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.

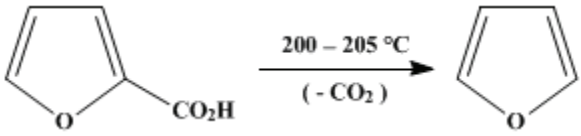
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.007.0040

Organic Syntheses, Coll. Vol. 1, p.274 (1941); Vol. 7, p.40 (1927).

FURAN



Submitted by W. C. Wilson
Checked by Roger Adams and C. G. Gauerke.

1. Procedure

In a 200-cc. round-bottomed flask is placed 80 g. (0.68 mole) of crude 2-furancarboxylic acid (p. 276) (usually about 95 per cent pure). To the neck of the flask is fitted an upright tube 2.5 cm. in diameter and 15 cm. long, with a side arm of the same diameter leading out about 2 cm. from the top of the tube. This side arm is extended into the bottom of an efficient (25-cm.) soda-lime tower (Note 1) immersed in a water bath held at 40° to prevent condensation of the furan. From the top of the soda-lime tower an outlet tube (0.5 cm. in diameter) is extended to the top of an upright water condenser, to the lower end of which is attached a receiving flask surrounded by ice and salt (Note 2).

The top of the upright tube in the reaction flask is closed by a cork stopper holding a glass plunger which may be used for pushing back into the flask any sublimed 2-furancarboxylic acid.

The acid is heated just to its boiling point (200–205°) (Note 3), when it decomposes with the evolution of furan and carbon dioxide. The small amounts of 2-furancarboxylic acid that sublime are pushed back from time to time. The distillate is finally redistilled, when it is found to boil at 31–34°/745 mm. The yield is 33–36 g. (72–78 per cent of the theoretical amount based on 2-furancarboxylic acid of 95 per cent purity) (Note 4).

2. Notes

1. The drying column removes carbon dioxide, moisture, and some of the other by-products which are formed. The product which is collected is nearly pure, and the redistillation completes the purification.
2. In a number of experiments a second receiver was used in addition to the first one, but it was found that practically all of the furan condensed in the first receiver.
3. Care should be taken in heating 2-furancarboxylic acid. If the temperature is too low, decomposition takes place too slowly; if too high, much 2-furancarboxylic acid sublimes and causes difficulty. In one run a thermometer was placed in the melted acid. It read 200–205° during the evolution of the furan.
4. In the preparation of considerable quantities of furan it is desirable to pass the gas through one or two wash bottles of potassium hydroxide before it reaches the soda-lime tower. The heat of absorption of carbon dioxide keeps these bottles warm enough to prevent condensation of furan. Also, the substitution of a 120–150 cm. column for the 15-cm. column allows greater volume and speed. By these modifications 200–300 g. of furan can be prepared per hour without difficulty (F. N. Peters, private communication).

3. Discussion

Furan can be prepared by heating 2-furancarboxylic acid in a sealed tube;[1] by the dry distillation of barium 2-furancarboxylate;[1] and by heating barium 2-furancarboxylate with soda-lime.[2] Considerably improved yields have been reported to result from heating 2-furancarboxylic acid with a small amount of copper sulfate or copper oxide in a high-boiling coal-tar base[3] or in quinoline.[4] Furan has also been prepared from furfural by passing it over hot soda-lime or dropping it into a fused mixture of sodium and potassium hydroxides.[5]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 566

References and Notes

1. Freundler, Compt. rend. **124**, 1157 (1897); Bull. soc. chim. (3) **17**, 613 (1897).
2. Limpricht, Ann. **165**, 281 (1873).
3. Gilman and Lousinian, Rec. trav. chim. **52**, 156 (1933).
4. Wagner and Simons, J. Chem. Education **13**, 270 (1936).
5. Hurd, Goldsby, and Osborne, J. Am. Chem. Soc. **54**, 2532 (1932).

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

soda-lime

sodium and potassium hydroxides

copper sulfate (7758-98-7)

carbon dioxide (124-38-9)

Furan (110-00-9)

2-furancarboxylic acid (88-14-2)

barium 2-furancarboxylate

copper oxide (1317-38-0)

Quinoline (91-22-5)

Furfural (98-01-1)