

# Asymmetric synthesis of 8-O-4'-neolignan perseal B

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Full details of an enantioselective total synthesis of 8-O-4'-neolignan perseal B are presented for the first time. The synthesis was achieved in 8 steps from vanillin and involved the asymmetric dihydroxylation reaction using AD-mix- $\alpha$  to give the key intermediate (1*S*,2*S*)-1-(4-(benzyloxy)-3-methoxyphenyl)propane-1,2,3-triol, and the Mitsunobu reaction between phenylpropanoid and vanillin formed perseal B. The synthetic method of perseal B exhibits a new route for 8-O-4'-neolignan.

**Key Words:** Neolignans; perseal B; enantioselective synthesis.

## Introduction

Neolignans are a class of secondary plant metabolites produced by oxidative dimerization of 2 phenylpropane (C6-C3) units, which are formed biogenetically through the shikimate pathway.<sup>1</sup> They are widespread in nature and are found in many kinds of plants.<sup>2-4</sup> Neolignans, especially 8-O-4' neolignans, exhibit a wide range of biological activities, including against *Trypanosoma cruzi*, and antileukemic, antitumor, antifungal, antimicrobial, insect antifeedant, and other diverse types of activity.<sup>5,6</sup>

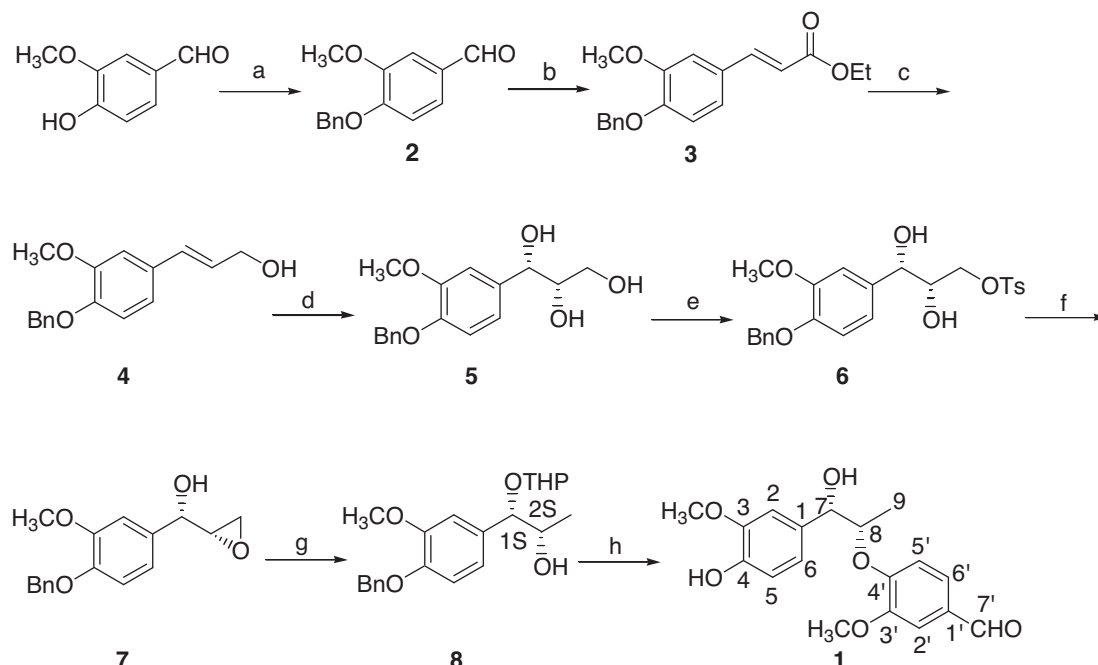
8-O-4' neolignans have stimulated substantial synthetic efforts due to their biological activity.<sup>2,7</sup> As reported previously, the synthesis of 8-O-4' neolignans was mainly achieved using oxidative coupling of monolignols and reduction of ketones, which were prepared from bromoketone and phenols in basic conditions.<sup>8-10</sup> However, these methods led to the mixture of *erythro*- and *threo*-isomers in all cases tested. Recently, Lourith developed a biosynthetic pathway for the synthesis of 8-O-4' neolignans in *Eucommia ulmoides*.<sup>7</sup> Curti reported the synthesis of 8-O-4' neolignans by modular synthesis.<sup>11</sup>

In this paper, the main objective of our study was to develop a new stereoselective synthesis approach to 8-O-4' neolignans perseal B, which was isolated from the leaves of *Persea obovatifolia*, and exhibited biological

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activities of against leishmaniasis and a tropical disease transmitted by mosquitoes.<sup>3,4</sup> As shown in the Scheme, we obtained 2 chiral centers by dihydroxylation of AD-mix- $\alpha$ , and condensated phenylpropanoid with vanillin using the Mitsunobu reaction. By this method, perseal B was synthesized stereoselectively for the first time.



