



A Publication  
of Reliable Methods  
for the Preparation  
of Organic Compounds

## Working with Hazardous Chemicals

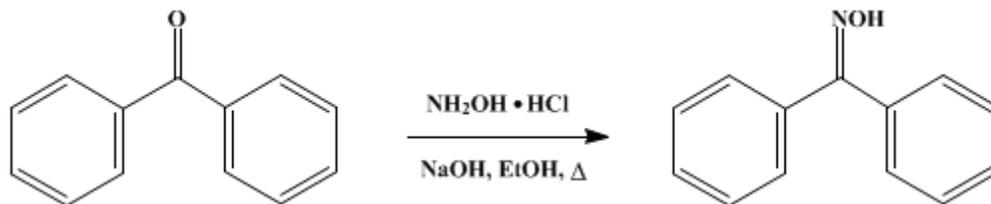
The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The 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](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.*

## BENZOPHENONE OXIME



Submitted by Arthur Lachman  
Checked by C. R. Noller

### 1. Procedure

A mixture of 100 g. (0.55 mole) of benzophenone (Org. Syn. Coll. Vol. I, 1941, 95), 60 g. (0.86 mole) of hydroxylamine hydrochloride (Org. Syn. Coll. Vol. I, 1941, 318), 200 cc. of 95 per cent ethyl alcohol, and 40 cc. of water is placed in a 2-l. round-bottomed flask. To this is added in portions, with shaking, 110 g. (2.75 moles) of powdered sodium hydroxide. If the reaction becomes too vigorous, cooling with tap water may be necessary. After all the sodium hydroxide has been added, the flask is connected to a reflux condenser, heated to boiling, and refluxed for five minutes. After cooling, the contents are poured into a solution of 300 cc. of concentrated hydrochloric acid in 2 l. of water. The precipitate is filtered with suction, thoroughly washed with water, and dried (Note 1). The yield is 106–107 g. (98–99 per cent of the theoretical amount) of a product melting at 141–142°. On crystallizing 20 g. from 80 cc. of methyl alcohol, 13 g. of crystalline material of the same melting point is obtained (Note 2).

### 2. Notes

1. This crude material dried overnight at about 40° is practically pure and if used at once is satisfactory for the preparation of diphenylmethane imine hydrochloride (p. 234).
2. In the presence of oxygen and traces of moisture, benzophenone oxime is gradually converted into a mixture of benzophenone and nitric acid.<sup>1</sup> A good method of preserving this oxime is to dry it in a vacuum desiccator, fill the desiccator with pure carbon dioxide, re-evacuate, and fill again with carbon dioxide. The preparation may then be transferred to a bottle, also filled with carbon dioxide, and sealed against access of air.

### 3. Discussion

Benzophenone oxime has been prepared in quantity by treating an aqueous alcoholic mixture of benzophenone and hydroxylamine hydrochloride with hydrochloric acid,<sup>2</sup> with sodium carbonate,<sup>3</sup> with alcoholic potassium hydroxide,<sup>4</sup> or with aqueous sodium hydroxide.<sup>5</sup> It has also been obtained by treating bisnitrosylbenzohydril with alcoholic potassium hydroxide,<sup>6</sup> and by the oxidation of  $\alpha$ -aminodiphenylmethane with magnesium persulfate solution.<sup>7</sup>

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 234

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### References and Notes

1. Hollemann, Rec. trav. chim. **13**, 429 (1894); Lachman, J. Am. Chem. Soc. **46**, 1478 (1924).
2. Beckmann, Ber. **19**, 989 (1886).
3. Janny, *ibid.* **15**, 2782 (1882).

4. Derick and Bornmann, J. Am. Chem. Soc. **35**, 1287 (1913).
  5. Lachman, *ibid.* **46**, 1481 (1924); **47**, 262 (1925).
  6. Behrend and Platner, Ann. **278**, 369 (1894).
  7. Bamberger and Seligman, Ber. **36**, 704 (1903).
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**Appendix**  
**Chemical Abstracts Nomenclature (Collective Index Number);**  
**(Registry Number)**

bisnitrosylbenzohydryl

ethyl alcohol (64-17-5)

hydrochloric acid (7647-01-0)

methyl alcohol (67-56-1)

sodium hydroxide (1310-73-2)

nitric acid (7697-37-2)

sodium carbonate (497-19-8)

oxygen (7782-44-7)

carbon dioxide (124-38-9)

potassium hydroxide (1310-58-3)

Benzophenone (119-61-9)

Hydroxylamine hydrochloride (5470-11-1)

Benzophenone oxime (574-66-3)

diphenylmethane imine hydrochloride (5319-67-5)

$\alpha$ -aminodiphenylmethane (91-00-9)

magnesium persulfate