



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.

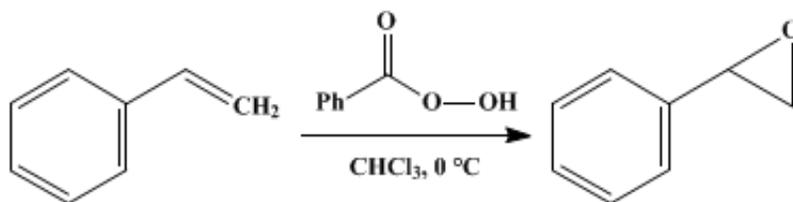
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.*

## STYRENE OXIDE

[Oxirane, 2-phenyl-]



Submitted by Harold Hibbert and Pauline Burt.

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### 1. Procedure

*Caution! Reactions and subsequent operations involving peracids and peroxy compounds should be run behind a safety shield. Peroxy compounds should be added to the organic material, never the reverse. For relatively fast reactions, the rate of addition of the peroxy compound should be slow enough so that it reacts rapidly and no significant unreacted excess is allowed to build up. The reaction mixture should be stirred efficiently while the peroxy compound is being added, and cooling should generally be provided since many reactions of peroxy compounds are exothermic. New or unfamiliar reactions, particularly those run at elevated temperatures, should be run first on a small scale. Reaction products should never be recovered from the final reaction mixture by distillation until all residual active oxygen compounds (including unreacted peroxy compounds) have been destroyed. Decomposition of active oxygen compounds may be accomplished by the procedure described in Korach, M.; Nielsen, D. R.; Rideout, W. H. *Org. Synth.* 1962, 42, 50 (*Org. Synth.* 1973, Coll. Vol. 5, 414). [Note added January 2011].*

To a solution of 42 g. (0.30 mole) of [perbenzoic acid](#) (p. 431) in 500 cc. of [chloroform](#) is added 30 g. (0.29 mole) of [styrene](#) (b.p. 141–143°) (p. 440). The solution is kept at 0° for twenty-four hours and is shaken frequently during the first hour. At the end of twenty-four hours, titration of an aliquot part of the solution shows that only the slight excess of [perbenzoic acid](#) remains ([Note 1](#)).

The [benzoic acid](#) is removed from the [chloroform](#) solution by shaking with an excess of 10 per cent [sodium hydroxide](#) solution, the alkali is removed by washing with water, and the [chloroform](#) solution is dried with anhydrous [sodium sulfate](#). It is then fractionated through an efficient distilling column (p. 130). Removal of the [chloroform](#) leaves a practically colorless liquid which distils at 188–192° (uncorr.) ([Note 2](#)). The yield is 24–26 g. (69–75 per cent of the theoretical amount).

### 2. Notes

1. The [perbenzoic acid](#) may be analyzed by mixing with an excess of acidified [potassium iodide](#) and titrating the liberated [iodine](#) with [sodium thiosulfate](#) (p. 434).
2. If the [styrene oxide](#) is distilled over a free flame some decomposition takes place as indicated by the formation of water. This is not observed if the product is distilled from an oil bath.

### 3. Discussion

[Styrene oxide](#) can be prepared by the action of [iodine](#), water, and [mercuric oxide](#) on [styrene](#).<sup>1</sup> The procedure described has been published.<sup>2</sup>

## References and Notes

1. Fourneau and Tiffeneau, Compt. rend. **140**, 1596 (1905).
  2. Hibbert and Burt, J. Am. Chem. Soc. **47**, 2240 (1925).
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### Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

sodium hydroxide (1310-73-2)

chloroform (67-66-3)

sodium sulfate (7757-82-6)

potassium iodide (7681-11-0)

sodium thiosulfate (7772-98-7)

Benzoic acid (65-85-0)

mercuric oxide (21908-53-2)

iodine (7553-56-2)

styrene (100-42-5)

Styrene oxide,  
Oxirane, 2-phenyl- (96-09-3)

Perbenzoic acid (93-59-4)