It should be pointed out that in the absence of Me_3SiCl no reduction occurs. And also the combination of $NaBH_4/Me_3SiCl$ was not effective. Reduction attempts using Lewis acids ($BF_3{\cdot}OEt_2$, $ZnCl_2$) were also fruitless.

Although there are several methods available for reductive cleavage of acetals and ketals, we believe that the present method offers considerable advantages in terms of simpleness, readily available reagents, and very mild conditions.[17]

Experimental Section

Melting points and boiling points are uncorrected. Kugelrohr distillation boiling points (bp) refer to the bath temperature. ^1H NMR spectra were recorded at 100 MHz on JEOL MH-100 with Me_4Si as an internal standard. High-resolution mass spectra were obtained with a JEOL HX-100 spectrometer. GLC analyses were performed on Shimadzu GC-7A flame ionization instruments with OV-1 or SE-30 columns.

Et_2O was distilled from sodium benzophenone ketyl before use, and CH_2Cl_2 was from CaH_2. Reagents were added via dry syringes through septa.

All starting derivatives in this study were prepared by known procedures[16] and purified by distillation. All products were identified through their ^1H NMR, IR, and high-resolution mass spectra.

General Procedure of the Reductive Cleavage of Acetals and Ketals with $Zn(BH_4)_2/Me_3SiCl$. To an Et_2O or CH_2Cl_2 solution (1.4 mL) of acetal or ketal (1 mmol) at 0 °C were added successively $Zn(BH_4)_2$ (0.15 M Et_2O solution; 3.4 mL, 0.5 mmol)[18] and Me_3SiCl (1.2 mmol). When Me_3SiCl was added, a small amount of gas evolution was observed. And the mixture was stirred at 0 °C or at room temperature until completion of the reaction, as monitored by TLC or GLC analysis. During the reaction the solution gradually turned cloudy white. After the reaction was quenched by addition of dilute HCl and conventional workup, the residue was purified by flash column chromatography[19] to provide the corresponding ethers.

Benzyl methyl ether (1): bp 68 °C (18 mmHg) [lit.[20] bp 59–60 °C (12 mmHg)]; IR (neat) 1435, 1385, 1100, 740, 700 cm^{-1}; ^1H NMR (CCl_4) δ 3.27 (3 H, s), 4.34 (2 H, s), 7.18 (5 H, s); mass spectrum, m/z (M^+) calcd for $C_8H_{10}O$ 122.0732, obsd 122.0747.

2-(Benzyloxy)ethanol (2): bp 72–73 °C (1 mmHg) [lit.[3a] bp 135 °C (13 mmHg)]; IR (neat) 3400, 1450, 1355, 1105, 1060, 735, 695 cm^{-1}; ^1H NMR (CCl_4) δ 2.92 (1 H, br s), 3.50 (4 H, m), 4.42 (2 H, s), 7.19 (5 H, s); mass spectrum, m/z (M^+) calcd for $C_9H_{12}O_2$ 152.0837, obsd 152.0824.

2-[(4-Methoxyphenyl)methoxy]ethanol (3): bp 146–147 °C (4.5 mmHg) [lit.[21] bp 109 °C (0.5 mmHg)]; IR (neat) 3400, 1610, 1247, 1107, 1067, 1032, 817 cm^{-1}; ^1H NMR (CCl_4) δ 3.17 (1 H, br), 3.46 (4 H, m), 3.67 (3 H, s), 4.33 (2 H, s), 6.71 (2 H, d, J = 8 Hz), 7.11 (2 H, d, J = 8 Hz); mass spectrum, m/z (M^+) calcd for $C_{10}H_{14}O_3$ 182.0943, obsd 182.0915.

2-[(4-Nitrophenyl)methoxy]ethanol (4): mp 46.5–47 °C (from hexane–Et_2O); IR ($CHCl_3$) 3600, 1605, 1520, 1350, 1110, 1055 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.58 (1 H, br), 3.55–3.90 (4 H, m), 4.63 (2 H, s), 7.48 (2 H, d, J = 8 Hz), 8.15 (2 H, d, J = 8 Hz); mass spectrum, m/z (M^+) calcd for $C_9H_{11}NO_4$ 197.0688, obsd 197.0678.

