



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.

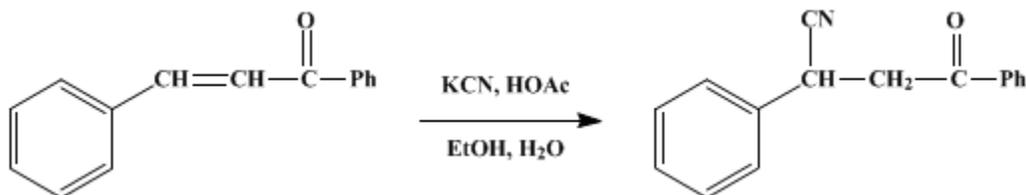
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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. 2, p.498 (1943); Vol. 10, p.80 (1930).*

## **$\alpha$ -PHENYL- $\beta$ -BENZOYLPROPIONITRILE**

[ $\alpha$ -Tolunitrile,  $\alpha$ -phenacyl-]



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### 1. Procedure

The following preparation, through the filtration of the crude solid and washing with water, should be carried out in a hood with a good draft.

Into a 5-l. flask or bottle, set in a water bath and fitted with a stirrer, thermometer, and separatory funnel, are placed 208 g. (1 mole) of [benzalacetophenone](#) ([Note 1](#)), 3.5 l. of 95 per cent [ethyl alcohol](#) ([Note 2](#)), and 60 g. (1 mole) of glacial [acetic acid](#) ([Note 3](#)). The mixture is warmed with stirring to 35°, and a solution of 130 g. (2 moles) of [potassium cyanide](#) in 375 cc. of water is added from the separatory funnel over a period of about fifteen minutes. The initial greenish color changes to yellow after all the [acetic acid](#) has reacted and the solution has become alkaline ([Note 3](#)). Stirring is continued for three hours, the temperature being maintained at 35°. During this time about half of the nitrile crystallizes. The flask is then loosely stoppered and left in a cool place for about fifty hours (conveniently out-of-doors in cold weather, but out of direct sunlight), after which the solid is filtered and washed, first with 500 cc. of cold 50 per cent [alcohol](#), and then with water until free from [potassium cyanide](#) ([silver nitrate](#) test). The yield of air-dried material melting at 125° is 220–227 g. (93–96 per cent of the theoretical amount). This product is sufficiently pure for most purposes, although it contains traces of a high-melting substance. If a product of higher melting point is desired, it may be recrystallized from 1 l. of 95 per cent [alcohol](#) or 375 cc. of [acetone](#). Pure [phenylbenzoylpropionitrile](#) melts at 127°.

### 2. Notes

1. Crude alkali-free, air-dried [benzalacetophenone](#) is used ([Org. Syn. Coll. Vol. I, 1941, 78](#)).
2. The preparation may be carried out in a more concentrated solution (1 l. of [alcohol](#)) with the same yield, but an inferior product is obtained. If this is done, it is best to stir for fifteen minutes at 50° after all the cyanide has been added and then cool in tap water. Since the product separates as an oil from a solution of this concentration, it is best to inoculate with a crystal of the nitrile.
3. If the solution becomes too alkaline, the nitrile formed will add to a second molecule of unsaturated ketone so readily that the product will consist almost entirely of a high-melting (284–286°) substance. For this reason it is essential to measure the [acetic acid](#) accurately; if too much is used, addition of [hydrocyanic acid](#) will not take place.

### 3. Discussion

$\alpha$ -Phenyl- $\beta$ -benzoylpropionitrile has been prepared by the action of [sodium](#) or [potassium cyanide](#) on  $\beta$ -chlorobenzylacetophenone<sup>1</sup> or [benzalacetophenone dibromide](#);<sup>2</sup> and by the addition of [hydrocyanic acid](#) to [benzalacetophenone](#) in the presence of [sodium](#) or [potassium cyanide](#).<sup>3</sup>

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 3, 358](#)

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## References and Notes

1. Anschütz and Montfort, Ann. **284**, 2 (1895); Rupe and Schneider, Ber. **28**, 960 (1895).
  2. Dodwadmath and Wheeler, Proc. Indian Acad. Sci. **2A**, 438 (1935) [C. A. **30**, 1771 (1936)].
  3. Hann and Lapworth, J. Chem. Soc. **85**, 1358 (1904); Lapworth and Wechsler, ibid. **97**, 41 (1910).
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## Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

ethyl alcohol,  
alcohol (64-17-5)

acetic acid (64-19-7)

hydrocyanic acid (74-90-8)

silver nitrate (7761-88-8)

potassium cyanide (151-50-8)

Benzalacetophenone (94-41-7)

acetone (67-64-1)

sodium (13966-32-0)

Benzalacetophenone dibromide (611-91-6)

$\alpha$ -Phenyl- $\beta$ -benzoylpropionitrile,  
 $\alpha$ -Tolunitrile,  $\alpha$ -phenacyl-,  
phenylbenzoylpropionitrile (6268-00-4)

$\beta$ -chlorobenzylacetophenone