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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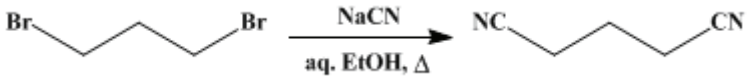
Organic Syntheses, Coll. Vol. 1, p.536 (1941); Vol. 5, p.103 (1925).

TRIMETHYLENE CYANIDE

[Glutaronitrile]



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1. Procedure

In a 5-l. round-bottomed flask fitted with a stopper holding a reflux condenser and a separatory funnel are placed 294 g. (6 moles) of sodium cyanide and 300 cc. of water. The flask is heated on a steam bath until most of the sodium cyanide is in solution. This requires two to three hours. A solution of 500 g. (2.47 moles) of trimethylene bromide (Note 1) in 1 l. of 95 per cent alcohol is then added through the separatory funnel over a period of forty to sixty minutes. The mixture is refluxed for thirty to forty hours (Note 2) on a steam bath. Then the solvent is removed, preferably under reduced pressure, using an oil bath.

The residue, consisting of sodium bromide, sodium cyanide, and trimethylene cyanide, is extracted with 300–400 cc. of ethyl acetate, which dissolves the trimethylene cyanide and does not dissolve the inorganic salts. This solution is filtered and the salt washed once with about 100 cc. of ethyl acetate. The ethyl acetate is distilled at ordinary pressure (Note 3) and the residual liquid is distilled under reduced pressure. The yield of trimethylene cyanide boiling at 144–147°/13 mm. or 131–134°/10 mm. is 180–200 g. (77–86 per cent of the theoretical amount).

2. Notes

1. Larger runs seem to give slightly lower yields.
2. The yield is lower if the heating is continued too long, owing to partial hydrolysis of the cyanide.
3. Very little ethyl acetate is lost in this procedure if it is distilled at ordinary pressures. If it is not entirely removed before the pressure is reduced, considerable foaming occurs.

3. Discussion

Trimethylene cyanide can be prepared by the action of potassium cyanide on trimethylene bromide.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 289

References and Notes

1. Reboul, Compt. rend. **82**, 1197 (1876); Henry, ibid. **100**, 742 (1885), Bull. soc. chim. (2) **43**, 617 (1885); Perkin, J. Chem. Soc. **55**, 702 (1889).

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

alcohol (64-17-5)

ethyl acetate (141-78-6)

sodium cyanide (143-33-9)

sodium bromide (7647-15-6)

Trimethylene bromide (109-64-8)

cyanide (57-12-5)

potassium cyanide (151-50-8)

Trimethylene cyanide,
Glutaronitrile (544-13-8)