Supporting Information

From intramolecular cyclization to intermolecular hydrolysis: $TMSCF_2Br$-enabled carbonylation of aldehydes/ketones and amines to α-hydroxyamides

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1 General information

Unless otherwise mentioned, all solvents and reagents were purchased from commercial sources. Tetrahydrofuran (THF), 1,4-dioxacyclohexane (1,4-Dioxane) and *N,N*-dimethylformamide (DMF) were used directly from a solvent purification system. All the melting points were uncorrected. NMR spectra were recorded on 400 or 600 MHz NMR spectrometer for ^{1}H, ^{19}F, and ^{13}C acquisitions. ^{1}H NMR chemical shifts were determined relative to internal $(CH_3)_4Si$ (TMS) at δ 0.00 ppm or to the signal of the residual protonated solvent: $CDCl_3$ at δ 7.26 ppm. ^{19}F NMR chemical shifts were determined relative to internal $CFCl_3$ at δ 0.00 ppm. ^{13}C NMR chemical shifts were determined relative to the signal of the solvent: $CDCl_3$ at δ 77.16 ppm. Data for ^{1}H, ^{19}F, ^{13}C NMR were recorded as follows: chemical shifts (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiple, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, ddd = doublet of doublet of doublets, br = broad, tt = triplet of triplets). Mass spectra were recorded on a high-resolution mass spectrometer in the EI, FI, ESI or DART mode.

2 Optimization of the reaction conditions

Typical procedures (Taking entry 1 and 8 in Table 1 as examples)

Entry 1:

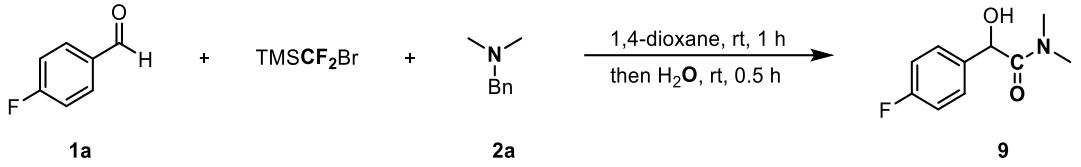
Under argon atmosphere, a solution of 4-fluorobenzaldehyde (**1a**, 0.2 mmol, 1.0 equiv.), $TMSCF_2Br$ (0.22 mmol, 1.1 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (0.22 mmol, 1.1 equiv.) in 1,4-dioxane (2 mL) was stirred at room temperature for an hour. Then *t*-BuOH (0.4 mmol, 2.0 equiv.) was added and the mixture was stirred at 100 °C for 0.5 hour. Yields were determined by ^{19}F NMR using 1-fluoronaphthalene as an internal standard.

Entry 8:

Under argon atmosphere, a solution of 4-fluorobenzaldehyde (**1a**, 0.2 mmol, 1.0 equiv.), $TMSCF_2Br$ (0.4 mmol, 2.0 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (0.4 mmol, 2.0 equiv.) in 1,4-dioxane (2 mL) was stirred at room temperature for an

hour. Then H_2O (0.2 mL) was added and the mixture was stirred at room temperature for 0.5 hour. Yields were determined by ^{19}F NMR using 1-fluoronaphthalene as an internal standard.

3 Procedures for the synthesis of α-hydroxyamides

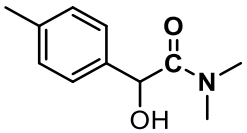


1a **2a** **9**

Typical procedures (Taking the compound **9** as an example)

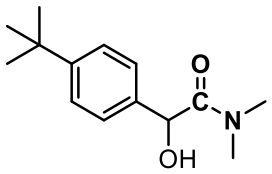
Under argon atmosphere, a solution of 4-fluorobenzaldehyde (**1a**, 0.5 mmol, 1.0 equiv.), $TMSCF_2Br$ (1.0 mmol, 2.0 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (**2a**, 1.0 mmol, 2.0 equiv.) in 1,4-dioxane (5 mL) was stirred at room temperature for an hour. Then H_2O (0.5 mL) was added and the mixture was stirred at room temperature for 0.5 hour. The mixture was quenched with water, and extracted with ethyl acetate for three times. The combined extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by flash column chromatography using petroleum ether/ethyl acetate to afford product **9**.

2-Hydroxy-*N,N*-dimethyl-2-(p-tolyl)acetamide (5)[1]



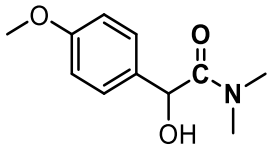
White solid (80 mg, 83% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). M.p.: 135 – 140 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18 (q, J = 8.1 Hz, 4H), 5.16 (s, 1H), 4.71 (s, 1H), 3.02 (s, 3H), 2.77 (s, 3H), 2.33 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.6, 138.4, 136.3, 129.8, 127.5, 71.4, 36.5, 36.3, 21.3; **MS** (EI, *m/z*): 193 $[M]^+$, 165, 121, 93; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{15}O_2N$) requires *m/z* 193.1097, found *m/z* 193.1102; **IR (film)**: 3307, 3030, 2908, 1634, 1504, 1403, 1261, 1076, 913, 809, 743, 687, 577 cm^{-1}.

2-(4-(*tert*-Butyl)phenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (6)



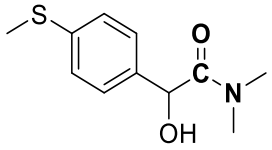
White solid (82 mg, 70% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). M.p.: 83 – 86 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7.29 – 7.18 (m, 2H), 5.19 (s, 1H), 4.71 (s, 1H), 3.03 (s, 3H), 2.78 (s, 3H), 1.30 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 151.5, 136.2, 127.2, 126.0, 71.4, 36.6, 36.4, 34.7, 31.4; **MS** (EI, *m/z*): 235 $[M]^+$, 207, 163; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{14}H_{21}O_2N$) requires *m/z* 235.1567, found *m/z* 235.1563; **IR (film)**: 3404, 2961, 2869, 1651, 1511, 1378, 1264, 1153, 1058, 857, 823, 657, 583 cm^{-1}.

2-Hydroxy-2-(4-methoxyphenyl)-*N*,*N*-dimethylacetamide (7)



White solid (80.4 mg, 76% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 114 – 116 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.04 (dd, *J* = 141.7, 8.7 Hz, 4H), 5.14 (s, 1H), 4.68 (s, 1H), 3.78 (s, 3H), 3.01 (s, 3H), 2.76 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 159.7, 131.6, 128.8, 114.5, 77.5, 77.2, 76.8, 71.1, 55.4, 36.5, 36.3; **MS** (EI, *m/z*): 209 $[M]^+$, 181, 137, 109; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{15}O_3N$) requires *m/z* 209.1046, found *m/z* 209.1053; **IR (film)**: 3312, 2932, 2833, 1633, 1508, 1457, 1420, 1403, 1259, 1177, 1071, 1030, 814, 737, 691, 641, 574 cm^{-1}.

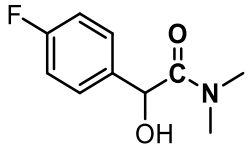
2-Hydroxy-*N*,*N*-dimethyl-2-(4-(methylthio)phenyl)acetamide (8)



White solid (96 mg, 85% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 130 – 132 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.23 (s, 4H), 5.16 (d, *J* = 4.6 Hz, 1H),

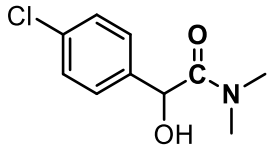
4.73 (d, J = 5.9 Hz, 1H), 3.02 (s, 3H), 2.77 (s, 3H), 2.47 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 139.2, 136.0, 128.0, 126.9, 71.2, 36.5, 36.4, 15.7; **MS** (EI, *m/z*): 225 $[M]^+$, 153, 109, 72; **HRMS** (EI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{15}O_2NS$) requires *m/z* 225.0818, found *m/z* 225.0820; **IR (film)**: 3316, 2918, 2360, 1633, 1489, 1425, 1403, 1258, 1074, 915, 806, 748, 669, 638, 536 cm^{-1}.

2-(4-Fluorophenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (9)



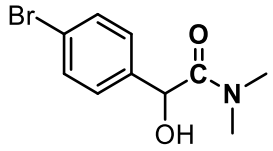
White solid (84 mg, 85% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 120 – 122 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.20 (m, 2H), 7.11 – 6.96 (m, 2H), 5.20 (s, 1H), 4.77 (s, 1H), 3.03 (s, 3H), 2.78 (s, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.27 (m, 1F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 162.7 (d, J = 247.2 Hz), 135.3 (d, J = 3.4 Hz), 129.4 (d, J = 8.4 Hz), 116.0 (d, J = 21.7 Hz), 70.8, 36.4 (d, J = 7.1 Hz). **MS** (EI, *m/z*): 197 $[M]^+$, 169, 125, 97, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O_2NF$) requires *m/z* 197.0847, found *m/z* 197.0845; **IR (film)**: 3256, 1635, 1602, 1506, 1403, 1264, 1225, 1072, 913, 819, 743, 574 cm^{-1}.

2-(4-Chlorophenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (10)



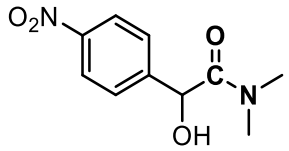
White solid (97 mg, 91% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 150 – 152 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.15 (m, 4H), 5.19 (s, 1H), 4.79 (s, 1H), 3.03 (s, 3H), 2.78 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 137.8, 134.5, 129.3, 129.0, 70.9, 36.5, 36.4; **MS** (FI, *m/z*): 213 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O_2NCl$) requires *m/z* 213.0551, found *m/z* 213.0558; **IR (film)**: 3297, 1636, 1505, 1403, 1261, 1076, 913, 814, 744, 641, 541 cm^{-1}.

2-(4-Bromophenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (11)



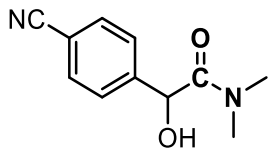
White solid (112.6 mg, 87% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). M.p.: 160 – 162 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 (dd, J = 118.5, 8.4 Hz, 4H), 5.16 (d, J = 3.9 Hz, 1H), 4.75 (d, J = 6.0 Hz, 1H), 3.02 (s, 3H), 2.77 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.0, 138.3, 132.3, 129.3, 122.7, 70.9, 36.5, 36.5; **MS** (EI, *m/z*): 257 $[M]^+$, 229, 185, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O_2NBr$) requires *m/z* 257.0046, found *m/z* 257.0047; **IR (film)**: 1635, 1507, 1405, 1260, 1075, 913, 812, 743, 633, 526 cm^{-1}.

2-Hydroxy-N,N-dimethyl-2-(4-nitrophenyl)acetamide (12)



White solid (96 mg, 86% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 1:1). M.p.: 167 – 169 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.7 Hz, 2H), 7.51 (d, J = 8.7 Hz, 2H), 5.30 (d, J = 5.9 Hz, 1H), 4.83 (d, J = 6.2 Hz, 1H), **^{13}C NMR** (101 MHz, $CDCl_3$) δ 3.05 (s, 3H), 2.81 (s, 3H); 171.3, 148.0, 146.1, 128.6, 124.4, 70.6, 36.6, 36.5; **MS** (EI, *m/z*): 224 $[M]^+$, 194, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O_4N_2$) requires *m/z* 224.0792, found *m/z* 224.0790; **IR (film)**: 3252, 1636, 1513, 1404, 1349, 1264, 1079, 913, 826, 743 , 728, 699, 637 cm^{-1}.

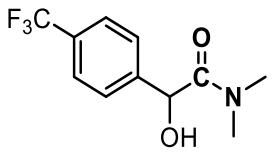
2-(4-Cyanophenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (13)



White solid (80 mg, 78% yield). R_f = 0.3 (petroleum ether/ethyl acetate = 1:1). M.p.: 135 – 137 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (dd, J = 87.6, 8.1 Hz, 4H), 5.24 (d, J = 5.3 Hz, 1H), 4.81 (d, J = 6.1 Hz, 1H), 3.01 (s, 3H), 2.78 (s, 3H); **^{13}C NMR** (101

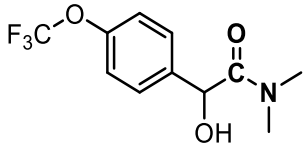
MHz, $CDCl_3$) δ 171.3, 144.2, 132.9, 128.3, 118.4, 112.5, 70.8, 36.5, 36.5; **MS** (EI, *m/z*): 204 $[M]^+$, 132, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{12}O_2N_2$) requires *m/z* 204.0893, found *m/z* 204.0892; **IR (film)**: 3253, 2935, 2228, 1638, 1501, 1423, 1404, 1263, 1157, 1079, 818, 736, 687, 648, 568 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(4-(trifluoromethyl)phenyl)acetamide (14)



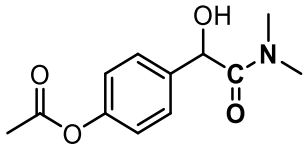
White solid (108 mg, 88% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). M.p.: 133 – 135 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 72.0, 8.0 Hz, 4H), 5.25 (s, 1H), 4.81 (s, 1H), 3.03 (s, 3H), 2.78 (s, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.74 (s, 3F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.8, 143.1, 130.8 (q, *J* = 32.6 Hz), 128.0, 126.1 (q, *J* = 3.8 Hz), 124.0 (q, *J* = 272.1 Hz), 120.0, 71.0, 36.5; **MS** (EI, *m/z*): 247 $[M]^+$, 175, 127, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{12}O_2NF_3$) requires *m/z* 247.0815, found *m/z* 247.0813; IR (film): 3214, 2930, 1635, 1500, 1420, 1403, 1331, 1157, 1124, 1080, 1069, 819, 636 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(4-(trifluoromethoxy)phenyl)acetamide (15)



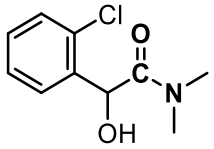
White solid (118 mg, 90% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). M.p.: 111 – 113 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 59.0, 8.3 Hz, 4H), 5.21 (s, 1H), 4.78 (s, 1H), 3.03 (s, 3H), 2.78 (s, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -57.90 (s, 3F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 149.3 (d, *J* = 2.0 Hz), 138.0, 129.1, 121.6, 117.9 (q, *J* = 257.5 Hz), 70.8, 36.5 (d, *J* = 5.5 Hz); **MS** (EI, *m/z*): 235, 191, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{12}O_3NF_3$) requires *m/z* 263.0764, found *m/z* 263.0767; **IR (film)**: 1737, 1637, 1505, 1404, 1215, 1155, 1079, 913, 743 cm^{-1}.

4-(2-(Dimethylamino)-1-hydroxy-2-oxoethyl)phenyl acetate (16)



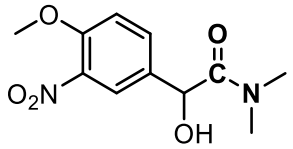
White solid (76 mg, 64% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 143 – 145 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 (dd, J = 96.3, 8.6 Hz, 4H), 5.19 (d, J = 6.3 Hz, 1H), 4.73 (d, J = 6.4 Hz, 1H), 3.01 (s, 3H), 2.78 (s, 3H), 2.28 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 169.3, 150.8, 136.8, 128.7, 122.2, 70.9, 36.5, 36.4, 21.2; **MS** (EI, *m/z*): 237 $[M]^+$, 209, 165, 123; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{12}H_{15}O_4N$) requires *m/z* 237.0996, found *m/z* 237.0993; **IR (film):** 1764, 1633, 1505, 1403, 1203, 1077, 913, 743 cm^{-1}.

2-(2-Chlorophenyl)-2-hydroxy-*N*,*N*-dimethylacetamide (17)



White solid (98 mg, 92% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 5:1). M.p.: 99 – 101 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 – 7.38 (m, 1H), 7.30 – 7.19 (m, 3H), 5.68 (s, 1H), 4.76 (s, 1H), 3.04 (s, 3H), 2.72 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.2, 136.8, 133.7, 129.9, 128.5, 127.9, 77.4, 77.1, 76.8, 67.8, 36.4, 36.0; **MS** (EI, *m/z*): 178, 141, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O_2NCl$) requires *m/z* 213.0551, found *m/z* 213.0555; **IR (film)**: 3394, 2931, 1651, 1500, 1474, 1441, 1378, 1261, 1065, 761, 737 cm^{-1}.

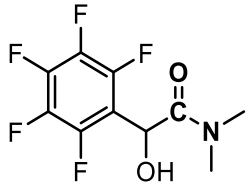
2-Hydroxy-2-(4-methoxy-3-nitrophenyl)-*N*,*N*-dimethylacetamide (18)



White solid (118 mg, 93% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 1:1). M.p.: 120 – 123 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, J = 2.4 Hz, 1H), 7.50 (dd, J = 8.7, 2.4 Hz, 1H), 7.08 (d, J = 8.7 Hz, 1H), 5.21 (s, 1H), 4.78 (s, 1H), 3.94 (s, 3H), 3.02 (s,

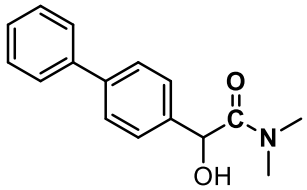
3H), 2.81 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.6, 153.0, 139.6, 133.2, 131.8, 124.9, 114.3, 77.5, 77.2, 76.8, 70.0, 56.8, 36.5 (d, J = 4 Hz); **MS** (EI, *m/z*): 254 $[M]^+$, 226, 182, 166, 107, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{11}H_{14}O_5N_2$) requires *m/z* 254.0897, found *m/z* 254.0904; **IR (film)**: 2994, 1769, 1758, 1644, 1530, 1373, 1246, 1056, 913, 743 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(perfluorophenyl)acetamide (19)



White solid (108 mg, 80% yield). R_f = 0.3 (petroleum ether/ethyl acetate = 5:1). M.p.: 111 – 112 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.48 (d, J = 5.5 Hz, 1H), 4.98 (d, J = 5.6 Hz, 1H), 3.06 (s, 3H), 2.77 (s, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -142.48 (m, 2F), -152.87 (m, 1F), -160.76 (m, 2F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.5, 147.1 – 145.3 (m), 144.5 – 143.4 (m), 143.3 – 142.2 (m), 141.1 – 139.7 (m), 139.4 – 138.6 (m), 137.4 – 135.6 (m), 115.9 – 110.3 (m), 78.7 – 75.3 (m), 61.6, 36.9, 35.9; **MS** (EI, *m/z*): 269 $[M]^+$, 197, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_8O_2NF_5$) requires *m/z* 269.0470, found *m/z* 269.0473; **IR (film)**: 3226, 1655, 1524, 1497, 1397, 1317, 1290, 1114, 1082, 993, 925, 857, 773 cm^{-1}.

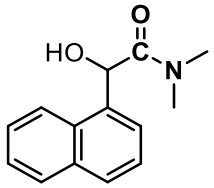
2-([1,1'-Biphenyl]-4-yl)-2-hydroxy-*N*,*N*-dimethylacetamide (20)



White solid (89 mg, 70% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). M.p.: 167 – 169 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (t, J = 7.2 Hz, 4H), 7.51 – 7.33 (m, 5H), 5.25 (d, J = 6.1 Hz, 1H), 4.80 (d, J = 6.3 Hz, 1H), 3.05 (s, 3H), 2.82 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 141.5, 140.6, 138.2, 128.9, 128.0, 127.9, 127.6, 127.2, 71.4, 36.6, 36.4; **MS** (EI, *m/z*): 255 $[M]^+$, 227, 183, 155, 72; **HRMS** (FI, *m/z*):

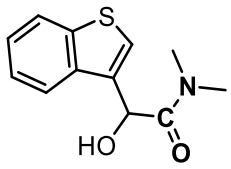
exact mass calculated for $[M]^+$ ($C_{16}H_{17}O_2N$) requires *m/z* 255.1254, found *m/z* 255.1252; **IR (film)**: 3318, 2914, 1632, 1498, 1400, 1260, 1075, 820, 755, 765, 733, 695 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(naphthalen-1-yl)acetamide (21)



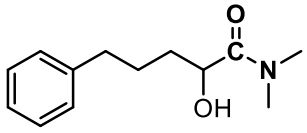
White solid (94 mg, 82% yield). R_f = 0.1 (petroleum ether/ethyl acetate = 5:1). M.p.: 98 – 100 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.30 (d, J = 8.5 Hz, 1H), 7.92 – 7.80 (m, 2H), 7.65 – 7.49 (m, 2H), 7.46 – 7.37 (m, 1H), 7.32 – 7.22 (m, 1H), 5.90 (s, 1H), 4.73 (s, 1H), 3.11 (s, 3H), 2.63 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 134.8, 134.3, 131.5, 129.4, 128.9, 127.0, 126.1, 125.5, 125.0, 123.4, 68.9, 36.6, 36.3; **MS** (EI, *m/z*): 229 $[M]^+$, 157, 129, 72; **HRMS** (EI, *m/z*): exact mass calculated for $[M]^+$ ($C_{14}H_{15}O_2N$) requires *m/z* 229.1097, found *m/z* 229.1101; **IR (film)**: 3395, 3046, 2930, 1647, 1507, 1379, 1261, 1161, 1056, 913, 803, 775, 743 cm^{-1}.

2-(Benzo[b]thiophen-3-yl)-2-hydroxy-*N*,*N*-dimethylacetamide (22)



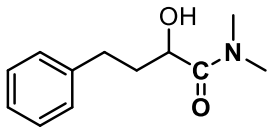
Pale yellow solid (99 mg, 84% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). M.p.: 93 – 95 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 (s, 1H), 6.48 – 6.17 (m, 4H), 5.30 (s, 1H), 4.57 (d, J = 5.9 Hz, 1H), 3.04 (s, 3H), 2.84 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.2, 152.2, 142.8, 110.7, 108.2, 100.1, 64.7, 36.5, 36.3; **MS** (EI, *m/z*): 235 $[M]^+$, 169, 141, 97, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{12}H_{13}O_2NS$) requires *m/z* 235.0662, found *m/z* 235.0658; **IR (film)**: 3106, 2902, 2849, 1644, 1498, 1399, 1256, 1065, 1009, 919, 830, 742, 637 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-5-phenylpentanamide (23)



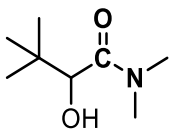
Yellow oil (95 mg, 86% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.24 (m, 2H), 7.23 – 7.15 (m, 3H), 4.37 (dd, J = 8.2, 3.0 Hz, 1H), 3.73 (br, 1H), 2.99 (s, 3H), 2.87 (s, 3H), 2.76 – 2.59 (m, 2H), 1.91 – 1.77 (m, 2H), 1.75 – 1.62 (m, 1H), 1.58 – 1.44 (m, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.4, 141.9, 128.5, 128.4, 125.9, 67.9, 36.3, 36.0, 35.4, 33.9, 26.4; **MS** (FI, *m/z*): 221 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{13}H_{19}O_2N$) requires *m/z* 221.1410, found *m/z* 221.1409; **IR (film)**: 3417, 3025, 2933, 2858, 1644, 1469, 1453, 1380, 1261, 1090, 913, 747, 700 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-4-phenylbutanamide (24)



Yellow oil (48 mg, 46% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 – 7.10 (m, 5H), 4.28 (dd, J = 9.0, 2.7 Hz, 1H), 3.80 (s, 1H), 2.93 (s, 3H), 2.85 – 2.69 (m, 5H), 1.94 – 1.82 (m, 1H), 1.76 (qd, J = 8.6, 4.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.3, 141.3, 128.7, 128.5, 126.1, 77.5, 77.2, 76.8, 67.0, 36.5, 36.2, 35.9, 31.3; **MS** (EI, *m/z*): 207 $[M]^+$, 103, 91, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{12}H_{17}O_2N$) requires *m/z* 207.1254, found *m/z* 207.1251; **IR (film)**: 3481, 3026, 2928, 2859, 1644, 1496, 1454, 1380, 1261, 1145, 1087, 753, 700, 492 cm^{-1}.

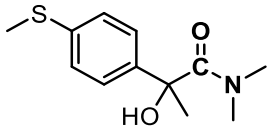
2-Hydroxy-*N*,*N*,3,3-tetramethylbutanamide (25)[2]



White solid (54 mg, 68% yield). R_f = 0.1 (petroleum ether/ethyl acetate = 5:1). M.p.: 52 – 55 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.16 (d, J = 9.1 Hz, 1H), 3.31 (d, J = 9.2 Hz,

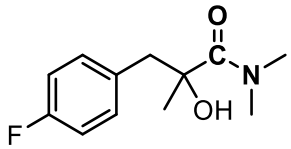
1H), 3.01 (s, 2H), 2.97 (s, 1H), 0.93 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.9, 77.5, 77.2, 76.8, 74.0, 38.0, 36.7, 35.9, 26.0; **MS** (EI, *m/z*): 159 $[M]^+$, 103, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_8H_{17}O_2N$) requires *m/z* 159.1254, found *m/z* 159.1257; **IR (film)**: 3447, 2955, 2870, 1636, 1505, 1480, 1365, 1258, 1145, 1047, 1020, 841 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(4-(methylthio)phenyl)propenamide (26)



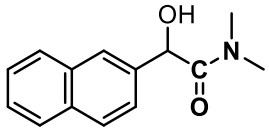
White solid (93 mg, 77% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). M.p.: 103 – 105 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.12 (m, 4H), 5.27 (s, 1H), 3.01 (s, 3H), 2.65 (s, 3H), 2.47 (d, *J* = 1.4 Hz, 3H), 1.80 (d, *J* = 1.9 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 175.0, 139.9, 138.3, 126.8, 126.1, 74.5, 38.4, 37.7, 24.8, 15.8; **MS** (EI, *m/z*): 239 $[M]^+$, 167, 151, 125; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{12}H_{17}O_2NS$) requires *m/z* 239.0975, found *m/z* 239.0977; **IR (film)**: 3373, 2980, 2924, 1623, 1492, 1438, 1398, 1363, 1258, 1096, 1014, 918, 822, 752, 653, 586 cm^{-1}.

3-(4-Fluorophenyl)-2-hydroxy-*N*,*N*,2-trimethylpropanamide (27)



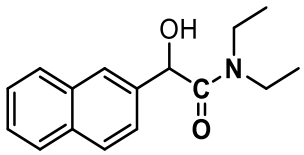
White solid (85 mg, 75% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). M.p.: 95 – 97 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 – 7.11 (m, 2H), 7.04 – 6.75 (m, 2H), 4.46 (s, 1H), 3.25 – 2.84 (m, 8H), 1.55 – 1.46 (m, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -116.3 – -116.5 (m, 1F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.9, 162.0 (d, *J* = 245.0 Hz), 132.2, 131.6, 131.5, 115.1, 114.9, 74.8, 45.2, 26.1; **MS** (EI, *m/z*): 207, 153, 116; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{12}H_{16}O_2NF$) requires *m/z* 225.1160, found *m/z* 225.1153; **IR (film)**: 2932, 1618, 1509, 1391, 1363, 1221, 1157, 1101, 913, 822, 746 cm^{-1}.

2-Hydroxy-*N*,*N*-dimethyl-2-(naphthalen-2-yl)acetamide （28）



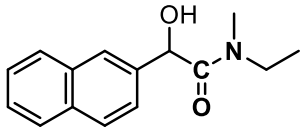
White solid (88 mg, 76% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). M.p.: 120 − 122 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (m, 4H), 7.47 (dd, J = 6.3, 3.3 Hz, 2H), 7.40 (d, J = 7.3 Hz, 1H), 5.35 (s, 1H), 4.88 (s, 1H), 3.00 (s, 3H), 2.74 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 136.4, 133.2, 133.1, 129.0, 128.0, 127.6, 126.8, 126.4, 126.3, 124.7, 71.6, 36.4, 36.2; **MS** (EI, *m/z*): 229 $[M]^+$, 201, 157, 129, 72; **HRMS** (EI, *m/z*): exact mass calculated for $[M]^+$ ($C_{14}H_{15}O_2N$) requires *m/z* 229.1097, found *m/z* 229.1102; **IR (film)**: 3315, 2938, 1644, 1505, 1263, 1162, 1066, 868, 754, 693, 623 cm^{-1}.

***N*,*N*-Diethyl-2-hydroxy-2-(naphthalen-2-yl)acetamide (29)**



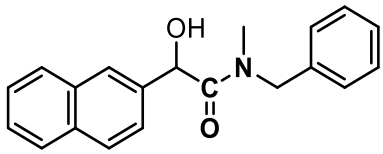
Yellow oil (107 mg, 83% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.72 (m, 4H), 7.58 – 7.37 (m, 3H), 5.35 (s, 1H), 3.53 (dq, J = 14.1, 7.1 Hz, 1H), 3.34 (dq, J = 14.1, 7.1 Hz, 1H), 3.20 (dq, J = 14.3, 7.1 Hz, 1H), 3.05 (dq, J = 14.4, 7.1 Hz, 1H), 1.14 (t, J = 7.1 Hz, 3H), 0.77 (t, J = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.4, 137.1, 133.4, 133.2, 129.1, 128.0, 127.7, 126.9, 126.4, 126.4, 124.8, 71.7, 41.0, 40.8, 13.2, 12.6; **MS** (EI, *m/z*): 257 $[M]^+$, 229, 157, 129, 100, 72; **HRMS** (EI, *m/z*): exact mass calculated for $[M]^+$ ($C_{16}H_{19}NO_2$) requires *m/z* 257.1410, found *m/z* 257.1418; **IR (film)**: 3070, 2974, 2929, 1685, 1637, 1464, 1381, 1360, 1277, 1066, 949, 819, 749, 478 cm^{-1}.

***N*-Ethyl-2-hydroxy-*N*-methyl-2-(naphthalen-2-yl)acetamide (30)**



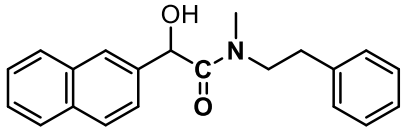
Yellow oil (87.8 mg, 72% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 5:1). A rotameric ratio of approx. 55:45 is observed in the ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 – 7.74 (m, 4H), 7.47 (d, J = 9.5 Hz, 2H), 7.40 (d, J = 8.2 Hz, 1H), 5.35 (s, 0.45×1H), 5.31 (s, 0.55×1H), 5.18 – 4.09 (br, 1H), 3.58 – 3.34 (m, 1H), 3.28 – 3.03 (m, 1H), 2.97 (s, 0.45×3H), 2.71 (s, 0.55×3H), 1.09 (t, J = 7.2 Hz, 0.55×3H), 0.74 (t, J = 7.1 Hz, 0.45×3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.8 (d, J = 9.1 Hz), 137.0, 136.5, 133.3 (d, J = 2.9 Hz), 129.0 (d, J = 2.8 Hz), 127.8 (d, J = 29.6 Hz), 126.9 (d, J = 15.5 Hz), 126.4 (d, J = 2.7 Hz), 124.8 (d, J = 2.7 Hz), 71.7 (d, J = 3.1 Hz), 43.4 (d, J = 35.4 Hz), 33.5 (d, J = 77.2 Hz), 12.2 (d, J = 50.3 Hz). **MS** (FI, *m/z*): 243 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{15}H_{17}NO_2$) requires *m/z* 243.1254, found *m/z* 243.1250; **IR (film)**: 3392, 2972, 2932, 1641, 1380, 1058, 820, 750 cm^{-1}.

***N*-Benzyl-2-hydroxy-*N*-methyl-2-(naphthalen-2-yl)acetamide (31)**



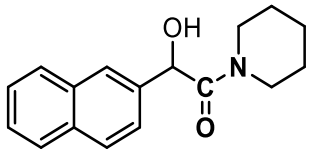
Yellow oil (112.2 mg, 74% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 4:1). Rotamers are observed in spectra. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.75 (m, 4H), 7.53 – 6.84 (m, 8H), 5.42 (d, J = 7.1 Hz, 1H), 4.89 (s, 1H), 4.78 – 4.05 (m, 2H), 3.02 – 2.66 (d, J = 112.3 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.8 (d, J = 21.8 Hz), 136.7, 136.3 (d, J = 7.7 Hz), 135.1, 133.3 (d, J = 3.6 Hz), 129.2 (d, J = 13.1 Hz), 128.8 (d, J = 16.4 Hz), 128.1 – 127.7 (m), 127.0 (d, J = 7.9 Hz), 126.6 – 126.4 (m), 124.8 (d, J = 17.7 Hz), 71.9 (d, J = 6.1 Hz), 52.2 (d, J = 20.4 Hz), 34.2 (d, J = 10.3 Hz); **MS** (FI, *m/z*): 305 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{20}H_{19}NO_2$) requires *m/z* 305.1410, found *m/z* 305.1412; **IR (film)**: 3403, 3057, 3028, 2926, 1644, 1494, 1543, 1383, 1267, 1060, 821, 749, 701 cm^{-1}.

2-Hydroxy-*N*-methyl-2-(naphthalen-2-yl)-*N*-phenethylacetamide (32)



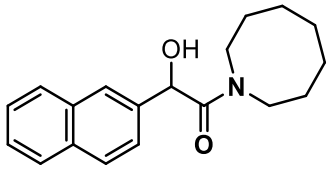
Yellow oil (144 mg, 90% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 2:1). A rotameric ratio of approx. 60:40 is observed in the ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 – 7.71 (m, 4H), 7.57 – 6.73 (m, 8H), 5.30 (d, J = 5.9 Hz, 0.6×1H), 5.15 (d, J = 5.8 Hz, 0.4×1H), 4.93 – 4.81 (m, 1H), 3.91 – 3.21 (m, 2H), 3.04 (s, 1H), 2.87 – 2.04 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.2 (d, J = 2.5 Hz), 138.4, 137.4, 136.9, 136.3, 133.4, 133.2 (d, J = 4.5 Hz), 129.2, 129.0, 128.8 – 128.6 (m), 128.4, 128.0, 127.7 (d, J = 5.9 Hz), 127.0, 126.8, 126.5 – 126.3 (m), 124.8, 71.8, 71.7, 50.9, 50.5, 35.3, 34.1, 33.9, 33.4. **MS** (FI, *m/z*): 319 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{21}H_{21}NO_2$) requires *m/z* 319.1567, found *m/z* 319.1571; **IR (film)**: 3395, 3057, 3026, 2931, 1644, 1495, 1454, 1385, 1288, 1251, 1166, 1061, 863, 820, 749, 701, 479 cm^{-1}.

2-Hydroxy-2-(naphthalen-2-yl)-1-(piperidin-1-yl)ethan-1-one (33)



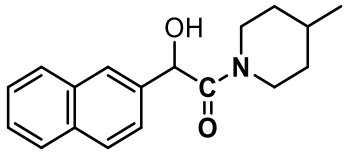
Pale yellow solid (107 mg, 79% yield). R_f = 0.7 (petroleum ether/ethyl acetate = 1:1). M.p.: 93 – 95 ºC. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 – 7.73 (m, 4H), 7.55 – 7.38 (m, 3H), 5.38 (s, 1H), 4.98 (s, 1H), 3.93 – 3.69 (m, 1H), 3.64 – 3.44 (m, 1H), 3.35 – 3.08 (m, 2H), 1.67 – 1.39 (m, 4H), 1.35 – 1.18 (m, 1H), 1.01 – 0.69 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.5, 137.1, 133.5, 133.3, 129.2, 128.1, 127.8, 126.9, 126.5, 124.9, 71.6, 46.0, 44.2, 25.5, 25.4, 24.3; **MS** (EI, *m/z*): 269 $[M]^+$, 241, 157, 129, 112; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{17}H_{19}NO_2$) requires *m/z* 269.1410, found *m/z* 269.1415; **IR (film)**: 2936, 2855, 1638, 1470, 1444, 1396, 1256, 1066, 1011, 914, 818, 748, 683, 478 cm^{-1}.

1-(Azocan-1-yl)-2-hydroxy-2-(naphthalen-2-yl)ethan-1-one (34)



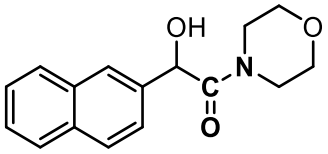
Yellow oil (80 mg, 54% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 3:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 – 7.77 (m, 4H), 7.51 – 7.41 (m, 3H), 5.36 (s, 1H), 5.03 (s, 1H), 3.60 – 3.39 (m, 2H), 3.31 – 3.10 (m, 2H), 1.87 – 1.75 (m, 2H), 1.59 – 1.12 (m, 8H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.8, 136.8, 133.2 (d, J = 9.5 Hz), 129.0, 127.9, 127.7, 127.0, 126.3, 124.9, 71.9, 48.5, 47.6, 26.5, 26.3, 25.7, 25.6, 23.8; **MS** (FI, *m/z*): 297 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{19}H_{23}NO_2$) requires *m/z* 297.1723, found *m/z* 297.1722; **IR (film)**: 3387, 2927, 2853, 1636, 1474, 1396, 1064, 819, 748 cm^{-1}.

2-Hydroxy-1-(4-methylpiperidin-1-yl)-2-(naphthalen-2-yl)ethan-1-one (35)



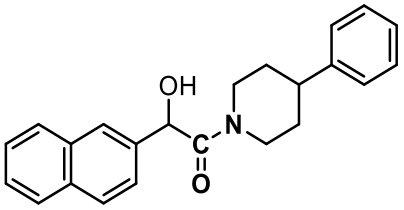
Yellow oil (124 mg, 87% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 – 7.73 (m, 4H), 7.50 (dt, J = 6.8, 3.7 Hz, 2H), 7.42 (t, J = 6.6 Hz, 1H), 5.38 (t, J = 5.5 Hz, 1H), 4.99 – 4.92 (m, 1H), 4.68 – 4.57 (m, 1H), 3.75 – 3.65 (m, 1H), 2.97 – 2.47 (m, 2H), 1.75 – 1.05 (m, 4H), 1.03 – 0.01 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.4 (d, J = 13.5 Hz), 137.0 (d, J = 20.1 Hz), 133.4 (d, J = 4.5 Hz), 133.2, 129.0 (d, J = 2.6 Hz), 128.0 (d, J = 4.1 Hz), 127.7 (d, J = 4.1 Hz), 126.9, 126.7, 126.4, 124.8 (d, J = 5.8 Hz), 71.5 (d, J = 10.6 Hz), 45.1 (d, J = 28.5 Hz), 43.4 (d, J = 9.7 Hz), 34.1, 33.6 (d, J = 5.7 Hz), 32.8, 30.7, 21.5 (d, J = 11.8 Hz); **MS** (FI, *m/z*): 283 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{18}H_{21}NO_2$) requires *m/z* 283.1567, found *m/z* 283.1565; **IR (film)**: 3391, 2949, 2925, 1640, 1456, 1397, 1270, 1242, 1068, 973, 819, 748 cm^{-1}.

2-Hydroxy-1-morpholino-2-(naphthalen-2-yl)ethan-1-one (36)



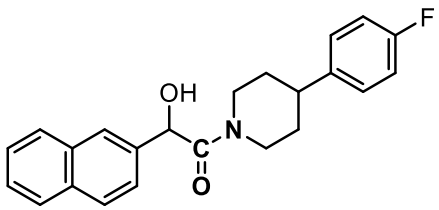
White solid (87 mg, 64% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 135 – 137 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 – 7.72 (m, 4H), 7.55 – 7.48 (m, 2H), 7.41 (dd, J = 8.5, 1.8 Hz, 1H), 5.37 (d, J = 5.4 Hz, 1H), 4.81 (d, J = 5.7 Hz, 1H), 3.85 – 3.50 (m, 4H), 3.48 – 2.95 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.1, 136.5, 133.4, 133.4, 129.4, 128.1, 127.9, 127.0, 126.7, 126.7, 124.7, 71.8, 66.7, 66.0, 45.4, 43.3; **MS** (EI, *m/z*): 271 $[M]^+$, 243, 157, 129; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{16}H_{17}NO_3$) requires *m/z* 271.1203, found *m/z* 271.1209; **IR (film)**: 3404, 2922, 2855, 1642, 1458, 1388, 1360, 1271, 1246, 1112, 1068, 1028, 913, 747, 478 cm^{-1}.

2-Hydroxy-2-(naphthalen-2-yl)-1-(4-phenylpiperidin-1-yl)ethan-1-one (37)



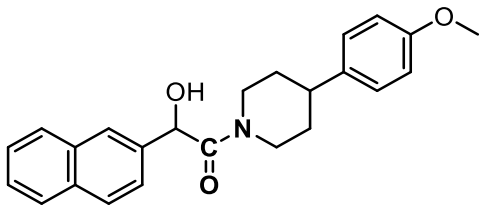
Yellow oil (124 mg, 93% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 2:1). A rotameric ratio of approx. 54:46 is observed in the ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 – 7.77 (m, 4H), 7.48 (s, 3H), 7.34 – 7.01 (m, 4H), 6.81 (d, J = 7.1 Hz, 1H), 5.45 (s, 0.46×1H), 5.42 (s, 0.54×1H), 4.83 (d, J = 12.9 Hz, 1H), 4.28 (s, 1H), 3.84 (d, J = 13.5 Hz, 1H), 3.08 – 2.49 (m, 3H), 2.11 – 0.41 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.7 (d, J = 11.1 Hz), 144.6 (d, J = 17.1 Hz), 136.9 (d, J = 25.2 Hz), 133.5 – 133.2 (m), 129.1 (d, J = 4.1 Hz), 128.6, 128.4, 128.0 (d, J = 9.3 Hz), 127.8 (d, J = 5.2 Hz), 127.1 – 126.3 (m), 124.8 (d, J = 4.0 Hz), 71.6 (d, J = 12.2 Hz), 45.5 (d, J = 34.4 Hz), 43.8 (d, J = 17.4 Hz), 42.3 (d, J = 19.0 Hz), 33.4, 32.8 (d, J = 6.4 Hz), 31.6; **MS** (FI, *m/z*): 345 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{23}H_{23}NO_2$) requires *m/z* 345.1723, found *m/z* 345.1718; **IR (film)**: 3394, 3055, 2936, 1640, 1452, 1396, 1268, 1069, 1006, 749 cm^{-1}.

1-(4-(4-Fluorophenyl)piperidin-1-yl)-2-hydroxy-2-(naphthalen-2-yl)ethan-1-one (38)



Yellow oil (162 mg, 89% yield). R_f = 0.6 (petroleum ether/ethyl acetate = 1:1). A rotameric ratio of approx. 56:44 is observed in the ^{19}F NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 – 7.77 (m, 4H), 7.53 – 7.40 (m, 3H), 7.09 (dd, J = 8.6, 5.4 Hz, 1H), 6.98 (t, J = 8.7 Hz, 1H), 6.85 – 6.70 (m, 2H), 5.45 (d, J = 6.0 Hz, 0.44×1H), 5.42 (d, J = 5.7 Hz, 0.56×1H), 5.00 (d, J = 6.1 Hz, 0.53×1H), 4.95 (d, J = 6.3 Hz, 0.44×1H), 4.83 (d, J = 12.4 Hz, 1H), 3.87 – 3.79 (m, 1H), 3.10 – 2.41 (m, 3H), 1.93 – 0.15 (m, 4H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -116.87 (tt, J = 8.6, 5.2 Hz, 0.44×1F), -117.06 (tt, J = 8.6, 5.2 Hz, 0.56×1F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.7 (d, J = 10.8 Hz), 140.4 – 140.1 (m), 137.0, 136.7, 133.4 (d, J = 7.2 Hz), 133.2, 129.1 (d, J = 2.2 Hz), 128.1 – 127.7 (m), 127.0, 126.7, 126.6 – 126.2 (m), 124.8 (d, J = 9.1 Hz), 115.2 (t), 71.6 (d, J = 10.4 Hz), 45.5, 45.2, 43.8, 43.6, 41.6, 41.4, 33.5, 32.9 (d, J = 11.8 Hz), 31.7; **MS** (FI, *m/z*): 363 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{23}H_{22}NO_2F$) requires *m/z* 363.1629, found *m/z* 363.1633; **IR (film)**: 3396, 2936, 2860, 1640, 1509, 1472, 1395, 1277, 1220, 1159, 1068, 1004, 833, 749 cm^{-1}.

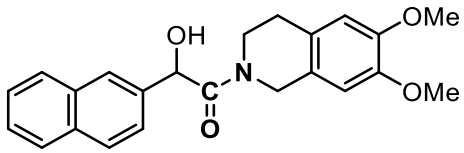
2-Hydroxy-1-(4-(4-methoxyphenyl)piperidin-1-yl)-2-(naphthalen-2-yl)ethan-1-one (39)



Yellow oil (142 mg, 76% yield). R_f = 0.5 (petroleum ether/ethyl acetate = 1:1). A rotameric ratio of approx. 54:46 is observed in the ^{1}H NMR. **^{1}H NMR** (400 MHz,

$CDCl_3$) δ 7.92 – 7.78 (m, 4H), 7.54 – 7.42 (m, 3H), 7.06 (d, J = 8.4 Hz, 1H), 6.85 (d, J = 8.4 Hz, 1H), 6.71 (dd, J = 22.4, 8.6 Hz, 2H), 5.47 (s, 0.46×1H), 5.43 (s, 0.54×1H), 5.01 (s, 1H), 4.82 (d, J = 13.4 Hz, 1H), 3.88 – 3.79 (m, 1H), 3.77 (s, 0.46×3H), 3.69 (s, 0.54×3H), 3.04 – 2.47 (m, 3H), 1.91 – 0.37 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.5 (d, J = 12.4 Hz), 158.1 (d, J = 18.4 Hz), 137.1 – 136.5 (m), 133.3 (d, J = 6.8 Hz), 133.1, 129.0 (d, J = 2.8 Hz), 127.9 (d, J = 9.8 Hz), 127.7 (d, J = 5.1 Hz), 127.4, 127.2, 126.9, 126.6, 126.4 (d, J = 5.8 Hz), 124.8 (d, J = 4.7 Hz), 71.5 (d, J = 11.6 Hz), 55.1 (d, J = 7.5 Hz), 45.6, 45.2, 43.7 (d, J = 16.5 Hz), 41.3 (d, J = 19.7 Hz), 33.6, 32.9 (d, J = 6.3 Hz), 31.8; **MS** (FI, *m/z*): 375 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{24}H_{25}NO_3$) requires *m/z* 375.1829, found *m/z* 375.1827; **IR (film)**: 3395, 2935, 2862, 1644, 1513, 1462, 1394, 1367, 1247, 1179, 1069, 828 cm^{-1}.

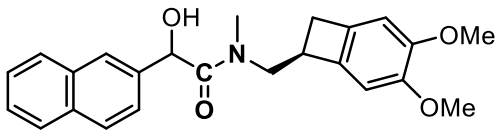
1-(6,7-Dimethoxy-3,4-dihydroisoquinolin-2(1H)-yl)-2-hydroxy-2-(naphthalen-2-yl)ethan-1-one (40)



Yellow oil (176 mg, 93% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). Rotamers are observed in spectrum. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.73 (m, 4H), 7.49 – 7.36 (m, 3H), 6.60 – 6.07 (m, 2H), 5.45 (d, J = 4.0 Hz, 1H), 5.17 – 4.19 (m, 3H), 4.06 (d, J = 15.4 Hz, 1H), 3.92 – 3.65 (m, 5H), 3.56 (s, 1H), 3.46 (t, J = 5.8 Hz, 1H), 2.85 – 1.93 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.2 (d, J = 10.6 Hz), 148.3 – 147.5 (m), 136.5 (d, J = 27.3 Hz), 133.1 (d, J = 2.8 Hz), 129.2, 127.9 (d, J = 11.4 Hz), 127.7 (d, J = 5.3 Hz), 126.8 (d, J = 2.7 Hz), 126.5 – 126.2 (m), 125.2, 124.7 (d, J = 2.4 Hz), 124.0, 123.3, 111.3 (d, J = 20.3 Hz), 109.2, 108.6, 71.9, 56.3 – 55.5 (m), 46.3, 44.8, 42.5, 41.3, 27.7 (d, J = 21.2 Hz); **MS** (FI, *m/z*): 377 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{23}H_{23}NO_4$) requires *m/z* 377.1622, found *m/z* 377.1619; **IR (film)**: 3399, 3055, 2935, 2836, 1644, 1518, 1454, 1257, 1115, 1049, 734 cm^{-1}.

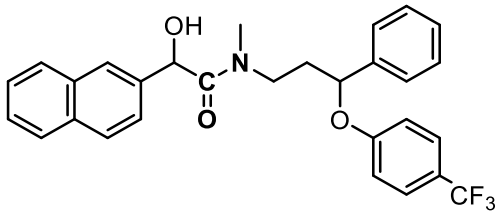
***N*-(((*S*)-3,4-Dimethoxybicyclo[4.2.0]octa-1(6),2,4-trien-7-yl)methyl)-2-hydroxy-*N*-**

methyl-2-(naphthalen-2-yl)acetamide (41)



Yellow oil (173 mg, 89% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 2:1). d.r. 60:40 (determined by HPLC). A mixture of 4 pairs of isomers (arising from rotational isomerism and diastereoisomerism). d.r. 60:40 (determined by HPLC). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 – 7.59 (m, 4H), 7.53 – 7.31 (m, 3H), 6.76 – 5.11 (m, 3H), 4.87 (br, 1H), 4.21 – 3.62 (m, 7H), 3.35 – 2.97 (m, 4H), 2.89 – 2.37 (m, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7 (d, J = 18.4 Hz), 172.3 (d, J = 18.0 Hz), 150.3, 150.2, 149.9, 149.7, 149.4 (d, J = 9.0 Hz), 137.3 – 136.2 (m), 134.8, 134.5, 134.1 – 133.1 (m), 129.2 – 128.9 (m), 128.1 – 127.6 (m), 127.2 – 126.7 (m), 126.6 – 126.2 (m), 125.0, 124.7, 124.5, 107.4 (d, J = 14.7 Hz), 106.9 (d, J = 23.7 Hz), 106.3 (d, J = 36.0 Hz), 71.7 (d, J = 14.1 Hz), 56.3 (d, J = 3.7 Hz), 56.2 – 55.9 (m), 55.6, 53.6, 52.5 (d, J = 14.9 Hz), 52.0, 40.8, 40.6, 40.0 (d, J = 4.6 Hz), 36.4, 35.9, 34.2 (d, J = 6.7 Hz), 33.9, 33.5, 32.8; **MS** (EI, *m/z*): 391 $[M]^+$; **HRMS** (EI, *m/z*): exact mass calculated for $[M]^+$ ($C_{24}H_{25}NO_4$) requires *m/z* 391.1778, found *m/z* 391.1781; **IR (film)**: 3399, 3054, 2923, 1650, 1591, 1490, 1384, 1059, 822, 750 cm^{-1}.

***N*-(3-(4-((Difluoro-λ^3-methyl)-λ^2-fluoraneyl)phenoxy)-3-phenylpropyl)-2-hydroxy-*N*-methyl-2-(naphthalen-2-yl)acetamide （42）**



Yellow oil (155 mg, 63% yield). R_f = 0.4 (petroleum ether/ethyl acetate = 2:1). A mixture of 4 pairs of isomers (arising from rotational isomerism and diastereoisomerism). d.r. 60:40 (determined by chiral HPLC). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, J = 25.8 Hz, 4H), 7.61 – 7.09 (m, 9H), 6.98 – 6.50 (m, 3H), 5.34 (d,

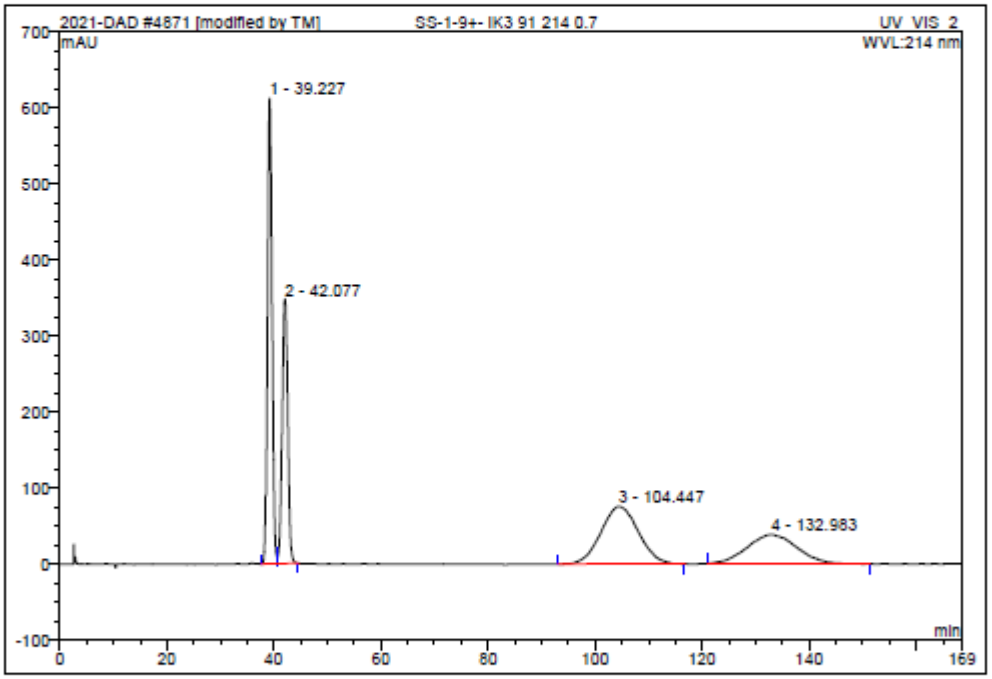
J = 2.8 Hz, 0.58×1H), 5.30 (d, *J* = 6.1 Hz, 0.42×1H), 5.15 – 4.59 (m, 1H), 3.89 – 3.21 (m, 2H), 3.09 – 2.67 (m, 3H), 2.29 – 1.41 (m, 2H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -60.5 – -64.4 (m, 3F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.5 (d, *J* = 5.9 Hz), 172.1 (d, *J* = 37.5 Hz), 160.2, 159.8 (d, *J* = 13.0 Hz), 140.3 (d, *J* = 12.2 Hz), 139.5, 137.1, 136.8, 136.3 (d, *J* = 3.1 Hz), 133.4 – 133.1 (m), 129.4 – 128.7 (m), 128.3 – 127.6 (m), 127.1 (d, *J* = 10.3 Hz), 126.6 (dd, *J* = 13.3, 9.8 Hz), 125.7 – 125.4 (m), 125.0, 124.6 (dd, *J* = 12.5, 3.5 Hz), 115.7 (d, *J* = 9.9 Hz), 115.4 (d, *J* = 12.4 Hz), 78.0 – 77.1 (m), 72.0, 71.7 (d, *J* = 7.3 Hz), 46.6, 46.2, 45.7, 45.3, 36.5, 36.0 (d, *J* = 8.5 Hz), 35.7, 35.0, 34.0 (d, *J* = 6.4 Hz); **MS** (FI, *m/z*): 493 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{29}H_{26}F_3NO_4$) requires *m/z* 493.1859, found *m/z* 493.1866; **IR (film)**: 3396, 3058, 2931, 1644, 1516, 1380, 1327, 1248, 1162, 1111, 1067, 836, 750 cm^{-1}; **Chiral HPLC** (IK3, 0.46×15 cm, 5 μm, *n*-hexane/isopropanol = 90/10, flow 0.7 mL/min, detection at 214 nm): retention time = 39.23 min (29.70%), 42.08 min (20.59%), 104.45 min (29.85%) and 132.98 min (19.85%).

Operator:TM Timebase:HPLC Sequence:2021-DAD Pa

2023-7-3 4:3

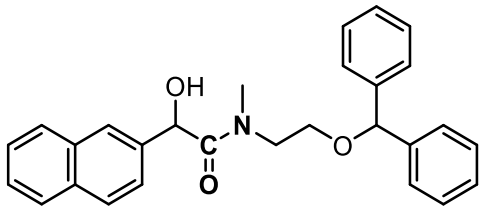
4871 SS-1-9+- IK3 91 214 0.7

Sample Name:	**SS-1-9+- IK3 91 214 0.7**	*Injection Volume:*	**3.0**
Vial Number:	**BD6**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**214.0**
Control Program:	**test-dad**	*Bandwidth:*	**4**
Quantif. Method:	**WXL**	*Dilution Factor:*	**1.0000**
Recording Time:	**2023-7-3 13:34**	*Sample Weight:*	**1.0000**
Run Time (min):	**168.54**	*Sample Amount:*	**1.0000**



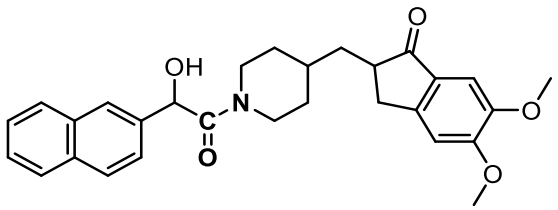
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	39.23	n.a.	612.229	608.551	29.70	n.a.	BM
2	42.08	n.a.	347.693	421.839	20.59	n.a.	MB
3	104.45	n.a.	75.602	611.608	29.85	n.a.	BMB*
4	132.98	n.a.	38.256	406.687	19.85	n.a.	BMB*
Total:			1073.780	2048.685	100.00	0.000	

***N*-(2-(Benzhydryloxy)ethyl)-2-hydroxy-*N*-methyl-2-(naphthalen-2-yl)acetamide (43)**



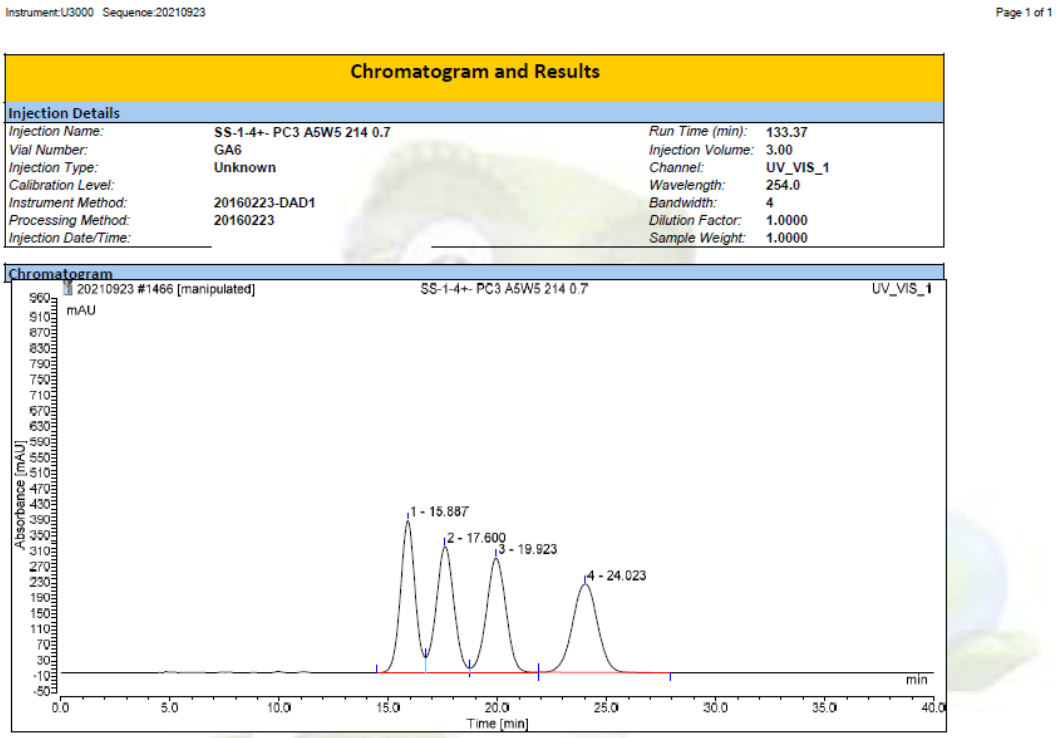
Yellow oil (179 mg, 84% yield). R_f = 0.8 (petroleum ether/ethyl acetate = 1:1). A rotameric ratio of approx. 62:38 is observed in the ^{1}H NMR. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 – 7.74 (m, 4H), 7.54 – 7.42 (m, 3H), 7.35 – 7.12 (m, 10H), 5.63 (s, 0.38×1H), 5.40 (s, 0.62×1H), 5.27 (s, 0.62×1H), 5.12 (s, 0.38×1H), 3.95 – 3.05 (m, 4H), 3.00 (s, 0.38×3H), 2.86 (s, 0.62×3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.6 (d, J = 53.1 Hz), 141.8 (d, J = 2.3 Hz), 141.6, 141.4, 137.0, 136.3, 133.3 – 133.0 (m), 129.0 (d, J = 2.9 Hz), 128.4 (d, J = 5.4 Hz), 128.3, 128.2, 128.0, 127.7 – 127.5 (m), 127.5 – 127.3 (m), 127.0, 126.8, 126.7 – 126.6 (m), 126.4, 126.3 (d, J = 4.0 Hz), 84.1, 83.9, 71.7 (d, J = 1.9 Hz), 66.8, 65.6, 49.1, 48.6, 36.2, 34.5; **MS** (FI, *m/z*): 425 $[M]^+$; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{28}H_{27}NO_3$) requires *m/z* 425.1985, found *m/z* 425.1992; **IR (film)**: 3400, 3057, 2936, 1651, 1493, 1453, 1384, 1062, 862, 820, 745, 703, 479 cm^{-1}.

