SUPPORTING INFORMATION for

"@-Tides": The 1,2-Dihydro-3(6*H*)-pyridinone Unit as a β-Strand Mimic

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General Experimental conditions. Thin-layer chromatography was performed using Merck silica gel 60 F_{254} plates. Analytical HPLC analysis was performed using a Waters 996 Photodiode Array Detector with a Waters 600 Controller and pump. The column was a Varian DYNAMAX-100 Å (4.6 mm × 250 mm) reverse-phase C18 column. Solvent mixtures for all chromatographic analyses included 0.1% TFA. The gradient was equilibrated in 9:1 H_2O/MeCN at 1 mL/min flow rate, followed by ramping to 1:19 H_2O/MeCN over 15 min. This solvent ratio was then run for an additional 15 min for a total run time of 30 min. Retention times were recorded for this gradient. Preparative HPLC purification was performed using a Waters 486 Tunable Absorbance Detector and a Waters 600 Controller and Pump. The column was a Varian DYNAMAX-100 Å (21.4 mm × 50 mm) reverse-phase C18 column. The preparative gradient was equilibrated in 9:1 H_2O/MeCN at 20 mL/min flow rate for 10 min, followed by ramping to 1:19 H_2O/MeCN over 25 min. This solvent ratio was then run for an additional 5 min for a total run time of 40 min. Liquid chromatography-mass spectrometry (LCMS) analysis was performed using a Hewlett Packard Series 1100 LCMS with a HP Series 1100 MSD and a Zorbax SB-C18 reverse-phase column (2.1 mm ID × 5 cm). The LCMS gradient was equilibrated in 9:1 H_2O/MeCN at 0.4 mL/min flow rate, followed by ramping to 1:19 H_2O/MeCN over 8 min. This solvent ratio was then run for an additional 3 min for a total run time of 11 min. Infrared (IR) spectra were recorded with a Perkin-Elmer 1600 Fourier transform infrared spectrophotometer and are reported in wavenumbers (cm^{-1}). Mass spectral data were obtained by the Mass Spectrometry Laboratory of the College of Chemistry, University of California, Berkeley or by LCMS (HP).

Abbreviations used: @ and Ach: the 1,2-dihydro-3(6*H*)-pyridinyl unit; DIEA: diisopropylethylamine.

3,5-Dimethoxypyridine. Prepared by a modification of the procedure reported by Testaferri et al. (Testaferri, L; Tiecco, M.; Tingoli, M.; Bartoli, D.; Massoli, A. *Tetrahedron* **1985**, *41*, 1373-84). A flame-dried flask equipped with a gas inlet, reflux condenser, and a bubbler was charged with degassed, anhydrous DMF (450 mL) and 3,5-dichloropyridine (40.6 g, 270 mmol). Potassium methoxide (58.3 g, 830 mmol) was added and the mixture was stirred vigorously at 72 °C for 16 h while a strong stream of nitrogen was passed through the solution. The DMF was removed under high vacuum pressure and the resulting oil was distilled under reduced pressure. The distillate was

lyophilized from benzene 3-5 times to removing residual DMF and afford 3,5-dimethoxypyridine as a light yellow oil (22.9 g, 162 mmol, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.82 (s, 6), 6.71 (dd, 2, J = 2.4), 7.95 (d, 1, J = 2.4); HRMS (FAB) m/z calcd for $C_7H_9NO_2$ (M^+) 139.0633, found: 139.0637.

***tert*-Butyl (2*S*,3*S*)-3-*tert*-Butoxy-2-{[5-oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}-butanoate (Alloc-Ach-Thr(*O*-*t*Bu) *t*-Butyl Ester, 4b).** To a solution of mesitylate **3** (335 mg, 884 μmol) in dry THF (3 mL) were added *O*-*t*-butyl-threonine *t*-butyl ester hydrochloride (225 mg, 972 μmol), anhydrous ytterbium(III)-trifluoromethanesulfonate ($Yb(OTf)_3$; 55.0 mg, 88.0 μmol), and DIEA (461 μL, 2.65 mmol) under a nitrogen atmosphere. After 24 h, saturated NH_4Cl (2 mL) was added, and the mixture was extracted with EtOAc (3 × 5 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated. Purification of the crude product by flash chromatography on silica using hexanes/EtOAc (1:1) gave vinylogous amide **4b** (294 mg, 716 μmol, 81%) as a light yellow oil. Proton and carbon spectra show peak doubling due to rotation of the vinylogous amide bond. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.18 (s, 9), 1.22-1.27 (m, 3), 3.74 (dd, 1, J = 1.4, 9); 4.02-4.10 (m, 2); 4.21 (m, 1); 4.30-4.40 (m, 2); 4.63 (s, 2), 5.23 (d, 1, J = 10.2), 5.32 (d, 1, J = 17.2), 5.53-5.59 (bs, 1); 5.93 (bm, 1); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.68, 21.25, 21.51, 22.03, 28.55, 28.57, 29.10, 29.27, 44.95, 51.13, 60.83, 61.51, 61.79, 67.09, 67.41, 68.94, 74.13, 74.92, 81.83, 83.27, 96.12, 118.40, 132.89, 169.25, 173.51, 191.54; MS (FAB) m/z (%) = 411 (M^+, 100), 355 (28); HRMS (FAB) m/z calcd for $C_{21}H_{34}N_2O_6$ (M+H$^+$): 411.2495; found: 411.2495.

***tert*-Butyl (2*S*)-4-Methyl-2-{[5-oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}pentanoate (Alloc-Ach-Leu *t*-Butyl Ester, 4c).** To a solution of mesitylate **3** (1.0 g, 2.6 mmol) in dry THF (11 mL) were added leucine *t*-butyl ester hydrochloride (0.5 g, 2.7 mmol), anhydrous $Yb(OTf)_3$ (1.64 g, 2.65 mmol), and DIEA (1.38 mL, 7.92 mmol) under a nitrogen atmosphere. After 24 h saturated NH_4Cl was added (10 mL), and the mixture was extracted with EtOAc (3 × 10 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated. Purification of the crude product by flash chromatography on silica using hexanes/EtOAc (1:1) gave vinylogous amide **4c** (0.71 g, 1.9 mmol, 74%) as a light yellow oil. Proton and carbon spectra show peak doubling due to rotation of the vinylogous amide bond. ^{1}H NMR (500 MHz, $CDCl_3$) δ = 0.94 (t, 3, J = 6.9); 0.95 (t, 3, J = 6.9); 1.33-1.84 (m, 2); 1.50 (s, 9); 3.94 (dd, 1, J = 7.1, 14.6); 4.00-4.14 (m, 2); 4.26-4.38 (m, 2); 4.62 (d, 2, J = 5.8); 5.20 (s, 1); 5.23 (ddd, 1, J = 1.3, 2.7, 13.4); 5.31 (ddd, 1, J = 1.5, 3.1, 17.3); 5.85-5.98 (m, 1); 6.02 (d, 1, J = 6.6); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.89, 22.21, 22.31, 22.80, 24.73, 24.81, 27.86, 27.92, 40.87, 44.09, 50.54, 53.29, 54.34, 66, 80.69, 82.6, 95.35, 117.92, 132.15, 132.26, 175.80; MS (FAB) m/z (%) = 598 (12), 536 (14), 450 (92), 367 (M^++198), 356 (24), 348 (14), 338 (44), 311 (100), 300 (16), 292 (22), 265 (24), 254 (12), 244 (56), 225 (24), 198 (56), 179 (12), 154 (32), 136 (26); HRMS (FAB) m/z calcd for $C_{19}H_{30}N_2O_5$ (M+H$^+$): 367.2235; found: 367.2233.

***tert*-Butyl (2*S*)-2-{[5-Oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}-3-phenylpropanoate (Alloc-Ach-Phe *t*-Butyl Ester, 4d).** To a solution of mesitylate **3** (180 mg, 0.46 mmol) in dry THF (6 mL) were added phenylalanine *t*-butylester hydrochloride (110 mg, 0.42 mmol), anhydrous $Yb(OTf)_3$

(20.0 mg, 0.028 mmol), and DIEA (0.24 mL, 1.4 mmol) under a nitrogen atmosphere. After 24 h saturated NH_4Cl was added (4 mL), and the mixture was extracted with EtOAc (3 × 5 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated. Purification of the crude product by flash chromatography on silica using hexanes/EtOAc (1:1) gave vinylogous amide **4d** (126 mg, 0.31 mmol, 75%) as a light yellow oil. Proton and carbon spectra show peak doubling due to rotation of the vinylogous amide bond. ^1H NMR (500 MHz, $CDCl_3$) δ 1.40 (s, 9), 3.09 (q, 3, *J* = 5, *J* = 6, *J* = 8, *J* = 6), 4.08 (d, 3, *J* = 2); 4.25 (m, 2); 4.57 (bs, 1); 5.27 (m, 3), 5.74 (bs, 1), 5.91 (bs, 1), 7.13 (d, 2, *J* = 7), 7.28 (m, 3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 27.82, 37.04, 44.10, 50.60, 56.29, 66.58, 83.16, 95.70, 117.85, 127.22, 128.46, 129.29, 132.22, 135.15, 154.80, 159.62, 169.61, 191.02; MS (FAB) *m/z* (%) = 401 (100) (M^++H), 345 (90); HRMS (FAB) *m/z* calcd for $C_{22}H_{28}N_2O_5$ ($M+H^+$): 401.2077; found: 401.2076.

Ac-Phe-Sar-Leu-Sar-IleNHMe (16). Peptide **16** was prepared on Wang resin using standard solid phase peptide coupling protocols (Hudson, D., *J. Org. Chem.* **1988**, *53*, 617) with HBTU and HOBt as coupling reagents. The resin bound peptide was acetylated using 3:1:2 CH_2Cl_2/pyridine/Ac_2O as solvent, mixing for 3 h. The peptide was cleaved from resin in 20% TFA in CH_2Cl_2 for 1 h and the solvent was subsequently removed under reduced pressure. The sample was redissolved in MeOH and filtered through a glass wool plug. The filtrate was concentrated *in vacuo* affording pure peptide as determined by HPLC (70% yield). The peptide was subsequently dissolved in 4.7 mL of dry THF and *N*-methylmorpholine (1.1 eq) was added at 0 °C. Isobutyl chloroformate (1.1 eq) was added slowly to the mixture while the temperature was maintained at 0 °C. Methylamine (2 M in THF, 10 eq) was added and the reaction mixture was stirred at rt for 3 h. The solvent was removed under reduced pressure and the coupling procedure was repeated. The solvent was filtered through a 0.45-μm Whatman HPLC filter and condensed. The resulting residue was purified by preparative HPLC to afford peptide **16** in 90% yield. ^1H NMR (500 MHz, $CDCl_3$-CD_3OH 10:1): δ = 0.84-0.91 (m, 6, H-32, H-33); 0.94-0.63 (m, 6, H-21, H-22); 1.06-1.08 (m, 1, H-31); 1.43–1.47 (m , 2, H-31, H-18); 1.64-1.69 (m, 2, H-19, H-18); 1.83-1.85 (m, 1, H-30); 1.91/1.92/1.95 (each s, 3 total, H-1 rotamers); 2.74/2.76 (s, 3, H-36 rotamers); 2.87 (s, 3H,H-27); 2.94-2.97 (m, 1, H-5); 3.02-3.06 (m, 1, H-5); 2.87 (s, 3, H-15); 3.50 (d, *J* = 16.2, 1, H-11/H-23); 3.89 (d, *J* = 16.8, 1, H-23/H-11); 3.96 (d, *J* = 15.8, 1, H-23/H-11); 4.09 (d, *J* = 15.9, 1, H-29); 4.40 (d, *J* = 19.3, 1, H-11/H-23); 4.74-4.75 (m, 1, H-17); 4.95 (t, *J* = 7.1, 1, H-4); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 10.72 (q, C-32); 15.19 (q, C-33); 21.34 (q, C-20, C-21); 22.15 (q, C-1); 23.04 (q, C-20, C-21); 24.49 (d, C-19); 24.51 (t, C-31); 25.76 (q, C-36); 36.22 (q, C-15, C-27); 36.33 (d, C-30); 36.53 (q, C-15, C-27); 37.63 (t, C-5); 40.19 (t, C-18); 17.85 (d, C-17); 50.83 (d, C-4); 51.40 (t, C-11/C-23); 51.82 (t, C-11/C-23); 57.89 (d, C-29); 126.99 (d, C-9); 128.34 (d, C-8); 129.09 (d, C-7); 135.79 (s, C-6); 168.54, 168.62, 170.77, 171.98, 172.64, 173.45 (C=O); HRMS (FAB) calcd for $C_{30}H_{49}N_6O_6$ (MH^+): 589.3714; found: 589.3713.

