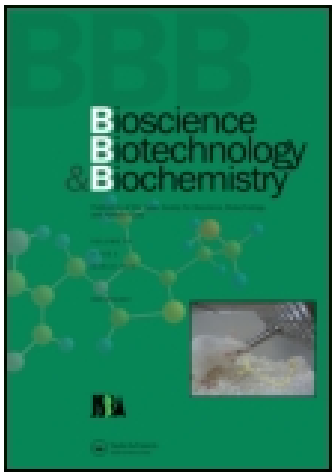

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Enantio- and Stereoselective Syntheses of Monodeuterium-labeled Glycerols

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Note

Enantio- and Stereoselective Syntheses of Monodeuterium-labeled Glycerols

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An efficient method is described for synthesizing and four possible diastereomers of stereochemically defined monodeuterated glycerols by utilizing Sharpless asymmetric dihydroxylation.

Key words: chirally monodeuterated glycerols; Sharpless asymmetric dihydroxylation

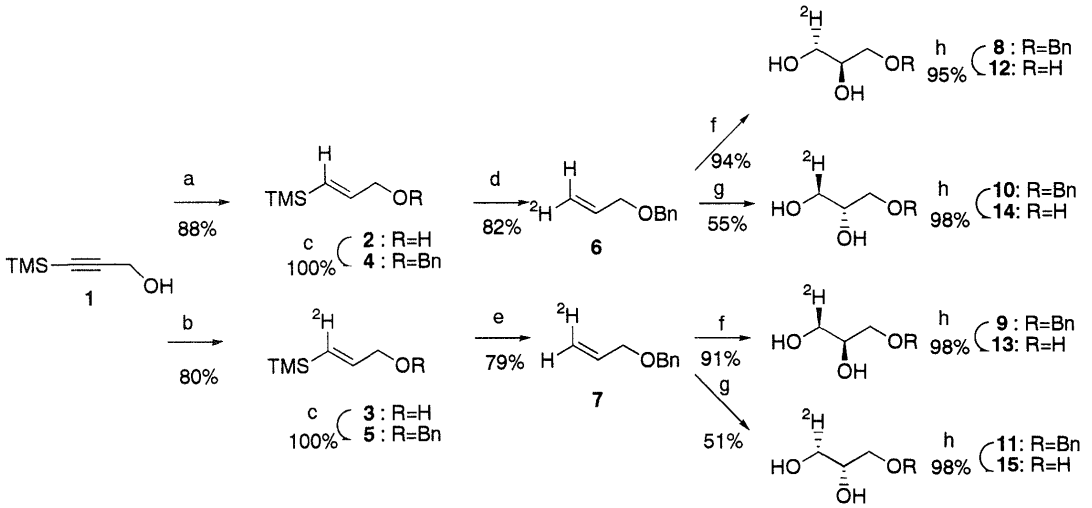
Studies on biosynthetic pathways and the cryptic stereochemistry involved in enzyme reactions have shown stereochemically defined labeled compounds to be useful reagents. Several syntheses of such compounds as chirally deuterium-labeled glycerols, including Kakinuma's versatile method, have been previously reported.[1] To determine the building unit of macrophomic acid,[2] we have developed an efficient and concise method for synthesizing chirally monodeuterated glycerols instead of using a chiral auxiliary. We chose a method for directly introducing the required stereochemistry to simplify the synthetic pathway. In this paper, we describe the syntheses of all four chirally deuterated diastereomers (*sn*-(3*R*)-[3-^{2}H]-, *sn*-(3*S*)-[3-^{2}H]-, *sn*-(1*S*)-[1-^{2}H]- and *sn*-(1*R*)-[1-^{2}H]-glycerols) by utilizing Sharpless asymmetric dihydroxylation.[3]

