

Electronic Supplementary Information (ESI)

Protecting-Group-Free Synthesis of Hydroxyesters from Amino Alcohols

Guillaume Reynard, Eve-Marline Joseph-Valcin and H el ene Lebel*.

*Department of Chemistry and Center in Green Chemistry and Catalysis (CGCC), Universit e de
Montr eal, P.O. Box 6128, Station Downtown, Montr eal, QC H3C 3J7, Canada*

Table of Contents

General	2
Synthesis of Nitrite Reagents	2
Figure S1. DSC-TGA of 1,3-(2,2-dimethyl)propanedinitrite	4
General Procedures	4
Characterization Data for Esters	5
NMR Spectra	18

General

Analytical thin-layer chromatography was performed using 0.25 mm silica gel 60-F plates. Visualization of the developed chromatogram was performed by UV absorbance, cerium ammonium molybdate, or aqueous potassium permanganate. Flash chromatography was performed using silica gel (230–400 mesh) with the indicated solvent system. Infrared spectra are reported in reciprocal centimeters (cm^{-1}). Only the most important and relevant frequencies are reported. Chemical shifts for ^1H NMR spectra were recorded in parts per million with the solvent resonance as the reference CDCl_3 ($\delta = 7.26$ ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qn = quintuplet, sept = septet, m = multiplet and br = broad), coupling constant in Hz and integration. Chemical shifts for ^{13}C NMR spectra are recorded in parts per million using the central peak of CDCl_3 ($\delta = 77.16$ ppm) as the reference. Chemical shifts for ^{19}F NMR spectra are recorded in parts per million using trifluorotoluene ($\delta = -63.72$ ppm) as an external reference. All ^{13}C NMR and ^{19}F NMR spectra were obtained with complete proton decoupling. All NMR yields were determined using quantitative ^1H NMR spectra using tetrachloroethane as an internal standard with a 10-sec relaxation time. High resolution mass spectra analysis was performed by the Centre régional de spectroscopie de masse de l'Université de Montréal.

Synthesis of Nitrite Reagents

1,3-Propanedinitrite. 1,3-Propanedinitrite was prepared from 1,3-propanediol, sodium nitrite and sulfuric acid according to the literature procedure.¹ We have never experienced any spontaneous explosion of this material, when heating 1,3-propanedinitrite in solution in chloroform or toluene. 95% of 1,3-propanedinitrite was recovered after heating a toluene solution of the reagent for 24 hours at 100 °C, whereas 68% of 1,3-propanedinitrite was recovered after refluxing in chloroform for 24 hours. The DSC-TGA spectra of 1,3-propanedinitrite in non-sealed crucible revealed a slow evaporation and no exotherm.¹ The DSC-TGA spectra of 1,3-propanedinitrite in a sealed stainless-steel crucible revealed an important, strong exothermic event from 122 to 155 °C, with an important loss of mass and a significant modification of temperature setpoint, consistent with a decomposition.² Heating 1,3-propanedinitrite concentrated or neat is thus *not recommended*, as autocatalytic decomposition may occur and overpressure might develop, due to the formation of gases. Distillation should also be avoided. To inform safe processing conditions, thermal stability evaluation is also recommended when synthesizing similar nitrite compounds.

¹ C. Audubert and H. Lebel, *Org. Lett.*, 2017, **19**, 4407-4410.

² G. Reynard, H. Mayrand and H. Lebel, *Can. J. Chem.*, 2020, DOI: 10.1139/cjc-2020-0028.

1,3-(2,2-Dimethyl)propanedinitrite.

In a 100 mL round bottom flask with magnetic stirring bar, sodium nitrite (16.6 g, 240 mmol, 2.40 equiv) was dissolved in H₂O (30 mL) under ambient atmosphere. 2,2-dimethyl-1,3-propanediol (10.4 g, 100 mmol, 1.00 equiv) was added and the solution was cooled to 0 °C. A solution of H₂SO₄ (12.8 mL, 240 mmol, 2.40 equiv) diluted in H₂O (30 mL) was added dropwise with an addition funnel over a period of 30 min. The reaction was stirred 60 min at 0 °C after the end of addition. The reaction was transferred in a separatory funnel and brine (30 mL) was added. The two layers were separated, and the organic layer was washed with brine, then dried over Na₂SO₄. The title compound was isolated as a yellow liquid (15.9 g, 93.9 mmol, 98% yield). 2,2-dimethyl-1,3-propyldinitrite was stored on Na₂SO₄ at rt during few months without significant decomposition. ¹H NMR (500 MHz, CDCl₃) δ 4.55 (s, 4H), 1.01 (s, 6H). The DSC-TGA spectra of 1,3-(2,2-dimethyl)propanedinitrite in a sealed stainless-steel crucible revealed an important, strong exothermic event from 124 to 174 °C, with an important loss of mass and a significant modification of temperature setpoint, consistent with a decomposition. Heating 1,3-(2,2-dimethyl)propanedinitrite concentrated or neat is thus *not recommended*, as autocatalytic decomposition may occur and overpressure might develop, due to the formation of gases. Distillation should also be avoided.

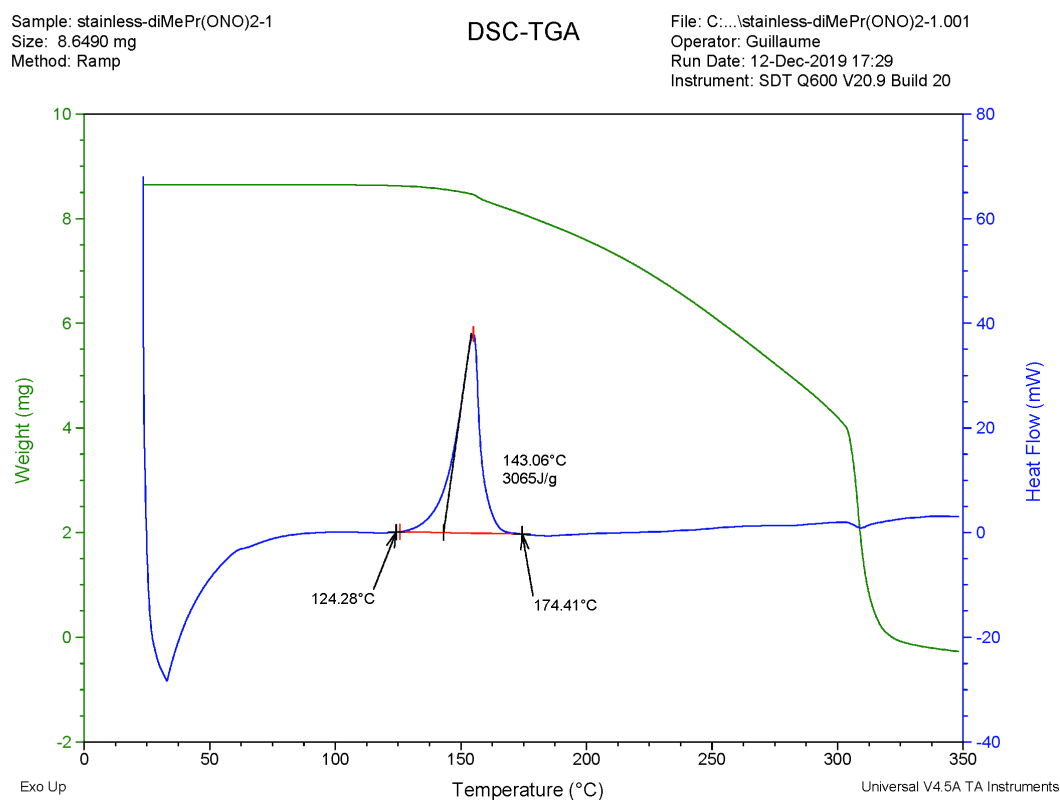


Figure S1. DSC-TGA of 1,3-(2,2-dimethyl)propanedinitrite

General Procedures

General Flow Procedure A for the Esterification of Carboxylic Acids with Amino alcohols

A 1 mL sample loop A was charged with a 2 M solution of the amino alcohol (2.00 mmol, 2.00 equiv) in THF. A 5 mL sample loop B was charged with a 0.2 M solution of the carboxylic acid (1.00 mmol) and a 0.32 M solution of 1,3-propanedinitrite (1.60 mmol, 1.60 equiv) in THF (the solution was prepared in a 10 mL gauge flask and injected in a 5 mL calibrate loop). When solubility issues occurred, a few drops methanol were added. The 2 loops were injected in the system with a total flow rate of 0.667 mL•min⁻¹ (0.111 mL•min⁻¹ for pump A and 0.556 mL•min⁻¹ for pump B) in a 4 x 10 mL reactor at 70 °C with a back-pressure regulator at 250 psi. After a residence time of 60 min, the product was collected in an opened flask. The solvent was removed under reduced pressure and the residue was purified by flash chromatography.

General Flow Procedure B for the Esterification of Carboxylic Acids with Amino alcohols

A 1 mL sample loop A was charged with a 2 M solution of amino alcohol (2.00 mmol, 2.00 equiv) in THF. A 5 mL sample loop B was charged with a 0.2 M solution of carboxylic acid (1.00 mmol) and a 0.32 M solution of 1,3-propanedinitrite (1.60 mmol, 1.60 equiv) in THF (the solution was prepared in a 10 mL gauge flask and injected in a 5 mL calibrate loop). When solubility issues occurred, a few drops methanol were added. The 2 loops were injected in the system with a total flow rate of 1.333 mL•min⁻¹ (0.222 mL•min⁻¹ for pump A and 1.111 mL•min⁻¹ for pump B) in a 4 x 10 mL reactor at 100 °C with a back-pressure regulator at 250 psi. After a residence time of 30 min, the product was collected in an opened flask. The solvent was removed under reduced pressure and the residue was purified by flash chromatography.

General Batch Procedure for One-Pot Esterification of carboxylic acid with amine

In a round bottom flask with a magnetic stirring bar, carboxylic acid (0.500 mmol, 1.00 equiv) was diluted in *tert*-butanol (4 mL). The resulting mixture was stirred at room temperature. 1,3-propanedinitrite (0.096 mL, 0.800 mmol, 1.60 equiv) was added, followed by the amino alcohol (1.25 mmol, 2.50 equiv). The flask was placed in an oil bath and the mixture was heated to reflux for 6 to 16 hours. The solvent was removed under reduced pressure. The crude mixture was purified by flash chromatography.

Characterization Data for Esters

3-Hydroxypropyl benzoate (1). The title compound was prepared according to the general flow procedure **A** using 3-aminopropane-1-ol (1.00 mL, 2.00 mmol, 2.0 M in THF) in loop A, and benzoic acid (122 mg, 1.00 mmol) and 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 30% EtOAc/hexanes. Hydroxyester **1** (169 mg, 0.938 mmol, 94 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.³ The title compound was also prepared according to the general batch procedure (6 h) using benzoic acid (61 mg, 0.50 mmol), 1,3-propanedinitrite (97.5 μ L, 0.80 mmol), and 3-aminopropane-1-ol (95.6 μ L, 1.25 mmol). Hydroxyester **1** was obtained as a colorless oil after purification (89.7 mg, 0.498 mmol, 99%). R_f 0.14 (30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.06 – 8.01 (m, 2H), 7.59-7.54 (m, 1H), 7.44 (t, J = 7.7 Hz, 2H), 4.49 (t, J = 6.1 Hz, 2H), 3.78 (t, J = 6.1 Hz, 2H), 2.17 (br. s., 1H,) 2.01 (qn, J = 6.1 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 167.0, 133.2, 130.2, 129.8, 128.6, 61.9, 59.3, 32.0.

2-Hydroxyethyl benzoate (2). The title compound was prepared according to the general flow procedure **A** using 2-aminoethan-1-ol solution (1.00 mL, 2.00 mmol, 2.0 M in THF) in loop A, and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 70% EtOAc/hexanes. Hydroxyester **2** (138 mg, 0.831 mmol, 83 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.⁴ The title compound was also prepared according to the general batch procedure (6 h) using benzoic acid (61.6 mg, 0.504 mmol), 1,3-propanedinitrite (98.4 μ L, 0.807 mmol), and 2-aminoethan-1-ol (76.1 μ L, 1.26 mmol). Hydroxyester **2** was obtained as a colorless oil after purification (61.6 mg, 0.368 mmol, 73%). R_f 0.28 (66% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, J = 7.4 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.42 (t, J = 7.4 Hz, 2H), 4.44 (t, J = 4.7 Hz, 2H), 3.93 (t, J = 4.7 Hz, 2H), 2.33-1.91 (s (br), 1H); ¹³C NMR (100 MHz, CDCl₃) δ 167.1, 133.2, 130.0, 129.8, 128.5, 66.7, 61.4.

4-Hydroxybutyl benzoate (3). The title compound was prepared according to the general flow procedure **A** using 4-aminobutan-1-ol solution (1.00 mL, 2.00 mmol, 2.0 M in THF) in loop A, and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 30% EtOAc/hexanes. Hydroxyester **3** (168 mg, 0.865 mmol, 87 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.⁵ The title compound was also prepared according to the general batch procedure (16 h) using benzoic acid (61.6 mg, 0.504 mmol), 1,3-

³ X. Wu, F. A. Cruz, A. Lu and V. M. Dong, *J. Am. Chem. Soc.*, 2018, **140**, 10126-10130

⁴ D. Lee, C. L. Williamson, L. Chan and M. S. Taylor, *J. Am. Chem. Soc.*, 2012, **134**, 8260-8267.

⁵ Nagasawa, S.; Sasano, Y.; Iwabuchi, Y., *Chem. - Eur. J.* 2017, **23**, 10276-10279.

propanedinitrite (98.4 μL , 0.807 mmol), and 4-aminobutan-1-ol (117 μL , 1.25 mmol). Hydroxyester **3** was obtained as a colorless oil after purification (95.2 mg, 0.490 mmol, 98%). R_f 0.28 (30% EtOAc/hexanes); ^1H NMR (500 MHz, CDCl_3) δ 8.05-8.00 (m, 2H), 7.56-7.52 (m, 1H), 7.44-7.41 (m, 2H), 4.35 (t, $J = 6.5$ Hz, 2H), 3.71 (t, $J = 6.5$ Hz, 2H), 1.89-1.82 (m, 3H), 1.75-1.68 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.8, 133.0, 130.4, 129.6, 128.5, 64.9, 62.4, 29.3, 25.3.

5-Hydroxypentyl benzoate (4). The title compound was prepared according to the general flow procedure **A** using 5-aminopentan-1-ol solution (1.00 mL, 2.00 mmol, 2.0 M in THF) in loop A, and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 30% EtOAc/hexanes. Hydroxyester **4** (178 mg, 0.855 mmol, 86 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.⁶ The title compound was also prepared according to the general batch procedure (16 h) using benzoic acid (61.2 mg, 0.500 mmol), 1,3-propanedinitrite (97.5 μL , 0.800 mmol), and 5-aminopentan-1-ol (136 μL , 1.25 mmol). Hydroxyester **4** was obtained as a colorless oil after purification (101 mg, 0.486 mmol, 97%). R_f 0.32 (30% EtOAc/hexanes); ^1H NMR (500 MHz, CDCl_3) δ 8.05-8.02 (m, 2H), 7.57-7.53 (m, 1H), 7.45-7.42 (m, 2H), 4.33 (t, $J = 6.6$ Hz, 2H), 3.67 (t, $J = 6.5$ Hz, 2H), 1.81 (qn, $J = 7.1$ Hz, 2H), 1.70-1.65 (m, 2H), 1.61 (s(br), 1H), 1.59-1.53 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.8, 133.0, 130.5, 129.7, 128.5, 65.0, 62.8, 32.4, 28.7, 22.5.

8-Hydroxyoctyl benzoate (5). The title compound was prepared according to the general flow procedure **A** using a 8-aminoctan-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, (2 mL) and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The 2 loops were injected in the system with a total flow rate of $0.667 \text{ mL}\cdot\text{min}^{-1}$ ($0.190 \text{ mL}\cdot\text{min}^{-1}$ for pump A and $0.476 \text{ mL}\cdot\text{min}^{-1}$ for pump B) in a $4 \times 10 \text{ mL}$ reactor at 70°C with a back-pressure regulator at 250 psi. After a residence time of 60 min, the product was collected in an opened flask. The crude mixture was purified by flash chromatography using 30% EtOAc/hexanes. Hydroxyester **5** (193 mg, 0.772 mmol, 77 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.⁷ The title compound was also prepared according to the general batch procedure (6 h) using benzoic acid (61.4 mg, 0.503 mmol), 1,3-propanedinitrite (98.1 μL , 0.807 mmol), and 8-aminoctan-1-ol (183 μL , 1.26 mmol). Hydroxyester **5** was obtained as a colorless oil after purification (683 mg, 0.332 mmol, 66%). R_f 0.22 (30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 8.07-8.01 (m, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.6$ Hz, 2H), 4.31 (t, $J = 6.6$ Hz, 2H), 3.63 (t, $J = 6.7$ Hz, 2H), 1.77 (qn, $J = 7.0$ Hz, 2H), 1.61-1.51 (m, 2H), 1.49-1.30 (m, 8H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.8, 132.9, 130.6, 129.7, 128.5, 65.2, 63.1, 32.9, 29.42, 29.36, 28.8, 26.1, 25.8.

⁶ Yang, Y.-Q.; Lu, Z.; Xu, X., *Asian J. Org. Chem.* 2019, **8**, 2192-2195.

⁷ H. Sharghi and M. H. Sarvari, *Tetrahedron*, 2003, **59**, 3627-3633.

3-Hydroxypropyl 4-fluorobenzoate (6). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 4-fluorobenzoic acid (140 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/Hexane. Hydroxyester **6** (178 mg, 0.899 mmol, 90% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.⁸ R_f 0.29 (40% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) δ 8.03-7.97 (m, 2H), 7.09-7.02 (m, 2H), 4.43 (t, $J = 6.2$ Hz, 2H), 3.74 (t, $J = 6.1$ Hz, 2H), 2.70 (s (br), 1H), 1.97 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.1, 165.8 (d, $J = 253.7$ Hz), 132.2 (d, $J = 9.3$ Hz), 126.4 (d, $J = 3.0$ Hz), 115.6 (d, $J = 22.0$ Hz), 62.1, 59.0, 31.8 ^{19}F NMR (282 MHz, CDCl_3) δ -106.9 (m).

3-Hydroxypropyl 4-nitrobenzoate (7). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 4-nitrobenzoic acid (167 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/Hexane. Hydroxyester **7** (209 mg, 0.929 mmol, 93% yield) was isolated as a pale-yellow oil. Spectral data matched those reported in the literature.⁸ R_f 0.26 (40% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) δ 8.25 (d, $J = 9.0$ Hz, 2H), 8.18 (d, $J = 9.0$ Hz, 2H), 4.51 (t, $J = 6.2$ Hz, 2H), 3.82-3.73 (m, 2H), 2.16 (s (br), 1H), 2.03 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.1, 150.6, 135.6, 130.8, 123.6, 62.9, 59.1, 31.7.

3-Hydroxypropyl 4-methoxybenzoate (8). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 4-methoxybenzoic acid (140 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/Hexane. Hydroxyester **8** (191 mg, 0.910 mmol, 91% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.⁹ R_f 0.23 (40% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 8.8$ Hz, 2H), 6.86 (d, $J = 8.9$ Hz, 2H), 4.39 (t, $J = 6.2$ Hz, 2H), 3.79 (s, 3H), 3.72 (t, $J = 6.1$ Hz, 2H), 2.88 (s (br), 1H), 1.95 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.8, 163.4, 131.6, 122.4, 113.6, 61.6, 59.0, 55.4, 31.9.

3-Hydroxypropyl 2-bromobenzoate (9). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2-bromobenzoic acid (201 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol)

⁸ C. Kuhakarn, W. Panchan, S. Chiampanichayakul, N. Samakkanad, M. Pohmakotr, V. Reutrakul and T. Jaipetch, *Synthesis*, 2009, 929-934.

⁹ B. Karimi and J. Rajabi, *Synthesis*, 2003, 2373-2377

in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/Hexane. Hydroxyester **9** (200 mg, 0.772 mmol, 77% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.¹⁰ R_f 0.24 (40% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.80 (dd, $J = 7.4$ Hz, 2.0 Hz, 1H), 7.67 (dd, $J = 7.5$ Hz, 1.6 Hz, 1H), 7.40-7.31 (m, 2H), 4.51 (t, $J = 6.2$ Hz, 2H), 3.82 (t, $J = 6.1$ Hz, 2H), 2.94-2.83 (br. s., 1H), 2.04 (qn, $J = 6.1$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 134.2, 132.6, 131.2, 127.2, 121.4, 62.7, 59.0, 31.6.

3-Hydroxypropyl 2-iodobenzoate (10). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2-iodobenzoic acid (248 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50% EtOAc/Hexane. Hydroxyester **10** (275 mg, 0.899 mmol, 90% yield) was isolated as a clear oil. R_f 0.40 (50% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) 7.96 (dd, $J = 8.0$, 1.1 Hz, 1H), 7.76 (dd, $J = 7.8$, 1.7 Hz, 1H), 7.38 (td, $J = 7.6$, 1.2 Hz, 1H), 7.13 (td, $J = 7.7$, 1.7 Hz, 1H), 4.48 (t, $J = 6.2$ Hz, 2H), 3.79 (t, $J = 6.1$ Hz, 2H), 2.23 (s (br), 1H), 2.00 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 167.1, 141.3, 135.3, 132.8, 131.0, 128.0, 94.0, 62.7, 59.3, 31.7; FTIR (cm^{-1}) (neat) 3500-3100, 3000-2800, 1718, 1547, 1265, 1042; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_{12}\text{I}\text{O}_3$ 306.9825, found 306.9820.

3-Hydroxypropyl 2,6-diiodobenzoate (11). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2,6-diiodobenzoic acid (374 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/hexane. Hydroxyester **11** (324 mg, 0.750 mmol, 75% yield) was isolated as a clear oil. R_f 0.38 (40% EtOAc/hexane); ^1H NMR (500 MHz, CDCl_3) δ 7.77 (d, $J = 8.0$ Hz, 2H), 6.76 (t, $J = 8.0$ Hz, 1H), 4.51 (t, $J = 6.2$ Hz, 2H), 3.84 (t, $J = 6.1$ Hz, 2H), 2.08 (s (br), 1H), 2.04 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 168.8, 145.3, 138.7, 132.0, 91.2, 63.6, 59.4, 31.4; FTIR (cm^{-1}) (neat) 3500-3100, 3000-2800, 1722, 1542, 1267, 1043; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{10}\text{H}_{10}\text{I}_2\text{O}_3\text{Na}$ 454.8633, found 370.8611.

2-(2-Hydroxyethoxy)ethyl benzoate (12). The title compound was prepared according to the general flow procedure **A** using a 2-(2-aminoethoxy)ethan-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 66% EtOAc/hexanes. Hydroxyester **12** (164 mg, 0.781 mmol, 78 % yield) was isolated as a colorless oil. Spectral data matched those reported in the literature.¹¹ The title compound was also prepared

¹⁰ S. Shinsei, *Heterocycles*, 2017, 94.

¹¹ T. Targel, P. Ramesh and M. Portnoy, *Eur. J. Org. Chem.*, 2018, 3017-3021.

according to the general batch procedure (6 h) using benzoic acid (61.6 mg, 0.504 mmol), 1,3-propanedinitrite (98.4 μ L, 0.807 mmol), and 2-(2-aminoethoxy)ethan-1-ol (0.131 mL, 1.26 mmol). Hydroxyester **12** was obtained as a colorless oil after purification (61.5 mg, 0.292 mmol, 58%). R_f 0.22 (50% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, $J = 7.2$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 4.49 (t, $J = 4.7$ Hz, 2H), 3.84 (t, $J = 4.7$ Hz, 2H), 3.75 (t, $J = 4.7$ Hz, 2H), 3.65 (t, $J = 4.7$ Hz, 2H), 2.21 (s (br), 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.7, 133.2, 130.1, 129.8, 128.5, 72.5, 69.3, 64.1, 61.8.

2-(2-(2-Hydroxyethoxy)ethoxy)ethyl benzoate (13). The title compound was prepared according to the general flow procedure **B** using a 2,2'-(ethane-1,2-diylbis(oxy))bis(ethan-1-ol) solution in THF (1.00 mL, 2.00 mmol) in loop A, and benzoic acid (122 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using EtOAc. Hydroxyester **13** (188 mg, 0.732 mmol, 73% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.¹¹ R_f 0.37 (100% EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 8.05-8.01 (m, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.43-7.37 (t, $J = 7.6$ Hz, 2H), 4.48-4.43 (m, 2H), 3.83-3.79 (m, 2H), 3.70-3.61 (m, 6H), 3.59-3.54 (m, 2H), 2.70 (s (br), 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 133.1, 130.1, 129.7, 128.4, 72.6, 70.7, 70.4, 69.2, 64.0, 61.7.

3-Hydroxypropyl furan-2-carboxylate (14). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2-furoic acid (112 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50% EtOAc/hexanes. Hydroxyester **14** (143 mg, 0.841 mmol, 84 % yield) was isolated as a clear oil. The title compound was also prepared according to the general batch procedure (16 h) using furoic acid (56.0 mg, 0.500 mmol), 1,3-propanedinitrite (97.4 μ L, 0.800 mmol), and 3-aminopropane-1-ol (95.5 μ L, 1.25 mmol). Hydroxyester **14** was obtained as a clear oil after purification (84.2 mg, 0.495 mmol, 99%); R_f 0.29 (50% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.59-7.55 (m, 1H), 7.18 (dd, $J = 0.8$ Hz, 3.5 Hz, 1H), 6.52-6.48 (m, 1H), 4.46 (t, $J = 6.2$ Hz, 2H), 3.75 (t, $J = 6$ Hz, 2H), 2.11 (s (br), 1H), 1.98 (qn, $J = 6.1$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 159.2, 146.5, 144.6, 118.3, 112.0, 62.1, 59.3, 31.9; FTIR (cm^{-1}) (neat) 3600-3200, 3140, 3000-2800, 1708, 1580, 1300-1100; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_8\text{H}_{10}\text{O}_4\text{Na}$ 193.0471, found 193.0475.

3-Hydroxypropyl benzo[d]thiazole-6-carboxylate (15). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and benzo[d]thiazole-6-carboxylic acid (179 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50 % EtOAc/hexanes. Hydroxyester **15** (166 mg, 0.700 mmol, 70 % yield) was isolated as a yellow solid. The title compound was also prepared according to the

general batch procedure (6 h) using benzo[d]thiazole-6-carboxylic acid (26.5 mg, 0.148 mmol), 1,3-propanedinitrite (29.0 μ L, 0.238 mmol), and 3-aminopropane-1-ol (29.1 μ L, 0.380 mmol). Hydroxyester **15** was obtained as a light yellow solid after purification (30.8 mg, 0.440 mmol, 88%). R_f 0.23 (50 % EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 9.13 (s, 1H), 8.63 (s, 1H), 8.17-8.11 (m, 2H), 4.52 (t, J = 6.2 Hz, 2H), 3.80 (t, J = 6.1 Hz, 2H), 2.67 (s (br), 1H), 2.03 (qn, J = 6.1 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.4, 157.5, 156.1, 133.8, 127.5, 127.4, 124.3, 123.5, 62.3, 59.1, 32.0; FTIR (cm^{-1}) (neat) 3000-2800, 1745, 1649, 1157, 1122, 1054; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{11}\text{NO}_3\text{NaS}$ 260.0344, found 260.0351.

3-Hydroxypropyl thiazole-4-carboxylate (16). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and thiazole-4-carboxylic acid (129 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using EtOAc. Hydroxyester **16** (151 mg, 0.807 mmol, 81% yield) was isolated as a pale-yellow oil. R_f 0.24 (100% EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 8.88-8.82 (m, 1H), 8.24 (d, J = 2.0 Hz, 1H), 4.51 (t, J = 6.2 Hz, 2H), 3.76 (t, J = 6.0 Hz, 2H), 2.01 (qn, J = 6.1 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 161.7, 153.7, 148.0, 127.7, 62.8, 59.4, 31.8; FTIR (cm^{-1}) (neat) 3000-2800, 1745, 1692, 1649, 1157, 1122, 1054; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_7\text{H}_{10}\text{NO}_3\text{S}$ 188.0376, found 188.0376.

3-Hydroxypropyl 5-methylpyrazine-2-carboxylate (17). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 5-methylpyrazine-2-carboxylic acid (138 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using EtOAc. Hydroxyester **17** (179 mg, 0.913 mmol, 91% yield) was isolated as a clear oil. R_f 0.19 (100% EtOAc); ^1H NMR (500 MHz, CDCl_3) 9.14-9.12 (m, 1H), 8.54-8.52 (m, 1H), 4.55 (t, J = 6.3 Hz, 2H), 3.76 (t, J = 5.9 Hz, 2H), 2.63 (s, 3H), 2.03 (qn, J = 6.1 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 164.4, 158.0, 145.4, 144.3, 140.5, 63.5, 59.2, 31.7, 22.0; FTIR (cm^{-1}) (neat) 3600-3100, 3000-2800, 1718, 1276, 1135, 1032; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_3\text{Na}$ 219.0740, found 219.0743.

3-Hydroxypropyl 4-methyl-1,2,3-thiadiazole-5-carboxylate (18). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 4-methyl-1,2,3-thiadiazole-5-carboxylic acid (144 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50% EtOAc/hexane. Hydroxyester **18** (158 mg, 0.782 mmol, 78% yield) was isolated as a clear oil. The title compound was also prepared according to the general batch procedure (16 h) using 4-methyl-1,2,3-thiadiazole-5-carboxylic acid (72.4 mg, 0.502 mmol), 1,3-propanedinitrite (98.6 μ L, 0.809 mmol), and 3-aminopropane-1-ol (98.7 μ L,

1.29 mmol). Hydroxyester **18** was obtained as a clear oil after purification (63.2 mg, 0.312 mmol, 62%). R_f 0.25 (50% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 4.51 (t, J = 6.3 Hz, 2H), 3.78 (t, J = 6 Hz, 2H), 2.97 (s, 3H), 2.01 (qn, J = 6.2 Hz, 2H), 1.66 (s (br), 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 162.6, 160.2, 139.4, 63.5, 59.1, 31.6, 14.1; FTIR (cm^{-1}) (neat) 3600-3100, 3000-2800, 1725, 1206, 1046; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_7\text{H}_{11}\text{N}_2\text{O}_3\text{S}$ 203.0484, found 203.0483.

3-Hydroxypropyl hexadecanoate (19). The title compound was prepared according to the general flow procedure **A** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and palmitic acid (270 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 25% EtOAc/hexanes. Hydroxyester **19** (176 mg, 0.561 mmol, 56 % yield) was isolated as a white solid. Spectral data matched those reported in the literature.¹² The title compound was also prepared according to the general batch procedure (6 h) using palmitic acid (131 mg, 0.509 mmol), 1,3-propanedinitrite (99.3 μL , 815 μmol), and 3-aminopropane-1-ol (97.4 μL , 1.27 mmol). Hydroxyester **19** was obtained as a solid after purification (156 mg, 0.498 mmol, 99%). R_f 0.23 (25% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 4.19 (t, J = 6.1 Hz, 2H), 3.64 (t, J = 6.1 Hz, 2H), 2.43 (s (br), 1H), 2.27 (t, J = 7.6 Hz, 2H), 1.83 (qn, J = 6.1 Hz, 2H), 1.65-1.55 (m, 2H), 1.33-1.19 (m, 24H), 0.84 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 174.5, 61.3, 59.1, 34.4, 32.0, 31.8, 29.77, 29.76, 29.75, 29.73, 29.72, 29.68, 29.5, 29.4, 29.3, 29.2, 25.1, 22.8, 14.2.

3-Hydroxypropyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate (20). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 1-(4-chlorophenyl)cyclopropane-1-carboxylic acid (197 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 40% EtOAc/hexanes. Hydroxyester **20** (237 mg, 0.933 mmol, 93 % yield) was isolated as a clear sticky oil. R_f 0.29 (40% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.24-7.30 (m, 4H), 4.19 (t, J = 6.0 Hz, 2H), 3.56 (t, J = 6.0 Hz, 2H), 1.77 (qn, J = 6.0 Hz, 2H), 1.67-1.73 (s (br), 1H), 1.61 (dd, J = 4.1 Hz, 7.1 Hz, 2H), 1.18 (dd, J = 3.9 Hz, 6.9 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 174.7, 138.0, 133.2, 131.9, 128.5, 62.6, 59.5, 31.7, 28.7, 16.9; FTIR (cm^{-1}) (neat) 3600-3200, 3000-2800, 1715, 1494, 1168, 1049, 1014; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{15}\text{O}_3\text{NaCl}$ 277.0602, found 277.0606.

3-Hydroxypropyl but-2-ynoate (21). The title compound was prepared according to the flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2-butyric acid (142 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50% EtOAc/Hexane.

¹² M. Ikubo, A. Inoue, S. Nakamura, S. Jung, M. Sayama, Y. Otani, A. Uwamizu, K. Suzuki, T. Kishi, A. Shuto, J. Ishiguro, M. Okudaira, K. Kano, K. Makide, J. Aoki and T. Ohwada, *J. Med. Chem.*, 2015, **58**, 4204-4219.

Hydroxyester **21** (118 mg, 0.830 mmol, 83 % yield) was isolated as a clear oil. The title compound was also prepared according to the general batch procedure (16 h) using 3-aminopropane-1-ol (95.6 μ L, 1.25 mmol), 2-butynoic acid (42.9 mg, 0.500 mmol), and 1,3-propanedinitrite (95.6 μ L, 1.25 mmol). The crude mixture was purified by flash chromatography using 50% EtOAc/hexane. Hydroxyester **21** (70.8 mg, 0.498 mmol, 99% yield) was isolated as a clear oil. R_f 0.35 (50% EtOAc/hexane); $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 4.29 (t, $J = 6.1$ Hz, 2H), 3.70 (t, $J = 6.1$ Hz, 2H), 2.00 (s (br), 1H), 1.97 (s, 3H), 1.89 (qn, $J = 6.1$ Hz, 2H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 154.1, 86.1, 72.4, 62.9, 59.1, 31.8, 3.9; FTIR (cm^{-1}) (neat) 3500-3000, 3000-2800, 2242, 1710, 1250, 1065; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_7\text{H}_{10}\text{O}_3\text{Na}$ 165.0522, found 165.0520.

tert-Butyl 3-(3-(3-hydroxypropoxy)-3-oxopropyl)-1H-indole-1-carboxylate (22). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 3-(1-(*tert*-butoxycarbonyl)-1H-indol-3-yl)propanoic acid (289 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 50% EtOAc/hexane. Hydroxyester **22** (319 mg, 0.919 mmol, 92% yield) was isolated as a clear oil. The title compound was also prepared according to the general batch procedure (6 h) using 3-(1-(*tert*-butoxycarbonyl)-1H-indol-3-yl)propanoic acid (144 mg, 0.498 mmol), 1,3-propanedinitrite (97.1 μ L, 796 mmol), and 3-aminopropane-1-ol (95.2 μ L, 1.24 mmol). Hydroxyester **22** was obtained as a clear oil after purification (171 mg, 0.493 mmol, 99%). R_f 0.40 (50% EtOAc/hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.19-8.07 (m, 1H), 7.55 (d, $J = 7.6$ Hz, 1H), 7.41 (s, 1H), 7.37-7.31 (m, 1H), 7.30-7.24 (m, 1H), 4.28 (t, $J = 6.1$ Hz, 2H), 3.66 (t, $J = 6.0$ Hz, 2H), 3.06 (t, $J = 7.5$ Hz, 2H), 2.76 (t, $J = 7.6$ Hz, 2H), 1.95 (s (br), 1H), 1.87 (qn, $J = 6.0$ Hz, 2H), 1.69 (s, 9H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 173.5, 149.9, 130.4, 124.6, 122.7, 122.6, 119.5, 118.9, 115.4, 83.7, 61.5, 59.2, 34.0, 31.8, 28.3, 20.5; FTIR (cm^{-1}) (neat) 3600-3100, 3000-2800, 1668, 1610, 1434, 1334, 1300; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_5$ 370.1624, found 370.1632.

3-Hydroxypropyl 4-oxo-4-phenylbutanoate (23). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 4-oxo-4-phenylbutanoic acid (178 mg, 1.00 mmol), 1,3-propanedinitrite (195 μ L, 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 60% EtOAc/hexanes. Hydroxyester **23** (184 mg, 0.779 mmol, 78 % yield) was isolated as a pale-yellow oil. The title compound was also prepared according to the general batch procedure (16 h) using 4-oxo-4-phenylbutanoic acid (89.0 mg, 0.500 mmol), 1,3-propanedinitrite (97.4 μ L, 0.800 mmol), and 3-aminopropane-1-ol (95.5 μ L, 1.25 mmol). Hydroxyester **23** was obtained as a clear oil after purification (106 mg, 0.450 mmol, 90%). R_f 0.25 (60% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.94-7.89 (m, 2H), 7.52 (tt, $J = 1.3, 7.4$ Hz, 1H), 7.44-7.38 (m, 2H), 4.20 (t, $J = 6.1$ Hz, 2H), 3.64 (t, $J = 6.1$ Hz, 2H), 3.27 (t, $J = 6.5$ Hz, 2H), 2.76 (s (br), 1H), 2.71 (t, $J = 6.5$ Hz, 2H),

1.82 (qn, $J = 6.1$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.3, 173.3, 136.4, 133.3, 128.6, 128.0, 61.6, 58.9, 33.3, 31.6, 28.2; FTIR (cm^{-1}) (neat) 3600-3100, 3080, 3000-2800, 1729, 1982, 1448, 1596, 1215, 1160, 1049; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_4\text{Na}$ 259.0941, found 259.0942.

3-Hydroxypropyl 2-hydroxy-2-methylpropanoate (24). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and 2-hydroxy-2-methylpropanoic acid (104 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 4 % MeOH/DCM. Hydroxyester **24** (159 mg, 0.981 mmol, 98 % yield) was isolated as a pale-yellow oil. R_f 0.40 (10 % MeOH/DCM); ^1H NMR (400 MHz, CDCl_3) δ 4.36 (t, $J = 6.1$ Hz, 2H), 3.73 (t, $J = 6.1$ Hz, 2H), 1.94 (qn, $J = 6.1$ Hz, 2H), 1.46 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.9, 72.2, 62.8, 59.1, 31.8, 27.4; FTIR (cm^{-1}) (neat) 3500-3200, 3000-2800, 1725, 1270 1147 1047; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_7\text{H}_{14}\text{O}_4\text{Na}$ 185.0785, found 185.0784.

(S)-3-Hydroxypropyl (tert-butoxycarbonyl)alaninate (25). The title compound was prepared according to the general flow procedure **A** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and (S)-(tert-butoxycarbonyl)alanine (189 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 60 % EtOAc/hexanes. Hydroxyester **25** (181 mg, 0.732 mmol, 73 % yield) was isolated as a pale-yellow oil. R_f 0.24 (60 % EtOAc/hexanes); ^1H NMR (300 MHz, CDCl_3) δ 5.23-5.09 (m, 1H), 4.24 (t, $J = 6.1$ Hz, 2H), 3.64 (t, $J = 6.1$ Hz, 2H), 2.67 (s (br), 1H), 1.84 (qn, $J = 6.1$ Hz, 2H), 1.39 (s, 9H), 1.33 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 173.8, 155.3, 80.0, 62.3, 58.8, 49.4, 31.6, 28.4, 18.5; FTIR (cm^{-1}) (neat) 3500-3200, 3000-2800, 1689, 1518, 1160; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{21}\text{NO}_5\text{Na}$ 270.1311, found 270.1312.

(S)-3-Hydroxypropyl (tert-butoxycarbonyl)serinate (26). The title compound was prepared according to the general flow procedure **B** using a 3-aminopropane-1-ol solution in THF (1.00 mL, 2.00 mmol) in loop A, and (S)-(tert-butoxycarbonyl)serine (205 mg, 1.00 mmol), 1,3-propanedinitrite (195 μL , 1.60 mmol) in THF (5 mL) in loop B. The crude mixture was purified by flash chromatography using 6 % MeOH/DCM. Hydroxyester **26** (240 mg, 0.91 mmol, 91 % yield) was isolated as a yellow sticky oil. The title compound was also prepared according to the general batch procedure (16 h) using (S)-(tert-butoxycarbonyl)serine (103 mg, 0.503 mmol), 1,3-propanedinitrite (98.1 μL , 0.805 mmol), and 3-aminopropane-1-ol (96.2 μL , 1.26 mmol). Hydroxyester **26** was obtained as a sticky oil after purification (95.4 mg, 0.362 mmol, 72%). R_f 0.27 (6 % MeOH/DCM); ^1H NMR (400 MHz, CDCl_3) δ 5.70-5.63 (m, 1H), 4.42-4.17 (m, 3H), 3.97 (m, 1H), 3.80 (dd, $J = 3.5, 11.3$ Hz, 1H), 7.78 (t, $J = 5.8$ Hz, 2H), 3.46-3.20 (s (br), 1H), 1.87 (qn, 5.9 Hz, 2H), 1.42 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.4, 156.0, 80.4, 63.2, 63.0, 59.1, 56.0, 31.3,

28.4; FTIR (cm^{-1}) (neat) 3500-3200, 1692, 1504, 1160, 1054; HRMS (ESI-TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{21}\text{NO}_6$ 286.1253, found 286.1261.

