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.

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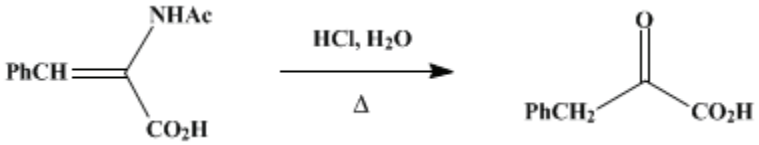
PHENYLPYRUVIC ACID

[Pyruvic acid, phenyl-]



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1. Procedure

Ten grams (0.05 mole) of α-acetaminocinnamic acid (p. 1) and 200 cc. of 1 *N* hydrochloric acid (Note 1) are placed in a 500-cc. flask fitted to an upright condenser with a ground-glass joint. Hydrolysis is completed by boiling for three hours. A few droplets of pale green oil may separate from the boiling solution; these are removed by filtration. The crystals of phenylpyruvic acid which separate from the filtrate on cooling (Note 2) are transferred to a Büchner funnel and washed with a little ice-cold water. The combined filtrate and washings are extracted with four 50-cc. portions of ether. The solvent is removed from the ether solution by evaporation at room temperature, finally in a vacuum desiccator (Note 3). The residue is combined with the first crop of crystals and dried in a vacuum desiccator over calcium chloride and potassium hydroxide. The yield is 7.2–7.7 g. (88–94 per cent of the theoretical amount), and the product melts at 150–154° (Note 4) and (Note 5).

2. Notes

1. Larger quantities of phenlypyruvic acid may be prepared by increasing the amounts of reactants proportionately. However, this is advisable only when the product is to be used immediately since phenylpyruvic acid begins to decompose after standing only a few days.
2. The amount of phenylpyruvic acid which separates from the filtrate is increased if the solution is allowed to stand in the refrigerator several days before filtration. No decomposition was noted when the product was kept suspended in cold, dilute acid.
3. The evaporation may be carried out conveniently at room temperature by passing a stream of dry air or inert gas over the surface of the solution under a glass bell.
4. The melting point varies considerably with the rate of heating.
5. Phenylpyruvic acid may be recrystallized from ethylene chloride, benzene, or chloroform, but losses due to instability of the compound are quite large.

3. Discussion

Phenylpyruvic acid has been prepared by the hydrolysis of α-benzoylaminocinnamic acid with alkalies or acids;[1], [2] by the acid hydrolysis of ethyl phenyloxalacetate;[3] by the acid hydrolysis of ethyl phenylcyanopyruvate;[2], [4] by dehydration of β-phenylglyceric acid with sulfuric acid;[5] and by the alkaline hydrolysis of α-acetaminocinnamic acid.[6]

References and Notes

1. Plöchl, Ber. **16**, 2817 (1883).
2. Erlenmeyer, Jr., Ann. **271**, 165, 173 (1892).
3. Wislicenus, Ber. **20**, 592 (1887).
4. Erlenmeyer, Jr., and Arbenz, Ann. **333**, 228 (1904); Hemmerlé, Ann. chim. (9) **7**, 229 (1917).
5. Dieckmann, Ber. **43**, 1034 (1910).

6. Bergmann and Stern, Ann. **448**, 27 (1926).

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

phenlypyruvic acid

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

hydrochloric acid (7647-01-0)

Benzene (71-43-2)

ether (60-29-7)

chloroform (67-66-3)

ethylene chloride (107-06-2)

potassium hydroxide (1310-58-3)

α-Acetaminocinnamic acid (5469-45-4)

Phenylpyruvic acid,
Pyruvic acid, phenyl- (156-06-9)

Ethyl phenylcyanopyruvate (6362-63-6)

α-Benzoylaminocinnamic acid (1155-48-2)

ethyl phenyloxalacetate

β-phenylglyceric acid