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CHLOROFLUOROANTIMONATES

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Chlorofluoroanions of antimony(V) have been reported by a number of workers [1,2] but structural characterisation has been seldom successful: on the basis of i.r. spectroscopy the anion in $\text{NaSbCl}_4\text{F}_2$ has cis-geometry [2]. Adams and Downs [3] have prepared $\text{Et}_4\text{N}(\text{SbBr}_2\text{F}_4)$ and proposed cis-geometry for this also. Nuclear (^{19}F) magnetic resonance spectroscopy has been attempted by Il'in *et al.* [4] but the interpretation is unconvincing since no Sb-F coupling was observed. We have now shown that anhydrous HF converts SbCl_6^- to SbF_6^- via $\text{fac-SbCl}_3\text{F}_3^-$, $\text{cis-SbCl}_2\text{F}_4^-$ and SbClF_5^- . Unambiguous evidence for the two cis-isomers has been obtained from a combination of ^{121}Sb , ^{123}Sb and ^{19}F n.m.r. spectroscopy of the compounds in Me_2CO or MeCN . Evidence from i.r. and Raman spectroscopy will also be presented as will a crystal structure determination of $\text{Et}_4\text{N}(\text{cis-SbCl}_2\text{F}_4)$.

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