3-(Benzyloxy)-1-propanol (5): bp 88–89 °C (0.25 mmHg) [lit.[3a] bp 110 °C (0.5 mmHg)]; IR (neat) 3400, 1450, 1365, 1090, 1070, 735, 695 cm^{-1}; ^1H NMR (CCl_4) δ 1.71 (2 H, quintet, J = 6 Hz), 2.84 (1 H, s), 3.4–3.65 (4 H, m), 4.38 (2 H, s), 7.18 (5 H, s); mass spectrum, m/z (M^+) calcd for $C_{10}H_{14}O_2$ 166.0994, obsd 166.0992.

Methyl 4-methoxypentanoate (6): bp 64–65 °C (19 mmHg) [lit.[22] bp 70–71 °C (20 mmHg)]; IR (neat) 1740, 1170, 1090 cm^{-1}; ^1H NMR (CCl_4) δ 1.09 (3 H, d, J = 6 Hz), 1.6–1.8 (2 H, m), 2.29 (2 H, t, J = 8 Hz), 3.21 (3 H, s), 3.1–3.4 (1 H, m), 3.59 (3 H, s); mass spectrum, m/z (M^+) calcd for $C_7H_{14}O_3$ 146.0943, obsd 146.0988.

2-(1-Nonyloxy)ethanol (7): bp 66–67 °C (0.4 mmHg); IR (neat) 3400, 1460, 1120, 1060 cm^{-1}; ^1H NMR (CCl_4) δ 0.90 (3 H, t, J = 6 Hz), 1.48 (12 H, br s), 1.55 (2 H, br m), 2.96 (1 H, br), 3.3–3.7 (6 H, m); mass spectrum, m/z (M^+) calcd for $C_{11}H_{24}O_2$ 188.1776, obsd 188.1798.

2-(Cyclohexyloxy)ethanol (8): bp 113–114 °C (23 mmHg) [lit.[3a] bp 100 °C (14 mmHg)]; IR (neat) 3400, 1450, 1365, 1110, 1100, 1060, cm^{-1}; ^1H NMR (CCl_4) δ 1.2–2.1 (11 H, m), 3.30 (1 H, br), 3.4–3.7 (4 H, m); mass spectrum, m/z (M^+) calcd for $C_8H_{16}O_2$ 144.1150, obsd 144.1160.

4-*tert*-Butylcyclohexyl methyl ether (9): bp 84–85 °C (13.5 mmHg) [lit.[3a] bp 90 °C (14 mmHg)]; IR (neat) 1480, 1470, 1455, 1370, 1110 cm^{-1}; ^1H NMR (CCl_4) δ 0.84 (9 H, s), 0.9–2.2 (9 H, m), 2.8–3.1 (1 H, m), 3.19, 3.20 (total 3 H, each s); mass spectrum, m/z (M^+) calcd for $C_{11}H_{22}O$ 170.1671, obsd 170.1658.

Benzyl ethyl ether (10): bp 72–73 °C (16 mmHg) [lit.[20] bp 70 °C (15 mmHg)]; IR (neat) 1495, 1450, 1375, 1355, 1115, 1105, 730, 695 cm^{-1}; ^1H NMR (CCl_4) δ 1.21 (3 H, t, J = 7 Hz), 3.44 (2 H, q, J = 7 Hz), 4.39 (2 H, s), 7.20 (5 H, s); mass spectrum, m/z (M^+) calcd for $C_9H_{12}O$ 136.0888, obsd 136.0865.

