Supplementary Material (ESI) for Chemical Communications

Rhodium(I)-Catalyzed Hydroaminomethylation of 2-isopropenylanilines as a Novel Route to 1,2,3,4-Tetrahydroquinolines

Tiago O. Vieira and Howard Alper*

[†]*Contribution from the Centre for Catalysis Research and Innovation, Department of Chemistry, University of Ottawa, 10 Marie Curie, Ottawa, Ontario, Canada K1N 6N5*

e-mail: howard.alper@uottawa.ca

Supporting Information

General:

The 2-isopropenyl aniline was purchased from Aldrich and rhodium(COD) chloride dimer from Strem, and were used without prior purification. Catalyst **1** was prepared as previously reported,[1] and toluene was not distilled before use. The other substrates were prepared through a two step sequence (MeLi addition to the appropriate 2-amino-acetophenones or methyl anthranilic esters, followed by dehydration of the resulting tertiary alcohol), as described in the literature.[2] Flash column chromatography was undertaken with silica gel (60 Å, 200−425 mesh) supplied by Aldrich. IR spectra were obtained with a Shimadzu FTIR−8400S spectrometer. Mass spectra were determined using a VG 7070E spectrometer. Solution ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$, containing TMS as internal standard, on a Bruker Avance 300 MHz. Chemical shifts (δ) are reported in ppm with the solvent signals as reference (^{1}H NMR at 7.26 ppm and ^{13}C NMR at 77.1 ppm), and coupling constants (*J*) are given in Hertz (Hz). Full spectroscopic and spectrometric characterization is provided only for new compounds.

Carbon monoxide is a powerful asphyxiant and should be used with care. To use and work with carbon monoxide safely, reactions must be carried out in a proper working fumehood with carbon monoxide detectors installed nearby.

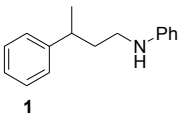
[1] J. J. Kim, H. Alper *Chem. Commun.* 2005, 3059.

[2] J. M. Bruce, P. Knowles *J. Chem. Soc.* 1964, 4046.

General Procedure for the Hydroaminomethylation:

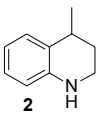
A glass liner, equipped with a magnetic stirring bar, containing the olefin (1.00 mmol), catalyst **1** (5.0 mol%, 25.6 mg) and toluene (2 mL) was placed, in a 45 mL autoclave. The autoclave was flushed three times with carbon monoxide and pressurized to 700 psi of CO and then 300 psi of H_2. The autoclave was then placed in an oil bath pre-set to 120 ^oC on a stirring hot plate. The total pressure increased to 1200 psi. After 48h, the autoclave was removed from the oil bath and cooled to room temperature prior to the release of excess carbon monoxide. The solvent was concentrated and the residue was purified by silica gel chromatography with a mixture of hexane and ethyl acetate (9:1) as the eluant to afford the desired products.

Compounds Prepared and Characterization:



1

Phenyl-(3-phenyl-butyl)-amine:[3] colorless oil; 80%; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.28 (d, *J*= 7.0 Hz, 3H), 1.88 (q, *J*= 7.2 Hz, 2H), 2.83 (sextet, *J*= 7.1 Hz, 1H), 2.92-3.08 (m, 2H), 3.47 (bs, 1H), 6.47-6.68 (m, 3H), 7.10-7.32 (m, 7H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.6, 37.9, 38.0, 42.3, 112.7 (2C), 117.1, 126.3, 127.0 (2C), 128.6 (2C), 129.2 (2C), 146.7, 148.4.



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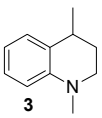
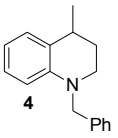
4-Methyl-1,2,3,4-tetrahydro-quinoline:[4] colorless oil; 89%; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.28 (d, *J*= 6.9 Hz, 3H), 1.62-1.72 (m, 1H), 1.92-2.02 (m, 1H), 2.85-2.96 (m, 1H), 3.21-3.35 (m, 2H), 3.82 (bs, 1H), 6.45-6.65 (m, 2H), 6.93-7.06 (m, 2H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.7, 29.9, 30.3, 39.0, 114.2, 117.0, 126.6, 126.8, 128.5, 144.3.



3

1,4-Dimethyl-1,2,3,4-tetrahydro-quinoline:[5] colorless oil; 92%; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.27 (d, *J*= 7.0 Hz, 3H), 1.63-1.73 (m, 1H), 1.97-2.08 (m, 1H), 2.88 (s, 3H + m, 1H), 3.21-3.27 (m, 2H), 6.58-6.66 (m, 2H), 7.03-7.10 (m, 2H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.8, 30.0, 30.8, 39.2, 48.3, 110.0, 116.2, 127.1, 127.8, 128.0, 146.2.



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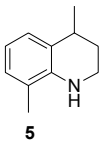
1-Benzyl-4-methyl-1,2,3,4-tetrahydro-quinoline: colorless oil; 98%; ν_{max}/cm^{-1} (neat) 1600, 1500, 1450, 738, 727, 694; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.31 (d, *J*= 7.2 Hz, 3H), 1.69-1.79 (m, 1H), 2.01-2.11 (m, 1H), 2.90-3.01 (m, 1H), 3.28-3.46 (m, 2H), 4.49 (s, 2H), 6.49-6.63 (m, 2H), 6.94-7.33 (m,

[3] T. Rische, P. Eilbracht *Synthesis* 1997, 1331.

[4] M. R. Pitts, J. R. Harrison, C. J. Moody *J. Chem. Soc., Perkin Trans. 1*, 2001, 955.

[5] A. R. Katritzky, B. Rachwal, S. Rachwal *J. Org. Chem.* 1995, **60**, 2588.

7H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.4, 29.7, 31.1, 46.7, 55.2, 111.0, 115.9, 126.6 (2C), 126.8, 127.2, 127.3, 128.0, 128.7 (2C), 138.9, 145.0; HRMS (EI) Calcd. for $C_{17}H_{19}N$ 237.1517, found 237.1528.



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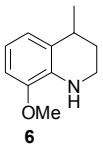
4,8-Dimethyl-1,2,3,4-tetrahydro-quinoline: yellowish oil; 85%; ν_{max}/cm^{-1} (neat) 3427, 1599, 1494, 1481, 1467, 1307, 767, 738; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.29 (d, *J*= 7.0 Hz, 3H), 1.64-1.73 (m, 1H), 1.92-2.03 (m, 1H), 2.07 (s, 3H), 2.88-2.99 (m, 1H), 3.30-3.43 (m, 2H), 3.69 (bs, 1H), 6.58 (t, *J*= 7.4 Hz, 1H), 6.88 (d, *J*= 7.3 Hz, 1H), 6.96 (d, *J*= 7.6 Hz, 1H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 17.4, 23.0, 29.7, 30.5, 39.1, 116.4, 121.1, 126.1, 126.5, 127.9, 142.2; HRMS (EI) Calcd. for $C_{11}H_{15}N$ 161.1204, found 161.1216.



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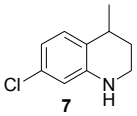
8-Methoxy-4-methyl-1,2,3,4-tetrahydro-quinoline: yellowish oil; 80%; ν_{max}/cm^{-1} (neat) 3427, 1500, 1332, 1251, 1242, 727; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.28 (d, *J*= 7.0 Hz, 3H), 1.62-1.73 (m, 1H), 1.93-2.04 (m, 1H), 2.88-2.98 (m, 1H), 3.25-3.39 (m, 2H), 3.81 (s, 3H), 4.30 (bs, 1H), 6.56-6.74 (m, 3H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.9, 29.8, 30.1, 38.5, 55.4, 107.2, 115.6, 120.7, 126.6, 134.1, 146.1; HRMS (EI) Calcd. for $C_{11}H_{15}NO$ 177.1154, found 177.1159.



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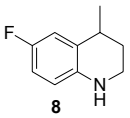
7-Chloro-4-methyl-1,2,3,4-tetrahydro-quinoline: yellowish oil; 73%; ν_{max}/cm^{-1} (neat) 3415, 1602, 1492, 1307, 1085, 1066, 870, 837, 788; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.24 (d, *J*= 7.0 Hz, 3H), 1.59-1.69 (m, 1H), 1.88-1.97 (m, 1H), 2.79-2.90 (m, 1H), 3.22-3.33 (m, 2H), 3.90 (bs, 1H), 6.42 (d, *J*= 2.1 Hz, 1H), 6.55 (dd, *J*= 8.1 and 2.1 Hz, 1H), 6.93 (d, *J*= 8.1 Hz, 1H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.4, 29.4, 29.9, 38.8, 113.4, 116.6, 124.9, 129.5, 132.0, 145.3; HRMS (EI) Calcd. for $C_{10}H_{12}ClN$ 181.0658, found 181.0653.



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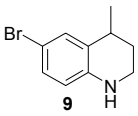
6-Fluoro-4-methyl-1,2,3,4-tetrahydro-quinoline: yellowish oil; 61%; ν_{max}/cm^{-1} (neat) 3414, 1500, 1244, 1147, 804; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.27 (d, *J*= 7.0 Hz, 3H), 1.60-1.72 (m, 1H), 1.91-2.02 (m, 1H), 2.83-2.93 (m, 1H), 3.19-3.33 (m, 2H), 3.72 (bs, 1H), 6.39 (dd, *J*= 8.7 and 4.9 Hz, 1H), 6.67 (ddd, *J*= 17.0, 8.6 and 2.9 Hz, 1H), 6.78 (dd, *J*= 9.7 and 2.6 Hz, 1H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.6, 29.9, 30.5, 39.4, 113.3 (*J*= 22.6 Hz), 114.6 (*J*= 21.1 Hz), 114.9 (*J*= 7.3 Hz), 128.1 (*J*= 6.1 Hz), 140.5 (*J*= 1.7 Hz), 155.7 (*J*= 233.9 Hz); HRMS (EI) Calcd. for $C_{10}H_{12}FN$ 165.0954, found 165.0958.



