

Supplementary material for Lee *et al.* (2001) *Proc. Natl. Acad. Sci. USA* **98** (4), 1427–1431. (10.1073/pnas.031384998)

## Experimental Section

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Gemini-300, a Varian Mercury-400, or a Varian Unity-500 spectrometer. Chemical shifts are reported in ppm from tetramethylsilane on the  $\delta$  scale. Infrared and mass spectra were recorded on Nicolet 680 DSP and Kratos MS 80RFT spectrometers, respectively. Melting points were taken on an Electrothermal melting point apparatus and are uncorrected. Optical rotation was taken on JASCO DIP-370 digital polarimeter. Thin-layer chromatography was performed with Whatman reagents 0.25-mm silica gel 60-F plates. All other reagents were purchased from Aldrich Chemical, Milwaukee, WI.

Stannane **3** (1, 2) and triflate **4** (3) were prepared by literature procedures. The coupling reaction used for **3** and **4** to give **5** was by a modified procedure after Farina *et al.* (4, 5).

**Compound 5.** To the solution of triflate **4** (3.33 g, 5.68 mmol) in dry *N*-methylpyrrolidinone (NMP, 25 mL) under an argon atmosphere was added tri(2-furyl)phosphine (53 mg, 0.23 mmol, 0.04 equiv.), tris(dibenzylideneacetone)dipalladium (52 mg, 0.11 mmol of Pd, 0.02 equiv Pd), and zinc chloride (1.50 g, 11.36 mmol). After stirring at room temperature for 15 min, the reaction mixture was treated with stannane **6** (3.52 g, 6.82 mmol) in NMP (30 mL) via cannula. The resultant solution was stirred at 40°C overnight. The solution was diluted with EtOAc and washed with water (three times), and brine, then dried over  $\text{Na}_2\text{SO}_4$ . The solvent was evaporated and the residue was taken up in acetonitrile and was further washed with hexanes (three times). The acetonitrile solution was then concentrated under vacuum and was purified by flash column chromatography (silica gel, 3:2 hexanes/ethyl acetate) to yield the title compound as a yellow amorphous powder (3.50 g, 93%). mp. 87–88°C;  $^1\text{H}$ NMR ( $\text{CDCl}_3$ ):  $\delta$  1.409–1.632 (overlapping s, 15H), 3.447–3.694 (m, 5H), 3.790 (s, 3H), 4.040 (dd, 1H,  $J = 6.0, 9.3$  Hz), 4.332 and 4.516 (two sets of bs, 1H), 4.923 (bs, 1H), 5.147 (bs, 2H), 5.767 (dd, 1H,  $J = 4.5, 9.0$  Hz), 5.870 (bs, 1H), 6.128 (bs, 1H), 6.868 (d, 2H,  $J = 8.4$  Hz), 7.001 (unresolved dd, 1H), 7.234–7.381 (m, 7H);  $^{13}\text{C}$ NMR ( $\text{CDCl}_3$ ):  $\delta$  14.32, 14.92, 18.24, 24.30, 25.27, 27.41, 27.55, 27.81, 28.55, 29.15, 43.99, 55.97, 58.27, 59.54, 59.93, 60.19, 68.50, 68.95, 80.79, 94.96, 114.64, 124.53, 126.14, 126.54, 127.10, 127.65, 128.40, 129.87, 130.15, 131.37, 133.55, 133.83, 134.40, 160.56, 162.16, 165.09, 171.88; IR (KBr): 3288, 1781, 1722, 1693, 1666  $\text{cm}^{-1}$ ; FAB HRMS (in *p*-nitrobenzylalcohol): 664.2697 ( $\text{M}^+ + \text{H}$ , calcd for  $\text{C}_{35}\text{H}_{42}\text{N}_3\text{O}_8\text{S}$  664.2692).