***l*-Menthyl methyl ether (11)**: bp 85–87 °C (14 mmHg) [lit.[23] bp 83 °C (12 mmHg)]; IR (neat) 1460, 1445, 1365, 1110, 1100 cm^{-1}; ^1H NMR (CCl_4) δ 0.76 (3 H, d, J = 7 Hz), 0.88 (3 H, d, J = 7 Hz), 0.93 (3 H, d, J = 6 Hz), 0.9–1.7 (7 H, m), 1.9–2.3 (2 H, m), 2.83 (1 H, dt, J = 10, 4 Hz), 3.24 (3 H, s); mass spectrum, m/z (M^+) calcd for $C_{11}H_{22}O$ 170.1671, obsd 170.1655.

Ethyl *l*-menthyl ether (12): bp 72 °C (9 mmHg); IR (neat) 1465, 1455, 1375, 1115, 1085 cm^{-1}; ^1H NMR (CCl_4) δ 0.76 (3 H, d, J = 7 Hz), 0.88 (3 H, d, J = 7 Hz), 0.91 (3 H, d, J = 6 Hz), 1.16 (3 H, t, J = 7 Hz), 0.9–1.8 (7 H, m), 1.9–2.4 (2 H, m), 2.90 (1 H, dt, J = 10, 4 Hz), 3.21 (1 H, dq, J = 9, 7 Hz), 3.58 (1 H, dq, J = 9, 7 Hz); mass spectrum, m/z (M^+) calcd for $C_{12}H_{24}O$ 184.1827, obsd 184.1814.

(17) Under these conditions cyclohexene was hydroborated. So there is a possibility that the real reducing species is borane. However, the present method is much more effective than the use of borane itself. See ref 7.

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Facile Preparation of (2*R*,3*S*)- and (2*S*,3*R*)-3-[[(4-Bromobenzyl)oxy]methyl]oxirane-2-methanol via Asymmetric Epoxidation

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The availability of small homochiral building blocks is an important consideration in the synthesis of enantiomerically pure natural products. For a projected synthesis of an insect pheromone, we required a C_4 unit like the epoxy alcohol **1a**. This highly oxygenated homochiral building block may be prepared via a multistep synthesis (seven steps, 18% overall yield) from D-(−)-tartaric acid.[1,2] In principle, epoxy alcohols **1** and **2** should also be easily available via Sharpless asymmetric epoxidation[3,4] of

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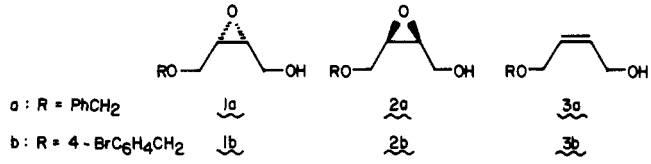
monoprotected *cis*-2-butene-1,4-diol **3**. Unfortunately, it is known[5] that asymmetric epoxidation of substrate bearing a cis electron-withdrawing substituent at C-3 is slow and does *not* proceed with complete enantioselectivity.

As Hanson and Sharpless[6] have recently disclosed a procedure for the catalytic asymmetric epoxidation of allylic alcohols in the presence of molecular sieves, which appears to be operationally extremely simple, we decided to investigate the possibility of catalytically epoxidizing an allylic alcohol **3** to produce *crystalline* epoxy alcohols **1** and **2** that could be recrystallized to optical purity.

We now report that epoxy alcohols **1b** and **2b** are stable crystalline materials that may be obtained enantiomerically pure in two simple steps in 65% overall yield from *cis*-2-butene-1,4-diol and 4-bromobenzyl bromide.



