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.

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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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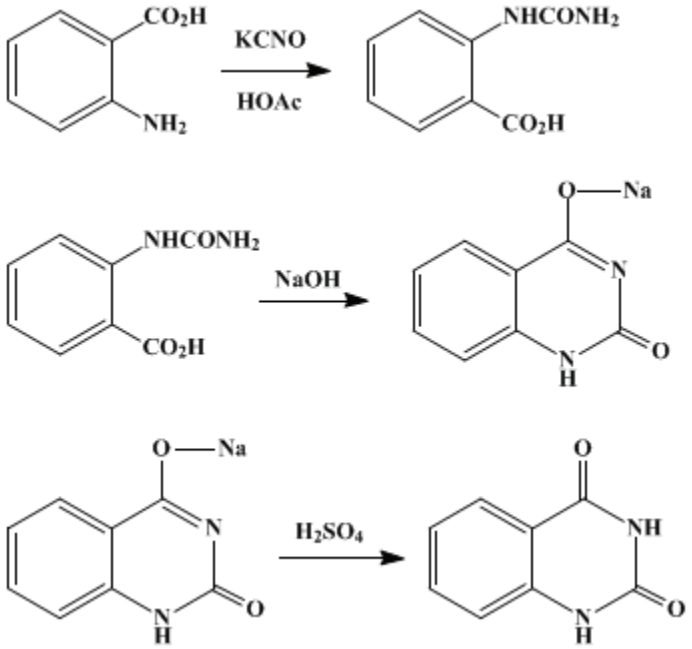
BENZOYLENE UREA

[2,4(1,3)-Quinazolinedione]



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1. Procedure

In a 3-l. beaker a mixture of 20 g. (0.146 mole) of anthranilic acid, 700 cc. of warm water (35°), and 11 cc. (11.6 g., 0.19 mole) of glacial acetic acid is stirred mechanically and allowed to cool to room temperature. A freshly prepared solution of 15 g. (0.185 mole) of potassium cyanate (Note 1) in 50 cc. of water is then added dropwise with stirring over a period of fifteen to twenty minutes (Note 2). The resulting pasty mixture is stirred for twenty minutes, and then 200 g. (5 moles) of flaked sodium hydroxide (Note 3) is added slowly in small portions. During this addition the reaction mixture is kept below 40° by cooling in a cold-water bath. A clear solution is obtained momentarily, but in a short time a fine granular precipitate of the hydrated monosodium salt of benzoylene urea precipitates. After the mixture has cooled overnight in an ice box, the precipitated sodium salt is collected on a Büchner funnel, using a hardened filter paper (Note 4). The colorless salt is dissolved in 1 l. of hot water (90–95°), and the solution is filtered and heated to boiling in a 3-l. beaker. The benzoylene urea is precipitated by adding dilute sulfuric acid (1:1) with vigorous stirring until the liquor is acid to litmus. The product separates as a hydrate which forms small, lustrous, colorless needles. The material is collected on a Büchner funnel, washed with 200 cc. of water, and dried in an oven at 100°. The yield is 19.5–20.5 g. (82–87 per cent of the theoretical amount) (Note 5).

2. Notes

1. The yield is highly dependent upon the quality of the potassium cyanate employed, and some samples were found worthless for the purpose. The yields given were realized using Eastman's regular grade of potassium cyanate.
2. If the addition is too rapid the odor of isocyanic acid (remindful of that of sulfur dioxide) becomes strong and the yield is diminished.
3. The commercial grade of flaked sodium hydroxide dissolves readily and is convenient to handle. Any silica present is not objectionable since it is removed by filtering the redissolved sodium salt before

precipitating the product with acid.
4. Schleicher and Schüll's hardened filters (No. 575, 9 cm.) are satisfactory.
5. The melting point of benzoylene urea is above 350°.

3. Discussion

Benzoylene urea has been prepared by passing cyanogen into a solution of anthranilic acid in alcohol and hydrolyzing the resulting 2-ethoxy-4-ketodihydroquinazoline;[1] by fusing anthranilic acid with urea;[2], [3] and by the action of aqueous cyanic acid on anthranilic acid.[2], [3], [4], [5] The procedure described is adapted from that of Bogert and Scatchard[5] with several modifications.

References and Notes

1. Griess, Ber. **2**, 415 (1869).
2. Griess, J. prakt. Chem. (2) **5**, 371 (1872).
3. Bogert and Scatchard, J. Am. Chem. Soc. **41**, 2056 (1919).
4. Gabriel and Colman, Ber. **38**, 3561 (1905); Scott and Cohen, J. Chem. Soc. **119**, 664 (1921).
5. Bogert and Scatchard, J. Am. Chem. Soc. **38**, 1611 (1916).

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

2,4(1,3)-Quinazolinedione

silica

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

sodium hydroxide (1310-73-2)

sulfur dioxide (7446-09-5)

cyanogen

urea (57-13-6)

Anthranilic Acid (118-92-3)

potassium cyanate (590-28-3)

Benzoylene urea (86-96-4)

isocyanic acid (75-13-8)

2-ethoxy-4-ketodihydroquinazoline

cyanic acid (420-05-3)

monosodium salt of benzoylene urea