



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.

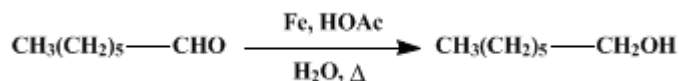
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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n-HEPTYL ALCOHOL



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1. Procedure

In a 12-l. flask fitted with a mechanical stirrer and a short reflux condenser are placed 1800 g. (32.2 atoms) of [iron filings](#) ([Note 1](#)), 3 l. (52.5 moles) of glacial [acetic acid](#), 3 l. of water, and 450 g. (3.95 moles) of [heptaldehyde](#) ([Note 2](#)). The mixture is heated on the steam bath, with stirring, for six to seven hours ([Note 3](#)). The flask is then fitted to an apparatus for steam distillation ([p. 479](#)) and the mixture distilled in a current of steam ([Note 4](#)) until no more oil passes over (7–8 l. of distillate). The oil is then separated, and the aqueous part distilled to recover a small quantity of dissolved or suspended [heptyl alcohol](#).

The combined product is mixed with 1 l. of 20 per cent [sodium hydroxide](#) solution and stirred on the steam bath for four hours, in order to hydrolyze a small proportion of [heptyl acetate](#). The oil is then separated and distilled, and the portion boiling at 172–176° is collected. The residue in the flask is mixed with about 100 cc. of water and distilled, whereupon a further small quantity of oil passes over with the steam. This distillate and the forerun are freed of the bulk of the water in a separatory funnel and distilled from a smaller flask. In this way the total yield of *n*-[heptyl alcohol](#) boiling at 172–176° (uncorr.) is 350–370 g. (75–81 per cent of the theoretical amount). The alcohol may be redistilled under reduced pressure, when it passes over almost without loss at 71–72°/12 mm.

2. Notes

1. The [iron filings](#) used were free from grease and oil. Ninety-five per cent passed an 80-mesh screen, and 60 per cent passed a 100-mesh screen. Certain lots of [iron filings](#) are not satisfactory for this reduction. Less difficulty is encountered with [iron powder](#) reduced with [hydrogen](#) than [iron](#) obtained in any other way, but some lots of [iron](#) reduced with [hydrogen](#) are not effective. Ruhoff and Reid¹ observed that the addition of a solution of [nickel chloride hexahydrate](#) will cause the reduction of [heptaldehyde](#) to start at once and will greatly increase the rate of reduction.

It is suggested, therefore, that, with a run of the size described above, a solution of 20 g. of [nickel chloride hexahydrate](#) in 50 cc. of water be added to the reaction mixture immediately after the addition of the aldehyde. If this is done, the reduction starts at once and is complete in two hours instead of the usual six to seven hours.

It is also recommended that the reaction mixture be divided between two 12-l. flasks and that 3 l. of water be added to each flask immediately at the end of the reaction. This prevents the reaction mixture from setting to a hard mass if the steam distillation is not carried out at once, and it also reduces the amount of foaming. (C. R. Noller and R. Bannerot, private communication.)

2. The [heptaldehyde](#) should be a freshly distilled product, boiling at 154–156°.

3. The reaction may be successfully carried out without a stirrer, but the time of heating must then be lengthened to twelve to fifteen hours.

4. It is advisable to begin the steam distillation at once, while the mixture is hot, and to avoid the use of a free flame, which might cause breakage of the flask. If foaming, or too great a volume, hinders a rapid distillation, it is well to decant part of the contents of the flask and to conduct the steam distillation in two portions. (Compare [Note 1](#).)

3. Discussion

[Heptyl alcohol](#) can be prepared by the reduction of [heptaldehyde](#) with [zinc](#) dust and [acetic acid](#),²

sodium amalgam and acetic acid,³ sodium in toluene and acetic acid,⁴ and hydrogen and a catalyst.⁵ Other methods include the reaction between pentane and ethylene oxide in the presence of anhydrous aluminum bromide,⁶ and the action of amylmagnesium bromide on ethylene oxide.⁷ The procedure described is based on a method by Hill and Nason for the reduction of cinnamaldehyde.⁸

References and Notes

1. Ruhoff and Reid, J. Am. Chem. Soc. **55**, 3827 (1933).
 2. Bouis and Carlet, Ann. **124**, 352 (1862); Krafft, Ber. **16**, 1723 (1883).
 3. Schorlemmer, Ann. **177**, 303 (1875); Cross, Ann. **189**, 2 (1877); Jourdan, Ann. **200**, 102 (1880).
 4. Levene and Taylor, J. Biol. Chem. **35**, 281 (1918).
 5. Adams and Carothers, J. Am. Chem. Soc. **45**, 1076 (1923); Bryusova and Kore, J. Applied Chem. (U.S.S.R.) **12**, 1457 (1939) [C. A. **34**, 6232 (1940)].
 6. I. G. Farbenind. A.-G., Fr. pat. 716,604 [C. A. **26**, 2198 (1932)].
 7. Vaughn, Spahr and Nieuwland, J. Am. Chem. Soc. **55**, 4207 (1933).
 8. Hill and Nason, *ibid.* **46**, 2236 (1924).
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Appendix

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

acetic acid (64-19-7)

hydrogen (1333-74-0)

sodium hydroxide (1310-73-2)

iron,
iron filings,
iron powder (7439-89-6)

toluene (108-88-3)

zinc (7440-66-6)

sodium (13966-32-0)

Ethylene oxide (75-21-8)

Heptyl alcohol,
n-HEPTYL ALCOHOL (111-70-6)

heptyl acetate (112-06-1)

nickel chloride hexahydrate (7791-20-0)

Pentane (109-66-0)

aluminum bromide

amylmagnesium bromide (693-25-4)

cinnamaldehyde

heptaldehyde (111-71-7)

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