Results and Discussion

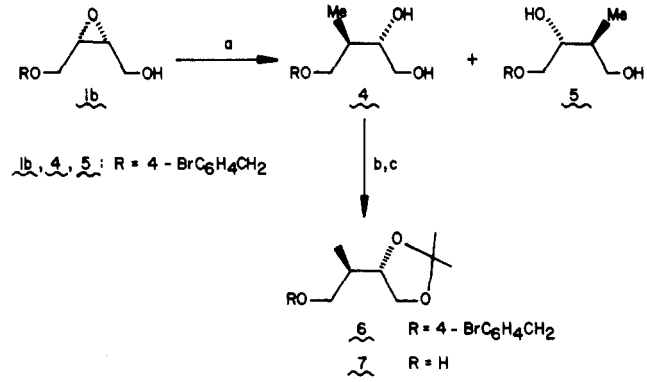
The monoprotected allylic alcohol **3b** was easily prepared by treating an excess of (inexpensive) *cis*-2-butene-1,4-diol with NaH, followed by 4-bromobenzyl bromide and catalytic n-Bu_4NI[7] in THF. Kugelrohr distillation afforded pure **3b** in 93% yield.

A variety of conditions to asymmetrically epoxidize **3b** were tried. As expected, most of the conditions tried afforded **1b** in good but not spectacular enantioselectivity. Significantly, however, the crude product was a solid that could be recrystallized to optical purity[8,9] in a single recrystallization. Thus, asymmetric epoxidation of **3b** using 10–20 mol % Ti(O-*i*-Pr)$_4$, (−)-diisopropyl tartrate (12–28 mol %), and TBHP (2–3 equiv) in the presence of 4-Å molecular sieves (20–50% w/w) consistently afforded epoxy alcohol **1b** in 84–87% ee. Reactions using 20 mol % Ti(O-*i*-Pr)$_4$ were faster than the corresponding reactions using 10 mol % Ti(O-*i*-Pr)$_4$; the former proceed to completion after 24 h at −20 to −12 °C while the latter were only 85–90% complete after the same time. The observed enantioselectivities were remarkably consistent, reflecting somewhat the dependability of the asymmetric epoxidation reaction. Furthermore, and very importantly, upon recrystallization from petroleum ether–ether the crude epoxy alcohol (consisting of a 93:7 mixture of enantiomers) afforded only a single enantiomer in 70% yield.

The epoxy alcohols **1b** and **2b** could thus be prepared in enantiomerically pure form in two simple steps (65% overall yield) from *cis*-2-butene-1,4-diol.

As the purification of **3b** involves only simple distillation and that of **1b** and **2b** only recrystallization, the described procedure should be amenable to relatively large-scale preparations. It is already known[12] that asymmetric epoxidation, especially the new catalytic procedure, works well on a large scale.

Scheme I[a]



[a] Key: (a) Me_3Al, 3 equiv, hexanes, room temperature, 2 h (4:5 = 4.8:1, combined yield 92%); (b) catalytic *p*-TsOH, $(MeO)_2CMe_2$, CH_2Cl_2, room temperature, 12 h (71% from **1b**); (c) 1 atm H_2, 10% w/w of 10% Pd/C, trace $NaHCO_3$, EtOH, room temperature, 3 h (98%).

Epoxy alcohol **1b** (and **2b**) should exhibit very similar chemistry to that of **1a** (and **2a**), which has been used as a chiral building block in a number of syntheses.[5a,13] For example, reaction of **1b** with Me_2CuLi (3 equiv, Et_2O, −20 °C) afforded the expected diols **4** and **5** (resulting from attack at C-3 and C-2, respectively) in a ratio of 1:2, while Me_3Al (2 equiv, hexanes, room temperature) gave the same diols in a ratio of 4.8:1 respectively. These results parallel those obtained previously[14,15] with **1a**.

The 4-bromobenzyl group has previously been used as an alcohol protecting group[16] that may be cleaved by hydrogenolysis. Thus, the (inseparable) mixture of diols **4** and **5** (from opening of **1b** with Me_3Al; Scheme I) was treated with 2,2-dimethoxypropane (catalytic *p*-TsOH, CH_2Cl_2) to afford the corresponding acetonides that were separated by flash chromatography. Treatment of the major acetonide **6** with H_2/Pd-C (1 atm, EtOH with $NaHCO_3$, room temperature, 3 h) then removed the 4-bromobenzyl protecting group to afford **7**.[17]

Many other manipulations of epoxy alcohols **1b** and **2b** should be possible. The convenient preparation of these homochiral building blocks described herein should facilitate their adoption as units to incorporate into enantiomerically pure natural (and unnatural) products.