3-Hydroxypropyl quinoline-6-carboxylate (27). The title compound was prepared according to the general batch procedure (6 h) using 3-aminopropane-1-ol (96.6 μL , 1.26 mmol), quinoline-6-carboxylic acid (87.5 mg, 0.505 mmol), and 1,3-propanedinitrite (98.6 μL , 0.808 mmol). The crude mixture was purified by flash chromatography using EtOAc. Hydroxyester **27** (79.8 mg, 0.343 mmol, 68% yield) was isolated as an off-white solid. R_f 0.30 (100% EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 8.97 (dd, $J = 1.7, 4.2$ Hz, 1H), 8.51 (d, $J = 1.8$ Hz, 1H), 8.25 – 8.19 (m, 2H), 8.09 (d, $J = 8.8$ Hz, 1H), 7.47-7.41 (m, 1H), 4.54 (t, $J = 6.2$ Hz, 2H), 3.83 (t, $J = 6.1$ Hz, 2H), 2.89 (s (br), 1H), 2.06 (qn, $J = 6.2$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.3, 152.4, 149.8, 137.6, 131.0, 129.6, 129.0, 128.2, 127.4, 121.9, 62.9, 58.9, 31.9; FTIR (cm^{-1}) (neat) 3500-3200, 3000-2800, 1711, 1603, 1505, 1462, 1276; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{14}\text{NO}_3$ 232.0970, found 232.09682.

3-Hydroxypropyl benzofuran-2-carboxylate (28). The title compound was prepared according to the general batch procedure (6 h) using 3-aminopropane-1-ol (0.150 mL, 2.00 mmol), and benzofuran-2-carboxylic acid (81.0 mg, 0.500 mmol), 1,3-propanedinitrite (96 μL , 0.80 mmol). The crude mixture was purified by flash chromatography using 45% EtOAc/hexane. Hydroxyester **28** (78.1 mg, 0.355 mmol, 71% yield) was isolated as a clear oil. R_f 0.33 (45% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, $J = 7.9$ Hz, 1H), 7.60-7.57 (m, 1H), 7.54 (d, $J = 0.9$ Hz, 1H), 7.48-7.43 (m, 1H), 7.33-7.28 (m, 1H), 4.55 (t, $J = 6.2$ Hz, 2H), 3.81 (t, $J = 6$ Hz, 2H), 2.04 (qn, $J = 6.1$ Hz, 2H), 1.98 (s (br), 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 160.0, 155.8, 145.4, 127.8, 127.0, 123.9, 122.9, 114.2, 112.4, 62.6, 59.2, 31.8; FTIR (cm^{-1}) (neat) 3600-3200, 3065, 300-2800, 1713, 1613, 1447, 1293, 1175, 747; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{13}\text{O}_4$ 221.0808, found 221.0808.

3-Hydroxypropyl-adamantane-1-carboxylate (29). The title compound was prepared according to the general batch procedure (16 h) using 3-aminopropane-1-ol (95.6 μL , 1.25 mmol), adamantane-1-carboxylic acid (90.1 mg, 0.500 mmol), and 1,3-propanedinitrite (95.6 μL , 1.25 mmol). The crude mixture was purified by flash chromatography using 25% EtOAc/hexane. Hydroxyester **29** (70.6 mg, 0.295 mmol, 59% yield) was isolated as a pale-yellow oil. R_f 0.28 (25% EtOAc/hexane); ^1H NMR (300 MHz, CDCl_3) δ 4.19 (t, $J = 6.1$ Hz, 2H), 3.64 (t, $J = 6.0$ Hz, 2H), 2.31 (s (br), 1H), 2.03-1.95 (m, 3H), 1.89-1.80 (m, 6H), 1.75-1.62 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 178.4, 61.1, 59.3, 40.9, 39.0, 36.6, 32.0, 28.0; FTIR (cm^{-1}) (neat) 3500-3200, 3000-2800, 1724, 1704, 1453, 1231, 1075; HRMS (ESI-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{14}\text{NO}_3$ 232.0970, found 232.09682.

Methyl [1,1'-biphenyl]-4-carboxylate (30). The title compound was prepared according to the general batch procedure (6 h) using 4-biphenylcarboxylic acid (99.1 mg, 0.500 mmol), 2,2-

dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and methylamine (0.250 mL, 2M solution in THF, 1.25 mmol). The crude mixture was purified by flash chromatography using 3% EtOAc/hexane. Ester **30** (67.9 mg, 0.320 mmol, 64% yield) was isolated as a white solid. Spectral data matched those reported in the literature.¹³ R_f 0.37 (3% EtOAc/hexane); ^1H NMR (300 MHz, CDCl_3) δ 8.12 (d, $J = 8.7$ Hz, 2H), 7.70-7.70 (m, 4H), 7.51-7.37 (m, 3H), 3.95 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 167.1, 145.7, 140.1, 130.2, 129.0, 128.9, 128.4, 127.4, 127.1, 52.2.

Methyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate (31). The title compound was prepared according to the general batch procedure (6 h) using 1-(4-chlorophenyl)cyclopropanecarboxylic acid (98.3 mg, 0.500 mmol), 2,2-dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and methylamine (0.250 mL, 2M solution in THF, 1.25 mmol). The crude mixture was purified by flash chromatography using 5% EtOAc/hexane. Ester **31** (99 mg, 470 μmol , 94% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.¹⁴ R_f 0.33 (5% EtOAc/hexane); ^1H NMR (300 MHz, CDCl_3) δ 7.26 (s, 4H), 3.61 (s, 3H), 1.60 (q, $J = 3.7$ Hz, 2H), 1.15 (q, $J = 3.7$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 174.7, 138.1, 133.1, 132.0, 128.4, 52.5, 28.5, 16.8.

Thiophen-2-ylmethyl (R)-4-((3R,5R,8R,9S,10S,13R,14S,17R)-3-hydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate (32). The title compound was prepared according to the general batch procedure (6 h) using lithocholic acid (188 mg, 0.500 mmol), 2,2-dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and 2-thiophenemethylamine (0.128 mL, 1.25 mmol). The crude mixture was purified by flash chromatography using 20% EtOAc/hexane. Ester **32** (157 mg, 0.345 mmol, 67% yield) was isolated as a yellow crystalline solid. Spectral data matched those reported in the literature.¹⁴ R_f 0.21 (20% EtOAc/hexane); ^1H NMR (300 MHz, CDCl_3) δ 7.31-7.28 (dd, $J = 5.1$ Hz, 1.2 Hz, 1H), 7.09-7.06 (m, 1H), 6.99-6.95 (m, 1H), 5.25 (m, 2H), 3.61 (m, 1H), 2.43-2.31 (m, 1H), 2.31-2.18 (m, 1H), 2.08 (s (br), 1H), 1.97-1.72 (m, 6H), 1.70-1.60 (m, 1H), 1.59-1.46 (m, 1H), 1.44-1.32 (m, 7H), 1.31-1.19 (m, 4H), 1.13-0.99 (m, 5H), 0.98-0.93 (m, 1H), 0.91 (s, 3H), 0.88 (d, $J = 6.3$ Hz, 3H), 0.61 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 174.0, 138.2, 128.1, 126.9, 126.8, 71.8, 60.4, 56.5, 56.0, 42.8, 42.2, 40.5, 40.2, 36.5, 35.9, 35.4, 35.3, 34.6, 31.3, 31.0, 30.6, 28.2, 27.3, 26.5, 24.3, 23.5, 20.9, 18.3, 12.1.

2-(N,N-Diethylamino)ethyl 2,2-diphenylacetate (33).

The title compound was prepared according to the general batch procedure (6 h) using 2,2-diphenylacetic acid (106 mg, 0.500 mmol), 2,2-dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and *N,N*-diethylethane-1,2-diamine (145 mg, 1.25 mmol). The crude mixture was purified by flash chromatography using 5% MeOH/AcOEt. Ester **33** (152 mg, 305 μmol , 98% yield) was

¹³ D. Martinez-Solorio, B. Melillo, L. Sanchez, Y. Liang, E. Lam, K. N. Houk and A. B. Smith, *J. Am. Chem. Soc.*, 2016, **138**, 1836-1839.

¹⁴ C. Audubert and H. Lebel, *Org. Lett.*, 2017, **19**, 4407-4410.

isolated as a clear oil. Spectral data matched those reported in the literature.¹⁵ R_f 0.31 (5% MeOH/AcOEt); ^1H NMR (400 MHz, CDCl_3) δ 7.35 – 7.27 (m, 10H), 5.06 (s, 1H), 4.25 (t, J = 6.0 Hz, 2H), 2.70 (t, J = 6.4 Hz, 2H), 2.53 (q, J = 6.8 Hz, 4H), 0.99 (t, J = 6.8 Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 172.6, 138.8, 128.8, 128.7, 127.4, 63.7, 57.3, 51.1, 47.7, 12.0.

Cyclopentyl cinnamate (34). The title compound was prepared according to the general batch procedure (6 h) using trans-cinnamic acid (74.1 mg, 0.500 mmol), 2,2-dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and cyclopentylamine (0.123 mL, 1.25 mmol). The crude mixture was purified by flash chromatography using 5% EtOAc/hexane. Ester **34** (78.9 mg, 0.365 mmol, 73% yield) was isolated as an orange oil. Spectral data matched those reported in the literature.¹⁶ R_f 0.40 (5% EtOAc/hexane); ^1H NMR (300 MHz, CDCl_3) δ 7.65 (d, J = 16.0 Hz, 1H), 7.55-7.48 (m, 2H), 7.42-7.33 (m, 3H), 6.42 (d, J = 16.0 Hz, 1H), 5.33-5.26 (m, 1H), 2.00-1.85 (m, 2H), 1.85-1.70 (m, 4H), 1.69-1.55 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 167.0, 144.4, 134.7, 130.2, 129.0, 128.1, 118.9, 77.3, 32.9, 23.9.

Adamantan-1-yl benzoate (35).

The title compound was prepared according to the general batch procedure (16 h) using benzoic acid (61.1 mg, 0.500 mmol), 2,2-dimethylpropane-1,3-dinitrite (130 mg, 0.800 mmol), and adamantylamine (197 mg, 1.25 mmol). The crude mixture was purified by flash chromatography using 7% EtOAc/hexane. Ester **35** (127 mg, 0.495 mmol, 99% yield) was isolated as a clear oil. Spectral data matched those reported in the literature.¹⁷ R_f 0.32 (7% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 8.01-7.97 (m, 2H), 7.51 (tt, J = 7.4 Hz, 1.3 Hz, 1H), 7.41 (t, J = 7.6 Hz, 2H), 2.27-2.26 (m, 6H), 2.22 (s, 3H), 1.72 (m, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.5, 132.4, 132.2, 129.5, 128.2, 81.0, 41.5, 36.3, 31.0.

Benzyl (S)-(2-oxotetrahydrofuran-3-yl)carbamate (36).

A 2 mL sample loop A was charged with a 0.125 M solution of (S)-4-amino-2-(((benzyloxy)-carbonyl)amino)butanoic acid (0.25 mmol, 1.00 equiv) in *tert*-butanol/ H_2O 5:1 v/v. A 1 mL sample loop B was charged with a 0.4 M solution of 1,3-propanedinitrite (0.40 mmol, 1.60 equiv) in *tert*-butanol/ H_2O 5:1 v/v. The 2 loops were injected in the system with a total flow rate of 1.333 $\text{mL}\cdot\text{min}^{-1}$ (0.889 $\text{mL}\cdot\text{min}^{-1}$ for pump A and 0.444 $\text{mL}\cdot\text{min}^{-1}$ for pump B) in a 4 x 10 mL reactor at 120 °C with a back-pressure regulator at 250 psi. After a residence time of 30 min, the product was collected in an opened flask. The solvent was removed under reduced pressure and the residue was purified by flash chromatography using 50% EtOAc/Hexane. Lactone **36** (54.7 mg, 0.232 mmol, 93% yield) was isolated as a white solid. Spectral data matched those reported in

¹⁵ S.-S. Yan, L. Zhu, J.-H. Ye, Z. Zhang, H. Huang, H. Zeng, C.-J. Li, Y. Lan and D.-G. Yu, *Chem. Sci.*, 2018, **9**, 4873-4878.

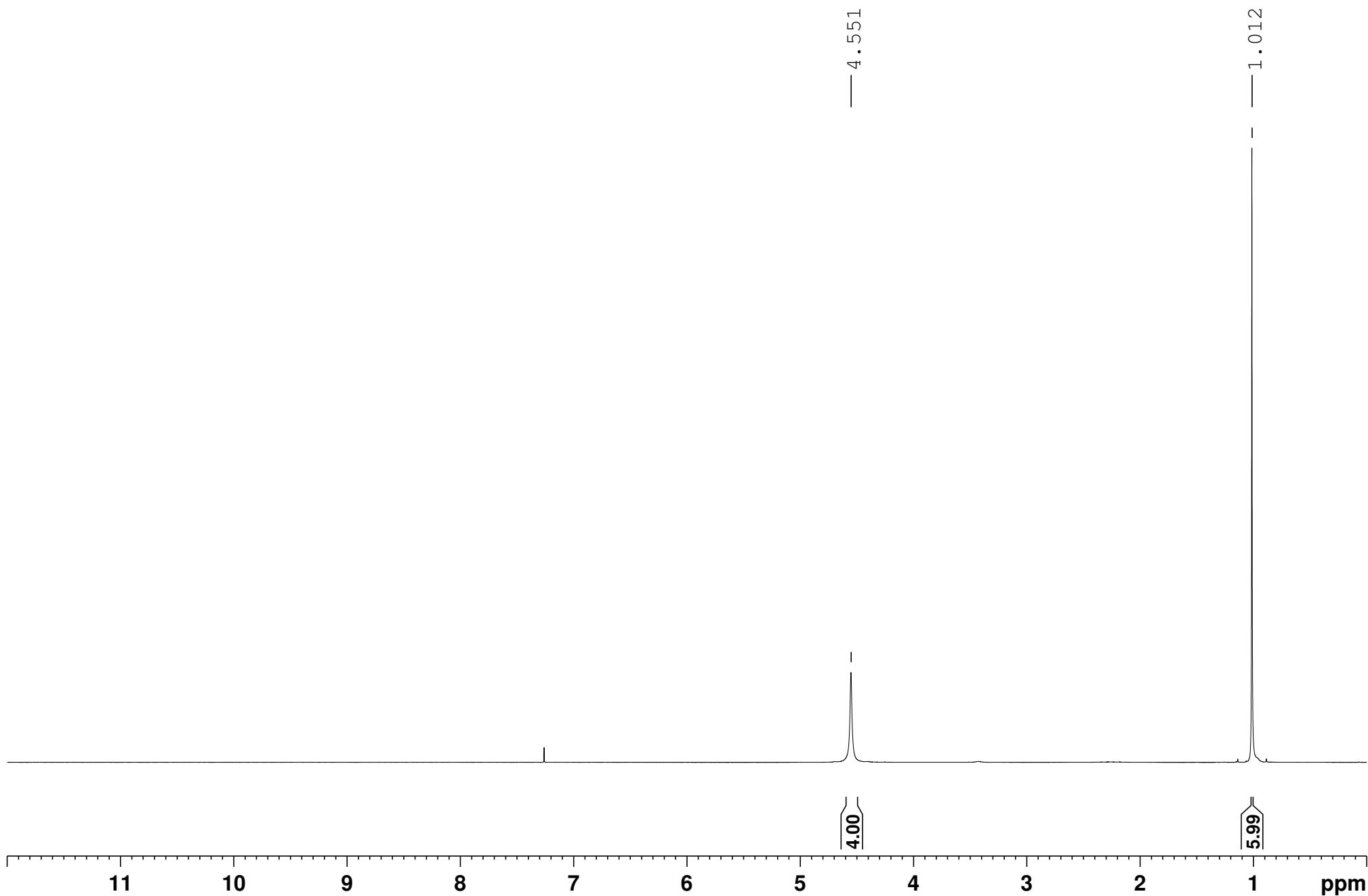
¹⁶ N. Iranpoor, H. Firouzabadi, A. Riazi and K. Pedrood, *J. Organomet. Chem.*, 2016, **822**, 67-73.

¹⁷ Z. Xin, T. M. Gøgsig, A. T. Lindhardt and T. Skrydstrup, *Org. Lett.*, 2012, **14**, 284-287.

the literature.¹⁸ R_f 0.28 (50% EtOAc/Hexane); ¹H NMR (300 MHz, CDCl₃) δ 7.39-7.30 (m, 5H), 5.49-5.35 (s (br), 1H), 5.12 (s, 2H), 4.49-4.34 (m, 2H), 4.29-4.17 (m, 1H), 2.82-2.69 (m, 1H), 2.29-2.12 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 175.1, 156.2, 136.0, 128.7, 128.5, 128.3, 67.5, 65.6, 50.6, 30.5.

¹⁸ S. P. Singh, A. Michaelides, A. R. Merrill and A. L. Schwan, *J. Org. Chem.*, 2011, **76**, 6825-6831.

1,3-(2,2-Dimethyl)Propanedinitrite - 1H - CDCl3 - 500 MHz

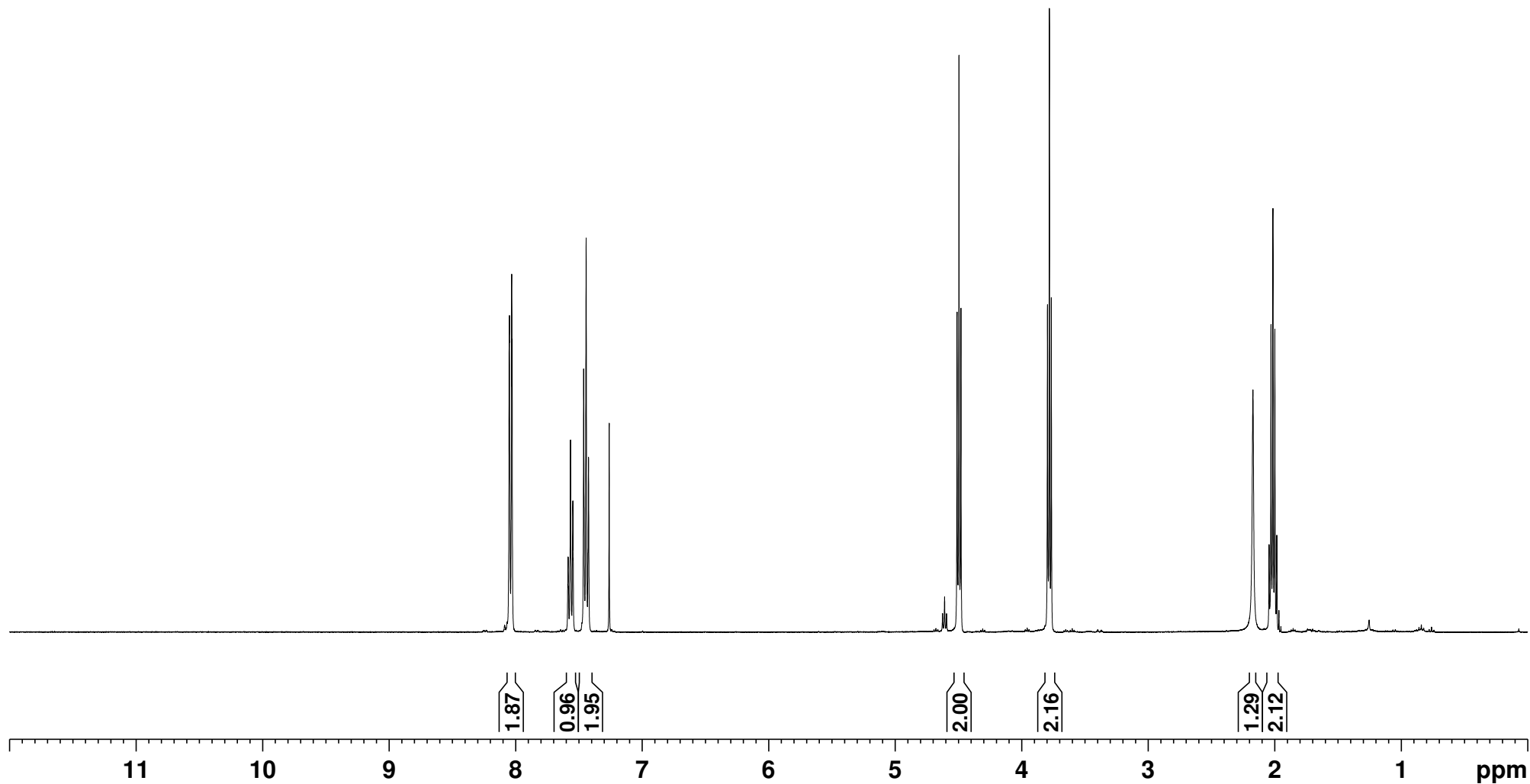


3-hydroxypropyl benzoate - 1H - CDCl3 - 400 MHz

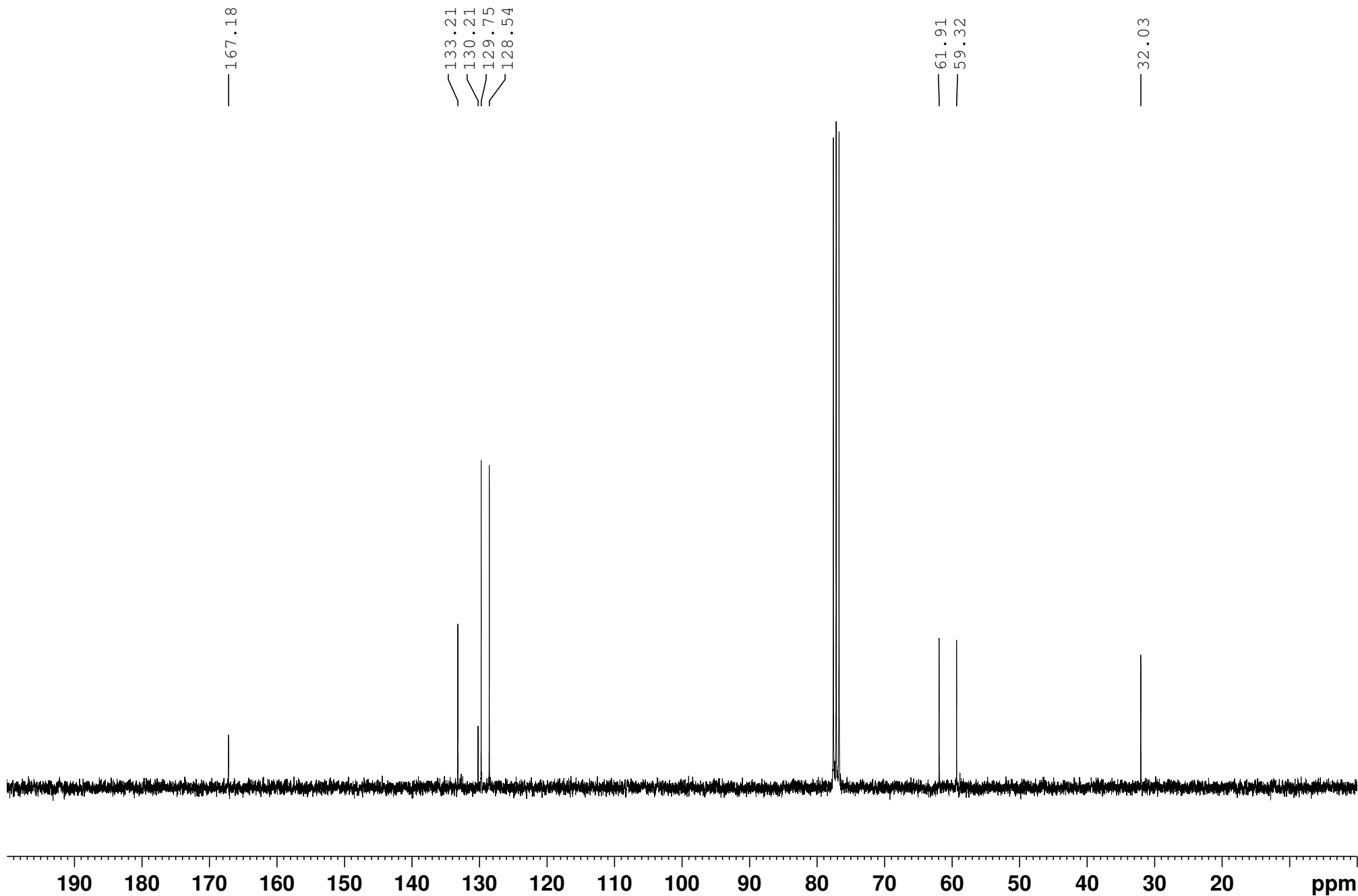
8.05
8.05
8.03
8.03
7.59
7.58
7.58
7.57
7.55
7.55
7.54
7.46
7.44
7.42
7.26

4.51
4.49
4.48
3.80
3.78
3.76

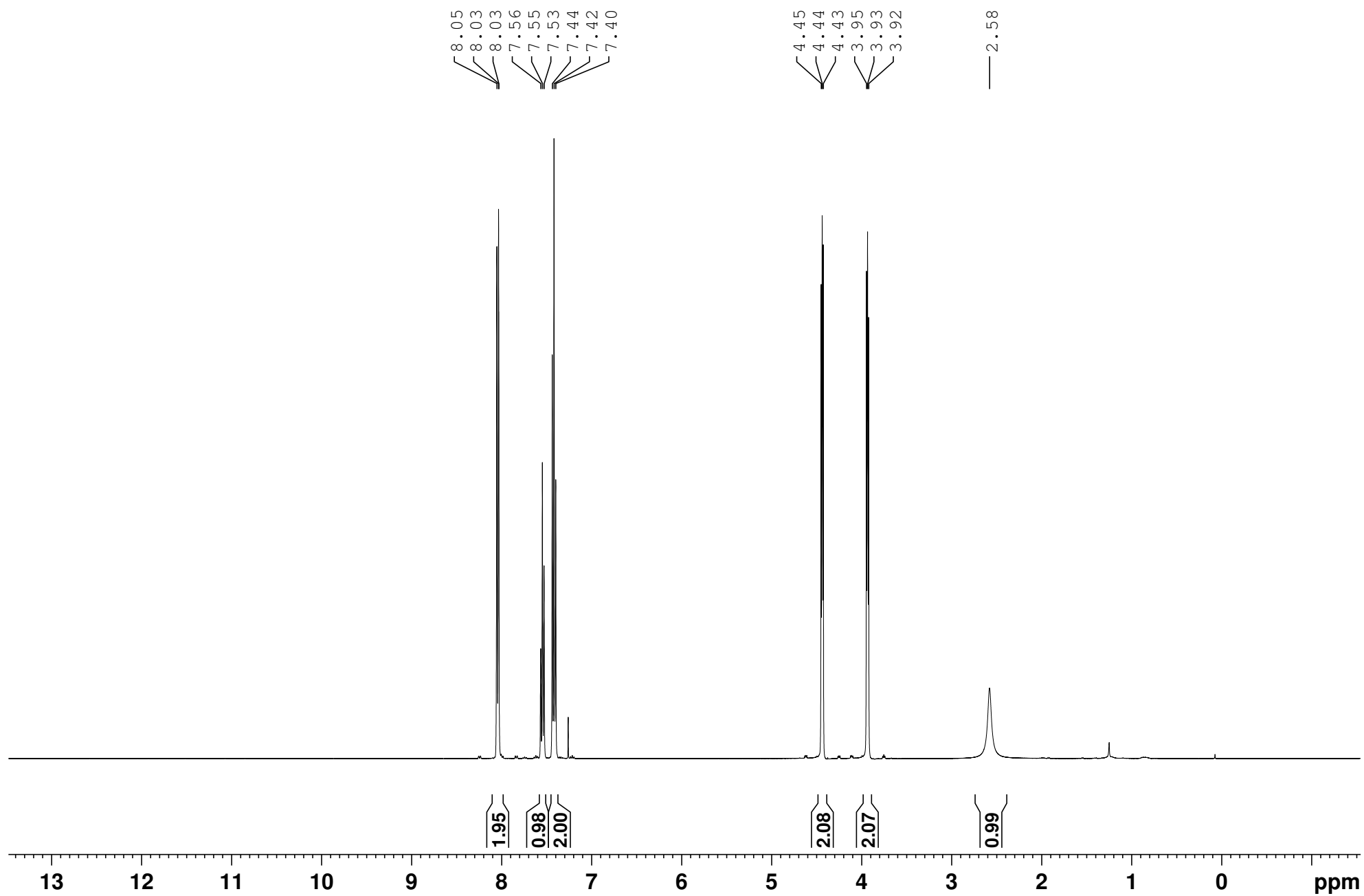
2.17
2.04
2.03
2.01
2.00
1.98



3-hydroxypropyl benzoate - ¹³C - CDCl₃ - 75 MHz



2-hydroxyethyl benzoate - 1H - CDCl3 - 400 MHz

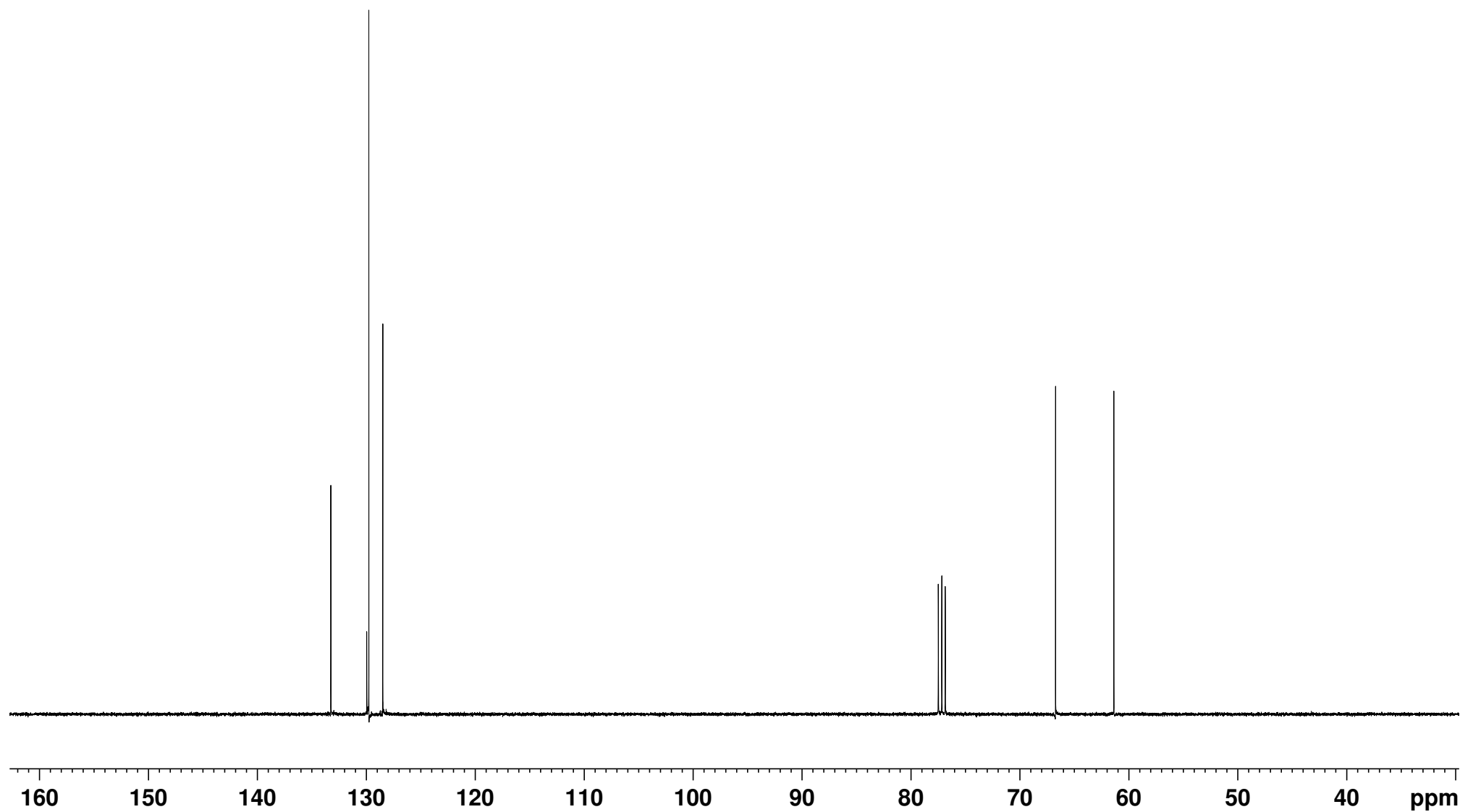


2-hydroxyethyl benzoate - ^{13}C - CDCl_3 - 100 MHz

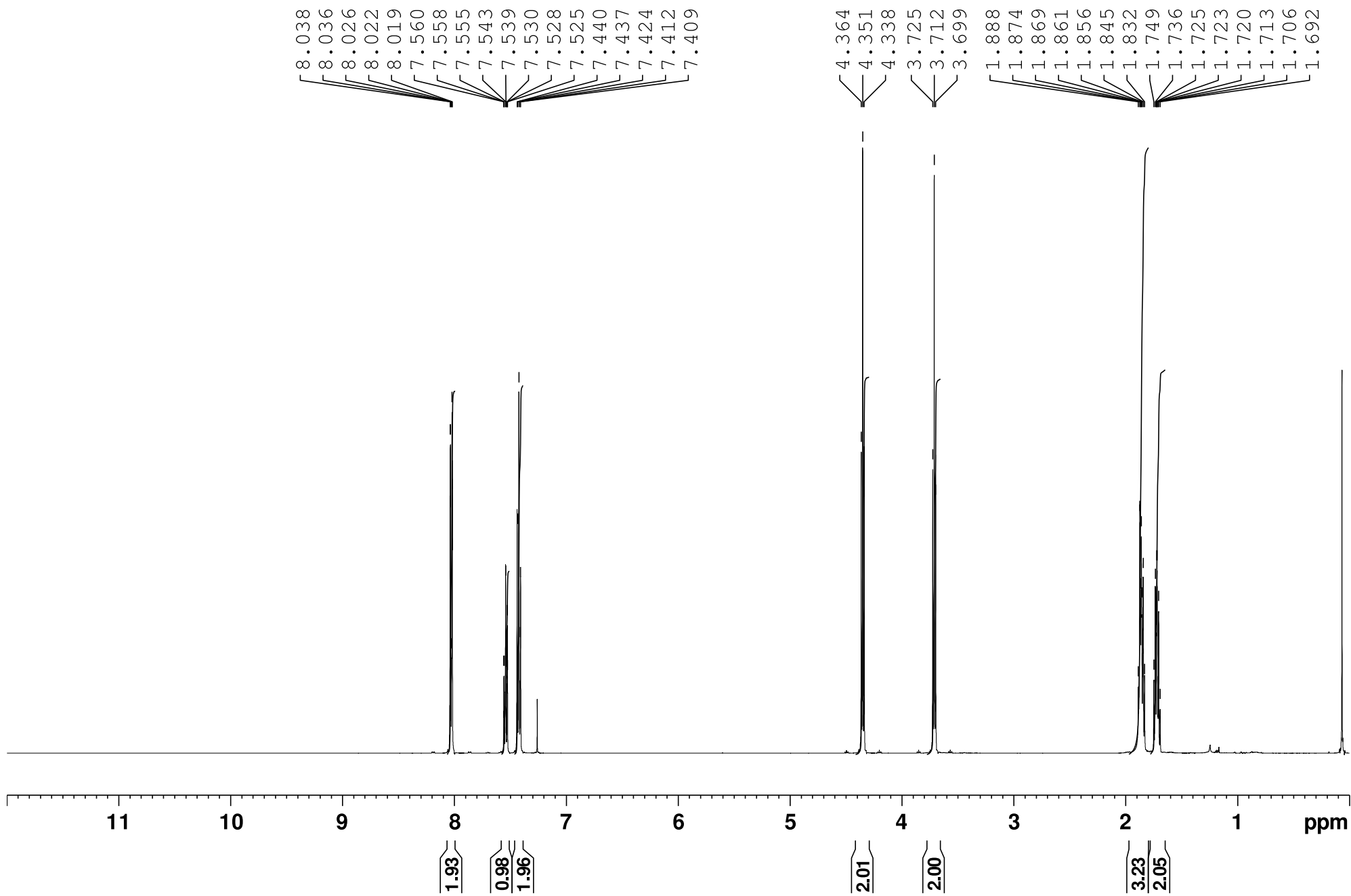
133.25
129.95
129.76
128.48

66.71

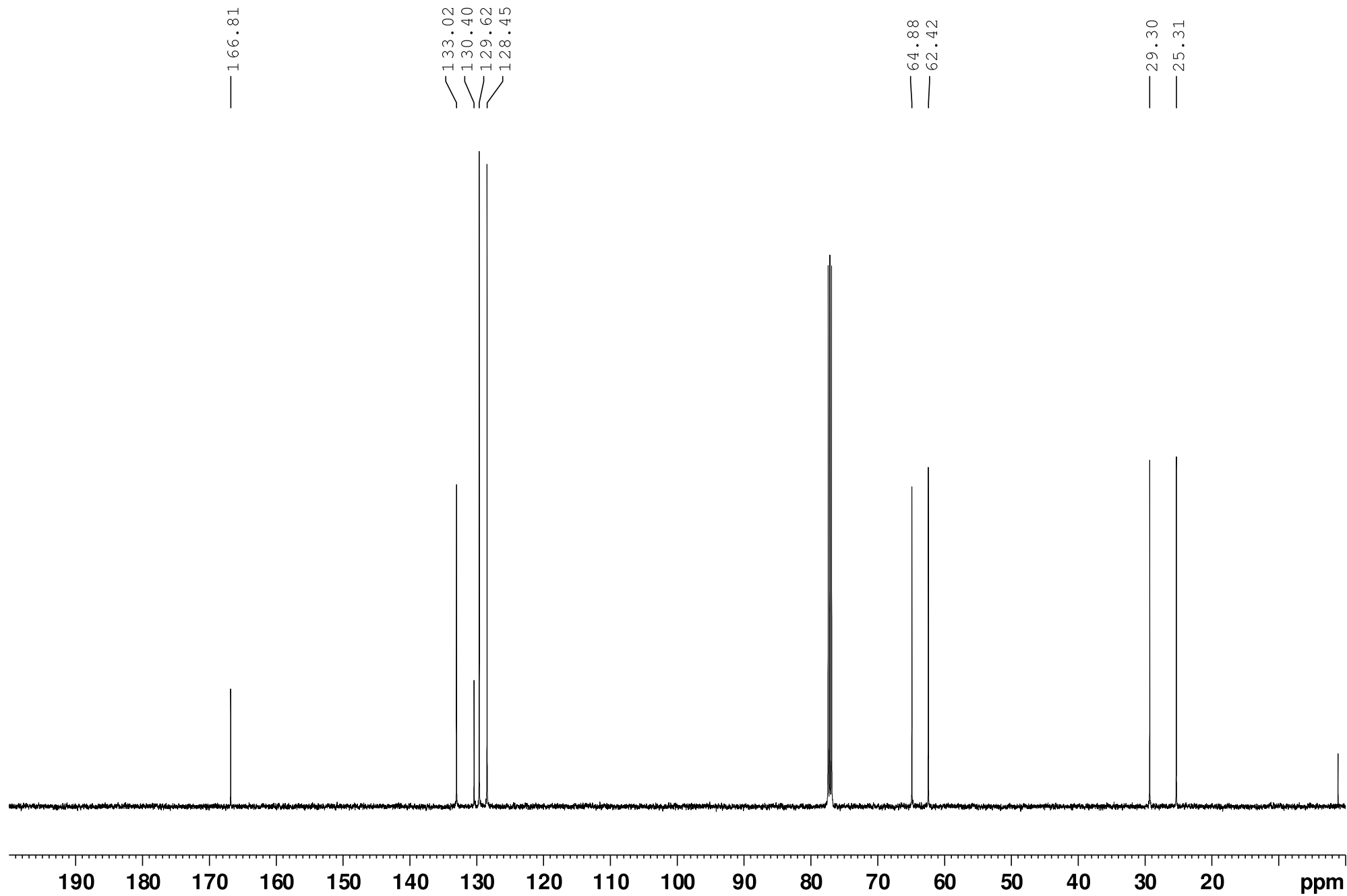
61.36



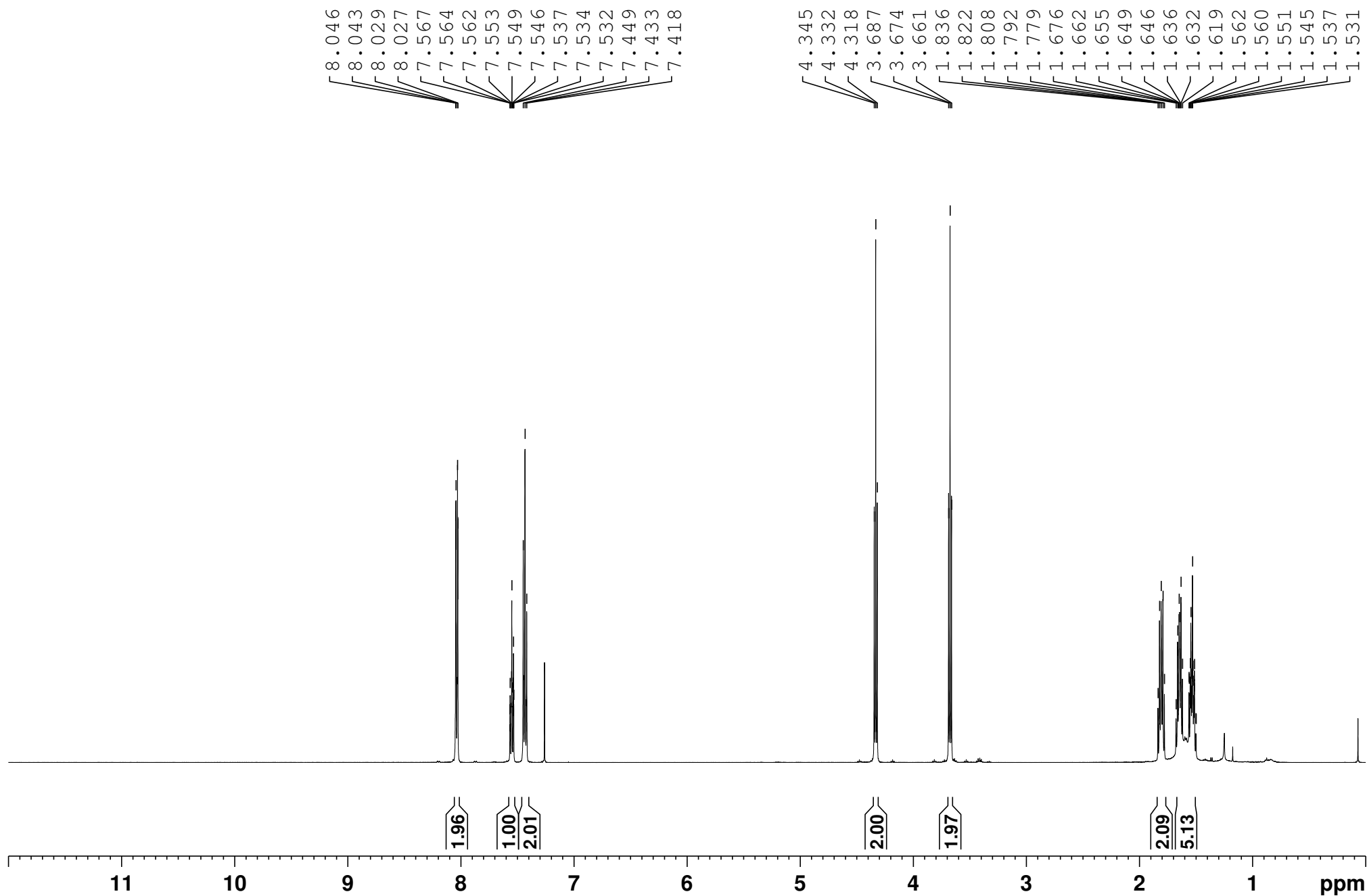
4-hydroxybutyl benzoate - 1H - CDCl3 - 500 MHz



