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.

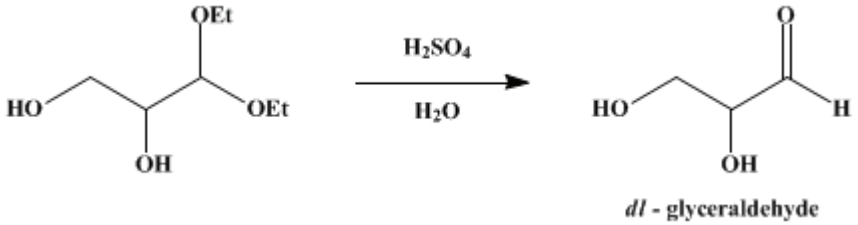
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.

DOI:10.15227/orgsyn.011.0050

Organic Syntheses, Coll. Vol. 2, p.305 (1943); Vol. 11, p.50 (1931).

dl-GLYCERALDEHYDE



dl - glyceraldehyde

Submitted by E. J. Witzemann, Wm. Lloyd Evans, Henry Hass, and E. F. Schroeder.
Checked by Frank C. Whitmore and Harry T. Neher.

1. Procedure

A mixture of 50 g. (0.3 mole) of *dl*-glyceraldehyde acetal (p. 307) and 500 cc. of 0.1 *N* sulfuric acid is allowed to stand for one week at about 20° (Note 1). Thirty cubic centimeters of glacial acetic acid is added; the mixture is neutralized carefully with barium hydroxide solution (Note 2), stirred with 5 g. of decolorizing carbon, and filtered. The filtrate is evaporated at 10 mm. pressure (Note 3). When no more water can be removed, the residue is treated with an equal volume of absolute alcohol to hasten crystallization. The crystals are collected on a filter and dried in a vacuum desiccator over soda-lime and calcium chloride. The yield of product melting at 137–139° is 22 g. (80 per cent of the theoretical amount).

2. Notes

1. During the entire preparation, including the evaporation, the temperature should be kept below 30°. If this precaution is rigidly observed, the glyceraldehyde crystallizes readily.
2. Samples of the filtered solution should give only a very slight opalescence when tested with barium hydroxide and with sulfuric acid.
3. The use of 10 mm. instead of 20 mm. pressure for the evaporation improves the quality of the glyceric aldehyde, making the difference between a syrup which may or may not crystallize and a product which crystallizes even from the concentrated solution.

3. Discussion

dl-Glyceraldehyde has been obtained by the oxidation of glycerol with nitric acid,[1] with bromine and sodium carbonate,[2] and with hydrogen peroxide in the presence of ferrous salts;[3] by the action of ultraviolet light on glycerol in neutral solution;[4] by the action of sunlight on glycerol in the presence of uranium sulfate;[5] by electrolysis of *dl*-erythronic acid;[6] by the hydrolysis of *dl*-glyceraldehyde acetal;[7] by the oxidation of acrolein;[8] by the oxidation of benzal-1,3-propenediol followed by hydrolysis;[9] and by the alkaline condensation of formaldehyde.[10]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 6, 919

References and Notes

1. Kiliani, Ber. **54**, 467 (1921); Fischer and Tafel, ibid. **20**, 3385 (1887).
2. Fischer and Tafel, ibid. **20**, 3385 (1887).
3. Fenton and Jackson, Chem. News **78**, 187 (1898); J. Chem. Soc. **75**, 5 (1899); Witzemann, J. Am. Chem. Soc. **36**, 2227 (1914).

4. Bierry, Henri, and Ranc, Compt. rend. **152**, 535 (1911).
5. Neuberg, Biochem. Z. **13**, 307 (1908).
6. Neuberg, Scott, and Lachmann, ibid. **24**, 157 (1910).
7. Wohl, Ber. **31**, 1800, 2395 (1898); Wohl and Neuberg, ibid. **33**, 3100 (1900); Evans and Hass, J. Am. Chem. Soc. **48**, 2706 (1926); Witzemann, ibid. **36**, 1913 (1914); Spoehr and Young, Carnegie Inst. Washington Yearbook, **25**, 177 (1925–1926); Exp. Sta. Record, **57**, 817 (1927) [C. A. **22**, 2368 (1928)].
8. Neuberg, Biochem. Z. **221**, 492 (1930); **255**, 1 (1932).
9. Fischer, Ahlström, and Richter, Ber. **64**, 611 (1931).
10. Kuzin, J. Gen. Chem. (U.S.S.R.) **8**, 592 (1938) [C. A. **33**, 1271 (1939)].

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

soda-lime

alcohol (64-17-5)

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

formaldehyde (50-00-0)

Acrolein (107-02-8)

glycerol (56-81-5)

nitric acid (7697-37-2)

sodium carbonate (497-19-8)

bromine (7726-95-6)

decolorizing carbon (7782-42-5)

hydrogen peroxide (7722-84-1)

barium hydroxide (17194-00-2)

glyceraldehyde,
glyceric aldehyde,
DL-Glyceraldehyde (56-82-6)

uranium sulfate

benzal-1,3-propenediol

DL-Glyceraldehyde acetal

dl-erythronic acid