



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.

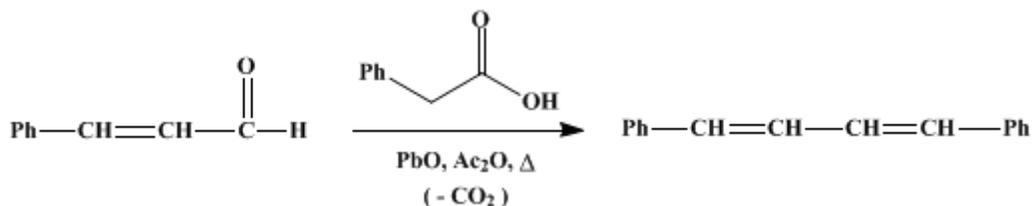
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.

Organic Syntheses, Coll. Vol. 2, p.229 (1943); Vol. 16, p.28 (1936).

1,4-DIPHENYLBUTADIENE

[Bisteryl]



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Checked by C. R. Noller and J. F. Carson.

1. Procedure

In a 1-l. round-bottomed flask equipped with a reflux condenser and protected by a calcium chloride tube are placed 150 g. (1.1 moles) of phenylacetic acid (*Org. Syn. Coll. Vol. I, 1941, 436*), 147 g. (1.1 moles) of freshly distilled cinnamic aldehyde, 122 g. of litharge, and 155 cc. of acetic anhydride. The mixture is boiled for five hours (*Note 1*), poured while still hot into a beaker, and allowed to stand overnight. The semisolid contents are stirred to a mush, filtered with suction in a large Büchner funnel, and pressed dry. The solid is washed on the funnel with two 35-cc. portions of ethyl alcohol, the material being stirred thoroughly before suction is applied. The cake is transferred to a beaker, stirred to a mush with 50 cc. of alcohol, and again filtered with suction. The material is washed with another 50-cc. portion of alcohol in the same manner (*Note 2*). The product, which is light yellow to tan in color, weighs 62–67 g. (27–29 per cent of the theoretical amount) (*Note 3*) and melts at 149.5–153.5°.

For purification the material is dissolved in 300 cc. of hot benzene; the solution is boiled three minutes with 5 g. of decolorizing carbon, and filtered hot, with gentle suction, through a warm Büchner funnel. The benzene filtrate is treated with 500 cc. of hot ethyl alcohol, heated to boiling, and then cooled to 10° in an ice bath, with shaking. After the crystals are filtered with suction and pressed thoroughly, 50 cc. of ethyl alcohol is allowed to soak into the cake and suction is applied again. The recrystallized product weighs 52–57 g. (23–25 per cent of the theoretical amount) (*Note 4*) and melts at 152.5–153.5°. This product is the trans-trans form of the diene.

2. Notes

1. During the first half hour, the flask should be heated gently and shaken several times to facilitate solution of the lead oxide.
2. Thorough washing is essential.
3. About 8 g. of crude hydrocarbon can be recovered from the original mother liquor, but the recovery is tedious and is not recommended.
4. An additional quantity of about 5.5 g. can be obtained by evaporating the mother liquor to 25 cc. and allowing to crystallize.

3. Discussion

The only method of preparative interest is the condensation of phenylacetic acid and cinnamic aldehyde.¹ The above procedure follows the method of Kuhn and Winterstein.² Bisteryl has been obtained also from styrylmagnesium bromide and cupric chloride,³ or azobenzene,⁴ and by the reduction of β-bromostyrene with hydrazine in the presence of palladium.⁵

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 5, 499*

References and Notes

1. Thiele and Schleussner, Ann. **306**, 198 (1899).
 2. Kuhn and Winterstein, Helv. Chim. Acta **11**, 103 (1928).
 3. Sakellarios and Kyrimis, Ber. **57**, 325 (1924); Gilman and Parker, J. Am. Chem. Soc. **46**, 2827 (1924).
 4. Gilman and Pickens, *ibid.* **47**, 2410 (1925).
 5. Busch and Weber, J. prakt. Chem. (2) **146**, 54 (1936).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

litharge

ethyl alcohol,
alcohol (64-17-5)

Benzene (71-43-2)

acetic anhydride (108-24-7)

decolorizing carbon (7782-42-5)

palladium (7440-05-3)

Phenylacetic acid (103-82-2)

cupric chloride (7758-89-6)

cinnamic aldehyde

hydrazine (302-01-2)

β -bromostyrene (103-64-0)

Azobenzene (103-33-3)

1,4-Diphenylbutadiene,
Bistryl (886-65-7)

styrylmagnesium bromide