Supporting Information

for

A simple and convenient one-pot synthesis of substituted isoindolin-1-ones via lithiation, substitution and cyclization of *N*'-benzyl-*N*,*N*-dimethylureas

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Characterization data of all compounds given in the article and NMR spectra and X-ray information for representative compounds. CCDC 737411, 737415, 762623, 762624, 766180, 766181 and 766182.

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Characterization Data for Products

Note: When the multiplicity of a proton NMR signal appears simpler than expected (for example if a signal expected to be a double doublet appears as a triplet because the two coupling constants are very similar), the observed apparent multiplicity is recorded without comment in the data reported below.

4-Methoxy-3-methylisoindolin-1-one (9). Yield: 0.28 g (1.58 mmol, 79%); mp 153–154 °C; IR (FT) ν/cm^{-1}: 3071, 2971, 1680, 1602, 1493, 1267, 1057; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.26 (br s, exch., 1H, NH), 7.47 (t, *J* = 8 Hz, 1H, H-6), 7.24 (d, *J* = 8 Hz, 1H, H-7), 7.15 (d, *J* = 8 Hz, 1H, H-5), 4.73 (q, *J* = 7 Hz, 1H, H-3), 3.92 (s, 3H, OCH_3), 1.55 (d, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 171.2 (s, C-1), 155.3 (s, C-4), 136.8 (s, C-7a), 133.9 (s, C-3a), 130.1 (d, C-6), 116.0 (d, C-7), 113.6 (d, C-5), 55.8 (q, OCH_3), 52.0 (d, C-3), 19.3 (q, CH_3); MS (EI) *m*/*z*: 177 ([M]$^+$, 24%), 162 (100), 146 (10), 119 (8), 105 (9), 91 (11), 84 (8), 49 (15); MS (CI) *m*/*z*: 195 ([M + NH_4]$^+$, 30%), 178 ([MH]$^+$, 100), 162 (8), 94 (11), 52 (50), 44 (41); HRMS CI: Calc for $C_{10}H_{12}NO_2$ [MH$^+$]: 178.0863; Found 178.0864.

3-Ethyl-4-methoxyisoindolin-1-one (10). Yield: 0.32 g (1.68 mmol, 84%); mp 150–152 °C; IR (FT) ν/cm^{-1}: 3303, 2922, 1679, 1602, 1493, 1271, 1049.^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.72 (s, exch., 1H, NH), 7.44 (t, *J* = 8 Hz, 1H, H-6), 7.23 (d, *J* = 8 Hz, 1H, H-7), 7.18 (d, *J* = 8 Hz, 1H, H-5), 4.57 (dd, *J* = 3, 7 Hz, 1H, H-3), 3.87 (s, 3H, OCH_3), 2.08 (ddq, *J* = 3, 14, 7 Hz, 1H, 1H of CH_2), 1.59 (d quintet, *J* = 14, 7 Hz, 1 H, 1 H of CH_2), 0.73 (t, *J* = 7 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.7 (s, C-1), 155.1 (s, C-4), 134.8 (s, C-7a), 134.4 (s, C-3a), 130.1 (d, C-6), 115.0 (d, C-7), 113.8 (d, C-5), 56.1 (d, C-3), 55.9 (q, OCH_3), 25.0 (t, CH_2), 9.2 (q, CH_3); MS (EI) *m*/*z*: 191 ([M]$^+$, 7%), 162 (100), 149 (5), 132 (6); MS (CI) *m*/*z*: 209 ([M + NH_4]$^+$, 9%), 192 ([MH]$^+$, 100), 176 (12), 162 (28), 94 (11), 59 (33), 44 (40); HRMS (CI): Calc for $C_{11}H_{14}NO_2$ [MH$^+$]:192.1019; Found 192.1018.

3-Butyl-4-methoxyisoindolin-1-one (11). Yield: 0.32 g (1.45 mmol, 72%); mp 130–131 °C (a known compound but no mp reported [76]); IR (FT) ν/cm^{-1}: 3314, 2955, 1678, 1600, 1490, 1276, 1053; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.57 (s, exch., 1H, NH), 7.47–7.41 (m, 2H, H-6 and H-7), 7.22 (dd, *J* = 2, 8 Hz, 1H, H-5), 4.69 (dd, *J* = 3, 7 Hz, 1H, H-3), 3.91 (s, 3H, OCH_3), 2.20 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.65 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.39–1.22 (m, 4H, C*H*$_2$C*H*$_2$CH$_3$), 0.88 (t, *J* = 7 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 171.5 (s, C-1), 155.3 (s, C-4), 135.6 (s, C-7a), 134.2 (s, C-3a), 130.0 (d, C-6), 116.1 (d, C-7), 113.5 (d, C-5), 56.4 (d, C-3), 55.8 (q, OCH_3), 32.4 (t, *C*H$_2$CH$_2$CH$_2$CH$_3$), 27.9 (t, *C*H$_2$CH$_2$CH$_3$), 23.0 (t, *C*H$_2$CH$_3$), 14.3 (q, CH_3); MS (EI) *m*/*z*: 219 ([M]$^+$, 4%), 188 (3), 162 (100), 148 (4), 132 (5), 119 (4), 91 (4), 77 (6); MS (CI) *m*/*z*: 237 ([M + NH_4]$^+$, 8%), 220 ([MH]$^+$, 100), 204 (9), 190 (11), 164 (18), 162 (17), 86 (17), 72 (38), 58 (41), 44 (39); HRMS (CI): Calc for $C_{13}H_{18}NO_2$ [MH$^+$]: 220.1332; Found 220.1332.

4-Methoxyisoindolin-1-one (12). Yield; 0.27 g (1.65 mmol, 82%); mp 190–192 °C (Lit [77] >200 °C); IR (FT) ν/cm^{-1}: 3286, 2870, 1682, 1585, 1440, 1266, 1052; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.60 (s, exch., 1H, NH), 7.46 (t, *J* = 8 Hz, 1H, H-6), 7.26 (d, *J* = 8 Hz, 1H, H-7), 7.19 (d, *J* = 8 Hz, 1H, H-5), 4.29 (s, 2H, H-3), 3.88 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 170.3 (s, C-1), 154.9 (s, C-4), 134.6 (s, C-3a), 131.8 (s, C-7a), 129.9 (d, C-6), 115.2 (d, C-7), 113.5 (d, C-5), 55.8 (q, OCH_3), 43.0 (t, C-3); MS (EI) *m*/*z*: 163 ([M]$^+$, 100%), 162 (42), 135 (12), 134 (22), 132 (44), 119 (22), 104 (18), 92 (15), 77 (26); MS (CI) *m*/*z*: 327 ([2 M + 1]$^+$, 6%), 181 ([M + NH_4]$^+$, 14) 164 ([MH]$^+$, 100), 134 (10); HRMS (CI): Calc for $C_9H_{10}NO_2$ [MH$^+$]: 164.0706; Found: 164.0707.

3-(Hydroxydiphenylmethyl)-4-methoxyisoindolin-1-one (13). Yield: 0.56 g (1.62 mmol, 81%); mp 206–208 °C; IR (FT) ν/cm^{-1}: 3301, 2981, 1690, 1599, 1491, 1267, 1038; ^{1}H NMR (400 MHz,

DMSO-d_6) δ (ppm): 7.89 (s, exch., 1H, NH), 7.52–7.18 (m, 11H, H-6 and 2 Ph), 7.10 (d, *J* = 8 Hz, 1H, H-7), 6.94 (d, *J* = 8 Hz, 1H, H-5), 5.84 (s, exch., 1H, OH), 5.76 (s, 1H, H-3), 3.19 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.7 (s, C-1), 155.0 (s, C-4), 145.8, 144.4 (2 s, C-1 of 2 Ph), 136.2 (s, C-7a), 131.9 (s, C-3a), 130.2 (s, C-6), 128.0, 127.4 (2 d, C-3/C-5 of 2 Ph), 127.1, 126.9 (2 d, C-2/C-6 of 2 Ph), 127.0, 126.8 (2 d, C-4 of 2 Ph), 115.0 (d, C-7), 113.9 (d, C-5), 79.7 (s, C-OH), 64.3 (d, C-3), 55.5 (q, OCH_3); MS (EI) *m*/*z*: 182 (22%), 163 (12), 105 (100), 77 (89), 51 (33); MS (CI) *m*/*z*: 346 ($[MH]^+$, 100%); MS (ES^+) *m*/*z*: 691 ($[2\ MH + 1]^+$, 12%), 346 ($[MH]^+$, 31), 164 (100); HRMS (ES^+): Calc for $C_{22}H_{20}NO_3$ [MH^+]: 346.1438; Found: 346.1440.

3-(1-Hydroxycyclohexyl)-4-methoxyisoindolin-1-one (14). Yield: 0.41 g (1.57 mmol, 78%); mp 206–207 °C; IR (FT) ν/cm^{-1}: 3301, 2952, 1690, 1590, 1470, 1240, 1047; ^1H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.45 (s, exch., 1H, NH), 7.43 (t, *J* = 8 Hz, 1H, H-6), 7.25 (d, *J* = 8 Hz, 1H, H-7), 7.22 (d, *J* = 8 Hz, 1H, H-5), 4.46 (s, 1H, H-3), 4.32 (s, exch., 1H, OH), 3.88 (s, 3H, OCH_3), 1.54–0.96 (m, 10H, cyclohexyl group); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.8 (s, C-1), 155.0 (s, C-4), 135.9 (s, C-7a), 132.0 (s, C-3a), 130.2 (d, C-6), 115.5 (d, C-7), 114.6 (d, C-5), 73.5 (s, C-1 of cyclohexyl group), 65.5 (d, C-3), 56.1 (q, OCH_3), 38.8, 33.7 (2 t, C-2/C-6 of cyclohexyl group), 25.8 (t, C-4 of cyclohexyl group), 21.5, 21.4 (2 t, C-3/C-5 of cyclohexyl group); MS (EI) *m*/*z*: 262 ($[MH]^+$, 11%), 262 ($[M]^+$, 2), 244 (32), 243 (100), 214 (33), 212 (21), 188 (48), 177 (19), 176 (34), 175 (55); MS (CI) *m*/*z*: 279 ($[M + NH_4]^+$, 2%), 262 ($[MH]^+$, 29), 244 (4), 181 (35), 164 (100), 134 (12), 116 (88), 98 (12), 55 (13); HRMS (CI): Calc for $C_{15}H_{20}NO_3$ [MH^+]: 262.1438; Found: 262.1435; Anal. Calc for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 69.13; H, 7.34; N, 5.48.

3-(1-Hydroxy-1-methylpentyl)-4-methoxyisoindolin-1-one (15). Yield: 0.41 g (1.56 mmol, 78%); Product **15** was a mixture of two diastereoisomers (**15a** and **15b**) in which many individual NMR signals could be identified, indicating that **15a**:**15b** = 47:53. IR (FT) ν/cm^{-1}: 3490, 3073, 1682,

1593, 1263, 1046; MS (EI) *m*/*z*: 163 (100%), 148 (10), 132 (13), 119 (11), 58 (15), 43 (18); MS (CI) *m*/*z*: 264 (30%) $[MH]^+$, 181 (7), 164 (18), 118 (100); HRMS (CI): Calc for $C_{15}H_{22}NO_3$ $[MH^+]$: 264.1600; Found: 264.1596; Crystallization of the crude product provided crystals of **15a**, mp 186–187 °C. Compound **15a** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.83 (s, exch., 1H, NH), 7.54 (dd, *J* = 1, 8 Hz, 1H, H-7), 7.49 (t, *J* = 8 Hz, 1H, H-6), 7.12 (dd, *J* = 1, 8 Hz, 1H, H-5), 4.72 (s, exch., 1H, OH), 4.08 (s, 1H, H-3), 3.99 (s, 3H, OCH_3), 1.34 (s, 3H, C*H*$_3$C-OH), 1.39–1.33 (m, 2H, C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.29–0.94 (m, 4H, C*H*$_2$C*H*$_2$CH$_3$), 0.79 (t, *J* = 7 Hz, 3H, CH$_2$C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 171.1 (s, C-1), 154.3 (s, C-4), 135.5 (s, C-7a), 132.5 (s, C-3a), 130.60 (d, C-6), 117.5 (d, C-7), 114.2 (d, C-5), 74.7 (s, C-OH), 66.9 (d, C-3), 56.49 (q, OCH_3), 36.8 (t, *C*H$_2$CH$_2$CH$_2$CH$_3$), 25.6 (t, *C*H$_2$CH$_2$CH$_3$), 24.8 (q, *C*H$_3$C-OH), 23.60 (t, *C*H$_2$CH$_3$), 14.4 (q, CH$_2$*C*H$_3$); Compound **15b** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.56 (s, exch., 1H, NH), 7.45 (dd, *J* = 1, 8 Hz, 1H, H-7), 7.41 (t, *J* = 8 Hz, 1H, H-6), 7.05 (dd, *J* = 1, 8 Hz, 1H, H-5), 4.66 (s, exch., 1H, OH), 4.36 (s, 1H, H-3), 3.92 (s, 3H, OCH_3), 1.59–1.33 (m, 6H, C*H*$_2$C*H*$_2$C*H*$_2$CH$_3$), 0.90 (t, *J* = 7 Hz, 3H, CH$_2$C*H*$_3$), 0.73 (s, 3H, C*H*$_3$C-OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 170.9 (s, C-1), 154.1 (s, C-4), 135.4 (s, C-7a), 132.9 (s, C-3a), 130.57 (d, C-6), 117.6 (d, C-7), 114.3 (d, C-5), 74.3 (s, C-OH), 64.7 (d, C-3), 56.52 (q, OCH_3), 40.5 (t, *C*H$_2$CH$_2$CH$_2$CH$_3$), 25.7 (t, *C*H$_2$CH$_2$CH$_3$), 23.62 (q, *C*H$_3$C-OH), 23.2 (t, *C*H$_2$CH$_3$), 14.5 (q, CH$_2$*C*H$_3$).

3-(1-Hydroxy-1-phenylethyl)-4-methoxyisoindolin-1-one (16). Yield: 0.43 g (1.61 mmol, 80%); Product **16** was a mixture of two diastereoisomers (**16a** and **16b**) in which many individual NMR signals could be identified, indicating that **16a**:**16b** = 56:44. IR (FT) ν/cm^{-1}: 3373, 3010, 1687, 1594, 1488, 1367, 1263, 1044; MS (EI) *m*/*z*: 265 (100%), 238 (22), 187 (35); MS (CI) *m*/*z*: 301 ($[M + NH_4]^+$, 3%), 284 ($[MH]^+$, 100), 268 (12); HRMS (CI): Calc for $C_{17}H_{18}NO_3$ $[MH]^+$: 284.1281; Found: 284.1281; Crystallization of the crude product provided crystals of **16a**, mp 235–236 °C. Compound **16a** (α-(*R**)-3-(*R**)- isomer): Anal. Calc for $C_{17}H_{17}NO_3$: C, 72.07; H, 6.05; N, 4.94.

Found: C, 71.98; H, 6.03; N, 4.94; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.61 (s, exch., 1H, NH), 7.32 (t, *J* = 8 Hz, 1H, H-6), 7.26 (d, *J* = 7 Hz, 2H, H-2/H-6 of Ph), 7.15 (t, *J* = 7 Hz, 2H, H-3/H-5 of Ph), 7.11–7.05 (m, 3H, H-5, H-7 and H-4 of Ph), 5.48 (s, exch., 1H, OH), 4.83 (s, 1H, H-3), 3.67 (s, 3H, OCH_3), 1.51 (s, 3H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.8 (s, C-1), 154.7 (s, C-4), 154.2 (s, C-1 of Ph), 135.5 (s, C-7a), 131.9 (s, C-3a), 130.2 (d, C-6), 127.5 (d, C-3/C-5 of Ph), 126.8 (d, C-4 of Ph), 125.6 (d, C-2/C-6 of Ph), 115.3 (d, C-7), 114.2 (d, C-5), 76.0 (s, C-OH), 65.7 (d, C-3), 56.1 (q, OCH_3), 26.6 (q, CH_3); Compound **16b** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.29 (s, exch., 1H, NH), 7.34 (t, *J* = 8 Hz, 1H, H-6), 7.18 (d, *J* = 7 Hz, 2H, H-2/H-6 of Ph), 7.12 (t, *J* = 7 Hz, 2H, H-3/H-5 of Ph), 7.01–6.88 (m, 3H, H-5, H-7 and H-4 of Ph), 5.43 (s, exch., 1H, OH), 4.77 (s, 1H, H-3), 3.72 (s, 3H, OCH_3), 1.74 (s, 3H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.0 (s, C-1), 155.4 (s, C-4), 154.8 (s, C-1 of Ph), 137.1 (s, C-7a), 131.1 (s, C-3a), 129.5 (d, C-6), 128.5 (d, C-3/C-5 of Ph), 128.1 (d, C-4 of Ph), 126.1 (d, C-2/C-6 of Ph), 115.9 (d, C-7), 114.5 (d, C-5), 76.0 (s, C-OH), 65.3 (d, C-3), 55.3 (q, OCH_3), 28.3 (q, CH_3).

3-(Hydroxy(4-methoxyphenyl)methyl)-4-methoxyisoindolin-1-one (17). Yield: 0.49 g (1.63 mmol, 81%); Product **17** was a mixture of two diastereoisomers (**17a** and **17b**) in which many individual NMR signals could be identified, indicating that **17a**:**17b** = 42:58. IR (FT) ν/cm^{-1}: 3304, 2872, 1677, 1604, 1512, 1273, 1048; MS (EI) *m*/*z*: 163 (46%), 136 (54), 135 (100), 119 (8), 107 (10), 92 (11), 77 (15); MS (CI) *m*/*z*: 300 ([MH]$^{+}$, 100%), 284 (8), 237 (24); HRMS (CI): Calc for $C_{17}H_{18}NO_4$ [MH^{+}]: 300.1230; Found: 300.1231; Crystallization of the crude product provided crystals of **17a**, mp 199–201 °C. Compound **17a** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.77 (s, exch., 1H, NH), 7.31 (t, *J* = 8 Hz, 1H, H-6), 7.16 (d, *J* = 8 Hz, 1H, H-5), 6.93 (d, *J* = 8 Hz, 1H, H-7), 6.89 (d, *J* = 9 Hz, 2H, H-2/H-6 of 4-methoxyphenyl group), 6.58 (d, *J* = 9 Hz, 2H, H-3/H-5 of 4-methoxyphenyl group), 5.73 (d, *J* = 3 Hz, exch., 1H, OH), 5.39 (t, *J* = 3 Hz, 1H, C*H*OH), 4.89 (d, *J* = 3 Hz, 1H, H-3), 3.97 (s, 3H, OCH_3), 3.60 (s, 3H, OCH_3); ^{13}C NMR

(100 MHz, DMSO-d_6) δ (ppm): 169.7 (s, C-1), 158.4 (s, C-4 of 4-methoxyphenyl), 155.0 (s, C-4), 134.9 (s, C-1 of 4-methoxyphenyl), 131.6 (s, C-7a), 131.5 (s, C-3a), 130.1 (d, C-6), 128.3 (d, C-2/C-6 of 4-methoxyphenyl), 114.9 (d, C-7), 113.6 (d, C-5), 112.5 (d, C-3/C-5 of 4-methoxyphenyl), 71.6 (d, CHOH), 61.5 (d, C-3), 56.0 (q, OCH_3), 55.1 (q, OCH_3); Compound **17b** (α-(*R**)-3-(*R**)-isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.01 (s, exch., 1H, NH), 7.31 (t, *J* = 8 Hz, 1H, H-6), 7.20 (d, *J* = 8 Hz, 1H, H-5), 6.90 (d, *J* = 8 Hz, 1H, H-7), 6.89 (d, *J* = 9 Hz, 2H, H-2/H-6 of 4-methoxyphenyl group), 6.58 (d, *J* = 9 Hz, 2H, H-3/H-5 of 4-methoxyphenyl group), 5.18 (dd, *J* = 3, 6 Hz, 1H, C*H*OH), 5.12 (d, *J* = 6 Hz, 1H, H-3), 4.76 (d, *J* = 3 Hz, exch., 1H, OH), 3.85 (s, 3H, OCH_3), 3.75 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 170.4 (s, C-1), 158.7 (s, C-4 of 4-methoxyphenyl), 155.2 (s, C-4), 135.7 (s, C-1 of 4-methoxyphenyl), 135.4 (s, C-7a), 133.1 (s, C-3a), 130.2 (d, C-6), 127.7 (d, C-2/C-6 of 4-methoxyphenyl), 115.0 (d, C-7), 113.8 (d, C-5), 112.6 (d, C-3/C-5 of 4-methoxyphenyl), 70.6 (d, CHOH), 62.0 (d, C-3), 56.0 (q, OCH_3), 55.5 (q, OCH_3).

3-(Hydroxy(phenyl)methyl)-4-methoxyisoindolin-1-one (18). Yield: 0.43 g (1.60 mmol, 80%); Product **18** was a mixture of two diastereoisomers (**18a** and **18b**) in which many individual NMR signals could be identified, indicating that **18a**:**18b** = 44:56. IR (FT) ν/cm^{-1}: 3201, 3070, 1672, 1603, 1492, 1269, 1053; MS (EI) *m*/*z*: 163 (22%), 132 (5), 116 (15), 105 (14), 86 (24), 84 (30), 51 (56), 49 (100); MS (CI) *m*/*z*: 270 ([MH]$^+$, 15%) 181 (7), 164 (100), 148 (8), 134 (7), 124 (14), 105 (23), 94 (13), 78 (12), 58 (28), 44 (27); HRMS (CI): Calc for $C_{16}H_{16}NO_3$ [MH$^+$]: 270.1125; Found: 270.1125; Crystallization of the crude product provided crystals of **18a**, mp 213–214 °C. Compound **18a** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.79 (s, exch., 1H, NH), 7.31 (t, *J* = 8 Hz, 1H, H-6), 7.16 (d, *J* = 8 Hz, 1H, H-7), 7.03–6.97 (m, 5H, Ph), 6.91 (d, *J* = 8 Hz, 1H, H-5), 5.82 (d, *J* = 3 Hz, exch., 1H, OH), 5.43 (t, 1H, *J* = 3 Hz, C*H*OH), 4.92 (d, *J* = 3 Hz, 1H, H-3), 3.98 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 175.0 (s, C-1), 160.3 (s, C-4), 144.8 (s, C-1 of Ph), 140.1 (s, C-7a), 136.8 (s, C-3a), 135.4 (d, C-6), 132.6 (d, C-4 of Ph), 132.5 (d, C-2/C-6 of Ph), 132.4 (d, C-3/C-5 of Ph), 120.1 (d, C-7), 118.8 (d, C-5), 77.2 (d, CHOH),

66.6 (d, C-3), 61.2 (q, OCH_3); Compound **18b** (α-(*R**)-3-((*R**)- isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.79 (s, exch., 1H, NH), 7.45-7.33 (m, 7H, H-6, H-7 and Ph), 7.17 (d, *J* = 8 Hz, 1H, H-5), 5.65 (d, *J* = 3 Hz, exch., 1H, OH), 5.22 (dd, 1H, *J* = 3, 6 Hz, C*H*OH), 4.96 (d, *J* = 6 Hz, 1H, H-3), 3.86 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 170.8 (s, C-1), 155.3 (s, C-4), 143.4 (s, C-1 of Ph), 139.7 (s, C-7a), 135.7 (s, C-3a), 130.1 (d, C-6), 128.4 (d, C-2/C-6 of Ph), 127.4 (d, C-4 of Ph), 126.6 (d, C-3/C-5 of Ph), 115.0 (d, C-7), 113.9 (d, C-5), 71.0 (d, CHOH), 61.8 (d, C-3), 56.0 (q, OCH_3).

Isoindolin-1-one (20). Yield: 0.19 g (1.43 mmol, 71%); mp 153–154 °C (Lit [78] 149–151 °C); IR (FT) ν/cm^{-1}: 3288, 2964, 1676, 1570, 1458, 1272, 1052; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.82 (d, *J* = 8 Hz, 1H, H-7), 7.66 (br, exch., 1H, NH), 7.49 (dt, *J* = 2, 8 Hz, 1H, H-5), 7.43–7.40 (m, 2H, H-4 and H-6), 4.41 (s, 2H, H-3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 172.0 (s, C-1), 143.7 (s, C-3a), 132.2 (s, C-7a), 131.8 (d, C-5), 128.0 (d, C-4), 123.8 (d, C-7), 123.2 (d, C-6), 45.7 (t, C-3); MS (APCI) *m*/*z*: 134 ([MH]$^+$, 100%); HRMS (APCI): Calc for C_8H_8NO [MH]$^+$: 134.0606; Found: 134.0604.

3-Methylisoindolin-1-one (21). Yield: 0.22 g (1.50 mmol, 75%); mp 117–118 °C (Lit [79] 115–116 °C); IR (FT) ν/cm^{-1}: 3176, 2934, 1678, 1602, 1457, 1265, 1043; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.35 (br, exch., 1H, NH), 7.77 (d, *J* = 8 Hz, 1H, H-7), 7.49 (dt, *J* = 2, 8 Hz, 1H, H-5), 7.38–7.34 (m, 2H, H-4 and H-6), 4.63 (q, *J* = 7 Hz, 1H, H-3), 1.43 (d, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.3 (s, C-1), 149.0 (s, C-3a), 131.8 (s, C-7a), 131.7 (d, C-5), 128.0 (d, C-4), 123.6 (d, C-7), 122.2 (d, C-6), 52.8 (d, C-3), 20.2 (q, CH_3); MS (ES$^+$) *m*/*z*: 295 ([2 M]$^+$, 9%), 189 ([M + MeCNH]$^+$, 100), 148 ([MH]$^+$, 34); HRMS (ES$^+$): Calc for $C_9H_{10}NO$ [MH]$^+$: 148.0762; Found: 148.0762.

3-Ethylisoindolin-1-one (22). Yield: 0.25 g (1.55 mmol, 77%); mp 103–105 °C (Lit [79] 104–105 °C); IR (FT) ν/cm^{-1}: 3312, 2945, 1677, 1589, 1472, 1267, 1043; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.46 (br, exch., 1H, NH), 7.76 (d, *J* = 8 Hz, 1H, H-7), 7.46 (dt, *J* = 2, 8 Hz, 1H, H-5), 7.35 (t, *J* = 8 Hz, 1H, H-6), 7.21 (d, *J* = 8 Hz, 1H, H-4), 4.52 (dd, *J* = 5, 7 Hz, 1H, H-3), 1.94 (ddq, *J* = 5, 14, 7 Hz, 1H, 1H of CH_2), 1.62 (d quintet, *J* = 14, 7 Hz, 1H, 1H of CH_2), 0.87 (t, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.6 (s, C-1), 147.6 (s, C-3a), 132.3 (s, C-7a), 131.7 (d, C-5), 128.5 (d, C-4), 123.6 (d, C-7), 122.4 (d, C-6), 58.2 (d, C-3), 27.3 (t, CH_2), 9.5 (q, CH_3); MS (APCI) *m*/*z*: 203 ([M + MeCNH]$^+$, 37%), 162 ([MH]$^+$, 100); HRMS (APCI): Calc for $C_{10}H_{12}NO$ [MH]$^+$: 162.0913; found: 162.0919.

3-Butylisoindolin-1-one (23). Yield: 0.29 g (1.53 mmol, 76%); mp 88–89 °C (Lit [79] 88–89 °C); IR (FT) ν/cm^{-1}: 3312, 2960, 1677, 1596, 1475, 1272, 1045; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.77 (d, *J* = 8 Hz, 1H, H-7), 7.66 (br, exch., 1H, NH), 7.49 (dt, *J* = 2, 8 Hz, 1H, H-5), 7.40–7.36 (m, 2H, H-4 and H-6), 4.55 (dd, *J* = 4, 7 Hz, 1H, H-3), 1.88 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.59 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.30–1.23 (m, 4H, C*H*$_2$C*H*$_2$CH$_3$), 0.82 (t, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.2 (s, C-1), 147.8 (s, C-3a), 132.0 (s, C-7a), 131.7 (d, C-5), 128.0 (d, C-4), 123.7 (d, C-7), 122.4 (d, C-6), 57.0 (d, C-3), 34.3 (t, *C*H$_2$CH$_2$CH$_2$CH$_3$), 27.6 (t, *C*H$_2$CH$_2$CH$_3$), 22.6 (t, *C*H$_2$CH$_3$), 13.9 (q, CH_3); MS (ES$^+$) *m*/*z*: 189 ([M]$^+$, 25%), 177 (56), 132 (100), 104 (23), 72 (32); HRMS (ES$^+$): Calc for $C_{12}H_{15}NO$ [M]$^+$: 189.1154; Found: 189.1154.

3-(Hydroxydiphenylmethyl)isoindolin-1-one (24). Yield: 0.47 g (1.49 mmol, 74%); mp 189–191 °C; IR (FT) ν/cm^{-1}: 3202, 2990, 1678, 1590, 1491, 1252, 1025; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.20 (br, exch., 1H, NH), 7.60 (d, *J* = 8 Hz, 1H, H-7), 7.52 (t, *J* = 8 Hz, 1H, H-5), 7.35 (d, *J* = 8 Hz, 1H, H-4), 7.30–7.00 (m, 11H, H-6 and 2 Ph), 6.44 (d, *J* = 6 Hz, 1H, H-3), 6.19 (s, exch., 1H, OH); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.3 (s, C-1), 145.2, 145.1 (2 s, C-1 of 2 Ph), 141.2 (s, C-3a), 134.2 (s, C-7a), 130.9 (d, C-5), 129.6 (d, C-4), 128.3, 128.2 (2 d, C-3/C-5 of

2 Ph), 127.4, 127.3 (2 d, C-2/C-6 of Ph), 126.4, 126.3 (2 d, C-4 of 2 Ph), 124.6 (d, C-7), 122.8 (d, C-6), 79.1 (s, C-OH), 67.5 (d, C-3); MS (ES^+) *m*/*z*: 316 ($[MH]^+$, 3%), 315 ($[M]^+$, 2), 297 (37), 268 (10), 182 (100), 133 (95), 105 (97); HRMS (ES^+): Calc for $C_{21}H_{18}NO_2$ $[MH]^+$: 316.1338; Found: 316.1338.

3-(Hydroxy(phenyl)methyl)isoindolin-1-one (25). Yield: 0.35 g (1.46 mmol, 73%); Product **25** was a mixture of two diastereoisomers (**25a** and **25b**) in which many individual NMR signals could be identified, indicating that **25a**:**25b** = 58:42. IR (FT) ν/cm^{-1}: 3237, 2957, 1669, 1599, 1512, 1050; MS (EI) *m*/*z*: 239 ($[M]^+$, 4%), 221 (100), 220 (51), 193 (33), 165 (89), 149 (86); MS (CI) *m*/*z*: 257 ($[M + NH_4]^+$, 12%), 240 ($[MH]^+$, 100), 224 (24); HRMS (CI): Calc for $C_{15}H_{14}NO_2$ $[MH]^+$: 240.1019; Found: 240.1018. Crystallization of the crude product provided crystals of **25a**, mp 174–175 °C. Compound **25a** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.69 (br, exch., 1H, NH), 7.48 (dd, *J* = 1, 8 Hz, 1H, H-7), 7.31 (dt, *J* = 1, 8 Hz, 1H, H-5), 7.37 (t, *J* = 8 Hz, 1H, H-6), 7.21 (br, 5H, Ph), 7.07 (d, *J* = 8 Hz, 1H, H-4), 5.92 (d, *J* = 4 Hz, exch., 1H, OH), 4.81 (d, *J* = 6 Hz, 1H, H-3), 4.73 (dd, *J* = 4, 6 Hz, 1H, C*H*OH); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 169.5 (s, C-1), 144.5 (s, C-1 of Ph), 141.1 (s, C-3a), 133.6 (s, C-7a), 131.0 (d, C-5), 128.3 (d, C-4), 127.8 (d, C-3/C-5 of Ph), 127.7 (d, C-2/C-6 of Ph), 127.6 (d, C-4 of Ph), 124.7 (d, C-7), 122.8 (d, C-6), 74.8 (d, CHOH), 61.9 (d, C-3); Compound **25b** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.38 (br, exch., 1H, NH), 7.72 (dd, *J* = 1, 8 Hz, 1H, H-7), 7.41 (dt, *J* = 1, 8 Hz, 1H, H-5), 7.30 (t, *J* = 8 Hz, 1H, H-6), 7.21-6.88 (m, 6H, H-4 and Ph), 5.60 (d, *J* = 4 Hz, exch., 1H, OH), 4.83 (t, *J* = 4 Hz, 1H, C*H*OH), 4.62 (d, *J* = 4 Hz, 1H, H-3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 169.2 (s, C-1), 144.1 (s, C-1 of Ph), 140.7 (s, C-3a), 134.2 (s, C-7a), 133.2 (d, C-5), 130.6 (d, C-4), 129.4 (d, C-3/C-5 of Ph), 129.1 (d, C-2/C-6 of Ph), 127.0 (d, C-4 of Ph), 124.0 (d, C-7), 122.4 (d, C-6), 74.5 (d, CHOH), 61.5 (d, C-3).

3-(Hydroxy(4-methoxyphenyl)methyl)isoindolin-1-one (26). Yield: 0.42 g (1.56 mmol, 78%); Product **26** was a mixture of two diastereoisomers (**26a** and **26b**) in which many individual NMR signals could be identified, indicating that **26a**:**26b** = 45:55. IR (FT) ν/cm^{-1}: 3301, 2886, 1678, 1602, 1510, 1271, 1042; MS (ES$^+$) *m*/*z*: 561 ([2 M + Na]$^+$, 12%), 539 ([2 M + 1]$^+$, 100), 311 ([M + MeCNNa]$^+$, 17), 270 ([M + MeCNH]$^+$, 17), 270 ([MH]$^+$, 100), 252 (58), 175 (6); HRMS (ES$^+$): Calc for $C_{16}H_{16}NO_3$ [MH]$^+$: 270.1130; Found: 270.1121. Crystallization of the crude product provided crystals of **26a**, mp 196–198 °C. Compound **26a** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.55 (br, exch., 1H, NH), 7.51–7.37 (m, 4H, H-4, H-5, H-6 and H-7), 7.13 (d, *J* = 9 Hz, 2H, H-2/H-6 of 4-methoxyphenyl group), 6.77 (d, *J* = 9 Hz, 2H, H-3/H-5 of 4-methoxyphenyl group), 5.69 (d, *J* = 4 Hz, exch., 1H, OH), 4.95 (t, *J* = 4 Hz, 1H, C*H*OH), 4.83 (d, *J* = 4 Hz, 1H, H-3), 3.70 (s, 3H, OCH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.0 (s, C-1), 158.8 (s, C-4 of 4-methoxyphenyl), 145.1 (s, C-3a), 133.6 (s, C-1 of 4-methoxyphenyl), 133.1 (s, C-7a), 131.2 (d, C-5), 128.6 (d, C-2/C-6 of 4-methoxyphenyl), 128.3 (d, C-7), 124.5 (d, C-4), 122.9 (d, C-6), 113.3 (d, C-3/C-5 of 4-methoxyphenyl), 73.9 (d, CHOH), 62.5 (d, C-3), 55.4 (q, OCH_3); Compound **26b** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6): δ = 8.67 (br, exch., 1 H, NH), 7.51–7.38 (m, 4 H, H-4, H-5, H-6 and H-7), 7.06 (d, *J* = 9 Hz, 2 H, H-2/H-6 of 4-methoxyphenyl group), 6.79 (d, *J* = 9 Hz, 2 H, H-3/H-5 of 4-methoxyphenyl group), 5.82 (d, *J* = 4 Hz, exch., 1 H, OH), 4.77 (d, *J* = 6 Hz, 1 H, H-3), 4.65 (dd, *J* = 4, 6 Hz, 1 H, C*H*OH), 3.68 (s, 3 H, OCH_3); ^{13}C NMR (125 MHz, DMSO-d_6): δ = 170.3 (s, C-1), 159.3 (s, C-4 of 4-methoxyphenyl), 145.0 (s, C-3a), 133.7 (s, C-1 of 4-methoxyphenyl), 133.4 (s, C-7a), 131.1 (d, C-5), 128.9 (d, C-2/C-6 of 4-methoxyphenyl), 128.4 (d, C-7), 124.8 (d, C-4), 122.9 (d, C-6), 113.4 (d, C-3/C-5 of 4-methoxyphenyl), 74.7 (d, CHOH), 62.1 (d, C-3), 55.4 (q, OCH_3).

6-Methoxyisoindolin-1-one (27). Yield: 0.23 g (1.41 mmol, 70%); mp 189–190 °C (a known compound but its mp was not reported [80,81]); IR (FT) ν/cm^{-1}: 3294, 2976, 1678, 1577, 1472, 1265, 1049; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.21 (br, exch., 1H, NH), 7.38 (d, *J* = 2 Hz, 1H,

H-7), 7.35 (d, *J* = 8 Hz, 1H, H-4), 7.14 (dd, *J* = 2, 8 Hz, 1H, H-5), 4.42 (s, 2H, H-3), 3.87 (s, 3H, OCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 172.2 (s, C-1), 160.0 (s, C-6), 135.9 (s, C-3a), 133.5 (s, C-7a), 124.0 (d, C-4), 120.3 (d, C-5), 106.3 (d, C-7), 55.7 (q, OCH_3), 45.4 (t, C-3); MS (ES^+) *m*/*z*: 205 ($[M + MeCNH]^+$, 100%), 164 ($[MH]^+$, 68); HRMS (ES^+): Calc for $C_9H_{10}NO_2$ $[MH]^+$:164.0706; Found: 164.0709.

6-Methoxy-3-methylisoindolin-1-one (28). Yield: 0.27 g (1.52 mmol, 76%); mp 160–161 °C; IR (FT) ν/cm^{-1}: 3165, 2979, 1679, 1600, 1487, 1261, 1046; ^1H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.21 (br, exch., 1H, NH), 7.33 (d, *J* = 2 Hz, 1H, H-7), 7.31 (d, *J* = 8 Hz, 1H, H-4), 7.13 (dd, *J* = 2, 8 Hz, 1H, H-5), 4.66 (q, *J* = 7 Hz, 1H, H-3), 3.86 (s, 3H, OCH_3), 1.48 (d, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.2 (s, C-1), 160.0 (s, C-6), 141.3 (s, C-3a), 133.0 (s, C-7a), 123.1 (d, C-4), 120.2 (d, C-7), 106.3 (d, C-5), 55.7 (q, OCH_3), 52.4 (d, C-3), 20.4 (q, CH_3); MS (ES^+) *m*/*z*: 219 (96%), 178 ($[MH]^+$, 100); HRMS (ES^+): Calc for $C_{10}H_{12}NO_2$ $[MH]^+$: 178.0863; Found: 178.0862.

3-Ethyl-6-methoxyisoindolin-1-one (29). Yield: 0.30 g (1.57 mmol, 78%); mp 137–138 °C; IR (FT) ν/cm^{-1}: 3300, 2942, 1678, 1598, 1475, 1270, 1045; ^1H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.16 (br, exch., 1H, NH), 7.33 (d, *J* = 8 Hz, 1H, H-4), 7.31 (s, 1H, H-7), 7.12 (dd, *J* = 2, 8 Hz, 1H, H-5), 4.56 (dd, *J* = 5, 7 Hz, 1H, H-3), 3.87 (s, 3H, OCH_3), 1.96 (ddq, *J* = 5, 14, 7 Hz, 1H, 1H of CH_2), 1.70 (d quintet, *J* = 14, 7 Hz, 1H, 1H of CH_2), 0.97 (t, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.4 (s, C-1), 160.0 (s, C-6), 139.8 (s, C-3a), 133.6 (s, C-7a), 123.2 (d, C-4), 120.1 (d, C-5), 106.3 (d, C-7), 57.8 (d, C-3), 55.7 (q, OCH_3), 27.5 (t, CH_2), 9.5 (q, CH_3); MS (EI) *m*/*z*: 191 ($[M]^+$, 21%), 162 (100), 147 (13), 134 (15), 119 (16); HRMS (EI): Calc for $C_{11}H_{13}NO_2$ $[M]^+$: 191.0946; Found: 191.0943.

3-Butyl-6-methoxyisoindolin-1-one (30). Yield: 0.34 g (1.55 mmol, 77%); mp 145–147 °C; IR (FT) ν/cm^{-1}: 3305, 2967, 1680, 1597, 1477, 1277, 1051; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.71 (br, exch., 1H, NH), 7.44 (d, *J* = 8 Hz, 1H, H-4), 7.15–7.03 (m, 2H, H-5 and H-7), 4.48 (dd, *J* = 3, 7 Hz, 1H, H-3), 3.81 (s, 3H, OCH_3), 1.84 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.48 (m, 1H, 1H of C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.31–1.21 (m, 4H, C*H*$_2$C*H*$_2$CH$_3$), 0.84 (t, *J* = 7 Hz, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 169.6 (s, C-1), 159.5 (s, C-6), 140.4 (s, C-3a), 134.3 (s, C-7a), 124.3 (d, C-4), 119.4 (d, C-5), 106.5 (d, C-7), 56.0 (d, C-3), 55.9 (q, OCH_3), 34.4 (t, *C*H$_2$CH$_2$CH$_2$CH$_3$), 27.3 (t, *C*H$_2$CH$_2$CH$_3$), 22.6 (t, *C*H$_2$CH$_3$), 14.3 (q, CH_3); MS (ES$^+$) *m*/*z*: 261 (100%), 220 ([MH]$^+$, 46), 204 (9), 164 (21); HRMS (ES$^+$): Calc for $C_{13}H_{18}NO_2$ [MH]$^+$: 220.1338; Found: 220.1347.

3-(Hydroxydiphenylmethyl)-6-methoxyisoindolin-1-one (31). Yield: 0.50 g (1.45 mmol, 72%); mp 159–160 °C; IR (FT) ν/cm^{-1}: 3205, 2994, 1677, 1594, 1493, 1255, 1027; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.12 (br, exch., 1H, NH), 7.71–7.16 (m, 10H, 2 Ph), 6.93 (d, *J* = 2 Hz, 1H, H-7), 6.75 (s, exch., 1H, OH), 6.69 (dd, *J* = 2, 8 Hz, 1H, H-5), 5.78 (d, *J* = 8 Hz, 1H, H-4), 5.40 (s, 1H, H-3), 3.68 (s, 3H, OCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 172.0 (s, C-1), 160.0 (s, C-6), 146.5, 144.7 (2 s, C-1 of 2 Ph), 136.4 (s, C-3a), 135.1 (s, C-7a), 128.6, 128.5 (2 d, C-3/C-5 of 2 Ph), 127.4, 127.4 (2 d, C-4 of 2 Ph), 126.3, 126.2 (2 d, C-2/C-6 of Ph), 124.6 (d, C-4), 119.2 (d, C-5), 106.6 (d, C-7), 78.4 (s, C-OH), 64.4 (d, C-3), 55.7 (q, OCH_3); MS (EI) *m*/*z*: 346 ([M + 1]$^+$, 33%), 345 ([M]$^+$, 10), 328 (45), 327 (100); MS (CI) *m*/*z*: 346 ([MH]$^+$, 100%), HRMS (CI): Calc for $C_{22}H_{20}NO_3$ [MH]$^+$: 346.1438; Found: 346.1443.

3-(Hydroxy(4-methoxyphenyl)methyl)-6-methoxyisoindolin-1-one (32). Yield: 0.45 g (1.50 mmol, 75%); Product **32** was a mixture of two diastereoisomers (**32a** and **32b**) in which many individual NMR signals could be identified, indicating that **32a**:**32b** = 62:38. IR (FT) ν/cm^{-1}: 3377, 2824, 1694, 1612, 1511, 1492, 1247, 1036; MS (EI) *m*/*z*: 163 (47%), 136 (52), 135 (100), 119 (5),

107 (20), 92 (26), 77 (53); MS (CI) *m/z*: 300 ([MH]$^+$, 100%), 181 (44); HRMS (CI): Calc for $C_{17}H_{18}NO_4$ [MH$^+$]: 300.1230; Found: 300.1225; Crystallization of the crude product provided crystals of **32a**, mp 172–175 °C. Compound **32a** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.69 (s, exch., 1H, NH), 7.12 (d, *J* = 9 Hz, 2H, H-2/H-6 of 4-methoxyphenyl group), 7.01-6.98 (m, 2H, H-5 and H-7), 6.86 (d, *J* = 8 Hz, 1H, H-4), 6.79 (d, *J* = 9 Hz, 2H, H-3/H-5 of 4-methoxyphenyl group), 5.79 (d, *J* = 4 Hz, exch., 1H, OH), 4.68 (d, *J* = 6 Hz, 1H, H-3), 4.56 (dd, *J* = 4, 6 Hz, 1H, C*H*OH), 3.75 (s, 3H, OCH_3), 3.71 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.4 (s, C-1), 159.8 (s, C-4 of 4-methoxyphenyl), 158.9 (s, C-6), 136.6 (s, C-1 of 4-methoxyphenyl), 135.1 (s, C-7a), 133.2 (s, C-3a), 128.6 (d, C-2/C-6 of 4-methoxyphenyl), 125.6 (d, C-4), 118.6 (d, C-7), 113.2 (d, C-3/C-5 of 4-methoxyphenyl), 106.1 (d, C-5), 74.9 (d, CHOH), 61.6 (d, C-3), 55.7 (q, OCH_3), 55.3 (q, OCH_3); Compound **32b** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.53 (s, exch., 1H, NH), 7.31 (d, *J* = 8 Hz, 1H, H-4), 7.05 (dd, *J* = 2, 8 Hz, 1H, H-5), 6.86 (d, *J* = 9 Hz, 2H, H-2/H-6 of 4-methoxyphenyl group), 6.80-6.76 (m, 3H, H-7 and H-3/H-5 of 4-methoxyphenyl group), 5.64 (d, *J* = 4 Hz, 1H, H-3), 4.90 (t, *J* = 4 Hz, 1H, C*H*OH), 4.74 (d, *J* = 4 Hz, exch., 1H, OH), 3.77 (s, 3H, OCH_3), 3.69 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm): 169.8 (s, C-1), 159.7 (s, C-4 of 4-methoxyphenyl), 158.7 (s, C-6), 137.1 (s, C-1 of 4-methoxyphenyl), 134.9 (s, C-7a), 133.0 (s, C-3a), 128.5 (d, C-2/C-6 of 4-methoxyphenyl), 125.3 (d, C-4), 118.7 (d, C-7), 113.1 (d, C-3/C-5 of 4-methoxyphenyl), 106.1 (d, C-5), 73.8 (d, CHOH), 61.9 (d, C-3), 55.7 (q, OCH_3), 55.2 (q, OCH_3).

6-Methylisoindolin-1-one (33). Yield: 0.22 g (1.50 mmol, 75%); mp 211–213 °C (Lit [82] 203–205 °C; Lit [83] 205 °C); IR (FT) ν/cm^{-1}: 3286, 2972, 1676, 1570, 1467, 1261, 1047; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.51 (br, exch., 1H, NH), 7.48 (s, 1H, H-7), 7.44 (d, *J* = 8 Hz, 1H, H-5), 7.39 (d, *J* = 8 Hz, 1H, H-4), 4.32 (s, 2H, H-3), 2.39 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.5 (s, C-1), 141.7 (s, C-3a), 137.6 (s, C-6), 133.2 (s, C-7a), 132.6 (d, C-5), 123.8 (d, C-7), 123.4 (d, C-4), 45.1 (t, C-3), 21.3 (q, CH_3); MS (ES$^+$) *m/z*: 295 ([2 M + 1]$^+$, 41%).

189 ([M + MeCNH]$^+$, 92), 148 ([MH]$^+$, 100); HRMS (ES$^+$): Calc for $C_9H_{10}NO$ [MH]$^+$: 148.0755; Found: 148.0762; Anal. Calc for C_9H_9NO: C, 73.45; H, 6.16; N, 9.52. Found: C, 73.32; H, 6.14; N, 9.41.

3,6-Dimethylisoindolin-1-one (34). Yield: 0.25 g (1.55 mmol, 78%); mp 161–162 °C; IR (FT) ν/cm^{-1}: 3187, 2986, 1677, 1598, 1478, 1266, 1042; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.59 (br, exch., 1H, NH), 7.45 (s, 1H, H-7), 7.44 (d, *J* = 8 Hz, 1H, H-5), 7.39 (d, *J* = 8 Hz, 1H, H-4), 4.56 (q, *J* = 6 Hz, 1H, H-3), 2.38 (s, 3H, CH_3), 1.33 (d, *J* = 6 Hz, 3H, C*H*$_3$CH); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.4 (s, C-1), 146.9 (s, C-3a), 137.7 (s, C-6), 132.8 (d, C-5), 132.7 (s, C-7a), 123.3 (d, C-7), 122.9 (d, C-4), 51.9 (d, C-3), 21.3 (q, CH_3), 20.8 (q, CH_3); MS (ES$^+$) *m*/*z*: 323 ([2 M + 1]$^+$, 22%), 203 ([M + MeCNH]$^+$, 96), 162 ([MH]$^+$, 100); HRMS (ES$^+$): Calc for $C_{10}H_{12}NO$ [MH]$^+$: 162.0925; Found: 162.0925.

3-Ethyl-6-methylisoindolin-1-one (35). Yield: 0.26 g (1.49 mmol, 75%); mp 165-166 °C; IR (FT) ν/cm^{-1}: 3307, 2949, 1679, 1597, 1470, 1272, 1049; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.27 (br, exch., 1H, NH), 7.58 (s, 1H, H-7), 7.28 (d, *J* = 8 Hz, 1H, H-5), 7.23 (d, *J* = 8 Hz, 1H, H-4), 4.49 (dd, *J* = 5, 7 Hz, 1H, H-3), 2.36 (s, 3H, CH_3), 1.91 (ddq, *J* = 5, 14, 7 Hz, 1H, 1H of CH_2), 1.62 (d quintet, *J* = 14, 7 Hz, 1H, 1H of CH_2), 0.89 (t, *J* = 7 Hz, 3H, C*H*$_3$CH$_2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 171.7 (s, C-1), 144.8 (s, C-3a), 137.9 (s, C-6), 132.7 (d, C-5), 132.4 (s, C-7a), 123.8 (d, C-4), 122.1 (d, C-7), 58.0 (d, C-3), 27.4 (t, CH_2), 21.3 (q, CH_3), 9.5 (q, *C*H$_3$CH$_2$); MS (APCI) *m*/*z*: 175 ([M]$^+$, 11%), 146 (100), 118 (12); HRMS (APCI): Calc for $C_{11}H_{13}NO$ [M]$^+$: 175.0997; Found: 175.1000.

3-(1-Hydroxycyclohexyl)-6-methylisoindolin-1-one (36). Yield: 0.35 g (1.43 mmol, 72%); mp 235–237 °C; IR (FT) ν/cm^{-1}: 3307, 2943, 1681, 1596, 1472, 1246, 1043; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.52 (br, exch., 1H, NH), 7.59 (d, *J* = 8 Hz, 1H, H-5), 7.41 (s, 1H, H-7), 7.35

(d, J = 8 Hz, 1H, H-4), 4.63 (s, exch., 1H, OH), 4.28 (s, 1H, H-3), 2.38 (s, 3H, CH_3), 1.61–0.93 (m, 10H, cyclohexyl); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.5 (s, C-1), 142.8 (s, C-3a), 137.6 (s, C-6), 133.8 (s, C-7a), 132.2 (d, C-5), 125.4 (d, C-4), 123.0 (d, C-7), 72.6 (s, C-1 of cyclohexyl group), 65.7 (d, C-3), 34.9, 30.4 (2 t, C-2/C-6 of cyclohexyl group), 25.9 (t, C-4 of cyclohexyl group), 21.31, 21.1 (2 t, C-3/C-5 of cyclohexyl group), 21.27;(q, CH_3); MS (ES^+) m/z: 491 ([2 M + 1]$^+$, 10%), 287 (79), 246 ([MH]$^+$, 100), 228 (18); HRMS (ES^+): Calc for $C_{15}H_{20}NO_2$ [MH]$^+$: 264.1494; Found: 246.1498; Anal. Calc for $C_{15}H_{19}NO_2$: C, 73.44; H, 7.81; N 5.71. Found: C, 73.81; H, 7.85; N 5.80.

3-(Hydroxydiphenylmethyl)-6-methylisoindolin-1-one (37). Yield: 0.56 g (1.70 mmol, 85%); mp 236–264 °C; IR (FT) ν/cm^{-1}: 3278, 3000, 1678, 1591, 1493, 1251, 1040; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.12 (br, exch., 1H, NH), 7.61–7.19 (m, 11H, 2 Ph and H-7), 7.11 (d, J = 8 Hz, 1H, H-5), 6.26 (d, J = 8 Hz, 1H, H-4), 5.81 (s, exch., 1H, OH), 5.72 (s, 1H, H-3), 2.33 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 172.4 (s, C-1), 145.5, 145.2 (2 s, C-1 of 2 Ph), 142.4 (s, C-3a), 137.6 (s, C-6), 134.4 (s, C-7a), 131.8 (d, C-5), 128.3, 128.2 (2 d, C-3/C-5 of 2 Ph), 127.3, 127.2 (2 d, C-4 of 2 Ph), 127.1, 127.0 (2 d, C-2/C-6 of 2 Ph), 124.3 (d, C-4), 123.0 (d, C-7), 79.0 (s, C-OH), 63.4 (d, C-3), 21.2 (q, CH_3); MS (ES^+) m/z: 659 ([2 M + 1]$^+$, 12%), 371 ([M + MeCNH]$^+$, 71), 330 ([MH]$^+$, 100), 312 (36), 189 (8); HRMS (ES^+): Calc for $C_{22}H_{20}NO_2$ [MH]$^+$: 330.1494; Found: 330.1487.

3-(1-Hydroxy-1-methylpentyl)-6-methylisoindolin-1-one (38). Yield: 0.38 g (1.54 mmol, 77%); Product **38** was a mixture of two diastereoisomers (**38a** and **38b**) in which many individual NMR signals could be identified, indicating that **38a**:**38b** = 55:45. IR (FT) ν/cm^{-1}: 3467, 3042, 1680, 1590, 1269, 1041; MS (ES^+) m/z: 517 ([2 M + Na]$^+$, 9%), 495 ([2 M + 1]$^+$, 44), 289 ([M + MeCNH]$^+$, 32), 248 ([MH]$^+$, 100), 230 (41), 148 (9), 100 (8); HRMS (ES^+): Calc for $C_{15}H_{22}NO_2$ [MH]$^+$: 248.1651; Found: 248.1646. Crystallization of the crude product provided crystals of **38a**,

mp 88–89 °C. Compound **38a** (α-(*R**)-3-(*R**)- isomer):^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.47 (br, exch., 1H, NH), 7.53 (d, *J* = 8 Hz, 1H, H-5), 7.42 (s, 1H, H-7), 7.35 (d, *J* = 8 Hz, 1H, H-4), 4.79 (s, exch., 1H, OH), 4.39 (br, 1H, H-3), 2.38 (s, 3H, CH_3), 1.33–1.09 (m, 6H, CH_2CH_2C$H_2$$CH_3$), 1.06 (s, 3H, C$H_3$C-OH), 0.79 (t, *J* = 7 Hz, 3H, C$H_3$$CH_2$); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.3 (s, C-1), 142.8 (s, C-3a), 137.7 (s, C-6), 133.9 (s, C-7a), 132.4 (d, C-5), 125.0 (d, C-4), 123.1 (d, C-7), 73.6 (s, C-OH), 65.5 (d, C-3), 36.3 (t, *C*$H_2$$CH_2$$CH_2$$CH_3$), 25.4 (t, *C*$H_2$$CH_2$$CH_3$), 24.6 (q, CH_3), 23.3 (t, *C*$H_2$$CH_3$), 21.3 (q, CH_3), 14.5 (q, CH_2*C*H_3); Compound **38b** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.59 (br, exch., 1H, NH), 7.56 (d, *J* = 8 Hz, 1H, H-5), 7.46 (s, 1H, H-7), 7.39 (d, *J* = 8 Hz, 1H, H-4), 4.88 (s, exch., 1H, OH), 4.23 (br, 1H, H-3), 2.26 (s, 3H, CH_3), 1.33–1.09 (m, 6H, CH_2CH_2C$H_2$$CH_3$), 1.00 (s, 3H, C$H_3$C-OH), 0.89 (t, *J* = 7 Hz, 3H, C$H_3$$CH_2$); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.8 (s, C-1), 142.7 (s, C-3a), 137.6 (s, C-6), 133.5 (s, C-7a), 132.5 (d, C-5), 124.9 (d, C-4), 122.8 (d, C-7), 73.8 (s, C-OH), 66.6 (d, C-3), 34.2 (t, *C*$H_2$$CH_2$$CH_2$$CH_3$), 25.3 (t, *C*$H_2$$CH_2$$CH_3$), 24.8 (q, CH_3), 23.6 (t, *C*$H_2$$CH_3$), 21.5 (q, CH_3), 14.4 (q, CH_2*C*H_3).

3-(Hydroxy(phenyl)methyl)-6-methylisoindolin-1-one (39). Yield: 0.40 g (1.58 mmol, 79%); Product **39** was a mixture of two diastereoisomers (**39a** and **39b**) in which many individual NMR signals could be identified, indicating that **39a**:**39b** = 54:48. IR (FT) ν/cm^{-1}: 3312, 2943, 1678, 1602, 1500, 1272, 1045; MS (APCI) *m*/*z*: 529 ([2 M + Na]$^{+}$, 61%), 507 ([2 M^{+} + 1], 93), 317 (15), 295 (22), 254 ([MH]$^{+}$, 89), 236 (100), 117 (12), 100 (22); HRMS (APCI): Calc for $C_{16}H_{16}NO_2$ [MH]$^{+}$: 254.1181; Found: 254.1182. Crystallization of the crude product provided crystals of **39a**, mp 228–229 °C. Compound **39a** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.02 (br, exch., 1H, NH), 7.41 (d, *J* = 2 Hz, 1H, H-7), 7.31–7.22 (m, 5H, Ph), 7.03 (dd, *J* = 2, 8 Hz, 1H, H-5), 6.33 (d, *J* = 8 Hz, 1H, H-4), 5.62 (d, *J* = 4 Hz, exch., 1H, OH), 4.67 (d, *J* = 7 Hz, 1H, H-3), 4.39 (dd, *J* = 4, 7 Hz, 1H, C*H*OH), 2.32 (s, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.3 (s, C-1), 141.1 (s, C-3a), 140.9 (s, C-1 of Ph), 137.9 (s, C-6), 133.3 (s, C-7a), 131.9 (d, C-5),

128.2 (d, C-7), 128.1 (d, C-3/C-5 of Ph), 127.7 (d, C-2/C-6 of Ph), 124.1 (d, C-4 of Ph), 123.2 (d, C-4), 76.9 (d, CHOH), 62.7 (d, C-3), 21.3 (q, CH_3); Compound **39b** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.00 (br, exch., 1H, NH), 7.50 (d, *J* = 2 Hz, 1H, H-7), 7.31–7.22 (m, 5H, Ph), 7.07 (dd, *J* = 2, 8 Hz, 1H, H-5), 6.28 (d, *J* = 8 Hz, 1H, H-4), 5.52 (d, *J* = 4 Hz, exch., 1H, OH), 4.73 (d, *J* = 4 Hz, 1H, H-3), 4.29 (t, *J* = 4 Hz, 1H, C*H*OH), 2.26 (s, 3H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.7 (s, C-1), 142.4 (s, C-3a), 142.1 (s, C-1 of Ph), 137.2 (s, C-6), 133.5 (s, C-7a), 132.5 (d, C-5), 128.6 (d, C-7), 128.0 (d, C-3/C-5 of Ph), 126.8 (d, C-2/C-6 of Ph), 125.7 (d, C-4 of Ph), 124.3 (d, C-4), 74.7 (d, CHOH), 61.4 (d, C-3), 21.2 (q, CH_3).

3-(Hydroxy(4-methoxyphenyl)methyl)-6-methylisoindolin-1-one (40). Yield: 0.47 g (1.66 mmol, 83%); Product **40** was a mixture of two diastereoisomers (**40a** and **40b**) in which many individual NMR signals could be identified, indicating that **40a**:**40b** = 42:58. IR (FT) ν/cm^{-1}: 3301, 2896, 1677, 1601, 1519, 1271, 1042; MS (ES$^+$) *m*/*z*: 589 ([2 M + Na]$^+$, 37%), 567 ([2 M + 1]$^+$, 64), 325 ([M + MeCNH]$^+$, 65), 284 ([MH]$^+$, 100), 266 (46); HRMS (ES$^+$): Calc for $C_{17}H_{18}NO_3$ [MH]$^+$: 284.1287; Found: 284.1290. Crystallization of the crude product provided crystals of **40a**, mp 211–213 °C.

Compound **40a** (α-(*R**)-3-(*S**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.47 (br, exch., 1H, NH), 7.31–7.29 (m, 3H, H-4, H-5 and H-7), 7.12 (d, *J* = 9, 2H, H-2/H-6 of 4-methoxyphenyl), 6.76 (d, *J* = 9, 2H, H-3/H-5 of 4-methoxyphenyl), 5.65 (d, *J* = 4 Hz, exch., 1H, OH), 4.92 (t, *J* = 4 Hz, 1H, C*H*OH), 4.77 (d, *J* = 4 Hz, 1H, H-3), 3.69 (s, 3H, OCH_3), 2.33 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 170.1 (s, C-1), 158.8 (s, C-4 of 4-methoxyphenyl), 142.3 (s, C-3a), 137.7 (s, C-6), 133.8 (s, C-7a), 133.2 (s, C-1 of 4-methoxyphenyl), 132.1 (d, C-5), 128.6 (d, C-2/C-6 of 4-methoxyphenyl), 124.2 (d, C-7), 123.1 (d, C-4), 113.3 (d, C-3/C-5 of 4-methoxyphenyl), 73.9 (d, CHOH), 62.3 (d, C-3), 55.4 (q, OCH_3), 21.3 (q, CH_3); Compound **40b** (α-(*R**)-3-(*R**)- isomer): ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 8.63 (br, exch., 1H, NH), 7.31–7.25 (m, 3H, H-4, H-5 and H-7), 7.13 (d, *J* = 9, 2H, H-2/H-6 of 4-methoxyphenyl), 6.79 (d, *J* = 9, 2H, H-3/H-5 of

4-methoxyphenyl), 5.79 (d, *J* = 4 Hz, exch., 1H, OH), 4.71 (d, *J* = 6 Hz, 1H, H-3), 4.58 (dd, *J* = 4, 6 Hz, 1H, C*H*OH), 3.70 (s, 3H, OCH_3), 2.32 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 169.2 (s, C-1), 158.5 (s, C-4 of 4-methoxyphenyl), 141.3 (s, C-3a), 137.2 (s, C-6), 133.4 (s, C-7a), 132.8 (s, C-1 of 4-methoxyphenyl), 131.5 (d, C-5), 128.4 (d, C-2/C-6 of 4-methoxyphenyl), 122.6 (d, C-7), 122.5 (d, C-4), 112.4 (d, C-3/C-5 of 4-methoxyphenyl), 74.8 (d, CHOH), 61.3 (d, C-3), 55.5 (q, OCH_3), 21.1 (q, CH_3).

3,3-Dibutylisoindolin-1-one (41). This compound was obtained as a side product, along with compound **40**, when 1-bromobutane was used as the electrophile; Yield: 25 mg (0.10 mmol, 5%); mp 88–89 °C; IR (FT) ν/cm^{-1}: 3300, 2960, 1681, 1591, 1473, 1273, 1046; ^1H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.74 (d, *J* = 8 Hz, 1H, H-7), 7.47 (t, *J* = 8 Hz, 1H, H-5), 7.36 (t, *J* = 8 Hz, 1H, H-6), 7.23 (d, *J* = 8 Hz, 1H, H-4), 7.16 (br s, exch., 1H, NH), 1.85–1.72 (m, 4H, 2 C$H_2$$CH_2CH_2CH_3$), 1.18–1.09 (m, 8H, 2 C$H_2CH_2$$CH_3$), 0.74 (t, *J* = 7 Hz, 6H, 2 CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.6 (s, C-1), 150.8 (s, C-3a), 133.3 (s, C-7a), 131.8 (d, C-5), 127.8 (d, C-4), 123.7 (d, C-7), 121.2 (d, C-6), 65.1 (s, C-3), 39.0 (t, 2 *C*$H_2CH_2CH_2CH_3$), 25.6 (t, 2 *C*$H_2CH_2CH_3$), 22.8 (t, 2 *C*H_2CH_3), 13.8 (q, 2 CH_3); MS (ES^+) *m*/*z*: 513 ($[2\ M + Na]^+$, 12%), 491 ($[2\ M + 1]^+$, 3), 309 ($[M + MeCNNa]^+$, 24), 287 ($[M + MeCNH]^+$, 97), 246 ($[MH]^+$, 100); HRMS (ES^+): Calc for $C_{16}H_{24}NO$ $[MH]^+$: 246.1858; Found: 246.1858.

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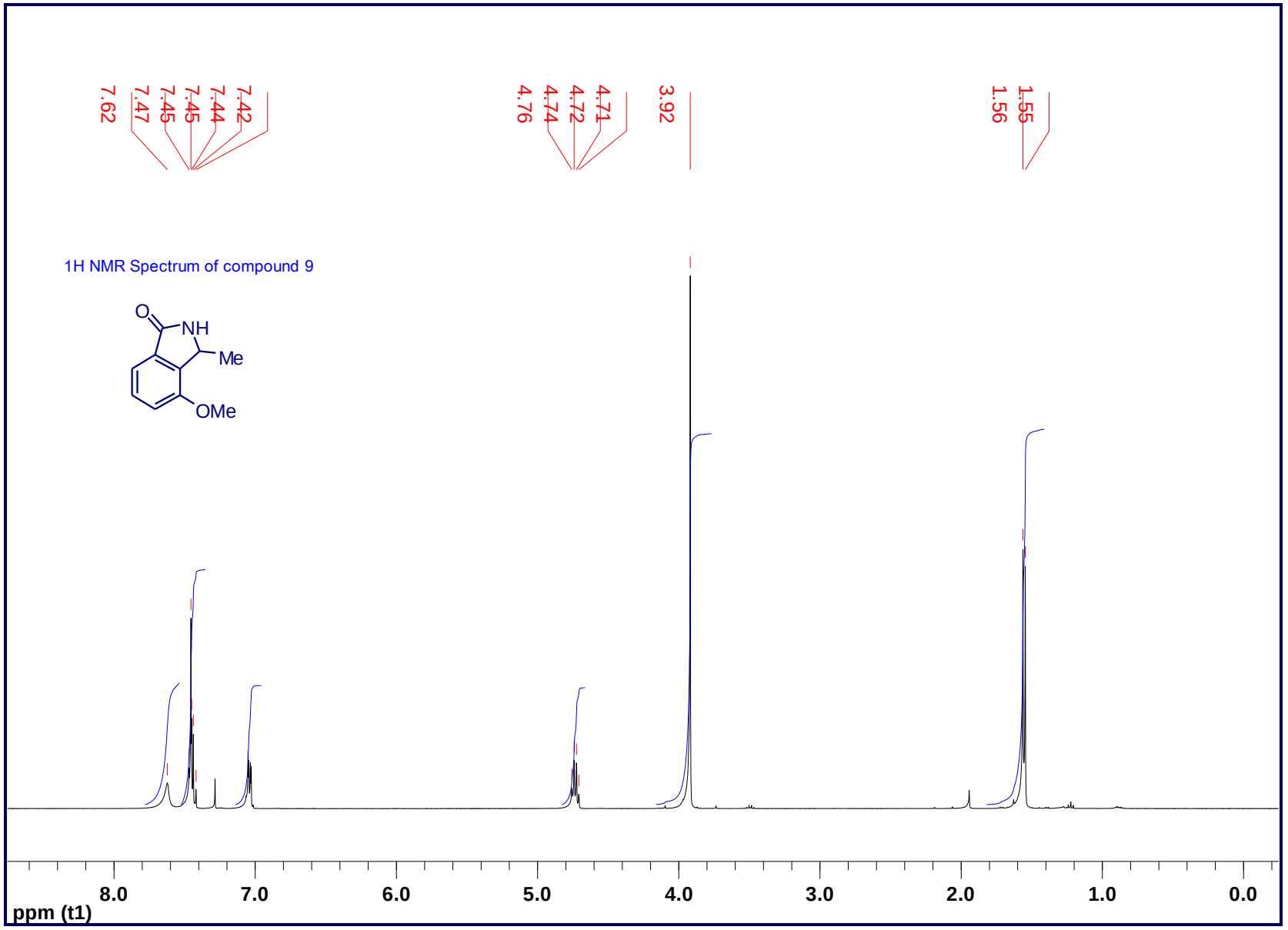
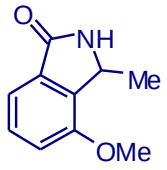
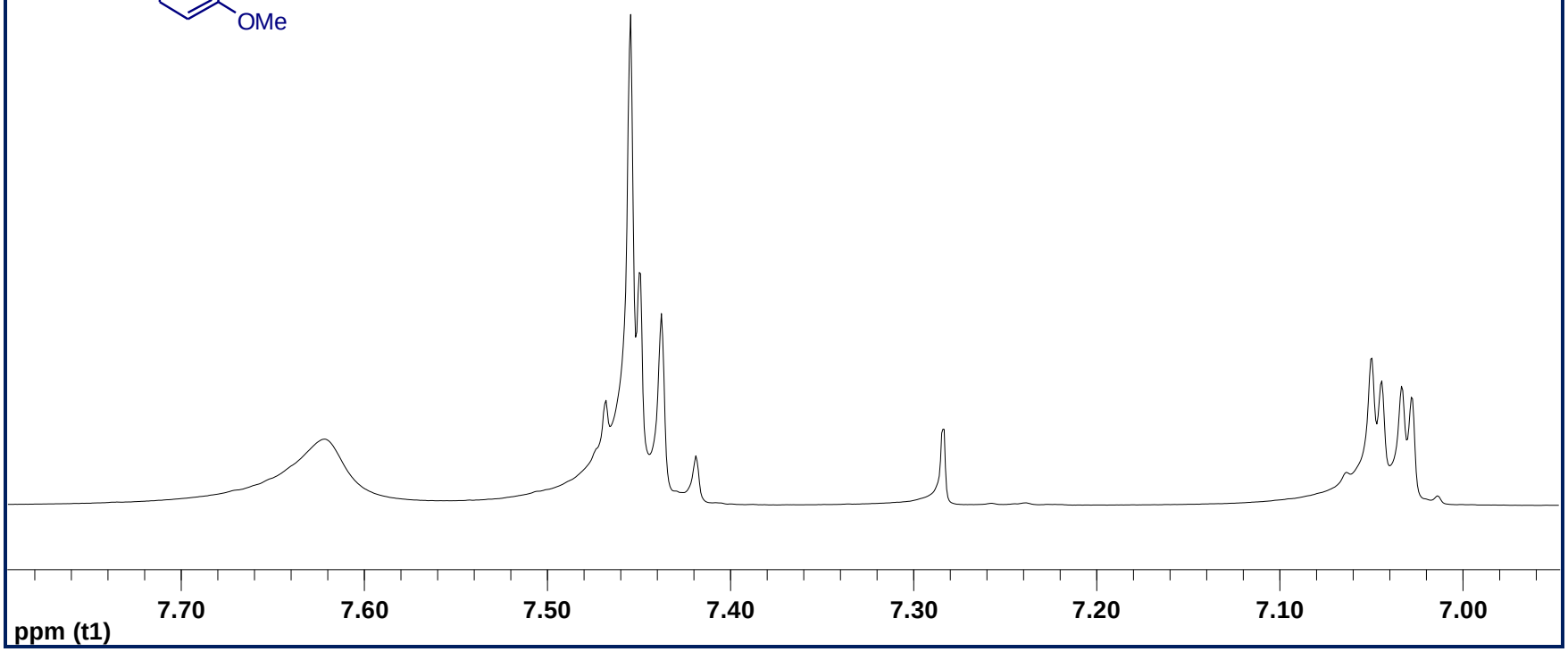
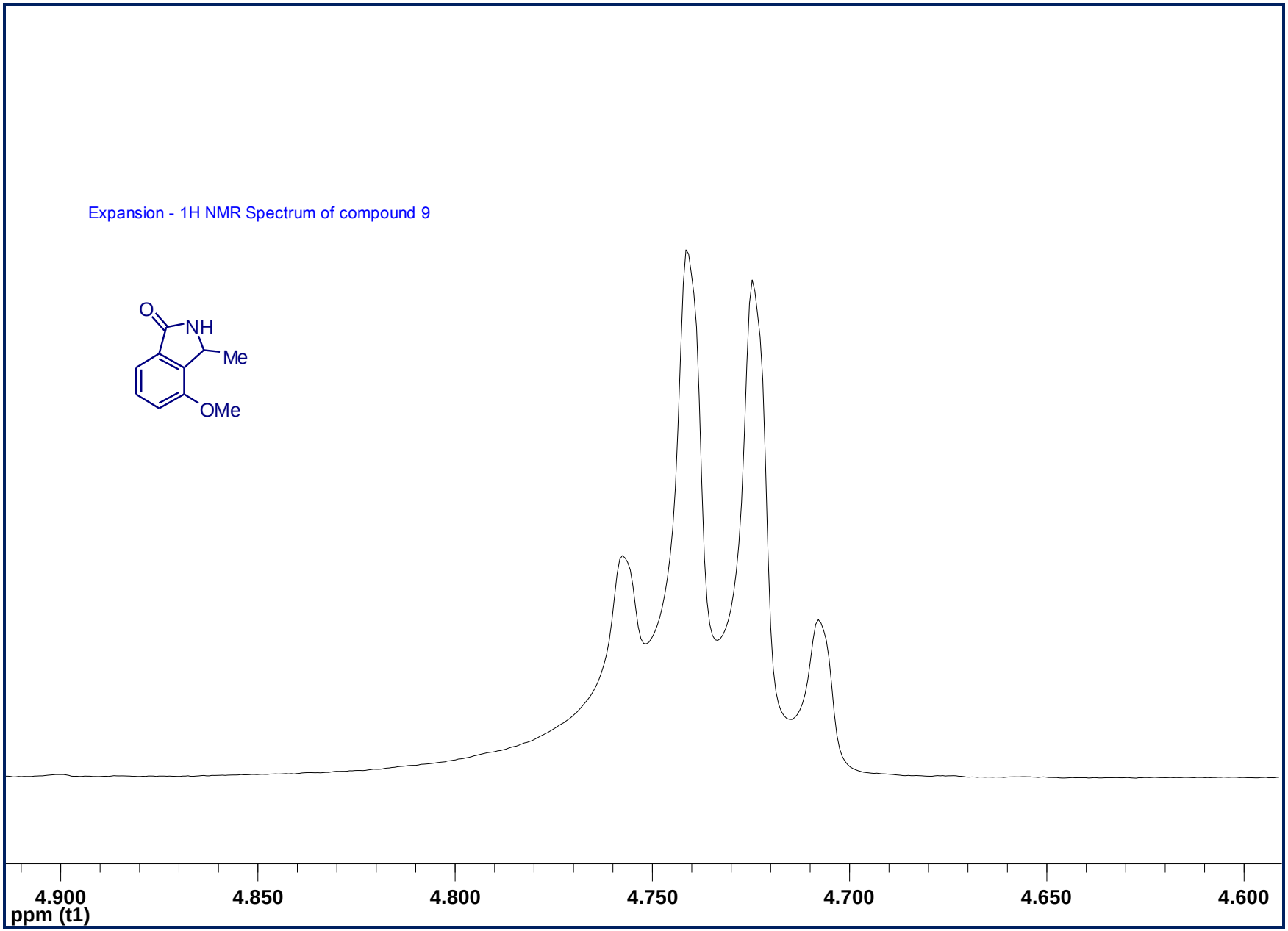
NMR Spectra for Some of the Synthesised Compounds

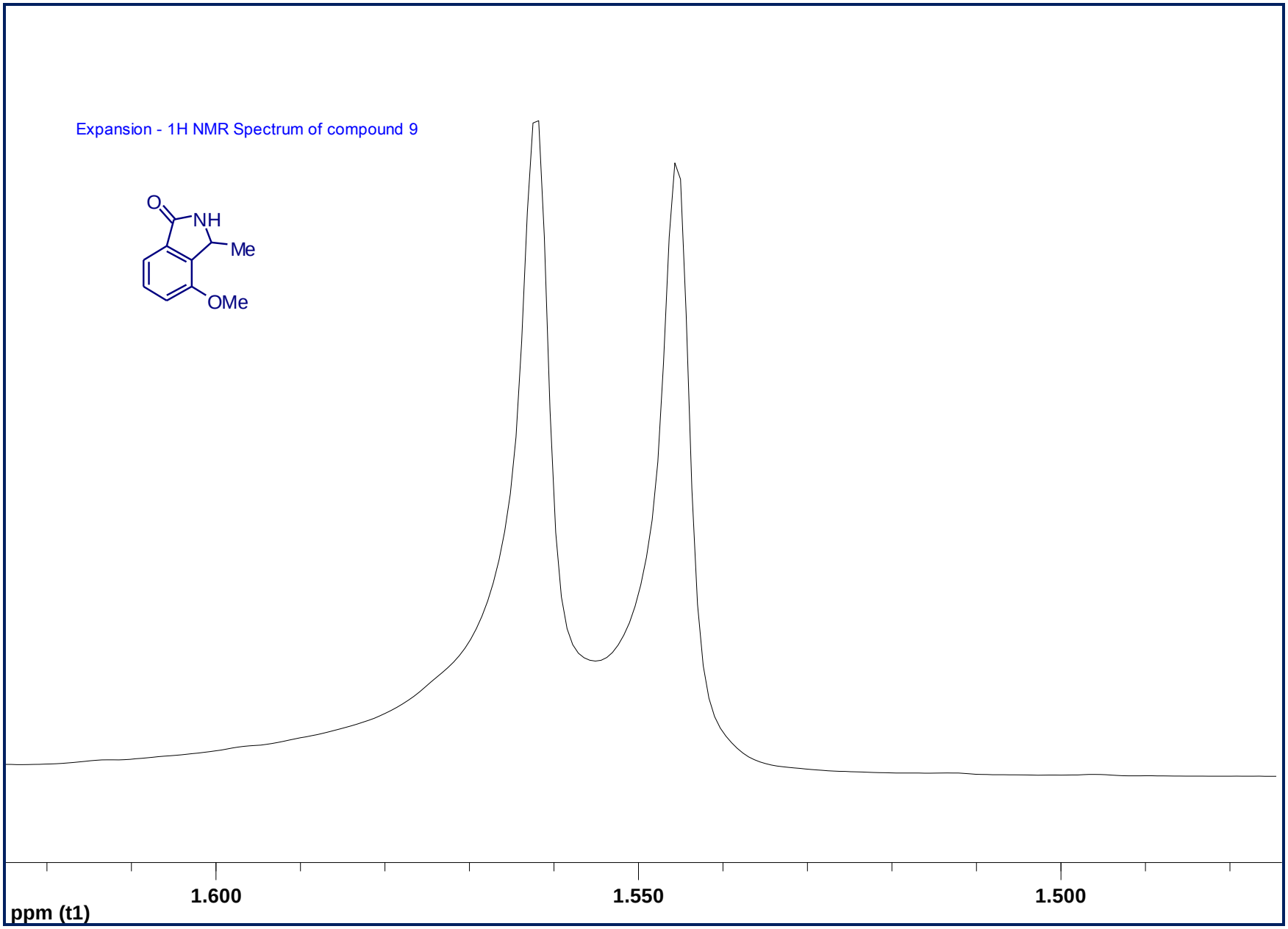

7.62
7.47
7.45
7.45
7.44
7.42
4.76
4.74
4.72
4.71
3.92
1.56
1.55
1H NMR Spectrum of compound 9
O
NH
Me
OMe
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

