

(100 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 (s, 5 H, ArH), 5.17 (s, 2 H, ArCH<sub>2</sub>), 5.00–4.67 (m, 1 H), 4.23–3.80 (m, 2 H), 3.73 (s, 3 H, OCH<sub>3</sub>), 3.60–3.23 (m, 1 H), 2.46 (d, 1 H,  $J = 16.6$  Hz), 2.02–1.60 (m, 4 H); IR (KBr) 1749, 1666 cm<sup>-1</sup>; MS (CI) 294 (MH<sup>+</sup>, 100). Anal. Calcd for C<sub>15</sub>H<sub>19</sub>NO<sub>5</sub>: C, 61.42; H, 6.53; N, 4.78. Found: C, 61.35, 6.53; N, 4.61.

**Bis(phenylmethyl) *cis*-4-Hydroxy-1,2-piperidinedicarboxylate (8).** A solution of the methyl ester 7 (2.6 g, 9.0 mmol) in 25 mL of dioxane and 2 mL of aqueous 1 N NaOH was stirred at room temperature for 18 h. The reaction mixture was treated with a saturated sodium bicarbonate solution until the pH = 8. The solvent was removed under reduced pressure, and the residue was dissolved in 2 mL of saturated aqueous sodium bicarbonate solution. The resulting solution was treated sequentially with Adogen-464 (3.64 g) in 20 mL of CH<sub>2</sub>Cl<sub>2</sub> and benzyl bromide (1.85 g, 10.8 mmol). The resulting solution was stirred at room temperature for 4 days. The reaction mixture was partitioned between water and CH<sub>2</sub>Cl<sub>2</sub>, and the organic phase was collected. The aqueous phase was extracted with additional CH<sub>2</sub>Cl<sub>2</sub>, and the combined organic extracts were dried (Na<sub>2</sub>SO<sub>4</sub>), filtered, and concentrated. The residue was purified by chromatography (silica gel, 9:1 CH<sub>2</sub>Cl<sub>2</sub>/MeOH) to give the product as an oil that was crystallized from ethyl acetate (3.32 g, 86%), mp 123–125 °C: <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>)  $\delta$  7.34 (s, 10 H, ArH), 5.16–5.10 (m, 4 H), 4.90 (dd, 1 H,  $J = 27.6, 6.6$  Hz), 4.16 (s, 1 H), 4.07–3.86 (m, 1 H), 3.66–3.36 (m, 1 H), 2.61–2.40 (d, 1 H,  $J = 16.7$  Hz), 2.00–1.85 (m, 1 H), 1.84–1.50 (m, 3 H); IR (neat) 1774, 1698 cm<sup>-1</sup>; MS (CI) 370 (MH<sup>+</sup>). Anal. Calcd for C<sub>21</sub>H<sub>23</sub>NO<sub>5</sub>: C, 68.28; H, 6.28; N, 3.79. Found: C, 68.31; H, 6.32; N, 3.63.

**Bis(phenylmethyl) *cis*-4-(Diphenylphosphonoxy)-1,2-piperidinedicarboxylate (9).** A solution of the alcohol 8 (0.92 g, 2.7 mmol), triethylamine (0.41 g, 4.1 mmol), and (dimethylamino)pyridine (0.49 g, 4.0 mmol) in 30 mL of CH<sub>2</sub>Cl<sub>2</sub> was treated with diphenylphosphoryl chloride (1.07 g, 4.0 mmol) over a 1–2-min period. The resulting solution was allowed to stir at room temperature for 24 h. The reaction mixture was transferred to

a separatory funnel and washed sequentially with water (20 mL), 0.5 N HCl (20 mL), and water (20 mL). The organic layer was dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated. The residue (2.08 g) was purified by chromatography (silica gel, 2% MeOH in CH<sub>2</sub>Cl<sub>2</sub>) to give the product as a colorless oil (1.62 g, 73%): <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>)  $\delta$  7.53–7.00 (m, 20 H, ArH), 5.32–4.77 (m, 6 H), 4.15–3.90 (m, 1 H), 3.61–3.27 (m, 1 H), 2.85–2.65 (m, 2 H), 2.12–1.55 (m, 3 H). Anal. Calcd for C<sub>33</sub>H<sub>32</sub>NO<sub>5</sub>P: C, 65.88; H, 5.36; N, 2.33. Found: C, 66.12; H, 5.38; N, 2.38.