2-((1-(2-Hydroxy-2-(naphthalen-2-yl)acetyl)piperidin-4-yl)methyl)-5,6-dimethoxy-2,3-dihydro-1H-inden-1-one (44)



Yellow oil (207 mg, 88% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 1:1). A mixture of 4 pairs of isomers (arising from rotational isomerism and diastereoisomerism). d.r. 50:50 (determined by chiral HPLC). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 – 7.69 (m, 4H), 7.49 (dd, J = 6.4, 3.2 Hz, 2H), 7.41 (dt, J = 8.5, 2.5 Hz, 1H), 7.12 (d, J = 20.4 Hz, 1H), 6.86 – 6.73 (m, 1H), 5.37 (s, 1H), 4.93 (s, 1H), 4.68 (t, J = 13.9 Hz, 1H), 3.99 – 3.91 (m, 3H), 3.88 (d, J = 8.5 Hz, 3H), 3.82 – 3.67 (m, 1H), 3.36 – 2.39 (m, 5H), 1.98 – 0.04 (m, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 207.1 (d, J = 3.9 Hz), 170.4 (d, J = 18.3 Hz), 155.5 (d, J = 5.3 Hz), 149.4 (d, J = 5.2 Hz), 148.4, 136.9, 136.8, 133.3 (d, J = 4.9 Hz), 133.1, 129.3 – 128.9 (m), 128.1 – 127.8 (m), 127.7 (d, J = 3.6 Hz), 127.6, 126.8 (d, J = 3.9 Hz), 126.6, 126.3 (d, J = 4.7 Hz), 124.7, 124.5 (d, J = 5.6 Hz), 107.3 (d, J = 4.9 Hz), 104.3 (d, J = 5.3 Hz), 71.5 (d, J = 9.7 Hz), 56.1

(d, J = 14.1 Hz), 45.3 – 44.4 (m), 43.2 (d, J = 14.5 Hz), 38.3 – 37.8 (m), 34.2, 34.0, 33.6 – 32.9 (m), 32.7, 32.2 – 31.8 (m), 31.6 – 31.0 (m), 30.6; **MS** (DART, *m/z*): 425 ($[M+H]^+$); **HRMS** (DART, *m/z*): exact mass calculated for $[M+H]^+$ ($C_{29}H_{32}NO_5$) requires *m/z* 474.2275, found *m/z* 474.2278; **IR (film)**: 3390, 2925, 1693, 1640, 1463, 1313, 1265, 1123, 1067, 1036, 732 cm^{-1}. **Chiral HPLC** (Lux 5u Cellulose-3, 0.46×25 cm, 5 μm, CH_3CN/H_2O = 50/50, flow 0.7 mL/min, detection at 214 nm): retention time = 15.89 min (24.90%), 17.60 min (25.11%), 19.92 min (25.11%) and 24.02 min (24.88%).



Instrument:U3000 Sequence:20210923 Page 1 of 1

Chromatogram and Results

Injection Details

Injection Name:	SS-1-4+- PC3 A5W5 214 0.7	*Run Time (min):*	133.37
Vial Number:	GA6	*Injection Volume:*	3.00
Injection Type:	Unknown	*Channel:*	UV_VIS_1
Calibration Level:		*Wavelength:*	254.0
Instrument Method:	20160223-DAD1	*Bandwidth:*	4
Processing Method:	20160223	*Dilution Factor:*	1.0000
Injection Date/Time:		*Sample Weight:*	1.0000

Chromatogram

Integration Results

No.	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Resolution (EP)	Asymmetry (EP)	K'	Plates (EP)
n.a.	15.887	302.4990	390.1664	24.901	1.28	n.a.	n.a.	2762
n.a.	17.600	304.9780	324.5777	25.105	1.50	n.a.	n.a.	2268
n.a.	19.923	305.0374	294.7748	25.110	2.20	1.00	n.a.	2373
n.a.	24.023	302.2736	227.0692	24.883	n.a.	0.99	n.a.	2097
Total:		**1214.788**	**1401.998**	**100.000**				

4 Gram-scale synthesis

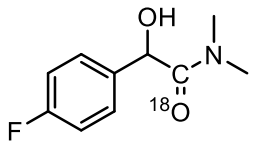
Under argon atmosphere, a solution of 4-fluorobenzaldehyde (**1**, 10.0 mmol, 1.0 equiv.), $TMSCF_2Br$ (20.0 mmol, 2.0 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (**2**, 20.0 mmol, 2.0 equiv.) in 1,4-dioxane (50 mL) was stirred at room temperature for

1.0 hour. Then H_2O (10 mL) was added and the mixture was stirred at room temperature for 0.5 hour. The mixture was quenched with water, and extracted with ethyl acetate for three times. The combined extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by flash column chromatography using petroleum ether/ethyl acetate to afford product (1.6 g, 81%).

5 The synthesis of ^{18}O-labeled *α*-hydroxyamides

Under argon atmosphere, a solution of 4-fluorobenzaldehyde (**1**, 0.2 mmol, 1.0 equiv.), $TMSCF_2Br$ (0.4 mmol, 2.0 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (**2**, 0.4 mmol, 2.0 equiv.) in 1,4-dioxane (2 mL) was stirred at room temperature for 1.0 hour. Then $H_2{}^{18}O$ (0.2 mL) was added and the mixture was stirred at room temperature for 0.5 hour. The mixture was quenched with water, and extracted with ethyl acetate for three times. The combined extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by flash column chromatography using petroleum ether/ethyl acetate to afford product. The ^{18}O content of sample [^{18}O]-*α*-hydroxyamides **46** was estimated peak heights of ^{16}O- and ^{18}O-parent peaks in mass spectrum. And the final **46** enriched in the ^{18}O isotope with 97%

2-(4-Fluorophenyl)-2-hydroxy-*N,N*-dimethylacetamide-^{18}*O* (45)



White solid (32 mg, 80% yield, 97% ^{18}*O*-isotopic purity.). R_f = 0.4 (petroleum ether/ethyl acetate = 1:1). M.p.: 118 – 120 ºC. **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.29 (dt, *J* = 9.7, 4.7 Hz, 2H), 7.05 (t, *J* = 8.5 Hz, 2H), 5.19 (d, *J* = 5.4 Hz, 1H), 4.75 (d, *J* = 6.1 Hz, 1H), 3.03 (s, 3H), 2.78 (s, 3H); **^{19}F NMR** (376 MHz, $CDCl_3$) *δ* -113.6 – -113.9 (m, 1F); **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 172.3, 164.0, 161.6, 135.3 (d, *J* = 3.4 Hz), 129.4 (d, *J* = 8.3 Hz), 116.1 (d, *J* = 21.6 Hz), 70.9, 36.5 (d, *J* = 6.3 Hz); **MS** (EI, *m/z*): 199 $[M]^+$, 169, 125, 97, 74; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{10}H_{12}O^{18}ONF$) requires *m/z* 199.0889, found *m/z* 199.0887. **IR (film)**: 3291, 2925,

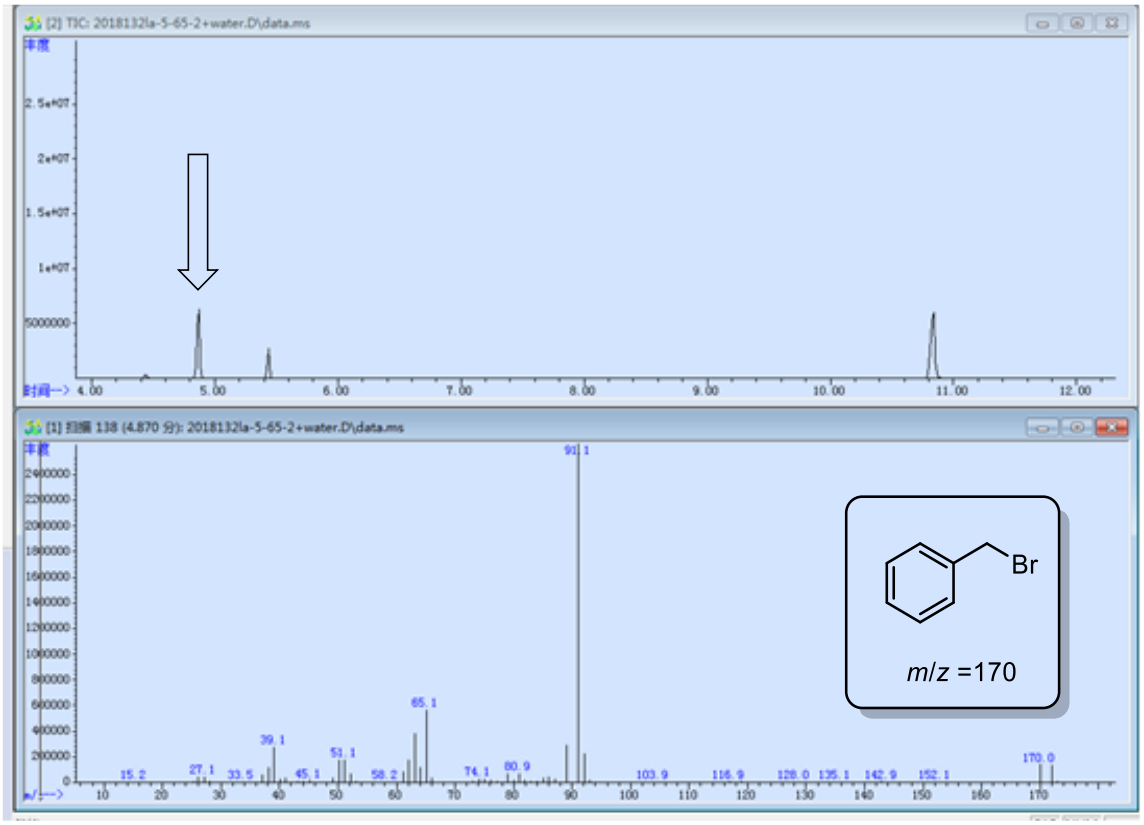
1612, 1506, 1403, 1261, 1255, 1156, 1701, 817, 749, 574 cm^{-1}.

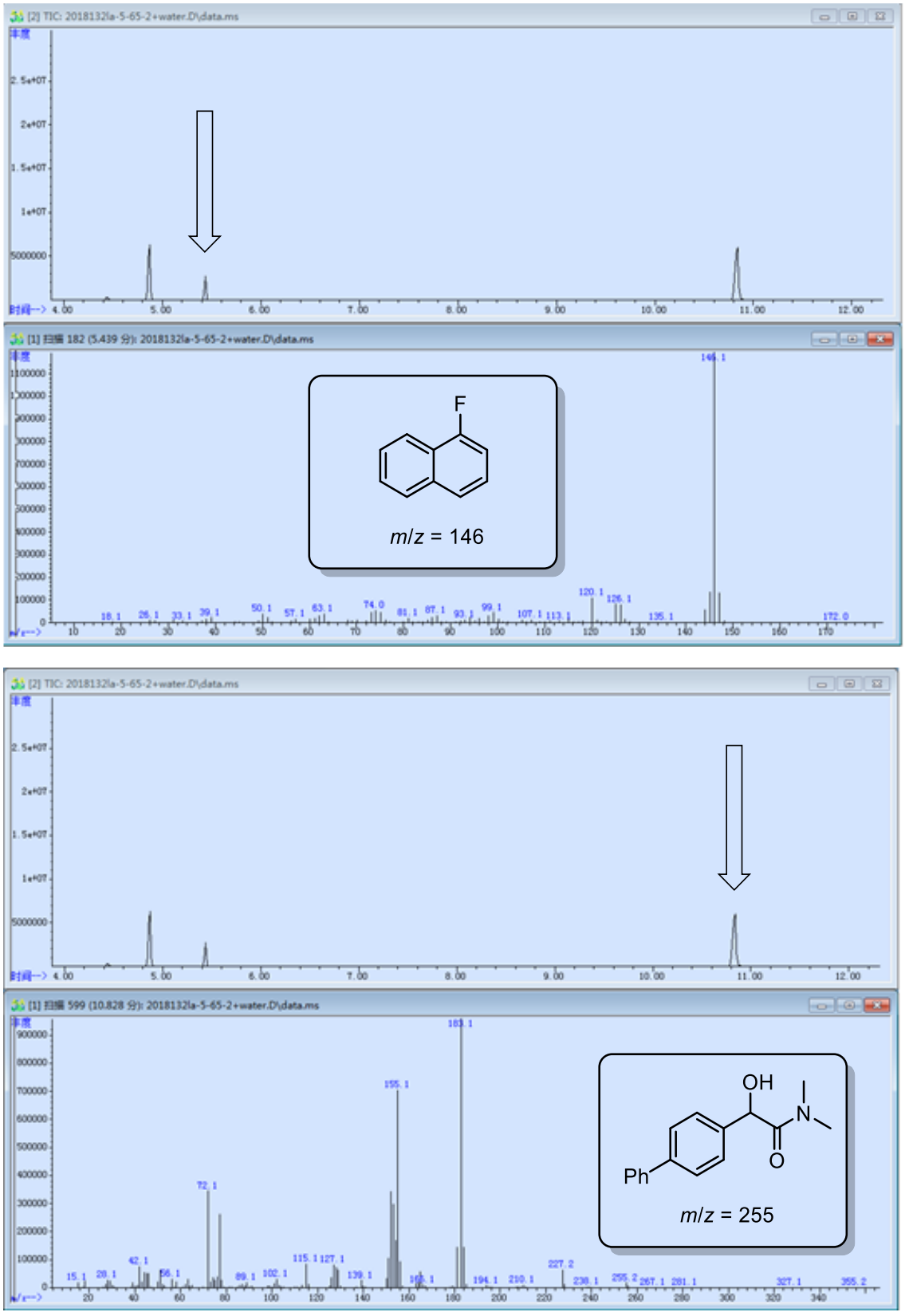
6 Mechanistic investigations

i. monitoring experiments (GC-MS and ^{19}F NMR)

Under argon atmosphere, a solution of 4-phenylbenzaldehyde (0.5 mmol, 1.0 equiv.), $TMSCF_2Br$ (1.0 mmol, 2.0 equiv.) and *N,N*-dimethyl-1-phenylmethanamine (**2a**, 1.0 mmol, 2.0 equiv.) in 1,4-dioxane (5 mL) was stirred at room temperature for 1.0 hour. 1-Fluoronaphthalene (30 μL) was added into the mixture as an internal standard, and the reaction was monitored by GC-MS and ^{19}F NMR. Then H_2O (0.5 mL) was added and the mixture was stirred at room temperature for 0.5 hour, the reaction was monitored again by GC-MS and ^{19}F NMR.

Peaks in GC spectrum were identified by the corresponding mass spectral signals, see:



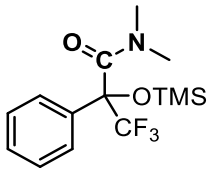


ii. The isolation of TMS-protected α-hydroxyamide

TMS-protected *α*-hydroxyamide (**46**) was successfully isolated when 2,2,2-

trifluoro-1-phenylethan-1-one was subject to the standard conditions.

3,3,3-Trifluoro-*N,N*-dimethyl-2-phenyl-2-((trimethylsilyl)oxy)propenamide (46)



Pale yellow solid (121.5 mg, 76% yield). R_f = 0.2 (petroleum ether/ethyl acetate = 5:1). M.p.: 91 – 93 ºC. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 – 7.44 (m, 2H), 7.43 – 7.30 (m, 3H), 2.92 (s, 3H), 2.61 (s, 3H), 0.26 (s, 9H); **^{19}F NMR** (376 MHz, $CDCl_3$) δ -74.0 (s, 3F); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.3, 135.7, 129.1, 128.4, 126.3, 123.5 (q, J = 286.7 Hz), 81.5 (q, J = 26.6 Hz), 37.7, 36.4, 0.8 ; **MS** (EI, *m/z*): 319 [M^+], 304, 247, 145, 105, 72; **HRMS** (FI, *m/z*): exact mass calculated for $[M]^+$ ($C_{14}H_{20}O_2NF_3Si$) requires *m/z* 319.1210, found *m/z* 319.1216; **IR (film)**: 3353, 2957, 1660, 1495, 1450, 1395, 1256, 1192, 1157, 1135, 1006, 955, 880, 846, 763, 714 cm^{-1}.

7 Procedures for the synthesis of substrates

$TMSCFCl_2$[3], $TMSCFClBr_2$[3], $TMSCCl_2Br$[4] and benzylamines[5] were prepared by the reported methods.

8 References

1 K. A. Mix, J. E. Lomax and R. T. Raines, Cytosolic Delivery of Proteins by Bioreversible Esterification. *J. Am. Chem. Soc.* 2017, **139**, 14396.

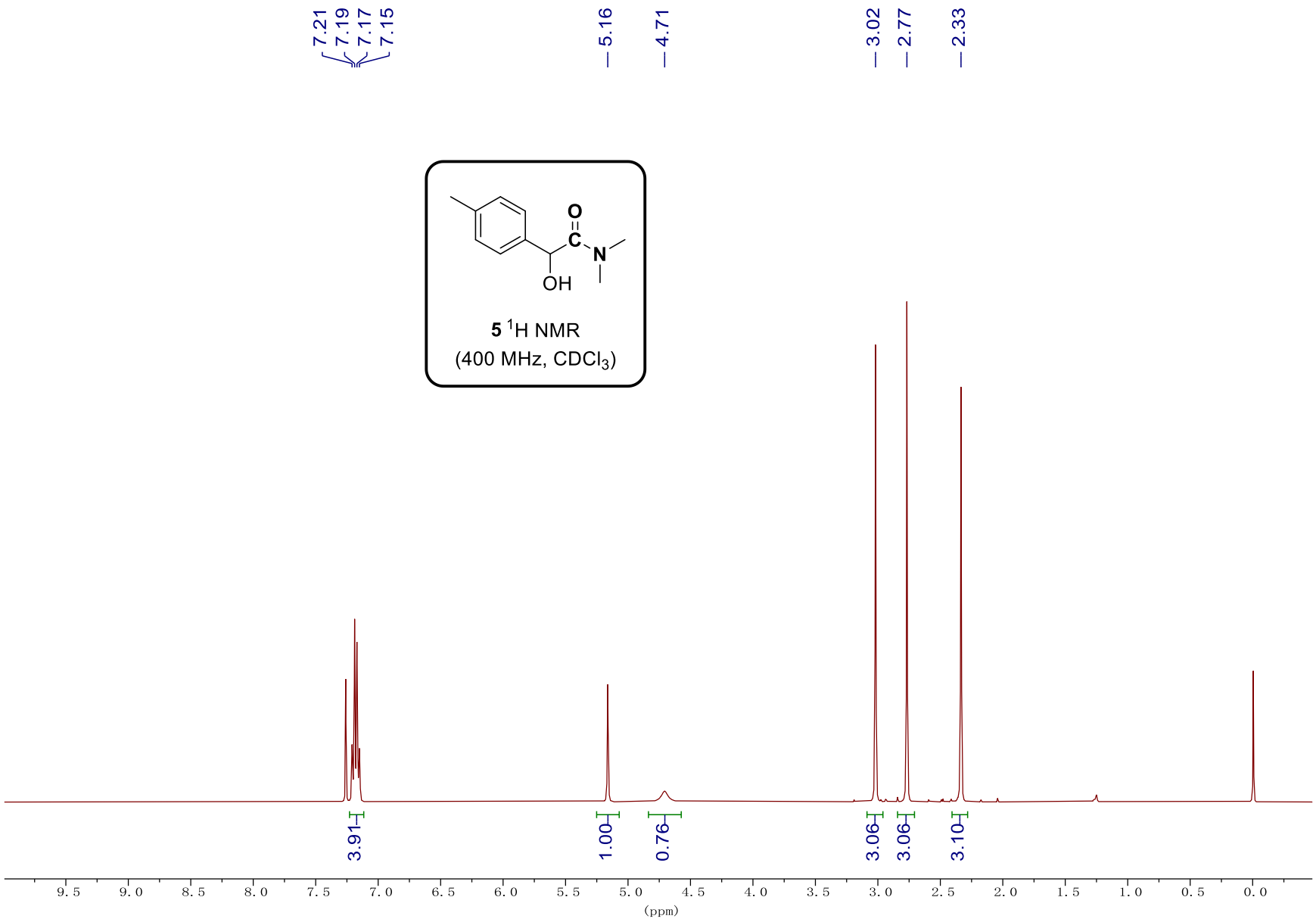
2 B. Bánhidai and U. Schöllkopf, (Dimethylcarbamoyl)lithium from Dimethylformamide and Lithium Diisopropylamide; Synthesis of α-Hydroxy N,N-Dimethylcarboxyamides. *Angew. Chem. Int. Ed.* 1973, **12**, 836.

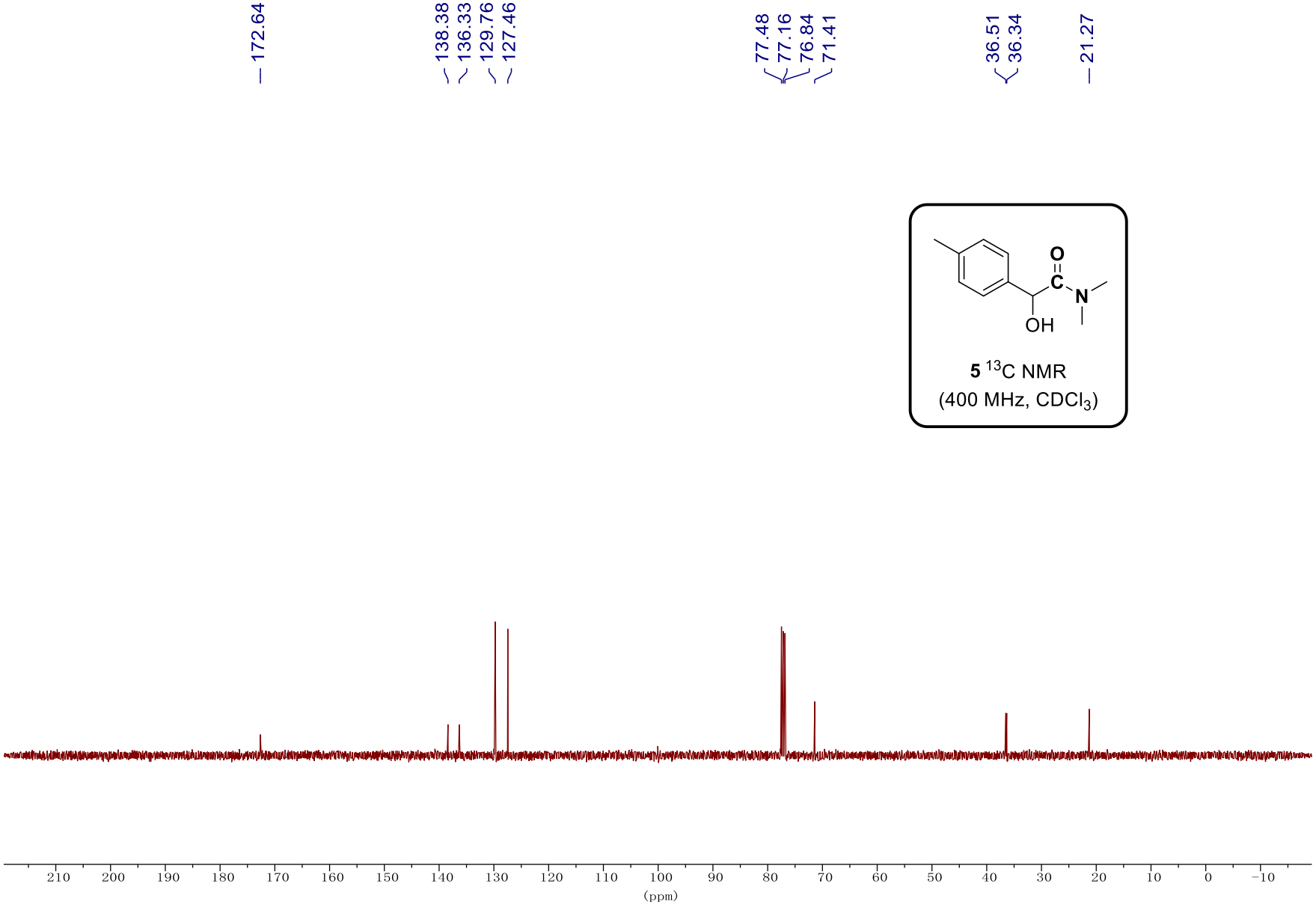
3 D. Chen, Z. Fan, L. Huang, K. Gao, P. Xiao, C. Ni and J. Hu, $TMSCFX_2$ (X = Cl, Br) as halofluorocarbene sources for the synthesis of halofluorocyclopropanes. *Chem. Commun.*, 2021, **57**, 319.

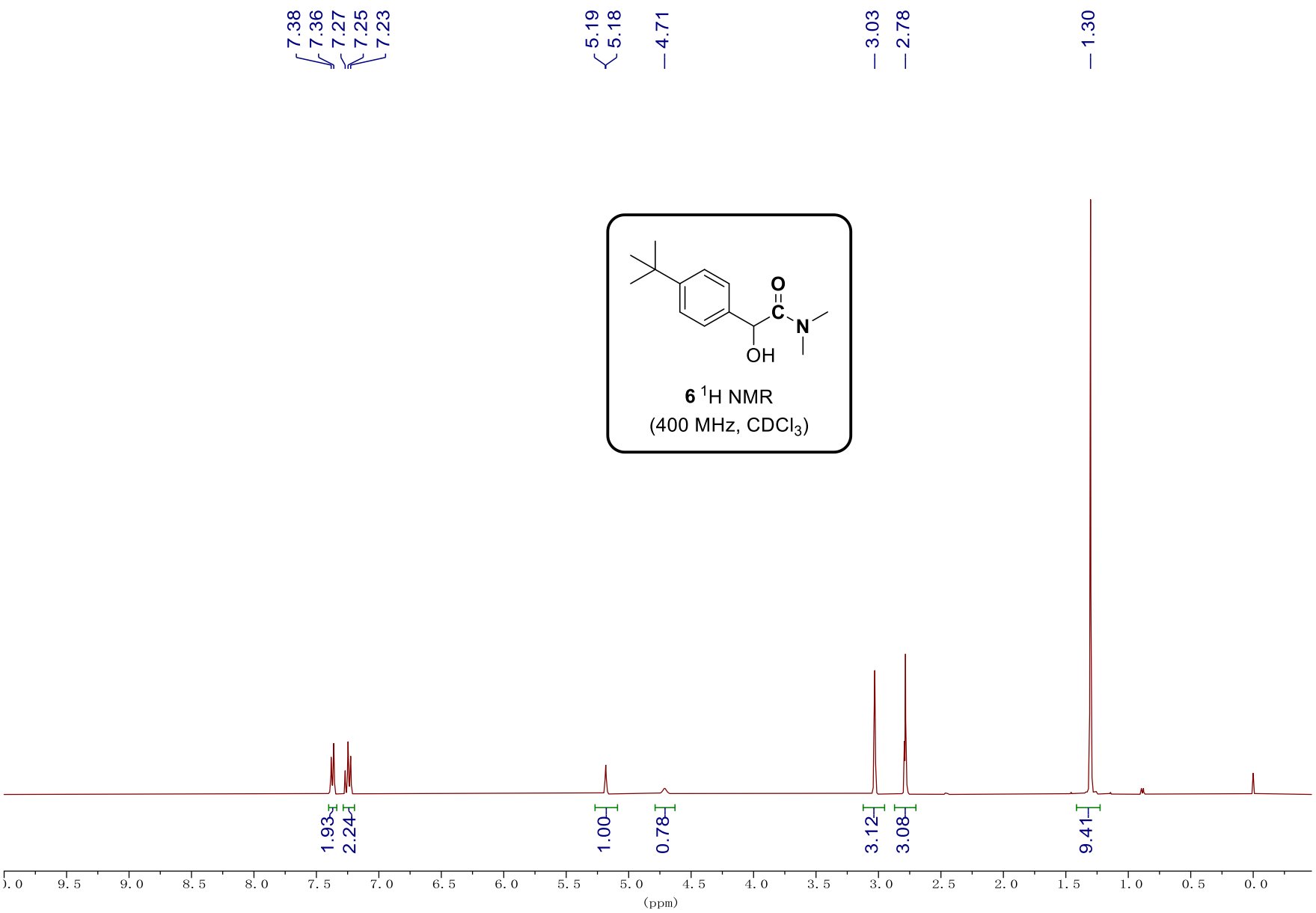
4 D. S. Lee, M. J. Duran-Pena, L. Burroughs and S. Woodward, Efficient Preparation of $TMSCCl_2Br$ and Its Use in Dichlorocyclopropanation of Electron-Deficient Alkenes. *Chem. Eur. J.*, 2016, **22**, 7609.

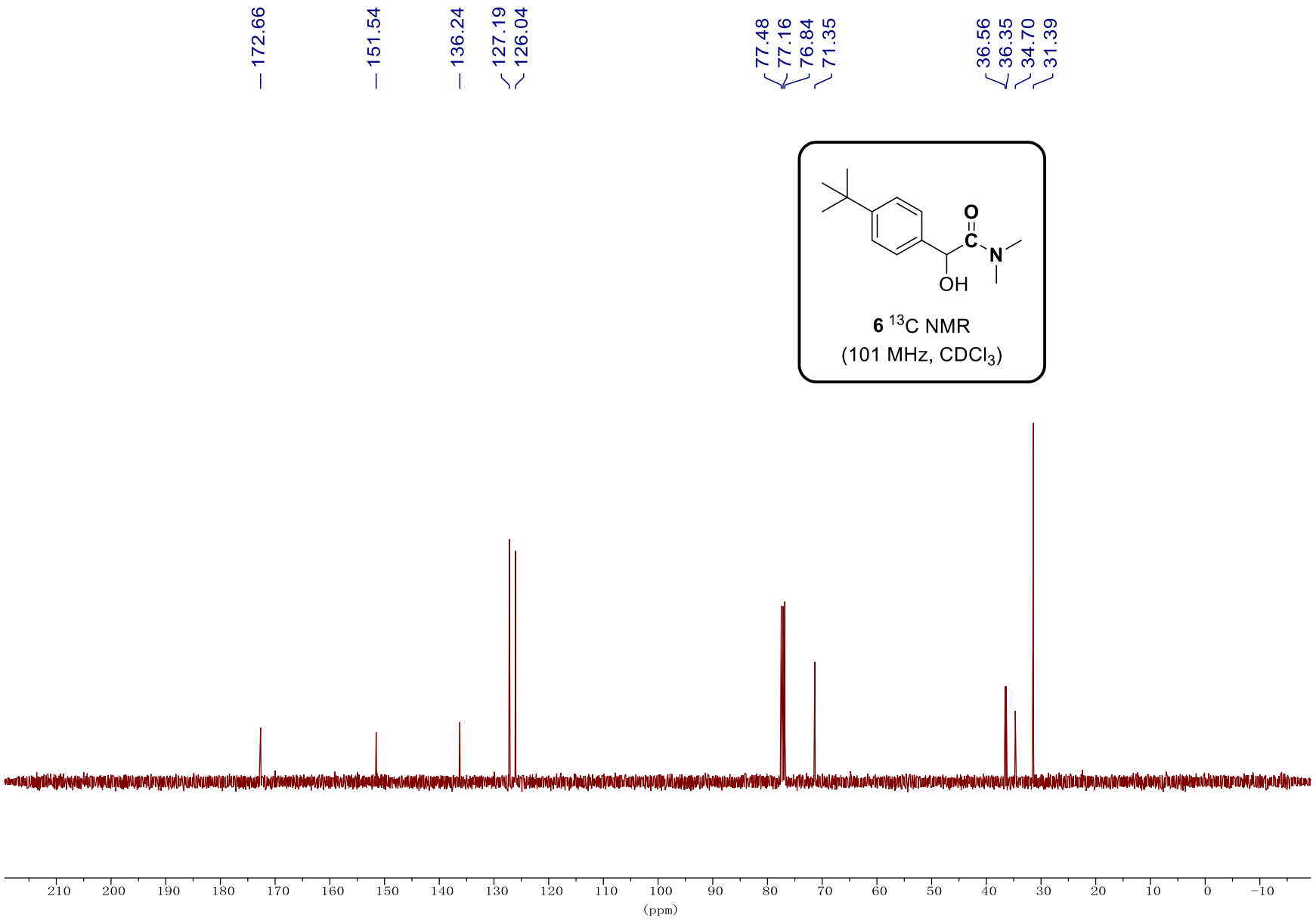
5 A. Liu, C. Ni, Q. Xie and J. Hu, TMSCF_2Br-Enabled Fluorination-Aminocarbonylation of Aldehydes: Modular Access to alpha-Fluoroamides. *Angew. Chem. Int. Ed.*, 2022, **61**, e202115467.

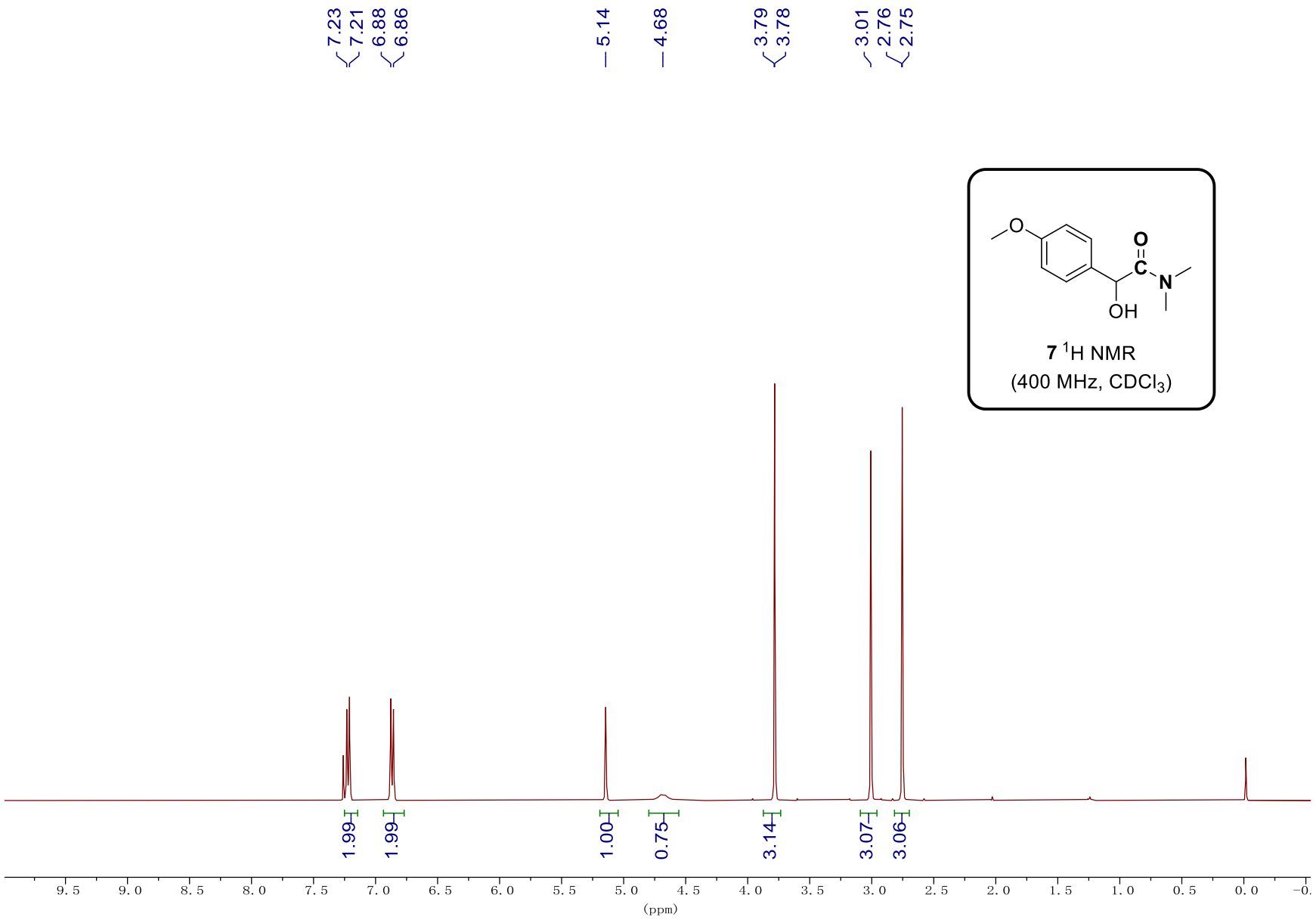
9 ^{1}H, ^{19}F and ^{13}C NMR spectra of products

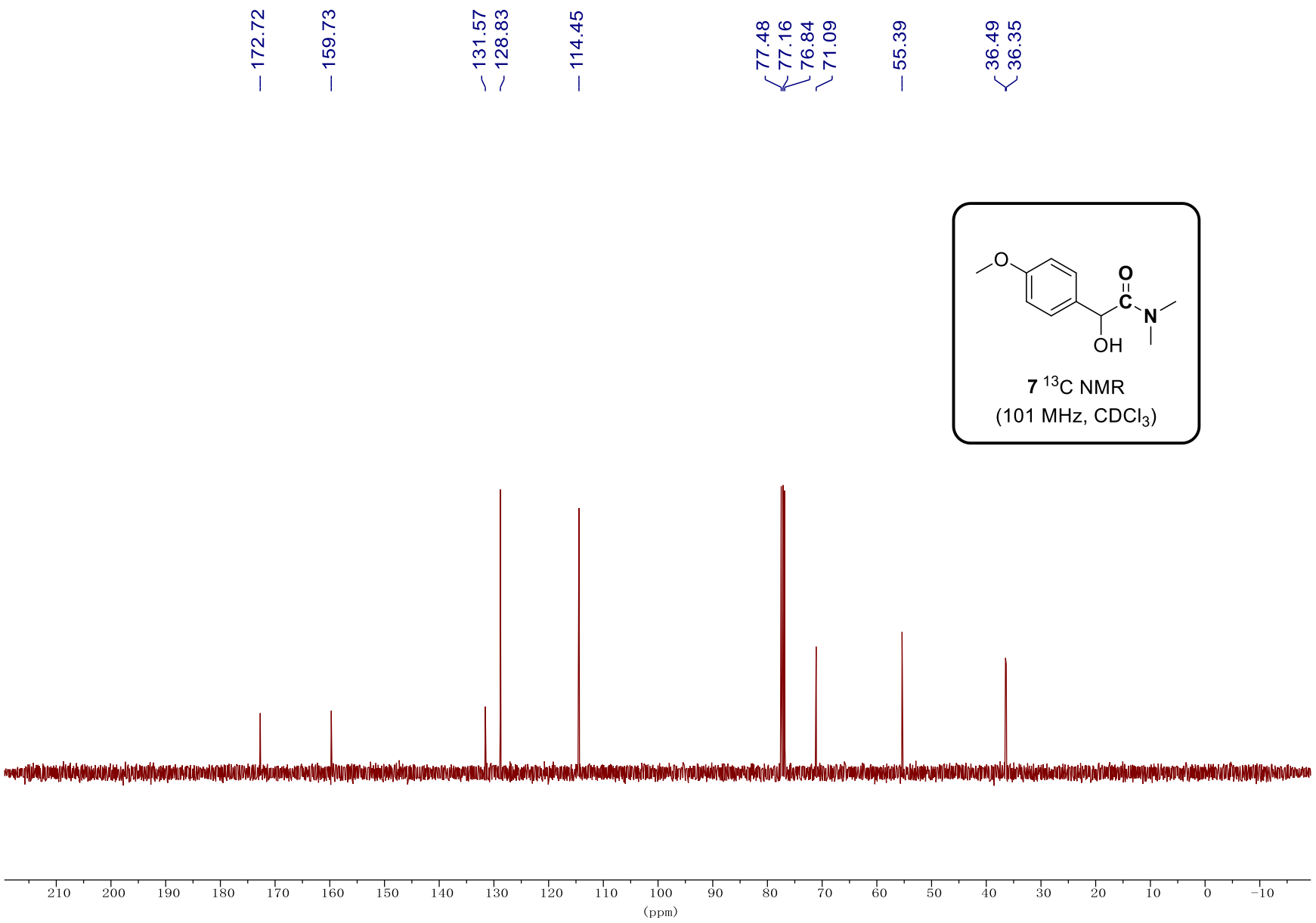

7.21
7.19
7.17
7.15
5.16
4.71
3.02
2.77
2.33
O
C
N
OH
5 1H NMR
(400 MHz, CDCl3)
3.91
1.00
0.76
3.06
3.06
3.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

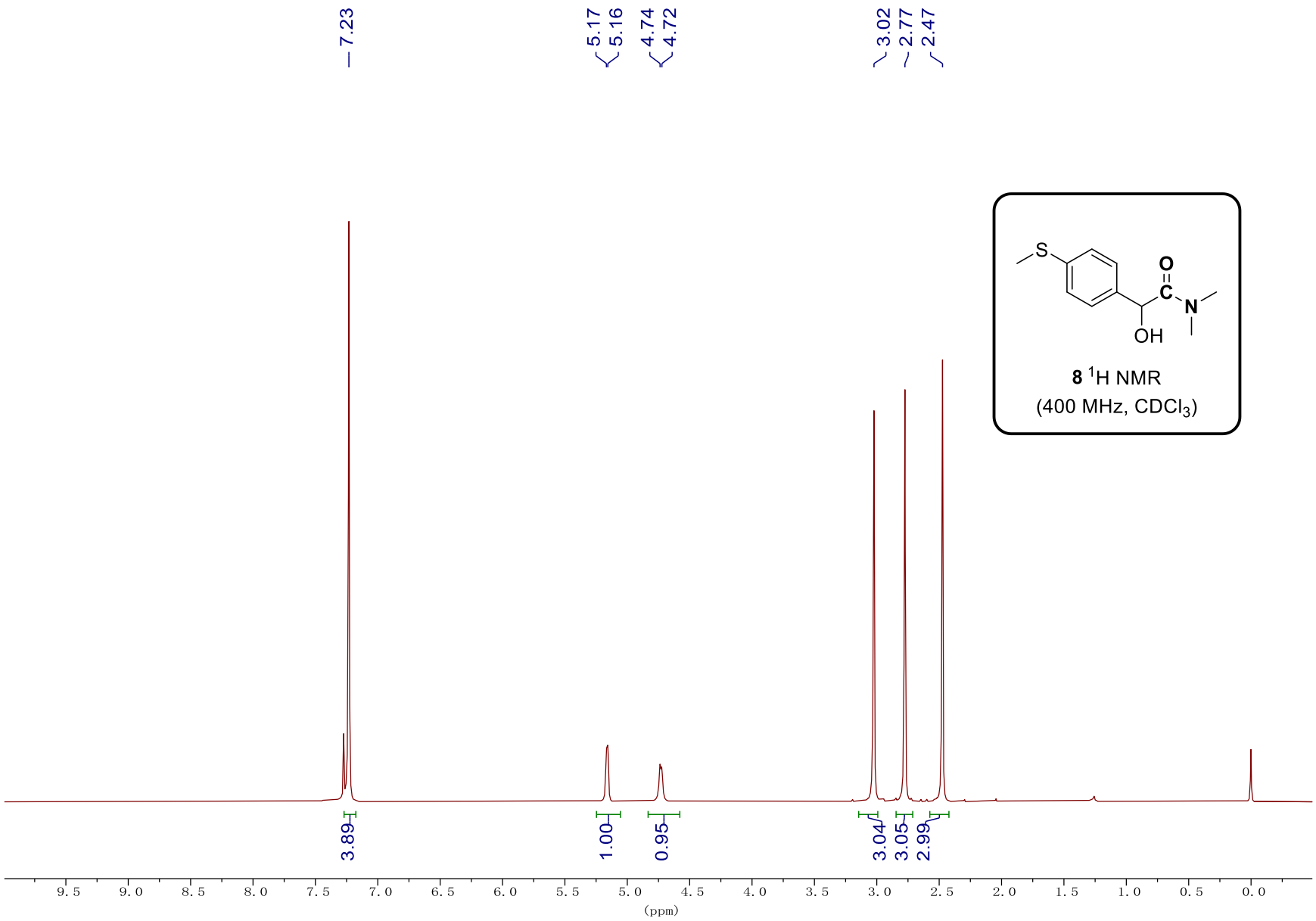

172.64
138.38
136.33
129.76
127.46
77.48
77.16
76.84
71.41
36.51
36.34
21.27
O
C
N
OH
5 13C NMR
(400 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

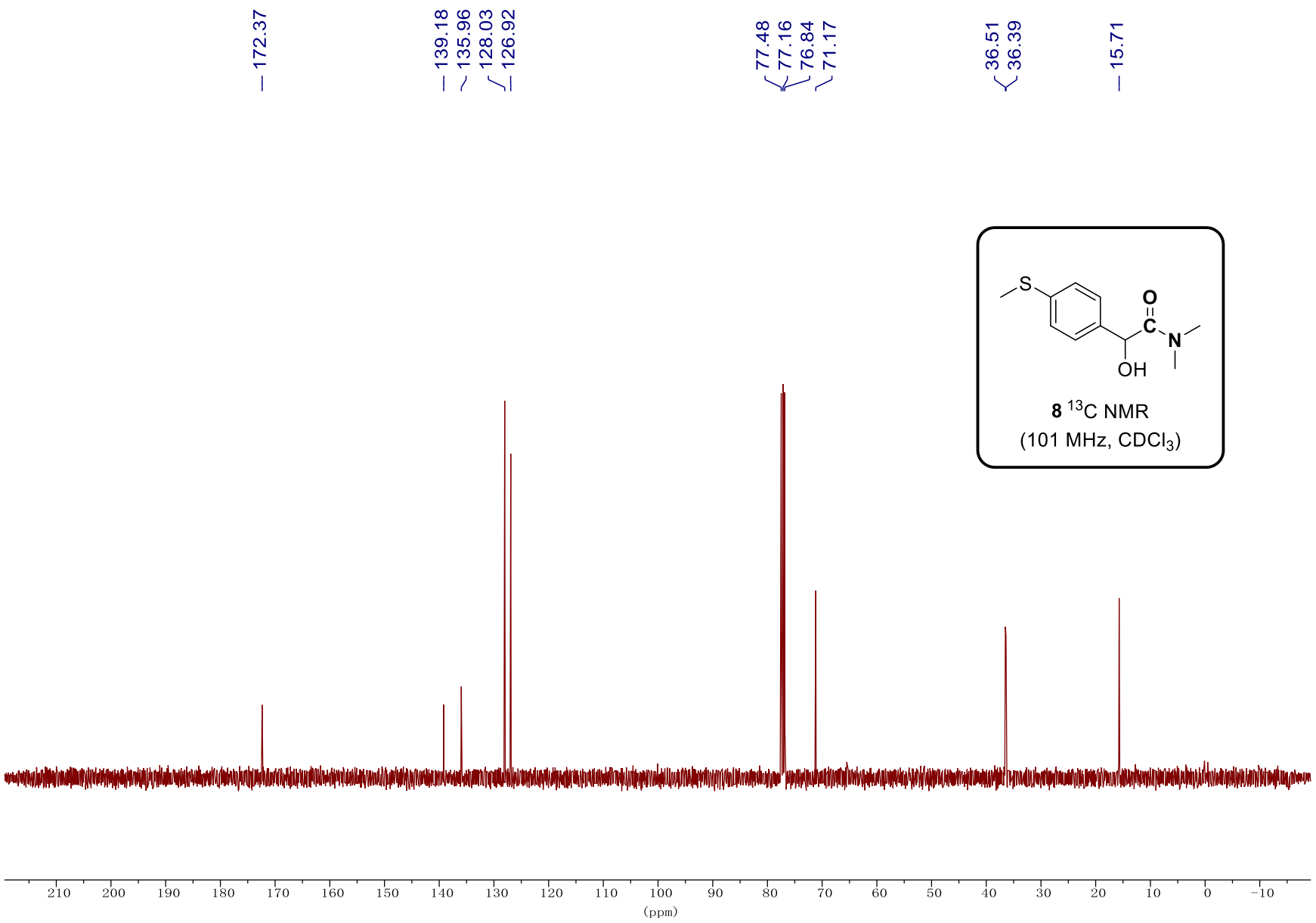

7.38
7.36
7.27
7.25
7.23
5.19
5.18
4.71
3.03
2.78
1.30
O
C
N
OH
6 1H NMR
(400 MHz, CDCl3)
1.93
2.24
1.00
0.78
3.12
3.08
9.41
(ppm)

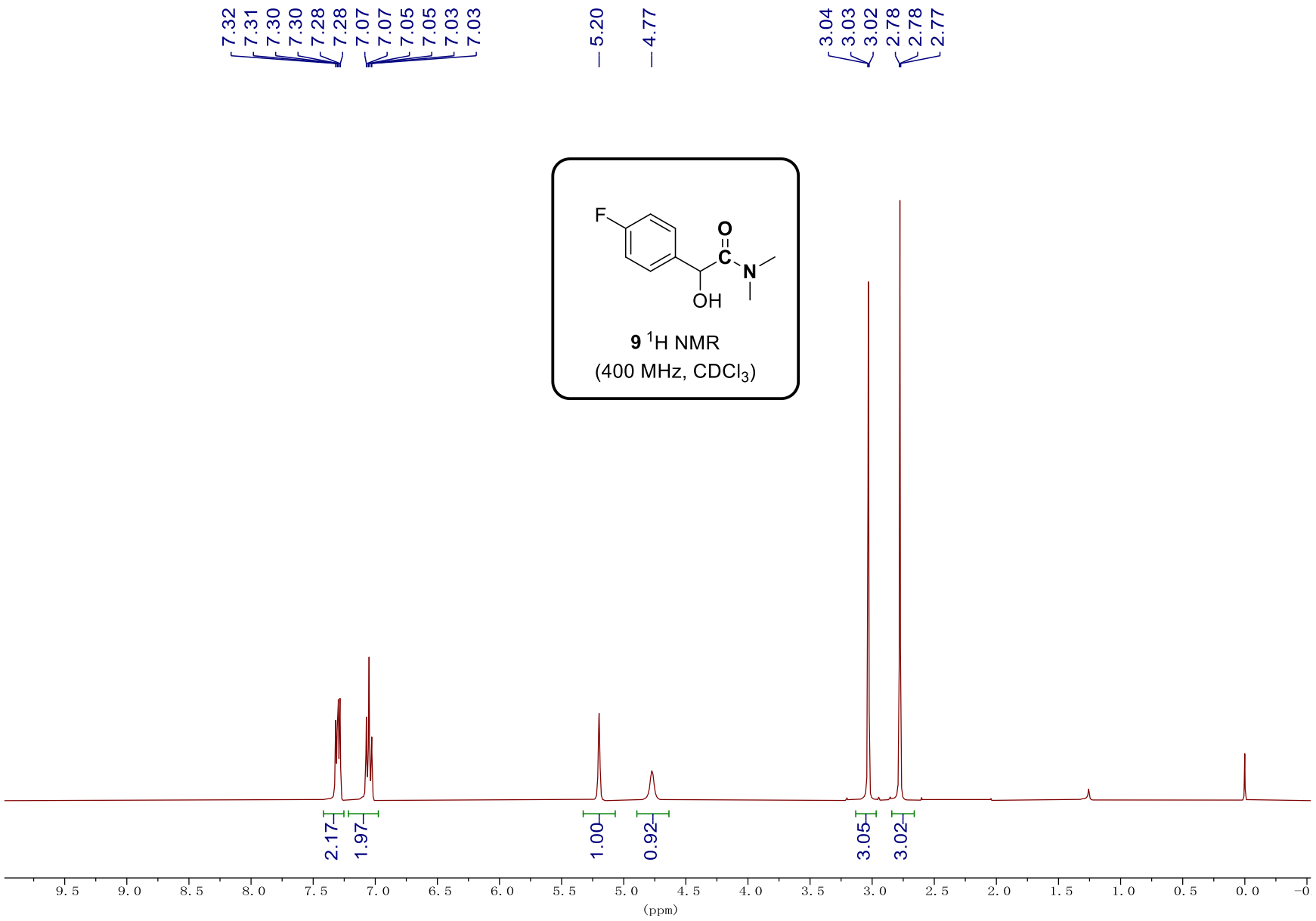

172.66
151.54
136.24
127.19
126.04
77.48
77.16
76.84
71.35
36.56
36.35
34.70
31.39
O
C
N
OH
6 13C NMR
(101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

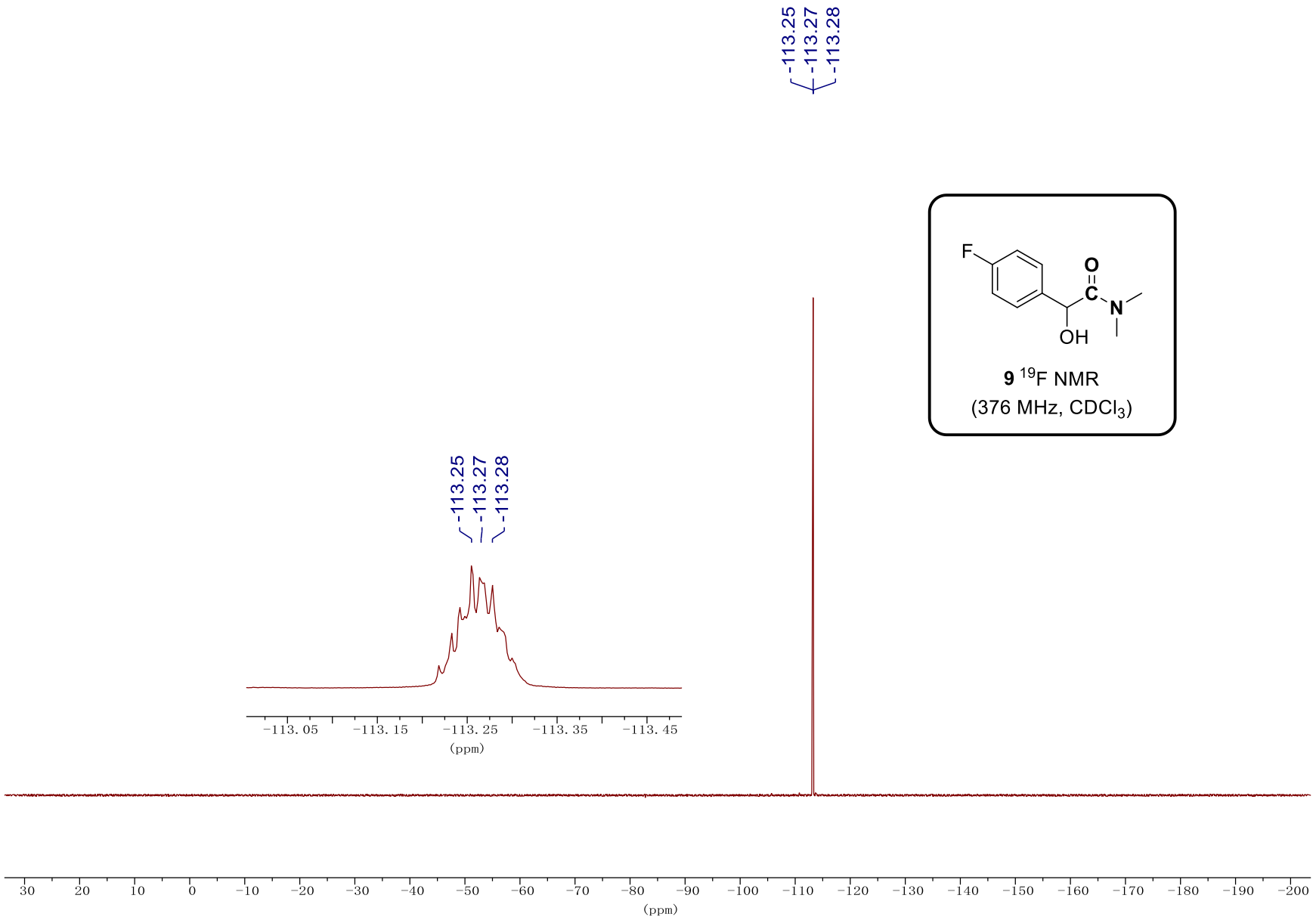

7.23
7.21
6.88
6.86
5.14
4.68
3.79
3.78
3.01
2.76
2.75
O
O
C
N
OH
7 1H NMR
(400 MHz, CDCl3)
1.99
1.99
1.00
0.75
3.14
3.07
3.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

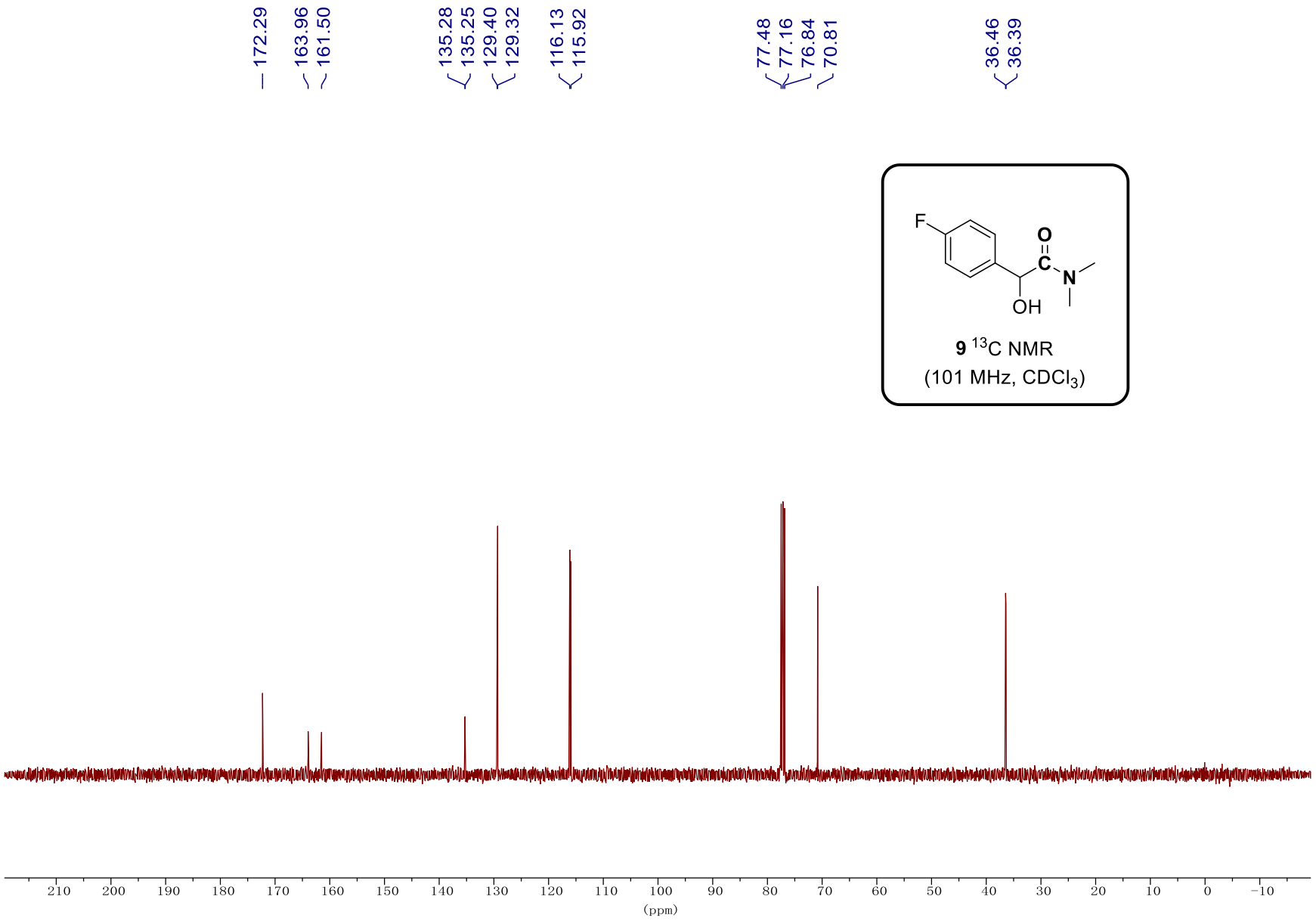

172.72
159.73
131.57
128.83
114.45
77.48
77.16
76.84
71.09
55.39
36.49
36.35
O
O
C
N
OH
7 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

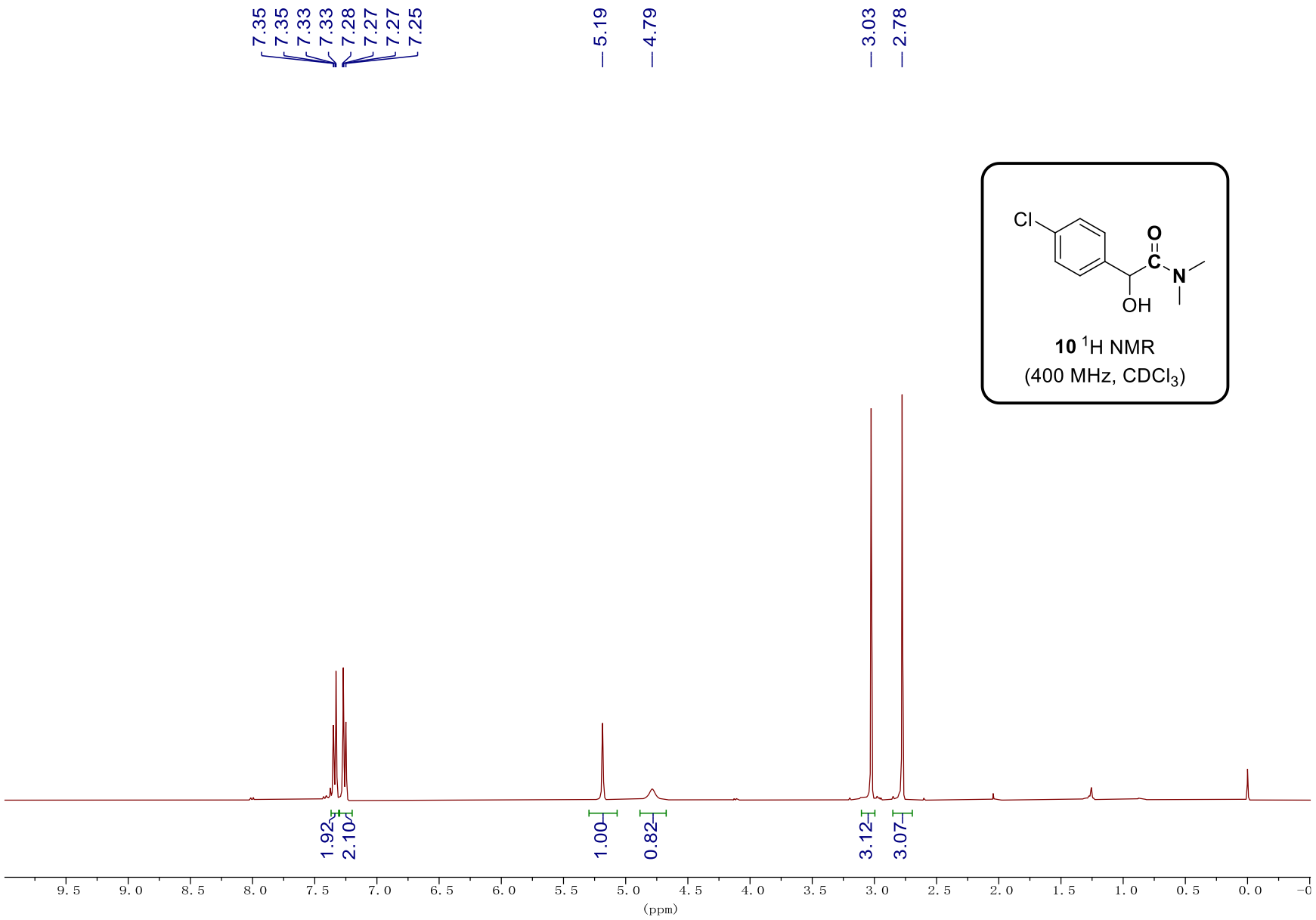

7.23
5.17
5.16
4.74
4.72
3.02
2.77
2.47
S
O
C
N
OH
8 ^{1}H NMR
(400 MHz, $CDCl_3$)
3.89
1.00
0.95
3.04
3.05
2.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

