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.

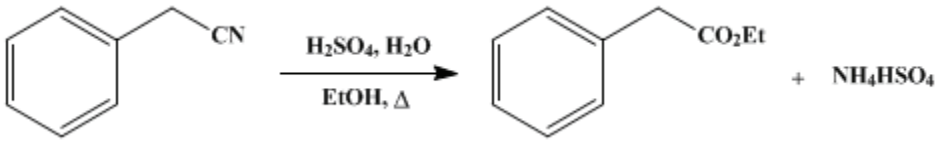
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.002.0027

Organic Syntheses, Coll. Vol. 1, p.270 (1941); Vol. 2, p.27 (1922).

ETHYL PHENYLACETATE

[α-Toluic acid, ethyl ester]



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1. Procedure

In a 3-l. round-bottomed flask, fitted with an efficient reflux condenser, are mixed 750 g. (918 cc.) of 95 per cent alcohol, 750 g. (408 cc.) of concentrated sulfuric acid and 450 g. (3.85 moles) of benzyl cyanide (Note 1). The mixture, which soon separates into two layers, is heated to boiling over a low flame, for six to seven hours, cooled, and poured into 2 l. of water, and the upper layer is separated. This is washed with a little 10 per cent sodium carbonate solution (Note 2) to remove small amounts of phenylacetic acid which may have been formed, and then distilled under reduced pressure. A small amount of water goes over first and then a pure product boiling 132–138°/32 mm. (120–125°/17–18 mm.) (Note 3). The yield is 525–550 g. (83–87 per cent of the theoretical amount).

2. Notes

1. The benzyl cyanide can be prepared according to the directions on p. 107; the product which boils over a 5° range should be used.
2. In washing the layer of ethyl phenylacetate with sodium carbonate it is sometimes advisable to add a certain amount of sodium chloride so that the ester will separate more readily.
3. The product obtained is water-clear and practically colorless. Although the product is collected over a 5° range, most of the liquid is found to boil over a 1° range, if distilled slowly without superheating. The boiling point of ethyl phenylacetate is near that of benzyl cyanide. However, a Kjeldahl analysis of the product shows that only a trace of nitrogen compounds is present.

3. Discussion

Ethyl phenylacetate can be prepared by the esterification of phenylacetic acid and alcohol by hydrochloric[1] or sulfuric acid;[2] and by the treatment of benzyl cyanide with alcohol and hydrogen chloride[3] or sulfuric acid, which is much more convenient in the laboratory.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 107
- Org. Syn. Coll. Vol. 2, 288

References and Notes

1. Radziszewski, Ber. **2**, 208 (1869).
2. Volhard, Ann. **296**, 2 (footnote) (1897); Senderens and Aboulenc, Compt. rend. **152**, 1855 (1911).
3. Wislicenus, Ber. **20**, 592 (1887); Ann. **296**, 361 (1897).

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

hydrochloric

alcohol (64-17-5)

sulfuric acid (7664-93-9)

hydrogen chloride (7647-01-0)

sodium chloride (7647-14-5)

sodium carbonate (497-19-8)

Benzyl cyanide (140-29-4)

Phenylacetic acid (103-82-2)

α-Toluic acid, ethyl ester (93-89-0)

Ethyl phenylacetate (101-97-3)