



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.

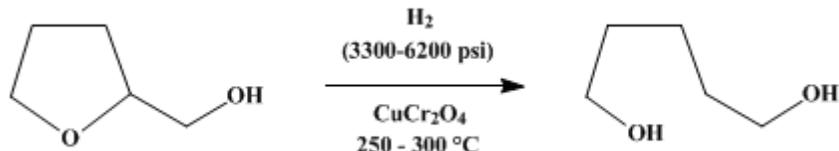
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.693 (1955); Vol. 26, p.83 (1946).

1,5-PENTANEDIOL



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Checked by Homer Adkins and Harry Billica.

1. Procedure

Five hundred and ten grams (5 moles) of pure [tetrahydrofurfuryl alcohol](#) ([Note 1](#)) and 50 g. of [copper chromite](#) ([Note 2](#)) are placed in a hydrogenation bomb having a void of 1.4 l. ([Note 3](#)). The bomb, in a suitable rocker assembly, is filled with [hydrogen](#) to a pressure of 3300 to 3500 lb. per sq. in. ([Note 4](#)). The bomb is rocked and heated. *The pressure must not at any time be allowed to go much above 6000 lb. per sq. in.* If the pressure rises higher than 6200 lb., the heating of the bomb should be stopped ([Note 5](#)). In a typical run, the pressure reaches a maximum of about 6100 lb. at 255° after 2 hours. The pressure then slowly falls to about 4400 lb. during another hour, as the temperature rises to a little over 300°. During the ensuing 6 hours, the pressure falls to about 3000 lb. while the temperature is held at 300–310°.

The bomb is allowed to cool to room temperature, where the pressure should be 1000–1100 lb. The contents of the bomb are poured into a beaker, and the bomb is rinsed twice with 100-ml. portions of [acetone](#). The catalyst is removed by centrifuging, and the reaction mixture is distilled through a fractionating column ([Note 6](#)). A fraction boiling in the range 60–140°, containing [α-methyltetrahydrofuran](#), water, and *n*-[amyl alcohol](#), is distilled at atmospheric pressure ([Note 7](#)). [Tetrahydrofurfuryl alcohol](#) (80–110 g.) is recovered, boiling at 65–70°/10 mm.; the [1,5-pentanediol](#) boiling at 118–120°/6 mm. is obtained in amounts varying from 200 g. to 244 g. The yield is 40–47% of the theoretical, without allowance being made for the recovered [tetrahydrofurfuryl alcohol](#). The residue of products boiling above the glycol amounts to 25–35 g.

2. Notes

1. The [tetrahydrofurfuryl alcohol](#) available from the Quaker Oats Company, or the practical grade from the Eastman Kodak Company, has been used. If the material available does not hydrogenate satisfactorily, it may be purified by hydrogenation over [Raney nickel](#) at 150°/100–200 atm. pressure. A sample of good quality boils at 177–178°/740 mm. and does not become dark-colored when a few milliliters are shaken with 1 drop of concentrated [sulfuric acid](#) at room temperature.
2. The catalyst is prepared as described in [Note 11](#) for the preparation of [copper chromite](#).¹
3. The hydrogenation bomb supplied under catalog No. 406–21, by the American Instrument Company, Silver Spring, Maryland, is satisfactory. The internal volume of the empty bomb should be at least 2.7 times that of the volume of [tetrahydrofurfuryl alcohol](#) added, as otherwise not enough [hydrogen](#) can be added for the completion of the reaction, without the use of excessively high pressures. The submitters maintained the pressure above 5500 lb. per sq. in. by intermittently adding [hydrogen](#).
4. Temperatures higher than about 300° and pressures higher than 6000 lb. per sq. in. should not be used in the vessels and with the gauges ordinarily supplied for "high-pressure" hydrogenations. *Only clean equipment, in first-class condition, and under careful control, can be used safely and successfully in carrying out reactions under the conditions described.*
5. If hydrogenolysis does not occur, the pressure would be about 7000 lb. when a temperature of 300° is reached. If the pressure rises above 6200, starting with 3500 lb. at room temperature, it is evident that the quality of the catalyst or the alcohol is not satisfactory. Further attempts to prepare the glycol should be made with alcohol and catalyst of better quality, or starting with a pressure sufficiently low (3000 lb.) so that a safe operating pressure will not be exceeded.

6. A Vigreux-type column 60 cm. in length and 2 cm. in outside diameter was used for the fractionation. The submitters state that they have also used a column of similar dimensions, packed with glass helices, for a fractionation at atmospheric pressure. They recommended the Vigreux column under reduced pressure, as used by the checkers.

7. When acetone was not used for washing out the bomb, a fraction weighing 70–75 g. was obtained by the submitters. After drying over anhydrous potassium carbonate, they obtained, by fractional distillation, 3–6 g. of α -methyltetrahydrofuran, b.p. 80–81°, and 23–28 g. of *n*-amyl alcohol, b.p. 137–138°. The alcohol obtained was pure, and neither secondary amyl nor butyl alcohols could be detected.

3. Discussion

1,5-Pentanediol has been prepared by the reduction of methyl^{2,3} or ethyl⁴ glutarate, of 2,3-dihydropyran,⁵ of δ -hydroxyvaleraldehyde,^{6,7,8,9,10} and of furfural.¹¹ It has also been prepared from pentamethylene bromide by conversion to the diacetate and subsequent hydrolysis.¹² The procedure described is a modification of that of Connor and Adkins.¹³

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 4, 746](#)

References and Notes

1. *Org. Syntheses Coll. Vol. 2*, 144 (1943).
2. Wojcik and Adkins, *J. Am. Chem. Soc.*, **55**, 4939 (1933).
3. Bennett and Reynolds, *J. Chem. Soc.*, **1935**, 138.
4. Adkins and Billica, *J. Am. Chem. Soc.*, **70**, 3121 (1948).
5. U. S. pat. 2,440,929 [*C. A.*, **42**, 5466 (1948)].
6. Paul, *Bull. soc. chim. France*, (5) **1**, 978 (1934).
7. Schniepp and Geller, *J. Am. Chem. Soc.*, **68**, 1646 (1946).
8. Woods and Saunders, *J. Am. Chem. Soc.*, **68**, 2111 (1946).
9. Ital. pat. 439,947 [*C. A.*, **44**, 5915 (1950)].
10. U. S. pat. 2,497,812 [*C. A.*, **44**, 6428 (1950)].
11. Brit. pat. 627,293 [*C. A.*, **44**, 2564 (1950)].
12. Bennett and Heathcoat, *J. Chem. Soc.*, **1929**, 273.
13. Connor and Adkins, *J. Am. Chem. Soc.*, **54**, 4678 (1932).

Appendix

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

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

[sulfuric acid](#) (7664-93-9)

[hydrogen](#) (1333-74-0)

[Raney nickel](#) (7440-02-0)

[acetone](#) (67-64-1)

[pentamethylene bromide](#) (111-24-0)

Furfural (98-01-1)

1,5-Pentanediol (111-29-5)

tetrahydrofurfuryl alcohol (97-99-4)

n-amyl alcohol (71-41-0)

COPPER CHROMITE

δ -hydroxyvaleraldehyde (4221-03-8)

α -methyltetrahydrofuran (96-47-9)