Concise Synthesis of Enantiomerically Pure *cis*- and *trans*-3-(Diphenylphosphino)-4-hydroxytetrahydrofurans

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3-(Mesyloxy)-4-hydroxytetrahydrofurans were prepared from L-ascorbic acid and D-isoascorbic acid, respectively. These widely applicable chiral building blocks were transformed into enantiomerically pure *cis*- and *trans*-3-(diphenylphosphino)-4-hydroxytetrahydrofurans.

Optically active chelating bisphosphines are among the most widely used ligands in transition-metal-catalyzed asymmetric organic reactions.[1] Although monophosphine ligands are reputated to be less useful in transition-metal catalysis, in a few cases they have been shown to be similiarly active or superior.[2] In some of these reactions, the presence of a hydroxy group may have a pronounced effect in terms of catalytic activity and enantioselectivity.[3] In conjunction with our particular interest in establishing remote hydroxy groups at defined distances in the phosphine ligand, able to interact with a second metal or the substrate itself,[4] we investigated routes for the preparation of chiral β-hydroxy phosphines. Perusal of the literature revealed that only a limited number of methods exist for the enantioselective design of such hydroxy phosphines.[5] Most synthetic approaches proceed by way of the addition of phosphides to chiral oxiranes to give *trans*-hydroxy phosphines.[6] This method requires the availability of chiral oxiranes. In some cases, racemic hydroxy phosphines may be resolved enzymatically.[7] Another method involves the conversion to phosphines of selected hydroxy groups starting from chiral polyhydroxy compounds.[8] However, this last strategy has been hampered by the lack of suitable protecting groups for those hydroxy groups which are not involved in the functional group interconversion. Most of the known protecting groups[9] suffer serious drawbacks, due either to their being attacked by phosphide ions or to their problematic deprotection in the presence of incorporated phosphine groups.

We describe here a facile route to enantiomerically pure *cis*- and *trans*-4-hydroxy-3-phosphinotetrahydrofurans, applying our chiral pool approach, which employs L-ascorbic acid and D-isoascorbic acid, respectively, as starting materials for the construction of chiral tetrahydrofuran derivatives.[10,11] The described monophosphines may serve either as ligands for transition-metal-catalyzed reactions, or as synthons for the preparation of other functionalized phosphines or diphosphines.[12]

Scheme I illustrates our synthetic route, starting from L-ascorbic acid (**1a**) and D-isoascorbic acid (**1b**). They have been converted by known methods to isopropylidenethreitol (**2a**) and -erythritol (**2b**), respectively.[13] Esterification of both hydroxy groups with methanesulfonyl chloride yields the bis(sulfonates) **3a**/**3b**. Removal of the acetal, by treatment with hot aqueous methanolic HCl, led to *in situ* cyclization to give tetrahydrofurans **4a** and **4b**. The evident facility and selectivity of the acid-catalyzed ring closure is surprising, since the secondary mesyl group is not affected as has been reported with reactions requiring basic conditions.[14] In principle, these