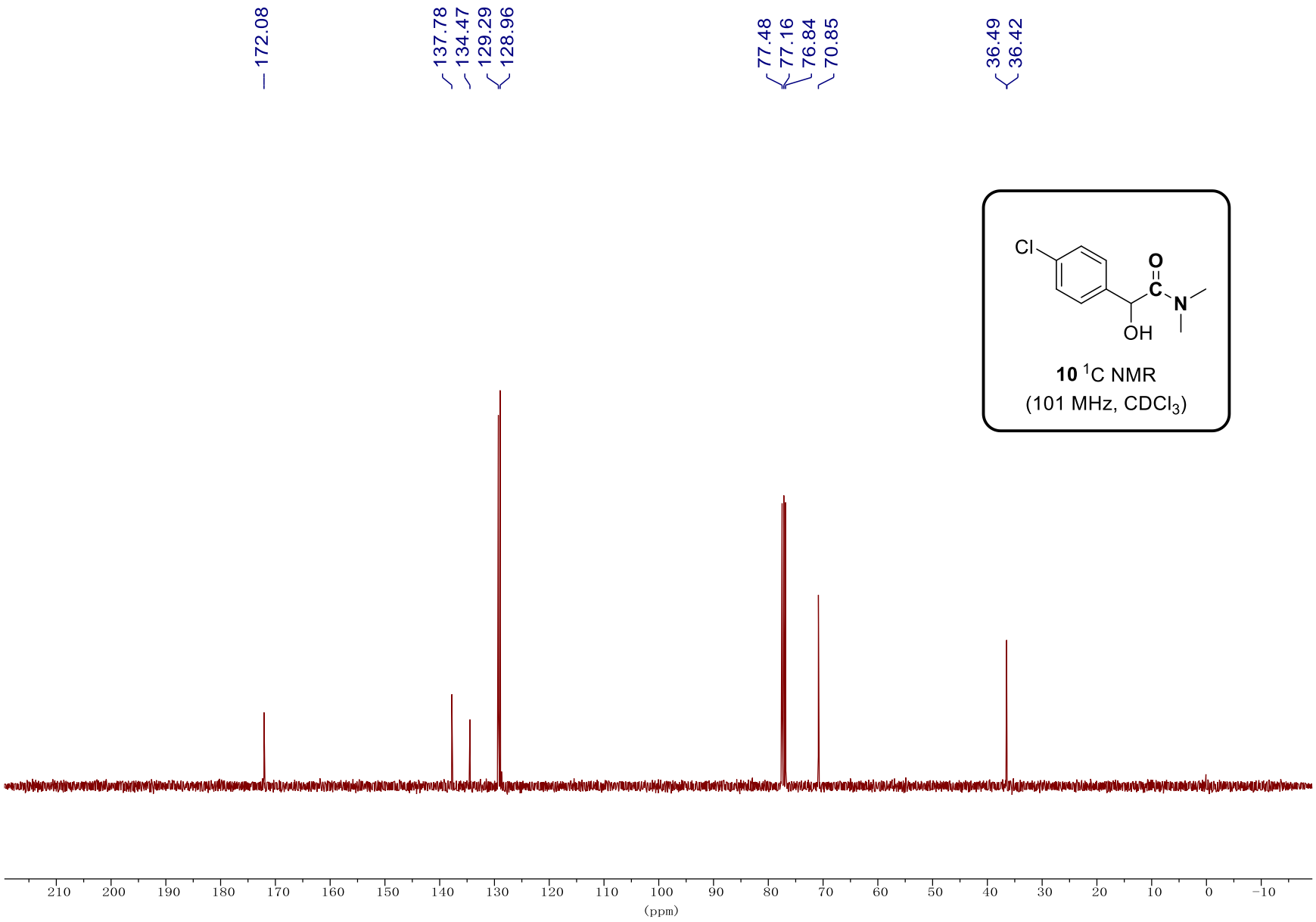

172.37
139.18
135.96
128.03
126.92
77.48
77.16
76.84
71.17
36.51
36.39
15.71
S
O
C
N
OH
8 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

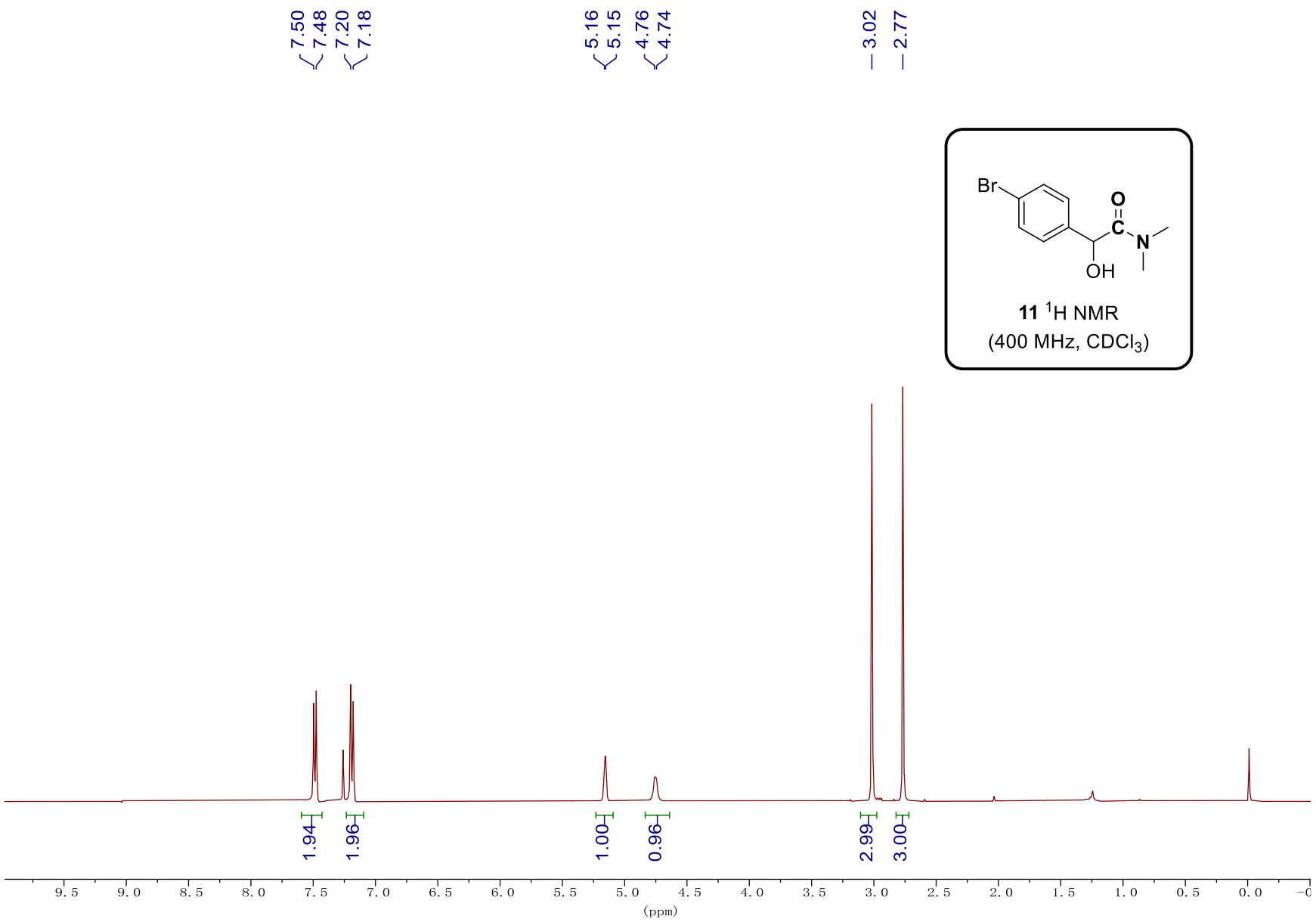

7.32
7.31
7.30
7.30
7.28
7.28
7.07
7.07
7.05
7.05
7.03
7.03
5.20
4.77
3.04
3.03
3.02
2.78
2.78
2.77
F
O
C
N
OH
9 1H NMR
(400 MHz, CDCl3)
2.17
1.97
1.00
0.92
3.05
3.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

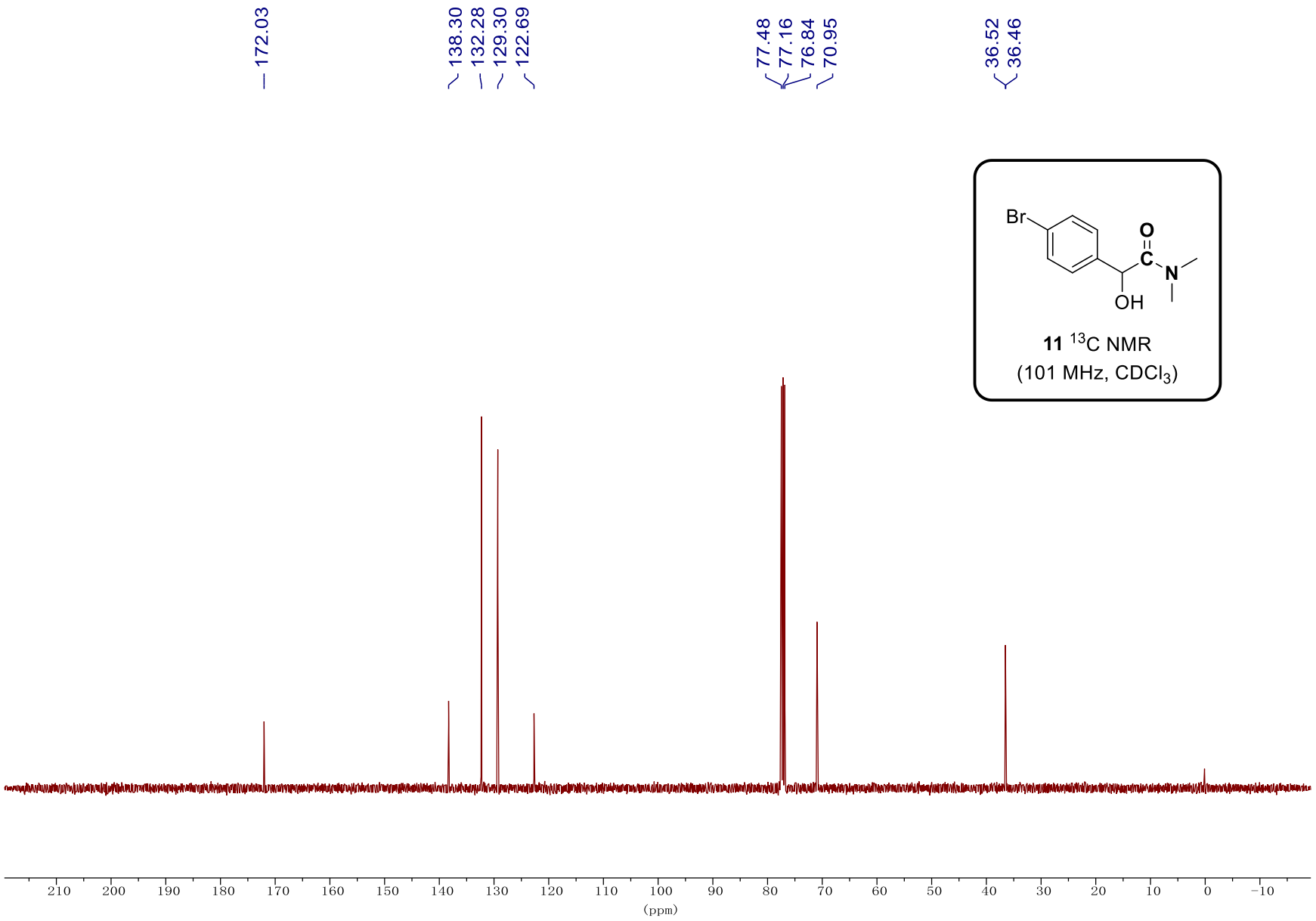

-113.25
-113.27
-113.28
F
O
C
N
OH
9 19F NMR
(376 MHz, CDCl3)
-113.25
-113.27
-113.28
-113.05
-113.15
-113.25
-113.35
-113.45
(ppm)
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
(ppm)

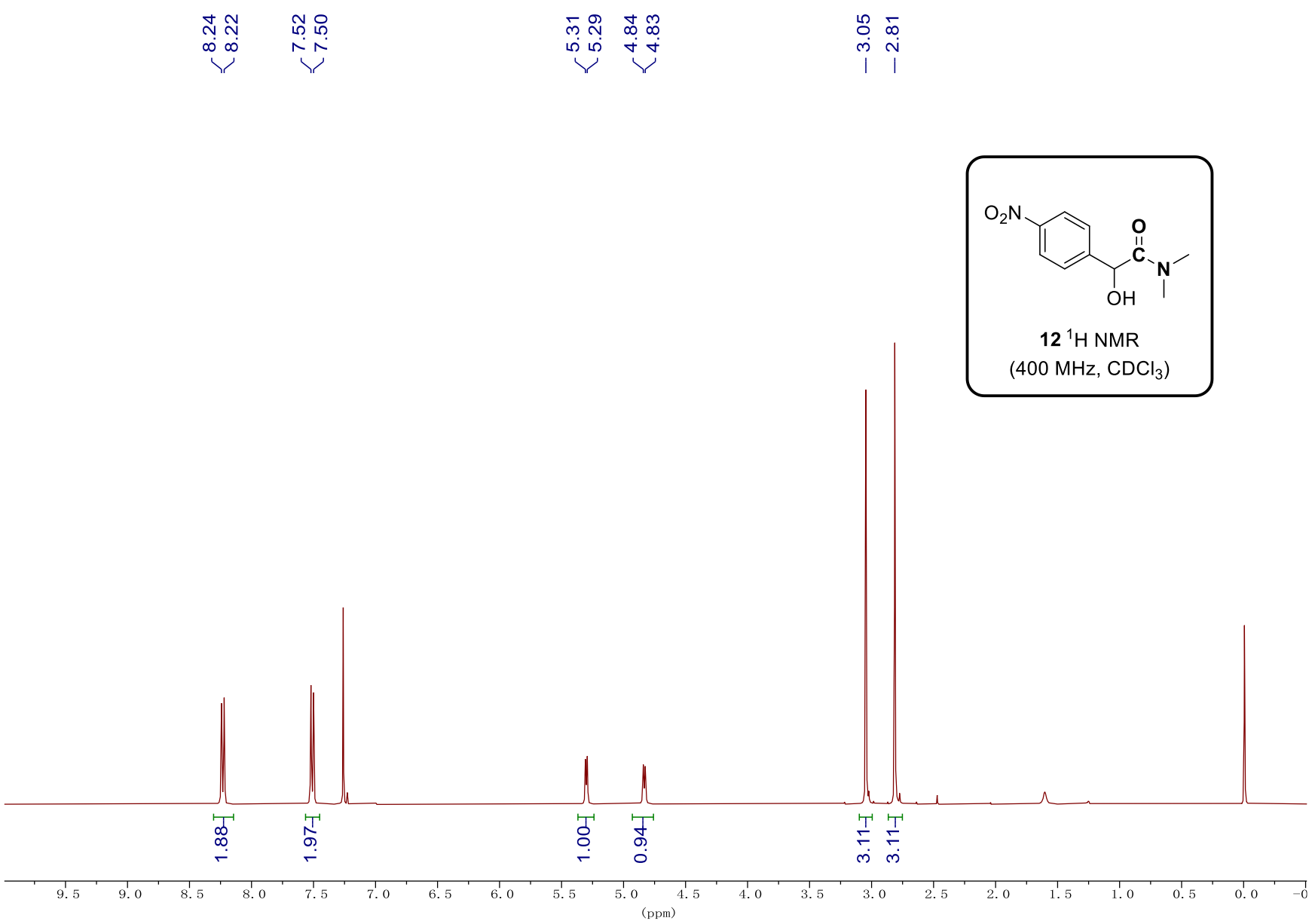

172.29
163.96
161.50
135.28
135.25
129.40
129.32
116.13
115.92
77.48
77.16
76.84
70.81
36.46
36.39
F
O
C
N
OH
9 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

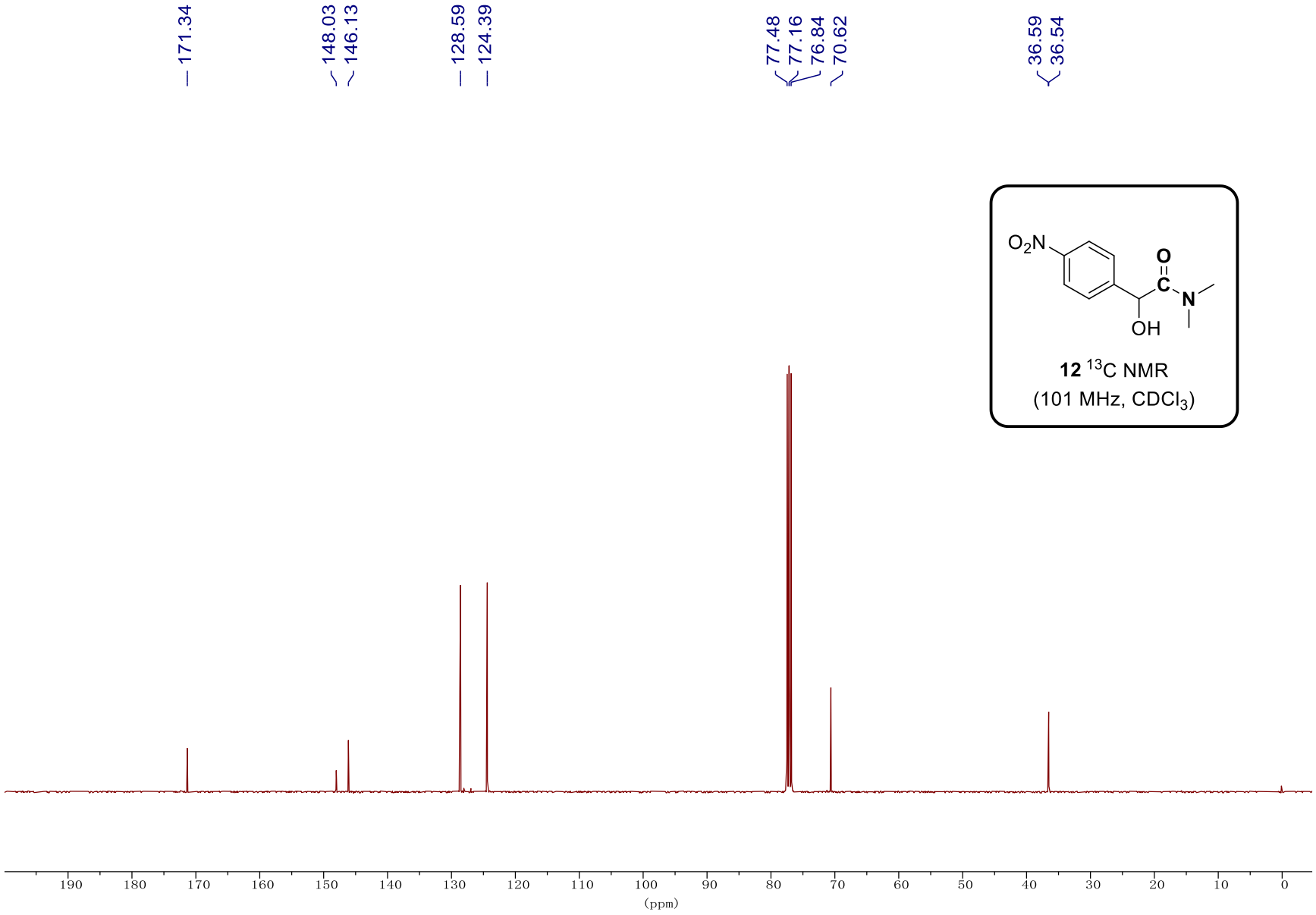

7.35
7.35
7.33
7.33
7.28
7.27
7.27
7.25
5.19
4.79
3.03
2.78
Cl
O
C
N
OH
10 1H NMR
(400 MHz, CDCl3)
1.92
2.10
1.00
0.82
3.12
3.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

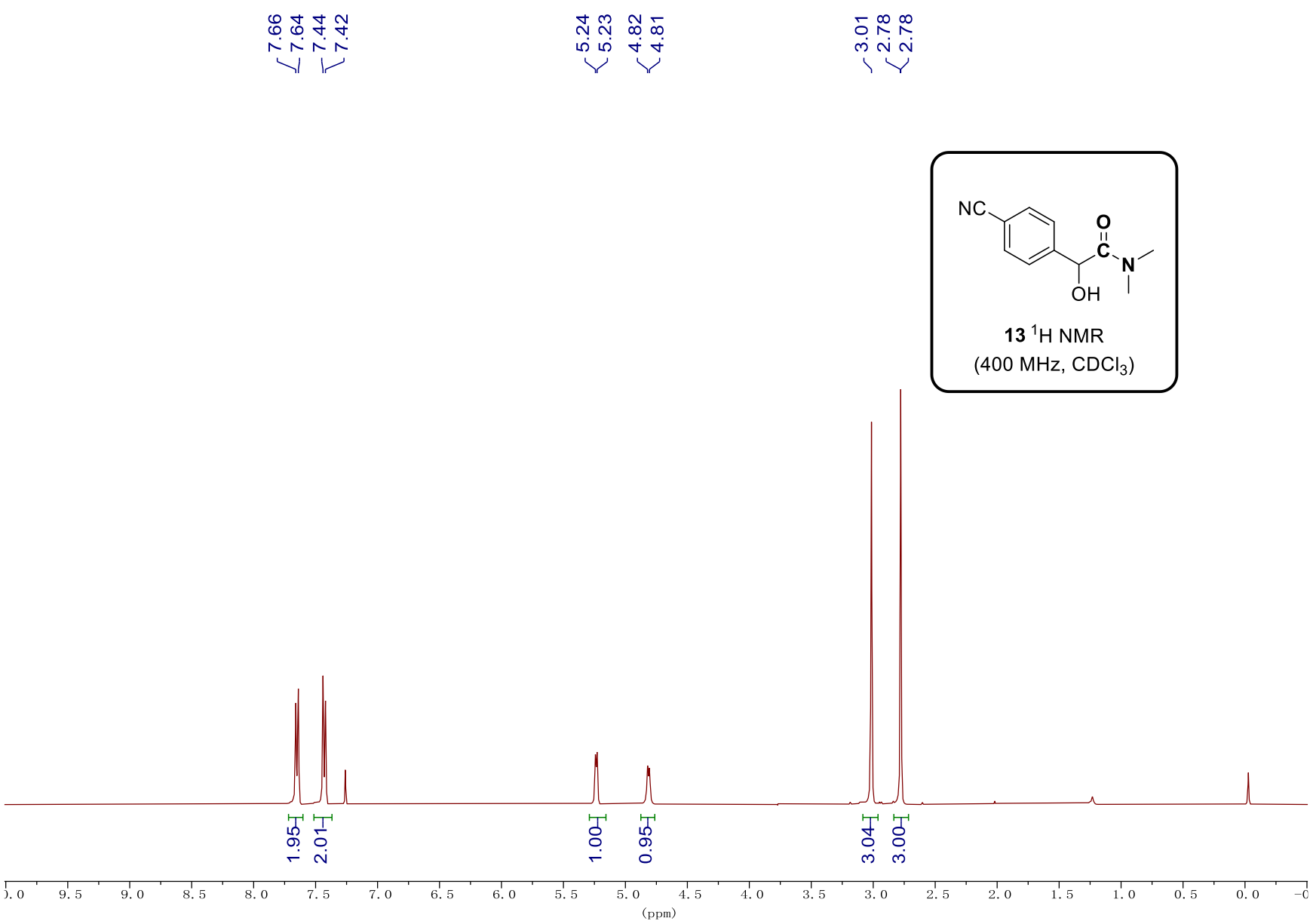

172.08
137.78
134.47
129.29
128.96
77.48
77.16
76.84
70.85
36.49
36.42
Cl
O
C
N
OH
10 1C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

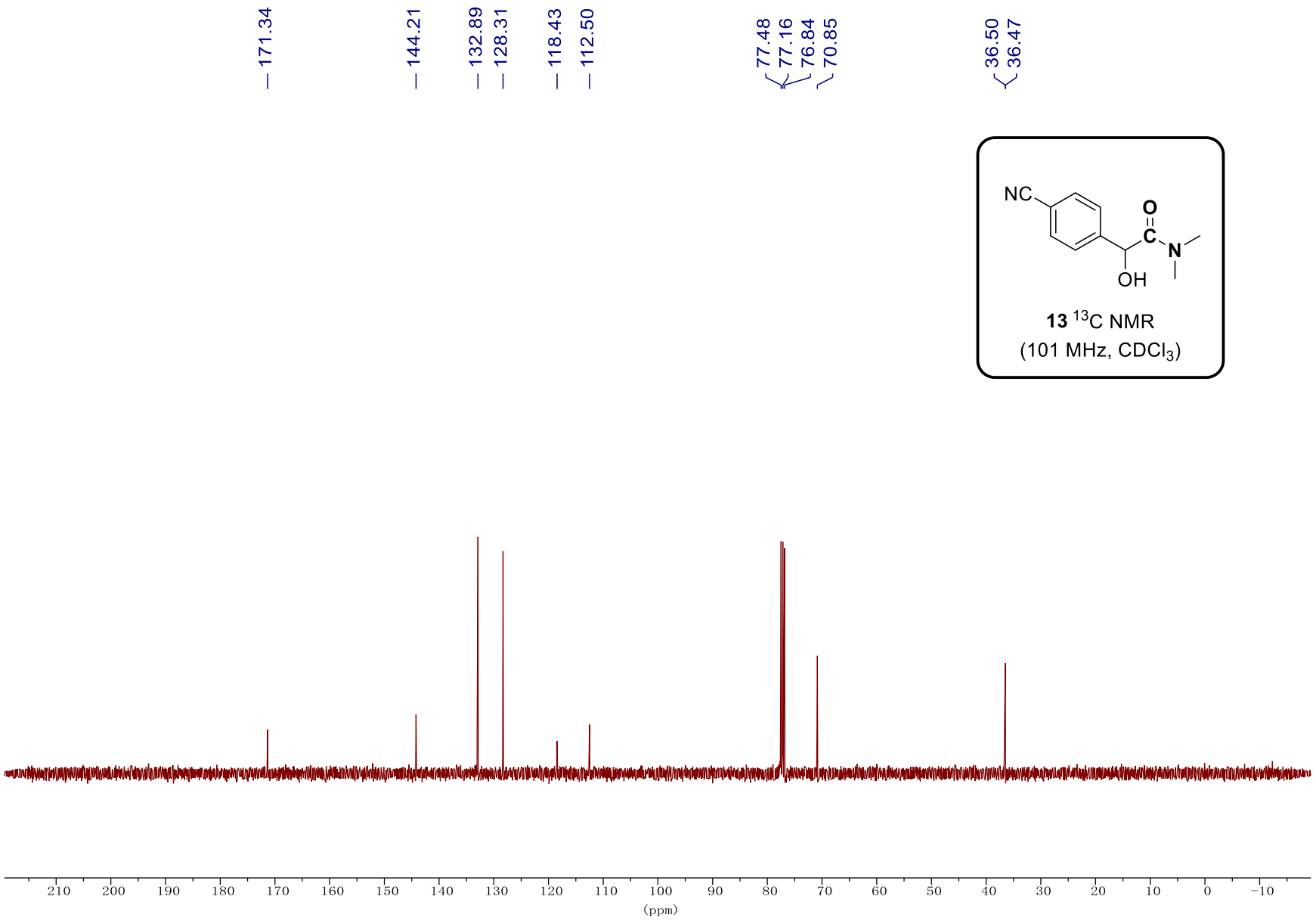

7.50
7.48
7.20
7.18
5.16
5.15
4.76
4.74
3.02
2.77
Br
O
C
N
OH
11 1H NMR
(400 MHz, CDCl3)
1.94
1.96
1.00
0.96
2.99
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

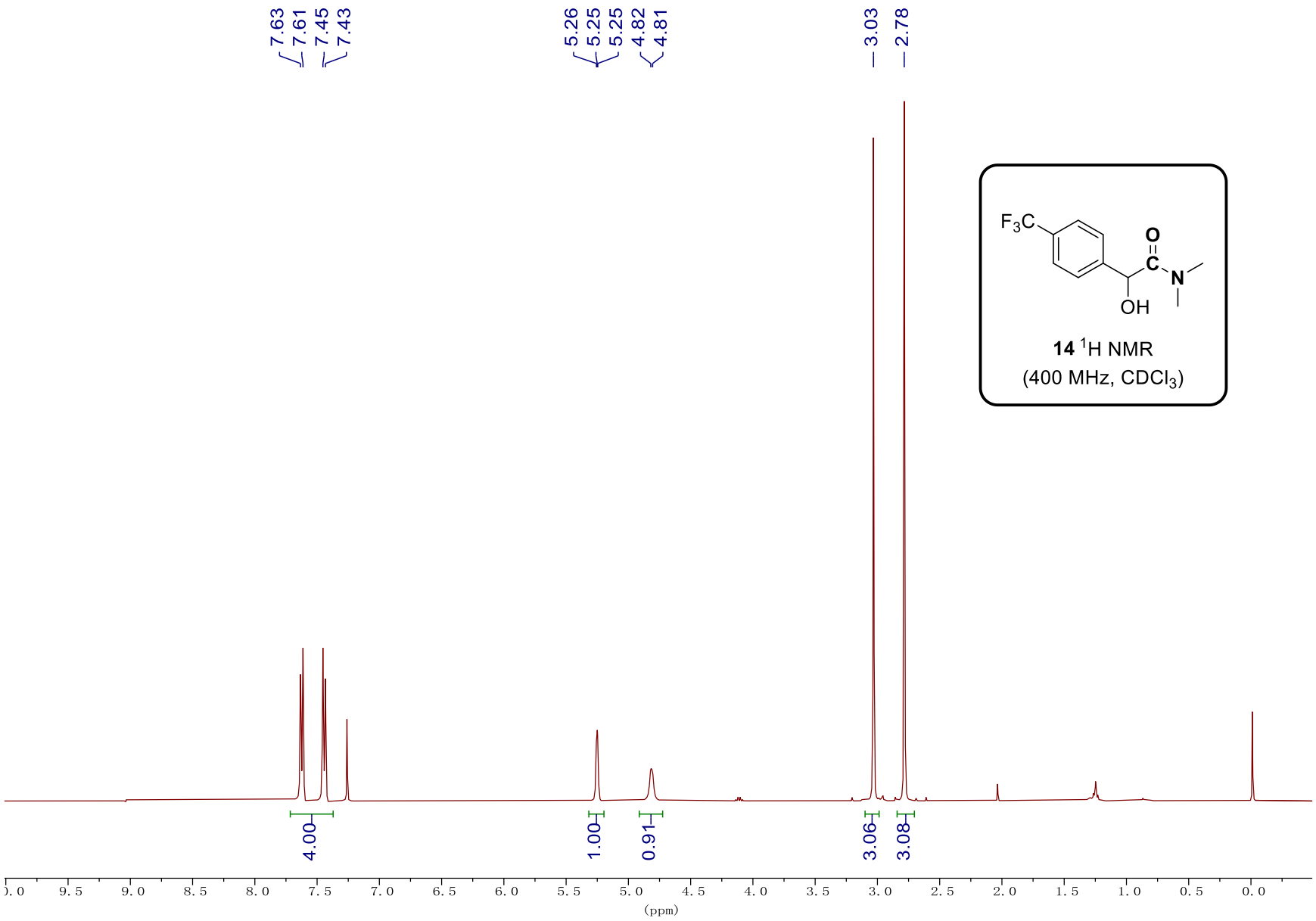

172.03
138.30
132.28
129.30
122.69
77.48
77.16
76.84
70.95
36.52
36.46
Br
O
C
N
OH
11 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

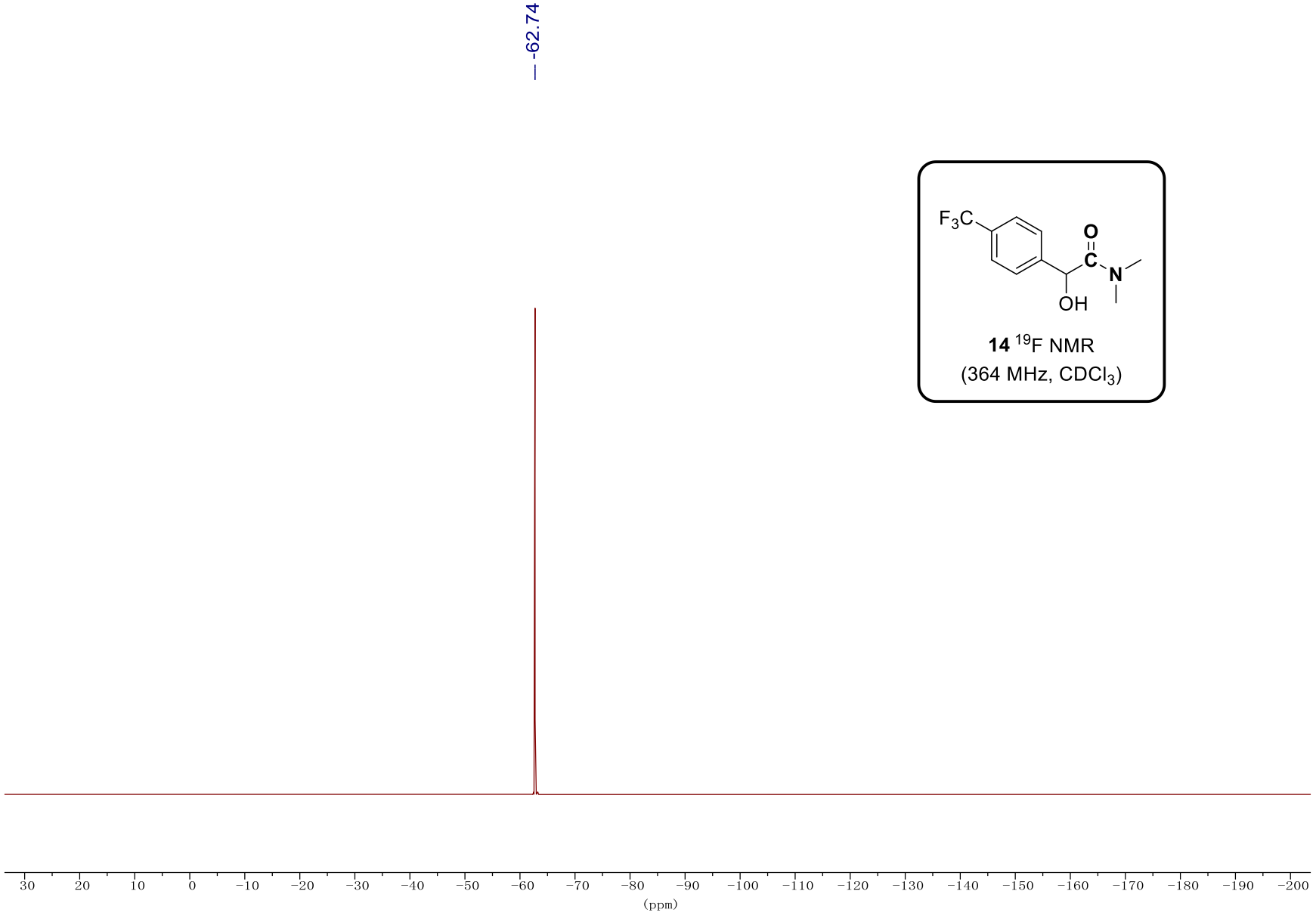

8.24
8.22
7.52
7.50
5.31
5.29
4.84
4.83
3.05
2.81
O2N
O
C
N
OH
12 1H NMR
(400 MHz, CDCl3)
1.88
1.97
1.00
0.94
3.11
3.11
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

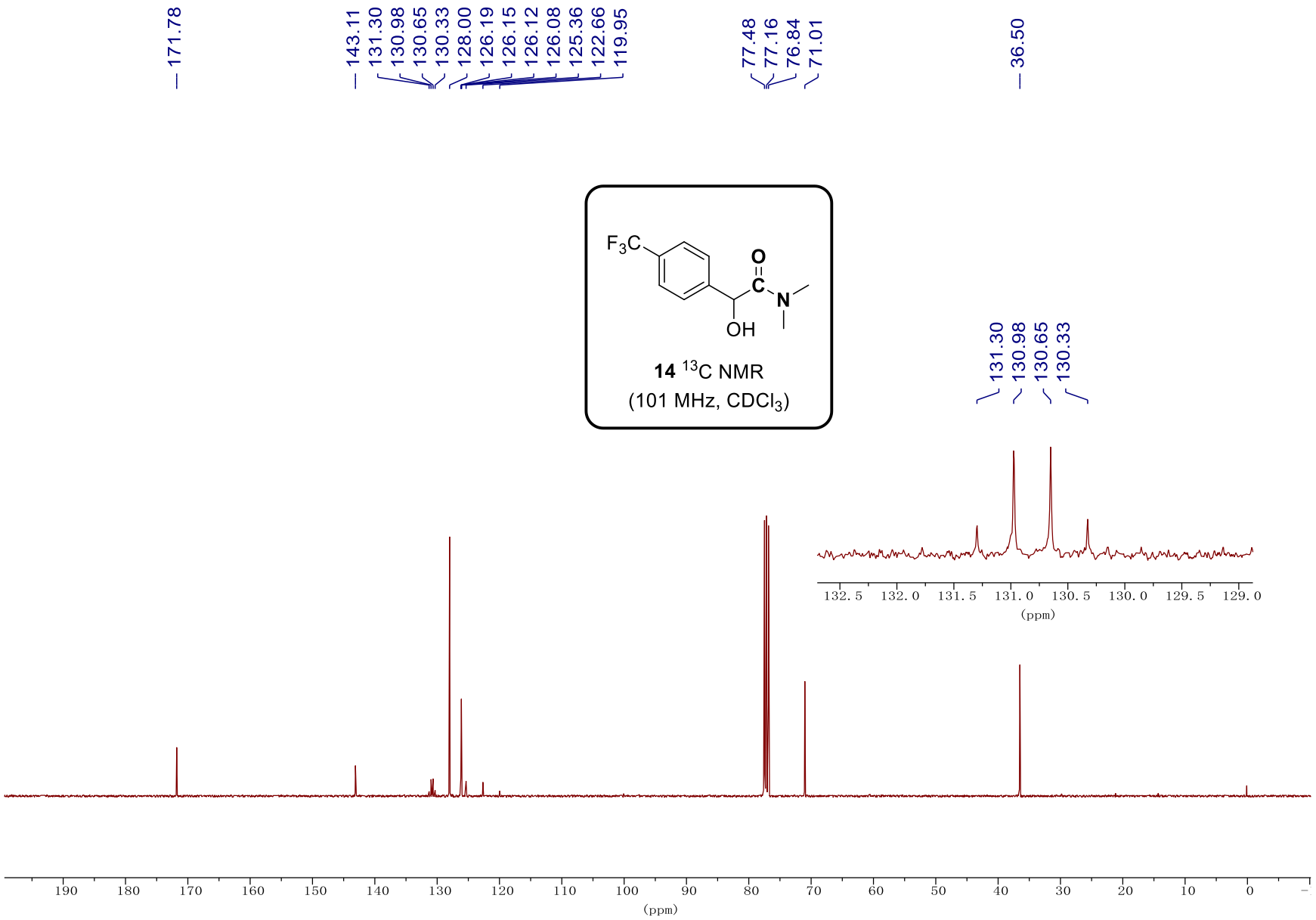

171.34
148.03
146.13
128.59
124.39
77.48
77.16
76.84
70.62
36.59
36.54
O2N
O
C
N
OH
12 13C NMR
(101 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

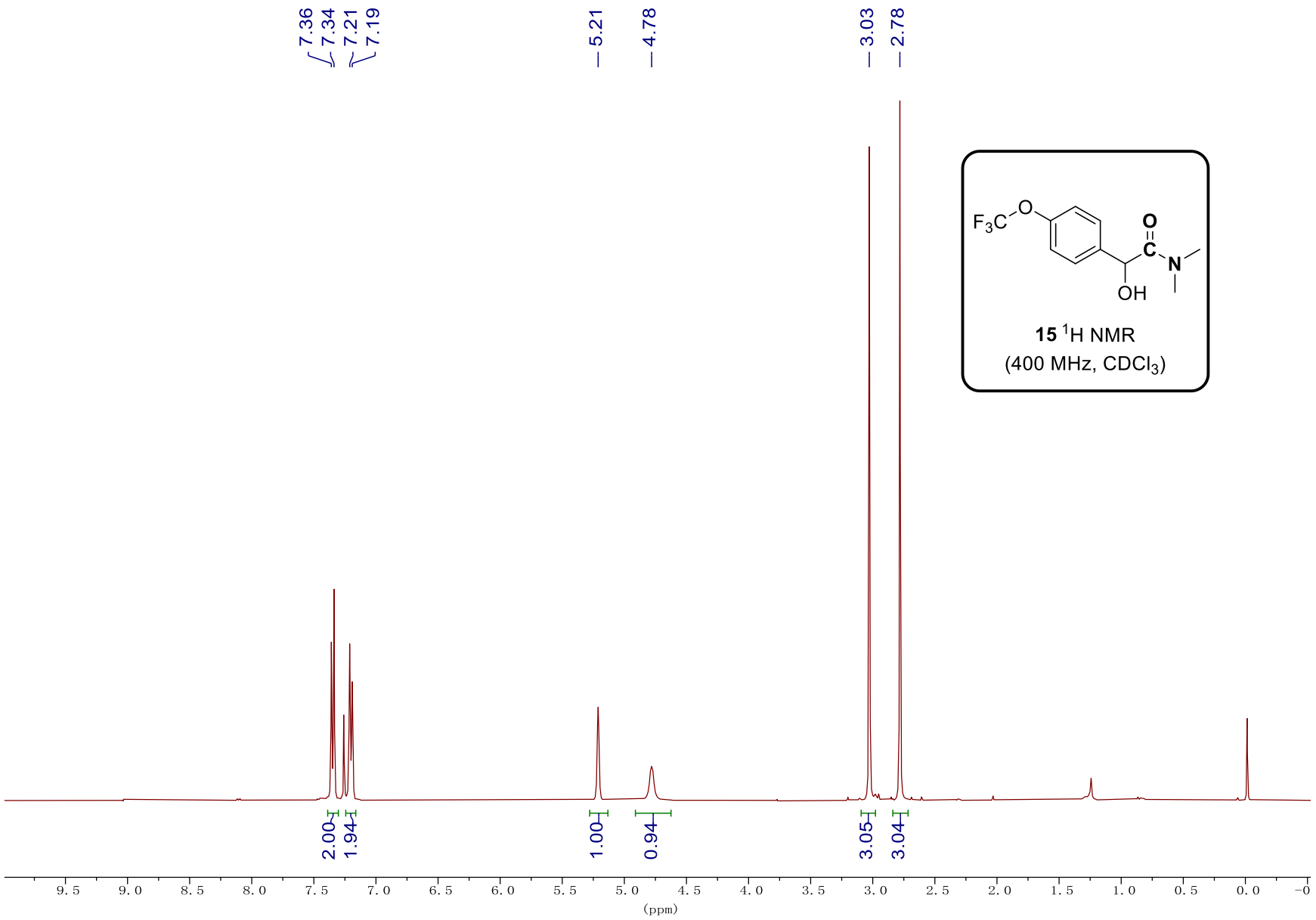

7.66
7.64
7.44
7.42
5.24
5.23
4.82
4.81
3.01
2.78
2.78
NC
O
C
N
OH
13 1H NMR
(400 MHz, CDCl3)
1.95
2.01
1.00
0.95
3.04
3.00
(ppm)

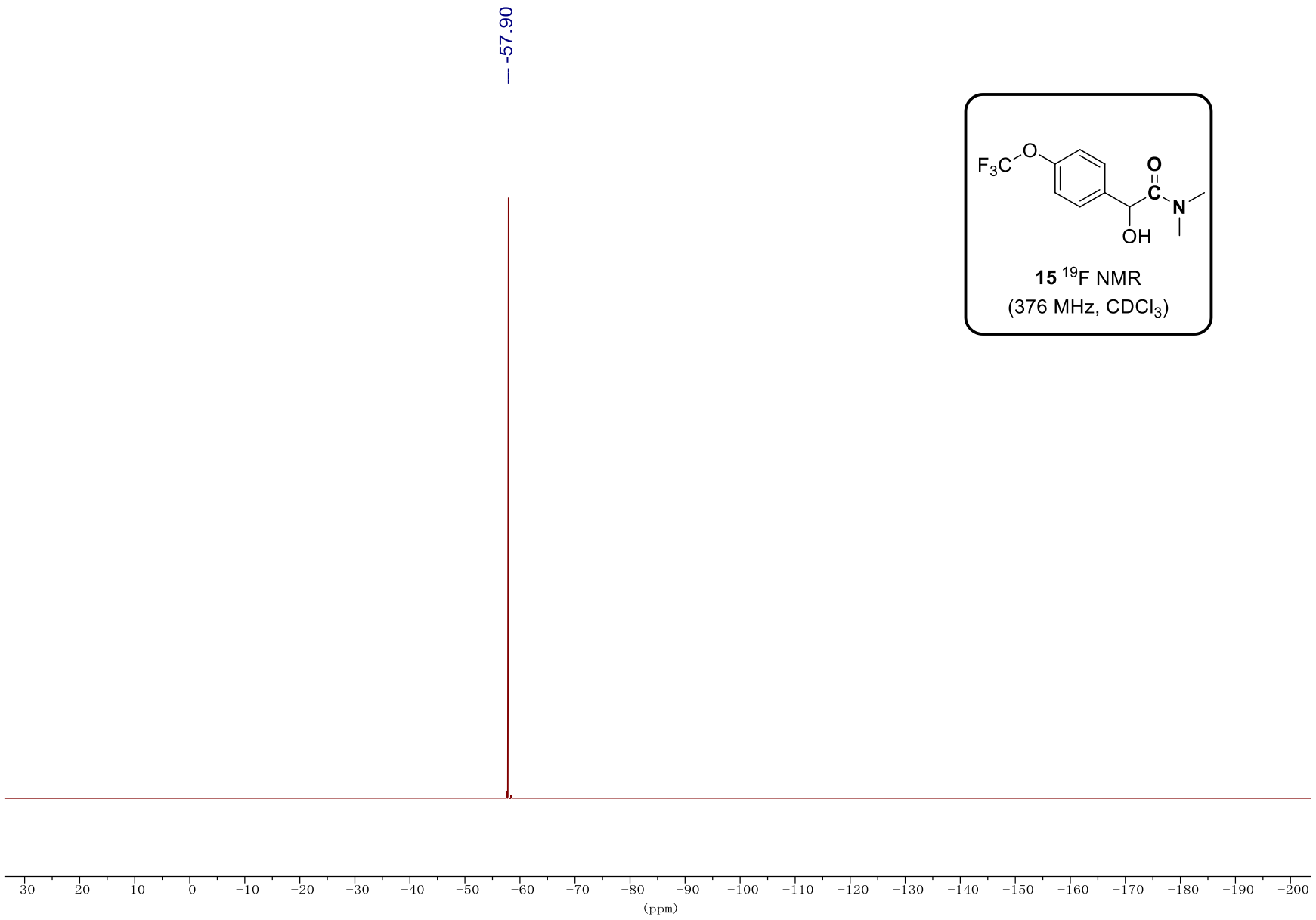

171.34
144.21
132.89
128.31
118.43
112.50
77.48
77.16
76.84
70.85
36.50
36.47
NC
O
C
N
OH
13 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

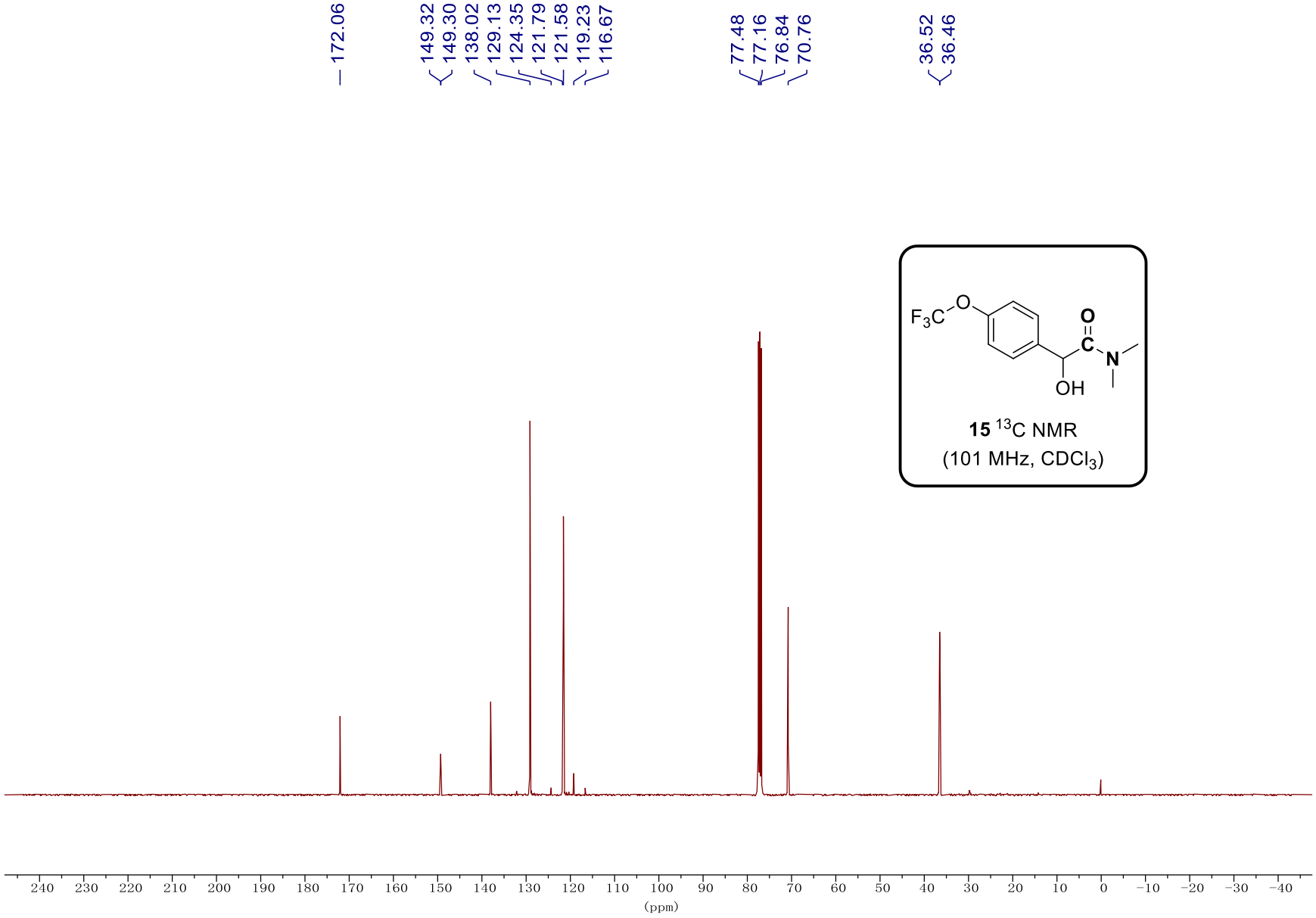

7.63
7.61
7.45
7.43
5.26
5.25
5.25
4.82
4.81
3.03
2.78
F3C
O
C
N
OH
14 1H NMR
(400 MHz, CDCl3)
4.00
1.00
0.91
3.06
3.08
(ppm)

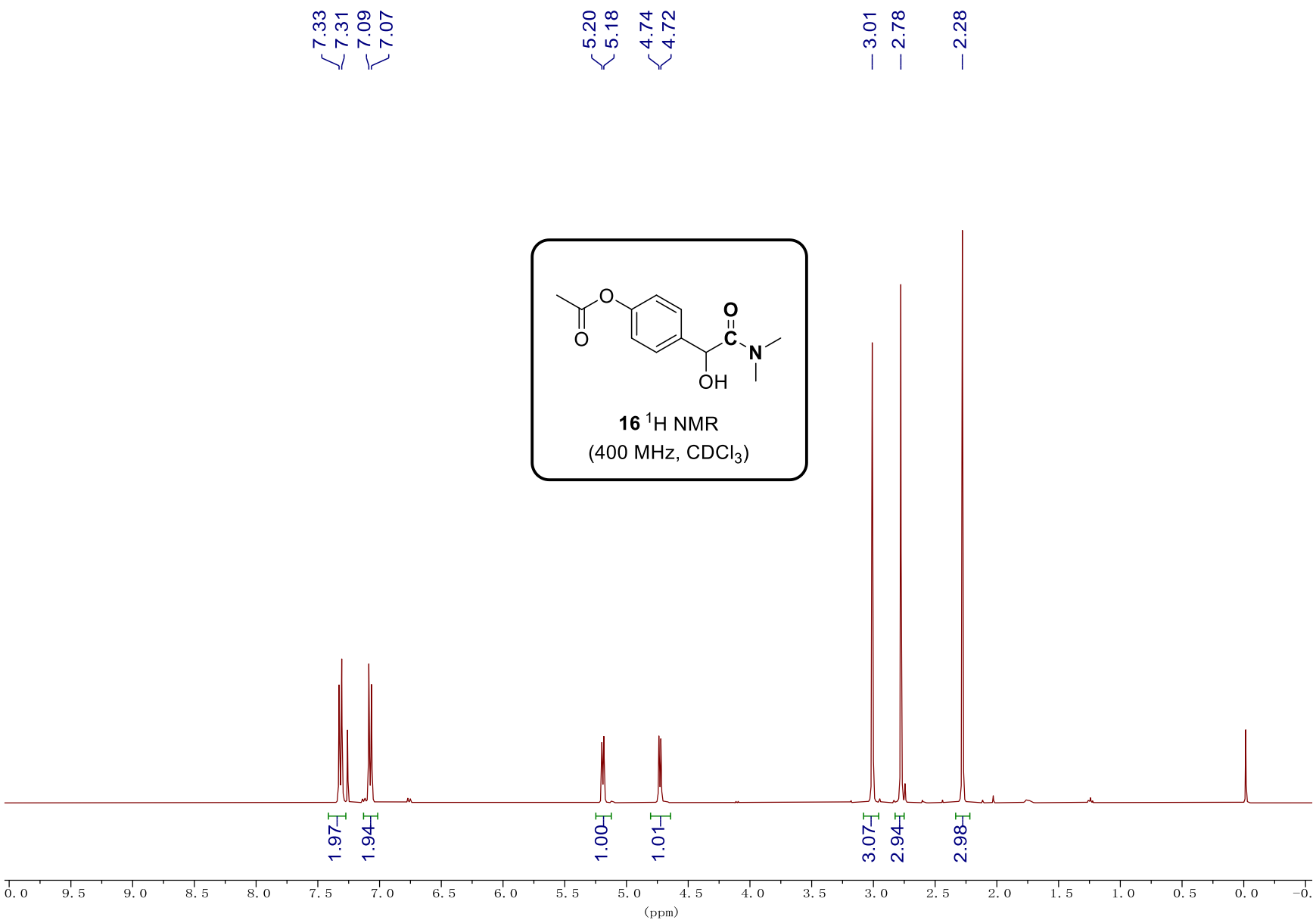

-62.74
F3C
O
C
N
OH
14 19F NMR
(364 MHz, CDCl3)
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
(ppm)

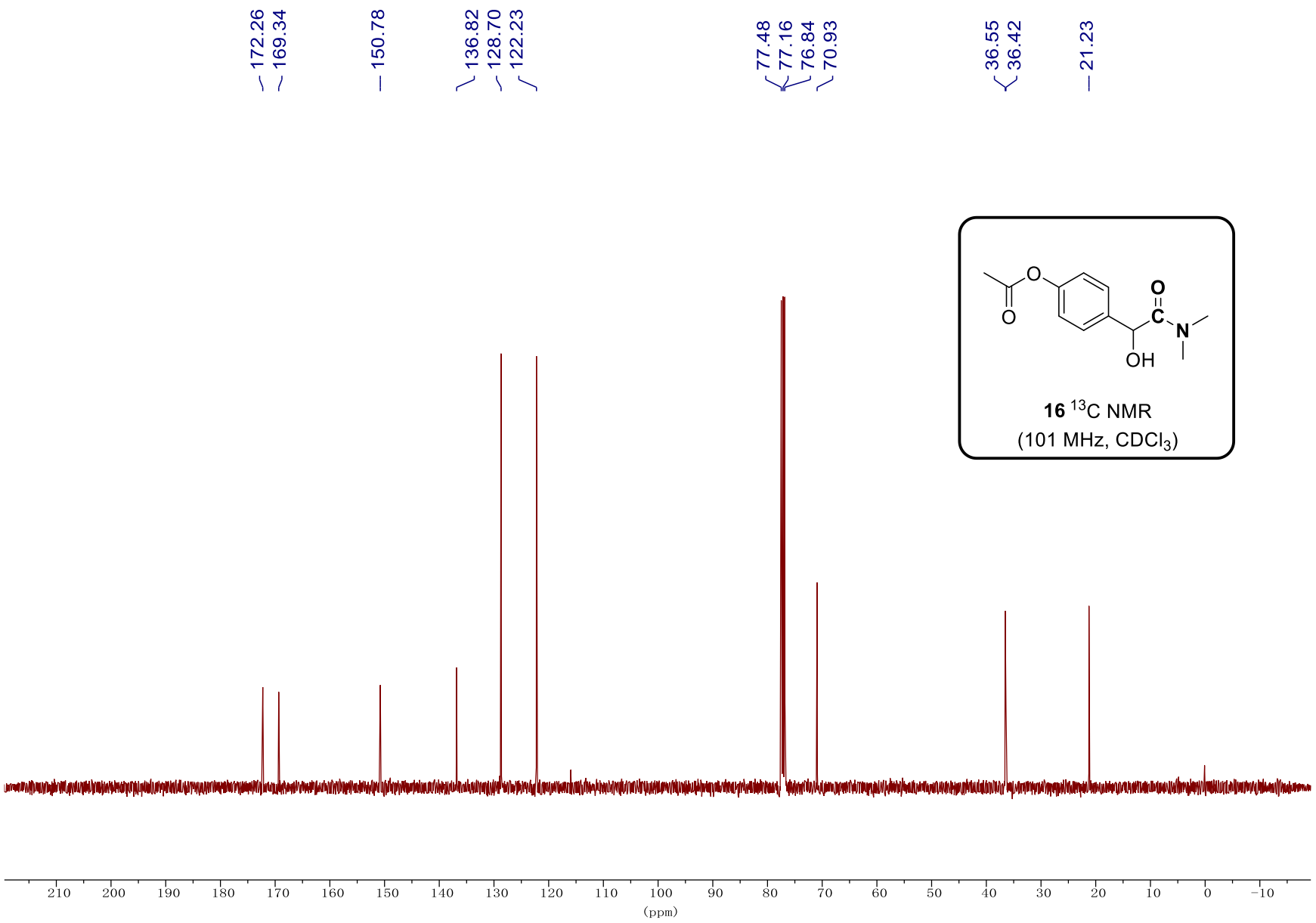

171.78
143.11
131.30
130.98
130.65
130.33
128.00
126.19
126.15
126.12
126.08
125.36
122.66
119.95
77.48
77.16
76.84
71.01
36.50
F3C
O
C
N
OH
14 13C NMR
(101 MHz, CDCl3)
131.30
130.98
130.65
130.33
132.5
132.0
131.5
131.0
130.5
130.0
129.5
129.0
(ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

