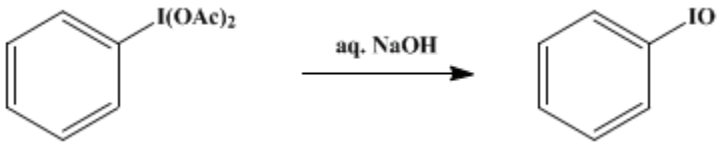
Organic Syntheses, Coll. Vol. 5, p.658 (1973); Vol. 43, p.60 (1963).

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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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

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