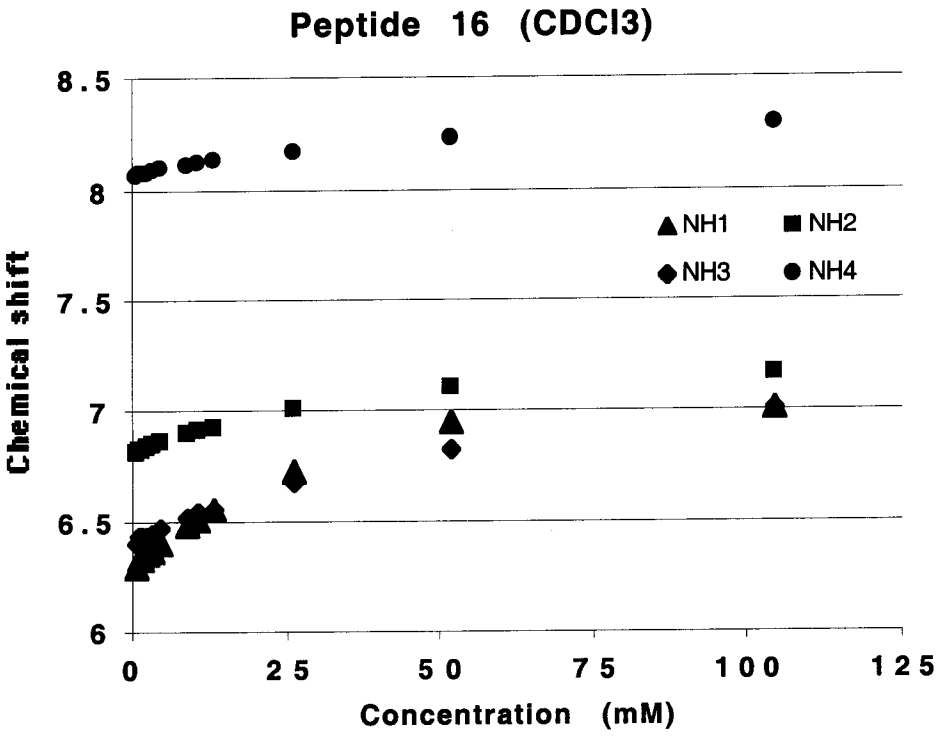
The following compounds were also prepared on solid phase using conventional Wang resin purchased from NovaBiochem:

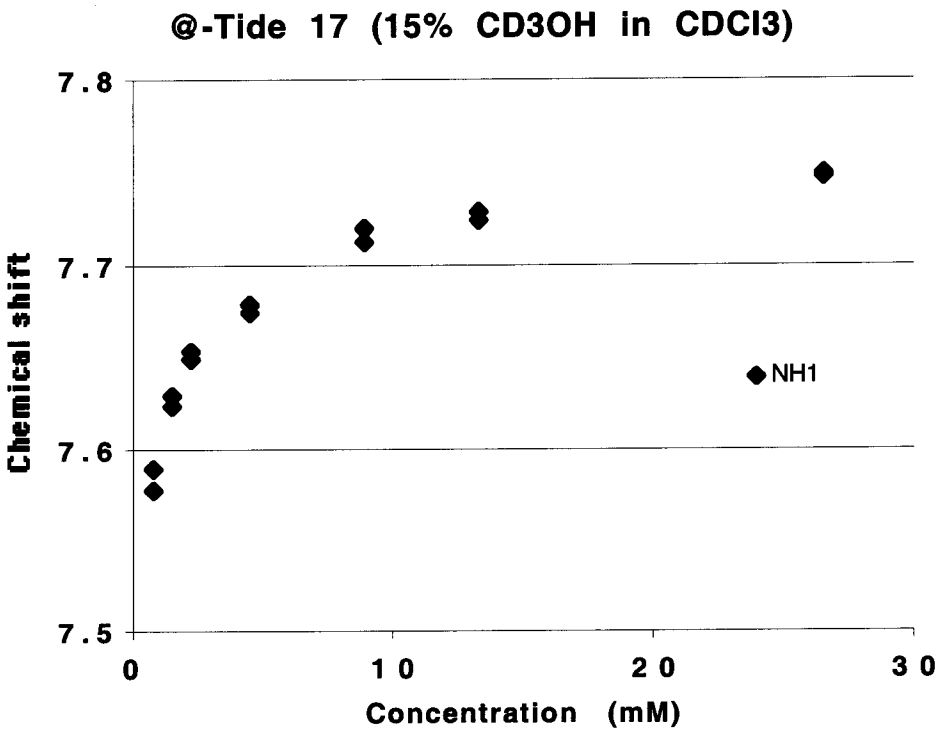
Ac-Leu-Ach-Val-Ach-Leu-Ach-Phe (17). Hepta-@-tide **17** bound to resin (0.1 g resin, 0.91 mmol/g loading) was cleaved yielding crude product, which was purified by preparative reverse-phase HPLC to afford hepta-@-tide **17** (13 mg, 0.02 mmol, 18% overall) as a light yellow foam. Retention time (analytical HPLC) = 15.5 min; ^{1}H NMR (500 MHz, CD_3OD) δ 0.95 (br m, 18), 1.31(br m, 7), 1.65 (br m, 6), 1.96 (d, 3), 2.03 (m, 0.6), 2.19 (m, 0.7), 3.03 (m, 0.8), 3.14 (m, 0.6), 4.14 (br m, 3.6), 4.34 (br m, 4), 4.47 (br m, 2), 4.61 (br m, 2), 4.94 (br m, 2), 5.08 (m, 2.4), 5.34 (m, 0.5), 7.26 (br m, 5); MS (FAB) *m/z* (%) 818 (M + H$^+$, 50); HRMS (FAB) *m/z* 818.4427 (M + H$^+$, $C_{43}H_{60}N_7O_9$ requires 818.4453).

Ac Phe-Ach-Leu-Ach-Val (18). Penta-@-tide **18** bound to resin (0.3 g resin, 0.91 mmol/g loading) was cleaved yielding crude pruduct, which was purified by preparative reverse-phase HPLC to afford penta-@-tide **18** (85 mg, 0.14 mmol, 51% overall) as a light yellow foam. The proton spectrum is complicated due to the presence of rotamers. Retention time (analytical HPLC) = 14.8 min; ^{1}H NMR (400 MHz, CD_3OD) δ 0.88 (br m, 12), 1.23 (br m, 0.3), 1.36 (br m, 0.5), 1.52 (br m, 1), 1.61 (m, 1), 1.77 (s, 1), 2.07 (m, 3), 2.20 (m, 1), 2.83 (m, 1.8), 3.68-3.85 (br m, 2), 3.95-4.07 (br m, 1.6), 4.17 (br m, 1.8), 4.36 (m, 1.3), 4.46 (m, 1.8), 4.59 (m, 0.8), 4.72 (d, 0.5, *J* = 17.5), 5.02 (m, 0.5), 5.09 (m, 0.5), 5.20 (m, 0.3), 7.21 (m, 5); ^{13}C NMR (125 MHz, CD_3OD) δ 19.10, 19.44, 22.37, 23.66, 26.28, 31.99, 39.18, 41.43, 43.39, 43.92, 46.61, 52.12, 53.19, 63.01, 94.95, 128.17, 129.71, 130.50, 137.87, 171.99, 172.92, 173.78; MS (FAB) *m/z* (%) 610 (M + H$^+$, 100); HRMS (FAB) *m/z* 610.3249 (M + H$^+$, $C_{32}H_{44}N_5O_7$ requires 610.3241).

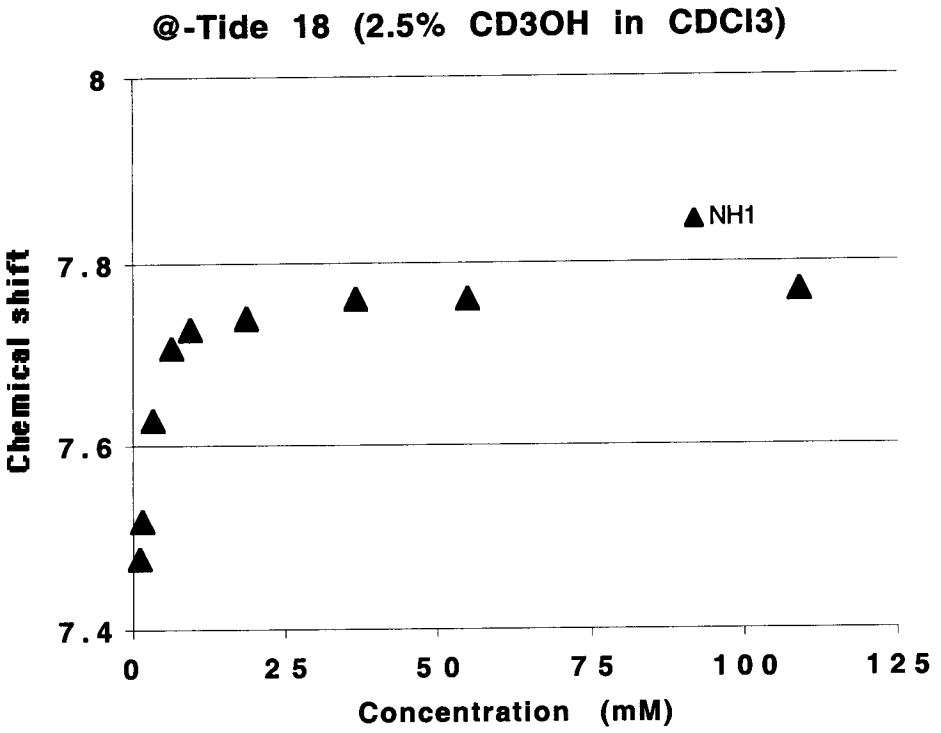
Ac-Leu-Ach-Val (19). Tri-@-tide **19** bound to resin (0.1 g resin, 0.91 mmol/g loading) was cleaved yielding crude product, which was purified by preparative reverse-phase HPLC to afford tri-@-tide **19** (28 mg, 0.08 mmol, 83% overall) as a light yellow foam. The proton spectrum is complicated due to the presence of rotamers. Retention time (analytical HPLC) = 13.6 min; ^{1}H NMR (500 MHz, CD_3OD) δ 0.92-1.09 (br m, 12), 1.29 (br s, 0.8), 1.42 (br m, 0.7), 1.56 (br m, 1), 1.65 (br m, 1.2), 1.96 (s, 3), 2.20 (br m, 1), 3.35 (s, 0.2), 3.84 (t, 0.9, *J* = 8.5, *J* = 7.0), 4.00 (d, 0.4, *J* = 17.5), 4.11 (s, 0.2), 4.15 (s, 0.4), 4.20 (s, 0.4), 4.23 (s, 0.2), 4.27 (s, 0.2), 4.30 (s, 0.2), 4.45 (d, 0.5, *J* = 17.5), 4.58 (s, 1), 4.62 (s, 0.3), 4.83 (m, 0.6), 5.17 (d, 0.9, *J* = 8.5); ^{13}C NMR (125 MHz, CD_3OD) δ 19.12, 19.44, 22.05, 22.33, 23.63, 26.18, 31.97, 41.66, 43.69, 46.85, 50.39, 53.17, 63.13, 94.92, 95.21, 164.64, 165.71, 173.25, 192.81, 194.24; MS (FAB) *m/z* (%) 368 (M + H$^+$, 100); HRMS (FAB) *m/z* 368.2187 (M + H$^+$, $C_{18}H_{30}N_3O_5$ requires 368.2186).

Concentration Dependence of ^1H NMR Chemical Shifts.

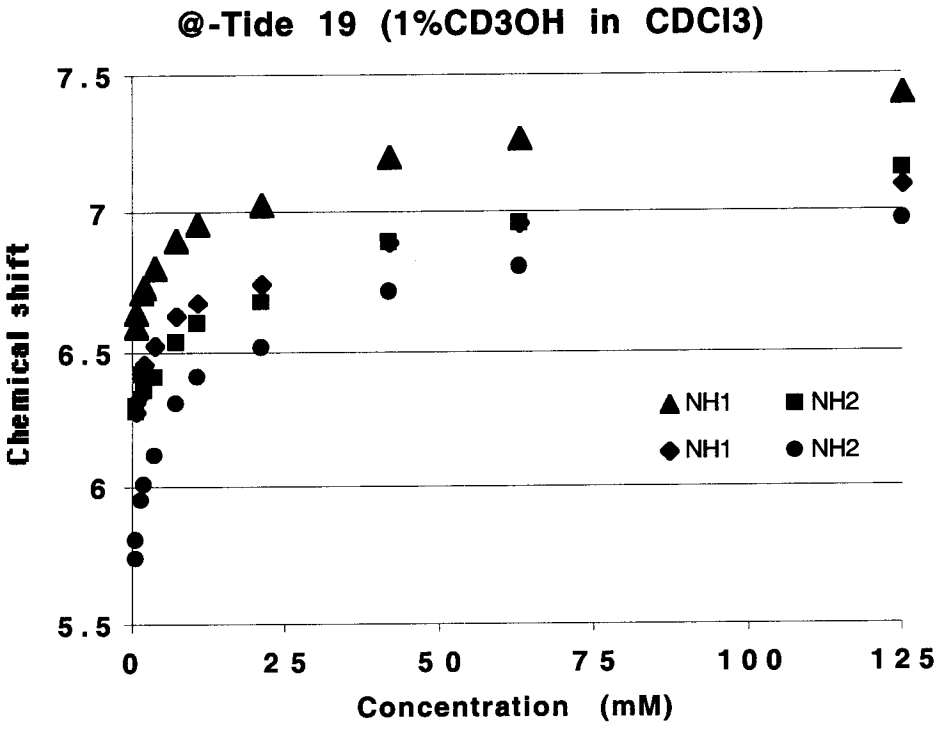




Note: The other resonances for 17 were obscured by the aromatic resonances during the course of the titration and were not used for the K_d determination.

