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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o-IODOPHENOL

[Phenol, *o*-iodo-]

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1. Procedure

A 2-l. round-bottomed, short-necked flask is equipped with a glass mechanical stirrer. A suspension of 165 g. (0.5 mole) of *o*-chloromercuriphenol (p. 161) (m.p. 147° or higher) in 500 cc. of chloroform is placed in the flask and treated with 127 g. (0.5 mole) of iodine, the stirring being continued until a small sample on filtration shows no unreacted iodine. This requires from one to two hours. The solid inorganic mercury compounds are removed by suction filtration. The filtrate is distilled from a 1-l. distilling flask on the water bath to remove most of the chloroform, which is saved for later runs (Note 1). The residual liquid is shaken vigorously with a solution of 5 g. of potassium iodide in 10 cc. of water, to remove dissolved mercuric iodide. The heavier layer, which still possesses a reddish color, is transferred without drying to a 200-cc. Claisen flask and distilled under reduced pressure. At first a small amount of chloroform and water distils over, after which the higher-boiling material is collected in a separate receiver. Small amounts of inorganic mercury compounds remain in the flask (Note 2).

The *o*-iodophenol solidifies upon cooling. The yield of material melting at 32–34° is 70 g. (63 per cent of the theoretical amount). The product obtained in this way is slightly yellow. If a purer product is desired, it may be redistilled under reduced pressure. Most of the crude material will distil over a 5° range (b.p. 130°/18 mm. and 186°/160 mm., m.p. 43°).

2. Notes

1. About 460 cc. of chloroform can be recovered, the time required being one hour.
2. The first distillation of the iodophenol should be carried out under distinctly reduced pressure, 40 mm. or lower. The redistillation may be conducted at a higher pressure if desired.
3. Since the iodophenol has a most persistent odor, care should be exercised in working with it.

3. Discussion

o-Iodophenol can be prepared by diazotization from *o*-aminophenol[1] and from *o*-iodoaniline;[2] by heating *o*-iodosalicylic acid;[3] by treating a dilute phenol solution with iodine and iodic acid;[4] from an alcoholic solution of phenol and iodine in the presence of mercuric oxide;[5] from dry sodium phenoxide and iodine in carbon disulfide;[6] and by treating *o*-chloromercuriphenol with iodine in aqueous potassium iodide.[7]

References and Notes

1. Gnehm, Ber. **8**, 820 (1875); Neumann, Ann. **241**, 68 (1887).
2. Holleman and Rinkes, Koninglijke Akad. Wetenschappen Amsterdam **19**, 67 (1910) [Chem. Zentr. II, 304 (1910)].
3. Lautemann, Ann. **120**, 315 (1861).
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7. Chi, Trans. Sci. Soc. China **7**, 169 (1932) [C. A. **26**, 5552 (1932)].

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

mercury compounds

chloroform (67-66-3)

phenol (108-95-2)

potassium iodide (7681-11-0)

o-aminophenol (95-55-6)

mercury (7439-97-6)

mercuric oxide (21908-53-2)

iodine (7553-56-2)

carbon disulfide (75-15-0)

mercuric iodide (7774-29-0)

iodic acid (7782-68-5)

iodophenol,
o-Iodophenol,
Phenol, o-iodo- (533-58-4)

sodium phenoxide

o-iodosalicylic acid

o-Chloromercuriphenol

o-iodoaniline (615-43-0)