structures que nous avons décrites: $In_{18}Sn_7S_{34}$ (Likforman *et al.*, 1987) et $In_{14}Sn_5S_{26}$ (Likforman *et al.*, 1988) un semblable phénomène existe, les structures diffèrent les unes des autres par la nature de l'assemblage de ces rubans de sept octaèdres.

Un autre trait commun à ces trois structures est l'environnement des atomes d'étain divalent qui se trouvent toujours à l'intérieur de prismes dicapés situés entre les rubans d'octaèdres InS_6, certains de ces prismes n'étant occupés que partiellement.

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Structure of Hexaammonium Dodecafluorotrisulfatotetraantimonate(III) at 300 K

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Abstract. $[NH_4]_6[Sb_4F_{12}(SO_4)_3]$, $M_r = 1111{\cdot}4$, trigonal, $P3$, $a = 17{\cdot}07\,(1)$, $c = 7{\cdot}515\,(8)$ Å, $V = 1896\,(1)$ Å^3, $Z = 3$, $D_m = 2{\cdot}90$, $D_x = 2{\cdot}92$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 46{\cdot}7$ cm^{-1}, $F(000) = 1566$, $T = 300$ K, $R = 0{\cdot}0442$ for 8711 observed reflections. There are six crystallographically independent Sb atoms in the unit cell, three of them located on threefold axes and the remaining three in general positions. Each Sb atom is six-coordinated; the Sb atom on the threefold axis has three F and three O atoms at the corners of the coordination octahedron whereas the Sb atoms in the general positions coordinate four F and two O atoms. The structure consists of three crystallographically independent polymeric chains which have been linked in a three-dimensional network by the N—H···O and N—H···F interactions. The chirality of one of the chains is opposite to that of the other two.

Introduction. It is known (Grigas, Davydovitch & Urbonavichus, 1978; Agarwal & Chand, 1985) that sulfate fluoride compounds of tervalent antimony with ammonium and alkali metals, crystallizing in the composition $M_6[Sb_4F_{12}(SO_4)_3]$ (M = Rb, Cs, NH_4), exhibit interesting physical properties. Davydovitch, Zemnukhova & Sigula (1973) reported that these compounds belong to the hexagonal system with $Z = 3$. The IR spectra – almost identical for the three compounds – suggested that the structures are isomorphous (Davydovitch, Sergyenko, Zemnukhova, Kharitonov & Kostin, 1974). The NQR experiments performed by Davydovitch, Zemnukhova & Kravchenko (1974) indicated that there are four types of Sb atoms in the unit cell. Dielectric studies (Grigas *et al.*, 1978) revealed anomalies on the ε' and ε'' curves in the temperature ranges 240–260 and 235–240 K for the NH_4 and Rb compounds, respectively. These authors stated that the room-temperature phase is paraelectric. It was suggested that below the transition temperature the crystal transforms to another polar phase. The EPR and optical absorption studies of the vanadyl-ion(VO^{2+})-doped single crystal of $[NH_4]_6[Sb_4F_{12}(SO_4)_3]$ indicated a phase transition at 256 K (Agarwal & Chand, 1985). A lack of detailed structural data has hindered further explanation of the physical properties of the crystal. This X-ray structure determination at room temperature was undertaken as part of our studies of the phase transition in this material.

