



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.

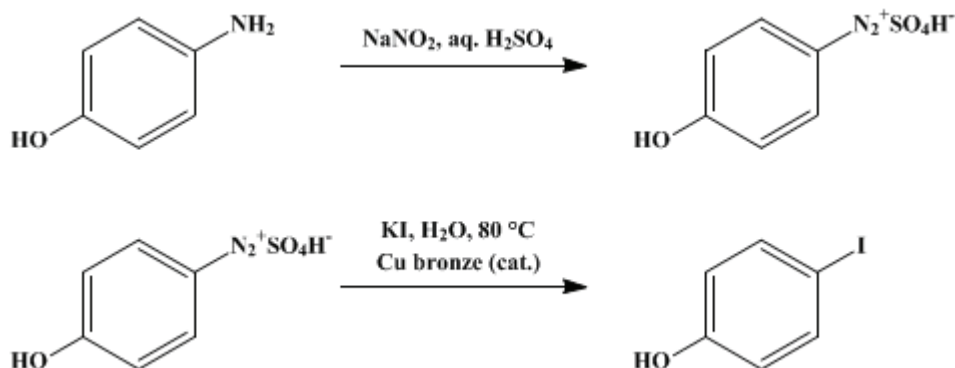
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.355 (1943); Vol. 15, p.39 (1935).

p-IODOPHENOL

[Phenol, *p*-iodo-]



Submitted by F. B. Dains and Floyd Eberly.
Checked by Reynold C. Fuson and H. H. Hully.

1. Procedure

One hundred nine grams (1 mole) of *p*-aminophenol (Note 1) is dissolved in a mixture of 500 g. of ice, 500 cc. of water, and 65 cc. (120 g., 1.2 moles) of concentrated sulfuric acid (sp. gr. 1.84). To this solution, kept in a freezing mixture at 0°, is added, during the course of an hour with constant mechanical stirring, a solution of 72 g. (1 mole) of 95 per cent sodium nitrite in 150 cc. of water. The stirring is continued twenty minutes longer, and then 20 cc. (37 g., 0.37 mole) of concentrated sulfuric acid is added.

This solution is poured into an ice-cold solution of 200 g. (1.2 moles) of potassium iodide in 200 cc. of water. After a few minutes, 1 g. of copper bronze (Note 2) is added, with continued stirring, and the solution is warmed slowly on the water bath. The temperature is kept at 75–80° until the evolution of nitrogen ceases; during this process the iodophenol separates as a heavy dark oil. After cooling to room temperature the reaction mixture is extracted three times with 165-cc. portions of chloroform and the combined extracts are washed with dilute thiosulfate solution. The solvent is removed on the water bath and the residue distilled under reduced pressure, the *p*-iodophenol coming over at 138–140°/5 mm. One crystallization from about 2 l. of ligroin (b.p. 90–110°) gives a colorless product melting sharply at 94°. The yield of recrystallized product is 153–159 g. (69–72 per cent of the theoretical amount).

2. Notes

1. The *p*-aminophenol used was a commercial product melting at 182–183° with decomposition.
2. Some commercial bronzes used for bronze paints are coated with a film of stearic acid. For chemical work an untreated pure copper bronze should be used.

3. Discussion

p-Iodophenol was first obtained as a by-product of the action of iodine on salicylic acid in alkaline solution or by heating iodosalicylic acid.¹ It has also been obtained by the action of iodine on phenol in alkaline solution² or in the presence of mercuric oxide,³ or by the action of iodine monochloride.⁴ It is best prepared by the diazotization of *p*-aminophenol and replacement of the diazonium group by iodine⁵ although it has also been obtained from *p*-iodoaniline by diazotization and replacement of the diazonium group by hydroxyl.⁶

References and Notes

1. Lautemann, Ann. **120**, 299 (1861); Kekulé, *ibid.* **131**, 221 (1864).
 2. Holleman and Rinkes, Rec. trav. chim. **30**, 96 (1911).
 3. Hlasiwetz and Weselsky, Ber. **2**, 523 (1869).
 4. Schützenberger and Sengenwald, Jahresb. **1862**, 413.
 5. Nölting and Wrzesinski, Ber. **8**, 820 (1875); Nölting and Stricker, *ibid.* **20**, 3021 (1887); Neumann, Ann. **241**, 74 (1887).
 6. Griess, Zeit. für Chem. **1865**, 427; Holleman and Rinkes, Rec. trav. chim. **30**, 95 (1911).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

ligroin

copper bronze

sulfuric acid (7664-93-9)

chloroform (67-66-3)

phenol (108-95-2)

potassium iodide (7681-11-0)

nitrogen (7727-37-9)

sodium nitrite (7632-00-0)

salicylic acid

mercuric oxide (21908-53-2)

iodine (7553-56-2)

iodophenol (533-58-4)

iodine monochloride (7790-99-0)

stearic acid (57-11-4)

iodosalicylic acid

p-aminophenol (123-30-8)

p-IODOANILINE (540-37-4)

p-IODOPHENOL,
Phenol, p-iodo- (540-38-5)

