

## SYNTHESIS OF OPTICALLY ACTIVE 2-PIPERIDYLGLYCINE

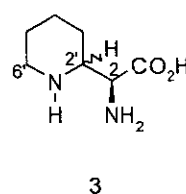
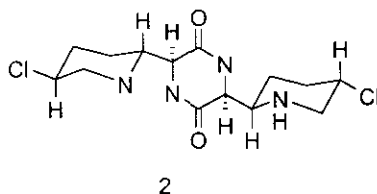
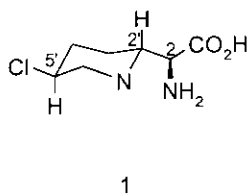
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**Abstract** - Both chiral *threo*- and *erythro*-2-piperidylglycines were synthesized from racemic 2-[(*N*-benzyloxycarbonyl)piperidin-2-yl]ethanoic acid. (4*S*,5*S*)-4-Methyl-5-phenyl-2-oxa-zolidinone was used as a chiral auxiliary in the resolution and azidation.

Many of  $\alpha,\beta$ -diamino acids have displayed various biological activities.<sup>1</sup> Streptolutin [1, 2-amino-2-(5-chloropiperidin-2-yl)ethanoic acid], one of  $\alpha,\beta$ -diamino acids, is the parent amino acid of antitumor agent DKP 593A (2). The agent was isolated from the soil microorganism, *Streptomyces griseoluteus* and reported to be effective against certain solid tumors.<sup>2,3</sup> The antitumor agent has been synthesized as a racemic mixture.<sup>4</sup> Streptolutine has three chiral centers whose configurations are (*R*) at C-2', (*S*) at C-5' and C-2, thus the stereochemical relationship between C-2' and C-5' is *trans*, and that of C-2' and C-2 is *threo*. In the attempted synthesis of streptolutin, the corresponding *erythro* isomer was formed as a major compound.<sup>5</sup>

2-Piperidylglycine [3, 2-amino-2-(piperidin-2-yl)ethanoic acid] has been studied as a model compound in the preparation of analogs of streptolutin and DKP 593A.<sup>6-8</sup> Heating 7-amino-7-carboxy-8-oxo-1-azabicyclo[4.2.0]octane in 5 M HCl gave a 2:1 mixture of *threo*- and *erythro*-2-piperidylglycines.<sup>7</sup> Hydrogenation of ethyl 2-piperidinylidene-*N*-(ethoxycarbonyl)glycinate on 10% Pd/C gave the corresponding *erythro*-glycine derivative.<sup>8</sup>



In the azidation at the C-2 position of a 2-(piperidin-2-yl)ethanoate, an *erythro*-azide is always predominant over a *threo* isomer.<sup>9</sup> In the meantime, a few compounds related to 2-piperidylglycine were

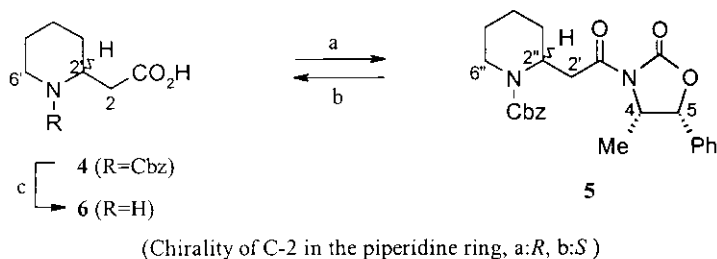
optically resolved. Pig liver esterase catalyzed enantioselectively the hydrolysis of racemic methyl 2-(*N*-acetylpiperidin-2-yl)ethanoate or acetate of 2-(*N*-acetylpiperidin-2-yl)ethan-1-ol to give the corresponding acid or alcohol in 7-24% ee.<sup>10</sup> 2-Piperidineethanol was also resolved through recrystallization of *d*-10-camphorsulfonate salt.<sup>11</sup>

Evans chiral imide enolates have been used in alkylation, aldol condensation and azidation.<sup>12,13</sup> So we thought that a chiral auxiliary may be utilized to obtain chiral 2-(piperidin-2-yl)ethanoic acid from the racemate and to introduce amino group diastereoselectively at the C-2 position.<sup>9</sup> In this note, we described the synthesis of chiral *threo*- and *erythro*-2-piperidylglycines using (4*S*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone as a chiral auxiliary.<sup>12</sup>

