Supporting Information for

Synthesis of Amino Acid-Derived Cyclic Acyl Amidines for Use in β-Strand Peptidomimetics

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General methods. Reagents and solvents were obtained from commercial suppliers and were used as received unless otherwise noted. Dichloromethane, acetonitrile (MeCN), triethylamine (TEA), and diisopropylethylamine (DIEA) were distilled from calcium hydride. Tetrahydrofuran (THF) was distilled from sodium/benzophenone ketal. Solvents were removed from samples under reduced pressure using a rotary evaporator, followed by drying under high vacuum. Flash chromatography was performed using 60 Å pore, 230-400 mesh silica gel and the solvent system indicated. Analytical thin-layer chromatography (TLC) was performed using silica gel 60 F_{254} plates. Compounds were visualized by staining with potassium permanganate, iodine, ninhydrin (for amines), and/or by UV absorbance.

Analytical high pressure liquid chromatography (HPLC) analysis was performed using a Photodiode Array Detector with Controller and Pump. The analytical column was a 4.6 mm x

250 mm reversed-phase C18 column. Retention times were recorded using gradient method A (flow rate 1 mL/min). Chiral analytical HPLC was performed using the chiral normal phase column indicated. Preparative HPLC purification was performed using a 21.4 mm x 250 mm reversed-phase C18 column equipped with a guard column. For purification purposes, the gradient program used was Method B (flow rate 15 mL/min). Liquid Chromatography-Mass Spectrometry (LCMS) analysis was performed using a C18 reversed-phase column (2.1 mm x 50 mm). Gradient method C was used for analysis (flow rate 0.4 mL/min).

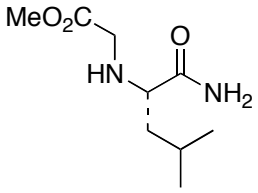
HPLC Method A		HPLC Method B		LCMS Method C	
Time	% CH_3CN (0.1% TFA)	Time	% CH_3CN	Time	% CH_3CN (0.1% TFA)
0	10	0	0	0	5
0-25	Ramp to 95	0-40	Ramp to 30	0-8	Ramp to 95
30	95	40-45	Ramp to 95	8-10	95
32	Ramp to 10	50	95	10-12	Ramp to 5
		55	Ramp to 0	12-14	5

Nuclear Magnetic Resonance (NMR) spectra were obtained using 400 or 500 MHz spectrometers with ^{13}C operating frequencies of 100 and 125 MHz. Spectral data are reported as chemical shifts (multiplicity, number of hydrogens, coupling constants in Hz). Chemical shifts were calibrated to TMS (0 ppm) or the residual solvent peak (1.94 for CHD_2CN, 2.50 for $(CHD_2)(CD_3)SO$, 3.31 for CHD_2OD, and 7.27 ppm for $CHCl_3$). Peaks for the major rotamer are reported, along with representative peaks of the minor rotamer. All ^{13}C NMR were proton decoupled and referenced to the solvent peak (1.24 for CD_3CN, 49.05 for CD_3OD, and 77.0 ppm for $CDCl_3$). TOCSY spectra were recorded with a mixing time of 100 ms. Concentrations of stock solutions of final products were determined by amino acid analysis (AAA).

Abbreviations used: Alloc, allyloxycarbonyl; DIEA, diisopropylethylamine; DME, ethylene glycol dimethyl ether; Fmoc, 9-fluorenylmethyloxycarbonyl; HATU, *N*-[(dimethylamino)-1*H*-1,2,3-triazolo-[4,5-b]pyridin-1-ylmethylene]-*N*-methylmethanaminium hexafluorophosphate *N*-oxide; MeCN, acetonitrile; TEA, triethylamine; TFA, trifluoroacetic acid.

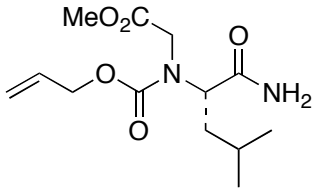
Synthesis of Thioamide 5

N-(2-Methoxy-2-oxoethyl)-L-leucine Amide.



To 2.0 g (12 mmol) of L-leucine amide hydrochloride (**4**) in 20 mL of dry THF at 0 °C was added 5.0 mL (29 mmol) of DIEA followed by slow addition of a solution of methyl bromoacetate (1.4 mL, 14 mmol) in dry THF (20 mL). The reaction solution was stirred under argon and allowed to warm up to room temperature over a 20-h period before it was diluted with 50 mL of saturated NH_4Cl. After concentration of the reaction mixture, the product was extracted with EtOAc (2x 50 mL). The organic layer was further washed with saturated $NaHCO_3$ (50 mL) and brine (50 mL), dried over Na_2SO_4, filtered, and concentrated to afford the product as a white solid (1.87 g, 77% crude yield); this material could be carried onto the next step without further purification. R_f = 0.26 ($KMnO_4$/basic, DCM/MeOH 95:5); retention time (analytical HPLC) = 13.1 min; IR (film) 1673, 1745, 2955 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.90 (d, 3, J = 4.7), 0.92 (d, 3, J = 4.7), 1.44 (m, 1, J = 5.5, 8.8, 14), 1.54 (m, 1, J = 4.8, 8.8, 14), 1.73 (m, 1), 2.1 (bs, 1), 3.08 (dd, 1, J = 4.8, 8.8), 3.29 (d, 1, J = 17.5), 3.43 (d, 1, J = 17.5), 3.68 (s, 3), 6.42 (bs, 1), 7.00 (bs, 1); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.8, 23.2, 24.9, 43.1, 49.2, 51.9, 61.2, 172.6, 178.1; MS (ES) m/z 203.1 ($M+H^+$), 158.1 ($M+H^+$-$CONH_2$); HRMS (FAB) m/z 203.1398 ($M+H^+$ $C_9H_{19}N_2O_3$ requires 203.1396).

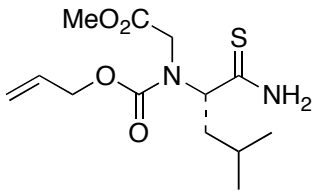
N-Allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-L-leucine Amide.



To 0.37 g (1.8 mmol) of N-(2-methoxy-2-oxoethyl)-L-leucine amide in 18 mL of dry DCM at 0 °C was added 2.0 mL (11.1 mmol) of DIEA followed by slow addition of allyl chloroformate (0.55 mL, 5.2 mmol). The reaction solution was stirred under argon and allowed to warm up to rt over a 20-h period before it was diluted with 50 mL of saturated NH_4Cl. After concentration of the reaction mixture, the product was extracted with EtOAc (3x 10 mL). The organic layer was further washed with saturated NH_4Cl (2x 15 mL), saturated $NaHCO_3$ (15 mL), and brine (15 mL), dried over Na_2SO_4, filtered, and concentrated. The crude product was purified by flash chromatography (EtOAc/hexanes 1:2 → 1:1 → 2:1) to afford the protected leucine amide as a yellow oil (0.41 g, 79% yield). R_f = 0.51 (ninhydrin, I_2,

EtOAc/hexanes 10:3); ^{1}H NMR (500 MHz, $CDCl_3$, rotamers) δ 0.92-0.99 (m, 6), 1.50-1.59 (m, 1), 1.59-1.66 (m, 1, J = 4.0), 1.79-1.85 (m, 0.6, J = 5.5), 1.90-1.96 (m, 0.5, J = 4.5), 3.74 (s, 1.6), 3.79 (s, 1.4), 3.95-4.06 (m, 2), 4.42 (m, 0.4), 4.59 (dt, 1, J = 1.5, 5.5), 4.63 (dd, 1, J = 4.2, 5.8), 4.71 (dd, 0.7, J = 6.0, 9.0), 5.19-5.35 (m, 2, J = 1.5) 5.84-5.90 (m, 1), 6.44 (bs, 0.6), 6.54 (bs, 0.4), 7.28 (bs, 0.6), 7.78 (bs, 0.4); ^{13}C NMR (125 MHz, $CDCl_3$, rotamers) δ 21.3/21.7, 23.0/23.2, 24.7/24.7, 37.5/38.2, 45.9/47.5, 52.4/52.7, 57.7/59.3, 66.7/67.0, 117.6/118.4, 131.8/132.1, 155.8/155.8, 171.0/171.8, 173.2/173.6; MS (ES) m/z 309.2 ($M+Na^+$), 242.1 ($M+H^+$-$CONH_2$); HRMS (FAB) m/z 287.1611 ($M+H^+$ $C_{13}H_{23}N_2O_5$ requires 287.1607).

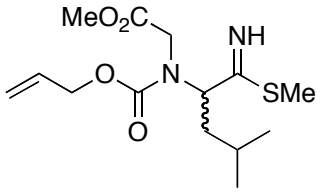
N-Allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-L-leucine Thioamide (5).



To 1.0 g (3.5 mmol) of N-allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-L-leucine amide in 50 mL of dry THF was added 0.99 g (2.4 mmol) of Lawesson's reagent, which turns the solution faint yellow and opaque. The reaction solution was stirred under argon at rt, and the reaction was stopped at 1.5 h when no more starting material was observed by TLC. After concentration of the reaction mixture, the crude product was redissolved in EtOAc and washed with saturated $NaHCO_3$ (2x 50 mL). The basic aqueous layer was acidified by addition of solid $KHSO_4$ and extracted with EtOAc (2 x 20 mL). The combined organic layers were further washed with brine (50 mL), dried over Na_2SO_4, filtered, and concentrated. The crude product was purified by flash chromatography (EtOAc/hexanes 1:2 → 1:1) to afford thioamide **5** as a white solid (0.82 g, 78%). R_f = 0.55 (UV, EtOAc/hexanes 1:1); retention time (analytical HPLC): 16.2 min; retention time (chiral HPLC, OD column, 85/15 hexanes/isopropanol): 7.1 min; $[\alpha]^{23}_D$ = -76.0 (*c* = 0.745, in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, rotamers) δ 0.94-0.96 (m, 6), 1.46-1.56 (m, 1), 1.64-1.72 (m, 1), 2.22 (m, 0.5, J = 6.0, 8.6, 14.5), 2.47 (m, 0.5, J = 4.0, 10.7, 14.8), 3.75 (s, 1.5), 3.82 (s, 1.5), 3.83 (m, 1), 4.09 (t, 1, J = 18.0), 4.61 (dd, 1, J = 1.4, 5.4), 4.65 (t, 1, J = 5.4), 4.86 (dd, 0.4, J = 3.6, 11.1), 4.93 (dd, 0.6, J = 6.1, 9.2), 5.21-5.35 (m, 2, J = 1.3), 5.82-5.92 (m, 1), 7.60 (bs, 0.5), 7.68 (bs, 0.5), 8.66 (bs, 0.5), 9.53 (bs, 0.5); ^{13}C NMR (125 MHz, $CDCl_3$, rotamers) δ 20.9/21.8, 22.9/23.4, 25.0/25.2, 40.3/41.4, 45.6/46.3, 52.5/53.0, 62.8/64.9, 67.0/67.3, 117.8/118.8, 131.6/132.0, 156.0/156.4, 171.1/172.5, 207.0/207.7; MS (ES) m/z 325.2 ($M+Na^+$), 303.1 ($M+H^+$); HRMS (FAB) m/z 303.1373 ($M+H^+$ $C_{13}H_{23}N_2O_4S$ requires 303.1378).

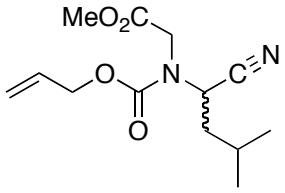
Additional Characterized Compounds

N-Allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-D,L-leucyl Methyl Thioimidate (9).



