



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.

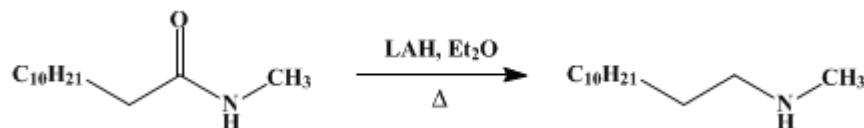
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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LAURYL METHYLAMINE

[Dodecylamine, N-methyl-]



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Checked by N. J. Leonard and C. W. Schimelpfenig.

1. Procedure

In a 5-l. three-necked flask, fitted with a ball-joint sealed stirrer and a Soxhlet extractor (70 mm. internal diameter \times 300 mm. length of body) carrying a large-capacity condenser, are placed 1.8 l. of dry ether (Note 1) and 38 g. (1 mole) of finely divided lithium aluminum hydride (Note 2). In the Soxhlet cup is placed 160 g. (0.75 mole) (Note 3) of N-methyl lauramide (m.p. 67–69°) (Note 4). The mixture is heated under gentle reflux with efficient stirring over a 5-hour period and then stirred overnight at room temperature (Note 5); the N-methyl lauramide is extracted from the cup during the first 3 hours.

The excess lithium aluminum hydride and the metallic complexes are decomposed by the careful addition of 82 ml. of distilled water, from a dropping funnel, to the well-stirred mixture. The reaction mixture is stirred for an additional 30 minutes, filtered with suction, and the solid is washed with several 100-ml. portions of ether. After the ether is removed from the filtrates, the residual oil is distilled under reduced pressure. The yield of lauryl methylamine, a colorless liquid boiling at 110–115°/1.2–1.5 mm., is 121–142 g. (81–95%) (Note 6).

2. Notes

1. It is preferable to use ether subjected to final drying by distillation from lithium aluminum hydride.
2. The yield depends upon the use of high-quality, fresh lithium aluminum hydride.
3. If a Soxhlet extractor having a smaller capacity is employed, the cup will have to be recharged during the course of the reaction.
4. N-Methyl lauramide, N-methyl myristamide, and N-methyl pelargonamide can be prepared in 95–98% yield by adaptation of the method used by Roe, Scanlan, and Swern² for the preparation of amides of oleic and 9,10-dihydroxystearic acids.
5. Stirring overnight is a matter of convenience. In the preparation of methyl nonylamine, refluxing for an hour after the addition of the amide was found by the submitters to be sufficient.
6. Methyl nonylamine and methyl myristylamine were prepared by the submitters in 89–92% yield using the same procedure; with methyl myristylamine a longer reflux period was required, owing to the lower solubility of the amide.

3. Discussion

Lauryl methylamine has been prepared by the reaction of lauryl alcohol with methylamine under pressure in the presence of catalysts at high temperature,³ by heating lauryl chloride with methylamine in alcoholic or aqueous medium under pressure,^{4,5,6} by the reaction of lauryl halides with aqueous methylamine,⁷ by the hydrolysis of *o*-carboxy-N-methyl-N-laurylbenzenesulfonamide,⁸ and by the pyrolysis of β -cyanoethyl lauryl methylamine.⁹ Cetyl methylamine has been prepared by the catalytic debenzoylation of benzylcetyl methylamine.¹⁰

References and Notes

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

ether (60-29-7)

Lauryl alcohol (112-53-8)

methylamine (74-89-5)

lithium aluminum hydride (16853-85-3)

Laurylmethylamine,
Dodecylamine, N-methyl- (7311-30-0)

N-methylauramide (27563-67-3)

N-methylpelargonamide

methylnonylamine (39093-27-1)

methylmyristylamine

lauryl chloride (112-52-7)

β -cyanoethylaurylmethylamine

Cetylmethylamine

benzylcetylmethylamine

o-carboxy-N-methyl-N-laurylbenzenesulfonamide

N-methylmyristamide (7438-09-7)