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.

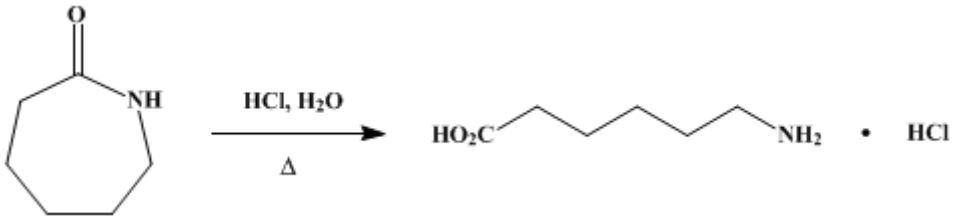
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.017.0007

Organic Syntheses, Coll. Vol. 2, p.28 (1943); Vol. 17, p.7 (1937).

ε-AMINOCAPROIC ACID

[Caproic acid, ε-amino-]



Submitted by J. C. Eck
Checked by Louis F. Fieser and C. H. Fisher.

1. Procedure

In a 500-cc. round-bottomed flask, 50 g. (0.44 mole) of 2-ketohexamethylenimine (p. 371) is added to a solution of 45 cc. of concentrated hydrochloric acid (sp. gr. 1.19) in 150 cc. of water. The solution is boiled for about one hour until it becomes clear (Note 1) and evaporated to dryness under reduced pressure on a steam bath.

The resulting ε-aminocaproic acid hydrochloride is converted to the free acid by a procedure similar to that used in the preparation of *dl*-alanine (Org. Syn. Coll. Vol. I, **1941**, 21). The hydrochloride is dissolved in 1 l. of water in a 1.5-l. beaker and treated successively with 50 g. of powdered litharge, 25 g. of powdered litharge, 5 g. of freshly precipitated lead hydroxide, 25 g. of powdered silver oxide (Note 2), and, finally, hydrogen sulfide. During this procedure, the original volume is maintained by the addition of small amounts of water.

After the complete removal of halogen and metallic ions, the solution is concentrated to a volume of about 100 cc., and 300 cc. of absolute alcohol is added. Then the amino acid is precipitated by slowly adding 500 cc. of ether with stirring and cooling.

The resulting ε-aminocaproic acid is collected on a suction filter and dried in a desiccator. The yield of ε-aminocaproic acid melting at 201–203° is 52.5–53.5 g. (90–92 per cent of the theoretical amount).

2. Notes

1. This indicates that the hydrolysis is complete.
2. The exact amount of silver oxide required may be determined by titrating a sample of the solution with silver nitrate by the Volhard method.

3. Discussion

ε-Aminocaproic acid has been prepared by the hydrolysis of ε-benzoylaminocapronitrile,[1] by the hydrolysis of ethyl δ-phthalimidobutylmalonate,[2] and from cyclohexanone oxime by rearrangement and hydrolysis.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 76
- Org. Syn. Coll. Vol. 4, 39

References and Notes

1. von Braun and Steindorff, Ber. **38**, 176 (1905); von Braun, ibid. **40**, 1839 (1907); Ruzicka and Hugoson, Helv. Chim. Acta **4**, 479 (1921); Marvel, MacCorquodale, Kendall, and Lazier, J. Am. Chem. Soc. **46**, 2838 (1924).
2. Gabriel and Maass, Ber. **32**, 1266 (1899).
3. Wallach, Ann. **312**, 188 (1900); Eck and Marvel, J. Biol. Chem. **106**, 387 (1934).

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

alcohol (64-17-5)

hydrochloric acid (7647-01-0)

ether (60-29-7)

lead hydroxide

silver oxide (20667-12-3)

hydrogen sulfide (7783-06-4)

silver nitrate (7761-88-8)

ε-AMINOCAPROIC ACID,
Caproic acid, ε-amino- (60-32-2)

2-Ketohexamethylenimine (105-60-2)

ε-aminocaproic acid hydrochloride

ε-benzoylaminocapronitrile

ethyl δ-phthalimidobutylmalonate

Cyclohexanone oxime (100-64-1)

DL-Alanine (302-72-7)

Copyright © 1921-2005, Organic Syntheses, Inc. All Rights Reserved