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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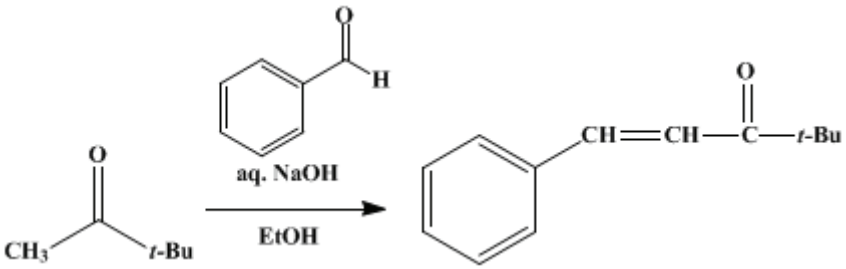
BENZALPINACOLONE

[1-Penten-3-one, 4,4-dimethyl-1-phenyl-]



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1. Procedure

In a 1500-cc. bottle are placed 100 g. (1 mole) of pinacolone (p. 462), 120 g. (1.13 moles) of freshly distilled benzaldehyde, 380 cc. of 95 per cent alcohol, 130 cc. of water, and 100 cc. of 10 per cent sodium hydroxide solution. The bottle is stoppered tightly, placed on a shaking machine, and agitated vigorously for about thirty-two hours (Note 1).

The reaction mixture is poured into a separatory funnel and diluted with an equal volume of water. The benzalpinacolone is separated by extracting three times with 300-cc. portions of benzene. The benzene extracts are combined and washed, first with water until the alkali is entirely removed, then with a saturated solution of sodium bisulfite, and finally two or three times with water. After drying over calcium chloride, the benzene is removed by distilling from a steam bath, and the residue is distilled under reduced pressure. The yield of distilled benzalpinacolone boiling at 143–146° /10 mm. is 165–175 g. (88–93 per cent of the theoretical amount). This product is slightly yellow and melts at 41–42°. It is pure enough for most purposes. It may be recrystallized from 95 per cent alcohol and is then obtained in almost pure white crystals, melting at 43°.

2. Notes

1. The time may be shortened to twenty-four hours without greatly reducing the yield.

3. Discussion

Benzalpinacolone can be prepared by the action of benzaldehyde on pinacolone in the presence of aqueous-alcoholic alkalies.[1] The procedure described is a modification of the method by Vorländer and Kalkow.[1]

References and Notes

1. Vorländer and Kalkow, Ber. **30**, 2269 (1897); Hill, Spear and Lachowicz, J. Am. Chem. Soc. **45**, 1559 (1923).

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

alcohol (64-17-5)

calcium chloride (10043-52-4)

Benzene (71-43-2)

sodium hydroxide (1310-73-2)

sodium bisulfite (7631-90-5)

benzaldehyde (100-52-7)

Benzalpinacolone,
1-Penten-3-one, 4,4-dimethyl-1-phenyl- (538-44-3)

Pinacolone (75-97-8)