with us his results for a similar approach to **2** and **3** prior to publication.

Tetrahydropyran as an Efficient Alcohol Protecting Group for the Synthesis of Penems: Synthesis of Sch 34343

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In the past few years, syntheses leading to (3*S*,4*R*)-3-[(R)-hydroxyethyl]-4-[(triphenylmethyl)thio]-2-azetidinone (**1**) have become available.[1] The conversion of azetidinone **1** to broad-spectrum antibiotic penems has been established by various groups.[2] This conversion usually requires the protection of the secondary hydroxy group. The trimethylsilyl (Me_3Si) group has been used for this purpose,[2a] but this group is not sufficiently stable. Though the *tert*-butyldimethylsilyl (TBDMS) group is stable,[2b] the group itself as well as the reagent reported for its removal, $(Bu)_4N^+F^-$, are not cost effective.

The analysis of the penem synthesis reported above[2a,b] indicated that the alcohol protecting group should be stable to basic conditions and that this group should be cleaved under mild acidic conditions. We report here that the tetrahydropyranyl group meets the above needs and that it can be used for an efficient synthesis of penems such as **9**. This synthesis is outlined in Scheme I.

The N-alkylation of **1** progressed smoothly in good yield to give **2** as a white foam. The protection of the hydroxy moiety of **2** with dihydropyran under the standard conditions[3] gave a 1:1 mixture of diastereomeric hydroxy-protected **3** as an oil in quantitative yield. The removal of the triphenylmethyl group of **3** was capricious. The quantity of methanol used in this reaction and the reaction time determined the yield of the reaction. When the reaction was run in methanol, the formation of **4** appeared to take place in the beginning of the reaction as judged by TLC, but **4** decomposed with time. An excellent yield for the removal of the triphenylmethyl group was realized when the reaction was carried out in CH_3CN with only 1 equiv of MeOH. The silver thiolate **4** was not isolated but was converted to the next intermediate.

Imidazole has been reported as a leaving group for the synthesis of penems.[2a] The reaction of azetidinone **4** with 1,1′-carbonothioylbis(1*H*-imidazole) (CTBI) proceeded in moderate yield to give **5** as a yellow oil. The reaction of azetidinone **4** with *O*-2-naphthalenyl carbonochloridothioate (NCCT) progressed in excellent yield to give **6** as an oil. The cyclization of **5** as well as **6** proceeded smoothly at low temperatures. In the case of **5**, after cyclization, the removal of the resultant imidazole from unstable and acid-sensitive **7** was necessary in order to avoid complications in the subsequent reactions. Some deprotection and some decomposition of **7** resulted during this workup as judged by TLC and NMR. Removal of β-naphthol from a solution of **7** after the cyclization of **6** was not necessary. Only some deprotection was apparent in the latter case as judged by TLC and NMR. Since NCCT is a stable solid, is readily available, and reacts with **4** in excellent yield whereas CTBI is an unstable solid, is difficult to purify, reacts with **4** in only moderate yield, and necessitates the removal of imidazole from a solution of **7**, NCCT is preferable over CTBI in the penem synthesis.

Compound **7**, derived from **5** or **6** (partially decomposed when derived from **5**), was S-alkylated in excellent yields to give **8** as an oil. For the purpose of characterization, **8** derived from **6** was chromatographed on a column of silica gel to separate it from β-naphthol. This resulted in some loss of material on the column and some deprotection that resulted in lowering of the yield.[4] Though the THP deprotection (of **8**) was possible with acetic acid in aqueous ethanol,[5] this reaction was very sluggish. THP deprotection with pyridinium *p*-toluenesulfonate (PPTS)[3] was rapid in the beginning of the reaction, but it slowed down considerably with time and hence at the end of 8 h *p*-toluenesulfonic acid (PTSA)[6] was added to the reaction mixture to accelerate the conversion of the remaining amount of **8** to **9**. This conversion progressed in excellent yield. Compound **9** thus obtained was similar to authentic **9** in melting point, NMR, IR, and TLC behavior.[7] The technique for the removal of the allyl protecting group from penems **9** to give the sodium salt of penems **10** has been reported previously.[7,8]

In conclusion, the synthesis of penems via the use of THP for the protection of the hydroxy group gave excellent overall yield. THP is compatible with the above synthesis, and it is cost effective. The use of β-naphthol was found to be superior to the use of imidazole as a leaving group in the above synthesis.