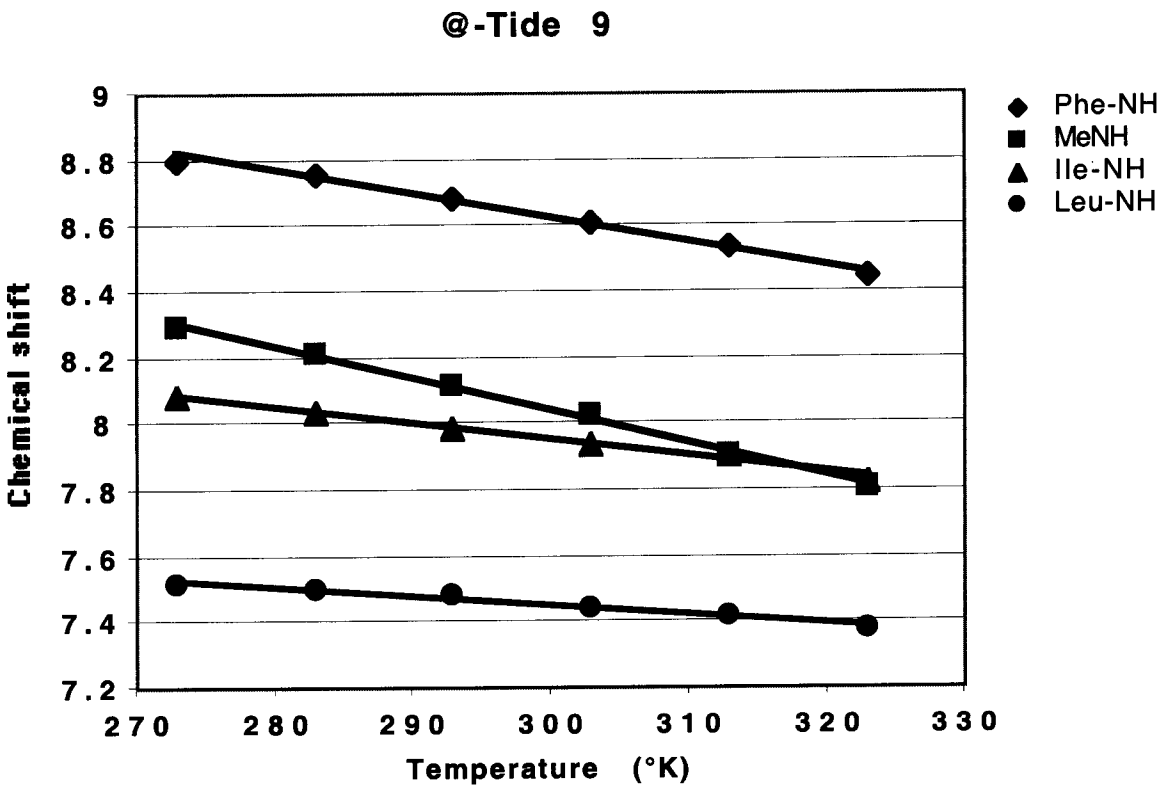


Note: The other resonances of **18** were obscured by the aromatic resonances during the course of the titration and were not used for the K_d determination.



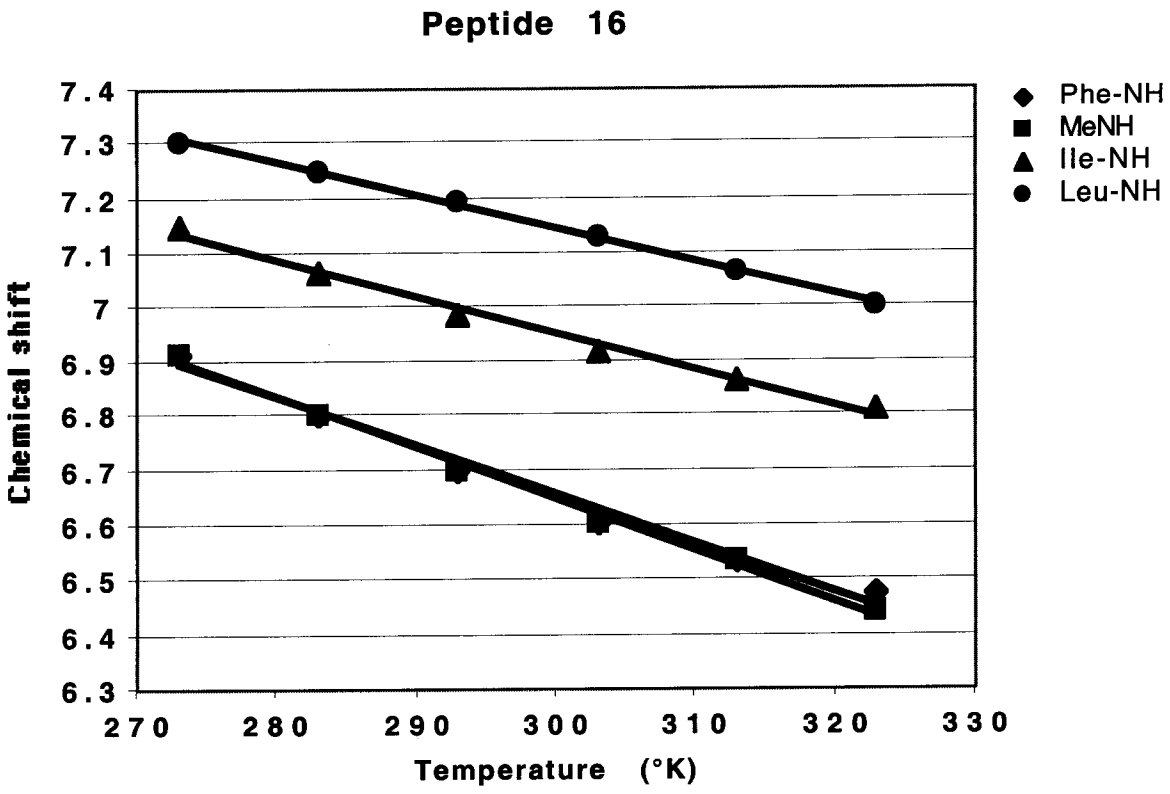
Sample Variable Temperature Data for @-Tide 9 (1% CD_3OH in $CDCl_3$).

Temperature (°K)	Phe-NH	MeNH	Ile-NH	Leu-NH
273	8.800	8.291	8.084	7.515
283	8.760	8.214	8.039	7.499
293	8.681	8.119	7.984	7.482
303	8.612	8.023	7.936	7.447
313	8.534	7.904	7.904	7.415
323	8.445	7.805	7.825	7.378

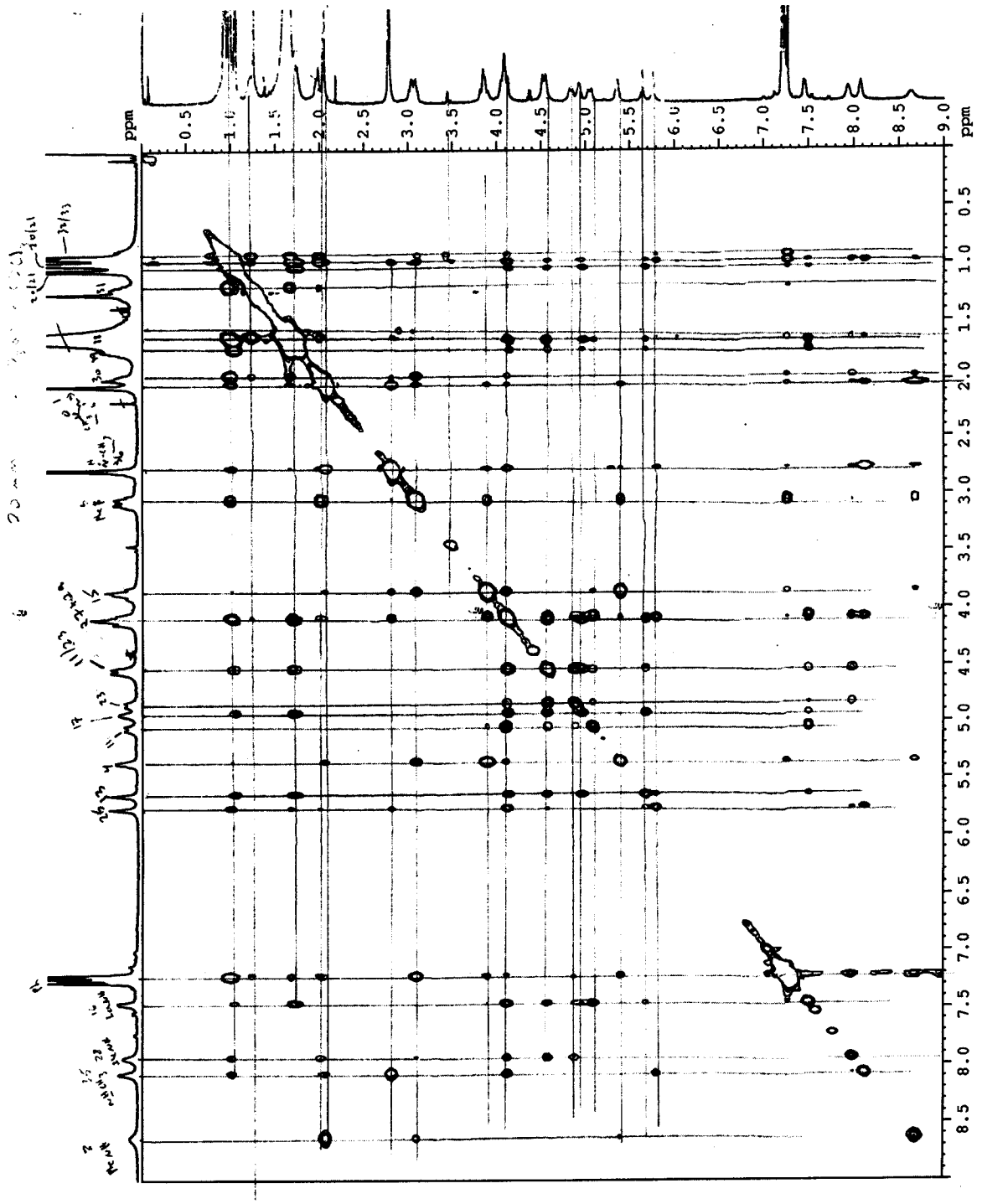


Sample Variable Temperature Data for Peptide 16 (1% CD_3OH in $CDCl_3$).

