

## Fluoride Crystal Structures. Part XXII.<sup>1</sup> Chloryl $\mu$ -Fluoro-bis[pentafluoroantimonate(v)]

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The interaction of chlorine, chlorine trifluoride, and antimony pentafluoride produced coloured solutions. After these had been set aside in Pyrex glass apparatus for several months, colourless crystals separated, and some of these were shown by crystallographic analysis to be the title compound. Crystals are monoclinic, space group  $P2_1/n$ ,  $Z = 4$ ,  $a = 7.50(1)$ ,  $b = 10.20(2)$ ,  $c = 13.24(2)$  Å,  $\beta = 94.4(3)^\circ$ . The structure was solved by the heavy-atom method and was refined by three-dimensional least-squares methods to  $R$  0.076 for 971 reflections. Although the molecular geometry is consistent with the ionic formulation  $[\text{ClO}_2]^+[\text{Sb}_2\text{F}_{11}]^-$  there is considerable interaction between the ions through fluorine bridging.

THERE has been considerable controversy over the formation of paramagnetic species in mixtures of chlorine fluorides with antimony pentafluoride. Two different e.s.r. spectra were observed under different conditions and were assigned<sup>2</sup> to the species  $\text{Cl}_2^+$  and  $\text{ClF}^+$ . These assignments were questioned,<sup>3</sup> and the suggestion was made that the species responsible for the spectra were  $\text{Cl}_2\text{O}^+$  and  $\text{ClOF}^+$ .

Careful preparative work in a passivated stainless

<sup>1</sup> Part XXI, A. J. Edwards and P. Taylor, *J.C.S. Dalton*, 1973, 2150.

<sup>2</sup> G. A. Olah and M. B. Comisarow, *J. Amer. Chem. Soc.*, 1968, **90**, 5033; 1969, **91**, 2172.

steel-Teflon vacuum line showed that no e.s.r. signals were obtained under these conditions, but that spectra could be generated when the reactants were handled in glass apparatus.<sup>4</sup> Thus the assignment of the paramagnetic materials as oxygen-containing species was supported. The observation was also made<sup>5</sup> that the addition of a small amount of water to a sample of  $[\text{ClF}_2]^+[\text{SbF}_6]^-$  in  $\text{SbF}_5$  enhanced the spectrum assigned

<sup>3</sup> R. S. Eachus, T. P. Sleight, and M. C. R. Symons, *Nature*, 1969, **222**, 769.

<sup>4</sup> K. O. Christe and J. S. Muirhead, *J. Amer. Chem. Soc.*, 1970, **91**, 7777.

<sup>5</sup> R. J. Gillespie and M. J. Morton, *Inorg. Chem.*, 1972, **11**, 591.

to  $\text{ClOF}^+$ , and  $\text{ClO}_2\text{F}^+$  was suggested as being a possible alternative assignment for this species.

We have obtained a yellow-orange, involatile liquid by mixing chlorine, chlorine trifluoride, and antimony pentafluoride in Pyrex glass apparatus at room temperature. E.s.r. spectra identical to those assigned previously<sup>2</sup> to  $\text{Cl}_2\text{O}^+$  and  $\text{ClOF}^+$  were obtained at room temperature and 80 °C respectively. Attempts to obtain crystals from the coloured liquid were initially unsuccessful, but after several months colourless crystals grew from the solution. Crystal-structure analysis has shown some of these crystals to be the title compound, and the formation of the oxychlorine cation supports the formulation of the paramagnetic species as  $\text{Cl}_2\text{O}^+$  and  $\text{ClOF}^+$  or  $\text{ClO}_2\text{F}^+$ .

#### EXPERIMENTAL

Chlorine trifluoride and antimony pentafluoride were prepared by the fluorination of the elements at ca. 250 °C. Chlorine (dried over phosphorus pentoxide) was condensed on to antimony pentafluoride in a rigorously dried Pyrex glass apparatus. On warming, the chlorine formed a yellow liquid layer above the  $\text{SbF}_5$  and then evaporated without reaction. The addition of small amounts of chlorine trifluoride to the mixture of chlorine and antimony pentafluoride, and warming to ca. 80°, caused a reaction involving the absorption of gaseous chlorine, and formation of an homogeneous liquid phase. This liquid had an extremely low vapour pressure at room temperature and could be distilled unchanged under vacuum at elevated temperatures.

