Large Laboratory Scale Synthesis of (2*S*,3*S*)-2-(4-Methoxybenzyloxy)-3,4-*O*-(3-pentylidene)-1,3,4-butantriol, a Versatile Chiral Building Block in Natural Product Synthesis[1)]

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(2*S*,3*S*)-2-(4-Methoxybenzyloxy)-3,4-*O*-(3-pentylidene)-1,3,4-butanetriol (3), a versatile chiral building block for an essential structural unit of complex natural products, was synthesized starting from dimethyl L-(+)-tartrate (5) on both a small scale and a large scale in good yield.

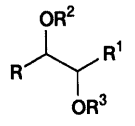
Keywords chiral building block; tartaric acid; protective group; acetal; diisobutylaluminum hydride

In multistep synthesis of complex natural products *via* fragment condensation, we usually need a variety of chiral building blocks, which are easily available in a large quantity as well as versatile and generally useful as starting materials. A structural unit with a vicinal C–O band (**1**) is often found in complex natural products, especially in polyketide-derived compounds, *e.g.*, in the antitumor polyether macrolides halichondrins,[2)] **1** appears seven times. Among four possible stereoisomeric forms of **1**, **2** is the most common and is expected to be readily synthesized from L-(+)-tartaric acid, which is identical with **2** with respect to configuration. Among carbohydrates, amino acids, hydroxyl acids, and terpenes, tartaric acid is one of the most versatile and convenient natural products as a chiral source. In the course of our synthetic study of halichondrin B, we required a large amount of a starting material with the configuration of **2**, which appears at three parts, C32–C33, C40–C41 and C47–C48, in halichondrin B.

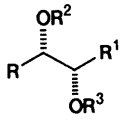
Enantiomerically pure 1,2-diol compounds can be readily synthesized from olefins by two typical oxidation methods, asymmetric epoxidation–regioselective epoxide ring opening[3)] and asymmetric dihydroxylation.[4)] In order to synthesize **2**, especially on a large scale, however, conversion of L-(+)-tartaric acid is much more practically preferred bacause it is one of the least expensive enantiomerically pure natural products and is available in large quantities. In this paper we report a synthesis of **3**, a versatile and easily handled chiral building block, on both a small scale and a large scale. Among several precedents for the preparation of chiral building blocks containing **2** starting from L-(+)-tartaric acid,[5)] Takano *et al.* synthesized variously protected derivatives such as **4**,[5d)] and **3** may provide an additional and generally useful building block.

Dimethyl L-(+)-tartrate (**5**) was treated with *p*-anisaldehyde dimethyl acetal in the presence of *p*-toluenesulfonic acid (TsOH) in the usual manner to give **6**[6)] in almost quantitative yield. Treatment of **6** with a mixture of lithium aluminum hydride ($LiAlH_4$) and aluminum chloride ($AlCl_3$) under the conditions successfully empolyed for the preparation of **4**[5a,c)] gave only a complex mixture. Reduction of **6** with $LiAlH_4$ readily gave the diol (**8**), which can be converted to **7**. However, since **7** is rather soluble in water, protection of the diol prior to reductive cleavage of the acetal ring was much better as an experimental procedure, and its selection is sometimes crucial. The diol of **8** was protected as a di-*tert*-butyldimethylsilyl (TBS) ether (**9**) under usual conditions. The five-membered *p*-methoxybenzylidene acetal of **9** was subjected to reductive cleavage with diisobutylaluminum hydride (DIBAH)[7)] to give the *p*-methoxybenzyl (MPM) ether (**10**) in good yield. Treatment of **10** with *dl*-camphorsulfonic acid (CSA) at room temperature gave the crude triol (**7**), which was dissolved in benzene without purification and treated with 3,3-dimethoxypentane[8)] in the presence of TsOH to give the title compound (**3**) in excellent yield without any detectable formation of the isomeric six-membered acetal.