In our first attempts to introduce the ^{2}H atom, attempted reduction of [3-^{2}H]propargyl alcohol with $LiAlH_4$, DIBAL, or Red-Al met such problems as randomization of the label and a serious loss of the desired product during purification due to its volatile nature. Reduction of benzyl propargyl ether with $LiAlH_4$ also caused undesired cleavage at the propargylic position to give benzyl alcohol. Thus, we planned to prepare the stereochemically defined monodeuterated allyl alcohol from 3-trimethylsilylpropargyl alcohol **1**. (*E*)-3-Trimethylsilyl-2-propen-1-ol (**2**), which is now commercially available, was prepared according to the literature method[4] (Scheme). After benzylation, desilylation of resultant benzyl ether **4** was then examined. Treatment with ^{2}HCl or I_2 in benzene–^2H_2O resulted in decomposition or recovery of the starting material. The use of [O^2H]-*p*-toluenesulfinic acid[5] smoothly effected the desilylation, but caused randomization of the label (*E*:*Z*, 2.3:1, 70% deuteration). The problem was finally solved by the use of [O^2H]acetic acid[6] under reflux for 48 h; **4** was successfully converted into (2*E*)-[3-^{2}H]-1-benzyloxy-2-propene (**6**; *E*:*Z*, 100:8).

(2*Z*)-[3-^{2}H]-1-Benzyloxy-2-propene (**7**) was synthesized under essentially the same protocol. Reduction of **1** and subsequent quenching with 2 M ^{2}HCl afforded **3**. After benzylation, resultant **5** was cleanly converted into (2*Z*)-[3-^{2}H]-1-benzyloxy-2-propene (**7**) by treating with acetic acid in a 79% yield (*E*:*Z*, 7:100).

Asymmetric dihydroxylation of **6** and **7** with AD-mix-β furnished 3-benzyloxy-1,2-propanediols **8** and **9** in good yields. Based on the ^{1}H-NMR analysis of the corresponding bis-*O*-(*R*)-α-methoxy-α-trifluoromethylphenylacetate (MTPA),*1 the enantiomeric purities of **8***2 and **9***3 were determined as 56 and 58% *e.e.*, respectively. Similarly, dihydroxylation of **6** and **7** with AD-mix-α furnished 3-benzyloxy-1,2-propanediols **10** and **11**. The enantiomeric purities of **10***2 and **11***3 were determined as 60 and 50% *e.e.*, respectively. Finally, standard hydrogenolysis of [^{2}H]-3-benzyloxy-1,2-propanediols **8**, **9**, **10**, and **11** afforded corresponding [^{2}H]glycerols **12**, **13**, **14**, and **15** in 95–98% yields.

This efficient 5-step method enabled us to synthesize all four diastereomers of chirally monodeuterated glycerols. As far as a study is concerned with diastereotopic methylene protons of glyceric acid, the low optical purity of the monodeuterated glycerols is not a problem since the label of *sn*-[1-^{2}H]-glycerol is



Scheme a: Red-Al, ether, 0°C, 1 h, then 2 M-HCl. b: Red-Al, ether, 0°C, 1 h, then 2 M-^{2}HCl. c: BnBr, *n*-Bu_4NI, THF, r.t., 18 h. d: CH_3COO^2H, reflux, 48 h. e: CH_3COOH, reflux, 48 h. f: AD-mix-β, *t*-BuOH, water, 0°C, 12 h. g: AD-mix-α, *t*-BuOH, water, 0°C, 12 h. h: 10% Pd–C, H_2, EtOH, 4 h.

*1 The optical purity of each MTPA ester was evaluated from the integration ratio between the methoxy signals: 3.42 ppm for 2*R*-isomers and 3.47 ppm for 2*S*-isomers, respectively.

*2 Each enantiomer contained 7% of the diastereomers derived from the corresponding *Z*-isomer.

*3 Each enantiomer contained 7% of the diastereomers derived from the corresponding *E*-isomer.

lost during glycolysis. Although our method has a disadvantage in the enantiomeric purity of the products, this could be overcome by the changing ligand of AD or by replacing a protective group such as phenyl or *p*-methoxyphenyl. Their allyl ethers have been reported to give superior *e.e.* in AD.[7]

We are grateful to Mr. K. Watanabe and Mrs. E. Fukushi in our deparatment for their measurements of mass spectra.

