

LOW TEMPERATURE FLUORINATION OF SULPHUR COMPOUNDS WITH ELEMENTAL FLUORINE

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Low temperature fluorination technique is adopted for fluorination of the following sulphur compounds in freon-11 medium (1) Sulphur dioxide (2) Thionyl chloride (3) Sulphuryl chloride (4) Tetrasulphur tetra nitride and (5) Sulphur bromide. All the compounds undergo oxidative fluorination to give rise to sulphur-fluorine compounds except sulphuryl chloride which resists fluorination. Sulphuryl chloride thus behaves as a good solvent medium for fluorination of other reactive compounds like elemental sulphur. Details of the experimental procedures adopted and the identification of the products will be presented.

ELECTROLYTIC PREPARATION OF BIS(FLUOROSULFURYL) PEROXIDE

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One of the known methods for producing bis (fluorosulfonyl) peroxide is electrolysis under vacuum at about -23°C of a dilute solution of potassium fluorosulfate in fluorosulfuric acid. This process has now been improved by increasing the concentration of the dissolved salt, thereby increasing the electrical conductivity and lowering the vapor pressure of the electrolyte. Both the rate and the current efficiency of production of $\text{S}_2\text{O}_8\text{F}_2$ have been increased and loss of HSO_3F by evaporation has been reduced. A current efficiency of about 92% has been found for a cell operating at about -8°C with platinum electrodes in an electrolyte containing about three moles of HSO_3F per mole of KSO_3F . Potassium fluorosulfate combines with fluorosulfuric acid to form the compound, $\text{KSO}_3\text{F} \cdot 3\text{HSO}_3\text{F}$, which melts at -10°C . The formation of compounds having compositions $\text{KSO}_3\text{F} \cdot x\text{HSO}_3\text{F}$ ($x=3-5$) are also indicated and will be discussed.