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.

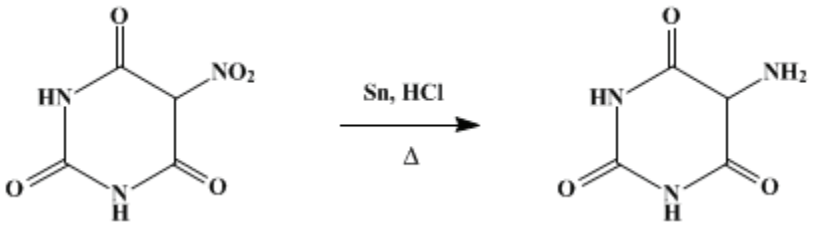
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.012.0084

Organic Syntheses, Coll. Vol. 2, p.617 (1943); Vol. 12, p.84 (1932).

URAMIL



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1. Procedure

In a 5-l. flask are placed 100 g. (0.44 mole) of nitrobarbituric acid (p. 440) and 600 cc. of concentrated hydrochloric acid, and the mixture is heated on a boiling water bath. To the hot mixture are added 250 g. (2.1 gram atoms) of mossy tin and 400 cc. of hydrochloric acid over a period of about thirty minutes; heating is continued until there is no yellow color in the liquid (Note 1). The solution is treated with about 3 l. more of concentrated hydrochloric acid and heated until all the white solid is in solution. Norite is added, and the hot mixture is filtered through a sintered-glass funnel (Note 2). The filtrate is allowed to stand in an icebox overnight, and then the precipitate (Note 3) of uramil is collected on a filter and washed with liberal quantities of dilute hydrochloric acid and finally with water (Note 4). The filtrate is concentrated under reduced pressure to about 1 l. and cooled overnight. The additional uramil thus obtained is collected on a Büchner funnel and added to the first product (Note 5). The uramil is dried in a desiccator over concentrated sulfuric acid, and finally over 40 per cent sodium hydroxide to remove hydrochloric acid (Note 6).

Uramil is a fine, white powder which becomes pink to red on standing. The yield of a product which does not melt below 400° is 40–46 g. (63–73 per cent of the theoretical amount).

2. Notes

1. Nitrobarbituric acid forms a yellow aqueous solution, but, as the color is weak in concentrated hydrochloric acid solution, no trace of it should show at the end of the reaction.
2. If a sintered-glass funnel is not available, the solution may be filtered through a half-inch layer of decolorizing carbon on a double filter paper. After the uramil is once dissolved in the concentrated hydrochloric acid it comes out of solution very slowly, and, if filtered promptly, the solution may be cooled to 60–80° with little loss of product.
3. If, as happens occasionally, the uramil does not crystallize, the solution must be concentrated and cooled again.
4. Unless the product is washed thoroughly it will contain tin salts.
5. To test for uramil an ammoniacal solution is boiled in the air. A positive test is the appearance of a pink color which gradually grows deeper. The reaction proceeds more rapidly in the presence of mercuric oxide.
6. If the material is dried in the air or in an oven, a pink product is almost always obtained. The pink color is produced more rapidly if ammonia or amines are present in the air.

3. Discussion

Uramil has been obtained by boiling alloxantin with ammonium chloride;[1] by reduction of nitrosobarbituric acid or nitrobarbituric acid with hydrogen iodide;[2] and by reduction of alloxan phenylhydrazone with tin and hydrochloric acid.[3]

References and Notes

1. Wöhler and Liebig, Ann. **26**, 309 (1838).
2. Baeyer, ibid. **127**, 223 (1863).
3. Kühling, Ber. **31**, 1973 (1898).

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

dilute hydrochloric acid

sulfuric acid (7664-93-9)

hydrochloric acid (7647-01-0)

ammonia (7664-41-7)

ammonium chloride (12125-02-9)

sodium hydroxide (1310-73-2)

tin (7440-31-5)

mercuric oxide (21908-53-2)

decolorizing carbon,
Norite (7782-42-5)

hydrogen iodide (10034-85-2)

Nitrobarbituric acid (480-68-2)

Uramil (118-78-5)

alloxantin (76-24-4)

nitrosobarbituric acid

alloxan phenylhydrazone