Experimental

IR spectra were taken with a HITACHI 285 spectrometer, and NMR spectra were recorded with JEOL GX 270 and Bruker AM500 FT-NMR spectrometers. Mass spectra were run on JEOL JMS-DX 300 and JMS-SX102A mass spectrometers. Tetrahydrofuran (THF) and diethyl ether were distilled from sodium-benzophenone under an argon atmosphere.

(2E)-[3-^2H]-3-Trimethylsilyl-2-propen-1-ol (**3**). To a solution of sodium bis(2-methoxyethoxy)aluminum hydride (a 3.4 M solution in toluene, 7.4 ml) in diethyl ether (10 ml) cooled with an ice bath to 3°C under argon, **1** (2.0 g, 16 mmol) in diethyl ether (5 ml) was cautiously added dropwise. After 10 min, the ice bath was removed and the reaction completed within 1 h. The mixture was cooled to 0°C and then quenched by the addition of deuterium chloride (99.5 atom %, Aldrich) in deuterium oxide (2.0 M, 20 ml). The organic extract was separated, and the aqueous phase was extracted with additional ether (40 ml). The combined extracts were dried over sodium sulfate and concentrated *in vacuo*. The residue was purified by short-path distillation to afford 1.63 g (80% yield, 99% deuteration) of **3** as a clear, colorless liquid, bp 74°C (20 mm). IR ν_{max} (neat) cm^{-1}: 3322 (br.), 2956, 2897, 2859, 1614, 1417, 1248, 1091, 1037, 838, 767, 692. NMR δ_H (270 MHz, $CDCl_3$): 0.08 (9H, s, $Si(C\underline{H}_3)_3$), 4.17 (2H, br. d, $J=3.9$ Hz, C_1–$\underline{H}$), 6.17 (1H, m, C_2–$\underline{H}$). EIMS *m/z*: 131 (M^+), 116, 114. EI-HRMS *m/z*: 131.0885 (M^+, 131.0876; calcd. for $C_6H_{13}{}^2HOSi$).

(2E)-3-Trimethylsilyl-1-benzyloxy-2-propene (**4**). To a solution of 70% sodium hydride in oil (1.25 g, 31 mmol) in tetrahydrofuran (40 ml) at 0°C under argon, **2** (2.03 g, 15.6 mmol) in tetrahydrofuran (10 ml) was cautiously added dropwise. After 15 min, benzyl bromide (3.86 ml, 31 mmol) and tetrabutylammonium iodide (576 mg, 1.56 mmol) were added. The suspension was warmed to room temperature and stirred overnight. The resulting suspension was cooled to 0°C, and the reaction was quenched by adding methanol (5 ml) and then water (60 ml). The organic extract was separated, and the aqueous phase was extracted with ether (60 ml). The combined extracts were dried over sodium sulfate and concentrated *in vacuo*. Column chromatography of the residue on silica gel (8:2, hexane/ethyl acetate) gave **4** (3.4 g, quantitative yield). IR ν_{max} (neat) cm^{-1}: 3065, 3031, 2955, 2926, 2854, 1622, 1455, 1248, 1102, 864, 838, 696. NMR δ_H (270 MHz, $CDCl_3$): 0.07 (9H, s, $Si(C\underline{H}_3)_3$), 4.05 (2H, dd, $J=5.6$, 1.3 Hz, C_1-$\underline{H}$), 4.51 (2H, s, Ar-$C\underline{H}_2$), 5.94 (1H, dt, $J=18$, 1.3 Hz, C_3–$\underline{H}$), 6.13 (1H, dt, $J=18$, 5.6 Hz, C_2–$\underline{H}$), 7.34 (5H, m, Ar–$\underline{H}$). EIMS *m/z*: 220 (M^+).

