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.

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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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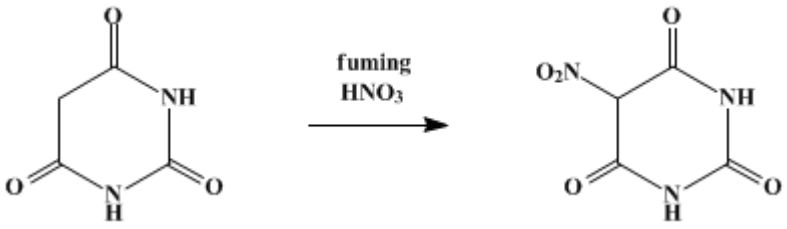
Organic Syntheses, Coll. Vol. 2, p.440 (1943); Vol. 12, p.58 (1932).

NITROBARBITURIC ACID

[Barbituric acid, 5-nitro-]



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1. Procedure

In a 2-l. flask, equipped with a mechanical stirrer and surrounded by an ice bath, is placed 143 cc. of fuming nitric acid (sp. gr. 1.52). Stirring is started, and 100 g. (0.61 mole) of barbituric acid (p. 60) is added over a period of two hours; the temperature is kept below 40° during the addition. The mixture is stirred for one hour after the barbituric acid has been added, and stirring is continued while 430 cc. of water is added and the solution is cooled to 10°. The mixture is filtered, and the residue is washed with cold water and dried on a glass tray at 60–80° (Note 1). The nitrobarbituric acid is dissolved by adding it to 860 cc. of boiling water in a 2-l. flask and heating the mixture on a boiling water bath while steam is blown in until solution is complete (Note 2). After filtration and cooling overnight, the crystals are removed, washed with cold water, and dried in trays in an oven at 90–95° for two to three hours. The product melts with decomposition at 181–183° when heated rapidly. The yield is 139–141 g. (Note 3). On drying the product at 110–115° for two to three hours, the yield is 90–94 g. (85–90 per cent of the theoretical amount) of an anhydrous compound which melts with decomposition at 176°.

2. Notes

1. Unless the product is dried before recrystallization it is difficult to remove all the nitric acid, and the final product will have a strong odor of nitric acid.
2. If a clear yellow solution is not obtained, Norite should be added before filtering.
3. This yield is slightly above the theoretical yield of 139 g., but this is probably due to a greater degree of hydration than is indicated in the formula.

3. Discussion

Nitrobarbituric acid has been prepared by oxidation of violuric acid,[1] and by treatment of barbituric acid with fuming nitric acid[2] or concentrated nitric acid.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 617

References and Notes

1. Ceresole, Ber. **16**, 1134 (1883).
2. Baeyer, Ann. **130**, 140 (1864).
3. Fredholm, Z. anal. Chem. **104**, 400 (1936).

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

nitric acid (7697-37-2)

Barbituric acid (67-52-7)

Nitrobarbituric acid,
Barbituric acid, 5-nitro- (480-68-2)

violuric acid (87-39-8)