



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.

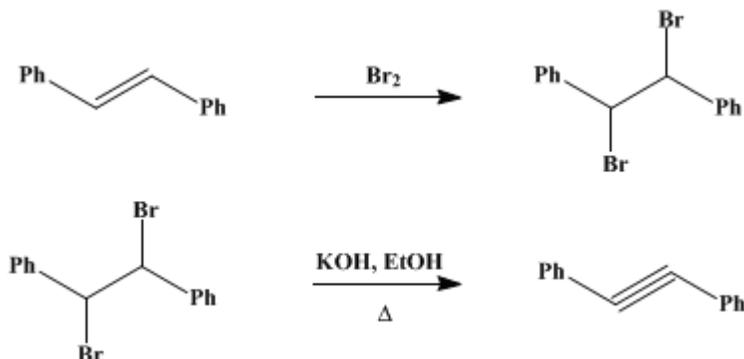
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.350 (1955); Vol. 22, p.50 (1942).

DIPHENYLACETYLENE

[Tolan]



Submitted by Lee Irvin Smith and M. M. Falkof.
Checked by W. E. Bachmann and Charles E. Maxwell.

1. Procedure

A solution of 45 g. (0.25 mole) of *trans*-stilbene in 750 ml. of ether is prepared in a 1-l. three-necked round-bottomed flask fitted with a reflux condenser, an efficient mechanical stirrer, and a dropping funnel. To the well-stirred solution there is added 13.8 ml. (43 g.; 0.27 mole) of bromine, during the course of 10 minutes. A solid begins to separate in 5 minutes, but stirring is continued for 1 hour. The product is collected on a Büchner funnel and washed with ether until it is white. The yield of stilbene dibromide, melting at 235–237°, is 65.8–69.1 g. (77–81%).

A solution of 90 g. of potassium hydroxide in 150 ml. of absolute ethanol is prepared in a 500-ml. round-bottomed flask fitted with a reflux condenser (Note 1). The solution is cooled somewhat, and the stilbene dibromide is added in several portions (Note 2). The mixture is then refluxed for 24 hours in an oil bath (Note 3). The hot mixture is poured into 750 ml. of cold water, and the product is removed by filtration and washed with 50 ml. of water. The crude diphenylacetylene is dried over calcium chloride in a vacuum desiccator for 18 hours at room temperature. The chunky, pale yellow crystals melt at 58–60° and weigh 33.7–37.8 g. By recrystallization of this product from 50 ml. of 95% ethanol there is obtained 29.2–30.5 g. (66–69% based on the stilbene) of pure diphenylacetylene, which forms white needles melting at 60–61° (Note 4).

2. Notes

1. Complete solution of the alkali in the alcohol is effected by placing the flask in an oil bath at 130–140°.
2. The addition of the stilbene dibromide causes an immediate, vigorous reaction, with the evolution of heat. It is necessary to replace the reflux condenser after each addition, until boiling has ceased.
3. An electrically heated oil bath is used to maintain a constant bath temperature of 130–140°.
4. The submitters obtained the same percentage yield on runs four times this size; the time employed was the same as that described here, except for the addition of bromine, which required 30 minutes.

3. Discussion

Diphenylacetylene has been prepared by the present method;¹ by the action of sodium amide on monochlorostilbene;² by the action of potassium amide on stilbene dichloride;³ by heating 1,1-diphenyl-2-chloroethane with sodium ethoxide in a sealed tube;⁴ and from benzil via the hydrazone.⁵

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 377
- Org. Syn. Coll. Vol. 5, 604
- Org. Syn. Coll. Vol. 6, 531

References and Notes

1. Smith and Hoehn, *J. Am. Chem. Soc.*, **63**, 1180 (1941); Söderbäck, *Ann.*, **443**, 161 (1925).
 2. Paillard and Wieland, *Helv. Chim. Acta*, **21**, 1363 (1938).
 3. Coleman and Maxwell, *J. Am. Chem. Soc.*, **56**, 133 (1934).
 4. Buttenberg, *Ann.*, **279**, 327 (1894)
 5. Schlenk and Bergmann, *Ann.*, **463**, 76 (1928)
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Appendix

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

hydrazone

ethanol (64-17-5)

calcium chloride (10043-52-4)

ether (60-29-7)

bromine (7726-95-6)

Benzil (134-81-6)

potassium hydroxide (1310-58-3)

Tolan (108-88-3)

sodium ethoxide (141-52-6)

1,1-diphenyl-2-chloroethane

sodium amide (7782-92-5)

stilbene

Diphenylacetylene (501-65-5)

Stilbene dibromide (5789-30-0)

monochlorostilbene

potassium amide

stilbene dichloride

trans-Stilbene (103-30-0)

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