**Compound 6.** To a solution of **5** (4.5 g, 6.79 mmol) in anhydrous MeOH (100 mL) was added Pd/C (10% Pd, 4.5 g). The mixture was stirred under  $\text{H}_2$  for 20h. After filtration, the catalyst was washed with MeOH and the combined MeOH solution was evaporated *in vacuo*. The residue was purified by column chromatography (silica gel, 100:1  $\text{CHCl}_3/\text{MeOH}$ ) to give the product as a white solid (2.73 g, 64%). mp. 182–183°C;

<sup>1</sup>HNMR (CDCl<sub>3</sub>): δ 1.479 (s, 9H), 1.636–1.709 (m, 2H), 2.140 (001 (m, 1H), 2.707 (m, 1H), 2.918 (bs, 1H), 3.339 (dd, 2H, *J* = 18.2, 65.4 Hz), 3.574 (bs, 3H), 3.644 (dd, 2H, *J* = 15.8, 19.0 Hz), 3.813 (s, 3H), 4.908 (d, 1H, *J* = 4.8 Hz), 5.050 (bd, 1H, *J* = 6.0 Hz), 5.181 (dd, 2H, *J* = 11.8, 22.2 Hz), 5.755 (dd, 1H, *J* = 4.8, 8.8 Hz), 6.567 (bd, 1H, *J* = 8.8 Hz), 6.900 (d, 2H, *J* = 8.8 Hz), 7.275–7.387 (m, 7H); <sup>13</sup>CNMR (CDCl<sub>3</sub>): δ 29.55, 29.87, 31.21, 44.25, 44.34, 53.73, 56.41, 58.56, 60.22, 66.59, 68.74, 80.75, 115.09, 123.78, 128.10, 128.72, 130.20, 130.56, 131.74, 134.97, 136.62, 157.62, 160.95, 163.04, 165.86, 172.53; IR (KBr): 3512–3335, 1778, 1705, 1683, 1662 cm<sup>-1</sup>; FAB HRMS (in *p*-nitrobenzylalcohol): 626.2543 (M<sup>+</sup> + H, calcd for C<sub>32</sub>H<sub>40</sub>N<sub>3</sub>O<sub>8</sub>S 626.2536).

**Compound 7.** To the suspension of Dess-Martin periodinane (403 mg, 0.95 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (20 mL) under an atmosphere of argon was added a solution of the alcohol **6** (495 mg, 0.79 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (10 mL) via cannula. The resultant mixture was stirred at room temperature overnight. The mixture was then diluted with CH<sub>2</sub>Cl<sub>2</sub> (50 mL) and mixed with a 20-mL solution of aqueous saturated NaHCO<sub>3</sub> containing 2 g of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. After stirring at room temperature for 20 min, the layers were separated and the organic phase was washed with aqueous saturated NaHCO<sub>3</sub>, water, and brine, then was dried over Na<sub>2</sub>SO<sub>4</sub>. The solvent was removed *in vacuo* to give the desired product (463 mg, 94%). mp. 75°C; <sup>1</sup>HNMR (CDCl<sub>3</sub>): δ 1.491 (s, 9H), 1.894 (unresolved m, 1H), 2.047–2.096 (m, 2H), 2.618–2.655 (m, 1H), 3.337 (dd, 2H, *J* = 18.4, 62.8 Hz), 3.649 (dd, 2H, *J* = 16.0, 20.0 Hz), 3.818 (s, 3H), 4.161 (unresolved dd, 1H), 4.907 (d, 1H, *J* = 5.2 Hz), 5.178 (dd, 2H, *J* = 11.8, 19.6 Hz), 5.491 (bd, 1H, *J* = 6.4 Hz), 5.777 (dd, 1H, *J* = 4.8, 8.8 Hz), 6.441 (bd, 1H, *J* = 8.8 Hz), 6.902 (d, 2H, *J* = 8.8 Hz), 7.275–7.393 (m, 7H), 9.505 (s, 1H); <sup>13</sup>CNMR (CDCl<sub>3</sub>): δ 28.61, 29.45, 29.55, 30.19, 44.35, 56.40, 58.46, 60.24, 60.67, 68.89, 81.36, 115.11, 124.41, 127.97, 128.77, 130.24, 130.55, 131.79, 134.61, 134.91, 156.79, 161.00, 162.93, 165.72, 172.46, 201.00; IR (KBr): 3482–3327, 1778, 1716, 1689, 1686, 1636 cm<sup>-1</sup>; FAB HRMS (in *p*-nitrobenzylalcohol): 624.2394 (M<sup>+</sup> + H, calcd for C<sub>32</sub>H<sub>38</sub>N<sub>3</sub>O<sub>8</sub>S 624.2379).

