

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Rb	0	0	0	0.0165 (4)
Ti	0	0	0.15141 (4)	0.0023 (4)
P	0.71964 (17)	0	1/4	0.0022 (5)
O(1)	0.2126 (4)	0.1469 (4)	0.30069 (10)	0.0057 (10)
O(2)	0.4659 (4)	0.3029 (4)	0.23157 (10)	0.0080 (10)

Table 2. Selected geometric parameters (\AA , $^\circ$)

TiO ₆ octahedron			
Ti—O(1) × 3	1.927 (3)	O(1)—Ti—O(1)	89.2 (1)
Ti—O(2) × 3	1.944 (3)	O(1)—Ti—O(2)	97.2 (1)
		O(1)—Ti—O(2)	86.5 (1)
		O(1)—Ti—O(2)	172.3 (1)
		O(2)—Ti—O(2)	87.6 (1)
PO ₄ tetrahedron			
P—O(2) × 2	1.517 (3)	O(2)—P—O(2)	110.7 (2)
P—O(1) × 2	1.530 (3)	O(2)—P—O(1)	109.0 (2)
		O(2)—P—O(1)	107.1 (2)
		O(1)—P—O(1)	113.9 (2)
Environment around Rb			
Rb—O(2) × 6	2.854 (2)	O(2)—Rb—O(2)	56.3 (1)
Rb—O(1) × 6	3.366 (2)	O(1)—Rb—O(1)	65.0 (1)
		O(2)—Rb—O(1)	45.5 (1)
		O(2)—Rb—O(1)	79.0 (1)
		O(2)—Rb—O(1)	86.4 (1)

Table 3. Comparison of cell parameters (\AA), bond lengths (\AA) and bond strengths (v.u.) for isostructural MTi₂(PO₄)₃ compounds

	NaTi ₂ (PO ₄) ₃ *		KTi ₂ (PO ₄) ₃ †		RbTi ₂ (PO ₄) ₃	
	Length	Strength	Length	Strength	Length	Strength
a	8.502	—	8.367	—	8.290	—
c	21.833	—	23.074	—	23.530	—
M—O(2)	2.290	0.27	2.745	0.19	2.857	0.20
M—O(1)	3.788	0	3.418	0.03	3.366	0.05
Ti—O(1)	1.896	0.80	1.918	0.76	1.927	0.74
Ti—O(2)	2.107	0.45	1.942	0.71	1.944	0.71
P—O(2)	1.530	1.27	1.524	1.29	1.517	1.31
P—O(1)	1.533	1.25	1.530	1.27	1.530	1.27

* Ivanov, Belokoneva, Egorov-Tismenko, Simonov & Belov (1980).

† Lunezheva, Maksimov, Mel'nikov & Muradyan (1989).

Data collection, cell refinement and data reduction were carried out using Stoe software. Precise cell refinement was performed by double-step-scan technique. The atomic coordinates for KTi₂(PO₄)₃ (Lunezheva, Maksimov, Mel'nikov & Muradyan, 1989) were used as an initial model. The structure was refined using *SHELX76* (Sheldrick, 1976).

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Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: DU1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ammonium Heptachlorooxo-diantimonate(III), (NH₄)₃[Sb₂Cl₇O]

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Abstract

Each Sb atom of the title compound is formally ψ -tetrahedral (AB_3E) with covalent bonds to two terminal Cl atoms and one bridging O atom. Three directed secondary interactions to three additional symmetrically bridging Cl atoms give each Sb atom a roughly octahedral coordination geometry. The bridging arising from the secondary interactions produces chains of [Sb₂Cl₄O]₂ units which propagate along the unit-cell *c* axis. The O, Cl(3) and Cl(4) atoms lie on crystallographic mirror planes, while Cl(5) resides on a twofold axis. The ammonium cations are hydrogen bonded to the Cl and O atoms.