**Scheme.** Reagents and conditions: (a) BnCl,  $K_2CO_3$ , EtOH, reflux, 4 h; (b)  $C_6H_6$ ,  $(Ph)_3PCHCOOEt$ , reflux, 10 h; (c) LAH,  $AlCl_3$ , THF, 10 h; (d) AD-mix- $\alpha$ ,  $MeSO_2NH_2$ ,  $tBuOH$ ,  $H_2O$ ,  $0^\circ C$ , 30 h; (e) TsCl, pyridine,  $CH_2Cl_2$ , rt; (f)  $K_2CO_3$ , MeOH, rt, 5 h (g) DHP, PPTS,  $CH_2Cl_2$ , LAH, THF, rt, 24 h; (h) vanillin,  $Ph_3P$ , DIAD, THF, reflux, 24 h; HCl, MeOH, rt, 8 h; Pd/C (10%),  $H_2$ , AcOEt, rt, 6 h.

## Experimental

### General

Melting points were taken on a Gallenkamp melting point apparatus and are uncorrected. Optical rotations were determined on a Perkin-Elmer 341 polarimeter. Chiral analysis was performed on a Varian Dynamax SD-300 using a chiralcel column CDMPC ( $150 \times 4.6$  mm D) with hexane/isopropyl alcohol as eluant. Infrared spectra were recorded on a Nicolet NEXUS 670 FT-IR. The  $^1H$ -NMR and  $^{13}C$ -NMR spectra were recorded on a Mercury Plus-300 MHz and Avance-200 MHz spectrometers. Mass spectra were recorded on a ZAB-HS spectrometer. HRMS were obtained on a Bruker Daltonics APEXII47e spectrometer. Flash column chromatography was performed on silica gel (200-300 mesh) and TLC inspections on silica gel GF254 plates.

**4-(benzyloxy)-3-methoxybenzaldehyde (2)** To a well-stirred solution of  $K_2CO_3$  in ethanol was added dropwise a mixture of vanillin (5.2 g, 0.035 mol) and benzyl chloride (3.64 g, 0.035 mol). The mixture was stirred and warmed to reflux for 4 h, and then concentrated in vacuo. The residue was dissolved in AcOEt

(300 mL), filtered, and washed with 10% aqueous NaOH and NaCl saturated solution 3 times consecutively. The extract was dried over MgSO<sub>4</sub>, filtered, and then concentrated in vacuo to remove AcOEt. The solids were recrystallized in C<sub>2</sub>H<sub>5</sub>OH to yield compound **2** as a colorless crystal (7.64 g, 92%). Mp 62-63 °C. <sup>1</sup>H-NMR (200 MHz, DMSO-d<sub>6</sub>), δ: 3.83 (s, 3H, OCH<sub>3</sub>), 5.17 (s, 2H, ArCH<sub>2</sub>O), 7.01-7.67 (m, 8H, Ar-H), 9.87 (s, 1H, CHO). EI-MS, *m/z*: 242 (M<sup>+</sup>, 14.3), 214 (3.2), 91 (100), 65 (13.5), 51 (5.2).

**(E)-ethyl 3-(4-(benzyloxy)-3-methoxyphenyl)acrylate (3)** Compound **2** (7.28 g, 0.30 mol) and (Ph)<sub>3</sub>PCHCOOEt (10.44 g, 0.030 mol) in dry benzene were refluxed for 10 h and concentrated in vacuo. Flash column chromatography of the residue gave compound **3** as a white crystal (6.78 g, 72%). Mp 68-72 °C. <sup>1</sup>H-NMR (200 MHz, CDCl<sub>3</sub>), δ: 1.44 (t, 3H, *J* = 7.2 Hz, COCH<sub>2</sub>CH<sub>3</sub>), 3.98 (s, 3H, OCH<sub>3</sub>), 4.26 (q, 2H, *J* = 7.2 Hz, COCH<sub>2</sub>CH<sub>3</sub>), 5.10 (s, 2H, ArCH<sub>2</sub>O), 6.25 (d, 1H, *J* = 14.2 Hz, ArCH=CH), 6.70-7.52 (m, 8H, ArH), 7.63 (d, 1H, *J* = 14.2 Hz, ArCH=CH). EI-MS, *m/z*: 312 (M<sup>+</sup>, 15), 267 (12), 221 (1.2), 153 (8), 91 (100).

