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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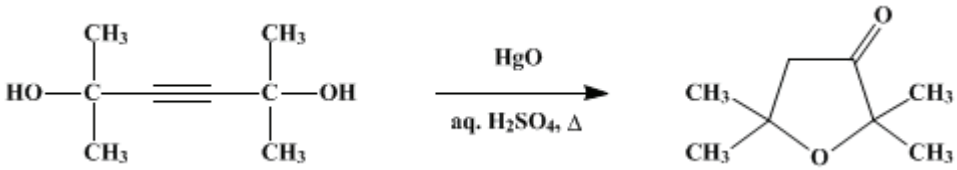
2,2,5,5-TETRAMETHYLTETRAHYDRO-3-KETOFURAN

[3(2)-Furanone, 4,5-dihydro-2,2,5,5-tetramethyl-]



Submitted by Melvin S. Newman and Walter R. Reichle[1].
Checked by Virgil Boekelheide and Graham Solomons.

1. Procedure

To the solution formed by dissolving 3 g. of mercuric oxide and 10 ml. of concentrated sulfuric acid (Note 1) in 1 l. of water is added 250 g. (1.76 moles) of 2,5-dimethyl-3-hexyne-2,5-diol (Note 2). The mixture is warmed with gentle swirling to dissolve the diol. At 80–90° the clear solution suddenly turns cloudy and the flask is immersed in a bath of water at about 20°. A colorless oil rises to the surface within a few minutes (Note 3).

The flask is then fitted for steam distillation, and 700–800 ml. of distillate is collected. After addition of 55 g. of sodium chloride to the distillate, the phases are separated in a 1-l. separatory funnel. The organic layer is dried by intermittent stirring with 25 g. of anhydrous magnesium sulfate for 6–10 hours.

The material is filtered and the residual solid washed twice with 50-ml. portions of low-boiling petroleum ether. The combined filtrates are concentrated and distilled through a short packed column to yield 190–205 g. (76–82%) of 2,2,5,5-tetramethyltetrahydro-3-ketofuran; b.p. 149–151°, n_D^{25} 1.4180.

2. Notes

1. Any other water-soluble mercury salt may be used.
2. Supplied by Air Reduction Chemical Company, 150 East 42nd Street, New York, N. Y.
3. The reaction is fairly exothermic. Cooling is advisable. An increase in acid and mercuric ion concentrations results in a faster reaction starting at a lower temperature.

3. Discussion

The procedure described is essentially that of Richet[2] which has been repeated.[3,4] The reaction is of interest since it provides a facile method of preparing tetrahydro-3-furanones which are useful reagents for alkylation in the Friedel-Crafts reaction.[4]

References and Notes

1. Department of Chemistry, Ohio State University, Columbus, Ohio.
2. M. Richet, *Ann. Chim.*, **3** (12), 317 (1948).
3. B. L. Murr, G. B. Hoey, and C. T. Lester, *J. Am. Chem. Soc.*, **77**, 4430 (1955).
4. H. A. Bruson, F. W. Grant, and E. Bobko, *J. Am. Chem. Soc.*, **80**, 3633 (1958).

Appendix

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

petroleum ether

3(2)-Furanone, 4,5-dihydro-2,2,5,5-tetramethyl-

sulfuric acid (7664-93-9)

sodium chloride (7647-14-5)

mercuric oxide (21908-53-2)

magnesium sulfate (7487-88-9)

2,2,5,5-Tetramethyltetrahydro-3-ketofuran (5455-94-7)

2,5-dimethyl-3-hexyne-2,5-diol (142-30-3)