Comment

The title compound was unintentionally isolated during the reaction of SbCl₃ with tetraethylene glycol in a 3:1 acetonitrile–methanol solution containing stoichiometric amounts of NH₄OH. The covalent Sb—Cl(1,2) and Sb—O distances are normal (Begley, Hall, Nunn & Sowerby, 1986; Hall & Sowerby, 1979; Rheingold, Landers, Dahlstrom & Zubieta, 1979). There are two types of bridging interaction produced by the secondary Sb—Cl contacts (Sawyer & Gillespie, 1986). The Cl(3) and the O atoms bridge two Sb atoms with an Sb...Sb separation of 3.582 (1) \AA . The Cl(5) and Cl(4) atoms form

nearly linear bridges and the Sb...Sb separations are correspondingly longer [5.757 (2) Å at the Cl(5) bridge and 6.411 (1) Å at the Cl(4) bridge]. Each bridging interaction in the polymeric chain is symmetric.

Although the H atoms could not be located, the N...O and N...Cl contact geometries indicate extensive hydrogen bonding, most likely with both single and bifurcated interactions. The possible hydrogen-bonded N...O contacts range from 2.94 (1) to 3.19 (2) Å and the N...Cl distances range from 3.17 (2) to 3.572 (9) Å.

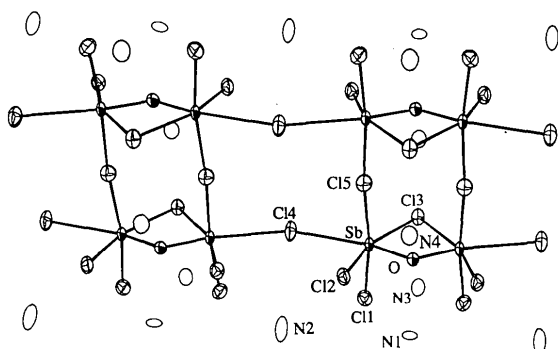


Fig. 1. ORTEP (Johnson, 1976) illustration of a portion of the polymeric chains in $(\text{NH}_4)_3[\text{Sb}_2\text{Cl}_7\text{O}]$. Ellipsoids are drawn at the 50% probability level.

Experimental

Crystal data

$(\text{NH}_4)_3[\text{Sb}_2\text{Cl}_7\text{O}]$
 $M_r = 561.79$
 Orthorhombic
Pnnm
 $a = 11.342$ (3) Å
 $b = 13.165$ (3) Å
 $c = 9.993$ (2) Å
 $V = 1492$ Å³
 $Z = 4$
 $D_x = 2.50$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 20\text{--}25^\circ$
 $\mu = 4.88$ mm⁻¹
 $T = 291$ K
 Parallelepiped
 $0.25 \times 0.15 \times 0.05$ mm
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: empirical
 $T_{\min} = 0.83$, $T_{\max} = 1.00$
 1538 measured reflections
 1538 independent reflections

816 observed reflections
 $[F_o \geq 5\sigma(F_o)]$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 13$
 $k = 0 \rightarrow 15$
 $l = 0 \rightarrow 11$
 3 standard reflections
 frequency: 60 min
 intensity variation: $\pm 2.5\%$

Refinement

Refinement on F
 $R = 0.041$
 $wR = 0.050$
 $S = 1.18$
 816 reflections
 71 parameters
 H-atom parameters not included

$w = 1/[\sigma^2(F_o) + 0.0004F_o^2]$
 $(\Delta/\sigma)_{\max} = 0.01$
 $\Delta\rho_{\max} = 0.8$ e Å⁻³
 $\Delta\rho_{\min} = -1.2$ e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Sb	0.78567 (6)	0.61713 (5)	0.67921 (7)	1.31
Cl(1)	0.5895 (3)	0.7007 (3)	0.7008 (3)	2.35
Cl(2)	0.6931 (3)	0.4563 (2)	0.7550 (3)	2.05
Cl(3)	0.8764 (4)	0.7694 (3)	1/2	2.00
Cl(4)	0.8190 (5)	0.6393 (4)	1	2.48
Cl(5)	1	1/2	0.6929 (5)	2.44
O	0.749 (1)	0.5598 (9)	1/2	1.73
N(1)	1/2	1/2	1/2	2.87
N(2)	1/2	1/2	1	3.69
N(3)	0.611 (1)	0.899 (1)	1/2	2.55
N(4)	0.847 (2)	0.332 (1)	1/2	2.44

