The Crystal Structure of the Adduct of Niobium Pentafluoride and Antimony Pentafluoride

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Summary Niobium pentafluoride forms with antimony pentafluoride a 1 : 1 adduct, the structure of which has been shown to be an endless chain arrangement, with a major contribution from the ionic form $[NbF_4]^+[SbF_6]^-$.

A RECENT n.m.r. study of niobium pentafluoride in liquid antimony pentafluoride concluded that dissolution rather than reaction occurred, although a fairly strong interaction was suggested.[1]

We have observed that the reaction of a slight excess of niobium pentafluoride with antimony pentafluoride produces a solid adduct, m.p. 60°, which can be removed from the excess of niobium pentafluoride by vacuum sublimation. Single crystals of the compound were isolated in evacuated Pyrex glass capillaries for *X*-ray examination.

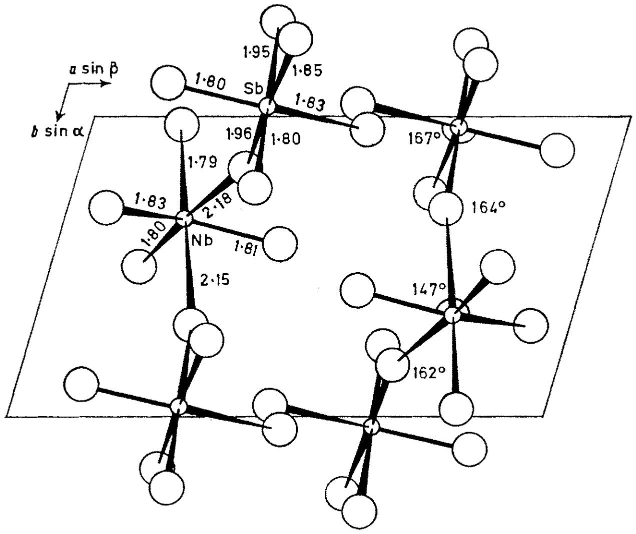

FIGURE. *Projection of the structure down* [001], *estimated standard deviations are* ±0·02 Å *and* ±0·8°

Crystal data were determined photographically using Weissenberg and precession techniques with Cu-K_α and Mo-K_α radiation: $NbSbF_{10}$; $M = 405$, triclinic, $a = 5{\cdot}64(1)$, $b = 9{\cdot}58(1)$, $c = 7{\cdot}38(1)$ Å, $\alpha = 87{\cdot}2(3)$, $\beta = 99{\cdot}9(3)$, $\gamma = 106{\cdot}5(3)°$, $U = 377$ Å^3. Space group $P\bar{1}(C_i^1$, No. 2).

The cell volume is consistent with $Z = 2$, since with 20 fluorine atoms in the unit cell the volume per fluorine atom is 18·8Å^3, similar to the value of 17·6Å^3 in niobium pentafluoride itself.[2] Intensity data were determined photometrically from integrated Weissenberg films, giving 903 independent reflections. The heavy-atom positions were derived from the three-dimensional Patterson function, and those of the fluorine atoms from a subsequent electron-density map. Full-matrix least-squares refinement of positional and isotropic temperature parameters has led to a value of 0·064 for *R*.

The structure, which is shown in the Figure, consists of a zigzag chain of alternating antimony and niobium atoms, linked asymmetrically by *cis*-bridging fluorine atoms. Four terminal fluorine atoms complete a distorted octahedral co-ordination of the metal atoms, the distortion being much more pronounced for niobium than for antimony. This chain arrangement is similar to that formed by vanadium pentafluoride[3] except for the asymmetric fluorine bridges.

Although the distances from either metal atom to terminal fluorine atoms are very similar, the Nb–F (bridge) distance of 2·16Å is significantly greater than the corresponding Sb–F (bridge) of 1·95Å, and the bridging fluorine atoms are obviously more closely associated with antimony than with niobium. Thus a contribution to the structure from the ionic form $[NbF_4]^+[SbF_6]^-$ is indicated. In line with this idea the F–Nb–F angle of 147° (for the fluorine atoms above and below the zigzag chain) can be considered as moving towards the theoretical value of 109° 28′, expected for a tetrahedral configuration for NbF_4^+, from the theoretical value of 180° in an undistorted octahedron.

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[1] T. K. Davies and K. C. Moss, *J. Chem. Soc.* (*A*), 1970, 1054.

[2] A. J. Edwards, *J. Chem. Soc.*, 1964, 3714.

[3] A. J. Edwards and G. R. Jones, *J. Chem. Soc.* (*A*), 1969, 1651.