(2E)-[3-^2H]-3-Trimethylsilyl-1-benzyloxy-2-propene (**5**). By the same procedure as that described for the preparation of **4**, 2.5 g (19.5 mmol) of **3** was converted to 4.3 g of **5** (quantitative yield). IR ν_{max} (neat) cm^{-1}: 3065, 3030, 2954, 2924, 2854, 1614, 1455, 1247, 1119, 1090, 838, 696. NMR δ_H (270 MHz, $CDCl_3$): 0.07 (9H, s, $Si(CH_3)_3$), 4.05 (2H, d, $J=5.3$ Hz, C_1–$\underline{H}$), 4.53 (2H, s, Ar-$C\underline{H}_2$), 6.11 (1H, m, C_2–$\underline{H}$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 221 (M^+). EI-HRMS *m/z*: 221.1370 (M^+, 221.1345; calcd for $C_{13}H_{19}{}^2HOSi$).

(2E)-[3-^2H]-1-Benzyloxy-2-propene (**6**). A solution of **4** (20 mg, 0.09 mmol) in acetic acid deuterium (0.3 ml, 98 atom%, Aldrich) and deuterium oxide (0.01 ml) was heated to reflux for 48 h. The reaction was quenched by 1 M sodium hydroxide, and the solution extracted with ether. The combined organic layers were successively washed with aqueous sodium hydrogen carbonate and brine, dried over sodium sulfate and concentrated *in vacuo*. Purification of the residue by PTLC (8:2, hexane/ethyl acetate) gave **6** (11 mg, 82% yield, 98% deuteration, *E*:*Z*; 100:8). IR ν_{max} (neat) cm^{-1}: 3064, 3031, 2854, 1495, 1454, 1360, 1121, 1090, 978, 736. NMR δ_H (270 MHz, $CDCl_3$): 4.03 (2H, dd, $J=5.9$, 1.3 Hz, C_1–$\underline{H}$), 4.53 (2H, s, Ar-$C\underline{H}_2$), 5.33 (1H, dt, $J=16$, 1.3 Hz, C_3–$\underline{H}$), 5.93 (1H, m, C_2–$\underline{H}$), 7.34 (5H, m, Ar-H). EIMS *m/z*: 149 (M^+), 119, 105, 91. EI-HRMS *m/z*: 149.0962 (M^+, 149.0950; calcd. for $C_{10}H_{11}{}^2HO$).

(2Z)-[3-^2H]-1-Benzyloxy-2-propene (**7**). By the same procedure as that described for the preparation of **6**, treatment of **5** (0.5 g, 2.28 mmol) with acetic acid gave **7** (0.268 g, 79% yield, 99% deuteration, *E*:*Z*; 7:100). IR ν_{max} (neat) cm^{-1}: 3088, 3062, 3030, 2854, 1454, 1363, 1248, 1117, 1076, 811, 736. NMR δ_H (270 MHz, $CDCl_3$): 4.05 (2H, dd, $J=6.9$, 1.3 Hz, C_1-$\underline{H}$), 4.53 (2H, s, Ar-$C\underline{H}_2$), 5.21 (1H, dt, $J=10$, 1.3 Hz, C_3–$\underline{H}$), 5.95 (1H, m, C_2–$\underline{H}$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 149 (M^+), 119, 105, 91. EI-HRMS *m/z*: 149.0983 (M^+, 149.0950; calcd for $C_{10}H_{11}{}^2HO$).

General procedure for Sharpless asymmetric dihydroxylation. To a solution of 1.876 g of AD-mix-β for (2*R*)-3-benzyloxy-1,2-propanediols or AD-mix-α for (2*S*)-3-benzyloxy-1,2-propanediols in *tert*-butyl alcohol (5 ml) and water (5 ml), 1-benzyloxy-2-propene (200 mg, 1.34 mmol) was added, and the mixture was stirred vigorously at 0°C for 12 h. The reaction was quenched by solid sodium sulfite, before the mixture was extracted with methylene chloride. The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated *in vacuo*. Purification of the residue by silica gel column chromatography (6:4, hexane/ethyl acetate) gave 3-benzyloxy-1,2-propanediol.

