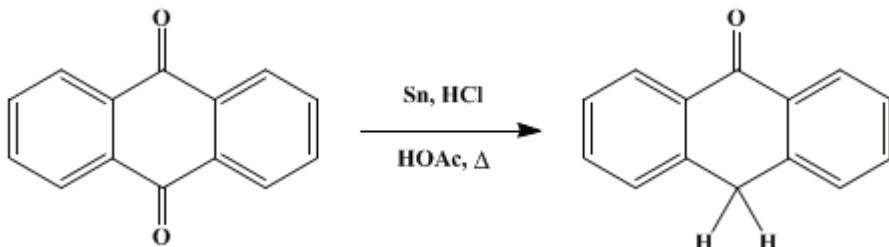


ANTHRONE



Submitted by Kurt H. Meyer

Checked by J. B. Conant and W. C. Boyd.

1. Procedure

In a 2-l. round-bottomed flask with a reflux condenser, 104 g. (0.5 mole) of **anthraquinone**, m.p. 276–280° (corr.), is mixed with 100 g. (0.86 atom) of granulated **tin**, and 750 cc. of glacial **acetic acid** is added. The contents of the flask are heated to boiling, and in the course of two hours, 298 g. (250 cc., 8.2 moles) of c.p. **hydrochloric acid** (sp. gr. 1.19) is added in 10-cc. portions to the boiling mixture. At the end of this time all the **anthraquinone** should have gone into solution; if not, more **tin** and **hydrochloric acid** are added.

The liquid is filtered with suction through a Gooch crucible with a fixed porous plate (**Note 1**), and 100 cc. of water is added. The **anthrone** crystallizes when the solution is cooled to about 10°. The crystals are filtered with suction on a Büchner funnel and washed with water. After drying on a porous plate the melting point of the material is about 153°. The yield is 80 g. (82.5 per cent of the theoretical amount). On recrystallization from a 3:1 mixture (**Note 2**) of **benzene** and petroleum ether about 60 g. of **anthrone** melting at 154–155° (corr.) is obtained (62 per cent of the theoretical amount).

2. Notes

1. The liquid can also be filtered through a fluted filter paper, but this is slower.
2. The proportions do not make much difference as far as the yield is concerned, but the substance is more soluble in mixtures rich in **benzene**. About 10–12 cc. of the 3:1 mixture is required for each gram of **anthrone**. The **anthrone** may be more readily dissolved if it is added to the estimated quantity of hot **benzene** on the steam bath, and the petroleum ether is then added. About two-thirds of the mother liquor may be distilled off through a condenser and used in later runs. The residual mother liquor deposits about 12 g. of rather impure **anthrone**.

3. Discussion

Anthrone can be prepared by the reduction of **anthraquinone** with **tin** and **hydrochloric acid**,¹ and with aluminum bronze,² or by cyclization of *o*-benzylbenzoic acid with liquid **hydrogen fluoride**.³

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 390

References and Notes

1. Meyer, Ann. **379**, 55 (1911).
2. Eckert and Pollak, Monatsh. **38**, 12 (1917).
3. Fieser and Hershberg, J. Am. Chem. Soc. **61**, 1278 (1939).

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

aluminum bronze

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

Benzene (71-43-2)

tin (7440-31-5)

Anthrone (90-44-8)

Anthraquinone (84-65-1)

hydrogen fluoride (7664-39-3)

o-benzylbenzoic acid (612-35-1)

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