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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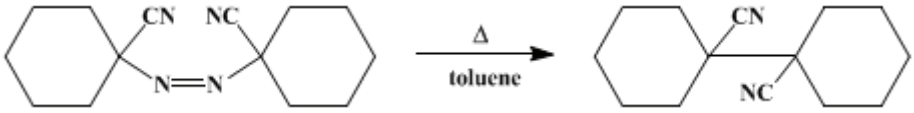
1,1'-DICYANO-1,1'-BICYCLOHEXYL

[[Bicyclohexyl]-1,1'-dicarbonitrile]



Submitted by C. G. Overberger and M. B. Berenbaum[1].
Checked by N. J. Leonard and E. H. Mottus.

1. Procedure

In a 200-ml. flask equipped with a reflux condenser are placed 20 g. (0.082 mole) of 1,1'-azo-*bis*-1-cyclohexanenitrile (p. 66) and 50 ml. of toluene. The solution is heated under gentle reflux for 8 hours, and the reaction mixture is placed in a refrigerator overnight. The product is collected on a Büchner funnel and air-dried; yield 11.5–12.2 g. (65–69%); m.p. 224.5–225.5° (Note 1).

2. Notes

1. Tetramethylsuccinonitrile can be prepared from 2,2'-azo-*bis*-isobutyronitrile in a similar manner.[2] In this case petroleum ether (b.p. 60–70°) is added to the reaction mixture before cooling it overnight. The yield is 75–81% of the theoretical amount; m.p. 168–170°. Sublimation at 20–50 mm. raises the melting point to 170.5–171.5° with very little loss of material.

3. Discussion

1,1'-Dicyano-1,1'-bicyclohexyl has been prepared previously by Hartman[3] in a similar manner. The procedure has been substantiated by Overberger, O'Shaughnessy, and Shalit.[4]

References and Notes

1. Polytechnic Institute of Brooklyn, Brooklyn, New York.
2. Thiele and Heuser, *Ann.*, **290**, 1 (1896).
3. Hartman, *Rec. trav. chim.*, **46**, 150 (1927).
4. Overberger, O'Shaughnessy, and Shalit, *J. Am. Chem. Soc.*, **71**, 2661 (1949).

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

petroleum ether

toluene (108-88-3)

Tetramethylsuccinonitrile (3333-52-6)

2,2'-Azo-bis-isobutyronitrile

1,1'-AZO-bis-1-CYCLOHEXANENITRILE (2094-98-6)

1,1'-DICYANO-1,1'-BICYCLOHEXYL,
[Bicyclohexyl]-1,1'-dicarbonitrile (18341-40-7)