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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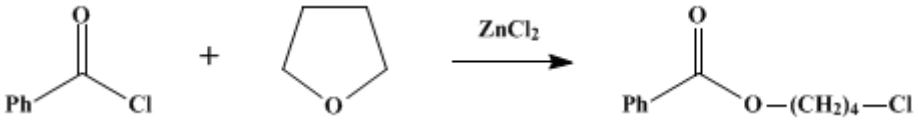
4-CHLOROBUTYL BENZOATE

[1-Butanol, 4-chloro-, benzoate]



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1. Procedure

In a 200-ml. round-bottomed flask fitted with an efficient reflux condenser are mixed 31.4 ml. (38 g., 0.27 mole) of freshly distilled benzoyl chloride, 28.2 ml. (25 g., 0.35 mole) of tetrahydrofuran (Note 1), and 5 g. of freshly fused zinc chloride. A vigorous reaction begins immediately, and after a few seconds, when the mixture starts to boil, external cooling is applied with an ice bath. After the initial reaction has subsided, the mixture is heated on a steam bath for 15 minutes, cooled, and dissolved in 100 ml. of benzene. The benzene solution is washed with 100 ml. of a 5% solution of sodium chloride and then with 100 ml. of a saturated solution of sodium bicarbonate. The benzene layer is dried over anhydrous sodium sulfate and fractionally distilled from a modified Claisen flask.

The product is collected at 140–143°/5 mm., 132–135°/2.5 mm.; n_D^{25} 1.5176 (Note 2). The yield is 45–48 g. (78–83%).

2. Notes

1. Good-quality commercial tetrahydrofuran may be used as received, or redistilled; b.p. 65–66°.
2. The product develops a slight yellow tint on standing.

3. Discussion

4-Chlorobutyl benzoate has been prepared by the action of benzoyl chloride on tetrahydrofuran in the presence of titanium chloride, stannic chloride,[1] or zinc chloride.[2]

References and Notes

1. Gol'dfarb and Smorgonskii, *J. Gen. Chem. U.S.S.R.*, **8**, 1516 (1938) [*C. A.*, **33**, 4593 (1939)].
2. Cloke and Pilgrim, *J. Am. Chem. Soc.*, **61**, 2667 (1939).

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

Benzene (71-43-2)

sodium bicarbonate (144-55-8)

sodium chloride (7647-14-5)

sodium sulfate (7757-82-6)

benzoyl chloride (98-88-4)

zinc chloride (7646-85-7)

stannic chloride (7646-78-8)

Tetrahydrofuran (109-99-9)

4-CHLOROBUTYL BENZOATE,
1-Butanol, 4-chloro-, benzoate (946-02-1)

titanium chloride (7550-45-0)