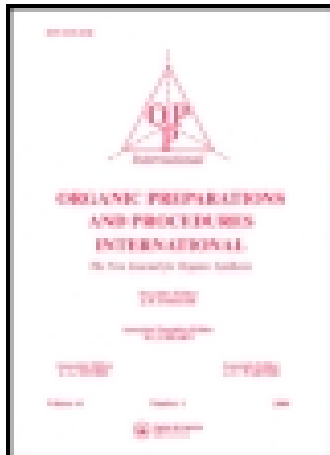


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IMPROVED ONE-POT PREPARATION OF TETRAMETHYLFLUOROFORMAMIDIUM HEXAFLUOROPHOSPHATE

Tomas Vojkovsky^a & Brian Drake^a

^a Selectide, Hoechst Marion Roussel, Dept. of Chemistry, 1580 E. Hanley Blvd, Tucson, AZ, 85704

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of KF (Aldrich 'spray-dried', 40.1 g, 0.69 mol) and KPF_6 (116.0 g, 0.63 mol) was added together with anhydrous acetonitrile (1 L) to the reaction flask. The reaction mixture was stirred at 55° for 3 h, then cooled to R.T. and filtered through a large coarse filtration funnel (the salts were washed with 3 x 150 mL of acetonitrile). The combined filtrates were evaporated and the resulting oily residue was taken up in hot dichloroethane (700 mL). The cloudy solution was filtered while hot using a medium-porosity filtration funnel and then concentrated to approximately 450 mL of volume. THF (600 mL) was added under stirring and the mixture was then shaken for 2 min to promote crystallization. The flask was cooled to R.T. The precipitate was collected under positive pressure of nitrogen and washed with THF (2 x 250 mL). A stream of nitrogen was passed through the product for 20 min, the drying process was completed in vacuum (0.3 mm Hg, 2 h), to yield 143.0 g (90 %) of white crystalline product, mp. 112-113°, lit.⁶ 111-112°.

¹H NMR spectra (DMSO, δ 3.150, d) were in accordance with published values.⁶

The above procedure was performed four times, with yields ranging from 86 to 90%.

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