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.

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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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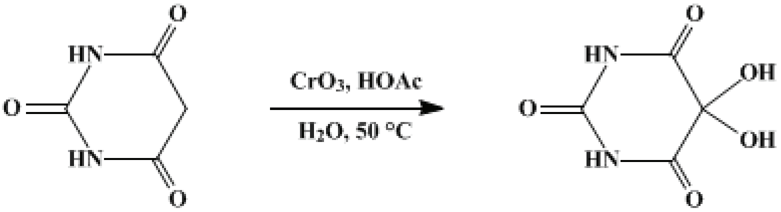
ALLOXAN MONOHYDRATE

[Barbituric acid, 5,5-dihydroxy-]



Submitted by A. V. Holmgren and Wilhelm Wenner[1].
Checked by T. L. Cairns and R. W. Upson.

1. Procedure

In a 2-l. three-necked, round-bottomed flask with glass joints are placed 850 g. of commercial glacial acetic acid and 100 ml. of water. The flask is fitted with a stirrer. One of the side necks carries a reflux condenser and a thermometer reaching to the bottom of the flask; the other is provided with a stopper which can be replaced by a powder funnel. The flask is surrounded by a water bath. At room temperature 156 g. (1.53 moles) of 98–99% chromium trioxide (Note 1) is added, and the mixture is stirred for about 15 minutes to effect solution of the oxidizing agent.

One hundred and twenty-eight grams (1 mole) of barbituric acid is added in the course of about 25 minutes in portions approximating 15–20 g. The temperature of the mixture rises from about 25–30° at the beginning of the reaction to 50° and is held at that value until all the barbituric acid has been added (Note 2). During the addition, alloxan monohydrate begins to crystallize. The temperature of the solution is held at 50° for 25–30 minutes after completion of the addition of barbituric acid. Then the reaction slurry, which contains the major amount of alloxan monohydrate in crystalline form, is cooled to 5–10° and filtered through a 5-in. Büchner funnel fitted with a piece of filter cloth. The product is washed while still on the funnel with cold glacial acetic acid until the washings are practically colorless. In order to effect rapid drying, the acetic acid is finally washed out of the filter cake by means of 100–200 ml. of ether. The yellow alloxan monohydrate weighs 120–125 g. (75–78%) after drying; m.p. 254° (dec.). It is pure enough for most purposes (Note 3).

2. Notes

1. This amount was found to give best yields.
2. It is very important that the temperature does not rise above 50°. If the addition of barbituric acid is carried out too rapidly, the temperature rise cannot be checked satisfactorily and the yield may drop considerably.
3. If entirely pure alloxan monohydrate is desired, this material is recrystallized according to the directions in an earlier volume of this series.[2]

3. Discussion

The methods for the preparation of alloxan have been reviewed earlier.[2] The present method is essentially that of Wenner.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 25

References and Notes

1. Hoffmann-La Roche, Inc., Nutley, New Jersey.
2. *Org. Syntheses Coll. Vol.* **3**, 37, 39 (1955).
3. Wenner, U. S. pat. 2,445,898 [*C. A.*, **43**, 2227 (1949)].

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

acetic acid (64-19-7)

ether (60-29-7)

Barbituric acid (67-52-7)

chromium trioxide (1333-82-0)

Alloxan monohydrate (2244-11-3)

Alloxan (50-71-5)

Barbituric acid, 5,5-dihydroxy- (3237-50-1)