Experimental Section

The ^1H NMR spectra were recorded on a Varian FT-80 or Varian XL 200. Chemical shifts are expressed in parts per million downfield from Me_4Si, and coupling constants are recorded in hertz. The infrared spectra were recorded on a Perkin-Elmer 1320 or Nicolet MX-IE FTIR spectrophotometer. Elemental microanalyses were conducted by Schering Analytical Research Services. Melting points were recorded on a Fisher-Johns hot-plate apparatus. The term flash chromatography refers to the method described by Still.[9] Dry THF was obtained by distillation from sodium benzophenone ketyl.

(3*S*,4*R*)-1-[[(Allyloxy)carbonyl]methyl]-3-[(*R*)-1-hydroxyethyl]-4-[(triphenylmethyl)thio]-2-azetidinone (2). Step a. To a stirred solution of 0.38 g (0.97 mmol) of **1** in 7 mL of CH_3CN at 25 °C under N_2 was added 0.44 g (1.95 mmol) of allyl iodoacetate followed by 0.40 g (1.23 mmol) of Cs_2CO_3. The reaction mixture was stirred at room temperature for 2 h and then at 40 °C for 1 h, then diluted with 90 mL of ether, and washed

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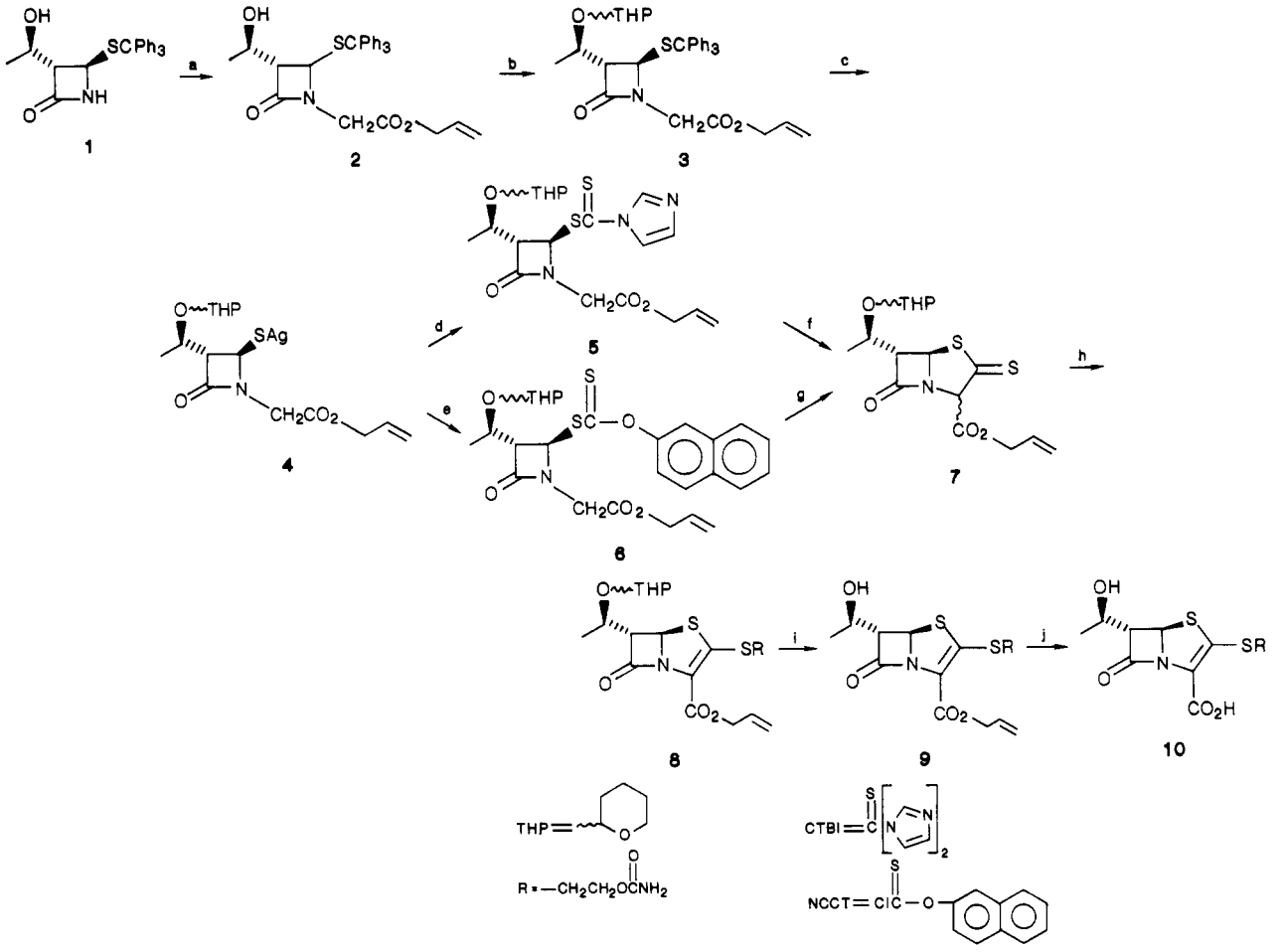
0022-3263/86/1951-4488$01.50/0

