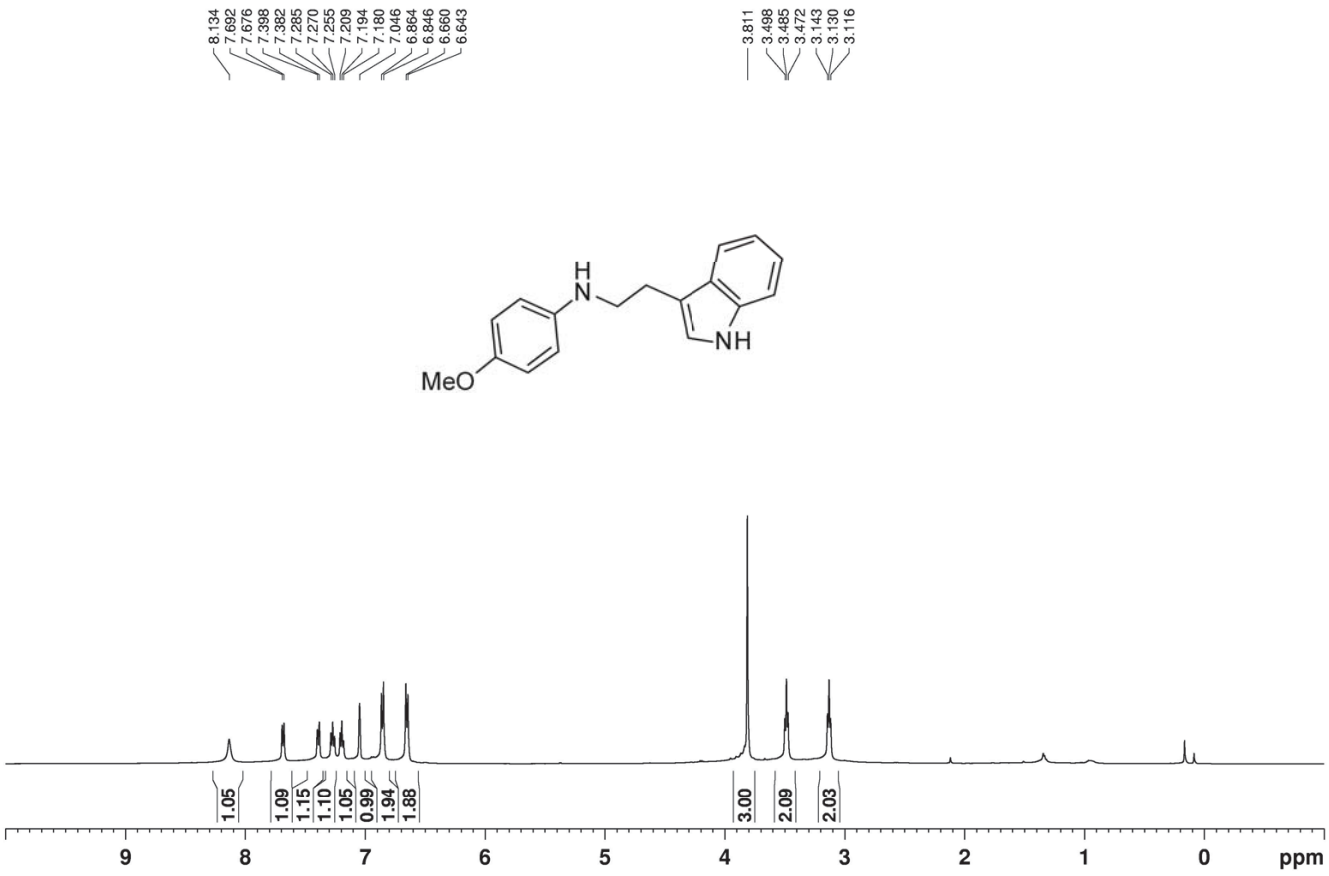
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7.209
7.194
7.180
7.046
6.864
6.846
6.660
6.643
3.811
3.498
3.485
3.472
3.143
3.130
3.116
H
N
NH
MeO
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1.09
1.15
1.10
1.05
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2.09
2.03
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6
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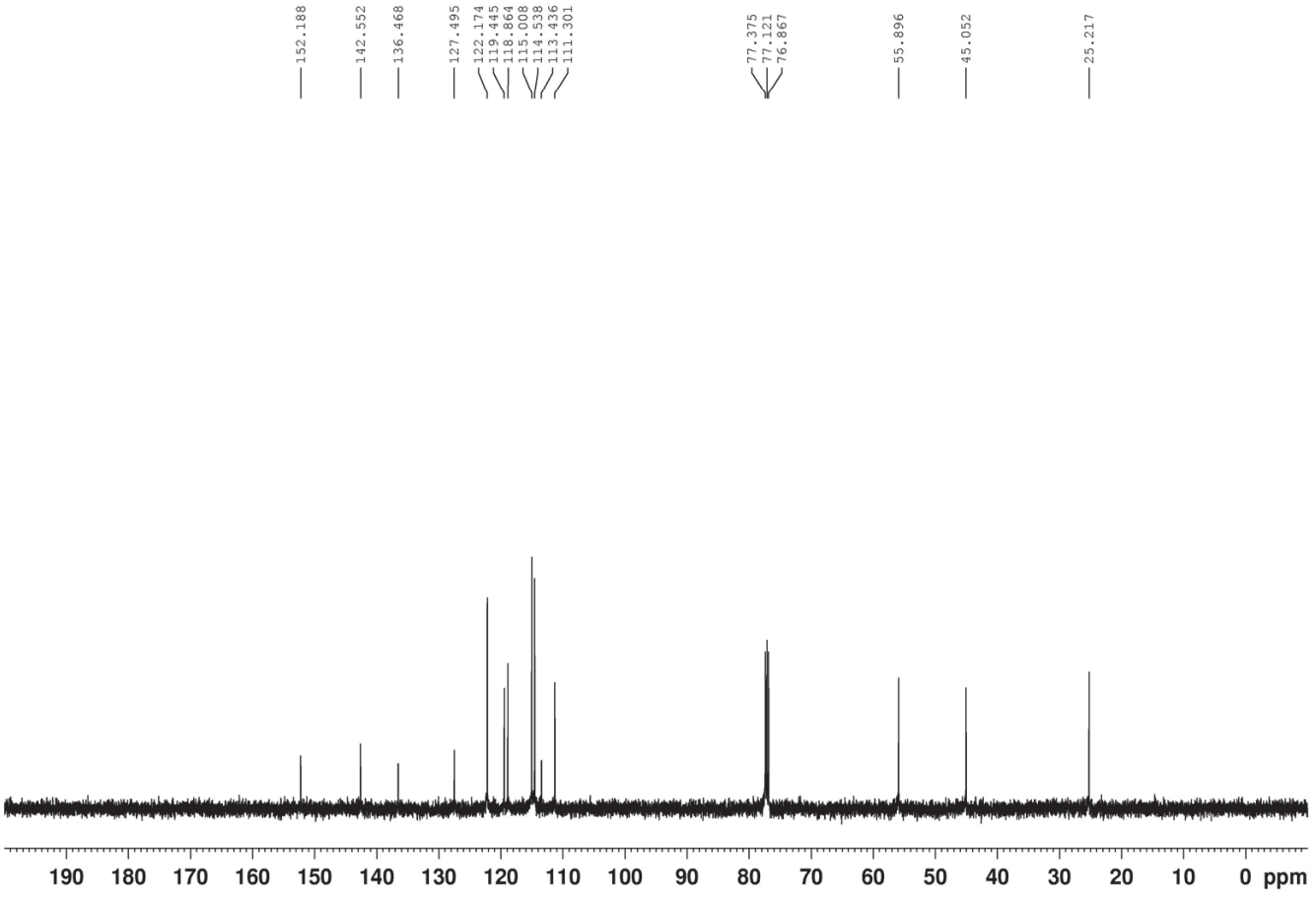
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114.538
113.436
111.301
77.375
77.121
76.867
55.896
45.052
25.217
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180
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160
150
140
130
120
110
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70
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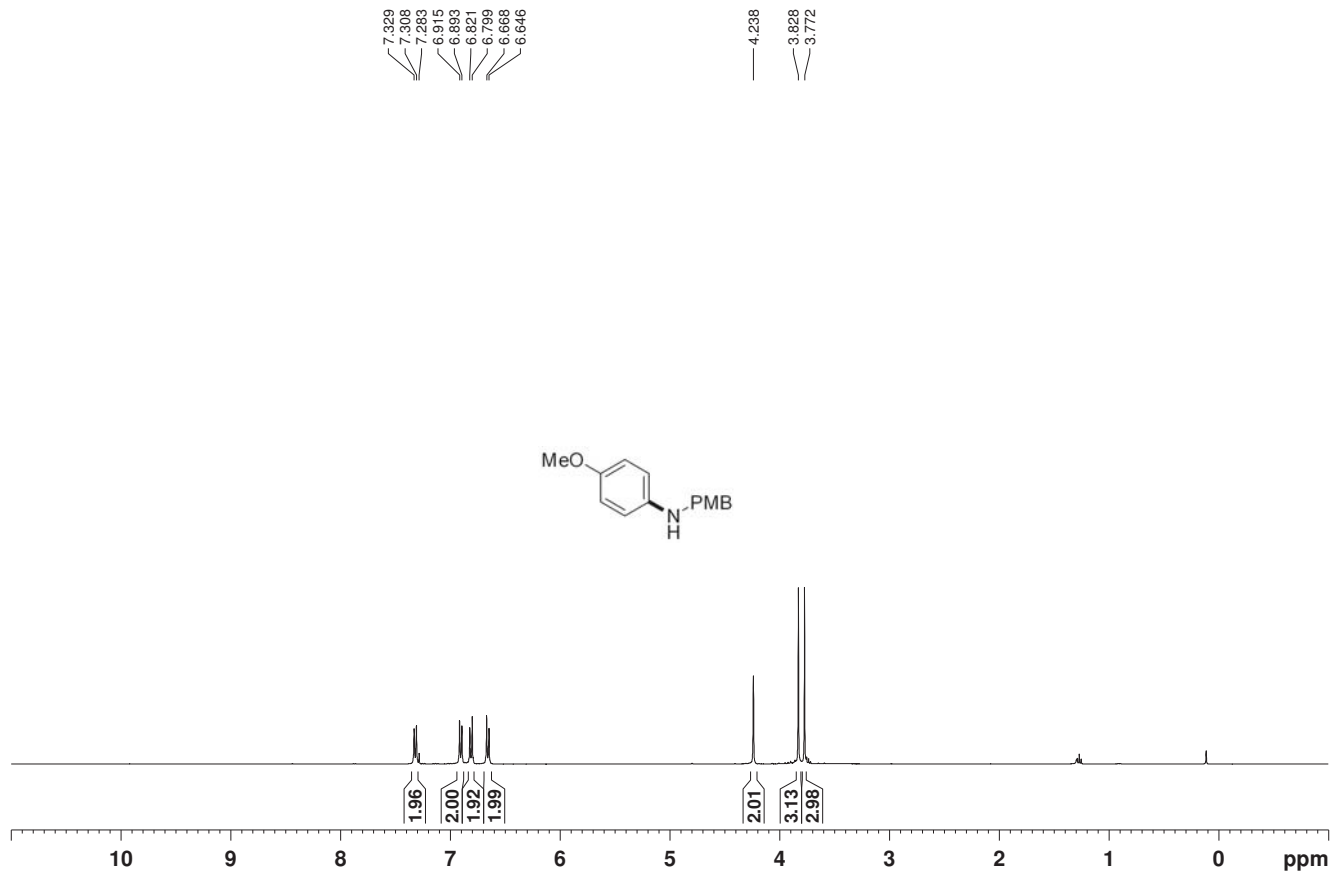
