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.

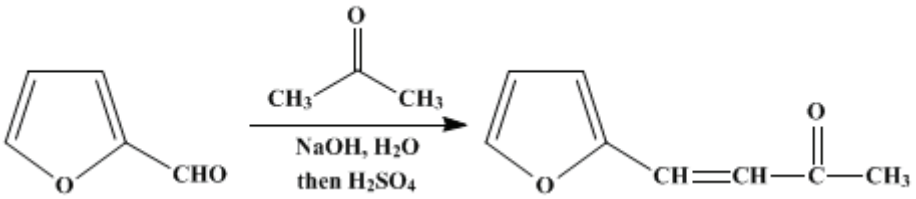
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.007.0042

Organic Syntheses, Coll. Vol. 1, p.283 (1941); Vol. 7, p.42 (1927).

2-FURFURALACETONE

[3-Buten-2-one, 4-(2-furyl)-]



Submitted by G. J. Leuck and L. Cejka.
Checked by Frank C. Whitmore and H. H. Rowley.

1. Procedure

In a 5-l. round-bottomed flask equipped with a mechanical stirrer are mixed 385 g. (335 cc., 3.8 moles) of 95 per cent furfural (p. 280) (Note 1) and 3 l. of water. Then 500 g. (630 cc., 8.6 moles) of acetone (Note 1) is added. The mixture is stirred and cooled to 10° and to it is added 75 cc. of 33 per cent sodium hydroxide solution, whereupon some heat is generated. Without cooling, the stirring is continued for four hours. At the end of this time 10 per cent sulfuric acid is added until the mixture is acid to litmus (about 350 cc.) (Note 2). The two layers which have formed are separated and the upper aqueous layer is distilled (Note 3) under ordinary pressure until the distillate no longer forms two layers.

The bottom layer of this distillate is added to the original lower layer and distilled under reduced pressure from a 1-l. modified Claisen flask (p. 130) provided with an air condenser and heated in an oil bath. The receiving flask is placed in a large funnel connected with the drain. A stream of cold water is run over the receiver. When a solid appears in the receiver, distillation is interrupted, the liquid distillate is discarded, and the distillation is continued. The product which distils at 114–118°/10 mm. (135–145°/50 mm.) (Note 4) weighs 310–340 g. (60–66 per cent of the theoretical amount, based on the furfural used). The yellow crystals melt at 37–39° and when melted show a sharp freezing point of 37° (Note 5).

2. Notes

1. Commercial chemicals were used. The use of purer chemicals, including furfural distilled over a 2° range under diminished pressure, gives no higher yield, but the product is lighter colored, although the boiling point and melting point are the same as with the crude materials.
Some commercial grades of acetone have a markedly deleterious effect on the yield of furfuralacetone (F. N. Peters, private communication). Accordingly, it is recommended that acetone of reasonably high purity be used.
2. When the alkali is neutralized, the mixture loses its milky appearance and forms definite layers.
3. The distillation of the water layer may be omitted, since it yields only 10–20 g. of the product.
4. A large residue of higher-boiling material remains in the flask. This residue contains much difurfuralacetone, the formation of which takes place to a considerable extent in spite of the large excess of acetone used.
5. The highest melting point recorded in the literature is 39–40°. The crystals gradually become reddish on standing even in the dark. This change is much slower when freshly distilled furfural is used.

3. Discussion

2-Furfuralacetone can be prepared by the condensation of furfural with acetone in the presence of bases.[1]

References and Notes

1. Schmidt, Ber. **14**, 574, 1459 (1881); Claisen, Ber. **14**, 2468 (1881); Claisen and Ponder, Ann. **223**, 137 (1884).

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

sulfuric acid (7664-93-9)

sodium hydroxide (1310-73-2)

acetone (67-64-1)

Furfural (98-01-1)

2-Furfuralacetone

3-Buten-2-one, 4-(2-furyl)- (623-15-4)

furfuralacetone

difurfuralacetone