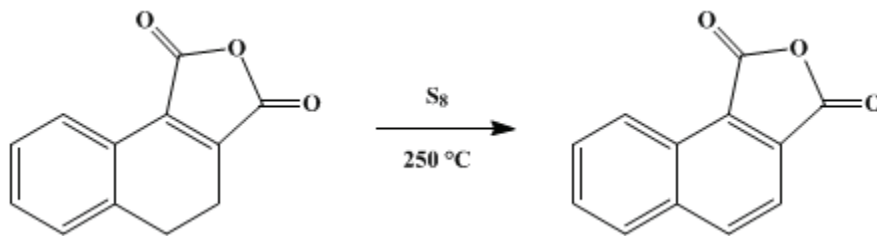


1,2-NAPHTHALIC ANHYDRIDE

[1,2-Naphthalenedicarboxylic anhydride]



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Checked by C. R. Noller and S. Kinsman.

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 \(5343-99-7\)](#)