Supporting Information

A New Total Synthesis of Patellamide A

Pablo García-Reynaga and Michael S. VanNieuwenhze*

Department of Chemistry, Indiana University, 800 E. Kirkwood Ave., Bloomington, IN 47401

mvannieu@indiana.edu

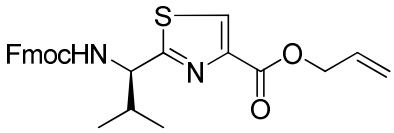
General Methods. Unless otherwise noted, all reactions were carried out in flame-dried glassware under an atmosphere of argon. All solvents were dried over activated alumina using a Seca Solvent system (Glass Countour). Amino acids and coupling reagents were purchased from Nova Biochem or Chem Impex. Burgess reagent and trifluoromethanesulfonic anhydride were purchased from Aldrich. All commercially available reagents were used as received.

^{1}H NMR spectra were measured at 400 MHz on a Varian Gemini-400, at 400 MHz on a Varian Inova-400, or at 500 MHz on a Varian VXR-500 instrument. 13C NMR spectra were measured at 100 MHz on a Varian Gemini spectrometer. Chemical shifts are reported relative to the central line of residual solvent. Infrared spectra were recorded using a Nicolet IR/42 spectrometer FT-IR (thin film, NaCl cells). High resolution mass spectra were obtained via electrospray ionization on an Agilent ESI-TOF spectrometer. Optical rotations were measured on a Perkin–Elmer polarimeter (Model 241) using a 1 mL capacity quartz cell with a 10 cm path length. Analytical thin layer chromatography (TLC) was performed using Whatman glass plates coated with a 0.25

mm thickness of silica gel containing PF 254 indicator, and compounds were visualized with UV light, cerium molybdate stain or ninhydrin stain.

Analytical high performance liquid chromatography (HPLC) was performed on a Beckman-Coulter instrument (System Gold) with diode array detection. Analysis was carried out using Phenomenex Jupiter reverse-phase (C18) column (10µ particle size, 300 Å pore size, 250 mm length x 4.6 mm diameter) with mobile phases consisting of either 1% trifluoroacetic acid in either water or acetonitrile. Preparatory HPLC purifications (Phenomenex Jupiter C18 reverse-phase column, 10µ particle size, 300 Å pore size, 250 S3 mm length x 21.2 mm diameter) were performed with a Waters Millipore Model 510 System with an automated gradient collector and a Model 2487 Dual Absorbance Detector. Flash chromatography purifications were performed using Silicycle 60 Å, 35-75 µm silica gel or Biotage purification system (SP1 HPFC system). All compounds purified by chromatography were sufficiently pure for use in further experiments, unless otherwise noted.

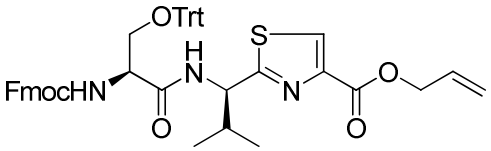
Experimental Section



Fmoc-D-Val-Thzl-OAllyl (2). Thiazole **2** was prepared according to the procedure outlined by Kelly[1]. $[\alpha]^{20}_{D}$ +37 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.76 (d, *J* = 7.38 Hz, 2H), 7.59 (d, *J* = 4.14 Hz, 2H), 7.39 (t, *J* = 7.26 Hz, 2H), 7.30 (t, *J* = 6.36 Hz, 2H), 6.11-5.96 (m, 1H), 5.59 (d, *J* = 8.83 Hz, 1H), 5.41 (dd, *J* = 17.18,

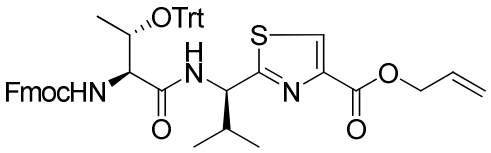
[1] You, S.-L.; Kelly, J.W. *J. Org. Chem.* **2003**, *68*, 9506.

1.24 Hz, 1H), 5.30 (dd, *J* = 10.38, 0.88 Hz, 1H), 4.99-4.90 (m, 1H), 4.85 (d, *J* = 5.73 Hz, 2H), 4.45 (d, *J* = 6.71 Hz, 2H), 4.22 (t, *J* = 6.46 Hz, 1H), 2.5-2.3 (m, 1H), 0.94 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 160.8, 156.0, 147.0, 143.7, 141.3, 131.8, 127.7, 127.2, 127.0, 125.0, 120.0, 118.9, 66.9, 65.9, 58.5, 47.2, 33.4, 19.4,17.6; IR (film) ν_{max} 3413, 3334, 2964, 1728, 1715, 1520, 1506, 1479, 1451, 1321, 1229, 1205, 1099, 1027, 984, 912, 759, 740; ESI MS *m/z* 450.23 $[M+Na]^+$.



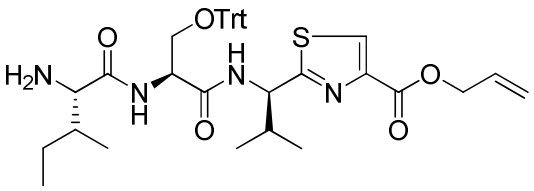
Fmoc-Ser(Trt)-D-Val-Thzl-OAllyl (9). (General Procedure for Piperidine-Mediated Deprotection of Fmoc Group and HBTU, HOBt-Mediated Peptide Coupling). Fmoc-D-Val-Thzl-OAllyl (1.40 g, 3.03 mmol) was dissolved in 25 mL of a 20% piperidine-DMF solution and allowed to stand at room temperature, with ocassional swirling, for 20 minutes. Solvent was then removed and the resulting residue was purified through a short column of silica gel eluted with 15% EtOAc/Hexanes followed by 10% MeOH/$CHCl_3$ to yield the free amine as an orange oil. Dissolution in dry DCM (15 mL) followed by sequential addition of Fmoc-Ser(Trt)-OH (1.72 g, 3.03 mmol), HOBt (430 mg, 3.18 mmol), and HBTU (1.21 g, 3.18 mmol) afforded a suspension which cleared up upon addition of DIEA (1.05 mL, 6.05 mmol). After stirring at room temperature for 12 h, the reaction mixture was diluted with EtOAc, washed with water, sat. aq. $NaHCO_3$, and brine. The organic layer was separated, dried with anhydrous Na_2SO_4 and purified by silica gel chromatography (15-33% EtOAc/Hexanes to afford tripeptide **3** (2.21 g, 2.79 mmol, 90%) as a white foam. $[\alpha]^{22}{}_D$ +14 (*c* 1.0, $CHCl_3$); ^{1}H

NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.56 (d, *J* = 7.00 Hz, 1H), 7.46-7.20 (m, 1H), 7.76 (d, *J* = 7.53 Hz, 1H), 7.17 (d, *J* = 8.47 Hz, 1H), 6.12-5.94 (m, 1H), 5.61 (d, *J* = 6.38 Hz, 1H), 5.40 (qd, *J* = 17.21, 1.43 Hz, 1H), 5.32-5.23 (m, 2H), 4.84 (d, *J* = 4.98 Hz, 1H), 4.43 (m, 1H), 4.36 (d, *J* = 6.70 Hz, 1H), 4.20 (t, *J* = 6.87 Hz, 1H), 3.77-3.66 (m, 1H), 3.24 (dd, *J* = 8.95, 6.14 Hz, 1H), 2.48-2.35 (m, 1H), 0.84 (dd, *J* = 23.59, 6.73 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 161.0, 156.2, 147.2, 143.8, 143.3, 141.4, 132.0, 128.9, 128.7, 128.2, 128.0, 127.9, 127.5, 127.3, 125.3, 120.2, 119.2, 87.7, 67.5, 66.2, 63.9, 56.8, 55.0, 47.2, 33.4, 19.7, 18.0; IR (film) ν_{max} 3422, 3318, 3063, 2961, 2934, 1719, 1495, 1449, 1227, 1207, 1096, 909, 737, 633; ESI MS *m/z* 814.30 $[M+Na]^+$, 830.20 $[M+K]^+$.



Fmoc-*allo*-Thr(Trt)-D-Val-Thzl-OAllyl (7). Compound **7** was synthesized from **2** and Fmoc-*allo*-Thr(Trt)-OH in 86% yield as a white foam by following the procedure used for the synthesis of **9**. $[\alpha]^{22}_D$ +25 (*c* 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.82-7.73 (m, 2H), 7.64-7.55 (m, 2H), 7.53-7.48 (m, 6H), 7.43-7.36 (m, 2H), 7.34-7.20 (m, 13H), 6.62 (d, J = 8.45 Hz, 1H), 6.06-5.96 (m, 1H), 5.40 (d, J = 17.87 Hz, 1H), 5.29 (d, J = 10.43 Hz, 1H), 5.08 (dd, J = 8.71, 6.86 Hz, 1H), 4.82 (d, J = 5.71 Hz, 2H), 4.45-4.19 (m, 3H), 4.14-4.08 (m, 1H), 3.61-3.57 (m, 1H), 2.41-2.29 (m, 1H), 1.03 (d, J = 6.35 Hz, 3H), 0.91-0.80 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 169.3, 160.7, 156.4, 146.8, 144.1, 143.7, 141.2, 131.7, 128.7, 127.9, 127.7, 127.3, 127.2, 127.1, 125.1, 125.0, 119.9, 118.9, 87.6, 69.6, 67.4, 65.9, 59.2, 59.1, 56.1, 46.9, 33.1, 19.4, 17.8, 17.0;

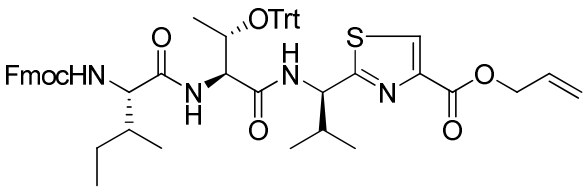
IR (film) ν_{max} 3422, 3383, 3320, 1719, 1505, 1450, 1228, 1203, 1086, 908, 758, 733, 704; ESI MS *m/z* 828.36 $[M+Na]^+$.



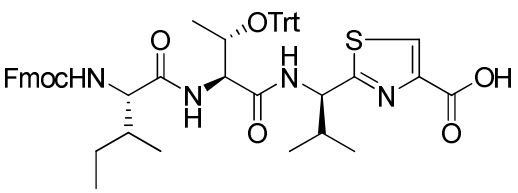
H-Ile-Ser(Trt)-D-Val-Thzl-OAllyl (6). Compound **10** was synthesized from **9** and Fmoc-Ile-OH in 88% yield by following the procedure used for the synthesis of **9**. with the exception that the precipitated solid product was filtered and collected. Trituration of the solid with 1:4 EtOAc/Hexanes afforded tetrapeptide **10** as a white solid, which was subjected to the next reaction conditions without further preparation.

The Fmoc-protected tetrapeptide above was dissolved in 20% piperidine-DMF and allowed to stand at room temperature for 20 minutes. Solvent was evaporated and the residue purified by silica gel chromatography (15% EtOAc/Hexanes followed by 10% MeOH/$CHCl_3$) to yield free amine **6** as an orange oil. $[\alpha]^{20}{}_D$ +1.0 (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, *J* = 7.32 Hz, 1H), 8.08-7.98 (m, 1H), 7.47 (d, *J* = 8.62 Hz, 1H), 7.42-7.36 (m, 6H), 7.31-7.16 (m, 9H), 6.07-5.94 (m, 1H), 5.44-5.35 (m, 1H), 5.33-5.16 (m, 2H), 4.83 (d, *J* = 5.74 Hz, 1H), 4.70-4.62 (m, 1H), 3.78-3.67 (m, 1H), 3.49-3.45 (m, 1H), 3.38-3.35 (m, 1H), 3.32-3.21 (m, 1H), 2.48-2.37 (m, 1H), 2.05-1.90 (m, 1H), 1.77-1.47 (m, 3H), 1.11-0.98 (m, 1H), 0.96-0.79 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.4, 171.9, 170.0, 160.8, 146.7, 143.3, 131.8, 128.5, 127.8, 127.2, 127.1, 118.8, 118.8, 86.9, 65.8, 62.9, 59.7, 56.5, 52.9, 46.8, 40.6, 37.7, 26.5, 25.0, 24.7, 23.5,

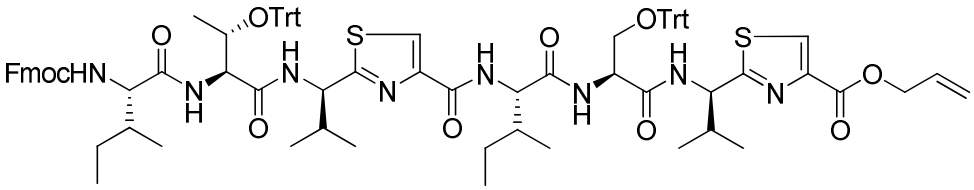
19.3, 17.5, 16.2, 11.9; IR (film) ν_{max} 3291, 3089, 3063, 3036, 3024, 2961, 2932, 2874, 1733, 1653, 1498, 1449, 1228, 1206, 1095, 912, 732, 706; ESI MS *m/z* 705.31 $[M+Na]^+$.



