

Supporting Information

Rapid Access to Cinnamamides and Piper Amides via Three Component Coupling of Arylaldehyde, Amines, and Meldrum's Acid

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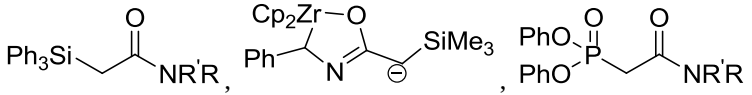
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Experimental:

General: All reactions involving air- or moisture-sensitive reagents or intermediates were carried out in oven-dried glassware under an argon atmosphere. Dichloromethane (CH_2Cl_2) was freshly distilled from phosphorus(V)oxide (P_2O_5). Commercial grade xylene, benzene and toluene were distilled over CaH_2 before use. All other solvents and reagents were purified according to standard procedures or were used as received from Aldrich, Acros, Merck and Spectrochem. ^1H , ^{13}C NMR spectroscopy: *Bruker 600 MHz* (at 298 K), *Bruker 400 MHz* (at 298 K). Chemical shifts, δ (in ppm), are reported relative to TMS δ (^1H) 0.0 ppm, δ (^{13}C) 0.0 ppm) which was used as the inner reference. Otherwise the solvents residual proton resonance and carbon resonance (CHCl_3 , δ (^1H) 7.26 ppm, δ (^{13}C) 77.2 ppm; CD_3OD , (^1H) 3.31 ppm, δ (^{13}C) 49.0 ppm) were used for calibration. Column chromatography: Merck or Spectrochem silica gel 60-120 under gravity. MS (ESI-HRMS): Mass spectra were recorded on an Agilent Accurate-Mass Q-TOF LC/MS 6520, and peaks are given in m/z (% of basis peak). IR: IR spectra were recorded on an Agilent Accurate-Mass Q-TOF LC/MS 6520.

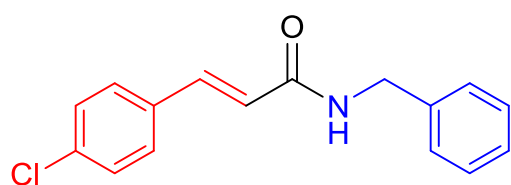
Table s1: Comparison with known methods.

Number of steps from Arylaldehyde ^a	Overall yield (range)	Coupling agents, reagents/catalyst, oxidants, base, etc. leading to chemical waste	Reference
Cinnamamide from cinnamaldehyde			
4-5	7-56%	Phosphine based Wittig salt, K- ^t OBu, cobalt(II) chloride, PPh ₃ , NH ₂ OH, diacetoxyiodobenzene (DIB), Ionic liquid, [Rh(OTf)(trop ₂ NH)(PPh ₃)], Methylmethacrylate, K ₃ PO ₄ , PhI(OAc) ₂ , Cs ₂ CO ₃ , K ₂ CO ₃ , N-hydroxysuccinimide (NHSI), N-heterocyclic carbene (NHC) (For structure see the manuscript. Schme-1), TEMPO, 3,3',5,5'-tetra- <i>tert</i> -butyldiphenylquinone, HBF ₄ .OEt ₂ , Trifluoromethane sulfonic acid (TfOH),	10a – 10l
Cinnamamide from cinnamic acid			
2	42 -90%	Pyridine (as solvent), piperidine, (Benzotriazol-1-yloxy)tris(dimethylamino)phosphonium hexafluorophosphate (BOP), 1- <i>tert</i> -butoxy-2- <i>tert</i> -butoxycarbonyl-1,2-dihydroisoquinoline (BDDI), PhSiH ₃ , Butyl(hydroxy)stannanone, PPh ₃ /CBrCl ₃ , Zr-complex, PPh ₃ /NCBT (<i>N</i> -chlorobenzotriazole), Et ₃ N, ZrCp ₂ Cl ₂ or ZrCl ₄ , silicone supported boronic acid, TiO ₂ , PhSiH ₂ .	9a - 9q
Cinnamamide from arylaldehyde			
1 (3 or more steps are used to prepare the ylide precursor)	38 - 90% (E/Z selectivity was poor for some cases)		11a - 11d
This method			
1	58 - 73%	No additional reagents used. CO ₂ and acetone are the byproducts.	

^aArylaldehyde was taken as the starting materials for the comparison.

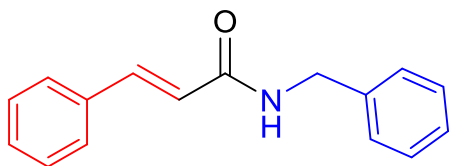
General procedure for the synthesis of cinnamamide (GP I): Arylaldehyde (0.94 – 0.56 mmol) was added to a solution of amine (0.94 – 0.56 mmol) and Meldrum's acid (0.94 – 0.56 mmol) in toluene (2 – 4 mL) and the mixture was refluxed (110 °C) for 8 h. After disappearance of starting materials (indicated by TLC), solvent was evaporated under reduced pressure. The crude mixture was purified by column chromatography to get analytically pure product.

(E)-N-benzyl-3-(4-chlorophenyl)acrylamide (6a)⁶: According to GP I, 4-chlorobenzaldehyde



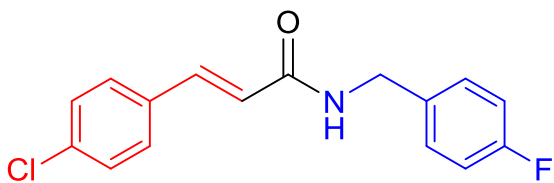
(0.10 g, 0.71 mmol), benzylamine (76 mg, 0.71 mmol) and Meldrum's acid (0.10 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6a** as white solid (0.14 g, 68%). ¹H NMR (600 MHz, CDCl₃) δ = 7.64 (d, *J* = 15.6 Hz, 1H), 7.43 (d, *J* = 8.4 Hz, 3H), 7.39 – 7.29 (m, 6H), 6.41 (d, *J* = 15.6 Hz, 1H), 6.07 (br. s, 1H), 4.59 (d, *J* = 5.8 Hz, 2H) ppm. ¹³C NMR (151 MHz, CDCl₃) δ = 165.5, 140.1, 138.1, 135.6, 133.2, 129.1, 129.0, 128.8, 127.9, 127.7, 120.9, 43.9 ppm.

(E)-N-benzylcinnamamide (6b)^{1,6}: According to GP I, benzaldehyde (0.1 g, 0.94 mmol), benzylamine (0.1 g, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was reacted for 8 h and



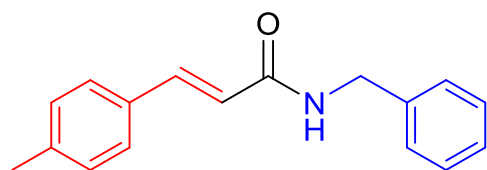
the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6b** as white solid (0.14 g, 63%). ¹H NMR (400 MHz, CDCl₃) δ = 7.56 (d, *J* = 15.6 Hz, 1H), 7.37 – 7.35 (m, 2H), 7.26 – 7.13 (m, 8H), 6.38 (d, *J* = 15.6 Hz, 1H), 6.36 (br. s, 1H), 4.43 (d, *J* = 5.8 Hz, 2H). ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 166.0, 141.3, 138.3, 134.8, 129.7, 128.8, 128.7, 127.9, 127.8, 127.5, 120.7, 43.82 ppm.

(E)-N-(4-fluorobenzyl)-3-(4-chlorophenyl)acrylamide (6c): According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), 4-fluorobenzylamine (89 mg, 0.71 mmol) and



Meldrum's acid (0.10 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6c** as colourless gum (0.13 g, 63%). FTIR: $\tilde{\nu}$ = 3288, 1654, 1622, 1510, 1224, 1091, 821 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ = 7.56 (d, *J* = 15.6 Hz, 1H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.30 – 7.20 (m, 4H), 6.96 (t, *J* = 8.4 Hz, 2H), 6.30 (d, *J* = 15.6 Hz, 1H), 5.84 (br. s, 1H), 4.48 (d, *J* = 5.7 Hz, 2H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 164.4, 139.4, 134.7, 132.9, 132.1, 128.6, 128.6, 128.1, 128.0, 119.7, 114.7, 114.5, 42.2 ppm. (Increased number of ¹³C signal is observed due to F-coupling). HRMS: Exact mass calculated for C₁₆H₁₃ClFNO ([M+H]⁺): 290.0742, Found: 290.0742.

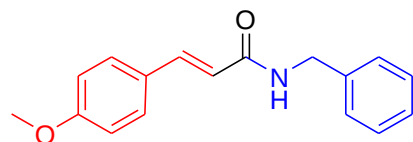
(E)-N-benzyl-3-*p*-tolylacrylamide (6d)⁷: According to GP I, 4-methylbenzaldehyde (0.10 g, 0.82 mmol), benzylamine (88 mg, 0.82 mmol) and Meldrum's acid (1.20 g, 0.82 mmol) was



reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6d** as white solid (0.13 g, 63%). ¹H NMR (600 MHz, CDCl₃) δ = 7.66 (d, *J* = 15.6

Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.37 – 7.31 (m, 4H), 7.31 – 7.26 (m, 1H), 7.16 (d, *J* = 7.8 Hz, 2H), 6.43 (d, *J* = 15.6 Hz, 1H), 6.29 (br. s, 1H), 4.56 (d, *J* = 5.6 Hz, 2H), 2.37 (s, 3H) ppm. ¹³C NMR (151 MHz, CDCl₃) δ = 166.1, 141.3, 140.0, 138.3, 132.1, 129.5, 128.7, 127.9, 127.8, 127.5, 119.5, 43.2, 21.4 ppm.

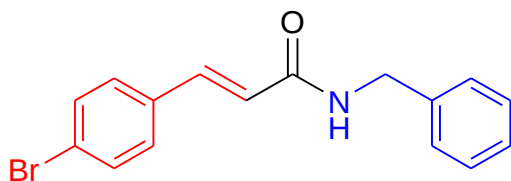
(E)-N-benzyl-3-(4-methoxyphenyl)acrylamide (6e)⁶: According to GP I, 4-methoxybenzaldehyde (0.10 g, 0.94 mmol), benzylamine (0.10 g, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was reacted for 8 h and the crude mixture was



purified by column chromatography (Silica gel; EtOAc: Hexane, 1:3) to get analytically pure product **6e** as white solid (0.18 g, 70%). ¹H NMR (400 MHz, CDCl₃) δ = 7.53 (d, *J* = 15.6

Hz, 1H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.27 – 7.16 (m, 5H), 6.77 (d, *J* = 8.7 Hz, 2H), 6.23 (d, *J* = 15.6 Hz, 1H), 6.10 (br. s, 1H), 4.46 (d, *J* = 5.8 Hz, 2H), 3.73 (s, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 166.2, 160.9, 140.9, 138.4, 129.4, 128.7, 127.9, 127.54, 127.48, 118.2, 114.3, 55.3, 43.8 ppm.

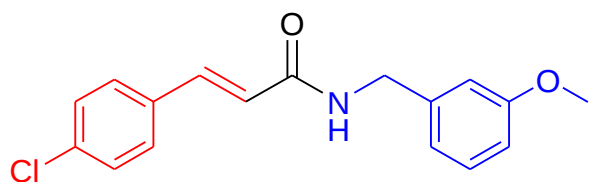
(E)-N-benzyl-3-(4-bromophenyl)acrylamide (6f)⁶: According to GP I, 4-bromo benzaldehyde (0.10 g, 0.54 mmol), benzylamine (58 mg, 0.54 mmol) and Meldrum's acid (78 mg, 0.54 mmol)



was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6f** as white solid (0.12 g, 70%). ¹H NMR (600 MHz,

CDCl₃) δ = 7.59 (d, *J* = 15.6 Hz, 1H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.35 – 7.26 (m, 7H), 6.40 (d, *J* = 15.6 Hz, 1H), 6.06 (br. s, 1H), 4.56 (d, *J* = 6.0 Hz, 2H). ¹³C NMR (151 MHz, CDCl₃) δ = 165.4, 140.2, 138.1, 133.7, 132.1, 129.2, 128.8, 127.9, 127.7, 123.9, 121.1, 43.9.

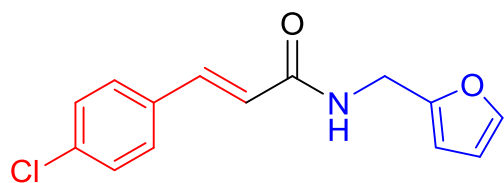
(E)-N-(4-methoxybenzyl)-3-(4-chlorophenyl)acrylamide (6g): According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), 3-methoxybenzylamine (98 mg, 0.71 mmol) and Meldrum's



acid (0.1 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6g** as colourless

gum (0.15 g, 68%). FTIR: $\tilde{\nu}$ = 2917, 2850, 1735, 1656, 1619, 1491, 1465, 1243, 1091, 1027, 821, 752 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ = 7.58 (d, J = 15.6 Hz, 1H), 7.41 (d, J = 8.4 Hz, 2H), 7.31 – 7.28 (m, 4H), 6.96 – 6.87 (m, 2H), 6.35 (d, J = 15.6 Hz, 1H), 6.12 (br. s, 1H), 4.57 (d, J = 6.0 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ = 165.3, 157.6, 139.6, 135.4, 133.4, 130.0, 129.0, 128.9, 126.1, 121.4, 120.8, 110.3, 55.4, 39.7 ppm. (Reduced number of ^{13}C signal is observed due to overlapping in aromatic region). HRMS: Exact mass calculated for $\text{C}_{17}\text{H}_{16}\text{ClNO}_2$ ($[\text{M}+\text{H}]^+$): 302.0942, Found: 302.0947.

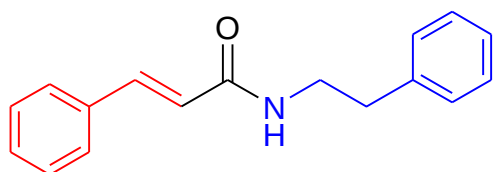
(E)-3-(4-chlorophenyl)-N-((furan-2-yl)methyl)acrylamide (6h): According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), furfurylamine (70 mg, 0.71 mmol) and Meldrum's acid



(0.1 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:3) to get analytically pure product **6h** as colourless gum (0.12 g, 62%). FTIR: $\tilde{\nu}$ = 3287,

1655, 1625, 1551, 1491, 1405, 1328, 1225, 1149, 1090, 975, 820, 737 cm^{-1} . ^1H NMR (600 MHz, CDCl_3) δ = 7.61 (d, J = 15.6 Hz, 1H), 7.42 (d, J = 8.4 Hz, 2H), 7.37 – 7.33 (m, 3H), 6.36 (d, J = 15.6 Hz, 1H), 6.34 – 6.33 (m, 1H), 6.29 – 6.27 (m, 1H), 5.95 (br. s, 1H), 4.57 (d, J = 5.4 Hz, 2H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ = 165.3, 151.0, 142.3, 140.3, 135.6, 133.2, 129.1, 129.0, 120.7, 110.6, 107.7, 36.7 ppm. HRMS: Exact mass calculated for $\text{C}_{14}\text{H}_{12}\text{ClNO}_2$ ($[\text{M}+\text{H}]^+$): 262.0629, Found: 262.0624.

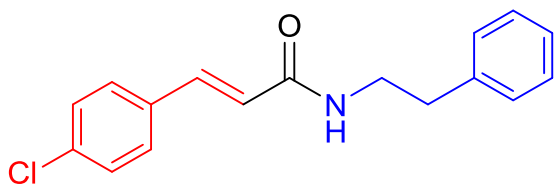
(E)-N-phenethylcinnamamide (6i)⁹: According to GP I, benzaldehyde (0.1 g, 0.94 mmol), phenethylamine (0.10 g, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was reacted for 8 h



and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:5) to get analytically pure product **6i** as colourless liquid (0.14 g,

61%). ^1H NMR (400 MHz, CDCl_3) δ = 7.54 (d, J = 15.6 Hz, 1H), 7.42 – 7.52 (m, 2H), 7.30 – 7.23 (m, 5H), 7.19 – 7.14 (m, 3H), 6.25 (d, J = 15.6 Hz, 1H), 5.64 (br. s, 1H), 3.61 – 3.56 (m, 2H), 2.84 – 2.80 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ = 165.9, 141.1, 138.9, 134.8, 129.7, 128.8, 128.8, 128.7, 127.8, 126.6, 120.6, 40.8, 35.7 ppm.

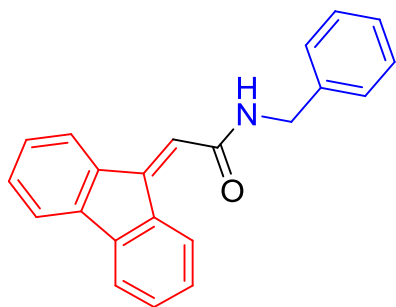
(E)-3-(4-chlorophenyl)-N-phenethylacrylamide (6j)⁸: According to GP I, 4-chloro benzaldehyde (0.1 g, 0.71 mmol), phenethylamine (86 mg, 0.71 mmol) and Meldrum's acid (0.1



g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:5) to get analytically pure product **6j** as colourless liquid

(0.13 g, 67%). ^1H NMR (400 MHz, CDCl_3) δ = 7.50 (d, J = 15.6 Hz, 1H), 7.34 (d, J = 8.4 Hz, 2H), 7.28 – 7.24 (m, 4H), 7.19 – 7.16 (m, 3H), 6.21 (d, J = 15.6 Hz, 1H), 5.56 (br. s, 1H), 3.62 – 3.57 (m, 2H), 2.82 (t, J = 6.8 Hz, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ = 165.5, 139.8, 138.8, 135.5, 133.3, 129.1, 128.9, 128.8, 128.7, 126.6, 121.1, 40.8, 35.6 ppm.

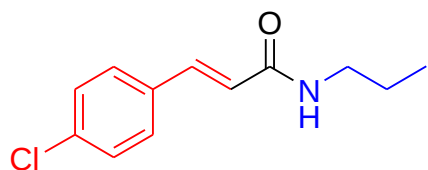
(E)-N-benzyl-3-(9H-fluoren-9-yl)acrylamide (6k): According to GP I, 9-fluorenone (0.10 g, 0.56 mmol), benzylamine (59.0 mg, 0.56 mmol) and Meldrum's acid (80 mg, 0.56 mmol) was



reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6k** as yellow solid (0.11 g, 60%). Mp: 167 – 169 °C. FTIR: $\tilde{\nu}$ = 3295, 2918, 2850, 1644, 1628, 1529, 1441, 1247, 1232, 780, 727, 698 cm^{-1} . ^1H NMR (600 MHz, CDCl_3) δ = 8.56 (d, J = 7.8 Hz, 1H), 7.67 - 7.64 (m, 2H), 7.41 -

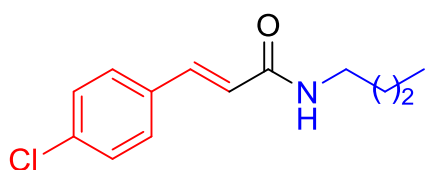
7.37 (m, 6H), 7.35 – 7.34 (m, 1H), 7.28 - 7.24 (m, 3H), 6.76 (s, 1H), 6.29 (br. s, 1H), 4.66 (d, J = 5.6 Hz, 2H) ppm. ^{13}C NMR (151 MHz, CDCl_3) δ = 166.1, 143.6, 141.9, 140.4, 138.6, 137.7, 135.3, 130.2, 129.9, 128.9, 128.2, 127.9, 127.79, 127.77, 127.3, 120.7, 119.8, 119.6, 117.1, 44.0 ppm. HRMS: Exact mass calculated for $\text{C}_{22}\text{H}_{17}\text{NO}$ ($[\text{M}+\text{H}]^+$): 312.1383, Found: 312.1388.

(E)-3-(4-chlorophenyl)-N-propylacrylamide (6l): According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), n-propylamine (42 mg, 0.71 mmol) and Meldrum's acid (0.10 g, 0.71 mmol)



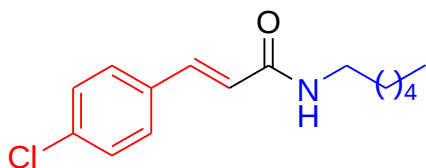
was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6l** as colourless gum (0.10 g, 68%). FTIR: $\tilde{\nu}$ = 3281, 2963, 2927, 1656, 1618, 1551, 1510, 1491, 1406, 1337, 1222, 1092, 1013, 979, 820 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ = 7.51 (d, J = 15.6 Hz, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 6.29 (d, J = 15.6 Hz, 1H), 5.57 (br. s, 1H), 3.32 – 3.27 (m, 2H), 1.56 – 1.50 (m, 2H), 0.90 (t, J = 7.4 Hz, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ = 165.6, 139.7, 135.5, 133.4, 129.1, 128.9, 121.2, 41.6, 22.9, 11.4 ppm. HRMS: Exact mass calculated for $\text{C}_{12}\text{H}_{14}\text{ClNO}$ ($[\text{M}+\text{H}]^+$): 224.0837, Found: 224.0837.

(E)-N-butyl-3-(4-chlorophenyl)acrylamide (6m)²: According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), n-butylamine (52 mg, 0.71 mmol) and Meldrum's acid (0.10 g, 0.71 mmol)



was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **6m** as colourless gum (0.10 g, 63%). ^1H NMR (400 MHz, CDCl_3) δ = 7.59 (d, J = 15.6 Hz, 1H), 7.45 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 8.4 Hz, 2H), 6.36 (d, J = 15.6 Hz, 1H), 5.60 (br. s, 1H), 3.44 – 3.39 (m, 2H), 1.62 – 1.54 (m, 2H), 1.46 – 1.39 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H). ppm. ^{13}C NMR (151 MHz, CDCl_3) δ = 165.5, 149.7, 139.6, 136.8, 129.1, 128.9, 121.3, 39.6, 31.7, 20.1, 13.8 ppm.

