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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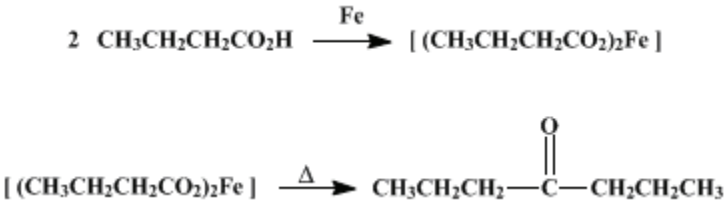
# 4-HEPTANONE



Submitted by Robert Davis, Charles Granito, and Harry P. Schultz[1].
Checked by William G. Dauben and Richard J. Shavitz.

## 1. Procedure

A mixture of 370 ml. (4 moles) of *n*-butyric acid (Note 1) and 123 g. (2.2 moles) of hydrogen-reduced iron powder (Note 2) is refluxed for 5 hours in a 1-l. flask equipped with a condenser (Note 3). The apparatus is converted for downward distillation while an atmosphere of nitrogen is maintained. The nitrogen sweep is then stopped, the flask is strongly heated, and the entire distillate collected.

The crude product is washed with two 20-ml. portions of 10% sodium hydroxide solution and with one 20-ml. portion of water. The 4-heptanone is dried over 5 g. of anhydrous sodium sulfate, filtered, and distilled. The yield of 4-heptanone, b.p. 142–144°, $n^{25}$D 1.4031–1.4036, is 157–171 g. (69–75%).

## 2. Notes

1. *n*-Butyric acid, b.p. 162–164°, from Eastman Organic Chemicals was redistilled before use.
2. Hydrogen-reduced iron powder from Fisher Scientific Company was used.
3. Severe foaming may force brief cessations of heating during the first hour. Boric acid (0.1 g.) somewhat diminishes the extent of foaming.

## 3. Discussion

The present procedure is that of Davis and Schultz.[2] 4-Heptanone has also been synthesized by virtually every general method known and listed for ketones in "Chemistry of Carbon Compounds," including liquid or vapor phase decarboxylation of *n*-butyric acid or its salts, oxidation of 4-heptanol, and hydration of 3-heptyne.[3]

## 4. Merits of the Preparation

This method is illustrative of a general method of preparing simple ketones from normal aliphatic carboxylic acids. It is especially useful because the starting materials are easily accessible, the yields good, and the procedure very simple.

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## References and Notes

1. Chemistry Department, University of Miami, Coral Gables, Florida 33124.
2. R. Davis and H. P. Schultz, *J. Org. Chem.*, **27**, 854 (1962).
3. J. G. Buchanan, N. A. Hughes, F. J. McQuillin, and G. A. Swan in S. Coffey, "Rodd's Chemistry of Carbon Compounds," Elsevier Publishing Company, New York, 1965, p. 53.

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## Appendix

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

sodium hydroxide (1310-73-2)

iron powder (7439-89-6)

sodium sulfate (7757-82-6)

nitrogen (7727-37-9)

n-butyric acid (107-92-6)

boric acid (10043-35-3)

4-Heptanone (123-19-3)

4-heptanol (589-55-9)

3-heptyne (2586-89-2)