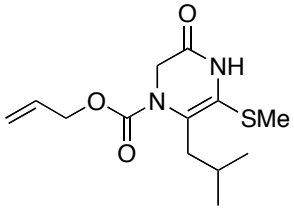
The crude thioimidate salt **8** can be redissolved in EtOAc and the organic layer washed with $NaHCO_3$ (1x 10 mL) to neutralize the salt, which can be monitored as the organic layer turns from dark yellow to clear or light yellow. The organic layer then is washed once with brine to give racemic **9** (84% yield). r_t(analytical HPLC) = 18.5 min; r_t(chiral HPLC, OD column, 85:15 hexanes/isopropanol, both enantiomers) = 8.2 min, 9.3 min; ^{1}H NMR (500 MHz, $CDCl_3$, rotamers) δ 0.87-0.92 (m, 6), 1.55-1.74 (m, 3), 2.21 (s, 1.2), 2.22 (s, 1.8), 3.64 (s, 1.8), 3.66 (s, 1.2), 3.81 (d, 0.4, J = 17.5), 3.85 (d, 0.6, J = 18.0), 4.00 (d, 1, J = 18.0), 4.56 (q, 0.6, J = 2.0), 4.57 (q, 0.6, J = 2.0), 4.61 (q, 0.4, J = 2.0), 4.62 (q, 0.4, J = 2.0), 4.83 (dd, 0.4, J = 5.0, 9.0), 4.98 (dd, 0.6, J = 6.0, 9.0), 5.12 (q, 0.25, J = 1.5), 5.14 (q, 0.25, J = 1.5), 5.17 (q, 0.2, J = 1.5), 5.19 (q, 0.5, J = 1.5), 5.23 (q, 0.3, J = 1.5), 5.28 (q, 0.2, J = 1.5), 5.32 (q, 0.2, J = 1.5), 5.82 (m, 0.6, J = 5.5, 10.5), 5.90 (m, 0.4, J = 5.5, 10.5), 9.24 (bs, 1); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.8/22.0, 22.8/22.9, 24.3/24.5, 38.8, 39.0, 51.9, 66.5/66.6, 117.1, 132.3/132.4, 155.7/155.8, 169.8/170.0; MS (ES) *m/z* 317.3 ($M+H^+$).

Allyl N-(2-Methoxy-2-oxoethyl)-N-(1(*S*)-cyano-3-methylbutyl) Carbamate (10).



Major side product from treatment of thioimidate **9** with base such as DIEA. R_f = (UV, EtOAc/hexanes 1:1); IR (film) 1716.2, 1757.9, 2242.3 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$, rotamers) δ 0.96 (d, 3, J = 6.5), 0.98 (d, 3, J = 6.5), 1.63-1.71 (m, 2), 1.79 (m, 1, J = 6.5), 3.76 (s, 3), 3.97 (d, 1, J = 18.0), 4.11 (d, 1, J = 18.0), 4.14 (d, 0.1, J = 16.5), 4.61 (d, 1.2, J = 5.5), 4.64-4.68 (m, 0.8), 5.03 (t, 0.3, J = 7.5), 5.21-5.36 (m, 2.7), 5.82-5.95 (m, 1); ^{13}C NMR (125 MHz, $CDCl_3$, rotamers) δ 21.9, 22.4, 24.8, 40.7/41.2, 45.8/46.4, 46.9/47.0, 52.6, 67.4/67.6, 117.5/117.7, 118.2/119.0, 131.8/132.0, 154.4/155.0, 169.3; MS (FAB) *m/z* 269 ($M+H^+$), 242 ($M+H^+$-CN).

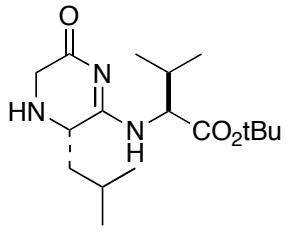
Allyl 3,4-Dihydro- 6-isobutyl-5-methylthio-3-oxopyrazine-1(2*H*)-carboxylate (12).



Side product from alkylation of thioimide **7**. R_f = 0.39 (UV, EtOAc/hexanes 1:1); r_t (analytical HPLC) = 15.7 min; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.93 (t, 6, J = 6.8), 1.72 (m, 1, J = 6.8), 2.29 (s, 3), 2.78 (d, 2, J = 6.4), 4.22 (s, 2), 4.64 (d, 2, J = 6.0), 5.27 (dd, 1, J = 1.2, 10.4),

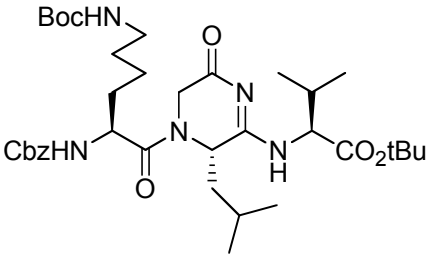
5.34 (dd, 1, J = 1.6, 17.2), 5.93 (m, 1), 7.57 (s, 1); MS (ES) *m/z* 285.1 ($M+H^+$), 244.1 ($M+H^+$-allyl)

4-((1(*S*)-(*t*-Butoxycarbonyl)-5,6-dihydro-2-methylpropyl)amino)-4,5-dihydro-5(*S*)-isobutyl-2(3*H*)-pyrazinone (@^{Leu}-Val *t*-Butyl Ester, 13).

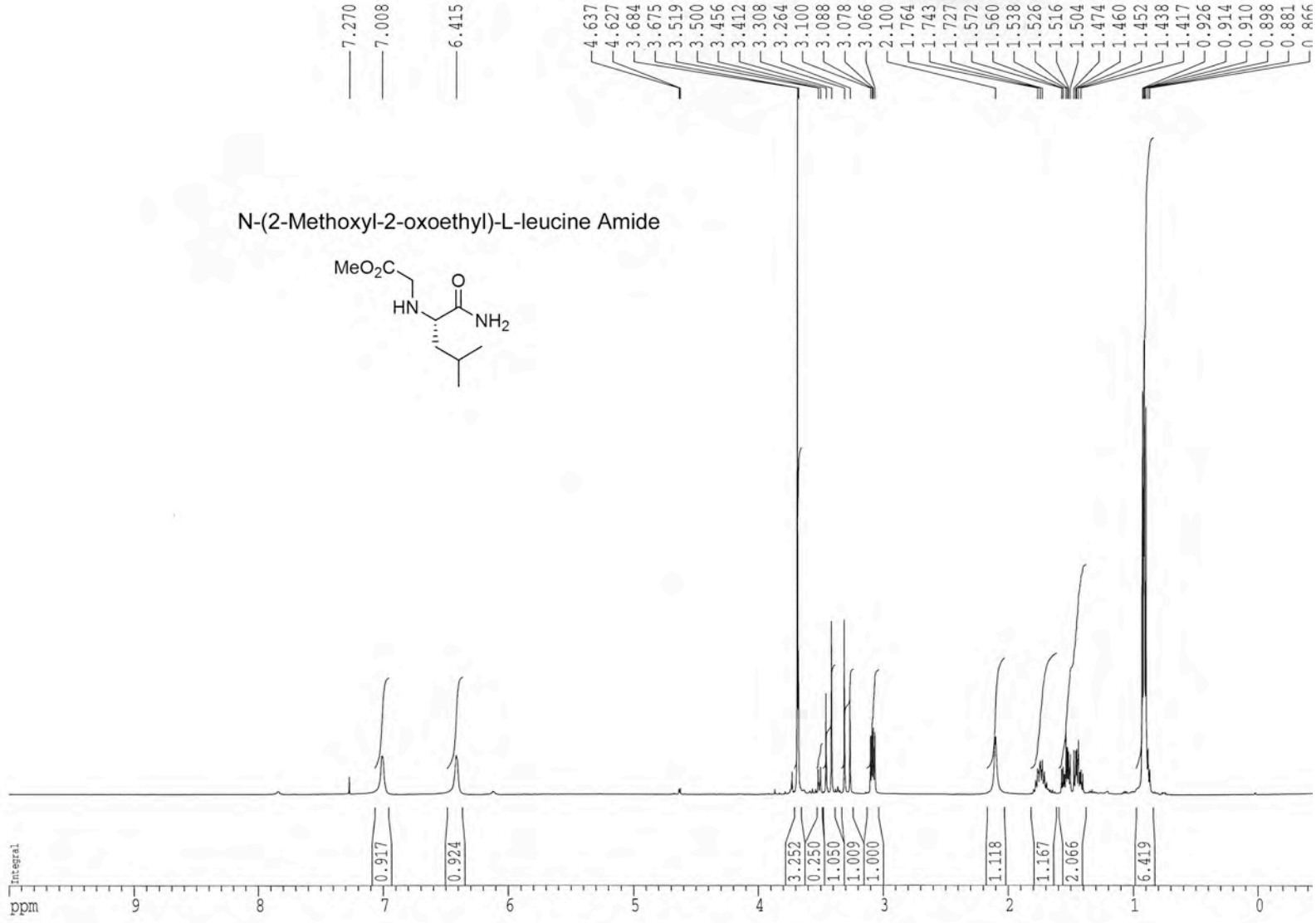


To 0.87 g (2.1 mmol) of the **3** in 10 mL of dry THF was added 10 mL of diethylamine (97 mmol), followed by $Pd(PPh_3)_4$ (0.48 g, 0.4 mmol). The reaction solution was stirred under argon at rt for 3 h. After concentration of the mixture, the crude product was purified by flash chromatography to separate the diastereomers (EtOH/EtOAc/TEA 1:99:0.5◊ 2:98:0.5◊ 3:97:0.5). The overall yield of the reaction was 69%. R_f = 0.32 (UV, EtOH/EtOAc/TEA 10:90:1); ^{1}H NMR (400 MHz, CD_3OD) δ 0.98-1.04 (m, 12), 1.39 (m, 1, J = 5.0, 9.6, 14.3), 1.49 (s, 9), 1.72 (m, 1, J = 4.6, 10.7, 15.3), 1.70-1.80 (m, 1), 2.12 (m, 1, J = 6.6), 3.36 (d,1, J = 18.3), 3.44 (d, 1, J = 18.3), 3.69 (dd, 1, J = 5.0, 10.8), 4.65 (d, 1, J = 5.9); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.1, 18.4, 21.2, 23.2, 24.5, 27.8, 28.0, 31.3, 39.0, 45.2, 51.1, 57.4, 82.6, 170.5, 174.5, 180.1; MS (ES) *m/z* 326.3 ($M+H^+$), 270.3 ($M+H^+$-tBu).

Cbz-Lys(Boc)-@^{Leu}-Val *t*-Butyl Ester (14).



