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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CHOLESTANONE

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1. Procedure

A solution of 50 g. (0.13 mole) of dihydrocholesterol (Note 1) in 500 cc. of benzene is added slowly with cooling (Note 2) to a solution of 68 g. (0.23 mole) of crystalline sodium dichromate, 50 cc. of glacial acetic acid, and 90 cc. of concentrated sulfuric acid in 300 cc. of water in a 3-l. flask. The mixture is agitated thoroughly in a shaking device or by efficient stirring (Note 3) for six hours at 25–30° (Note 4).

The benzene solution is separated and washed twice with 100 cc. of water, once with 200 cc. of 5 per cent potassium hydroxide, and twice with water. If the solution is not colorless it is clarified with 1 g. of Norite. The benzene is removed by distillation, and the resulting syrup is dissolved in 300 cc. of alcohol by heating. The solution on cooling deposits cholestanone as well-formed needles. The yield of collected, washed, and air-dried material, m.p. 129–130°, is 41.5–42 g. (83–84 per cent of the theoretical amount). The addition of 80 cc. of water to the filtrate gives about 2 g. of material melting at 125–126°.

2. Notes

1. Material melting at 140–141° (p. 191) is satisfactory. The presence of a trace of cholesterol is not objectionable since this is converted into acidic products which are removed in the course of the purification.
2. When the solutions are mixed without cooling the temperature rises to about 60° and the yield is somewhat less.
3. The checkers employed a Hershberg stirrer (p. 117).
4. The agitation may be continued twice as long without appreciable difference in yield. Six hours is regarded as the minimum time for the quantity specified.

3. Discussion

Cholestanone has been prepared by the oxidation of dihydrocholesterol with chromic anhydride in acetic acid solution.[1] The yield is sometimes diminished as a result of the partial acetylation of the sterol.

References and Notes

1. Diels and Abderhalden, Ber. **39**, 884 (1906); Willstätter and Mayer, ibid. **41**, 2199 (1908); Vavon and Jakubowicz, Bull. soc. chim. (4) **53**, 584 (1933).

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

alcohol (64-17-5)

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

Benzene (71-43-2)

Norite (7782-42-5)

potassium hydroxide (1310-58-3)

sodium dichromate (7789-12-0)

chromic anhydride

Cholestanone (566-88-1)

Dihydrocholesterol (80-97-7)

Cholesterol (57-88-5)