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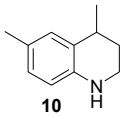
6-Bromo-4-methyl-1,2,3,4-tetrahydro-quinoline: yellowish amorphous solid; 72%; ν_{max}/cm^{-1} (CH_2Cl_2) 3421, 1598, 1494, 1300, 808; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.25 (d, *J*= 7.0 Hz, 3H), 1.59-1.69 (m, 1H), 1.88-1.98 (m, 1H), 2.80-2.91 (m, 1H), 3.20-3.34 (m, 2H), 3.81 (bs, 1H), 6.33 (d, *J*= 8.5 Hz, 1H), 7.02 (dd, *J*= 8.5 and 2.3 Hz, 1H), 7.12 (d, *J*= 2.3 Hz, 1H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.4, 29.4, 30.3, 38.9, 108.3, 115.6, 128.6, 129.4, 130.9, 143.2; HRMS (EI) Calcd. for $C_{10}H_{12}BrN$ 225.0153, found 225.0139.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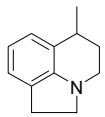
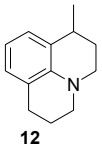
4,6-Dimethyl-1,2,3,4-tetrahydro-quinoline: yellowish oil; 80%; ν_{max}/cm^{-1} (neat) 3396, 1618, 1508, 1303, 806; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.28 (d, *J*= 7.0 Hz, 3H), 1.61-1.71 (m, 1H), 1.92-2.21 (m, 1H), 2.82-2.93 (m, 1H), 3.18-3.33 (m, 2H), 3.71 (bs, 1H), 6.40 (d, *J*= 8.3 Hz, 1H), 6.76-6.87 (m, 2H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 20.6, 22.9, 30.1, 30.2, 39.2, 114.5, 126.2, 126.8, 127.4, 129.1, 141.9; HRMS (EI) Calcd. for $C_{11}H_{15}rN$ 161.1204, found 161.1224.


11

6-Methyl-1,2,5,6-tetrahydro-4H-pyrrolo[3,2,1-*ij*]quinoline:[6] yellowish oil; 70%; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.27 (d, *J*= 7.0 Hz, 3H), 1.73-1.84 (m, 1H), 2.09-2.19 (m, 1H), 2.85-3.31 (m, 7H), 6.64 (t, *J*= 7.4 Hz, 1H), 6.90-6.95 (m, 2H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.3, 28.7, 29.0, 31.8, 45.8, 55.1, 118.5, 122.0, 124.9, 125.1, 128.8, 149.5.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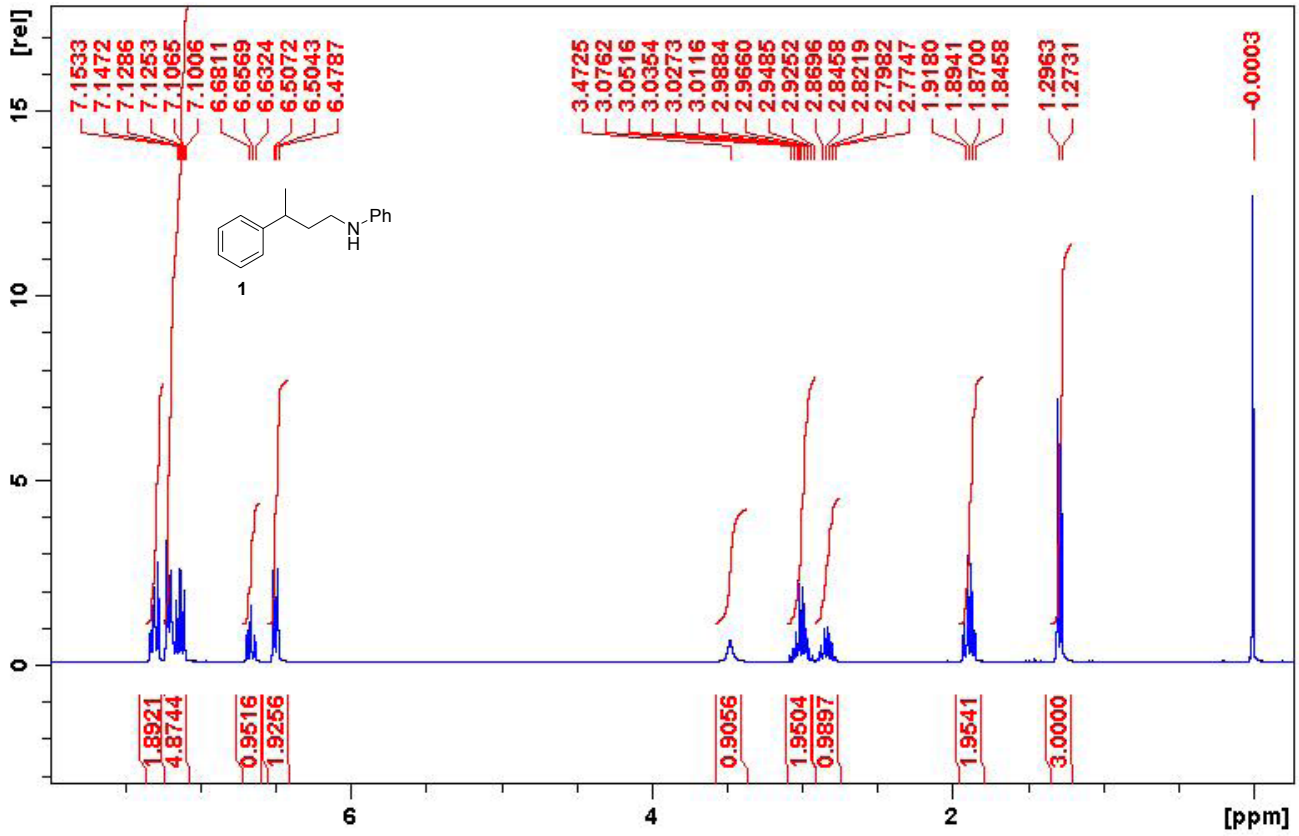
1-Methyl-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-*ij*]quinoline: colorless oil, 73%, ν_{max}/cm^{-1} (neat) 1595, 1488, 1456, 1444, 1301, 736; δ_H (300 MHz; $CDCl_3$; Me_4Si) 1.26 (d, *J*= 7.0 Hz, 3H), 1.62-1.72 (m, 1H), 1.92-2.07 (m, 3H), 2.73-2.90 (m, 3H), 3.02-3.19 (m, 4H), 6.52 (t, *J*= 7.4 Hz, 1H), 6.77-6.89 (m, 2H); δ_C (75.4 MHz; $CDCl_3$; Me_4Si) 22.1, 23.2, 27.8, 29.7, 30.8, 47.1, 50.2, 115.7, 121.5, 126.1, 126.8, 127.0, 142.4; HRMS (EI) Calcd. for $C_{13}H_{17}N$ 187.1361, found 187.1353.