For a large-scale synthesis it is necessary to minimize the number of purification steps, and chromatographic separation must be particularly avoided. In the kilogram-scale synthesis of **3** starting from **5**, we purified only one (**6**) among six intermediates by recrystallization at the



1



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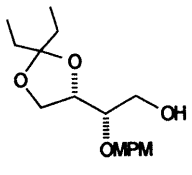
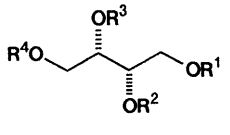


3



4a : $R^1=R^2=R^3=Bn$, $R^4=H$
4b : $R^1=R^2=Bn$, $R^3=R^4=H$
4c : $R^1=R^3=Bn$, $R^2=R^4=H$

Chart 1

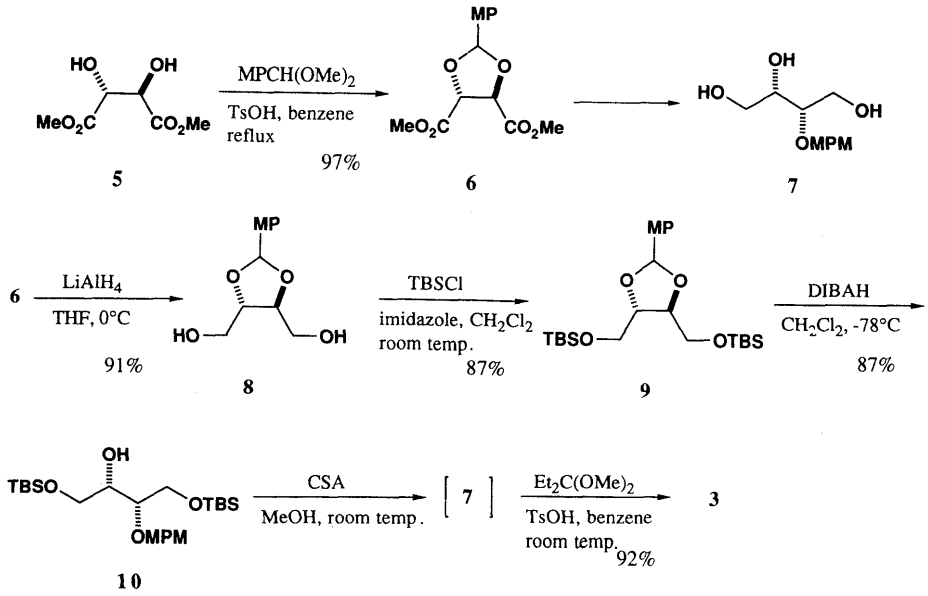


Chart 2

first step, and all other steps until the final product (**3**) were carried out without purification. Finally, crude **3** was converted to the 3,5-dinitrobenzoate, which was recrystallized from *n*-hexane–ethyl acetate and then hydrolyzed with alkali to give purified **3**. The overall yield of **3** from **5** was 63%.

Experimental

Optical rotations were measured with a JASCO DIP-370 digital polarimeter. IR spectra were taken with a JASCO IRA-2 spectrometer. ^{1}H- and ^{13}C-NMR spectra were recorded on a JEOL JNM GX-270 or JEOL EX-400 specrometer. Mass spectra were measured using a JEOL JMX DX-300 or JEOL JMX HX-100 spectrometer.