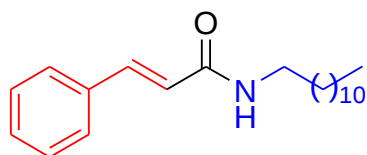
(E)-3-(4-chlorophenyl)-N-hexylacrylamide (6n): According to GP I, 4-chlorobenzaldehyde (0.1g, 0.71 mmol), n-hexylamine (72 mg, 0.71 mmol) and Meldrum's acid (0.1 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel;



EtOAc: Hexane, 1:5) to get analytically pure product **6n** as white solid (0.12 g, 62%). Mp: 113 – 115°C. FTIR: $\tilde{\nu}$ = 3286, 2927, 2857, 1654, 1619, 1545, 1491, 1339, 1221, 1094, 972, 820 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ = 7.50 (d, J = 15.4 Hz, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 6.27 (d, J = 15.4 Hz, 1H), 5.52 (br. s, 1H), 3.34 – 2.29 (m, 2H), 1.42 – 1.05 (m, 8H), 0.84 – 0.81 (m, 3H) ppm. ^{13}C NMR (151 MHz, CDCl_3) δ = 165.5, 139.5, 135.4, 133.4,

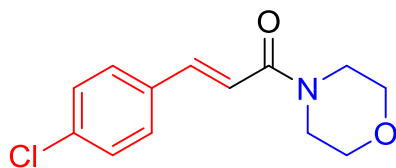
129.1, 128.9, 121.3, 39.85, 31.48, 29.6, 26.6, 22.6, 14.0 ppm. HRMS: Exact mass calculated for $C_{15}H_{20}ClNO$ ($[M+H]^+$): 266.1306, Found: 266.1304.

(E)-N-decylcinnamamide (6o)¹²: According to GP I, benzaldehyde (0.10 g, 0.94 mmol), n-dodecylamine (0.18 g, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was reacted for 8 h



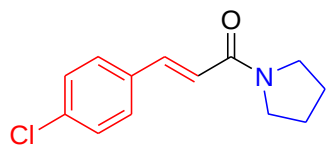
and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:5) to get analytically pure product **6o** as white solid (0.19 g, 71%). 1H NMR (400 MHz, $CDCl_3$) δ = 7.62 (d, J = 15.6 Hz, 1H), 7.53 – 7.45 (m, 2H), 7.40 – 7.31 (m, 3H), 6.39 (d, J = 15.6 Hz, 1H), 5.59 (br. s, 1H), 3.41 – 3.36 (m, 2H), 1.60 – 1.53 (m, 7H), 1.33 – 1.26 (m, 13H), 0.88 (t, J = 6.8 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ = 165.8, 140.8, 134.9, 129.6, 128.8, 127.7, 120.8, 39.8, 31.9, 29.7, 29.64, 29.62, 29.58, 29.54, 29.3, 29.3, 26.0, 22.7, 14.1 ppm.

(E)-3-(4-chlorophenyl)-1-morpholinoprop-2-en-1-one (6p)⁴: According to GP I, 4-chlorobenzaldehyde (0.1 g, 0.71 mmol), morpholine (62 mg, 0.71 mmol) and Meldrum's acid



(0.10 g, 0.71 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:2) to get analytically pure product **6p** as white solid (0.11 g, 61%). Mp: 142 – 143°C. FTIR: $\tilde{\nu}$ = 2966, 2917, 2851, 1648, 1610, 1494, 1430, 1405, 1230, 1112, 1048, 1011, 820 cm^{-1} . 1H NMR (600 MHz, $CDCl_3$) δ = 7.64 (d, J = 15.4 Hz, 1H), 7.44 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 6.81 (d, J = 15.4 Hz, 1H), 3.76 – 3.62 (m, 8H). ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 165.3, 141.9, 135.6, 133.6, 129.1, 129.0, 117.0, 66.8, 46.3, 42.5 ppm. HRMS: Exact mass calculated for $C_{13}H_{14}ClNO_2$ ($[M+H]^+$): 252.0786, Found: 252.0789.

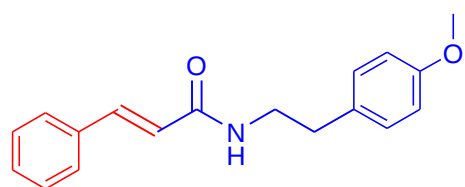
(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one (6q)³: According to GP I, 4-chlorobenzaldehyde (0.10 g, 0.71 mmol), pyrrolidine (51 mg, 0.71 mmol) and Meldrum's acid (0.10 g, 0.71 mmol) was reacted for 8 h and the crude mixture was



purified by column chromatography (Silica gel; EtOAc: Hexane, 1:2) to get analytically pure product **6q** as white solid (0.12 g, 70%). 1H NMR (400 MHz, $CDCl_3$) δ = 7.62 (d, J = 15.6 Hz, 1H), 7.43 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 6.68 (d, J = 15.6 Hz, 1H), 3.62 – 3.55 (m, 4H), 2.02 – 1.97 (m,

2H), 1.91 – 1.85 (m, 2H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ = 164.4, 140.3, 135.3, 133.9, 129.00, 119.4, 46.6, 46.1, 26.1, 24.3 ppm.

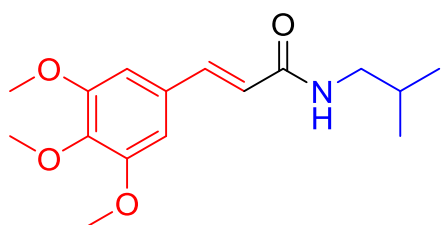
(E)-N-(4-methoxyphenethyl)cinnamamide (8a)⁷: According to GP I, benzaldehyde (0.1 g, 0.94 mmol), 4-methoxyphenethylamine (0.14 g, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol)



was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **8a** as colourless gum (0.18 g, 68%). ^1H NMR (400 MHz, CDCl_3) δ = 7.62 (d, J = 15.4 Hz,

1H), 7.49 – 7.46 (m, 2H), 7.36 – 7.34 (m, 3H), 7.14 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 6.33 (d, J = 15.4 Hz, 1H), 5.70 (br. s, 1H), 3.80 (s, 3H), 3.65 – 3.60 (m, 2H), 2.85 – 2.81 (m, 2H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ = 165.9, 158.3, 141.1, 134.8, 130.8, 129.8, 129.7, 128.8, 127.8, 120.5, 114.1, 55.3, 41.0, 34.7 ppm.

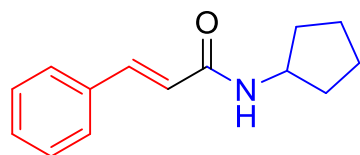
(E)-N-isobutyl-3-(3,4,5-trimethoxyphenyl)acrylamide (8b): According to GP I, 3,4,5-trimethoxybenzaldehyde (0.10 g, 0.51 mmol), isobutylamine (37 mg, 0.51 mmol) and Meldrum's



acid (73 mg, 0.51 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:3) to get analytically pure product **8b** as white solid (90 mg, 60%). Mp: 145 – 146°C. FTIR: $\tilde{\nu}$ = 3302, 2917, 2850, 1736, 1655, 1619, 1581, 1546, 1508,

1467, 1417, 1323, 1281, 1241, 1210, 993, 823 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ = 7.48 (d, J = 15.4 Hz, 1H), 6.66 (s, 2H), 6.27 (d, J = 15.4 Hz, 1H), 5.70 (br. s, 1H), 3.81 (s, 6H), 3.80 (s, 3H), 3.18 – 3.14 (m, 2H), 1.81 – 1.75 (m, 1H), 0.89 (d, J = 6.8 Hz, 6H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ = 165.9, 153.4, 141.0, 139.5, 130.4, 120.0, 104.9, 61.0, 56.1, 47.1, 28.6, 20.2 ppm. HRMS: Exact mass calculated for $\text{C}_{16}\text{H}_{23}\text{NO}_4$ ($[\text{M}+\text{H}]^+$): 294.1700, Found: 294.1700.

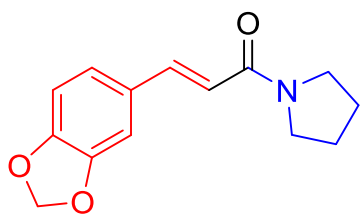
(E)-N-cyclopentylcinnamamide (8c)¹²: According to GP I, benzaldehyde (0.10 g, 0.94 mmol), cyclopentylamine (82 mg, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was reacted for



8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **8c** as white solid (0.14 g, 70%). Mp: 144 – 145°C. FTIR: $\tilde{\nu}$ = 3256, 2959, 2870, 1655, 1615, 1543, 1449, 1338, 1225, 978, 765, 701 cm^{-1}

¹H NMR (400 MHz, CDCl₃) δ = 7.61 (d, *J* = 15.6 Hz, 1H), 7.50 – 7.48 (m, 2H), 7.36 – 7.34 (m, 3H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.63 (br. s, 1H), 4.37 – 4.32 (m, 1H), 2.10 – 2.02 (m, 2H), 1.72 – 1.61 (m, 4H), 1.47 – 1.41 (m, 2H). ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 165.5, 140.8, 134.9, 129.6, 128.8, 127.7, 120.9, 51.5, 33.3, 23.8 ppm. HRMS: Exact mass calculated for C₁₄H₁₇NO ([M+H]⁺): 216.1383, Found: 216.1389.

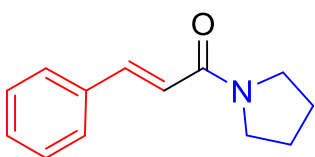
(E)-3-(benzo[d][1,3]dioxol-5-yl)-1-(pyrrolidin-1-yl)prop-2-en-1-one (8d)⁷: According to GP I, piperonyl aldehyde (0.10 g, 0.66 mmol), pyrrolidine (47 mg, 0.66 mmol) and Meldrum's acid (96



mg, 0.66 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:1) to get analytically pure product **8d** as white solid (98 mg, 58%).

¹H NMR (400 MHz, CDCl₃) δ = 7.57 (d, *J* = 15.4 Hz, 1H), 7.03 – 6.95 (m, 2H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.53 (d, *J* = 15.4 Hz, 1H), 5.95 (s, 2H), 3.60 – 3.54 (m, 4H), 2.00 – 1.95 (m, 2H), 1.90 – 1.83 (m, 2H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 164.8, 148.9, 148.2, 141.4, 129.8, 123.8, 116.9, 108.5, 106.4, 101.4, 46.5, 46.0, 26.1, 24.3 ppm.

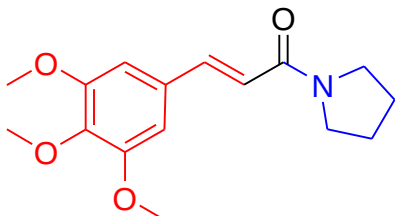
(E)-3-phenyl-1-(pyrrolidin-1-yl)prop-2-en-1-one (8e)^{6,7}: According to GP I, benzaldehyde (0.10 g, 0.94 mmol), pyrrolidine (67 mg, 0.94 mmol) and Meldrum's acid (0.14 g, 0.94 mmol) was



reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:1) to get analytically pure product **8e** as white solid (0.13 g, 72%). ¹H NMR (400 MHz, CDCl₃) δ = 7.63 (d, *J* = 15.6 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.35 – 7.23

(m, 3H), 6.66 (d, *J* = 15.6 Hz, 1H), 3.64 – 3.57 (m, 4H), 2.03 – 1.97 (m, 2H), 1.93 – 1.88 (m, 2H) ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 164.7, 141.7, 135.4, 129.5, 128.8, 127.8, 118.9, 46.6, 46.1, 26.2, 24.4 ppm.

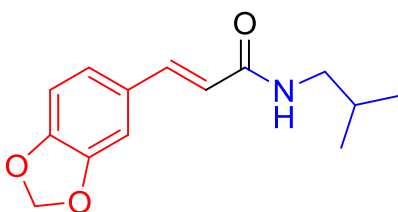
(E)-3-(3,4,5-trimethoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one (8f)⁵: According to GP I, 3,4,5-trimethoxybenzaldehyde (0.10 g, 0.51 mmol), pyrrolidine (36 mg, 0.51 mmol) and



Meldrum's acid **1** (73 mg, 0.51 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:1) to get analytically pure product **8f** as white solid (91 mg, 64%). ¹H NMR (600 MHz, CDCl₃) δ =

7.59 (d, $J = 15.4$ Hz, 1H), 6.73 (s, 2H), 6.60 (d, $J = 15.4$ Hz, 1H), 3.87 (s, 6H), 3.85 (s, 3H), 3.64 – 3.62 (m, 2H), 3.59 – 3.56 (m, 2H), 2.02 – 1.96 (m, 3H), 1.92 – 1.85 (m, 2H). ^{13}C NMR (151 MHz, CDCl_3) $\delta = 164.7, 153.4, 141.8, 139.6, 130.9, 118.1, 105.1, 60.9, 56.2, 46.6, 46.1, 26.1, 24.3$ ppm.

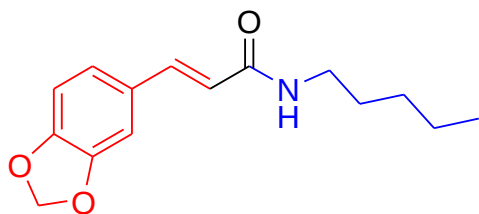
(E)-3-(benzo[d][1,3]dioxol-5-yl)-N-isobutylacrylamide (8g)⁷: According to GP I, piperonyl aldehyde (0.10 g, 0.66 mmol), isobutylamine (50 mg, 0.66 mmol) and Meldrum's acid (96 mg,



0.66 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:2) to get analytically pure product **8g** as white solid (0.12 g, 73%). ^1H NMR (400 MHz, CDCl_3) $\delta = 7.54$ (d, $J = 15.5$

Hz, 1H), 7.00 – 6.97 (m, 2H), 6.79 (d, $J = 7.8$ Hz, 1H), 6.24 (d, $J = 15.5$ Hz, 1H), 5.99 (s, 2H), 5.73 (br. s, 1H), 3.23 – 3.20 (m, 2H), 1.87 – 1.81 (m, 1H), 0.95 (d, $J = 6.8$ Hz, 6H). ppm. ^{13}C NMR (101 MHz, CDCl_3) $\delta = 166.2, 149.0, 148.2, 140.9, 129.2, 123.9, 118.6, 108.5, 106.3, 101.4, 47.2, 28.6, 20.2$ ppm.

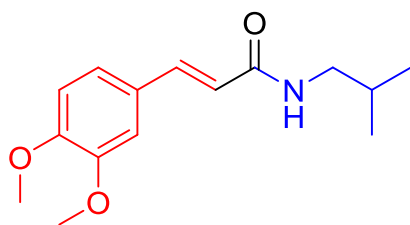
(E)-3-(benzo[d][1,3]dioxol-5-yl)-N-pentylacrylamide (8h)¹¹: According to GP I, piperonyl aldehyde (0.10 g, 0.66 mmol), amylamine (58 mg, 0.66 mmol) and Meldrum's acid (96 mg, 0.66



mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **8h** as white solid (0.11 g, 64%). ^1H NMR (400 MHz, CDCl_3) $\delta = 7.53$ (d, $J = 15.6$ Hz, 1H), 7.01 – 6.95 (m, 2H), 6.79 (d, $J = 7.6$

Hz, 1H), 6.20 (d, $J = 15.6$ Hz, 1H), 5.98 (s, 2H), 5.59 (br. s, 1H), 3.39 – 3.34 (m, 2H), 1.60 – 1.53 (m, 2H), 1.35 – 1.33 (m, 4H), 0.90 (t, $J = 6.8$ Hz, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3) $\delta = 166.0, 149.0, 148.2, 140.5, 129.3, 123.8, 118.9, 108.5, 106.3, 101.4, 39.8, 29.4, 29.1, 22.4, 14.0$ ppm.

(E)-N-isobutyl-3-(3,4-dimethoxyphenyl)acrylamide (8i)⁷: According to GP I, 3,4-dimethoxybenzaldehyde (0.10 g, 0.60 mmol), isobutylamine (44 mg, 0.60 mmol) and Meldrum's



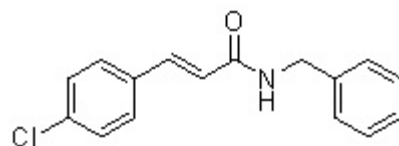
acid (87 mg, 0.60 mmol) was reacted for 8 h and the crude mixture was purified by column chromatography (Silica gel; EtOAc: Hexane, 1:4) to get analytically pure product **8i** as white solid (0.10 g, 65%). ¹H NMR (600 MHz, CDCl₃) δ = 7.58 (d, *J* = 15.6 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 1H), 7.03 (s, 1H), 6.86 (d, *J*

= 8.4 Hz, 1H), 6.27 (d, *J* = 15.6 Hz, 1H), 5.59 (br. s, 1H), 3.912 (s, 3H), 3.907 (s, 3H), 3.23 – 3.21 (m, 2H), 1.86 – 1.82 (m, 1H), 0.96 (d, *J* = 7.2 Hz, 6H). ppm. ¹³C NMR (101 MHz, CDCl₃) δ = 166.2, 150.5, 149.1, 140.9, 127.8, 121.9, 118.6, 111.1, 109.6, 56.0, 55.9, 47.1, 28.7, 20.2 ppm.

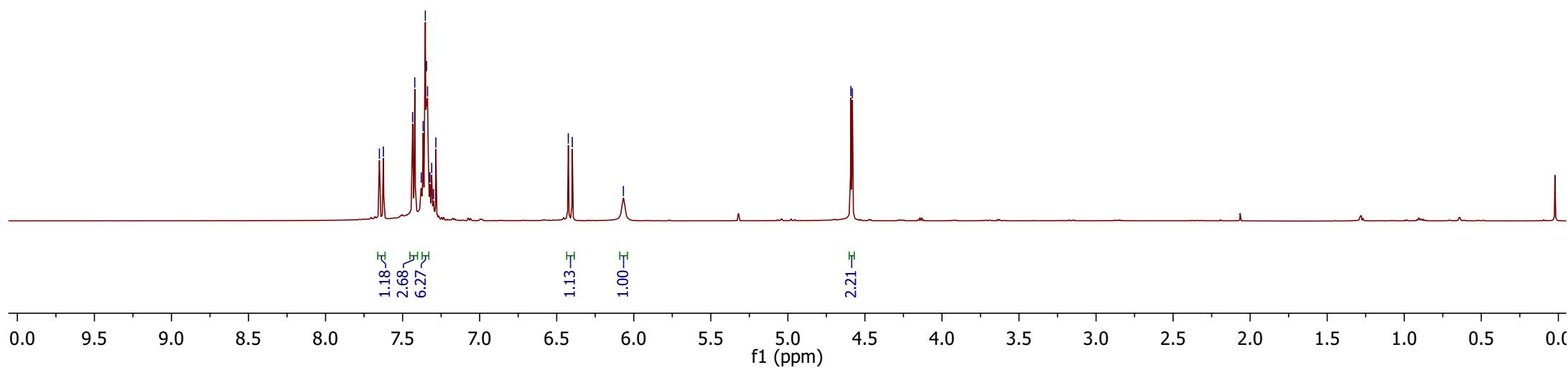
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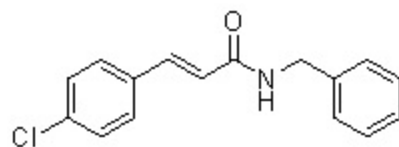
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6a





6a

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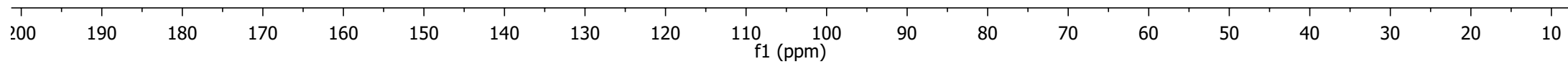
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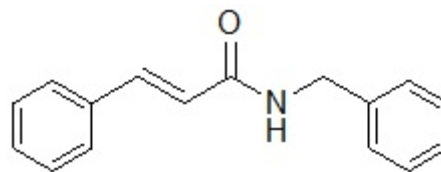
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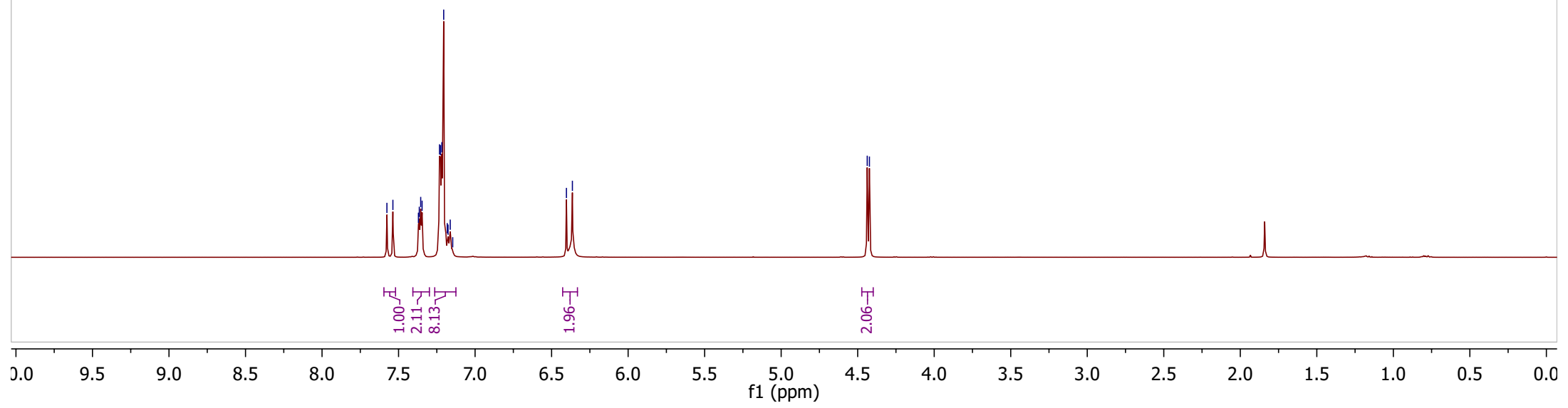
— 43.922

