



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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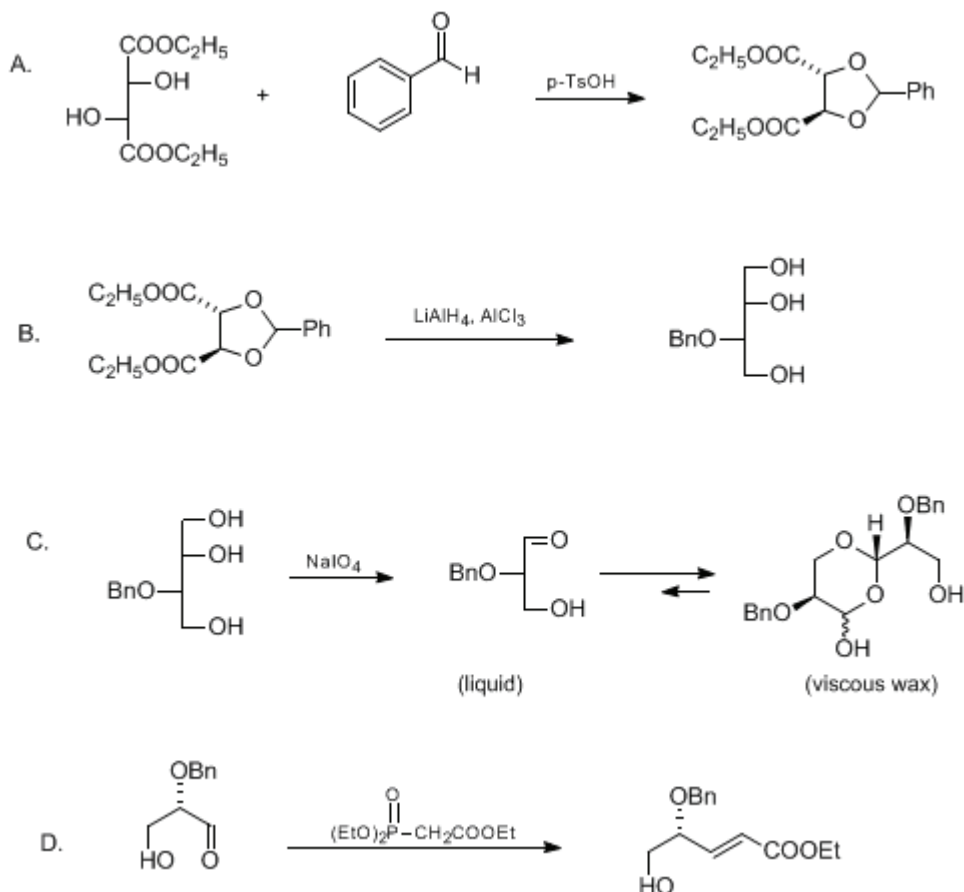
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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(-)-2-O-BENZYL-L-GLYCERALDEHYDE AND ETHYL (R,E)-4-O-BENZYL-4,5-DIHYDROXY-2-PENTENOATE

[Propanal, 3-hydroxy-2-(phenylmethoxy)-, (R)- and 2-Pentenoic acid, 5-hydroxy-4-(phenylmethoxy)-, ethyl ester, [R-(E)]-]



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Checked by Joseph P. Bullock and Louis S. Hegedus.

1. Procedure

Caution: Lithium aluminum hydride is sensitive to mechanical shock and very reactive towards moisture and other protic substances; its dust is very irritating to skin and mucous membranes. It should not be allowed to come into contact with metallic species or apparatus, including metal spatulas, because of the potential danger of metal ion-promoted detonation.

