

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 accessed text can be free 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.

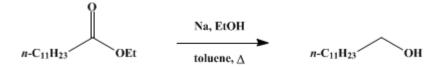
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

Organic Syntheses, Coll. Vol. 2, p.372 (1943); Vol. 10, p.62 (1930).

LAURYL ALCOHOL

[Dodecyl alcohol]



Submitted by S. G. Ford and C. S. Marvel. Checked by Frank C. Whitmore and D. J. Loder.

1. Procedure

The central neck of a 5-l. three-necked round-bottomed flask is fitted with a stopper carrying a mercury-sealed mechanical stirrer. One of the side necks is connected by means of a short piece of heavy rubber tubing to a large reflux condenser about 2 m. long, with an inner tube 2.5 cm. in diameter (Note 1). The third neck is fitted with a separatory funnel.

In the flask are placed 70 g. (3 moles) of sodium and 200 cc. of dry toluene (Note 2). The flask is heated in an oil bath until the sodium is melted. The stirrer is then started; when the sodium is finely divided, the oil bath is removed and the mixture allowed to cool. Stirring must be continued during the cooling in order to keep the sodium finely divided.

When the mixture has cooled to about 60°, there are added from the separatory funnel, first, a solution of 114 g. (0.5 mole) of ethyl laurate (Note 3) in 150 cc. of absolute alcohol (Note 4), then 500 cc. more of alcohol, as rapidly as is possible (Note 5) without loss of material through the condenser. The time required for the addition of the ester solution and the alcohol is less than five minutes, usually two or three minutes. When the reaction has subsided, the flask is heated on a steam bath until the sodium is completely dissolved (Note 6). The mixture is then steam-distilled to remove the toluene and ethyl alcohol. The contents of the flask are transferred to a separatory funnel while still hot and washed three times with 200-cc. portions of hot water to remove the sodium laurate (Note 7). The lauryl alcohol is extracted with ether from the cooled mixture and the washings. The combined ether extracts are washed with water, sodium carbonate solution, and again with water, and dried over anhydrous magnesium sulfate. The ether is evaporated and the lauryl alcohol distilled under diminished pressure. The yield is 60–70 g. (65–75 per cent of the theoretical amount) of a product boiling at 143–146°/18 mm, or 198–200°/135 mm. (Note 8).

2. Notes

- 1. The reaction is very vigorous and, unless the condenser has a wide bore, finely divided sodium may be forced out the top and bad fires may result. The inner tube of the condenser may advantageously be made of brass or copper.
- 2. The toluene is dried by distillation; the first 10 per cent is discarded and the remainder is stored over sodium until used.
- 3. The ethyl laurate used was prepared by the alcoholysis of cocoanut oil and fractionation of the resulting esters. The material boiled at 127–132°/5 mm. See Org. Syn. 20, 69, and Organic Chemical Reagents III, Univ. Illinois Bull. 19 (6) 62 (1921).
- 4. The grade of absolute alcohol used in the reduction is very important. Alcohol dried with magnesium methoxide (Org. Syn. Coll. Vol. I, **1941**, 249) was used in this preparation. Alcohol dried over lime gives very low yields.
- 5. The best yields are obtained when the reductions are carried out rapidly. If the reaction seems to be about to get out of control, the stirrer is stopped and the mixture is cooled with an ice pack.
- 6. When several reductions are being made, time is saved by transferring the mixture at this point to another flask, thus having the original apparatus ready for another reduction.

- 7. Unless the sodium laurate is carefully removed, it causes trouble some emulsions.
- 8. Ethyl undecylenate has been reduced to undecylenyl alcohol (b.p. 123–125°/6 mm.) in 70 per cent yields; ethyl myristate to myristyl alcohol (b.p. 170–173°/20 mm.; m.p. 39–39.5°) in 70–80 per cent yields; ethyl palmitate to cetyl alcohol (b.p. 178–182°/12 mm.; m.p. 48.5–49.5°) in 70–78 per cent yields by this same procedure.

3. Discussion

Lauryl alcohol has been prepared by the reduction of the aldehyde;¹ by the reduction of esters of lauric acid with sodium and absolute alcohol² or with sodium, liquid ammonia and absolute alcohol,³ or catalytically;⁴ and by the reduction of lauramide with sodium and amyl alcohol.⁵ The method in the above procedure is essentially that described by Levene and Allen.²

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 246
- Org. Syn. Coll. Vol. 2, 468

References and Notes

- **1.** Krafft, Ber. **16**, 1718 (1883); Sivkov and Novikova, J. Applied Chem. (U.S.S.R.) **13**, 1272 (1940) [C. A. **35**, 2110 (1941)].
- Bouveault and Blanc, Bull. soc. chim. (3) 31, 674 (1904); Ger. pat. 164,294 [Frdl. 8, 1260 (1905–07)]; Levene and Allen, J. Biol. Chem. 27, 443 (1916); Marvel and Tanenbaum, J. Am. Chem. Soc. 44, 2649 (1922); Adams and Marvel, Org. Chem. Reagents IV, Univ. Illinois Bull. 20 (8) 54 (1922).
- 3. Chablay, Compt. rend. 156, 1021 (1913); Ann. chim. (9) 8, 215 (1917).
- **4.** Adkins and Folkers, J. Am. Chem. Soc. **53**, 1095 (1931); Böhme A.-G., Brit. pat. 356,606 [C. A. **26**, 5573 (1932)].
- 5. Scheuble and Loebl, Monatsh. 25, 348 (1904).

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

ethyl alcohol, alcohol (64-17-5)

ammonia (7664-41-7)

ether (60-29-7)

sodium carbonate (497-19-8)

Lauryl alcohol, dodecyl alcohol (112-53-8)

copper (7440-50-8)

toluene (108-88-3)

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sodium (13966-32-0)

magnesium methoxide

amyl alcohol (71-41-0)

magnesium sulfate (7487-88-9)

Cetyl alcohol (36653-82-4)

ethyl laurate (106-33-2)

sodium laurate (629-25-4)

Ethyl undecylenate

Undecylenyl alcohol

ethyl myristate (124-06-1)

Myristyl alcohol (112-72-1)

ethyl palmitate (628-97-7)

esters of lauric acid (143-07-7)

lauramide (1120-16-7)
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