[6] D. E. Burke, J. M. Cook, P. W. Le Quesne, *J. Am. Chem. Soc.* 1973, **95**, 546.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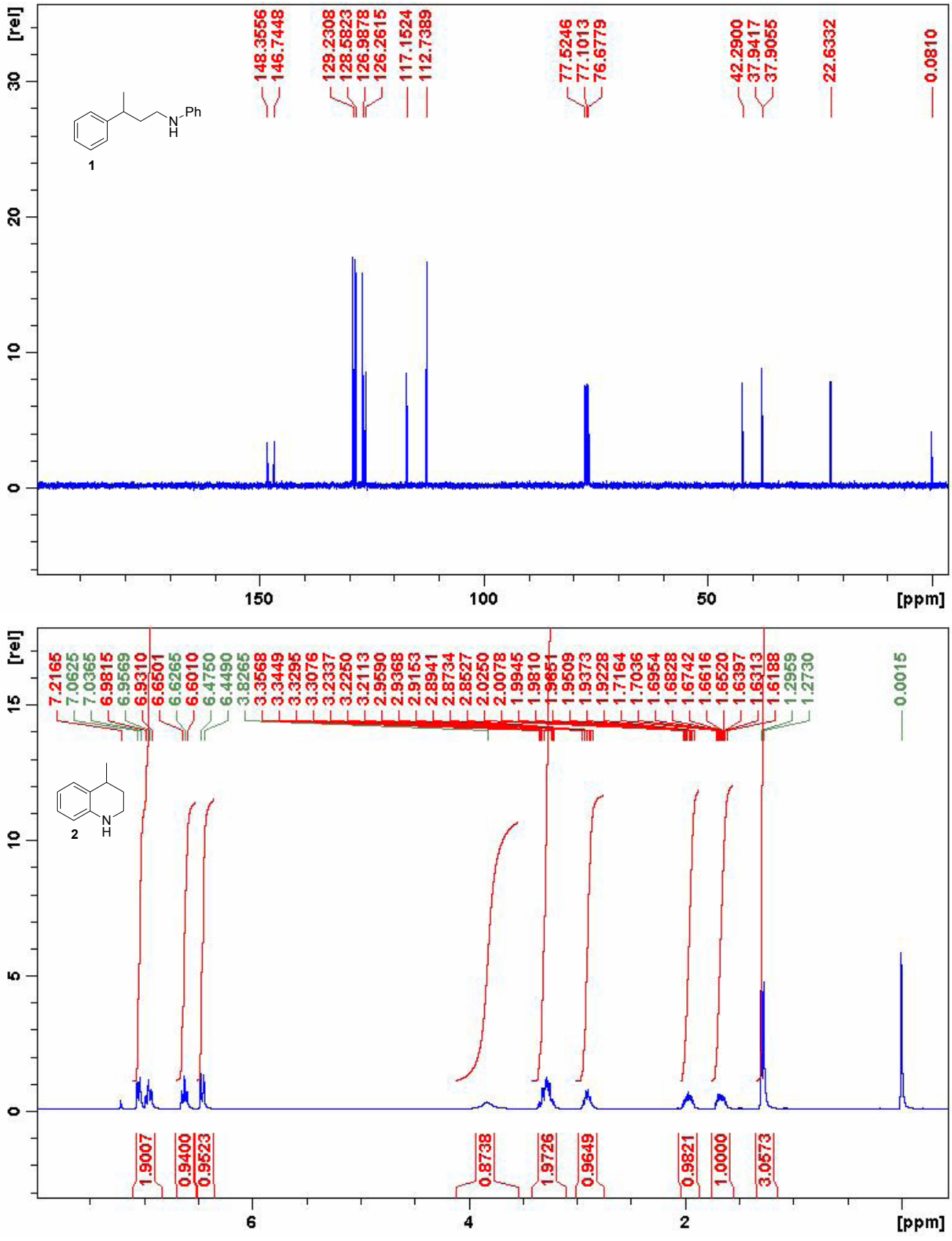
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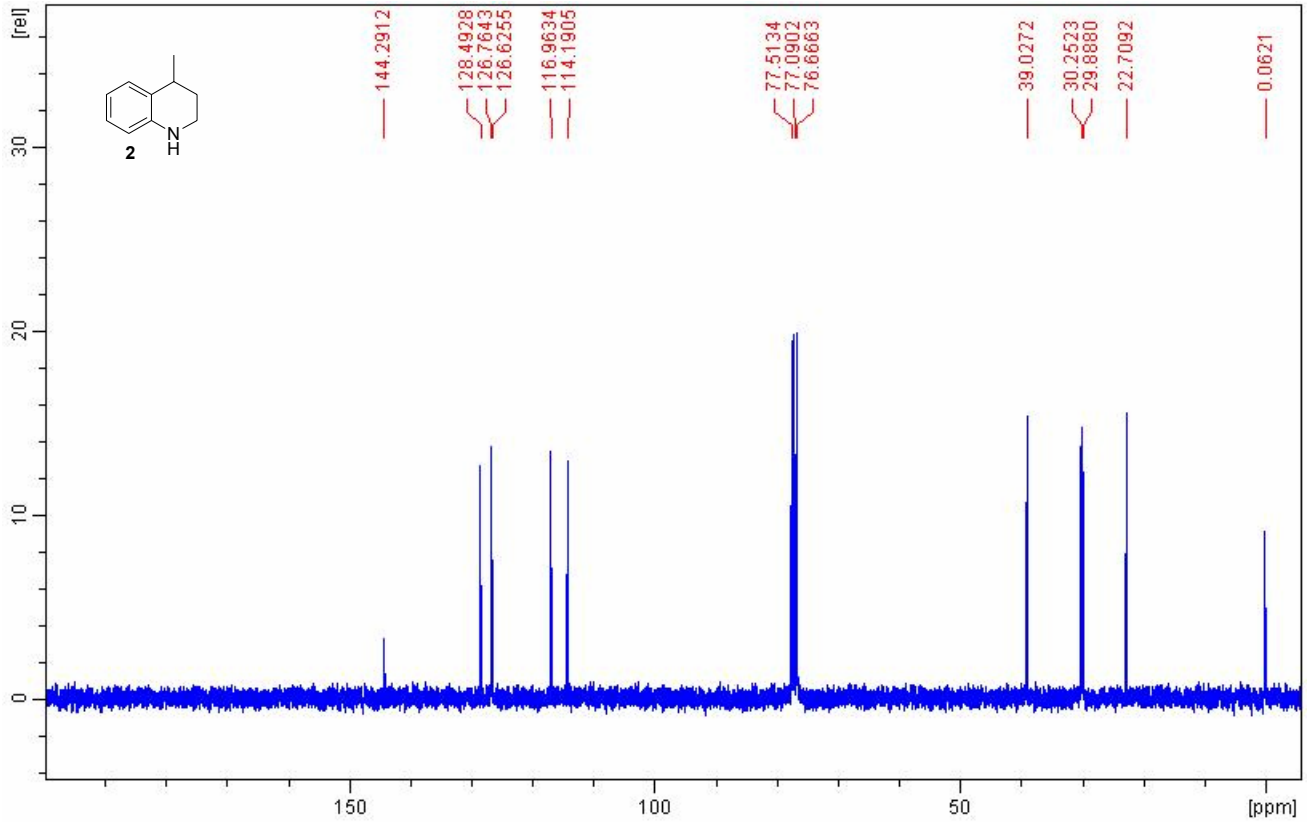
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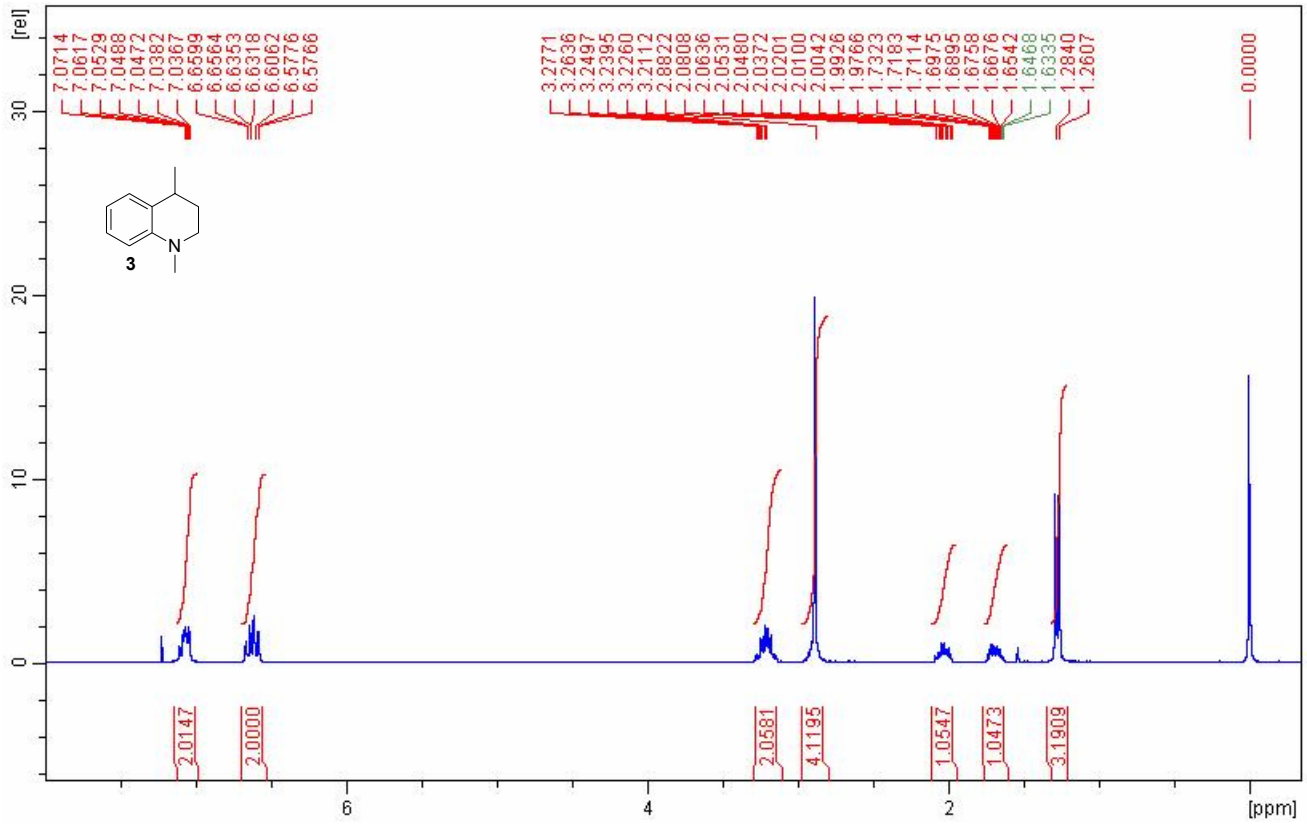