Racemic 2-[(*N*-benzyloxycarbonyl)piperidin-2-yl]ethanoic acid (**4**) was reacted with (4*S*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone to give diastereomeric carboxamide (**5**).<sup>9</sup> By column chromatography, the mixture of **5a** and **5b** was separated without any difficulty. When (*S*)-4-benzyl-2-oxazolidinone was used as a chiral auxiliary to prepare a carboxamide, the resulting mixture of two diastereoisomers was difficult to be separated by column chromatography.

The stereochemistry of the piperidine ring was determined by comparison with the known optical rotation. Chiral 2-(piperidin-2-yl)ethanoic acid (**6**) was prepared by the following sequence; hydrolysis of carboxamide (**5**) by LiOH-H<sub>2</sub>O<sub>2</sub> followed by hydrogenolysis of the resulting acid on 10% Pd/C. The [α]<sub>D</sub> values of **6**, derived from one of **5** having lower R<sub>f</sub> value, was +31.2°. Because the [α]<sub>D</sub> values of (*S*)-2-(piperidin-2-yl)ethanoic acid (**6b**) was reported to be +33.5°,<sup>14</sup> the compound having lower R<sub>f</sub> value was assigned to be **5b** and the other one was **5a** in a mixture of **5** (Scheme 1).

Scheme 1. Determination of the Stereochemistry in the Piperidine Ring.



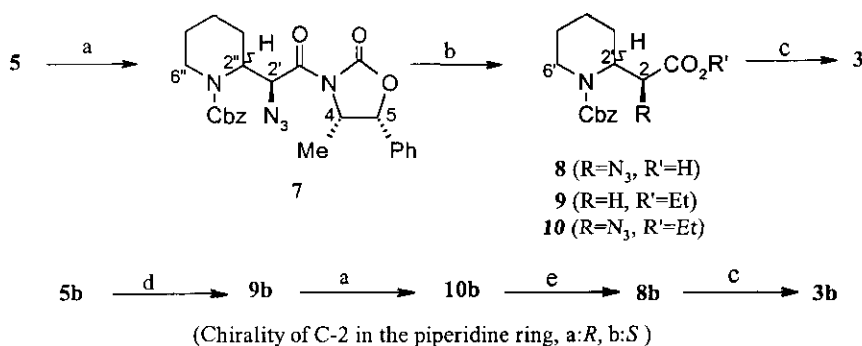
Reaction conditions: (a) i) pivaloyl chloride, Et<sub>3</sub>N, ii) (4*S*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone, *n*-BuLi; (b) i) separation, ii) LiOH, H<sub>2</sub>O<sub>2</sub>; (c) H<sub>2</sub>, 10% Pd/C.

Carboxamide (**5a**) was treated with KHMDS and 2,4,6-triisopropylbenzenesulfonyl azide (trisyl azide) to give azide (**7a**) which has *S* chirality at the C-2', since the stereochemistry was governed by the methyl

and phenyl groups of the oxazolidinone. As a result, the *threo* isomer was in hand. The reaction of azido carboxamide (**7a**) with LiOH-H<sub>2</sub>O<sub>2</sub> gave azido acid (**8a**), which was hydrogenated on 10% Pd/C to afford *threo*-2-piperidylglycine (**3a**). The *threo* stereochemistry was confirmed by comparing its <sup>1</sup>H NMR spectra in 2M DCl/D<sub>2</sub>O with spectra for *threo*- and *erythro*-2,3-diaminobutanoic acids.<sup>7</sup>

Since azide (**7b**), derived from carboxamide (**5b**), was difficult in separation from the unreacted **5b** upon azidation, other methodology was employed to synthesize *erythro*-2-piperidylglycine (**3b**). Carboxamide (**5b**) was converted to ethyl ester (**9b**) which was reacted with trisyl azide to give a mixture of *erythro*-azide (**10b**) and the enantiomer of **10a** in a ratio of 3.5 : 1.<sup>9</sup> Perpendicular model was suggested to explain the *erythro* preference in the azidation.<sup>15</sup> Ester (**10b**) was purified by flash column chromatography and was converted to *erythro*-2-piperidylglycine (**3b**) by the above mentioned method. The *erythro* stereochemistry was also confirmed by its <sup>1</sup>H NMR spectrum (Scheme 2).