Experimental Section

General Procedures. Melting points were taken on a Buchi SMP-20 melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer 983 infrared spectrophotometer. ^1H NMR (250-MHz) and ^{13}C NMR (62.9-MHz) spectra were recorded on a Bruker AM-250 spectrometer using $CDCl_3$ as solvent. Chemical shifts are reported vs. tetra-

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(8) No signals for the other diastereomer could be observed by 250-MHz ^1H NMR analysis of the derived Mosher[11] ester [derived from (+)-α-methoxy-α-(trifluoromethyl)phenylacetyl chloride] in benzene-d_6, and no change in optical rotation [$[\alpha]^{22}_D$ +17.4° (*c* 1.5, $CHCl_3$)] was observed on further recrystallization.

(9) It is interesting to note that the pure enantiomers exhibit mp 52–52.5 °C while the racemic epoxy alcohol [prepared by $VO(acac)_2$-catalyzed epoxidation[10] of **3b** or, more conveniently, by mixing together equal amounts of **1b** and **2b**] has mp 48–48.5 °C.

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methylsilane (^{1}H, δ 0.0) or $CDCl_3$ (^{13}C, δ 77.0) as internal standards. Optical rotations were measured on a Jasco DIP-360 digital polarimeter. Mass spectra were recorded on a MAT CH7 mass spectrometer. THF was distilled from sodium/benzophenone ketyl, and CH_2Cl_2 was distilled from CaH_2. Anhydrous TBHP in CH_2Cl_2 was prepared and titrated by the method of Hanson and Sharpless.[6] (*R*)-(+)-α-Methoxy-α-(trifluoromethyl)phenylacetyl chloride (MTPA-Cl) was prepared (99% yield) from (*R*)-(+)-α-methoxy-α-(trifluoromethyl)phenylacetic acid (Aldrich) by treatment with oxalyl chloride (5 equiv, catalytic DMF, reflux 1 h) followed by Kugelrohr distillation [air bath temperature 80–90 °C (9 torr)]. Other chemicals were used as received from Aldrich Chemical Co. Elemental analyses were performed by M-H-W Laboratories, Phoenix, AZ.

(*Z*)-4-[(4-Bromobenzyl)oxy]-2-buten-1-ol (3b). To a cold (0 °C) stirred suspension of NaH (4.8 g, 0.12 mol of a 60% dispersion in oil previously washed with 3 × 70 mL of dry petroleum ether) in dry THF (400 mL) under an atmosphere of argon was added *cis*-2-butene-1,4-diol (49 mL, 53 g, 0.6 mol) over 10 min. The ice bath was removed and the resulting tan solution was stirred at room temperature for 30 min. Tetrabutylammonium iodide (300 mg) and 4-bromobenzyl bromide (25.0 g, 0.100 mol) were then added, and the reaction mixture was stirred at room temperature for 15 h. Water (5 mL) was added, and the THF was removed by rotary evaporation. The residue was taken up in Et_2O (250 mL) and washed with 5 × 100 mL of water. Drying ($MgSO_4$), followed by concentration and Kugelrohr distillation [air bath temperature 120–130 °C (0.2 torr)] afforded the monoprotected diol **3b** as a light yellow oil [23.8 g (93%)]. It was homogeneous by TLC (petroleum ether:ether 1:1): IR (film) 3380 (br), 3020, 2860, 1590, 1485, 1070, 1010, 840, 800 cm^{-1}; ^{1}H NMR δ 4.04 (d, 2 H, J = 5.8 Hz), 4.09 (d, 2 H, J = 5.8 Hz), 4.42 (s, 2 H), 5.71 (AB of ABX_2Y_2 system, 2 H, Δδ 0.08, J_{AB} = 11.3, J_{AX} = J_{VY} = 5.8 Hz), 7.18, 7.44 (AA′BB′ system, 4 H, J_{AB} = 8.3 Hz); ^{13}C NMR (δ 57.8, 65.4, 71.1, 121.2, 127.1, 129.0, 131.1, 132.2, 136.6; MS, m/z 258 (1.0, M$^+$, ^{81}Br), 256 (2.0, M$^+$, ^{79}Br), 188 (45), 187 (100), 186 (94), 185 (100), 184 (57), 183 (100), 172 (81), 171 (100), 170 (78), 169 (100), 159 (26), 157 (33), 107 (71).