Table 2. Selected geometric parameters (Å, °)

Sb—Cl(1)	2.492 (3)	Sb—Cl(2)	2.482 (3)
Sb—Cl(3)	2.879 (3)	Sb—Cl(4)	3.241 (1)
Sb—Cl(5)	2.8820 (8)	Sb—O	1.987 (5)
Cl(1)—Sb—Cl(2)	88.4 (1)	Cl(1)—Sb—Cl(3)	93.8 (1)
Cl(2)—Sb—Cl(3)	159.3 (1)	Cl(1)—Sb—Cl(4)	88.8 (1)
Cl(2)—Sb—Cl(4)	79.9 (1)	Cl(3)—Sb—Cl(4)	120.7 (1)
Cl(1)—Sb—Cl(5)	170.2 (1)	Cl(2)—Sb—Cl(5)	83.45 (8)
Cl(3)—Sb—Cl(5)	95.8 (1)	Cl(4)—Sb—Cl(5)	84.4 (1)
Cl(1)—Sb—O	93.5 (3)	Cl(2)—Sb—O	82.1 (3)
Cl(3)—Sb—O	77.2 (3)	Cl(4)—Sb—O	161.8 (3)
Cl(5)—Sb—O	90.8 (3)	Sb—Cl(3)—Sb ⁱ	76.9 (1)
Sb—Cl(4)—Sb ⁱ	163.1 (2)	Sb—Cl(5)—Sb ⁱⁱⁱ	174.6 (2)
Sb—O—Sb ⁱⁱ	128.7 (6)		

Symmetry codes: (i) $x, y, 2 - z$; (ii) $x, y, 1 - z$; (iii) $2 - x, 1 - y, z$.

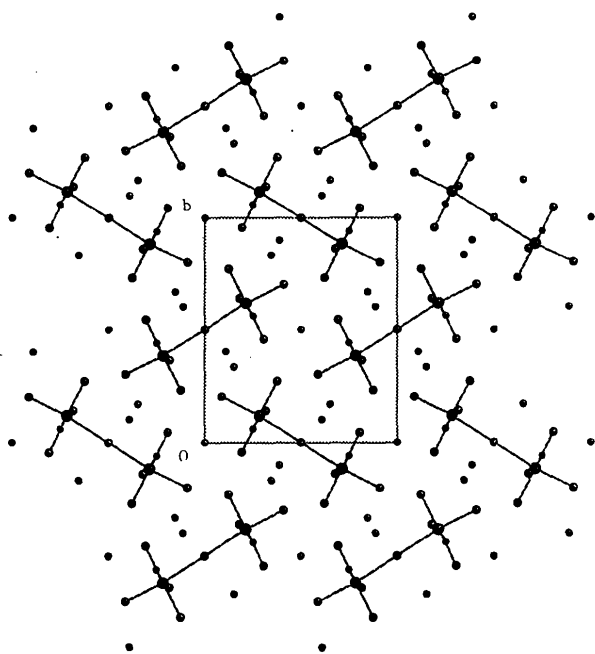


Fig. 2. SYBYL (Tripos Associates, 1993) representation of the unit-cell packing as viewed down the c axis. The polymeric chains are viewed end on.

Computer programs utilized include *SHELXS86* (Sheldrick, 1990) for structure solution, *SHELX76* (Sheldrick, 1976) for structure refinement, *ORTEP* (Johnson, 1976) for the preparation of Fig. 1 and *SYBYL* (Tripos Associates, 1993) for the preparation of Fig. 2.

Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: BR1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexagonal $\text{Yb}_6\text{Cr}_{4+x}\text{Al}_{43-x}$ ($x = 1.76$) with a New Structure Type

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Abstract

The title compound contains one Yb, two Cr and seven Al sites, one of which is about half occupied by Cr. The coordination polyhedron of Yb has 17 vertices and composition $[\text{Cr}(\text{Al}, \text{Cr})_2\text{Al}_{13}\text{Yb}]$, while those around Cr are $[\text{Al}_{10}\text{Yb}_2]$ and $[\text{Al}_{12}]$, and that around the mixed site is $[\text{Al}_9\text{Yb}_3]$.

