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.

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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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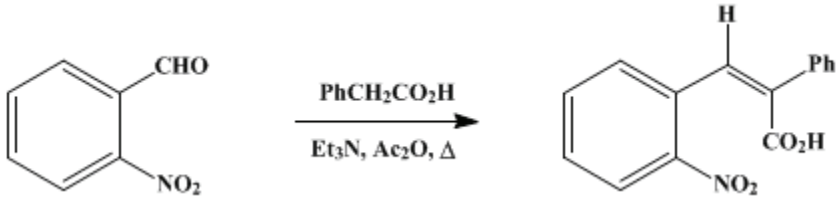
Organic Syntheses, Coll. Vol. 4, p.730 (1963); Vol. 35, p.89 (1955).

trans-*o*-NITRO-α-PHENYLCINNAMIC ACID

[Acrylic acid, *trans*-3-(*o*-nitrophenyl)-2-phenyl-]



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1. Procedure

A mixture of 30.2 g. (0.20 mole) of *o*-nitrobenzaldehyde,[2] 40 g. (0.29 mole) of phenylacetic acid, 100 ml. (1.08 moles) of acetic anhydride, and 20 g. (0.20 mole) of triethylamine is refluxed for 15 minutes in a 500-ml. flask. The solution is cooled to 90°, and 100 ml. of cold water is added over a 5-minute period at a rate that maintains the temperature above 90° (Note 1). The solution is filtered at 95–100° and cooled to 20°. *trans*-*o*-Nitro-α-phenylcinnamic acid precipitates in the form of light-orange crystals. It is separated by filtration and washed with 60 ml. of 50% acetic acid and with water. The dried acid weighs 39–42 g. (72–78%) and melts at 195–198°, which corresponds to a purity of about 98% (Note 2). After recrystallization from 500 ml. of toluene, it is in the form of yellow prisms weighing 38–39 g. (71–72%) and melting at 197.8–198.3°.

2. Notes

1. If the temperature gets too high, more cold water may be added. If the water is added too rapidly at first, the temperature drops below 90° and the rate of hydrolysis becomes very slow. Heating such an incompletely hydrolyzed mixture above 90° may cause it to boil violently.
2. Approximate melting points of mixtures of the *trans*- and *cis*-*o*-nitro-α-phenylcinnamic acids are as follows:

Per Cent *cis*	Sinter Point	M.P.
33		134–185°
23	130°	136–187°
13	131°	165–192°
8	135°	184–196°
3.5	140°	192–198°
1.5	170°	195–198°
—	196°	197.8–198.3°

3. Discussion

trans-*o*-Nitro-α-phenylcinnamic acid has been prepared by the condensation of *o*-nitrobenzaldehyde with sodium phenylacetate in the presence of acetic anhydride with[3] or without[4] fused zinc chloride as a catalyst. It has also been prepared by the condensation of *o*-nitrobenzaldehyde with phenylacetic acid in the presence of acetic anhydride and triethylamine.[5]

References and Notes

1. University of South Carolina, Columbia, South Carolina.
2. *Org. Syntheses Coll. Vol.* **3**, 641 (1955).
3. Pschorr, *Ber.*, **29**, 496 (1896).
4. Oglialoro and Rosini, *Gazz. chim. ital.*, **20**, 396 (1890).
5. Bakunin and Peccerillo, *Gazz. chim. ital.*, **65**, 1145 (1935).

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

acetic acid (64-19-7)

acetic anhydride (108-24-7)

toluene (108-88-3)

Phenylacetic acid (103-82-2)

zinc chloride (7646-85-7)

o-Nitrobenzaldehyde (552-89-6)

sodium phenylacetate (114-70-5)

triethylamine (121-44-8)

trans-o-Nitro-α-phenylcinnamic acid,
Acrylic acid, trans-3-(o-nitrophenyl)-2-phenyl- (19319-35-8)