(R)-2,3-O-Cyclohexylideneglyceraldehyde, a Versatile Intermediate for Asymmetric Synthesis of Chiral Alcohol

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Grignard addition to (*R*)-2,3-*O*-cyclohexylideneglyceraldehyde **(IIa)** gave rise to column chromatographically separable diastereo alcohols **2** and **3** including highly functionalized and synthetically exploitable homoallylic alcohols **2e** and **3e** and homopropargylic alcohols **2f** and **3f**. Compound **3c** on functional manipulation gave rise to (−)-coriolic acid synthon **6**.

Diastereoselective synthesis of enantiomeric alcohols via nucleophilic addition of organometallic to carbonyl in the presence of varied chiral auxiliaries has drawn considerable attention over the ages.[1] In this endeavor a number of functionalized α-alkoxy,[2a] β-alkoxy,[2b] α,β-dialkoxy,[2c,3] α,β-epoxide,[2d] and other α- or β-hetero-substituted[2e−g] carbonyls have been chosen as targets. Of them, (*R*)-2,3-isopropylideneglyceraldehyde **(Ia)** and its (*S*)-isomer **Ib** have been most widely used due to their easy availability, synthetically exploitable high functional density, and structural simplicity.[3] Consequently, additions of different types of nucleophiles viz. alkyl, aryl, allyl, vinyl, masked formyl,[3b] etc. through their organometallics (Mg, Li, Zn, Cu,[3c] Cr, Ti, B, Si, In,[3d] etc.) to **I** or its analogs[3e] under varied conditions (temperature, solvent, presence of metal salt, etc.) have been studied. Usually, most of these additions afford poor to medium diastereoselectivity especially for organolithium, organozinc, and organomagnesium whereas high but not absolute selectivity is observed in the case of some difficultly obtained organometallics of a limited variety of nucleophiles.

So far, addition to **I** with moderate diastereoselectivity has not been considered to be synthetically useful, and the resulting diastereoisomeric alcohols are not easily separable with conventional procedures. There are reports of separation of their derivatives such as benzyl,[3f] benzoate,[3g] and substituted acetate[3h] through column chromatography. But these derivatives may not always be compatible with the subsequent reaction's protocol. Moreover, mild acidic environment during reaction or workup can lead to deketalization of the sensitive isopropylidene moiety of **I**, causing difficulty in selective manipulation of the hydroxyl groups.

In our ongoing program on the synthesis of insect pheromones,[4] marine natural products,[5] and other biologically important compounds, we need pure and substantial amounts of each diastereoisomeric alcohol resulting from alkylation, allylation, and propargylation of conventionally protected glyceraldehyde. In this endeavor, we found (*R*)-cyclohexylideneglyceraldehyde **(IIa)** to be a good candidate for this purpose. Moreover, it is free from the drawbacks generally associated with **I** for the latter's high water solubility, high volatility, and increased affinity for polymerization. We prepared **IIa** with a modification of the reported[6] procedure employing 2 equiv of $NaIO_4$ in 60% aqueous acetonitrile for cleavage of **1**. The aldehyde **IIa**, thus obtained almost quantitatively, was sufficiently pure for use. Its solution (THF) was treated with the corresponding Grignards, prepared in appropriate solvents (as shown in Table 1), at −50 °C to give the addition products. Reports of some stereocontrolled nucleophilic additions to **IIa** and its enantiomer **IIb**[8] are available in the literature,[7] but none of their products conforms to our present synthetic program.

All the Grignard additions we performed resulted in good yield with low to medium diastereoselectivity with predominant formation of the *anti* isomer. In each case the diastereoisomeric alcohols were separated nicely by ordinary column chromatography (SiO_2) without any need for being derivatized. The *syn* and *anti* relationship

