



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.

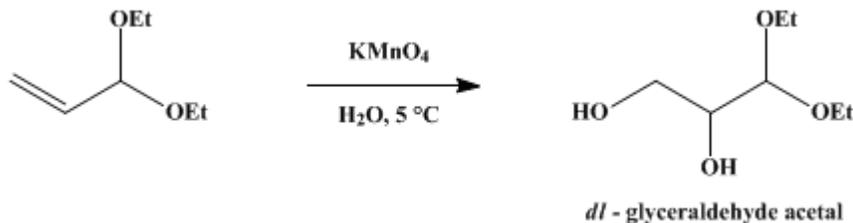
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.307 (1943); Vol. 11, p.52 (1931).

***dl*-GLYCERALDEHYDE ETHYL ACETAL**



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1. Procedure

In a 3-l. open flask, equipped with a mechanical stirrer and a thermometer and cooled in an ice bath, is placed a suspension of 65 g. (0.5 mole) of [acrolein acetal](#) (p. 17) in 600 cc. of water. The suspension is cooled to 5° ([Note 1](#)), and a solution of 80 g. (0.5 mole) of [potassium permanganate](#) in 1.5 l. of water is added, with stirring, at the rate of about 25 cc. per minute. The temperature is kept as near 5° as possible during the addition. Soon after the stirring is stopped, the mixture sets to a gel ([Note 2](#)). After standing for two hours, the mixture is heated for one hour on the steam bath and then filtered by suction on a 30-cm. Büchner funnel. The residual [manganese dioxide](#) is pressed thoroughly and washed with 150 cc. of cold water. The filtrate (about 2.3 l.) is kept cool and treated with 1.2 kg. of freshly dehydrated commercial [potassium carbonate](#). The layers are separated and the water layer is extracted with four 100-cc. portions of [ether](#). The [ether](#) extracts are added to the crude acetal layer, and the mixture, which may consist of two layers ([Note 3](#)), is dried over 10 g. of [potassium carbonate](#). After removal of the [ether](#), the residue is distilled under reduced pressure. The yield of product boiling at 120–121°/8 mm. is 55 g. (67 per cent of the theoretical amount).

2. Notes

1. The oxidation is very sensitive to changes in temperature. The best results are obtained at 5°; a slight variation causes a marked decrease in the yield.
2. If the mixture does not set to a gel, the yield is likely to be poor. This is usually due to poor temperature control.
3. Sometimes two layers appear at first, but these disappear when the [potassium carbonate](#) is added.

3. Discussion

dl-Glyceraldehyde acetal has been prepared by heating [hydroxychloropropionaldehyde acetal](#) with [potassium carbonate](#) solution;¹ by treating [glyceraldehyde](#) with alcoholic [hydrogen chloride](#);² and by oxidation of [acrolein acetal](#) with [potassium permanganate](#).³

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 2, 305](#)

References and Notes

1. Wohl, Ber. **31**, 1799 (1898).
2. Wohl and Neuberg, *ibid.* **33**, 3103 (1900); Witzemann, J. Am. Chem. Soc. **36**, 2229 (1914).
3. Wohl, Ber. **31**, 1799 (1898); Evans and Hass, J. Am. Chem. Soc. **48**, 2706 (1926); Witzemann, *ibid.* **36**, 1912 (1914); Spoehr and Young, Carnegie Inst. Washington Yearbook, **25**, 177 (1925–

1926); Expt. Sta. Record, **57**, 817 (1927) [C. A. **22**, 2368 (1928)]; Fischer and Baer, Helv. Chim. Acta **18**, 516 (1935).

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

potassium carbonate (584-08-7)

hydrogen chloride (7647-01-0)

ether (60-29-7)

potassium permanganate (7722-64-7)

manganese dioxide (1313-13-9)

Acrolein acetal

glyceraldehyde (56-82-6)

hydroxychloropropionaldehyde acetal

DL-Glyceraldehyde acetal

dl-GLYCERALDEHYDE ETHYL ACETAL