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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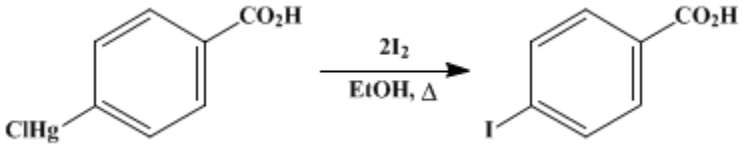
*Organic Syntheses, Coll. Vol. 1, p.325 (1941); Vol. 7, p.58 (1927).*

# *p*-IODOBENZOIC ACID

## [Benzoic acid, *p*-iodo-]

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## 1. Procedure

One hundred fifty grams (0.59 mole) of iodine is dissolved in 2.5 l. of 95 per cent ethyl alcohol in a 5-l. round-bottomed flask set on a tripod and provided with a mechanical stirrer and a reflux condenser. To this solution is added 300 g. (0.84 mole) of powdered dry crude *p*-chloromercuribenzoic acid (p. 159) (Note 1). The mixture is stirred and heated. The acid gradually goes into solution and the color of iodine disappears. The hot stirred mixture is treated with iodine until a yellow color persists for at least ten minutes. This is conveniently done by weighing out iodine in 10-g. portions, dissolving in a little alcohol, and adding through the condenser. The total amount of iodine required depends on the purity of the mercurated acid. It should not exceed 210 g. (0.83 mole)If any insoluble material is left after an excess of iodine has been added, it is removed by rapid filtration through a preheated suction filter.

On cooling, the filtrate yields about 175 g. of *p*-iodobenzoic acid. Concentration of the mother liquor yields crystals which are contaminated with mercuric iodide. The latter may be removed by grinding the crystals in a mortar with water and enough sodium iodide or potassium iodide to destroy the red color of the mercuric iodide (Note 2). The mixture is filtered and the crystals are washed with a little sodium iodide solution and then with water. The washed *p*-iodobenzoic acid melts at 266–267°. The total yield is 150–170 g. (72–81 per cent of the theoretical amount).

## 2. Notes

1. The crude *p*-chloromercuribenzoic acid (p. 159) may be sucked as dry as possible and transferred in this pasty form to the iodine solution. If, however, it has caked at all, it must be thoroughly dried and powdered. A respirator should be worn, as the dust is rather irritating.
2. Another method of removing most of the mercuric iodide is to grind the crystals, suspend them in a little water, and pour off the lighter suspension of iodobenzoic acid, leaving the heavy mercuric iodide behind. Washing with iodide solution is necessary to remove the last of the mercuric iodide.

## 3. Discussion

*p*-Iodobenzoic acid can be prepared by the oxidation of *p*-iodotoluene with chromic acid mixture[1] and with nitric acid;[2] by hydrolysis of the nitrile obtained from *p*-iodonitrobenzene and potassium cyanide;[3] and from *p*-aminobenzoic acid by diazotization.[4]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 159

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## References and Notes

**1.** Körner, Z. Chem. 327 (1868).

2. Cohen and Raper, J. Chem. Soc. **85**, 1273 (1904).
3. Richter, Ber. **4**, 553 (1871).
4. Marshall, Ber. **28**, 338 (1895).

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

ethyl alcohol (64-17-5)

nitric acid (7697-37-2)

potassium iodide (7681-11-0)

potassium cyanide (151-50-8)

iodine (7553-56-2)

chromic acid (7738-94-5)

mercuric iodide (7774-29-0)

sodium iodide (7681-82-5)

iodobenzoic acid (88-67-5)

p-iodotoluene (624-31-7)

p-Chloromercuribenzoic acid (59-85-8)

p-iodobenzoic acid,
Benzoic acid, p-iodo- (619-58-9)

p-iodonitrobenzene (636-98-6)

p-aminobenzoic acid (150-13-0)