



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.

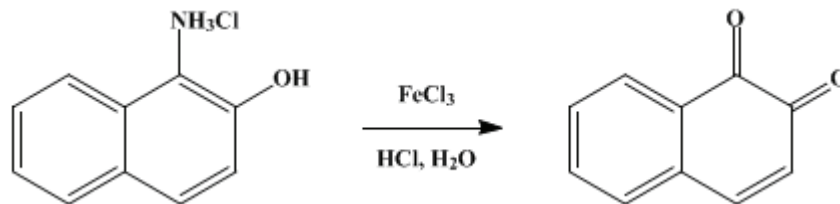
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.430 (1943); Vol. 17, p.68 (1937).

1,2-NAPHTHOQUINONE



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1. Procedure

For the best results this preparation must be carried out rapidly. The vessels and reagents required should be made ready in advance. The oxidizing solution is prepared by dissolving 240 g. (0.89 mole) of [ferric chloride hexahydrate](#) in a mixture of 90 cc. of concentrated [hydrochloric acid](#) and 200 cc. of water with heating, cooling to room temperature by the addition of 200–300 g. of ice, and filtering the solution by suction.

Eighty grams (0.41 mole) of 1,2-aminonaphthol hydrochloride of a high degree of purity ([Note 1](#)) is placed in a 5-l. round-bottomed flask and covered with a solution of 5 cc. of [hydrochloric acid](#) in 3 l. of water which has been heated to 35°. The material is dissolved quickly by shaking (one to two minutes), and the solution is filtered rapidly by suction from a trace of residue and transferred to a clean 5-l. flask ([Note 2](#)). The oxidizing solution is added all at once while rotating the flask vigorously in order to mix the two solutions thoroughly. The quinone separates at once as a voluminous, microcrystalline, yellow precipitate. The product is collected on a Büchner funnel, washed well with water ([Note 3](#)), and then for more thorough washing it is transferred to a large beaker, stirred for a few minutes with 2 l. of water (at 30°), and again collected. The filter cake is cut into slices and dried on filter paper at room temperature in an atmosphere free from acid fumes. The yield is 60–61 g. (93–94 per cent of the theoretical amount).

The material is pure golden yellow in color and melts with decomposition at 145–147°, with some softening at about 140°. It contains none of the black, sparingly soluble dinaphthylidiquinhydrone and dissolves without residue in [alcohol](#) or [benzene](#). Under the microscope it is seen to consist of a mass of well-formed, fine needles. By crystallization from alcohol or [benzene](#) it may be obtained as orange-red needles of good appearance, but the process is unreliable, it involves much loss of material, and the point of decomposition is lowered by 10–20°. It is thus best to use or to preserve the quinone in the form originally obtained; in this condition it is essentially pure and it will keep indefinitely. The material should not be pulverized, for it then becomes highly electrified.

2. Notes

1. The quality of the [naphthoquinone](#) is closely dependent upon the purity of the starting material. The crystalline product described on [p. 36](#) may be used without recrystallization and, indeed, without being dried. The excess of oxidizing agent specified is sufficient to allow for a yield of [aminonaphthol](#) slightly higher than that given; if the yield is lower the larger excess of [ferric chloride](#) does no harm.
2. The solution at this point should be clear, but it may acquire a rather pronounced orange-yellow color when viewed in bulk. It should not, however, turn purple.
3. The yellow color of the wash water is due to a slight solubility of the quinone.

3. Discussion

The only satisfactory method of preparing [1,2-naphthoquinone](#) (β -[naphthoquinone](#)) is by the oxidation of 1,2-aminonaphthol in acid solution, and the chief problem involved is the preparation of this intermediate in suitable yield and purity. This problem and the literature pertaining to it are

discussed on p. 38. Most reports of the preparation of the [aminonaphthol](#) include some description of its oxidation, but the only particularly helpful comment on the reaction is that [ferric chloride](#) is a better oxidizing agent than [chromic acid](#) because at a low temperature it does not attack the quinone, even when present in excess.¹

One other method of preparing [β-naphthoquinone](#) is from 1,2-bromonaphthol through the ketonitrobromide.² Though the parent quinone itself is such a sensitive compound that the material so obtained decomposes within a few hours, the method is of considerable value for the preparation of certain substituted β-naphthoquinones.³

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 3, 465](#)

References and Notes

1. Groves, J. Chem. Soc. **45**, 298 (1884).
 2. Fries, Ann. **389**, 315 (1912).
 3. Fries and Schimmelschmidt, *ibid.* **484**, 245 (1930).
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Appendix

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

1,2-Aminonaphthol hydrochloride

1,2-aminonaphthol

dinaphthylidiquinhydrone

1,2-bromonaphthol

ketonitrobromide

[alcohol \(64-17-5\)](#)

[hydrochloric acid \(7647-01-0\)](#)

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

[aminonaphthol](#)

[chromic acid \(7738-94-5\)](#)

[ferric chloride \(7705-08-0\)](#)

[naphthoquinone \(130-15-4\)](#)

[1,2-Naphthoquinone,
β-naphthoquinone \(524-42-5\)](#)

ferric chloride hexahydrate (10025-77-1)

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