Scheme I



thoroughly with water containing traces of Na_2SO_3. The organic phase was dried over Na_2SO_4 and concentrated in vacuo to give 0.42 g (90%) of the title compound as a white foam, suitable for further reactions.

The analytical sample was purified by flash column chromatography [silica gel, ether/petroleum ether (44:55)]: NMR ($CDCl_3$) δ 1.2 (d, 3 H, J = 7 Hz, CH_3CH), 1.85 (d, 1 H, J = 6 Hz, OH, exchanged with D_2O), 2.8 and 3.75 (d, 2 H, J = 18 Hz, N-CH_2), 3.4 (dd, 1 H, J = 3 and 6 Hz, H3), 3.95 (m, 1 H, $CHCH_3$), 4.45 (m, 3 H, H4 and $COOCH_2$), 5.25 (m, 2 H, $=CH_2$), 5.75 (m, 1 H, CH=), 7.05–7.45 (m, 15 H, phenyl); $[\alpha]_D$ −123.4 (c 0.52, Me_2SO) IR (KBr) 3340, 1770, 1740 cm^{-1}; MS (CI, NH_3) m/e 488 $(M + H)^+$, 505 $(M + NH_4)^+$. Anal. Calcd for $C_{29}H_{29}N_1O_4S$: C, 71.47; H, 5.95; N, 2.87; S, 6.57. Found: C, 70.97; H, 6.16; N, 2.75; S, 6.43.

(3*S*,4*R*)-1-[[(Allyloxy)carbonyl]methyl]-3-[(*R*)-1-(tetrahydropyranyloxy)ethyl]-4-[(triphenylmethyl)thio]-2-azetidinone (3). Step b. To a stirred solution of 1.36 g (2.79 mmol) of **2** in 12 mL of CH_2Cl_2 at room temperature was added 0.35 g (4.16 mmol) of dihydropyran followed by 0.07 g (0.27 mmol) of pyridinium p-toluenesulfonate. The reaction mixture was stirred at 30 °C for 5 h, and CH_2Cl_2 was removed in vacuo. The resultant oil was taken in 100 mL of Et_2O, washed with brine followed by distilled water, dried over Na_2SO_4, and then concentrated in vacuo to give 1.59 g (quantitative) of the title compound as a glassy solid, suitable for further reactions: NMR ($CDCl_3$) δ 1.15 (two d, J = 6.5 Hz, CH_3CH), 1.55 (br, THP methylenes), 3.05 and 3.75 (two sets of d, J = 18 and 3 Hz, N-CH_2), 3.4 (m, H3), 4.45 (m, H4 and $COOCH_2$), 5.2 (m, $=CH_2$), 5.75 (m, CH=), 7.1–7.5 (m, phenyl); MS (FAB, thioglycerol) m/e 572 $(M + H)^+$.