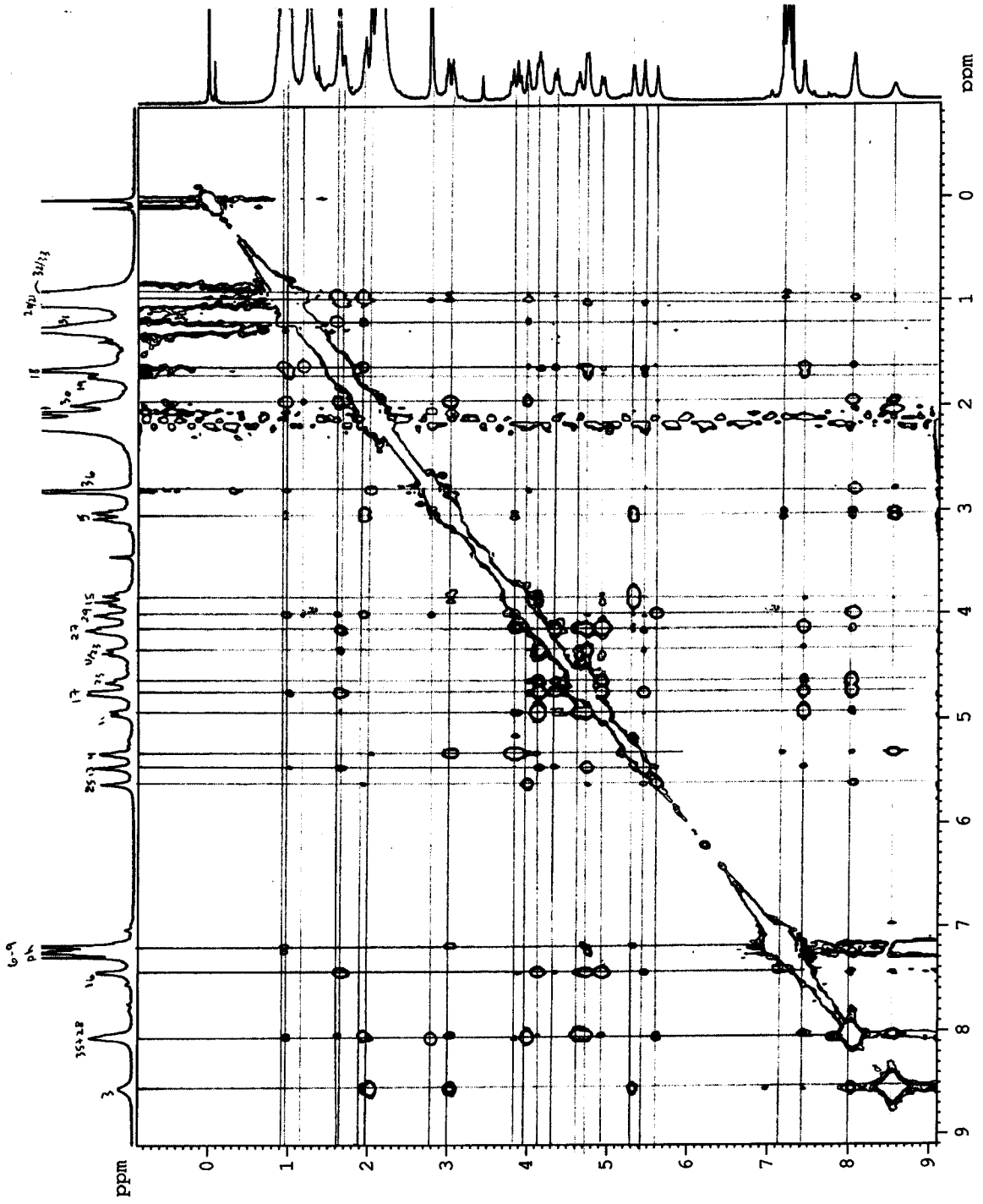
Temperature (°K)	Phe-NH	MeNH	Ile-NH	Leu-NH
273	6.915	6.915	7.148	
283	6.803	6.803	7.065	7.246
293	6.698	6.698	6.986	7.195
303	6.604	6.604	6.920	7.128
313	6.534	6.534	6.863	7.065
323	6.478	6.440	6.812	7.003



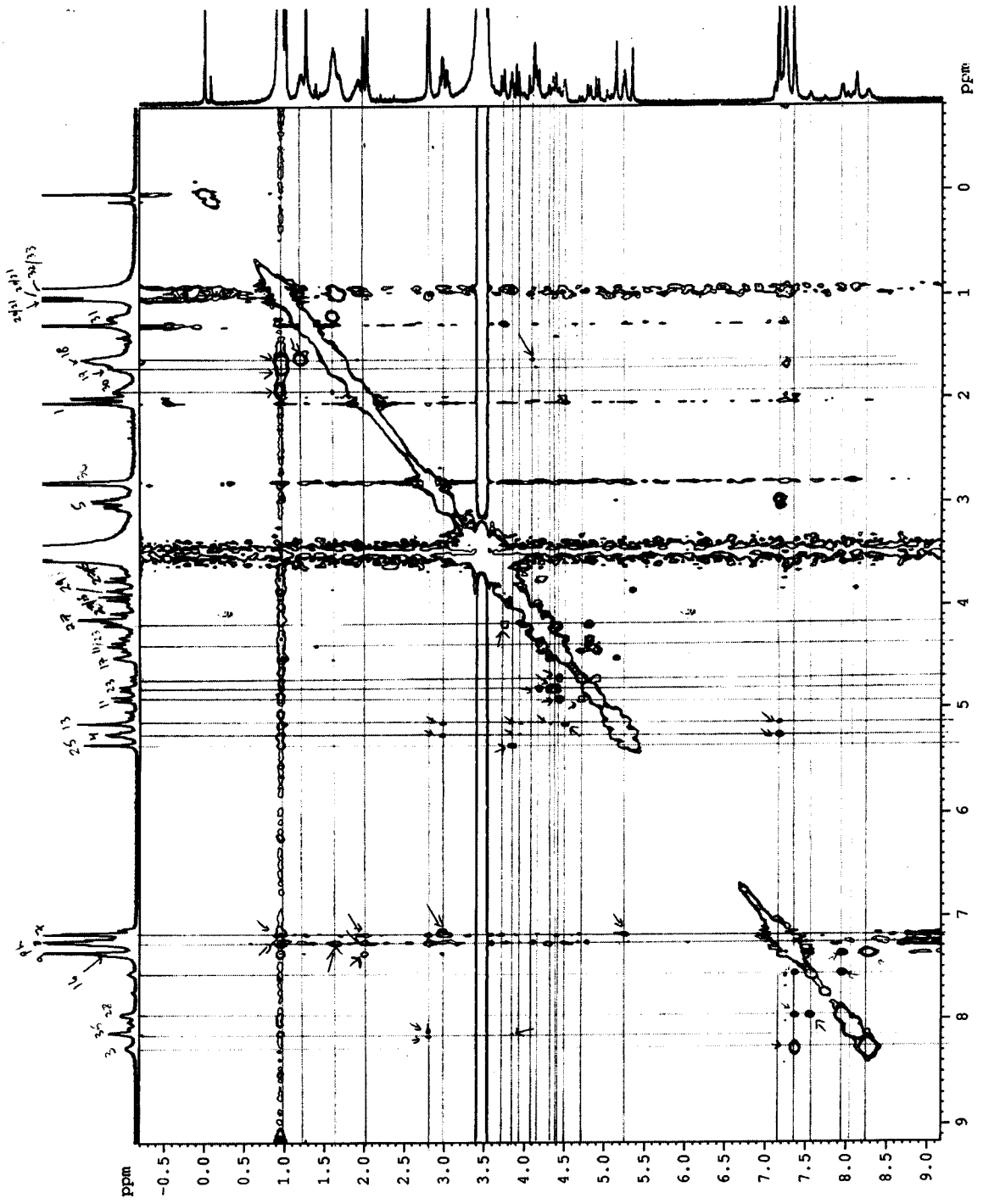
NOESY Spectrum of @-Tide 9: concentration: 20 mM
solvent: 1% $CD_3OH/CDCl_3$
temperature: 20 °C



NOESY Spectrum of @-Tide 9: concentration: 35 mM
solvent: 2.5% $CD_3OH/CDCl_3$
temperature: 20 °C

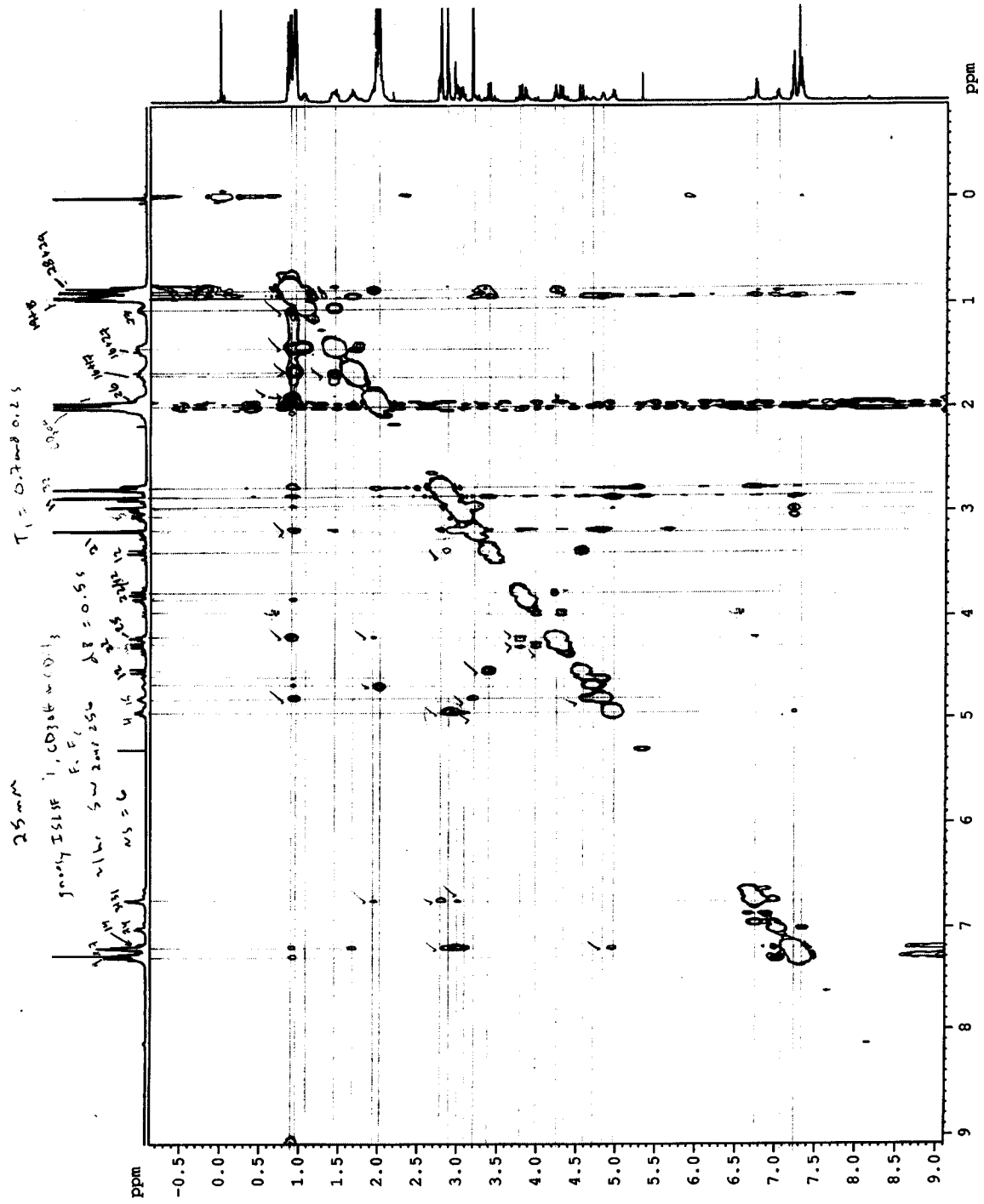


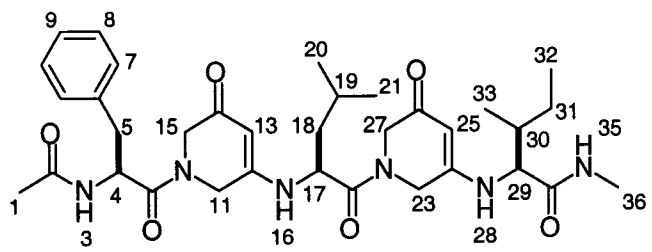
NOESY Spectrum of @-Tide 9: concentration: 22 mM
solvent: 10% $CD_3OH/CDCl_3$
temperature: 20 °C

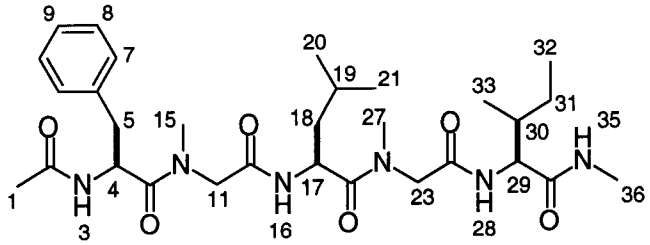


NOESY Spectrum of Peptide 16:

concentration: 25 mM
solvent: 1% $CD_3OH/CDCl_3$
temperature: 20 °C





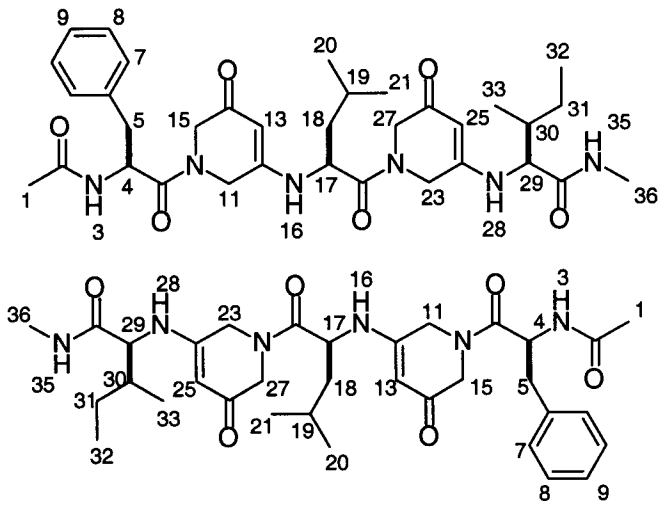


Intramolecular NOEs

	@-Tide 9			Peptide 16
% $CD_3OH/CDCl_3$:	1%	2.5%	10%	1%
1-3	•	•		
1-4	•			
1-7,8,9			•	
1-5	•			
3-4	•	•		
3-5	•	•		•
3-9			•	
4-5	•	•	•	•
4-7,8	•	•	•	•
4-15	•	•	•	
5-7,8	•	•	•	•
5-13			•	
5-15	•			•
7,8-13			•	
7,8-15	•			
7,8-18	•		•	
7,8-20,21			•	
11-13			•	
11-15	•			•
11-16	•	•		
11-17				•
13-16 (very weak)	•	•		
13-17	•	•	•	
13-18	•			
13-20,21		•	•	
16-18		•		•
16-19	•			
16-20,21	•			•
17-18		•		
17-19	•			
17-20,21	•	•		