To 74.5 mg (0.22 mmol) of **13** in 10 mL of dry DCM and 1 mL of dry DMF was added 60 μL (0.34 mmol) of DIEA, 0.13 g (0.34 mmol) of Cbz-Lys(Boc), and 0.13 g (0.34 mmol) of HATU. The reaction mixture was stirred under nitrogen at rt for 20 h. After the solution was concentrated, the crude product was purified by flash chromatography (EtOAc/hexanes 1:2 → 1:1 → 2:1) to afford **13** (113 mg, 72% yield). R_f = 0.18 (UV, EtOAc/hexanes 1:1). Retention time (analytical HPLC) = 26.8 min; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.79-1.05 (m, 12), 1.30-1.64 (m, 9), 1.43 (s, 18), 2.17 (m, 1, J = 6.5), 2.95-3.15 (m, 2), 4.05-4.07 (m, 0.2), 4.15 (d, 0.8, J = 18.0), 4.29 (d, 0.7, J = 18.0), 4.53-4.63 (m, 1), 4.65-4.78 (m, 0.8), 4.88-5.00 (m, 1.3), 5.06 (d, 1, J = 12.0), 5.12 (d, 1, J = 12.0), 5.68-5.83 (m, 1), 6.61-6.71 (m, 1), 7.24-7.39 (m, 5), 8.10-8.30 (m, 1); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.51, 17.90, 18.46, 18.91, 22.13, 22.54, 22.83, 24.72, 27.55, 27.70, 27.83, 28.22, 29.40, 30.42, 31.80, 31.90, 39.47, 39.91, 45.42, 48.52, 51.13, 57.91, 64.14, 66.61, 82.95, 127.71, 127.86, 128.22, 136.17, 155.61, 156.29, 170.35, 171.84, 172.09, 175.96; MS (ES) m/z 688.3 ($M+H^+$), 632.2 ($M+H^+$-tBu); HRMS (FAB) m/z 688.4301 ($M+H^+$ $C_{36}H_{58}N_5O_8$ requires 688.4285).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N-(2-Methoxyl-2-oxoethyl)-L-leucine Amide
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Integral
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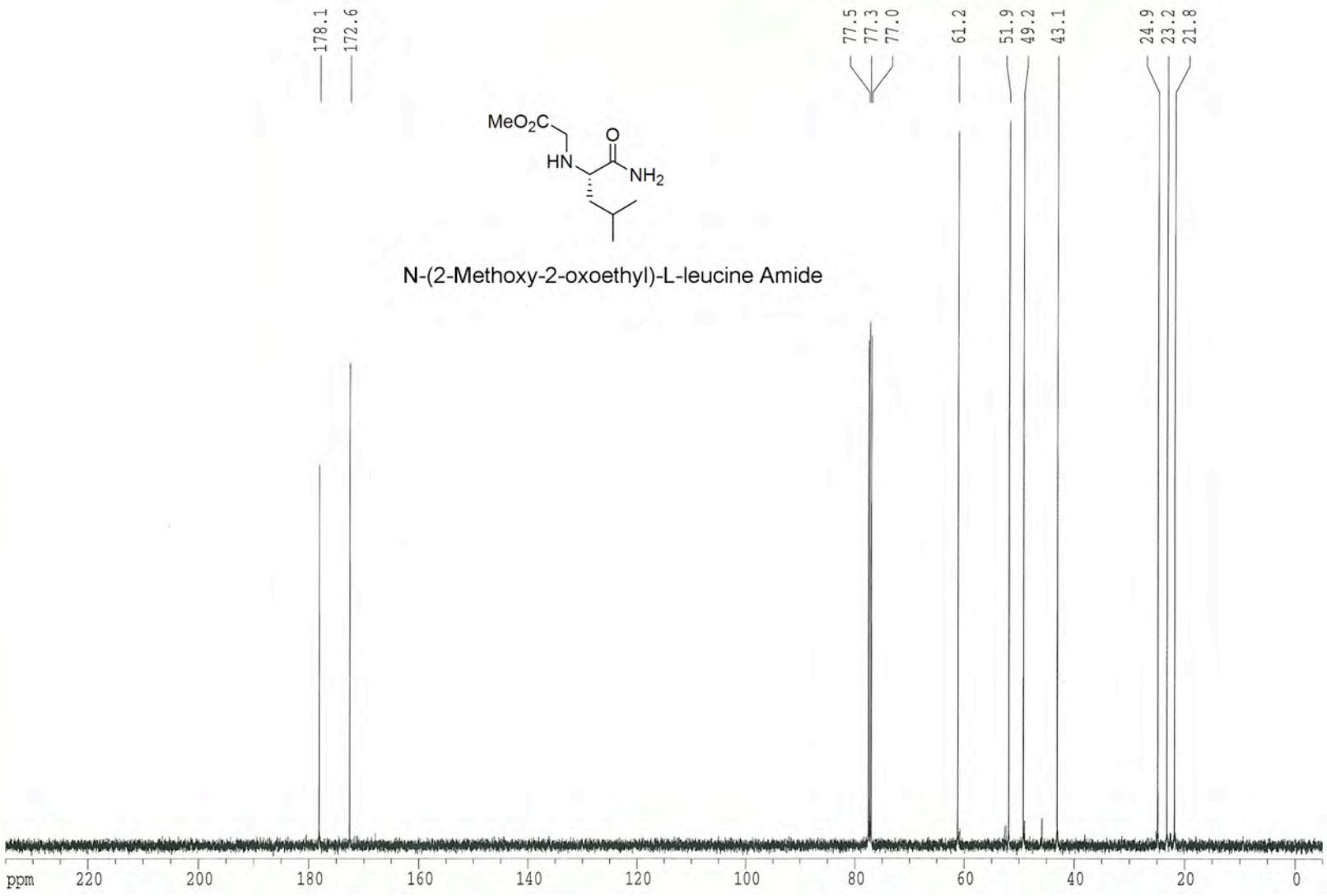
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O
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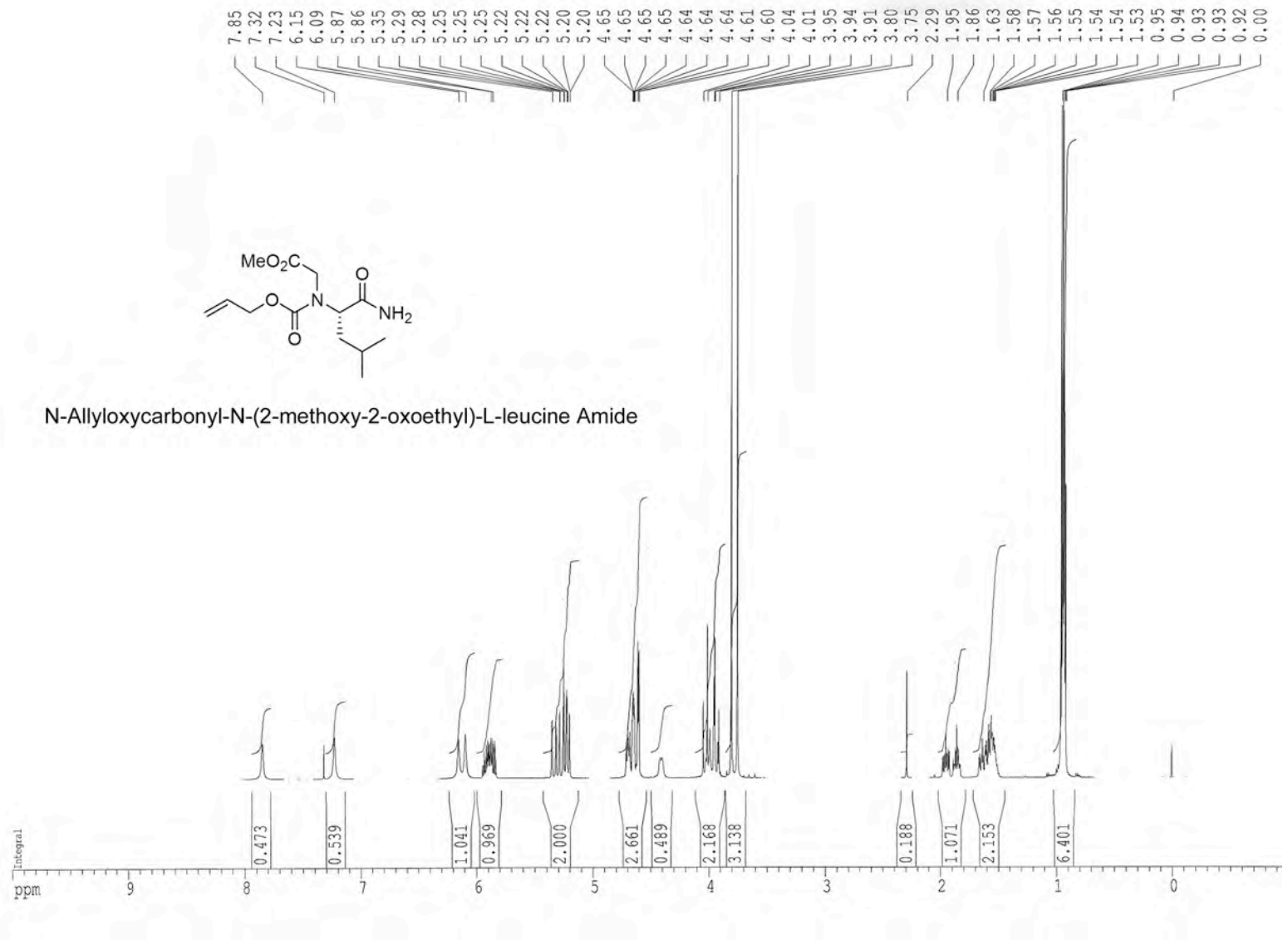
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2.29
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1.54
1.54
1.53
0.95
0.94
0.93
0.93
0.92
0.00
MeO2C
O
N
O
O
O
NH2
N-Allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-L-leucine Amide
Integral
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0.539
1.041
0.969
2.000
2.661
0.489
2.168
3.138
0.188
1.071
2.153
6.401
ppm
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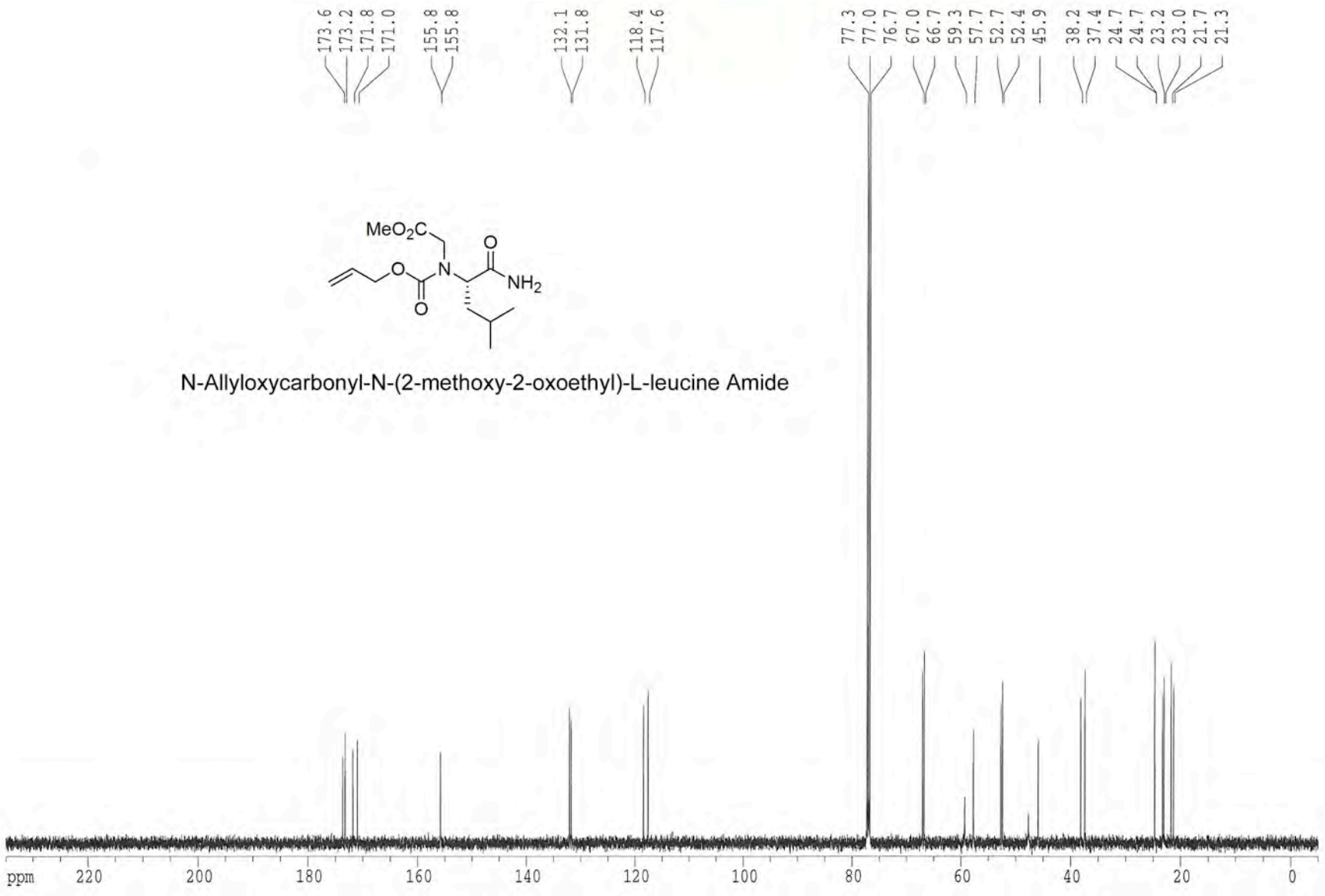
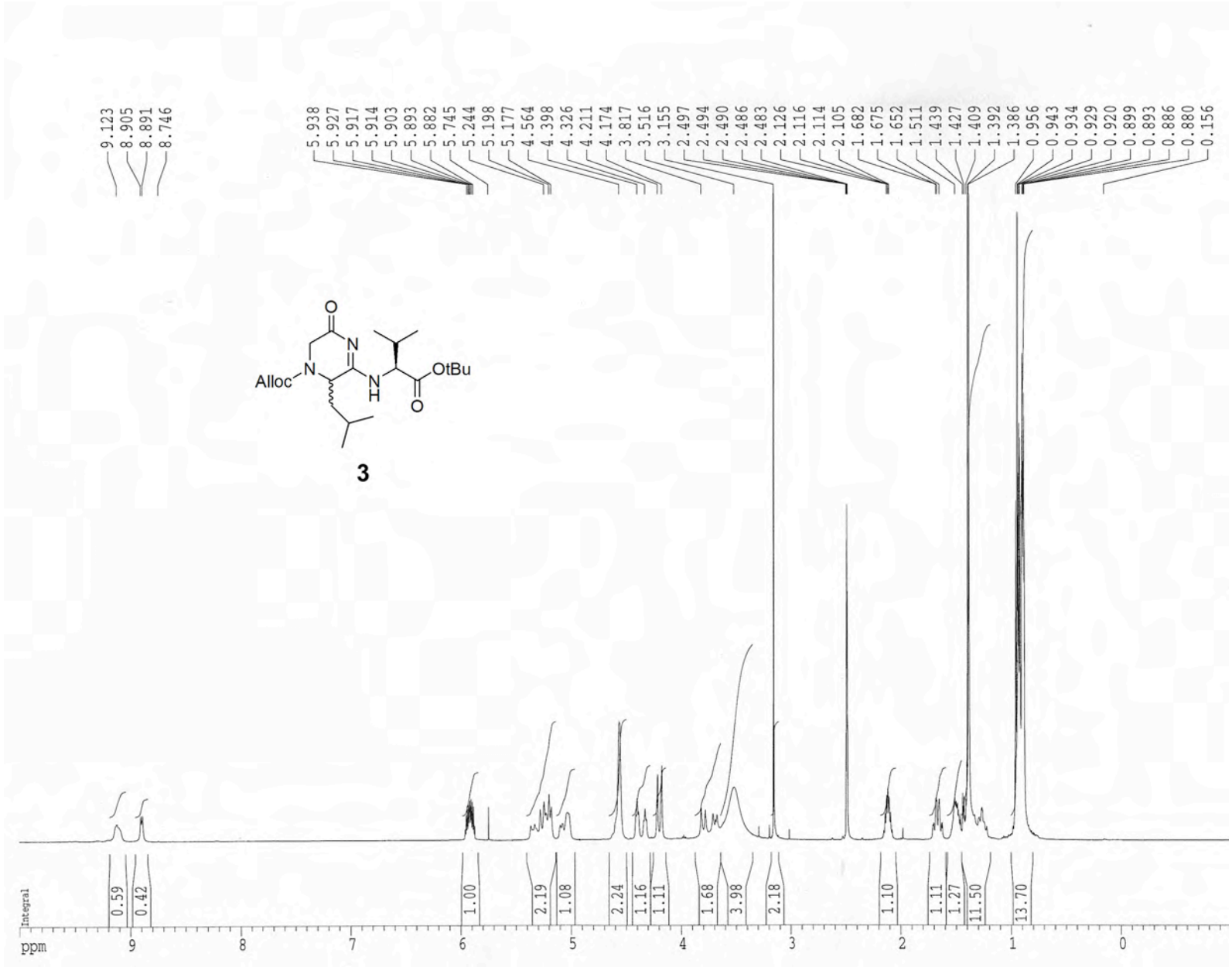
8
7
6
5
4
3
2
1
0



