



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.

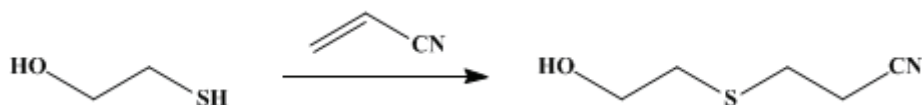
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.458 (1955); Vol. 29, p.52 (1949).

β-(2-HYDROXYETHYLMERCAPTO)PROPIONITRILE

[Propionitrile, β-(2-hydroxyethylmercapto)-]



Submitted by Leon L. Gershbein

Checked by Charles D. Hurd, Cliff S. Hamilton, and John A. Stephens.

1. Procedure

In a 500-ml. three-necked round-bottomed flask equipped with a sealed stirrer, a reflux condenser, a dropping funnel, and a thermometer is placed 78 g. (70 ml., 1 mole) of **2-mercaptoethanol** (Note 1). Into the dropping funnel is poured 67 ml. (54.3 g., 1 mole) of **acrylonitrile** (Note 2), and after the addition of about 3 ml. of the nitrile, with stirring, the contents are warmed with a water bath to about 35–40° for 5 minutes. The remainder of the **acrylonitrile** is then added dropwise during 10 minutes. The temperature soon mounts to about 65° and is kept between 55° and 60° by intermittent short cooling with water until it only slowly increases or remains stationary at 55–60° (Note 3). Forty milliliters of **acrylonitrile** is then added all at once, cooling being applied if necessary, and the contents are stirred for 16 hours at room temperature. The product is distilled from a 250-ml. Claisen flask after removal of excess **acrylonitrile** under reduced pressure. The yield of nitrile distilling at 178–180°/14 mm., n_D^{25} 1.5101, as a colorless viscous liquid is 121–123 g. (92–94%) (Note 4).

2. Notes

1. The **2-mercaptoethanol** was obtained from Carbide and Carbon Chemicals Corporation.
2. Commercial **acrylonitrile** may be used without further purification.
3. This requires about 30 minutes. As an inhibition period generally occurs, care must be taken in the initiation of the reaction and subsequent moderation of the heat evolved, but this operation can easily be controlled.
4. In the presence of alcoholic **sodium hydroxide**, either **2-mercaptoethanol** or **β-(2-hydroxyethylmercapto) propionitrile** is converted to the dicyanoethylated product, **4-oxa-7-thiadecanedinitrile**, $\text{NCCH}_2\text{-CH}_2\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN}$. This basic agent can also be applied to the general reaction of thiophenols or mercaptans with **acrylonitrile**.

3. Discussion

This method is a modification of the directions of Hurd and Gershbein.¹ The compound has been made also² with **piperidine** as the basic catalyst.

References and Notes

1. Hurd and Gershbein, *J. Am. Chem. Soc.*, **69**, 2331 (1947).
 2. Gribbins, Miller, and O'Leary, U. S. pat. 2,397,960 [*C. A.*, **40**, 3542 (1946)].
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sodium hydroxide (1310-73-2)

piperidine (110-89-4)

acrylonitrile (107-13-1)

β -(2-Hydroxyethylmercapto)propionitrile,
Propionitrile, β -(2-hydroxyethylmercapto)-,
 β -(2-hydroxyethylmercapto) propionitrile (15771-37-6)

2-mercaptoethanol (60-24-2)

4-oxa-7-thiadecanedinitrile