Samples of the liquid were distilled into thin-walled Pyrex capillaries and sealed under vacuum. After several months, the colour of the liquid darkened, and colourless crystals formed. A very volatile colourless compound was also formed, which, from its properties, was assumed to be silicon tetrafluoride, formed by reaction of the glass.

Single crystals were sealed in small sections of capillary tube and investigated by X-ray methods. Four different types of crystal have been found, but only one of these has been fully characterised (with the results reported here) owing to the difficulty of obtaining single crystals suitable for data collection.

*Crystal Data.*— $\text{ClF}_{11}\text{O}_2\text{Sb}_2$ ,  $M = 520$ , Monoclinic,  $a = 7.50(1)$ ,  $b = 10.20(2)$ ,  $c = 13.24(2)$  Å,  $\beta = 94.4(3)^\circ$ ,  $U = 1010$  Å<sup>3</sup>,  $Z = 4$ ,  $D_c = 3.42$ ,  $F(000) = 936$ . Space group  $P2_1/n$  ( $C_{2h}^2$ , No. 14 in a non-standard setting) from systematic absences:  $0k0$  when  $k \neq 2n$  and  $h0l$  when  $h + l \neq 2n$ .  $\text{Cu-K}\alpha$  ( $\lambda = 1.5418$  Å) and  $\text{Mo-K}\alpha$  ( $\lambda = 0.7107$ ;  $\mu = 62.3$  cm<sup>-1</sup>) radiations. Single-crystal precession and Weissenberg photographs.

The density of the crystals was not measured but  $Z = 4$  gives a volume per light atom of 19.4 Å<sup>3</sup>, consistent with the corresponding volume in similar compounds<sup>1</sup> of 18–20 Å<sup>3</sup>.

*Structure Determination.*—Integrated intensities were collected about the  $b$  axis (layers  $h0-6l$ ) by use of  $\text{Mo-K}\alpha$  radiation and a Nonius integrating camera. The relative intensities of 971 independent reflections were measured with a photometer of similar design to that described by Jeffery,<sup>6</sup> and were corrected for Lorentz and polarisation factors.

The structure was solved by conventional heavy-atom

\* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue (items less than 10 pp. are sent as full-size copies).

<sup>6</sup> J. W. Jeffery, *J. Sci. Instr.*, 1963, **40**, 494.

methods.<sup>7</sup> Refinement of the positional and isotropic temperature parameters, and layer scale-factors was achieved by full-matrix least-squares methods with all light atoms assigned fluorine scattering factors. The function  $\Sigma w(|F_o| - |F_c|)^2$  was minimised, at first with unit weights and in the final stages of the refinement with  $\sqrt{w} = 1$  when  $|F_o| < 61$ , and  $\sqrt{w} = 61/|F_o|$  when  $|F_o| > 61$ . When refinement was almost complete (at  $R$  0.18) an absorption correction was applied by use of the computer programme ABSCOR. The crystal shape could only be defined approximately (as a rectangular prism with dimensions  $0.5 \times 0.5 \times 0.1$  mm) since it was coated with liquid, but after application of the correction and further cycles of refinement, the final  $R$  was 0.076. In these last cycles oxygen scattering factors were assigned to the two close neighbours of the chlorine atom, but this gave negligible changes in  $R$  and in their temperature factors. The final parameter shifts were  $< 0.1\sigma$  and an analysis of the variation of  $w\Delta^2$  with increasing  $\sin \theta/\lambda$  and with increasing fractions of  $|F_o|$  was satisfactory. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 21039 (7 pp., 1 microfiche),\* and the final positional parameters and isotropic temperature factors, with their estimated standard deviations, in Table 1. Interatomic distances and angles are given in Table 2.