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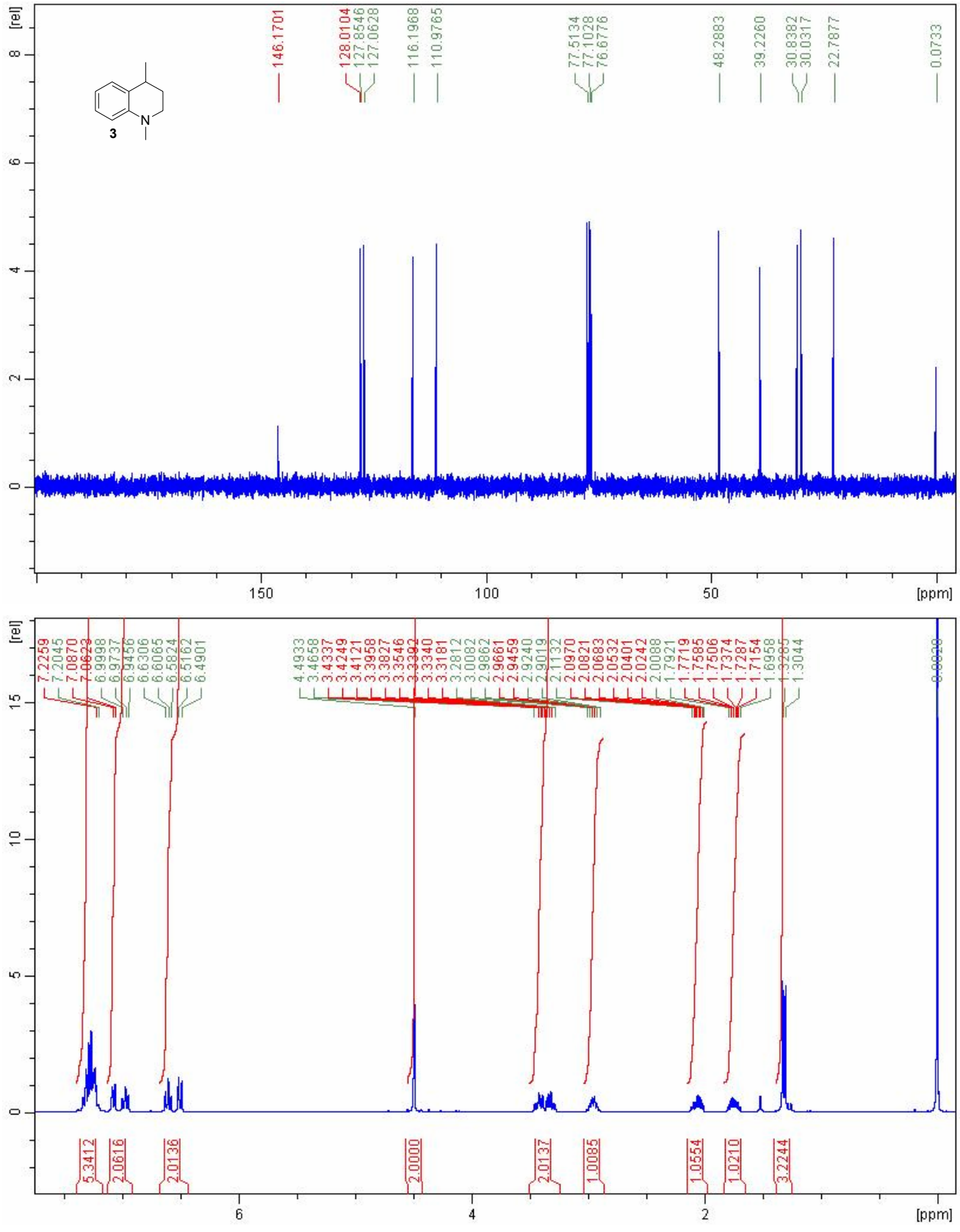
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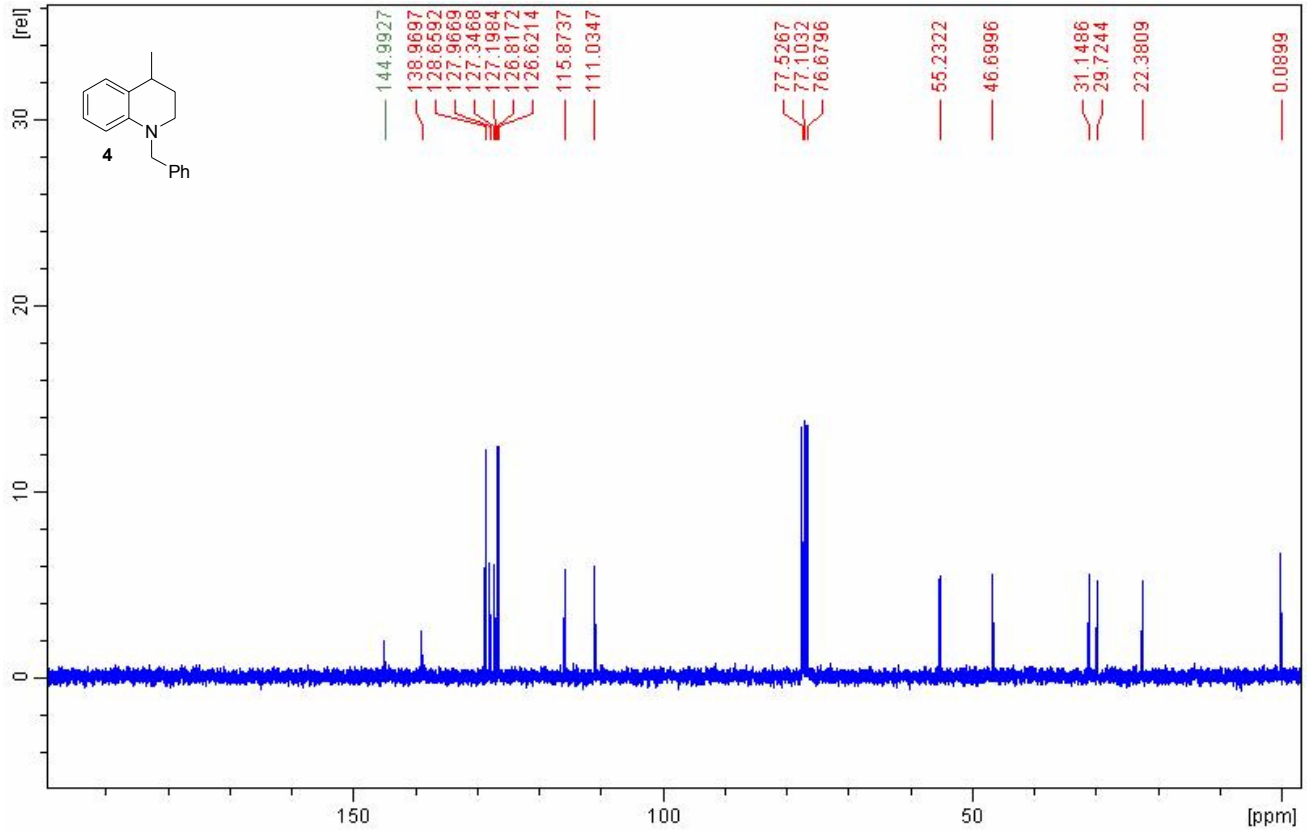
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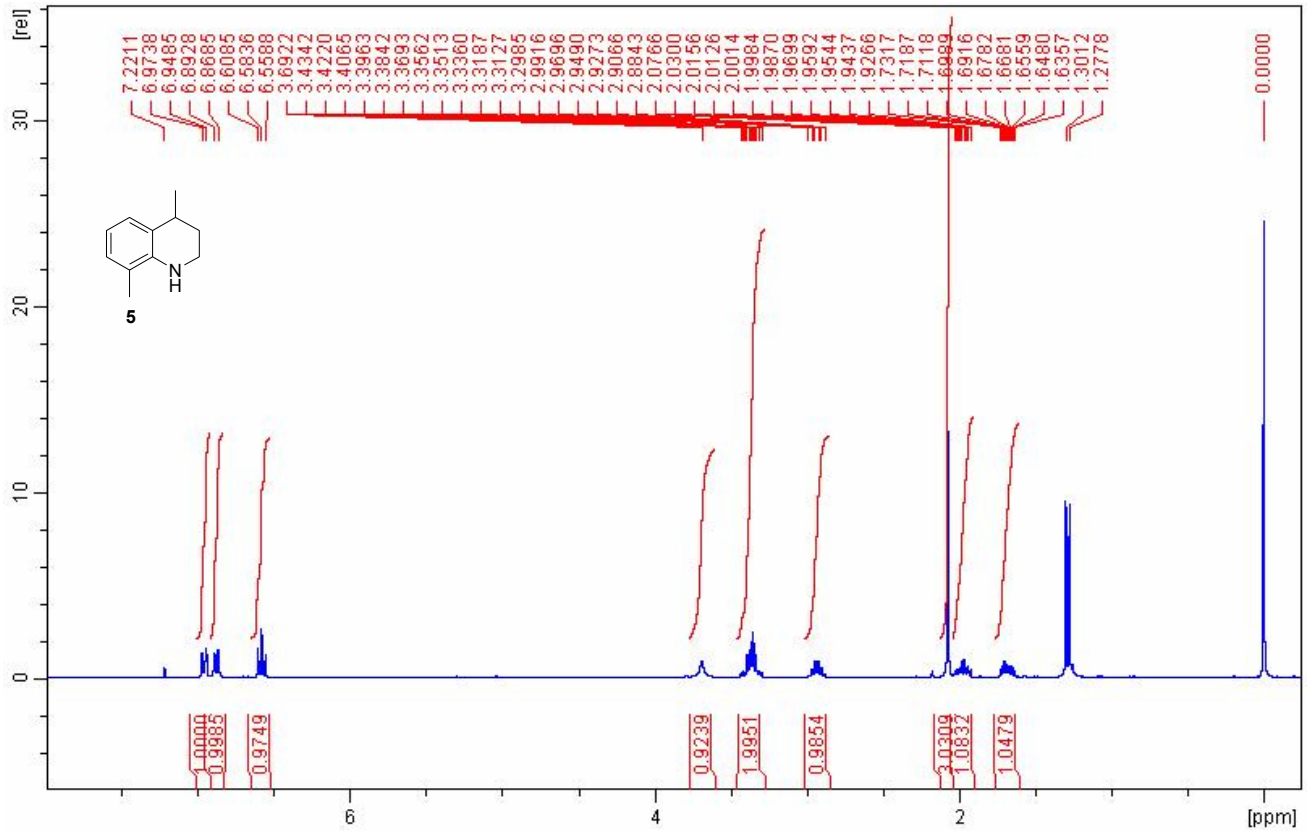