A. *Diethyl (-)-2,3-O-benzylidene-L-tartrate.* A 1-L, round-bottomed flask equipped with a Dean-Stark trap is charged with 103 g (500 mmol) of diethyl L-tartrate (Note 1), 53.1 g (500 mmol) of benzaldehyde (Note 2), 600 mL of cyclohexane and 2.80 g (14.7 mmol) of p-toluenesulfonic acid monohydrate (Note 3). The stirred mixture is heated with azeotropic removal of water (Note 4). The solution is allowed to cool to room temperature and then is concentrated by rotary evaporation (30 mm, 30°C). The residual yellow oil is dissolved in 400 mL of diethyl ether (Note 5), transferred to a 1-L separatory funnel, and washed with saturated aqueous potassium bicarbonate (200 mL), and with water (2 × 200 mL). The ethereal layer is dried over magnesium sulfate and filtered. The solution is

concentrated by rotary evaporation (30 mm, 30°C), followed by removal of solvent at 0.02 mm at room temperature. The solid crude product is collected by suction filtration through a sintered-glass funnel to give diethyl (–)-2,3-O-benzylidene-L-tartrate as yellow crystals. Trituration of the crude solid product with 150 mL of hexanes and collection of the purified product by suction filtration through a sintered-glass funnel gives 105 g (71%) of purified product, mp 47°C (Note 6).

B. (+)-2-O-Benzyl-L-threitol.² A 2-L, three-necked, round-bottomed flask is oven-dried (140°C) and flushed with nitrogen. The flask is equipped with an efficient mechanical stirrer, a 250-mL pressure-equalizing addition funnel, and a reflux condenser equipped with a mineral oil bubbler that is connected to a nitrogen source. A slight pressure of gas is maintained in the apparatus throughout the course of the reaction. The flask is charged with 16.5 g (434 mmol) of lithium aluminum hydride (Note 7), and cooled to –30°C using an isopropyl alcohol-dry ice bath. Then 167 mL of dry diethyl ether (Note 8) is added with vigorous stirring and a solution of 57.8 g (434 mmol) of aluminum chloride in 134 mL of dry diethyl ether (Note 8), (Note 9) is added dropwise during 40 min. Dry dichloromethane (134 mL) (Note 10) is placed in the addition funnel and added rapidly, while the temperature is allowed to rise to 0°C. A solution of 64.7 g (220 mmol) of diethyl (–)-2,3-O-benzylidene-L-tartrate in dry dichloromethane (134 mL) is added dropwise during 30 min (Note 11). The mixture is stirred for 1 hr at room temperature and heated to reflux for an additional 2 hr. The mixture is cooled to –20°C as above, and 14 mL of de-ionized water, followed by a solution of 31.5 g (561 mmol) of potassium hydroxide in 46 mL of de-ionized water, is added cautiously. The cooling bath is removed and the mixture is stirred at room temperature until the grey color (probably due to unreacted lithium aluminum hydride) has completely disappeared (Note 12). Efficient stirring is required throughout to ensure good yields. The mixture is filtered through a glass-sintered funnel containing a 2-cm pad of Celite, and the inorganic precipitate is extracted with 0.5 L of dichloromethane in a Soxhlet apparatus for 3 days. The combined extracts and filtrate are evaporated under reduced pressure (30 mm, 30°C). After drying over phosphorus pentoxide (P₄O₁₀) in an evacuated (1 mm) desiccator, 42.4 g (91%) of colorless crystals, mp 71–73°C, are obtained and used as such for step C. Recrystallization from dichloromethane (Note 10) gives 37.4 g (80%) of (+)-2-O-benzyl-L-threitol, mp 75–76°C (Note 13).

C. (–)-2-O-Benzyl-L-glyceraldehyde.³ A solution of 9.22 g (43.4 mmol) of (+)-2-O-benzyl-L-threitol (Note 14) in 100 mL of water is stirred vigorously, while 9.28 g (43.4 mmol) of sodium periodate (Note 15) is added in ca. 1-g portions over 40 min. The mixture is stirred for 2 hr at room temperature, then the pH is adjusted to 7.0 by addition of solid potassium carbonate (Note 16). The mixture is transferred to a 500-mL separatory funnel and extracted with dichloromethane (3 × 150 mL). The combined organic layers are dried over magnesium sulfate for 15 min and concentrated on a rotary evaporator (30 mm, 30°C). The remaining pale-yellow crude oil is transferred to a 25-mL round-bottomed flask and purified by short-path distillation in a preheated oil bath (0.025 mm, 160°C) (Note 17), to yield 6.32 g (80%) of (–)-2-O-benzyl-L-glyceraldehyde, a colorless oil that on standing turns more and more viscous, and after several weeks at room temperature forms a waxy solid (Note 18).

