Organic Syntheses

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.

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.

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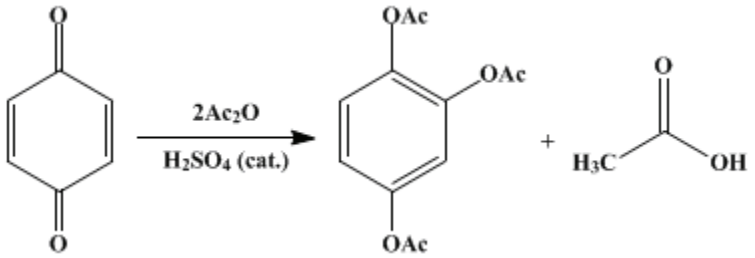
HYDROXYHYDROQUINONE TRIACETATE

[1,2,4-Benzenetriol, triacetate]



Submitted by E. B. Vliet
Checked by Roger Adams and H. O. Calvery.

1. Procedure

Twelve grams of concentrated sulfuric acid is added to 180 g. (167 cc., 1.8 moles) of acetic anhydride in a 600-cc. beaker, after which 60 g. (0.55 mole) of quinone (Note 1) is added gradually in small portions with constant mechanical stirring. The temperature rises and should be held at 40–50° during the addition of the quinone. A temperature higher than 50° leads to decomposition of the product whereas a temperature lower than 40° causes a much slower reaction. Good temperature control can be obtained by using care in adding the quinone and by the use of a cooling bath and efficient stirring. The reaction goes smoothly and does not evolve an excessive amount of heat.

After all the quinone has been added, the solution is allowed to stand. It should be watched closely at first to see that the temperature does not rise above 50°, but at the same time it is well not to cool it below 40°. When the mixture starts to cool of its own accord, a precipitate begins to form. As soon as the mixture has cooled to about 25° (Note 2), it is poured into 750 cc. of cold water, whereupon a white precipitate separates. The mixture is cooled to about 10° and filtered with suction. The product is recrystallized from 250 cc. of 95 per cent ethyl alcohol and dried. The drying is best done in a vacuum desiccator, through which a very small stream of air is allowed to sweep. Calcium chloride is the best drying agent.

The product is practically white and rather granular; it melts at 96–97°. The yield is 120–123 g. (86–87 per cent of the theoretical amount). The product is fairly reactive and tends to decompose upon standing (Note 3), especially if care is not used in drying and handling. If it is to be used as a starting material for other syntheses, it is advisable to use it as soon as possible.

2. Notes

1. The quality of the product depends to a large extent upon the purity of the quinone; if the latter is dark, it is advisable to crystallize it from benzene before using it. Directions for the preparation and purification of quinone are given on p. 482.
2. It is quite desirable to have the reaction mixture cooled to about 25° before it is added to the water. If it is warm when added to the water, an oil will separate which solidifies slowly in the form of lumps.
3. The hydroxyhydroquinone triacetate keeps quite satisfactorily in an atmosphere of carbon dioxide.

3. Discussion

Hydroxyhydroquinone triacetate can be prepared by acetylating hydroxyhydroquinone formed by the alkali fusion of hydroquinone,[1] and by the procedure described which is essentially that of Thiele.[2]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 360

References and Notes

1. Barth and Schreder, Monatsh. **5**, 590 (1884).
2. Thiele, Ber. **31**, 1247 (1898); Bayer and Co., Ger. pat. 101,607 and 107,508 [Frdl. **5**, 155, 156 (1901)]; Bargellini and Avrutin, Gazz. chim. ital. **40** (2), 349 (1910).

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

ethyl alcohol (64-17-5)

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

Benzene (71-43-2)

acetic anhydride (108-24-7)

hydroquinone (123-31-9)

carbon dioxide (124-38-9)

Hydroxyhydroquinone triacetate,
1,2,4-Benzenetriol, triacetate (613-03-6)

Quinone (106-51-4)

hydroxyhydroquinone (533-73-3)