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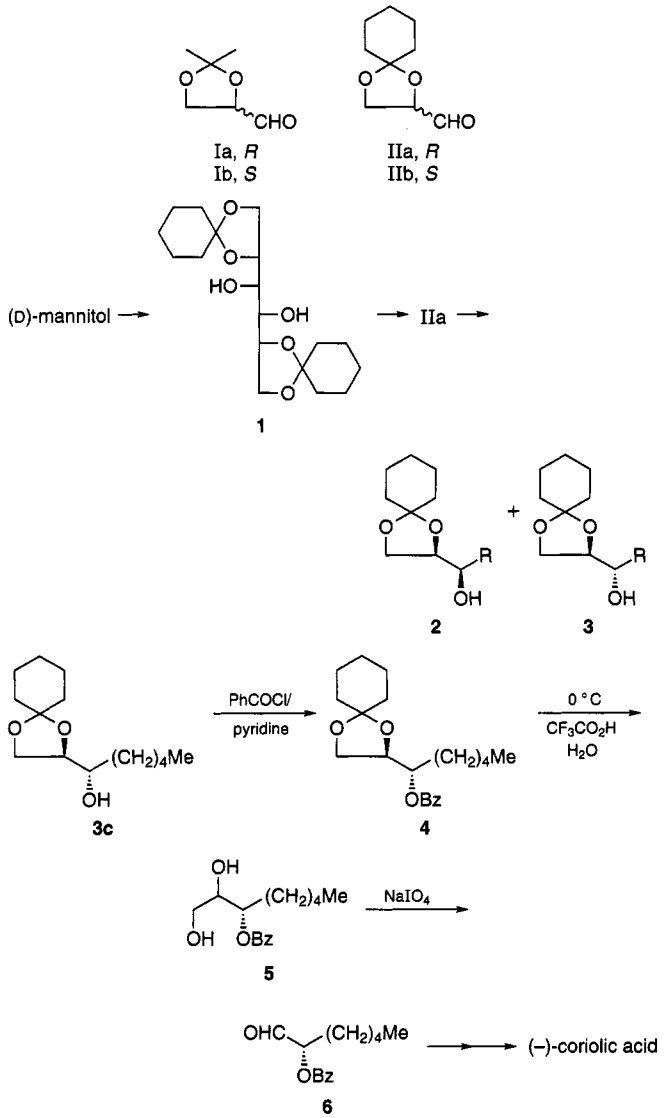
Table 1 1

entry	grignard	solvent	% yield (2)	% yield (3)
a	MeMgI	Et_2O	24.4	56.4
b	nBuMgBr	THF	23.5	63.5
c	$nC_5H_{11}MgBr$	THF	25.8	62.9
d	$nC_8H_{17}MgBr$	THF	18.9	71.0
e	$H_2C{=}CHCH_2MgI$	Et_2O	19.5	64.8
f	$HC{\equiv}CCH_2MgBr$	Et_2O	12.9	69.5

Scheme 1



was established by comparing their ^{1}H-NMR data for $-CHO-$ and $-CH_2O-$ with the reported pattern.[3h,9] In almost all cases the *syn* isomer shows three separable multiplets in the region of δ 3.5–4.1, and the *anti* isomer shows a complex multiplet in the region of δ 3.7–4.2 for the alkyl addition product and two separable multiplets for homoallylic and homopropargylic alcohols.

The efficacy of this method of preparation of diastereomeric alcohols from **IIa** is due to its operational simplicity. Hence, it should be amenable to large scale synthesis. Its novelty is further established from the effective separation of diastereomeric homoallylic (entry e) and homopropargylic (entry f) alcohols which proved unsuccessful while using **I** as reported recently by Schmid et al.[3d] These alcohols are extremely useful for many synthetic elaborations due to the diverse nature of functionalities present in them.

The alcohol **(3c)** was benzoylated and subsequently deketalized. Cleavage of the resulting diol **(5)** afforded **6**,[10] a useful synthon of (−)-coriolic acid,[11] which is a natural defensive substance acting against rice blast disease. The spectral and optical data are in accordance with those reported.[10]

By use of our methodology hopefully another series of diastereomeric alcohols can be obtained from the enantiomer **IIb**.

Experimental Section

All bp's are uncorrected. All the anhydrous reactions were carried out under argon atmosphere using freshly distilled anhydrous solvents. Unless otherwise mentioned, the organic extracts were dried over anhydrous Na_2SO_4.