(4*S*,5*S*)-4,5-Bis(carbomethoxy)-2-(4-methoxyphenyl)-1,3-dioxolane (6) A flask equipped with a Dean–Stark trap containing molecular sieves was charged with a solution of dimethyl L-(+)-tartrate (**5**) (45.0 g, 0.25 mol) in benzene (500 ml), *p*-anisaldehyde dimethyl acetal (134.2 g, 0.74 mol) and TsOH·H_2O (1.0 g, 5.3 mmol). This mixture was refluxed for 16 h under argon, and allowed to cool to room temperature. After addition of Et_3N (1 ml), the mixture was evaporated *in vacuo*. The residue was chromatographed on a silica gel column (*n*-hexane–EtOAc 3 : 1—1 : 1) to give **6** as colorless fine prisms (72.8 g, 97%), mp 71—72 °C (*n*-hexane–Et_2O), $[\alpha]_D^{27}$ −35.1° (*c* = 1.11, MeOH). IR $\nu_{max}^{CH_2Cl_2}$ cm^{-1}: 3060, 2960, 1750, 1620, 1520, 1440, 1255, 1175, 1105, 1035. ^{1}H-NMR ($CDCl_3$) δ: 3.82 (s, 3H), 3.84 (s, 3H), 3.87 (s, 3H), 4.84 (d, 1H, *J* = 4.0 Hz), 4.96 (d, 1H, *J* = 4.0 Hz), 6.10 (s, 1H), 6.87—6.96 (m, 2H), 7.47—7.55 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ: 52.82, 52.84, 55.29, 77.20, 77.34, 106.72, 113.78, 127.34, 128.72, 160.95, 169.54, 170.14. MS *m/z* (%): 296 (M^+, 5.6), 295 (16), 237 (36), 152 (94), 135 (100), 121 (45), 108 (25), 77 (12), 59 (10). *Anal.* Calcd for $C_{14}H_{16}O_7$: C, 56.76; H, 5.44. Found: C, 56.51; H, 5.21.

(2*S*,3*S*)-2,3-*O*-(4-Methoxybenzylidene)-1,2,3,4-butanetetraol (8) A solution of **6** (10.0 g, 33.8 mmol) in THF (100 ml) was added dropwise to a stirred suspension of $LiAlH_4$ (2.5 g, 65.9 mmol) in THF (25 ml) at 0 °C under argon, and the mixture allowed to warm to room temperature with stirring. After 1 h, the reaction was quenched by careful addition of MeOH, and then 15% NaOH (2.5 ml) was added. After 30 min, H_2O (7.5 ml) was added, and the stirring was continued for 30 min. The mixture was dried over Na_2SO_4, filtered with the aid of Celite, and washed with CH_2Cl_2. The filtrate was evaporated *in vavuo*, and the residue was chromatographed on a silica gel column (CH_2Cl_2–MeOH 10 : 1) to give **8** as a colorless viscous oil (7.4 g, 91%), which solidified to a crystalline powder on standing in a refrigerator, mp 52—53 °C, $[\alpha]_D^{25}$ +10.5° (*c* = 1.92, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 3360, 2930, 1615, 1520, 1440, 1390, 1305, 1250, 1175, 1025. ^{1}H-NMR ($CDCl_3$) δ: 1.88—2.16 (bt, 2H, *J* = 6.0 Hz), 3.72—4.00 (m, 4H), 3.81 (s, 3H), 4.14—4.24 (m, 2H), 5.93 (s, 1H), 6.86—6.97 (m, 2H), 7.35—7.46 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ: 55.31, 62.29, 62.40, 78.29, 79.23, 103.82, 113.69, 113.86, 127.99, 129.28, 160.62. MS *m/z* (%): 240 (M^+, 21), 239 (65), 209 (33), 137 (83), 135 (100), 121 (36). HR-MS Calcd for $C_{12}H_{16}O_5$: 240.0970. Found: 240.0999. *Anal.* Calcd for $C_{12}H_{16}O_5$: C, 59.99; H, 6.71. Found: C, 59.83; H, 6.60.