Intramolecular NOEs, cont'd

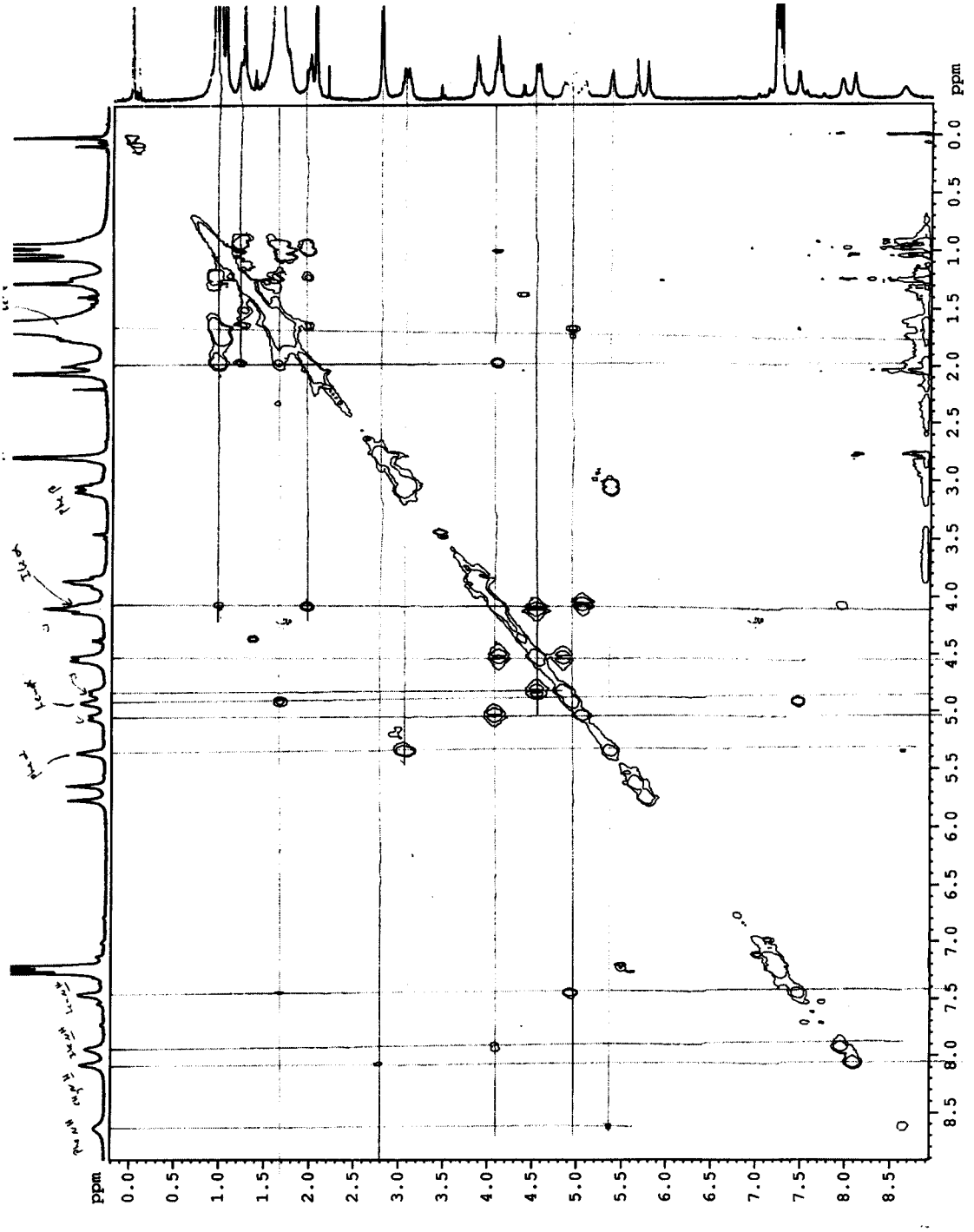
	@-Tide 9			Peptide 16
% $CD_3OH/CDCl_3$:	1%	2.5%	10%	1%
17-27				•
18-20,21	•	•	•	•
18-27		•		
18-31		•	•	
19-20,21	•	•	•	
20,21-27				•
23-27		•	•	
23-28	•			
23-29				•
23-32,33				•
25-29		•	•	
25-32,33	•			
25-36	•			
28-30	•			•
28-32,33	•			
29-30		•		•
29-32,33		•		•
29-35	•		•	
30-32,33	•	•	•	•
30-35				•
30-36	•			
31-32,33	•	•		•
32,33-36	•	•	•	
35-36	•		•	•



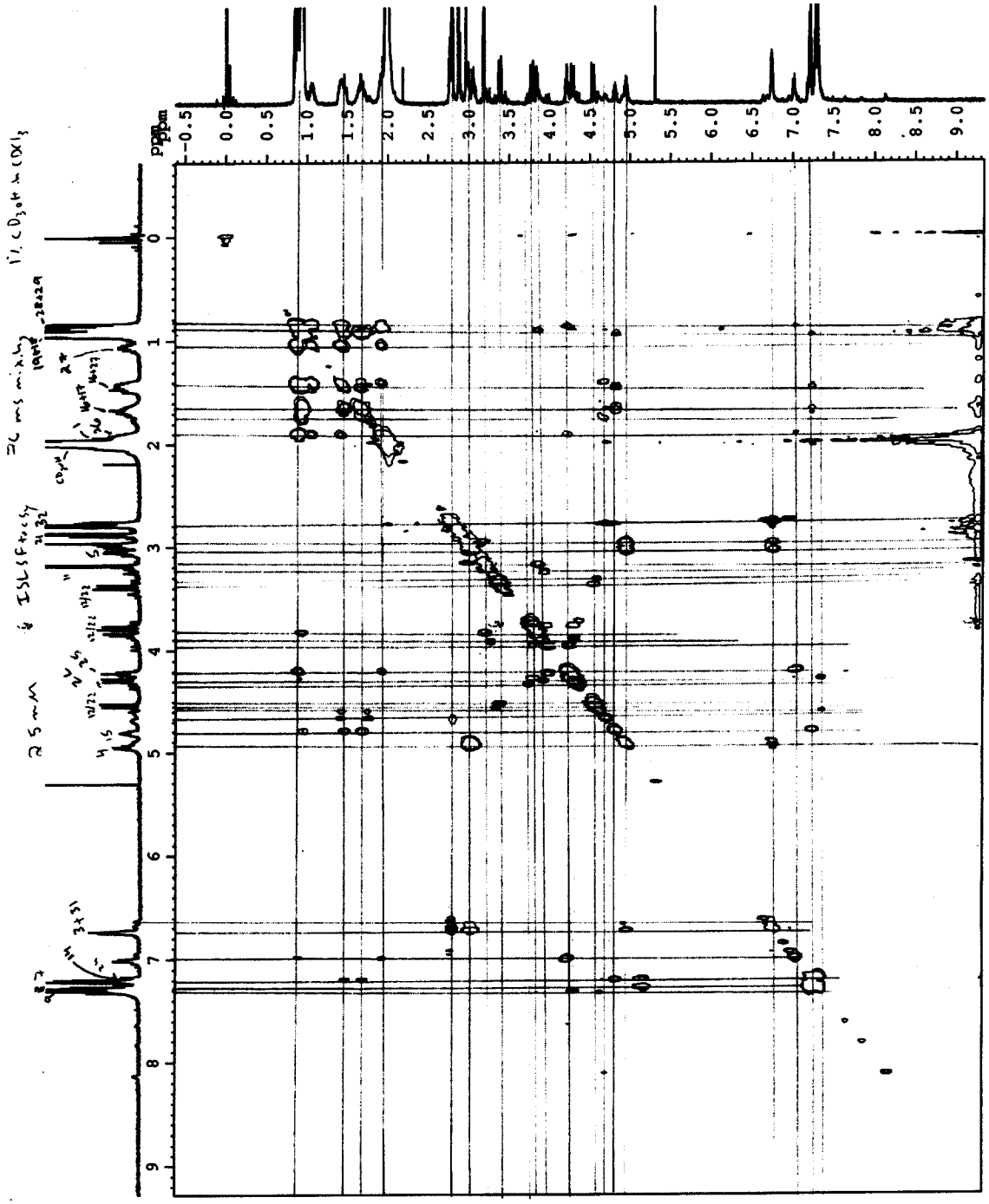
Intermolecular NOEs

	@-Tide 9	
% $CD_3OH/CDCl_3$:	1%	2.5%
1-32,33	•	
1-35	•	
1-36	•	•
5-28	•	•
5-30	•	•
5-32,33	•	
7,8-23	•	
7,8-30	•	
7,8-31	•	
7,8-32,33	•	•
11-23	•	•
11-28		•
16-23	•	

TOCSY Spectrum of @-Tide 9: concentration: 20 mM
solvent: 1% $CD_3OH/CDCl_3$
temperature: 20 °C
mixing time: 48 ms

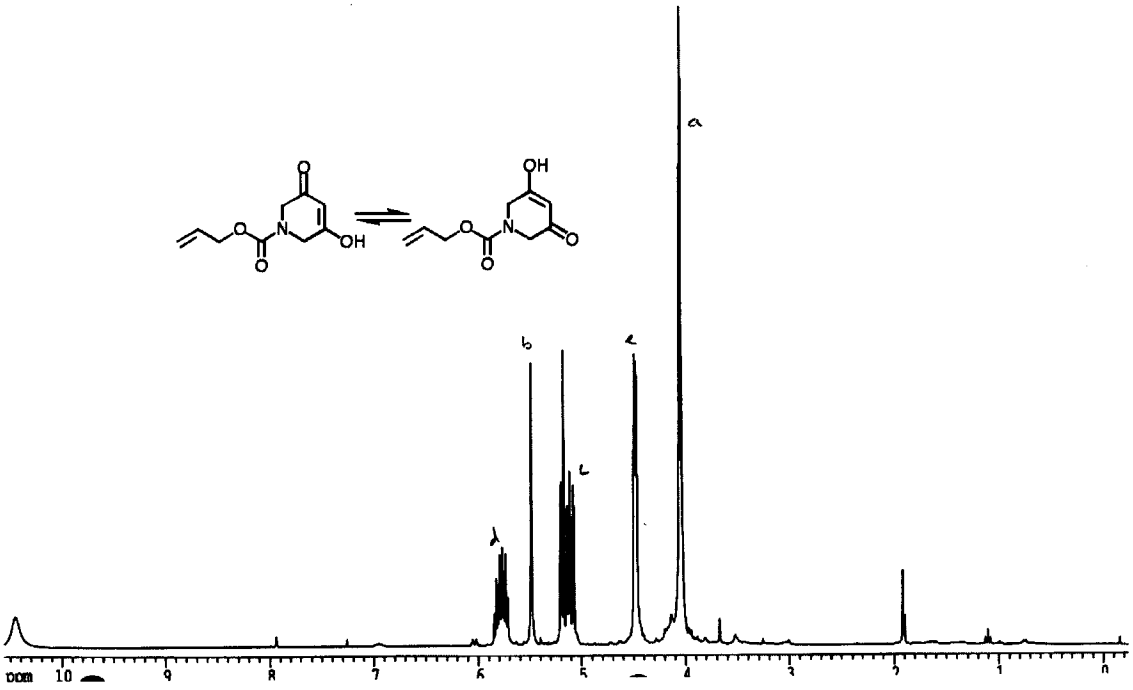


TOCSY Spectrum of Peptide **16**: concentration: 25 mM
solvent: 1% $CD_3OH/CDCl_3$
temperature: 20 °C
mixing time: 76 ms

