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Electronic Supplementary Information

A Non-Dissociative Open-Flask Hydroboration with Ammonia Borane: Ready Synthesis of Ammonia-Trialkylboranes and Aminodialkylboranes

P. Veeraraghavan Ramachandran,* Michael P. Drolet, and Ameya S. Kulkarni

Department of Chemistry, Purdue University, 560 Oval Drive, West Lafayette, Indiana 47907-2084.

*Email: chandran@purdue.edu

Supporting Information

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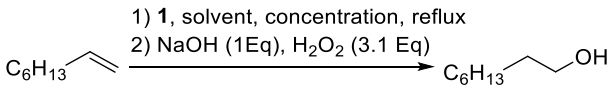
General Considerations

Unless otherwise noted, all reactions were carried out in dry glassware open to air. All solvents were used as received commercially. ^{1}H, ^{13}C, and ^{11}B NMR spectra were recorded at room temperature on a Varian INOVA 300 MHz NMR spectrophotometer. Chemical shifts (δ values) are reported in parts per million (ppm) and are referenced to BF_3-Et_2O for ^{11}B NMR. Data are reported as: δ value, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad) and integration. HRMS data were collected on a FinniganMAT XL95 spectrometer via direct probe injection. All olefins were purchased from commercial sources and were distilled before use. Ammonia borane (AB, **1**) was synthesized via our previously reported procedure.[1] Avoid contact with easily reduced, flammable compounds (e.g. acetone), which may combust upon contact with AB.[2,3]

Optimization of Reaction Conditions with oct-1-ene (Table 3):

The reaction was extremely slow (^{11}B NMR spectroscopy) until the mixture reached reflux (THF). Solvent concentration was targeted first for optimization with the stoichiometry being 3:1 for 1-octene:AB (Table 3, Entries 1-3). Reactions at 2 M concentration in tetrahydrofuran (THF) with respect to AB provided the highest yields for octanol. At 4 M, addition of the olefin resulted in precipitation of AB, generating a heterogeneous reaction mixture. Stoichiometry of oct-1-ene to AB was optimized at 2 M in THF. At 1:1 equivalency (Entry 4), significant amounts of AB degradation products were observed. At 2:1 equivalency (Entry 5), a mixture of aminodioctylborane to trioctylborane-ammonia complex was observed in a ratio of approximately 60:40, which could not be purified. An equivalency of 3:1 cleanly provided a single signal at δ -6 ppm and was chosen for further optimization (Entry 6). AB was seen to be essentially insoluble in dichloromethane, diethyl ether, pentane, or neat in oct-1-ene (Entries 6-9). After extended reaction times and subsequent oxidation, only trace amounts of octan-1-ol were observed. Acetonitrile was found to give similar results to THF without any reaction with the solvent (Entry 10).

Table 3. Optimization of hydroboration of oct-1-ene with ammonia borane.



Entry	Solvent	Concentration (M)	Equiv. Olefin	Time (h)	Yield (%)
1	THF	1	3	1	84
2	THF	2	3	1	89
3	THF	4	3	1	89
4	THF	2	1	5	92
5	THF	2	2	2	88
6	CH_2Cl_2	2	3	19	Trace
7	Et_2O	2	3	19	Trace
8	Pentane	2	3	19	Trace
9	neat	2	3	22	Trace
10	CH_3CN	2	3	2	89

Representative procedure for the synthesis of ammonia-trialkylboranes (**3**) and oxidation to alcohols (**4**).

To a dry 25 mL round bottom flask containing a magnetic stir bar was added 0.154 g ammonia borane (5 mmol, 1 equiv.), 2.5 mL THF, and 2.4 mL 1-octene (15 mmol, 3 equiv.). The flask was fitted with a water-cooled reflux condenser and the reaction mixture rapidly brought to reflux in a 90 ^oC oil bath. The reaction was stirred for 1 h open to air, after which time an aliquot was analyzed by ^{11}B NMR spectroscopy, which showed complete disappearance of the peak due to AB and a new singlet at δ -6 ppm. The reaction mixture was cooled in an ice-water bath and oxidized with the dropwise addition of 1.7 mL 3 M NaOH (5 mmol, 1 equiv), followed by the dropwise addition of 1.7 mL 30% H_2O_2 (15.5 mmol, 3.1 equiv). The reaction contents were allowed to warm to room temperature with continued stirring for 3 h. The reaction mixture was extracted with diethyl ether and the combined organic extracts washed with brine, dried over sodium sulfate, and concentrated *in vacuo* to furnish 1.74 g octanol (**4a**) in 89% yield. The ratio of the 1^o- and 2^o-ols was determined by ^1H NMR as 98:2.

Representative procedure for the synthesis of essentially pure aminodialkylboranes (**5k-5o and 5q**).

Caution: Due to the liberation of flammable hydrogen gas, the reactions were carried out in a well-ventilated hood. Following a similar procedure as above, 1.6 mL (+)-3-carene (**2q**) (10 mmol, 2 equiv), 0.154 g ammonia borane (5 mmol, 1 equiv), and 2.5 mL THF were refluxed under nitrogen for 1 h, after which time the reaction mixture was analyzed by ^{11}B NMR spectroscopy to show a peak at δ 48 ppm. Removal of solvent *in vacuo* yielded 1.486 g of

aminodi-4-isocaranylborane (**5q**) as a slightly turbid, viscous liquid in 97% yield. Oxidation of **5** was carried out as with ammonia-trialkylborane complexes (**3**).

Large scale preparation of aminodicyclohexylborane (**5j**). Purification by distillation.

Following a similar procedure as above, 10.1 mL cyclohexene (**2j**) (100 mmol), 1.54 g ammonia borane (50 mmol), and 25 mL THF were refluxed for 1 h, after which time the reaction mixture was analyzed by ^{11}B NMR spectroscopy to show peaks at δ 48 ppm and δ -6 ppm in a 9:1 ratio. The solvent was removed *in vacuo* and the organic residue distilled under reduced pressure to yield aminodicyclohexylborane (**5j**) as a clear, colorless liquid in 77% yield.

Preparation of aminodiisopinocampheylborane (**5p**). Removal of trialkylborane-ammonia complex.

Following a similar procedure as above, 1.6 mL (+)-α-pinene (**2p**) (10 mmol, 2 equiv), 0.154 g ammonia borane (5 mmol), and 2.5 mL THF were refluxed for 1 h, after which time the reaction mixture was analyzed by ^{11}B NMR to show peaks at δ 48 ppm and δ -6 ppm. The solvent was removed *in vacuo*, and the residue was suspended in 2.5 mL anhydrous pentane, whereupon a white solid formed. The suspension was filtered through a bed of celite and the solid residue washed twice with 2.5 mL pentane. The organic solvent was removed to yield 1.486 g of aminodiisopinocampheylborane (**5p**) as a clear, colorless, viscous liquid in 92% yield.

Characterization data:

trioctylborane-ammonia (**3a**)

Not isolated. Clear, colorless solution in THF. ^{1}H NMR (300 MHz, Tetrahydrofuran-*d8*) δ 3.41 (s, 3H), 1.26 (s, 30H), 1.16 – 0.97 (m, 6H), 0.87 (t, *J* = 6.7 Hz, 9H), 0.25 – -0.02 (m, 6H). ^{13}C NMR (75 MHz, Tetrahydrofuran-*d8*) δ 35.84, 33.34, 31.17, 30.80, 27.56, 23.93, 14.98. **LRMS** (EI) calcd for $C_{24}H_{54}BN$ $[M]^+$: *m/*z, 367, found 367.

octan-1-ol (**4a**)

89% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.64 (t, *J* = 6.6 Hz, 2H), 1.56 (m, 2H), 1.42 – 1.14 (m, 10H), 0.88 (t, *J* = 6.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 63.25, 33.08, 32.10, 29.63, 29.58, 26.05, 22.96, 14.40.[4]

decan-1-ol (**4b**)

90% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.55 (t, *J* = 6.7 Hz, 2H), 2.83 (br s, 1H), 1.49 (m, 2H), 1.23 (m, 14H), 0.84 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 62.96, 33.00, 32.18, 29.91, 29.74, 29.61, 26.07, 22.96, 14.38.[5]

2-methyloctan-1-ol (**4c**)

77% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.46 (ddd, *J* = 29.6, 10.5, 6.2 Hz, 2H), 1.70 – 1.52 (m, 1H), δ 1.48 – 1.17 (m, 9H), 1.16 – 1.02 (m, 1H), 0.89 (dd, *J* = 12.7, 6.8 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 68.66, 36.06, 33.45, 32.17, 29.91, 27.25, 22.98, 16.91, 14.43.[6]

cis-Myrtanol (**4d**)

71% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.62 – 3.46 (m, 2H), 2.37 (dt, *J* = 9.5, 6.3 Hz, 1H), 2.31 – 2.16 (m, 1H), 2.08 – 1.79 (m, 5H), 1.54 – 1.37 (m, 1H), 1.19 (s, 3H), 0.97 (s, 3H), 0.93 (d, *J* = 9.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 67.79, 44.61, 43.13, 41.72, 38.89, 33.45, 28.29, 26.33, 23.67, 19.15.[7]

2-phenylethanol (**4e**) & 1-phenylethanol (minor) (82:18)

83% yield (combined). Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 6.96 (m, 6H), 4.82 (q, *J* = 6.4 Hz, 0.23H), 3.77 (t, *J* = 6.6 Hz, 2H), 2.81 (t, *J* = 6.7 Hz, 2H), 2.44 (br s, 0.23H), 2.00 (br s, 1H), 1.45 (d, *J* = 6.5 Hz, 0.73H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.57, 129.04, 128.54, 126.47, 125.43, 63.72, 39.36, 25.33.[4,8]

2-butoxyethan-1-ol (**4f**)

59% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.71 – 3.65 (t, 2H), 3.53 – 3.33 (m, 4H), 2.54 (br s, 1H), 1.71 – 1.44 (m, 2H), 1.44 – 1.14 (m, 2H), 0.88 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 71.96, 71.05, 70.55, 61.56, 31.70, 26.48, 19.30, 13.94.[9]

5-bromopentan-1-ol (**4g**)

78% yield. Colorless liquid; turned yellow then brown over time. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.62 (t, *J* = 6.2 Hz, 2H), 3.42 (t, *J* = 6.7 Hz, 2H), 2.80 (br s, 1H), 1.98 – 1.77 (m, 2H), 1.68 – 1.37 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 62.38, 33.98, 32.63, 31.83, 24.59.[10,11]

4-(tert-butyldimethylsilyloxy)butan-1-ol (**4h**)

81% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.67 – 3.51 (m, 4H), 3.14 (br s, 1H), 1.68 – 1.51 (m, 4H), 0.85 (d, *J* = 2.5 Hz, 9H), 0.03 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 63.51, 62.73, 30.23, 30.02, 26.14, 18.55, -5.10.[12]

aminodicyclohexylborane (**5j**)

77% yield after distillation *in vacuo*. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.67 (br s, 2H), 1.69 (m, 11H), 1.38 – 1.16 (m, 6H), 1.15 – 0.99 (m, 3H), 0.99 – 0.83 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 29.47, 28.35, 27.58. ^{11}B NMR (96 MHz, $CDCl_3$) δ 47.52. **HRMS** (EI) calcd for $C_{12}H_{24}BN$ $[M]^+$: *m/z*, 193.1996, found 193.1997.

cyclohexanol (**4j**)

67% yield. Pale yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.73 – 3.52 (m, 1H), 1.89 (m, 2H), 1.81 – 1.66 (m, 2H), 1.65 – 1.48 (m, 1H), 1.39 (m, 1H), 1.34 – 1.05 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 70.04, 35.48, 25.60, 24.37.[13]

amino-di-*exo*-norbornylborane (**5k**)

99% yield. Clear, colorless viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.51 (br s, 2H), 2.13 (s, 2H), 2.06 (s, 2H), 1.53 – 1.34 (m, 4H), 1.35 – 1.06 (m, 8H), 1.02 (s, 4H), 0.87 – 0.73 (dt, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 38.72, 38.60, 38.28, 38.20, 37.21, 34.01, 33.94, 33.20, 29.52. ^{11}B NMR (96 MHz, $CDCl_3$) δ 48.04. **HRMS** (EI) calcd for $C_{14}H_{24}BN$ $[M]^+$: *m/z*, 217.1996, found 217.1990.

exo-2-norborneol (**4k**)

81% yield. White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.85 – 3.68 (m, 1H), 2.25 (s, 1H), 2.14 (d, J = 3.8 Hz, 1H), 1.71 – 1.51 (m, 3H), 1.51 – 1.34 (m, 1H), 1.34 – 1.20 (m, 1H), 1.12 (ddd, J = 9.8, 2.5, 1.4 Hz, 1H), 1.07 – 0.96 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.13, 44.56, 42.59, 35.68, 34.66, 28.38, 24.70.[14,15]

aminodiheptylboranes (**5l**)

97% yield as mixture of three potential compounds (3,3’ or 4,4’or 3,4’). Slightly turbid, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.69 (br s, 2H), 1.50 – 1.13 (m, 16H), 1.04 – 0.76 (m, 14H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 34.50, 33.25, 31.82, 31.35, 24.60, 23.67, 23.28, 22.69, 15.04, 14.50, 14.04. ^{11}B NMR (96 MHz, $CDCl_3$) δ 49.25.

heptan-3-ol and heptan-4-ol (1:1 mixture) (**4l**)

87% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.60 (m, 1H), 3.57 – 3.45 (m, 1H), 1.77 (br s, 2H), 1.62 – 1.19 (m, 16H), 0.93 (d, J = 3.8 Hz, 12H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 73.44, 71.57, 39.97, 36.94, 30.44, 28.21, 23.15, 19.21, 14.51, 10.29.[16,17]

aminodioct-4-ylborane (**5m**)

95% yield. Slightly turbid, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.66 (s, 2H), 1.41 – 1.10 (m, 21H), 0.87 (m, 13H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 34.54, 31.89, 31.77, 23.67, 22.69, 15.03, 14.49. ^{11}B NMR (96 MHz, $CDCl_3$) δ 48.95. **LRMS** (APCI) calcd for $C_{16}H_{36}BN$ $[M-H]^-$: *m/z*, 253.3, found 253.3.

octan-4-ol (**4m**)

72% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.68 – 3.51 (m, 1H), 2.55 – 2.31 (br s, 1H), 1.57 – 1.22 (m, 9H), 0.98 – 0.84 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 71.61, 39.82, 37.35, 28.08, 22.99, 19.05, 14.30, 14.25.[18]

aminodi(2-methylpent-3-yl)borane (**5n**)

92% yield. Slightly turbid, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.80 (br s, 2H), 1.85 – 1.56 (m, 2H), 1.53 – 1.15 (m, 4H), 1.00 – 0.76 (m, 20H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 28.60, 28.23, 24.12, 23.37, 21.32, 21.21, 20.51, 20.38, 14.39, 13.81. ^{11}B NMR (96 MHz, $CDCl_3$) δ 56.40 (borinic acid), 48.74 (major), 34.15 (boronic acid), 17.67 (boric acid), 0.26. **LRMS** (APCI) calcd for $C_{12}H_{28}BN$ $[M]^+$: *m/z*, 197.2, found 197.3. **HRMS** (EI) calcd for $C_{12}H_{27}BO$ $[M]^+$: *m/z*, 198.2149, found 198.2150 (hydrolysis product).

2-methylpentan-3-ol (**4n**)

87% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.26 (m, 1H), 2.24 (br s, 1H), 1.67 – 1.52 (m, 1H), 1.57-1.46 (m, 1H), 1.44-1.33 (m, 1H), 0.88 (t, *J* = 7.5 Hz, 3H), 0.84 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 78.23, 33.23, 27.01, 19.08, 17.37, 10.52.[19]

aminodi-2-methylcyclohexylborane (**5o**)

97% yield. Clear, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.65 (br s, 2H), 1.80 – 1.55 (m, 12H), 1.41 – 0.87 (m, 6H), 0.83 (d, *J* = 6.5 Hz, 6H), 0.58 (ddd, *J* = 23.7, 12.2, 2.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 77.66, 77.24, 76.81, 39.23, 37.17, 37.05, 34.85, 34.18, 30.17, 29.76, 28.03, 27.81, 27.44, 23.60, 23.48, 1.42. ^{11}B NMR (96 MHz, $CDCl_3$) δ 48.56.

trans-2-methylcyclohexanol (**4o**)

87% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.17 – 3.05 (dt, 1H), 1.94 (m, 1H), 1.82 – 1.45 (m, 4H), 1.25 (m, 5H), 1.01 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 76.09, 40.11, 35.47, 33.81, 25.78, 25.31, 18.75.[20]

aminodiisopinocampheylborane (**5p**)

92% yield after precipitation of triisopinocampheylborane-ammonia complex. Clear, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.60 (br s, 2H), 2.24 (ddd, *J* = 15.4, 6.2, 2.0 Hz, 2H), 1.98 (ddt, *J* = 8.3, 4.9, 2.1 Hz, 6H), 1.76 – 1.68 (m, 2H), 1.53 (ddd, *J* = 13.1, 8.1, 2.4 Hz, 2H), 1.28 (dt, *J* = 10.8, 8.2 Hz, 2H), 1.12 (s, 6H), 1.03 (s, 6H), 0.90 (d, *J* = 7.1 Hz, 6H), 0.63 (d, *J* = 9.3 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 48.66, 42.10, 39.06, 35.02, 30.89, 28.92, 23.62, 23.12. ^{11}B NMR (96 MHz, $CDCl_3$) δ 47.81, -4.65. **HRMS** (EI) calcd for $C_{20}H_{36}BN$ $[M]^+$: 301.2935, found 301.2937.

(-)-isopinocampheol (**4p**)

98% yield. Pale yellow crystals. ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.07 (dt, *J* = 9.6, 5.0 Hz, 1H), 2.59 – 2.44 (m, 1H), 2.44 – 2.31 (m, 1H), 2.01 – 1.87 (m, 2H), 1.80 (dt, *J* = 5.9, 1.9 Hz, 1H), 1.71 (ddd, *J* = 13.9, 4.7, 2.6 Hz, 1H), 1.57 (s, 1H), 1.28 – 1.18 (m, 3H), 1.12 (t, *J* = 8.0 Hz, 3H), 1.09 – 0.98 (m, 1H), 0.95 (d, *J* = 16.9 Hz, 3H). 13C NMR (75 MHz, $CDCl_3$) δ 71.09, 47.90, 47.32, 41.84, 38.88, 38.27, 34.28, 27.80, 23.81, 20.87.[7]

aminodi-4-isocaranylborane (**5q**)

97% yield. Clear, colorless, viscous liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.51 (br s, 2H), 2.03 – 1.80 (m,2), 1.79 – 1.42 (m, 4H), 1.19 – 0.89 (m, 2H), 0.98 (s, 12H), 0.79 (d, *J* = 6.4 Hz, 6H), 0.87 – 0.57 (m, 4H), 0.50 – 0.15 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 33.46, 30.34, 29.84, 29.70, 22.64, 21.43, 18.27, 17.62, 15.72. ^{11}B NMR (96 MHz, $CDCl_3$) δ 49.45, 0.31. **HRMS** (EI) calcd for $C_{20}H_{36}BN$ $[M]^+$: 301.2935, found 301.2936.

4,7,7-trimethylbicyclo[4.1.0]heptan-3-ol (**4q**)

84% yield. Clear, colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.07 (td, *J* = 9.7, 6.8 Hz, 1H), 2.10 (dd, *J* = 14.0, 6.6 Hz, 1H), 1.97 (m, 1H), 1.75 (s, 1H), 1.57 (m, 1H), 1.30 – 1.13 (m, 1H), 1.05 – 0.97 (m, 1H), 0.97 (s, 3H), 0.92 (d, *J* = 6.4 Hz, 3H), 0.90 (s, 3H), 0.88 – 0.65 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 74.95, 36.85, 30.90, 29.20, 28.81, 22.27, 20.51, 18.19, 18.03, 16.35.[21]

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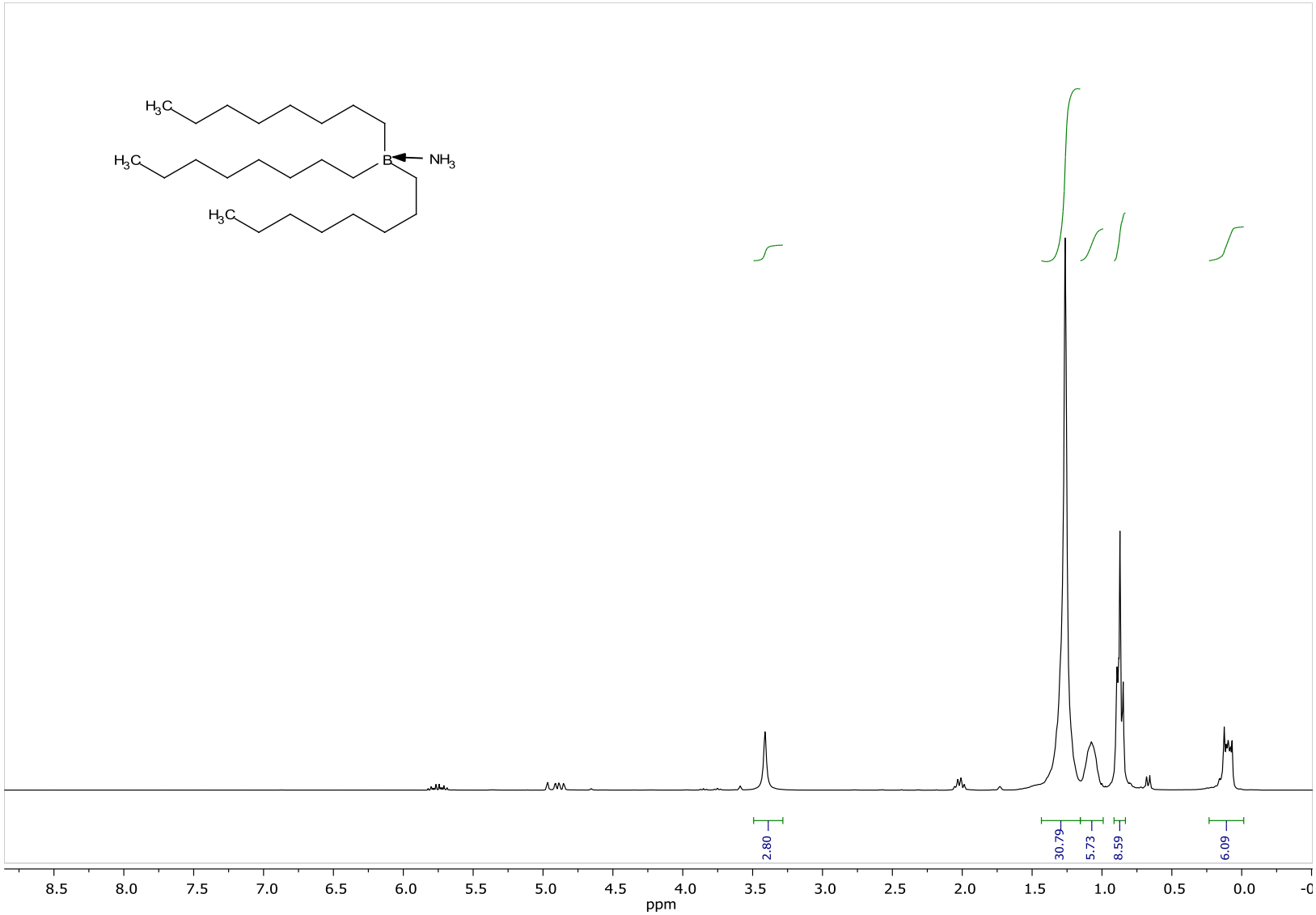
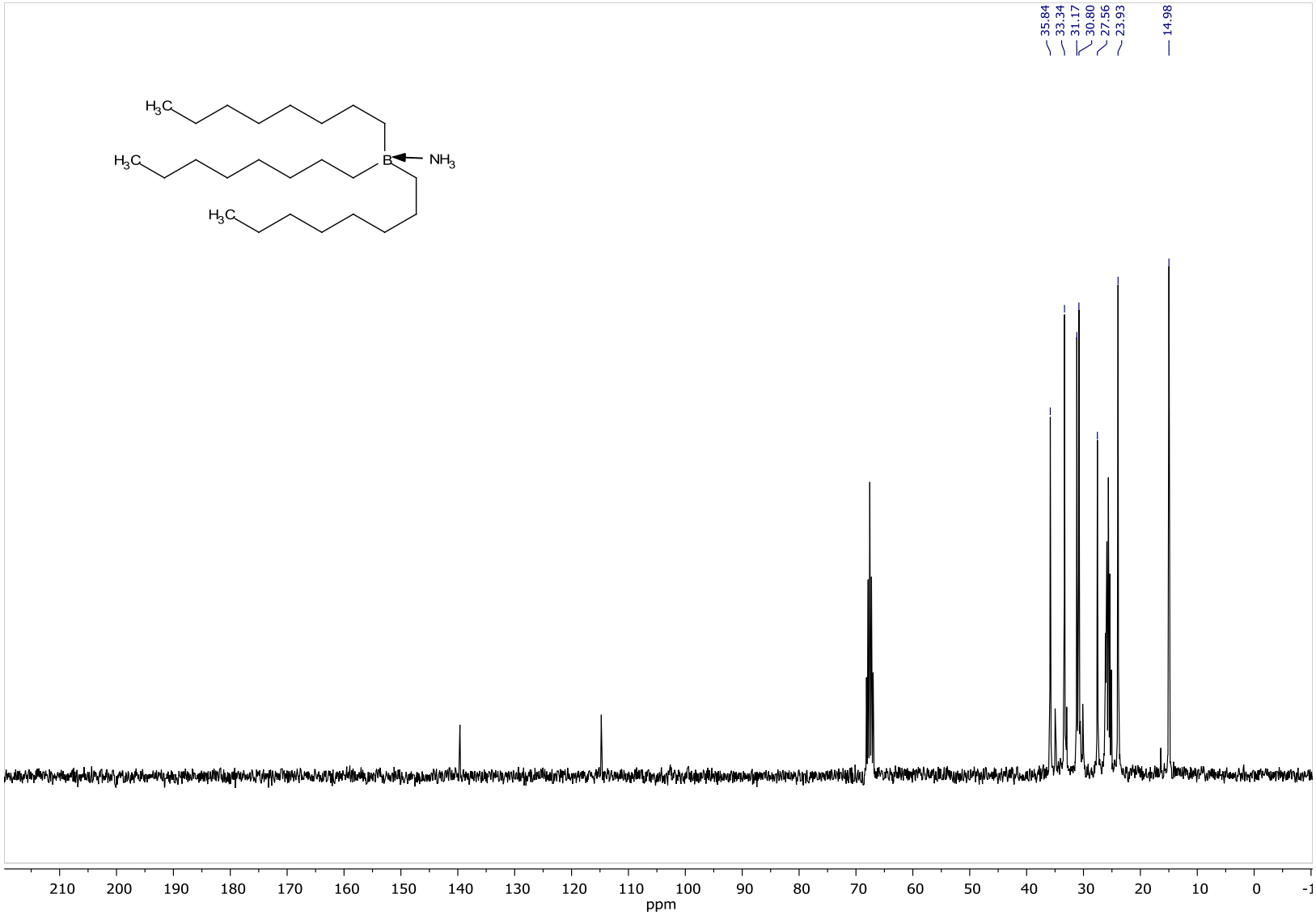
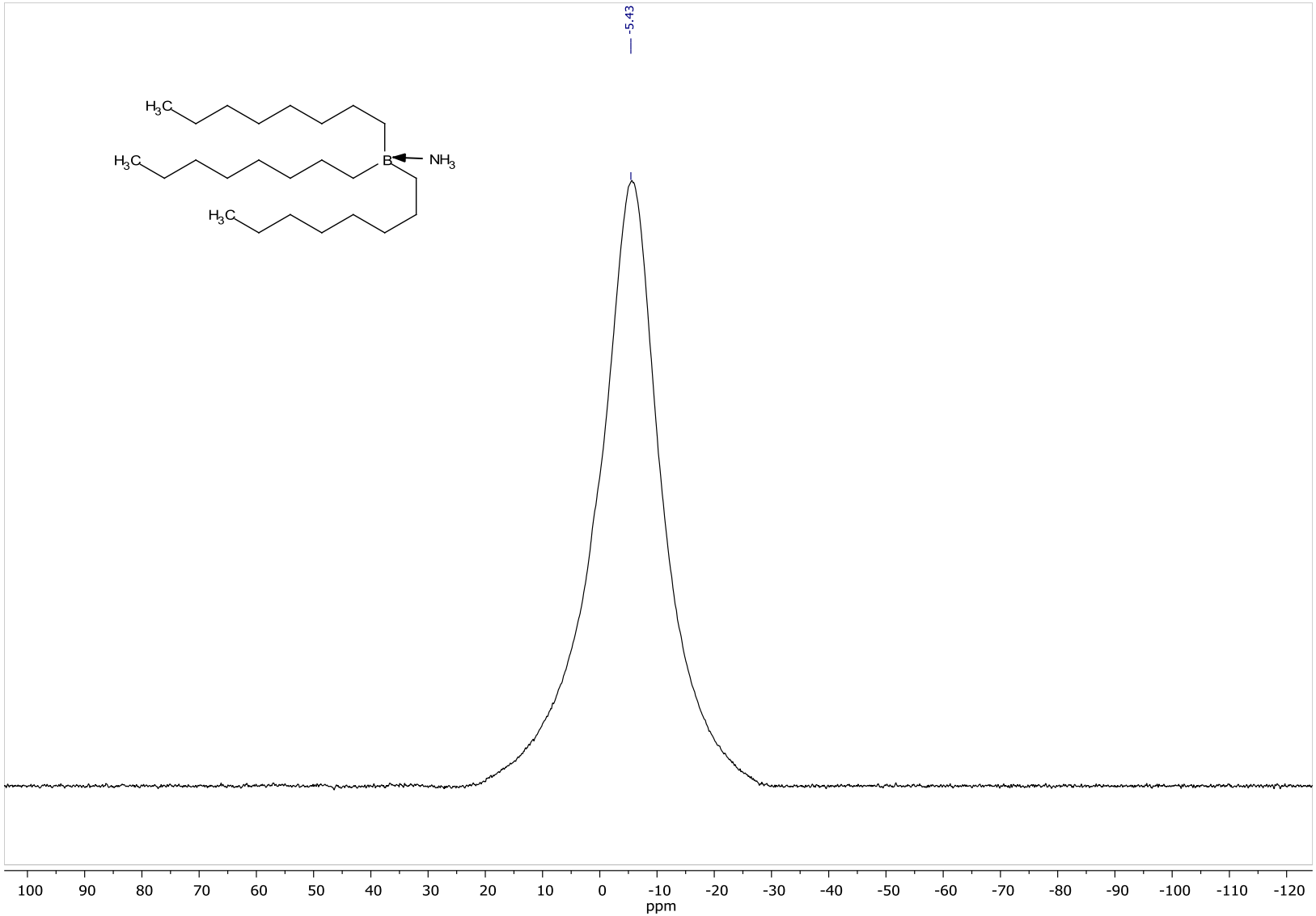