N-Allyloxycarbonyl-N-(2-methoxy-2-oxoethyl)-L-leucine Amide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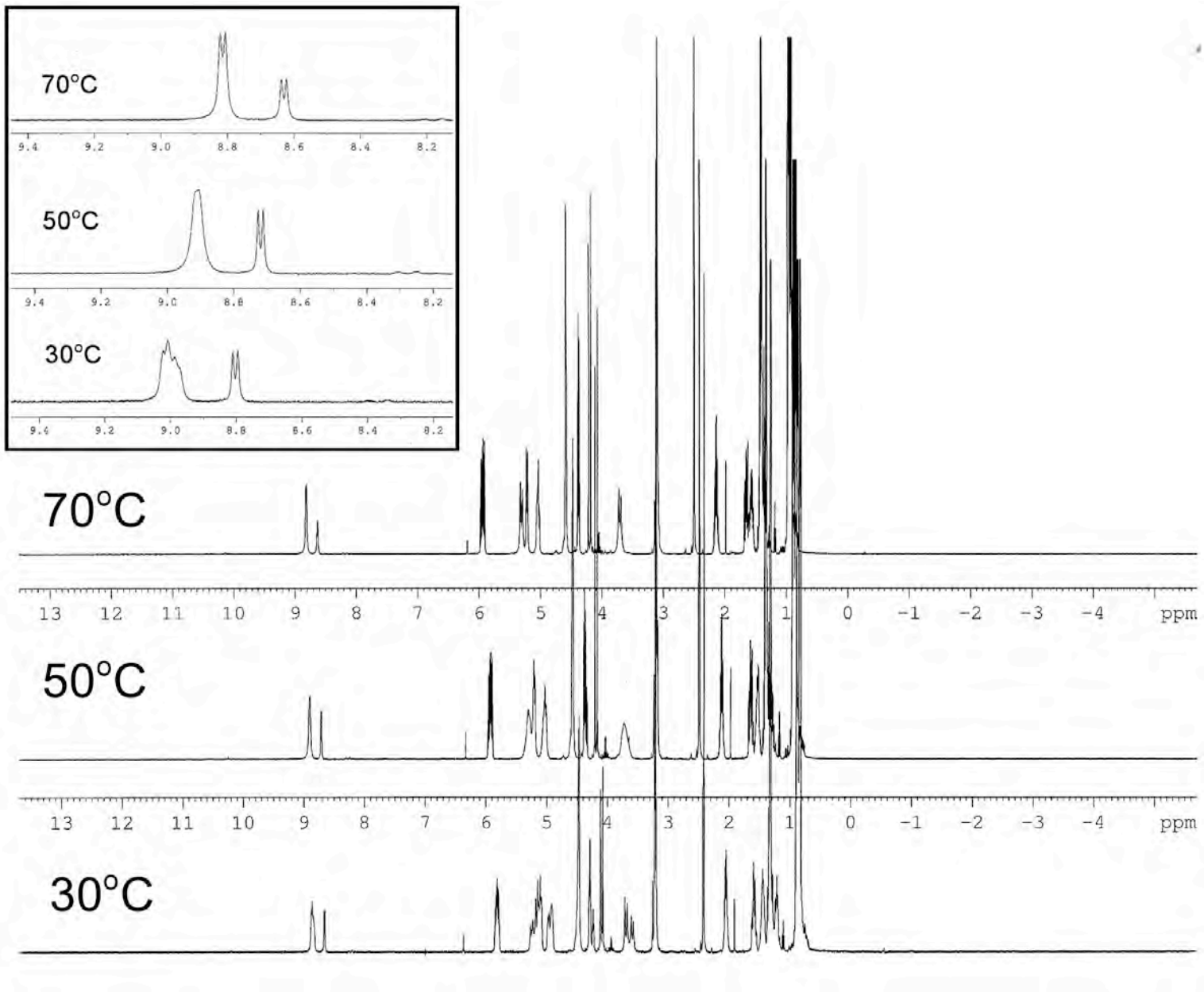
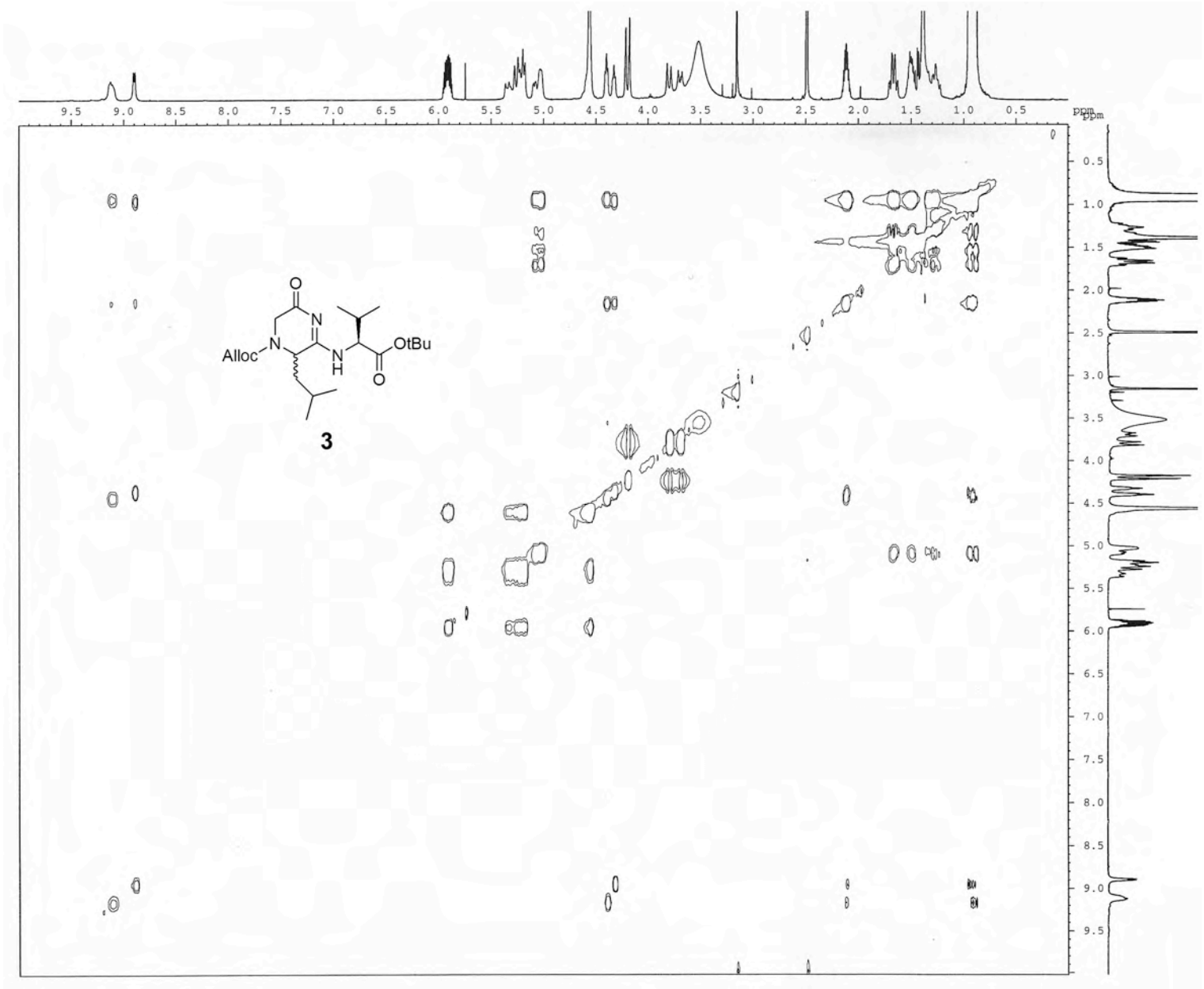
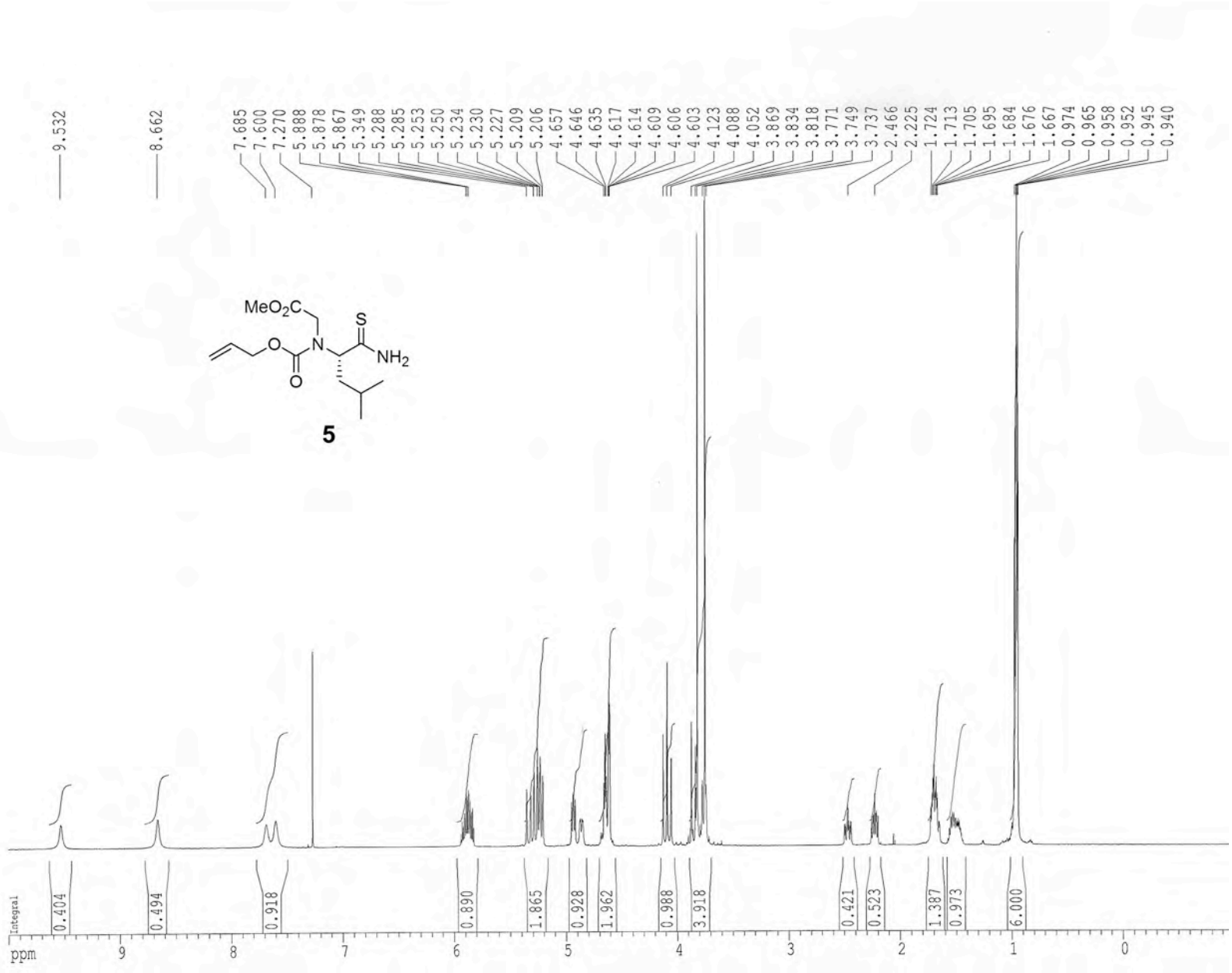
8.905
8.891
8.746
5.938
5.927
5.917
5.914
5.903
5.893
5.882
5.745
5.244
5.198
5.177
4.564
4.398
4.326
4.211
4.174
3.817
3.516
3.155
2.497
2.494
2.490
2.486
2.483
2.126
2.116
2.114
2.105
1.682
1.675
1.652
1.511
1.439
1.427
1.409
1.392
1.386
0.956
0.943
0.934
0.929
0.920
0.899
0.893
0.886
0.880
0.156
Alloc
OtBu
3
Integral
0.59
0.42
1.00
2.19
1.08
2.24
1.16
1.11
1.68
3.98
2.18
1.10
1.11
1.27
11.50
13.70
ppm


