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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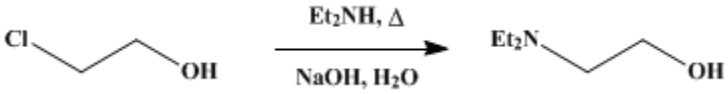
β-DIETHYLAMINOETHYL ALCOHOL

[Ethanol, 2-diethylamino-]



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1. Procedure

In a 2-l. flask provided with a reflux condenser and a dropping funnel is placed 380 g. (5.2 moles) of diethylamine (b.p. 52–60°). The diethylamine is heated to boiling over a steam bath, and 320 g. (4 moles) of ethylene chlorohydrin is added from the dropping funnel during the course of about one hour. Heating is then continued for eight hours more. The reaction mixture is allowed to cool, and a solution of 230 g. of sodium hydroxide in 350 cc. of water is added fairly rapidly with constant shaking. Two layers form immediately, and sodium chloride is precipitated. The latter is dissolved by the addition of 400 cc. of water, and then 500 cc. of benzene is added and the mixture is stirred mechanically for five minutes. The benzene layer is separated and the aqueous layer is extracted three times more, 500 cc. of benzene being used for each extraction. The combined benzene extracts are dried over about 100 g. of solid potassium carbonate, with mechanical stirring until the turbidity of the solution has disappeared. The solution is distilled from a 3-l. flask provided with a 50-cm. column (packed with glass or Carborundum) and a thermometer dipping in the liquid. Distillation is continued until the temperature of the liquid reaches 100° and that at the top of the column is 85°. The residue is transferred to a 1-l. Claisen flask having a 30-cm. column, and is distilled under reduced pressure. Cuts are taken at 45°/20 mm., 45–64°/18 mm., and 64–65°/18 mm. The last fraction amounts to about 290 g. The first two fractions are redistilled and more β-diethylaminoethyl alcohol is obtained (Note 1). The total yield is 320–330 g. (68–70 per cent of the theoretical amount).

2. Notes

1. The physical properties of β-diethylaminoethyl alcohol are described in detail by Headlee, Collett, and Lazzell.[1]

3. Discussion

β-Diethylaminoethyl alcohol has been prepared by reduction of diethylaminoacetic ester with sodium and alcohol;[2] by the action of ethylene chlorohydrin on diethylamine;[3] by the action of ethylene oxide on diethylamine;[1] and from ethanolamine and ethyl sulfate.[4]

References and Notes

1. Horne and Shriner, J. Am. Chem. Soc. **54**, 2928 (1932); Headlee, Collett, and Lazzell, ibid. **55**, 1066 (1933).
2. Gault, Compt. rend. **145**, 126 (1907); Bull. soc. chim. (4) **3**, 369 (1908).
3. Ladenburg, Ber. **14**, 1878 (1881); Soderman and Johnson, J. Am. Chem. Soc. **47**, 1394 (1925).
4. Carbide and Carbon Chemicals Corporation, Fr. pat. 792,046 [C. A. **30**, 4176 (1936)].

Appendix

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

diethylaminoacetic ester

alcohol (64-17-5)

potassium carbonate (584-08-7)

Benzene (71-43-2)

sodium hydroxide (1310-73-2)

sodium chloride (7647-14-5)

sodium (13966-32-0)

Ethylene oxide (75-21-8)

ethylene chlorohydrin (107-07-3)

diethylamine (109-89-7)

ethanolamine (141-43-5)

β-Diethylaminoethyl alcohol,
Ethanol, 2-diethylamino- (100-37-8)

ethyl sulfate