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.

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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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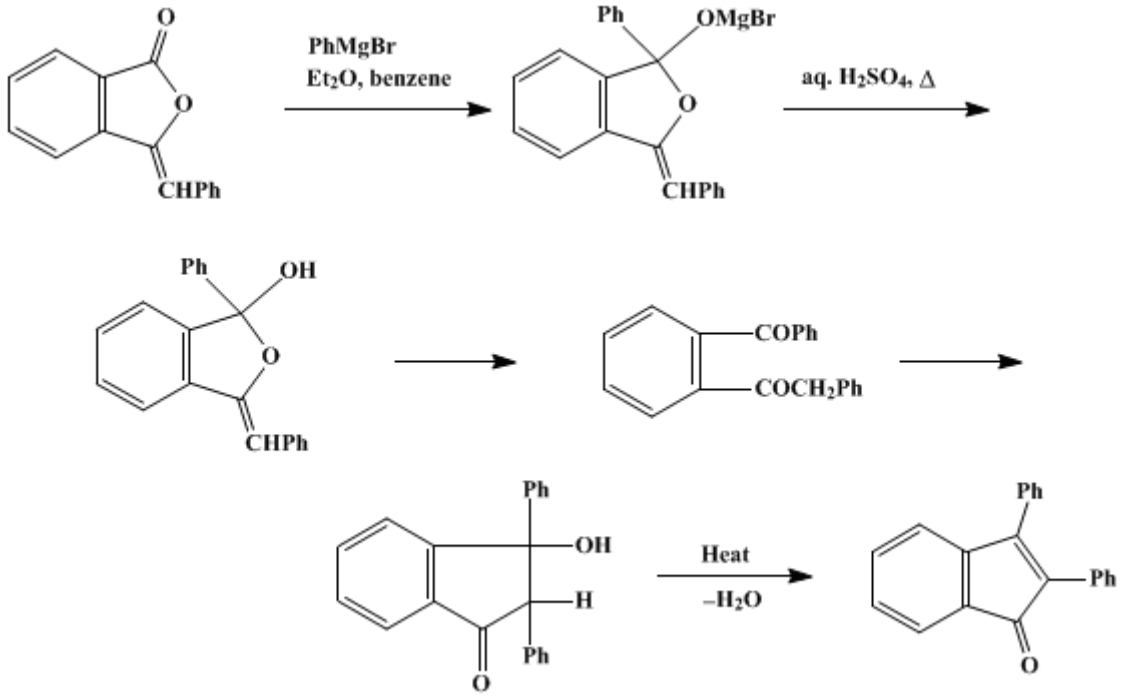
2,3-DIPHENYLINDONE (2,3-DIPHENYL-1-INDENONE)

[Indone, 2,3-diphenyl-]



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1. Procedure

A solution of phenylmagnesium bromide is prepared in the usual manner[1] using 12.2 g. (0.5 gram atom) of magnesium, 78.5 g. (52 ml., 0.5 mole) of bromobenzene, and 500 ml. of absolute ether in a 2-l. round-bottomed three-necked flask fitted with a reflux condenser, a mechanical stirrer, and a dropping funnel. No unreacted magnesium should remain; if any does, an additional 1–2 ml. of bromobenzene should be added.

To this vigorously stirred solution is added slowly a solution of 44.5 g. (0.20 mole) of benzalphthalide[2] in 400 ml. of dry benzene. After about half of this solution has been added, the magnesium complex begins to separate; it hinders the stirring somewhat. When all the phthalide solution has been admitted (about 1 hour), the reflux condenser is replaced by a still head carrying a thermometer and attached to a condenser set for downward distillation. The bulk of the solvent is then removed; this requires about 30 minutes, the temperature of the vapor remaining at 50° for about half this time and rising to about 65° toward the end. About 220–230 ml. of distillate is obtained. The flask and contents are then immersed in an ice bath, and the magnesium complex is decomposed by the slow addition of a cold solution of 15 ml. of concentrated sulfuric acid in 300 ml. of water (Note 1) with rapid stirring. The upper benzene layer is separated and transferred to a 1-l. Claisen flask, and the solvent is removed by distillation from a steam bath; this requires about 4 hours. The residual thick red syrup is transferred to a 125-ml. Claisen flask with a wide side arm set up for vacuum distillation. The residue is heated under a pressure of about 10 mm. to remove all low-boiling material (Note 2) and then is distilled under reduced pressure. The fraction boiling at 215–255°/6 mm. (195–220°/1 mm.) is collected, most of the distillate coming over at 235–240°/6 mm. The distillate is dissolved in 50 ml. of boiling benzene, 200 ml. of hot 95% ethanol is added, and the solution is chilled in an ice bath for 2 hours. The red, crystalline 2,3-diphenylindone is collected on a filter, washed with 50 ml. of cold 95%

ethanol, and air-dried. The yield is 34–40 g. (60–71%) of red crystals melting at 149–151° (Note 3) and (Note 4).

2. Notes

1. The decomposition is vigorous, and the acid must be admitted slowly at first but may be added more rapidly toward the end.
2. During this heating any carbinol present is dehydrated. Low and erratic yields usually indicate incomplete dehydration.
3. The product may be recrystallized by dissolving it in 50 ml. of boiling benzene and diluting with 200 ml. of hot ethanol. The recovery of material, m.p. 150–151°, is about 90%.
4. This procedure is capable of considerable variation, by which other indenones may be secured. For example, the benzalphthalide may be replaced by other phthalides made from (*a*) other aldehydes, or (*b*) other anhydrides; the phenylmagnesium bromide can be replaced by other Grignard reagents.

3. Discussion

2,3-Diphenylindone has been prepared by the action of phenylmagnesium bromide upon benzalphthalide[3,4] and by ring closure from α,β-diphenylcinnamic acid[5,6] or from 2,3,3-triphenyl-3-hydroxypropionic acid.[7,8]

References and Notes

1. *Org. Syntheses* Coll. Vol. **1**, 226 (1941).
2. *Org. Syntheses* Coll. Vol. **2**, 61 (1943).
3. Löwenbein and Ulich, *Ber.*, **58**, 2662 (1925).
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6. Weitz and Scheffer, *Ber.*, **54**, 2341 (1921).
7. Ivanoff and Ivanoff, *Compt. rend.*, **226**, 1199 (1948).
8. Ivanoff and Ivanoff, *Annuaire univ. Sofia, Faculté sci.*, **44**, (2), 121 (1947–1948) [*C. A.*, **44**, 3960 (1950)].

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

Indone, 2,3-diphenyl-

magnesium complex

2,3-Diphenylindone

ethanol (64-17-5)

sulfuric acid (7664-93-9)

Benzene (71-43-2)

ether (60-29-7)

magnesium (7439-95-4)

bromobenzene (108-86-1)

Phenylmagnesium bromide (100-58-3)

Benzalphthalide (575-61-1)

2,3-DIPHENYL-1-INDENONE (1801-42-9)

α,β-diphenylcinnamic acid

2,3,3-triphenyl-3-hydroxypropionic acid