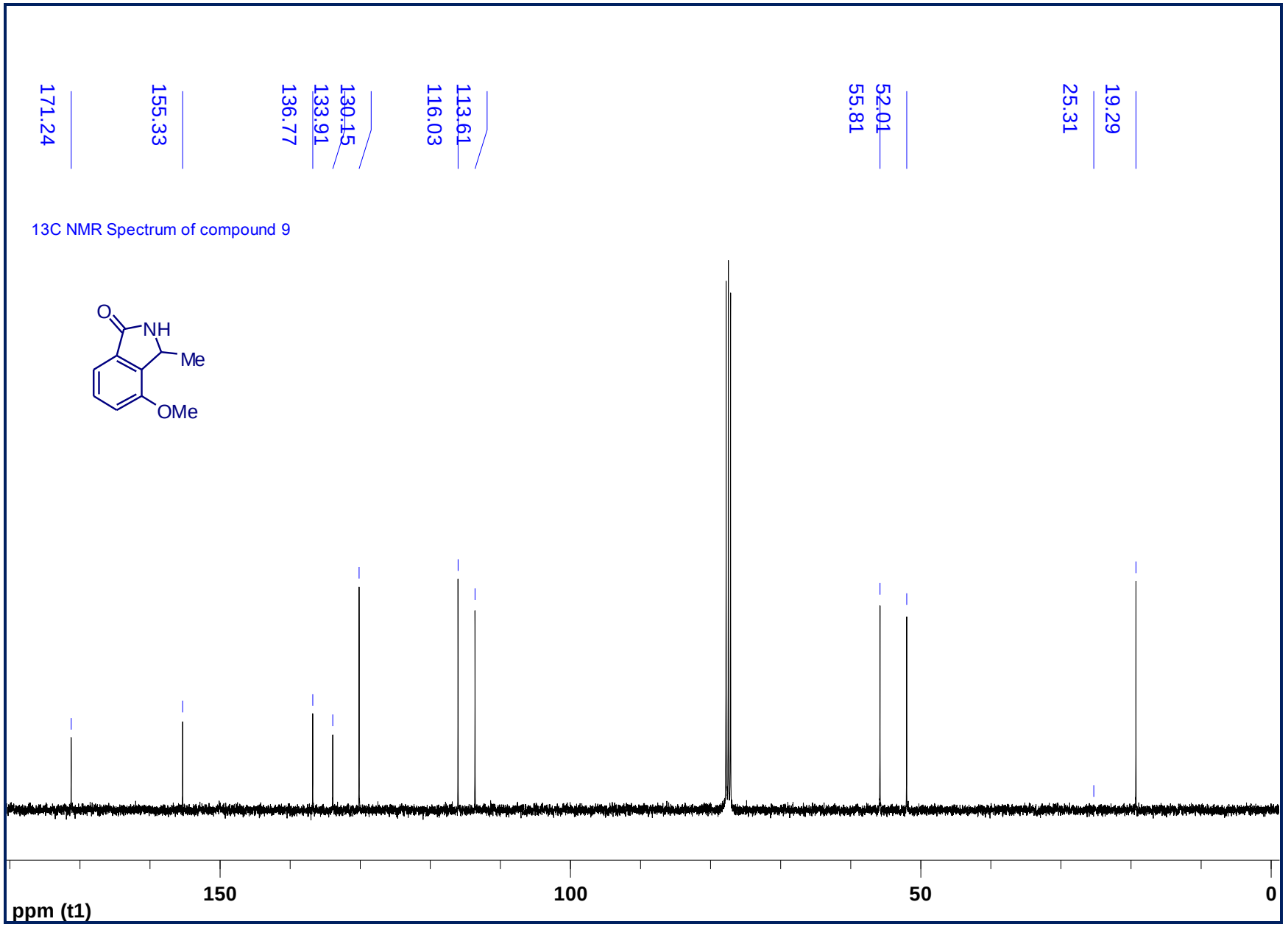
Expansion - 1H NMR Spectrum of compound 9

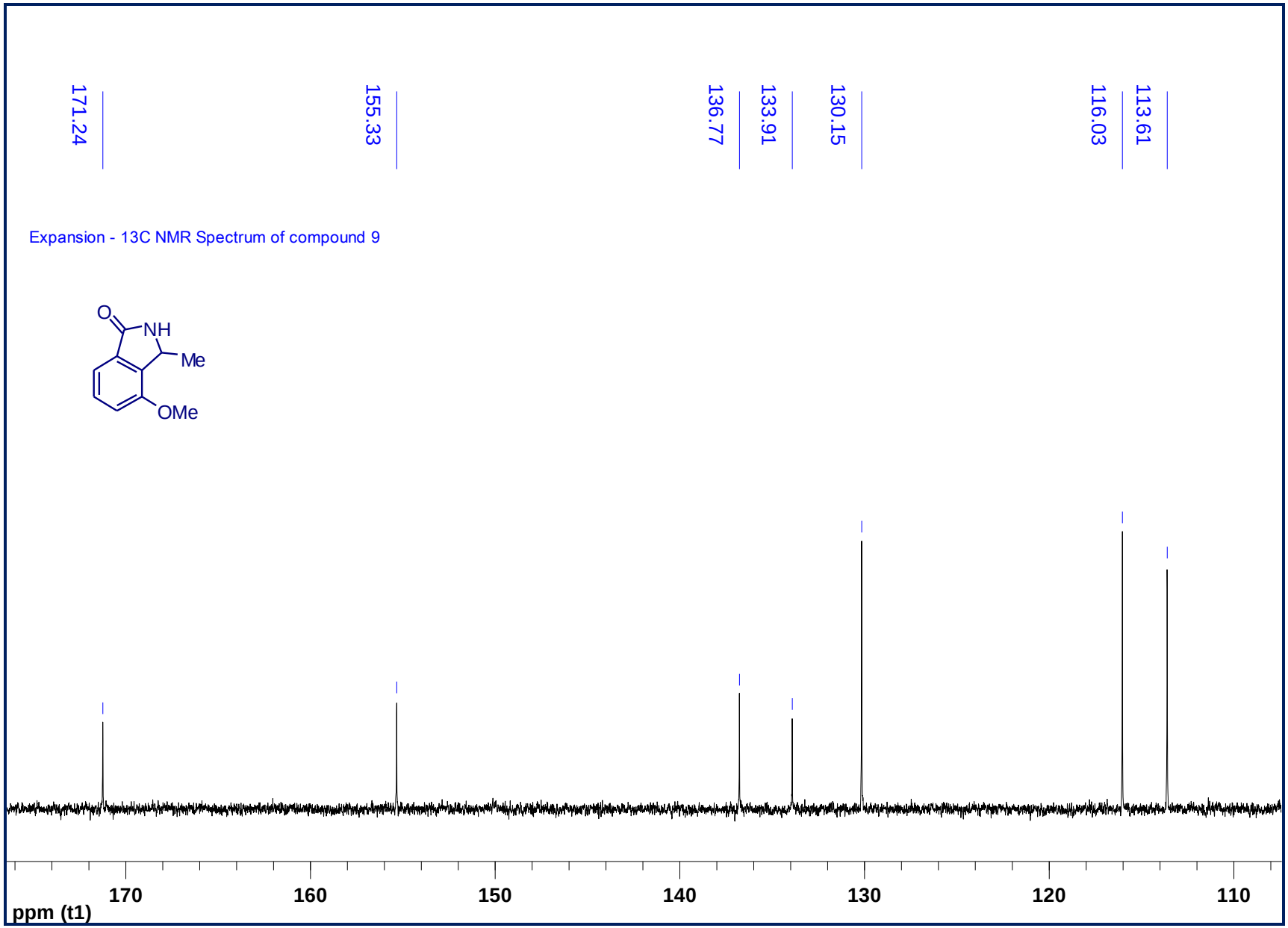


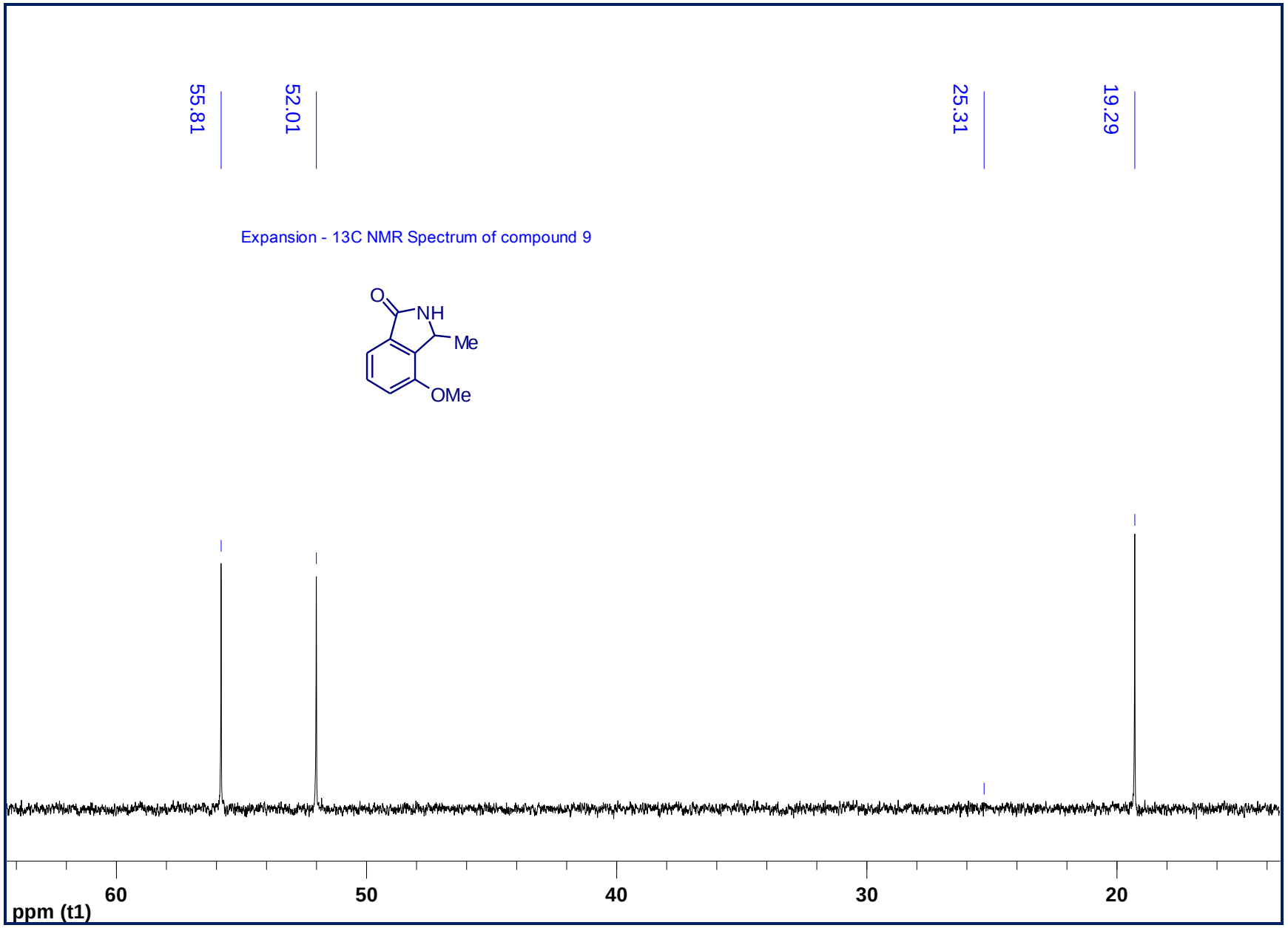


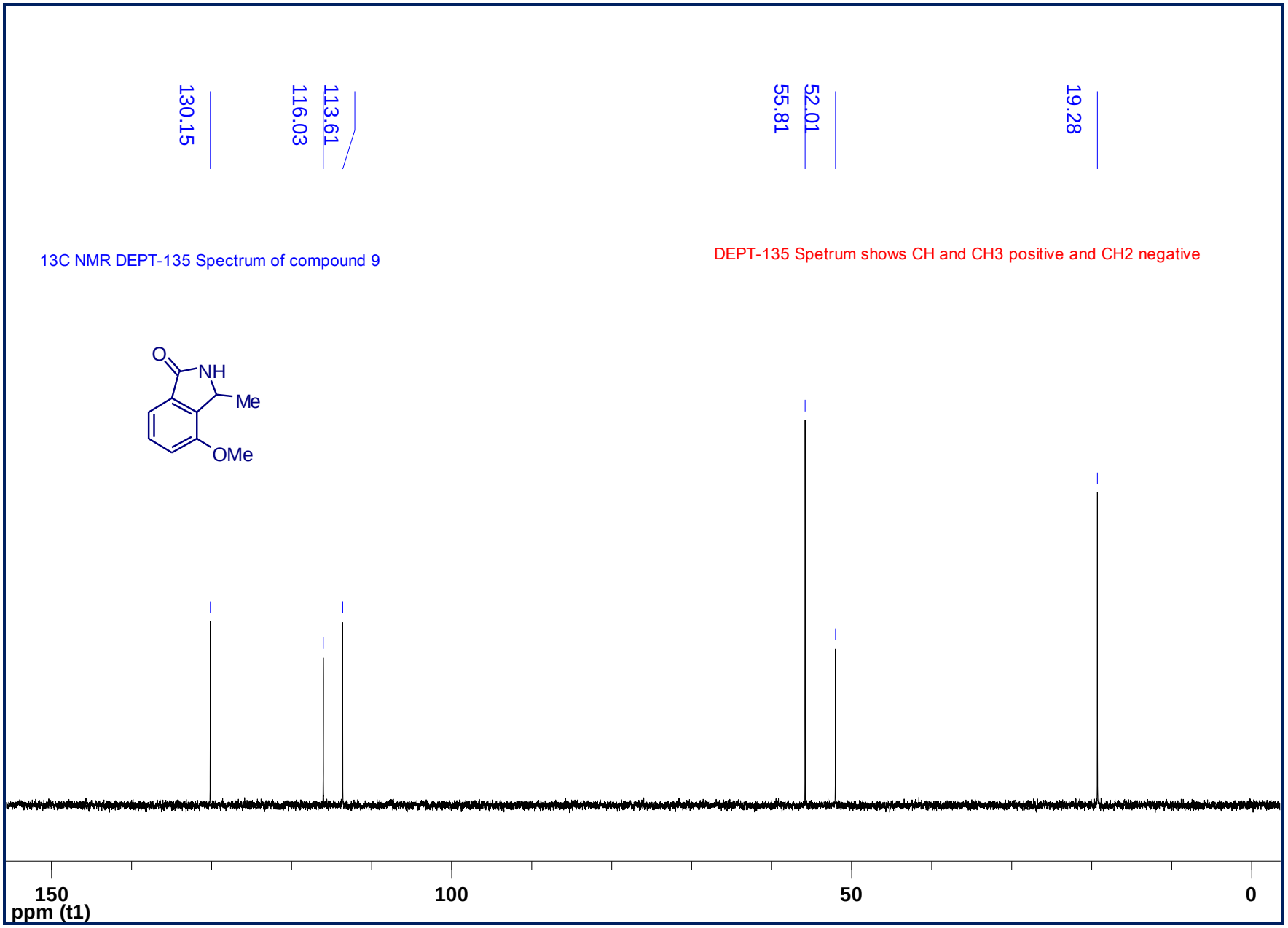

Expansion - 1H NMR Spectrum of compound 9
O
NH
Me
OMe
4.900
4.850
4.800
4.750
4.700
4.650
4.600
ppm (t1)

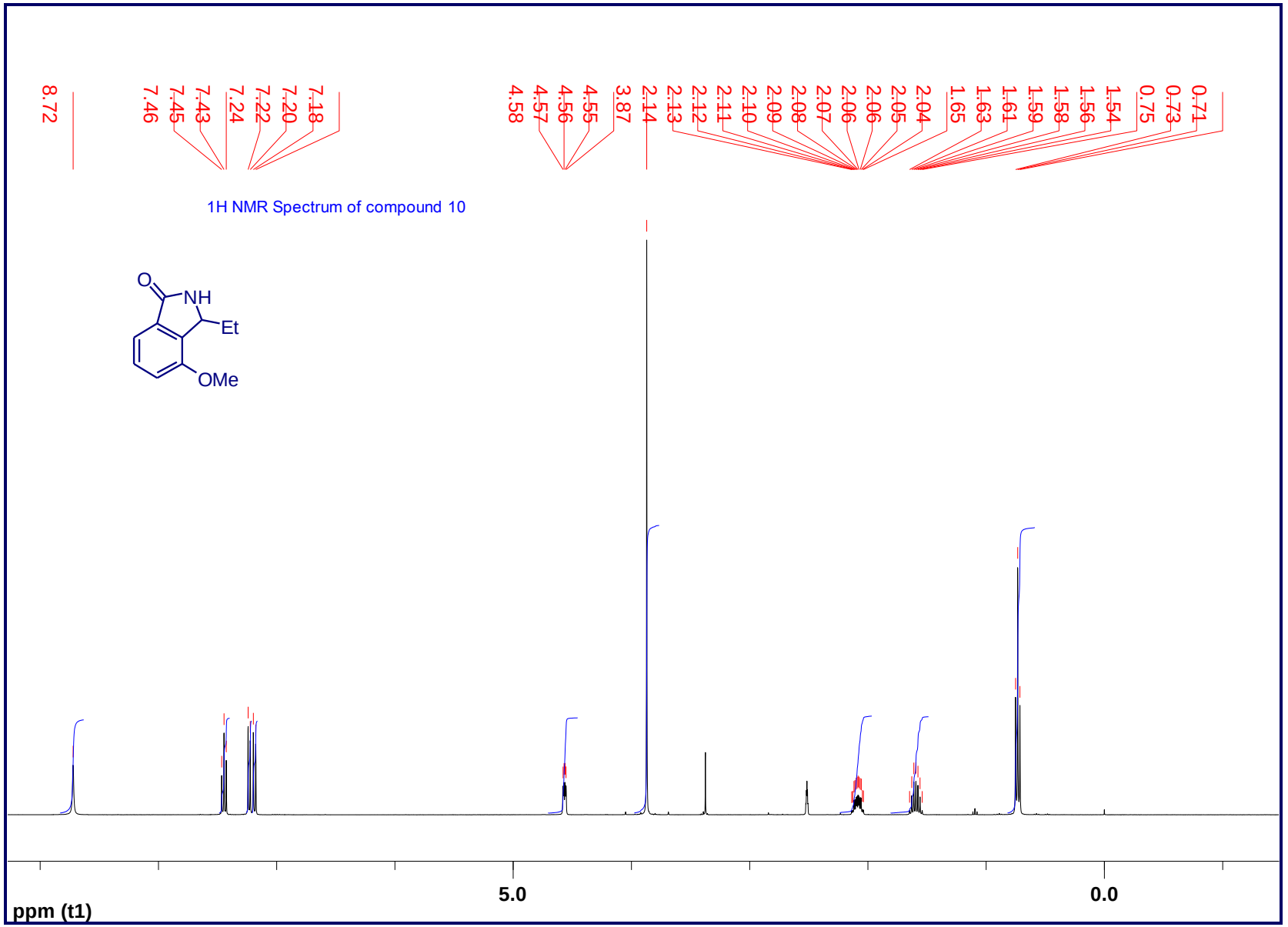

Expansion - 1H NMR Spectrum of compound 9
O
NH
Me
OMe
1.600
1.550
1.500
ppm (t1)


171.24
155.33
136.77
133.91
130.15
116.03
113.61
55.81
52.01
25.31
19.29
13C NMR Spectrum of compound 9
O
NH
Me
OMe
150
100
50
0
ppm (t1)

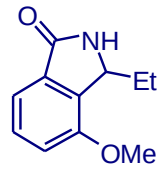

171.24
155.33
136.77
133.91
130.15
116.03
113.61
Expansion - 13C NMR Spectrum of compound 9
O
NH
Me
OMe
170
160
150
140
130
120
110
ppm (t1)

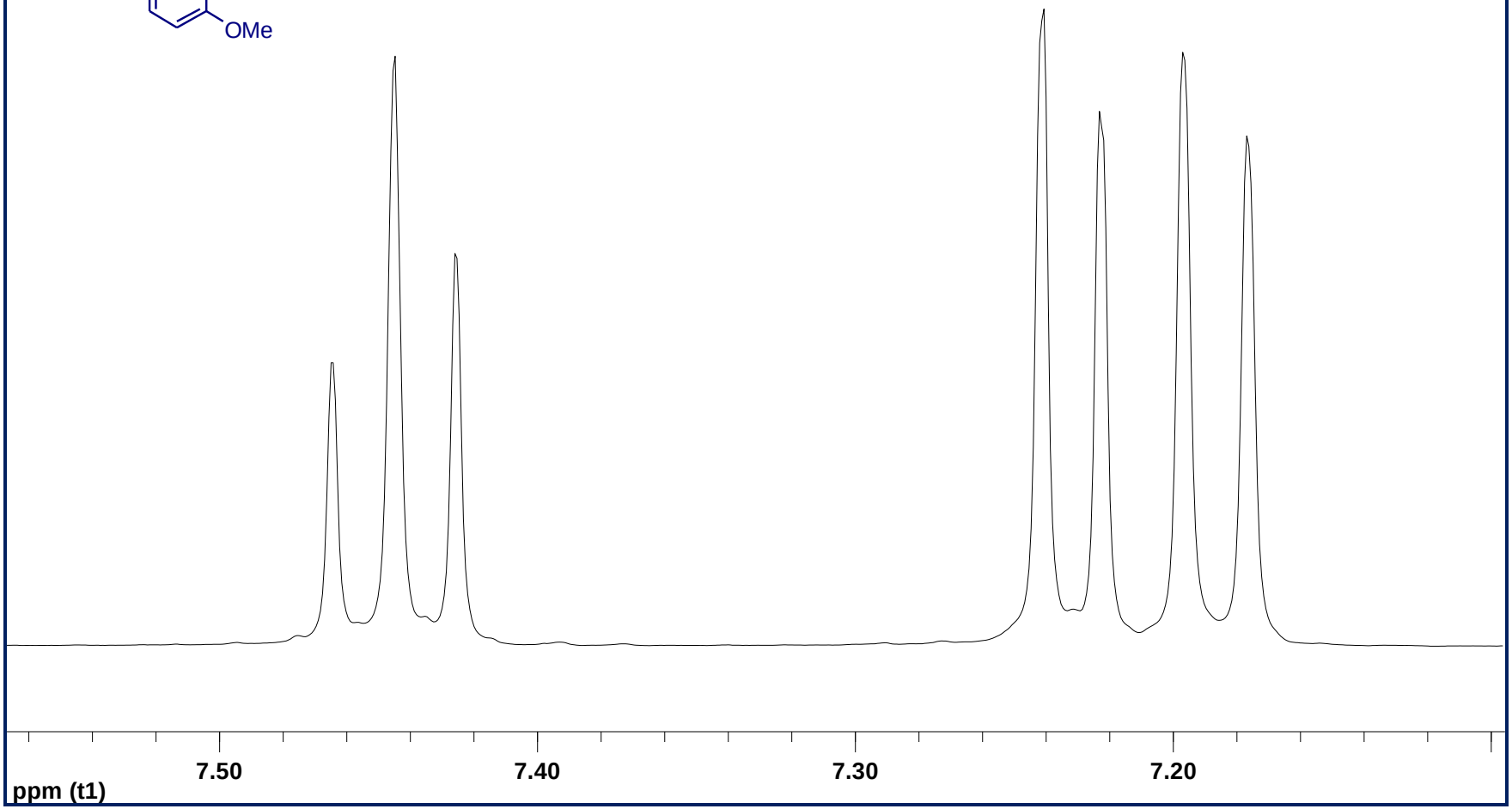

55.81
52.01
25.31
19.29
Expansion - 13C NMR Spectrum of compound 9
O
NH
Me
OMe
60
50
40
30
20
ppm (t1)


130.15
116.03
113.61
55.81
52.01
19.28
13C NMR DEPT-135 Spectrum of compound 9
DEPT-135 Spetrum shows CH and CH3 positive and CH2 negative
O
NH
Me
OMe
150
100
50
0
ppm (t1)

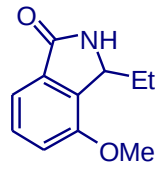

8.72
7.46
7.45
7.43
7.24
7.22
7.20
7.18
4.58
4.57
4.56
4.55
3.87
2.14
2.13
2.12
2.11
2.10
2.09
2.08
2.07
2.06
2.06
2.05
2.04
1.65
1.63
1.61
1.59
1.58
1.56
1.54
0.75
0.73
0.71
1H NMR Spectrum of compound 10
O
NH
Et
OMe
5.0
0.0
ppm (t1)

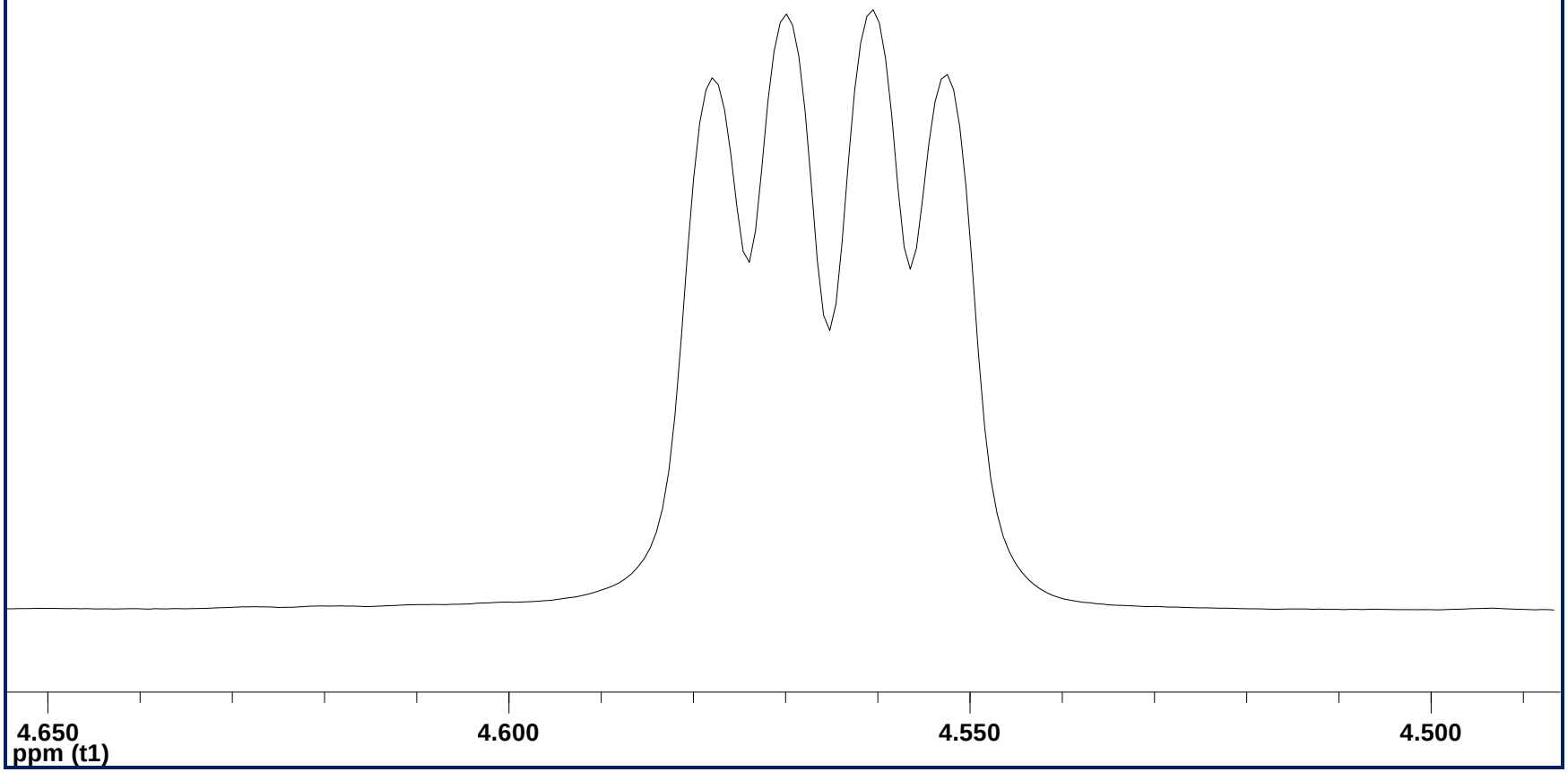
Expansion - 1H NMR Spectrum of compound 10



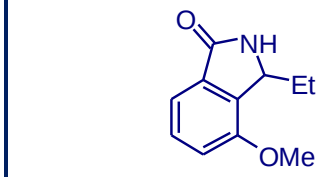


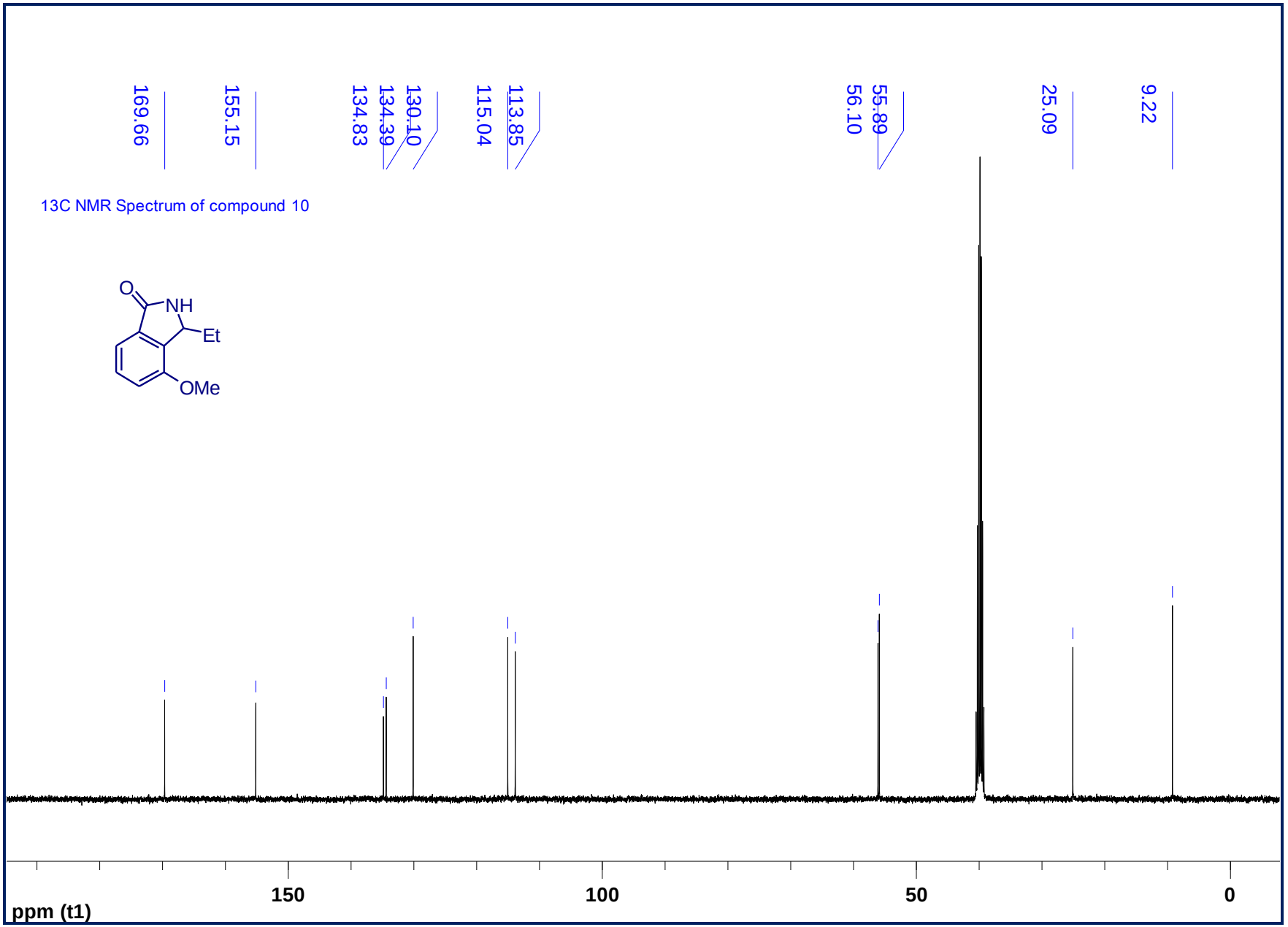
Expansion - 1H NMR Spectrum of compound 10

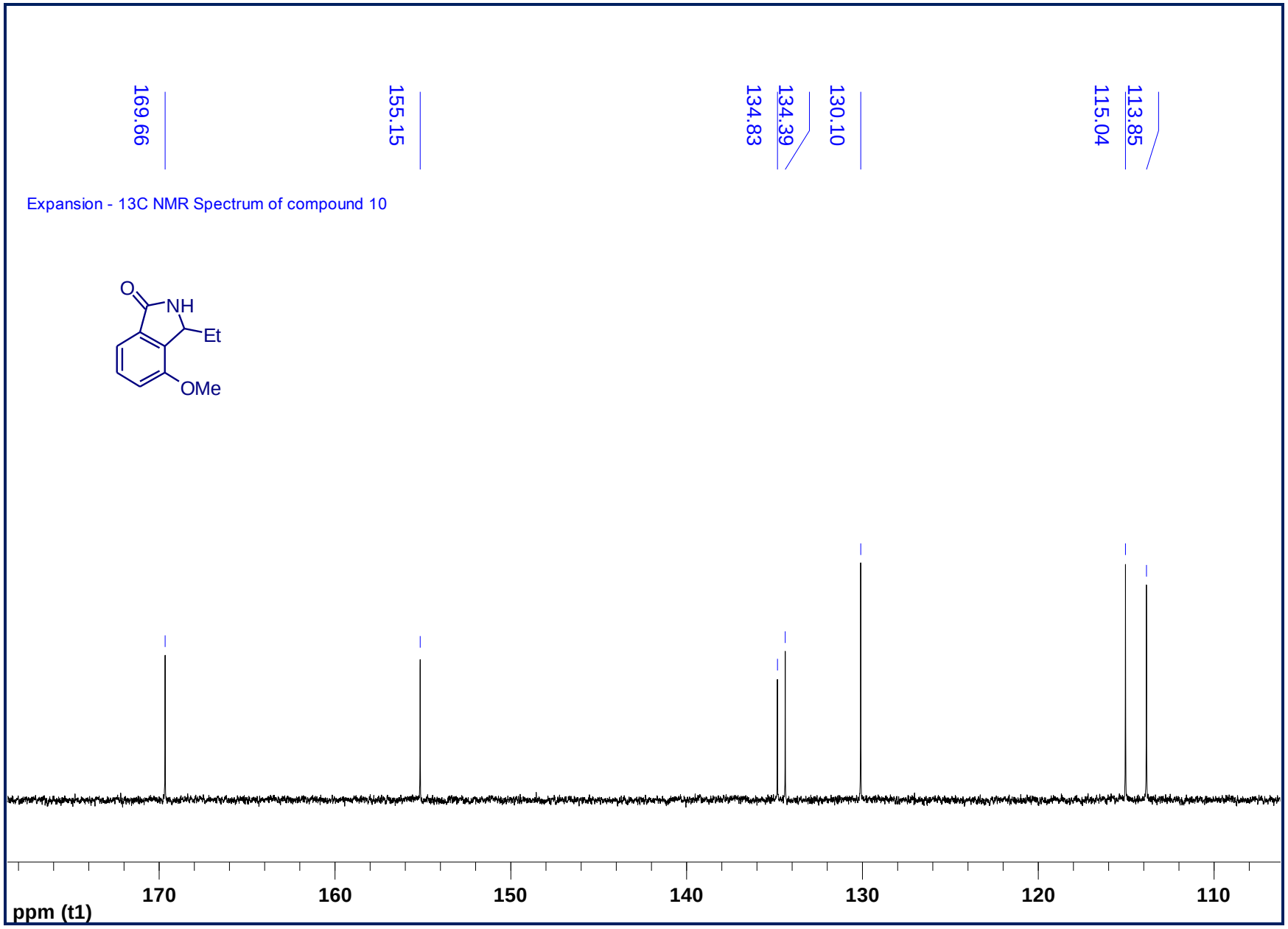


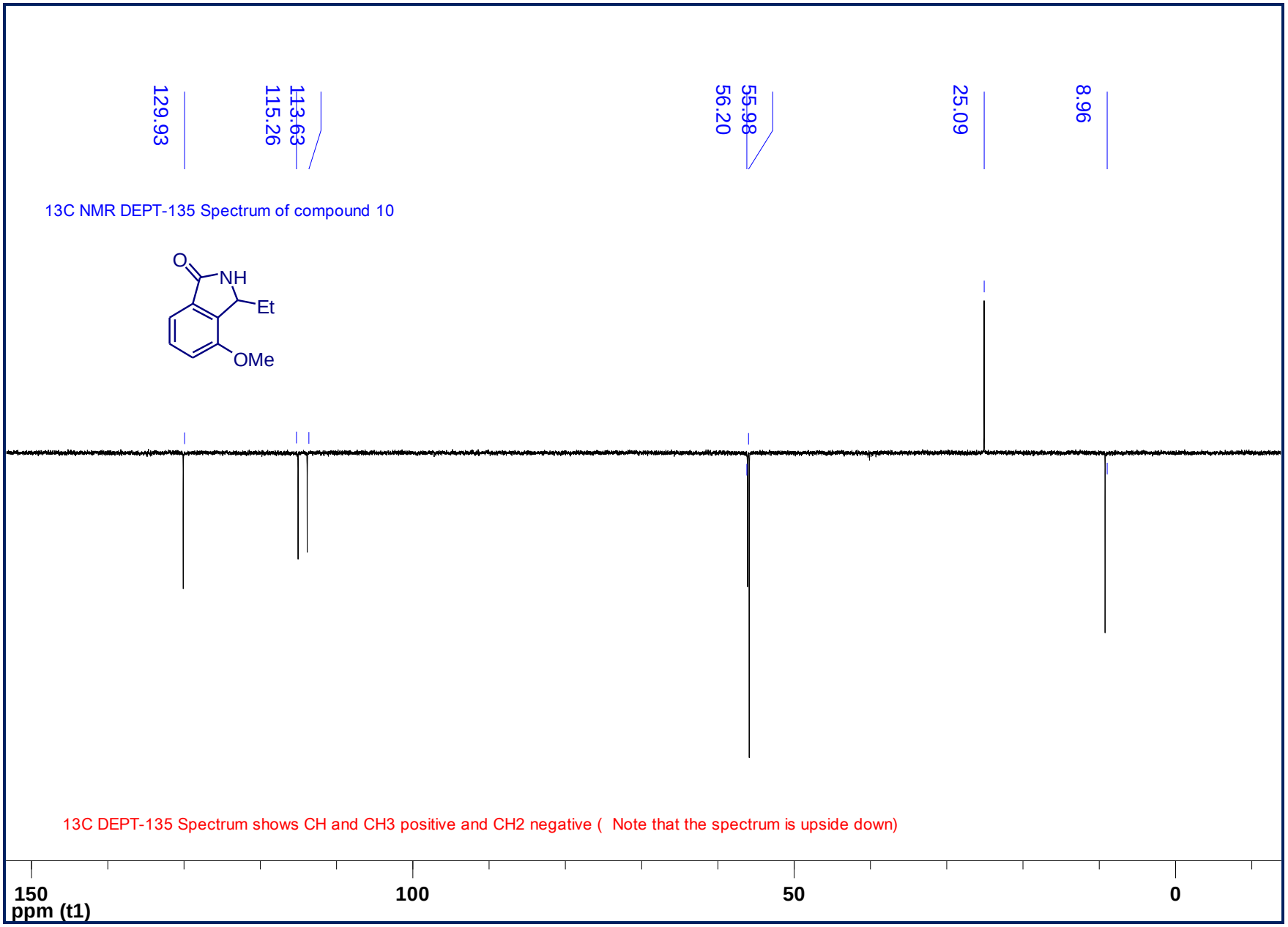


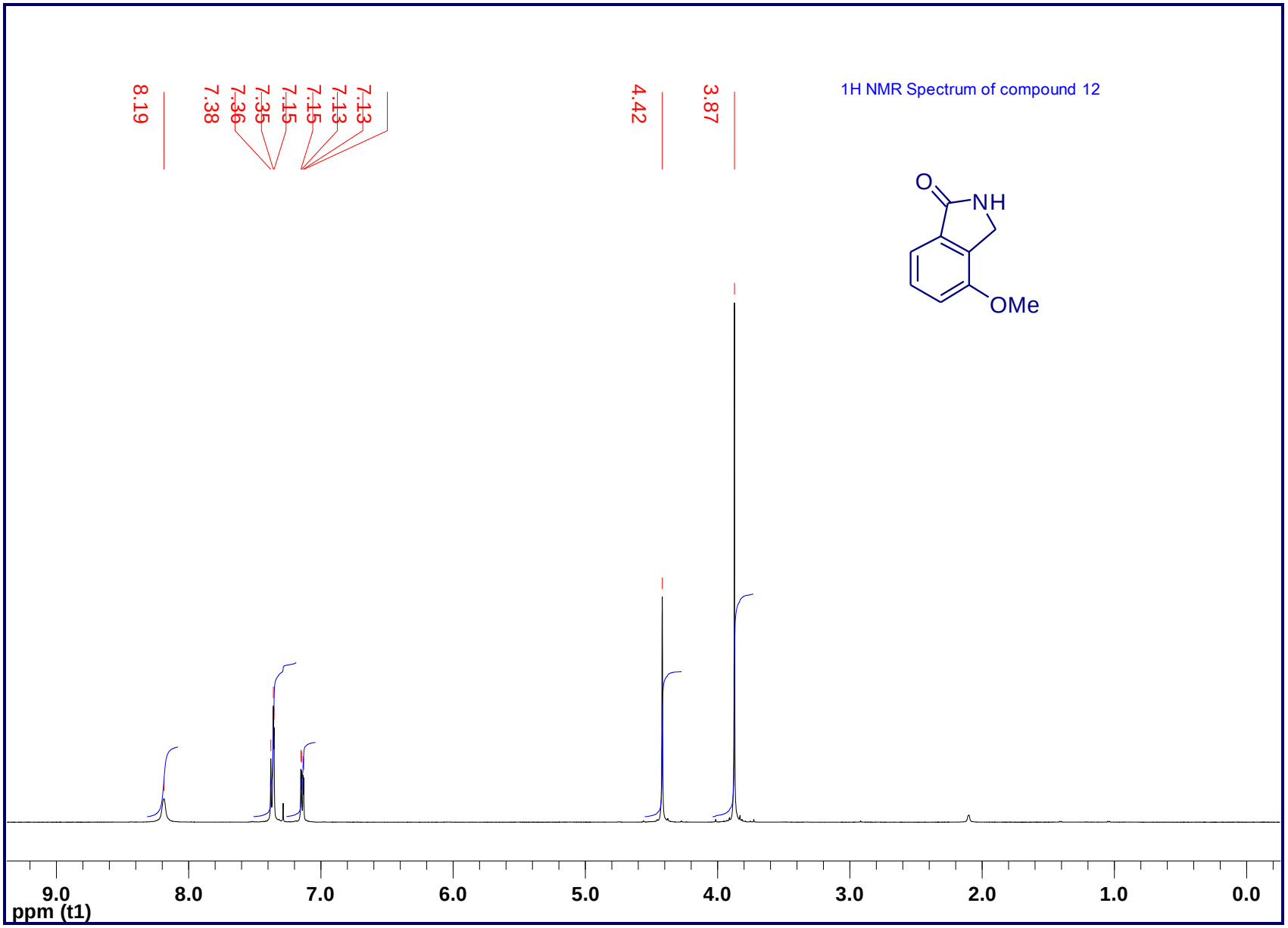
Expansion - 1H NMR Spectrum of compound 10



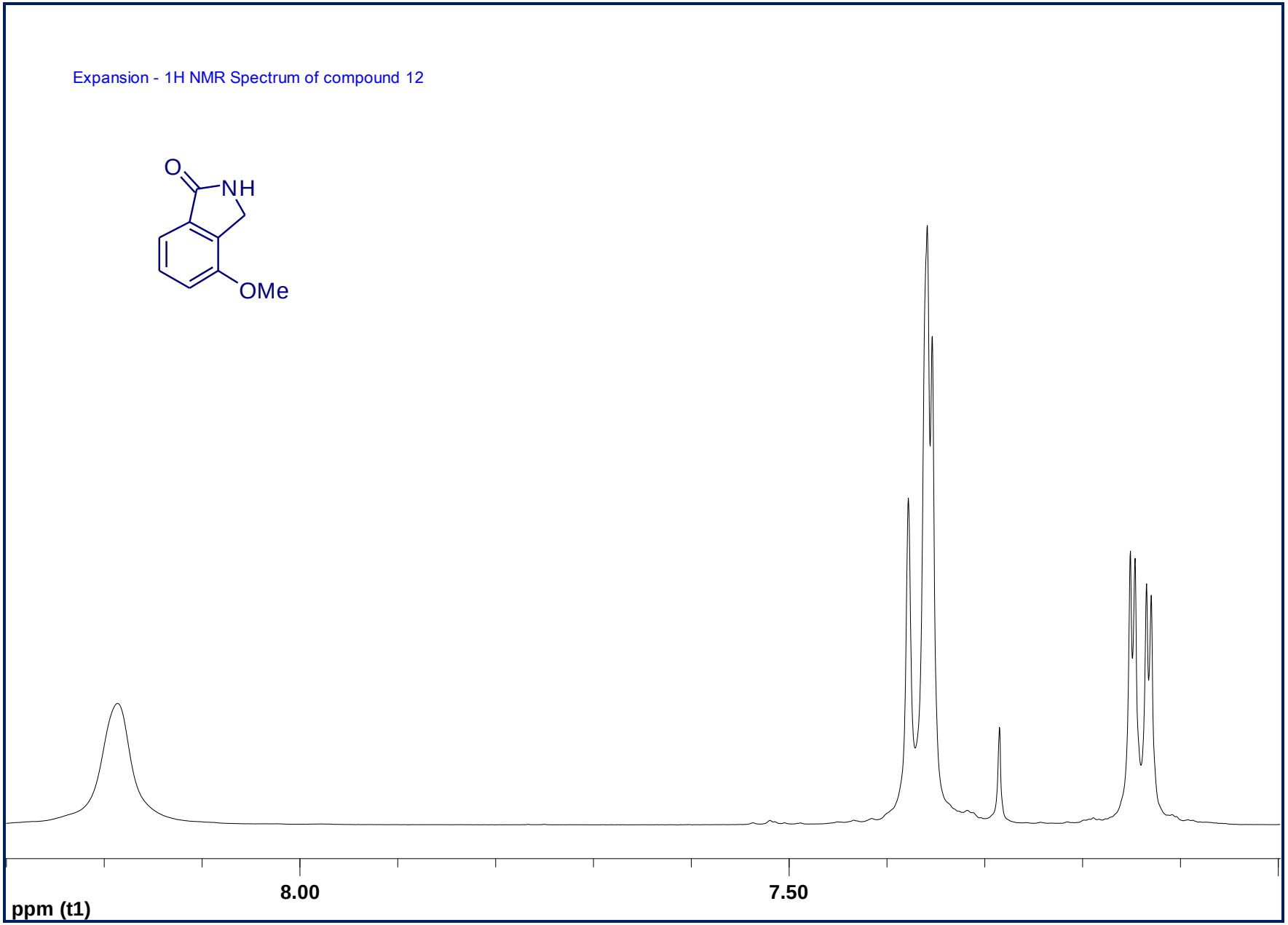

169.66
155.15
134.83
134.39
130.10
115.04
113.85
56.10
55.89
25.09
9.22
13C NMR Spectrum of compound 10
O
NH
Et
OMe
150
100
50
0
ppm (t1)

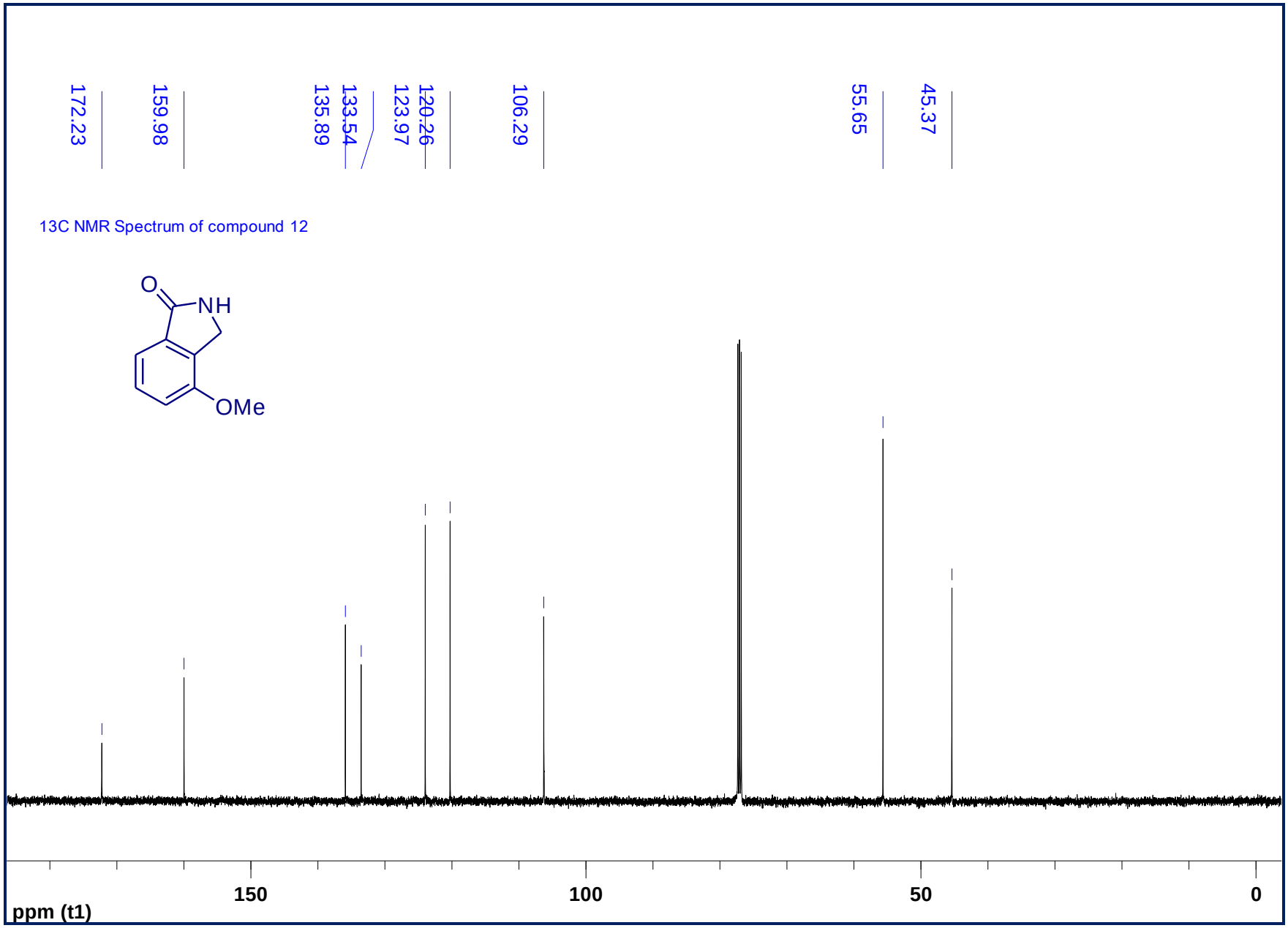

169.66
155.15
134.83
134.39
130.10
115.04
113.85
Expansion - 13C NMR Spectrum of compound 10
O
NH
Et
OMe
170
160
150
140
130
120
110
ppm (t1)

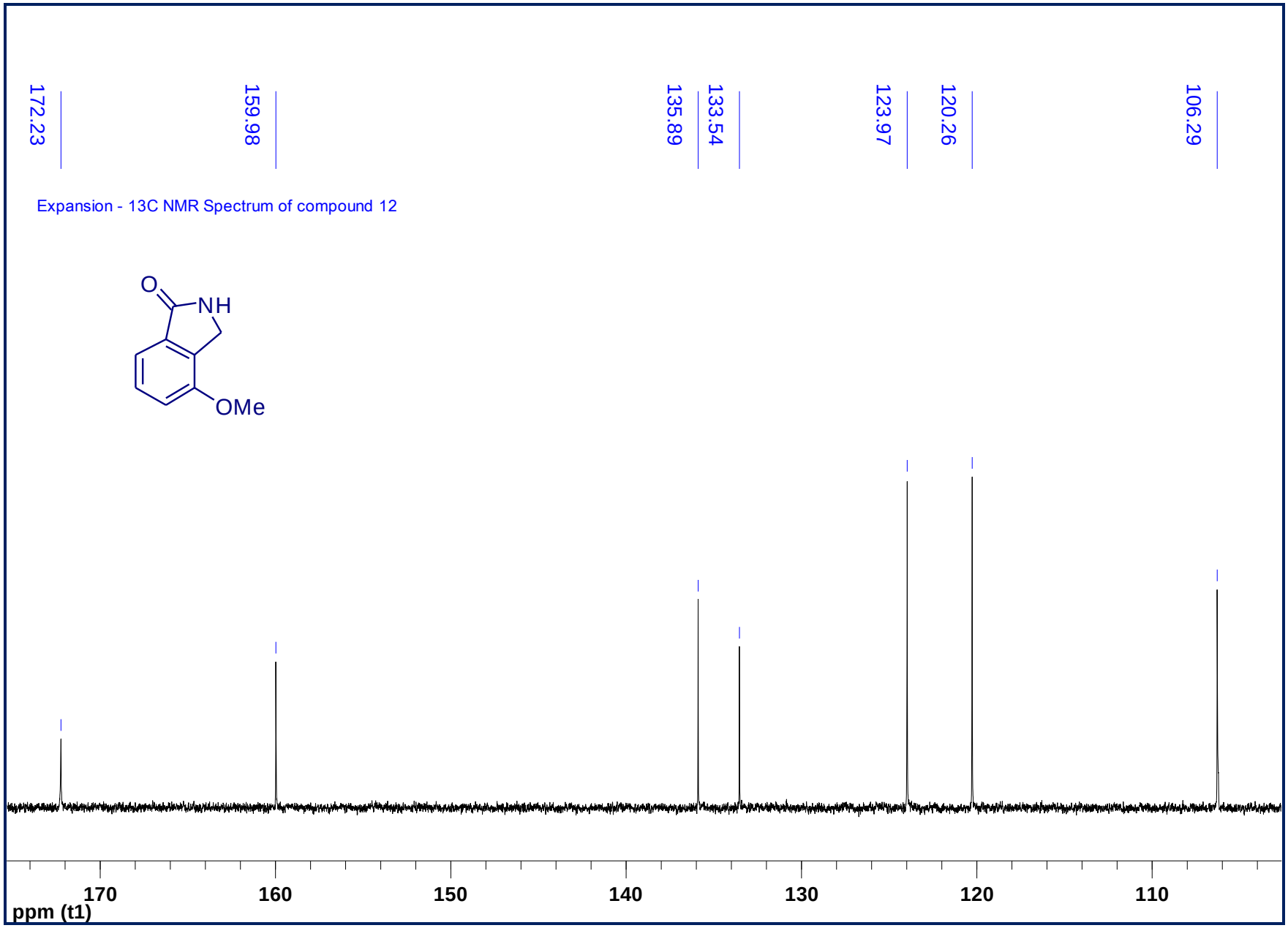

129.93
115.26
113.63
56.20
55.98
25.09
8.96
13C NMR DEPT-135 Spectrum of compound 10
O
NH
Et
OMe
13C DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative (Note that the spectrum is upside down)
150
100
50
0
ppm (t1)

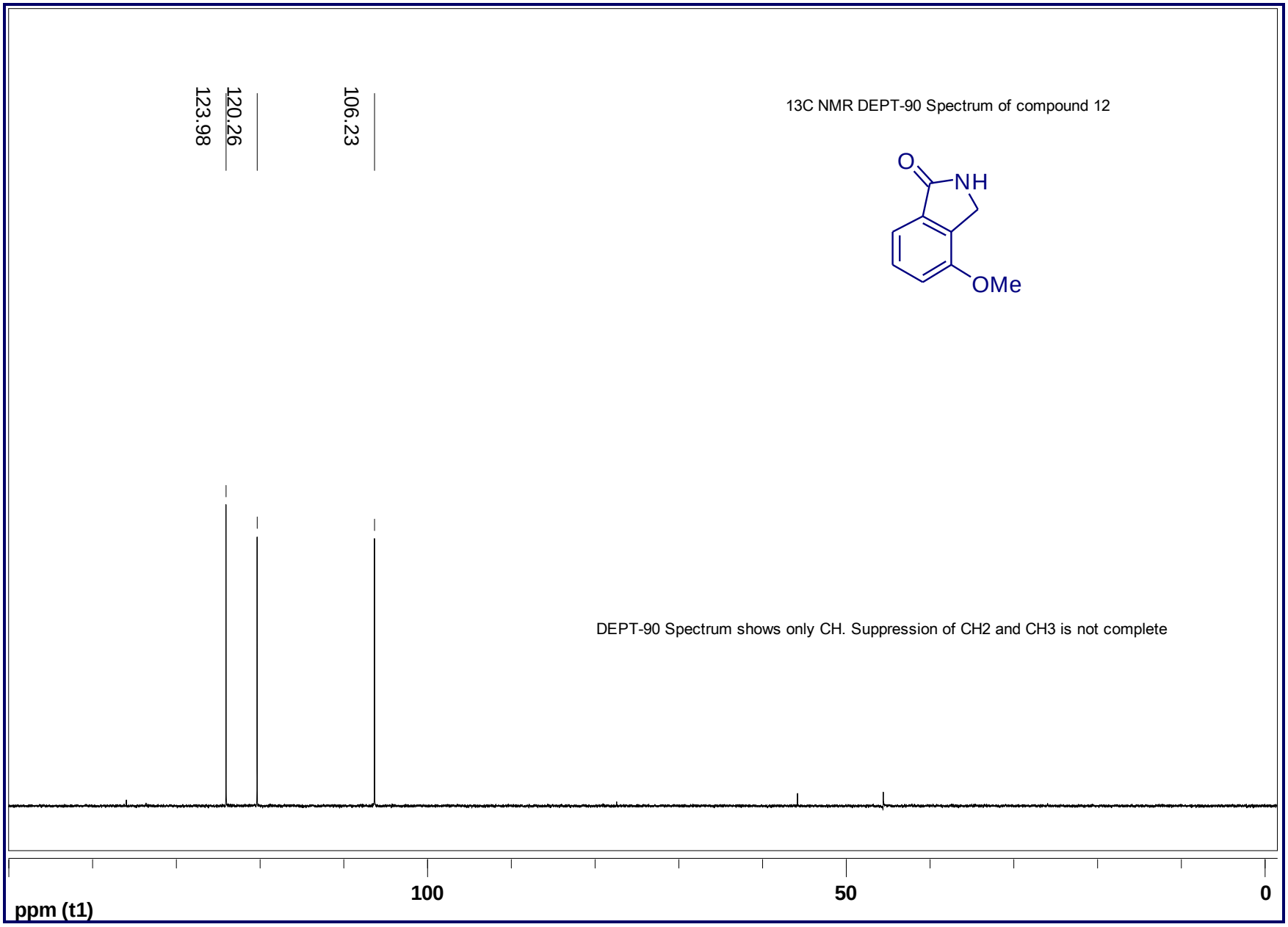

1H NMR Spectrum of compound 12
8.19
7.38
7.36
7.35
7.15
7.15
7.13
7.13
7.13
4.42
3.87
O
NH
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

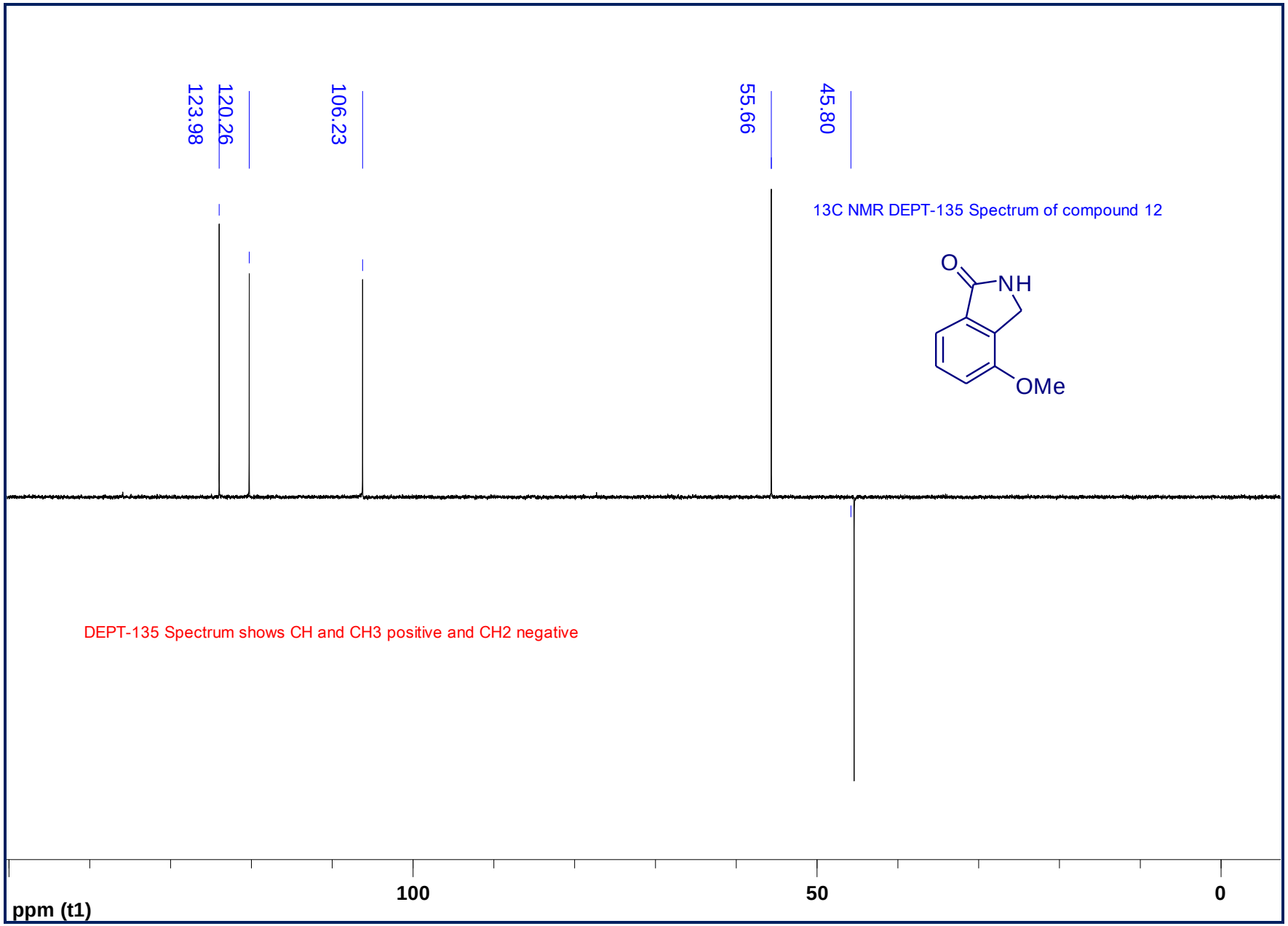
Expansion - 1H NMR Spectrum of compound 12

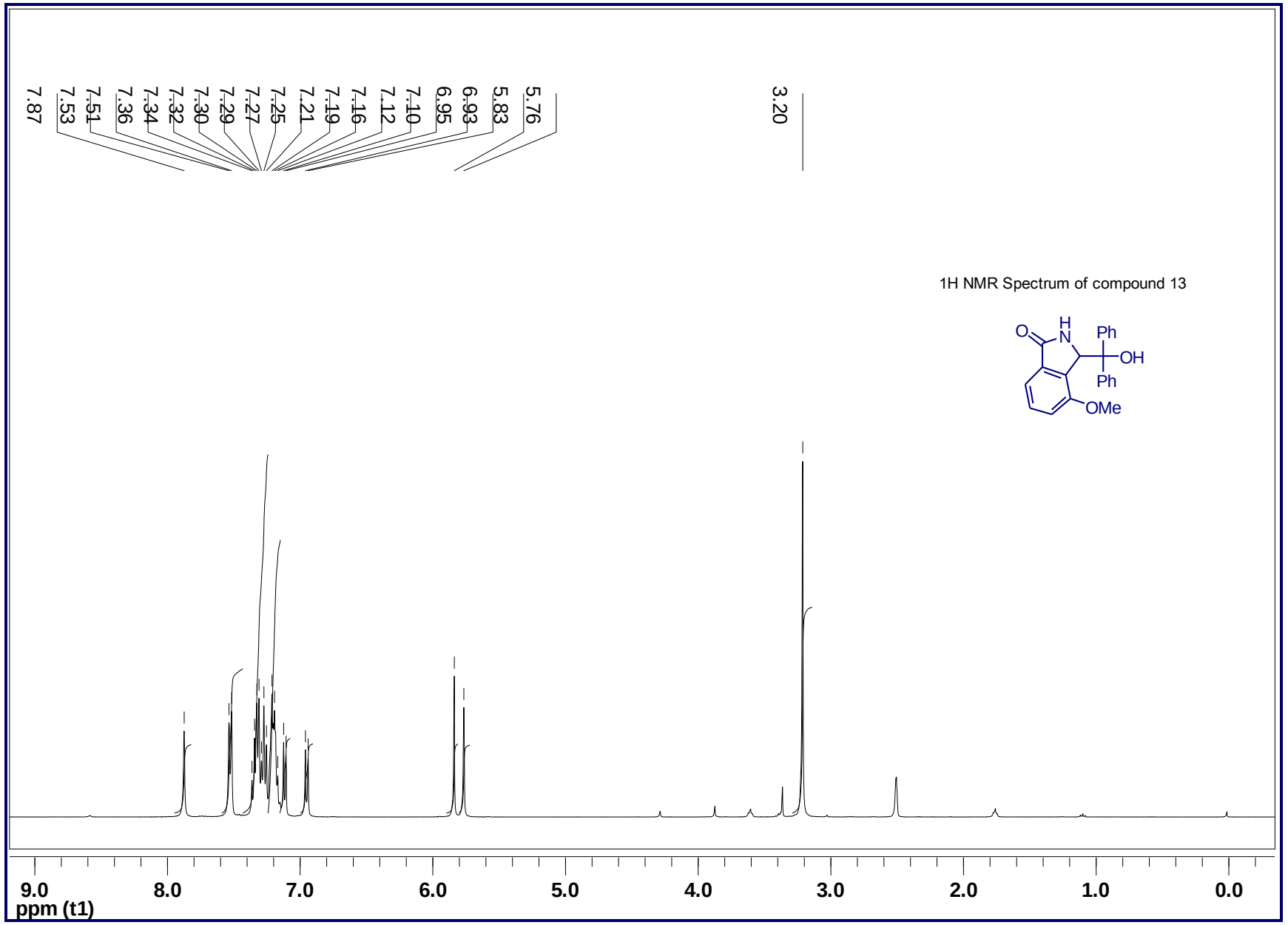



172.23
159.98
135.89
133.54
123.97
120.26
106.29
55.65
45.37
13C NMR Spectrum of compound 12
O
NH
OMe
150
100
50
0
ppm (t1)

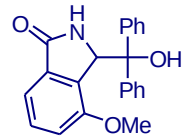

172.23
159.98
135.89
133.54
123.97
120.26
106.29
Expansion - 13C NMR Spectrum of compound 12
O
NH
OMe
170
160
150
140
130
120
110
ppm (t1)

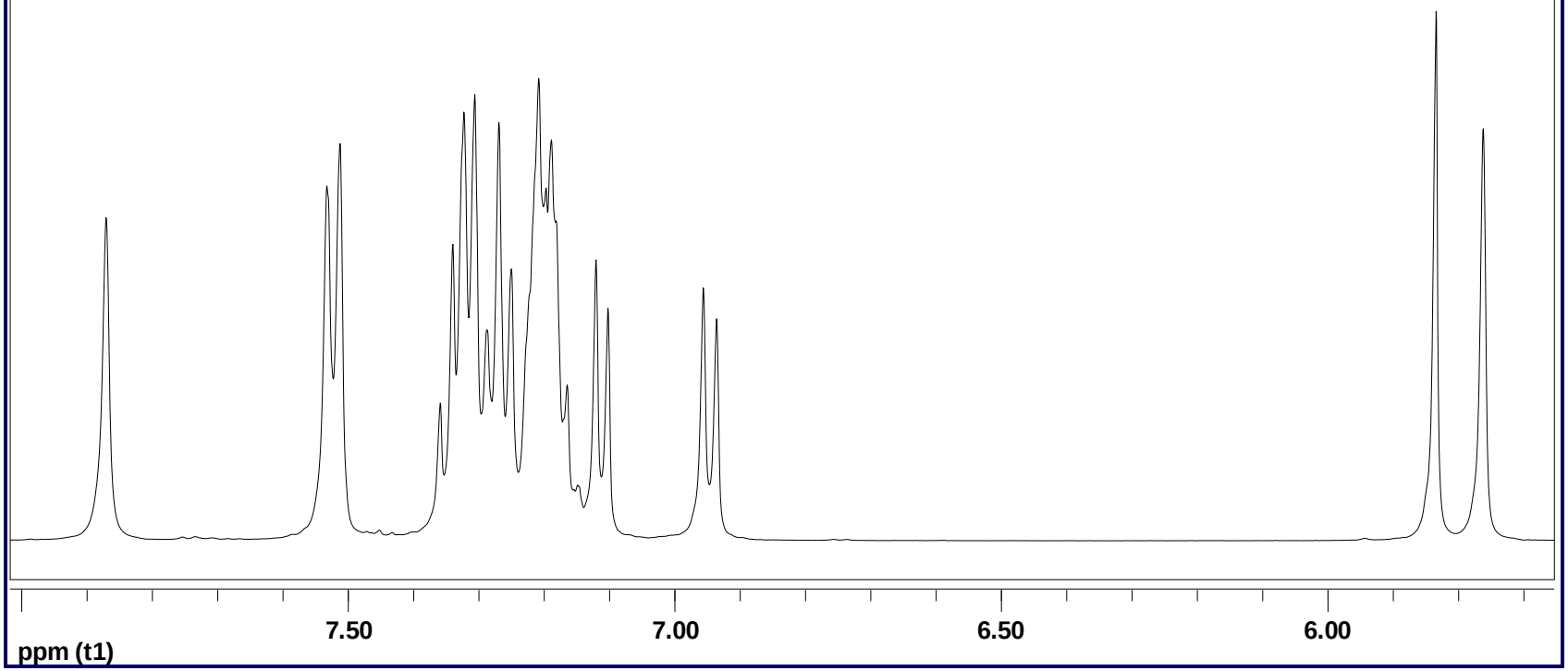

123.98
120.26
106.23
13C NMR DEPT-90 Spectrum of compound 12
O
NH
OMe
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete
100
50
0
ppm (t1)

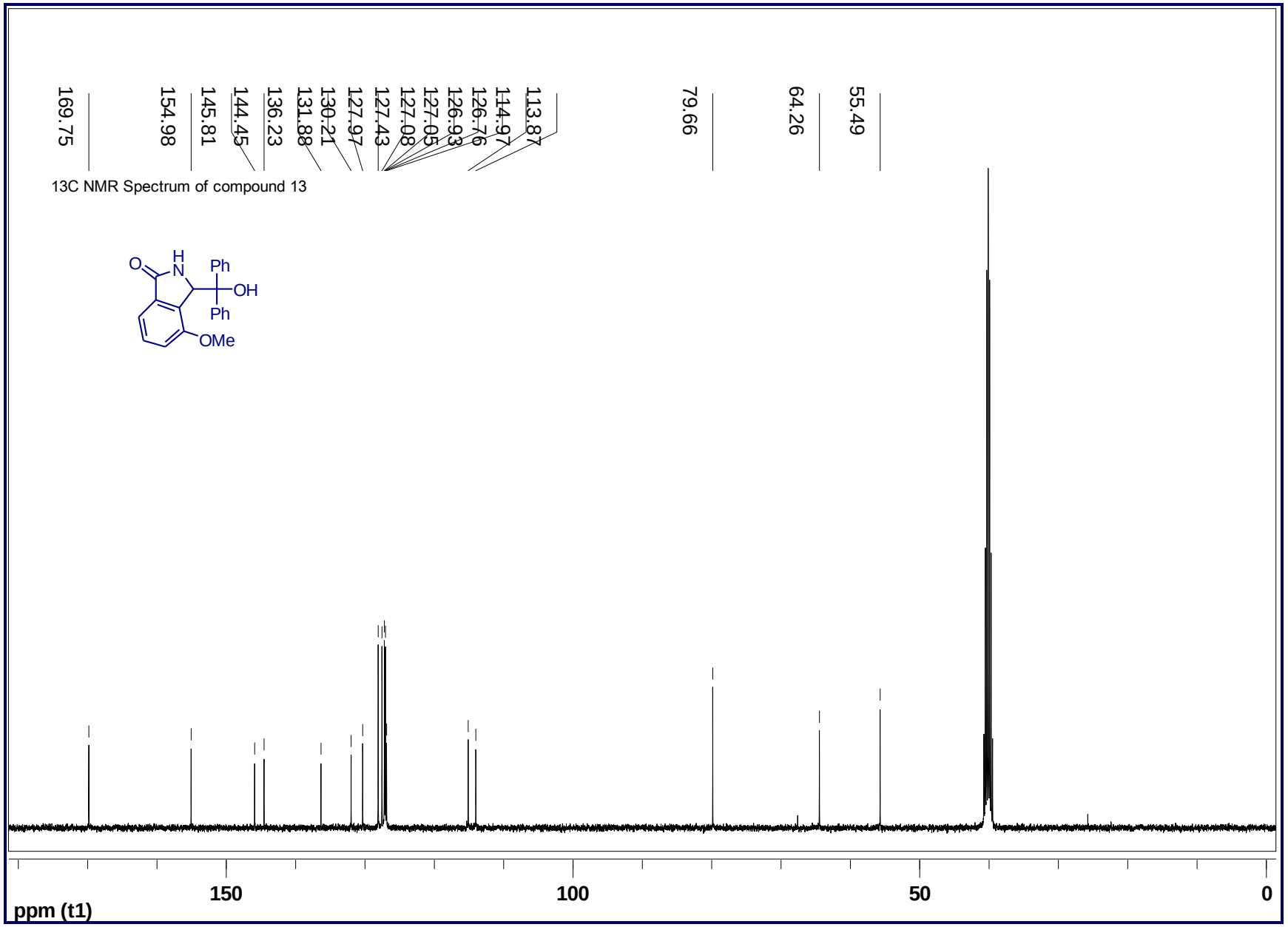

123.98
120.26
106.23
55.66
45.80
13C NMR DEPT-135 Spectrum of compound 12
O
NH
OMe
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative
100
50
0
ppm (t1)

