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.

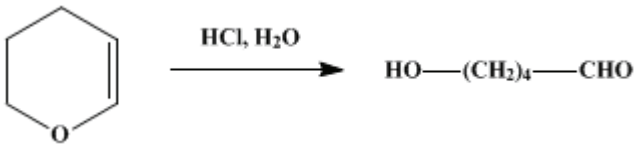
*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

DOI:10.15227/orgsyn.027.0043

*Organic Syntheses, Coll. Vol. 3, p.470 (1955); Vol. 27, p.43 (1947).*

# 5-HYDROXYPENTANAL

## [Valeraldehyde, δ-hydroxy-]




Submitted by G. Forrest Woods, Jr.
Checked by Cliff S. Hamilton and Wm. V. Ruyle.

## 1. Procedure

In a 1-l. three-necked flask provided with a Hershberg stirrer are mixed 300 ml. of water, 25 ml. of concentrated hydrochloric acid (Note 1), and 100 g. of 2,3-dihydropyran (p. 276). The mixture is stirred until the solution has become homogeneous and then for an additional 20 minutes (Note 2). After the addition of a few drops of phenolphthalein indicator to the mixture, the acid is neutralized with 20% sodium hydroxide; enough alkali is added so that a faint pink color just persists.

The solution is then transferred to a continuous extractor and extracted with ether for about 16 hours. The ether extract is added in convenient portions to a 250-ml. modified Claisen flask fitted with a condenser and a suitable fraction-cutter for distillation under reduced pressure. The ether is removed by distillation under the diminished pressure of a water pump, and the residue is then distilled at about 10 mm. pressure. After a small fore-run which weighs 2–5 g., the product distils as a clear, colorless, viscous oil at 62–66°/9–10 mm.; $n_D^{25}$ 1.4513. The yield is 90–95 g. (74–79%) (Note 3) and (Note 4).

## 2. Notes

1. The quantity of acid is arbitrary. This amount was chosen to minimize the time of hydration. The order of addition of the reactants should be that stated above. Less acid may be used with increased reaction time; more should not be used.
2. About 5–10 minutes is required before the solution becomes homogeneous. Some heat is evolved during the hydration. The submitter has found that the amount of 2,3-dihydropyran may be increased up to 300 g. without using more water or acid; however, if larger quantities of the pyran are used, the pyran must be added dropwise to the acid solution with cooling.
3. A convenient apparatus for the distillation is a modified Claisen flask whose side arm is provided with a short water-cooled condenser. A fraction-cutter of the "pig" type is satisfactory.
4. According to the submitter the product can be converted smoothly to pentamethylene glycol by hydrogenation over Raney nickel at 90° and 2000 lb. pressure. It will also undergo reductive alkylamination by a procedure similar to that described for 2-isopropylaminoethanol (p. 501).

## 3. Discussion

5-Hydroxypentanal has been prepared only by the method of Paul,[1] of which the above is an adaptation.

## References and Notes

**1.** Paul, *Bull. soc. chim. France*, (5), **1**, 976 (1934).

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

hydrochloric acid (7647-01-0)

ether (60-29-7)

sodium hydroxide (1310-73-2)

Raney nickel (7440-02-0)

pentamethylene glycol (111-29-5)

phenolphthalein (77-09-8)

2,3-Dihydropyran

5-Hydroxypentanal,
Valeraldehyde, δ-hydroxy- (4221-03-8)

2-isopropylaminoethanol (109-56-8)