



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.

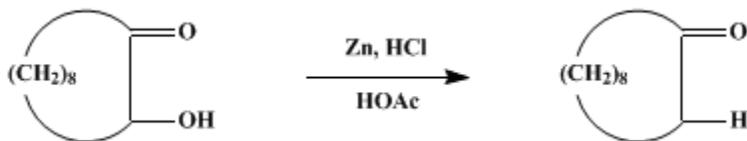
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.218 (1963); Vol. 36, p.14 (1956).

CYCLODECANONE



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1. Procedure

A 1-l. round-bottomed three-necked flask is fitted with a sealed stirrer (Note 1), a dropping funnel, and a reflux condenser, through which a thermometer extends nearly to the bottom of the flask. In the flask are thoroughly mixed 40.5 g. (0.62 g. atom) of zinc dust (Note 2) and 100 ml. of glacial acetic acid, and to this mixture is added 42.5 g. (0.25 mole) of sebacoin (p.840) (Note 3). The mixture is stirred rapidly, and 90 ml. of concentrated C.P. hydrochloric acid is added dropwise during a period of 5 to 10 minutes, or as fast as control of foaming and temperature permits. The temperature must be kept between 75 and 80° (Note 4), and cooling by a water bath may be necessary during the addition of the hydrochloric acid. Stirring is continued for 1.5 hours at 75–80°. Thirty minutes after the initial addition of hydrochloric acid, and again 30 minutes later, 90-ml. portions of concentrated hydrochloric acid are added to the mixture while the temperature is maintained at 75–80°. After the reaction is complete, the remaining zinc is separated from the cooled mixture by decantation (Note 5). The liquid phase is diluted with 700 ml. of saturated aqueous sodium chloride solution and extracted with four 250-ml. portions of ether, each of which is first used to wash the residual zinc (Note 6). The ether extracts are combined and washed with 250 ml. of saturated sodium chloride solution, three 250-ml. portions of 10% sodium carbonate solution (foaming!), and finally 250 ml. of saturated sodium chloride solution. The ethereal solution is dried over anhydrous magnesium sulfate (about 25 g. is needed). After the drying agent has been removed by filtration and the solvent by distillation, the residue is distilled at reduced pressure through an efficient column (Note 7). After a small fore-run consisting mostly of cyclodecane, cyclodecanone is collected at 99–101°/8 mm. The yield is 29–30 g. (75–78%), n_D^{25} 1.4808–1.4810 (Note 8).

2. Notes

1. A metal stirrer must not be used. A simple glass stirrer with a ball-joint seal is satisfactory.
2. Mallinckrodt technical grade may be used.² If Mallinckrodt analytical reagent zinc dust is used, the reaction temperature must be maintained at 50–55° instead of 75–80°.
3. Pure sebacoin gives a colorless product. A sebacoin-sebacil mixture must first be purified by recrystallization from pentane as described (p.841). The sebacil apparently is not reduced completely according to the accompanying directions and thus may contaminate the product (Note 7).
4. The reaction temperature is important. At temperatures below 75° some sebacoin remains unreduced, while at temperatures above 80° considerable cyclodecane is formed. The submitters report that the reaction run at the reflux temperature gives cyclodecanone in 27% yield and cyclodecane in 32% yield.
5. The product should be isolated and distilled as quickly as possible inasmuch as the unreacted sebacoin is readily oxidized to sebacil, which cannot be separated from the cyclodecanone by simple distillation.
6. The residual zinc may be pyrophoric.
7. For efficient separation of cyclodecanone from cyclodecane, a 60-cm. column of the simple Podbielniak type³ may be used. Removal of sebacil cannot be accomplished readily by fractional distillation, since cyclodecanone and sebacil have virtually identical boiling points.
8. Cyclodecanone regenerated from its semicarbazone, m.p. 203.5–205.5°, has n_D^{25} 1.4806.

3. Discussion

The procedure described is a modification of the directions of Prelog, Frenkiel, Kobelt, and Barman.⁴ [Cyclodecanone](#) has been prepared by the dehydration of sebacoin followed by catalytic hydrogenation,⁵ by the pyrolysis of the thorium or yttrium salt of nonane-1,9-dicarboxylic acid,⁶ and by the ring enlargement of [cyclononanone](#),⁷ as well as by the reduction of sebacoin.⁸

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 5, 277](#)

References and Notes

1. Massachusetts Institute of Technology, Cambridge 39, Massachusetts.
 2. Brown and Borkowski, *J. Am. Chem. Soc.*, **74**, 1901 (1952).
 3. Cason and Rapoport, *Laboratory Text in Organic Chemistry*, 2nd ed., p. 294, Prentice-Hall, Englewood Cliffs, New Jersey, 1962.
 4. Prelog, Frenkiel, Kobelt, and Barman, *Helv. Chim. Acta*, **30**, 1741 (1947).
 5. Stoll, *Helv. Chim. Acta*, **30**, 1837 (1947).
 6. Ruzicka, Stoll, and Schinz, *Helv. Chim. Acta*, **9**, 249 (1926); **11**, 670 (1928).
 7. Kohler, Tishler, Potter, and Thompson, *J. Am. Chem. Soc.*, **61**, 1057 (1939).
 8. Blomquist, Burge, and Sucsy, *J. Am. Chem. Soc.*, **74**, 3636 (1952).
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Appendix

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

Sebacoin

sebacoin-sebacil

thorium or yttrium salt of nonane-1,9-dicarboxylic acid

[hydrochloric acid](#) (7647-01-0)

[acetic acid](#) (64-19-7)

[ether](#) (60-29-7)

[sodium chloride](#) (7647-14-5)

[sodium carbonate](#) (497-19-8)

[zinc](#) (7440-66-6)

[Pentane](#) (109-66-0)

[magnesium sulfate](#) (7487-88-9)

[Cyclodecanone](#) (1502-06-3)

[cyclodecane \(293-96-9\)](#)

[cyclononanone \(3350-30-9\)](#)

[Sebacil \(96-01-5\)](#)