

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 accessed of charge text can be free 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.

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. 3, p.806 (1955); Vol. 23, p.92 (1943).

# **TETRAPHENYLCYCLOPENTADIENONE**

[Cyclopentadienone, tetraphenyl-]



Submitted by John R. Johnson and Oliver Grummitt. Checked by Nathan L. Drake and Stuart Haywood.

#### **1. Procedure**

In a 500-ml. round-bottomed flask, 21 g. (0.1 mole) of benzil and 21 g. (0.1 mole) of dibenzyl ketone (Note 1) are dissolved in 150 ml. of hot ethanol. The flask is fitted with a reflux condenser, the temperature of the solution is raised nearly to the boiling point, and a solution of 3 g. of potassium hydroxide in 15 ml. of ethanol is added slowly in two portions through the condenser. When the frothing has subsided the mixture is refluxed for 15 minutes and then cooled to  $0^{\circ}$ . The dark crystalline product is filtered with suction and washed with three 10-ml. portions of 95% ethanol. The product melts at 218–220° and weighs 35–37 g. (91–96%) (Note 2).

#### 2. Notes

1. The dibenzyl ketone should melt at 34–35°.

2. This product is sufficiently pure for most purposes. It may be crystallized from a mixture of ethanol and benzene, using 155–160 ml. solvent for 5 g. of tetraphenylcyclopentadienone; the melting point of the recrystallized material is 219–220°.

## 3. Discussion

Tetraphenylcyclopentadienone has been prepared by the action of phenylmagnesium bromide on benzaldiphenylmaleide,<sup>1</sup> and by reduction, dehydration, and oxidation of the methylenedesoxybenzoin obtained by condensing formaldehyde with desoxybenzoin.<sup>2</sup> The present procedure is essentially that of Dilthey.<sup>3</sup>.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 807
- Org. Syn. Coll. Vol. 5, 604
- Org. Syn. Coll. Vol. 5, 1037

## **References and Notes**

- 1. Lowenbein and Uhlich, Ber., 58, 2662 (1925).
- 2. Ziegler and Schnell, Ann., 445, 266 (1925).
- **3.** Dilthey and Quint, *J. prakt. Chem.*, (2) **128**, 146 (1930); Ger. pat. 575,857 [*Frdl.*, **20**, 503 (1933); *C. A.*, **28**, 1356 (1934)].

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

Desoxybenzoin

benzaldiphenylmaleide

methylenedesoxybenzoin

ethanol (64-17-5)

Benzene (71-43-2)

formaldehyde (50-00-0)

Benzil (134-81-6)

potassium hydroxide (1310-58-3)

Phenylmagnesium bromide (100-58-3)

dibenzyl ketone (102-04-5)

Tetraphenylcyclopentadienone, Cyclopentadienone, tetraphenyl- (479-33-4)

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