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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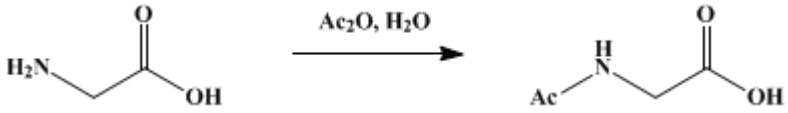
Organic Syntheses, Coll. Vol. 2, p.11 (1943); Vol. 19, p.4 (1939).

ACETYLGLYCINE

[Aceturic acid]



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1. Procedure

In a 1-l. Erlenmeyer flask provided with a mechanical stirrer are placed 75 g. (1 mole) of glycine (Org. Syn. Coll. Vol. I, **1941**, 298) and 300 cc. of water. The mixture is stirred vigorously until the glycine is almost completely dissolved, when 215 g. (2 moles) of 95 per cent acetic anhydride (Note 1) is added in one portion. Vigorous stirring is continued for fifteen to twenty minutes, during which time the solution becomes hot and acetylglycine may begin to crystallize. The solution is placed in the refrigerator (Note 2) overnight to effect complete crystallization. The precipitate is collected on a Büchner funnel, washed with ice-cold water, and dried at 100–110°. This product weighs 75–85 g. and melts at 207–208°. The combined filtrate and washings are evaporated to dryness under reduced pressure on a water bath at 50–60°. The residue on recrystallization from 75 cc. of boiling water yields a second fraction, of 20–30 g., which melts at 207–208° after being washed with ice-cold water and dried at 100–110°. An additional 4–6 g. of only slightly less pure product may be obtained from the mother liquor by concentration. The total yield is 104–108 g. (89–92 per cent of the theoretical amount) (Note 3).

2. Notes

1. The equivalent quantity of 90 per cent acetic anhydride may be used.
2. The refrigerator used by the checkers maintained a temperature of 5–7°.
3. The method may be employed to acetylate most α-amino acids with only slight modifications depending upon the solubility of the particular amino acid. When optically active amino acids are acetylated, there is little or no racemization.[1]

3. Discussion

Acetylglycine has been prepared by the interaction of acetyl chloride and the silver salt of glycine in dry ether or benzene;[2], [3] by the action of acetic anhydride on glycine suspended in warm benzene;[3] by heating glycine with acetic anhydride;[4] by treating an aqueous solution of glycine or its sodium salt with ketene;[5] and by treating an aqueous alkaline solution of glycine with acetic anhydride.[6]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 1

References and Notes

1. Behr and Clarke, J. Am. Chem. Soc. **54**, 1631 (1932).
2. Kraut and Hartmann, Ann. **133**, 105 (1865).
3. Curtius, Ber. **17**, 1665 (1884).
4. Radenhausen, J. prakt. Chem. (2) **52**, 437 (1895).
5. Bergmann and Stern, Ber. **63**, 437 (1930).

6. Chattaway, J. Chem. Soc. **1931**, 2495.

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

glycine or its sodium salt

Benzene (71-43-2)

ether (60-29-7)

acetic anhydride (108-24-7)

acetyl chloride (75-36-5)

Glycine (513-29-1)

Ketene (463-51-4)

Acetylglycine,
Aceturic acid (543-24-8)

silver salt of glycine