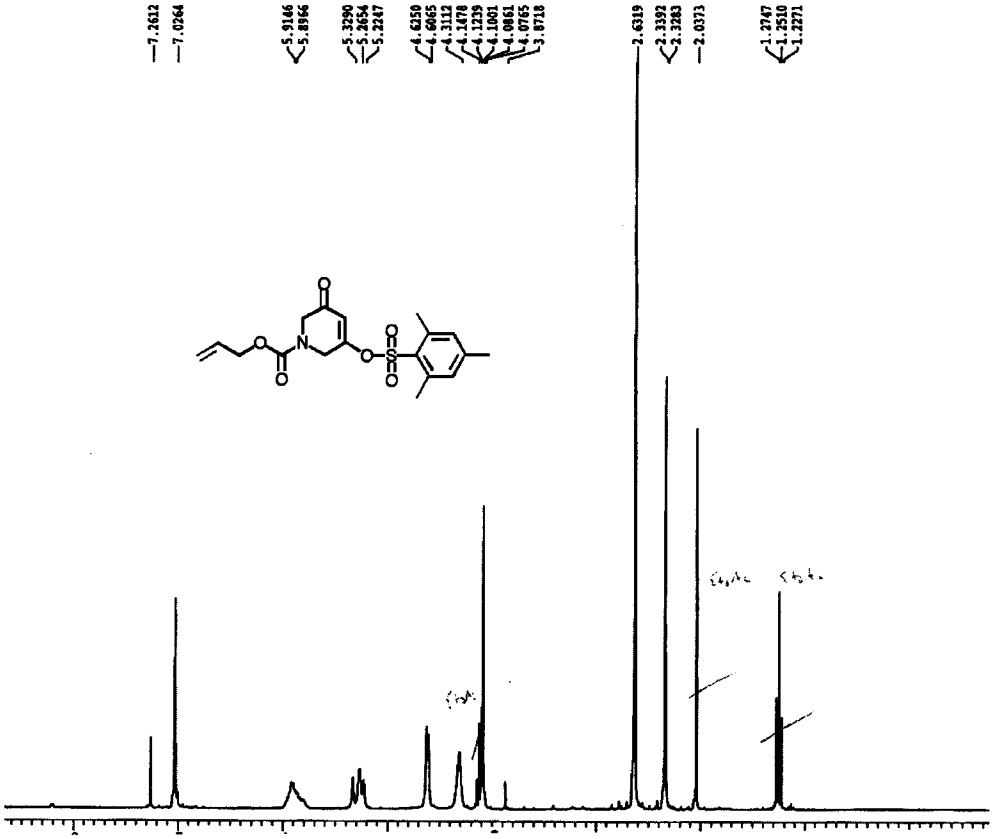


^{1}H NMR spectra (solvent is $CDCl_3$ unless otherwise indicated)

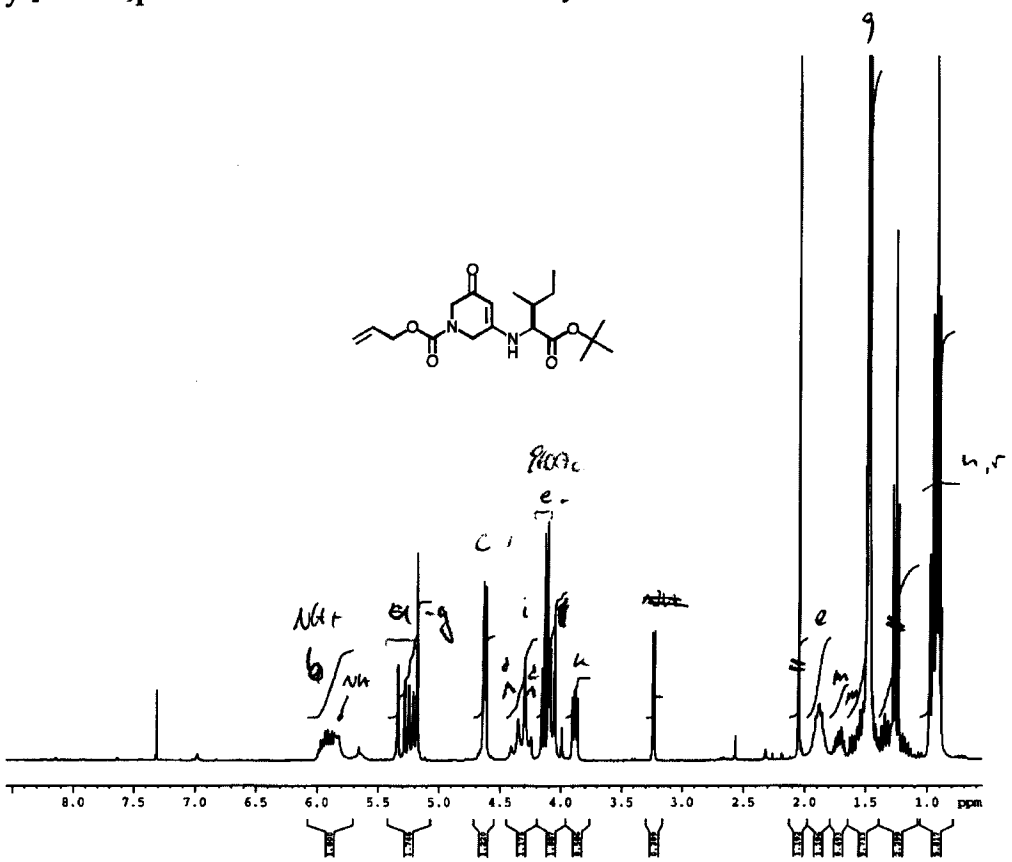
Prop-2-enyl 5-Hydroxy-3-oxo-1,2,6-trihydropyridine-1-carboxylate (2).



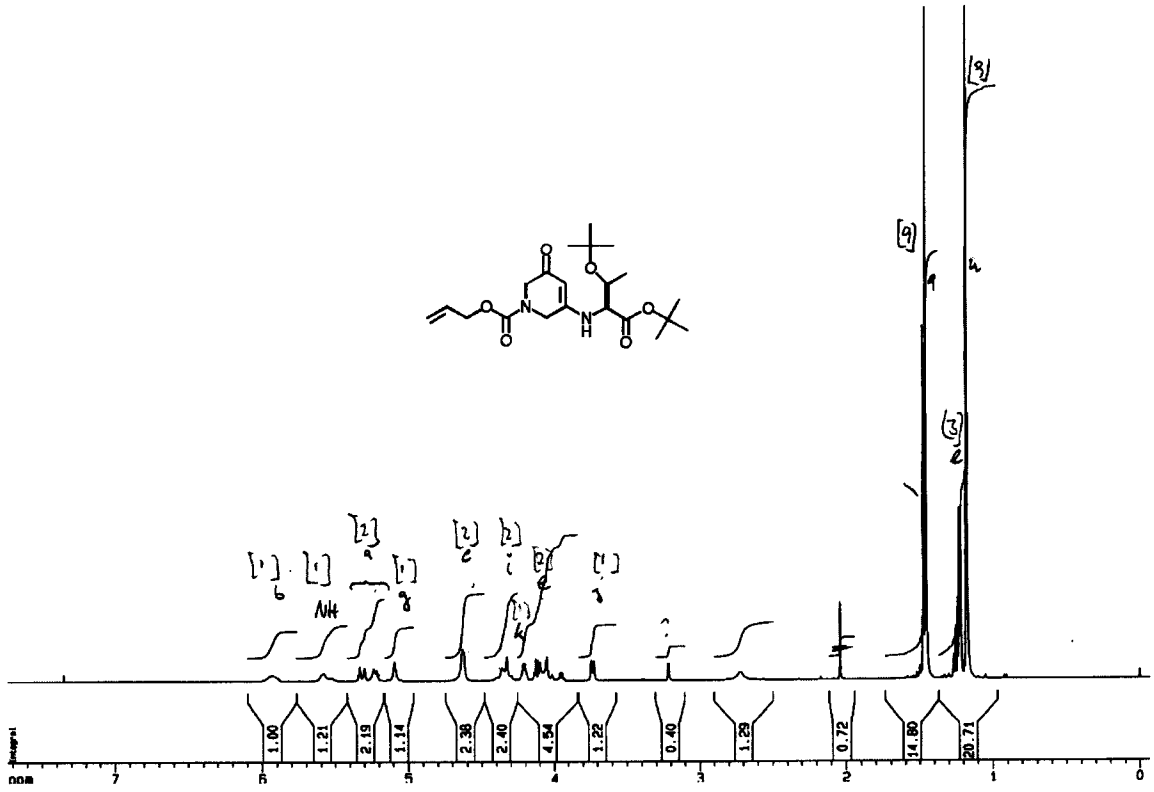
Prop-2-enyl 3-Oxo-5-[(2,4,6-trimethylphenyl)sulfonyloxy]-1,2,6-trihydropyridine-1-carboxylate (3).



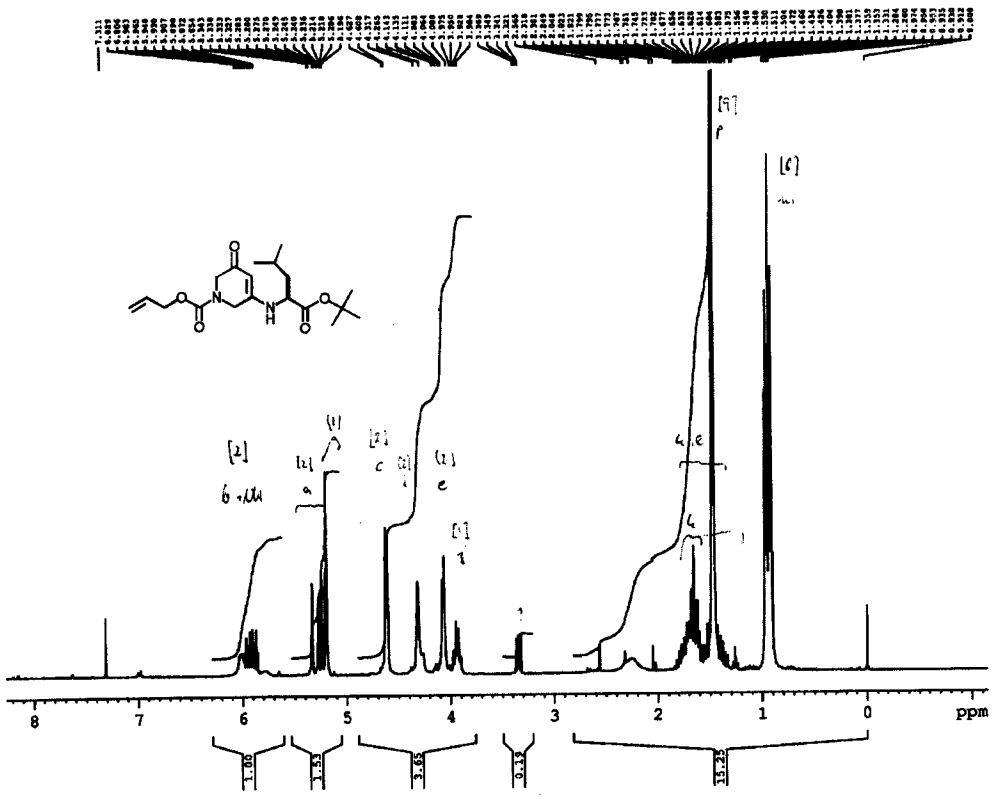
tert-Butyl (2*S*,3*S*)-3-Methyl-2-{[5-oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}pentanoate (Alloc-Ach-Ile *t*-Butyl Ester, 4a).



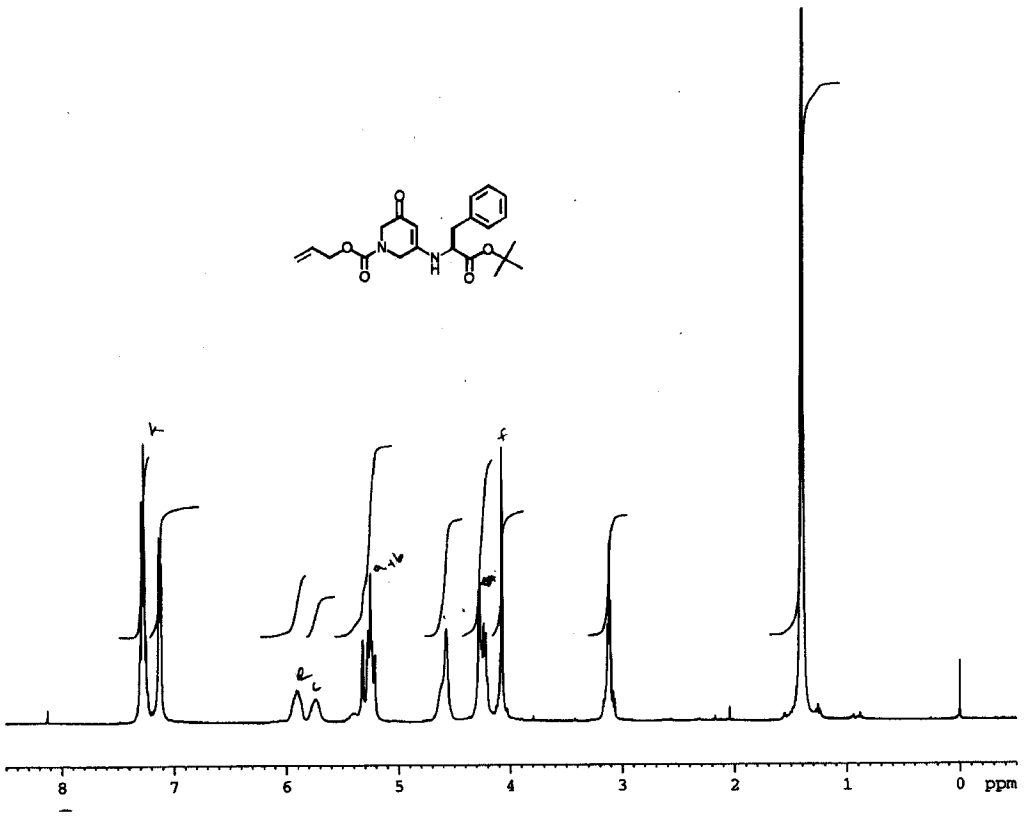
tert-Butyl (2*S*,3*S*)-3-*tert*-Butoxy-2-{[5-oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]-amino}butanoate (Alloc-Ach-Thr(*O*-*t*Bu) *t*-Butyl Ester, 4b).



