



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.

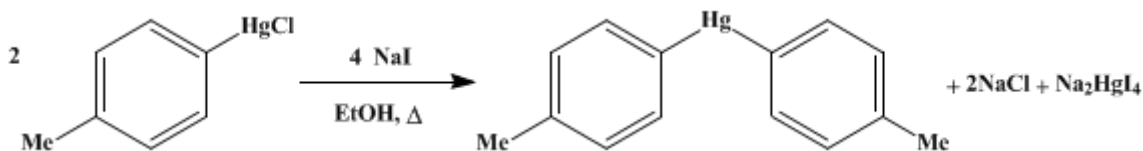
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.

Organic Syntheses, Coll. Vol. 1, p.231 (1941); Vol. 3, p.65 (1923).

DI-*p*-TOLYLMERCURY

[Mercury, di-*p*-tolyl-]



Submitted by F. C. Whitmore, N. Thurman, and Frances H. Hamilton.
Checked by J. B. Conant and F. C. Whidden.

1. Procedure

In a 2-l. round-bottomed flask fitted with a reflux condenser and an efficient glass stirrer, are placed 700 cc. of 95 per cent ethyl alcohol, 100 g. (0.3 mole) of recrystallized *p*-tolylmercuric chloride (p. 519), and 160 g. (1.1 moles) of sodium iodide (Note 1) (75 per cent excess). The mixture is boiled with vigorous stirring (Note 2) for fifteen hours; the solid does not entirely enter into solution at any time. The mixture is then thoroughly cooled and filtered by suction and the solid washed with water until free of inorganic halide. It is then dried in an air oven at 40° and recrystallized from 500 cc. of boiling xylene (Note 3). In this way 45–50 g. (78–85 per cent of the theoretical amount) of a product melting at 238° is obtained.

2. Notes

- Mercury compounds of the type $RHgX$ can be changed to those of the type R_2Hg , by almost any neutral or alkaline reagent which tends to change mercuric ions to metallic mercury or to a stable complex ion. Some of these reagents are inorganic iodides, thiocyanates, thiosulfates, sulfides, hydrosulfites, ferrous hydroxide, sodium amalgam, metallic sodium, and sodium stannite. Preliminary experiments indicated that potassium thiocyanate was a good reagent for preparing di-*p*-tolylmercury; but it was later found that the use of sodium iodide with vigorous stirring gave even better results.
- Stirring decreases the time required for the reaction and increases the yield. Without stirring it is practically impossible to obtain a product which is free from halogen.
- Since the di-*p*-tolylmercury has practically the same solubility in xylene as the tolylmercuric iodide and chloride, the latter will be present in the recrystallized product if the reaction has not gone to completion. A trace of unchanged product may readily be detected by the metallic sodium test for inorganic halides.

3. Discussion

Di-*p*-tolylmercury can be prepared from *p*-bromotoluene with sodium amalgam;¹ from *p*-tolylmercuric chloride with copper in pyridine,² or with hydrazine hydrate and sodium carbonate;³ from *p*-tolyllithium and mercuric chloride;⁴ and from the double salt of *p*-toluene-diazonium chloride and mercuric chloride with copper powder in cold acetone.⁵

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 2*, 381

References and Notes

- Dreher and Otto, *Ann.* **154**, 171 (1870); Wurtz, *Compt. rend.* **68**, 1298 (1869); La Coste and Michaelis, *Ann.* **201**, 246 (1880).

2. Hein and Wagler, Ber. **58**, 1499 (1925).
 3. Gilman and Barnett, Rec. trav. chim. **55**, 565 (1936).
 4. Austin, J. Am. Chem. Soc. **54**, 3726 (1932).
 5. Nesmejanov and Kahn, Ber. **62**, 1018 (1929).
-

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

mercuric ions

metallic mercury

inorganic iodides

thiocyanates

thiosulfates

sulfides

hydrosulfites

tolylmercuric iodide and chloride

double salt of p-toluene-diazonium chloride

ethyl alcohol (64-17-5)

sodium carbonate (497-19-8)

copper,
copper powder (7440-50-8)

acetone (67-64-1)

pyridine (110-86-1)

sodium,
metallic sodium (13966-32-0)

mercuric chloride (7487-94-7)

p-tolylmercuric chloride (539-43-5)

xylene (106-42-3)

sodium stannite

sodium iodide (7681-82-5)

ferrous hydroxide

potassium thiocyanate (333-20-0)

hydrazine hydrate (7803-57-8)

p-Bromotoluene (106-38-7)

DI-p-TOLYLMERCURY,
Mercury, di-p-tolyl- (537-64-4)

p-tolyl lithium