TABLE 1

Final atomic positional and thermal parameters, with estimated standard deviations in parentheses

Atom	$x/a$	$y/b$	$z/c$	$B/\text{Å}^2$
Cl	-0.3273(9)	0.1666(7)	0.3944(6)	2.71(15)
O(1)	-0.3266(27)	0.0527(22)	0.4357(19)	3.67(42)
O(2)	-0.1925(30)	0.2501(23)	0.4116(18)	4.34(48)
Sb(1)	0.1626(2)	-0.0370(2)	0.2732(1)	2.15(4)
Sb(2)	-0.1801(2)	0.1622(2)	0.1044(1)	2.27(4)
F(1)	0.2901(25)	-0.0999(21)	0.1741(16)	4.42(39)
F(2)	0.2539(24)	-0.1512(18)	0.3679(15)	3.90(35)
F(3)	-0.0167(22)	-0.1470(18)	0.2308(14)	3.48(33)
F(4)	0.0220(25)	0.0379(20)	0.3573(16)	4.51(39)
F(5)	0.0475(21)	0.0911(17)	0.1694(13)	3.03(29)
F(6)	0.3298(26)	0.0897(22)	0.3090(16)	4.63(41)
F(7)	-0.2823(23)	0.0886(18)	0.2110(14)	3.61(33)
F(8)	-0.1994(27)	0.0014(21)	0.0398(17)	4.48(43)
F(9)	-0.1249(26)	0.3100(20)	0.1820(15)	4.31(39)
F(10)	-0.0468(26)	0.2299(21)	0.0092(15)	4.62(41)
F(11)	-0.3856(24)	0.2271(19)	0.0464(15)	3.99(36)

In the non-standard setting of the space group the equivalent positions are:  $x, y, z$ ;  $\bar{x}, \bar{y}, \bar{z}$ ;  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ;  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ .

#### DISCUSSION

Since oxygen and fluorine cannot be distinguished by X-ray methods, in the presence of heavy atoms, we have assigned the identity of the compound in the present study from a comparison of molecular dimensions and stereochemistry with those for similar compounds.  $[\text{ClF}_2]^+[\text{Sb}_2\text{F}_{11}]^-$  is a possible formulation for the present compound based on the starting materials for the preparation, and has not been characterised. The dimensions of the  $\text{ClF}_2^+$  cation have been established from crystal-structure determinations<sup>8,9</sup> on  $[\text{ClF}_2]^+[\text{SbF}_6]^-$  and  $[\text{ClF}_2]^+[\text{AsF}_6]^-$ , and a useful comparison can be made with the present results.

<sup>7</sup> 'International Tables for X-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, p. 202.

<sup>8</sup> A. J. Edwards and R. J. C. Sills, *J. Chem. Soc. (A)*, 1970, 2697.

<sup>9</sup> H. Lynton and J. Passmore, *Canad. J. Chem.*, 1971, **49**, 2539.

TABLE 2

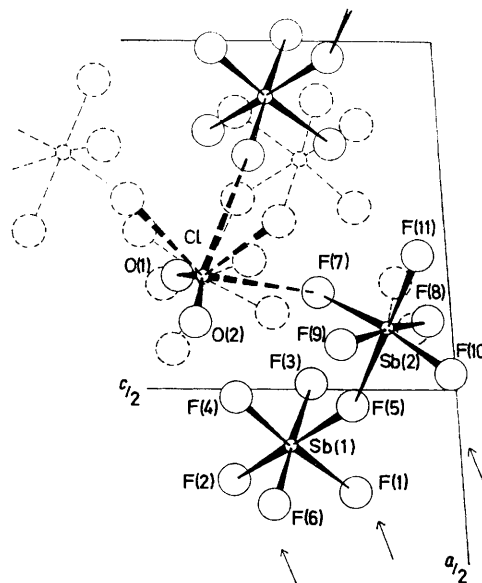
Interatomic distances (Å) and angles (°), with estimated deviations in parentheses