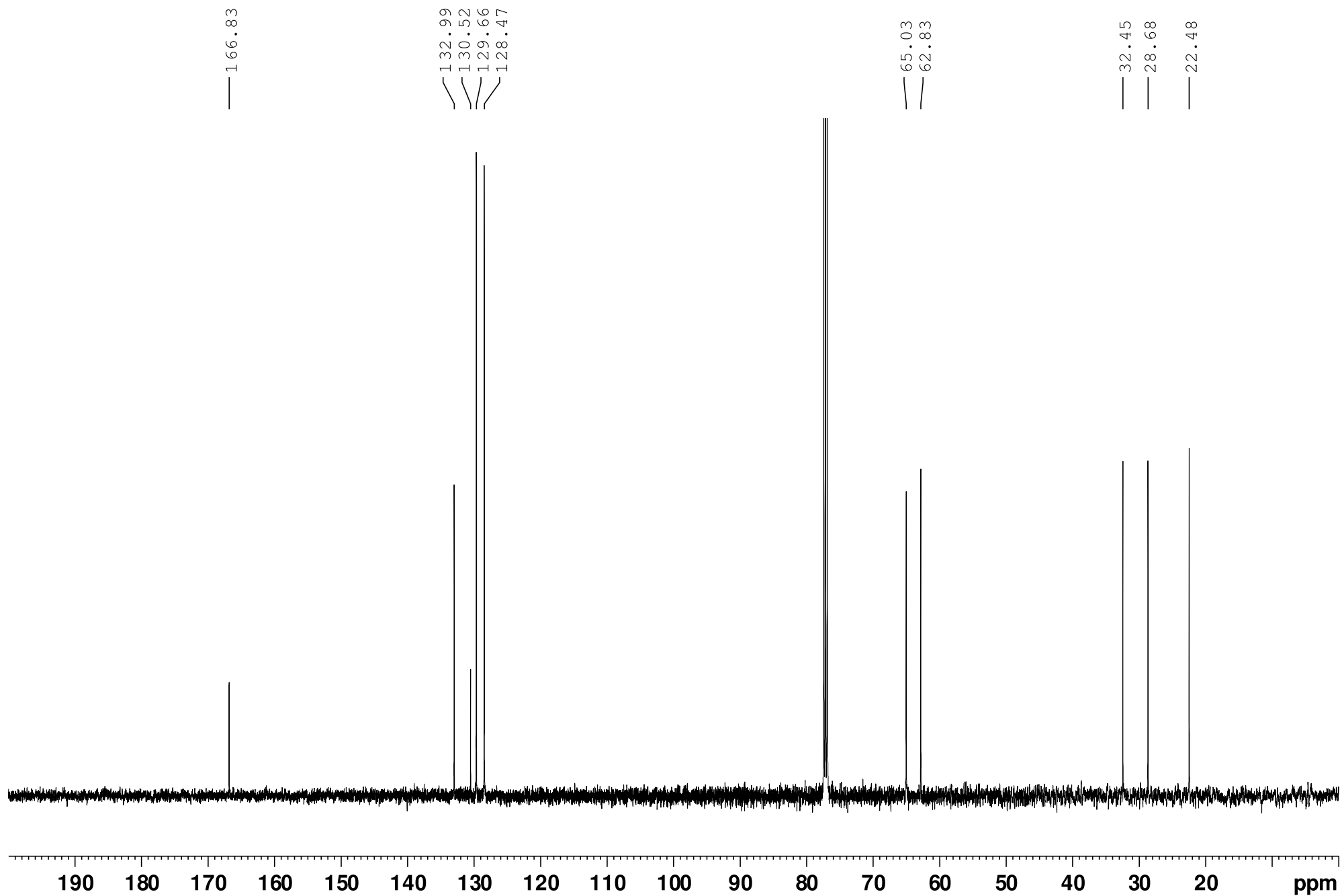
4-hydroxybutyl benzoate - ^{13}C - CDCl_3 - 125 MHz



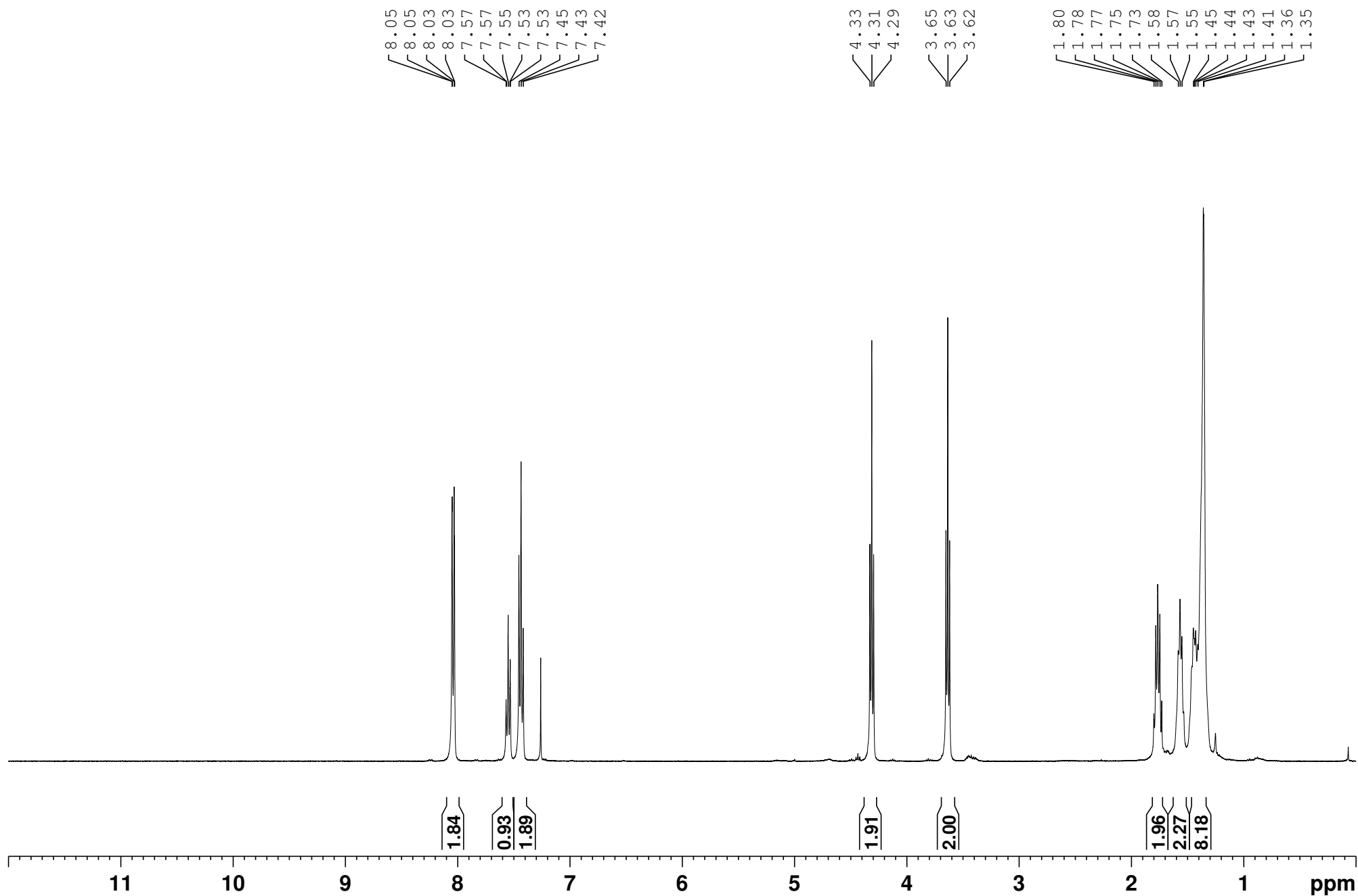
5-hydroxypentyl benzoate - 1H - CDCl3 - 500 MHz



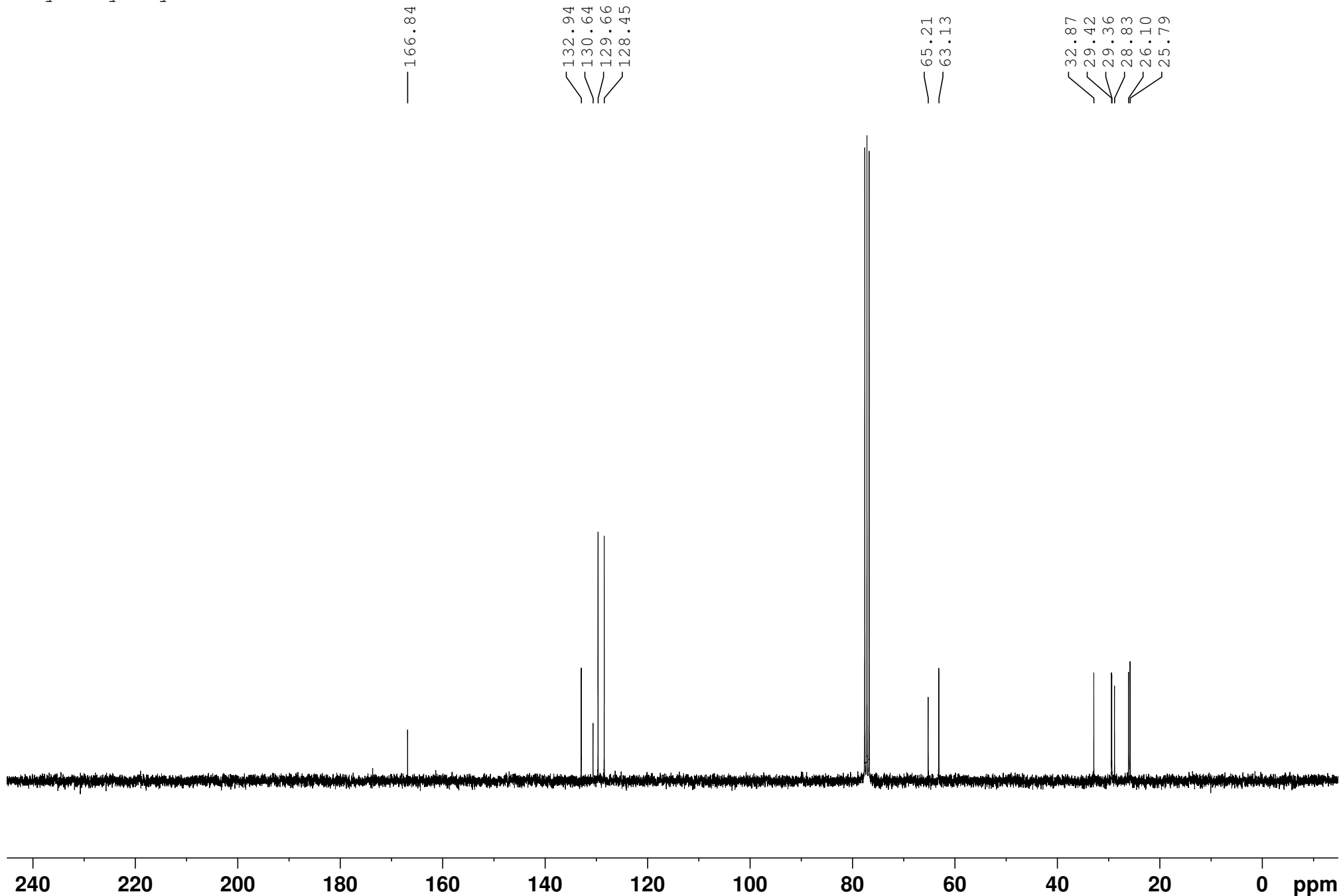
5-hydroxypentyl benzoate - ^{13}C - CDCl_3 - 125 MHz



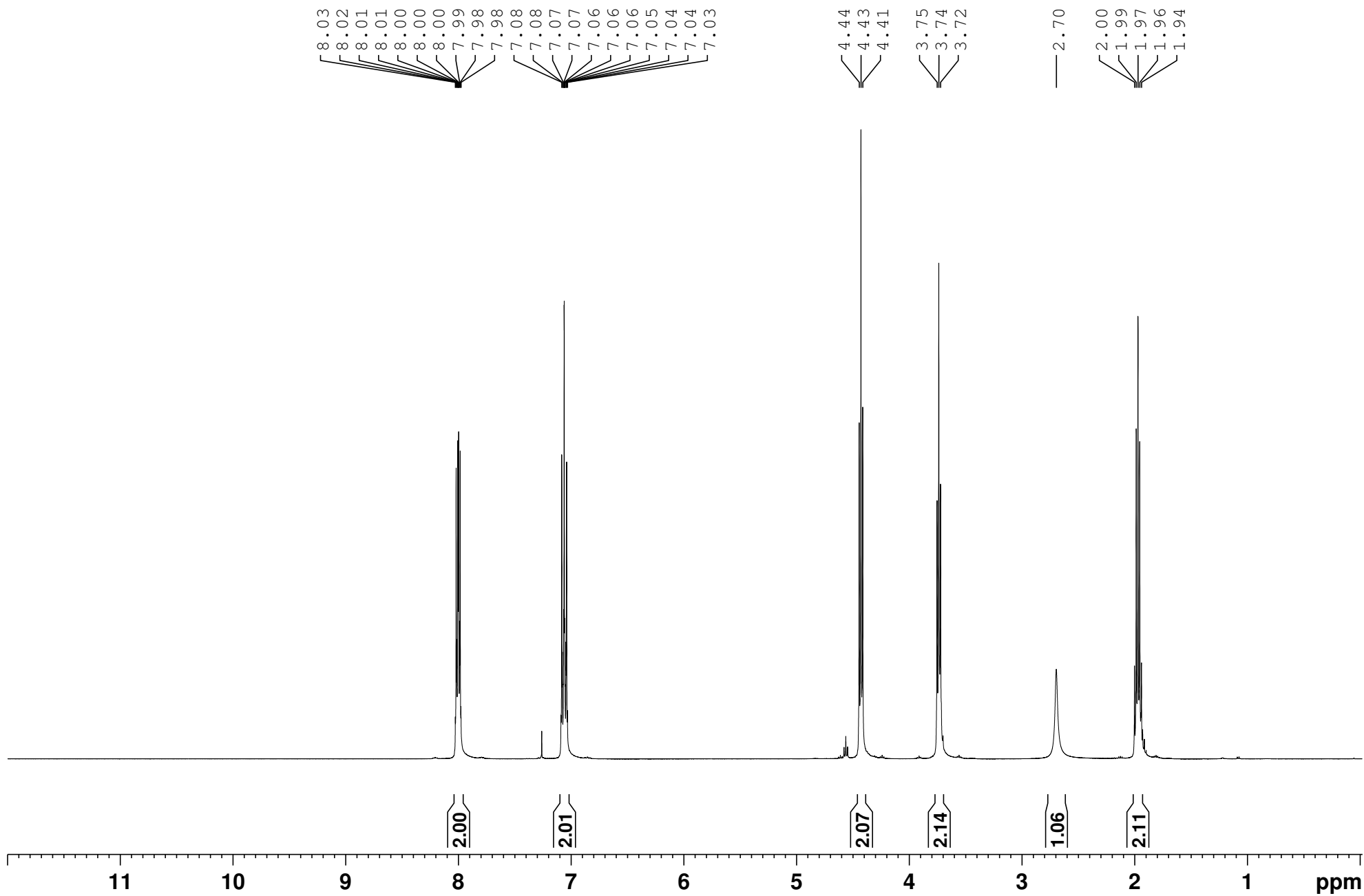
8-hydroxyoctyl benzoate - 1H - CDCl3 - 400 MHz



8-hydroxyoctyl benzoate - ^{13}C - CDCl_3 - 75 MHz



3-hydroxypropyl 4-fluorobenzoate - 1H - CDCl3 - 400 MHz



3-hydroxypropyl 4-fluorobenzoate - ¹³C - CDCl₃ - 75 MHz

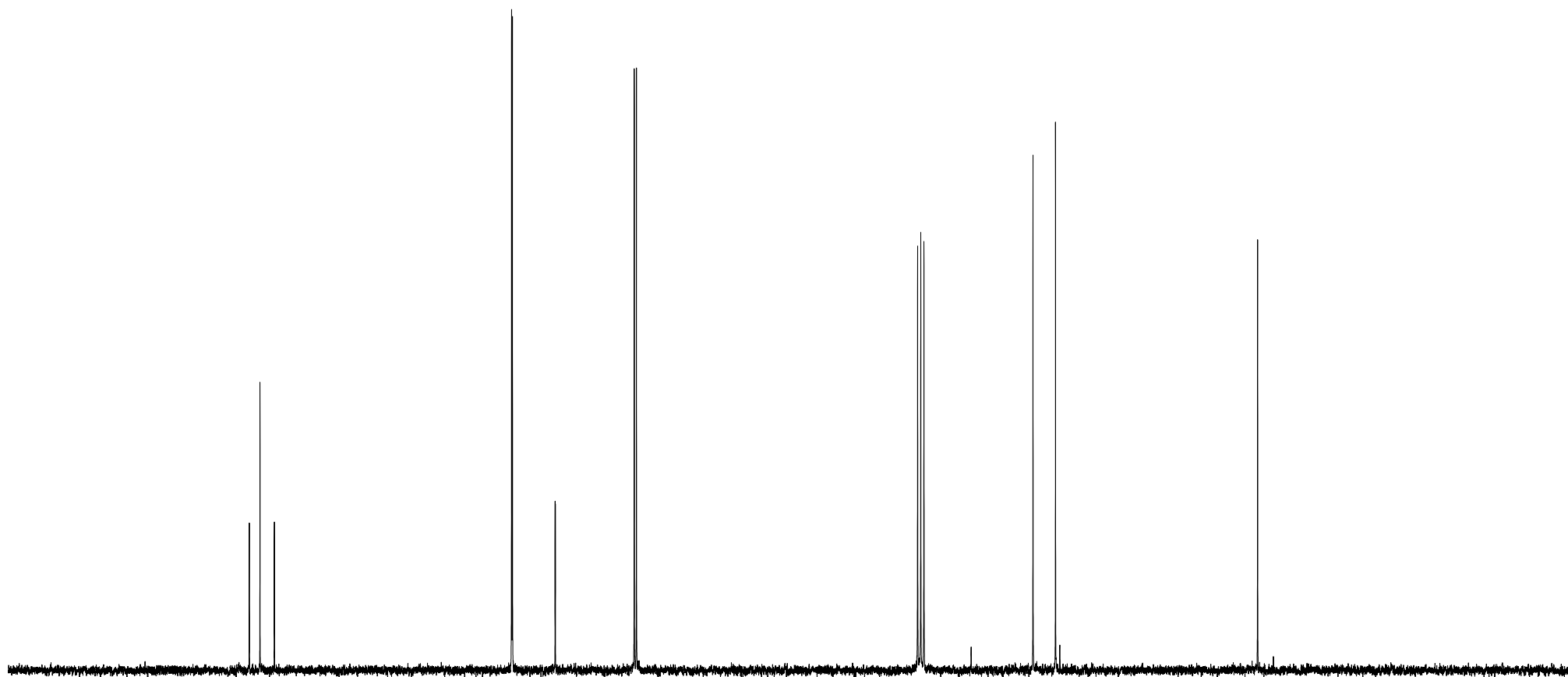
167.52
166.09
164.15

132.23
132.11
126.37
126.33

115.72
115.43

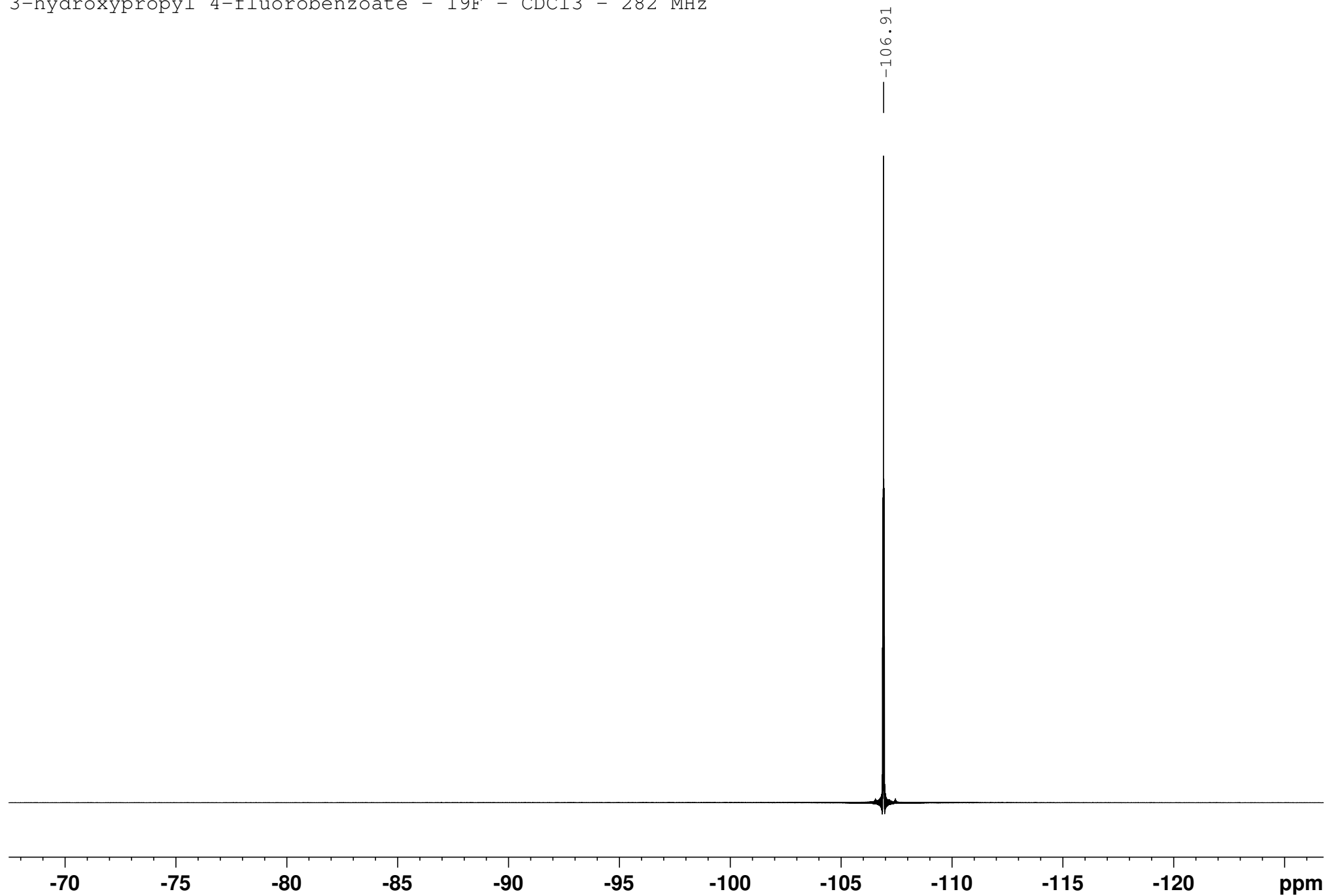
62.06
59.03

31.83

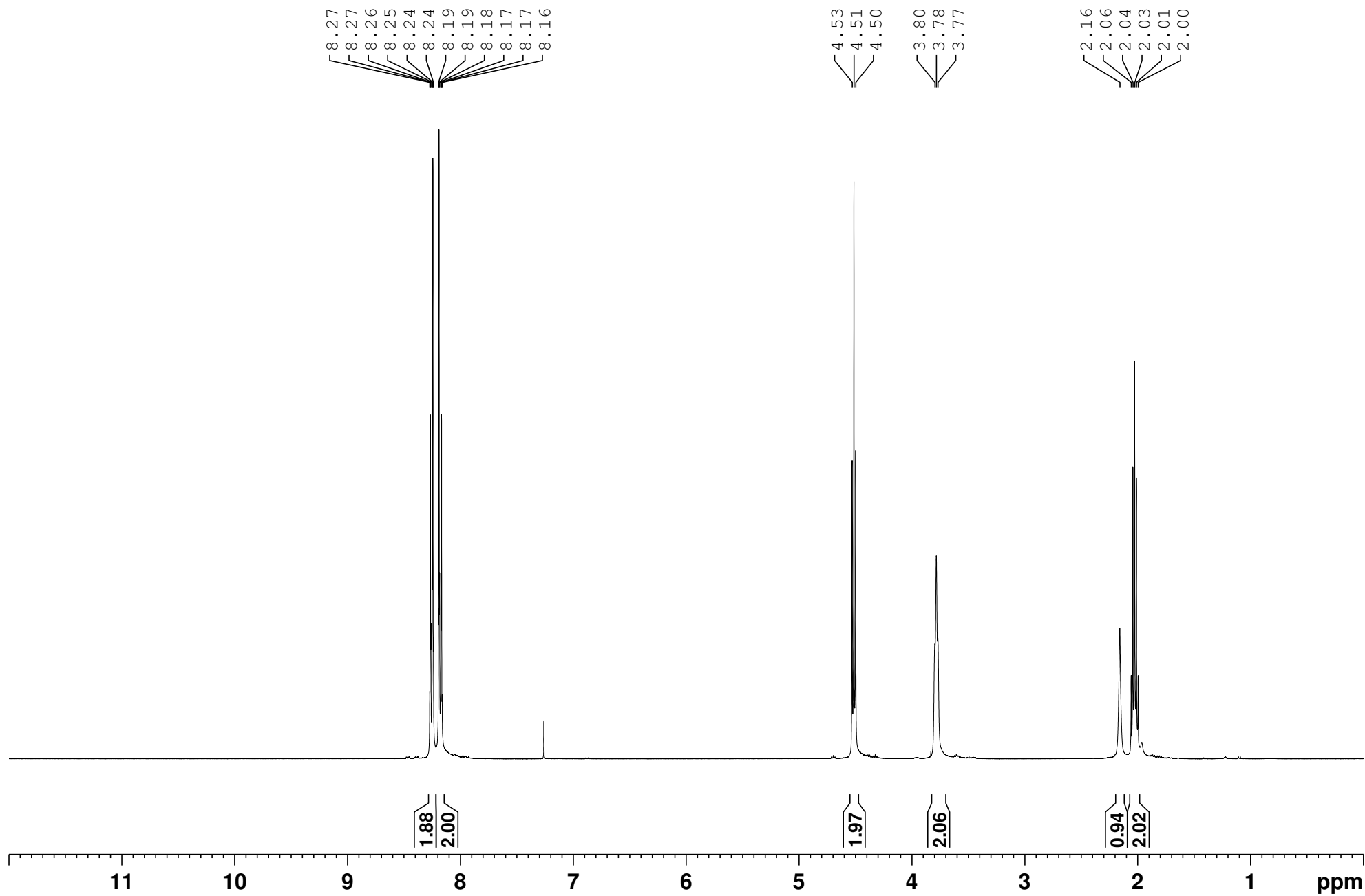


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

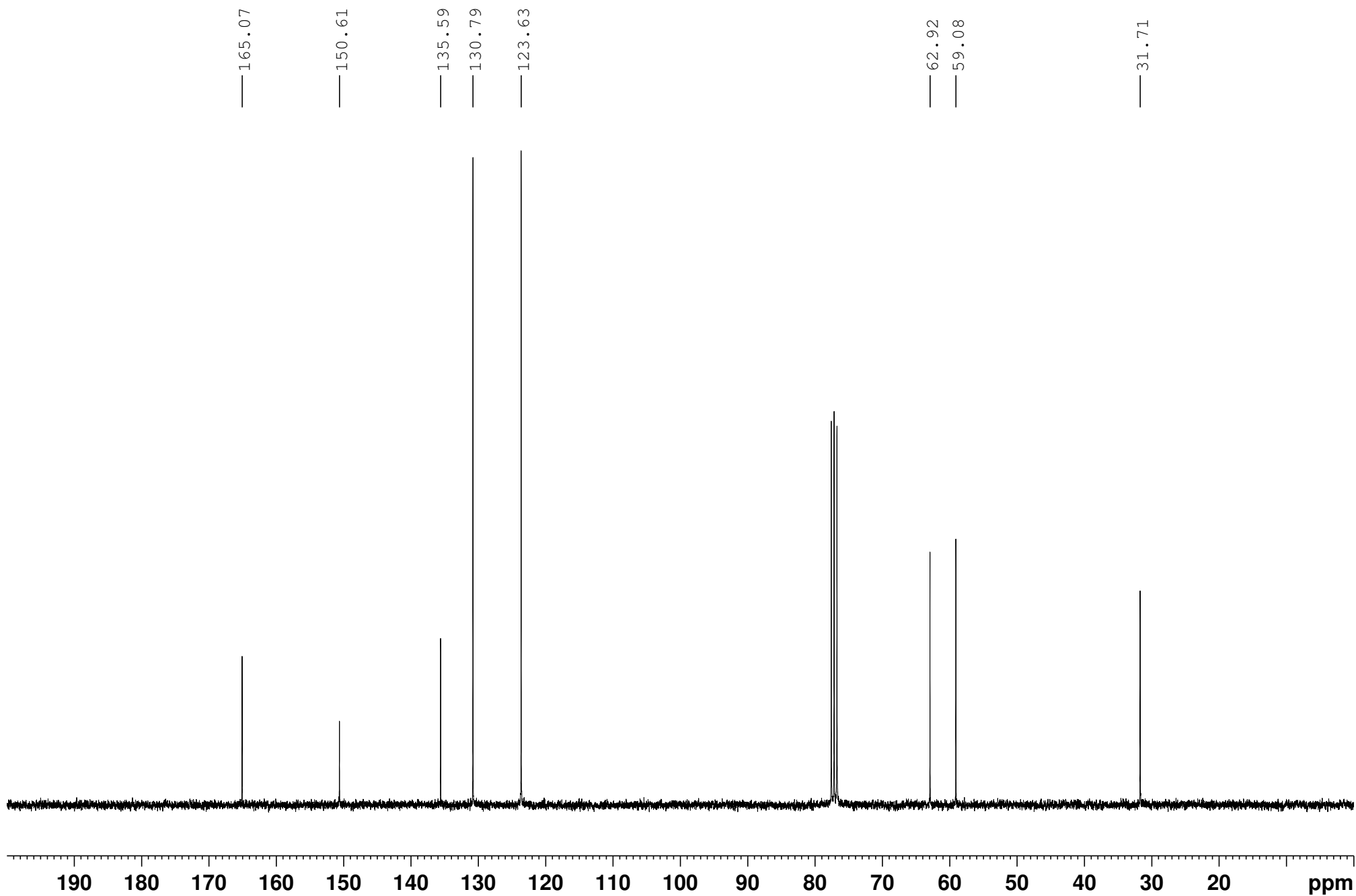
3-hydroxypropyl 4-fluorobenzoate - ^{19}F - CDCl_3 - 282 MHz



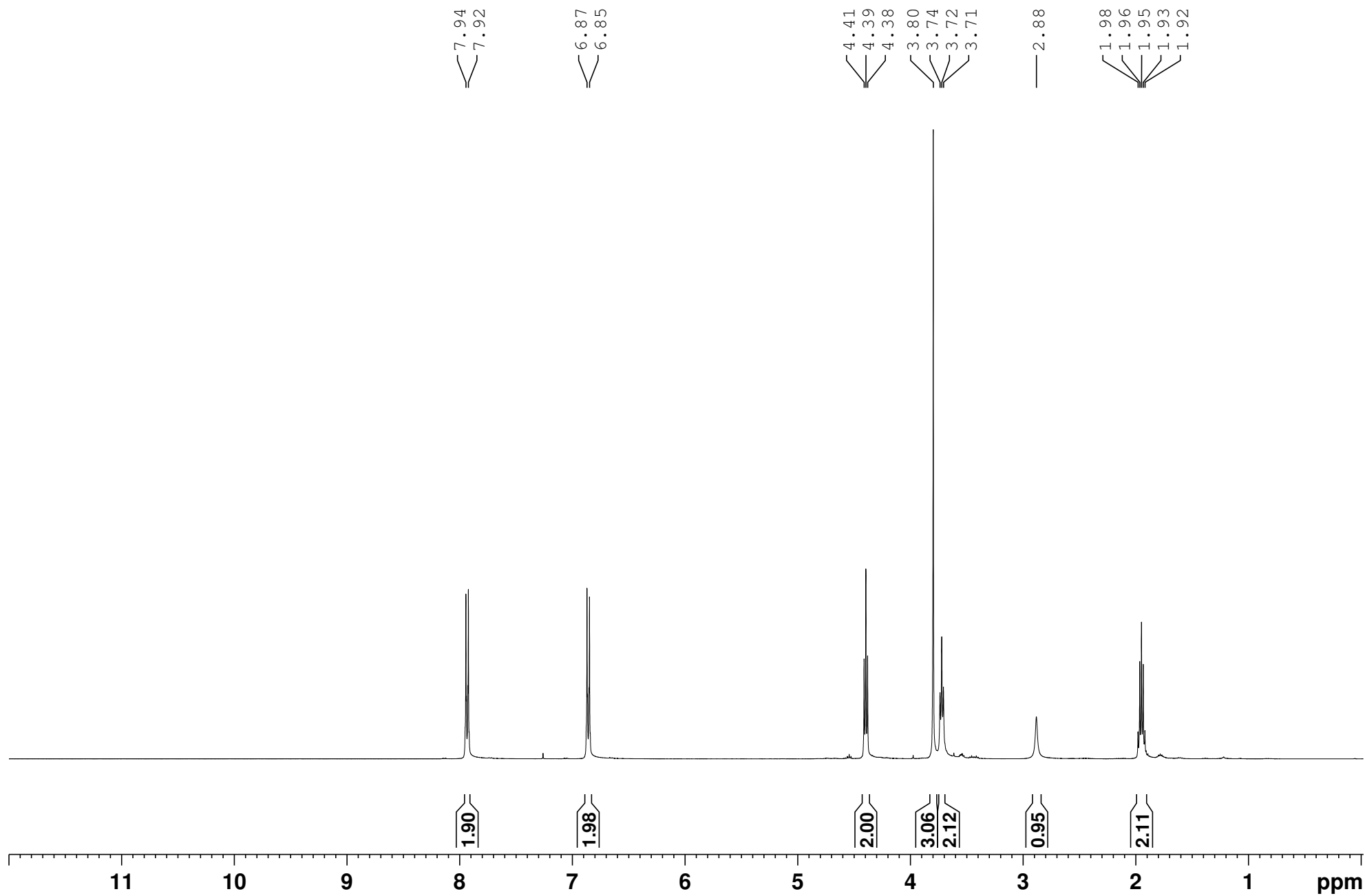
3-hydroxypropyl 4-nitrobenzoate - 1H - CDCl3 - 400 MHz



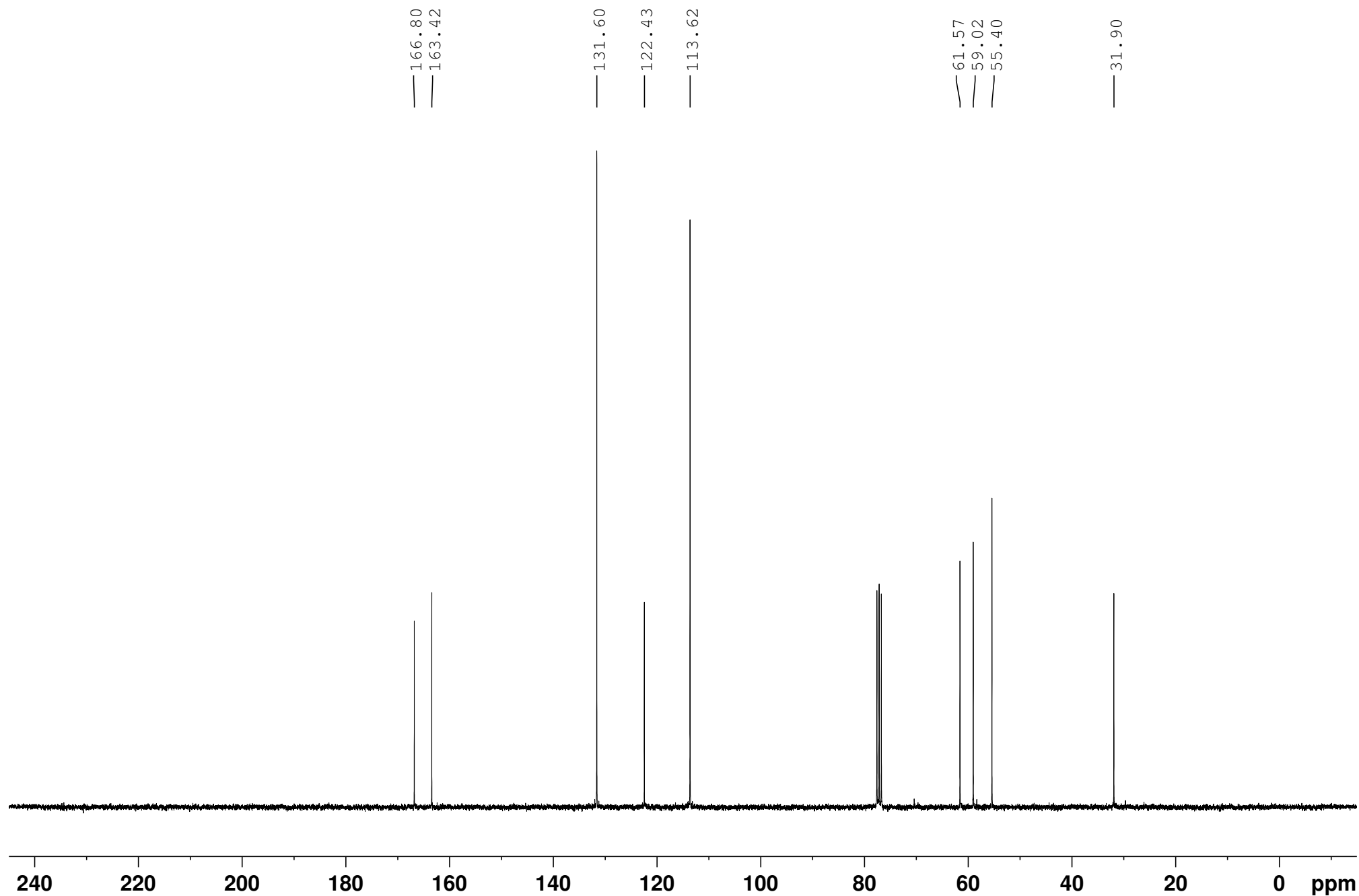
3-hydroxypropyl 4-nitrobenzoate - ^{13}C - CDCl_3 - 75 MHz



