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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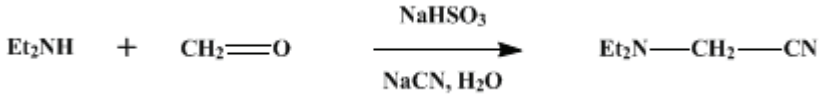
DIETHYLAMINOACETONITRILE

[Acetonitrile, diethylamino-]



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1. Procedure

This preparation should be carried out under a good hood since poisonous hydrogen cyanide may be evolved.

To a solution of 312 g. (3 moles) of sodium bisulfite in 750 ml. of water in a 3-l. beaker is added 225 ml. of a 37–40% formaldehyde solution, and the mixture is warmed to 60°. After cooling to 35°, 219 g. (309 ml., 3 moles) of diethylamine is added with hand stirring, and the mixture is allowed to stand for 2 hours. The beaker containing the reaction mixture is placed under a good hood, and to it is added a solution of 147 g. (3 moles) of sodium cyanide dissolved in 400 ml. of water with efficient stirring so that the two layers are thoroughly mixed. After 1.5 hours the upper nitrile layer is separated and dried over 25 g. of Drierite; it weighs 299–309 g. (90–92%). The crude product is purified by distillation; the portion boiling at 61–63°/14 mm., n_D^{25} 1.4230, amounts to 298–302 g. (88–90%) (Note 1).

2. Notes

1. Higher homologs have been prepared from other aldehydes.

3. Discussion

This procedure is essentially that recorded in the literature.[1]

References and Notes

1. Knoevenagel and Mercklin, *Ber.*, **37**, 4089 (1904).

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

Drierite

formaldehyde (50-00-0)

sodium cyanide (143-33-9)

hydrogen cyanide (74-90-8)

sodium bisulfite (7631-90-5)

diethylamine (109-89-7)

Diethylaminoacetonitrile,
Acetonitrile, diethylamino- (3010-02-4)