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.

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.

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Organic Syntheses, Coll. Vol. 2, p.560 (1943); Vol. 12, p.66 (1932).

SUCCINIC ANHYDRIDE

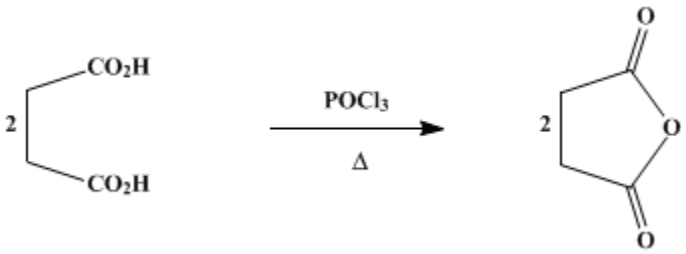
[I]

Submitted by Louis F. Fieser and E. L. Martin.
Checked by C. R. Noller

1. Procedure

In a 1-l. round-bottomed flask, fitted with a reflux condenser attached to a gas trap, are placed 118 g. (1 mole) of succinic acid and 215 cc. (235 g., 3 moles) of acetyl chloride. The mixture is gently refluxed on the steam bath until all the acid dissolves; this requires one and a half to two hours. The solution is allowed to cool undisturbed and is finally chilled in an ice bath. The succinic anhydride, which separates in beautiful crystals, is collected on a Büchner funnel, washed with two 75-cc. portions of ether, and dried in a vacuum desiccator. The yield of material melting at 118–119° is 93–95 g. (93–95 per cent of the theoretical amount).

[II]



Submitted by R. L. Shriner and H. C. Struck.
Checked by C. R. Noller, F. B. Hilmer, and J. D. Pickens.

1. Procedure

Two hundred thirty-six grams (2 moles) of succinic acid (Note 1) and 153.5 g. (1 mole) of phosphorus oxychloride are placed in a 1-l. Claisen flask. One neck of the flask is equipped with a reflux condenser connected to a gas trap; the other neck and the side arm of the flask are closed with corks. The mixture is heated (Note 2) with a free flame for about fifty minutes until no more hydrogen chloride is evolved. The condenser is removed and the flask is arranged for distillation, with a tube leading from the side arm of the receiver to the drain in order to carry off the vapors. A few cubic centimeters of distillate is collected below 255°, at which temperature the receiver is changed and succinic anhydride is collected from 255–260° (Note 3). The product melts at 118–120° and weighs 164–192 g. (82–96 per cent of the theoretical amount).

The succinic anhydride may be purified by dissolving 50 g. of the crude material in 35 cc. of acetic anhydride. The hot solution is cooled in an ice bath. The crystals are filtered with suction, washed with two 20-cc. portions of cold, absolute ether, and air-dried quickly at 40°. The yield of the pure anhydride,

which melts at 119–120°, is 43.5 g.

2. Notes

1. Succinic acid having a melting point of 189–190° was used. Less pure material gives a lower-melting product.
2. The reaction foams considerably at the start, hence slow and careful heating is necessary. It is best to heat the flask directly with the flame, making certain that all parts of the mixture are heated about equally.
3. The tarry mass left in the distilling flask may be easily removed by warm dilute sodium hydroxide.

3. Discussion

Succinic anhydride has been prepared from succinic acid with phosphorus pentachloride,[1], [2] phosphorus oxychloride,[2], [3] phosphorus pentoxide,[4] thionyl chloride,[5] acetyl chloride,[3], [6], [7] or succinyl chloride;[8] from barium or sodium succinate with benzoyl chloride,[1] acetyl chloride,[9] acetic anhydride,[10] or benzophenone dichloride;[11] from succinyl chloride and oxalic acid[6] or sodium acetate;[9] and from ethyl succinate and benzoyl chloride.[12]

The procedure in Org. Syn. Coll. Vol. I, **1941**, 91, for preparing benzoic anhydride when applied to succinic acid and acetic anhydride gives a 72 per cent yield of succinic anhydride. Procedure I above has the advantage of convenience; Procedure II is more economical.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 81

References and Notes

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4. d'Arcet, Ann. chim. phys. (2) **58**, 288 (1835).
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7. Schulz, Ber. **18**, 2459 (1885).
8. Anschütz, ibid. **10**, 1883 (1877).
9. Heintz, Jahresb. **1859**, 279.
10. Oddo and Manuelli, Gazz. chim. ital. **26** (II) 482 (1896).
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12. Kraut, Ann. **137**, 254 (1866).

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

barium or sodium succinate

hydrogen chloride (7647-01-0)

ether (60-29-7)

acetic anhydride (108-24-7)

sodium acetate (127-09-3)

sodium hydroxide (1310-73-2)

phosphorus pentachloride (10026-13-8)

acetyl chloride (75-36-5)

thionyl chloride (7719-09-7)

ethyl succinate

Oxalic acid (144-62-7)

Succinic acid (110-15-6)

benzoyl chloride (98-88-4)

Benzoic anhydride (93-97-0)

Phosphorus Oxychloride (21295-50-1)

benzophenone dichloride

Succinic anhydride (108-30-5)

succinyl chloride (543-20-4)

phosphorus pentoxide (1314-56-3)