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THE FLUORINATION OF SILYLATED PHOSPHITES BY PERFLUOROPROPYLENE OXIDE

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The chemical properties of perfluoroolefin oxides have not been studied extensively. We have found that silyl ethers of dialkyl phosphites undergo oxidative fluorination efficiently by perfluoropropylene oxide (I) under mild conditions to give the corresponding dialkyl fluorophosphates. This reaction has not been described previously.

$$CF_2-CF-CF_3 + (RO)_2POSi \rightarrow Me_3(RO)_2P$$

$$(I) \qquad (II) \qquad (IIIa-c)$$

R = Me (a), Et (b), Pr (c)

General Procedure. A sample of 0.01-0.015 mole phosphite (II) was added to a steel test tube and a 1.5-fold excess of (I) was condensed at from -50 to -40°C. The test tube was sealed, brought to room temperature, and maintained for 10 h. After opening, the reaction mixture was distilled in vacuum to give dialkyl fluorophosphates (III). (IIIa): bp 39-44°C (9 mm), δP (H₃PO₄, ppm): -6.3, J_{P-F} = 972 Hz, δF (CF₃CO₂H, ppm): -9.9, J_{P-F} = 976 Hz (37%) [1]. (IIIb): bp 58-65°C (9 mm), δP) -8.5, J_{P-F} = 976 Hz, δF) -4.7, J_{P-F} = 976 Hz (56%) [1]. (IIIc): bp 79-92°C (9 mm), δP) -8.3, J_{P-F} = 977 Hz, δF) -5.3, J_{P-F} = 970 Hz (45%) [1].

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