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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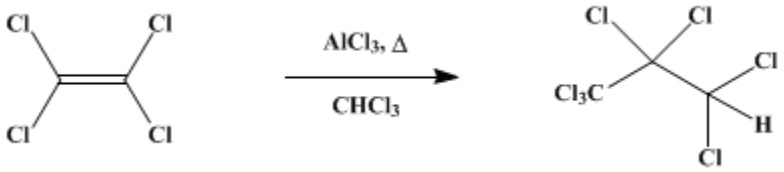
unsym.-HEPTACHLOROPROPANE

[Propane, 1,1,1,2,2,3,3-heptachloro-]



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1. Procedure

In a 1-l. round-bottomed flask, equipped with a reflux condenser carrying a calcium chloride tube, are placed 166 g. (103 cc., 1 mole) of technical tetrachloroethylene, 300 g. (200 cc., 2.5 moles) of dry chloroform, and 27 g. (0.2 mole) of anhydrous aluminum chloride. The mixture is refluxed gently on the steam bath for fifteen hours (Note 1), cooled to room temperature, and poured into a 1-l. separatory funnel half filled with crushed ice. The organic layer is washed several times with water and dried over calcium chloride or soluble anhydrite. By fractionation at atmospheric pressure through an efficient column, 160–165 g. of chloroform is recovered. Distillation of the *unsym*.-heptachloropropane fraction at diminished pressure gives material boiling at 110–113°/10 mm., or 137–140°/32 mm., and melting at 29–30°. The yield is 250–266 g. (88–93 per cent of the theoretical amount) (Note 2).

2. Notes

1. A small amount of hydrogen chloride is evolved in the initial stages of the reaction.
2. According to Prins[1] the heptachloropropane can be isolated easily by pouring the reaction mixture into water and removing the unreacted materials by steam distillation. The process is stopped when the product begins to distil, and on cooling the residue is obtained as a colorless solid of the correct melting point.

3. Discussion

The method is essentially that discovered by Böeseken and Prins[2] and studied further by Prins.[1], [3] Pentachloroethane can be used in place of tetrachloroethylene, as it is converted into the unsaturated compound in the presence of aluminum chloride. *unsym*.-Heptachloropropane has been obtained also by the action of phosphorus pentachloride on pentachloroacetone,[4] and by treating dichloroacetyl chloride with aluminum chloride.[5]

References and Notes

1. Prins, Rec. trav. chim. **54**, 249 (1935).
2. Böeseken and Prins, Chem. Zentr. **1911**, I, 466.
3. Prins, J. prakt. Chem. (2) **89**, 414 (1914).
4. Fritsch, Ann. **297**, 312 (1897).
5. Böeseken, Rec. trav. chim. **29**, 109 (1910).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

unsym.-HEPTACHLOROPROPANE

calcium chloride (10043-52-4)

hydrogen chloride (7647-01-0)

phosphorus pentachloride (10026-13-8)

chloroform (67-66-3)

aluminum chloride (3495-54-3)

pentachloroethane (76-01-7)

HEPTACHLOROPROPANE,
Propane, 1,1,1,2,2,3,3-heptachloro- (594-89-8)

tetrachloroethylene (127-18-4)

pentachloroacetone (1768-31-6)

dichloroacetyl chloride (79-36-7)