



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.

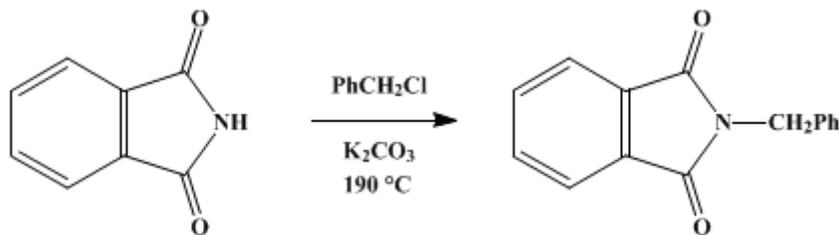
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

Organic Syntheses, Coll. Vol. 2, p.83 (1943); Vol. 12, p.10 (1932).

BENZYL PHTHALIMIDE

[Phthalimide, N-benzyl-]



Submitted by Richard H. F. Manske

Checked by Henry Gilman and H. J. Harwood.

1. Procedure

An intimate mixture of 166 g. (1.2 moles) of anhydrous potassium carbonate (Note 1) and 294 g. (2 moles) of phthalimide is treated with 506 g. (4 moles) of benzyl chloride (Note 2), and the mixture is heated in an oil bath at 190° under a reflux condenser for three hours (Note 3). While the mixture is still hot, the excess benzyl chloride is removed by steam distillation (Note 4). Near the end of this operation the benzyl phthalimide crystallizes. It is advisable to cool the mixture rapidly with very vigorous agitation so that the material is in as fine a state of division as possible. The solid is filtered on a large Büchner funnel, thoroughly washed with water, and drained as completely as possible by suction. It is then washed once with 400 cc. of 60 per cent alcohol and drained again. The yield of this product, melting at 100–110°, is 340–375 g. (72–79 per cent of the theoretical amount). It is conveniently purified by crystallizing from glacial acetic acid. The recovery in the crystallization is about 80 per cent, and the pure product melts at 116° (corr.) (Note 5).

2. Notes

1. The potassium carbonate is conveniently dehydrated by heating in a large basin over a moderate flame. It must be ground to a very fine powder and mixed with the phthalimide in a mortar.
2. A good grade of benzyl chloride having a boiling range of 3° was used.
3. There is no apparent advantage in using mechanical stirring.
4. The excess benzyl chloride is recovered from the distillate and dried with calcium chloride. About 200–300 g. is recovered.
5. Using the same general procedure trimethylene bromide furnishes γ -bromopropylphthalimide and, as a by-product, α,γ -diphthalimidopropane, while β -phenylethyl bromide gives β -phenylethylphthalimide.

3. Discussion

Benzyl phthalimide can be prepared from potassium phthalimide and benzyl chloride;¹ from phthalimide, potassium carbonate, and benzyl chloride;² and from phthalimide, sodium ethoxide, and benzyl chloride.³ The two latter procedures avoid the preparation of potassium phthalimide. Phthalic anhydride, benzylamine, and glacial acetic acid on refluxing also furnish benzyl phthalimide.⁴ The procedure described has been published.²

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 3, 256*

1. Gabriel, Ber. **20**, 2227 (1887).
 2. Ing and Manske, J. Chem. Soc. **1926**, 2348.
 3. Weisz and Lányi, Magyar Chem. Folyóirat **39**, 153 (1933) [C. A. **28**, 5815 (1934)].
 4. Vanags, Acta Univ. Latviensis, Kim. Fakultat. Ser. 4, No. 8, 405 (1939) [C. A. **34**, 1983 (1940)].
-

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

calcium chloride (10043-52-4)

potassium carbonate (584-08-7)

acetic acid (64-19-7)

Trimethylene bromide (109-64-8)

phthalic anhydride (85-44-9)

sodium ethoxide (141-52-6)

benzyl chloride (100-44-7)

Potassium Phthalimide (1074-82-4)

Phthalimide (85-41-6)

BENZYL PHTHALIMIDE

Phthalimide, N-benzyl- (2142-01-0)

γ -bromopropylphthalimide

β -phenylethyl bromide (103-63-9)

β -phenylethylphthalimide

benzylamine (100-46-9)

α,γ -diphthalimidopropane