



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.

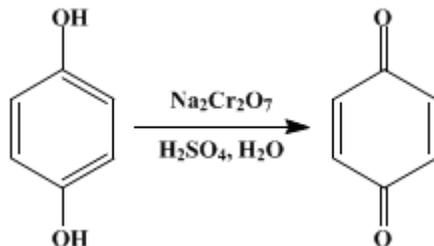
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.482 (1941); Vol. 2, p.85 (1922).

QUINONE



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1. Procedure

In a 2.5-l. beaker, 100 g. (0.91 mole) of [hydroquinone](#) is dissolved in 2 l. of water heated to about 50°. After the solid is completely dissolved, the solution is cooled to 20°, 100 g. (54.4 cc.) of concentrated [sulfuric acid](#) is slowly poured in, and the mixture is again cooled to 20° ([Note 1](#)). A concentrated solution of technical [sodium dichromate](#) is prepared by dissolving 140 g. (0.47 mole) in 65 cc. of water ([Note 2](#)). This solution is then added gradually to the [hydroquinone](#) solution, with the use of a mechanical stirrer ([Note 3](#)), the mixture being cooled so that the temperature never rises above 30°. At first a greenish-black precipitate forms, but, upon further addition of the [sodium dichromate](#) solution, the color changes to yellowish green. As soon as this color remains permanent (a slight excess of [sodium dichromate](#) does no harm) the reaction is complete. This requires about one-half to three-quarters of an hour; 90–110 cc. of [sodium dichromate](#) solution is necessary. The mixture is then cooled to about 10° and filtered with suction. As much water as possible is pressed out of the crystals.

The filtrate is extracted twice, 150 cc. of [benzene](#) being used for each extraction. The precipitate of [quinone](#) is transferred to a 1-l. beaker, and 500 cc. of [benzene](#), including the 300 cc. used to extract the filtrate, is added. The mixture is now heated with stirring on a steam bath, and as soon as most of the [quinone](#) has dissolved the [benzene](#) layer is decanted into another beaker. It is dried while hot by stirring a short time with a little [calcium chloride](#), and then filtered through an ordinary funnel into a 1-l. distilling flask before it cools. There is a certain amount of [quinone](#) which does not go into the 500 cc. of [benzene](#), so that the residue is extracted a second time with about 100 cc. of [benzene](#), which is dried and filtered with the first extract. During these extractions ([Note 4](#)), the [benzene](#) should not be at the boiling point, as this will cause a considerable volatilization of the [quinone](#).

The distilling flask is now attached to a condenser set for downward distillation, and the [benzene](#) is distilled. As soon as the [quinone](#) starts to separate, the residue in the flask is transferred to a beaker and cooled in an ice bath. The precipitate is filtered off with suction and the product spread out for a short time to dry. The product is yellow in color and weighs 75 to 80 g. (76–81 per cent of the theoretical amount). Material made in this way will hold its yellow color over long periods of time, provided it is protected from light.

The [benzene](#) distillate is yellow and contains some [quinone](#). This, as well as the [benzene](#) from the final filtration of the [quinone](#) crystals, may be used in a subsequent run and thus raises the yield of the subsequent runs to about 85–90 g. (86–92 per cent of the theoretical amount) ([Note 5](#)).

2. Notes

1. If impure [hydroquinone](#) is used, a black, sticky precipitate will usually appear after the addition of the [sulfuric acid](#) to the [hydroquinone](#) solution. This should be removed, before the oxidation is started, by filtration without suction through a fluted filter.
2. When technical [sodium dichromate](#) is used, the solution should be filtered with suction, before it is added to the [hydroquinone](#), in order to remove any insoluble impurities.

3. As the mixture becomes thick during the oxidation, it is very necessary to use a stirrer which will keep the whole mass agitated by reaching to the sides and bottom of the beaker.
4. In the laboratory it is convenient to make several small runs of the size indicated, as far as the oxidation is concerned; but the [benzene](#) extractions can be combined.
5. It is also possible to obtain good yields of [quinone](#) in the following manner: 1500 cc. of water, 465 g. of concentrated [sulfuric acid](#), and 300 g. (2.7 moles) of [hydroquinone](#) are mixed in a 3-l. beaker. The mixture is cooled to 0°, and 330 g. (1.1 moles) of [sodium dichromate](#) is added in powdered form, the temperature being kept below 5° at all times. This method requires a longer time and much more care in the control of conditions than the procedure described.

3. Discussion

[Quinone](#) can be prepared by the oxidation of [benzene](#),¹ [aniline](#),² and [hydroquinone](#)³ by a miscellany of oxidizing agents. The oxidation of [hydroquinone](#) is rapid and convenient and therefore desirable for the laboratory.

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 1, 317](#)
- [Org. Syn. Coll. Vol. 2, 553](#)

References and Notes

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2. Hofmann, Jahresber. 415 (1863); Nietzki, Ber. **10**, 1934 (1877), Ber. **19**, 1468 (1886); Ann. **215**, 127 (1882); Liebermann, Ber. **10**, 2005 (1877); Hesse, Ann. **200**, 240 (1880); Seyda, Ber. **16**, 687 (1883); Schniter, Ber. **20**, 2283 (1887); Bamberger and Tschirner, Ber. **31**, 1524 (1898); Darmstädter, Ger. pat. 109,012 [Frld. **5**, 664 (1897–1900)]; Boehringer and Sons, Ger. pat. 117,129 [Frld. **6**, 112 (1900–02)]; Perkin, J. Soc. Dyers and Colourists, **36**, 138 (1920).
3. Wöhler, Ann. **51**, 152 (1844); Clark, Am. Chem. J. **14**, 555 (1892); Craven and Duncan, J. Chem. Soc. **127**, 1489 (1925); McCoy, J. Chem. Education, **14**, 494 (1937).

Appendix

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

[calcium chloride](#) (10043-52-4)

[sulfuric acid](#) (7664-93-9)

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

[aniline](#) (62-53-3)

[hydroquinone](#) (123-31-9)

sodium dichromate (7789-12-0)

Quinone (106-51-4)

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