The stillpot residue (1.0 g) was chromatographed on 35 g of silica with petroleum ether–ether (20:1) as eluant to furnish the corresponding diprotected diol as a thick oil (0.5 g): IR (film) 3020, 2860, 1590, 1485, 1090, 1070, 1010, 840, 800 cm^{-1}; ^{1}H NMR δ 4.03 (m, 4 H), 4.42 (s, 4 H), 5.75–5.79 (sym m, 2 H), 7.18, 7.45 (AA′BB′ system, J_{AB} = 8.5 Hz). ^{13}C NMR δ 65.7, 71.2, 121.3, 129.1, 129.2, 131.3, 137.0; MS, m/z 428 (0.3, M$^+$, ^{81}Br – ^{81}Br), 426 (0.7, M$^+$, ^{79}Br – ^{81}Br), 424 (0.3, M$^+$, ^{79}Br – 79 Br), 257 (18), 255 (18), 247 (16), 245 (19), 240 (14), 238 (14), 189 (24), 188 (100), 187 (100), 186 (100), 185 (100), 183 (54), 172 (78), 171 (100), 170 (79), 169 (100), 107 (60).

(2*R*,3*S*)-3-[[(4-Bromobenzyl)oxy]methyl]oxirane-2-methanol (1b). To a cold (–20 °C) stirred suspension of powdered 4-Å molecular sieves (6.2 g) in dry CH_2Cl_2 (400 mL) under an atmosphere of argon was added titanium isopropoxide (4.54 g, 16 mmol), D-(–)-diisopropyl tartrate (5.12 g, 22 mmol) and *tert*-butyl hydroperoxide (36 mL of a 4.4 M solution in CH_2Cl_2, 160 mmol). The slurry was stirred at –20 °C for 30 min. Allylic alcohol **3b** (20.7 g, 80 mmol) was then added as a solution in CH_2Cl_2 (30 mL), and the reaction mixture was stirred at –20 °C for 2 h and then stored in a –20 °C freezer overnight (actual temperature –20 to –12 °C). After 24 h, TLC (petroleum ether:ether 1:2) indicated no remaining allylic alcohol and a single lower R_f product. The reaction was vigorously stirred and quenched with water (90 mL). The mixture was allowed to warm to room temperature and stirred for 60 min. A solution of 30% NaOH in brine (20 mL) was then added. After 30 min of vigorous stirring, the phases were separated and the aqueous layer was extracted with CH_2Cl_2 (2 × 40 mL). The combined organic layer was dried ($MgSO_4$) and filtered through a pad of Celite. Concentration afforded a TBHP-containing colorless oil; removal of excess TBHP in vacuo (0.1 torr) gave a white solid that still contained traces of TBHP.

Purification of a small amount (200 mg) of this material by flash chromatography (petroleum ether:ether = 1:2) gave chemically pure epoxy alcohol, $[\alpha]^{22}_D$ + 14.7° (*c* 1.5, $CHCl_3$, 85% ee). Acetylation of some (20 mg) of this alcohol (excess Ac_2O, pyridine, catalytic DMAP) gave the corresponding acetate. Analysis using the chiral shift reagent Eu(hfc)$_3$ (250-MHz ^{1}H NMR in benzene-d_6, hfc = 3-[(heptafluoropropyl)hydroxymethylene]-*d*-camphorate) indicated an optical purity of 86%.