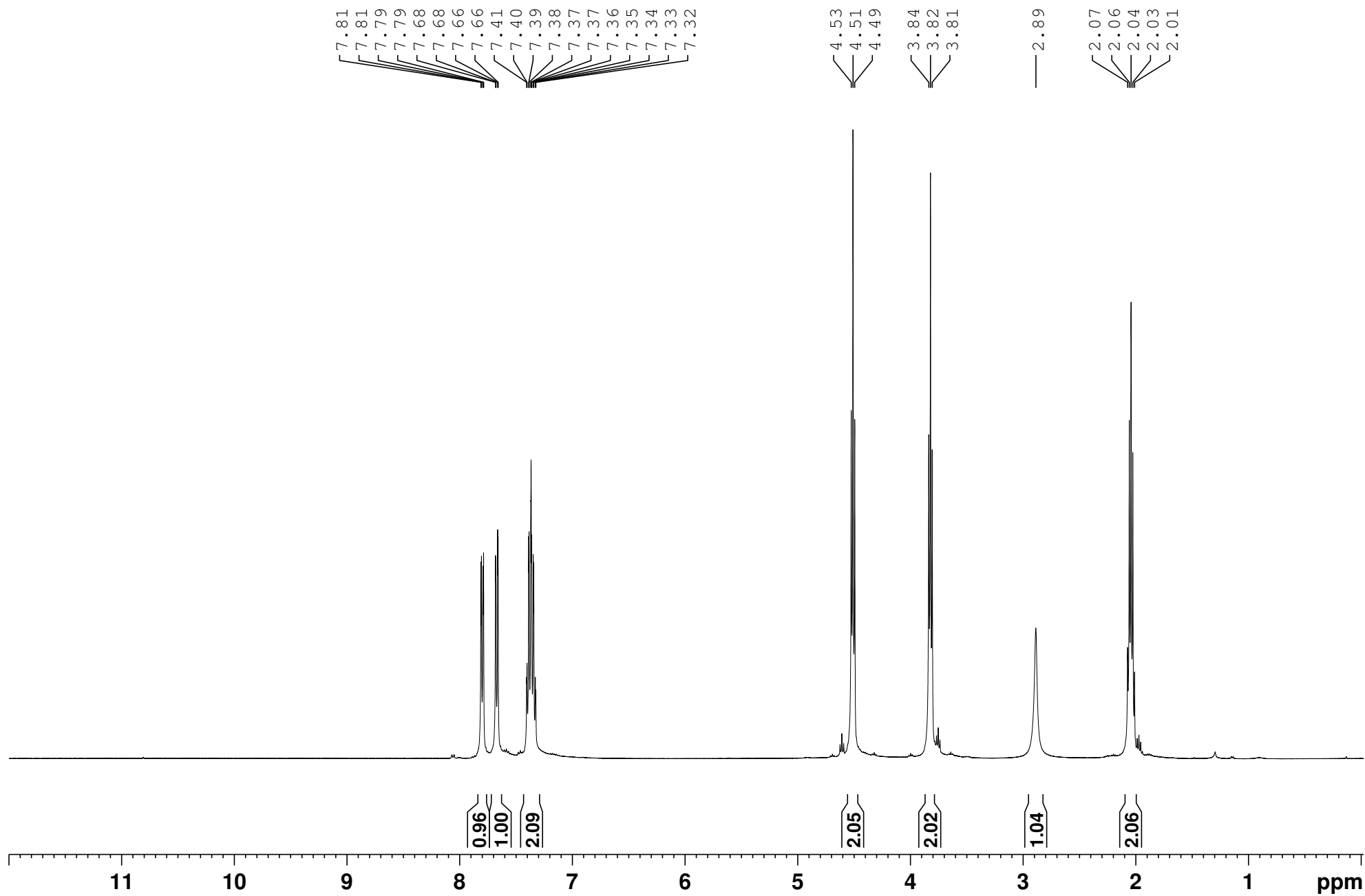
3-hydroxypropyl 4-methoxybenzoate - ¹H - CDCl₃ - 400 Mz



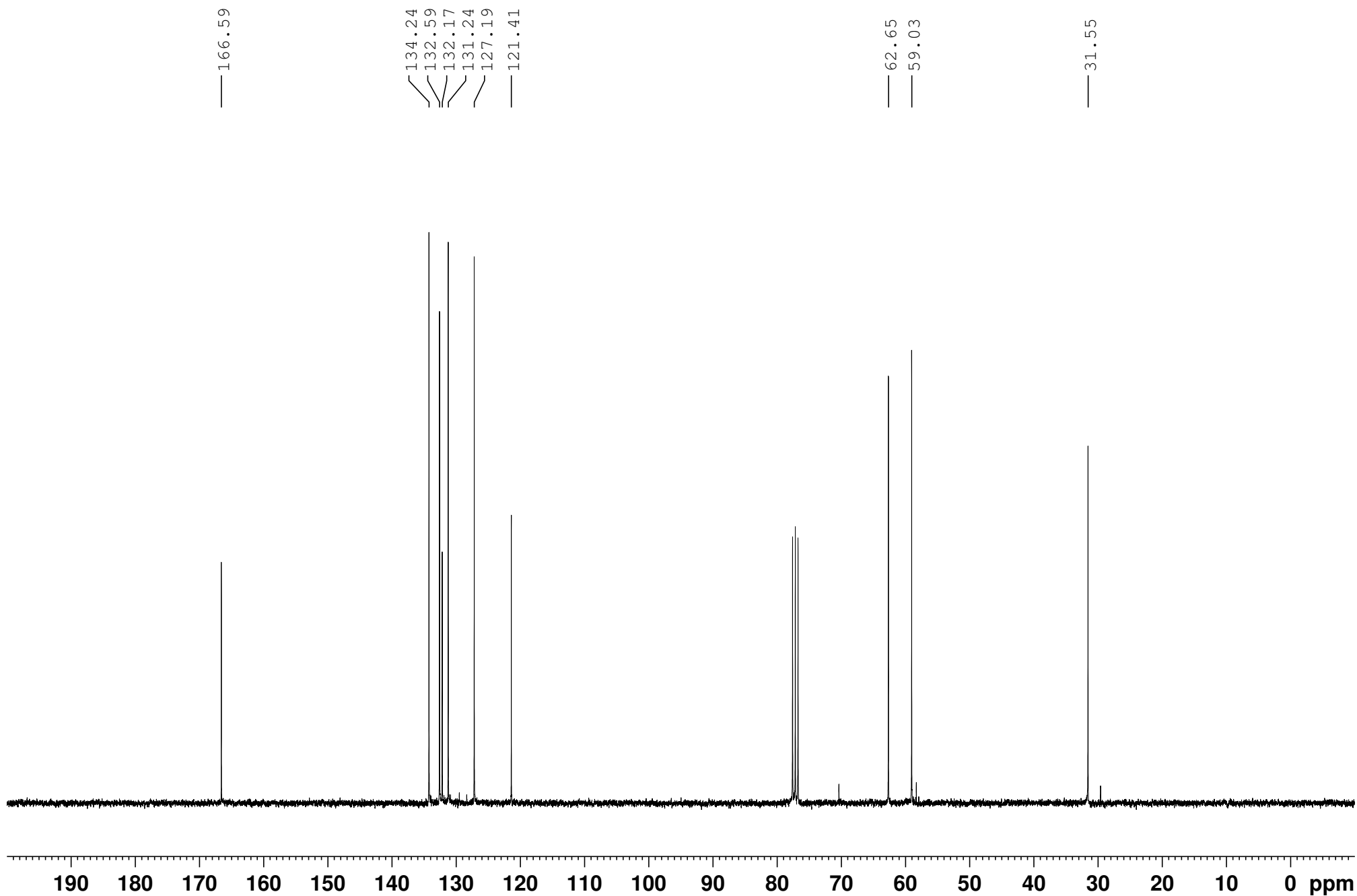
3-hydroxypropyl 4-methoxybenzoate - ¹³C - CDCl₃ - 75 MHz



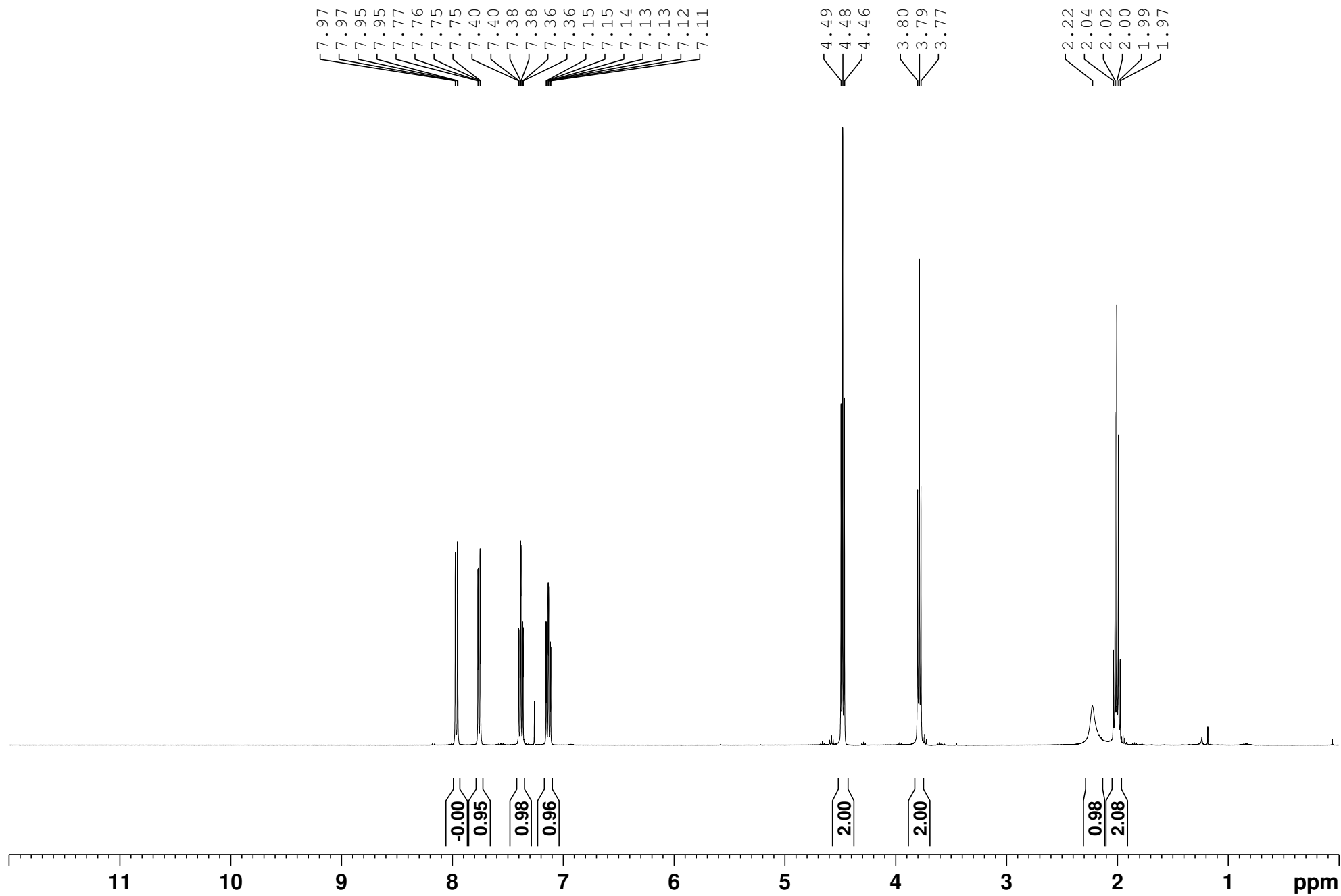
3-hydroxypropyl 2-bromobenzoate - 1H - CDCl3 - 400 MHz



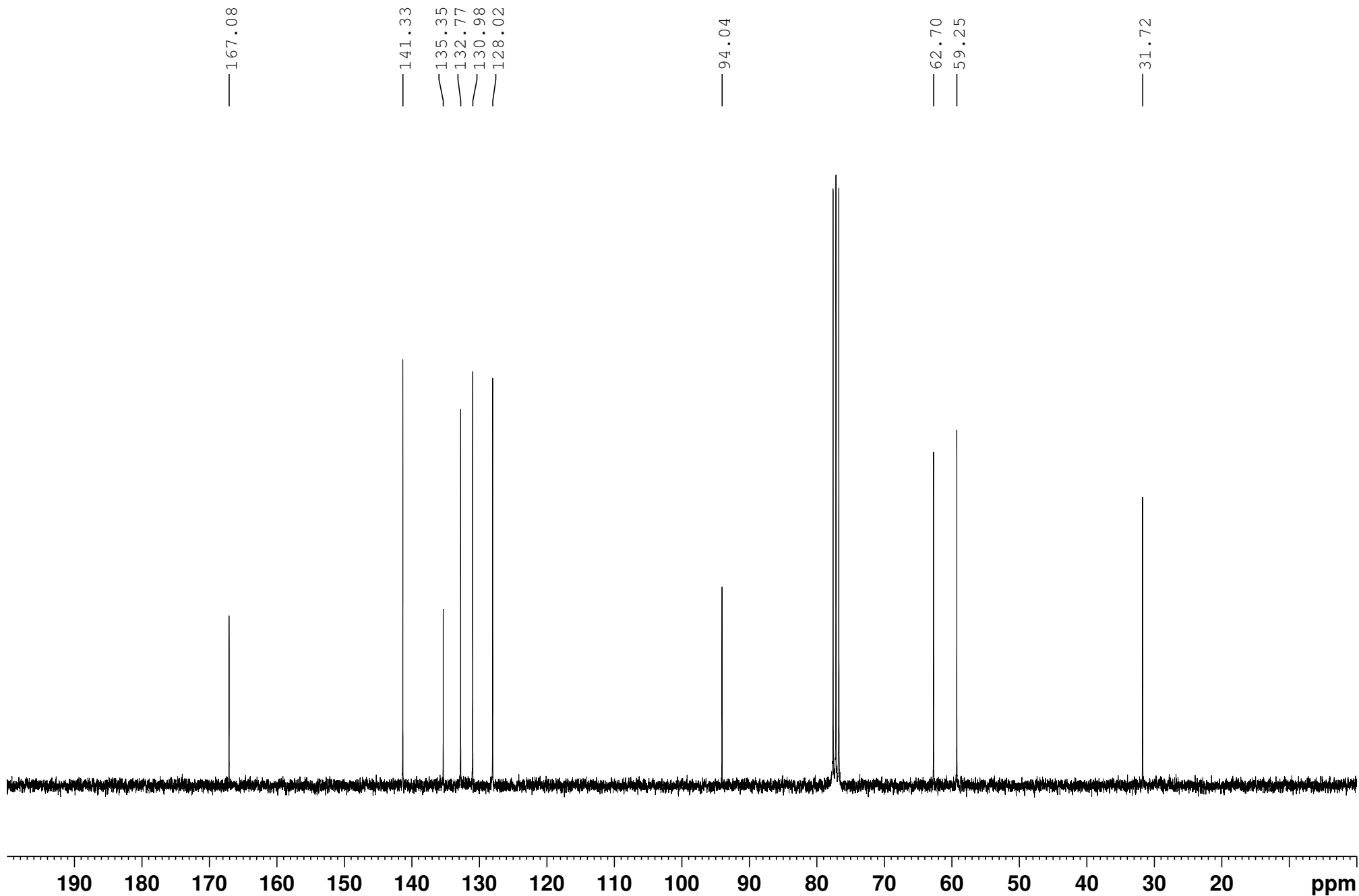
3-hydroxypropyl 2-bromobenzoate - ^{13}C - CDCl_3 - 75 MHz



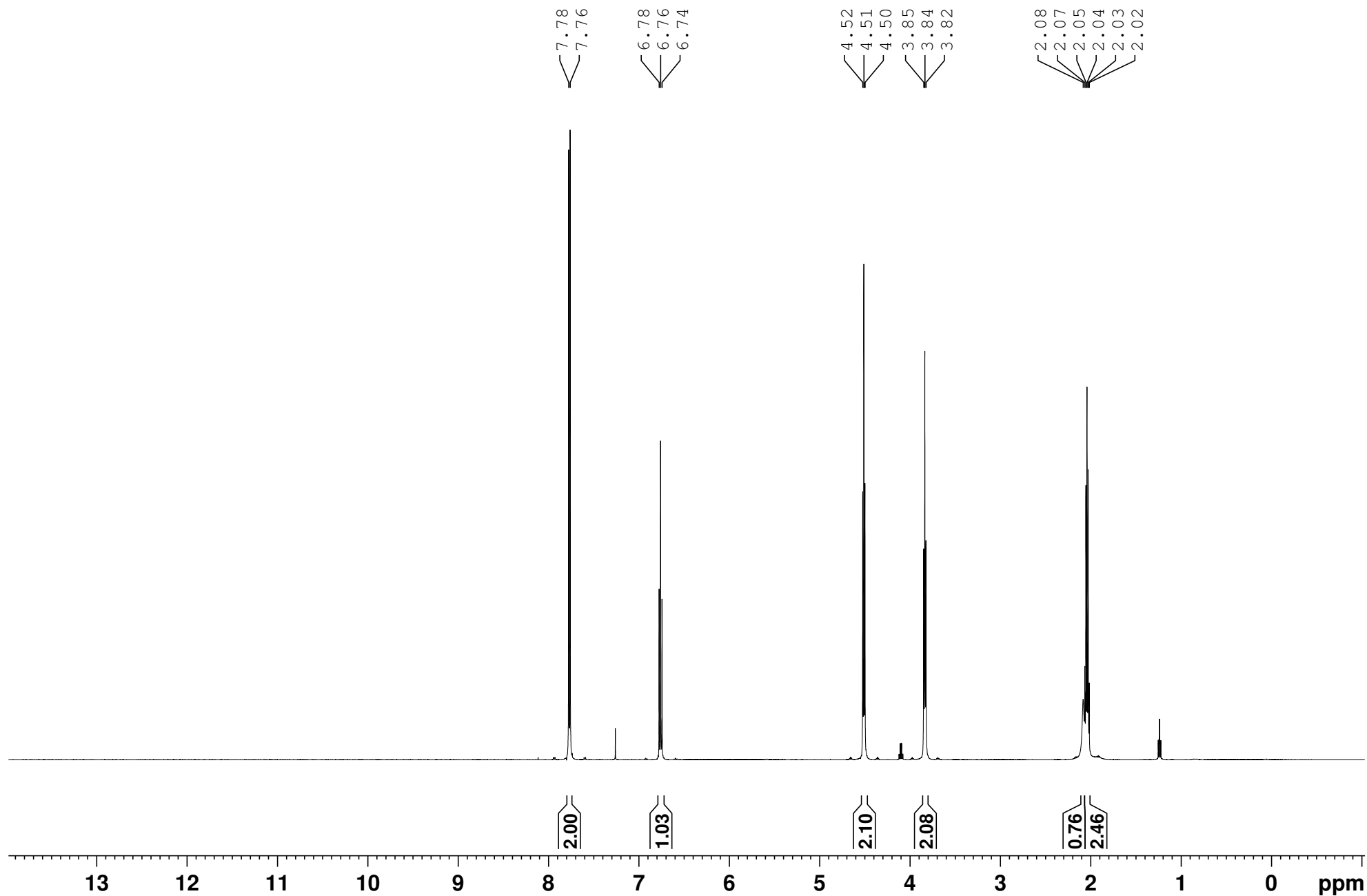
3-hydroxypropyl 2-iodobenzoate - 1H - CDCl3 - 400 MHz



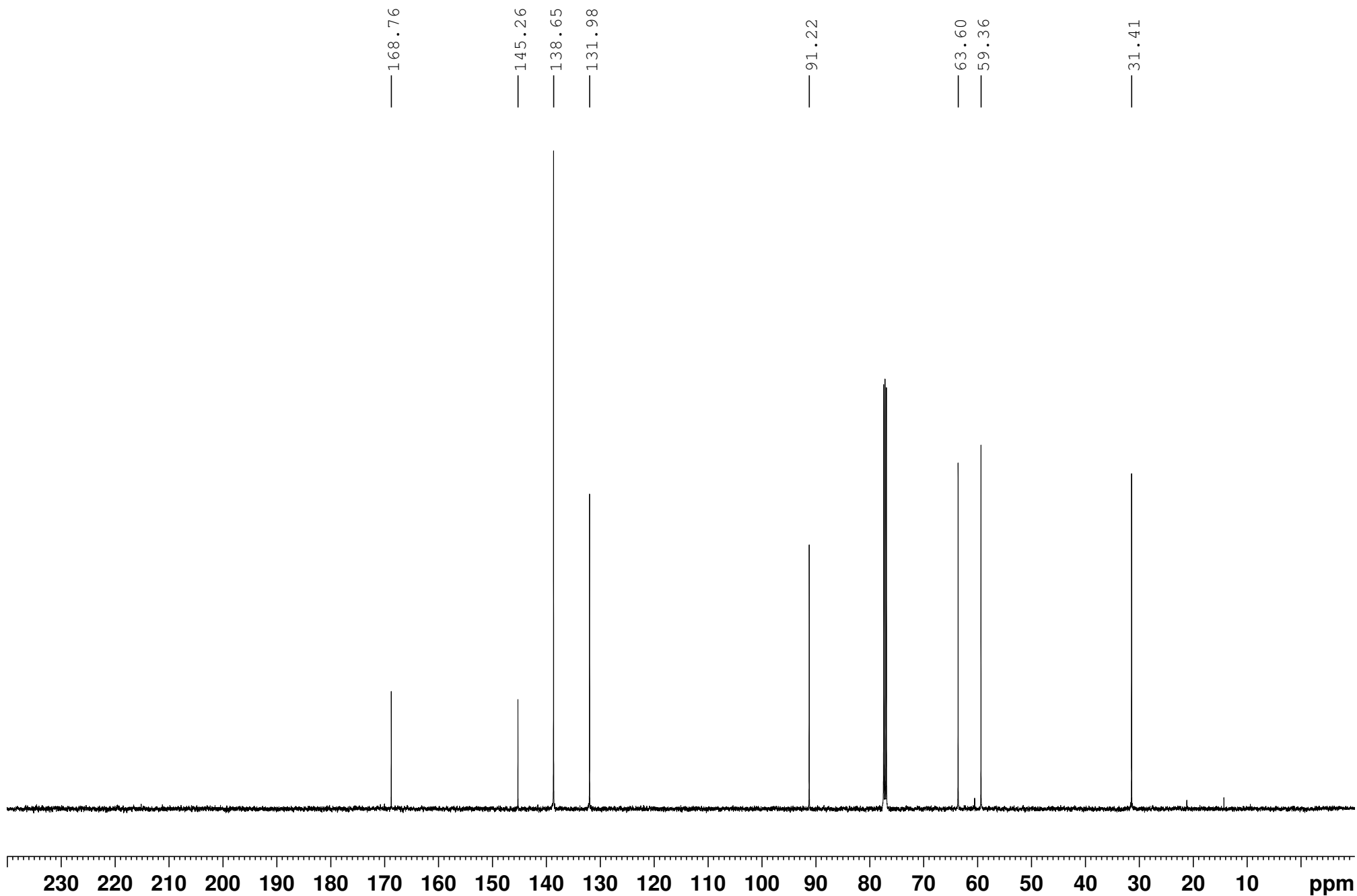
3-hydroxypropyl 2-iodobenzoate - ¹³C - CDCl₃ - 75 MHz



3-hydroxypropyl 2,6-diiodobenzoate - 1H - CDCl3 - 500 MHz



3-hydroxypropyl 2,6-diiodobenzoate - ^{13}C - CDCl_3 - 125 MHz

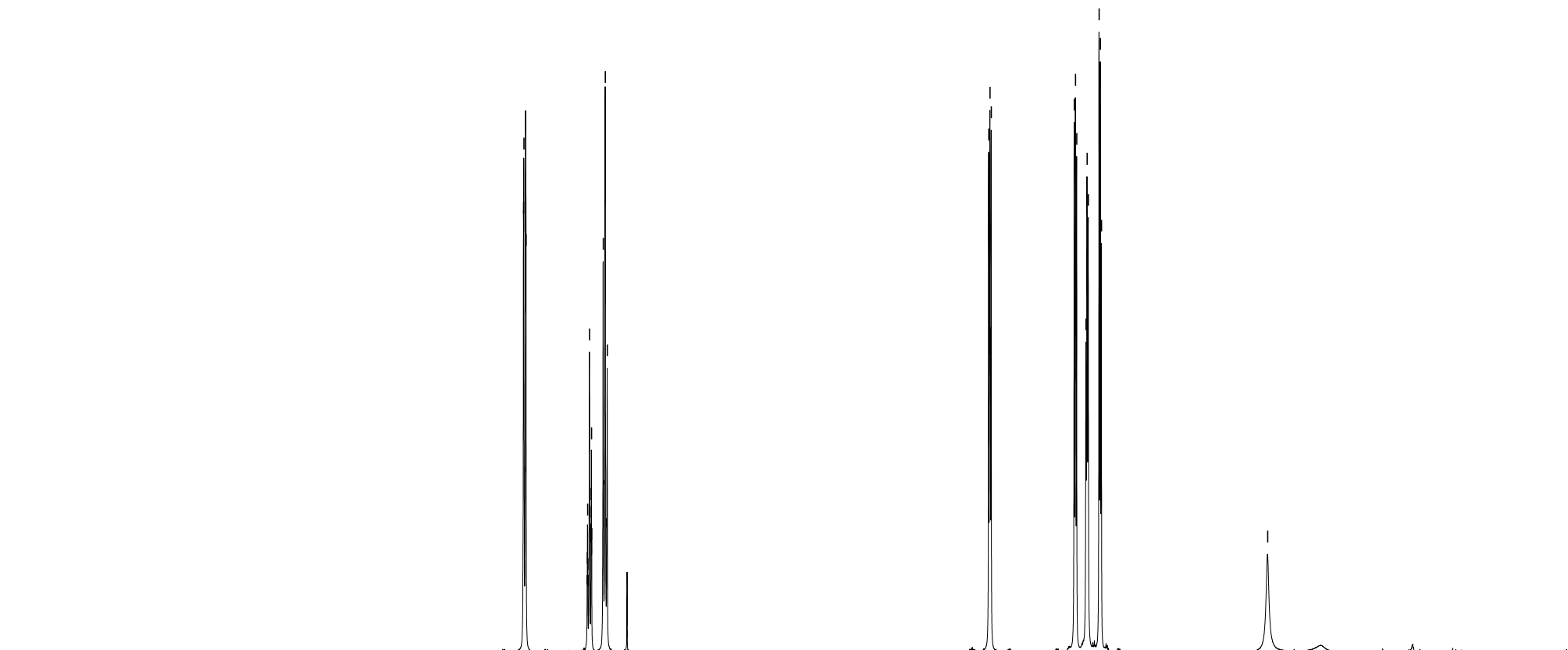


2-(2-hydroxyethoxy)ethyl benzoate - 1H - CDCl3 - 500 MHz

8.053
8.051
8.037
8.034
7.566
7.563
7.560
7.548
7.545
7.536
7.533
7.531
7.443
7.427
7.412

4.490
4.481
4.471
3.836
3.826
3.817
3.746
3.737
3.728
3.645
3.636
3.627

— 2.355



2.00

1.01

2.06

2.07

2.07

2.07

2.07

0.96

11

10

9

8

7

6

5

4

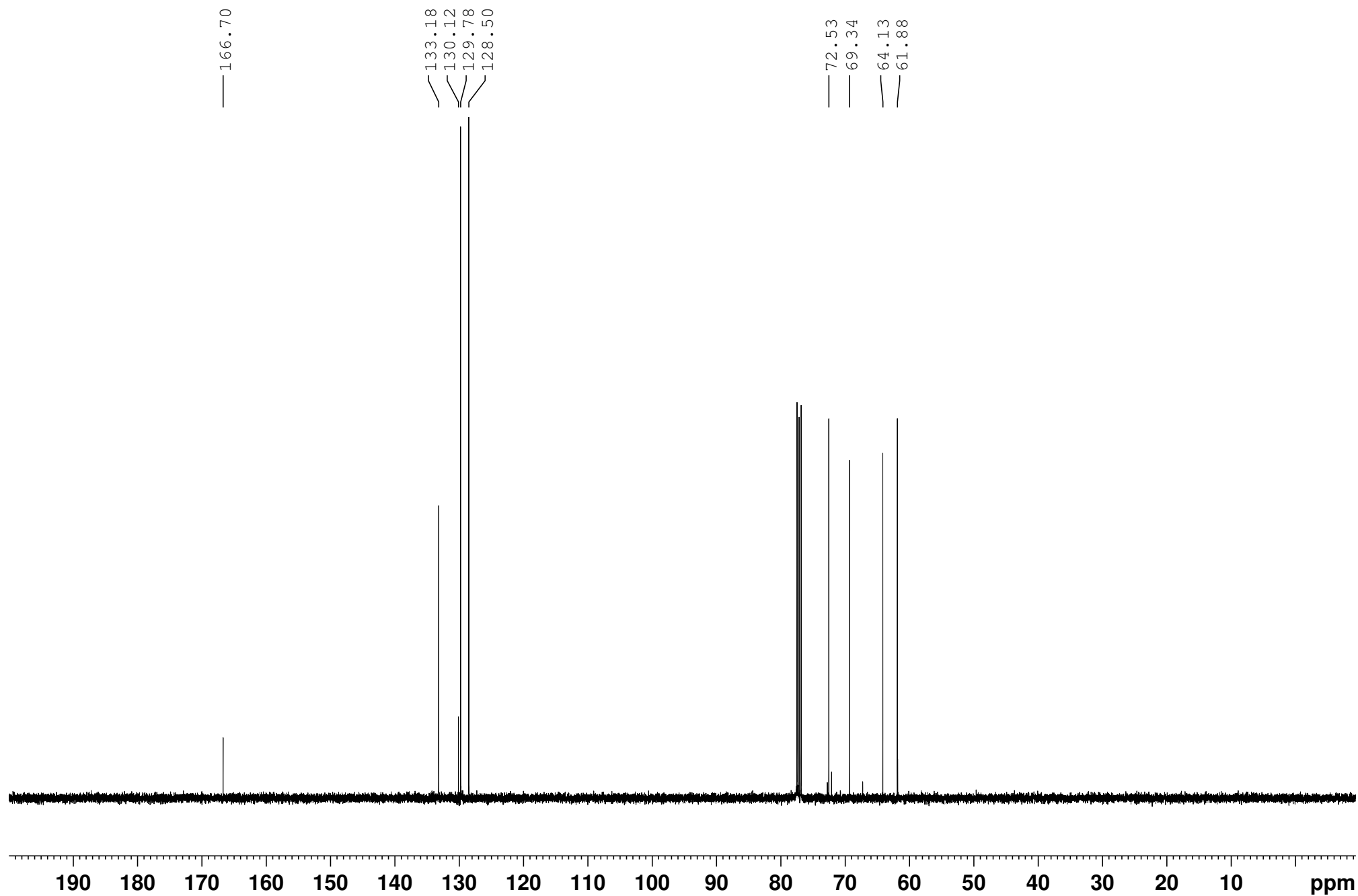
3

2

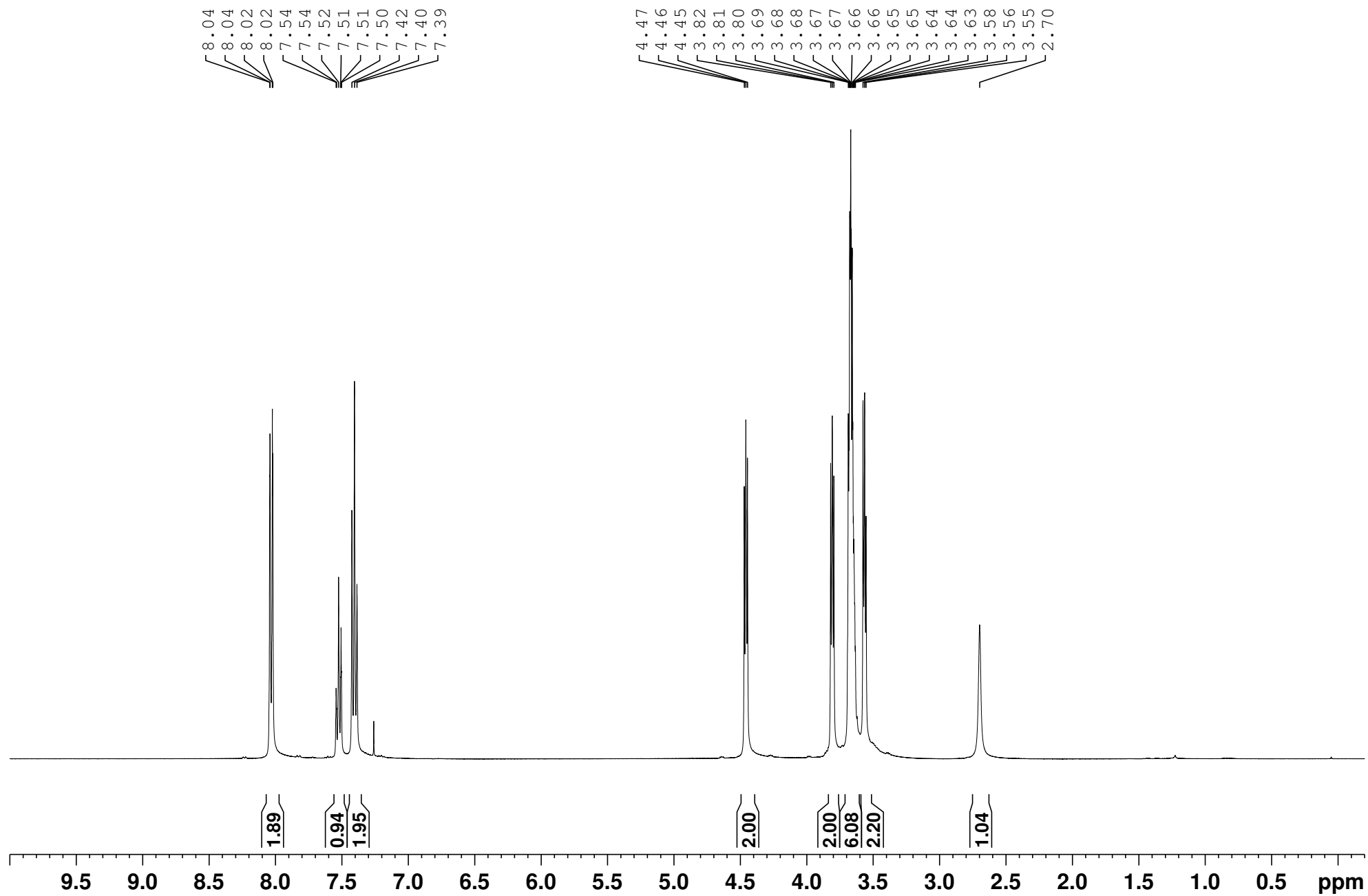
1

ppm

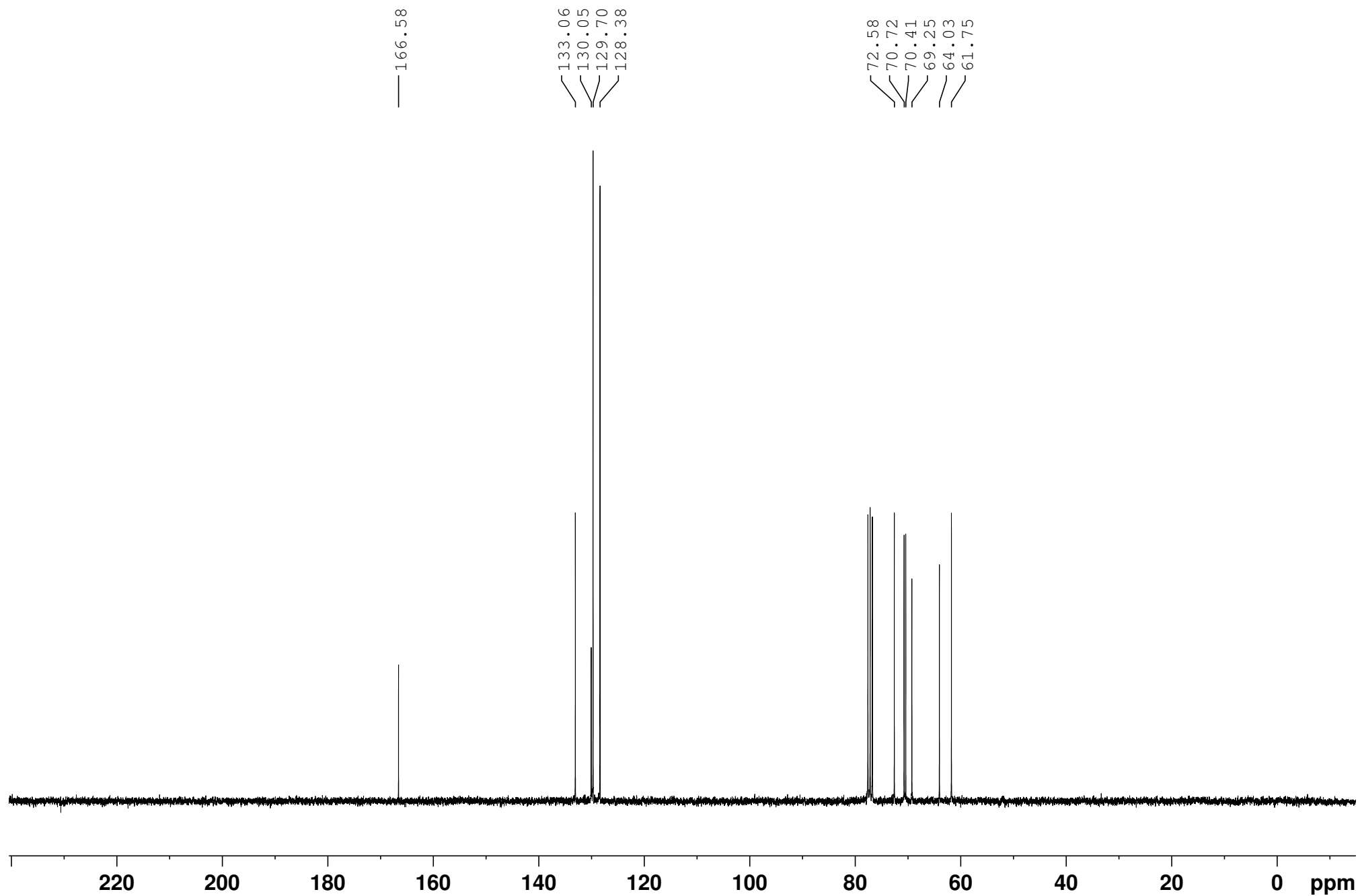
2-(2-hydroxyethoxy)ethyl benzoate - ^{13}C - CDCl_3 - 100 MHz



2-(2-(2-hydroxyethoxy)ethoxy)ethyl benzoate - 1H - CDCl3 - 400 MHz



2-(2-(2-hydroxyethoxy)ethoxy)ethyl benzoate - ^{13}C - CDCl_3 - 75 MHz



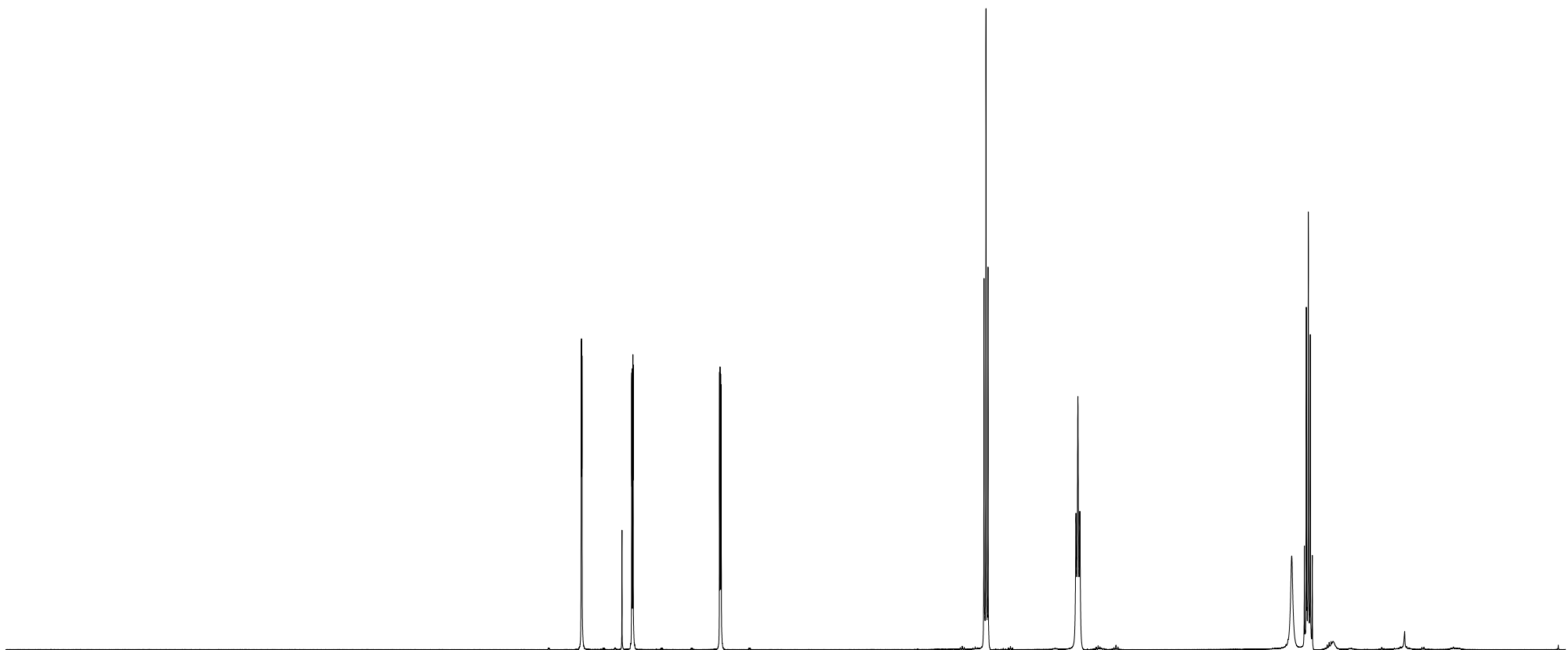
3-hydroxypropyl furan-2-carboxylate - 1H - CDCl3 - 400 MHz