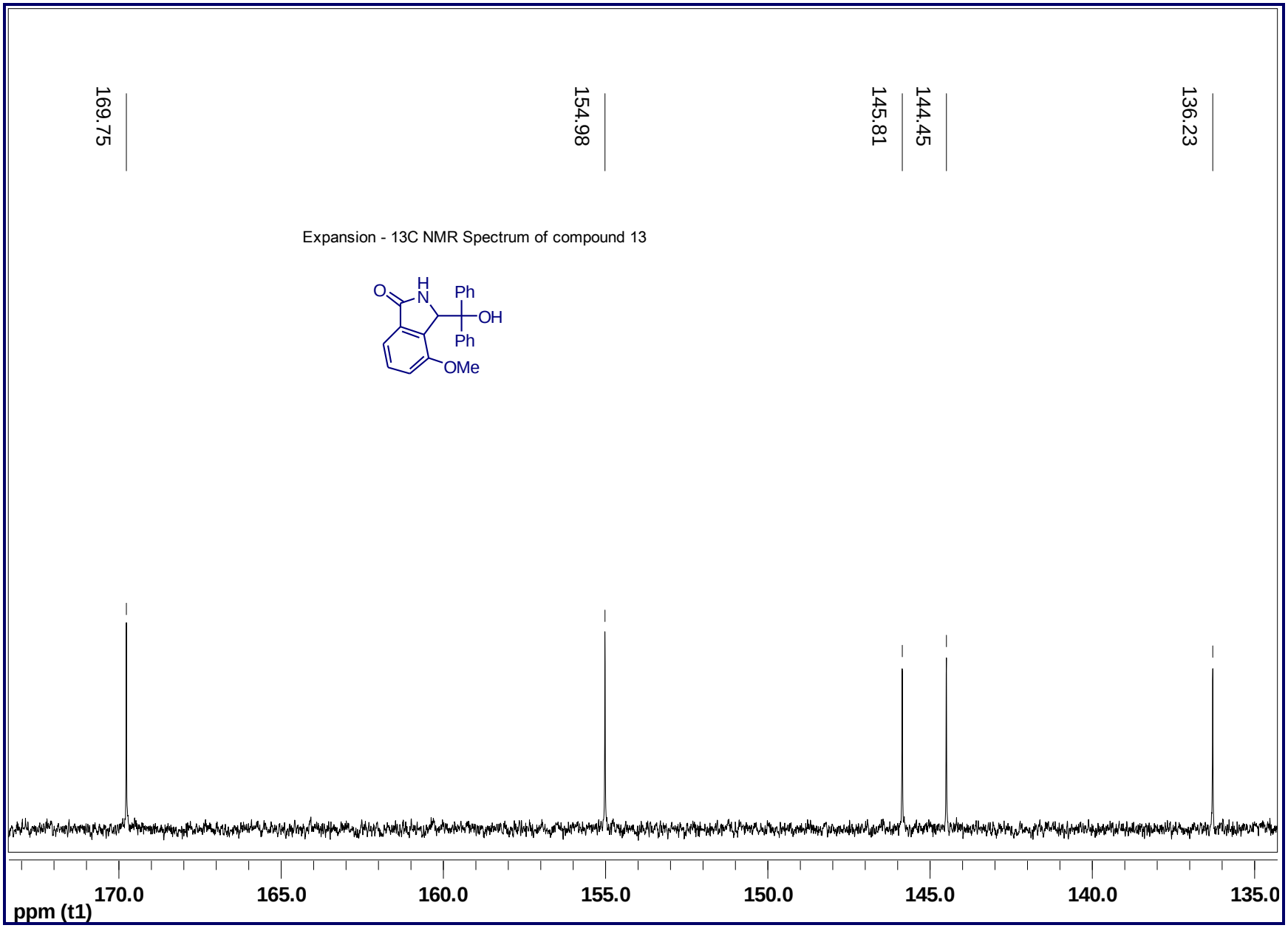

7.87
7.53
7.51
7.36
7.34
7.32
7.30
7.29
7.27
7.25
7.21
7.19
7.16
7.12
7.10
6.95
6.93
5.83
5.76
3.20
1H NMR Spectrum of compound 13
O
H
N
Ph
OH
Ph
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

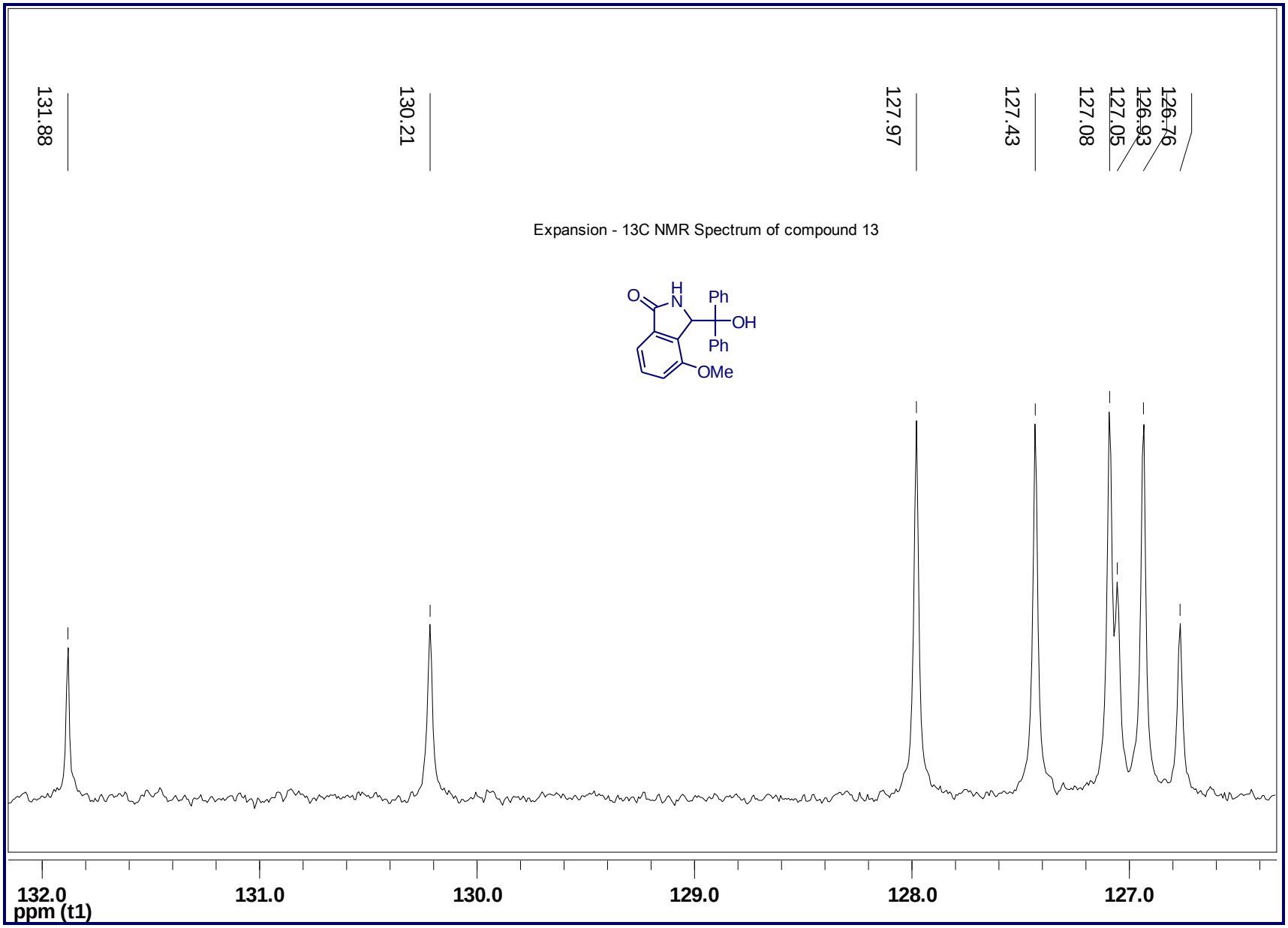
Expansion - 1H NMR Spectrum of compound 13

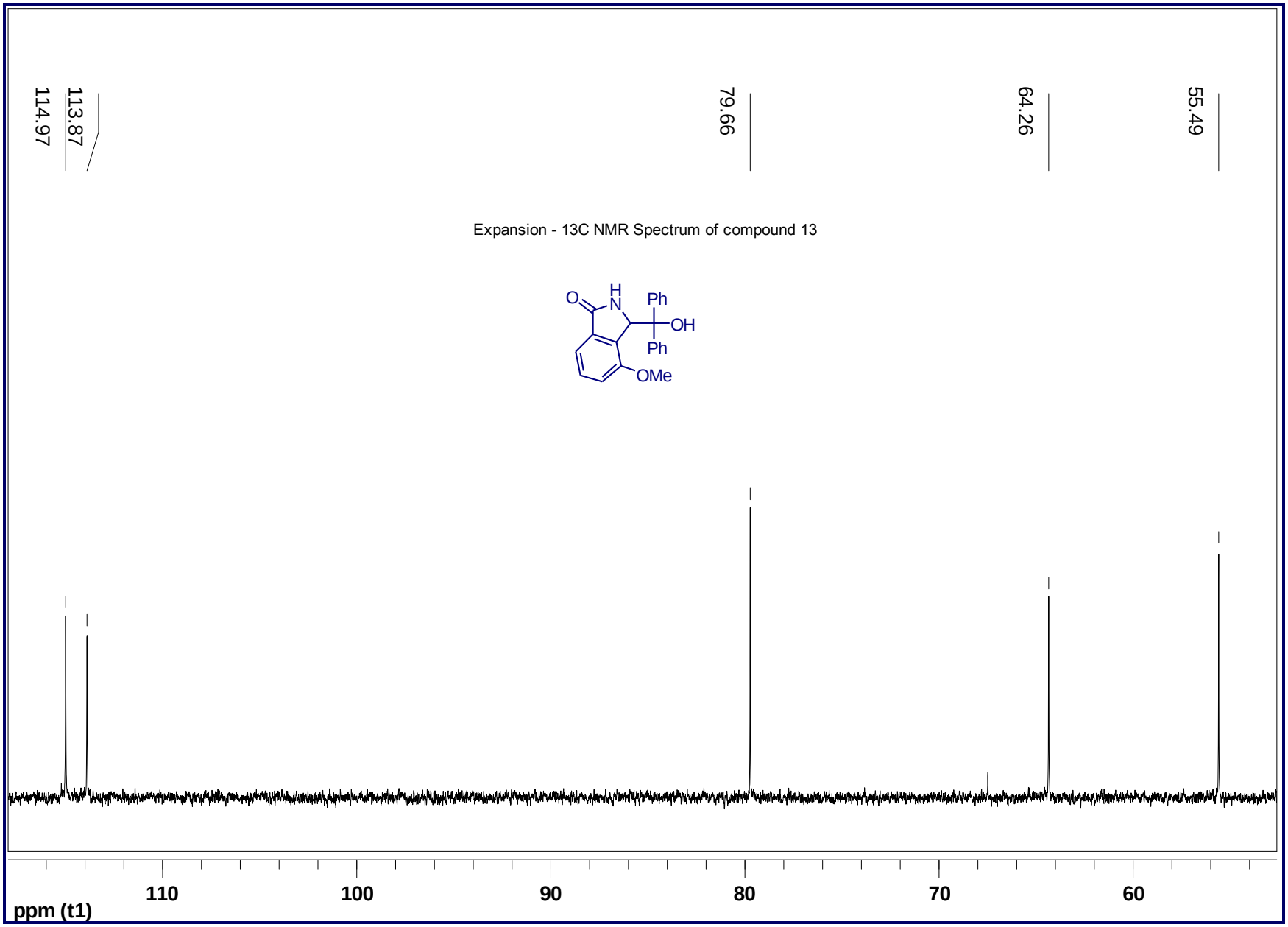


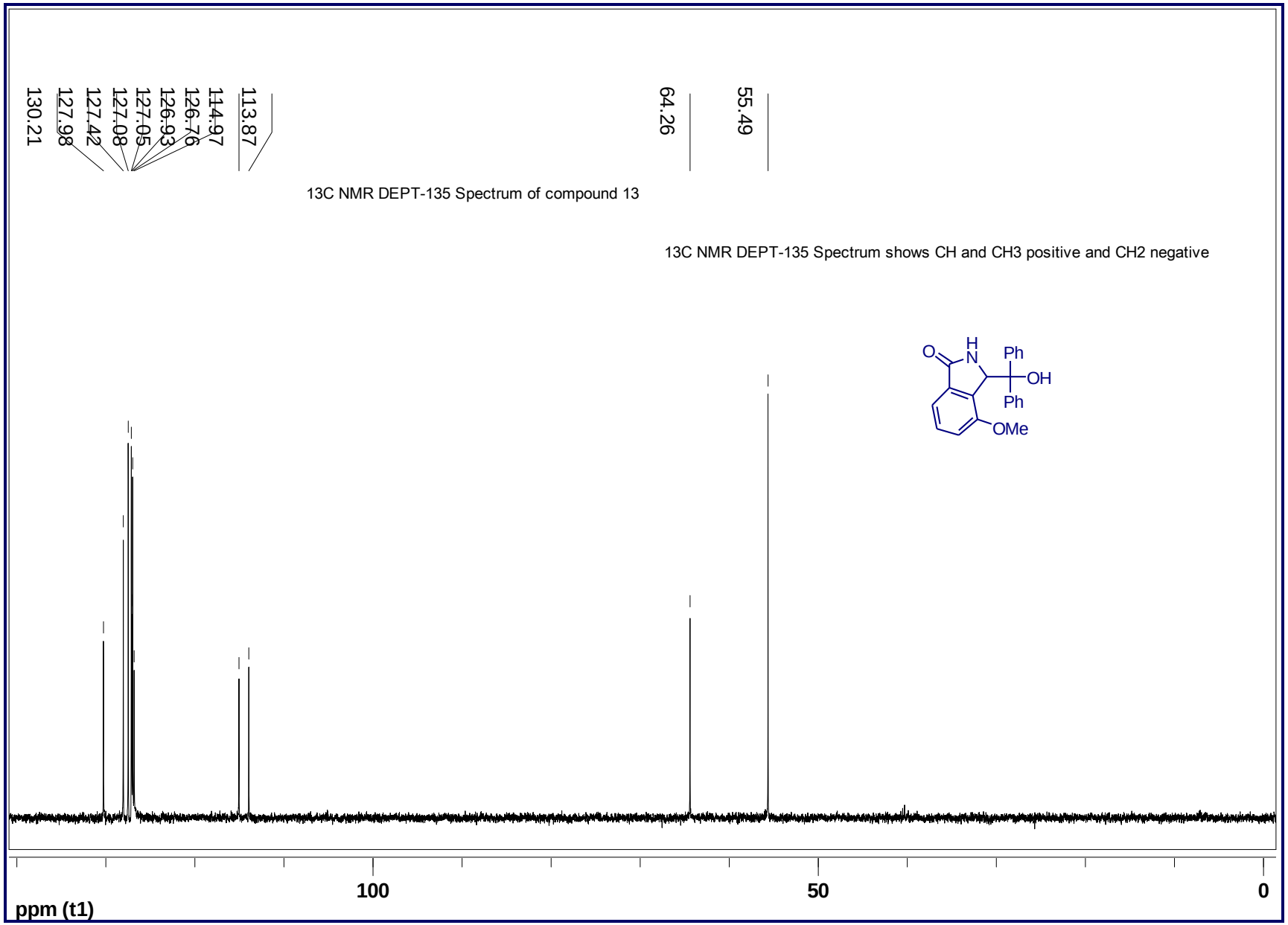


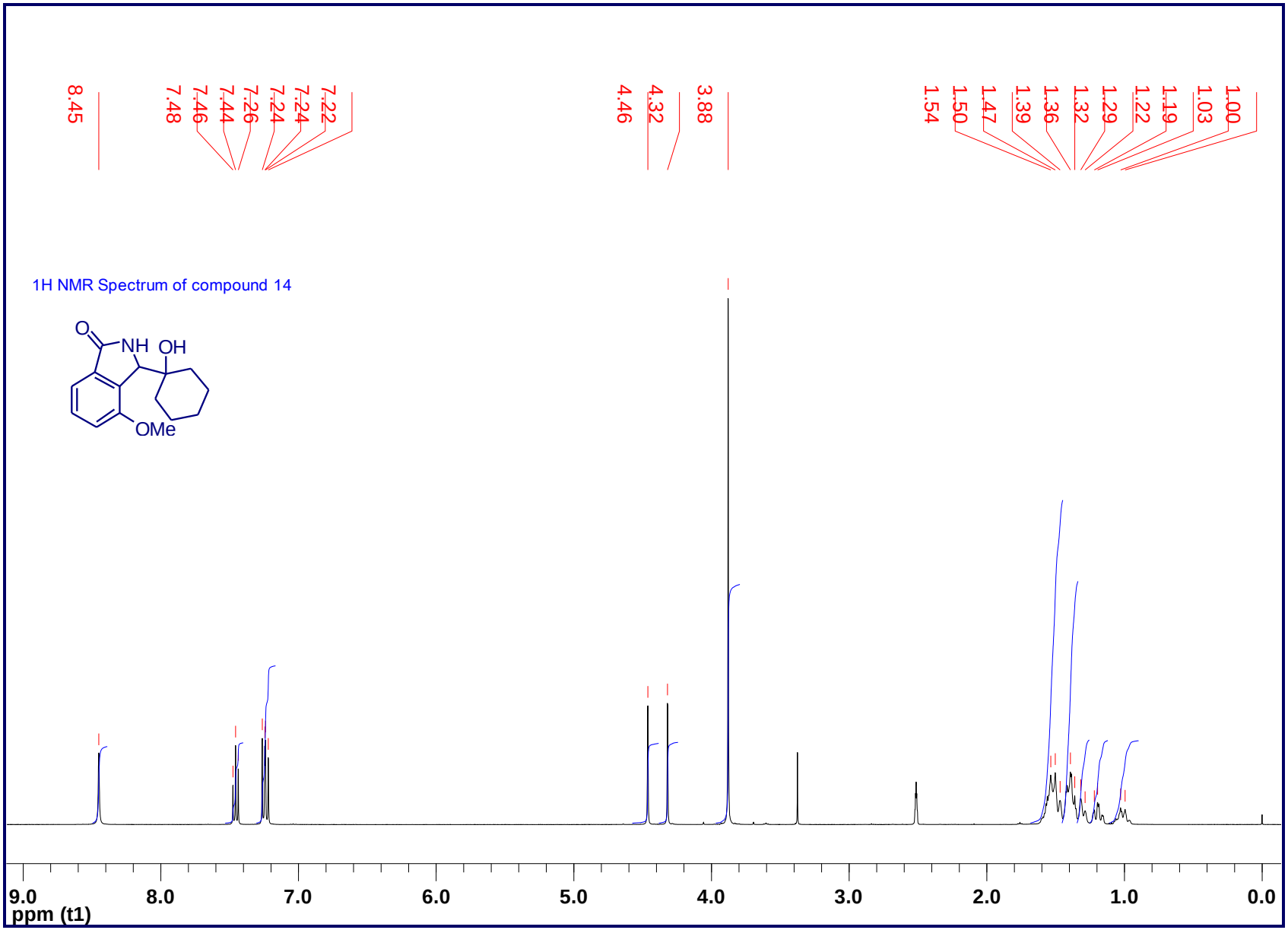

169.75
154.98
145.81
144.45
136.23
131.88
130.21
127.97
127.43
127.08
127.05
126.93
126.76
114.97
113.87
79.66
64.26
55.49
13C NMR Spectrum of compound 13
O
H
N
Ph
OH
Ph
OMe
150
100
50
0
ppm (t1)


169.75
154.98
145.81
144.45
136.23
Expansion - 13C NMR Spectrum of compound 13
O
H
N
Ph
OH
Ph
OMe
170.0
165.0
160.0
155.0
150.0
145.0
140.0
135.0
ppm (t1)

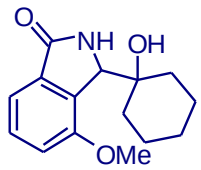

131.88
130.21
127.97
127.43
127.08
127.05
126.93
126.76
Expansion - 13C NMR Spectrum of compound 13
O
H
N
Ph
OH
Ph
OMe
132.0
131.0
130.0
129.0
128.0
127.0
ppm (t1)

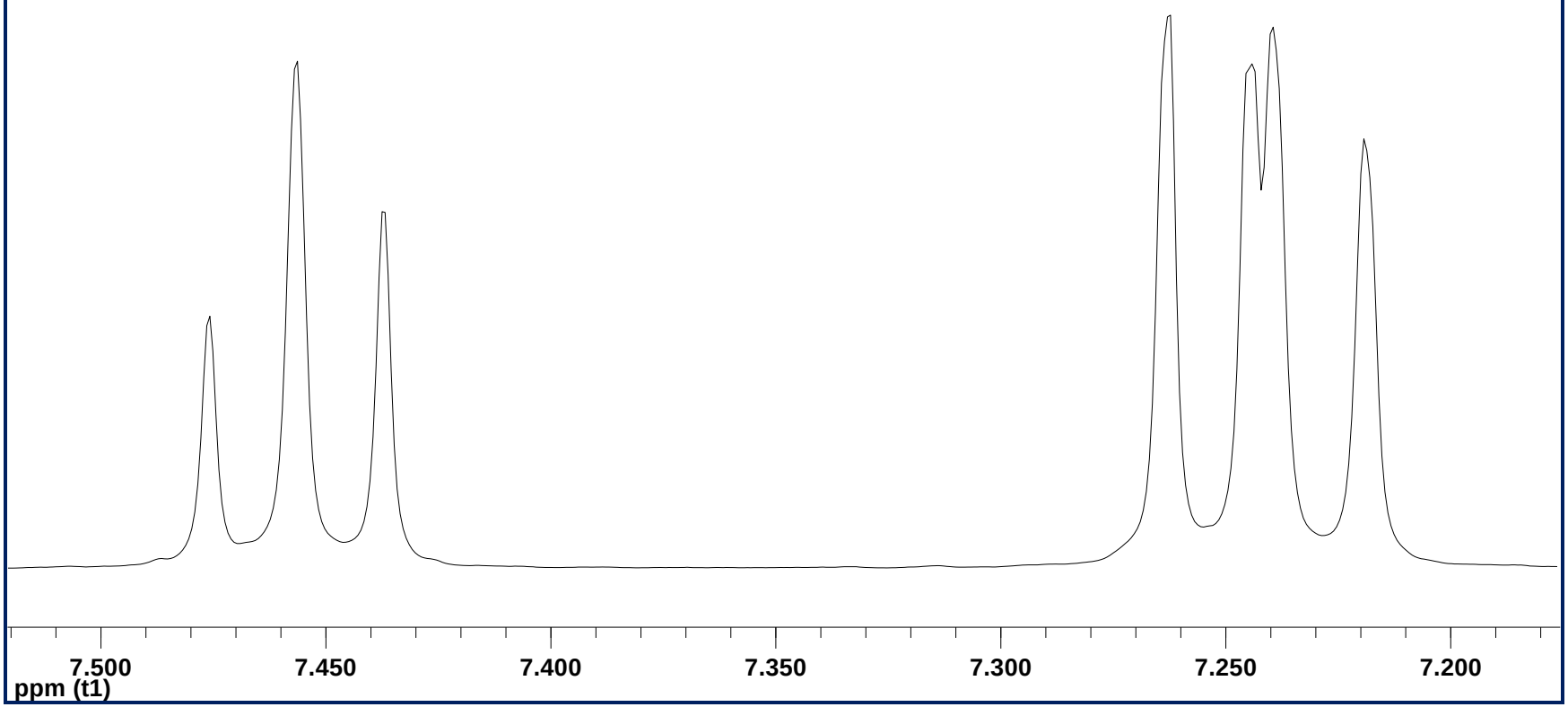

114.97
113.87
79.66
64.26
55.49
Expansion - 13C NMR Spectrum of compound 13
O
H
N
Ph
OH
Ph
OMe
110
100
90
80
70
60
ppm (t1)

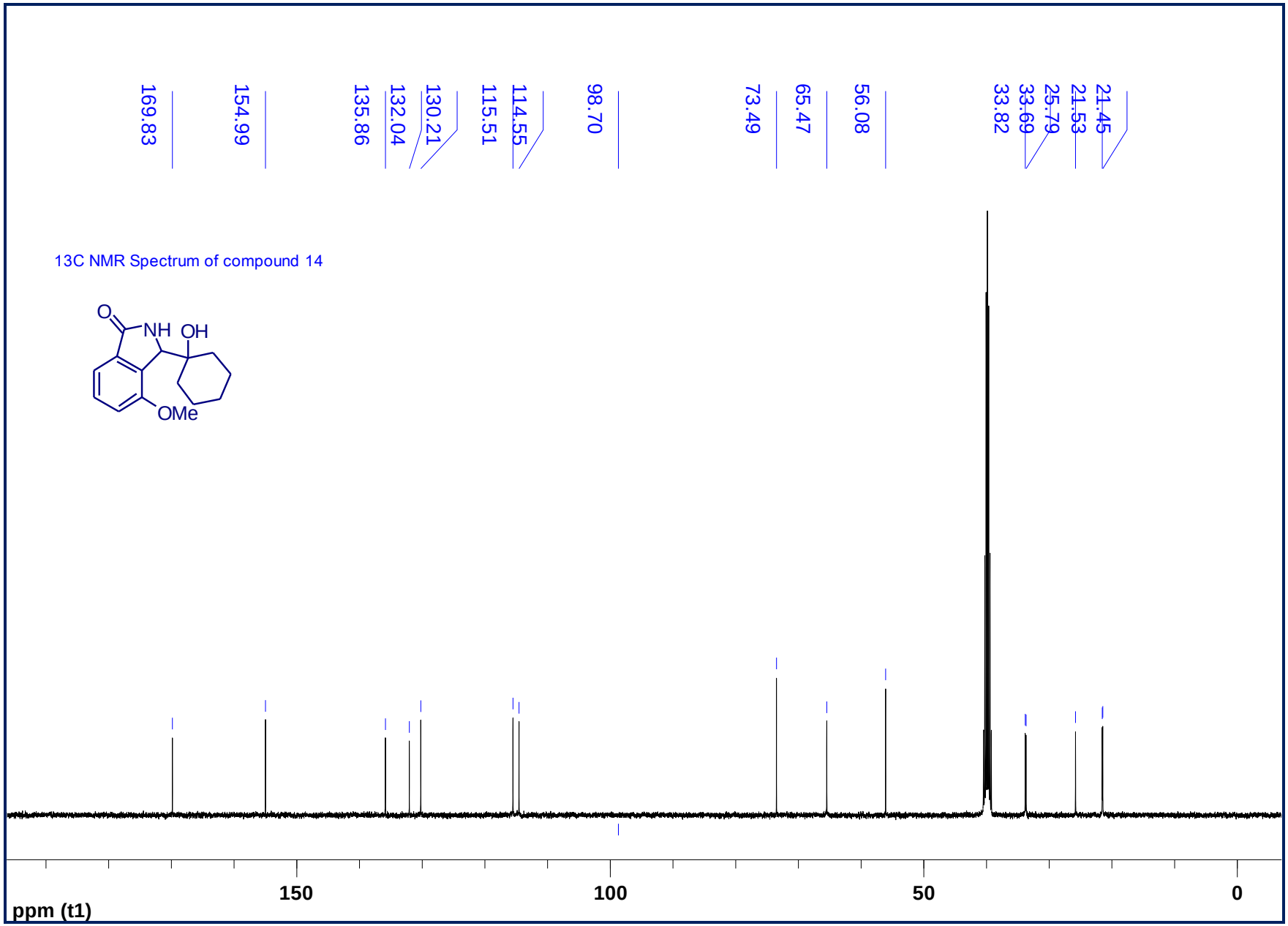

130.21
127.98
127.42
127.08
127.05
126.93
126.76
114.97
113.87
64.26
55.49
13C NMR DEPT-135 Spectrum of compound 13
13C NMR DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative
O
H
N
Ph
OH
Ph
OMe
100
50
0
ppm (t1)

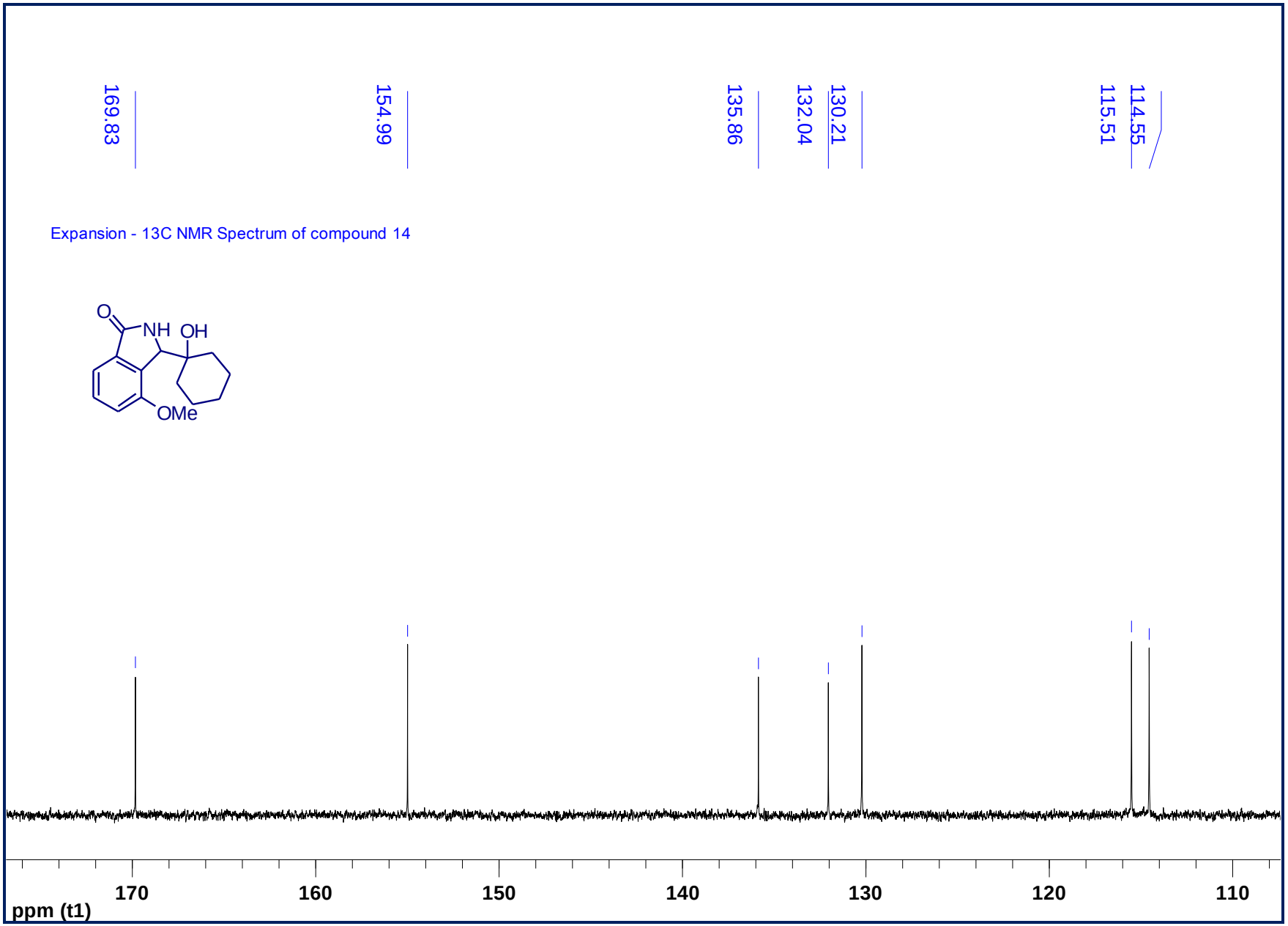

8.45
7.48
7.46
7.44
7.26
7.24
7.24
7.22
4.46
4.32
3.88
1.54
1.50
1.47
1.39
1.36
1.32
1.29
1.22
1.19
1.03
1.00
1H NMR Spectrum of compound 14
O
NH
OH
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

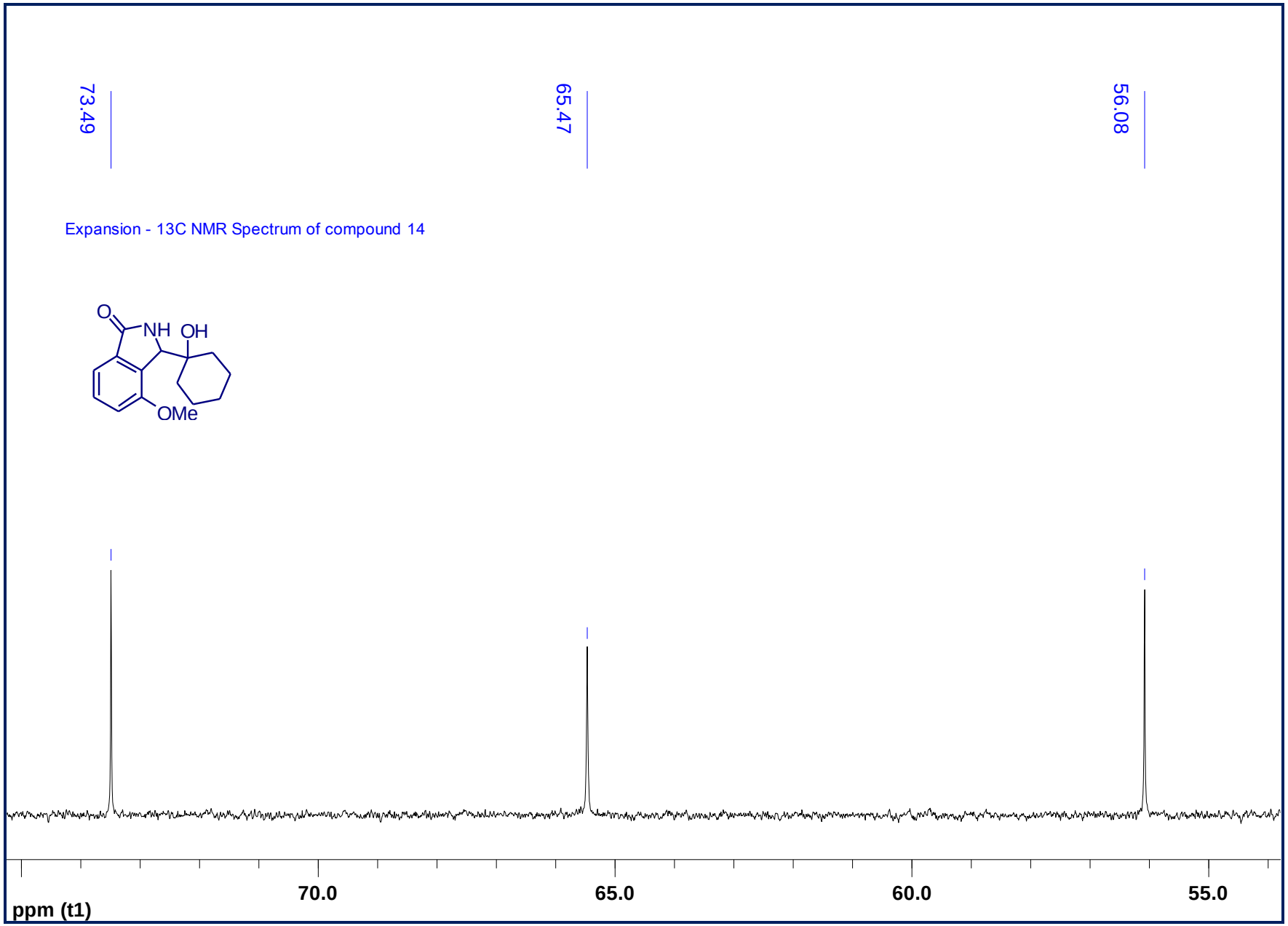
Expansion - 1H NMR Spectrum of compound 14

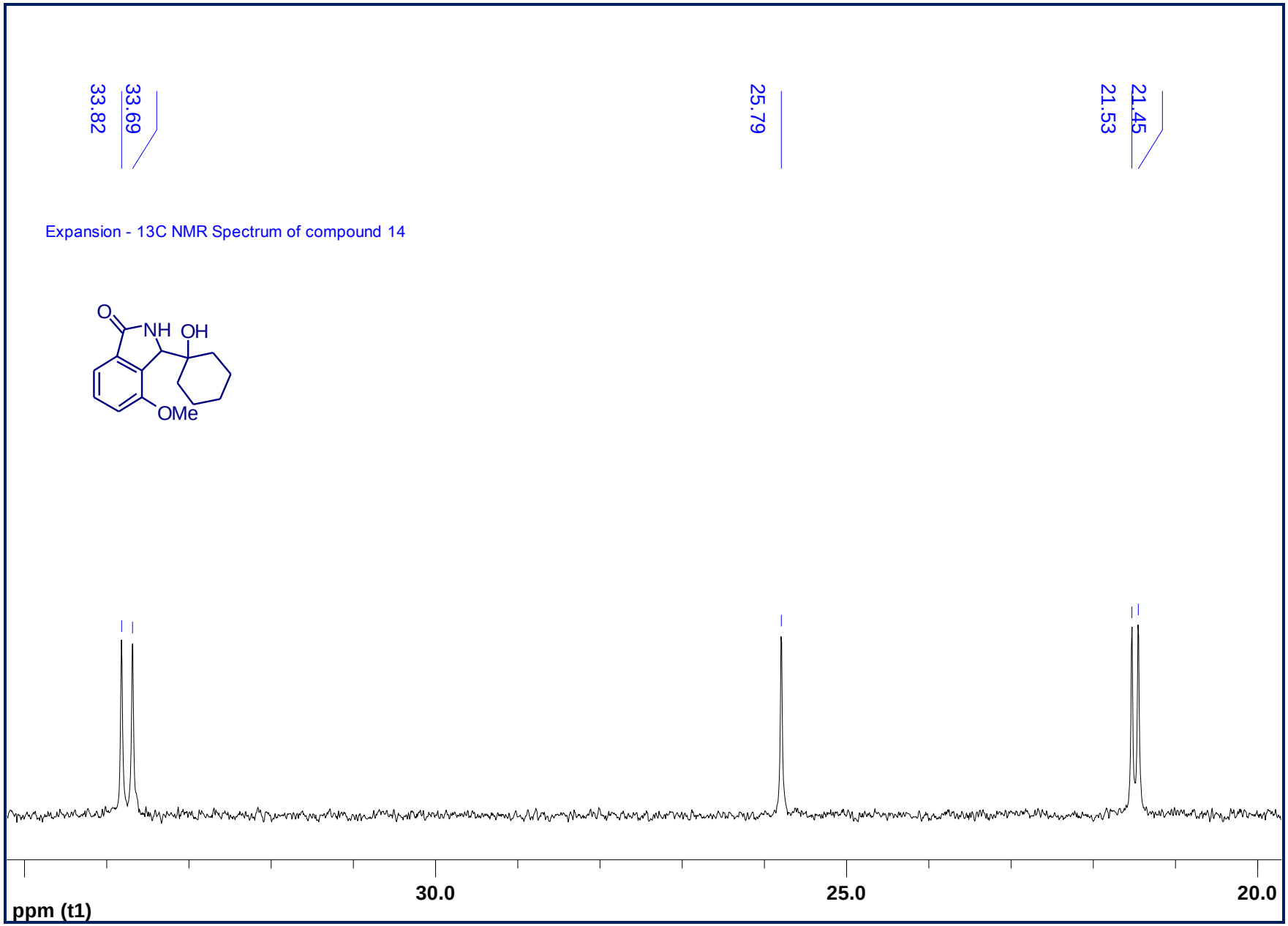


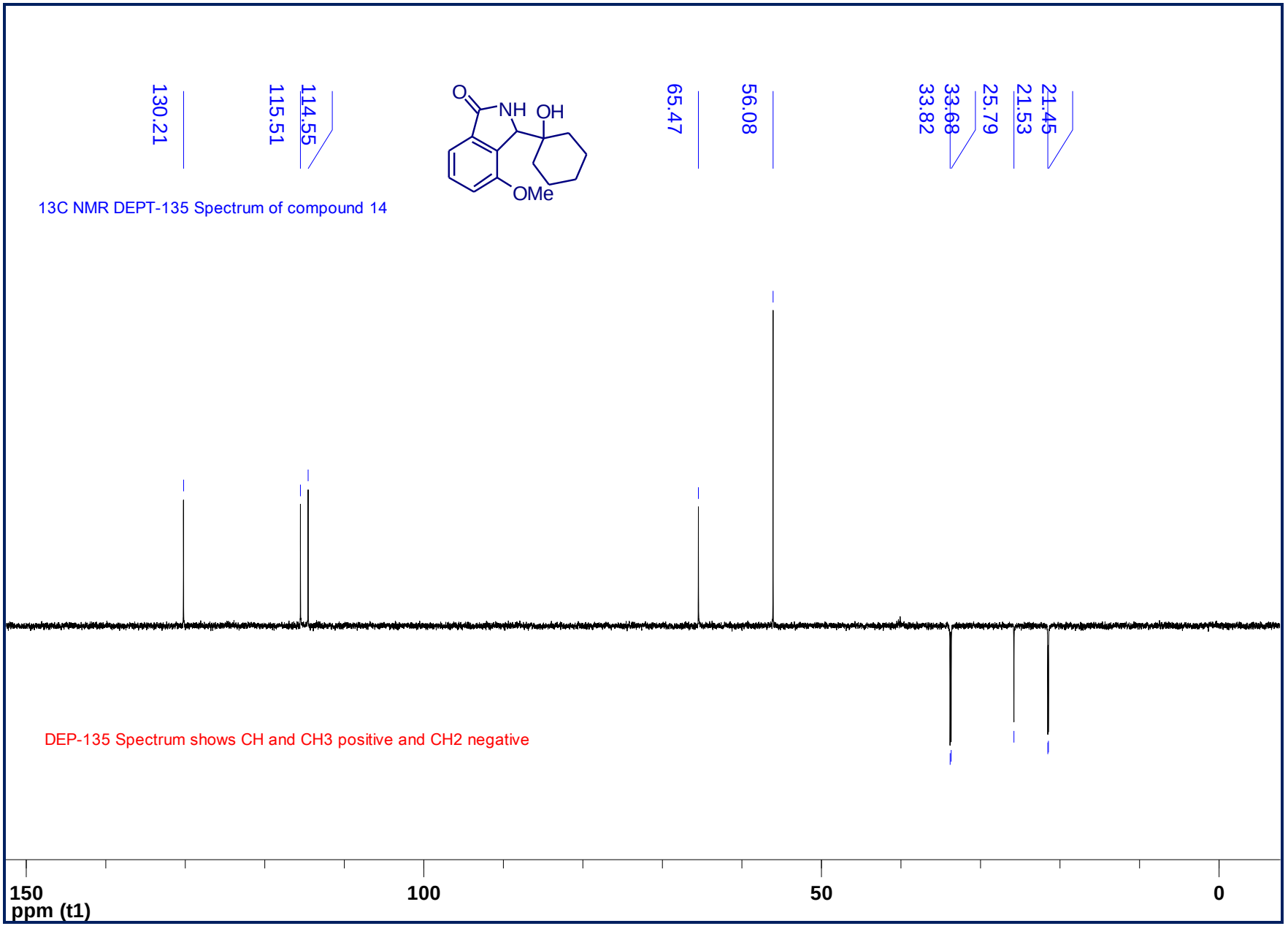


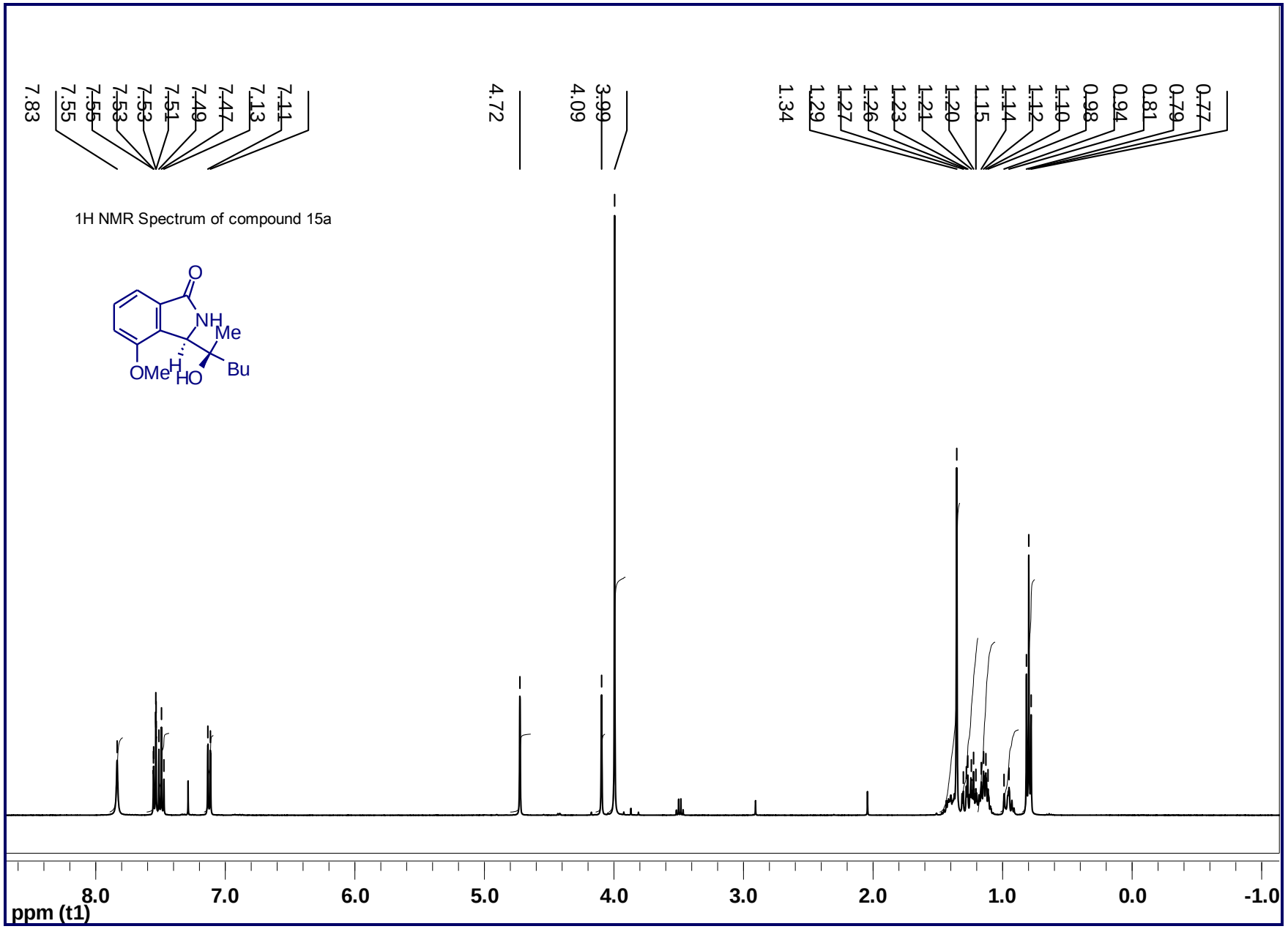

169.83
154.99
135.86
132.04
130.21
115.51
114.55
98.70
73.49
65.47
56.08
33.82
33.69
25.79
21.53
21.45
13C NMR Spectrum of compound 14
O
NH
OH
OMe
150
100
50
0
ppm (t1)


169.83
154.99
135.86
132.04
130.21
115.51
114.55
Expansion - 13C NMR Spectrum of compound 14
O
NH
OH
OMe
170
160
150
140
130
120
110
ppm (t1)

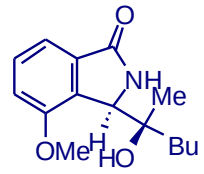

73.49
65.47
56.08
Expansion - 13C NMR Spectrum of compound 14
O
NH
OH
OMe
70.0
65.0
60.0
55.0
ppm (t1)

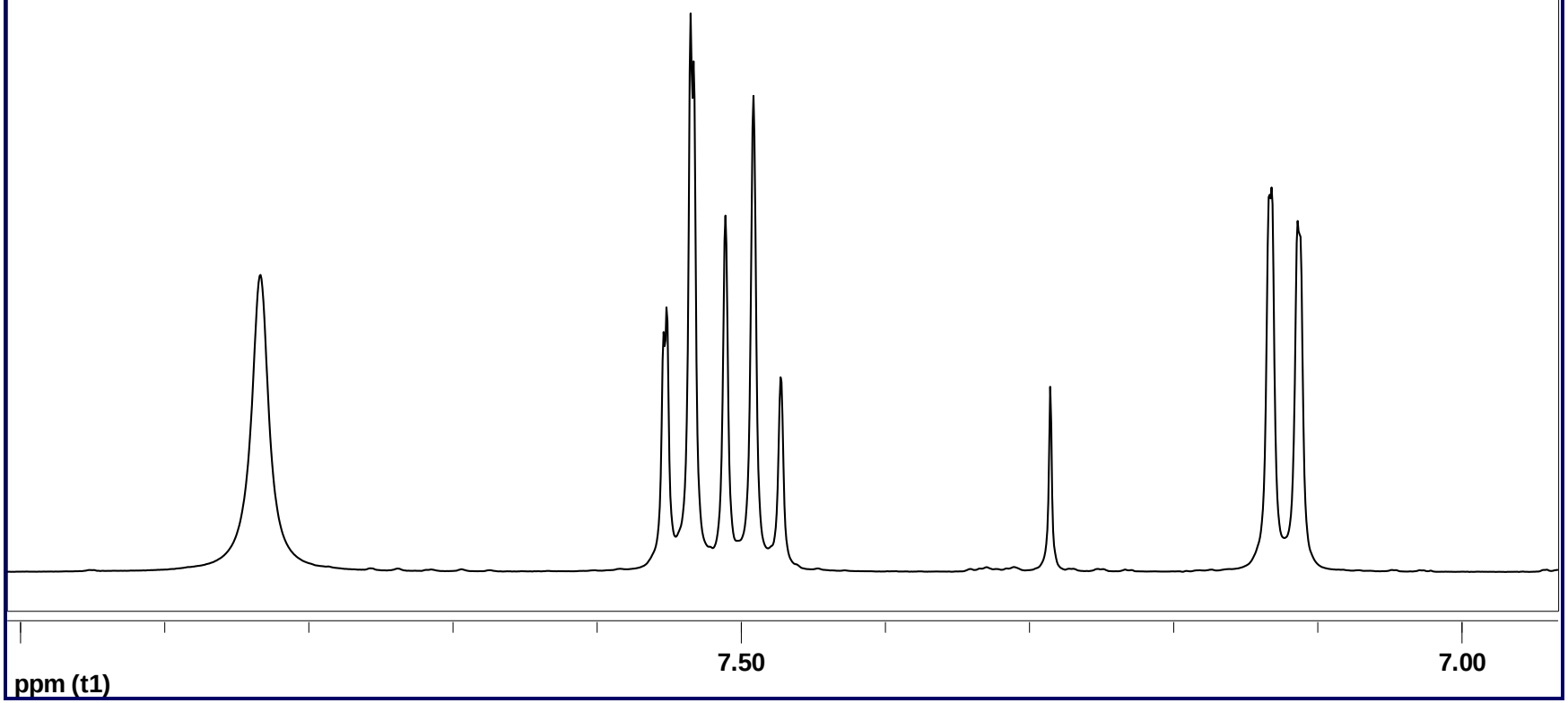

33.82
33.69
25.79
21.53
21.45
Expansion - 13C NMR Spectrum of compound 14
O
NH
OH
OMe
30.0
25.0
20.0
ppm (t1)

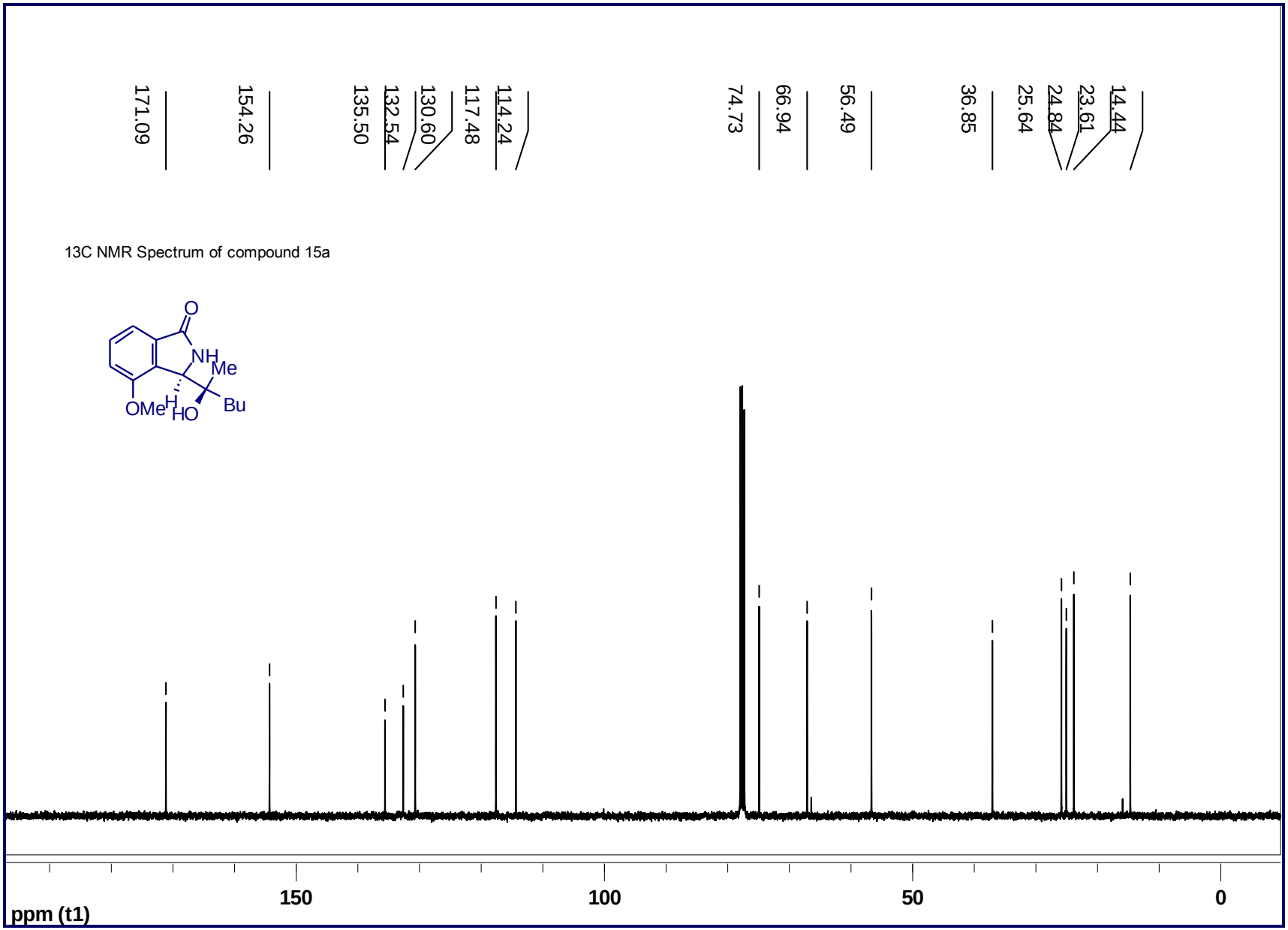

130.21
115.51
114.55
65.47
56.08
33.82
33.68
25.79
21.53
21.45
O
NH
OH
OMe
13C NMR DEPT-135 Spectrum of compound 14
DEP-135 Spectrum shows CH and CH3 positive and CH2 negative
150
ppm (t1)
100
50
0

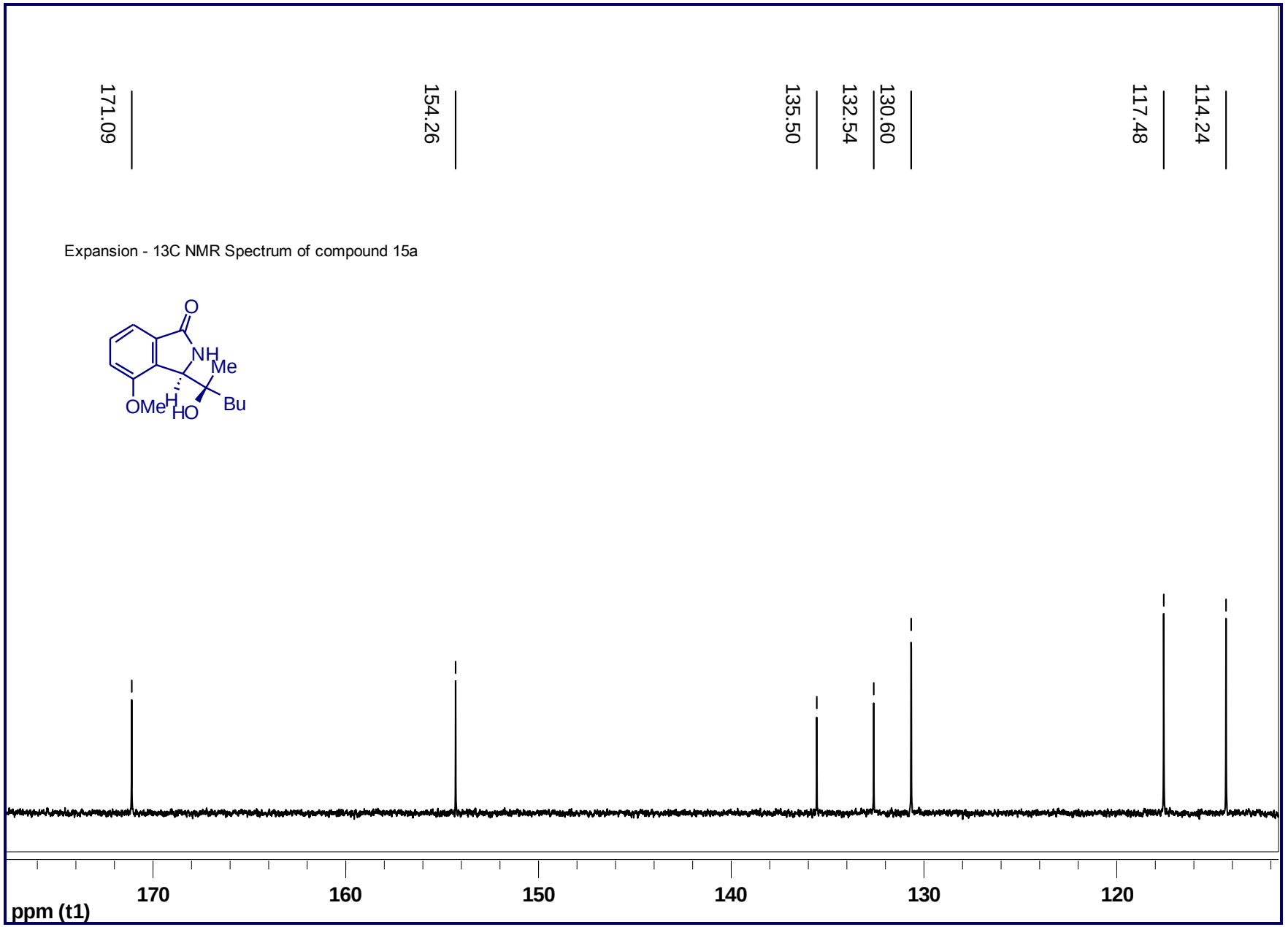

7.83
7.55
7.55
7.53
7.53
7.51
7.49
7.47
7.13
7.11
4.72
4.09
3.99
1.34
1.29
1.27
1.26
1.23
1.21
1.20
1.15
1.14
1.12
1.10
0.98
0.94
0.81
0.79
0.77
1H NMR Spectrum of compound 15a
O
NH
Me
OMe
H
HO
Bu
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (t1)

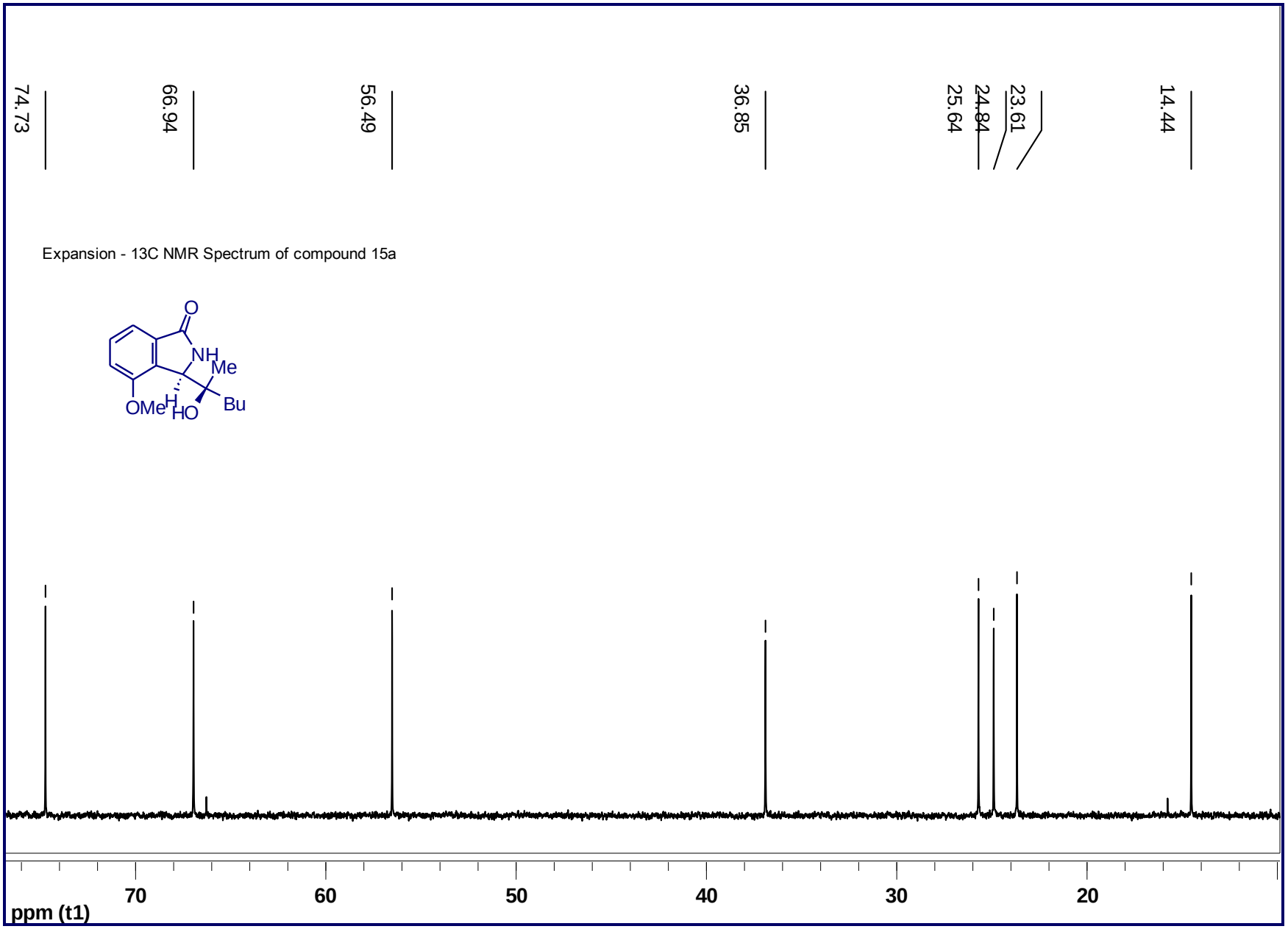
Expansion - 1H NMR Spectrum of compound 15a

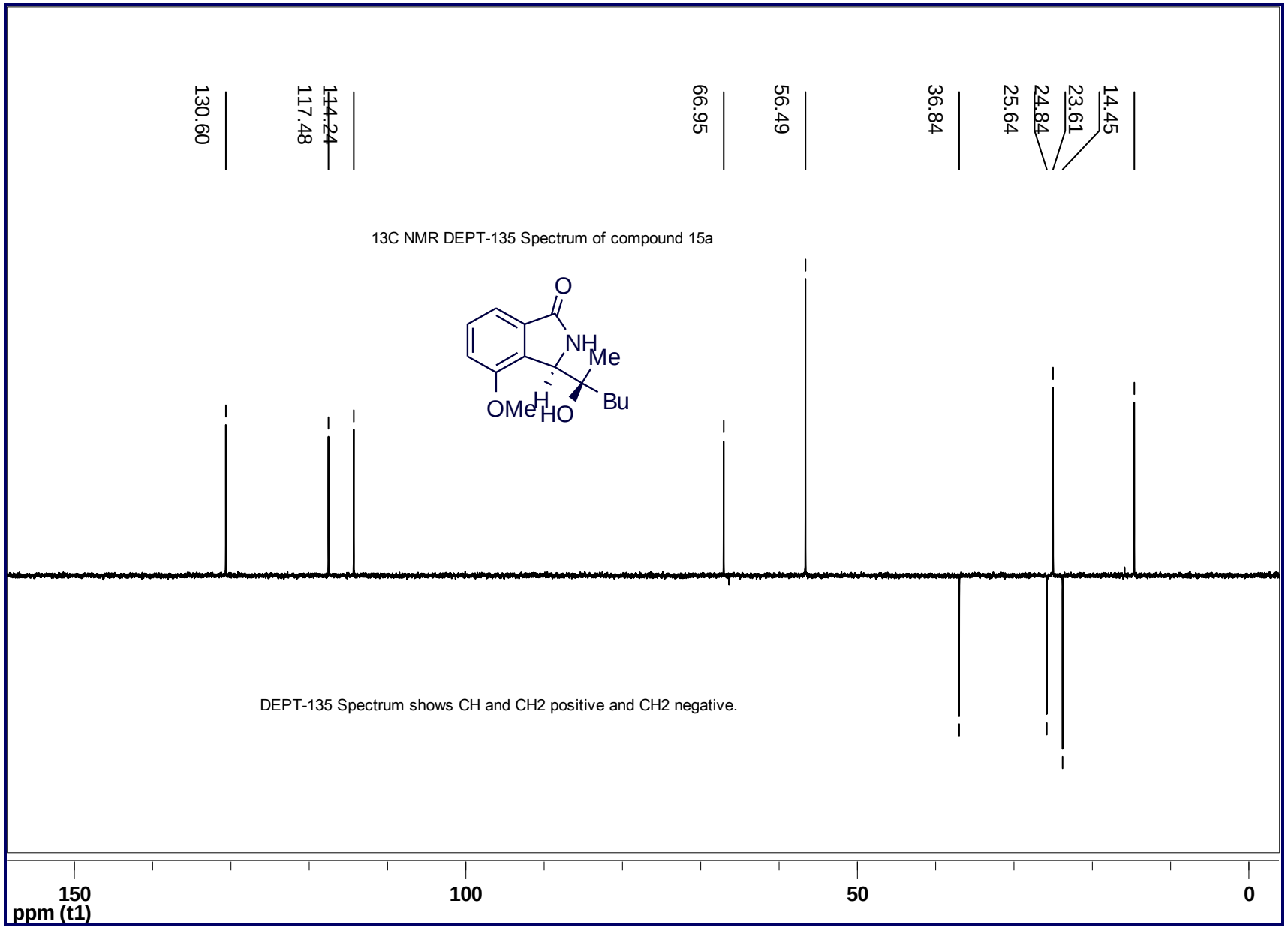


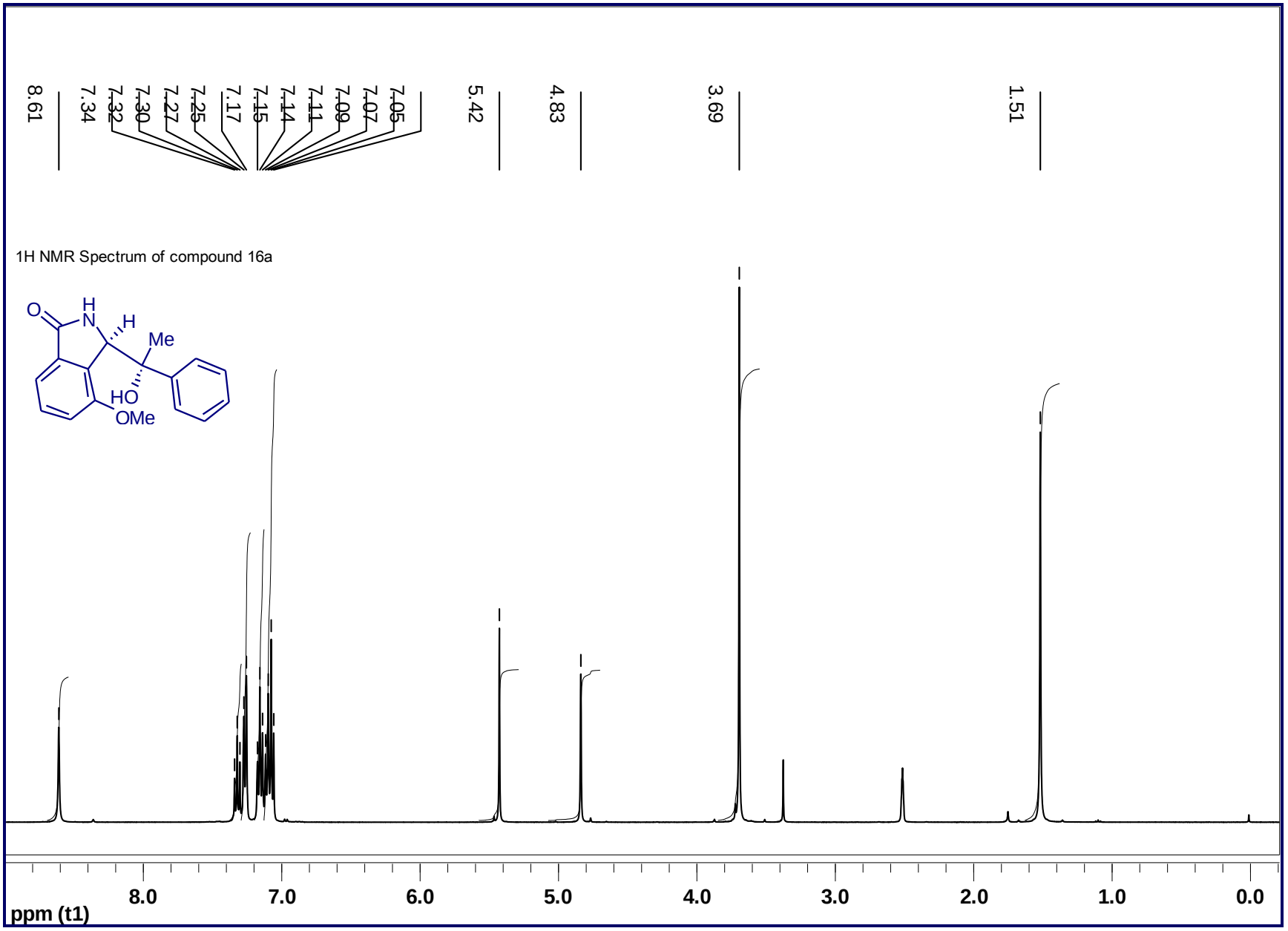



171.09
154.26
135.50
132.54
130.60
117.48
114.24
74.73
66.94
56.49
36.85
25.64
24.84
23.61
14.44
13C NMR Spectrum of compound 15a
O
NH
Me
OMe
H
HO
Bu
150
100
50
0
ppm (t1)

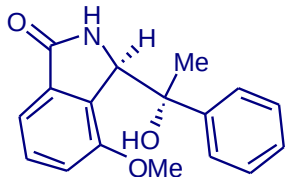

171.09
154.26
135.50
132.54
130.60
117.48
114.24
Expansion - 13C NMR Spectrum of compound 15a
O
NH
Me
OMe
H
HO
Bu
170
160
150
140
130
120
ppm (t1)


74.73
66.94
56.49
36.85
25.64
24.84
23.61
14.44
Expansion - 13C NMR Spectrum of compound 15a
O
NH
Me
H
OMe
HO
Bu
70
60
50
40
30
20
ppm (t1)

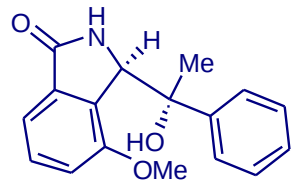

130.60
117.48
114.24
66.95
56.49
36.84
25.64
24.84
23.61
14.45
13C NMR DEPT-135 Spectrum of compound 15a
O
NH
Me
OMe
H
HO
Bu
DEPT-135 Spectrum shows CH and CH2 positive and CH2 negative.
150
100
50
0
ppm (t1)


8.61
7.34
7.32
7.30
7.27
7.25
7.17
7.15
7.14
7.11
7.09
7.07
7.05
5.42
4.83
3.69
1.51
1H NMR Spectrum of compound 16a
O
H
N
H
Me
HO
OMe
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

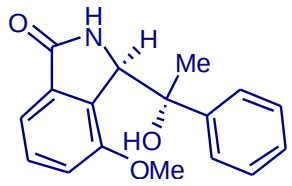
Expansion - 1H NMR Spectrum of compound 16a

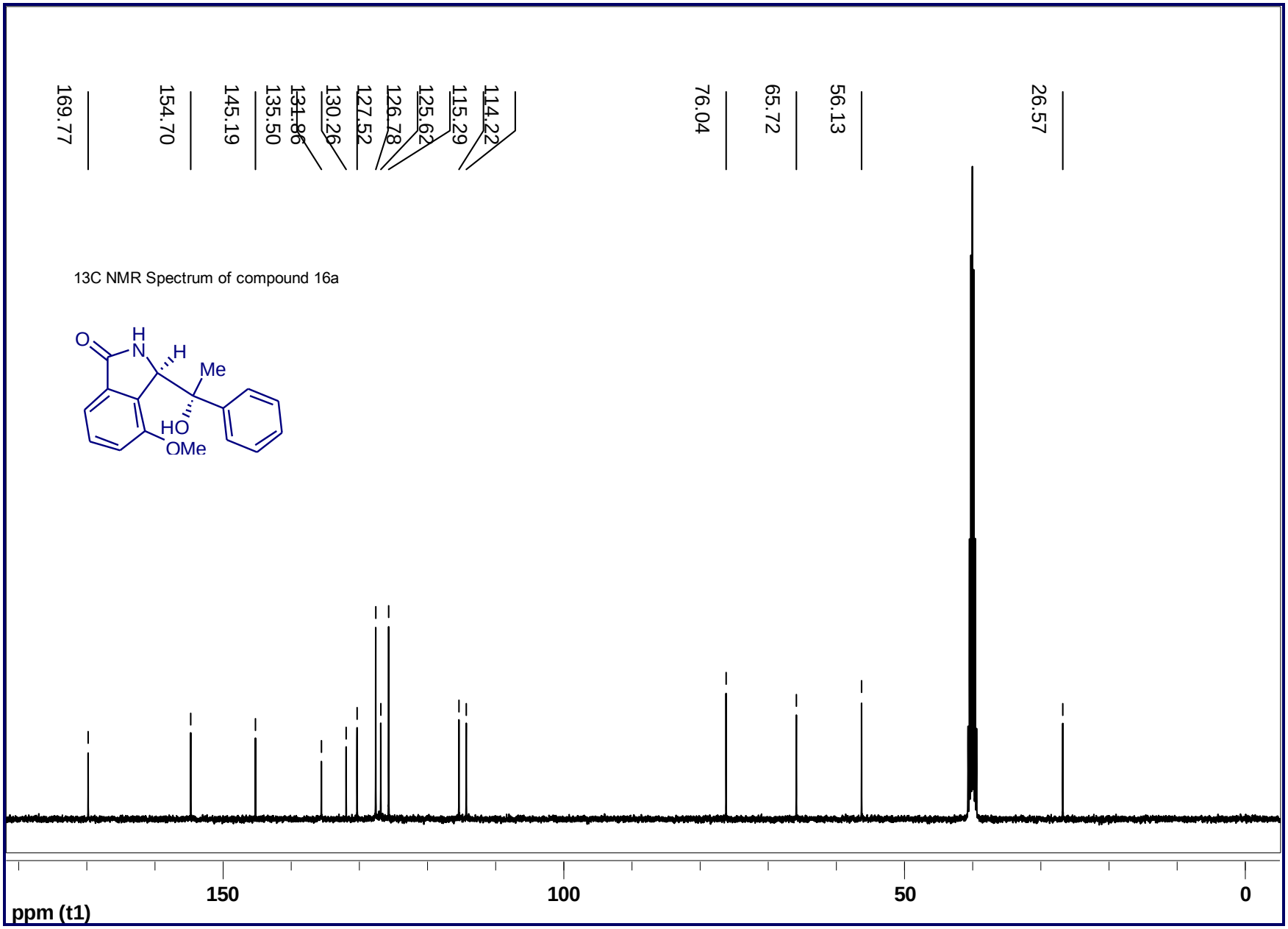


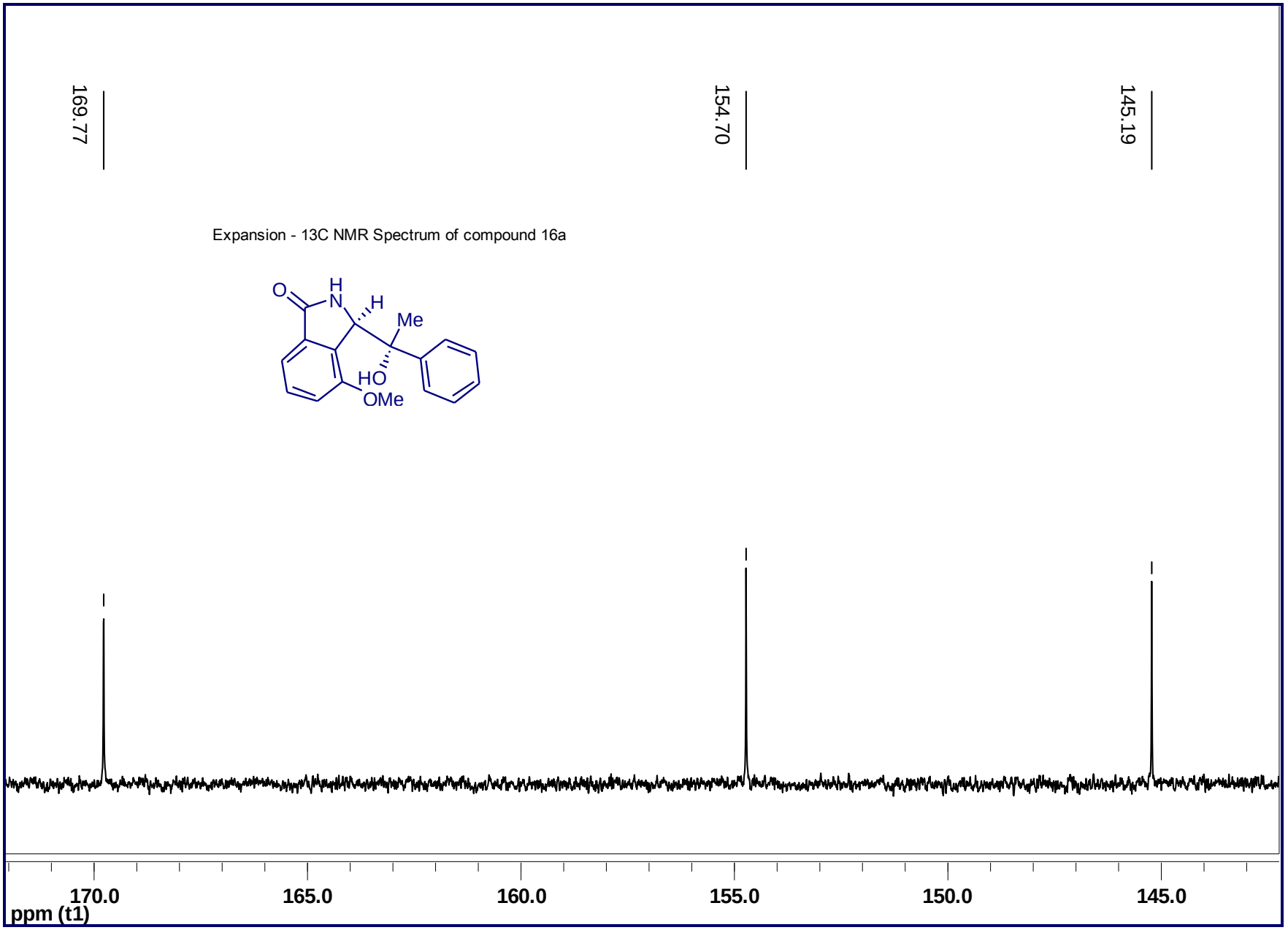
Expansion - 1H NMR Spectrum of compound 16a