(1R,2R)-[1-^2H]-3-Benzyloxy-1,2-propanediol (**8**). **6** (200 mg, 1.34 mmol) was converted to **8** (230 mg, 94%). IR ν_{max} (neat) cm^{-1}: 3391 (br.), 2915, 2867, 1651, 1454, 1366, 1208, 1094, 739, 698. NMR δ_H (270 MHz, $CDCl_3$): 2.15 (1H, br. s, O$\underline{H}$), 2.66 (1H, br. s, OH), 3.58 (3H, m, C_1–H, C_3–$\underline{H}_2$), 3.89 (1H, m, C_2–$\underline{H}$), 4.55 (2H, s, Ar-$C\underline{H}_2$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 183 (M^+), 107, 91. EI-HRMS *m/z* 183.1007 (M^+, 183.1005; calcd. for $C_{10}H_{13}{}^2HO_3$).

(1S,2R)-[1-^2H]-3-Benzyloxy-1,2-propanediol (**9**). **7** (120 mg, 0.81 mmol) was converted to **9** (135 mg, 91%). IR ν_{max} (neat) cm^{-1}: 3752 (br.), 3392, 2918, 1455, 1364, 1313, 1095, 738, 698. NMR δ_H (270 MHz, $CDCl_3$): 2.15 (1H, br. s, O$\underline{H}$), 2.66 (1H, br. s, O$\underline{H}$), 3.58 (3H, m, C_1–$\underline{H}$, C_3–$\underline{H}_2$), 3.89 (1H, m, C_2–$\underline{H}$) 4.55 (2H, s, Ar-$C\underline{H}_2$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 183 (M^+), 107, 91. EI-HRMS *m/z*: 183.1007 (M^+, 183.1005; calcd. for $C_{10}H_{13}{}^2HO_3$).

(1S,2S)-[1-^2H]-3-Benzyloxy-1,2-propanediol (**10**). **6** (710 mg, 4.7 mmol) was converted to **10** (480 mg, 55%). IR ν_{max} (next) cm^{-1}: 3391 (br.), 3031, 2915, 2867, 1651, 1494, 1454, 1366, 1208, 1094, 739, 698. NMR δ_H (270 MHz, $CDCl_3$): 2.15 (1H, br. s, OH), 2.66 (1H, br. s, OH), 3.58 (3H, m, C_1–$\underline{H}$, C_3–$\underline{H}_2$), 3.88 (1H, m, C_2–$\underline{H}$), 4.55 (2H, s, Ar-$C\underline{H}_2$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 183 (M^+), 107, 91. EI-HRMS *m/z*: 183.0991 (M^+, 183.1005; calcd. for $C_{10}H_{13}{}^2HO_3$).

(1R,2S)-[1-^2H]-3-Benzyloxy-1,2-propanediol (**11**). **7** (147 mg, 0.99 mmol) was converted to **11** (93 mg, 51%). IR ν_{max} (neat) cm^{-1}: 3391 (br.), 2867, 1652, 1455, 1366, 1313, 1094, 739, 698. NMR δ_H (270 MHz, $CDCl_3$): 2.14 (1H, br. s, O$\underline{H}$), 2.64 (1H, br. s, O$\underline{H}$), 3.58 (3H, m, C_1–$\underline{H}$, C_3–$\underline{H}_2$), 3.90 (1H, m, C_2–$\underline{H}$), 4.55 (2H, s, Ar-$C\underline{H}_2$), 7.34 (5H, m, Ar-$\underline{H}$). EIMS *m/z*: 183 (M^+), 107, 91. EI-HRMS *m/z*: 183.0992 (M^+, 183.1005; calcd. for $C_{10}H_{13}{}^2HO_3$).