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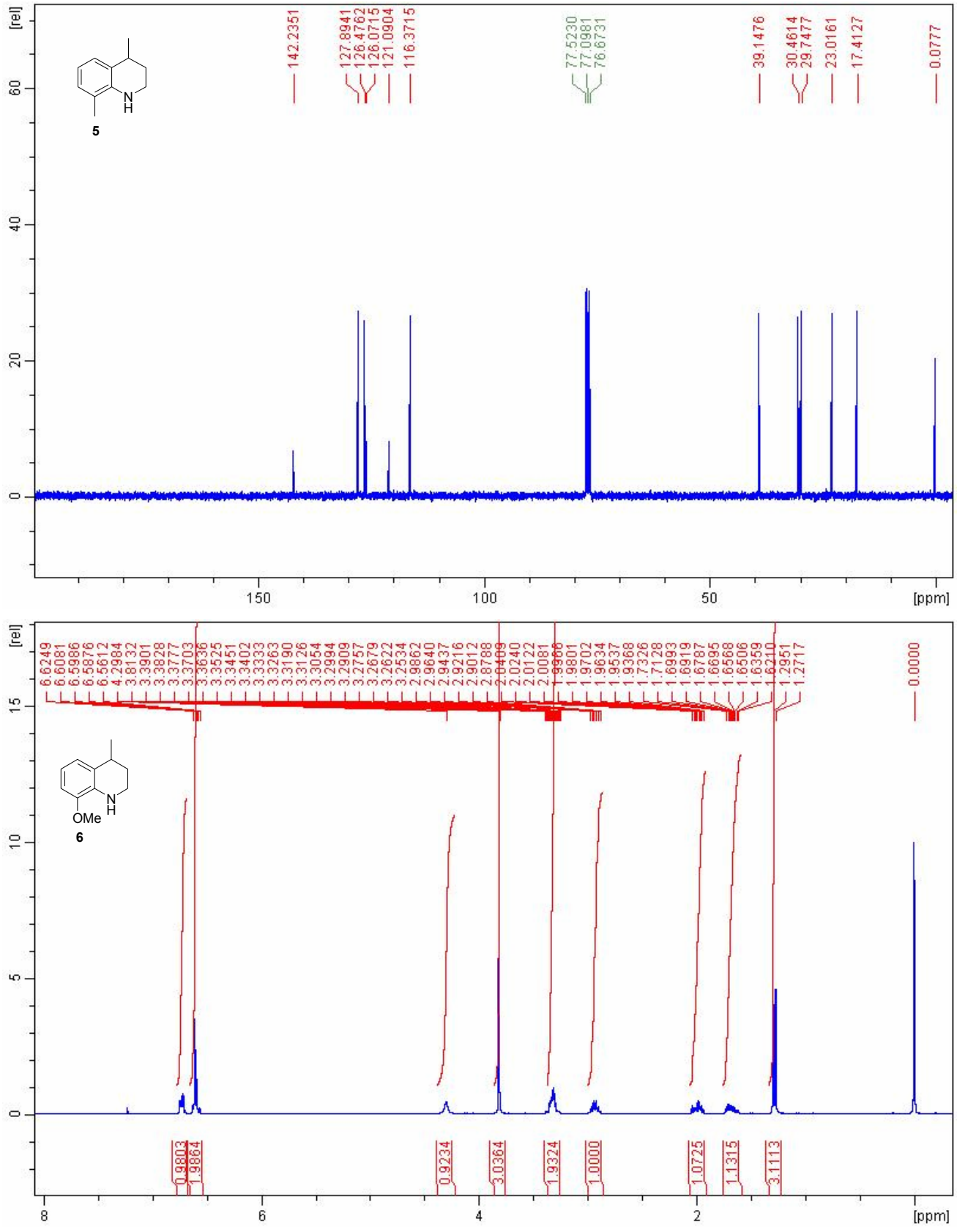
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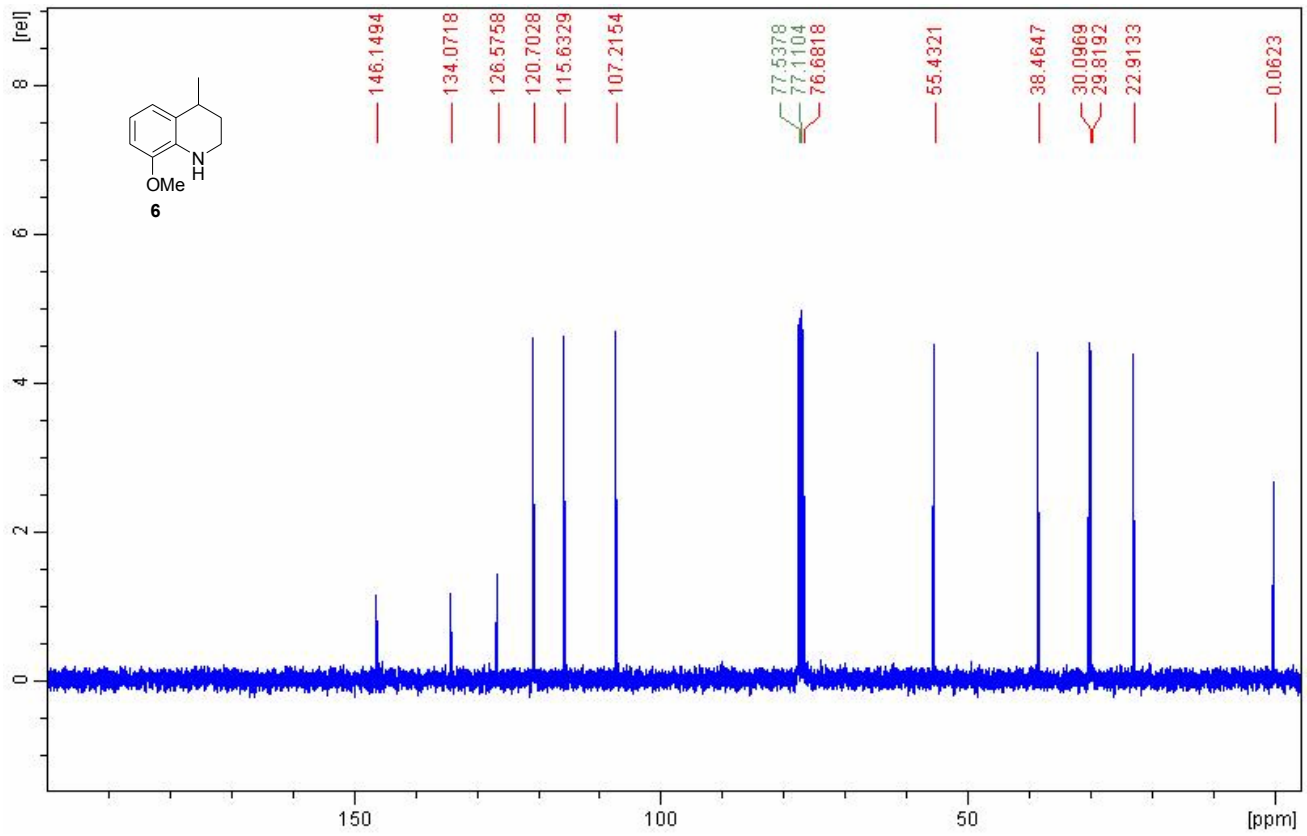
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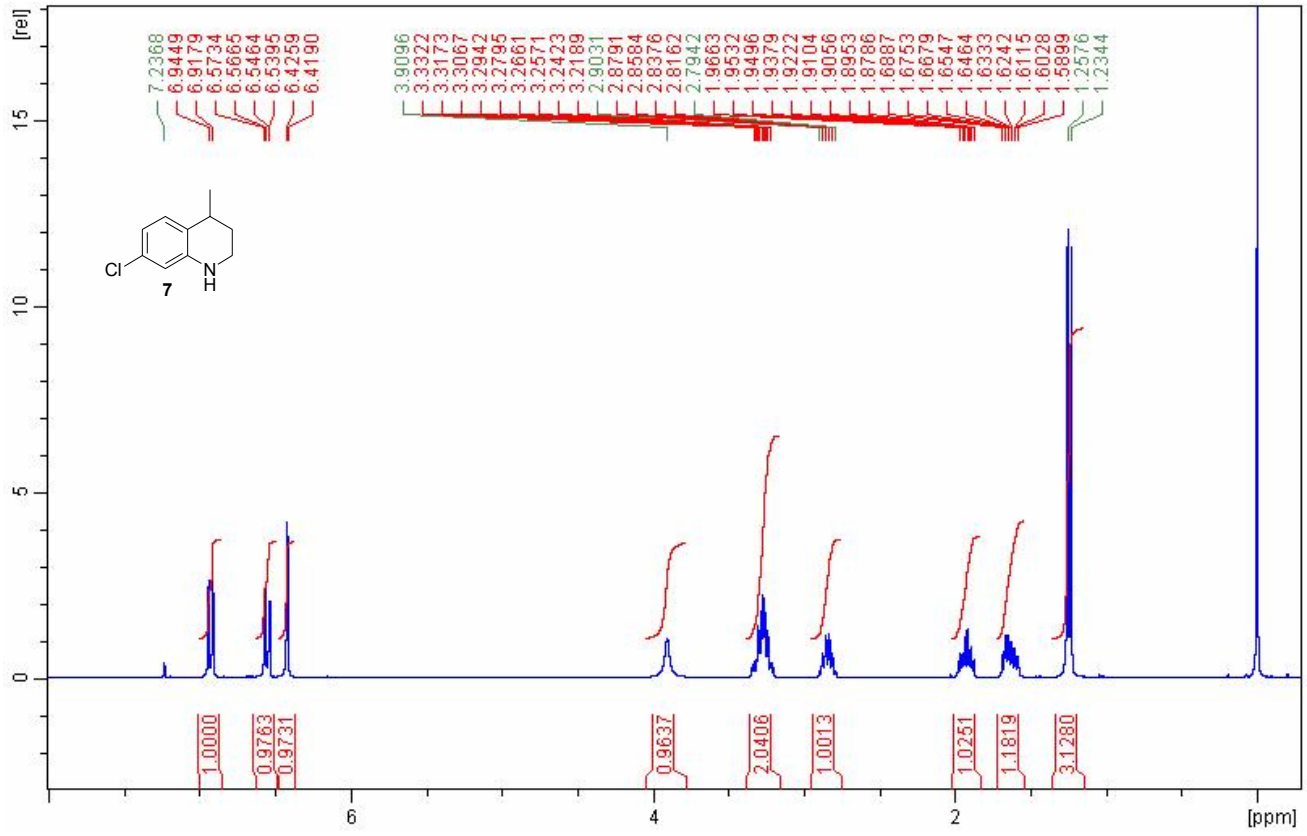