**(E)-3-(4-(benzyloxy)-3-methoxyphenyl)prop-2-en-1-ol (4)**. At 0 °C, to a suspension of LiAlH<sub>4</sub> (2.40 g, 63.4 mmol) in dry THF (100 mL), dry AlCl<sub>3</sub> (2.82 g, 21.2 mmol) was added and stirred for 20 min. Then compound **3** (6.6 g, 21.2 mmol) was added. The mixture was stirred for 10 h at room temperature, and then quenched with H<sub>2</sub>O, filtered, and concentrated in vacuo. Flash column chromatography of the residue gave **4** as a white crystal (5.0 g, 88%). Mp 79-80 °C. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): 3.88 (s, 3H, OCH<sub>3</sub>), 4.28 (d, 2H, *J* = 6.0 Hz, CH<sub>2</sub>OH), 4.61 (s, 1H, OH), 5.13 (s, 2H, ArCH<sub>2</sub>O), 6.24 (dt, 1H, *J* = 6.0, 15.9 Hz, ArCH=CH), 6.53 (d, 1H, *J* = 15.9 Hz, ArCH=CH), 6.68-7.44 (m, 8H, ArH). EI-MS, *m/z*: 270 (M<sup>+</sup>, 25), 179 (12), 151 (8), 119 (18), 91 (100).

**(1S,2S)-1-(4-(benzyloxy)-3-methoxyphenyl)propane-1,2,3-triol (5)** To a stirred solution of *t*-BuOH (50 mL) and H<sub>2</sub>O (50 mL) was added AD-mix-a (14 g) and MeSO<sub>2</sub>NH<sub>2</sub> (960 mg), and the mixture was stirred at room temperature until both phases were clear, and then cooled to 0 °C. Compound **4** (2.7 g, 10 mmol) was added at once, and the mixture was stirred vigorously at 0 °C until TLC revealed the absence of **4**. The reaction was quenched at 0 °C by addition of Na<sub>2</sub>SO<sub>3</sub> (15 g), then warmed to room temperature, and stirred for 0.5 h. The reaction mixture was extracted with AcOEt (3 × 100 mL) and dried over MgSO<sub>4</sub>; then the AcOEt was distilled off. Flash chromatography of the residue over silica gel gave compound **5** (2.9 g, 95%) as a white crystal in 93% e.e. Mp 153-155 °C. [α]<sub>D</sub><sup>20</sup> = -71.0 (c 1.0, acetone). IR (KBr/cm<sup>-1</sup>): 3410, 2935, 2840, 1655, 1516, 1460, 1155, 990. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): 3.40 (dd, 1H, *J* = 3.0, 9.4 Hz, CH<sub>2</sub>OH), 3.47 (dd, 1H, *J* = 3.0, 9.4 Hz, CH<sub>2</sub>OH), 3.60-3.73 (m, 1H, CHOHCH<sub>2</sub>OH), 3.82 (s, 3H, OCH<sub>3</sub>), 4.61 (d, *J* = 6.5 Hz, 1H, ArCHOH), 5.10 (s, 2H, ArCH<sub>2</sub>OAr), 6.86-7.04 (m, 3H, ArH), 7.31-7.51 (m, 5H, ArH). EI-MS, *m/z*: 304 (M<sup>+</sup>, 0.9), 286 (5), 243 (17), 153 (24), 91 (100). HRMS calcd for C<sub>17</sub>H<sub>24</sub>NO<sub>5</sub> (M+NH<sub>4</sub><sup>+</sup>): 322.1649. Found: 322.1652. The data are consistent with the literature.<sup>12</sup>