**Compound 8.** To the solution of the aldehyde **7** (2.3 g, 3.69 mmol) in acetone (75 mL) at 0 °C was added dropwise 1 M Jones reagent (4.4 mL, 4.4 mmol). The resultant mixture was stirred for 30 min at 0 °C. 2-Propanol (5 mL) was added to the mixture and stirring was continued for an additional 30 min. The solvent was removed *in vacuo* and the residue was taken up in EtOAc and washed with water (twice) and brine, then was dried over MgSO<sub>4</sub>. The solvent was removed *in vacuo* to give the crude product as a yellowish solid (2.18 g, 92%), which was used in the next step without further purification. <sup>1</sup>HNMR (CDCl<sub>3</sub>): δ 1.446 (s, 9H), 1.957 (m, 2H), 2.135 (m, 1H), 2.753 (m, 1H), 3.274 (dd, 2H, *J* = 18.2, 56.2 Hz), 3.594 (bs, 2H), 3.760 (s, 3H), 4.272 (bs, 1H), 4.852 (d, 1H, *J* = 4.4 Hz), 5.130 (dd, 2H, *J* = 11.8, 24.0 Hz), 5.533 (bd, 1H, *J* = 7.6 Hz), 5.689 (dd, 1H, *J* = 4.8, 8.4 Hz), 6.660 (bd, 1H, *J* = 7.6 Hz), 6.845 (d, 2H, *J* = 8.4 Hz), 7.225–7.304 (m, 7H); <sup>13</sup>CNMR (CDCl<sub>3</sub>): δ 28.36, 29.45, 30.47, 42.88, 53.13, 55.23, 57.51, 59.13, 60.51, 67.60, 80.21, 113.95, 123.13, 126.97, 127.41, 128.90, 129.35, 130.56, 134.06, 134.64, 155.90, 159.76, 161.79, 164.78, 172.10, 175.15.

**Compound 10.** To the solution of the acid **8** (453 mg, 0.71 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (25 mL) was added HOBt (96 mg, 0.71 mmol), DCC (143 mg, 0.69 mmol) and the dipeptide

D-alanyl-D-alanine *tert*-butyl ester (**9**) (**6**) (230 mg, 1.06 mmol). The resultant mixture was stirred at room temperature overnight. The precipitated DCU was filtered out and the filtrate was concentrated *in vacuo*. EtOAc (25 ml) was added to precipitate another crop of DCU, which was filtered. The filtrate was washed with 5% NaHCO<sub>3</sub>, water, 1 M HCl, water, and brine, then dried over MgSO<sub>4</sub>. The solvent was removed *in vacuo* and the residue was purified by column chromatography (silica gel, 50:1, then 30:1 CH<sub>2</sub>Cl<sub>2</sub>/2-propanol) to give the product as a yellow solid (408 mg, 69%). mp. 128–130°C; <sup>1</sup>HNMR (CD<sub>3</sub>OD): δ 1.373–1.386 (overlapping d, 6H), 1.451 and 1.461 (two overlapping s, 18H), 1.885 (m, 2H), 2.385 (m, 1H), 2.592 (m, 1H), 3.367–3.632 (two overlapping dd, 4H, *J* = 18.0, 103.0, 14.5, 26.5 Hz), 3.786 (s, 3H), 3.941 (dd, 1H, *J* = 6.5, 12.0 Hz), 4.249 (m, 1H), 4.411 (m, 1H), 5.019 (d, 1H, *J* = 5.0 Hz), 5.178 (dd, 2H, *J* = 12.0, 49.5 Hz), 5.644 (d, 1H, *J* = 5.0 Hz), 6.900 (d, 2H, *J* = 8.5 Hz), 7.219–7.353 (m, 7H); <sup>13</sup>CNMR (CD<sub>3</sub>OD): δ 16.58, 16.92, 17.46, 27.52, 28.09, 28.61, 30.01, 30.97, 42.45, 55.01, 55.41, 58.36, 59.91, 67.77, 78.78, 80.12, 81.84, 81.99, 114.20, 123.74, 127.27, 128.03, 128.85, 129.50, 130.78, 135.49, 135.73, 157.04, 160.63, 162.79, 165.45, 172.47, 173.59, 173.79, 173.84; IR (KBr): 3504–3313, 1781, 1719, 1685, 1659, 1615 cm<sup>-1</sup>; FAB HRMS (in *p*-nitrobenzylalcohol): 838.3718 (M<sup>+</sup> + H, calcd for C<sub>42</sub>H<sub>56</sub>N<sub>5</sub>O<sub>11</sub>S 838.3697).

