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.

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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.

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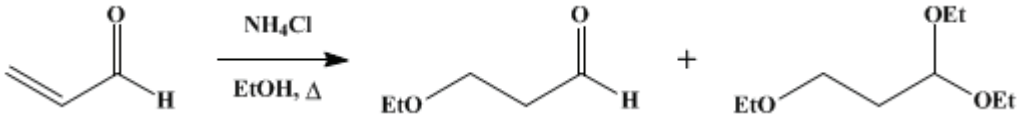
β-ETHOXYPROPIONALDEHYDE ACETAL

[Propionaldehyde, β-ethoxy-, diethyl acetal]



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1. Procedure

In a 1-l. three-necked round-bottomed flask fitted with a reflux condenser, a mechanical stirrer, and a thermometer dipping below the surface of the mixture are placed 158 g. (2.8 moles) of acrolein (Note 1), 500 ml. of absolute ethanol, and 10 g. of ammonium chloride (Note 2). The material is stirred for 1 hour without external heating; the temperature of the mixture should rise to about 30°. Over a 3-hour period the temperature is raised to 80°, and the mixture is maintained at this temperature for an additional hour. The flask is then cooled, and anhydrous sodium or magnesium sulfate is added as a drying agent.

After 24 hours the mixture is filtered and distilled under reduced pressure through a good column (Note 3). The following fractions are collected (Note 4):

Below 23°/20 mm.	Acrolein and alcohol
38–42°/16 mm.	β-Ethoxypropionaldehyde
75–78°/16 mm.	β-Ethoxypropionaldehyde acetal

The yield of β-ethoxypropionaldehyde is 22–24 g. (7–8%) and of β-ethoxypropionaldehyde acetal is 166–193 g. (31–39%), n_D^{20} 1.4067, d_4^{15} 0.898.

2. Notes

1. Commercial acrolein may be employed. It should be dried over anhydrous sodium sulfate.
2. Reagent grade ammonium chloride is used. It should be washed several times with absolute ethanol.
3. A 1-m. Fenske column packed with 3/32-in. glass helices was employed by the submitters.
4. Ammonium chloride separates during the distillation. It is advisable to decant the liquid into a clean flask for distillation of the last two fractions.

3. Discussion

β-Ethoxypropionaldehyde acetal is best prepared from acrolein by reaction with ethanol.[1,2]

References and Notes

1. Hall and Stern, *Chemistry & Industry*, **1950**, 775.
2. *Org. Syntheses*, **25**, 1 (1945).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

ethanol (64-17-5)

ammonium chloride (12125-02-9)

Acrolein (107-02-8)

sodium sulfate (7757-82-6)

magnesium sulfate (7487-88-9)

β-Ethoxypropionaldehyde acetal (7789-92-6)

Propionaldehyde, β-ethoxy-, diethyl acetal

β-Ethoxypropionaldehyde (2806-85-1)