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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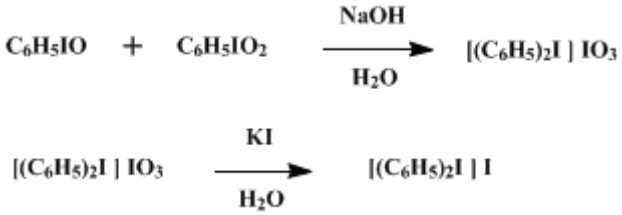
Organic Syntheses, Coll. Vol. 3, p.355 (1955); Vol. 22, p.52 (1942).

DIPHENYLIODONIUM IODIDE

[Iodonium compounds, diphenyl—iodide]



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1. Procedure

A mixture of 22 g. (0.1 mole) of iodosobenzene (p. 483), 24 g. (0.1 mole) of iodoxybenzene (p. 485), and 200 ml. of 1 *N* sodium hydroxide (Note 1) is gently stirred for 24 hours. The resulting brown slurry is thoroughly stirred with 1 l. of cold water, and, after the mixture settles, the supernatant solution of diphenyliodonium iodate is decanted through a filter. The solid residue is extracted twice with 500-ml. portions of water, and the extracts are separated, by decantation through a filter, from the small amount of tarry residue. An aqueous solution of 20 g. (0.12 mole) of potassium iodide is added to the combined filtrates. After the bulky white precipitate of diphenyliodonium iodide has stood for an hour or two, with occasional shaking, it is filtered with suction, washed with water, and dried on porous tile at room temperature. The product weighs 29–30 g. (70–72%) and melts at 172–175° with vigorous decomposition.

2. Notes

1. Since the reaction is catalyzed by hydroxide ions, the amount of base may be varied within wide limits. It is advantageous to grind the solid reactants with 50 ml. of water before the alkali is added.

3. Discussion

Diphenyliodonium iodide is obtained when sulfur dioxide or aqueous potassium iodide is added to a solution containing diphenyliodonium iodate.[1,2] Such solutions have been prepared by the action of moist silver oxide, or of aqueous sodium or potassium hydroxide, on an equimolar mixture of iodosobenzene and iodoxybenzene;[1,2] by shaking iodosobenzene with moist silver oxide;[3] by shaking iodoxybenzene with sodium hydroxide;[2] and by steam distillation of a mixture of iodosobenzene and iodoxybenzene.[2] Diphenyliodonium iodide has been prepared by heating iodoxybenzene with aqueous potassium iodide and barium hydroxide.[4]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 483
- Org. Syn. Coll. Vol. 3, 485
- Org. Syn. Coll. Vol. 6, 928

References and Notes

1. Hartmann and Meyer, *Ber.*, **27**, 504, 506 (1894).
2. Hartmann and Meyer, *Ber.*, **27**, 1598 (1894).
3. Hartmann and Meyer, *Ber.*, **27**, 503 (1894).

4. Willgerodt, *Ber.*, **29**, 2009 (1896).

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

sodium or potassium hydroxide

sodium hydroxide (1310-73-2)

silver oxide (20667-12-3)

sulfur dioxide (7446-09-5)

potassium iodide (7681-11-0)

barium hydroxide (17194-00-2)

DIPHENYLIODONIUM IODIDE (2217-79-0)

Iodosobenzene (536-80-1)

Iodoxybenzene (696-33-3)

diphenyliodonium iodate