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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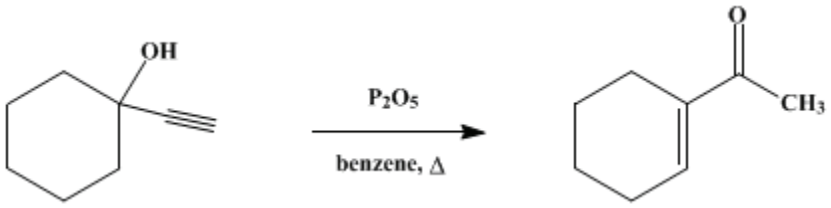
1-ACETYLCYCLOHEXENE

[Ketone, 1-cyclohexenyl methyl]



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1. Procedure

In a 500-ml. round-bottomed flask are placed 40 g. (0.32 mole) of 1-ethynylcyclohexanol (p. 416), 250 ml. of dry benzene, 10 g. of phosphorus pentoxide, and a boiling chip. A reflux condenser is attached to the flask, and the benzene solution is refluxed gently on a steam cone for 2.5 hours. At the end of that time the contents of the flask are cooled and the benzene is decanted from the phosphorus pentoxide, washed once with 100 ml. of 5% sodium bicarbonate solution, and dried over 15 g. of anhydrous sodium sulfate. The benzene is removed by distillation at atmospheric pressure, and the acetylcyclohexene is carefully fractionated at reduced pressure, through a 15-cm. helix-packed column. The yield of material boiling at 85–88°/22 mm., n_D^{20} 1.4892, is 22.5–28 g. (56–70%).

3. Discussion

1-Acetylcyclohexene has been prepared by treating cyclohexene with acetyl chloride and aluminum chloride,[2,3,4,5] by treating 1-ethynylcyclohexanol with oxalic acid[6] or 85% aqueous formic acid,[5,7,8,9] and by the dehydrohalogenation and hydrolysis of ethylidenecyclohexane nitrosochloride.[10] 1-Acetylcyclohexene and its homologs also have been prepared by the addition of a suitable diene to vinylacetylene in the presence of water and a mercury salt.[11]

References and Notes

1. This investigation was carried out under the sponsorship of the Office of Rubber Reserve, Reconstruction Finance Corporation, in connection with the Government Synthetic Rubber Program.
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9. Chanley, *J. Am. Chem. Soc.*, **70**, 246 (1948).
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11. U. S. pat. 2,301,515 [*C. A.*, **37**, 2015 (1943)].

Appendix

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

ethylidenecyclohexane nitrosochloride

Benzene (71-43-2)

acetyl chloride (75-36-5)

sodium bicarbonate (144-55-8)

Cyclohexene (110-83-8)

sodium sulfate (7757-82-6)

formic acid (64-18-6)

Oxalic acid (144-62-7)

mercury (7439-97-6)

aluminum chloride (3495-54-3)

1-Acetylcyclohexene,
Ketone, 1-cyclohexenyl methyl,
acetylcyclohexene (932-66-1)

1-Ethynylcyclohexanol (78-27-3)

vinylacetylene (689-97-4)

phosphorus pentoxide (1314-56-3)