D. Ethyl (–)-[R-(E)]-4-O-benzyl-4,5-dihydroxy-2-pentenoate. To a 500-mL, round-bottomed, two-necked flask, fitted with a nitrogen inlet (Note 19) and a stopper, is added 3.32 g of a suspension of sodium hydride in paraffin, containing 65% sodium hydride. The suspension is washed three times with 40 mL and once with 20 mL of hexanes (Note 20) to remove the paraffin. The residue is freed from remaining hexanes under vacuum (0.01 mm) to give ca. 2.16 g (ca. 90 mmol) of sodium hydride. The flask is fitted with a magnetic stirring bar and the stopper is exchanged for a septum. Then 100 mL of tetrahydrofuran (Note 21) is added and the suspension is cooled to 0°C. Triethyl phosphonoacetate (22.87 g, 102 mmol) (Note 22) is added to the stirred sodium hydride/tetrahydrofuran suspension by means of a 50-mL syringe over a period of 20 min. The mixture is cooled to –78°C (acetone/dry ice) and a solution of 10.81 g (60 mmol) of (–)-2-O-benzyl-L-glyceraldehyde in 80 mL of tetrahydrofuran (Note 21) is added by means of a syringe over 20 min. (The checkers transferred this solution via a cannula.) The mixture is stirred for an additional 15 min at –78°C. The temperature is allowed to rise to 0°C within 30 min, and finally is kept at room temperature for an additional 45 min. The reaction is quenched with 150 mL of saturated ammonium chloride solution and extracted three times with ether (500, 200, 200 mL). The combined organic layers are washed with a mixture of saturated sodium bicarbonate/brine (1:1, 160 mL). The aqueous layer is washed with ether (3 × 100 mL). The combined organic layers are dried over magnesium sulfate, filtered, and evaporated to dryness to give a pale

yellow oil. The crude product is purified by column chromatography over silica gel (Note 23) with petroleum ether/ethyl acetate 1/1 as eluent (Note 24), to yield 11.7 g (78%) of analytically pure ethyl (-)-[R-(E)]-4-O-benzyl-4,5-dihydroxy-2-pentenoate (Note 25).

