



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.

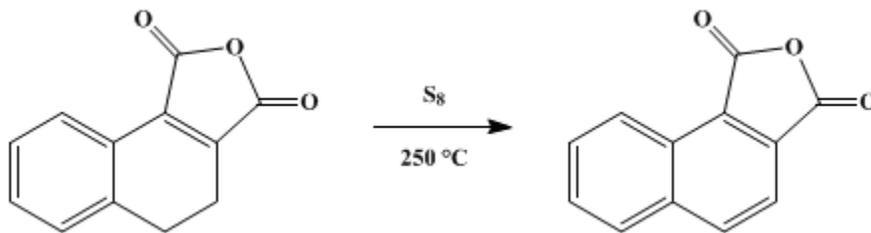
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.423 (1943); Vol. 18, p.59 (1938).

1,2-NAPHTHALIC ANHYDRIDE

[1,2-Naphthalenedicarboxylic anhydride]



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1. Procedure

In a 50-cc. Claisen distilling flask with a 50-cc. sealed-on distilling flask as a receiver are placed 20 g. (0.1 mole) of 3,4-dihydro-1,2-naphthalic anhydride (p. 194) and 3.2 g. (0.1 gram atom) of sulfur. After the flask is immersed in a bath (Note 1) previously heated to 230–235° and shaken until the globule of sulfur has dissolved (fifteen to twenty minutes), the temperature is raised to 250° for thirty minutes (Note 2). The residue is distilled under reduced pressure (Note 3), and the distillate is crystallized from 150 cc. of benzene to which 50 cc. of ligroin (b.p. 60–80°) has been added at the boiling point. The yield is 15–18 g. (76–91 per cent of the theoretical amount) of light yellow needles melting at 166–167°.

2. Notes

1. A Wood's metal bath or a mixture (m.p. about 150°) of ten parts of potassium nitrate and seven and one-half parts of sodium nitrite may be used.
2. If the heating at 250° is continued until hydrogen sulfide is no longer evolved (about ten hours), the product, after recrystallization, is lighter in color and shrinks less before melting.
3. The material comes over between 210° and 215° at 12–13 mm. with the bath at 260°.

3. Discussion

1,2-Naphthalic anhydride has been prepared by the hydrolysis of the dinitrile of 1,2-naphthalic acid;¹ by the oxidation of suitably substituted hydrocarbons or ketones;² and by the dehydrogenation of the 3,4-dihydro compound with bromine³ or with sulfur.⁴

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 3, 300*

References and Notes

1. Cleve, *Ber.* **25**, 2475 (1892); Waldmann, *J. prakt. Chem.* (2) **127**, 197 (1930); Cook, *J. Chem. Soc.* **1932**, 462.
 2. Freund and Fleischer, *Ann.* **399**, 186, 210 (1913); Kruber and Schade, *Ber.* **68**, 11 (1935).
 3. von Auwers and Möller, *J. prakt. Chem.* (2) **109**, 141 (1925).
 4. Fieser and Hershberg, *J. Am. Chem. Soc.* **57**, 1853 (1935).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

ligroin

dinitrile of 1,2-naphthalic acid

[Benzene \(71-43-2\)](#)

[hydrogen sulfide \(7783-06-4\)](#)

[bromine \(7726-95-6\)](#)

[sodium nitrite \(7632-00-0\)](#)

[sulfur \(7704-34-9\)](#)

[potassium nitrate \(7757-79-1\)](#)

[3,4-Dihydro-1,2-naphthalic anhydride \(37845-14-0\)](#)

[1,2-Naphthalic anhydride,](#)
[1,2-Naphthalenedicarboxylic anhydride \(5343-99-7\)](#)