



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](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.

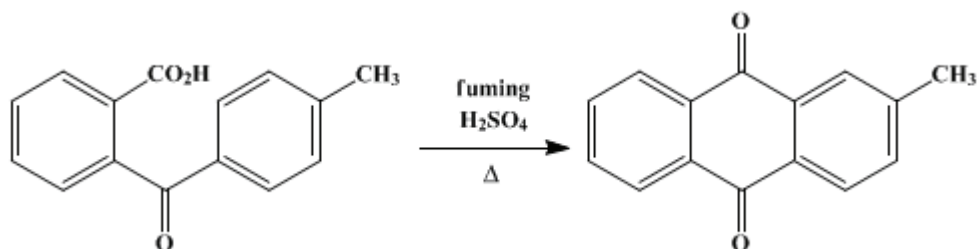
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

*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. 1, p.353 (1941); Vol. 4, p.43 (1925).*

## **β-METHYLANTHRAQUINONE**

[Anthraquinone, 2-methyl-]



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### 1. Procedure

The *p*-toluylo-benzoic acid which is obtained from 100 g. of [phthalic anhydride](#) (p. 517), and which should weigh 157 g. (0.65 mole), is mixed with 1400 g. (725 cc.) of fuming [sulfuric acid](#) (20 per cent anhydride) ([Note 1](#)) in a 2-l. flask protected by a calcium chloride tube, and the mixture is heated on the steam bath for two hours with occasional shaking ([Note 2](#)). The clear, deep red solution is poured when cold upon cracked ice in a 4-l. beaker. The [methyl anthraquinone](#) separates and is digested for twenty minutes by passing in steam, after which it is filtered by suction. A flannel cloth is used in the filter, or a filter plate may be cemented into a Büchner funnel with water-glass. The precipitate is washed well with hot water, after which it is returned to the beaker and digested as before with hot water to which is added a slight excess of [ammonia](#), beyond that required to neutralize any acid present.

The product is filtered and dried to constant weight. The filtrate will be clear and will give no precipitate with [hydrochloric acid](#) if the conditions of condensation have been correct.

The [β-methylanthraquinone](#) is pale tan in color and weighs from 118 to 130 g. (81–90 per cent of the theoretical amount, based upon the weight of acid taken). It is practically pure, melting at 173°. Upon crystallization from alcohol in the presence of animal charcoal, it forms long, silken, almost colorless needles, melting constantly at 173.5° (176° corr.).

### 2. Notes

1. Some investigators have used concentrated instead of fuming [sulfuric acid](#) for the condensation, but the yield is usually low and the product is always colored bright yellow by some impurity which cannot be removed by crystallization.
2. The time allowed for condensation may be shortened to one-half, without affecting the yield, by maintaining the temperature at 125–130°.

### 3. Discussion

[β-Methylanthraquinone](#) can be prepared by the oxidation of [β-methylanthracene](#)<sup>1</sup> and material of the same origin, obtained by the benzene-extraction of crude commercial [anthraquinone](#);<sup>2</sup> from the intramolecular condensation of *p*-toluylo-benzoic acid;<sup>3</sup> and from *o*-[carbomethoxybenzoyl chloride](#) and [toluene](#).<sup>4</sup>

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 1, 517](#)

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## References and Notes

1. Fischer, Ber. **8**, 675 (1875); J. prakt. Chem. (2) **79**, 560 (1909); Fischer and Gross, J. prakt. Chem. (2) **82**, 232 (1910); Fischer, Falco, and Gross, *ibid.* (2) **83**, 210 (1911); Lavaux, Ann. chim. phys. (8) **20**, 445 (1910); I. G. Farbenind. A.-G., Brit. pat. 253,911 and Swiss pat. 124,266 [Chem. Zentr. II, 1820 (1928)].
  2. Perkin and Cope, J. Chem. Soc. **65**, 843 (1894); Wachendorff and Zincke, Ber. **10**, 1485 (1877); Börnstein, Ber. **15**, 1820 (1882); Römer and Link, Ber. **16**, 696 (1883).
  3. Elbs, J. prakt. Chem. (2) **33**, 319 (1886); (2) **41**, 4 (1890); Gresby, Ann. **234**, 239 (1886); Limpricht, Ann. **299**, 300 (1898); Ber. **28**, 1134 (1895); Limpricht and Weigand, Ann. **311**, 180 (1900); Heller and Schülke, Ber. **41**, 3632 (1908); Krassovski, J. Russ. Phys. Chem. Soc. **46**, 1067 (1914) [Chem. Zentr. **I** 999 (1915)].
  4. Smith, J. Am. Chem. Soc. **43**, 1922 (1921).
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## Appendix

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

p-Toluylo-benzoic acid

sulfuric acid (7664-93-9)

hydrochloric acid (7647-01-0)

ammonia (7664-41-7)

phthalic anhydride (85-44-9)

Anthraquinone (84-65-1)

toluene (108-88-3)

$\beta$ -Methylanthraquinone,  
Anthraquinone, 2-methyl- (84-54-8)

methyl anthraquinone

$\beta$ -methylanthracene (613-12-7)

o-carbomethoxybenzoyl chloride