Organic Syntheses

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.
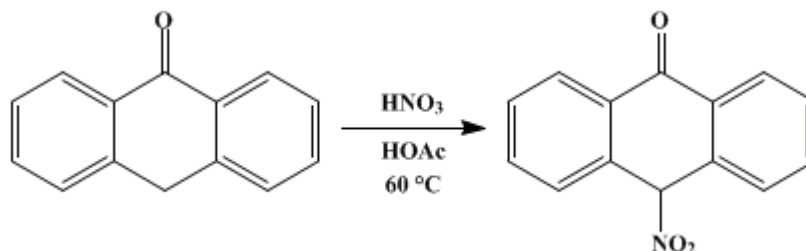
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.390 (1941); Vol. 8, p.78 (1928).

NITROANTHRONE

[Anthrone, 10-nitro-]

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

1. Procedure

In a 1-l. beaker equipped with a separatory funnel, a mechanical stirrer, and a thermometer, 20 g. (0.1 mole) of anthrone (p. 60) is dissolved in 300 cc. of glacial acetic acid. While the mixture is kept at 60° and stirred, a solution of 10.5 g. (7 cc., 0.16 mole) of fuming nitric acid (sp. gr. 1.5) in 50 cc. of glacial acetic acid is run in during one hour.

On cooling to about 10°, 15 g. of nitroanthrone separates out in long yellowish-white needles. After the addition of 100 cc. of water to the mother liquor, 6 g. more, somewhat darker in color, crystallizes in three to four hours.

On recrystallization from about 300 cc. of a 1:1 benzene-petroleum ether (40–60°) mixture, 16.5 g. of nitroanthrone melting at 140° (corr.) is obtained (67 per cent of the theoretical amount) (Note 1).

2. Notes

1. By dissolving in hot alkali (about 300 cc. of water and 30 g. of sodium hydroxide per gram of nitroanthrone) and precipitating with acid below 10°, the red nitroanthranol may be obtained, which on standing slowly changes back to the nitroanthrone.

3. Discussion

Nitroanthrone can be prepared by the nitration of anthracene in isobutyl alcohol,[1] and by the nitration of anthrone.[2]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 445

References and Notes

1. Perkin and Mackenzie, J. Chem. Soc. **61**, 868 (1892); Meisenheimer and Connerade, Ann. **330**, 177 (1904).
2. Meyer and Sander, Ann. **396**, 149 (1913).

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

benzene-petroleum ether

acetic acid (64-19-7)

sodium hydroxide (1310-73-2)

nitric acid (7697-37-2)

Anthrone (90-44-8)

anthracene (120-12-7)

Nitroanthrone

Anthrone, 10-nitro- (6313-44-6)

nitroanthranol

isobutyl alcohol (78-83-1)