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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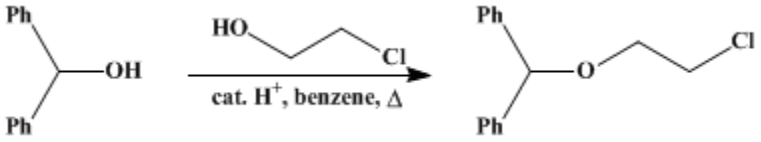
BENZHYDRYL β-CHLOROETHYL ETHER

[Ether, benzohydryl 2-chloroethyl]



Submitted by Shigehiko Sugasawa and Kunio Fujiwara[1].
Checked by N. J. Leonard, P. D. Thomas, and L. A. Miller.

1. Procedure

In a 500-ml. three-necked round-bottomed flask equipped with a sealed stirrer, a reflux condenser, and a dropping funnel are placed 36 g. (0.45 mole) of ethylene chlorohydrin (Note 1), 5 ml. of concentrated sulfuric acid, and 35 ml. of benzene. The mixture is warmed on a water bath, and to it is added, with efficient stirring, a solution of 55 g. (0.30 mole) of benzhydrol (Note 2) in 65 ml. of benzene (Note 3) during 30–50 minutes. The reaction mixture is heated at the reflux temperature for an additional 4 hours with stirring. To the cooled mixture is added about 35 ml. of benzene, and the combined benzene layer is washed with water and dried over calcium chloride. The drying agent is removed, the benzene is evaporated, and the residue is distilled under reduced pressure. The benzhydryl β-chloroethyl ether is collected at 144–148°/1.0 mm. (174–177°/4 mm.) as a colorless, viscous oil, n_D^{30} 1.5651, which should be removed from the receiver to a beaker or Erlenmeyer flask immediately after the distillation. The oil solidifies to a hard white mass, m.p. 27.4–27.8°, when kept in an ice chest (Note 4). The yield is 60.0–65.3 g. (81–88%).

2. Notes

1. Commercial ethylene chlorohydrin is dried over anhydrous sodium sulfate and distilled before use; b.p. 126–127°/743 mm. Excess is used to avoid the formation of dibenzhydryl ether as a by-product.
2. Eastman Kodak Company benzhydrol, m.p. 67–67.5°, can be used directly.
3. It is necessary to warm the mixture in order to complete the solution of benzhydrol in the benzene.
4. The checkers found this product to be analytically pure without recourse to further purification.

3. Discussion

This method is based on the process of the submitters.[2]

References and Notes

1. Pharmaceutical Institute, Medical Faculty, University of Tokyo, Tokyo, Japan.
2. Sugasawa and Fujiwara, *J. Pharm. Soc. Japan*, **71**, 365 (1951) [*C. A.*, **46**, 951*h* (1952)]; Jap. pat. 184,243 (Aug. 12, 1949).

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

Ether, benzohydryl 2-chloroethyl

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

Benzene (71-43-2)

sodium sulfate (7757-82-6)

ethylene chlorohydrin (107-07-3)

Benzhydryl β-chloroethyl ether (32669-06-0)

benzhydrol (91-01-0)

dibenzhydryl ether (574-42-5)