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.

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.

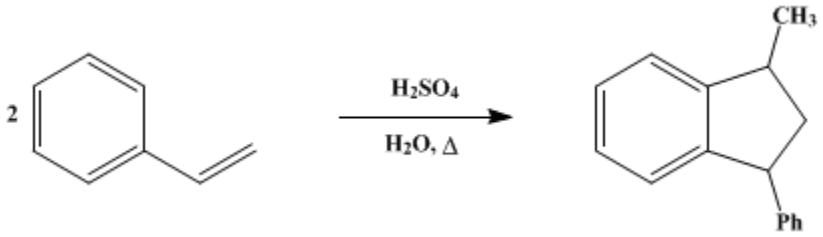
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.035.0083

Organic Syntheses, Coll. Vol. 4, p.665 (1963); Vol. 35, p.83 (1955).

1-METHYL-3-PHENYLINDANE

[Indan, 1-methyl-3-phenyl-]



Submitted by Milton J. Rosen[1]
Checked by R. T. Arnold and William K. Witsiepe.

1. Procedure

In a 500-ml. three-necked round-bottomed flask fitted with a mechanical stirrer and a reflux condenser are placed 50 g. (0.48 mole) of styrene (Note 1) and a previously cooled solution of 100 ml. of concentrated sulfuric acid in 150 ml. of water. The mixture is stirred vigorously (Note 2) and heated under reflux in an oil bath for approximately 4 hours.

Without interrupting stirring or heating, 50 ml. of concentrated sulfuric acid is added slowly through the condenser, and the mixture is stirred and heated for an additional 12 hours (Note 3).

The reaction mixture is cooled, cautiously poured into 250 ml. of cold water with stirring, and allowed to separate into layers. The upper hydrocarbon layer is removed, and the lower layer is extracted with three 50-ml. portions of ether. The combined ether extracts and hydrocarbon layer are washed successively with about 30 ml. each of a saturated solution of sodium bicarbonate, water, and a saturated solution of calcium chloride, and then dried over anhydrous calcium chloride. The ether is removed by distillation, and the product is distilled under reduced pressure. The yield of 1-methyl-3-phenylindane, b.p. 168–169°/16 mm. (Note 4), n_D^{20} 1.5811 ± 0.0005, is 38.5–40.5 g. (77–81%) (Note 5).

2. Notes

1. Commercial styrene, distilled from a water bath at about 80–100 mm. pressure just before use, is employed.
2. It is essential to use an efficient stirring device, capable of forming a dispersion of the styrene in the acid layer.
3. The sulfuric acid is added in two portions in order to minimize higher-polymer formation.[2]
4. The product also distils at 150–151°/6.5 mm.
5. The submitter states that α-methylstyrene can be converted to 1,1,3-trimethyl-3-phenylindane, b.p. 154–155°/8 mm.., m.p. 50.4–52.1°, by the same general procedure. The yield is 86–89% of the theoretical amount. The 1,1,3-trimethyl-3-phenylindane may be purified further by one recrystallization from three times its weight of 80% isopropyl alcohol. The yield of purified product, m.p. 51.8–52.3°, is 80–83% (based on the original weight of monomer used).

3. Discussion

1-Methyl-3-phenylindane has been prepared by the treatment of dimeric styrene[3] with aqueous sulfuric acid, and by the hydrogenation of 1-methyl-3-phenyl-Δ^2-indene.[4]

References and Notes

1. Brooklyn College, Brooklyn, New York.
2. Rosen, *J. Org. Chem.*, **18**, 1701 (1953).
3. Spoerri and Rosen, *J. Am. Chem. Soc.*, **72**, 4918 (1950).
4. Müller and Körmendy, *J. Org. Chem.*, **18**, 1237 (1953).

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

1-methyl-3-phenyl-Δ^2-indene

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

ether (60-29-7)

sodium bicarbonate (144-55-8)

isopropyl alcohol (67-63-0)

styrene (100-42-5)

1-Methyl-3-phenylindane,
Indan, 1-methyl-3-phenyl- (6416-39-3)

α-methylstyrene (98-83-9)

1,1,3-trimethyl-3-phenylindane