(a) Distances			
Cl-O(1)	1.28(3)	Cl-O(2)	1.33(3)
Sb(1)-F(1)	1.80(2)	Sb(2)-F(5)	1.99(2)
Sb(1)-F(2)	1.81(2)	Sb(2)-F(7)	1.82(2)
Sb(1)-F(3)	1.81(2)	Sb(2)-F(8)	1.85(2)
Sb(1)-F(4)	1.77(2)	Sb(2)-F(9)	1.85(2)
Sb(1)-F(5)	2.04(2)	Sb(2)-F(10)	1.80(2)
Sb(1)-F(6)	1.84(2)	Sb(2)-F(11)	1.80(2)
F(1) ... F(2)	2.65(3)	F(7) ... F(9)	2.59(3)
F(1) ... F(6)	2.63(3)	F(7) ... F(5)	2.57(3)
F(1) ... F(3)	2.52(3)	F(7) ... F(8)	2.55(3)
F(1) ... F(5)	2.66(3)	F(7) ... F(11)	2.66(3)
F(4) ... F(2)	2.59(3)	F(10) ... F(9)	2.54(3)
F(4) ... F(6)	2.50(3)	F(10) ... F(5)	2.60(3)
F(4) ... F(3)	2.53(3)	F(10) ... F(8)	2.64(3)
F(4) ... F(5)	2.57(3)	F(10) ... F(11)	2.62(3)
F(2) ... F(6)	2.65(3)	F(5) ... F(8)	2.59(3)
F(2) ... F(3)	2.62(3)	F(5) ... F(9)	2.59(3)
F(5) ... F(6)	2.70(3)	F(11) ... F(8)	2.70(3)
F(5) ... F(3)	2.62(3)	F(11) ... F(9)	2.69(3)
O(1) ... O(2)	2.28(3)	Cl ... F(7)	2.60(2)
Cl ... F(10 <sup>I</sup> )	2.55(2)	Cl ... F(3 <sup>II</sup> )	2.73(2)
Cl ... F(6 <sup>III</sup> )	2.84(2)		
(b) Angles			
F(2)-Sb(1)-F(1)	94.7(9)	F(11)-Sb(2)-F(7)	94.9(8)
F(2)-Sb(1)-F(3)	92.8(9)	F(11)-Sb(2)-F(8)	95.5(9)
F(2)-Sb(1)-F(4)	93.1(9)	F(11)-Sb(2)-F(9)	94.9(9)
F(2)-Sb(1)-F(6)	93.4(9)	F(11)-Sb(2)-F(10)	93.6(9)
F(1)-Sb(1)-F(3)	88.6(9)	F(7)-Sb(2)-F(8)	88.3(9)
F(1)-Sb(1)-F(6)	92.8(9)	F(7)-Sb(2)-F(9)	89.7(8)
F(4)-Sb(1)-F(3)	89.9(9)	F(10)-Sb(2)-F(8)	92.6(9)
F(4)-Sb(1)-F(6)	87.8(9)	F(10)-Sb(2)-F(9)	87.9(9)
F(5)-Sb(1)-F(1)	87.6(8)	F(5)-Sb(2)-F(7)	85.0(7)
F(5)-Sb(1)-F(3)	85.5(7)	F(5)-Sb(2)-F(8)	84.9(8)
F(5)-Sb(1)-F(4)	84.6(8)	F(5)-Sb(2)-F(9)	84.7(8)
F(5)-Sb(1)-F(6)	88.2(8)	F(5)-Sb(2)-F(10)	86.5(8)
Sb(1)-F(5)-Sb(2)	146.1(9)	O(1)-Cl-O(2)	121.9(1.5)
O(1)-Cl-F(7)	97.1(1.2)	O(2)-Cl-F(10 <sup>I</sup> )	101.8(1.2)
O(1)-Cl-F(10 <sup>I</sup> )	95.9(1.2)	O(2)-Cl-F(10 <sup>II</sup> )	98.6(1.2)
O(1)-Cl-F(6 <sup>III</sup> )	83.9(1.1)	O(2)-Cl-F(3 <sup>II</sup> )	86.7(1.1)
