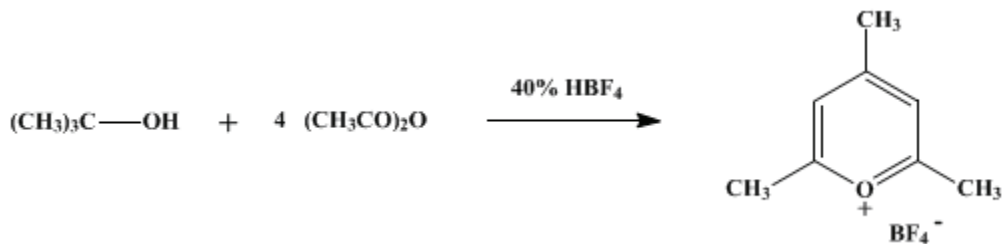


2,4,6-TRIMETHYLPYRYLIUM TETRAFLUOROBORATE



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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-trimethylpyrylium 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-trimethylpyrylium 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)