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.

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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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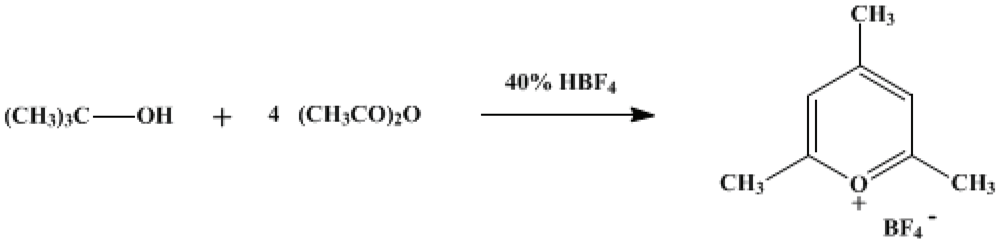
2,4,6-TRIMETHYLPYRYLIUM TETRAFLUOROBORATE



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Checked by Dorothy G. McMahan and Henry E. Baumgarten.

1. Procedure

In a 250-ml. Erlenmeyer flask provided with a magnetic stirrer bar and a thermometer containing 50 ml. (54 g., 530 mmol.) of acetic anhydride and 4.0 ml. (3.1 g., 41 mmol.) of dry *t*-butyl alcohol, 7 ml. (39 mmol.) of 40% fluoroboric acid (Note 1) are added, initially very cautiously, and then in *ca.* 0.2-ml. portions until all has been added, at such a rate that the final temperature reaches *ca.* 100°. A dark yellow-brown color develops. The solution is allowed to cool spontaneously to 80°, and then is chilled to 5° in an ice bath. Separation of the salt begins and is completed by the addition of 100 ml. of ether. After filtration on a Büchner funnel the salt is washed with 30 ml. of ether, yielding 3.9–4.1 g. (47–50%) of a colorless or pale yellow product, m.p. 218–220° (decomp.) (Note 2). The crude product may be recrystallized from ethanol-methanol 1:1 (*ca.* 70–80 ml.) containing a few drops of fluoroboric acid, affording 3.4–3.5 g. (41–43%) of colorless prisms, m.p. 224–226° (decomp.) (Note 3).

2. Notes

1. The checkers used 6 ml. of 48% fluoroboric acid obtained from the Ozark Mahoning Co.
2. The checkers collected two crops of crystals, the second precipitating during the filtration of the first. The crude product was dried between filter papers overnight.
3. The yields reported are those of the checkers obtained with 48% fluoroboric acid and are about 30% higher than those (27–32%) reported by the submitters. Larger scale preparations (five or more times the quantities described here) have been carried out by the submitters. Such preparations require external cooling.

3. Discussion

See the Discussion section of 2,4,6-trimethylpyrilium trifluoromethanesulfonate.[2]

References and Notes

1. Institute of Atomic Physics, Bucharest, Rumania.
2. A. T. Balaban and A. J. Boulton, this volume, p. 1114.

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

2,4,6-trimethylpyrilium trifluoromethanesulfonate ether (60-29-7)

acetic anhydride (108-24-7)

ethanol-methanol (504-63-2)

t-butyl alcohol (75-65-0)

2,4,6-Trimethylpyrylium tetrafluoroborate (773-01-3)

fluoroboric acid (16872-11-0)