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4.238
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3.772
MeO
PMB
N
H
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2.00
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6
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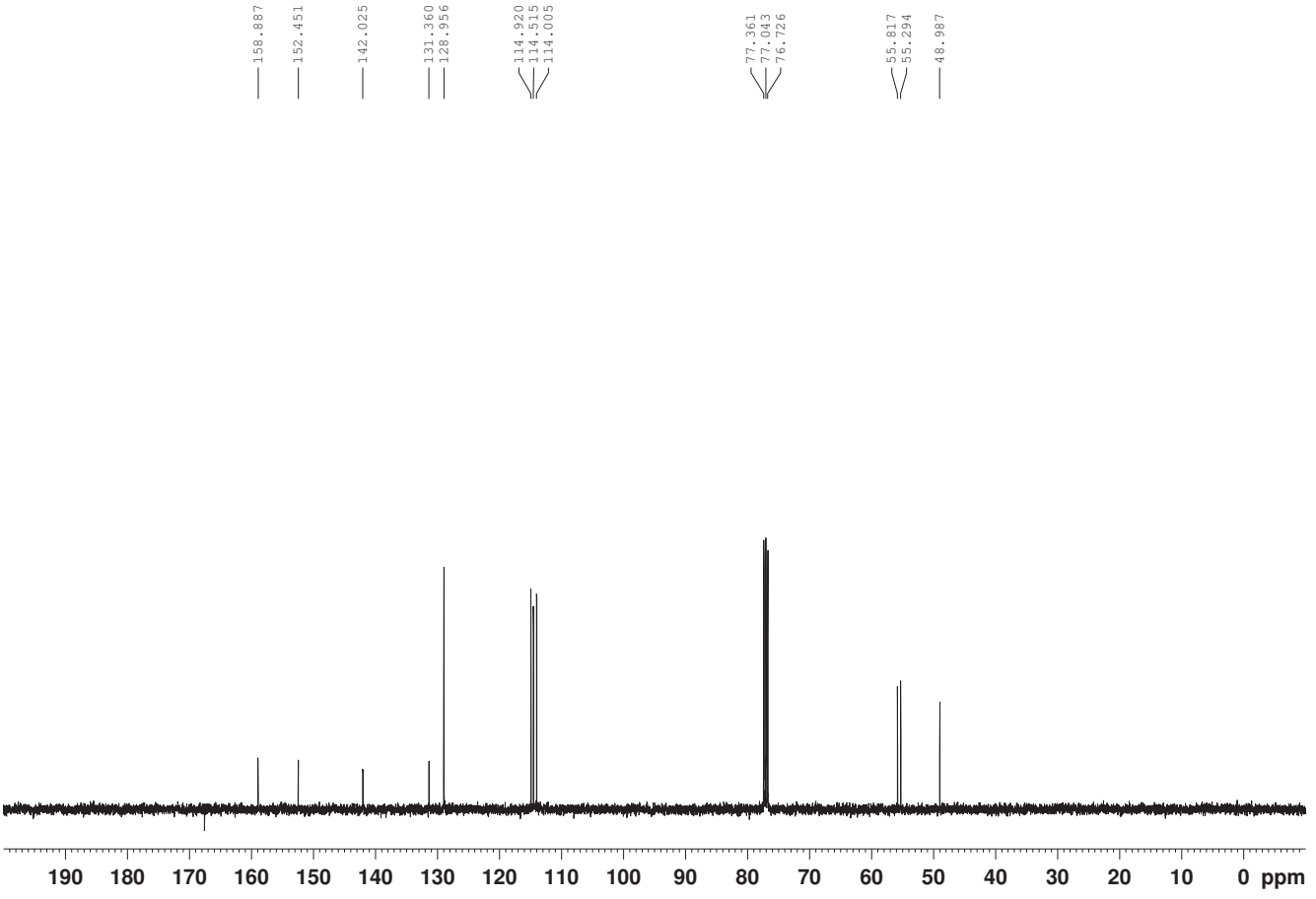
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114.005
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76.726
55.817
55.294
48.987
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150
140
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110
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70
60
50
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30
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10
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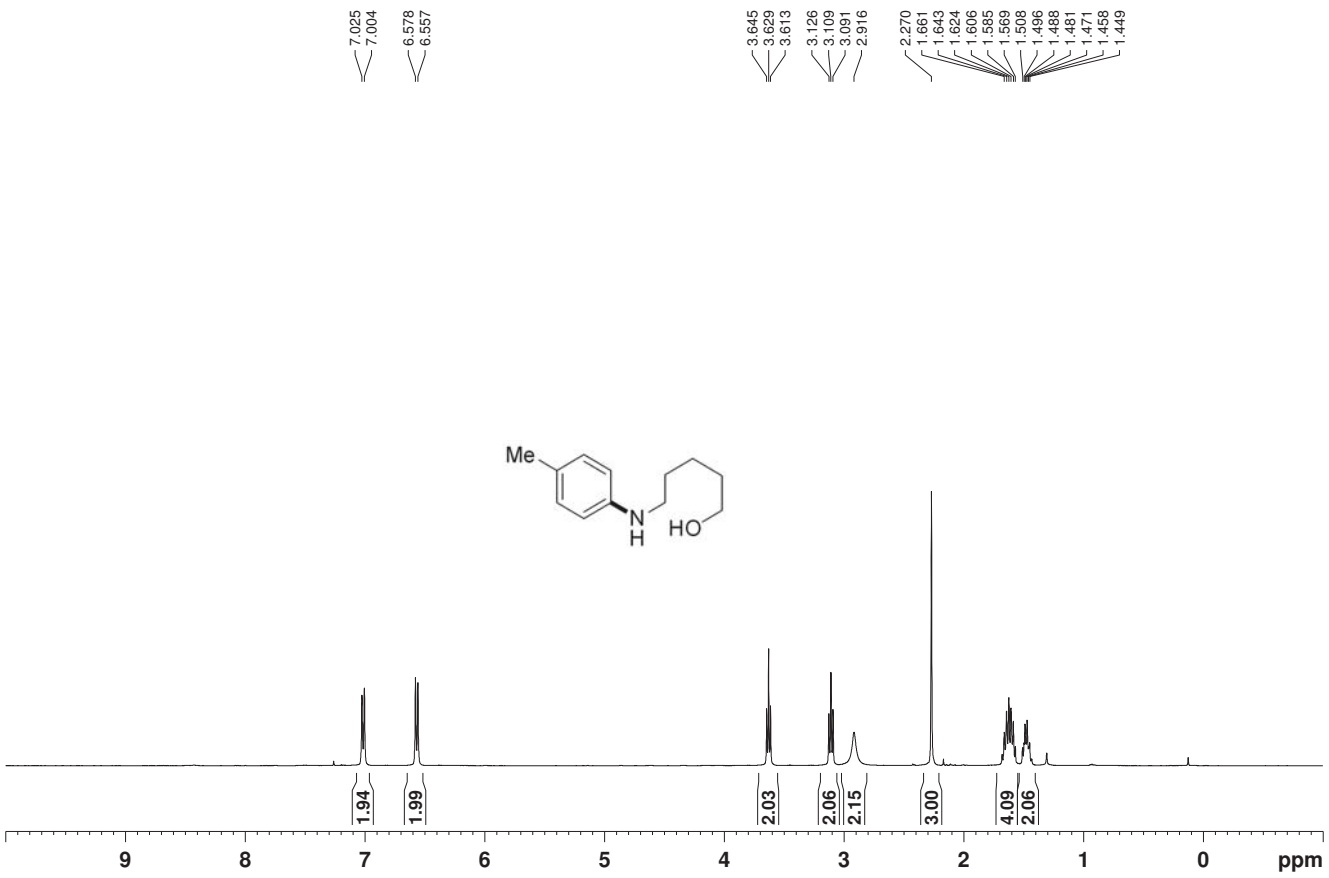