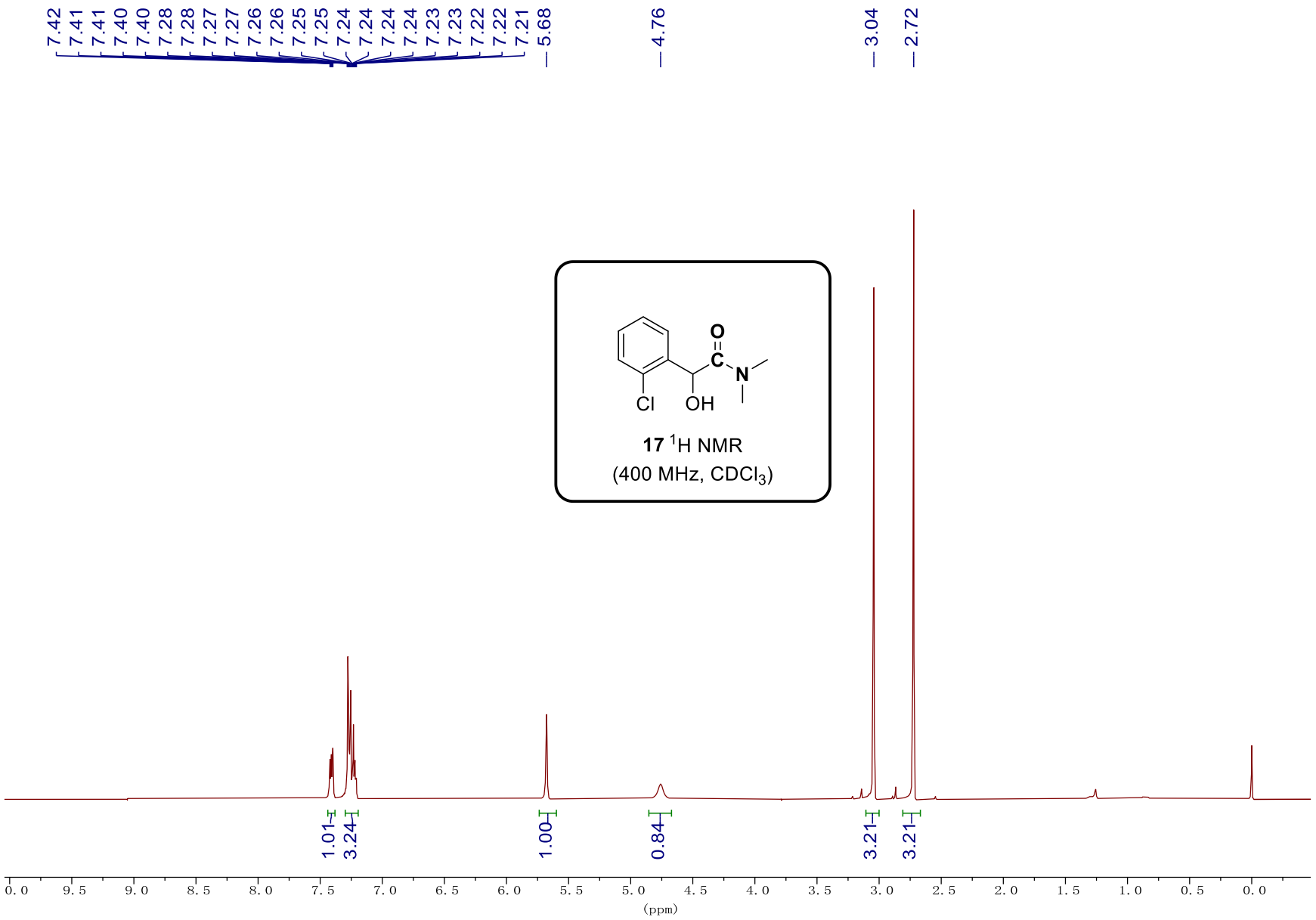

7.36
7.34
7.21
7.19
5.21
4.78
3.03
2.78
2.00
1.94
1.00
0.94
3.05
3.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0
(ppm)
F3C
O
O
C
N
OH
15 1H NMR
(400 MHz, CDCl3)

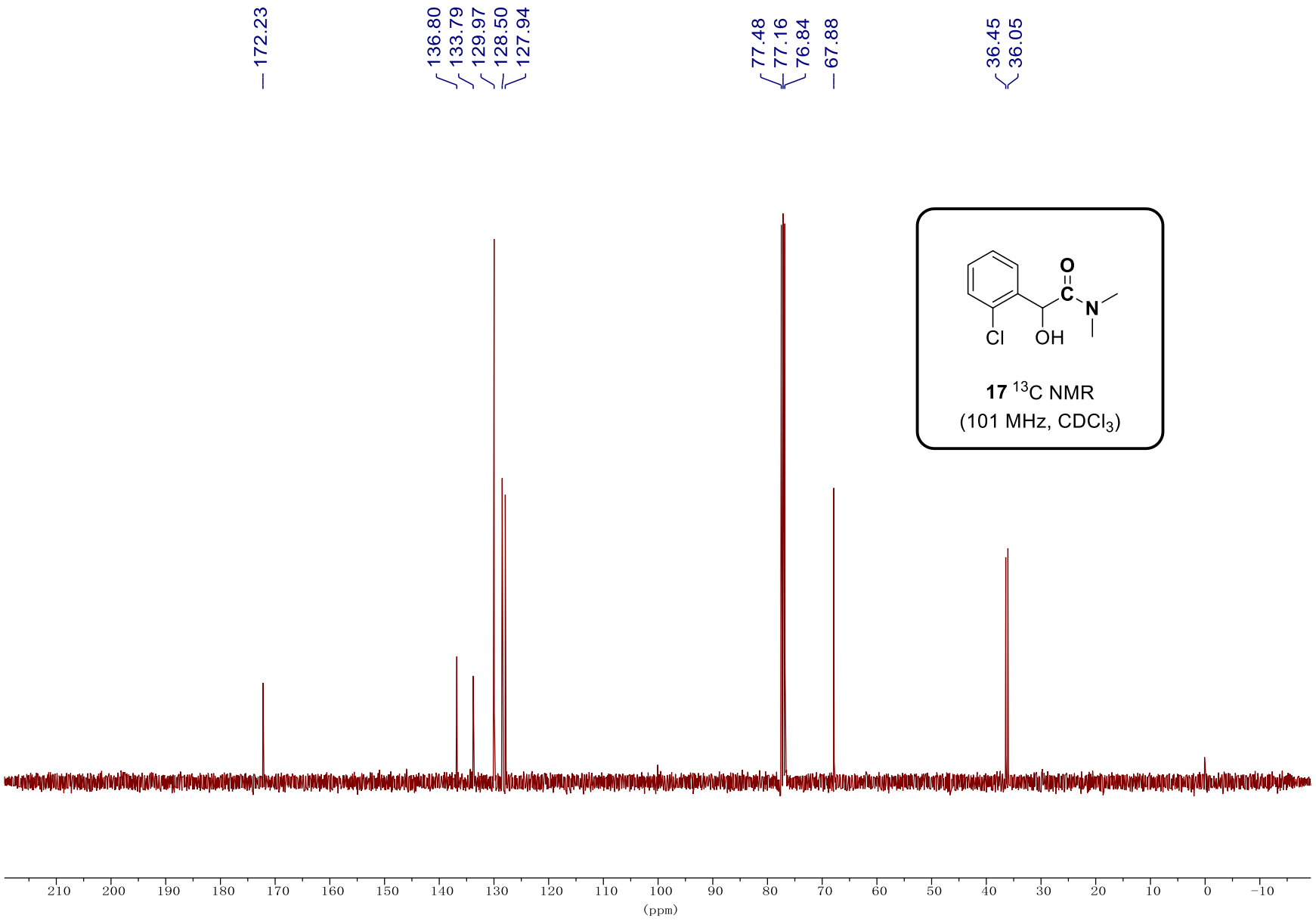

-57.90
F3C
O
O
C
N
OH
15 19F NMR
(376 MHz, CDCl3)
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
(ppm)

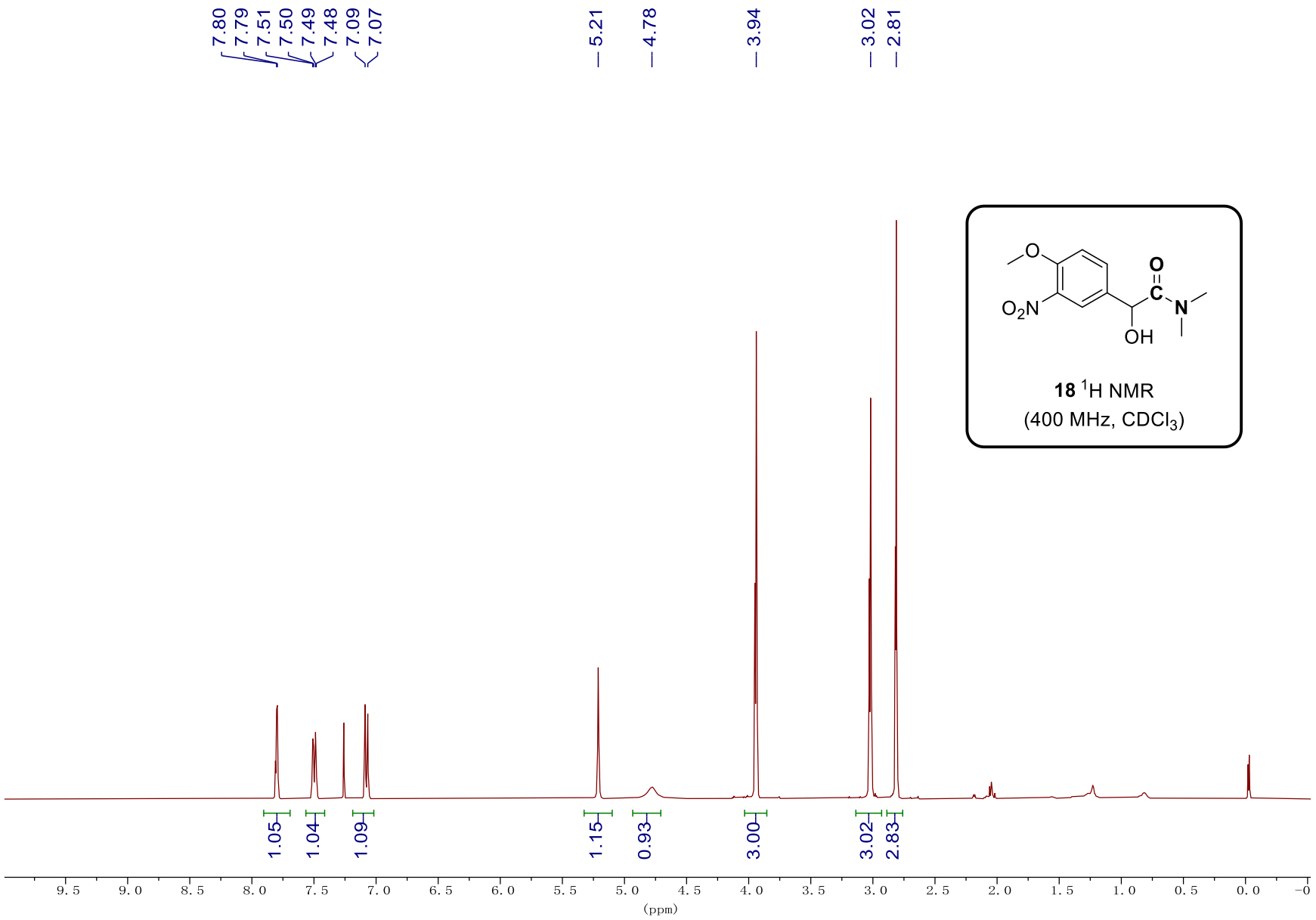

172.06
149.32
149.30
138.02
129.13
124.35
121.79
121.58
119.23
116.67
77.48
77.16
76.84
70.76
36.52
36.46
F3C
O
O
C
N
OH
15 13C NMR
(101 MHz, CDCl3)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
(ppm)

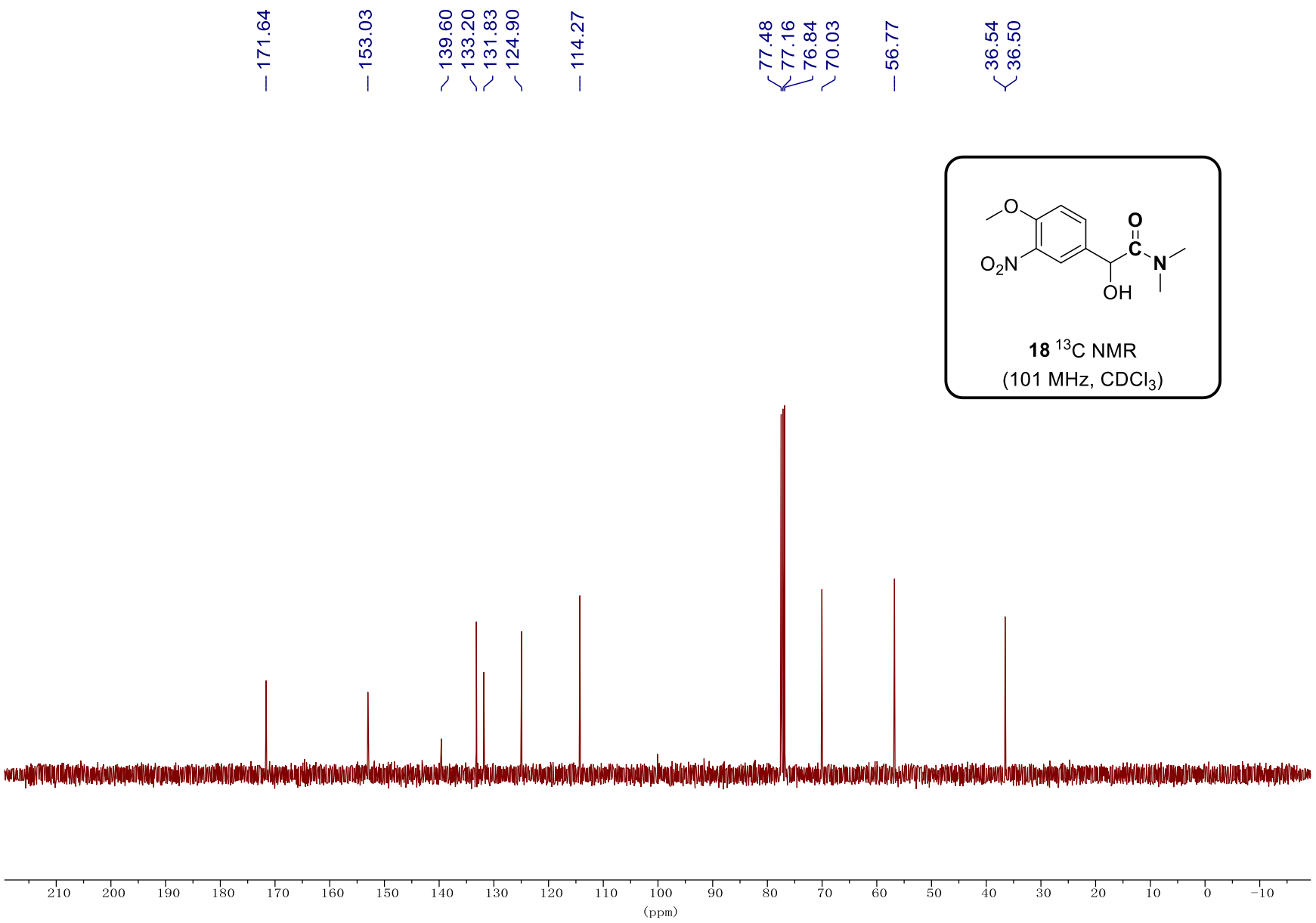

7.33
7.31
7.09
7.07
5.20
5.18
4.74
4.72
3.01
2.78
2.28
O
O
O
C
N
OH
16 1H NMR
(400 MHz, CDCl3)
1.97
1.94
1.00
1.01
3.07
2.94
2.98
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.
(ppm)

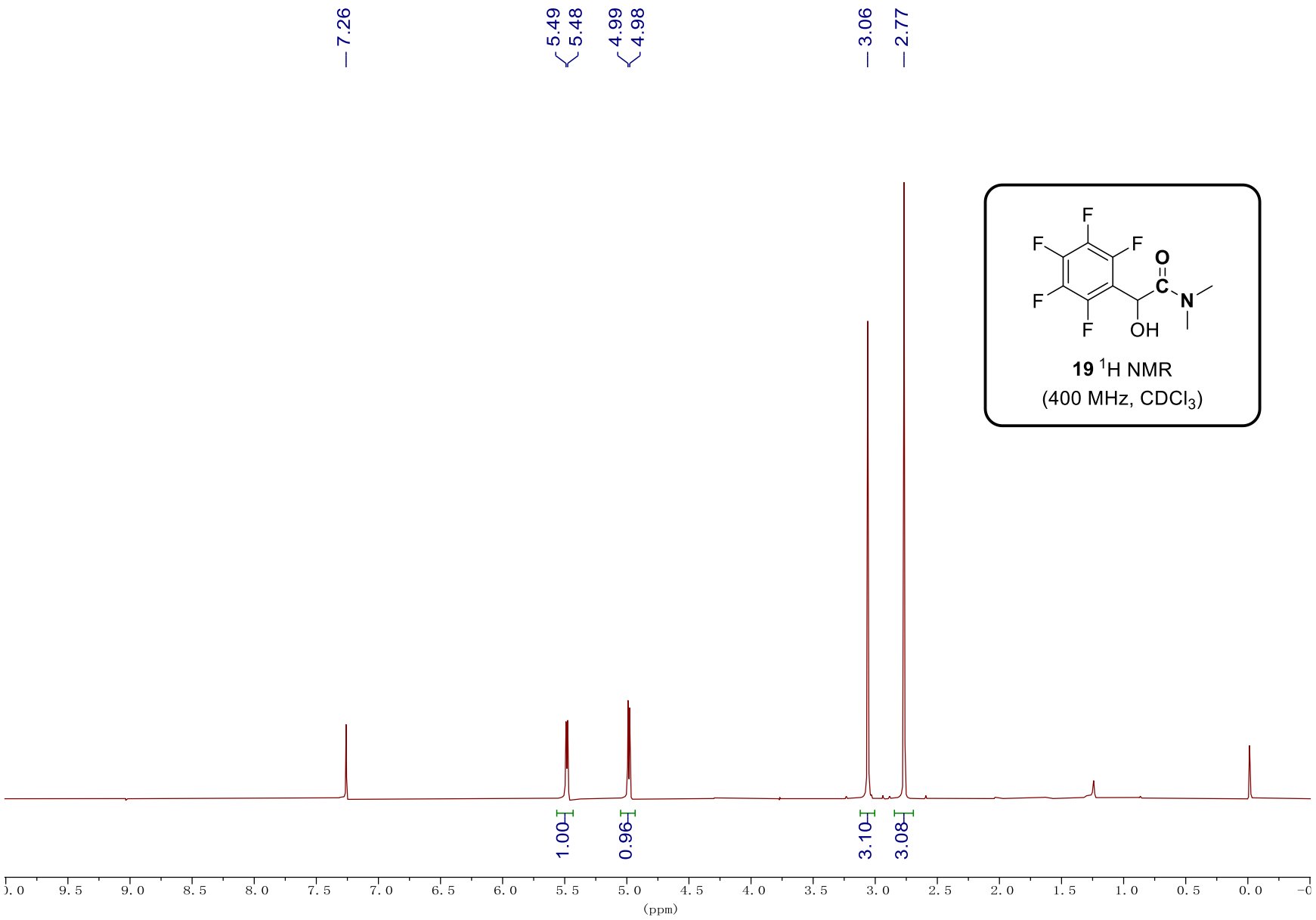

172.26
169.34
150.78
136.82
128.70
122.23
77.48
77.16
76.84
70.93
36.55
36.42
21.23
O
O
O
C
N
OH
16 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

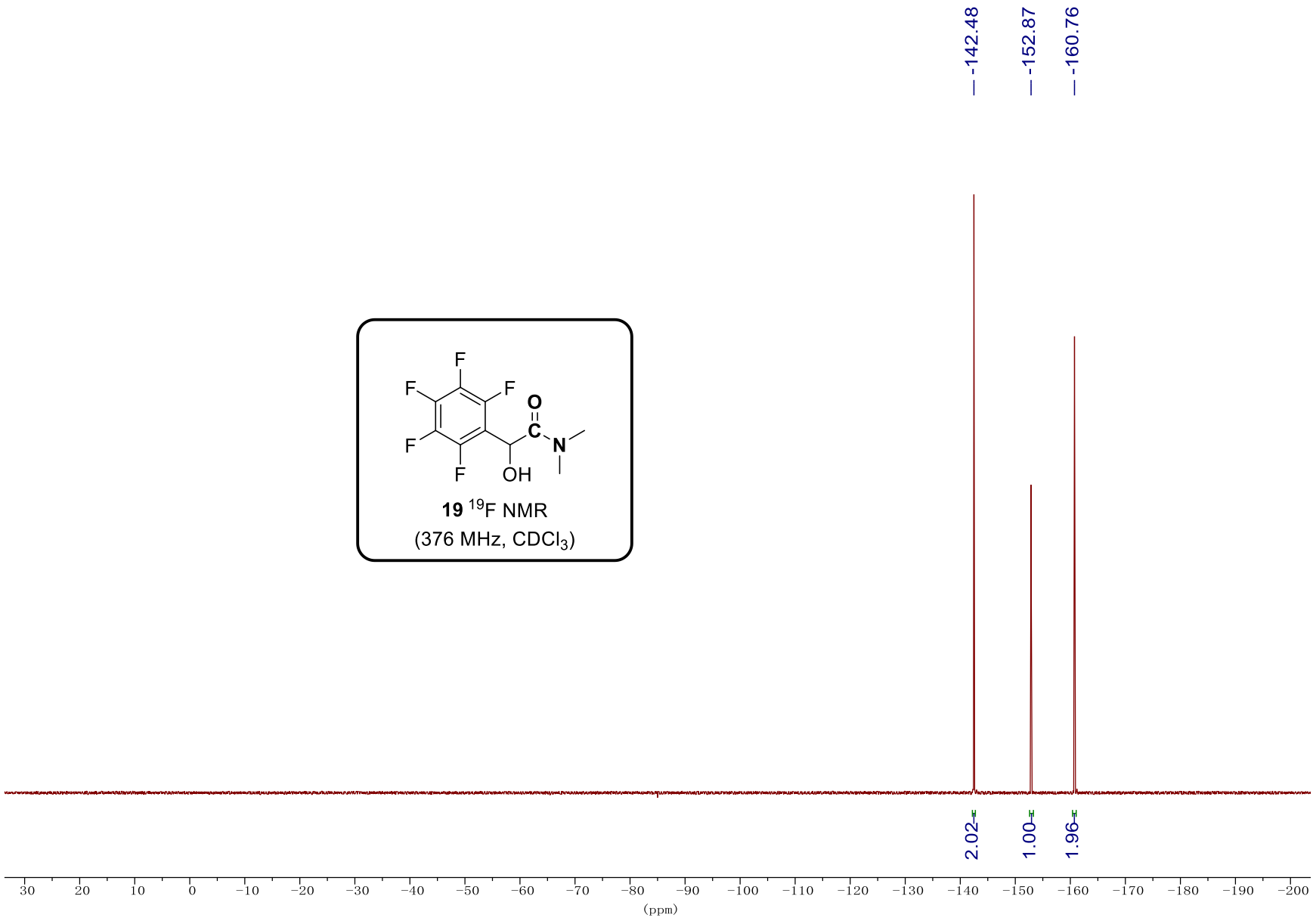

7.42
7.41
7.41
7.40
7.40
7.28
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.24
7.24
7.23
7.23
7.22
7.22
7.21
5.68
4.76
3.04
2.72
O
C
N
Cl
OH
17 1H NMR
(400 MHz, CDCl3)
1.01
3.24
1.00
0.84
3.21
3.21
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)


172.23
136.80
133.79
129.97
128.50
127.94
77.48
77.16
76.84
67.88
36.45
36.05
O
C
N
Cl
OH
17 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)


7.80
7.79
7.51
7.50
7.49
7.48
7.09
7.07
5.21
4.78
3.94
3.02
2.81
1.05
1.04
1.09
1.15
0.93
3.00
3.02
2.83
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)
O
O2N
O
C
N
OH
18 1H NMR
(400 MHz, CDCl3)


171.64
153.03
139.60
133.20
131.83
124.90
114.27
77.48
77.16
76.84
70.03
56.77
36.54
36.50
O
O2N
O
C
N
OH
18 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)


7.26
5.49
5.48
4.99
4.98
3.06
2.77
F
F
F
F
F
O
C
N
OH
19 1H NMR
(400 MHz, CDCl3)
1.00
0.96
3.10
3.08
(ppm)


-142.48
-152.87
-160.76
F
F
F
F
F
O
C
N
OH
19 19F NMR
(376 MHz, CDCl3)
2.02
1.00
1.96
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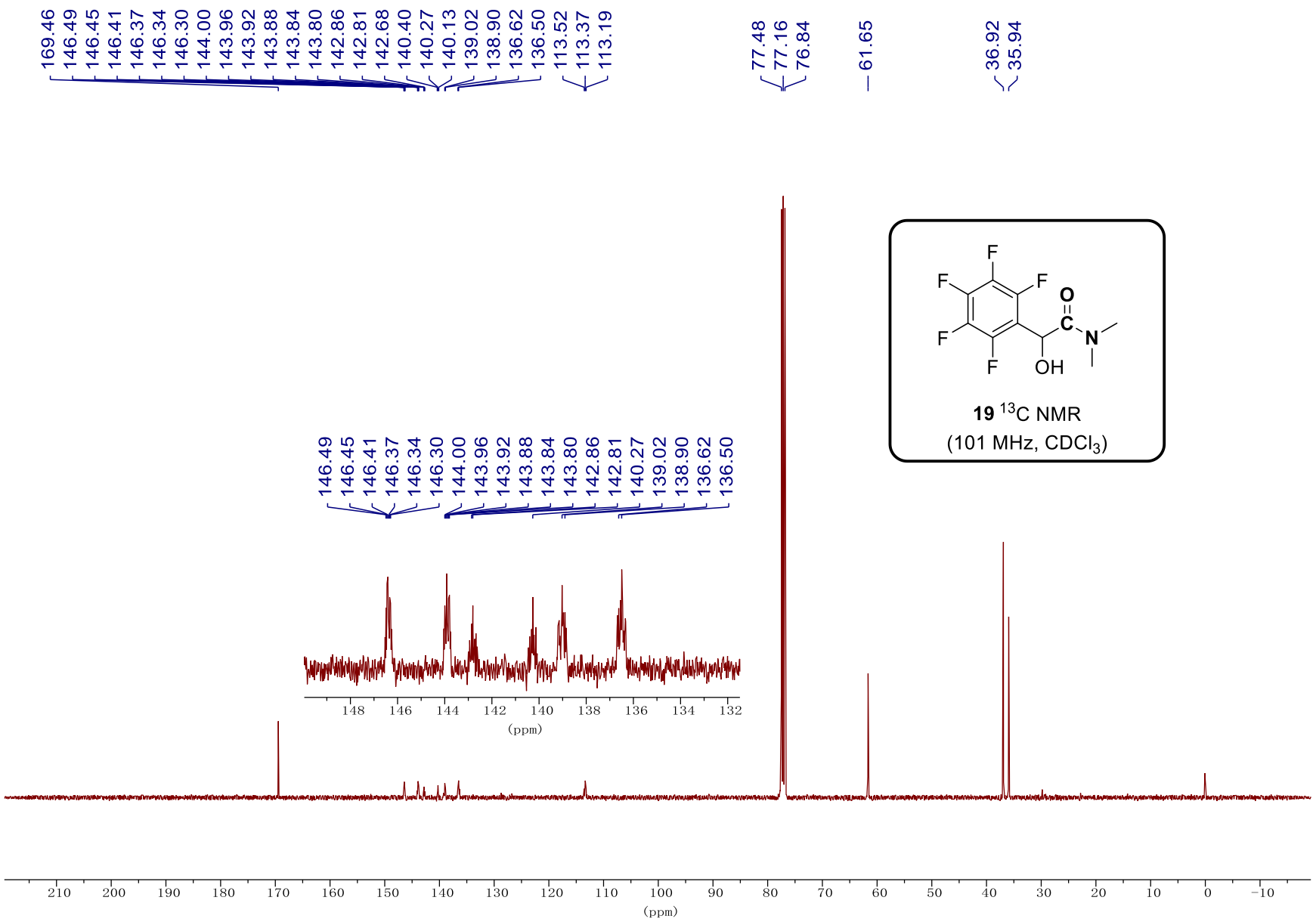
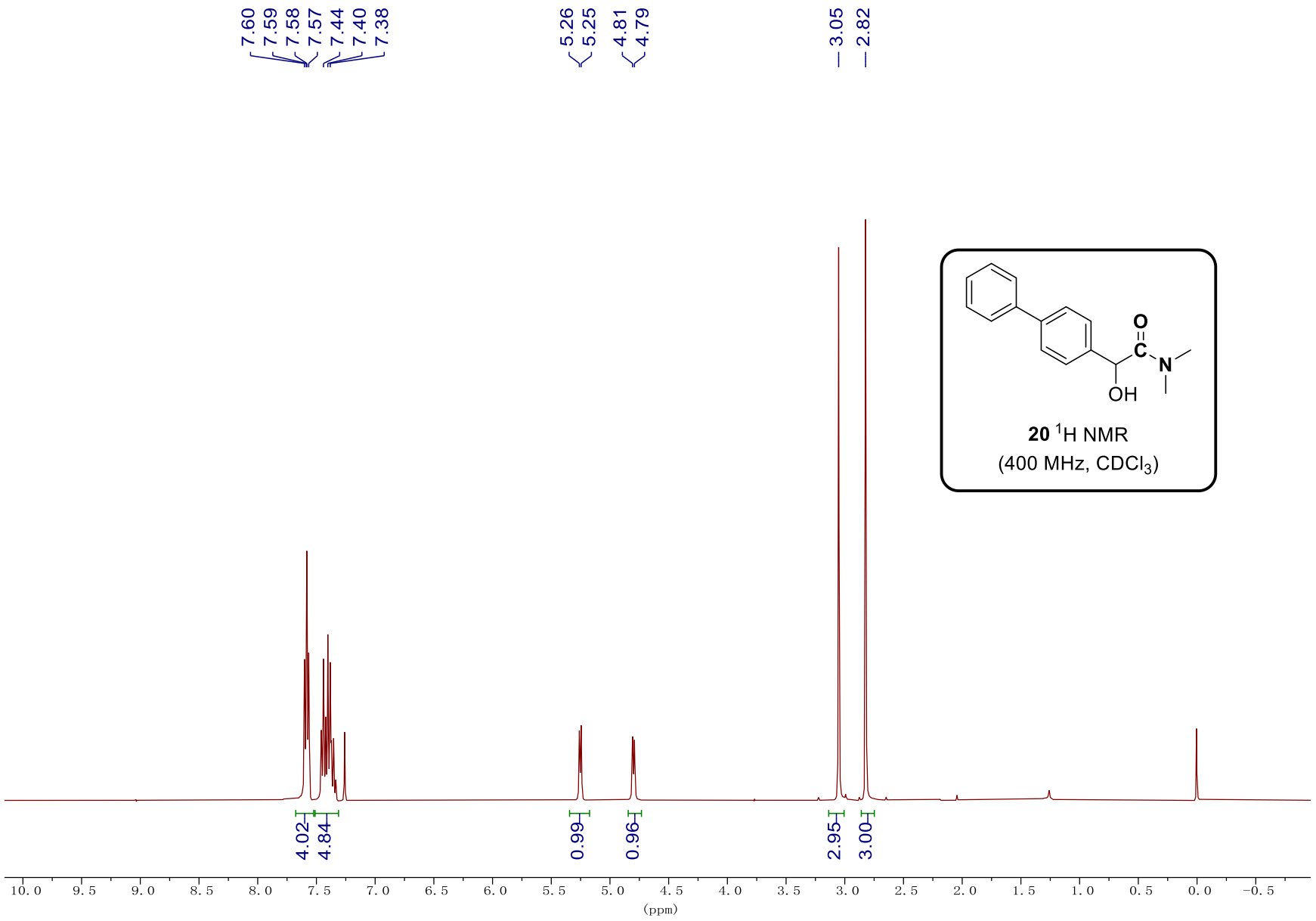
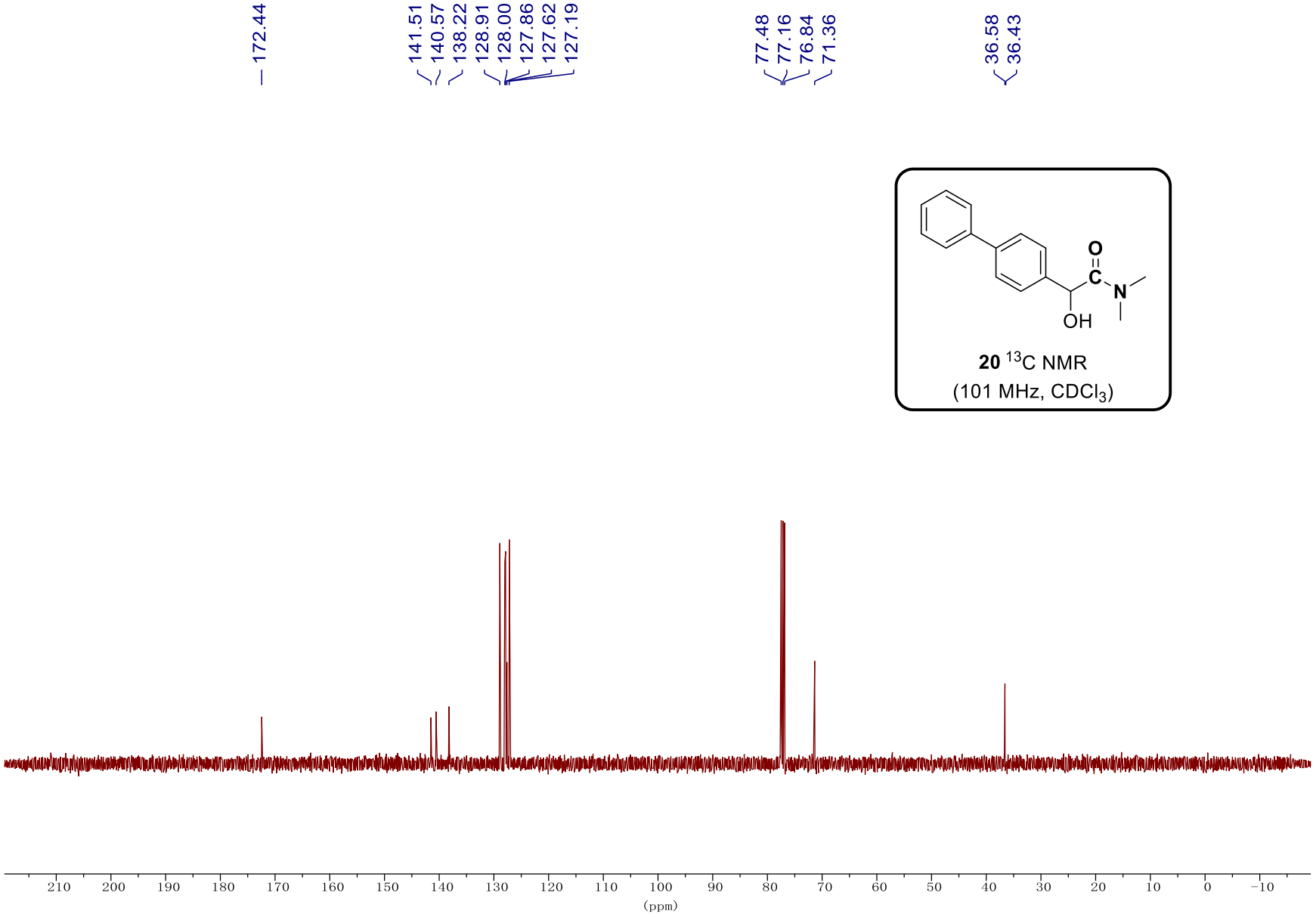
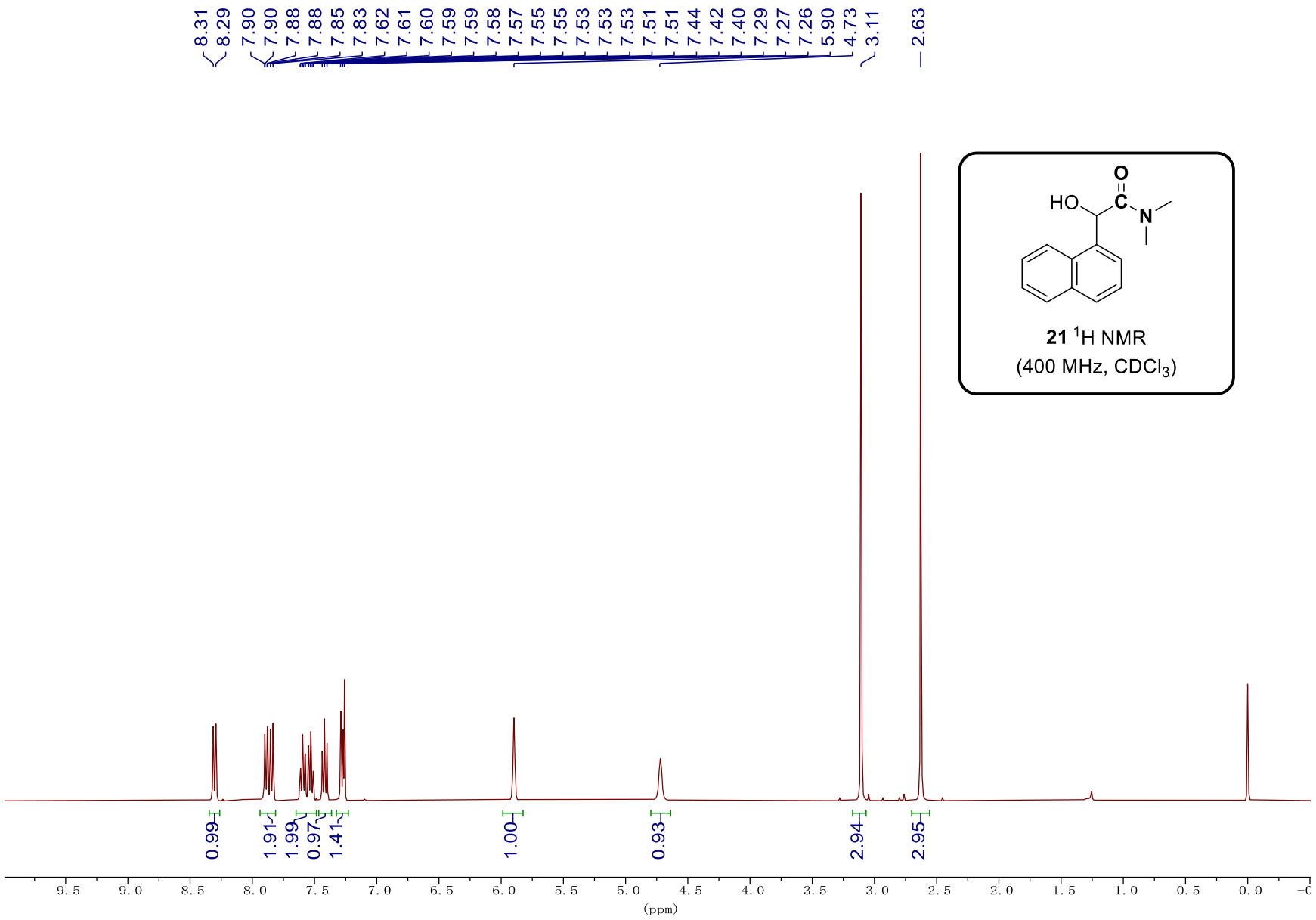
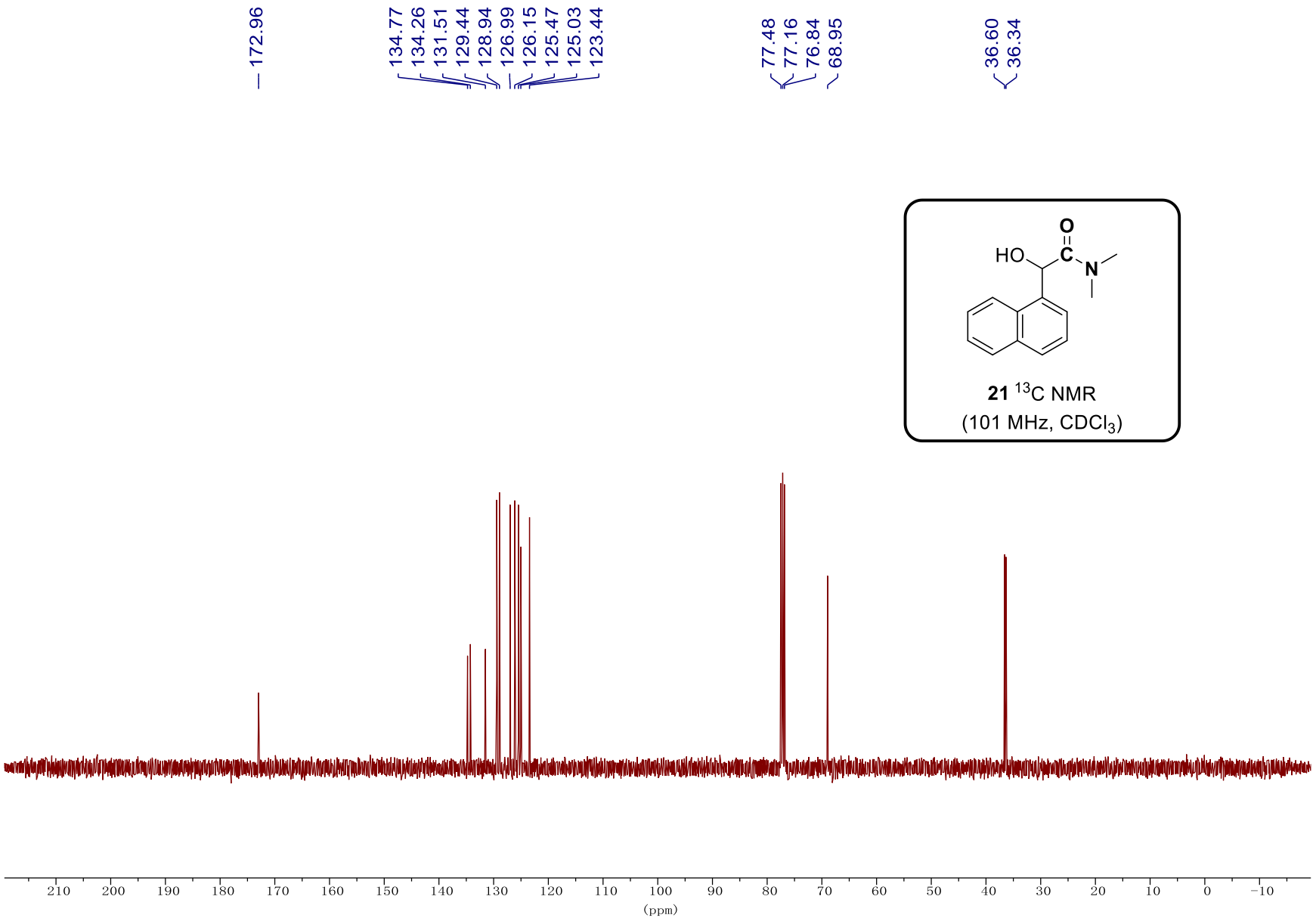
(ppm)

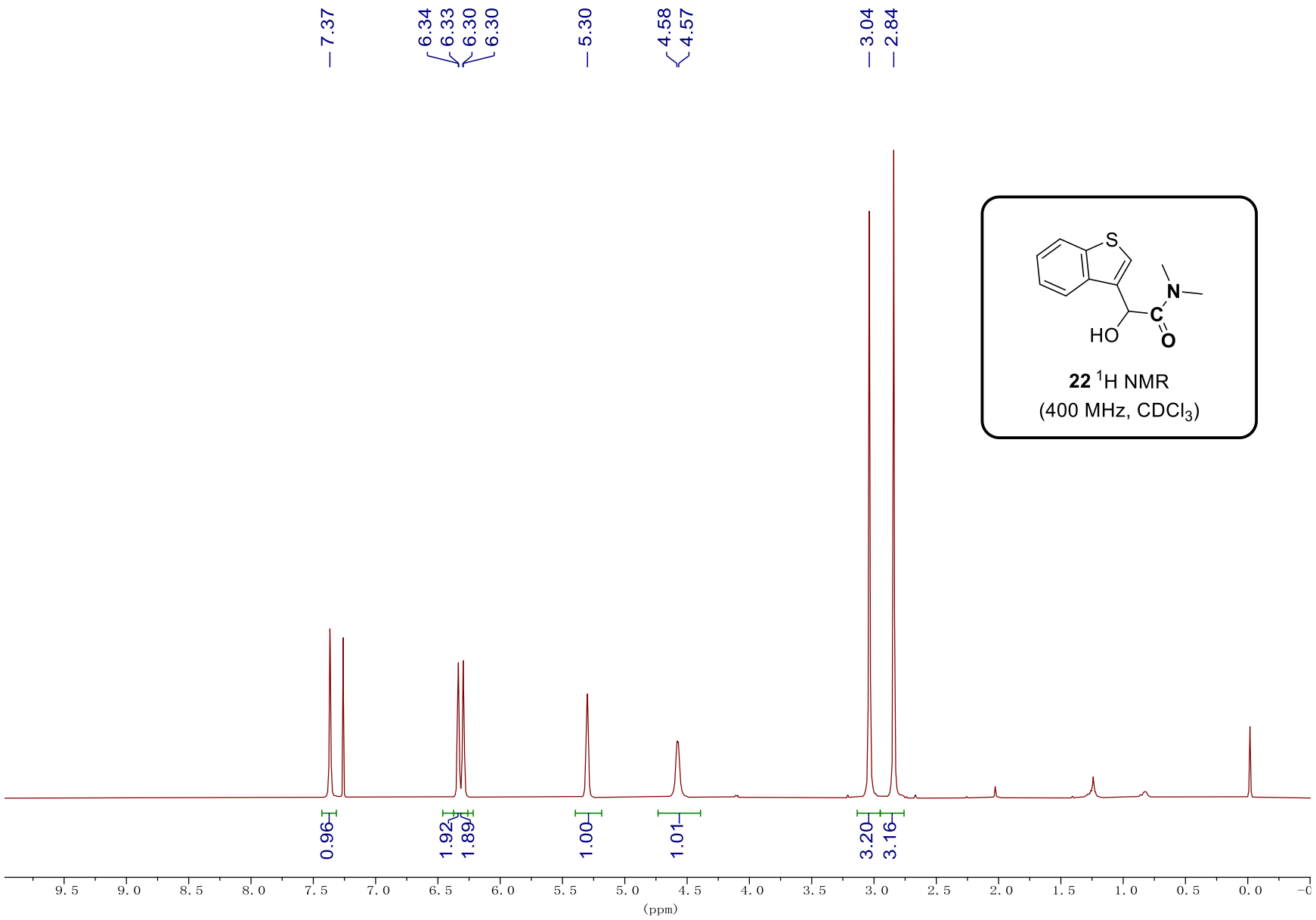

169.46
146.49
146.45
146.41
146.37
146.34
146.30
144.00
143.96
143.92
143.88
143.84
143.80
142.86
142.81
142.68
140.40
140.27
140.13
139.02
138.90
136.62
136.50
113.52
113.37
113.19
77.48
77.16
76.84
61.65
36.92
35.94
F
F
F
F
F
O
C
N
OH
19 13C NMR
(101 MHz, CDCl3)
148
146
144
142
140
138
136
134
132
(ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

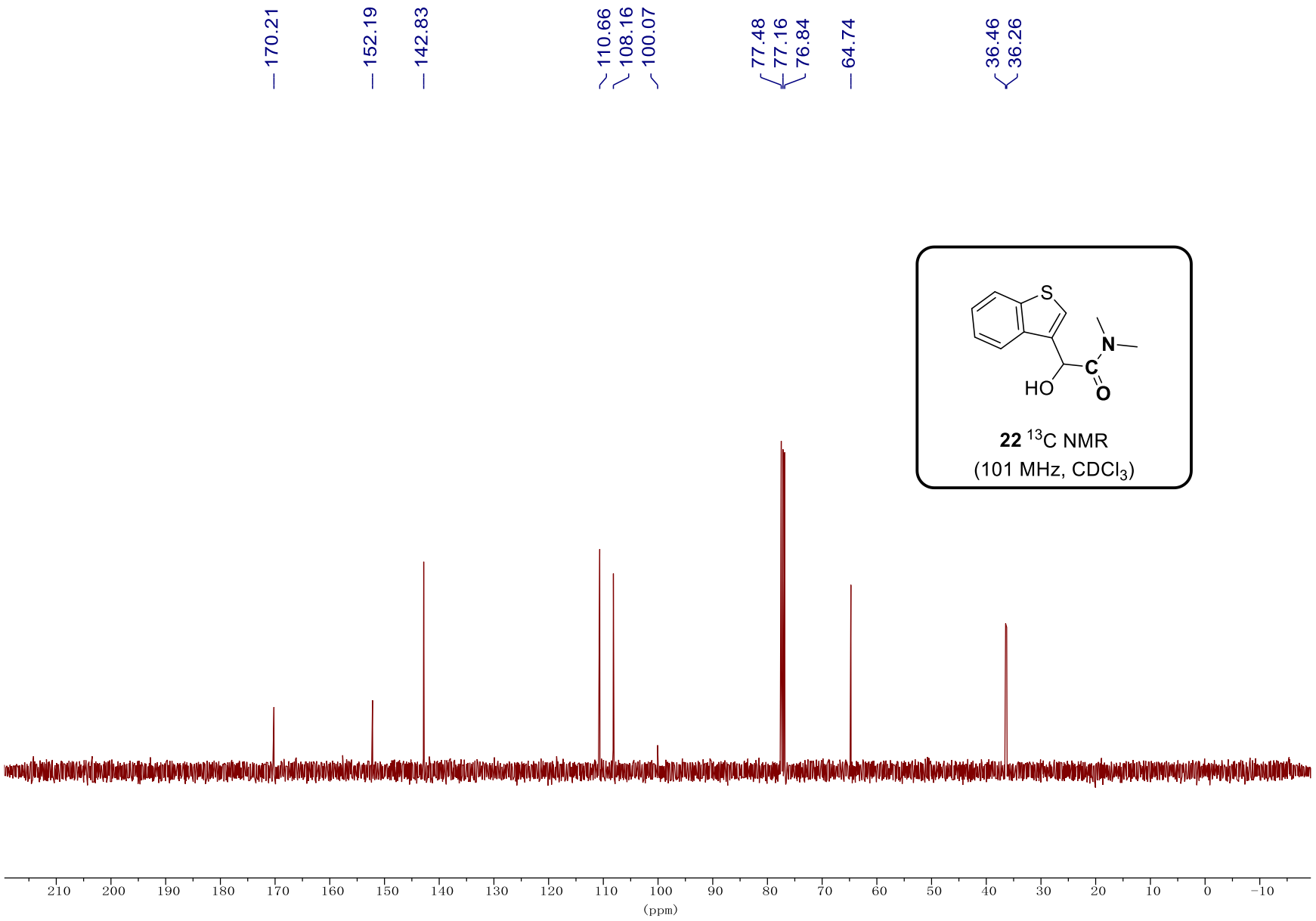

7.60
7.59
7.58
7.57
7.44
7.40
7.38
5.26
5.25
4.81
4.79
3.05
2.82
O
C
N
OH
20 1H NMR
(400 MHz, CDCl3)
4.02
4.84
0.99
0.96
2.95
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
(ppm)

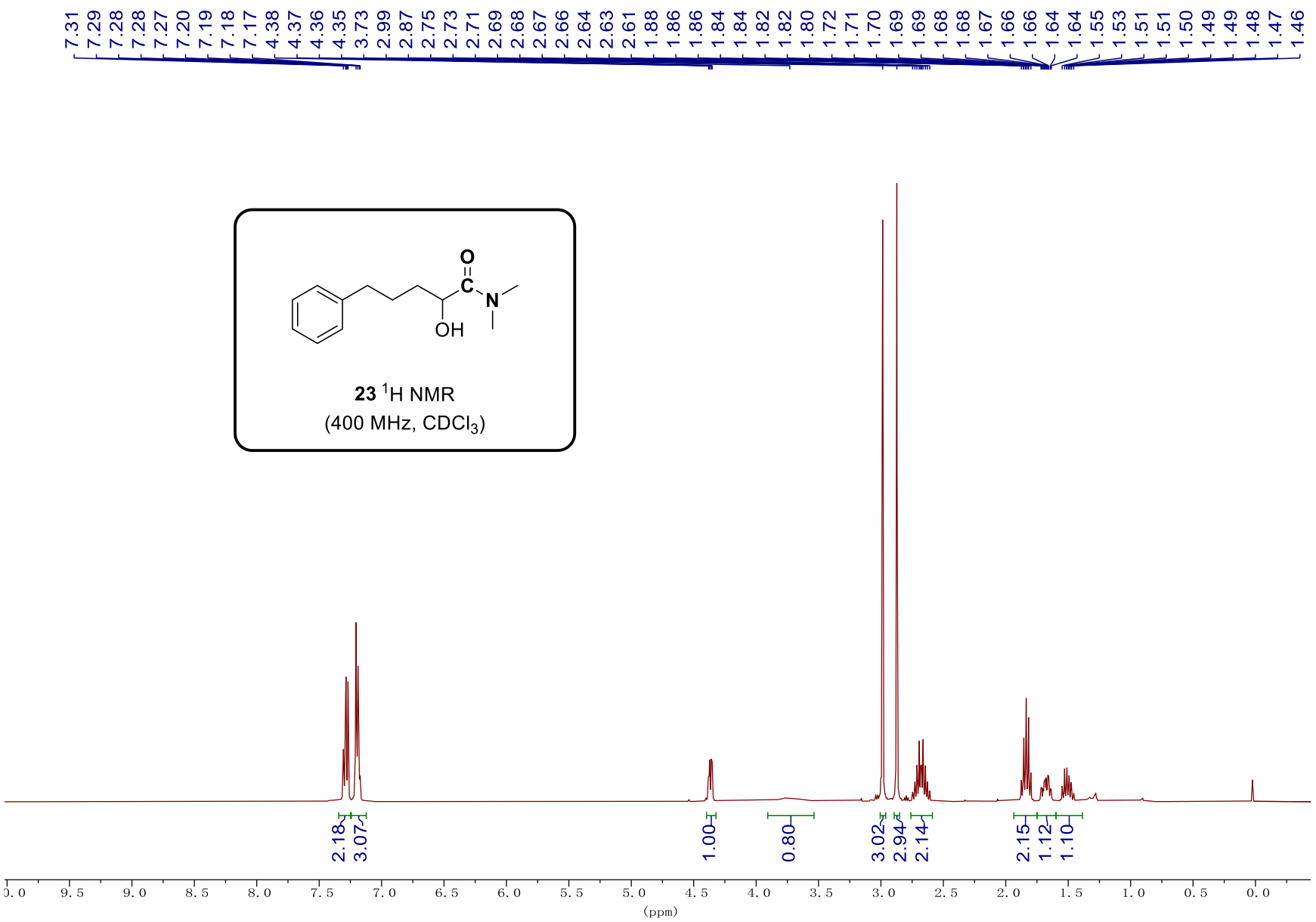

172.44
141.51
140.57
138.22
128.91
128.00
127.86
127.62
127.19
77.48
77.16
76.84
71.36
36.58
36.43
O
C
N
OH
20 13C NMR
(101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

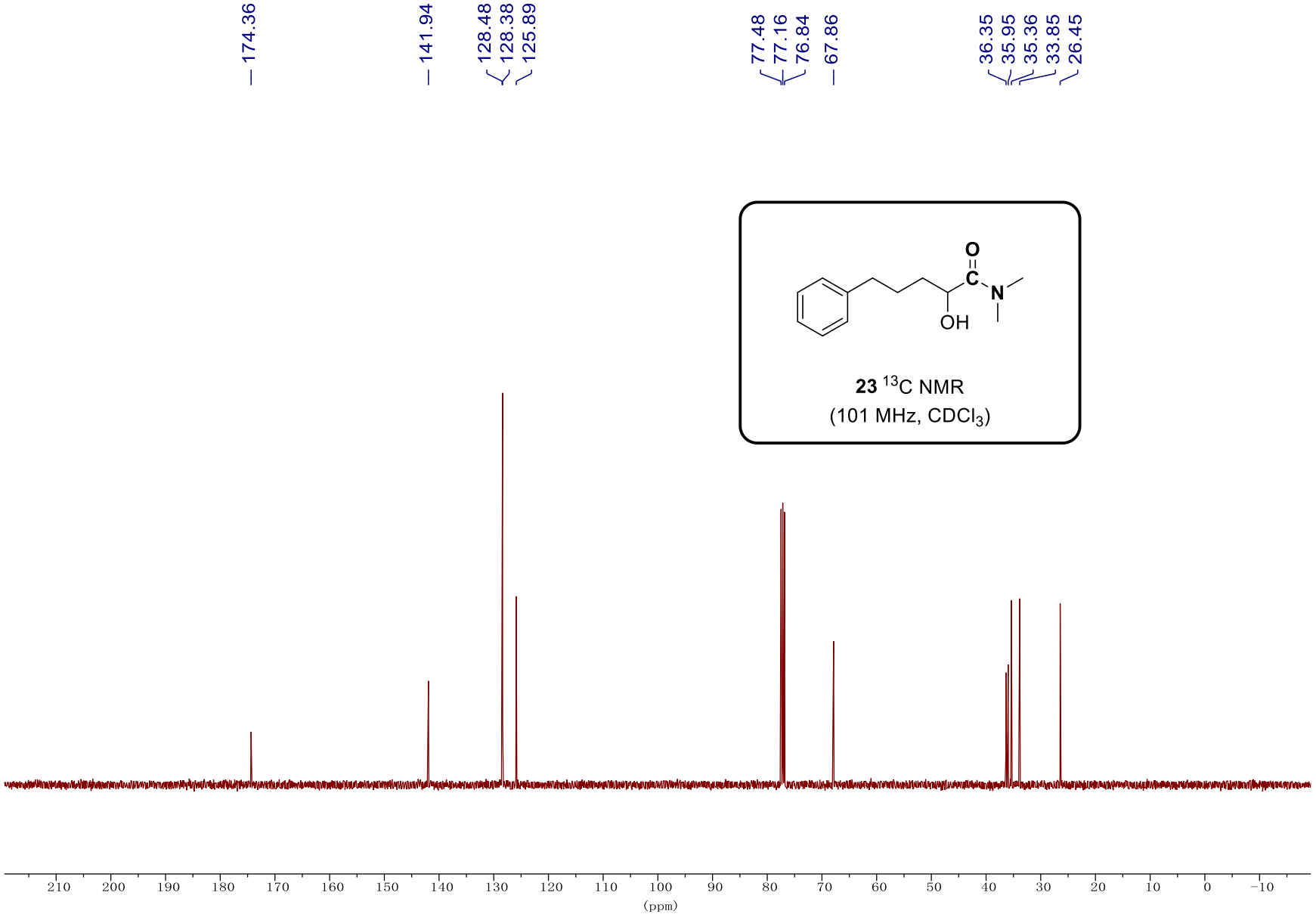

8.31
8.29
7.90
7.90
7.88
7.88
7.85
7.83
7.62
7.61
7.60
7.59
7.59
7.58
7.57
7.55
7.55
7.53
7.53
7.53
7.51
7.51
7.44
7.42
7.40
7.29
7.27
7.26
5.90
4.73
3.11
2.63
HO
O
C
N
21 1H NMR
(400 MHz, CDCl3)
0.99
1.91
1.99
0.97
1.41
1.00
0.93
2.94
2.95
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

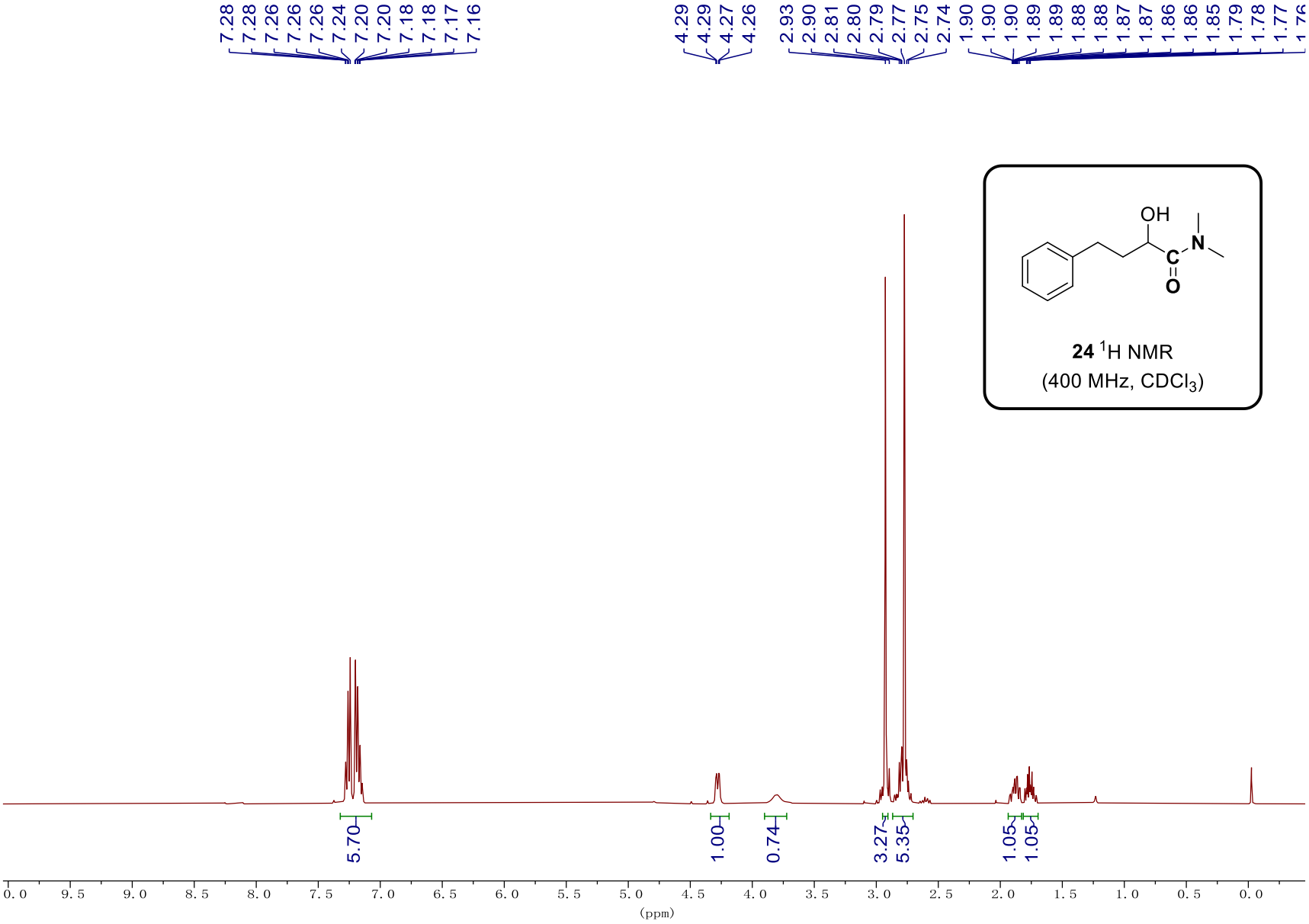

172.96
134.77
134.26
131.51
129.44
128.94
126.99
126.15
125.47
125.03
123.44
77.48
77.16
76.84
68.95
36.60
36.34
HO
O
C
N
21 13C NMR
(101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