Fmoc-Ile-*allo*-Thr(Trt)-D-Val-Thzl-OAllyl (8). Compound **8** was synthesized from **7** and Fmoc-Ile-OH in 92% yield as a white foam by following the procedure used for the synthesis of **9**. $[\alpha]^{20}{}_D$ +7.4 (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.78 (d, *J* = 7.21 Hz, 2H), 7.58-7.51 (m, 2H), 7.50-7.36 (m, 6H), 7.32-7.18 (m, 13H), 6.80 (d, *J* = 8.07 Hz, 1H), 6.60 (d, *J* = 7.04 Hz, 1H), 6.07-5.89 (m, 1H), 5.37 (d, *J* = 17.16 Hz, 1H), 5.31-5.19 (m, 2H), 5.10 (t, *J* = 7.19 Hz, 1H), 4.78 (d, *J* = 5.29 Hz, 2H), 4.54 (dd, *J* = 10.05, 6.81 Hz, 1H), 4.35-4.27 (m, 1H), 4.21 (t, *J* = 6.17 Hz, 1H), 4.14-4.05 (m, 1H), 4.01 (t, *J* = 5.46 Hz, 1H), 3.97-3.91 (m, 1H), 2.49-2.34 (m, 1H), 1.99-1.83 (m, 2H), 1.17-1.05 (m, 1H), 1.02-0.77 (m, 15H), 1.55-1.37 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 171.0, 169.0, 160.7, 156.6, 146.6, 144.1, 143.6, 143.5, 141.2, 87.4, 69.6, 67.1, 65.8, 60.4, 57.3, 56.5, 47.0, 36.8, 32.9, 24.7, 19.3, 17.6, 17.1, 15.6, 11.4; IR (film) ν_{max} 3312, 3060, 2961, 2933, 1711, 1655, 1510, 1449, 1227, 1207, 1084, 1032, 911, 760, 739, 707; ESI MS *m/z* 941.31 $[M+Na]^+$.

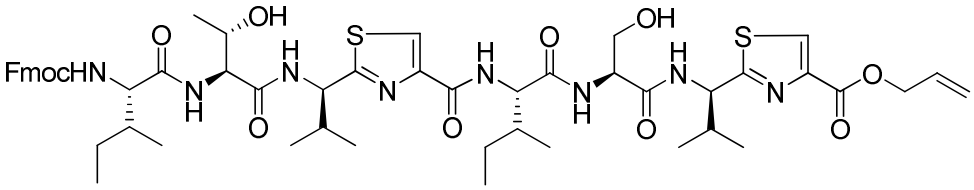


Fmoc-Ile-*allo*-Thr(Trt)-D-Val-Thzl-OH (5). To a solution of allyl ester **8** (1.50 g, 1.63 mmol) in 8.16 mL CH_2Cl_2 was added $Pd(PPh_3)_4$ (47 mg, 0.041 mmol), followed by $PhSiH_3$ (403 µL, 3.26 mmol). After stirring for 6 h, the brown solution was concentrated *in vacuo* and purified by silica-gel chromatography (33% Hex/EtOAc, then 10% MeOH/$CHCl_3$), affording the carboxylic acid **5**(1.41 g, 1.604 mmol, 98%) as a light brown foam. The product was used in the next step without further purification.



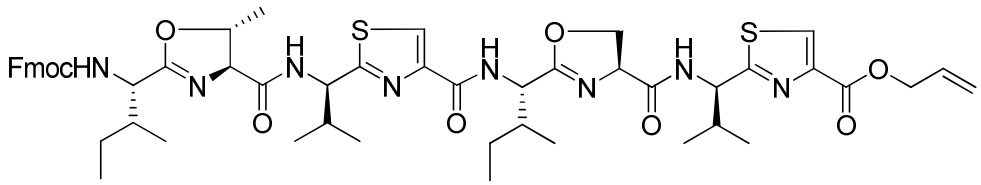
Fmoc-Ile-*allo*-Thr(Trt)-D-Val-Thzl- Ile-Ser(Trt)-D-Val-Thzl-OAllyl (11). To a solution of carboxylic acid **5** (1.35 g, 1.54 mmol) in CH_2Cl_2 (7.7 mL) was added HOBt (218 mg, 1.61 mmol), HBTU (612 mg, 1.61 mmol) and DIEA (535 µL, 3.07 mmol). Free amine **6** (1.46 g, 1.61 mmol) was subsequently added and the mixture stirred at room temperature overnight. The reaction mixture was then diluted with EtOAc, washed with water, sat. aq. $NaHCO_3$ and brine. The organic layer was separated and dried with anhydrous Na_2SO_4. After filtration and evaporation of the solvent *in vacuo*, the oily residue was purified by silica gel chromatography (33% EtOAc/Hex) to give octapeptide **11** (2.10 g, 1.36 mmol, 84%) as a white foam. $[\alpha]^{21}$ +9.4 (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.79-7.56 (m, 6H), 7.50-7.00 (m, 33H), 6.80-6.63 (m, 4H), 5.98-5.84 (m, 1H), 5.38-5.14 (m, 4H), 5.04-4.95 (m, 1H), 4.80-4.59 (m, 3H), 4.36 (t, *J* = 11.0 Hz, 1H), 4.19 (t, *J* = 5.1 Hz, 1H), 4.14-3.94 (m, 4H), 3.79 (d, *J* = 9.3 Hz, 1H), 3.02 (dd, *J* = 8.9, 3.2 Hz, 1H), 2.65-2.53 (m, 1H), 2.33-2.14 (m, 1H), 1.91-1.76 (m, 2H), 1.44-

1.26 (m, 2H), 1.11-1.02 (m, 1H), 1.01-0.72 (m, 31H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 218.5, 172.3, 171.9, 171.4, 170.0, 169.7, 169.2, 161.8, 160.8, 156.6, 148.3, 146.8, 146.6, 144.1, 143.4, 143.3, 143.0, 141.2, 131.8, 128.7, 127.8, 127.3, 127.1, 127.1, 124.9, 124.8, 124.7, 124.5, 120.0, 120.0, 118.8, 87.5, 86.7, 86.7, 81.9, 69.1, 68.3, 67.2, 67.0, 65.8, 65.7, 63.0, 60.4, 59.1, 57.6, 57.0, 56.1, 56.0, 53.1, 52.9, 46.9, 37.0, 36.8, 32.8, 32.3, 25.0, 24.9, 24.8, 24.7, 20.0, 19.7, 19.6, 19.4, 19.3, 18.0, 17.2, 17.1, 15.7, 15.6, 15.5, 15.2, 11.6, 11.5, 11.1; IR (film) ν_{max} 3396, 3301, 3059, 2964, 2932, 2874, 1646, 1536, 1490, 1449, 1228, 1209, 1092, 1034, 909, 759, 34, 702; ESI MS *m/z* 1565.52 $[M+Na]^+$.



Fmoc-Ile-*allo*-Thr-D-Val-Thzl- Ile-Ser-D-Val-Thzl-OAllyl (12). To a stirring solution of the trityl-protected peptide **11** (500 mg, 323 μmol) in CH_2Cl_2 (8 mL) was added TFA (66.7 μL), followed by PhSH (66.7 μL, 648 μmmol). The reaction was monitered by TLC and was complete within 5 min. Solvent wasthen removed and the residue triturated with hexanes to yield diol **12** (340 mg, 321 μmol, 99%) as a white solid. $[\alpha]^{24}_{D}$ +5.0 (*c* 1.0, DMSO); ^{1}H NMR (500 MHz, DMSO-*d*6) δ 8.59-8.48 (m, 2H), 8.48 (s, 1H), 8.33 (d, *J* = 8.02 Hz, 1H), 8.17 (s, 1H), 7.93-7.81 (m, 3H), 7.71-7.65 (m, 2H), 7.41-7.32 (m, 2H), 7.30-7.23 (m, 2H), 6.09-5.94 (m, 1H), 5.39 (dd, *J* = 17.22, 1.32 Hz, 1H), 5.27 (dd, *J* = 10.47, 0.85 Hz, 1H), 5.01-4.88 (m, 3H), 4.87-4.73 (m, 3H), 4.56-4.45 (m, 2H), 4.40 (t, *J* = 8.08 Hz, 1H), 4.32-4.14 (m, 3H), 3.93 (t, *J* = 8.34 Hz, 1H), 3.87-3.76 (m, 1H), 3.66-3.54 (m, 2H), 2.37-2.22 (m, 2H), 1.86-1.75 (m, 1H), 1.74-1.62 (m,

1H), 1.50-1.31 (m, 2H), 1.07 (d, *J* = 6.17 Hz, 3H), 0.96-0.70 (m, 28H); ^{13}C NMR (100 MHz, DMSO-*d*6) δ 174.4, 174.3, 172.0, 171.5, 171.4, 171.1, 161.3, 160.8, 156.9, 149.8, 146.4, 144.8, 144.7, 141.6, 133.4, 130.2, 128.6, 128.0, 126.3, 124.9, 121.0, 119.2, 90.2, 67.9, 66.5, 66.0, 62.6, 60.0, 59.2, 57.3, 55.9, 47.6, 38.5, 37.3, 33.1, 33.0, 25.3, 21.1, 20.3, 20.2, 18.9, 18.8, 16.3, 12.0, 11.8; IR (KBr) ν_{max} 3317(br), 3068, 2966, 2932, 2879, 1681(br), 1524(br), 984, 760, 739; ESI MS *m/z* 1081.44 $[M+Na]^+$.

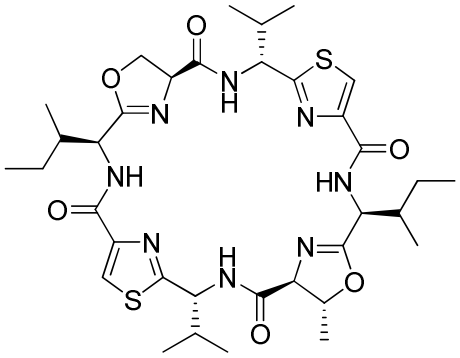


Fmoc-Ile-*allo*-Thr(Oxzn)-D-Val-Thzl-Ile-Ser(Oxzn)-D-Val-Thzl-OAllyl (4).

