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.

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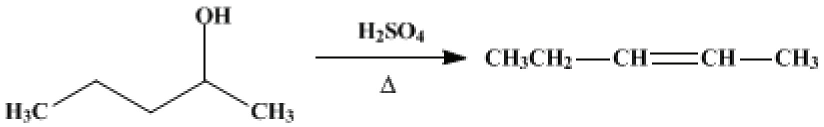
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.007.0076

Organic Syntheses, Coll. Vol. 1, p.430 (1941); Vol. 7, p.76 (1927).

2-PENTENE



Submitted by James F. Norris
Checked by Frank C. Whitmore

1. Procedure

To a cooled mixture of 200 cc. of water and 200 cc. of concentrated sulfuric acid in a 1-l. round-bottomed flask is added slowly 176 g. (214 cc., 2 moles) of pentanol-2 (b.p. 118–120°) (Note 1). The flask is connected with a long efficient condenser for distillation (Note 2). The end of the condenser is provided with an adapter which passes well into a receiver surrounded by ice water (Note 2). The flask is heated on a boiling-water bath as long as distillation takes place (two to three hours).

The distillate is shaken in the receiver with about 25 cc. of a 5 per cent solution of sodium hydroxide to remove any trace of sulfur dioxide that may be present. The hydrocarbon is separated and dried with 10 g. of anhydrous calcium chloride. It is then distilled, the fraction boiling at 35–41° (Note 3) being collected. The yield is 92–112 g. (65–80 per cent of the theoretical amount).

2. Notes

1. Pentanol-2 is now manufactured on a large scale from petroleum. As the quality of the commercial alcohol varies, the product should be distilled before use and the proper fraction taken. The material was kindly supplied by the Petroleum Chemical Corporation, Cambridge, Mass. (A. D. Little, Inc.).
2. Because of the volatility of the product, unusual precautions must be taken in its preparation to avoid loss.
3. Most of the product distils at 36–37°. The product may contain a small amount of the isomeric pentene-1. By redistilling it through a 25-cm. fractionating column containing small pieces of glass rod, the pure hydrocarbon can be readily obtained. Pentene-2 boils at 36.39° ± 0.04°/760 mm., melts at −138° ± 2°, has the density $d_{4°}^{15°}$ 0.6555 and n_D^{25} 1.3839.

3. Discussion

Pentene-2 can be prepared by the action of alcoholic potash on 3-iodopentane[1] or 3-bromopentane;[2] by dehydration of pentanol-2,[3] or pentanol-3;[4] by pyrolysis of the acetate of pentanol-3;[5] and by the catalytic reduction of pentine-2.[6]

References and Notes

1. Wagner and Saytzev, Ann. **175**, 373 (1875).
2. Lucas and Moyse, J. Am. Chem. Soc. **47**, 1461 (1925); Sherrill, Otto, and Pickett, ibid. **51**, 3028 (1929).
3. Tissier, Bull. soc. chim. (3) **9**, 100 (1893); Sherrill, Baldwin, and Haas, J. Am. Chem. Soc. **51**, 3037 (1929).
4. Sherrill, Otto, and Pickett, J. Am. Chem. Soc. **51**, 3028 (1929); Leendertse, Tulleners, and Waterman, Rec. trav. chim. **53**, 715 (1934).
5. Wibaut and van Pelt, Jr., ibid. **57**, 1057 (1938).
6. Bourguel, Bull. soc. chim. (4) **41**, 1475 (1927).

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

alcoholic potash

acetate of pentanol-3

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

sodium hydroxide (1310-73-2)

sulfur dioxide (7446-09-5)

2-Pentene,
Pentene-2 (646-04-8)

pentanol-2 (6032-29-7)

pentene-1 (109-67-1)

3-iodopentane (1809-05-8)

3-bromopentane (1809-10-5)

pentanol-3 (584-02-1)

pentine-2 (627-21-4)