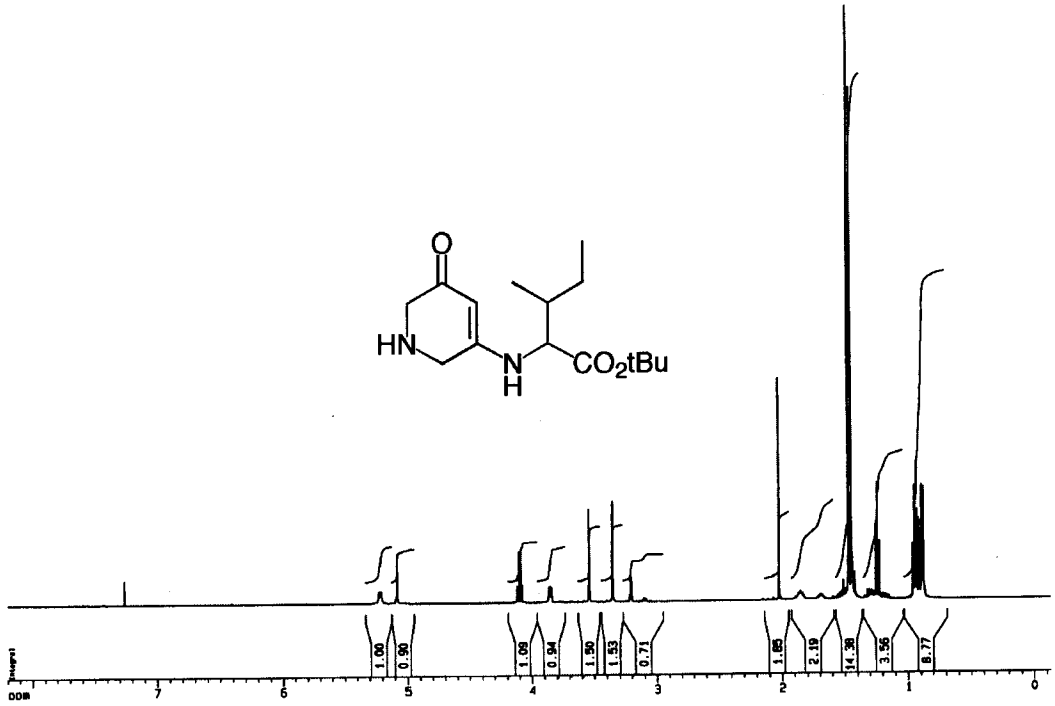
***tert*-Butyl (2*S*)-4-Methyl-2-{[5-oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}-pentanoate (Alloc-Ach-Leu *t*-Butyl Ester, 4c).**



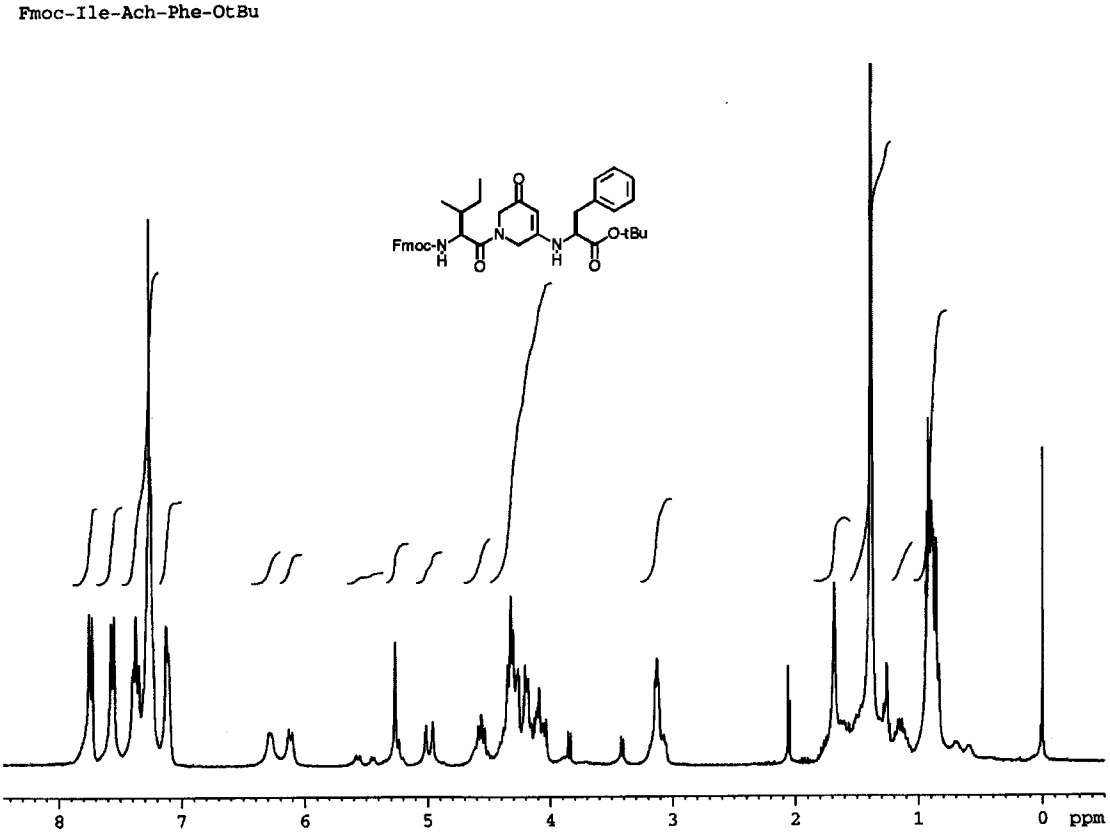
***tert*-Butyl (2*S*)-2-{[5-Oxo-1-(prop-2-enyloxycarbonyl)-1,2,6-trihydropyridyl]amino}-3-phenyl-propanoate (Alloc-Ach-Phe *t*-Butyl Ester, 4d).**



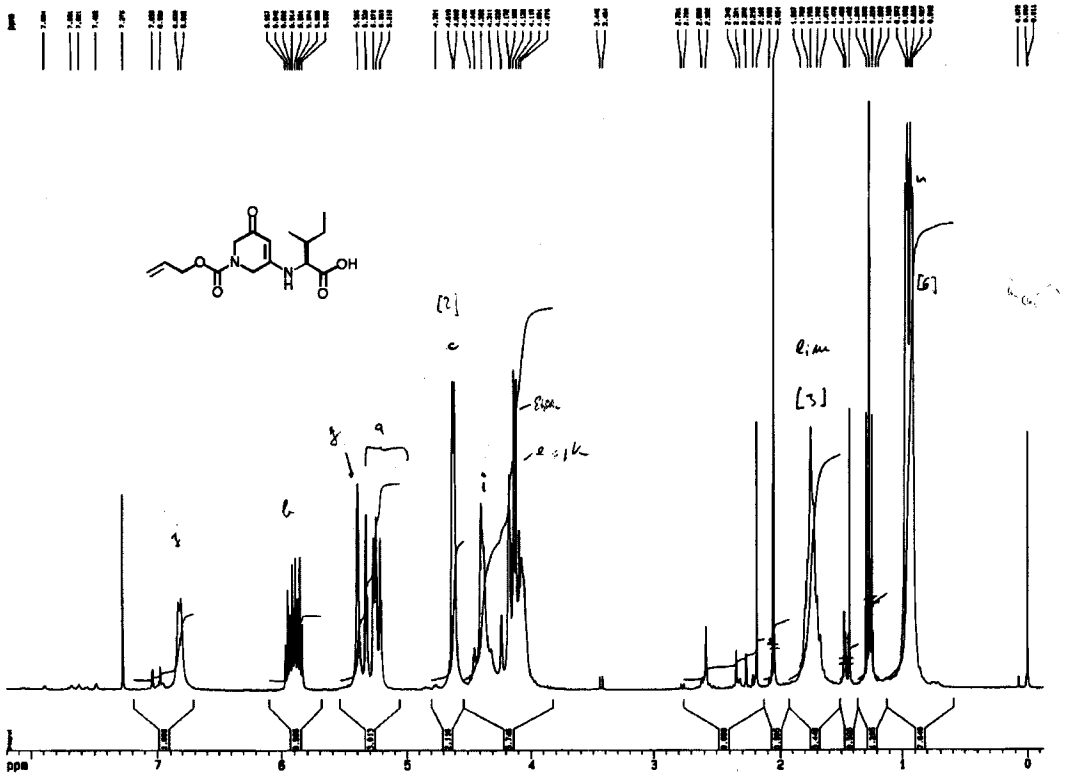
***tert*-Butyl (2*S*,3*S*)-3-Methyl-2-[(5-oxo-1,2,6-trihydro-3-pyridyl)amino]pentanoate (Ach-Ile *t*-Butyl Ester).**



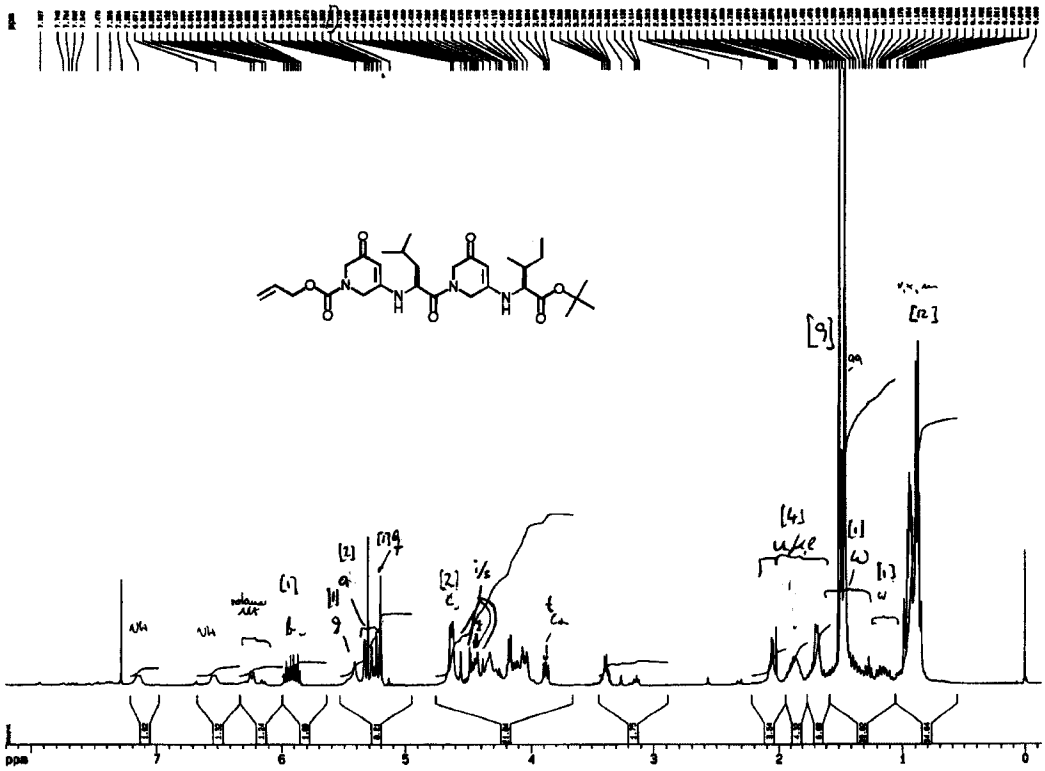
Fmoc-Ile-Ach-Phe *t*-Butyl Ester (5).