7.57
7.57
7.57
7.57
7.26
7.18
7.18
7.18
7.17
6.51
6.50
6.50
6.50

4.47
4.46
4.44

3.77
3.75
3.74

2.11
2.01
1.99
1.98
1.96
1.95



11

10

9

8

7

6

5

4

3

2

1

ppm

3-hydroxypropyl furan-2-carboxylate - ^{13}C - CDCl_3 - 75 MHz

— 159.16

— 146.55

— 144.63

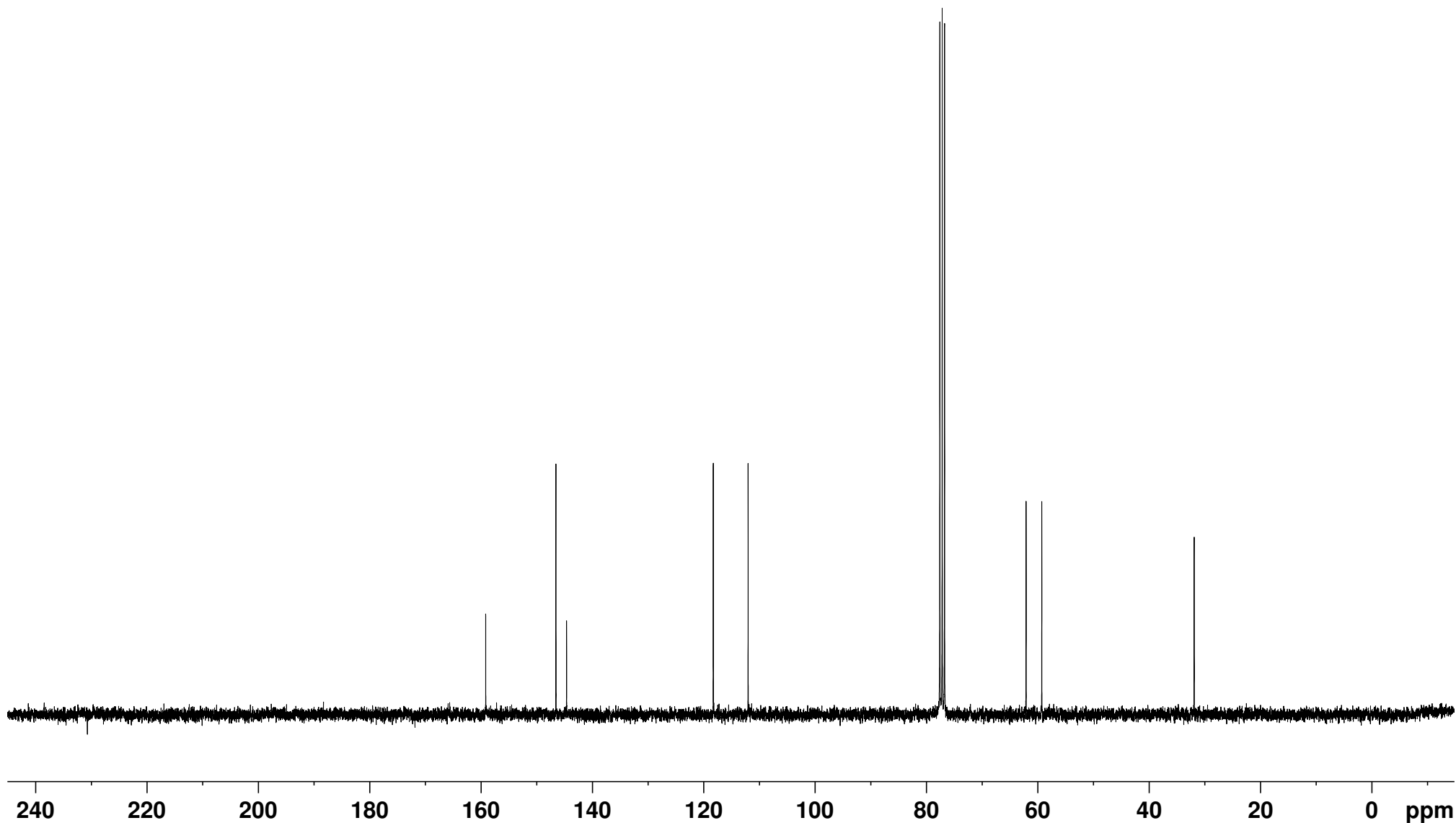
— 118.28

— 112.02

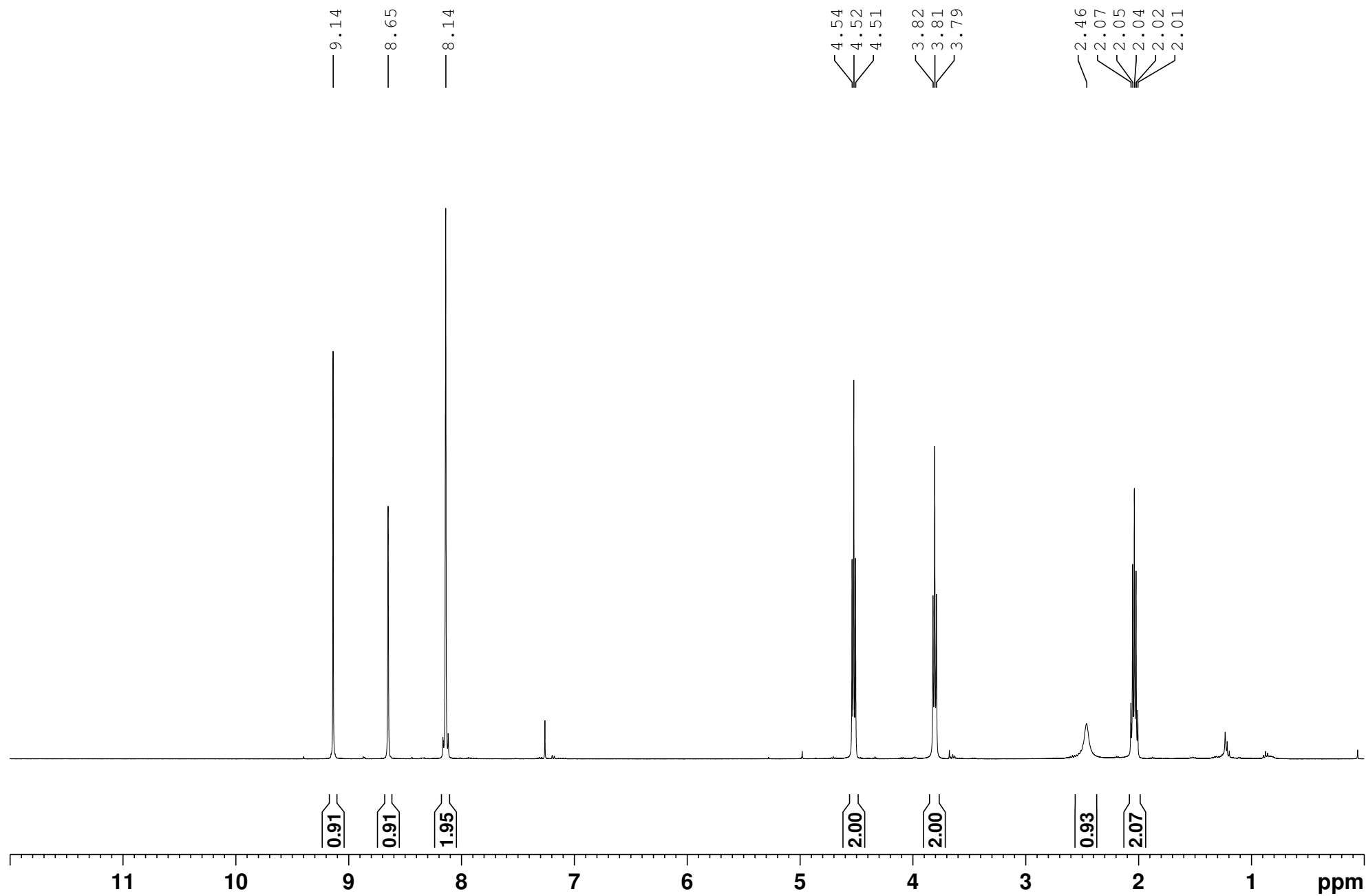
— 62.09

— 59.27

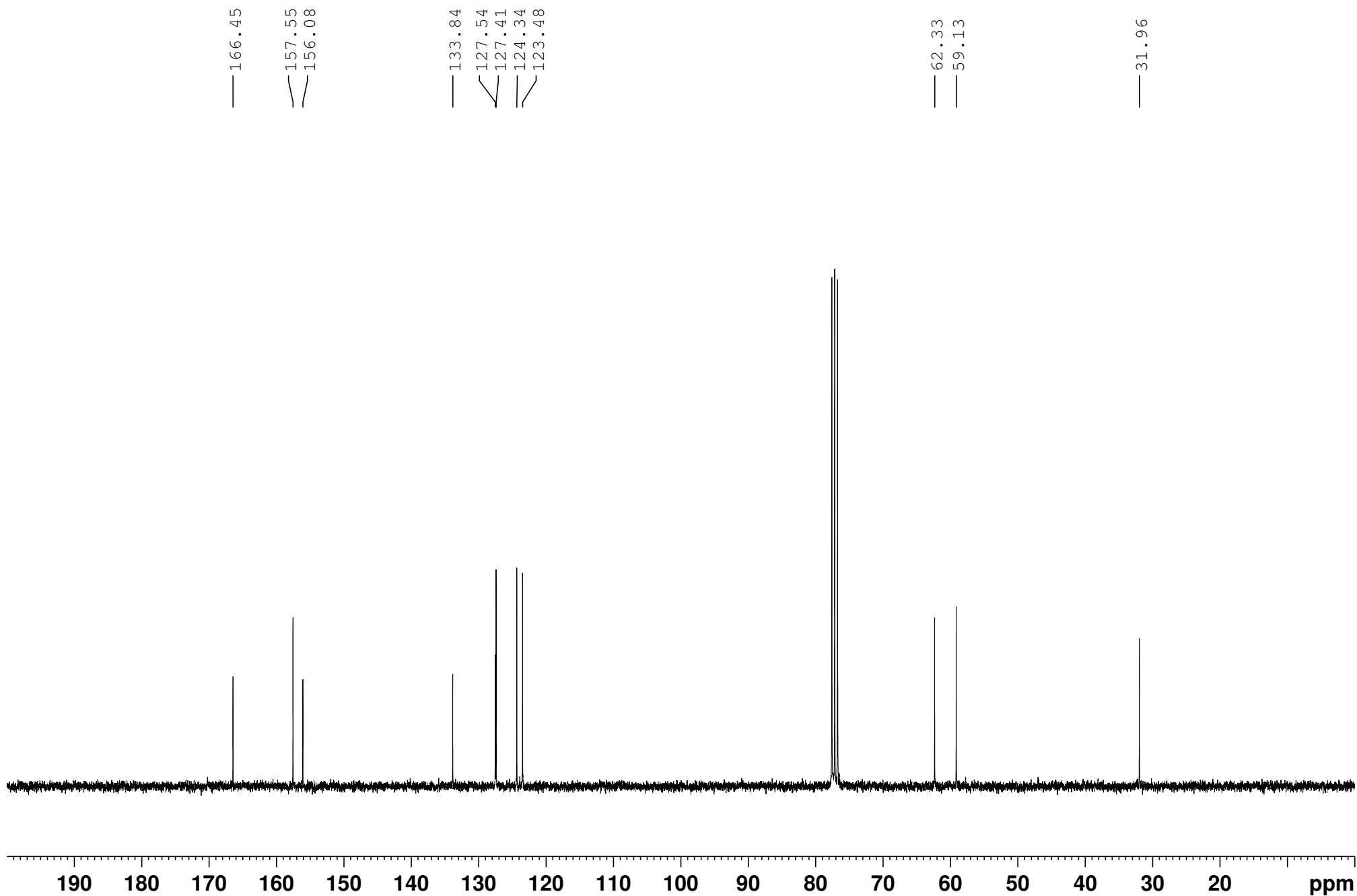
— 31.89



3-hydroxypropyl benzo[d]thiazole-6-carboxylate - 1H - CDCl3 - 400 MHz



3-hydroxypropyl benzo[d]thiazole-6-carboxylate - ^{13}C - CDCl_3 - 75 MHz



3-hydroxypropyl thiazole-4-carboxylate - 1H - CDCl3 - 400 MHz

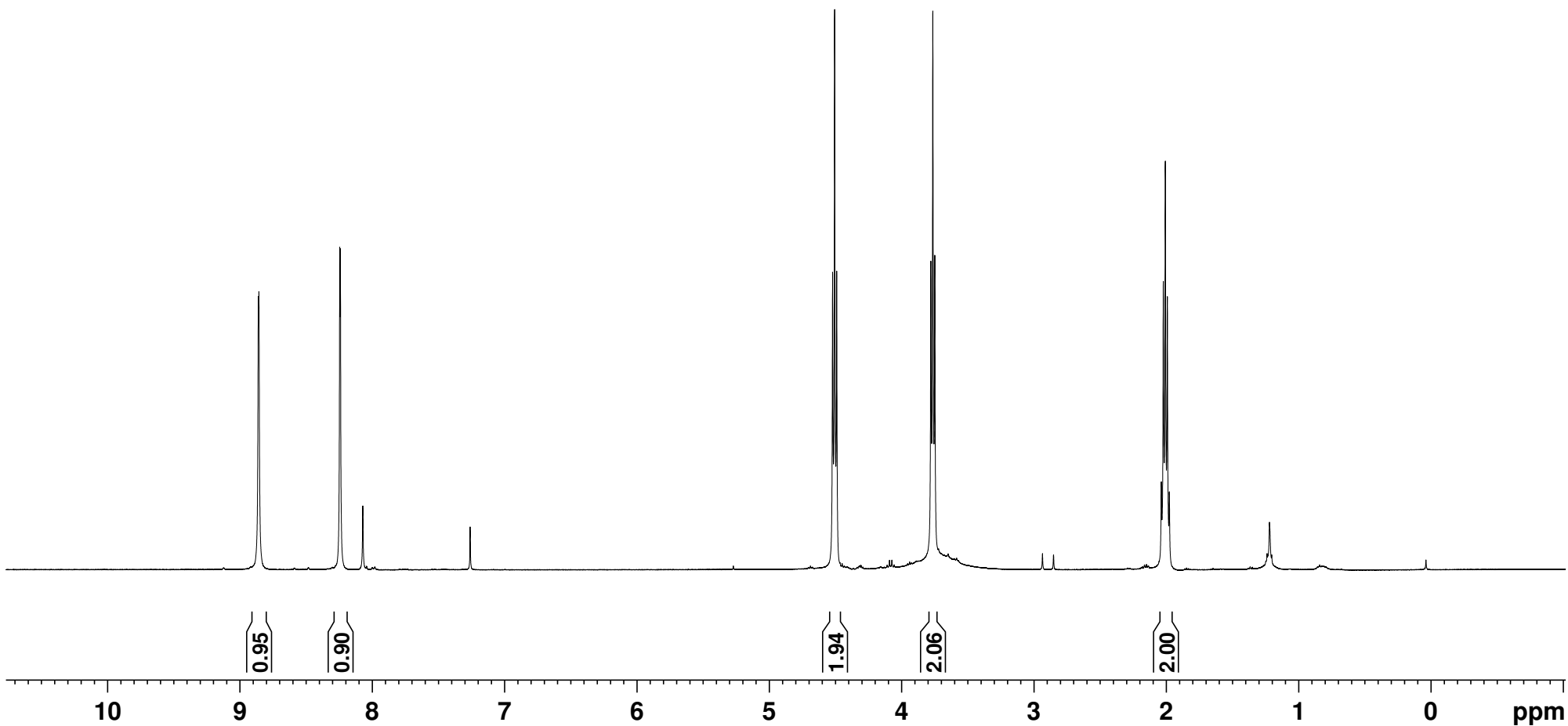
8.86
8.86

8.25
8.24

4.52
4.51
4.49

3.78
3.76
3.75

2.04
2.02
2.01
1.99
1.98



3-hydroxypropyl thiazole-4-carboxylate - ¹³C - CDCl₃ - 75 MHz

— 161.70

— 153.68

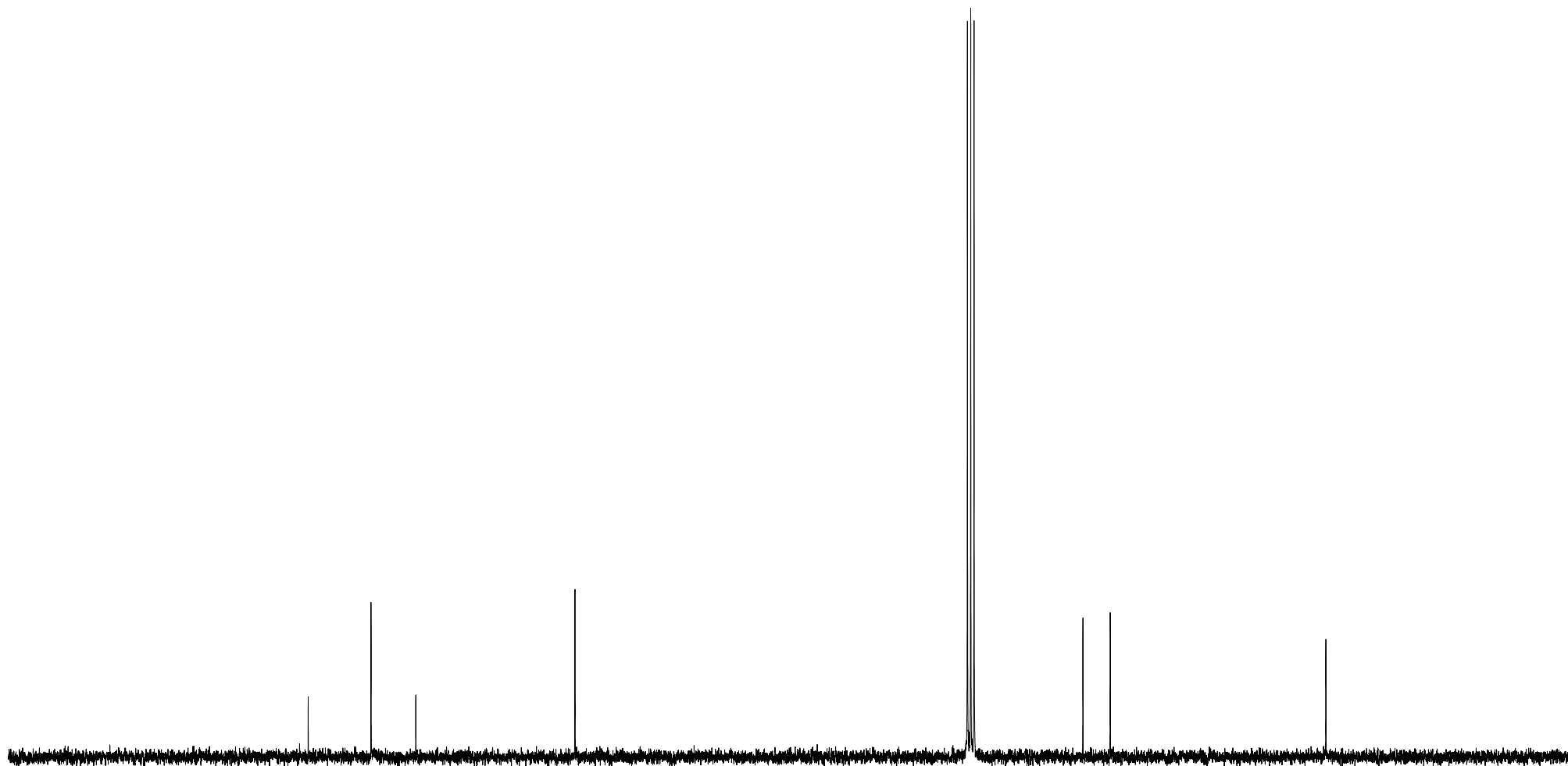
— 147.97

— 127.66

— 62.85

— 59.36

— 31.85



190

180

170

160

150

140

130

120

110

100

90

80

70

60

50

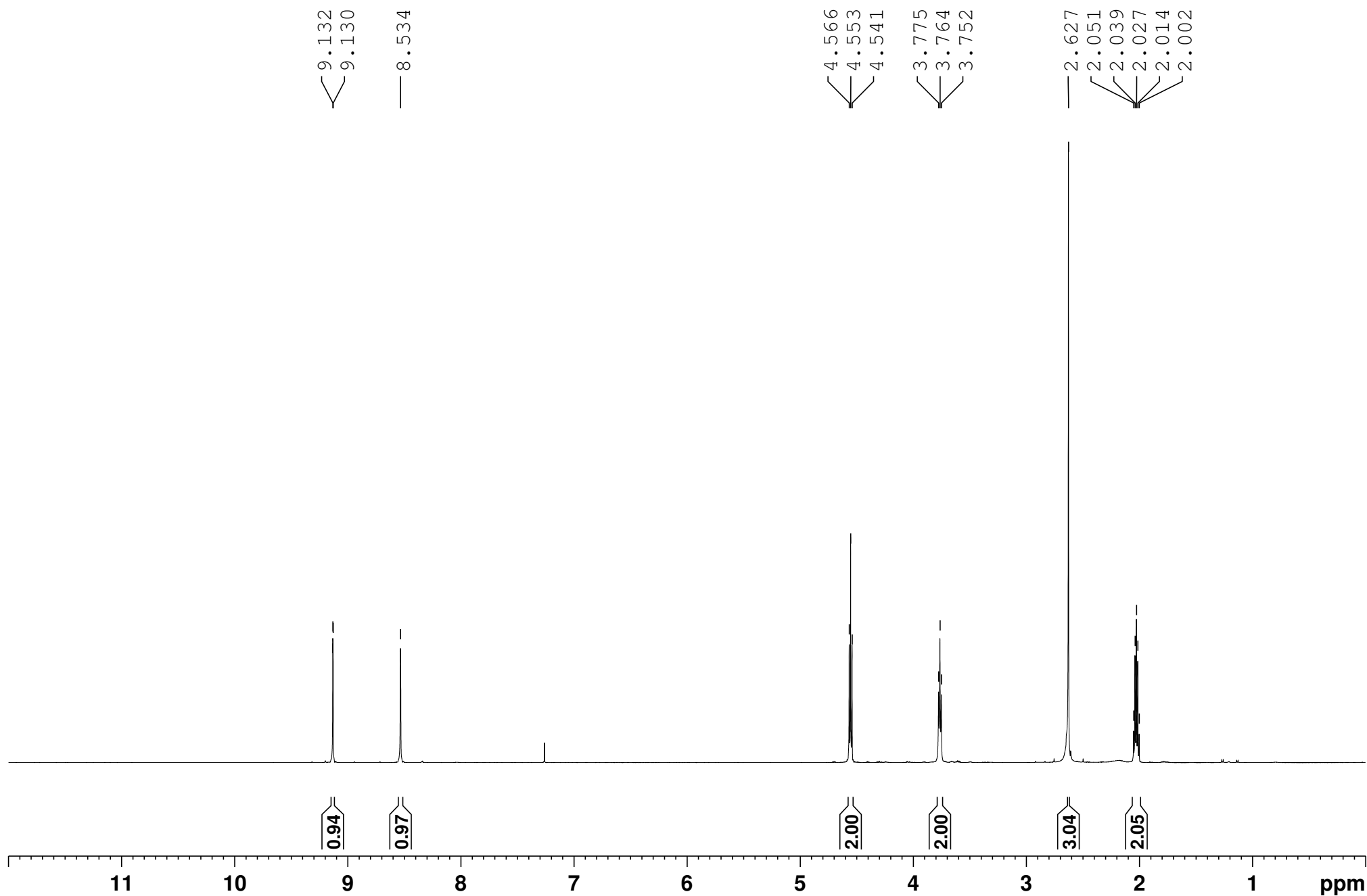
40

30

20

ppm

3-hydroxypropyl 5-methylpyrazine-2-carboxylate - 1H - CDCl3 - 500 MHz



3-hydroxypropyl 5-methylpyrazine-2-carboxylate - ¹³C - CDCl₃ - 125 MHz

— 164.443

— 158.025

— 145.382

— 144.348

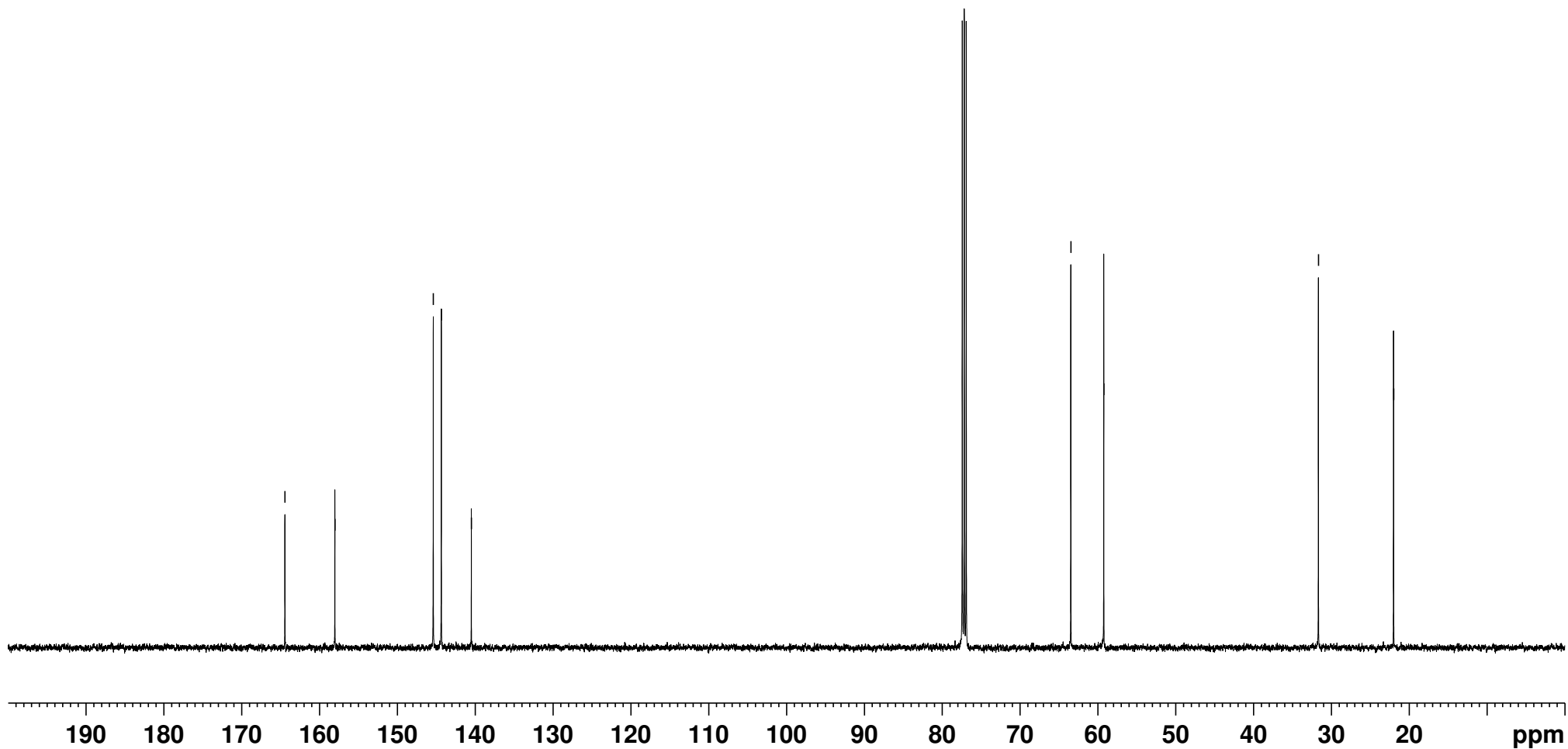
— 140.491

— 63.468

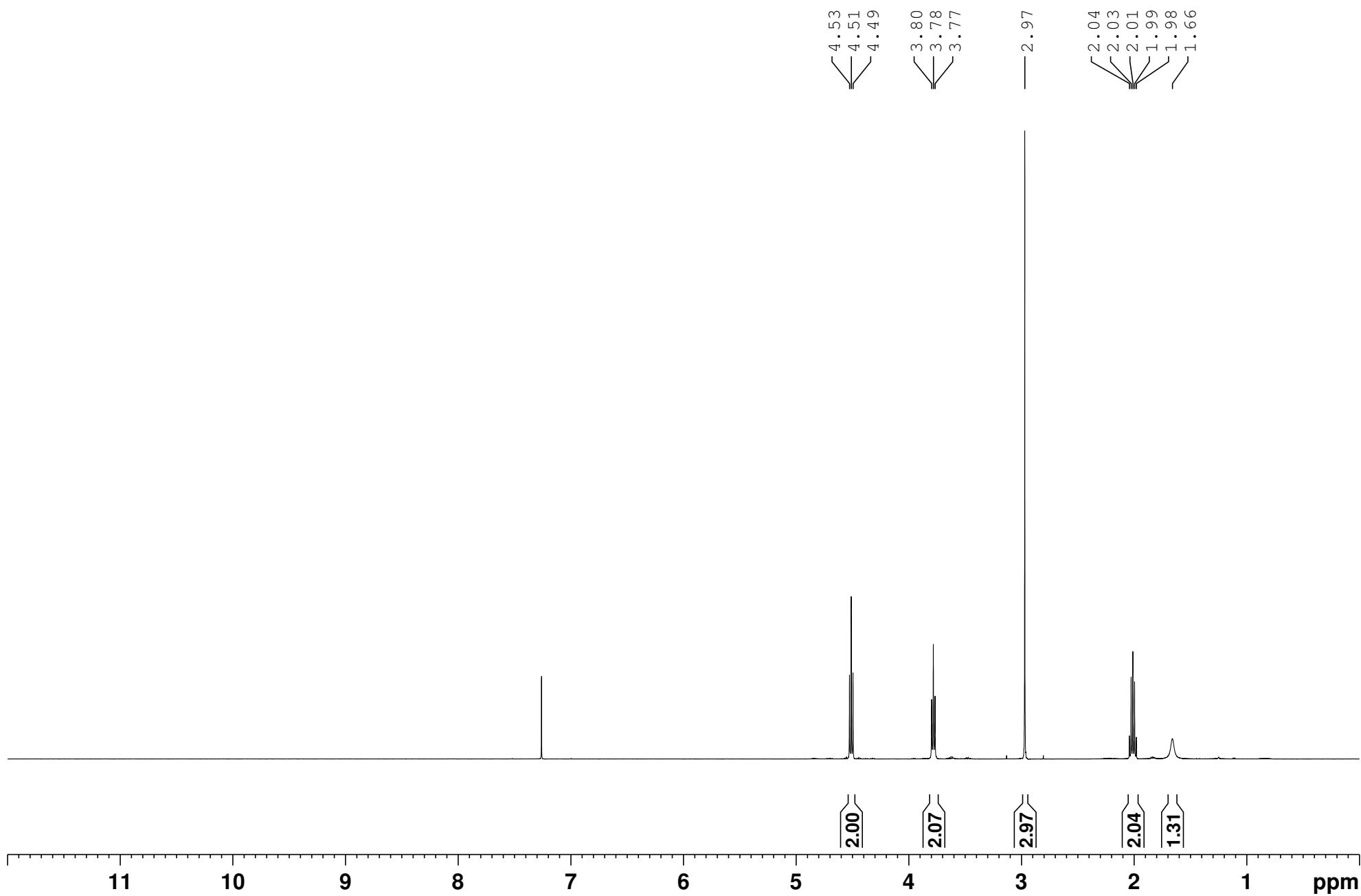
— 59.235

— 31.658

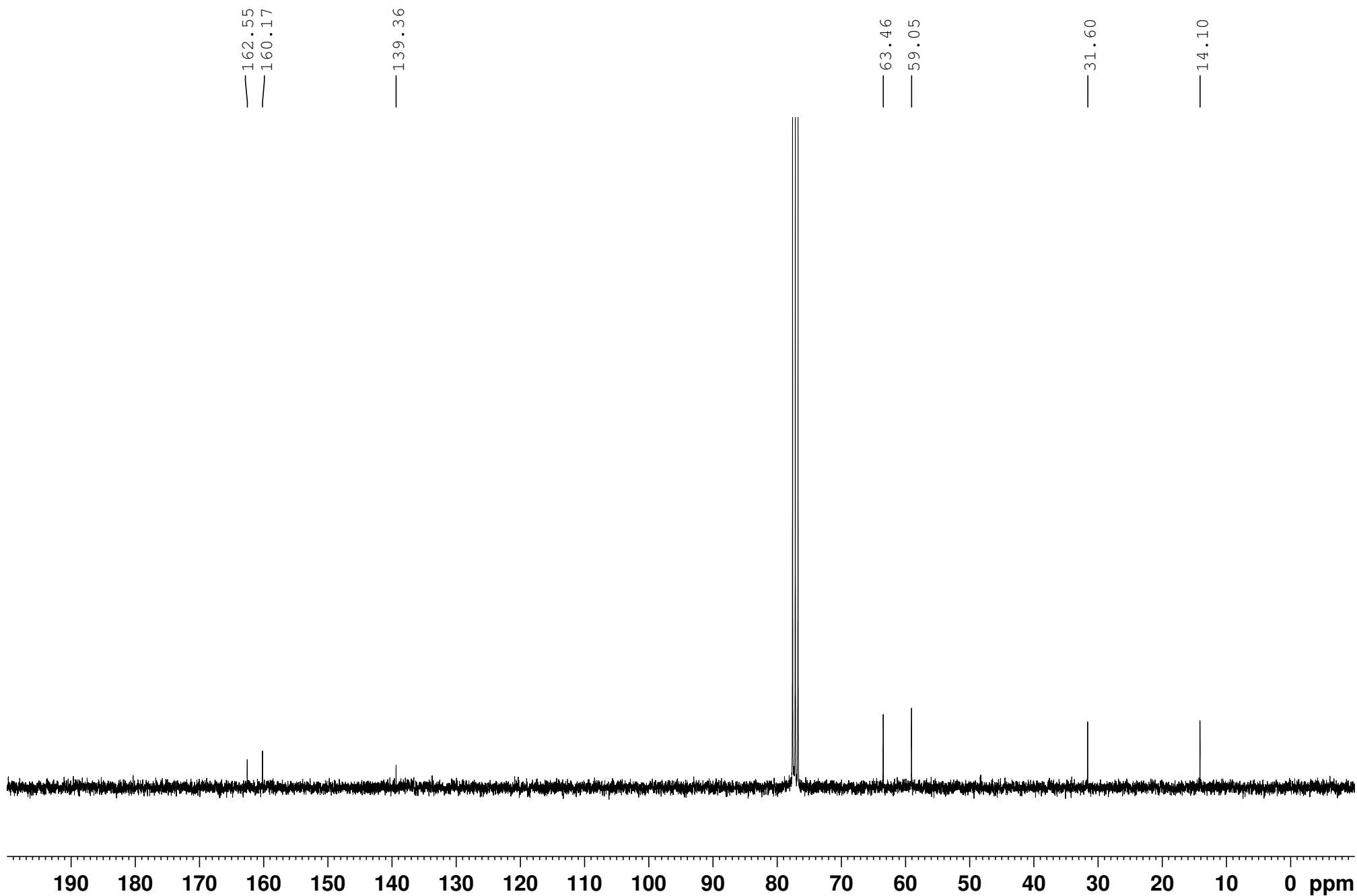
— 22.001



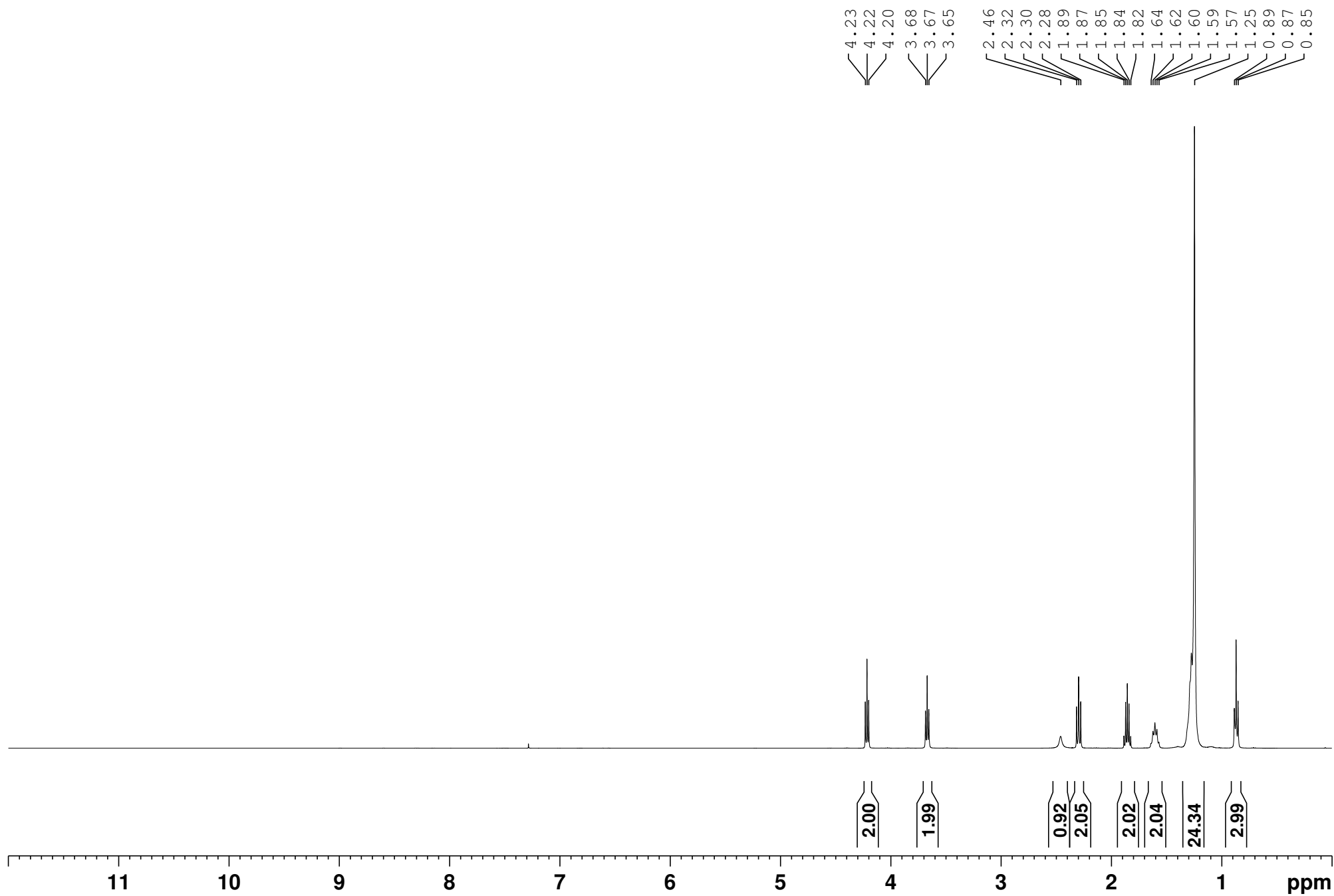
3-hydroxypropyl 4-methyl-1,2,3-thiadiazole-5-carboxylate - 1H - CDCl3 - 400 MH



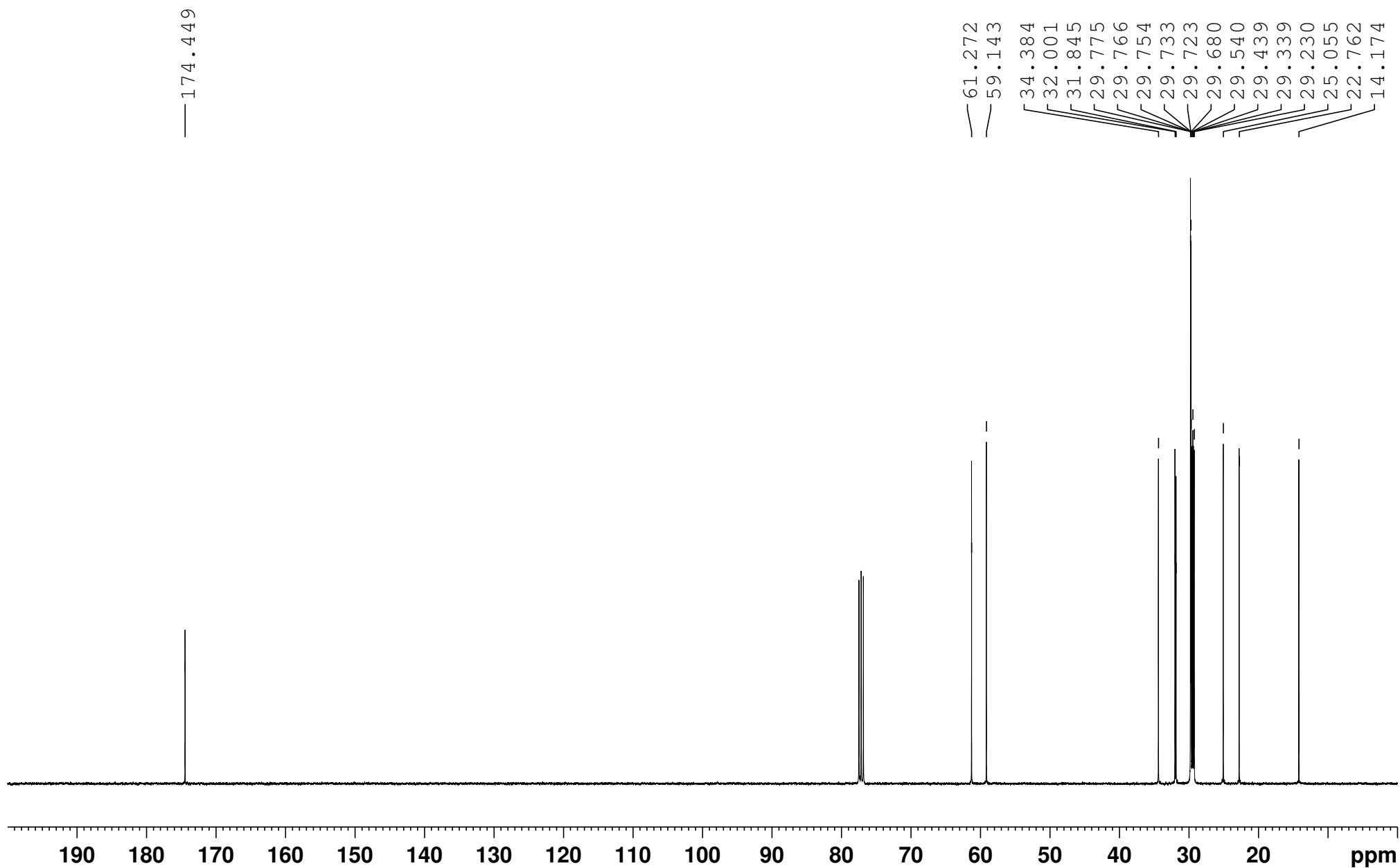
3-hydroxypropyl 4-methyl-1,2,3-thiadiazole-5-carboxylate - ^{13}C - CDCl_3 - 75 MHz