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1.661
1.643
1.624
1.606
1.585
1.569
1.508
1.496
1.488
1.481
1.471
1.458
1.449
Me
N
H
HO
1.94
1.99
2.03
2.06
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3.00
4.09
2.06
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6
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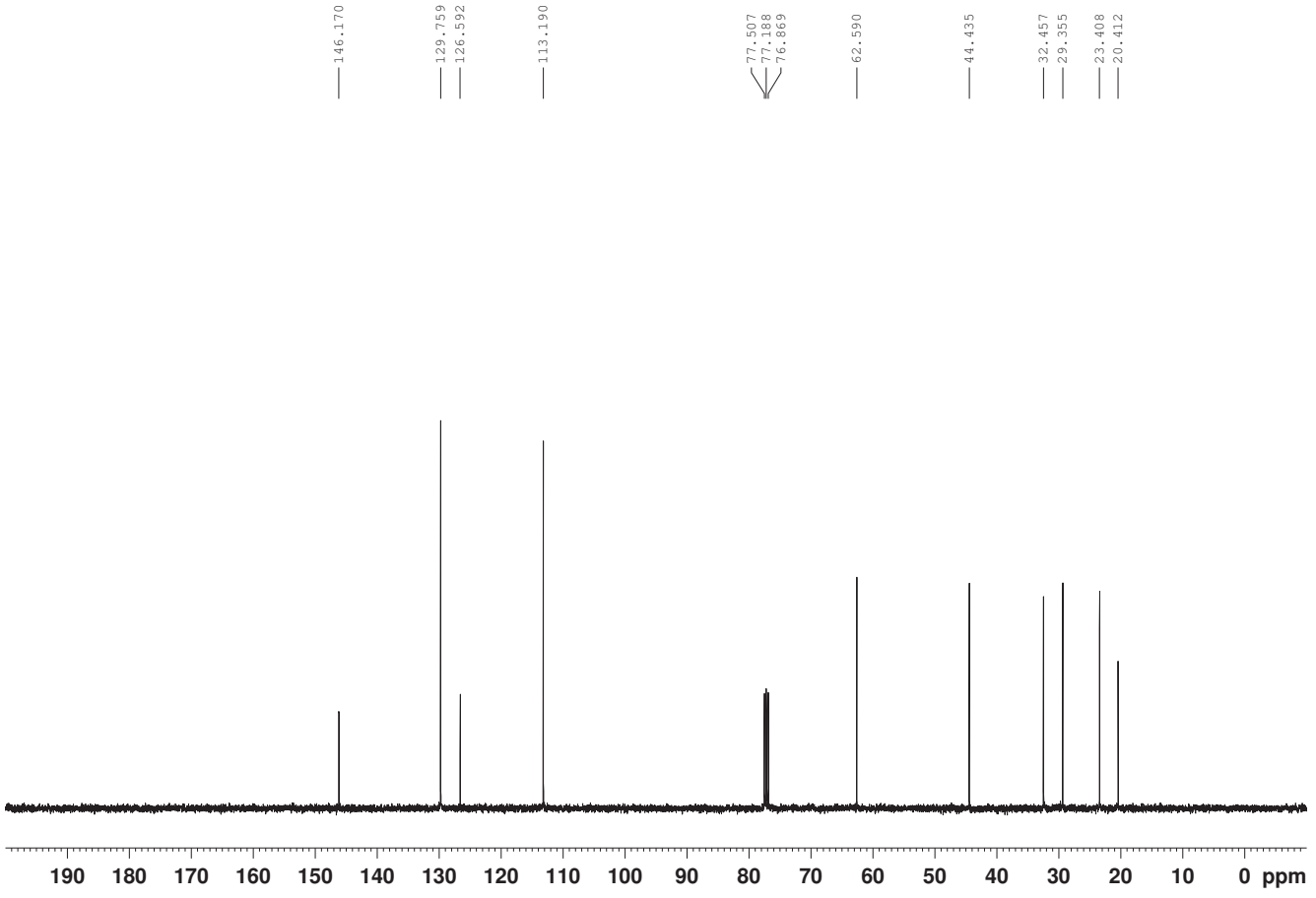
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62.590
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29.355
23.408
20.412
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150
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110
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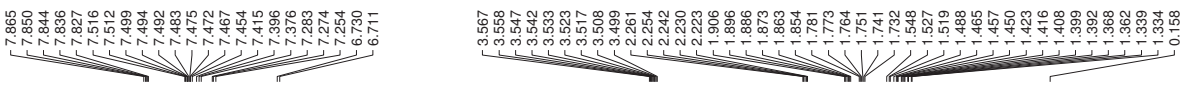
20
10
0 ppm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7.492
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7.475
7.472
7.467
7.454
7.415
7.396
7.376
7.283
7.274
7.254
6.730
6.711

