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.

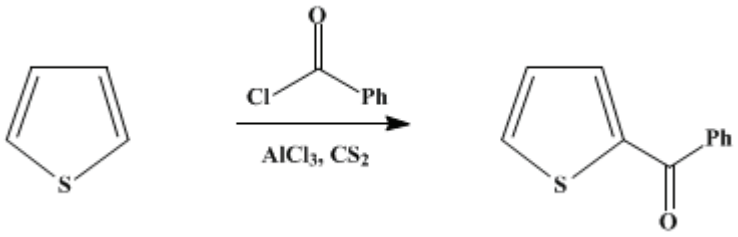
These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.012.0062

Organic Syntheses, Coll. Vol. 2, p.520 (1943); Vol. 12, p.62 (1932).

PHENYL THIENYL KETONE

[Ketone, phenyl 2-thienyl]



Submitted by Wesley Minnis
Checked by Roger Adams and H. D. Cogan.

1. Procedure

In a 1-l. three-necked flask, equipped with a mechanical stirrer, a reflux condenser, and a thermometer (with bulb immersed in the liquid), are placed 100 g. (0.75 mole) of anhydrous aluminum chloride and 300 g. of carbon disulfide (Note 1). The suspension is cooled to 15–25°, and a solution of 60 g. (0.71 mole) of thiophene (p. 578) and 105 g. (0.75 mole) of benzoyl chloride in 225 g. of carbon disulfide is added through the condenser, with stirring, over a period of three and one-half hours (Note 2). The solution is allowed to warm up to room temperature, and stirring is continued for three more hours; the reaction mixture is then allowed to stand overnight. The mixture is refluxed on the water bath for three and one-half hours, cooled, poured on ice, and extracted with ether. The ether extract is washed successively with sodium carbonate solution and water, and then dried over calcium chloride. The ether is removed by distillation on the water bath, and the residue is distilled under reduced pressure. The yield of product boiling at 200–209°/30–40 mm. is 117–120 g. (88–90 per cent of the theoretical amount). On crystallization from 1 l. of petroleum ether (b.p. 65–110°) there is obtained 110–112 g. of product melting at 52°. Another crystallization from petroleum ether gives a product which melts at 55–56°. The loss on the second crystallization is about 10 per cent.

2. Notes

1. The carbon disulfide was dried over calcium chloride.
2. Thiophene and aluminum chloride react vigorously in carbon disulfide suspension. Subsequent addition of a carbon disulfide solution of benzoyl chloride produces a tar, and a low yield of ketone results.

3. Discussion

Phenyl thienyl ketone has been prepared by treatment of benzoyl chloride with thienylmercuric chloride;[1] by treatment of thiophene with benzoyl chloride in the presence of thienylmercuric chloride,[2] phosphorus pentoxide,[3] stannic chloride,[4] and aluminum chloride.[5] It has also been prepared from thienylmagnesium iodide and benzonitrile.[6]

References and Notes

1. Volhard, Ann. **267**, 179 (1892); Steinkopf and Killingstad, ibid. **532**, 288 (1937).
2. Steinkopf and Bauermeister, ibid. **403**, 70 (1914).
3. Steinkopf, ibid. **413**, 349 (1917).
4. Stadnikoff and Rakowsky, Ber. **61**, 269 (1928); Goldfarb, J. Russ. Phys.-Chem. Soc. **62**, 1073 (1930) [C. A. **25**, 2719 (1931)].

5. Comey, Ber. **17**, 790 (1884).
6. Thomas and Couderc, Bull. soc. chim. (4) **23**, 289 (1918).

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

petroleum ether

calcium chloride (10043-52-4)

ether (60-29-7)

benzonitrile (100-47-0)

sodium carbonate (497-19-8)

benzoyl chloride (98-88-4)

aluminum chloride (3495-54-3)

carbon disulfide (75-15-0)

Thiophene (110-02-1)

stannic chloride (7646-78-8)

Phenyl thienyl ketone,
Ketone, phenyl 2-thienyl (135-00-2)

thienylmercuric chloride

thienylmagnesium iodide

phosphorus pentoxide (1314-56-3)