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.

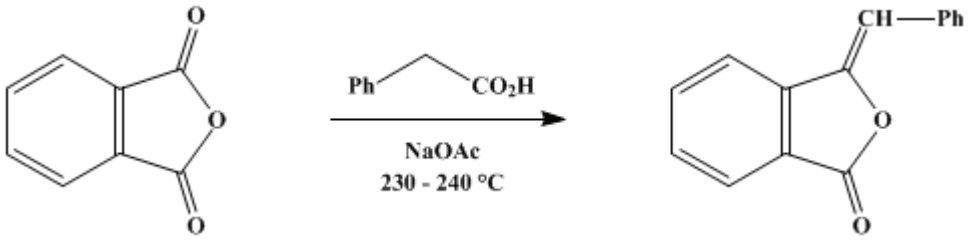
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.013.0010

Organic Syntheses, Coll. Vol. 2, p.61 (1943); Vol. 13, p.10 (1933).

BENZALPHTHALIDE

[Phthalide, 3-benzylidene-]



Submitted by Richard Weiss
Checked by John R. Johnson and H. R. Snyder.

1. Procedure

In a 500-cc. round-bottomed flask with a short neck (not longer than 3 cm.) are placed 100 g. (0.67 mole) of phthalic anhydride (Note 1), 110 g. (0.8 mole) of phenylacetic acid (Org. Syn. Coll. Vol. I, **1941**, 436), and 2.6 g. of freshly fused sodium acetate. A few chips of porous plate are added, and the flask is provided with a cork bearing a thermometer, which reaches almost to the bottom, and a wide, bent glass tube leading to a condenser. The tube ends just at the lower edge of the cork and does not protrude into the neck of the flask. The flask is imbedded up to the neck in a sand bath and is heated rapidly until the thermometer reaches 230°; then the temperature is raised slowly until the water produced in the reaction and some entrained organic matter pass out through the exit tube. The water is collected in a small vessel and its quantity noted from time to time in order to follow the progress of the reaction. The operation should be conducted so that the temperature rises from 230 to 240° in the course of about two hours. The reaction is maintained at 240° until the distillation of water ceases; this requires about one additional hour.

The flask now contains a brown mass covered with a film. The stopper is removed, and a test portion is taken out by means of a glass rod. The test portion is placed in a test tube or small beaker, treated with a little alcohol, and heated to boiling. When the reaction is complete, the material dissolves readily in the hot alcohol and crystallizes on cooling.

When this test has been found to be satisfactory, the flask is allowed to cool to 90–95°, and the product is dissolved in 400 cc. of boiling alcohol. The solution is filtered from insoluble matter and allowed to cool. The yellow crystals of benzalphthalide are filtered with suction and washed with 40–50 cc. of cold alcohol. The product weighs 115–116 g. and melts at 95–97°; for purification it is recrystallized from 370–380 cc. of alcohol. The yield of pure benzalphthalide, m.p. 100–101°, is 106–110 g. (71–74 per cent of the theoretical amount).

2. Notes

1. A good grade of sublimed phthalic anhydride should be used (m.p. 129–131°); if this is not available the ordinary phthalic anhydride can be purified by sublimation.

3. Discussion

Benzalphthalide has been prepared only from phthalic anhydride and phenylacetic acid in the presence of sodium acetate. The procedure given here is essentially that of Gabriel.[1]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 353

References and Notes

1. Gabriel, Ber. **18**, 3470 (1885).

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

alcohol (64-17-5)

sodium acetate (127-09-3)

phthalic anhydride (85-44-9)

Phenylacetic acid (103-82-2)

Benzalphthalide,
Phthalide, 3-benzylidene- (575-61-1)