(3*S*,4*R*)-1-[[(Allyloxy)carbonyl]methyl]-3-[(*R*)-1-(tetrahydropyranyloxy)ethyl]-4-(argentiothio)-2-azetidinone (4). Step c. A cold (0 °C) solution of 0.223 g (1.31 mmol) of $AgNO_3$ dissolved in a mixture of 0.116 g (1.42 mmol) of pyridine, 0.04 g (1.31 mmol) of methanol, and 3 mL of acetonitrile was added to a cold (0 °C) stirred solution of 0.75 g (1.31 mmol) of **3** in 6 mL of CH_3CN kept in the dark and under N_2. The reaction mixture was stirred at room temperature for 18 h, and then the solvents were removed in vacuo. The resultant viscous oil was dissolved in 60 mL of CH_2Cl_2, washed with 2 × 30 mL of double-distilled, deionized water, dried over $MgSO_4$, and concentrated in vacuo to give 0.80 g of an oil containing **4** and triphenylmethyl methyl ether. Compound **4** was not isolated, but the CH_2Cl_2 solution of the above oil was used for the further reactions.

(3*S*,4*R*)-1-[[(Allyloxy)carbonyl]methyl]-3-[(*R*)-1-(tetrahydropyranyloxy)ethyl]-4-[[imidazol-1-yl(thiocarbonyl)]thio]-2-azetidinone (5). Step d. To a stirred solution of 0.46 g of the mixture of **4** and triphenylmethyl methyl ether [obtained from 0.43 g (0.75 mmol) of **3** as described above in step c] in 10 mL of CH_2Cl_2 under N_2 at room temperature was added a solution of 0.22 g (90% pure, 1.11 mmol) of 1,1-carbonothioylbis(1*H*-imidazole) in 10 mL of CH_2Cl_2 slowly over 5 min. The reaction mixture was stirred for 1 h and then filtered. The solid residue was washed twice with 10 mL of CH_2Cl_2, and the combined filtrate was concentrated in vacuo to a yellow oil. Silica gel column chromatography [EtOAc/CH_2Cl_2 (10:90 changing to 30:70)] of this oil gave 0.22 g (67% from **3**) of the title compound as a yellow oil (due to streaking on the column, about 0.04 g (12%) of the product was left on the column): NMR ($CDCl_3$) δ 1.3 (two d, J = 7 Hz, CH_3CH), 1.65 (br, THP methylenes), 3.55 (m, H3), 3.85 and 4.25 (two d, J = 18 Hz, N-CH_2), 4.7 (m, $COOCH_2$), 5.3 (m, $=CH_2$), 5.8 (m, CH=), 6.1 (d, J = 1.5 Hz, H4), 7.1 (m, Im), 7.8 (m, Im), 8.45 (m, Im); MS (FAB, thioglycerol) m/e 440 $(M)^+$, 296 (M − SCSIm).

(3*S*,4*R*)-1-[[(Allyloxy)carbonyl]methyl]-3-[(*R*)-1-(tetrahydropyranyloxy)ethyl]-4-[[β-naphthoxy(thiocarbonyl)]thio]-2-azetidinone (6). Step e. To a stirred solution of 0.23 g of the mixture of **4** and triphenylmethyl methyl ether (obtained from 0.21 g (0.36 mmol) of **3** as described in step c above) in 5 mL of CH_2Cl_2 at 0 °C under N_2 was added a cold (0 °C) solution of 0.08 g (0.36 mmol) of *O*-2-naphthalenyl carbonochloridothioate in 3 mL of CH_2Cl_2 over 5 min. The reaction mixture was stirred for 30 min and filtered through Celite to remove AgCl. The Celite

pad was washed twice with 10 mL of CH_2Cl_2, and the combined filtrate was concentrated in vacuo to obtain a red oil. Chromatographic separation of the title compound was achieved by using a short silica gel flash column [CH_2Cl_2 followed by CH_2Cl_2/EtOAc (9:1)] to obtain 0.17 g (94% from **3**) of **6** as an oil: NMR ($CDCl_3$) δ 1.3 (two d, J = 7 Hz, CH_3CH), 1.6 (br, THP methylenes), 3.5 (dd, J = 3 and 6 Hz, H3), 3.9 and 4.4 (two d, J = 18 Hz, N-CH_2), 4.55 (m, $COOCH_2$), 5.2 (m, $=CH_2$), 5.8 (m, CH=), 6.05 (d, J = 2 Hz, H4), 7.2-8 (m, aromatic); MS (FAB, thioglycerol) m/e 516 $(M + H)^+$.

