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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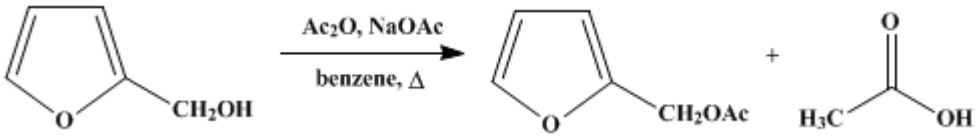
2-FURYLMETHYL ACETATE

[Furfuryl alcohol, acetate]



Submitted by The Miner Laboratories
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1. Procedure

A mixture of 1 l. of benzene, 600 g. (529 cc., 6.1 moles) of 2-furylcarbinol (p. 276) (Note 1), 225 g. (2.7 moles) of fused powdered sodium acetate, and 650 g. (602 cc., 6.4 moles) of a good grade of acetic anhydride are placed in a 5-l. round-bottomed flask (Note 2) fitted with a mechanical stirrer and a reflux condenser provided with a calcium chloride tube. The flask is heated on a steam bath (Note 1) for four hours with stirring to prevent caking of the sodium acetate.

The reaction mixture is allowed to cool and is poured into 4 l. of cold water (Note 3). The upper layer is separated and allowed to stand for two hours over about 500 cc. of 5 per cent sodium carbonate solution with frequent shaking or mechanical stirring. This decomposes any excess of acetic anhydride. It is finally washed with about 3 l. of water. The benzene solution is distilled under ordinary pressure to remove the benzene (800–900 cc. is recovered). The 2-furylmethyl acetate is distilled under diminished pressure, b.p. 69–70°/7 mm. The yield is 750–800 g. (87–93 per cent of the theoretical amount) (Note 4).

2. Notes

1. If the water-insoluble form of the alcohol (p. 278) is used, the mixture should be allowed to stand under a reflux condenser for about two hours before it is heated. During this period a certain amount of heat is evolved, and sometimes it is sufficient to cause the benzene to boil vigorously, thus making cooling necessary. After it has stood the required time, the mixture is refluxed as described above.
2. If a two- or three-necked flask is available it can be used to advantage.
3. The washing can best be done in a large separatory apparatus prepared by cutting the bottom out of an 18-l. (5-gallon) glass carboy and wiring into its neck a piece of glass tubing carrying a rubber tube and pinch clamp. The inverted carboy forms a useful separatory funnel of large capacity.
4. Saponification of the product with standard potassium hydroxide shows 93–94 per cent ester. It contains some furfuryl alcohol, the removal of which by fractional distillation is difficult, because the boiling points of the alcohol (169°/752 mm.) and the ester (175°/764 mm.) are so close together. Furfuryl acetate darkens on standing. It may be redistilled with little loss to give an almost colorless product.

3. Discussion

2-Furylmethyl acetate can be prepared by heating 2-furylcarbinol with acetic anhydride alone, or with acetic anhydride and sodium acetate.[1]

References and Notes

1. Wissell and Tollens, Ann. **272**, 303 (1892); Zanetti, J. Am. Chem. Soc. **47**, 535 (1925).

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

Benzene (71-43-2)

acetic anhydride (108-24-7)

sodium acetate (127-09-3)

sodium carbonate (497-19-8)

potassium hydroxide (1310-58-3)

2-FURYLCARBINOL,
Furfuryl alcohol (98-00-0)

2-Furylmethyl acetate

Furfuryl alcohol, acetate,
Furfuryl acetate (623-17-6)