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.

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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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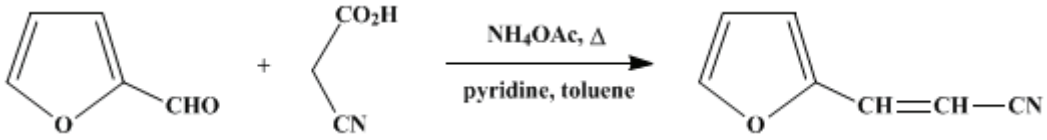
3-(2-FURYL)ACRYLONITRILE

[2-Furanacrylonitrile]



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1. Procedure

A mixture of 105.6 g. (1.1 moles) of freshly distilled furfural, 87.0 g. (1.0 mole) of 98% cyanoacetic acid (Note 1), 3.0 g. of ammonium acetate, 200 ml. of toluene, and 110 ml. of pyridine is placed in a 1-l. round-bottomed flask equipped with a Stark and Dean water trap and reflux condenser. The mixture is boiled under reflux for 2 days. The theoretical quantity of water is collected in the trap within 1 hour. Upon completion of the reflux period, the solvent is removed under reduced pressure by heating on a water bath. The residue, distilled through a 15-cm. Vigreux column at 11 mm. pressure, yields 88.6–93.3 g. (74.5–78%) of colorless liquid boiling at 95–97°, n_D^{25} 1.5823–1.5825.

2. Notes

1. Cyanoacetic acid was obtained from Distillation Products Industries, Rochester, New York, and used without further purification.

3. Discussion

The method described is a modification of the procedure used by Ghosez[2] to synthesize cinnamonitrile. 3-(2-Furyl)acrylonitrile has been prepared by catalytic condensation of furfural with acetonitrile in the vapor phase at 320°,[3] by dehydration of the corresponding amide over phosphorus pentachloride,[4] and by decarboxylation of 3-(2-furyl)-2-cyanoacrylic acid.[5]

References and Notes

1. University of Kentucky, Lexington, Kentucky.
2. J. Ghosez, *Bull. Soc. Chim. Belges.*, **41**, 477 (1932).
3. M. M. Brubaker, U. S. pat. 2,341,016 (1944) [*C. A.*, **38**, 4272 (1944)].
4. H. Gilman and A. P. Hewlett, *Iowa State Coll. J. Sci.*, **4** (1), 27 (1929).
5. R. Heuck, *Ber.*, **27**, 2624 (1894).

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

ammonium acetate (631-61-8)

acetonitrile (75-05-8)

phosphorus pentachloride (10026-13-8)

pyridine (110-86-1)

toluene (108-88-3)

cyanoacetic acid (372-09-8)

Furfural (98-01-1)

3-(2-Furyl)acrylonitrile,
2-Furanacrylonitrile (7187-01-1)

Cinnamonitrile (4360-47-8)

3-(2-furyl)-2-cyanoacrylic acid