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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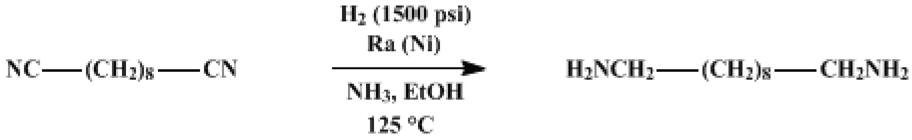
DECAMETHYLENEDIAMINE

[1,10-Decanediamine]



Submitted by B. S. Biggs and W. S. Bishop.
Checked by C. F. H. Allen and John R. Byers, Jr..

1. Procedure

A high-pressure bomb of about 1.1-l. capacity is charged with 82 g. (0.50 mole) of sebaconitrile and about 6 g. of Raney nickel catalyst (Note 1) suspended in 25 ml. of 95% ethanol, an additional 25 ml. of ethanol being used to rinse in the catalyst. The bomb is closed (Note 2), and about 68 g. (4 moles) of liquid ammonia is introduced from a tared 5-lb. commercial cylinder (Note 3). Hydrogen is then admitted at tank pressure (1500 lb.), and the temperature is raised to 125°. The reaction starts at about 90° and proceeds rapidly at 110–125°. When hydrogen is no longer absorbed (1–2 hours) the heater is shut off and the bomb allowed to cool. The hydrogen and ammonia are allowed to escape, and the contents of the bomb are rinsed out with two 100-ml. portions of 95% ethanol. The ethanolic solution is filtered quickly through a layer of decolorizing carbon (Note 4) to remove the catalyst and transferred to a 500-ml. Claisen flask having a modified side arm and connected by ground-glass joints to a receiver (Note 5). The ethanol is removed by distillation at atmospheric pressure, the receiver is changed, and the decamethylenediamine is distilled under reduced pressure. It boils at 143–146°/14 mm. (Note 6) and solidifies, on cooling, to a white solid, freezing point 60°. The yield is 68–69 g. (79–80%) (Note 7), (Note 8), and (Note 9).

2. Notes

1. Raney nickel catalyst, already prepared and suspended in water, can be obtained from the Gilman Paint and Varnish Company, Chattanooga, Tennessee.
2. The safety disk should be made of steel, nickel, or other suitable material. It must not be of copper, which is readily attacked by ammonia under pressure.
3. The ammonia may be introduced by a number of methods. A suitable one is given under α-phenylethylamine (p. 717). The amount of ammonia is not critical, but maximum yields are obtained when 6–8 moles is used per mole of dinitrile. The purpose of the ammonia is to suppress secondary amine formation.[1,2]
4. A 9-cm. Büchner funnel is used. The decolorizing carbon is deposited from a slurry in ethanol.
5. As decamethylenediamine combines with atmospheric carbon dioxide rapidly, any solutions left standing should be protected by a drying tube containing solid potassium hydroxide. When air is admitted to the apparatus at the end of the distillation it should be through such a tube.
6. Decamethylenediamine should not be allowed to solidify in a bottle or Erlenmeyer flask, since it will probably break such a container. A tared round-bottomed flask less than half filled is advisable.
7. A dermatitis is induced in susceptible individuals by decamethylenediamine.
8. The submitters reported yields of 85–90% on runs four times this size.
9. Other boiling points are 139–140°/12 mm.; 126–127°/5 mm.

3. Discussion

Decamethylenediamine has been obtained by reduction of sebaconitrile either catalytically[2] or by sodium and ethanol,[3] or by lithium aluminum hydride.[4] It has also been obtained by hydrolysis of the condensation product from decamethylene iodide and phthalimide,[5] and from decamethylene glycol in the presence of Raney nickel and ammonia at 220–260°.[6]

References and Notes

1. Brit. pat. 490,922 [*C. A.*, **33**, 993 (1939)].
2. Schwoegler and Adkins, *J. Am. Chem. Soc.*, **61**, 3499 (1939).
3. Phookan and Krafft, *Ber.*, **25**, 2253 (1892).
4. Nystrom and Brown, *J. Am. Chem. Soc.*, **70**, 3738 (1948).
5. von Braun, *Ber.*, **42**, 4551 (1909).
6. U. S. pat. 2,412,209 [*C. A.*, **41**, 1237 (1947)].

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

ethanol (64-17-5)

ammonia (7664-41-7)

hydrogen (1333-74-0)

carbon dioxide (124-38-9)

Raney nickel (7440-02-0)

decolorizing carbon (7782-42-5)

potassium hydroxide (1310-58-3)

sodium (13966-32-0)

Phthalimide (85-41-6)

Decamethylene glycol (112-47-0)

α-Phenylethylamine (3886-69-9)

lithium aluminum hydride (16853-85-3)

Decamethylenediamine,
1,10-Decanediamine (646-25-3)

Sebaconitrile (1871-96-1)

decamethylene iodide (16355-92-3)