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.

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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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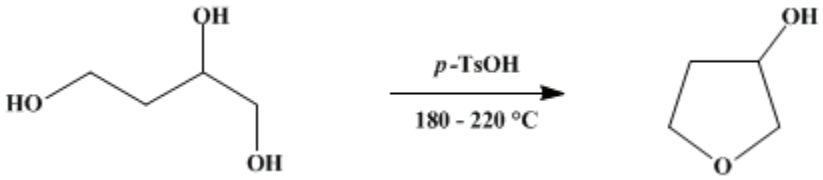
3-HYDROXYTETRAHYDROFURAN

[Furan, 3-hydroxy-1,2,3,4-tetrahydro-]



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1. Procedure

A 500-ml. flask is charged with 318 g. (3 moles) of 1,2,4-trihydroxybutane (Note 1) and 3 g. of *p*-toluenesulfonic acid monohydrate. A few Carborundum boiling chips are added, the flask is equipped with a 30.5-cm. Vigreux column, condenser, and receiver arranged for vacuum distillation, and the contents are heated, with swirling, to dissolve the acid (Note 2). The flask is then heated in a bath held at 180–220° so that 300–306 g. of distillate, b.p. 85–87°/22 mm., is collected over a period of 2–2.5 hours (Note 3). The colorless liquid obtained is refractionated, the same apparatus being used, and two fractions are collected: the first, 50–60 g., b.p. 42–44°/24 mm., n_D^{25} 1.3343, is mainly water. After a negligible intermediate fraction, 215–231 g. (81–88%) of pure 3-hydroxytetrahydrofuran, b.p. 93–95°/26 mm., n_D^{25} 1.4497, d_4^{20} = 1.095, is collected (Note 4).

2. Notes

1. Supplied by the General Aniline and Film Corporation.
2. Considerable darkening occurs even when the acid is well dispersed. The yield appears not to be affected.
3. Other temperatures are: b.p. 75–77°/16 mm.; 90–92°/28 mm. This first distillate contains 14% (±3%) of water as determined by interpolation of the refractive indices.
4. Calcd. M_D = 21.64. Found: 21.72. As obtained by this single fractionation the submitters found the alcohol to be analytically pure: Calcd. for $C_4H_8O_2$: C, 54.53; H, 9.14. Found: C, 54.74; H, 9.32. Others have reported: b.p. 50°/1 mm., n_D^{18} 1.4486, d_4^{20} = 1.090,[2] and b.p. 81°/13 mm., $d>^{18}$ = 1.07, n_D^{18} 1.4478.[3]

3. Discussion

3-Hydroxytetrahydrofuran has been obtained during the preparation of 1,2,4-trihydroxybutane,[3] by hydrolysis of 4-chloromethyl-1,3-dioxane[2] and by acid catalyzed dehydration of 1,2,4-trihydroxybutane.[4] The present procedure is similar to that described by Reppe.[4]

References and Notes

1. Tulane University, New Orleans, Louisiana.
2. Price and Krishnamurti, *J. Am. Chem. Soc.*, **72**, 5335 (1950).
3. Pariselle, *Ann. chim. (Paris)*, [8]**24**, 315 (1911).
4. Reppe, *Ann.*, **596**, 1 (1955), see p. 112; DBP 841 592 (1942), BASF (H. Krzikalla, E. Woldan).

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

Carborundum

3-Hydroxytetrahydrofuran,
Furan, 3-hydroxy-1,2,3,4-tetrahydro- (453-20-3)

1,2,4-trihydroxybutane (42890-76-6)

4-chloromethyl-1,3-dioxane

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