The bulk solid was suspended in petroleum ether (100 mL), and sufficient ether was added to form a solution at ambient temperature. The solution was slowly cooled to –20 °C (freezer) and left for a few hours. The white needles were collected by suction filtration, washed with petroleum ether (2 × 10 mL), and dried in vacuo to afford epoxy alcohol **1b** [15.3 g (70%)]. This material was homogeneous by TLC: mp 52–52.5 °C; IR ($CHCl_3$) 3600, 3430, 3000, 2860, 1600, 1490, 1090 cm^{-1}; ^{1}H NMR ($CDCl_3$, D_2O) δ 3.22 (1 H, dt, H_2, J = 4.4, 5.3 Hz), 3.29 (1 H, dt, H_3, J = 4.4, 5.3 Hz), 3.68 (d, 2 H, H_4, J = 5.3 Hz), 3.73 (d, 2 H, H_1, J = 5.3 Hz), 4.52 (2 H, AB q, Δδ 0.10, J_{AB} = 12.0 Hz), 7.21, 7.48 (AA′BB′ system, 4 H, J_{AB} = 8.3 Hz); ^{13}C NMR δ 54.8, 55.7, 60.5, 68.1, 72.5, 121.7, 129.3, 131.5, 136.4; MS, m/z 274 (5.8, M$^+$, ^{81}Br), 272 (5.2, M$^+$, ^{79}Br), 213 (21), 211 (22), 188 (73), 187 (100), 186 (100), 185 (100), 184 (90), 183 (84), 172 (59), 171 (100), 170 (63), 169 (100), 159 (34), 157 (46), 132 (14), 119 (15), 107 (53), 106 (25); $[\alpha]^{22}_D$ +17.4° (*c* 1.5, $CHCl_3$). Anal. Calcd for $C_{11}H_{13}BrO_3$: C, 48.37; H, 4.80; Br, 29.25. Found: C, 48.35; H, 5.00; Br, 29.41.

Analysis (250 MHz, ^{1}H NMR, C_6D_6) of the Mosher ester (excess (+)-MTPA-Cl, Et_3N, catalytic DMAP in CH_2Cl_2) from the crystalline epoxy alcohol indicated the presence of only one diastereomer. Peaks due to the other diastereomer (obtained from **2b**) were not detected; in particular, the signals due to the epoxide protons (δ 2.79 for **1b**-OMTPA and δ 2.75 for **2b**-OMTPA) for each diastereomer were quite distinctive.

Further recrystallization did not affect the specific rotation of the epoxy alcohol.

(2*S*,3*R*)-3-[[(4-Bromobenzyl)oxy]methyl]oxirane-2-methanol (2b). Essentially the same procedure as for **1b** was followed with the obvious exception that L-(+)-diisopropyl tartrate was used in place of (–)-DIPT. The following materials were used: 4-Å molecular sieves (2 g), CH_2Cl_2 (150 mL), titanium isopropoxide (1.7 g, 6 mmol), (+)-DIPT (1.9 g, 8.2 mmol), TBHP (4.4 M in CH_2Cl_2, 13.7 mL, 60 mmol), and allylic alcohol **3b** (7.77 g, 30 mmol). Reaction at –20 °C for 24 h subsequent workup afforded a white solid (crude ee 86%). Recrystallization from petroleum ether–ether gave epoxy alcohol **2b** as white needles: (6.02 g (73%); $[\alpha]^{22}_D$–17.3° (*c* 1.5, $CHCl_3$); spectral data identical with those for **1b**.

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A Convenient Synthesis of Sulfinate Esters from Sulfonyl Chlorides

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The reaction of an organometallic reagent with a diastereomerically pure sulfinate ester of menthol[1] continues to be the method most often employed for the preparation of optically active sulfoxides, despite recent advances in the asymmetric oxidation of sulfides.[2] These sulfoxides have proven to be valuable intermediates for asymmetric synthesis.[3] The requisite sulfinate esters are generally

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