Scheme 2. Synthesis of Chiral 2-Piperidylglycine.



Reaction conditions: (a) KHMDS, trisyl azide; (b) LiOH, H<sub>2</sub>O<sub>2</sub>; (c) H<sub>2</sub>, 10% Pd/C; (d) NaOEt, EtOH; (e) LiOH.

## EXPERIMENTAL

All chemicals were reagent grade (Aldrich Chemical Co.) and were used as purchased without further purification. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Varian Gemini 2000 (200MHz) spectrometer in CDCl<sub>3</sub>, unless otherwise stated. Optical rotations were recorded on a Perkin-Elmer polarimeter 241. Melting points were determined on a Yamato MP-21 and were uncorrected. Elemental analysis was performed by a Xytel EA-1110 at Inha University.

**(4S,5S)-3-[2-((N-Benzyloxycarbonyl)piperidin-2-yl)-1-oxoethyl]-4-methyl-5-phenyl-2-oxazolidinone (5).** To a solution of 2-[(N-benzyloxycarbonyl)piperidin-2-yl]ethanoic acid (**4**, 2.73 g, 9.83 mmol) in THF (25 mL) was added triethylamine (1.30 mL, 12.8 mmol) and pivaloyl chloride (1.30 mL, 10.8 mmol) at -

78 °C.<sup>9,13</sup> The slurry was stirred at -78 °C for 10 min and 0 °C for 50 min and then re-cooled to -78 °C. In a separate flask a solution of (4*S*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone (6.80 g, 10.8 mmol) in THF (15 mL) was cooled to -78 °C. To this solution was added 1.6M *n*-BuLi in hexane (13.5 mL, 21.6 mmol). The metalated oxazolidinone was added *via* cannula to the white slurry prepared as described above. The resulting slurry was stirred for 15 min at -78 °C, warmed to rt, and then stirred for 1.5 h. The reaction was quenched by sodium bisulfate (11.9 g, 4.00 mmol). THF was removed *in vacuo*, and the mixture was diluted with water and extracted with CH<sub>2</sub>Cl<sub>2</sub> (40 mL x 3). The extract was dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated *in vacuo*. Purification by flash column chromatography (20% EtOAc/*n*-hexane) gave **5a** (1.50 g, 35%) and **5b** (0.86 g, 20%). **5a**: mp 93 °C - 95 °C (EtOAc/*n*-hexane). <sup>1</sup>H NMR δ 0.79 (d, *J* = 6.5 Hz, 3H, CH<sub>3</sub>), 1.40-1.90 (m, 6H, H-3", H-4", H-5"), 2.93-3.20 (m, 2H, H-2'), 3.32-3.55 (m, 1H, H-6a"), 4.02-4.24 (m, 1H, H-6e"), 4.24-4.48 (m, 1H, H-4), 4.85-5.00 (m, 1H, H-2e"), 5.10-5.17 (m, 2H, benzyl), 5.52-5.68 (m, 1H, H-5), 7.10-7.45 (m, 10H, Ph); <sup>13</sup>C NMR δ 168.7, 153.8, 151.6, 135.5, 131.8, 126.87, 126.83, 126.14, 125.6, 124.0, 77.2, 65.2, 53.1, 46.4, 38.0, 34.8, 27.4, 23.6, 17.3, 12.8; [α]<sub>D</sub><sup>20</sup> +3.3° (c 2.0, CHCl<sub>3</sub>). Anal. Calcd for C<sub>25</sub>H<sub>28</sub>N<sub>2</sub>O<sub>5</sub>: C, 68.79; H, 6.47; N, 6.42. Found: C, 68.74; H, 6.48; N, 6.49.