**(2S,3S)-3-(4-(benzyloxy)-3-methoxyphenyl)-2,3-dihydroxypropyl 4-methylbenzenesulfonate (6)** At room temperature to a solution of TsCl (1.52 g, 8 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (60 mL) was added compound **5** (2.4 g, 8 mmol) and pyridine (30 mL). The mixture was stirred vigorously until TLC revealed the absence of **5**. The mixture was quenched with HCl solution and then extracted with AcOEt. The organic layer was washed with NaHCO<sub>3</sub> and NaCl saturated solution consecutively, then dried over MgSO<sub>4</sub>, and concentrated in vacuo. Flash chromatography of the residue over silica gel gave compound **6** (3.0 g, 83%) as amorphous powder in 95% e.e. [α]<sub>D</sub><sup>20</sup> = -42.3 (c 0.5, CHCl<sub>3</sub>). IR (KBr/cm<sup>-1</sup>): 3420, 2941, 2845, 1641, 1520, 1422, 1318, 1235, 1102, 1023. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): 2.44 (s, 3H, ArCH<sub>3</sub>), 3.90 (s, 3H, ArOCH<sub>3</sub>), 3.84-3.96

(m, 1H, *CHOHCH*<sub>2</sub>OTs), 3.98-4.11 (m, 2H, *CH*<sub>2</sub>OTs), 4.60 (d, 1H, *J* = 6.0 Hz, *ArCHOH*), 5.14 (s, 2H, *ArCH*<sub>2</sub>OAr), 6.72-7.76 (m, 12H, ArH). <sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>),  $\delta$ : 21.6 (*ArCH*<sub>3</sub>), 56.0 (*OCH*<sub>3</sub>), 70.3 (*CH*<sub>2</sub>OTs), 70.9 (*CHOHCH*<sub>2</sub>OTs), 73.4 (*ArCH*<sub>2</sub>OAr), 73.7 (*ArCHOHCH*), 109.9, 113.7, 118.8, 127.2, 127.2, 127.9, 127.9, 128.6, 128.6, 129.9, 129.9, 132.5, 132.5, 136.9, 136.9, 146.1, 148.1, 149.8. EI-MS, *m/z*: 458 (*M*<sup>+</sup>, 2.4), 286 (1.2), 268 (0.7), 243 (4.8), 123 (7.8), 91 (100). HRMS calcd for C<sub>24</sub>H<sub>30</sub>NO<sub>7</sub>S (*M*+NH<sub>4</sub><sup>+</sup>): 476.1738. Found: 476.1732.

**(*S*)-(4-(benzyloxy)-3-methoxyphenyl)((*S*)-oxiran-2-yl)methanol (7)** To a solution of K<sub>2</sub>CO<sub>3</sub> (0.32 g, 2.2 mmol) in methanol was added compound **6** (2.0 g, 4.4 mmol). The mixture was stirred at room temperature for 5 h; then the reaction mixture was concentrated in vacuo. The residue was dissolved in AcOEt and washed with water and NaCl saturated solution 3 times consecutively. The extract was dried over MgSO<sub>4</sub> and concentrated in vacuo. Flash column chromatography of the residue over silica gel gave compound **7** (1.2 g, 96%) as colorless liquid in 92% e.e.  $[\alpha]_D^{20} = -36.2$  (c 0.6, CHCl<sub>3</sub>). IR (KBr/cm<sup>-1</sup>): 3450, 2983, 2936, 2840, 1732, 1604, 1592, 1512, 1465, 1262, 1140, 1027, 916. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>),  $\delta$ : 2.80-2.89 (m, 2H, *CHOCH*<sub>2</sub>), 3.16-3.25 (m, 1H, *CHCH*<sub>2</sub>O), 3.97 (s, 3H, *OCH*<sub>3</sub>), 4.43 (d, 1H, *J* = 5.8 Hz, *ArCHOHCH*), 5.17 (s, 2H, *ArCH*<sub>2</sub>OAr), 6.87-7.45 (m, 8H, ArH). <sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>),  $\delta$ : 45.6 (*CHCH*<sub>2</sub>O), 56.2 (*OMe*-3), 56.7 (*CHCH*<sub>2</sub>O), 71.3 (*ArCH*<sub>2</sub>OAr), 74.8 (*ArCHOHCH*), 109.3, 113.9, 121.5, 126.7, 127.2, 127.2, 127.9, 128.5, 128.5, 137.1, 147.3, 149.6. EI-MS, *m/z*: 286 (*M*<sup>+</sup>, 5), 243 (0.7), 165 (0.4), 123 (0.2), 91 (100). HRMS calcd for C<sub>17</sub>H<sub>22</sub>NO<sub>4</sub> (*M*+NH<sub>4</sub><sup>+</sup>): 304.1544. Found: 304.1547. The data are consistent with the literature.<sup>12</sup>

