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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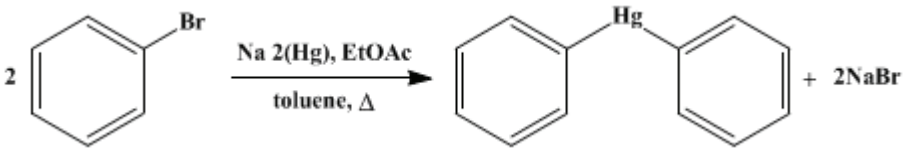
DIPHENYLMERCURY

[Mercury, diphenyl-]



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1. Procedure

In a 1-l. round-bottomed flask fitted with a reflux condenser are placed 900 g. of 3 per cent sodium amalgam (Note 1), 180 g. (1.15 moles) of bromobenzene, 200 cc. of dry toluene or dry xylene, and 10 cc. of ethyl acetate. The mixture is refluxed with frequent shaking for twelve hours in an oil bath at 130°.

The mixture is transferred (Hood) (Note 2) while still hot to a fluted filter paper in a 20-cm. glass funnel, leaving behind as much mercury as possible (Note 3). The funnel is made part of a special extraction apparatus as described on p. 375. The diphenylmercury is extracted with 600 cc. of boiling benzene for about ten hours (Note 4).

The solution is distilled under reduced pressure on an oil bath, the temperature of which is raised to 110° near the end of the distillation. The solid residue left in the flask after removal of the solvent is taken out and washed until nearly white with 95 per cent alcohol which has been cooled in an ice bath. This requires about four washings of 50 cc. of alcohol each (Note 5). The yield is 65–75 g. (32–37 per cent of the theoretical amount). The melting point is 121–123°.

2. Notes

1. For the preparation of sodium amalgam see Note 1 on p. 554.
2. Diphenylmercury is very poisonous. The vapors of the benzene solution must not be breathed.
3. Care must be taken to remove all the diphenylmercury; but if too much mercury is removed, it may break the filter paper or weight it down so that the benzene vapors cannot rise around it. It is sometimes best to put stirring rods down around the sides of the paper to insure a free path for the benzene vapors.
4. In some cases the extraction may not be complete in this length of time.
5. Some diphenylmercury may be recovered by combining the washings from several runs.

3. Discussion

Diphenylmercury can be prepared by the action of sodium on a mixture of bromobenzene and mercuric chloride;[1] from sodium amalgam and phenylmercuric iodide[2] or bromobenzene;[3] from phenylmercuric bromide and potassium sulfide;[2] from phenylmercuric acetate and sodium stannite;[4] from the double salt of benzenediazonium chloride and mercuric chloride by the action of powdered copper;[5] and from phenylmagnesium bromide and mercuric chloride[6] or bromide.[7]

References and Notes

1. Michaelis and Reese, Ber. **15**, 2877 (1882).
2. Dreher and Otto, Ber. **2**, 542 (1869).
3. Wurtz, Compt. rend **68**, 1300 (1869); Calvery, J. Am. Chem. Soc. **48**, 1009 (1926).
4. Dimroth, Ber. **35**, 2853 (1902); Maynard, J. Am. Chem. Soc. **46**, 1510 (1924).

5. Nesmejanov and Kahn, Ber. **62**, 1018 (1929).
6. Pfeiffer and Truskier, Ber. **37**, 1127 (1904); Steinkopf, Bielenberg and Augestad-Jensen, Ann. **430**, 41 (1923); Borgstrom and Dewar, J. Am. Chem. Soc. **51**, 3387 (1929); Blicke and Smith, ibid. **51**, 3479 (1929).
7. Bachmann, ibid. **55**, 2830 (1933).

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

double salt of benzenediazonium chloride

powdered copper

alcohol (64-17-5)

Benzene (71-43-2)

ethyl acetate (141-78-6)

bromide (24959-67-9)

mercury (7439-97-6)

toluene (108-88-3)

sodium (13966-32-0)

bromobenzene (108-86-1)

mercuric chloride (7487-94-7)

xylene (106-42-3)

Phenylmagnesium bromide (100-58-3)

Diphenylmercury,
Mercury, diphenyl- (587-85-9)

phenylmercuric iodide

phenyl-mercuric bromide

potassium sulfide (1312-73-8)

phenylmercuric acetate

sodium stannite

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