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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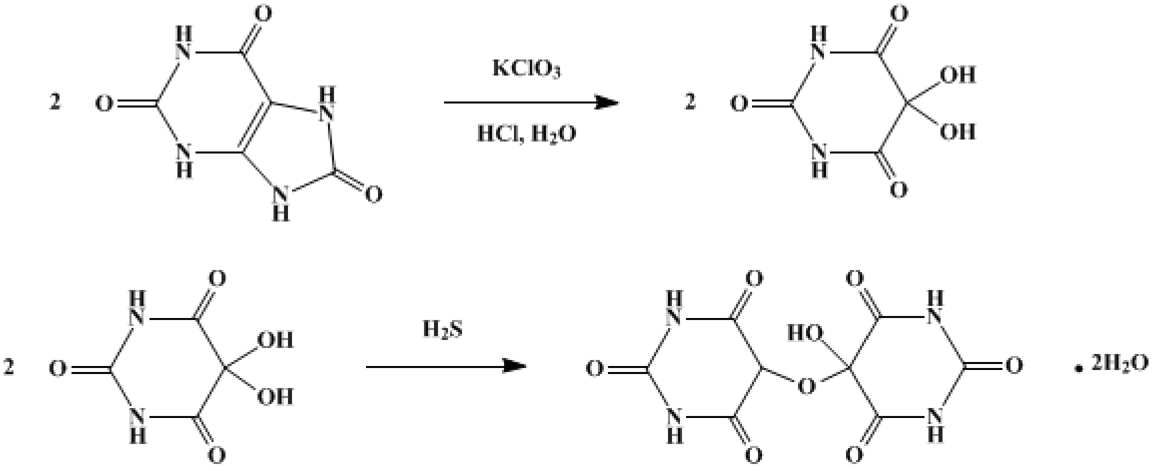
ALLOXANTIN DIHYDRATE



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1. Procedure

In a 500-ml. three-necked flask, provided with a stirrer, are placed 15 g. (0.09 mole) of finely powdered uric acid, 30 g. (25.2 ml.) of concentrated hydrochloric acid, and 40 ml. of water. The mixture is warmed to 30°, the stirrer is started, and 4 g. (0.014 mole) of finely powdered potassium chlorate is added in small portions during a period of not less than 45 minutes (Note 1). Most of the uric acid dissolves; any undissolved material is removed by filtration through a fritted-glass filter. The clear filtrate is diluted with 30 ml. of water, and a rapid stream of hydrogen sulfide is led into it until it is saturated (about 10–15 minutes). Sulfur and alloxantin separate, and the mixture is cooled for 2–3 hours in an ice bath until the separation is complete.

The solid is collected on a Büchner funnel and washed with three 30-ml. portions of cold water. The alloxantin is dissolved by boiling the wet solid for 15 minutes with 250 ml. of water, and the *hot* solution is filtered to remove the sulfur (Note 2). Alloxantin dihydrate crystallizes from the filtrate in glistening plates which should be pressed as dry as possible on a Büchner filter, washed with about 30 ml. of ether, and dried in a vacuum desiccator (Note 3). The yield is 8–10 g. (55–69%) (Note 4). The product melts with decomposition at 234–238° (Note 5), and is pure enough for most purposes.

2. Notes

1. It is important that the temperature of the reaction mixture be kept near 30°. The potassium chlorate must be added slowly.
2. Since alloxantin is difficulty soluble, it is desirable to make a second extraction of the sulfur to be certain that all the alloxantin has been removed.
3. The compound gradually turns pink on standing in the air. It should be stored in a tightly stoppered bottle or kept in a vacuum desiccator over calcium chloride.
4. If larger amounts of material are desired it is best to oxidize several 15-g. portions of uric acid and to combine the sulfur-alloxantin mixtures for the extraction.
5. The water of crystallization may be removed by heating the dihydrate at 120–150° under reduced pressure for 2 hours. The melting points reported in the literature vary considerably. The anhydrous material turns yellow at about 225–230° and decomposes at temperatures ranging from 238–242° to 253–255°, depending on the rate of heating. The instantaneous decomposition temperatures determined on the Maquenne block were 270–275°.

3. Discussion

This procedure is essentially that described in the laboratory manual by Fischer and Helferich.[1]

Alloxantin has been obtained by the oxidation of uric acid with nitric acid, followed by reduction with hydrogen sulfide;[2] by oxidation of uric acid with potassium chlorate, followed by reduction with stannous chloride;[3] by condensation of alloxan with dialuric acid in aqueous solution;[4] and by oxidation of dialuric acid.[5] A preparation from alloxan has been published.[6]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 37
- Org. Syn. Coll. Vol. 3, 46
- Org. Syn. Coll. Vol. 4, 25

References and Notes

1. Fischer and Helferich, *Anleitung zur Darstellung organischer Präparate*, 10th ed., pp. 66, 67, Braunschweig, 1922; Doja and Mokeet, *J. Indian Chem. Soc.*, **13**, 542 (1936).
2. Deniges, *Bull. soc. pharm. Bordeaux*, **66**, 8–12 (1928) [*C. A.*, **23**, 4160 (1929)].
3. Davidson and Epstein, *J. Org. Chem.*, **1**, 305 (1936).
4. Wöhler and Liebig, *Ann.*, **26**, 279 (1838); Behrend and Friederichs, *Ann.*, **344**, 1 (1906).
5. Baeyer, *Ann.*, **127**, 11 (1863).
6. *Org. Syntheses*, **33**, 3 (1953).

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

calcium chloride (10043-52-4)

hydrochloric acid (7647-01-0)

ether (60-29-7)

nitric acid (7697-37-2)

hydrogen sulfide (7783-06-4)

stannous chloride

sulfur (7704-34-9)

potassium chlorate (3811-04-9)

uric acid

alloxantin (76-24-4)

Alloxan (50-71-5)

Alloxantin dihydrate (6011-27-4)

dialuric acid (444-15-5)