3.567
3.558
3.547
3.542
3.533
3.523
3.517
3.508
3.499
2.261
2.254
2.242
2.230
2.223
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1.873
1.863
1.854
1.781
1.773
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1.751
1.741
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1.519
1.488
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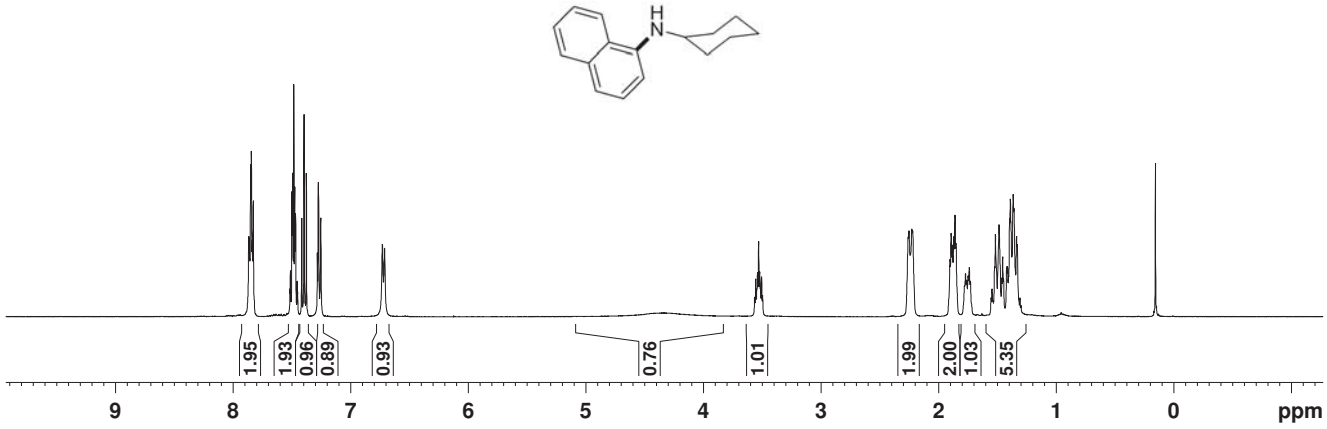
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4
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0
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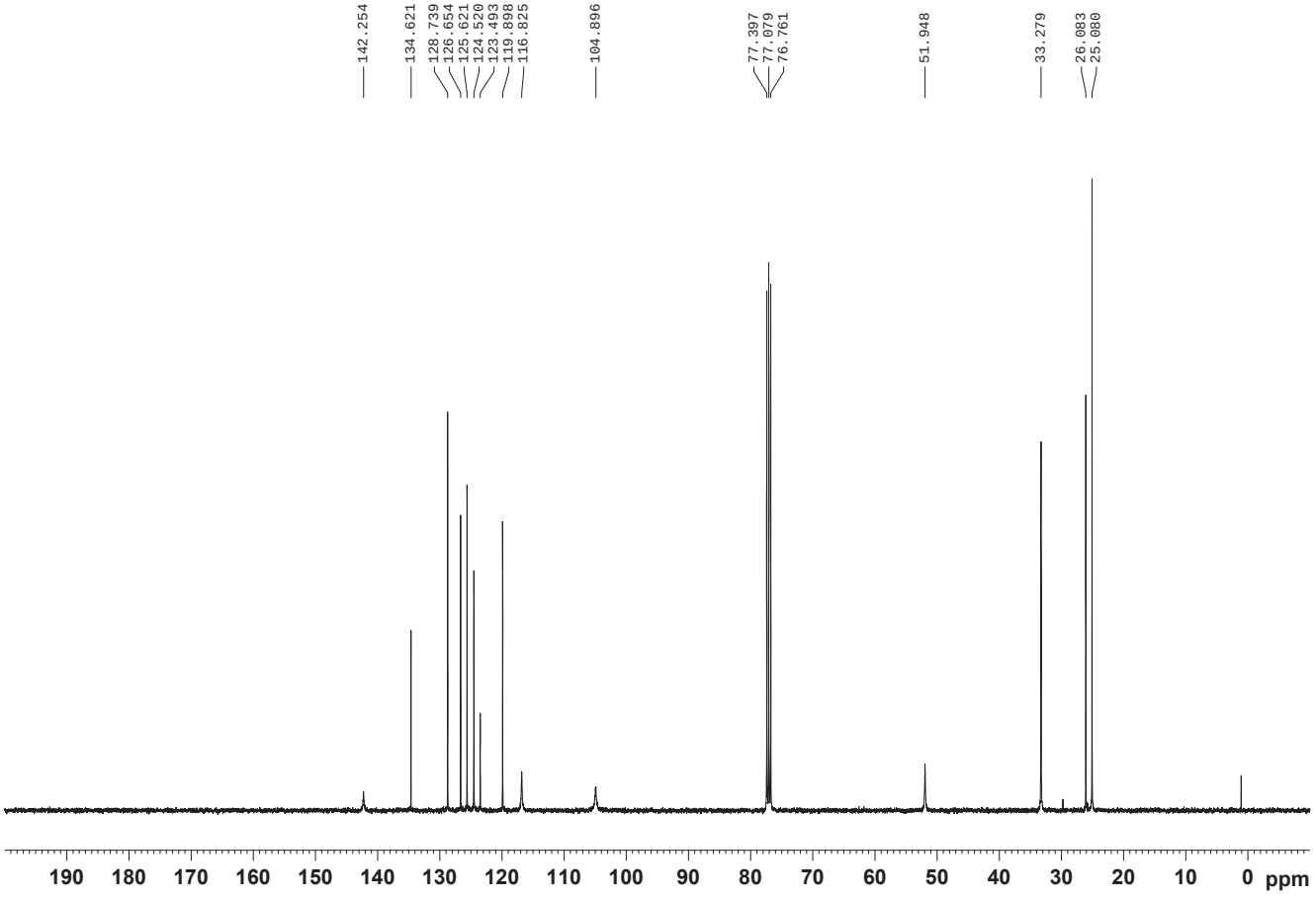
