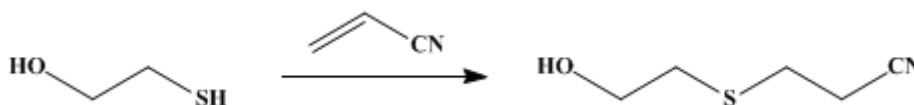


## $\beta$ -(2-HYDROXYETHYLMERCAPTO)PROPIONITRILE

### [Propionitrile, $\beta$ -(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  $\beta$ -(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.<sup>1</sup> The compound has been made also<sup>2</sup> with piperidine as the basic catalyst.

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### 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)

$\beta$ -(2-Hydroxyethylmercapto)propionitrile,  
Propionitrile,  $\beta$ -(2-hydroxyethylmercapto)-,  
 $\beta$ -(2-hydroxyethylmercapto) propionitrile (15771-37-6)

2-mercaptoethanol (60-24-2)

4-oxa-7-thiadecanedinitrile