



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.

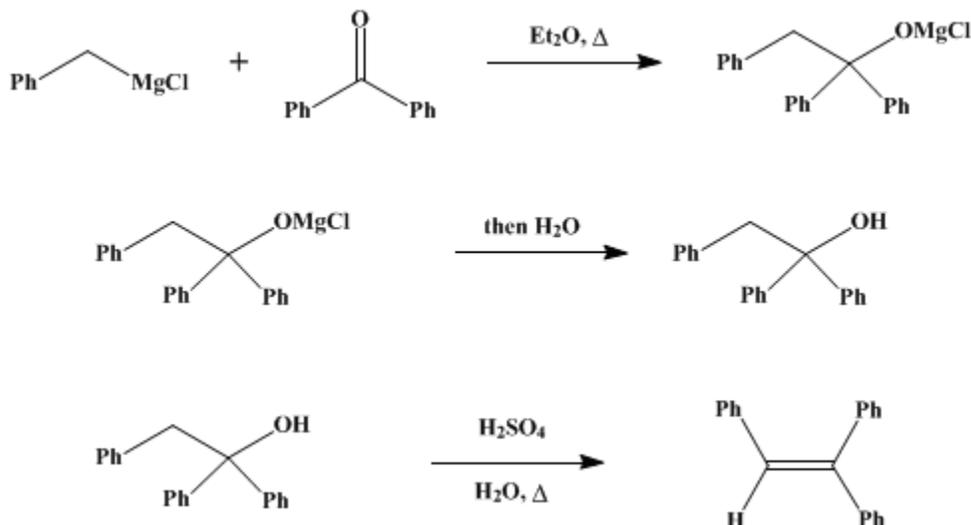
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.606 (1943); Vol. 17, p.89 (1937).

TRIPHENYLETHYLENE

[Ethylene, triphenyl-]



Submitted by Homer Adkins and Walter Zartman.

Checked by C. R. Noller and F. M. McMillan.

1. Procedure

In a 3-l. three-necked flask, fitted with a mechanical stirrer, a reflux condenser, and a separatory funnel, are placed 24.3 g. (1 gram atom) of **magnesium turnings**, 500 cc. of absolute **ether**, a crystal of **iodine**, and a 5- to 10-cc. portion of 126.5 g. (115 cc., 1 mole) of freshly distilled **benzyl chloride** (b.p. 177–179°). In a few minutes the reaction starts (**Note 1**) and is controlled if necessary by cooling with a wet towel. The stirrer is started, and the balance of the **benzyl chloride** is run in as fast as the refluxing will permit. The addition requires from one to two hours, and when completed the mixture is refluxed on the steam bath with stirring for three hours. With the stirrer still running, 182 g. (1 mole) of **benzophenone** (*Org. Syn. Coll. Vol. I, 1941, 95*) dissolved in 500 cc. of absolute **ether** is added at such a rate that the mixture refluxes rapidly. This requires about twenty minutes, and then the reaction mixture is allowed to stand for two hours (**Note 2**).

The flask is placed in an ice bath, 700 g. of cracked ice is added, and the **magnesium hydroxide** is dissolved by adding 500 cc. of cold 20 per cent **sulfuric acid**. The **ether** layer is separated, and the water layer extracted with two 200-cc. portions of **ether**. The **ether** is distilled from the combined extracts, and the residual liquid is refluxed for two hours with 100 cc. of 20 per cent **sulfuric acid** to dehydrate the carbinol. The layers are separated and the product vacuum-distilled (**Note 3**). The fraction boiling at 215–225°/15 mm. weighs 160–170 g. and melts at 60–68° (**Note 4**). Crystallization from 900 cc. of hot 95 per cent **alcohol** and cooling to 0° gives 140–150 g. of the hydrocarbon melting at 68–69° (54–59 per cent of the theoretical amount). Concentration of the mother liquor to 150 cc. and cooling gives 5–10 g., m.p. 65–67.5° (**Note 5**).

2. Notes

1. If the reaction does not start within thirty minutes, the mixture is warmed on a water bath with the stirrer running.
2. Slightly better yields are obtained if the reaction mixture is allowed to stand overnight.
3. The product should not be washed with water as the presence of a trace of **sulfuric acid** during the distillation seems to be necessary to complete the dehydration.

4. Seeding is usually necessary to induce crystallization.
5. By the same procedure, [stilbene](#) may be prepared from [benzaldehyde](#) and [benzylmagnesium chloride](#) in 25–35 per cent yield.

3. Discussion

[Triphenylethylene](#) has been prepared by the reaction between [phenylmagnesium bromide](#) and [desoxybenzoin](#) or [ethyl phenylacetate](#);¹ and by the reaction between [diphenylketene-quinoline](#) and [benzaldehyde](#).² The procedure given above is an adaptation of that published by Hell and Wiegandt.³ The intermediate [diphenylbenzylcarbinol](#) can be dehydrated very effectively by heating with [potassium bisulfate](#) or by boiling its solution in glacial [acetic acid](#).⁴

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 5, 952](#)

References and Notes

1. Klages and Heilmann, *Ber.* **37**, 1455 (1904).
 2. Staudinger and Kon, *Ann.* **384**, 89 (1911).
 3. Hell and Wiegandt, *Ber.* **37**, 1429 (1904).
 4. Van de Kamp and Sletzinger, *J. Am. Chem. Soc.* **63**, 1880 (1941).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[Desoxybenzoin](#)

[diphenylketene-quinoline](#)

[alcohol](#) (64-17-5)

[sulfuric acid](#) (7664-93-9)

[acetic acid](#) (64-19-7)

[ether](#) (60-29-7)

[magnesium turnings](#) (7439-95-4)

[potassium bisulfate](#) (7646-93-7)

[benzaldehyde](#) (100-52-7)

[iodine](#) (7553-56-2)

[Benzophenone](#) (119-61-9)

[benzyl chloride](#) (100-44-7)

benzylmagnesium chloride (6921-34-2)

magnesium hydroxide

Phenylmagnesium bromide (100-58-3)

Ethyl phenylacetate (101-97-3)

Triphenylethylene,
Ethylene, triphenyl- (58-72-0)

stilbene

diphenylbenzylcarbinol (4428-13-1)