



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](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.

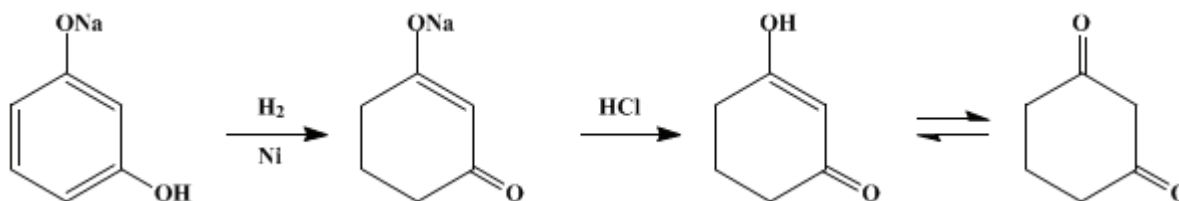
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 3, p.278 (1955); Vol. 27, p.21 (1947).*

## DIHYDRORESORCINOL

### [1,3-Cyclohexanedione]



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Checked by Nathan L. Drake, G. Forrest Woods, and I. W. Tucker.

### 1. Procedure

A solution of 24 g. (0.6 mole) of sodium hydroxide, 100 ml. of water, and 55 g. (0.5 mole) of resorcinol is placed in an apparatus for high-pressure hydrogenation together with 10 g. of reduced Universal Oil Products hydrogenation catalyst (Note 1) or Raney nickel (see p. 181). The pressure in the bomb is raised to 1000–1500 lb. with hydrogen, and the temperature is adjusted to 50° (Note 2). The bomb is shaken and the reaction allowed to proceed for 10 to 12 hours, during which time 0.5 mole of hydrogen is absorbed (Note 3).

The apparatus is allowed to cool to room temperature, the pressure is released, and the catalyst is removed by filtration. The filtrate is made acid to Congo red with concentrated hydrochloric acid, and the solution is cooled to 0° in an ice-salt bath and held at that temperature for 30 minutes before filtration. The dihydroresorcinol which crystallizes is separated by filtration and dried; 50–60 g. of crude dry product containing sodium chloride is obtained.

The crude dihydroresorcinol is dissolved in 125–150 ml. of hot benzene, filtered to remove the sodium chloride, and allowed to crystallize. The solid is separated by filtration and dried overnight in a vacuum desiccator. The product melts at 103–104° and weighs 48–53 g. (85–95%) (Note 4).

### 2. Notes

1. The Universal Oil Products hydrogenation catalyst consists of a mixture of nickel, nickel oxides, and kieselguhr compressed into pills containing 50–55% nickel. Before use in a liquid-phase hydrogenation of this sort, the catalyst must be reduced in a stream of hydrogen at 430°. The reduced catalyst is cooled in the stream of hydrogen and may be kept under alcohol. It also may be saturated with carbon dioxide and kept in a sealed bottle. The pillated material should be pulverized before use in this preparation. The pelleted Universal Oil Products hydrogenation catalyst may be purchased from Universal Oil Products Company, 310 S. Michigan Ave., Chicago 4, Illinois.

2. It is particularly important that the temperature should not rise above 50°; at higher temperatures complex condensation products result.

3. It is important that hydrogenation be complete; incompletely hydrogenated material yields an oily product which is exceedingly difficult to crystallize. The usual time of hydrogen absorption is 4 to 5 hours with Universal Oil Products catalyst and about 4 hours with Raney nickel. The extra time mentioned in the procedure avoids any chance of incomplete reduction.

4. Dihydroresorcinol is unstable; it can be stored only a short time. If it is not used immediately, it should be stored under an inert gas in a brown bottle in a refrigerator.

### 3. Discussion

Dihydroresorcinol has been prepared by the reduction of resorcinol with sodium amalgam,<sup>1</sup> by reduction of hydroxyhydroquinone or its carboxylic acid with sodium amalgam,<sup>2</sup> by hydrolysis of its

dioxime,<sup>3</sup> or by cyclization of ethyl  $\gamma$ -acetylbutyrate.<sup>4</sup> The present method of preparation is essentially that of Klingenfuss.<sup>5</sup>

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 5, 539](#)
- [Org. Syn. Coll. Vol. 5, 743](#)

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## References and Notes

1. Merling, *Ann.*, **278**, 28 (1894).
  2. Thiele and Jaeger, *Ber.*, **34**, 2841 (1901).
  3. Kötze and Grethe, *J. prakt. Chem.*, (2) **80**, 502 (1909).
  4. Vorländer, *Ann.*, **294**, 270 (1897); Schilling, *Ann.*, **308**, 190 (1899).
  5. Klingenfuss (to Hoffmann-La Roche, Inc.), U. S. pat. 1,965,499 [*C. A.*, **28**, 5476 (1934)].
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## Appendix

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

nickel oxides

kieselguhr

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

[Benzene \(71-43-2\)](#)

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

[sodium hydroxide \(1310-73-2\)](#)

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

[carbon dioxide \(124-38-9\)](#)

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

[sodium \(13966-32-0\)](#)

[hydroxyhydroquinone \(533-73-3\)](#)

[resorcinol \(108-46-3\)](#)

[Dihydroresorcinol](#)

[1,3-Cyclohexanedione \(504-02-9\)](#)

ethyl  $\gamma$ -acetylbutyrate (13984-57-1)

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