



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.

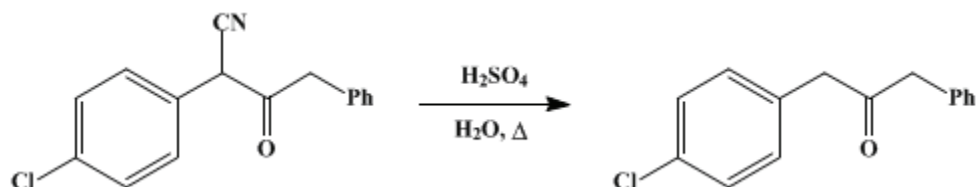
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.176 (1963); Vol. 35, p.32 (1955).*

## 1-(*p*-CHLOROPHENYL)-3-PHENYL-2-PROPANONE

### [2-Propanone, 1-(*p*-chlorophenyl)-3-phenyl-]



Submitted by Stephen B. Coan and Ernest I. Becker<sup>1</sup>.

Checked by Charles C. Price and G. Venkat Rao.

### 1. Procedure

In a 250-ml. three-necked flask equipped with a stirrer and a condenser are placed 75 ml. of 60% sulfuric acid and 25 g. (0.093 mole) of  $\alpha$ -(4-chlorophenyl)- $\gamma$ -phenylacetoacetonitrile (p. 174). While being stirred, the mixture is heated at reflux until the evolution of carbon dioxide ceases (Note 1) and (Note 2). The mixture is cooled, poured into 200 ml. of ice water, and extracted three times with 150-ml. portions of ether. The ether solution is washed once with 50 ml. of water, twice with 100-ml. portions of 10% sodium hydroxide solution, and then with 50 ml. of water. After drying over sodium sulfate and filtering, the ether is distilled on a steam bath, affording 15–17 g. (66–75%) of 1-(*p*-chlorophenyl)-3-phenyl-2-propanone, m.p. 34.5–35.5°. Recrystallization from 160 ml. of petroleum ether (b.p. 40–60°) gives 12–13 g. (53–57%) of product, m.p. 35.9–36.5° (Note 3).

### 2. Notes

1. The hydrolysis and decarboxylation of the nitrile require from 18 to 24 hours.
2. The evolution of carbon dioxide is conveniently observed by passing the effluent gases through a saturated solution of barium hydroxide.
3. The submitters report that a similar procedure has been used in the preparation of other monosubstituted dibenzyl ketones from  $\alpha$ -phenyl- $\gamma$ -(4-substituted phenyl)acetoacetonitriles.

Product	Yield, %	M.P.
1,3-Diphenyl-2-propanone	65–71	34.2–34.5°
1-( <i>p</i> -Tolyl)-3-phenyl-2-propanone	66	30.8–31.2°
1-( <i>p</i> -Bromophenyl)-3-phenyl-2-propanone	50	53.8–54.2°
1-( <i>p</i> -Fluorophenyl)-3-phenyl-2-propanone	50	36.0–36.5°
1-( <i>p</i> -Methylthiophenyl)-3-phenyl-2-propanone	40	43.9–44.2°
1-( <i>p</i> -Methoxyphenyl)-3-phenyl-2-propanone <sup>a</sup>	19	46.6–47.4°

<sup>a</sup> It is necessary to employ 5 ml. of glacial acetic acid and 5 ml. of 20% aqueous hydrochloric acid, instead of the sulfuric acid, per gram of ketonitrile as reaction solvent in order to obtain the desired product.

### 3. Discussion

This method of preparation of 1-(*p*-chlorophenyl)-3-phenyl-2-propanone has been reported by Coan and Becker.<sup>2</sup> The method utilized is a modification of that described for the formation of 1,3-diphenyl-2-propanone from  $\alpha$ , $\gamma$ -diphenylacetoacetonitrile.<sup>3,4</sup>

### References and Notes

1. Polytechnic Institute of Brooklyn, Brooklyn 2, New York.
  2. Coan and Becker, *J. Am. Chem. Soc.*, **76**, 501 (1954).
  3. Meyer, *J. prakt. Chem.*, [2] **52**, 81 (1895).
  4. Walther and Schickler, *J. prakt. Chem.*, [2] **55**, 305 (1897).
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**Appendix**  
**Chemical Abstracts Nomenclature (Collective Index Number);**  
**(Registry Number)**

petroleum ether

$\alpha$ -phenyl- $\gamma$ -(4-substituted phenyl)acetoacetonitriles

sulfuric acid (7664-93-9)

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ether (60-29-7)

sodium hydroxide (1310-73-2)

sodium sulfate (7757-82-6)

carbon dioxide (124-38-9)

barium hydroxide (17194-00-2)

1,3-Diphenyl-2-propanone (102-04-5)

$\alpha$ -(4-CHLOROPHENYL)- $\gamma$ -PHENYLACETOACETONITRILE (35741-47-0)

$\alpha,\gamma$ -Diphenylacetoacetonitrile

1-(p-CHLOROPHENYL)-3-PHENYL-2-PROPANONE,  
2-Propanone, 1-(p-chlorophenyl)-3-phenyl- (35730-03-1)

1-(p-Tolyl)-3-phenyl-2-propanone

1-(p-Bromophenyl)-3-phenyl-2-propanone

1-(p-Fluorophenyl)-3-phenyl-2-propanone

1-(p-Methylthiophenyl)-3-phenyl-2-propanone