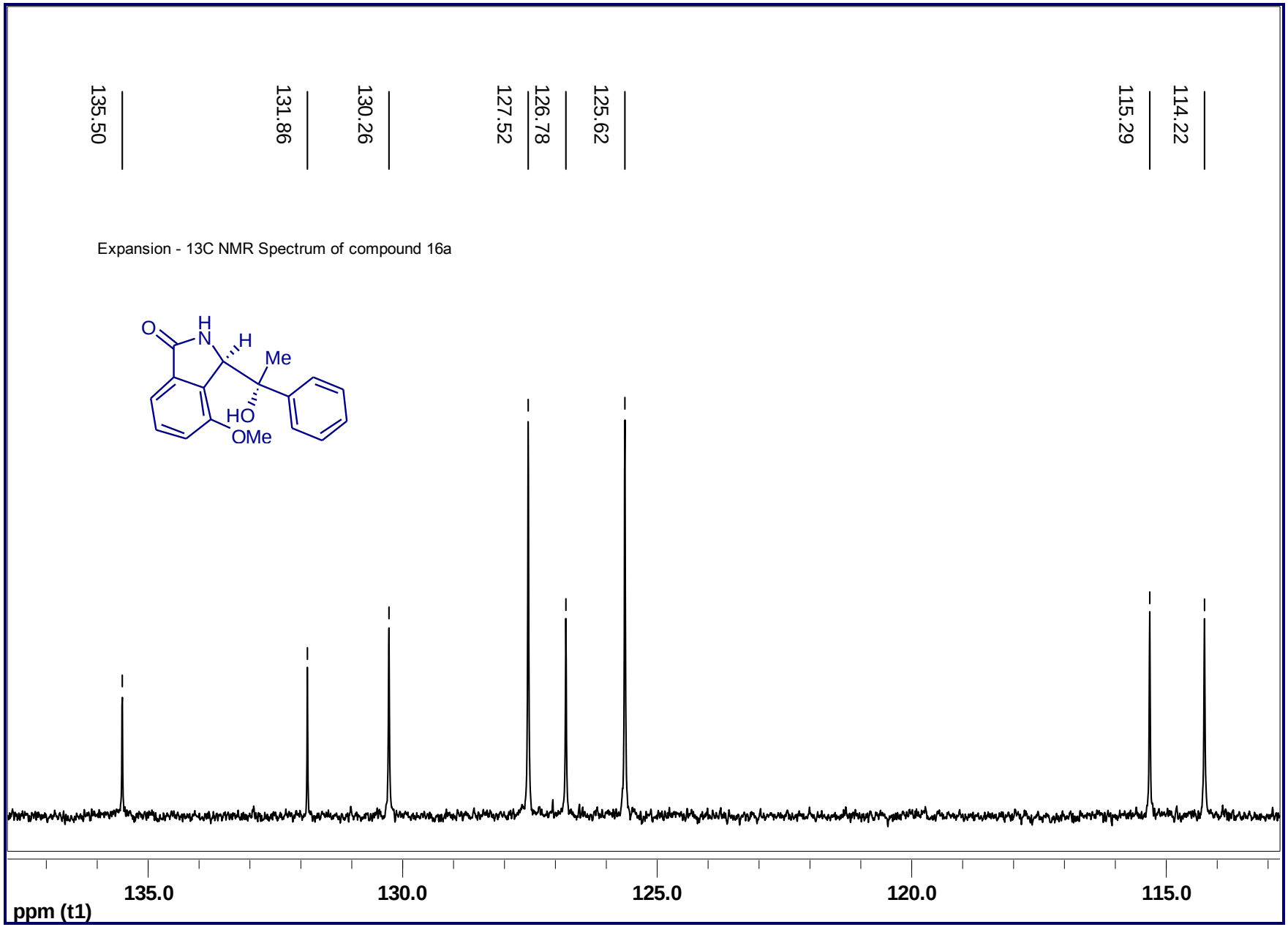


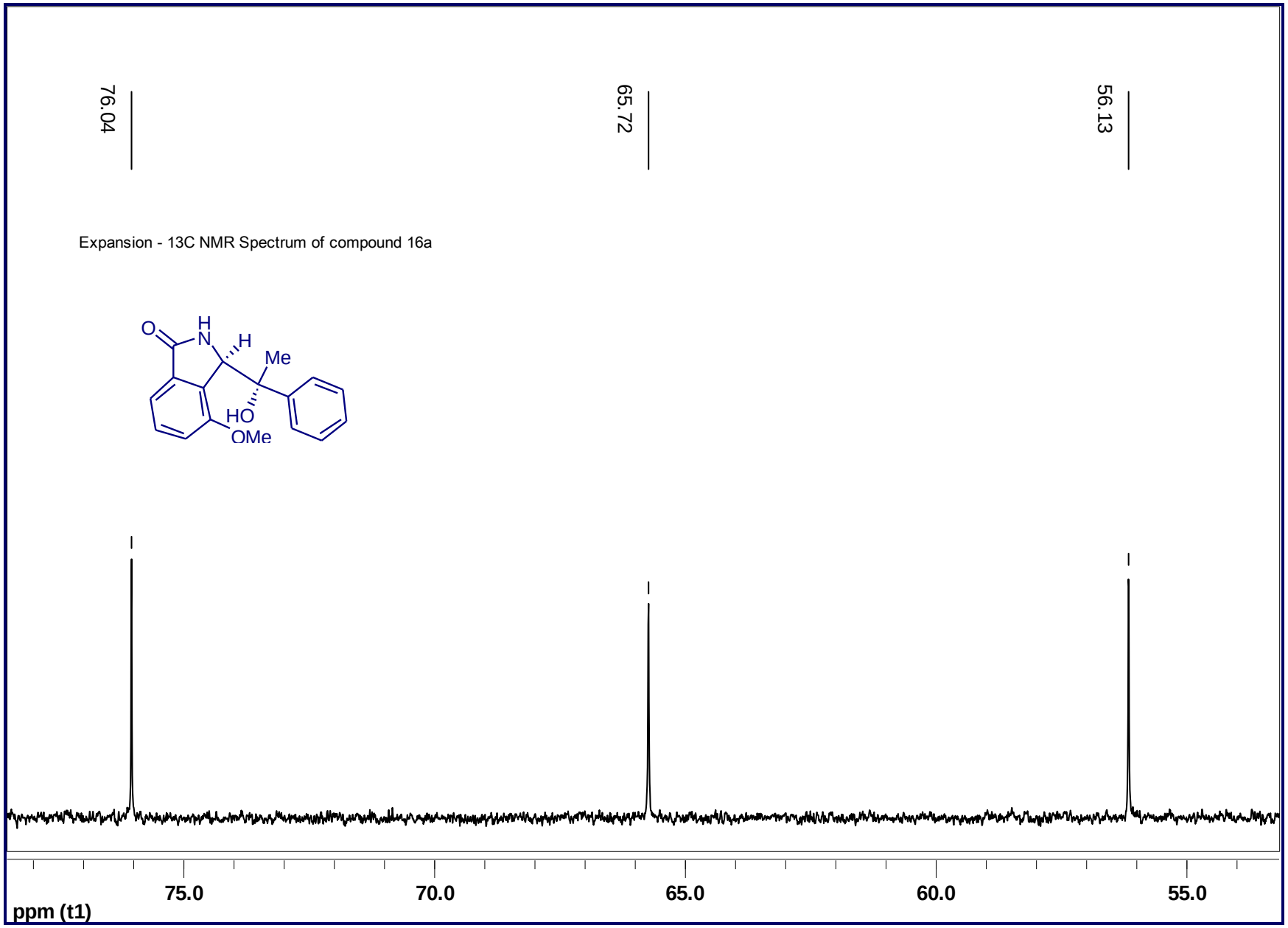
Expansion - 1H NMR Spectrum of compound 16a

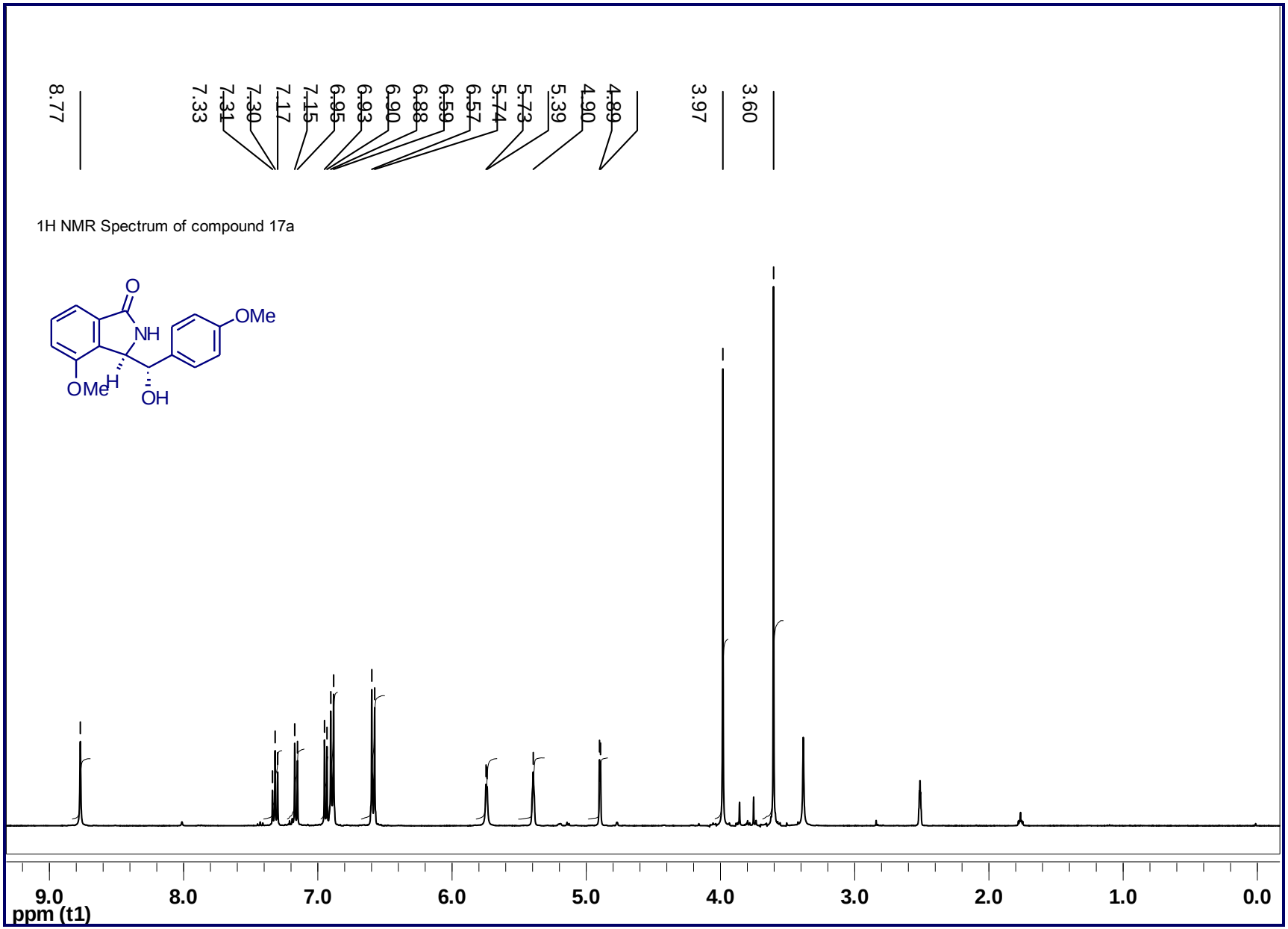



169.77
154.70
145.19
135.50
131.86
130.26
127.52
126.78
125.62
115.29
114.22
76.04
65.72
56.13
26.57
13C NMR Spectrum of compound 16a
O
H
N
H
Me
HO
OMe
150
100
50
0
ppm (t1)

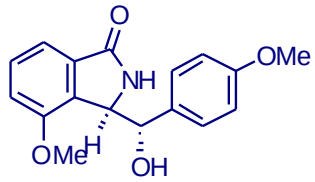

169.77
154.70
145.19
Expansion - 13C NMR Spectrum of compound 16a
O
H
N
H
Me
HO
OMe
170.0
165.0
160.0
155.0
150.0
145.0
ppm (t1)

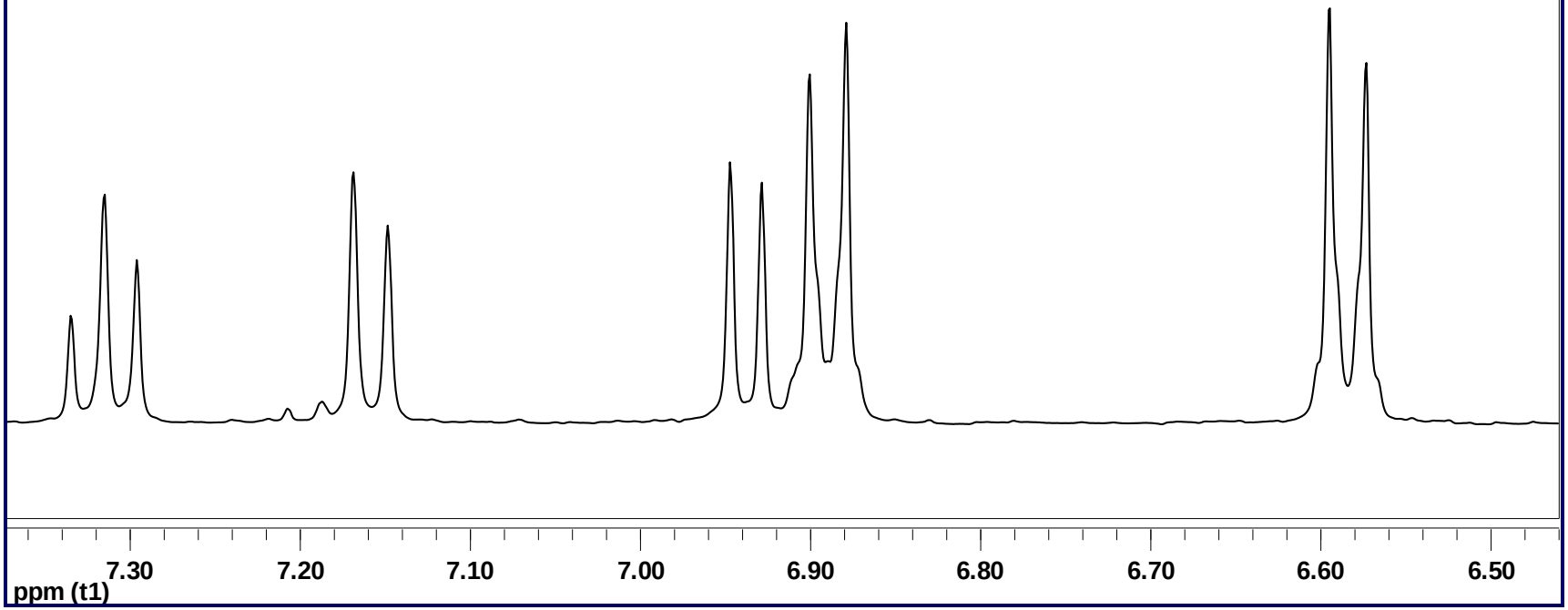

135.50
131.86
130.26
127.52
126.78
125.62
115.29
114.22
Expansion - 13C NMR Spectrum of compound 16a
O
H
N
H
Me
HO
OMe
135.0
130.0
125.0
120.0
115.0
ppm (t1)


76.04
65.72
56.13
Expansion - 13C NMR Spectrum of compound 16a
O
H
N
H
Me
HO
OMe
75.0
70.0
65.0
60.0
55.0
ppm (t1)

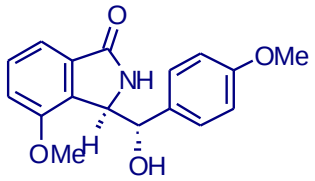

8.77
7.33
7.31
7.30
7.17
7.15
6.95
6.93
6.90
6.88
6.59
6.57
5.74
5.73
5.39
4.90
4.89
3.97
3.60
1H NMR Spectrum of compound 17a
O
NH
OMe
OMe
H
OH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

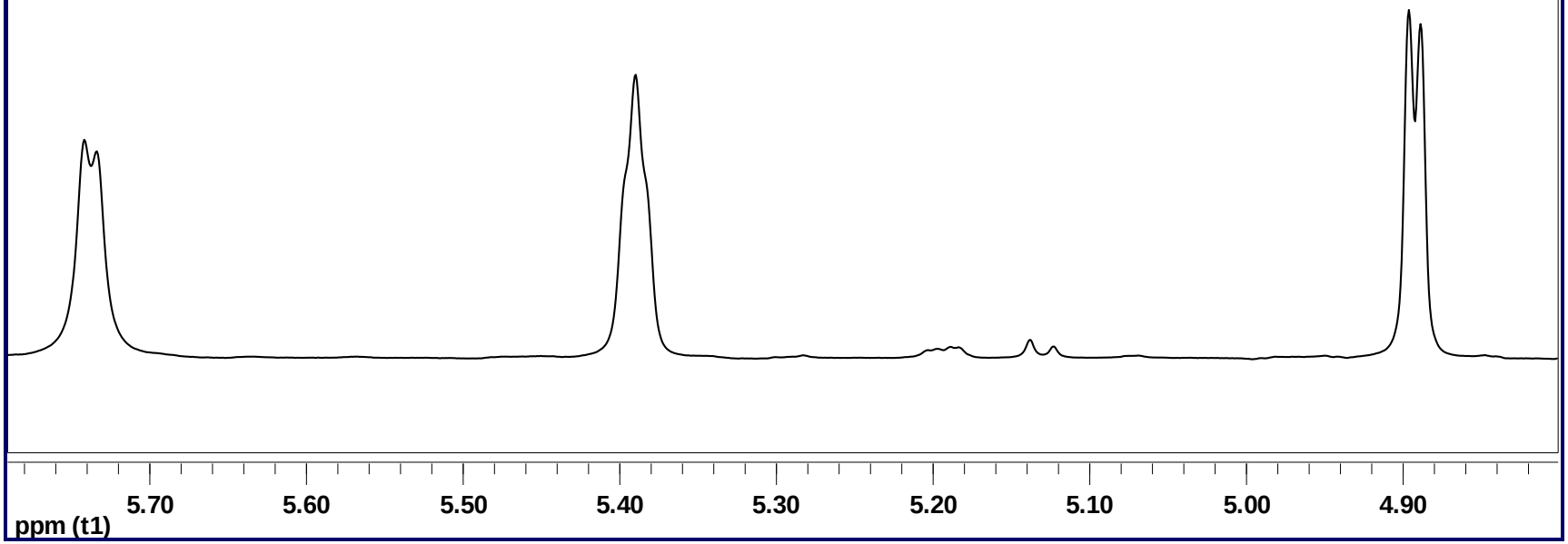
Expansion - 1H NMR Spectrum of compound 17a

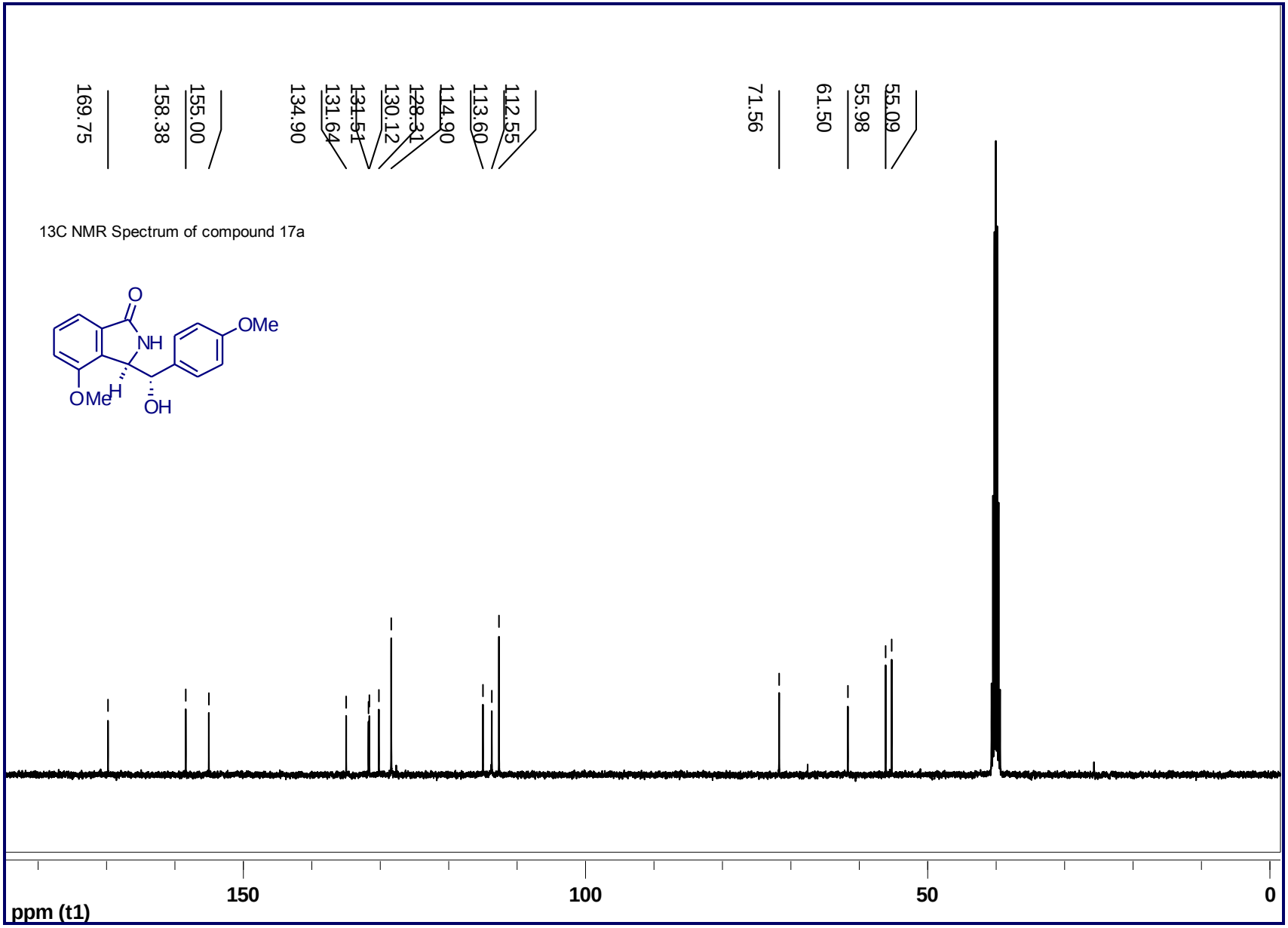


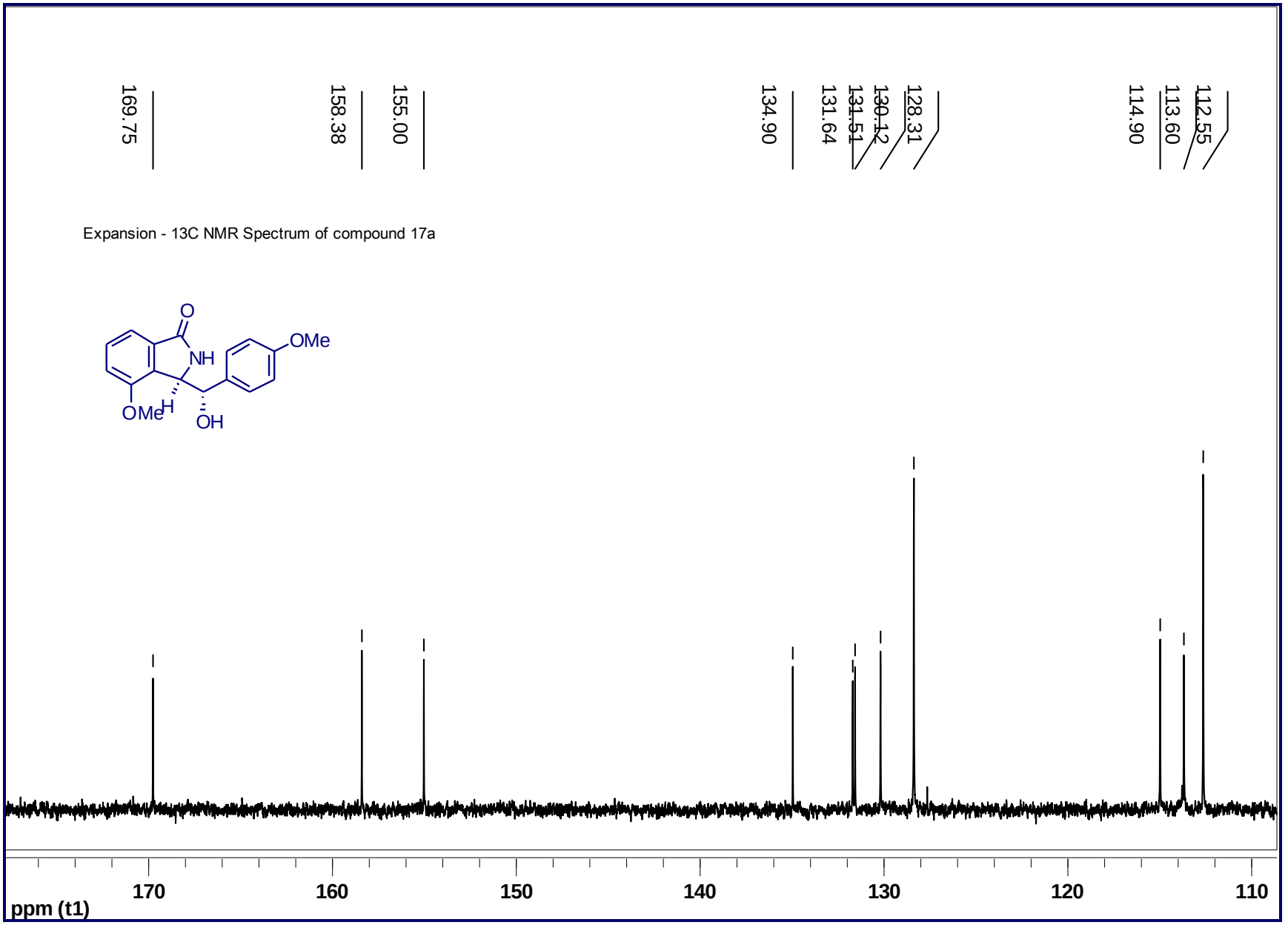


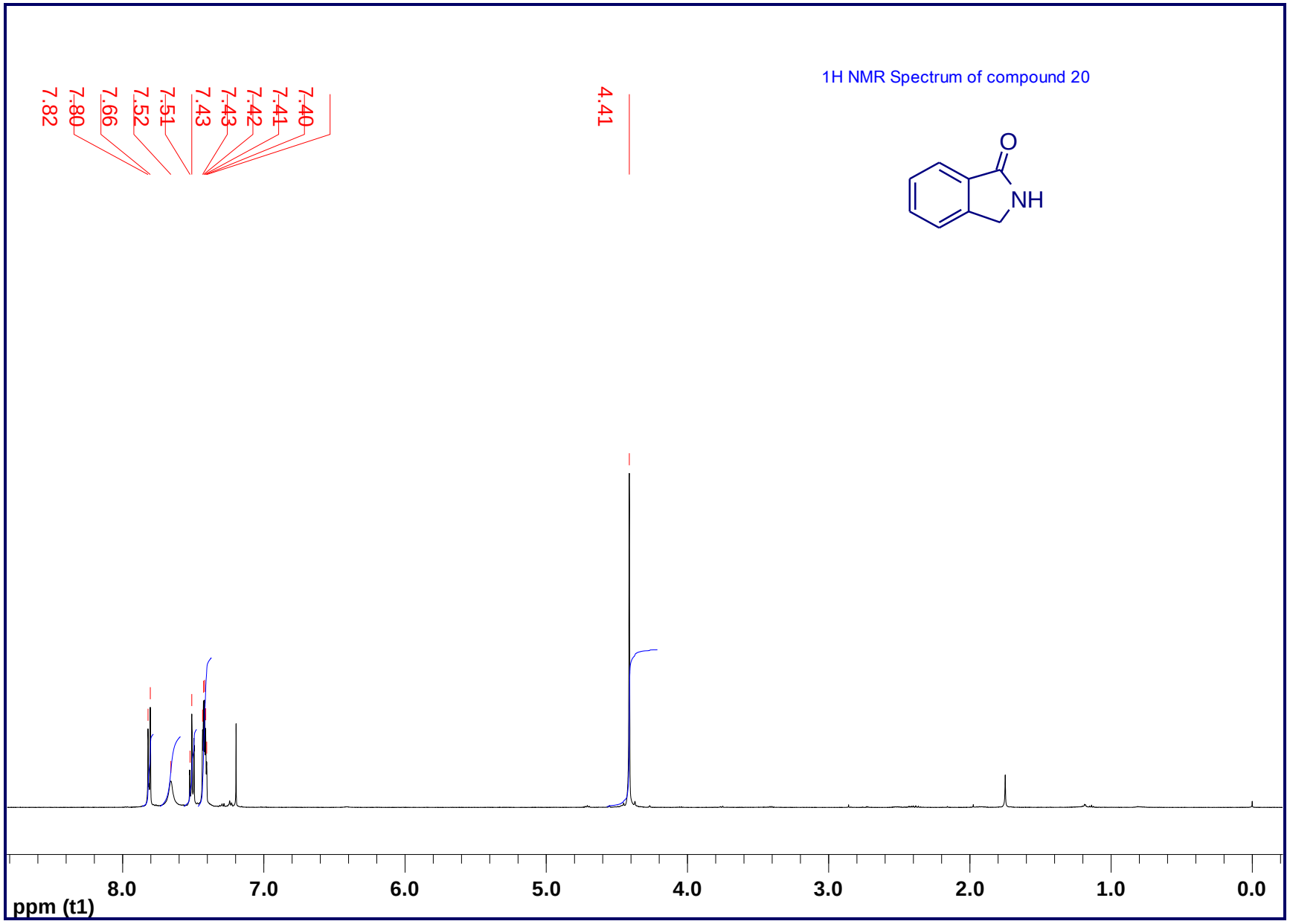
Expansion - 1H NMR Spectrum of compound 17a

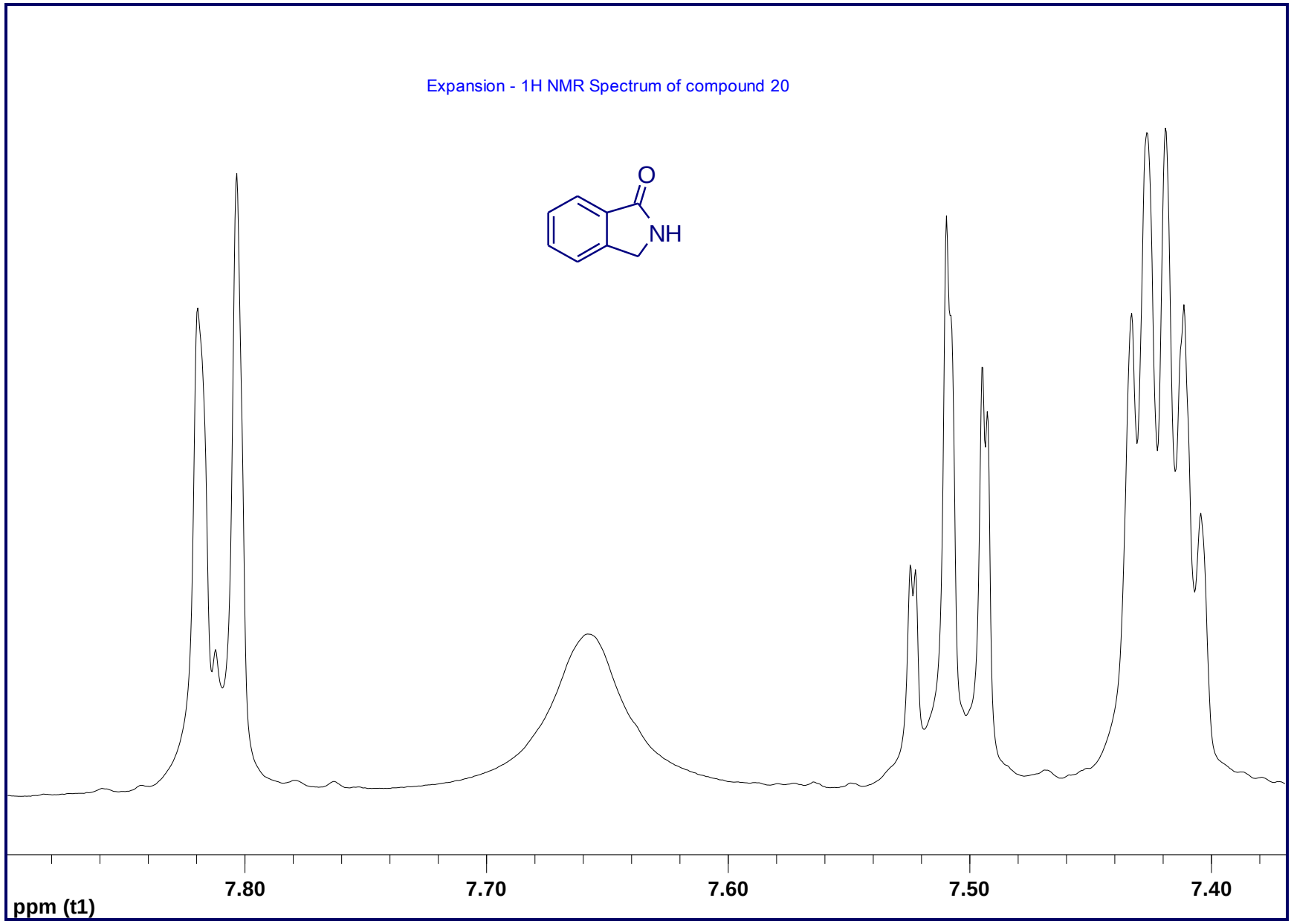


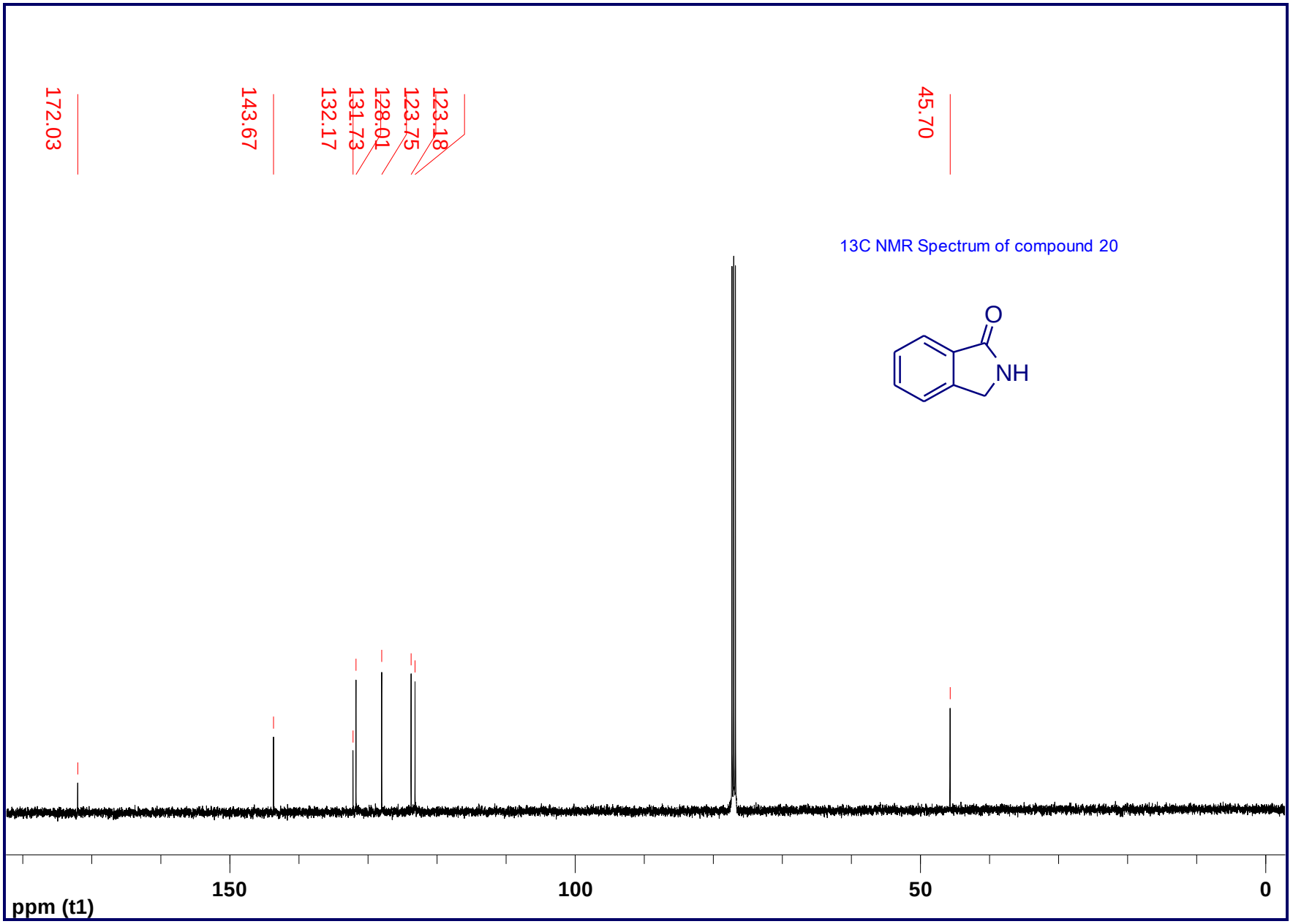


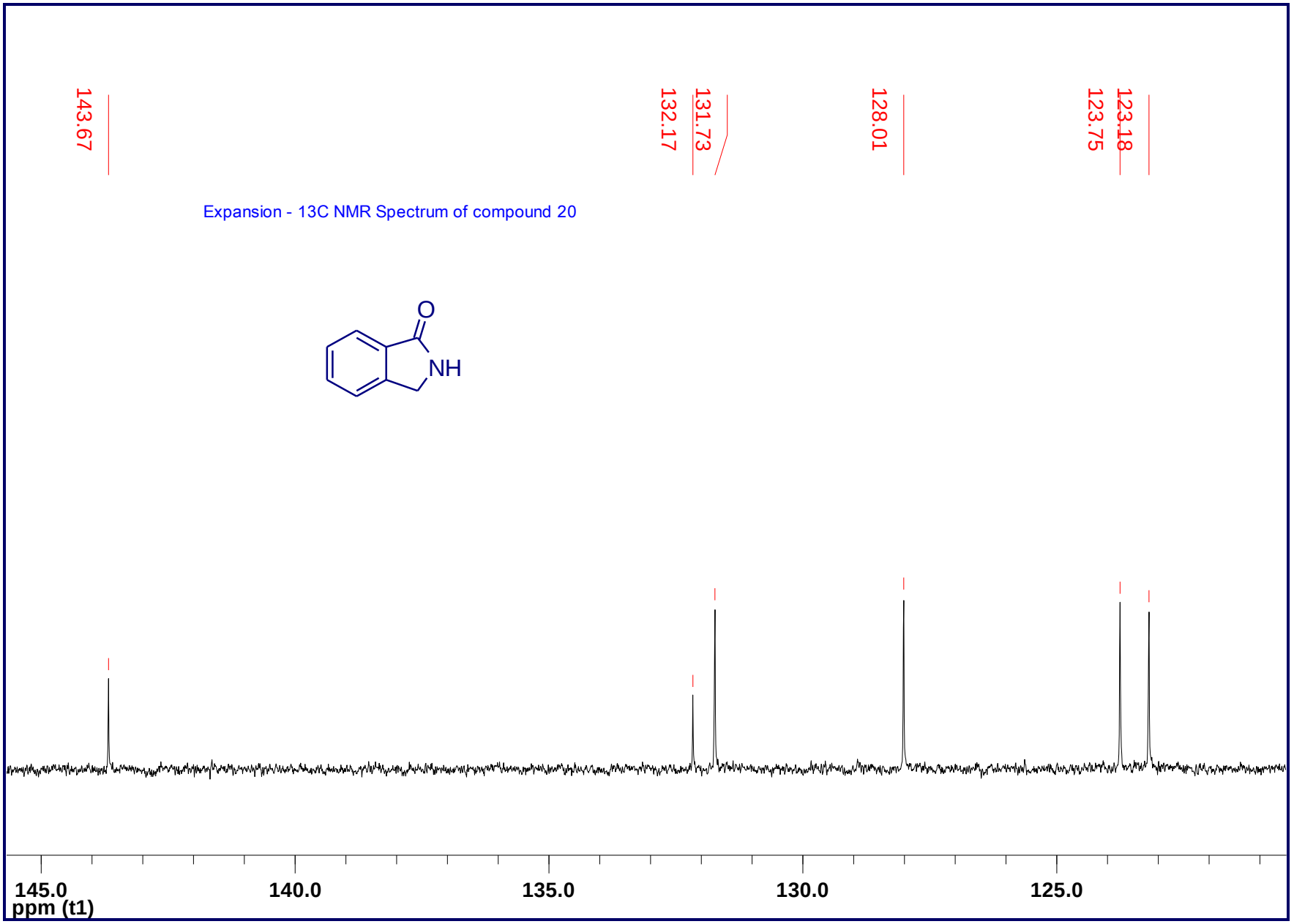

169.75
158.38
155.00
134.90
131.64
131.51
130.12
128.31
114.90
113.60
112.55
71.56
61.50
55.98
55.09
13C NMR Spectrum of compound 17a
O
NH
OMe
OMe
H
OH
150
100
50
0
ppm (t1)

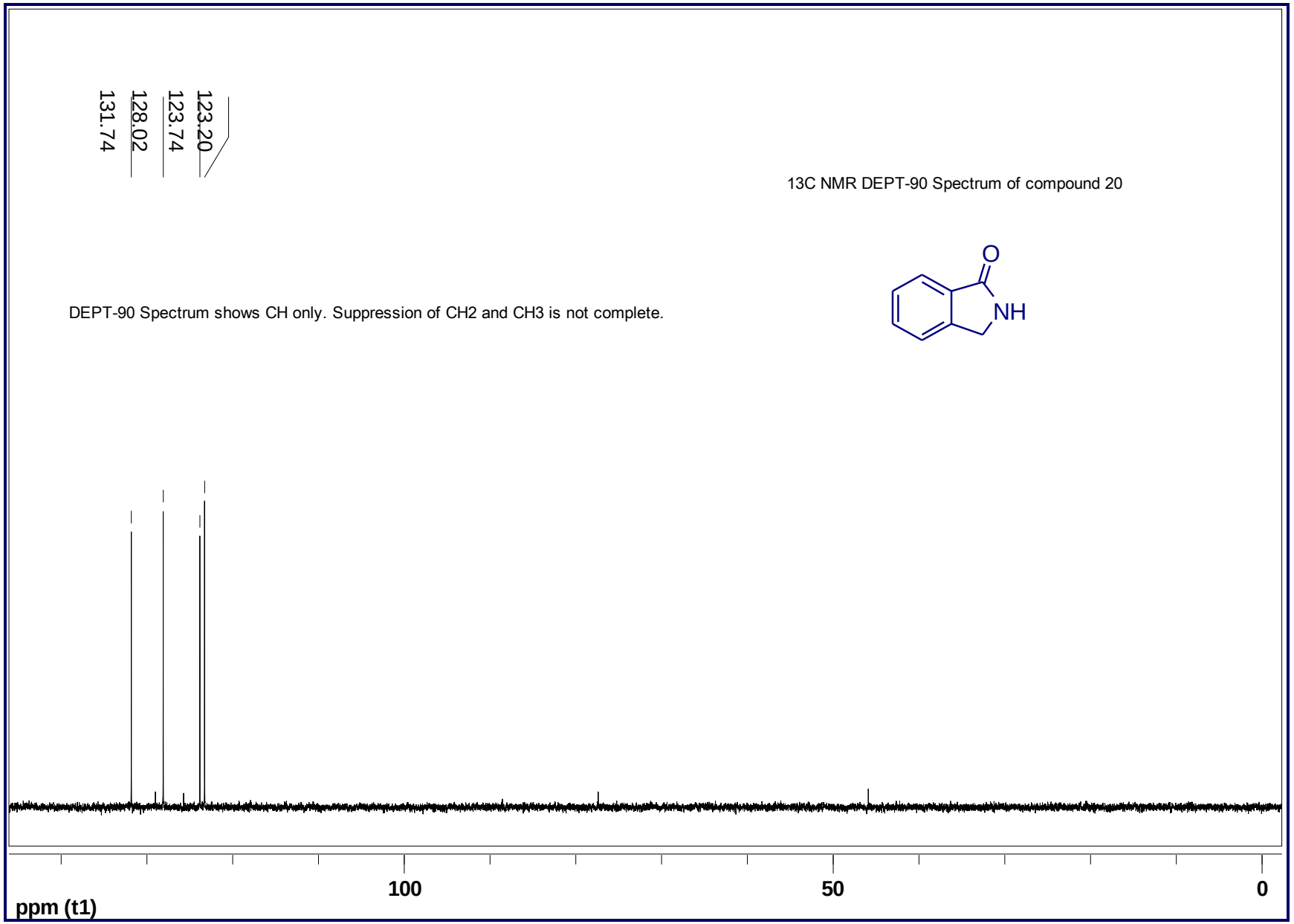

169.75
158.38
155.00
134.90
131.64
131.51
130.12
128.31
114.90
113.60
112.55
Expansion - 13C NMR Spectrum of compound 17a
O
NH
OMe
OMe
H
OH
170
160
150
140
130
120
110
ppm (t1)

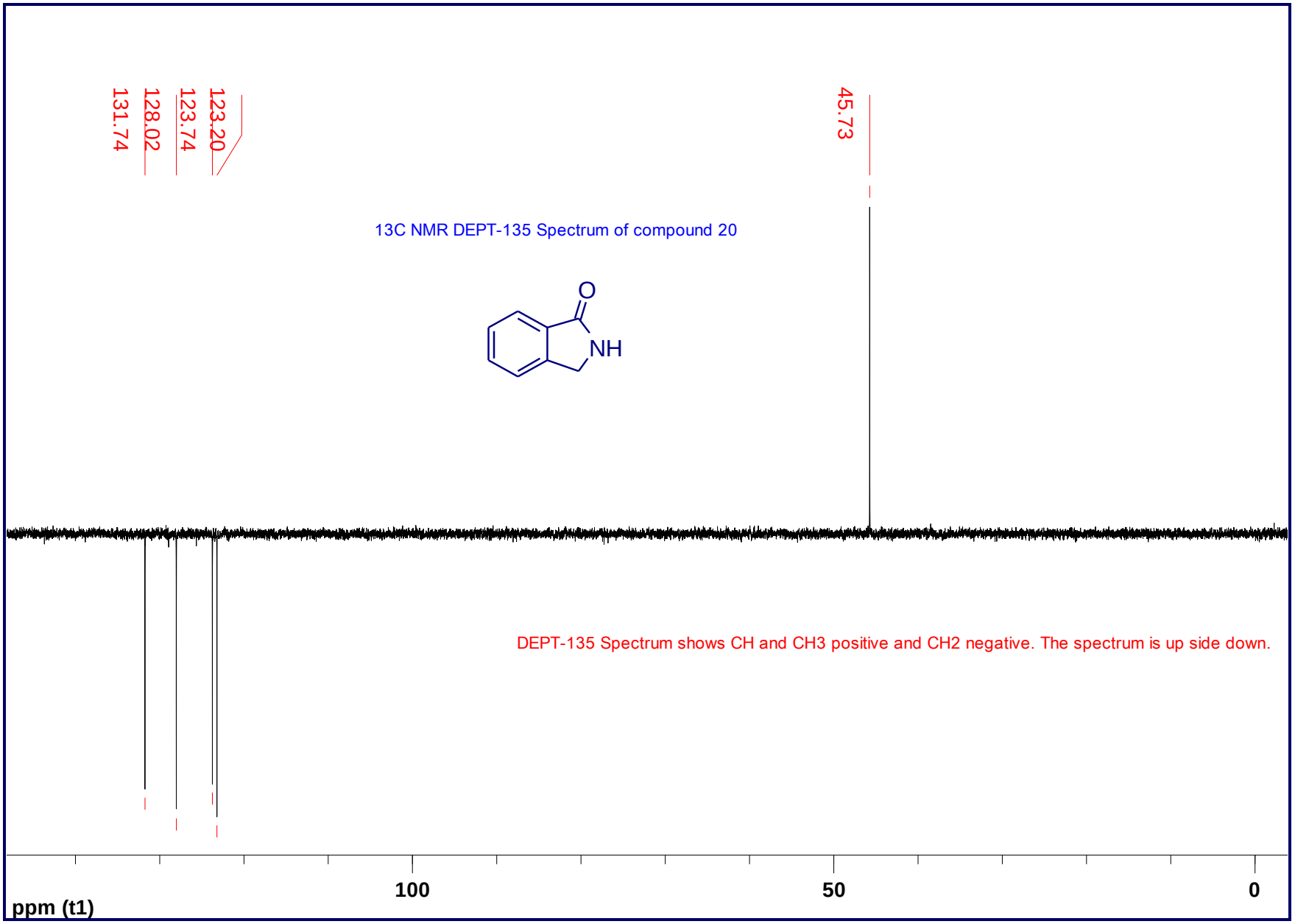

1H NMR Spectrum of compound 20
7.82
7.80
7.66
7.52
7.51
7.43
7.43
7.42
7.41
7.40
4.41
O
NH
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

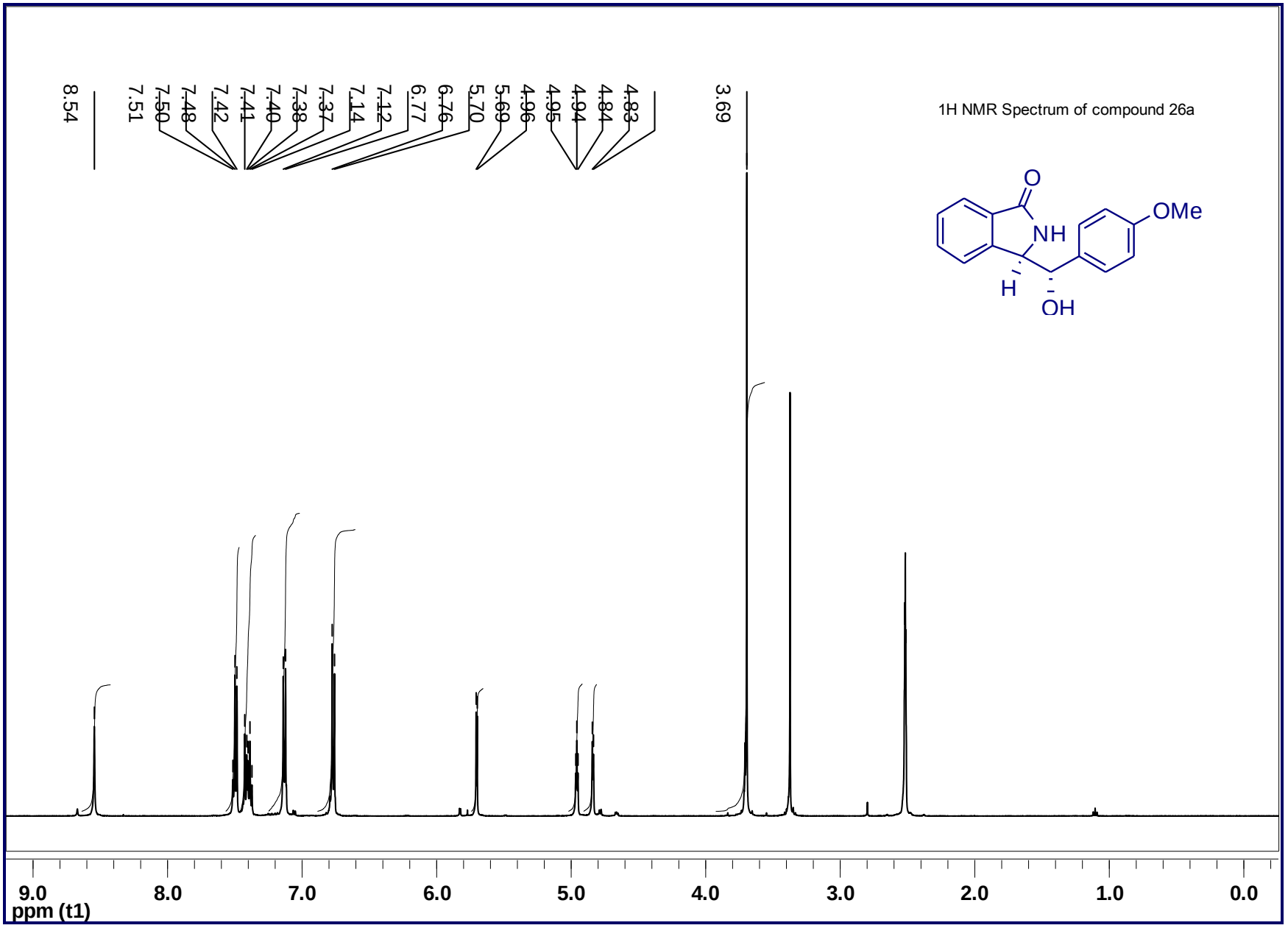

Expansion - 1H NMR Spectrum of compound 20
O
NH
7.80
7.70
7.60
7.50
7.40
ppm (t1)


172.03
143.67
132.17
131.73
128.01
123.75
123.18
45.70
13C NMR Spectrum of compound 20
O
NH
150
100
50
0
ppm (t1)

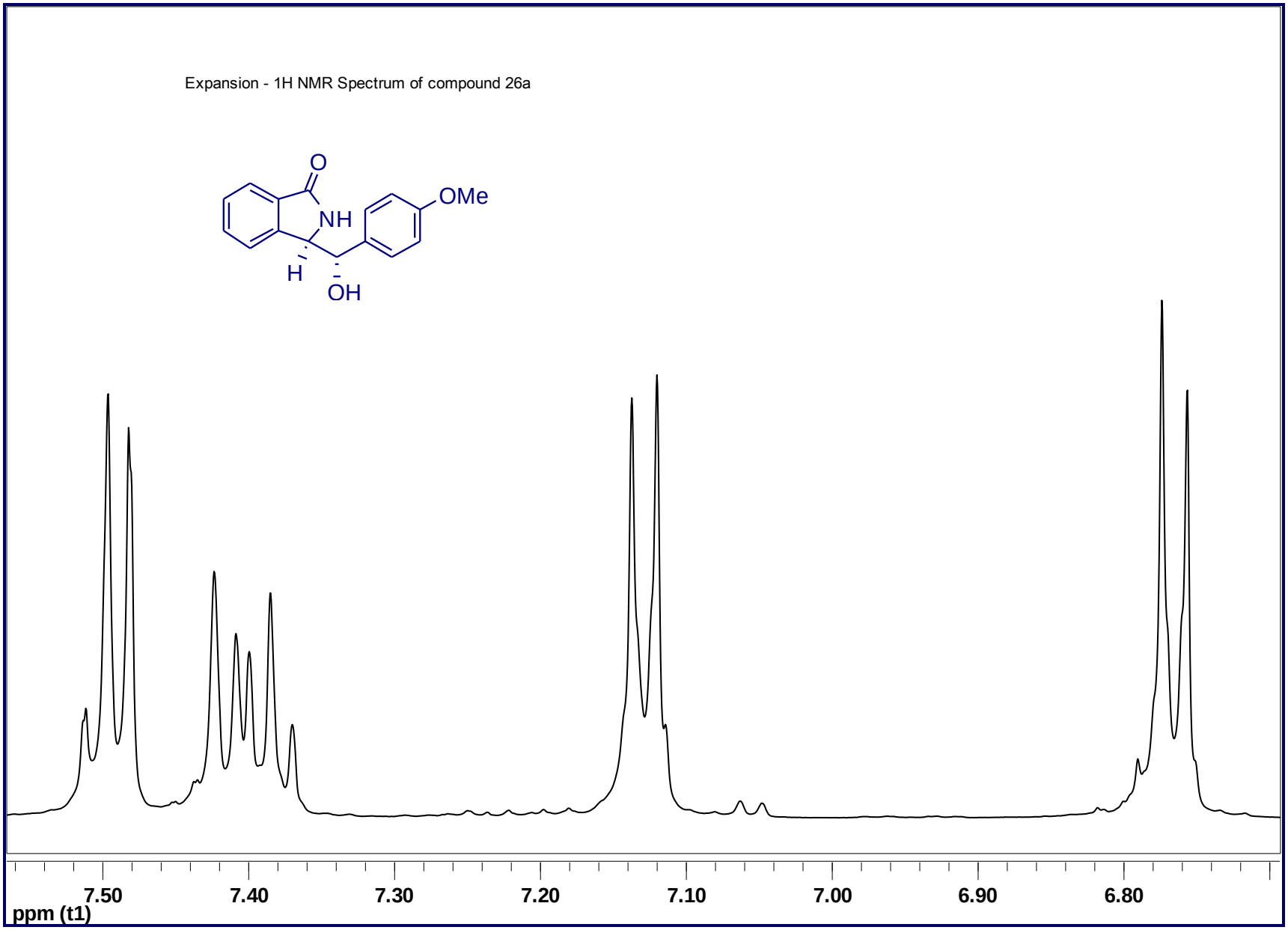

143.67
132.17
131.73
128.01
123.75
123.18
Expansion - 13C NMR Spectrum of compound 20
O
NH
145.0
ppm (t1)
140.0
135.0
130.0
125.0


131.74
128.02
123.74
123.20
13C NMR DEPT-90 Spectrum of compound 20
DEPT-90 Spectrum shows CH only. Suppression of CH2 and CH3 is not complete.
O
NH
100
50
0
ppm (t1)

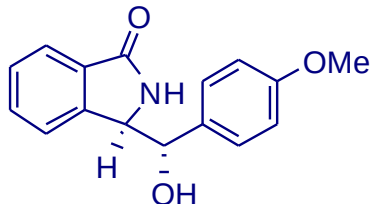

131.74
128.02
123.74
123.20
45.73
13C NMR DEPT-135 Spectrum of compound 20
O
NH
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative. The spectrum is up side down.
100
50
0
ppm (t1)

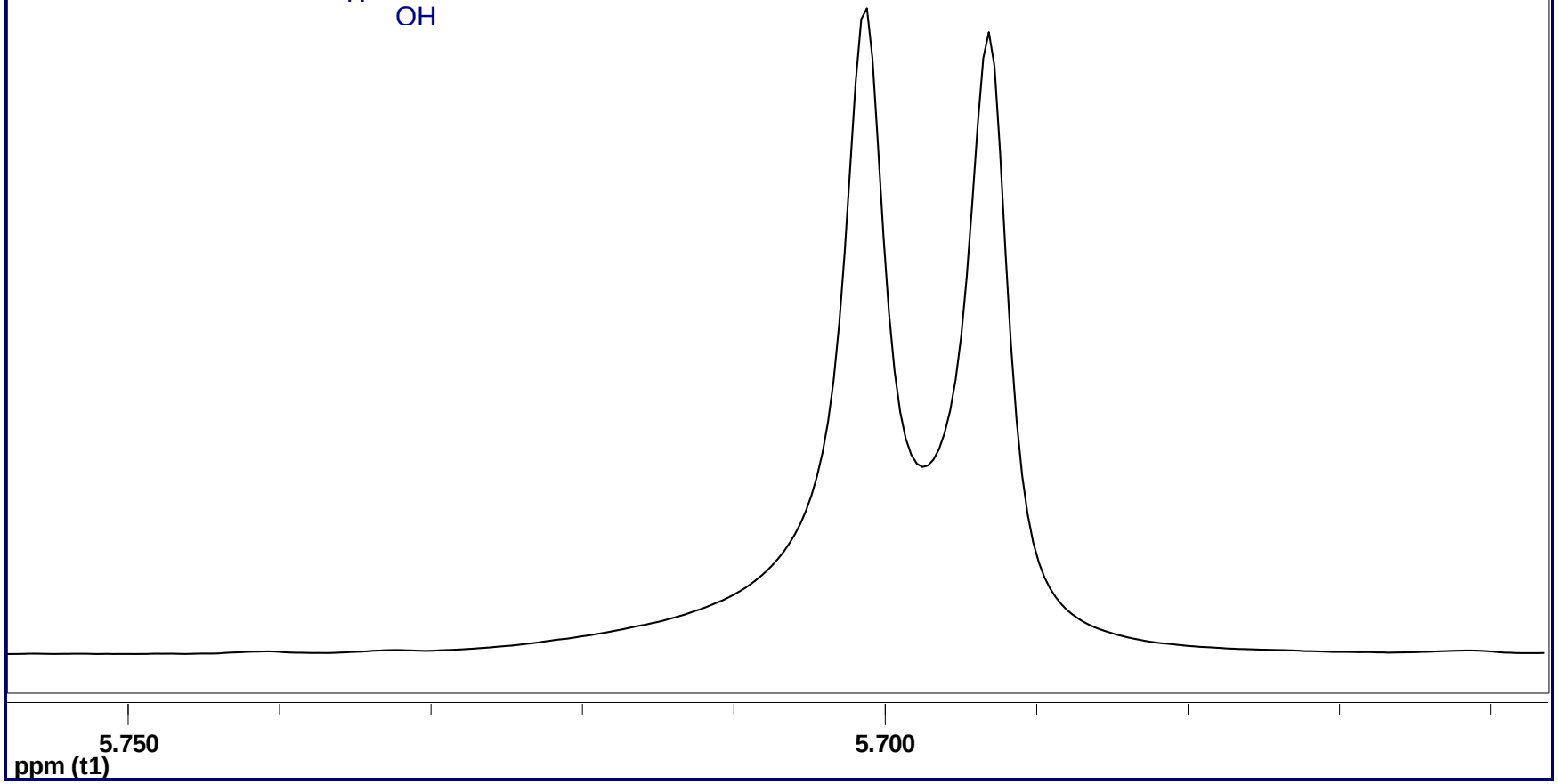

1H NMR Spectrum of compound 26a
8.54
7.51
7.50
7.48
7.42
7.41
7.40
7.38
7.37
7.14
7.12
6.77
6.76
5.70
5.69
4.96
4.95
4.94
4.84
4.83
3.69
O
NH
OMe
H
OH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

Expansion - 1H NMR Spectrum of compound 26a

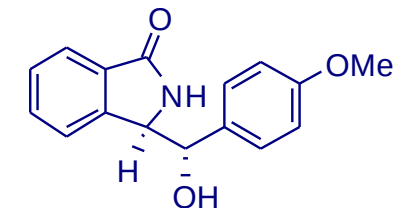


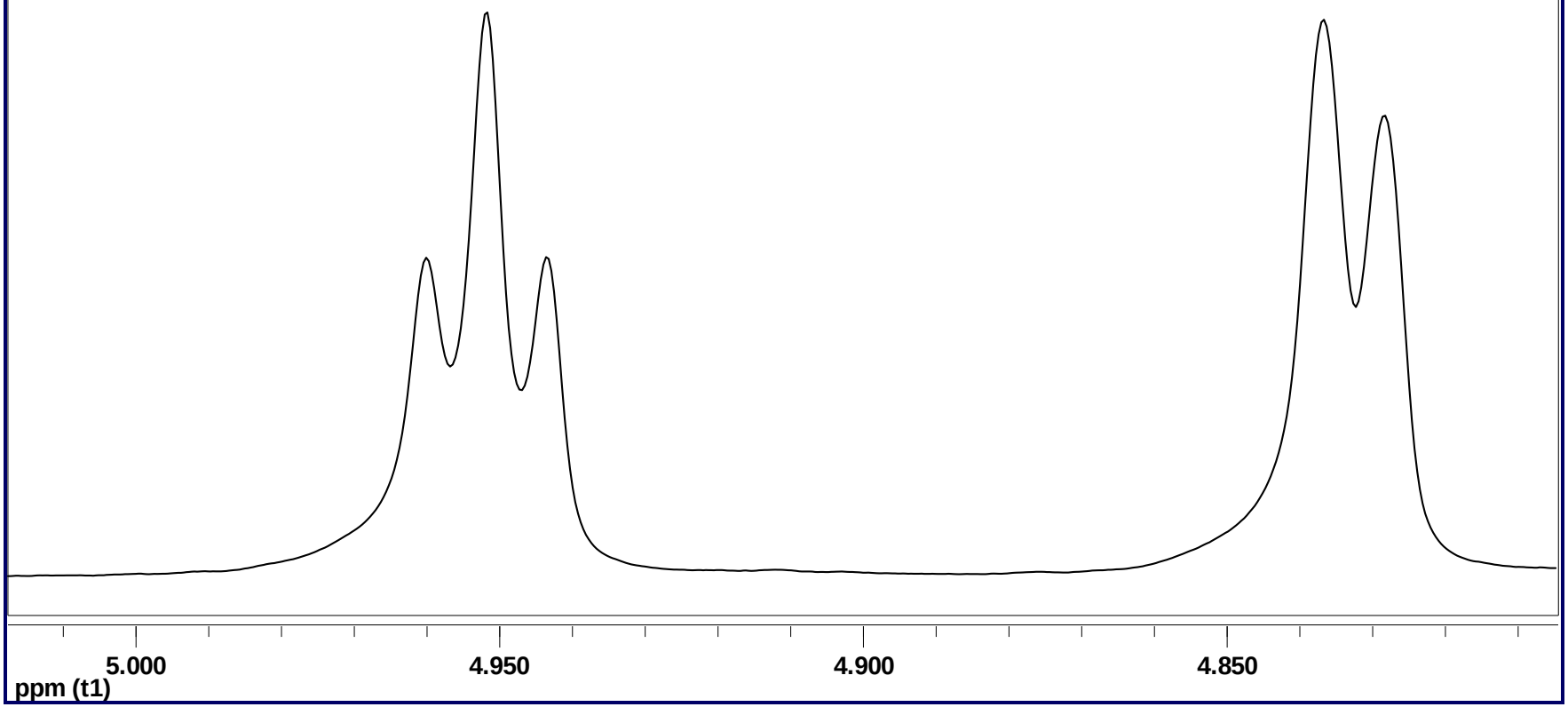
Expansion - 1H NMR Spectrum of compound 26a

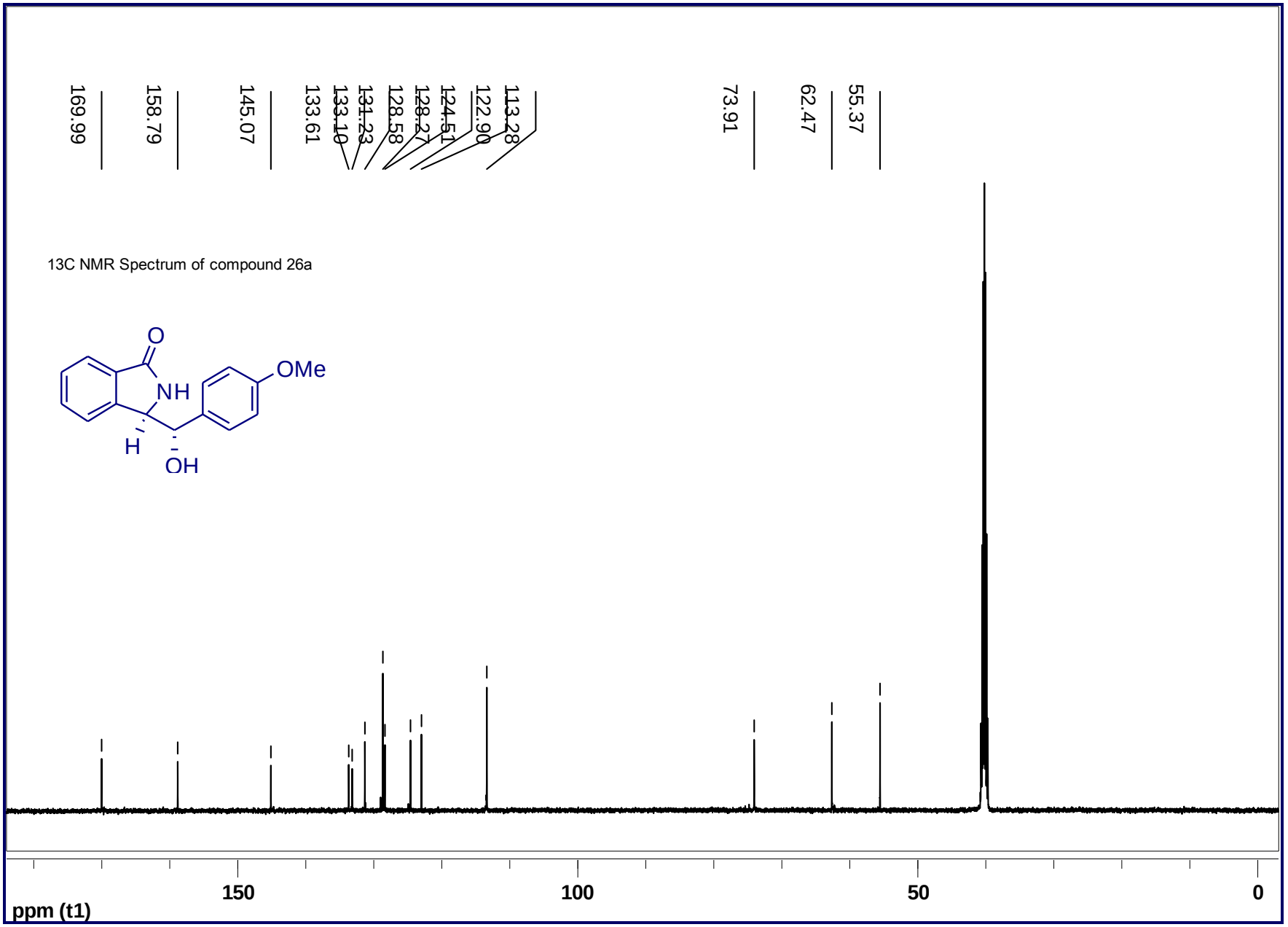


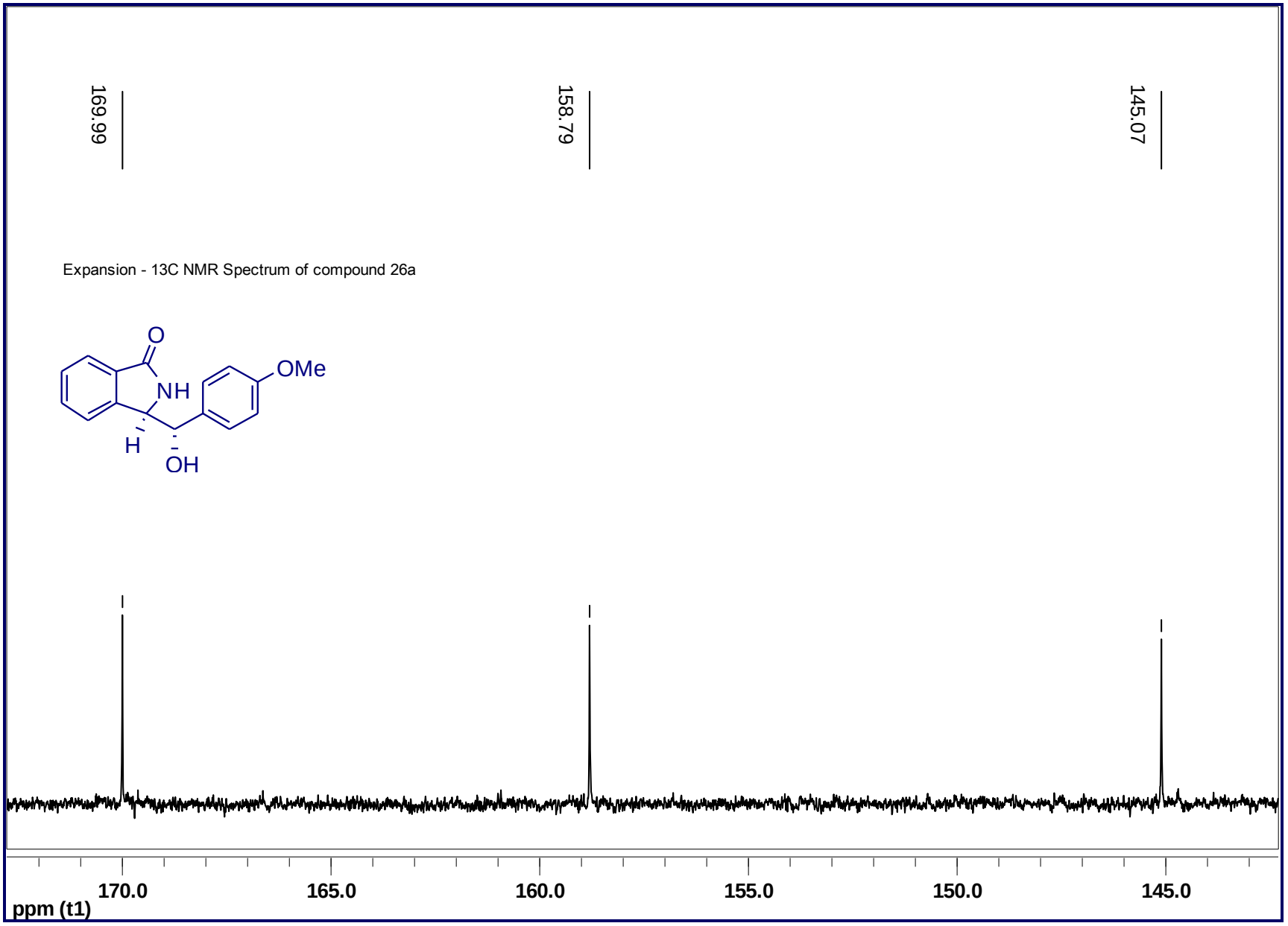


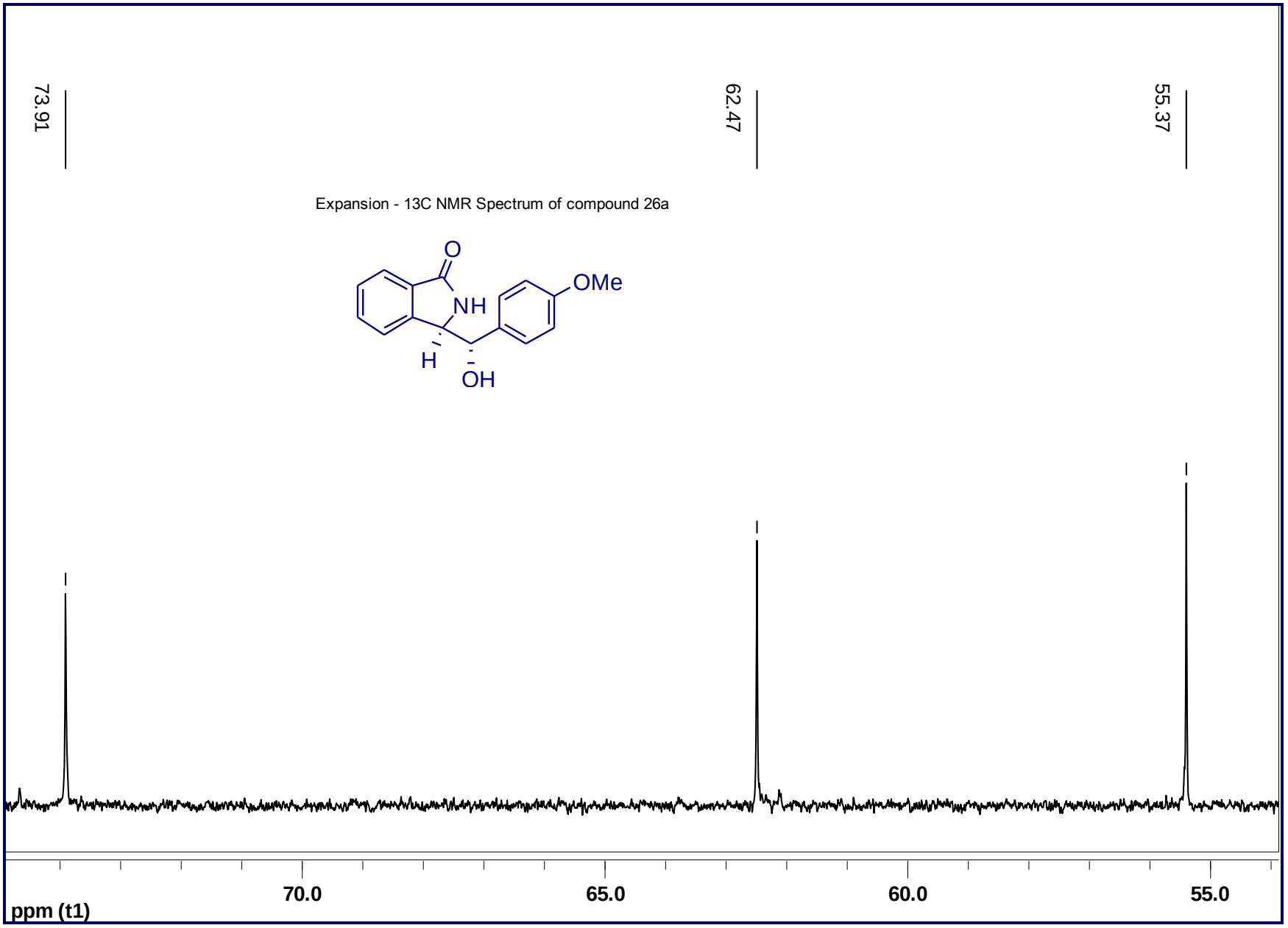
Expansion - 1H NMR Spectrum of compound 26a