***cis*-4-(Phosphonoxy)-2-piperidinecarboxylic Acid (1).** A solution of ester 9 (1.10 g, 1.8 mmol) was dissolved in 35 mL of trifluoroacetic acid and 40 mL of acetic acid. The resulting solution was hydrogenated over PtO<sub>2</sub> for 2.5 h. The catalyst was removed by filtration, and the filtrate was concentrated. The residue was partitioned between water and CH<sub>2</sub>Cl<sub>2</sub>. The aqueous layer was collected and concentrated to give a white solid. The solid material was treated with a 1:1 EtOH/H<sub>2</sub>O solution and heated to 50 °C. The resulting suspension was cooled to room temperature and the solid collected by filtration. The collected material was dried under vacuum (100 °C) to give the product as a white solid (0.28 g, 69%), mp 282–284 °C dec: <sup>1</sup>H NMR (250 MHz, D<sub>2</sub>O)  $\delta$  4.40–4.27 (m, 1 H, CHOP), 3.75 (dd, 1 H,  $J = 12.5, 3.0$  Hz NCHCOOH), 3.54–3.49 (m, 1 H), 3.07 (dt, 1 H,  $J = 13.0, 3.0$  Hz), 2.64–2.53 (m, 1 H), 2.31–2.22 (m, 1 H), 1.82–1.68 (m, 2 H); IR (KBr) 1718 cm<sup>-1</sup>; MS (FAB) 209 (M - 16). Anal. Calcd for C<sub>6</sub>H<sub>12</sub>NO<sub>5</sub>P: C, 32.01; H, 5.37; N, 6.22. Found: C, 32.10; H, 5.47; N, 5.83.

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**Registry No.** 1, 133192-42-4; 3, 17150-62-8; 5, 133192-43-5; 6, 133192-44-6; 7, 133192-45-7; 8, 133192-46-8; 9, 133192-47-9; NMDA, 6384-92-5; HCOCOOH, 298-12-4.

## Additions and Corrections

Vol. 55, 1990

Edward E. Schweizer,\* Cao Zhisong, Arnold Rheingold, and Martha Bruch. Reactions of Azines. 15. Preparation of Pyrazolo[1,5-c][1,3]oxazepin-6-ones.

Page 6363, column 2, lines 9–13, should read as follows: Therefore, the major isomer has a *Z* configuration, that is, the hydrogen atom on C6 is on the same side as the C4-methyl group. The ylidenes 6 have a characteristic absorption around 5.86 ± 0.17 ppm for C6-H (see Table III), thus suggesting that they should all be in the *Z* form.

Page 6364, column 1, lines 7–10, should read as follows: However, when R was phenyl or methyl and R<sup>1</sup> = R<sup>2</sup> = Ph (6g and 6f) or R = Ph and R<sup>1</sup> = R<sup>2</sup> = Me (6e), the rearrangement reaction did not take place under refluxing xylene.

Vol. 56, 1991

Jeffrey Aubé,\* Marlys Hammond, Elyse Gherardini, and Fusao Takusagawa. Syntheses and Rearrangements of Spirocyclic Oxaziridines Derived from Unsymmetrical Ketones.

Page 502, footnote *a* to Table I should read "Ketones were racemic and reacted with (*R*)- $\alpha$ -MBA unless otherwise noted."

In addition, some errors in absolute configuration and locant descriptors occur in the compound names given in the Experimental Section. The structures drawn in the text are correct and do correspond to those identified by numbers in the Experimental Section. None of the conclusions of the paper are altered. The correct prefixes follow: 2, [2*S*-[2*R*\*(*S*\*),3*R*\*5*S*\*]]; 3, [2*R*-[2*R*\*(*S*\*),3*S*\*5*R*\*]]; 11, [2*S*-[2*R*\*(*S*\*),3*R*\*4*R*\*]]; 12, [2*S*-[2*R*\*(*S*\*),3*R*\*4*S*\*]]; 15, [2*S*-(2*R*\*(*S*\*),3*S*\*4*S*\*)]; 14, [2*S*-(2*R*\*(*S*\*),3*R*\*4*R*\*)]; 17, [2*S*-(2*R*\*(*S*\*),3*R*\*4*S*\*)]; 18, [2*S*-(2*R*\*(*S*\*),3*R*\*4*R*\*)]; 5a, [*R*-(*R*\**S*\*)]; 26, [*S*-(*R*\**S*\*)]; 28, [*S*-(*R*\**S*\*)]; 29, [*S*-(*R*\**R*\*)].