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.

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.

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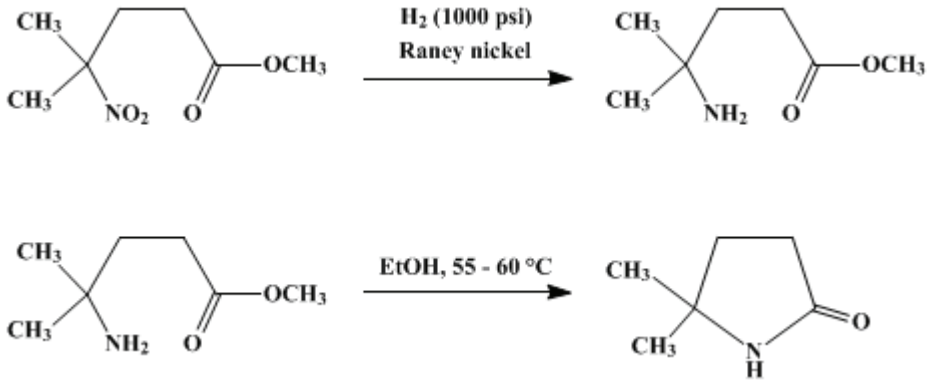
5,5-DIMETHYL-2-PYRROLIDONE

[2-Pyrrolidinone, 5,5-dimethyl-]



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1. Procedure

To a solution of 148 g. (0.845 mole) of methyl γ-methyl-γ-nitrovalerate (p.652) in 500 ml. of commercial absolute ethanol (total volume about 632 ml.) in a 2.5-l. rocking high-pressure bomb is added 12.5–25.0 g. (Note 1) of W-5[2] Raney nickel catalyst (Note 2), previously rinsed with absolute ethanol. The bomb head and fittings are placed in position, including a thermocouple attached to a semi-automatic heating control (Micromax). Hydrogen is introduced into the bomb until the pressure reaches 1000 1b. per sq. in. (Note 3).

The bomb is rocked and the temperature of the solution is raised carefully to 55° during the course of 0.5–1.0 hour (Note 4). The hydrogen uptake begins at 40–51°, and during the reaction period the temperature is held at 55–60°. The rate of pressure drop is 50–100 lb. per sq. in. each 15 minutes. The rocking of the bomb is continued, and the temperature is maintained at 60° until the pressure reading is constant for 1 hour, in order to ensure completion of the reaction.

After the bomb has been cooled the contents are removed and allowed to stand until the catalyst has settled (Note 5). The mixture is filtered, and the filtrate is transferred to a Claisen flask placed in an oil bath. The solvent is removed by distillation at atmospheric pressure, and the oil-bath temperature is raised to 200°. After temporary cooling, the residue in the flask is distilled under reduced pressure. The 5,5-dimethyl-2-pyrrolidone boils at 126.5–128.5°/12 mm. and solidifies in the receiver. The yield is 84–92 g. (88–96%); m.p. 42–43° (Note 6).

2. Notes

1. It is unnecessary and may in fact be dangerous to use a larger amount of catalyst.
2. The checkers have found that commercial grade Raney nickel (Gilman Paint and Varnish Company) is a satisfactory substitute for W-5 catalyst. The yields obtained with the two catalysts are identical, but the hydrogenation requires 2–3 hours with commercial catalyst compared with 1–1.5 hours for W-5 catalyst.
3. The theoretically required drop in hydrogen pressure for a free space of 1868 ml. and equivalent to 2.535 moles of hydrogen is 507 lb. per sq. in. The theoretical pressure drop will vary with the free space when bombs of different capacity are used. The observed pressure drop usually exceeds the theoretical by about 10%.
4. Since batches of Raney nickel may vary in activity, caution must be exercised during the heating period. The temperature should not exceed 60° at any time.

5. Filtration through Hiflo Super-Cel (Johns-Manville Company) speeds the operation but lowers the final yield.
6. The product can be recrystallized from petroleum ether (b.p. 30–38°).

3. Discussion

5,5-Dimethyl-2-pyrrolidone has been prepared by the hydrolysis of 5-imino-2,2-dimethylpyrrolidine in the presence of Raney nickel[3] or by the hydrogenation of 5-amino-2,2-dimethylpyrroline-N-oxide in the presence of Raney nickel.[3] The preparation by this method has been published.[4]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 354

References and Notes

1. The Upjohn Company, Kalamazoo, Michigan.
2. Adkins and Billica, *J. Am. Chem. Soc.*, **70**, 695 (1948).
3. Buckley and Elliott, *J. Chem. Soc.*, **1947**, 1508.
4. Moffett and White, *J. Org. Chem.*, **17**, 407 (1952).

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

petroleum ether

ethanol (64-17-5)

hydrogen (1333-74-0)

Raney nickel (7440-02-0)

5,5-Dimethyl-2-pyrrolidone,
2-Pyrrolidinone, 5,5-dimethyl- (5165-28-6)

5-amino-2,2-dimethylpyrroline-N-oxide

5-imino-2,2-dimethylpyrrolidine

Methyl γ-methyl-γ-nitrovalerate (16507-02-1)