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.

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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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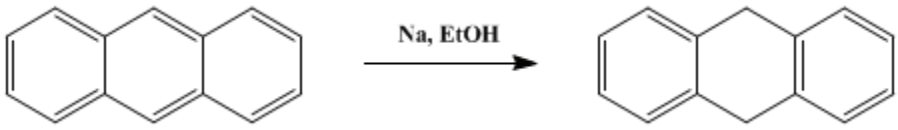
9,10-DIHYDROANTHRACENE

[Anthracene, 9,10-dihydro-]



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1. Procedure

In a 2-l., three-necked, round-bottomed flask fitted with a rubber-tube sealed mechanical glass stirrer, a reflux condenser (Note 1), and a thermometer reaching to the bottom of the flask are placed 50 g. (0.28 mole) of anthracene (Note 2) and 750 ml. of commerical absolute ethanol. The suspension obtained is stirred and heated (Note 3) to 50°, and 75 g. (3.25 g. atom) of freshly cut sodium is added in quantities of about 10 g. each to the stirred mixture over a period of 5 minutes. The reaction mixture boils vigorously (Note 4) and stirring is continued for 15 minutes longer. The reaction mixture is then cooled and carefully diluted with 1 l. of water. The white-yellow solid which separates is a mixture of 9,10-dihydroanthracene and anthracene, and it is collected on a Büchner funnel, washed with 400 ml. of water, and dried in air.

The dry white-yellow solid is suspended in 500 ml. of commercial absolute ethanol in a 1-l., three-necked, round-bottomed flask fitted with a rubber-tube sealed mechanical glass stirrer, a reflux condenser (Note 1), and a thermometer reaching to the bottom of the flask. The suspension is stirred and heated (Note 3) to 50°, and 50 g. (2.17 g. atom) of freshly cut sodium is added in quantities of about 10 g. each to the stirred mixture over a period of 5 minutes. The reaction mixture boils vigorously (Note 4), and stirring is continued for an additional 15 minutes. The reaction mixture is then cooled and carefully diluted with 750 ml. of water. The white solid which separates is 9,10-dihydroanthracene, and it is collected on a Büchner funnel, washed with 300 ml. of water, and dried in air. It is recrystallized from ethanol (about 250–300 ml. of solvent is required), and the crystals are collected on a Büchner funnel, washed with 20 ml. of cold ethanol, and dried in air. The yield of dry 9,10-dihydroanthracene in the form of broad, colorless needles, m.p. 108–109°, is 38–40 g. (75–79%) (Note 5).

2. Notes

1. An efficient 12-in., double-surface, all-glass condenser should be used with an outlet tube carrying the evolved hydrogen into a good hood vent.
2. A purified grade of anthracene (blue fluorescence, m.p. 216°) should be used.
3. An electric heating mantle should be used. No free flames should be present anywhere near the reaction flask.
4. The reaction may be controlled by removing the heat source or slowing down the rate of stirring or both.
5. The 9,10-dihydroanthracene may be purified further by steam distillation from an aqueous suspension followed by recrystallization of the dried product from ethanol.

3. Discussion

The procedure described is adapted from the preparation outlined by Wieland.[2]

4. Merits of Preparation

9,10-Dihydroanthracene has been used as one of the hydrogen transfer reagents in a series of homolytic hydrogen transfer reactions by Braude, Jackman, and Linstead[3] and as a hydrogen donor for

the hydrogenation of thiyl radicals to form thiols.[4]

References and Notes

1. Department of Chemistry, The City University, London, England.
2. H. Wieland, *Ber.* **45**, 492 (1912).
3. E. A. Braude, L. M. Jackman, and R. P. Linstead, *J. Chem. Soc.*, 3548 (1954).
4. A. F. Bickel and E. C. Kooyman, *Nature*, **170**, 211 (1952).

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

ethanol (64-17-5)

hydrogen (1333-74-0)

sodium (13966-32-0)

anthracene (120-12-7)

9,10-Dihydroanthracene,
Anthracene, 9,10-dihydro- (613-31-0)