



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.

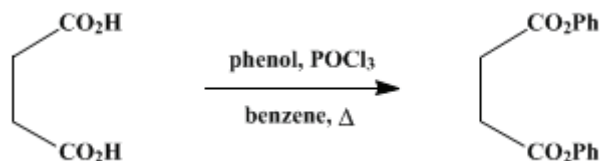
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

Organic Syntheses, Coll. Vol. 4, p.390 (1963); Vol. 34, p.44 (1954).

DIPHENYL SUCCINATE

[Succinic acid, diphenyl ester]



Submitted by Guido H. Daub and William S. Johnson¹.

Checked by James Cason, Robert A. Wessman, and William G. Dauben.

1. Procedure

A mixture of 118 g. (1 mole) of [succinic acid](#), 188 g. (2 moles) of [phenol](#), and 138 g. (83 ml., 0.9 mole) of [phosphorus oxychloride](#) ([Note 1](#)) is placed in a 2-l. round-bottomed flask fitted with an efficient reflux condenser capped with a calcium chloride tube ([Note 2](#)) and ([Note 3](#)). The mixture is heated on a steam bath in a hood ([Note 3](#)) for 1.25 hours, 500 ml. of [benzene](#) is added, and the refluxing is continued for an additional hour. The hot [benzene](#) solution is decanted from the red syrupy residue of [phosphoric acid](#) and filtered by gravity into a 1-l. Erlenmeyer flask. The syrupy residue is extracted with two 100-ml. portions of hot [benzene](#), which are also filtered into the Erlenmeyer flask. The combined [benzene](#) solutions are concentrated to a volume of about 600 ml. ([Note 4](#)), and the pale yellow solution is allowed to cool, whereupon the [diphenyl succinate](#) separates as colorless crystals. It is filtered with suction on a Büchner funnel, washed with three 50-ml. portions of [ether](#), and dried on a porous plate at 40°. The yield of [diphenyl succinate](#), m.p. 120–121°, is 167–181 g. (62–67%) ([Note 5](#)).

2. Notes

1. The use of a larger proportion of [phosphorus oxychloride](#) failed to improve the yield and in general gave an inferior product.
2. Ground-glass joints are preferred; however, burnt-cork stoppers may be used.
3. Provision should be made to dispose of the [hydrogen chloride](#) which is evolved during the reaction.
4. If the mother liquor is concentrated further before removal of [phenol](#) and [phosphorus oxychloride](#) a dark, impure product is obtained.
5. If desired, a second crop may be obtained by the following procedure: The mother liquor is extracted in a 1-l. separatory funnel with eight 50-ml. portions of 5% [potassium hydroxide](#) solution. Each extract is extracted in turn with a 50-ml. portion of [benzene](#). The two [benzene](#) solutions are washed in turn with a 50-ml. portion of water and two 50-ml. portions of saturated [sodium chloride](#) solution. All the [benzene](#) solutions are combined, dried over anhydrous [sodium sulfate](#), filtered, and concentrated to a volume of 150 ml. Cooling yields a second crop of [diphenyl succinate](#), which is collected, washed, and dried as described for the first crop. The yield is 13–27 g. (5–10%), m.p. 118–120°.

3. Discussion

The above procedure is a modification of that described by Rasinski.² [Diphenyl succinate](#) has also been prepared by the reaction of [phenol](#) and [succinic acid](#) in the presence of [phosphorus pentoxide](#) in [toluene](#)³ and by the treatment of [phenol](#) with [succinyl chloride](#).⁴

References and Notes

1. University of Wisconsin, Madison, Wisconsin.
2. Rasinski, *J. prakt. Chem.*, **26**, 63 (1882). Bischoff and Hedenstrom, *Ber.*, **35**, 4073 (1902).
3. Bakunin, *Gazz. chim. ital.*, **30**, 358 (1900).

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

hydrogen chloride (7647-01-0)

Benzene (71-43-2)

ether (60-29-7)

phenol (108-95-2)

sodium chloride (7647-14-5)

sodium sulfate (7757-82-6)

Succinic acid (110-15-6)

Phosphorus Oxychloride (21295-50-1)

potassium hydroxide (1310-58-3)

toluene (108-88-3)

phosphoric acid (7664-38-2)

succinyl chloride (543-20-4)

Diphenyl succinate,
Succinic acid, diphenyl ester (621-14-7)

phosphorus pentoxide (1314-56-3)