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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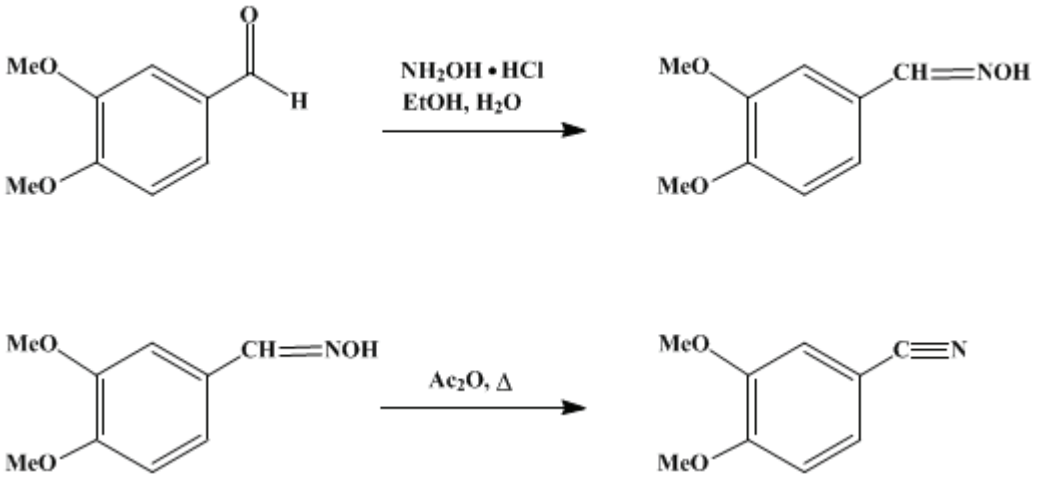
Organic Syntheses, Coll. Vol. 2, p.622 (1943); Vol. 15, p.85 (1935).

VERATRONITRILE



Submitted by J. S. Buck and W. S. Ide.
Checked by John R. Johnson and E. Amstutz.

1. Procedure

In a 1-l. round-bottomed flask, 83 g. (0.5 mole) of veratraldehyde (p. 619) is dissolved in 200 cc. of warm 95 per cent alcohol, and a warm solution of 42 g. (0.6 mole) of hydroxylamine hydrochloride (Org. Syn. Coll. Vol. I, **1941**, 318) in 50 cc. of water is added. The two solutions are mixed thoroughly, and a solution of 30 g. (0.75 mole) of sodium hydroxide in 40 cc. of water is introduced. After the mixture has stood for two and one-half hours at room temperature, 250 g. of crushed ice is added and the solution is saturated with carbon dioxide. This causes the separation of the aldoxime as an oil which solidifies on standing overnight in an ice chest (Note 1). The crystalline oxime is filtered with suction, washed thoroughly with water, and allowed to dry in the air. The yield of oxime is 88–89 g. (97–98 per cent of the theoretical amount).

The veratraldoxime is placed with 100 g. of acetic anhydride (94–96 per cent) in a 300-cc. round-bottomed flask provided with a ground-glass air condenser (Note 2), and heated cautiously. A vigorous reaction takes place, and when this occurs the flame is removed. After the reaction has subsided the solution is boiled gently for twenty minutes and then poured carefully, with stirring, into 300 cc. of cold water. The stirring is continued, and on cooling the nitrile separates in small, almost colorless crystals, which are filtered and dried in the air. The veratronitrile thus obtained is quite pure; it weighs 57–62 g. (70–76 per cent of the theoretical amount, based upon the veratraldehyde), and melts at 66–67°.

2. Notes

1. Occasionally the oxime does not solidify after standing overnight. If this happens it is advisable to separate the oily layer, treat with crushed ice, and induce crystallization by scratching. If a seed crystal of the oxime is available no difficulty is experienced.
2. If a cork or rubber stopper is used the product is likely to be colored.

3. Discussion

Veratronitrile has been obtained from 4-aminoveratrole by diazotization and treatment with cuprous cyanide,[1] and by heating veratrylglyoxylic acid with hydroxylamine.[2] The general method employed above has been used for the preparation of a variety of substituted aromatic nitriles.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 44
- Org. Syn. Coll. Vol. 2, 619

References and Notes

1. Moureu, Bull. soc. chim. (3) **15**, 650 (1896).
2. Garelli, Gazz. chim. ital. **20**, 700 (1890).
3. Marcus, Ber. **24**, 3650 (1891); Pschorr, Ann. **391**, 33 (1912).

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

alcohol (64-17-5)

acetic anhydride (108-24-7)

sodium hydroxide (1310-73-2)

Cuprous Cyanide (544-92-3)

carbon dioxide (124-38-9)

Hydroxylamine hydrochloride (5470-11-1)

hydroxylamine (7803-49-8)

4-Aminoveratrole (6315-89-5)

Veratronitrile (2024-83-1)

Veratraldehyde (120-14-9)

veratrylglyoxylic acid (2460-33-5)

veratraldoxime