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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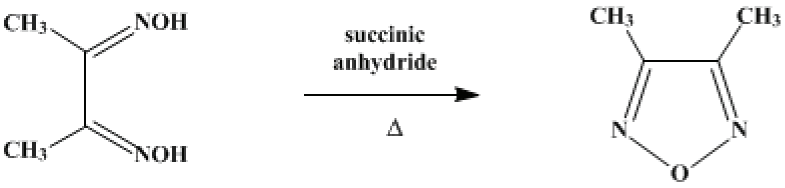
DIMETHYLFURAZAN

[Furazan, 3,4-dimethyl-]



Submitted by Lyell C. Behr and John T. Brent[1].
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1. Procedure

One hundred grams (1 mole) of succinic anhydride and 116 g. (1 mole) of dimethylglyoxime (Note 1), both finely ground, are intimately mixed and introduced into a 1-l. three-necked flask which is equipped with a sealed mechanical stirrer, a thermometer reaching nearly to the bottom of the flask, and an outlet tube connected to a water-cooled condenser arranged for distillation. The mixture is heated slowly with an electric mantle or oil bath, and stirring is commenced as soon as practicable (Note 2). The mixture liquefies at about 100°, and a rapid reaction begins at 150–170° accompanied by a sudden rise in temperature. This initial rapid reaction can be controlled readily by removing the heater when the temperature reaches 170° and applying a cooling bath until the inside temperature is 150° (Note 3). Heat is then applied again, and distillation of the product begins at a flask temperature of 160° and continues until the temperature reaches 200°. The stirrer is stopped, the receiver is changed, and, after the flask and contents have cooled to about 120°, 50 ml. of water is added. The thermometer is replaced by an inlet tube, and steam is passed in until no more insoluble material passes over. Usually collection of about 200–300 ml. of distillate is sufficient. The distillate is extracted with two 100-ml. portions of ether, and the extracts are combined with the dimethylfurazan obtained by direct distillation (Note 4). The ether solution is dried for a short time over anhydrous magnesium sulfate. The drying agent is removed by filtration, and the ether is evaporated on a steam bath. The residue is distilled at atmospheric pressure through a short column, and after a fore-run, consisting chiefly of biacetyl, the dimethylfurazan distils at 154–159° as a colorless liquid, n_D^{25} 1.4234–1.4243, m.p. −7.2 to −6.6°. The yield is 59–63 g. (60–64%).

2. Notes

1. A good grade of both reagents should be used. The better-quality products supplied by the Eastman Kodak Company are satisfactory.
2. The mixture may be difficult to stir mechanically below its melting point unless a powerful motor is used.
3. The temperature usually climbs to about 190° during this interval, and some dimethylfurazan distils rapidly.
4. The product obtained by direct distillation contains some water, but the dimethylfurazan can be readily separated.

3. Discussion

Dimethylfurazan has been obtained from dimethylglyoxime by heating with water, aqueous ammonia, or aqueous sodium hydroxide.[2,3] The usual acid dehydrating agents fail.

References and Notes

1. Mississippi State University, State College, Mississippi. Work supported in part by a gift from the Research Corporation.
2. Wolff, *Ber.*, **28,** 69 (1895).
3. Rimini, *Gazz. chim. ital.,* **25,** II, 266 (1895).

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

ammonia	(7664-41-7)
ether	(60-29-7)
sodium hydroxide	(1310-73-2)
magnesium sulfate	(7487-88-9)
Succinic anhydride	(108-30-5)
DIMETHYLGLYOXIME	(95-45-4)
biacetyl	(431-03-8)
Dimethylfurazan, Furazan, 3,4-dimethyl-	(4975-21-7)