



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. 2, p.17 (1943); Vol. 11, p.1 (1931).

ACROLEIN ACETAL

[Acrolein, diethyl acetal]

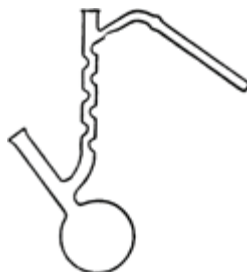


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1. Procedure

To 340 g. (6 moles) of dry, powdered potassium hydroxide (Note 1) in a 500-cc. short-necked round-bottomed flask (Note 2) is added 167 g. (1 mole) of β -chloropropionaldehyde acetal (p. 137). The mixture is shaken vigorously and attached at once to a three-bulbed Glinsky or other suitable column, connected to a water condenser set for distillation (Note 3). The flask is heated in an oil bath at 210–220° until nothing more distils (Note 4). The distillate is transferred to a separatory funnel and the lower aqueous layer is removed. The acrolein acetal is dried over 10 g. of potassium carbonate, filtered, and distilled from a modified Claisen flask (Fig. 2). The yield is 98 g. (75 per cent of the theoretical amount) of a product which boils at 122–126°.

Fig. 2



2. Notes

1. The powdered potassium hydroxide should pass a 60-mesh sieve (24 per cm.). The dryness of the powder is of the utmost importance. Water must be avoided as much as possible. Therefore, the potassium hydroxide should be fused at 350° for two hours and then pulverized as rapidly as possible. A 24-cm. (10-in.) disk pulverizer having a capacity of about 200 g. per minute is recommended. If the potassium hydroxide is not fused before pulverizing, the yield of acetal drops to about 60 per cent.
2. The concentrated potassium hydroxide left at the completion of the reaction attacks the glass rapidly. For this reason iron retorts made from 10-cm. (4-in.) pipe should be used when many runs are to be made.
3. The reaction between the acetal and the alkali is very vigorous.
4. A large low-boiling fraction indicates too much moisture in the potassium hydroxide used. See (Note 1).

3. Discussion

Acrolein acetal has been prepared by treatment of β -chloropropionaldehyde acetal with dry, powdered potassium hydroxide,¹ and from acrolein, ethyl orthoformate, and ammonium nitrate in boiling alcohol.²

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 2, 307](#)
- [Org. Syn. Coll. Vol. 4, 21](#)

References and Notes

1. Wohl, Ber. **31**, 1798 (1898); Witzemann, J. Am. Chem. Soc. **36**, 1911 (1914); Spoehr and Young, Carnegie Inst. Washington Yearbook, **25**, 176 (1925–1926); Expt. Sta. Record, **57**, 817 (1927) [C. A. **22**, 2368 (1928)].
2. Fischer and Baer, Helv. Chim. Acta **18**, 516 (1935).

Appendix

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

[alcohol \(64-17-5\)](#)

[potassium carbonate \(584-08-7\)](#)

[Acrolein \(107-02-8\)](#)

[potassium hydroxide \(1310-58-3\)](#)

[ammonium nitrate](#)

[Ethyl orthoformate](#)

[Acrolein acetal](#)

[Acrolein, diethyl acetal](#)

[β-chloropropionaldehyde acetal \(35573-93-4\)](#)