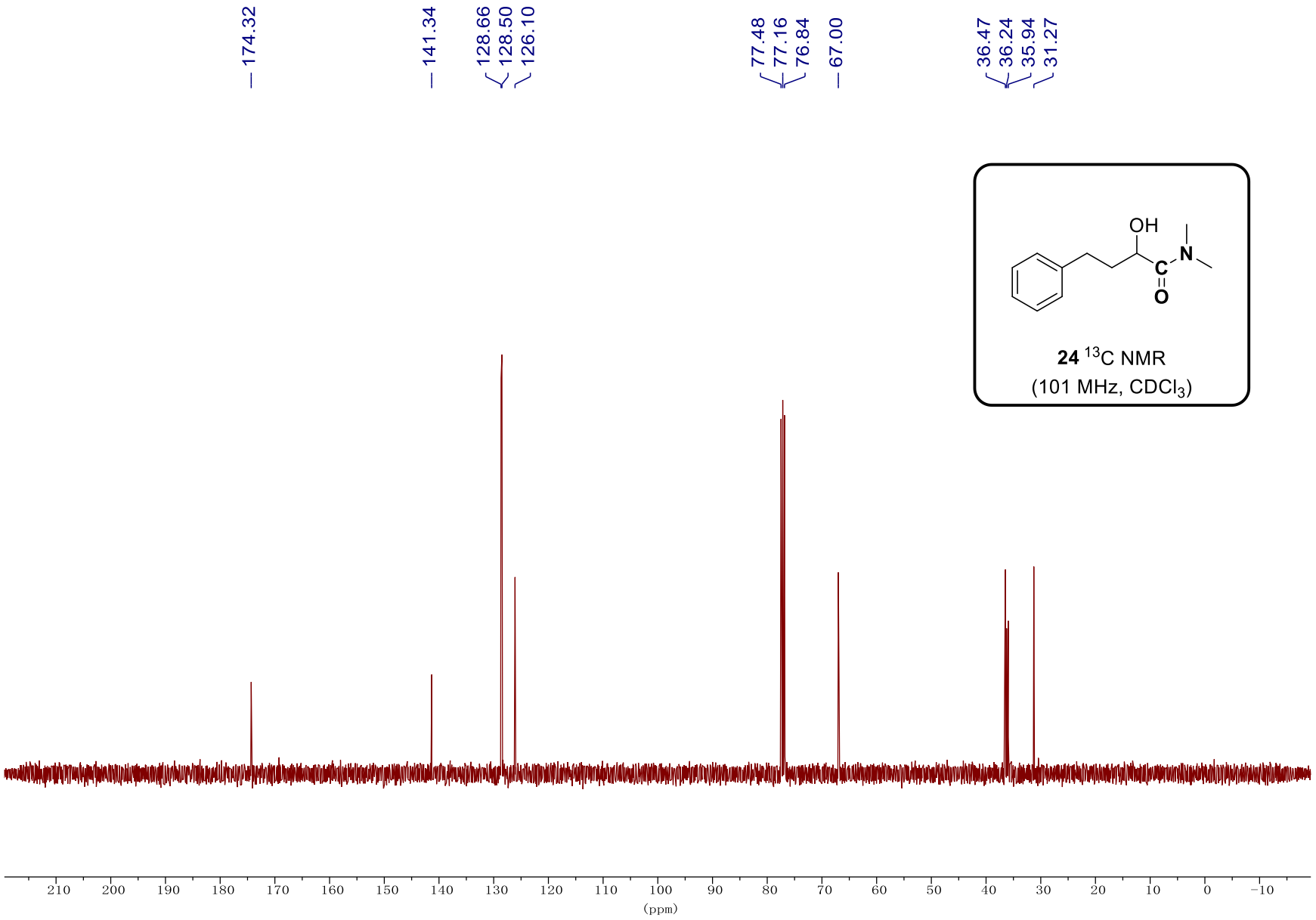

7.37
6.34
6.33
6.30
6.30
5.30
4.58
4.57
3.04
2.84
0.96
1.92
1.89
1.00
1.01
3.20
3.16
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)
S
N
C
O
HO
22 1H NMR
(400 MHz, CDCl3)

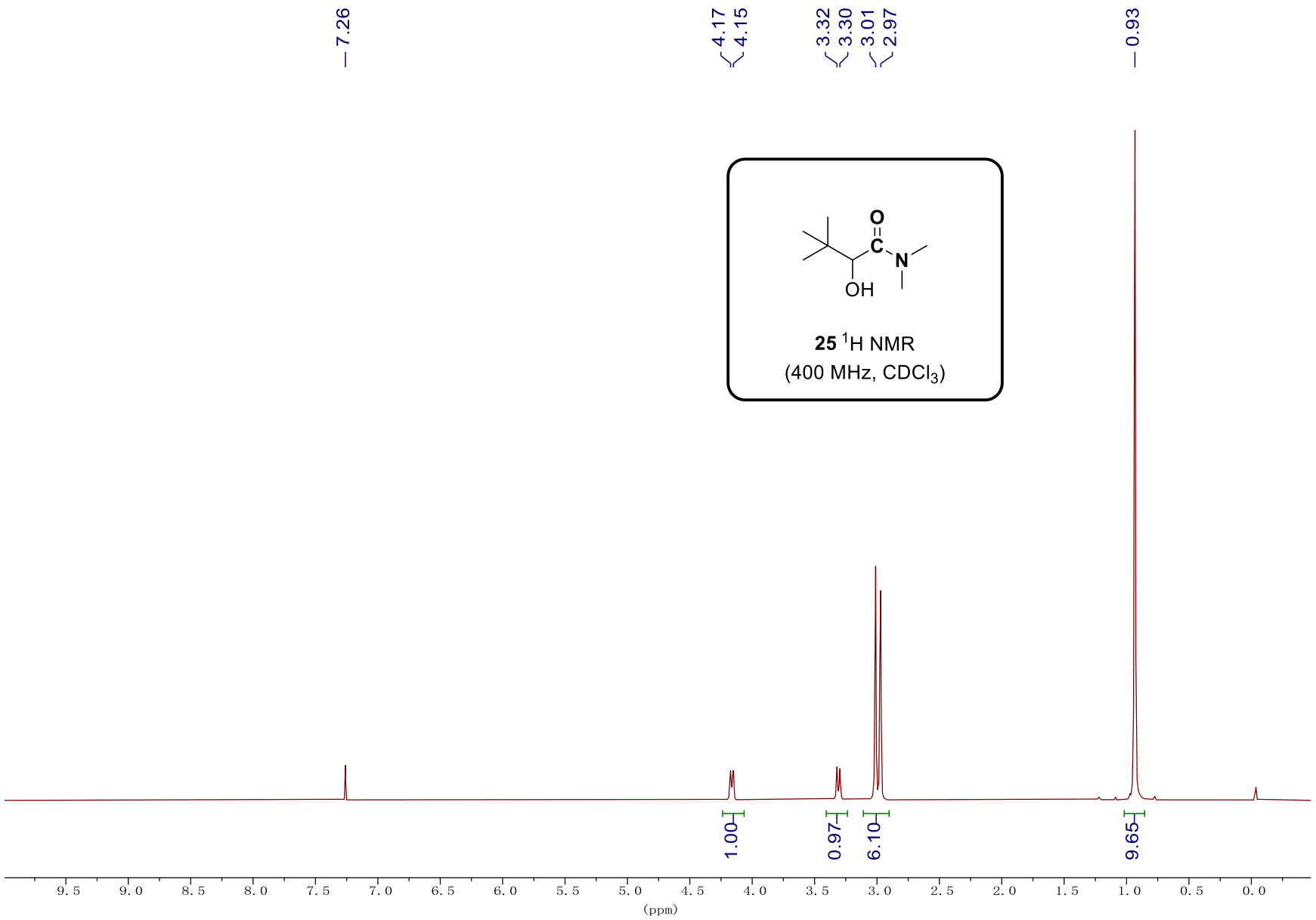

170.21
152.19
142.83
110.66
108.16
100.07
77.48
77.16
76.84
64.74
36.46
36.26
S
N
C
O
HO
22 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

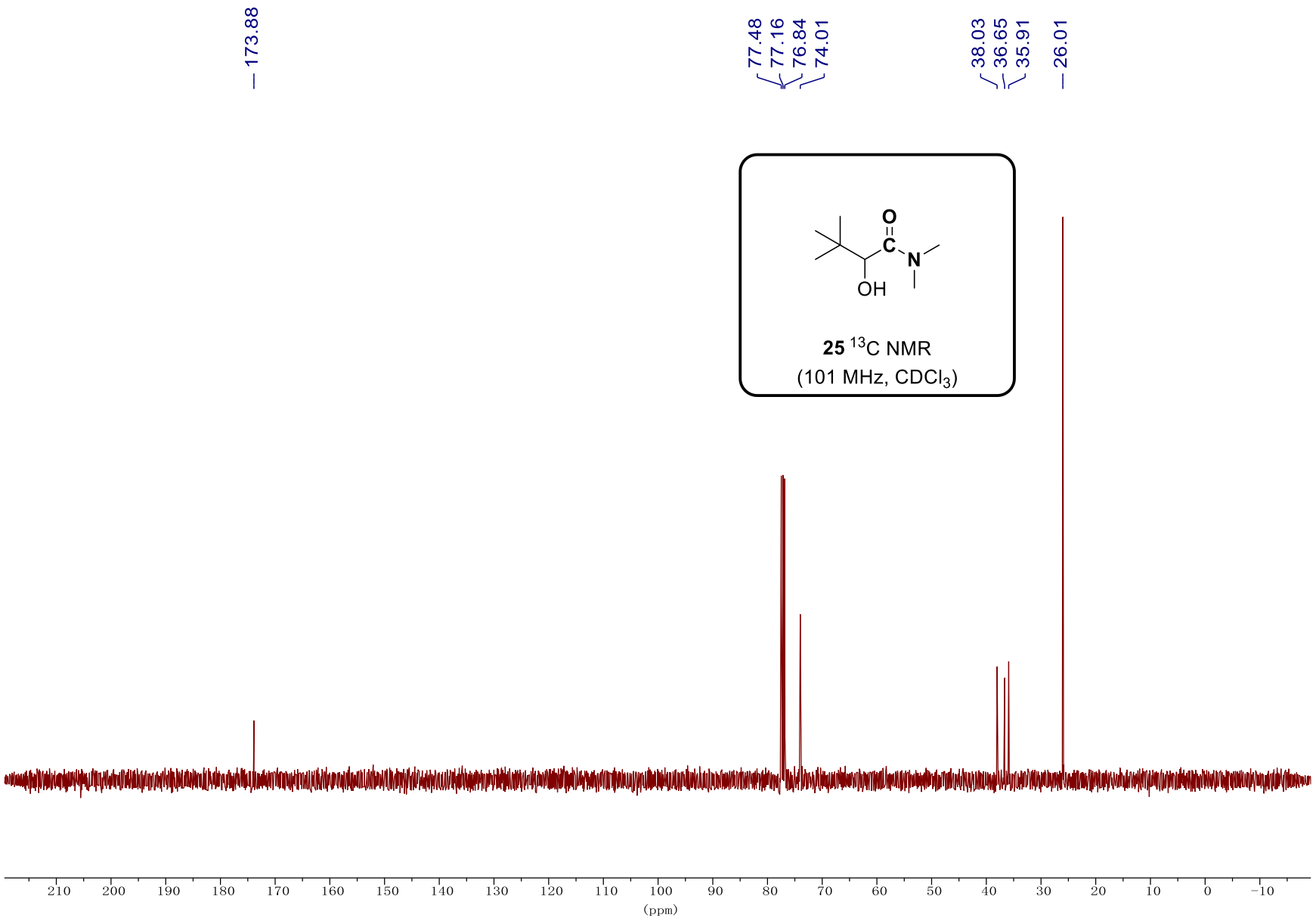

7.31 7.29 7.28 7.28 7.27 7.20 7.19 7.18 7.17 4.38 4.37 4.36 4.35 3.73 2.99 2.87 2.75 2.73 2.71 2.69 2.68 2.67 2.66 2.64 2.63 2.61 1.88 1.86 1.86 1.84 1.84 1.82 1.82 1.80 1.72 1.71 1.70 1.69 1.69 1.68 1.68 1.67 1.66 1.66 1.64 1.64 1.55 1.53 1.51 1.51 1.50 1.49 1.49 1.48 1.47 1.46
O
C
N
OH
23 1H NMR
(400 MHz, CDCl3)
2.18
3.07
1.00
0.80
3.02
2.94
2.14
2.15
1.12
1.10
(ppm)

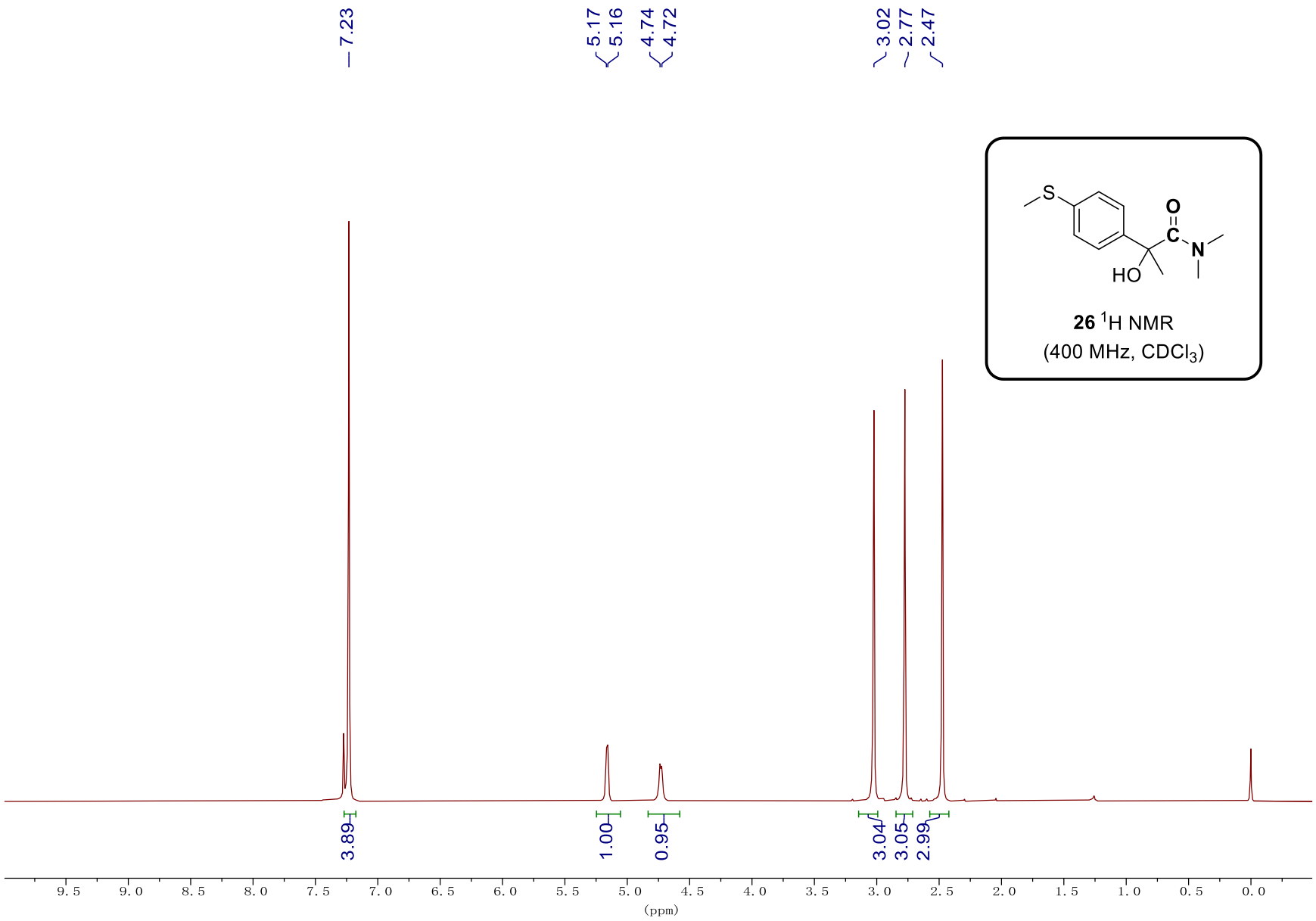

174.36
141.94
128.48
128.38
125.89
77.48
77.16
76.84
67.86
36.35
35.95
35.36
33.85
26.45
O
C
N
OH
23 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

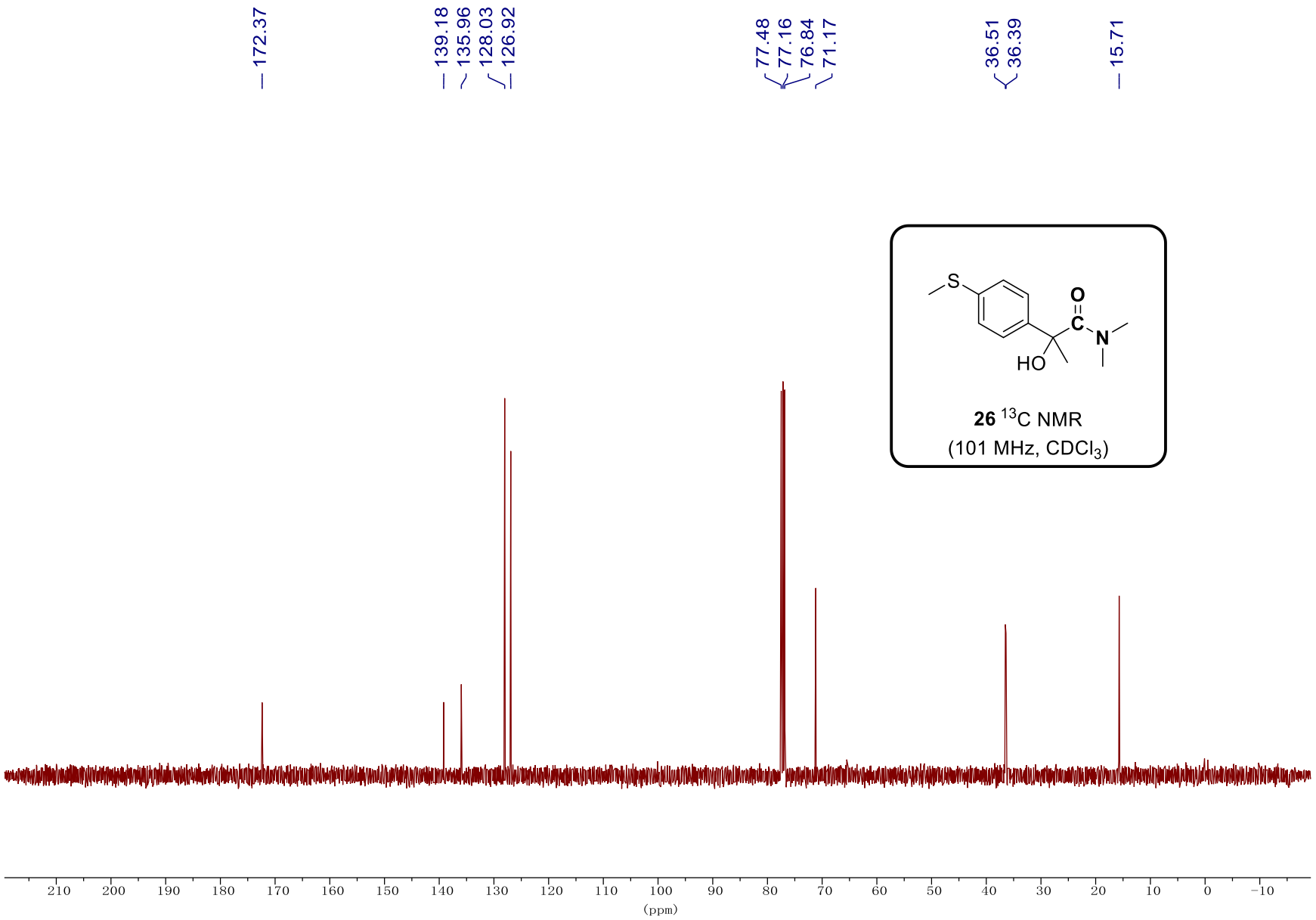

7.28
7.28
7.26
7.26
7.26
7.24
7.20
7.20
7.18
7.18
7.17
7.16
4.29
4.29
4.27
4.26
2.93
2.90
2.81
2.80
2.79
2.77
2.75
2.74
1.90
1.90
1.90
1.89
1.89
1.88
1.88
1.87
1.87
1.86
1.86
1.85
1.79
1.78
1.77
OH
N
C
O
24 1H NMR
(400 MHz, CDCl3)
5.70
1.00
0.74
3.27
5.35
1.05
1.05
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

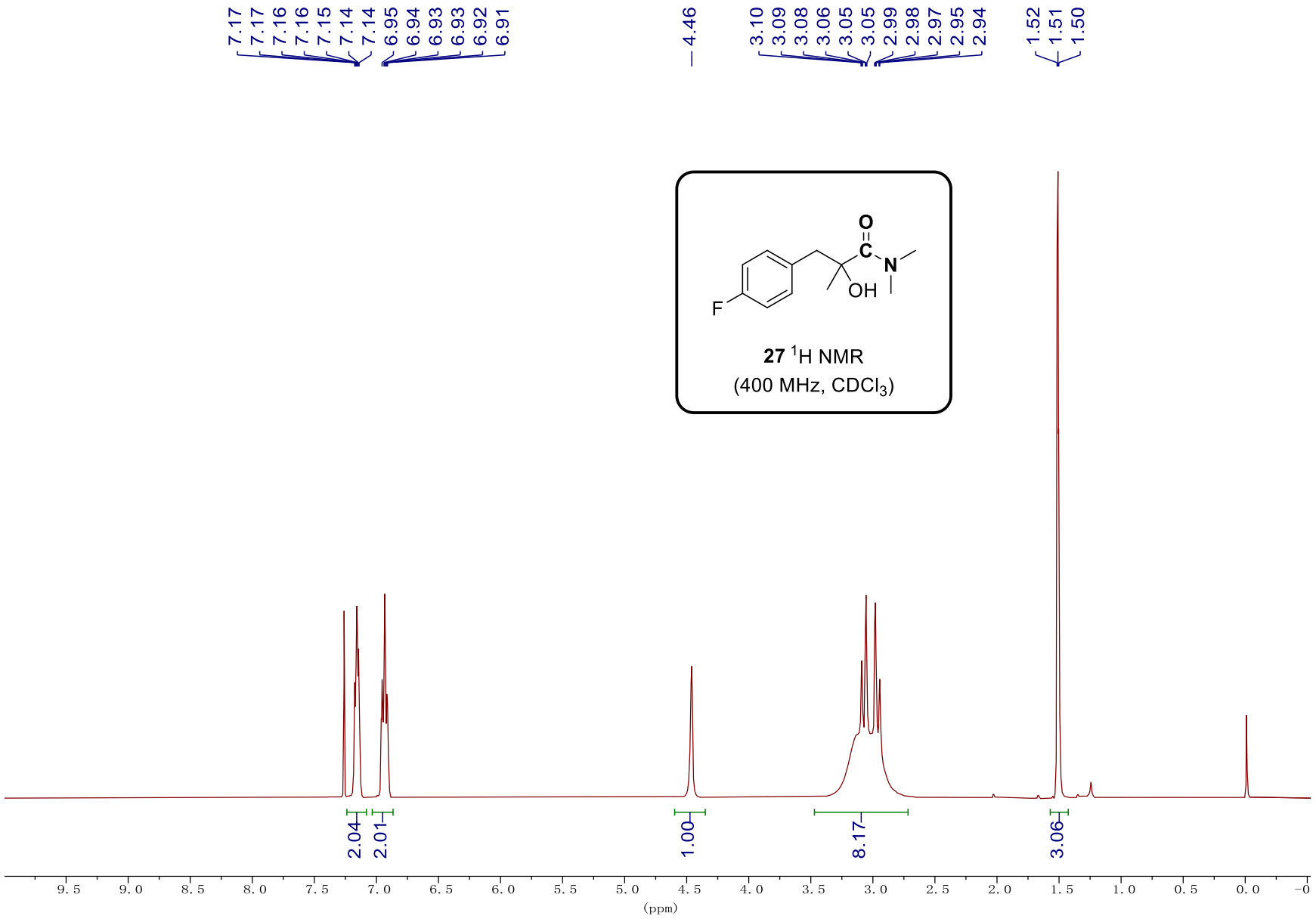

174.32
141.34
128.66
128.50
126.10
77.48
77.16
76.84
67.00
36.47
36.24
35.94
31.27
OH
N
C
O
24 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

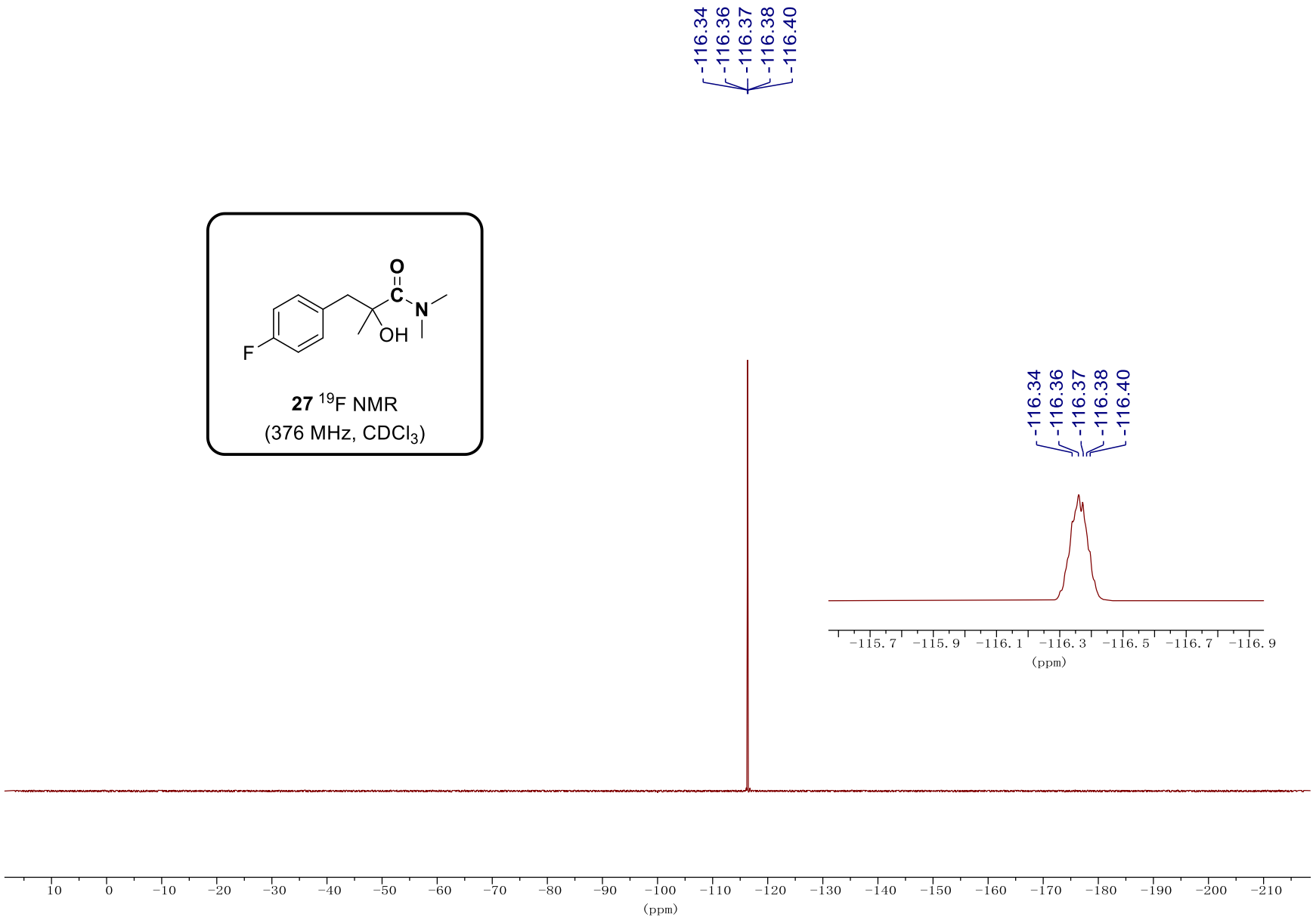

7.26
4.17
4.15
3.32
3.30
3.01
2.97
0.93
O
C
N
OH
25 1H NMR
(400 MHz, CDCl3)
1.00
0.97
6.10
9.65
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

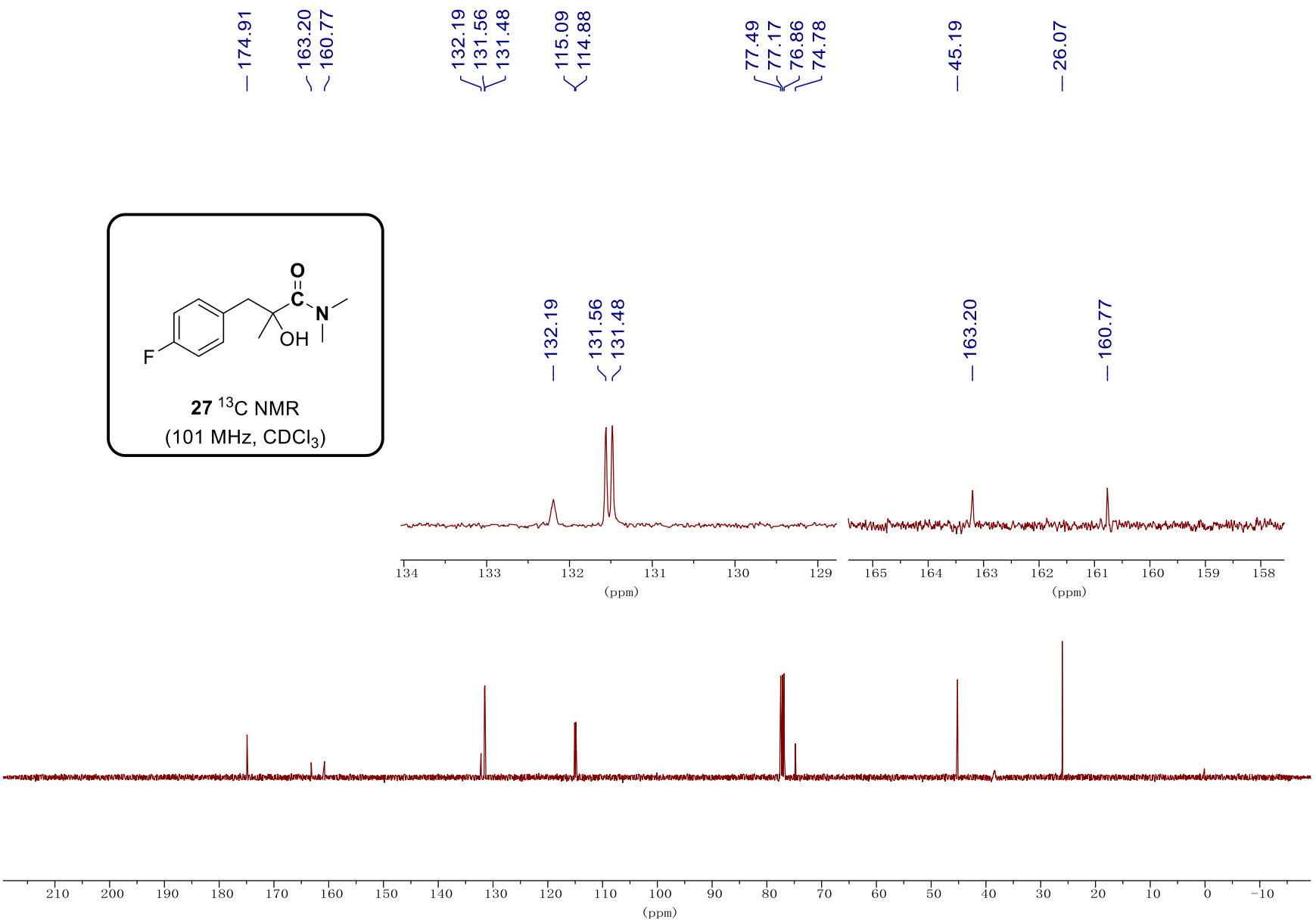

173.88
77.48
77.16
76.84
74.01
38.03
36.65
35.91
26.01
O
C
N
OH
25 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

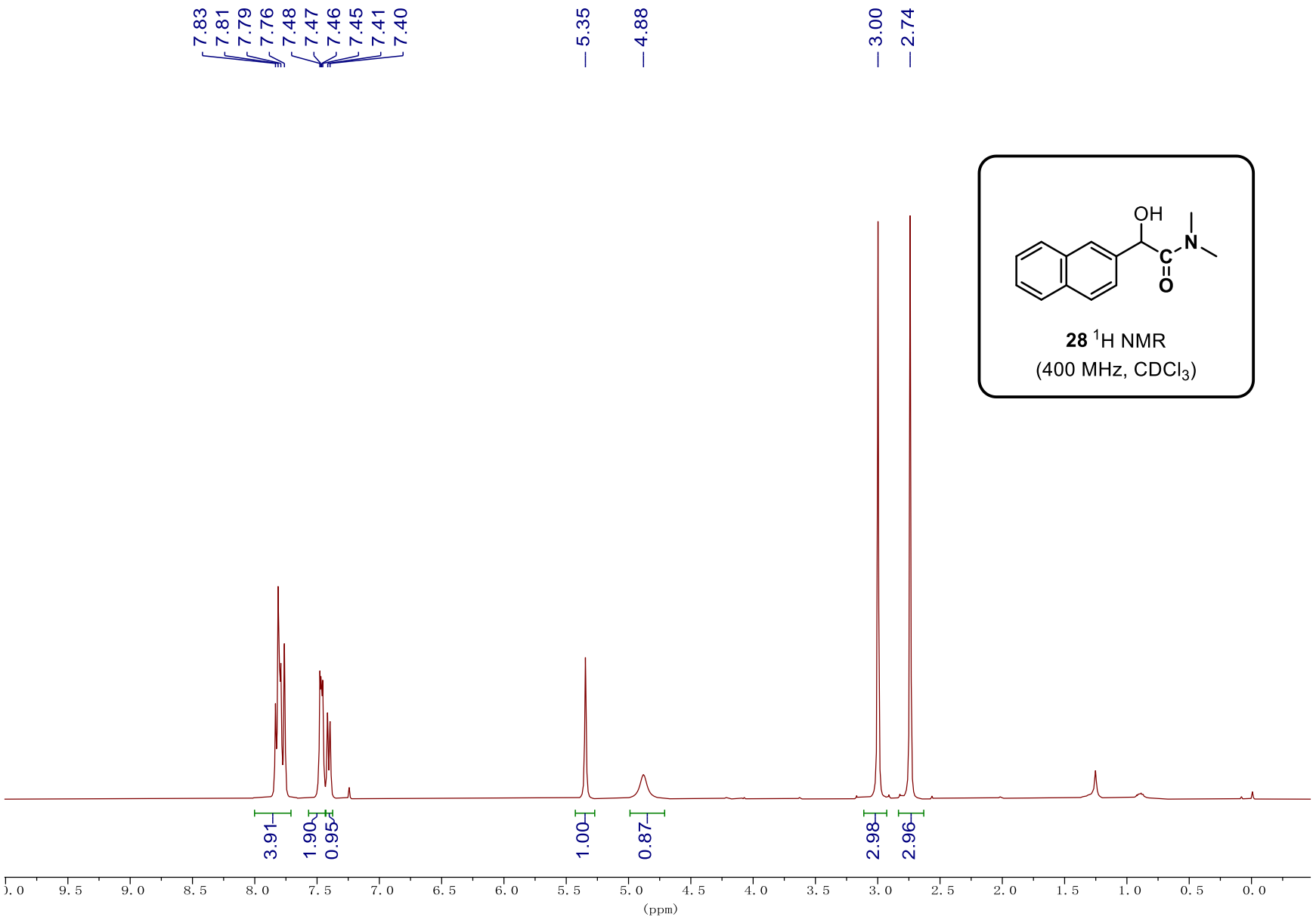

7.23
5.17
5.16
4.74
4.72
3.02
2.77
2.47
S
O
C
N
HO
26 1H NMR
(400 MHz, CDCl3)
3.89
1.00
0.95
3.04
3.05
2.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

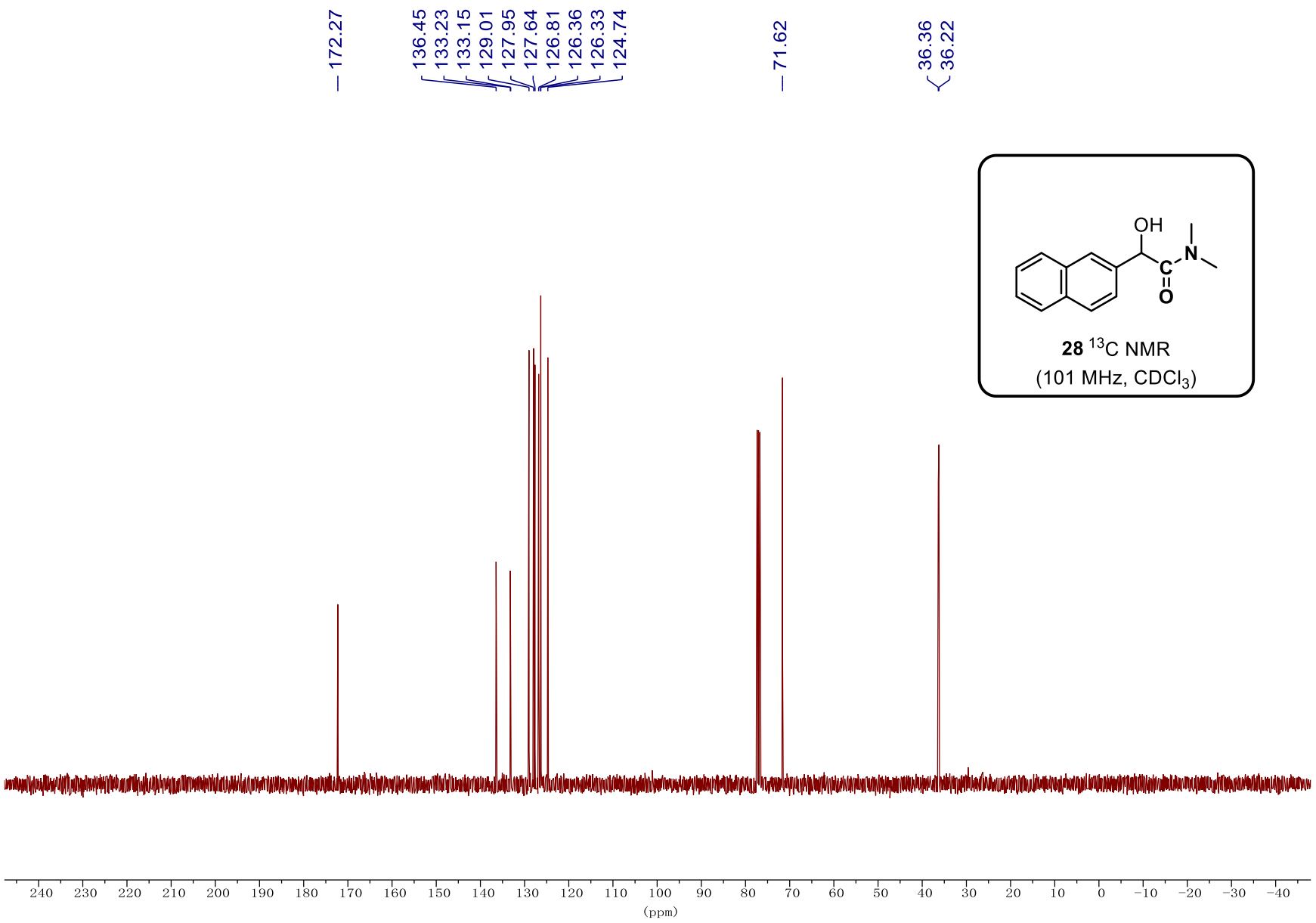

172.37
139.18
135.96
128.03
126.92
77.48
77.16
76.84
71.17
36.51
36.39
15.71
S
O
C
N
HO
26 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

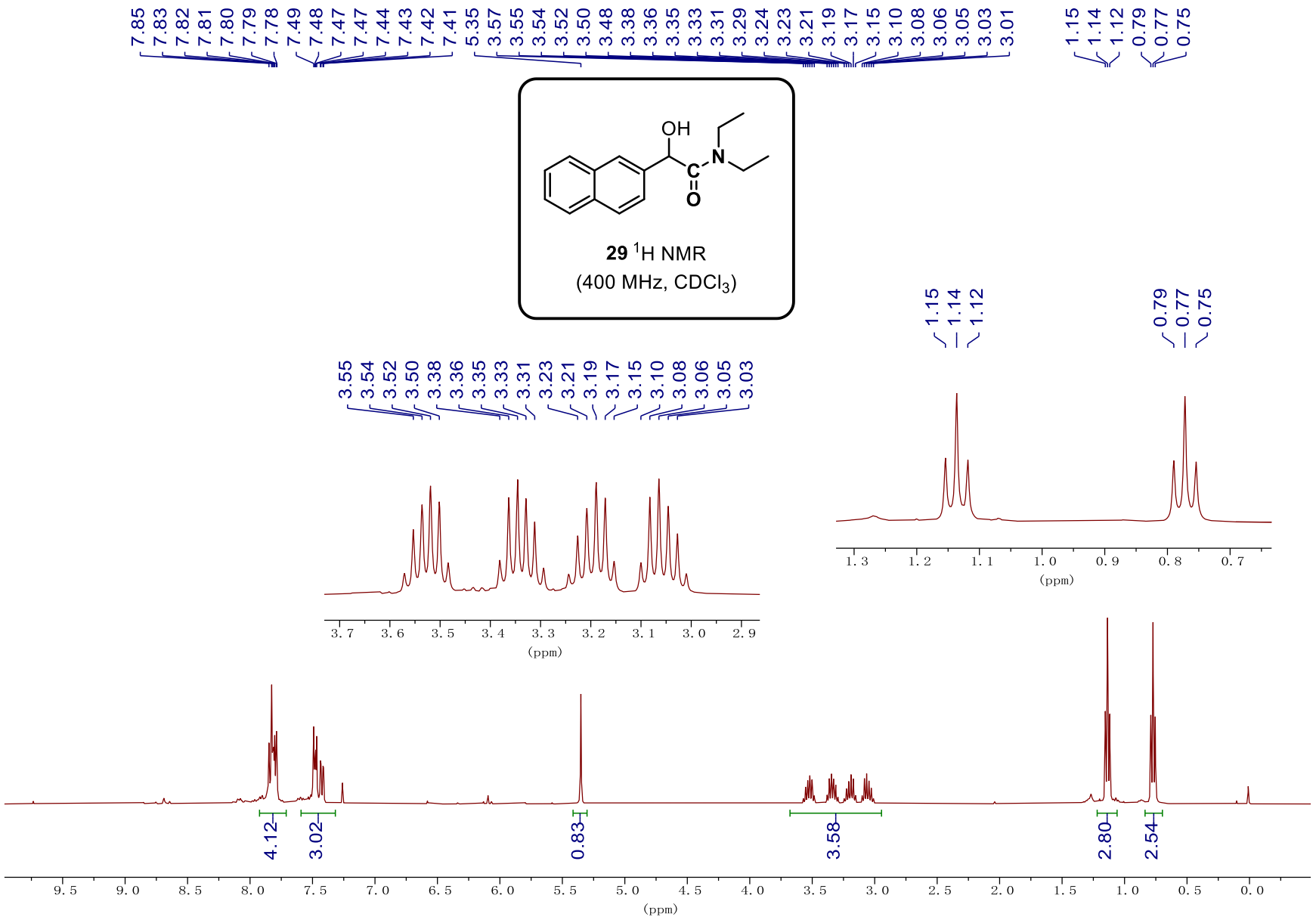

7.17
7.17
7.16
7.16
7.15
7.14
7.14
6.95
6.94
6.93
6.93
6.92
6.91
4.46
3.10
3.09
3.08
3.06
3.05
3.05
2.99
2.98
2.97
2.95
2.94
1.52
1.51
1.50
O
C
N
OH
F
27 1H NMR
(400 MHz, CDCl3)
2.04
2.01
1.00
8.17
3.06
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

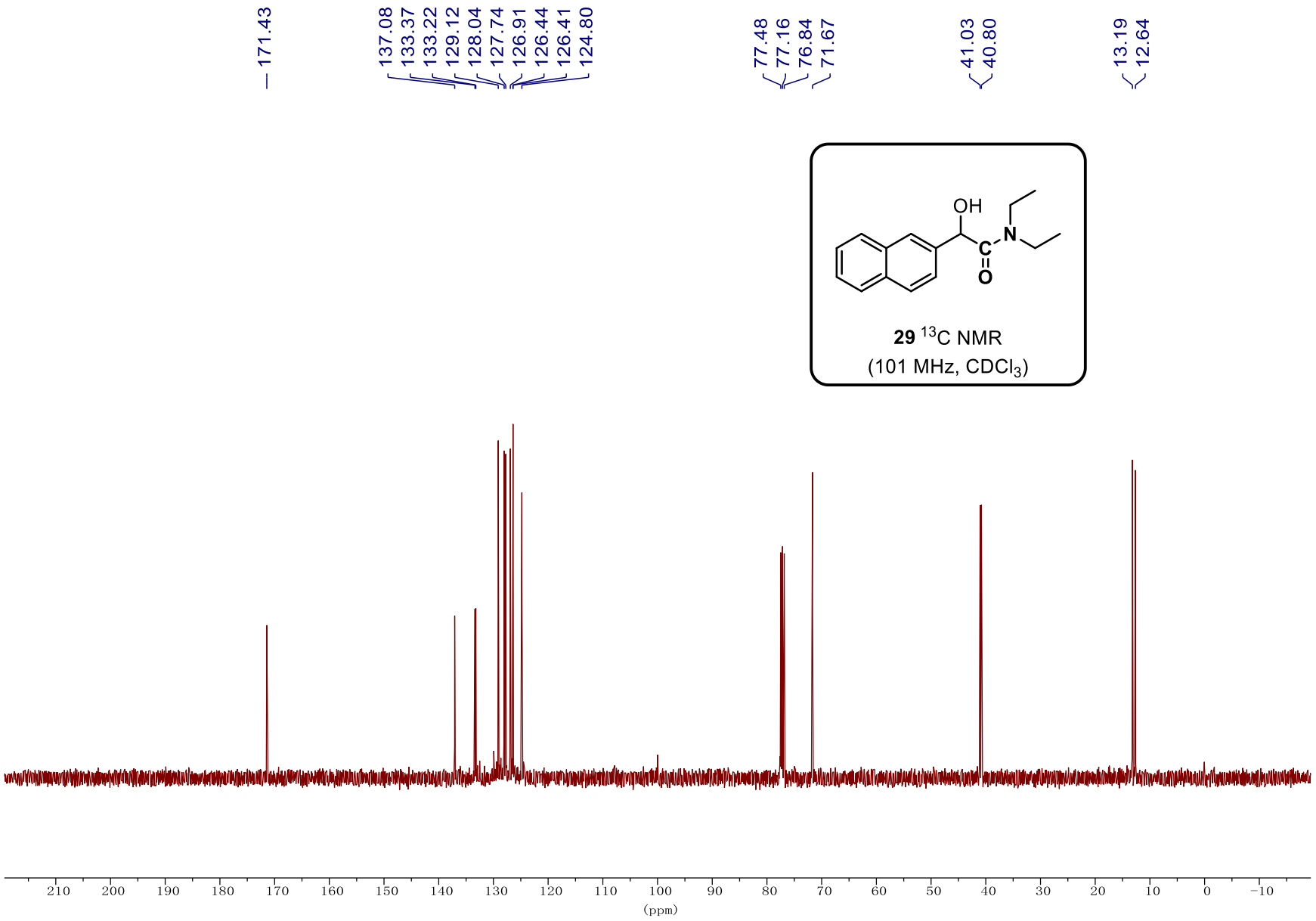

-116.34
-116.36
-116.37
-116.38
-116.40
O
C
N
OH
F
27 19F NMR
(376 MHz, CDCl3)
-116.34
-116.36
-116.37
-116.38
-116.40
-115.7 -115.9 -116.1 -116.3 -116.5 -116.7 -116.9
(ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
(ppm)

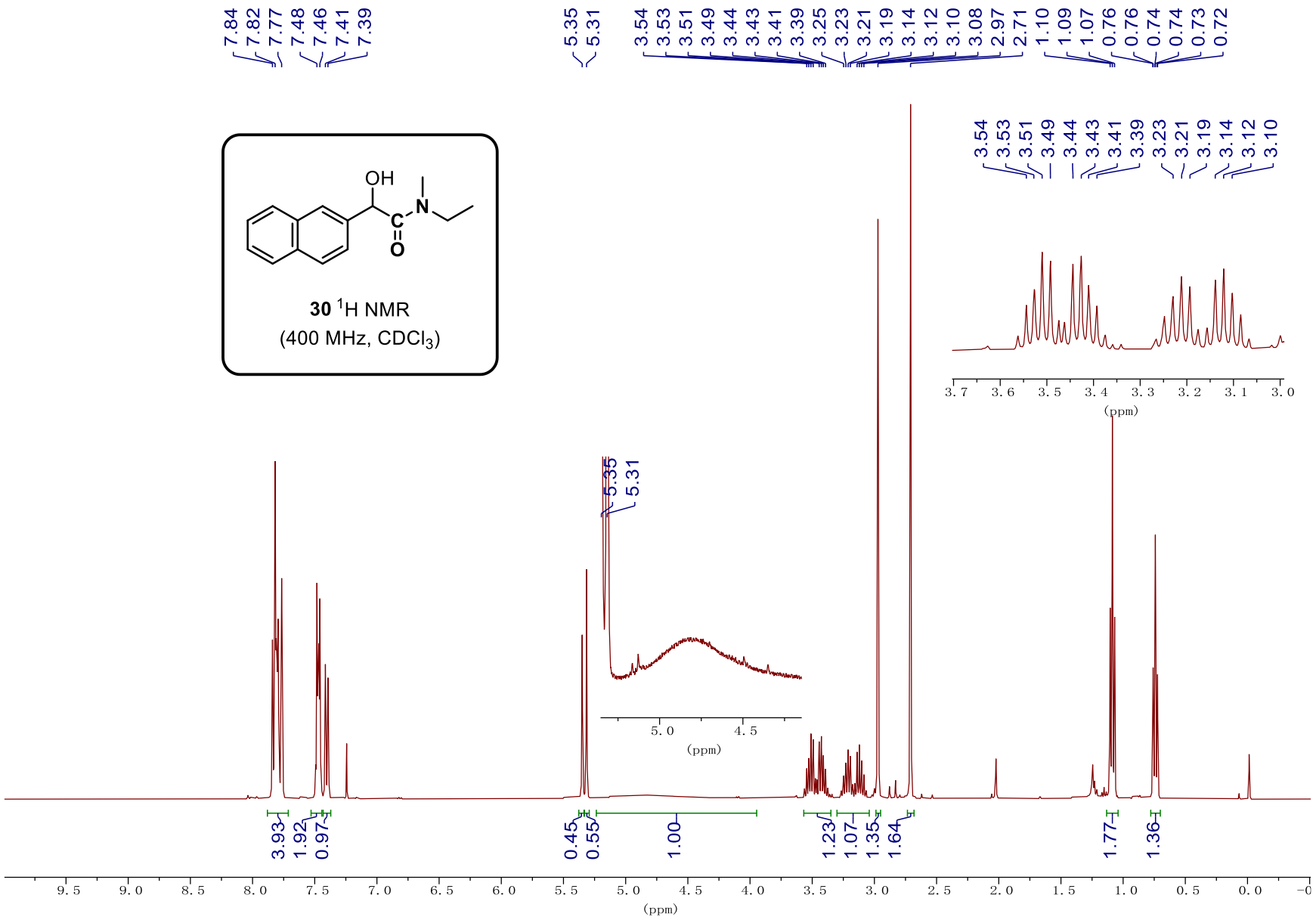

174.91
163.20
160.77
132.19
131.56
131.48
115.09
114.88
77.49
77.17
76.86
74.78
45.19
26.07
O
C
N
OH
F
27 13C NMR
(101 MHz, CDCl3)
132.19
131.56
131.48
163.20
160.77
134 133 132 131 130 129
165 164 163 162 161 160 159 158
(ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

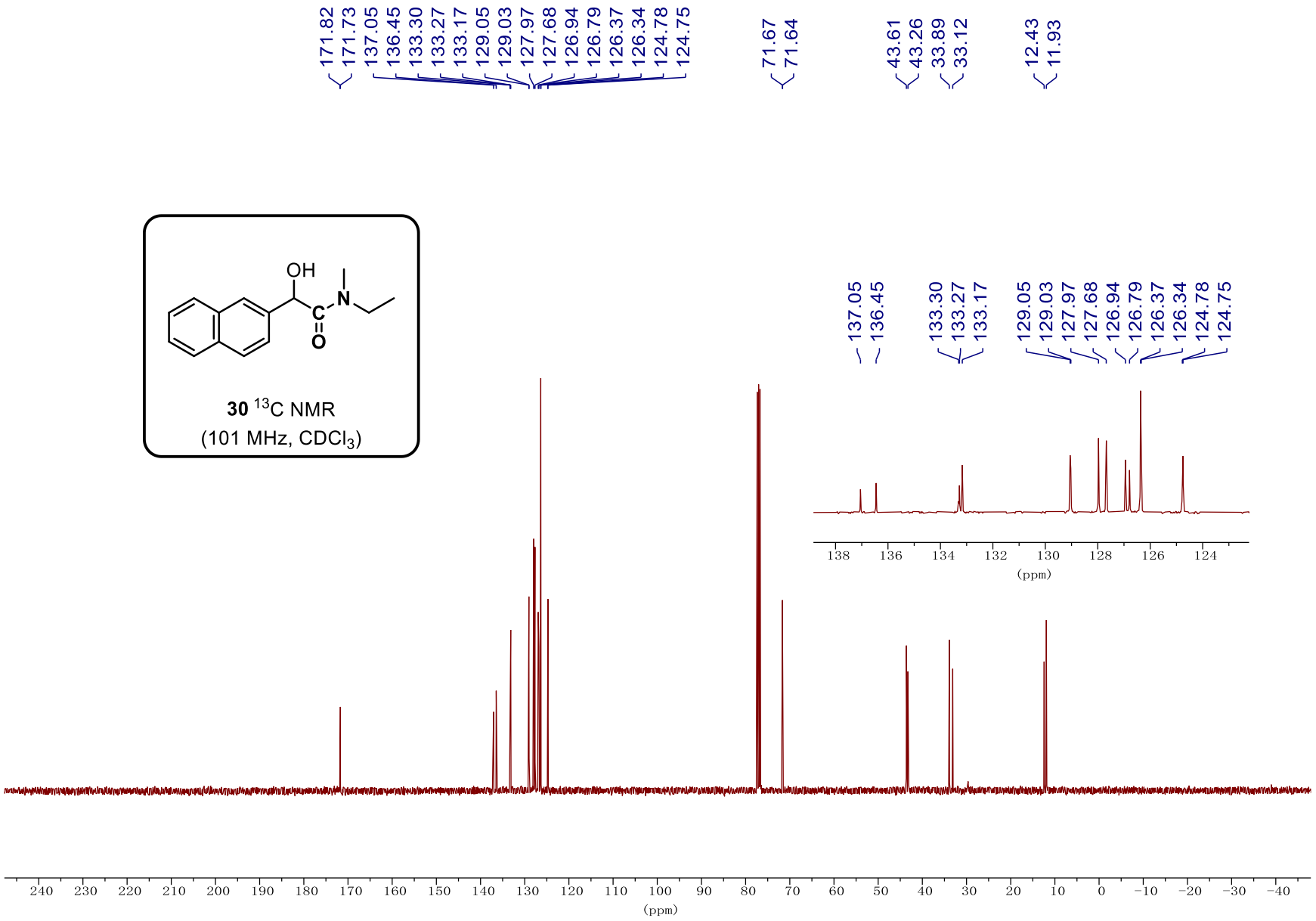

7.83
7.81
7.79
7.76
7.48
7.47
7.46
7.45
7.41
7.40
5.35
4.88
3.00
2.74
OH
N
C
O
28 1H NMR
(400 MHz, CDCl3)
3.91
1.90
0.95
1.00
0.87
2.98
2.96
(ppm)

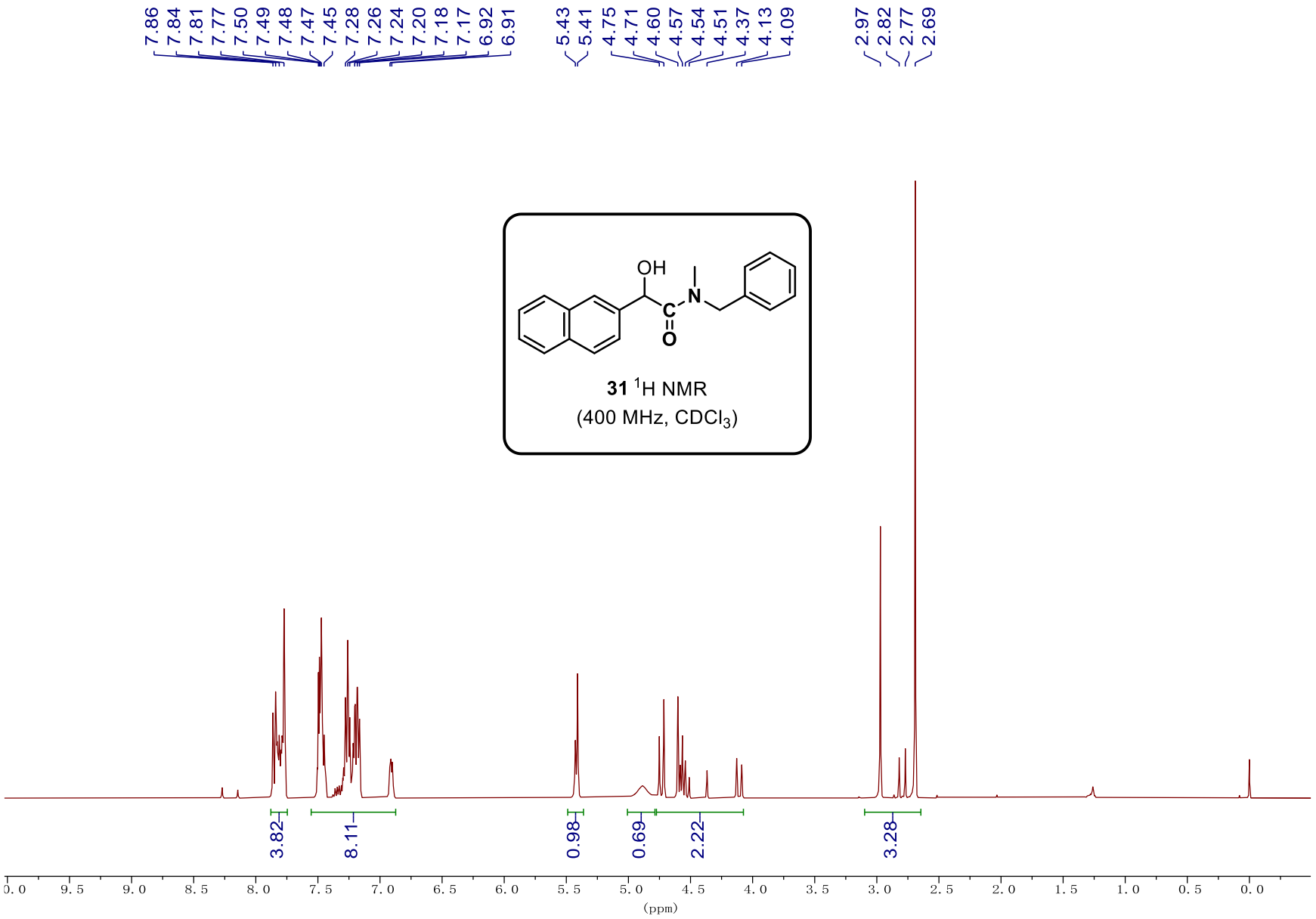

172.27
136.45
133.23
133.15
129.01
127.95
127.64
126.81
126.36
126.33
124.74
71.62
36.36
36.22
OH
N
C
O
28 13C NMR
(101 MHz, CDCl3)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
(ppm)