**5b** (oil): <sup>1</sup>H NMR δ 0.79 (d, *J* = 4.0 Hz, 3H, CH<sub>3</sub>), 1.33-1.85 (m, 6H, H-3", H-4", H-5"), 2.93-3.35 (m, 3H, H-2', H-6a"), 4.00-4.20 (m, 1H, H-6e"), 4.58-4.80 (m, 1H, H-4), 4.85-5.08 (m, 1H, H-2e"), 5.08 (bs, 2H, benzyl), 5.58 (d, *J* = 7.2 Hz, 1H, H-5), 7.15-7.50 (m, 10H, Ph); <sup>13</sup>C NMR δ 168.9, 153.7, 151.4, 135.3, 131.7, 127.1, 126.8, 126.2, 125.9, 124.1, 77.4, 65.2, 53.1, 46.4, 38.0, 34.5, 27.4, 23.5, 17.2, 12.4; [α]<sub>D</sub><sup>20</sup> -24.2° (c 2.0, CHCl<sub>3</sub>). Anal. Calcd for C<sub>25</sub>H<sub>28</sub>N<sub>2</sub>O<sub>5</sub>: C, 68.79; H, 6.47; N, 6.42. Found: C, 68.74; H, 6.48; N, 6.49.

**(3(2*S*),3(2(2*R*)),4*S*,5*S*)-3-[2-Azido-2-((*N*-benzyloxycarbonyl)piperidin-2-yl)-1-oxoethyl]-4-methyl-5-phenyl-2-oxazolidinone (7a)**. To a solution of **5a** (1.00 g, 2.29 mmol) in THF (15 mL) at -78 °C was added dropwise 0.5M KHMDS in toluene (5.5 mL, 2.75 mmol). The solution was stirred for 30 min and treated with a pre-cooled solution of trisyl azide (0.78 g, 2.52 mmol) in THF (5 mL) *via* cannula. The mixture was stirred for 5 min at -78 °C and then quenched by rapid addition of acetic acid (0.66 mL, 4.80 mmol). After the white slurry was stirred for 2 h at rt, it was diluted with CH<sub>2</sub>Cl<sub>2</sub>. The mixture was washed with water, and the water layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (50 mL x 3). Combined organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated *in vacuo*. Purification by flash column chromatography (20% EtOAc/*n*-hexane) gave an oil (0.744 g, 68%). <sup>1</sup>H NMR δ 0.89 (d, *J* = 6.5 Hz, 3H, CH<sub>3</sub>), 1.35-1.95 (m, 6H, H-3", H-4", H-5"), 3.05-3.35 (m, 1H, H-6a"), 4.00-4.20 (m, 1H, H-6e"), 4.35-4.52 (m, 1H, H-4), 4.73-4.90 (m, 1H, H-2e"), 5.00-5.35 (m, 2H, benzyl), 5.35-5.57 (m, 1H, H-2'), 5.69 (d, *J* = 7.2 Hz, H-5), 7.15-7.50 (m, 10H, Ph); <sup>13</sup>C NMR δ 169, 153.7, 151.4, 135.3, 131.7, 127.1, 126.9, 126.2, 125.7, 124.1, 77.6, 65.6, 61.7, 53.8, 46.4, 38.9, 28.0, 24.5, 17.8, 12.9; [α]<sub>D</sub><sup>20</sup> +8.44° (c 2.9, CHCl<sub>3</sub>). HRMS (FAB+)

calcd for  $C_{25}H_{28}N_5O_5(MH^+)$  478.2090, found 478.2089.

