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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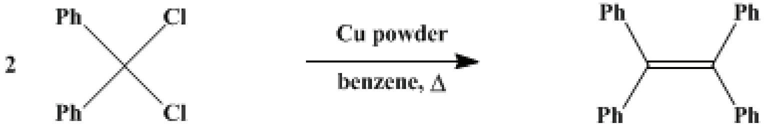
TETRAPHENYLETHYLENE

[Ethylene, tetraphenyl-]



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1. Procedure

A solution of 75 g. (0.32 mole) of diphenyldichloromethane (Note 1) in 250 ml. of anhydrous benzene is placed in a 500-ml. round-bottomed flask fitted with a reflux condenser. To the solution is added 50 g. (0.78 g. atom) of powdered copper (Note 2). The mixture is boiled gently for 3 hours. The hot solution is filtered, and 250 ml. of absolute ethanol is added to the filtrate. On cooling 25–31 g. (47–60%) of light yellow crystals, m.p. 222–224°, are obtained. The mother liquor is concentrated to about 200 ml. by distillation from a 1-l. Claisen flask. Cooling the residue yields 6–12 g. of yellow product. Crystallization of this crude material from a 1:1 by volume mixture (12 ml. for each gram) of absolute ethanol and benzene gives an additional 2.5–10 g. of tetraphenylethylene, m.p. 223–224°. The total yield is 29–37 g. (55–70%).

2. Notes

1. Diphenyldichloromethane is conveniently prepared from benzophenone and phosphorus pentachloride.[2] A product of b.p. 180–181°/17 mm. is obtained in about 90% yield.
2. The checkers used bronze powder obtained from George Benda, Inc., Boonton, New Jersey. Some varieties of copper powder tended to form a dense paste which did not disperse readily and resulted in lower yields.

3. Discussion

This procedure is adapted from the method of Schlenk and Bergmann.[3] Tetraphenylethylene has been prepared by the reaction of diphenylmethane with diphenyldichloromethane;[4] by the reaction of diphenyldichloromethane with silver or zinc;[4] by the reaction of thiobenzophenone with copper;[5] by the reaction of diphenylmethane with sulfur;[6] by the reduction of benzophenone with amalgamated zinc in the presence of hydrochloric acid;[7] by the rearrangement of 1,2,2,2-tetraphenylethanol with acetyl chloride;[8] by the reaction of diphenylmethane with bromine, followed by treatment of the product with sodium iodide in acetone;[9] by the reaction of 1,2-difluoro-1,2-diphenylethylene with phenyllithium;[10] by the reaction of diphenylchloromethane with sodium or potassium amide in liquid ammonia;[11] and by treatment of 2,2,3,3-tetraphenylpropionitrile with potassium amide in liquid ammonia.[12]

References and Notes

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11. Hauser, Brasen, Skell, Kantor, and Brodhag, *J. Am. Chem. Soc.*, **78**, 1653 (1956).
12. Hauser and Brasen, *J. Am. Chem. Soc.*, **78**, 82 (1956).

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

amalgamated zinc

sodium or potassium amide

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

ammonia (7664-41-7)

Benzene (71-43-2)

phosphorus pentachloride (10026-13-8)

acetyl chloride (75-36-5)

bromine (7726-95-6)

sulfur (7704-34-9)

copper,
copper powder (7440-50-8)

acetone (67-64-1)

Benzophenone (119-61-9)

zinc (7440-66-6)

diphenyldichloromethane (2051-90-3)

Diphenylmethane (101-81-5)

sodium iodide (7681-82-5)

Phenyllithium (591-51-5)

silver (7440-22-4)

Thiobenzophenone (1450-31-3)

potassium amide

Tetraphenylethylene,
Ethylene, tetraphenyl- (632-51-9)

1,2,2,2-tetraphenylethanol

1,2-difluoro-1,2-diphenylethylene

diphenylchloromethane (90-99-3)

2,2,3,3-tetraphenylpropionitrile