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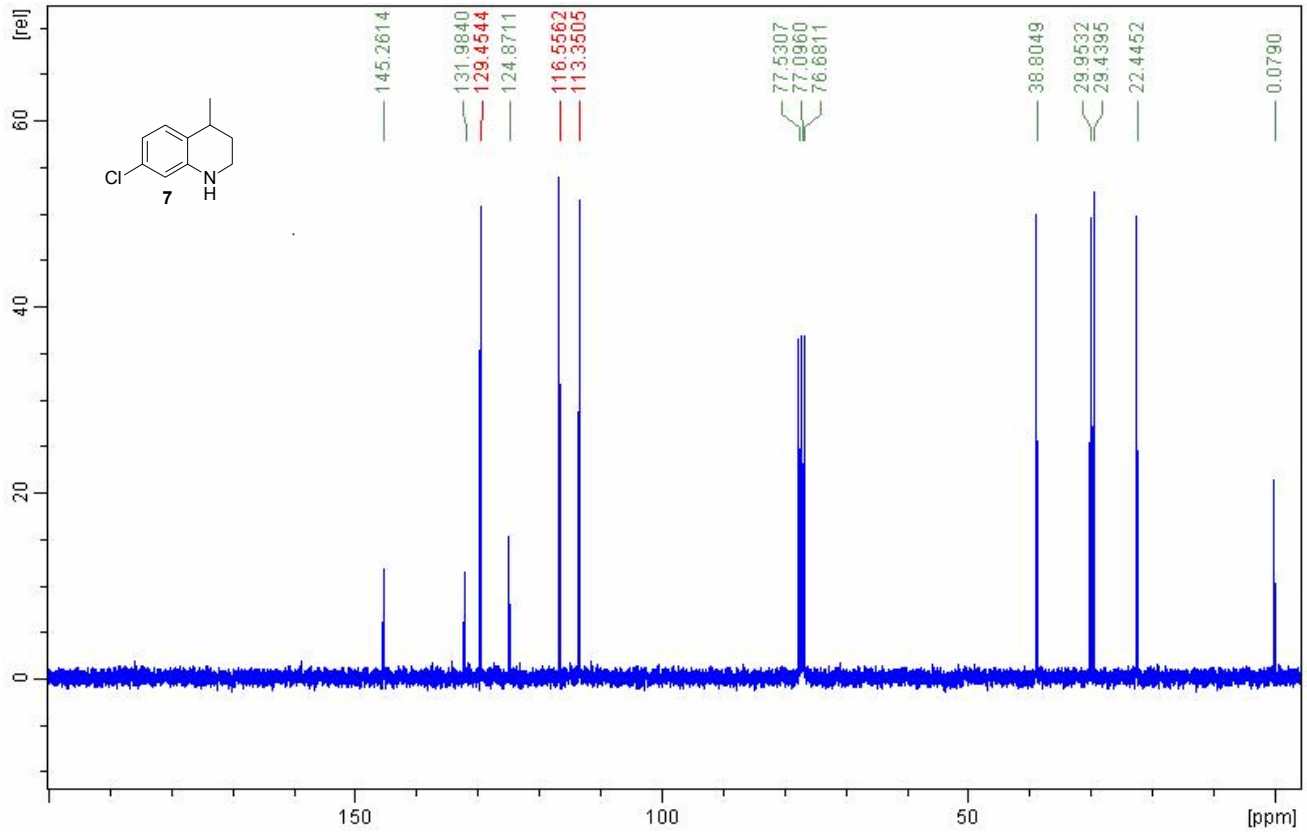
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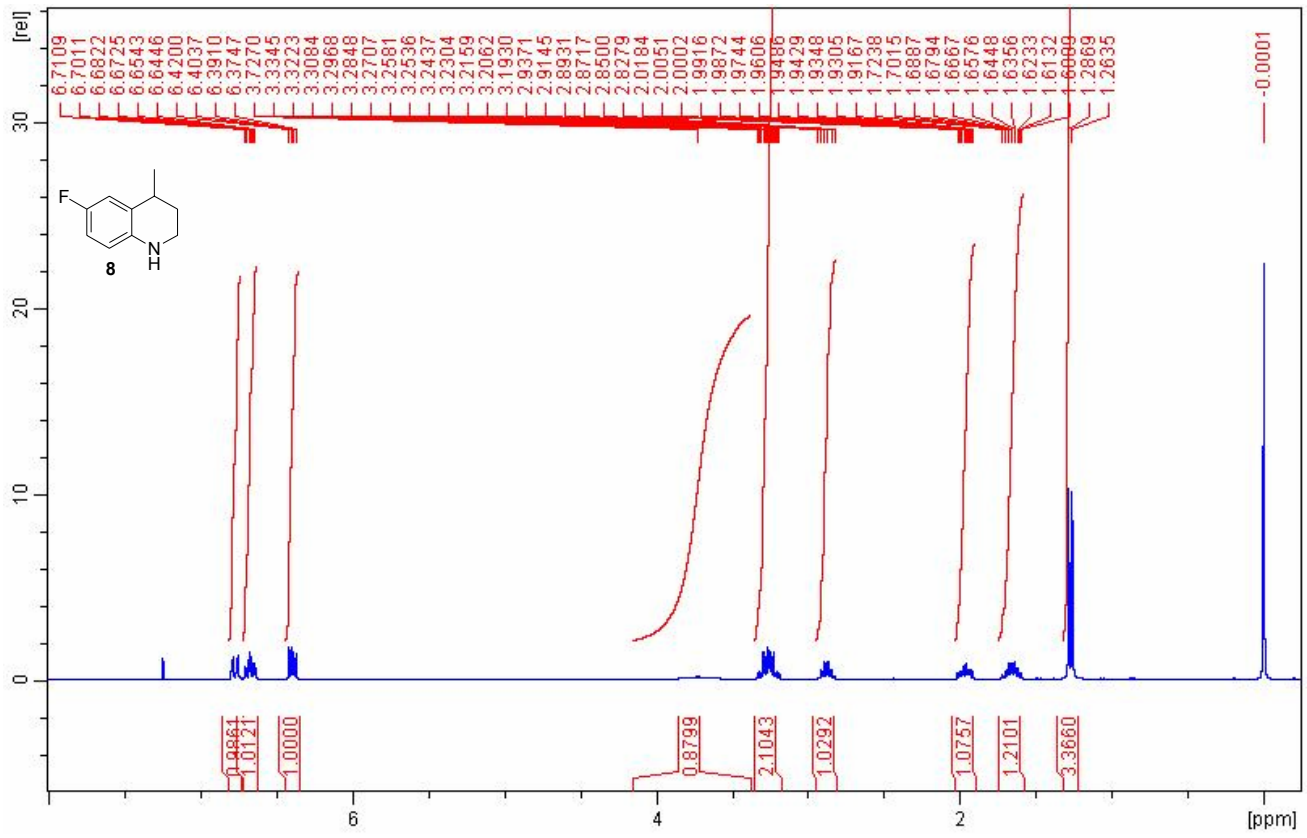
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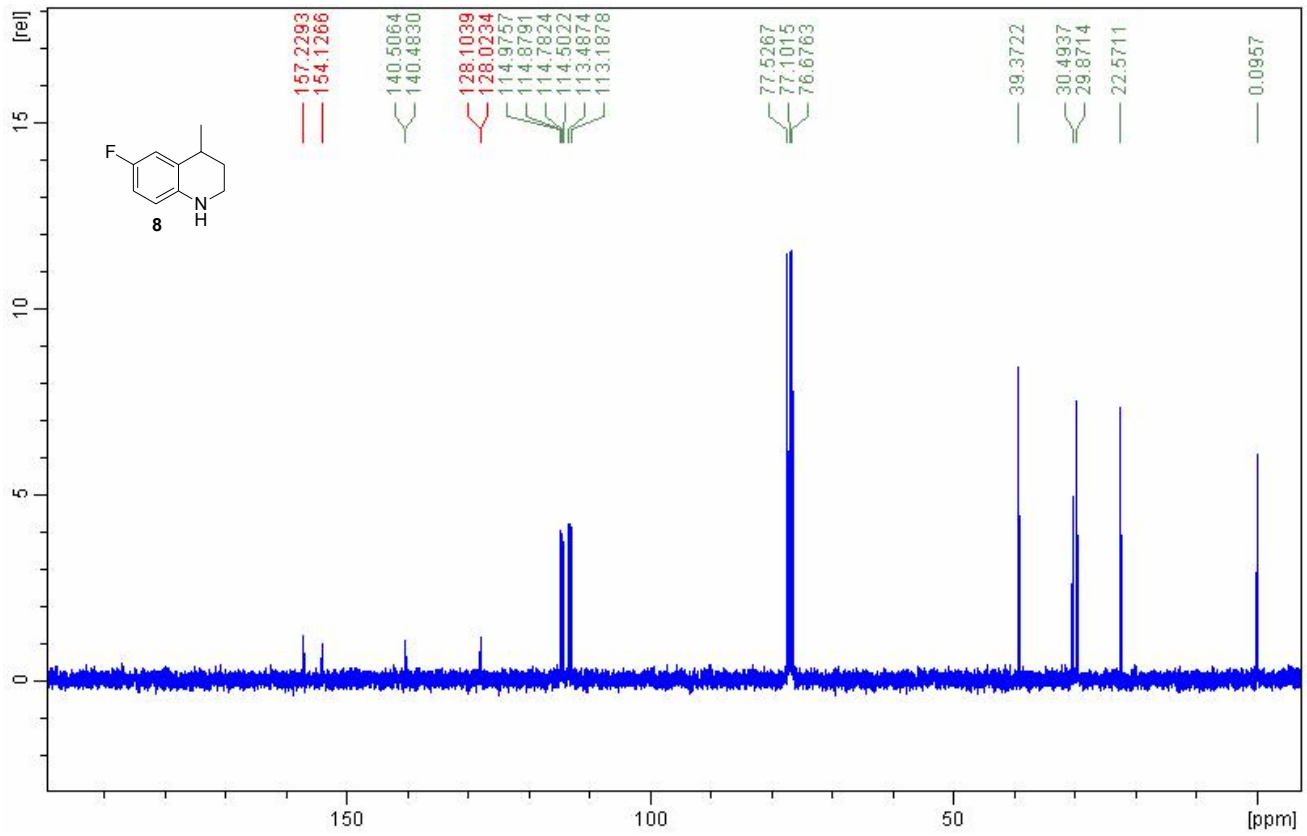
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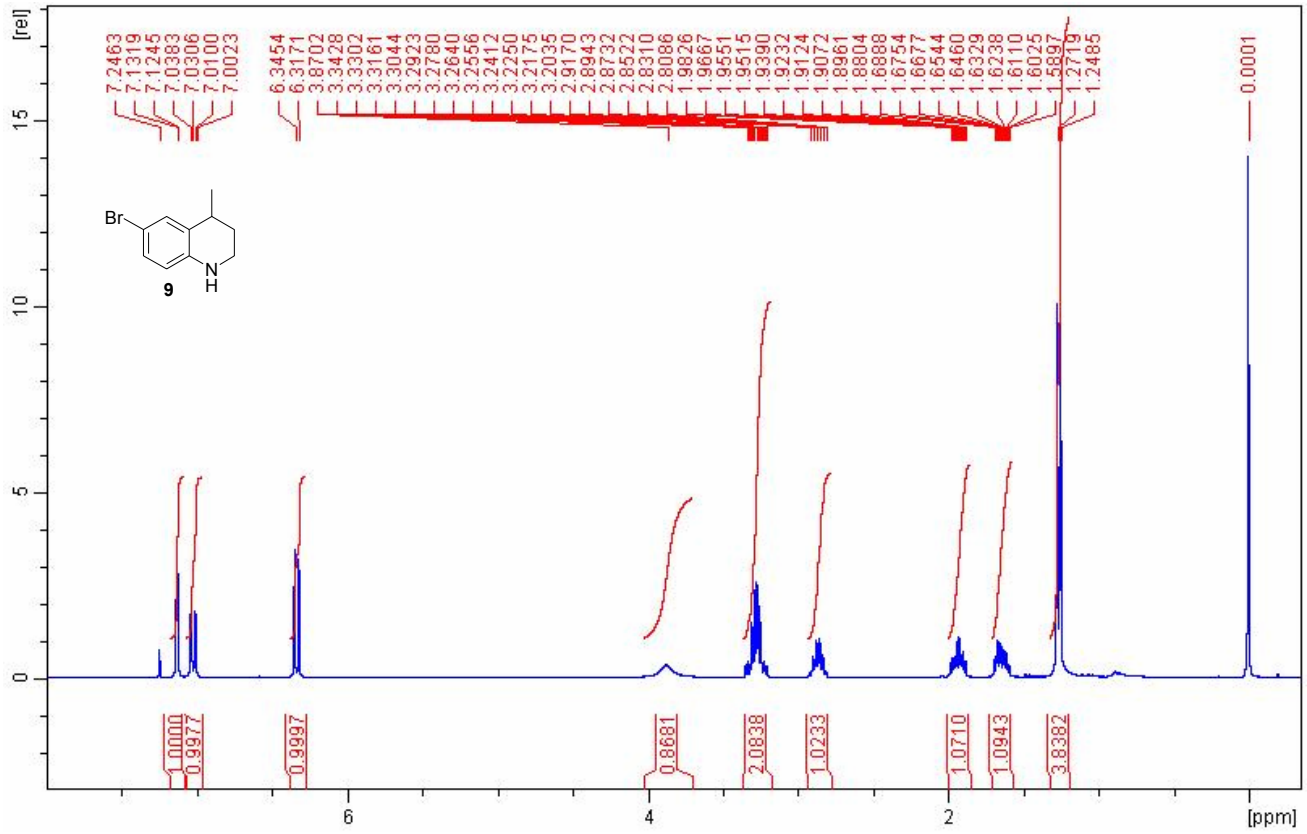
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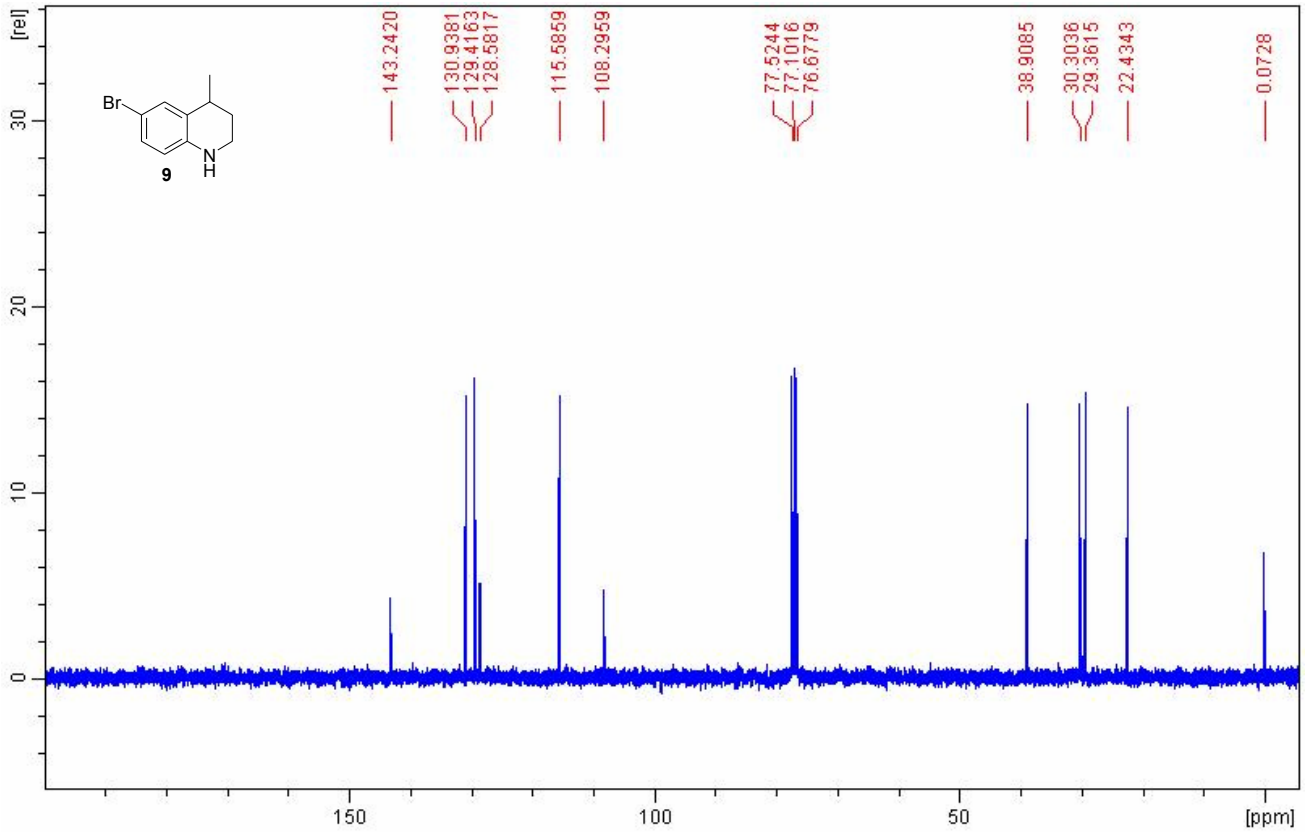
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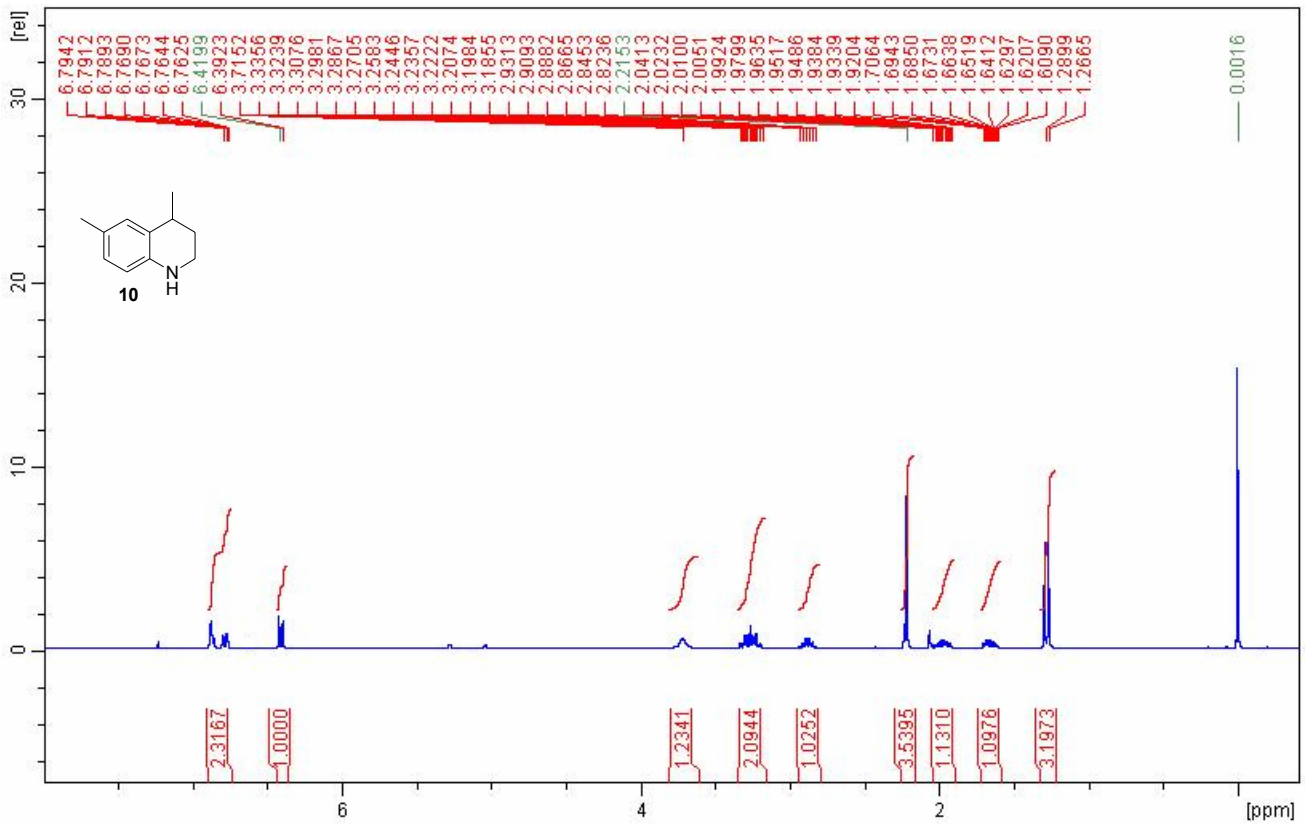
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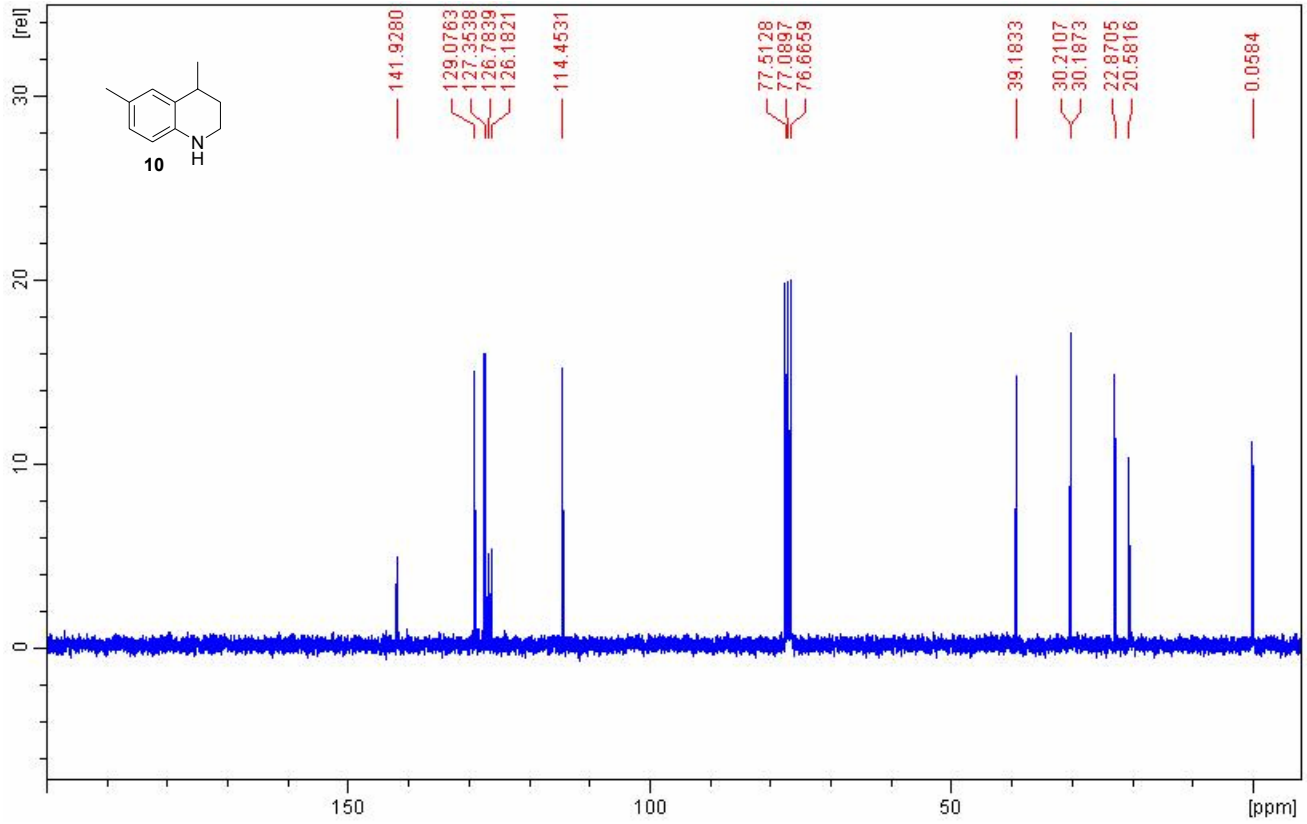
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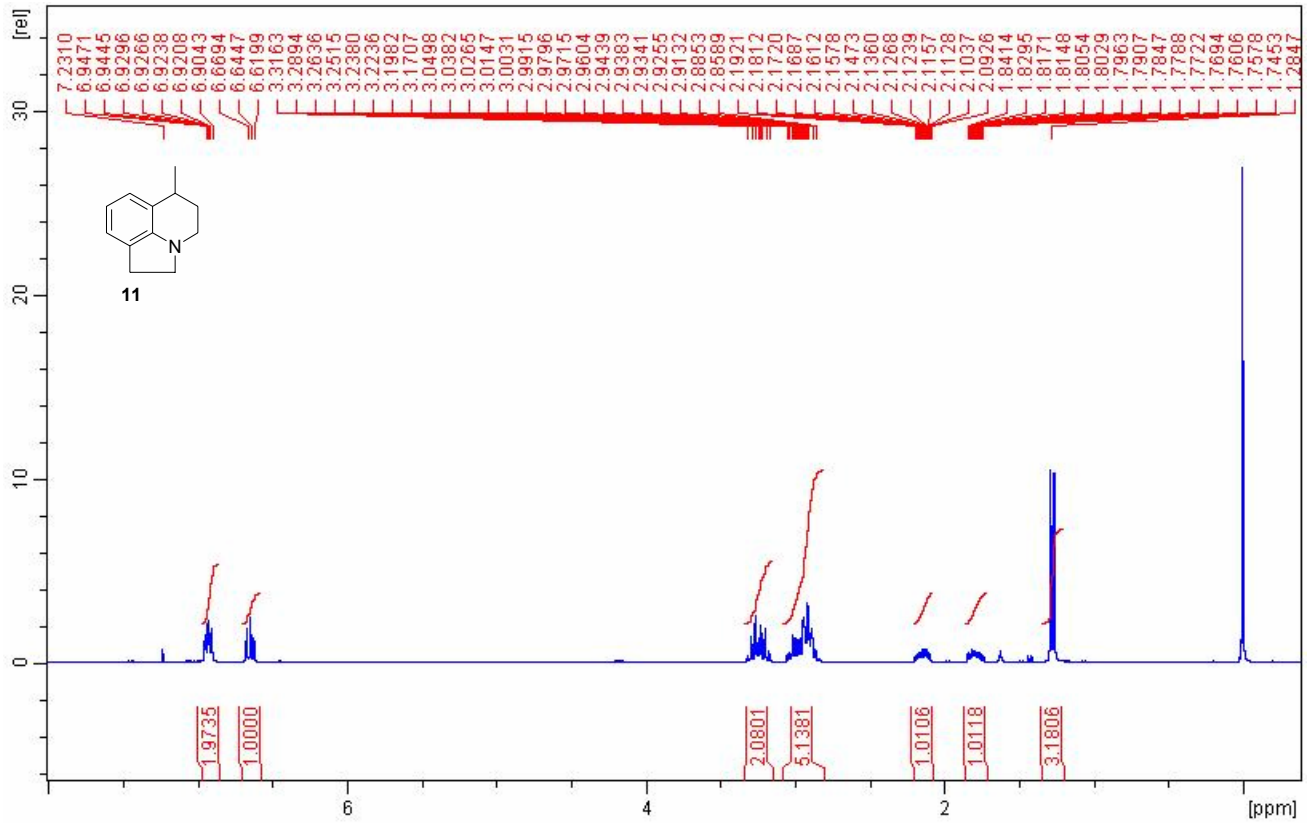
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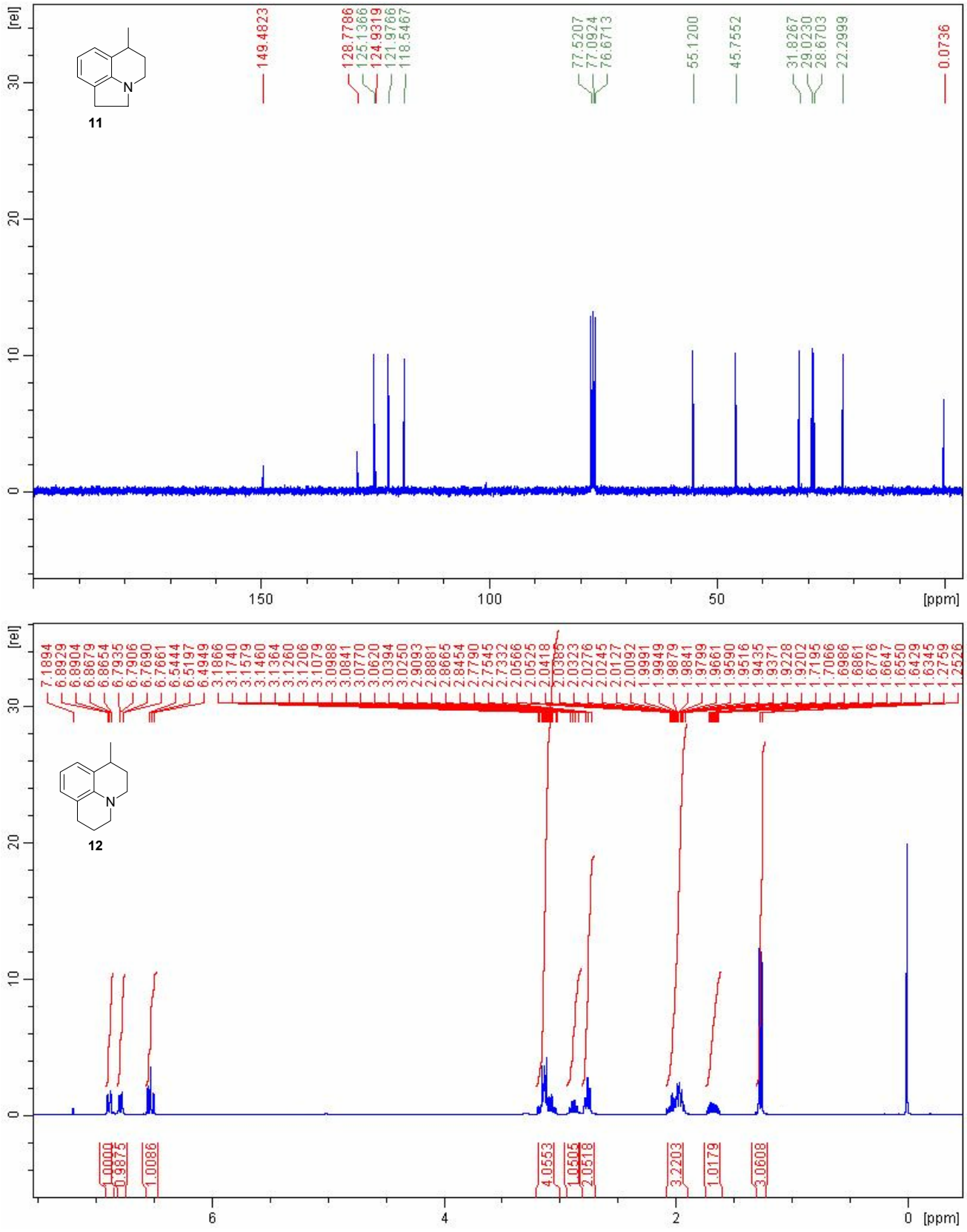
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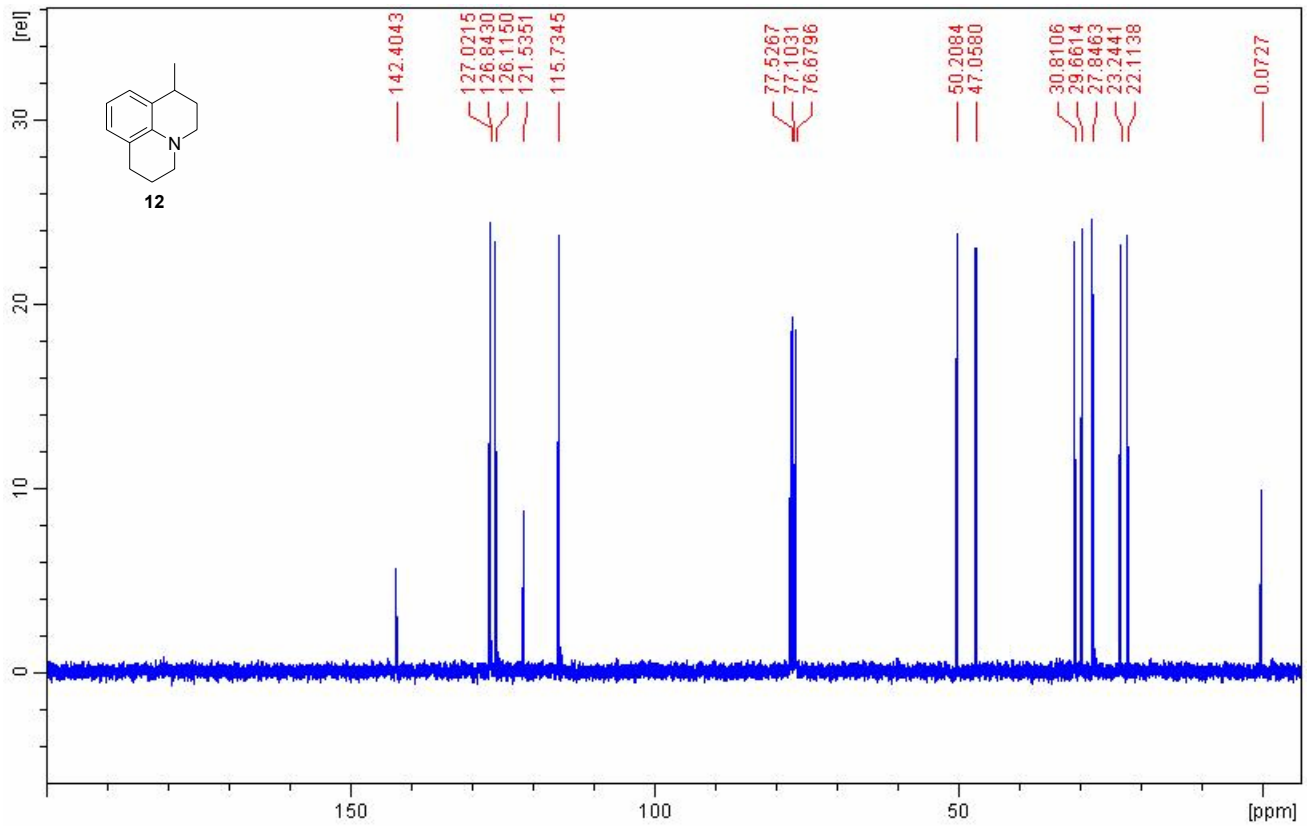
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