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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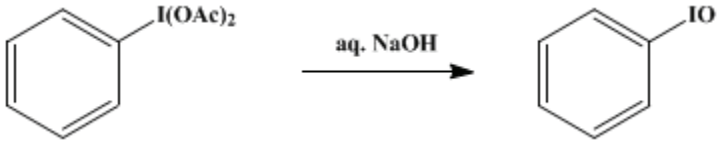
Organic Syntheses, Coll. Vol. 5, p.658 (1973); Vol. 43, p.60 (1963).

IODOSOBENZENE

[Benzene, iodoso-]



Submitted by H. Saltzman and J. G. Sharefkin[1].
Checked by Melvin S. Newman and Narinder Gill.

1. Procedure

Caution! This compound explodes if heated to 210°.

Finely ground iodosobenzene diacetate[2] (32.2 g., 0.10 mole) is placed in a 250-ml. beaker, and 150 ml. of 3*N* sodium hydroxide is added over a 5-minute period with vigorous stirring. The lumps of solid that form are triturated with a stirring rod or spatula for 15 minutes, and the reaction mixture stands for an additional 45 minutes to complete the reaction. One hundred milliliters of water is added, the mixture is stirred vigorously, and the crude, solid iodosobenzene is collected on a Büchner funnel. The wet solid is returned to the beaker and triturated in 200 ml. of water. The solid is again collected on the Büchner funnel, washed there with 200 ml. of water, and dried by maintaining suction. Final purification is effected by triturating the dried solid in 75 ml. of chloroform in a beaker. The iodosobenzene is separated by filtration (Note 1) and air-dried; weight 18.7–20.5 g. (85–93%); m.p. 210° *(Caution! Explodes!)*. Iodometric titration[3] shows the product to be more than 99% pure (Note 2).

2. Notes

1. The filtrate yields unreacted diacetate on evaporation.
2. The purity of the iodosobenzene depends on the purity of the diacetate used.

3. Discussion

Iodosobenzene has been prepared by the action of sodium or potassium hydroxide solution on iodobenzene dichloride[3,4] and by addition of water to the dichloride.[5]

4. Merits of the Preparation

This method of preparing iodosobenzene is preferable to older ones based on iodosobenzene dichloride because iodosobenzene diacetate[2] is more stable and more conveniently prepared than the dichloride[3] and the overall yield is greater (75% versus 54%).

The procedure seems to be a general way of preparing iodosoarenes with electron-donating substituents, for the submitters have used it to obtain good yields of *o*-, *m*- and *p*-iodosotoluene, 2- and 4-iodoso-*m*-xylene, 2-iodoso-*p*-xylene, *o*-iodosophenetole, and 4-iodosobiphenyl.

Iodosoarenes are useful in the preparation of iodonium salts, $Ar_2I^+X^-$.[6]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 5, 660

References and Notes

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2. J. G. Sharefkin and H. Slatzman, this volume, p. 660.
3. H. J. Lucas, E. R. Kennedy, and M. W. Formo, *Org. Syntheses*, Coll. Vol. **3**, 483 (1955).
4. C. Willgerodt, *Ber.*, **25**, 3494 (1892); **26**, 357, 1802 (1893); P. Askenasy and V. Meyer, *Ber.*, **26**, 1354 (1893); C. Hartmann and V. Meyer, *Ber.*, **27**, 502 (1894).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

o-, m- and p-iodosotoluene

2- and 4-iodoso-m-xylene

sodium hydroxide (1310-73-2)

chloroform (67-66-3)

potassium hydroxide (1310-58-3)

sodium (13966-32-0)

iodobenzene dichloride (2401-21-0)

Iodosobenzene,
Benzene, iodoso- (536-80-1)

Iodosobenzene diacetate (3240-34-4)

iodosobenzene dichloride

4-iodosobiphenyl

2-iodoso-p-xylene

o-iodosophenetole