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Experimental. Colorless prismatic crystals of $[NH_4]_6$-$[Sb_4F_{12}(SO_4)_3]$ were obtained by Czapla (1986) by slow evaporation from an aqueous solution of $[NH_4]_2SO_4$ and SbF_3 in molar ratio 3:4; D_m determined by flotation in $CHBr_3/CBr_4$ mixture. Trigonal system and Laue class C_{3i} unequivocally determined from Weissenberg photographs, lack of systematic absences indicated space groups $P3$ or $P\bar{3}$. A small specimen was cut from a large crystal and shaped into an approximate sphere ($2R = 0{\cdot}35$ mm) by polishing with blotting paper moistened in water. Syntex $P2_1$ diffractometer, graphite monochromator, lattice parameters refined by least-squares fit of 15 reflections in range $21 \leq 2\theta \leq 26°$. A total of 13 573 reflections measured below $2\theta = 65°$, hkl range: h 0 to 24, k −25 to 25 and l −11 to 11, $\theta/2\theta$ scan technique, two standard reflections measured after every 50 reflections, variation ±13% connected with apparatus instability during 20 d of data collection; no indications of crystal decay. Absorption correction applied as for spherical crystal with $\mu R = 0{\cdot}8$. Equivalent reflections averaged giving a set of 9021 reflections with $F(hkl)$ and $F(\bar{h}\bar{k}\bar{l})$ (Bijvoet pairs not averaged) of which 8711 with $I_o > 2\sigma(I_o)$ used in final refinement; $R_{\rm int} = 0{\cdot}035$.

The choice of the noncentrosymmetric space group $P3$ was originally based on a positive piezoelectric effect measured at room temperature by Grigas *et al.* (1978) and confirmed by our observation of optical activity. Structure solved by direct-methods program *MULTAN*80 (Main *et al.*, 1980). The Sb and S atoms were located in the E map with the highest combined figures of merit. The remaining heavy atoms were found from a difference Fourier synthesis. Refinement with *SHELX*76 (Sheldrick, 1976) based on F values (Sb, S, F and O anisotropic, N isotropic). Geometry calculations performed with Syntex (1976) *XTL/XTLE* system. Empirical correction for extinction applied according to the formula $F_{\rm corr} = F(1-XF^2\sin\theta)$ where X refined to $2{\cdot}2\,(6) \times 10^{-7}$. $\sum w||F_o| - |F_c||^2$ minimized, $w = 1/[\sigma^2(F_o) + g(F_o)^2]$, refined value of $g = 8{\cdot}762\,(2) \times 10^{-3}$. Final $R = 0{\cdot}043$, $wR = 0{\cdot}047$, $R_G = 0{\cdot}06$, for 305 refined parameters $(\Delta/\sigma)_{\max} = 0{\cdot}011$, minimum and maximum height on difference Fourier map $-1{\cdot}43$ and $1{\cdot}97$ e Å^{-3} in vicinity of Sb atoms; H-atom positions were not resolvable. Neutral-atom scattering factors including corrections for anomalous dispersion for all atoms taken from *International Tables for X-ray Crystallography* (1974). Conoscopic figures and Airy's spirals from the sample used in data collection were not very sharp but identified the crystal as dextrorotatory. However, it was impossible to measure optical activity in this sample since it was too small to obtain sufficiently resolved optical rotation. Thus, the slices from six different samples of thickness from 0·15 to 0·43 mm were observed in conoscopic illumination with a quarter-wave plate. Each of the slices was dextrorotatory. The angle of optical rotation in the He/Ne laser light ($\lambda = 6328$ Å) was $\alpha = 7°$ for the plate of thickness 0·43 mm. The absolute structure was determined using all Bijvoet pairs in refinement of models with xyz and $\bar{x}\bar{y}\bar{z}$ configuration. The model with $\bar{x}\bar{y}\bar{z}$ coordinates gave $R = 0{\cdot}048$ and $wR = 0{\cdot}052$ hence the enantiomeric structure can safely be rejected. Table 1 presents the atomic configuration for the dextrorotatory crystal.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^2)

$U_{\rm eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma)$. For N atoms $U_{\rm iso}$ values are given.