(*R*)-(+)-Cyclohexylideneglyceraldehyde (IIa). To a stirred solution of **1** (68.4 g, 0.2 mol) in 300 mL of aqueous acetonitrile (60%) at 0–10 °C under nitrogen atmosphere was added $NaIO_4$ (85.6 g, 0.4 mol) in small portions over a period of 40 min. The mixture was stirred for 1 h more and filtered. The filtrate was mixed with water and extracted with chloroform. The combined organic layer was washed with water and brine and dried. Solvent removal under reduced pressure afforded **IIa** in almost quantitative yield. This was sufficiently pure and hence used as such for the next step. A part of the residue was distilled under vacuum for characterization: bp 96–98 °C/10 mmHg (lit.[6] bp 90–93 °C/2 mmHg); $[\alpha]^{23}$ +60.5° (*c* 3.5, benzene) [lit.[6] $[\alpha]^{25}$ +61.5° (*c* 3.4, benzene)]; IR (film) 2720, 1740, 1455, 1375, 1340, 1235, 1180, 920; ^{1}H-NMR ($CDCl_3$) δ 1.4–1.6 (m, 10H), 4.0–4.2 (m, 2H), 4.3–4.4 (m, 1H), 9.75 (d, J = 2.0 Hz, 1H).

General Procedure for Preparation of 2 and 3. To a stirred solution of Grignard reagent at −50 °C, prepared by treating halide (0.075 mol) with a suspension of Mg (1.92 g, 0.08 g equiv) in an appropriate solvent (100 mL) (as shown in Table 1), was added **IIa** (5.1 g, 0.03 mol) in THF (60 mL) over a period of 1 h. The mixture was stirred at −50 °C for 3 h and at room temperature overnight to ensure completion of the reaction. Saturated aqueous NH_4Cl was added followed by extraction with ether. The organic layer was washed with water and brine, dried, and evaporated under reduced pressure. The residue was column chromatographed (SiO_2) eluting with 0–25% ethyl acetate in hexane resulting in separation of pure **2** and **3**. Chromatography was repeated twice for entries a, e and f and once for entries b–d for complete separation of the diastereoisomers. In all cases the minor *syn* isomer was eluted first followed by the major *anti* isomer. TLC (16 cm plate) showed a spot for each diastereoisomer whose R_f (with solvent system) values are given below.

1,2-Cyclohexylidenebutane-1,2,3-triol (2a and 3a). Minor (2*R*,3*R*)-isomer (2a): yield 1.34 g (24.4%); R_f 0.62 (25% EtOAc/ hexane); $[\alpha]^{20}$ +3.5° (*c* 1.06, $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1290, 1180, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 1.06 (d, J = 6.2 Hz, 3H), 1.4–1.6 (m, 10H), 2.7 (br s, D_2O exchangeable, 1H), 3.5 (m, 1H), 3.7–3.8(m, 1H), 3.9–4.1 (m, 2H).

Major (2*R*,3*S*)-isomer (3a): yield 3.14 g (56.4%); R_f 0.57 (25% EtOAc/ hexane); $[\alpha]^{20}$ +7.4° (*c* 1.15, $CHCl_3$); IR 3450, 1460, 1380, 1340, 1290, 1180, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 1.06 (d, J = 6.2 Hz, 3H), 1.4–1.6 (m, 10H), 2.5 (bs, D_2O exchangeable, 1H), 3.7–4.2 (m, 2H).

Anal. Calcd for $C_{10}H_{18}O_3$: C, 64.49; H, 9.74. Found: C, 64.71; H, 9.61 for **2a**; C, 64.62; H, 9.55 for **3a**.

1,2-Cyclohexylideneheptane-1,2,3-triol (2b and 3b). Minor (2*R*,3*R*)-isomer (2b): yield 1.61 g (23.5%); R_f 0.81 (20% EtOAc/ hexane); $[\alpha]^{23}$ +3.7° (*c* 1.17, $CHCl_3$); IR (film) 3450,

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1460, 1380, 1340, 1285, 1180, 1130, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.91 (t, J = 6.5 Hz, 3H), 1.1–1.3 (m, 6H), 1.4–1.6 (m, 10H), 2.1 (br s, D_2O exchangeable, 1H), 3.5 (m, 1H), 3.7 (m, 1H), 3.9–4.1 (m, 2H).

