



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.

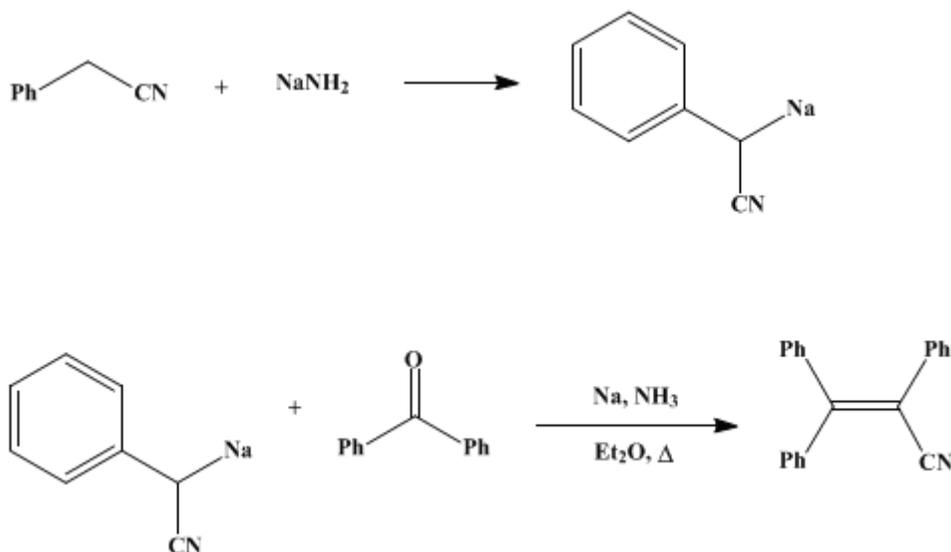
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.387 (1963); Vol. 31, p.52 (1951).

α,β -DIPHENYLCINNAMONITRILE

[Acrylonitrile, 2,3,3-triphenyl-]



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1. Procedure

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

A solution of sodium amide in liquid ammonia is prepared according to a procedure previously described (Note 1) in a 2-l. three-necked round-bottomed flask fitted with a reflux condenser attached to a soda-lime tower which is connected to a gas absorption trap,² a mercury-sealed mechanical stirrer, and an inlet tube. Anhydrous liquid ammonia (350 ml.) is introduced through the inlet tube, and about 0.3 g. of hydrated ferric nitrate and 13.7 g. (0.59 g. atom) of freshly cut sodium are added (Note 1) and (Note 2).

After the conversion of sodium to sodium amide is complete, the inlet tube is replaced with a 500-ml. dropping funnel and the flask is cooled in a bath of Dry Ice and trichloroethylene. Benzyl cyanide (69 g., 0.59 mole) (Note 3) is added dropwise with stirring during about 20 minutes. The cooling bath is removed, and the solution is stirred for an additional period of 20 minutes, after which 700 ml. of dry ether is added slowly through the dropping funnel. The solution is allowed to stand or is warmed gently with a water bath until it comes to room temperature. The rate of addition of ether and subsequent warming are controlled so that the ammonia which is vaporized passes through the gas absorption trap rather than escaping in part through the mercury seal of the stirrer. When most of the ammonia has been removed, an additional 300-ml. portion of dry ether is added (Note 4). The flask is then heated on a hot water bath. By turning off the cooling water of the reflux condenser for a short time, a small amount of ether is allowed to distil out of the reaction mixture in order to remove as much of the ammonia as possible (Note 5). The condenser water is then turned on again, and dry nitrogen lines under a positive pressure of 2–3 cm. of mercury (maintained by a T-tube dipping into mercury) are attached to the top of the dropping funnel and the reflux condenser. A solution of 200 g. (1.1 moles) of benzophenone (Note 6) and (Note 7) in 300 ml. of dry ether (Note 8) is added through the dropping funnel during a period of 15 minutes, and the mixture is heated under reflux with stirring for 24 hours. At the end of this period the solution contains a reddish brown precipitate. The mixture is allowed to cool, and 250 ml. of water

is added slowly through the dropping funnel. The aqueous and ether layers are separated in a 2-l. separatory funnel, and the water layer is filtered directly through a 15-cm. Büchner funnel. The aqueous filtrate is discarded. The ether layer also is run into the funnel, and the filtrate is concentrated to a volume of about 80 ml. The solid that separates is added to the solid previously collected on the filter. The crude reddish brown product is recrystallized from 600 ml. of glacial acetic acid. α,β -Diphenylcinnamonitrile separates as white crystals which are collected on a suction filter, washed with 100 ml. of water, and dried at 110° for 24 hours. The yield of a pure product melting at 166–167° is 83–110 g. (50–66%).

2. Notes

1. The procedures for preparing sodium amide and the sodium derivative of benzyl cyanide are described in *Organic Syntheses*.³ Another procedure for preparing sodium amide (p.763) is convenient and should be equally satisfactory.
2. If vaporization of ammonia reduces the liquid volume to less than 250 ml. before the conversion of sodium to sodium amide is complete, more ammonia should be added through the inlet tube.
3. The benzyl cyanide used should be washed with warm 50% sulfuric acid to remove benzyl isocyanide⁴ and redistilled.
4. Some of the ether is lost through the reflux condenser during vaporization of the ammonia.
5. Traces of ammonia remain in the reaction mixture after this procedure.
6. A smaller excess of benzophenone results in a lowered yield.
7. The benzophenone should be dried by redistillation or storage over phosphorus pentoxide in a vacuum desiccator.
8. The mixture of benzophenone and ether should be warmed gently until the solid dissolves.

3. Discussion

α,β -Diphenylcinnamonitrile has been prepared by the condensation of benzophenone with the sodium⁵ or lithium⁶ derivative of benzyl cyanide, or from benzophenone, benzyl cyanide, and sodium ethoxide.⁷ It has been obtained also by heating benzyl cyanide with benzophenone dichloride at 200°.⁸

References and Notes

1. State University of Iowa, Iowa City, Iowa.
2. *Org. Syntheses Coll. Vol. 2*, 4 (1943).
3. *Org. Syntheses Coll. Vol. 3*, 219 (1955).
4. *Org. Syntheses Coll. Vol. 1*, 108 (1941).
5. Bodroux, *Bull. soc. chim. France*, [4] **9**, 758 (1911).
6. Ivanov and Vasilev, *Compt. rend. acad. bulgare sci.*, **10**, No. 1, 53 (1957) [*C. A.*, **52**, 5353 (1958)].
7. Stobbe and Zeitschel, *Ber.*, **34**, 1967 (1901).
8. Heyl and Meyer, *Ber.*, **28**, 1798, 2785 (1895).

Appendix

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

sodium derivative of benzyl cyanide

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

ammonia (7664-41-7)

ether (60-29-7)

nitrogen (7727-37-9)

mercury (7439-97-6)

Benzophenone (119-61-9)

sodium (13966-32-0)

benzophenone dichloride

sodium ethoxide (141-52-6)

Benzyl cyanide (140-29-4)

Benzyl isocyanide (10340-91-7)

sodium amide (7782-92-5)

ferric nitrate

Acrylonitrile, 2,3,3-triphenyl-,
 α,β -Diphenylcinnamionitrile (6304-33-2)

phosphorus pentoxide (1314-56-3)