**(1*S*,2*S*)-1-(4-(benzyloxy)-3-methoxyphenyl)-1-(tetrahydro-2*H*-pyran-2-yloxy)propan-2-ol (8)** To a solution of compound **7** (0.86 g, 3.0 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> were added DHP (0.26 g, 3.0 mmol) and a catalytic amount of PPTS. The mixture was stirred at room temperature until TLC revealed the absence of **7**. Then the mixture was concentrated in vacuo. The residue was added to a solution of LiAlH<sub>4</sub> (120 mg, 3.0 mmol) in dry THF. The mixture was stirred at room temperature for 24 h. The mixture was quenched with H<sub>2</sub>O, filtered, and concentrated in vacuo. Flash column chromatography of the residue over silica gel gave compound **8** (0.92 g, 82%) as a colorless liquid in 93% e.e.  $[\alpha]_D^{20} = -31.8$  (c 0.3, CHCl<sub>3</sub>). IR (KBr/cm<sup>-1</sup>): 3418, 2936, 2869, 1593, 1513, 1457, 1418, 1380, 1263, 1226, 1137, 1075, 1028, 809. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>),  $\delta$ : 1.00 (d, 3H, *CHOHCH*<sub>3</sub>), 1.44-1.72 (m, 6H, *CH*<sub>2</sub>), 3.20 (m, 1H, *CH*<sub>2</sub>*CH*<sub>2</sub>O), 3.45 (m, 1H, *CH*<sub>2</sub>*CH*<sub>2</sub>O), 3.89 (s, 3H, *OCH*<sub>3</sub>), 3.84-4.05 (m, 1H, *CHOHCH*<sub>3</sub>), 4.16 (d, 1H, *J* = 7.5 Hz, *ArCHOTHP*), 4.84 (s, 1H, *OCHO*), 5.14 (s, 2H, *ArCH*<sub>2</sub>OAr), 6.82-6.90 (m, 3H, ArH), 7.30-7.44 (m, 5H, ArH). <sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>),  $\delta$ : 18.2 (*HOHCH*<sub>3</sub>), 19.3 (*CH*<sub>2</sub>*CH*<sub>2</sub>), 25.2 (*CH*<sub>2</sub>*CH*<sub>2</sub>), 30.5 (*CH*<sub>2</sub>*CH*<sub>2</sub>), 55.9 (*CH*<sub>3</sub>O), 62.4 (*CH*<sub>2</sub>*CHOHCH*), 71.0 (*CHOH*), 71.1 (*ArCH*<sub>2</sub>O), 85.7 (*ArCHOTHP*), 100.0 (*OCHO*), 110.8, 113.4, 119.7, 127.3, 127.3, 127.8, 128.5, 128.5, 133.3, 137.1, 147.6, 149.3. EI-MS, *m/z*: 372 (*M*<sup>+</sup>, 0.4), 328 (1.6), 288 (0.3), 271 (0.6), 243 (37), 91 (98), 85 (100). HRMS calcd for C<sub>22</sub>H<sub>32</sub>NO<sub>5</sub> (*M*+NH<sub>4</sub><sup>+</sup>): 390.2273. Found: 390.2275.

**4-((1*S*,2*S*)-1-hydroxy-1-(4-hydroxy-3-methoxyphenyl)propan-2-yloxy)-3-methoxybenzaldehyde (1)** A mixture of (1*S*,2*S*)-**8** (0.74 g, 2.0 mmol), vanillin (0.46 g, 3.0 mmol), triphenylphosphine (0.80 g, 3.0 mmol), and diethylazodicarboxylate (0.48 mL, 3.0 mmol) was heated to reflux in anhydrous THF (30 mL) for 24 h under nitrogen. The mixture was concentrated under reduced pressure. The residue was added to a solution of HCl in MeOH (0.3 N, 50 mL), and the mixture was stirred at room temperature for 8 h. The solution was neutralized with NaHCO<sub>3</sub> saturated solution. Subsequently the solvent was concentrated in vacuo and the residue was taken up in AcOEt. The organic phase was separated, washed with H<sub>2</sub>O, dried with Na<sub>2</sub>SO<sub>4</sub>,