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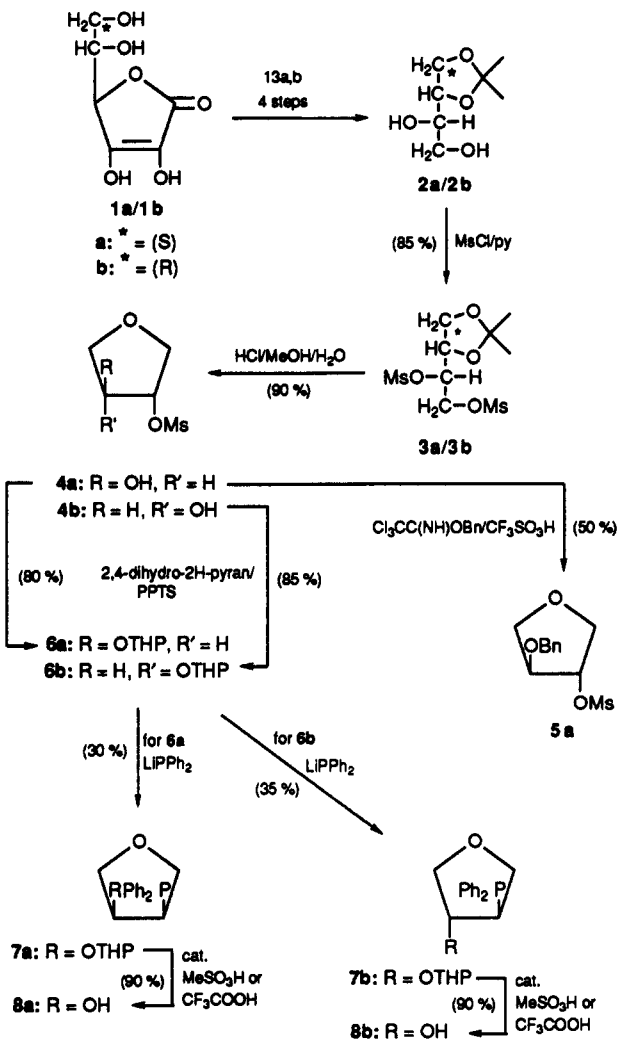
0022-3263/93/1958-6814$04.00/0

Scheme I



tetrahydrofurans should also be accessible from diastereomers of tartaric acid.[15] However, our more generalized approach exhibits advantages. The dissymmetrization of *meso*-alcohols represented by derivatives **2b** and **4b** has been achieved without the help of enzymes, etc.[16] The formation of dimesylated tetrahydrofurans as byproducts was simultaneously avoided.

The protection of the hydroxy group is the key step in the following synthesis of the hydroxy phosphines.[17] In our first approach the benzylation of **4a** under acidic conditions,[18] which was used in order to avoid the formation of achiral *cis*-tetrahydroepoxyfuran, gave the benzyl ether **5a** which could, in turn, be converted to the corresponding phosphine. However, all attempts to cleave the benzyl ether in the presence of the phosphine under a variety of conditions (e.g. catalytic or stoichiometric palladium on charcoal or prolonged heating with lithium alanate in THF) failed. This was also the case when the phosphine group had been protected as the corresponding phosphine–borane.[19] Protection of the hydroxy group as its tetrahydropyranyl ether proved more advantageous. Thus, reaction of alcohols **4a** and **4b** with 2,4-dihydro-2*H*-pyran in the presence of catalytic amounts of pyridinium *p*-toluenesulfonate (PPTS)[20] gave **6a** and **6b**. The tetrahydropyran derivatives were formed as diastereomeric mixtures which differed in configuration at the newly created stereogenic center.[21] Nucleophilic displacement of the mesyl group by diphenylphosphide yields the phosphine derivatives **7a** and **7b** with inversion of configuration at C-3. Removal of the functional group with a catalytic amount of methanesulfonic acid or trifluoracetic acid in aqueous methanol gave the desired hydroxy phosphines **8a** and **8b** which were purified by flash chromatography. It is noteworthy that under the described conditions of acetal cleavage, the phosphine remains untouched, as do the stereocenters. This is in contrast to hydrolyses utilizing more nucleophilic acids such as HCl.[22]

In conclusion, this procedure efficiently converts L-ascorbic acid and D-isoascorbic acid to chiral 3-phosphino-4-hydroxytetrahydrofurans in acceptable overall yield. It demonstrates the marked versatility of tetrahydrofurans **4** as building blocks and simultaneously stresses the importance of protecting groups in the synthesis of hydroxy phosphines. We are currently investigating the application of the synthesized compounds in asymmetric catalysis.

Experimental Section

All experiments involving phosphines were conducted under argon using conventional Schlenck techniques. Flash chromatography was performed on silica gel 60 (230–400 mesh, Merck). Melting points are corrected. Optical rotations were measured at 22 + 2 °C. ^{1}H NMR spectra were recorded at 250 MHz using tetramethylsilane as an internal standard. ^{13}C and ^{31}P NMR spectra were recorded at 100 and 60 MHz, respectively.