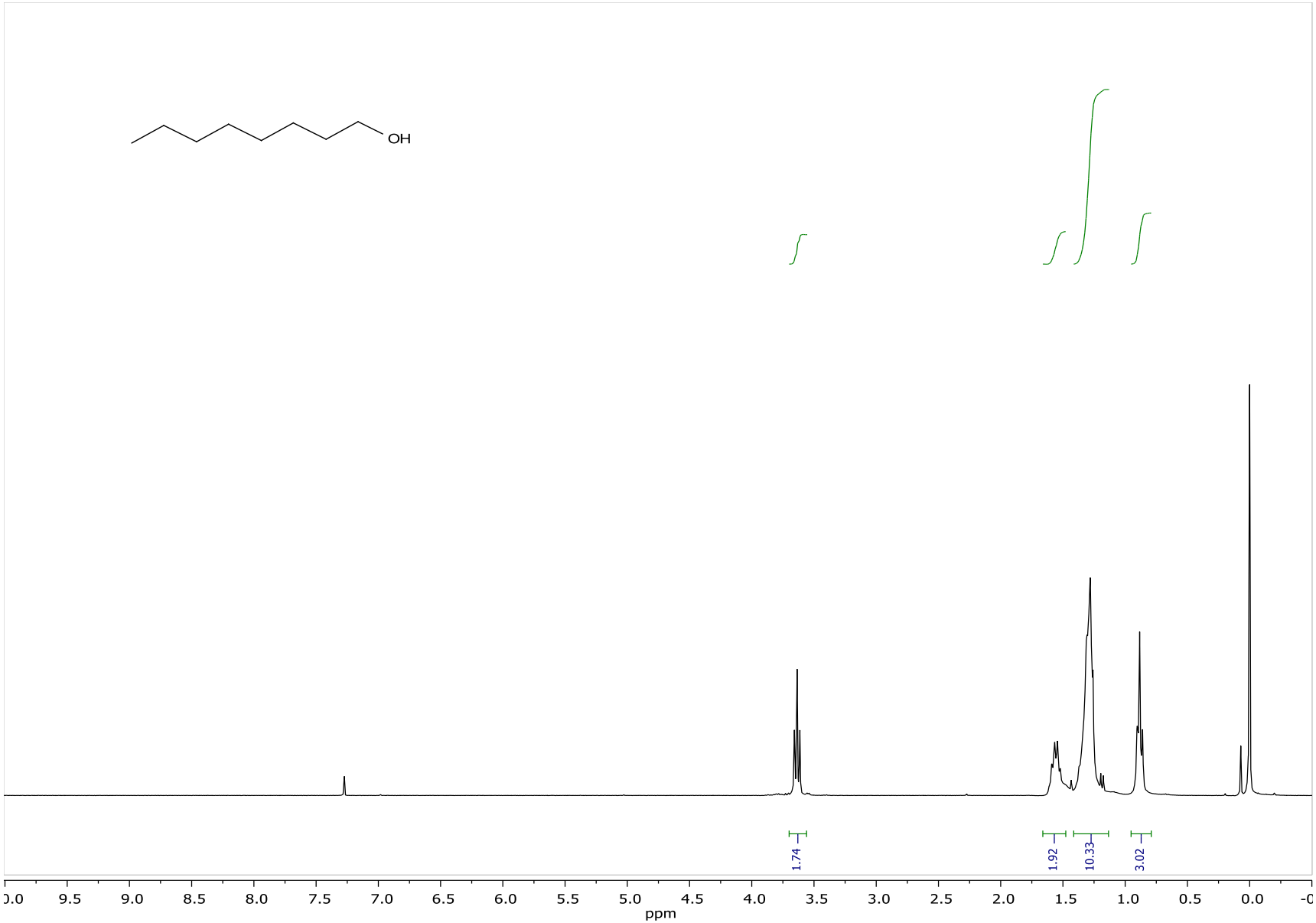
^{1}H NMR of **3a** (300 MHz, THF-*d8*)



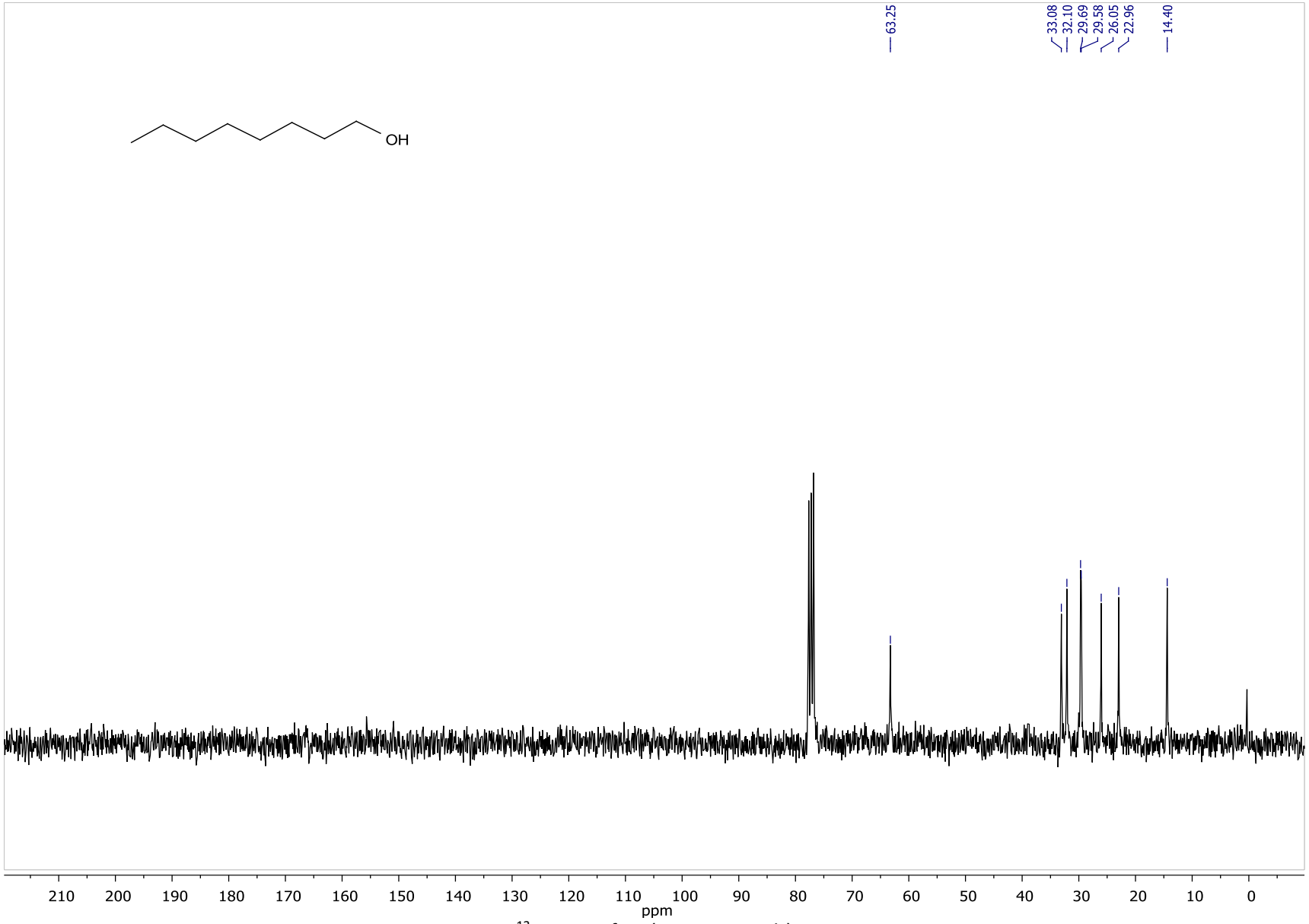
^{13}C NMR of **3a** (300 MHz, THF-*d8*)



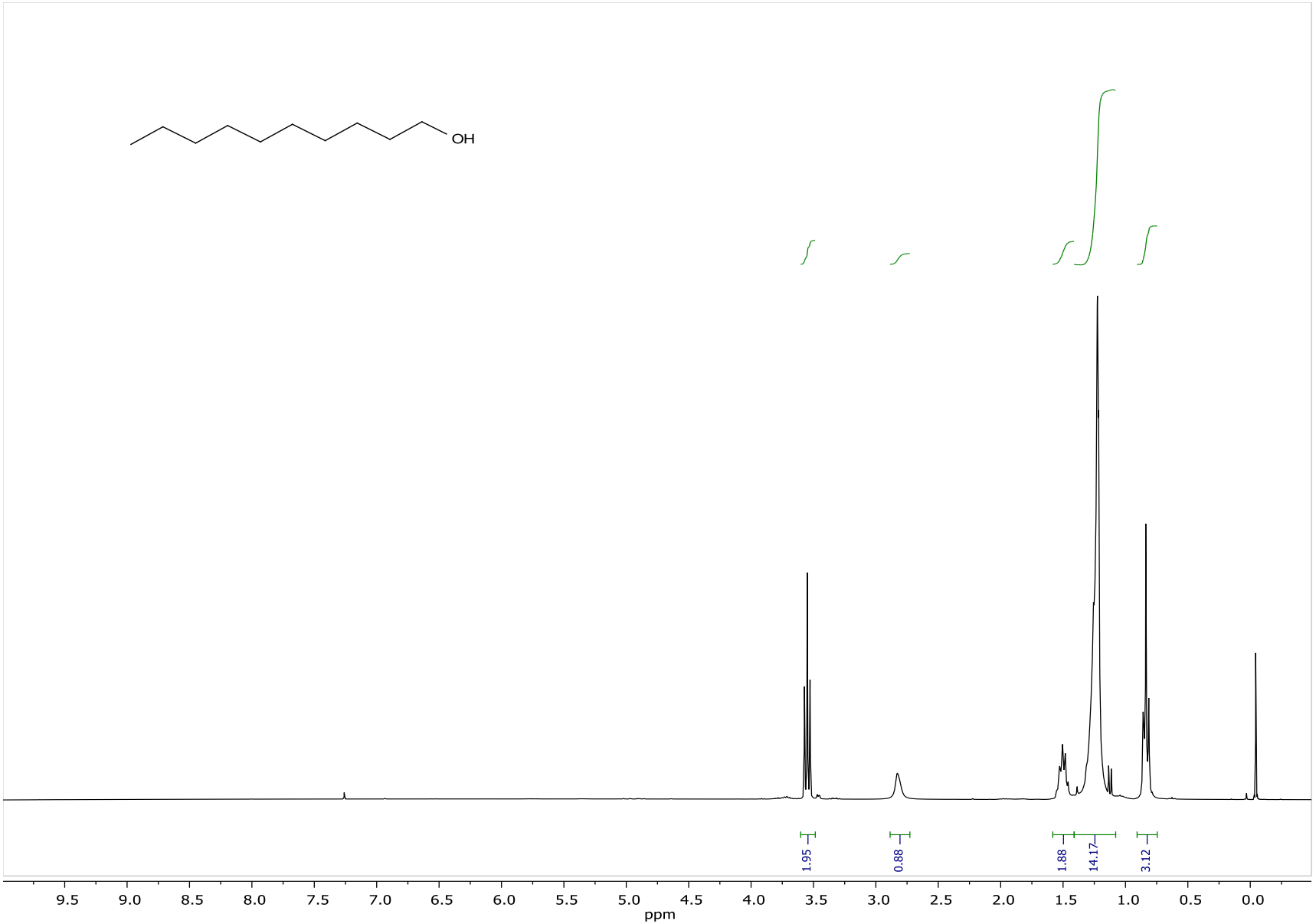
^{11}B NMR of **3a** (300 MHz, THF-*d8*)



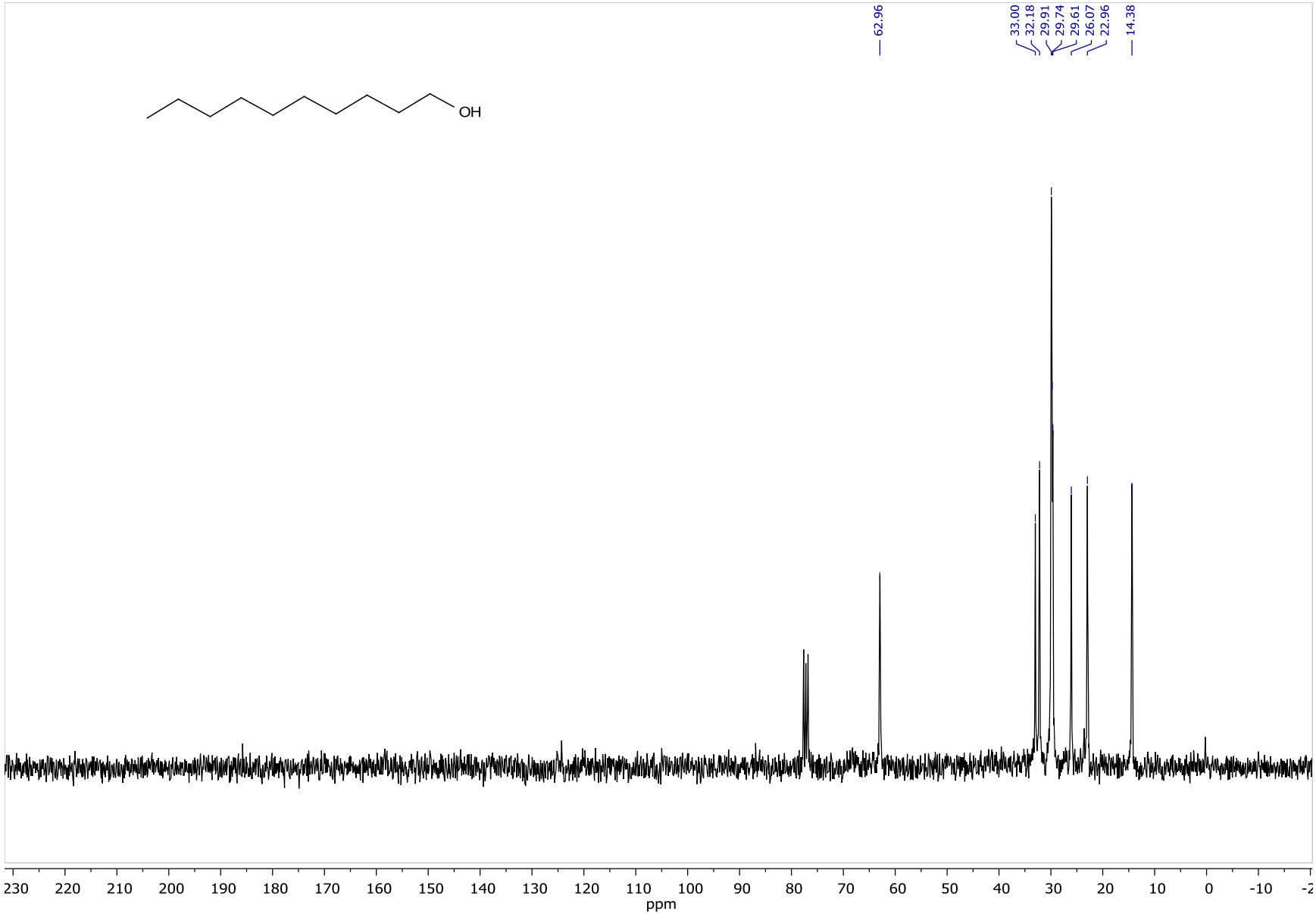
^{1}H NMR of **4a** (300 MHz, $CDCl_3$)



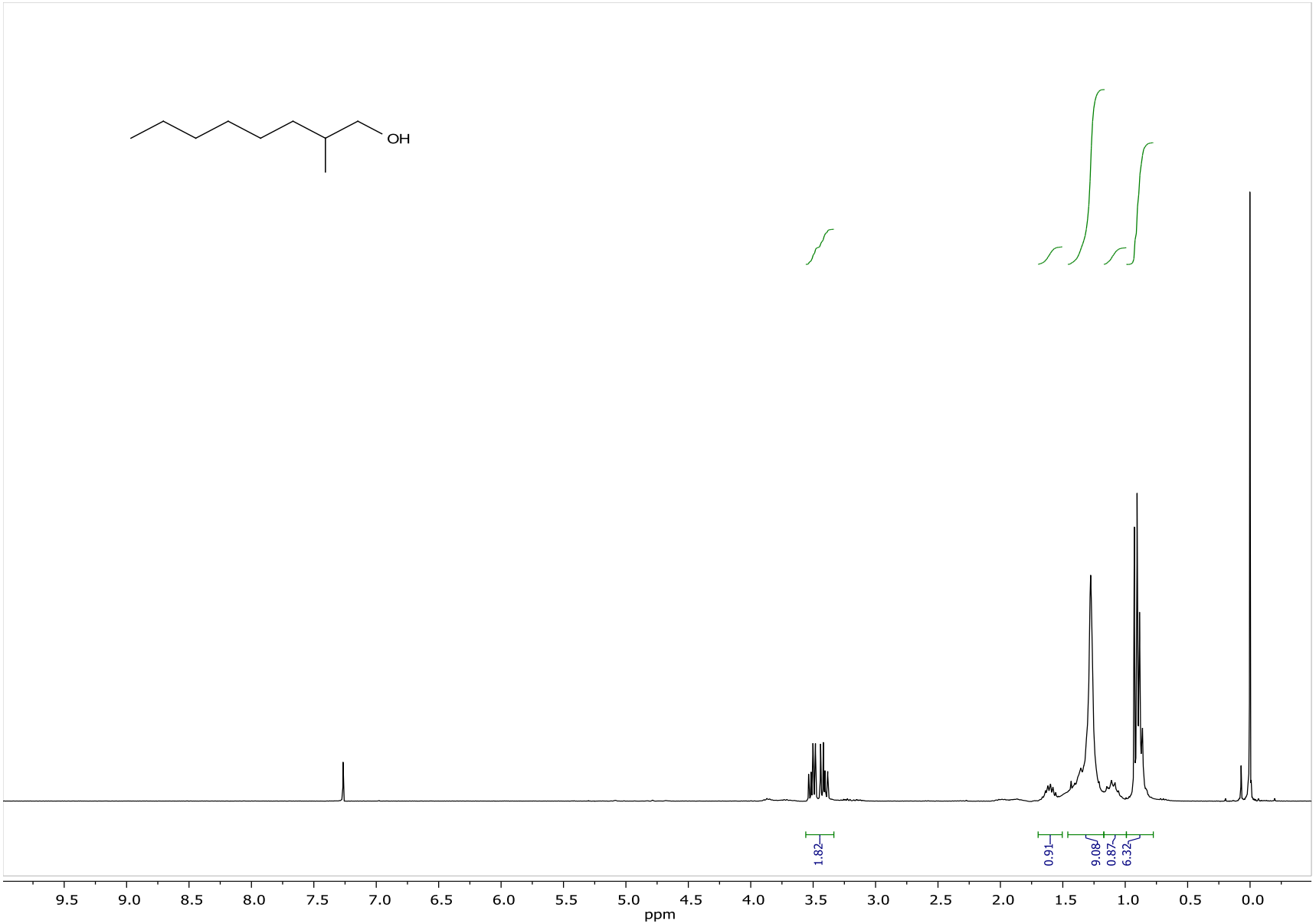
^{13}C NMR of **4a** (75 MHz, $CDCl_3$)



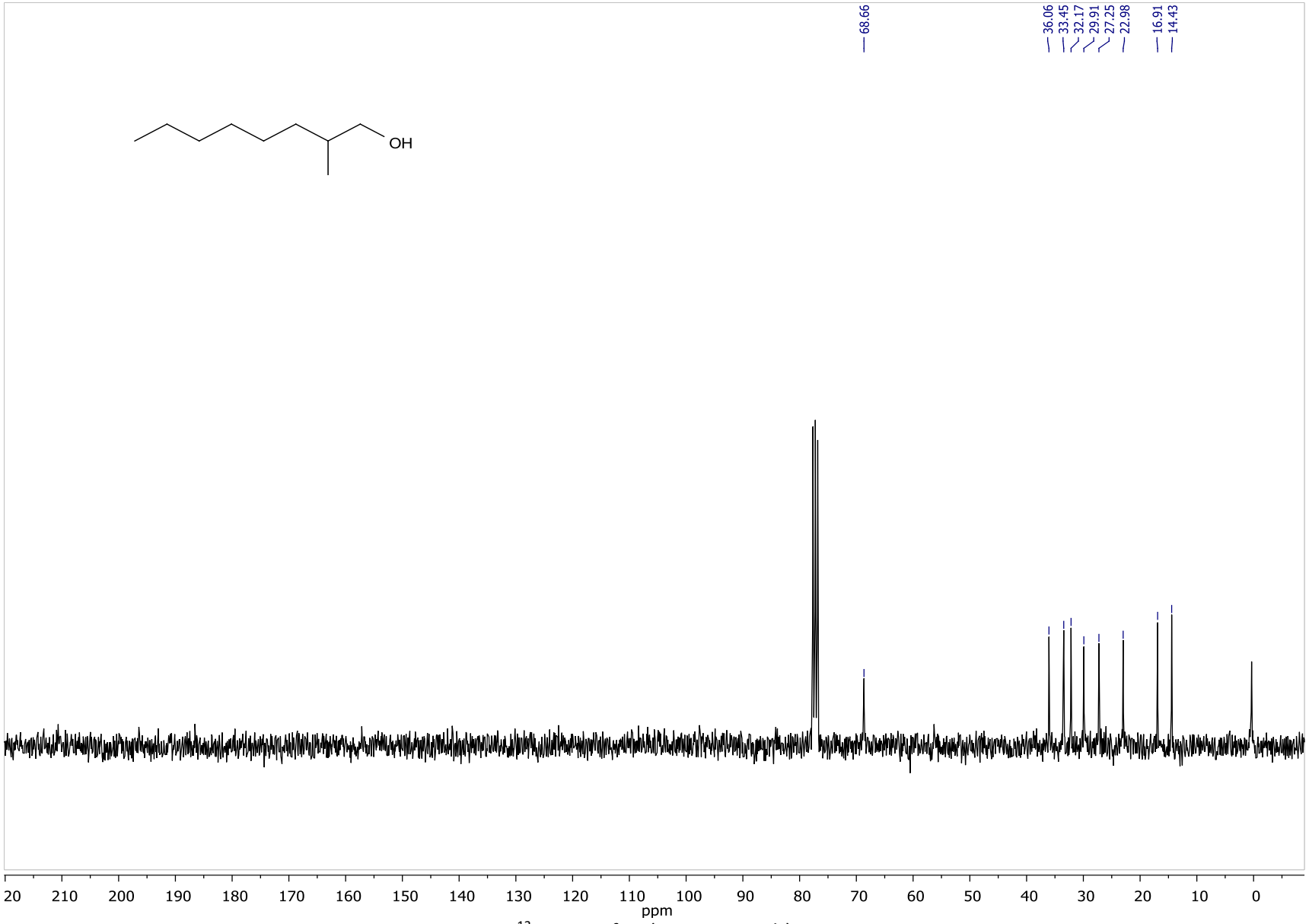
^{1}H NMR of **4b** (300 MHz, $CDCl_3$)



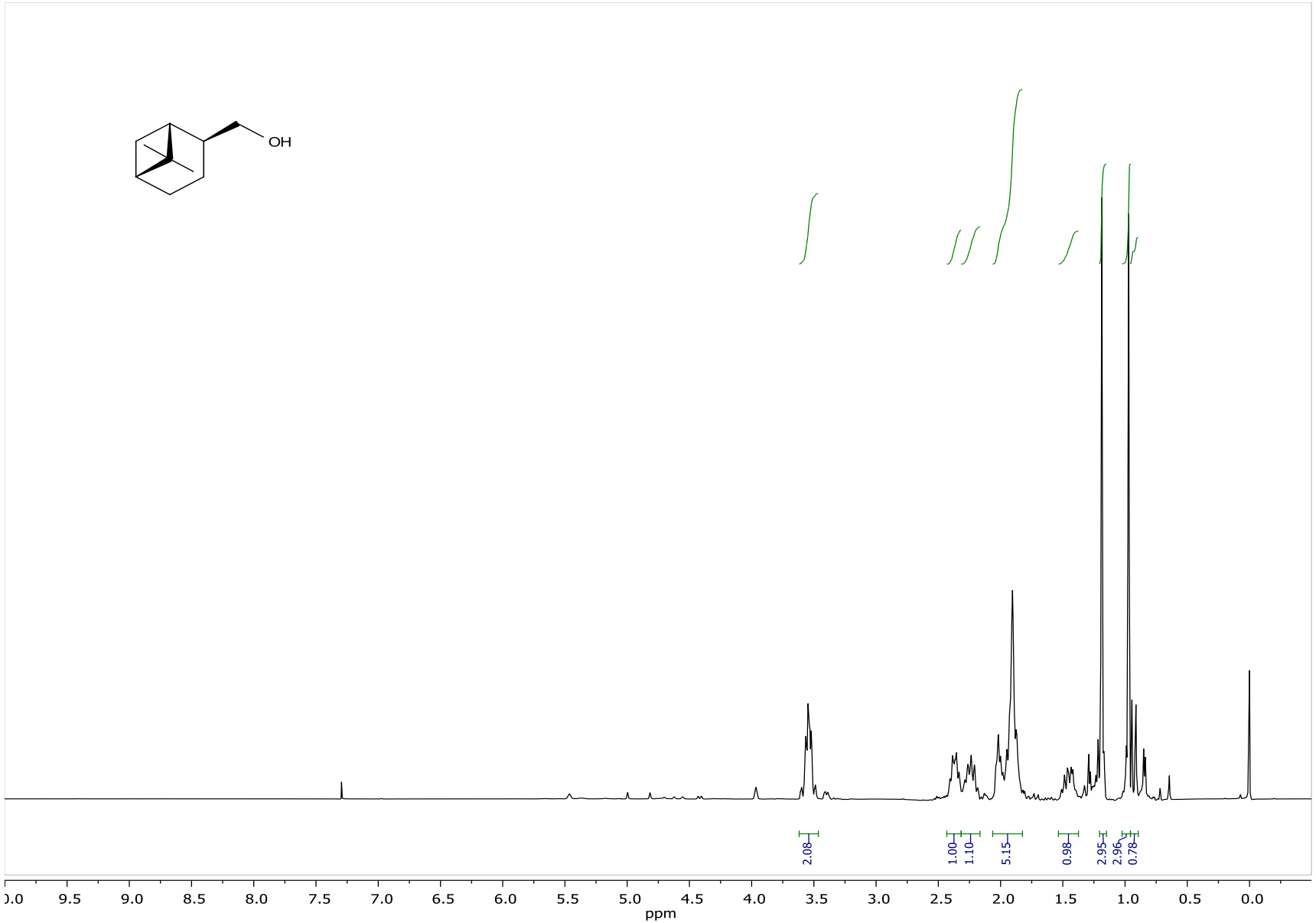
^{13}C NMR of **4b** (75 MHz, $CDCl_3$)



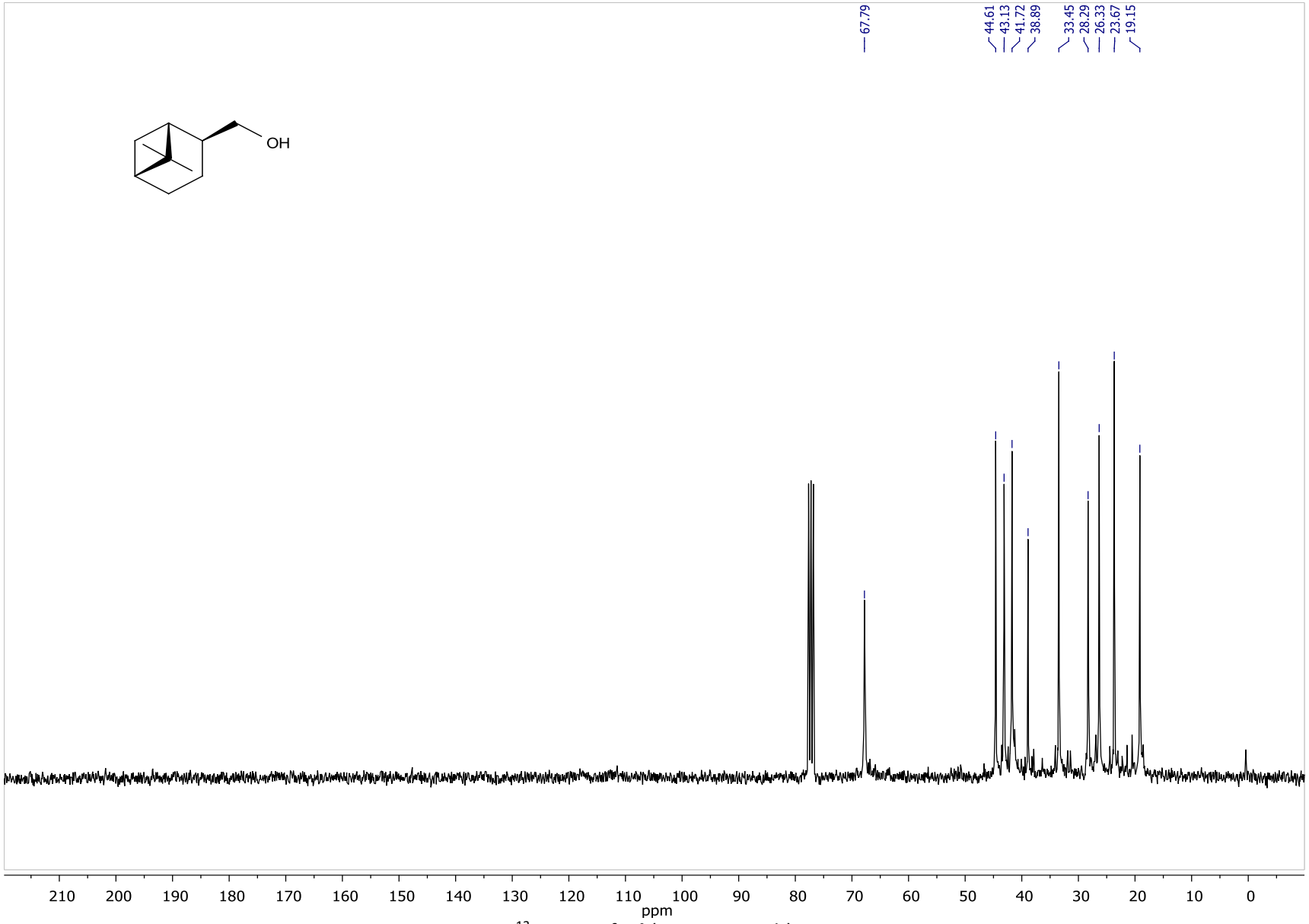
^{1}H NMR of **4c** (300 MHz, $CDCl_3$)



