

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 accessed of charge text can be free 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. 3, p.452 (1955); Vol. 28, p.68 (1948).

HYDROQUINONE DIACETATE

[Hydroquinone, diacetate]



Submitted by W. W. Prichard Checked by Homer Adkins and Maynette Vernsten.

1. Procedure

One drop of concentrated sulfuric acid is added to a mixture of 110 g. (1.0 mole) of hydroquinone and 206 g. (190.3 ml., 2.02 moles) of acetic anhydride (Note 1) in a 1-l. Erlenmeyer flask. The mixture is stirred gently by hand; it warms up very rapidly, and the hydroquinone dissolves. After 5 minutes the clear solution is poured onto about 800 ml. of crushed ice. The white crystalline solid which separates is collected on a Büchner filter and washed with 1 l. of water. The filter cake is pressed occasionally to facilitate the removal of water; the solid is dried to constant weight over phosphorus pentoxide in a vacuum desiccator. The nearly pure product weighs 186–190 g. (96–98%) and melts at 121–122° (Note 2); it can be recrystallized from dilute ethanol (Note 3).

2. Notes

1. The use of commercial acetic anhydride in this preparation sometimes results in appreciably lower yields. The checkers used the freshly redistilled reagent.

2. The melting point is recorded in the literature^{1,2} as 121° and as $123-124^{\circ}$.

3. Recrystallization from 50% ethanol (by weight) permits a 93–94% recovery of material melting at 121.5–122.5°; about 365 g. (400 ml.) of the solvent is required for 100 g. of the crude product.

3. Discussion

Hydroquinone diacetate has been prepared by the treatment of hydroquinone with acetic anhydride, both in the presence^{1,3,4} and in the absence^{5,6,7} of strong acid catalysts, by the treatment of the sodium salt of hydroquinone with acetic anhydride, and by the reaction of hydroquinone with acetic anhydride in the presence of sodium acetate.^{8,9} It has also been prepared from hydroquinone and acetyl chloride;¹⁰ the acetylation with acetyl chloride is reported to be improved by the addition of metallic magnesium.¹¹

This preparation is referenced from:

• Org. Syn. Coll. Vol. 3, 280

References and Notes

- 1. Ciusa and Sollazzo, Chem. Zentr., 114, II, 615 (1943).
- 2. Chattaway, J. Chem. Soc., 1931, 2495.
- **3.** Henle, *Ann.*, **350**, 344 (1906).
- 4. Reychler, Chem. Zentr., 79, I, 1042 (1908).
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- 6. Hesse, Ann., 200, 244 (1880).
- 7. Kaufmann, Ber., 42, 3482 (1909).
- 8. Sarauw, Ann., 209, 128 (1881).

- 9. Buchka, Ber., 14, 1327 (1881).
- 10. Nietzki, Ber., 11, 470 (1878).
- **11.** Spasov, Ann. univ. Sofia II, Faculté phys.-math., livre 2, **35**, 289 (1938–1939) [C. A., **34**, 2343 (1940)].

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

ethanol (64-17-5)

sulfuric acid (7664-93-9)

acetic anhydride (108-24-7)

sodium acetate (127-09-3)

acetyl chloride (75-36-5)

hydroquinone (123-31-9)

magnesium (7439-95-4)

Hydroquinone diacetate, Hydroquinone, diacetate (1205-91-0)

phosphorus pentoxide (1314-56-3)

sodium salt of hydroquinone

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