



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](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.

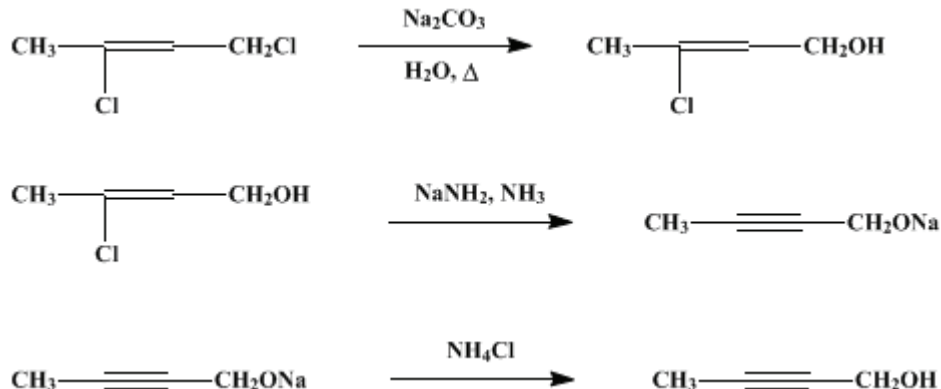
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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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## 2-BUTYN-1-OL



Submitted by P. J. Ashworth, G. H. Mansfield, and M. C. Whiting<sup>1</sup>.  
Checked by John C. Sheehan, George Buchi, and Walfred S. Saari.

### 1. Procedure

*Caution! The experimental procedure involving liquid ammonia should be conducted in a hood.*

In a 3-l. three-necked round-bottomed flask fitted with a reflux condenser and a mercury-sealed stirrer, 250 g. (2 moles) of 1,3-dichloro-2-butene (Note 1) and 1.25 l. of 10% sodium carbonate are heated at reflux temperature for 3 hours. The 3-chloro-2-buten-1-ol is extracted with three 300-ml. portions of ether, which are then dried over anhydrous magnesium sulfate. The ether is removed by distillation through a 20-cm. Fenske column, and the residue is distilled from a 250-ml. Claisen flask, yielding 134 g. (63%) of 3-chloro-2-buten-1-ol, b.p. 58–60°/8 mm.,  $n_D^{20}$  1.4670.

A solution of sodium amide in liquid ammonia is prepared according to the procedure described on p. 763 using a 4-l. Dewar flask equipped with a plastic cover (Note 2) and a mechanical stirrer. Anhydrous liquid ammonia (3 l.) is introduced through a small hole in the plastic cover, and 1.5 g. of hydrated ferric nitrate is added followed by 65 g. (2.8 g. atoms) of clean, freshly cut sodium. The mixture is stirred until all the sodium is converted into sodium amide, after which 134 g. (1.26 moles) of 3-chloro-2-buten-1-ol is added over a period of 30 minutes. The mixture is stirred overnight, then 148 g. (2.8 moles) of solid ammonium chloride is added in portions at a rate that permits control of the exothermic reaction. The mixture is transferred to a metal bucket (5-l., preferably of stainless steel) and allowed to stand overnight in the hood while the ammonia evaporates. The residue is extracted thoroughly with five 250-ml. portions of ether, which is removed by distillation through a 20-cm. Fenske column. Distillation of the residue yields 66–75 g. (75–85%) of 2-butyne-1-ol, b.p. 55°/8 mm.,  $n_D^{20}$  1.4550 (Note 3).

### 2. Notes

- 1,3-Dichloro-2-butene was obtained from Eastman Kodak Company. Redistillation of the crude material did not appreciably alter the yield in the first stage of the reaction.
- The checkers used an ordinary flask, well insulated, for this reaction.
- Others have reported b.p. 91–93°/50 mm.,  $n_D^{25}$  1.4630;<sup>2</sup> b.p. 160.5°,  $n_D^{25}$  1.4635;<sup>3</sup> b.p. 160–161°,  $n_D^{25}$  1.4635, for 3-chloro-2-buten-1-ol, and b.p. 87–88°/100 mm.,  $n_D^{25}$  1.4520;<sup>2</sup> b.p. 140–141°,  $n_D^{25}$  1.4517<sup>4</sup> for 2-butyne-1-ol.

### 3. Discussion

2-Butyn-1-ol has been prepared as above for the first stage<sup>2,3,4</sup> but using aqueous sodium hydroxide<sup>2</sup>

and alcoholic [potassium hydroxide](#)<sup>4</sup> for the second stage. The reaction between [propynylmagnesium bromide](#) and [formaldehyde](#) also has been employed.<sup>5,6,7</sup>

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### References and Notes

1. Victoria University of Manchester, Manchester, England.
2. Hatch and Nesbitt, *J. Am. Chem. Soc.*, **72**, 729 (1950).
3. Hatch and Hudson, *J. Am. Chem. Soc.*, **72**, 2505 (1950).
4. Hatch and Chiola, *J. Am. Chem. Soc.*, **73**, 360 (1951).
5. Yvon, *Compt. rend.*, **180**, 748 (1925).
6. Hurd and Cohen, *J. Am. Chem. Soc.*, **53**, 1074 (1931).
7. Schulte and Reiss, *Chem. Ber.*, **87**, (1954).

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

[ammonia](#) (7664-41-7)  
[ether](#) (60-29-7)  
[ammonium chloride](#) (12125-02-9)  
[sodium hydroxide](#) (1310-73-2)  
[formaldehyde](#) (50-00-0)  
[sodium carbonate](#) (497-19-8)  
[potassium hydroxide](#) (1310-58-3)  
[sodium](#) (13966-32-0)  
[magnesium sulfate](#) (7487-88-9)  
[sodium amide](#) (7782-92-5)  
[ferric nitrate 2-Butyn-1-ol](#) (764-01-2)  
[1,3-dichloro-2-butene 3-Chloro-2-buten-1-ol](#) (40605-42-3)  
[propynylmagnesium bromide](#)