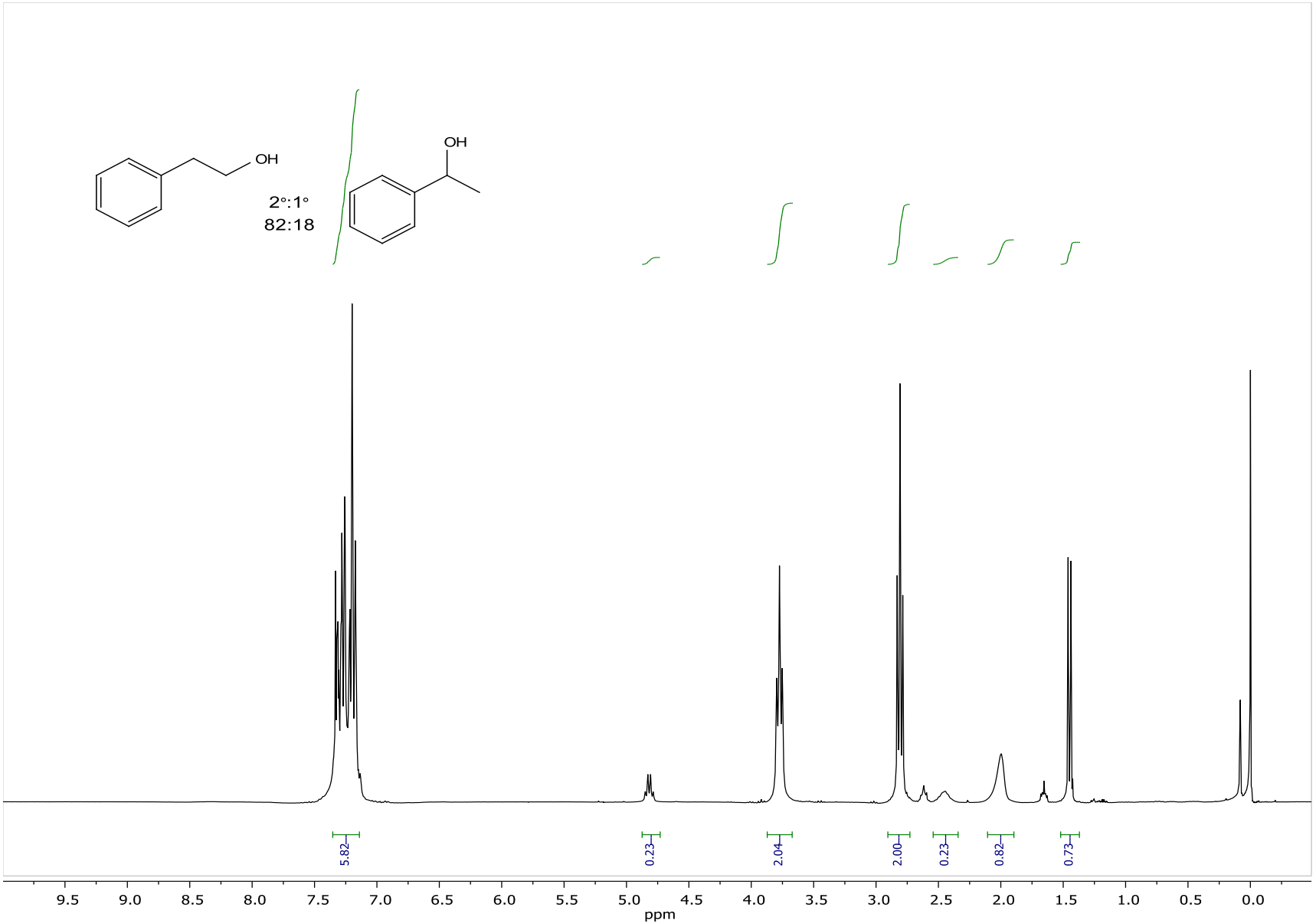
^{13}C NMR of **4c** (75 MHz, $CDCl_3$)



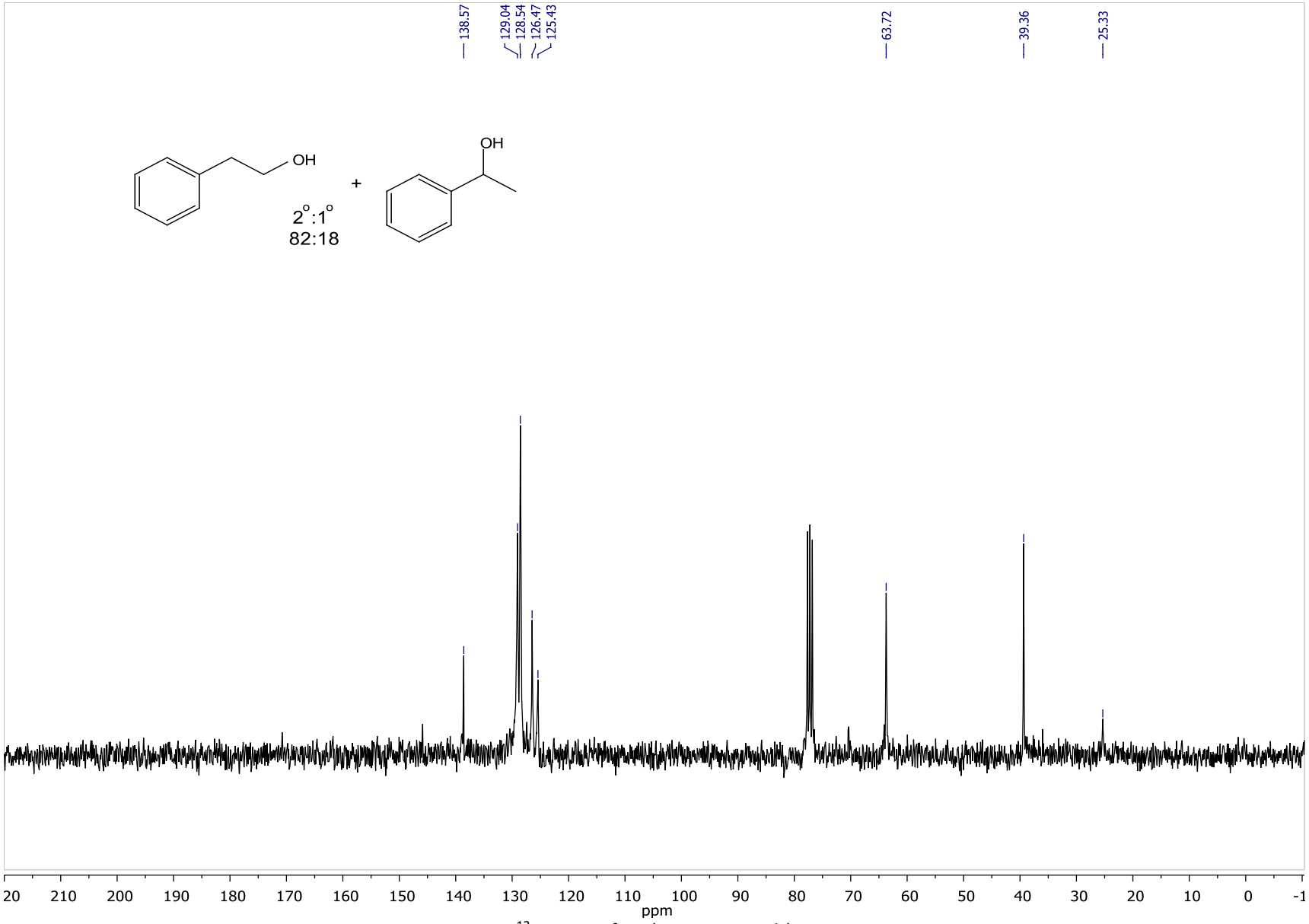
^{1}H NMR of **4d** (300 MHz, $CDCl_3$)



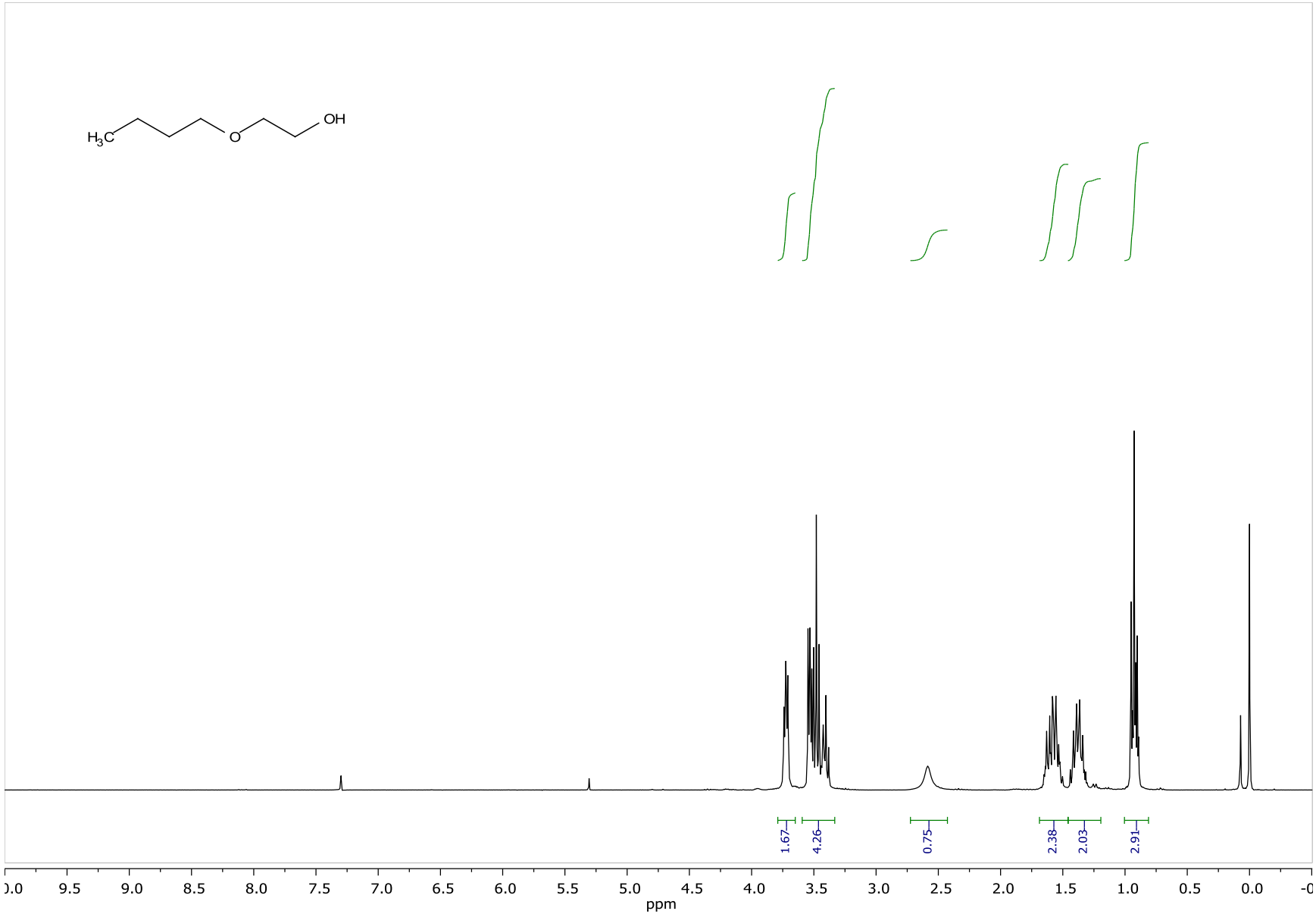
^{13}C NMR of **4d** (75 MHz, $CDCl_3$)



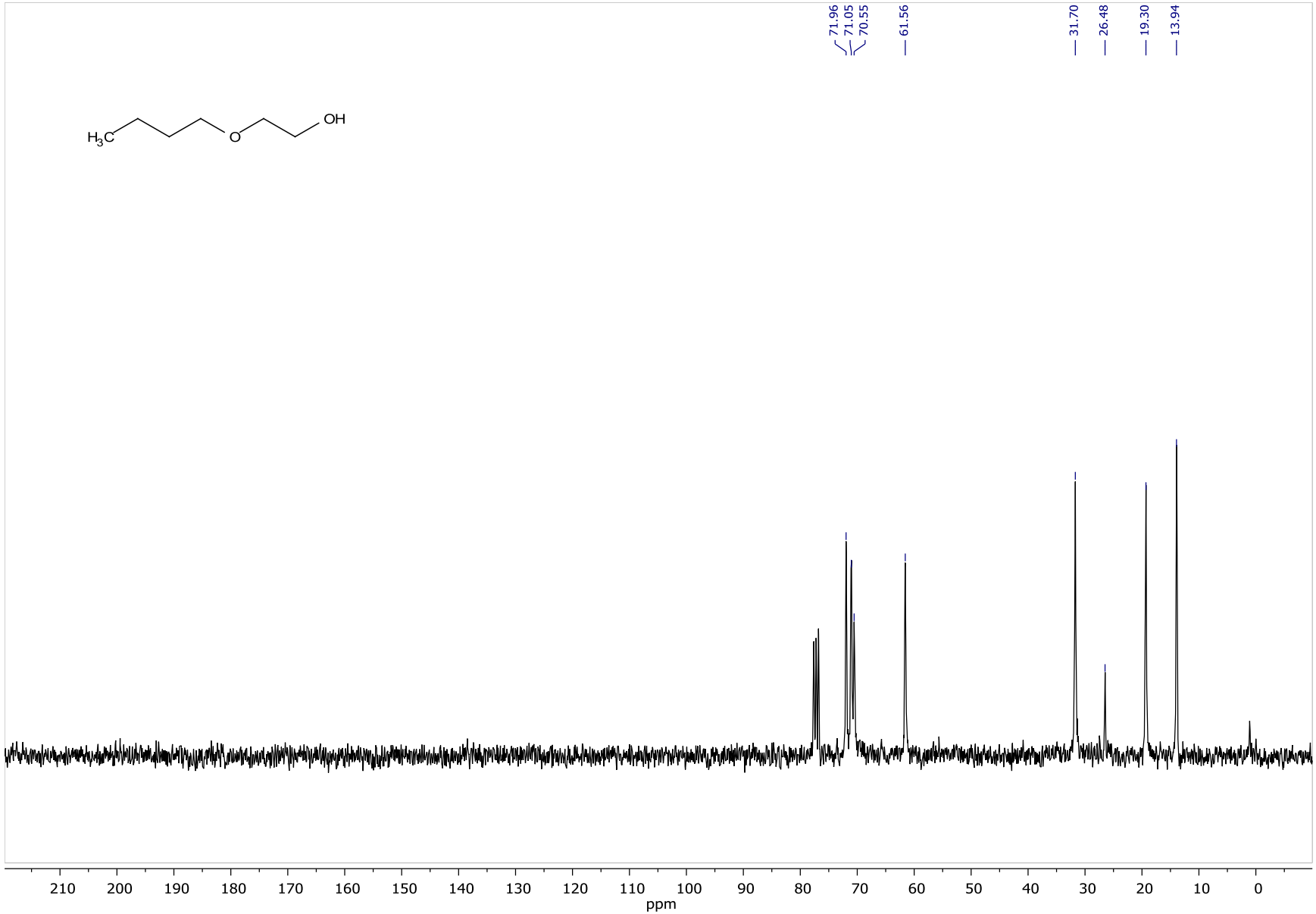
^{1}H NMR of **4e** (300 MHz, $CDCl_3$)



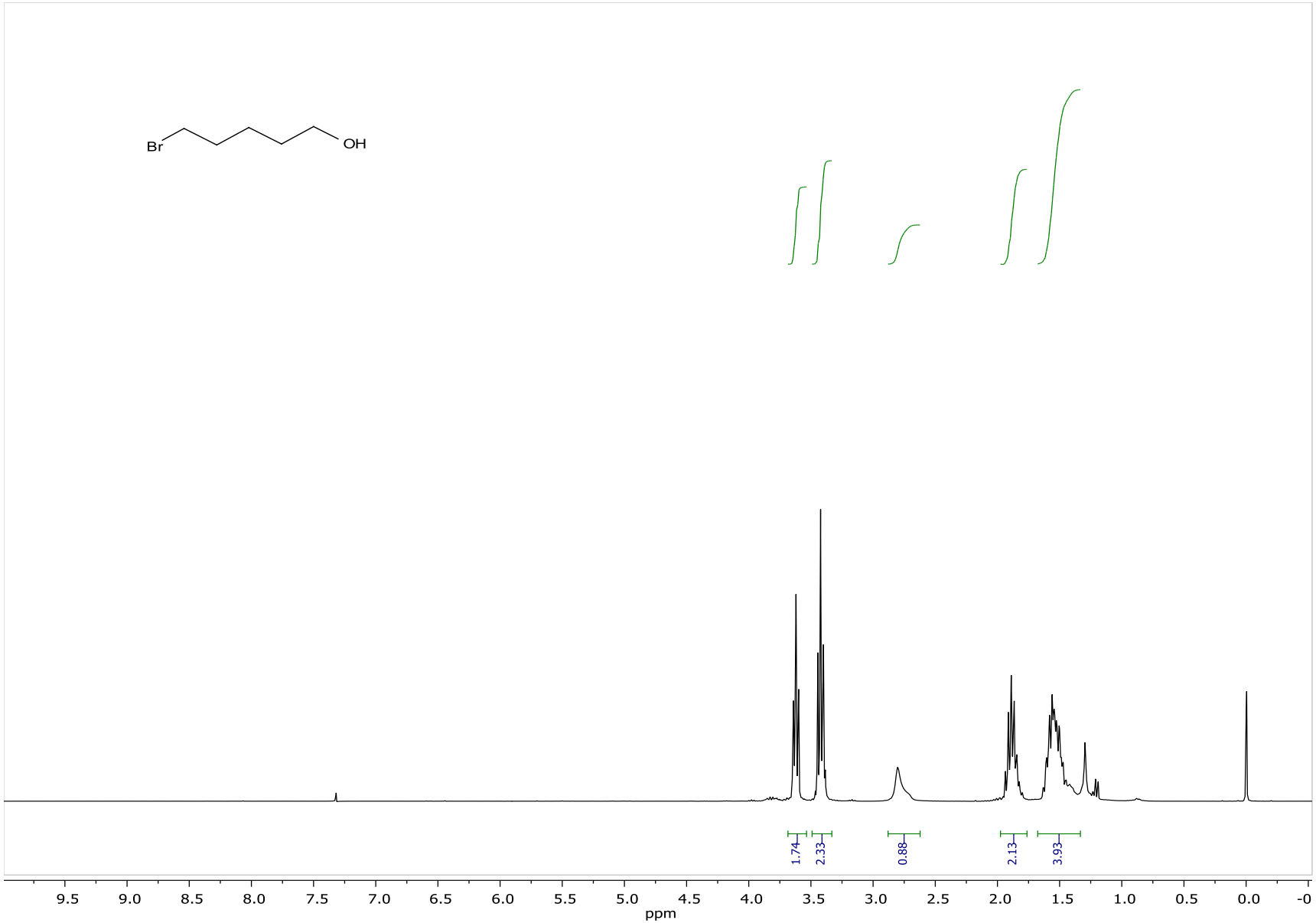
^{13}C NMR of **4e** (75 MHz, $CDCl_3$)



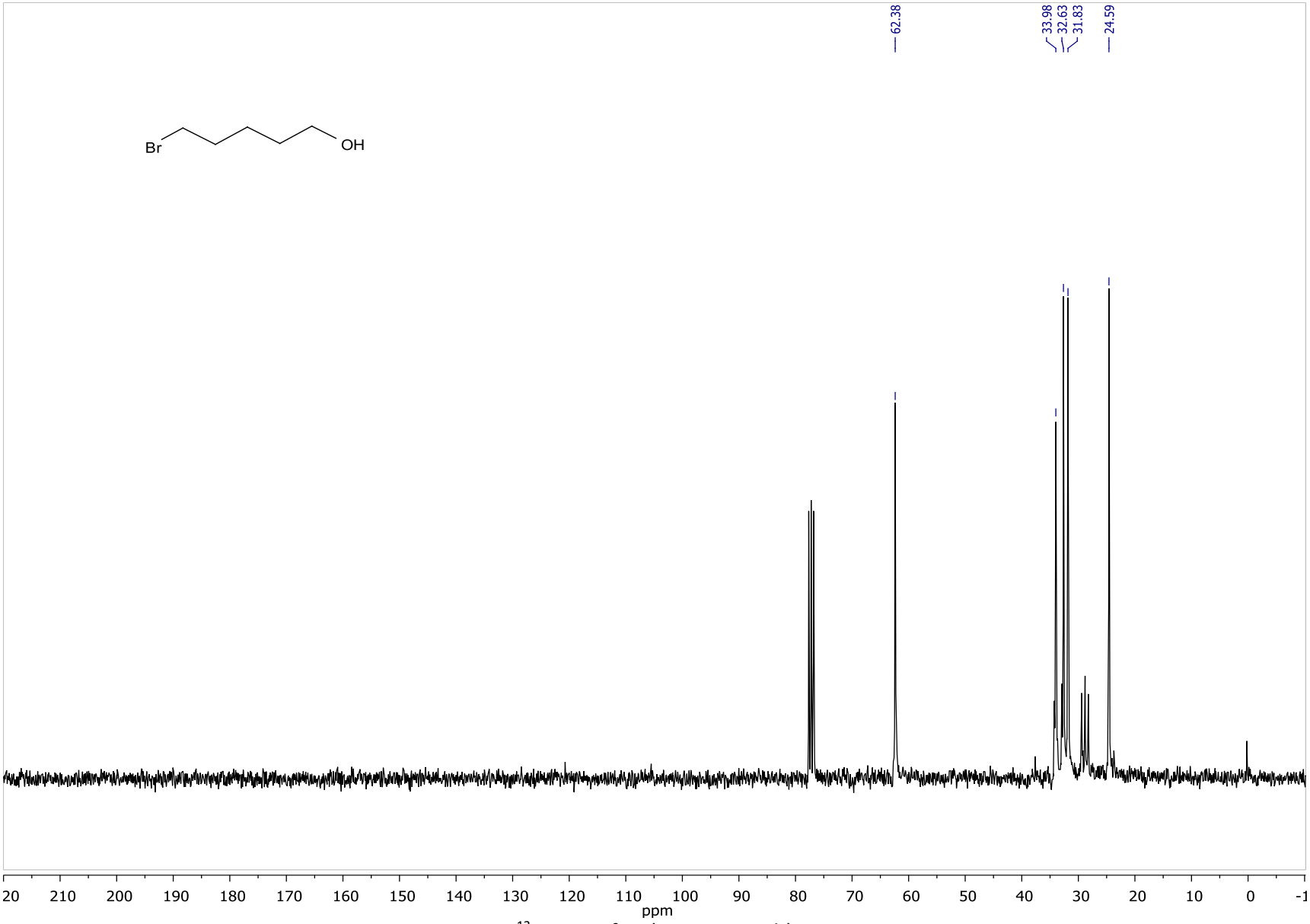
^{1}H NMR of **4f** (300 MHz, $CDCl_3$)



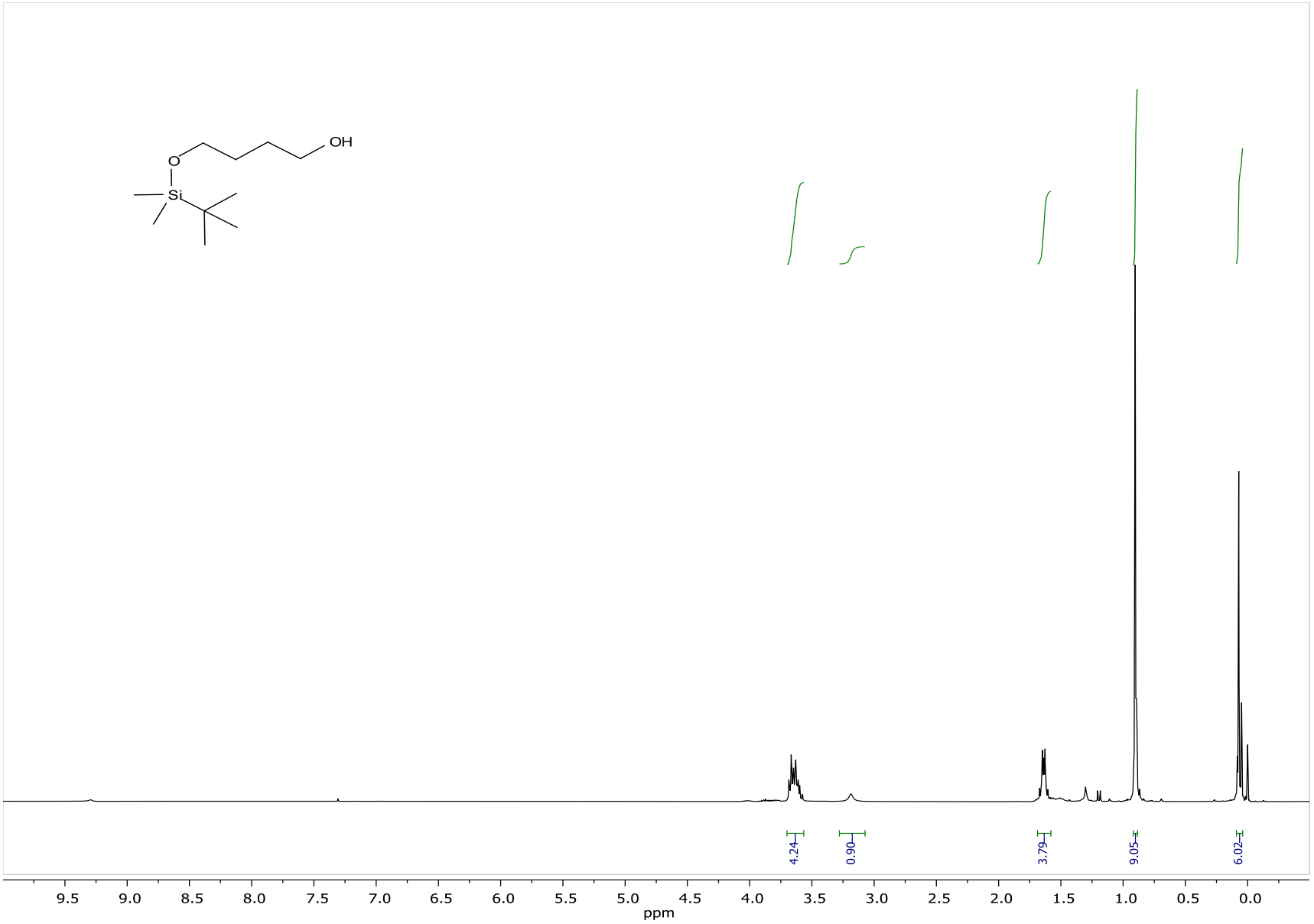
^{13}C NMR of **4f** (75 MHz, $CDCl_3$)



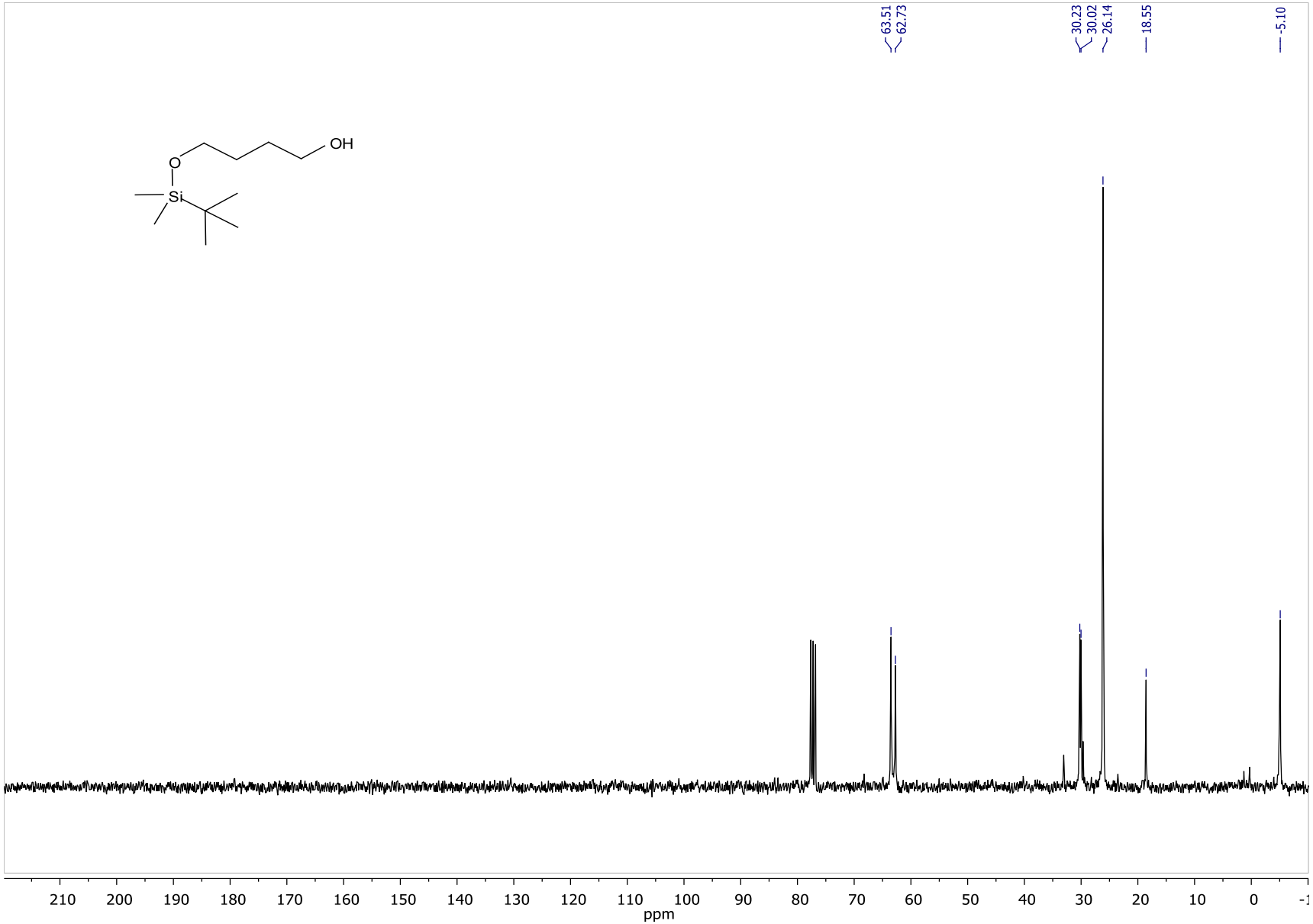
^{1}H NMR spectrum of **4g** (300 MHz, $CDCl_3$)