2. Notes

1. Diethyl L-tartrate (99%) was obtained from Janssen Chimica, Brüggen, Germany or Aldrich Chemical Company, Inc.
2. Benzaldehyde (99+%) from Aldrich Chemical Company, Inc., was used as received.
3. p-Toluenesulfonic acid monohydrate (99%) was obtained from Fluka Feinchemikalien GmbH, Neu-Ulm, Germany or from Aldrich Chemical Company, Inc.
4. The mixture becomes homogeneous at reflux temperature. The reaction usually takes about 16 hr at an 0.5 mole-scale as indicated by the amount of water separated.
5. Diethyl ether (technical grade) was distilled over potassium hydroxide.
6. The spectral properties of diethyl (-)-2,3-O-benzylidene-L-tartrate are as follows: ¹H NMR (250 MHz, CDCl₃) δ: 1.32, 1.35 (2 t, 6 H, J = 7.1, 2 CH₂CH₃), 4.23, 4.28 (2 q, 4 H, J = 7.1, 2 CH₂CH₃), 4.83, 4.95 (2 d, 2 H, J = 4.0, 2 CHO), 6.16 (s, 1 H, C₆H₅), 7.40, 7.58 (2 m, 5 H, C₆H₅), ⁴[α]_D²⁰ -30.7° (CHCl₃, c 2.20), ⁵mp 45°C.^{4,5}
7. Lithium aluminum hydride was obtained in 25-g samples (98%) from Merck-Schuchardt, Hohenbrunn, Germany or Aldrich Chemical Company, Inc.
8. Diethyl ether was dried first by distillation over potassium hydroxide, then by distillation from lithium aluminum hydride.
9. In order to dissolve aluminum chloride in dry diethyl ether, a flask is charged with the aluminum chloride and the diethyl ether is added in 10-mL portions with vigorous mechanical stirring to give a dark solution. The flask must be cooled in an ice bath.
10. Dichloromethane (technical grade) was distilled over phosphorus pentoxide.
11. If lithium aluminum hydride and aluminum chloride are not of high purity, an excess of 10% of each should be used. Otherwise a mixture with products of incomplete reduction is obtained.
12. The reaction mixture is heated to reflux with 200 mL of tetrahydrofuran; the precipitate then obtained is very easy to filter off.
13. The spectral properties of (+)-2-O-benzyl-L-threitol are as follows: ¹H NMR (250 MHz, CDCl₃) δ: 2.58, 2.76, 2.98 (bs, 3 H, 3 OH), 3.44–3.88 (m, 6 H, 2 CH₂OH, H-2, H-3), 4.56, 4.69 (AB, 2 H, J = 11.6, CH₂C₆H₅), 7.16–7.34 (m, 5 H, C₆H₅), ⁴[α]_D²⁵ +17.5° (EtOH, c 1.14).⁵
14. In several experiments it was found that the yield of (-)-2-O-benzyl-L-glyceraldehyde is somewhat lower when the reaction is performed on a larger scale.
15. Sodium periodate (98%) was obtained from Fluka Feinchemikalien GmbH, Neu-Ulm, Germany or from Fisher Scientific Company.
16. The pH was controlled with Merck Universal-Indikatorpapier or pHDrion Vivid 1–11 Jumbo pH paper, Micro Essential Laboratory, Brooklyn, NY, USA.
17. A silicone-oil bath is preheated to 160°C (the checkers used a sand bath). The flask of the evacuated apparatus filled with the crude product is immersed totally in the bath until no more distillate is collected. For optimum results, the distillation should be performed within 10 to 15 min. Slower distillation leads to lower yields because of thermal decomposition.
18. It is not possible to give exact spectral properties of (-)-2-O-benzyl-L-glyceraldehyde because of rapid di- and/or oligomerization. In order to check the optical purity of the product, it is convenient to compare the equilibrium value of specific rotation, as obtained after 6 days in ethanol solution at room temperature; ⁴[α]_D²² -33.2° (EtOH, c 0.083). In succeeding reactions (see Discussion and Step D), it was determined from NMR shift experiments that these products [e.g., (i) the Z-selective Wittig enoate product (with ethoxycarbonylmethylene-triphenylphosphorane) and the penten-5-olide therefrom (after acid-catalyzed lactonization,^{4,6}) and (ii) the E-enoate (Horner product)^{4,6}] contained enantiomers in ratios of >96:4.
19. Nitrogen was dried by means of a Sicapent® (E. Merck) drying tube. The checkers used argon.
20. The checkers used hexanes (technical grade) distilled over 3 Å molecular sieves. The submitters used pentane distilled from sodium.
21. Tetrahydrofuran was purified by distillation under nitrogen from a purple solution of sodium and benzophenone.
22. Triethyl phosphonoacetate was purified by distillation (bp 143°C, 9 mm). The checkers obtained this

compound (99%) from Aldrich Chemical Company, Inc., and used it as received.

23. The submitters used 28 g of Kieselgel 60, E. MERCK, 0.040–0.063 mm (250–400 mesh); column: 28 cm × 2.5 cm. The checkers used 100 g of silica gel, 0.032–0.063 mm, Selecto Scientific, Norcross, GA, USA, catalog # 162824; column: 40 cm × 5.5 cm.

