



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.

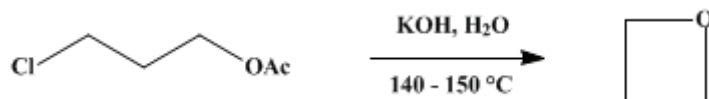
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.835 (1955); Vol. 29, p.92 (1949).

TRIMETHYLENE OXIDE

[Oxetane]



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1. Procedure

A 3-l. Pyrex flask (Note 1) is fitted with an efficient mechanical stirrer, a dropping funnel, a thermometer, and an upright tube (Note 2) to act as an air condenser and to carry the trimethylene oxide vapors through a spiral condenser. In the flask are placed 1344 g. (24 moles) of potassium hydroxide and 120 ml. of water. The contents are heated to about 140° with stirring (Note 3). The upper part of the flask should be kept sufficiently hot so that the potassium hydroxide mush which is thrown up on the sides does not solidify. The spiral condenser is then packed with an ice-salt mixture, and 1092 g. (8 moles) of γ -chloropropyl acetate (p. 203) (Note 4) is slowly run into the flask by means of the dropping funnel so that the distillate comes over at the rate of about 1 drop a second. The time required for the addition is 1.5–3 hours. After all the ester has been added, the mixture is stirred, with continued heating at 140–150°, until the distillate amounts to 250–400 g., depending on how efficiently the air condenser has separated the trimethylene oxide from higher-boiling substances. About 100 g. of potassium hydroxide pellets is added to the crude distillate, which is then distilled through a 25-cm. fractionating column packed with $\frac{1}{4}$ -in. Berl Saddles. By careful distillation the trimethylene oxide can be obtained in a single fractionation, the portion boiling at 45–50° being collected. The higher-boiling residue still contains some trimethylene oxide, but consists mostly of water, allyl alcohol, and unchanged γ -chloropropyl acetate. The yield is 195–205 g. (42–44%). Redistillation over freshly fused potassium hydroxide gives a product boiling at 47–48°; n_{23D} 1.3905.

2. Notes

1. A half-gallon jacketed iron autoclave may be employed.
2. A plain 90-cm. 10-mm. tube or a 30-cm. Vigreux column is equally satisfactory.
3. An electric heating mantle is advisable. The reaction is exothermic, and if it appears to become too vigorous the current is temporarily discontinued.
4. The submitter states that a yield of 20–25% may be obtained when trimethylene chlorohydrin is substituted for the acetate, with 2 moles of potassium hydroxide and 10 ml. of water per mole of chlorohydrin.

3. Discussion

This procedure is an improvement upon that of Reboul,¹ and of Derick and Bissell.² Reboul, as also did Ipatow,³ used trimethylene chlorohydrin, but the yield is higher from the acetate.

References and Notes

1. Reboul, *Ann. chim.*, (5) **14**, 495 (1878).
2. Derick and Bissell, *J. Am. Chem. Soc.*, **38**, 2478 (1916).
3. Ipatow, *J. Russ. Phys. Chem. Soc.*, **46**, 67 (1914) [*C. A.*, **8**, 1965 (1914); *Chem. Zentr.*, **85**, I, 2161 (1914)].

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

Allyl alcohol (107-18-6)

potassium hydroxide,
potassium hydroxide pellets (1310-58-3)

Trimethylene chlorohydrin (627-30-5)

Trimethylene oxide,
Oxetane (503-30-0)

γ -Chloropropyl acetate (628-09-1)