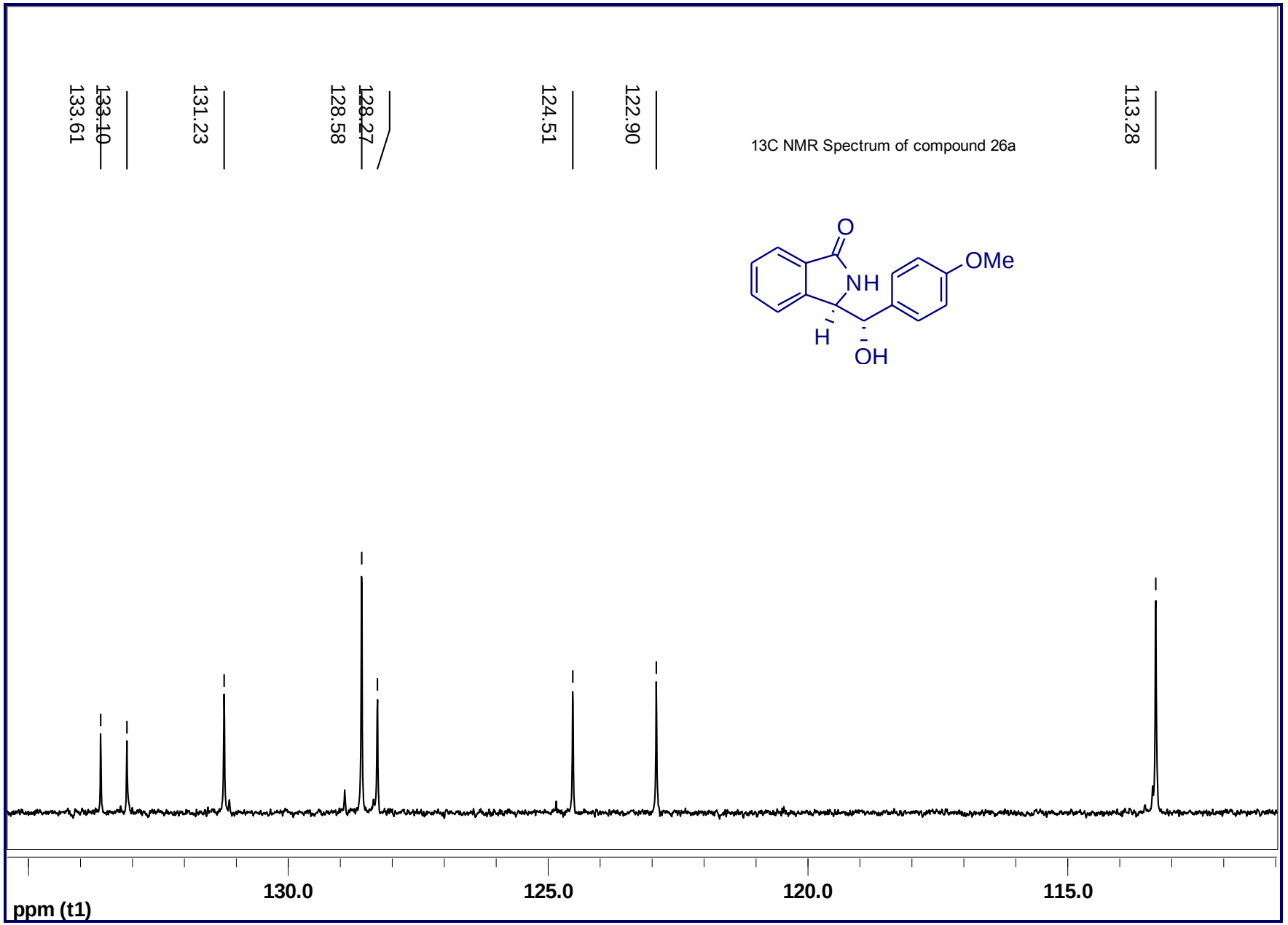


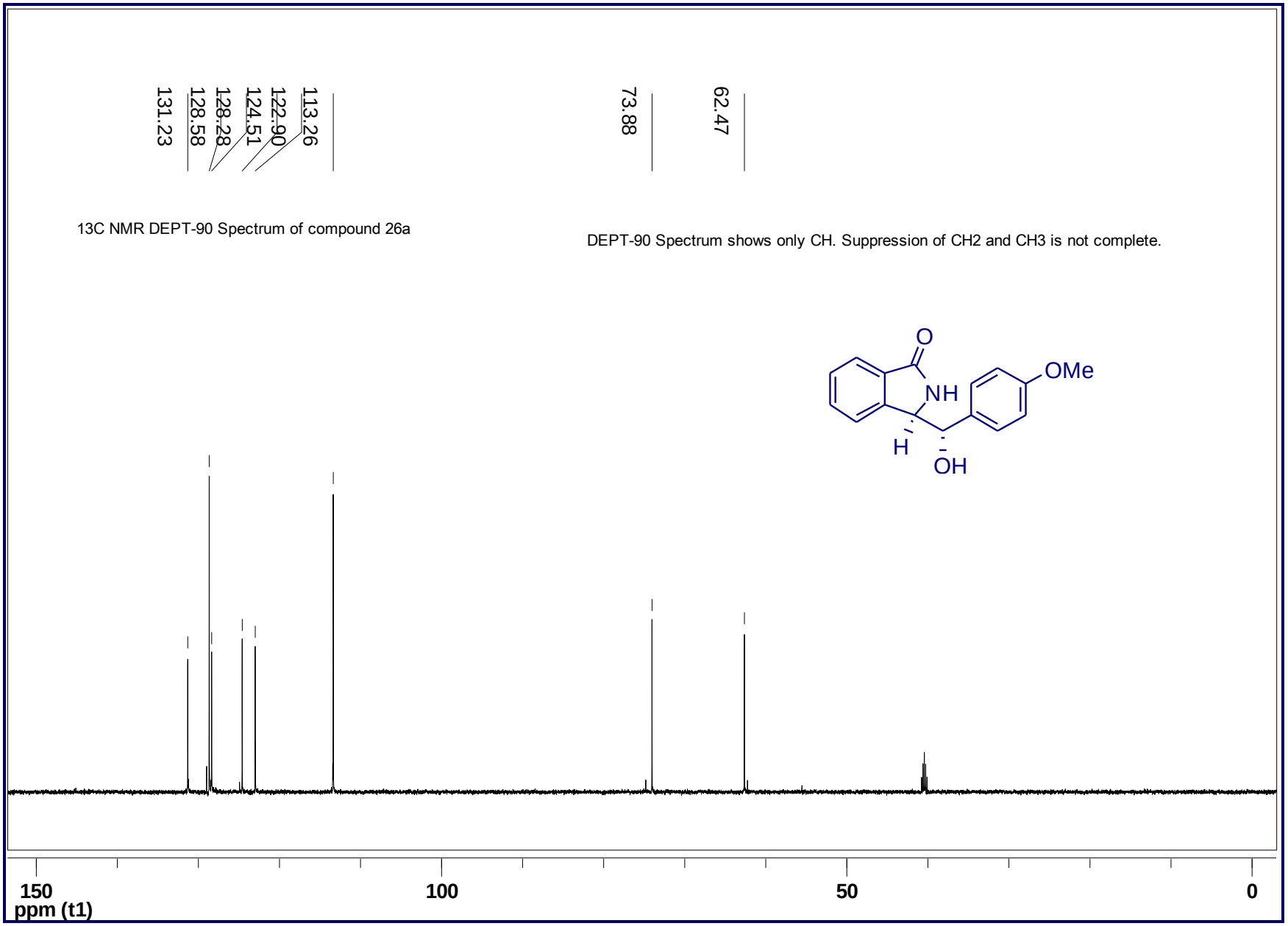


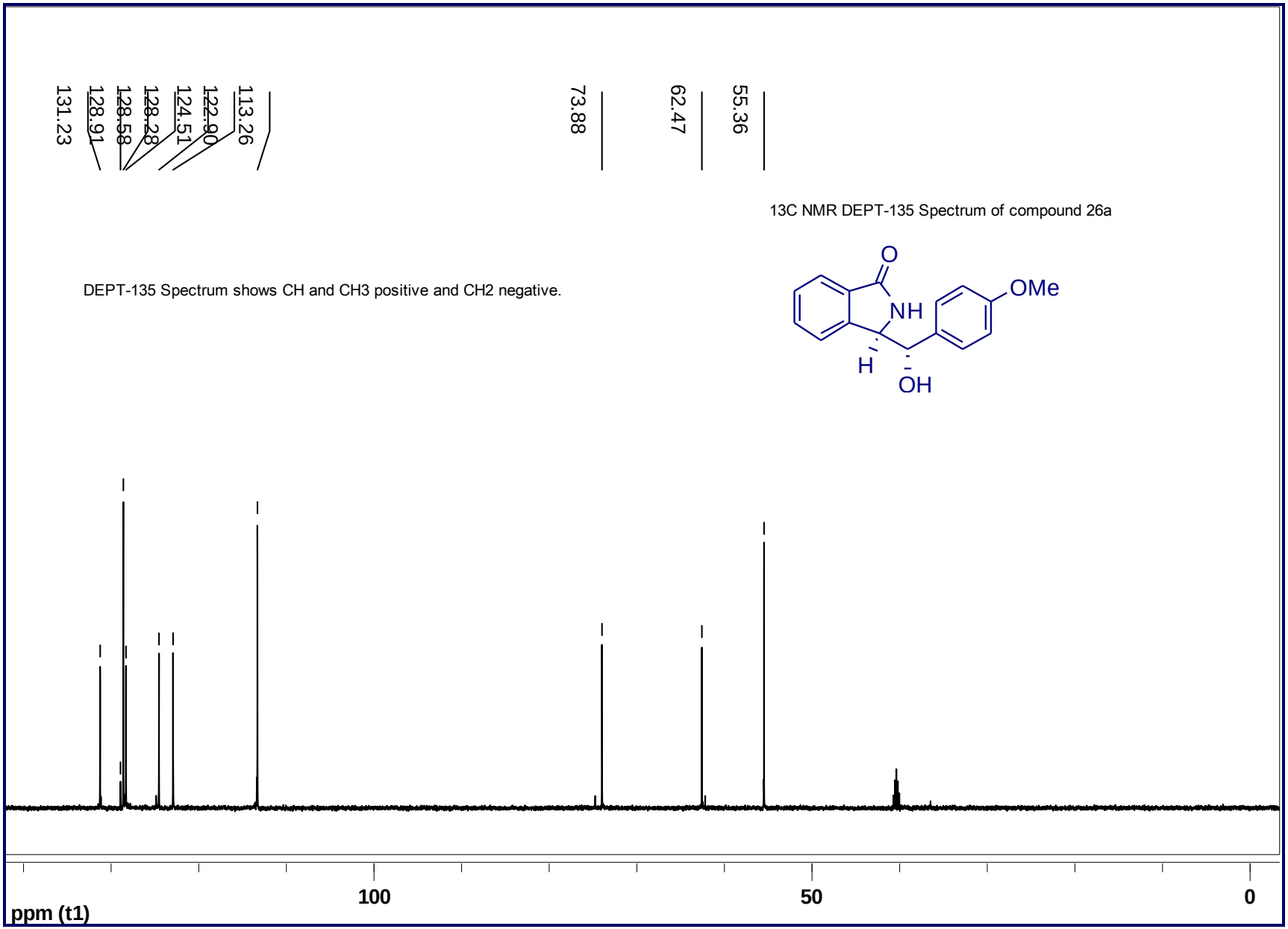

169.99
158.79
145.07
133.61
133.10
131.23
128.58
128.27
124.51
122.90
113.28
73.91
62.47
55.37
13C NMR Spectrum of compound 26a
O
NH
OMe
H
OH
150
100
50
0
ppm (t1)

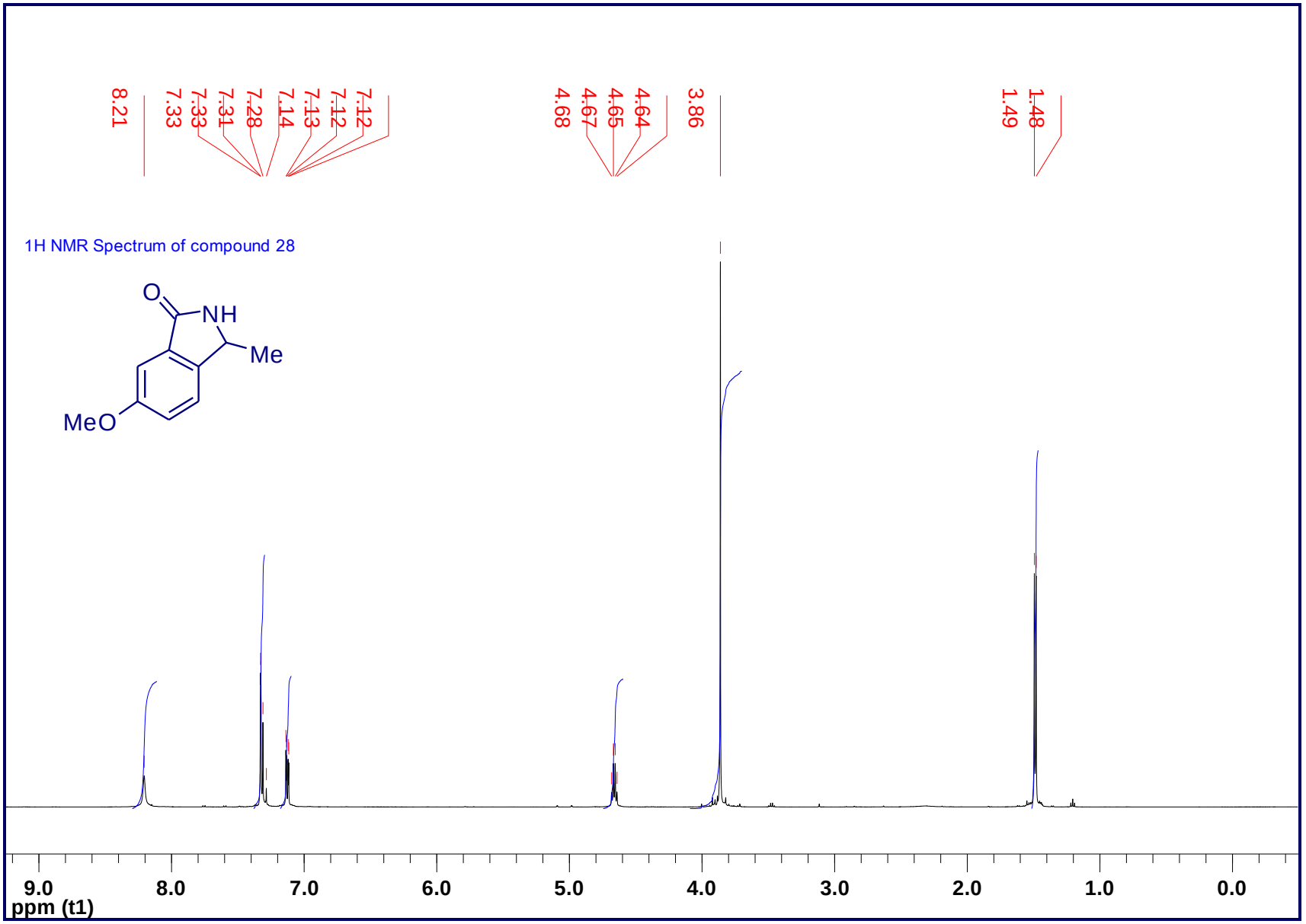

169.99
158.79
145.07
Expansion - 13C NMR Spectrum of compound 26a
O
NH
OMe
H
OH
170.0
165.0
160.0
155.0
150.0
145.0
ppm (t1)


73.91
62.47
55.37
Expansion - 13C NMR Spectrum of compound 26a
O
NH
OMe
H
OH
70.0
65.0
60.0
55.0
ppm (t1)


133.61
133.10
131.23
128.58
128.27
124.51
122.90
113.28
13C NMR Spectrum of compound 26a
O
NH
OMe
H
OH
130.0
125.0
120.0
115.0
ppm (t1)

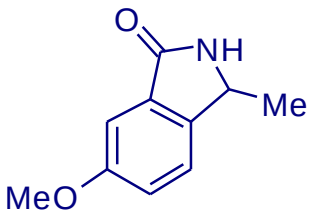

131.23
128.58
128.28
124.51
122.90
113.26
73.88
62.47
13C NMR DEPT-90 Spectrum of compound 26a
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete.
O
NH
OMe
H
OH
150
ppm (t1)
100
50
0

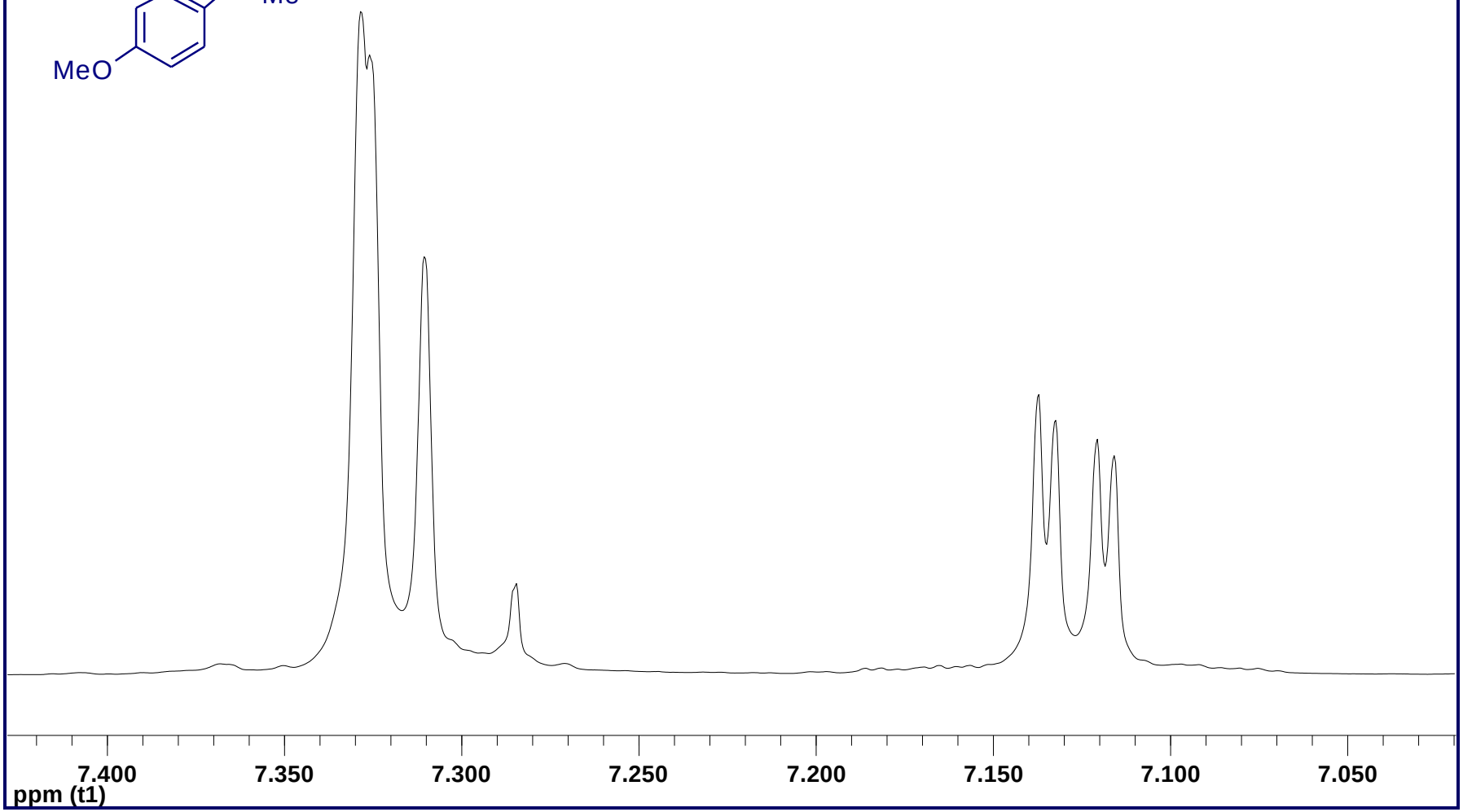

131.23
128.91
128.58
128.28
124.51
122.90
113.26
73.88
62.47
55.36
13C NMR DEPT-135 Spectrum of compound 26a
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
O
NH
OMe
H
OH
100
50
0
ppm (t1)

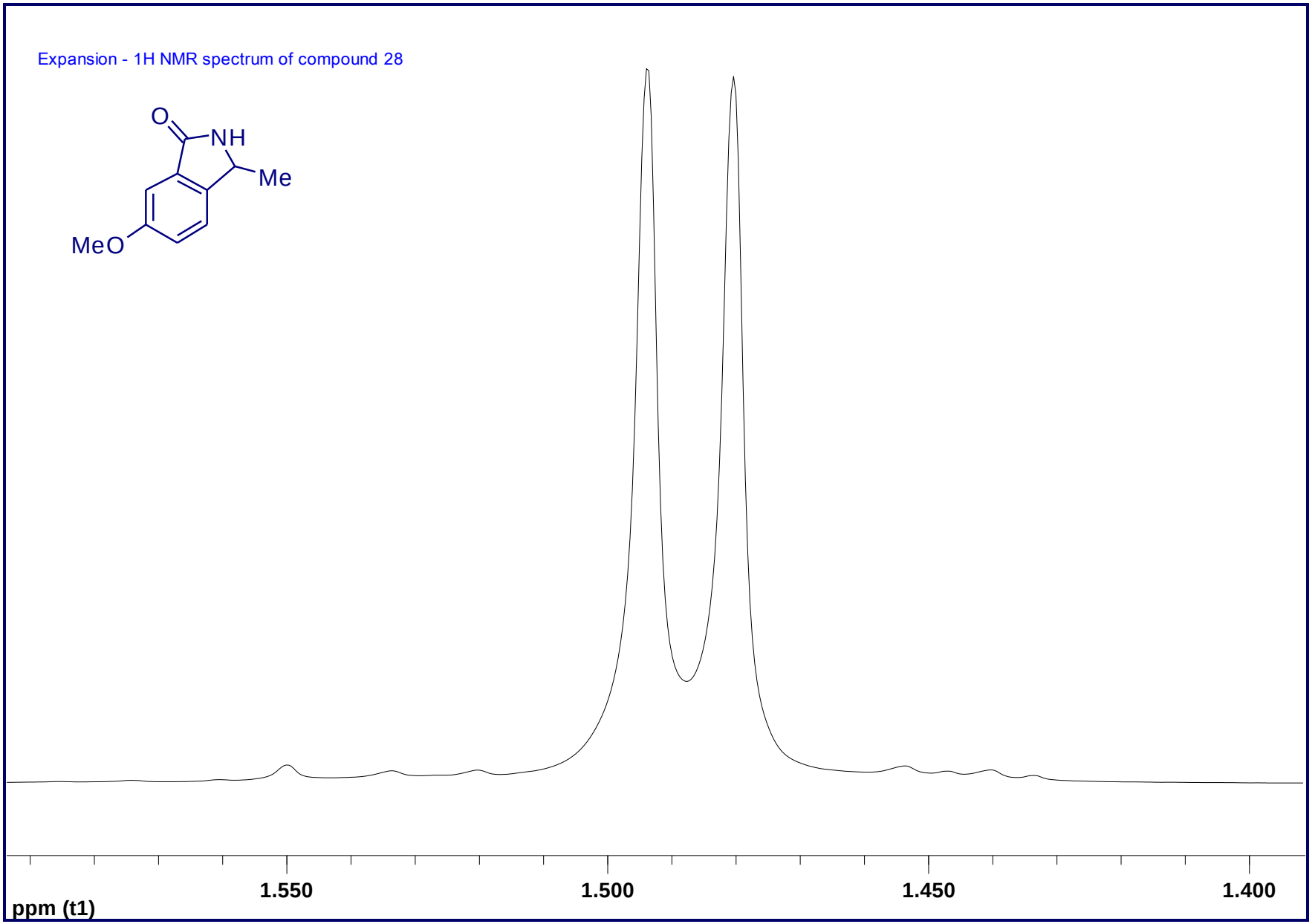

8.21
7.33
7.33
7.31
7.28
7.14
7.13
7.12
7.12
4.68
4.67
4.65
4.64
3.86
1.49
1.48
1H NMR Spectrum of compound 28
O
NH
Me
MeO
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

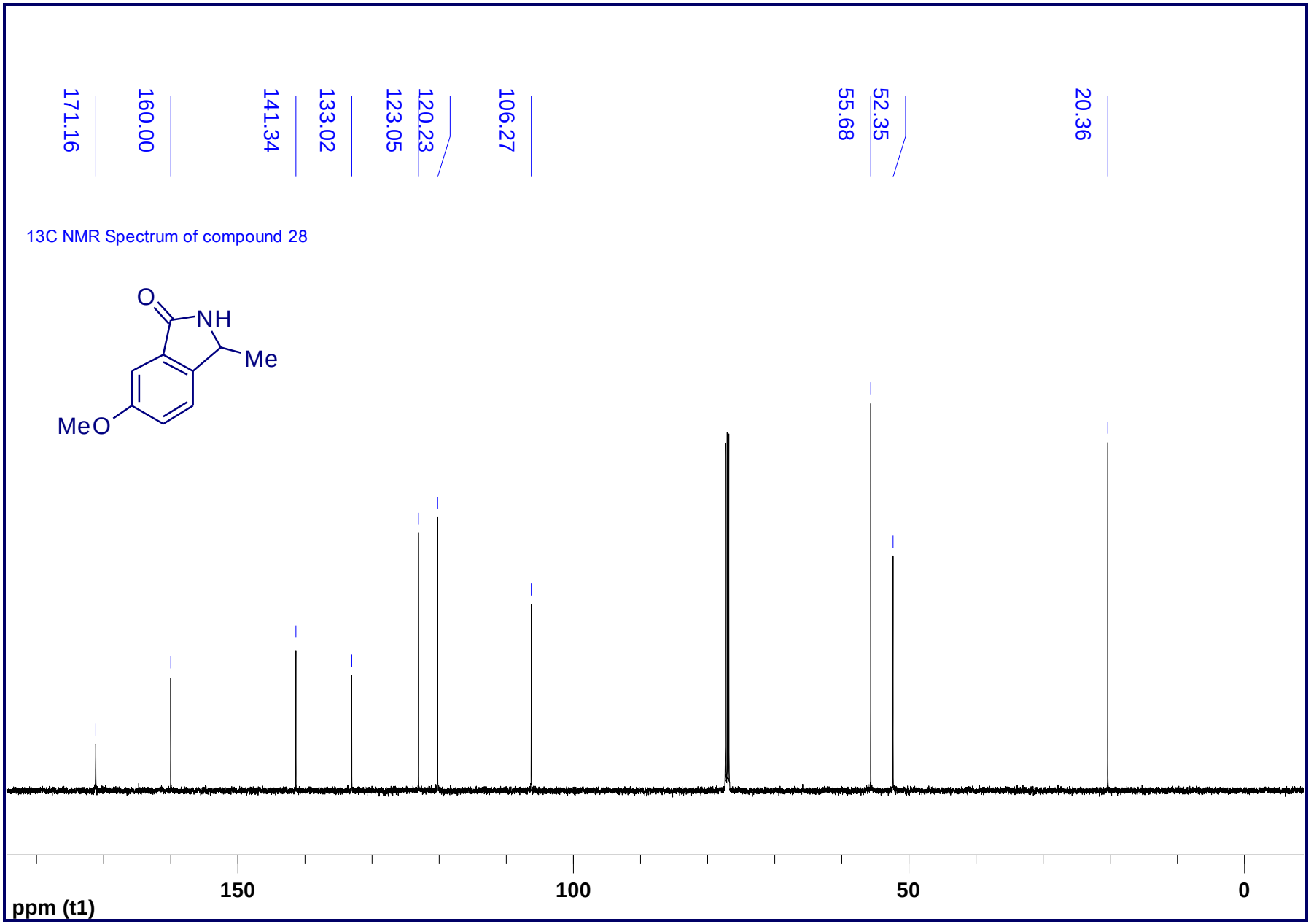
Expansion - 1H NMR spectrum of compound 28

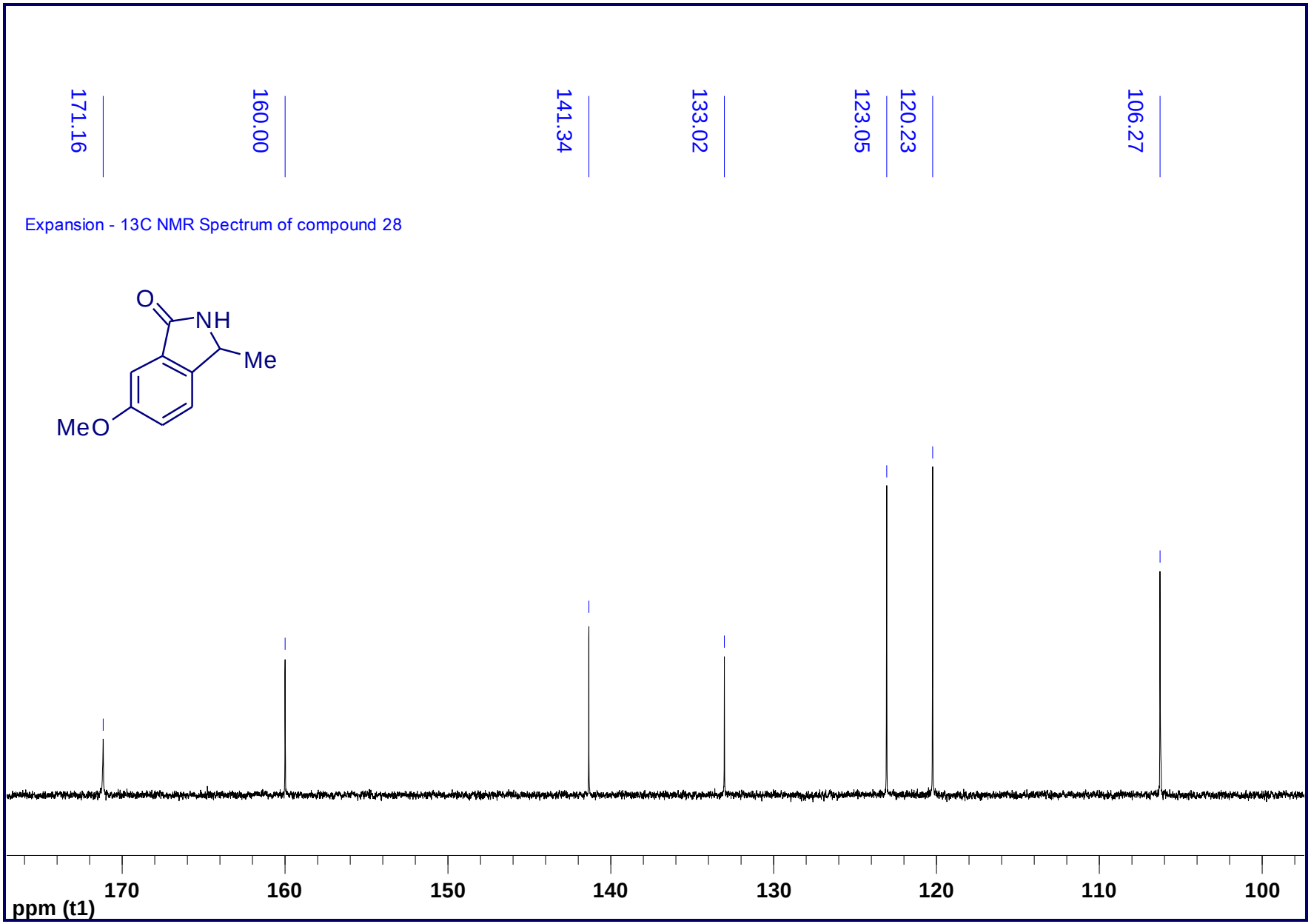
Expansion - 1H NMR spectrum of compound 28

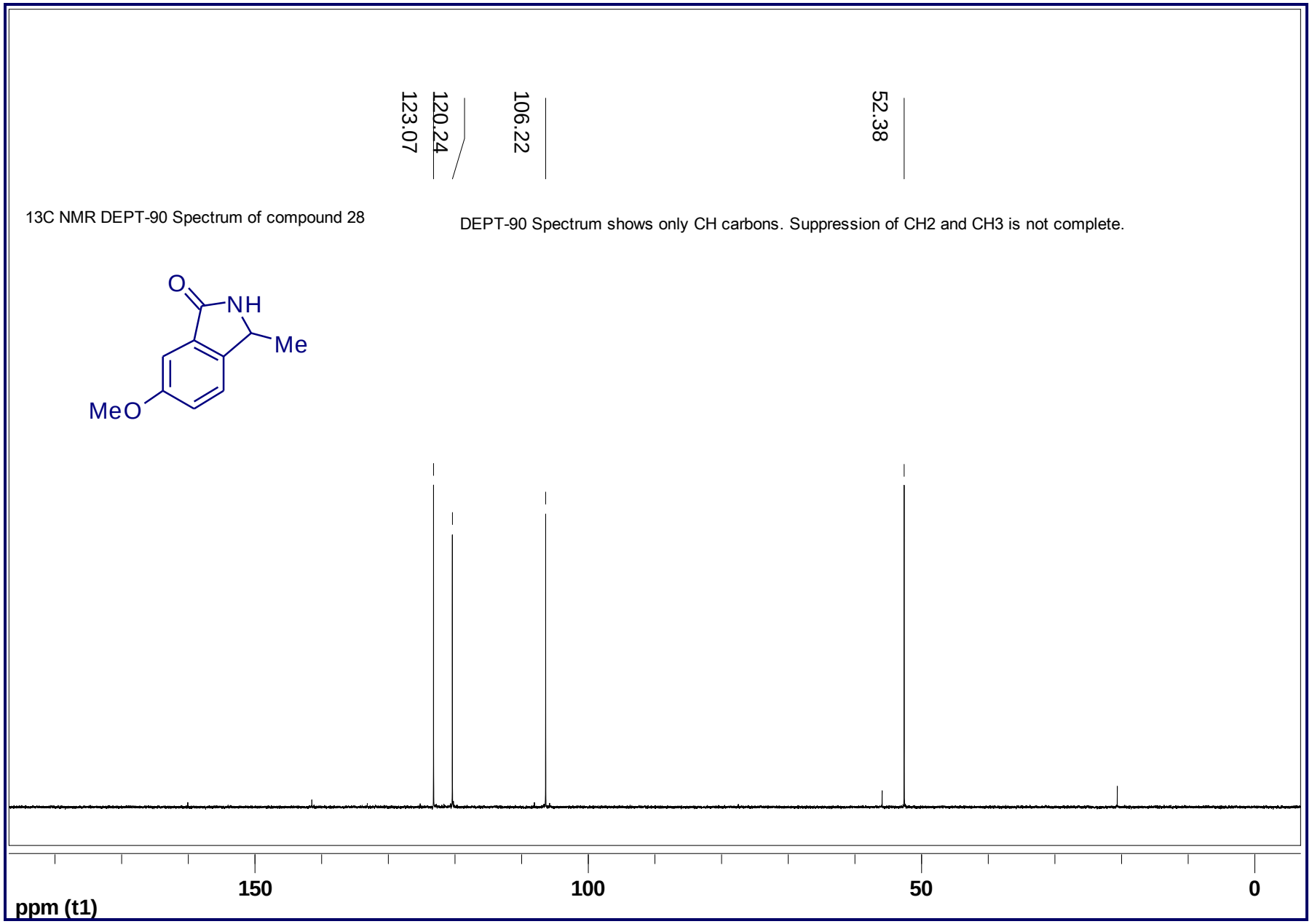


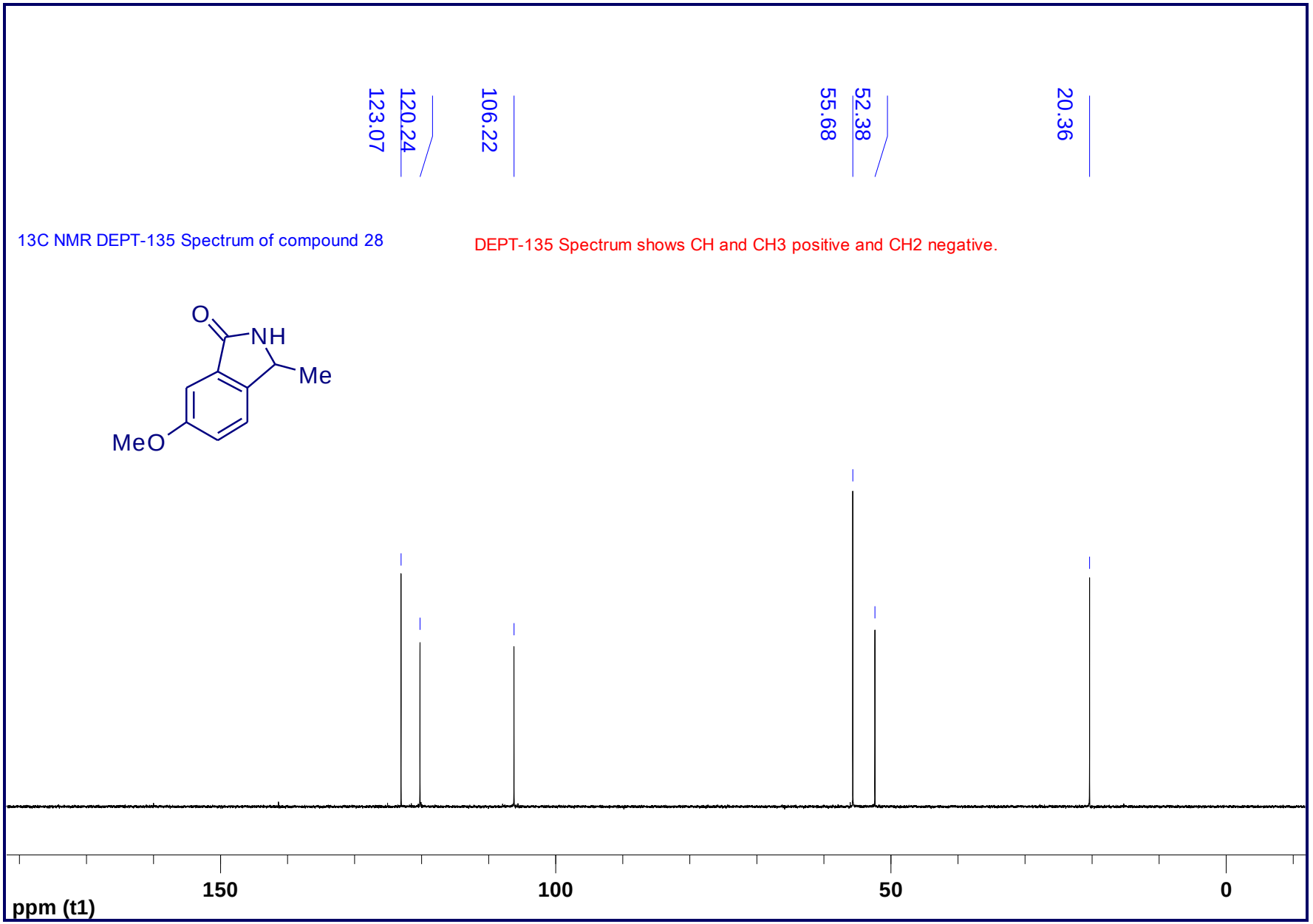


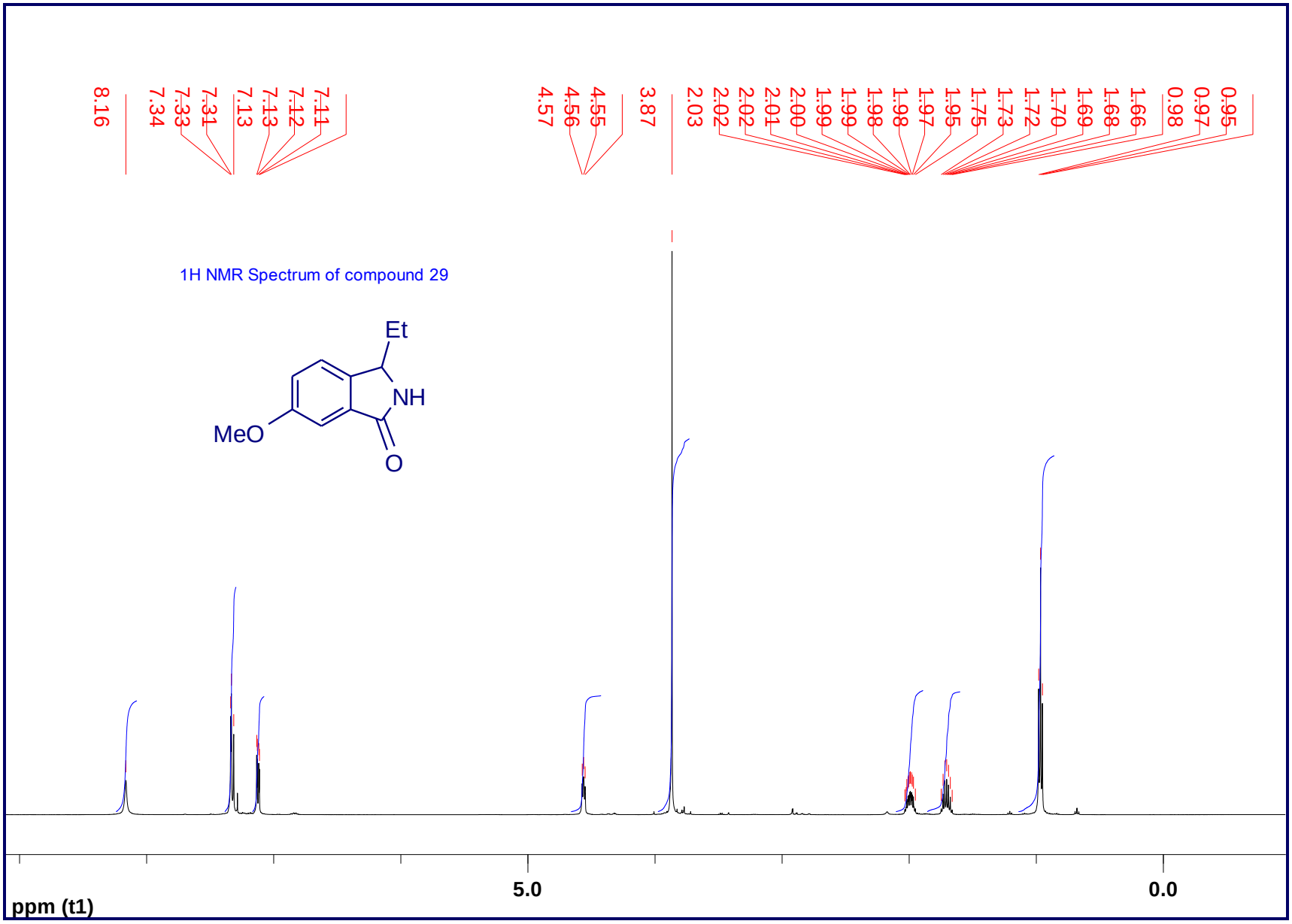

Expansion - 1H NMR spectrum of compound 28
O
NH
Me
MeO
1.550
1.500
1.450
1.400
ppm (t1)

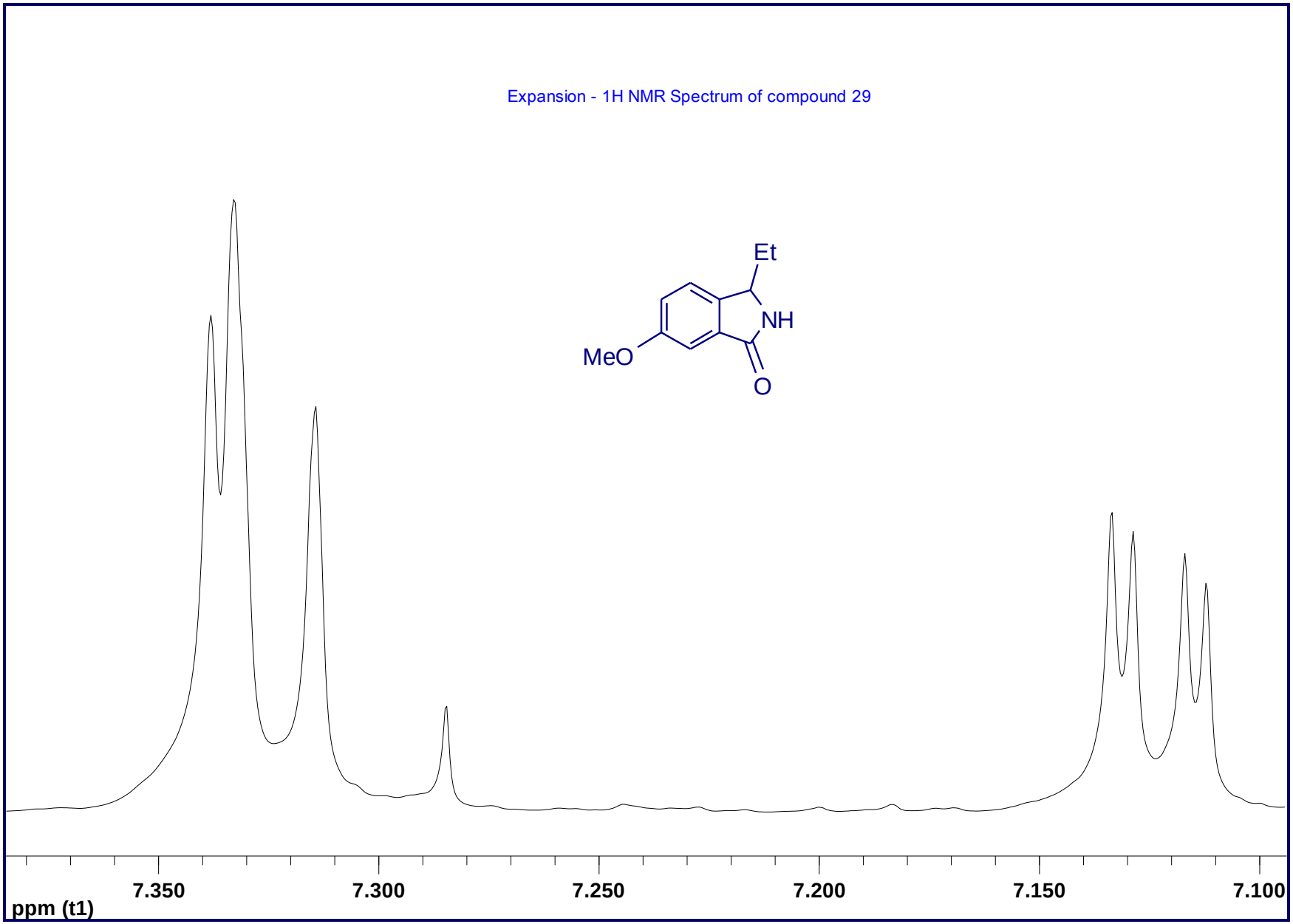

171.16
160.00
141.34
133.02
123.05
120.23
106.27
55.68
52.35
20.36
13C NMR Spectrum of compound 28
O
NH
Me
MeO
150
100
50
0
ppm (t1)

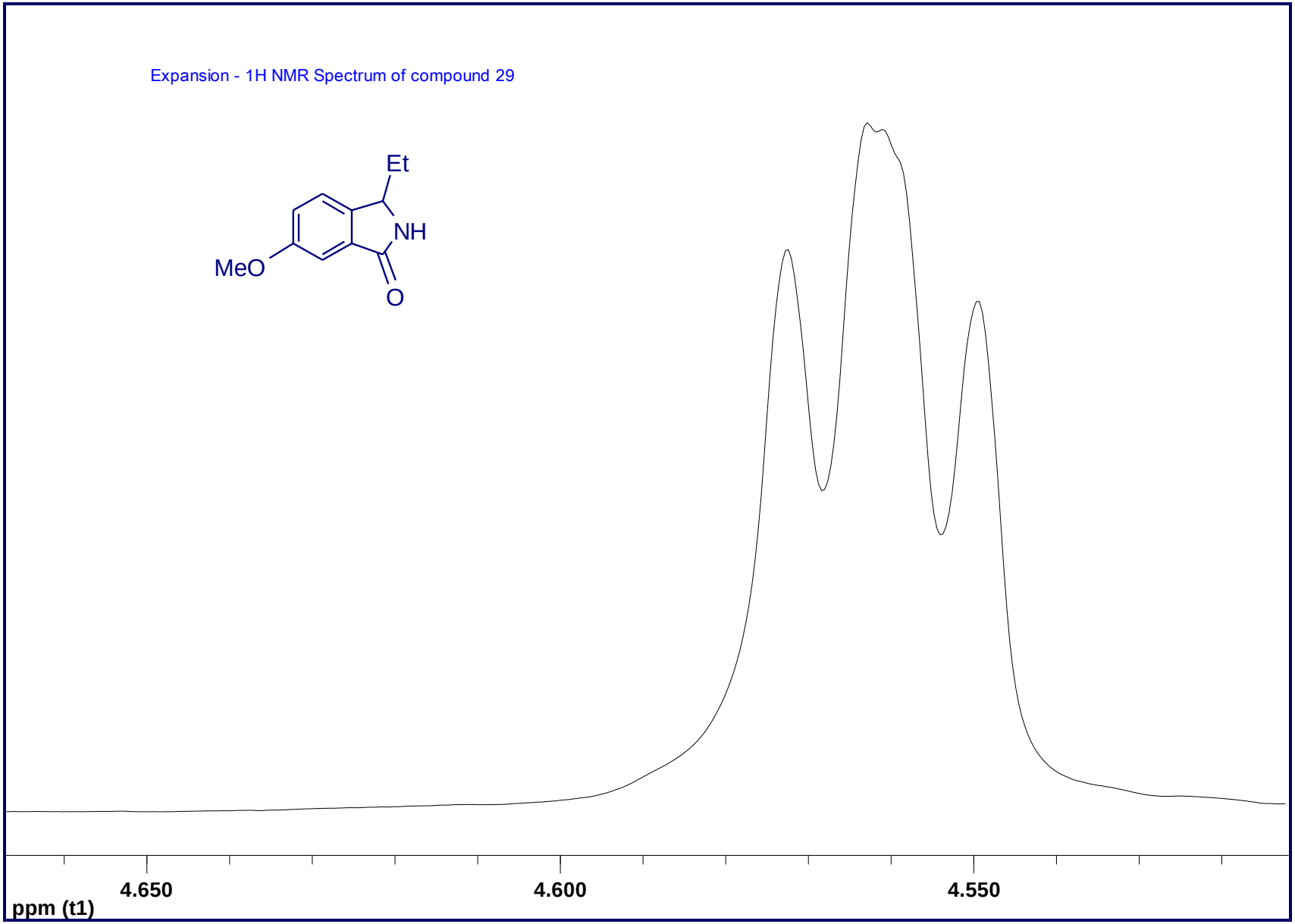

171.16
160.00
141.34
133.02
123.05
120.23
106.27
Expansion - 13C NMR Spectrum of compound 28
O
NH
Me
MeO
170
160
150
140
130
120
110
100
ppm (t1)

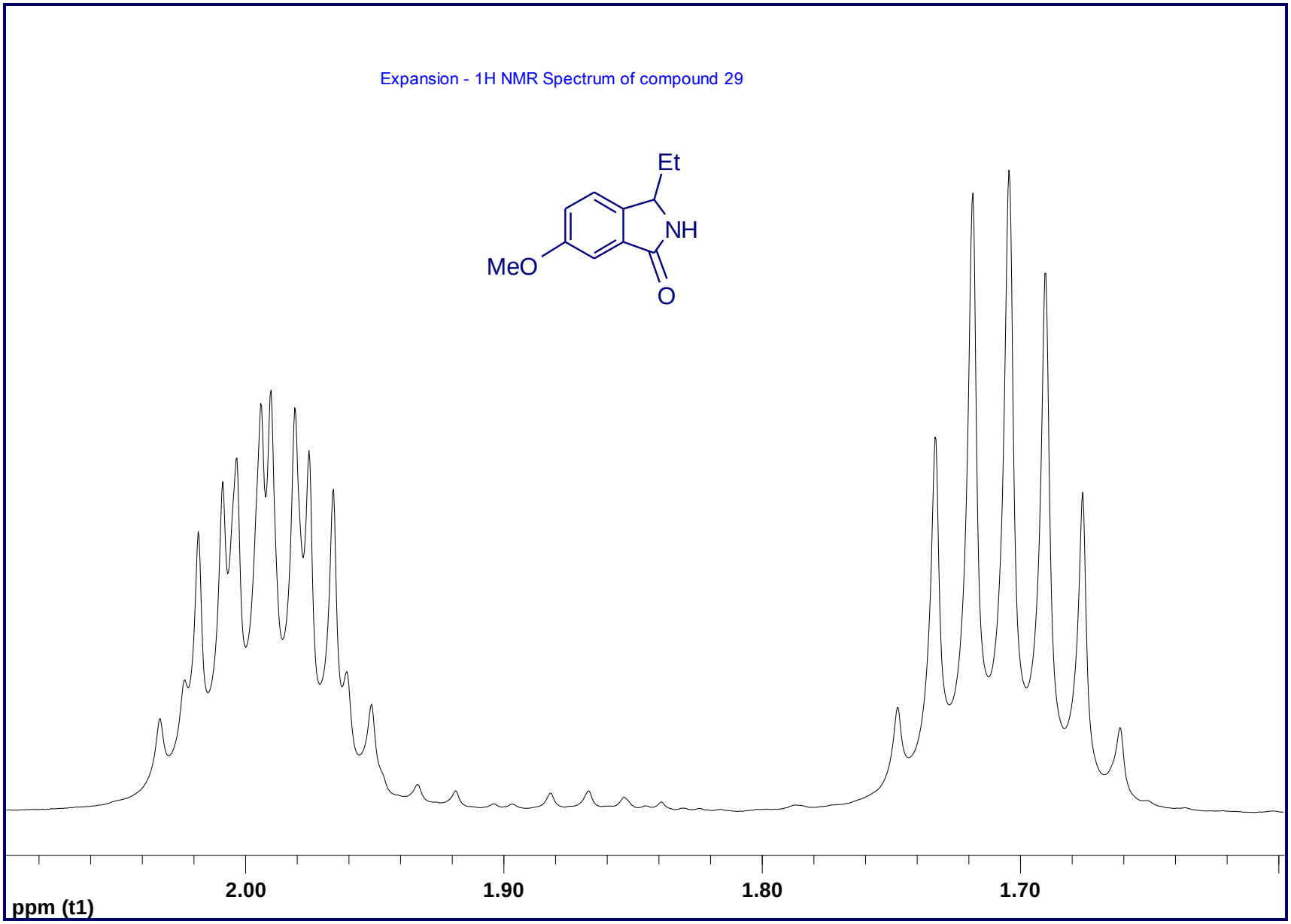

123.07
120.24
106.22
52.38
13C NMR DEPT-90 Spectrum of compound 28
DEPT-90 Spectrum shows only CH carbons. Suppression of CH2 and CH3 is not complete.
O
NH
Me
MeO
150
100
50
0
ppm (t1)

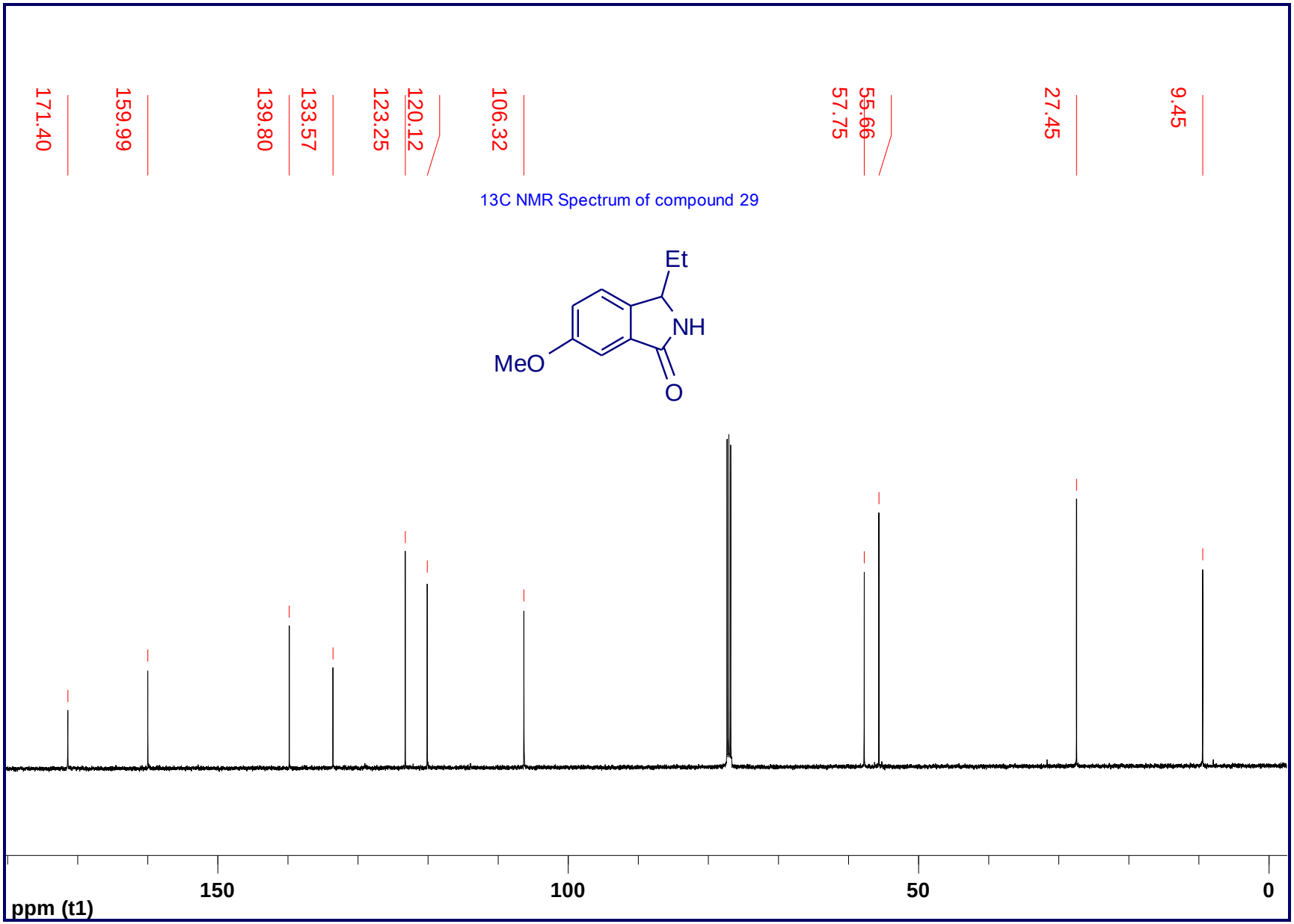

123.07
120.24
106.22
55.68
52.38
20.36
13C NMR DEPT-135 Spectrum of compound 28
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
O
NH
Me
MeO
150
100
50
0
ppm (t1)

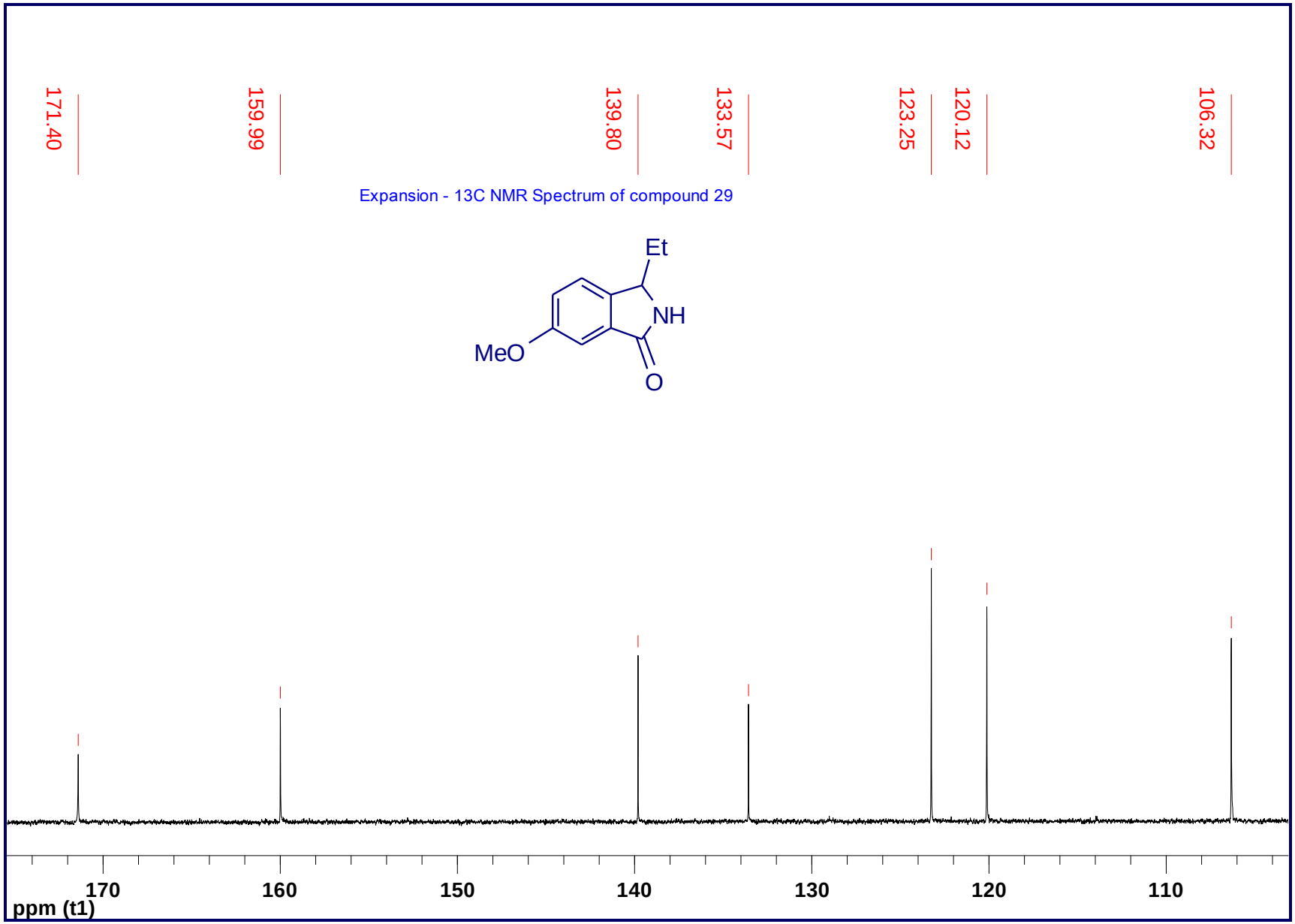

8.16
7.34
7.33
7.31
7.13
7.13
7.12
7.11
4.57
4.56
4.55
3.87
2.03
2.02
2.02
2.01
2.00
1.99
1.99
1.98
1.98
1.97
1.95
1.75
1.73
1.72
1.70
1.69
1.68
1.66
0.98
0.97
0.95
1H NMR Spectrum of compound 29
Et
NH
MeO
O
5.0
0.0
ppm (t1)

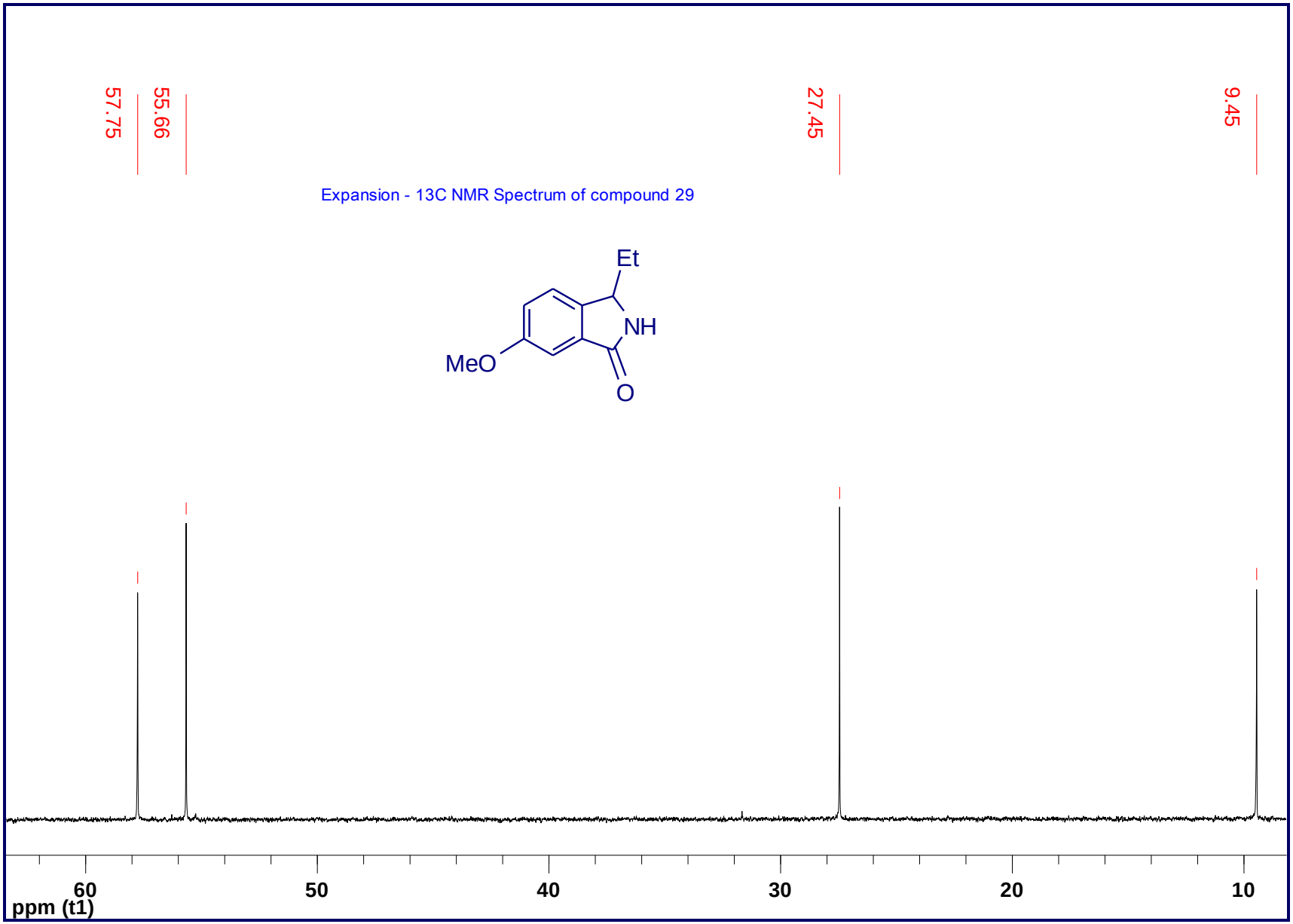

Expansion - 1H NMR Spectrum of compound 29
Et
NH
MeO
O
7.350
7.300
7.250
7.200
7.150
7.100
ppm (t1)

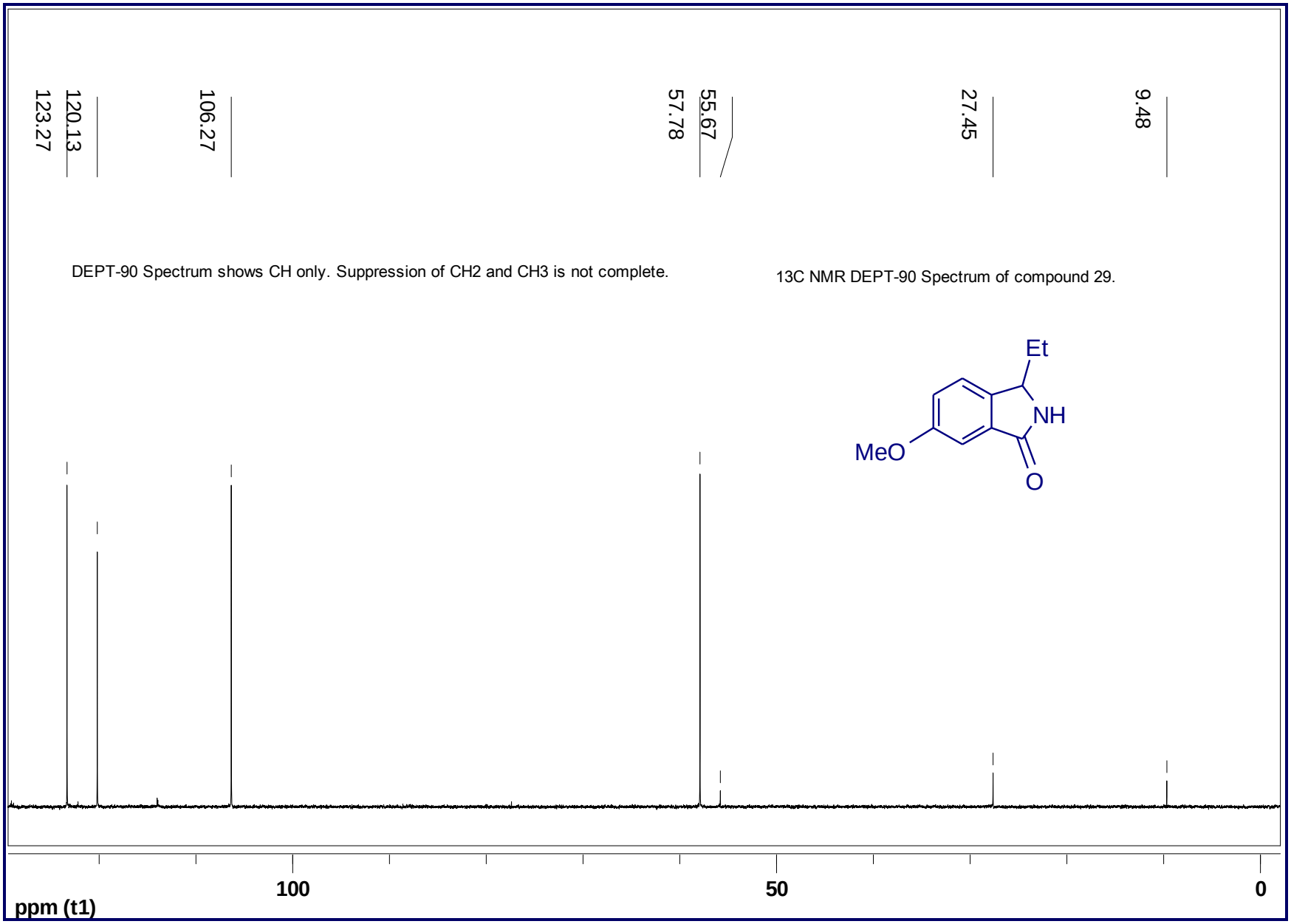

Expansion - 1H NMR Spectrum of compound 29
Et
NH
MeO
O
4.650
4.600
4.550
ppm (t1)

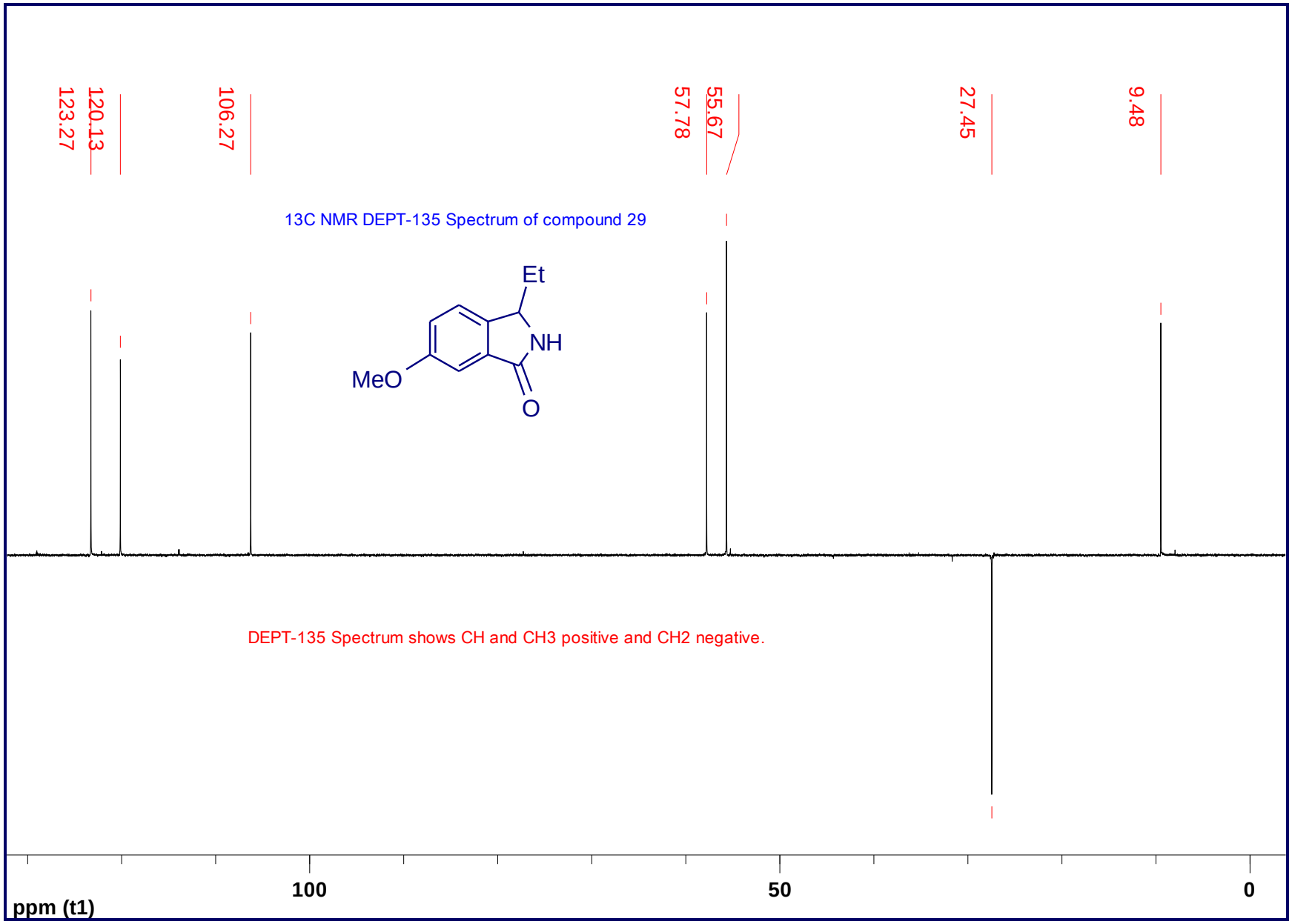

Expansion - 1H NMR Spectrum of compound 29
Et
NH
MeO
O
2.00
1.90
1.80
1.70
ppm (t1)

