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N, N-DIETHYLAMIDOFLUOROCHLOROPHOSPHITE

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We are the first to show that O-methyl-N,N-diethylaminochlorophosphite (I) reacts with zinc fluoride to form N,N-diethylaminofluorochlorophosphite (II), which is converted by the action of antimony trifluoride into N,N-diethylaminodifluorophosphite (III).



Fluorochlorophosphite (II) is a thermally stable liquid which fumes in the air. Its structure was supported by 31 P NMR spectroscopy (the spectra were taken on a Bruker HX-90 spectrometer at 36.43 MHz with 85% H₃PO₄ as the external standard).

A sample of 9.73 g chlorophosphite (I) was added dropwise slowly to 2.99 g zinc fluoride. The reaction mass was maintained at 20°C for 2.5 h and fractionated to give 6.37 g (70%) fluorochlorophosphite (II), bp 56-57°C (20 mm), $n_D^{2^\circ}$ 1.4150, $d_4^{2^\circ}$ 1.1447. ³¹P NMR spectrum: δ + 171.1 ppm, J_{PF} = 1167 Hz. Found: C, 30.11; H, 6.18; C1, 22.64; F, 12.16; N, 8.55; P, 19.91%. Calculated for C₄H₁₀FCINP: C, 30.28; H, 6.31; C1, 22.40; F, 11.99; N, 6.31; P, 19.56%.

A sample of 1.5 g antimony trifluoride was added to 3.1 g phosphite (II) and the mixture was maintained for 1 h to give 2.5 g (90%) difluorophosphite (III), bp 43°C (75 mm), n_D^{20} 1.3860, d_4^{20} 1.0357. ³¹P NMR spectrum: δ +145.5 ppm, $J_{\rm PF}$ = 1194 Hz (see the work of Schmutz-ler [1]).

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