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.

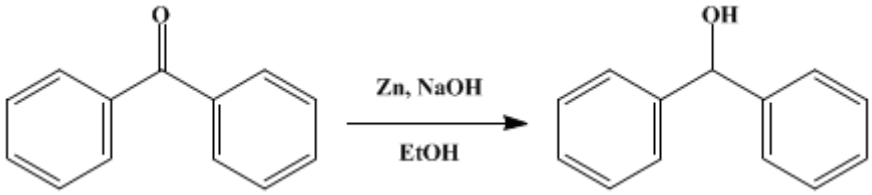
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

DOI:10.15227/orgsyn.008.0024

Organic Syntheses, Coll. Vol. 1, p.90 (1941); Vol. 8, p.24 (1928).

BENZOHYDROL



Submitted by F. Y. Wiselogle and H. Sonneborn, III.
Checked by C. S. Marvel and J. F. Kaplan.

1. Procedure

In a 3-l. round-bottomed flask fitted with a mechanical stirrer are placed 200 g. of technical flake sodium hydroxide, 200 g. of benzophenone, 2 l. of 95 per cent alcohol, and 200 g. of technical zinc dust. The stirrer is started and the mixture slowly warms to about 70° spontaneously. After two to three hours the mixture, which has started to cool, is filtered with suction, and the residue is washed twice with 100-cc. portions of hot alcohol (Note 1).

The filtrate is poured into five volumes of ice water acidified with about 425 cc. of concentrated commercial hydrochloric acid. The benzohydrol separates as a white crystalline mass and is filtered by suction. The yield of crude air-dried product melting at 65° is 194–196 g. (96–97 per cent of the theoretical amount). From 200 g. of crude product in 200 cc. of hot alcohol there is obtained, after cooling in an ice-salt mixture, filtering, and drying, 140–145 g. of product melting at 68°. The benzohydrol remaining in the mother liquors may be precipitated with water.

2. Notes

1. The filtered zinc dust is inflammable; it must not be allowed to dry in contact with combustible material.

3. Discussion

Benzohydrol can be prepared by reducing benzophenone with sodium amalgam;[1] with metallic calcium and alcohol;[2] with hydrogen in the presence of a catalyst;[3] with zinc, aluminum, or sodium in strongly alkaline solutions;[4] with zinc dust and alcoholic potassium hydroxide solution;[5] electrolytically;[6] with magnesium and absolute alcohol;[7] with magnesium and ammonium chloride in 95 per cent alcohol;[7] with isopropyl alcohol and a little sodium isopropoxide;[8] and with aluminum isopropoxide.[9]

References and Notes

1. Linnemann, Ann. **125**, 230 (1863); **133**, 6 (1865).
2. Marschalk, Ber. **43**, 642 (1910).
3. Vavon, Compt. rend. **155**, 287 (1912).
4. Böeseken and Cohen, Verhandel Akad. Wetenschappen Amsterdam, **16**, 91 (1913) [Chem. Zentr. I, 1375 (1915)].
5. Montagne, Rec. trav. chim. **25**, 402 (1906).
6. Müller, Z. Electrochem. **16**, 240 (1910).
7. Zeckmeister and Rom, Ann. **468**, 117 (1929).
8. Bachmann, J. Am. Chem. Soc. **55**, 391 (1933).
9. Lund, Ber. **70**, 1520 (1937).

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

Benzohydrol

alcohol (64-17-5)

hydrochloric acid (7647-01-0)

ammonium chloride (12125-02-9)

hydrogen (1333-74-0)

sodium hydroxide (1310-73-2)

magnesium (7439-95-4)

aluminum (7429-90-5)

potassium hydroxide (1310-58-3)

Benzophenone (119-61-9)

zinc (7440-66-6)

calcium (7440-70-2)

sodium (13966-32-0)

isopropyl alcohol (67-63-0)

sodium isopropoxide (683-60-3)

aluminum isopropoxide