70°C
50°C
30°C
9.4 9.2 9.0 8.8 8.6 8.4 8.2
70°C
50°C
30°C
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 ppm


Alloc
OtBu
3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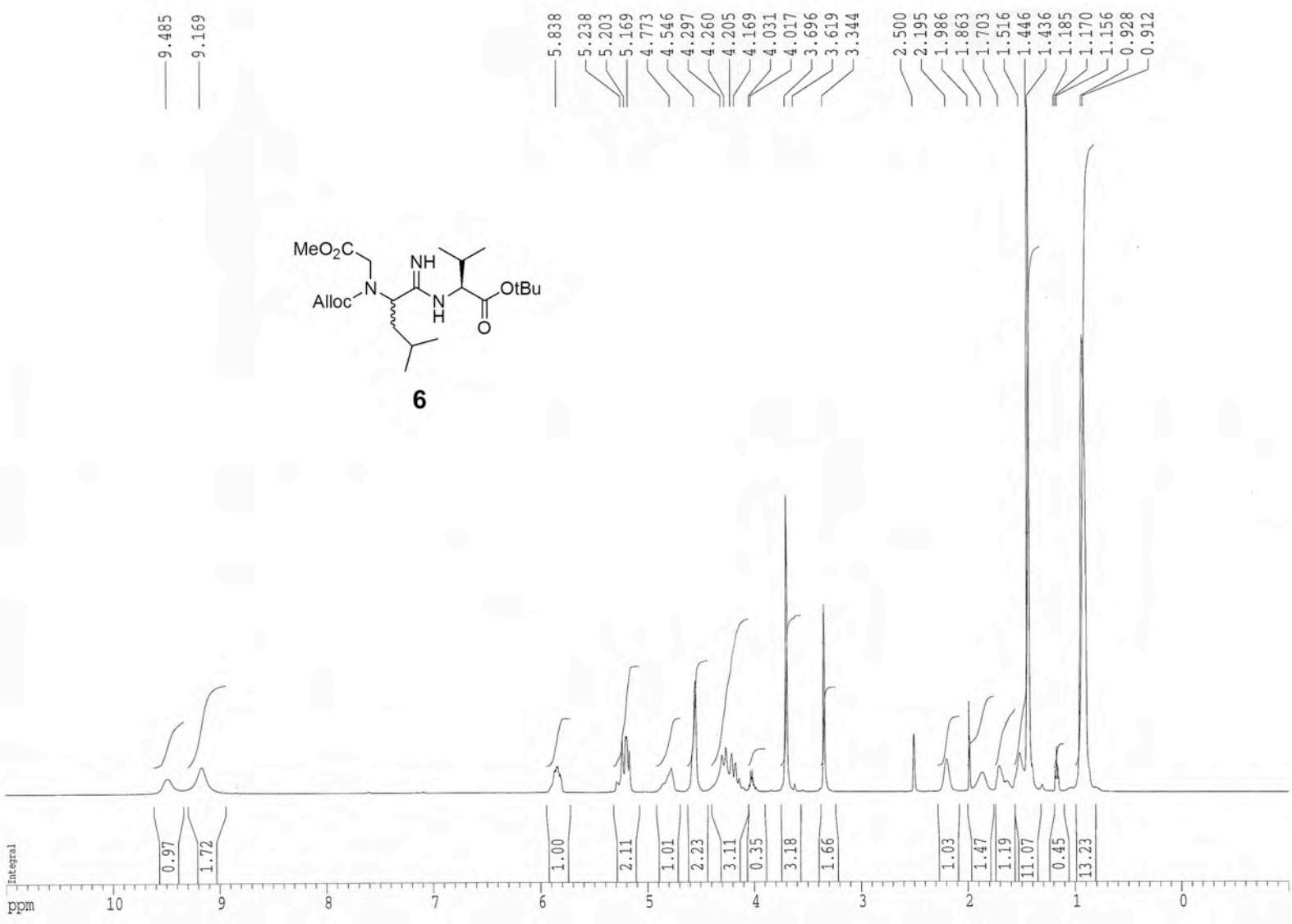
7.685
7.600
7.270
5.888
5.878
5.867
5.349
5.288
5.285
5.253
5.250
5.234
5.230
5.227
5.209
5.206
4.657
4.646
4.635
4.617
4.614
4.609
4.606
4.603
4.123
4.088
4.052
3.869
3.834
3.818
3.771
3.749
3.737
2.466
2.225
1.724
1.713
1.705
1.695
1.684
1.676
1.667
0.974
0.965
0.958
0.952
0.945
0.940
MeO2C
S
O
N
NH2
O
O
5
Integral
0.404
0.494
0.918
0.890
1.865
0.928
1.962
0.988
3.918
0.421
0.523
1.387
0.973
6.000
ppm
9
8
7
6
5
4
3
2
1
0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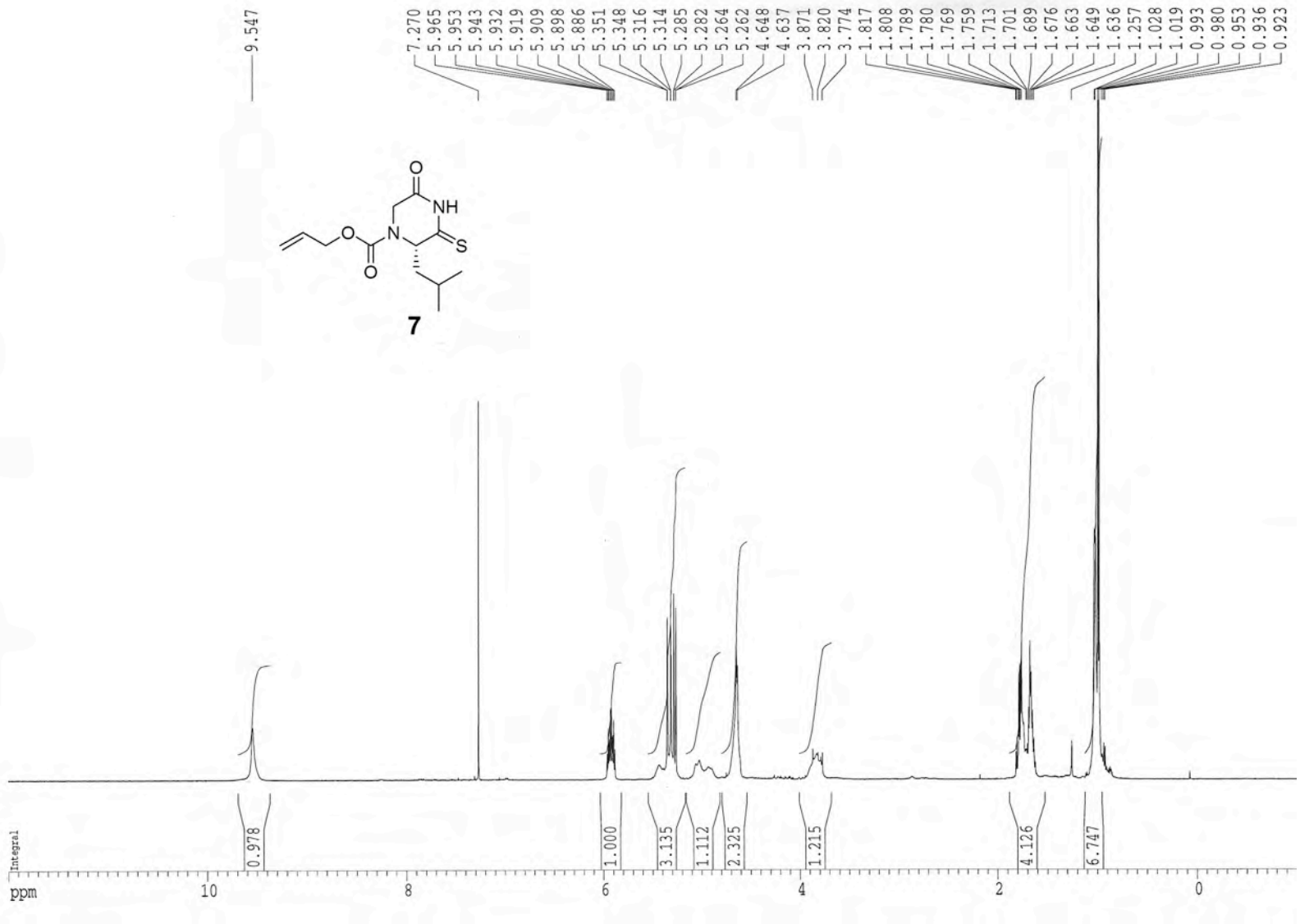
9.169
5.838
5.238
5.203
5.169
4.773
4.546
4.297
4.260
