



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.

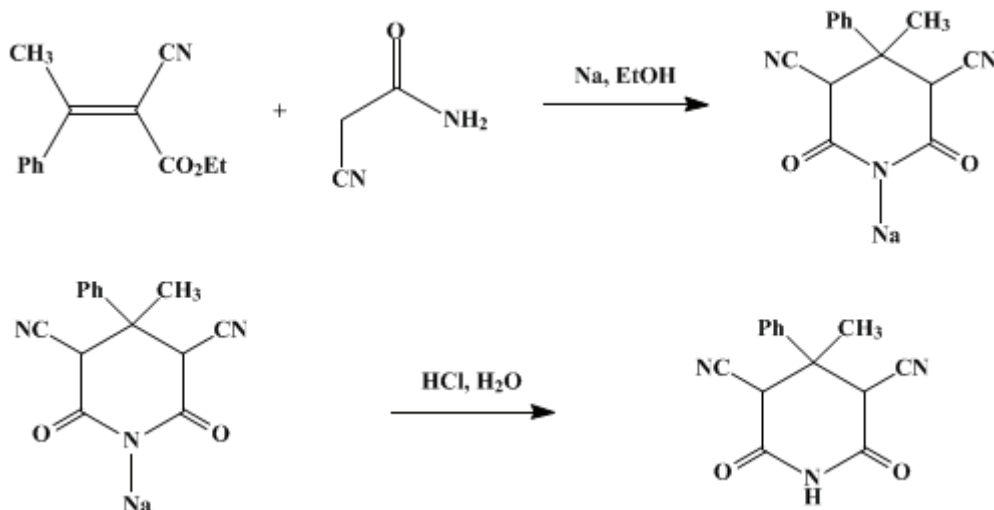
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.662 (1963); Vol. 39, p.52 (1959).

β -METHYL- β -PHENYL- α,α' -DICYANOGLUTARIMIDE

[Glutarimide, 2,4-dicyano-3-methyl-3-phenyl-]



Submitted by S. M. McElvain and David H. Clemens¹.

Checked by W. E. Parham, Perry W. Kirklin, Jr., and Wayland E. Noland.

1. Procedure

In a 2-l. Erlenmeyer flask fitted with a reflux condenser and arranged for magnetic stirring are placed 400 ml. of absolute ethanol (Note 1) and 11.5 g. (0.5 g. atom) of sodium added in small portions. After the sodium has reacted (Note 2), the clear solution is cooled to room temperature, and 42.0 g. (0.5 mole) of finely powdered cyanoacetamide (Note 3) is added with stirring over a period of 1 minute. Immediately thereafter 107.6 g. (0.5 mole) of ethyl (1-phenylethylidene)cyanoacetate (p. 463) is added. After about 20 minutes, the mixture becomes homogeneous and is allowed to stand at room temperature for 2 hours. Water (650 ml.) is added, followed by 100 ml. of concentrated hydrochloric acid in 1 portion. The resulting suspension is stirred thoroughly with a glass rod and placed in a refrigerator overnight. The product is then filtered by suction. The filter cake is sucked as dry as possible using a rubber dam, stirred to a paste with a mixture of 150 ml. of water and 50 ml. of 95% ethanol, and sucked dry. This process is repeated using 200 ml. of water, and the product is dried to constant weight in an oven at 45°. The yield is 114–116 g. (90–92%) of the dicyanoglutarimide, m.p. 274–278° (dec.) (Note 4).

2. Notes

1. Commercially available absolute ethanol is used without further drying.
2. The checkers report that the sodium ethoxide solution should be used promptly in order to avoid the formation of colored impurities.
3. Eastman Kodak white label grade is used after grinding in a mortar.
4. Recrystallization from absolute ethanol gives glistening plates melting at 286–287° (dec.).

3. Discussion

β -Methyl- β -phenyl- α,α' -dicyanoglutarimide has been prepared in low yield by the Guareschi condensation of acetophenone, ethyl cyanoacetate, and ammonia.²

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 4, 664](#)

References and Notes

1. University of Wisconsin, Madison, Wisconsin.
 2. Phalnikar and Nargund, *J. Univ. Bombay*, **6**, Pt. II, 102 (1937) [*C. A.*, **32**, 3763 (1938)].
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[β-METHYL-β-PHENYL-α,α'-DICYANOGLUTARIMIDE](#)

[ethanol \(64-17-5\)](#)

[hydrochloric acid \(7647-01-0\)](#)

[ammonia \(7664-41-7\)](#)

[Acetophenone \(98-86-2\)](#)

[sodium \(13966-32-0\)](#)

[sodium ethoxide \(141-52-6\)](#)

[CYANOACETAMIDE \(107-91-5\)](#)

[Ethyl cyanoacetate \(105-56-6\)](#)

[ETHYL \(1-PHENYLETHYLIDENE\)CYANOACETATE](#)

[Glutarimide, 2,4-dicyano-3-methyl-3-phenyl- \(6936-95-4\)](#)