Following Wipf's procedure,[2] diol **12** (100 mg, 94.4μmol) was suspended in THF (12.4 mL), and the Burgess reagent (74.9 mg, 314 μmol) added. The mixture was then stirred at 55 ˚C for 1 h and at 77 ˚C for 4 h. After evaporation of the solvent *in vacuo*, the residue was purified by silica gel chromatography (40-100% EtOAc/Hexanes) to produce the bis-oxazoline **4** (77 mg, 0.075 mmol, 80%) as a white foam. $[\alpha]^{22}_{D}$ +44 (*c* 1.0, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.81-7.66 (m, 3H), 7.60-7.48 (m, 1H), 7.45-7.24 (m, 5H), 7.18 (d, *J* = 8.5 Hz, 1H), 6.09-5.91 (m, 1H), 5.46-5.34 (m, 2H), 5.32-5.10 (m, 3H), 4.88-4.72 (m, 4H), 4.61-4.34 (m, 4H), 4.29-4.15 (m, 3H), 2.64-2.39 (m, 2H), 2.06-1.94 (m, 1H), 1.91-1.80 (m, 1H), 1.49 (d, *J* = 6.2 Hz, 3H), 1.32-1.15 (m, 24H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 172.2, 171.5, 171.4, 169.6, 168.9, 160.7, 160.4, 155.8, 149.4, 146.9, 143.6, 143.5, 141.2, 131.7, 127.7, 127.0, 126.9, 124.9, 124.7, 123.0, 120.0, 118.8, 80.7, 77.2, 74.3, 70.8, 68.1, 66.8, 65.8, 56.5, 56.4, 54.0, 51.8, 47.0,

[2] Wipf, P.; Fritch, P.C. *J. Am. Chem. Soc.*, **1996**, *118*, 12358.

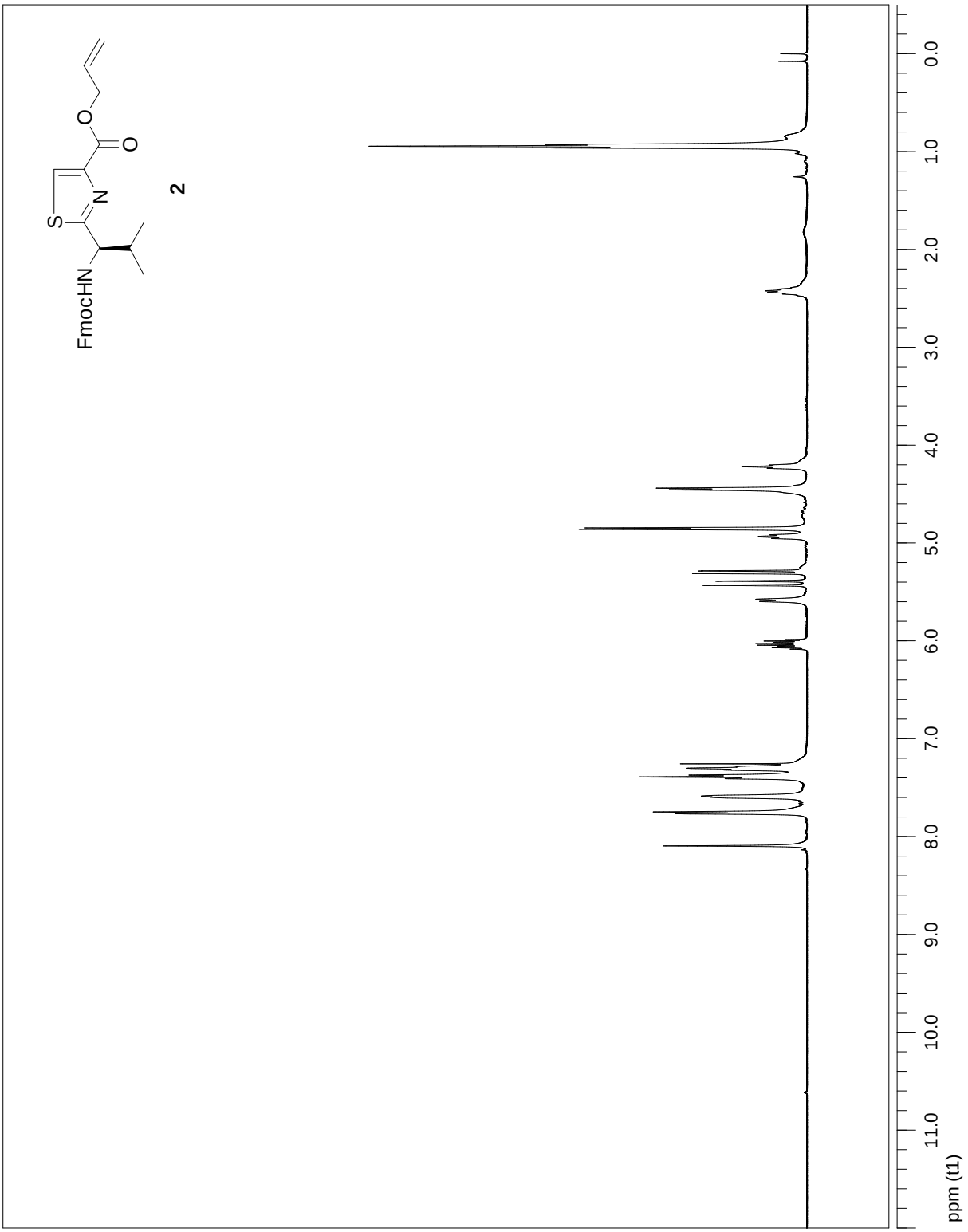
37.4, 37.3, 32.6, 32.5, 25.1, 25.1, 21.8, 19.4, 19.3, 17.6, 17.4, 15.3, 15.2, 11.1; IR (film) ν_{max} 3391, 3313, 2964, 2932, 2876, 1806, 1718, 1670, 1539, 1386, 1371, 1334, 1321, 1228, 1203, 1033, 1984, 910, 733; ESI MS *m/z* 1023.30 $[M+H]^+$, 1045.42 $[M+Na]^+$.



Patellamide A (1). To a solution of **4** (77 mg, 75 μmol) in CH_2Cl_2 (752 μL) was added $Pd(PPh_3)_4$ (2.17 mg, 1.88 μmmol), followed by $PhSiH_3$ (20.4 μL, 150 μmmol), and the solution was stirred under Argon at room temperature for 6 h. After removal of the solvent *in vacuo*, the residue was purified by silica gel chromatography (33% EtOAc/Hexanes, then 10% MeOH/$CHCl_3$) to yield the free acid as a light brown foam. The product was used in the next step without further purification.

The above Fmoc-protected acid was dissolved in 20% piperidine-DMF (3 mL) and allowed to stand at room temperature, with ocassional swirling, for 20 minutes. After evaporation of the solvent, the residue was triturated with hexanes (3x15 mL) to remove the dibenzofulvene byproducts. The resulting residue was dissolved in 2:1 CH_2Cl_2:DMF (6 mL) and slowly added to a solution of PyBOP (78 mg, 151 μmol), DIEA (16.0 μL, 90 μmol) and DMAP (18.4 mg, 151 μmol) in 2:1 CH_2Cl_2:DMF (16.7 mL) over 21 h using a syringe pump. After completion of the addition, the solution was stirred for another 3 hours followed by the removal of solvent *in vacuo*. The resulting residue was dissolved