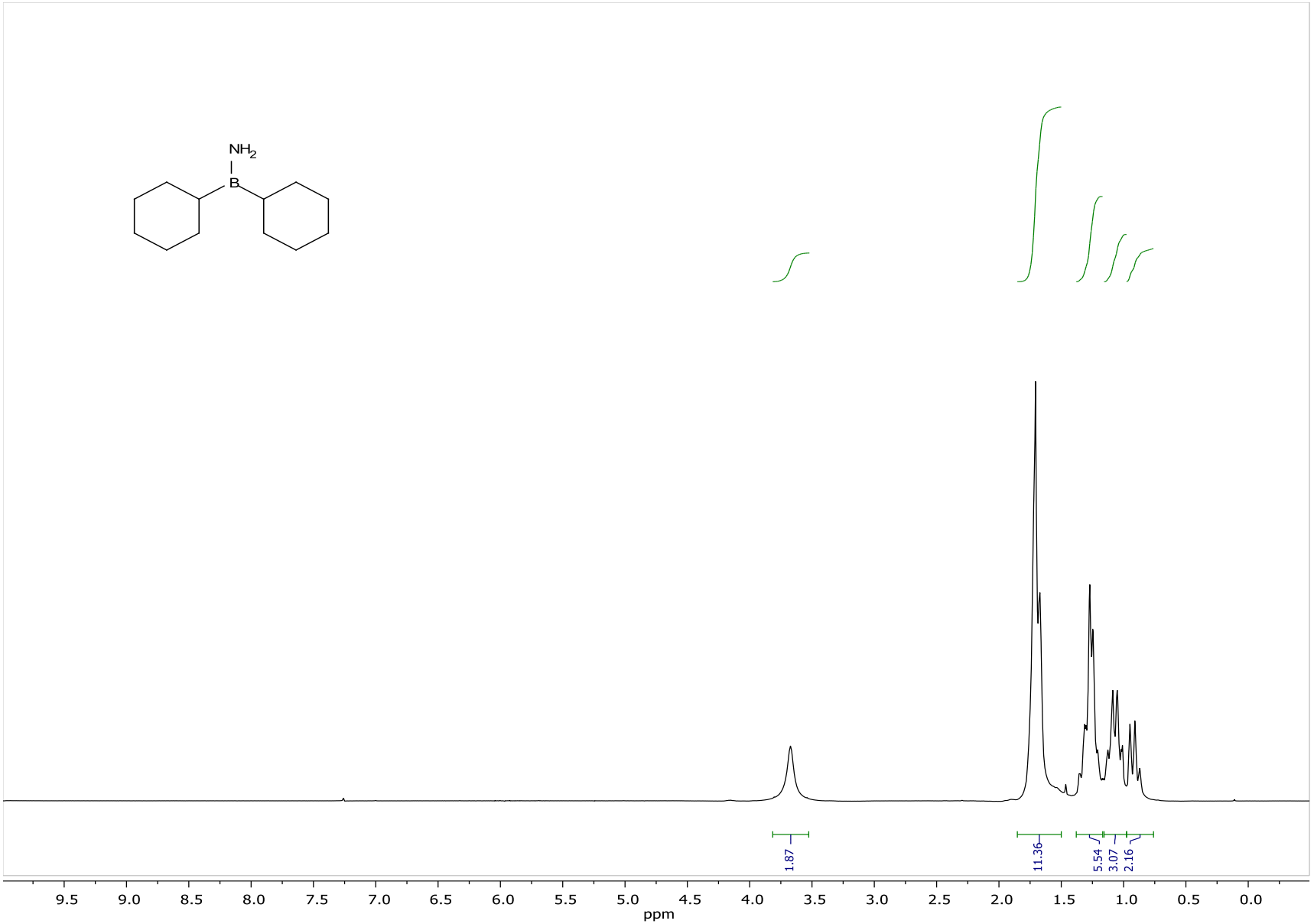
^{13}C NMR of **4g** (75 MHz, $CDCl_3$)



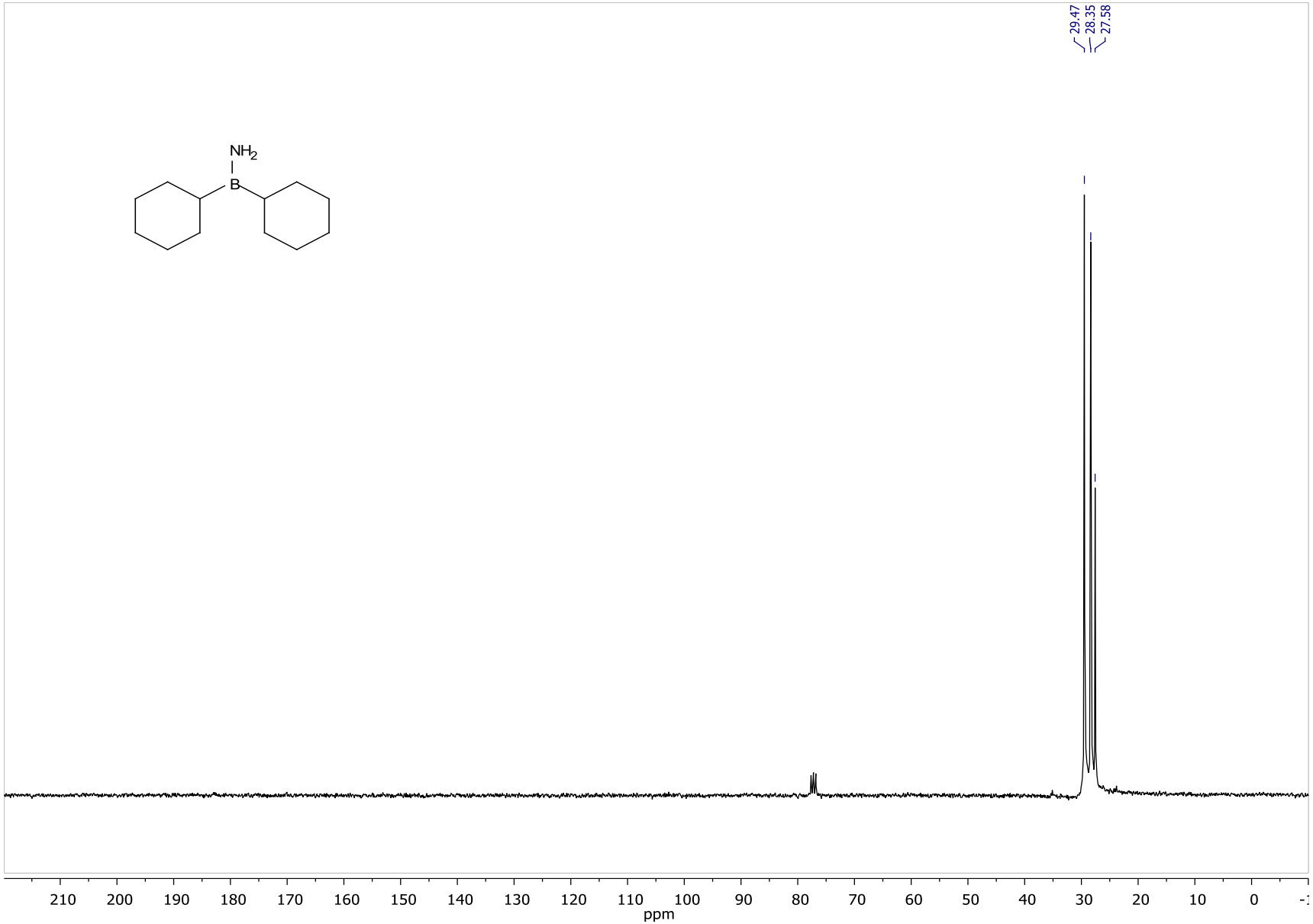
^{1}H NMR of **4h** (300 MHz, $CDCl_3$)



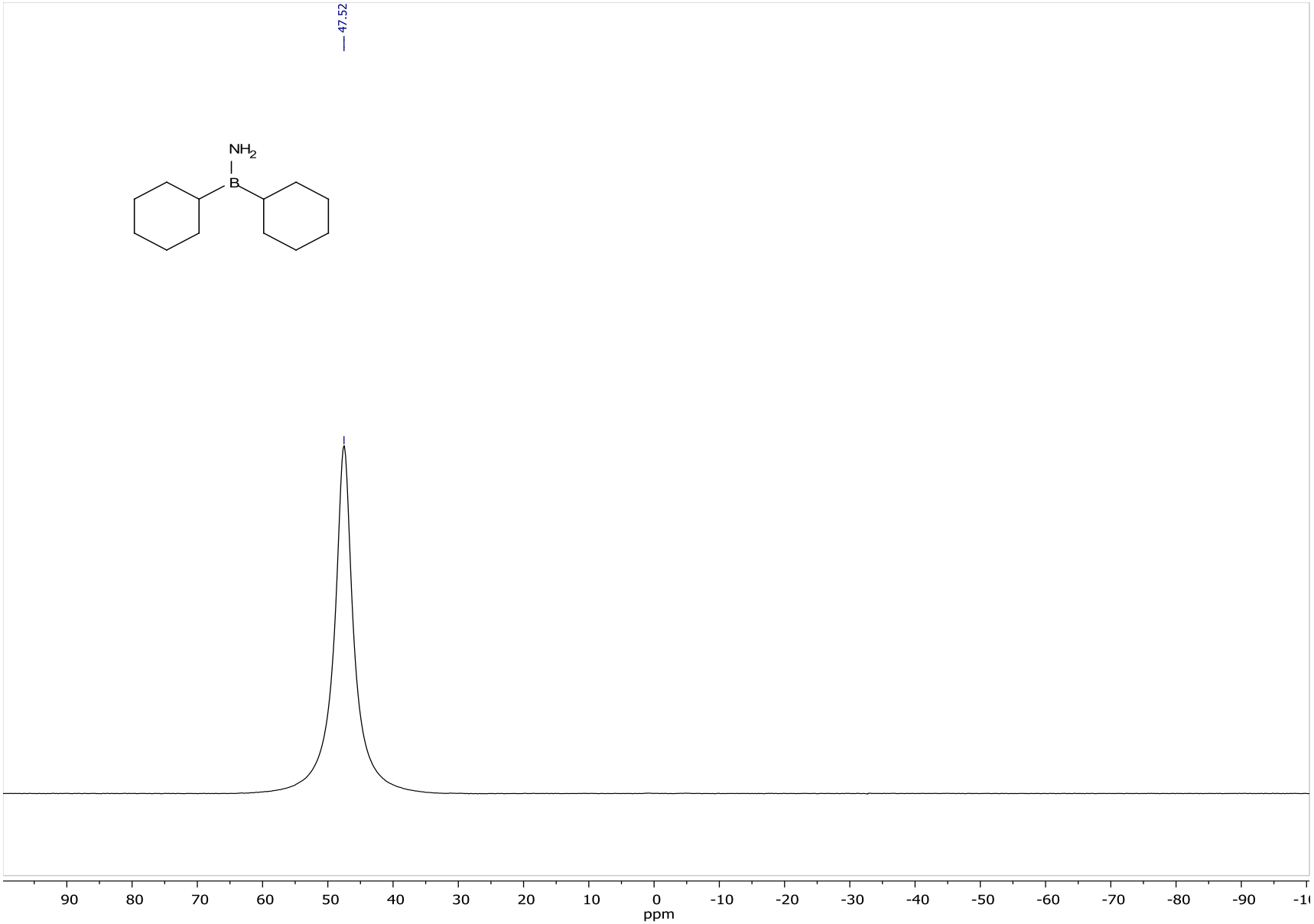
^{13}C NMR of **4h** (75 MHz, $CDCl_3$)



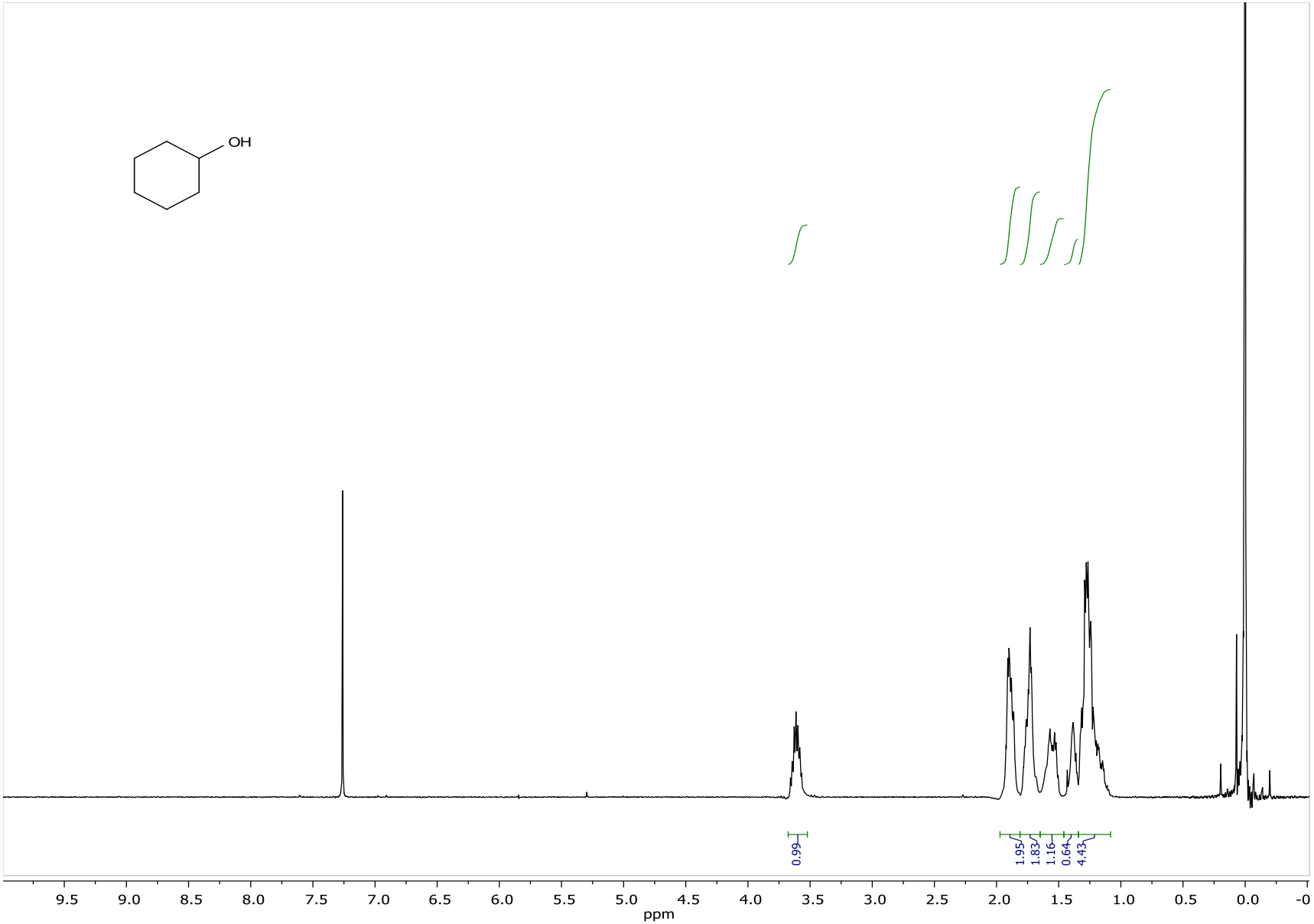
^{1}H NMR of **5j** (300 MHz, $CDCl_3$)



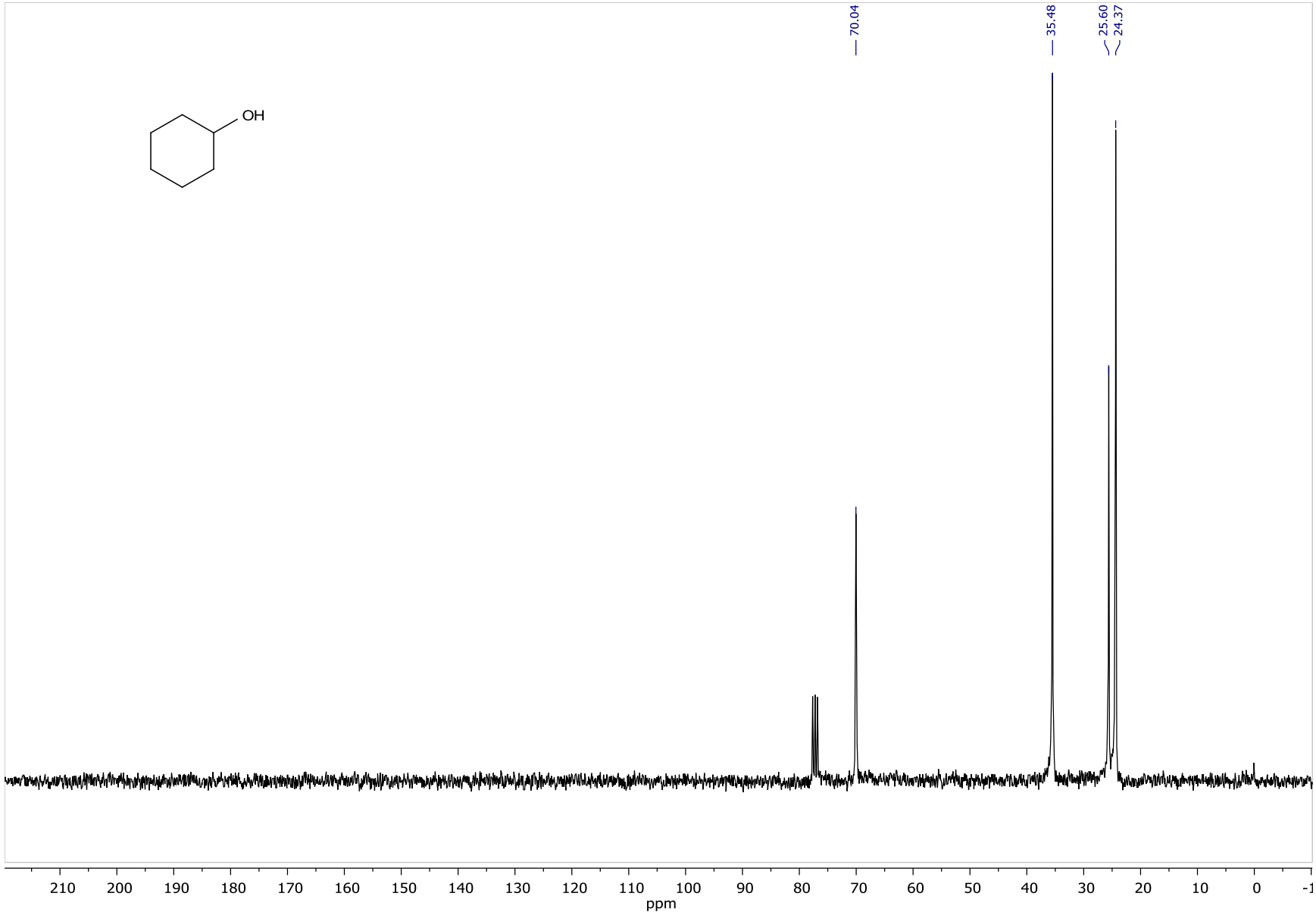
^{13}C NMR of **5j** (75 MHz, $CDCl_3$)



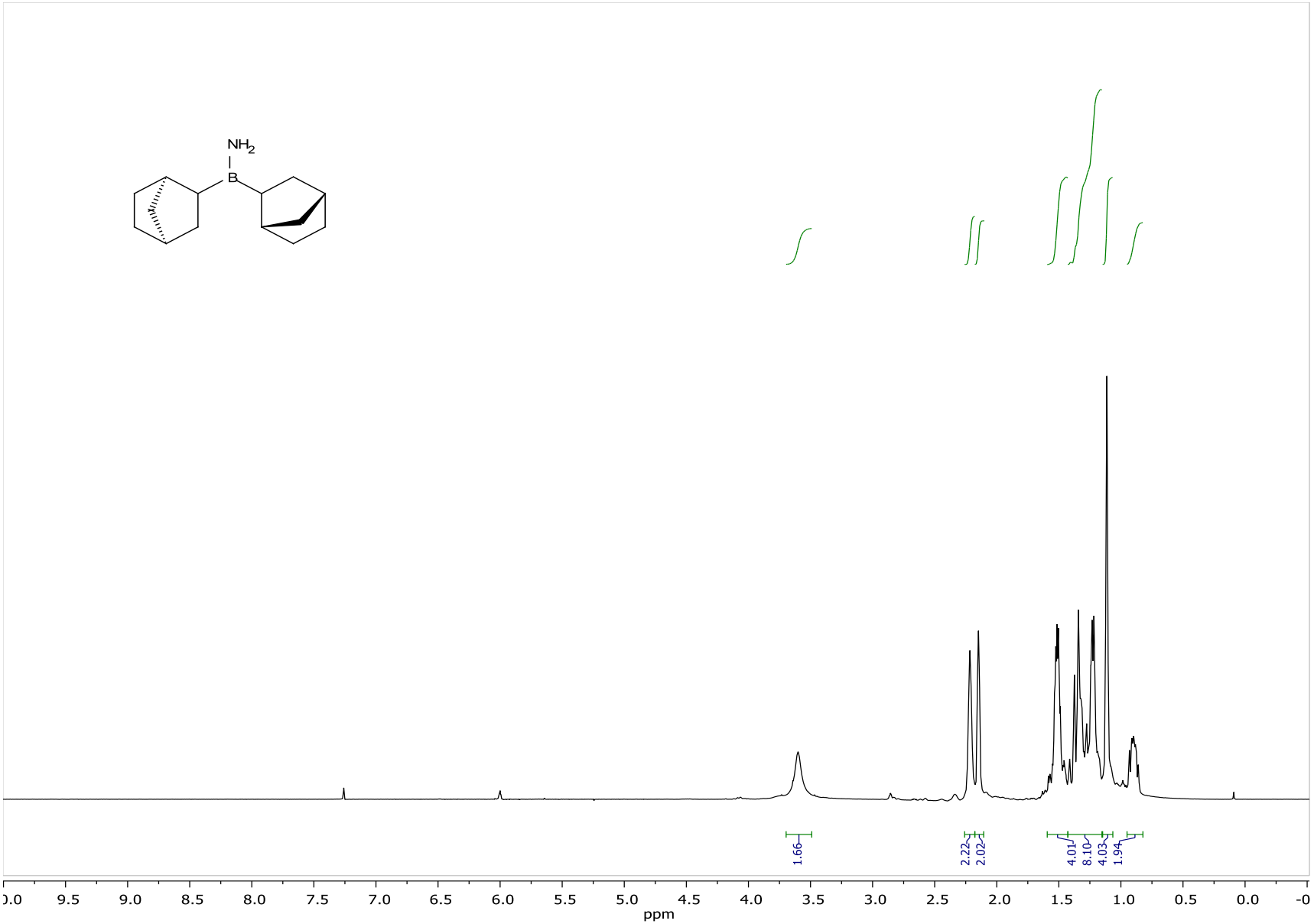
^{11}B NMR of **5j** (96 MHz, $CDCl_3$)



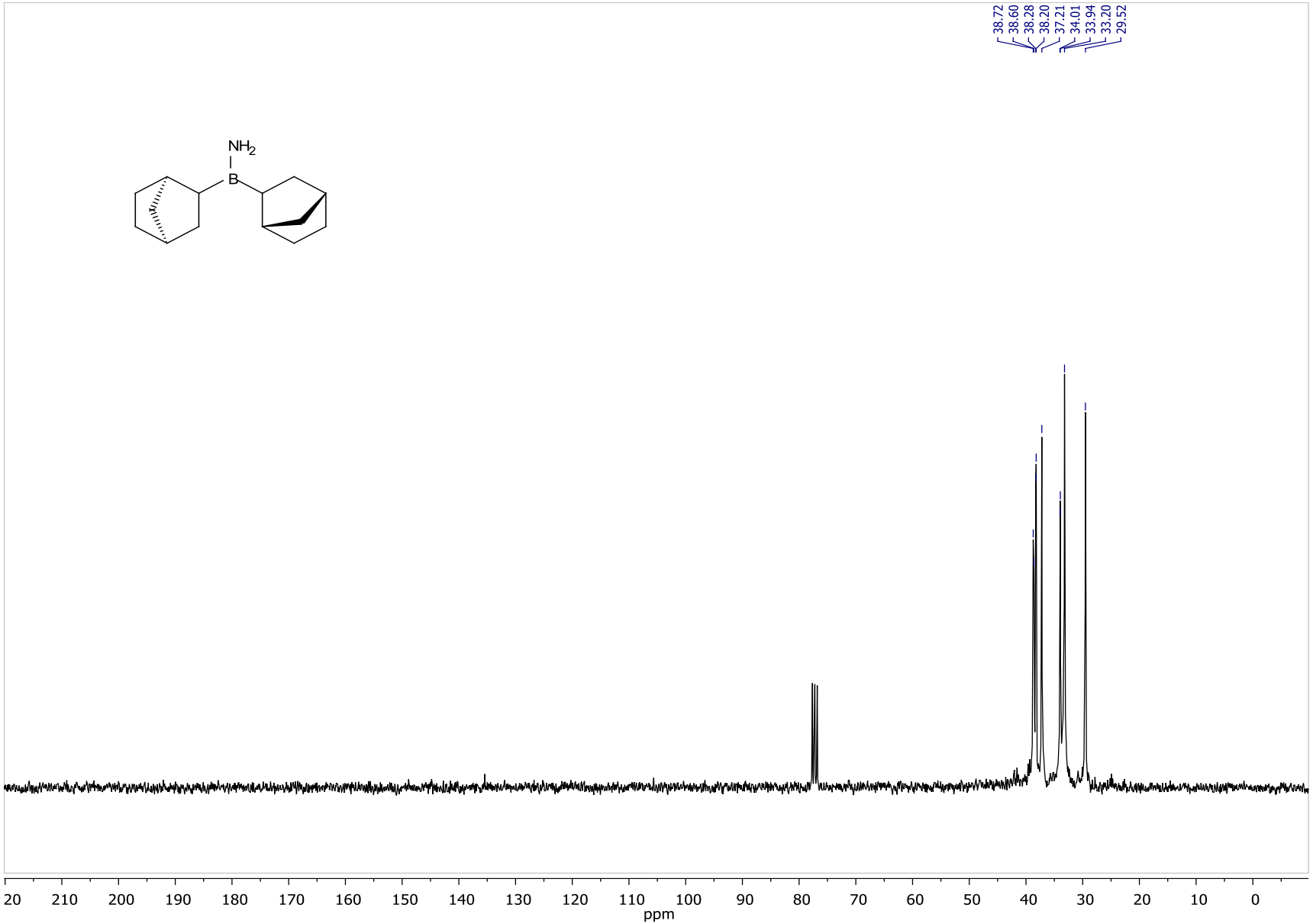
^{1}H NMR of **4j** (300 MHz, $CDCl_3$)



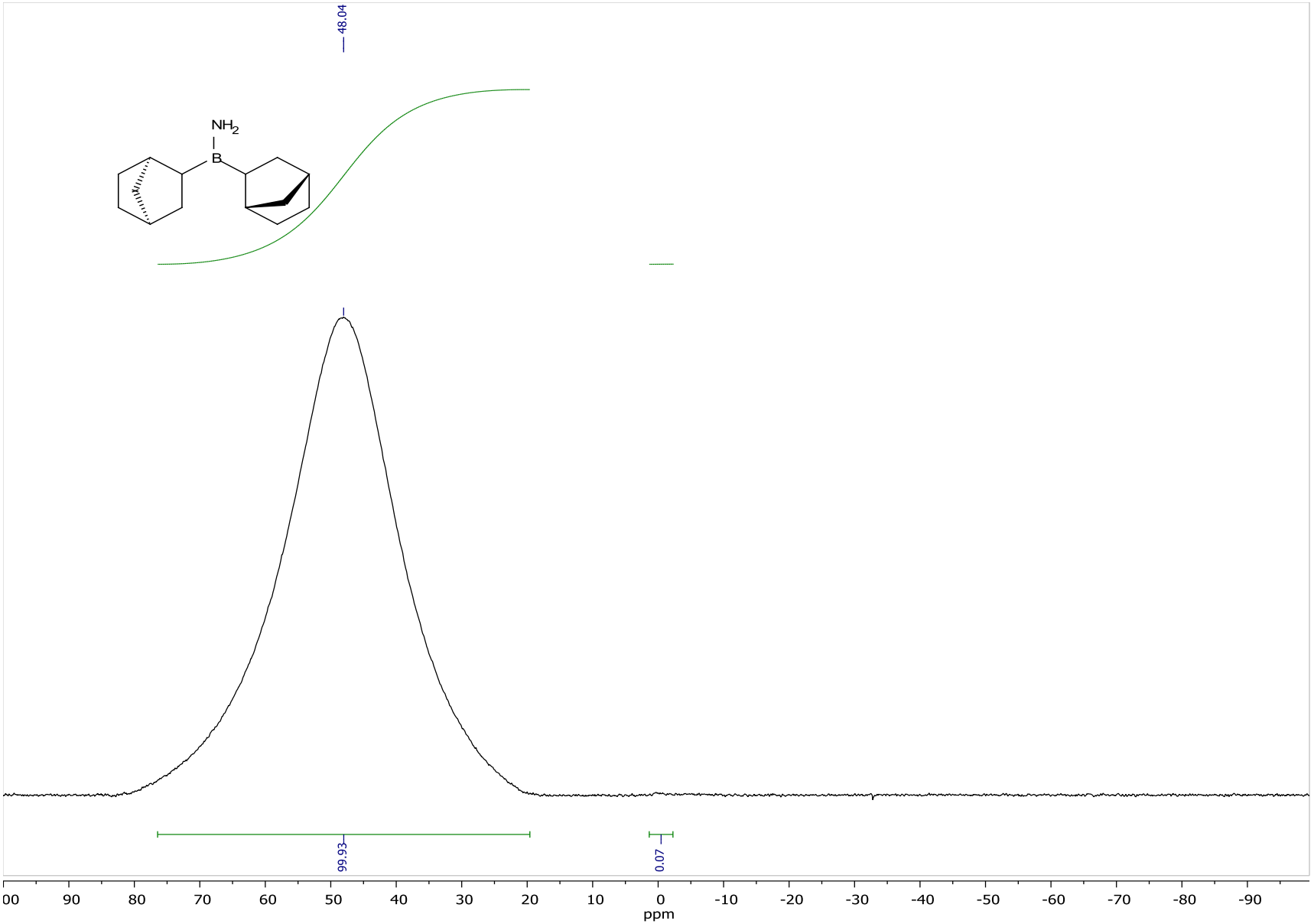
^{13}C NMR of **4j** (75 MHz, $CDCl_3$)