Major (2*R*,3*S*)-isomer (3b): yield 4.34 g (63.5%); R_f 0.70 (20% EtOAc/ hexane); $[\alpha]^{23}$ +9.2° (*c* 1.21, $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1290, 1180, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.92 (t, J = 6.5 Hz, 3H), 1.1–1.3 (m, 6H), 1.4–1.6 (m, 10H), 2.6 (br s, D_2O exchangeable, 1H), 3.7–4.2 (m, 2H).

Anal. Calcd for $C_{13}H_{24}O_3$: C, 68.38; H, 10.59. Found: C, 68.61; H, 10.81 for **2b**; C, 68.52; H, 10.65 for **3b**.

1,2-Cyclohexylideneoctane-1,2,3-triol (2c and 3c). Minor (2*R*,3*R*)-isomer (2c): yield 1.87 g (25.8%); R_f 0.82 (20% EtOAc/ hexane); $[\alpha]^{20}$ +4.1° (*c* 1.15 $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1285, 1180, 1130, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.91 (t, J = 6.5 Hz, 3H), 1.1–1.3 (m, 8H), 1.4–1.6 (m, 10H), 2.4 (br s, D_2O exchangeable, 1H), 3.5 (m, 1H), 3.7 (m, 1H), 3.9–4.1 (m, 2H).

Major (2*R*,3*S*)-isomer (3c): yield 4.57 g (62.9%); R_f 0.71 (20% EtOAc/ hexane); $[\alpha]^{20}$ +8.2° (*c* 1.18, $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1290, 1130, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.92 (t, J = 6.5 Hz, 3H), 1.1–1.3 (m, 8H), 1.4–1.6 (m, 10H), 2.3 (br s, D_2O exchangeable, 1H), 3.7–4.2 (m, 2H).

Anal. Calcd for $C_{14}H_{26}O_3$: C, 69.38; H, 10.81. Found: C, 69.19; H, 10.64 for **2c**; C, 69.32; H, 10.75 for **3c**.

1,2-Cyclohexylideneundecane-1,2,3-triol (2d and 3d). Minor (2*R*,3*R*)-isomer (2d): yield 1.61 g (18.9%); R_f 0.79 (15% EtOAc/ hexane); $[\alpha]^{25}$ +2.7° (*c* 1.36, $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1285, 1180, 1130, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.91 (t, J = 6.5 Hz, 3H), 1.1–1.3 (m, 14H), 1.4–1.6 (m, 10H), 2.2 (br s, D_2O exchangeable, 1H), 3.5 (m, 1H), 3.7 (m, 1H), 3.9–4.1 (m, 2H).

Major (2*R*,3*S*)-isomer (3d): yield 6.04 g (71.0%); R_f 0.68 (15% EtOAc/ hexane); $[\alpha]^{25}$ +6.2° (*c* 1.15, $CHCl_3$); IR (film) 3450, 1460, 1380, 1340, 1290, 1130, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 0.92 (t, J = 6.5 Hz, 3H), 1.1–1.4 (m, 14H), 1.4–1.6 (m, 10H), 2.8 (br s, D_2O exchangeable, 1H), 3.7–4.2 (m, 2H).

Anal. Calcd for $C_{17}H_{32}O_3$: C, 71.78; H, 11.34. Found: C, 71.61; H, 11.55 for **2d**; C, 71.62; H, 11.48 for **3d**.

1,2-Cyclohexylidene-5-hexene-1,2,3-triol (2e and 3e). Minor (2*R*,3*R*)-isomer (2e): yield 1.24 g (19.5%); R_f 0.80 (20% EtOAc/ hexane); $[\alpha]^{23}$ +5.3° (*c* 1.34, $CHCl_3$); IR (film) 3450, 3005, 1650, 1475, 1390, 1340, 1305, 1180, 1130, 1060, 995, 920; ^{1}H-NMR ($CDCl_3$) δ 1.4–1.6 (m, 10H), 2.24 (t, J = 6.5 Hz, 2H overlapped with a bs, D_2O exchangeable, 1H), 3.5–3.6 (m, 1H), 3.7–3.8 (m, 1H), 3.9–4.1 (m, 2H), 5.0–5.2 (m, 2H), 5.7–6.0 (m, 1H).

