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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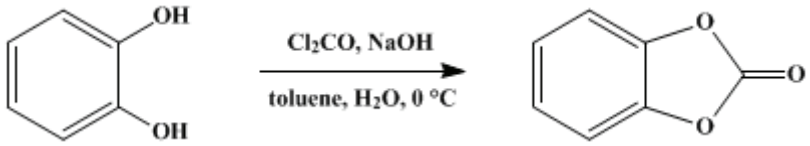
o-PHENYLENE CARBONATE

[Carbonic acid, *o*-phenylene ester]



Submitted by R. S. Hanslick, W. F. Bruce, and A. Mascitti[1].
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1. Procedure

Caution! This preparation should be conducted in a hood to avoid exposure to toxic phosgene.

In a 5-l. three-necked flask, filled with nitrogen, 110 g. (1.0 mole) of recrystallized catechol (Note 1) is dissolved in 250 ml. of deaerated water (Note 2) containing 88 g. (2.2 moles) of sodium hydroxide. The flask is fitted with a gas inlet tube, a thermometer dipping into the liquid, and an efficient glass mechanical stirrer with a gas-tight rubber slip seal and is immersed in an ice-salt bath. A positive nitrogen pressure of about 1 cm. is maintained by attaching the inlet tube to a source of nitrogen through a line containing a T-tube dipping into mercury. A solution of 200–225 g. (2.0–2.3 moles) of commercial phosgene in 750 ml. of toluene is prepared at 0° by bubbling the gas into toluene in a tared flask (Note 3), and the solution is added to the flask in portions of about 50 ml. with good mechanical stirring over a period of 60 to 75 minutes. During the addition the temperature is maintained at 0–5° by periodic addition to the mixture of clean cracked ice, free from dirt and iron rust. After addition of the toluene solution of phosgene is completed, the mixture is stirred at 0–5° for 1 hour and then allowed to come to room temperature. The mixture is filtered with suction, and the solid is pressed on the funnel to remove as much water as possible. The aqueous portion of the filtrate is separated, and the solid on the funnel is added to the toluene in the filtrate and dissolved by warming. The warm toluene solution is filtered and distilled under reduced pressure (water aspirator) until the product begins to crystallize. The residue is warmed to redissolve the solid, and then chilled. The *o*-phenylene carbonate is collected on a suction filter and dried in a vacuum desiccator; the yield is 98–110 g., m.p. 119–120°.

Concentration of the filtrate yields a second crop of impure product, which is recrystallized from toluene and then melts at 119–120°. The combined yield of pure white *o*-phenylene carbonate from the first and second crops is 107–116 g. (79–85%).

2. Notes

1. Catechol obtained from the Koppers Company, Pittsburgh, Pennsylvania, was recrystallized from toluene.
2. Water that was deaerated by boiling was used, and an atmosphere of nitrogen essentially free from oxygen (such as the Seaford grade of the Air Reduction Company) was maintained, in order to prevent discoloration of the alkaline solution of catechol due to oxidation.
3. Phosgene from a commercial cylinder was used (Matheson Company or Ohio Chemical Company). For the preparation of a solution of phosgene in toluene see *Organic Syntheses*.[2]

3. Discussion

o-Phenylene carbonate has been prepared by the distillation of *o*-hydroxyphenyl ethyl carbonate[3] and by the reaction of catechol with phosgene.[3,4]

References and Notes

1. Research Laboratory, Wyeth Institute, Philadelphia, Pennsylvania.
2. *Org. Syntheses Coll. Vol.* **3**, 167 (1955).
3. Einhorn and Lindenberg, *Ann.*, **300**, 141 (1898).
4. Nachfolger, Ger. pat. 72,806 [*Chem. Zentr.*, **65 I**, 805 (1894)].

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

iron rust

sodium hydroxide (1310-73-2)

oxygen (7782-44-7)

nitrogen (7727-37-9)

mercury (7439-97-6)

toluene (108-88-3)

phosgene (75-44-5)

Catechol (120-80-9)

o-Phenylene carbonate,
Carbonic acid, o-phenylene ester (2171-74-6)

o-hydroxyphenyl ethyl carbonate

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