

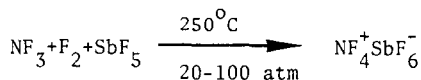
SHORT COMMUNICATION

Simplified Synthesis of $\text{NF}_4^+\text{SbF}_6^-$

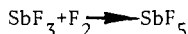
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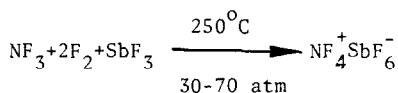
In NF_4^+ chemistry, the $\text{NF}_4^+\text{SbF}_6^-$ salt plays a key role. It is most amenable to large scale production and serves as a starting material for the metathetical syntheses of numerous other NF_4^+ salts [1 - 4]. The most convenient previously reported [5] method involved the reaction of SbF_5 with an excess of NF_3 and F_2 at elevated temperature and pressure according to:



In view of its appreciable cost and its detrimental physical and chemical properties, it was desirable to replace SbF_5 by a starting material which is cheaper, more readily available and easier to handle. Since it is well known [6] that, under conditions similar to those of the above $\text{NF}_4^+\text{SbF}_6^-$ synthesis, SbF_3 can be fluorinated by F_2 to SbF_5 ,



a direct synthesis of $\text{NF}_4^+\text{SbF}_6^-$ from SbF_3 , F_2 and NF_3 was logical. The possible combination of the two steps was experimentally verified, as shown by the following equation:



Although no efforts were made to maximize all the reaction parameters, the high yield and purity of the thus obtained $\text{NF}_4^+\text{SbF}_6^-$ demonstrates the feasibility of this simplified synthesis.

EXPERIMENTAL

A prepassivated (with ClF_3) 95 ml monel cylinder was loaded in the dry Nitrogen atmosphere of a glove box with SbF_3 (31 mmol). The cylinder was connected to a metal vacuum system, evacuated, and charged at -196°C with NF_3 (65 mmol) and F_2 (98 mmol). The cylinder was heated for five days to 250°C . The excess of unreacted NF_3 and F_2 was pumped off at ambient temperature, leaving behind a white crystalline residue (10 g, weight expected for 31 mmol of $\text{NF}_4\text{SbF}_6 = 10.1$ g). Based on its infrared and Raman spectra and its chemical analysis, this solid consisted of high purity $\text{NF}_4^+\text{SbF}_6^-$. It did not contain any detectable amounts of polyantimonate [7] salts.

ACKNOWLEDGEMENTS

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