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123.493
119.898
116.825
104.896
77.397
77.079
76.761
51.948
33.279
26.083
25.080
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100
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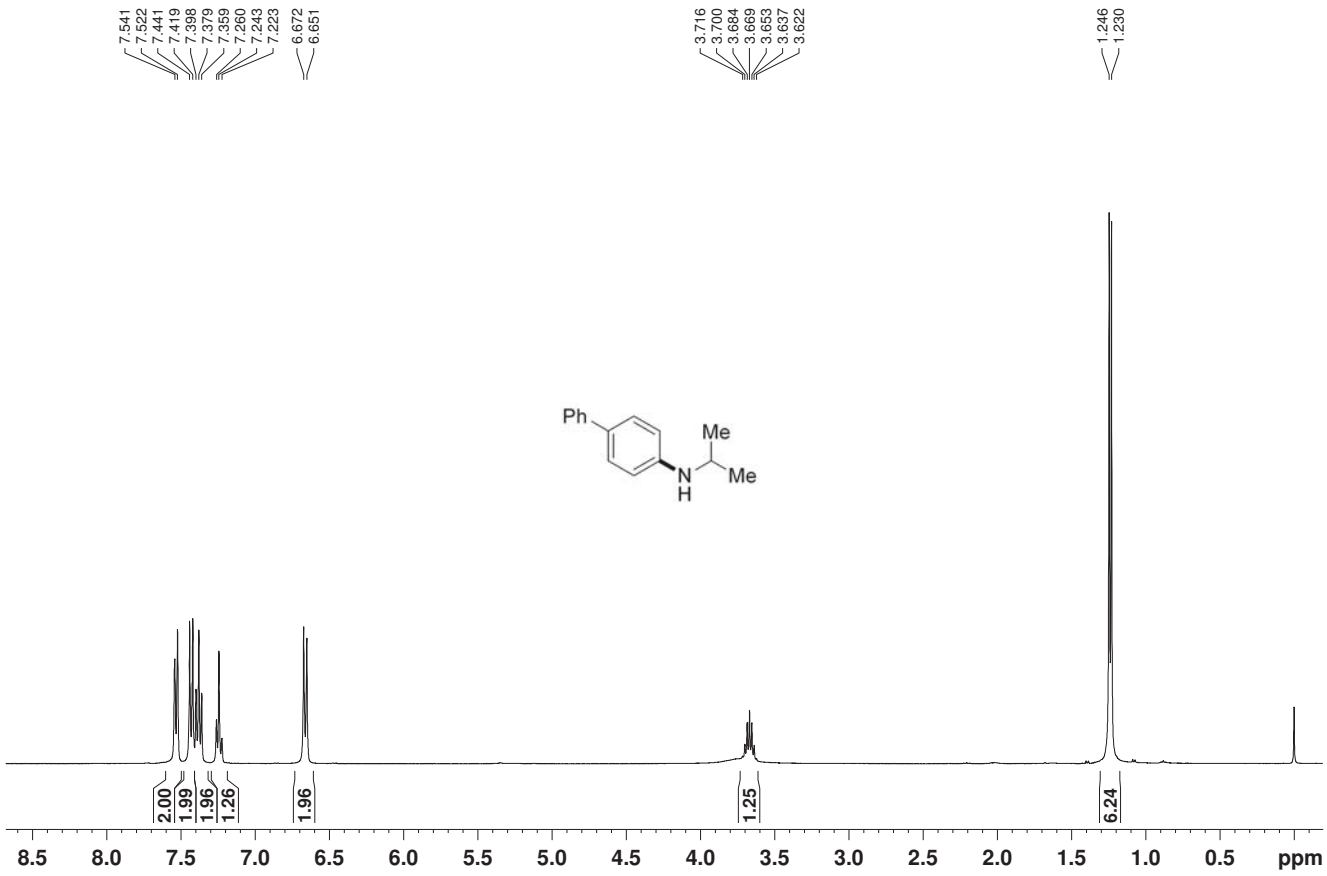
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6.651
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3.700
3.684
3.669
3.653
3.637
3.622
1.246
1.230
Ph
Me
N
H
Me
2.00
1.99
1.96
1.26
1.96
1.25
6.24
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
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2.0