**(2S,2(2R))-2-Azido-[(N-benzyloxycarbonyl)piperidin-2-yl]ethanoic acid (8a).** To a solution of **7a** (0.68 g, 1.42 mmol) in a mixture of THF and water (3:1, 15 mL) was added slowly LiOH (0.12 g, 2.88 mmol) and  $H_2O_2$  (35%, 1.38 g, 14.2 mmol). The reaction mixture was stirred for 1 h, and quenched by sodium bisulfate (1.97 g, 14.2 mmol). THF was removed *in vacuo*, and the resulting mixture was diluted by water, acidified to pH 1-2 with 1.0N HCl, and extracted with ethyl acetate (30 mL x 3). The combined organic extracts were dried, and concentrated *in vacuo*. Purification of the residue by column chromatography (10% MeOH/ $CH_2Cl_2$ ) gave a solid (0.41 g, 82%). mp 134 °C-135 °C ( $CH_2Cl_2$ /n-hexane);  $^1H$  NMR  $\delta$  1.40-1.86 (m, 6H, H-3', H-4', H-5'), 2.80-2.89 (m, 1H, H-6a'), 4.07-4.25 (m, 2H, H-6e', H-2), 4.62-4.80 (m, 1H, H-2a'), 5.11-5.28 (m, 2H, benzyl), 7.34 (s, 5H, Ph);  $^{13}C$  NMR  $\delta$  19.0, 20.7, 24.7, 26.2, 39.8, 51.9, 61.0, 67.7, 127.9, 128.0, 136.3, 156.0, 173.5;  $[\alpha]_D^{20} -21.0^\circ$  (c 0.5,  $CHCl_3$ ). Anal. Calcd For  $C_{15}H_{18}N_4O_4$ : C 56.60, H 5.70, N 17.60. Found: C 56.03, H 6.08, N 17.41.

**(2S,2(2R))-2-Amino-2-(piperidin-2-yl)ethanoic acid (3a).** To a solution of **8a** (0.12 g, 0.38 mmol) in MeOH (15mL) was added 10% Pd/C (0.012 g). The black slurry was stirred under hydrogen at atmospheric pressure for 1 h. The slurry was then filtered through celite, and the filtrate was concentrated *in vacuo*. The residue was recrystallized from MeOH/ Et<sub>2</sub>O to give a solid (0.049 g, 81%). mp 204 °C-205 °C;  $^1H$  NMR (2.0M  $DCI/D_2O$ )  $\delta$  1.18-1.82 (m, 6H, H-3', H-4', H-5'), 2.78-2.89 (m, 1H, H-6a'), 3.20-3.27 (m, 1H, H-6e'), 3.44-3.48 (m, 1H, H-2a'), 4.20 (d,  $J = 3.3$  Hz, 1H, H-2);  $^{13}C$  NMR (2.0M  $DCI/D_2O$ )  $\delta$  18.6, 18.8, 20.6, 44.2, 50.6, 52.9, 164.9;  $[\alpha]_D^{20} +22.1^\circ$  (c 0.24, 1.0N HCl). Anal. Calcd For  $C_7H_{14}N_2O$ : C 53.15, H 8.92, N 17.71. Found: C 53.24, H 8.89, N 17.66.

**Ethyl (S)-2-[(N-benzyloxycarbonyl)piperidin-2-yl]ethanoate (9b).**<sup>9</sup> To a precooled solution of **5b** (1.83 g, 4.19 mmol) in EtOH (10 mL) was added slowly NaOEt (0.294 g, 4.19 mmol) in EtOH (5 mL). The reaction mixture was stirred for 2 h and the EtOH was removed *in vacuo*. The residue was added into water and extracted with  $CH_2Cl_2$ . The organic layer was dried, filtered, and concentrated *in vacuo* to give an oil. Purification of the crude product by column chromatography (20% EtOAc/n-hexane) gave an oil (1.04 g, 81%).  $^1H$  NMR  $\delta$  1.21 (t,  $J=7.2$  Hz, 3H,  $CH_3$ ), 1.35-1.95 (m, 6H, H-3', H-4', H-5'), 2.52-2.65 (m, 2H, H-2), 2.78-2.96 (m, 1H, H-6a'), 3.95-4.15 (m, 1H, H-6e'), 4.07 (q,  $J = 7.4$  Hz, 2H,  $-OCH_2$ ), 4.72-4.86 (m, 1H, H-2e'), 5.13 (br s, 2H, benzyl);  $^{13}C$  NMR  $\delta$  12.4, 17.0, 23.5, 26.5, 33.6, 37.8, 46.5, 58.2, 65.3, 126.1, 126.2, 126.8, 135.3, 153.7, 169.6;  $[\alpha]_D^{20} -7.2^\circ$  (c 2.1,  $CHCl_3$ ); MS (EI) m/z 305 ( $M^+$ ).