sn-(3R)-[3-^2H]-Glycerol (**12**). To a solution of **8** (100 mg, 0.55 mmol) in ethanol (1 ml), 10% palladium on charcoal (20 mg) was added, and the mixture was stirred vigorously under a hydrogen atmosphere at room temperature for 4 h. The suspension was filtered, and the filtrate was concentrated *in vacuo* to give **12** (49 mg, 95% yield). NMR δ_H (270 MHz, CD_3OD): 3.55 (3H, m, C_1–$\underline{H}_2$, C_3–$\underline{H}$), 3.62 (1H, dt, $J=10$, 5.9 Hz, C_2–$\underline{H}$). FIMS *m/z*: 94 (MH^+), 93 (M^+), 75. FI-HRMS *m/z*: 93.0557 (M^+, 93.0535; calcd for $C_3H_7O_3{}^2H$).

sn-(3S)-[3-^2H]-Glycerol (**13**). By the same procedure as that described for the preparation of **12**, 100 mg (0.55 mmol) of **9** was converted to 50 mg of **13** (98%). NMR δ_H (270 MHz, CD_3OD): 3.55 (3H, m, C_1–$\underline{H}_2$, C_3–$\underline{H}$), 3.62 (1H, dt, $J=10$, 5.6 Hz, C_2–$\underline{H}$). FIMS *m/z*: 94 (MH^+), 93 (M^+), 75. FI-HRMS *m/z*: 93.0533 (M^+, 93.0535; calcd. for $C_3H_7O_3{}^2H$).

sn-(1S)-[1-^2H]-Glycerol (**14**). By the same procedure as that described for the preparation of **12**, 100 mg (0.55 mmol) of **10** was converted to 45 mg of **14** (87%). NMR δ_H (270 MHz, CD_3OD): 3.55 (3H, m, C_1–$\underline{H}$, C_2–$\underline{H}_2$), 3.62 (1H, dt, $J=10$, 5.9 Hz, C_2–$\underline{H}$). FIMS *m/z*: 94 (MH^+), 93 (M^+). FI-HRMS *m/z*: 93.0536 (M^+, 93.0535; calcd. for $C_3H_7O_3{}^2H$).

sn-(1R)-[1-^2H]-Glycerol (**15**). By the same procedure as that described for the preparation of **12**, 100 mg (0.55 mmol) of **11** was converted to

43 mg of **15** (84%). NMR δ_H (270 MHz, CD_3OD): 3.55 (3H, m, C_1–H, C_3–H_2), 3.62 (1H, dt, J=10, 5.6 Hz, C_2–H). FIMS *m/z*: 94 (MH^+), 93 (M^+). FI-HRMS *m/z*: 93.0552 (M^+, 93.0535; calcd. for $C_3H_7O_3{}^2H$).

