



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.

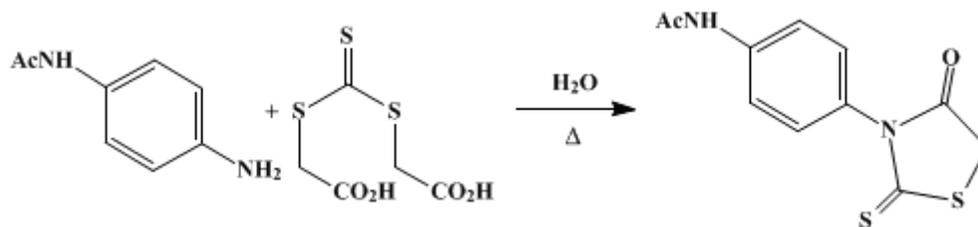
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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N-(*p*-ACETYLAMINOPHENYL)RHODANINE

[Rhodanine, 3-(*p*-acetamidophenyl)-]



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Checked by John D. Roberts and Stanley L. Manatt.

1. Procedure

In a 2-l. round-bottomed flask fitted with a mechanical stirrer and a reflux condenser are placed 30.0 g. (0.20 mole) of *p*-aminoacetanilide (Note 1) and 400 ml. of water. The mixture is heated on a steam bath with stirring, and to the clear solution is added at once a hot solution of 45.2 g. (0.20 mole) of trithiocarbodiglycolic acid (p. 967) in 500 ml. of water. Heating and stirring are continued for 5 hours (Note 2). The steam bath is then replaced by an ice bath, and the reaction mixture is cooled to 20–25°. The precipitate is removed by suction filtration. The solid is transferred to a 500-ml. Erlenmeyer flask containing 200 ml. of water. The mixture is heated on the steam bath to 70–75° while the lumps are crushed by a glass rod to obtain a homogeneous mixture. The mixture is filtered with suction while hot, and the flask is cleaned by rinsing it with small amounts of hot water. The solid on the filter is sucked as dry as possible and then transferred to a 2-l. round-bottomed flask fitted with a reflux condenser. Glacial acetic acid (1.5 l.) is added and the mixture is heated in an oil bath to vigorous reflux for 5 minutes (Note 3). A small amount of solid does not dissolve, and this is removed by filtration while hot (Note 4). The filtrate is stirred mechanically and cooled to 15–20° by an ice bath and kept at this temperature for 1 hour. The slightly yellow crystals are collected by suction filtration, washed successively with 25 ml. of glacial acetic acid, 100 ml. of ethanol, and 100 ml. of ether. The yield of air-dried material is 26–28 g. (49–53% yield). The compound decomposes on heating above 240° (Note 5).

2. Notes

1. *p*-Aminoacetanilide (white label) supplied by Eastman Kodak Company was used.
2. Within 10 minutes a precipitate is formed; the greater part of the reaction product is present after 2 hours' heating.
3. The purification should be carried out in a hood, since gas escapes during the heating and hot acetic acid is irritating to the eyes. The checkers used a 2-l. heating mantle instead of an oil bath.
4. The filtration of the hot acetic acid solution should be done with care. The flask was surrounded by a towel and rubber gloves were worn. The filtration can best be done in two steps. Approximately half of the hot acetic acid solution is filtered through a large, fluted filter paper; the other half is heated again to reflux and then filtered through another fluted filter paper. Filtration through a steam-heated Büchner funnel may sometimes be troublesome, since the suction accelerates crystallization causing plugging of the funnel stem.
5. Analytical values: Calcd. for C₁₁H₁₀N₂O₂S₂: C, 49.62; H, 3.78; N, 10.52; S, 24.08. Found: C, 49.76; H, 3.76; N, 10.36; S, 24.07.

3. Discussion

This procedure is based on the method of Holmberg² for preparing N-substituted rhodanines. The synthesis of N-(*p*-acetylaminophenyl)rhodanine has not yet been reported in the literature.

References and Notes

1. The Upjohn Company, Kalamazoo, Michigan.
 2. Holmberg, *J. prakt. Chem.*, **81**, 451 (1910).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

trithiocarbodiglycolic acid

[ethanol \(64-17-5\)](#)

[acetic acid \(64-19-7\)](#)

[ether \(60-29-7\)](#)

[N-\(p-ACETYLAMINOPHENYL\)RHODANINE,
Rhodanine, 3-\(p-acetamidophenyl\)- \(53663-36-8\)](#)

[p-aminoacetanilide \(122-80-5\)](#)