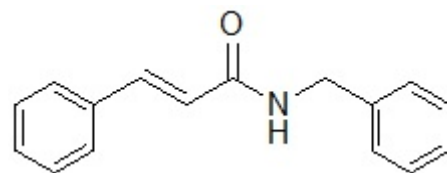


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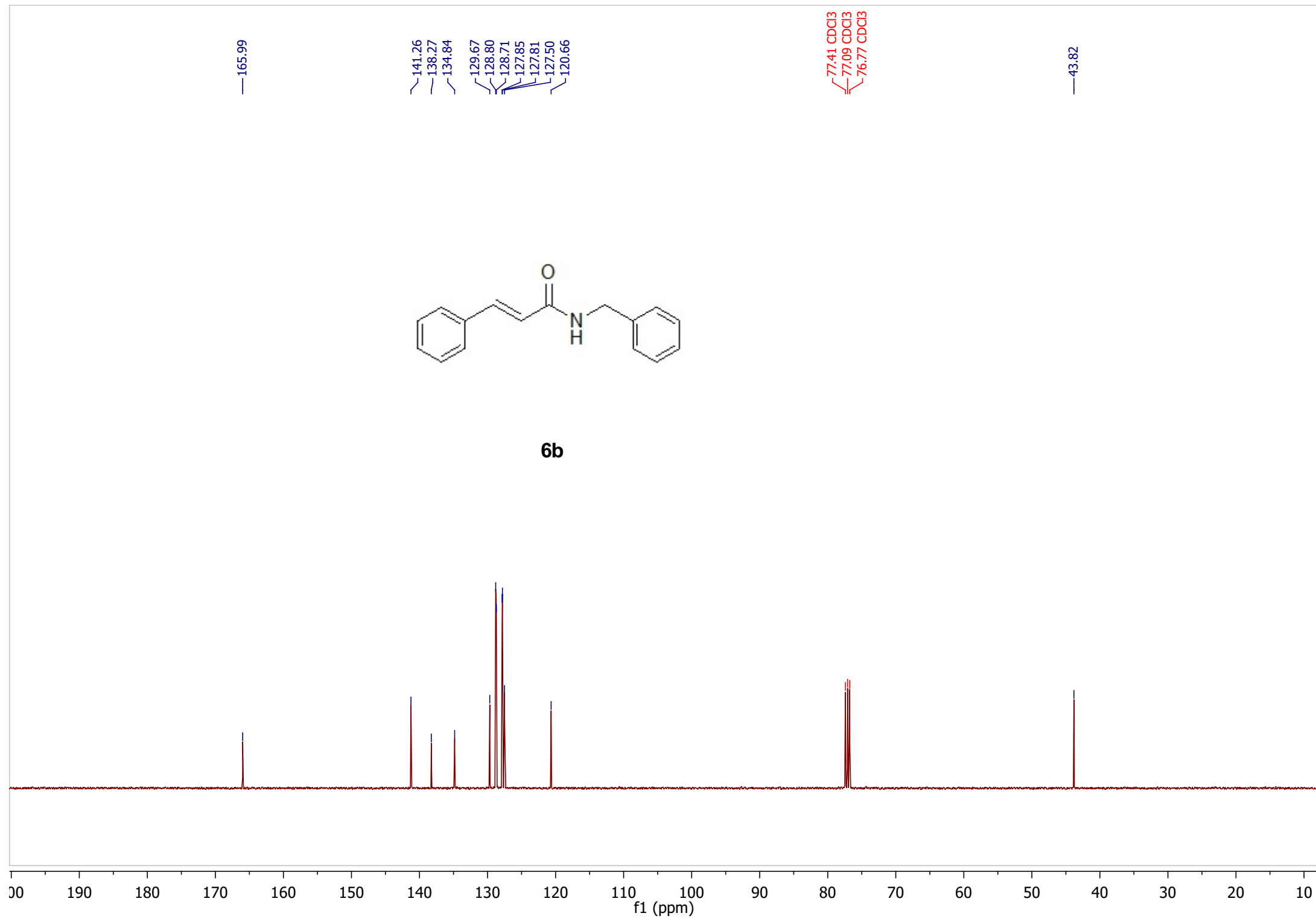


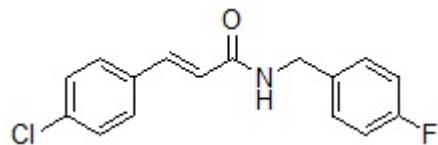
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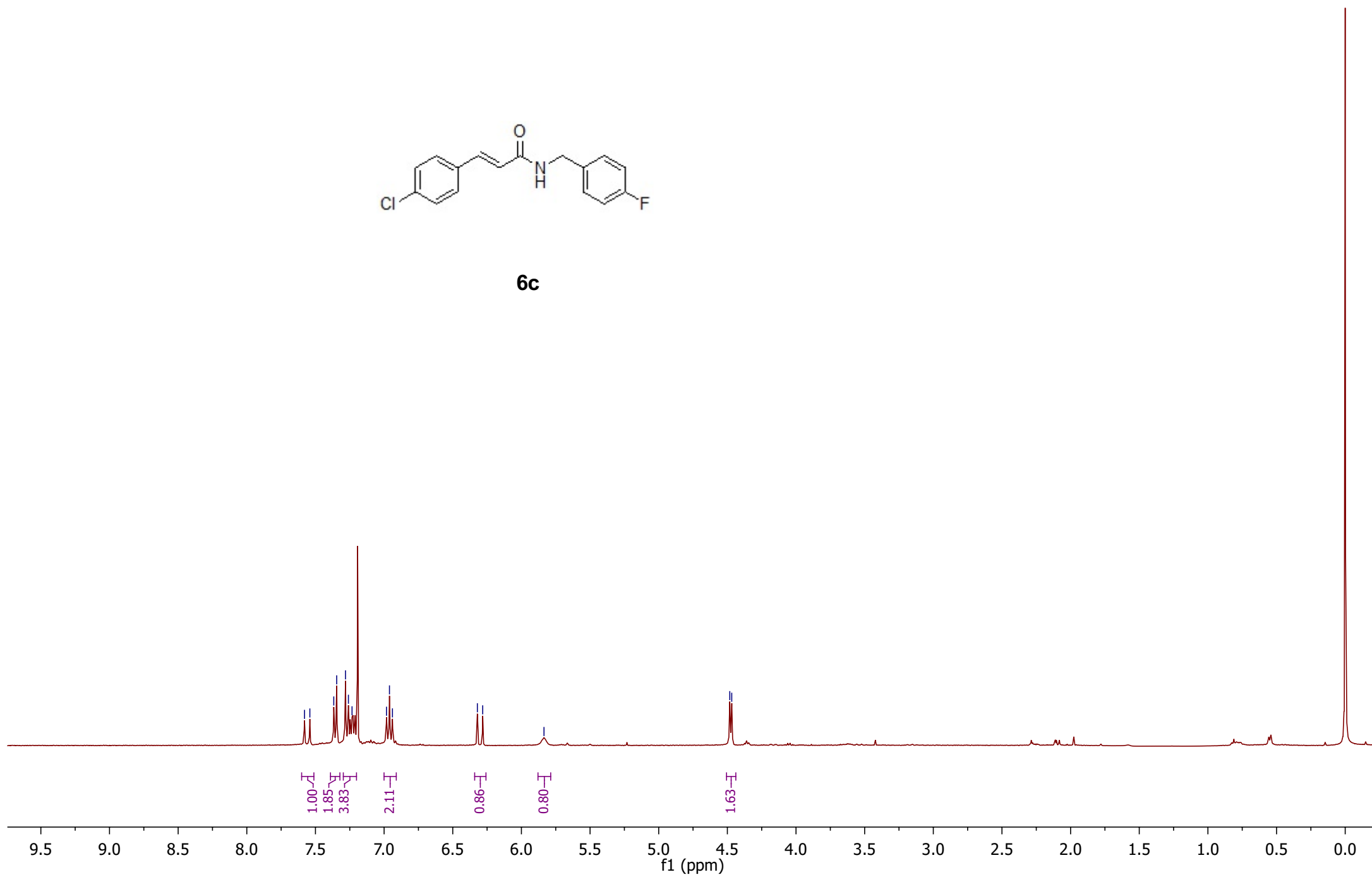
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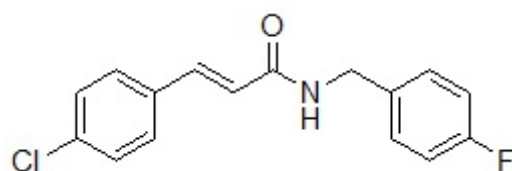




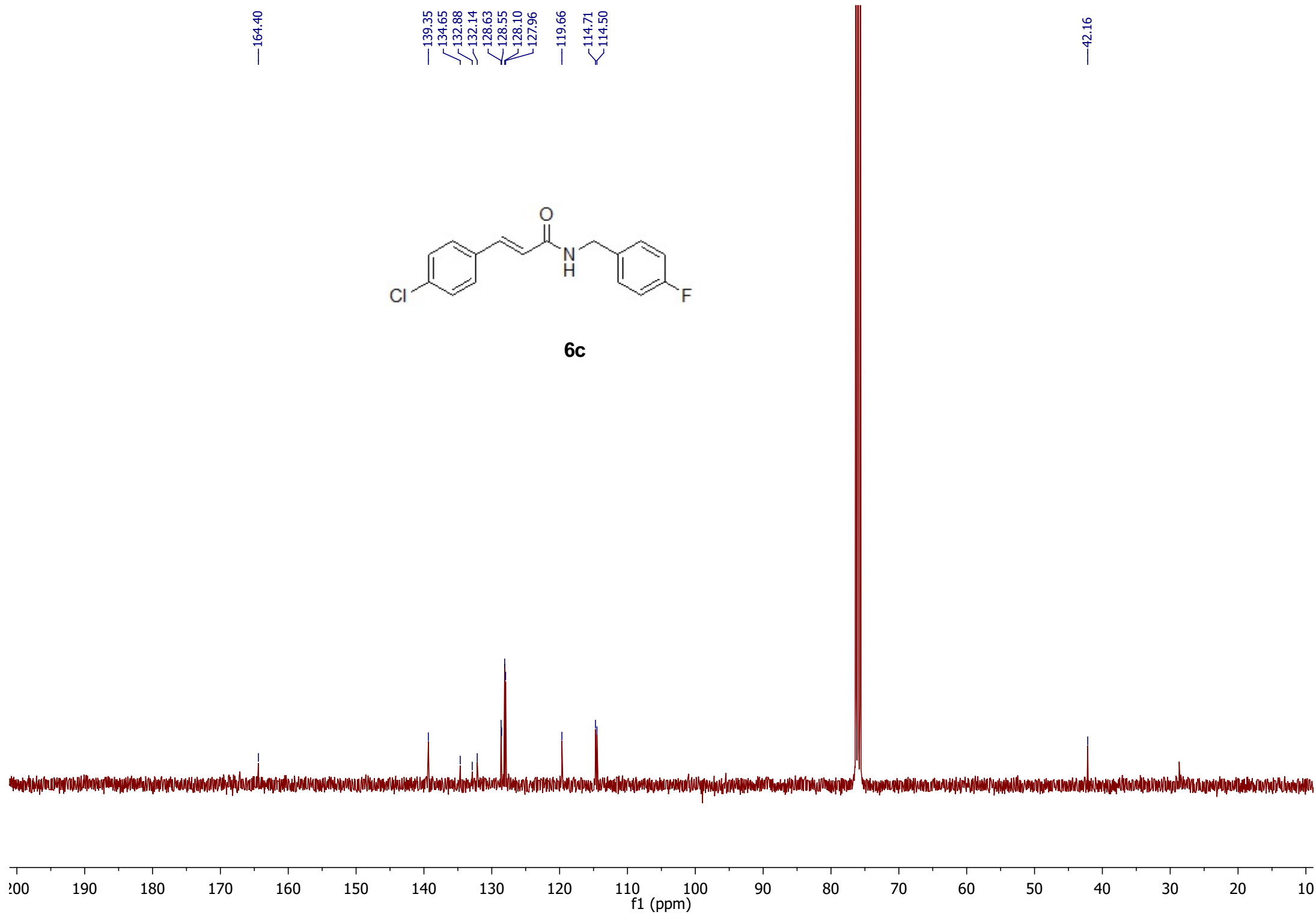
6c

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6.282
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4.468





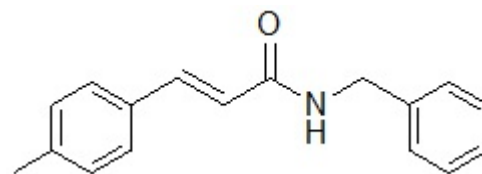
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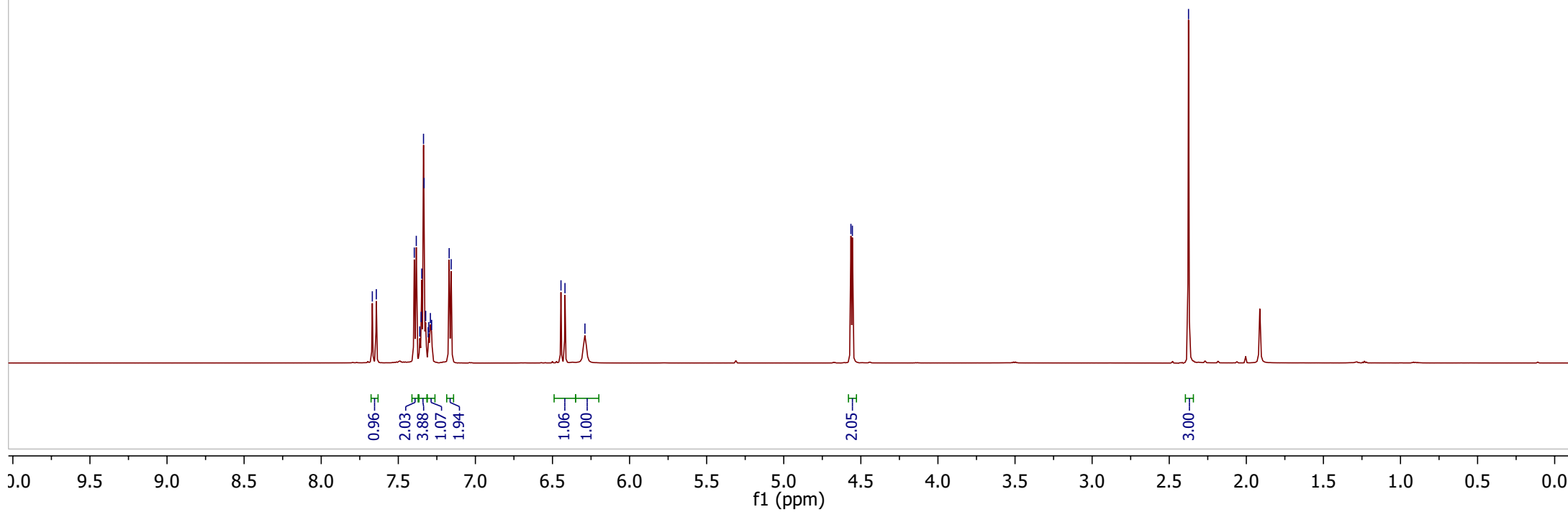
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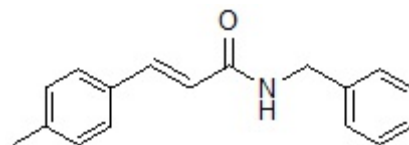
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4.555

2.375



6d





6d

—166.11

—141.29

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—138.31

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—129.53

—128.71

—127.88

—127.79

—127.51

—119.50

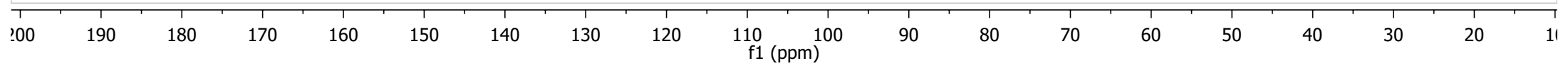
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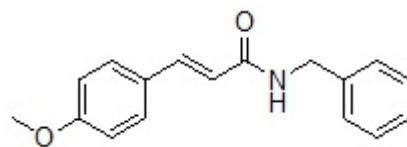
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76.85 CDCl₃

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—21.40





6e

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4.449
3.725

0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

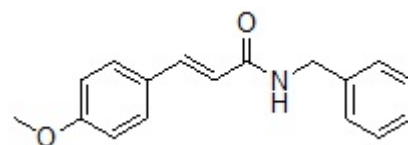
1.00
1.99
4.85

1.92

0.99

1.98

2.95



6e

— 166.22
— 160.90

— 140.94
— 138.38

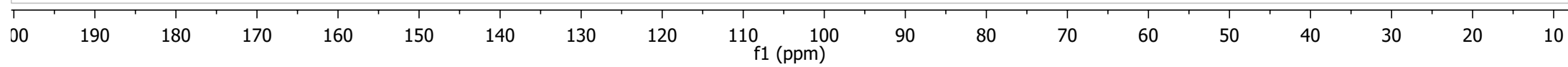
129.36
128.70
127.87
127.54
127.48

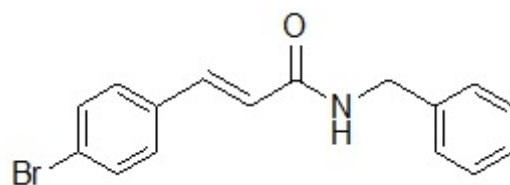
— 118.15
— 114.25

77.37 CDCl₃
77.05 CDCl₃
76.74 CDCl₃

— 55.34

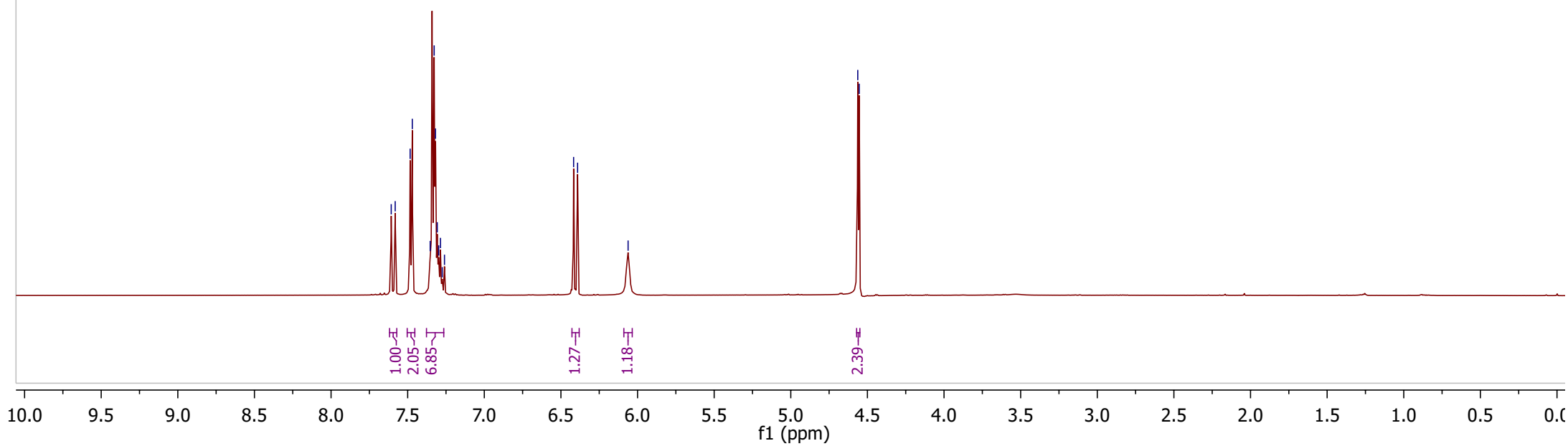
— 43.80

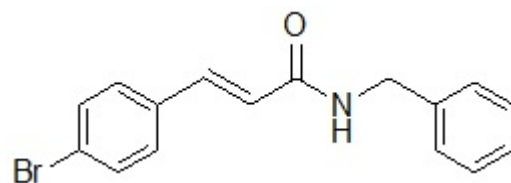




6f

7.607
7.581
7.484
7.470
7.353
7.327
7.319
7.307
7.298
7.286
7.275
7.260
6.417
6.391
6.062
4.563
4.553





