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.

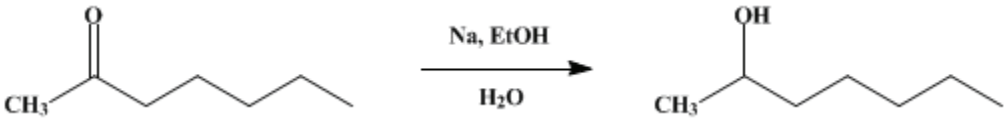
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.010.0060

Organic Syntheses, Coll. Vol. 2, p.317 (1943); Vol. 10, p.60 (1930).

2-HEPTANOL



Submitted by Frank C. Whitmore and T. Otterbacher.
Checked by Henry Gilman and H. J. Harwood.

1. Procedure

In a 3-l. round-bottomed flask, fitted with an efficient Liebig condenser (100 by 1 cm.), 228 g. (2 moles) of methyl *n*-amyl ketone (Org. Syn. Coll. Vol. I, **1941**, 351) is dissolved in a mixture of 600 cc. of 95 per cent alcohol and 200 cc. of water. One hundred thirty grams (5.6 gram atoms) of sodium in the form of wire is gradually added through the condenser. During the addition of the sodium the flask is cooled with running water (Note 1) so that the reaction does not become unduly violent (Note 2).

When the sodium has dissolved (Note 3), 2 l. of water is added and the mixture is cooled to 15°. The upper oily layer is then separated, washed with 50 cc. of 1:1 hydrochloric acid and then with 50 cc. of water, dried over 20 g. of anhydrous sodium sulfate, and distilled with a fractionating column (Note 4). After a small fore-run of low-boiling liquid, the pure heptanol distils at 155–157.5°. The yield is 145–150 g. (62–65 per cent of the theoretical amount).

2. Notes

1. If no cooling is used, condensation products are formed and the yield of heptanol is reduced considerably.
2. The temperature can be held conveniently below 30° by cooling with ice. Such cooling when accompanied by stirring is particularly helpful during the early addition of the sodium (either as wire or in small pieces). With cooling and stirring, very little refluxing takes place and after the addition of about 60 g. of sodium the reaction slows down to such an extent that large amounts of sodium can be added at once without danger of excessive heating.
3. The time required for addition of the sodium may be significantly decreased by the use of mechanical stirring. Although the yield is not increased appreciably by stirring, frothing is prevented and for this reason the sodium may be added more rapidly.
4. The submitters used a Young column with 20 disks 3 cm. apart. The checkers used a Glinsky three-bulbed column.

3. Discussion

2-Heptanol has been prepared by the action of *n*-amylmagnesium bromide on acetaldehyde,[1] and by the reduction of methyl *n*-amyl ketone in alcoholic solution by means of sodium.[2]

References and Notes

1. Henry, Rec. trav. chim. **28**, 446 (1909).
2. Thoms and Mannich, Ber. **36**, 2544 (1903); Pickard and Kenyon, J. Chem. Soc. **99**, 58 (1911).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

alcohol (64-17-5)

acetaldehyde (75-07-0)

hydrochloric acid (7647-01-0)

sodium sulfate (7757-82-6)

sodium (13966-32-0)

heptanol (111-70-6)

n-amylmagnesium bromide (693-25-4)

METHYL n-AMYL KETONE (110-43-0)

2-Heptanol (543-49-7)