	x	y	z	$U_{\rm eq}$
Sb(*A*)	0·0	0·0	0·40709 (9)	1·88 (2)
Sb(*B*)	$\frac{1}{3}$	$\frac{2}{3}$	0·77048 (9)	1·78 (2)
Sb(*C*)	$\frac{2}{3}$	$\frac{1}{3}$	0·31164 (9)	1·68 (1)
Sb(2)	−0·16260 (3)	−0·02780 (2)	0·9100*	1·95 (2)
Sb(3)	0·46503 (2)	0·82231 (2)	0·26414 (6)	1·87 (1)
Sb(4)	0·83907 (2)	0·38993 (3)	0·81608 (7)	2·05 (1)
S(1)	−0·16885 (9)	−0·19834 (9)	0·1755 (2)	1·97 (5)
S(2)	0·30499 (9)	0·83539 (9)	0·5348 (2)	1·86 (5)
S(3)	0·81958 (9)	0·53946 (9)	0·5436 (2)	2·04 (5)
F(1)	−0·1042 (4)	−0·0529 (4)	0·5646 (6)	3·5 (2)
F(2)	0·4349 (4)	0·7385 (4)	−0·0710 (8)	4·1 (2)
F(3)	0·6787 (3)	0·4293 (3)	0·1526 (7)	3·6 (2)
F(21)	−0·1911 (4)	0·0368 (3)	−0·2683 (6)	3·3 (2)
F(31)	0·4596 (3)	0·9010 (3)	0·0809 (5)	3·1 (2)
F(41)	0·9051 (3)	0·4777 (3)	1·0022 (6)	2·9 (2)
F(22)	−0·2466 (3)	−0·0114 (3)	0·0540 (7)	3·4 (2)
F(32)	0·5356 (3)	0·9319 (3)	0·3979 (6)	2·8 (2)
F(42)	0·9480 (3)	0·4443 (3)	0·6794 (6)	3·0 (2)
F(23)	−0·2667 (4)	−0·1337 (3)	0·8103 (7)	4·1 (2)
F(33)	0·5785 (3)	0·8490 (4)	0·1544 (6)	3·1 (2)
F(43)	0·8894 (4)	0·3198 (4)	0·9267 (7)	3·5 (2)
O(11)	−0·2480 (4)	−0·2846 (4)	0·2220 (8)	3·4 (2)
O(12)	−0·0953 (4)	−0·1744 (4)	0·3059 (8)	3·2 (2)
O(13)	−0·1386 (3)	−0·2067 (4)	−0·0048 (6)	2·7 (2)
O(14)	−0·1928 (5)	−0·1256 (4)	0·1709 (7)	3·3 (2)
O(21)	0·2902 (6)	0·9059 (5)	0·5950 (11)	4·6 (3)
O(22)	0·3672 (4)	0·8273 (4)	0·6550 (9)	3·9 (2)
O(23)	0·3438 (4)	0·8547 (4)	0·3518 (7)	2·9 (2)
O(24)	0·2170 (4)	0·7475 (4)	0·5299 (8)	3·3 (2)
O(31)	0·8831 (5)	0·6271 (4)	0·4667 (8)	4·2 (2)
O(32)	0·8150 (5)	0·4656 (4)	0·4351 (10)	4·2 (3)
O(33)	0·8499 (5)	0·5350 (4)	0·7250 (7)	3·3 (2)
O(34)	0·7299 (4)	0·5318 (5)	0·5544 (9)	3·8 (3)
N(1)	0·7229 (4)	−0·0638 (4)	0·4239 (8)	3·5 (1)
N(2)	0·3624 (5)	0·4272 (5)	0·7782 (9)	4·0 (1)
N(3)	0·7889 (4)	0·7317 (4)	0·6295 (8)	3·1 (1)
N(4)	−0·0569 (4)	0·4074 (4)	0·3137 (8)	3·3 (1)
N(5)	0·8465 (4)	0·6061 (4)	0·0895 (8)	3·5 (1)
N(6)	0·6016 (4)	0·7045 (5)	0·9868 (8)	3·5 (1)

* Held invariant to fix the origin of the coordinate system.

