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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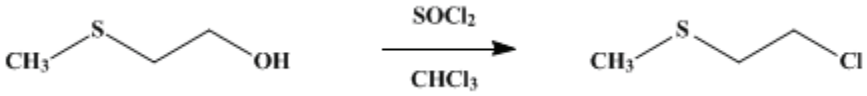
Organic Syntheses, Coll. Vol. 2, p.136 (1943); Vol. 14, p.18 (1934).

β-CHLOROETHYL METHYL SULFIDE

[Sulfide, β-chloroethyl methyl]



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1. Procedure

In a 1-l. three-necked flask are mixed 150 g. (1.63 moles) of β-hydroxyethyl methyl sulfide (p. 345) (Note 1) and 200 g. of dry chloroform (Note 2). The flask is placed on a steam bath and is fitted with a dropping funnel, a mechanical stirrer, and a condenser. The condenser is fitted with a trap to remove the vapors of hydrogen chloride and sulfur dioxide. A solution of 204 g. (1.7 moles) (Note 3) of thionyl chloride in 135 cc. of dry chloroform is added dropwise to the β-hydroxyethyl methyl sulfide over a period of about two hours (Note 4). The reaction mixture is stirred vigorously during this addition and for about four hours after the addition is complete. The chloroform is distilled on the steam bath and the residue is distilled under reduced pressure. The yield is 135–153 g. (75–85 per cent of the theoretical amount) of a product boiling at 55–56°/30 mm. (Note 5).

2. Notes

1. Quantities of material seven times as large as the above may be used without decreasing the percentage yield of the product.
2. The chloroform is dried by distillation, and the fraction boiling at 60–61° is used.
3. The thionyl chloride is redistilled, and the fraction boiling over a two-degree range is employed.
4. The reaction mixture is heated once when about half the thionyl chloride has been added in order to keep the chloroform refluxing gently. Heating after the complete addition of the thionyl chloride is undesirable.
5. β-Chloroethyl methyl sulfide is a vesicant and must be handled with care. It boils at 140° under atmospheric pressure.

3. Discussion

The above method is essentially that described in the literature.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 384

References and Notes

1. Kirner, J. Am. Chem. Soc. **50**, 2452 (1928).

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

hydrogen chloride (7647-01-0)

thionyl chloride (7719-09-7)

chloroform (67-66-3)

sulfur dioxide (7446-09-5)

β-Chloroethyl methyl sulfide,
Sulfide, β-chloroethyl methyl (542-81-4)

β-Hydroxyethyl methyl sulfide (5271-38-5)