4.205
4.169
4.031
4.017
3.696
3.619
3.344
2.500
2.195
1.986
1.863
1.703
1.516
1.446
1.436
1.185
1.170
1.156
0.928
0.912
MeO2C
NH
Alloc
N
N
H
OtBu
O
6
Integral
0.97
1.72
1.00
2.11
1.01
2.23
3.11
0.35
3.18
1.66
1.03
1.47
1.19
11.07
0.45
13.23
ppm
10
9
8
7
6
5
4
3
2
1
0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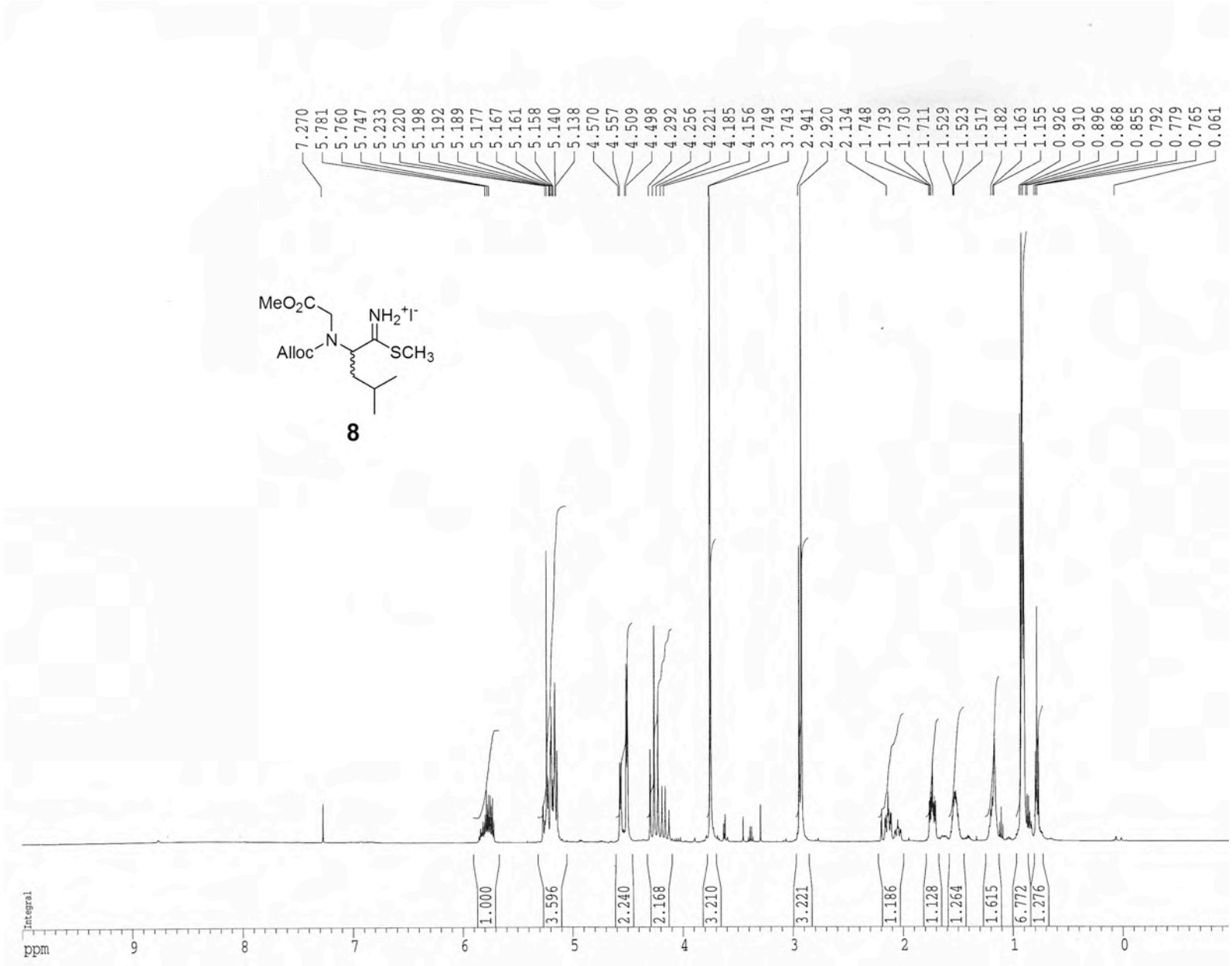
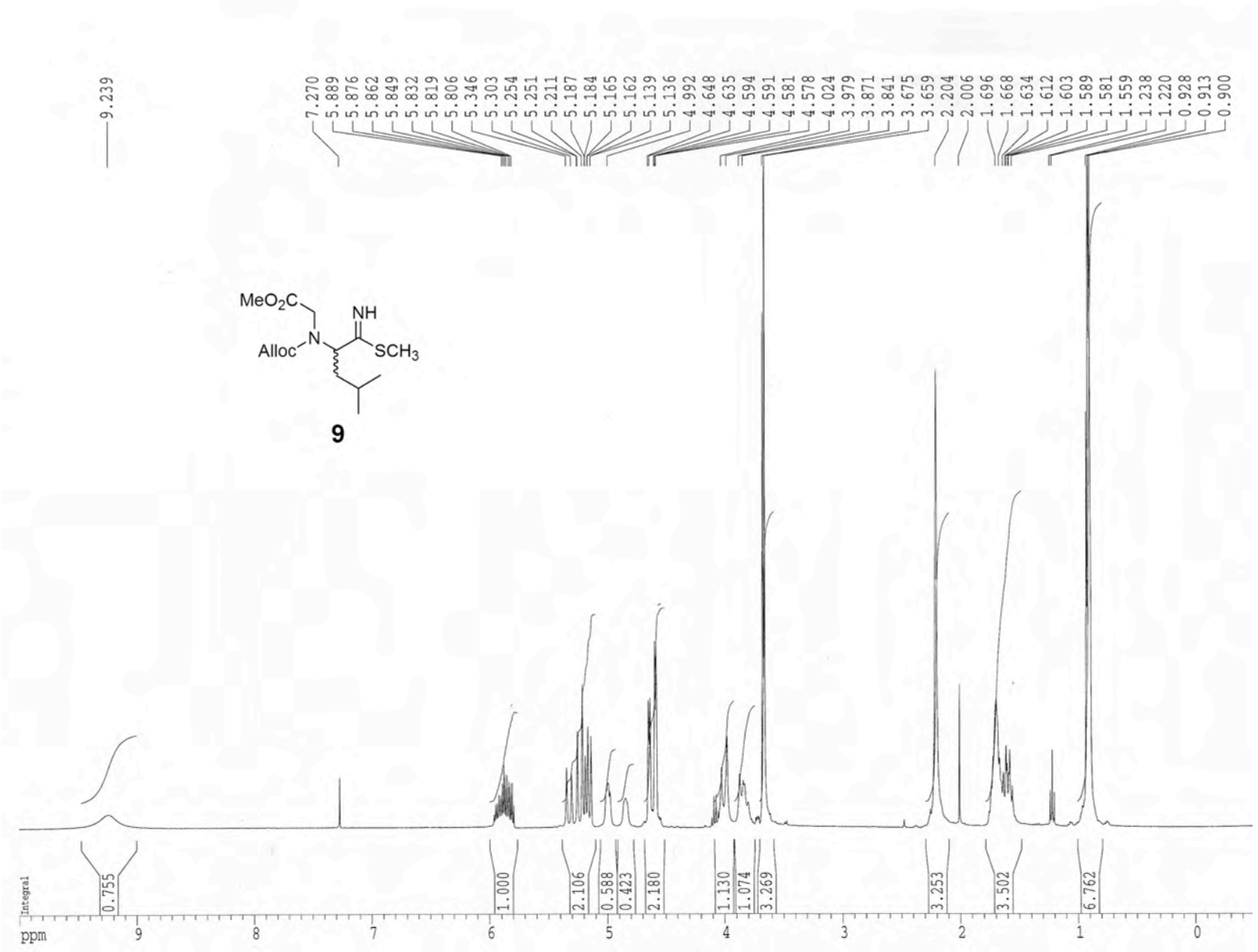
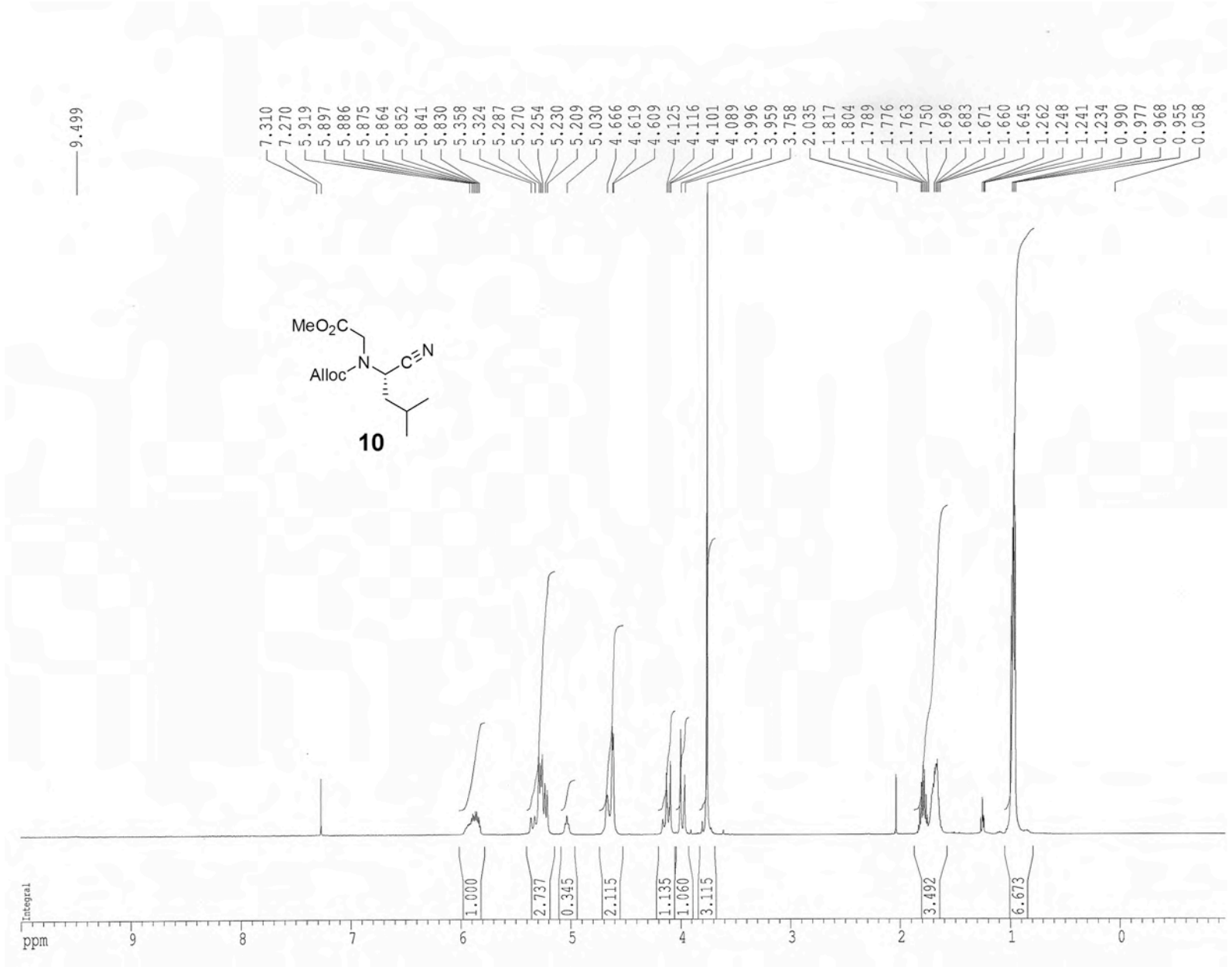
7.270
5.965
5.953
5.943
5.932
5.919
5.909
5.898
5.886
5.351
5.348
5.316
5.314
5.285
5.282
5.264
5.262
4.648
4.637
3.871
3.820
3.774
1.817
1.808
1.789
1.780
1.769
1.759
1.713
1.701
1.689
1.676
1.663
1.649
1.636
1.257
1.028
1.019
0.993
0.980
0.953
0.936
0.923
O
NH
O
N
O
S
O
7
Integral
0.978
1.000
3.135
1.112
2.325
1.215
4.126
6.747
ppm
10
8
6
4
2
0


