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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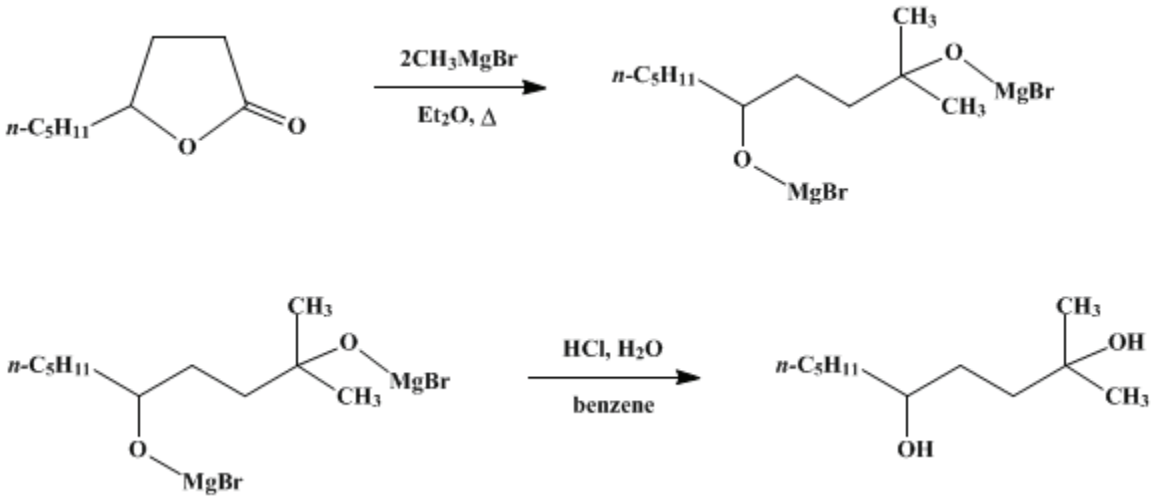
2-METHYL-2,5-DECANEDIOL

[2,5-Decanediol, 2-methyl-]



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1. Procedure

A 2-l. flask containing 1.0 l. of anhydrous ether (Note 1) is fitted with a stopper bearing an inlet tube dipping below the surface of the ether and an outlet tube protected by a calcium chloride drying tube. After the ether has been cooled thoroughly in an ice-salt bath, the flask is placed on a balance and cold methyl bromide (Note 2) is introduced through the inlet tube until the gain in weight is 200 g. (2.1 moles).

In a 3-l. three-necked flask, equipped with a sealed mechanical stirrer, reflux condenser, and a pressure-equalizing separatory funnel (Note 3), are placed 48 g. (2 g. atoms) of magnesium turnings, 500 ml. of anhydrous ether, and a small crystal of iodine. The cold methyl bromide solution is transferred to the separatory funnel and slowly added, with stirring. The reaction starts spontaneously, and the remainder of the methyl bromide is added at a rate such that the solution boils gently under reflux. Generally, the addition is complete at the end of 1–2 hours and all the magnesium should be dissolved. After the stirred solution of methylmagnesium bromide is well cooled by using an ice bath, a solution of 78.0 g. (0.5 mole) of γ-nonanoic lactone (Note 4) in 100 ml. of dry ether is added slowly over a period of 30 minutes. When the addition is complete, the mixture is placed on a steam bath and boiled under reflux for 2 hours. Then the condenser is arranged for downward distillation (Note 5), and the ether is removed.

To the thick, syrupy residue is added 200 ml. of benzene, and, after the solution is cooled in an ice bath and the condenser is set for reflux, 350 ml. of water is slowly added through the separatory funnel, with stirring. This is followed by the cautious addition of 325 ml. of a 20% solution of hydrochloric acid, and stirring is continued untill all the precipitate dissolves. The organic layer is then separated, and the aqueous layer and flask are washed with 50 ml. benzene. The combined benzene extracts are washed successively with water, a 5% solution of sodium carbonate, and again with water. Concentration of the benzene solution gives 88.5 g. of an oily residue. Careful fractional distillation (Note 6) of this residue gives, after a fore-run, 53.0 g. (57%) of the pure 2-methyl-2,5-decanediol boiling at 65–69°/2 mm., n_D^{25} 1.4420.

2. Notes

1. Commercial anhydrous ether should be dried over sodium or sodium hydride before use.
2. Commercial methyl bromide (Eastman Kodak Company, Rochester, New York) was used without purification.
3. The separatory funnel is fitted to an adapter tube extending to the bottom of the flask so that the methyl bromide solution is introduced below the surface of the mixture. A drying tube is placed in the condenser outlet.
4. Commercial γ-nonanoic lactone (Aldrich Chemical Co., Milwaukee, Wisconsin) was purified by distillation prior to use. The refractive index of the pure lactone is n_D^{25} 1.4449.
5. As the removal of ether proceeds, the viscous solution becomes difficult to stir and stirring may be stopped without harm.
6. The checkers found that an ordinary Vigreux column wasineffective in separating lower-boiling impurities. An efficient fractionating column 1m. in length and of 5 mm. I.D. gave excellent results. The infrared spectrum of the product gave no evidence of impurities.

3. Discussion

The preparation of 2-methyl-2,5-decanediol has not been described elsewhere in the literature.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 350

References and Notes

1. École de Chimie Industrielle de Lyon and Établissement Descollonges Frères (Lyon).

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

hydrochloric acid (7647-01-0)

Benzene (71-43-2)

ether (60-29-7)

magnesium,
magnesium turnings (7439-95-4)

sodium carbonate (497-19-8)

iodine (7553-56-2)

sodium (13966-32-0)

methyl bromide (74-83-9)

methylmagnesium bromide (75-16-1)

sodium hydride (7646-69-7)

2-Methyl-2,5-decanediol,
2,5-Decanediol, 2-methyl- (53731-34-3)

γ-nonanoic lactone (104-61-0)

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