6f

—165.44

—140.16

—138.05

—133.71

—132.05

—129.20

—128.78

—127.92

—127.65

—123.87

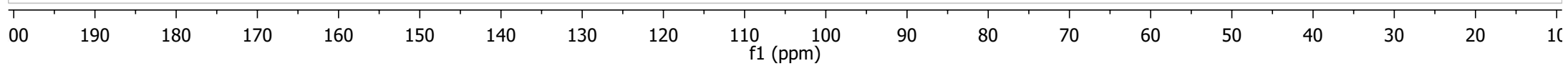
—121.08

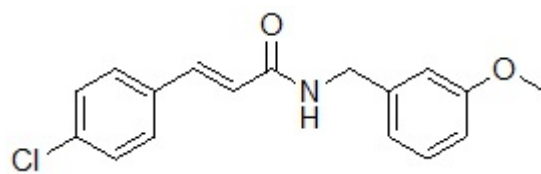
77.24 CDCl₃

77.03 CDCl₃

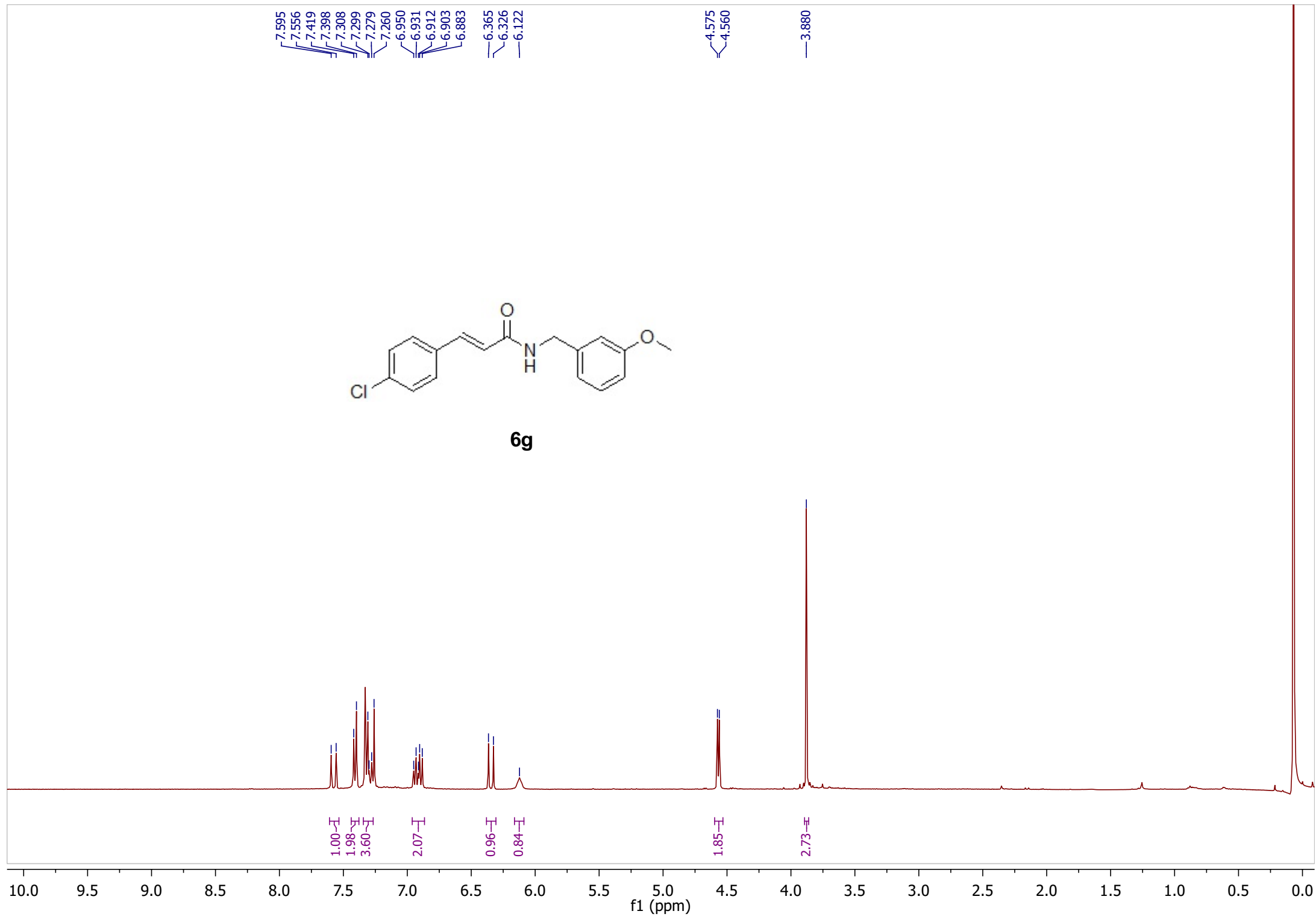
76.82 CDCl₃

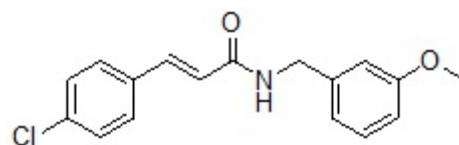
—43.93





6g





6g

—165.26

—157.56

—139.62

—135.37

—133.42

—130.00

—129.02

—128.93

—126.05

—121.40

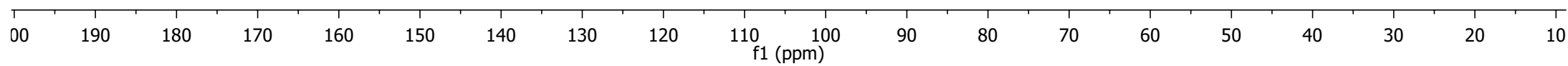
—120.79

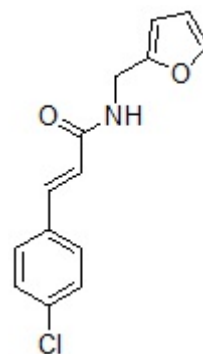
—110.34

77.36 CDCl₃
77.05 CDCl₃
76.73 CDCl₃

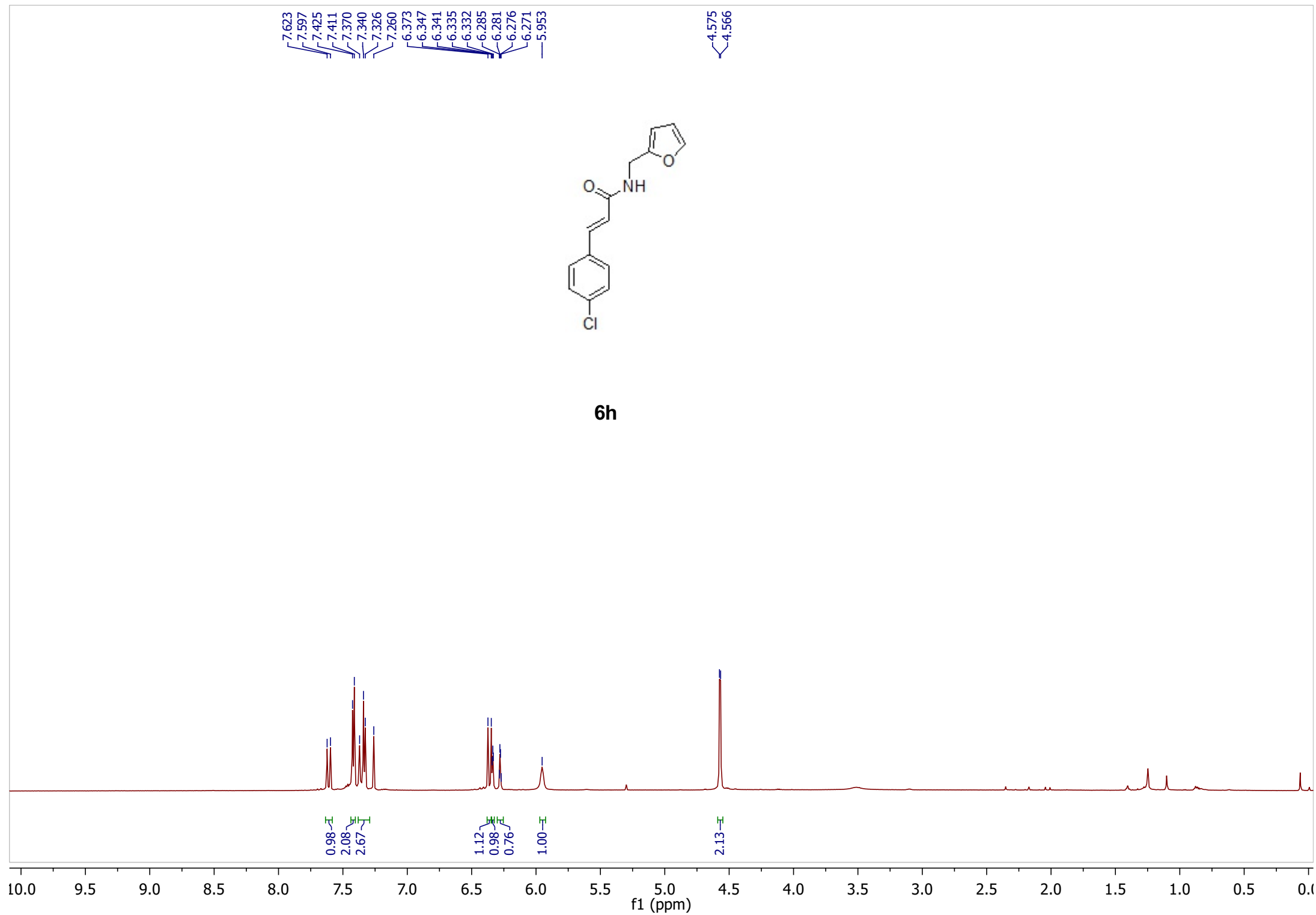
—55.39

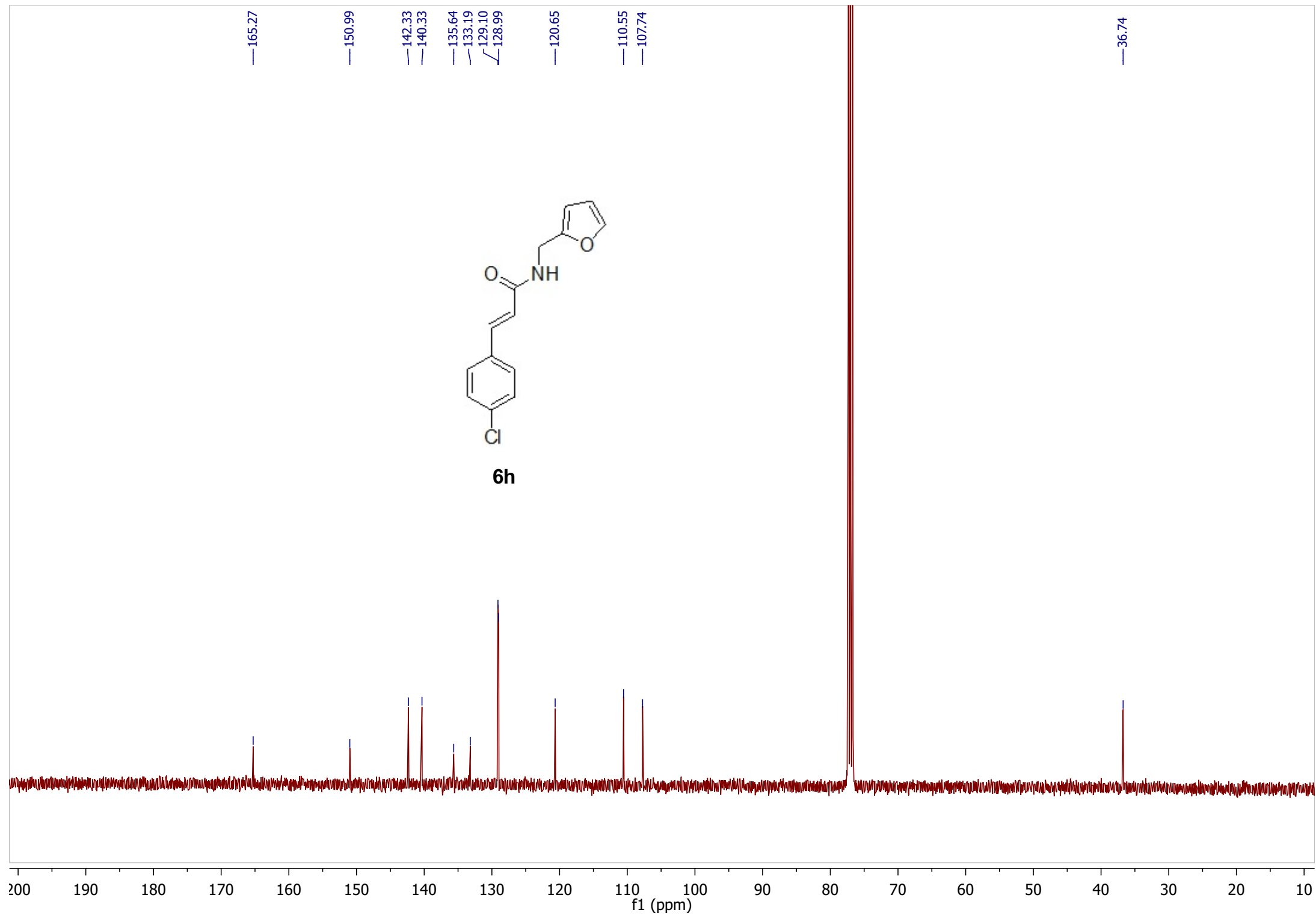
—39.67





6h



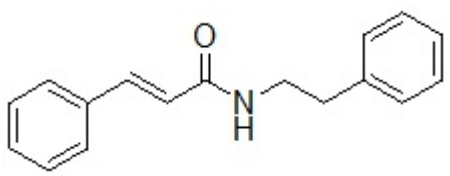


7.562
7.523
7.415
7.405
7.396
7.391
7.303
7.290
7.280
7.268
7.263
7.252
7.233
7.187
7.185
7.170
7.162
7.142
6.272
6.233

