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.

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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.

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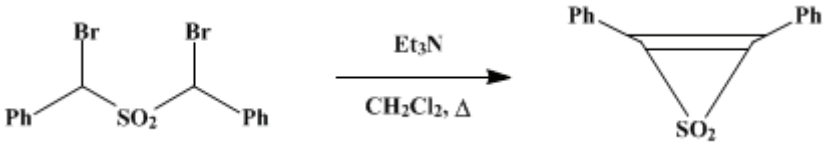
2,3-DIPHENYLVINYLENE SULFONE

[Thiirene, diphenyl-, 1,1-dioxide]



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1. Procedure

Caution! Benzene has been identified as a carcinogen; OSHA has issued emergency standards on its use. All procedures involving benzene should be carried out in a well-ventilated hood, and glove protection is required.

To a magnetically stirred solution of 6.36 g. (0.0157 mole) of crude α,α'-dibromodibenzyl sulfone (m.p. 135–150°; *Org. Synth.*, **Coll. Vol. 6**, 403 (1988)) in 40 ml. of dichloromethane contained in a 100-ml., round-bottomed flask fitted with a reflux condenser there is added in one portion 5.05 g. (0.0500 mole) of triethylamine. The solution is heated at reflux with stirring for 3 hours (Note 1), filtered, and the precipitate washed with 5 ml. of cold (0°) dichloromethane. The combined dichloromethane solution is washed with two 20-ml. portions of 3 *N* hydrochloric acid followed by one 10-ml. portion of water. Removal of the solvent by distillation from a water bath at 30° with the aid of a water aspirator gives 3.0 g. (80%) of the vinylene sulfone as a tan solid, m.p. 116–126° (dec.). The crude solid is washed with 5 ml. of cold (0°) ethanol and recrystallized from about 10 ml. of benzene, yielding 2.4–2.5 g. (63–67%) of the pure sulfone as tiny, snow-white needles, m.p. 123–126° (dec.) (Note 2).

2. Notes

1. After about 45 minutes triethylamine hydrobromide began to precipitate.
2. The submitters obtained a similar percent yield on ten times the scale.

3. Discussion

2,3-Diphenylvinylene sulfone has been prepared by dehydrohalogenation of either α,α'-dibromodibenzyl sulfone[2] [3] or α,α'-dichlorodibenzyl sulfone,[4] as well as by oxidation of 2,3-diphenylvinylene sulfoxide.[5] The procedure described here illustrates a general technique for the synthesis of diarylvinylene sulfones and has been extended to a number of substituted derivatives.[5,6] Aliphatic derivatives have been synthesized in other ways.[3,7,8] The vinylene sulfones are a group of compounds of considerable theoretical interest.[2,3,9] [10] [11] [12] [13]

References and Notes

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

2,3-Diphenylvinylene sulfone

2,3-diphenylvinylene sulfoxide

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

Benzene (71-43-2)

sulfone (7446-09-5)

sulfur (7704-34-9)

dichloromethane (75-09-2)

triethylamine (121-44-8)

triethylamine hydrobromide (636-70-4)

α,α'-Dibromodibenzyl sulfone (21966-50-7)

Thiirene, diphenyl-, 1,1-dioxide (5162-99-2)

vinylene sulfone

α,α'-dichlorodibenzyl sulfone