7.270
5.781
5.760
5.747
5.233
5.220
5.198
5.192
5.189
5.177
5.167
5.161
5.158
5.140
5.138
4.570
4.557
4.509
4.498
4.292
4.256
4.221
4.185
4.156
3.749
3.743
2.941
2.920
2.134
1.748
1.739
1.730
1.711
1.529
1.523
1.517
1.182
1.163
1.155
0.926
0.910
0.896
0.868
0.855
0.792
0.779
0.765
0.061
MeO2C
NH2+I-
Alloc
N
SCH3
8
Integral
1.000
3.596
2.240
2.168
3.210
3.221
1.186
1.128
1.264
1.615
6.772
1.276
ppm
9
8
7
6
5
4
3
2
1
0


9.239
7.270
5.889
5.876
5.862
5.849
5.832
5.819
5.806
5.346
5.303
5.254
5.251
5.211
5.187
5.184
5.165
5.162
5.139
5.136
4.992
4.648
4.635
4.594
4.591
4.581
4.578
4.024
3.979
3.871
3.841
3.675
3.659
2.204
2.006
1.696
1.668
1.634
1.612
1.603
1.589
1.581
1.559
1.238
1.220
0.928
0.913
0.900
MeO2C
NH
Alloc
N
SCH3
9
Integral
0.755
1.000
2.106
0.588
0.423
2.180
1.130
1.074
3.269
3.253
3.502
6.762
ppm
9
8
7
6
5
4
3
2
1
0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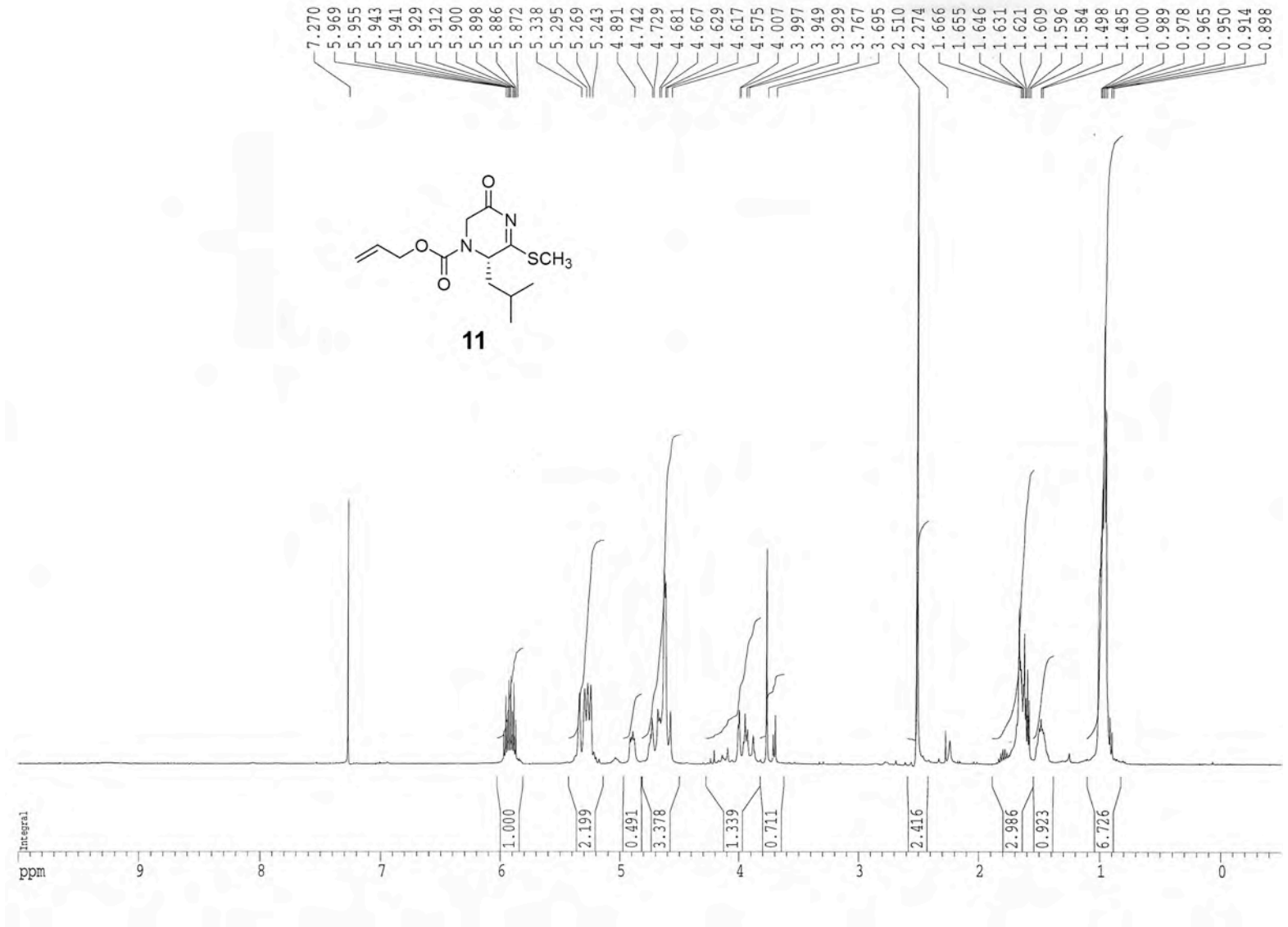
7.310
7.270
5.919
5.897
5.886
5.875
5.864
5.852
5.841
5.830
5.358
5.324
5.287
5.270
5.254
5.230
5.209
5.030
4.666
4.619
4.609
4.125
4.116
4.101
4.089
3.996
3.959
3.758
2.035
1.817
1.804
1.789
1.776
1.763
1.750
1.696
1.683
1.671
1.660
1.645
1.262
1.248
1.241
1.234
0.990
0.977
0.968
0.955
0.058
MeO2C
Alloc
N
C≡N
10
Integral
1.000
2.737
0.345
2.115
1.135
1.060
3.115
3.492
6.673
ppm
9
8
7
6
5
4
3
2
1
0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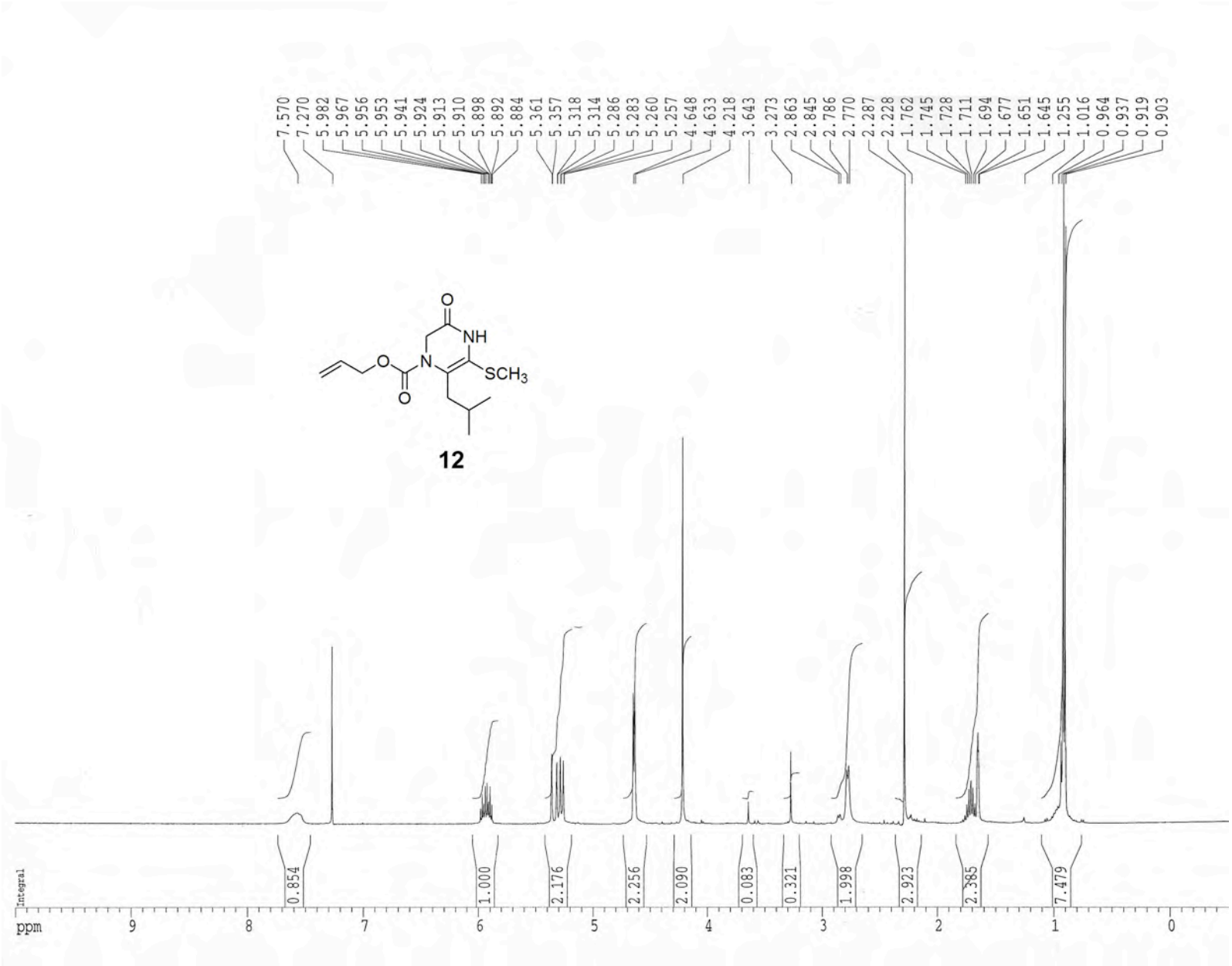
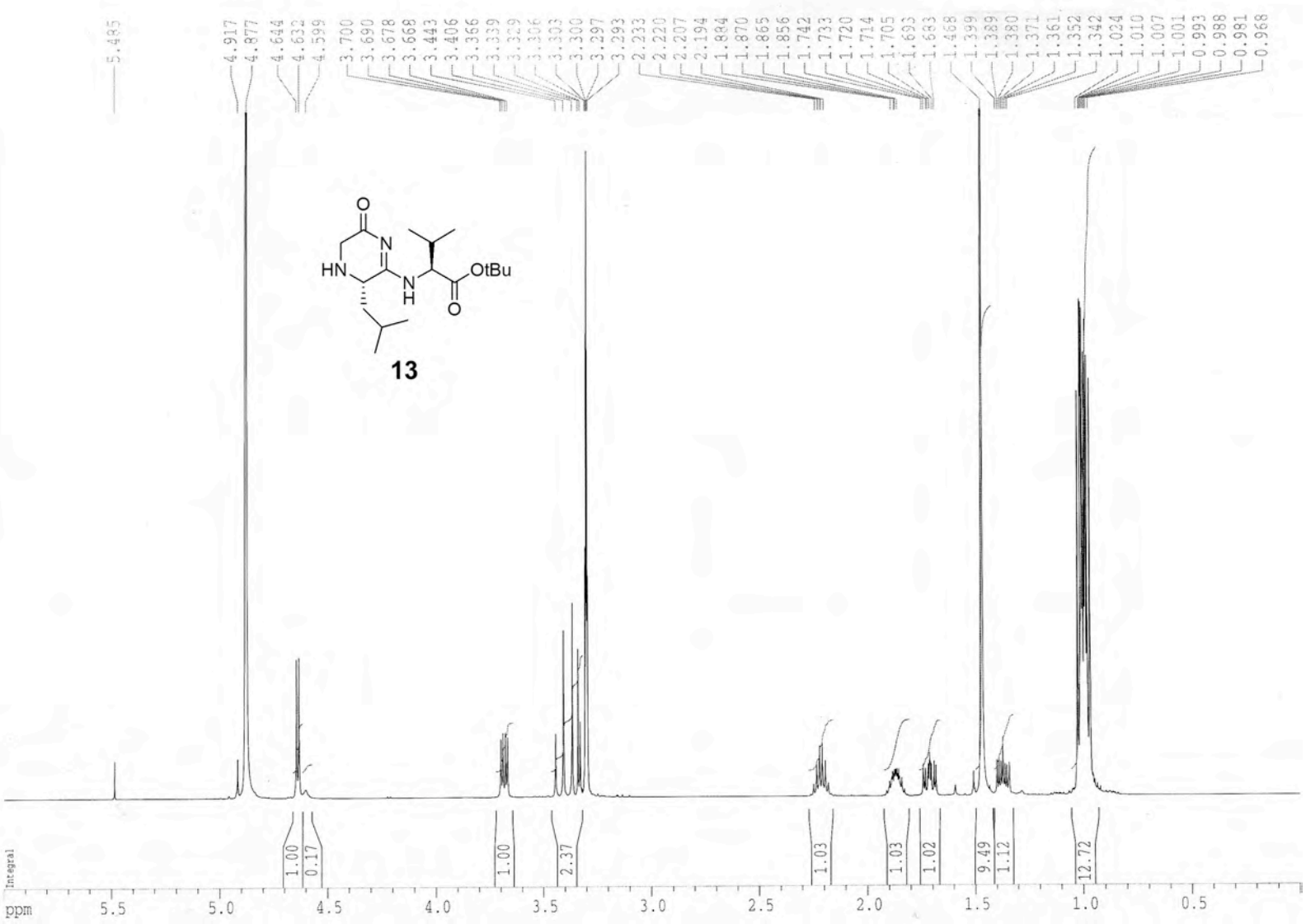
5.969
5.955
5.943
5.941
5.929
5.912
5.900
5.898
5.886
5.872
5.338
5.295
5.269
5.243
4.891
4.742
4.729
4.681
4.667
4.629
4.617
4.575
4.007
3.997
3.949
3.929
3.767
3.695
2.510
2.274
1.666
1.655
1.646
1.631
1.621
1.609
1.596
1.584
1.498
1.485
1.000
0.989
0.978
0.965
0.950
0.914
0.898
SCH3
11
Integral
1.000
2.199
0.491
3.378
1.339
0.711
2.416
2.986
0.923
6.726
ppm
9
8
7
6
5
4
3
2
1
0


