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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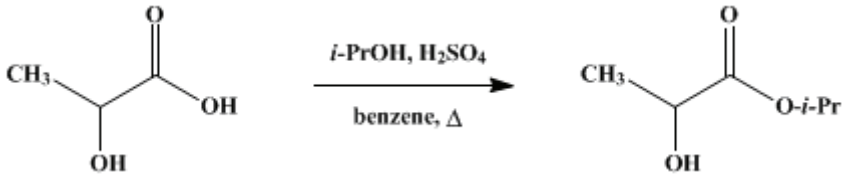
ISOPROPYL LACTATE

[Lactic acid, isopropyl ester]



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1. Procedure

In a 3-l. round-bottomed flask (Note 1) fitted with a 1-meter fractionating column[1] are placed 450 g. (7.5 moles) of anhydrous isopropyl alcohol (Note 2), 212 g. (2 moles) of u.s.p. 85 per cent lactic acid, 1 l. of benzene, and 5 cc. of concentrated sulfuric acid. The flask is placed in an air bath on an electric hot plate (Note 3) and heated until the benzene-isopropyl alcohol-water ternary mixture distils at 66.5°. Distillation is continued slowly (six to seven hours) until the temperature at the head of the column rises to and persists at 71–72° (isopropyl alcohol-benzene binary mixture), and no further separation of water occurs. Ten grams of precipitated calcium carbonate is then added to the mixture, and distillation is continued until the temperature rises to 80° in order to remove most of the benzene and excess isopropyl alcohol (Note 4) and (Note 5). The contents of the flask are then filtered into a modified Claisen flask and distilled under reduced pressure. Cuts are taken to 60°, 60–75°, 75–80°, and 80–100° at 32 mm. The fraction boiling at 75–80°/32 mm. is isopropyl lactate and weighs 130–160 g. By redistilling the high and low fractions an additional 30–60 g. is obtained, bringing the total yield to 160–180 g. (60–68 per cent of the theoretical amount). The ester may be redistilled at atmospheric pressure (with some loss due to decomposition) at 166–168°.

2. Notes

1. With larger amounts of material it is desirable to employ a two-necked flask; the spare neck is used for introducing the calcium carbonate later in the process.
2. Commercial anhydrous alcohol was used in this preparation. Isopropyl alcohol is very difficult to dry satisfactorily. The water binary mixture, boiling at 80.35°, contains 12.1 per cent of water by weight. The ternary mixture with benzene, boiling at 66.5°, contains 73.8 per cent benzene, 18.7 per cent isopropyl alcohol, and 7.5 per cent water. Hence by adding 120 g. of dry benzene to 100 g. of the isopropyl alcohol-water binary mixture, and distilling until the temperature reaches 82°, there will remain 55 to 60 g. of nearly dry isopropyl alcohol.
3. A water or steam bath or oil bath may be used.
4. The temperature of the vapors should not be allowed to rise above 72° before the addition of the calcium carbonate. If too much alcohol is removed before the acid is neutralized, charring and resinification take place with a decrease in the yield of ester.
5. The recovered benzene and excess isopropyl alcohol may be dried by distillation and used in a subsequent run.

3. Discussion

Isopropyl lactate has been prepared by heating isopropyl alcohol and lactic acid in a sealed tube at 170°,[2] and from silver lactate and isopropyl iodide, together with the isopropyl ester of α-isopropoxypropionic acid.[3] Direct esterification of lactic acid with isopropyl alcohol, using sulfuric acid, has hitherto given less than a 20 per cent yield of impure ester.

References and Notes

1. Clarke and Rahrs, Ind. Eng. Chem. **15**, 349 (1923).
2. Silva, Bull. soc. chim. (2) **17**, 97 (1872).
3. Purdie and Lander, J. Chem. Soc. **73**, 298 (1898).

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

sulfuric acid (7664-93-9)

Benzene (71-43-2)

calcium carbonate (471-34-1)

isopropyl alcohol (67-63-0)

lactic acid (50-21-5)

Isopropyl lactate,
Lactic acid, isopropyl ester (63697-00-7)

silver lactate (128-00-7)

isopropyl iodide (75-30-9)

isopropyl ester of α-isopropoxypropionic acid