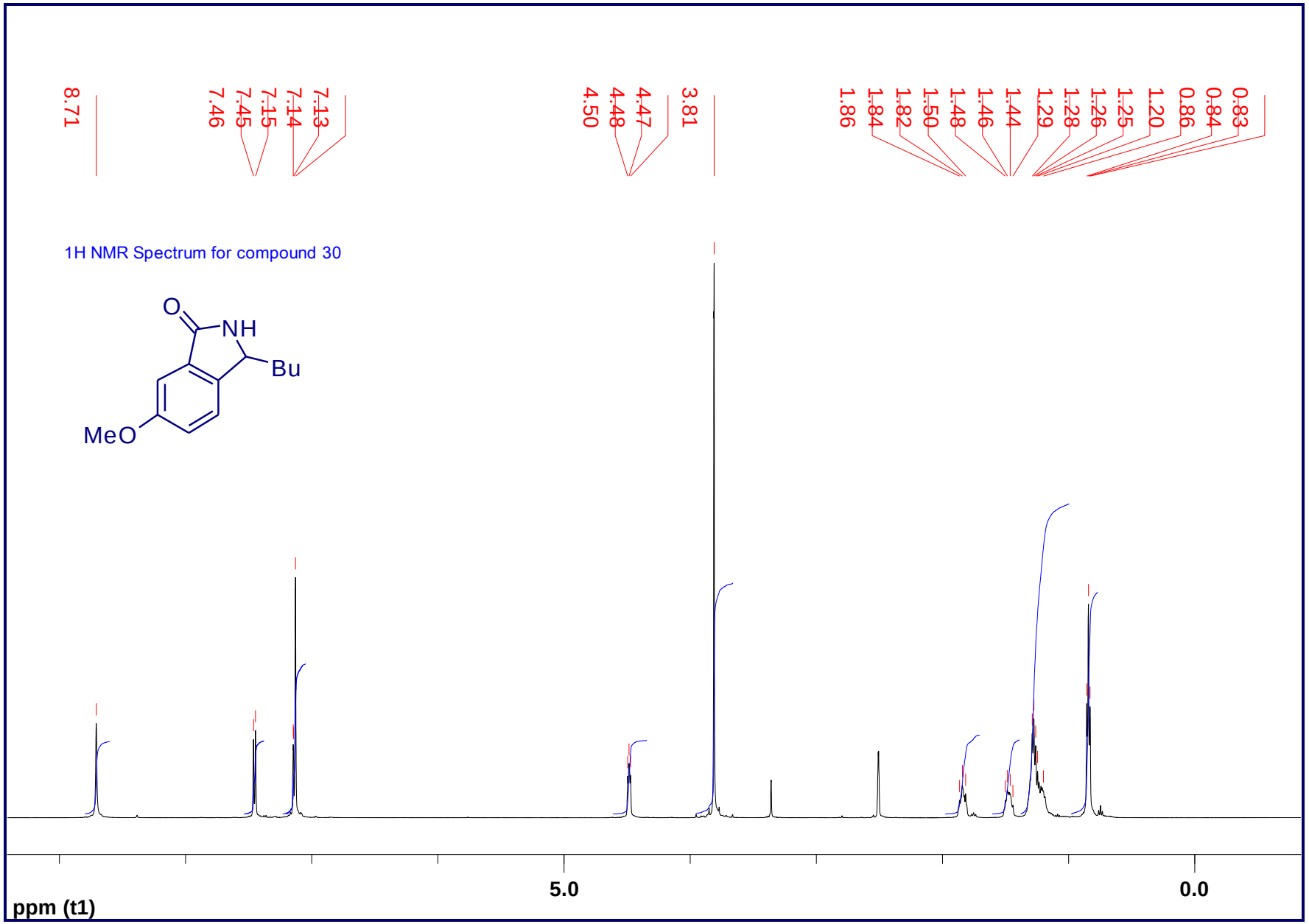

171.40
159.99
139.80
133.57
123.25
120.12
106.32
57.75
55.66
27.45
9.45
13C NMR Spectrum of compound 29
Et
NH
MeO
O
150
100
50
0
ppm (t1)


171.40
159.99
139.80
133.57
123.25
120.12
106.32
Expansion - 13C NMR Spectrum of compound 29
Et
NH
MeO
O
170
160
150
140
130
120
110
ppm (t1)

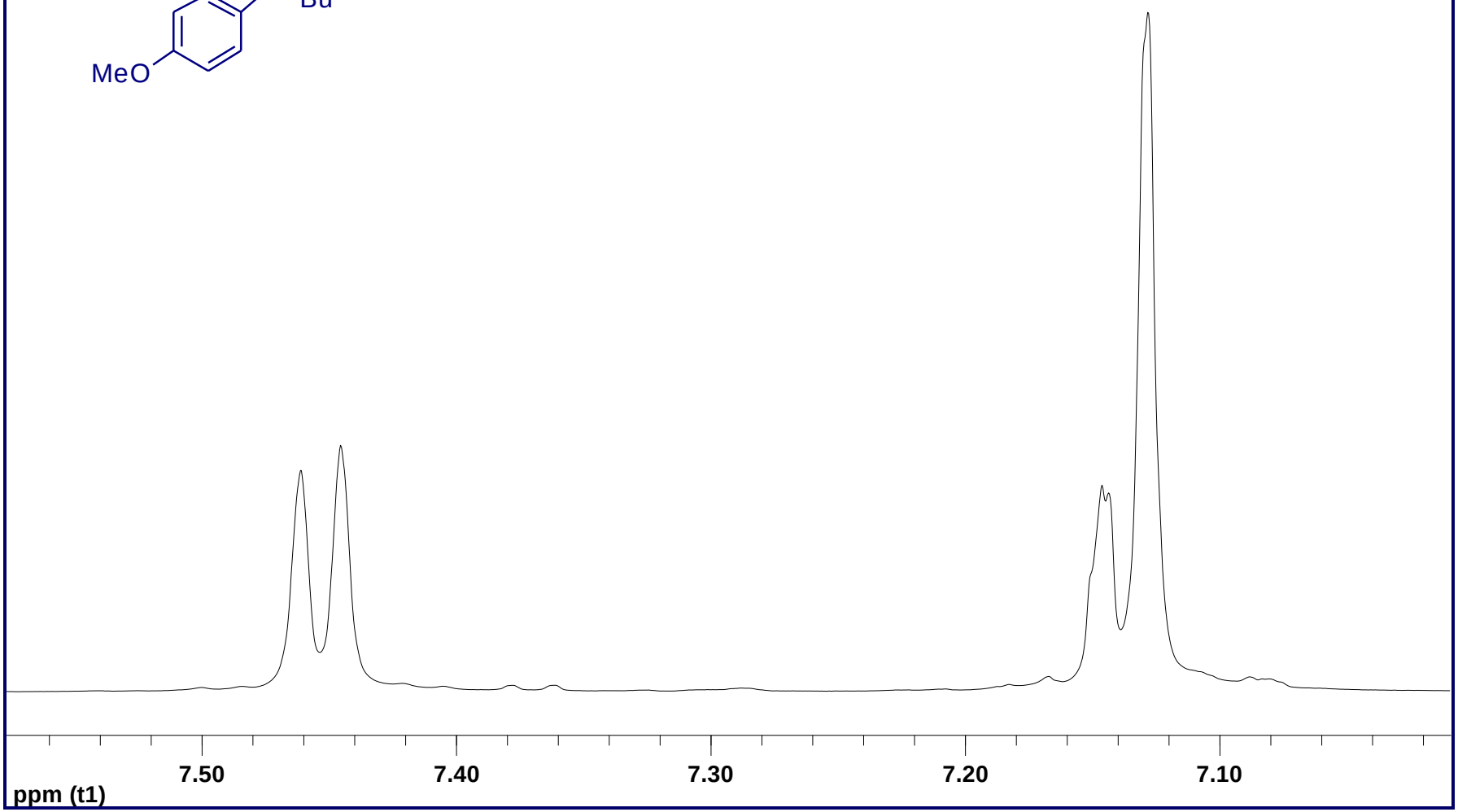

57.75
55.66
27.45
9.45
Expansion - 13C NMR Spectrum of compound 29
Et
NH
MeO
O
60
50
40
30
20
10
ppm (t1)


123.27
120.13
106.27
57.78
55.67
27.45
9.48
DEPT-90 Spectrum shows CH only. Suppression of CH2 and CH3 is not complete.
13C NMR DEPT-90 Spectrum of compound 29.
Et
NH
MeO
O
100
50
0
ppm (t1)

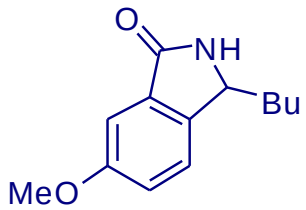

123.27
120.13
106.27
57.78
55.67
27.45
9.48
13C NMR DEPT-135 Spectrum of compound 29
Et
NH
MeO
O
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
100
50
0
ppm (t1)

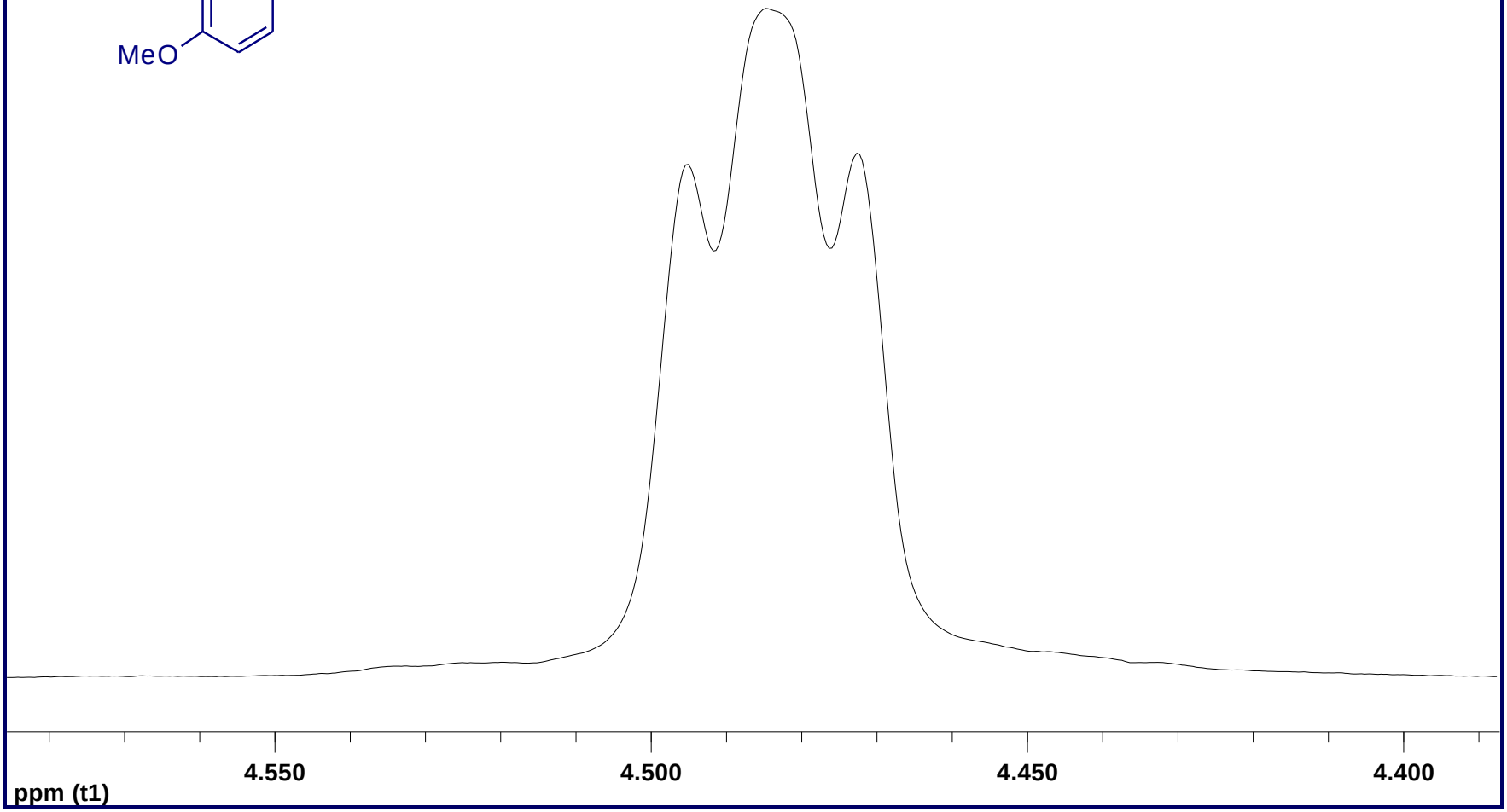

8.71
7.46
7.45
7.15
7.14
7.13
4.50
4.48
4.47
3.81
1.86
1.84
1.82
1.50
1.48
1.46
1.44
1.29
1.28
1.26
1.25
1.20
0.86
0.84
0.83
1H NMR Spectrum for compound 30
O
NH
Bu
MeO
5.0
0.0
ppm (t1)

Expansion - 1H NMR spectrum for compound 30

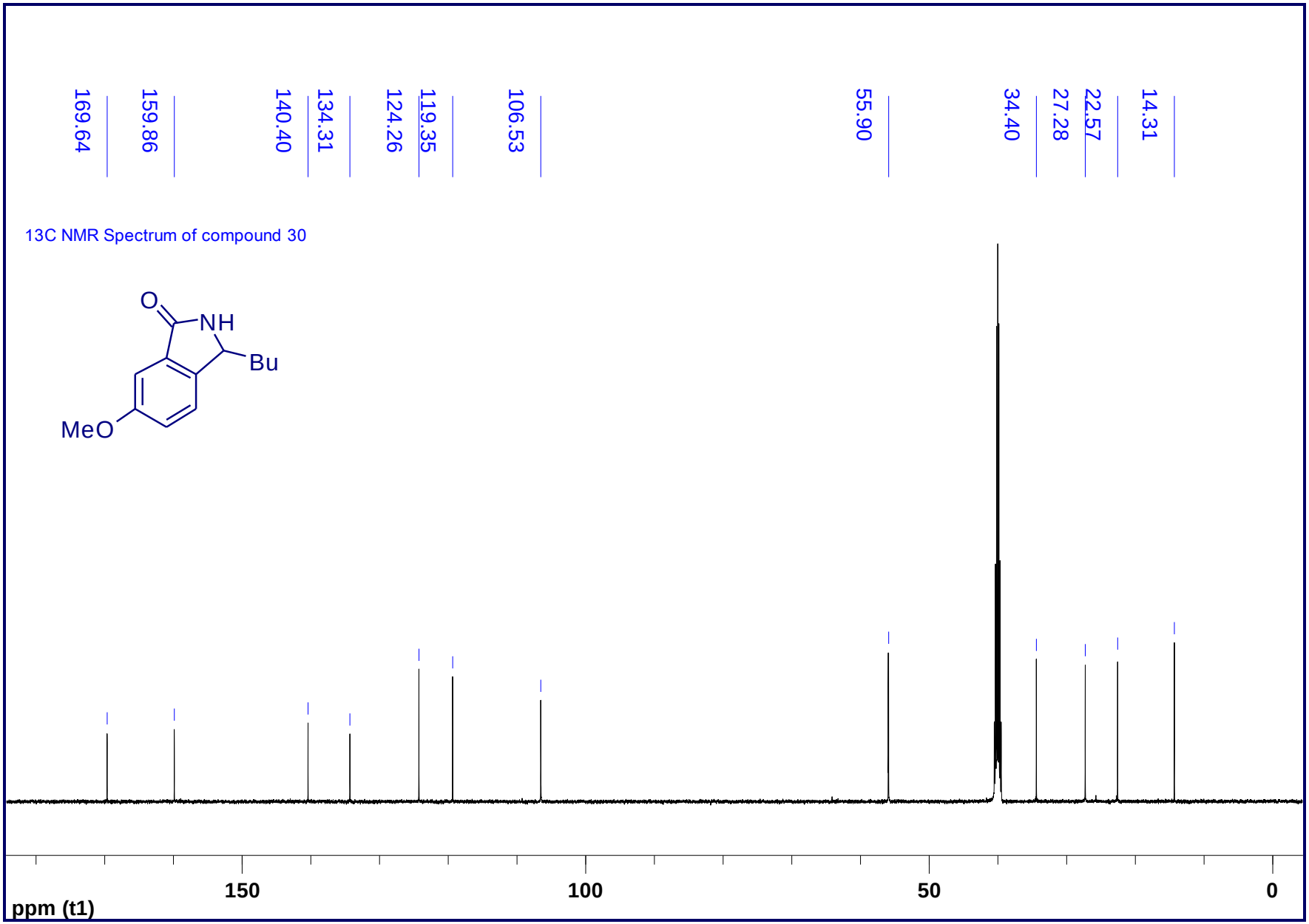


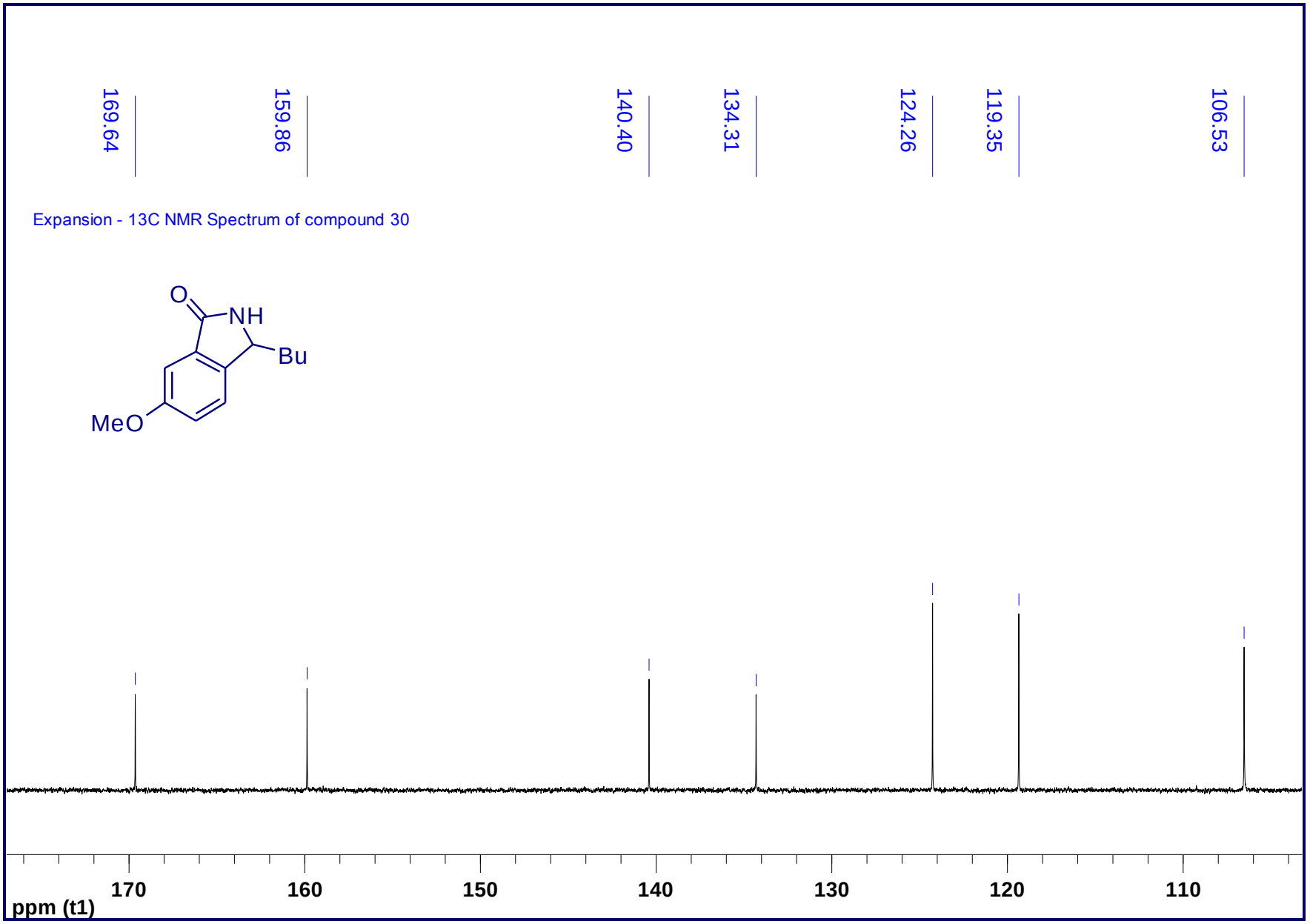
Expansion - 1H NMR spectrum for compound 30

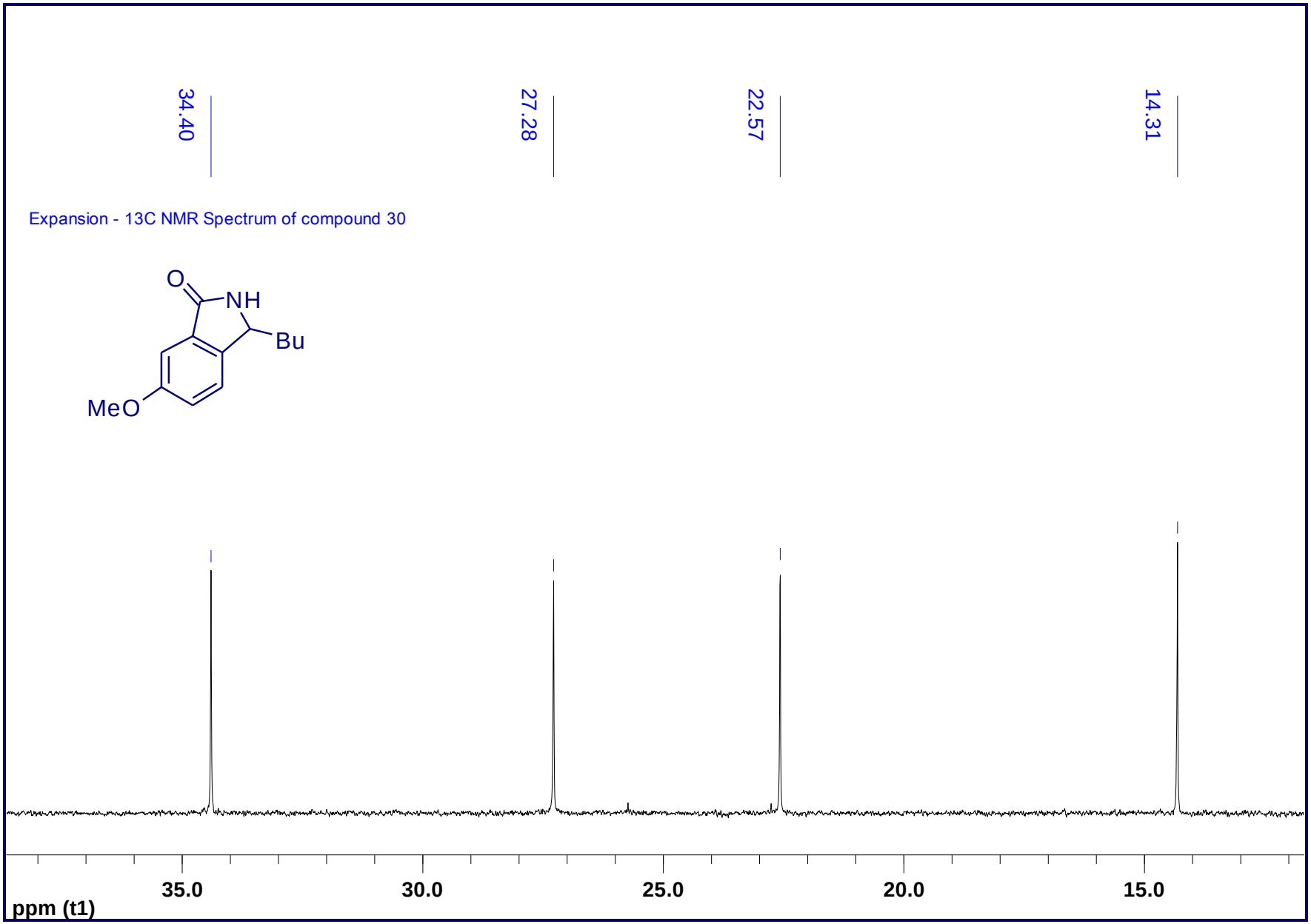


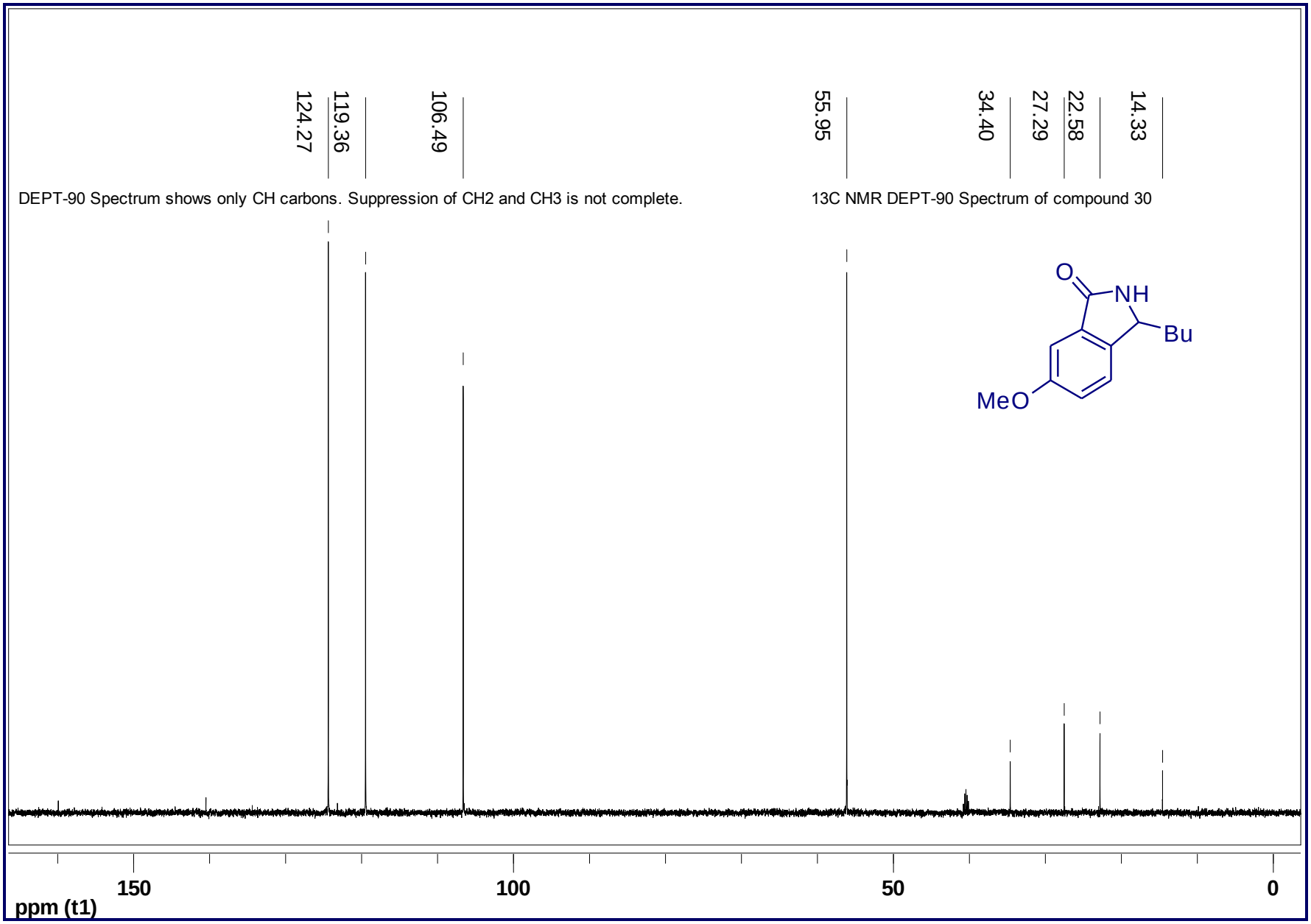


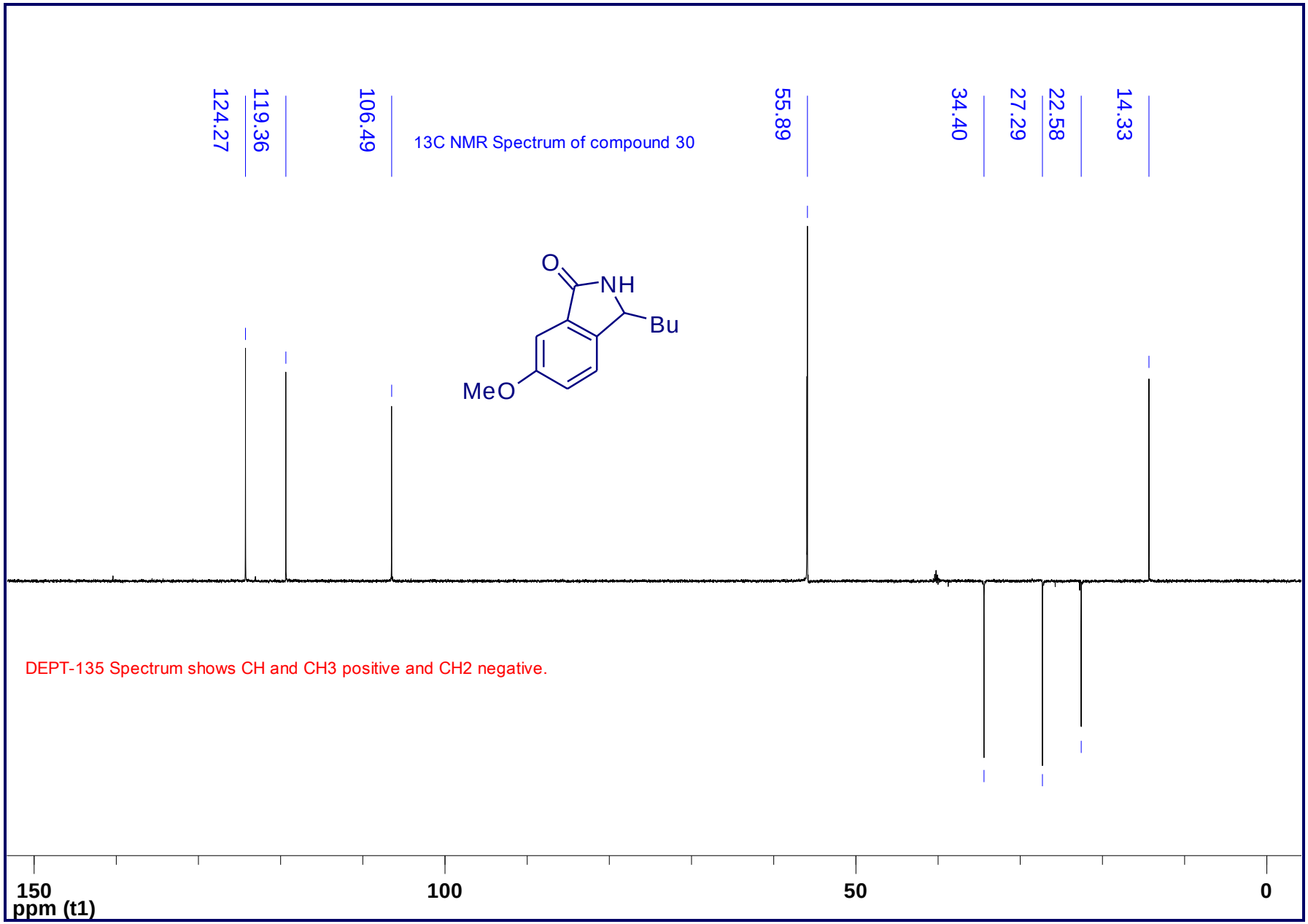
Expansion - 1H NMR spectrum for compound 30

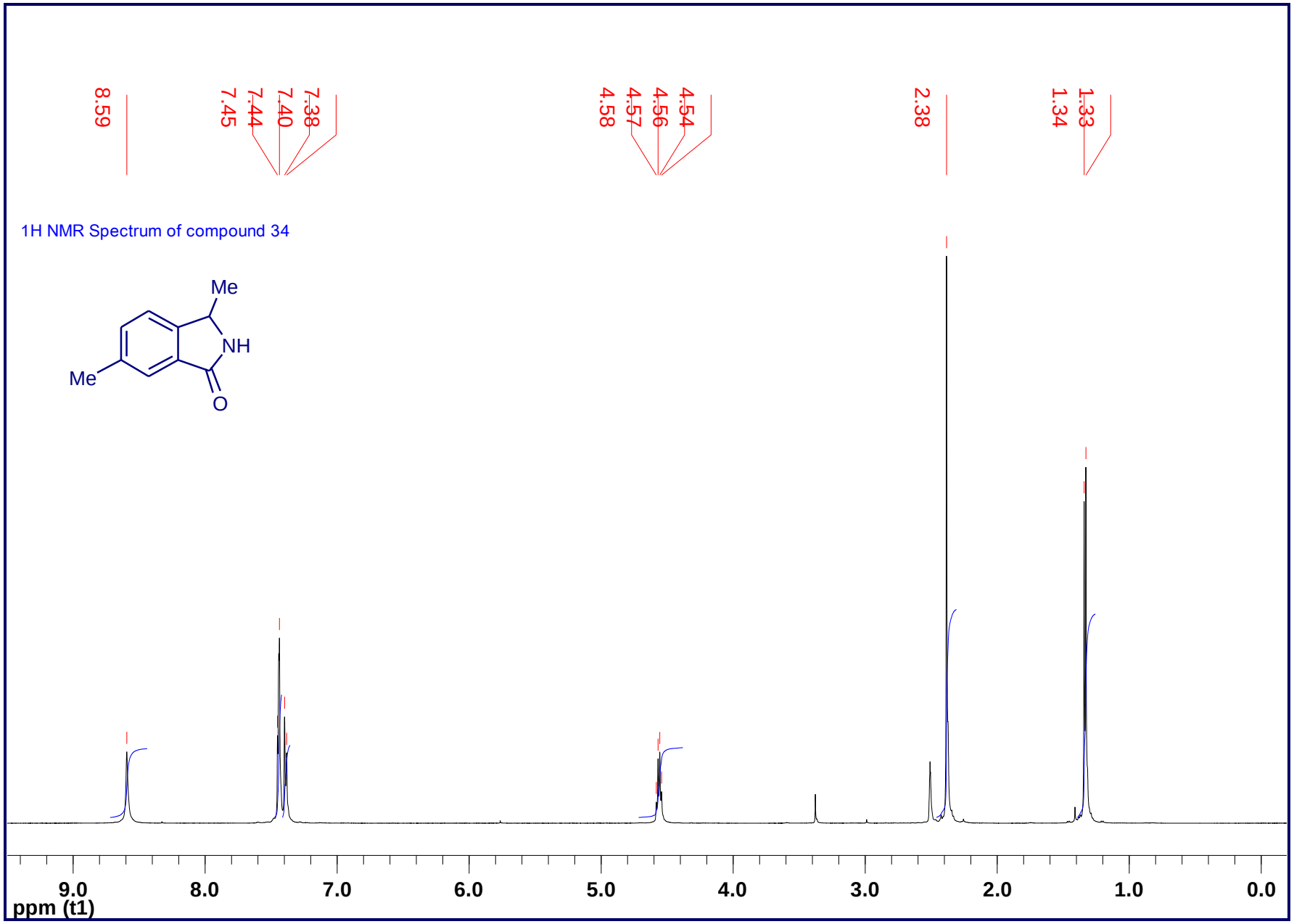

169.64
159.86
140.40
134.31
124.26
119.35
106.53
55.90
34.40
27.28
22.57
14.31
13C NMR Spectrum of compound 30
O
NH
Bu
MeO
150
100
50
0
ppm (t1)


169.64
159.86
140.40
134.31
124.26
119.35
106.53
Expansion - 13C NMR Spectrum of compound 30
O
NH
Bu
MeO
170
160
150
140
130
120
110
ppm (t1)

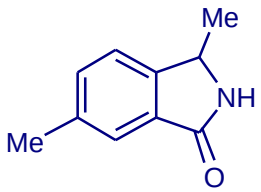

34.40
27.28
22.57
14.31
Expansion - 13C NMR Spectrum of compound 30
O
NH
Bu
MeO
35.0
30.0
25.0
20.0
15.0
ppm (t1)

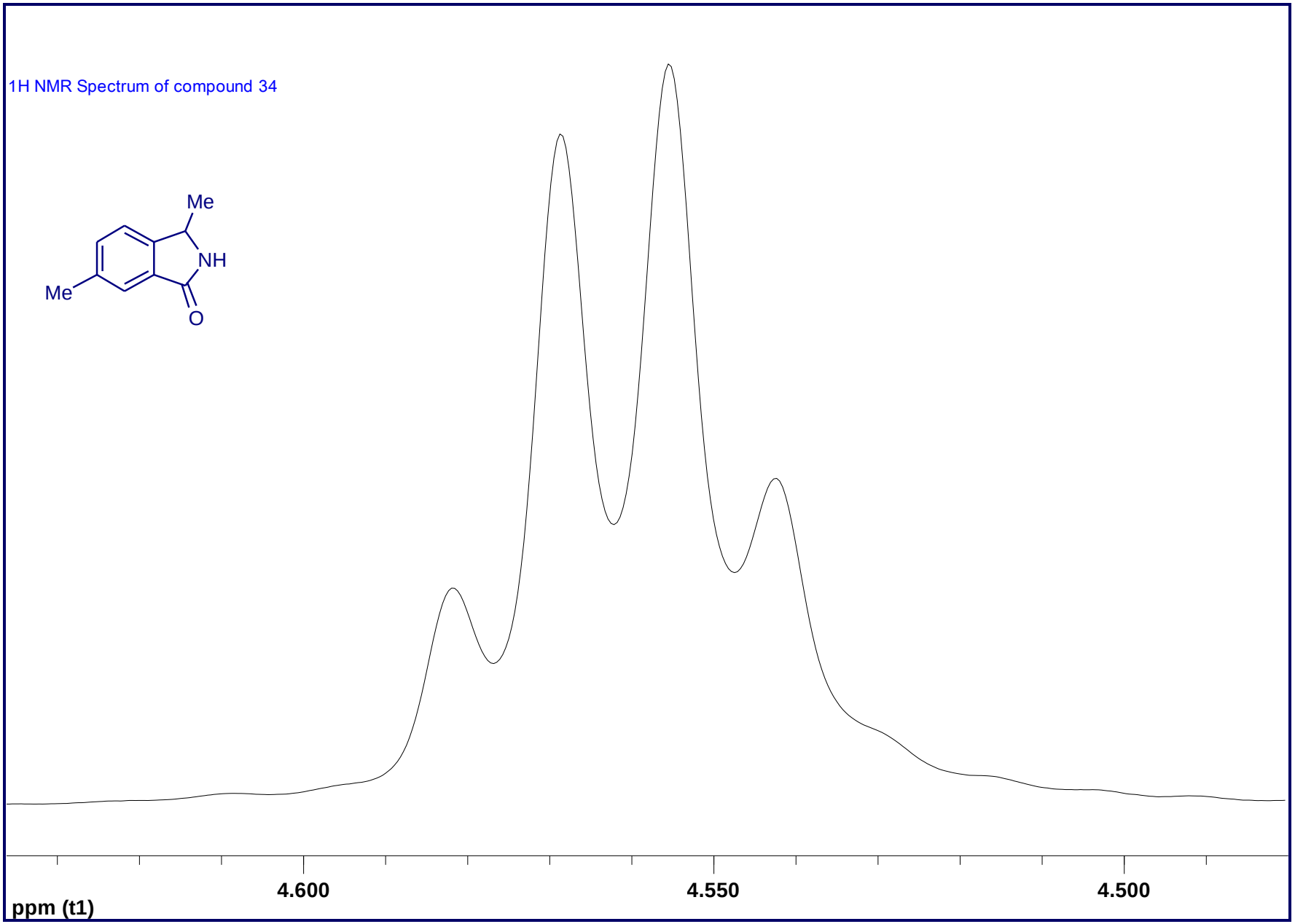

124.27
119.36
106.49
55.95
34.40
27.29
22.58
14.33
DEPT-90 Spectrum shows only CH carbons. Suppression of CH2 and CH3 is not complete.
13C NMR DEPT-90 Spectrum of compound 30
O
NH
Bu
MeO
150
100
50
0
ppm (t1)

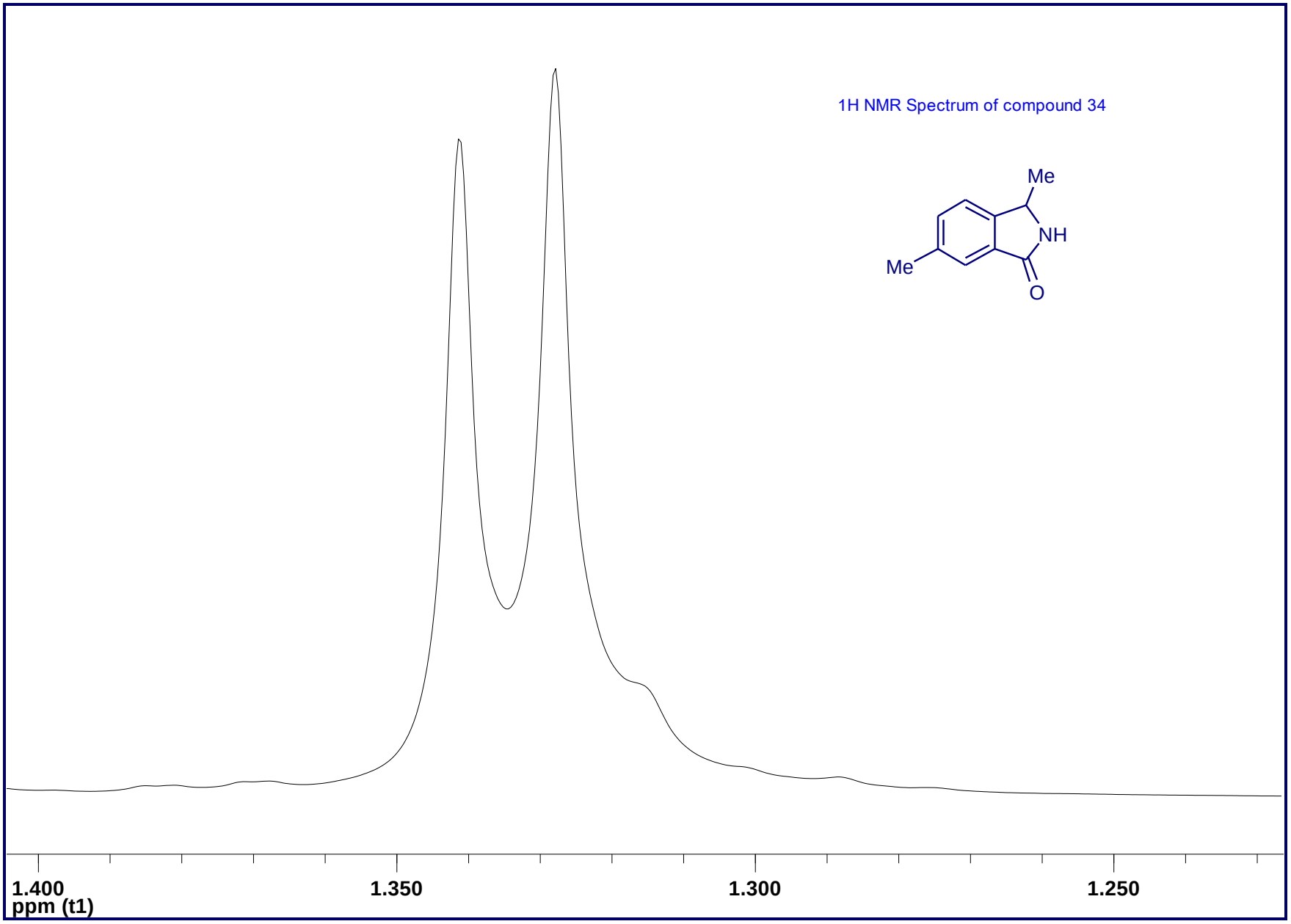

124.27
119.36
106.49
13C NMR Spectrum of compound 30
55.89
34.40
27.29
22.58
14.33
O
NH
Bu
MeO
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
150
100
50
0
ppm (t1)

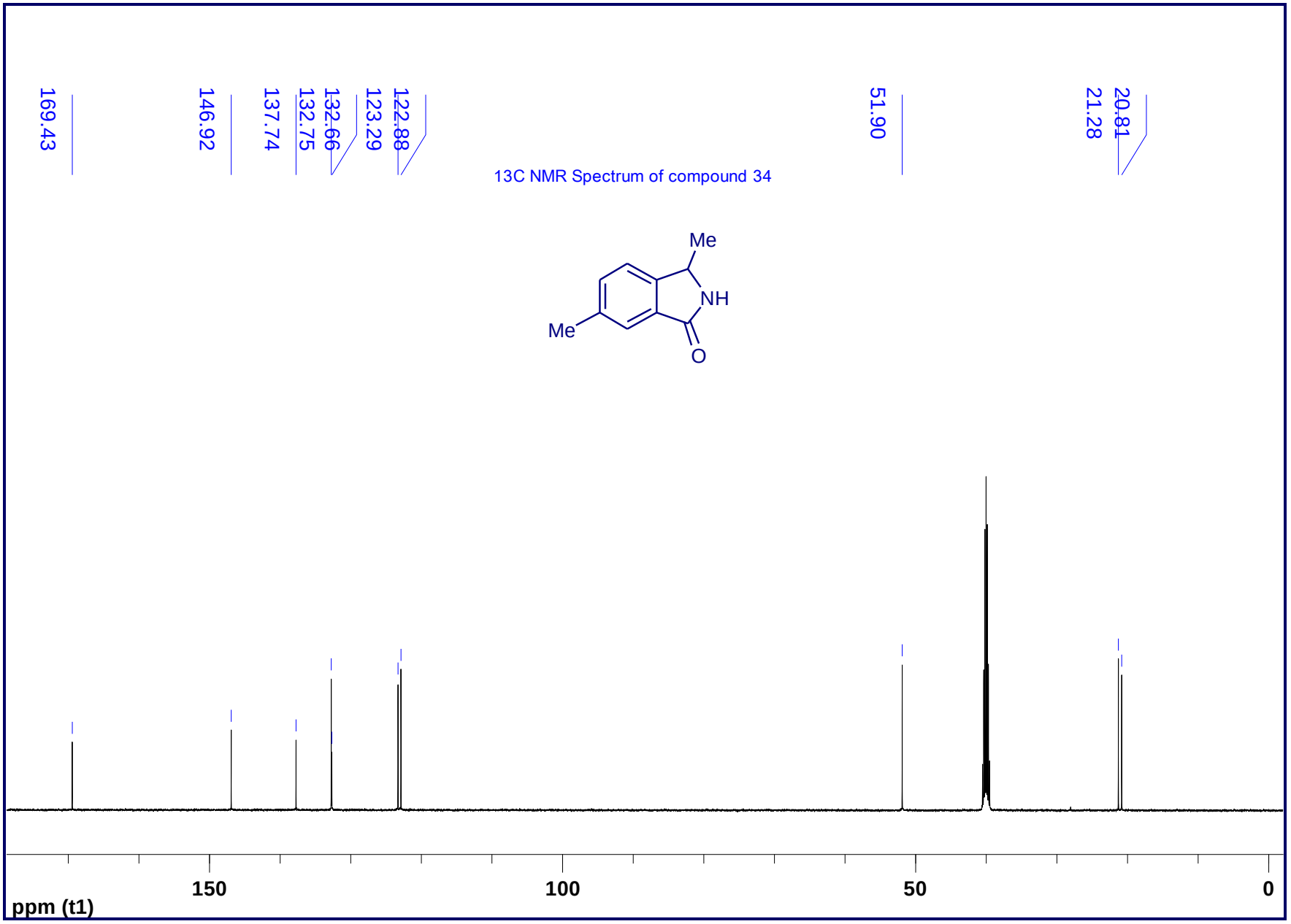

8.59
7.45
7.44
7.40
7.38
4.58
4.57
4.56
4.54
2.38
1.34
1.33
1H NMR Spectrum of compound 34
Me
NH
Me
O
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

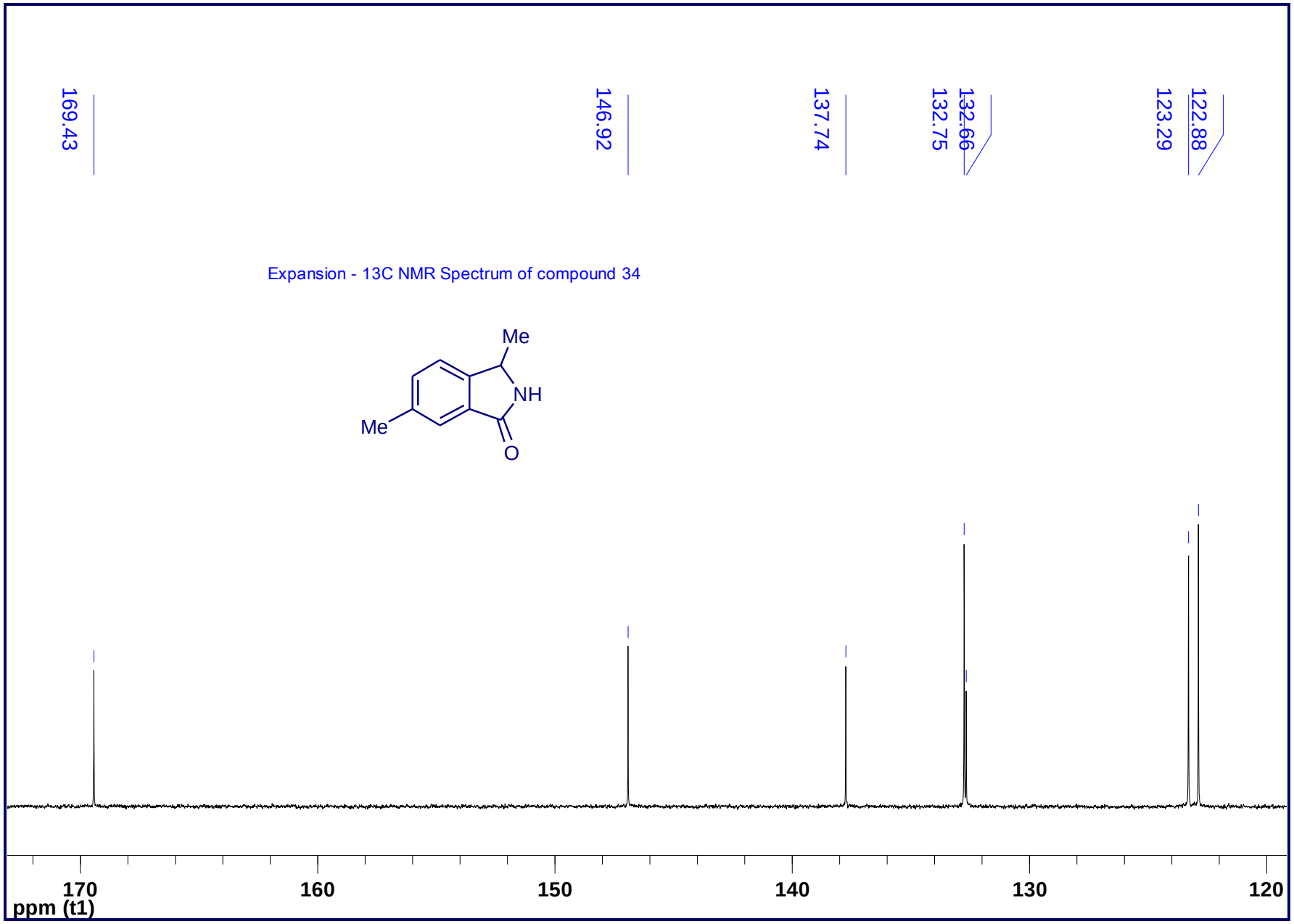
1H NMR Spectrum of compound 34

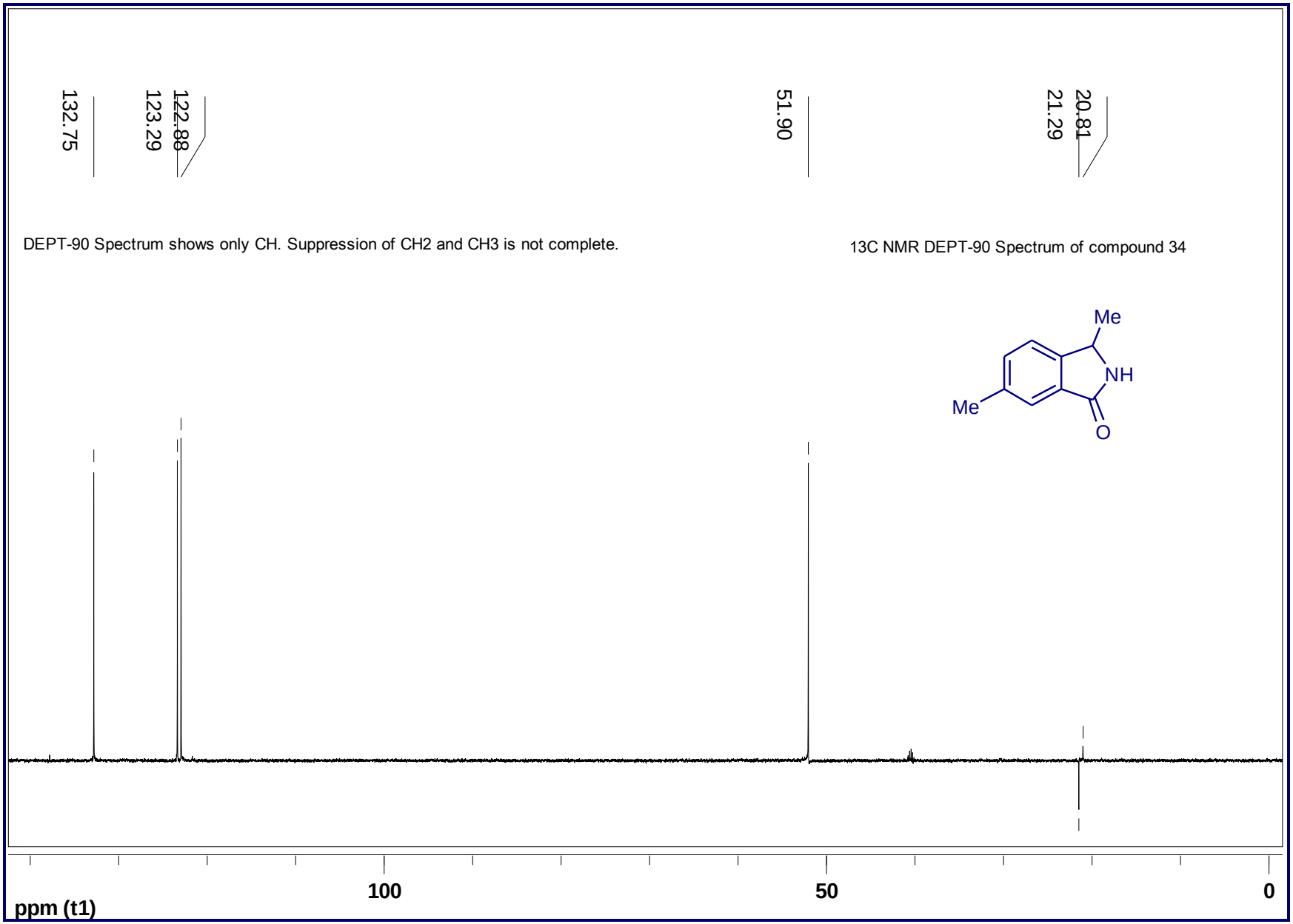


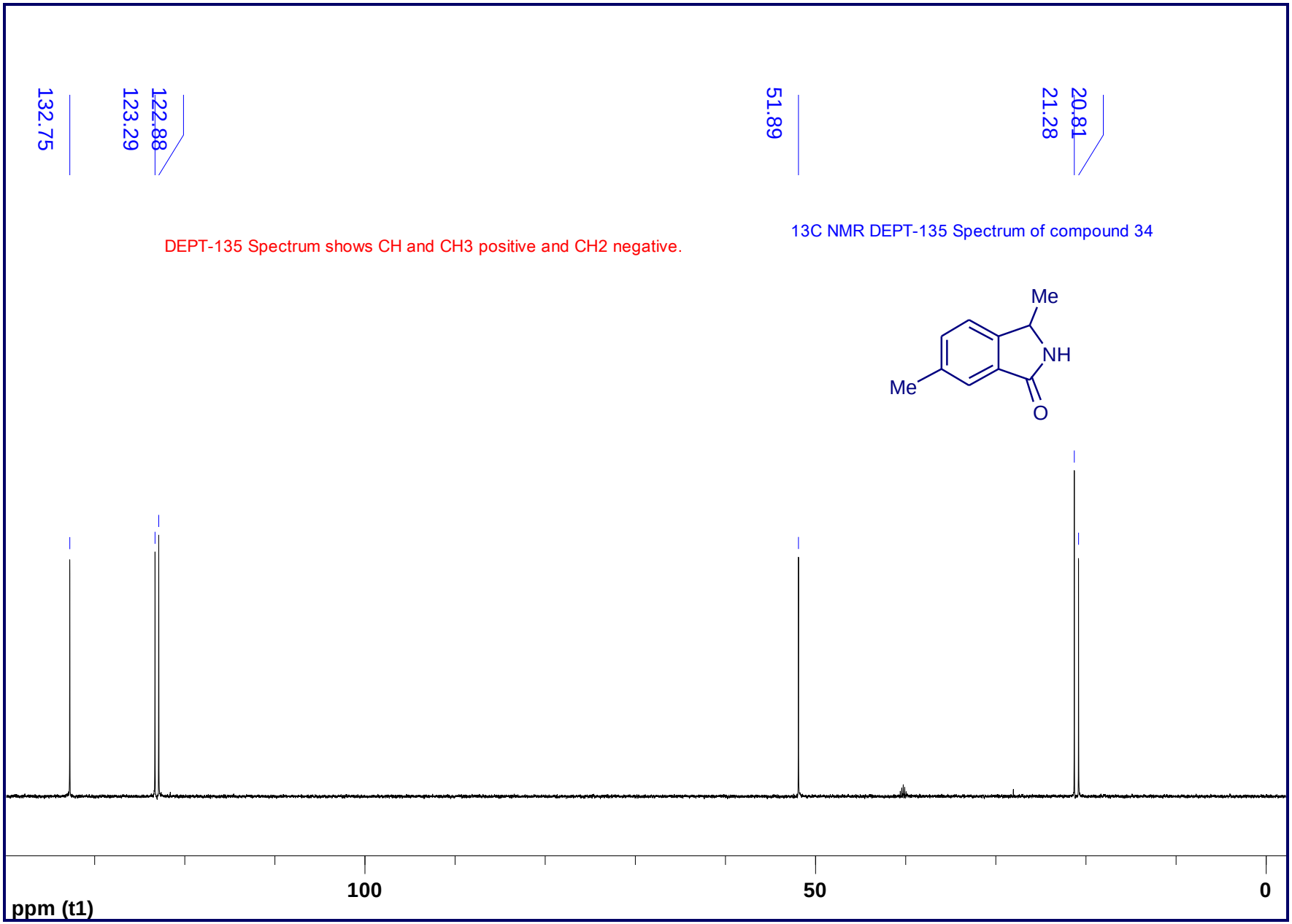

1H NMR Spectrum of compound 34
Me
NH
Me
O
4.600
4.550
4.500
ppm (t1)

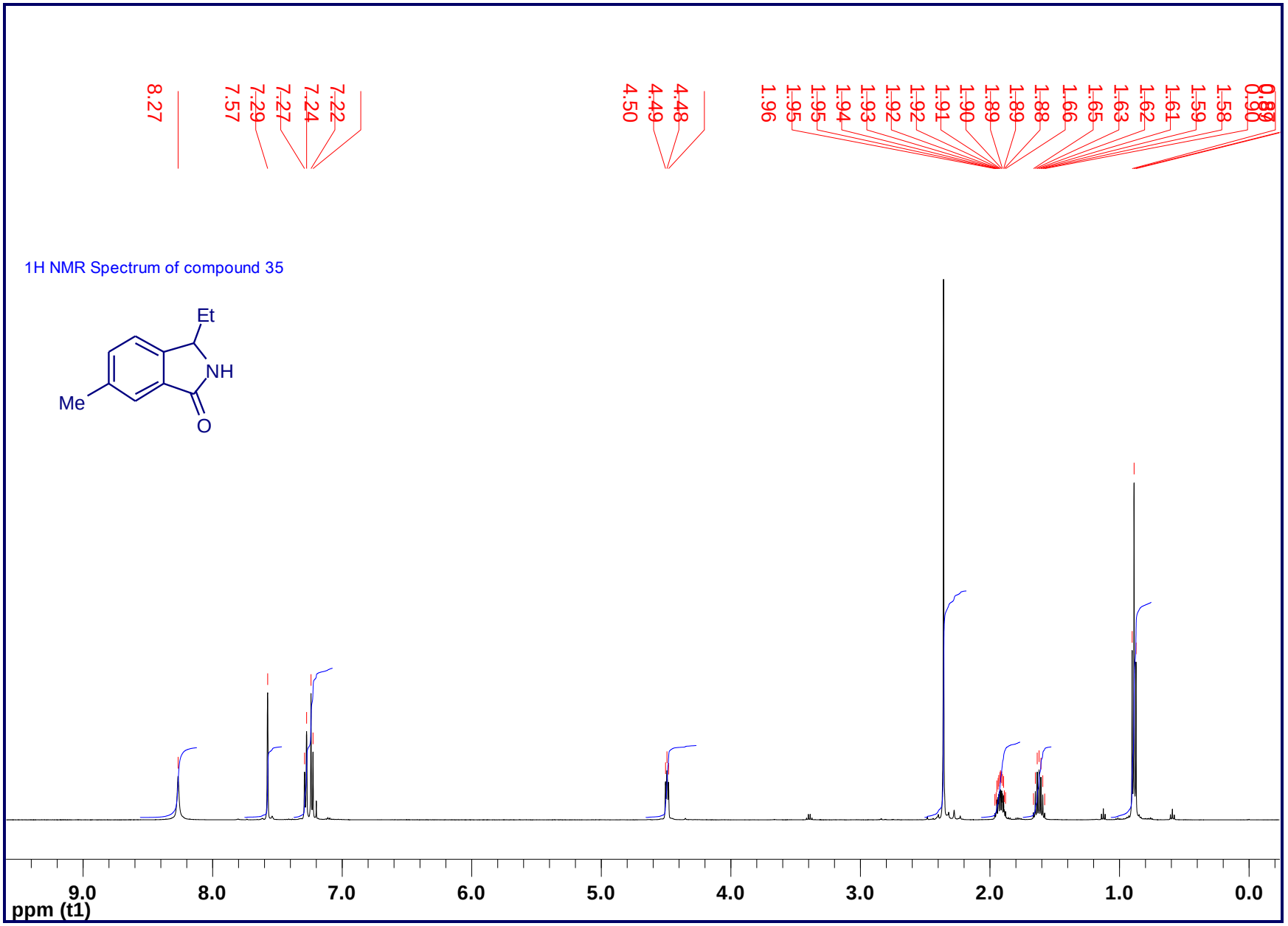

1H NMR Spectrum of compound 34
Me
NH
Me
O
1.400
ppm (t1)
1.350
1.300
1.250


169.43
146.92
137.74
132.75
132.66
123.29
122.88
51.90
21.28
20.81
13C NMR Spectrum of compound 34
Me
NH
Me
O
150
100
50
0
ppm (t1)

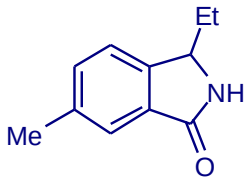

169.43
146.92
137.74
132.75
132.66
123.29
122.88
Expansion - 13C NMR Spectrum of compound 34
Me
NH
Me
O
170
160
150
140
130
120
ppm (t1)


132.75
123.29
122.88
51.90
21.29
20.81
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete.
13C NMR DEPT-90 Spectrum of compound 34
Me
NH
Me
O
100
50
0
ppm (t1)

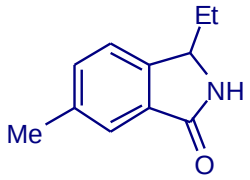

132.75
123.29
122.88
51.89
21.28
20.81
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
13C NMR DEPT-135 Spectrum of compound 34
Me
NH
Me
O
100
50
0
ppm (t1)

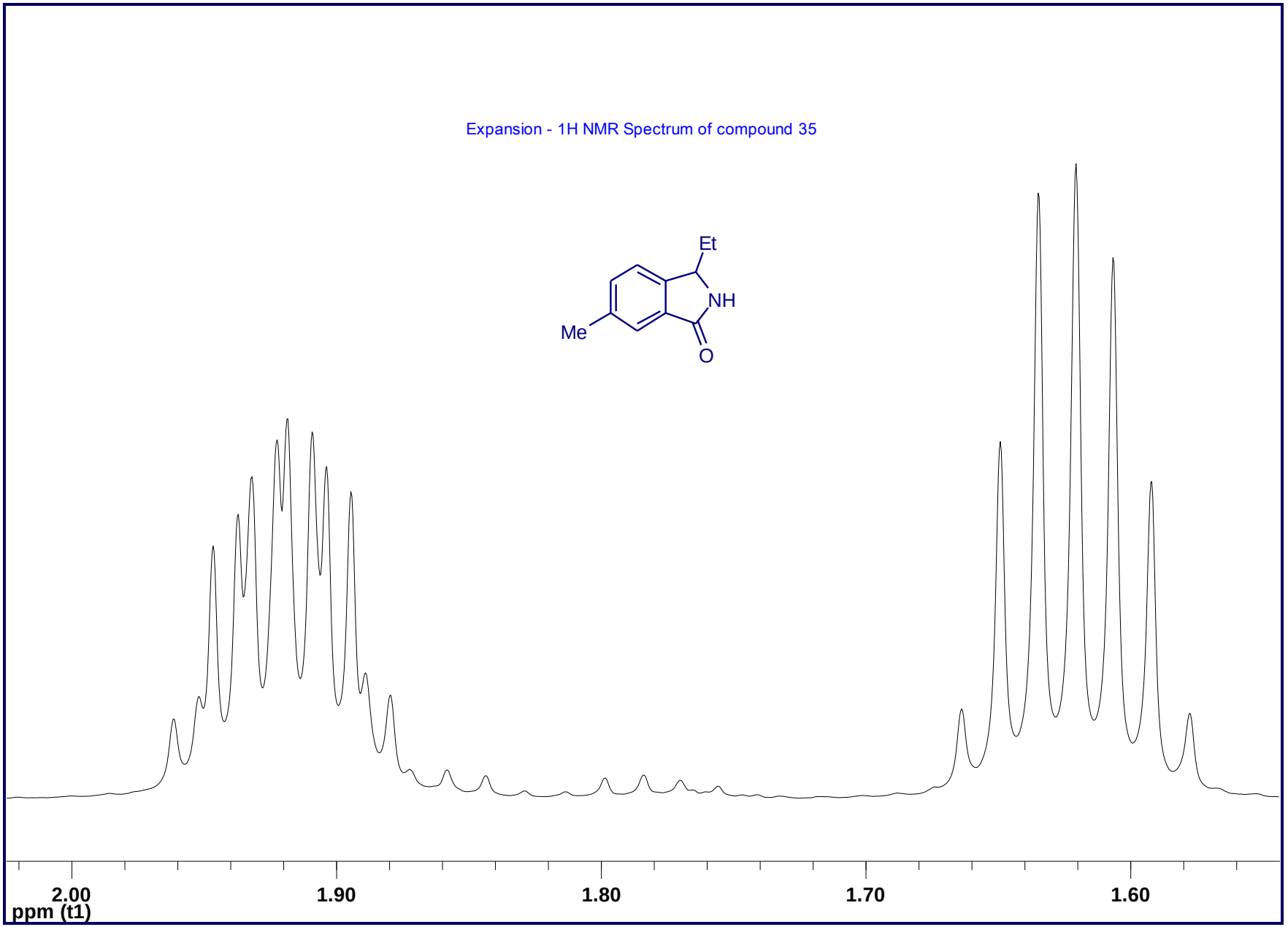

8.27
7.57
7.29
7.27
7.24
7.22
4.50
4.49
4.48
1.96
1.95
1.95
1.94
1.93
1.92
1.92
1.91
1.90
1.89
1.89
1.88
1.66
1.65
1.63
1.62
1.61
1.59
1.58
0.90
1H NMR Spectrum of compound 35
Et
NH
Me
O
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

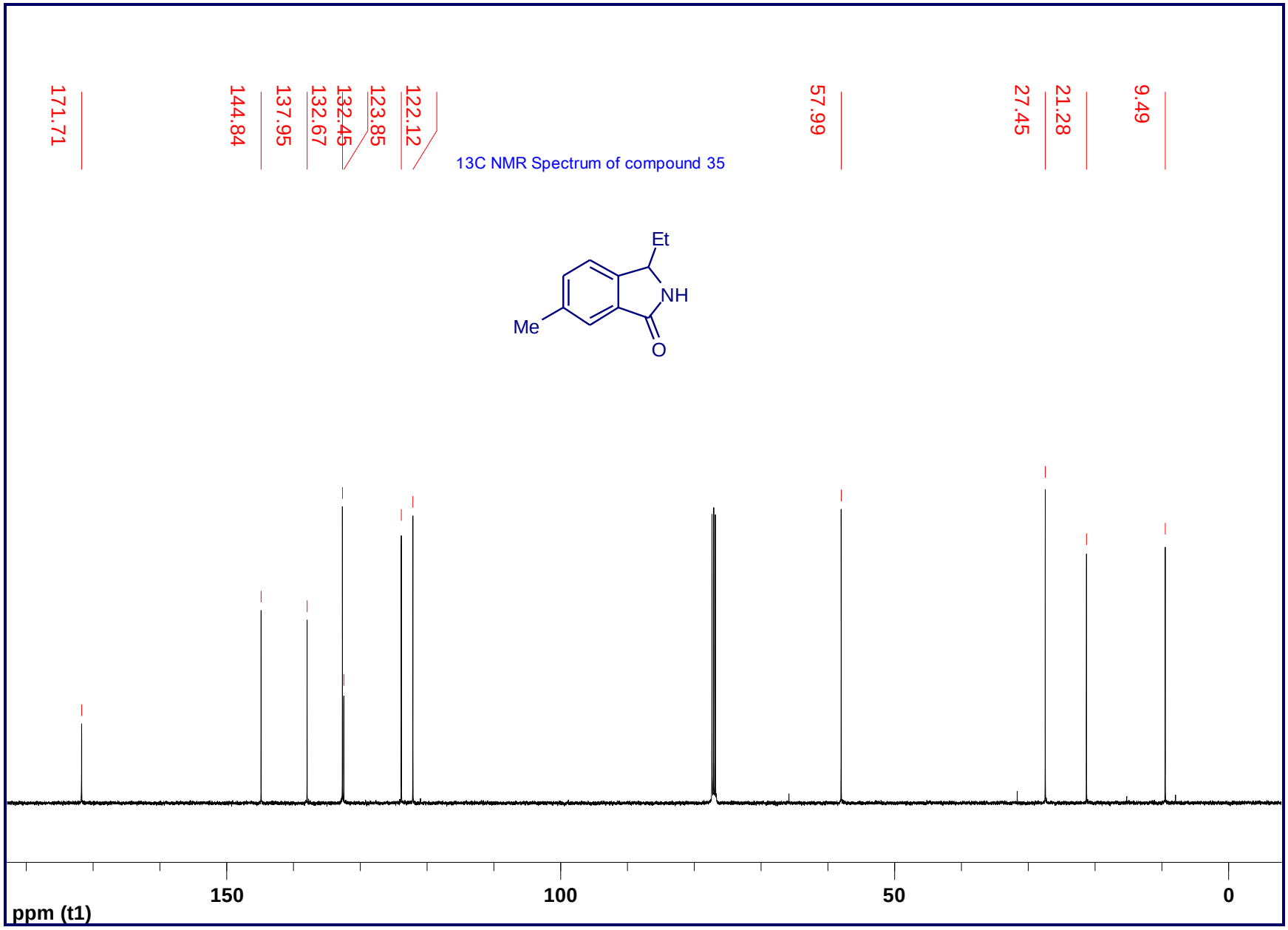
Expansion - 1H NMR Spectrum of compound 35

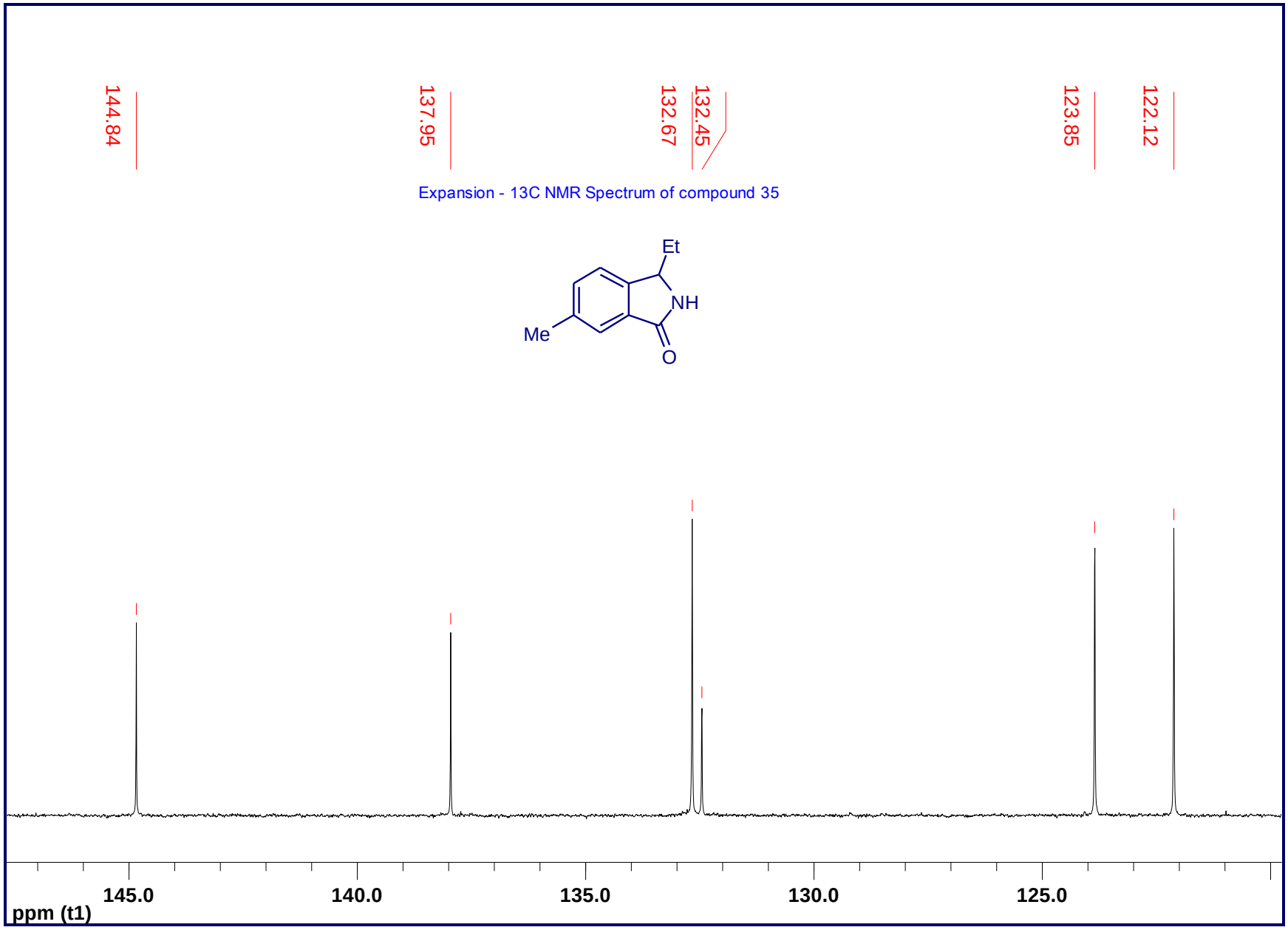


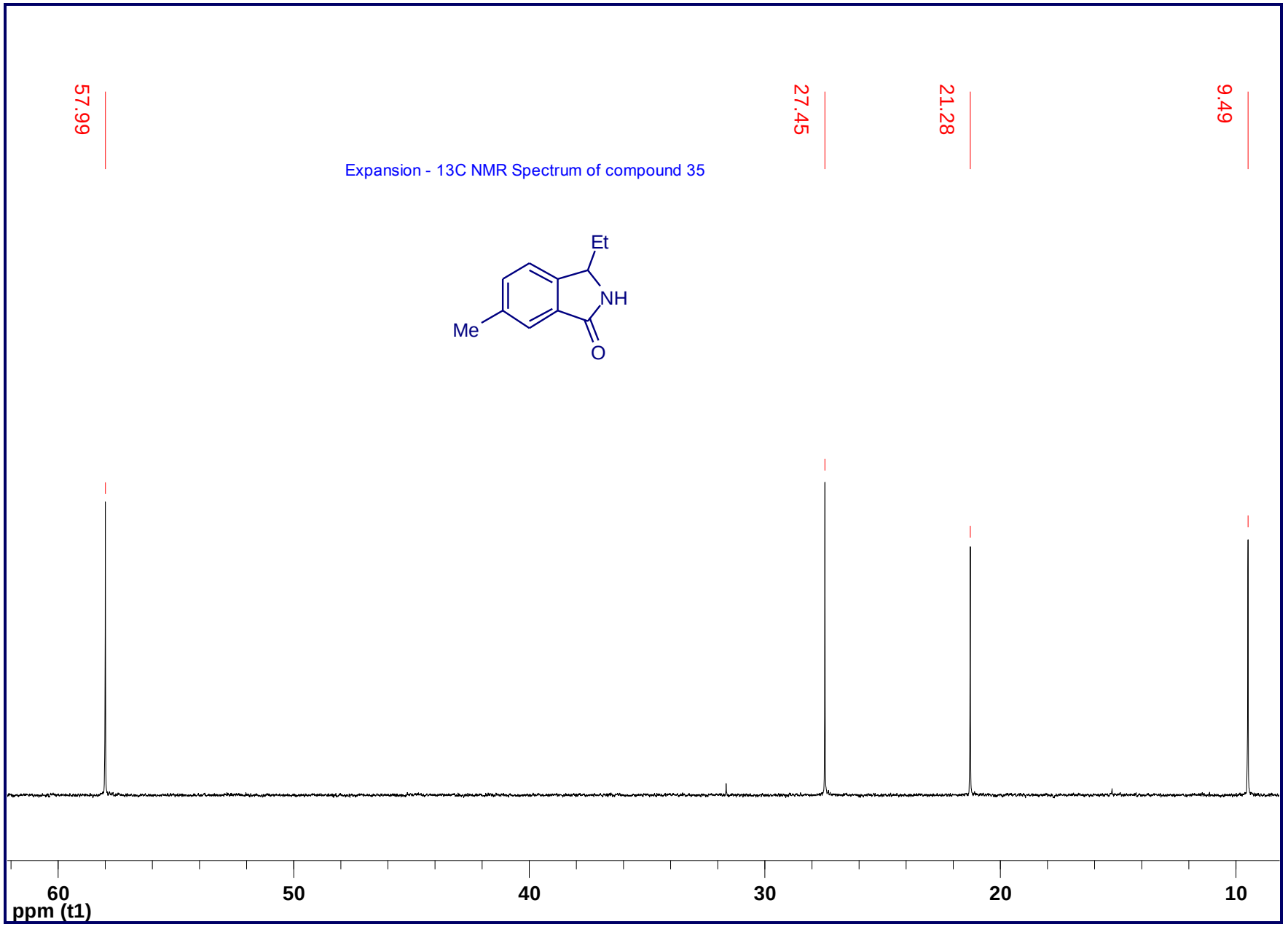
Expansion - 1H NMR Spectrum of compound 35