5.640

3.613
3.596
3.580
3.563

2.837
2.819
2.802



6i

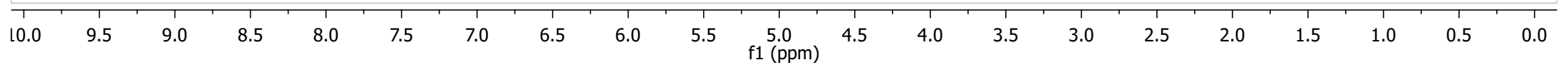
1.00
2.17
5.14
2.85

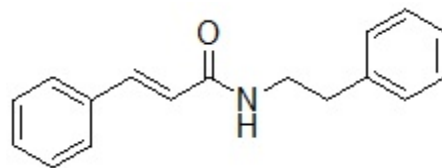
1.26

1.14

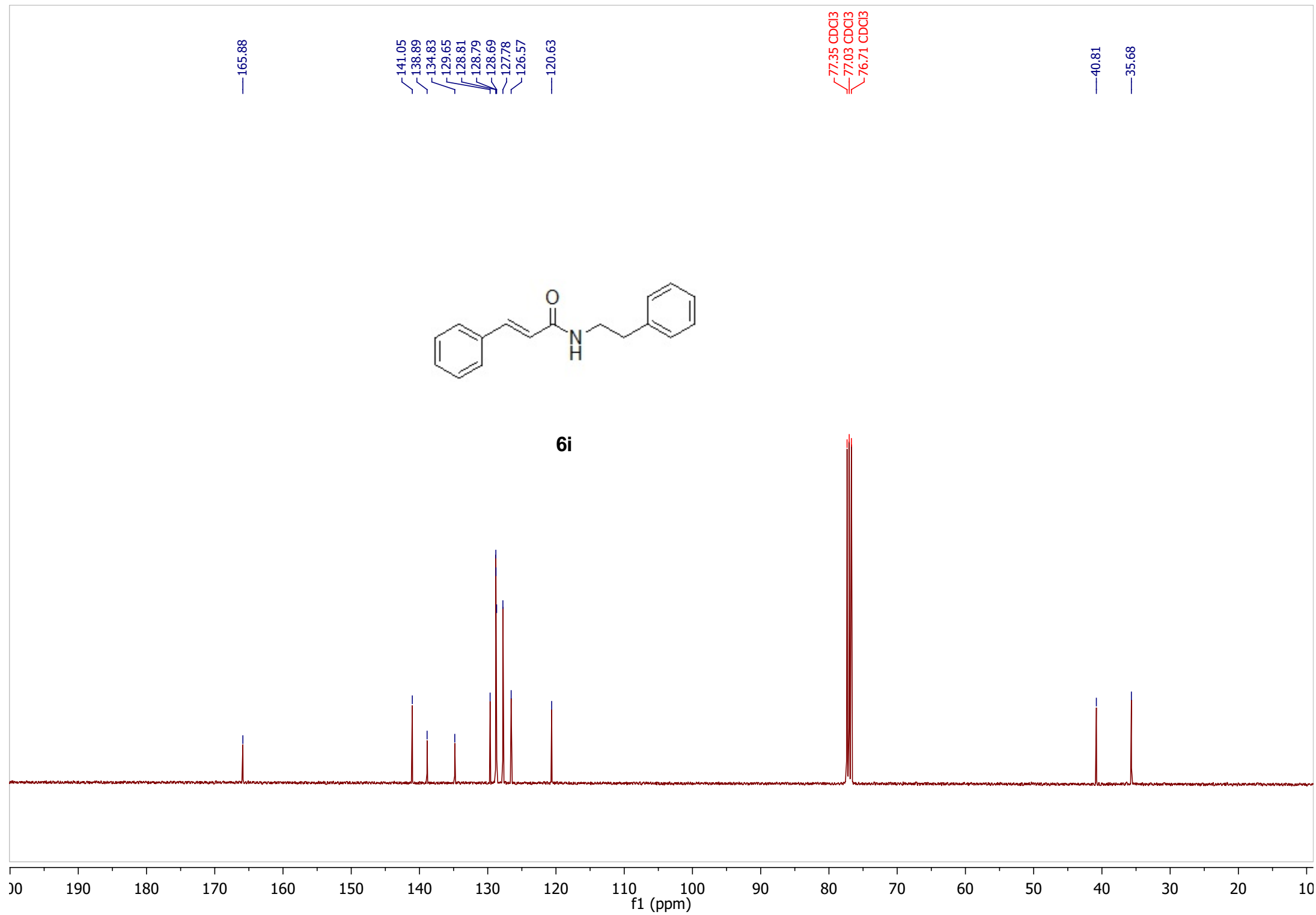
2.44

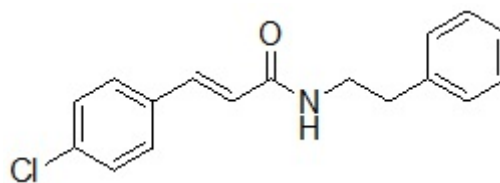
2.22



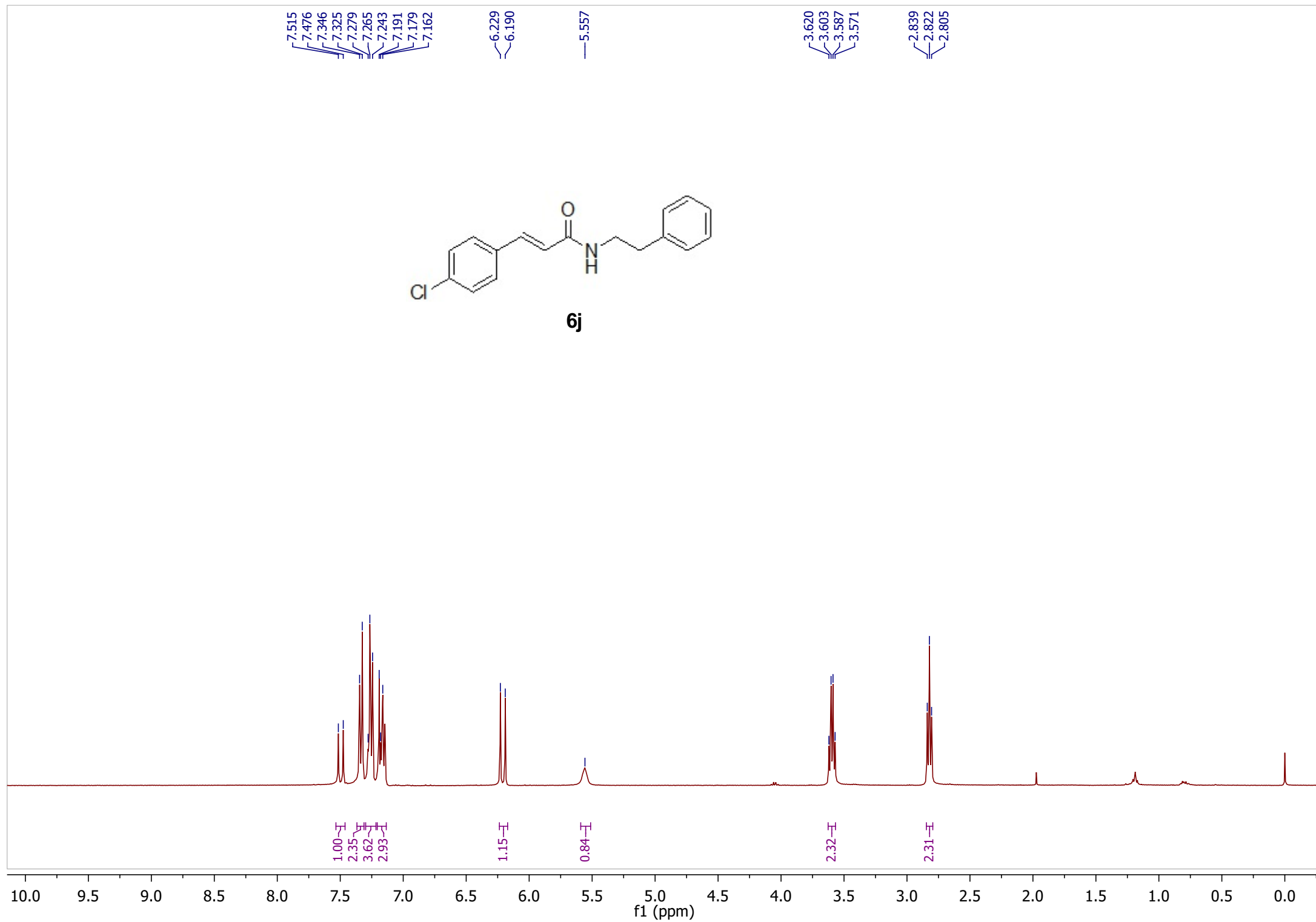


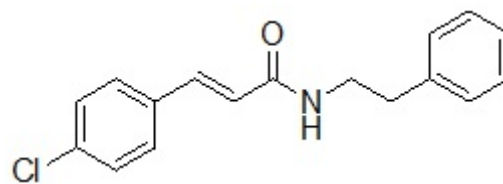
6i





6j





6j

— 165.50

— 139.75

— 138.80

— 135.50

— 133.32

— 129.05

— 128.94

— 128.79

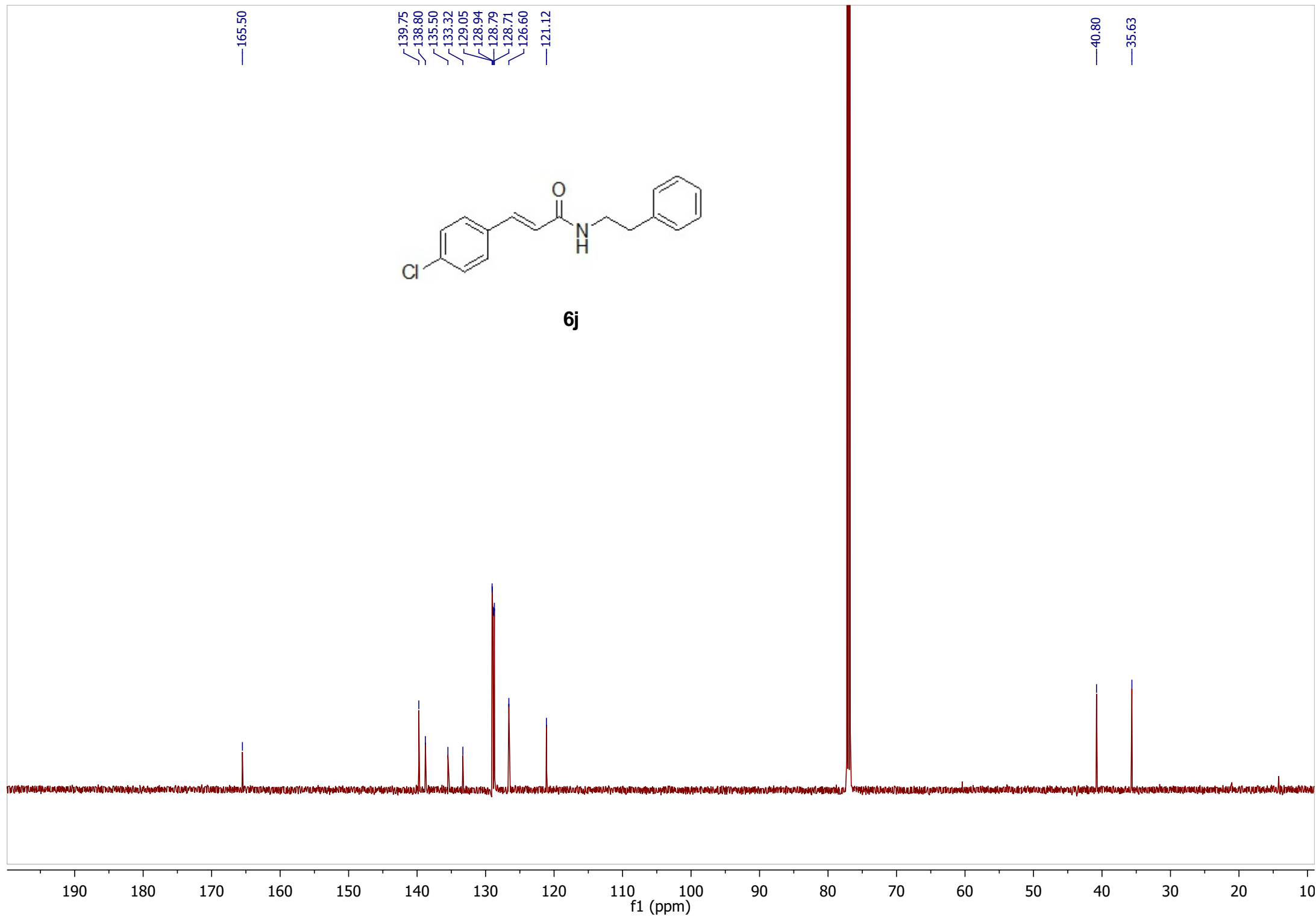
— 128.71

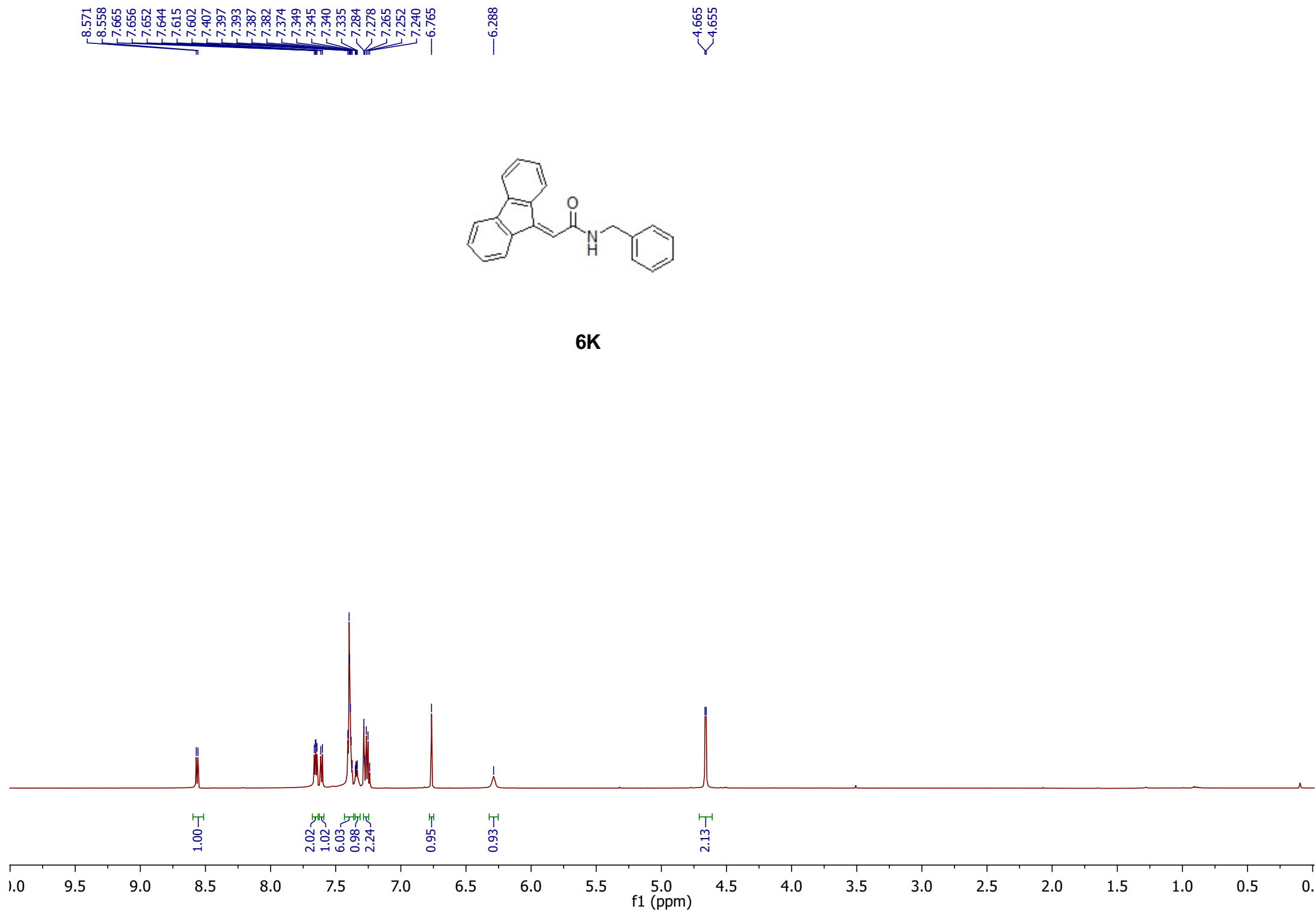
— 126.60

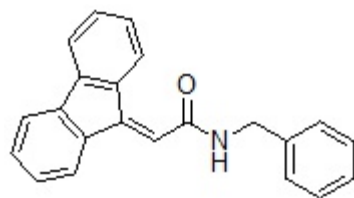
— 121.12

— 40.80

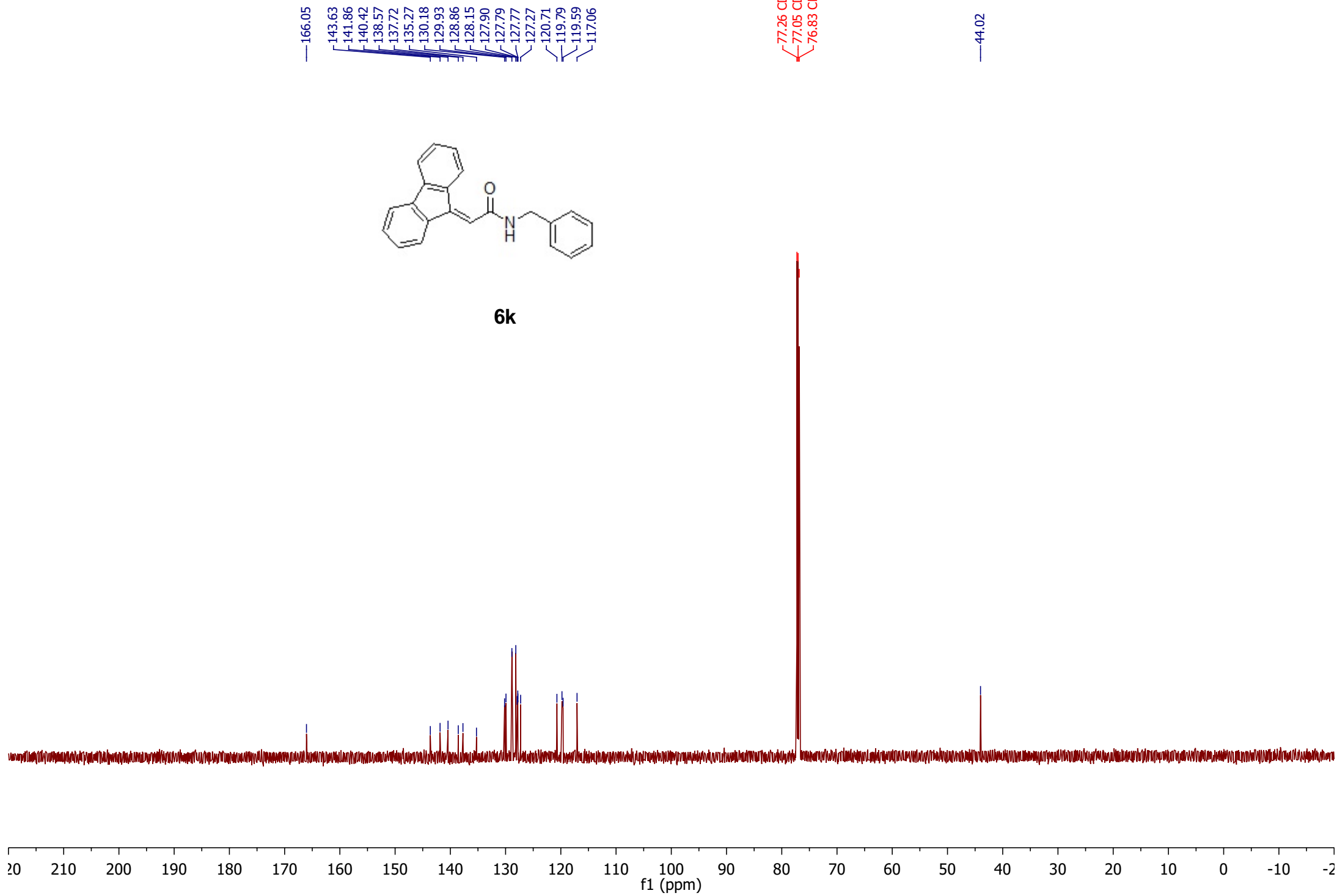
— 35.63

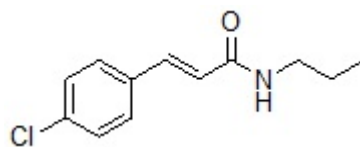




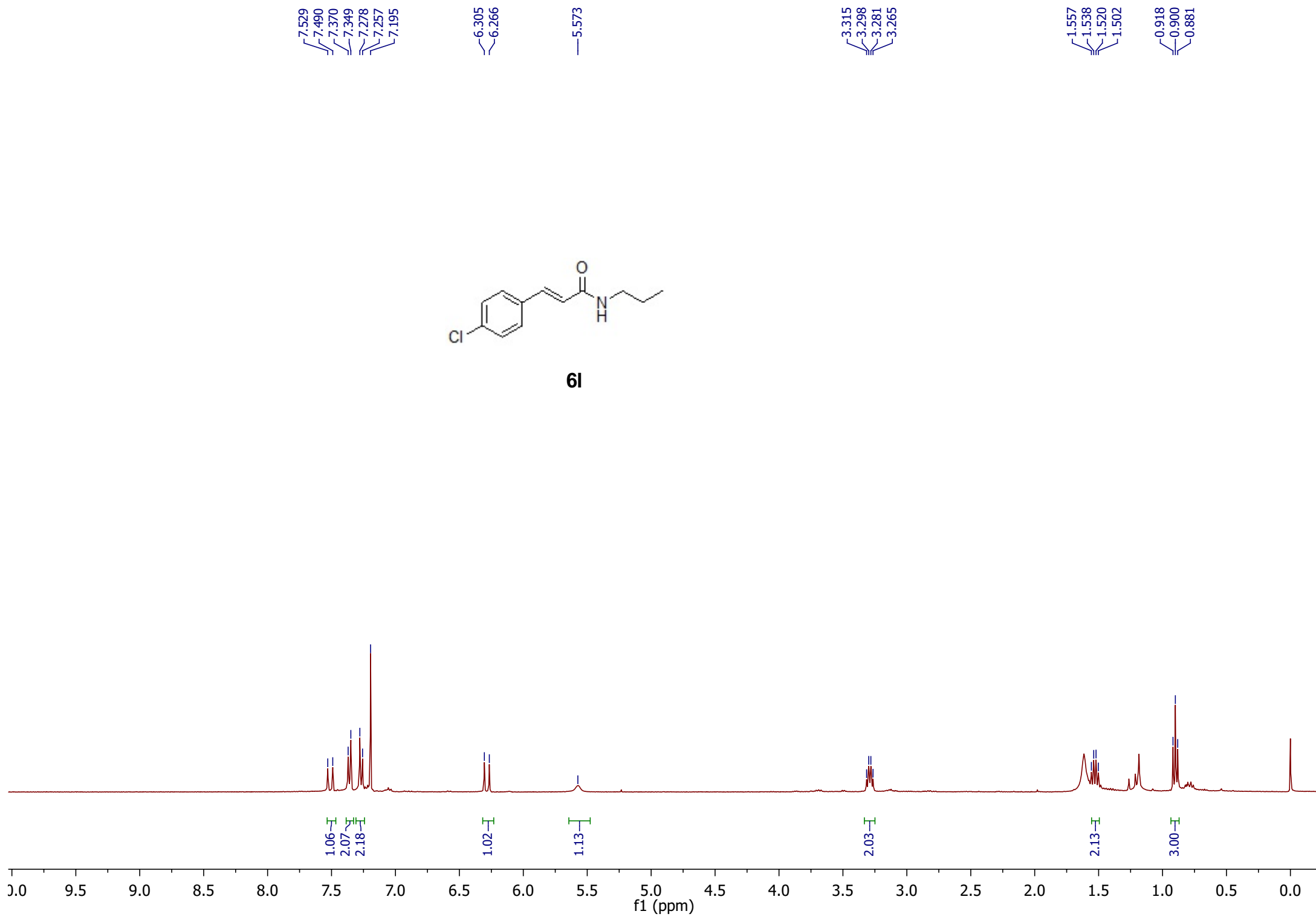


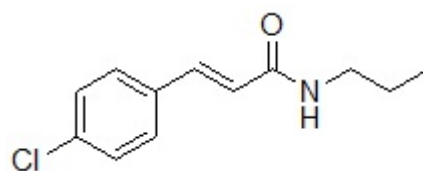
6k



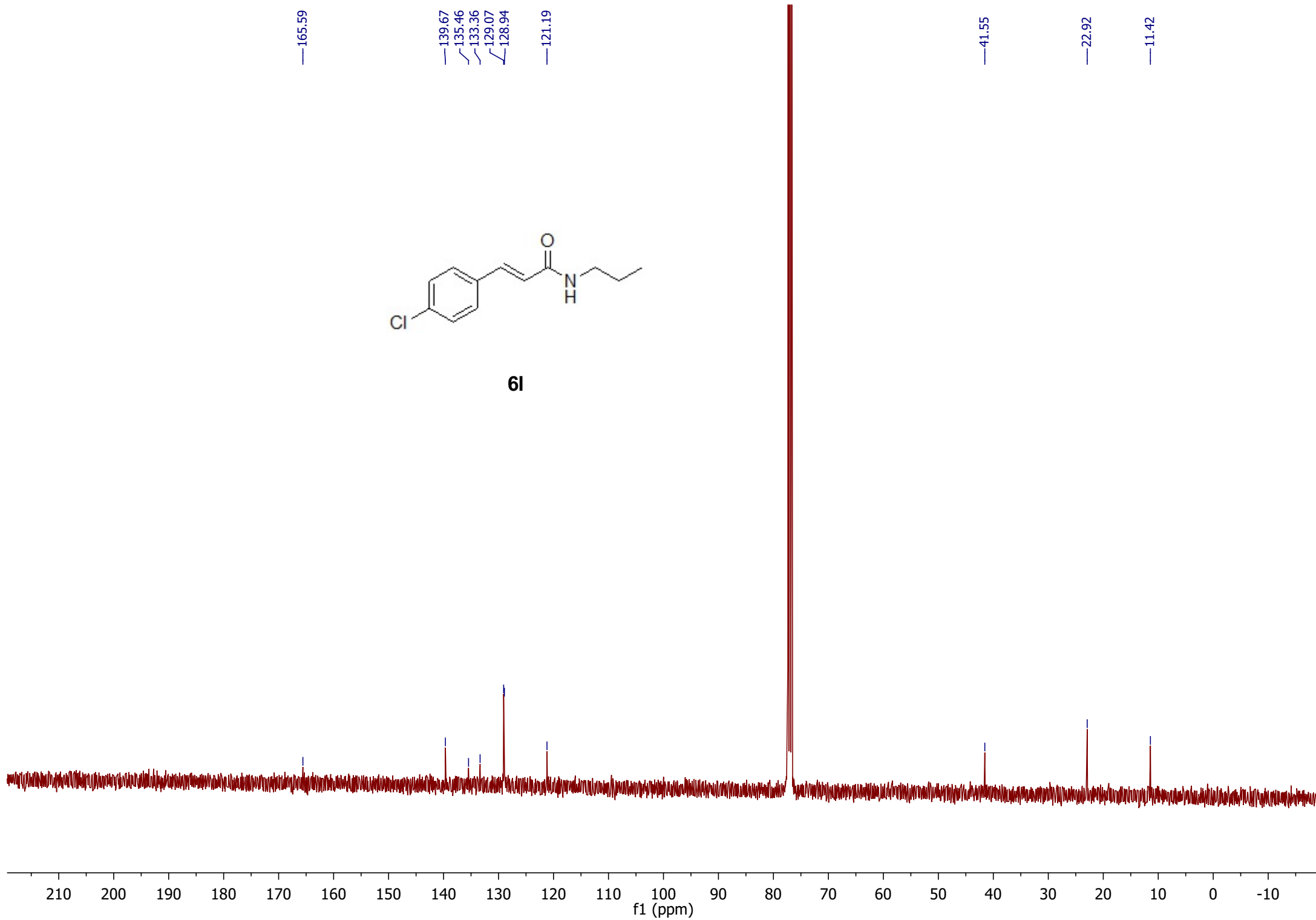


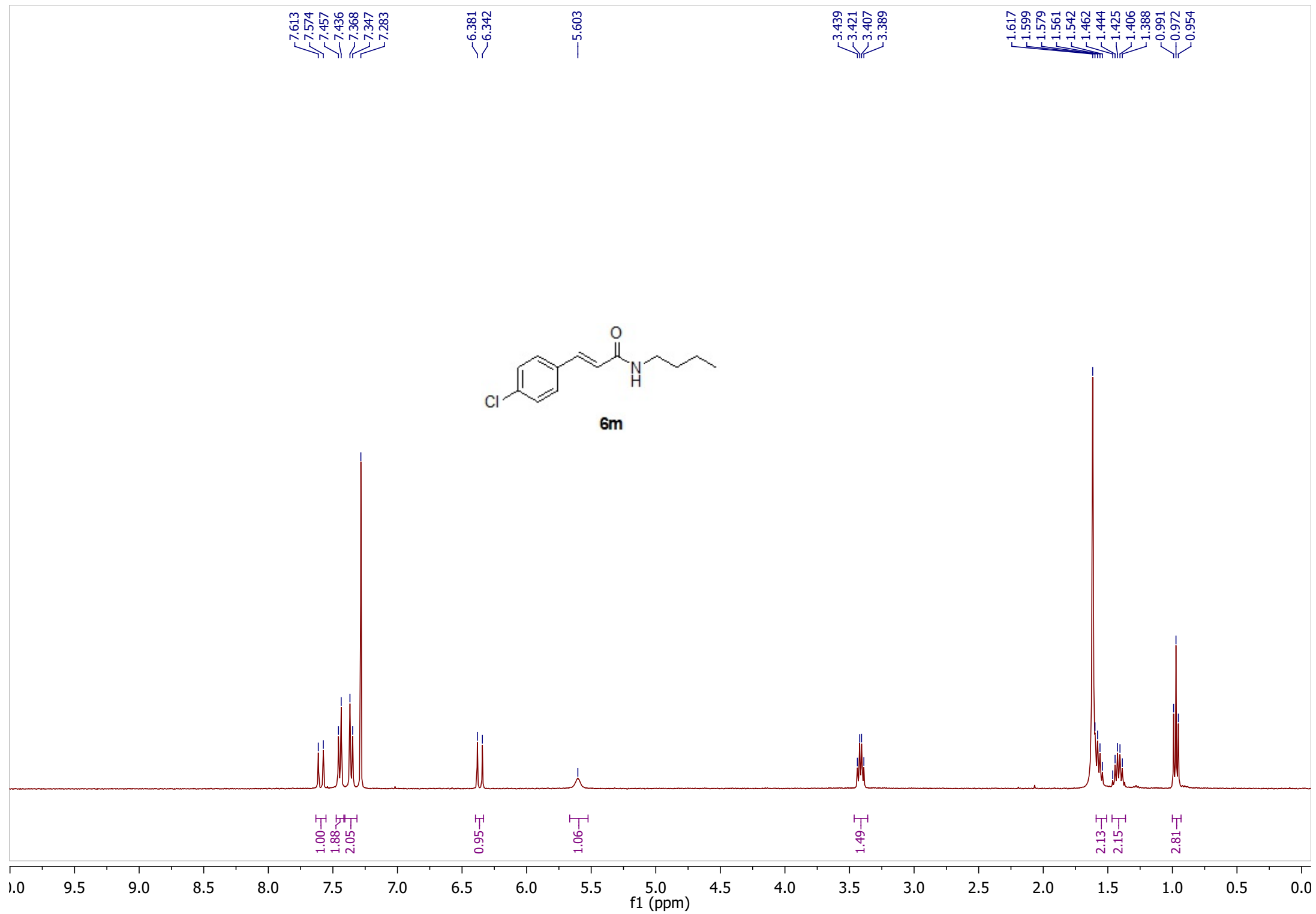
6l

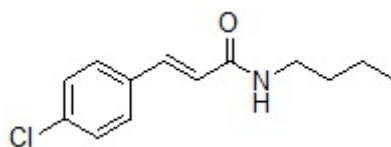




6l







6m

— 165.52

— 139.53

— 135.42

— 133.41

— 129.05

— 128.90

— 121.32

77.32 CDCl₃

77.00 CDCl₃

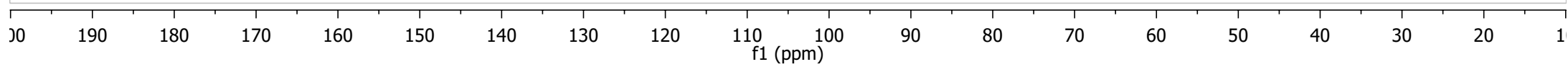
76.69 CDCl₃

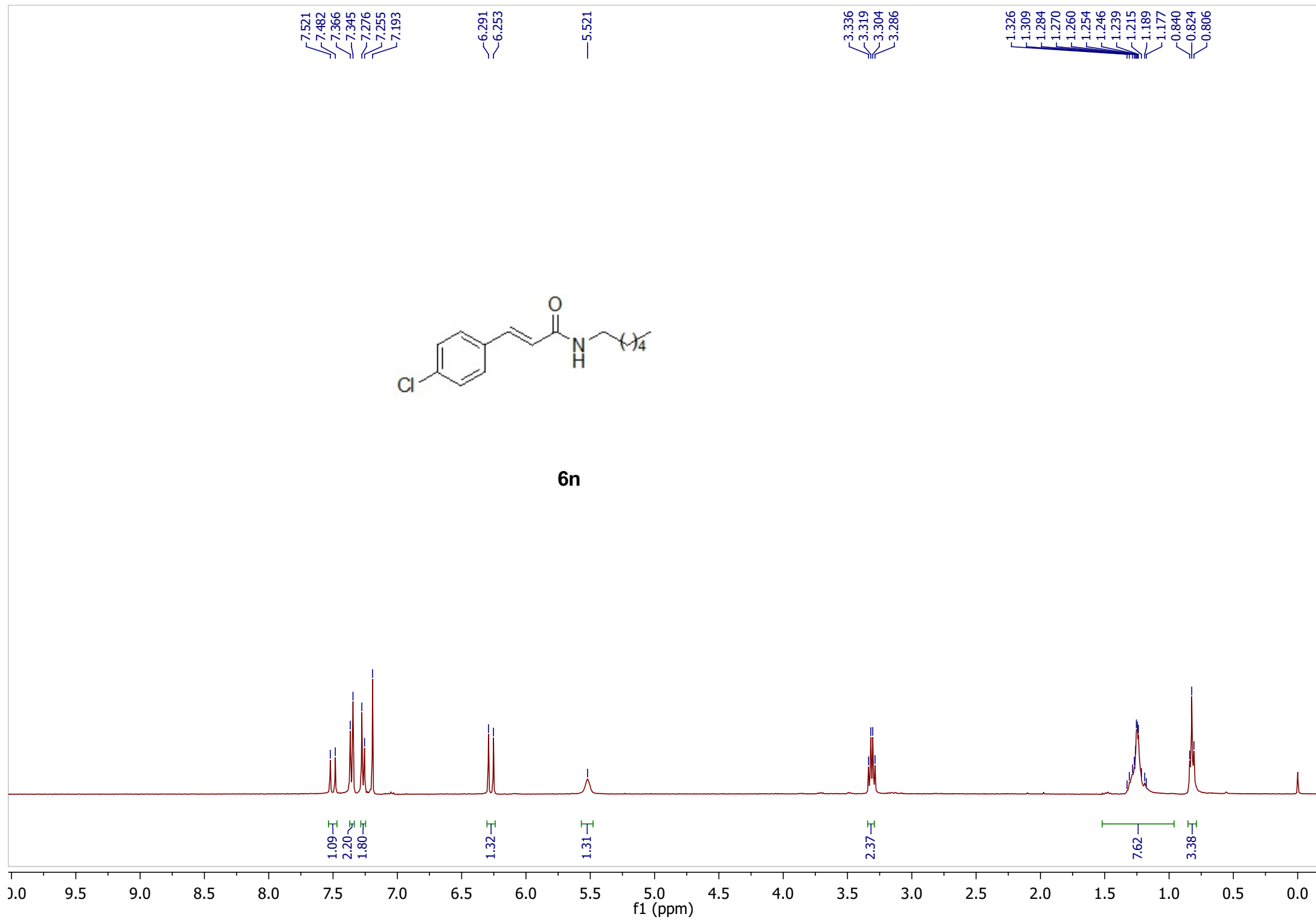
— 39.54

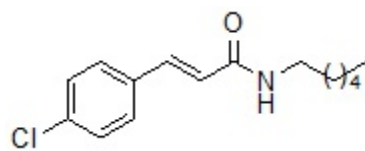
— 31.73

— 20.10

— 13.73







6n

165.48

139.53

135.42

133.42

129.05

128.90

121.33

77.21 CDCl₃

77.00 CDCl₃