24. Ethyl acetate and petroleum ether (technical grade; boiling range 40–80°C) were purified by distillation. The checkers used ethyl acetate/hexanes 10/90 followed by ethyl acetate/hexanes 35/65 as eluent; ethyl acetate (HPLC grade) was obtained from Mallinckrodt Specialty Chemicals Company, Paris, KY, USA and hexanes (technical grade) were distilled over 3 Å molecular sieves.

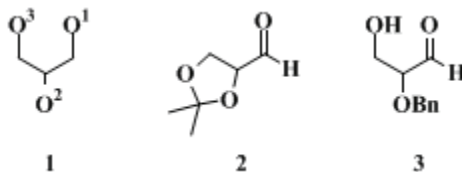
25. The analytical data (after chromatography) were as follows: Calcd. for C₁₄H₁₈O₄ (250.29): C, 67.18; H, 7.25. Found: C, 67.00; H, 7.20. The E/Z ratio was found to be >97:3 (determined by HPLC). Rt_E = 3.72 min; Rt_Z = 4.28 min, eluent petroleum ether/ethyl acetate 6/4 (LiChrosorb Si 60 column, E. Merck]. TLC: R_f = 0.34 (petroleum ether/ethyl acetate 1/1). GLC analysis: Column PS086/.32 mm × 20 m glass capillary, 95:5 methyl/phenylsilicone. Program: T₁, 40°C/(1 min), rate 10°C/min, T₂, 300°C, 0.5 bar hydrogen pressure; Rt_Z = 16.47 min; Rt_E = 17.25 min. E/Z ratio was found to be >97:3 (determined by GLC). [α]_D²² –75.8° (CHCl₃, c 1.192, E/Z >97:3), bp 125–130°C (0.001 mm). ¹³C NMR (63 MHz, CDCl₃) δ: 14.2 (OCH₂CH₃), 60.7 (OCH₂CH₃), 64.6 (C-5), 71.5 (CH₂Ph), 79.0 (C-4), 123.9 (C-2), 127.9, 128.0, 128.3, 128.6, 137.6 (C₆H₅), 144.3 (C-3), 165.9 (C-1); ¹H NMR (250 MHz, CDCl₃) δ: 1.30 (t, 3 H, J = 7.1, CH₃), 2.32 (dd, 1 H, J = 5.2, 7.9, OH), 3.65 (m, 2 H, CH₂OH), 4.14 (m, 1 H, 4-H), 4.22 (q, 2 H, J = 7.1, OCH₂CH₃), 4.41, 4.45 (AB, 2 H, J = 11.6, CH₂Ph), 6.11 (dd, 1 H, J = 1.3, 15.8, 2-H), 6.85 (dd, 1 H, J = 6.1, 15.8, 3-H), 7.34 (m, 5 H, C₆H₅).

Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995 and "Neue Datenblätter für gefährliche Arbeitsstoffe nach der Gefahrstoffverordnung", Welzbacher, U. (Ed.); WEKA Fachverlage, Kissing, 1991.

3. Discussion

Optically active C₃-building blocks of type **1** are key starting compounds in organic synthesis.^{7 8 9 10}
^{11 12} The most important member of this class is 2,3-O-isopropylidenglyceraldehyde **2**.^{7,8,9,10,11,12,13 14 15}



Both enantiomers of **2** are readily available from D-mannitol and from L-gulonolactone, respectively, and have been used in many reactions in a straightforward manner, because of the free aldehyde and the protected hydroxy functions. Unfortunately, **2** tends to trimerize and partial racemization has often been encountered on prolonged storage, thus preventing shipping/commercialization.¹⁶ A recent paper on large-scale preparation of glyceraldehyde acetone **2**, however, indicates suitable stability even at room temperature when certain precautions are met.^{17 18 19}

Glyceraldehyde derivatives with one protected and one free hydroxy function could, in principle offer new options because 2O/3O are more strongly differentiated, and further, since the free hydroxy group does or may cause different regio- and stereoselectivity in the next or subsequent steps. 2-O-Benzylglyceraldehyde, **3**, because of its di- or oligomeric form, is configurationally stable at room temperature.⁶ The D-form has previously been prepared from D-mannitol in four steps, with an overall yield of 5%,²⁰ or in 9 steps with 4%,²¹ and from D-glucose in six steps with an overall yield of 50%.²²

