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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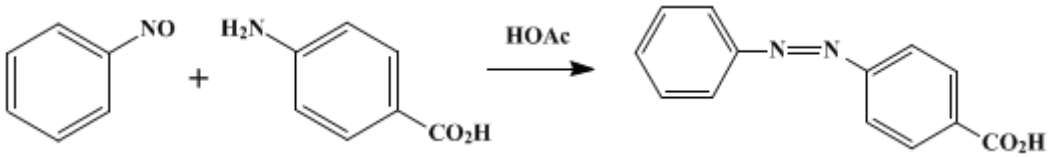
Organic Syntheses, Coll. Vol. 3, p.711 (1955); Vol. 25, p.86 (1945).

p-PHENYLAZOBENZOIC ACID

[Benzoic acid, *p*-phenylazo-]



Submitted by Harry D. Anspon
Checked by W. E. Bachmann and N. C. Deno.

1. Procedure

Fifty-four grams (0.39 mole) of *p*-aminobenzoic acid is dissolved in 390 ml. of warm glacial acetic acid in a 1-l. Erlenmeyer flask. The solution is cooled to room temperature, 42 g. (0.39 mole) of nitrosobenzene (p. 668) is added, and the mixture is shaken until the nitrosobenzene dissolves. The flask is stoppered, and the solution is allowed to stand for 12 hours at room temperature. The product begins to crystallize after about 15 minutes.

The *p*-phenylazobenzoic acid is collected on a Büchner funnel (Note 1) and washed with acetic acid and with water. The yield of air-dried acid melting at 245–247° cor. is 62 g. (70%). By recrystallization from 95% ethanol (60 ml. per g.) the acid is obtained as orange-gold plates which melt at 248.5–249.5° cor.; the yield is 54 g. (61%).

2. Notes

1. The solution is not cooled below room temperature before filtering; cooling below 20° brings down impurities.

3. Discussion

The method employed here is essentially the one described by Angeli and Valori.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 712

References and Notes

1. Angeli and Valori, *Atti accad. Lincei*, **22**, I, 132 (1913) [*C. A.*, **7**, 2223 (1913)].

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

ethanol (64-17-5)

acetic acid (64-19-7)

Nitrosobenzene (586-96-9)

p-aminobenzoic acid (150-13-0)

p-Phenylazobenzoic acid,
Benzoic acid, p-phenylazo- (1562-93-2)