



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.

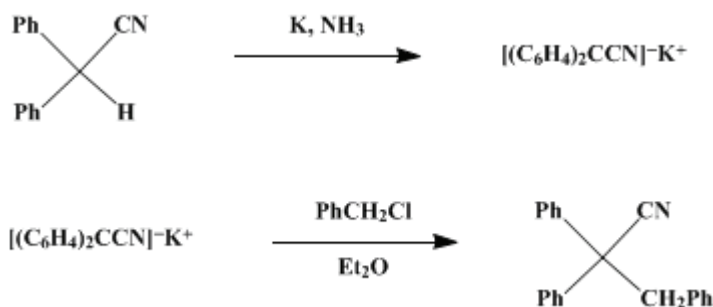
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

Organic Syntheses, Coll. Vol. 4, p.962 (1963); Vol. 39, p.73 (1959).

α,α,β -TRIPHENYLPROPIONITRILE

[Propionitrile, 2,2,3-triphenyl-]



Submitted by C. R. Hauser and W. R. Dunnivant¹.
Checked by Virgil Boekelheide and Donald R. Arnold.

1. Procedure

Caution! This preparation should be conducted in a hood to avoid exposure to ammonia.

A suspension of **potassium amide** (0.23 mole) in liquid **ammonia** is prepared in a 1-l. three-necked flask equipped with an air condenser (without drying tube), a ball-sealed mechanical stirrer, and a dropping funnel. Commercial anhydrous liquid **ammonia** (500 ml.) is introduced into the flask from a cylinder through an inlet tube. To the stirred **ammonia** is added a small piece of **potassium** metal. After the appearance of a blue color, a few crystals (about 0.25 g.) of **ferric nitrate hydrate** are added, followed by small pieces of **potassium** (**Note 1**) until 9.0 g. (0.23 g. atom) has been added. After all the **potassium** has been converted to the amide (**Note 2**), 44.6 g. (0.23 mole) of **diphenylacetonitrile** (**Note 3**) is added and the resulting greenish-brown solution is stirred for 5 minutes. To this is added, over 10 minutes, 30.5 g. (0.24 mole) of **benzyl chloride** (**Note 4**) in 100 ml. of anhydrous **ether**. The orange solution is stirred for 1 hour, and the **ammonia** is then evaporated on a steam bath as 300 ml. of anhydrous **ether** is being added. To the **ether** solution is added 300 ml. of water, whereupon the crude nitrile precipitates. The **ether** is then removed by distillation and the crude nitrile is collected on a Büchner funnel. The yield of crude, light-tan α,α,β -triphenylpropionitrile is 64 g. (98–99%). The nitrile is dissolved in 1.3 l. of hot **ethanol**, treated with **Norit**, and filtered. The filtrate is held at room temperature overnight, and the product collected by filtration. A second crop is obtained by concentration of the mother liquor. The total yield of α,α,β -triphenylpropionitrile, m.p. 126.5–127.5°, is 62.2–65.5 g. (95–99% yield) (**Note 5**).

2. Notes

1. The **potassium** is cut in about 0.5-g. pieces, stored under kerosene, and blotted with filter paper before addition.
2. Conversion is indicated by the discharge of the deep-blue color. This generally requires about 20 minutes.
3. **Diphenylacetonitrile** supplied by the Eastman Kodak Company was used without purification.
4. Eastman Kodak Company practical grade **benzyl chloride** was vacuum-distilled; the fraction, b.p. 63°/12 mm., was used.
5. Under comparable conditions the corresponding alkylations of **diphenylacetonitrile** with α -phenethyl chloride and benzhydryl chloride have been effected to form 2,3,3-triphenylbutyronitrile and 2,2,3,3-tetraphenylpropionitrile in yields of 88 and 96% respectively.²

3. Discussion

The method used is that of Hauser and Brasen.²

The benzylation of diphenylacetonitrile with benzyl chloride to form α,α,β -triphenylpropionitrile has been previously effected in 83% yield by sodium ethoxide in ethanol,³ in 67% yield by methylmagnesium iodide in ether,⁴ and in unreported yield by sodium amide in ether.⁵

References and Notes

1. Duke University, Durham, North Carolina. Work supported by the Office of Ordnance Research.
 2. Hauser and Brasen, *J. Am. Chem. Soc.*, **78**, 82 (1956).
 3. Neure, *Ann.*, **250**, 140 (1889).
 4. Sisido, Nozaki, and Kurihara, *J. Am. Chem. Soc.*, **72**, 2270 (1950).
 5. Ramart, *Bull. soc. chim. France*, [4] **35**, 196 (1924).
-

Appendix

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

ethanol (64-17-5)

ammonia (7664-41-7)

ether (60-29-7)

Norit (7782-42-5)

sodium ethoxide (141-52-6)

benzyl chloride (100-44-7)

potassium (7440-09-7)

methylmagnesium iodide (917-64-6)

α -phenethyl chloride (622-24-2)

sodium amide (7782-92-5)

Diphenylacetonitrile (86-29-3)

potassium amide

ferric nitrate hydrate

benzhydryl chloride (90-99-3)

α,α,β -TRIPHENYLPROPIONITRILE,
Propionitrile, 2,2,3-triphenyl- (5350-82-3)

2,3,3-triphenylbutyronitrile

2,2,3,3-tetraphenylpropionitrile

Copyright © 1921-2005, Organic Syntheses, Inc. All Rights Reserved