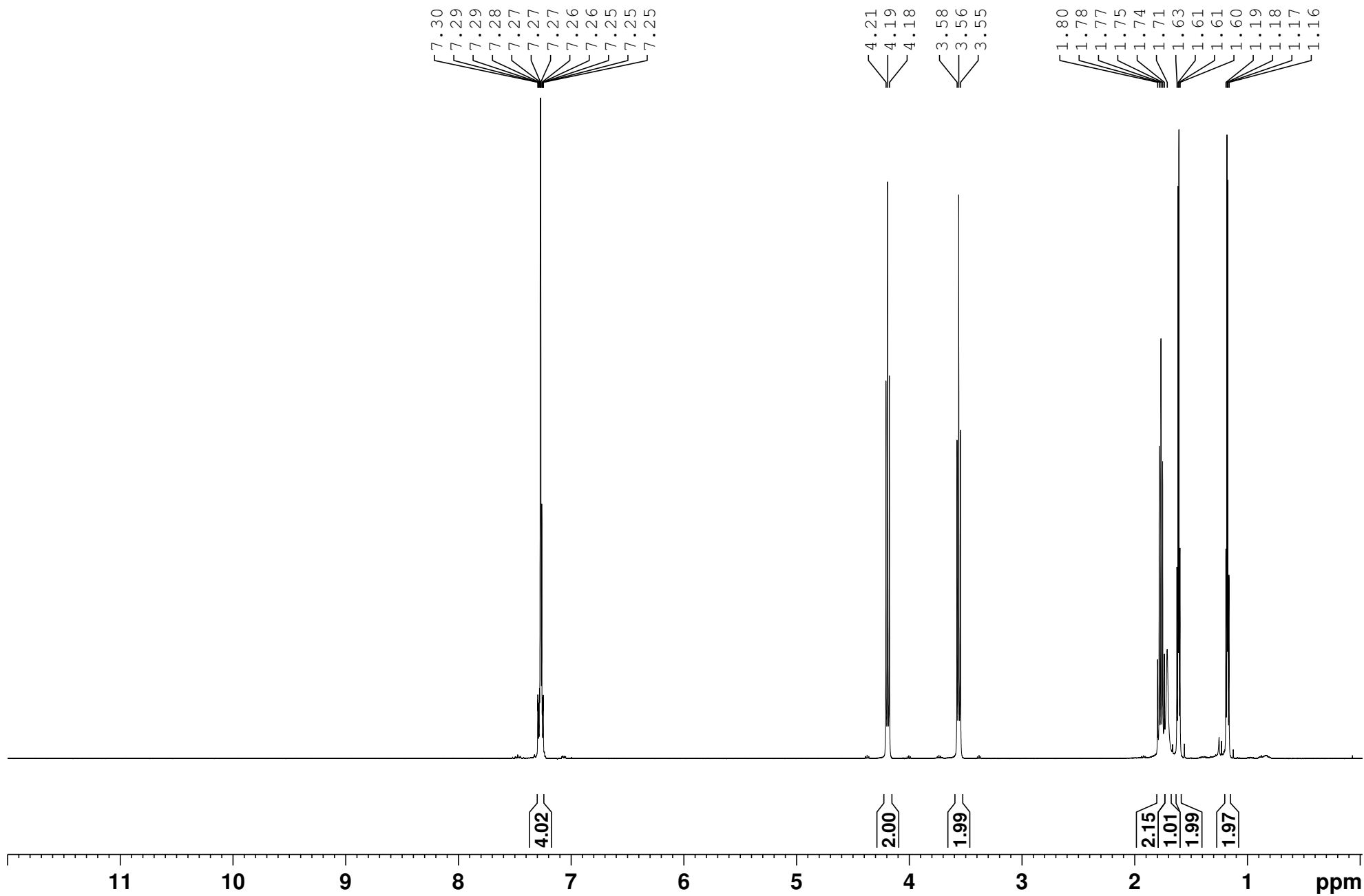
3-hydroxypropyl hexadecanoate - 1H - CDCl3 - 400 MHz



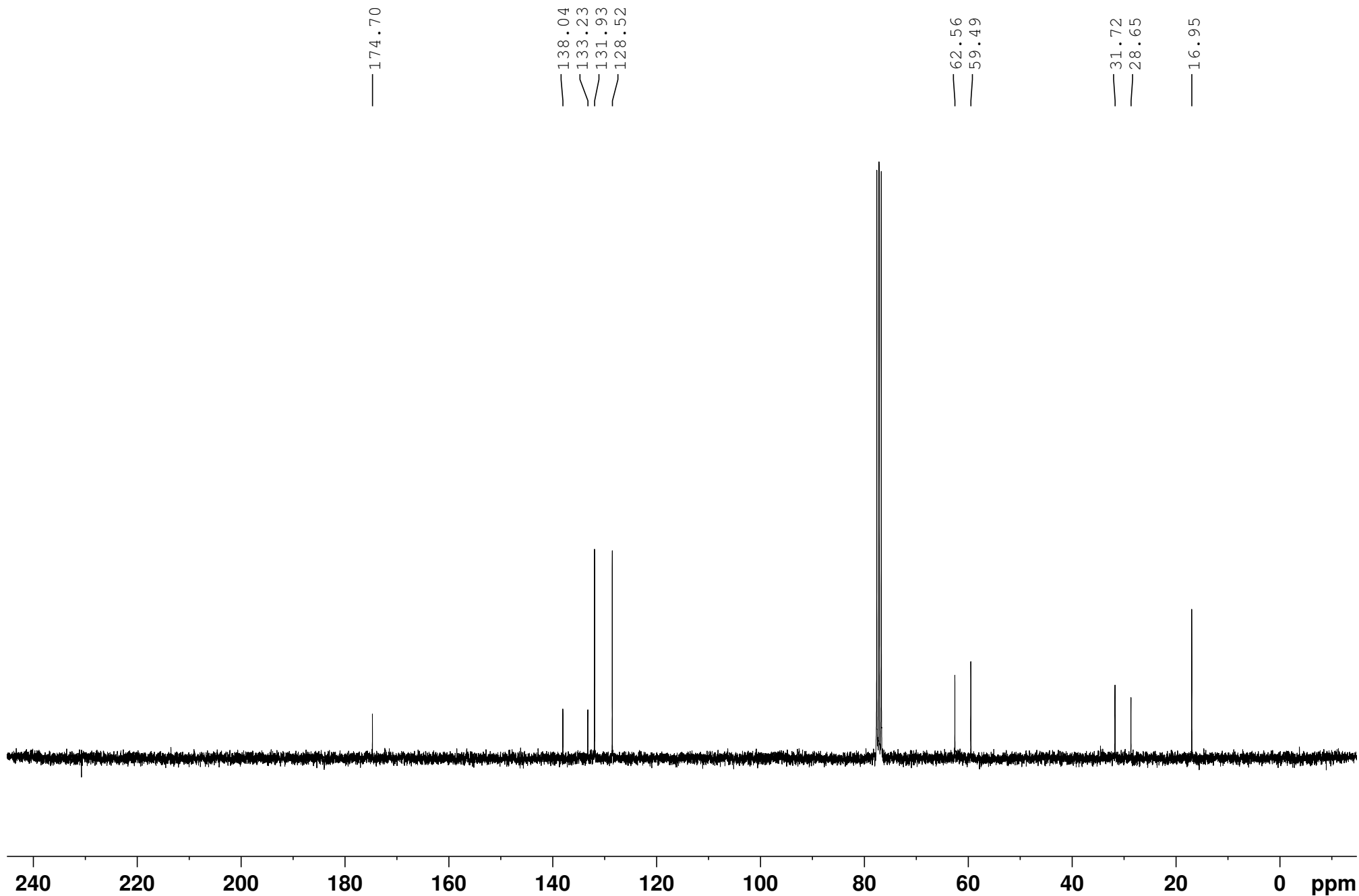
3-hydroxypropyl hexadecanoate - ^{13}C - CDCl_3 - 100 MHz



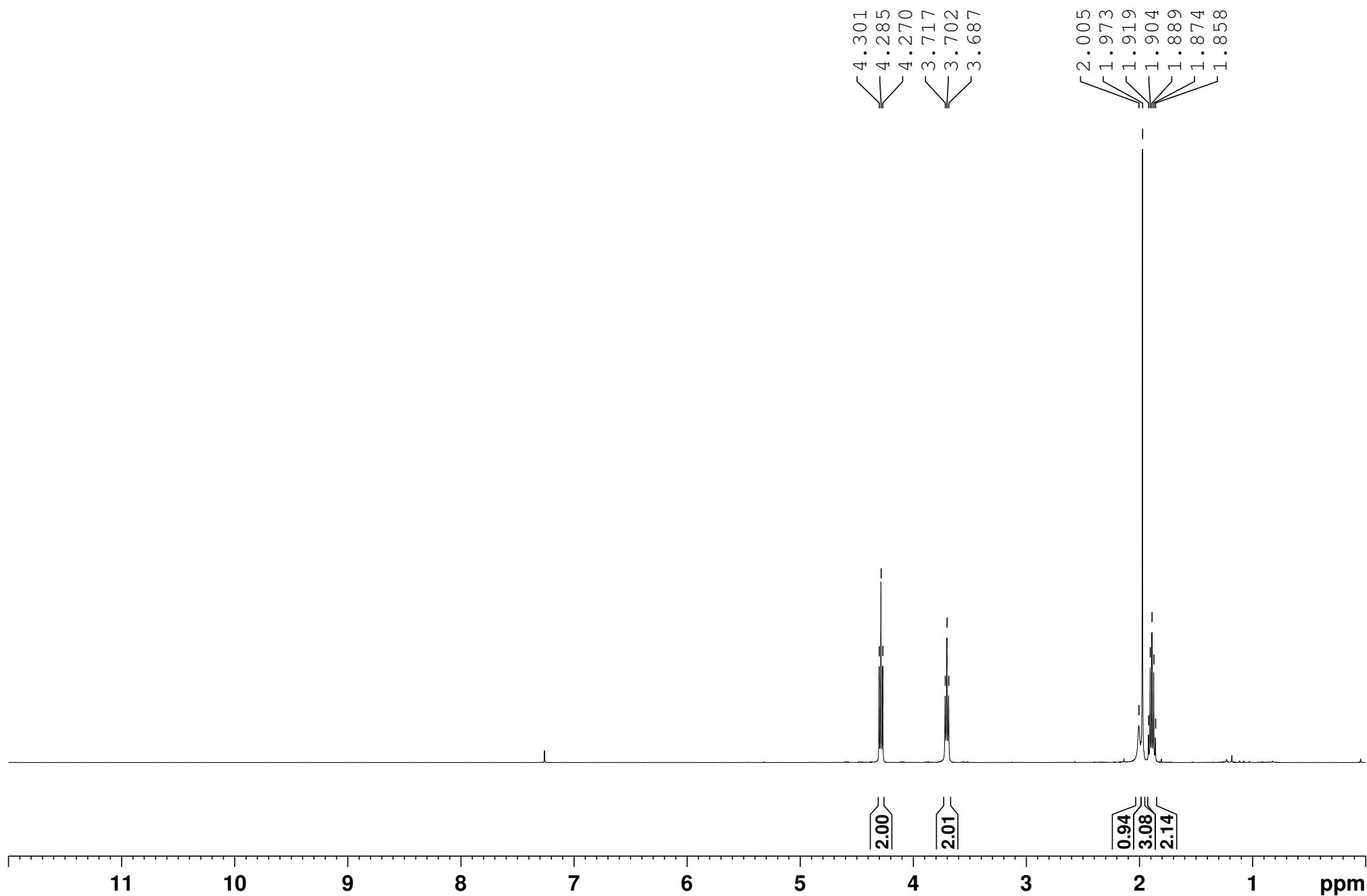
3-hydroxypropyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate - 1H - CDCl3 - 40



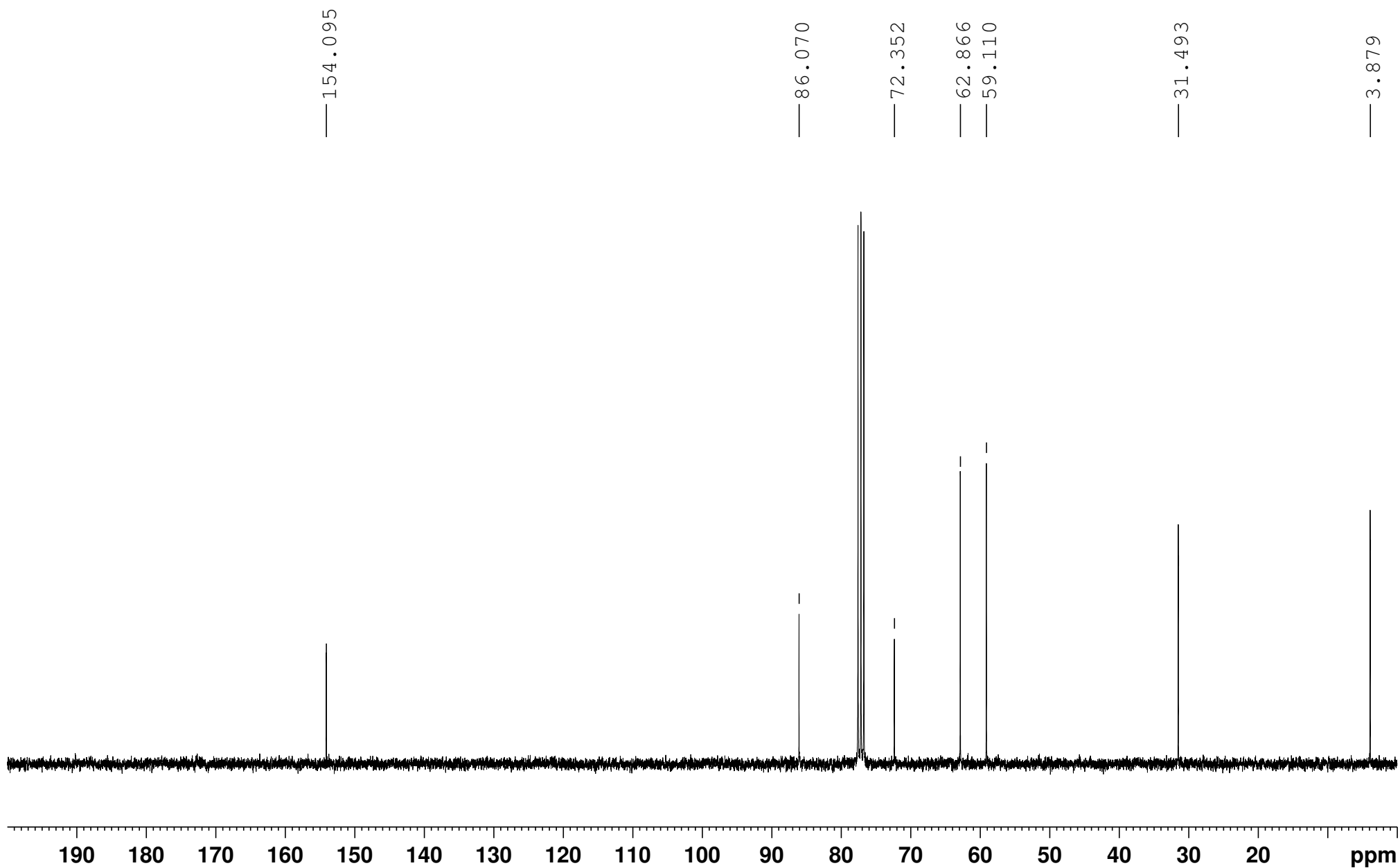
3-hydroxypropyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate - ^{13}C - CDCl_3 - 7



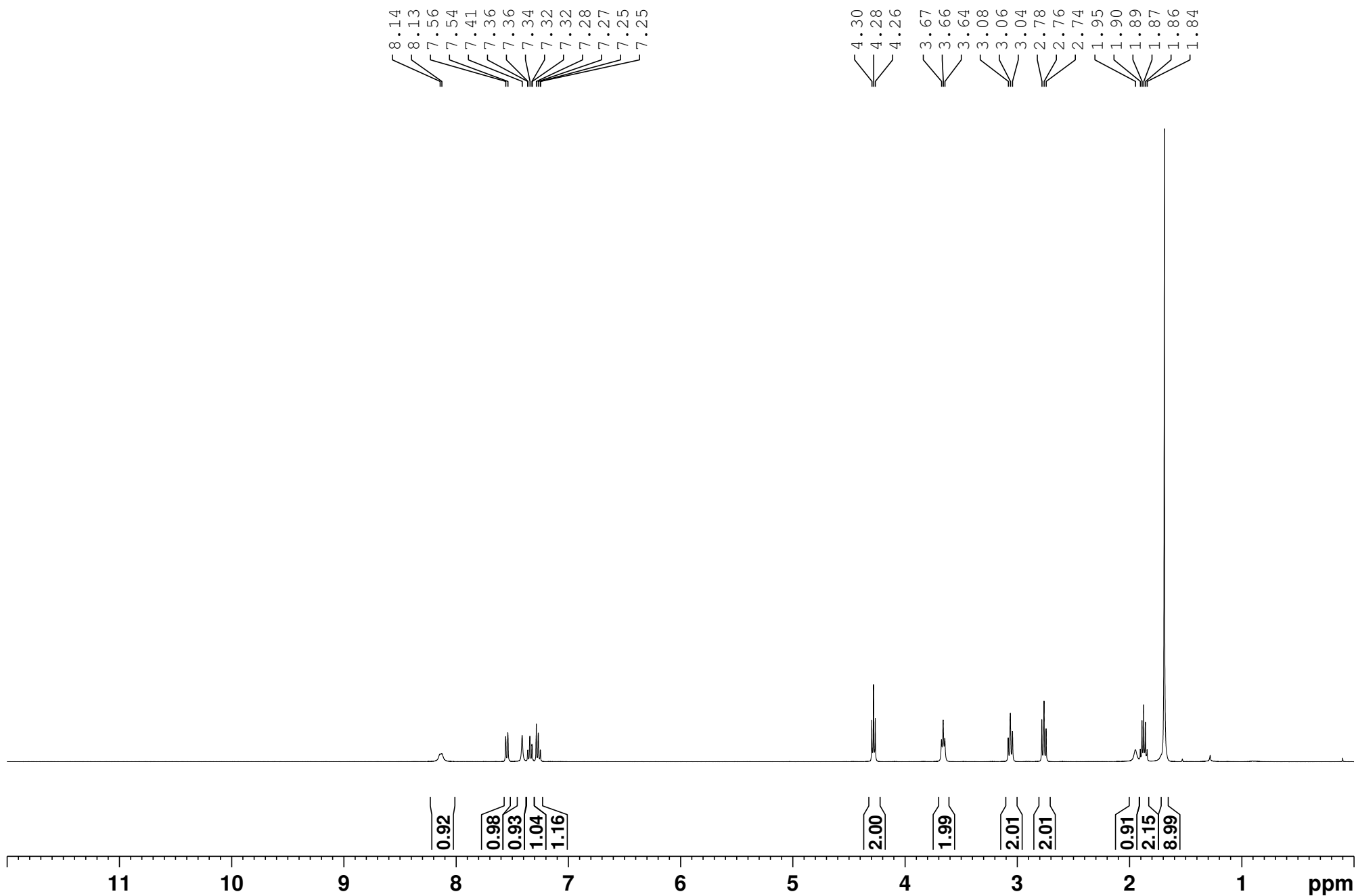
3-hydroxypropyl but-2-ynoate - 1H - CDCl3 - 400 MHz



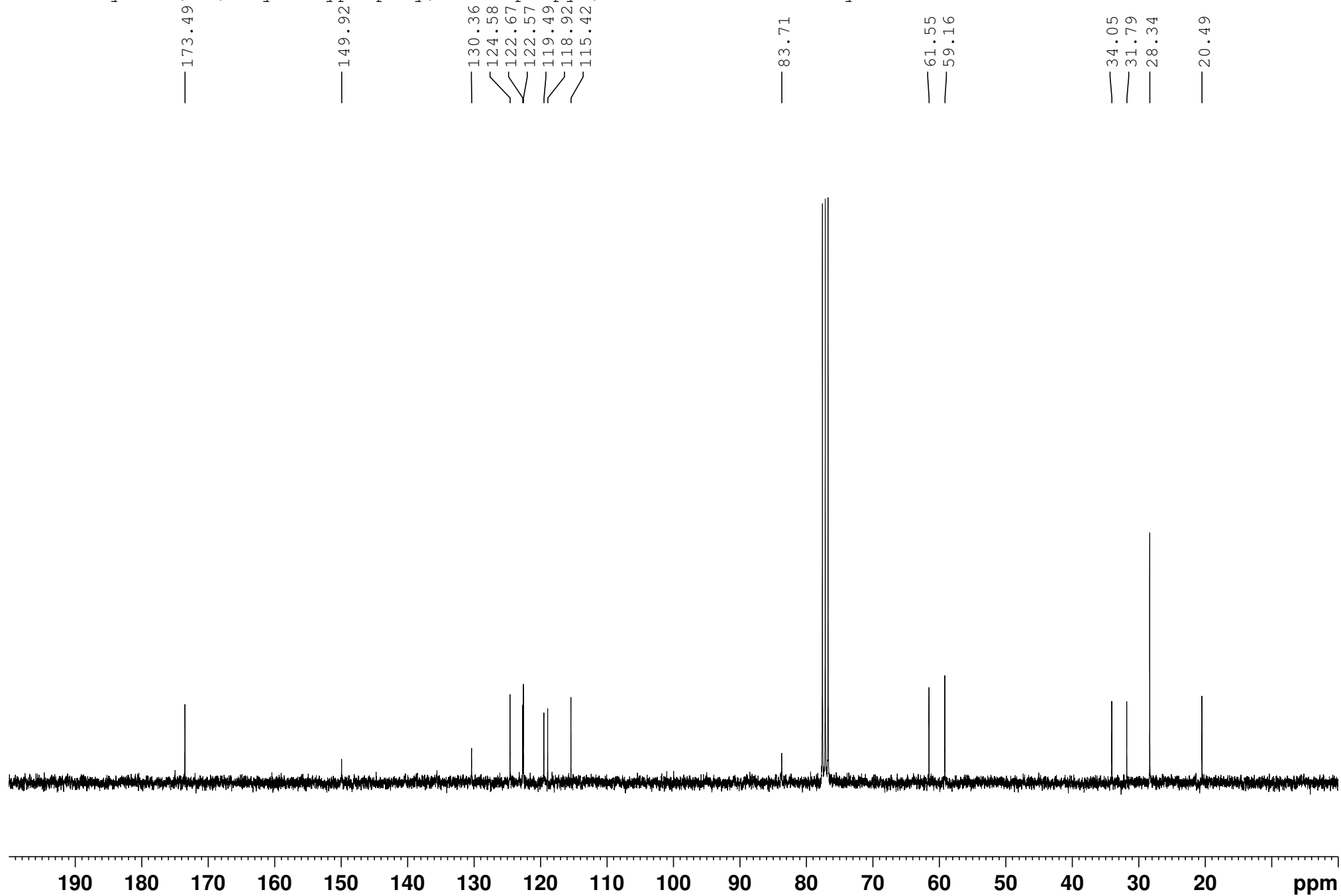
3-hydroxypropyl but-2-ynoate - ¹³C - CDCl₃ - 75 MHz



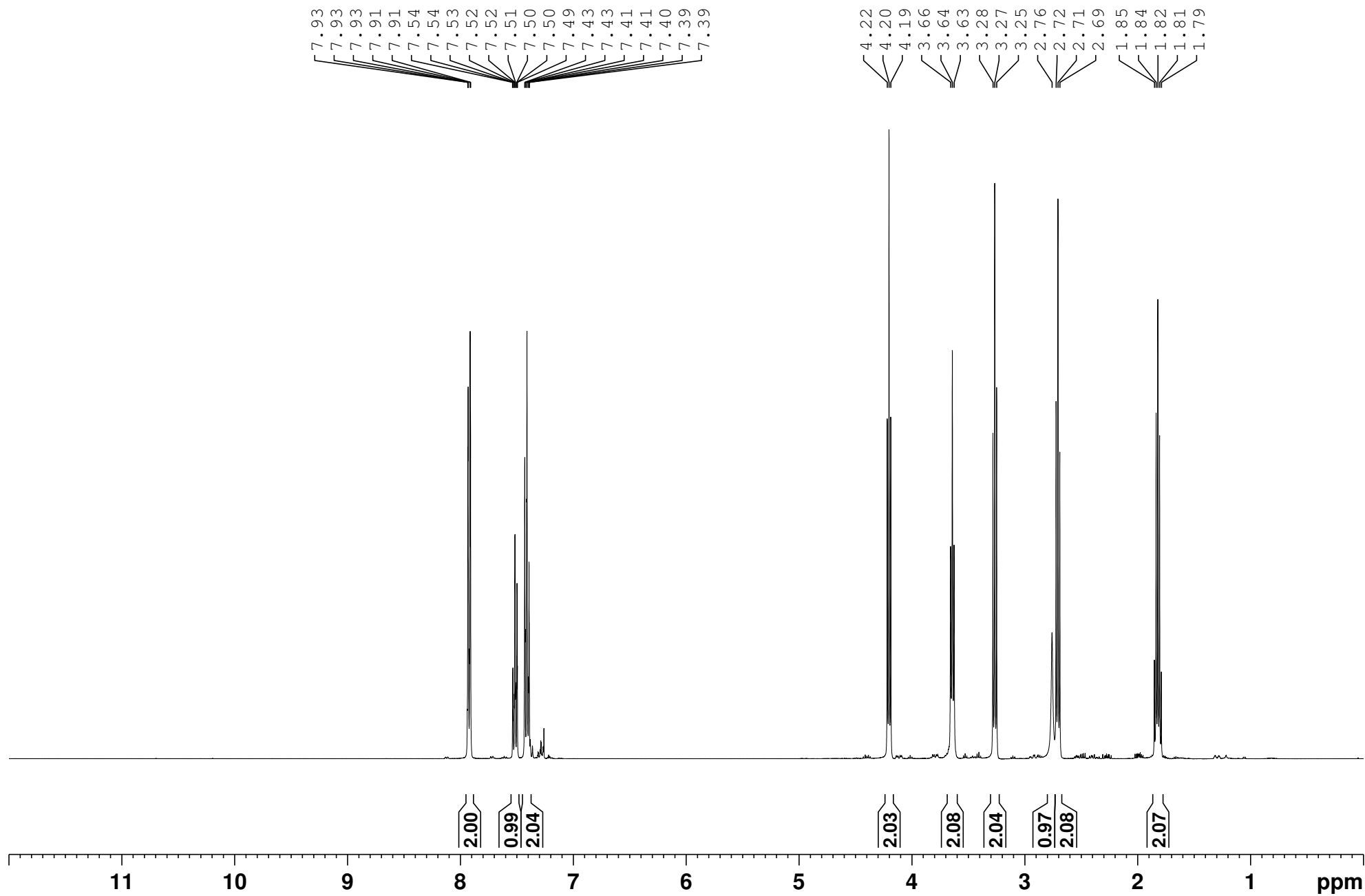
tert-butyl 3-(3-(3-hydroxypropoxy)-3-oxopropyl)-1H-indole-1-carboxylate - 1H -



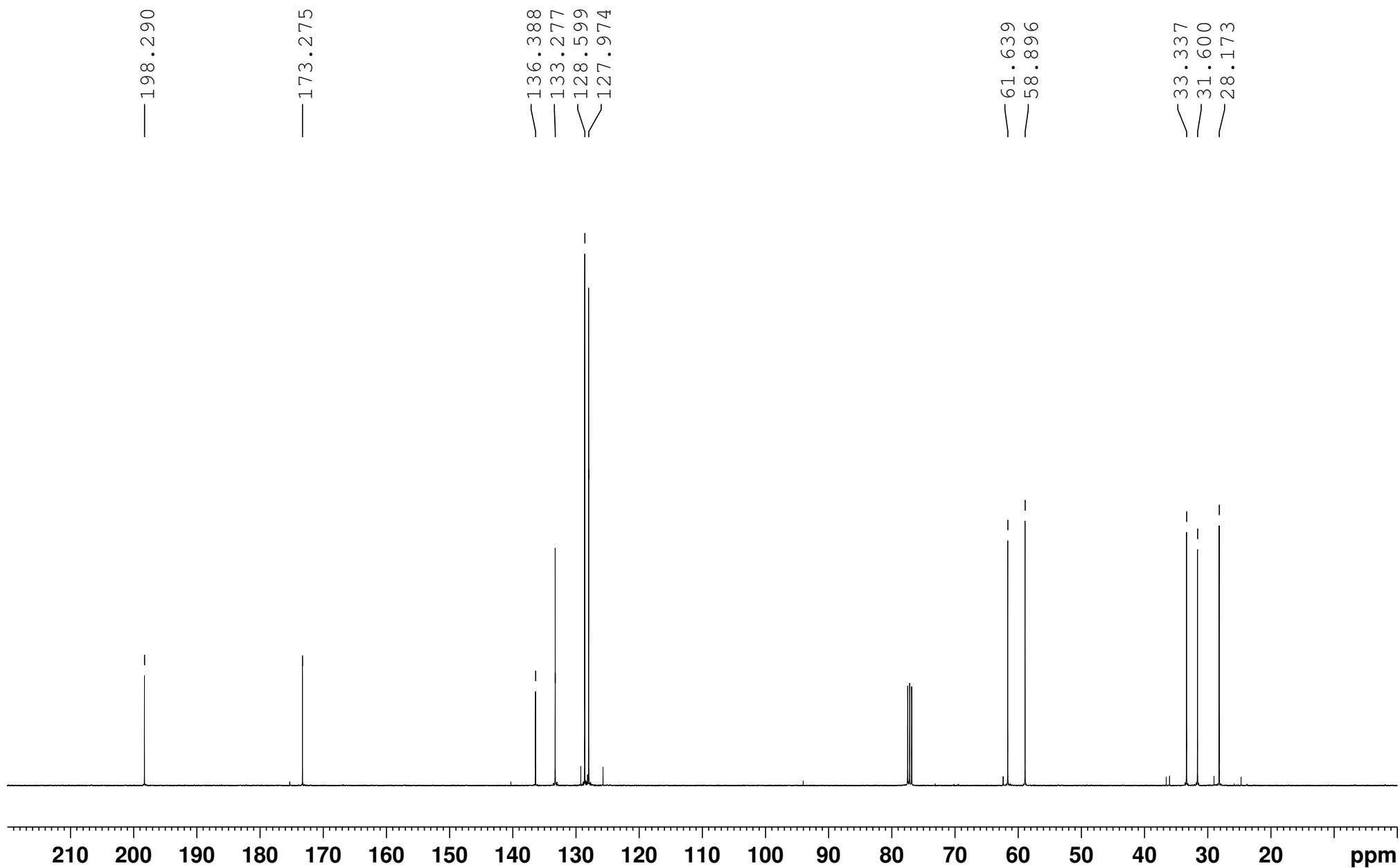
tert-butyl 3-(3-(3-hydroxypropoxy)-3-oxopropyl)-1H-indole-1-carboxylate - ¹³C



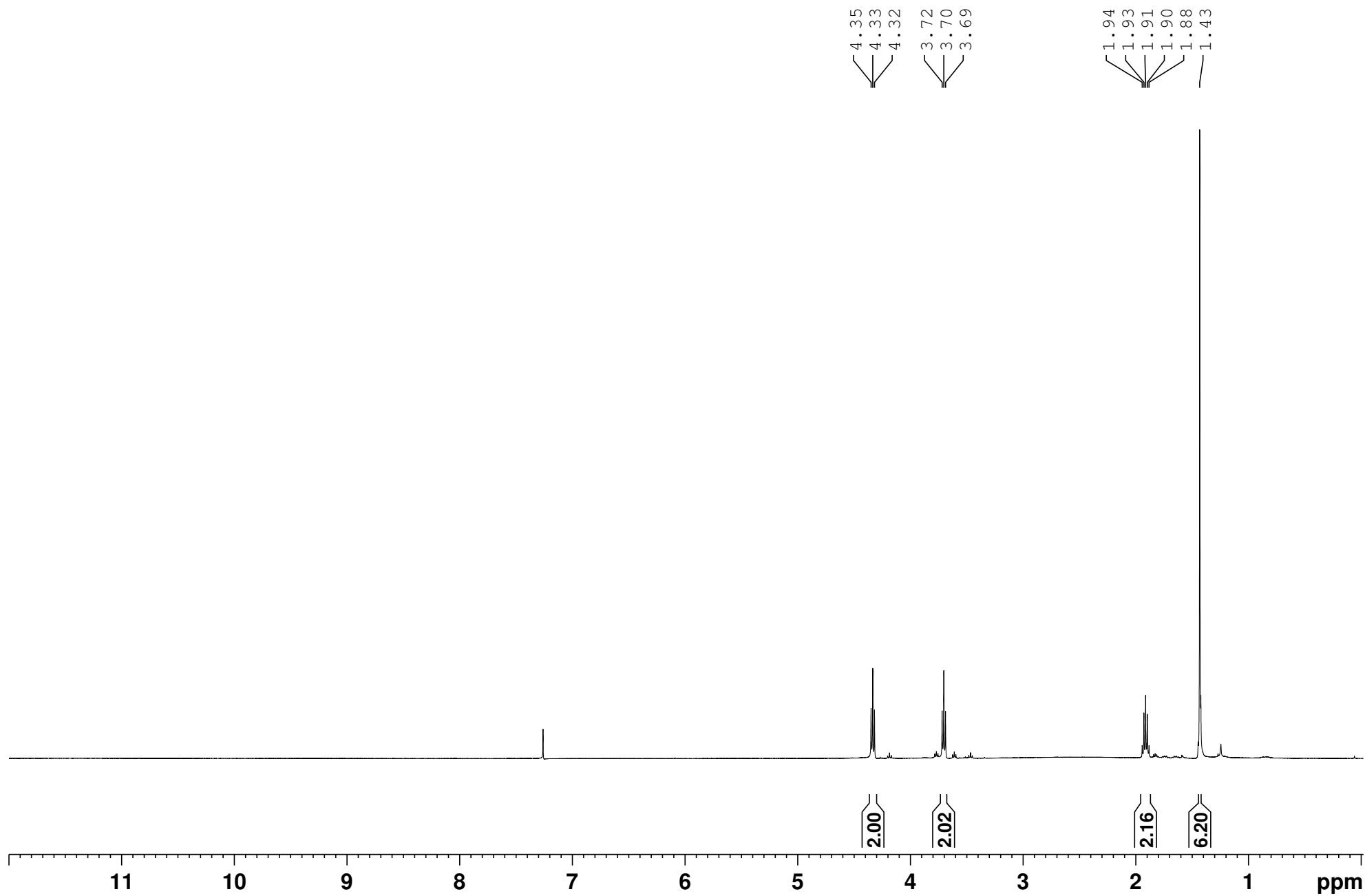
3-hydroxypropyl 4-oxo-4-phenylbutanoate - 1H - CDCl3 - 400 MHz



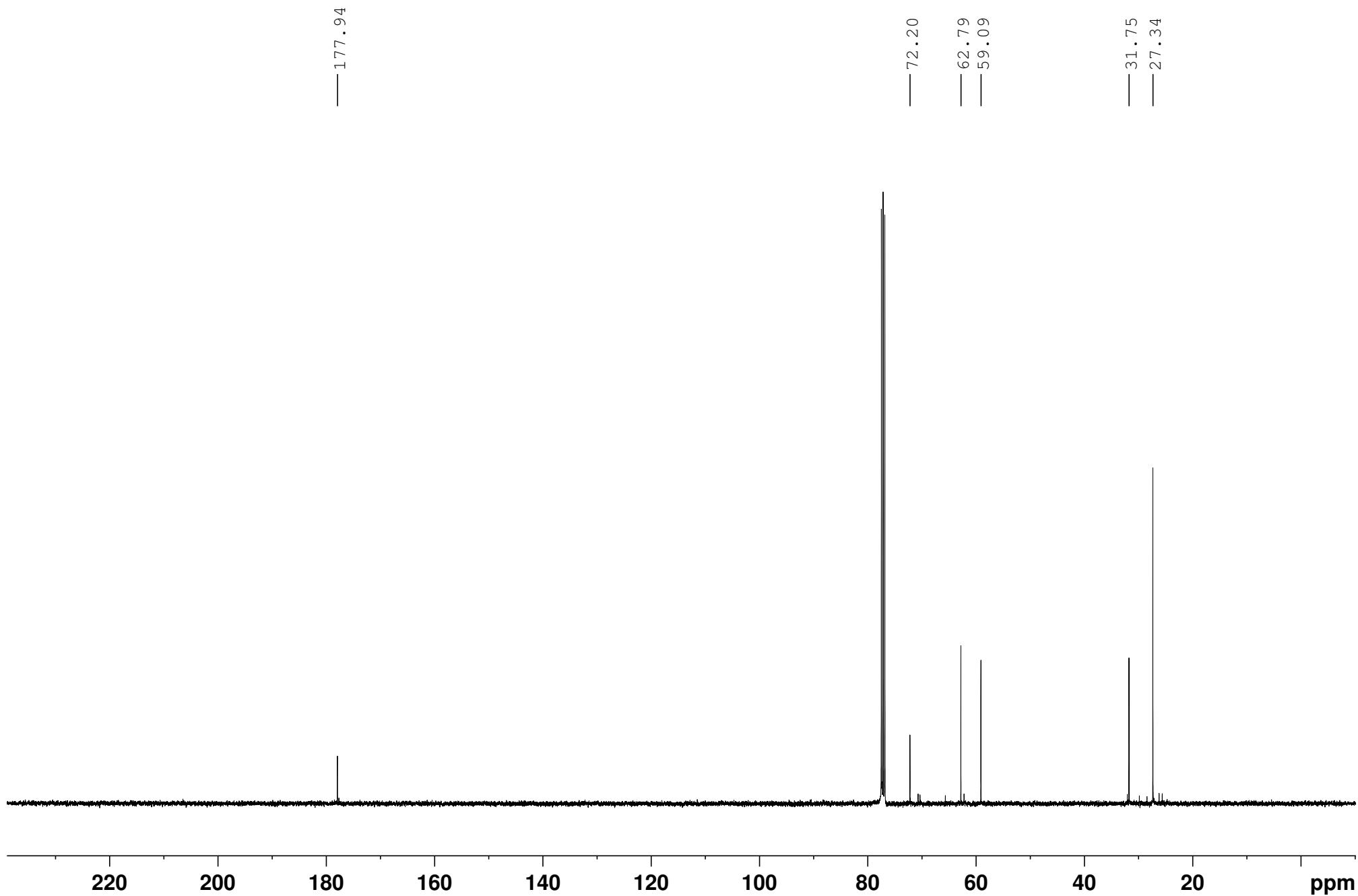
3-hydroxypropyl 4-oxo-4-phenylbutanoate - ^{13}C - CDCl_3 - 100 MHz



3-hydroxypropyl 2-hydroxy-2-methylpropanoate - 1H - CDCl3 - 400 MHz



3-hydroxypropyl 2-hydroxy-2-methylpropanoate - ¹³C - CDCl₃ - 100 MHz



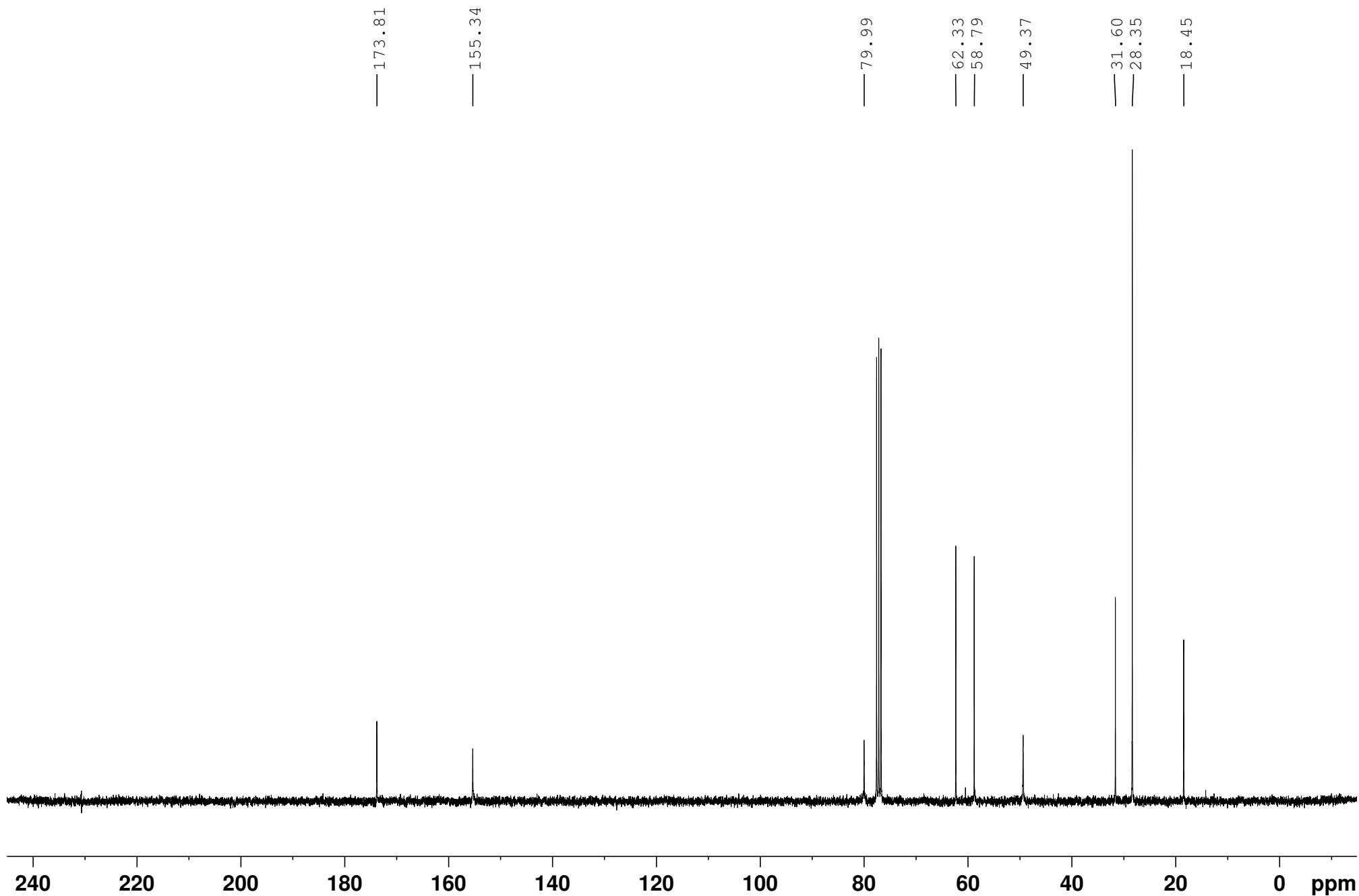
3-hydroxypropyl (tert-butoxycarbonyl)alaninate - 1H - CDCl3 - 300 MHz

