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.

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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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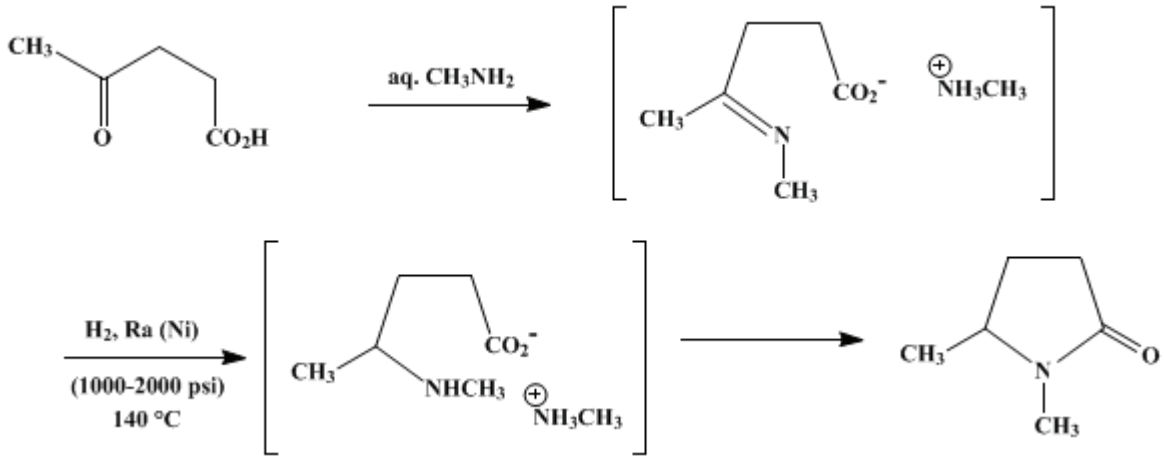
Organic Syntheses, Coll. Vol. 3, p.328 (1955); Vol. 27, p.28 (1947).

1,5-DIMETHYL-2-PYRROLIDONE

[2-Pyrrolidone, 1,5-dimethyl-]



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1. Procedure

One hundred and ninety-four grams (170 ml., 1.67 moles) of levulinic acid (Note 1) and 500 ml. of 35% aqueous methylamine (sp. gr., 0.89; 3.94 moles of methylamine) (Note 2) are placed in a 2-l. steel reaction vessel of a high-pressure hydrogenation apparatus. Ten grams of Raney nickel catalyst[1] is added, the vessel is closed, and hydrogen is admitted to a pressure of 1000–2000 lb. The bomb is then heated with continuous agitation to 140° and maintained at that temperature for 5 hours (Note 3). The contents are removed, and the bomb is washed with two 100-ml. portions of water. After removal of the catalyst by filtration (Note 4), the filtrate is distilled under reduced pressure using a good column. A low-boiling fore-run of water and methylamine distils first, followed by the product, a colorless liquid boiling at 84–86°/13 mm.; 102–104°/27 mm.; n_D^{25} 1.4611 (Note 5). The yield is 140–146 g. (74–77%).

2. Notes

1. A. E. Staley Manufacturing Company's Grade A levulinic acid was used.
2. Commercial 35% aqueous methylamine solution was used.
3. The course of the reaction may be followed by the drop in hydrogen pressure, the decrease depending upon the size of the bomb employed.
4. Because of its pyrophoric nature, the nickel catalyst should not be allowed to dry on the filter. A convenient alternative procedure for removing catalyst is to centrifuge the reaction mixture.
5. The product of some runs has a light yellow tint, but the color appears to cause no complications in subsequent reactions of the material.

3. Discussion

1,5-Dimethyl-2-pyrrolidone has been prepared by the reaction of 5-methyl-2-pyrrolidone with excess methyl iodide[2] and by the catalytic hydrogenation of a mixture of levulinic acid and methylamine.[3]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 6, 615

References and Notes

1. *Org. Syntheses* Coll. Vol. **3**, 181 (1954).
2. Senfter and Tagel, *Ber.*, **27**, 2313 (1894).
3. Hoffman-LaRoche and Co., Ger. pat. 609,244 [*C. A.*, **29**, 3116 (1935)].

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

hydrogen (1333-74-0)

nickel,
Raney nickel (7440-02-0)

Methyl iodide (74-88-4)

LEVULINIC ACID (123-76-2)

methylamine (74-89-5)

1,5-Dimethyl-2-pyrrolidone,
2-Pyrrolidone, 1,5-dimethyl- (5075-92-3)

5-methyl-2-pyrrolidone (108-27-0)

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