Journal of Fluorine Chemistry, 15 (1980) 83–84

Received: October 3, 1979

SHORT COMMUNICATION

Simplified Synthesis of $NF_4^+SbF_6^-$

WILLIAM W. WILSON AND KARL O. CHRISTE*

Rocketdyne Division of Rockwell International, Canoga Park, Calif. 91304, (USA)

In NF_4^+ chemistry, the $NF_4^+SbF_6^-$ 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:

$$NF_3+F_2+SbF_5 \xrightarrow[20\text{-}100\ atm]{250^oC} NF_4^+SbF_6^-$$

In view of its appreciable cost and its detremental 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 NF_4SbF_6 synthesis, SbF_3 can be fluorinated by F_2 to SbF_5,

$$SbF_3+F_2 \longrightarrow SbF_5$$

a direct synthesis of $NF_4^+SbF_6^-$ 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:

$$NF_3+2F_2+SbF_3 \xrightarrow[30\text{-}70\ atm]{250^oC} NF_4^+SbF_6^-$$

Although no efforts were made to maximize all the reaction parameters, the high yield and purity of the thus obtained $NF_4^+SbF_6^-$ 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_5 (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 crystallinic residue (10 g, weight expected for 31 mmol of NF_4SbF_6=10.1 g). Based on its infrared and Raman stectra and its chemical analysis, this solid consisted of high purity $NF_4^+SbF_6^-$. It did not contain any detectable amounts of polyantimonate [7] salts.

ACKNOWLEDGEMENTS

We are indebted to Drs. C. J. Schack and L. R. Grant and Mr. R. D. Wilson for helpful discussions and to the U. S. Army Research Office for financial support.

REFERENCES

1 K. O. Christe, C. J. Schack and R. D. Wilson, Inorg. Chem., 16 (1977) 849.

2 K. O. Christe and C. J. Schack, Inorg. Chem., 16 (1977) 353.

3 K. O. Christe, Inorg. Chem., 16 (1977) 2238.

4 K. O. Christe, W. W. Wilson and C. J. Schack, J. Fluorine Chem., 11 (1978) 71.

5 K. O. Christe, C. J. Schack and R. D. Wilson, J. Fluorine Chem., 8 (1976) 541.

6 A. A. Woolf and N. N. Greenwood, J. Chem. Soc., (1950) 2200.

7 K. O. Christe, C. J. Schack and R. D. Wilson, Inorg. Chem., 15 (1976) 1275.