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.

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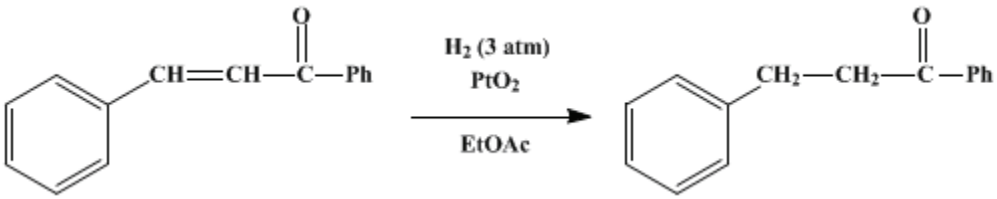
Organic Syntheses, Coll. Vol. 1, p.101 (1941); Vol. 8, p.36 (1928).

BENZYLACETOPHENONE

[Propiophenone, β-phenyl-]



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1. Procedure

A solution of 20.8 g. (0.1 mole) of benzalacetophenone (p. 78) (Note 1) in 150 cc. of c.p. ethyl acetate (Note 2) is placed in the reaction bottle of the catalytic reduction apparatus (p. 61), and 0.2 g. of platinum oxide catalyst (p. 463) is added. The apparatus is evacuated, then filled with hydrogen, and the mixture shaken with hydrogen until 0.1 mole has been absorbed. The time required is usually about fifteen to twenty-five minutes (Note 3). The platinum is filtered off and the solvent removed from the filtrate by distillation. The benzylacetophenone is recrystallized from about 25 cc. of alcohol and melts at 72–73°. The yield is 17–20 g. (81–95 per cent of the theoretical amount).

2. Notes

1. The benzalacetophenone should be freshly recrystallized from alcohol just before using and should melt at 57°.
2. Owing to the comparatively slight solubility of benzalacetophenone in alcohol, ethyl acetate is used as a solvent during the reduction.
3. If 0.1 g. of catalyst is used the reduction requires about three hours; an increase in catalyst to 0.5 g. causes the reduction to take place in three or four minutes. The exact time depends to a considerable extent on the grade of benzalacetophenone used and it is not certain that the purest will necessarily be reduced in the shortest time.

3. Discussion

Benzylacetophenone can be prepared by the reduction of benzalacetophenone with zinc and acetic acid[1] and catalytically with palladium and hydrogen;[2] by the oxidation of the corresponding carbinol with chromic acid;[3] and by the hydrolysis of ethyl benzylbenzoylacetate.[4]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 61
- Org. Syn. Coll. Vol. 1, 463

References and Notes

1. Schneidewind, Ber. **21**, 1325 (1888); Harries and Hübner, Ann. **296**, 327 (1897).
2. Straus and Grindel, Ann. **439**, 294 (1924).
3. Bauer, Compt. rend. **154**, 1094 (1912).
4. Perkin and Stenhouse, J. Chem. Soc. **59**, 1007 (1891).

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

alcohol (64-17-5)

acetic acid (64-19-7)

ethyl acetate (141-78-6)

hydrogen (1333-74-0)

platinum oxide

Benzalacetophenone (94-41-7)

Benzylacetophenone,
Propiophenone, β-phenyl- (1083-30-3)

platinum (7440-06-4)

zinc (7440-66-6)

palladium (7440-05-3)

chromic acid (7738-94-5)

ethyl benzylbenzoylacetate