(2*S*,3*S*)-1,4-Bis(*tert*-butyldimethylsilyloxy)-2,3-*O*-(4-methoxybenzylidene)-2,3-butanediol (9) Imidazole (25.7 g, 0.38 mol) and TBS chloride (57.0 g, 0.38 mol) were added to a stirred solution of **8** (41.3 g, 0.17 mol) in CH_2Cl_2 (600 ml) at 0 °C under argon. The reaction mixture was stirred at room temperature for 30 min, then the reaction was quenched with H_2O, and the whole was extracted with CH_2Cl_2. The extract was dried over Na_2SO_4 and evaporated *in vauco* to leave an oil, which was chromatographed on a silica gel column (*n*-hexane–EtOAc 10 : 1) to give **9** as a colorless oil (78.5 g, 97%), $[\alpha]_D^{28}$ +10.5° (*c* = 1.11, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 2900, 2850, 1610, 1520, 1460, 1250, 1080. ^{1}H-NMR ($CDCl_3$) δ: 0.08 (s, 12H), 0.90 (s, 9H), 0.92 (s, 9H), 3.74—3.90 (m, 4H), 3.80 (s, 3H), 4.08 (dt, 1H, *J* = 6.0, 4.0 Hz), 4.18 (dt, 1H, *J* = 5.0, 6.0 Hz), 5.91 (s, 1H), 6.85—6.90 (m, 2H), 7.24—7.42 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ: −5.38, 18.32, 25.90, 55.29, 63.93, 64.01, 79.15, 104.03, 113.62, 128.14, 130.03, 160.38. MS *m/z* (%): 468 (M^+, 7.1), 467 (13), 411 (7.8), 143 (23), 117 (86), 89 (100), 73 (99). HR-MS (FAB) Calcd for $C_{24}H_{44}NaO_5Si_2$ (M^+ + Na): 491.2625. Found: 491.2633. HR-MS Calcd for $C_{24}H_{44}O_5Si_2$: 468.2730. Found: 468.2765.

(2*S*,3*S*)-1,4-Bis(*tert*-butyldimethylsilyloxy)-3-(4-methoxybenzyloxy)-2-butanol (10) A 0.94 M solution of DIBAH in *n*-hexane (19.0 ml, 17.9 mmol) was added dropwise to a stirred solution of **9** (1.64 g, 3.5 mmol) in CH_2Cl_2 (16 ml) at −78 °C under argon. After 1 h, the reaction mixture was allowed to warm to −30 °C, and stirred for 30 min, the reaction was quenched with MeOH. Brine was added, and the mixture was extracted with CH_2Cl_2. The extract was dried over Na_2SO_4 and evaporated *in vacuo* to leave an oil, which was chromatographed on a silica gel column (*n*-hexane–EtOAc 3 : 1) to give **10** as a colorless oil (1.34 g, 81%), $[\alpha]_D^{27}$ +23.4° (*c* = 1.16, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 3480, 2930, 2850, 1615, 1520, 1465, 1390, 1250, 1175, 1100. ^{1}H-NMR ($CDCl_3$) δ: 0.05 (s, 6H), 0.06 (s, 6H), 0.89 (s, 9H), 2.53 (d, 1H, *J* = 6.0 Hz), 3.55—3.85 (m, 6H), 3.80 (s, 3H), 4.53 (d, 1H, *J* = 11.0 Hz), 4.69 (d, 1H, *J* = 11.0 Hz), 6.85—6.89 (m, 2H), 7.24—7.28 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ: −5.45, −5.37, 16.23, 25.88, 55.26, 63.05, 63.78, 71.71, 72.89, 78.03, 113.75, 129.55, 130.66, 159.24. MS *m/z* (%): 470 (M^+, 0.3), 469 (1.1), 364 (2.2), 122 (10), 121 (100). HR-MS (FAB) Calcd for $C_{24}H_{46}NaO_5Si_2$ (M^+ + Na): 493.2781. Found: 493.2766.

(2*S*,3*S*)-2-(4-Methoxybenzyloxy)-3,4-*O*-(3-pentylidene)-1,3,4-butanetriol (3) A solution of **10** (115.8 mg, 0.24 mmol) and *dl*-CSA (4 mg, 0.02 mmol) in MeOH (1 ml) was stirred at room temperature for 1 h. After addition of Et_3N (20 μl), the mixture was evaporated and dried *in vacuo*. The resulting crude triol was dissolved in benzene (1 ml), and to this solution were added 3,3-dimethoxypentane (15 μl, 0.49 mmol) and TsOH·H_2O (5.3 mg, 0.03 mmol). The reaction mixture was stirred at room temperature for 3 h, then the reaction was quenched with Et_3N