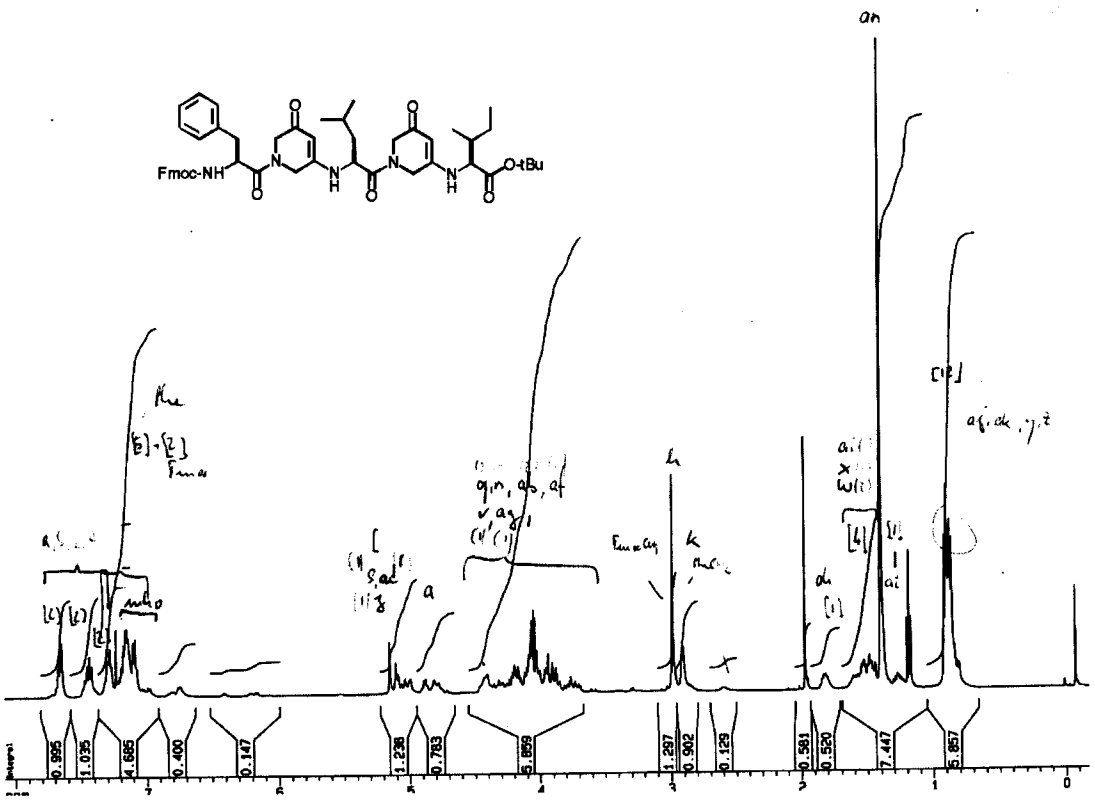
(2*S*)-4-Methyl-2-[[5-oxo-1-(prop-2-enyloxycarbonyl)(3-oxo-1,2,6-trihydropyridyl)]amino]pentanoic acid (Alloc-Ach-Leu, 6).



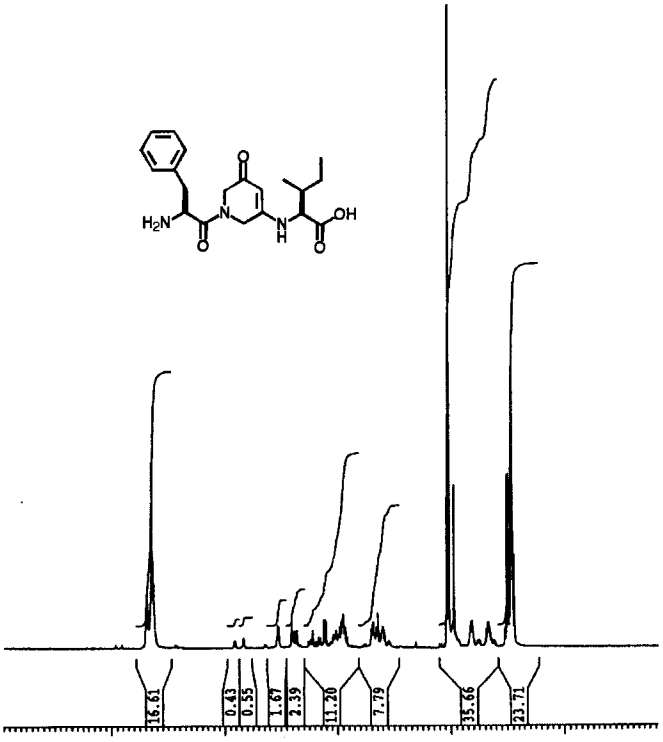
Alloc-Ach-Leu-Ach-Ile *t*-Butyl Ester (7).



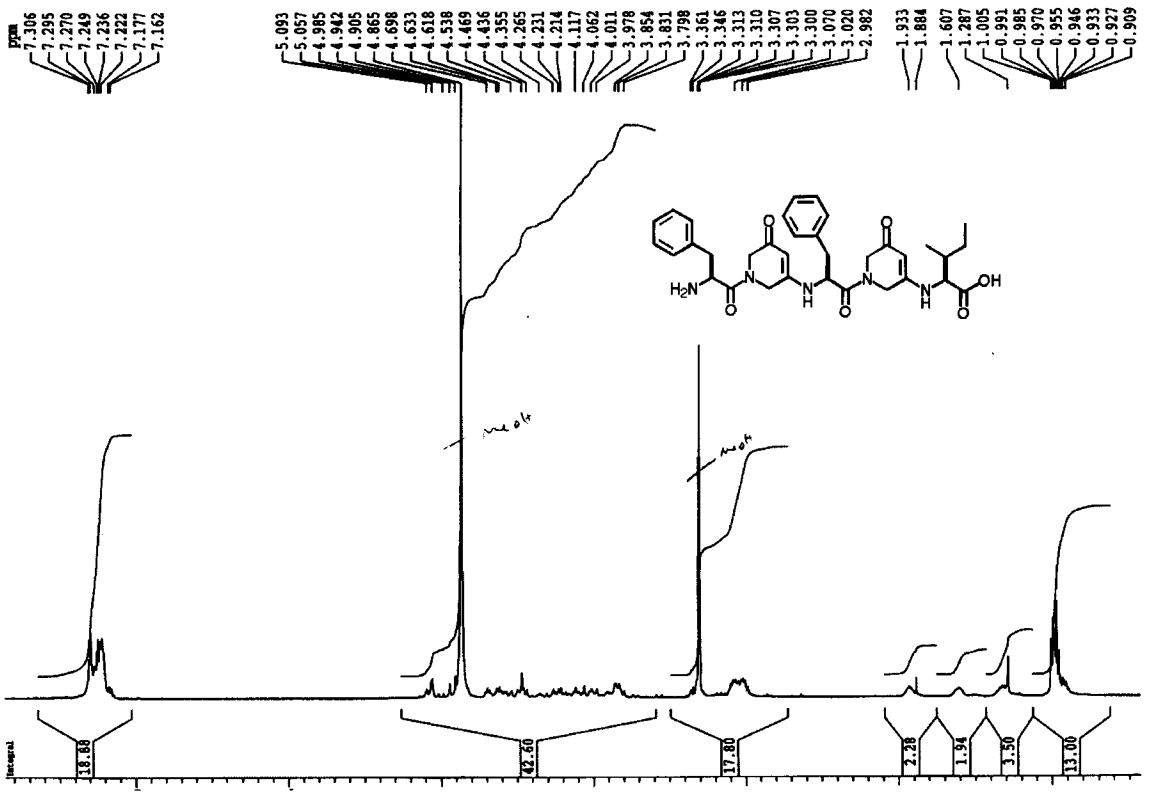
Fmoc-Phe-Ach-Leu-Ach-Ile *t*-Butyl Ester (8). (solvent: 5% $CD_3OH/CDCl_3$)



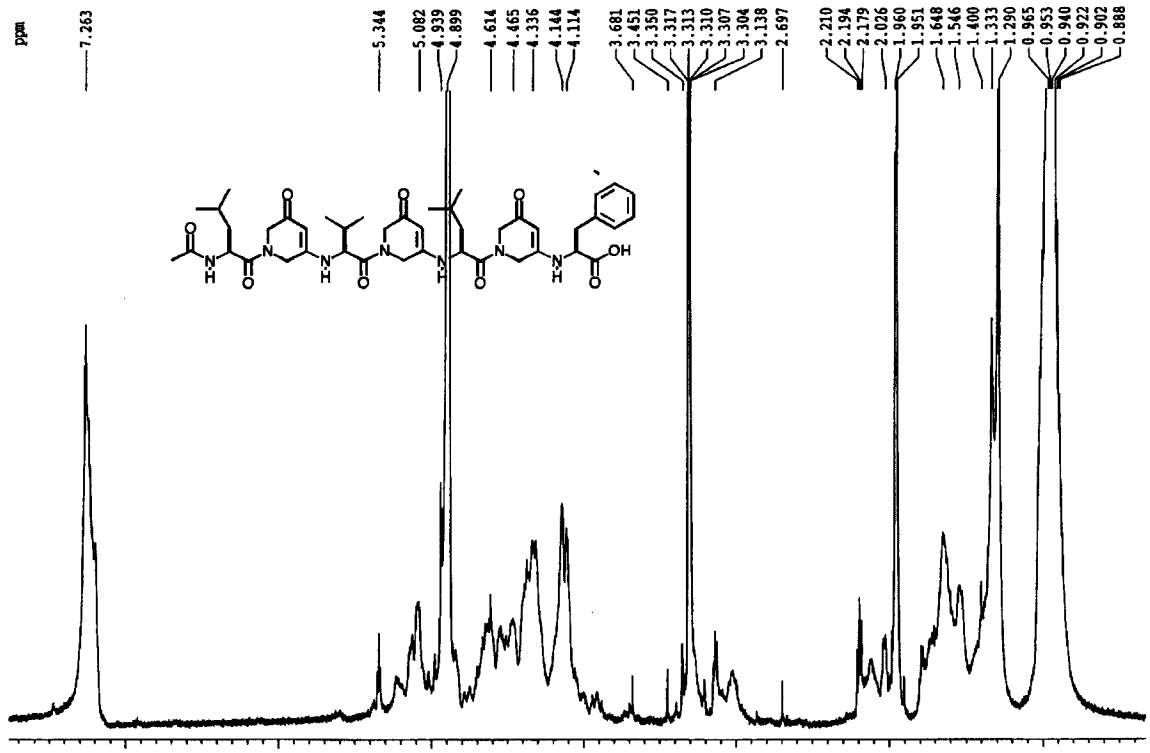
Phe-Ach-Ile (13). (solvent: acetone-d_6)



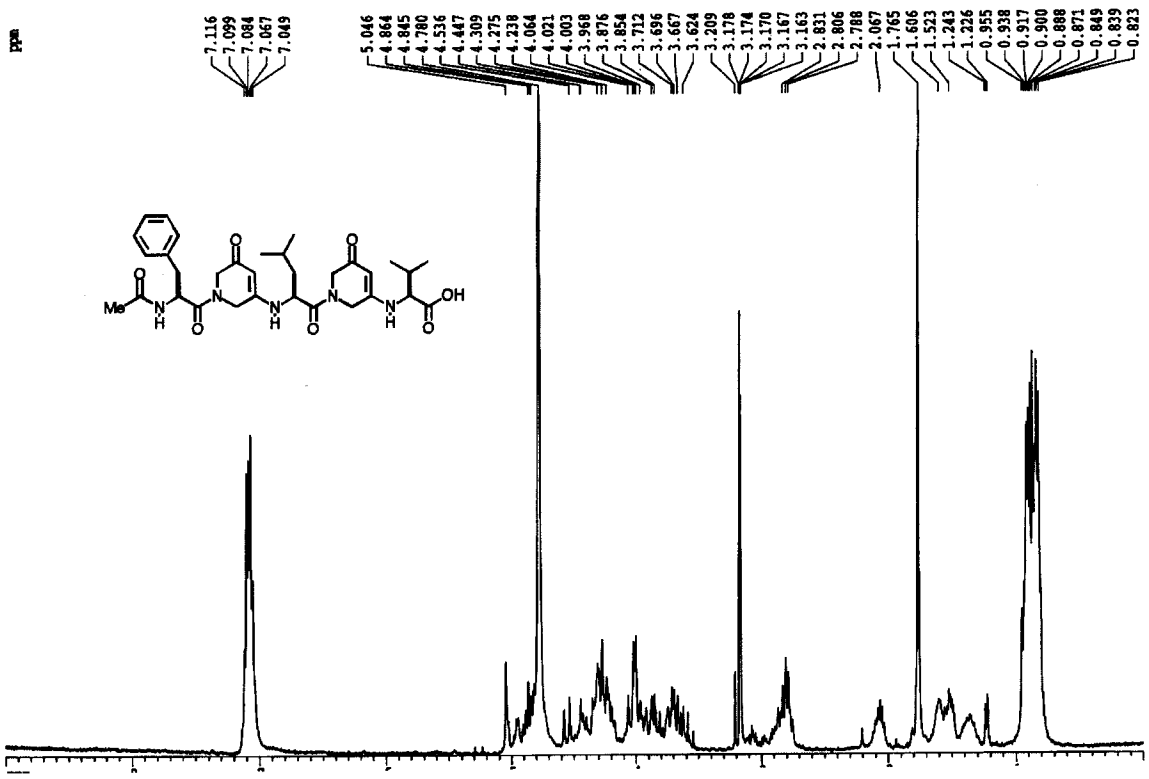
Phe-Ach-Phe-Ach-Ile (15). (solvent: methanol-d_4)



Ac-Leu-Ach-Val--Ach-Leu-Ach-Phe (17). (solvent: methanol-d_4)



Ac-Phe-Ach-Leu-Ach-Val (18). (solvent: methanol-d_4)



Ac-Leu-Ach-Val (19). (solvent: methanol-d_4)