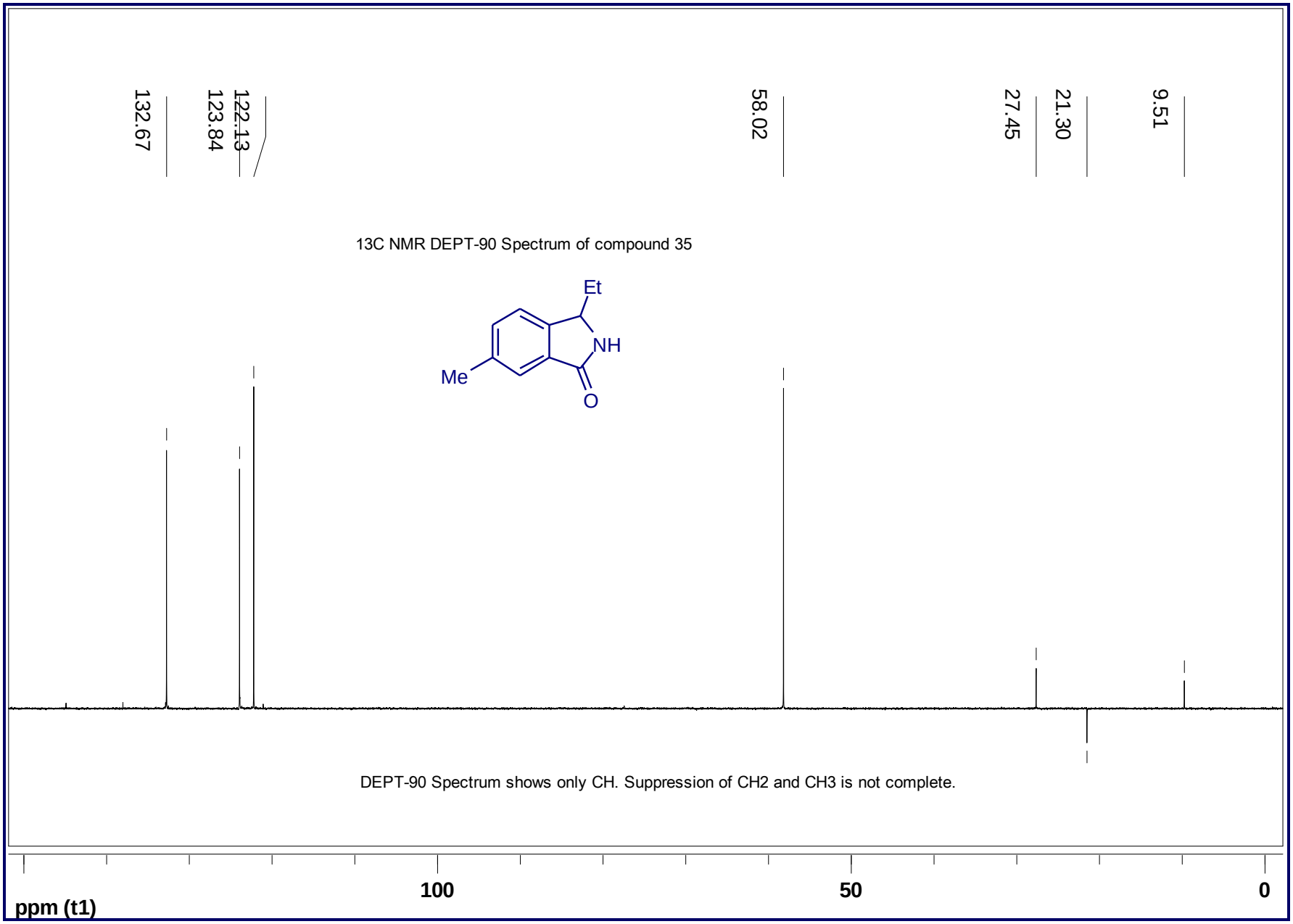


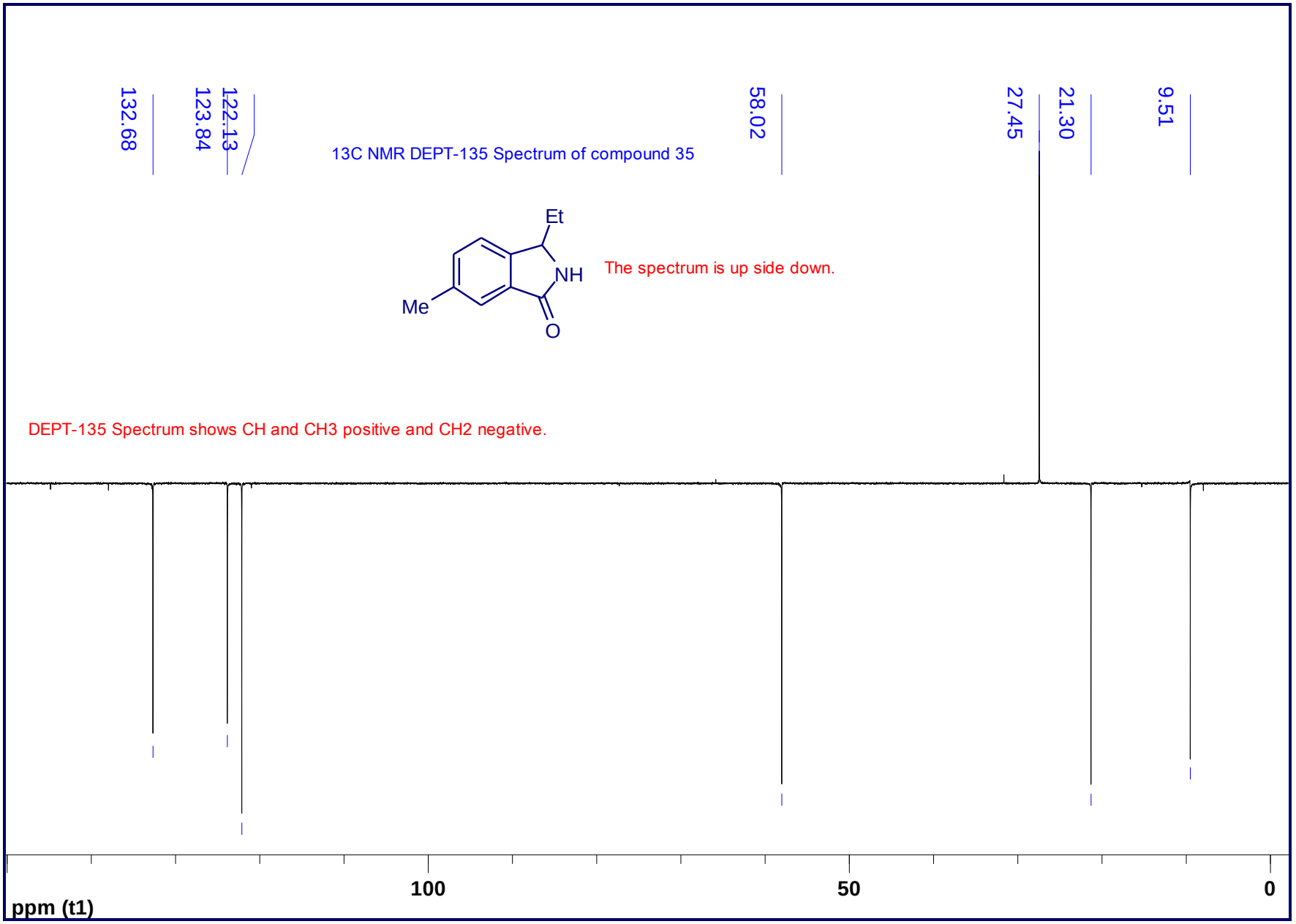

Expansion - 1H NMR Spectrum of compound 35
Et
NH
Me
O
2.00
1.90
1.80
1.70
1.60
ppm (t1)

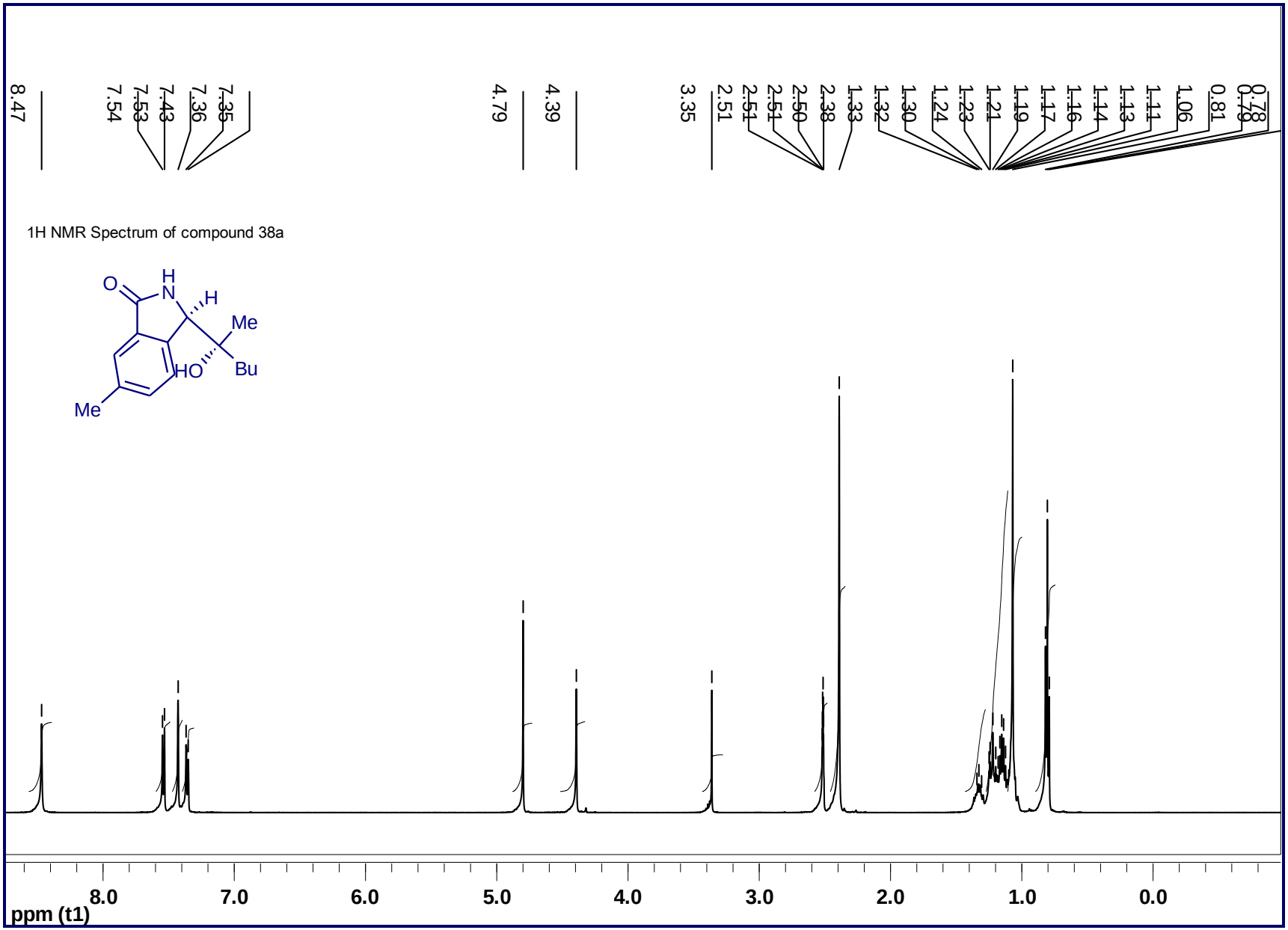

171.71
144.84
137.95
132.67
132.45
123.85
122.12
57.99
27.45
21.28
9.49
13C NMR Spectrum of compound 35
Et
NH
Me
O
150
100
50
0
ppm (t1)


144.84
137.95
132.67
132.45
123.85
122.12
Expansion - 13C NMR Spectrum of compound 35
Et
NH
Me
O
145.0
140.0
135.0
130.0
125.0
ppm (t1)

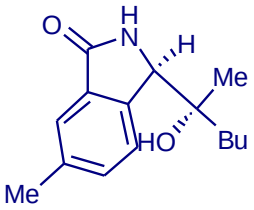

57.99
27.45
21.28
9.49
Expansion - 13C NMR Spectrum of compound 35
Et
NH
Me
O
60
50
40
30
20
10
ppm (t1)

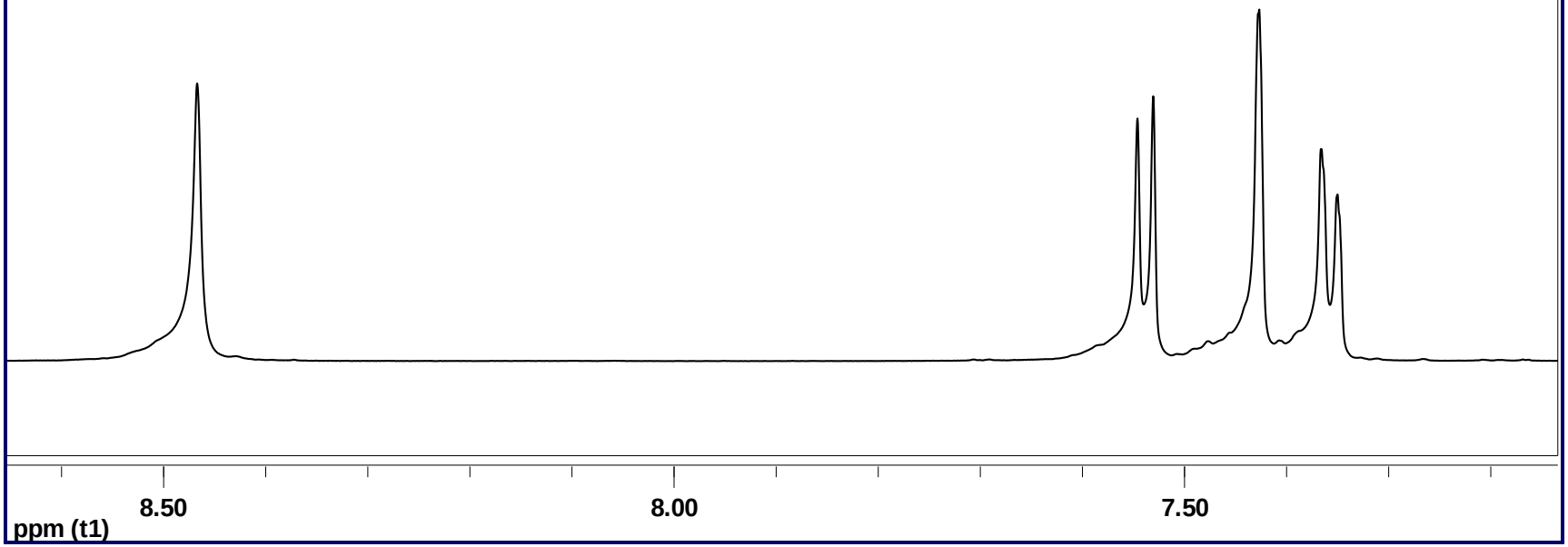

132.67
123.84
122.13
58.02
27.45
21.30
9.51
13C NMR DEPT-90 Spectrum of compound 35
Et
NH
Me
O
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete.
100
50
0
ppm (t1)


132.68
123.84
122.13
58.02
27.45
21.30
9.51
13C NMR DEPT-135 Spectrum of compound 35
Et
NH
Me
O
The spectrum is up side down.
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
100
50
0
ppm (t1)

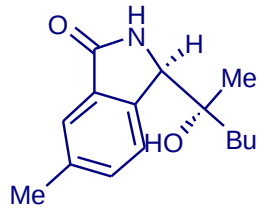

8.47
7.54
7.53
7.43
7.36
7.35
4.79
4.39
3.35
2.51
2.51
2.51
2.50
2.38
1.33
1.32
1.30
1.24
1.23
1.21
1.19
1.17
1.16
1.14
1.13
1.11
1.06
0.81
0.79
0.78
1H NMR Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

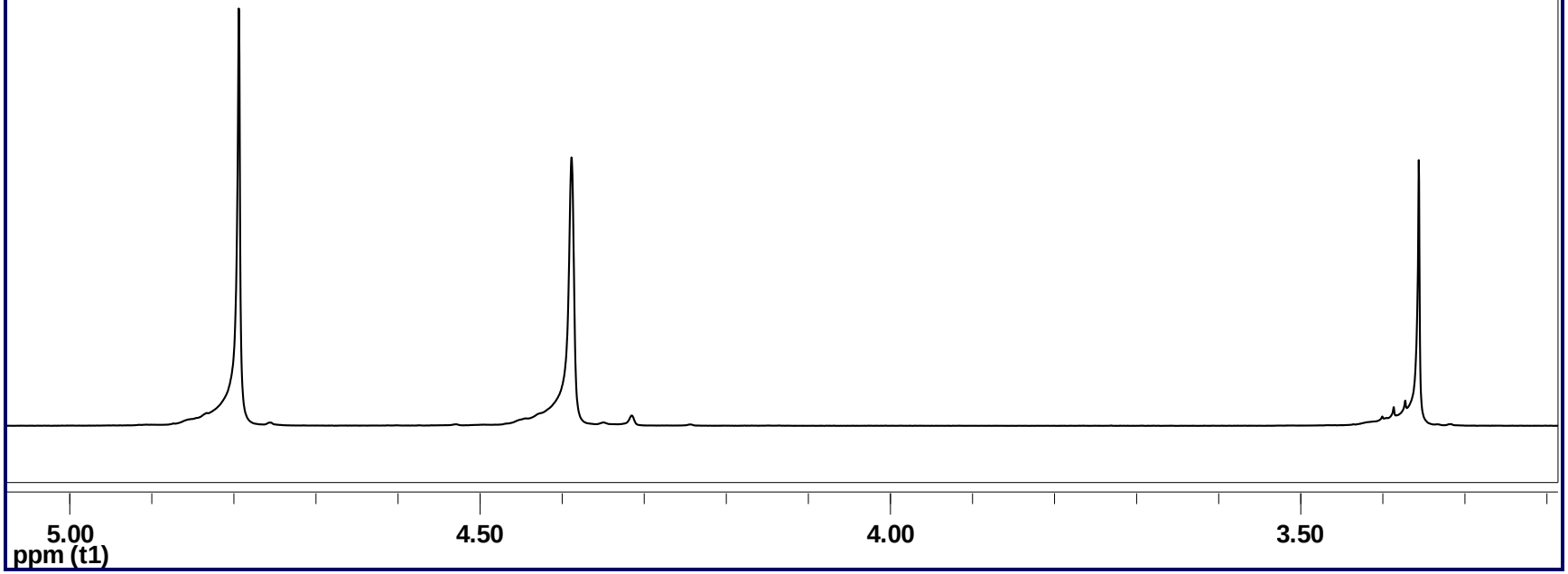
Expansion - 1H NMR Spectrum of compound 38a



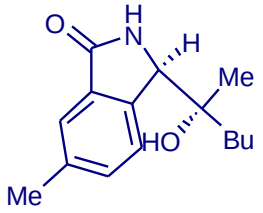


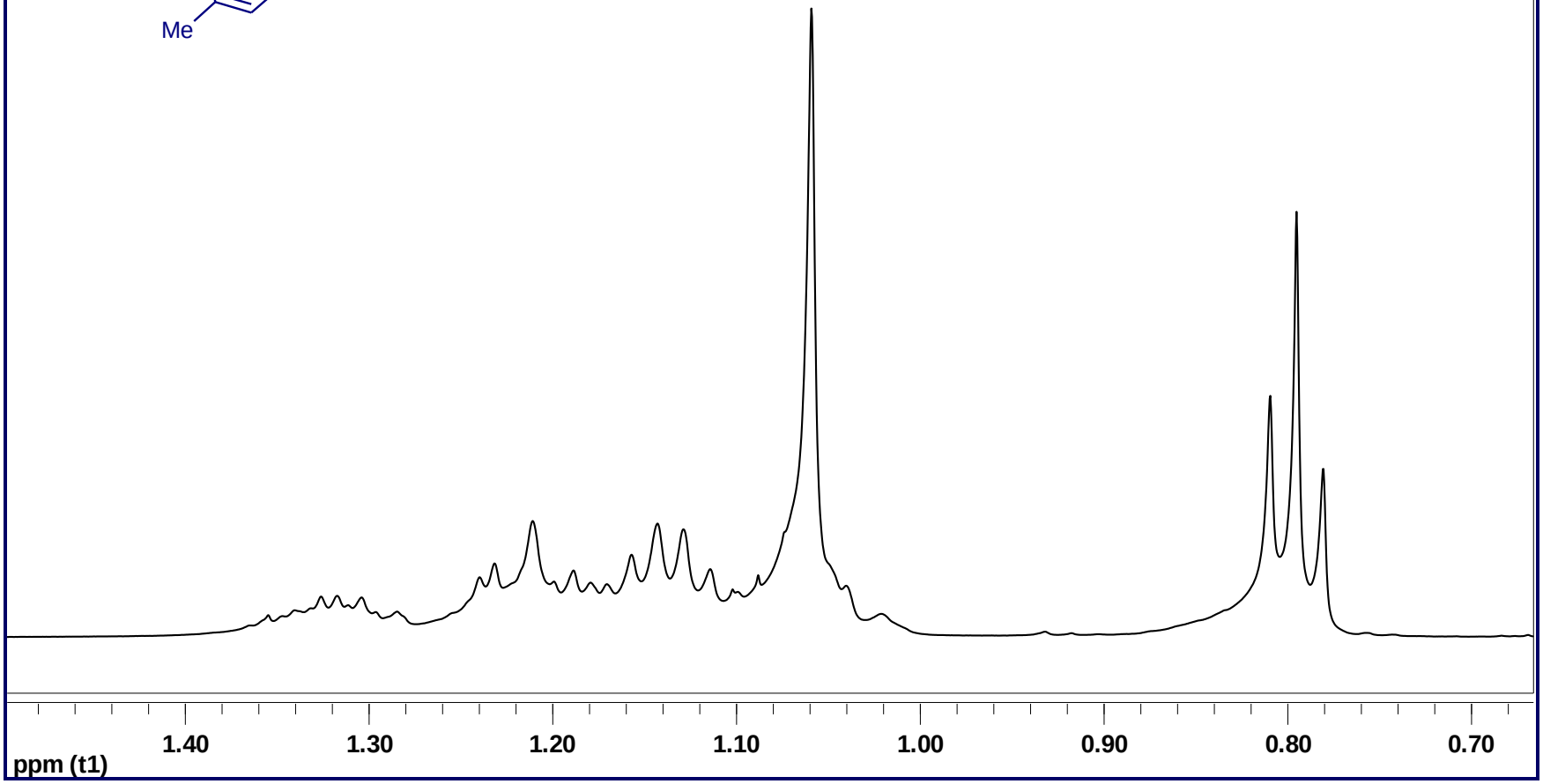
Expansion - 1H NMR Spectrum of compound 38a

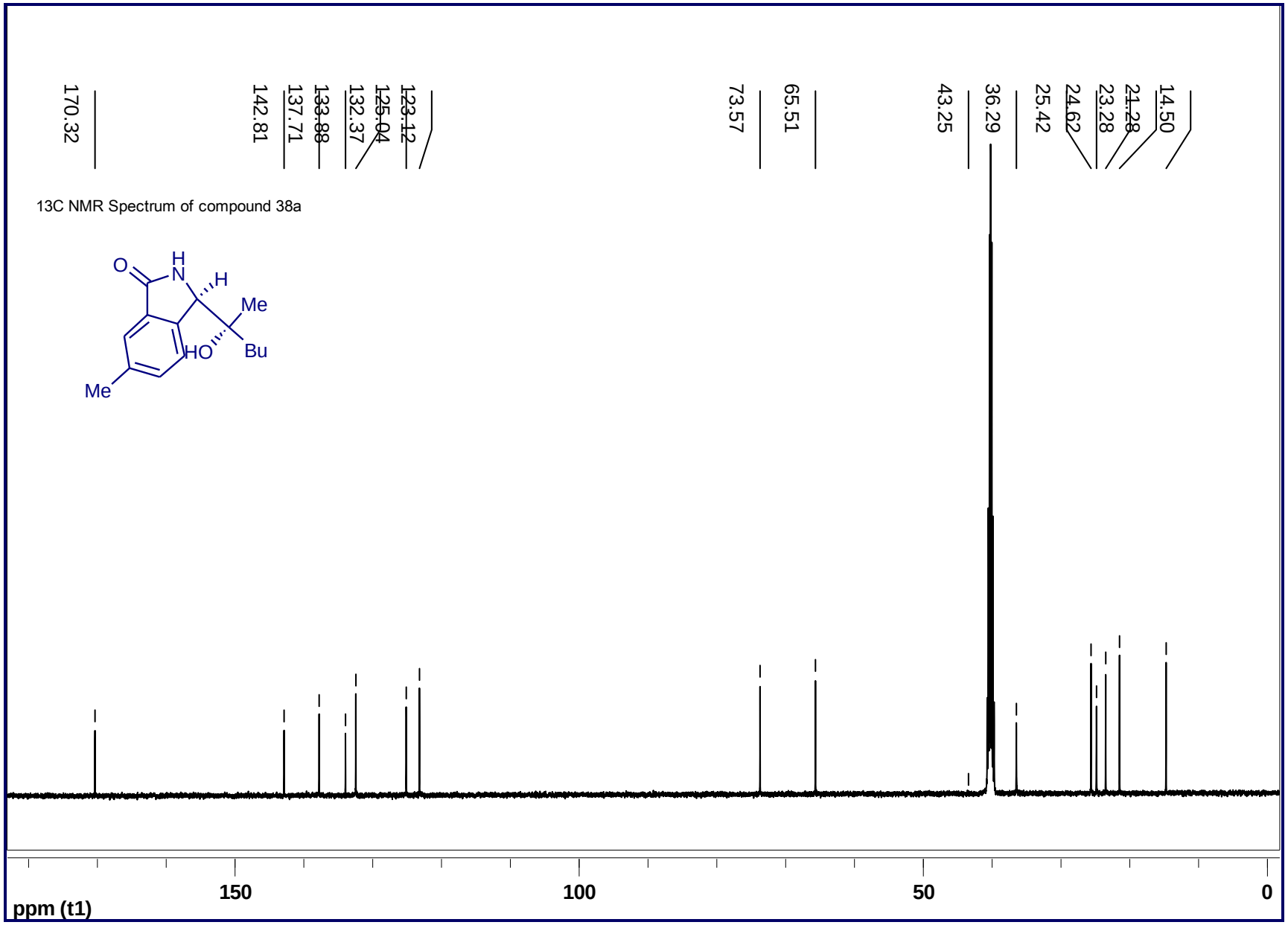


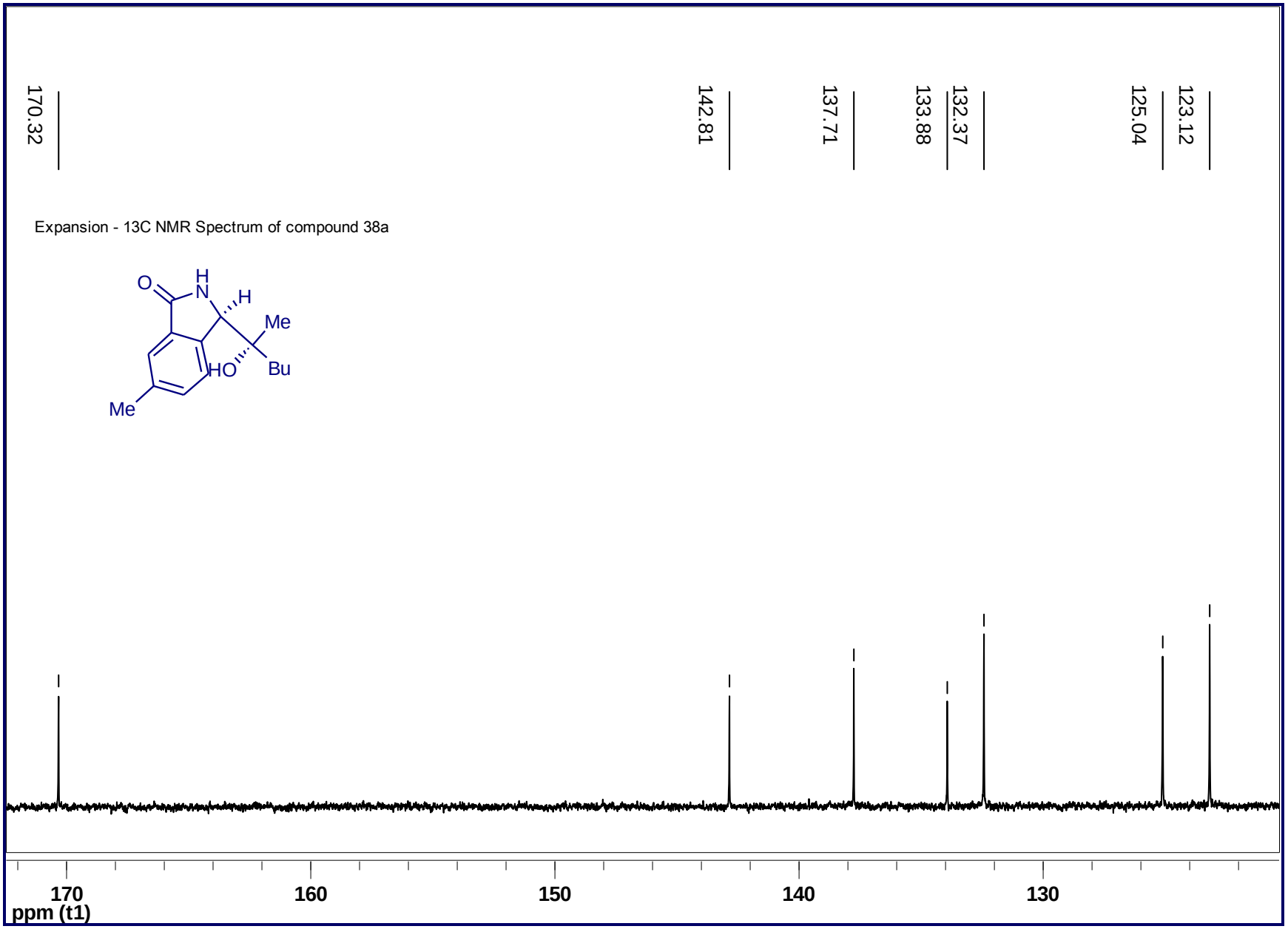


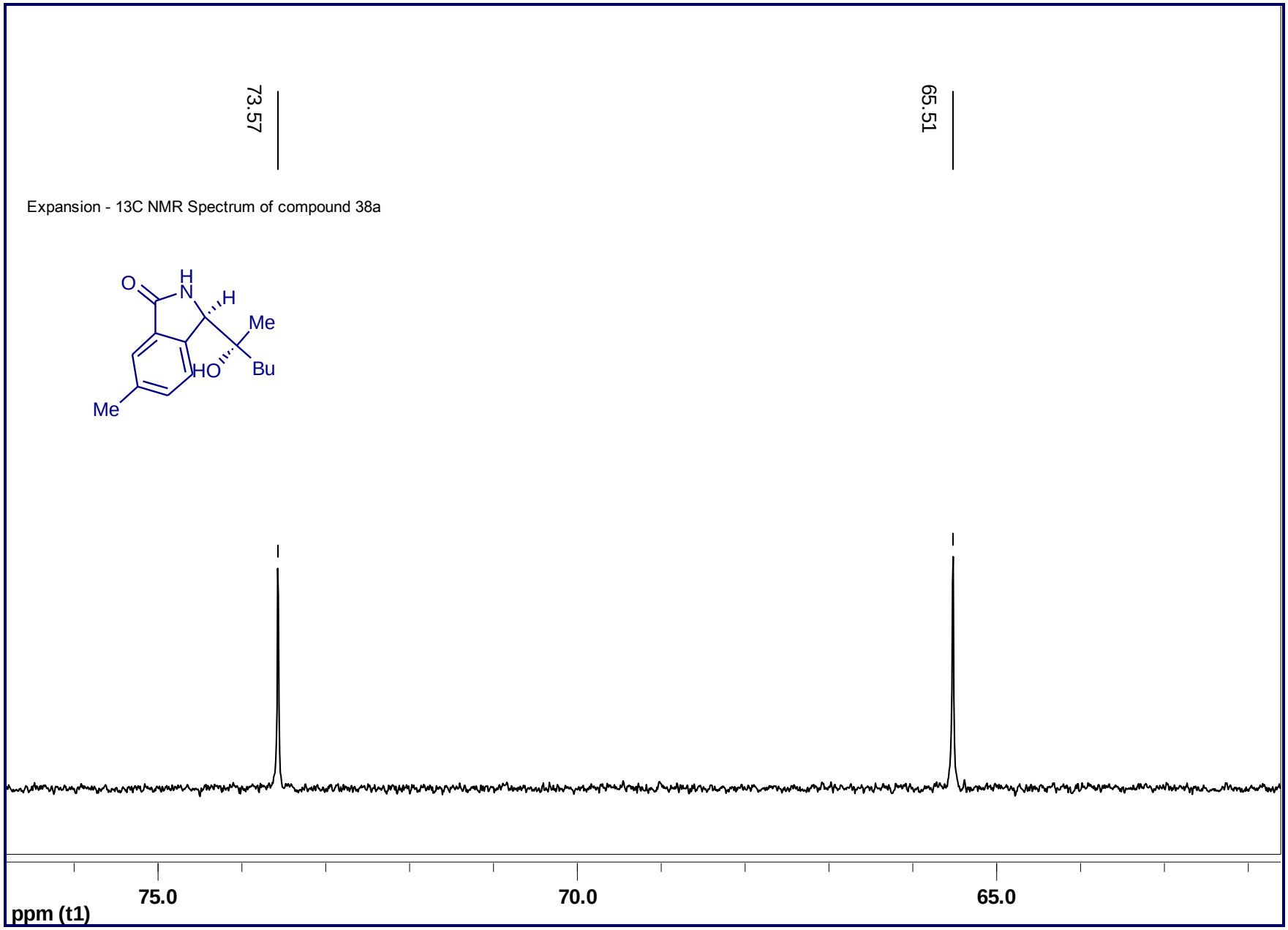
Expansion - 1H NMR Spectrum of compound 38a

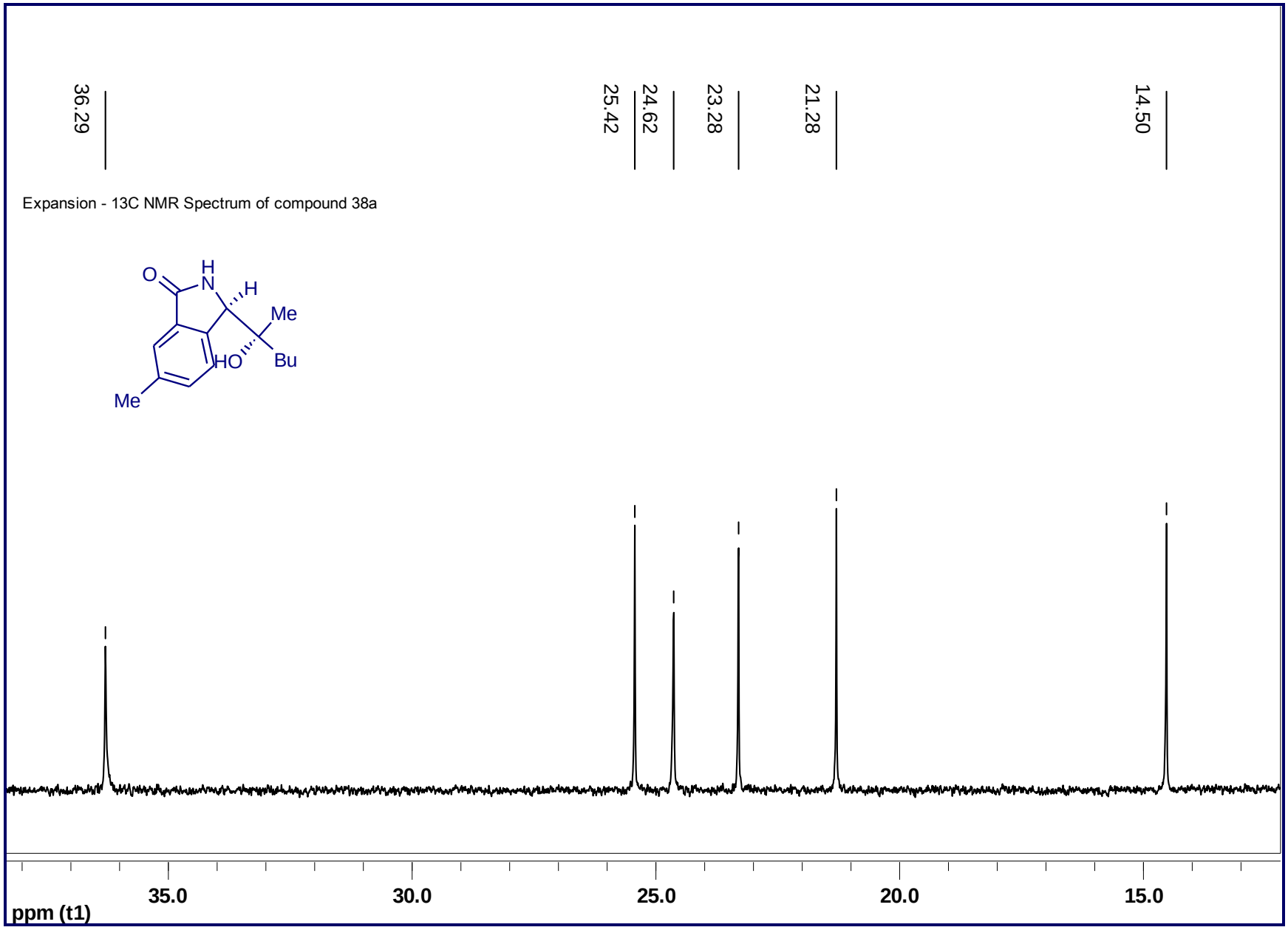


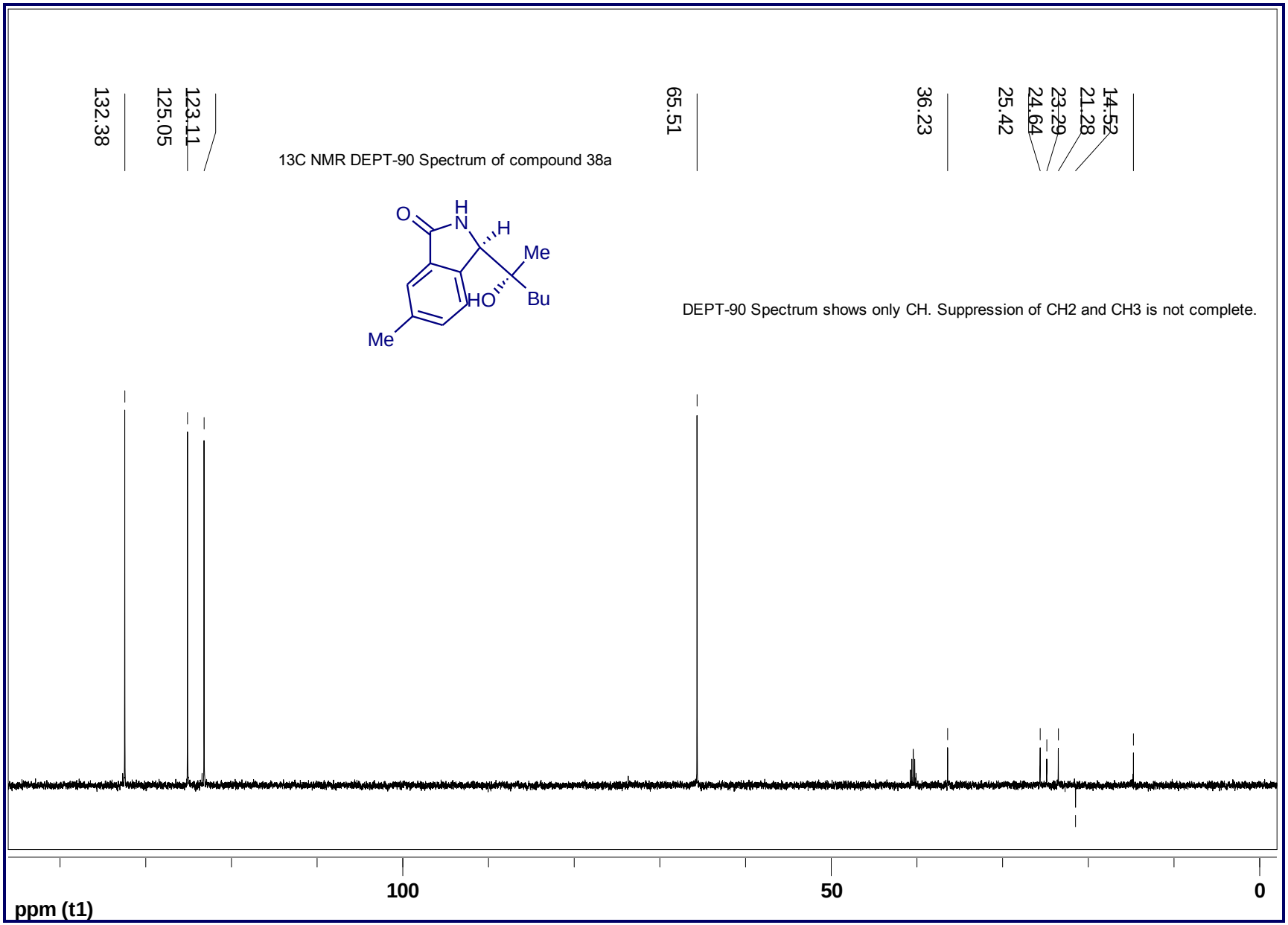


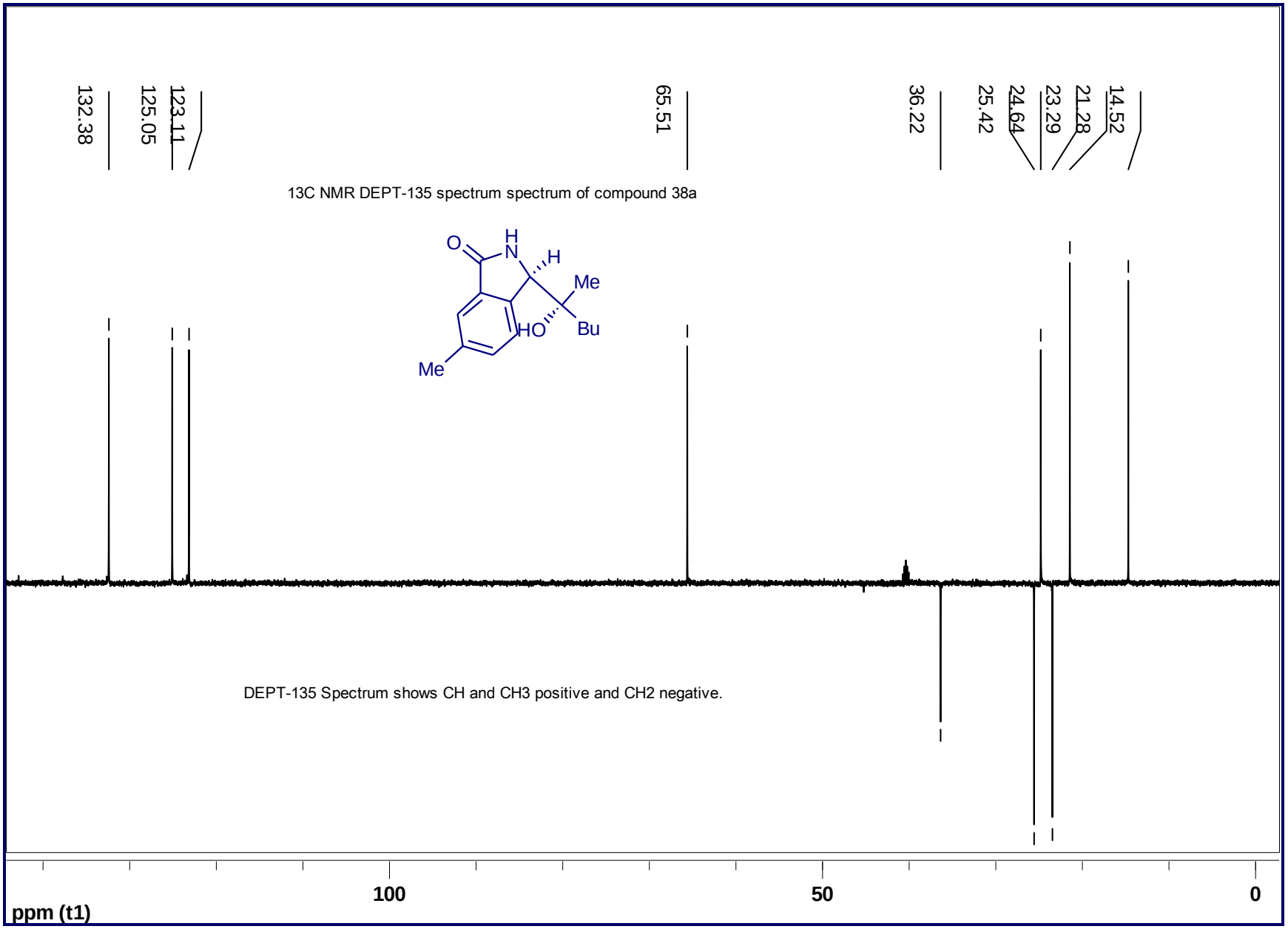

170.32
142.81
137.71
133.88
132.37
125.04
123.12
73.57
65.51
43.25
36.29
25.42
24.62
23.28
21.28
14.50
13C NMR Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
150
100
50
0
ppm (t1)

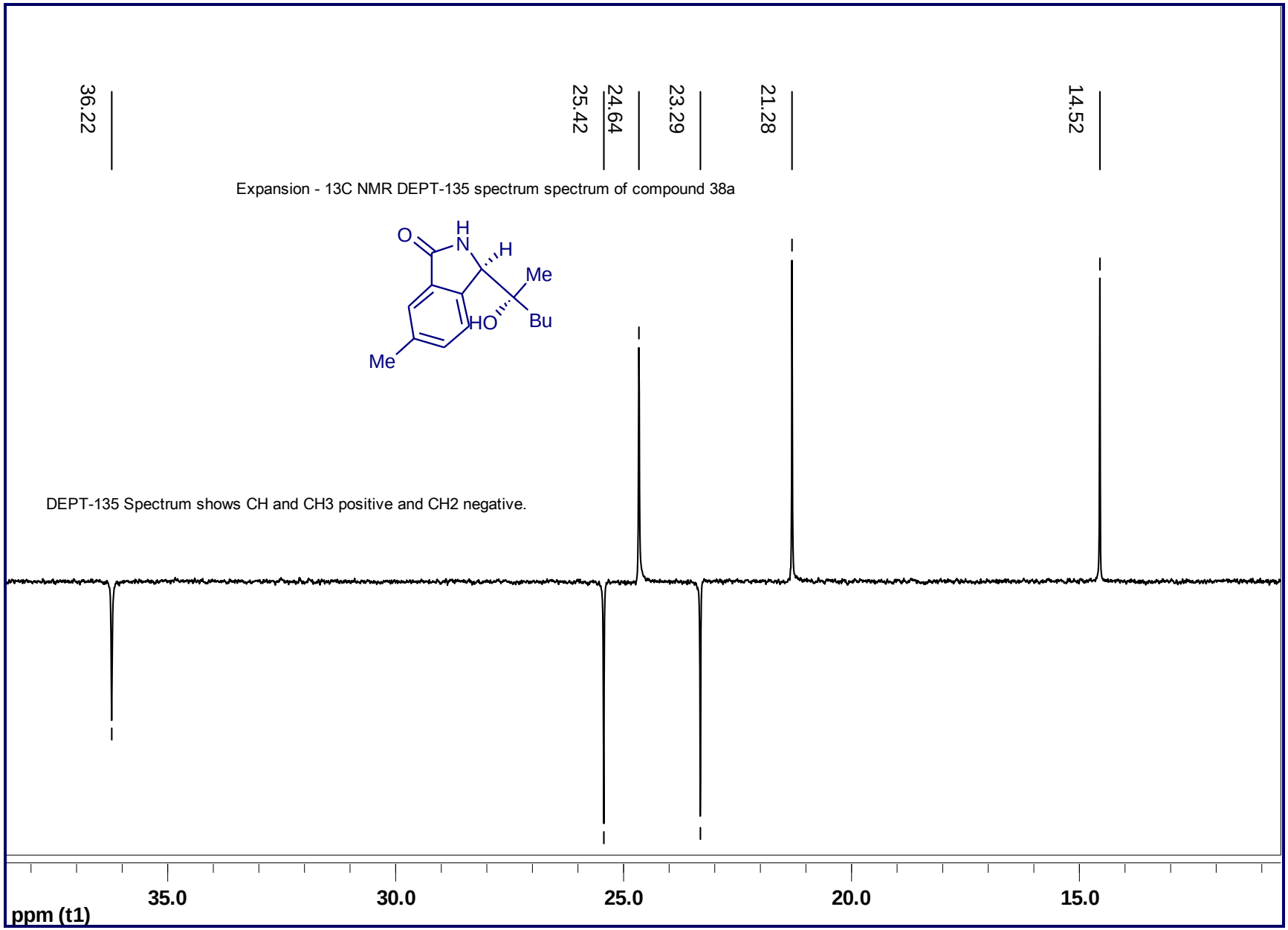

170.32
142.81
137.71
133.88
132.37
125.04
123.12
Expansion - 13C NMR Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
170
160
150
140
130
ppm (t1)

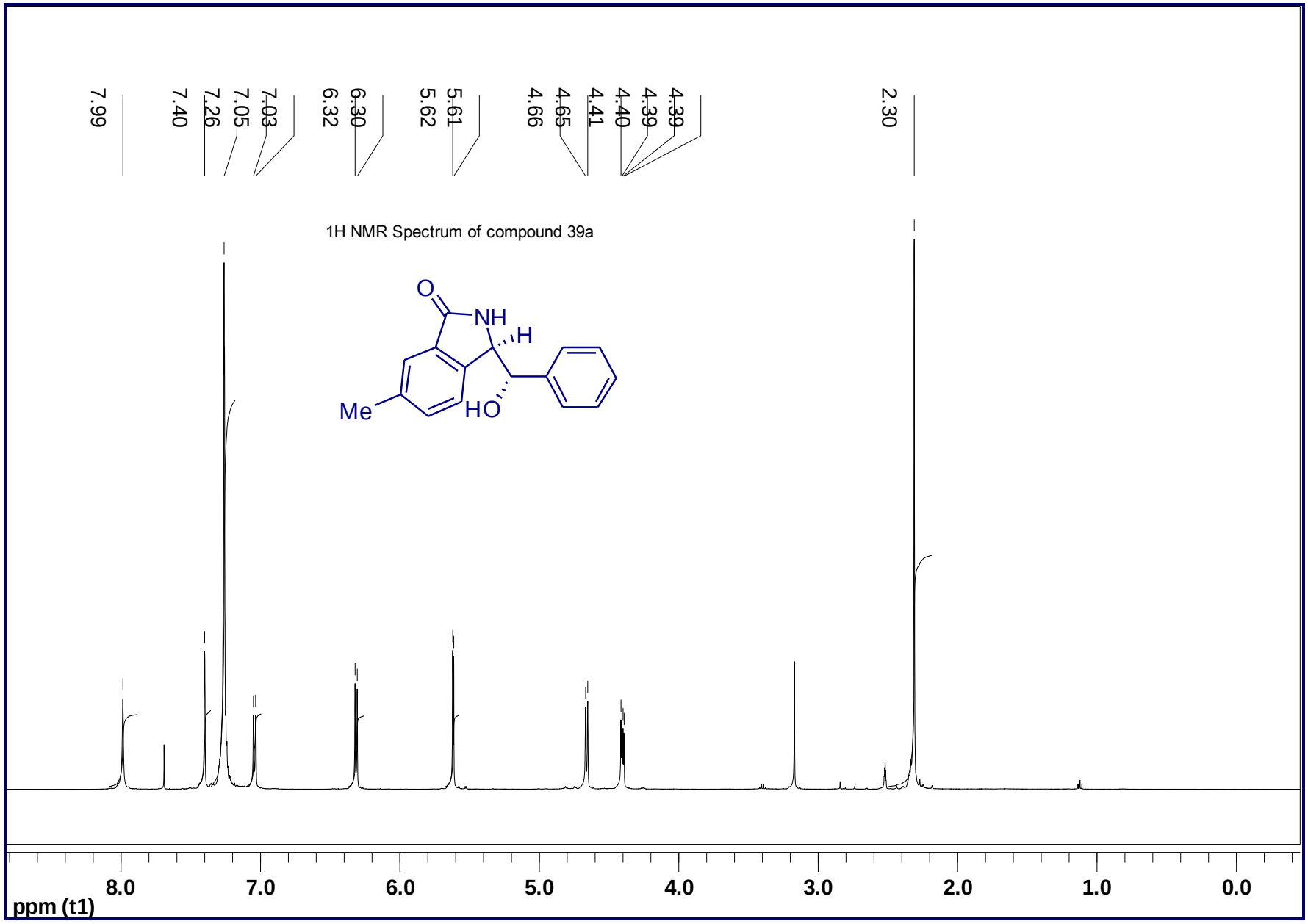

73.57
65.51
Expansion - 13C NMR Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
75.0
70.0
65.0
ppm (t1)


36.29
25.42
24.62
23.28
21.28
14.50
Expansion - 13C NMR Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
35.0
30.0
25.0
20.0
15.0
ppm (t1)

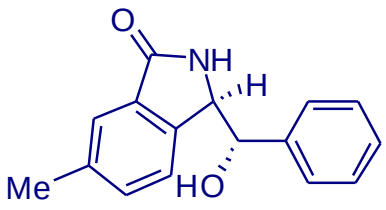

132.38
125.05
123.11
65.51
36.23
25.42
24.64
23.29
21.28
14.52
13C NMR DEPT-90 Spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete.
100
50
0
ppm (t1)


132.38
125.05
123.11
65.51
36.22
25.42
24.64
23.29
21.28
14.52
13C NMR DEPT-135 spectrum spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
100
50
0
ppm (t1)

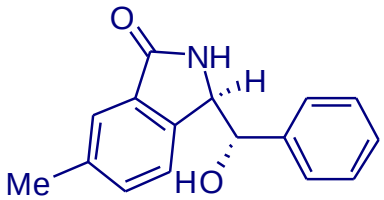

36.22
25.42
24.64
23.29
21.28
14.52
Expansion - 13C NMR DEPT-135 spectrum spectrum of compound 38a
O
H
N
H
Me
HO
Bu
Me
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
35.0
30.0
25.0
20.0
15.0
ppm (t1)

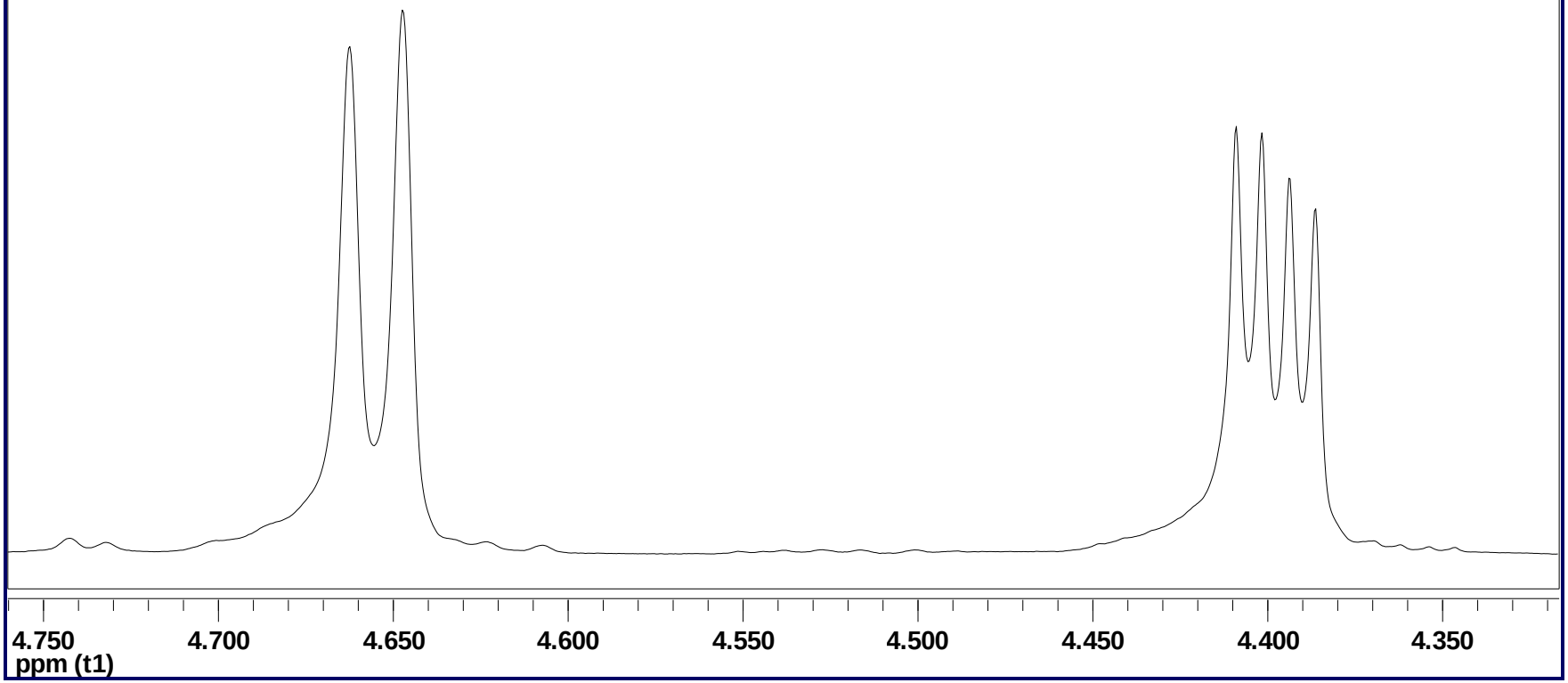

7.99
7.40
7.26
7.05
7.03
6.32
6.30
5.62
5.61
4.66
4.65
4.41
4.40
4.39
4.39
2.30
1H NMR Spectrum of compound 39a
O
NH
H
Me
HO
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

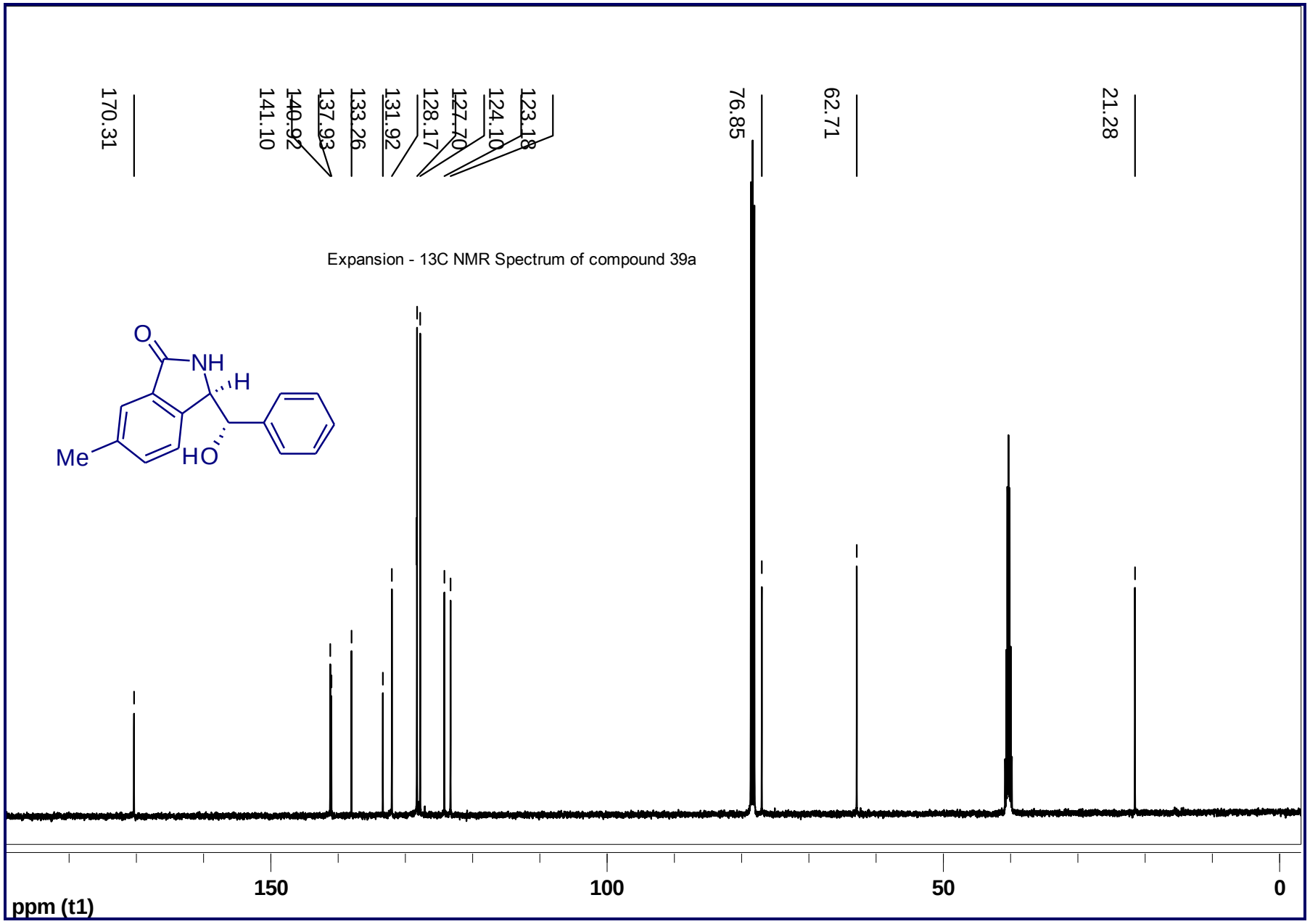
Expansion - 1H NMR Spectrum of compound 39a

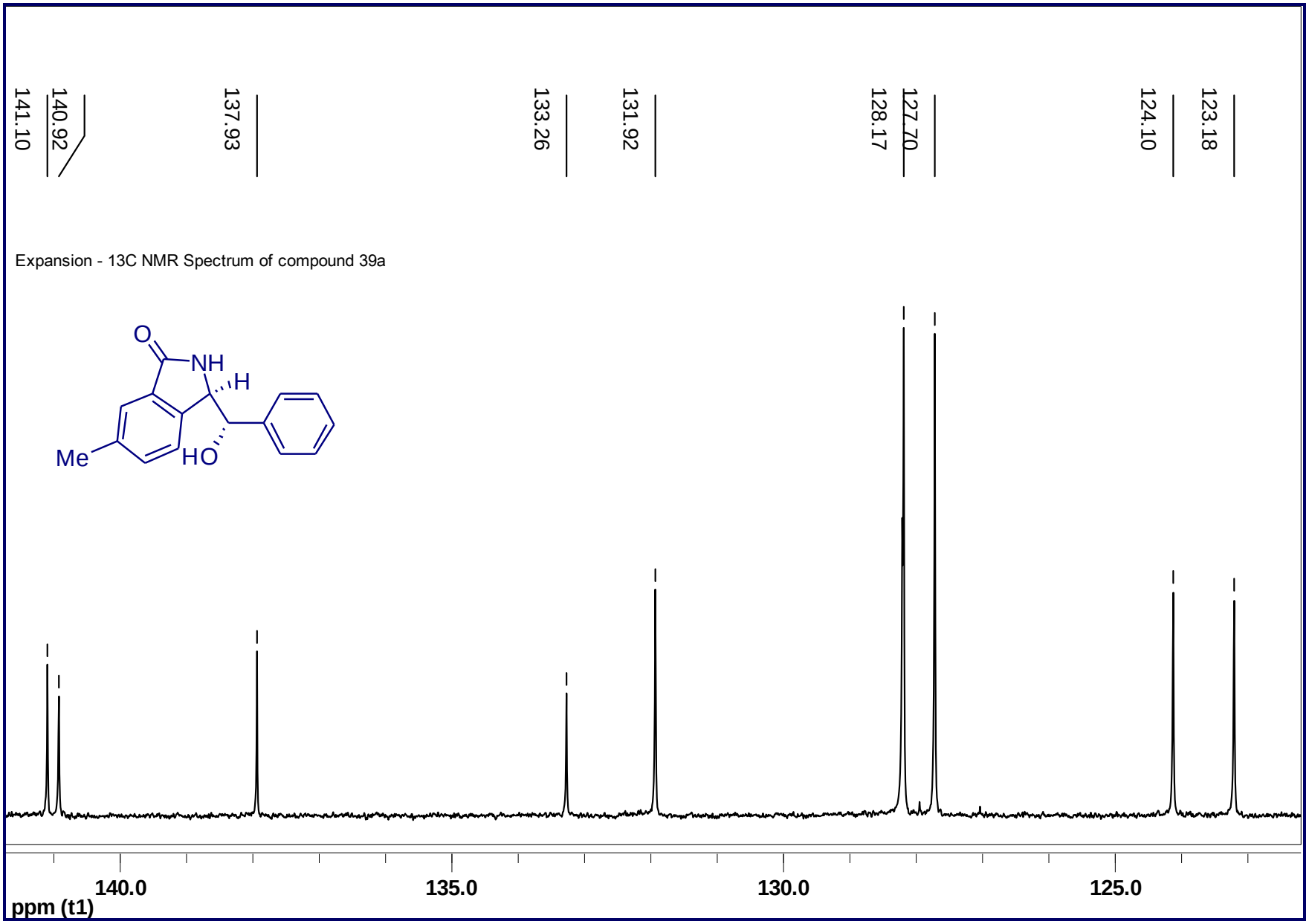


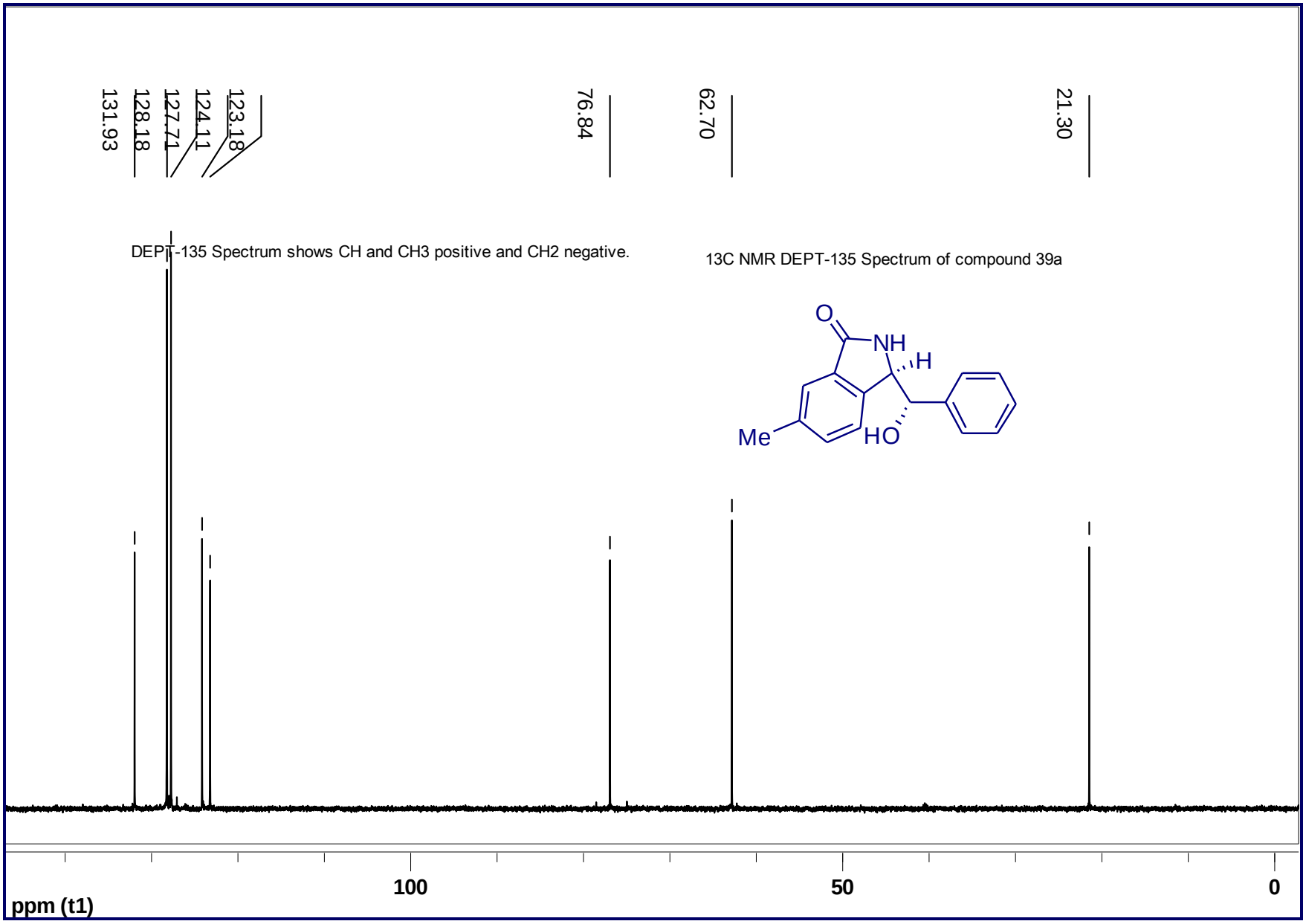
Expansion - 1H NMR Spectrum of compound 39a

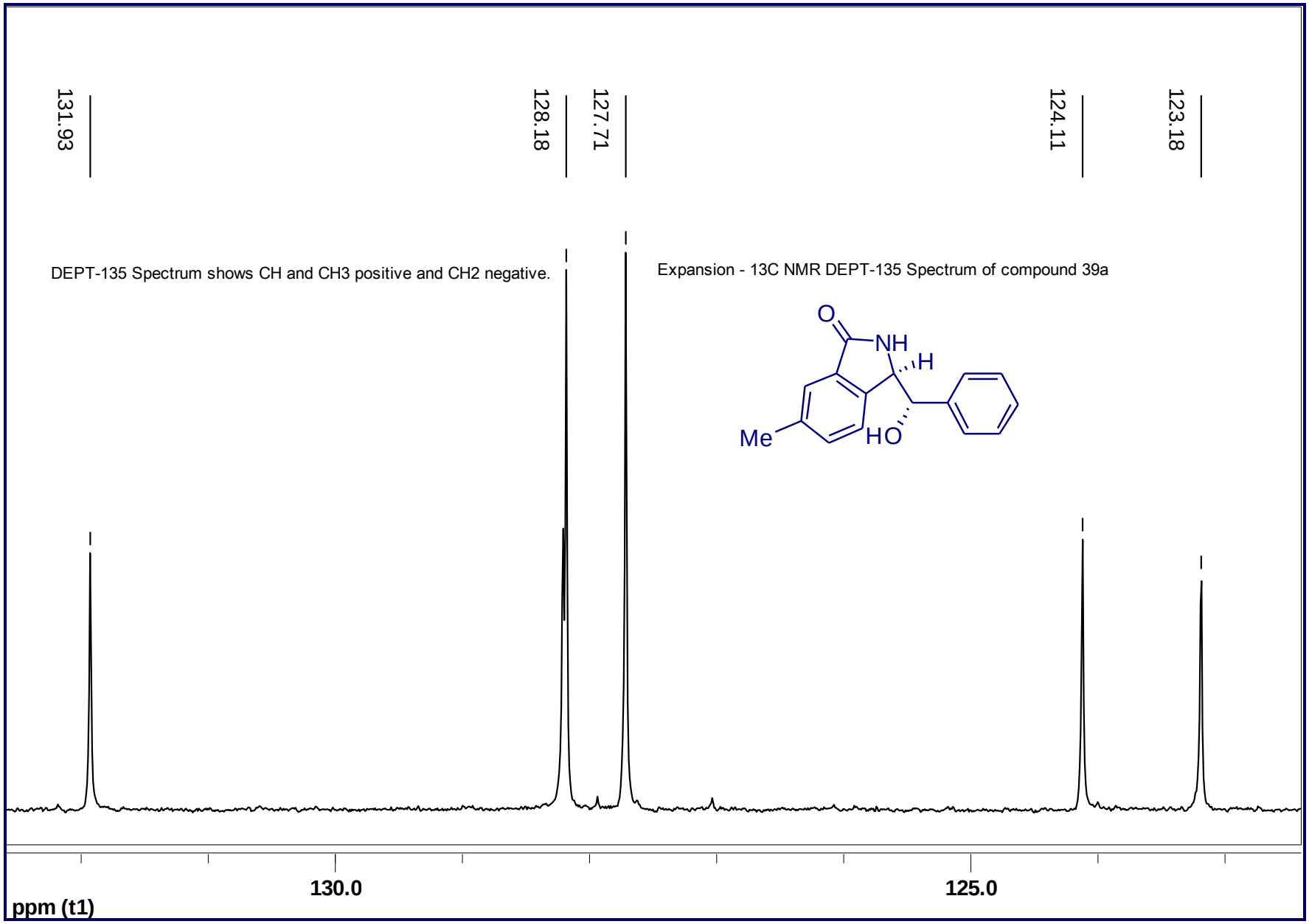


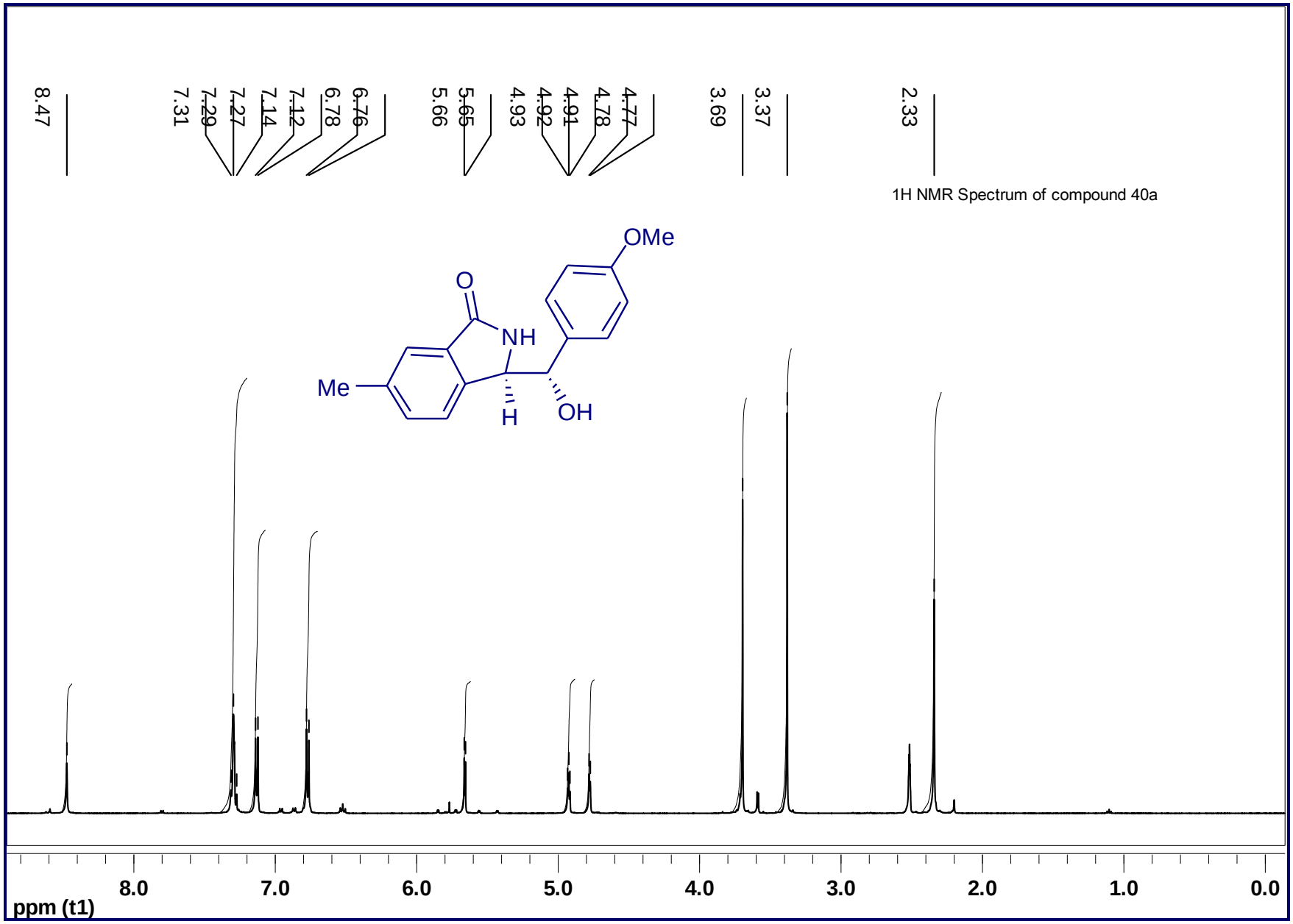


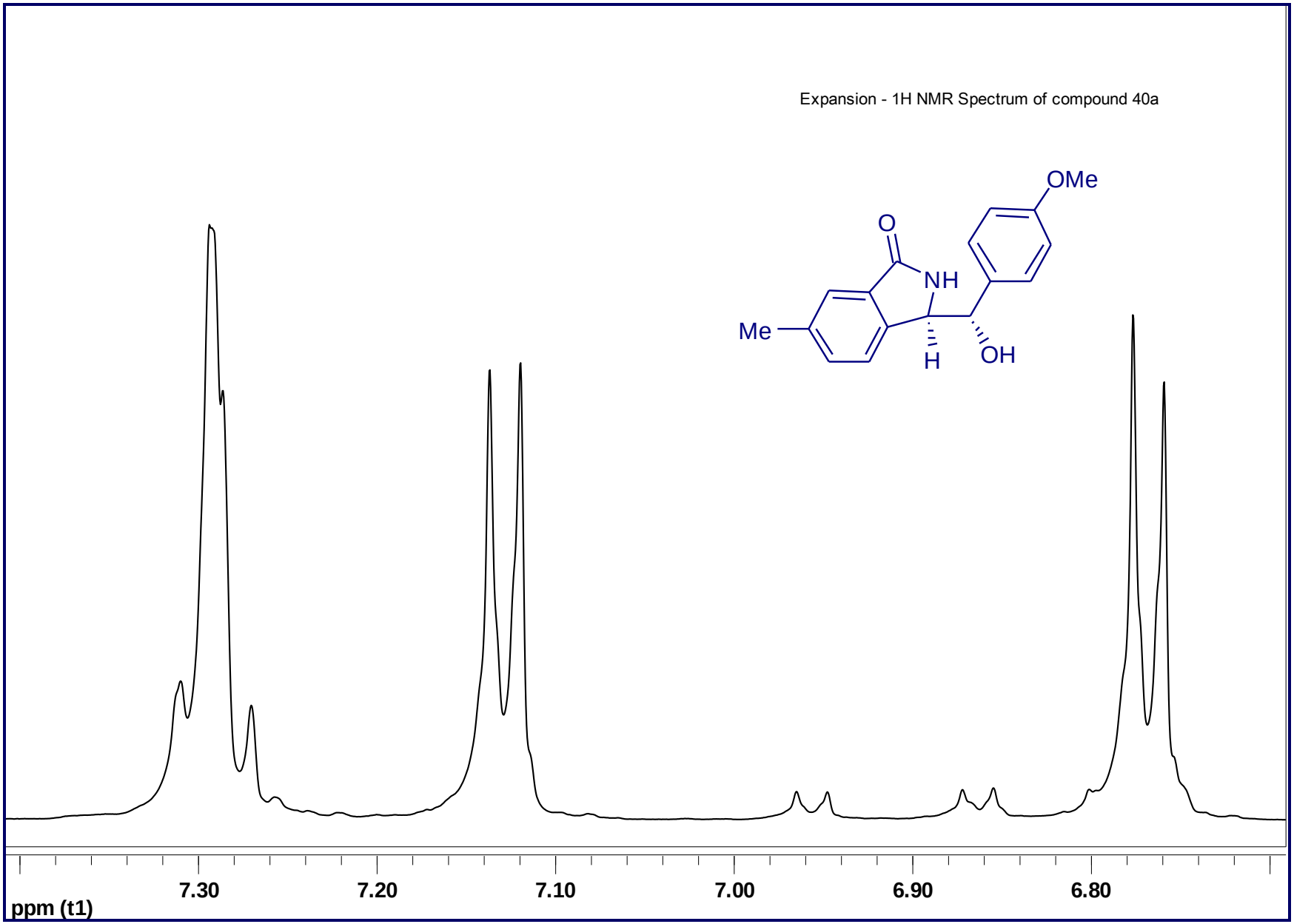

170.31
141.10
140.92
137.93
133.26
131.92
128.17
127.70
124.10
123.18
76.85
62.71
21.28
Expansion - 13C NMR Spectrum of compound 39a
O
NH
H
Me
HO
150
100
50
0
ppm (t1)