in EtOAc and washed with water, sat. aq. $NaHCO_3$, and brine. Purification by silica gel chromatography (15-50% EtOAc/Hexanes) produced the title compound (31 mg, 75 μmol, 55%) as an amorphous solid. $[\alpha]^{21}_D$ +110 (*c* 0.1, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (m, 2H), 7.82 (s, 1H), 7.40 (d, *J* = 10.1 Hz, 2H), 5.28-5.17 (m, 2H), 4.94-4.73 (m, 4H), 4.69-4.62 (m, 1H), 4.59-4.52 (m, 1H), 4.30 (d, *J* = 5.5 Hz, 1H), 2.39-2.25 (m, 2H), 2.10-1.84 (m, 1H), 1.48 (d, *J* = 6.2 Hz, 3H), 1.18-1.05 (m, 12H), 0.86-0.70 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 171.6, 169.5, 169.2, 168.5, 160.5, 149.5, 149.4, 123.1, 81.7, 73.6, 72.2, 67.4, 55.1, 55.0, 52.5, 52.2, 37.1, 36.9, 33.4, 24.9, 24.8, 21.8, 19.3, 18.0, 15.1, 15.0, 11.1, 10.6; IR (film) ν_{max} 3385, 2957, 2920, 2851, 1683, 1538, 1505, 1487, 1462, 1261, 1093, 1023, 866, 799; HR-EIMS *m/z* 742.3292 $[M+H]^+$ (742.3289 expected for $C_{35}H_{50}O_6N_8S_2$).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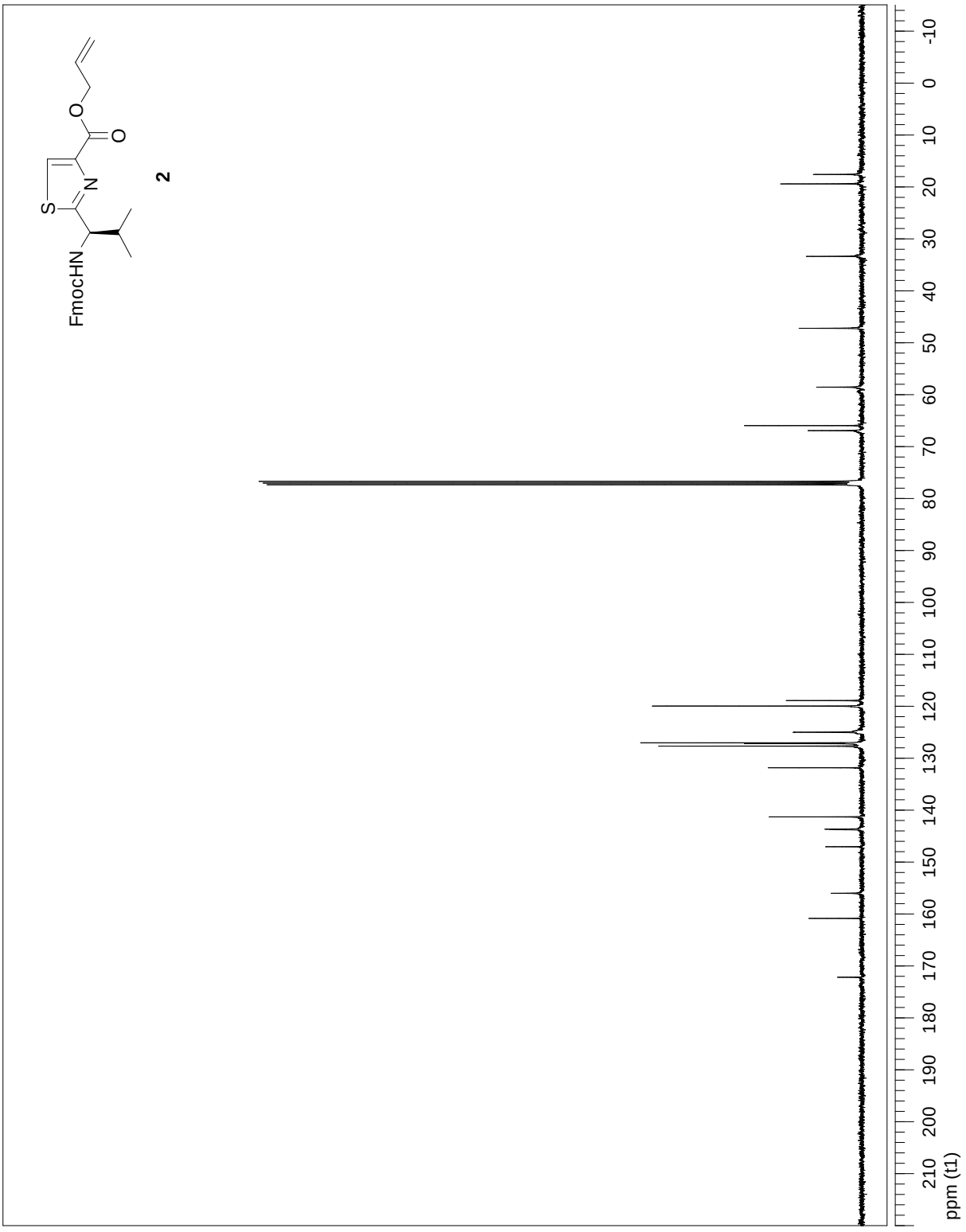
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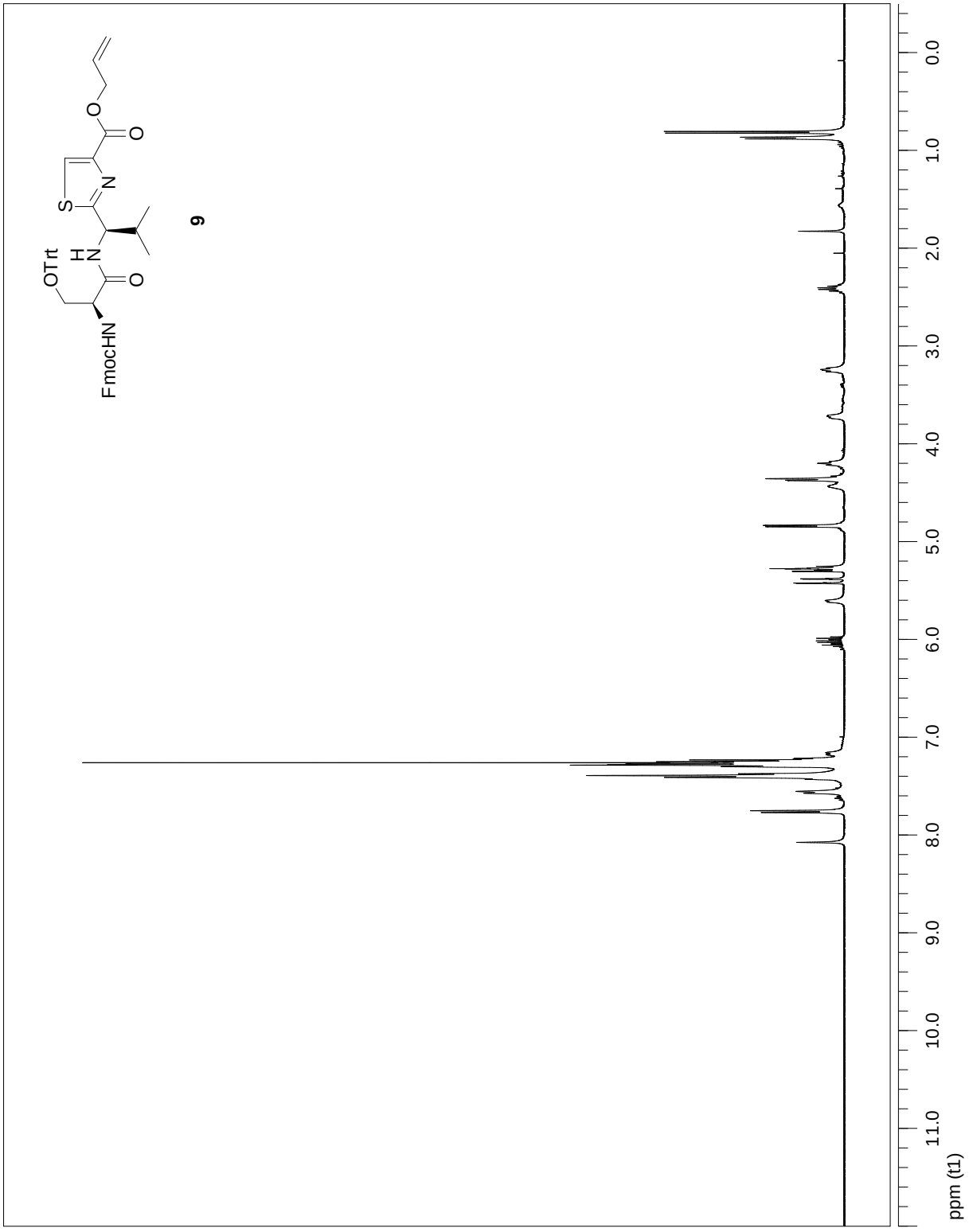
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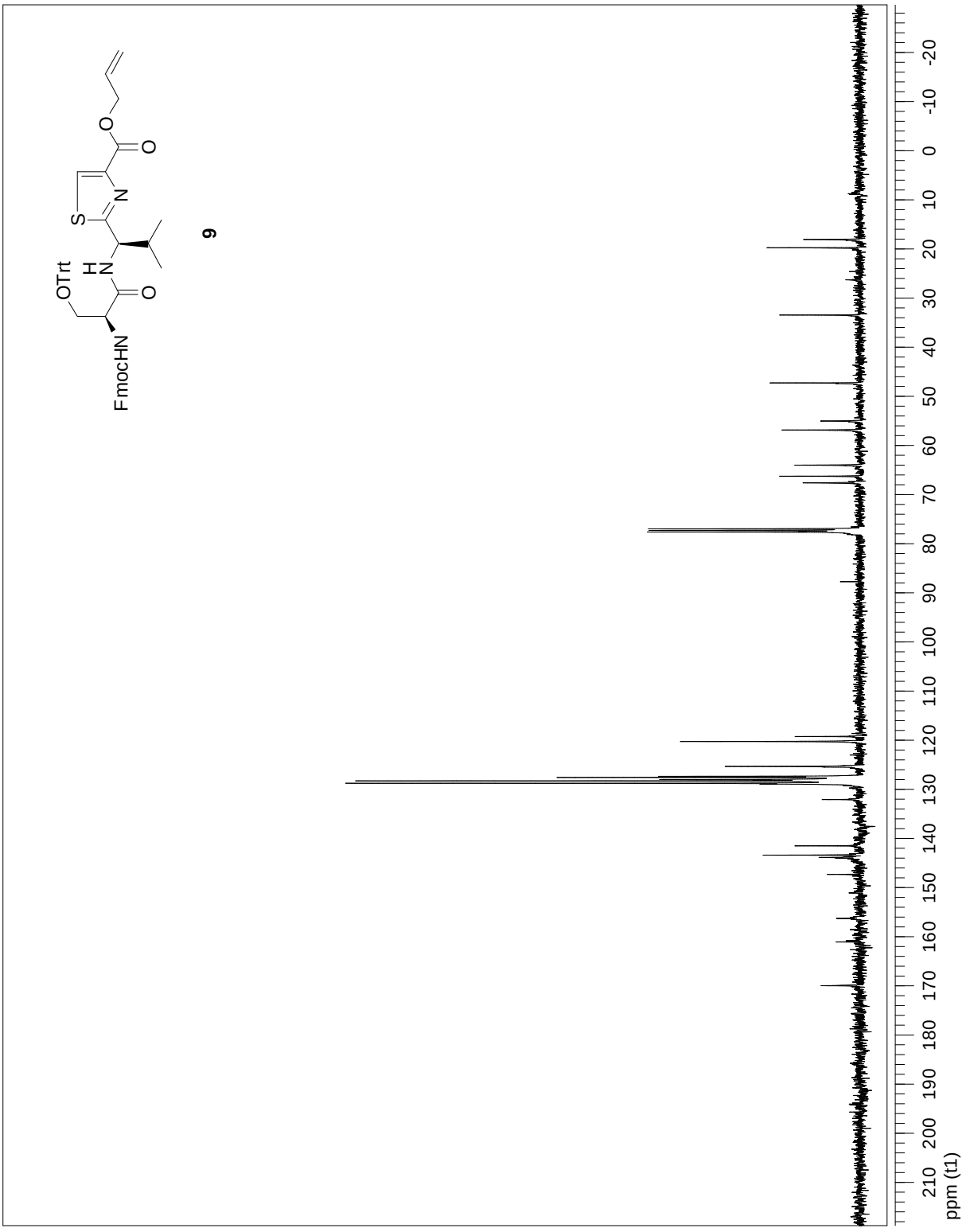
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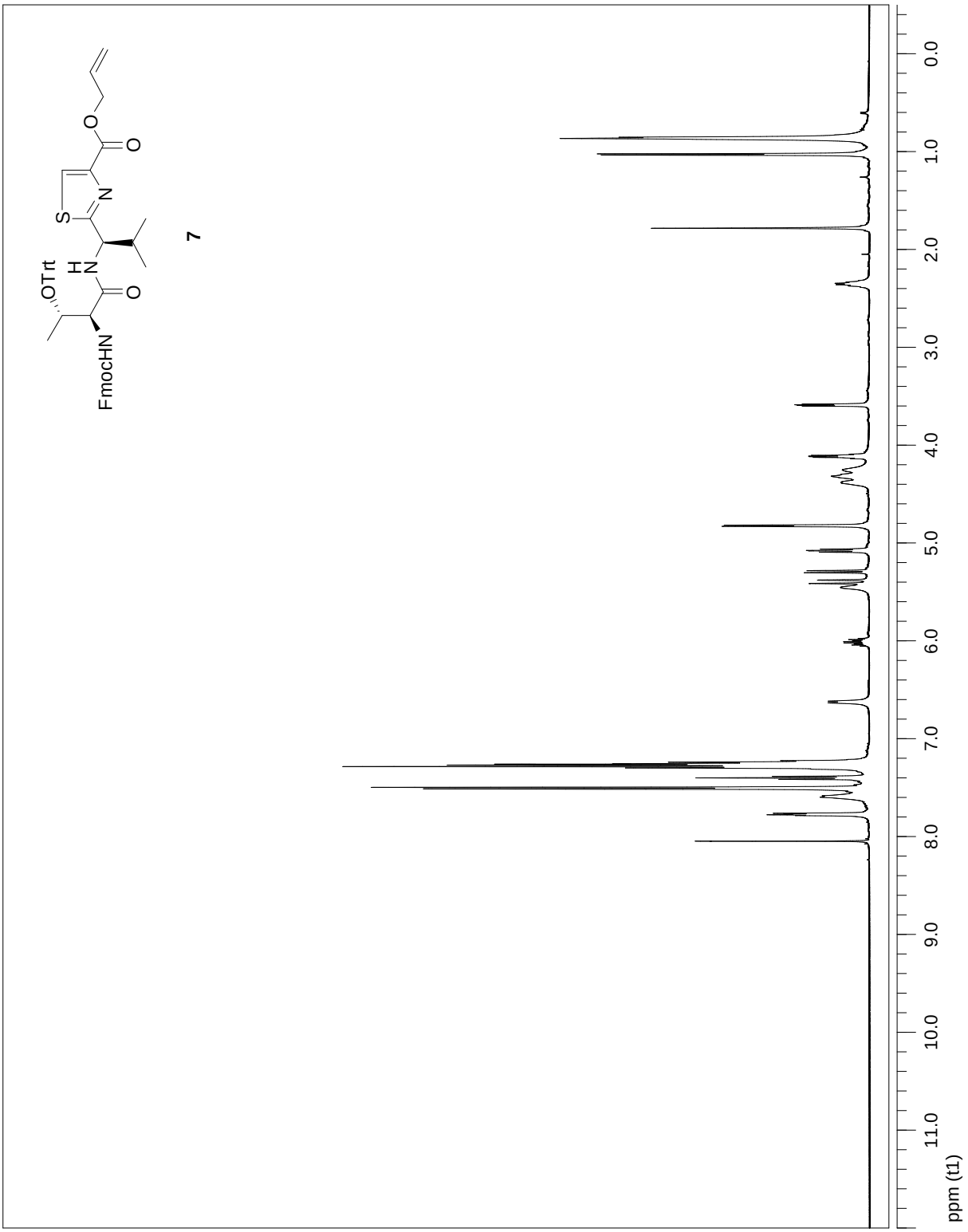
