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.

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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.

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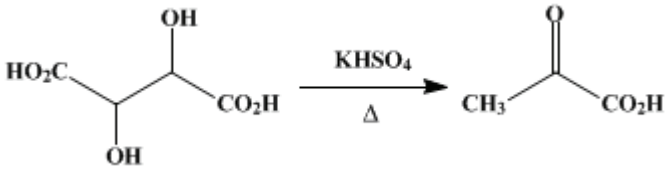
Organic Syntheses, Coll. Vol. 1, p.475 (1941); Vol. 4, p.63 (1925).

PYRUVIC ACID



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1. Procedure

An intimate mixture of 600 g. (4.4 moles) of finely powdered, freshly *fused* potassium acid sulfate and 400 g. (2.7 moles) of powdered tartaric acid, prepared by grinding them together in a mortar, is placed in a 3-l. round-bottomed Pyrex flask connected with a condenser which is filled with water but does not have any water flowing through it. The mixture is heated by means of an oil bath maintained at a temperature between 210 and 220° until liquid no longer distils over. Some foaming takes place (Note 1), but, if fused potassium acid sulfate is used and the temperature of the bath does not rise above 220°, it is not difficult to control. The distillate is then fractionated under reduced pressure. Pyruvic acid passes over at 75–80°/25 mm. and the yield is 117–128 g. (50–55 per cent of the theoretical amount) (Note 2).

2. Notes

1. If the mixture foams badly, it may be kept from frothing over by heating the upper part of the flask with a free flame.
2. The cake left in the reaction flask may be removed readily by inverting over a steam jet.

3. Discussion

Pyruvic acid can be prepared by the hydrolysis of α,α-dichloropropionic acid,[1], [2] α,α-dibromopropionic acid,[2] acetyl cyanide,[3] and oxal-acetic ester;[3] and by the distillation of tartaric acid or glyceric acid.[4] Better results are obtained, however, by the distillation of tartaric acid in the presence of a dehydrating agent such as potassium bisulfate,[5] and the procedure described was adopted after a study of a variety of dehydrating agents and various experimental conditions. The ethyl ester can be prepared by the catalytic oxidation of ethyl lactate,[6] and the acid has been obtained by the oxidation of methylglyoxal bisulfite.[7]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 610

References and Notes

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2. Kowski, Ann. **342**, 132 (1905).
3. Claisen and Shadwell, Ber. **11**, 620, 1563 (1878); Wislicenus, Ann. **246**, 327 (1888).
4. Berzelius, Ann. Physik (2) **36**, 1 (1835); Moldenhauer, Ann. **131**, 338 (1864); Böttinger, Ann. **188**, 314 (1877).
5. Erlenmeyer, Ber. **14**, 321 (1881); Simon, Bull. soc. chim. (3) **13**, 335 (1895); de Jong, Rec. trav. chim. **19**, 278 (1900); Döbner, Ann. **242**, 269 (1887); Wohl and Maag, Ber. **43**, 2188 (1910).

6. Häussler, U. S. pat. 1,164,195 [C. A. **21**, 746 (1927)].
7. Neuberg and Kobel, Biochem. Z. **258**, 365 (1933).

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

ethyl ester

potassium acid sulfate

oxal-acetic ester

potassium bisulfate (7646-93-7)

tartaric acid (87-69-4)

Pyruvic acid (127-17-3)

α,α-dichloropropionic acid (75-99-0)

α,α-dibromopropionic acid

acetyl cyanide (631-57-2)

glyceric acid (600-19-1)

ethyl lactate (687-47-8)

methylglyoxal bisulfite