



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.

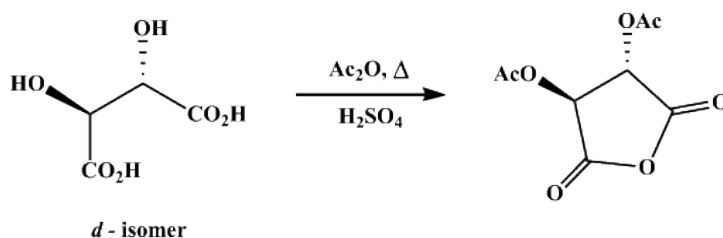
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.242 (1963); Vol. 35, p.49 (1955).

DIACETYL-*d*-TARTARIC ANHYDRIDE

[Tartaric anhydride, diacetate of *d*-]



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1. Procedure

In a 500-ml. three-necked round-bottomed flask fitted with a liquid-sealed stirrer and two reflux condensers (Note 1) is placed 40 g. (0.27 mole) of anhydrous, powdered *d*-tartaric acid (Note 2). A solution of 1.2 ml. of concentrated sulfuric acid in 136 g. (126 ml., 1.33 moles) of acetic anhydride is added, and the stirrer is started. The mixture warms up, and the tartaric acid goes into solution. The solution is heated gently (Note 1) under reflux with stirring for 10 minutes. The solution is poured into a beaker and cooled for 1 hour in an ice bath. The crude crystalline product is collected on a 15-cm. Büchner funnel (Note 3), washed twice with 20-ml. portions of dry benzene, stirred mechanically with 175 ml. of cold absolute ether, filtered, and placed in a vacuum desiccator over phosphorus pentoxide and paraffin shavings for 24 hours. The yield of diacetyl-*d*-tartaric anhydride is 41–44.5 g. (71–77%), m.p. 133–134° (Note 4), $[\alpha]_D^{20}$ 97.2° in dry chloroform ($c = 0.47$).

2. Notes

1. The reaction may be quite vigorous at its start, and the use of a large flask with two condensers is advised.
2. The anhydrous *d*-tartaric acid was obtained from Matheson, Coleman and Bell, East Rutherford, New Jersey.
3. Additional but lower-grade product may be acquired by pouring the mother liquor into petroleum ether and filtering the mixture. The recovered product is washed twice with absolute ether, filtered, and dried. About 7 g. of product, m.p. 129–131°, is thus obtained.
4. The product is not stable and should be prepared only as needed. It may be kept in a vacuum desiccator over phosphorus pentoxide and paraffin, but the melting point drops about 1 degree during the first 4 days and then remains constant at approximately 132–134°. If placed in an ordinary stoppered bottle, the product becomes gummy and the melting point falls to about 100° within 3 days. Attempts to recrystallize the anhydride invariably led to decomposition and lowered melting point.

3. Discussion

The acetylation of *d*-tartaric acid with acetic anhydride has been effected by means of sulfuric acid,^{2,3} hydrogen chloride,⁴ or 85% phosphoric acid.⁵

References and Notes

1. State University of Iowa, Iowa City, Iowa.
2. Chattaway and Parkes, *J. Chem. Soc.*, **123**, 663 (1923).
3. Roberts, *J. Chem. Soc.*, **1952**, 3315.

4. Lucas and Baumgarten, *J. Am. Chem. Soc.*, **63**, 1653 (1941).
 5. Fuchs (to Emulsol Corp.), Brit. pat. 674,710 [*C. A.*, **47**, 4904 (1953)].
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

petroleum ether

Tartaric anhydride, diacetate of d-

sulfuric acid (7664-93-9)

hydrogen chloride (7647-01-0)

Benzene (71-43-2)

ether (60-29-7)

acetic anhydride (108-24-7)

chloroform (67-66-3)

phosphoric acid (7664-38-2)

tartaric acid (87-69-4)

phosphorus pentoxide (1314-56-3)

D-tartaric acid (147-71-7)

Diacetyl-D-tartaric anhydride