(2*S*,3*S*)-1,2-*O*-Isopropylidene-3,4-di-*O*-mesylbutane-1,2,3,4-tetrol (3a). To a solution of 2.43 g (15 mmol) of (2*S*,3*S*)-1,2-*O*-isopropylidenebutane-1,2,3,4-tetrol (**2a**)[13a] in 7.2 mL of dry pyridine at 0 °C was added 3.5 mL of methanesulfonyl chloride (30 mmol). The mixture was stirred at 0 °C for 15 min and then allowed to warm to room temperature. Stirring was continued for 6 h. The reaction mixture was poured onto excess ice/water and extracted with CH_2Cl_2 (3 × 100 mL). The combined extracts were washed with 5% aqueous H_2SO_4, $NaHCO_3$ solution, and

(14) For the base-promoted cyclization of (2*S*,3*S*)-1-*O*-tosylthreitol to (3*S*,4*S*)-3,4-dihydroxytetrahydrofuran see Jackson, W. R., Lovel, C. G. *Aust. J. Chem.* **1982**, *35*, 2069.

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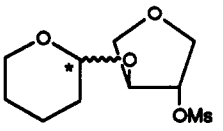

(22) Acid hydrolysis of DIOP with aqueous HCl (Deschenaux, R.; Stille, J. K. *J. Org. Chem.* **1985**, *50*, 2299) gave rise to the formation of considerable quantities of quarternary phosphonium compounds: Börner, A.; Ward, J.; Kortus, K; Kagan, H. B. *Tetrahedron: Asymmetry*, accepted for publication.

water, dried (Na_2SO_4), and concentrated. Recrystallization from ethanol afforded **3a** (4.05 g, 85%): mp 85 °C; $[\alpha]_D$ = −2.9° (*c* 1, acetone); ^{1}H NMR ($CDCl_3$) δ 4.81 (1H, ddd), 4.36 (2H, m), 4.28 (1H, ddd), 4.07 (1H, dd, *J* = 9.1, 6.7 Hz), 3.89 (1H, dd, *J* = 9.1, 5.5 Hz), 3.13 (3H, s), 3.08 (3H, s), 1.48 (3H, s), 1.35 (3H, s); ^{13}C NMR ($CDCl_3$) δ 110.5 (Me_2C), 78.6 (C-3), 73.9 (C-2), 67.6 (C-4), 65.2 (C-1), 38.8, 37.7 (CH_3SO_2), 26.1, 25.0 (CH_3C); IR (KBr) 1347, 1328, 1171, 1087 cm^{-1}; MS *m/e* 303 ($M^+ - CH_3$, 80), 165 ($M^+ - CH_3 - CH_3SO_3 - C_2H_3O$, 8), 101 ($C_5H_9O_2^+$, 55), 79 ($CH_3SO_2^+$, 32), 69 (45), 43 ($C_2H_3O^+$, 100). Anal. Calcd for $C_9H_{18}O_8S_2$: C, 33.95; H, 5.70. Found: C, 33.69; H, 5.57.

(2*R*,3*S*)-1,2-*O*-Isopropylidene-3,4-di-*O*-mesylbutane-1,2,3,4-tetrol (3b). This compound was prepared by the method used for **3a** starting from (2*R*,3*S*)-1,2-*O*-isopropylidenebutane-1,2,3,4-tetrol (**2b**)[13b]. The resultant colorless oil was been purified by flash chromatography (*n*-hexane/AcOEt 1/1) to give **3b** (85%): $[\alpha]_D$ = −8.5° (*c* 1; $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 4.77 (1H, ddd, *J* = 6.8 Hz), 4.59 (1H, dd, *J* = 2.4 Hz), 4.38 (1H, dd, *J* = 11.9, 5.4 Hz), 4.24 (1H, ddd, *J* = 6.8 Hz), 4.13 (1H, dd, *J* = 6.2 Hz), 4.03 (1H, dd, *J* = 9.0, 4.9 Hz), 3.13 (3H, s), 3.07 (3H, s), 1.43 (3H, s), 1.37 (3H, s); ^{13}C NMR (CDCl3) δ 110.5 (Me_2C), 77.9 (C-3), 73.3 (C-2), 67.9 (C-4), 66.0 (C-1), 38.8, 37.6 (CH_3SO_2), 26.5, 24.8 (CH_3C). Anal. Calcd for $C_9H_{18}O_8S_2$: C, 33.95; H, 5.70. Found: C, 33.75; H, 5.75.

