



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.

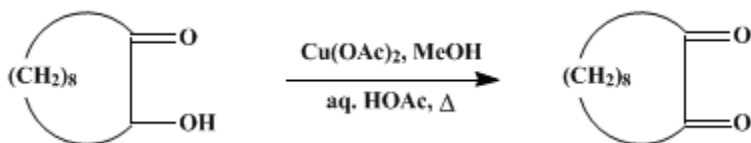
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.838 (1963); Vol. 36, p.77 (1956).

SEBACIL

[1,2-Cyclodecanedione]



Submitted by A. T. Blomquist and Albert Goldstein¹.

Checked by N. J. Leonard and J. C. Little.

1. Procedure

Fifty-one grams (0.30 mole) of sebacoin (Note 1), 25 ml. of methanol, 120 g. of cupric acetate monohydrate (0.60 mole), and 300 ml. of 50% aqueous acetic acid are mixed in a 1-l. flask equipped with an efficient mechanical stirrer and a reflux condenser. The mixture is heated over a free flame until refluxing occurs. The color of the mixture changes from blue to red at approximately 75°. Refluxing is continued for 1 minute. The mixture is then allowed to cool, with stirring, to about 40° (Note 2). The mixture is filtered through filter aid ("Celite") on a sintered glass funnel to remove cuprous oxide, and the filtrate is transferred to a 2-l. separatory funnel (Note 3). Saturated aqueous sodium chloride solution. (310 ml.) is added to the filtrate, which is then extracted with three 150-ml. portions of ether. The combined ether extracts are washed with three 250-ml. portions of saturated salt solution, four 250-ml. portions of 5% sodium bicarbonate solution (foaming!), and once again with 250 ml. of saturated salt solution. The ether solution is then dried over 20 g. of anhydrous sodium or magnesium sulfate. The ether is removed by distillation at atmospheric pressure, and the residue is transferred to a 100-ml. flask and distilled under vacuum. The yield of sebacil is 44.4–45.1 g. (88–89%), b.p. 104–106°/10 mm. (Note 4).

2. Notes

1. The sebacoin-sebacil mixture obtained from the sebacoin preparation (p. 840) was used.
2. The sebacil may tend to crystallize during filtration if the reaction mixture is too cool.
3. The "Celite"-cuprous oxide mixture is extracted with three 50-ml. portions of ether, and the combined extracts are used as the first ether portion for the extraction of the sebacil-containing aqueous solution.
4. 1,2-Cyclononanedione has been prepared from 2-hydroxycyclononane in 67–72% yield by this procedure.²

3. Discussion

1,2-Cyclodecanedione has also been prepared by oxidation of sebacoin with chromium trioxide in acetic acid.^{3,4} Cupric acetate in acetic acid has been used for oxidation of an α -hydroxyketone by Ruggli and Zeller.⁵

References and Notes

1. Cornell University, Ithaca, New York.
 2. Blomquist, Liu, and Bohrer, *J. Am. Chem. Soc.*, **74**, 3643 (1952).
 3. Blomquist, Burge, and Sucsy, *J. Am. Chem. Soc.*, **74**, 3636 (1952).
 4. Prelog, Schenker, and Günthard, *Helv. Chim. Acta*, **35**, 1610 (1952).
 5. Ruggli and Zeller, *Helv. Chim. Acta*, **28**, 741 (1945).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

sodium or magnesium sulfate

Sebacoin

sebacoïn-sebacil

Celite

acetic acid (64-19-7)

methanol (67-56-1)

ether (60-29-7)

sodium bicarbonate (144-55-8)

sodium chloride (7647-14-5)

cuprous oxide

cupric acetate (142-71-2)

chromium trioxide (1333-82-0)

cupric acetate monohydrate (6046-93-1)

1,2-Cyclodecanedione,
Sebacil (96-01-5)

1,2-Cyclononanedione

2-hydroxycyclononane