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.

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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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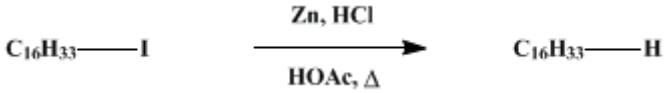
n-HEXADECANE



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1. Procedure

In a 2-l. round-bottomed flask, fitted with a liquid-sealed mechanical stirrer, a gas inlet tube, and a tube to carry off hydrogen chloride and acetic acid vapors (Note 1), are placed 915 cc. of glacial acetic acid, 327 g. (5 gram atoms) of zinc dust, and 352 g. (1 mole) of cetyl iodide (m.p. 20–22°) (p. 322). The mixture is saturated with dry hydrogen chloride and then stirred and heated on a steam bath. At the end of every five hours of heating, the mixture is again saturated with hydrogen chloride. After the reaction has proceeded for twenty-five hours, the mixture is allowed to cool, and the layer of hexadecane, which rises to the top of the reaction mixture, is separated. The residue is poured into 3 l. of water and filtered on a Büchner funnel to remove the zinc dust. The zinc dust is washed with 500 cc. of water and then with 250 cc. of ether. The combined water layers are extracted with two 500-cc. portions of ether. The ether extracts are combined and added to the hexadecane, and the resulting solution is washed with two 250-cc. portions of 20 per cent sodium hydroxide and then with water until free of alkali. The ether solution is dried with 150 g. of anhydrous sodium sulfate, filtered, and distilled from a 500-cc. modified Claisen flask with a fractionating side arm. The yield of *n*-hexadecane boiling at 156–158°/14 mm. and melting at 16–17° is 192 g. (85 per cent of the theoretical amount).

2. Notes

1. If the reaction is run in a hood, an open flask may be used.

3. Discussion

n-Hexadecane has been prepared by the reduction of cetyl iodide with zinc and hydrochloric acid in alcohol[1] or acetic acid,[2], [3] with the zinc-copper couple,[3] and with hydrogen and a palladium catalyst.[3] The hydrocarbon has also been prepared by treating octyl iodide with sodium;[4] by heating mercury dioctyl alone or with zinc dust;[5] by heating palmitic acid with hydriodic acid and red phosphorus;[6] and by reducing 1-hexadecene.[7]

n-Hexadecane has been obtained as a by-product from the preparation of octylmagnesium bromide[8] and from the action of sodium on a mixture of octyl bromide and ethyl bromide,[9] and it is one of the products formed on heating sodium stearate[10] or cetyl ether.[11]

References and Notes

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

red phosphorus

zinc-copper couple

mercury dioctyl

hydrogen chloride,
hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ether (60-29-7)

hydrogen (1333-74-0)

sodium hydroxide (1310-73-2)

Ethyl bromide (74-96-4)

Octyl bromide (111-83-1)

sodium sulfate (7757-82-6)

zinc (7440-66-6)

sodium (13966-32-0)

palladium (7440-05-3)

hydriodic acid (10034-85-2)

cetyl iodide (544-77-4)

Hexadecane,
n-HEXADECANE (544-76-3)

octyl iodide (629-27-6)

palmitic acid (57-10-3)

1-hexadecene (629-73-2)

octylmagnesium bromide (17049-49-9)

sodium stearate (822-16-2)

cetyl ether (4113-12-6)