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.

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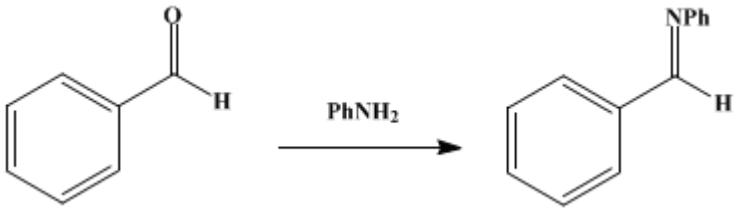
Organic Syntheses, Coll. Vol. 1, p.80 (1941); Vol. 8, p.22 (1928).

BENZALANILINE

[Aniline, N-benzylidene-]



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1. Procedure

In a 500-cc. three-necked, round-bottomed flask provided with a mechanical stirrer is placed 106 g. (1 mole) of benzaldehyde (Note 1), and 93 g. (1 mole) of aniline is added with rapid stirring. After a few seconds a reaction occurs with evolution of heat and separation of water. The mixture is allowed to stand fifteen minutes and is then poured, with vigorous stirring, into 165 cc. of 95 per cent alcohol in a 600-cc. beaker. Crystallization begins in about five minutes, and the mixture is allowed to stand, first ten minutes at room temperature, and then thirty minutes in ice water. The almost solid mass is next transferred to a large Büchner funnel, filtered by suction, pressed out, and air-dried. The yield of pure benzalaniline melting at 52° is 152–158 g. (84–87 per cent of the theoretical amount).

By concentrating the mother liquor to about one-half of its original volume at room temperature or lower, under reduced pressure by means of a water pump, an additional 10 g. of benzalaniline may be obtained. This is of good quality and melts at 51° (Note 2).

2. Notes

1. Freshly distilled reagents must be used to obtain the yields described. Distillation through a column is recommended. The benzaldehyde should be washed with 5 per cent sodium carbonate solution before being distilled under reduced pressure with minimum atmospheric exposure.
2. Removal of the alcohol by distillation at ordinary pressure gives a much darker product.
If a product of high purity is desired, it may be obtained by recrystallization from 85 per cent alcohol.

3. Discussion

Benzalaniline can be prepared from aniline and benzaldehyde[1] either without a solvent or in dilute alcohol saturated with carbon dioxide.[2]

References and Notes

1. Laurent and Gerhardt, Jahresber. 488 (1850); Bogert, Org. Syn. **5**, 13 (1925).
2. Pyl, Ber. **60**, 287 (1927).

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

alcohol (64-17-5)

aniline (62-53-3)

sodium carbonate (497-19-8)

carbon dioxide (124-38-9)

benzaldehyde (100-52-7)

Benzalaniline,
Aniline, N-benzylidene- (538-51-2)