76.79 CDCl₃

39.85

31.48

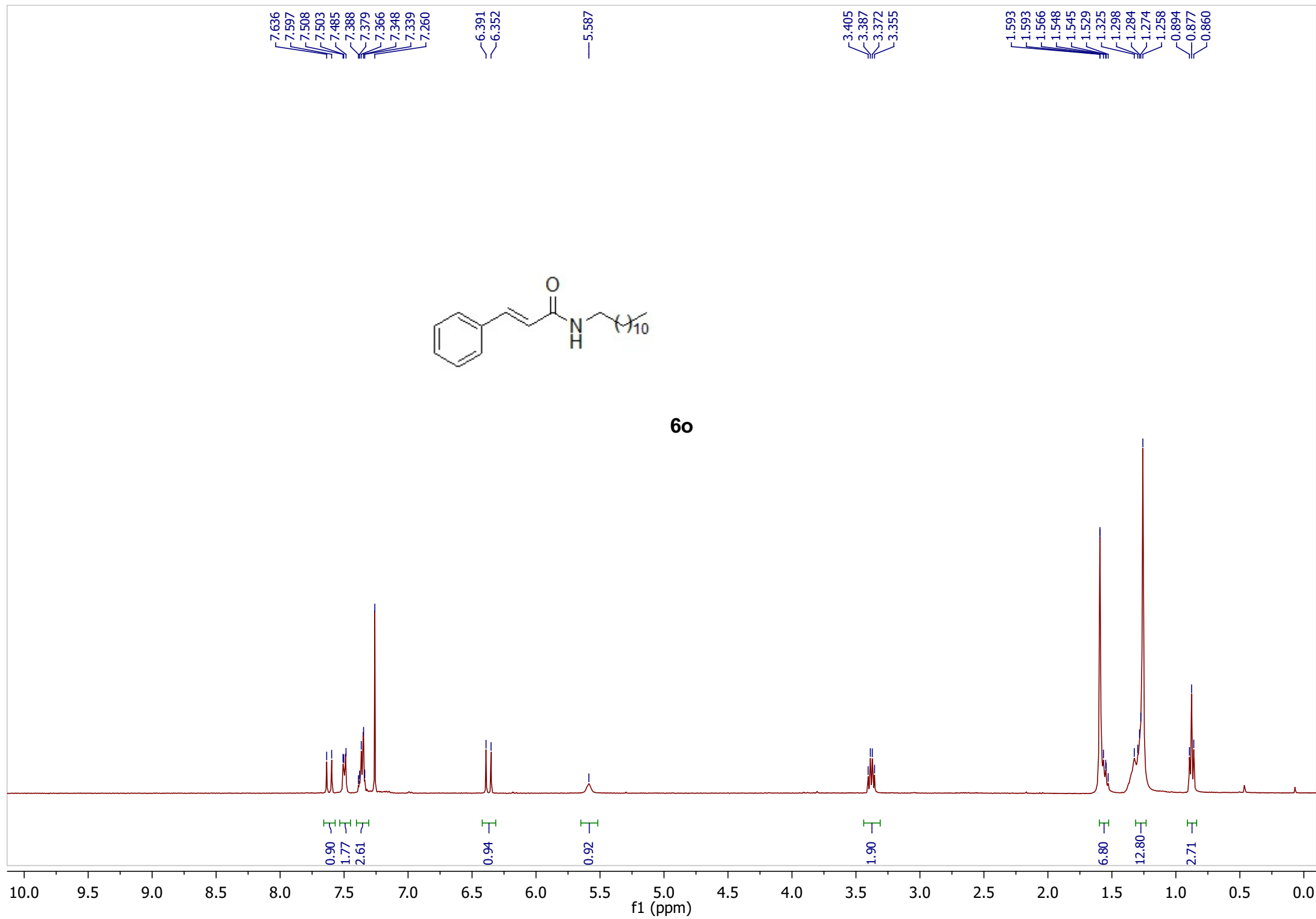
29.63

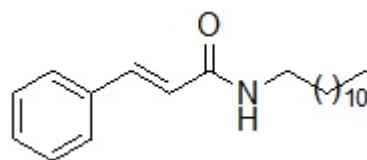
26.62

22.55

14.00

f1 (ppm)





6o

— 165.80

— 140.84

— 134.93

— 129.57

— 128.78

— 127.73

— 120.80

77.32 CDCl₃

77.00 CDCl₃

76.68 CDCl₃

— 39.83

— 31.91

— 29.69

— 29.64

— 29.62

— 29.58

— 29.54

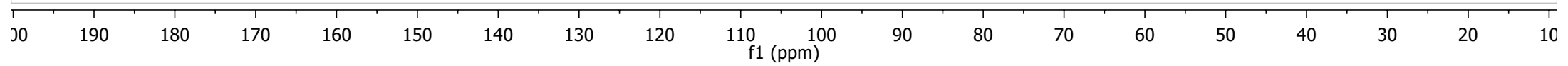
— 29.33

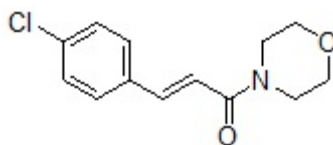
— 29.31

— 26.97

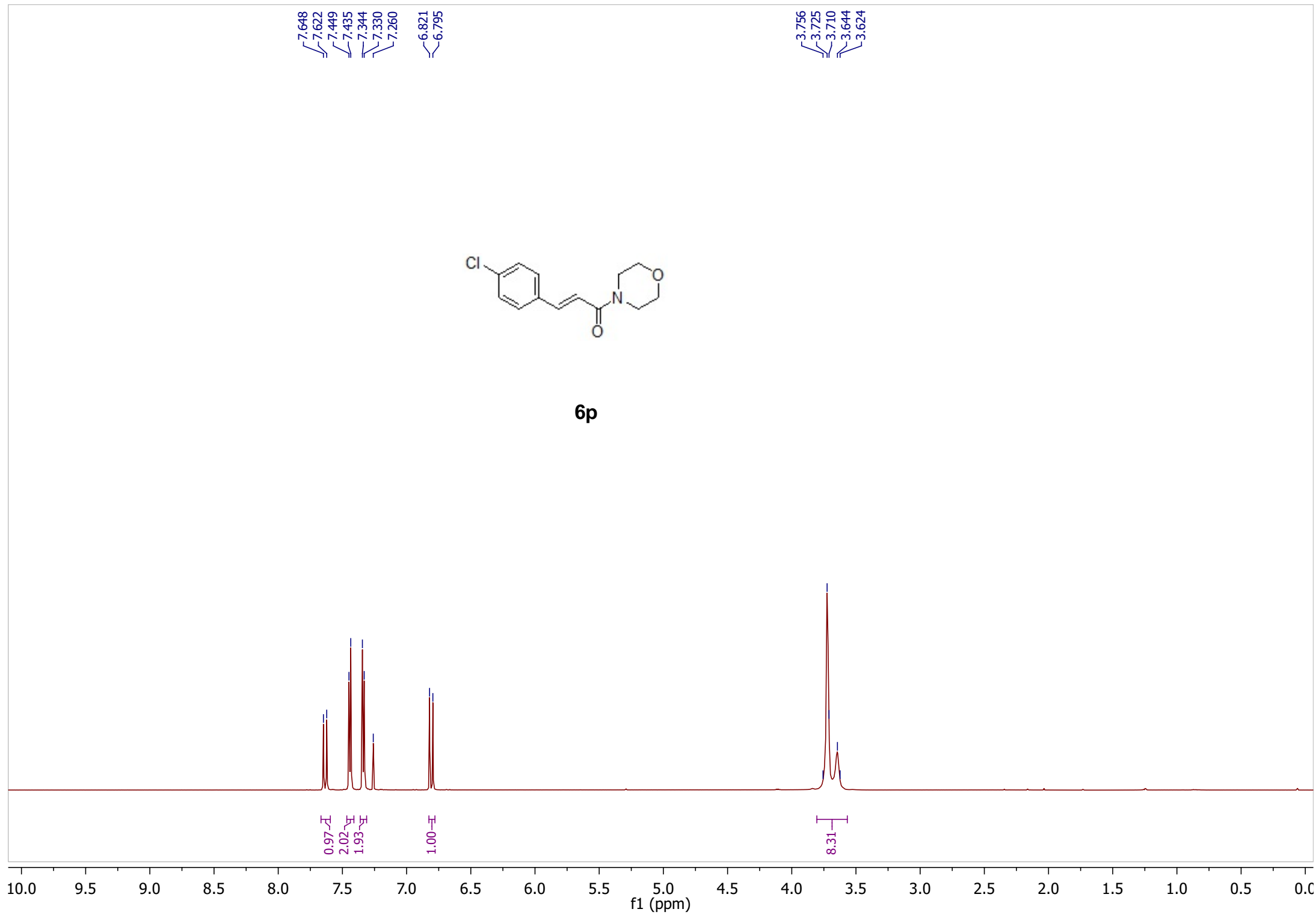
— 22.67

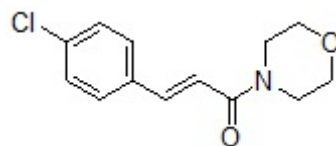
— 14.09





6p





6p

—165.28

—141.84

—135.58

—133.62

—129.08

—128.95

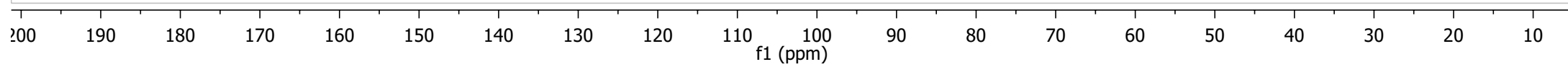
—117.11

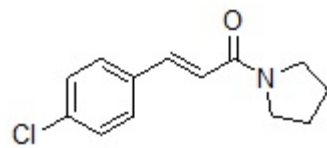
77.24 CDCl₃
77.03 CDCl₃
76.82 CDCl₃

—66.84

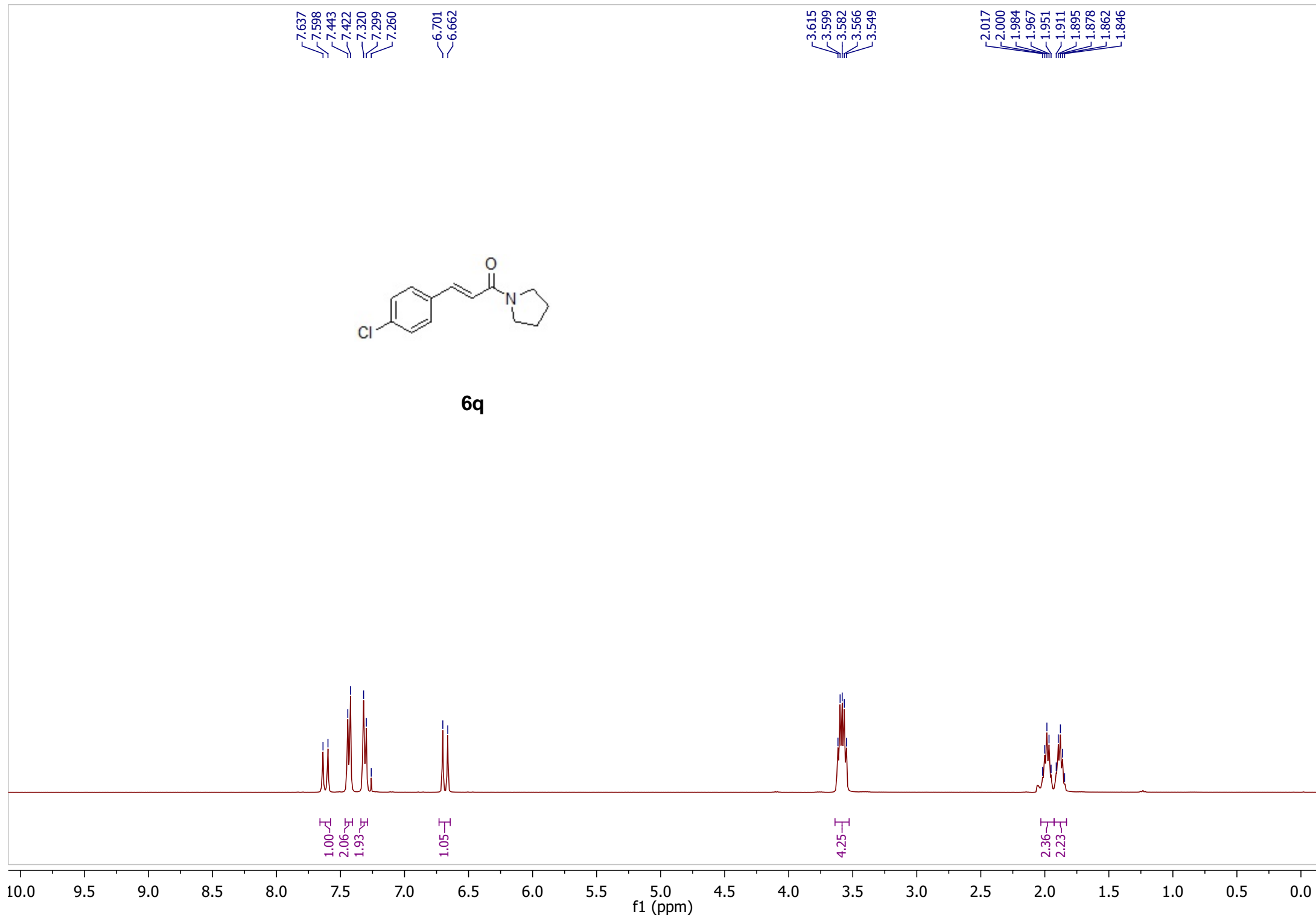
—46.26

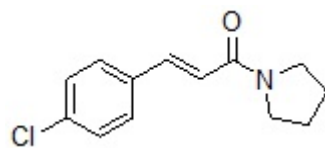
—42.53





6q





6q

— 164.38

— 140.26

— 135.28

— 133.86

— 128.99

— 119.44

77.38 CDCl₃
77.06 CDCl₃
76.74 CDCl₃

46.59
46.08

26.13
24.32

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

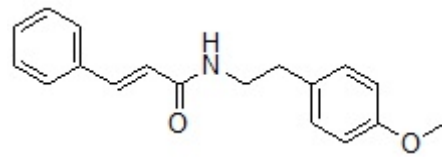
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7.597
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7.481
7.472
7.468
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7.352
7.344
7.339
7.260
7.150
7.129
6.874
6.852

