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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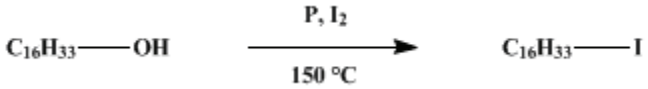
n-HEXADECYL IODIDE

[Hexadecane, 1-iodo-]



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1. Procedure

Two hundred forty-two grams (1 mole) of cetyl alcohol (Note 1), 10 g. (0.32 gram atom) of red phosphorus, and 134 g. (1.06 gram atoms) of resublimed iodine are placed in a 3-l. round-bottomed flask and heated in an oil bath until the alcohol has melted. The flask is then fitted with a reflux condenser and a liquid-sealed mechanical stirrer. With stirring, the mixture is heated at 145–150° (temperature of the oil bath) for five hours. When the reaction mixture has cooled, the cetyl iodide is removed by extracting once with a 250-cc. portion and twice with 125-cc. portions of commercial ether. The combined ether extracts are filtered free of phosphorus and washed with 500 cc. of cold water, 250 cc. of 5 per cent sodium hydroxide solution, and again with 500 cc. of water. The ether solution is dried over anhydrous calcium chloride. After removal of the ether by distilling on a steam bath, the iodide is distilled under reduced pressure. The main fraction, distilling at 220–225°/22 mm. (210– 215°/12 mm.), weighs 300 g. (85 per cent of the theoretical amount) and melts at 18–20° (Note 2). Redistillation gives 275 g. (78 per cent of the theoretical amount) boiling at 220–223°/22 mm. (203–205°/9 mm.) and melting at 20–22° (Note 3).

2. Notes

1. Cetyl alcohol prepared according to the directions given on p. 374 and melting at 48–49° is satisfactory. If a poorer grade of cetyl alcohol is used, the yield may be reduced to 70 per cent.
2. This material is probably pure enough for most work. Melting points as high as 25° are recorded in the literature.
3. Traces of iodine come over when the distillation starts and the fore-run is therefore strongly colored. When distillation is started again after being interrupted traces of iodine again appear in the first few drops of the main distillate. A more nearly colorless distillate is obtained if the fractions are cut without interrupting the distillation.

3. Discussion

The method described is essentially that of Smith.[1] Several other workers have used a similar method.[2] Cetyl iodide has also been prepared by heating cetyl alcohol with yellow phosphorus and iodine in carbon disulfide solution;[3] by repeatedly passing dry hydrogen iodide into the molten alcohol and permitting the reaction mass to stand between additions;[4] and by heating cetyl alcohol or cetyl stearate with 55 per cent hydriodic acid to a temperature of 120° during two hours.[5]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 320
- Org. Syn. Coll. Vol. 6, 830

References and Notes

1. Smith, J. Chem. Soc. **1932**, 738.
2. Fridau, Ann. **83**, 9 (1852); Levene, West, and van der Scheer, J. Biol. Chem. **20**, 523 (1915); Delcourt, Bull. soc. chim. Belg. **40**, 284 (1931) [C. A. **25**, 5661 (1931)].
3. Gascard, Ann. chim. (9) **15**, 372 (1921).
4. Krafft, Ber. **19**, 2219 (1886).
5. Guyer, Bieler, and Hardmeier, Helv. Chim. Acta **20**, 1466 (1937).

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

red phosphorus

calcium chloride (10043-52-4)

ether (60-29-7)

sodium hydroxide (1310-73-2)

PHOSPHORUS (7723-14-0)

iodine (7553-56-2)

carbon disulfide (75-15-0)

hydriodic acid,
hydrogen iodide (10034-85-2)

cetyl iodide,
Hexadecane, 1-iodo-,
n-HEXADECYL IODIDE (544-77-4)

Cetyl alcohol (36653-82-4)

cetyl stearate (1190-63-2)