Major (2*R*,3*S*)-isomer (3e): yield 4.12 g (64.8%); R_f 0.73 (20% EtOAc/ hexane); $[\alpha]^{25}$ +10.2° (*c* 1.41, $CHCl_3$); IR (film) 3450, 3005, 1650, 1475, 1390, 1340, 1305, 1130, 1120, 1060, 995, 920; ^{1}H-NMR ($CDCl_3$) δ 1.4–1.6 (m, 10H), 2.0 (br s, D_2O exchangeable, 1H), 2.1–2.3 (m, 2H), 3.6–3.8 (m, 1H), 3.9–4.2 (m, 3H), 5.0–5.2 (m, 2H), 5.7–6.0 (m, 1H).

Anal. Calcd for $C_{12}H_{20}O_3$: C, 67.89; H, 9.50. Found: C, 67.64; H, 9.65 for **2e**; C, 67.82; H, 9.25 for **3e**.

1,2-Cyclohexylidenehex-5-yne-1,2,3-triol (2f and 3f). Minor (2*R*,3*R*)- isomer (2f): yield 0.82 g (12.9%); R_f 0.68 (20% EtOAc/ hexane); $[\alpha]^{23}$ +5.7° (*c* 1.25, $CHCl_3$); IR (film) 3450, 3320, 1470, 1390, 1340, 1300, 1180, 1130, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 1.4–1.6 (m, 10H), 2.05 (t, J = 1.5 Hz, 1H), 2.4–2.5 (m, 2H overlapped with a D_2O exchangeable bs, 1H), 3.5–3.6 (m, 1H), 3.7–3.8 (m, 1H), 3.9–4.1 (m, 2H).

Major (2*R*,3*S*)-isomer (3f): yield 4.38 g (69.5%); R_f 0.62 (20% EtOAc/ hexane); $[\alpha]^{25}$ +9.5° (*c* 1.22, $CHCl_3$); IR (film) 3450, 3320, 1470, 1390, 1340, 1300, 1130, 1120, 1060, 950; ^{1}H-NMR ($CDCl_3$) δ 1.4–1.6 (m, 10H), 2.05 (t, J = 1.5 Hz, 1H), 2.4–2.6 (m, 2H, overlapped with a bs, D_2O exchangeable, 1H), 3.5–3.7 (m, 1H), 3.8–4.1 (m, 3H).

Anal. Calcd for $C_{12}H_{18}O_3$: C, 68.54; H, 8.63. Found: C, 68.80; H, 8.81 for **2f**; C, 68.72; H, 8.55 for **3f**.

(2*S*)-(Benzoyloxy)heptanal (6). To a solution of **3c** (1.4 g, 0.057 mol) in THF (25 mL) at 0 °C was added benzoyl chloride (2 mL, 0.17 mol) dropwise over a period of 30 min. The mixture was stirred for 2 h at 0 °C and 2 h at room temperature, poured into water, and extracted with ether. Usual workup and solvent removal under reduced pressure afforded the residue benzoate **4**. The crude **4** was mixed with 90% aqueous trifluoroacetic acid (15 mL), stirred for 4 h at 0 °C, and diluted with $CHCl_3$ and water. The aqueous layer was extracted with $CHCl_3$. The combined organic layer was washed successively with 2% aqueous $NaHCO_3$ and water until neutral. Solvent removal under reduced pressure gave the residue diol **5**. The product was not purified further and was dissolved in 60% aqueous acetonitrile (20 mL) and treated with $NaIO_4$ (1.5 g) according to a similar experimental procedure as for the preparation of **IIa**. The residue after solvent removal under reduced pressure was chromatographed (SiO_2, 0–10% ethyl acetate in hexane) to furnish **6** (0.424 g, 68%): $[\alpha]^{20}$ −31.1° (*c* 1.41, CH_2Cl_2) [lit.[10] $[\alpha]^{20}$ −31° (*c* 1.4, CH_2Cl_2)]; IR (film) 3010, 2720, 1720, 1650, 1140, 750; ^{1}H-NMR ($CDCl_3$) δ 0.89 (t, J = 6.5 Hz, 3H), 1.2–1.9 (m, 8H), 5.2 (t, J = 6.2 Hz, 1H), 7.3–8.2 (m, 5H), 9.7 (s, 1H).

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