**Compound 12.** To a mixture of (*S*)- $\alpha$ -*tert*-butyl  $\delta$ -benzyl glutamate (**7**) (1.21 g, 4.12 mmol) and triethylamine (0.56 ml, 4.12 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (25 ml) was added acetic anhydride (0.50 ml, 4.94 mmol) and the resultant solution was stirred at room temperature overnight. More CH<sub>2</sub>Cl<sub>2</sub> was added to dilute the solution and the organic phase was washed with 1 M HCl, water, and brine, then was dried over MgSO<sub>4</sub>. The solvent was removed *in vacuo* and the residue was purified by flash column chromatography (silica gel, 2:1 EtOAc/hexanes) to give (*S*)- $\alpha$ -*tert*-butyl  $\delta$ -benzyl *N*-acetylglutamate as a colorless oil (1.3 g, 94%). [ $\alpha$ ]<sub>D</sub><sup>25</sup> -17.5 (*c* 2.43, CHCl<sub>3</sub>); <sup>1</sup>HNMR (CDCl<sub>3</sub>): δ 1.449 (s, 9H), 1.962 (overlapping s and m, 4H), 2.191 (m, 1H), 2.421 (m, 2H), 4.512, (unresolved ddd, 1H), 5.106 (s, 2H), 6.169 (bd, 1H, *J* = 6.8 Hz), 7.310–7.353 (m, 5H); <sup>13</sup>CNMR (CDCl<sub>3</sub>): δ 23.22, 27.76, 28.03, 30.40, 52.16, 66.57, 82.58, 128.32, 128.37, 128.64, 135.79, 169.98, 171.22, 172.80; IR (neat): 3292 br, 1738, 1734, 1660 cm<sup>-1</sup>; EI HRMS 279.1109 (M<sup>+</sup> - C<sub>4</sub>H<sub>8</sub>), calcd for C<sub>14</sub>H<sub>17</sub>NO<sub>5</sub> 279.1107). This product (628 mg) was then dissolved in EtOAc (20 ml) and Pd/C (5%, Degussa type, 70 mg) was added. The mixture was stirred under a hydrogen atmosphere at room temperature for 2 h. The Pd/C was filtered out and the solvent of the filtrate was removed *in vacuo* to give the crude product as a white solid (426 mg), which was used without further purification. mp. 102–104°C; [ $\alpha$ ]<sub>D</sub><sup>25</sup> = -18.1 (*c* 1.2, CHCl<sub>3</sub>); <sup>1</sup>HNMR (CDCl<sub>3</sub>): δ 1.453 (s, 9H), 1.915 (m, 1H), 2.023 (s, 3H), 2.170 (m, 1H), 2.402 (m, 2H), 4.524 (unresolved ddd, 1H), 6.502 (bs, 1H), 10.714 (bs, 1H); <sup>13</sup>CNMR (CDCl<sub>3</sub>): δ 23.05, 27.76, 28.01, 30.19, 52.20, 82.89, 170.89, 171.30, 176.95; IR (KBr): 3364, 1716, 1637, 1556 cm<sup>-1</sup>; EI HRMS 172.0612 (M<sup>+</sup> - C<sub>4</sub>H<sub>9</sub>O), calcd for C<sub>7</sub>H<sub>10</sub>NO<sub>4</sub> 172.0610).

