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.

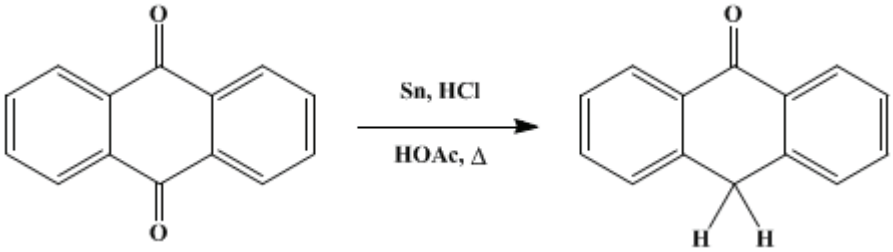
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.

DOI:10.15227/orgsyn.008.0008

Organic Syntheses, Coll. Vol. 1, p.60 (1941); Vol. 8, p.8 (1928).

ANTHRONE



Submitted by Kurt H. Meyer
Checked by J. B. Conant and W. C. Boyd.

1. Procedure

In a 2-l. round-bottomed flask with a reflux condenser, 104 g. (0.5 mole) of anthraquinone, m.p. 276–280° (corr.), is mixed with 100 g. (0.86 atom) of granulated tin, and 750 cc. of glacial acetic acid is added. The contents of the flask are heated to boiling, and in the course of two hours, 298 g. (250 cc., 8.2 moles) of c.p. hydrochloric acid (sp. gr. 1.19) is added in 10-cc. portions to the boiling mixture. At the end of this time all the anthraquinone should have gone into solution; if not, more tin and hydrochloric acid are added.

The liquid is filtered with suction through a Gooch crucible with a fixed porous plate (Note 1), and 100 cc. of water is added. The anthrone crystallizes when the solution is cooled to about 10°. The crystals are filtered with suction on a Büchner funnel and washed with water. After drying on a porous plate the melting point of the material is about 153°. The yield is 80 g. (82.5 per cent of the theoretical amount). On recrystallization from a 3:1 mixture (Note 2) of benzene and petroleum ether about 60 g. of anthrone melting at 154–155° (corr.) is obtained (62 per cent of the theoretical amount).

2. Notes

1. The liquid can also be filtered through a fluted filter paper, but this is slower.
2. The proportions do not make much difference as far as the yield is concerned, but the substance is more soluble in mixtures rich in benzene. About 10–12 cc. of the 3:1 mixture is required for each gram of anthrone. The anthrone may be more readily dissolved if it is added to the estimated quantity of hot benzene on the steam bath, and the petroleum ether is then added. About two-thirds of the mother liquor may be distilled off through a condenser and used in later runs. The residual mother liquor deposits about 12 g. of rather impure anthrone.

3. Discussion

Anthrone can be prepared by the reduction of anthraquinone with tin and hydrochloric acid,[1] and with aluminum bronze,[2] or by cyclization of *o*-benzylbenzoic acid with liquid hydrogen fluoride.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 390

References and Notes

1. Meyer, Ann. **379**, 55 (1911).
2. Eckert and Pollak, Monatsh. **38**, 12 (1917).
3. Fieser and Hershberg, J. Am. Chem. Soc. **61**, 1278 (1939).

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

aluminum bronze

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

Benzene (71-43-2)

tin (7440-31-5)

Anthrone (90-44-8)

Anthraquinone (84-65-1)

hydrogen fluoride (7664-39-3)

o-benzylbenzoic acid (612-35-1)