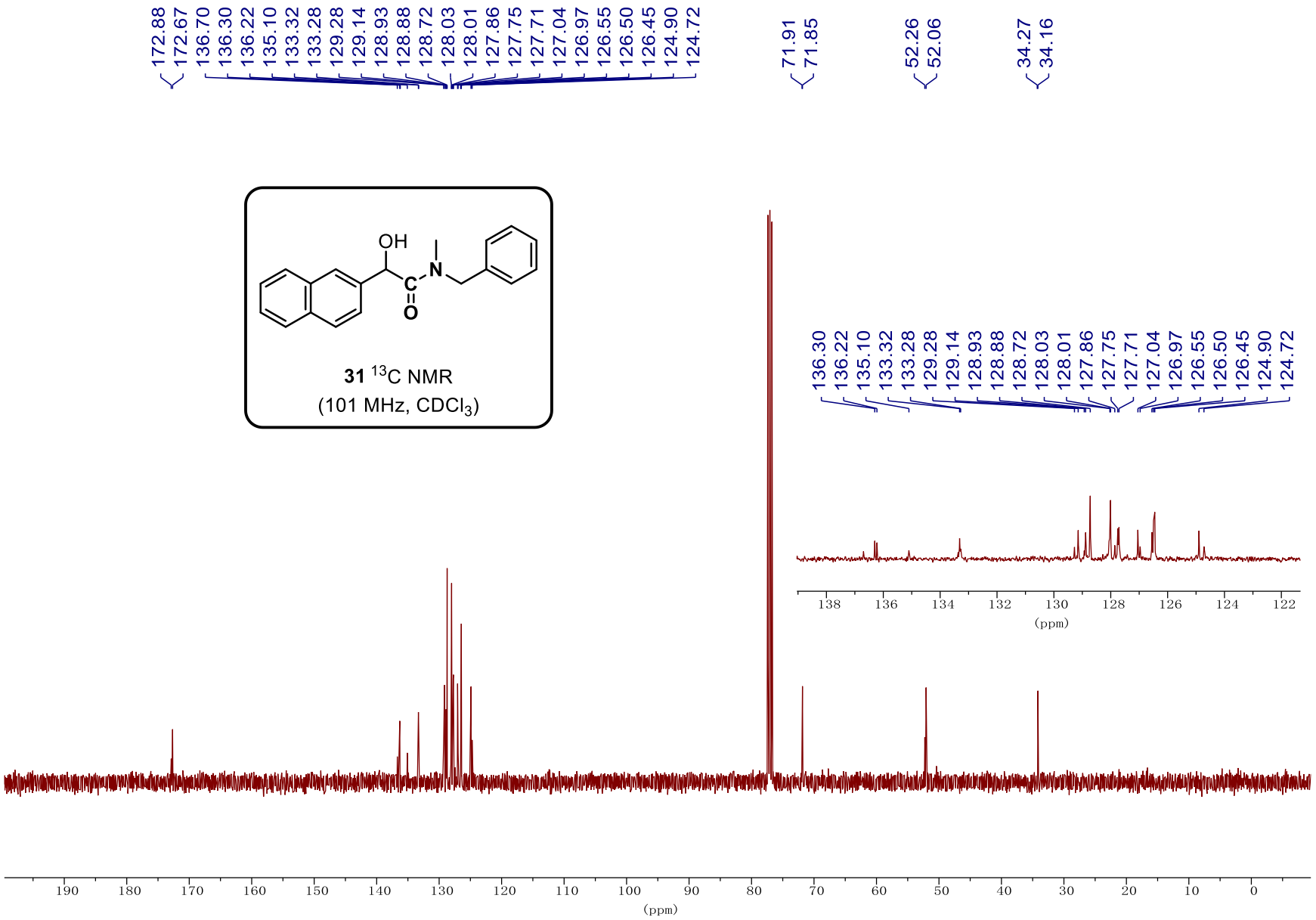

7.85 7.83 7.82 7.81 7.80 7.79 7.78 7.49 7.48 7.47 7.47 7.44 7.43 7.42 7.41 5.35 3.57 3.55 3.54 3.52 3.50 3.48 3.38 3.36 3.35 3.33 3.31 3.29 3.24 3.23 3.21 3.19 3.17 3.15 3.10 3.08 3.06 3.05 3.03 3.01 1.15 1.14 1.12 0.79 0.77 0.75
OH
N
C
O
29 1H NMR
(400 MHz, CDCl3)
3.55 3.54 3.52 3.50 3.38 3.36 3.35 3.33 3.31 3.23 3.21 3.19 3.17 3.15 3.10 3.08 3.06 3.05 3.03
1.15 1.14 1.12
0.79 0.77 0.75
(ppm)
4.12 3.02 0.83 3.58 2.80 2.54
(ppm)

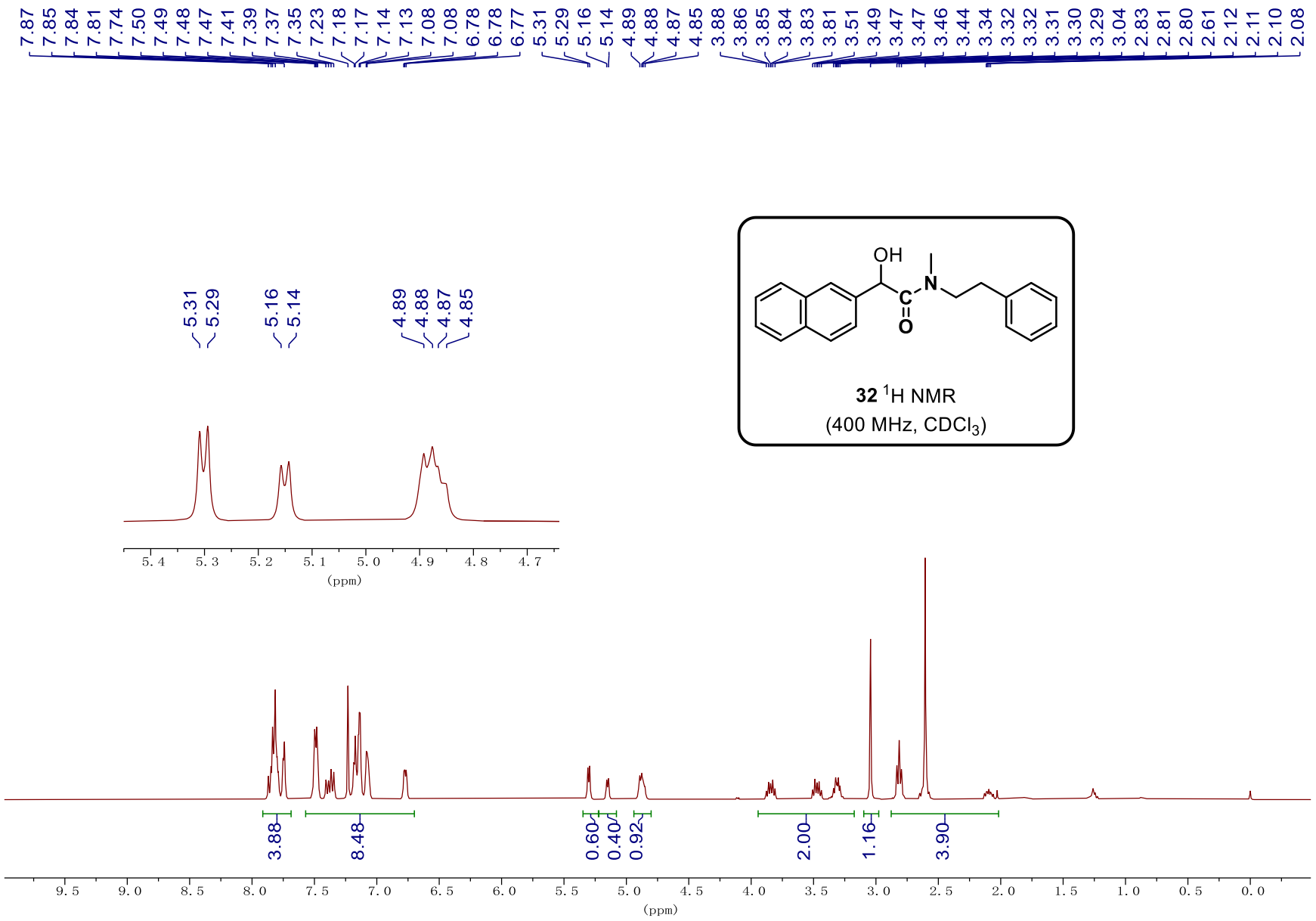

171.43
137.08
133.37
133.22
129.12
128.04
127.74
126.91
126.44
126.41
124.80
77.48
77.16
76.84
71.67
41.03
40.80
13.19
12.64
OH
N
C
O
29 13C NMR
(101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

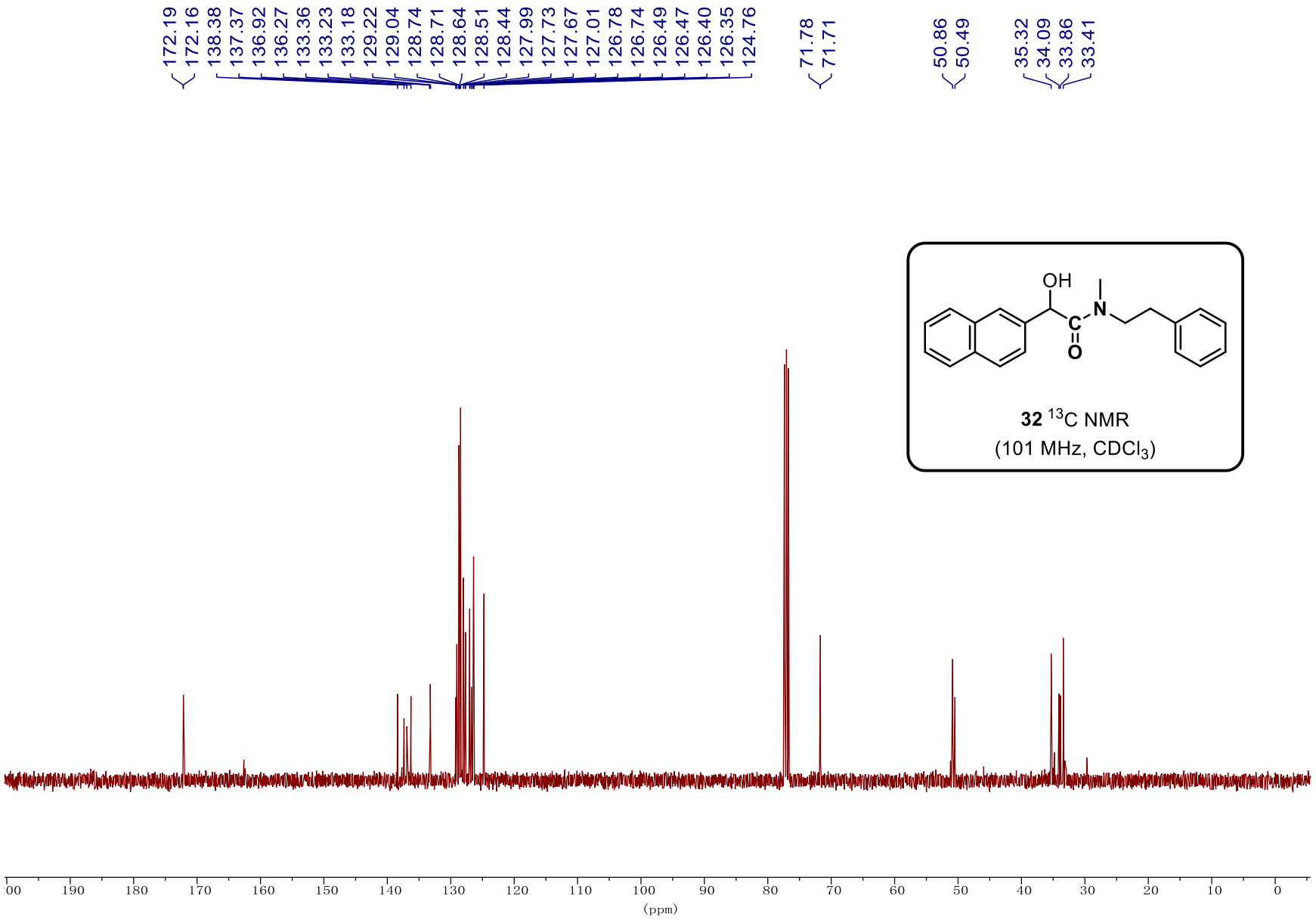

7.84
7.82
7.77
7.48
7.46
7.41
7.39
5.35
5.31
3.54
3.53
3.51
3.49
3.44
3.43
3.41
3.39
3.25
3.23
3.21
3.19
3.14
3.12
3.10
3.08
2.97
2.71
1.10
1.09
1.07
0.76
0.76
0.74
0.74
0.73
0.72
OH
N
C
O
30 1H NMR
(400 MHz, CDCl3)
(ppm)
3.93
1.92
0.97
0.45
0.55
1.00
1.23
1.07
1.35
1.64
1.77
1.36

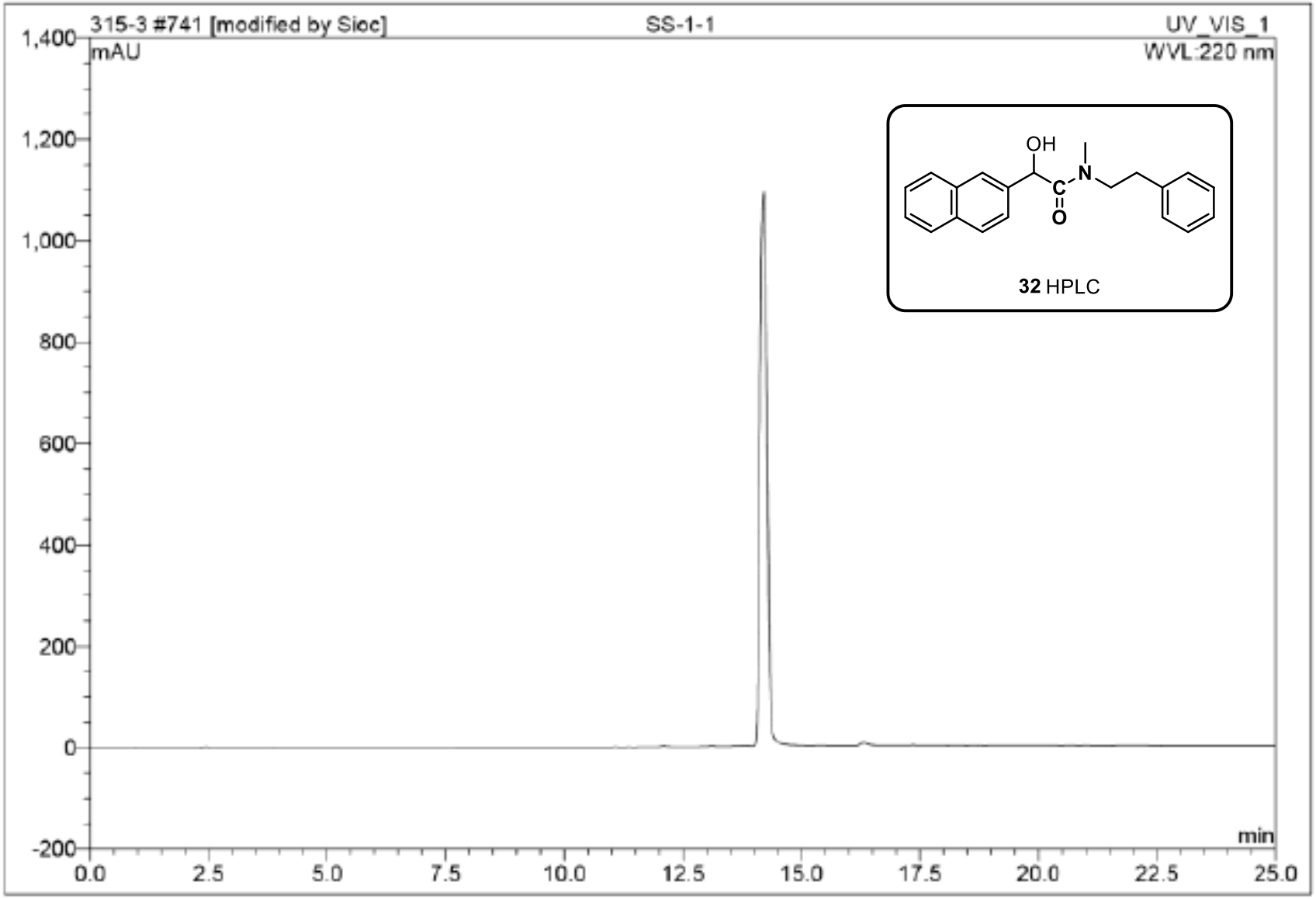

171.82
171.73
137.05
136.45
133.30
133.27
133.17
129.05
129.03
127.97
127.68
126.94
126.79
126.37
126.34
124.78
124.75
71.67
71.64
43.61
43.26
33.89
33.12
12.43
11.93
OH
N
C
O
30 13C NMR
(101 MHz, CDCl3)
138
136
134
132
130
128
126
124
(ppm)
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
(ppm)

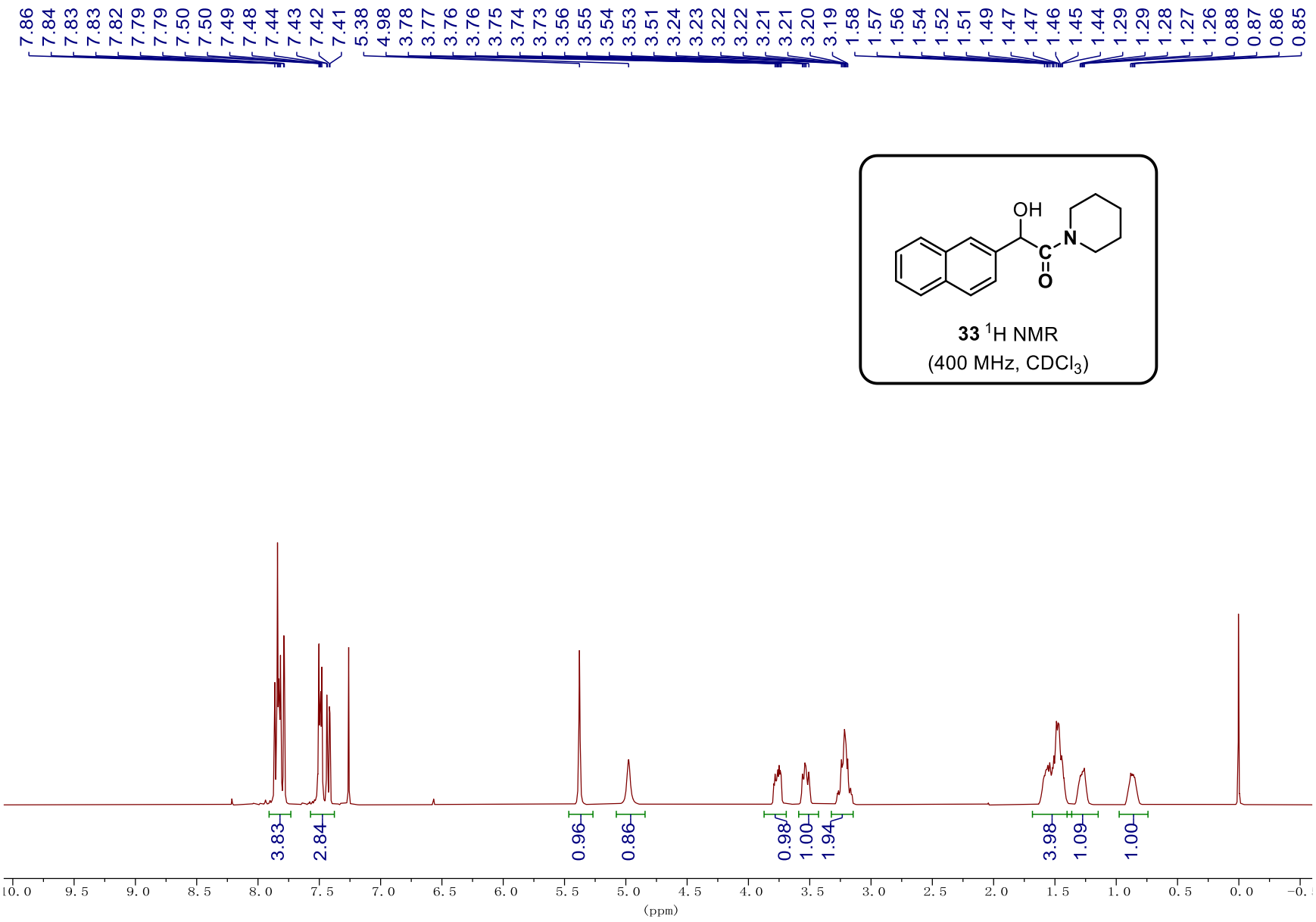

7.86
7.84
7.81
7.77
7.50
7.49
7.48
7.47
7.45
7.28
7.26
7.24
7.20
7.18
7.17
6.92
6.91
5.43
5.41
4.75
4.71
4.60
4.57
4.54
4.51
4.37
4.13
4.09
2.97
2.82
2.77
2.69
OH
N
C
O
31 1H NMR
(400 MHz, CDCl3)
3.82
8.11
0.98
0.69
2.22
3.28
(ppm)

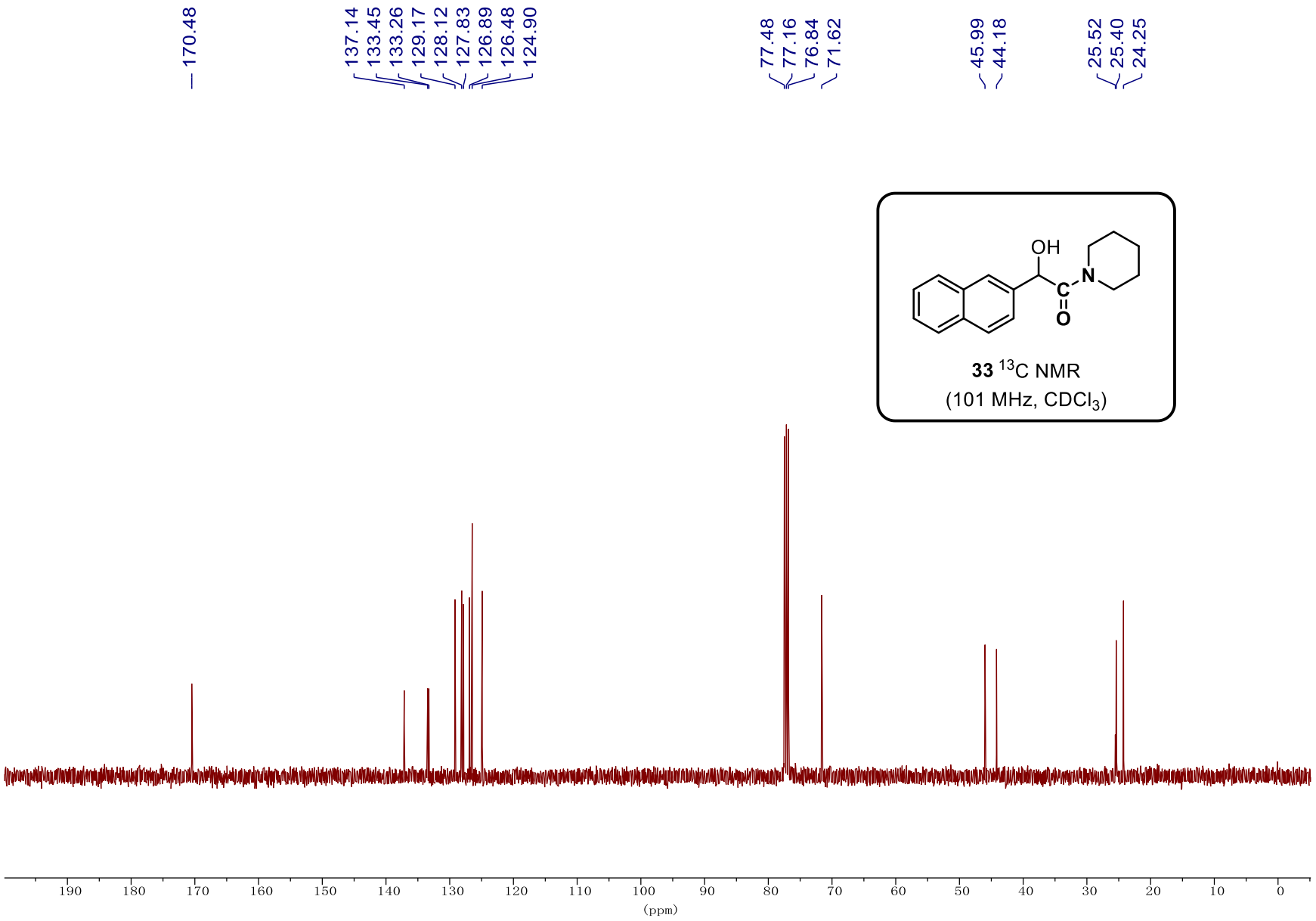

172.88
172.67
136.70
136.30
136.22
135.10
133.32
133.28
129.28
129.14
128.93
128.88
128.72
128.03
128.01
127.86
127.75
127.71
127.04
126.97
126.55
126.50
126.45
124.90
124.72
71.91
71.85
52.26
52.06
34.27
34.16
OH
N
C
O
31 13C NMR
(101 MHz, CDCl3)
(ppm)
(ppm)

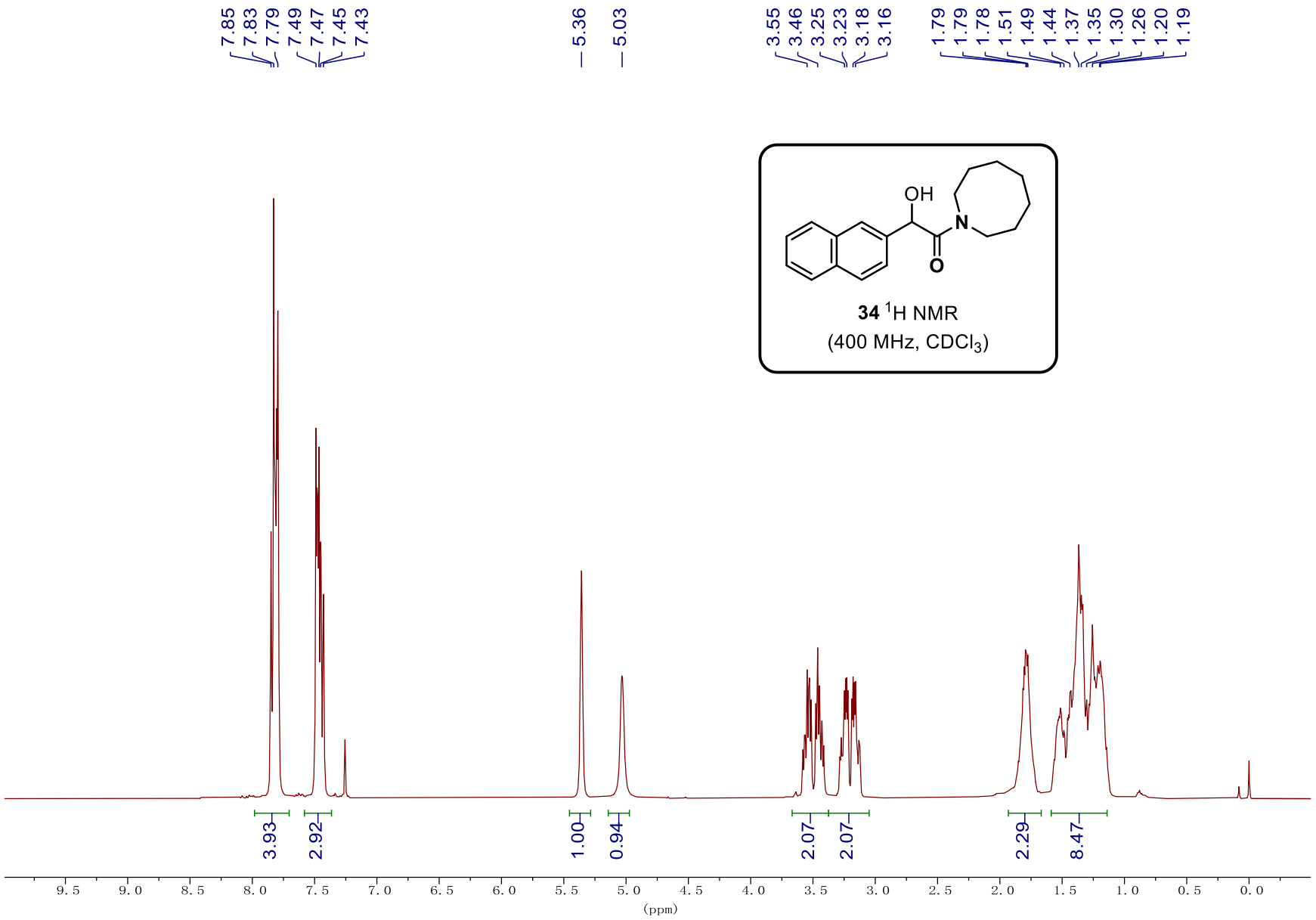

7.87 7.85 7.84 7.81 7.74 7.50 7.49 7.48 7.47 7.41 7.39 7.37 7.35 7.23 7.18 7.17 7.14 7.13 7.08 7.08 6.78 6.78 6.77
5.31 5.29 5.16 5.14 4.89 4.88 4.87 4.85
3.88 3.86 3.85 3.84 3.83 3.81 3.51 3.49 3.47 3.47 3.46 3.44 3.34 3.32 3.32 3.31 3.30 3.29 3.04 2.83 2.81 2.80 2.61 2.12 2.11 2.10 2.08
OH
N
C
O
32 1H NMR
(400 MHz, CDCl3)
(ppm)
3.88 8.48 0.60 0.40 0.92 2.00 1.16 3.90

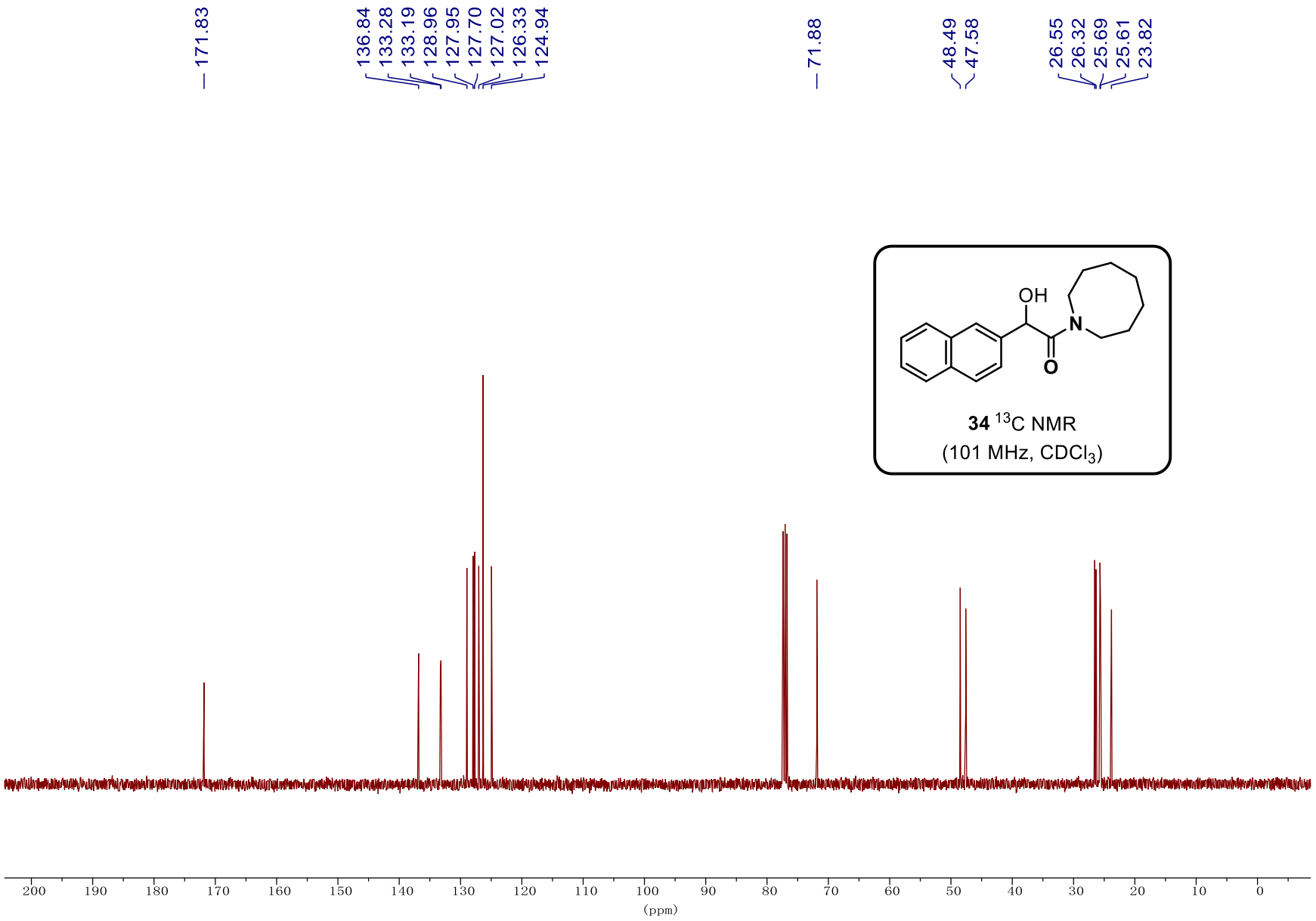

172.19
172.16
138.38
137.37
136.92
136.27
133.36
133.23
133.18
129.22
129.04
128.74
128.71
128.64
128.51
128.44
127.99
127.73
127.67
127.01
126.78
126.74
126.49
126.47
126.40
126.35
124.76
71.78
71.71
50.86
50.49
35.32
34.09
33.86
33.41
OH
N
C
O
32 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

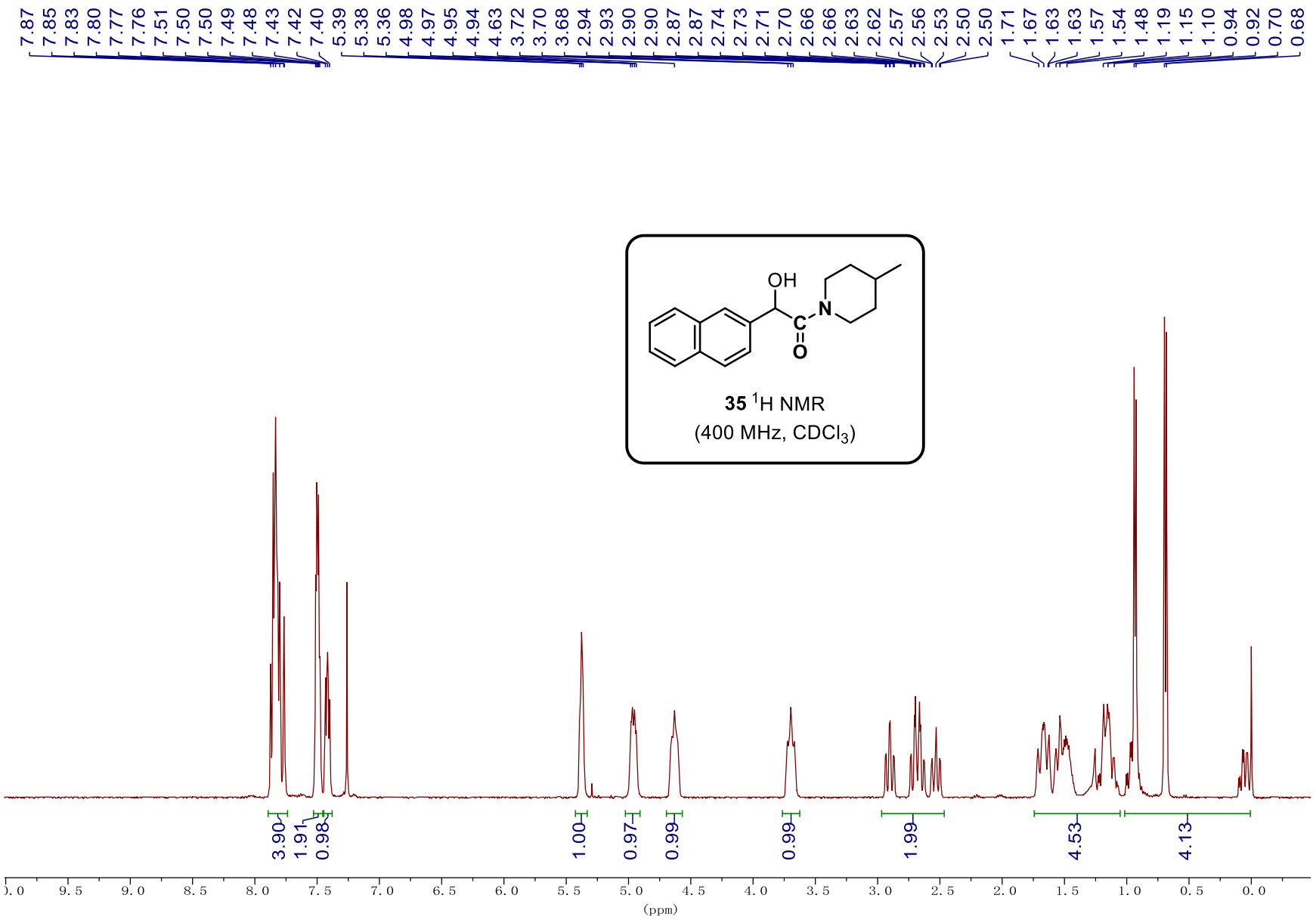

315-3 #741 [modified by Sioc]
SS-1-1
UV_VIS_1
WVL:220 nm
mAU
1,400
1,200
1,000
800
600
400
200
0
-200
min
0.0
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
OH
N
C
O
32 HPLC

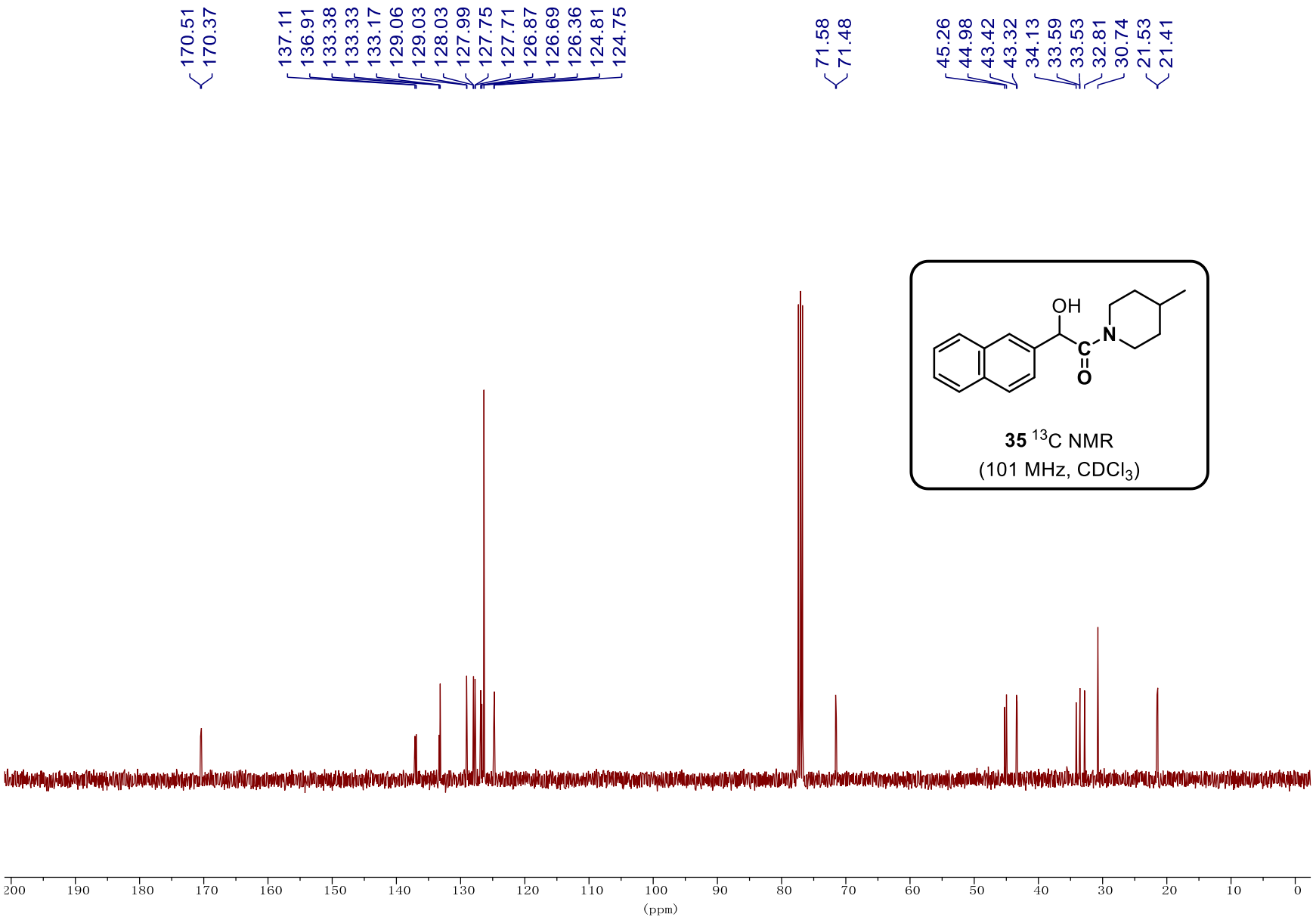

7.86
7.84
7.83
7.83
7.82
7.79
7.79
7.50
7.50
7.49
7.48
7.44
7.43
7.42
7.41
5.38
4.98
3.78
3.77
3.76
3.76
3.75
3.74
3.73
3.56
3.55
3.54
3.53
3.51
3.24
3.23
3.22
3.22
3.21
3.21
3.20
3.19
1.58
1.57
1.56
1.54
1.52
1.51
1.49
1.47
1.47
1.46
1.45
1.44
1.29
1.29
1.28
1.27
1.26
0.88
0.87
0.86
0.85
OH
N
C
O
33 1H NMR
(400 MHz, CDCl3)
3.83
2.84
0.96
0.86
0.98
1.00
1.94
3.98
1.09
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

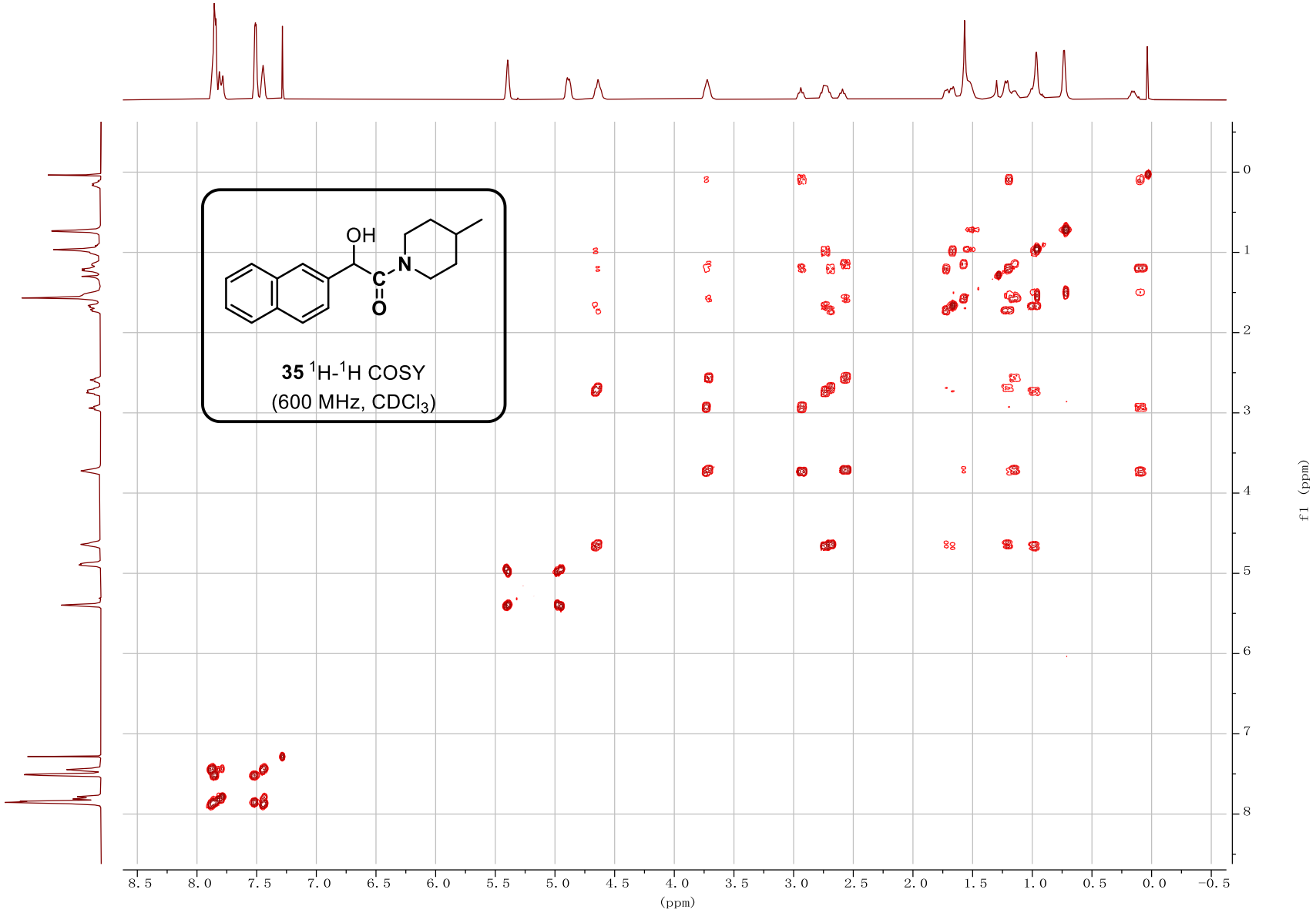

170.48
137.14
133.45
133.26
129.17
128.12
127.83
126.89
126.48
124.90
77.48
77.16
76.84
71.62
45.99
44.18
25.52
25.40
24.25
OH
N
C
O
33 13C NMR
(101 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

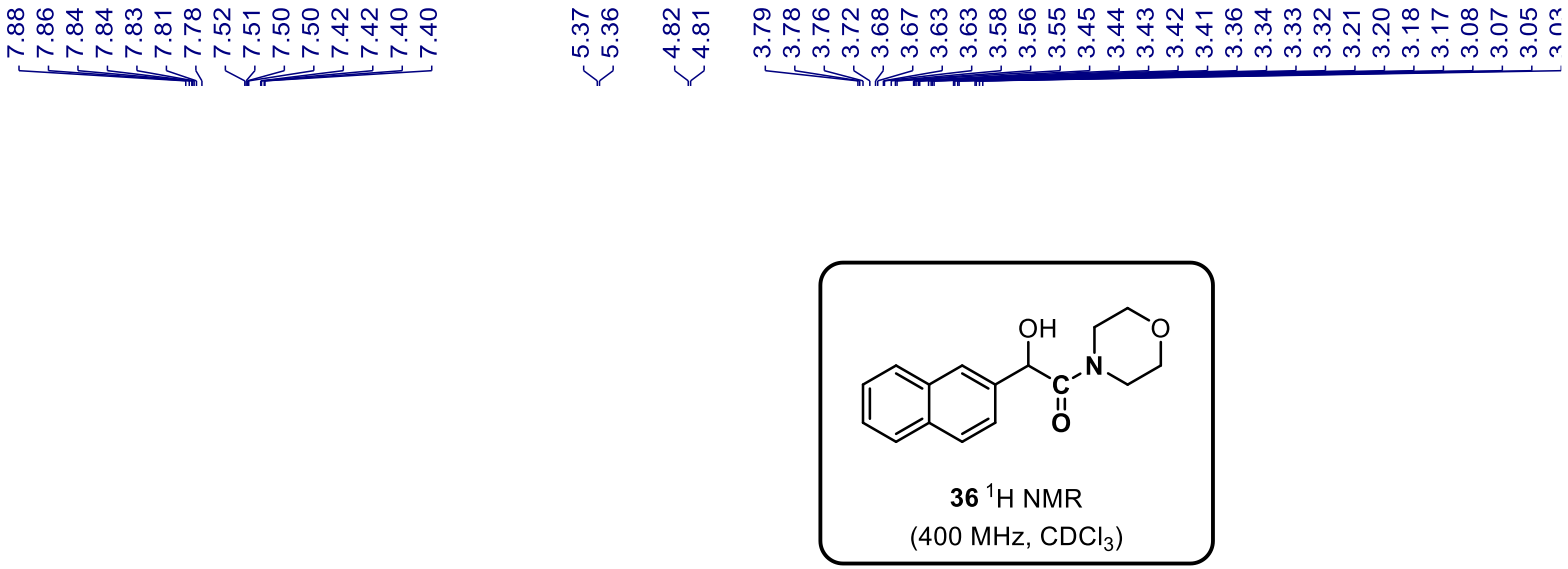

7.85
7.83
7.79
7.49
7.47
7.45
7.43
5.36
5.03
3.55
3.46
3.25
3.23
3.18
3.16
1.79
1.79
1.78
1.51
1.49
1.44
1.37
1.35
1.30
1.26
1.20
1.19
OH
N
O
34 1H NMR
(400 MHz, CDCl3)
3.93
2.92
1.00
0.94
2.07
2.07
2.29
8.47
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

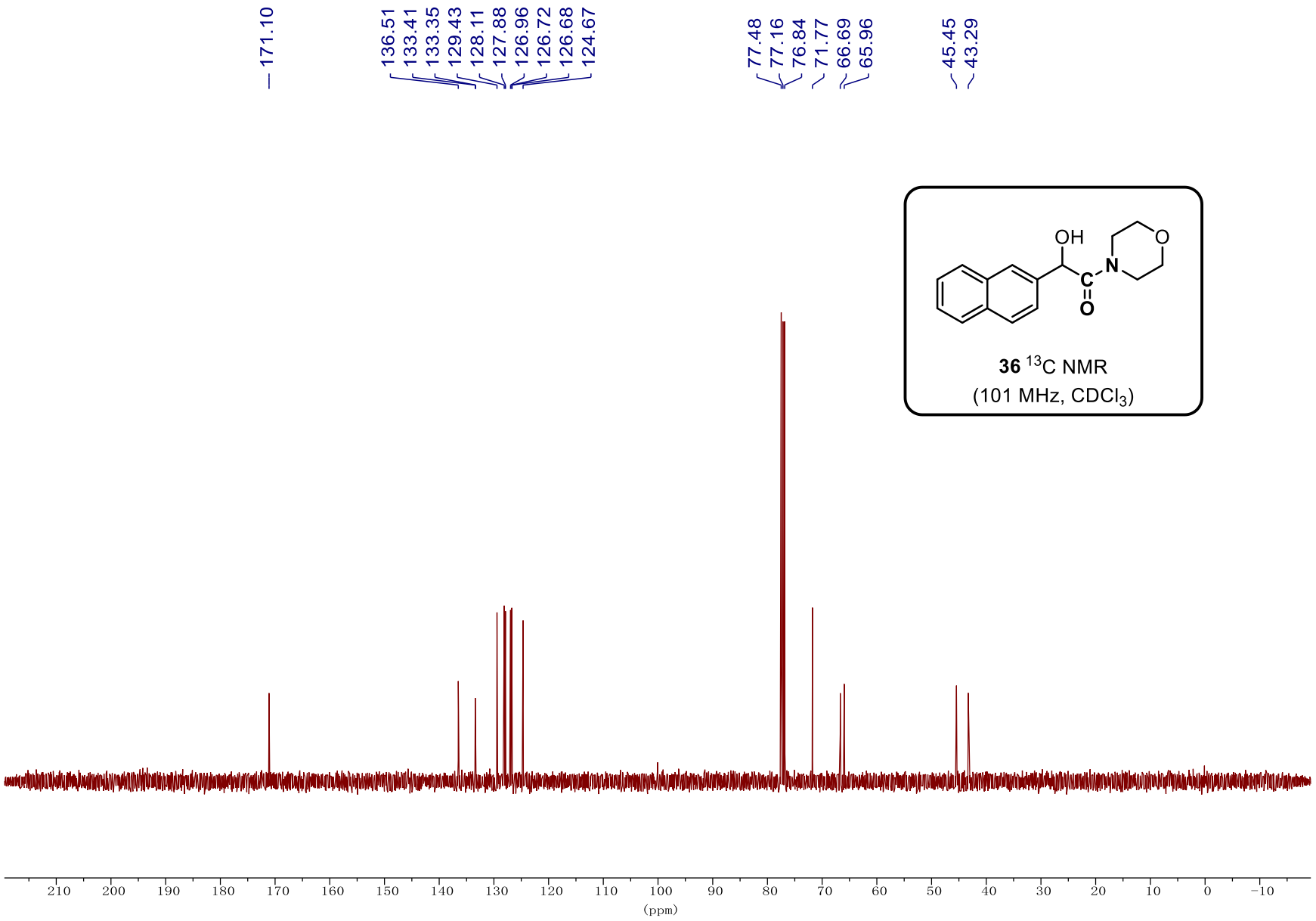

171.83
136.84
133.28
133.19
128.96
127.95
127.70
127.02
126.33
124.94
71.88
48.49
47.58
26.55
26.32
25.69
25.61
23.82
OH
N
O
34 13C NMR
(101 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

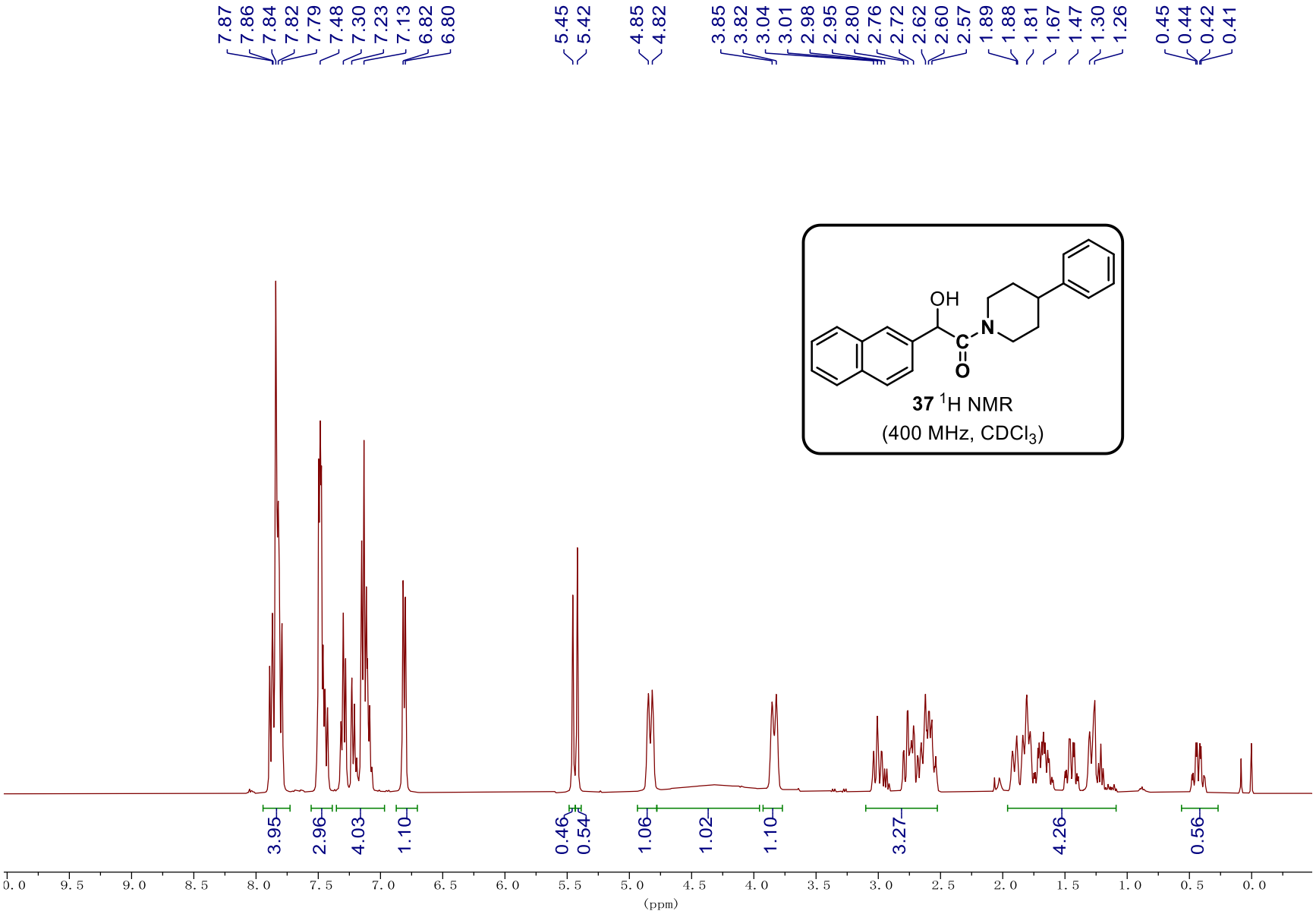

7.87 7.85 7.83 7.80 7.77 7.76 7.51 7.50 7.50 7.49 7.48 7.43 7.42 7.40 5.39 5.38 5.36 4.98 4.97 4.95 4.94 4.63 3.72 3.70 3.68 2.94 2.93 2.90 2.90 2.87 2.87 2.74 2.73 2.71 2.70 2.66 2.66 2.63 2.62 2.57 2.56 2.53 2.50 2.50 1.71 1.67 1.63 1.63 1.57 1.54 1.48 1.19 1.15 1.10 0.94 0.92 0.70 0.68
OH
N
C
O
35 1H NMR
(400 MHz, CDCl3)
3.90 1.91 0.98 1.00 0.97 0.99 0.99 1.99 4.53 4.13
(ppm)

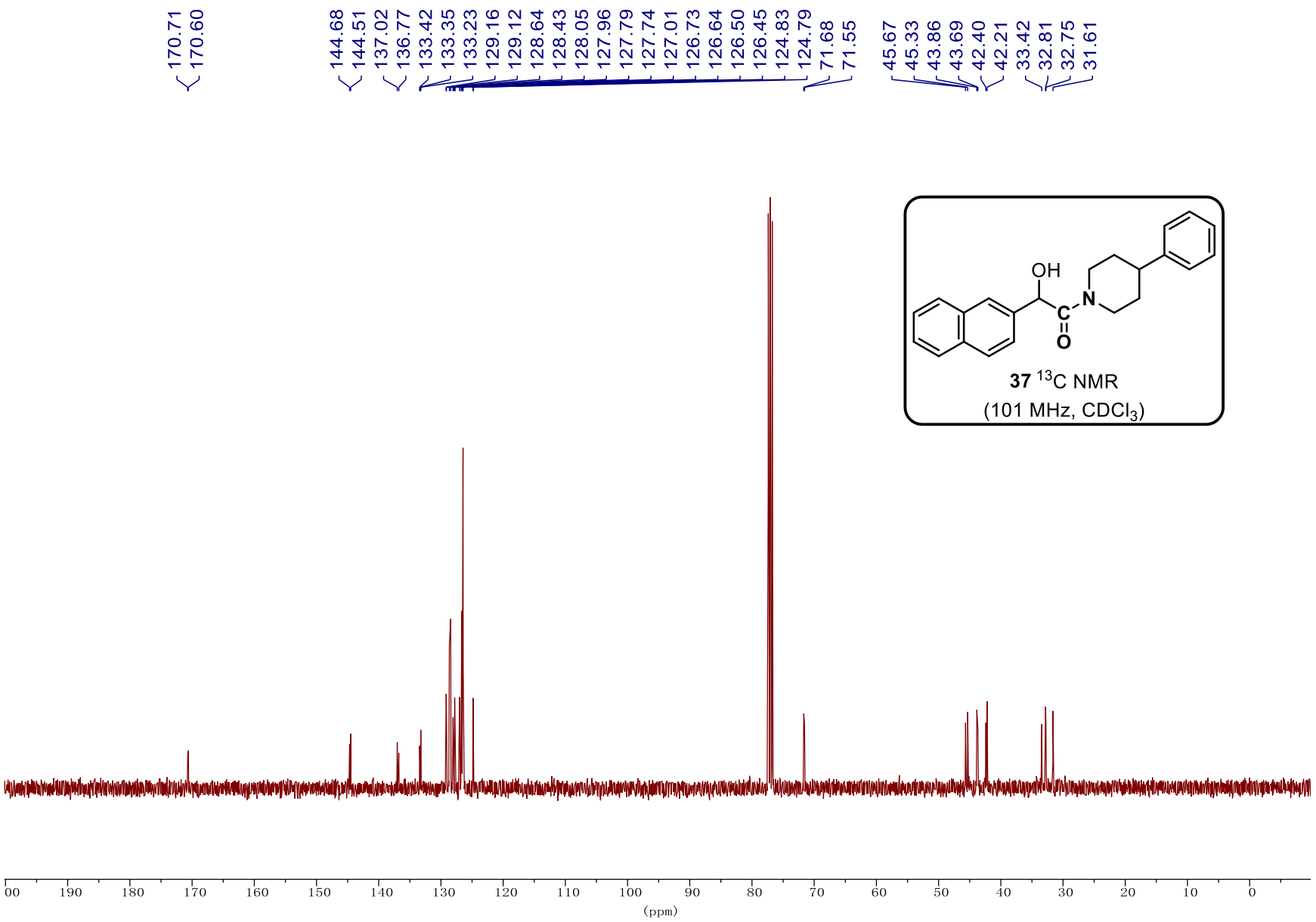

170.51
170.37
137.11
136.91
133.38
133.33
133.17
129.06
129.03
128.03
127.99
127.75
127.71
126.87
126.69
126.36
124.81
124.75
71.58
71.48
45.26
44.98
43.42
43.32
34.13
33.59
33.53
32.81
30.74
21.53
21.41
OH
N
C
O
35 13C NMR
(101 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