**Ethyl (2S,2(2S))-2-azido-[(N-benzyloxycarbonyl)piperidin-2-yl]ethanoate (10b).**<sup>9</sup> The procedure outlined above for the azidation of **5a** was followed employing **9b**. The crude product (**10b**) was purified by column chromatography (20% EtOAc/n-hexane) to give an oil (45%).  $^1H$  NMR  $\delta$  1.20 (t,  $J = 7.0$  Hz, 3H,  $CH_3$ ), 1.40-2.02 (m, 6H, H-3', H-4', H-5'), 2.85-3.05 (m, 1H, H-6a'), 4.09 (q,  $J = 7.0$  Hz, 2H,  $OCH_2$ ),

3.98-4.10 (m, 1H, H-6e'), 4.53-4.68 (m, 1H, H-2e'), 5.10 (m, 2H, benzyl), 7.25-7.40 (m, 5H, Ph);  $^{13}\text{C}$  NMR 12.3, 17.3, 23.1, 24.4, 37.9, 50.2, 59.0, 60.3, 65.7, 126.3, 126.8, 134.9, 153.9, 167.6; [ $\alpha$ ] $^{20}_{\text{D}}$ +19.8° (c 2.3,  $\text{CHCl}_3$ ); MS (CI)  $m/z$  347 ( $\text{MH}^+$ ).

**(2S,2(2S))-2-Azido-[(N-benzyloxycarbonyl)piperidin-2-yl]ethanoic acid (8b).** To a solution of **10b** (0.469 g, 1.42 mmol) in a mixture of THF and water (3:1, 15 mL) was added slowly LiOH (0.12 g, 2.83 mmol) at 0°C. the reaction mixture was stirred for 1 h, and THF was removed *in vacuo*. The resultant was diluted by water, acidified to pH 1-2 with 1.0N HCl, and extracted with ethyl acetate. The combined organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated *in vacuo*. Purification of the residue by column chromatography (10% MeOH/ $\text{CH}_2\text{Cl}_2$ ) gave an oil (0.42 g, 84%).  $^1\text{H}$  NMR  $\delta$  1.40-1.95 (m, 6H, H-3', H-4', H-5'), 2.96-3.08 (m, 1H, H-6a'), 4.06-4.20 (m, 2H, H-6e', H-2), 4.52-4.64 (m, 1H, H-2a'), 5.03-5.17 (m, 2H, benzyl), 7.34 (br s, 5H, Ph);  $^{13}\text{C}$  NMR  $\delta$  17.0, 22.8, 23.3, 50.4, 58.7, 66.3, 126.2, 126.9, 134.5, 154.5, 170.8; [ $\alpha$ ] $^{20}_{\text{D}}$ -6.9° (c 1.8,  $\text{CHCl}_3$ ). Anal. Calcd For  $\text{C}_{15}\text{H}_{18}\text{N}_4\text{O}_4$ : C 56.60, H 5.70, N 17.60. Found: C 56.03, H 6.08, N 17.41.

**(2S,2(2S))-2-Amino-2-(piperidin-2-yl)ethanoic acid (3b).** The procedure outlined above for **3a** was followed employing **8b**. mp 234°C-235°C (MeOH/Et $_2$ O);  $^1\text{H}$  NMR (2.0 M DCl/D $_2$ O)  $\delta$  1.12-1.63 (m, 6H, H-3', H-4', H-5'), 2.64-2.75 (m, 1H, H-6a'), 3.10-3.20 (m, 1H, H-6e'), 3.30-3.47 (m, 1H, H-2a'), 4.00 (d,  $J$ = 6.4 Hz, 1H, H-2);  $^{13}\text{C}$  NMR (2.0 M DCl/D $_2$ O)  $\delta$  18.5, 22.2, 43.6, 51.2, 52.7, 164.9; [ $\alpha$ ] $^{20}_{\text{D}}$ -10.0° (c 0.05, 1.0 N HCl). Anal. Calcd For  $\text{C}_7\text{H}_{14}\text{N}_2\text{O}$ : C 53.15, H 8.92, N 17.71. Found: C 53.24, H 8.89, N 17.66.

## ACKNOWLEDGMENT

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