Discussion. Final atomic parameters are given in Table 1.* There are three kinds of similar, but not identical, polymeric chains (denoted *A*, *B* and *C*) in the crystal. In each chain the central Sb atom [Sb(*A*), Sb(*B*) or Sb(*C*)] occupies a special position on the threefold axis while the remaining three Sb atoms are situated in general positions (Fig. 1). Each Sb atom is six-coordinated and the coordination octahedron comprises two groups of bonds. There are three equivalent short bonds between

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* Lists of structure factors, anisotropic thermal parameters, and bond lengths and angles for the sulfate ions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44898 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the central Sb and the F atoms with a mean length of 1·95 (1) Å for the three chains. Each of these F atoms is simultaneously linked to one of the Sb atoms in a general position, forming weak F—Sb bonds of length 2·890 (4), 2·814 (5) and 2·878 (4) Å for the chains *A*, *B* and *C*, respectively. The remaining three coordination sites of the central Sb atoms are occupied by sulfate O atoms. Two other O atoms from each of the sulfate groups bridge two Sb atoms located in general positions. Each sulfate group is thus tridentate. The three remaining coordination sites of Sb atoms in general positions are occupied by F with short Sb—F bonds. In this manner linear polymeric chains are formed about the threefold axis. In each chain the coordination sphere of the central Sb atom is built up by three F and three O atoms, while four F and two O atoms surround the Sb atoms in general positions. The F(1), F(2) and F(3) atoms form Sb···Sb bridges with mean distance 4·54 (5) Å (Table 2). The two polymeric chains *A* and *B* are shown in Fig. 2 in a perspective view, close to but not exactly along [001]. When comparing Figs. 1 and 2 it can be noticed that chains *B* and *C* have the same chirality whereas chain *A*, located about the threefold axis at 00*z*, shows opposite chirality. Such a conformation with chains of opposing chirality within a given crystal has not been previously observed and was not considered by Glazer & Stadnicka (1986) in their review on the origin of optical activity in crystal structures.

The sulfate ions form distorted tetrahedra. The S—O bond lengths vary from 1·421 (8) to 1·501 (1) Å; the mean S—O distance is 1·471 (21) Å. The O—S—O angles range from 108·1 (4) to 110·7 (4)°; the mean bond angle is 109·5 (9)°.

Although the hydrogen positions of the ammonium ions have not been determined, the intermolecular contacts shown in Table 3 indicate interactions of the type N—H···O and N—H···F.

We are grateful to Doc. Z. Czapla (Institute of Physics, University of Wrocław) for providing us with a

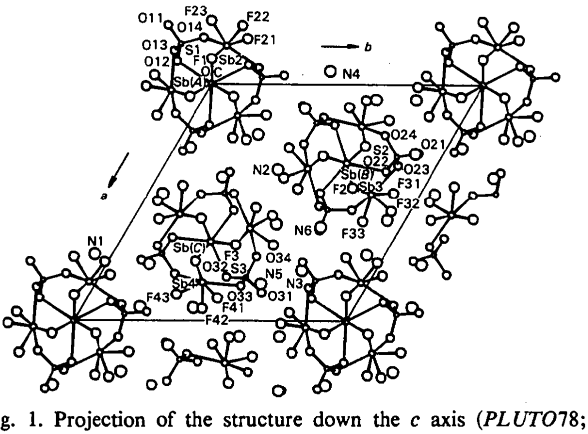
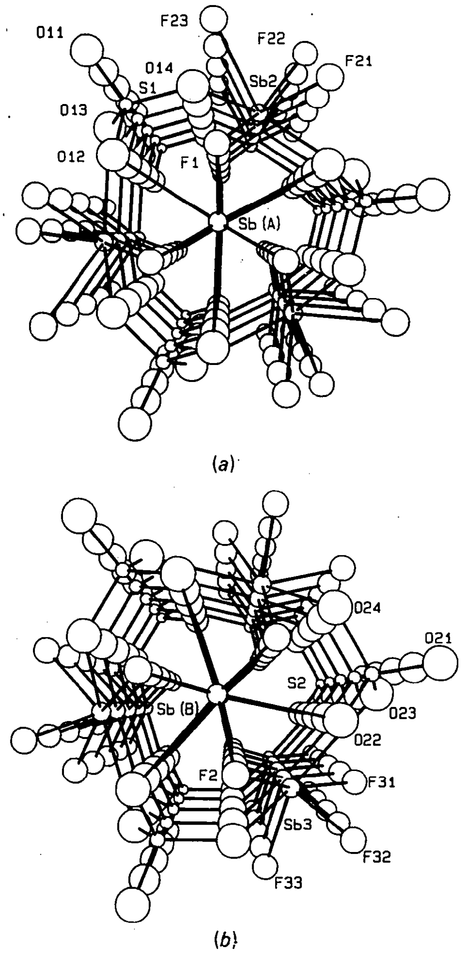


