



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.

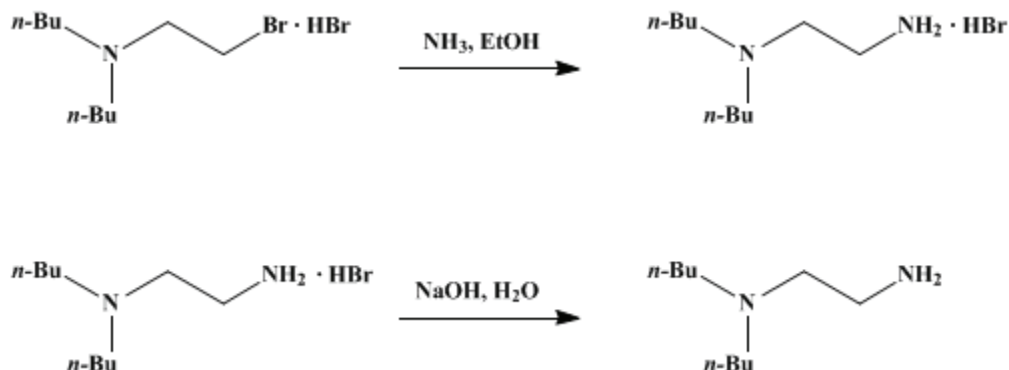
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.254 (1955); Vol. 23, p.23 (1943).

β -DI-*n*-BUTYLAMINOETHYLAMINE

[Ethylenediamine, N,N-dibutyl-]



Submitted by Lawrence H. Amundsen, Karl W. Krantz, and James J. Sanderson.
Checked by R. L. Shriner and John L. Rendall.

1. Procedure

A rubber tube from an inverted cylinder of liquid ammonia is attached to a glass tube which extends beneath the surface of 325 ml. of 90% ethanol in a 500-ml. Erlenmeyer flask. Ammonia is passed into the ethanol until the weight increases by 41 g. (2.4 moles). The flask is cooled in ice water occasionally in order to hasten the absorption of the ammonia.

The ethanolic ammonia is added to 49 g. (0.16 mole) of di-*n*-butylaminoethyl bromide hydrobromide [*Org. Syntheses Coll. Vol. 2, 92 (1943)*] (Note 1) in a 1-l. round-bottomed flask. The flask is stoppered tightly and allowed to stand for 6 days at room temperature. The solution is then transferred to an evaporating dish and stirred while it is evaporated to a crystalline paste under a hood. The crystalline paste is transferred to a 400-ml. beaker, and to it is added a solution of 16 g. of sodium hydroxide in 18 ml. of water. The solution separates into an upper layer of brown oil and a lower layer which contains a considerable amount of undissolved sodium bromide. About 10 ml. of water is added to dissolve most of the precipitate. The whole is then extracted with three 50-ml. portions of benzene. The combined extracts are placed in a 250-ml. flask over 15 g. of potassium carbonate and set aside to dry overnight.

The dried benzene extract is placed in a 250-ml. flask fitted with a 30-cm. fractionating column, and the benzene is removed by distillation at atmospheric pressure. Fractionation of the residue is carried out under reduced pressure using a 50-ml. Claisen flask with a built-in 12- to 15-cm. Vigreux column. The fraction boiling at 100–103°/13 mm. is collected; it weighs 11 to 14.7 g. (41–55%) (Note 2).

2. Notes

1. It is not necessary to isolate the di-*n*-butylaminoethyl bromide hydrobromide. The crude di-*n*-butylaminoethyl bromide hydrobromide prepared from 405 g. (2.34 moles) of di-*n*-butylaminoethanol is taken up in 600 ml. of water, and the tarry impurities are removed by extraction with three 200-ml. portions of benzene. About 400 ml. of water is evaporated from this solution, and to it is then added 2.8 l. of ethanolic ammonia prepared from absolute ethanol; the concentration of the ethanol in the reaction mixture should be 90% with respect to water. The yield is 114 g. (28% based on 405 g. of the aminoethanol). The over-all yield obtained by the procedure described above is 27% based on the aminoethanol.

2. The submitter reports that diethylaminoethylamine and di-*n*-propylaminoethylamine have also been prepared by this method.

3. Discussion

Di-*n*-butylaminoethylamine has been prepared by the action of ethanolic ammonia on di-*n*-butylaminoethyl bromide hydrobromide,¹ and by the reduction of di-*n*-butylaminoacetonitrile with sodium in butanol.²

References and Notes

1. Amundsen and Krantz, *J. Am. Chem. Soc.*, **63**, 305 (1941).
 2. Bloom, Breslow, and Hauser, *J. Am. Chem. Soc.*, **67**, 539 (1945).
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Appendix

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

ethanol (64-17-5)

potassium carbonate (584-08-7)

ammonia (7664-41-7)

Benzene (71-43-2)

sodium hydroxide (1310-73-2)

sodium bromide (7647-15-6)

butanol (71-36-3)

sodium (13966-32-0)

aminoethanol (141-43-5)

Ethylenediamine, N,N-dibutyl-,
 β -DI-*n*-BUTYLAMINOETHYLAMINE,
Di-*n*-butylaminoethylamine (3529-09-7)

diethylaminoethylamine (100-36-7)

di-*n*-butylaminoethyl bromide hydrobromide

di-*n*-butylaminoethanol (102-81-8)

di-*n*-propylaminoethylamine

di-*n*-butylaminoacetonitrile