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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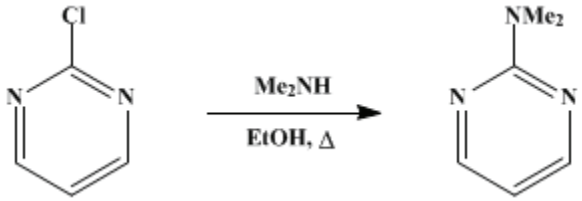
2-(DIMETHYLAMINO)PYRIMIDINE

[Pyrimidine,2-dimethylamino-]



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Checked by Charles C. Price and T. L. V. Ulbricht.

1. Procedure

In a 250-ml. three-necked flask equipped with a reflux condenser and a gas-inlet tube are placed 45.6 g. (0.4 mole) of 2-chloropyrimidine (p. 182) and 150 ml. of absolute ethanol. The mixture is refluxed for 6 hours while anhydrous dimethylamine is bubbled into the solution (Note 1). The solution is cooled, and 100 ml. of ethanol is removed by distillation using a water aspirator. The residue is chilled in an ice bath for 1 hour, and 75 ml. of ether is added to cause precipitation of dimethylamine hydrochloride. After the removal of dimethylamine hydrochloride and solvent, the residue is distilled at reduced pressure from a Claisen flask (Note 2). The fraction boiling at 85–86°/28 mm. is collected; yield 40–42.5 g. (81–86%), n_D^{25} 1.5420 (Note 3).

2. Notes

1. Anhydrous dimethylamine may be conveniently prepared by allowing 25% aqueous dimethylamine to drop onto solid potassium hydroxide, the gas evolved being dried by passage over solid potassium hydroxide.
2. The compound is hygroscopic, and care should be taken to prevent exposure to air.
3. N-Methylaminopyrimidine is similarly prepared; b.p. 96–98°/28 mm., m.p. 57.5–58.5° (65% yield).

3. Discussion

Similar procedures for this preparation have been reported by Brown and Short[2] and by Copenhaver and Kleinschmidt.[3]

References and Notes

1. Polytechnic Institute of Brooklyn, Brooklyn 2, New York.
2. Brown and Short, *J. Chem. Soc.*, **1953**, 331.
3. Copenhaver and Kleinschmidt, Brit. pat. 663,303 [*C. A.*, **46**, 10212 (1952)].

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

ethanol (64-17-5)

ether (60-29-7)

potassium hydroxide (1310-58-3)

dimethylamine (124-40-3)

dimethylamine hydrochloride (506-59-2)

2-Chloropyrimidine (1722-12-9)

2-(Dimethylamino)pyrimidine,
Pyrimidine,2-dimethylamino- (5621-02-3)

N-Methylaminopyrimidine