Fig. 1. Projection of the structure down the *c* axis (*PLUTO78*; Motherwell & Clegg, 1978).

Table 2. *Bond distances and selected interatomic distances* (Å) *for* Sb *atoms with e.s.d.'s in parentheses*

Sb(*A*)—F(1)	1·942 (5)	Sb(2)—F(1)	2·890 (4)
Sb(*A*)—O(12)	2·691 (6)	Sb(2)—F(21^{ii})	1·945 (4)
Sb(*A*)···S(1)	3·612 (2)	Sb(2)—F(22^{ii})	1·925 (5)
Sb(*A*)···Sb(2^{i})	4·535 (3)	Sb(2)—F(23)	1·942 (5)
Sb(*A*)···Sb(2)	4·571 (3)	Sb(2)—O(13^{iv})	2·548 (4)
		Sb(2)—O(14^{i})	2·457 (5)
		Sb(2)···Sb(2^{iii})	4·454 (3)
Sb(*B*)—F(2^{ii})	1·950 (5)	Sb(3)—F(2)	2·814 (5)
Sb(*B*)—O(22)	2·650 (7)	Sb(3)—F(31)	1·959 (4)
Sb(*B*)···S(2)	3·614 (2)	Sb(3)—F(32)	1·926 (4)
Sb(*B*)···Sb(3^{ii})	4·461 (3)	Sb(3)—F(33)	1·938 (4)
Sb(*B*)···Sb(3)	4·541 (3)	Sb(3)—O(23)	2·483 (6)
		Sb(3)—O(24^{v})	2·538 (5)
		Sb(3)···Sb(3^{v})	4·292 (3)
Sb(*C*)—F(3)	1·954 (4)	Sb(4)—F(3^{vi})	2·878 (4)
Sb(*C*)—O(32)	2·580 (6)	Sb(4)—F(41)	1·945 (4)
Sb(*C*)···S(3)	3·612 (2)	Sb(4)—F(42)	1·910 (4)
Sb(*C*)···Sb(4^{i})	4·541 (3)	Sb(4)—F(43)	1·973 (4)
Sb(*C*)···Sb(4)	4·596 (3)	Sb(4)—O(33)	2·486 (6)
		Sb(4)—O(34^{vii})	2·675 (7)
		Sb(4)···Sb(4^{viii})	4·500 (3)

Symmetry code: (i) $x, y, z-1$; (ii) $x, y, z+1$; (iii) $-x, x-y, z$; (iv) $y-x, -x, z$; (v) $y-x, 1-x, z$; (vi) $-y, x-y, z$; (vii) $1-x+y, 1-x, z$; (viii) $1-x, x-y, z$.



(*a*)

(*b*)

Fig. 2. Perspective views of the two polymeric chains with opposing chirality. (*a*) Chain *A* is located about the threefold axis at 00*z* and (*b*) chain *B* about the threefold axis at $\frac{1}{3}\frac{2}{3}z$.