O(1)-Cl-F(3 <sup>III</sup> )	151.3(1.1)	O(2)-Cl-F(6 <sup>III</sup> )	154.1(1.2)
F(7)-Cl-F(6 <sup>III</sup> )	73.8(6)	F(10 <sup>I</sup> )-Cl-F(6 <sup>III</sup> )	75.3(6)
F(7)-Cl-F(3 <sup>III</sup> )	74.1(6)	F(10 <sup>I</sup> )-Cl-F(3 <sup>II</sup> )	78.9(6)
F(7)-Cl-F(10 <sup>I</sup> )	144.8(7)	F(6 <sup>III</sup> )-Cl-F(3 <sup>II</sup> )	67.5(6)
Cl-F(7)-Sb(2)	133.7(9)	Cl-F(10 <sup>I</sup> )-Sb(2 <sup>I</sup> )	172.2(1.1)
Cl-F(6 <sup>III</sup> )-Sb(1 <sup>III</sup> )	151.2(1.1)	Cl-F(3 <sup>II</sup> )-Sb(1 <sup>II</sup> )	153.7(9)
(c) Contacts < 3.5 Å			
Cl ... F(4)	3.00(2)	F(7) ... F(3)	3.12(2)
F(7) ... F(4)	2.93(3)	F(8) ... F(3)	3.17(3)
O(1) ... F(4)	2.89(3)	O(1) ... F(7)	3.04(3)
O(2) ... F(4)	2.82(3)	O(2) ... F(7)	3.16(3)
O(2) ... F(9)	3.18(3)	O(1) ... F(10 <sup>I</sup> )	2.97(3)
O(2) ... F(10 <sup>I</sup> )	3.05(3)	F(2) ... F(11 <sup>II</sup> )	3.31(3)
F(3) ... F(7 <sup>II</sup> )	3.21(3)	F(3) ... F(9 <sup>II</sup> )	3.04(3)
F(3) ... F(11 <sup>II</sup> )	3.35(3)	F(3) ... O(2 <sup>II</sup> )	2.96(3)
F(7) ... F(9 <sup>II</sup> )	3.27(3)	F(8) ... O(2 <sup>II</sup> )	2.78(3)
O(1) ... F(9 <sup>II</sup> )	2.93(3)	F(7) ... F(6 <sup>III</sup> )	3.27(3)
O(1) ... F(6 <sup>III</sup> )	2.99(3)	F(1) ... F(8 <sup>IV</sup> )	3.03(3)
F(1) ... F(10 <sup>IV</sup> )	3.21(3)	F(1) ... F(11 <sup>IV</sup> )	3.32(3)
F(3) ... F(10 <sup>IV</sup> )	3.36(3)	F(5) ... F(8 <sup>IV</sup> )	3.22(3)
F(8) ... F(8 <sup>IV</sup> )	3.25(4)	F(8) ... F(10 <sup>IV</sup> )	3.10(3)
F(4) ... F(11 <sup>V</sup> )	3.50(3)	F(6) ... F(10 <sup>V</sup> )	3.30(3)
O(2) ... F(11 <sup>V</sup> )	2.81(3)	F(1) ... F(6 <sup>VI</sup> )	3.30(3)
F(1) ... F(9 <sup>VI</sup> )	3.17(3)	F(2) ... F(5 <sup>VI</sup> )	3.08(3)
F(2) ... F(9 <sup>VI</sup> )	2.94(3)	F(2) ... F(10 <sup>VI</sup> )	2.90(3)
F(3) ... F(6 <sup>VI</sup> )	3.09(3)		

