



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.

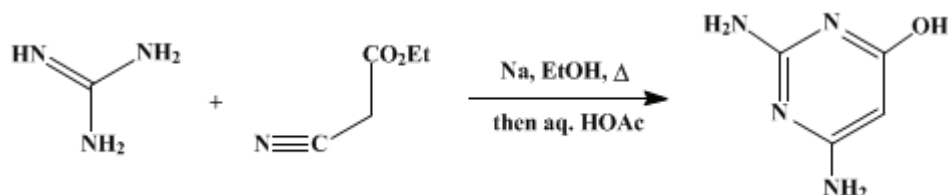
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.245 (1963); Vol. 32, p.45 (1952).

2,4-DIAMINO-6-HYDROXYPYRIMIDINE

[4-Pyrimidinol, 2,6-diamino-]



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1. Procedure

A solution of sodium ethoxide is prepared from 23 g. (1 g. atom) of sodium and 250 ml. of anhydrous ethanol in a 1-l. round-bottomed flask fitted with a reflux condenser carrying a calcium chloride drying tube. After the sodium has dissolved, the solution is cooled and 113 g. (1 mole) of ethyl cyanoacetate is added (Note 1). This mixture is allowed to stand while a second solution of sodium ethoxide of the same volume and concentration is prepared. To this solution is added 97 g. (1.02 moles) of guanidine hydrochloride. The sodium chloride is separated by filtration, and the clear filtrate containing guanidine is added to the solution of ethyl sodiocyanoacetate. This mixture is heated for 2 hours under reflux and is then evaporated to dryness at atmospheric pressure. The solid product is dissolved in 325 ml. of boiling water and acidified with 67 ml. of glacial acetic acid. Upon cooling of the solution, 101–103 g. (80–82%) of yellow needles separates; m.p. 260–270° (dec.) (Note 2).

2. Notes

1. Eastman Kodak Company white label grade of ethyl cyanoacetate was used by the checkers.
2. Analyses carried out by the checkers showed that the product is quite pure.

3. Discussion

This procedure is a modification of the method of Traube,² which has been studied by Berezovskii and Strel'chunas.³ The reaction has been carried out in the presence of alkali or alkali earth hydroxides.⁴

References and Notes

1. Eastman Kodak Company, Rochester, New York.
 2. Traube, *Ber.*, **33**, 1371 (1900); Ger. pat. 135,371 [*Frdl.*, **6**, 1192 (1900–1902)].
 3. Berezovskii and Strel'chunas, *Trudy Vsesoyuz. Nauch. Issledovatel. Vitamin Inst.*, **5**, 28 (1954) [*C. A.*, **51**, 7379 (1957)].
 4. Heinrich and Buth (to Wickhen Products Inc.), U. S. pat. 2,673,204 [*C. A.*, **49**, 1825 (1955)]; Sugino, Jap. pat. 2733 (1953) [*C. A.*, **49**, 2527 (1955)].
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

ethanol (64-17-5)

acetic acid (64-19-7)

sodium chloride (7647-14-5)

sodium (13966-32-0)

sodium ethoxide (141-52-6)

Ethyl cyanoacetate (105-56-6)

guanidine (113-00-8)

2,4-Diamino-6-hydroxypyrimidine,
4-Pyrimidinol, 2,6-diamino- (56-06-4)

guanidine hydrochloride (50-01-1)

ethyl sodiocyanoacetate