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1.0
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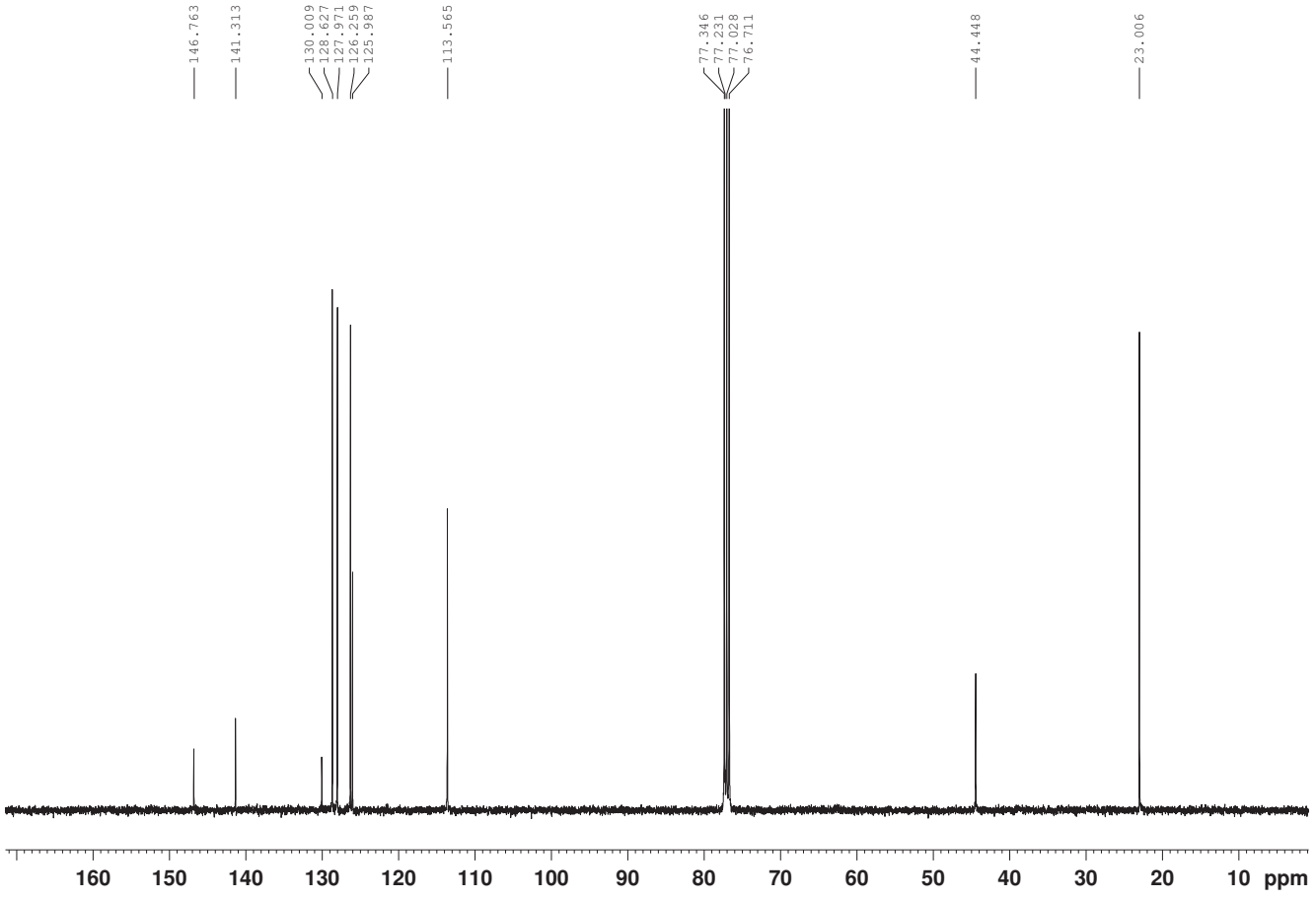
ppm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125.987
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77.231
77.028
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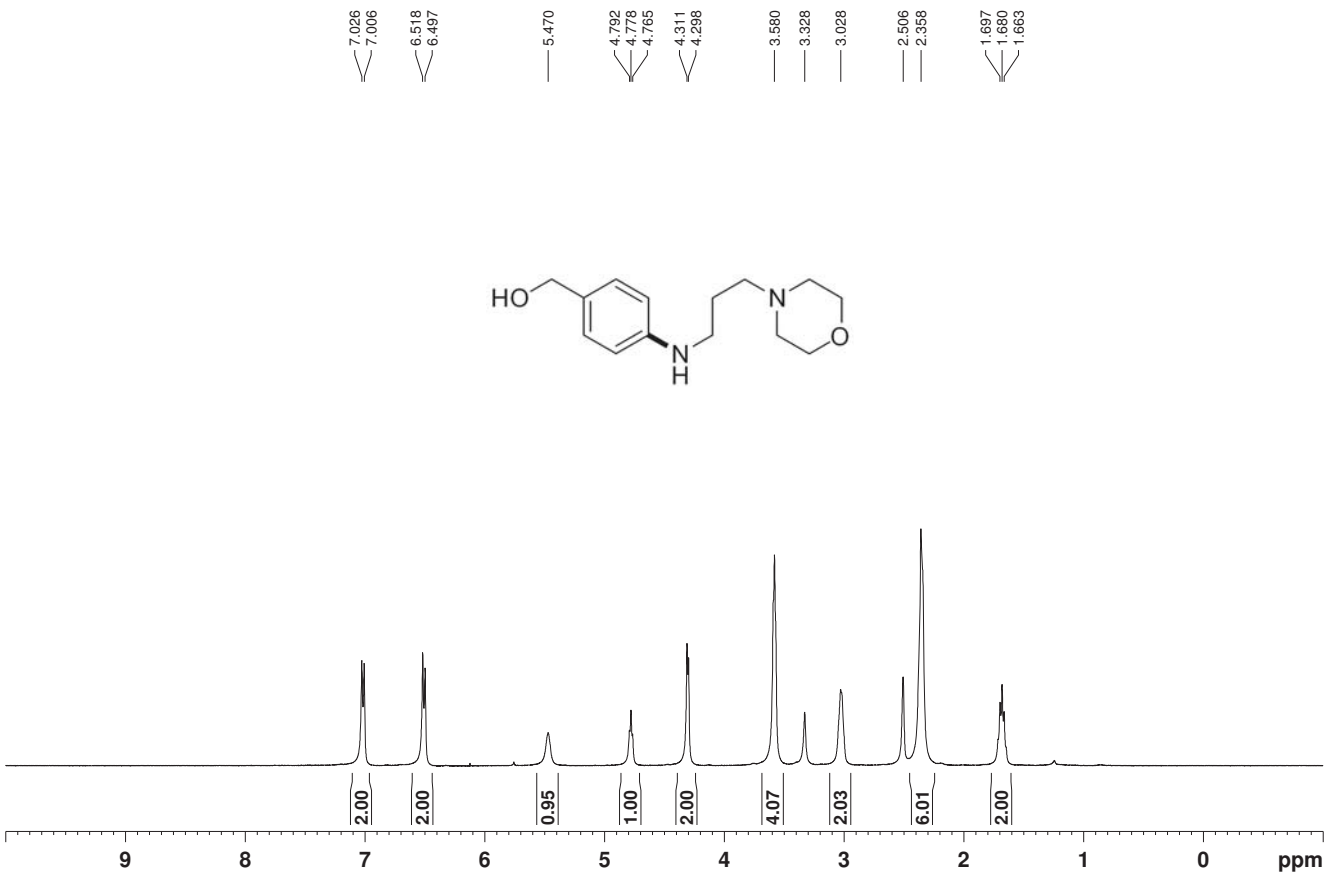
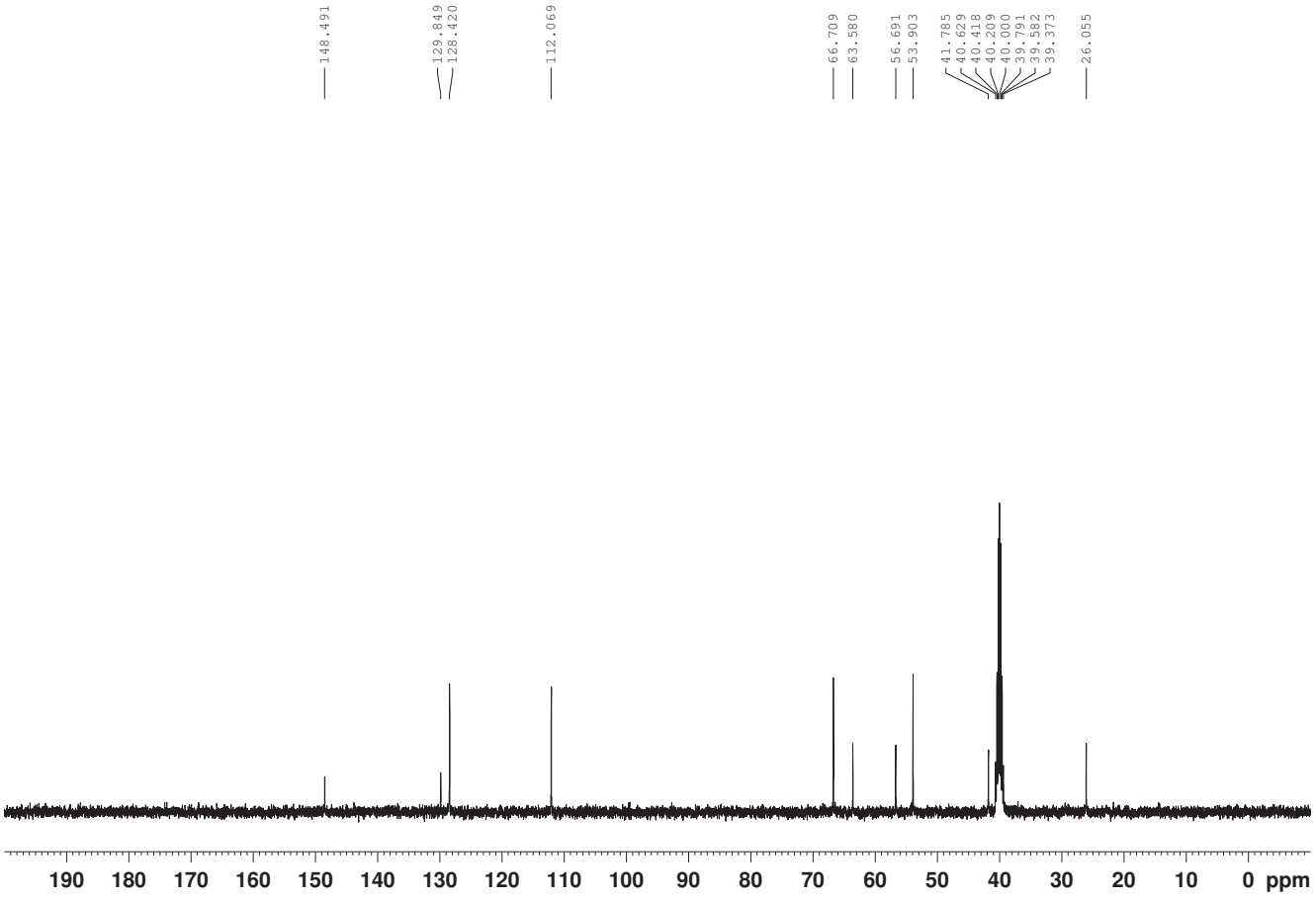
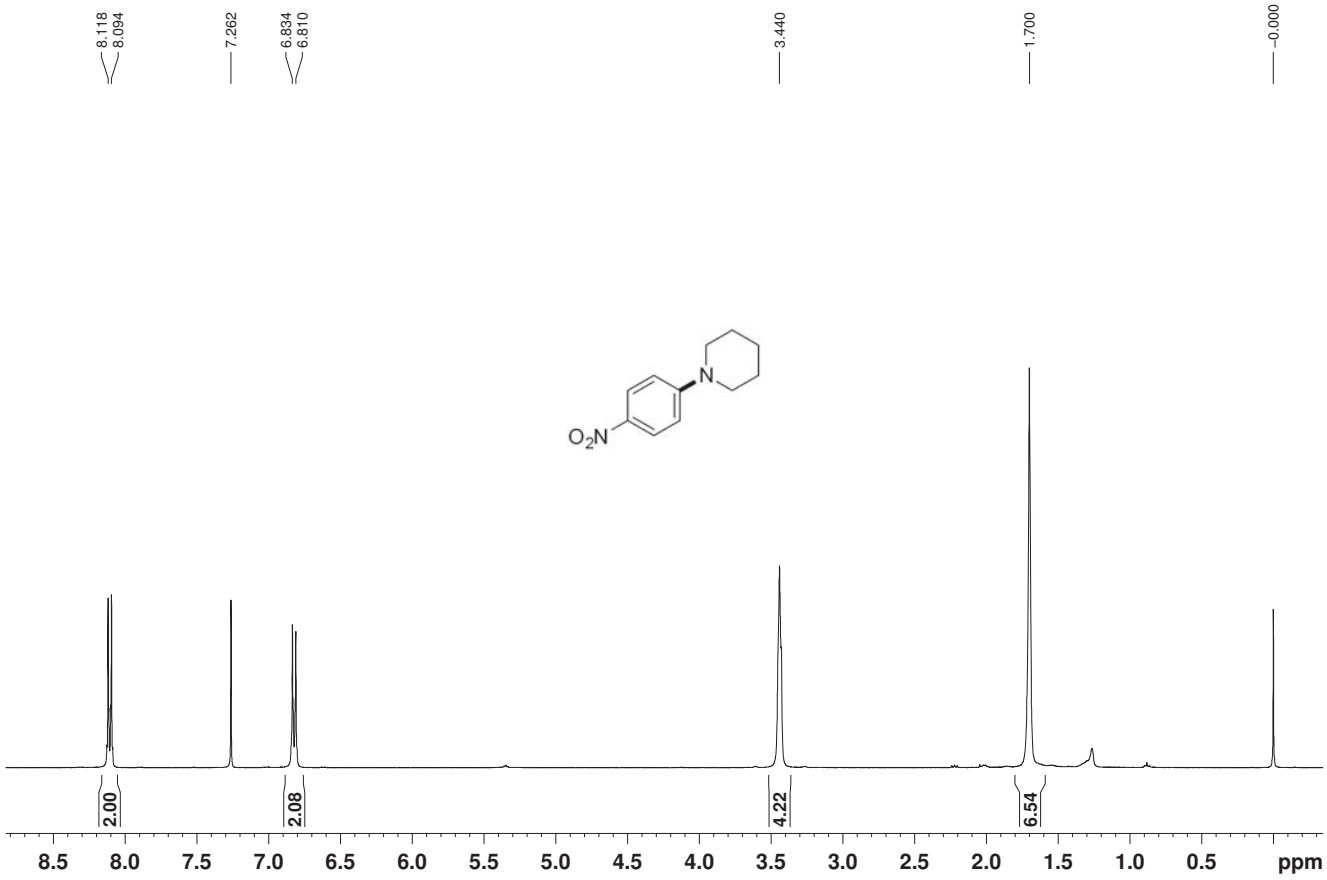
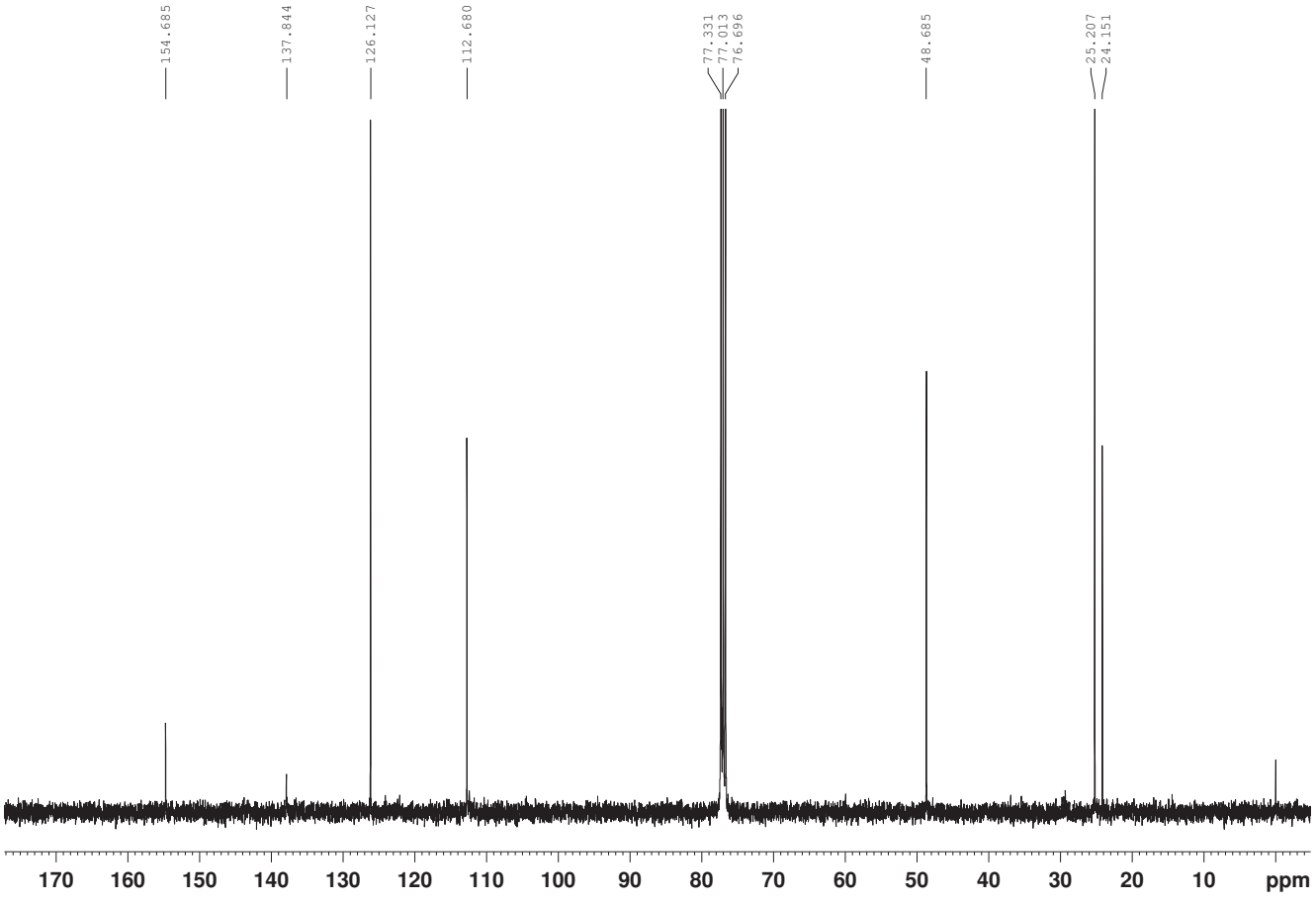
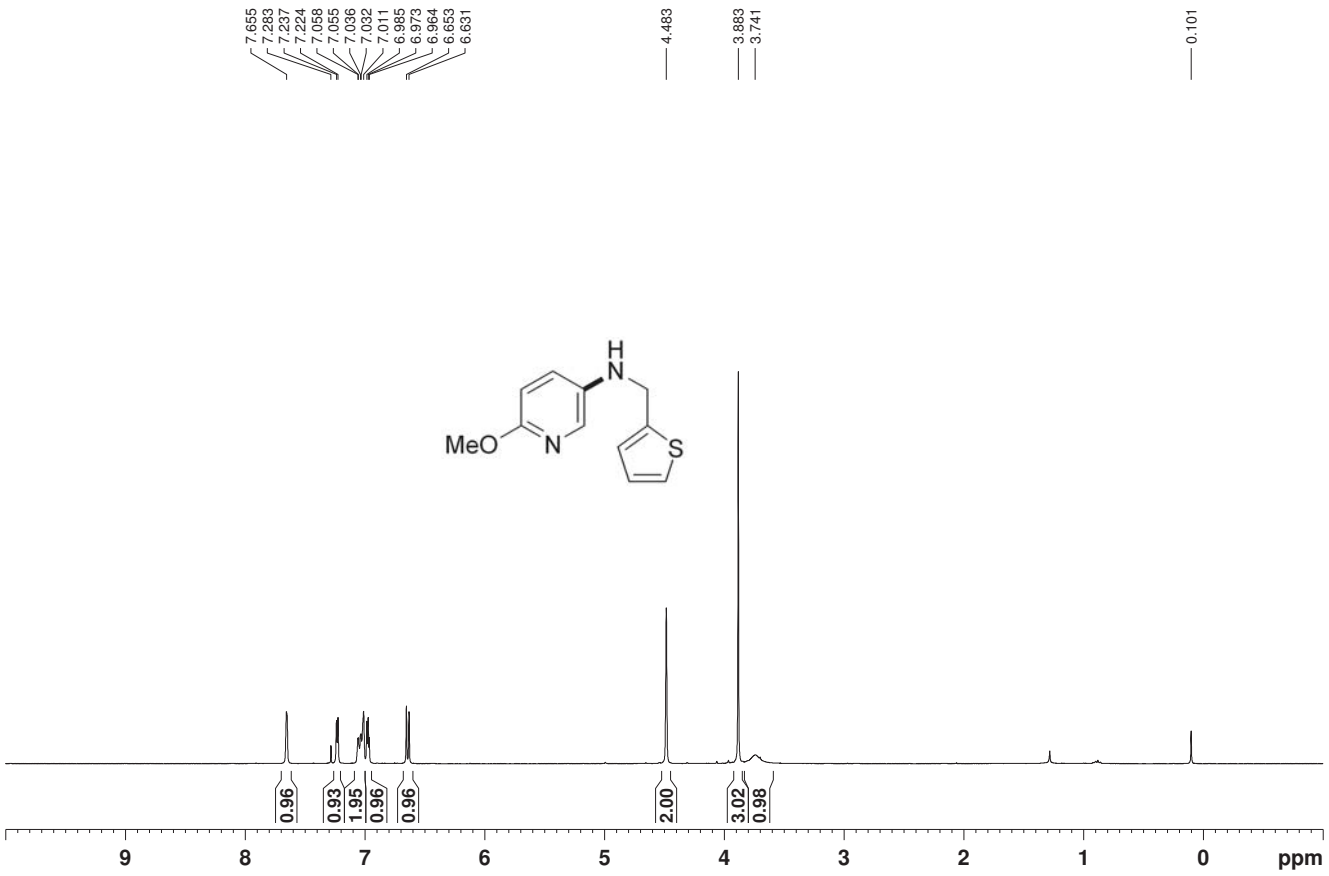
44.448
23.006
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