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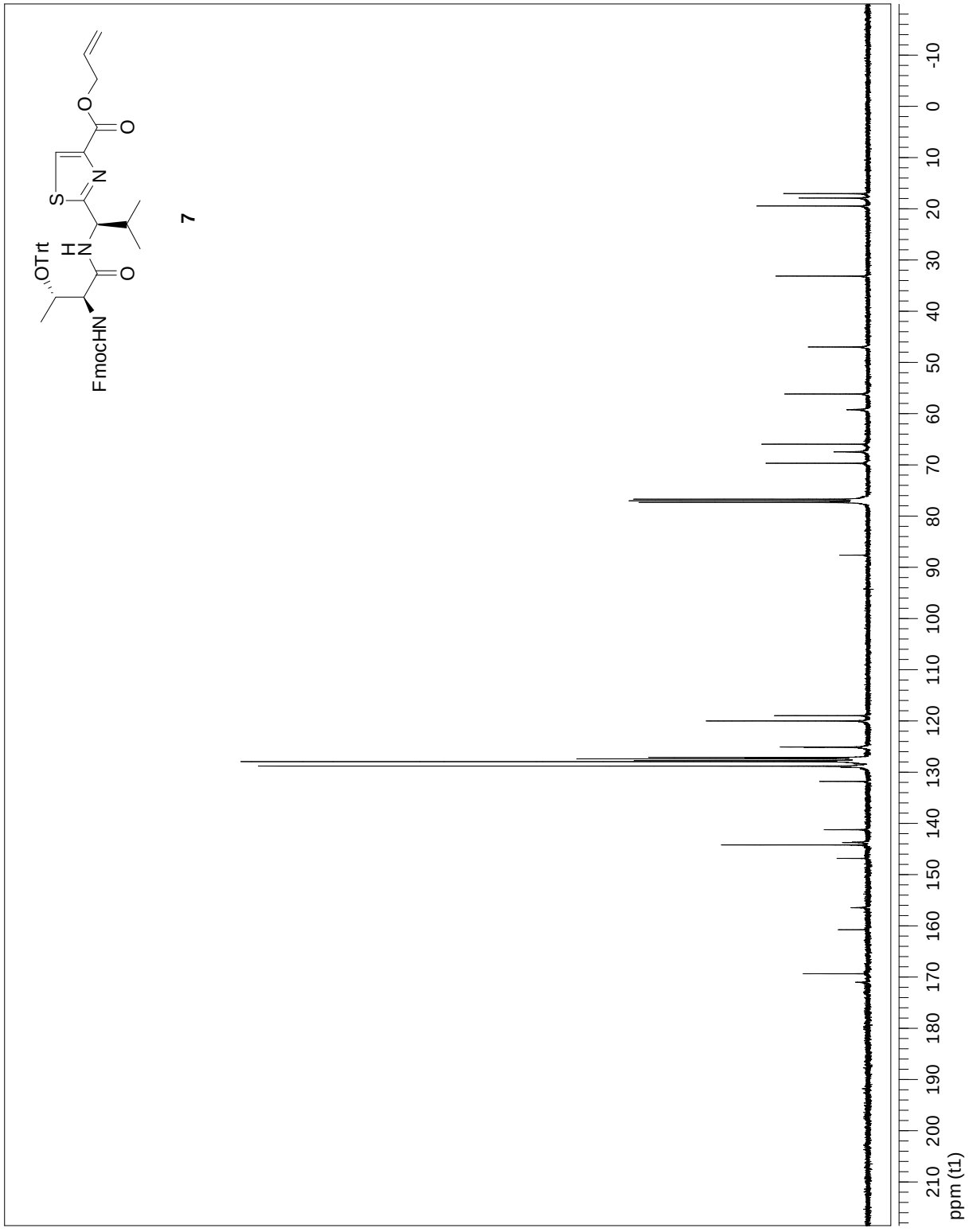
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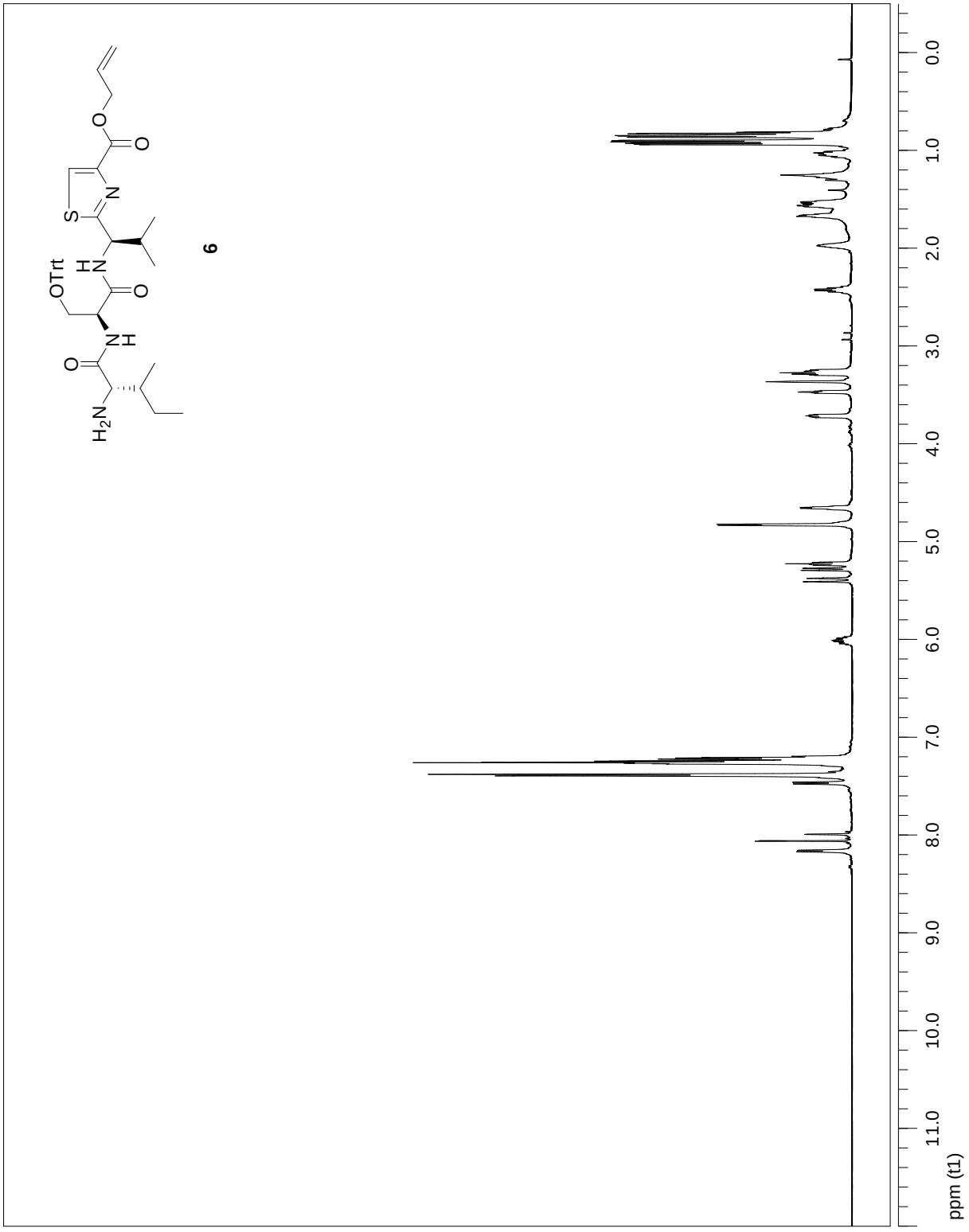
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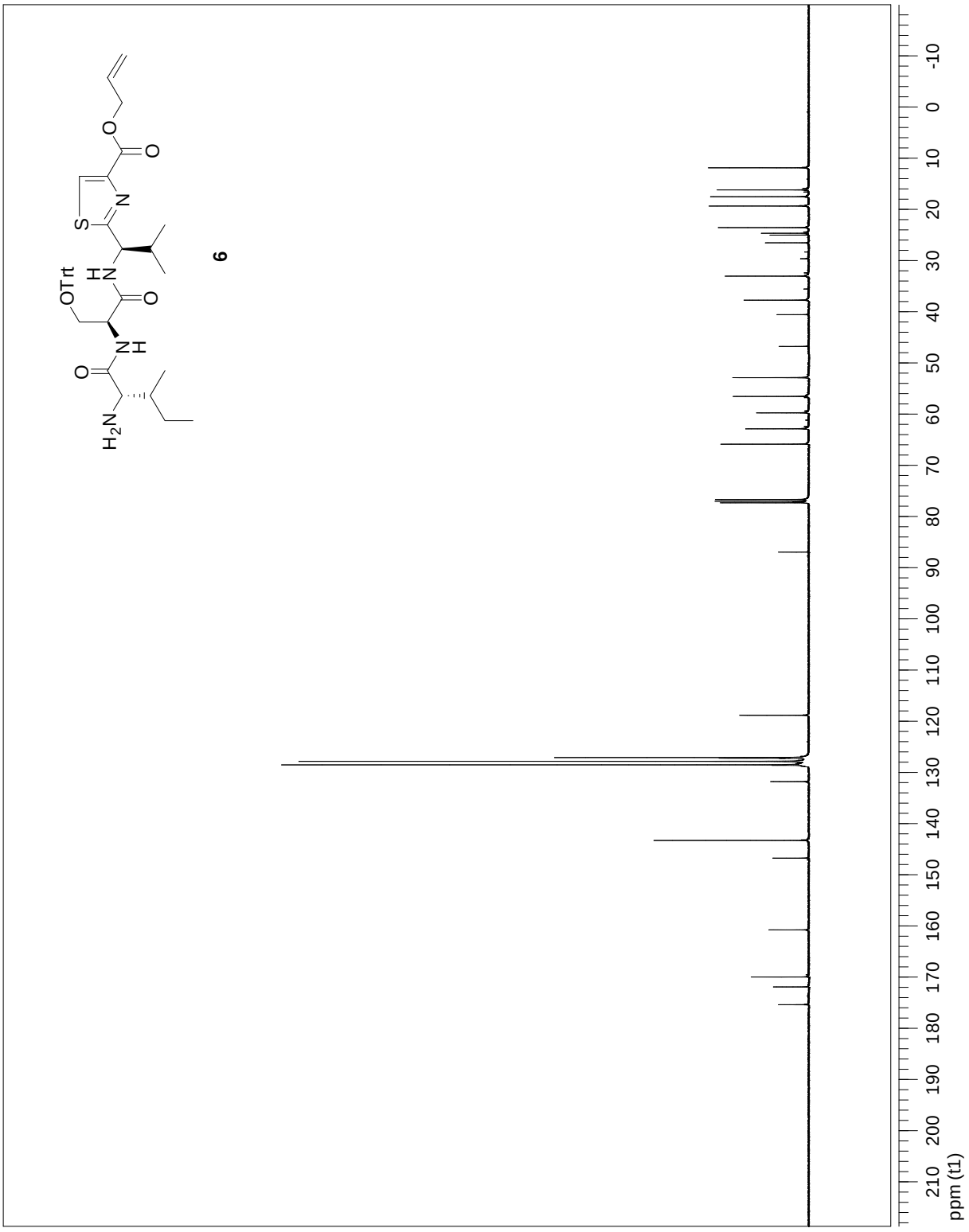
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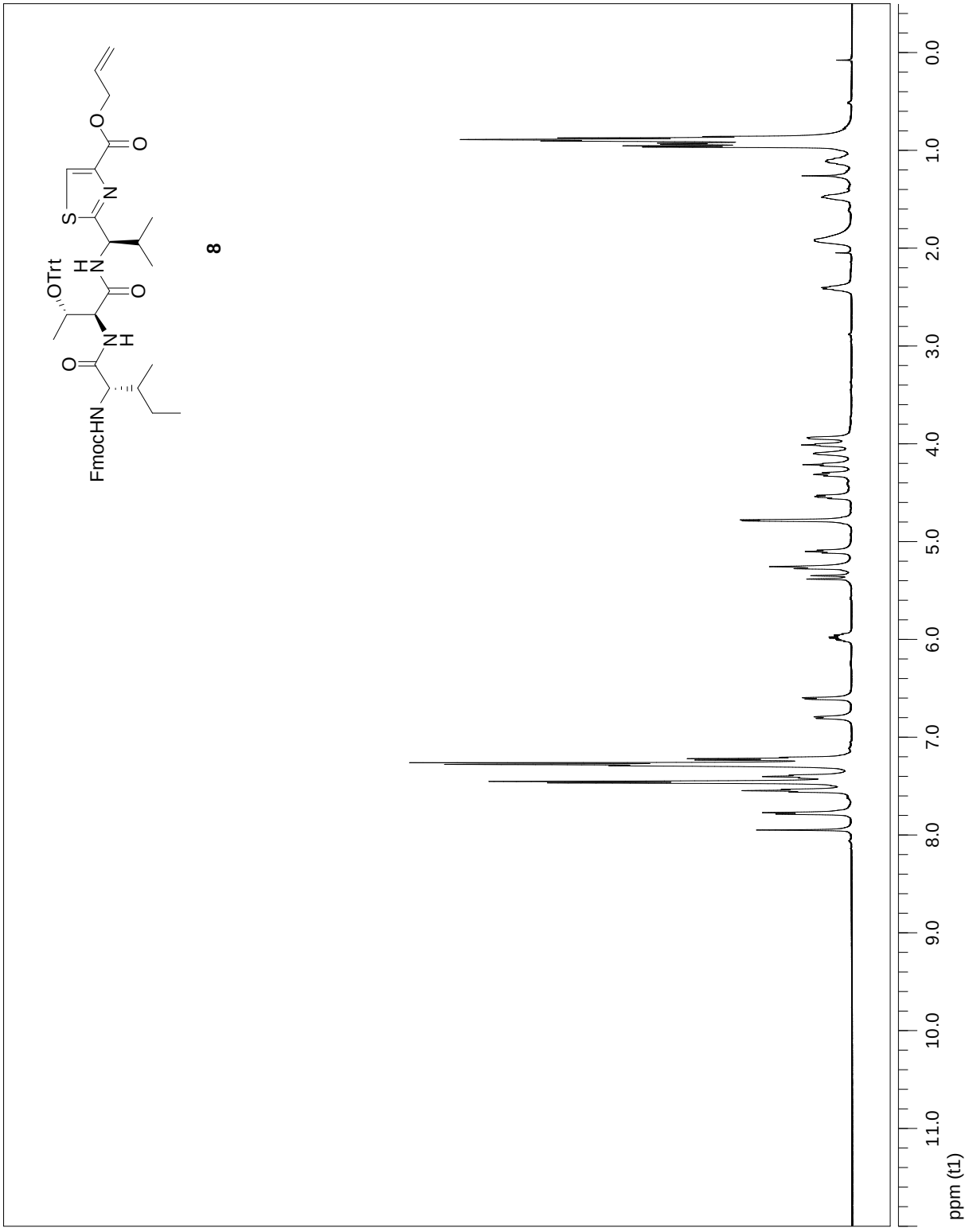
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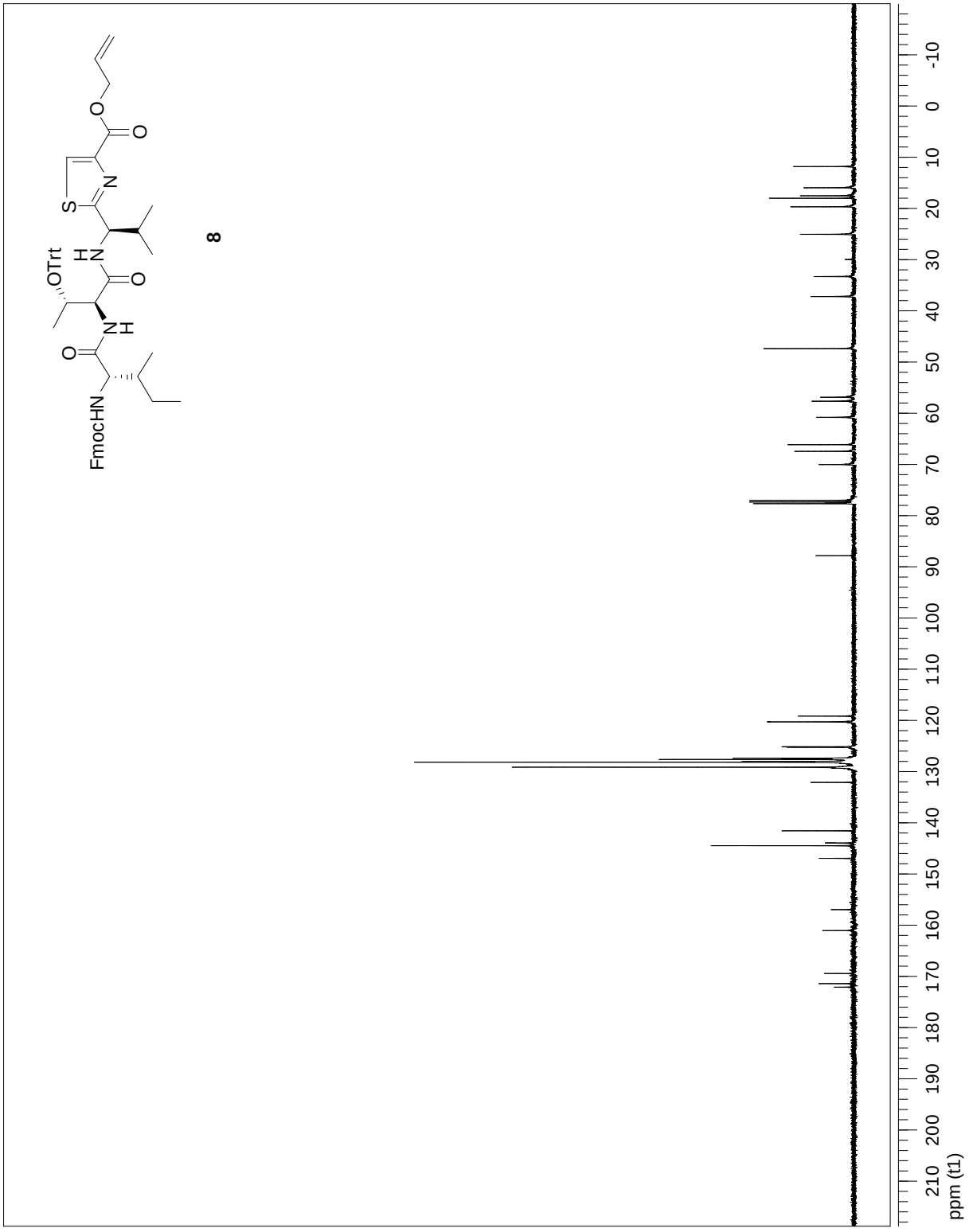
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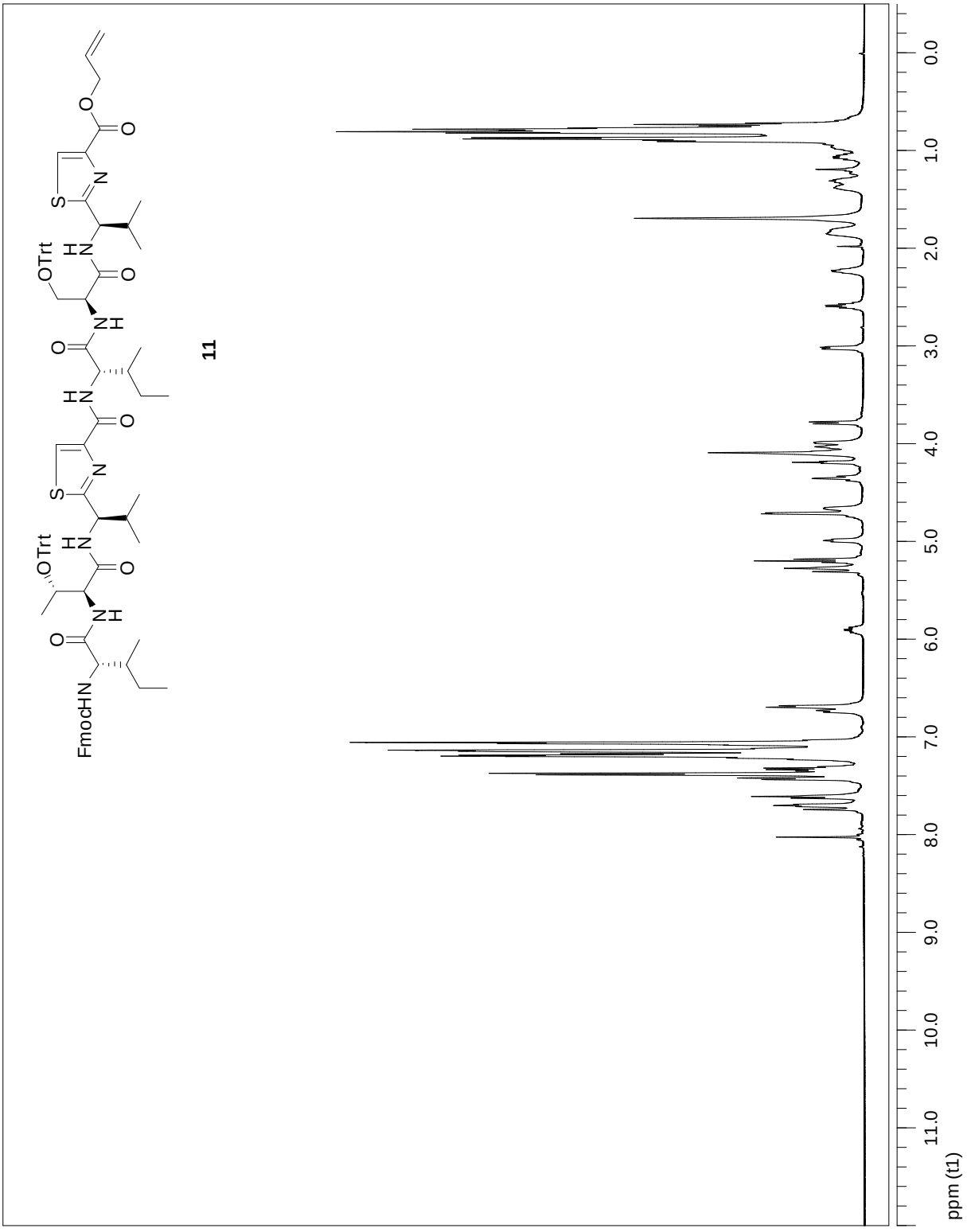
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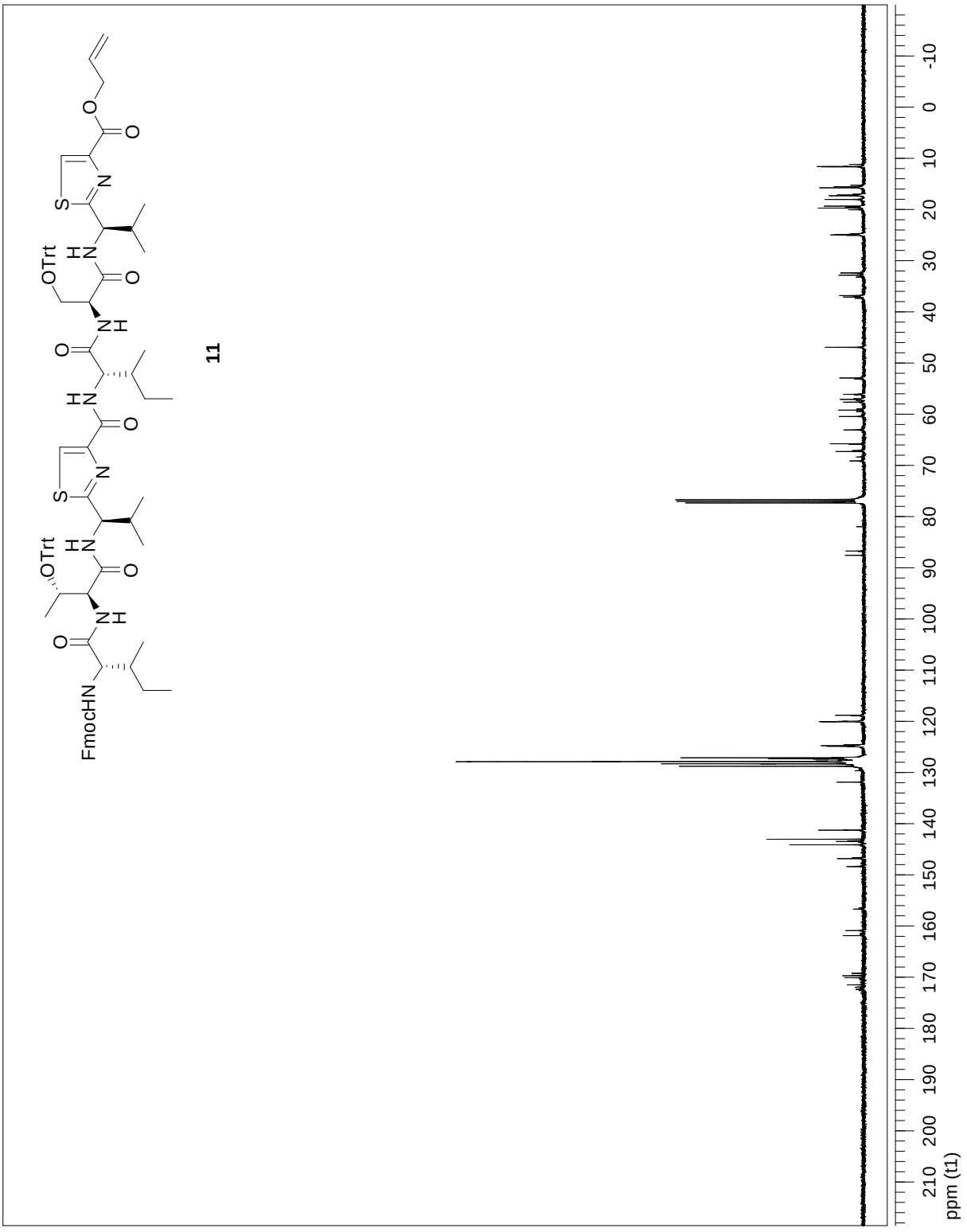