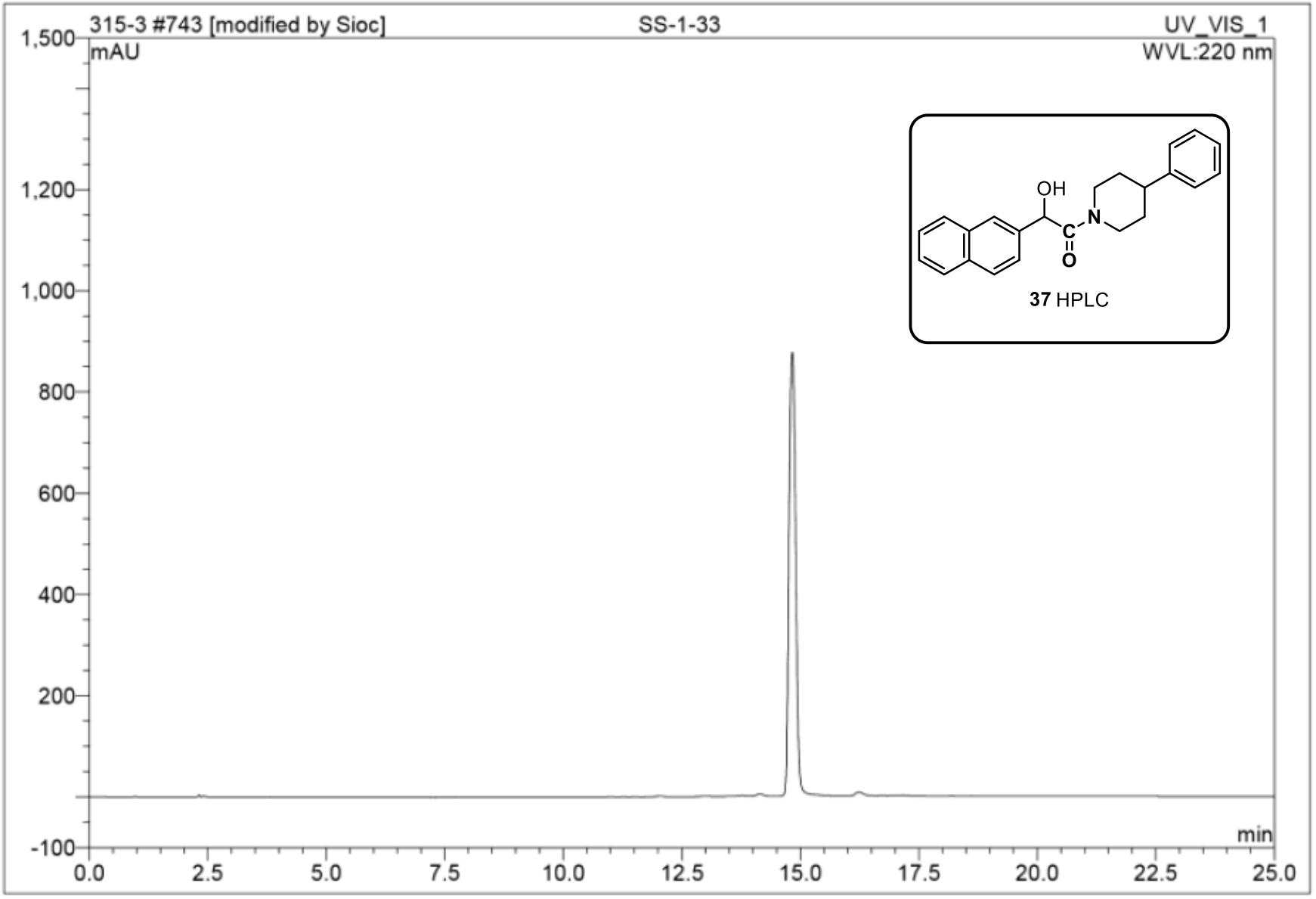

OH
N
C
O
35 ^{1}H-^{1}H COSY
(600 MHz, $CDCl_3$)
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
(ppm)
0 1 2 3 4 5 6 7 8
f1 (ppm)

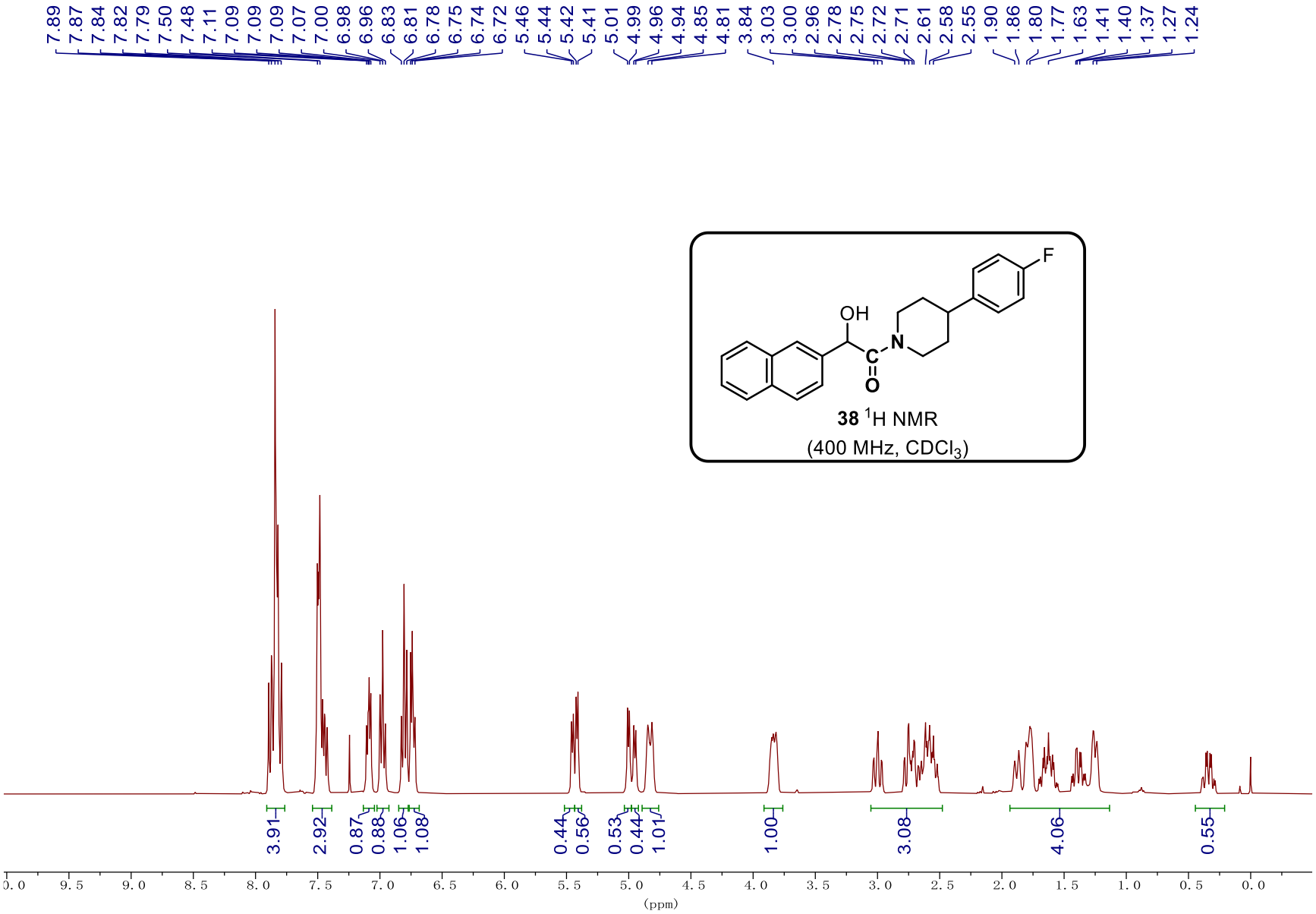

7.88
7.86
7.84
7.84
7.83
7.81
7.78
7.52
7.51
7.50
7.50
7.42
7.42
7.40
7.40
5.37
5.36
4.82
4.81
3.79
3.78
3.76
3.72
3.68
3.67
3.63
3.63
3.58
3.56
3.55
3.45
3.44
3.43
3.42
3.41
3.36
3.34
3.33
3.32
3.21
3.20
3.18
3.17
3.08
3.07
3.05
OH
O
N
C
O
36 1H NMR
(400 MHz, CDCl3)
3.91
1.91
0.95
1.00
0.92
8.28
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

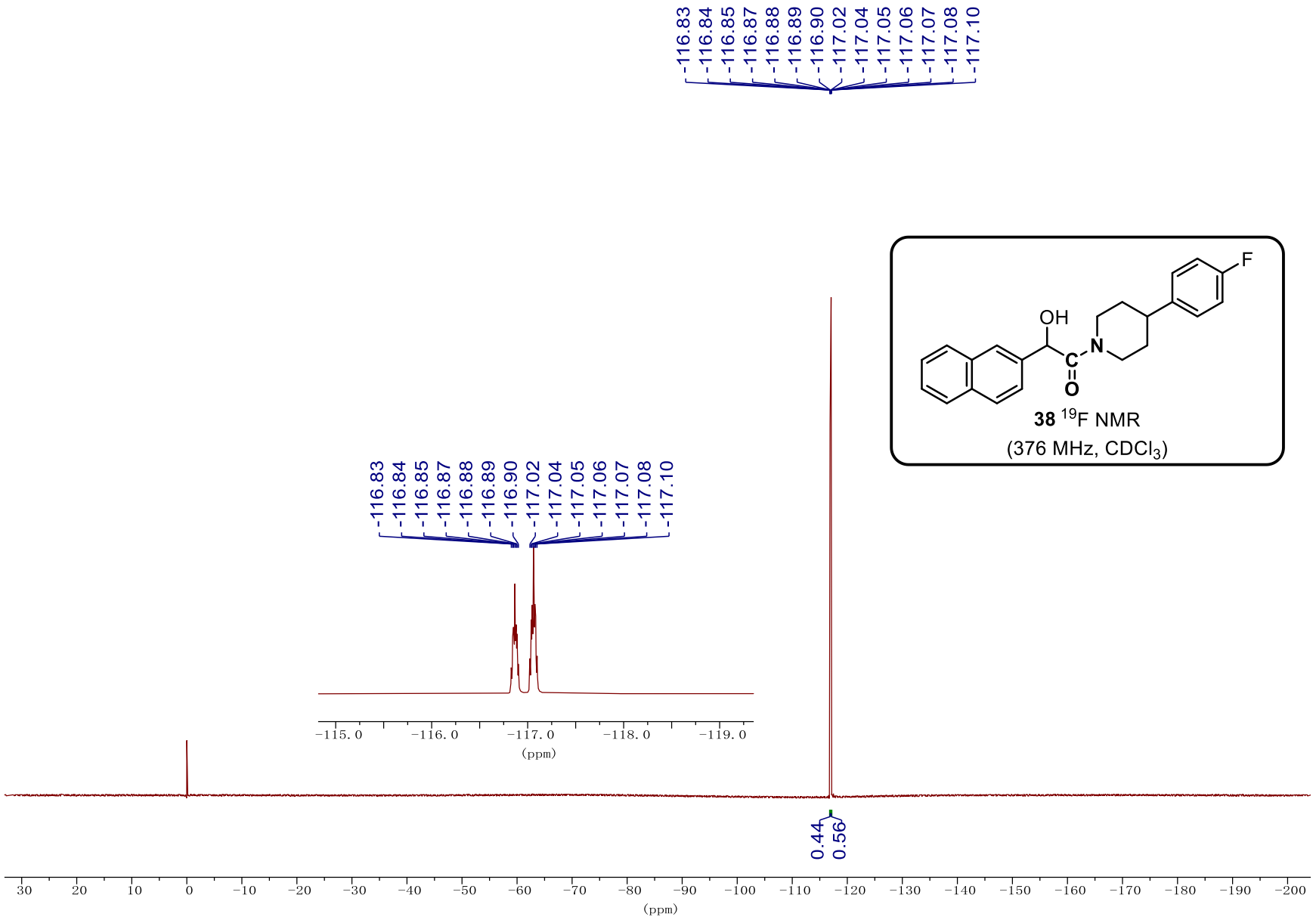

171.10
136.51
133.41
133.35
129.43
128.11
127.88
126.96
126.72
126.68
124.67
77.48
77.16
76.84
71.77
66.69
65.96
45.45
43.29
OH
O
N
C
O
36 13C NMR
(101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
(ppm)

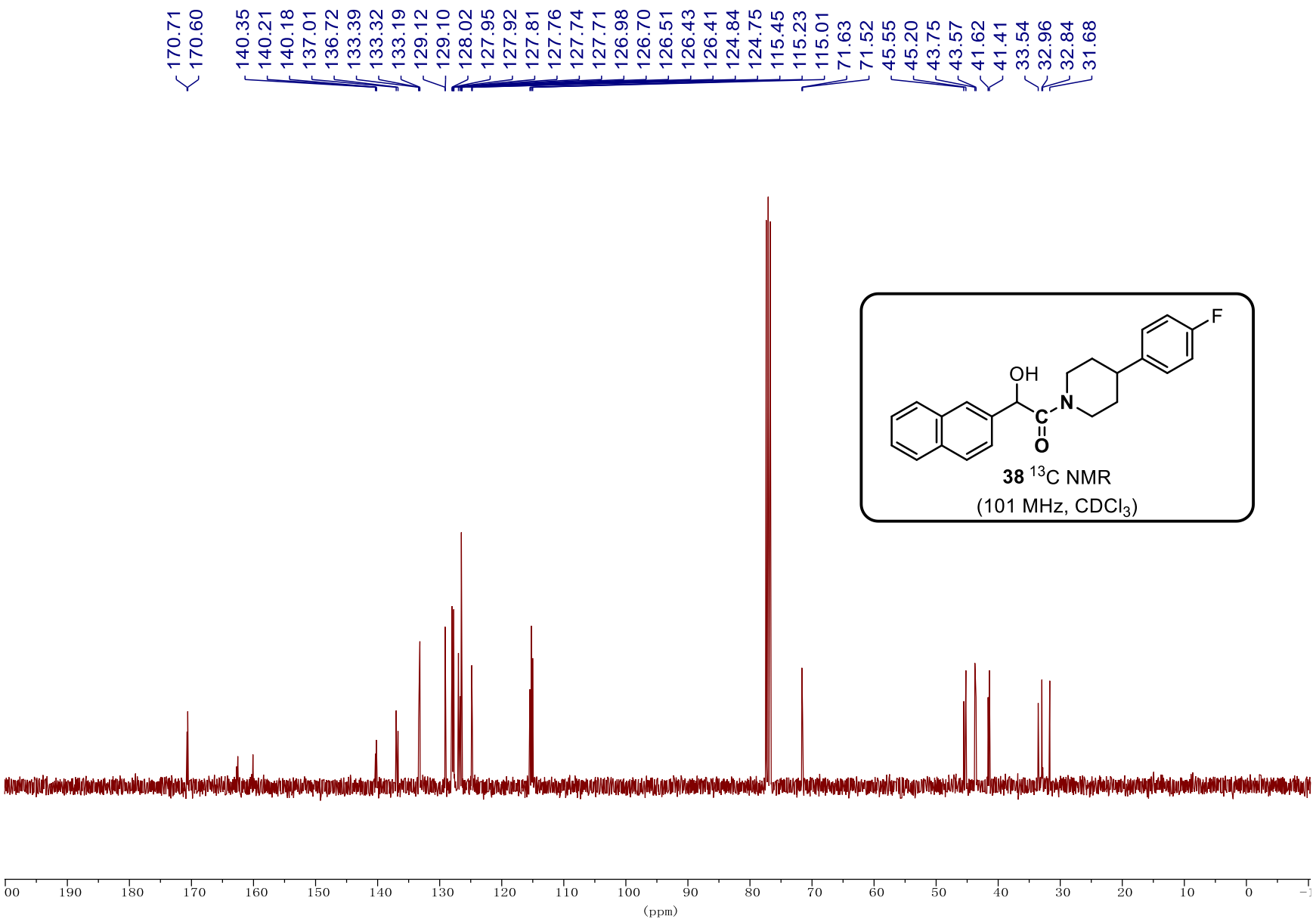

7.87
7.86
7.84
7.82
7.79
7.48
7.30
7.23
7.13
6.82
6.80
5.45
5.42
4.85
4.82
3.85
3.82
3.04
3.01
2.98
2.95
2.80
2.76
2.72
2.62
2.60
2.57
1.89
1.88
1.81
1.67
1.47
1.30
1.26
0.45
0.44
0.42
0.41
OH
N
C
O
37 1H NMR
(400 MHz, CDCl3)
3.95
2.96
4.03
1.10
0.46
0.54
1.06
1.02
1.10
3.27
4.26
0.56
(ppm)

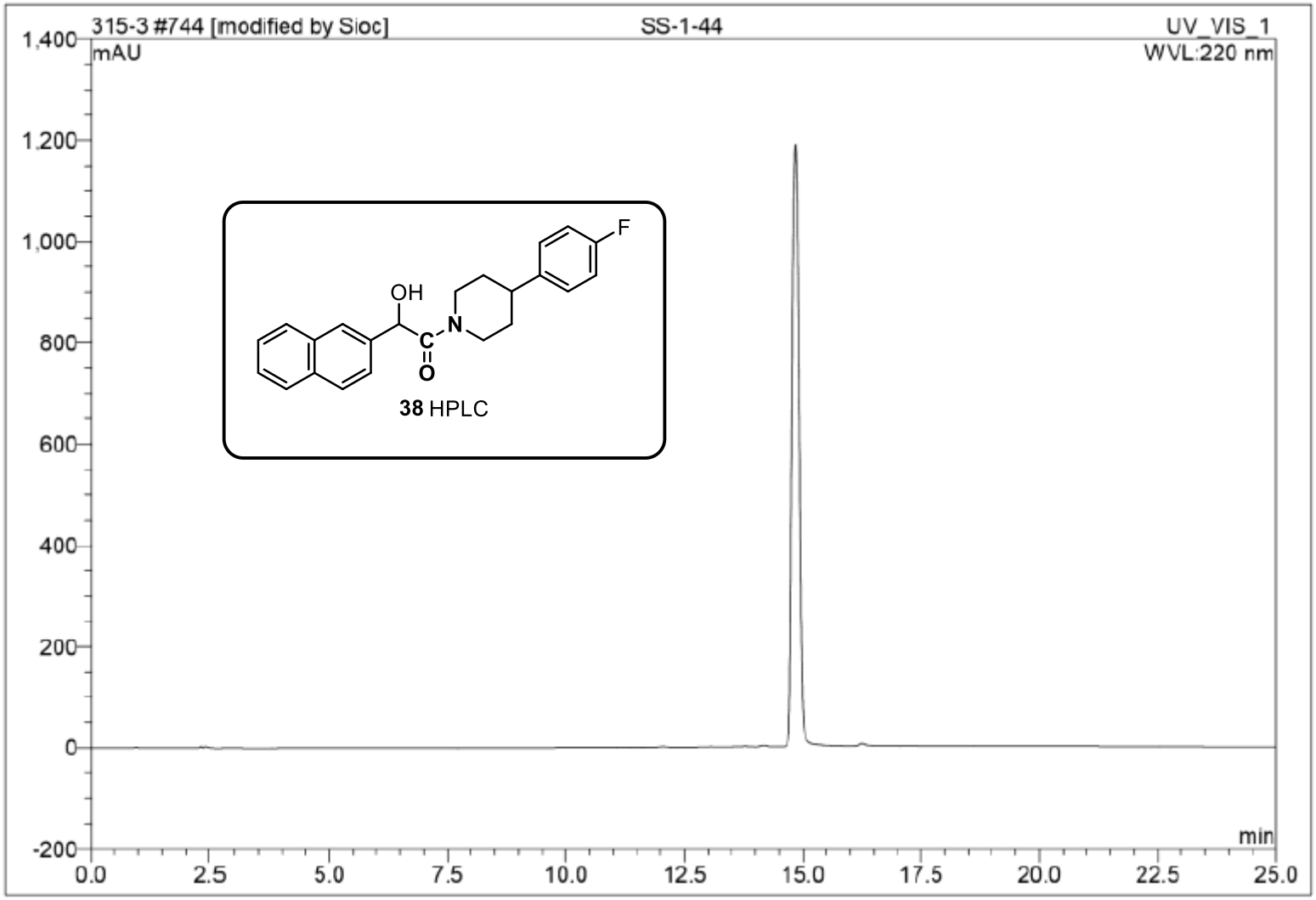

170.71
170.60
144.68
144.51
137.02
136.77
133.42
133.35
133.23
129.16
129.12
128.64
128.43
128.05
127.96
127.79
127.74
127.01
126.73
126.64
126.50
126.45
124.83
124.79
71.68
71.55
45.67
45.33
43.86
43.69
42.40
42.21
33.42
32.81
32.75
31.61
OH
N
C
O
37 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

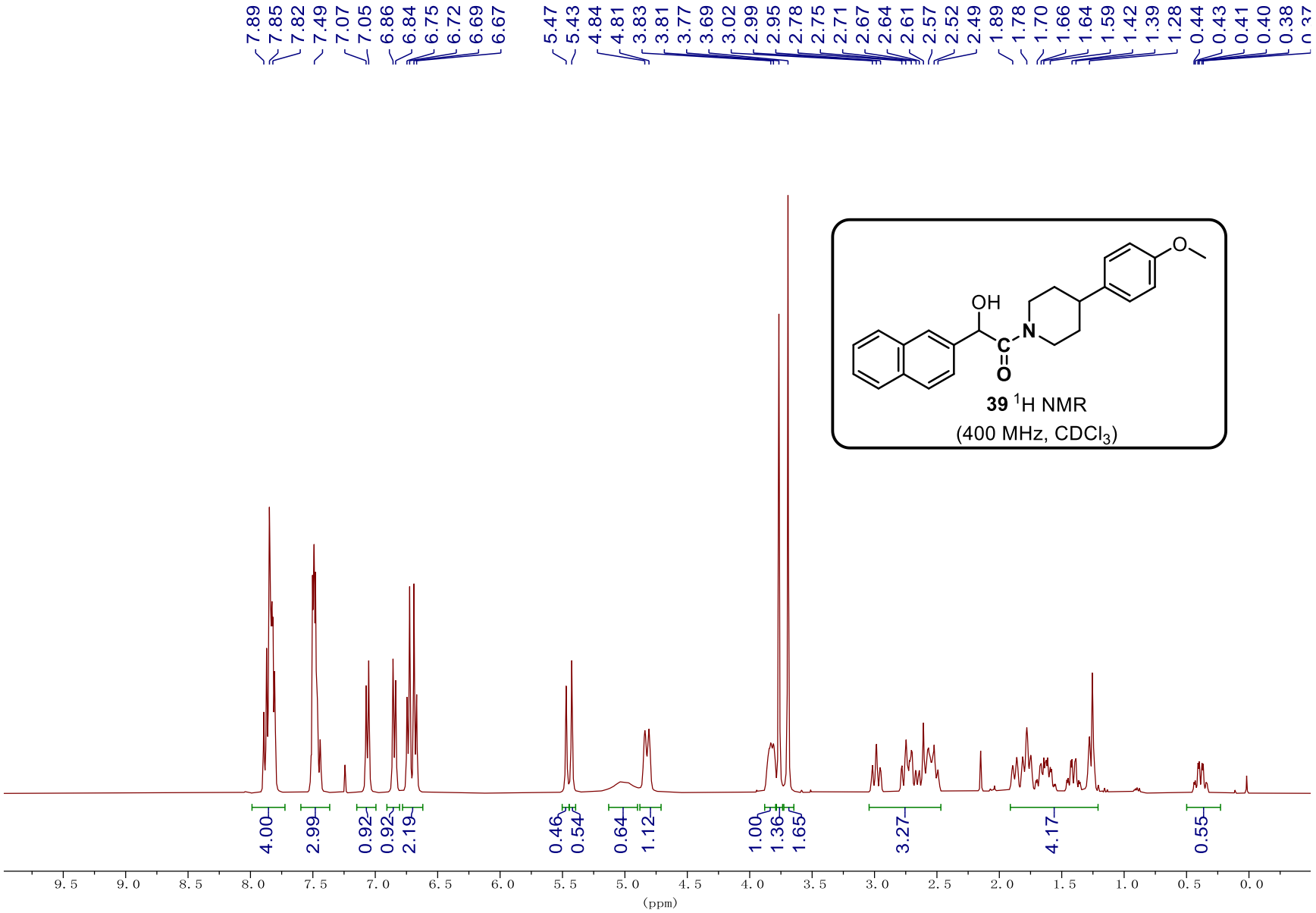

315-3 #743 [modified by Sioc]
SS-1-33
UV_VIS_1
WVL:220 nm
mAU
1,500
1,200
1,000
800
600
400
200
-100
min
0.0
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
OH
N
C
O
37 HPLC

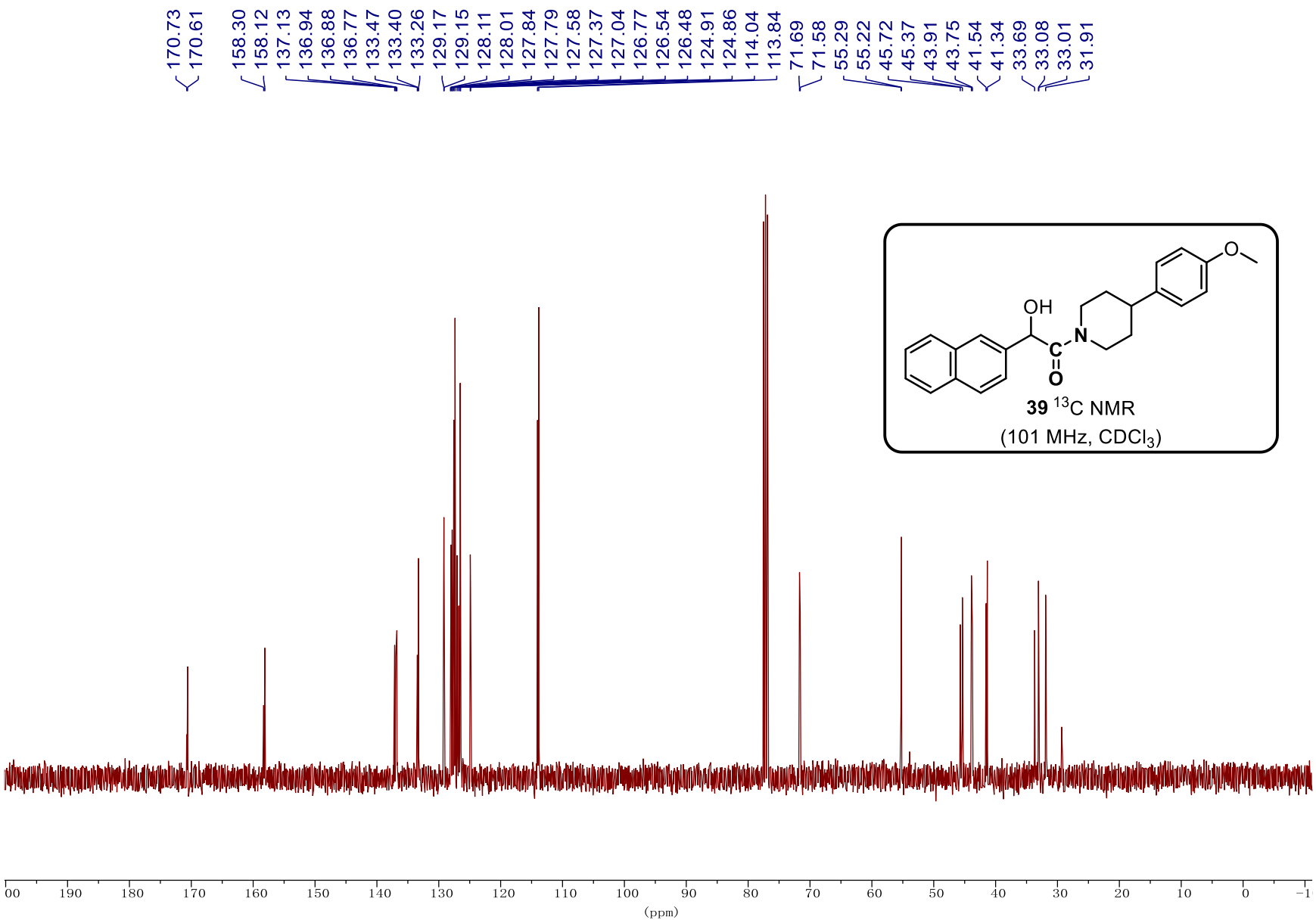

7.89 7.87 7.84 7.82 7.79 7.50 7.48 7.11 7.09 7.09 7.09 7.07 7.00 6.98 6.96 6.83 6.81 6.78 6.75 6.74 6.72
5.46 5.44 5.42 5.41 5.01 4.99 4.96 4.94 4.85 4.81 3.84 3.03 3.00 2.96 2.78 2.75 2.72 2.71 2.61 2.58 2.55
1.90 1.86 1.80 1.77 1.63 1.41 1.40 1.37 1.27 1.24
F
OH
N
C
O
38 1H NMR
(400 MHz, CDCl3)
3.91 2.92 0.87 0.88 1.06 1.08 0.44 0.56 0.53 0.44 1.01 1.00 3.08 4.06 0.55
(ppm)

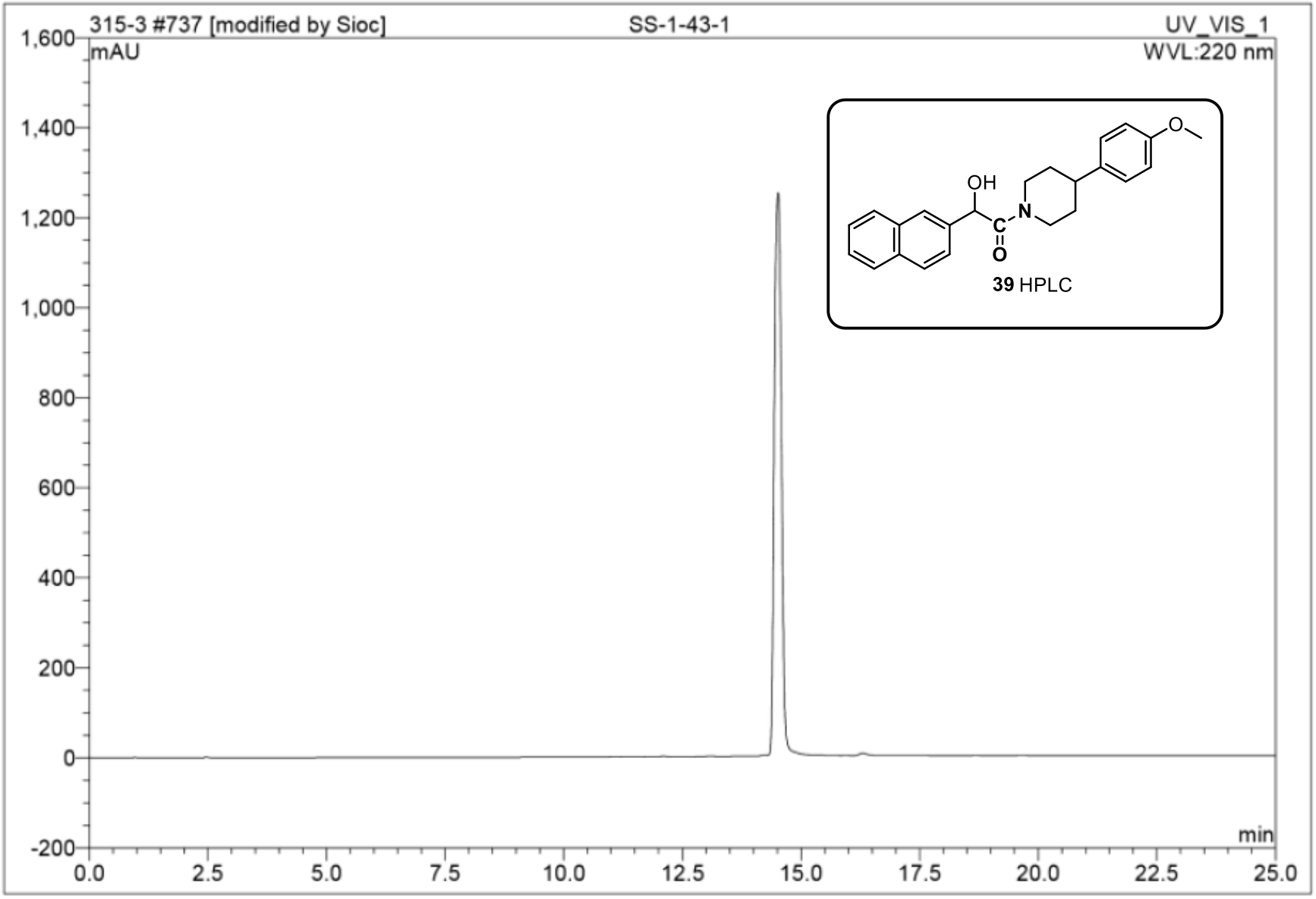

-116.83
-116.84
-116.85
-116.87
-116.88
-116.89
-116.90
-117.02
-117.04
-117.05
-117.06
-117.07
-117.08
-117.10
F
OH
N
C
O
38 19F NMR
(376 MHz, CDCl3)
-115.0
-116.0
-117.0
-118.0
-119.0
(ppm)
0.44
0.56
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
(ppm)

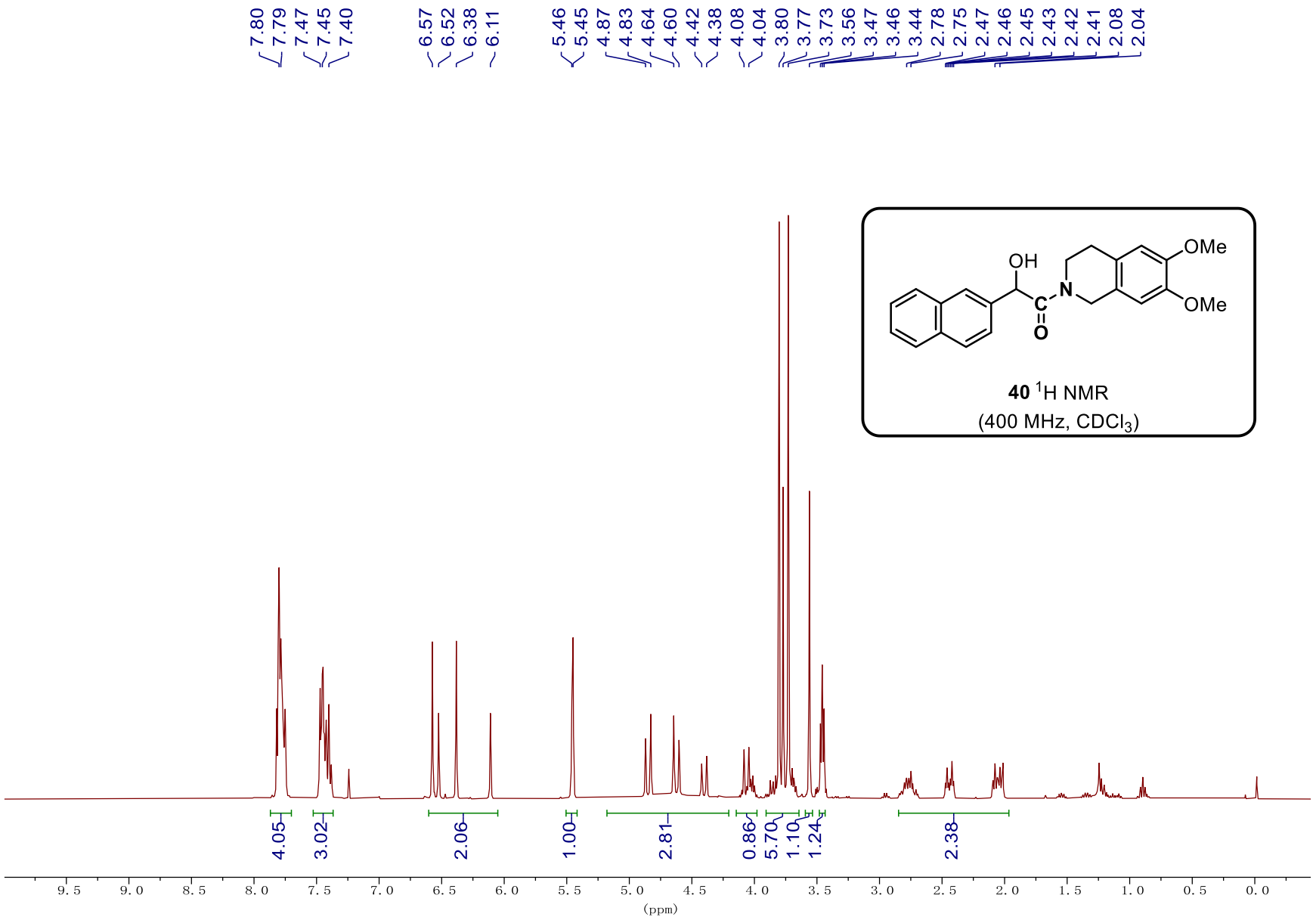

170.71
170.60
140.35
140.21
140.18
137.01
136.72
133.39
133.32
133.19
129.12
129.10
128.02
127.95
127.92
127.81
127.76
127.74
127.71
126.98
126.70
126.51
126.43
126.41
124.84
124.75
115.45
115.23
115.01
71.63
71.52
45.55
45.20
43.75
43.57
41.62
41.41
33.54
32.96
32.84
31.68
F
OH
N
C
O
38 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

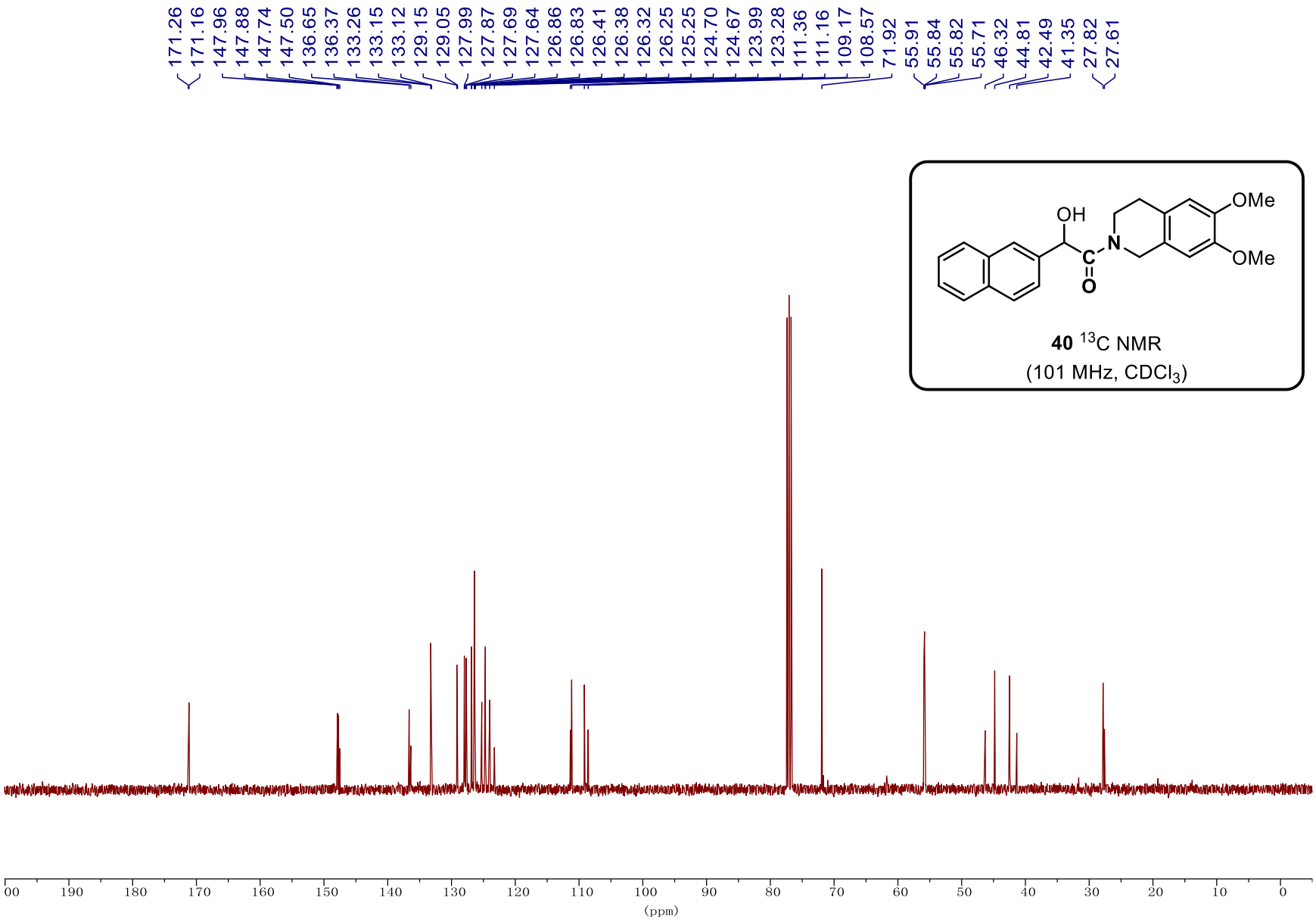

315-3 #744 [modified by Sioc]
SS-1-44
UV_VIS_1
WVL:220 nm
mAU
1,400
1,200
1,000
800
600
400
200
0
-200
min
0.0
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
OH
N
C
O
F
38 HPLC

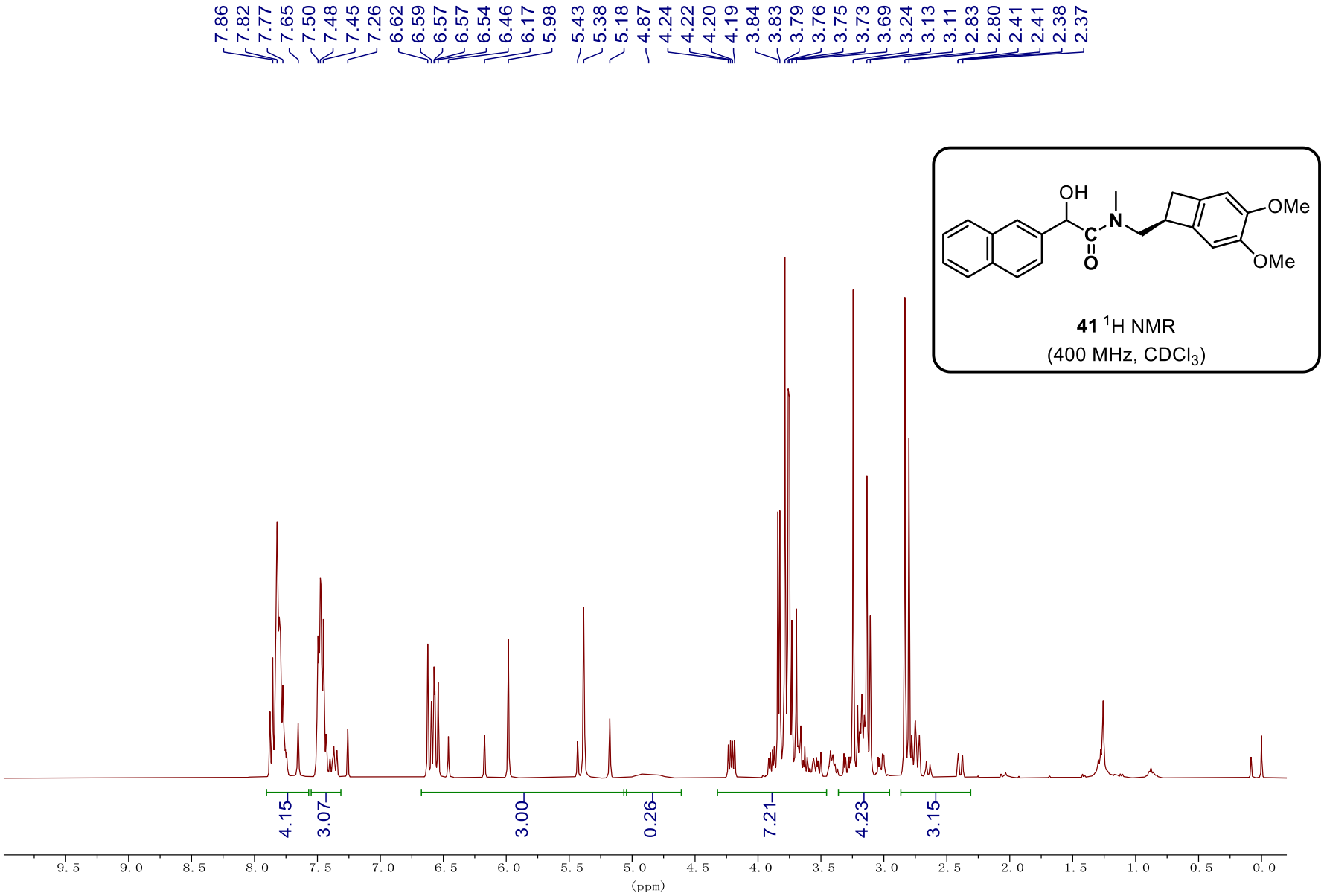

7.89
7.85
7.82
7.49
7.07
7.05
6.86
6.84
6.75
6.72
6.69
6.67
5.47
5.43
4.84
4.81
3.83
3.81
3.77
3.69
3.02
2.99
2.95
2.78
2.75
2.71
2.67
2.64
2.61
2.57
2.52
2.49
1.89
1.78
1.70
1.66
1.64
1.59
1.42
1.39
1.28
0.44
0.43
0.41
0.40
0.38
OH
N
C
O
O
39 1H NMR
(400 MHz, CDCl3)
4.00
2.99
0.92
0.92
2.19
0.46
0.54
0.64
1.12
1.00
1.36
1.65
3.27
4.17
0.55
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

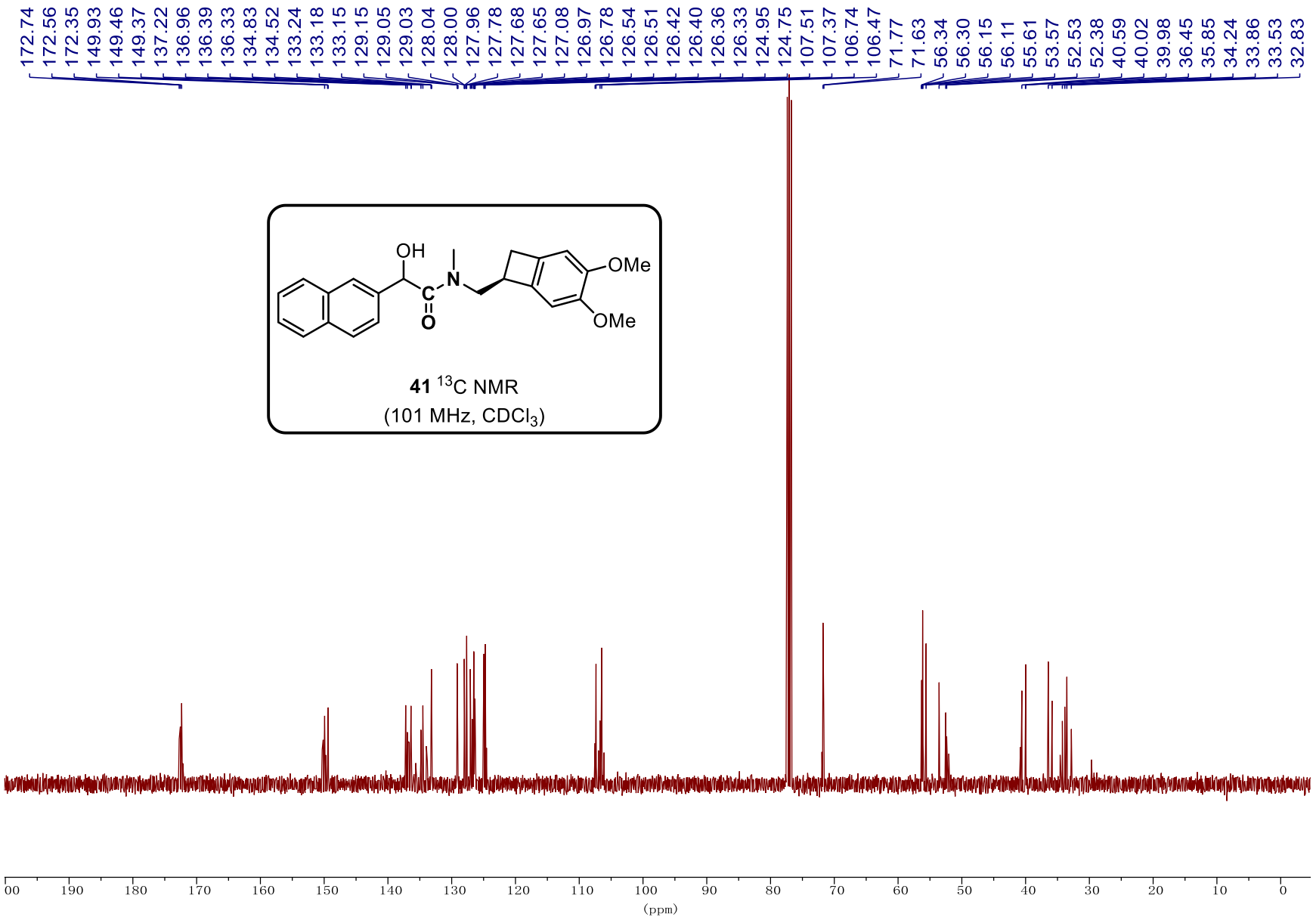

170.73
170.61
158.30
158.12
137.13
136.94
136.88
136.77
133.47
133.40
133.26
129.17
129.15
128.11
128.01
127.84
127.79
127.58
127.37
127.04
126.77
126.54
126.48
124.91
124.86
114.04
113.84
71.69
71.58
55.29
55.22
45.72
45.37
43.91
43.75
41.54
41.34
33.69
33.08
33.01
31.91
OH
N
C
O
O
39 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−1
(ppm)

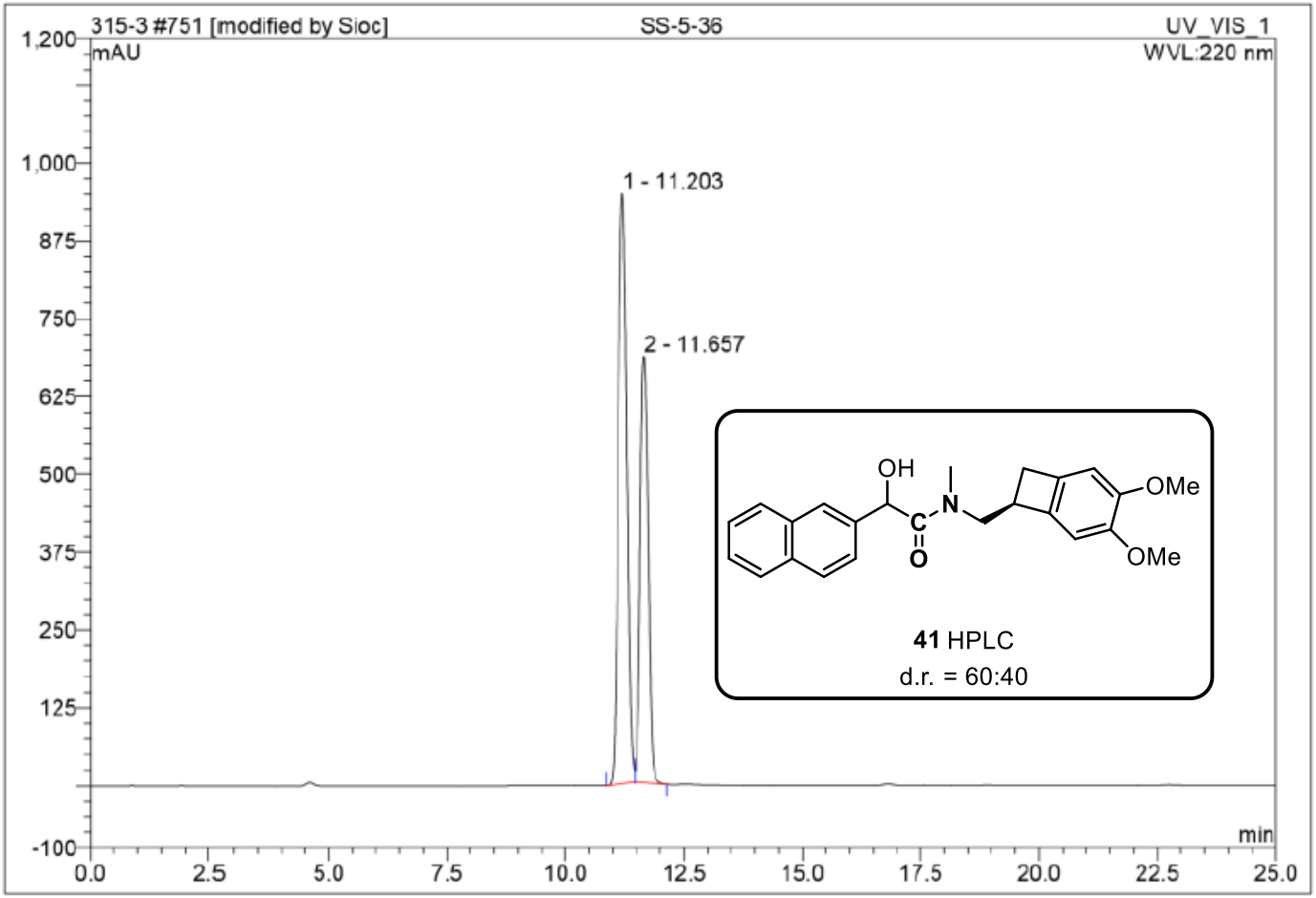

315-3 #737 [modified by Sioc]
SS-1-43-1
UV_VIS_1
WVL:220 nm
mAU
1,600
1,400
1,200
1,000
800
600
400
200
0
-200
min
0.0
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
OH
N
C
O
O
39 HPLC


7.80
7.79
7.47
7.45
7.40
6.57
6.52
6.38
6.11
5.46
5.45
4.87
4.83
4.64
4.60
4.42
4.38
4.08
4.04
3.80
3.77
3.73
3.56
3.47
3.46
3.44
2.78
2.75
2.47
2.46
2.45
2.43
2.42
2.41
2.08
2.04
OH
N
C
O
OMe
OMe
40 1H NMR
(400 MHz, CDCl3)
4.05
3.02
2.06
1.00
2.81
0.86
5.70
1.10
1.24
2.38
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

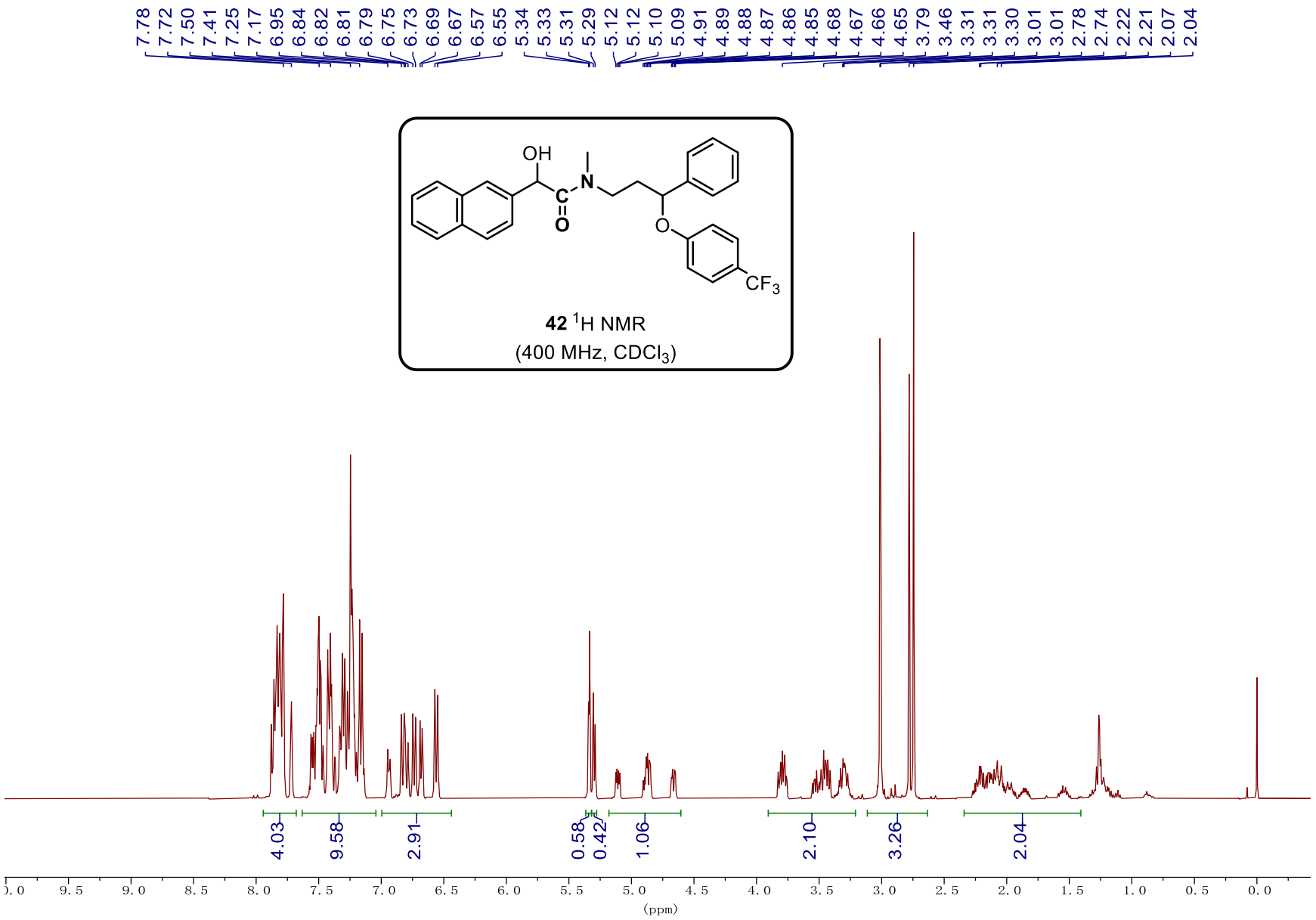

171.26
171.16
147.96
147.88
147.74
147.50
136.65
136.37
133.26
133.15
133.12
129.15
129.05
127.99
127.87
127.69
127.64
126.86
126.83
126.41
126.38
126.32
126.25
125.25
124.70
124.67
123.99
123.28
111.36
111.16
109.17
108.57
71.92
55.91
55.84
55.82
55.71
46.32
44.81
42.49
41.35
27.82
27.61
OH
N
C
O
OMe
OMe
40 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

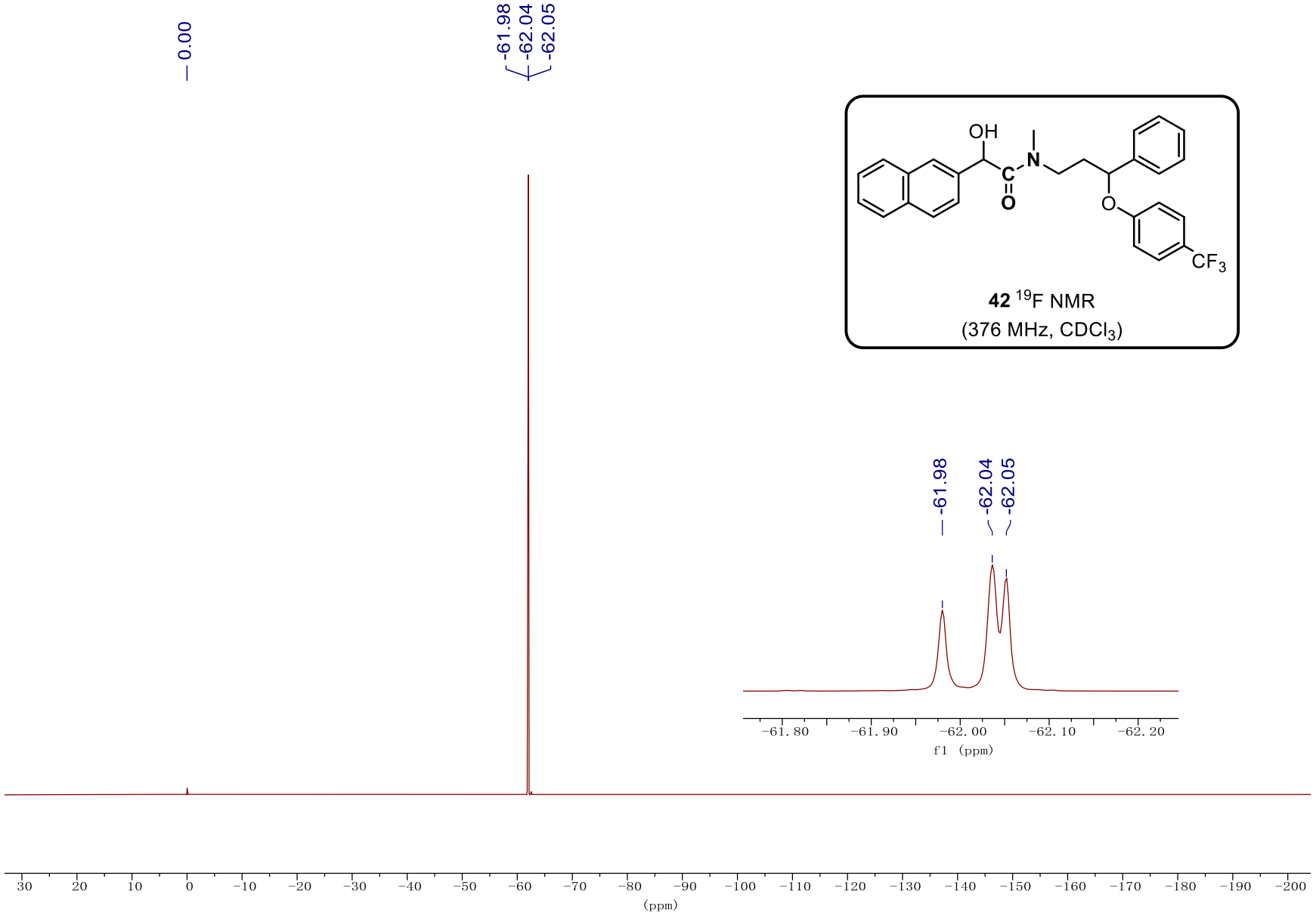

7.86
7.82
7.77
7.65
7.50
7.48
7.45
7.26
6.62
6.59
6.57
6.57
6.54
6.46
6.17
5.98
5.43
5.38
5.18
4.87
4.24
4.22
4.20
4.19
3.84
3.83
3.79
3.76
3.75
3.73
3.69
3.24
3.13
3.11
2.83
2.80
2.41
2.41
2.38
2.37
OH
N
C
O
OMe
OMe
41 1H NMR
(400 MHz, CDCl3)
4.15
3.07
3.00
0.26
7.21
4.23
3.15
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