Table 3. *The* N···F *and* N···O *contacts* (Å) *with e.s.d.'s in parentheses*

N(1)···F(21$^{\text{i}}$)	2·816 (8)	N(2)···F(31$^{\text{iv}}$)	2·850 (8)
F(22$^{\text{ii}}$)	2·884 (8)	F(32$^{\text{v}}$)	2·922 (9)
O(14$^{\text{ii}}$)	2·883 (8)	F(42$^{\text{x}}$)	2·973 (8)
O(31$^{\text{vii}}$)	2·939 (9)	O(24$^{\text{xi}}$)	2·979 (9)
N(3)···F(21$^{\text{viii}}$)	2·803 (8)	N(4)···F(41$^{\text{xii}}$)	2·850 (8)
O(13$^{\text{vi}}$)	2·978 (8)	F(42$^{\text{xiii}}$)	2·812 (8)
O(21$^{\text{v}}$)	2·801 (8)	O(32$^{\text{xii}}$)	2·962 (8)
O(12$^{\text{ix}}$)	3·037 (9)	O(11$^{\text{xiv}}$)	2·986 (9)
N(5)···F(3)	2·986 (9)	N(6)···F(23$^{\text{xv}}$)	2·872 (8)
F(41$^{\text{xvii}}$)	2·902 (9)	F(31$^{\text{iv}}$)	2·855 (8)
F(43$^{\text{xvi}}$)	2·859 (9)	F(33$^{\text{iii}}$)	2·963 (9)
O(31)	2·889 (9)	O(21$^{\text{v}}$)	2·955 (9)

Symmetry code: none x, y, z; (i) $x+1, y, z+1$; (ii) $x+1, y, z$; (iii) $x, y, z+1$; (iv) $y-x, 1-x, z+1$; (v) $y-x, 1-x, z$; (vi) $1+x, 1+y, z+1$; (vii) $1-x+y, 1-x, z$; (viii) $1-y, 1+y-x, z+1$; (ix) $x-1, y-1, z$; (x) $1-y, y-x, z$; (xi) $1-y, 1+y-x, z$; (xii) $x-1, y, z-1$; (xiii) $x-1, y, z$; (xiv) $y-x, -x, z$; (xv) $x+1, y+1, z$; (xvi) $1-y, y-x, z-1$; (xvii) $x, y, z-1$.

sample and for stimulating suggestions for this study. AW wishes to thank Dr K. Stadnicka (Jagiellonian University, Kraków) for many helpful discussions on the optical studies of the crystal. A special debt of gratitude is owed to Dr M. Wołcyrz and J. Jańczak from our department for much help in preparing the figures, and to MERA ELZAB, Zabrze, for supporting our laboratory with the 79100 Monitor.

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Structure of Nickel(II) Perbromate Hexahydrate at 169 K

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Abstract. $Ni(BrO_4)_2.6H_2O$, $M_r = 454·61$, trigonal, $P\bar{3}$, $a = 7·817\ (2)$, $c = 5·235\ (1)$ Å, $V = 277·03\ (14)$ Å^3, $Z = 1$, $D_x = 2·725$ g cm^{-3}, $\lambda(\text{Mo}\ K\bar{\alpha}) = 0·71069$ Å, $\mu = 89·7$ cm^{-1}, $F(000) = 222$, $T = 169$ K, $R = 0·029$ for 417 unique reflections with $I > 0$. The structure consists of stacks of hydrated nickel ions and stacks of perbromate groups with hydrogen bonds from a given water molecule involving three stacks of perbromate groups. The water O atoms form a very slightly distorted octahedron about nickel while the perbromate geometry is virtually ideally tetrahedral. This structure and that of mercury(II) perchlorate hexahydrate, though assigned in different space groups, are very similar and are interestingly related as described in the text.

Introduction. Interest has arisen in this laboratory concerning detailed similarities and differences in the structures of corresponding simple salts containing perbromate or perchlorate anions. Since at the inception of these investigations very little structural information was available for perbromates, it appeared worthwhile to determine the structures of a number of simple perbromate salts. We have previously reported on the structure of barium perbromate trihydrate (Gerkin, Reppart & Appelman, 1988).

Experimental. Nickel perbromate was prepared by the aqueous reaction of suspended nickel(II) hydroxide with a slight excess of ~2*M* perbromic acid.‡ [The

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‡ The synthesis of the perbromic acid was performed at Argonne National Laboratory while WJR was a Thesis Parts Program participant. Program administered by the Argonne Division of Educational Programs with funding from the US Department of Energy.

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