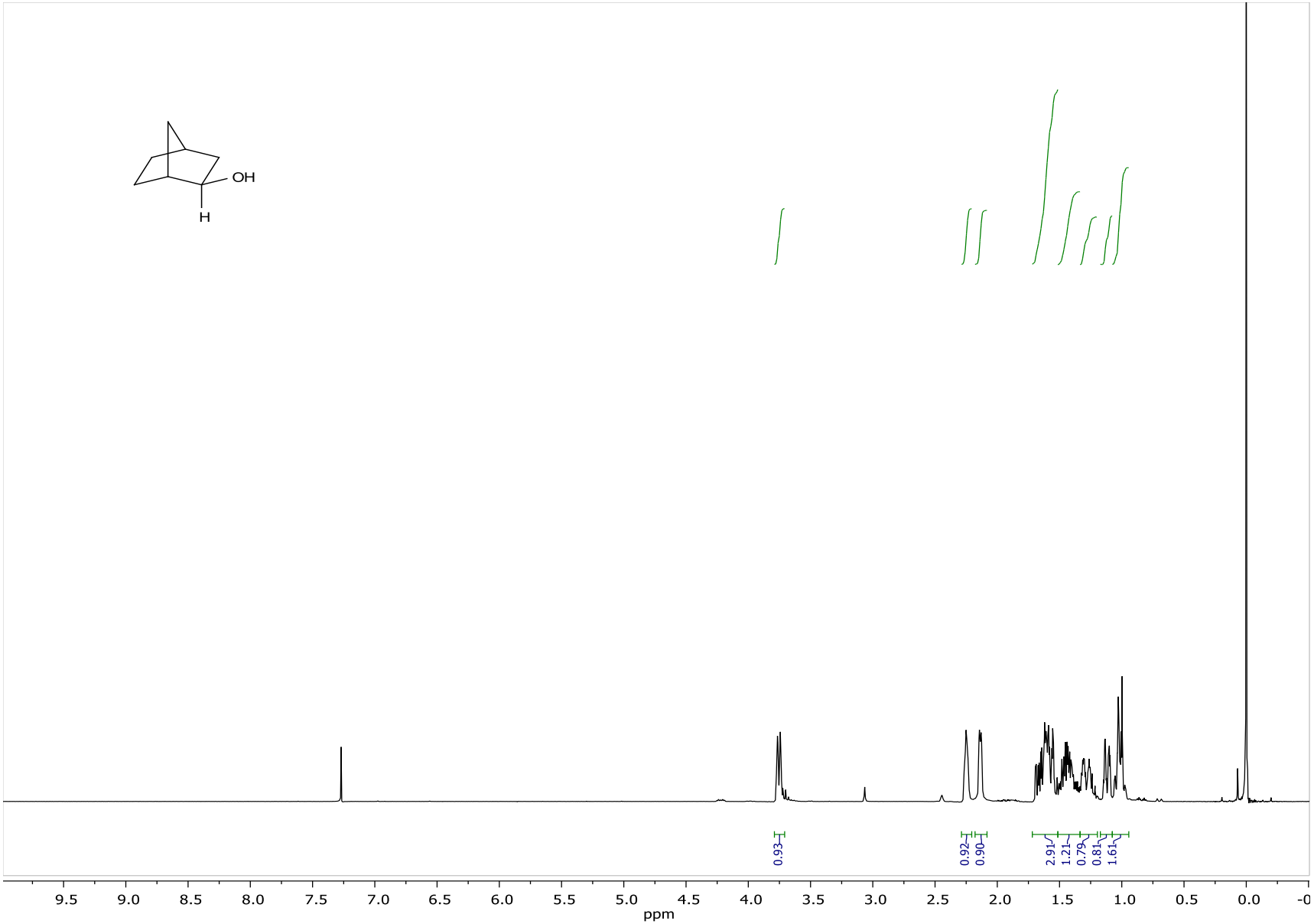
^{1}H NMR of **5k** (300 MHz, $CDCl_3$)



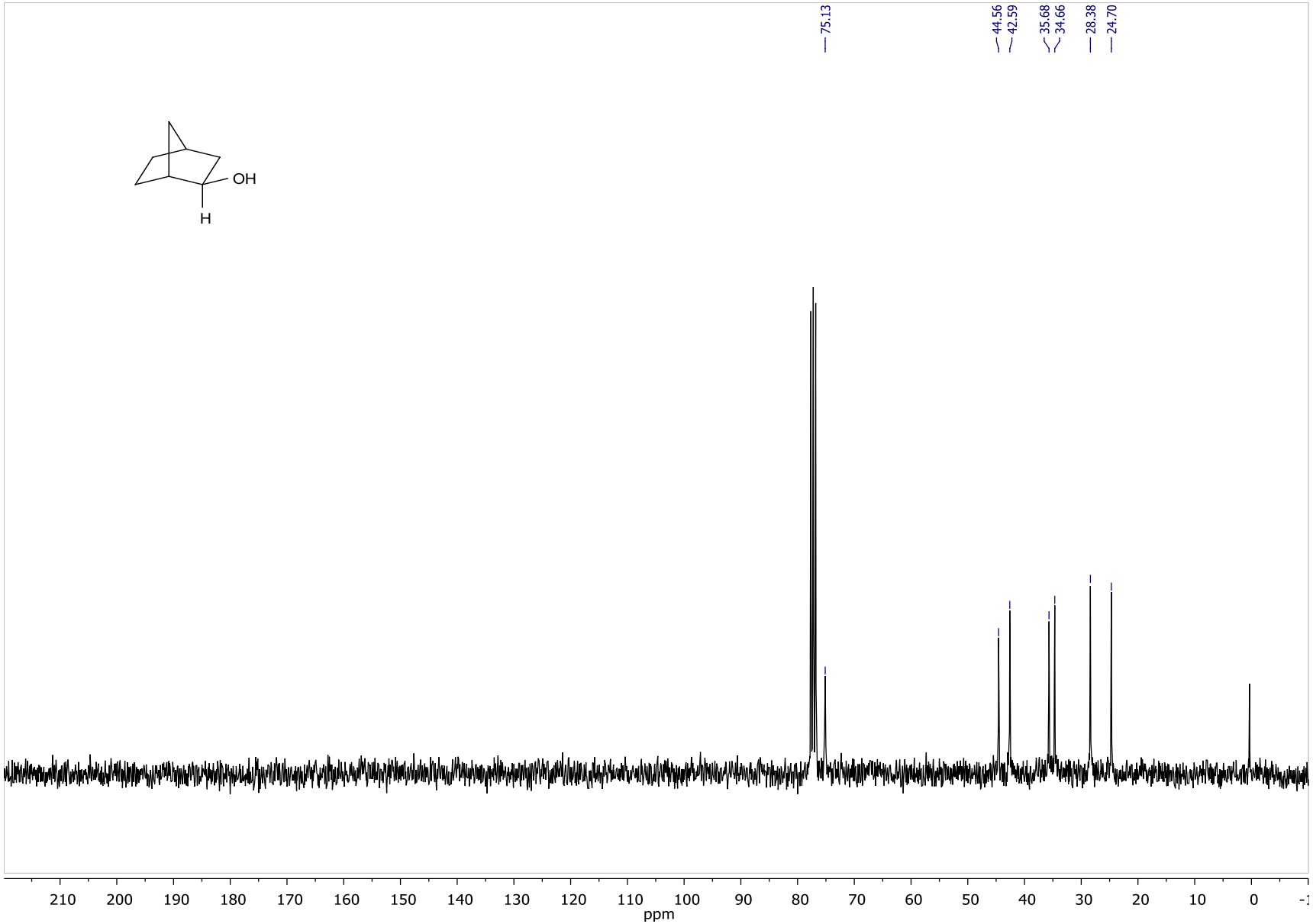
^{13}C NMR of **5k** (75 MHz, $CDCl_3$)



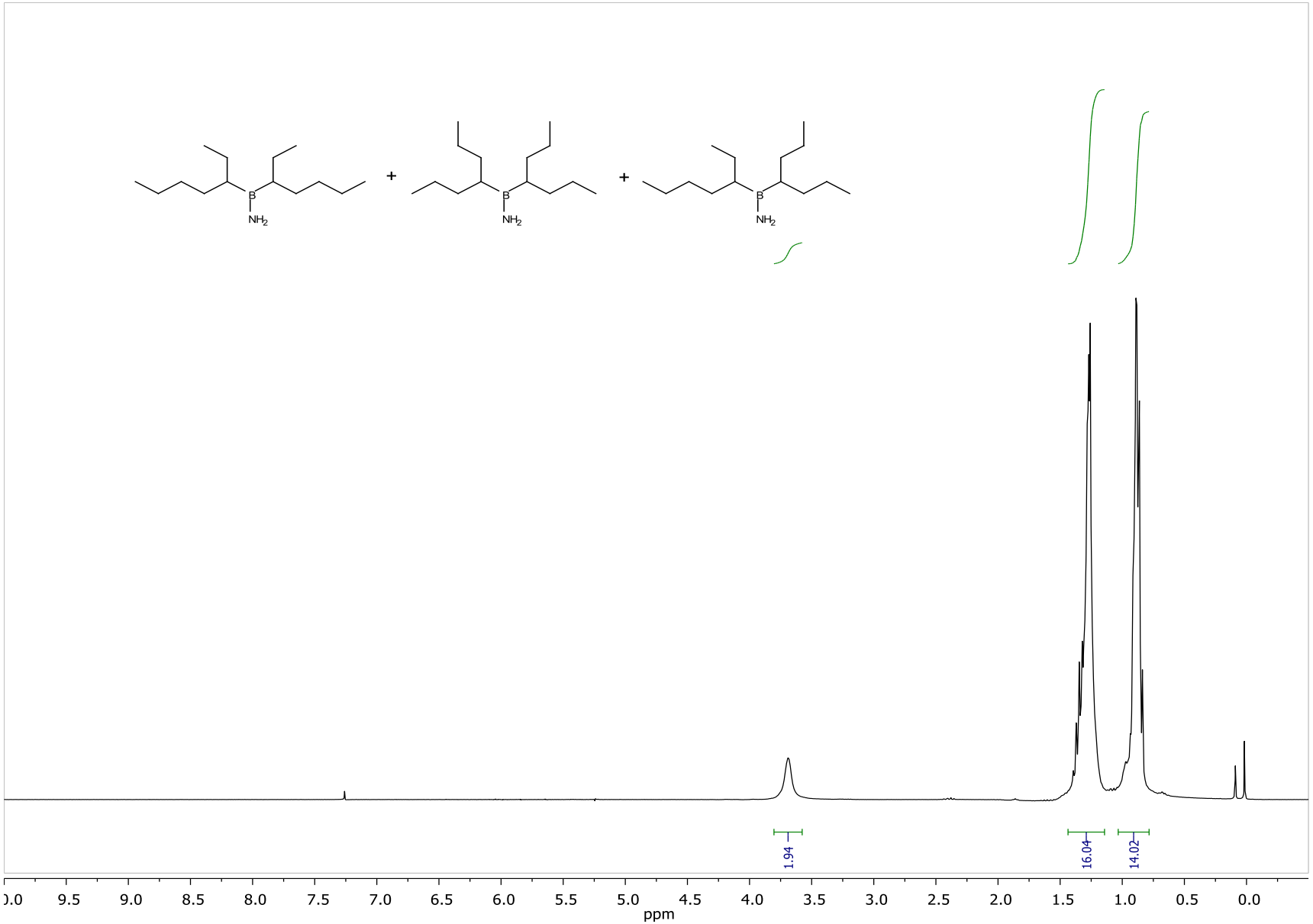
^{11}B NMR of **5k** (96 MHz, $CDCl_3$)



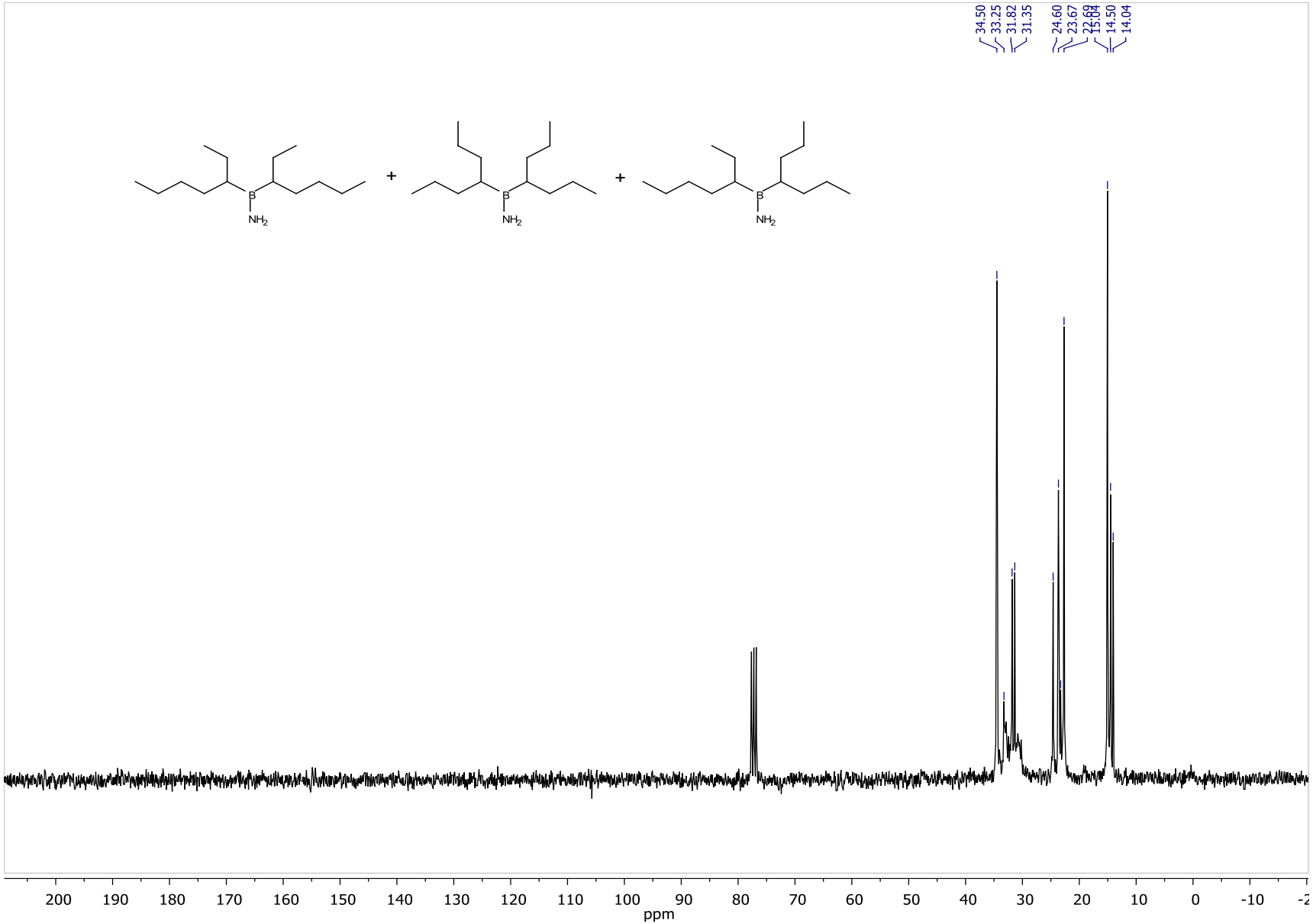
^{1}H NMR of **4k** (300 MHz, $CDCl_3$)



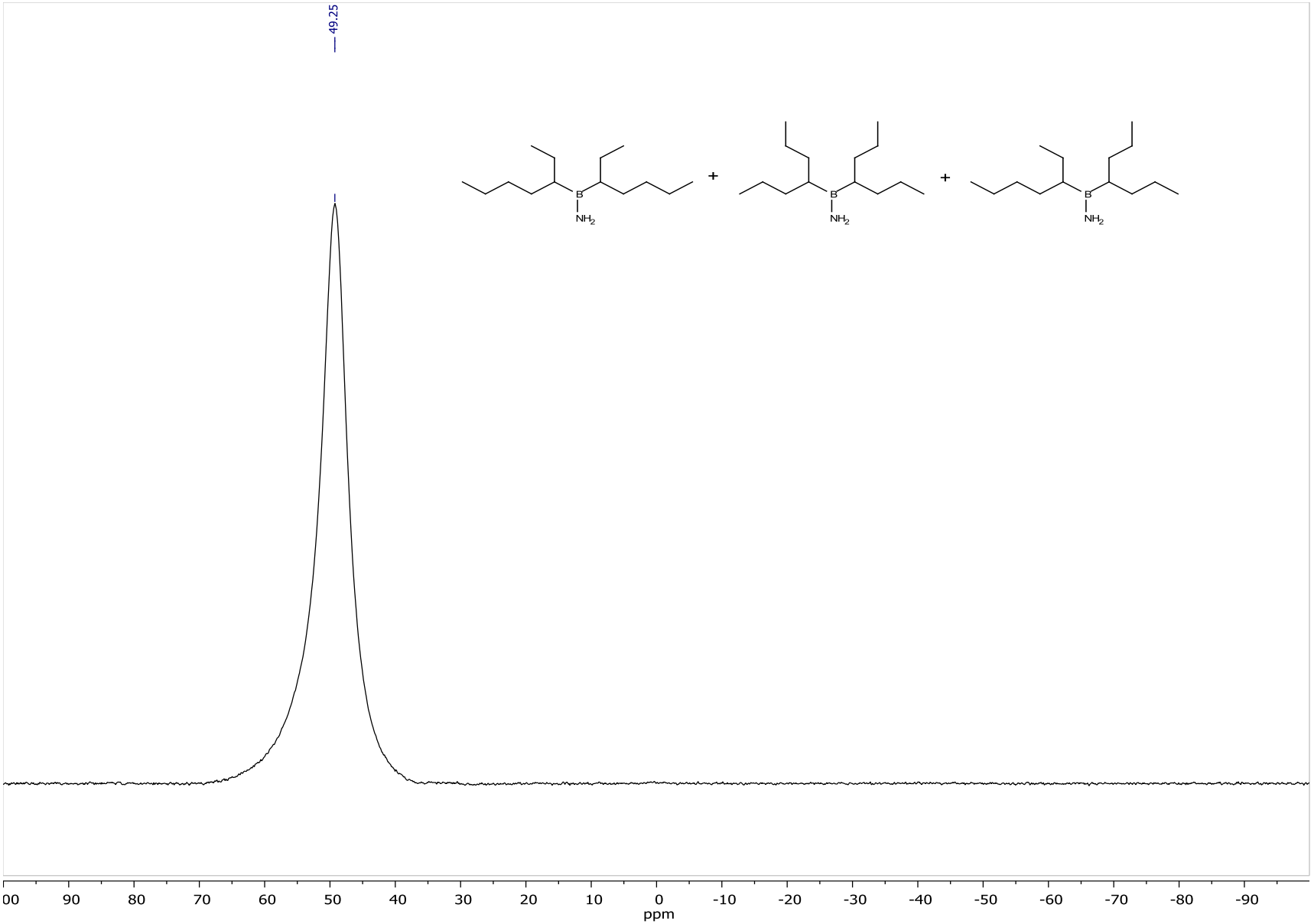
^{13}C NMR of **4k** (75 MHz, $CDCl_3$)



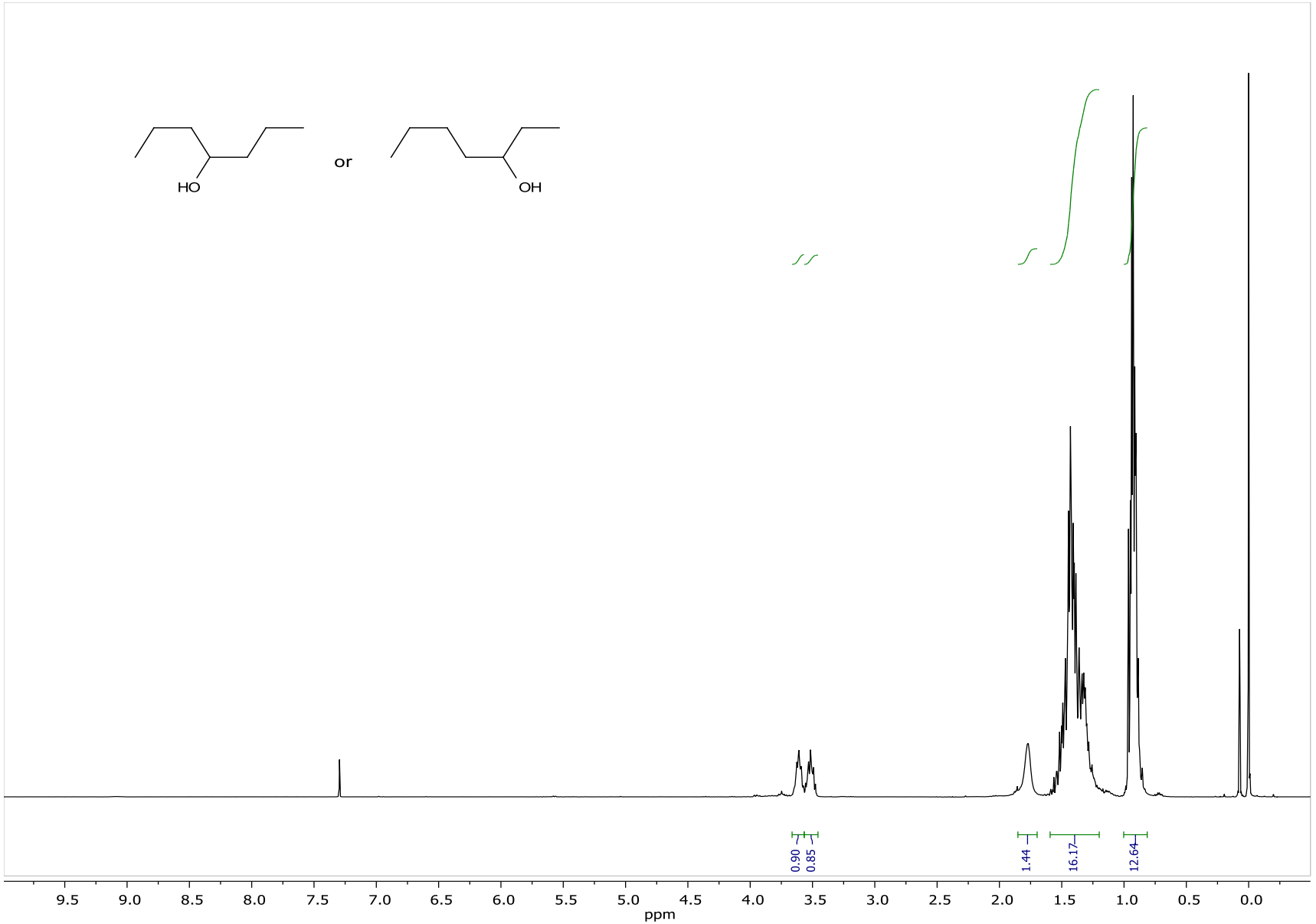
^{1}H NMR of **5l** (300 MHz, $CDCl_3$)



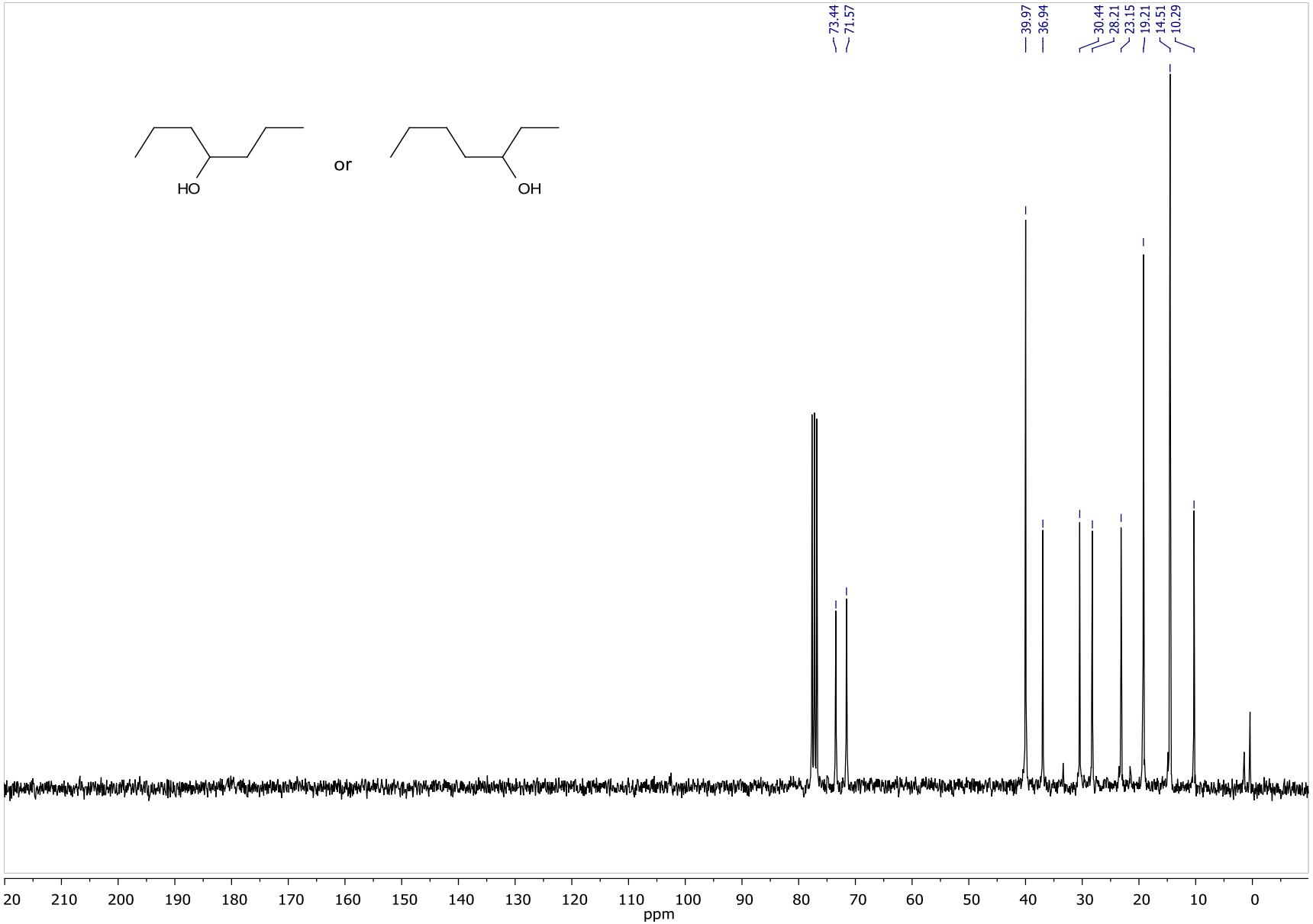
^{13}C NMR of **5l** (75 MHz, $CDCl_3$)



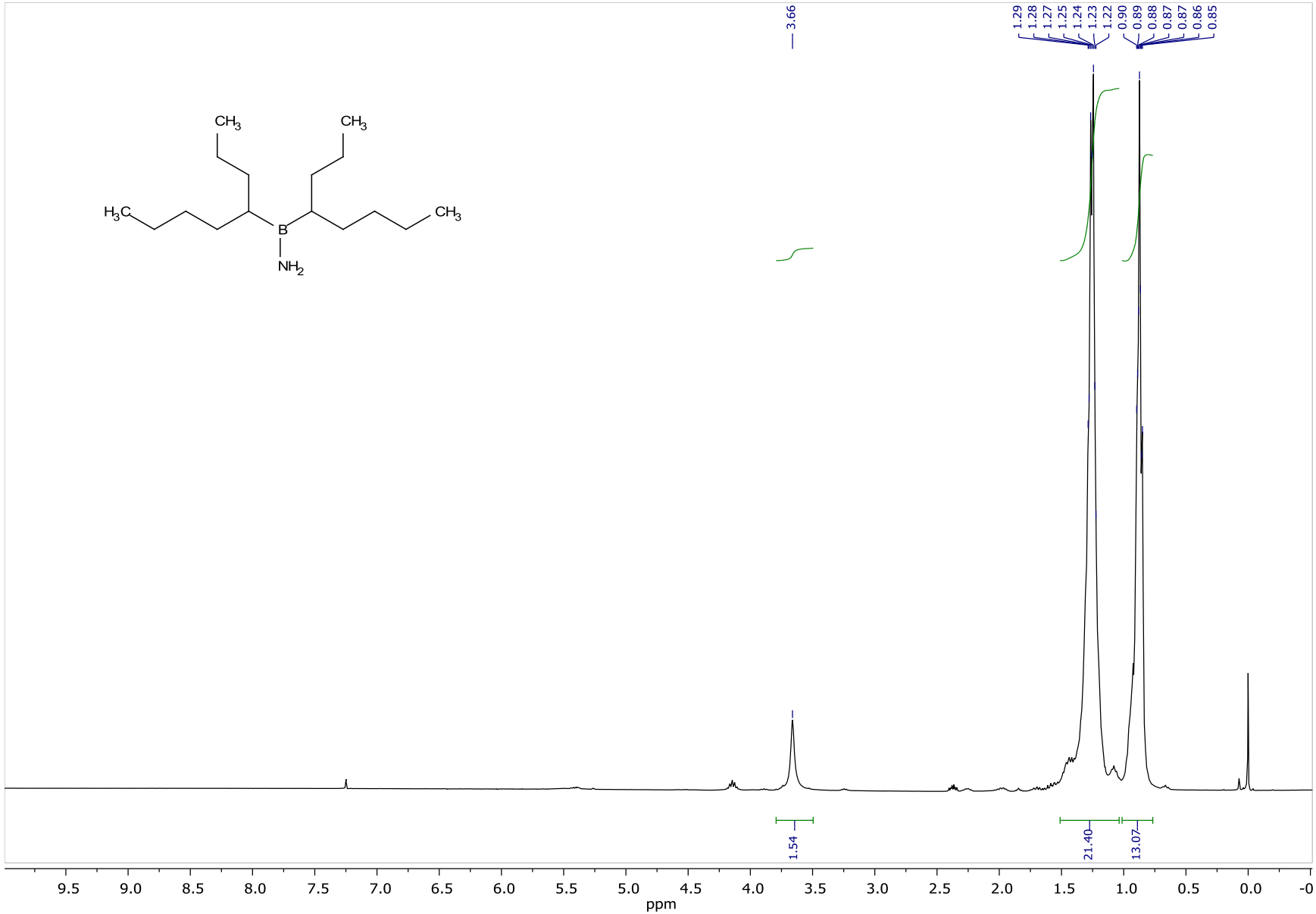
^{11}B NMR of **5l** (96 MHz, $CDCl_3$)