Roman numerals as superscripts refer to atoms in the positions:

I	$-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$	IV	$-x, -y, -z$
II	$-\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$	V	$\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$
III	$-1 + x, y, z$	VI	$\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$

In the atomic arrangement shown in the Figure, the nearest light atom neighbours to the chlorine and antimony atoms define the  $\text{ClO}_2^+$  and  $\text{Sb}_2\text{F}_{11}^-$  ions. The  $\text{ClO}_2^+$  ion is V-shaped, with a mean Cl-O distance of 1.31 Å and an O-Cl-O angle of 122°. This distance is much shorter than the mean Cl-F distance (1.54 Å) in the  $\text{ClF}_2^+$  ion, as expected for a multiple bond to oxygen compared with a single bond to fluorine. The O-Cl-O angle of 122° is much larger than the F-Cl-F angle



Projection of the structure down [010] showing the atom numbering and the co-ordination of the chlorine atom. Arrows indicate planes of approximately close-packed atoms

(103°) and the value is similar to that (114°) predicted for  $\text{ClO}_2^+$  from spectral measurements<sup>10</sup> on  $[\text{ClO}_2]^+[\text{AsF}_6]^-$ .

The characteristic co-ordination of chlorine in the  $\text{ClF}_2^+$  ion is completed by two long contacts to fluorine atoms in the anions of the compound, to give a distorted square-planar arrangement.<sup>8,9</sup> The co-ordination of the chlorine atom in the present structure is quite different, with four interionic fluorine bridges giving a more complex arrangement (see Figure and later discussion). We therefore conclude that the structure analysis has established the existence of  $[\text{ClO}_2]^+[\text{Sb}_2\text{F}_{11}]^-$ . Salts containing the chloryl cation have previously been characterised, but were prepared directly from the interaction of  $\text{ClO}_2\text{F}$  and the appropriate fluoride ion acceptor molecule.<sup>10,11</sup>

On valence-shell electron-pair repulsion (VSEPR) theory an angular configuration is predicted for  $\text{ClO}_2^+$ , with a non-bonding pair of electrons completing a trigonal planar co-ordination around the chlorine atom. Repulsions between the multiple bonds to oxygen and the non-bonding pair of electrons appear to be similar, as the O-Cl-O angle of 122° is not significantly different from the 120° predicted for a symmetrical trigonal planar arrangement. The Cl-O distance is extremely short,

<sup>10</sup> K. O. Christe, C. J. Schack, D. Pilipovich, and W. Sawodny, *Inorg. Chem.*, 1969, **8**, 2489.

<sup>11</sup> C. J. Schack and D. Pilipovich, *Inorg. Chem.*, 1970, **9**, 387; H. A. Carter and F. Aubke, *Canad. J. Chem.*, 1970, **48**, 3466.

although the estimated standard deviation is large. The isoelectronic  $\text{SO}_2$  molecule has S-O 1.432 Å, and the  $\text{ClO}_2$  molecule has Cl-O 1.475 Å. The Cl-O distance in the cation would be expected to be shorter than in  $\text{ClO}_2$ , because of the removal of the odd electron, and because of the positive charge. The  $\text{OSF}_3^+$  cation<sup>12</sup> in  $[\text{OSF}_3]^+[\text{AsF}_6]^-$  has S-O 1.35 Å, and the difference between that and the present Cl-O distance is not statistically significant at the level of accuracy of our structure determination.

Apart from the two directly bonded oxygen atoms, the chlorine atom has four fluorine atom neighbours at 2.55, 2.60, 2.73, and 2.84 Å. The weak bridging interactions to the chlorine atom in  $[\text{ClF}_2]^+[\text{SbF}_6]^-$  are 2.33 and 2.43 Å, so that the interactions for  $\text{ClO}_2^+$  are comparable, at least for the two shorter contacts. These two contacts give the chlorine atom a distorted tetrahedral co-ordination by two oxygen and two fluorine atoms. This can be considered to be based on a distorted trigonal bipyramidal arrangement, if the non-bonding pair of electrons is included.

The two oxygen atoms and the non-bonding pair are in equatorial positions, with O-Cl-O 122° (theoretical 120°), and the two fluorine atoms in axial positions, with F...Cl...F 145° (theoretical 180°). This arrangement is stereochemically quite similar to that of the arsenic atom in the structure<sup>13</sup> of  $\text{AsF}_3, \text{SbF}_5$ . In that structure there is a large contribution from the ionic form  $[\text{AsF}_2]^+[\text{SbF}_6]^-$ , and the two nearest interionic contacts to fluorine atoms complete a similar arrangement, based on a trigonal bipyramidal stereochemistry. The

change from two fluorine to two oxygen atoms in the equatorial positions introduces a larger repulsive effect on the axial fluorine atoms, and causes them to be displaced to the opposite side of the central atom. Thus the value of the F...As...F angle (164°) becomes 215° for F...Cl...F. The two longer Cl...F contacts (2.73 and 2.84 Å), which complete the co-ordination of the chlorine atom, give a greatly distorted octahedral arrangement around chlorine, which parallels the arrangement in the arsenic compound where there are two As...F contacts of 2.73 Å.

The Sb-F-Sb angle of 146° in the  $\text{Sb}_2\text{F}_{11}^-$  ion is smaller than that of 161° in the anion<sup>1</sup> in  $[\text{TeF}_3]^+[\text{Sb}_2\text{F}_{11}]^-$ , and the configuration is closer to staggered than eclipsed. Since it can be seen from the Figure that there is an approach to close packing of the light atoms in the structure, this bridge angle may be indicative of an approximately hexagonal close-packed, rather than cubic close-packed, arrangement.

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[4/301 Received, 15th February, 1974]

<sup>12</sup> C. Lau, H. Lynton, J. Passmore, and P. Y. Shiew, *J.C.S. Dalton*, 1973, 2535.

<sup>13</sup> A. J. Edwards and R. J. C. Sills, *J. Chem. Soc. (A)*, 1971, 942.