Allyl (5*R*,6*S*)-2-[[2-(Carbamoyloxy)ethyl]thio]-6-[(*R*)-1-(tetrahydropyranyloxy)ethyl]penem-3-carboxylate (8). Steps f-h. (A) From 6. Step g. To a vigorously stirred solution of 0.89 g (1.72 mmol) of **6** in 40 mL of dry THF at -78 °C was added 2.5 mL (2.5 mmol) of 1 M $LiN(SiMe_3)_2$ in hexane. The reaction mixture was stirred for 10 min, and then 1.27 mL of glacial CH_3COOH followed by 85 mL of EtOAc was added. This cold (0 °C) solution was quickly washed with 25 mL of 2% aqueous tartaric acid solution followed by 30 mL of cold (5 °C) distilled H_2O. The cold aqueous phases were back-extracted with cold (0 °C) EtOAc; the EtOAc extract was treated as above and combined with EtOAc/THF extract. The combined, cold (0 °C) organic phase was dried over $MgSO_4$ and then concentrated in vacuo at $\leq$0 °C to obtain 0.93 g of an orange oil containing **7** and β-naphthol. Compound **7** was not isolated from this oil but was directly converted to **8** as given below.

Step h. To a stirred solution of the above oil in 11 mL of THF was added 0.41 g (2.06 mmol) of iodoethyl carbamate, followed by 0.17 g (2.06 mmol) of $NaHCO_3$ in 2 mL of H_2O. Approximately 1 mL of CH_3CN was added after 5 min to avoid the formation of two phases. The reaction mixture was stirred at room temperature for 18 h, diluted with 120 mL of CH_2Cl_2, and washed thoroughly with 25 mL of brine containing traces of Na_2SO_3, followed by 25 mL of distilled H_2O. The organic phase was dried over Na_2SO_4 and concentrated in vacuo to obtain 1.05 g (quantitative from **6**) of oil consisting of the title compound and β-naphthol.

For the purpose of characterization, the above oil was column chromatographed [silica gel, CH_2Cl_2 followed by CH_2Cl_2/MeOH (96:4)] to obtain 0.58 g (73.4% from **6**) of **8** as a viscous oil and 0.03 g (4.7% from **6**) of **9** (resulting from tetrahydropyranyl ether cleavage during the workup and column chromatography). **8**: NMR ($CDCl_3$) δ 1.3 (two d, J = 7 Hz, CH_3CH), 1.6 (br, THP methylenes), 3.15 (m, SCH_2), 3.75 (dd, J = 1.5 and 6 Hz, H6), 4.3 (m, CH_2OCO), 4.6 (m, CO_2CH_2), 5.3 (m, $=CH_2$), 5.6 (d, J = 1.5 Hz, H7), 5.8 (m, CH=), 7.2 (br, 2 H, $CONH_2$); MS (FAB, thioglycerol) m/e 458 $(M)^+$.

(B) From 5. Step f. A solution of 0.09 g (0.20 mmol) of **5** in 15 mL of dry THF was converted to **7** as described above under step g with one difference. After the reaction mixture was quenched with 0.15 mL of glacial CH_3COOH, the EtOAc solution was washed thoroughly with 30 mL of 2% aqueous tartaric acid to remove imidazole. This reaction gave 0.07 g of **7** as an orange oil. [TLC [silica gel, EtOAc/CH_2Cl_2 (2:8)] of this oil indicated slight desilylation and slight decomposition. See text for details.]

Step h. Compound **7** was converted to compound **8** as described above in 85% (from **5**) yield.

Allyl (5*R*,6*S*)-2-[[2-(Carbamoyloxy)ethyl]thio]-6-[(*R*)-1-hydroxyethyl]penem-3-carboxylate (9). Step i. To a stirred solution of 0.35 g (0.76 mmol) of **8** in 10 mL of 80% aqueous EtOH under N_2 was added 0.04 g (0.15 mmol) of pyridinium p-toluenesulfonate. The reaction mixture was stirred at 45° C for 8 h, and then 0.03 g (0.15 mmol) of p-toluenesulfonic acid was added. The reaction mixture was stirred for 1 h more, and the solvent was removed in vacuo. The resultant oil was taken in 120 mL of EtOAc and washed with distilled H_2O, distilled H_2O containing a small amount of $NaHCO_3$, and finally distilled H_2O. The organic phase was dried over Na_2SO_4 and then concentrated in vacuo to obtain a yellow solid. Silica gel column chromatography [MeOH/CH_2Cl_2 (5:95)] of this solid gave 0.27 g (96%) of the title compound as a white solid.