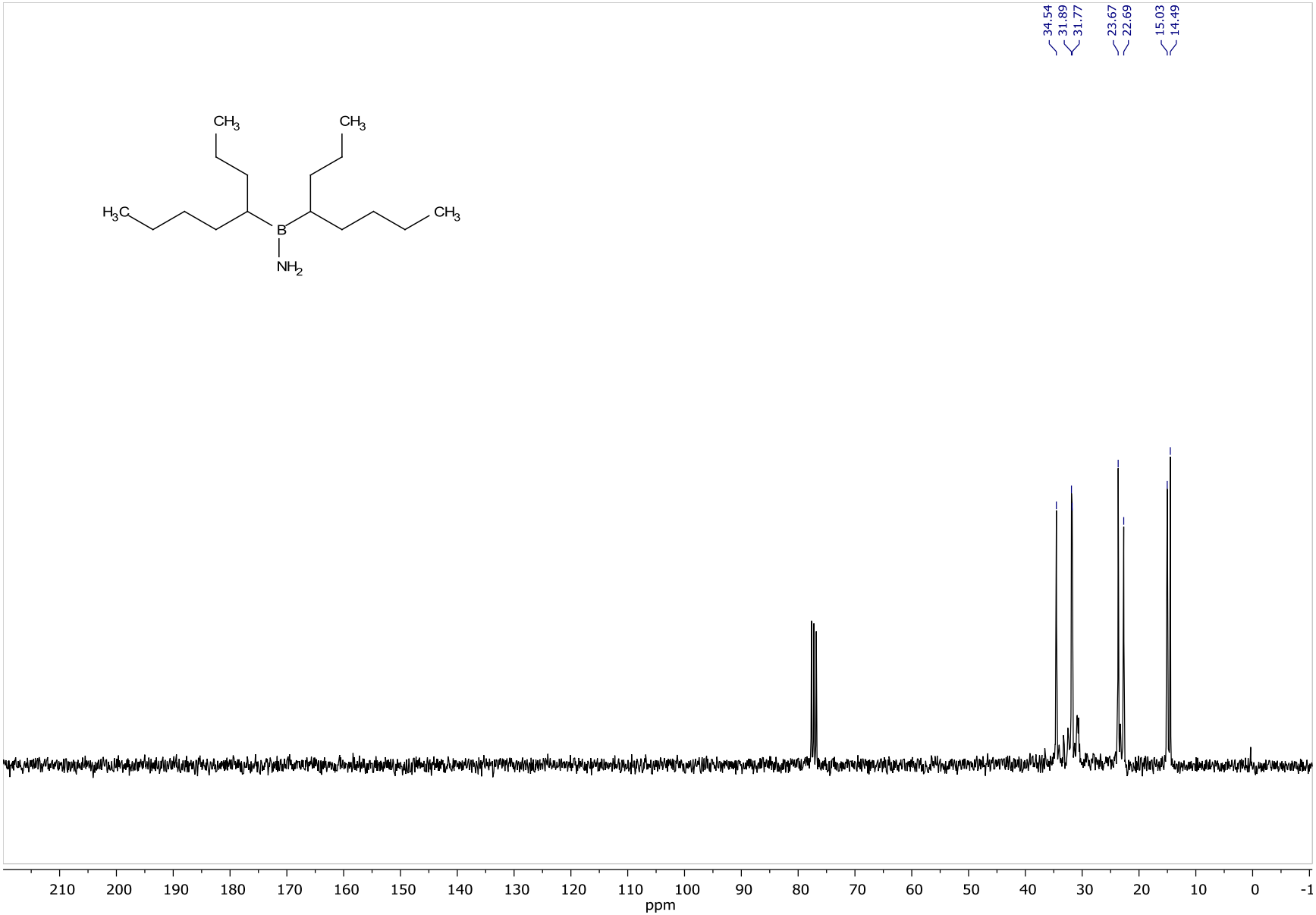
^{1}H NMR of **4l** (300 MHz, $CDCl_3$)



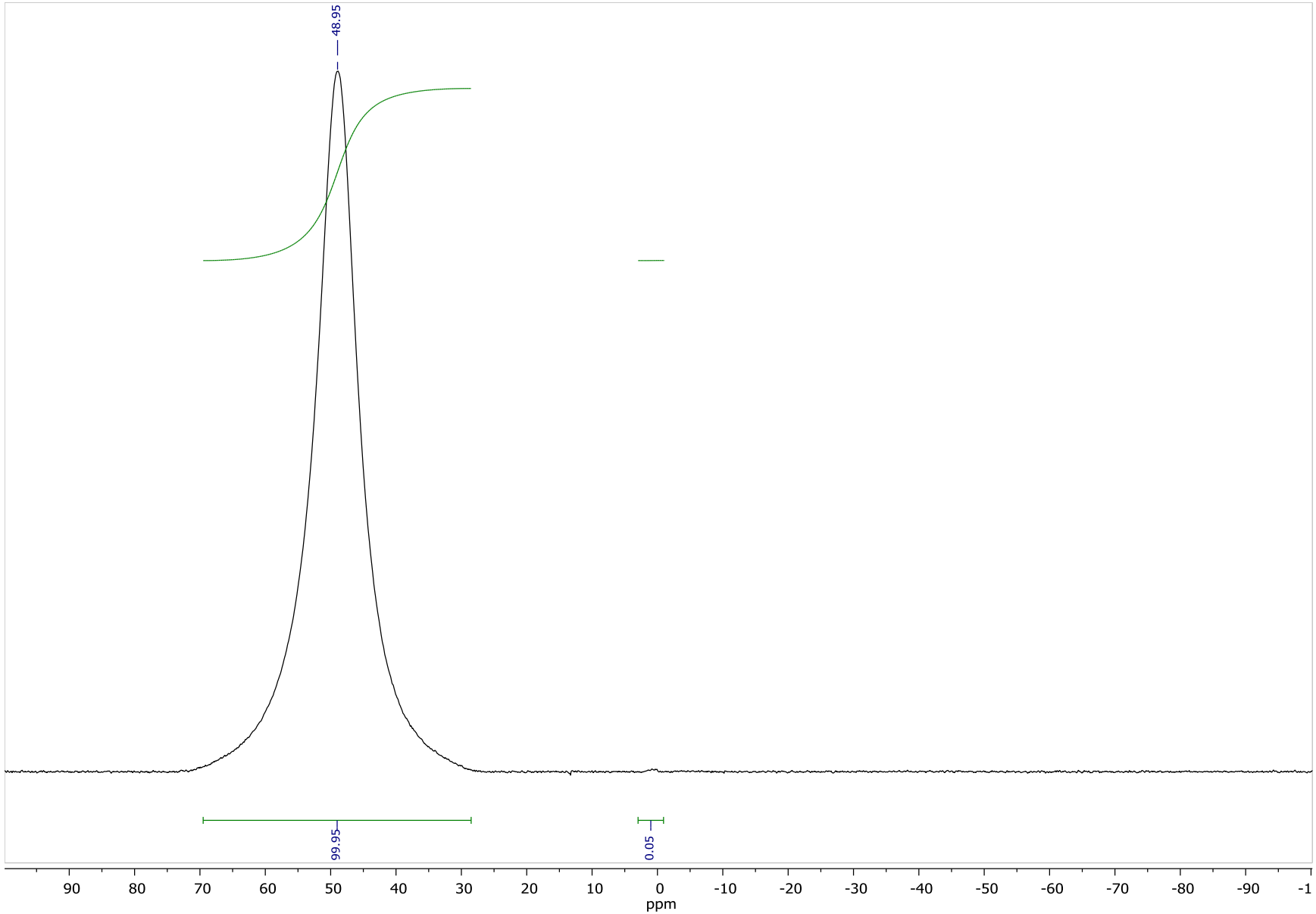
^{13}C NMR of **4l** (75 MHz, $CDCl_3$)



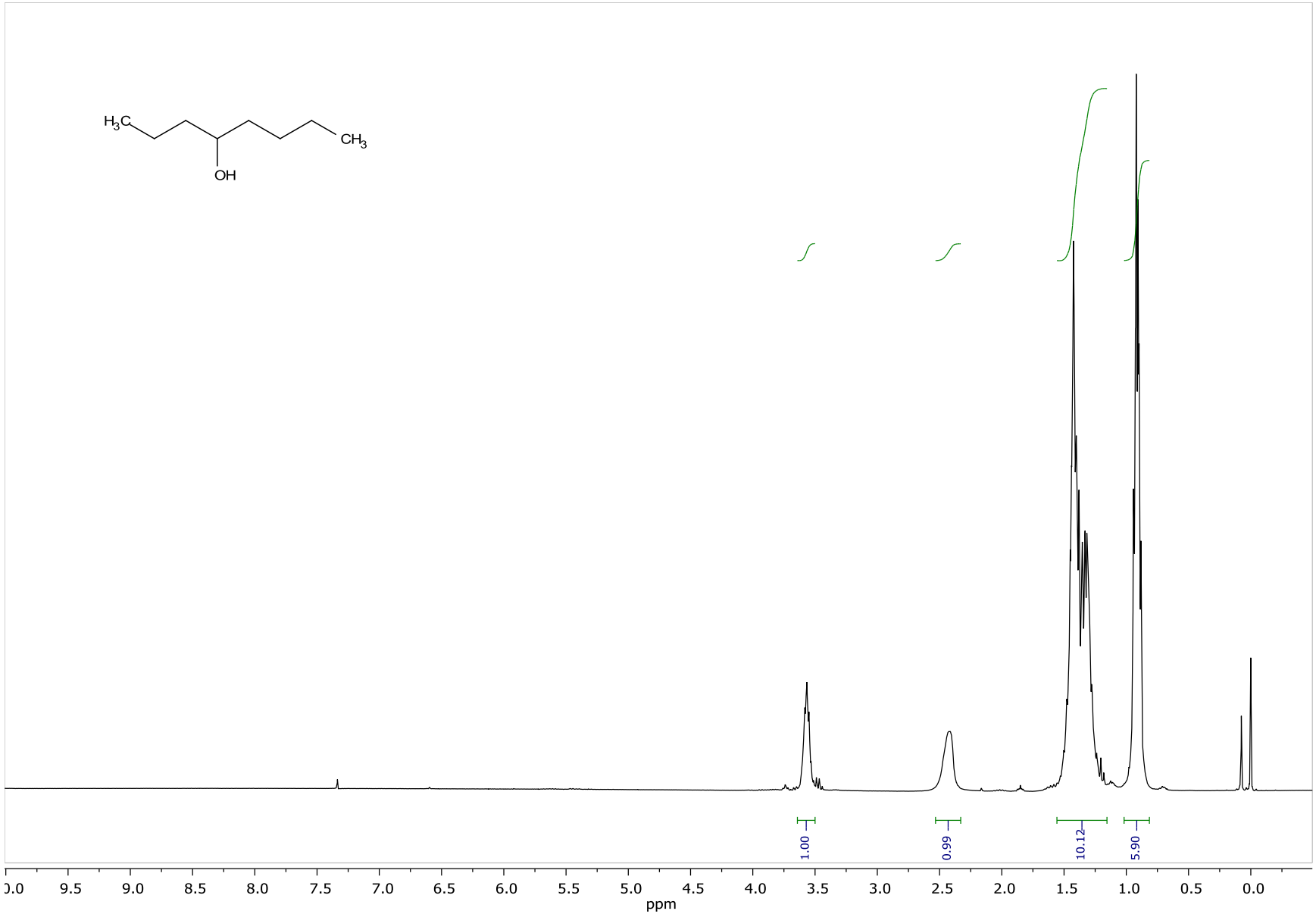
^{1}H NMR of **5m** (300 MHz, $CDCl_3$)



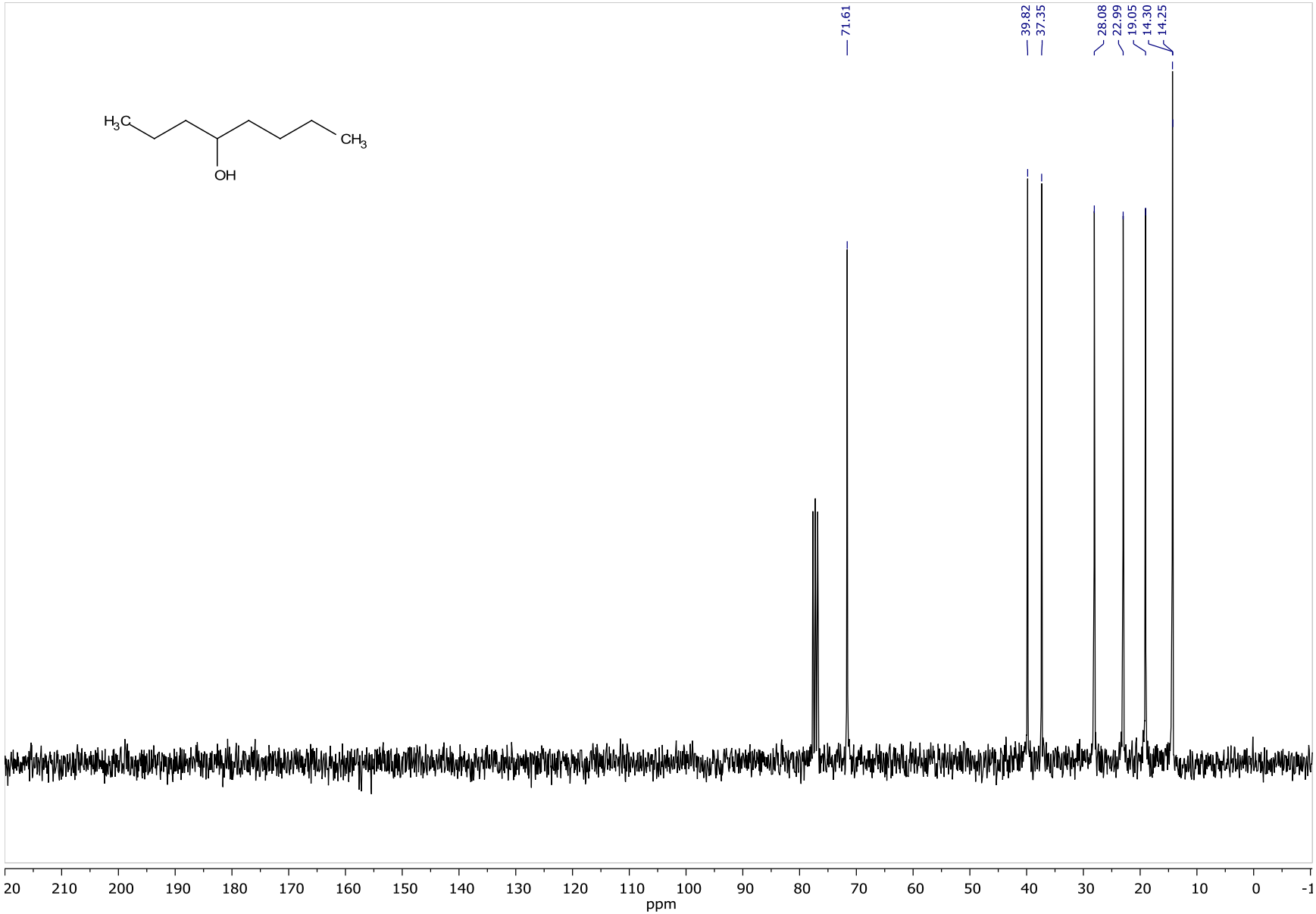
^{13}C NMR of **5m** (75 MHz, $CDCl_3$)



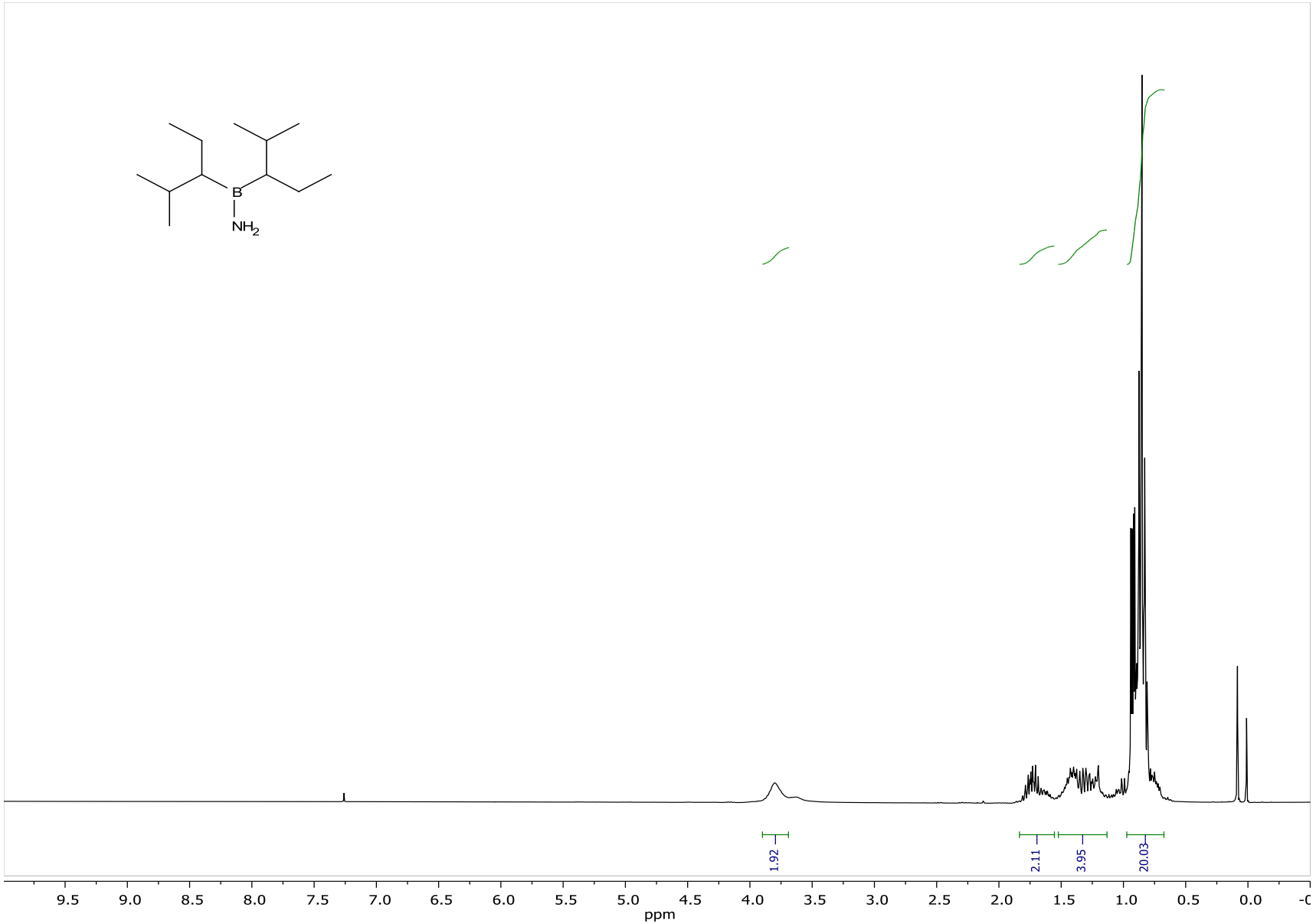
^{11}B NMR of **5m** (96 MHz, $CDCl_3$)



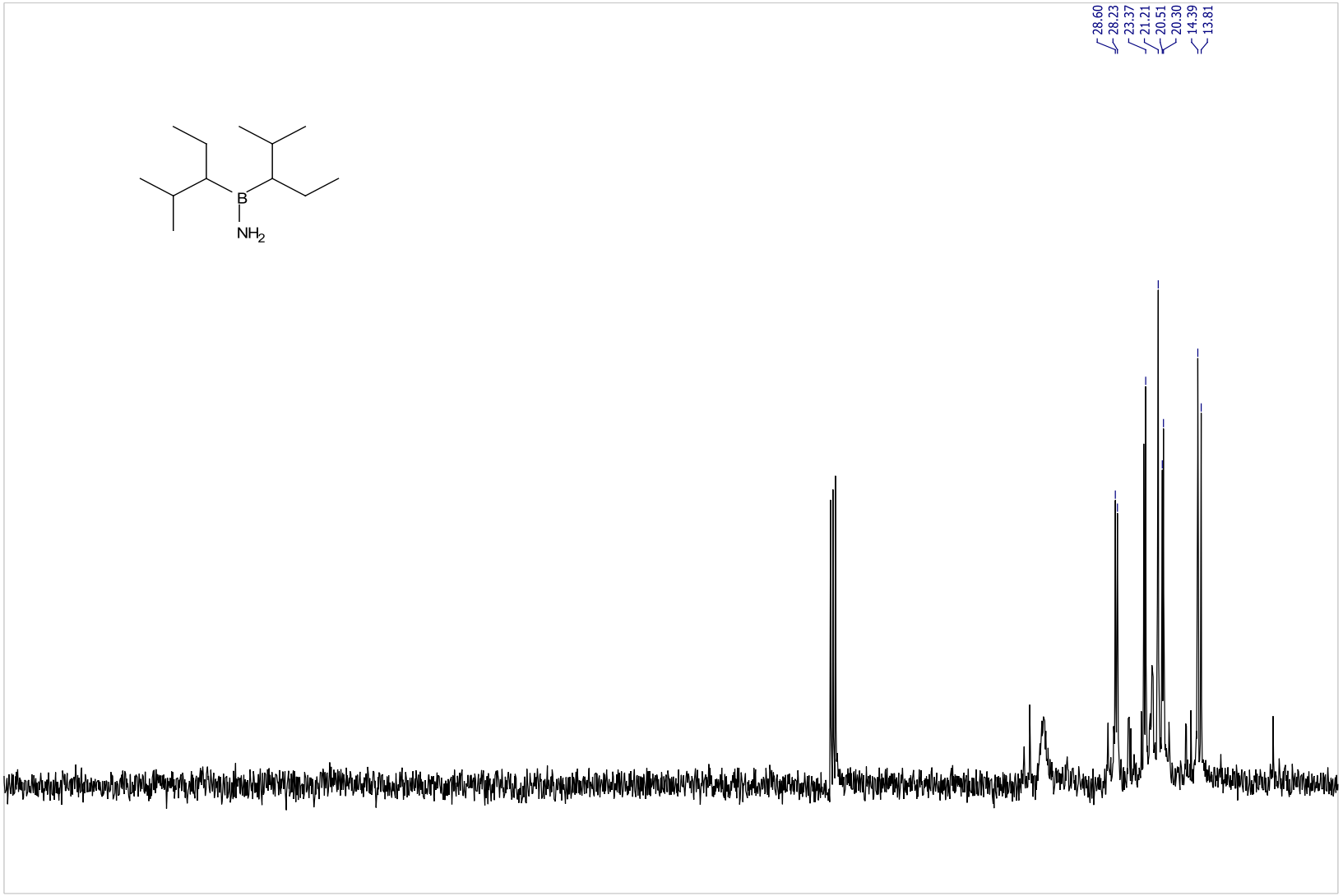
^{1}H NMR of **4m**(300 MHz, $CDCl_3$)



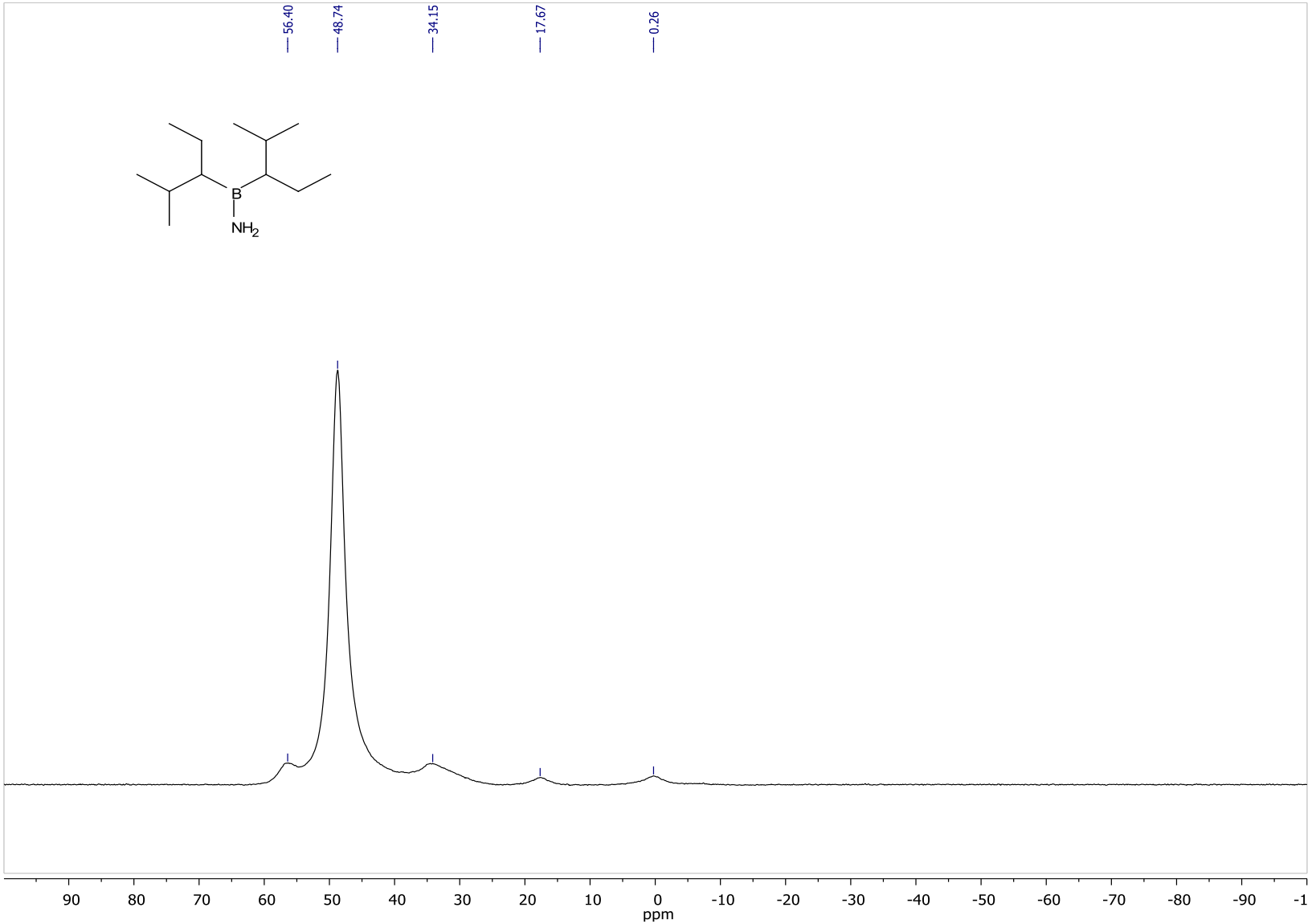
^{13}C NMR of **4m** (75 MHz, $CDCl_3$)



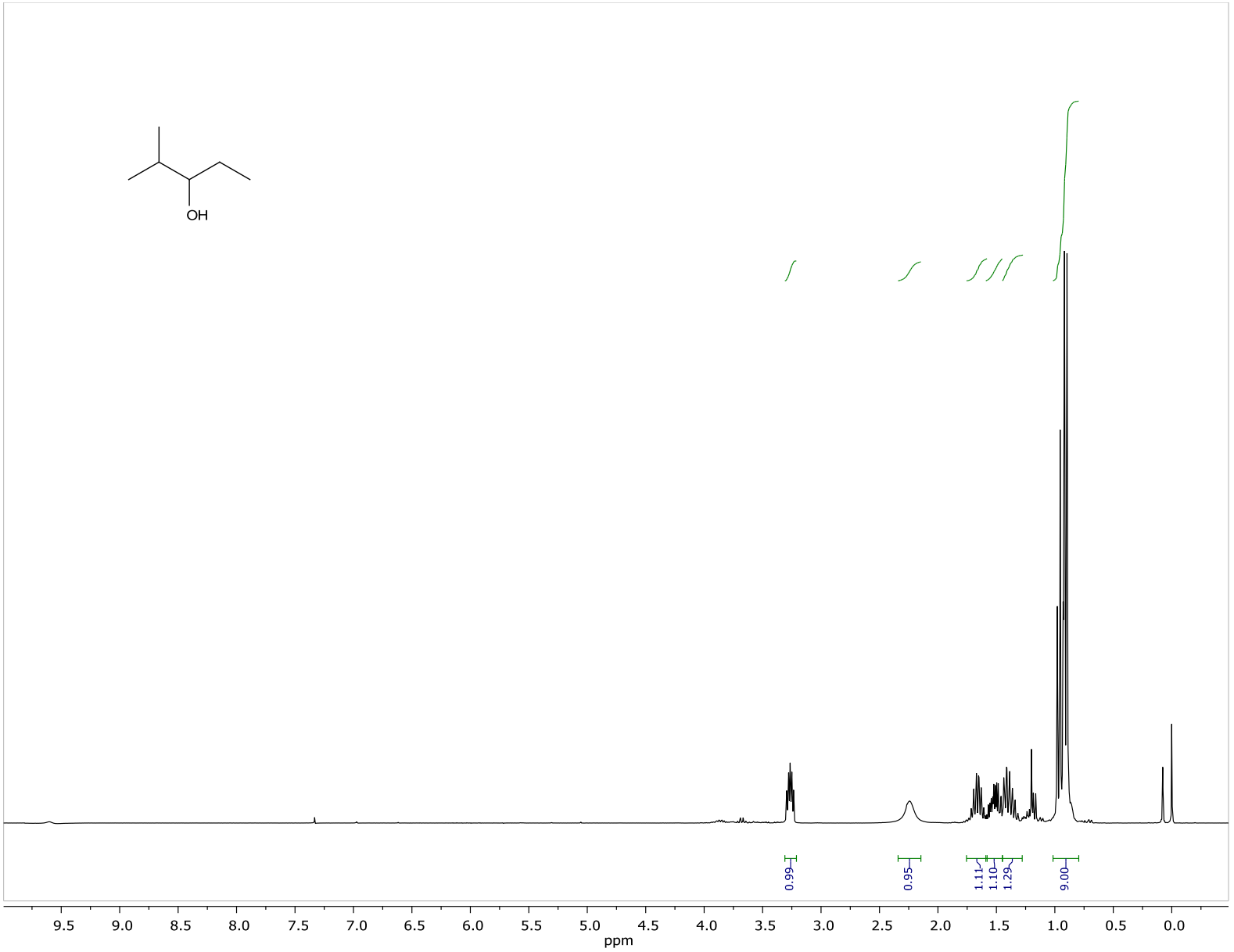
^{1}H NMR of **5n** (300 MHz, $CDCl_3$)



