Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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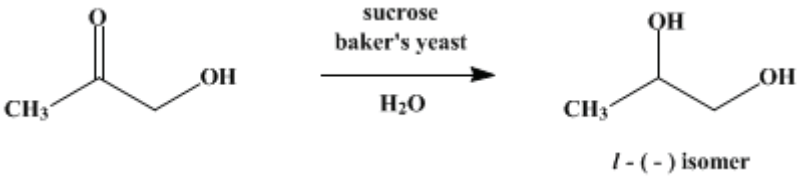
Organic Syntheses, Coll. Vol. 2, p.545 (1943); Vol. 10, p.84 (1930).

l-PROPYLENE GLYCOL

[1,2-Propanediol, *l*-]



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1. Procedure

A solution of 1 kg. of sucrose in 9 l. of water is placed in a 20-l. bottle provided with a gas trap. A paste of baker's yeast (Note 1) is made by breaking up 1 kg. of yeast and gradually stirring in 1 l. of water. This is then added to the sugar solution and the mixture is allowed to stand at room temperature until a lively evolution of gas starts (from one to three hours). To the vigorously fermenting solution 100 g. (1.35 moles) of freshly prepared acetol (p. 5) is added, and the mixture is allowed to stand at room temperature until the reaction subsides (Note 2). The bottle is then transferred to an incubator at 32°, when the fermentation recommences. At the end of three days the reaction is generally completed, and the solution when tested with Fehling's reagent gives a negligible test for reducing sugars.

At this point 20–30 g. of short glass fiber or asbestos is added and the yeast is filtered by suction. The filtrate is concentrated to a thick syrup under diminished pressure on a water bath, the temperature being kept below 40° (Note 3). The residue (about 200 cc.) is taken up in a mixture of 400 cc. of absolute alcohol and 100 cc. of dry ether (Note 4). The precipitate formed is removed by centrifuging, the supernatant liquid is decanted, and the residue is extracted with a mixture of 200 cc. of 98.5 per cent alcohol and 100 cc. of dry ether (Note 5). The combined alcohol-ether solutions are concentrated under diminished pressure at 35–40° to a thick syrup. The residue is again taken up in a mixture of 400 cc. of 98.5 per cent alcohol and 100 cc. of dry ether and centrifuged (Note 5). The supernatant liquid is concentrated under diminished pressure and distilled from a modified Claisen flask. The yield of the crude product boiling at 86–91°/12 mm. is approximately 100 g. The crude material is refractionated and collected at 88–90°/12 mm. or 187–189°/760 mm. The final product (Note 4) is a colorless liquid having a density 1.04 and specific rotation $[\alpha]_D^{20} = -15.0°$. The yield is 50–60 g. (49–58 per cent of the theoretical amount) (Note 6).

2. Notes

1. Fleischmann's yeast is satisfactory.
2. The addition of the acetol may cause the reaction to slacken for a time.
3. The evaporation must be carried out at as low a temperature as is practicable. A suitable device for this evaporation is given in Org. Syn. Coll. Vol. I, **1941**, 427.
4. The product reacts slightly acid. If an entirely neutral *l*-propylene glycol is desired, the syrup first obtained should be made neutral with a solution of sodium methoxide in methyl alcohol, and again concentrated and extracted as indicated.
5. If a centrifuge is not available the same result may be obtained by adding about 15 g. of short glass fiber or asbestos to the solution, stirring the solution mechanically or shaking it vigorously for five minutes, and filtering with suction.
6. The optically active glycols are convenient starting materials for the preparation of optically active carbinols, hydroxyacids, etc. The biological method of asymmetric reduction is perhaps the only convenient method for the preparation of these glycols. The steps in the preparation of other optically active glycols are identical with those in the preparation of *l*-propylene glycol from acetone. In the

synthesis of certain glycols it is convenient to prepare the chloroketone by oxidizing the corresponding chlorohydrin, the succeeding steps being the same as those in the synthesis of *l*-propylene glycol.

3. Discussion

l-Propylene glycol has been obtained from the optically inactive glycol by the action of bacteria,[1] and by means of strychnine compounds.[2] The present method is based on that of Färber, Nord, and Neuberg.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 478
- Org. Syn. Coll. Vol. 7, 356
- Org. Syn. Coll. Vol. 8, 332

References and Notes

1. LeBel, Jahresb. **1881**, 512.
2. Grün, Ber. **52**, 260 (1919).
3. Färber, Nord, and Neuberg, Biochem. Z. **112**, 313 (1920).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

Fehling's reagent

glycol

alcohol (64-17-5)

Acetol (64-19-7)

methyl alcohol (67-56-1)

ether (60-29-7)

sucrose

acetone (67-64-1)

sodium methoxide (124-41-4)

L-Propylene glycol,
1,2-Propanediol, l- (57-55-6)