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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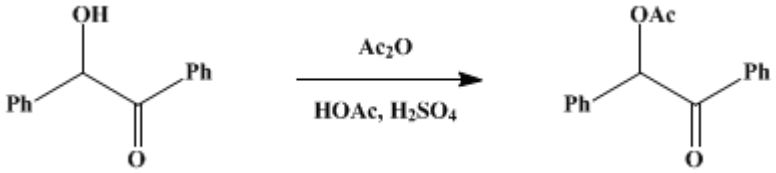
Organic Syntheses, Coll. Vol. 2, p.69 (1943); Vol. 12, p.1 (1932).

BENZOIN ACETATE



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1. Procedure

To a mixture of 212 g. (1 mole) of benzoin (Note 1), 200 cc. of glacial acetic acid, and 200 cc. (2.1 moles) of acetic anhydride, in a 1-l. beaker provided with a mechanical stirrer, is added slowly, with stirring, 20 cc. of concentrated c.p. sulfuric acid. This requires five minutes, during which the benzoin quickly dissolves and the temperature rises to about 50°. The beaker is placed on the steam bath for twenty minutes (Note 2). The mixture is allowed to cool somewhat, transferred to a large dropping funnel, and added *slowly* to 2.5 l. of water vigorously stirred in a 4-l. (1-gal.) crock during thirty minutes (Note 3). Stirring is continued for one hour. The mixture is filtered by suction on a 30-cm. Büchner funnel, and the crystals are sucked as dry as possible and spread on filter paper. After about two hours the crystals are transferred to a 1-l. beaker and warmed to about 60° with 400 cc. of 95 per cent ethyl alcohol. The clear solution is cooled with stirring to 5° and filtered by suction. The air-dried benzoin acetate, melting at 80–82°, weighs 220–230 g. (86 to 90 per cent of the theoretical amount). Another crystallization from 400 cc. of alcohol removes the slight yellow tinge and gives a product melting at 81.5–82.5°, with a loss of about 10 g.

2. Notes

1. The benzoin (Org. Syn. Coll. Vol. I, **1941**, 94) need not be recrystallized.
2. The mixture should not be heated longer or more vigorously.
3. If the product solidifies in lumps, the lumps must be removed, crushed to a paste in a large mortar, and returned to the mixture for stirring.

3. Discussion

The only method of preparative interest is the acetylation of benzoin, either with acetyl chloride[1] or with acetic anhydride.[2] The melting point of benzoin acetate was reported by Zinin as "below 100°," by Jena and Limpricht as 75°, but by later investigators as 82–83°.

References and Notes

1. Zinin, Ann. **104**, 120 (1857); Jena and Limpricht, ibid. **155**, 92 (1870); Päpcke Ber. **21**, 1336 (1888).
2. Francis and Keane, J. Chem. Soc. **99**, 346 (1911).

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

ethyl alcohol,
alcohol (64-17-5)

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

acetic anhydride (108-24-7)

acetyl chloride (75-36-5)

Benzoin (119-53-9)

Benzoin acetate (574-06-1)