5.16
5.15
4.26
4.24
4.22
3.66
3.64
3.62
2.67
1.88
1.86
1.84
1.82
1.80
1.39
1.34
1.32



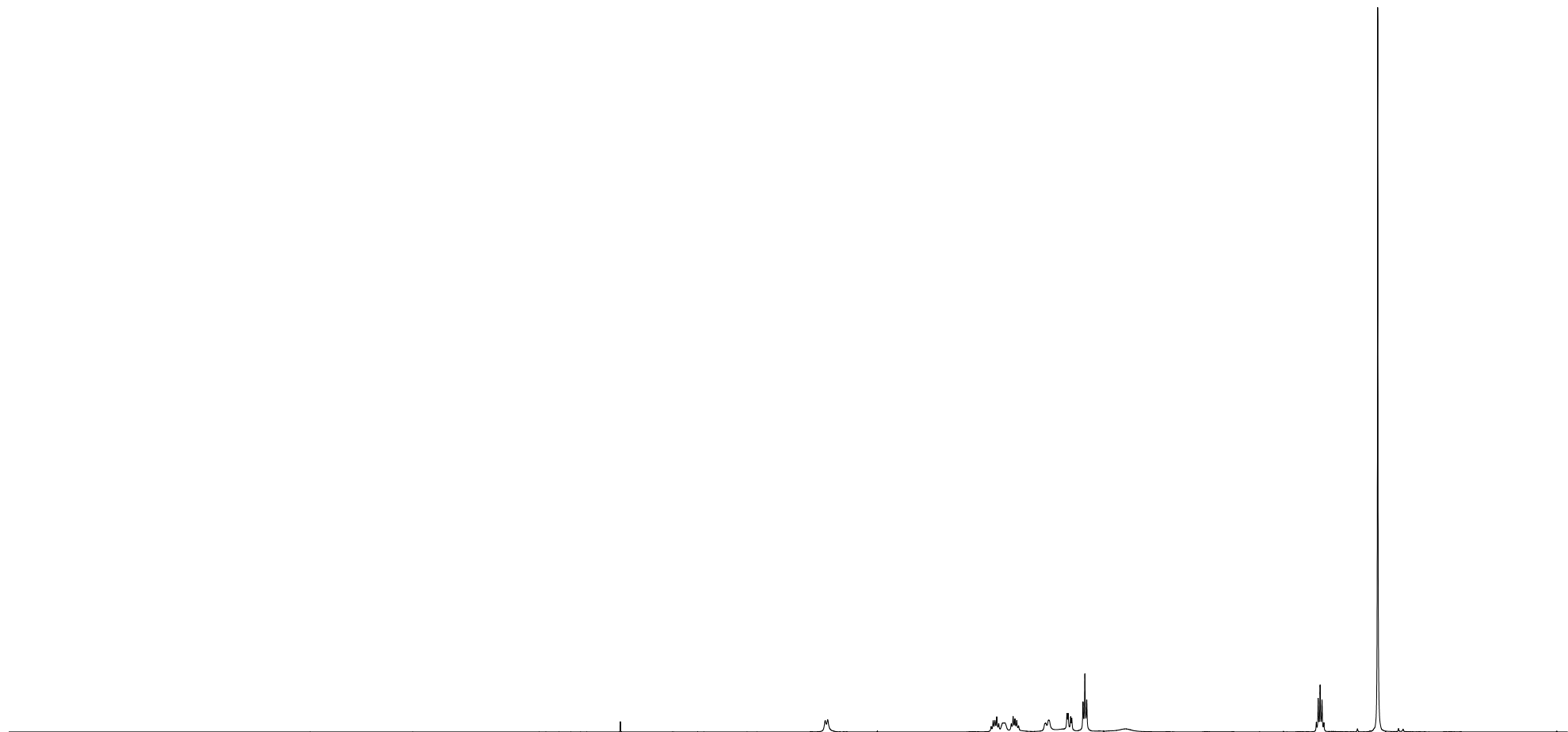
11 10 9 8 7 6 5 4 3 2 1 ppm

3-hydroxypropyl (tert-butoxycarbonyl)alaninate - ^{13}C - CDCl_3 - 75 MHz



3-hydroxypropyl (tert-butoxycarbonyl)serinate - 1H - CDCl3 - 400 MHz

5.71
5.69
4.43
4.41
4.40
4.38
4.37
4.34
4.33
4.32
4.27
4.26
4.24
4.23
4.21
4.00
3.98
3.98
3.84
3.83
3.81
3.80
3.72
3.70
3.69
3.39
1.92
1.90
1.89
1.87
1.86



1.00

1.08

0.88

1.20

1.23

1.29

2.20

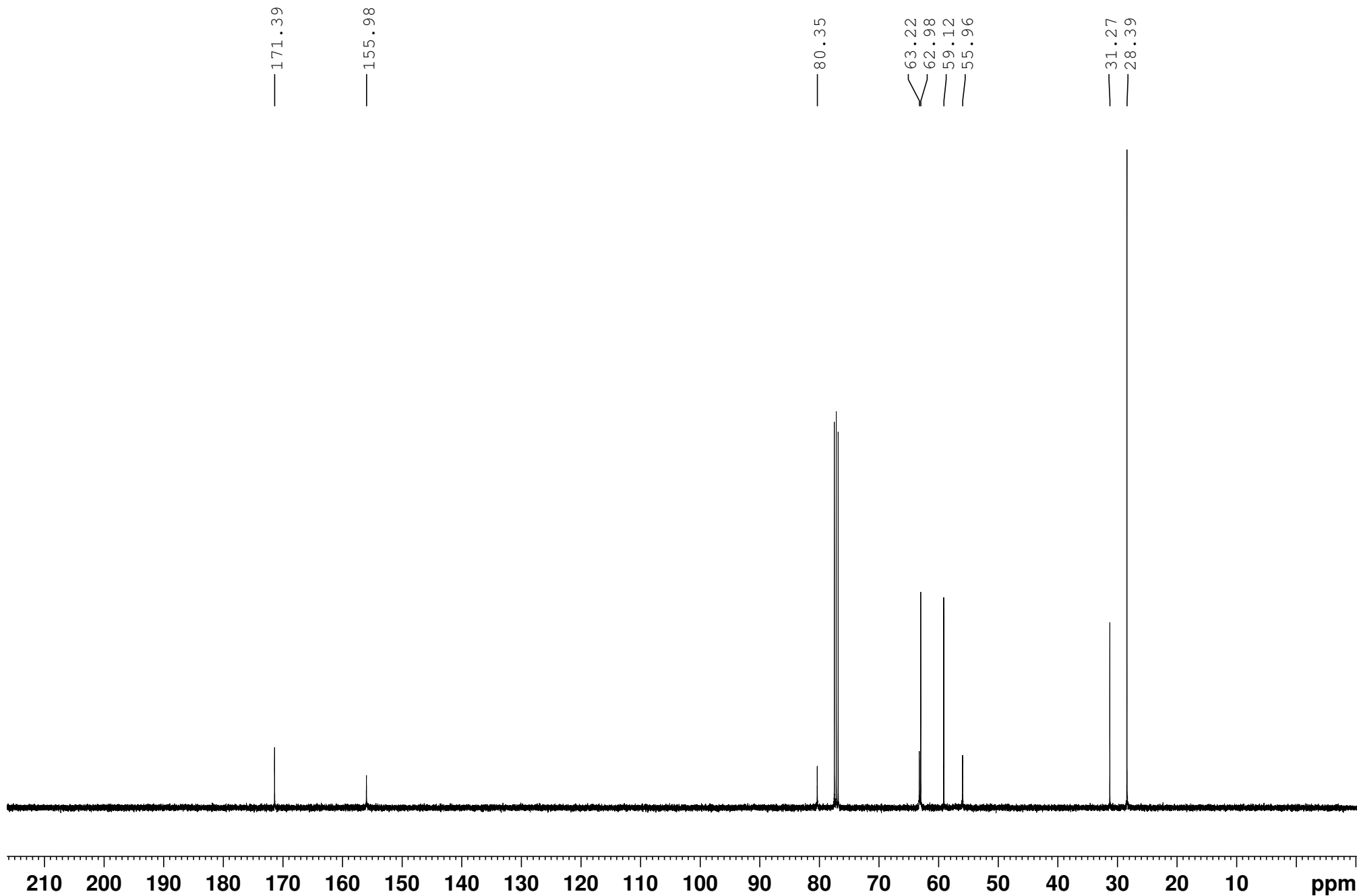
0.96

2.09

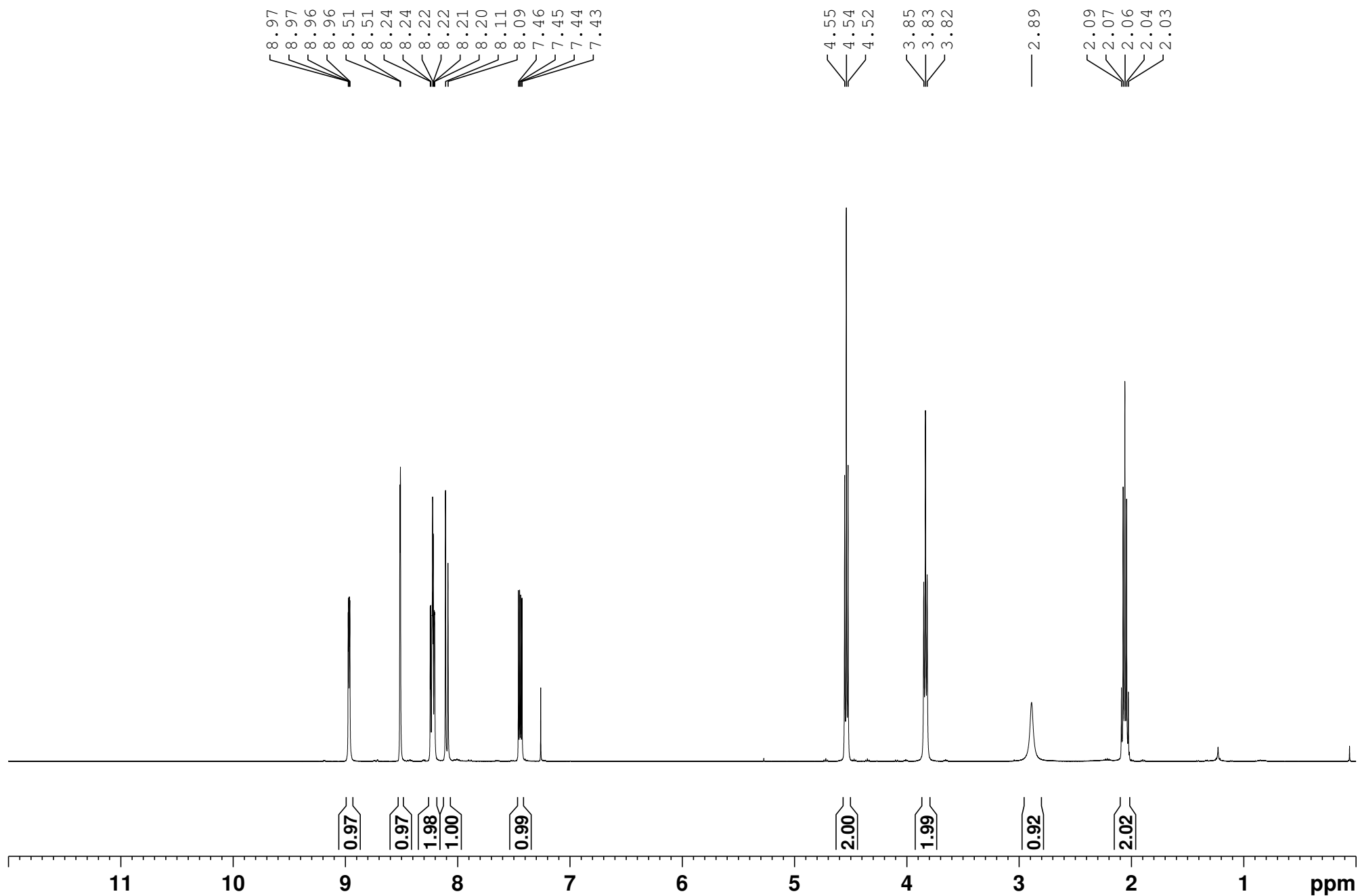
9.16

11 10 9 8 7 6 5 4 3 2 1 ppm

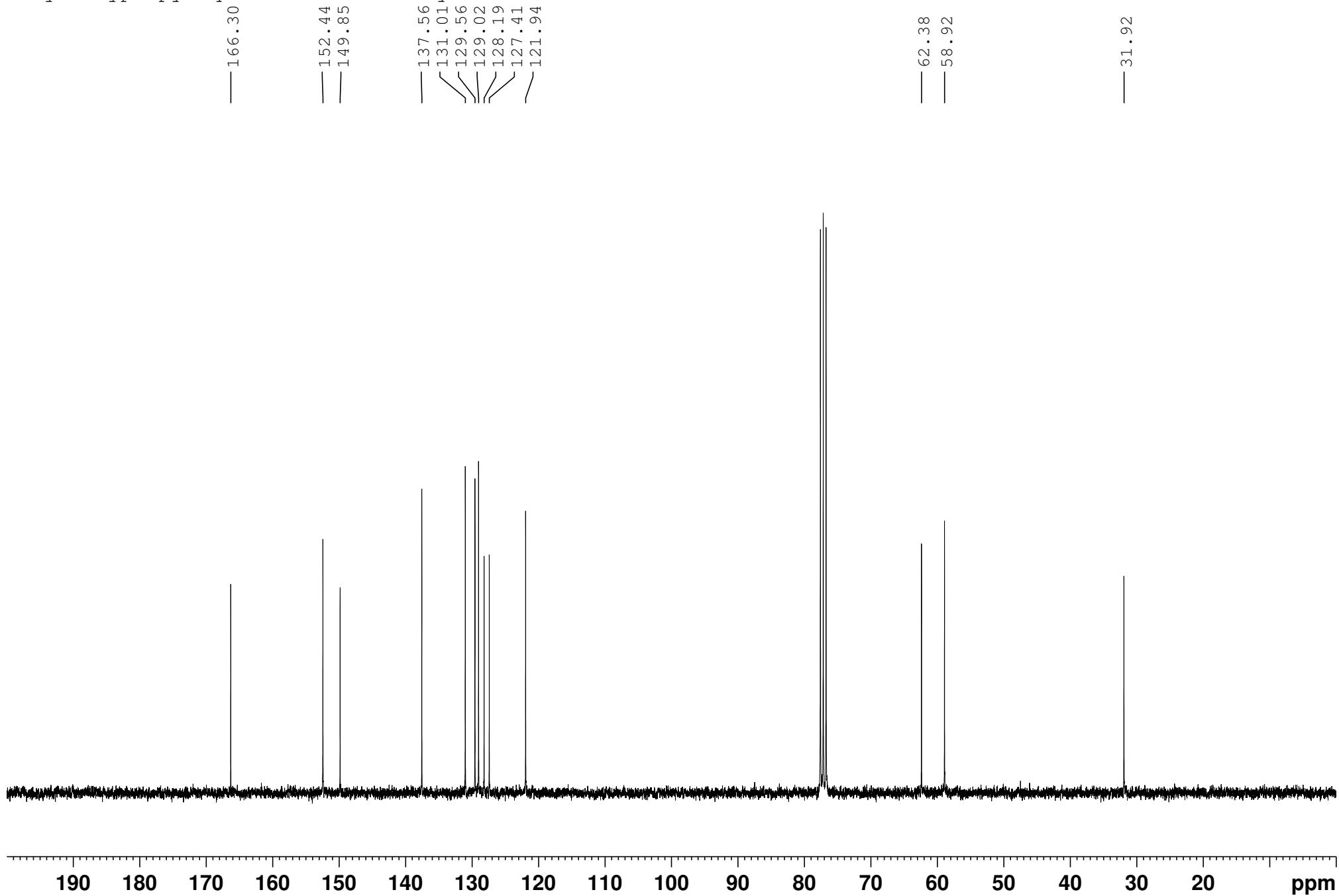
3-hydroxypropyl (tert-butoxycarbonyl)serinate - ^{13}C - CDCl_3 - 100 MHz



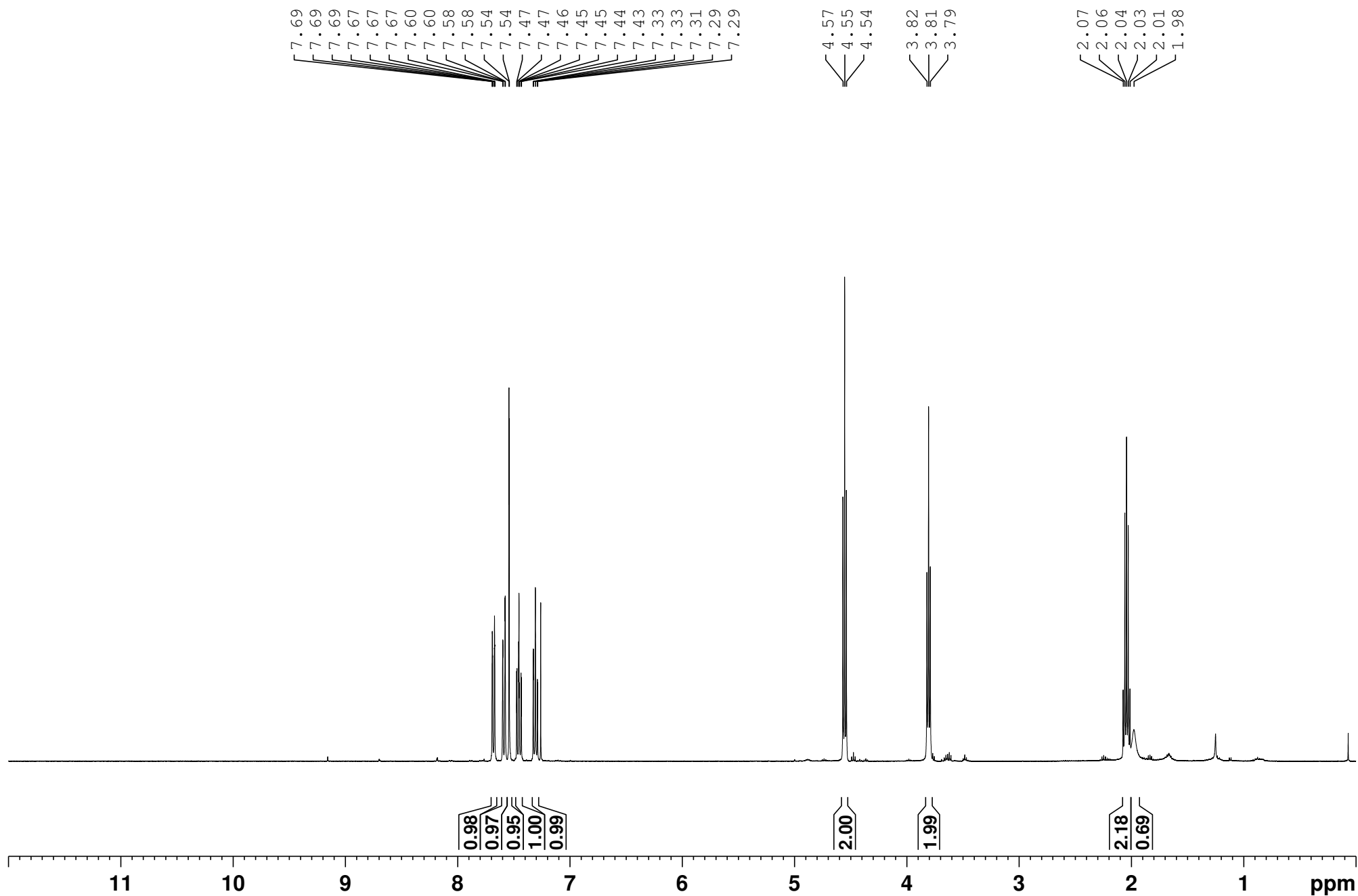
3-hydroxypropyl quinoline-6-carboxylate - 1H - CDCl3 - 400 MHz



3-hydroxypropyl quinoline-6-carboxylate - ^{13}C - CDCl_3 - 75 MHz

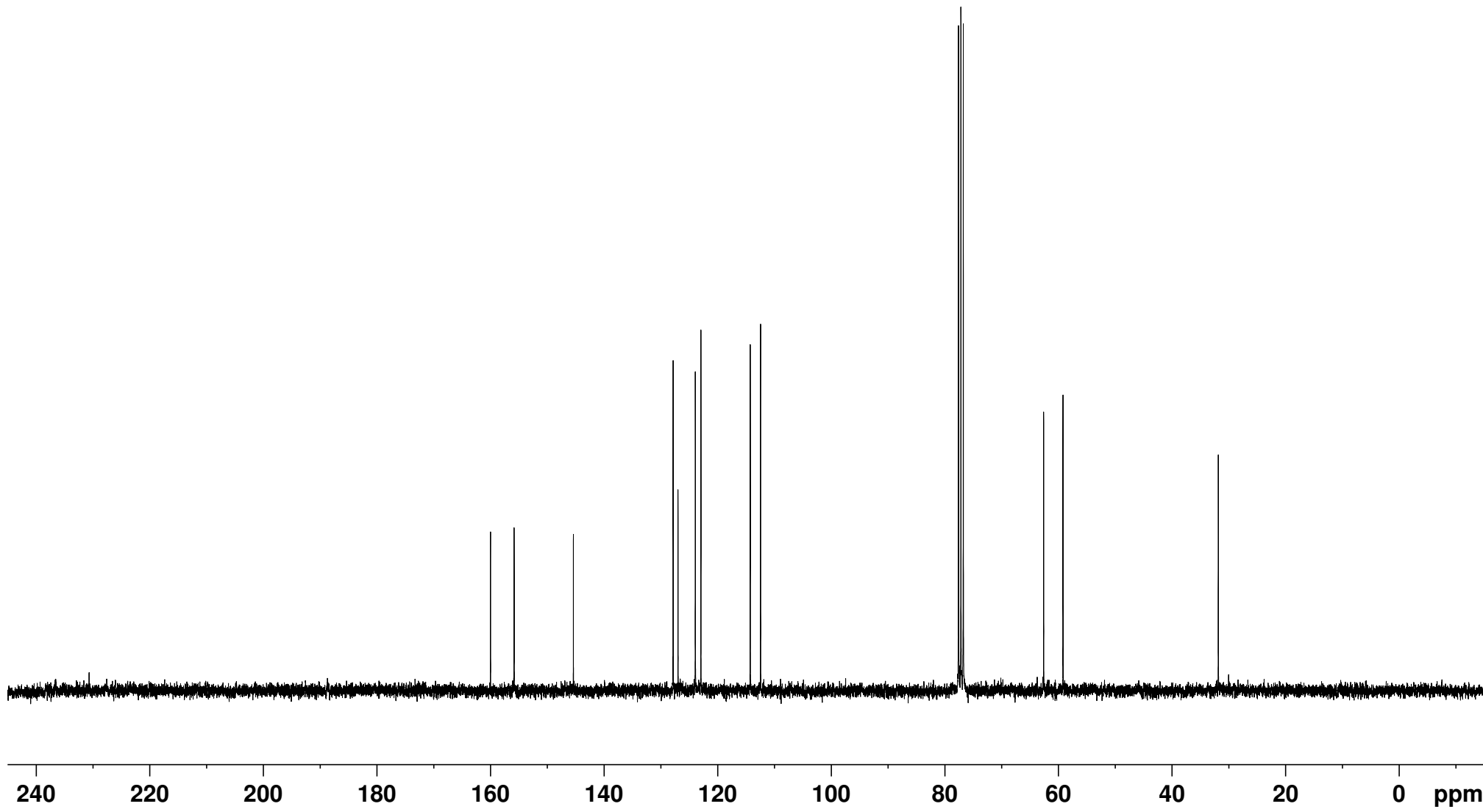


3-hydroxypropyl benzo-furan-2-carboxylate - 1H - CDCl3 - 400 MHz

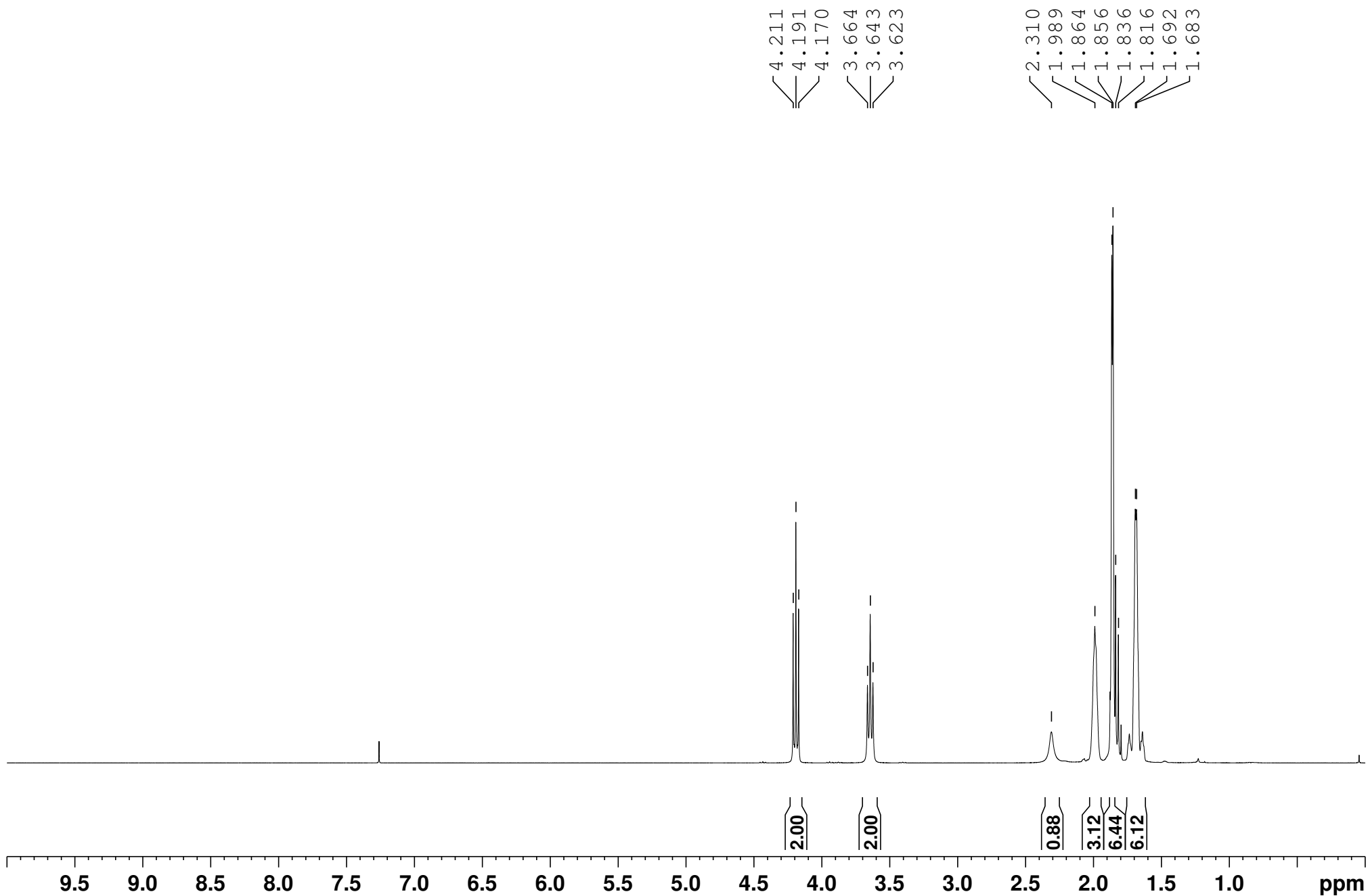


3-hydroxypropyl benzofuran-2-carboxylate - ^{13}C - CDCl_3 - 75 MHz

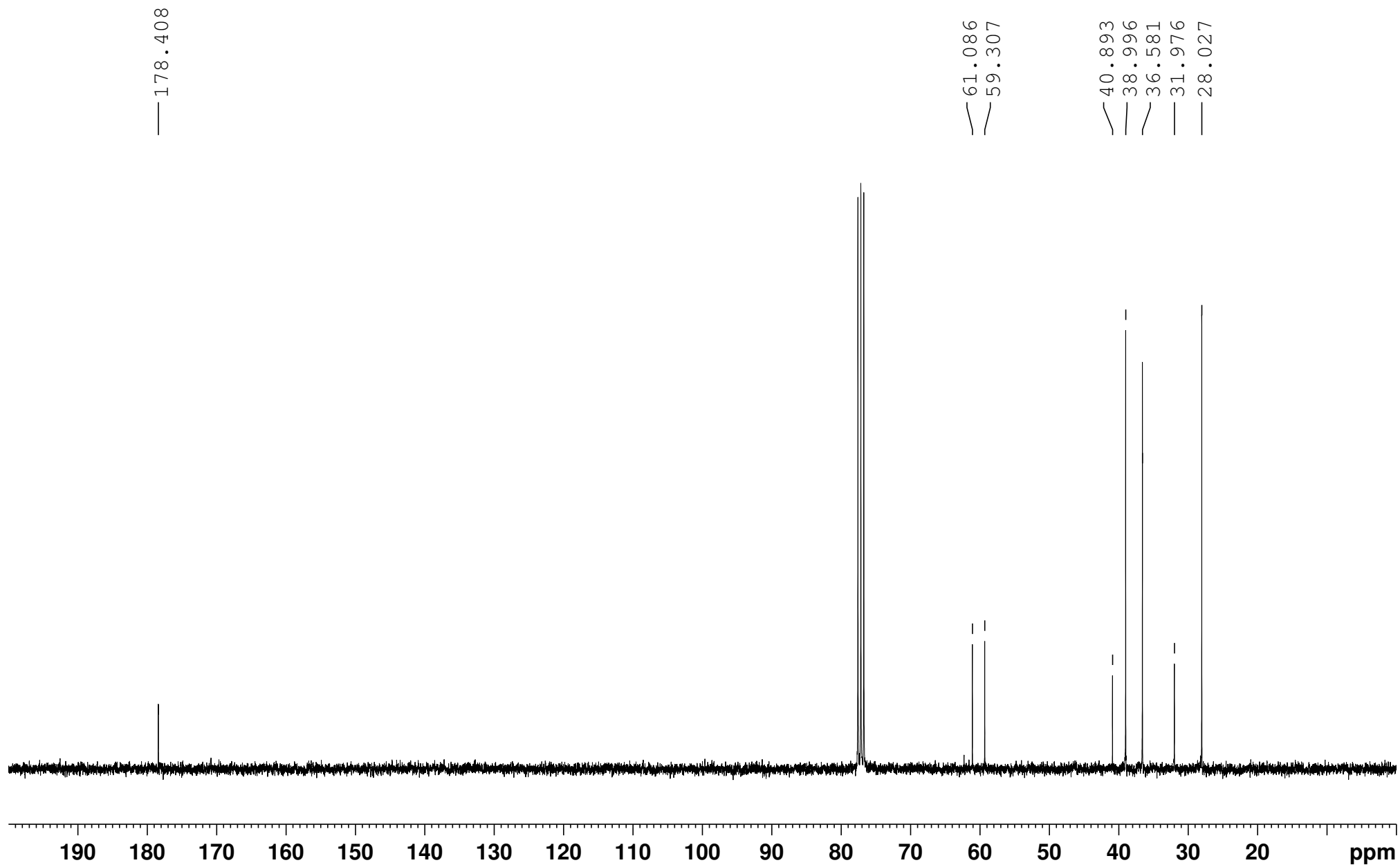
159.99
155.83
145.40
127.83
126.97
123.93
122.94
114.25
112.44
62.59
59.18
31.83



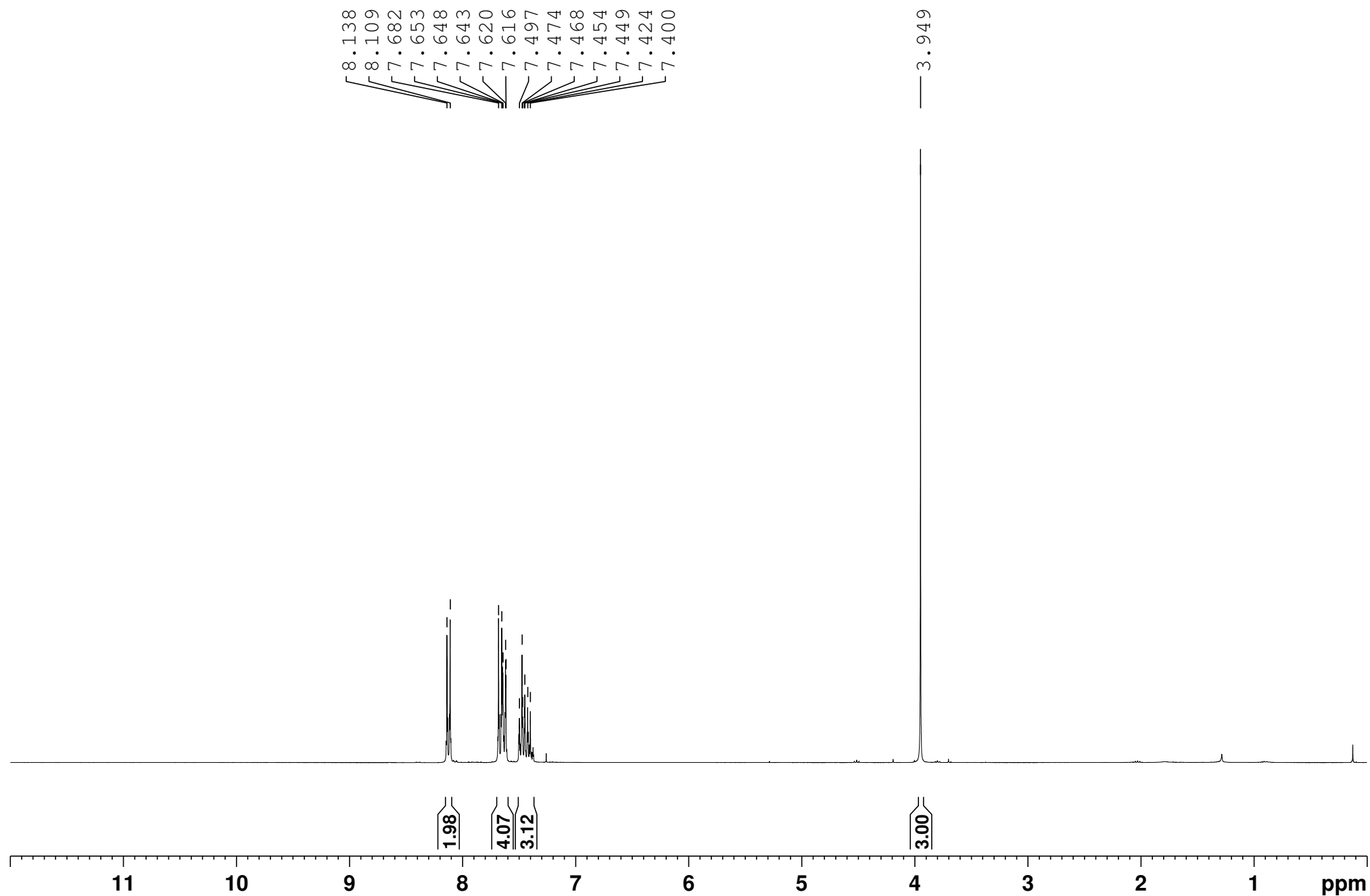
3-hydroxypropyl-adamantane-1-carboxylate - 1H - CDCl3 - 300 MHz



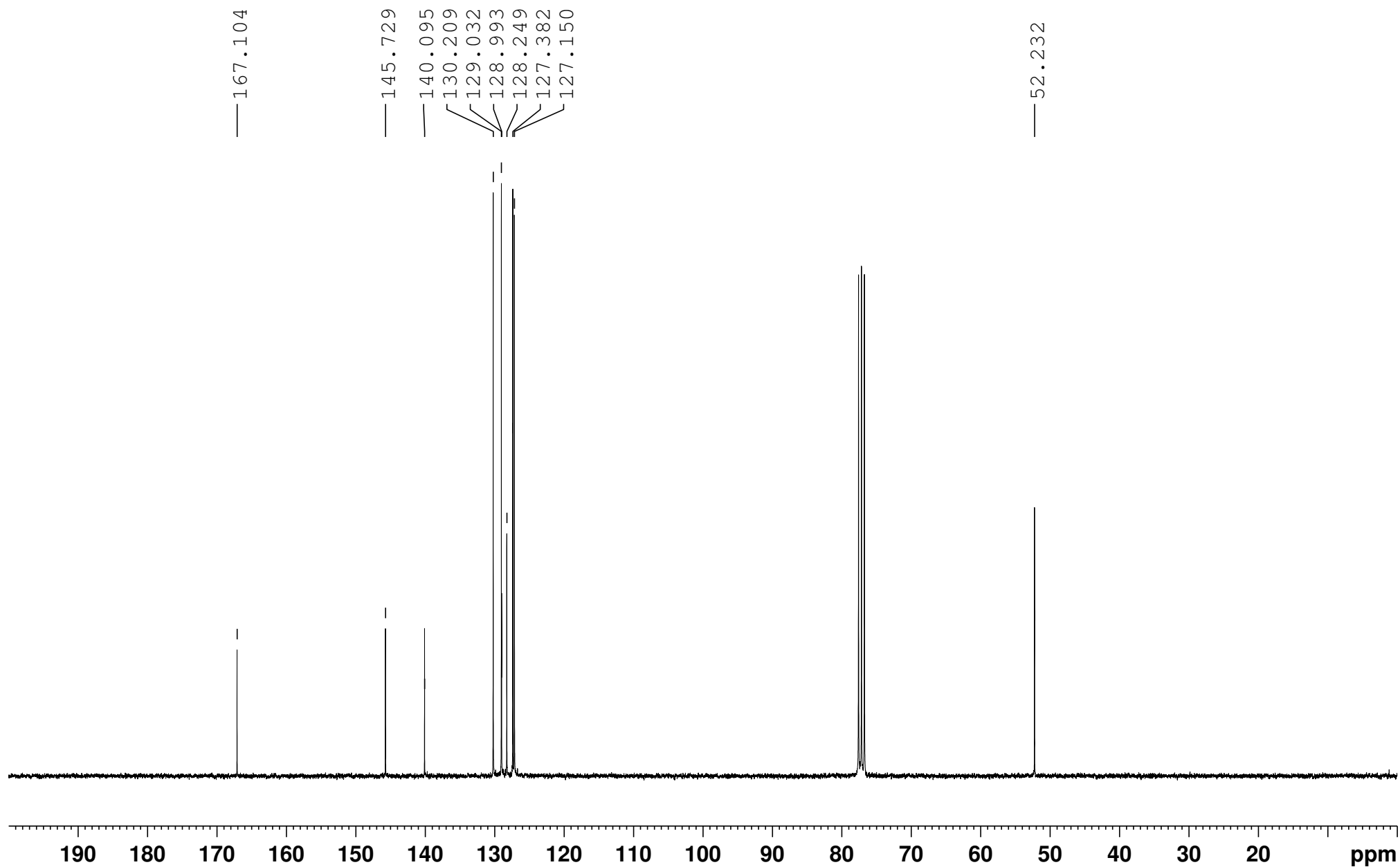
3-hydroxypropyl-adamantane-1-carboxylate - ^{13}C - CDCl_3 - 75 MHz