(3*S*,4*S*)-3-(Mesyloxy)-4-hydroxytetrahydrofuran (4a). A solution of 2.0 g (6.3 mmol) of **3a** in 70 mL of methanol and 0.4 mL of concentrated hydrochloric acid was heated under reflux, and the reaction was monitored by TLC (toluene/acetone 7/3). After completion of the reaction the solvent was evaporated. To the resultant colorless oil was added 50 mL of dry ethanol. The resultant clear solution was again concentrated. This procedure was repeated three times and the resultant product recrystallized from ethanol to give **4a** (1.04 g, 90%): mp 107–108 °C; $[\alpha]_D$ = +16.5° (*c* 1, acetone); ^{1}H NMR (acetone) δ 4.84 (1H, m), 4.32 (1H, m), 3.92 (1H, dd, *J* = 10.8, 3.9 Hz), 3.85 (1H, dd, *J* = 9.7, 4.5 Hz), 3.82–3.74 (2H, m, 1 proton exchangeable with D_2O), 3.53 (1H, dd, *J* = 9.7, 2.1 Hz), 3.07 (3H, s); ^{13}C NMR (acetone) δ 86.5 (C-4), 75.8 (C-3), 74.1 (C-2), 71.7 (C-5), 38.3 (CH_3SO_2); IR (KBr) 3322, 1418, 1350, 1175 cm^{-1}; MS *m/e* 139 ($M^+ - C_2H_3O$, 13), 122 ($M^+ - C_2H_4O_2$, 52), 86 ($C_4H_6O_2^+$, 40), 79 ($CH_3SO_2^+$, 75), 57 ($C_3H_5O^+$, 47), 43 ($C_2H_3O^+$, 100). Anal. Calcd for $C_5H_{10}O_5S$: C, 32.96; H, 5.53. Found: C, 32.68; H, 5.43.

(3*S*,4*R*)-3-(Mesyloxy)-4-hydroxytetrahydrofuran (4b). This compound was prepared by the method used for **4a** starting from **3b** to yield **4b** (90%): mp 78 °C; $[\alpha]_D$ = +11.3° (*c* 1, acetone); ^{1}H NMR ($CDCl_3$) δ 5.02 (1H, m), 4.42 (1H, m), 4.04 (1H, dd, *J* = 5.6 Hz), 3.96 (1H, dd, *J* = 5.9 Hz), 3.91 (1H, dd, *J* = 10.4, 4.3 Hz), 3.67 (1H, dd, *J* = 9.6, 5.5 Hz), 3.08 (3H, s) 2.38 (1H, b, exchangeable with D_2O); ^{13}C NMR ($CDCl_3$) δ 79.0 (C-3), 70.0, 70.6, 71.9 (C-4, C-2, C-5) 38.5 (CH_3SO_2); IR (KBr): 3310, 1349, 1175, 1053 cm^{-1}; MS *m/e* 139 ($M^+ - C_2H_3O$, 26), 122 ($M^+ - C_2H_4O_2$, 100), 86 ($C_4H_6O_2^+$, 25), 79 ($CH_3SO_2^+$, 78). Anal. Calcd for $C_5H_{10}O_8S$: C, 32.96; H, 5.53. Found: C, 33.25; H, 5.50.

