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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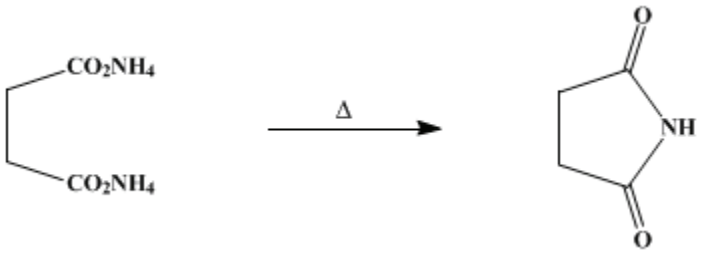
Organic Syntheses, Coll. Vol. 2, p.562 (1943); Vol. 16, p.75 (1936).

SUCCINIMIDE



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1. Procedure

In a 1-l. distilling flask, fitted with a side arm 40 cm. in length and not less than 10 mm. in internal diameter (Note 1), is placed 236 g. (2 moles) of succinic acid. To this is added slowly, with cooling and shaking, 270 cc. (243 g., 4 moles) of 28 per cent aqueous ammonia (sp. gr. 0.90). Most of the acid dissolves, forming a clear solution. The flask is set for downward distillation, and a water-cooled 500-cc. distilling flask is attached to the side arm. Provision may be made for removal of ammonia from the side tube of the receiver. The mixture is heated gently over a free flame; solution takes place rapidly, and a small amount of uncombined ammonia passes over with the first portions of the distillate. The temperature of the vapor rises to 100° and remains at this point until about 200 cc. of water has distilled. The flame is then increased, and the ammonium succinate begins to decompose with evolution of ammonia; the temperature of the vapor falls to 97° during the distillation of the next 30 cc. When the vapor temperature has risen to 102°, the receiver is changed and an intermediate fraction collected from 102° to 275°. Succinimide then distils and is collected over the range 275–289°, largely at 285–289°. Decomposition takes place to a small extent with formation of a black tar; the distillation is stopped when the tarry residue begins to decompose with evolution of yellow fumes.

The crude succinimide, which solidifies completely, amounts to about 168 g. The intermediate fraction is redistilled from a smaller flask and furnishes about 10 g. more of crude succinimide boiling between 275° and 289°. The two portions of crude succinimide are combined and crystallized from 95 per cent ethyl alcohol, employing 1 cc. of solvent for every gram of product. If the mixture is chilled to 0° for some hours before filtration and about 25 cc. of cold alcohol is employed for washing the crystals, the first crop amounts to 163–164 g. (82–83 per cent of the theoretical amount). On concentrating the mother liquor to one-third of its volume, a second crop of 4–5 g. can be secured (Note 2). The product melts at 123–125° and contains no water of crystallization.

2. Notes

1. It is essential to employ a side arm of at least this diameter in order to avoid clogging by crystals of succinimide when this first passes over.
2. A further small quantity of a less pure product can be obtained by evaporating the dark mother liquor to dryness and recrystallizing the residue from fresh 95 per cent alcohol.

3. Discussion

Succinimide has usually been prepared by heating succinic acid in a current of ammonia[1], [2] or by distilling ammonium succinate.[1], [3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 19

References and Notes

1. Fehling, Ann. **49**, 198 (1844).
2. Franchimont and Friedmann, Rec. trav. chim. **25**, 79 (1906).
3. Bunge, Ann. Suppl. **7**, 118 (1870); Menschutkin, Ann. **162**, 166 (1872).

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

ethyl alcohol,
alcohol (64-17-5)

ammonia (7664-41-7)

Succinic acid (110-15-6)

Succinimide (123-56-8)

ammonium succinate