(20 μl), and the mixture was evaporated *in vacuo*. The residue was chromatographed on a silica gel column (*n*-hexane–EtOAc 3 : 1) to give **3** as a colorless oil (67.9 mg, 92%), $[\alpha]_D^{23}$ −28.3° (c=1.07, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 3450, 2950, 1600, 1500, 1240. ^{1}H-NMR ($CDCl_3$) δ: 0.90 (t, 3H, J=7.5 Hz), 0.91 (t, 3H, J=7.5 Hz), 1.59—1.72 (m, 4H), 2.13 (br s, 1H), 3.52—3.59 (m, 2H), 3.66 (m, 1H), 3.69 (dd, 1H, J=8.0, 8.5 Hz), 3.81 (s, 3H), 4.00 (dd, 1H, J=6.5, 8.0 Hz), 4.26 (ddd, 1H, J=6.0, 6.5, 8.5 Hz), 4.62 (d, 1H, J=11.5 Hz), 4.75 (d, 1H, J=11.5 Hz), 6.86—6.91 (m, 2H), 7.28—7.32 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ: 8.07, 8.22, 29.13, 29.59, 55.27, 61.69, 66.18, 72.47, 77.27, 79.05, 113.35, 113.88, 129.57, 130.34, 159.35. MS m/z (%): 310 (M^+, 0.1), 295 (0.2), 281 (2.8), 224 (7.3), 193 (2.8), 129 (15), 121 (100). HR-MS Calcd for $C_{17}H_{26}O_5$: 310.1795. Found: 310.1604.

Large-Scale Synthesis of 3 A solution of **5** (1021 g, 5.73 mol), *p*-anisaldehyde dimethyl acetal (2089 g, 11.46 mol), and $TsOH \cdot H_2O$ (22 g, 0.12 mol) in benzene (5 l) in a vessel equipped with a Dean–Stark trap containing molecular sieves was heated under reflux for 27 h under argon. The reaction mixture was allowed to cool to room temperature, then the reaction was quenched with Et_3N (20 ml), and the mixture was evaporated *in vacuo*. The residue was recrystallized from *n*-hexane–Et_2O to give **6** as colorless fine crystals (1417 g, 83%).

A solution of **6** (600 g, 2.03 mol) in THF (2.5 l) was added dropwise to a stirred suspension of $LiAlH_4$ (150 g, 3.95 mol) in THF (2 l) at 0 °C under argon. After 1 h, the reaction was quenched with MeOH, then H_2O (150 ml), 15% NaOH (150 ml) and H_2O (350 ml) were added, and stirring was continued for 30 min. A large amount of Celite (*ca.* 2 kg) was added, and the mixture was centrifuged. The remaining solid materials were extracted five times with CH_2Cl_2 by repeated stirring in CH_2Cl_2 and centrifugal,separation. The combined extracts were dried over Na_2SO_4 and evaporated *in vacuo* to leave crude **8** as a yellow oil (482 g, 99%).

Imidazole (791 g, 11.62 mol) and TBS chloride (1632 g, 10.83 mol) were added to a stirred solution of **8** (1269 g, 5.28 mol) in CH_2Cl_2 (5 l) at 0 °C under argon. The reaction mixture was allowed to warm to room temperature, stirred for 1 h, then the reaction was quenched with brine, and the mixture was extracted with CH_2Cl_2. The extract was dried over Na_2SO_4 and evaporated *in vacuo* to leave crude **9** as a yellow oil (2494 g, 100%).

A 1.5 M solution of DIBAH in toluene (2.61, 3.9 mol) was added dropwise to a stirred solution of **9** (825 g, 1.76 mol) in CH_2Cl_2 (3 l) at −40 °C under argon. After 1 h, the excess reagent was decomposed with MeOH, and H_2O (555 ml) and 15% NaOH (555 ml) were added. The mixture was stirred for 30 min, then dried over Na_2SO_4, and filtered with the aid of Celite. The filtrate was evaporated *in vacuo* to leave crude **10** as a yellow oil (745 g, 90%).