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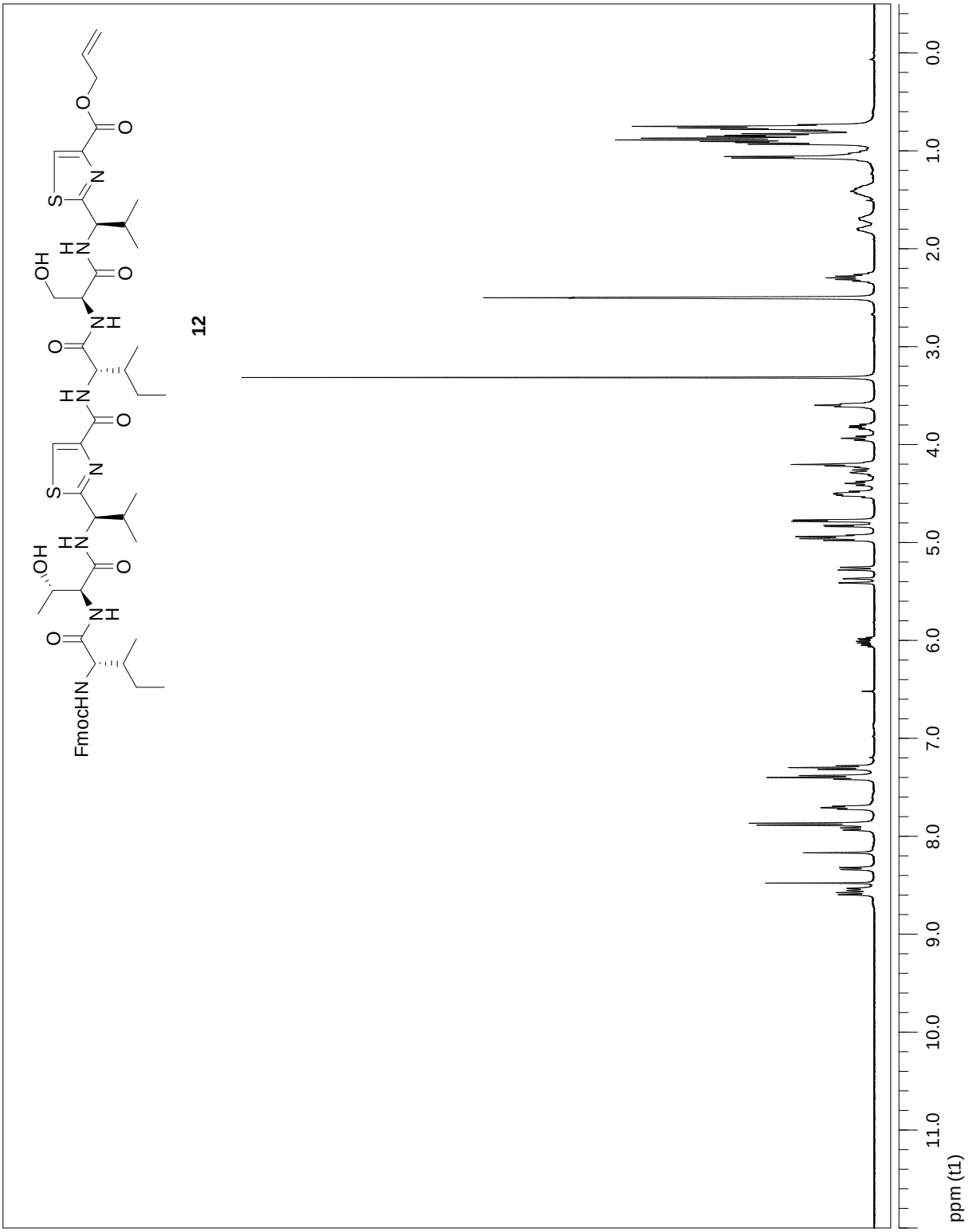
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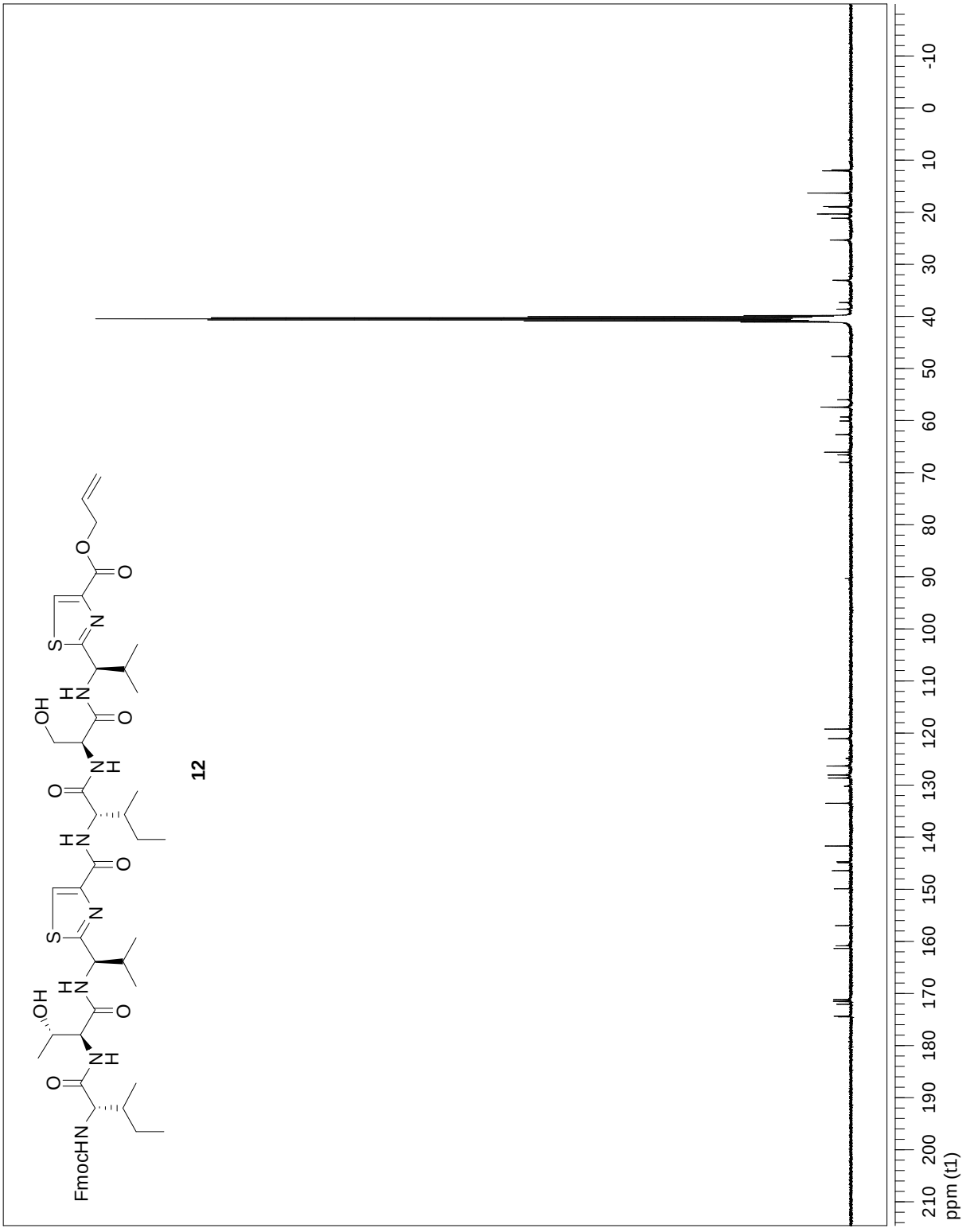
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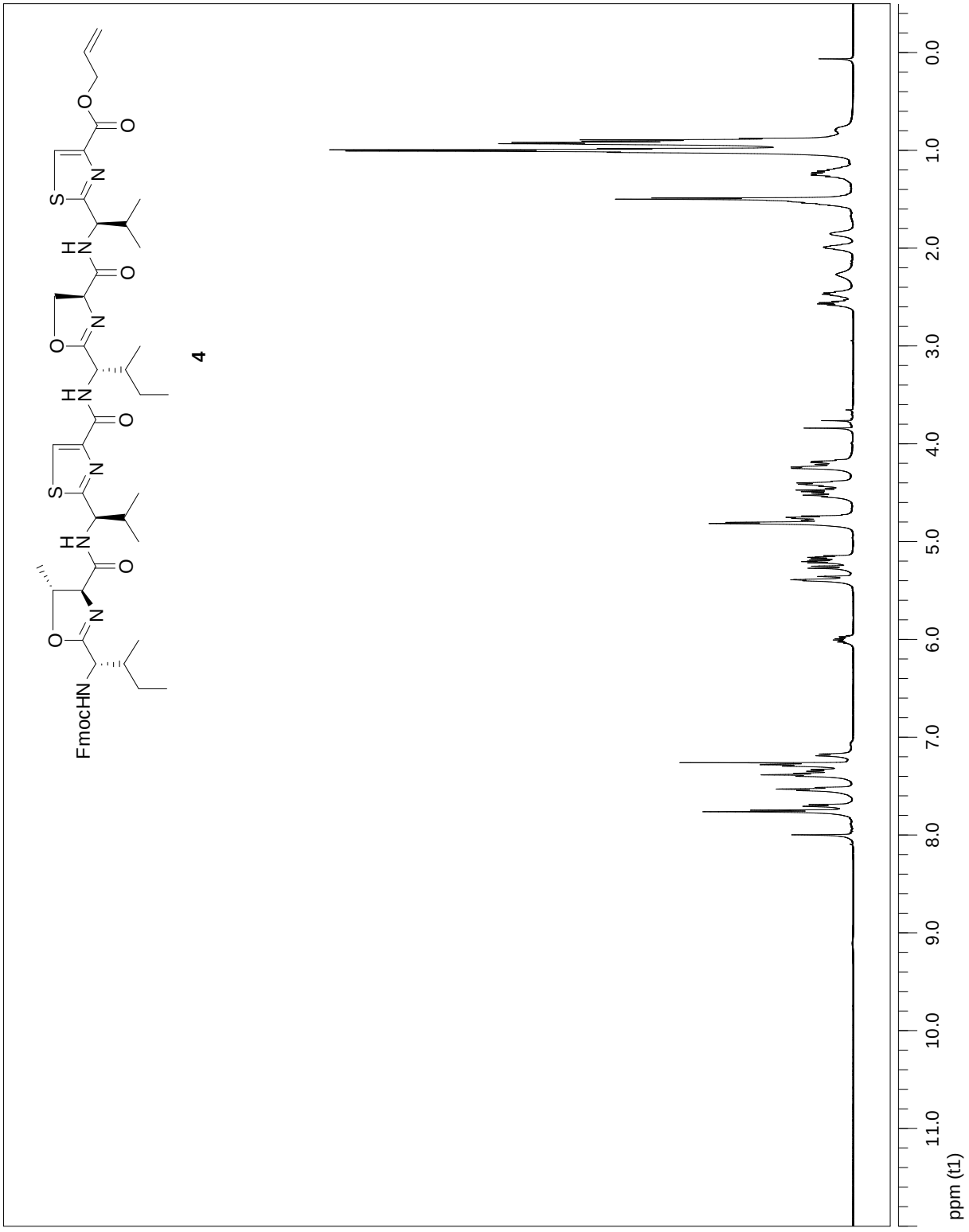