141.10
140.92
137.93
133.26
131.92
128.17
127.70
124.10
123.18
Expansion - 13C NMR Spectrum of compound 39a
O
NH
H
Me
HO
140.0
135.0
130.0
125.0
ppm (t1)

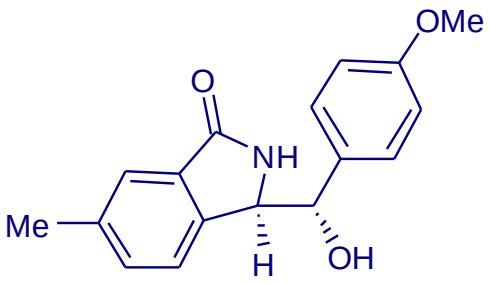

131.93
128.18
127.71
124.11
123.18
76.84
62.70
21.30
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
13C NMR DEPT-135 Spectrum of compound 39a
O
NH
H
Me
HO
100
50
0
ppm (t1)

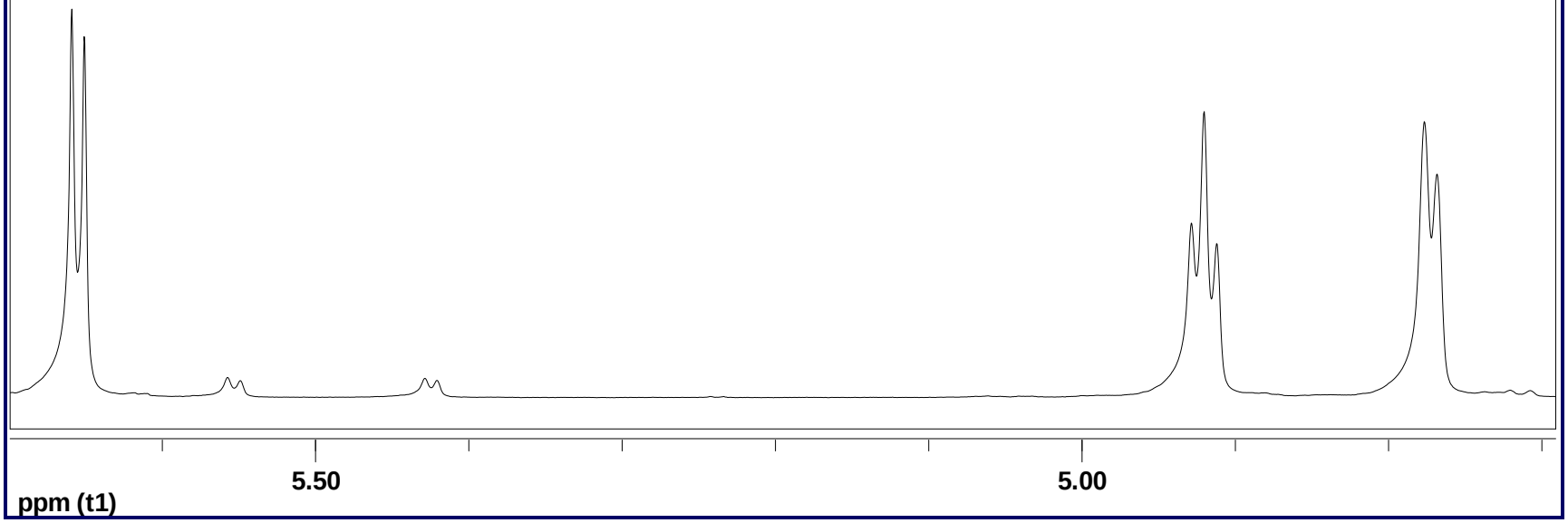

131.93
128.18
127.71
124.11
123.18
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
Expansion - 13C NMR DEPT-135 Spectrum of compound 39a
O
NH
H
Me
HO
130.0
125.0
ppm (t1)

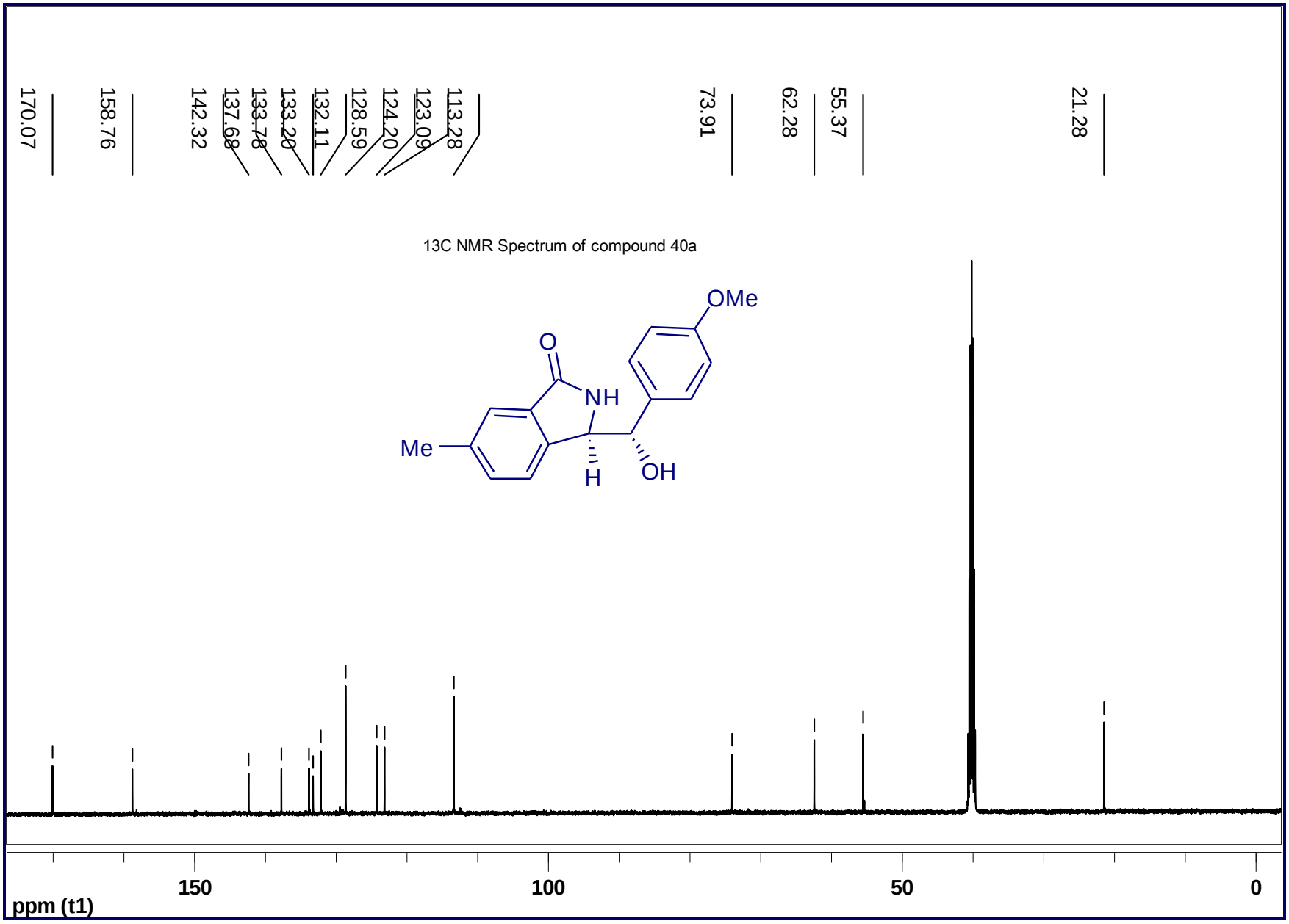

8.47
7.31
7.29
7.27
7.14
7.12
6.78
6.76
5.66
5.65
4.93
4.92
4.91
4.78
4.77
3.69
3.37
2.33
1H NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

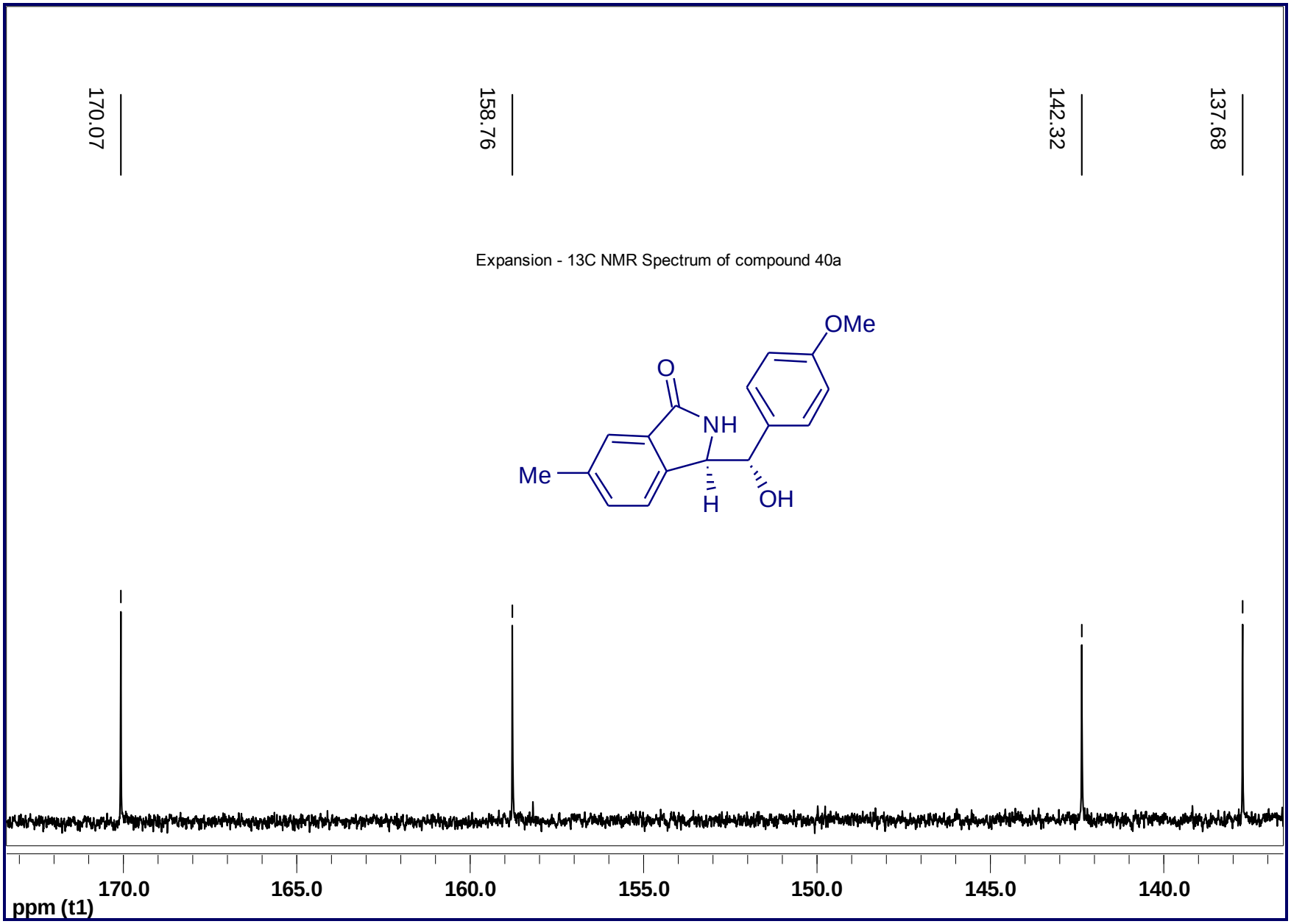

Expansion - 1H NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
7.30
7.20
7.10
7.00
6.90
6.80
ppm (t1)

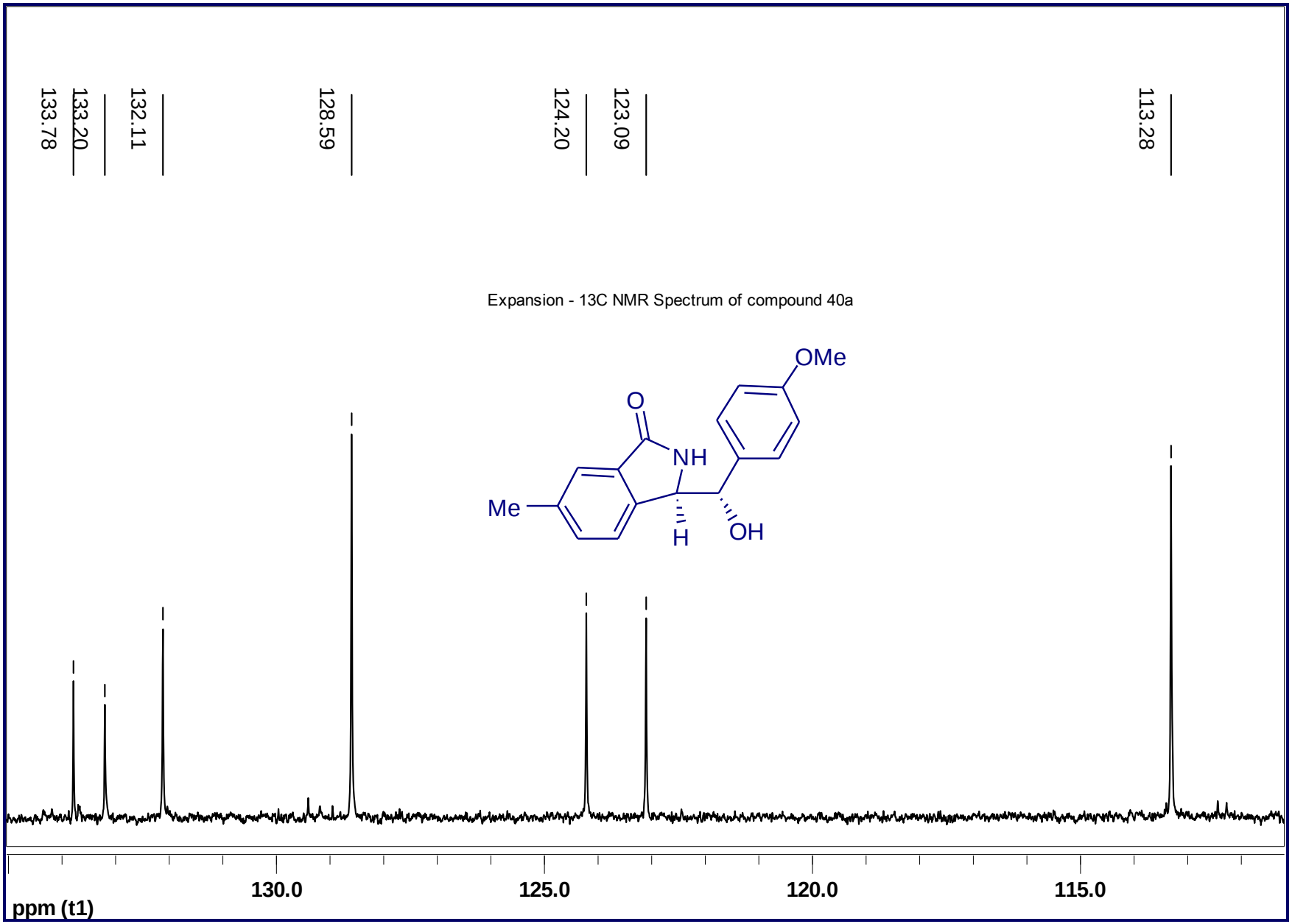
Expansion - 1H NMR Spectrum of compound 40a

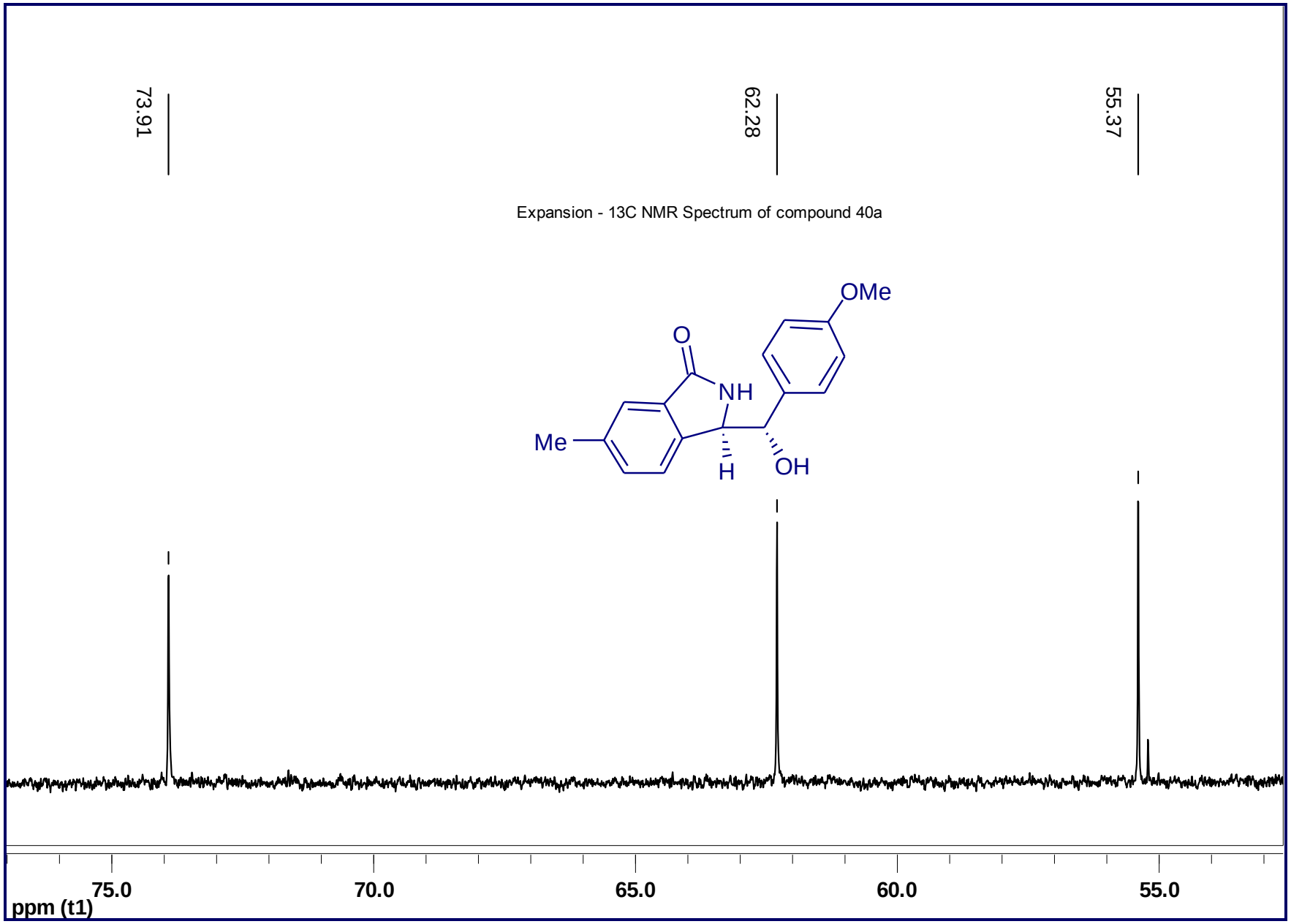


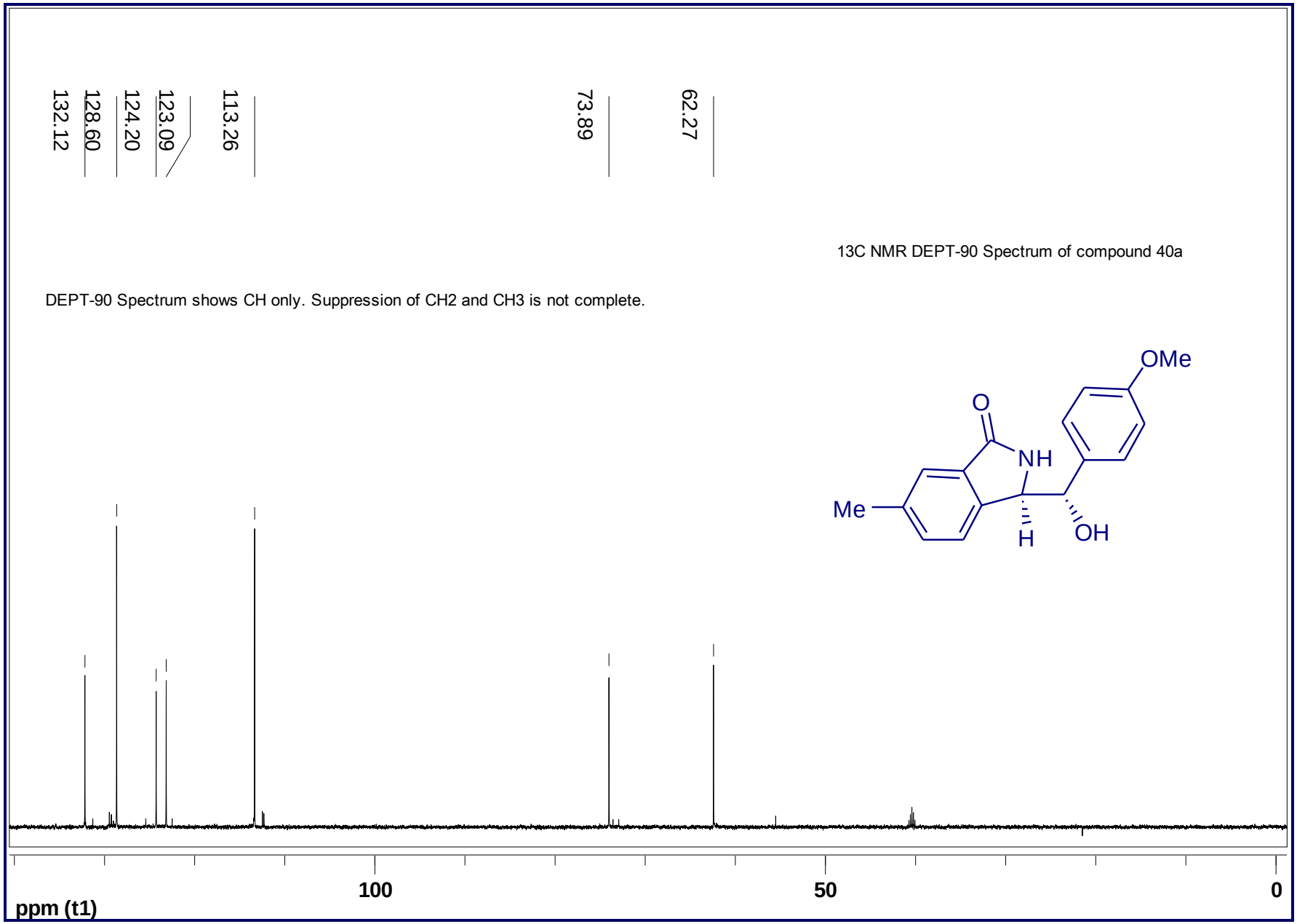


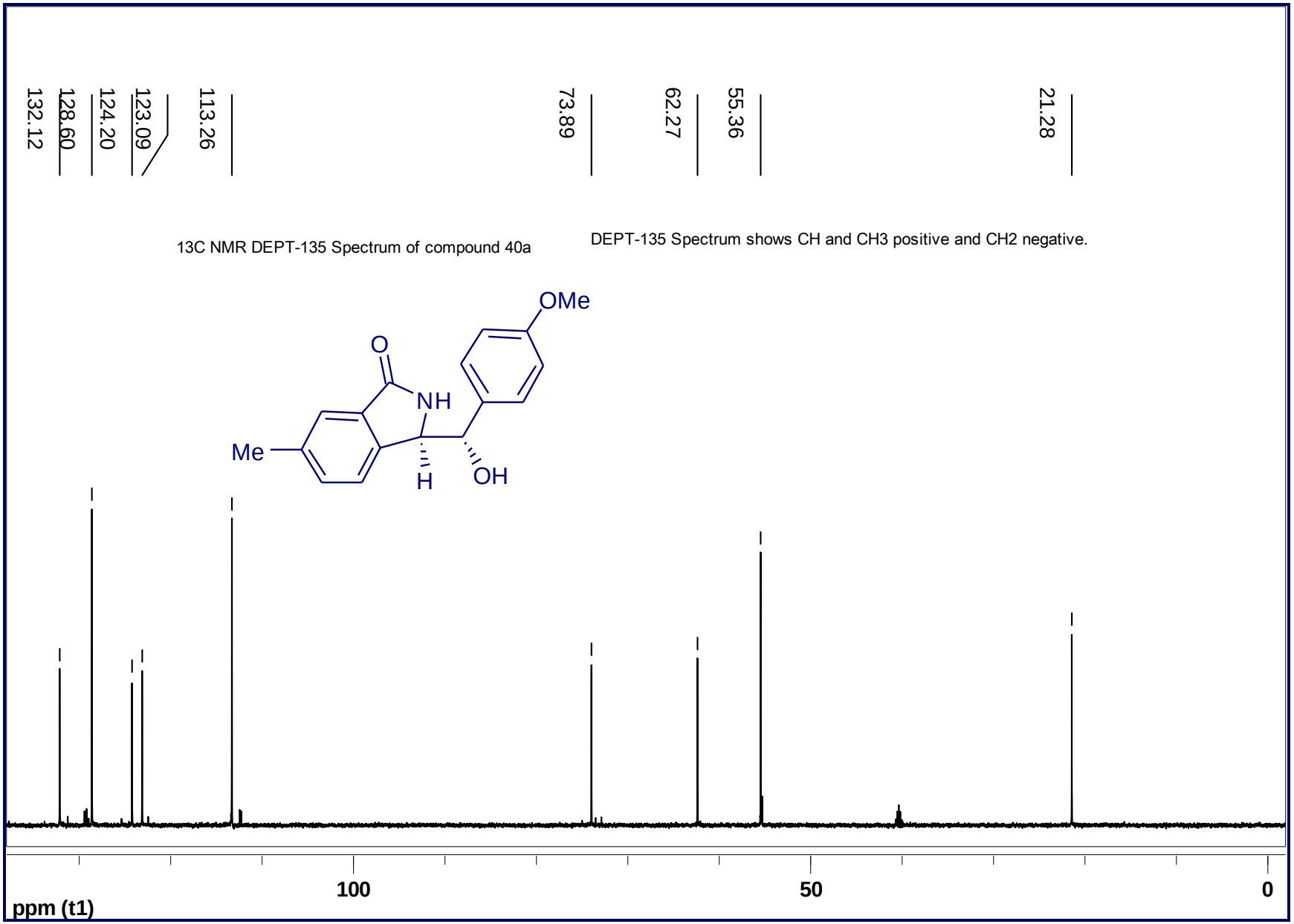

170.07
158.76
142.32
137.68
133.78
133.20
132.11
128.59
124.20
123.09
113.28
73.91
62.28
55.37
21.28
13C NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
150
100
50
0
ppm (t1)

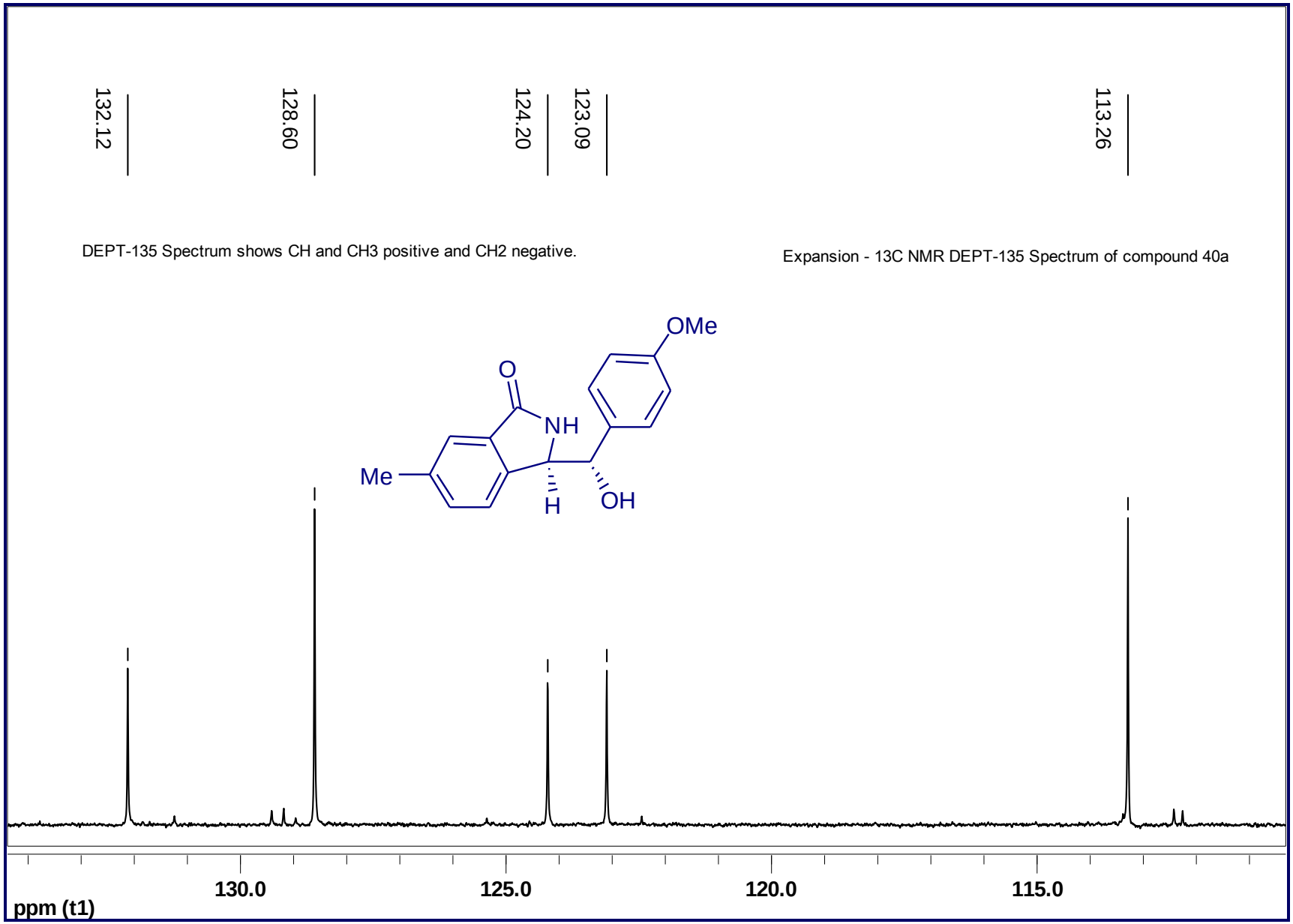

170.07
158.76
142.32
137.68
Expansion - 13C NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
170.0
165.0
160.0
155.0
150.0
145.0
140.0
ppm (t1)

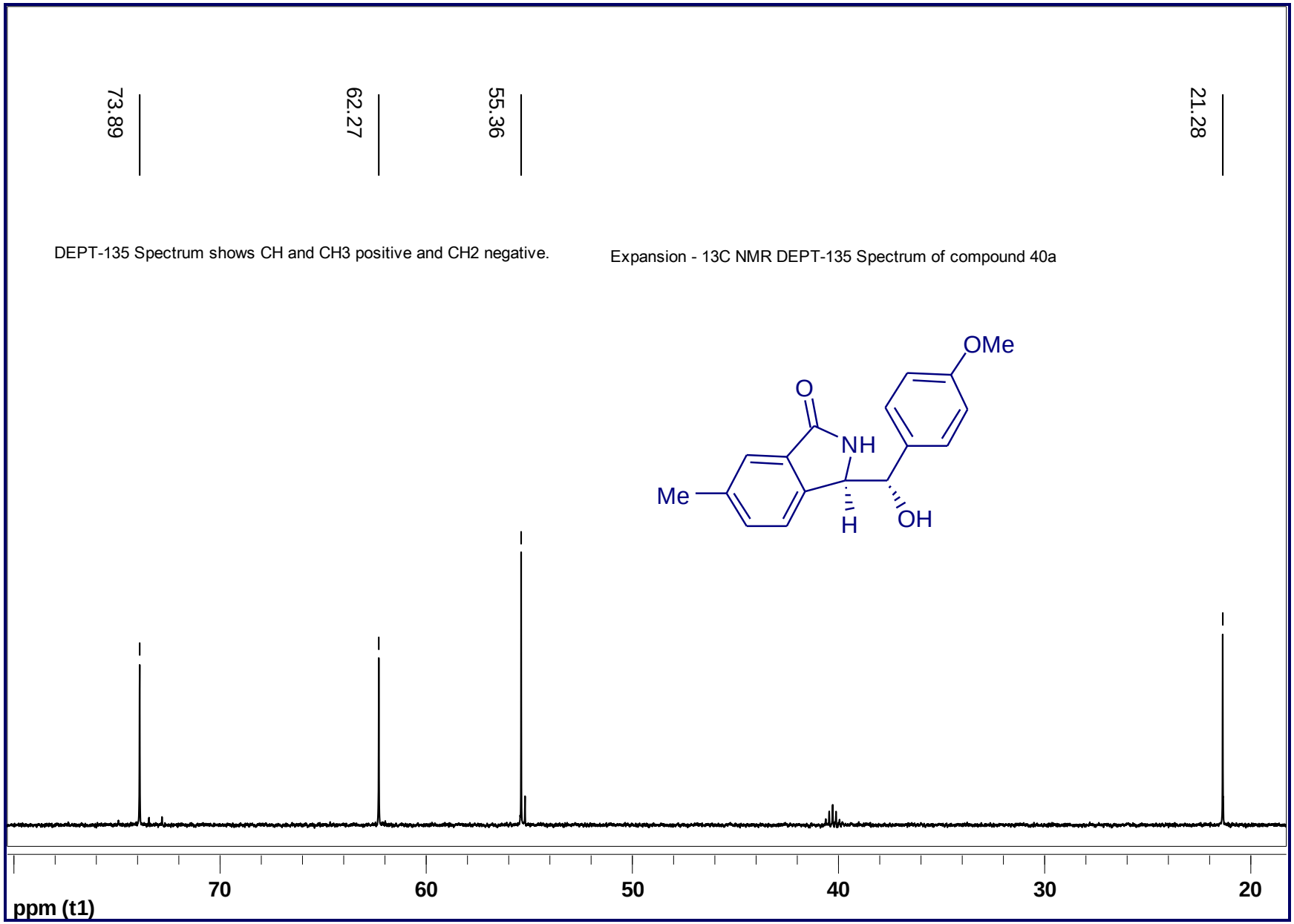

133.78
133.20
132.11
128.59
124.20
123.09
113.28
Expansion - 13C NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
130.0
125.0
120.0
115.0
ppm (t1)

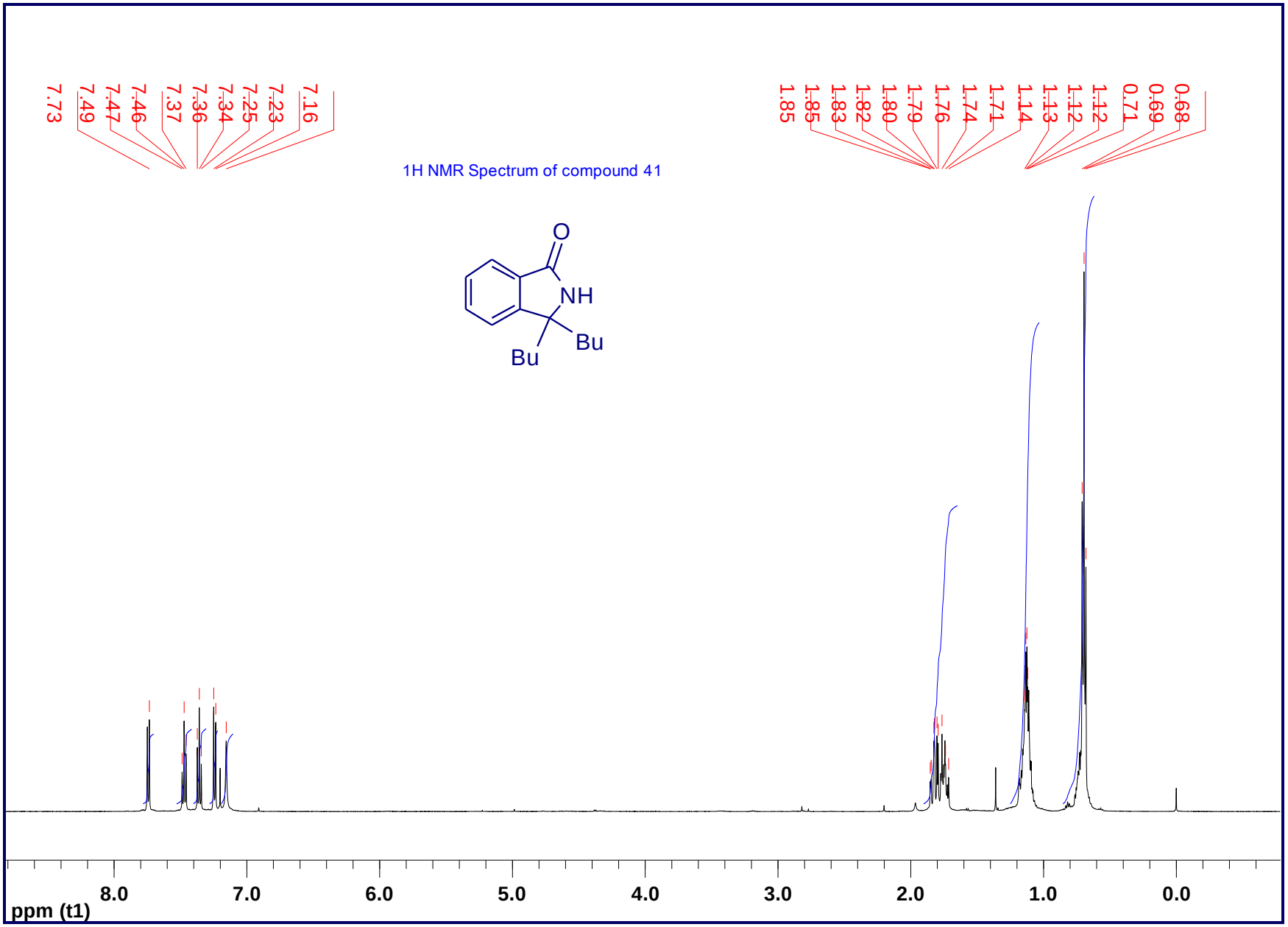

73.91
62.28
55.37
Expansion - 13C NMR Spectrum of compound 40a
OMe
O
NH
Me
H
OH
75.0
70.0
65.0
60.0
55.0
ppm (t1)


132.12
128.60
124.20
123.09
113.26
73.89
62.27
13C NMR DEPT-90 Spectrum of compound 40a
DEPT-90 Spectrum shows CH only. Suppression of CH2 and CH3 is not complete.
OMe
O
NH
Me
H
OH
100
50
0
ppm (t1)

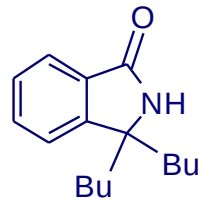

132.12
128.60
124.20
123.09
113.26
73.89
62.27
55.36
21.28
13C NMR DEPT-135 Spectrum of compound 40a
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
OMe
O
NH
Me
H
OH
100
50
0
ppm (t1)

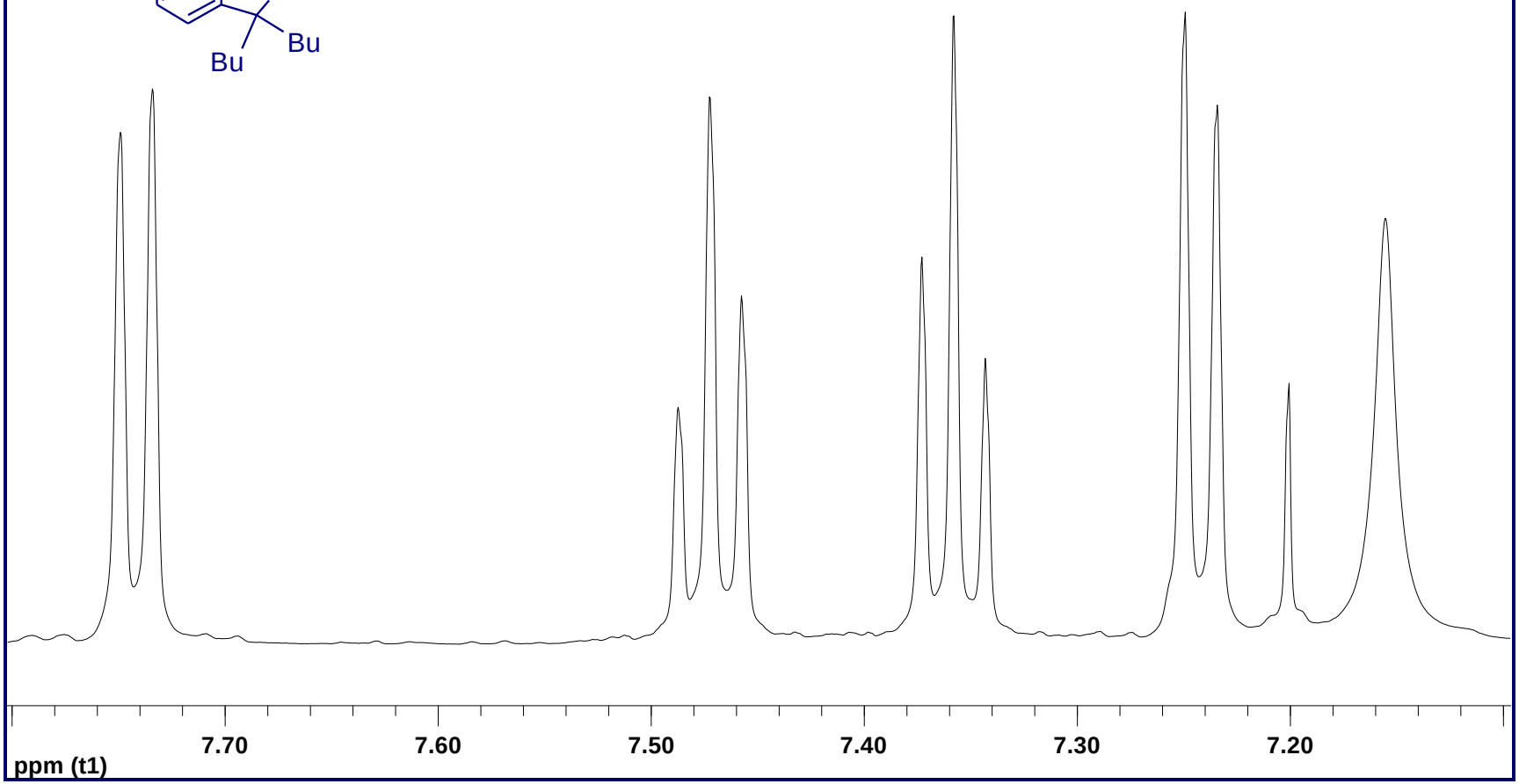

132.12
128.60
124.20
123.09
113.26
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
Expansion - 13C NMR DEPT-135 Spectrum of compound 40a
OMe
O
NH
Me
H
OH
130.0
125.0
120.0
115.0
ppm (t1)

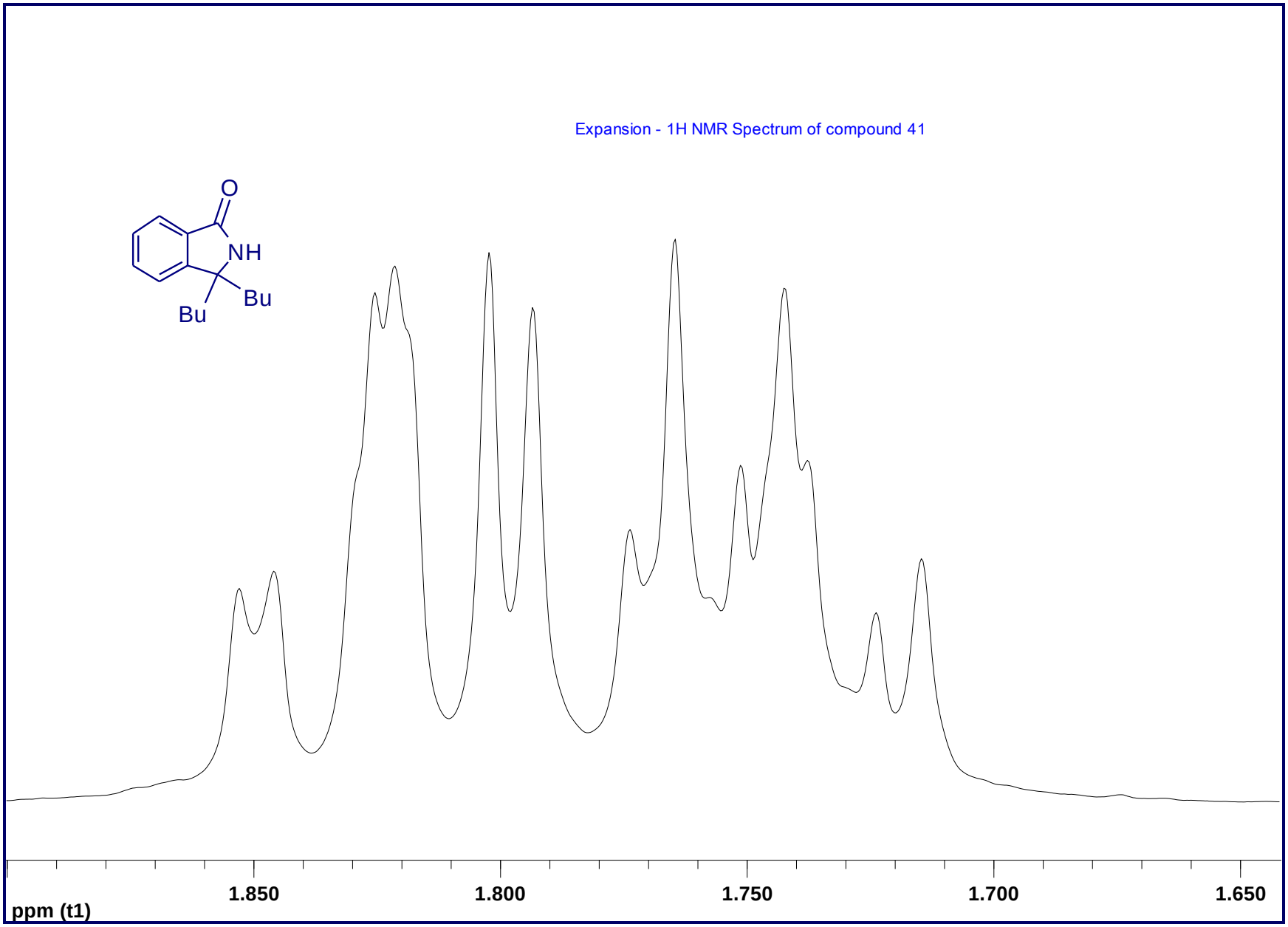

73.89
62.27
55.36
21.28
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
Expansion - 13C NMR DEPT-135 Spectrum of compound 40a
OMe
O
NH
Me
H
OH
70
60
50
40
30
20
ppm (t1)


7.73
7.49
7.47
7.46
7.37
7.36
7.34
7.25
7.23
7.16
1.85
1.85
1.83
1.82
1.80
1.79
1.76
1.74
1.71
1.14
1.13
1.12
1.12
0.71
0.69
0.68
1H NMR Spectrum of compound 41
O
NH
Bu
Bu
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

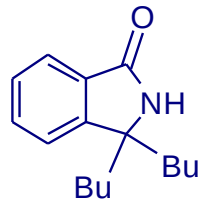
Expansion - 1H NMR Spectrum of compound 41

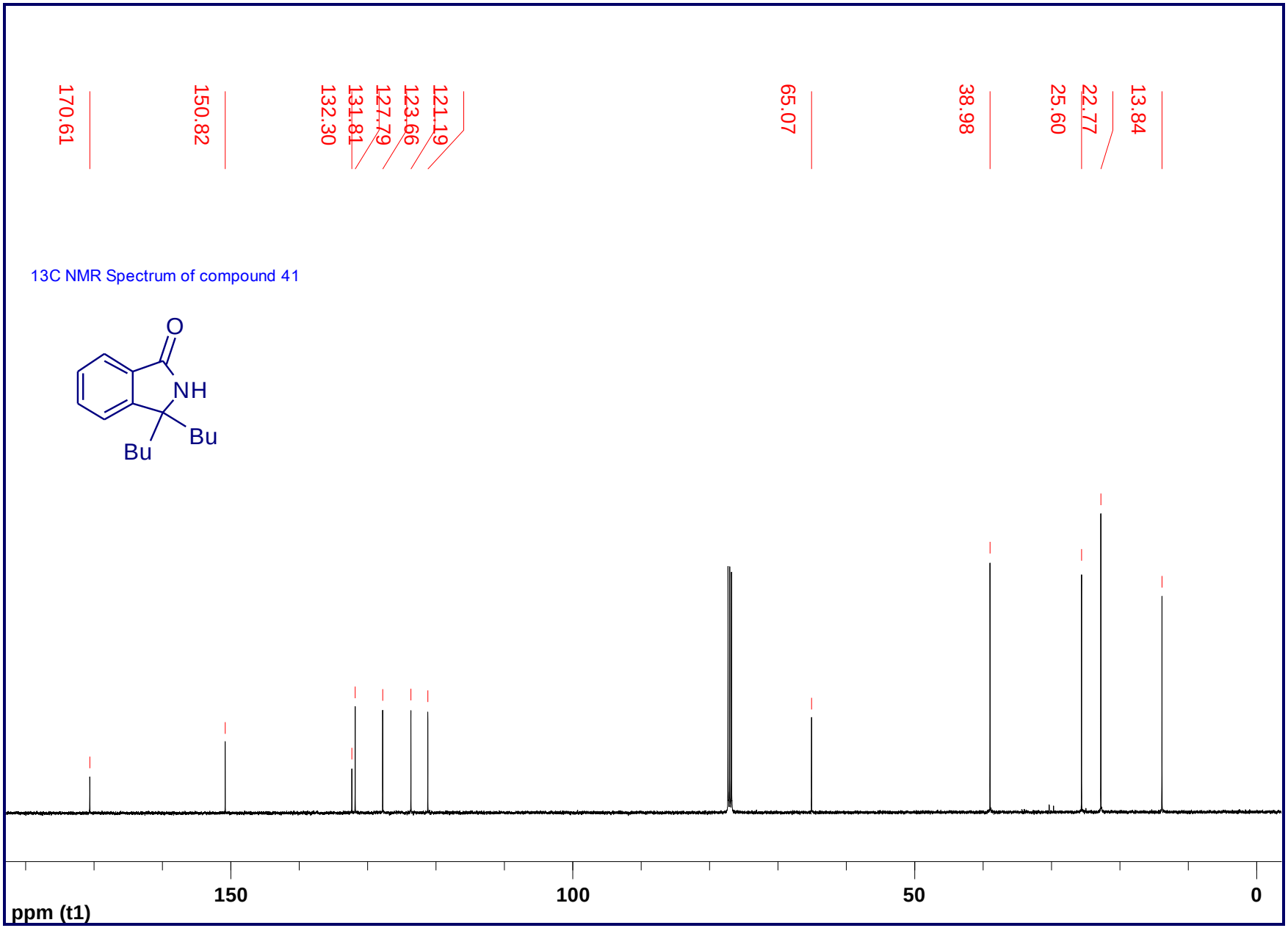


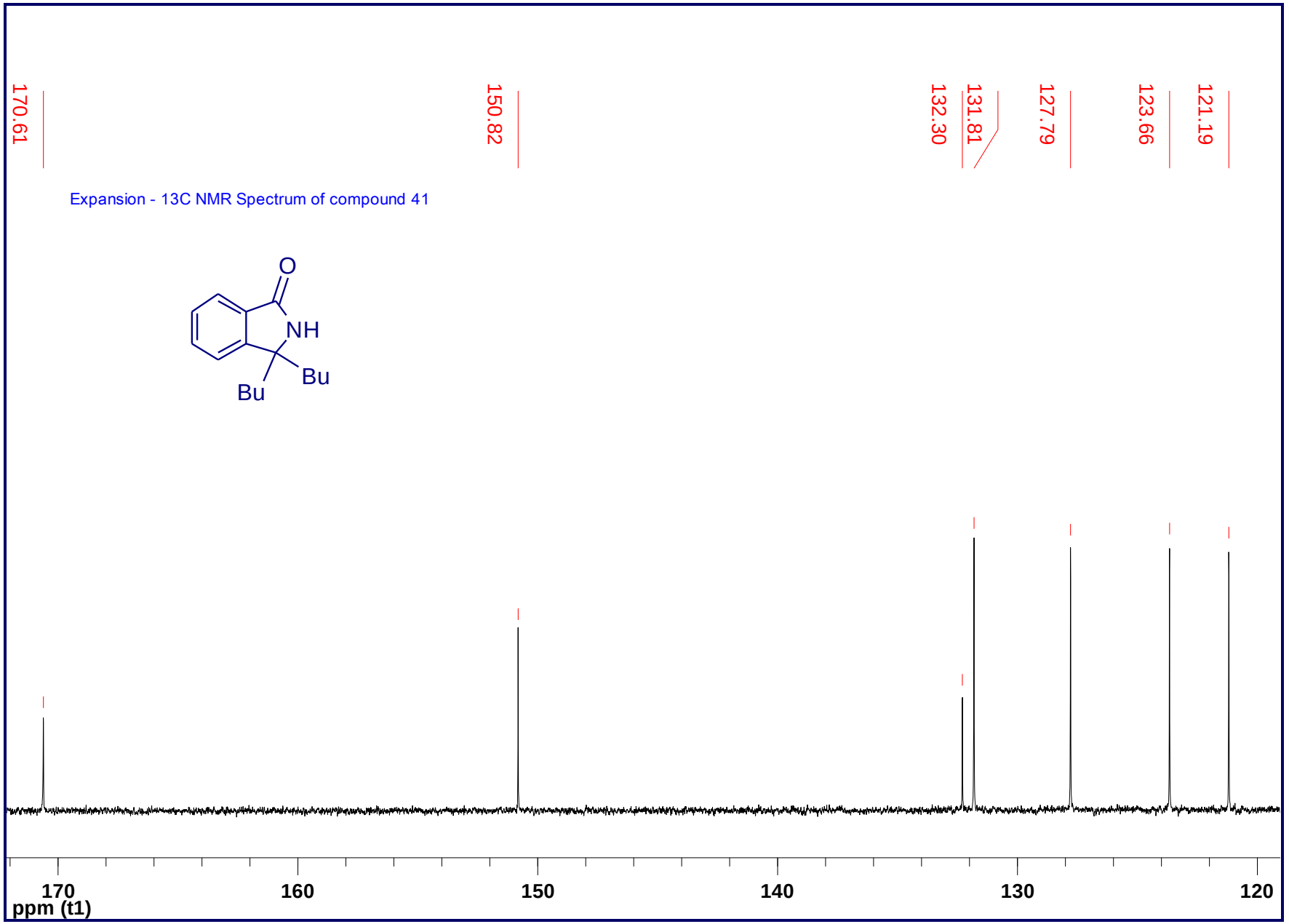


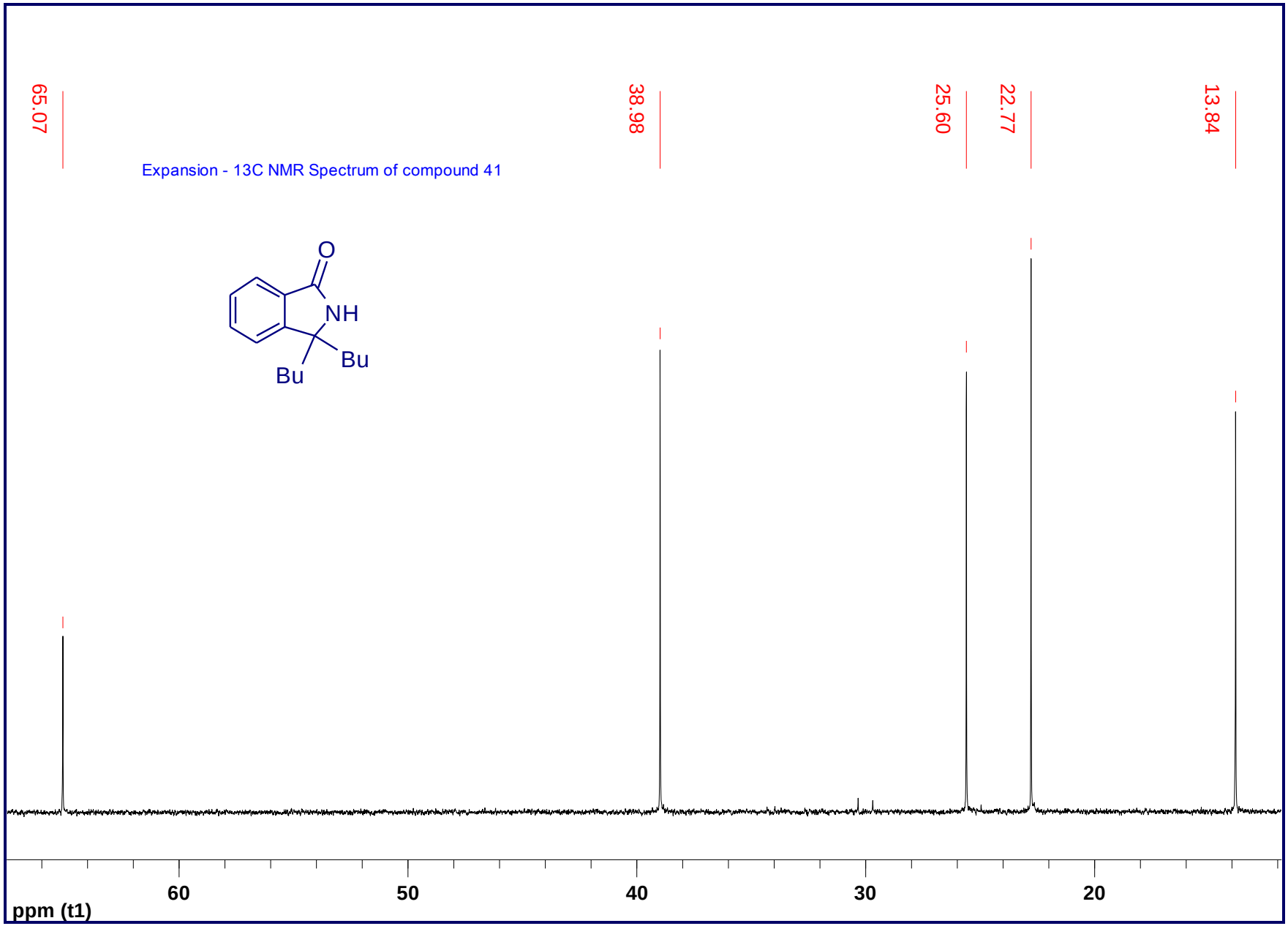

Expansion - 1H NMR Spectrum of compound 41
O
NH
Bu
Bu
1.850
1.800
1.750
1.700
1.650
ppm (t1)

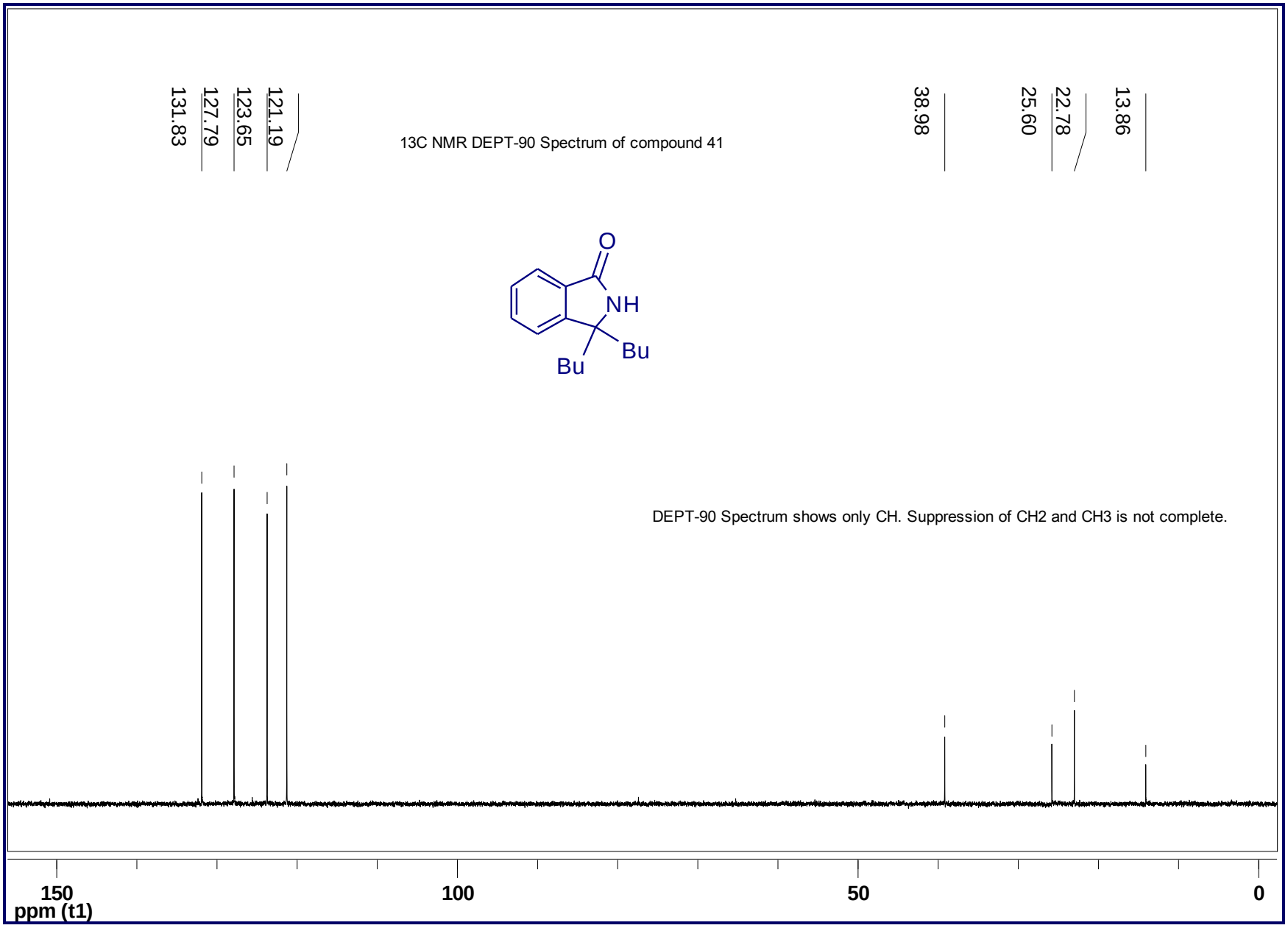
Expansion - 1H NMR Spectrum of compound 41

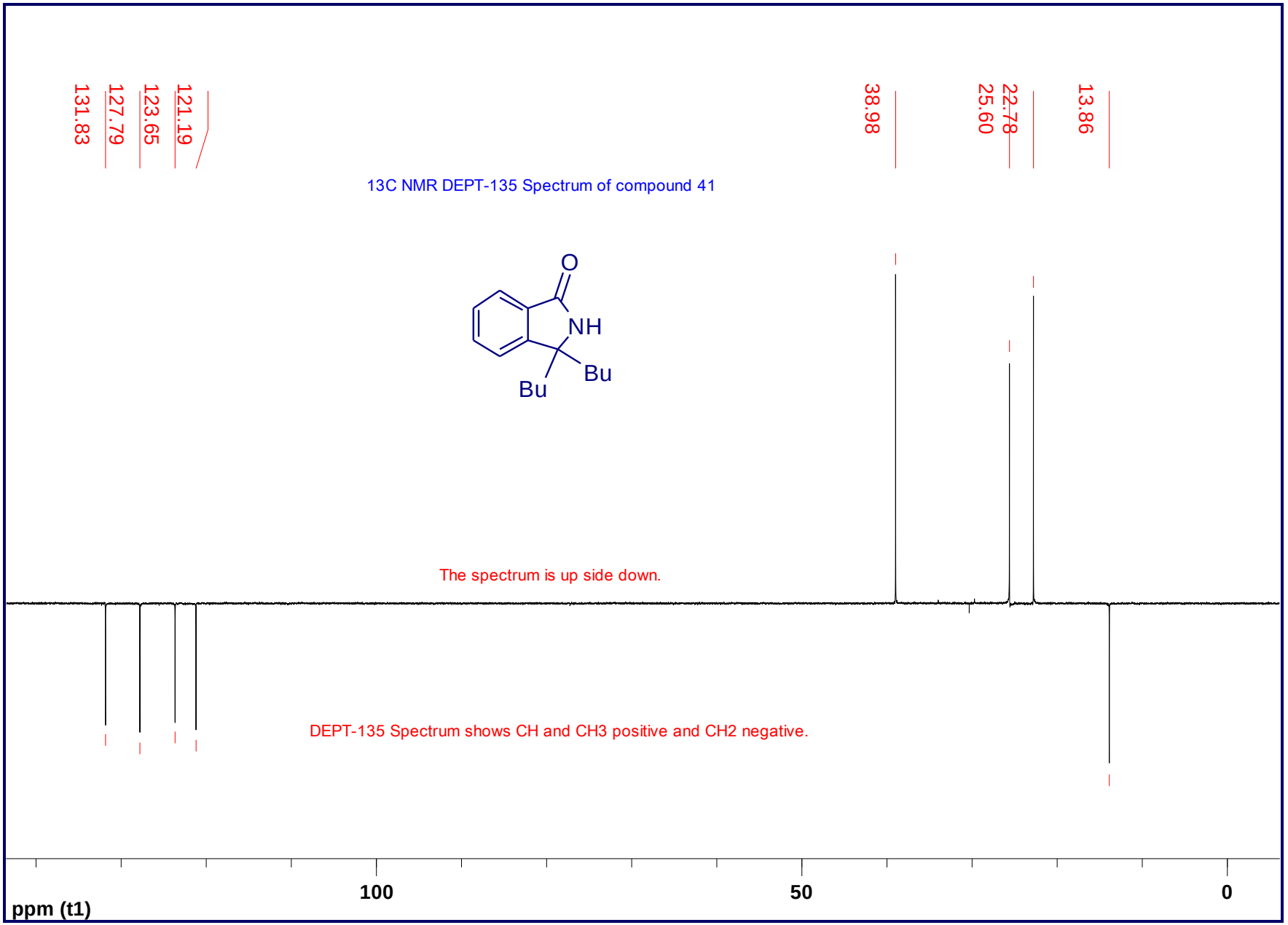



170.61
150.82
132.30
131.81
127.79
123.66
121.19
65.07
38.98
25.60
22.77
13.84
13C NMR Spectrum of compound 41
O
NH
Bu
Bu
150
100
50
0
ppm (t1)

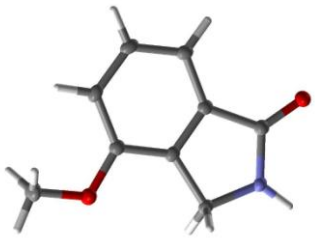
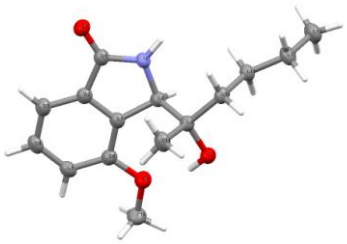

170.61
150.82
132.30
131.81
127.79
123.66
121.19
Expansion - 13C NMR Spectrum of compound 41
O
NH
Bu
Bu
170
160
150
140
130
120
ppm (t1)


65.07
38.98
25.60
22.77
13.84
Expansion - 13C NMR Spectrum of compound 41
O
NH
Bu
Bu
60
50
40
30
20
ppm (t1)

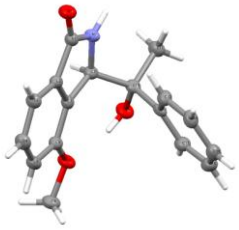
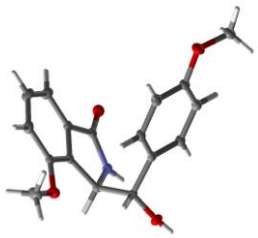
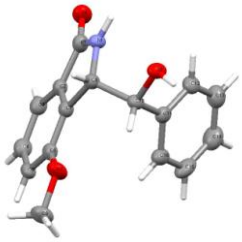

131.83
127.79
123.65
121.19
38.98
25.60
22.78
13.86
13C NMR DEPT-90 Spectrum of compound 41
O
NH
Bu
Bu
DEPT-90 Spectrum shows only CH. Suppression of CH2 and CH3 is not complete.
150
100
50
0
ppm (t1)


131.83
127.79
123.65
121.19
38.98
25.60
22.78
13.86
13C NMR DEPT-135 Spectrum of compound 41
O
NH
Bu
Bu
The spectrum is up side down.
DEPT-135 Spectrum shows CH and CH3 positive and CH2 negative.
100
50
0
ppm (t1)

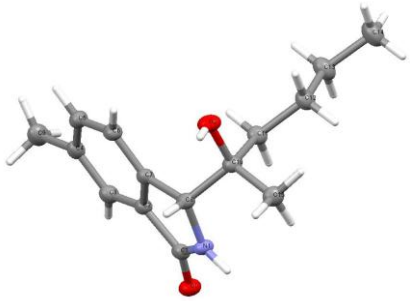
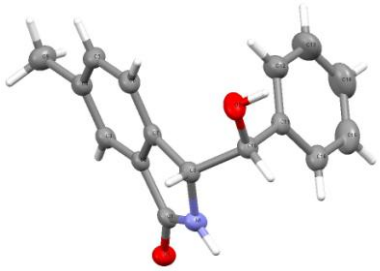
Crystal and Structure Refinement Data of Compounds 12 and 15

Structure		
	12	**15**
CCDC reference	737411	762624
Empirical formula of crystal unit	$C_9H_9NO_2$	$C_{15}H_{21}NO_3$
Formula weight of crystal unit	163.17	263.33
Temperature/K	150(2)	150(2)
Crystal system	Triclinic	Monoclinic
Space group	P-1	P21/a
a/Å	7.2640(5)	9.7517(5)
b/Å	7.8940(6)	13.5852(7)
c/Å	8.3280(8)	11.0154(4)
α/°	104.845(3)	90
β/°	114.462(4)	95.266(3)
γ/°	103.622(5)	90
V/Å^3	387.21(5)	1453.15(12)
Z	2	4
ρ_{cal}/Mgm^{-3}	1.400	1.204
μ/mm^{-1}	0.100	0.083
Crystal size/mm^3	0.25 x 0.24 x 0.18	0.30 x 0.06 x 0.06
Reflections collected	2379	5380
Independent	1714	3240
R(int)	0.0301	0.0661
Parameters	110	176
R1	0.0512	0.0725
wR2	0.1220	0.1445

Crystal and Structure Refinement Data of Compounds 16-18

Structure			
	16	**17**	**18**
CCDC reference	766180	737415	762623
Empirical formula of crystal unit	$C_{17}H_{17}NO_3$	$C_{17}H_{17}NO_4$	$C_{18}H_{19}NO_4$
Formula weight of crystal unit	283.32	299.32	313.34
Temperature/K	150(2)	293(2)	150(2)
Crystal system	Triclinic	Centrosymmetric	Triclinic
Space group	P-1	P-1	P-1
a/Å	8.9567(5)	7.5240(3)	7.5698(6)
b/Å	9.0050(3)	9.4100(3)	9.2785(8)
c/Å	9.3788(4)	11.9960(5)	12.2934(10)
α/°	67.768(3)	83.877(2)	78.473(4)
β/°	89.017(3)	77.311(2)	82.365(4)
γ/°	81.593(3)	68.559(2)	70.596(3)
V/Å^3	692.09(5)	770.92(5)	795.91(11)
Z	2	2	2
ρ_{cal}/Mgm^{-3}	1.360	1.289	1.307
μ/mm^{-1}	0.093	0.092	0.093
Crystal size/mm^3	0.20 x 0.10 x 0.10	0.20 x 0.15 x 0.15	0.30 x 0.30 x 0.10
Reflections collected	4270	5071	4991
Independent	3038	3513	3467
R(int)	0.0345	0.0334	0.0536
Parameters	194	202	238
R1	0.0511	0.0570	0.0774
wR2	0.1271	0.1277	0.1657

Crystal and Structure Refinement Data of Compounds 38 and 39

Structure		
	38	**39**
CCDC reference	766182	766181
Empirical formula of crystal unit	$C_{36}H_{56}N_2O_4$	$C_{16}H_{15}NO_2$
Formula weight of crystal unit	580.83	253.29
Temperature/K	150(2)	150(2)
Crystal system	Triclinic	Monoclinic
Space group	P-1	C2/c
a/Å	7.1881(4)	23.4249(10)
b/Å	10.5950(4)	7.8424(3)
c/Å	12.5474(5)	15.7782(4)
α/°	112.175(2)	90
β/°	96.371(2)	113.612(2)
γ/°	101.774(2)	90
V/Å^3	847.46(7)	2655.90(17)
Z	1	8
ρ_{cal}/Mgm^{-3}	1.138	1.267
μ/mm^{-1}	0.073	0.084
Crystal size/mm^3	0.40 x 0.30 x 0.02	0.15 x 0.15 x 0.10
Reflections collected	5995	8198
Independent	3840	3039
R(int)	0.0434	0.0544
Parameters	195	174
R1	0.0656	0.0504
wR2	0.1716	0.1337