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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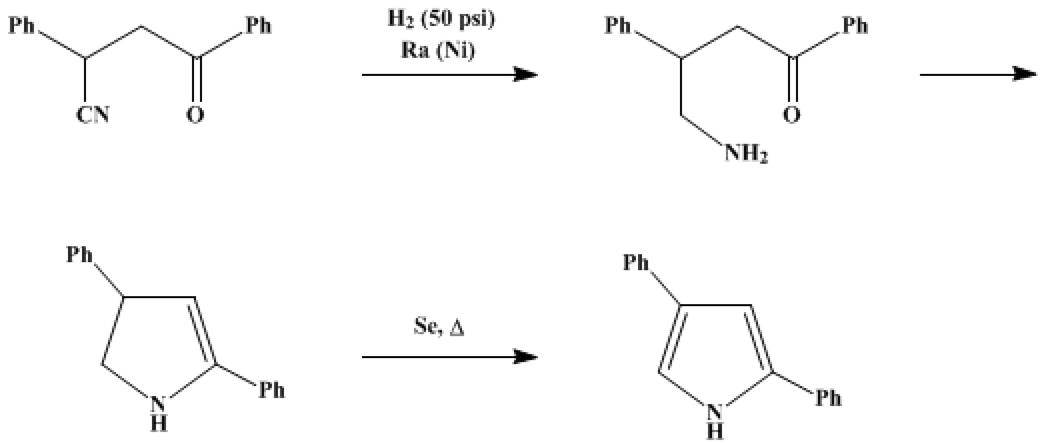
Organic Syntheses, Coll. Vol. 3, p.358 (1955); Vol. 27, p.33 (1947).

2,4-DIPHENYLPYRROLE

[Pyrrole, 2,4-diphenyl-]



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1. Procedure

A suspension of 61 g. (0.26 mole) of α-phenyl-β-benzoylpropionitrile[1] (Note 1) in 150 ml. of methanol and 1 level teaspoonful of Raney nickel catalyst[2,3] (Note 2) are placed in a 400–500 ml. pressure bottle (Note 3) connected to a low-pressure reduction apparatus. The bottle is alternatively evacuated and filled with hydrogen twice, and the reduction is conducted at 80–90° by shaking with hydrogen at an initial pressure of 50 lb. After 2 moles of hydrogen is absorbed the reduction is discontinued, the solution is filtered to separate the catalyst, and the solvent is removed from the green filtrate by distillation under reduced pressure. The crude 2,4-diphenyl-2-pyrroline (Note 4) is rinsed into a 300-ml. Kjeldahl flask with a little methanol, which is removed by distillation at reduced pressure (water pump) while the flask is immersed in a bath at 100°. Selenium (10 g.) is added, and the mixture is heated in a metal bath at 245–265° for 5 hours while a slow stream of nitrogen is passed over the surface of the liquid. This operation is conducted in a well-ventilated hood. The molten mass is poured from the reaction flask into a beaker (Note 5), and the product is extracted with 300 ml. of boiling toluene. The green toluene solution is filtered through a cotton plug to remove a small amount of suspended selenium, and the filtrate is cooled. The 2,4-diphenylpyrrole separates as light green crystals which melt at 174–176° and weigh 24–26 g. (42–46%) (Note 6). A second crop of 2–3 g. (4–5%), m.p. 160–170°, can be obtained by concentrating the filtrate and cooling.

2. Notes

1. The α-phenyl-β-benzoylpropionitrile[1] was used without recrystallization (m.p. 123–125°).
2. The time required for the hydrogenation was 2.5 hours with a very active nickel catalyst, and longer with a less active catalyst. Raney nickel prepared according to [2] is a particularly active catalyst and gives excellent results in this preparation.
3. A 500-ml. Pyrex centrifuge bottle is satisfactory.
4. The submitters state that distillation yields pure 2,4-diphenyl-2-pyrroline, b.p. 170–172°/3 mm., 203–205°/8 mm., which solidifies on cooling (yield 80–90%).
5. If the contents of the flask are not poured into a beaker while molten, it is difficult to remove the cake of selenium. If more than one preparation is to be made, the contents of the flask are allowed to cool and the product is extracted with hot toluene. The selenium can be reused.
6. The product is sufficiently pure for most purposes. Recrystallization from toluene gives a 90–93% recovery of a very light-green product, m.p. 179–180° (cor.). The submitters state that the product

obtained in the first crystallization has this melting point if the 2,4-diphenyl-2-pyrroline is purified by distillation before dehydrogenation.

3. Discussion

2,4-Diphenylpyrrole has been prepared only by dehydrogenation of 2,4-diphenyl-2-pyrroline with selenium, Raney nickel, and nickel supported on pumice.[4]

References and Notes

1. *Org. Syntheses* Coll. Vol. **2**, 498 (1943)
2. Pavlic and Adkins, *J. Am. Chem. Soc.*, **68**, 1471 (1946).
3. *Org. Syntheses* Coll. Vol. **3**, 176, 181 (1954).
4. Rogers, *J. Chem. Soc.*, **1943**, 594.

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

methanol (67-56-1)

hydrogen (1333-74-0)

nitrogen (7727-37-9)

nickel,
Raney nickel (7440-02-0)

toluene (108-88-3)

selenium

α-Phenyl-β-benzoylpropionitrile (6268-00-4)

2,4-Diphenylpyrrole,
Pyrrole, 2,4-diphenyl- (3274-56-4)

2,4-Diphenyl-2-pyrroline