6.345
6.306

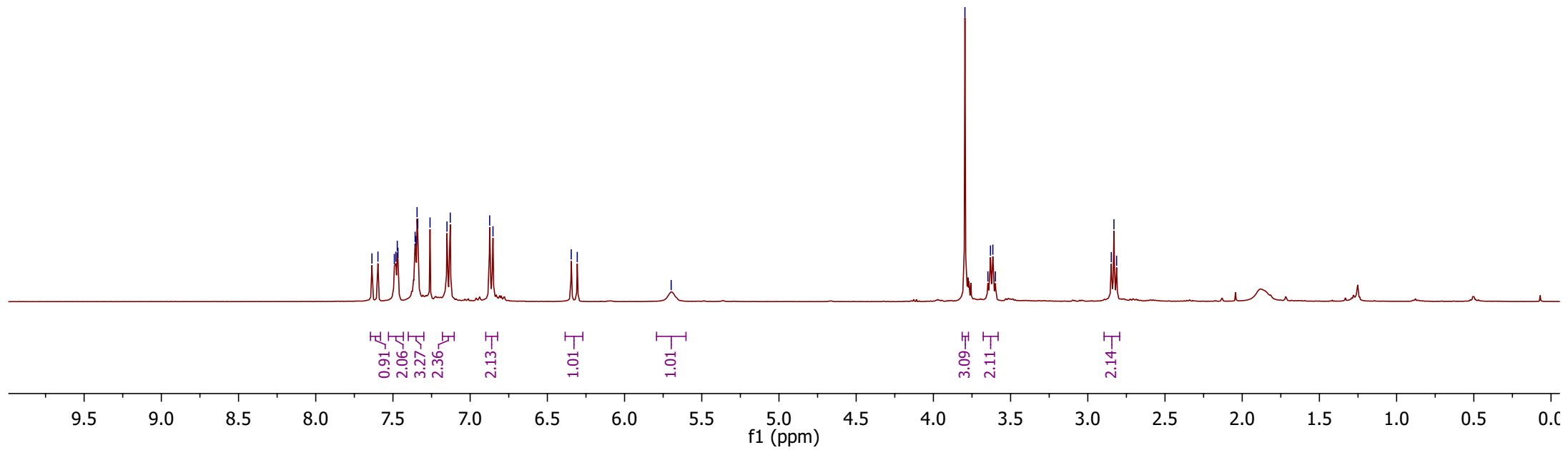
5.698

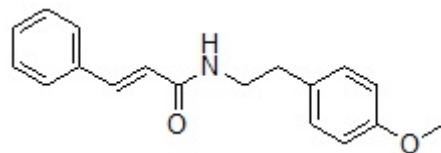
3.796
3.647
3.630
3.615
3.598

2.848
2.831
2.813

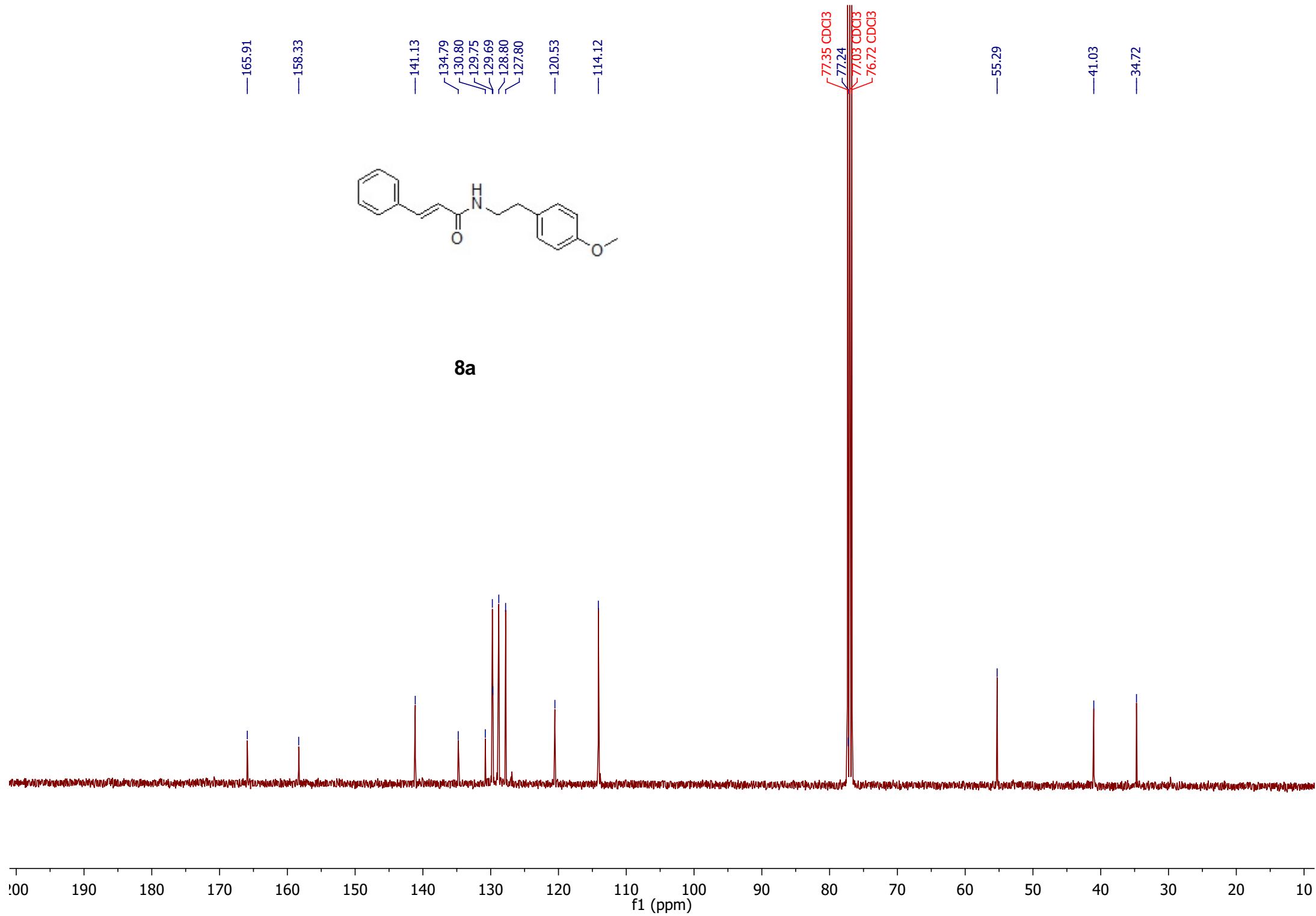


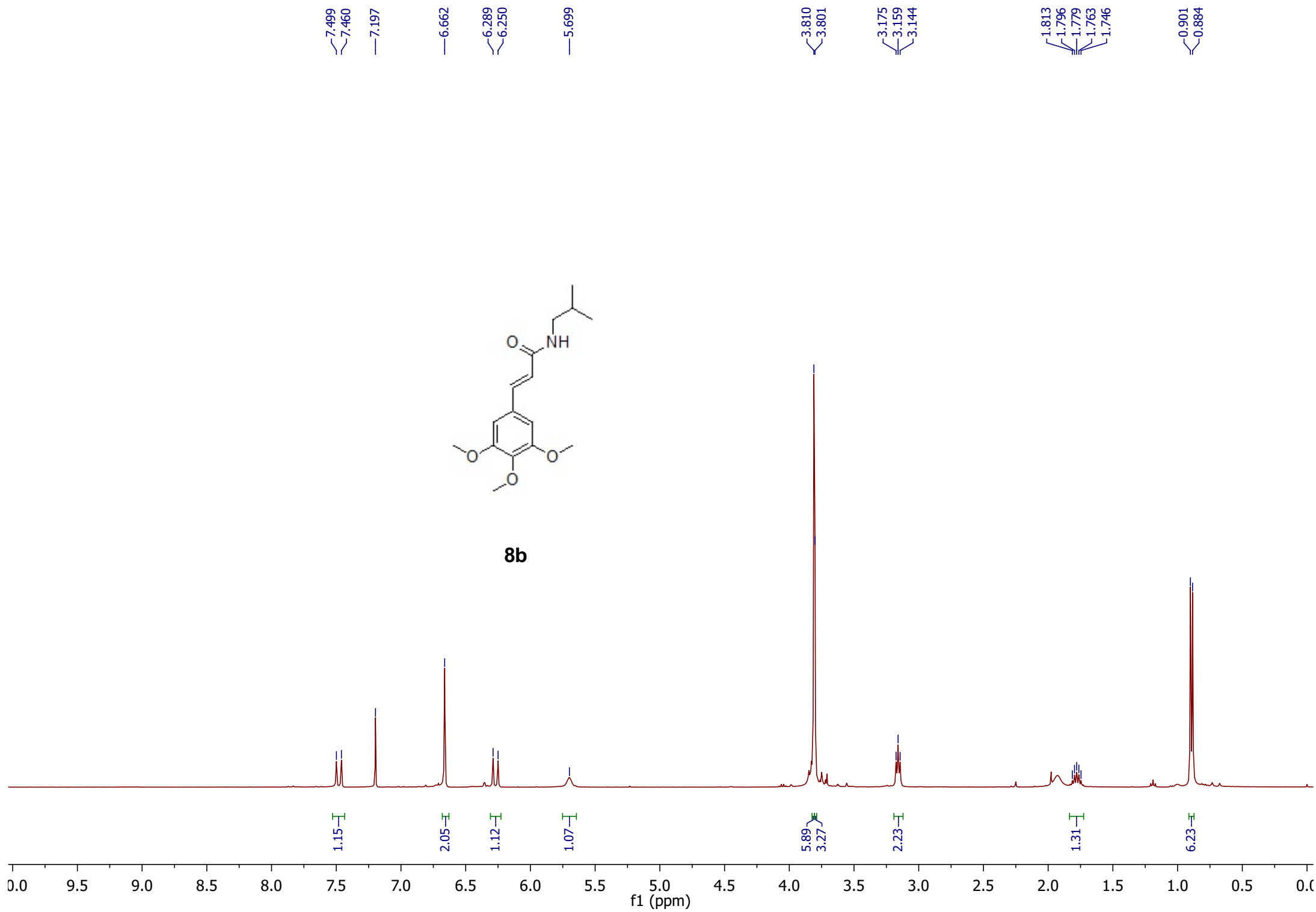
8a

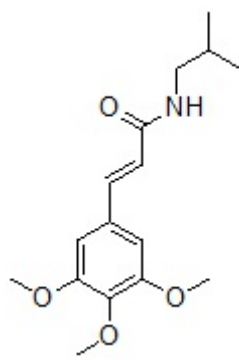




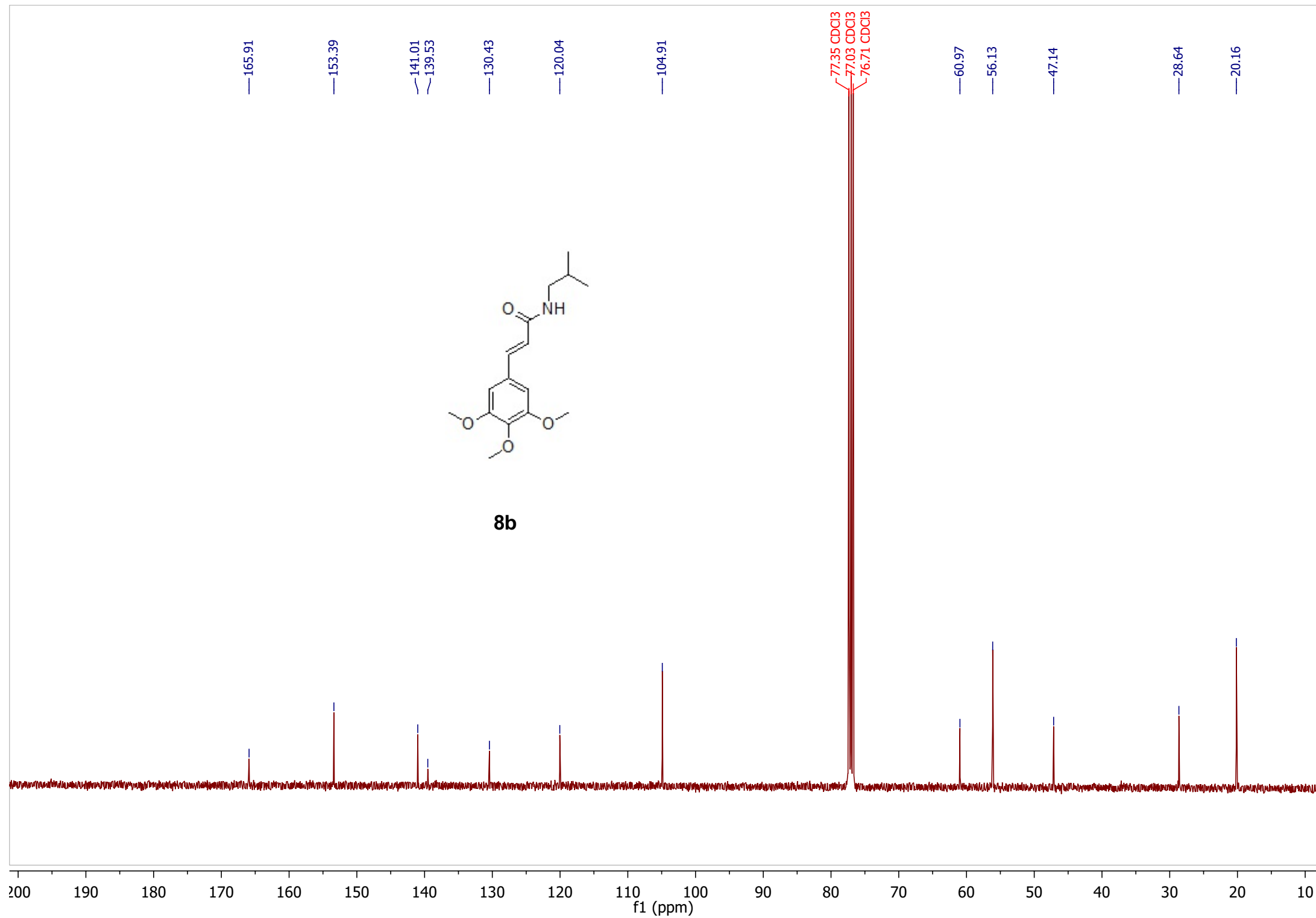
8a

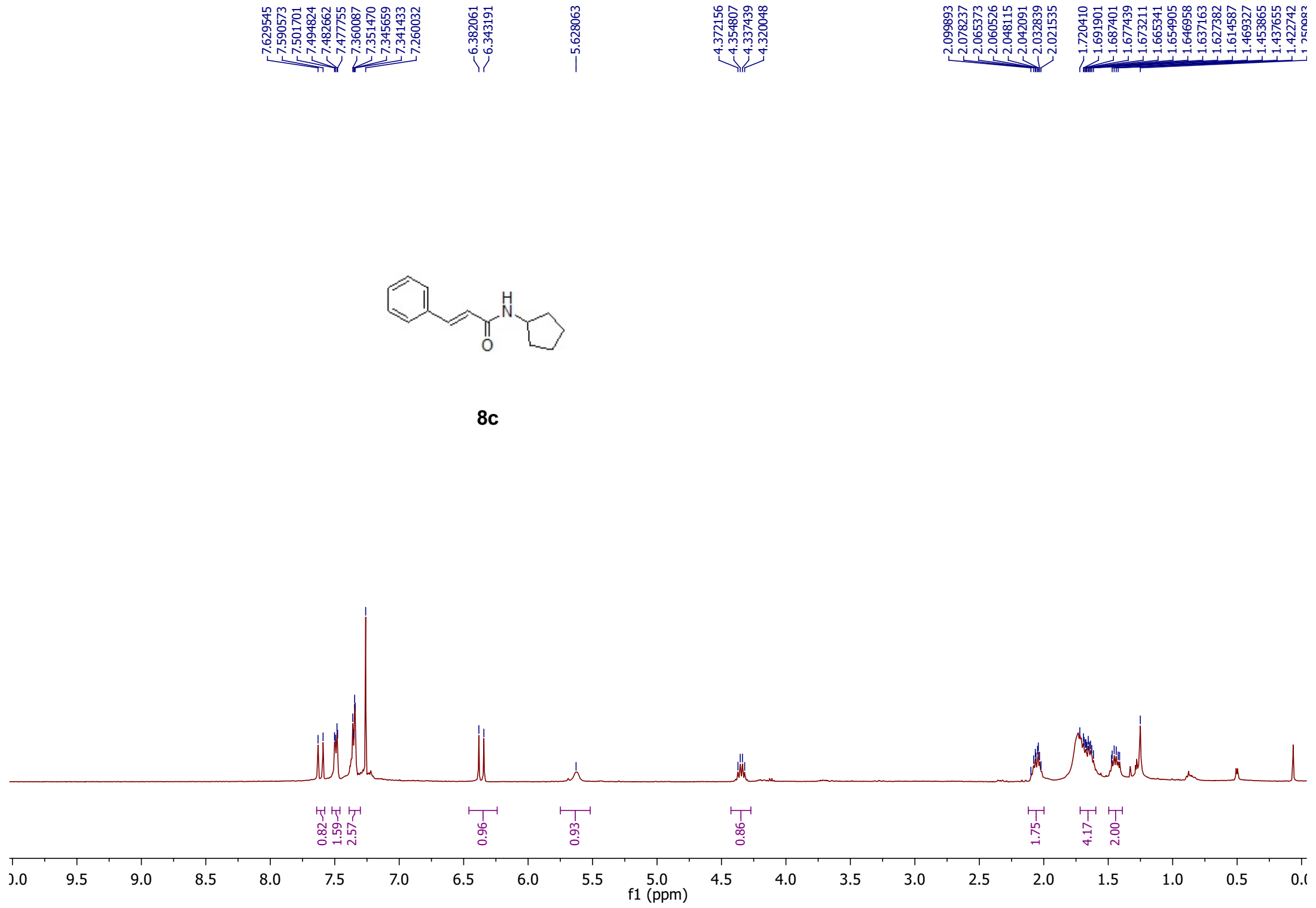


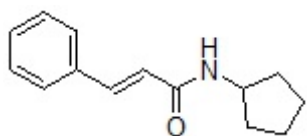




8b







8c

— 165.47

— 140.79

— 134.93

— 129.56

— 128.79

— 127.74

— 120.87

77.33 CDCl₃

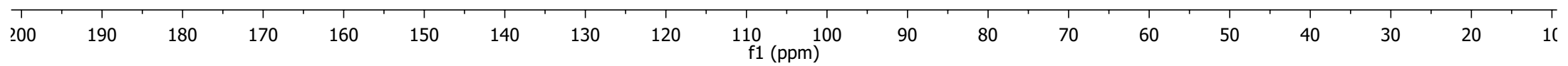
77.00 CDCl₃

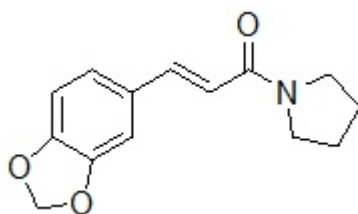
76.70 CDCl₃

— 51.45

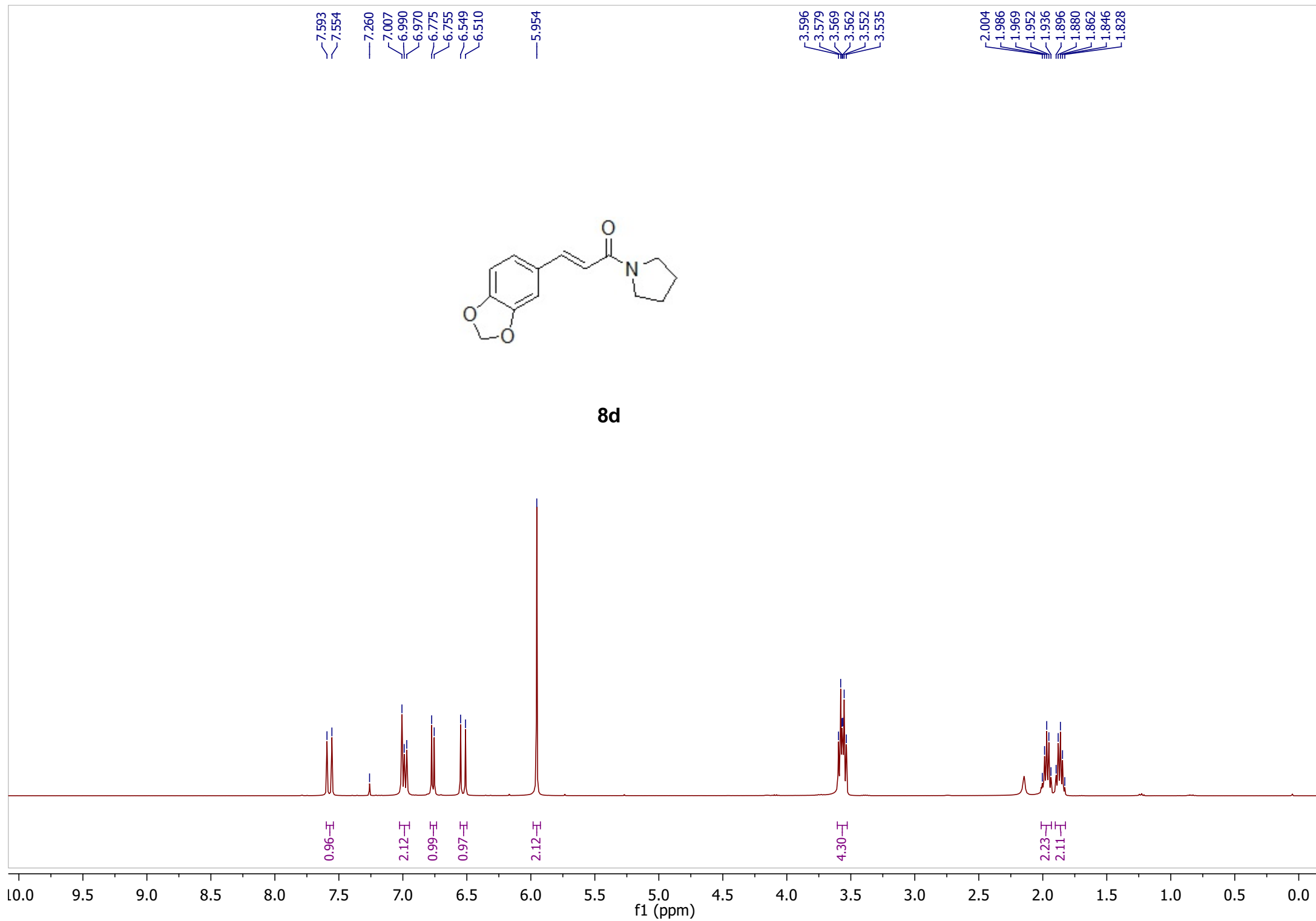
— 33.26

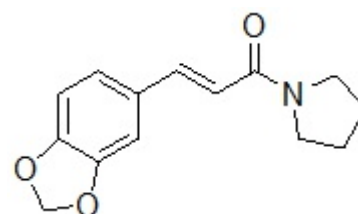
— 23.78



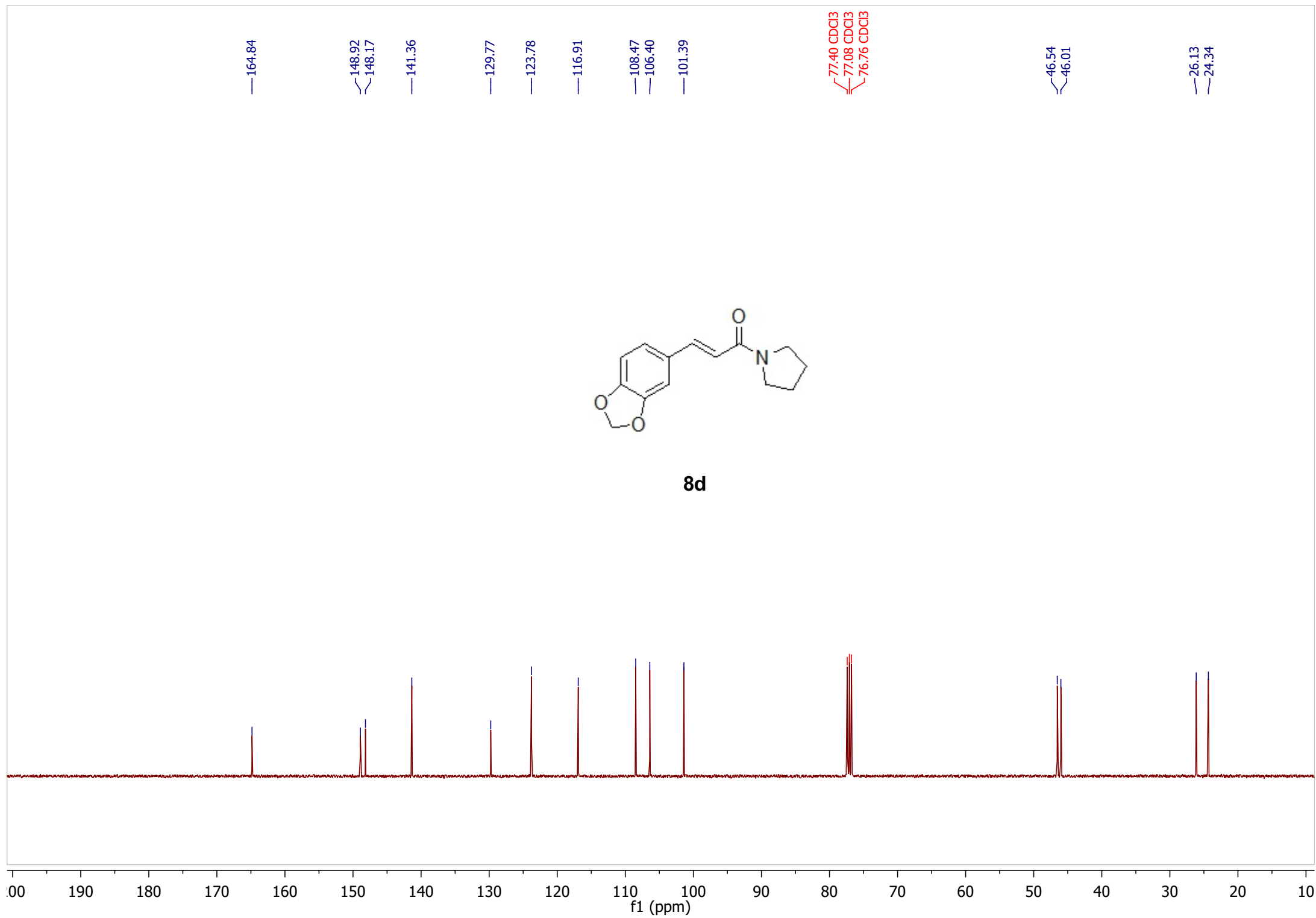


8d





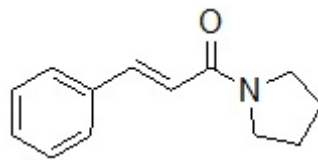
8d



7.713
7.674
7.536
7.531
7.516
7.513
7.385
7.378
7.363
7.345
7.331
7.260
6.747
6.708

3.641
3.624
3.607
3.590
3.573

2.033
2.017
2.000
1.983
1.967
1.929
1.913
1.895
1.878



8e

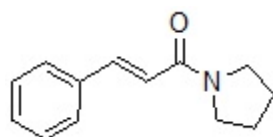
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