and concentrated in vacuo. To a stirred solution of residue (0.43 g, 1.1 mmol) in methanol (70 mL) was added 10% palladized charcoal (50 mg). After stirring for 8 h at room temperature under atmospheric pressure of hydrogen, the solvent was concentrated under reduced pressure. Flash column chromatography of the residue over silica gel gave compound (1*S*,2*S*)-**1** (0.29 g, 44%) as amorphous powder in 91% e.e.  $[\alpha]_D^{20} = +58.3$  (c 0.6, CHCl<sub>3</sub>). IR (KBr/cm<sup>-1</sup>): 3420, 2930, 2850, 1675, 1590, 1510. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$ : 1.22 (d, 3H,  $J = 6.4$  Hz, Me-8), 3.90 (s, 3H, OMe-3), 3.96 (s, 3H, OMe-3'), 4.40 (m, 1H, H-8), 4.71 (d, 1H,  $J = 8.0$  Hz, H-7), 6.88-6.94 (m, 3H, Ar-H), 7.05-7.46 (m, 3H, Ar-H'), 9.86 (s, 1H, CHO). <sup>13</sup>C-NMR (75 MHz, CDCl<sub>3</sub>),  $\delta$ : 16.4 (Me-9), 56.0 (OMe-3), 56.0 (OMe-3'), 78.0 (C-7), 82.1 (C-8), 109.3 (C-2), 109.9 (C-6'), 114.2 (C-5), 115.2 (C-5'), 120.6 (C-6), 126.3 (C-2'), 131.0 (C-1), 131.4 (C-1'), 145.8 (C-4), 146.7 (C-3), 150.8 (C-3), 153.2 (C-4'), 190.8 (CHO). EI-MS,  $m/z$ : 332 (M<sup>+</sup>, 3.46), 180 (84.7), 153 (83.8), 151 (100), 135 (46). HRMS calcd for C<sub>18</sub>H<sub>24</sub>NO<sub>6</sub> (M+NH<sub>4</sub><sup>+</sup>): 350.1605. Found: 350.1599. The data are consistent with the literature.<sup>3</sup>

## Results and discussion

As shown in the Scheme, synthesis of the target compound **1** began from vanillin. Protection of the hydroxyl group with benzyl bromide afforded compound **2**. The Wittig reaction was carried out between compound **2** and (Ph)<sub>3</sub>PCHCOOEt, and unsaturated ester (*E*)-**3** was synthesized. Reduction of (*E*)-**3** with LAH gave the corresponding unsaturated alcohol (*E*)-**4** in high yields. It is interesting to note that compound **4** was treated with AD-mix- $\alpha$  to afford directly compound (1*S*,2*S*)-**5** in 93% e.e.<sup>13</sup> In fact, this reaction was a dihydroxylation of AD-mix- $\alpha$  to the double bond of **4** to obtain 2 chiral centers. Treatment of (1*S*,2*S*)-**5** with sterically hindered acid chlorides TsCl in pyridine provided the primary tosylate (1*S*,2*S*)-**6**. Because of the steric hindrance, this reaction only occurred in primary hydroxyl. This was followed by ring closure using K<sub>2</sub>CO<sub>3</sub> in methanol to afford (1*S*,2*S*)-**7**. The hydroxy group of **7** was protected by DHP, and then ring opening using LiAlH<sub>4</sub> in THF gave (1*S*,2*S*)-**8**. Mitsunobu reaction between compound **8** and vanillin could give (1*S*,2*S*)-**1**. The Mitsunobu reaction provided an uncharacterized mixture and the absolute configuration at the C-8 stereogenic center was not inverted completely.<sup>14</sup> Then the THP group was removed with 0.3N HCl in MeOH and the benzyl group was cleaved by hydrogenation with a 10% palladium on charcoal catalyst, and (1*S*,2*S*)-perseal B **1** was obtained. The data of (1*S*,2*S*)-perseal B **1** were agreement with those reported in the literature.<sup>3</sup>

In summary, we have developed an efficient chiral synthetic method to give perseal B. With cheap materials, short experimental procedures, mild conditions, and simple operations, the route will exhibit more potential value in the future.

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