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.

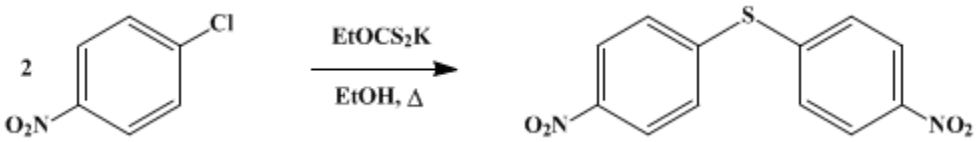
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.028.0082

Organic Syntheses, Coll. Vol. 3, p.667 (1955); Vol. 28, p.82 (1948).

p-NITROPHENYL SULFIDE

[Sulfide, bis-(*p*-nitrophenyl)]



Submitted by Charles C. Price and Gardner W. Stacy.
Checked by Cliff S. Hamilton and Paul D. Berry.

1. Procedure

In a 1-l. round-bottomed flask equipped with a reflux condenser are placed 157.5 g. (1 mole) of *p*-chloronitrobenzene, 160 g. (1 mole) of potassium xanthate (Note 1), and 450 ml. of 95% ethanol. This reaction mixture is heated under reflux on a steam bath for 48 hours. The crystalline product, which deposits from solution during the course of the reaction, is collected by filtration, crushed into small particles in a mortar, and washed twice with hot ethanol and once with hot water. The yield of *p*-nitrophenyl sulfide melting at 158–160° is 105–113 g. (76–82%). This product is pure enough for most purposes. Recrystallization from glacial acetic acid (15 ml. per gram) raises the melting point to 160–161°.

2. Notes

1. Potassium xanthate may be prepared in the following manner: With heating, 300 g. (5.36 moles) of potassium hydroxide is dissolved in 3 l. of absolute ethanol. The solution is then cooled in an ice bath, and the temperature is kept below 10° while carbon disulfide is added in portions with stirring until the solution is no longer alkaline; about 456 g. (360 ml., 5.95 moles) of carbon disulfide is required. The potassium xanthate is collected by suction filtration and air-dried on large sheets of filter paper; yield, 429–472 g. (50–55%).

3. Discussion

p-Nitrophenyl sulfide has been prepared by the reaction between *p*-chloronitrobenzene and sodium sulfide.[1] This is not a practical means of preparation, however, because of the variety of substances formed.[2] The method described has been published.[3] A preparation of pure *p*-nitrophenyl sulfide has been reported from *p*-chloronitrobenzene and sodium thiosulfate.[4]

References and Notes

1. Nietski and Bothof, *Ber.*, **27**, 3261 (1894).
2. Kehrmann and Bauer, *Ber.*, **29**, 2362 (1896).
3. Price and Stacy, *J. Am. Chem. Soc.*, **68**, 498 (1946).
4. Dall'Olio, *Chimica e industria*, **28**, 73 (1946) [*C. A.*, **41**, 380 (1947)].

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

ethanol (64-17-5)

acetic acid (64-19-7)

sodium thiosulfate (7772-98-7)

potassium hydroxide (1310-58-3)

carbon disulfide (75-15-0)

sodium sulfide (1313-82-2)

p-chloronitrobenzene (100-00-5)

potassium xanthate

p-Nitrophenyl sulfide,
Sulfide, bis-(p-nitrophenyl) (1223-31-0)