



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.

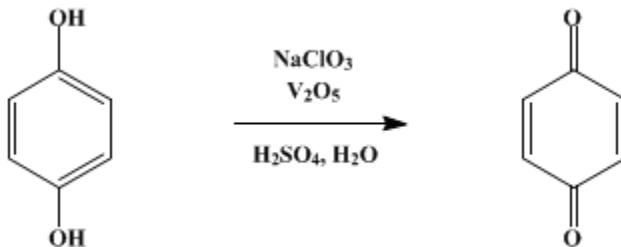
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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. 2, p.553 (1943); Vol. 16, p.73 (1936).

QUINONE



Submitted by H. W. Underwood, Jr. and W. L. Walsh.
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1. Procedure

In a 2-l. round-bottomed flask equipped with a mechanical stirrer are placed 1 l. of 2 per cent sulfuric acid, 0.5 g. of vanadium pentoxide (Note 1), 110 g. (1 mole) of hydroquinone, and 60 g. (0.56 mole) of sodium chlorate. The mixture is vigorously stirred for about three hours, when the green quinhydrone first formed is converted to yellow quinone. The temperature of the reaction mixture rises to about 40° (Note 2). The reaction is usually complete in three and one-half to four hours. The flask is then disconnected and cooled under the tap, the mixture is filtered with suction, and the quinone is washed once with about 100 cc. of cold water. After drying in a desiccator over calcium chloride, the product weighs 86–90 g. and melts at 110–112°. This material is pure enough for most purposes. Extraction of the filtrate and washings with four 100-cc. portions of benzene yields a further 12–14 g. of quinone and brings the total amount to 99–104 g. (92–96 per cent of the theoretical yield) (Note 3).

Very pure quinone can be obtained either by vacuum sublimation or recrystallization from boiling ligroin (b.p. 90–120°). One hundred grams of the crude quinone requires about 1.2 l. of ligroin for recrystallization and yields 92–97 g. of bright yellow quinone, m.p. 111–113°.

2. Notes

1. Vanadium pentoxide may be obtained from the Vanadium Corporation of America or prepared from ammonium metavanadate as described in Note 2, p. 302.
2. If quantities larger than one mole of hydroquinone are used the temperature should not be allowed to rise above 40°.
3. By the use of a suitable organic solvent the same oxidizing agent can be employed for the preparation of anthraquinone. A mixture of 90 g. (0.51 mole) of finely powdered pure anthracene, 0.5 g. of vanadium pentoxide, 76 g. of sodium chlorate, 1 l. of glacial acetic acid, and 200 cc. of 2 per cent sulfuric acid is warmed under reflux until a vigorous reaction commences. The source of heat is removed, and the reaction allowed to proceed for about twenty minutes. The mixture is refluxed for one hour longer and then cooled in ice. The light yellow solid is filtered with suction, washed well with water, and dried at 110°. The yield is 92–96 g. (88–91 per cent of the theoretical amount) of a product melting at 273–275°.

The sodium chlorate-vanadium pentoxide mixture is not very powerful, and, although it attacks easily the particularly reactive anthracene, it is not suitable for the conversion of hydrocarbons of the naphthalene and phenanthrene series into the corresponding quinones or for the oxidation of acenaphthene or fluorene (observation of the checkers).

3. Discussion

The oxidation of hydroquinone by means of sodium dichromate in sulfuric acid solution and references to other methods of preparation are given in an earlier volume of this series.¹

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 62
- Org. Syn. Coll. Vol. 2, 539

References and Notes

1. Org. Syn. Coll. Vol. I, 1941, 482.
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

ligroin

quinhydrone

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

Benzene (71-43-2)

hydroquinone (123-31-9)

ammonium metavanadate (7803-55-6)

vanadium pentoxide

Anthraquinone (84-65-1)

Naphthalene (91-20-3)

sodium dichromate (7789-12-0)

sodium chlorate (7775-09-9)

anthracene (120-12-7)

Quinone (106-51-4)

fluorene (86-73-7)

phenanthrene (85-01-8)

sodium chlorate-vanadium pentoxide

acenaphthene (83-32-9)

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