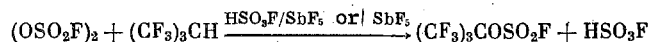


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A large number of examples have been reported for the radical abstraction of hydrogen atoms from fluorinated and nonfluorinated hydrocarbons by peroxydisulfuryl difluoride,  $(\text{OSO}_2\text{F})_2$ , with the formation of  $\text{HSO}_3\text{F}$  and the corresponding esters of fluorosulfuric acid [1]. This reaction begins at from 10 to 20°C. On the other hand, it has not been possible to obtain a reaction between  $(\text{OSO}_2\text{F})_2$  with CH acids [2]. In the present work, we found that one of the strongest CH acids,  $(\text{CF}_3)_3\text{CH}$ , does not react with  $(\text{OSO}_2\text{F})_2$  in a closed volume even at 120-130°C. The addition of  $\text{HSO}_3\text{F}$  to the reaction mixture did not lead to this reaction. However, the reaction begins in the presence of  $\text{SbF}_5$  or a mixture of  $\text{SbF}_5$  and  $\text{HSO}_3\text{F}$  at from 50 to 60°C



Apparently some polarization of the O-O bond occurs in this reaction as well as in the reaction with fluorochlorohydrocarbons [3] in highly acidic media ( $\text{HSO}_3\text{F}/\text{SbF}_5$  or  $\text{SbF}_5$ ) and  $(\text{OSO}_2\text{F})_2$  as an electrophilic reagent.

A mixture of 7.8 g (0.039 mole)  $(\text{OSO}_2\text{F})_2$ , 9.4 g (0.043 mole)  $(\text{CF}_3)_3\text{CH}$ , and 1 g (0.0046 mole)  $\text{SbF}_5$  was heated in a sealed glass ampule for 2 h at 60-90°C. Fractionation of the reaction mass gave 11.2 g (89.6%) tert-perfluorobutyl fluorosulfate bp 69-70°C,  $d_4^{26}$  1.761,  $n_D^{26}$  1.28. Found: C 15.31; F 60.14; S 9.83%. Calculated for  $\text{C}_4\text{F}_{10}\text{O}_3\text{S}$ : C 15.09; F 59.75; S 10.06%.  $^{19}\text{F}$  NMR spectrum (ppm, relative to  $\text{CF}_3\text{CO}_2\text{H}$ ): -11.8 s ( $3\text{CF}_3$ ), -125.4 s (SF).

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