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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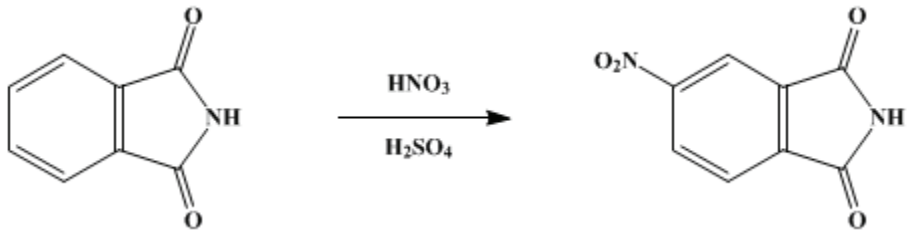
Organic Syntheses, Coll. Vol. 2, p.459 (1943); Vol. 16, p.58 (1936).

4-NITROPHTHALIMIDE

[Phthalimide, 4-nitro-]



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1. Procedure

Two hundred forty cubic centimeters (5.7 moles) of c.p. fuming nitric acid (sp. gr. 1.50) is added to 1.4 l. of concentrated c.p. sulfuric acid (sp. gr. 1.84) in a 3-l. beaker, and the mixture is cooled in an ice bath. As soon as the temperature of the mixed acids reaches 12°, 200 g. (1.36 moles) of commercial phthalimide is stirred in as rapidly as possible while the temperature of the nitrating mixture is kept between 10° and 15°. The reaction mixture is allowed to warm to room temperature in the ice bath as the ice melts, and left overnight.

The clear, pale yellow solution is poured slowly with vigorous stirring onto 4.5 kg. of cracked ice; the temperature of this mixture must not rise above 20°. The crude nitration product is filtered through cloth on a 20-cm. Büchner funnel, using suction, and the mass is pressed as dry as possible. The cake is removed and stirred vigorously with 2 l. of *ice water*. The solid is filtered; the cake is removed and stirred again with 2 l. of *ice water*. This washing is repeated four times. The crude product, after drying in the air, melts at 185–190° and weighs 165–174 g. (63–66 per cent of the theoretical amount). This material is purified by crystallization from 3 to 3.2 l. of 95 per cent ethyl alcohol. This furnishes 136–140 g. (52–53 per cent of the theoretical amount) of 4-nitrophthalimide melting at 198°.

3. Discussion

4-Nitrophthalimide has been prepared from 4-nitrophthalic acid[1] and by nitrating phthalimide.[2] The procedure described is a modification of the method of Levy and Stephen.[2]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 457

References and Notes

1. Seidel, Ber. **34**, 4351 (1901); Seidel and Bittner, Monatsh. **23**, 420 (1902); Bogert and Renshaw, J. Am. Chem. Soc. **30**, 1137 (1908).
2. Levy and Stephen, J. Chem. Soc. **1931**, 79.

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

ethyl alcohol (64-17-5)

sulfuric acid (7664-93-9)

nitric acid (7697-37-2)

Phthalimide (85-41-6)

4-nitrophthalic acid (610-27-5)

4-Nitrophthalimide,
Phthalimide, 4-nitro- (89-40-7)