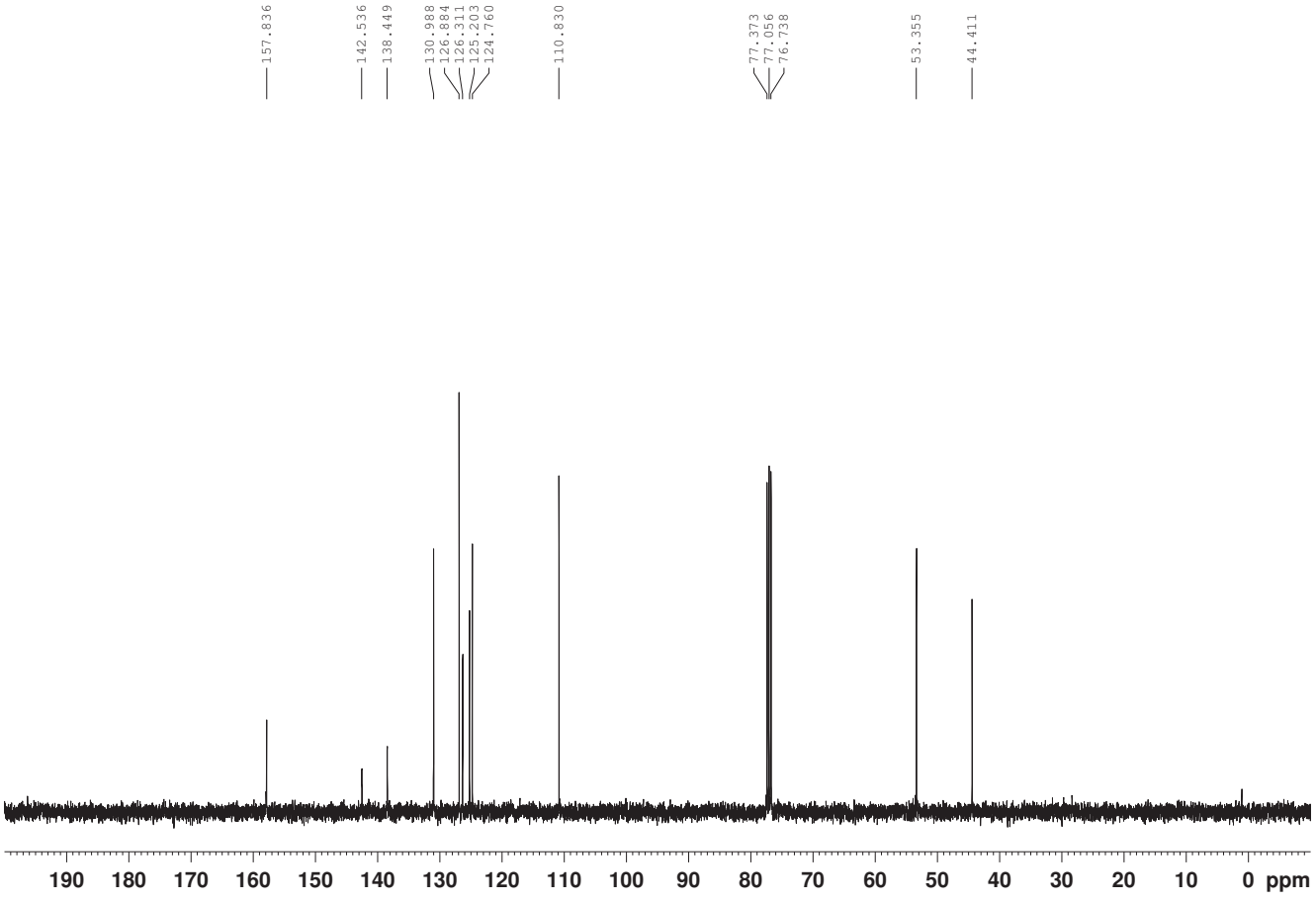

7.026
7.006
6.518
6.497
5.470
4.792
4.778
4.765
4.311
4.298
3.580
3.328
3.028
2.506
2.358
1.697
1.680
1.663
HO
N
O
N
H
2.00
2.00
0.95
1.00
2.00
4.07
2.03
6.01
2.00
9
8
7
6
5
4
3
2
1
0
ppm


148.491
129.849
128.420
112.069
66.709
63.580
56.691
53.903
41.785
40.629
40.418
40.209
40.000
39.791
39.582
39.373
26.055
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

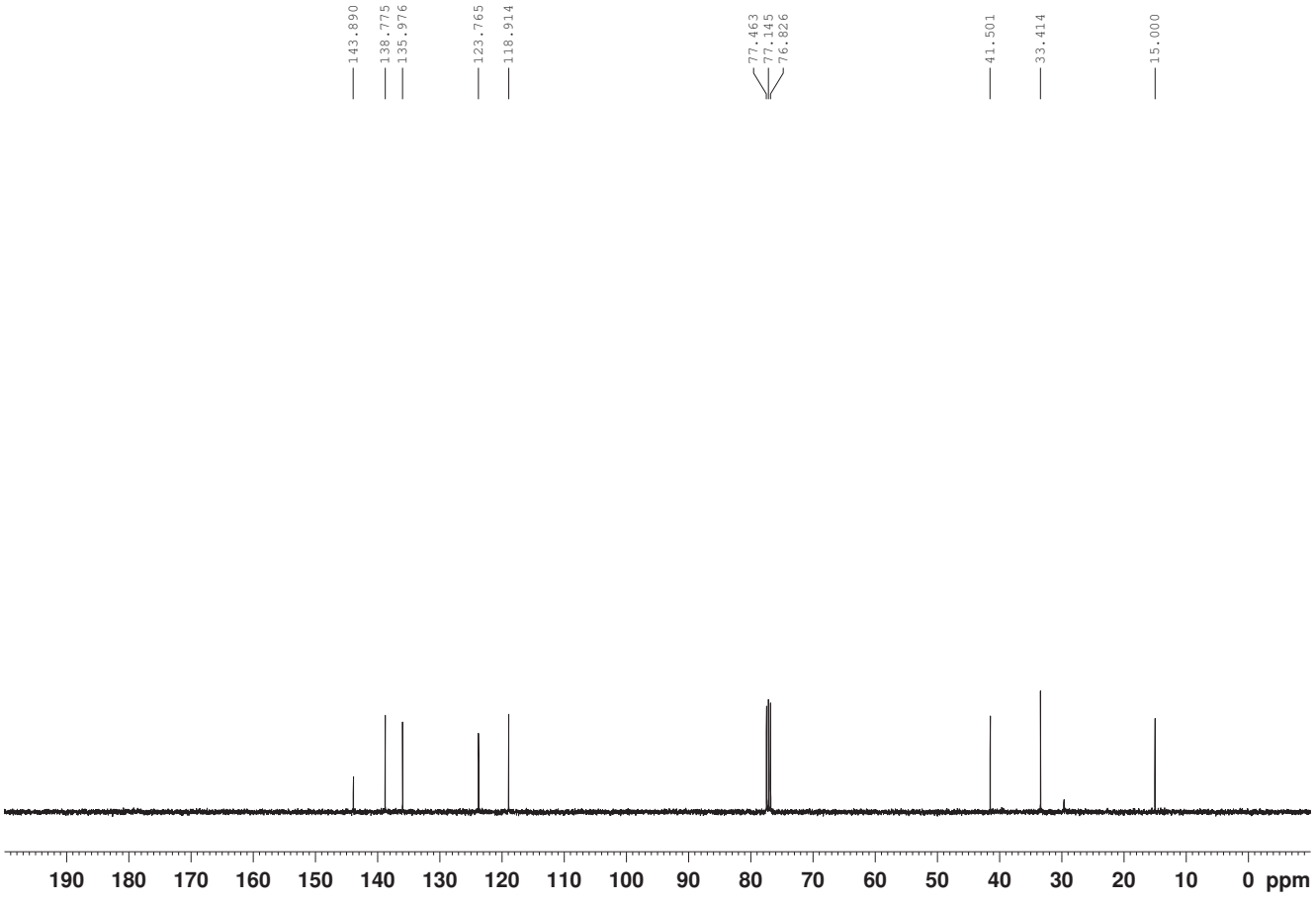

8.118
8.094
7.262
6.834
6.810
3.440
1.700
0.000
O_2N
N
2.00
2.08
4.22
6.54
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

