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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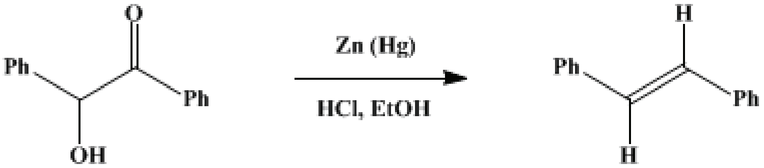
# *trans*-STILBENE



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## 1. Procedure

In a 4-l. beaker, equipped with a powerful mechanical stirrer which reaches nearly to the bottom, are placed 500 ml. of water and 50 g. of mercuric chloride. The stirrer is started, and 200 g. (3.06 gram atoms) of zinc dust (Note 1) is rapidly sifted into the suspension. Stirring is continued until the mercuric chloride dissolves (about 20–30 minutes). The zinc is then allowed to settle, the supernatant liquid is removed by decantation, and the amalgam is filtered and washed with 200 ml. of water. The zinc amalgam is returned to the beaker, which is now surrounded by an ice bath, and 500 ml. of 95% ethanol and 100 g. (0.48 mole) of benzoin are added. The stirrer is started, and 500 ml. of concentrated hydrochloric acid is added through a dropping funnel at such a rate that addition is complete in about 2 hours; throughout the reaction the temperature is maintained below 15°. Stirring is continued for about 2 hours more.

About 2 l. of cold water is added to the reaction mixture, and the insoluble material is collected on a Büchner funnel. The precipitate is transferred to a 2-l. beaker and extracted with two 600-ml. portions of hot ethanol. The combined extracts, on cooling, deposit long needles of stilbene which weigh 55–59 g. and melt at 116–121°. These are filtered with suction and are recrystallized from 600 ml. of 95% ethanol. The yield is 45–48 g. (53–57%) of colorless needless melting at 123–124°.

## 2. Notes

1. The zinc dust used was obtained from J. T. Baker and Company.

## 3. Discussion

The procedure described is essentially that of Ballard and Dehn.[1] Stilbene has also been prepared by reduction of desoxybenzoin,[2] benzaldehyde[3] and benzil;[2,4] by dehydrogenation of ethylbenzene,[5] toluene,[5,6,7] and bibenzyl;[6,8] by alkaline reduction of phenylnitromethane,[9] phenylnitroacetonitrile,[9] and desoxybenzoin;[10] by distillation of benzyl sulfone,[11] benzyl sulfide,[11,12] calcium cinnamate,[13] cinnamic acid,[14] phenyl cinnamate,[15,16] and diphenyl fumarate,[11] by dehydrohalogenation of α,α'-dichlorobibenzyl[17] and benzyl chloride;[18] by dehalogenation of α,α,α',α'-tetrachlorobibenzyl[19] and benzal chloride;[20] by the coupling of cinnamic acid and phenyldiazonium chloride;[21] by dehydration of benzylphenylcarbinol,[17,22] benzyl ether,[23] and benzyl alcohol;[23,24] by treatment of benzaldehyde ethyl mercaptal with Raney nickel;[25] by pyrolysis of methyl benzyl dithiocarbonate;[26] and by catalytic desulfurization of trithiobenzaldehyde.[27] The diazonium coupling reaction[21] appears to have the widest applicability for the synthesis of substituted stilbenes.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 5, 428

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

Desoxybenzoin

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

phenyldiazonium chloride

benzaldehyde (100-52-7)

Raney nickel (7440-02-0)

Benzil (134-81-6)

Benzoin (119-53-9)

toluene (108-88-3)

zinc (7440-66-6)

benzyl chloride (100-44-7)

Benzyl alcohol (100-51-6)

benzyl ether (103-50-4)

mercuric chloride (7487-94-7)

ethylbenzene (100-41-4)

cinnamic acid (621-82-9)

benzal chloride (98-87-3)

Phenylnitromethane (622-42-4)

stilbene

α,α'-dichlorobibenzyl

Phenyl cinnamate (2757-04-2)

bibenzyl (103-29-7)

phenylnitroacetonitrile

benzyl sulfone (620-32-6)

calcium cinnamate

diphenyl fumarate

benzaldehyde ethyl mercaptal

methyl benzyl dithiocarbonate

trithiobenzaldehyde

Benzyl sulfide (538-74-9)

trans-Stilbene (103-30-0)

α,α,α',α'-tetrachlorobibenzyl

benzylphenylcarbinol (614-29-9)