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.

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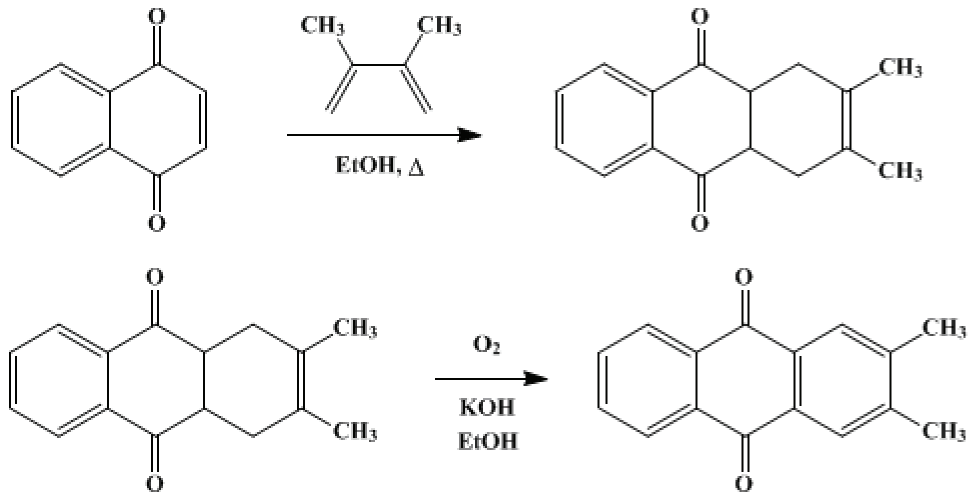
Organic Syntheses, Coll. Vol. 3, p.310 (1955); Vol. 22, p.37 (1942).

2,3-DIMETHYLANTHRAQUINONE

[Anthraquinone, 2,3-dimethyl-]



Submitted by C. F. H. Allen and Alan Bell.
Checked by R. L. Shriner and John C. Robinson, Jr..

1. Procedure

A solution of 80 g. (0.5 mole) of 1,4-naphthoquinone [*Org. Syntheses* Coll. Vol. **1**, 375 (1932), 383 (1941)] and 80 g. (1 mole) of 2,3-dimethylbutadiene-1,3 (p. 312) in 300 ml. of alcohol is refluxed for 5 hours, using a 1-l. round-bottomed flask and an efficient reflux condenser. The solution is cooled and placed in a refrigerator for 10–12 hours. The crystalline mass is then broken up with a spatula, and the addition product is filtered and washed with 50 ml. of cold ethanol. The product forms white, feathery crystals melting at 147–149° (Note 1). The yield is 116 g. (96% based on the 1,4-naphthoquinone).

For the dehydrogenation, 40 g. of the addition product is dissolved in 600 ml. of 5% ethanolic potassium hydroxide solution (Note 2) in a 1-l. three-necked flask equipped with a reflux condenser and inlet tube. A current of air is bubbled through the solution for 24 hours; considerable heat is generated, and the initial green color soon changes to yellow. The yellow quinone that has separated is then filtered with suction and is washed, first with 200 ml. of water, then with 100 ml. of ethanol, and finally with 50 ml. of ether. The yield of air-dried product (m.p. 209–210°) is 36.5–37.5 g. (94–96%) (Note 3). The over-all yield for both steps is 90% (Note 4).

2. Notes

1. The addition product is usually pure enough for the next step. It may be purified by recrystallization from acetone, ethanol, or methanol, and then it melts sharply at 150°. If the crude product is deeply colored, it should be recrystallized, using a decolorizing carbon.
2. This is prepared by dissolving 30 g. of potassium hydroxide in 570 g. of 95% ethanol.
3. The melting points given in the literature vary from 183°[1] to 208°.[2]
4. Essentially the same percentage yield has been obtained using three times the amounts given.

3. Discussion

2,3-Dimethylanthraquinone has been obtained by ring closure of the corresponding *o*-benzoylbenzoic acid;[1,2,3] by oxidation of the corresponding anthrone;[4] by decarboxylation of 2,3-dimethylanthraquinone-5-carboxylic acid;[5] from 2-chloro- and 2,3-dichloro-1,4-naphthoquinone and 2,3-dimethylbutadiene-1,3 by the action of sodium hydroxide;[6] and from 2-methyl-1,4-naphthoquinone and 2,3-dimethylbutadiene-1,3 with subsequent dehydrogenation by sulfur.[7] The addition product employed in the present procedure has been described.[8]

References and Notes

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2. Heller, *Ber.*, **43**, 2891 (1910).
3. Fairbourne, *J. Chem. Soc.*, **119**, 1573 (1921).
4. Limpricht and Martens, *Ann.*, **312**, 103 (1900).
5. Fieser and Newton, *J. Am. Chem. Soc.*, **64**, 917 (1942).
6. Ger. pat. 500,160 [*Frdl.*, **17**, 1143 (1932); *C. A.*, **24**, 4790 (1930)].
7. Fieser and Seligman, *J. Am. Chem. Soc.*, **56**, 2690 (1934).
8. Fieser and Webber, *J. Am. Chem. Soc.*, **62**, 1362 (1940).

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

2-chloro- and 2,3-dichloro-1,4-naphthoquinone

ethanol (64-17-5)

methanol (67-56-1)

ether (60-29-7)

sodium hydroxide (1310-73-2)

sulfur (7704-34-9)

Anthrone (90-44-8)

acetone (67-64-1)

decolorizing carbon (7782-42-5)

potassium hydroxide (1310-58-3)

Quinone (106-51-4)

1,4-Naphthoquinone (130-15-4)

2,3-Dimethylanthraquinone,
Anthraquinone, 2,3-dimethyl- (6531-35-7)

2,3-dimethylbutadiene-1,3 (513-81-5)

2,3-dimethylanthraquinone-5-carboxylic acid

2-methyl-1,4-naphthoquinone

o-benzoylbenzoic acid (85-52-9)