1.00
2.03
2.97

1.03

4.02

2.05
1.97



8e

— 164.72

— 141.69

— 135.39

— 129.49

— 128.75

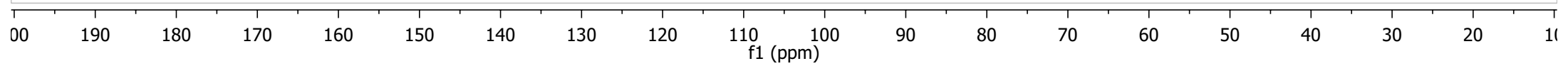
— 127.82

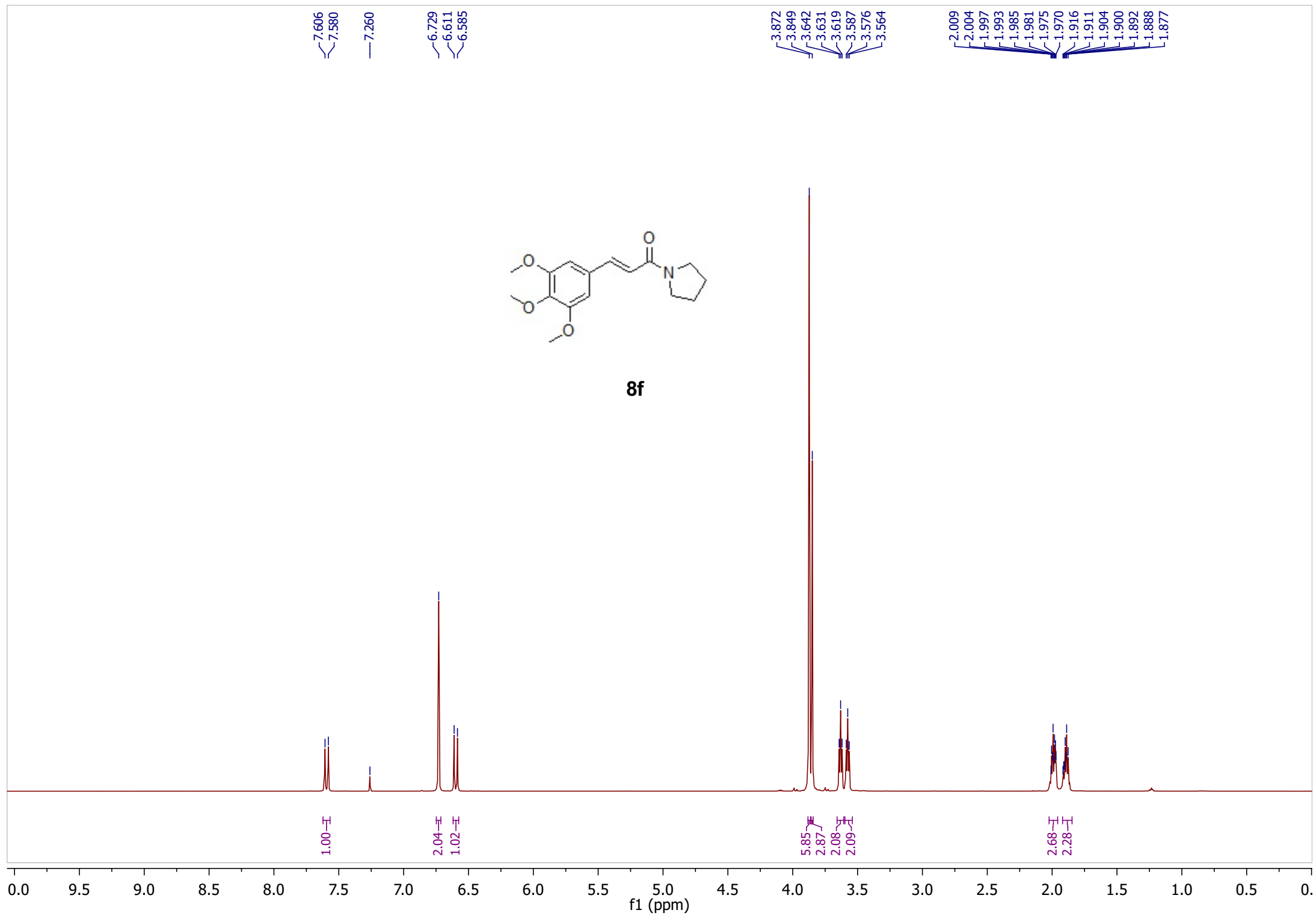
— 118.90

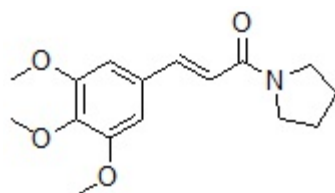
77.35 CDCl₃
77.03 CDCl₃
76.72 CDCl₃

46.60
46.05

26.15
24.35







8f

— 164.65

— 153.38

— 141.78

— 139.58

— 130.92

— 118.10

— 105.12

77.27 CDCl₃
77.05 CDCl₃
76.84 CDCl₃

— 60.93

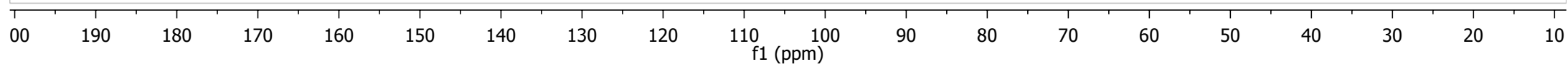
— 56.20

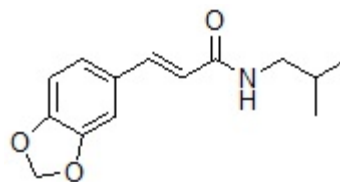
— 46.63

— 46.08

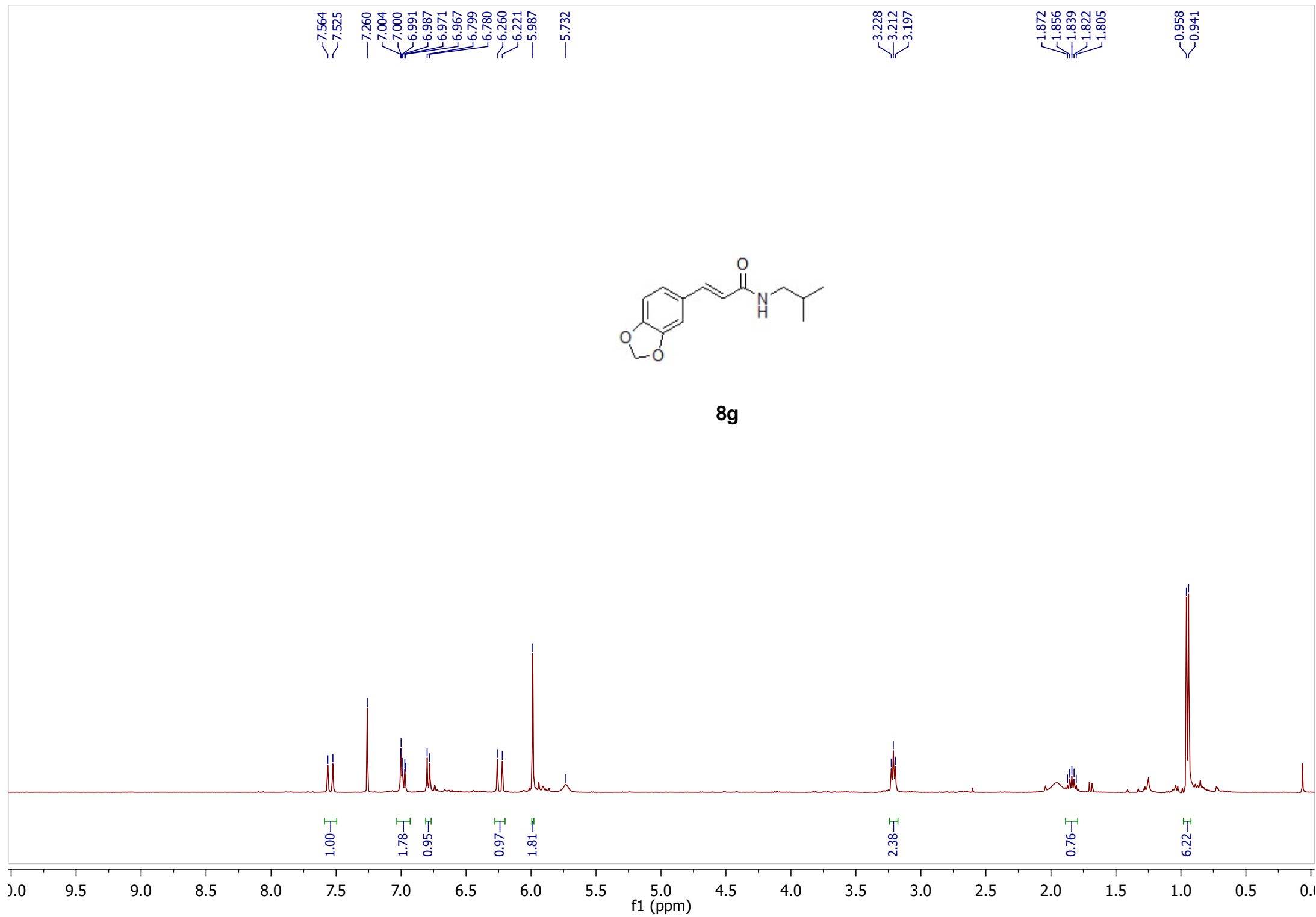
— 26.13

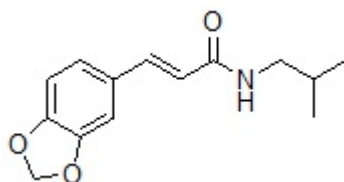
— 24.34



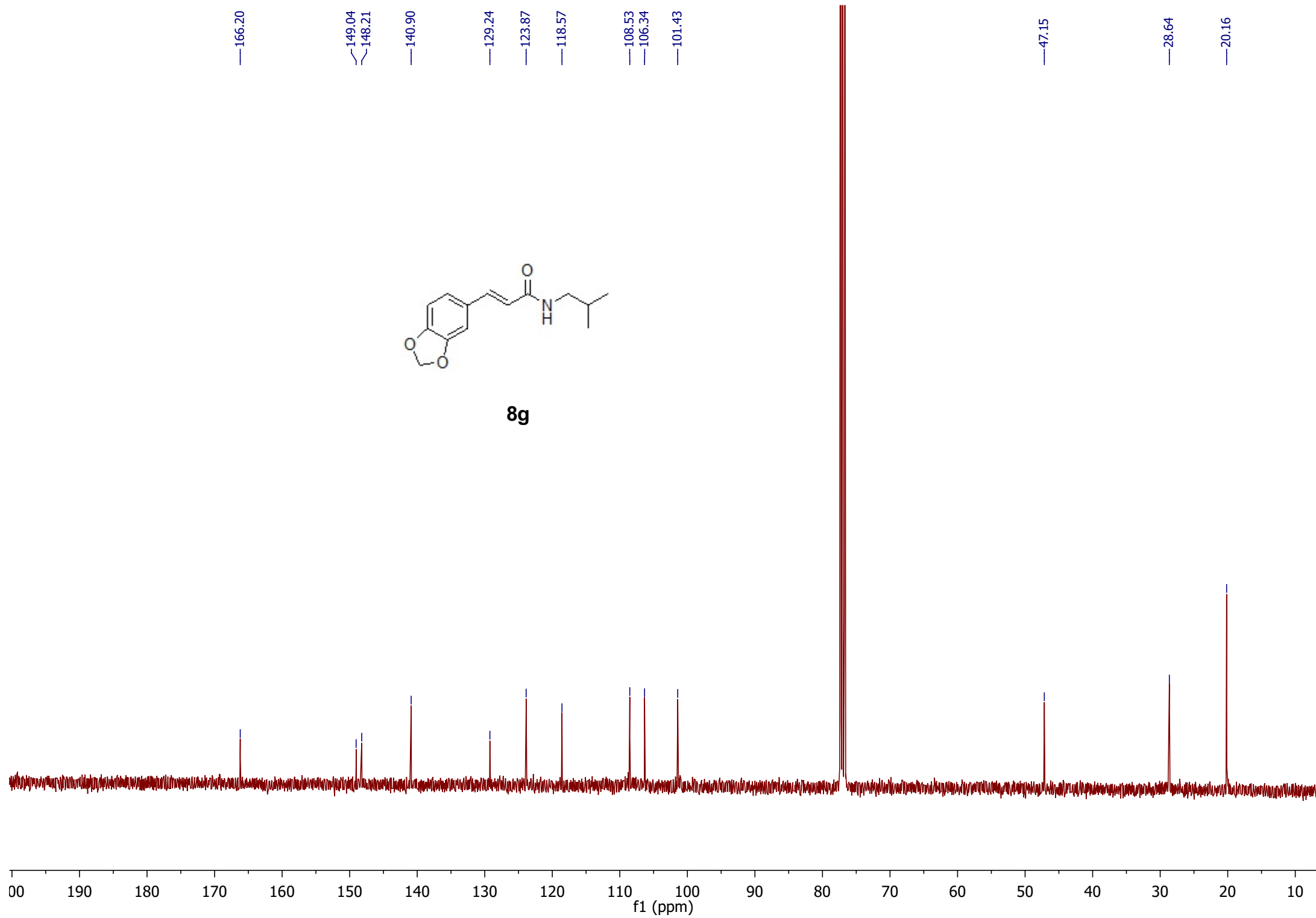


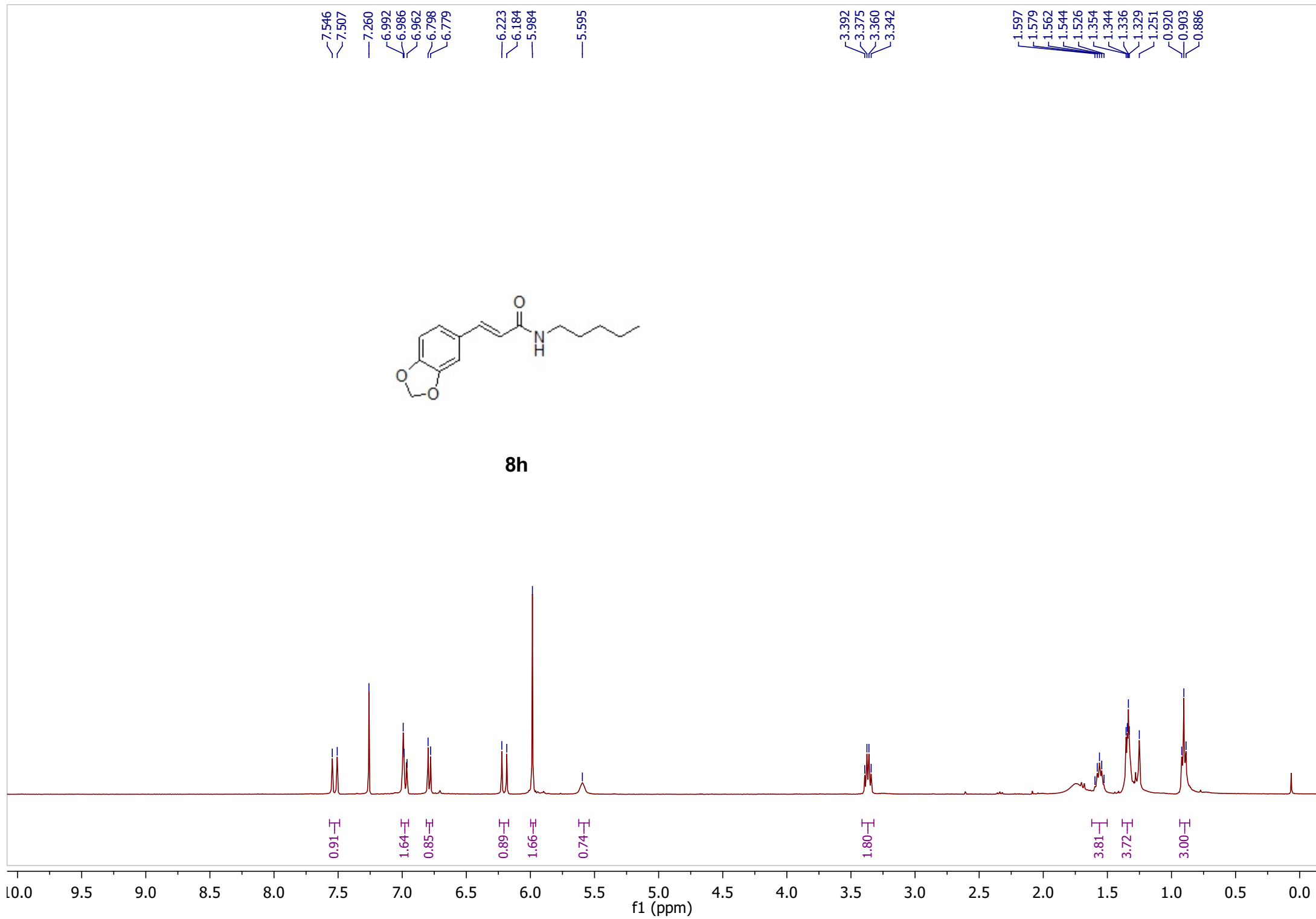
8g

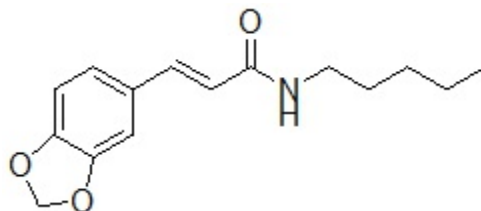




8g







8h

—166.00

148.95
148.19

—140.50

—129.32

—123.75

—118.88

—108.51

—106.31

—101.40

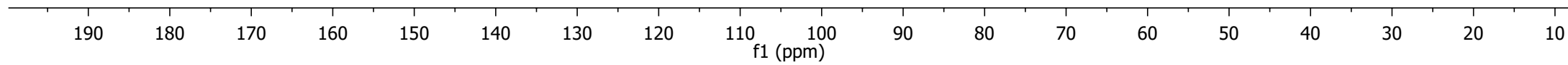
77.35 CDCl₃
77.03 CDCl₃
76.71 CDCl₃

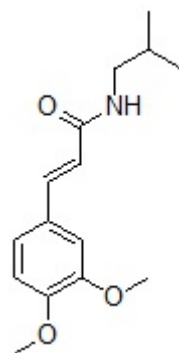
—39.76

29.39
29.11

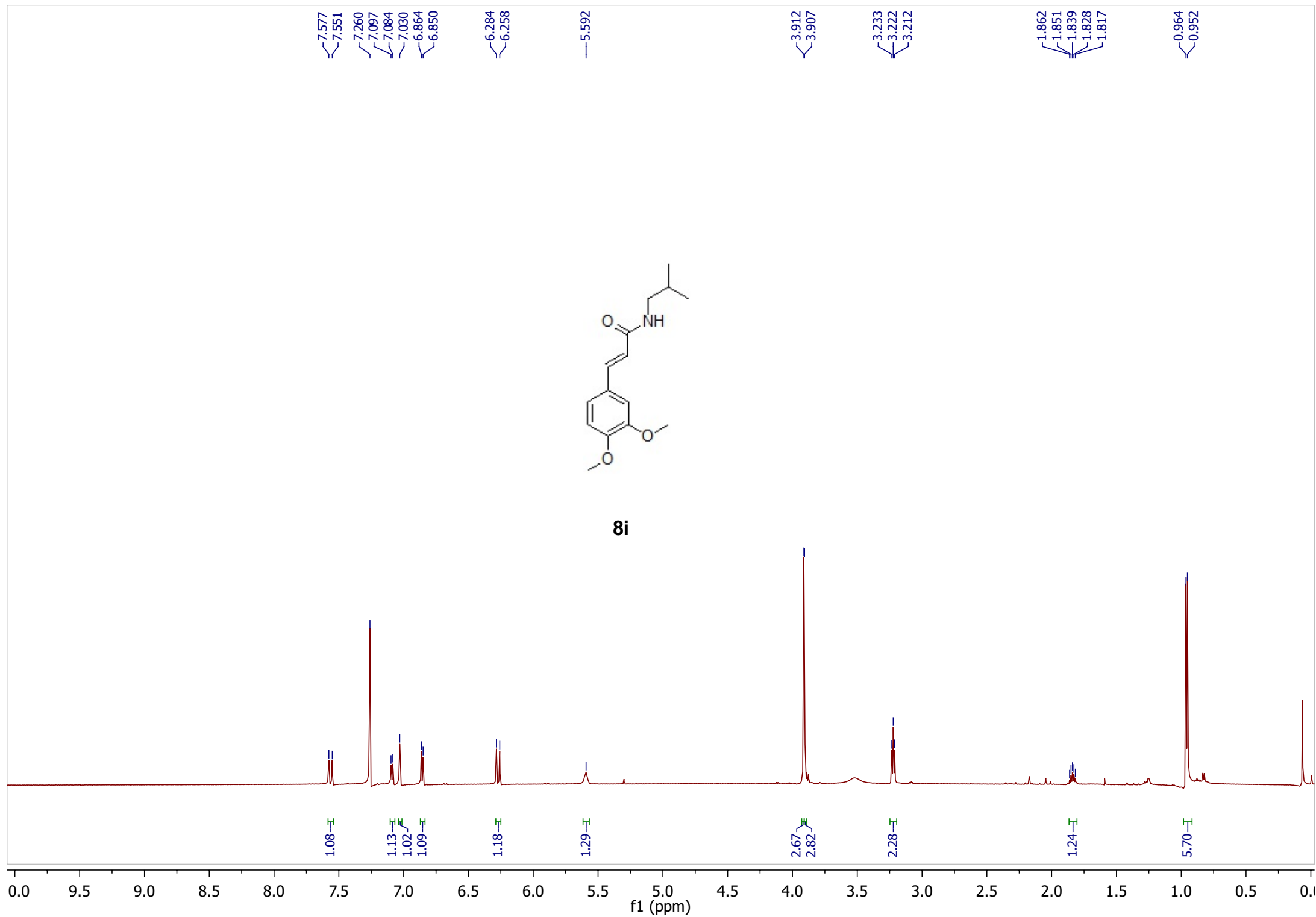
—22.38

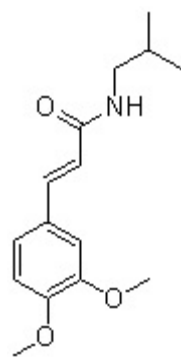
—13.99





8i





8i

166.216

150.524
149.099

140.851

127.832

121.921
118.583

111.063
109.626

55.949
55.867

47.099

28.663

20.167