7.570
7.270
5.982
5.967
5.956
5.953
5.941
5.924
5.913
5.910
5.898
5.892
5.884
5.361
5.357
5.318
5.314
5.286
5.283
5.260
5.257
4.648
4.633
4.218
3.643
3.273
2.863
2.845
2.786
2.770
2.287
2.228
1.762
1.745
1.728
1.711
1.694
1.677
1.651
1.645
1.255
1.016
0.964
0.937
0.919
0.903
O
NH
N
O
O
O
SCH3
12
Integral
0.854
1.000
2.176
2.256
2.090
0.083
0.321
1.998
2.923
2.385
7.479
ppm
9
8
7
6
5
4
3
2
1
0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4.917
4.877
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4.632
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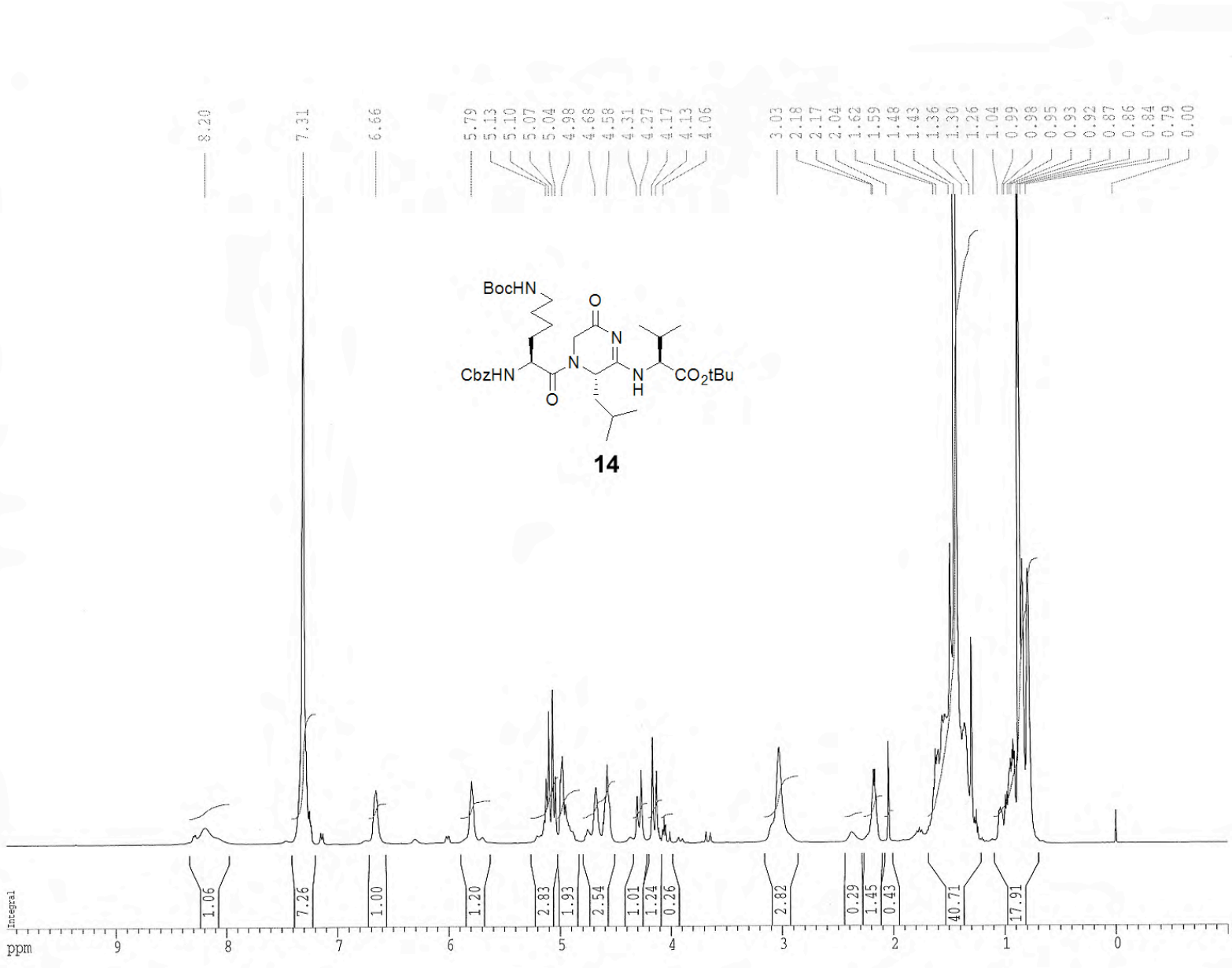
3.700
3.690
3.678
3.668
3.443
3.406
3.366
3.339
3.329
3.306
3.303
3.300
3.297
3.293
2.233
2.220
2.207
2.194
1.884
1.870
1.865
1.856
1.742
1.733
1.720
1.714
1.705
1.693
1.683
1.468
1.399
1.389
1.380
1.371
1.361
1.352
1.342
1.034
1.010
1.007
1.001
0.993
0.988
0.981
0.968
HN
N
N
H
O
O
OtBu
13
Integral
1.00
0.17
1.00
2.37
1.03
1.03
1.02
9.49
1.12
12.72
ppm
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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6.66
5.79
5.13
5.10
5.07
5.04
4.98
4.68
4.58
4.31
4.27
4.17
4.13
4.06
3.03
2.18
2.17
2.04
1.62
1.59
1.48
1.43
1.36
1.30
1.26
1.04
0.99
0.98
0.95
0.93
0.92
0.87
0.86
0.84
0.79
0.00
BocHN
CbzHN
CO2tBu
14
Integral
1.06
7.26
1.00
1.20
2.83
1.93
2.54
1.01
1.24
0.26
2.82
0.29
1.45
0.43
40.71
17.91
ppm
9
8
7
6
5
4
3
2
1
0