



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.

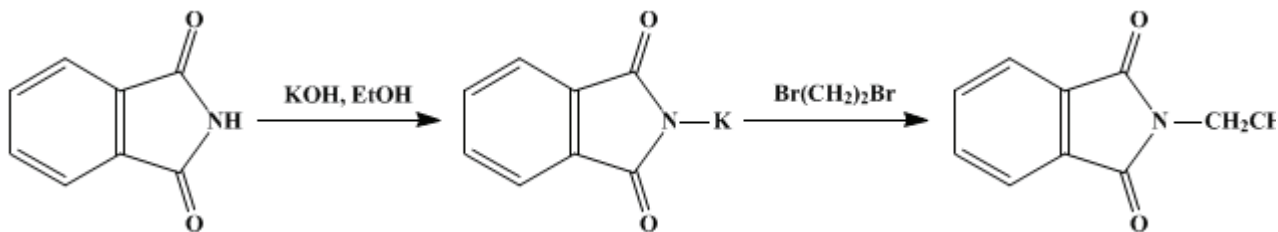
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.119 (1941); Vol. 7, p.8 (1927).

β-BROMOETHYLPHTHALIMIDE

[Phthalimide, *N*-(2-bromoethyl)-]



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1. Procedure

(A) *Potassium Phthalimide*.—In a 2-l. round-bottomed flask fitted with a reflux condenser are placed 80 g. (0.54 mole) of [phthalimide](#) ([Note 1](#)) and 1600 cc. of absolute [alcohol](#) ([p. 249](#)). The mixture is gently boiled for about fifteen minutes or until no more of the [phthalimide](#) dissolves ([Note 2](#)). The hot solution is decanted from any solid into a specially prepared solution of 30.5 g. (0.54 mole) of [potassium hydroxide](#) ([Note 3](#)). A precipitate of [potassium phthalimide](#) separates at once. The mixture is stirred and cooled quickly to room temperature, and the precipitate is filtered with suction. To the alcoholic mother liquors a second 80-g. portion of [phthalimide](#) is added, and the entire process is repeated. The two crops of crystals are united ([Note 4](#)) and washed with 200 cc. of [acetone](#) to remove any unchanged [phthalimide](#). The yield of air-dried [potassium phthalimide](#) is 160–180 g. (80–90 per cent of the theoretical amount).

(B) *β-Bromoethylphthalimide*.—In a 1-l. two-necked round-bottomed flask fitted with an efficient stirrer and a reflux condenser are placed 150 g. (0.81 mole) of [potassium phthalimide](#) and 450 g. (2.4 moles) of [ethylene dibromide](#) ([Note 5](#)). The stirrer is started and the mixture is heated for about twelve hours in an oil bath maintained at 180–190°. The condenser is then set for distillation, and the excess of [ethylene dibromide](#) is distilled under reduced pressure. The recovery of the [bromide](#) amounts to 290–295 g. ([Note 6](#)).

The crude [bromoethylphthalimide](#) is extracted from the [potassium bromide](#) by refluxing with 300 cc. of [alcohol](#) (98–100 per cent) ([Note 7](#)) until the dark oil is entirely dissolved. This requires about one-half hour. The hot solution is filtered with suction, and the residue of salt is washed with a little hot alcohol. The alcohol is distilled under reduced pressure, and the dry residue is refluxed with 500 cc. of [carbon disulfide](#) for about fifteen minutes in order to separate the soluble [bromoethylphthalimide](#) from the insoluble [diphthalimidoethane](#) ([Note 8](#)). The warm solution is filtered with suction. The [carbon disulfide](#) is distilled under diminished pressure ([Note 9](#)). The [bromoethylphthalimide](#) remains as light tan crystals which melt at 78–80°. The yield is 140–160 g. (69–79 per cent of the theoretical amount).

The product thus obtained is pure enough for most purposes. However, a purer product may be obtained by recrystallization from dilute alcohol in the presence of [decolorizing carbon](#). When 50 g. of crude product is dissolved in 200 cc. of 75 per cent [alcohol](#), boiled for about ten minutes with 5 g. of [decolorizing carbon](#) ([Norite](#)), filtered, and the solution cooled to 0°, 40 g. of white product is obtained. The recrystallized product melts at 80–81°. A second recrystallization raises the melting point to 82–83°.

2. Notes

1. The [phthalimide](#) used was the crude product obtained as described on [p. 457](#).
2. The crude product always contains a small amount of material insoluble in alcohol.

3. It is convenient to prepare enough [potassium hydroxide](#) solution for the two precipitations at one time. This solution is prepared by dissolving 61 g. of [potassium hydroxide](#) in 60 cc. of water. To this solution is then added 180 cc. of absolute [alcohol](#). One-half of the resulting solution is used for each 80-g. portion of [phthalimide](#).
4. After the first crop has been filtered, most of it should be removed and the second crop should be filtered through the same paper. The alcoholic filtrate on distillation yields about 1500 cc. of 98 per cent [alcohol](#).
5. The [ethylene dibromide](#) which was used boiled at 129–131°.
6. The last 15 to 20 g. of this recovered product distils very slowly.
7. The alcohol recovered from the preparation of the [potassium phthalimide](#) may be used for this extraction.
8. About 10 g. of crude [diphthalimidoethane](#) is obtained.
9. If the [carbon disulfide](#) is distilled at ordinary pressures, the residue melts and turns very dark. The crude yield is not lowered, but the purification is made more difficult.

3. Discussion

The procedure described is based on that of Gabriel.¹

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 1, 271](#)
- [Org. Syn. Coll. Vol. 2, 25](#)
- [Org. Syn. Coll. Vol. 3, 256](#)
- [Org. Syn. Coll. Vol. 4, 106](#)

References and Notes

1. Gabriel, Ber. **20**, 2225 (1887); **21**, 566 (1888); **22**, 1137 (1889).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[alcohol](#) (64-17-5)

[bromide](#) (24959-67-9)

[acetone](#) (67-64-1)

[decolorizing carbon](#),
[decolorizing carbon \(Norite\)](#) (7782-42-5)

[potassium hydroxide](#) (1310-58-3)

[carbon disulfide](#) (75-15-0)

[β-Bromoethylphthalimide](#),
[bromoethylphthalimide](#)

Potassium Phthalimide (1074-82-4)

Phthalimide (85-41-6)

ethylene dibromide (106-93-4)

potassium bromide (7758-02-3)

diphthalimidoethane

Phthalimide, N-(2-bromoethyl)- (574-98-1)