The light compound was analysed first and the $(P+1)/P$ ratio was determined. Two samples of known composition and the heavy compound were then run. The light reference compound was finally analysed again to verify a constant $(P+1)/P$ ratio. The samples to be determined were then run and the determination was ended by running the two samples of known composition. The composition of the known samples determined by mass spectrometry agreed within 1 % with the calculated values. The reproducibility was better then 0.5 %.

Acknowledgements. The author wishes to thank Prof. Lars Melander and Prof. William H. Saunders, Jr., for many fruitful discussions and for reading the manuscript prior to publication. Many thanks are due to Mrs. Lena Kagevi for technical aid in the determination of the mass spectra. Financial support from *The Swedish Natural Science Research Council* is gratefully acknowledged.

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Received June 5, 1971.

Preparation and Crystal Structures of Various Forms of SbOF

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The heating of mixtures of SbF_3 and Sb_2O_3 in the mole ratio 1:1 in sealed gold or platinum capsules at temperatures between 150 and 450°C results in the formation of four different forms of SbOF. Rapid quenching in ice-cold water from temperatures 350 – 400°C gives a colourless glass. When this material, or a mixture of SbF_3 and Sb_2O_3, is heated for a few hours at 350°C, a crystalline substance is obtained. Its X-ray powder pattern can be indexed in the cubic system, and we define this form as H-SbOF.

H-SbOF transforms at temperatures around 275°C into an orthorhombic form which we name M-SbOF. This form can also be prepared by the direct reaction of SbF_3 and Sb_2O_3. During the hydrolysis of SbF_3 at 150°C, beautiful, rodshaped crystals were formed with a composition analyzed to be SbOF. Heat treatment of the glass, or of a mixture of Sb_2O_3 and SbF_3, at temperatures around 150°C, resulted in the same phase, named L-SbOF.

The crystallographic constants for H-, L-, and M-SbOF are given in Table 1. Single crystal data have been collected for the L- and M-forms, and the crystal structure of the L-form has been refined to an R-factor of 6.5 %.[1] The structure of M-SbOF has also been solved, and refinement is in progress.[2]

The configuration around antimony in L-SbOF is typical for an element having a stereochemically active lone pair, and is

Table 1. Crystallographic constants for the various forms of SbOF.

	L-SbOF	M-SbOF	H-SbOF
Space group	*Pnma* (No. 62)	*Pbca* (No. 61)	
a Å	8.873	11.618	11.72
b Å	4.099	5.616	
c Å	5.483	12.281	
d_{calc}	5.22	5.20	5.17

the same as has been observed in the structures of Sb_2O_4 and $SbNbO_4$,[3] and $SbPO_4$.[4] There are four anions bonded to antimony, and they are arranged in such a way that the lone pair in the equatorial plane completes a trigonal bipyramidal configuration. This arrangement of electron pairs was predicted by Gillespie and Nyholm [5] for five-configuration, and is defined by them as AX_4E, where A is the central atom, X ligand and E unshared electron pair. The distortions of the trigonal bipyramidal configuration that occur are explained by the valence shell electron pair repulsion theory of Gillespie and Nyholm. A stereochemically active lone pair is larger than a bonding pair and takes up more room on the surface of an atom. The bonding pairs are therefore squeezed together and the equatorial angle is greatly reduced from 120°.

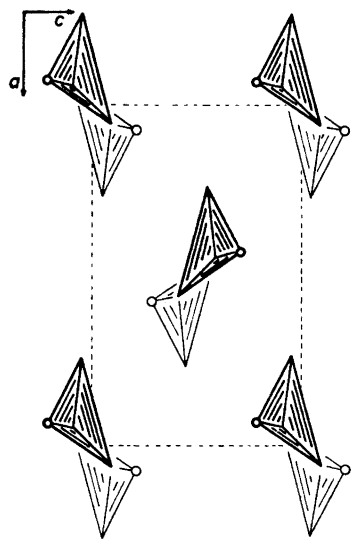

Fig. 1. The structure of L-SbOF projected down the *b* axis. The heavier and lighter polyhedra are *b*/2 apart, as are the Sb-atoms, marked with heavy and light circles. All atoms are on $y=0$ or 1/2.

In L-SbOF the angle for the grouping O – Sb – F situated in the equatorial plane is found to be 96°. In the structure, the polyhedra then edge-share to form endless chains in the *b* direction as shown in Fig. 1. After refinement of the structure it was further assumed that the bridging anions were oxygen, and the terminal fluorine. This reduced the final *R*-factor from 6.6 % to 6.5 %. The distances obtained were for Sb – O 1.99 Å, and for Sb – F 1.95 Å.

The authors are grateful to the *Computer Division of the National Rationalization Agency* for the use of the Univac 1108 Computer in Lund.

This investigation forms a part of a research program sponsored by the *Swedish Natural Science Research Council.*

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Received June 15, 1971.