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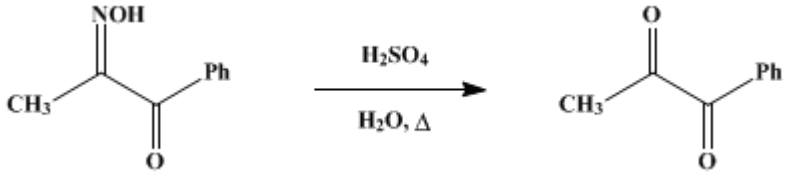
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.023.0001

Organic Syntheses, Coll. Vol. 3, p.20 (1955); Vol. 23, p.1 (1943).

ACETYLBENZOYL

[1,2-Propanedione, 1-phenyl-]



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1. Procedure

In a 1-l. flask arranged for steam distillation (Note 1), 50 g. (0.31 mole) of isonitrosopropiophenone (*Org. Syntheses*, **16**, 44; Coll. Vol. **2**, 363) and 500 g. of 10% sulfuric acid are mixed, and the mixture is distilled with steam until about 2 l. of distillate is collected. During the distillation the flask is heated so that the volume of the reaction mixture is kept roughly constant. The distillation requires about 6 hours, and at the end of this time the liquid in the flask is clear (Note 2).

The lower, yellow layer of diketone in the distillate is separated; the water layer is then saturated with salt and extracted with ether, one 80-ml. and two 25-ml. portions being used for each liter of the aqueous solution. The ether extracts are combined with the diketone proper and dried over sodium sulfate. The ether is removed on the steam bath, and the residual material is distilled from a Claisen flask under reduced pressure. Acetylbenzoyl is collected at 114–116°/20 mm. (Note 3). The yield is 30–32 g. (66–70% of the theoretical amount) (Note 4).

2. Notes

1. A spray trap should be placed between the flask and the condenser; otherwise some isonitrosoketone will be carried over. The checkers used a "Kjeldahl Connecting Bulb, Cylindrical Type," illustrated as item 2020 in the Pyrex Catalog, LP–34, for 1954.
2. On being cooled, the reaction mixture deposits 3–7 g. of the dioxime of acetylbenzoyl, m.p. 234–236° dec.[1]
3. Other boiling points reported for acetylbenzoyl are 216–218°, 164–165°/116 mm., and 102–103°/12 mm.
4. According to the submitters the reaction can be carried out with about the same percentage yields using five times the amounts of material specified above.

3. Discussion

Acetylbenzoyl has been prepared by dehydrogenation of acetylphenylcarbinol over copper at 350°;[2] by oxidation of 1-methyl-2-phenylethylene glycol;[3] by action of amyl nitrite on isonitrosopropiophenone;[4] and by acid hydrolysis of isonitrosopropiophenone[5] or of isonitrosobenzyl methyl ketone.[6] The last-named reaction is reported to furnish quantitative yields of acetylbenzoyl, but the starting material is not easily accessible. Treatment of propiophenone with amyl nitrite (2 moles) without isolating the isonitroso compound has been mentioned as a method of preparing acetylbenzoyl,[4] but the yield of diketone is much better when the isonitroso compound is isolated.[7] Acetylbenzoyl also has been prepared by the oxidation of phenyl acetone with selenium dioxide.[8]

References and Notes

1. Müller and Pechmann, *Ber.*, **22**, 2128 (1889).
2. Mailhe, *Bull. soc. chim. France*, (4) **15**, 326 (1914).
3. Zincke and Zehn, *Ber.*, **43**, 855 (1910).
4. Manasse, *Ber.*, **21**, 2177 (1888).
5. Pechmann and Müller, *Ber.*, **21**, 2119 (1888).
6. Kolb, *Ann.*, **291**, 286 (1896); Borsche, *Ber.*, **40**, 740 (1907).
7. Coles, Manske, and Johnson, *J. Am. Chem. Soc.*, **51**, 2269 (1929).
8. Wegmann and Dahn, *Helv. Chim. Acta*, **29**, 1247 (1946).

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

dioxime of acetylbenzoyl

sulfuric acid (7664-93-9)

ether (60-29-7)

sodium sulfate (7757-82-6)

copper (7440-50-8)

selenium dioxide (7446-08-4)

amyl nitrite (463-04-7)

Propiophenone (93-55-0)

phenyl acetone (103-79-7)

ISONITROSOPROPIOPHENONE

1,2-Propanedione, 1-phenyl- (579-07-7)

1-methyl-2-phenylethylene glycol

Acetylbenzoyl (579-07-7)

acetylphenylcarbinol

isonitrosobenzyl methyl ketone