The analytical sample was crystallized from CH_3CN: mp 152-152.5 °C; NMR (Me_2SO-D_6/$CDCl_3$) δ 1.17 (d, 3 H, J = 7 Hz, CH_3CH), 3.15 (m, 2 H, SCH_2), 3.65 (dd, 1 H, J = 1.5 and 6 Hz, H6), 3.8-4.3 (m, 3 H, CH_3CH and CH_2OCO), 4.6 (m, 2 H, $COOCH_2$), 5.1-5.4 (m, 2 H, $=CH_2$), 5.65 (d, 1 H, J = 1.5 Hz, H5), 5.75 (m, 1 H, CH=), 6.45 (br, 2 H, $CONH_2$); IR (Nujol) 3420, 3305, 1780, 1690, 1610 cm^{-1}; MS (FAB, thioglycerol) m/e 375 $(M + H)^+$; $[\alpha]_D$ +188.9 (c 0.46, Me_2SO). Anal. Calcd for $C_{14}H_{18}N_2O_6S_2$: C, 44.93; H, 4.81; N, 7.48; S, 17.13. Found: C, 44.88; H, 4.84; N, 7.44; S, 16.85.

(5*R*,6*S*)-2-[[2-(Carbamoyloxy)ethyl]thio]-6-[(*R*)-1-hydroxyethyl]penem-3-carboxylic Acid (10). Step j. The allyl ester deblocking on penems was accomplished as reported before:[7,8] NMR (Me_2SO-d_6) δ 1.15 (d, 3 H, J = 7 Hz, CH_3CH), 3.13 (m, 2, SCH_2), 3.76 (dd, 1 H, J = 1.52 Hz, H_6), 3.98 (br, H, $CHCH_3$), 4.12 (m, 2, CH_2OCO), 5.68 (d, 1 H, J = 1.5 Hz, H_5), 6.55 (br, 2 H, NH_2); IR (CH_2Cl_2) 3400, 1790, 1710, 1690 cm^{-1}; MS (FAB, thioglycerol) m/e 335 (M + H); $[\alpha]_D$ +208.7 (c 0.3, DMF).

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Synthesis of β-Benzyl *N*-(*tert*-Butoxycarbonyl)-L-*erythro*-β-(benzyloxy)aspartate from (*R*,*R*)-(+)-Tartaric Acid

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Various diastereomers of β-hydroxyaspartic acid occur in microorganisms and in fungi as free amino acids[1] and as constituents of peptides.[2] The free amino acid has also been found in urine from several mammals, including man.[3] Recently, L-*erythro*-β-hydroxyaspartic acid was found in several of the vitamin K dependent plasma proteins involved in the blood clotting cascade.[4] The role of this amino acid in these proteins is unknown. To facilitate further investigations of the biological role of β-hydroxyaspartic acid, we have developed a synthesis of β-benzyl-*N*-(*tert*-butoxycarbonyl)-L-*erythro*-β-(benzyloxy)aspartate (**8**), a derivative suitable for "solid-phase" peptide synthesis.

One of the main synthetic approaches to *erythro*-β-hydroxyaspartic acid has relied on the ammonolysis of *trans*-epoxysuccinic acid. Previously, resolution of the product obtained from racemic *trans*-epoxysuccinic acid was required.[5] Preparations of the chiral *trans*-epoxysuccinic acid esters have recently been reported.[6] Starting from (*R*,*R*)-(+)-tartaric acid and proceeding via diethyl (-)-*trans*-epoxysuccinate, L-*erythro*-β-hydroxyaspartic acid is thus obtained[7] in a procedure involving three consecutive inversions of one of the stereocenters in (*R*,*R*)-(+)-tartaric acid. We now report a synthesis of β-benzyl *N*-(*tert*-butoxycarbonyl)-L-*erythro*-β-(benzyloxy)aspartate (**8**)

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