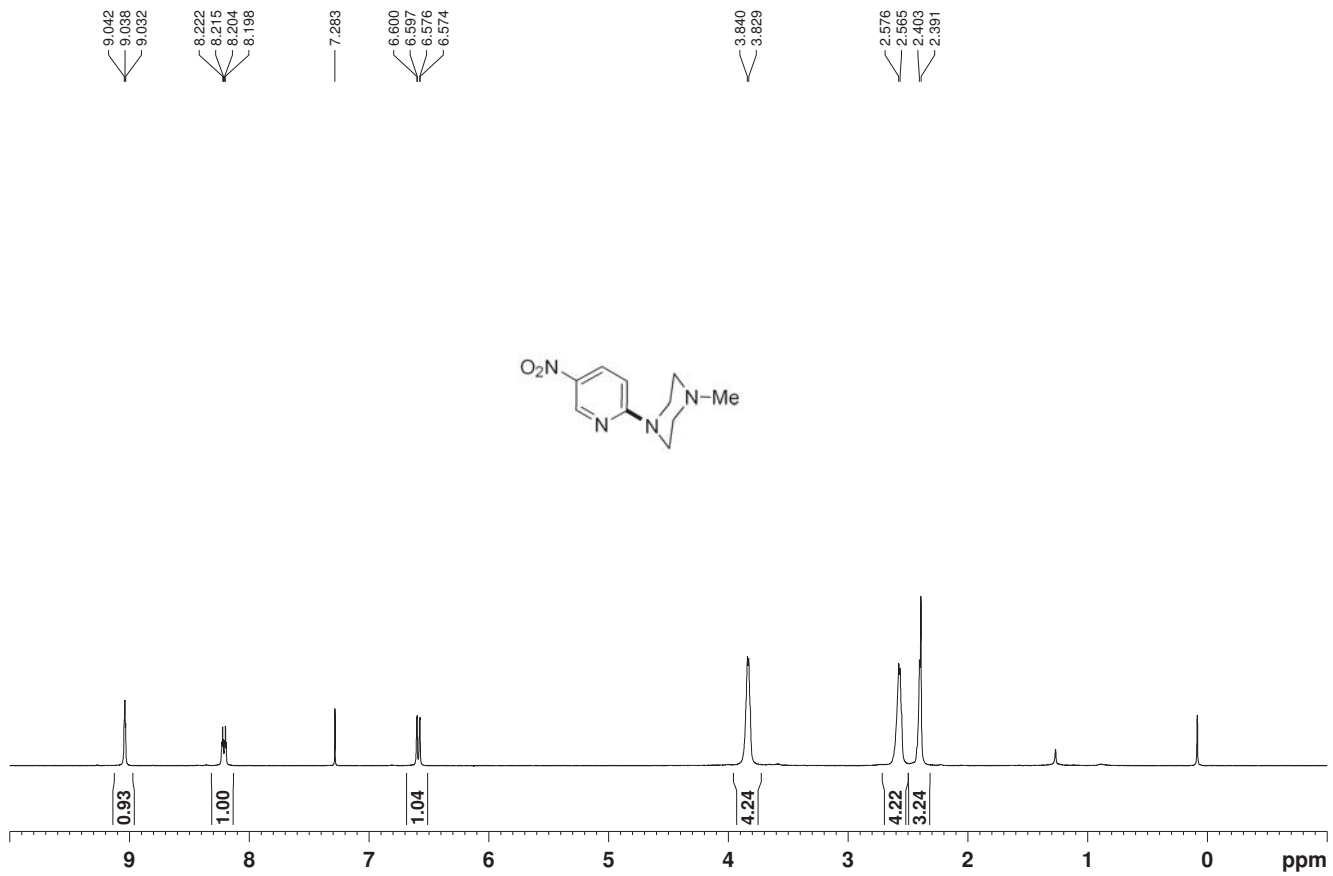

154.685
137.844
126.127
112.680
77.331
77.013
76.696
48.685
25.207
24.151
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

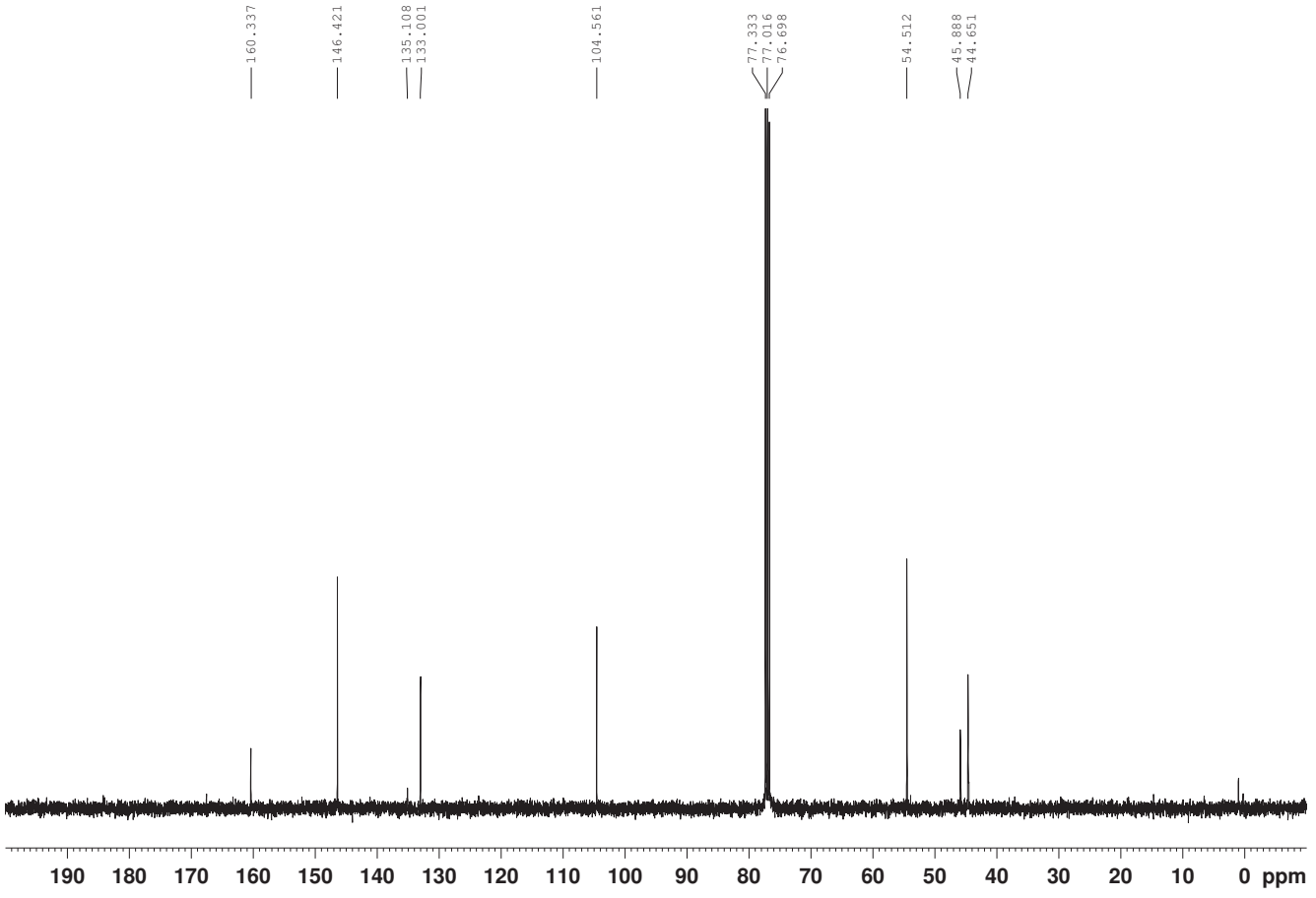

7.655
7.283
7.237
7.224
7.058
7.055
7.036
7.032
7.011
6.985
6.973
6.964
6.653
6.631
4.483
3.883
3.741
0.101
MeO
N
H
N
S
0.96
0.93
1.95
0.96
0.96
2.00
3.02
0.98
9
8
7
6
5
4
3
2
1
0
ppm


157.836
142.536
138.449
130.988
126.884
126.311
125.203
124.760
110.830
77.373
77.056
76.738
53.355
44.411
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


8.035
7.944
7.935
7.079
7.068
7.059
7.048
6.891
6.871
4.280
3.330
3.314
3.298
2.758
2.742
2.726
2.091
H
N
S
Me
N
0.97
1.02
1.00
1.02
1.08
2.09
2.09
3.10
9
8
7
6
5
4
3
2
1
0
ppm


143.890
138.775
135.976
123.765
118.914
77.463
77.145
76.826
41.501
33.414
15.000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


9.042
9.038
9.032
8.222
8.215
8.204
8.198
7.283
6.600
6.597
6.576
6.574
3.840
3.829
2.576
2.565
2.403
2.391
O2N
N
N
N–Me
0.93
1.00
1.04
4.24
4.22
3.24
9
8
7
6
5
4
3
2
1
0
ppm


160.337
146.421
135.108
133.001
104.561
77.333
77.016
76.698
54.512
45.888
44.651
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm