Supporting Information

# Experimental and Computational Probes of a Self-Assembled Capsule

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**(1) General** NMR spectra were recorded on a Bruker DRX-600 spectrometer. Proton ($^{1}$H) chemical shifts, reported in parts per million (ppm), were indirectly referenced to external tetramethylsilane employing resonances due to trace monoprotio-solvent as an internal reference. Deuterated NMR solvents were obtained from Cambridge Isotope Laboratories, Inc., and used without further purification.

**(2) Materials** Compounds **3a**, **6a** – **6c**, and **5** were purchased from Aldrich Chemical Company, and were used as received. The compound **6e** was purchased from Acros Organics, and were used as received. The starting materials for other biaryls, 3-bromopyridine, 2-bromopyridine, 2-bromo-5-methylpyridine, 4-bromotoluene, 5-bromopyrimidine, *p*-tolylboronic acid, 4-ethylphenylboronic acid,

4-*n*-propylphenylboronic acid, and 4-*n*-butylphenylboronic acid, were purchased from Aldrich Chemical Company., and were used without further purification. The 5-bromo-2-methylpyridine was purchased from Apollo Scientific Ltd., and was used without further purification. The reagents for cross-coupling reactions, $Pd(OAc)_2$, $K_3PO_4$, $K_2CO_3$, and tetra-*n*-butylammonium bromide were purchased from Aldrich Chemical Company. The catalyst $Pd(OAc)_2(Cy_2NH)_2$ was prepared according to the literature.[1] The cylindrical cavitand **1** was prepared according to the literature.[2]

**(3) Complete list of author for Guassian, Inc., Pittsburgh, PA, 2002:** Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Montgomery, Jr., J. A.; Vreven, T.; Kudin, K. N.; Burant, J. C.; Millam, J. M.; Iyengar, S. S.; Tomasi, J.; Barone, V.; Mennucci, B.; Cossi, M.; Scalmani, G.; Rega, N.; Petersson, G. A.; Nakatsuji, H.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Klene, M.; Li, X.; Knox, J. E.; Hratchian, H. P.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Ayala, P. Y.; Morokuma, K.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Zakrzewski, V. G.; Dapprich, S.; Daniels, A. D.; Strain, M. C.; Farkas, O.; Malick, D. K.; Rabuck, A. D.; Raghavachari, K.; Foresman, J. B.; Ortiz, J. V.; Cui, Q.; Baboul, A. G.; Clifford, S.; Cioslowski, J.; Stefanov, B. B.; Liu, G.; Liashenko, A.; Piskorz, P.; Komaromi, I.; Martin, R. L.; Fox, D. J.; Keith, T.; Al-Laham, M. A.; Peng, C. Y.; Nanayakkara, A.; Challacombe, M.; Gill, P. M. W.; Johnson, B.; Chen, W.; Wong, M. W.; Gonzalez, C.; and Pople, J. A.; Gaussian, Inc., Wallingford CT, 2004.

**(4) Preparation of biaryls in Figure 1b**. The biaryls **2a** – **2g**, **3b** – **3g**, **6d**, and **7a** – **7c** were prepared according to the Suzuki-Miyaura cross-coupling under aerobic conditions.[1] The synthesis of biaryl **4** was referred to the literature.[3] Typical procedures about **2f** and **4** were described below, as well as data of other biaryls. The $^1$H NMR and $^{13}$C NMR spectra of **2a** – **2g**, **3b** – **3g**, **4**, **6d**, and **7a** – **7c** are also attached in from page 8S – 44S.

**2f**: In the air, to a 50 mL one neck flask $K_3PO_4$ (850 mg, 4 mmol), $Pd(OAc)_2(Cy_2NH)_2$ (23 mg, 0.04 mmol), 5-bromo-2-methylpyridine (344 mg, 2 mmol), 4-*n*-propylboronic acid (394 mg, 2.4 mmol), and EtOH (6 mL) were added. After stirring at ambient temperature

for overnight, EtOAc (50 mL) was added, and the mixture was washed with water and brine, and dried over $Na_2SO_4$. Evaporation of the solvent provided the crude product, which was purified by column chromatography to afford the desired products in 50% yield (210 mg) as pale yellow crystals. $^1$H NMR (600 MHz, $C_6D_6$) δ 8.91 (d, *J* = 2.4 Hz, 1H), 7.40 (dd, *J* = 2.4 Hz, 7.8 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.74 (d, *J* = 7.8 Hz, 1H), 2.47 (s, 3H), 2.43 (t, *J* = 7.2 Hz, 2H), 1.52 (dt, *J* = 7.2 Hz, 7.2 Hz, 2H), 0.849 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.3, 148.0, 142.2, 136.0, 134.1, 133.8, 129.4, 127.1, 122.9, 37.9, 24.8, 24.2, 13.9. HRMS (ESI, $MH^+$): Calcd. For $C_{15}H_{18}N$: 212.1434. Found: 212.1440.

**4**[3]: Under a nitrogen atmosphere, a mixture of $K_2CO_3$ (1.10 g, 8 mmol), $Pd(OAc)_2$ (90 mg, 0.4 mmol), tetra-*n*-butylammonium bromide (1.29 g, 4 mmol) and 5-bromo-2-methylpyridine (1.37 g, 8 mmol) in a mix of DMF (0.9 mL) / $H_2O$ (0.35 mL) was stirred for 2 minutes at 115 °C. Isopropanol (480 mg, 8 mmol) was added. After stirring for 48 h, the reaction mixture was allowed to cool to ambient temperature. To the mixture was water and ether, and the organic phase was washed with water and brine, and dried over $Na_2SO_4$. Evaporation of the solvent provided the crude product, which was purified by column chromatography to afford the desired products in 51% yield (377 mg) as white crystals. $^1$H NMR (600 MHz, $C_6D_6$) δ 8.70 (d, *J* = 2.4 Hz, 2H), 7.18 (dd, *J* = 2.4 Hz, 7.8 Hz, 2H), 6.66 (d, *J* = 7.8 Hz, 2H), 2.44 (s, 6H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.9, 147.9, 134.0, 130.8, 123.0, 24.2. HRMS (ESI, $MH^+$): Calcd. For $C_{12}H_{13}N_2$: 185.1073. Found: 185.1067.

**2a**[5]: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.97 (d, *J* = 1.2 Hz, 1H), 8.52 (d, *J* = 3.6 Hz, 1H), 7.39 – 7.37 (m, 1H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 6.78 – 6.77 (m, 1H), 2.10 (s, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 148.8, 148.7, 137.7, 136.6, 135.5, 133.6, 129.9, 127.2, 123.4, 21.0. HRMS (ESI, $MH^+$): Calcd. For $C_{12}H_{11}N$: 170.0964. Found: 170.0970.

**2b**: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.89 (d, *J* = 2.4 Hz, 1H), 7.39 (dd, *J* = 2.4 Hz, 7.8 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 7.8 Hz, 1H), 2.48 (s, 3H), 2.11 (s, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.3, 148.0, 137.4, 135.7, 134.1, 133.8, 129.9, 127.1, 122.9, 24.2, 21.0. HRMS (ESI, $MH^+$): Calcd. For $C_{13}H_{13}N$: 184.1121. Found: 184.1123.

**2c**: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 8.98 (d, *J* = 1.8 Hz, 1H), 8.52 (dd, *J* = 1.8 Hz, 4.8 Hz, 1H), 7.41 – 7.39 (m, 1H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 7.8 Hz, 2H), 6.78 (dd, *J* = 4.8 Hz, 8.4 Hz, 1H), 2.44 (q, *J* = 7.8 Hz, 2H), 1.08 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 148.8, 148.7, 144.1, 136.6, 135.8, 133.7, 128.8, 127.4, 123.4, 28.8, 15.7. HRMS (ESI, $MH^+$): Calcd. For $C_{13}H_{14}N$: 184.1121. Found: 184.1128.

**2d**: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 8.91 (s, 1H), 7.40 (dd, *J* = 2.4 Hz, 7.8 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 6.74 (d, *J* = 7.8 Hz, 1H), 2.47 (s, 3H), 2.45 (q, *J* = 7.8 Hz, 2H), 1.09 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.3, 148.0, 143.8, 135.9, 134.1, 133.8, 128.7, 127.2, 122.9, 28.8, 24.2, 15.8. HRMS (ESI, $MH^+$): Calcd. For $C_{14}H_{16}N$: 198.1277. Found: 198.1280.

**2e**: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 8.99 (d, *J* = 2.4 Hz, 1H), 8.52 (dd, *J* = 2.4 Hz, 4.8 Hz, 1H), 7.41 – 7.39 (m, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 2.42 (t, *J* = 7.8 Hz, 2H), 1.52 (dt, *J* = 7.8 Hz, 7.8 Hz, 2H), 0.84 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 148.8, 148.7, 142.5, 136.6, 135.8, 133.7, 129.7, 127.3, 123.4, 37.9, 24.8, 13.9. HRMS (ESI, $MH^+$): Calcd. For $C_{14}H_{16}N$: 198.1277. Found: 198.1284.

**2g**[6]: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 9.00 (d, *J* = 2.4 Hz, 1H), 8.53 – 8.52 (m, 1H), 7.42 – 7.40 (m, 1H), 7.26 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 6.79 – 6.77 (m, 1H), 2.46 (t, *J* = 7.8 Hz, 2H), 1.49 (dd, *J* = 7.8 Hz, 7.8 Hz, 2H), 1.24 (dt, *J* = 7.2 Hz, 7.8 Hz, 2H), 0.86 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 148.8, 148.7, 142.8, 136.6, 135.8, 133.7, 129.4, 127.3, 123.4, 35.6, 33.9, 22.6, 14.1. HRMS (ESI, $MH^+$): Calcd. For $C_{15}H_{18}N$: 212.1434. Found: 212.1434.

**3b**[7]: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 8.49 (d, *J* = 2.4 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.11 (d, *J* = 7.8 Hz, 2H), 6.99 (dd, *J* = 2.4 Hz, 8.4 Hz, 1H), 2.13 (s, 3H), 1.85 (s, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 155.0, 150.5, 138.5, 137.3, 136.8, 131.0, 129.7, 127.0, 119.2, 21.2, 17.8. MS (EI, *m/z*): 183 ($M^+$).

**3c**: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 8.55 – 8.53 (m, 1H), 8.02 (d, *J* = 7.8 Hz, 2H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.24 – 7.21 (m, 1H), 7.12 (d, *J* = 7.8 Hz, 2H), 6.75 – 6.73 (m, 1H), 2.55 (q, *J* =

7.2 Hz, 1H), 1.20 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.6, 149.9, 145.3, 137.5, 136.2, 128.5, 127.3, 121.7, 119.8, 28.9, 15.6. HRMS (ESI, $MH^+$): Calcd. For $C_{13}H_{14}N$: 184.1121. Found: 184.1128.

**3d**: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.49 – 8.48 (m, 1H), 8.17 (d, *J* = 7.8 Hz, 2H), 7.36 (d, *J* = 7.8 Hz, 2H), 7.16 – 7.14 (m, 1H), 6.99 (dd, *J* = 2.4 Hz, 7.8 Hz, 1H), 2.47 (q, *J* = 7.8 Hz, 2H), 1.85 (s, 3H), 1.09 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 155.0, 150.4, 144.9, 137.5, 136.9, 131.1, 128.5, 127.1, 119.4, 28.9, 17.8, 15.7. HRMS (ESI, $MH^+$): Calcd. For $C_{14}H_{16}N$: 198.1277. Found: 198.1283.

**3e**: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.60 – 8.58 (m, 1H), 8.14 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.14 – 7.11 (m, 3H), 6.65 (m, 1H), 2.44 (t, *J* = 7.2 Hz, 2H), 1.51 (dt, *J* = 7.2 Hz, 7.8 Hz, 2H), 0.83 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.6, 150.0, 143.7, 137.5, 136.3, 129.1, 127.2, 121.7, 119.8, 38.0, 24.7, 13.9. HRMS (ESI, $MH^+$): Calcd. For $C_{14}H_{16}N$: 198.1277. Found: 198.1279.

**3f**: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.51 – 8.49 (m, 1H), 8.19 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.16 – 7.14 (m, 3H), 7.00 – 6.97 (m, 1H), 2.45 (t, *J* = 7.8 Hz, 2H), 1.85 (s, 3H), 1.53 (dt, *J* = 7.8 Hz, 7.2 Hz, 2H), 0.85 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 155.1, 150.5, 143.3, 137.6, 136.9, 131.1, 129.1, 127.0, 119.3, 38.0, 24.8, 17.8, 13.9. HRMS (ESI, $MH^+$): Calcd. For $C_{15}H_{18}N$: 212.1434. Found: 212.1436.

**3g**[8]: $^1$H NMR (600 MHz, $C_6D_6$) δ 8.60 – 8.59 (m, 1H), 8.15 (d, *J* = 7.8 Hz, 2H), 7.38 – 7.37 (m, 1H), 7.13 (d, *J* = 7.8 Hz, 2H), 7.12 – 7.10 (m, 1H), 6.64 (dd, *J* = 4.8 Hz, 7.2 Hz, 1H), 2.48 (t, *J* = 7.8 Hz, 2H), 1.49 (dd, *J* = 7.8 Hz, 7.2 Hz, 2H), 1.23 (dt, *J* = 7.8 Hz, 7.2 Hz, 2H), 0.85 (t, *J* = 7.8 Hz, 3H). HRMS (ESI, $MH^+$): Calcd. For $C_{15}H_{18}N$: 212.1434. Found: 212.1439.

**6d**[9]: $^1$H NMR (600 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 2.49 (q, *J* = 7.8 Hz, 2H), 2.16 (s, 3H), 1.12 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 143.1, 139.1, 138.9, 136.6, 129.8, 128.6, 127.3, 127.2, 28.8, 21.1, 15.9. MS (EI, *m/z*): 196 ($M^+$).

**7a**[10]: $^{1}$H NMR (600 MHz, $CDCl_3$) δ 9.18 (s, 1H), 8.93 (s, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 2.42 (s, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.1, 154.7, 139.1, 134.2, 131.3, 130.1, 126.8, 21.2. MS (EI, *m/z*): 170 ($M^+$).

**7b**: $^{1}$H NMR (600 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.69 (s, 2H), 7.02 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 2.40 (q, *J* = 7.2 Hz, 2H), 1.06 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 157.9, 154.7, 144.9, 134.2, 132.2, 128.9, 127.1, 28.8, 15.6. HRMS (ESI, $MH^+$): Calcd. For $C_{12}H_{13}N_2$: 185.1073. Found: 185.1079.

**7c**: $^{1}$H NMR (600 MHz, $C_6D_6$) δ 9.27 (s, 1H), 8.69 (s, 2H), 7.03 (d, *J* = 7.8 Hz, 2H), 6.95 (d, *J* = 7.8 Hz, 2H), 2.38 (t, *J* = 7.2 Hz, 2H), 1.48 (dd, *J* = 7.8 Hz, 7.2 Hz, 2H), 0.83 (t, *J* = 7.8 Hz, 3H). $^{13}$C NMR (150 MHz, $C_6D_6$) δ 158.0, 154.7, 143.4, 134.1, 132.2, 129.5, 127.0, 37.8, 24.7, 13.8. HRMS (ESI, $MH^+$): Calcd. For $C_{13}H_{15}N_2$: 199.1230. Found: 199.1226.

**(5) Competitive encapsulation studies**

In the competitive encapsulations in Table 1 and Table 2, the concentration of **1** was 4 mM, and that of each guest was performed in 50 mM. The mixture of the host and guests was warmed and sonicated to dissolve the host-guest complexes, and placed at ambient temperature for at least 30 min. The ratios were based on the peaks of alkyl side-chains located at the upfield of $^{1}$H NMR spectra, and determined on the integral values and/or weighing papers of the magnified peaks. All the $^{1}$H NMR spectra of encapsulated guests and the pairwise competition of Table 1 and Table 2 are shown in from page 45S to 57S.

**(6) Reference**

1) Tao, B.; Boykin, D. W. *J. Org. Chem.* **2004**, *69*, 4330 – 4335.

2) Hayashida, O.; Sebo, L.; Rebek, J., Jr. *J. Org. Chem.* **2002**, *67*, 8291 – 8298.

3) Hassan, J.; Penalva, V.; Lavenot, L.; Gozzi, C.; Lemaire, M. *Tetrahedron* **1998**, *54*, 13793 – 13804.

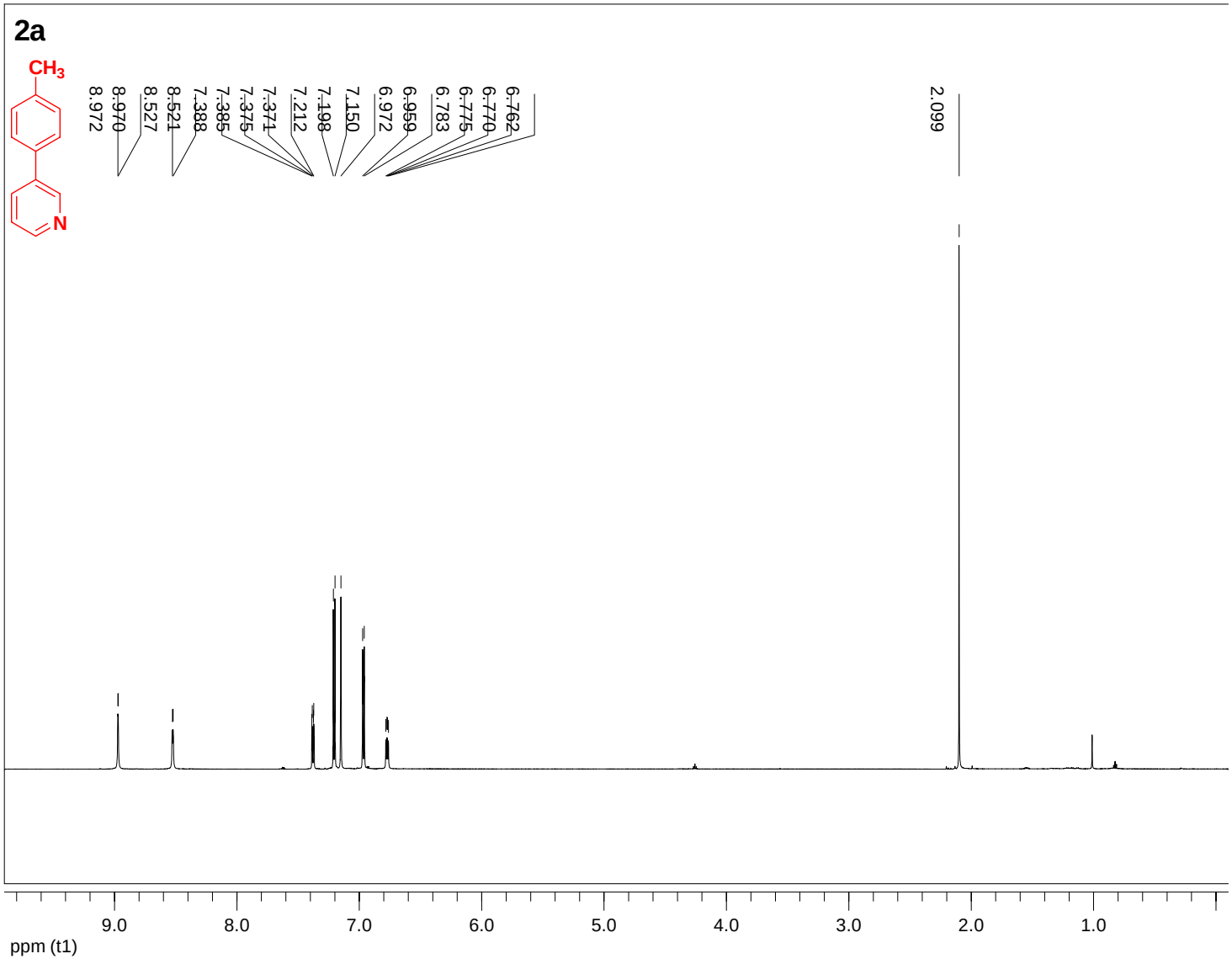
4) Cherioux, F.; Attias, A.-J.; Maillotte, H. *Advanced Functional Materials* **2002**, *12*, 203 – 208.

5) Inada, K.; Miyaura, N. *Tetrahedron* **2000**, *56*, 8661 – 8664.

6) Barder, T. E.; Walker, S. D.; Martinelli, J. R.; Buchwald, S. L. *J. Am. Chem. Soc.* **2005**,

*127*, 4685 – 4696.
7) Jung, S.; Kang, Y.; Kim, H.-S.; Kim, Y.-H.; Lee, C.-L., Kim, J.-J.; Lee, S.-K.; Kwon, S.-K. *Eur. J. Inorg. Chem.* **2004**, 3415 – 3423.
8) Chia, W.-L.; Shen, S. –W.; Lin, H.-C. *Tetrahedron Lett.* **2001**, *42*, 2177 – 2179.
9) Cho, C.-H.; Yun, H.-S.; Park, K. *J. Org. Chem.* **2003**, *68*, 3017 -3025.
10) Kano, S.; Yuasa, Y.; Shibuya, S.; Hibino, S. *Heterocycles* **1982**, *19*, 1079 – 1082.

**(7) $^{1}$H and $^{13}$C NMR spectra of biaryls 2a-g (page 8S – 21S), 3b-g (page 22S – 34S) 4 (page 35S – 36S), 6d (page 37S – 38S), 7a-c (page 39S – 44S), and all $^{1}$H NMR spectra of encapsulated biaryls (page 45S – 52S) and competitive encapsulation studies (page 53S – 57S).**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6.972
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6.775
6.770
6.762
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1.0
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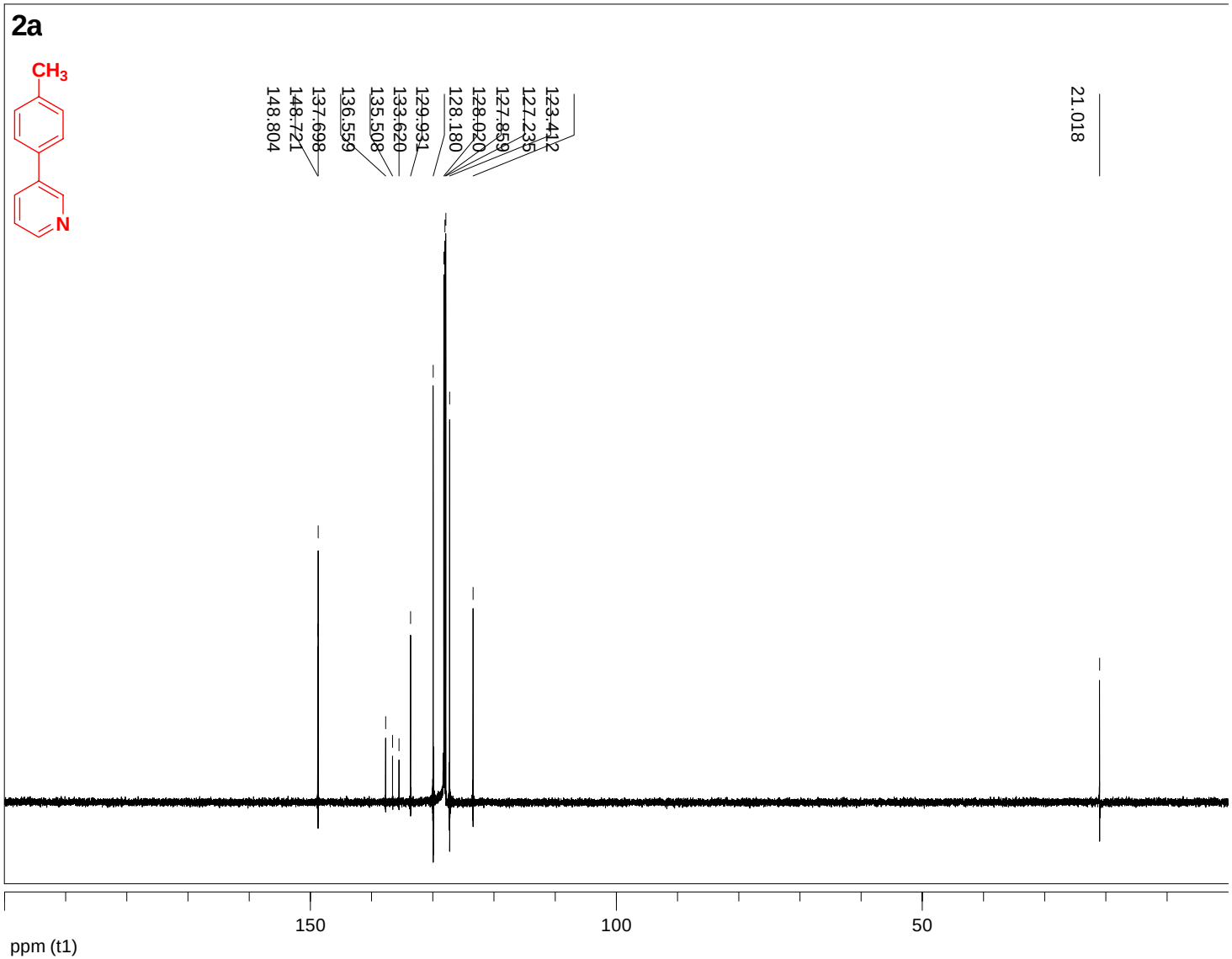
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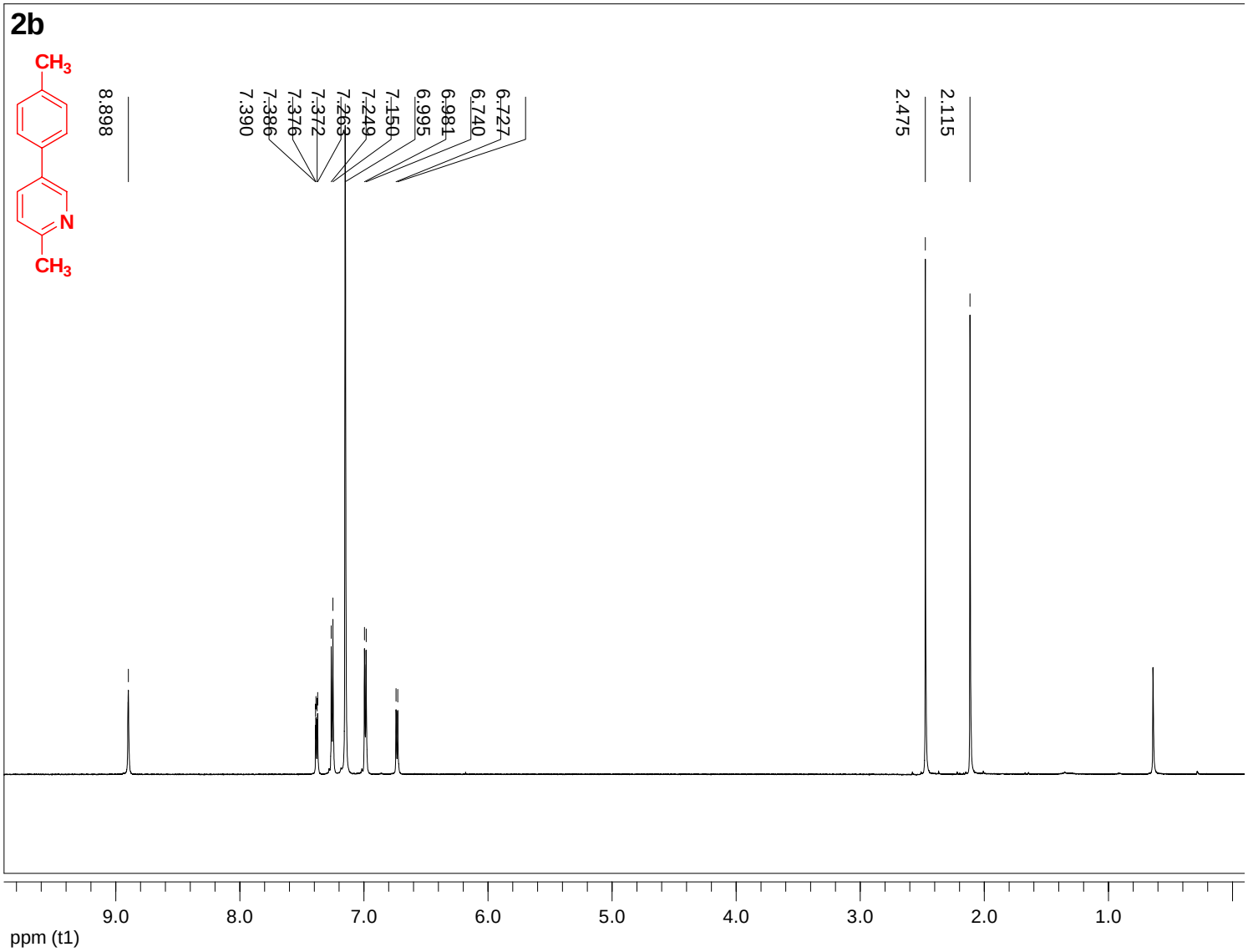
ppm (t1)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6.740
6.727
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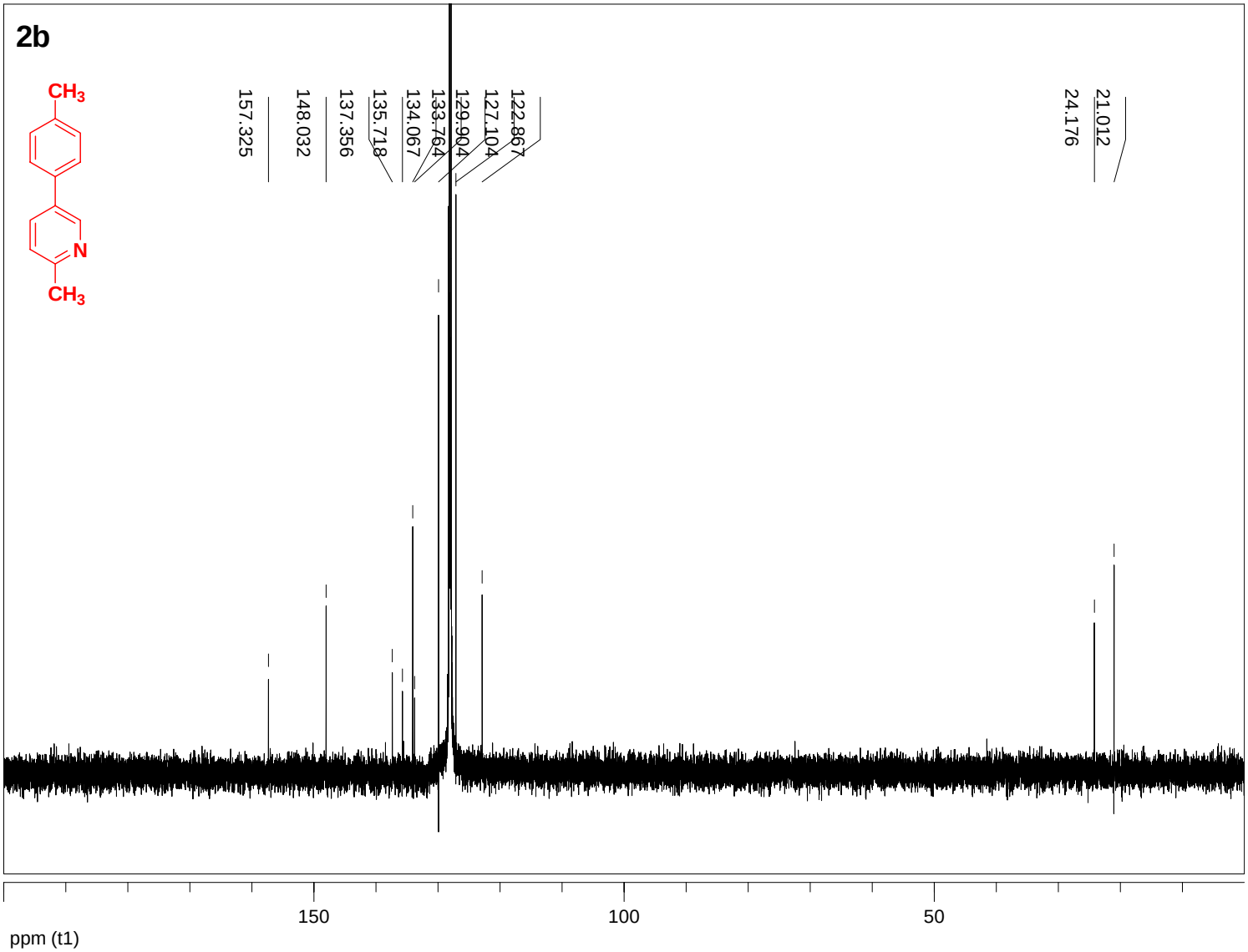
ppm (t1)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CH3
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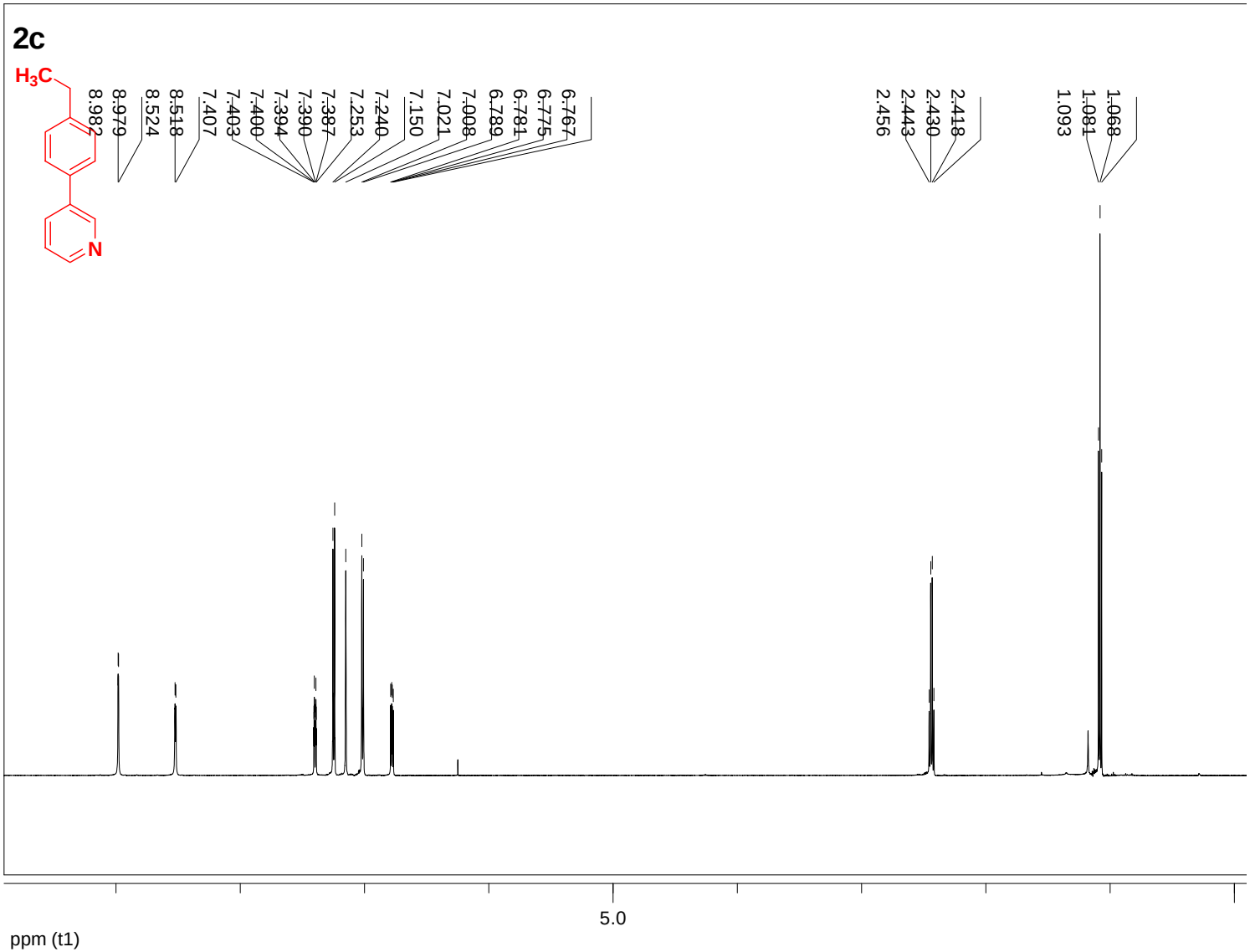
ppm (t1)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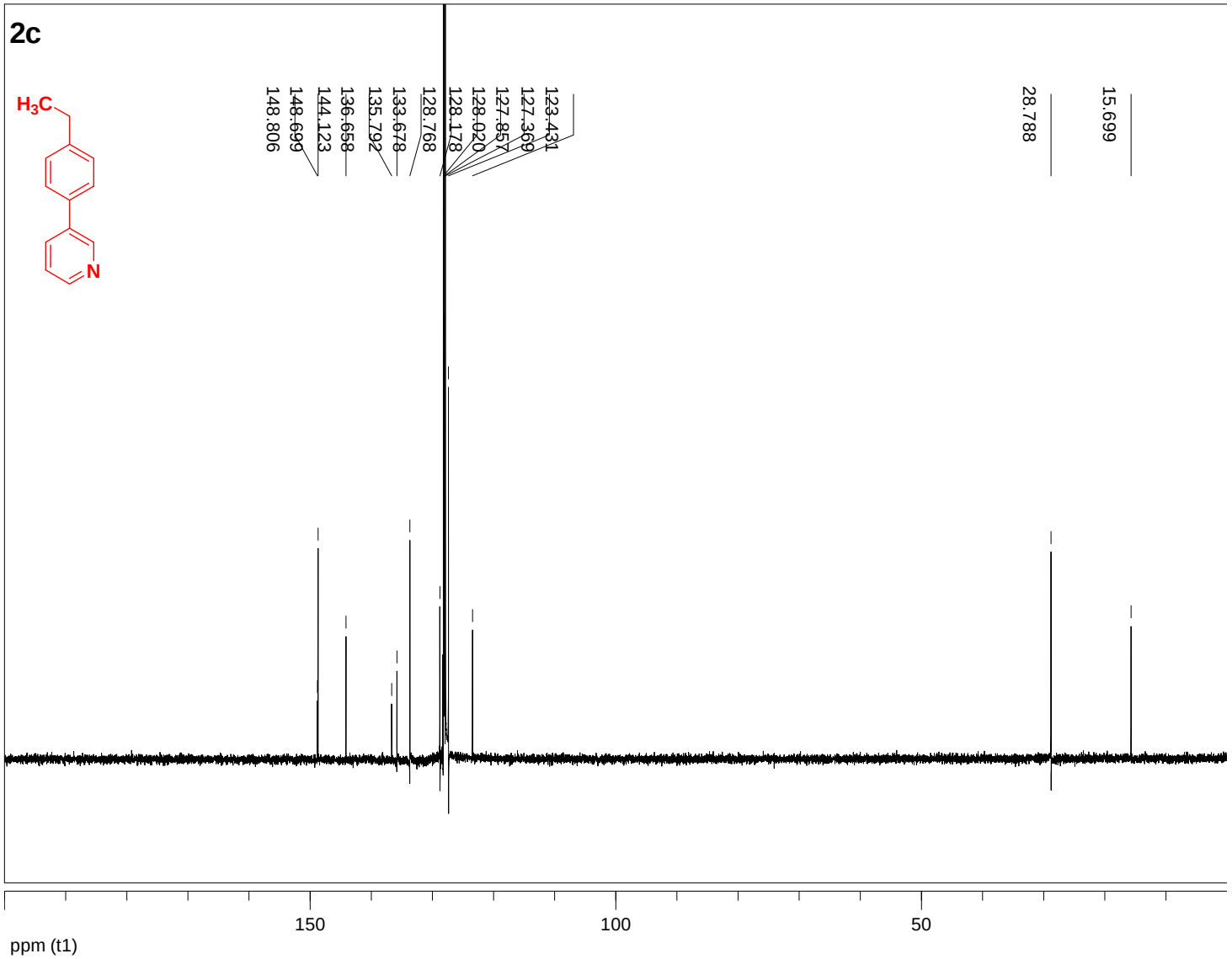
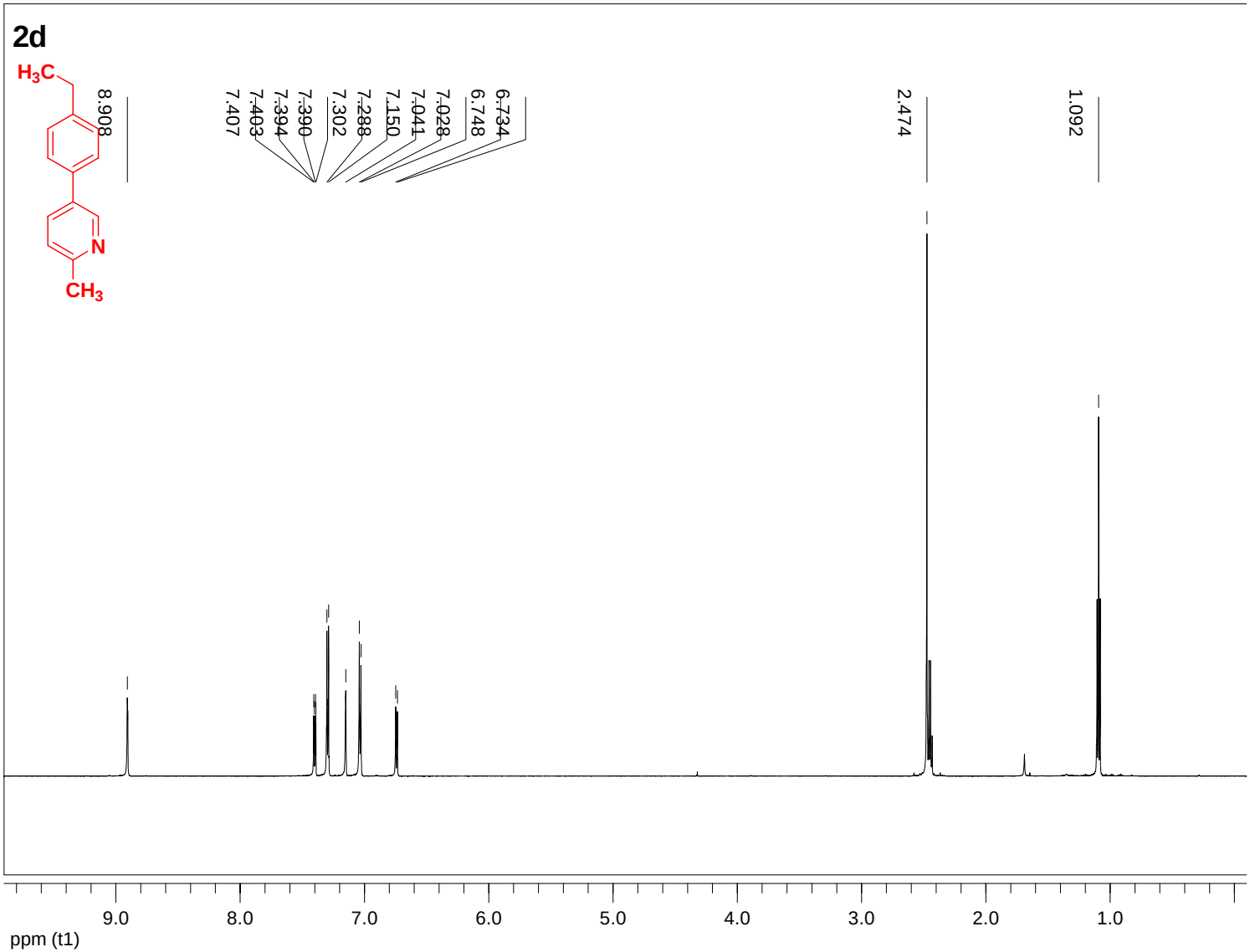
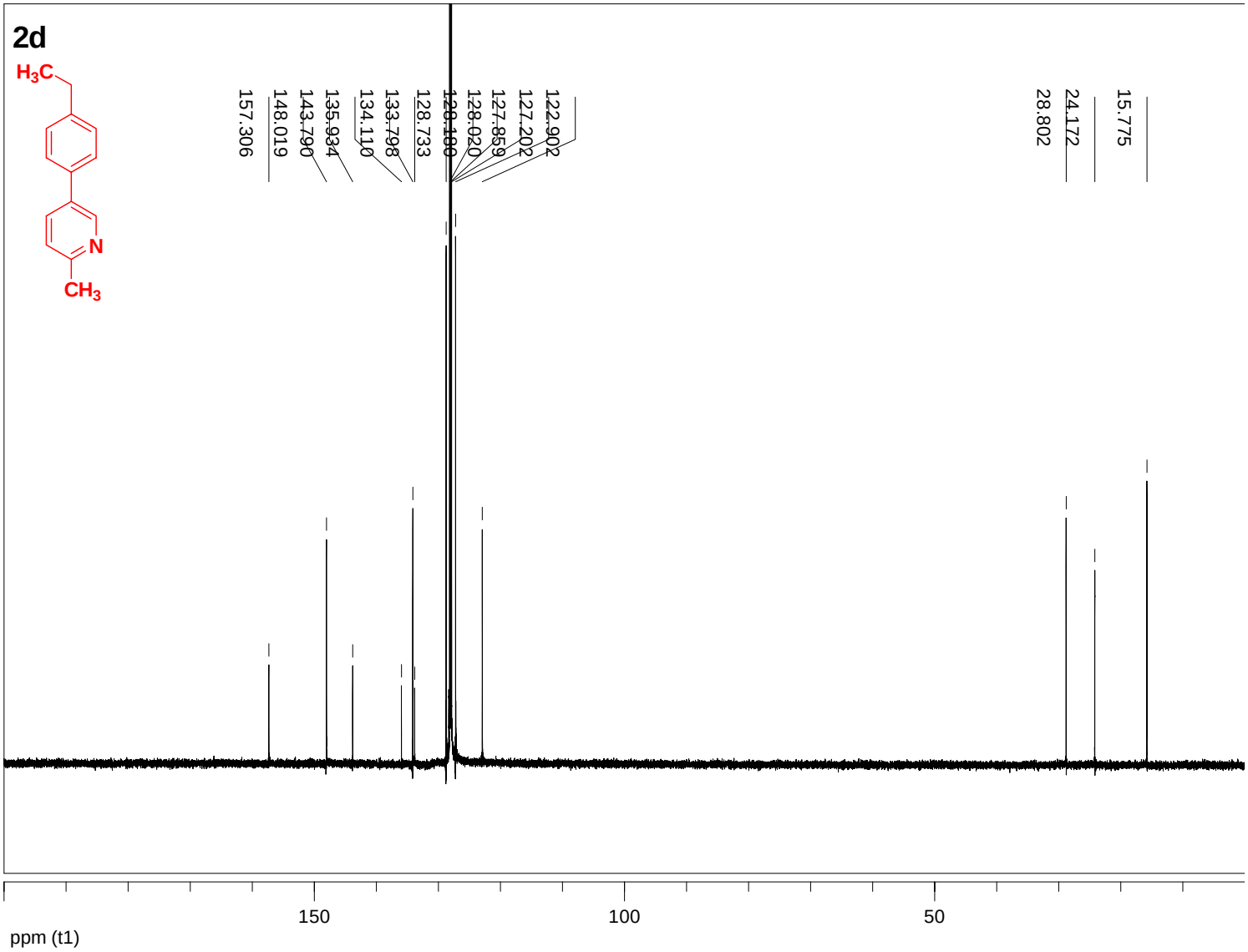
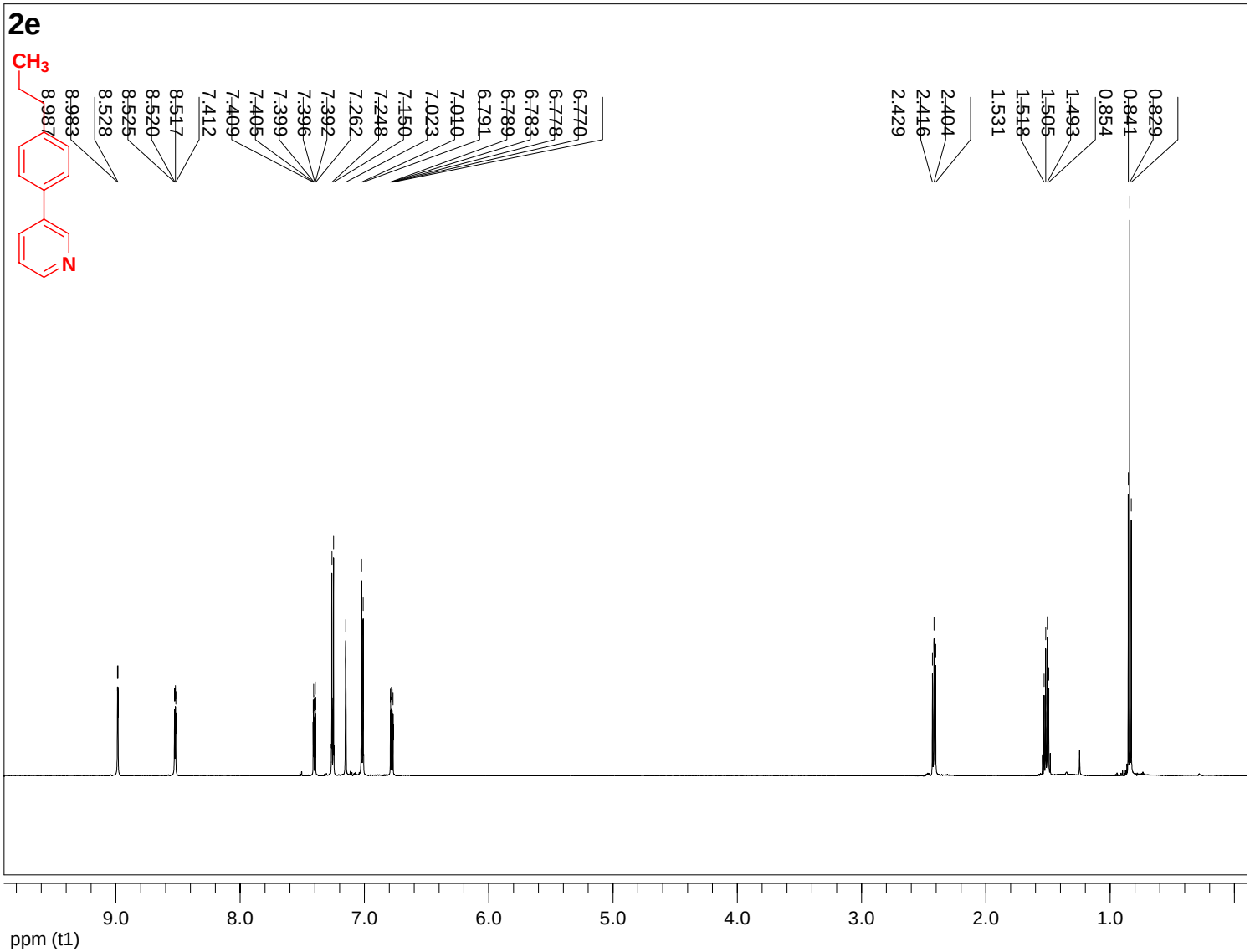
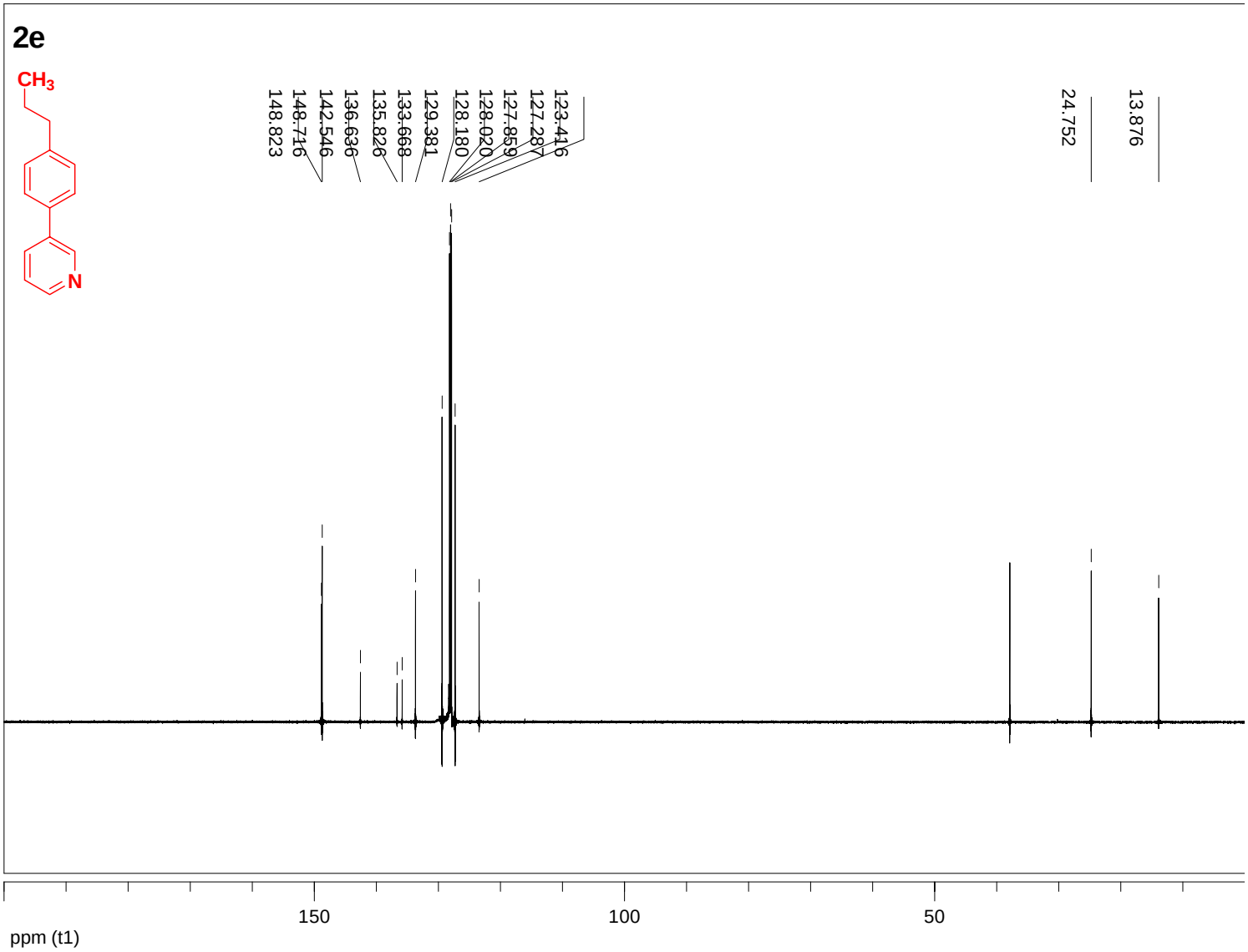
6.781
6.775
6.767
2.456
2.443
2.430
2.418
1.093
1.081
1.068
5.0
ppm (t1)

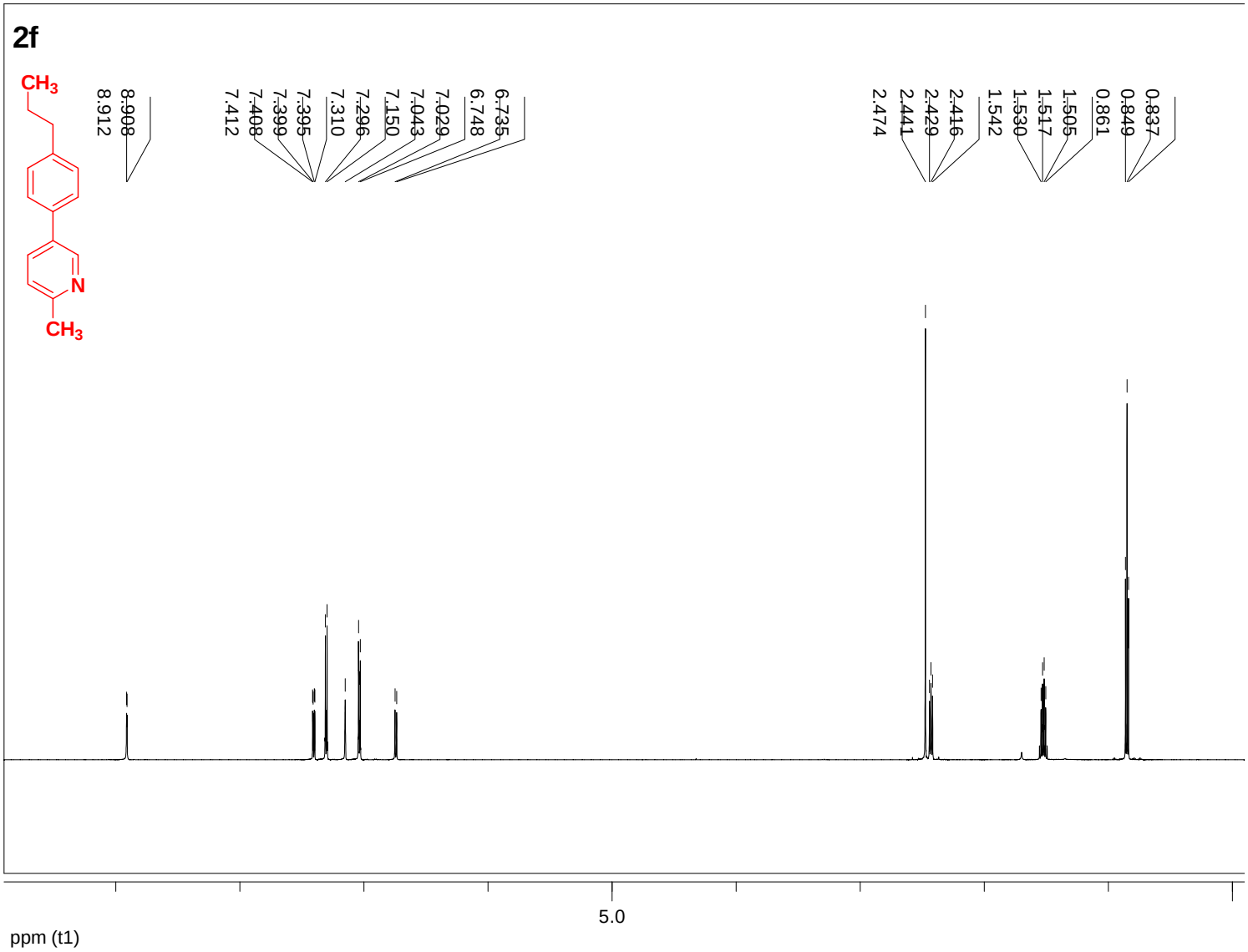

2c
H3C
N
148.806
148.699
144.123
136.658
135.792
133.678
128.768
128.178
128.020
127.857
127.369
123.431
28.788
15.699
150
100
50
ppm (t1)

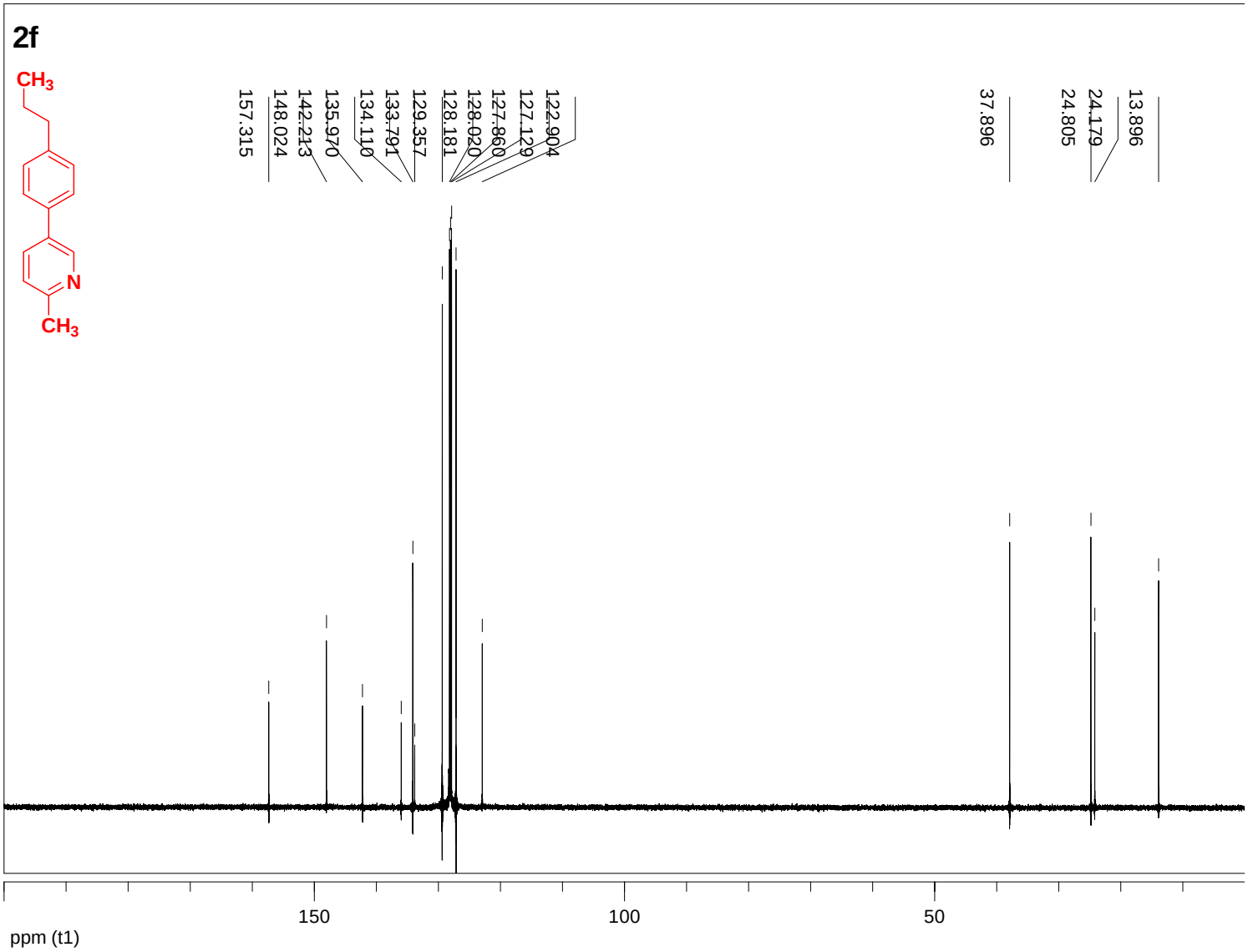

2d
H3C
N
CH3
8.908
7.407
7.403
7.394
7.390
7.302
7.288
7.150
7.041
7.028
6.748
6.734
2.474
1.092
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

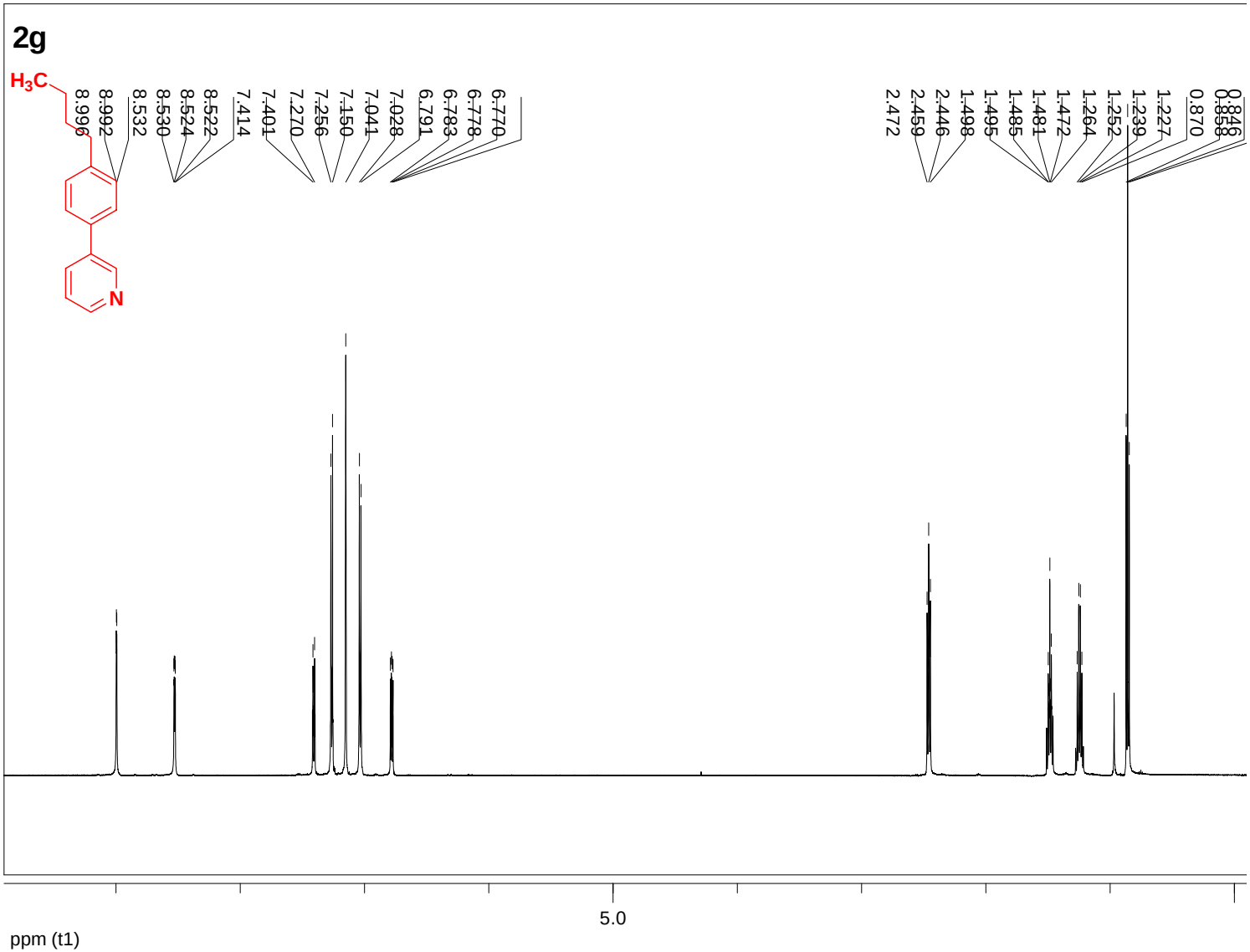

2d
H3C
N
CH3
157.306
148.019
143.790
135.934
134.110
133.798
128.733
128.180
128.020
127.859
127.202
122.902
28.802
24.172
15.775
150
100
50
ppm (t1)

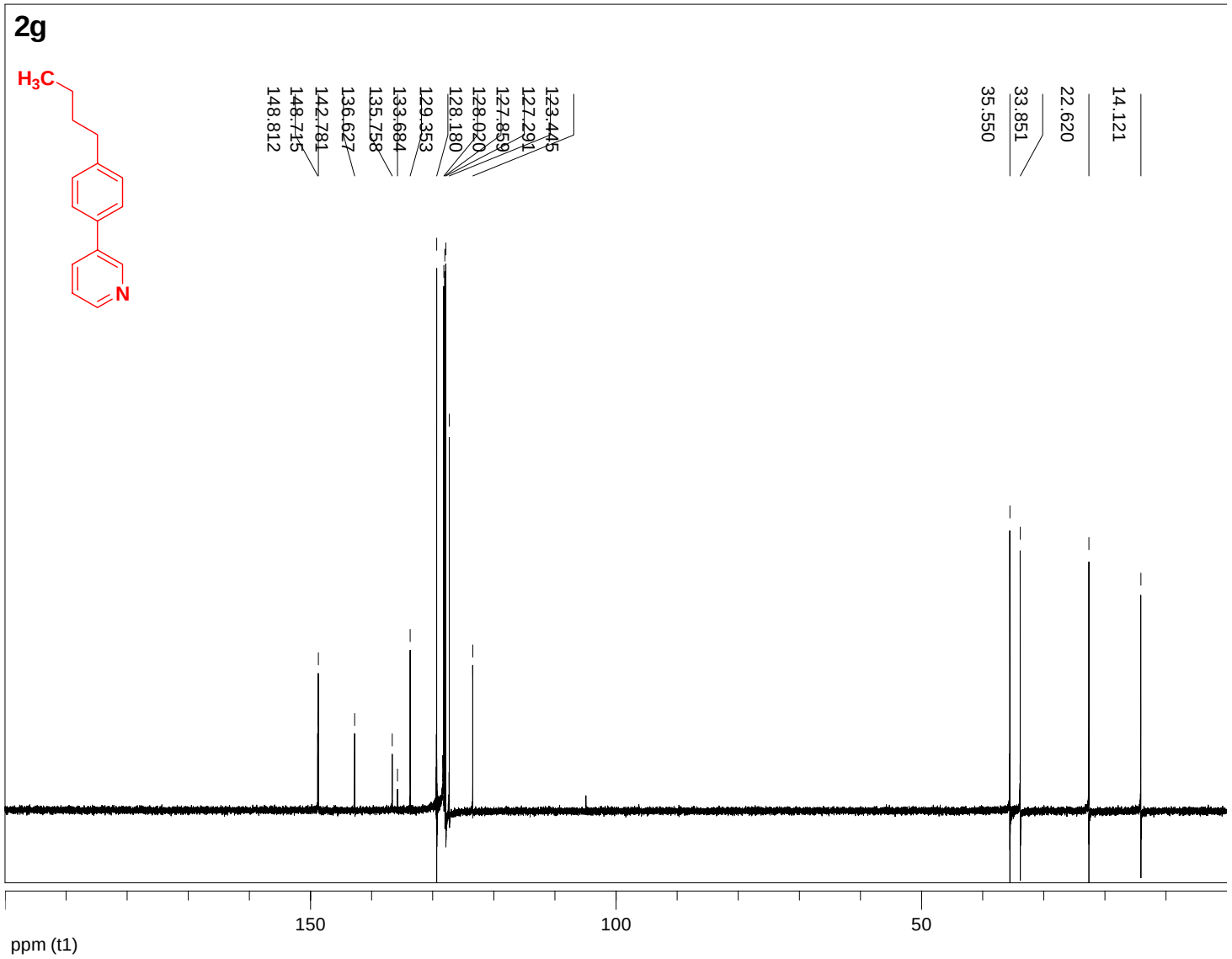

2e
CH3
N
8.987
8.983
8.528
8.525
8.520
8.517
7.412
7.409
7.405
7.399
7.396
7.392
7.262
7.248
7.150
7.023
7.010
6.791
6.789
6.783
6.778
6.770
2.429
2.416
2.404
1.531
1.518
1.505
1.493
0.854
0.841
0.829
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

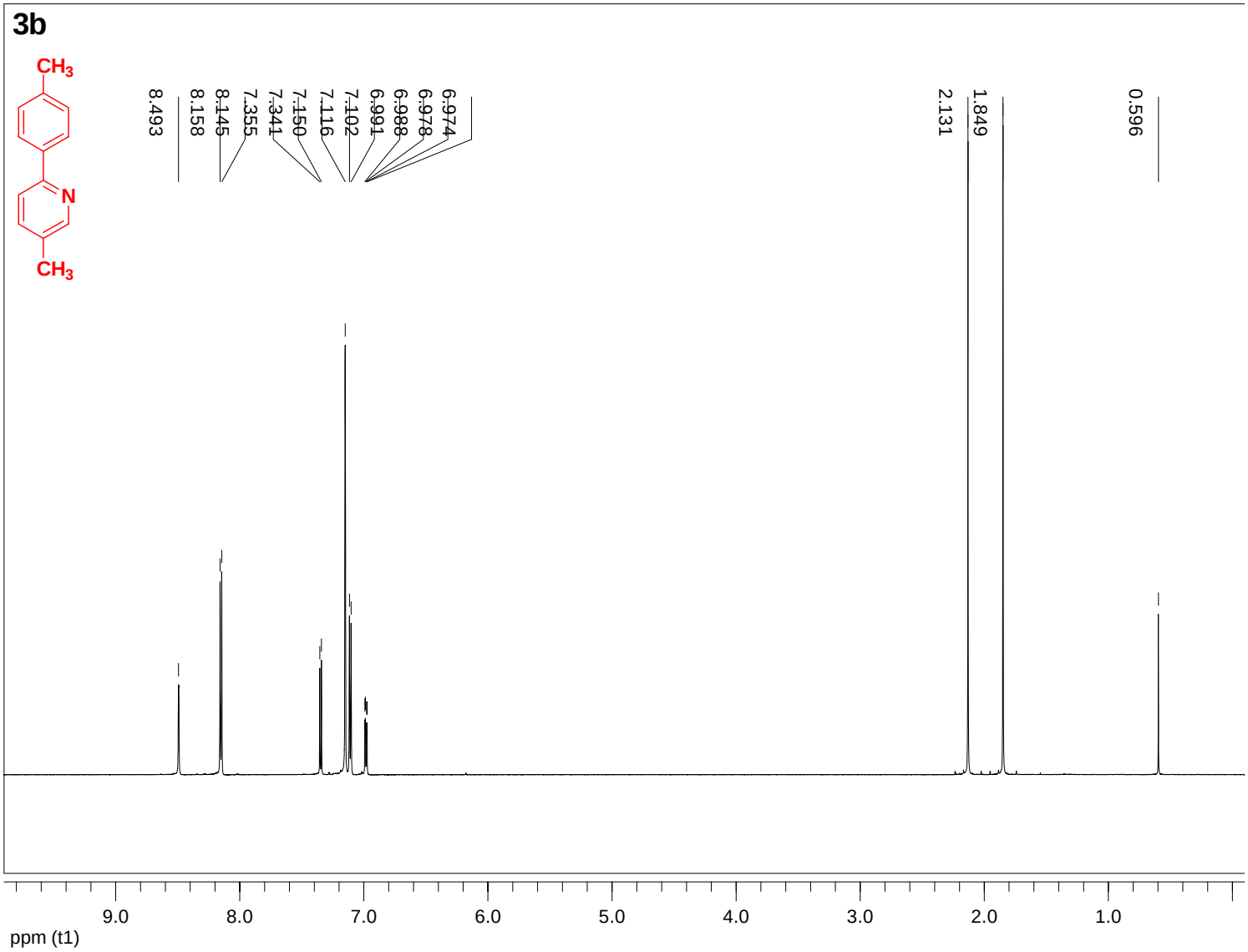

2e
CH3
N
148.823
148.716
142.546
136.636
135.826
133.668
129.381
128.180
128.020
127.859
127.287
123.416
24.752
13.876
150
100
50
ppm (t1)

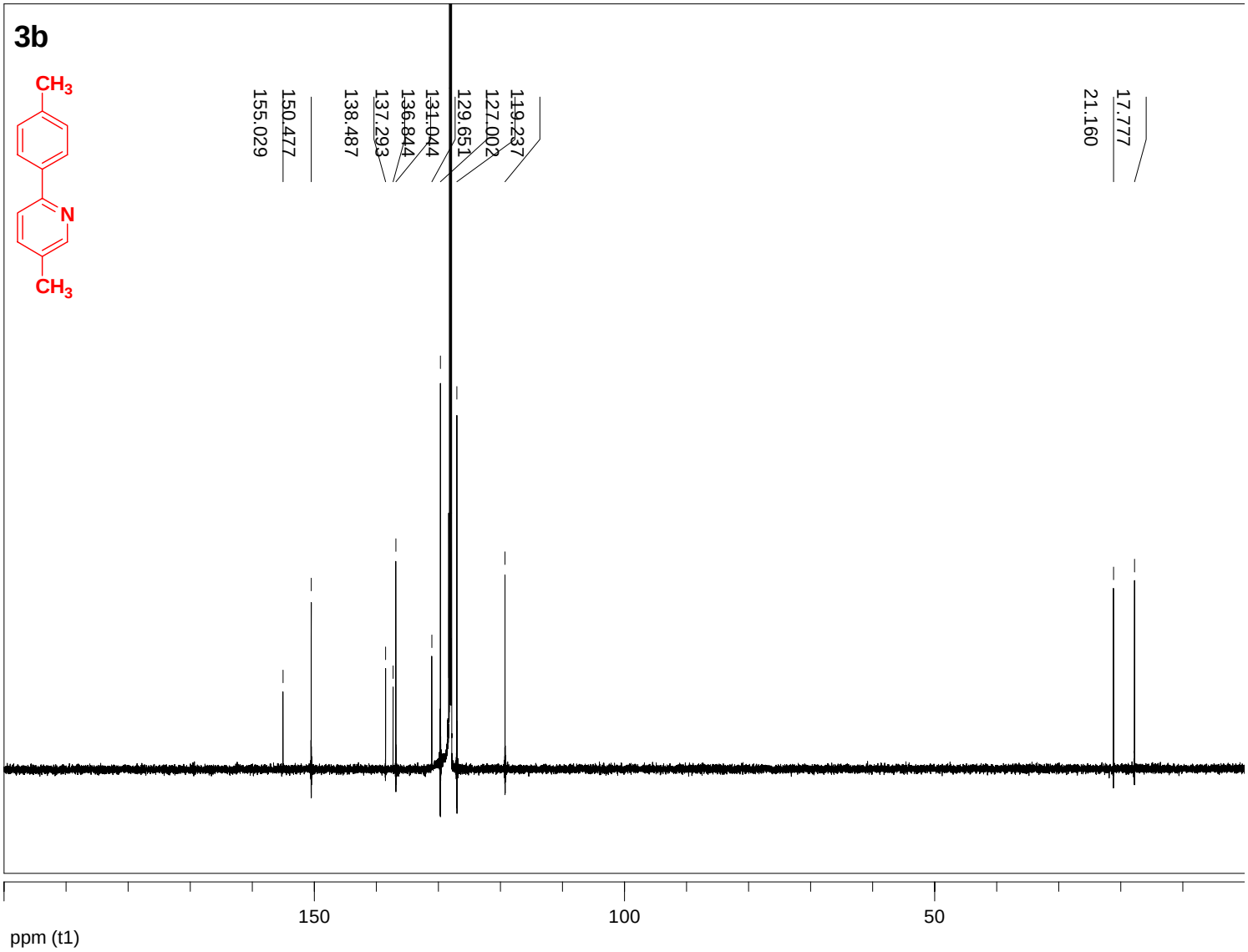

2f
CH3
N
CH3
8.908
8.912
7.412
7.408
7.399
7.395
7.310
7.296
7.150
7.043
7.029
6.748
6.735
2.474
2.441
2.429
2.416
1.542
1.530
1.517
1.505
0.861
0.849
0.837
5.0
ppm (t1)

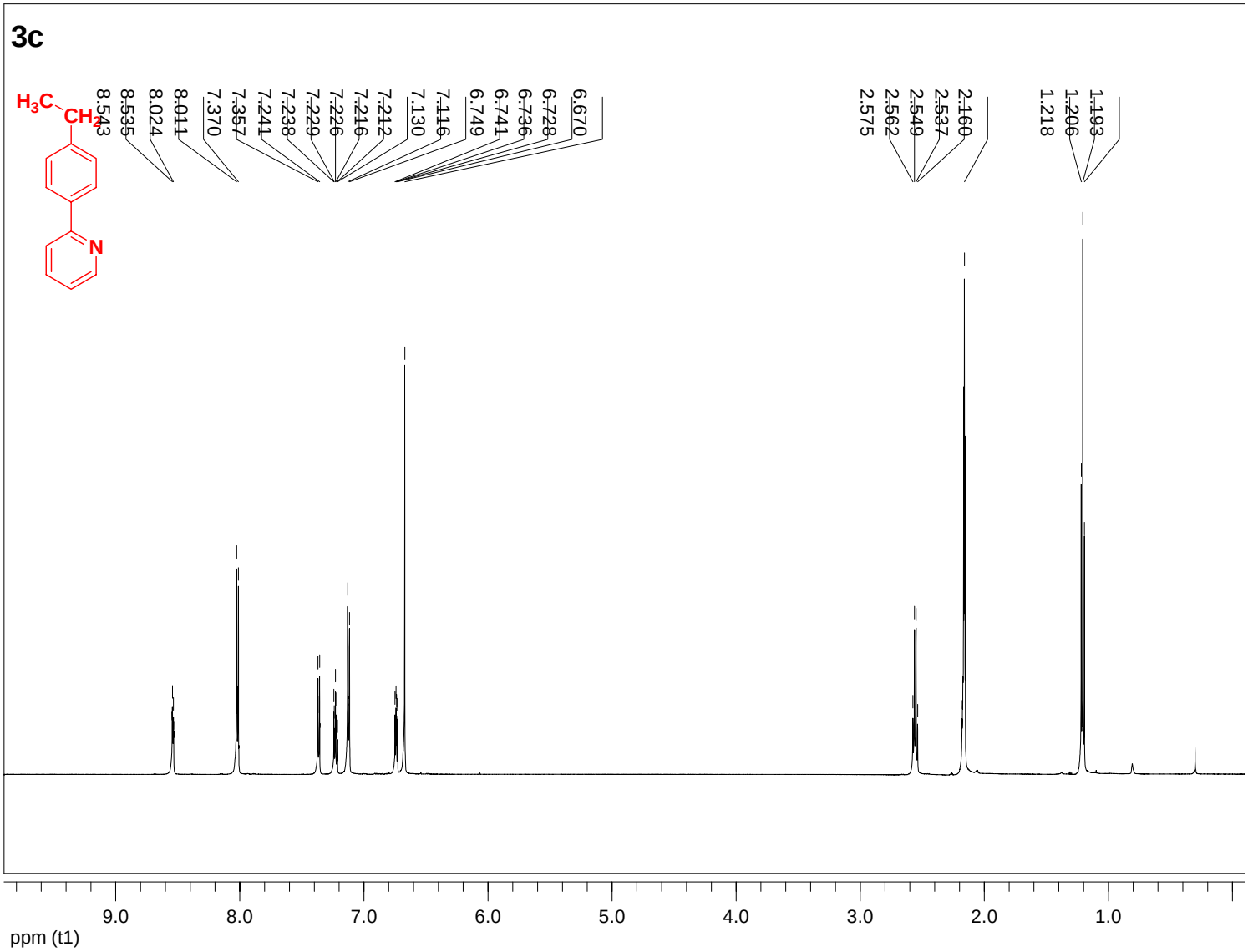

2f
CH3
N
CH3
157.315
148.024
142.213
135.970
134.110
133.791
129.357
128.181
128.020
127.860
127.129
122.904
37.896
24.805
24.179
13.896
150
100
50
ppm (t1)

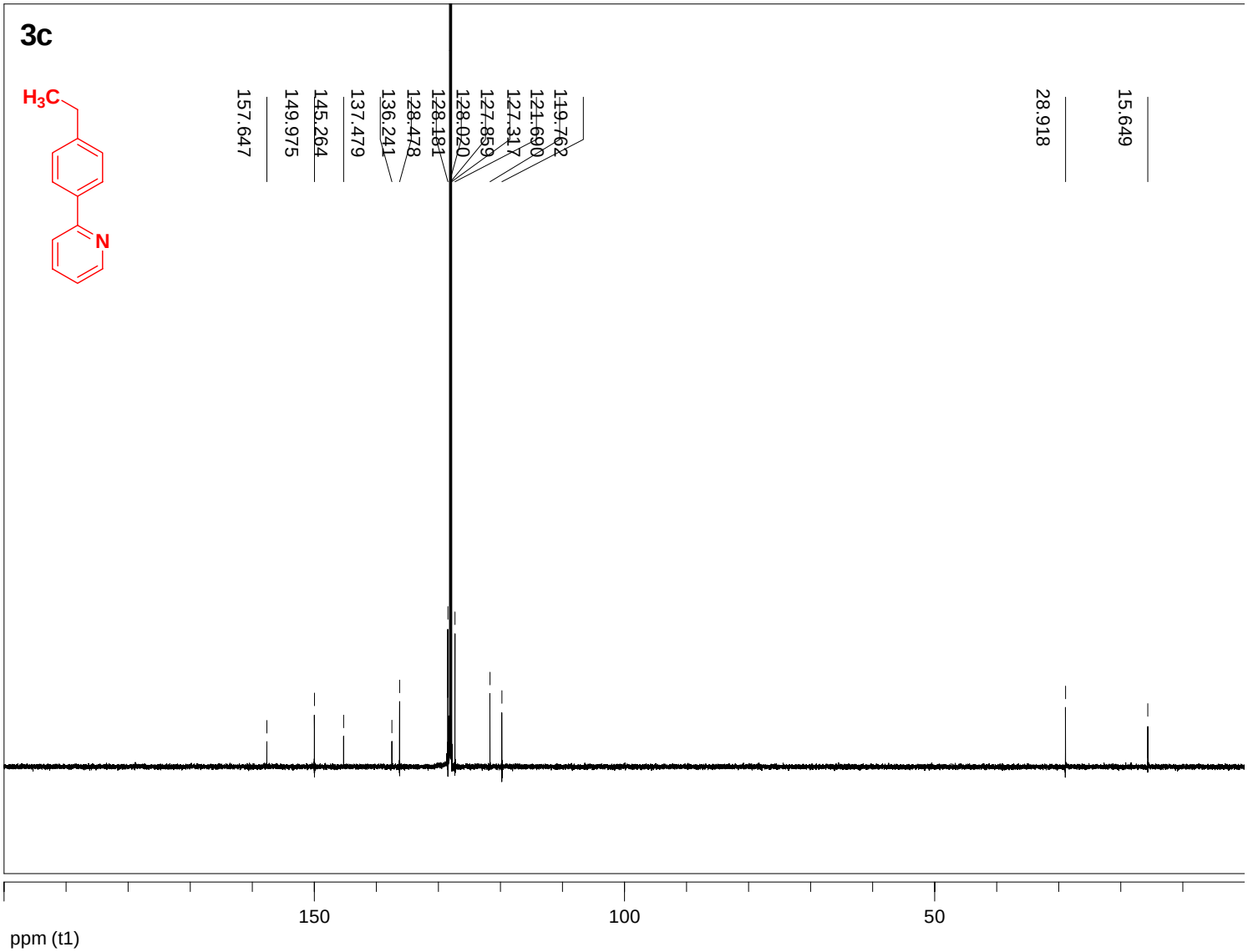

2g
H3C
N
8.996
8.992
8.532
8.530
8.524
8.522
7.414
7.401
7.270
7.256
7.150
7.041
7.028
6.791
6.783
6.778
6.770
2.472
2.459
2.446
1.498
1.495
1.485
1.481
1.472
1.264
1.252
1.239
1.227
0.870
0.858
0.846
5.0
ppm (t1)

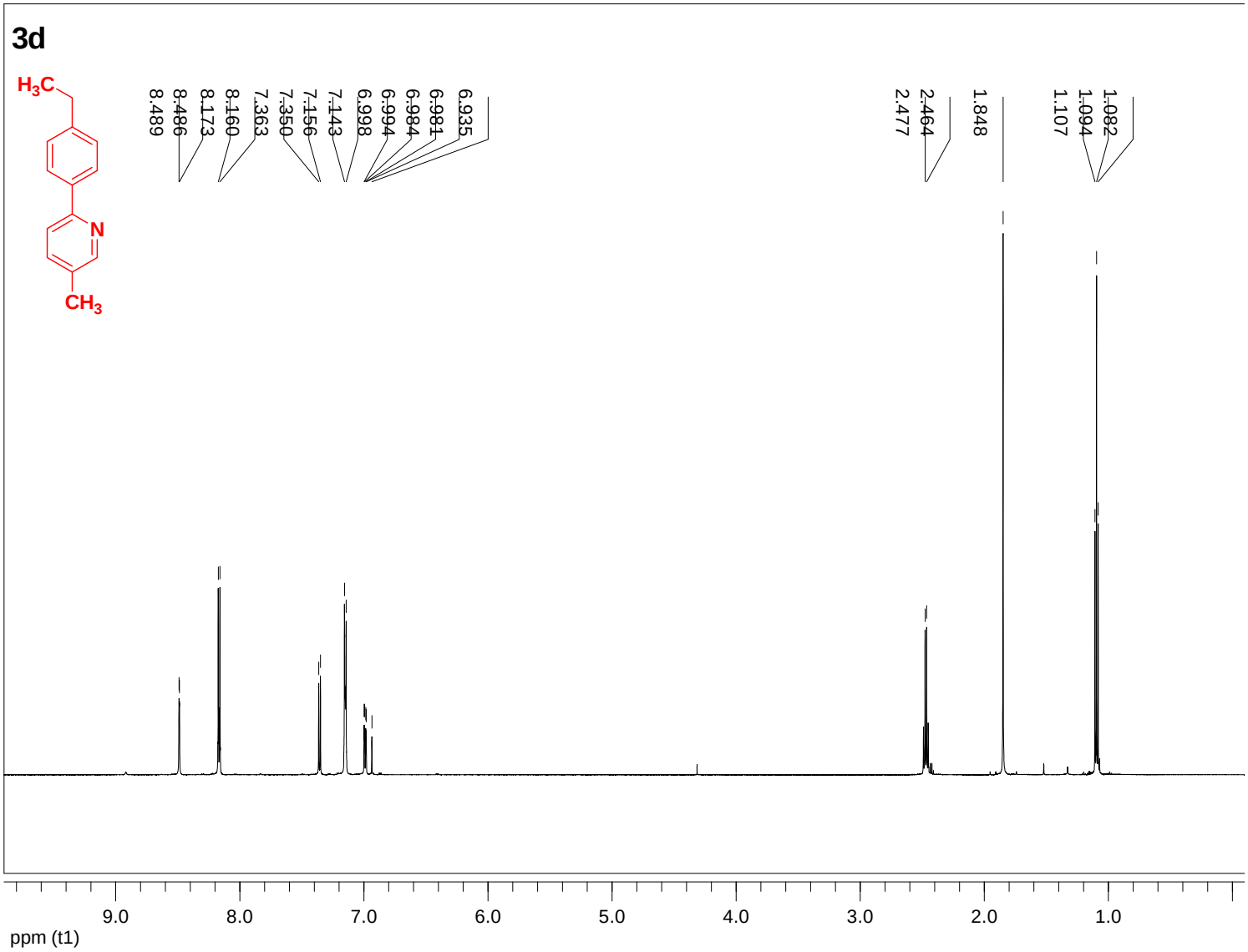

2g
H3C
N
148.812
148.715
142.781
136.627
135.758
133.684
129.353
128.180
128.020
127.859
127.291
123.445
35.550
33.851
22.620
14.121
150
100
50
ppm (t1)

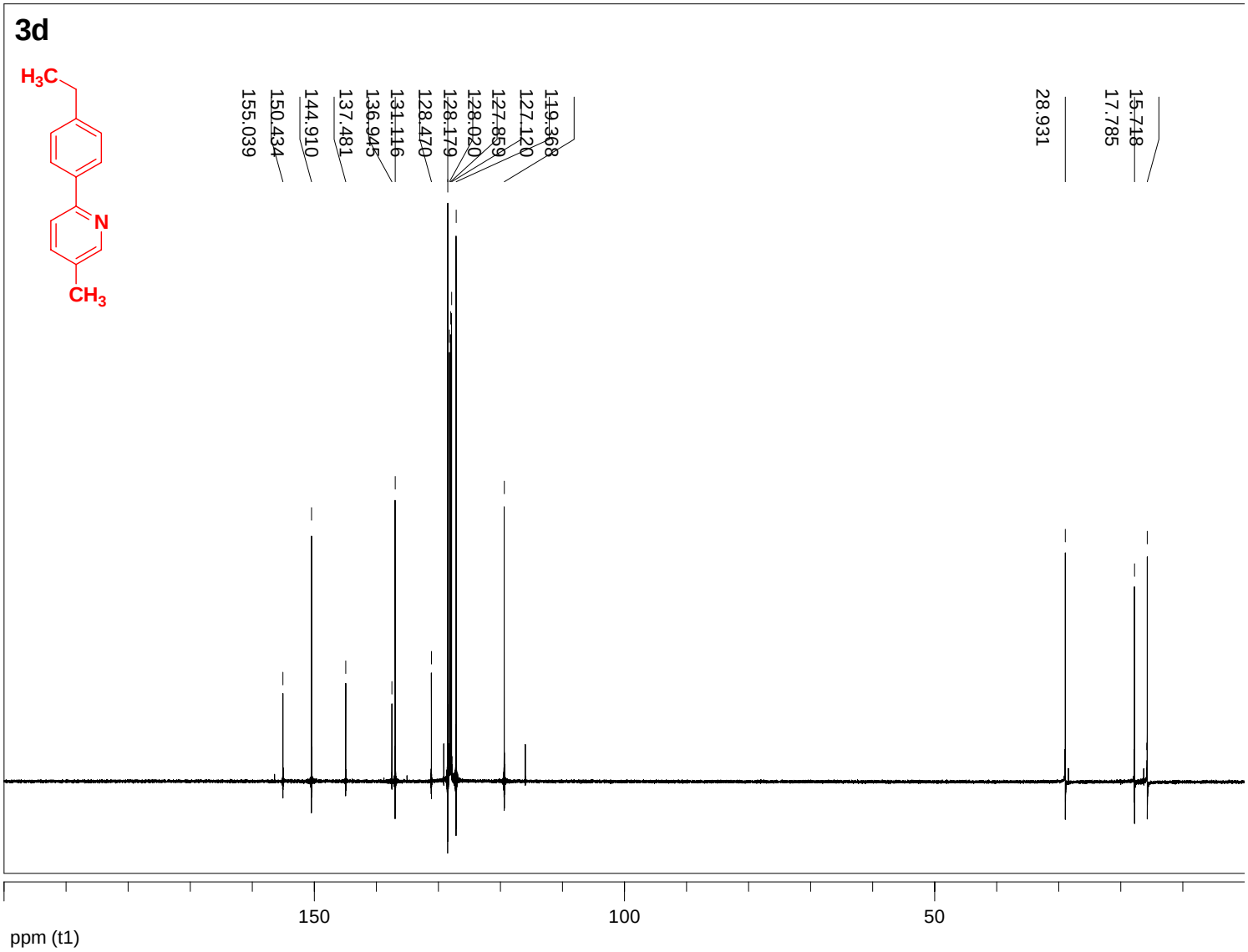

3b
CH3
N
CH3
8.493
8.158
8.145
7.355
7.341
7.150
7.116
7.102
6.991
6.988
6.978
6.974
2.131
1.849
0.596
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

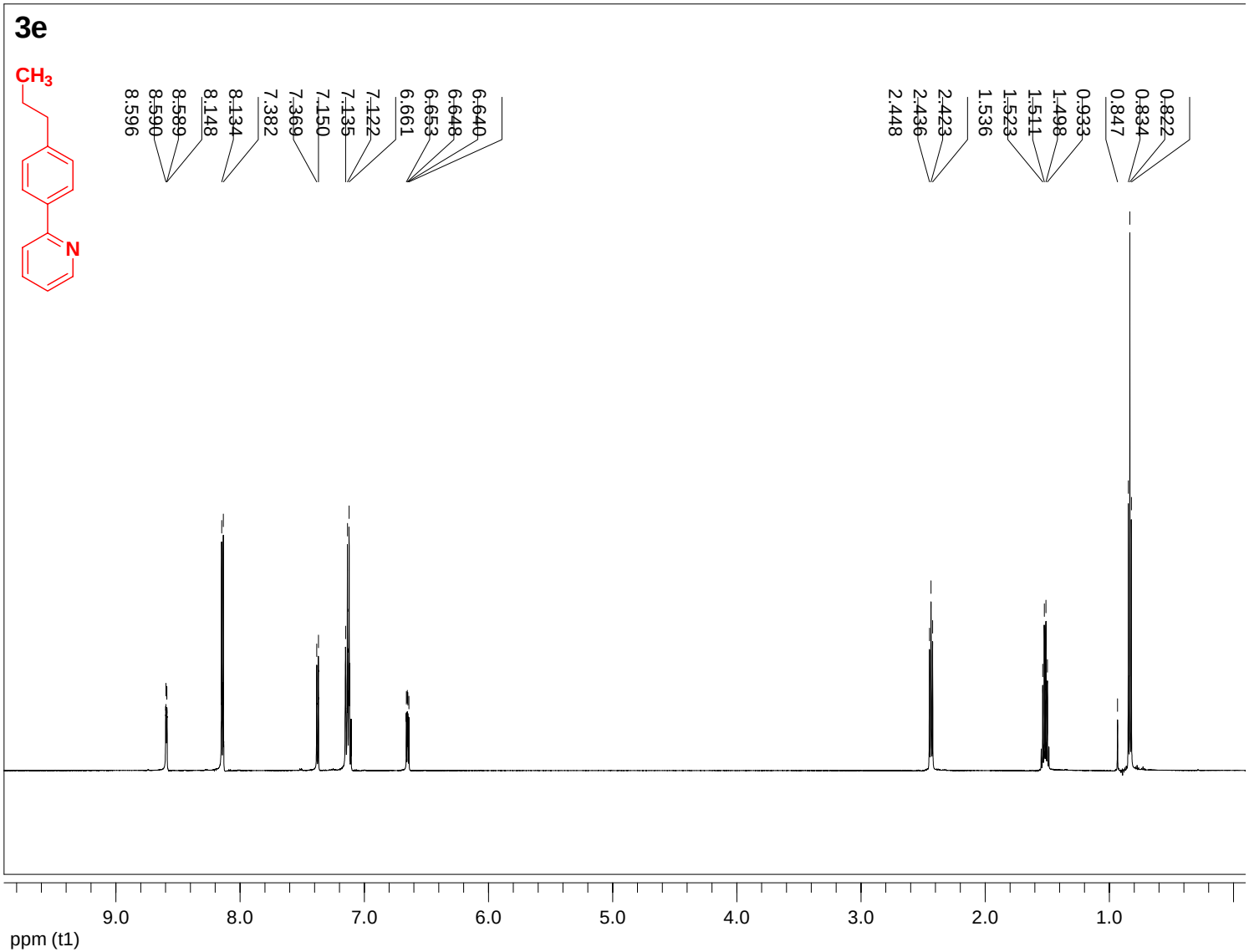

3b
CH3
N
CH3
155.029
150.477
138.487
137.293
136.844
131.044
129.651
127.002
119.237
21.160
17.777
150
100
50
ppm (t1)

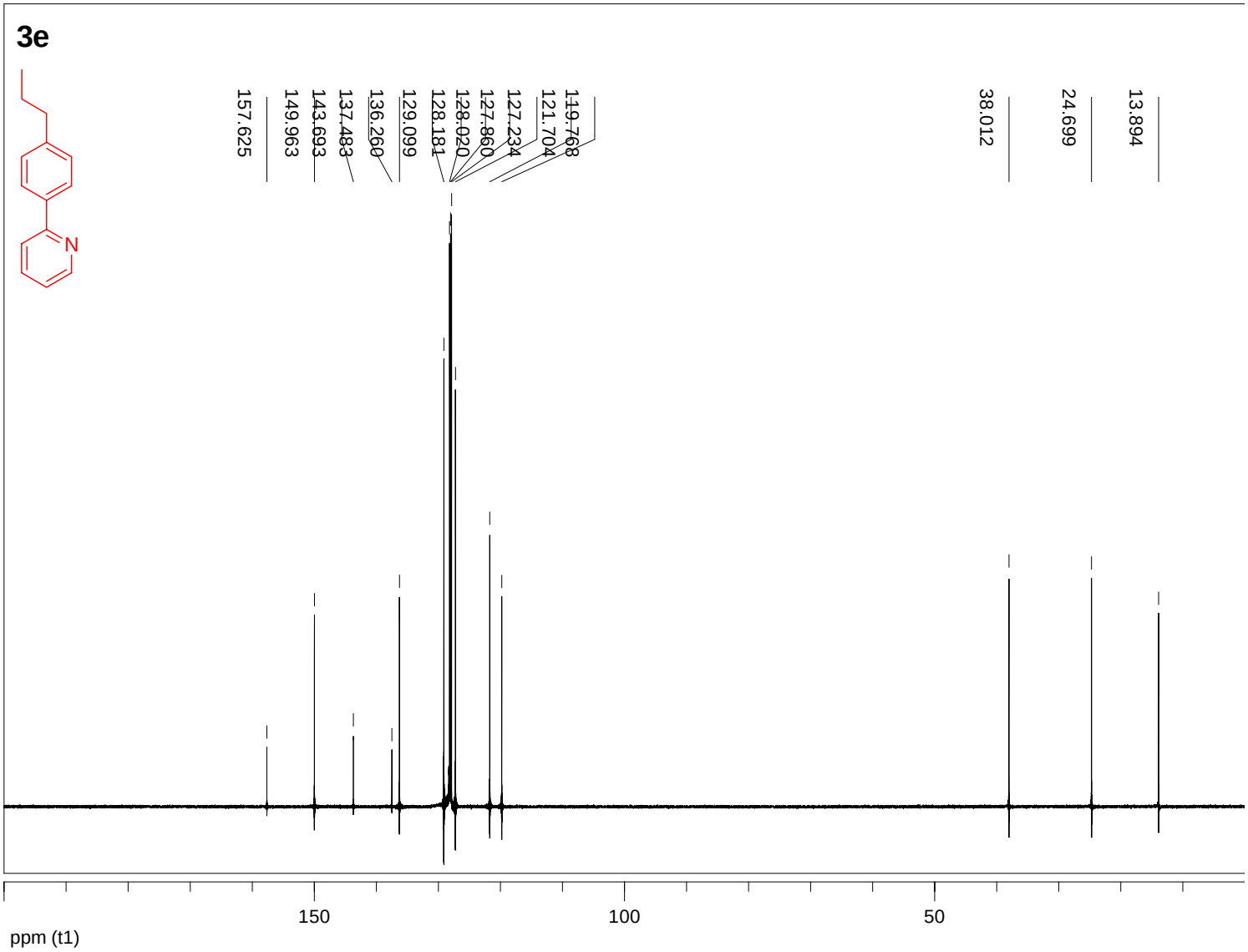

3c
H3C
CH2
N
8.543
8.535
8.024
8.011
7.370
7.357
7.241
7.238
7.229
7.226
7.216
7.212
7.130
7.116
6.749
6.741
6.736
6.728
6.670
2.575
2.562
2.549
2.537
2.160
1.218
1.206
1.193
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

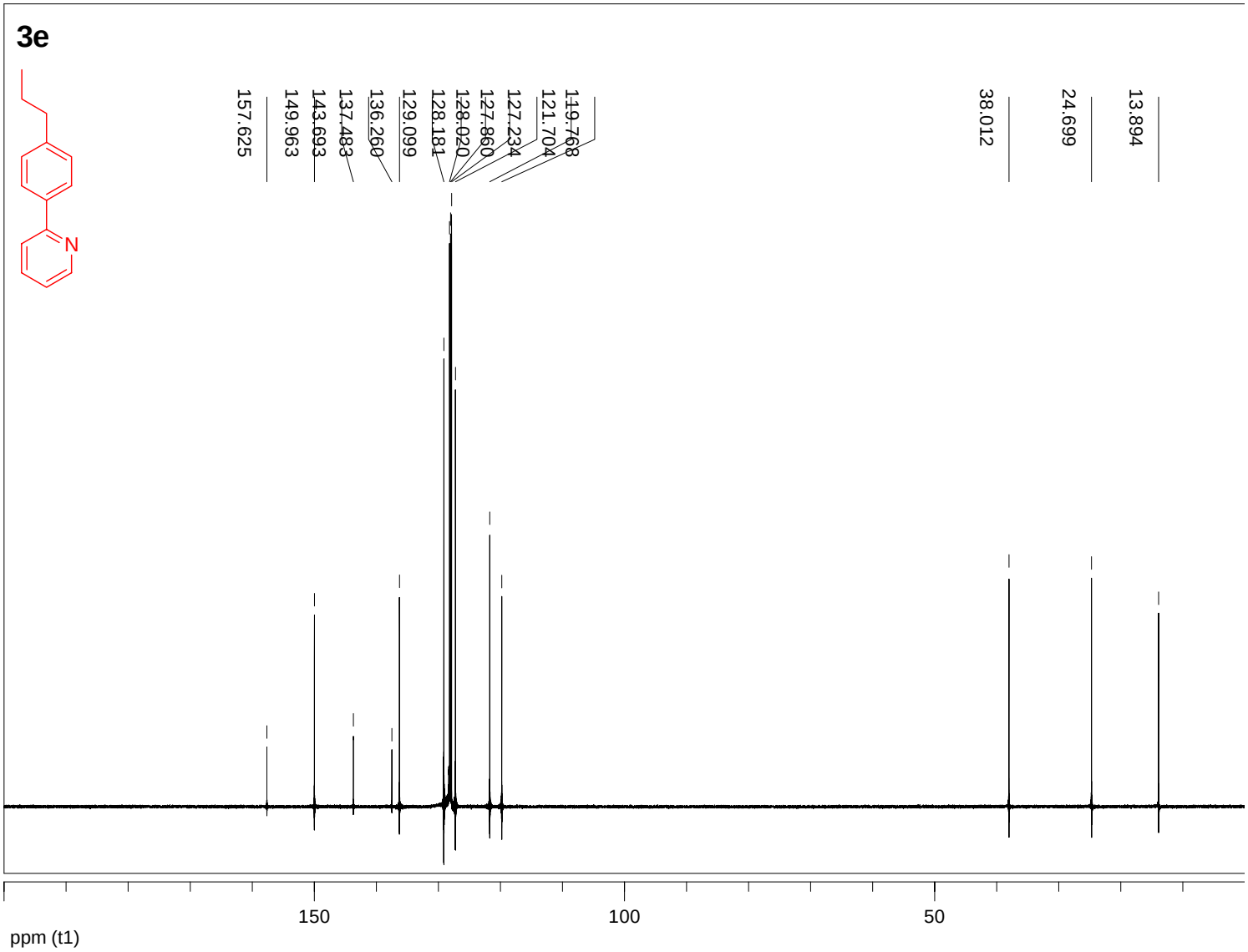

3c
H3C
N
157.647
149.975
145.264
137.479
136.241
128.478
128.181
128.020
127.859
127.317
121.690
119.762
28.918
15.649
150
100
50
ppm (t1)

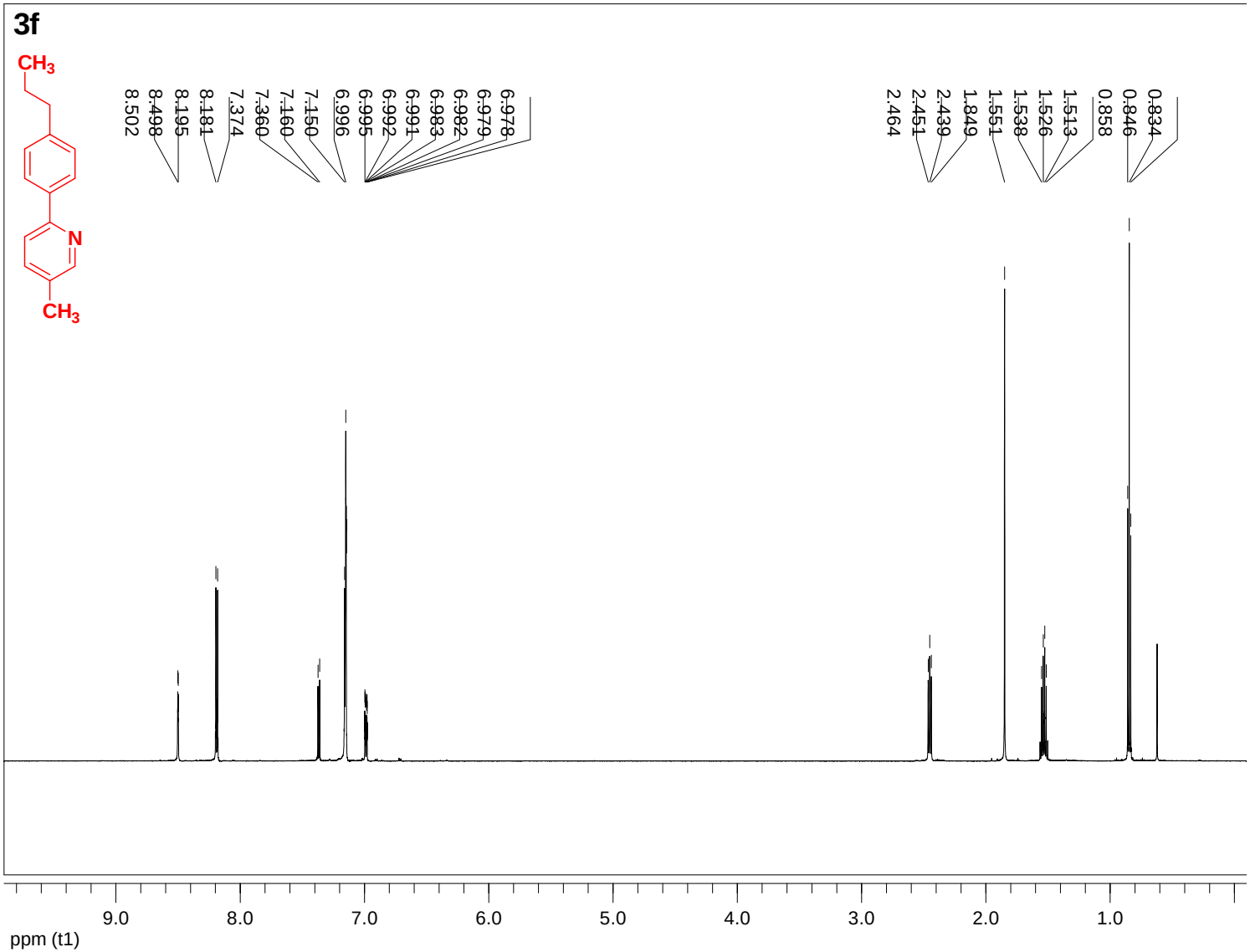

3d
H3C
N
CH3
8.489
8.486
8.173
8.160
7.363
7.350
7.156
7.143
6.998
6.994
6.984
6.981
6.935
2.477
2.464
1.848
1.107
1.094
1.082
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

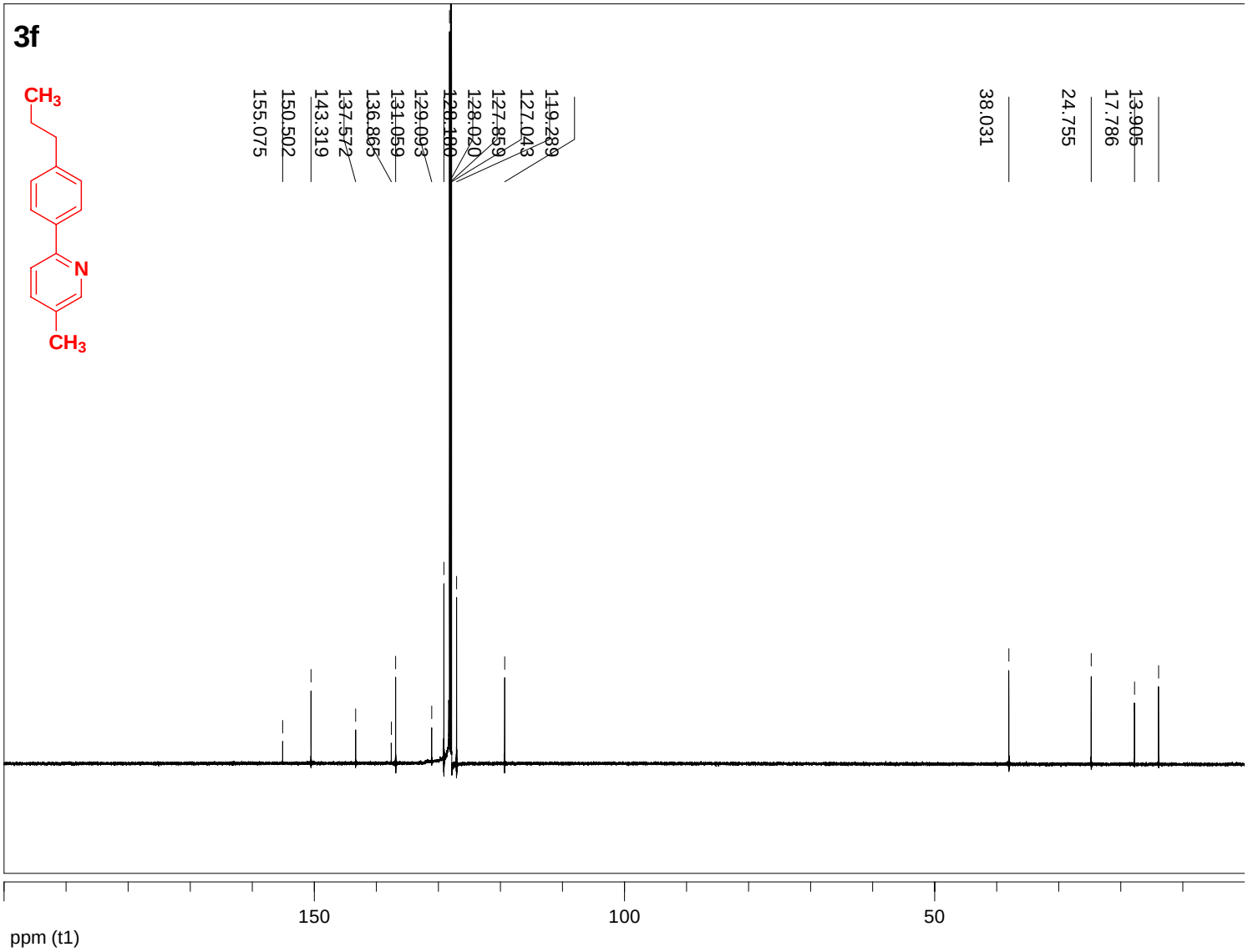

3d
H3C
N
CH3
155.039
150.434
144.910
137.481
136.945
131.116
128.470
128.179
128.020
127.859
127.120
119.368
28.931
17.785
15.718
150
100
50
ppm (t1)

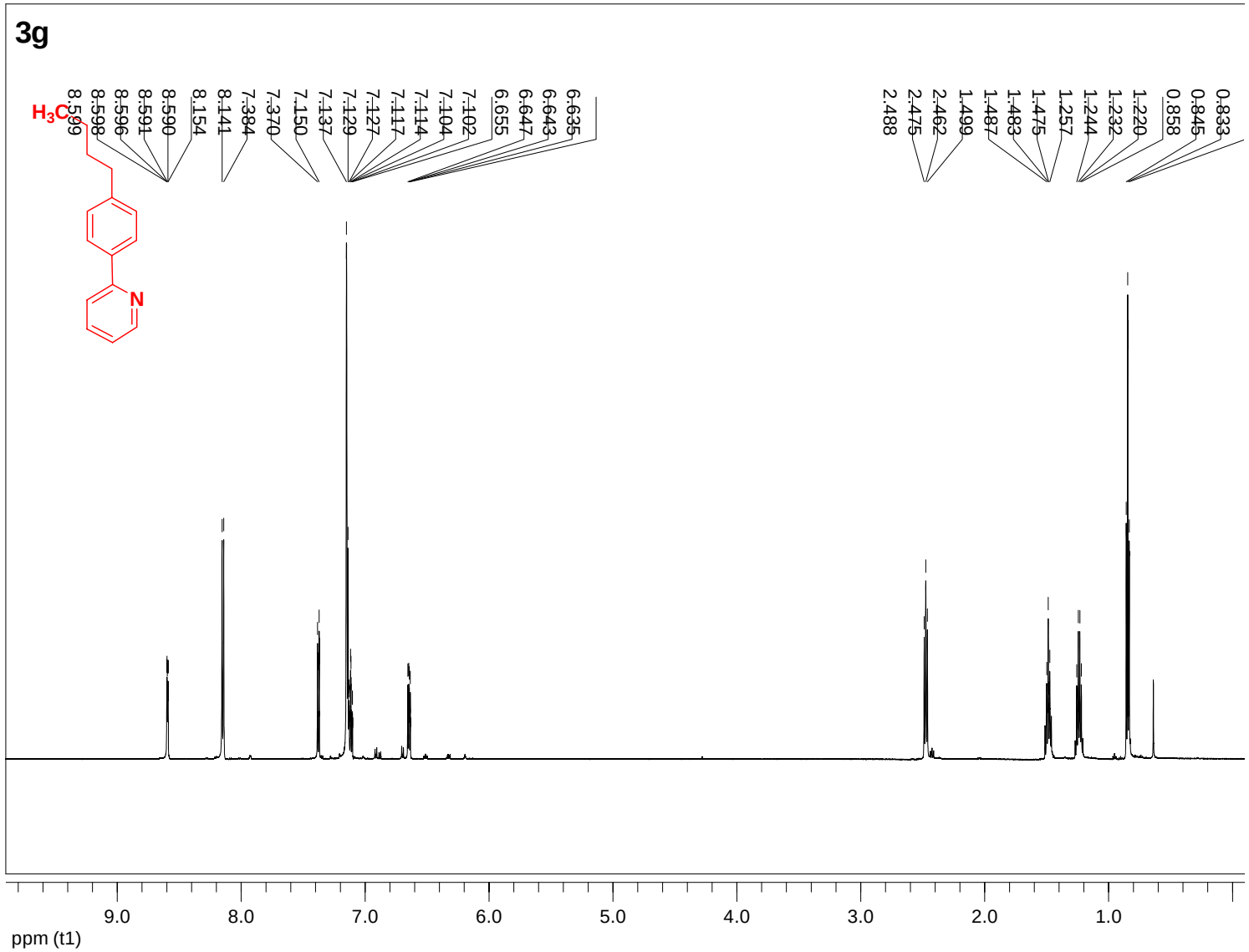

3e
CH3
N
8.596
8.590
8.589
8.148
8.134
7.382
7.369
7.150
7.135
7.122
6.661
6.653
6.648
6.640
2.448
2.436
2.423
1.536
1.523
1.511
1.498
0.933
0.847
0.834
0.822
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

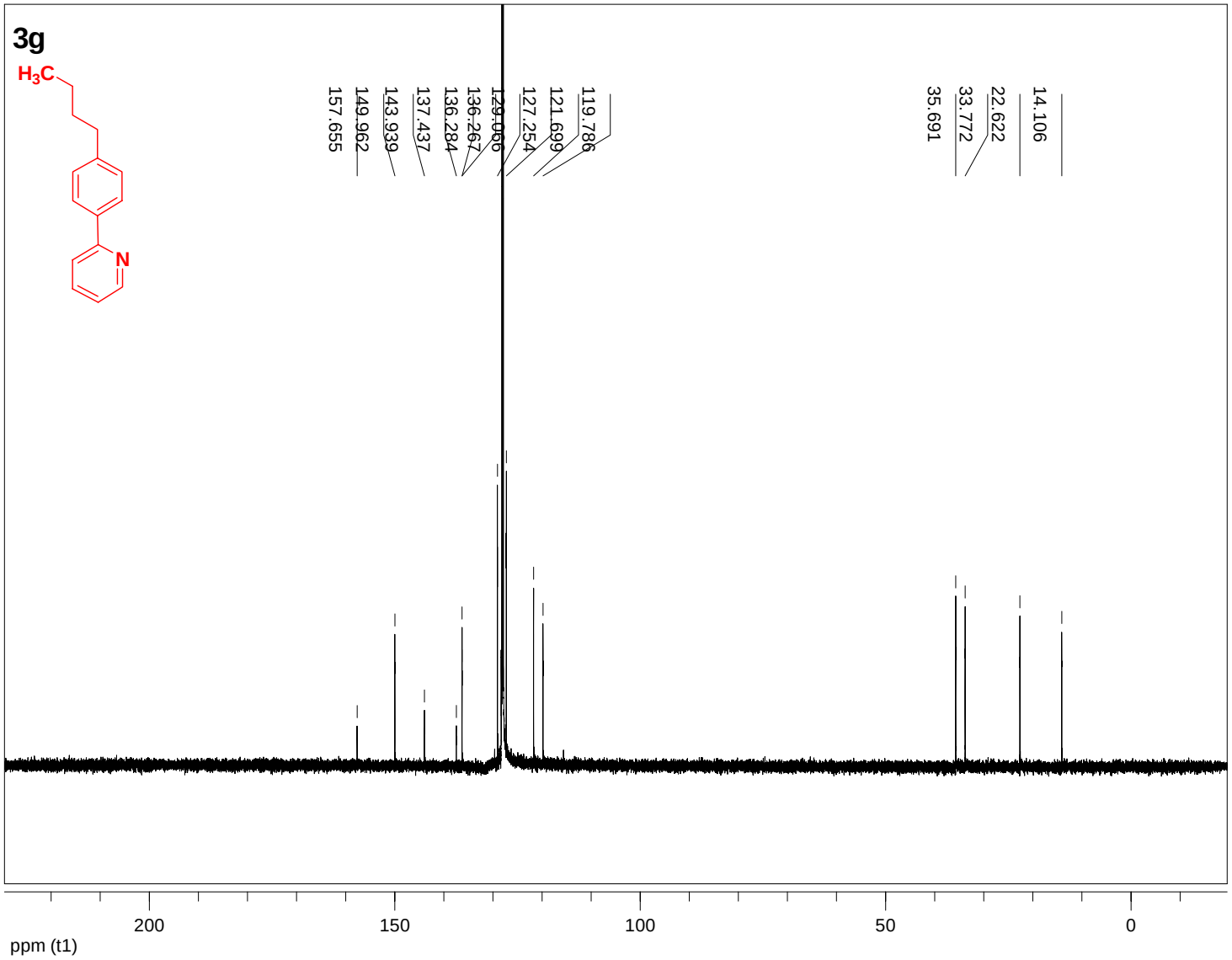

3e
N
157.625
149.963
143.693
137.483
136.260
129.099
128.181
128.020
127.860
127.234
121.704
119.768
38.012
24.699
13.894
150
100
50
ppm (t1)

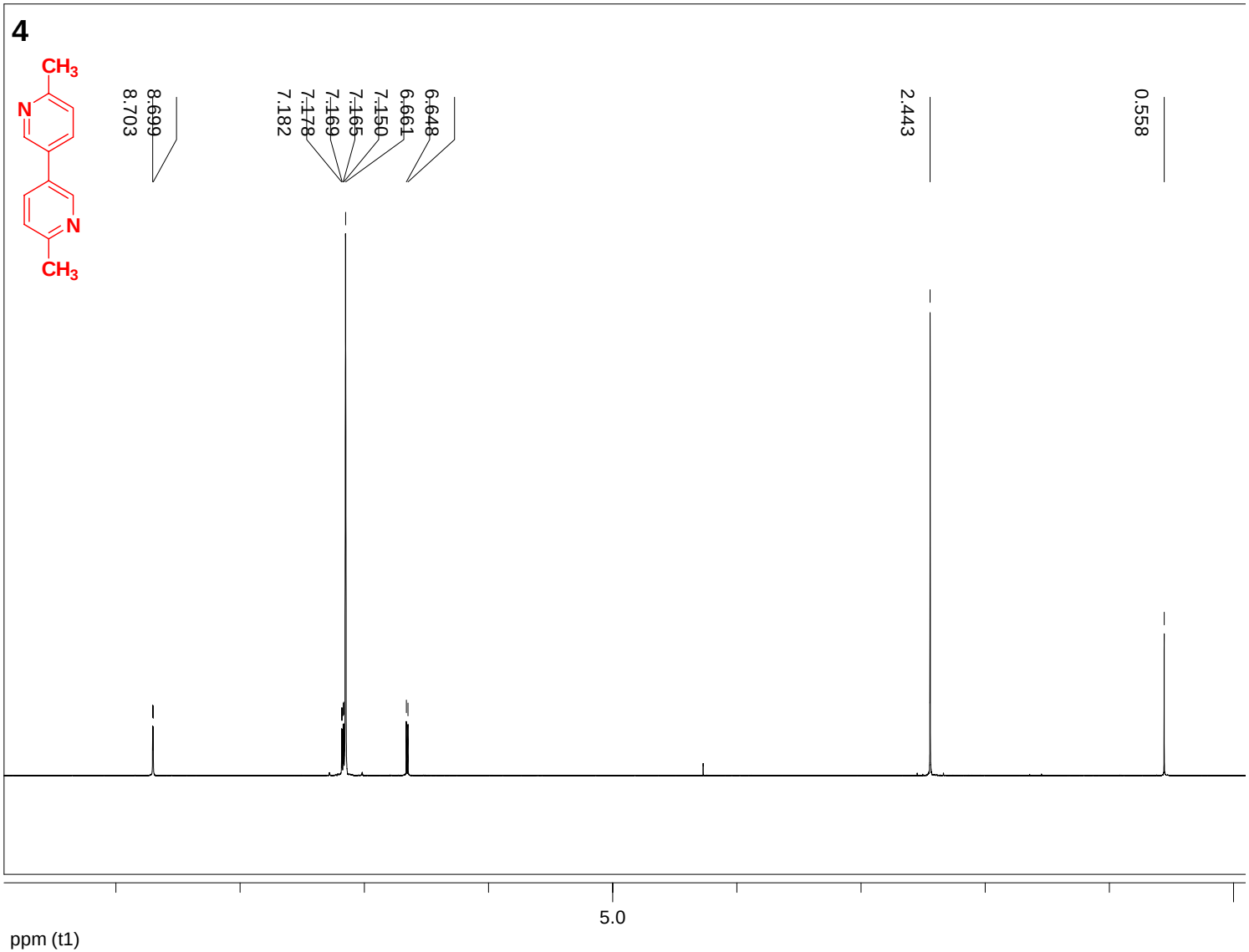

3e
N
157.625
149.963
143.693
137.483
136.260
129.099
128.181
128.020
127.860
127.234
121.704
119.768
38.012
24.699
13.894
150
100
50
ppm (t1)

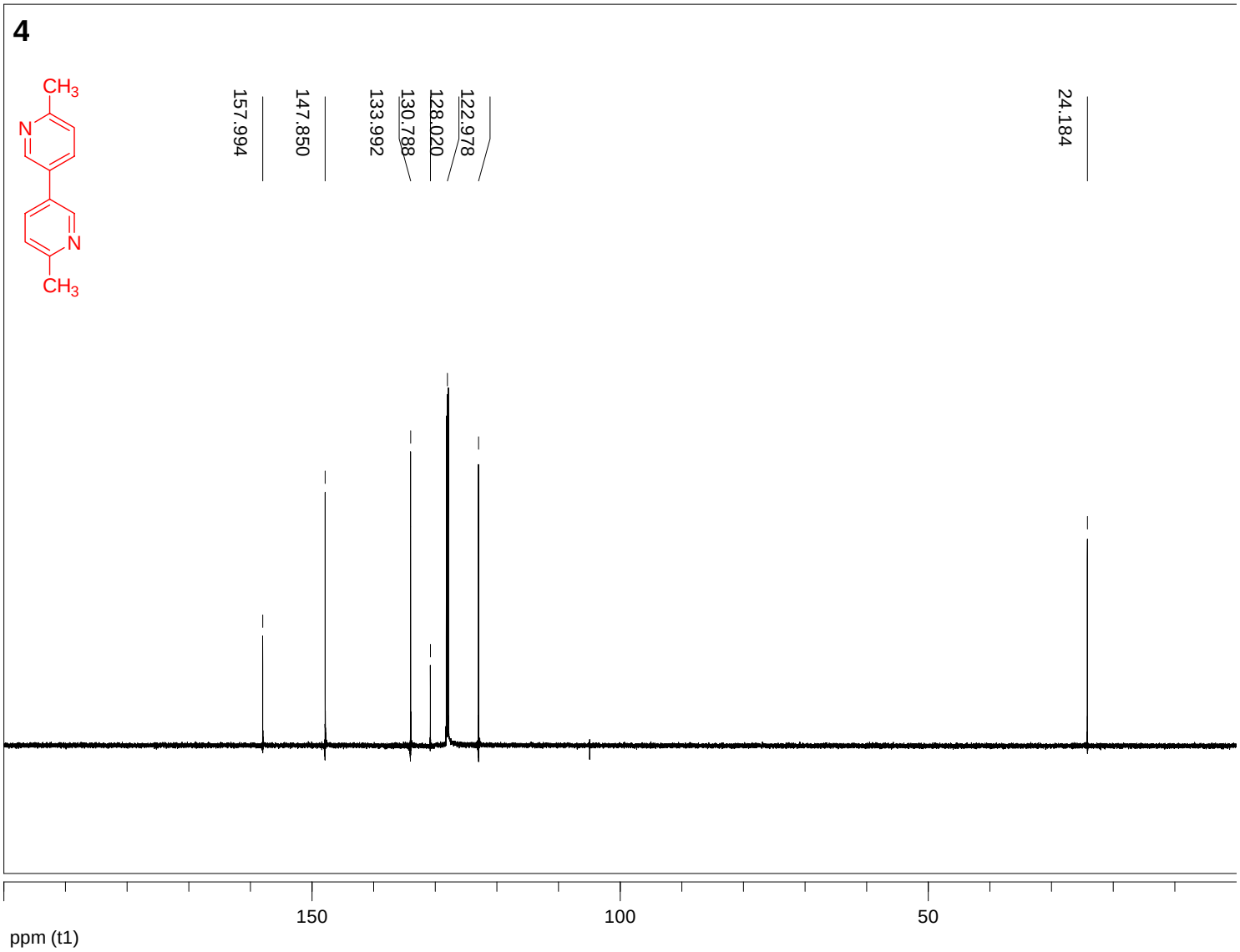

3f
CH3
N
CH3
8.502
8.498
8.195
8.181
7.374
7.360
7.160
7.150
6.996
6.995
6.992
6.991
6.983
6.982
6.979
6.978
2.464
2.451
2.439
1.849
1.551
1.538
1.526
1.513
0.858
0.846
0.834
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

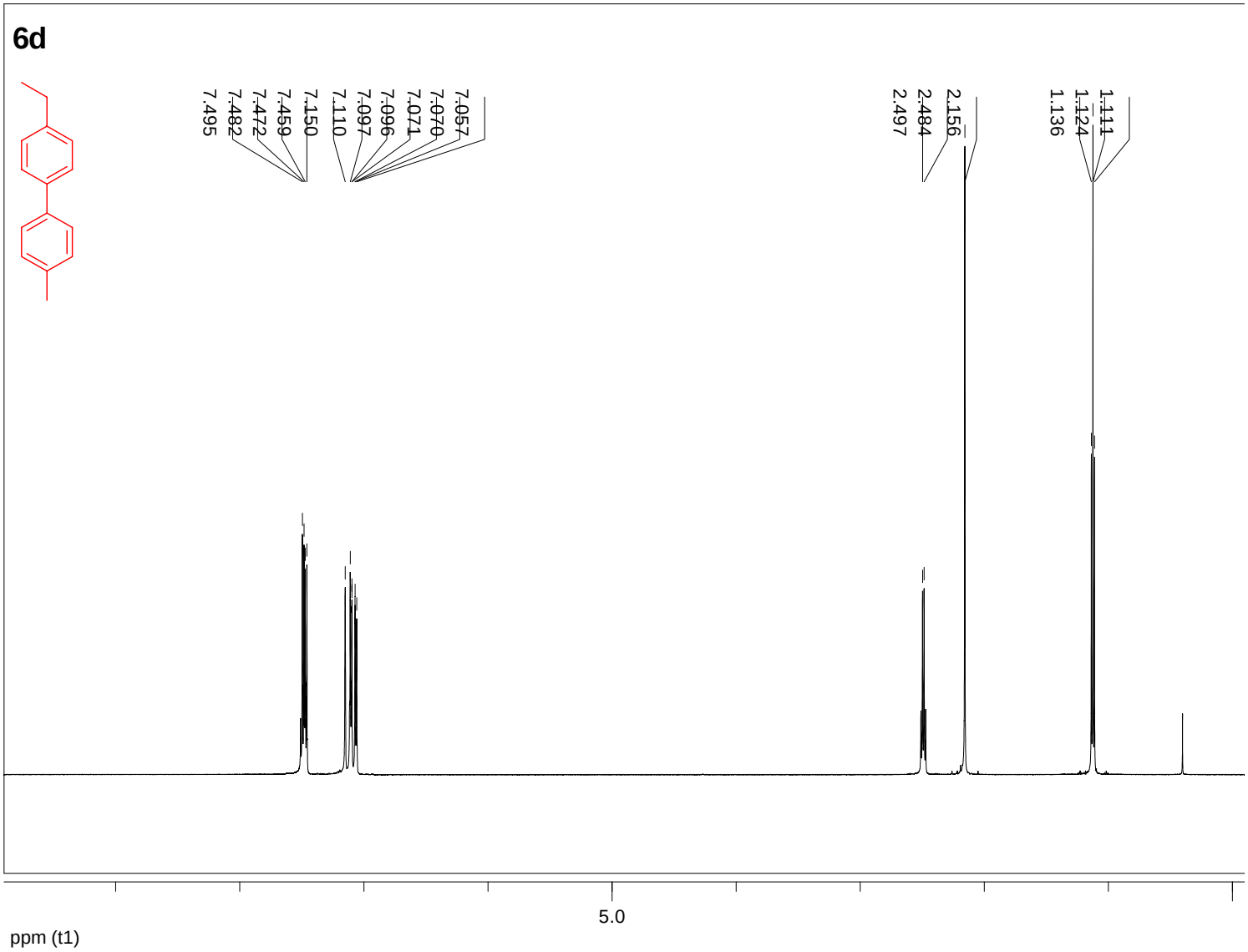

3f
CH3
N
CH3
155.075
150.502
143.319
137.572
136.865
131.059
129.093
128.180
128.020
127.859
127.043
119.289
38.031
24.755
17.786
13.905
150
100
50
ppm (t1)

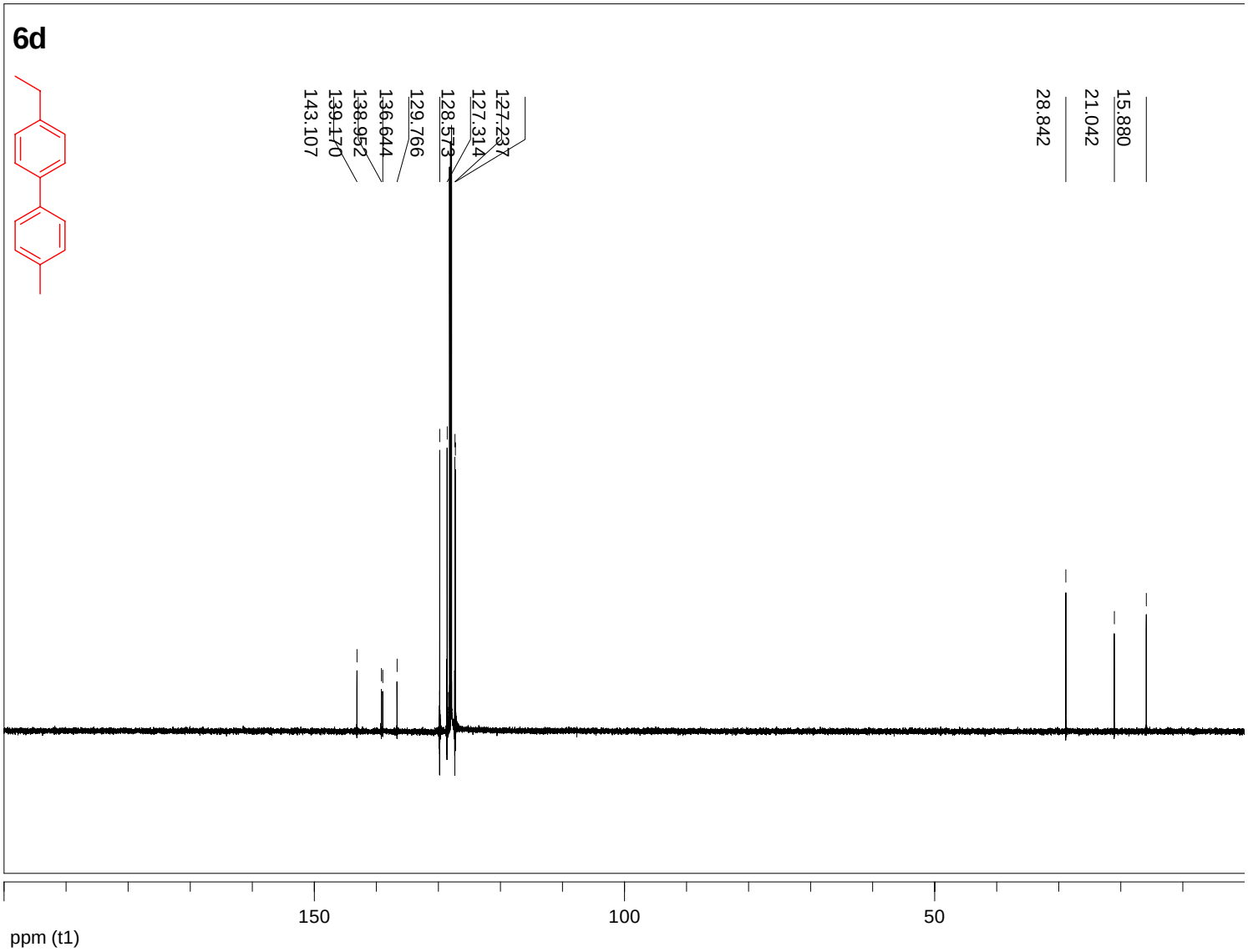

3g
8.599
8.598
8.596
8.591
8.590
8.154
8.141
7.384
7.370
7.150
7.137
7.129
7.127
7.117
7.114
7.104
7.102
6.955
6.647
6.643
6.635
2.488
2.475
2.462
1.499
1.487
1.483
1.475
1.257
1.244
1.232
1.220
0.858
0.845
0.833
H3C
N
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

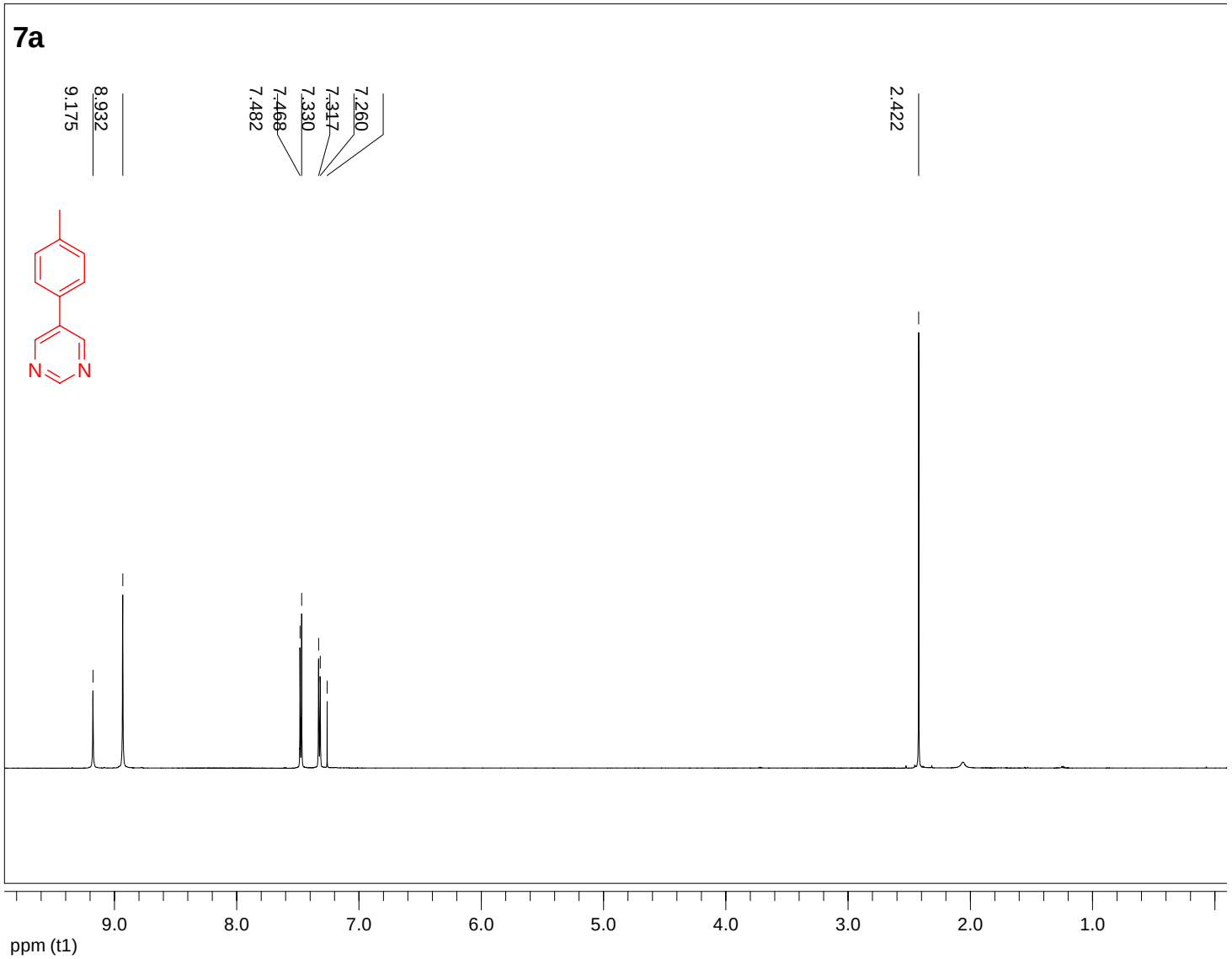

3g
H3C
N
157.655
149.962
143.939
137.437
136.284
136.267
129.066
127.254
121.699
119.786
35.691
33.772
22.622
14.106
200
150
100
50
0
ppm (t1)

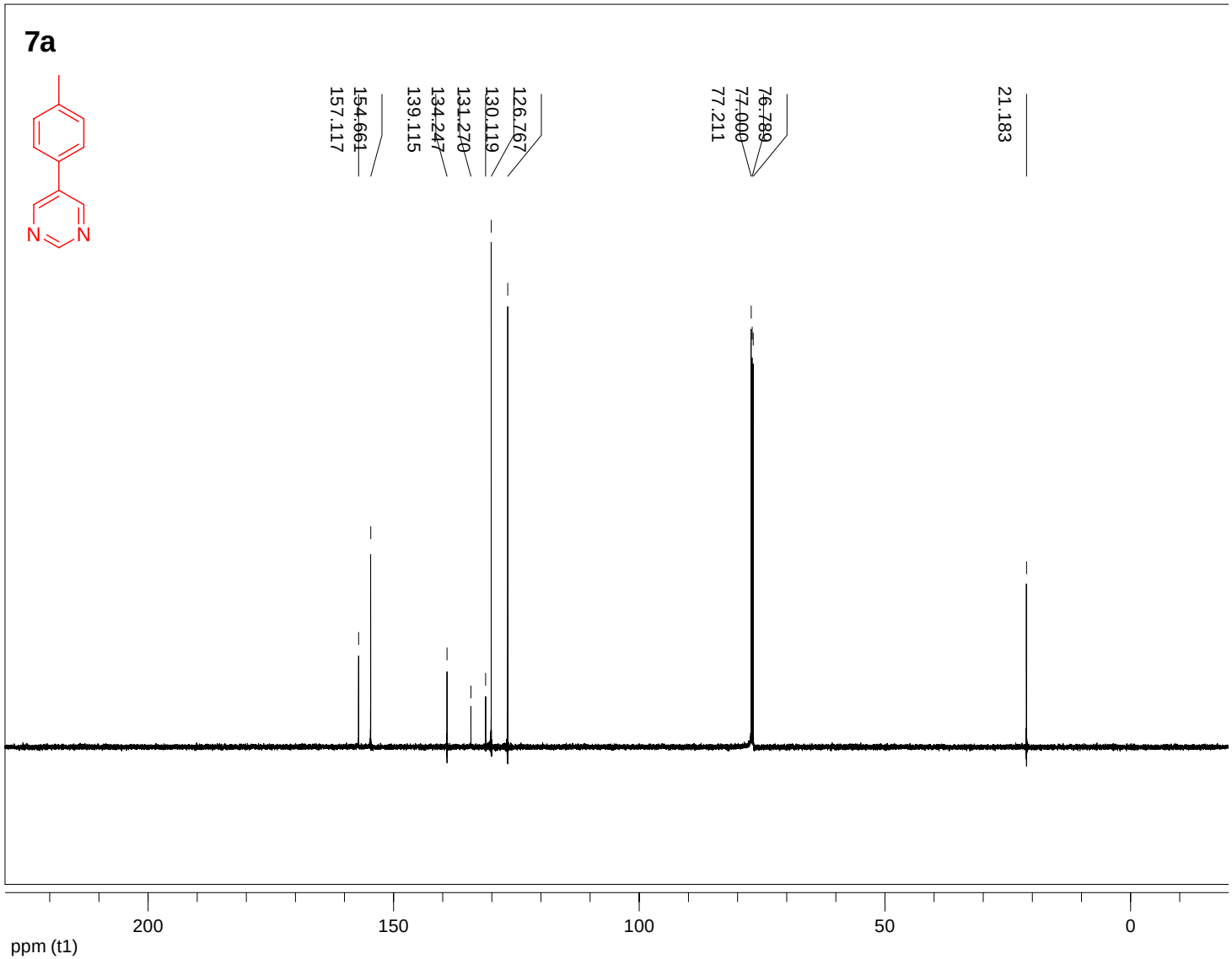

4
CH3
N
N
CH3
8.703
8.699
7.182
7.178
7.169
7.165
7.150
6.661
6.648
2.443
0.558
5.0
ppm (t1)

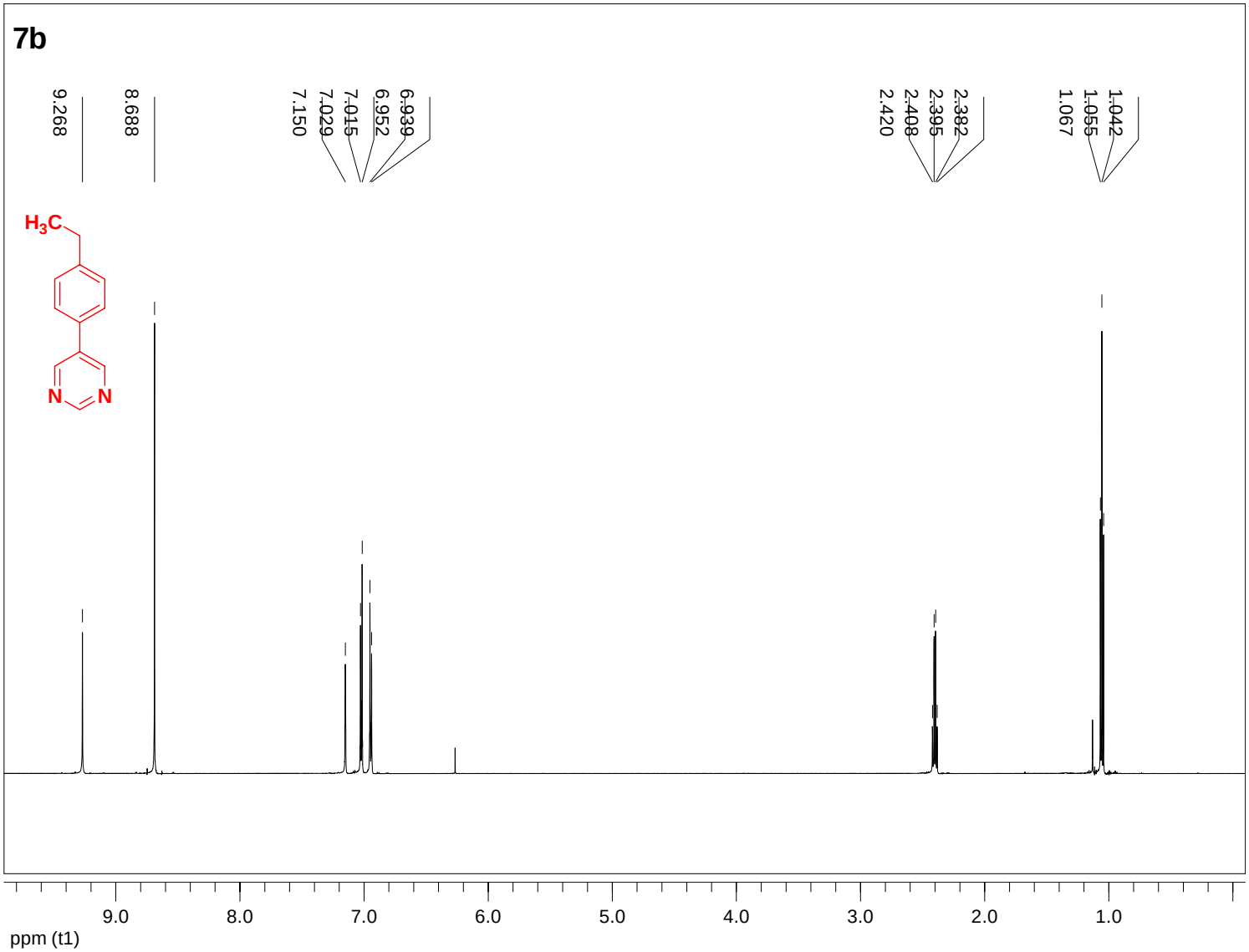

4
CH3
N
N
CH3
157.994
147.850
133.992
130.788
128.020
122.978
24.184
150
100
50
ppm (t1)

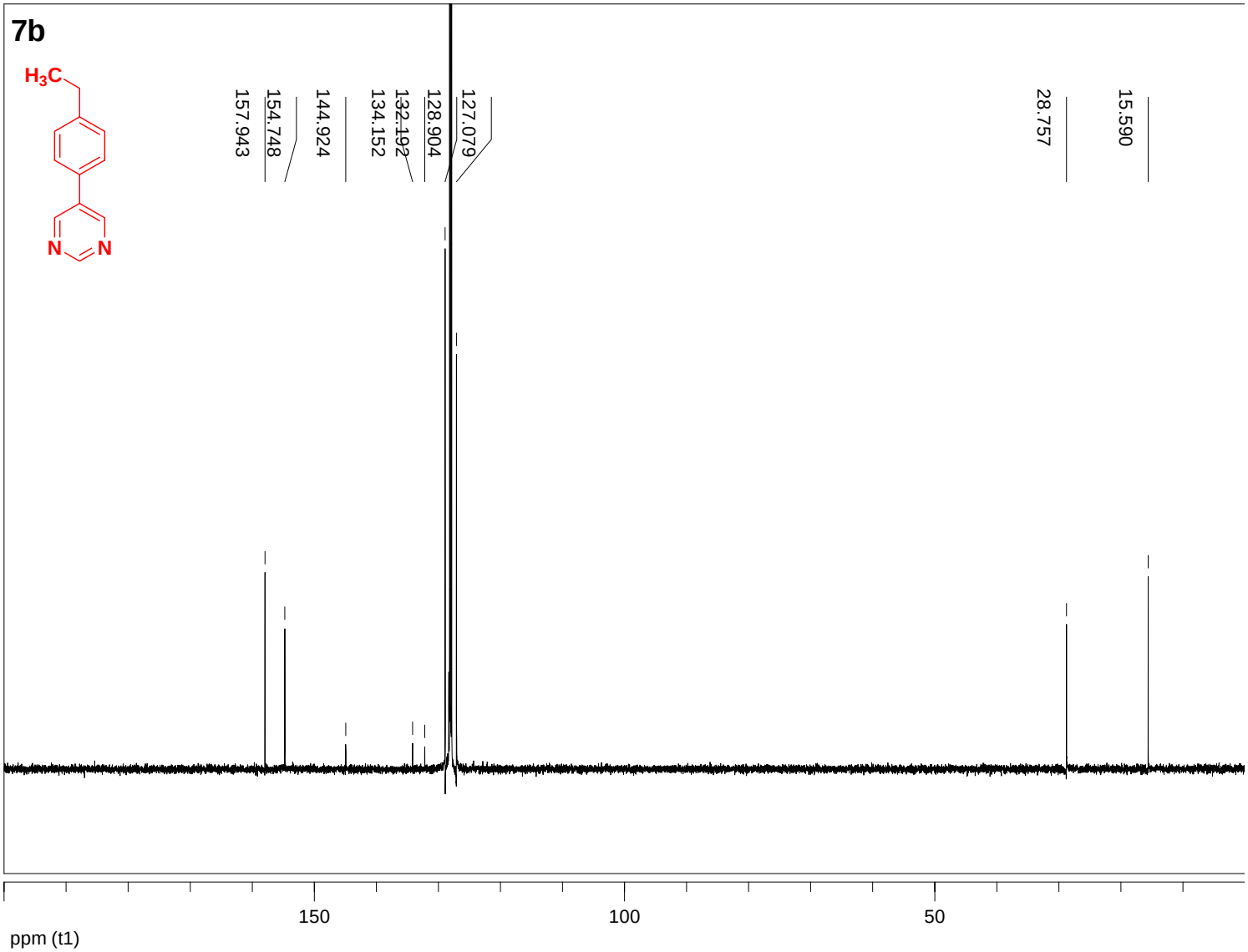

6d
7.495
7.482
7.472
7.459
7.150
7.110
7.097
7.096
7.071
7.070
7.057
2.497
2.484
2.156
1.136
1.124
1.111
5.0
ppm (t1)

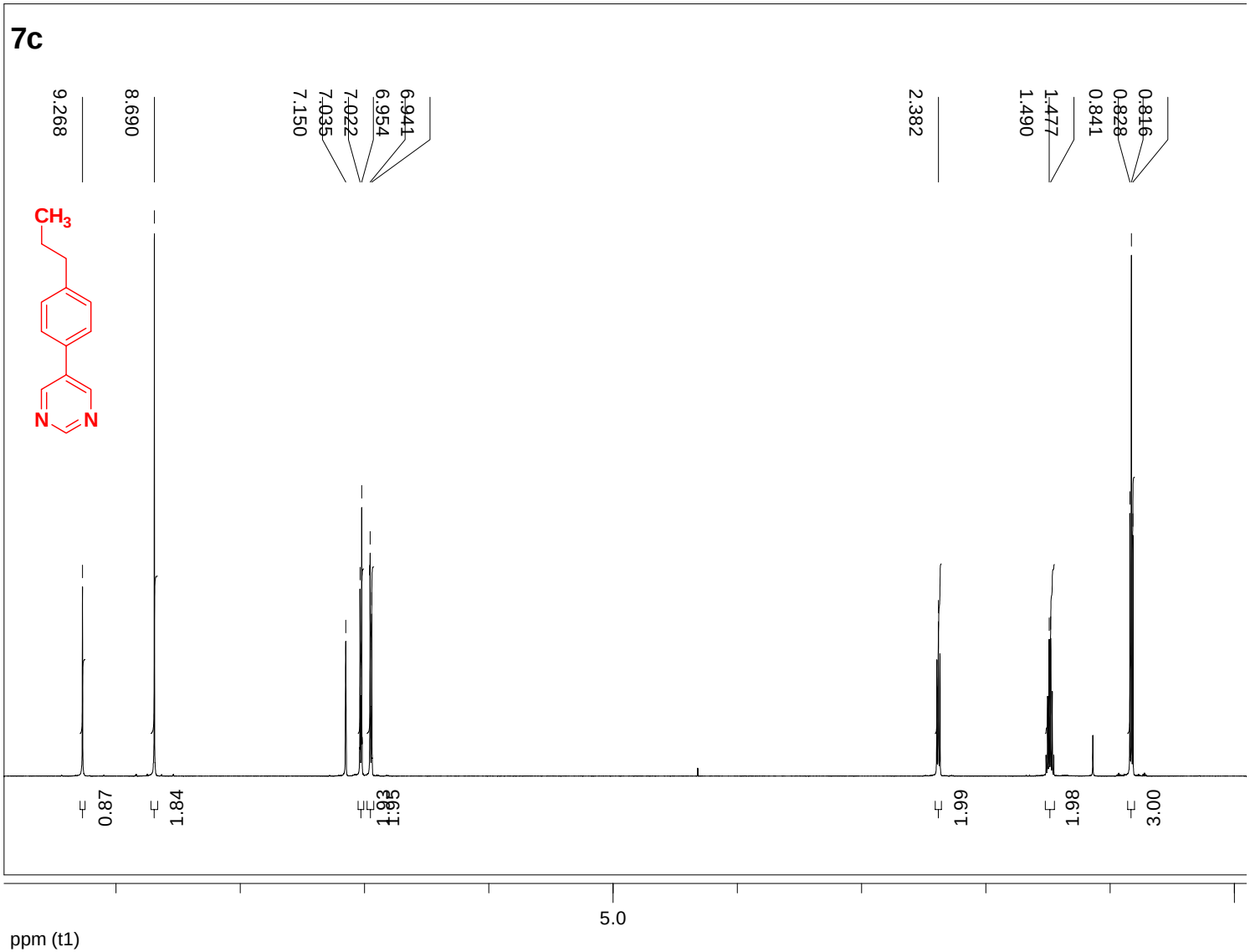

6d
143.107
139.170
138.952
136.644
129.766
128.573
127.314
127.237
28.842
21.042
15.880
150
100
50
ppm (t1)

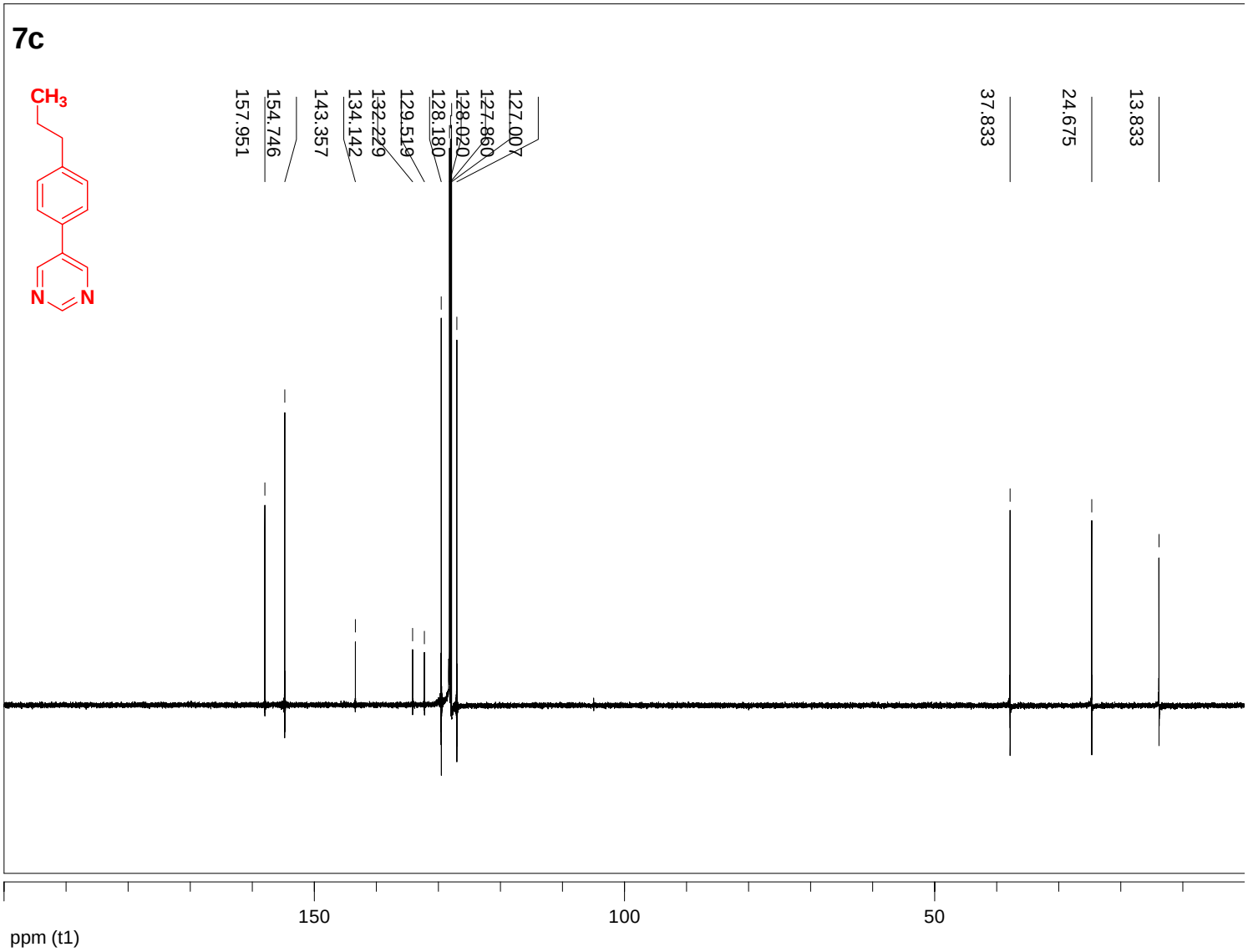

7a
9.175
8.932
7.482
7.468
7.330
7.317
7.260
2.422
N
N
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

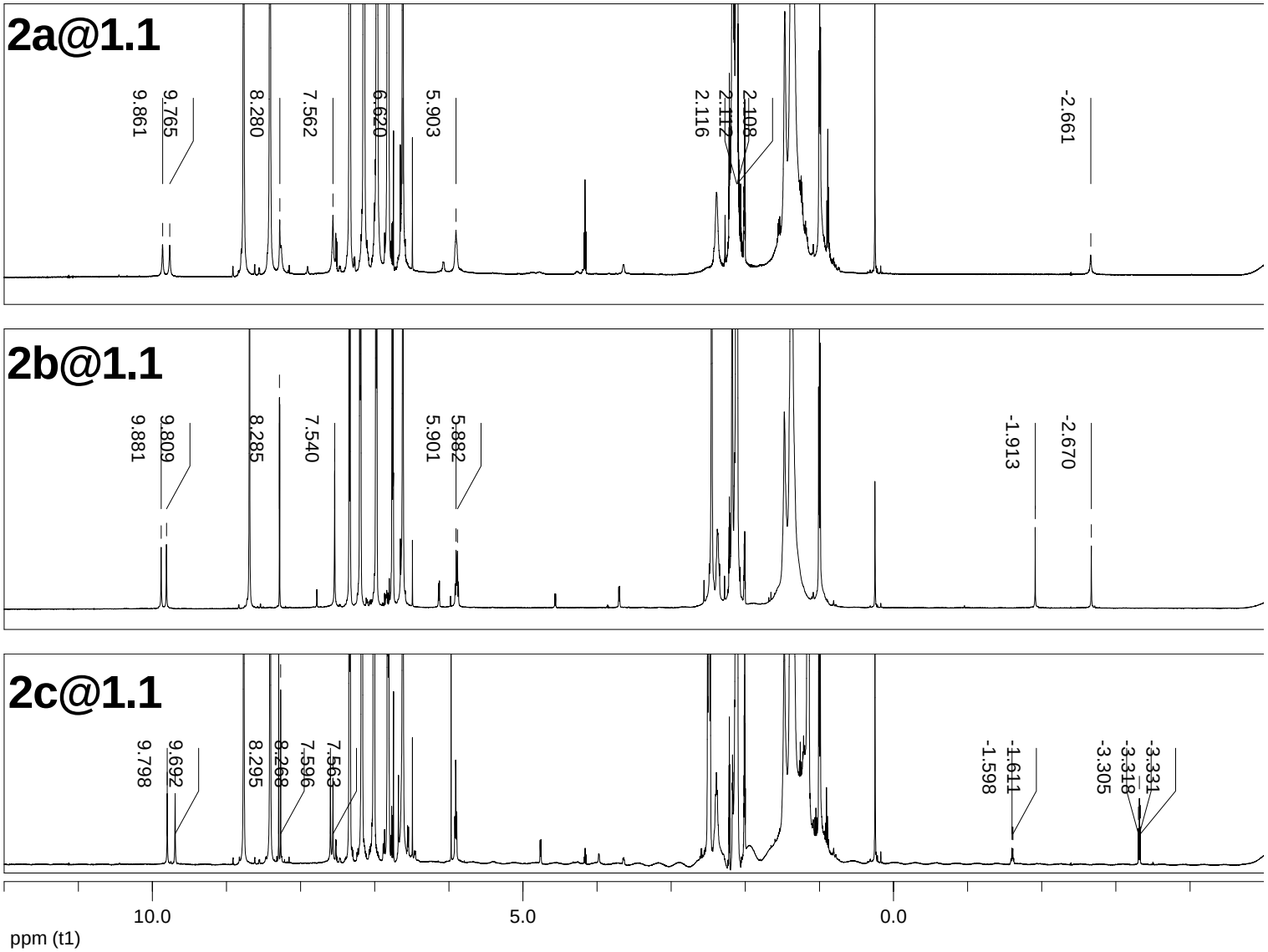

7a
157.117
154.661
139.115
134.247
131.270
130.119
126.767
77.211
77.000
76.789
21.183
200
150
100
50
0
ppm (t1)

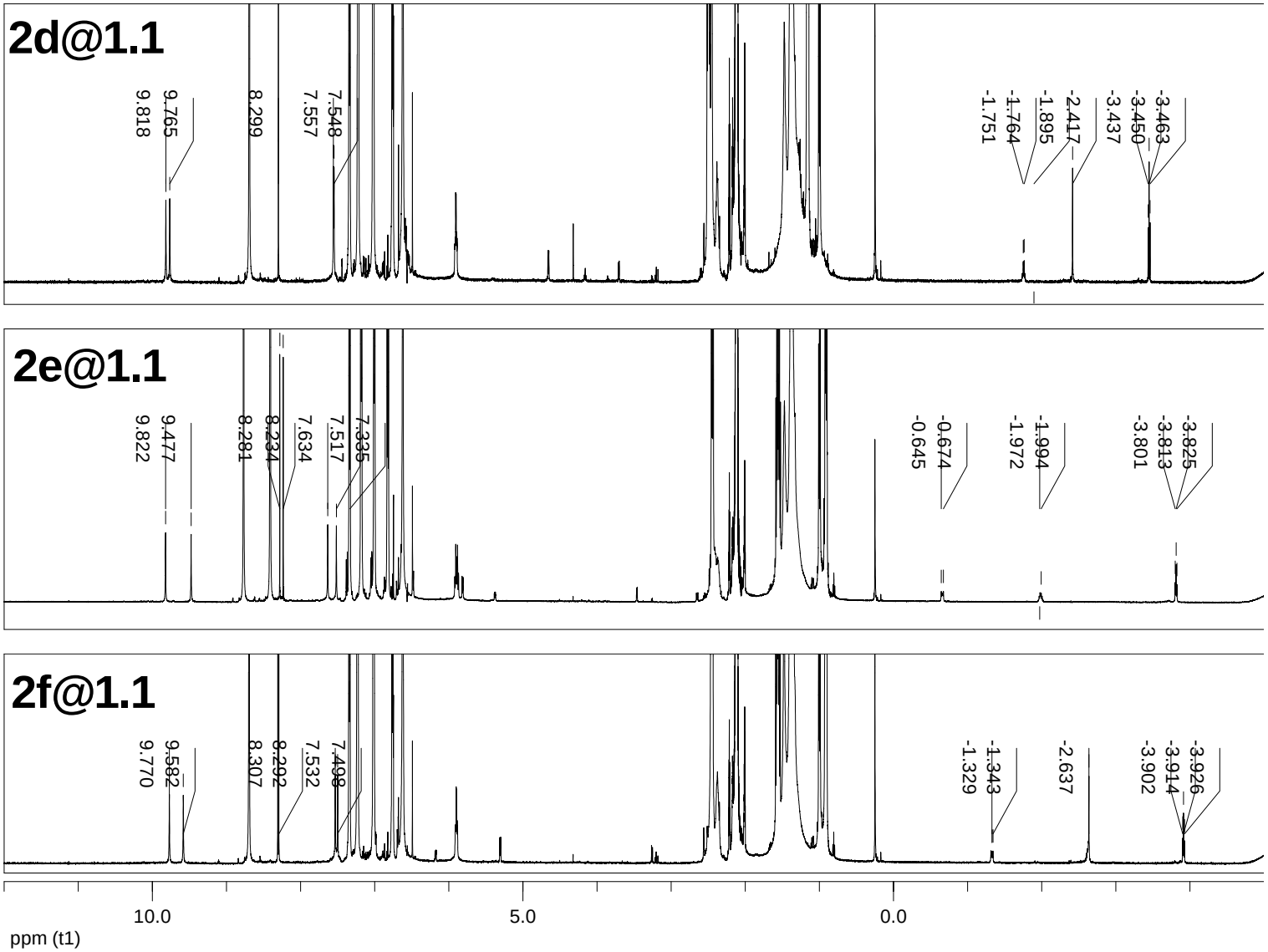

7b
9.268
8.688
7.150
7.029
7.015
6.952
6.939
2.420
2.408
2.395
2.382
1.067
1.055
1.042
H3C
N
N
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

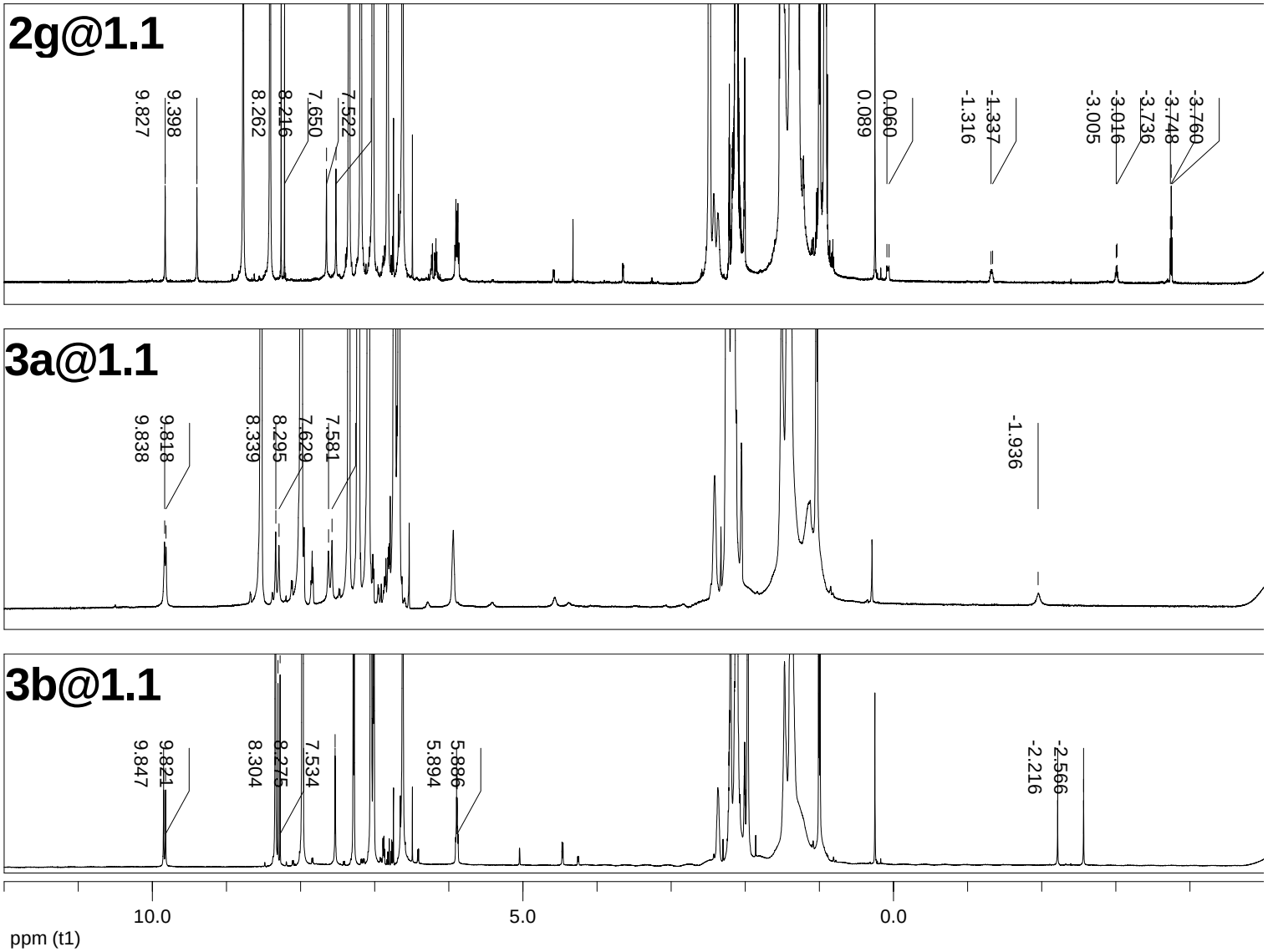

7b
H3C
N
N
157.943
154.748
144.924
134.152
132.192
128.904
127.079
28.757
15.590
150
100
50
ppm (t1)

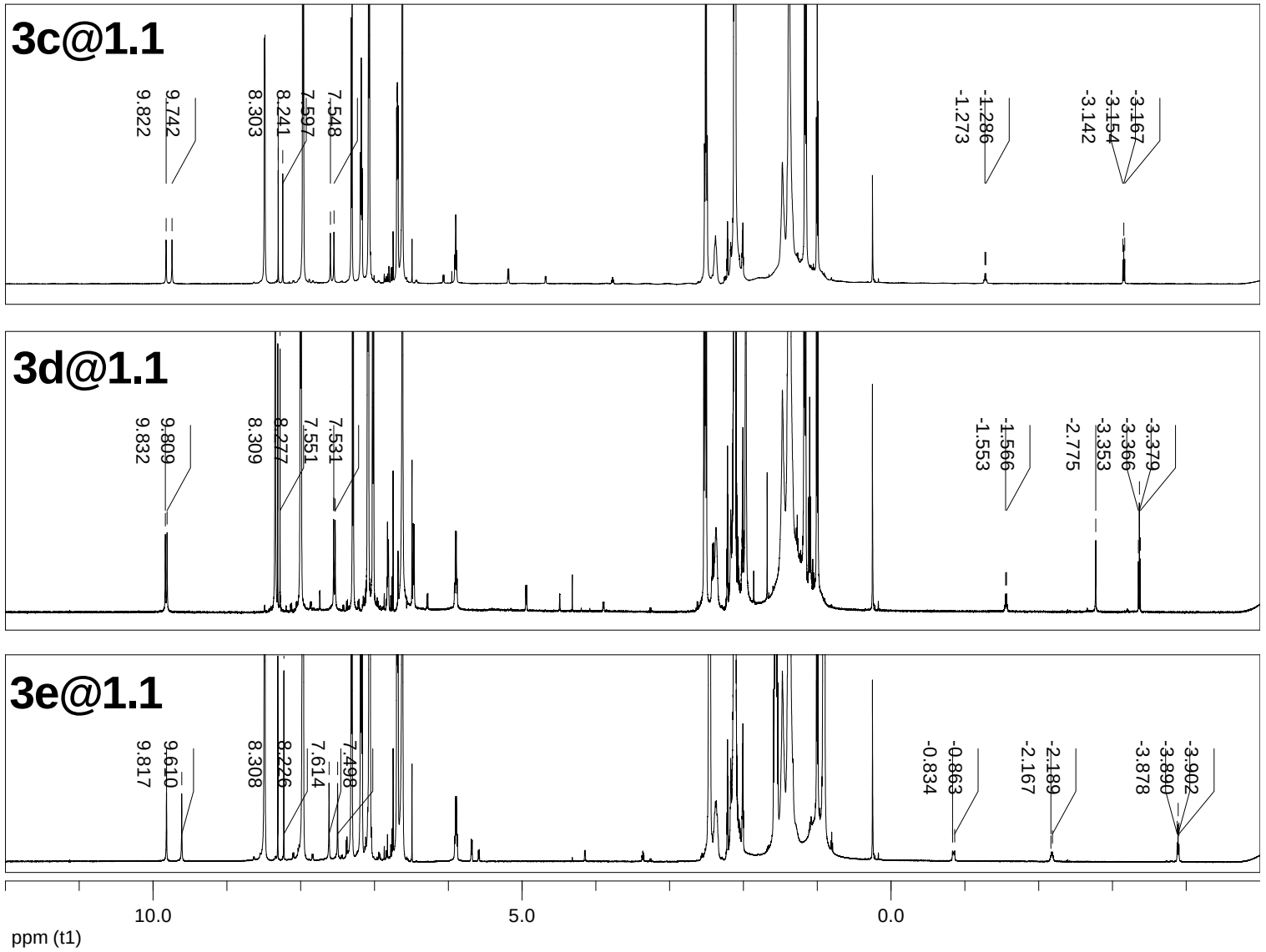

7c
9.268
8.690
7.150
7.035
7.022
6.954
6.941
2.382
1.490
1.477
0.841
0.828
0.816
CH3
N
N
0.87
1.84
1.99
1.98
3.00
5.0
ppm (t1)

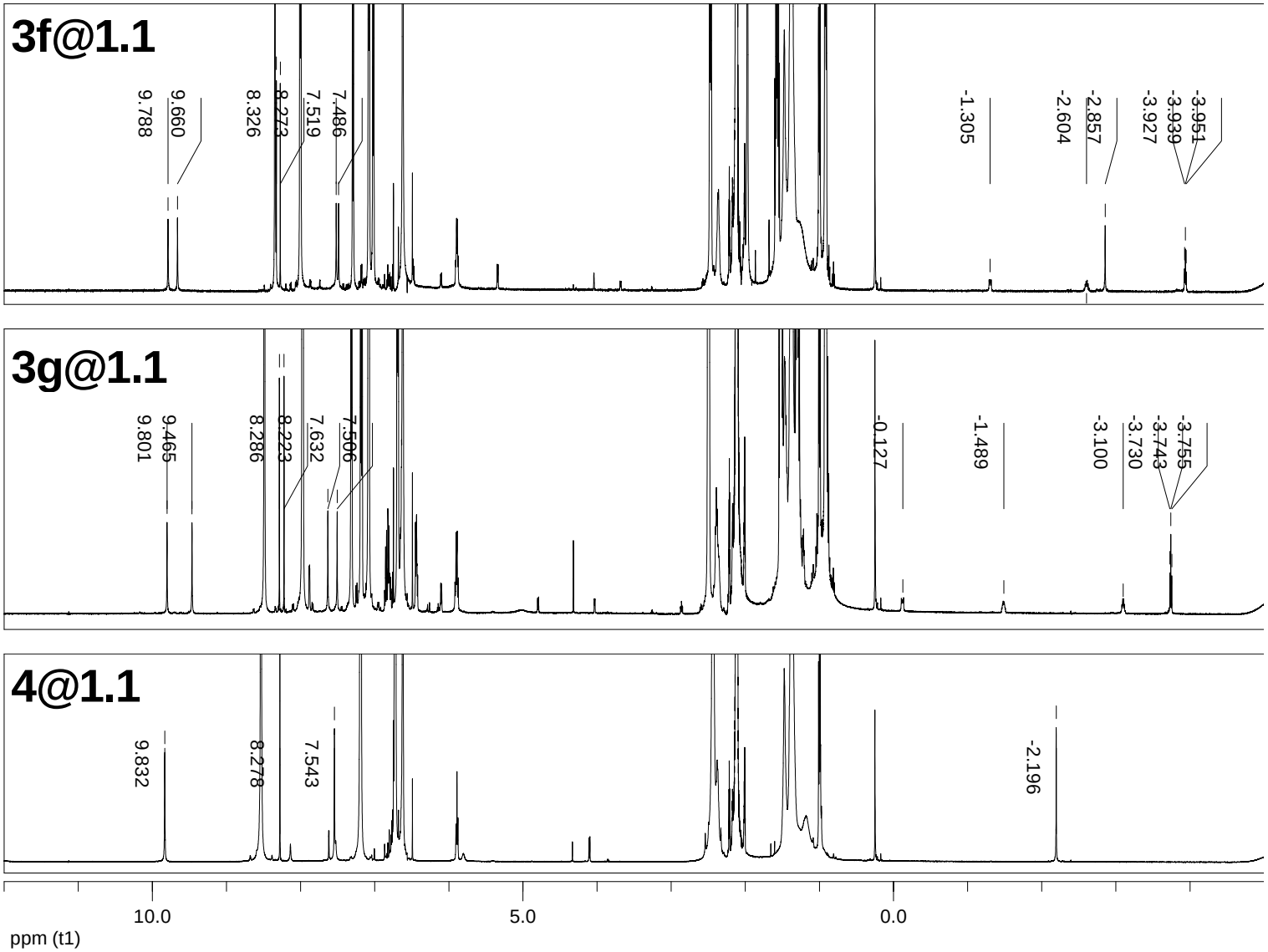

7c
CH3
N
N
157.951
154.746
143.357
134.142
132.229
129.519
128.180
128.020
127.860
127.007
37.833
24.675
13.833
150
100
50
ppm (t1)

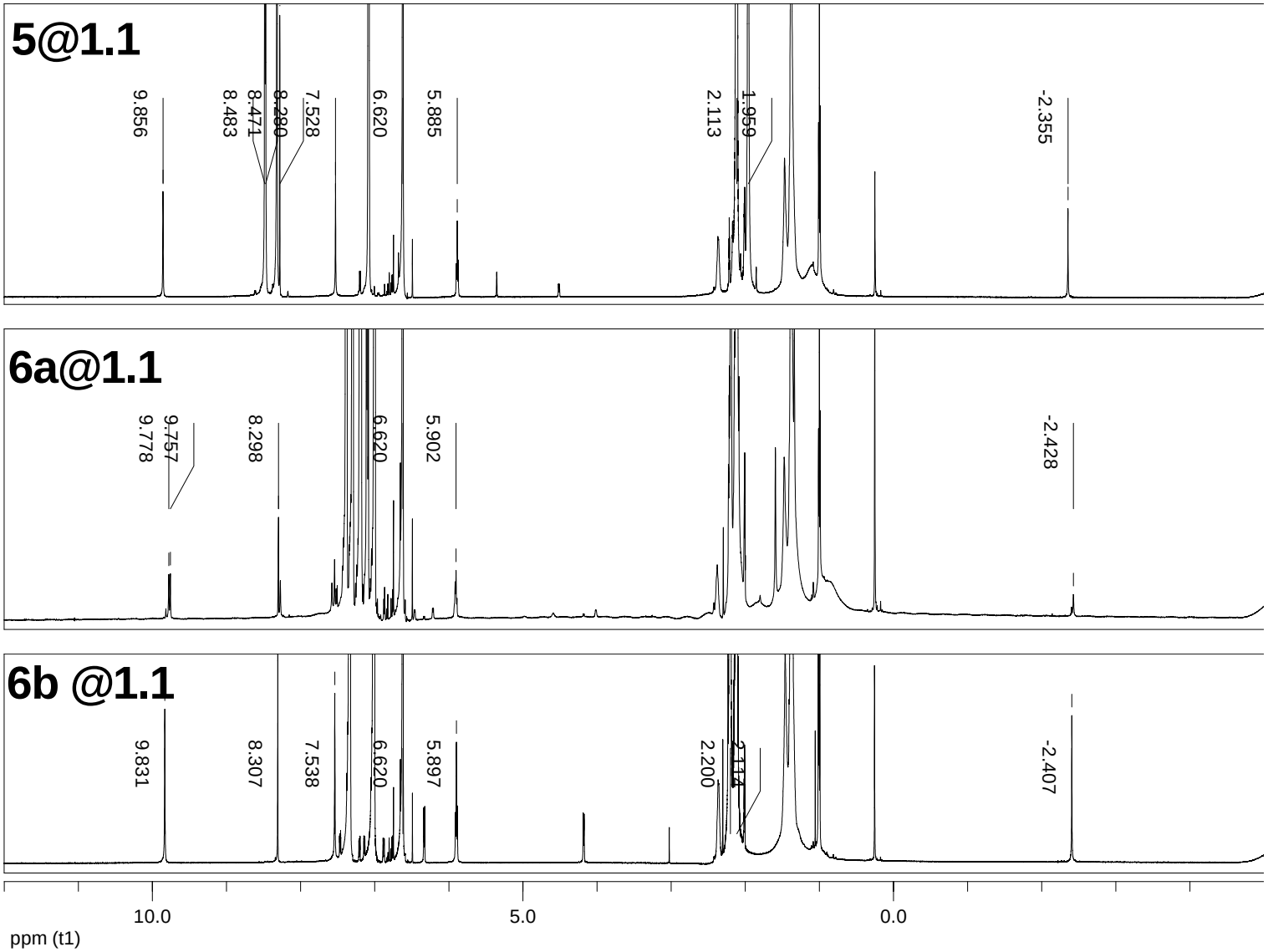

2a@1.1
9.861
9.765
8.280
7.562
6.620
5.903
2.116
2.112
2.108
-2.661
2b@1.1
9.881
9.809
8.285
7.540
5.901
5.882
-1.913
-2.670
2c@1.1
9.798
9.692
8.295
8.268
7.596
7.563
-1.598
-1.611
-3.305
-3.318
-3.331
10.0
5.0
0.0
ppm (t1)

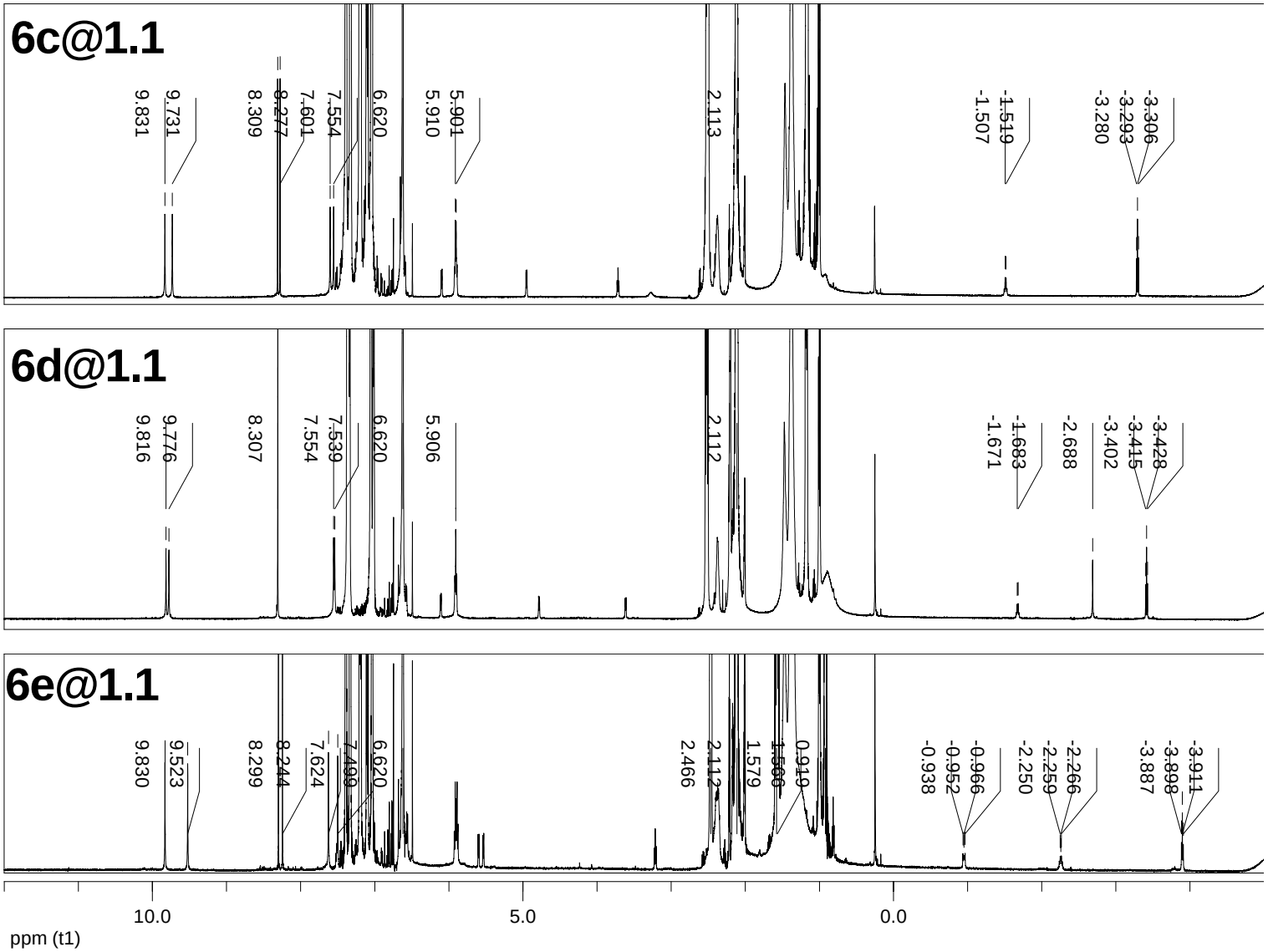

2d@1.1
9.818
9.765
8.299
7.557
7.548
-1.751
-1.764
-1.895
-2.417
-3.437
-3.450
-3.463
2e@1.1
9.822
9.477
8.281
8.234
7.634
7.517
7.335
-0.645
-0.674
-1.972
-1.994
-3.801
-3.813
-3.825
2f@1.1
9.770
9.582
8.307
8.292
7.532
7.498
-1.329
-1.343
-2.637
-3.902
-3.914
-3.926
10.0
5.0
0.0
ppm (t1)

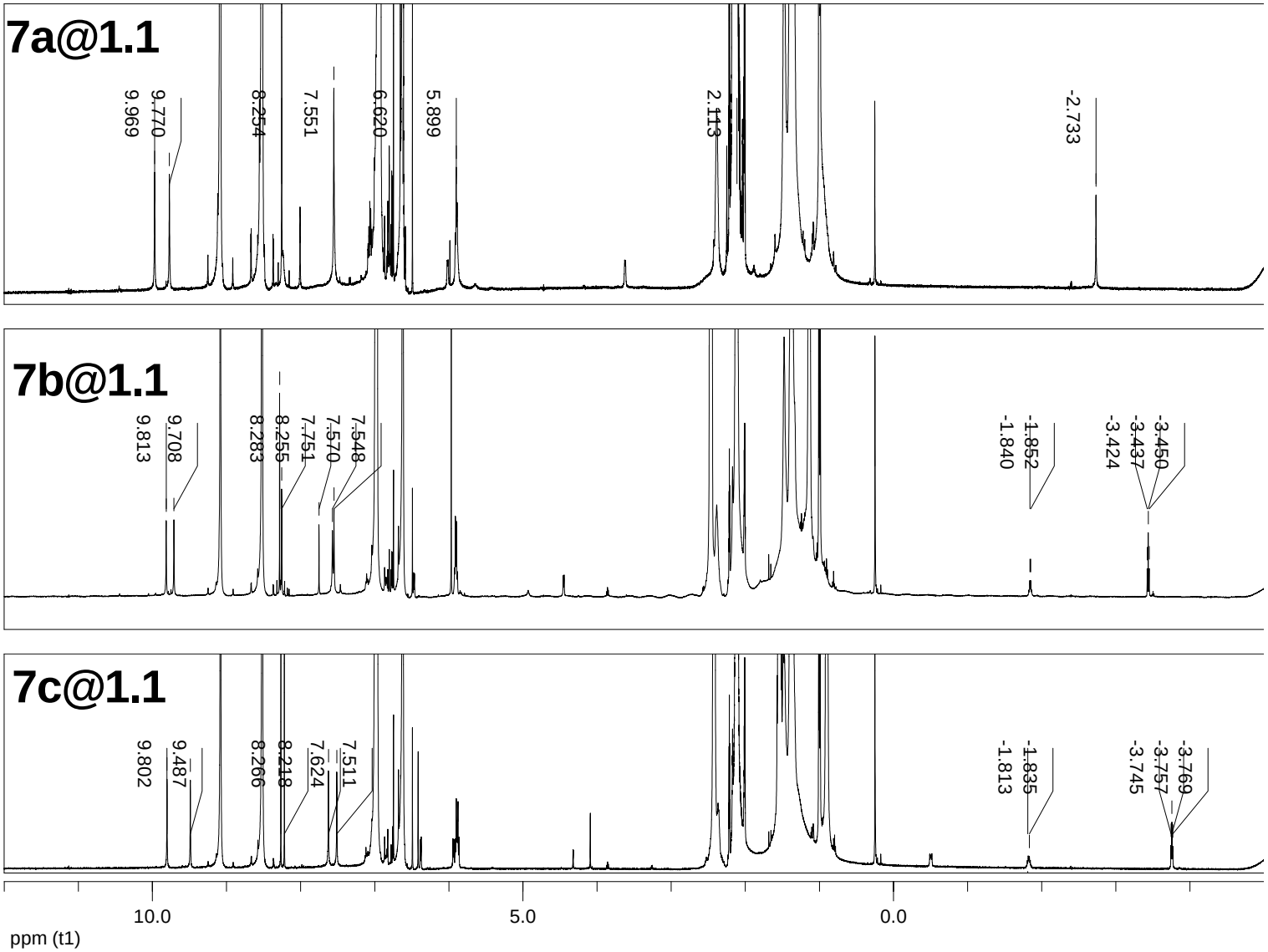

2g@1.1
9.827
9.398
8.262
8.216
7.650
7.522
0.089
0.060
-1.316
-1.337
-3.005
-3.016
-3.736
-3.748
-3.760
3a@1.1
9.838
9.818
8.339
8.295
7.629
7.581
-1.936
3b@1.1
9.847
9.821
8.304
8.275
7.534
5.894
5.886
-2.216
-2.566
10.0
5.0
0.0
ppm (t1)


3c@1.1
9.822
9.742
8.303
8.241
7.597
7.548
-1.286
-1.273
-3.167
-3.154
-3.142
3d@1.1
9.832
9.809
8.309
8.277
7.551
7.531
-1.566
-1.553
-2.775
-3.353
-3.366
-3.379
3e@1.1
9.817
9.610
8.308
8.226
7.614
7.498
-0.834
-0.863
-2.167
-2.189
-3.878
-3.890
-3.902
10.0
5.0
0.0
ppm (t1)


3f@1.1
9.788
9.660
8.326
8.273
7.519
7.486
-1.305
-2.604
-2.857
-3.927
-3.939
-3.951
3g@1.1
9.801
9.465
8.286
8.223
7.632
7.506
-0.127
-1.489
-3.100
-3.730
-3.743
-3.755
4@1.1
9.832
8.278
7.543
-2.196
10.0
5.0
0.0
ppm (t1)


5@1.1
9.856
8.483
8.471
8.290
7.528
6.620
5.885
2.113
1.959
-2.355
6a@1.1
9.778
9.757
8.298
6.620
5.902
-2.428
6b @1.1
9.831
8.307
7.538
6.620
5.897
2.200
2.114
-2.407
10.0
5.0
0.0
ppm (t1)


6c@1.1
9.831
9.731
8.309
8.277
7.601
7.554
6.620
5.910
5.901
2.113
-1.507
-1.519
-3.280
-3.293
-3.306
6d@1.1
9.816
9.776
8.307
7.554
7.539
6.620
5.906
2.112
-1.671
-1.683
-2.688
-3.402
-3.415
-3.428
6e@1.1
9.830
9.523
8.299
8.244
7.624
7.499
6.620
2.466
2.112
1.579
1.566
0.919
-0.938
-0.952
-0.966
-2.250
-2.259
-2.266
-3.887
-3.898
-3.911
10.0
5.0
0.0
ppm (t1)


7a@1.1
9.969
9.770
8.254
7.551
6.620
5.899
2.113
-2.733
7b@1.1
9.813
9.708
8.283
8.255
7.751
7.570
7.548
-1.852
-1.840
-3.424
-3.437
-3.450
7c@1.1
9.802
9.487
8.266
8.218
7.624
7.511
-1.835
-1.813
-3.745
-3.757
-3.769
10.0
5.0
0.0
ppm (t1)

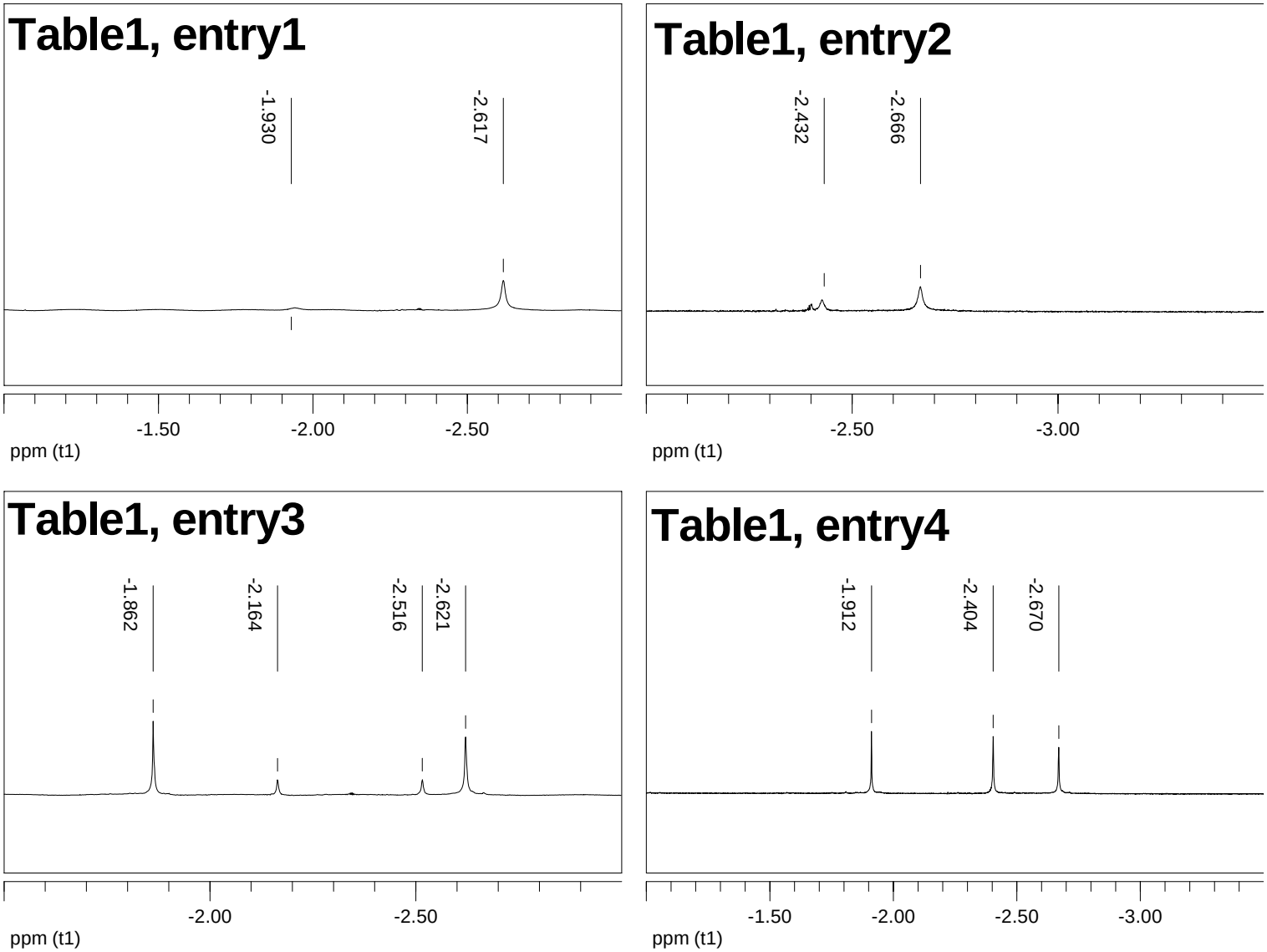


Table1, entry1
-1.930
-2.617
-1.50
-2.00
-2.50
ppm (t1)
Table1, entry2
-2.432
-2.666
-2.50
-3.00
ppm (t1)
Table1, entry3
-1.862
-2.164
-2.516
-2.621
-2.00
-2.50
ppm (t1)
Table1, entry4
-1.912
-2.404
-2.670
-1.50
-2.00
-2.50
-3.00
ppm (t1)

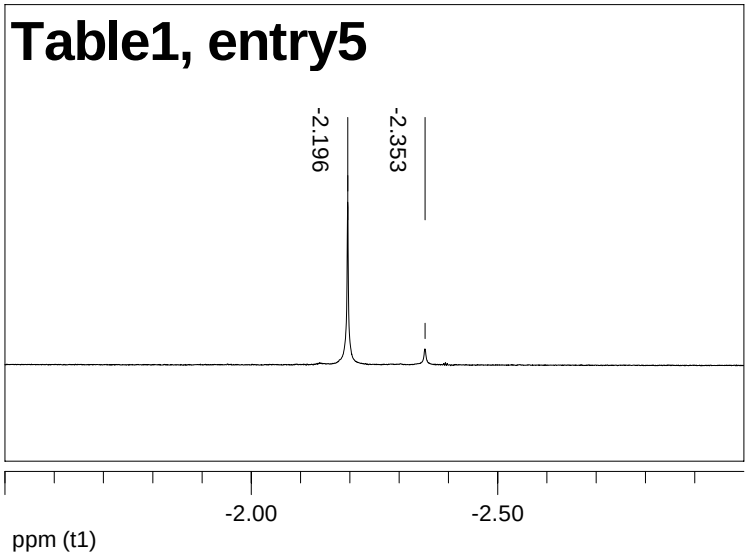


Table1, entry5
-2.196
-2.353
-2.00
-2.50
ppm (t1)

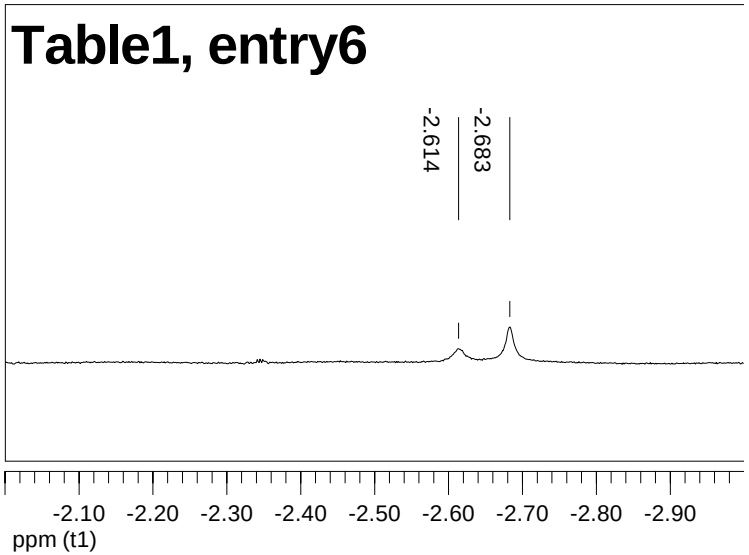


Table1, entry6
-2.614
-2.683
-2.10 -2.20 -2.30 -2.40 -2.50 -2.60 -2.70 -2.80 -2.90
ppm (t1)

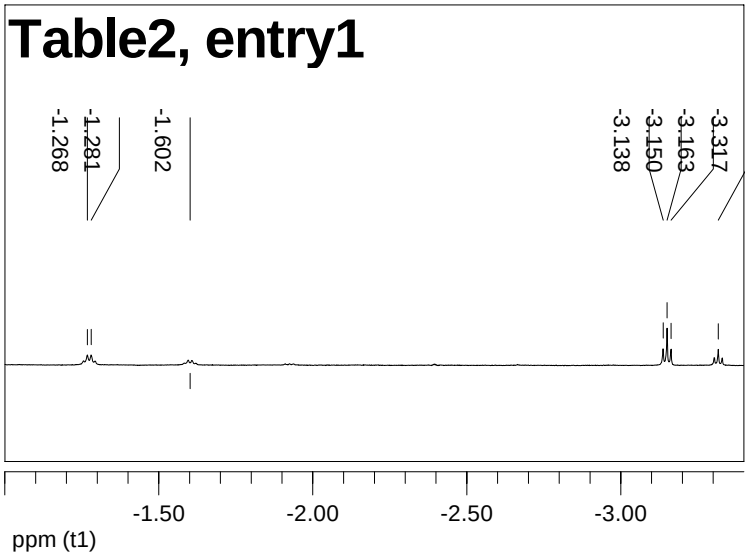


Table2, entry1
-1.268
-1.281
-1.602
-3.138
-3.150
-3.163
-3.317
-1.50 -2.00 -2.50 -3.00
ppm (t1)

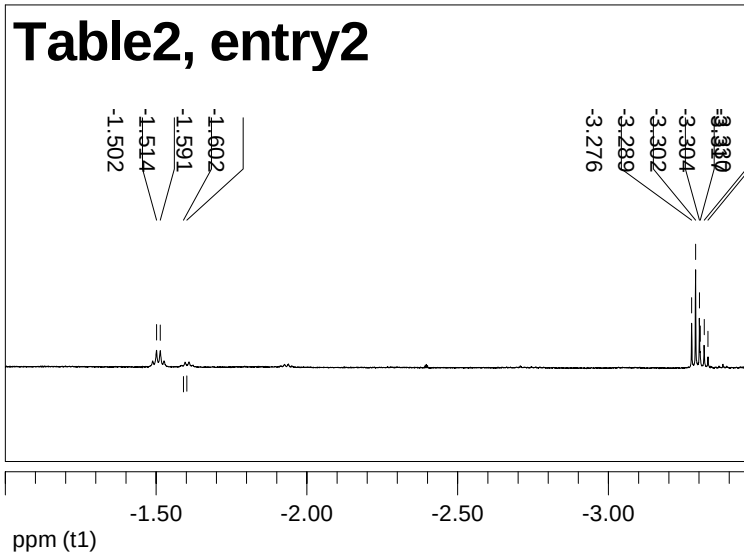


Table2, entry2
-1.502
-1.514
-1.591
-1.602
-3.276
-3.289
-3.302
-3.304
-1.50 -2.00 -2.50 -3.00
ppm (t1)

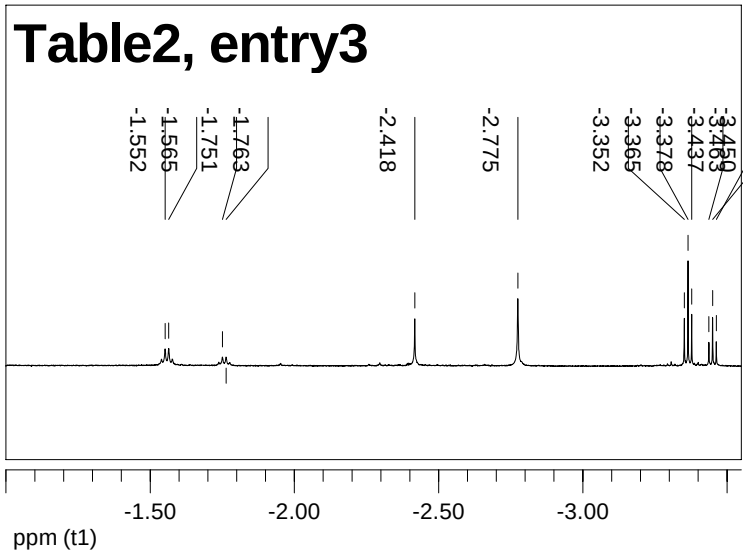


Table2, entry3
-1.552
-1.565
-1.751
-1.763
-2.418
-2.775
-3.352
-3.365
-3.378
-3.437
-3.459
-3.463
-1.50
-2.00
-2.50
-3.00
ppm (t1)

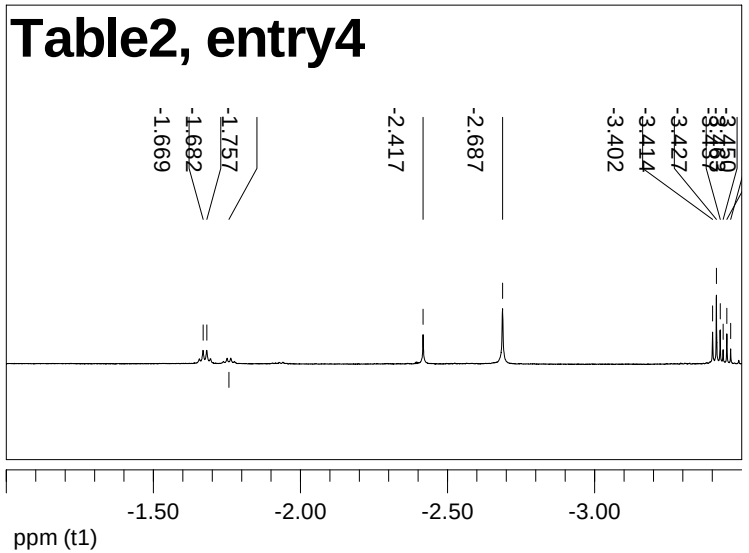


Table2, entry4
-1.669
-1.682
-1.757
-2.417
-2.687
-3.402
-3.414
-3.427
-1.50
-2.00
-2.50
-3.00
ppm (t1)

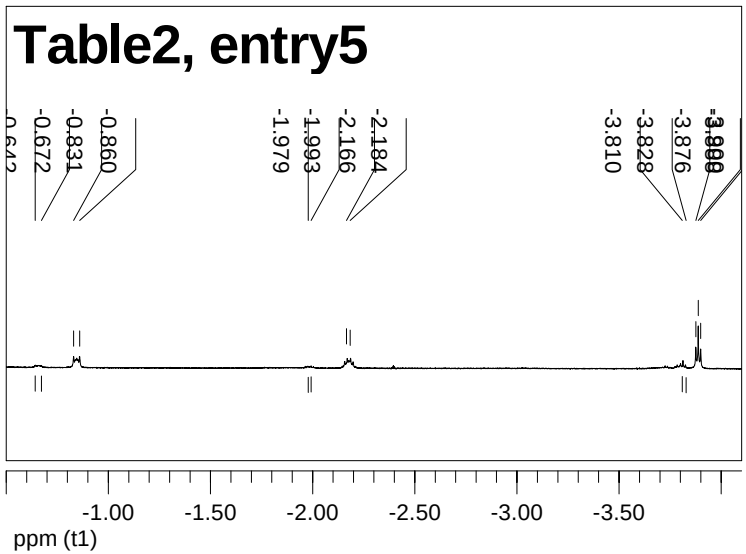


Table2, entry5
-0.672
-0.831
-0.860
-1.979
-1.993
-2.166
-2.184
-3.810
-3.828
-3.876
-1.00
-1.50
-2.00
-2.50
-3.00
-3.50
ppm (t1)

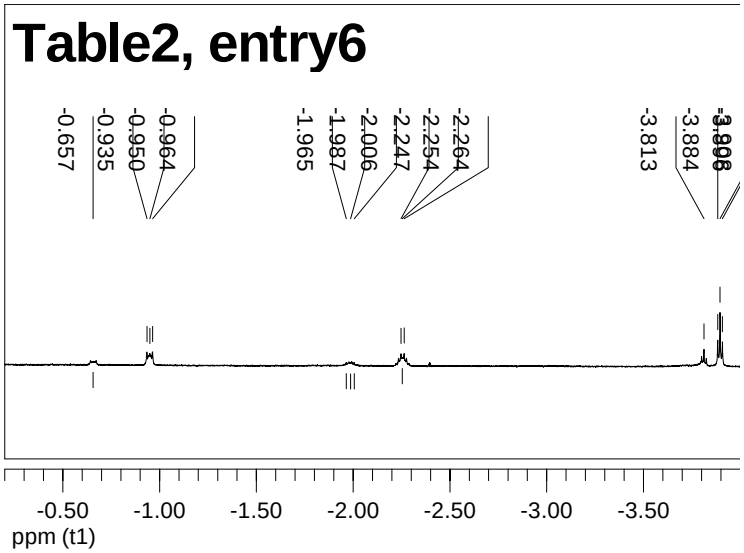


Table2, entry6
-0.657
-0.935
-0.950
-0.964
-1.965
-1.987
-2.006
-2.247
-2.254
-2.264
-3.813
-3.884
-0.50
-1.00
-1.50
-2.00
-2.50
-3.00
-3.50
ppm (t1)

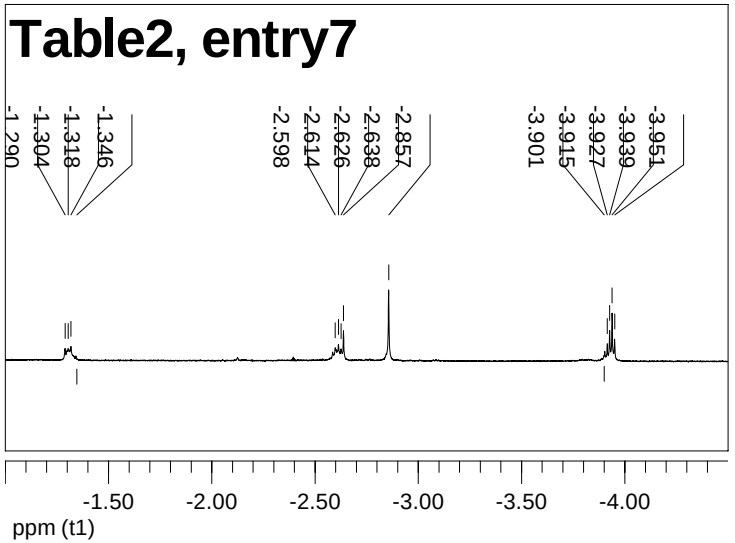


Table2, entry7
-1.290
-1.304
-1.318
-1.346
-2.598
-2.614
-2.626
-2.638
-2.857
-3.901
-3.915
-3.927
-3.939
-3.951
-1.50
-2.00
-2.50
-3.00
-3.50
-4.00
ppm (t1)

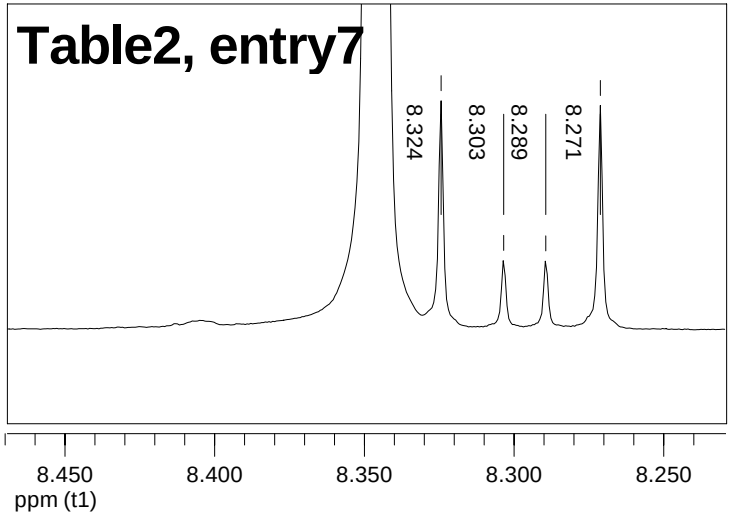


Table2, entry7
8.324
8.303
8.289
8.271
8.450
8.400
8.350
8.300
8.250
ppm (t1)

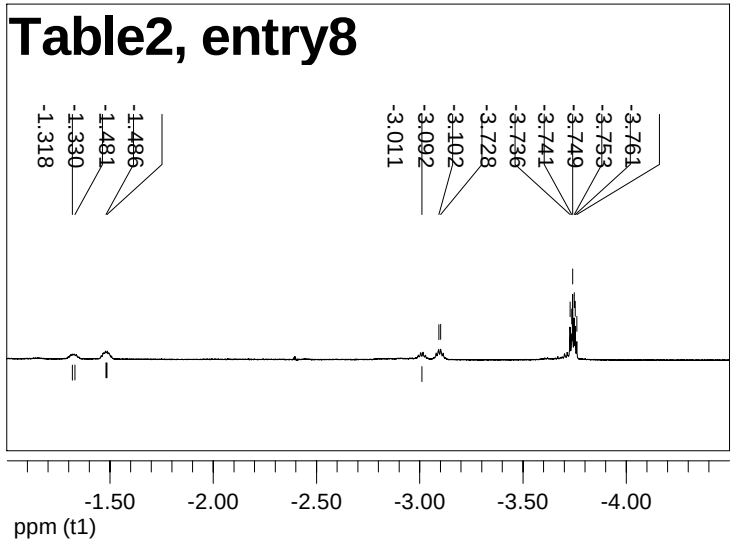


Table2, entry8
-1.318
-1.330
-1.481
-1.486
-3.011
-3.092
-3.102
-3.728
-3.736
-3.741
-3.749
-3.753
-3.761
-1.50
-2.00
-2.50
-3.00
-3.50
-4.00
ppm (t1)

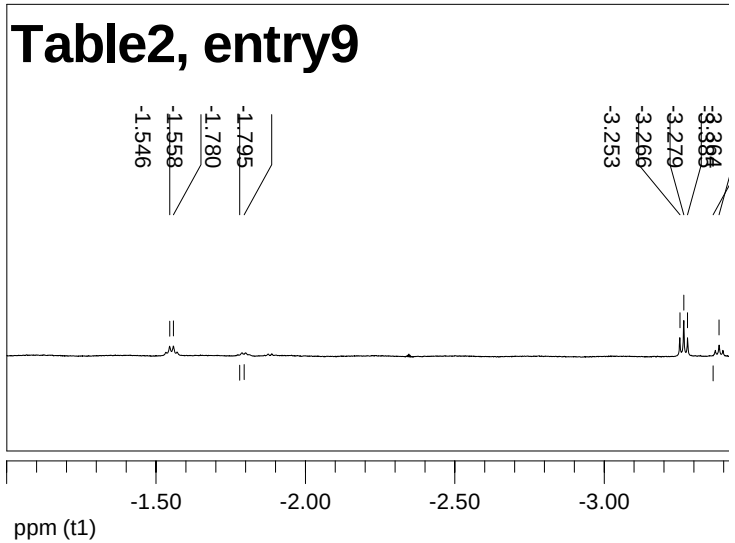


Table2, entry9
-1.546
-1.558
-1.780
-1.795
-3.253
-3.266
-3.279
-1.50
-2.00
-2.50
-3.00
ppm (t1)

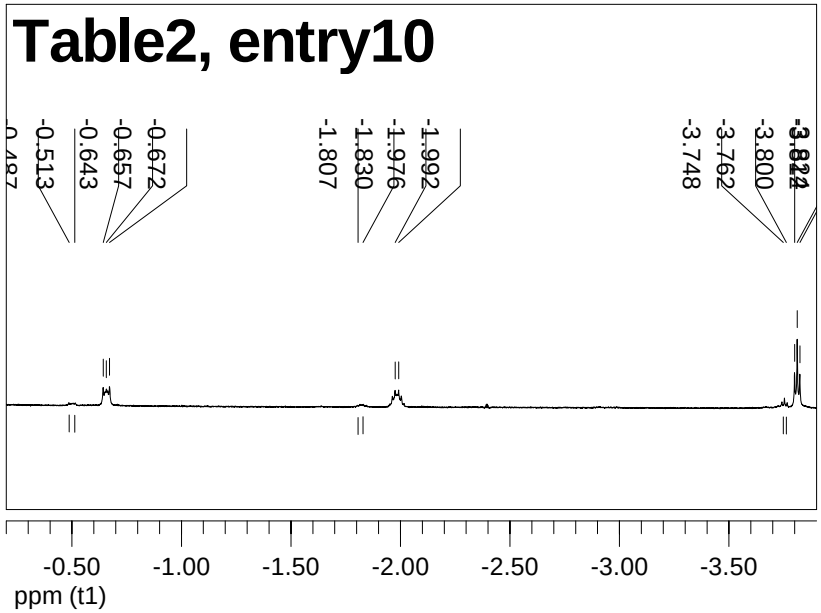


Table2, entry10
-0.487
-0.513
-0.643
-0.657
-0.672
-1.807
-1.830
-1.976
-1.992
-3.748
-3.762
-3.800
-0.50
-1.00
-1.50
-2.00
-2.50
-3.00
-3.50
ppm (t1)