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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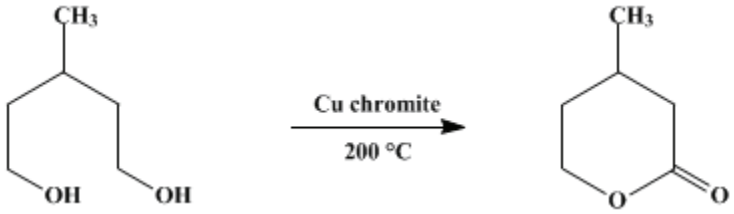
Organic Syntheses, Coll. Vol. 4, p.677 (1963); Vol. 35, p.87 (1955).

β-METHYL-δ-VALEROLACTONE

[Valeric acid, 5-hydroxy-3-methyl-, δ-lactone]



Submitted by Raymond I. Longley, Jr. and William S. Emerson[1].
Checked by T. L. Cairns and W. W. Gilbert.

1. Procedure

A 1-l. three-necked flask fitted with an efficient stirrer, a thermometer, and a reflux condenser attached to a device for measuring gas evolution (Note 1) is charged with 197 g. (1.67 moles) of 3-methyl-1,5-pentanediol (p. 660) and 10 g. of copper chromite (Note 2). The mixture is heated rapidly to 200° (Note 3) with good stirring and is held at 195–205° for 1.5–3.0 hours, during which time 3.1 cu. ft. of hydrogen is evolved (Note 4). The product is distilled directly from the flask with stirring through a 2 by 120 cm. Vigreux column (Note 5). The yield of β-methyl-δ-valerolactone is 172–180 g. (90–95%), b.p. 110–111°/15 mm., n_D^{25} 1.4495.

2. Notes

1. A standard wet test meter may be used.
2. Copper chromite is prepared according to *Organic Syntheses*[2] and washed with sodium bicarbonate solution. The glycol is slurried with sodium bicarbonate and filtered before use.
3. At this point gas evolution becomes so rapid that the temperature tends to drop slightly.
4. If gas evolution subsides more catalyst may be added.
5. The column is substituted for the reflux condenser in the same set-up. Stirring during distillation prevents serious bumping.

3. Discussion

β-Methyl-δ-valerolactone has been prepared by heating 3-methyl-1,5-pentanediol with copper chromite in the liquid phase,[3] by passing the vapors of 3-methyl-1,5-pentanediol over copper on pumice,[3] by heating 2-methoxy-4-methyl-3,4-dihydro-2H-pyran with water and copper chromite,[3] by treating 3-methylglutaraldehyde with aqueous alkali,[3] and by reducing β-methylglutaric anhydride with sodium and ethanol.[4] The present method was first developed by Kyrides and Zienty.[5]

References and Notes

1. Monsanto Chemical Company, Dayton 7, Ohio.
2. *Org. Syntheses Coll. Vol.* **2**, 142 (1943).
3. Longley, Emerson, and Shafer, *J. Am. Chem. Soc.*, **74**, 2012 (1952); Emerson, Longley, and Shafer (to Monsanto Chemical Co.), U. S. pat. 2,680,118 [*C. A.*, **49**, 6315 (1955)].
4. Sircar, *J. Chem. Soc.*, **1928**, 898.
5. Kyrides and Zienty, *J. Am. Chem. Soc.*, **68**, 1385 (1946).

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

ethanol (64-17-5)

hydrogen (1333-74-0)

sodium bicarbonate (144-55-8)

copper (7440-50-8)

sodium (13966-32-0)

COPPER CHROMITE

2-methoxy-4-methyl-3,4-dihydro-2H-pyran (53608-95-0)

β-Methylglutaric anhydride (4166-53-4)

3-Methyl-1,5-pentanediol (4457-71-0)

3-methylglutaraldehyde (6280-15-5)

β-Methyl-δ-valerolactone,
Valeric acid, 5-hydroxy-3-methyl-, δ-lactone (1121-84-2)