



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.

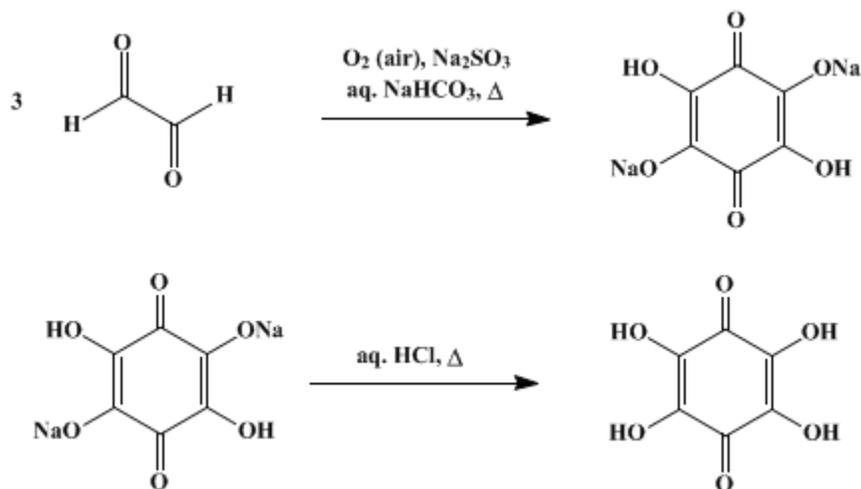
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.*

*Organic Syntheses, Coll. Vol. 5, p.1011 (1973); Vol. 42, p.90 (1962).*

## TETRAHYDROXYQUINONE

[Quinone, tetrahydroxy-]



Submitted by A. J. Fatiadi and W. F. Sager<sup>1</sup>.

Checked by B. C. McKusick and J. K. Williams.

### 1. Procedure

A 5-l., three-necked round-bottomed flask is fitted with a thermometer, an air-inlet tube of 10-mm. diameter extending to within approximately 1 cm. of the bottom, and an outlet tube connected to an aspirator (Note 1). A solution of 400 g. (3.17 moles) of anhydrous sodium sulfite and 150 g. (1.79 moles) of anhydrous sodium bicarbonate (Note 2) in 3 l. of water is heated to 40–45° in the flask. Six hundred grams (480 ml., 3.11 moles) of 30% glyoxal solution (Note 3) is added, and a brisk stream of air is drawn through the solution for 1 hour without application of heat. Within a few minutes, greenish black crystals of the sodium salt of tetrahydroxyquinone begin to separate. The flask is warmed to between 80° and 90° over a period of an hour. The air current is then stopped, and the mixture is heated to incipient boiling and set aside for 30 minutes. It is then cooled to 50° (Note 4), and the sodium salt of tetrahydroxyquinone is separated by filtration and washed successively with 50 ml. of cold 15% sodium chloride solution, 50 ml. of cold 1:1 methanol-water, and 50 ml. of methanol. The air-dried salt weighs 20–21 g.

The salt is added to 250 ml. of 2*N* hydrochloric acid, and the mixture is heated to incipient boiling. The resultant solution is cooled in an ice bath, and the glistening black crystals of tetrahydroxyquinone that precipitate are collected on a Büchner funnel and washed with ice water to give 11–15 g. (6.2–8.4%) of product. The quinone fails to melt on a hot plate at 320° (Note 5).

### 2. Notes

1. A tube of 10-mm. diameter is necessary to prevent the clogging of the outlet that occurs if tubing of smaller diameter is used.
2. Equivalent amounts of hydrated salt may be used.
3. Dow commercial grade 30% glyoxal solution is satisfactory.
4. It is not necessary to cool below this temperature since crystallization is essentially complete at 50°.
5. This material is pure enough for reduction to hexahydroxybenzene<sup>2</sup> and most other purposes. A purer product can be obtained by dissolving the crude tetrahydroxyquinone in acetone and adding petroleum ether of b.p. 60–80° to precipitate it.

### 3. Discussion

The procedure employed for [tetrahydroxyquinone](#) is based on an observation by Homolka.<sup>3</sup> [Tetrahydroxyquinone](#) may also be prepared by treatment of the [glyoxal-bisulfite](#) addition compound with [sodium carbonate](#)<sup>3</sup> or [magnesium hydroxide](#) and [potassium cyanide](#)<sup>4</sup> or by treatment of 50% [glyoxal](#) with [sodium hydrosulfite](#).<sup>5</sup>

### 4. Merits of the Preparation

[Tetrahydroxyquinone](#) is of interest because of its application in analytical chemistry to the determination of [barium](#) and as a complexing agent for many ions.<sup>6</sup> Moreover, it serves as a convenient source not only of the reduction product [hexahydroxybenzene](#)<sup>2</sup> but also of the oxidation products [rhodizonic acid](#) and triquinoyl and of the product of catalytic reduction, *meso*-inositol.

This procedure serves as a particularly simple method for preparing [tetrahydroxyquinone](#). The low yield is more than offset by the simplicity of the set-up, the ease of manipulation, and the low cost and ready availability of the starting materials.

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 5, 595](#)

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### References and Notes

1. Department of Chemistry, The George Washington University, Washington, D. C.
  2. [A. J. Fatiadi and W. F. Sager, this volume, p. 595.](#)
  3. B. Homolka, *Ber.*, **54**, 1393 (1921).
  4. R. Kuhn, G. Quadbeck, and E. Röhm, *Ann.*, **565**, **1** (1949).
  5. B. Eistert and G. Bock, *Angew. Chem.*, **70**, 595 (1958).
  6. J. H. Yoe and L. A. Sarver, "Organic Analytical Reagents," John Wiley and Sons, Inc., New York, 1941 (out of print); S. J. Kocher, *Ind. Eng. Chem., Anal. Ed.*, **9**, 331 (1937).
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### Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[petroleum ether](#)

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

[methanol \(67-56-1\)](#)

[glyoxal \(107-22-2\)](#)

[sodium sulfite \(7757-83-7\)](#)

[sodium bicarbonate \(144-55-8\)](#)

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

sodium carbonate (497-19-8)  
potassium cyanide (151-50-8)  
sodium hydrosulfite (7775-14-6)  
acetone (67-64-1)  
magnesium hydroxide  
barium (7440-39-3)  
Quinone (106-51-4)  
glyoxal-bisulfite (18381-20-9)  
Hexahydroxybenzene (608-80-0)  
Tetrahydroxyquinone,  
Quinone, tetrahydroxy- (319-89-1)  
rhodizonic acid  
sodium salt of tetrahydroxyquinone  
meso-inositol (87-89-8)