**Compound 13.** To a solution of **10** (130 mg, 0.16 mmol) and NaI (47 mg, 0.31 mmol) in anhydrous acetonitrile (2 ml) at -20°C was added chlorotrimethylsilane (40  $\mu$ l, 0.31 mmol). The cooling bath was removed and the reaction mixture was stirred at room temperature for 4 h. A few drops of MeOH were added to quench the reaction and the solvent was removed *in vacuo*. The residue was redissolved in CH<sub>2</sub>Cl<sub>2</sub> and washed with

10% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, water, and brine, then was dried over MgSO<sub>4</sub>. The solvent was removed *in vacuo* to give **11**. This sample was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and to this solution was added HOBt (60 mg, 0.44 mmol), (*S*)- $\alpha$ -*tert*-butyl *N*-acetylglutamate (**12**) (80 mg, 0.33 mmol), and DCC (64 mg, 0.31 mmol). The resultant mixture was stirred at room temperature for 24 h. The precipitated DCU was filtered and the filtrate was washed with 5% NaHCO<sub>3</sub>, water, 1N HCl, water, and brine, then dried over MgSO<sub>4</sub>. The solvent was removed *in vacuo* and the product was purified by column chromatography (silica gel, 20:1, then 10:1 CH<sub>2</sub>Cl<sub>2</sub>/2-propanol) to give the product as a white solid (50 mg, 44% 2 steps). mp 191–193°C; <sup>1</sup>HNMR (CD<sub>3</sub>OD):  $\delta$  1.270–1.454 (overlapping s and d, 24H), 1.840–2.401 (m, 10H), 2.592–2.649 (m, 1H), 3.378–3.624 (two overlapping dd, 4H, *J* = 14.5, 27.0, 18.5, 87.5 Hz), 3.777 (s, 3H), 4.186–4.261 (two overlapping dd, 2H, *J* = 6.0, 7.5, 7.5, 15.0 Hz), 4.288 (dd, 1H, *J* = 4.5, 9.5 Hz), 4.382 (dd, 1H, *J* = 7.5, 14.5 Hz), 5.008 (d, 1H, *J* = 5.0 Hz), 5.147 (dd, 2H, *J* = 11.5, 35.5 Hz), 5.627 (d, 1H, *J* = 4.5 Hz), 6.880 (d, 2H, *J* = 8.5 Hz), 7.213–7.330 (m, 7H); <sup>13</sup>CNMR (CD<sub>3</sub>OD):  $\delta$  16.36, 17.02, 21.46, 27.22, 27.42, 28.22, 29.51, 29.62, 30.24, 31.73, 42.06, 49.08, 49.10, 52.75, 53.74, 54.64, 57.94, 59.47, 67.43, 77.77, 78.03, 78.29, 81.44, 81.87, 113.78, 123.29, 126.86, 127.44, 128.43, 129.05, 130.32, 135.15, 135.56, 160.13, 162.35, 165.01, 171.32, 172.07, 172.13, 172.40, 173.40, 173.64; IR (neat): 3322–3266 br, 1772, 1734, 1718, 1699, 1695, 1683, 1668, 1647, 1636 cm<sup>-1</sup>; FAB HRMS (in *p*-nitrobenzylalcohol + trace NaCl): 987.4116 (M<sup>+</sup> + Na<sup>+</sup>, calcd for C<sub>48</sub>H<sub>64</sub>N<sub>6</sub>O<sub>13</sub>SNa 987.4149).

