



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.

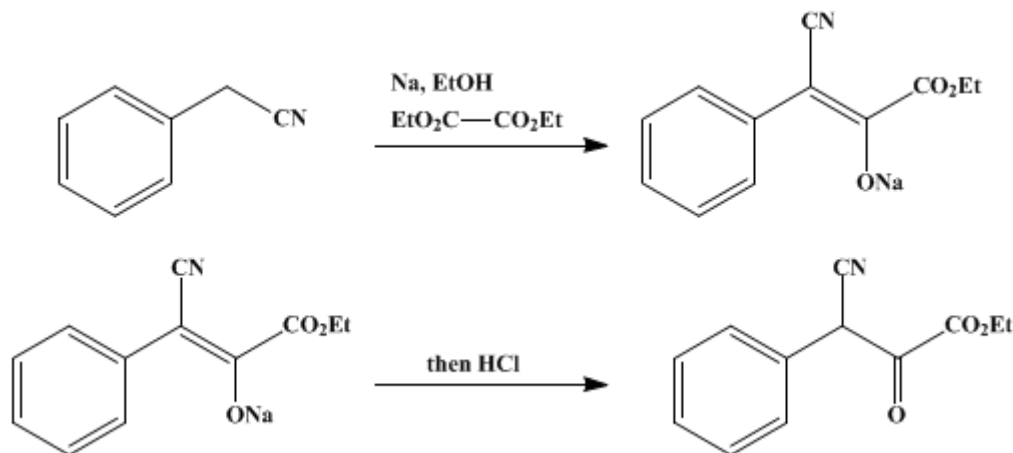
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.287 (1943); Vol. 11, p.40 (1931).*

## ETHYL PHENYLCYANOPYRUVATE

[Pyruvic acid, cyanophenyl-, ethyl ester]



Submitted by Roger Adams and H. O. Calvery.  
Checked by J. B. Conant and Doris Blumenthal.

### 1. Procedure

In a 3-l. round-bottomed flask, fitted with a reflux condenser, is placed 650 cc. of absolute alcohol (Note 1), and to it 46 g. (2 gram atoms) of sodium is added as rapidly as possible without loss of material through the condenser. If the sodium does not entirely dissolve, heat is applied. To the hot sodium ethoxide solution 312 g. (2.1 moles) of ethyl oxalate (Org. Syn. Coll. Vol. I, 1941, 261) is added as rapidly as possible. Then, immediately, 234 g. (2 moles) of benzyl cyanide (Org. Syn. Coll. Vol. I, 1941, 107) is added, and the reaction mixture is allowed to stand overnight. The solution is transferred to a 3-l. beaker and treated with 250–300 cc. of water (Note 2). It is then warmed to 35° and made strongly acid to litmus with concentrated hydrochloric acid. Mechanical stirring is used during the acidification. On cooling to ordinary temperatures the ester crystallizes. The yield is 360–385 g. of lemon-yellow crystals melting at 126–128°. On recrystallization from 60 per cent alcohol, the ester melts at 130°. The final yield is 300–325 g. (69–75 per cent of the theoretical amount).

### 2. Notes

1. Absolute alcohol, prepared according to the directions in Org. Syn. Coll. Vol. I, 1941, 259, is satisfactory.
2. This amount of water dilutes the alcohol so that the ester crystallizes well. If more water is used an oily product separates and then solidifies.

### 3. Discussion

The only method of preparation mentioned in the literature is that given above.<sup>1</sup>

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 531

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### References and Notes

1. Erlenmeyer, Ann. **271**, 173 (1892).

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

alcohol (64-17-5)

hydrochloric acid (7647-01-0)

sodium (13966-32-0)

sodium ethoxide (141-52-6)

Benzyl cyanide (140-29-4)

Ethyl oxalate

Ethyl phenylcyanopyruvate,  
Pyruvic acid, cyanophenyl-, ethyl ester (6362-63-6)