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.

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m-THIOCRESOL[1]

WARNING

Diazonium xanthates (ArN=NSCSOC2Hs) can detonate, and this procedure should be followed carefully to ensure decomposition of the xanthate as it is formed. Under no circumstances should the diazonium solution and the potassium ethyl xanthate be mixed cold and the mixture subsequently heated. A severe detonation has been reported when such a procedure was employed during the preparation of thiocresol.

It has been observed[2] that the dropwise addition of an aqueous solution of potassium ethyl xanthate to a cold (0°) aqueous solution of diazotized orthanilic acid results in the immediate loss of nitrogen when a trace of nickel ion is present in the stirred diazonium solution.[3] The catalyst can be added as nickelous chloride or simply by using a nichrome wire stirrer. When no nickel ion is added and a glass stirrer is employed, the diazonium xanthate precipitates and requires heat (32°) to effect decomposition.

The use of a nichrome stirrer or a catalytic amount of nickel ion is recommended[1] for such reactions to minimize the accumulation of diazonium xanthate; however, the catalytic role of nickel ion has not been explored with other diazonium salts.

References and Notes

1. *Org. Syntheses*, Coll. Vol. **3**, 809 (1955).
2. William E. Parham and William R. Hasek, unpublished work.
3. William R. Hasek, Ph.D. Thesis, The University of Minnesota, 1953, p. 121.