(3*S*,4*S*)-4-(Benzyloxy)-3-(mesyloxy)tetrahydrofuran (5a). To a stirred solution of 0.5 g (2.75 mmol) of **4a** and 0.61 mL of benzyl 2,2,2-trichloroethanimidate (3.30 mmol) in a mixture of 50 mL of cyclohexane and 15 mL of CH_2Cl_2 was added 0.04 mL of trifluoromethanesulfonic acid under argon and the mixture stirred overnight. The crystalline trichloroethanimidate was removed by filtration and the filtrate washed with 50 mL of saturated aqueous $NaHCO_3$. The organic layer was dried (Na_2SO_4) and evaporated and the residue purified by flash chromatography (*n*-hexane/AcOEt 1/1) to give **5a** as a colorless oil (0.37 g, 50%): $[\alpha]_D$ = +1.2° (*c* 1.15, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 7.40–7.26 (5H, m), 5.09 (1H, m), 4.57 (2H, dd, *J* = 13.8 Hz), 4.24 (1H, m), 4.06–3.94 (3H, m), 3.74 (1H, dd, *J* = 10.1, 2.5 Hz), 3.00 (3H, s); ^{13}C NMR ($CDCl_3$) 137.2, 128.6, 128.3, 127.9 (arom), 82.3, 82.2 (C-3, C-4), 72.0, 71.8, 71.4 (C-2, C-5, OCH_2Ph), 38.6 (CH_3SO_2); IR (KBr) 1364, 1176 cm^{-1}; MS *m/e* 272 (M^+, 40), 151 ($M^+ - C_7H_7 - CH_2O$, 36), 107 ($C_7H_7O^+$, 100), 91 ($C_7H_7^+$, 100), 79 ($CH_3SO_2^+$, 50), 70 ($C_4H_6O^+$, 62). Anal. Calcd for $C_{12}H_{16}O_5S$: C, 52.93; H, 5.92. Found: C, 52.71; H, 5.95.

(3*S*,4*S*,2′*rac*)-3-(Mesyloxy)-4-*O*-(tetrahydropyran-2′-yl)-tetrahydrofuran-4-ol (6a). To a solution of 0.91 g (5 mmol) of **4a** in 35 mL of dry CH_2Cl_2 was added 125 mg of pyridinium *p*-toluenesulfonate (PPTS) and 0.68 mL of 3,4-dihydro-2*H*-pyran. The mixture was stirred for 5 h at room temperature. The solution was diluted with 100 mL ether and washed twice with half-saturated brine and water. After drying the ether solution (Na_2SO_4) the solvent was evaporated and the residue purified by flash chromatography (*n*-hexane/AcOEt 1/1) to furnish the diastereomeric mixture of **6a** as a colorless oil (1.05 g, 80%): ^{1}H NMR ($CDCl_3$) δ 5.27/5.08 (1H, 2 × m), 4.80/4.68 (1H, 2 × m), 4.52/4.41 (1H, 2 × m), 4.18–3.75, 3.55 (6H, 2 × m), 3.04/3.05 (3H, 2 × s), 1.85–1.35 (6H, m); ^{13}C-NMR ($CDCl_3$) δ 99.5/98.2 (C-2′), 84.0/82.7 (C-3), 81.0/79.4 (C-4), 72.3/72.3, 71.6/71.2 (C-5, C-2), 63.2/62.7 (C-6′), 38.6/38.2 (CH_3SO_2), 30.7/30.5 (C-3′), 25.2/25.1, 19.6/19.3 (C-4′, C-5′). Anal. Calcd for $C_{10}H_{18}O_6S$: C, 45.11; H, 6.82. Found: C, 44.98; H, 6.92.

