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.

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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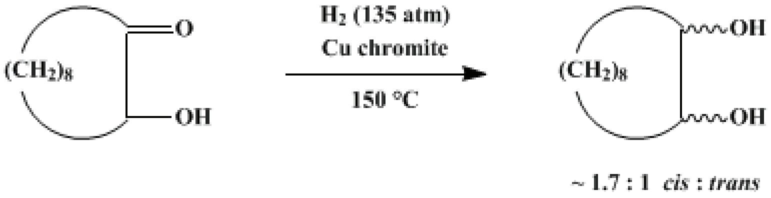
Organic Syntheses, Coll. Vol. 4, p.216 (1963); Vol. 36, p.12 (1956).

1,2-CYCLODECANEDIOL



Submitted by A. T. Blomquist and Albert Goldstein[1].
Checked by N. J. Leonard and F. H. Owens.

1. Procedure

A mixture of 140 g. (0.82 mole) of sebacoin (p. 840) (Note 1), 50 g. of copper chromite catalyst[2] (Note 2), and 230 ml. of 95% ethanol is placed in an 800-ml. high-pressure hydrogenation bomb (Note 3). Hydrogen is admitted to the bomb at 135 atm., and the bomb is heated to 150°. When this temperature is reached, the shaker is started. The temperature is stabilized at 150° after its initial rapid fluctuation, and the hydrogenation is allowed to proceed until the mixture ceases to absorb hydrogen (2–4 hours). The heating and shaking are discontinued, and the bomb is allowed to cool to room temperature. The excess hydrogen is vented, and the bomb is dismantled.

The *cis*-1,2-cyclodecanediol will have crystallized out of solution, while the *trans*-diol remains in the ethanol. The entire mixture is washed out of the bomb with 95% ethanol (about 1 l.). The *cis*-glycol is redissolved by heating the ethanolic mixture at reflux temperature. Filter aid ("Celite") is added to the mixture, and the hot mixture is filtered through a bed of filter aid on a Büchner funnel to remove the catalyst. The ethanol is removed from the filtrate by distillation on a steam bath under water-pump pressure. The residue is dissolved in a minimum of hot 1:1 benzene-ethanol solution (about 250 ml.), and the *cis*-diol crystallizes upon cooling to room temperature. The crystals are collected by filtration, and the mother liquor is concentrated to dryness on a steam bath under water-pump pressure. The residue is again dissolved in a minimum of hot 1:1 benzene-ethanol (about 100 ml.), and additional *cis*-diol crystallizes upon cooling in a refrigerator. The process is repeated using 50 ml. of 1:1 benzene-ethanol solvent. The total yield of *cis*-1,2-cyclodecanediol is 68–73 g. (48–52%), m.p. 137–138°.

The *trans*-diol remains in the mother liquor and may be recovered by complete evaporation of the solvent followed by recrystallization of the residue from pentane. The yield of *trans*-1,2-cyclodecanediol is 38–45 g. (27–32%), m.p. 53–54°.

2. Notes

1. A sebacoin-sebacil mixture may be used.
2. A commercial catalyst was employed by the submitters: Harshaw Chemical Company, CU-0202P; 556–002.
3. A suitable apparatus is the "Aminco" high-pressure hydrogenation apparatus, manufactured by the American Instrument Company, Silver Spring, Maryland.

3. Discussion

1,2-Cyclodecanediol has been prepared by the hydrogenation of sebacoin in the presence of Raney nickel[3] or platinum,[4] by the reduction of sebacoin with aluminum isopropoxide[4] or lithium aluminum hydride,[4] and by the oxidation of cyclodecene with osmium tetroxide and pyridine.[3]

References and Notes

1. Cornell University, Ithaca, New York.
2. *Org. Syntheses Coll. Vol.* **2**, 142 (1943), Note 11.
3. Prelog, Schenker, and Günthard, *Helv. Chim. Acta*, **35**, 1598 (1952); Prelog, Urech, Bothner-By, and Würsch, *Helv. Chim. Acta*, **38**, 1095 (1955).
4. Blomquist, Burge, and Sucsy, *J. Am. Chem. Soc.*, **74**, 3636 (1952).

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

Sebacoin

ethanol (64-17-5)

hydrogen (1333-74-0)

platinum (7440-06-4)

nickel (7440-02-0)

pyridine (110-86-1)

aluminum isopropoxide

Pentane (109-66-0)

benzene-ethanol (60-12-8)

COPPER CHROMITE

lithium aluminum hydride (16853-85-3)

osmium tetroxide (20816-12-0)

1,2-Cyclodecanediol (21014-77-7)

cyclodecene

cis-1,2-cyclodecanediol

trans-1,2-cyclodecanediol