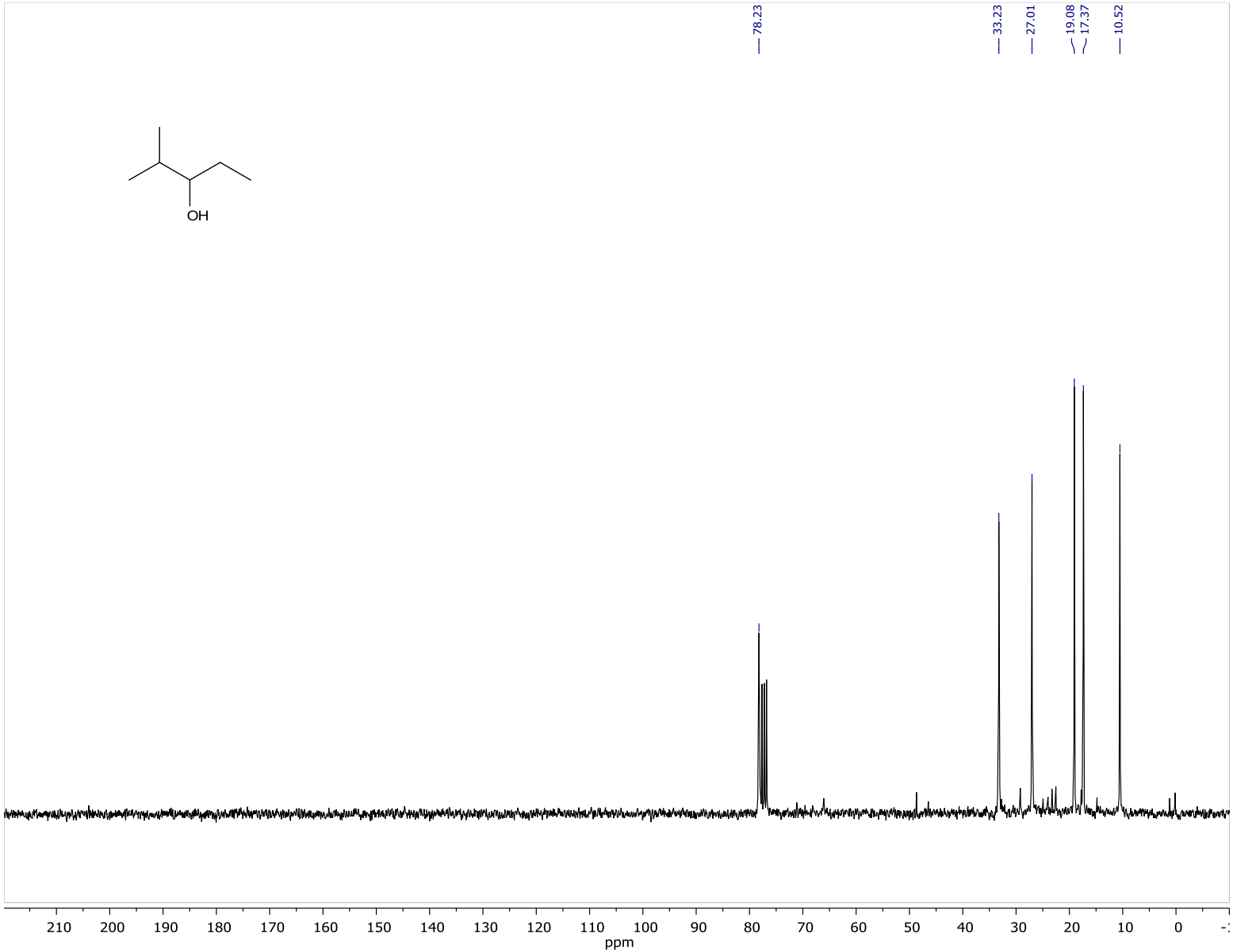
^{13}C NMR of **5n** (300 MHz, $CDCl_3$)



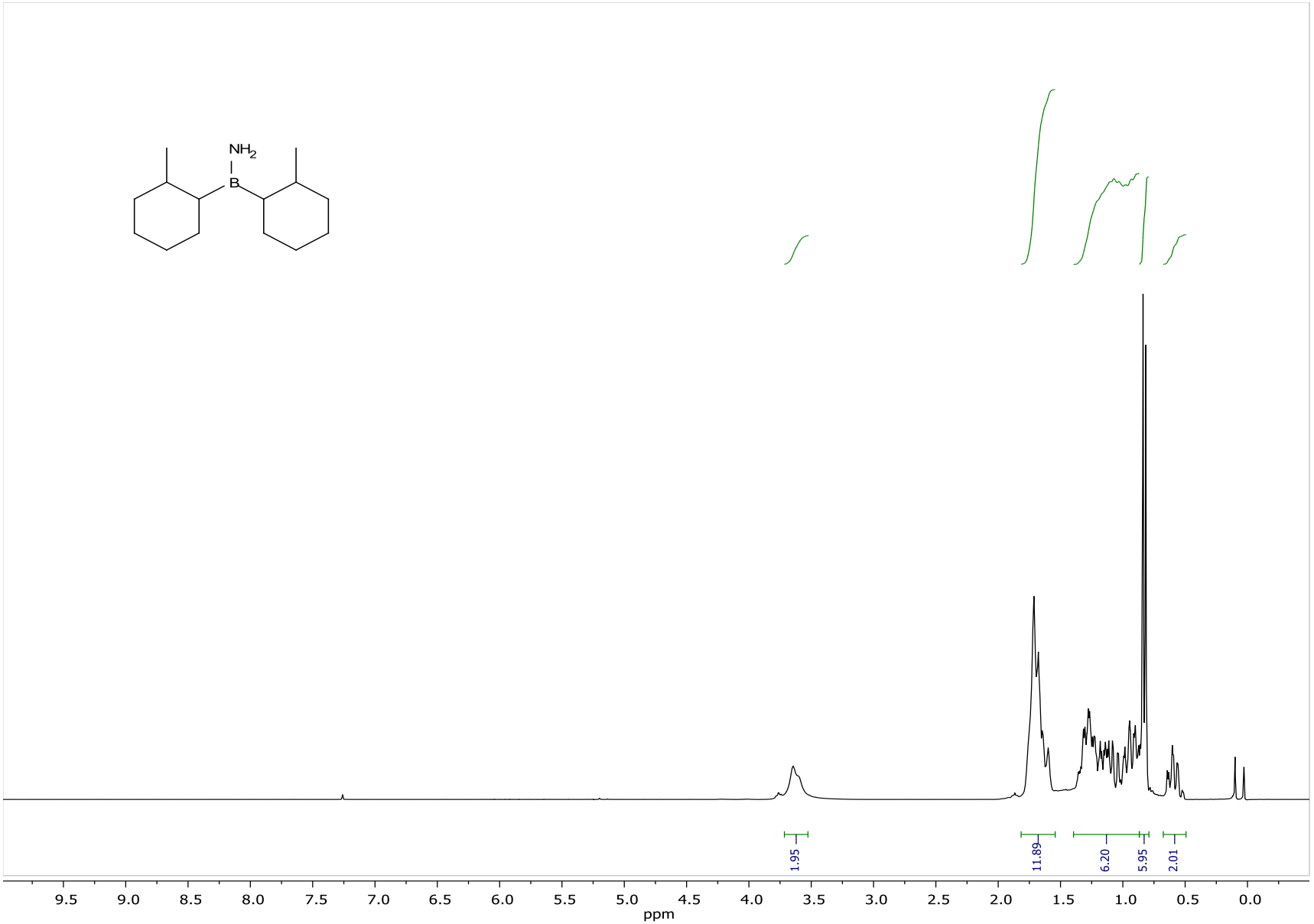
^{11}B NMR of **5n** (96 MHz, $CDCl_3$)



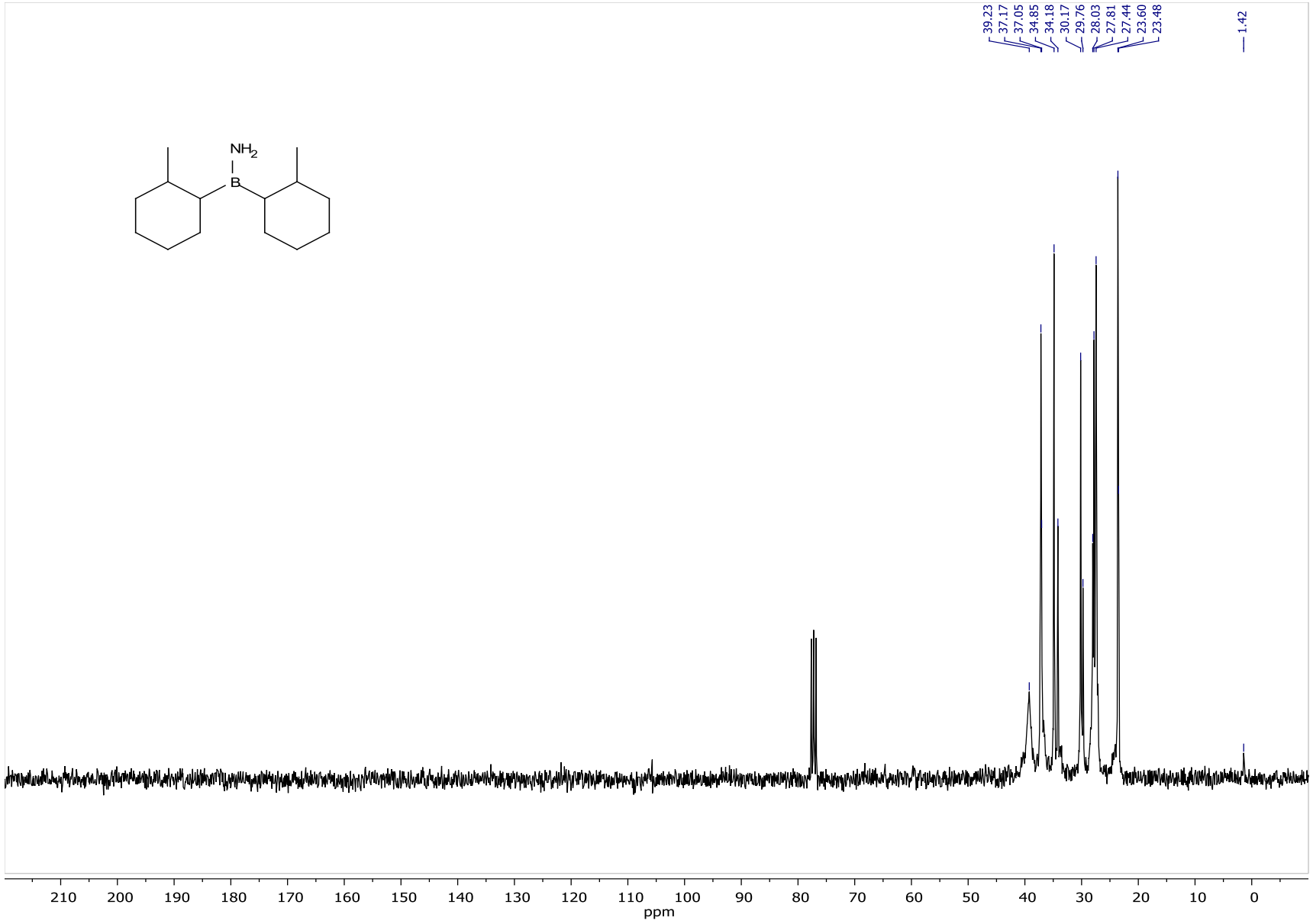
^{1}H NMR of **4n** (300 MHz, $CDCl_3$)



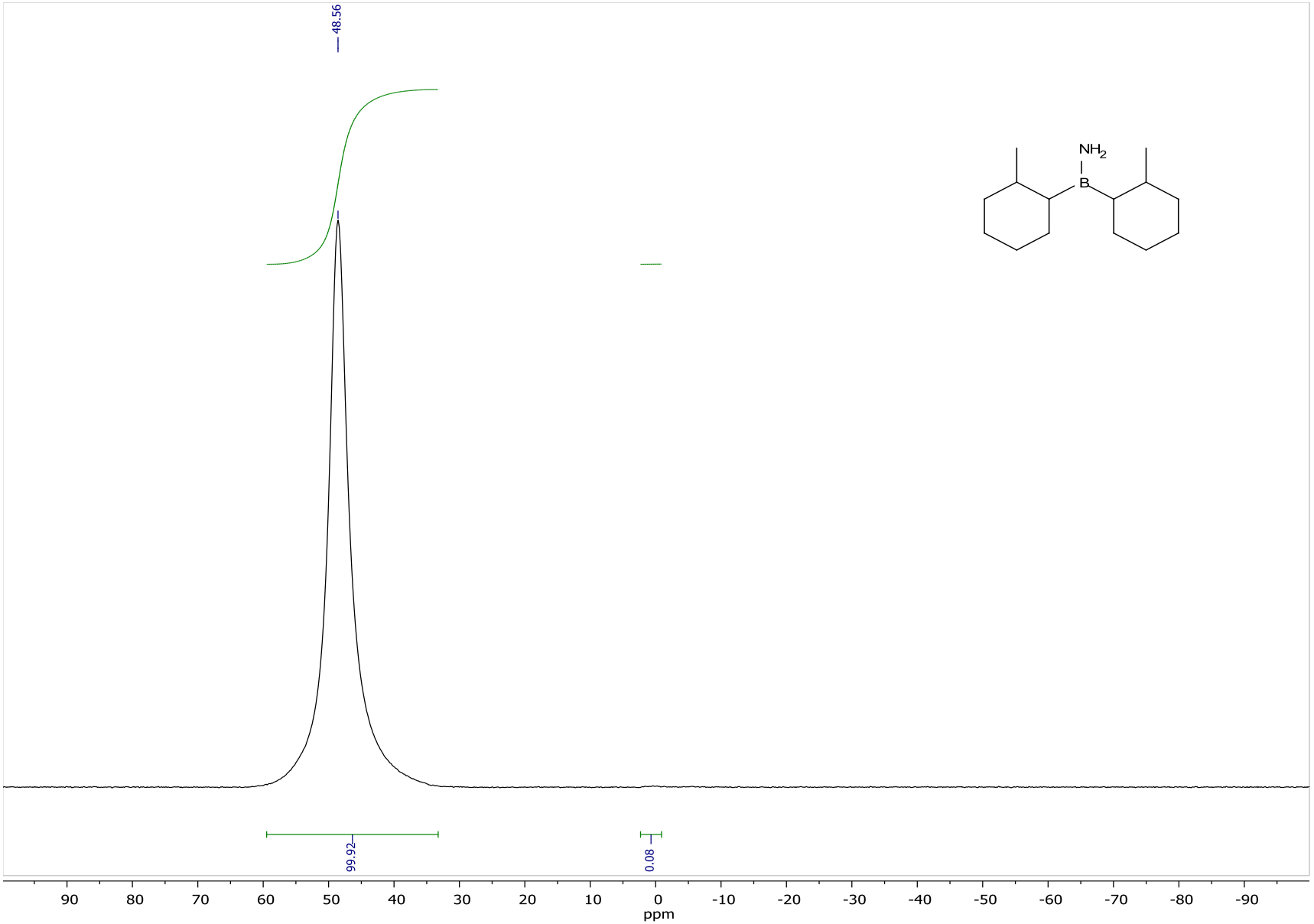
^{13}C NMR of **4n** (300 MHz, $CDCl_3$)



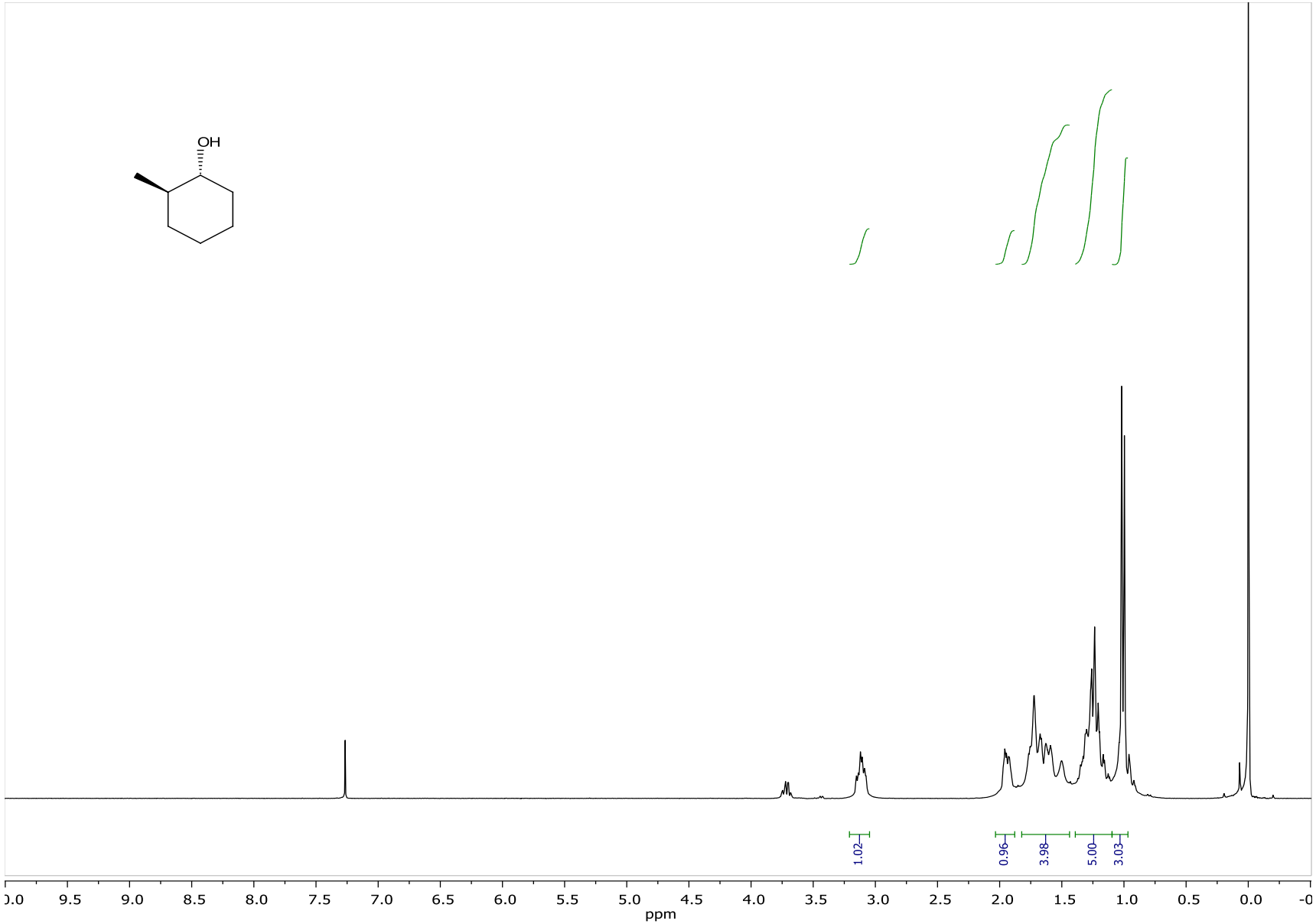
^{1}H NMR of **5o** (300 MHz, $CDCl_3$)



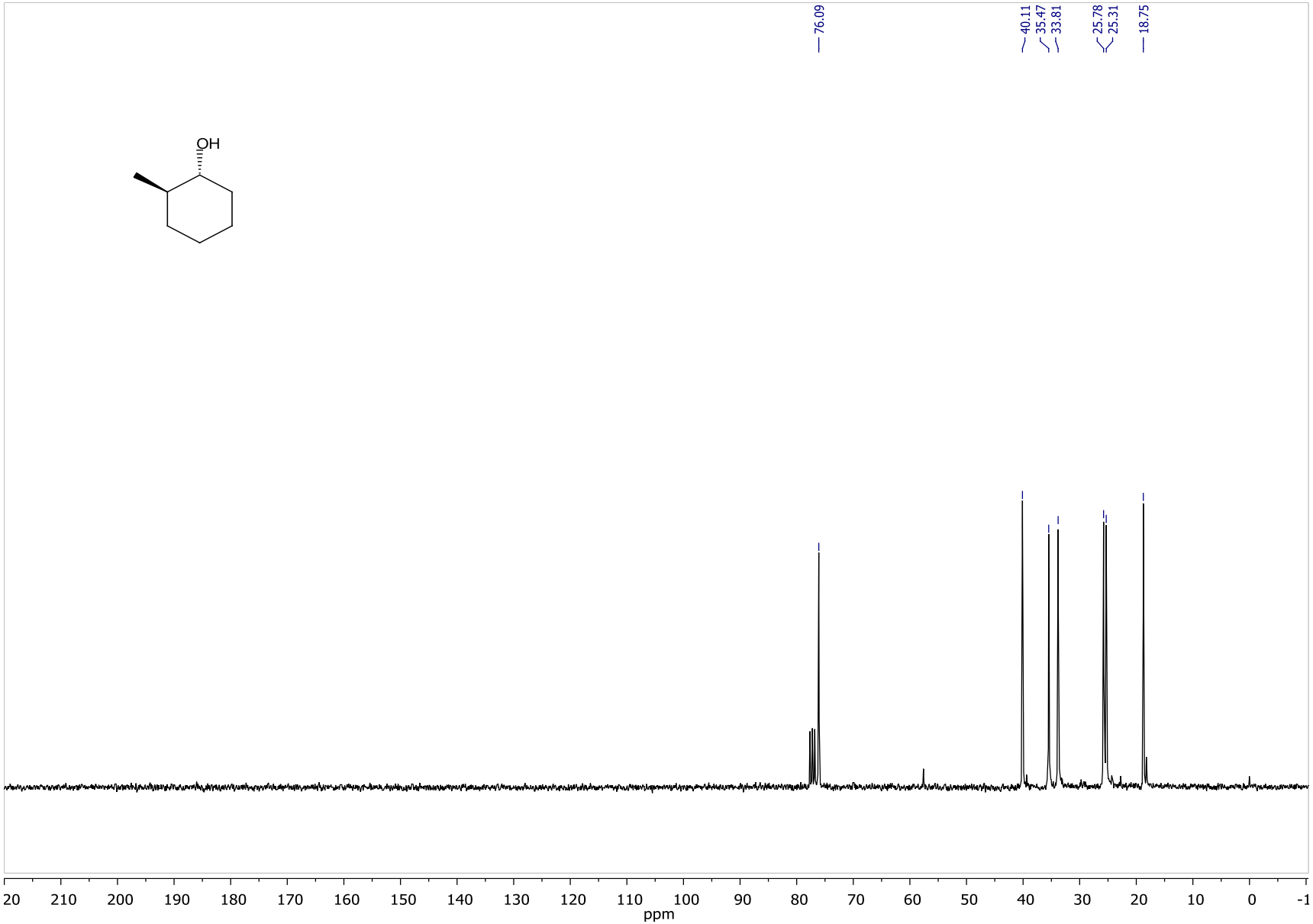
^{13}C NMR of **5o** (75 MHz, $CDCl_3$)



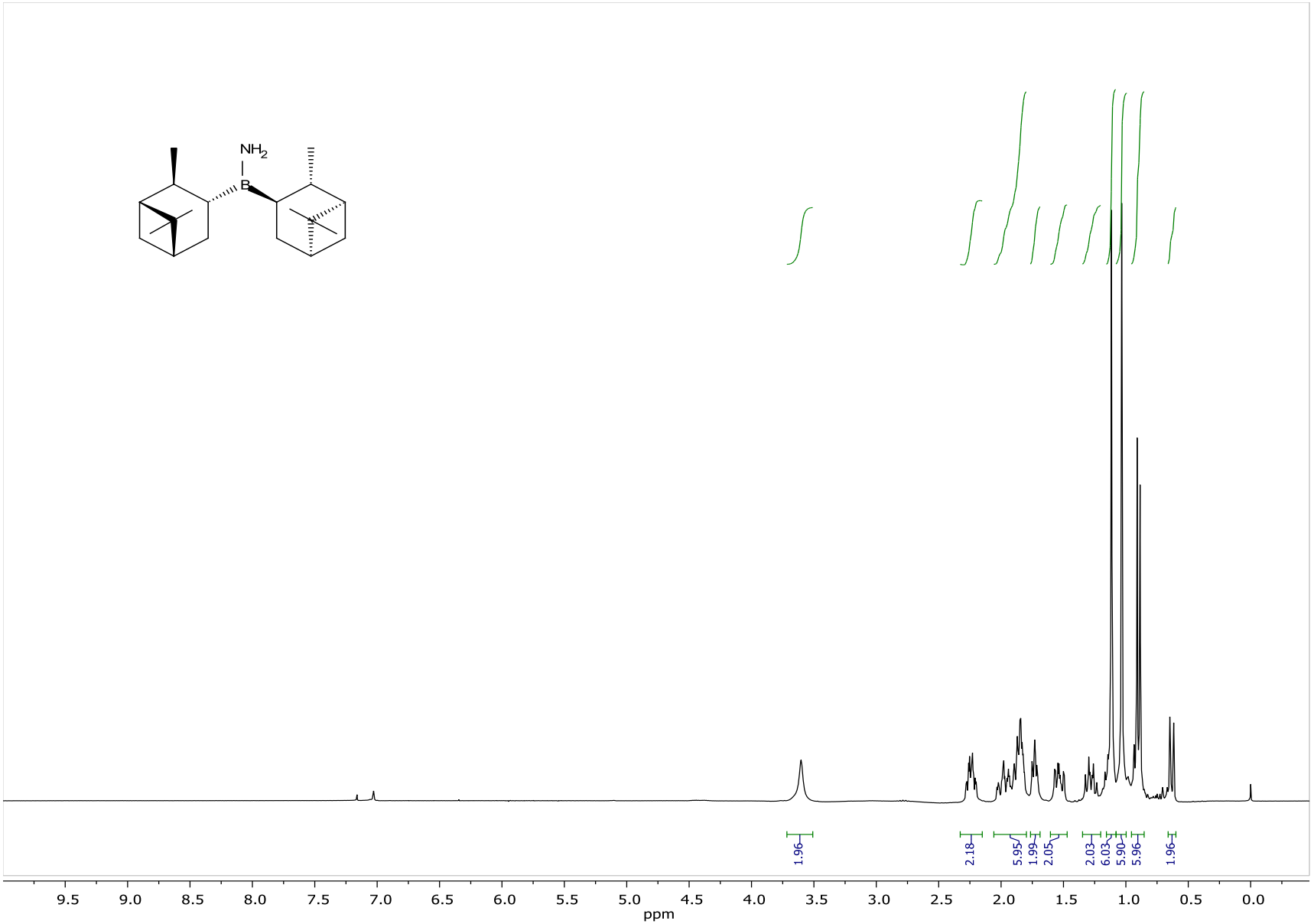
^{11}B NMR of **5o** (96 MHz, $CDCl_3$)



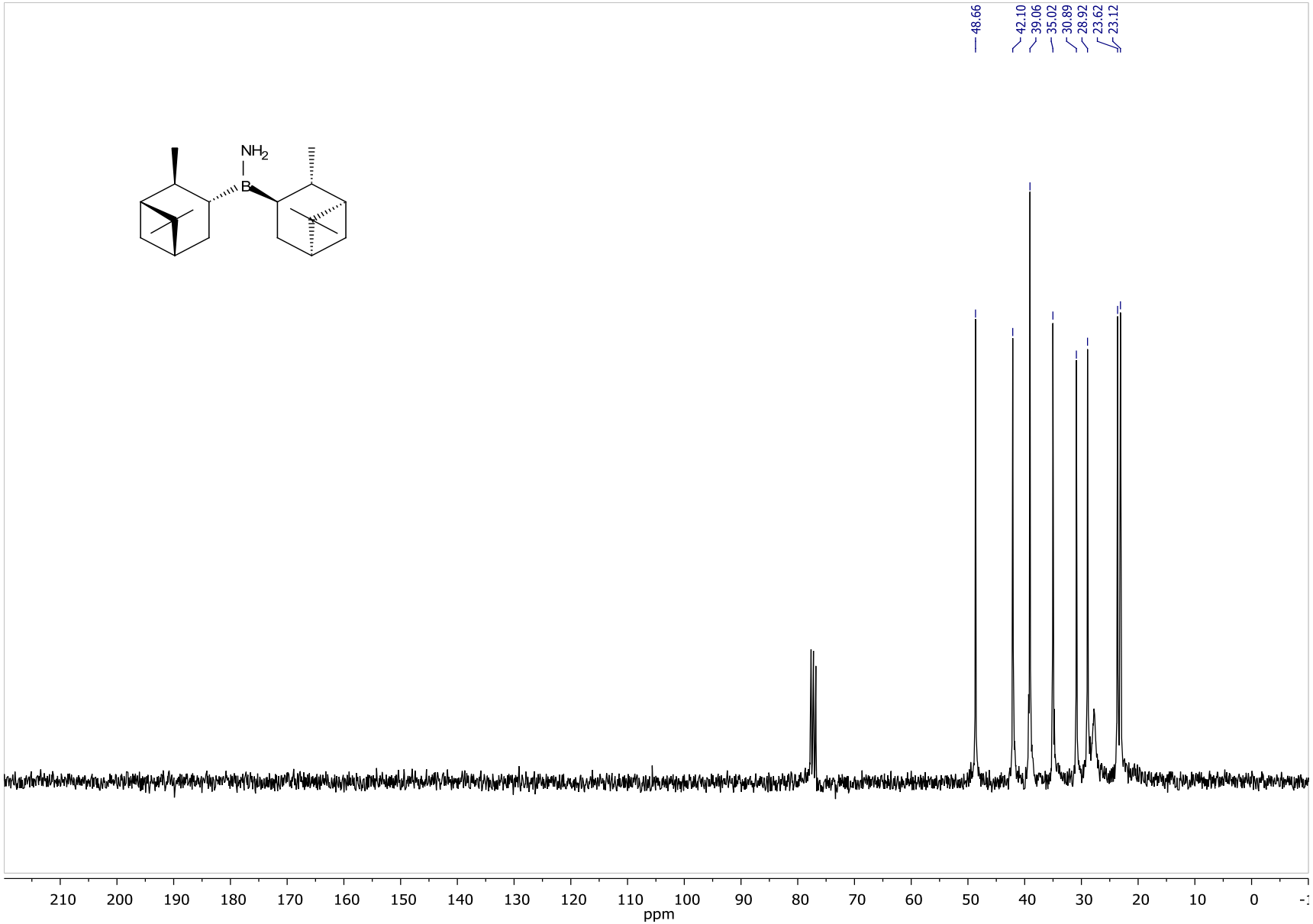
^{1}H NMR of **4o** (300 MHz, $CDCl_3$)