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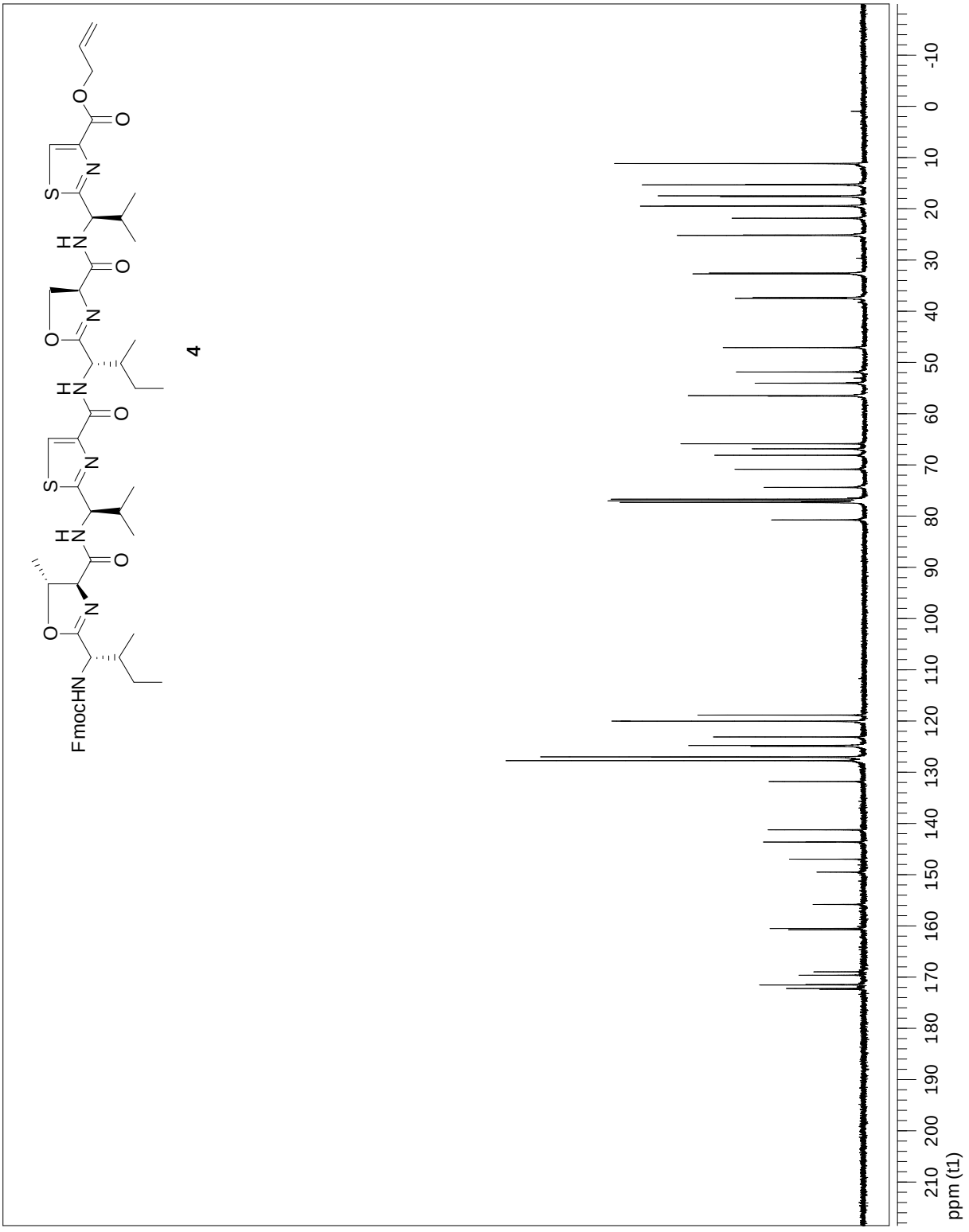
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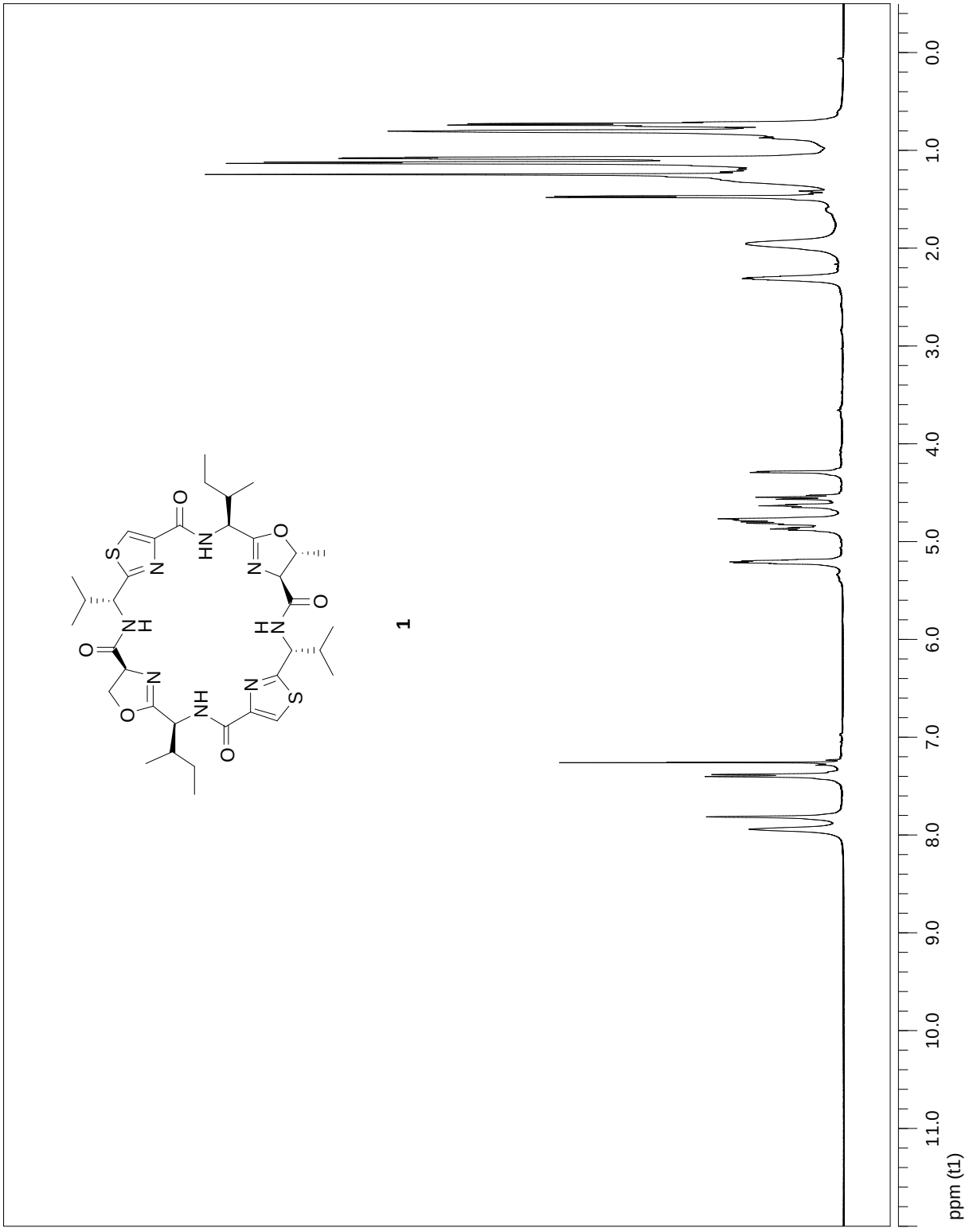
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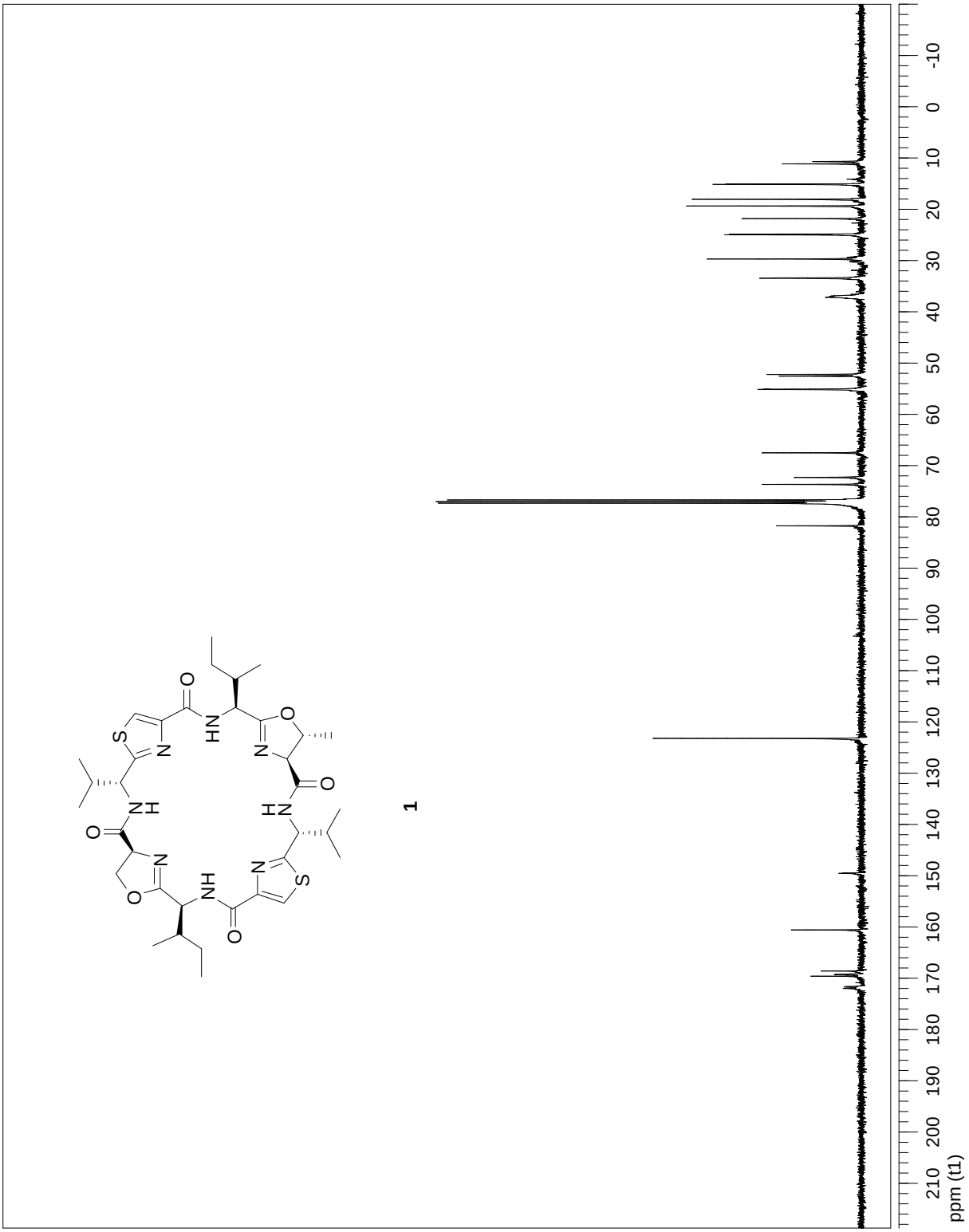
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