A solution of **10** (2037 g, 4.33 mol) and *dl*-CSA (40 g, 0.17 mol) in MeOH (10 l) was stirred for 1.5 h at room temperature. After dropwise addition of Et_3N (48 ml), the reaction mixture was evaporated *in vacuo*. The residue was dissolved in benzene (7 l), and 3,3-dimethoxypentane (1350 g, 10.21 mol) and $TsOH \cdot H_2O$ (74 g) were added. The reaction mixture was stirred for 2 h at room temperature, then the reaction was quenched with Et_3N (110 ml), and the mixture was evaporated *in vacuo*. The residue was extracted with CH_2Cl_2. The extract was washed with 1 N HCl and brine, dried over Na_2SO_4, and evaporated *in vacuo* to leave crude **3** as a yellow oil (1280 g, 95%). 3,5-Dinitrobenzoyl chloride (1137 g, 4.93 mol) was added portionwise to a stirred solution of crude **3** (1280 g, 4.11 mol) and Et_3N (825 ml, 5.92 mol) in CH_2Cl_2 (7 l) at 0 °C. The reaction mixture was stirred for 30 min at room temperature, then the reaction was quenched with saturated aqueous $NaHCO_3$, and the mixture was extracted with CH_2Cl_2. The extract was washed with brine, dried over Na_2SO_4, and evaporated *in vacuo*. The residue was recrystallized from *n*-hexane–EtOAc to give the 3,5-dinitrobenzoate as colorless needles (1844 g, 89%), mp 81—82 °C, $[\alpha]_D^{27}$ −52.8° (c=0.13, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3020, 1740, 1635, 1620, 1555, 1520, 1470, 1355, 1290, 1260, 1220, 1175, 1085. ^{1}H-NMR ($CDCl_3$) δ: 0.92 (t, 3H, J=7.5 Hz), 0.95 (t, 3H, J=7.5 Hz), 1.66 (q, 2H, J=7.5 Hz), 1.71 (q, 2H, J=7.5 Hz), 3.70 (s, 3H), 3.80—3.93 (m, 2H), 4.07 (dd, 1H, J=7.5, 8.0 Hz), 4.32 (dd, 1H, J=7.5, 13.0 Hz), 4.45—4.56 (m, 2H), 4.60 (d, 1H, J=11.5 Hz), 4.72 (d, 1H, J=11.5 Hz), 6.70—6.73 (m, 2H), 7.21—7.24 (m, 2H), 9.03 (d, 2H, J=2.0 Hz), 9.23 (t, 1H, J=2.0 Hz). ^{13}C-NMR ($CDCl_3$) δ: 8.15, 8.27, 28.75, 29.44, 55.13, 65.80, 65.88, 72.88, 75.96, 76.18, 113.71, 113.75, 122.38, 129.35, 129.75, 129.89, 133.51, 148.57, 159.27, 162.31. MS (FAB) m/z (%): 527 (M^++Na, 9.5), 475 (4.8), 413 (27), 391 (33), 369 (5.6), 313 (4.0), 279 (6.5), 195 (9.7), 149 (69), 121 (100). HR-MS Calcd for $C_{24}H_{28}O_{10}N_2Na$ (M^++Na): 527.1642. Found: 527.1638. *Anal.* Calcd for $C_{24}H_{28}N_2O_{10}$: C, 57.14; H, 5.59; N, 5.55. Found: C, 57.08; H, 5.47; N, 5.56.

A 15% NaOH solution (1.5 l) was added slowly to a stirred solution of the 3,5-dinitrobenzoate (1421 g, 2.82 mol) in THF (2.5 l). The reaction mixture was stirred for 1 h at room temperature. The THF layer was separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, and evaporated *in vacuo*. The residue was passed through a short silica gel column (*n*-hexane–EtOAc 1 : 1) to give **3** as a colorless oil (882 g, 100%).

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