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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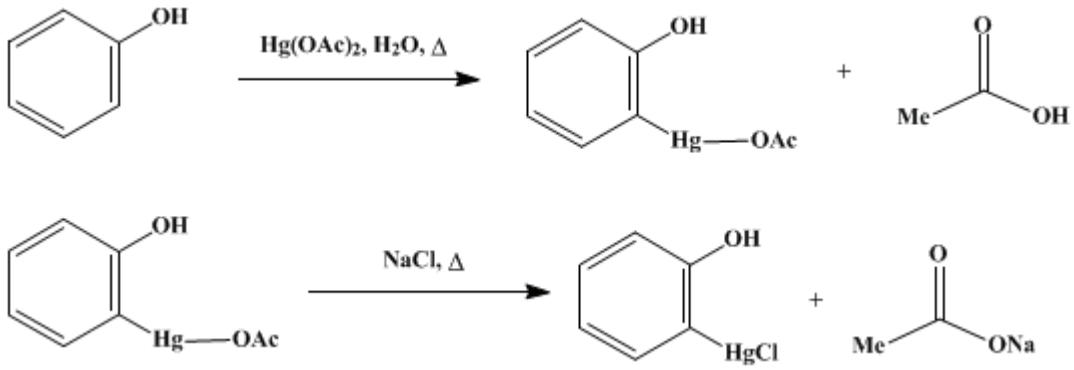
Organic Syntheses, Coll. Vol. 1, p.161 (1941); Vol. 4, p.13 (1925).

o-CHLOROMERCURIPHENOL

[Phenol, *o*-chloromercuri-]



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1. Procedure

Two liters of water is heated to boiling in a 3-l. flask. Mean-while 50 g. (0.53 mole) of phenol, in a 250-cc. tall beaker provided with a small glass mechanical stirrer, is heated to 170° on an electric heater (Note 1). The heat is then turned off and 100 g. (0.31 mole) of powdered mercuric acetate is added gradually (five to ten minutes) to the stirred phenol. After all of the mercuric acetate has dissolved in the phenol, the mercuration mixture is poured slowly into the hot water, the burner having previously been removed (Note 2). The beaker is rinsed out with some of the hot water. The mixture is boiled for five minutes and then filtered through filter paper in a large Büchner funnel which has been previously heated by blowing steam through it. The pink residue (Note 3) consists of a small amount of dimercurated phenol and some polymerization products.

The filtrate is again brought to boiling in a clean flask and treated with a solution of 20 g. (0.34 mole) of sodium chloride in 200 cc. of boiling water. The precipitate formed is *p*-chloromercuriphenol together with some colored impurities (Note 4). The mixture is heated to boiling and filtered through a large preheated Büchner funnel. The filtrate on cooling deposits white feathery crystals of *o*-chloromercuriphenol. The mixture is allowed to stand at least twelve hours and then filtered. The crystals are air-dried. They should be colorless and should melt above 147° (m.p. of pure substance 152°). If the product is pink or melts low it should be recrystallized from hot water. The average yield of fifteen experiments was 45 g. of ortho compound melting above 147°, which corresponds to 44 per cent of the theoretical amount (Note 5).

2. Notes

1. Mercuration at lower temperatures than that recommended gives a poorer yield of the ortho compound.
2. If the heat is not removed from under the water before the mercurated phenol is added, the mixture is likely to froth over.
3. Long heating of the mercuration mixture increases the amount of pink by-product.
4. The yield of impure para compound obtained as a by-product varies from 10 to 30 g.
5. A saturated solution of bromine in glycerol should be kept at hand as an antidote for phenol burns. If all undissolved bromine is allowed to settle out before the solution is used, there is no danger of bromine burns.
Limewater is also highly recommended for phenol burns.

3. Discussion

o-Chloromercuriphenol can be prepared by the mercuration of phenol in water solution[1] and with only phenol as the solvent.[2]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 326

References and Notes

1. Dimroth, Ber. **35**, 2855 (1902).
2. Whitmore and Middleton, J. Am. Chem. Soc. **43**, 622 (1921).

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

dimercurated phenol

mercurated phenol

para compound

Limewater

glycerol (56-81-5)

phenol (108-95-2)

sodium chloride (7647-14-5)

bromine (7726-95-6)

mercuric acetate (1600-27-7)

o-Chloromercuriphenol,
Phenol, o-chloromercuri-

p-chloromercuriphenol