Organic Syntheses

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.

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.

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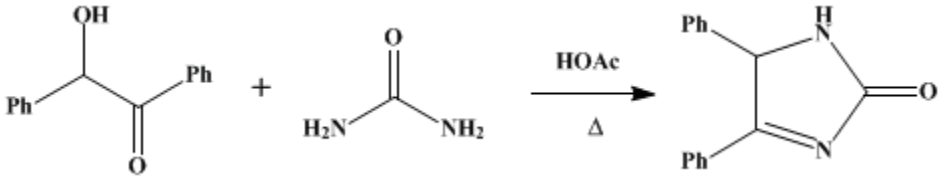
Organic Syntheses, Coll. Vol. 2, p.231 (1943); Vol. 12, p.34 (1932).

4,5-DIPHENYLGLYOXALONE

[2(5)-Imidazolone, 4,5-diphenyl-]



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1. Procedure

A mixture of 212 g. (1 mole) of benzoin (Note 1), 110 g. (1.8 moles) of urea and 800 cc. of glacial acetic acid in a 2-l. round-bottomed flask is heated under a reflux condenser for seven hours (Note 2). The hot solution is quickly poured into a 2-l. beaker (Note 3) and allowed to stand at least three hours. The cold mixture is then transferred by means of a large wooden spoon or spatula to a 30-cm. Büchner funnel and sucked as dry as possible with the help of a rubber dam. The crystals are pressed down during the suction filtration. The filtrate is discarded. The crystals are returned to the beaker, stirred mechanically with 500 cc. of ether for thirty minutes, filtered again by suction (Note 4), and spread out to dry at least overnight (Note 5). The product is dissolved by heating with 1 l. of glacial acetic acid in a 2-l. round-bottomed flask attached to a reflux condenser (Note 6), and the clear solution is poured, with mechanical stirring, into 2.5 l. of water in a 4-l. (1-gal.) crock. Stirring is continued for thirty minutes (Note 7). The mixture is filtered on a 30-cm. Büchner funnel and pressed and sucked as dry as possible. The crystals are transferred to the 2-l. beaker and stirred mechanically for ten minutes with 1 l. of water. After filtration, the washing with water is repeated. The product is then returned to the beaker, stirred with 750 cc. of ether, and filtered again. The material is then air-dried to constant weight. The yield of white fluffy crystals of diphenylglyoxalone melting at 330–335° (corr.) (Note 8) is 220–230 g. (93–97 per cent of the theoretical amount).

2. Notes

1. The benzoin (Org. Syn. Coll. Vol. I, **1941**, 94) need not be recrystallized.
2. At first the color is reddish orange but later it changes to dark yellow.
3. If left in the flask the product solidifies too much to be removed readily.
4. Unreacted benzoin is removed by the ether, in which diphenylglyoxalone is only sparingly soluble.
5. The product is difficult to dry completely.
6. This step is not successful if less than 1 l. of glacial acetic acid is used.
7. The water removes unreacted urea.
8. The melting point is conveniently taken on the surface of mercury heated in a test tube with the thermometer dipping in the mercury.

3. Discussion

The only method of preparative interest is the interaction between benzoin and urea in acetic acid solution as described by Biltz[1] and Chattaway.[2]

References and Notes

1. Biltz, Ann. **368**, 173 (1909).
2. Chattaway and Coulson, J. Chem. Soc. **1928**, 1363.

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

4,5-Diphenylglyoxalone

2(5)-Imidazolone, 4,5-diphenyl-

diphenylglyoxalone

acetic acid (64-19-7)

ether (60-29-7)

mercury (7439-97-6)

Benzoin (119-53-9)

urea (57-13-6)