Typical procedure for preparating the bis-O-(R)-α-methoxy-α-trifluoromethylphenylacetate of monobenzylglycerols. To a solution of monobenzylglycerol (1 mg, 5.5 mmol) in methylene chloride (100 ml), 1,3-dicyclohexylcarbodiimide (5.6 mg, 27.3 mmol), (*R*)-α-methoxy-α-trifluoromethylphenylacetic acid (6.6 mg, 27.3 mmol) and 4-dimethylaminopyridine (1.4 mg, 11 mmol) were added, and the mixture was stirred at room temperature for 18 h. Purification of the residue by preparative thin-layer chromatography (7 : 3, hexane/ethyl acetate) gave an MTPA ester (3.3 mg, 97% yield). MTPA ester of **8**: NMR δ_H (500 MHz, C_6D_6): 3.21 (2H, d, J=5.2 Hz, C_3–H_2), 3.39 (3H, s, OCH_3), 3.42 (3H, s, OCH_3), 4.10 (1H, d, J=11 Hz, Ar-CH), 4.11 (1H, d, J=11 Hz, Ar-CH), 4.13 (1H, d, J=5.1 Hz, C_1–H), 5.41 (1H, m, C_2–H), 7.10–7.30 (11H, m, Ar-H), 7.71 (4H, m, Ar-H). EI-HRMS *m/z*: 615.1813 (M^+, 615.1800; calcd. for $C_{30}H_{27}{}^2HF_6O_7$). MTPA ester of **9**: NMR δ_H (500 MHz, C_6D_6): 3.21 (2H, d, J=5.1 Hz, C_3–H_2), 3.39 (3H, s, OCH_3), 3.42 (3H, s, OCH_3), 4.10 (1H, d, J=11 Hz, Ar-CH), 4.11 (1H, d, J=11 Hz, Ar-CH), 4.43 (1H, d, J=2.6 Hz, C_1–H), 5.41 (1H, m, C_2–H), 7.10–7.30 (11H, m, Ar-H), 7.71 (4H, m, Ar-H). EI-HRMS *m/z*: 615.1780 (M^+, 615.1800; calcd. dor $C_{30}H_{27}{}^2HF_6O_7$). MTPA ester of **10**: NMR δ_H (500 MHz, C_6D_6): 3.26 (1H, dd, J=10, 4.1 Hz, C_3–H), 3.36 (1H, dd, J=10, 4.6 Hz, C_3–H), 3.37 (3H, s, OCH_3), 3.47 (3H, s, OCH_3), 3.97 (1H, d, J=5.1 Hz, C_1–H), 4.14 (1H, d, J=11 Hz, Ar-CH), 4.18 (1H, d, J=11 Hz, Ar-CH), 5.42 (1H, m, C_2–H), 7.10–7.30 (11H, m, Ar-H), 7.75 (4H, m, Ar-H). EI-HRMS *m/z*: 615.1763 (M^+, 615.1800; calcd. for $C_{30}H_{27}{}^2HF_6O_7$). MTPA ester of **11**: NMR δ_H (500 MHz, C_6D_6): 3.23 (1H, dd, J=10, 4.1 Hz, C_3–H), 3.35 (1H, dd, J=10, 4.6 Hz, C_3–H), 3.37 (3H, s, OCH_3), 3.47 (3H, s, OCH_3), 4.14 (1H, d, J=11 Hz, Ar-CH), 4.18 (1H, d, J=11 Hz, Ar-CH), 4.40 (1H, d, J=2.0 Hz, C_1–H), 5.42 (1H, m, C_2–H), 7.10–7.30 (11H, m, Ar-H). 7.75 (4H, m, Ar-H). EI-HRMS *m/z*: 615.1810 (M^+, 615.1800; calcd. for $C_{30}H_{27}{}^2HF_6O_7$).

References

1) K. Kakinuma, Y. Iihama, I. Takagi, K. Ozawa, N. Yamauchi, N. Imamura, Y. Esumi, and M. Uramoto, *Tetrahedron*, **48**, 3763–3774 (1992) and references cited therein.
2) H. Oikawa, K. Yagi, K. Watanabe, M. Honma, and A. Ichihara, *J. Chem. Soc., Chem. Commun.*, **1997**, 97–98.
3) K. B. Sharpless, W. Amberg, Y. L. Bennani, G. A. Crispino, J. Hartumg, K.-S. Joeng, H.-L. Kwong, K. Morikawa, Z.-M. Wang, D. Xu, and X.-L. Zhang, *J. Org. Chem.*, **57**, 2768–2771 (1992).
4) T. K. Jones and S. E. Denmark, *Org. Synth.*, **64**, 182–189 (1985).
5) G. Büchi and H. Wüest, *Tetrahedron Lett.*, **49**, 4305–4306 (1977).
6) P. F. Hudrlik, R. H. Schwartz, and J. C. Hogan, *J. Org. Chem.*, **44**, 155–157 (1979).
7) Z.-M. Wang, X.-L. Zhang, and K. B. Sharpless, *Tetrahedron Lett.*, **34**, 2267–2270 (1993).