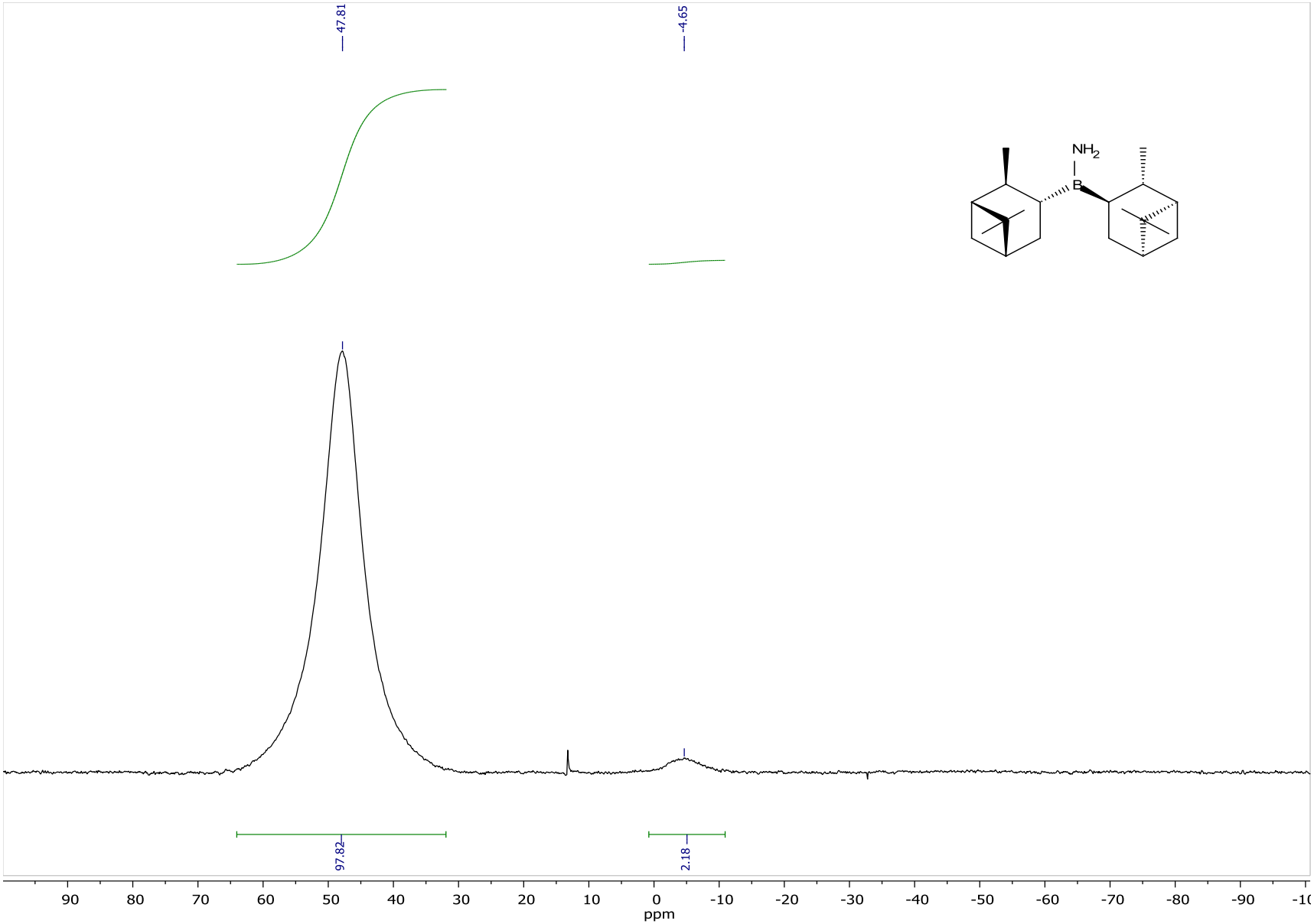
^{13}C NMR of **4o** (75 MHz, $CDCl_3$)



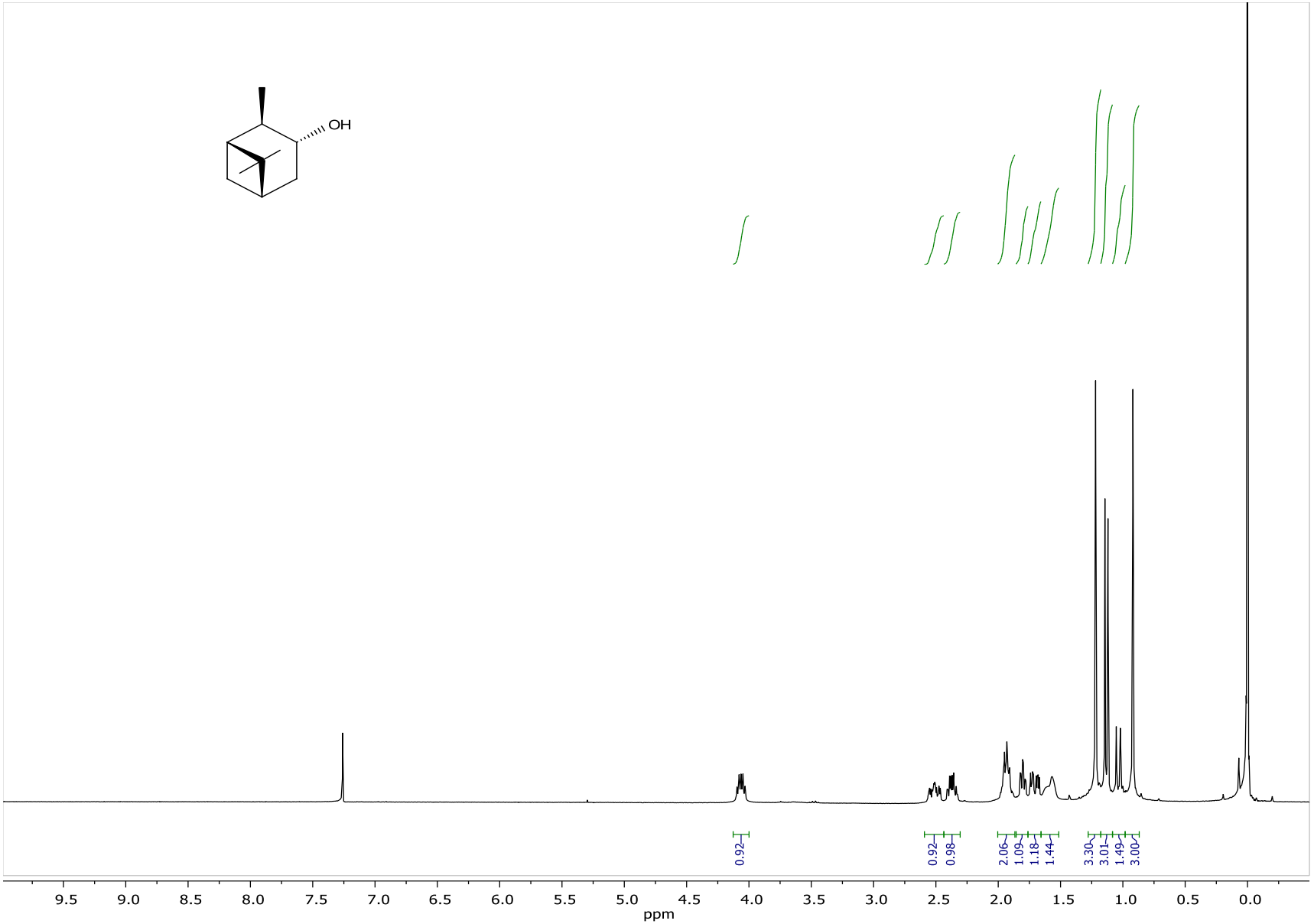
^{1}H NMR of **5p** (300 MHz, $CDCl_3$)



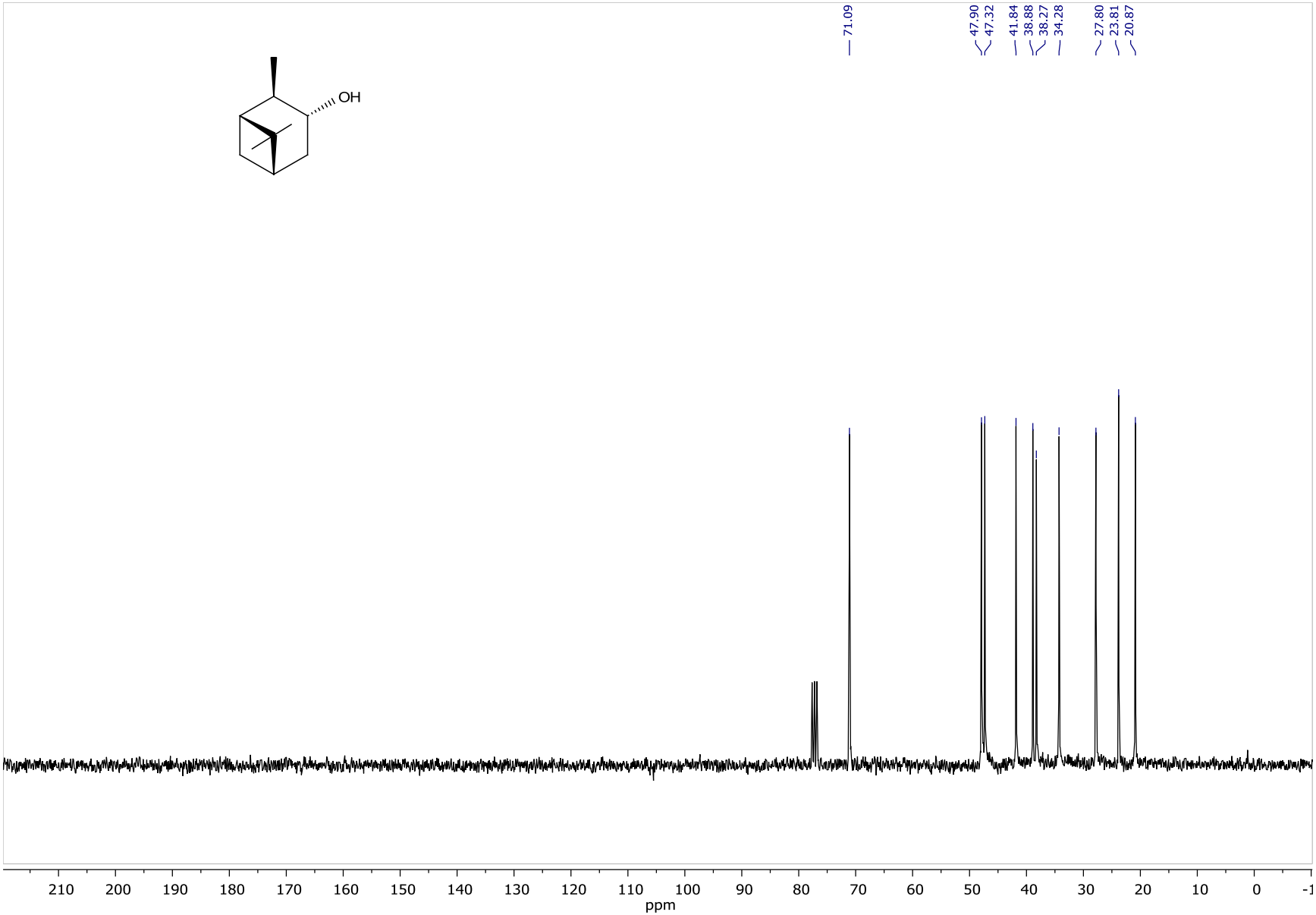
^{13}C NMR of **5p** (75 MHz, $CDCl_3$)



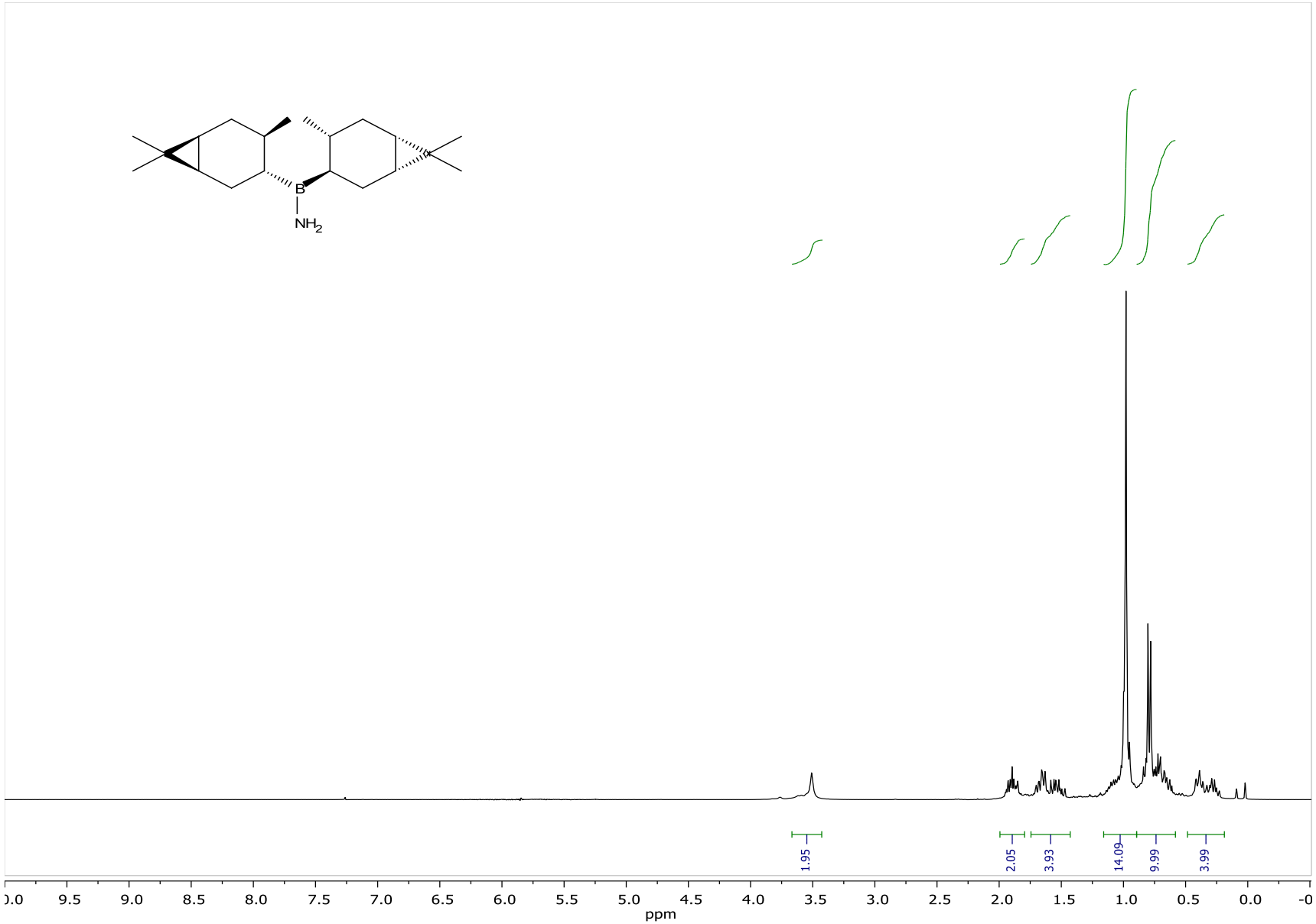
^{11}B NMR of **5p** (96 MHz, $CDCl_3$)



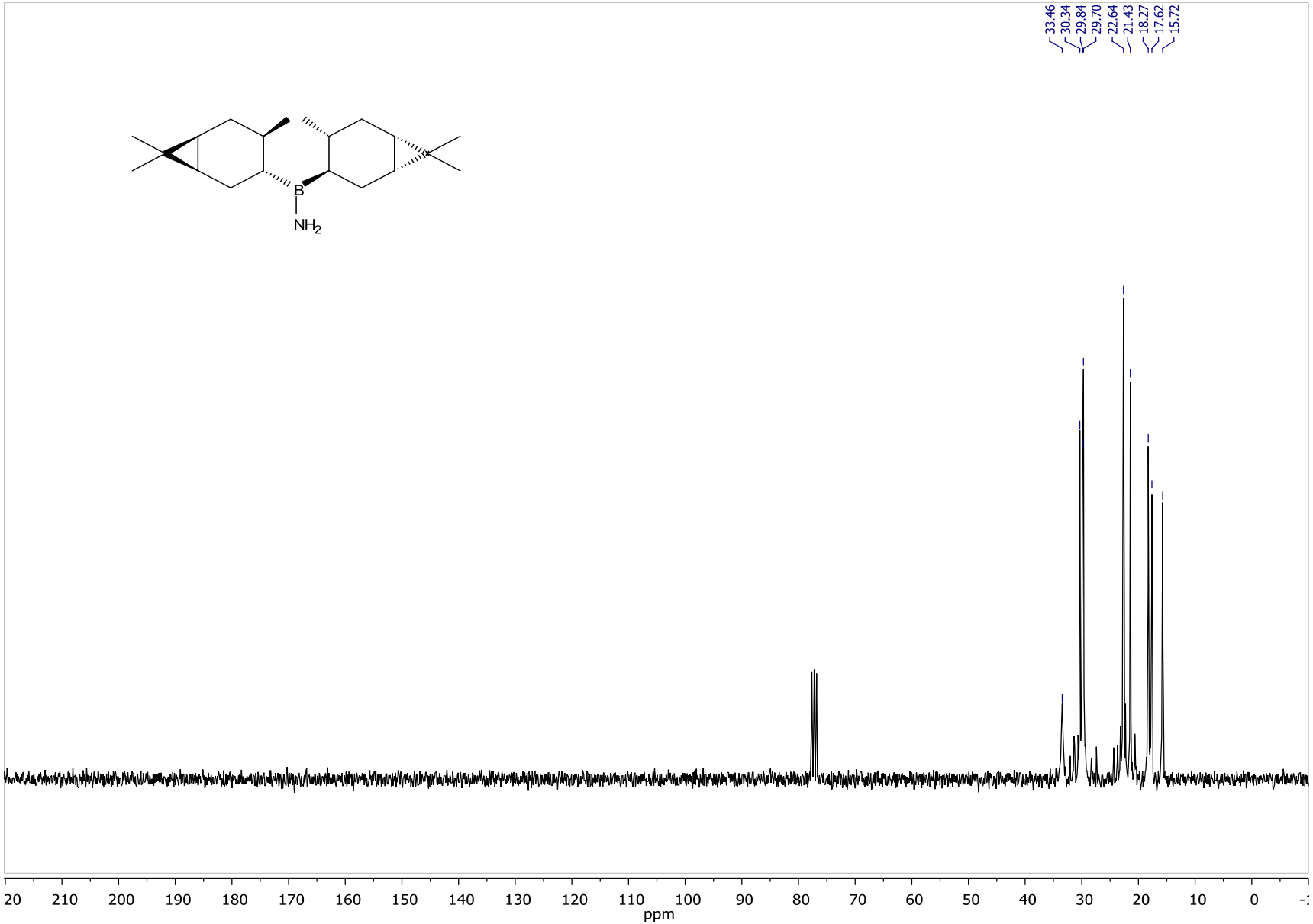
^{1}H NMR of **4p** (300 MHz, $CDCl_3$)



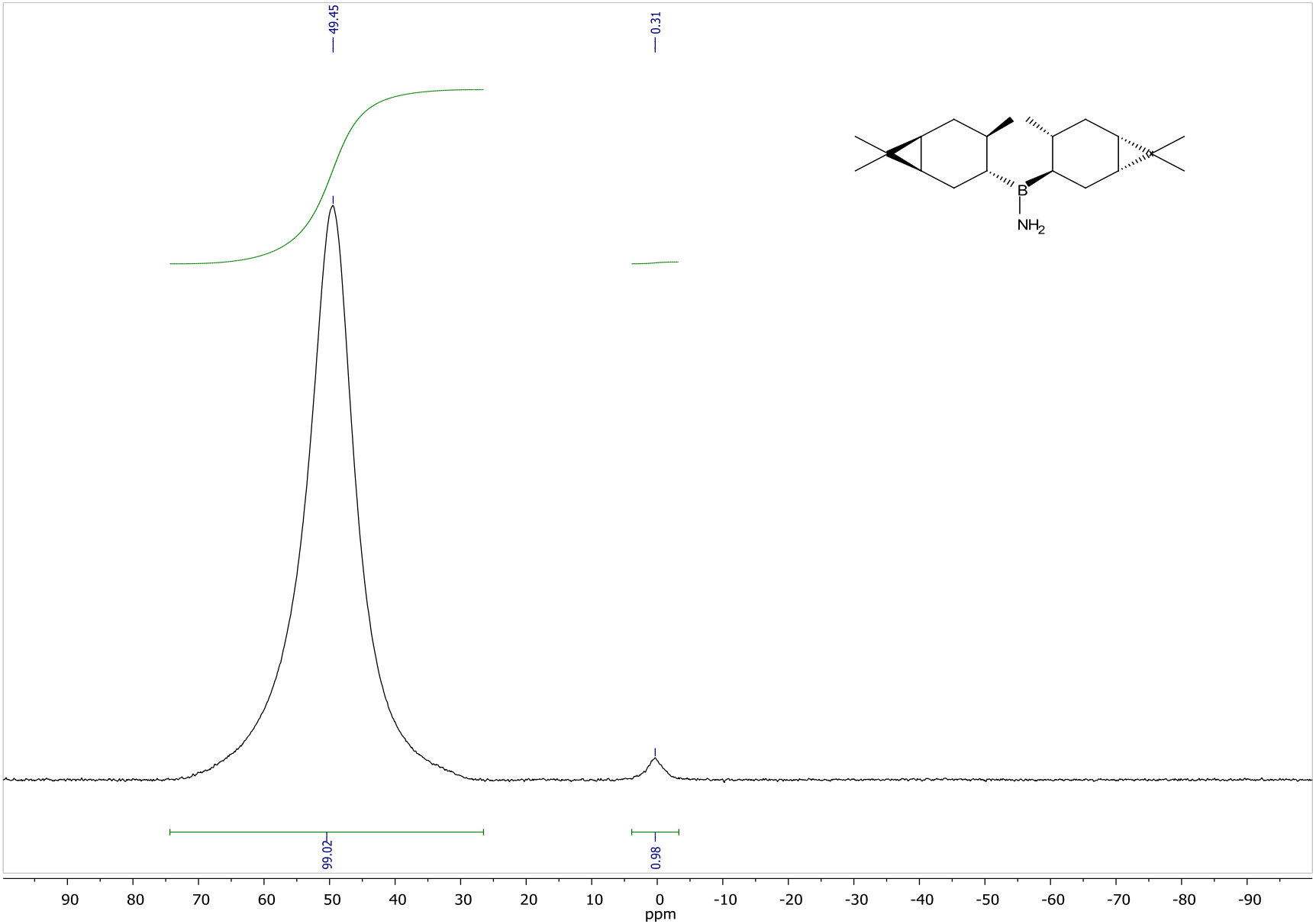
^{13}C NMR of **4p** (75 MHz, $CDCl_3$)



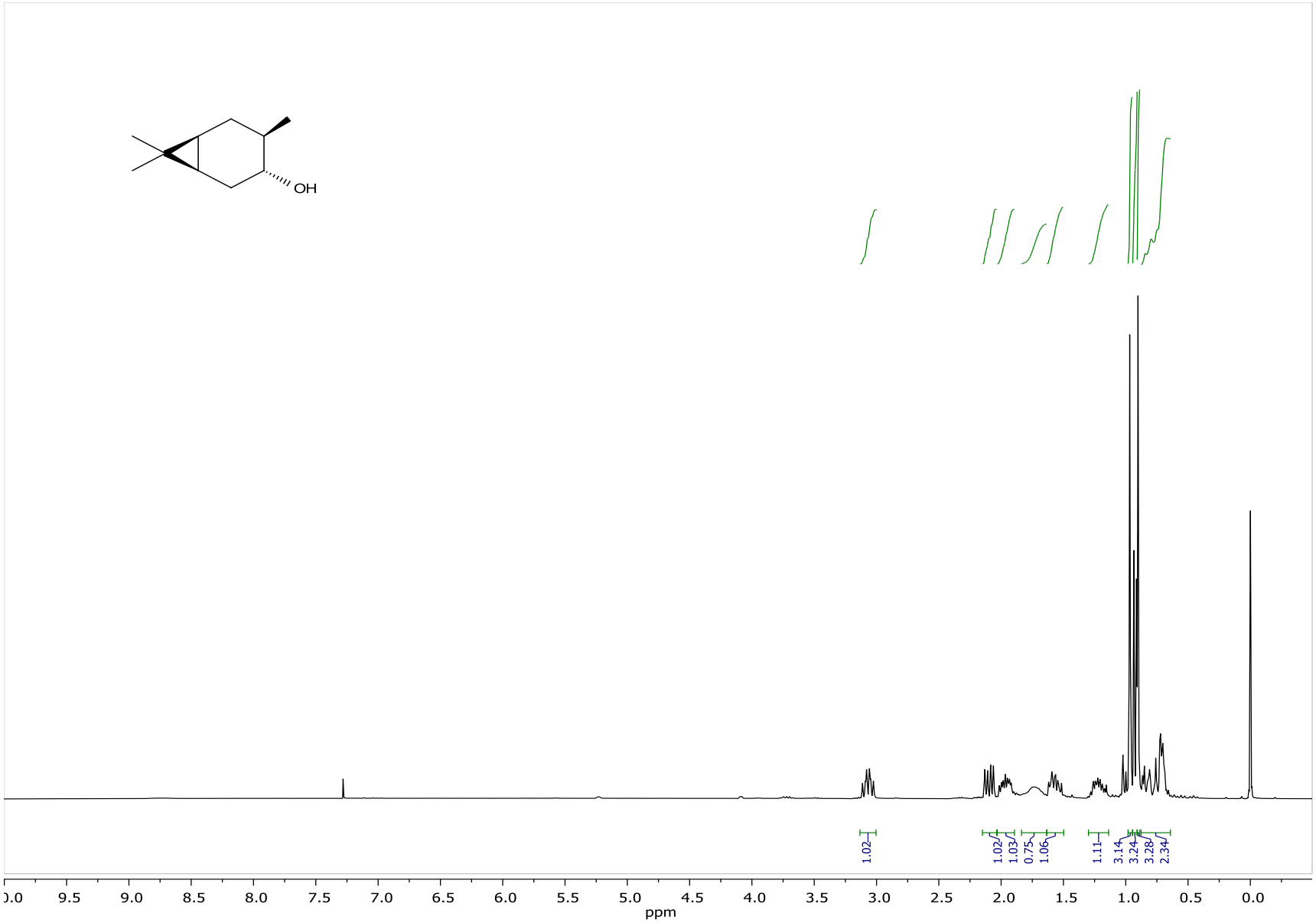
^{1}H NMR of **5q** (300 MHz, $CDCl_3$)



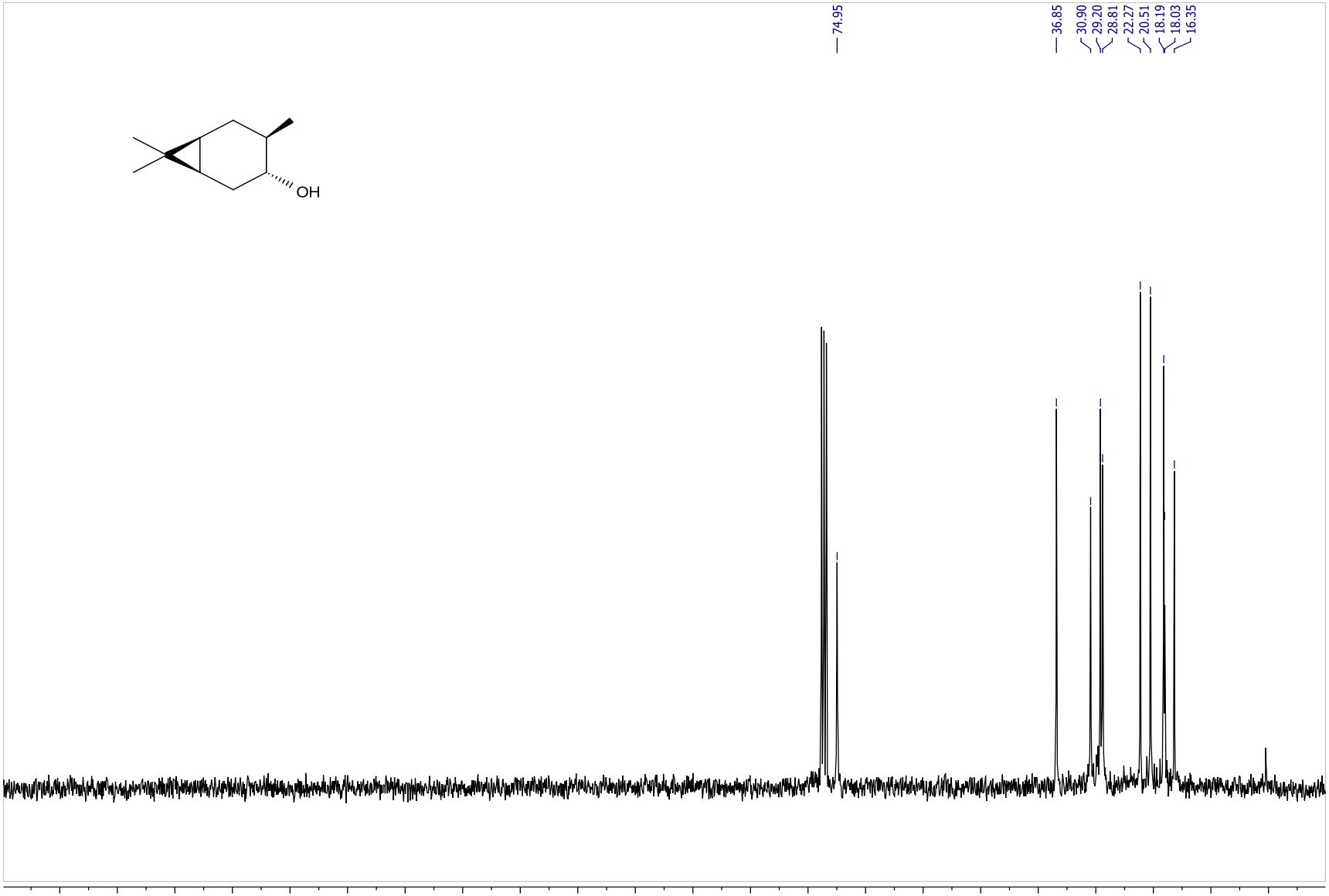
^{13}C NMR of **5q** (75 MHz, $CDCl_3$)



^{11}B NMR of **5q** (96 MHz, $CDCl_3$)



^{1}H NMR of **4q** (300 MHz, $CDCl_3$)



^{13}C NMR of **4q** (75 MHz, $CDCl_3$)