

FLUOROSULFATE DERIVATIVES OF MOLYBDENUM

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There appear to be no stable mononuclear, binary fluorosulfates of the type $\text{Mo}(\text{SO}_3\text{F})_n$, and attempts to oxidize molybdenum metal by oxidation with bis(fluorosulfonyl)peroxide, $\text{S}_2\text{O}_6\text{F}_2$, in the presence or absence of HSO_3F , result in the formation of Mo(IV) oxyfluorosulfates--the well known $\text{MoO}_2(\text{SO}_3\text{F})_2$, and a previously unknown yellow oil of the composition $\text{MoO}(\text{SO}_3\text{F})_4$. The characterization of this material, and three new different routes to molybdenum fluorosulfate derivatives will be discussed:

- (i) The oxidation of MoF_5 is found to yield $\text{MoF}_5(\text{SO}_3\text{F})$ as a colourless, highly volatile liquid. This compound is characterized by mass spectrometry, ^{19}F - and ^{95}Mo -NMR, and IR and Raman spectroscopy, and found to display interesting spectroscopic properties.
- (ii) The metal oxidation by $\text{S}_2\text{O}_6\text{F}_2$ in HSO_3F in the presence of CsSO_3F , in the hope of isolating ternary complexes of the composition $\text{Cs}_n[\text{Mo}(\text{SO}_3\text{F})_m]$ with $n = 1$ or 2 and $m = 6-8$.
- (iii) The solvolysis of the binuclear $\text{Mo}_2(\text{CH}_3\text{CO}_2)_4$ in HSO_3F resulting in the formation of $\text{Mo}_2(\text{SO}_3\text{F})_4$, which is characterized by vibrational spectroscopy and NMR.