



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.

TETRAMETHYLBIPHOSPHINE DISULFIDE

Warning: It has been reported^{1,2} that serious explosions have occurred during the preparation of tetramethylbiphosphine disulfide by the method described in Inorganic Syntheses.³ No such incidents have been reported in the synthesis of the compound published in this series,⁴ but the two procedures are sufficiently similar that caution is indicated. The following precautions are strongly urged:

1. The phosphorus trichloride sulfide (PSCl_3) should be distilled before use.
2. The reaction vessel should be cooled with an ice-salt bath (rather than an acetone dry ice bath as specified in the published procedure) during the addition of the PSCl_3 solution to the Grignard reagent. The reaction temperature should be monitored carefully. If it falls below -5° , the addition should be stopped and the reaction mixture cautiously rewarmed to 0 – 5°C before addition is resumed.
3. The reaction apparatus should be shielded throughout the addition of the PSCl solution and the subsequent warming of the reaction mixture.

References and Notes

1. Bercaw, J. E. *Chem. Eng. News* **1984**, 62(18), 4, April 30.
2. Davies, S. G. *Chem. Britain* **1984**, 20(5), 403, May.
3. Butter, S. A.; Chatt, J. *Inorg. Synth.* **1974**, 15, 186.
4. Parshall, G. W. *Org. Synth.*, **1965**, 45, 102; *Org. Synth., Coll. Vol. V* **1973**, 1016.

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