**Compound 1.** A mixture of **13** (13 mg, 0.013 mmol) and anisole (1 drop) in trifluoroacetic acid (TFA) (0.5 ml) was stirred at 0 °C for 5 min, followed by an additional 25 min at room temperature. The volatiles were removed *in vacuo* and the residue was dissolved in 2:1 MeOH/CHCl<sub>3</sub> (1.5 ml). To this solution was added sodium 2-ethylhexanoate (40 mg, 0.24 mmol) and the resulting mixture was stirred at room temperature for 1 h. The solvent was removed *in vacuo* and the residue was triturated with di-isopropyl ether. The mixture was centrifuged and the supernatant decanted. The pellet was washed with two portions of di-isopropyl ether, along with centrifugation, as described. The final pellet on the bottom of the centrifuge tube was dried *in vacuo* to give an off-white fine powder (10 mg, 97% for two steps). mp 220°C (decomp); <sup>1</sup>HNMR (CD<sub>3</sub>OD):  $\delta$  1.360 (two overlapping d, 6H, two alanyl-CH<sub>3</sub>), 1.851–2.530 (m, 11H, four CH<sub>2</sub> and acetyl-CH<sub>3</sub>), 3.508–3.753 (m, 4H, cephem-H<sub>2</sub> and H<sub>2</sub>, and benzyl CH<sub>2</sub>), 4.145–4.396 (m, 4H, four amino acid  $\alpha$ -H), 4.970 (d, 1H, *J* = 5 Hz, cephem-H<sub>6</sub>), 5.587 (d, 1H, *J* = 5 Hz, cephem-H<sub>7</sub>), 7.220–7.331 (m, 5H, aromatic); <sup>13</sup>CNMR (CD<sub>3</sub>OD):  $\delta$  11.68, 13.28, 16.88, 18.04, 21.81, 22.83, 26.38, 27.15, 29.01, 29.47, 30.26, 32.12, 33.07, 42.02, 49.41, 50.75, 51.33, 53.87, 54.45, 57.62, 59.10, 122.76, 126.73, 128.33, 129.03, 129.75, 135.36, 146.06, 168.77, 171.36, 172.53, 173.19, 173.46, 174.61, 177.36, 178.41; IR (KBr): 3432 br, 1752, 1742, 1736, 1654, 1648, 1637, 1629, 1624, 1617, 1602, 1571 cm<sup>-1</sup>; FAB HRMS (in *p*-nitrobenzylalcohol): 799.1993 (M<sup>+</sup> + H, calcd for C<sub>32</sub>H<sub>38</sub>N<sub>6</sub>O<sub>12</sub>SNa<sub>3</sub> 799.1962).

**Table 2. Thermal factor data reported in Å<sup>2</sup>**

	Average B <sub>iso</sub>	Highest B <sub>iso</sub>	Lowest B <sub>iso</sub>
Protein	12.7	78.0	7.2
Ligand	32.2	77.4	11.7
Solvent	32.1	62.5	8.2

**Table 3. Progress of the refinement as measured by the crystallographic residuals, *R* and *R*<sub>free</sub><sup>†</sup> using data with a 2-σ cutoff in CNS and no σ cutoff in SHELXL**

Refinement stage	<i>R</i>	<i>R</i> <sub>free</sub>
After isotropic refinement in the program CNS, with 411 solvent molecules	0.214	0.230
Same model after isotropic refinement in the program SHELXL	0.187	0.210
After adding solvent molecules selected by SHELXL	0.182	0.207
Same model after anisotropic refinement in SHELXL	0.137	0.172
After adding 35 alternate protein side-chain conformations and ligand	0.127	0.161
After adding hydrogen atoms with good geometry and peaks at least 3σ on   <i>F</i> <sub>o</sub> – <i>F</i> <sub>c</sub>   density maps (10 % of theoretical number of hydrogen atoms)	0.112	0.154
After use of SHELXL recommended weighting scheme	0.113	0.153
Residual using all data, no σ cutoff and no reserve data set for <i>R</i> <sub>free</sub>	0.114	

$${}^{\dagger}R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}, \text{ where } F_o \text{ and } F_c \text{ are observed and calculated structure factors.}$$

*R*<sub>free</sub> = A residual, calculated as above, but using 5% of the data, randomly chosen, that were not included in the refinement.

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