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.

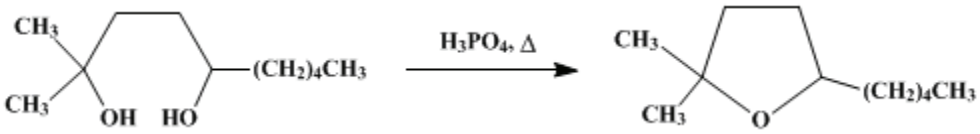
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.038.0025

Organic Syntheses, Coll. Vol. 4, p.350 (1963); Vol. 38, p.25 (1958).

5,5-DIMETHYL-2-*n*-PENTYLTETRAHYDROFURAN

[Furan, tetrahydro-2,2-dimethyl-5-pentyl-]



Submitted by J. Colonge and R. Marey[1].
Checked by V. Boekelheide and H. Kaempfen.

1. Procedure

In a 100-ml., three-necked, round-bottomed flask, fitted with a sealed mechanical stirrer, a reflux condenser, and a thermometer reaching to the bottom of the flask, are placed 37.6 g. (0.2 mole) of 2-methyl-2,5-decanediol (p.601) and 17 g. of 85% phosphoric acid. The limpid liquid obtained is heated and maintained at 125° for 40 minutes. Then the acidic lower layer is discarded, and the organic layer is washed with three or four 50-ml. portions of lukewarm distilled water.

Distillation of the resulting crude oil using a simple fractionating column gives 32–33 g. (94–97%) of pure 5,5-dimethyl-2-*n*-pentyltetrahydrofuran as a colorless liquid boiling at 31–33°/1.5 mm.; n_D^{25} 1.4257 (Note 1).

2. Notes

1. The submitters have also prepared 5,5-dimethyl-2-heptyltetrahydrofuran, n_D^{25} 1.4360, by a similar dehydration of 2-methyl-2,5-undecanediol obtained from the reaction of methylmagnesium bromide and γ-undecanoic acid lactone.

3. Discussion

There is no report on the preparation of 5,5-dimethyl-2-*n*-pentyltetrahydrofuran.

References and Notes

1. École de Chimie Industrielle de Lyon and Établissement Descollonges Frères (Lyon).

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

phosphoric acid (7664-38-2)

methylmagnesium bromide (75-16-1)

Furan, tetrahydro-2,2-dimethyl-5-pentyl-,
5,5-DIMETHYL-2-n-PENTYLTETRAHYDROFURAN (53684-53-0)

2-Methyl-2,5-decanediol (53731-34-3)

5,5-dimethyl-2-heptyltetrahydrofuran

2-methyl-2,5-undecanediol

γ-undecanoic acid lactone (104-67-6)