As described here, both enantiomers of **3** can be prepared in three steps from commercially available diethyl D- and L-tartrate in up to 70% over-all yield.^{2,3,4,6} Procedures to obtain the benzylidene acetal,^{23,24} with the ensuing reduction step,^{23,24} are based on previous literature reports. Both enantiomers of **3** have been used in highly stereoselective nitroaldol additions.^{4,25} Imines, nitrones, oximes, and nitrile oxides derived therefrom were recently employed in a variety of additions/cycloadditions.^{26 27 28,29}

³⁰ ³¹ ³² ³³ (-)-2-O-Benzyl-L-glyceraldehyde has further been used for the preparation of protected (2S,4R)-4-hydroxyornithine, via a Horner-Emmons reaction to the corresponding α,β -didehydroamino acid derivatives and subsequent diastereoselective hydrogenation.³⁴ Transformation of aldehyde **3** in Z-selective Wittig or E-selective Horner reactions^{4,6,35} (see Step D³⁶), formation of the dimethyl acetal^{5,37} or of the corresponding nitro compound by oxidation of the oxime,^{5,37} represent further recent uses of **3**.

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 10, 405](#)

References and Notes

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2. (+)-2-O-Benzyl-L-threitol is available from Fluka Feinchemikalien GmbH at ca. DM 60/g;
3. 2-O-Benzylglyceraldehyde is available from Merck-Schuchardt, D- and L- at a price of ca. DM 140/g.
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36. We thank Mr. Timo Gräther for experimental assistance concerning Step D.
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

petroleum ether

hexanes

brine

dimethyl acetal

glyceraldehyde acetonide

ETHYL (R,E)-4-O-BENZYL-4,5-DIHYDROXY-2-PENTENOATE

Diethyl (–)-2,3-O-benzylidene-L-tartrate

Ethyl (–)-[R-(E)]-4-O-benzyl-4,5-dihydroxy-2-pentenoate

sodium hydride in paraffin

penten-5-olide

diethyl D- and L-tartrate

benzylidene acetal

methylsilicone

phenylsilicone

ethanol (64-17-5)

potassium carbonate (584-08-7)

ethyl acetate (141-78-6)

ether,
diethyl ether (60-29-7)

ammonium chloride (12125-02-9)

sodium bicarbonate (144-55-8)

nitrogen (7727-37-9)

cyclohexane (110-82-7)

benzaldehyde (100-52-7)

acetone (67-64-1)

aluminum chloride (3495-54-3)

potassium hydroxide (1310-58-3)

Benzophenone (119-61-9)

sodium (13966-32-0)

Pentane (109-66-0)

d-Glucose (492-62-6)

dichloromethane (75-09-2)

magnesium sulfate (7487-88-9)

Tetrahydrofuran (109-99-9)

lithium aluminum hydride (16853-85-3)

sodium hydride (7646-69-7)

argon (7440-37-1)

potassium bicarbonate (298-14-6)

triethyl phosphonoacetate (867-13-0)

Sodium periodate (7790-28-5)

diethyl L-tartrate

ethoxycarbonylmethylene-triphenylphosphorane (1099-45-2)

phosphorus pentoxide (1314-56-3)

D-mannitol (69-65-8)

p-toluenesulfonic acid monohydrate (6192-52-5)

(-)-2-O-Benzyl-L-glyceraldehyde,
2-O-Benzylglyceraldehyde

Propanal, 3-hydroxy-2-(phenylmethoxy)-, (R)-

2-Pentenoic acid, 5-hydroxy-4-(phenylmethoxy)-, ethyl ester, [R-(E)]- (119770-84-2)

(+)-2-O-Benzyl-L-threitol

2,3-O-isopropylidenglyceraldehyde (22323-80-4)

L-gulonolactone

(2S,4R)-4-hydroxyornithine