(3*S*,4R,2′*rac*)-3-(Mesyloxy)-4-*O*-(tetrahydropyran-2′-yl)-tetrahydrofuran-4-ol (6b). This compound was prepared by the method used for **6a** starting from **4b** to give the diastereomeric mixture **6b** as a colorless oil (85%): ^{1}H NMR ($CDCl_3$) δ 5.12 (1H, m), 4.75/4.58 (1H, 2 × m) 4.38 (1H, m), 4.12–3.98, 3.75, 3.52 (6H, 2 × m), 3.12/3.08 (3H, 2 × s), 1.77–1.40 (6H, m); ^{13}C NMR ($CDCl_3$) δ 99.7/99.0 (C-2′), 80.2/78.1 (C-3), 75.0/74.6 (C-4), 71.7/71.0 (C-5), 69.5/68.1 (C-2), 63.7/62.6 (C-6′), 38.8/38.7 (CH_3SO_2), 30.6/30.2 (C-3′), 25.2/25.2, 20.0/19.2 (C-4′, C-5′). Anal. Calcd for $C_{10}H_{18}O_6S$: C, 45.11; H, 6.82. Found: C, 45.01; H, 6.83.

(3*R*,4*S*,2′*rac*)-3-(Diphenylphosphino)-4-*O*-(tetrahydropyran-2′-yl)tetrahydrofuran-4-ol (7a). A solution of lithium diphenylphosphide was generated from 0.14 g of lithium strips and 0.98 mL (5.3 mmol) of freshly distilled chlorodiphenylphosphine in dry THF. The resultant deep red solution was added at 0 °C to a solution of 1.0 g (3.75 mmol) of **6a** in dry THF over a period of 15 min. The solution was stirred for a further 2 h at room temperature and then the solvent removed. The residue was dissolved in 5 mL of ethanol and allowed to stand in a refrigerator. After a few hours a solid precipitated from the solution, which was filtered and dried. The crude phosphine was subjected to flash chromatography to give the diastereomeric mixture of **7a** as a white solid (0.38 g, 30%): ^{1}H NMR ($CDCl_3$) δ 7.60–7.25 (10H, m), 4.68/4.51 (1H, 2 × m), 4.25–3.00 (8H, m), 1.35–1.80 (6H, m); ^{13}C NMR ($CDCl_3$) δ 137.8–128.4 (arom), 100.8 (d, $^4J_{C-P}$ = 1.8 Hz)/94.6 (C-2′), 80.5 (d, $^2J_{C-P}$ = 5.0 Hz)/74.9 (d, $^2J_{C-P}$ = 5.7 Hz) (C-4), 62.6/60.9 (C-6′), 70.9 (d, $^2J_{C-P}$ = 25.8 Hz)/70.8 (d, $^2J_{C-P}$ = 24.0 Hz) (C-2), 75.2 (d, $^3J_{C-P}$ = 2.6 Hz)/72.5 (d, $^3J_{C-P}$ = 3.1 Hz) (C-5), 43.7 (d, $^1J_{C-P}$ = 11.5 Hz)/43.2 (d, $^1J_{C-P}$ = 13.5 Hz) (C-3), 30.3/30.1 (C-3′), 25.4/25.3, 19.4/18.3 (C-4′, C-5′); ^{31}P-NMR ($CDCl_3$) δ −22.2/−22.3 (s). Anal. Calcd for $C_{21}H_{25}O_3P$: C, 70.78; H, 7.07. Found: C, 70.83; H, 7.12.

(3*R*,4*R*,2′*rac*)-3-(Diphenylphosphino)-4-*O*-(tetrahydropyran-2′-yl) tetrahydrofuran-4-ol (7b). This compound was prepared by the method used for **7a** to give the diastereomeric mixture of **7b** (35%): ^{1}H NMR ($CDCl_3$) δ 7.61–7.15 (10 H, m), 4.52–2.92 (9H, m), 1.70–1.24 (6H, m); ^{13}C NMR ($CDCl_3$) δ 137.2–128.3 (arom), 99.4/96.8 (C-2′), 82.1 (d, $^2J_{C-P}$ = 23.0 Hz)/79.3 (d, $^2J_{C-P}$ = 26.0 Hz)(C-4), 63.1/61.7 (C-6′), 70.7 (d, $^2J_{C-P}$ = 23.1 Hz)/70.6 (d, $^2J_{C-P}$ = 20.6 Hz)(C-2), 74.3 (d, $^3J_{C-P}$ = 4.0 Hz)/72.4 (d, $^3J_{C-P}$ = 3.1 Hz) (C-5), 43.9 (d, $^1J_{C-P}$ = 11.6 Hz)/43.7 (d, $^1J_{C-P}$ = 11.6 Hz) (C-3), 30.8/30.6 (C-3′), 25.3/25.3, 20.0/19.0 (C-4′, C-5′); ^{31}P NMR ($CDCl_3$) δ −12.0/−11.0 (s). Anal. Calcd for $C_{21}H_{25}O_3P$: C, 70.78; H, 7.07. Found: C, 70.99; H, 6.85.

