



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.

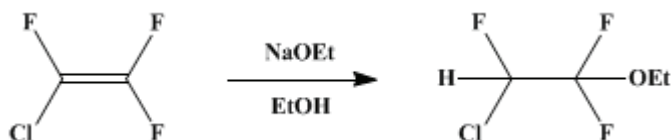
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries 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.184 (1963); Vol. 34, p.16 (1954).

2-CHLORO-1,1,2-TRIFLUOROETHYL ETHYL ETHER

[Ether, 2-chloro-1,1,2-trifluoroethyl ethyl]



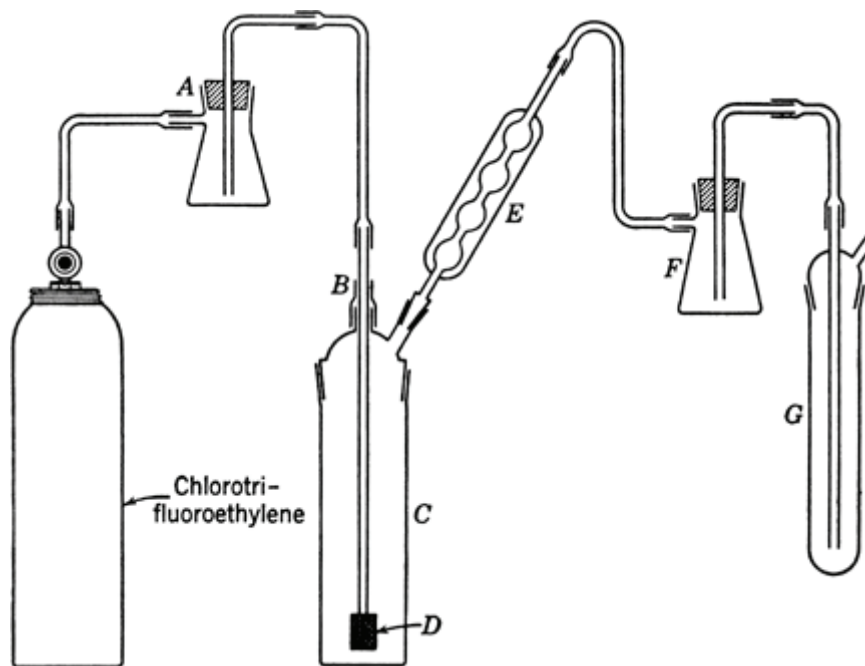
Submitted by Bruce Englund¹

Checked by R. S. Schreiber and Burris D. Tiffany.

1. Procedure

The apparatus used is shown in Fig. 6 (Note 1). A solution of sodium ethoxide prepared by dissolving 2.5 g. (0.11 g. atom) of clean sodium in 230 g. (292 ml., 5 moles) of absolute ethanol under anhydrous conditions is added to the reaction tube C. Tube C thus charged is weighed and placed in position with the gas inlet tube, fitted with fritted-glass dispersion cylinder D, extending nearly to the bottom. Several inches of ethanol is placed in tube G, which serves as a flow indicator. The traps A and F are provided to protect against suck-back if the gas flow is interrupted. The seal at B is a sleeve of rubber tubing. Any efficient reflux condenser E is satisfactory.

Fig. 6.



Chlorotrifluoroethylene (Note 2) is introduced at such a rate, controlled by the needle valve, that it is essentially all absorbed in the reaction tube C, as indicated by the escape of little or no gas through tube G. At this rate, 233 g. (2 moles) (Note 3) is absorbed in 2–2.5 hours, during which the temperature rises to the point of reflux (Note 4).

When the required amount of chlorotrifluoroethylene has been absorbed, the reaction mixture is poured into 500 ml. of water. The product separates as a light-yellow oil, which is separated, washed with 250 ml. of water, and dried over 20 g. of anhydrous calcium chloride. From 233 g. of chlorotrifluoroethylene (2 moles), the yield of crude product is 300–315 g. (92–97%). This material is

suitable for use in preparation of [ethyl chlorofluoroacetate](#) (p. 423). It may be fractionated through an efficient column to give 285–300 g. (88–92%) of pure [chlorotrifluoroethyl ethyl ether](#), b.p. 87–88°, n_D^{25} 1.3427.

2. Notes

1. The reaction tube *C* may be of any convenient size. The tube used by the submitter was 300 by 55 mm., fitted with a 55/35 standard taper ground-glass joint. The checkers used a 500-ml. calibrated dropping funnel which was convenient for following the increase in volume during the absorption of the [chlorotrifluoroethylene](#). For safety, a heavy grease such as Cello-Grease was used in the stopcock.
2. The [chlorotrifluoroethylene](#) used was inhibited polymerization grade, supplied by the Kinetic Chemicals Division, Organic Chemicals Department, E. I. du Pont de Nemours and Company, Wilmington, Delaware.
3. The amount of [chlorotrifluoroethylene](#) absorbed is determined by weighing the reaction tube *C* and contents. A convenient alternative is to note the increase in volume of the reaction mixture, which in this case amounts to 165 ml.
4. The rate of absorption is slow at first but increases as the temperature rises.

3. Discussion

[2-Chloro-1,1,2-trifluoroethyl ethyl ether](#) has been prepared by the base-catalyzed addition of ethanol to [1-chloro-1,2,2-trifluoroethylene](#).^{2,3,4}

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 4, 423](#)

References and Notes

1. Chemical Department, Experimental Station, E. I. du Pont de Nemours and Company, Wilmington, Delaware.
2. Hanford and Rigby, U. S. pat. 2,409,274 [*C. A.*, **41**, 982 (1947)].
3. Park, Vail, Lea, and Lacher, *J. Am. Chem. Soc.*, **70**, 1550 (1948).
4. Barr, Rapp, Pruett, Bahner, Gibson, and Lafferty, *J. Am. Chem. Soc.*, **72**, 4480 (1950).

Appendix

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

[ethanol](#) (64-17-5)

[calcium chloride](#) (10043-52-4)

[sodium](#) (13966-32-0)

[sodium ethoxide](#) (141-52-6)

[chlorotrifluoroethyl ethyl ether](#),
[2-Chloro-1,1,2-trifluoroethyl ethyl ether](#),
[Ether, 2-chloro-1,1,2-trifluoroethyl ethyl](#) (310-71-4)

Chlorotrifluoroethylene,
1-chloro-1,2,2-trifluoroethylene (79-38-9)

ethyl chlorofluoroacetate (401-56-9)