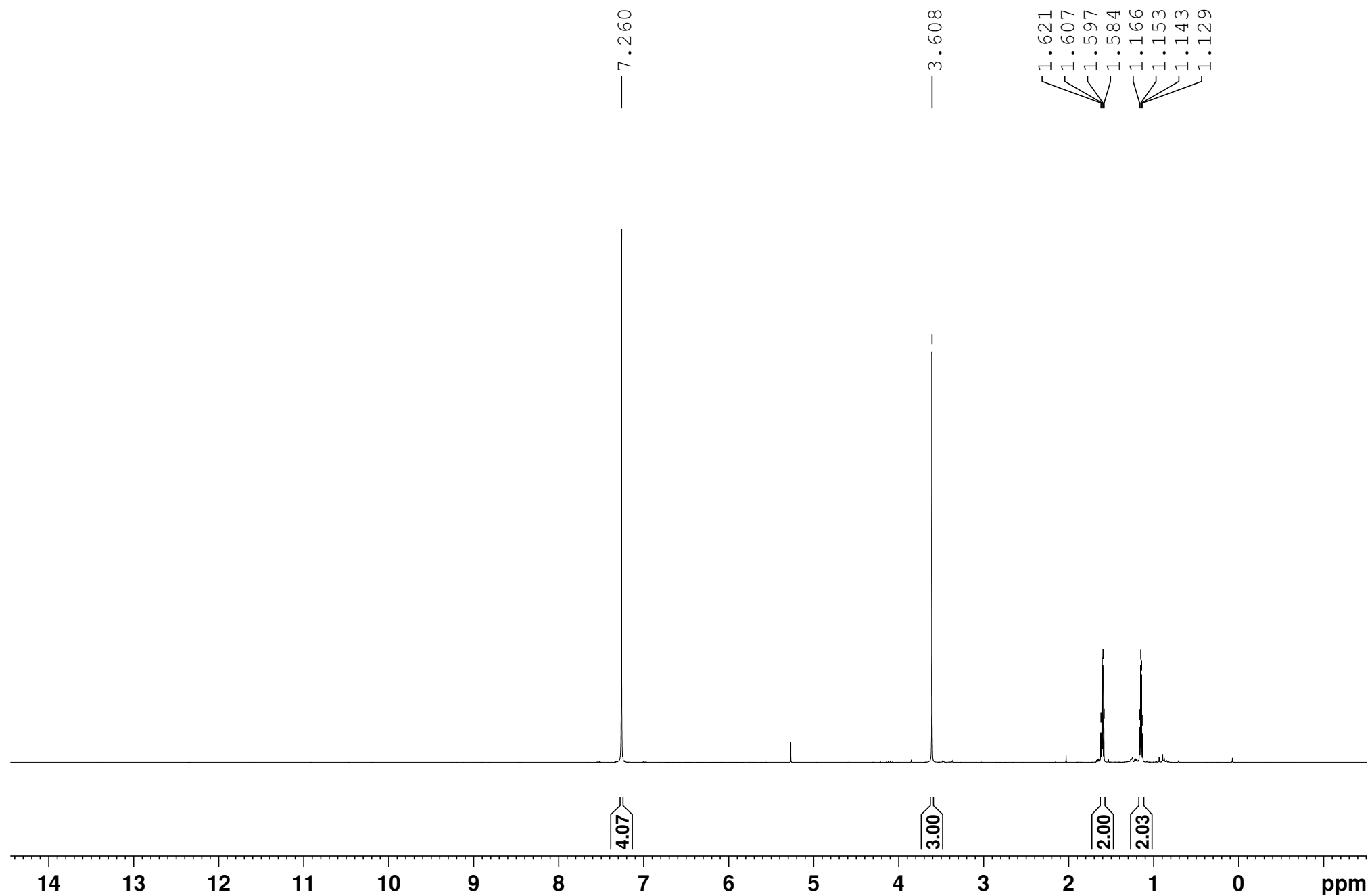
methyl [1,1'-biphenyl]-4-carboxylate - 1H - CDCl3 - 300 MHz



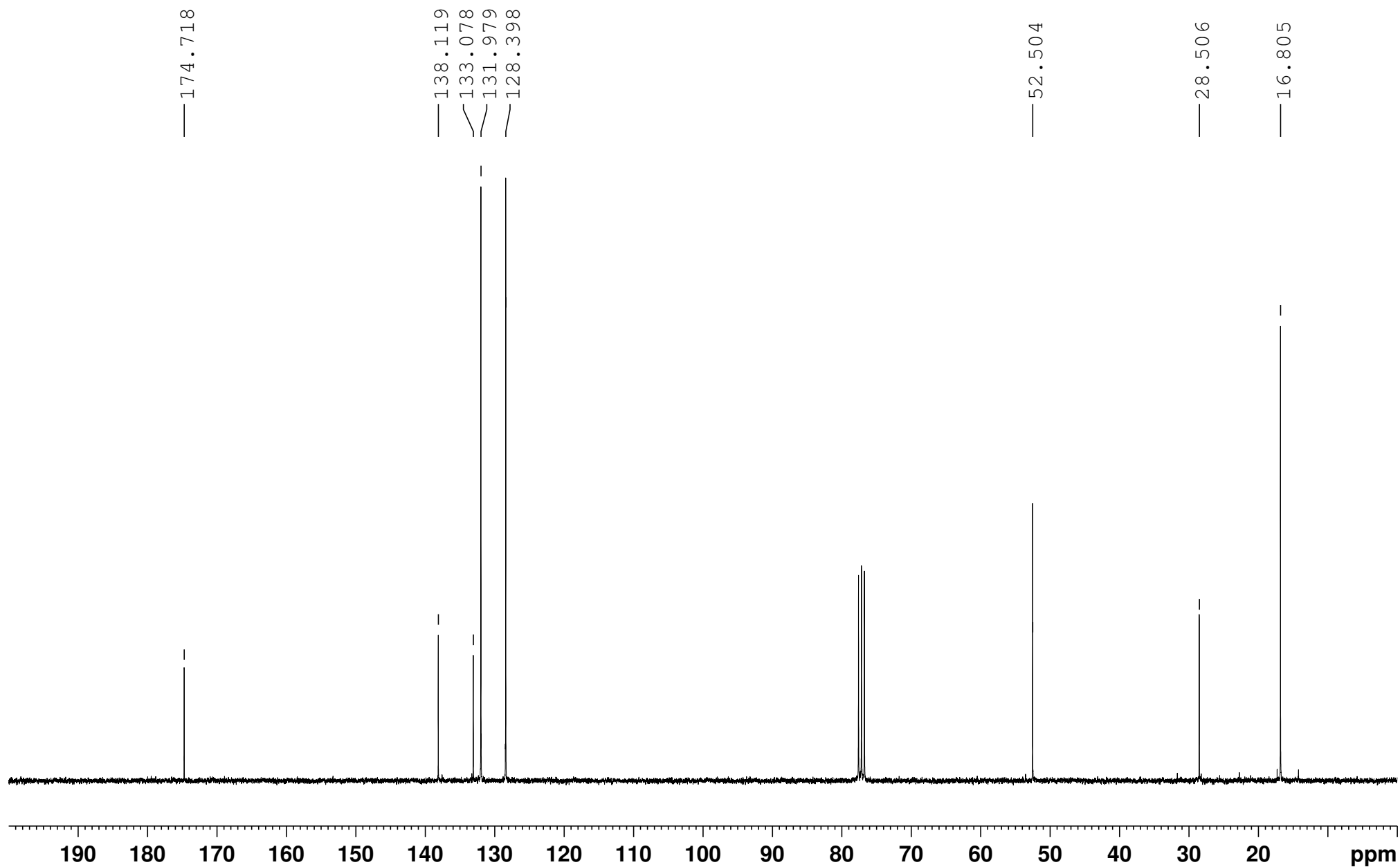
methyl [1,1'-biphenyl]-4-carboxylate - ¹³C - CDCl₃ - 75 MHz



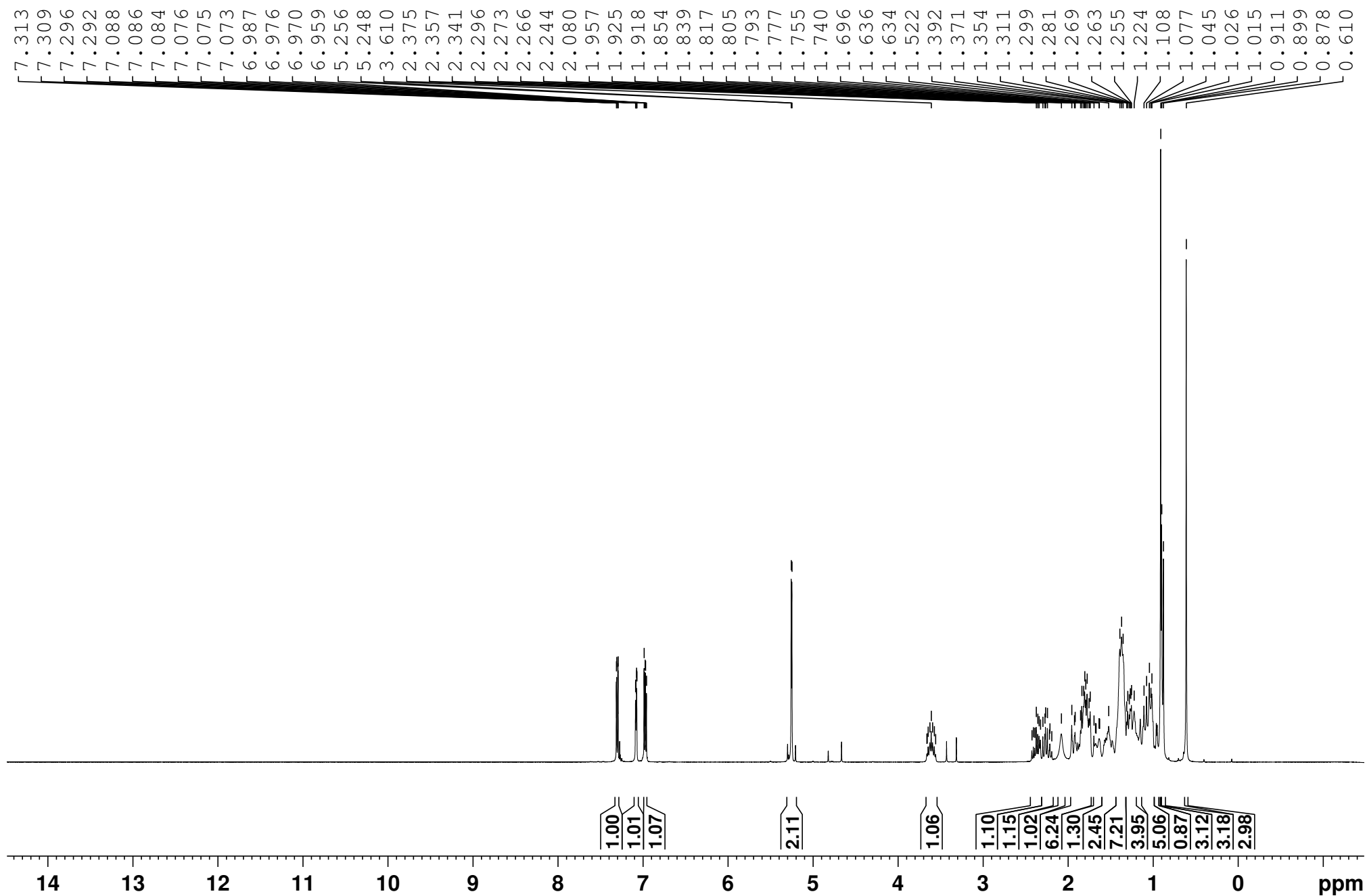
methyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate - 1H - CDCl3 - 300 MHz



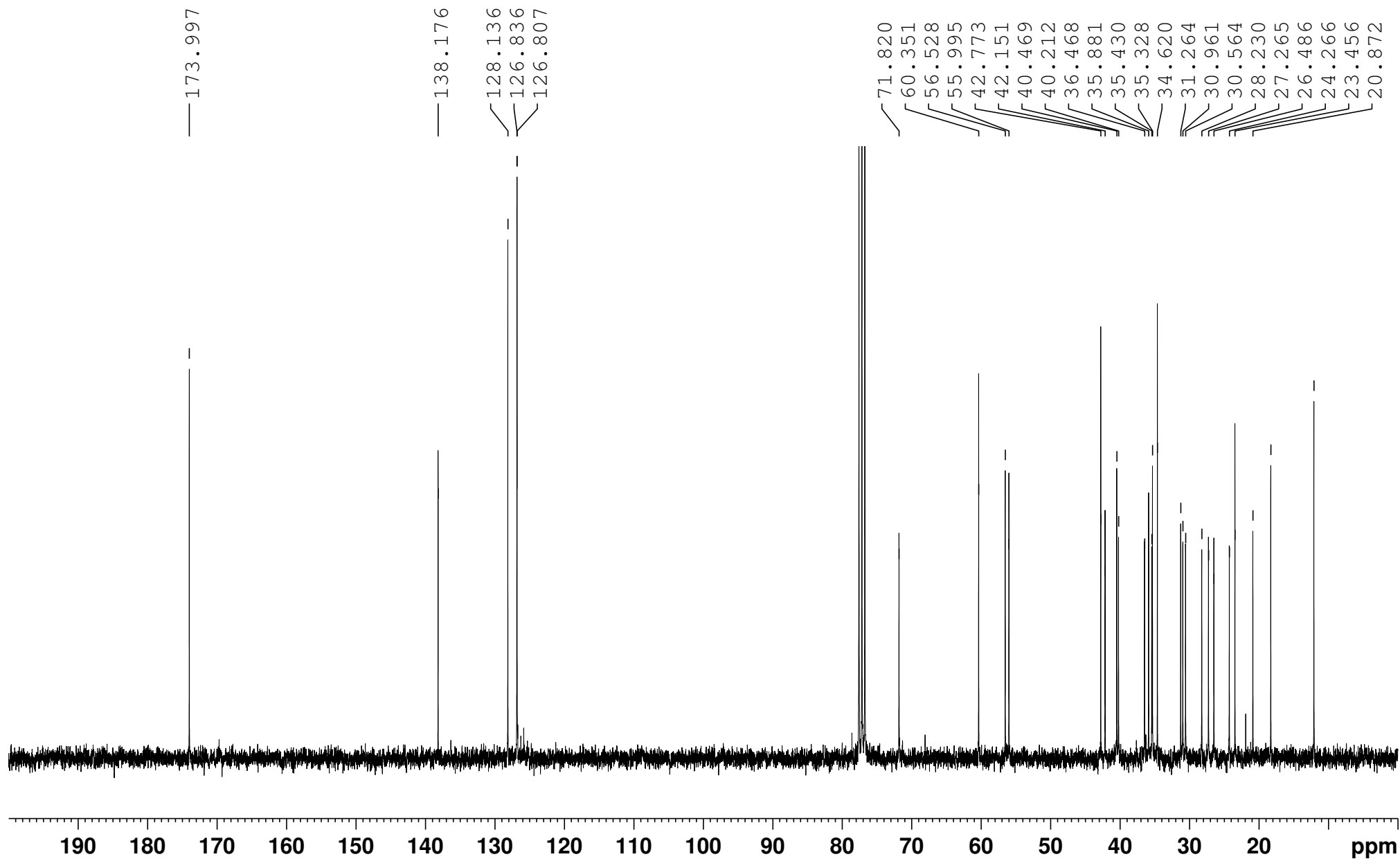
methyl 1-(4-chlorophenyl)cyclopropane-1-carboxylate - ¹³C - CDCl₃ - 75 MHz



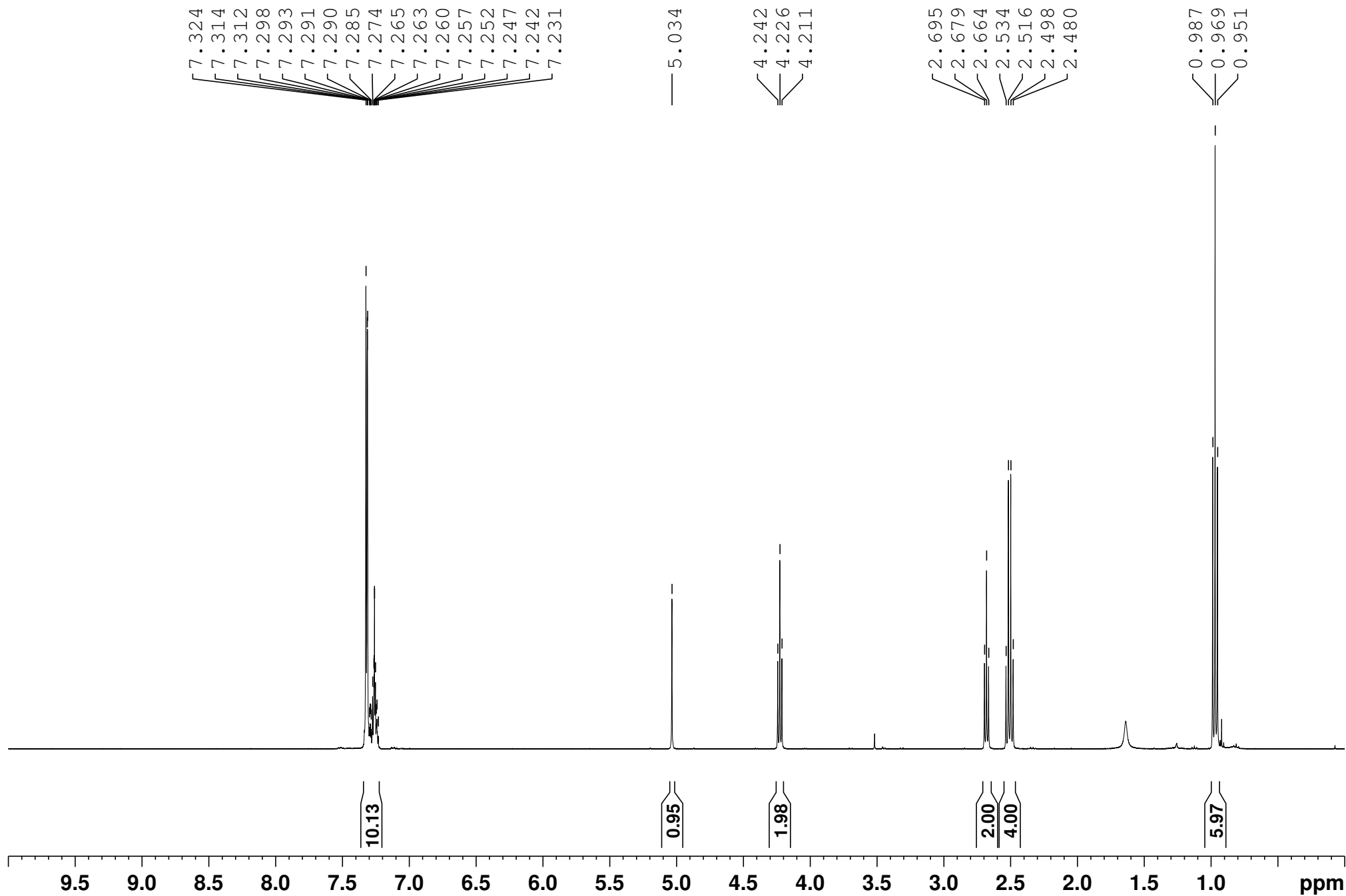
thiophen-2-ylmethyl (R)-4-((3R,5R,8R,9S,10S,13R,14S,17R)-3-hydroxy-10,13-dimethylhexadecahydro-1H-cycloper
[a]phenanthren-17-yl)pentanoate - 1H - CDCl3 - 300 MHz



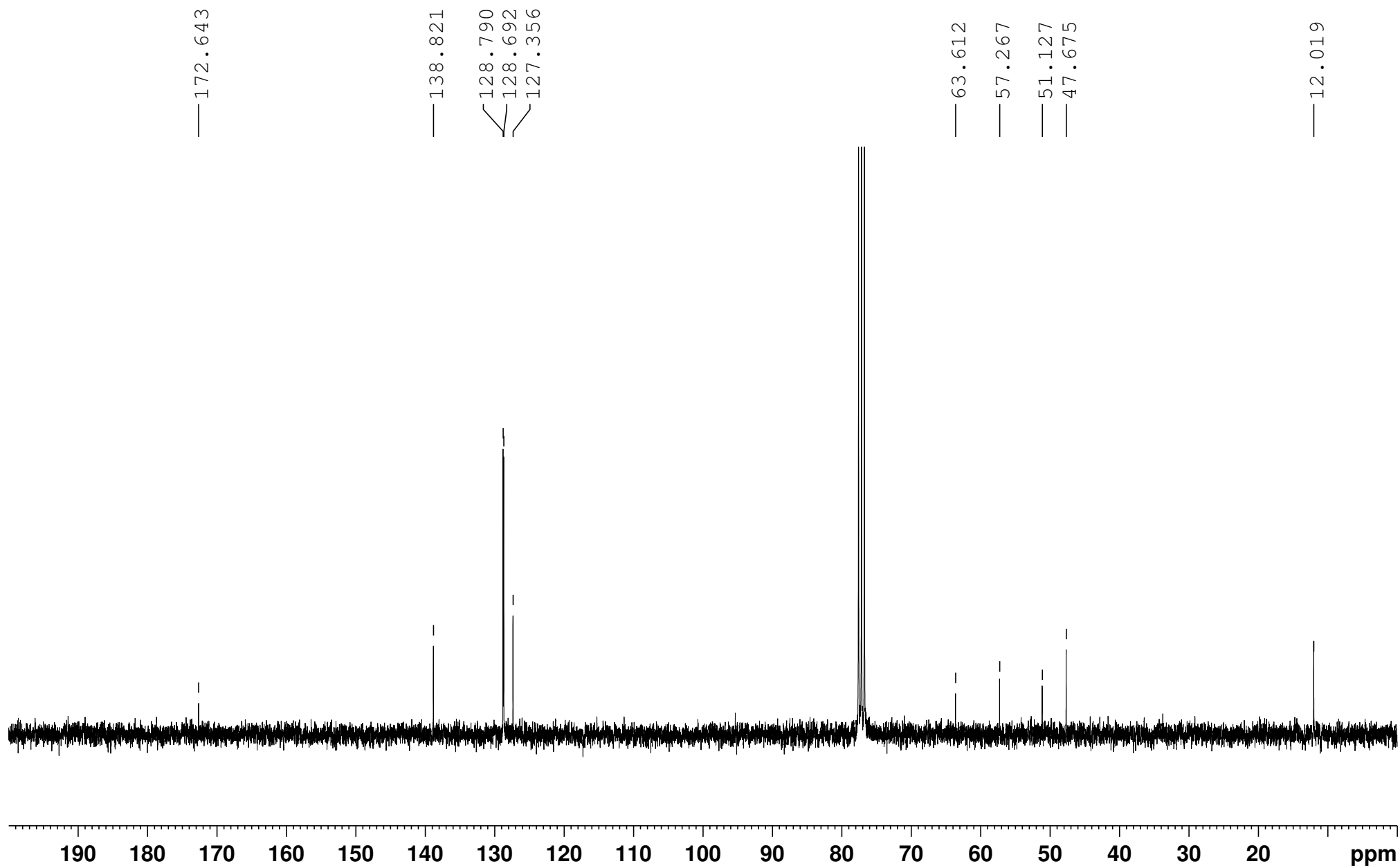
thiophen-2-ylmethyl (R)-4-((3R,5R,8R,9S,10S,13R,14S,17R)-3-hydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate - ¹³C - CDCl₃ - 75 MHz



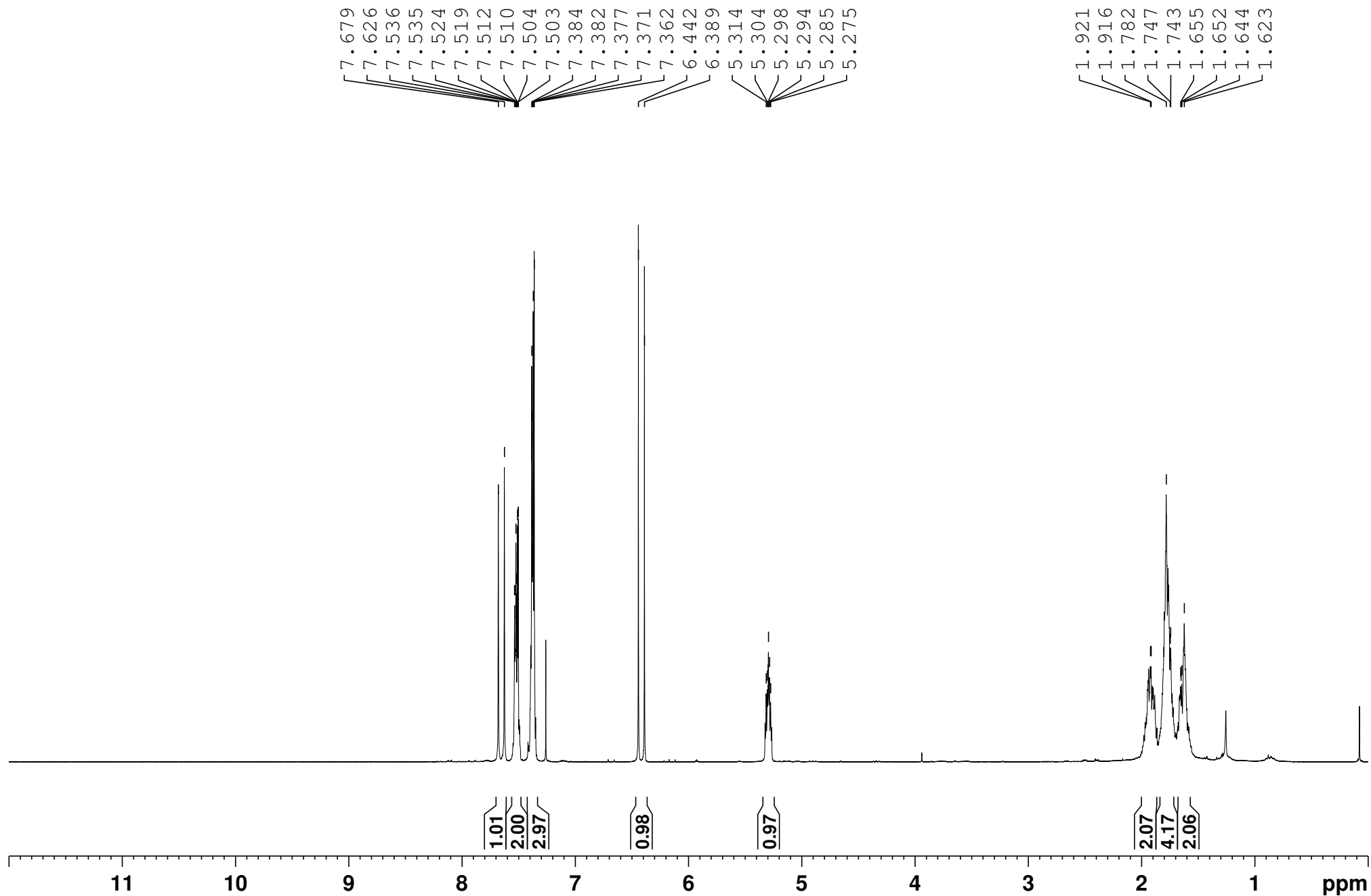
2-(diethylamino)ethyl 2,2-diphenylacetate - 1H - CDCl3 - 400 MHz



2-(diethylamino)ethyl 2,2-diphenylacetate - ¹³C - CDCl₃ - 75 MHz



Cyclopentyl cinnamate - 1H - CDCl3 - 300 MHz



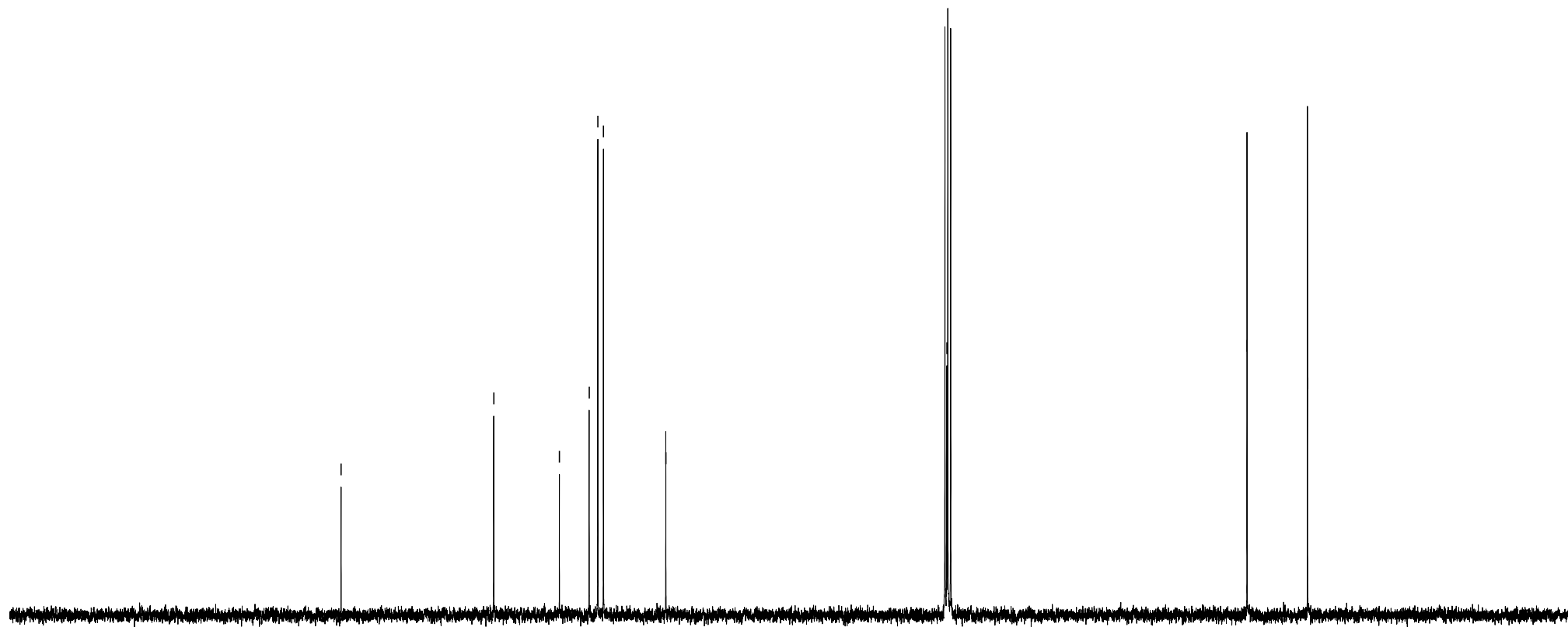
Cyclopentyl cinnamate - ¹³C - CDCl₃ - 75 MHz

— 166.968
— 144.373
— 134.652
— 130.249
— 128.968
— 128.137
— 118.903

— 77.309

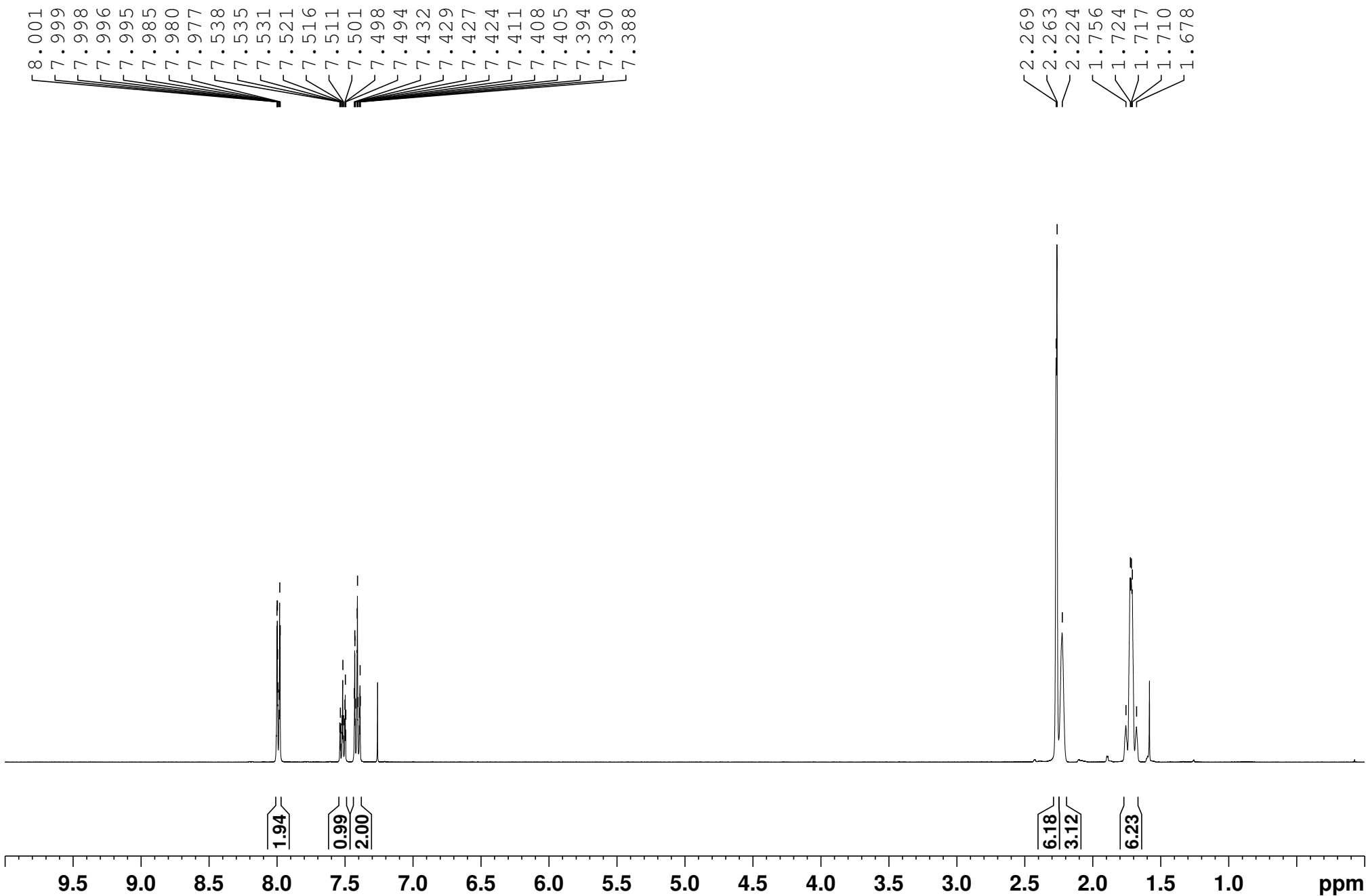
— 32.869

— 23.921

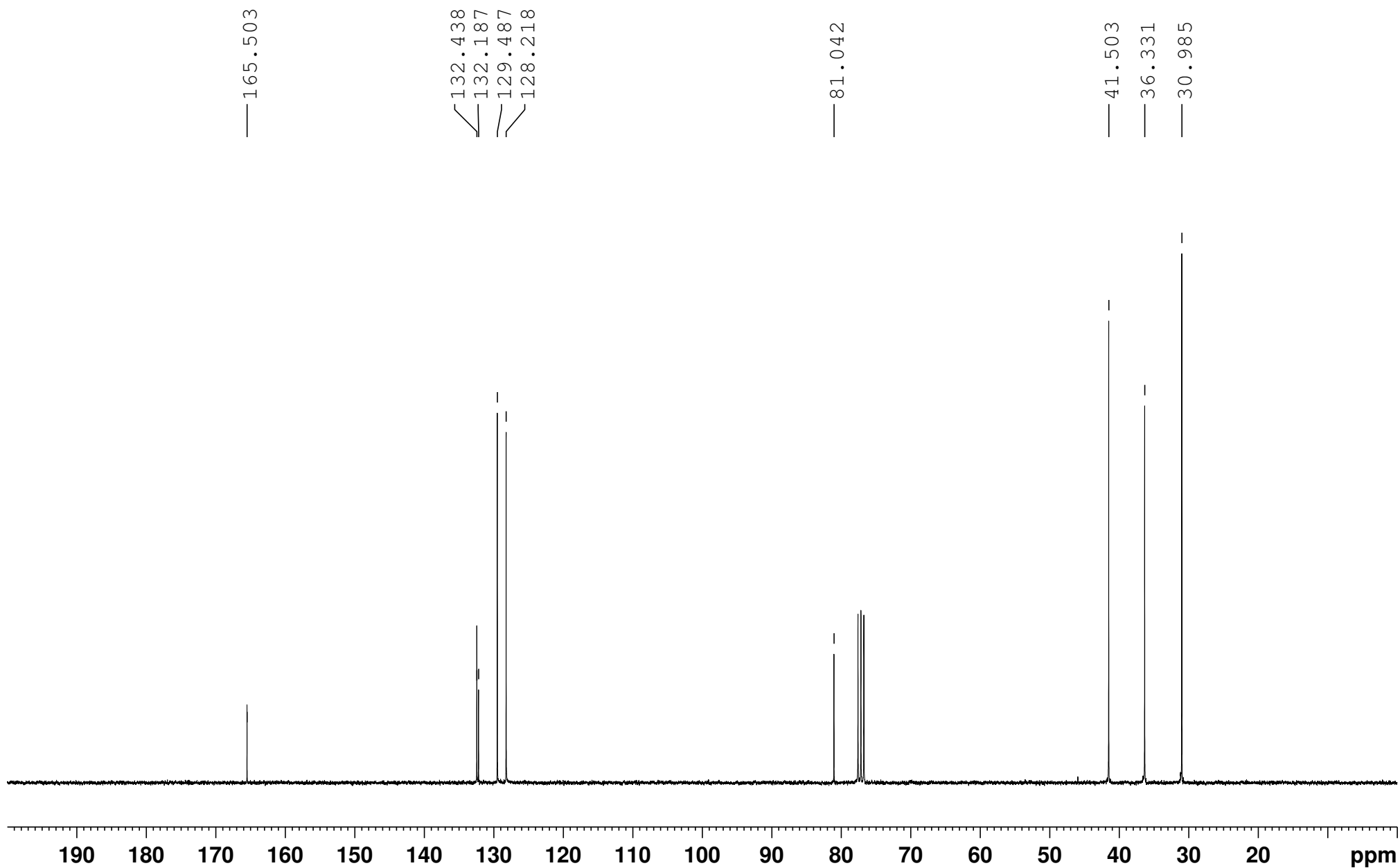


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

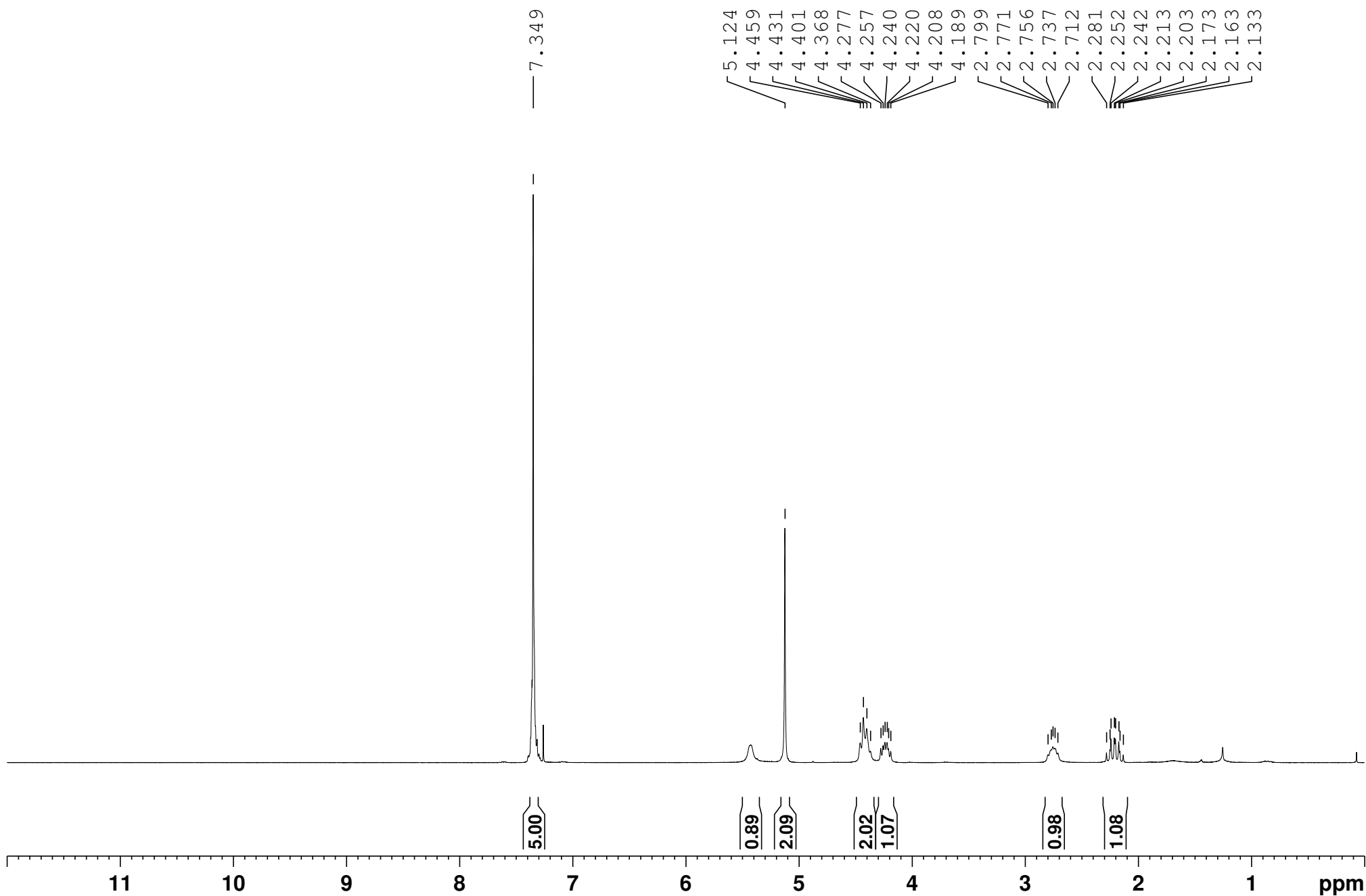
adamantan-1-yl benzoate - 1H - CDCl3 - 400 MHz



adamantan-1-yl benzoate - ^{13}C - CDCl_3 - 75 MHz



Benzyl (S)-(2-oxotetrahydrofuran-3-yl) carbamate - 1H - CDCl3 - 300 MHz



Benzyl (S)-(2-oxotetrahydrofuran-3-yl) carbamate - ¹³C - CDCl₃ - 75 MHz