(3*R*,4*S*)-3-(Diphenylphosphino)-4-hydroxytetrahydrofuran (8a). A solution of 250 mg (0.75 mmol) of diastereomeric mixture **7a** in 50 mL of methanol, 5 mL of water, and 60 μL of methanesulfonic acid was heated under reflux for 4 h. The solution was cooled to room temperature and the solvents removed under reduced pressure. The residue was purified by flash chromatography (CH_2Cl_2/AcOEt 20/1) to yield the hydroxy phosphine **8a** as a white solid (184 mg, 90%): mp 87–88 °C; $[\alpha]_D$ = −18.8° (*c* 0.5, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 7.60–7.30 (10H, m), 4.40 (1H, m), 3.92–3.81 (4H, m), 3.03 (1H, m), 2.05 (1H, exchangeable with D_2O); ^{31}C NMR ($CDCl_3$) δ 137.3–129.0 (arom), 76.4 (C-4, d, $^2J_{C-P}$ = 3.1 Hz), 73.7 (C-5, d, $^3J_{C-P}$ = 7.7 Hz), 70.2 (C-2, d, $^2J_{C-P}$ = 24.0 Hz), 45.1 (C-3, d, $^1J_{C-P}$ = 10.3 Hz); ^{31}P NMR ($CDCl_3$) δ −14.0 (s); IR (KBr) 3392, 3050, 1434, 1262, 1099 cm^{-1}; MS *m/e* 272 (M^+, 30), 255 ($M^+ - OH$, 9), 213 ($M^+ - OH - C_2H_2O$, 30), 186 ($HPPh_2^+$, 100), 108 (PPh^+, 93), 91 ($C_7H_7^+$, 8), 77 ($C_6H_5^+$, 9). Anal. Calcd for $C_{16}H_{17}O_2P$: C, 70.58; H, 6.30. Found: C, 70.76; H, 6.12.

(3*R*,4*R*)-3-(Diphenylphosphino)-4-hydroxytetrahydrofuran (8b). This compound was prepared by the method used for **8a** to give, after flash chromatography (CH_2Cl_2/AcOEt 20/1), the hydroxy phosphine **8b** as a colorless oil(90%): $[\alpha]_D = -19.8°$ (*c* 1.1, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 7.60–7.30 (10H, m), 4.20 (1H, m, J = 10.3, 4.0, 2.5, 1.1 Hz), 4.06 (1H, ddd, J= 12.1, 9.3, 8.0 Hz), 3.70 (2H, m), 3.48 (1H, J = 12.9, 9.3, 6.7 Hz), 2.85 (1H, m), 2.38 (1H, exchangeable with D_2O); ^{31}C NMR ($CDCl_3$) δ 137.3–128.7 (arom), 76.0 (C-4, d, $^2J_{P-C}$ = 24.5 Hz), 75.5 (C-5, d, $^3J_{P-C}$ = 3.7 Hz), 70.8 (C-2, d, $^2J_{P-C}$ = 23.0 Hz), 45.9 (C-3, d, $^1J_{P-C}$ = 11.4 Hz); ^{31}P NMR ($CDCl_3$) δ −11.8 (s); IR (neat) 3394, 3071, 1434, 1330, 1181, 1081 cm^{-1}. Anal. Calcd for $C_{16}H_{17}O_2P$: C, 70.58; H, 6.30. Found: C, 70.55; H, 6.22.

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