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.

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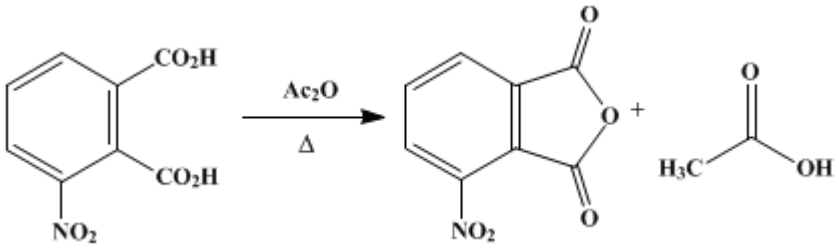
3-NITROPHTHALIC ANHYDRIDE

[Phthalic anhydride, 3-nitro-]



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1. Procedure

To 211 g. (1 mole) of 3-nitrophthalic acid (p. 408) in a 300-cc. round-bottomed flask fitted with a reflux condenser is added 205 g. (190 cc., 2 moles) of acetic anhydride (99–100 per cent). The mixture is heated to gentle boiling until the acid is completely dissolved and then for about ten minutes longer. The hot mixture is poured (Hood) into a 15-cm. porcelain dish and allowed to cool. The crystal mass is ground thoroughly in a mortar and filtered by suction. The crystals are returned to the mortar and the filtrate (about 140 cc.) is placed in a 300-cc. distilling flask. The crystals are ground with 150 cc. of alcohol-free ether (Note 1) and filtered. They are again returned to the mortar and similarly washed.

After drying in the air for a short time, the product is dried to constant weight at 105°. The yield of product melting at 163–164° is 170–180 g. (88–93 per cent of the theoretical amount). The acetic acid filtrate is distilled with a thermometer in the liquid until the temperature is 150°. The distillate amounts to about 120 cc. The residue is poured into the mortar and, after cooling, is ground with some of the ether used in washing the original crystals. Thus about 10 g. of a product melting at 160–163° is obtained.

2. Notes

1. Dry ether is recommended because ordinary ether contains alcohol, and some monoethyl ester may form. Ordinary u.s.p. ether which has stood over calcium chloride for two days is satisfactory.

3. Discussion

3-Nitrophthalic anhydride can be prepared by heating 3-nitrophthalic acid under various conditions[1] and by the action of acetic anhydride,[2] essentially as in the procedure described. The direct nitration of phthalic anhydride yields 3-nitrophthalic anhydride together with the isomeric 4-nitro compound.[3]

References and Notes

1. Kahn, Ber. **35**, 3859 (1902).
2. McKenzie, J. Chem. Soc. **79**, 1137 (1901).
3. Hayashi and Kawasaki, J. Soc. Chem. Ind. Japan **36**, Suppl. binding 121 (1933) [C. A. **27**, 2947 (1933)].

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

alcohol-free ether

alcohol (64-17-5)

calcium chloride (10043-52-4)

acetic acid (64-19-7)

ether (60-29-7)

acetic anhydride (108-24-7)

3-Nitrophthalic acid (603-11-2)

phthalic anhydride (85-44-9)

3-Nitrophthalic anhydride,
Phthalic anhydride, 3-nitro- (641-70-3)