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.

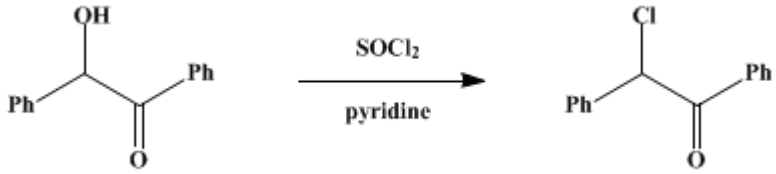
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.012.0020

Organic Syntheses, Coll. Vol. 2, p.159 (1943); Vol. 12, p.20 (1932).

DESYL CHLORIDE

[Acetophenone, α-chloro-α-phenyl-]



Submitted by A. M. Ward
Checked by C. S. Marvel and Tse-Tsing Chu.

1. Procedure

In a 1-l. beaker are placed 100 g. (0.47 mole) of benzoin (Org. Syn. Coll. Vol. I, **1941**, 94), and 50 g. (57 cc.) of pyridine. The mixture is heated until a solution is obtained, then cooled in an ice bath until solid. The mass is coarsely ground, and 75 g. (46 cc.; 0.63 mole) of thionyl chloride is added slowly with vigorous stirring and cooling in a water bath. After each addition of thionyl chloride, the reaction mixture becomes quite hot, and considerable amounts of sulfur dioxide and hydrogen chloride are evolved. At first the mass becomes pasty and then soon sets to a light yellow solid. After about an hour, water is added and the solid is coarsely ground and filtered. It is finely triturated twice with water, filtered by suction, and pressed as dry as possible. The white powder is dried to constant weight over sulfuric acid or calcium chloride. The yield of crude product is about 125 g. The compound is dissolved in 450 cc. of boiling 95 per cent alcohol (Note 1), filtered, and the filtrate cooled by running water. There is obtained 77 g. of colorless crystals which, after drying in the air, melt at 66–67°. On cooling the mother liquor in an ice-salt mixture, there is obtained an additional 9 g. of crystals melting at 65–66°. Further cooling of the filtrate yields no more product. The total yield is 80–86 g. (74–79 per cent of the theoretical amount) (Note 2) and (Note 3).

2. Notes

1. The product may be recrystallized from petroleum ether (b.p. 40–60°), but this solvent is less satisfactory for large amounts of material.
2. When the preparation is carried out with one-fifth these quantities the yield is only about 70 per cent of the theoretical amount.
3. Desyl chloride decomposes and becomes brown when exposed to sunlight, but is quite stable if kept in dark bottles.

3. Discussion

Desyl chloride has been prepared by the action of thionyl chloride on benzoin,[1] and on *l*-benzoin.[2] It has also been prepared by the action of hydrogen chloride on azibenzil.[3]

References and Notes

1. Schroeter, Ber. **42**, 2348 (1909).
2. McKenzie and Wren, J. Chem. Soc. **97**, 481 (1910).
3. Curtius and Lang, J. prakt. Chem. (2) **44**, 547 (1891).

Appendix

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

petroleum ether

azibenzil

alcohol (64-17-5)

calcium chloride (10043-52-4)

sulfuric acid (7664-93-9)

hydrogen chloride (7647-01-0)

thionyl chloride (7719-09-7)

sulfur dioxide (7446-09-5)

pyridine (110-86-1)

Benzoin,
l-benzoin (119-53-9)

Desyl chloride,
Acetophenone, α-chloro-α-phenyl- (447-31-4)