



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.

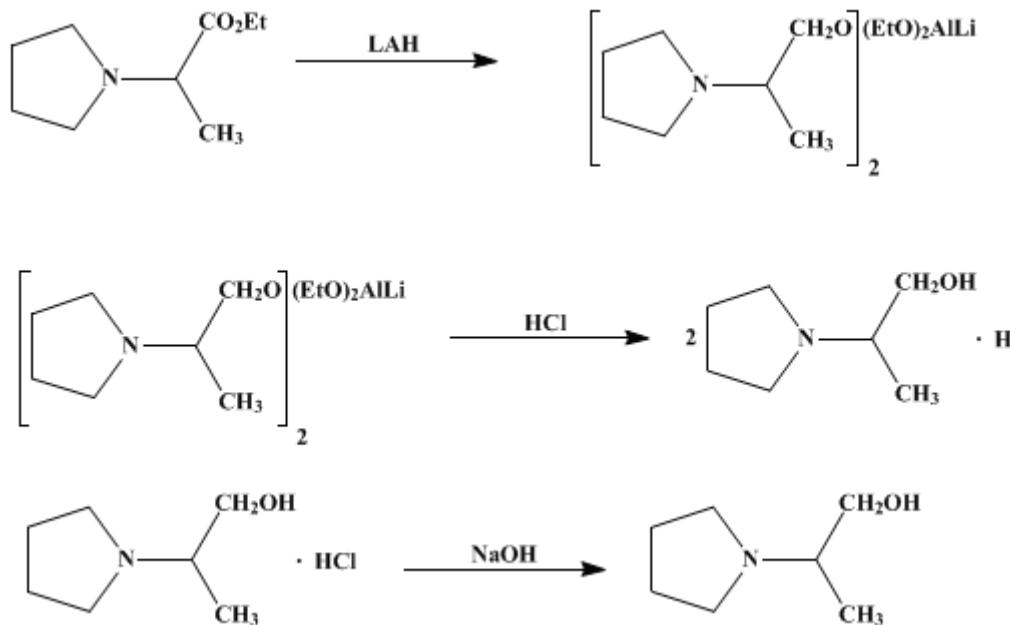
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.834 (1963); Vol. 33, p.82 (1953).

2-(1-PYRROLIDYL)PROPANOL

[1-Pyrrolidineethanol, β -methyl-]



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1. Procedure

A 2-l. three-necked round-bottomed flask in an electric heating mantle is fitted with a mercury-sealed Hershberg stirrer, a dropping funnel, and an efficient reflux condenser topped with a tube containing soda lime and calcium chloride. In this flask are placed 21.3 g. (0.56 mole) of pulverized lithium aluminum hydride (Note 1) and 300 ml. of dry ether. The mixture is heated under reflux until most of the hydride has dissolved. A solution of 157.3 g. (0.92 mole) of ethyl α -(1-pyrrolidyl)propionate (p.466) in 200 ml. of dry ether is then added slowly with vigorous stirring at such a rate that the solvent refluxes gently. When the addition is complete and the initial reaction subsides, the mixture is stirred at the reflux temperature an additional 30 minutes. The excess lithium aluminum hydride is decomposed by adding 50 ml. of ethyl acetate slowly with stirring. This is followed by 600 ml. of 6*N* hydrochloric acid, added slowly with vigorous stirring.

The mixture is transferred to a separatory funnel. The water layer is separated, washed once with ether, and made strongly alkaline by the addition of 1 l. of 6*N* sodium hydroxide (Note 2). The mixture is returned to the original 2-l. three-necked flask. An attachment for continuous ether extraction is placed in one of the side necks,² and 6-in. extension columns are placed in the other two necks. The stirrer is fitted through the center column and neck. Ether is added through the column attached to the side neck until the proper ether level is attained. A stopper is then placed on this column. The stirrer is run at such a rate that gentle swirling is accomplished without hindering the separation of the ether layer, and the continuous extraction with ether is continued until test with pH paper indicates that the ether coming over contains no more basic material. The ether solution of the product is thoroughly dried over anhydrous potassium carbonate (Note 3). The drying agent is removed by filtration, and the solvent by distillation at atmospheric pressure and finally at water-pump pressure. The residue is distilled at reduced pressure through a short fractionating column; b.p. 95–96°/20 mm. (109–110°/40 mm., 80°/11 mm.); n_{D}^{20} 1.4780; n_{D}^{25} 1.4758; d_4^{25} 0.9733. The yield is 95–106 g. (80–90%).

2. Notes

1. The hydride can be pulverized rapidly and safely by breaking the large pieces with a spatula, followed by careful crushing with a mortar and pestle. Caution must be observed because the solid may inflame on prolonged grinding or abrasion. The hydride dust is caustic and irritating.
2. At this point the mixture should be a mobile milky slurry. The submitter suggests that, if the mixture is too thick to extract, more water or base may be added.
3. Sufficient drying agent should be used so that no aqueous liquid phase appears.

3. Discussion

2-(1-Pyrrolidyl)propanol has been prepared by the lithium aluminum hydride reduction of ethyl α -(1-pyrrolidyl)propionate.³

References and Notes

1. The Upjohn Company, Kalamazoo, Michigan.
2. *Org. Syntheses Coll. Vol. 1*, 277 (1941).
3. Moffett, *J. Org. Chem.*, **14**, 862 (1949).

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

potassium carbonate (584-08-7)

hydrochloric acid (7647-01-0)

ethyl acetate (141-78-6)

ether (60-29-7)

sodium hydroxide (1310-73-2)

lithium aluminum hydride (16853-85-3)

2-(1-Pyrrolidyl)propanol,
1-Pyrrolidineethanol, β -methyl- (53663-19-7)

Ethyl α -(1-pyrrolidyl)propionate (26846-86-6)