

## RHENIUM NITRIDE TETRAFLUORIDE AND RELATED COMPOUNDS

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Azidotrimethylsilane,  $\text{Me}_3\text{SiN}_3$ , reacts with  $\text{ReF}_6$  at  $-50^\circ$  in Genetron 113 (1,1,2-trichlorotrifluoroethane) to give, after suitable treatment, the nitrile fluoride  $\text{ReNF}_4$  (10%) and a black solid (I). (I) yields  $\text{ReF}_5(\text{NCl})$  with  $\text{ClF}_3$  and  $\text{ReF}_5(\text{NF})$  with  $\text{XeF}_2$  in Genetron 113.  $\text{ReNF}_4$  is a very reactive pale yellow crystalline solid which yields  $\text{NH}_3$  with  $\text{OH}^-$  aq.  $\text{ReF}_5(\text{NCl})$  (purple) and  $\text{ReF}_5(\text{NF})$  (orange) both have nearly linear Re-N-X groups; the nitrogen halogen bond lengths (N-Cl =  $1.56\text{\AA}$ ; N-F =  $1.26\text{\AA}$ ) are short. The  $^{19}\text{F}$  n.m.r. spectrum of  $\text{ReF}_5(\text{NCl})$  at  $-10^\circ$  in  $\text{ClF}_3$  solution shows a well resolved doublet (90Hz, + 180.4 p.p.m.) and a quintet (+ 163.5 p.p.m.) relative to  $\text{CFCl}_3$ .