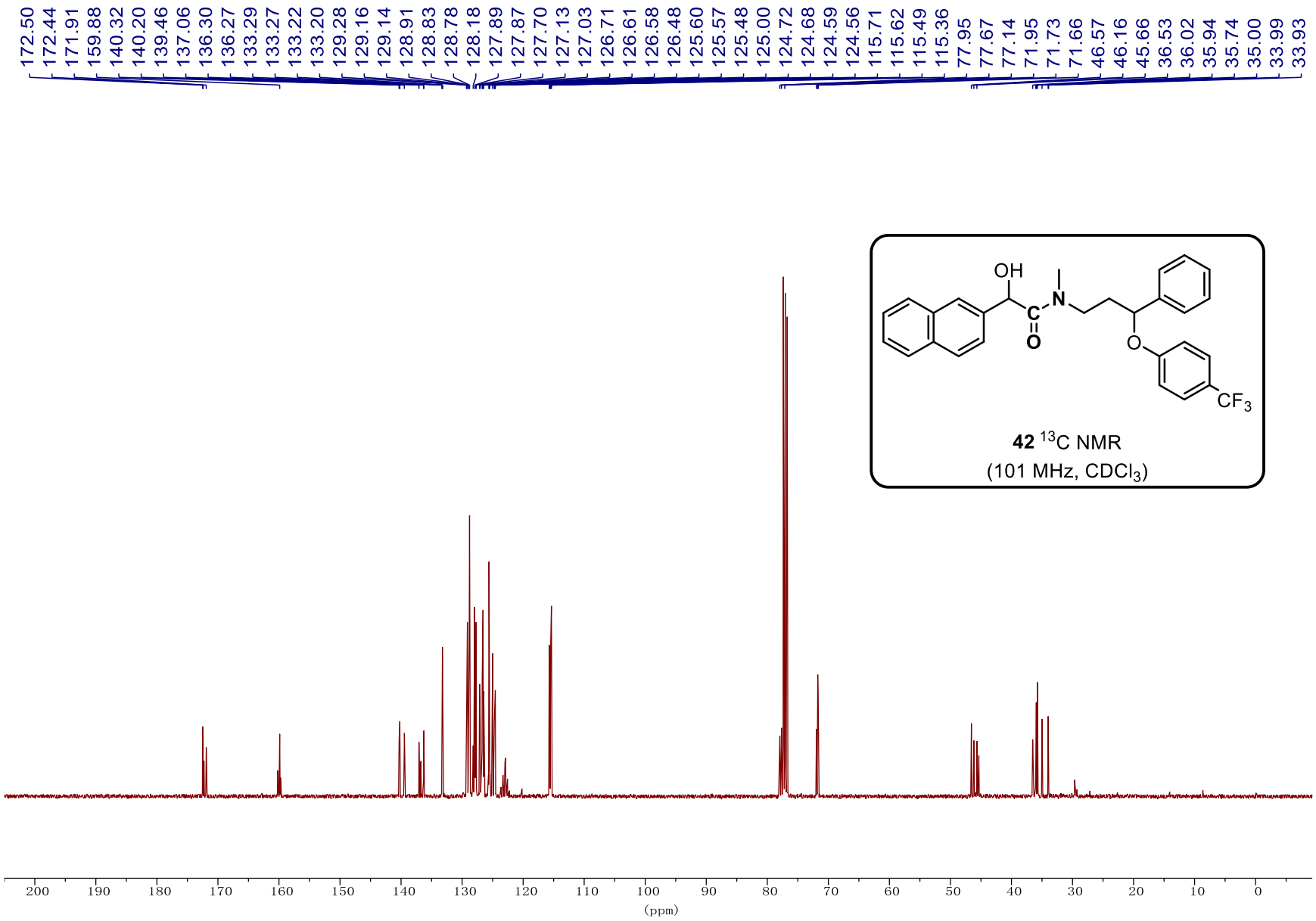

172.74
172.56
172.35
149.93
149.46
149.37
137.22
136.96
136.39
136.33
134.83
134.52
133.24
133.18
133.15
129.15
129.05
129.03
128.04
128.00
127.96
127.78
127.68
127.65
127.08
126.97
126.78
126.54
126.51
126.42
126.40
126.36
126.33
124.95
124.75
107.51
107.37
106.74
106.47
71.77
71.63
56.34
56.30
56.15
56.11
55.61
53.57
52.53
52.38
40.59
40.02
39.98
36.45
35.85
34.24
33.86
33.53
32.83
OH
N
C
O
OMe
OMe
41 13C NMR
(101 MHz, CDCl3)
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

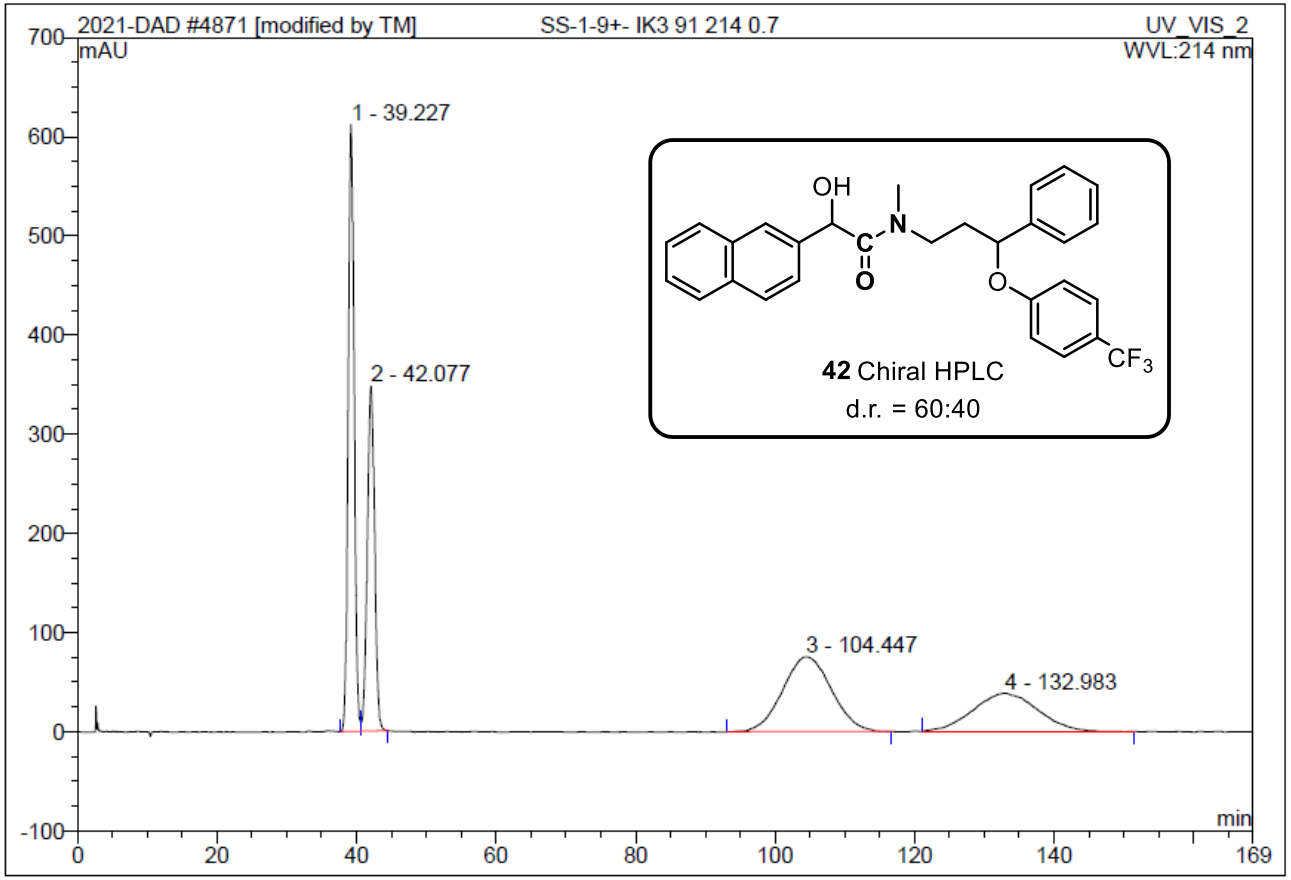


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	11.20	n.a.	947.205	196.962	60.05	n.a.	BM *
2	11.66	n.a.	683.902	131.042	39.95	n.a.	MB*
Total:			1631.107	328.004	100.00	0.000	

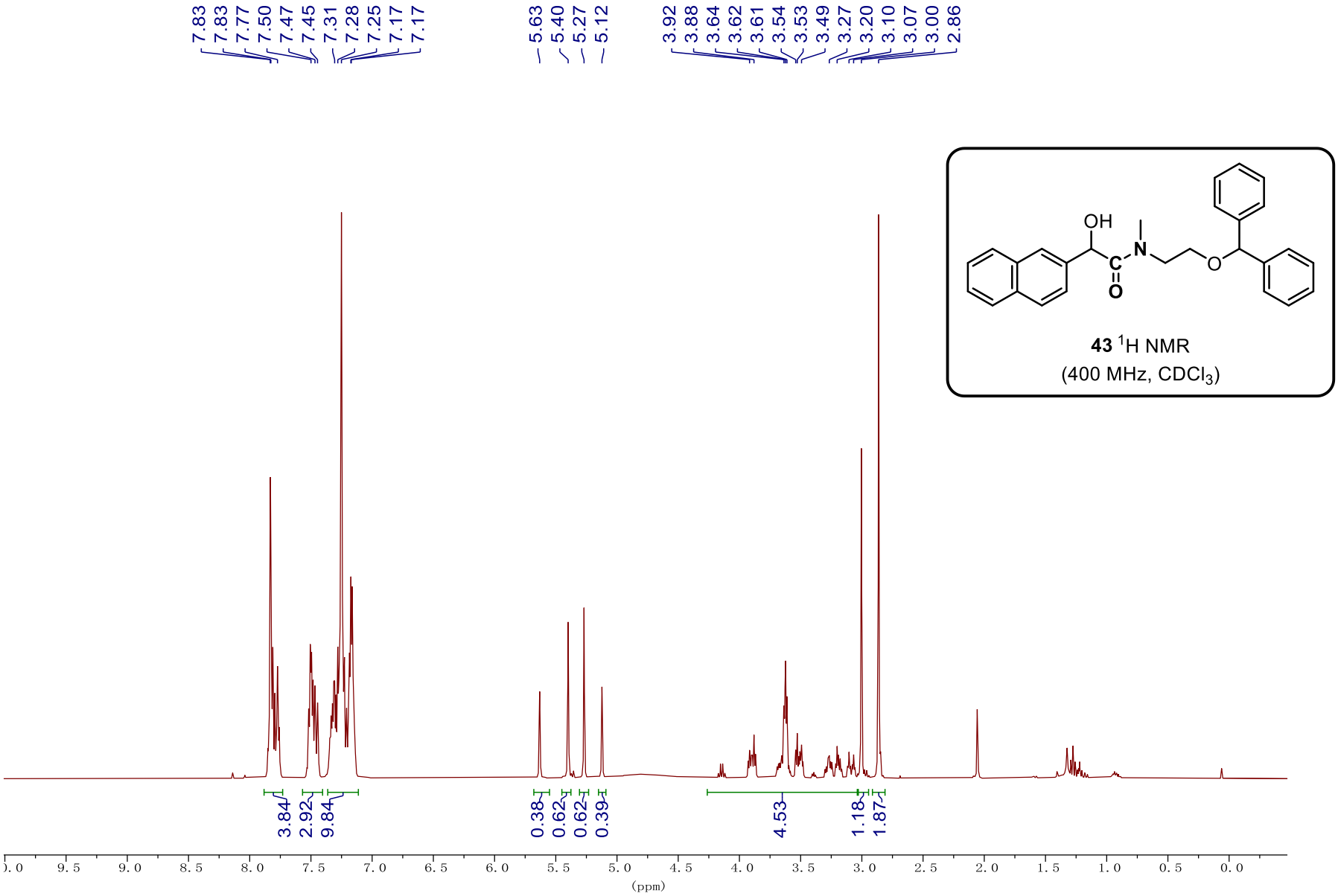

7.78
7.72
7.50
7.41
7.25
7.17
6.95
6.84
6.82
6.81
6.79
6.75
6.73
6.69
6.67
6.57
6.55
5.34
5.33
5.31
5.29
5.12
5.12
5.10
5.09
4.91
4.89
4.88
4.87
4.86
4.85
4.68
4.67
4.66
4.65
3.79
3.46
3.31
3.31
3.30
3.01
3.01
2.78
2.74
2.22
2.21
2.07
2.04
OH
N
C
O
O
CF3
42 1H NMR
(400 MHz, CDCl3)
4.03
9.58
2.91
0.58
0.42
1.06
2.10
3.26
2.04
(ppm)

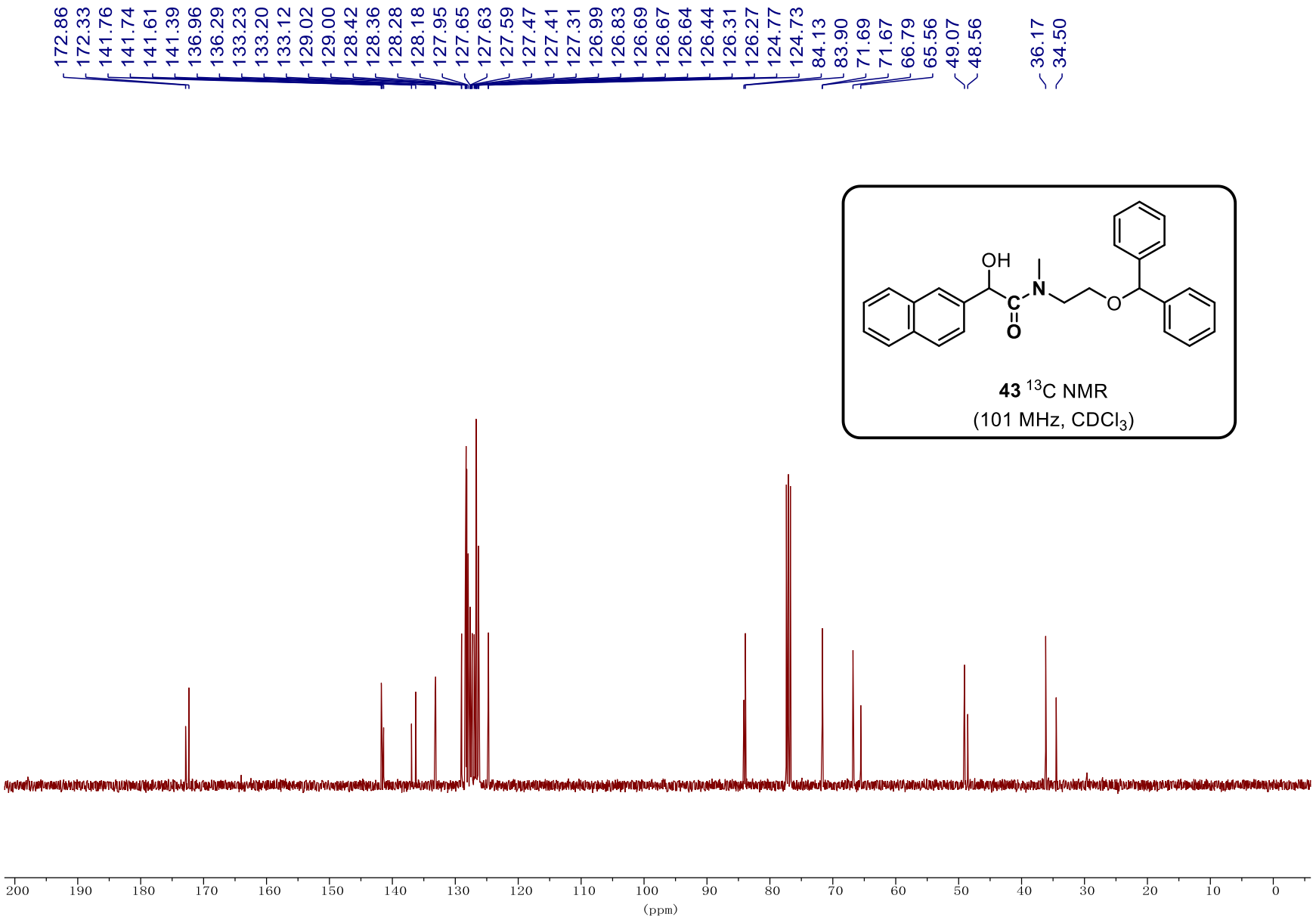

0.00
-61.98
-62.04
-62.05
OH
N
C
O
O
CF3
42 19F NMR
(376 MHz, CDCl3)
-61.98
-62.04
-62.05
-61.80
-61.90
-62.00
-62.10
-62.20
f1 (ppm)
30
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
(ppm)

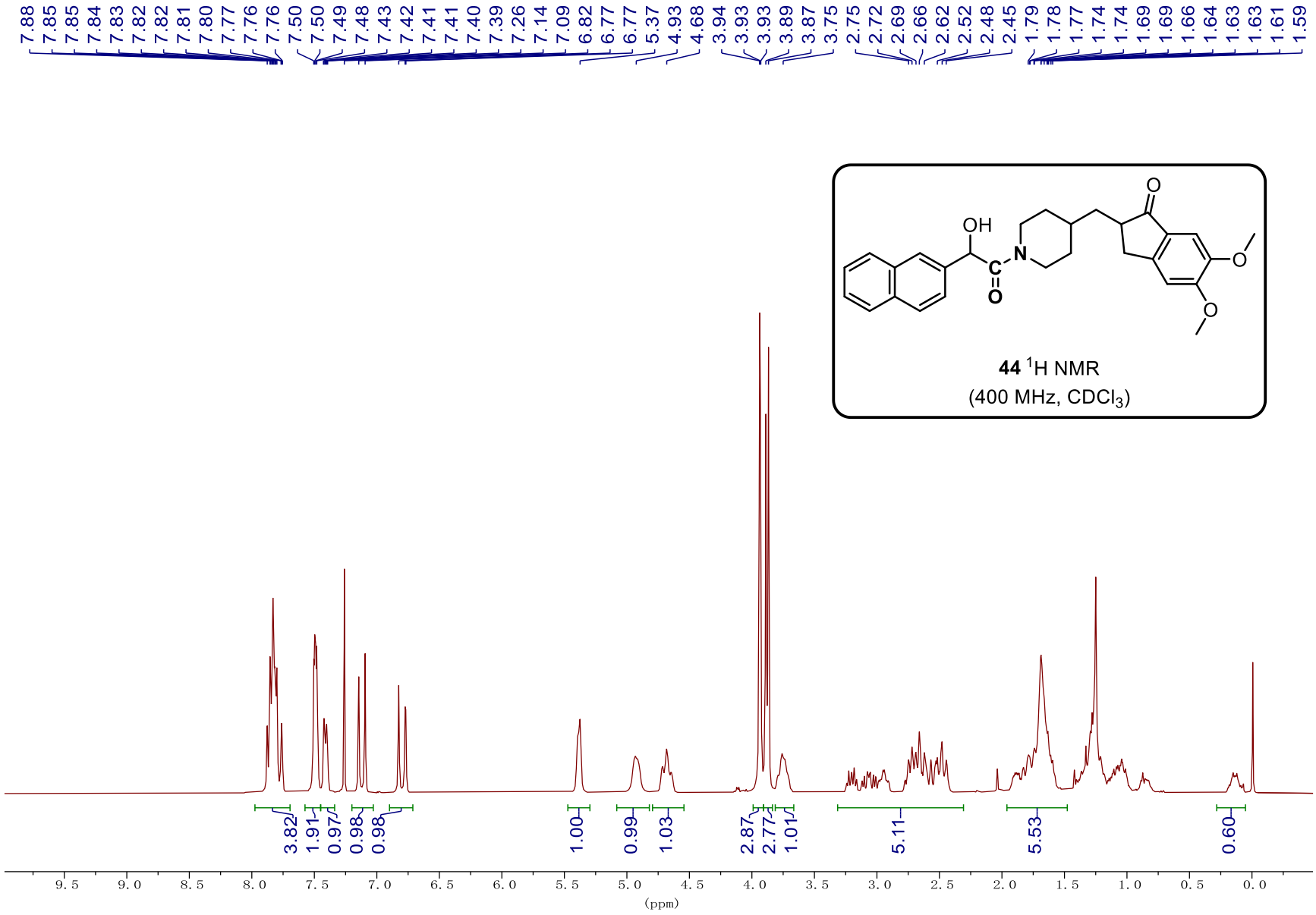

172.50
172.44
171.91
159.88
140.32
140.20
139.46
137.06
136.30
136.27
133.29
133.27
133.22
133.20
129.28
129.16
129.14
128.91
128.83
128.78
128.18
127.89
127.87
127.70
127.13
127.03
126.71
126.61
126.58
126.48
125.60
125.57
125.48
125.00
124.72
124.68
124.59
124.56
115.71
115.62
115.49
115.36
77.95
77.67
77.14
71.95
71.73
71.66
46.57
46.16
45.66
36.53
36.02
35.94
35.74
35.00
33.99
33.93
OH
C
O
N
O
CF3
42 13C NMR
(101 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

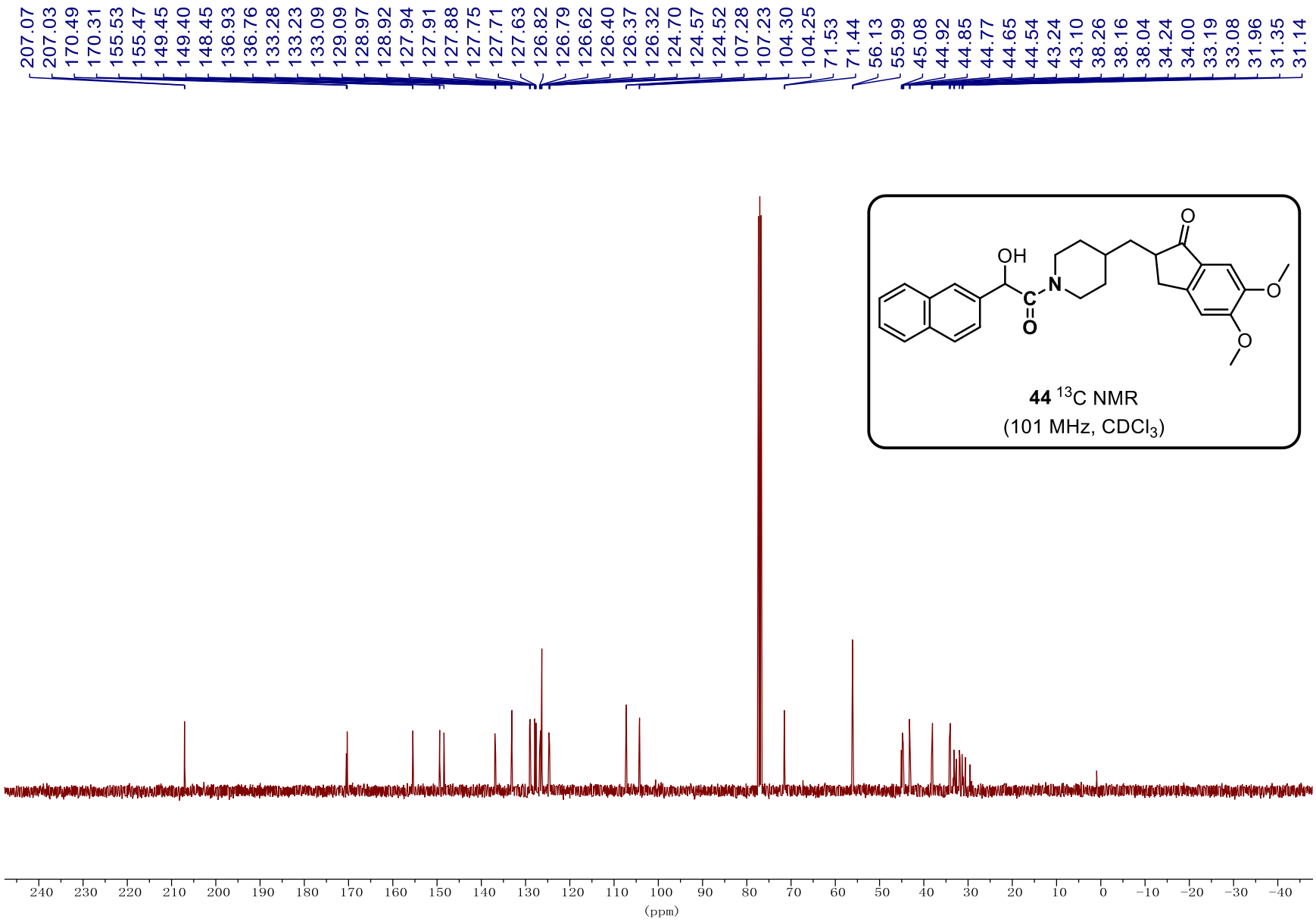


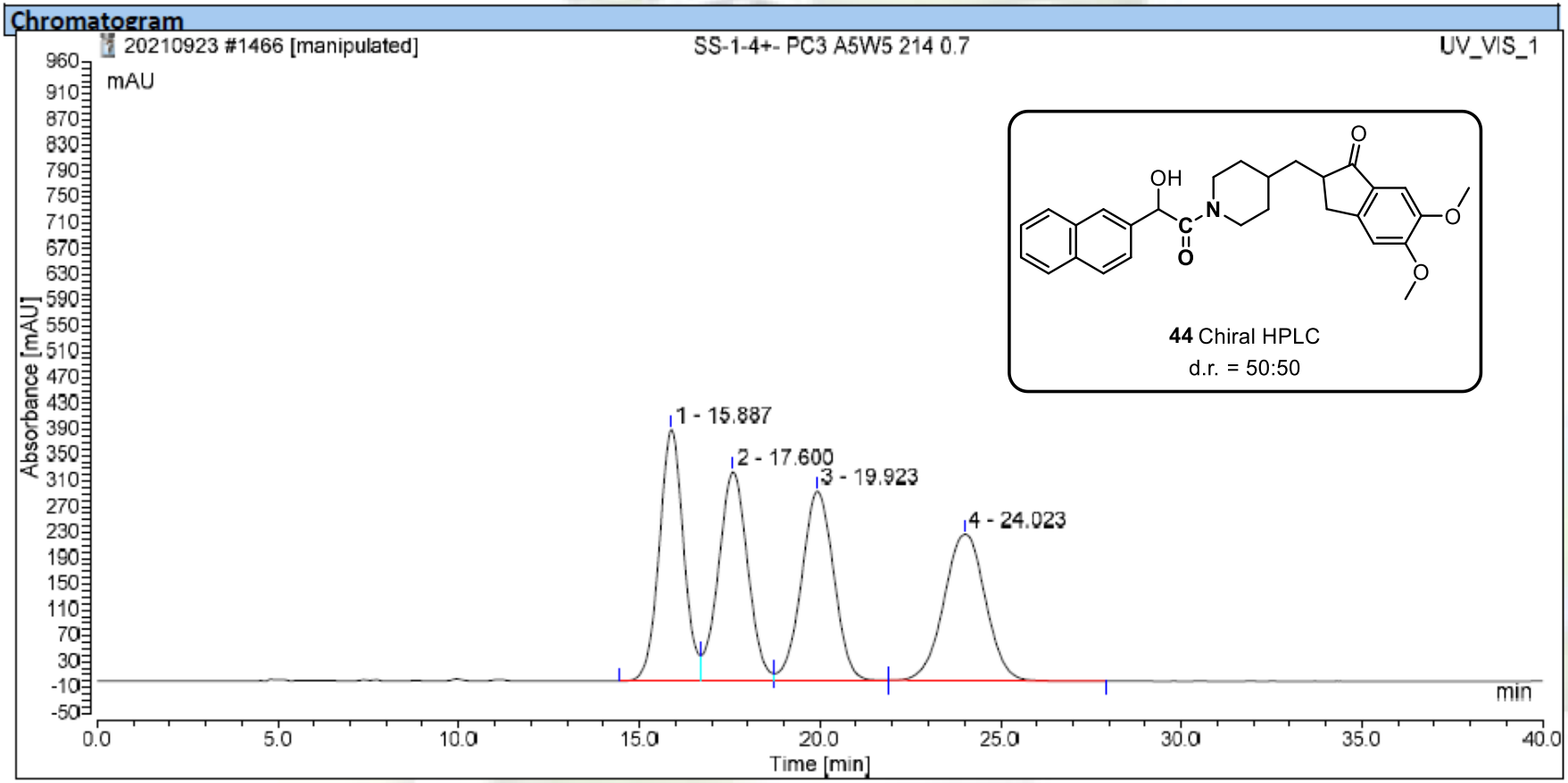
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	39.23	n.a.	612.229	608.551	29.70	n.a.	BM
2	42.08	n.a.	347.693	421.839	20.59	n.a.	MB
3	104.45	n.a.	75.602	611.608	29.85	n.a.	BMB*
4	132.98	n.a.	38.256	406.687	19.85	n.a.	BMB*
Total:			1073.780	2048.685	100.00	0.000	


7.83
7.83
7.77
7.50
7.47
7.45
7.31
7.28
7.25
7.17
7.17
5.63
5.40
5.27
5.12
3.92
3.88
3.64
3.62
3.61
3.54
3.53
3.49
3.27
3.20
3.10
3.07
3.00
2.86
OH
N
C
O
O
43 1H NMR
(400 MHz, CDCl3)
3.84
2.92
9.84
0.38
0.62
0.62
0.39
4.53
1.18
1.87
(ppm)


172.86
172.33
141.76
141.74
141.61
141.39
136.96
136.29
133.23
133.20
133.12
129.02
129.00
128.42
128.36
128.28
128.18
127.95
127.65
127.63
127.59
127.47
127.41
127.31
126.99
126.83
126.69
126.67
126.64
126.44
126.31
126.27
124.77
124.73
84.13
83.90
71.69
71.67
66.79
65.56
49.07
48.56
36.17
34.50
OH
N
C
O
O
43 13C NMR
(101 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

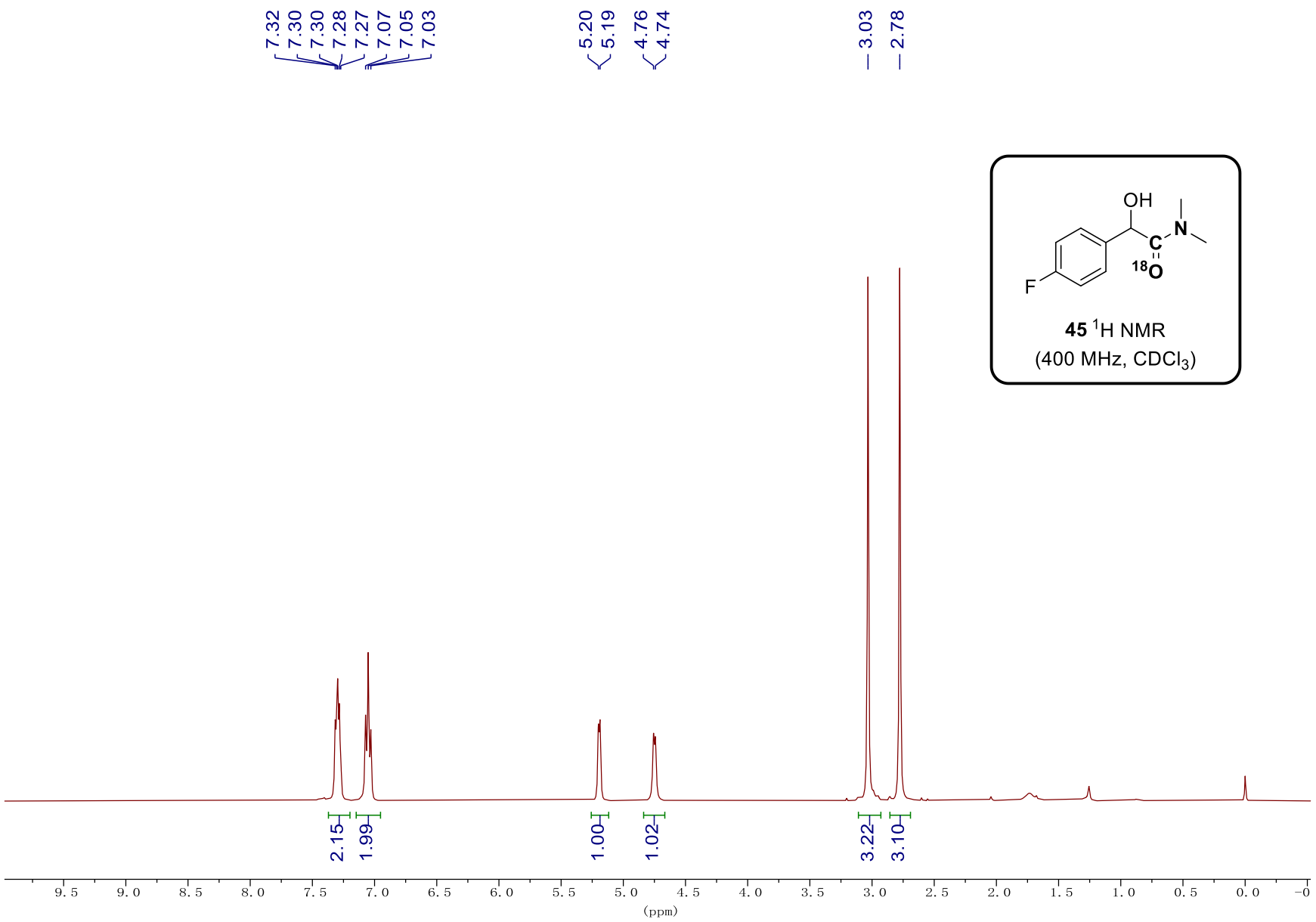

7.88 7.85 7.85 7.84 7.83 7.82 7.82 7.81 7.80 7.77 7.76 7.76 7.50 7.50 7.49 7.48 7.43 7.42 7.41 7.41 7.40 7.39 7.26 7.14 7.09 6.82 6.77 6.77 5.37 4.93 4.68 3.94 3.93 3.93 3.89 3.87 3.75 2.75 2.72 2.69 2.66 2.62 2.52 2.48 2.45 1.79 1.78 1.77 1.74 1.74 1.69 1.69 1.66 1.64 1.63 1.63 1.61 1.59
OH
N
C
O
O
O
O
44 1H NMR
(400 MHz, CDCl3)
3.82 1.91 0.97 0.98 0.98 1.00 0.99 1.03 2.87 2.77 1.01 5.11 5.53 0.60
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

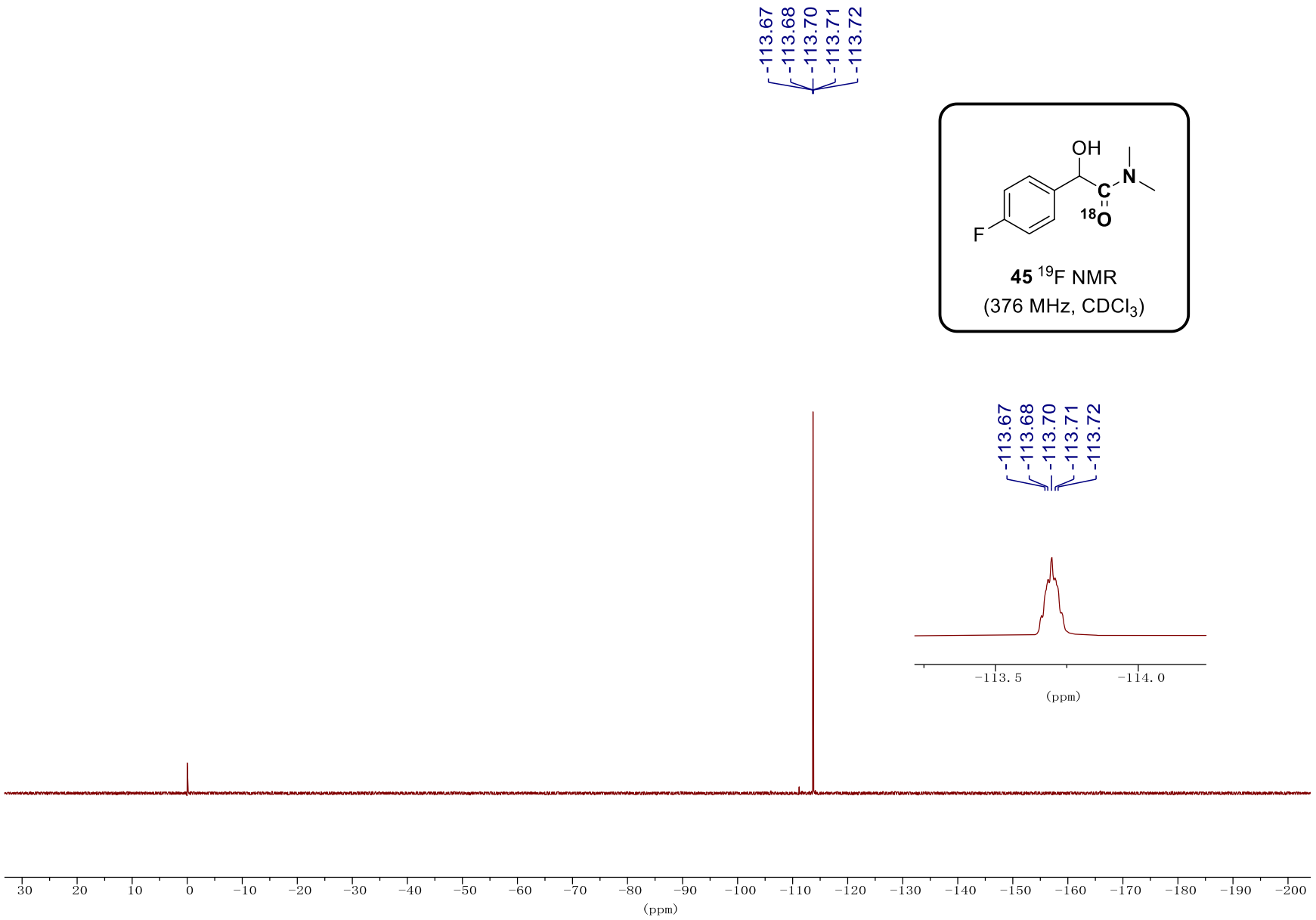

207.07
207.03
170.49
170.31
155.53
155.47
149.45
149.40
148.45
136.93
136.76
133.28
133.23
133.09
129.09
128.97
128.92
127.94
127.91
127.88
127.75
127.71
127.63
126.82
126.79
126.62
126.40
126.37
126.32
124.70
124.57
124.52
107.28
107.23
104.30
104.25
71.53
71.44
56.13
55.99
45.08
44.92
44.85
44.77
44.65
44.54
43.24
43.10
38.26
38.16
38.04
34.24
34.00
33.19
33.08
31.96
31.35
31.14
OH
N
C
O
O
O
O
44 13C NMR
(101 MHz, CDCl3)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
(ppm)

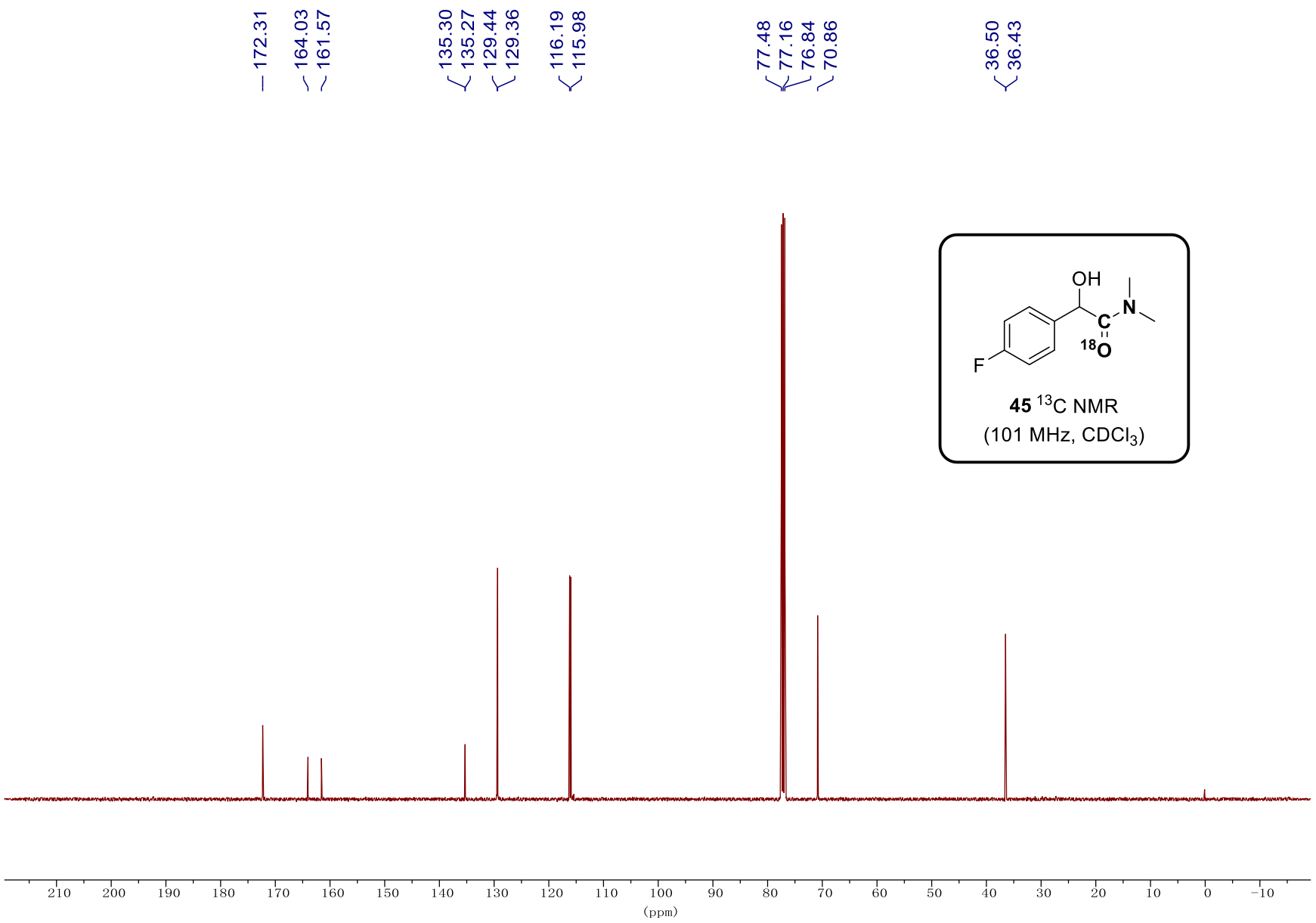


Integration Results

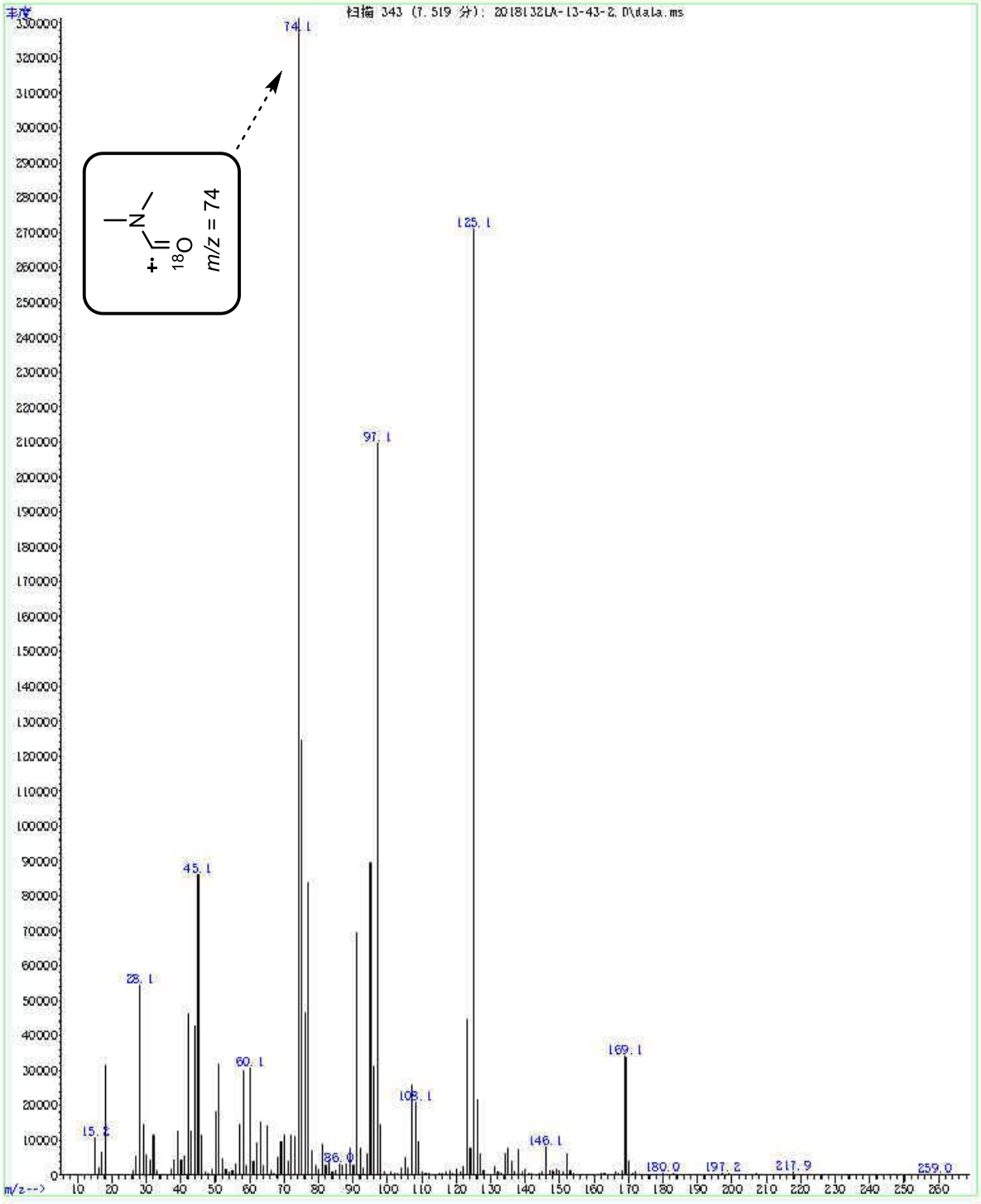
No.	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Resolution (EP)	Asymmetry (EP)	K'	Plates (EP)
n.a.	15.887	302.4990	390.1664	24.901	1.28	n.a.	n.a.	2762
n.a.	17.600	304.9780	324.5777	25.105	1.50	n.a.	n.a.	2268
n.a.	19.923	305.0374	294.7748	25.110	2.20	1.00	n.a.	2373
n.a.	24.023	302.2736	227.0692	24.883	n.a.	0.99	n.a.	2097
Total:		**1214.788**	**1401.998**	**100.000**				

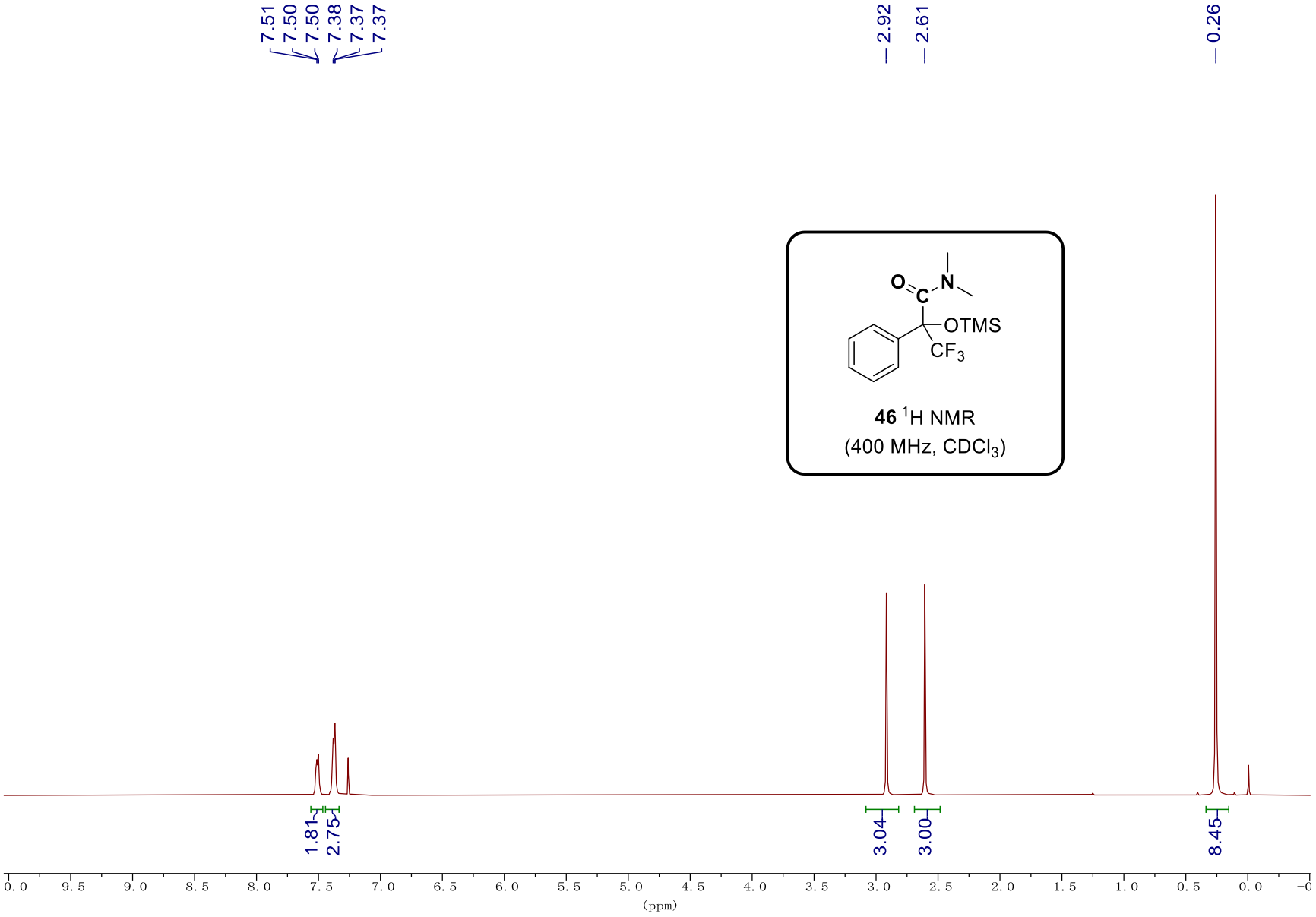

7.32
7.30
7.30
7.28
7.27
7.07
7.05
7.03
5.20
5.19
4.76
4.74
3.03
2.78
OH
N
C
18O
F
45 1H NMR
(400 MHz, CDCl3)
2.15
1.99
1.00
1.02
3.22
3.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

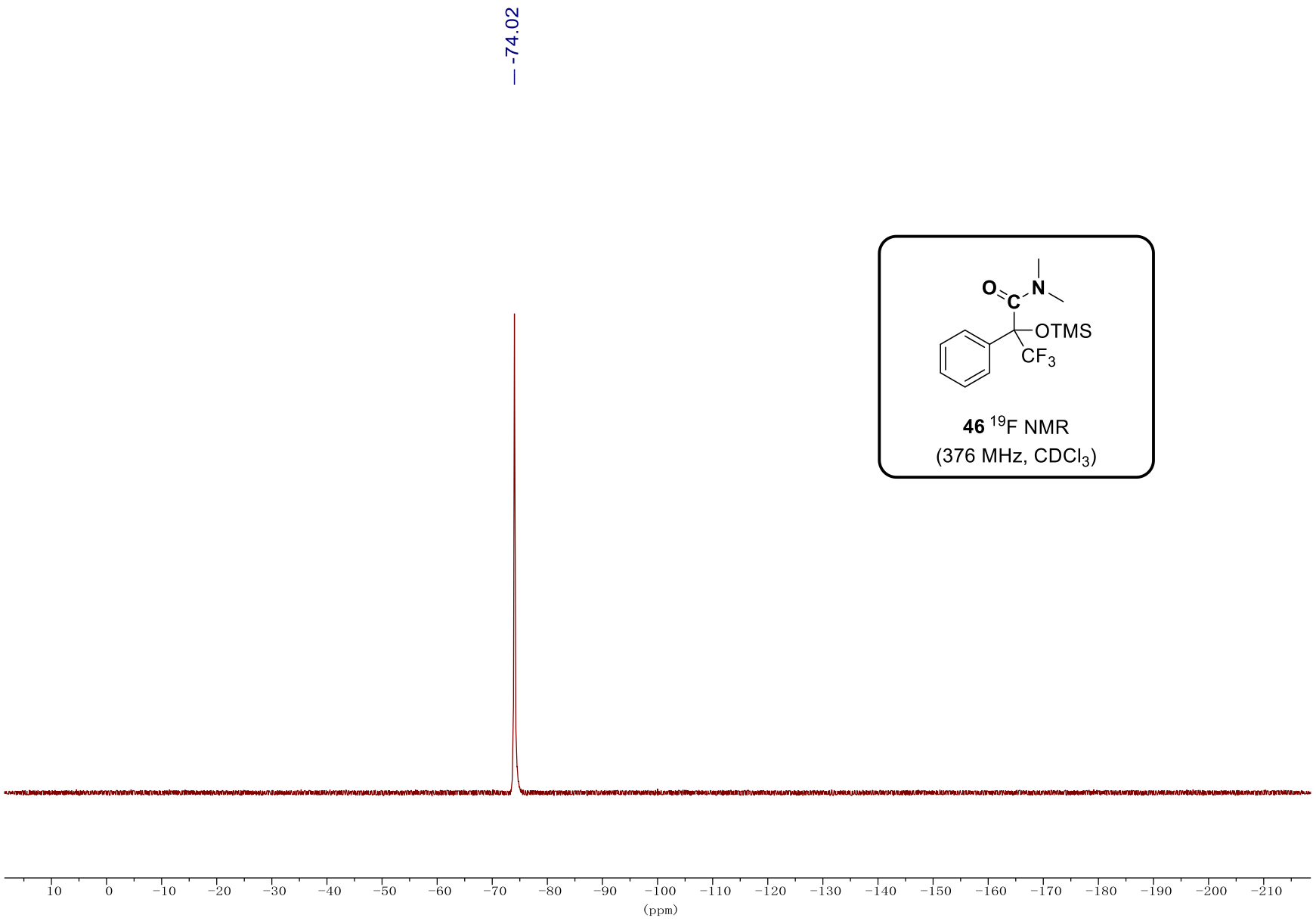

-113.67
-113.68
-113.70
-113.71
-113.72
OH
N
C
18O
F
45 19F NMR
(376 MHz, CDCl3)
-113.67
-113.68
-113.70
-113.71
-113.72
-113.5
-114.0
(ppm)
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
(ppm)

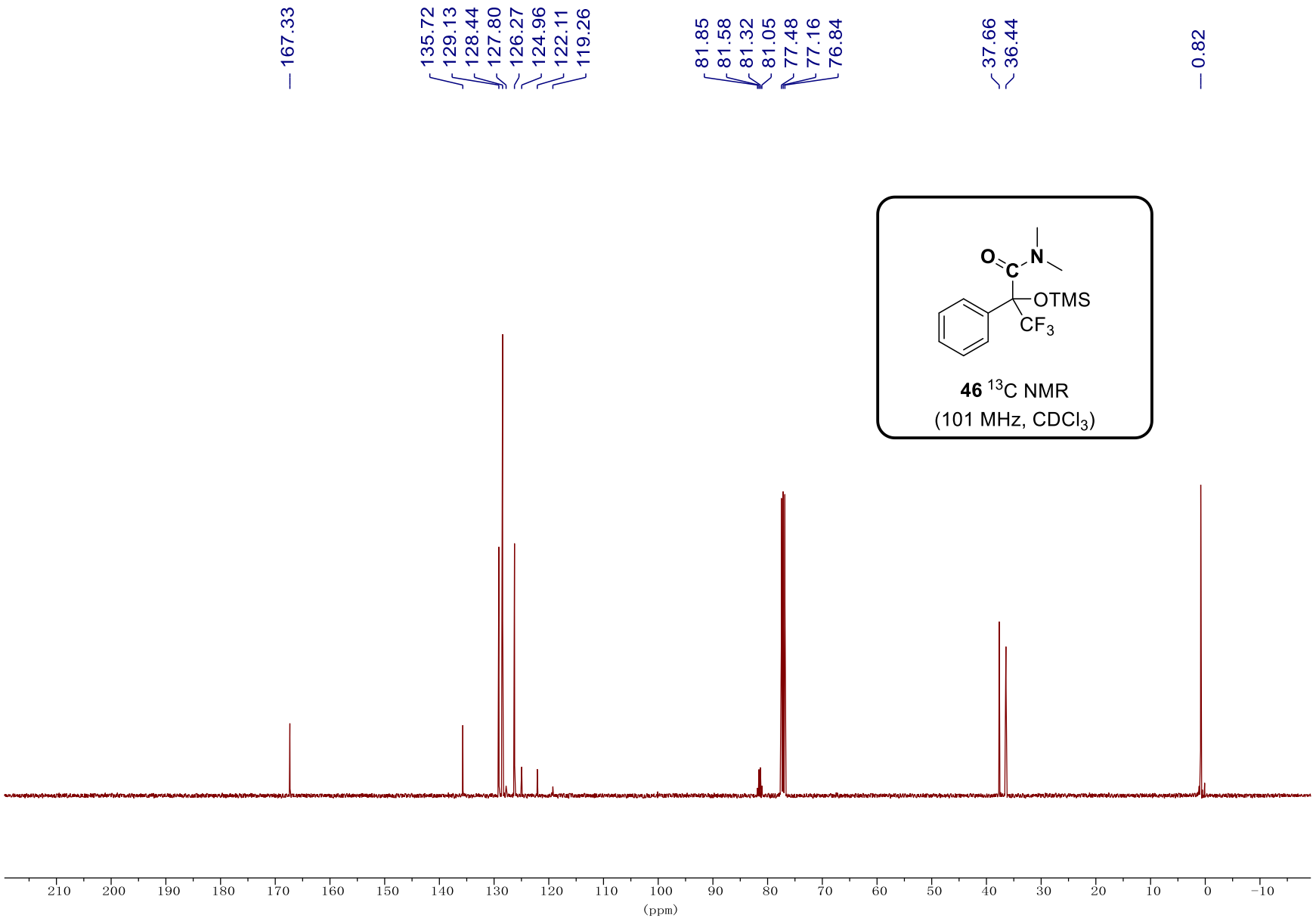

172.31
164.03
161.57
135.30
135.27
129.44
129.36
116.19
115.98
77.48
77.16
76.84
70.86
36.50
36.43
OH
N
C
18O
F
45 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)

文件　　: D:\LA\LA13\20181132LA-13-43-2.D
操作员　　:
已采集　　: 17 Apr 2023　17:04　　, 使用采集方法 LA-QUICKTEST-HIGH.M
仪器:　5975CCMS
样品名称　　:
其他信息　　:
样品瓶号　　: 8




7.51
7.50
7.50
7.38
7.37
7.37
2.92
2.61
0.26
O
C
N
OTMS
CF3
46 1H NMR
(400 MHz, CDCl3)
1.81
2.75
3.04
3.00
8.45
(ppm)


-74.02
O
C
N
OTMS
CF3
46 19F NMR
(376 MHz, CDCl3)
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
(ppm)


167.33
135.72
129.13
128.44
127.80
126.27
124.96
122.11
119.26
81.85
81.58
81.32
81.05
77.48
77.16
76.84
37.66
36.44
0.82
O
C
N
OTMS
CF3
46 13C NMR
(101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
(ppm)