



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.

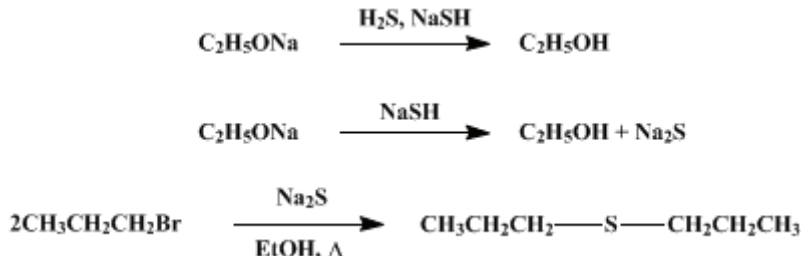
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.

Organic Syntheses, Coll. Vol. 2, p.547 (1943); Vol. 15, p.72 (1935).

***n*-PROPYL SULFIDE**



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1. Procedure

In a 2-l. round-bottomed flask, fitted with a reflux condenser and a 6-mm. glass tube closed at the upper end with a rubber tube and pinch-cock and reaching to the bottom of the flask, are placed 800 cc. of absolute alcohol and 50.6 g. (2.2 gram atoms) of clean sodium cut in small pieces. When the sodium is completely dissolved, one half of the solution is transferred to a 2-l. three-necked, round-bottomed flask fitted with a dropping funnel, a liquid-sealed mechanical stirrer, and a reflux condenser closed by a calcium chloride tube. The flask containing the remainder of the solution is connected again to the reflux condenser, and hydrogen sulfide from a cylinder is introduced by means of the glass tube at the rate of about two bubbles per second until the solution is saturated (about six hours is required). This solution of sodium hydrogen sulfide is added to the sodium ethoxide solution in the three-necked flask, and the mixture is refluxed for one hour. After cooling to room temperature, sufficient absolute alcohol (about 200 cc.) is added to dissolve all the sodium sulfide (Note 1).

To this solution of sodium sulfide is added dropwise with stirring 246 g. (2 moles) of *n*-propyl bromide (Org. Syn. Coll. Vol. I, 1941, 37 and p. 359 above). After all the bromide has been added the flask is heated on a steam cone for eight hours (Note 2), during which time the mixture should not be stirred too vigorously. It is then cooled and added to 2 l. of 25 per cent aqueous sodium chloride solution contained in a separatory funnel. The mixture is shaken to ensure thorough mixing, allowed to stand until the layers have separated, and the upper oily layer of propyl sulfide removed and dried with anhydrous sodium sulfate. The lower layer is extracted with five 200-cc. portions of petroleum ether (b.p. 25–45°) (Note 3), the extract dried with 20 g. of sodium sulfate, and the petroleum ether distilled through a 60-cm. fractionating column until the temperature of the vapors passing over reaches 60°. The residue is added to the crude propyl sulfide previously separated and the combined portions distilled. The yield of product boiling at 140–143° is 80–100 g. (68–85 per cent of the theoretical amount) (Note 4).

2. Notes

1. It is best to have the sodium sulfide completely dissolved before it is added to the bromide; subsequent extraction is facilitated, however, if the solution is kept at a minimum volume.
2. In order to be certain that no propyl sulfide is being lost during the refluxing, the condenser should be connected to a trap containing an aqueous solution of mercuric chloride. Condenser water cooled to 5–10° is recommended.
3. To avoid contamination of the product the petroleum ether should be distilled before being used. Ethyl ether is not suitable for this extraction.
4. *n*-Butyl and *sec.*-amyl sulfides can be prepared by procedures essentially the same as that given here.

3. Discussion

n-Propyl sulfide has always been prepared by the action of an alcoholic solution of an alkali sulfide

on a *n*-propyl halide.¹

References and Notes

1. Cahours, Compt. rend. **76**, 133 (1973); Winssinger, Bull. soc. chim. (2) **48**, 109 (1887).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

petroleum ether

alcohol (64-17-5)

ethyl ether (60-29-7)

sodium chloride (7647-14-5)

hydrogen sulfide (7783-06-4)

bromide (24959-67-9)

n-PROPYL BROMIDE (106-94-5)

sodium sulfate (7757-82-6)

sodium (13966-32-0)

sodium ethoxide (141-52-6)

mercuric chloride (7487-94-7)

sodium sulfide (1313-82-2)

sodium hydrogen sulfide

Propyl sulfide,
n-PROPYL SULFIDE (111-47-7)

n-butyl sulfide (544-40-1)

n-amyl sulfide