



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.

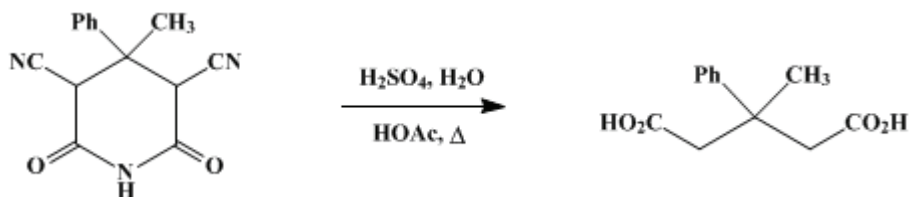
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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. 4, p.664 (1963); Vol. 39, p.54 (1959).*

## **$\beta$ -METHYL- $\beta$ -PHENYLGLUTARIC ACID**

**[Glutaric acid, 3-methyl-3-phenyl-]**



Submitted by S. M. McElvain and David H. Clemens<sup>1</sup>.

Checked by W. E. Parham, Perry W. Kirklin, Jr., and Wayland E. Noland.

### 1. Procedure

In a 3-l. round-bottomed flask fitted with a small glass paddle stirrer and a reflux condenser are placed 101 g. (0.4 mole) of  $\beta$ -methyl- $\beta$ -phenyl- $\alpha,\alpha'$ -dicyanoglutarimide (p. 662), and a mixture of 500 ml. of water, 500 g. of concentrated sulfuric acid, and 400 ml. of glacial acetic acid. Without starting the stirrer (Note 1), the mixture is heated under reflux for 2 hours. Then the stirrer is cautiously started, and reflux is continued for a total of 80 hours. The reaction mixture is transferred to a 6-l. Erlenmeyer flask, 3 l. of water is added and the mixture cooled in a refrigerator overnight. The precipitated acid is filtered by suction (Note 2), washed with 100 ml. of water, and air-dried to constant weight. The crude, dry product is swirled for 5 minutes with 200 ml. of benzene, filtered by suction, washed with two 100-ml. portions of benzene, and again air-dried. The yield of acid, m.p. 136–140°, is 64.5–68 g. (72.5–76.5%) (Note 3) and (Note 4).

### 2. Notes

1. Use of the stirrer during the first 2 hours of the hydrolysis results in excessive foaming.
2. In some runs a small amount of dark tar adhered to the side of the flask. This material was not isolated in the filtration.
3. The product is pure enough for most purposes, but it may be further purified by recrystallization from water (100 ml. for 20 g. of acid) to give material melting at 140–142° in 95% yield.
4. If the alkyl group of a  $\beta$ -alkyl- $\beta$ -phenyl- $\alpha,\alpha'$ -dicyanoglutarimide is larger than the methyl group, the hydrolysis to the corresponding glutaric acid should be modified as described by McElvain and Clemens, *J. Am. Chem. Soc.*, **80**, 3915 (1958).

### 3. Discussion

$\beta$ -Methyl- $\beta$ -phenylglutaric acid has been prepared by the hydrolysis of  $\beta$ -methyl- $\beta$ -phenyl- $\alpha,\alpha'$ -dicyanoglutarimide with sulfuric acid, and also by acid hydrolysis of the condensation product of 1,1-dichloroethylbenzene and ethyl sodiomalonate.<sup>2</sup>

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### References and Notes

1. University of Wisconsin, Madison, Wisconsin.
  2. Phalnikar and Nargund, *J. Univ. Bombay*, **6**, Pt. II, 102 (1937) [*C. A.*, **32**, 3763 (1938)].
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**(Registry Number)**

$\beta$ -METHYL- $\beta$ -PHENYL- $\alpha,\alpha'$ -DICYANOGLUTARIMIDE

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

Benzene (71-43-2)

$\beta$ -Methyl- $\beta$ -phenylglutaric acid,  
Glutaric acid, 3-methyl-3-phenyl- (4160-92-3)

1,1-dichloroethylbenzene

ethyl sodiomalonate