



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.

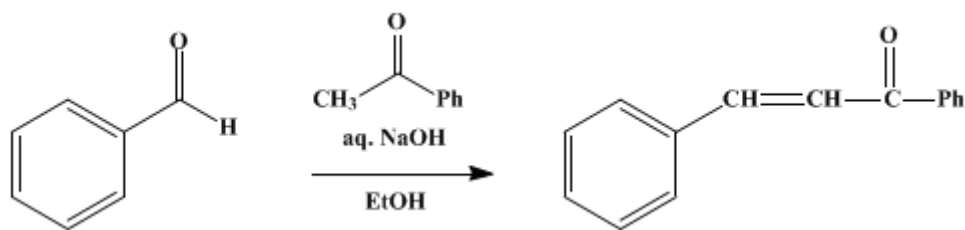
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.78 (1941); Vol. 2, p.1 (1922).

BENZALACETOPHENONE

[Chalcone]



Submitted by E. P. Kohler and H. M. Chadwell.

Checked by H. T. Clarke and R. P. Leavitt.

1. Procedure

A solution of 218 g. (5.5 moles) of [sodium hydroxide](#) in 1960 g. of water and 1000 g. (1225 cc.) of 95 per cent [alcohol](#) are introduced into a 5.5-l. bottle which is loosely covered with a perforated disk of cardboard, supplied with an effective stirrer, and supported in a larger vessel so as to permit cooling with cracked ice. Into the alkaline solution, 520 g. (4.3 moles) of pure [acetophenone](#) ([Note 1](#)) is poured, the bottle is rapidly surrounded with cracked ice, and the stirrer started; 460 g. (4.3 moles) of u. s. p. [benzaldehyde](#) ([Note 2](#)) is then added at once. The temperature of the mixture should not be below 15° and it should not be allowed to rise above 30° during the reaction ([Note 3](#)). If it tends to do so, the stirring is not sufficiently vigorous.

It is advantageous, though not essential, to inoculate the mixture with a little powdered [benzalacetophenone](#) after stirring for one-half hour. After two to three hours, the mixture becomes so thick that the stirring is no longer effective. The stirrer is then removed and the mixture left to itself in an ice box for about ten hours. The mixture now is a thick paste composed of small shot-like grains suspended in an almost colorless liquid. It is cooled in a freezing mixture and then either centrifuged or filtered on a large Büchner funnel, washed with water until the washings are neutral to litmus, and finally washed with 200 cc. of 95 per cent [alcohol](#), which has previously been cooled to 0°. After thorough drying in the air, the crude product weighs about 880 g. (97 per cent of the theoretical amount) and melts at 50–54°. It is sufficiently pure for most purposes but tenaciously holds traces of water. It is most readily purified by recrystallization from four to four and one-half times its weight of 95 per cent [alcohol](#) ([Note 4](#)). Eight hundred and eighty grams of crude product give 770 g. (85 per cent of the theoretical amount) of light yellow material (m. p. 55–57°) and 40–50 g. that require recrystallization.

2. Notes

1. The [acetophenone](#) should be as pure as possible (m. p. 20°). Commercial [acetophenone](#) contains variable quantities of impurities which reduce the yield. By distilling commercial [acetophenone](#) with the help of a good still-head (preferably under diminished pressure) and using only the fraction which boils at 201–202° (76–77° /10 mm.) greater quantities of [benzalacetophenone](#) can be obtained than by using the entire sample.
2. Commercial [benzaldehyde](#) can be used in place of the purer product, but the amount used must be increased to make up for the impurities which are present.
3. If the temperature is too low, or the stirring too slow, the product separates as an oil, which later solidifies in large lumps. If the temperature is allowed to rise above 30°, secondary reactions diminish both the yield and the purity of the product. The most favorable temperature is 25°.
4. In recrystallizing [benzalacetophenone](#), the alcohol should be saturated at 50°. If the solution is saturated above this temperature, the [benzalacetophenone](#) tends to separate as an oil. The solution should be allowed to cool gradually, and should finally be chilled in a freezing mixture.

3. Discussion

Benzalacetophenone can be prepared from benzaldehyde and acetophenone, by the use of either acid¹ or alkaline condensing agents. The alkaline agents are superior and those generally used are a 30 per cent solution of sodium methoxide at low temperatures² and alcoholic sodium hydroxide.³ Preliminary experiments showed that condensation with sodium methoxide requires a long time and gives a product which is difficult to handle in large quantities.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 101
- Org. Syn. Coll. Vol. 1, 205
- Org. Syn. Coll. Vol. 2, 167
- Org. Syn. Coll. Vol. 2, 236
- Org. Syn. Coll. Vol. 2, 498
- Org. Syn. Coll. Vol. 5, 1135

References and Notes

1. Claisen and Claparède, Ber. **14**, 2463 (1881); Straus and Grindel, Ann. **439**, 276 (1924); Breslow and Hauser, J. Am. Chem. Soc. **62**, 2385 (1940).
 2. Claisen, Ber. **20**, 657 (1887).
 3. Kostanecki and Rossbach, Ber. **29**, 1492 (1896); Schlenck and Bergmann, Ann. **463**, 234 (1928).
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Appendix

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

alcohol (64-17-5)

sodium hydroxide (1310-73-2)

Benzalacetophenone,
Chalcone (94-41-7)

benzaldehyde (100-52-7)

Acetophenone (98-86-2)

sodium methoxide (124-41-4)