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Comment

Aluminium-rich rare-earth (*R*) transition-metal (*T*) compounds of approximate composition $RT\text{Al}_8$ are known for $R = \text{Y}, \text{Dy}, \text{Sm}, \text{Tb}$ and $T = \text{V}, \text{Cr}$ (Zarechnyuk, Rykhal' & German, 1971; Rykhal', Zarechnyuk & Mats'kiv, 1979; Zarechnyuk, Yanson, Ostrovskaya & Shevchuk, 1988). Investigation of the system $\text{Yb}-\text{Cr}-\text{Al}$ at 773 K revealed the existence of a similar compound.

The structure of $\text{Yb}_6\text{Cr}_{4+x}\text{Al}_{43-x}$ is of a new type. It contains one Yb, two Cr and seven Al sites, one of which is occupied partially by Cr (Fig. 1). The coordination polyhedron of Yb has 17 vertices and composition $[\text{Cr}(\text{Al}, \text{Cr})_2\text{Al}_{13}\text{Yb}]$. The polyhedra of the two ordered chromium sites Cr(1) and Cr(2), and the disordered metal site Al(6) ($\equiv \text{Al}_{0.56}\text{Cr}_{0.44}$) each have 12 vertices; their compositions are $[\text{Al}_{10}\text{Yb}_2]$, $[\text{Al}_{12}]$ and $[\text{Al}_9\text{Yb}_3]$, respectively. The polyhedra around Cr(1) and Al(6) each have a deformed icosahedral shape, while that around Cr(2) has a regular icosahedral shape, as in many other Al-rich transition-metal compounds (Kripyakevich, 1977). The polyhedra around the other six Al sites all each have 12 vertices and are deformed icosahedra or bicapped pentagonal prisms $[\text{Al}(7)]$. The polyhedra around the Cr(1) sites are linked parallel to the hexagonal plane in groups of three *via* common Al atoms and these units are linked perpendicular to the hexagonal plane to form columns along $[001]$ at $x = 0, y = 0$ *via* Cr(2) icosahedra (Fig. 2). Adjacent columns are connected *via* interpenetrated Al(6) and Yb polyhedra. All other Al polyhedra interpenetrate with Cr(1), Cr(2), Al(6) or Yb polyhedra. The architecture of the columns of Cr polyhedra (without the Yb atoms) resembles that of hexagonal V_4Al_{23} (Smith & Ray, 1957). This structure also contains two transition-metal sites. One of these is icosahedrally coordinated with Al atoms [as in

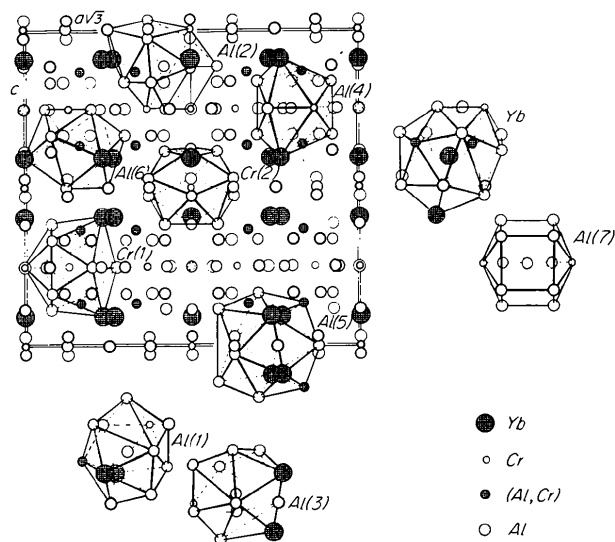


Fig. 1. Structural projection of hexagonal $\text{Yb}_6\text{Cr}_{4+x}\text{Al}_{43-x}$ along $[010]$ showing the coordination polyhedra.