

**K₂SrNb₆Cl₁₈, dipotassium strontium
hexaniobium octadecachloride**

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The structure of dipotassium strontium hexaniobium octadecachloride is based on $[\text{Nb}_6\text{Cl}_{12}^i\text{Cl}_6^a]^{4-}$ units (*i* and *a* denote 'inner' and 'outer' ligands, respectively), which have crystallographically imposed $\bar{3}$ symmetry, linked together by K^+ and Sr^{2+} cations. The K^+ cation occupies a tetrahedral site in the face-centered cubic lattice of cluster units, and is bonded to 12 Cl ligands. The Sr atom is located in an octahedral site and is bonded to six outer Cl ligands.

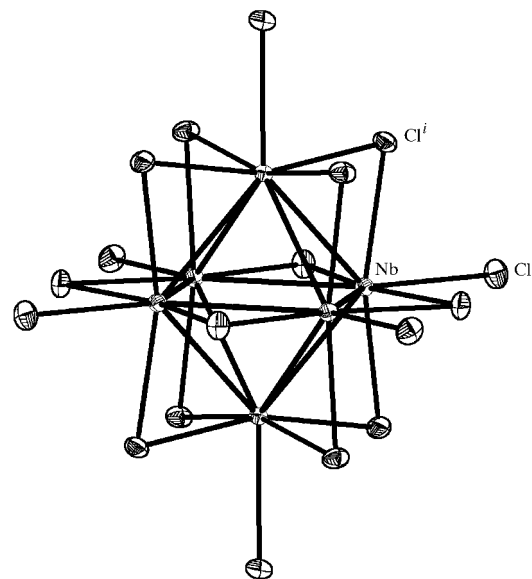
Comment

A large number of metal-rich niobium halides with $[\text{Nb}_6\text{Cl}_{18}]^{n-}$ -type cluster units have been previously crystallized using a variety of metal cations, for instance, $A\text{RENb}_6\text{Cl}_{18}$ (where *A* is an alkali and *RE* is a rare earth element; Ihmaine *et al.*, 1989) and $A\text{TiNb}_6\text{Cl}_{18}$ (*A* is an alkali or group 13 element; Nagele *et al.*, 2000). Recently, Pb was used to prepare the cluster compound $\text{Cs}_2\text{PbNb}_6\text{Cl}_{18}$ (Gulo *et al.*, 2001).

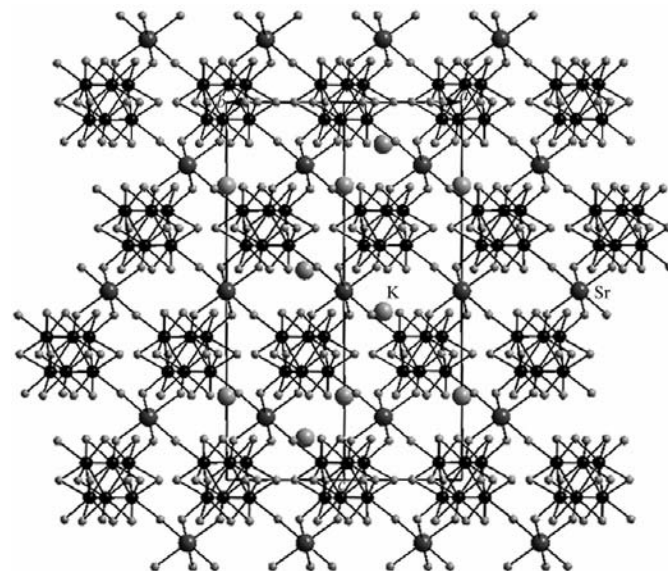
In the present paper, we describe the structure of the new cluster compound dipotassium strontium hexaniobium octadecachloride, $\text{K}_2\text{SrNb}_6\text{Cl}_{18}$, crystallized using Sr^{2+} . The new compound crystallizes in the trigonal space group $R\bar{3}$. The structure is based on discrete anionic cluster units, $[(\text{Nb}_6\text{Cl}_{12}^i\text{Cl}_6^a)]^{4-}$ (where *i* and *a* denote 'inner' and 'outer' ligands, respectively). The cluster unit consists of an Nb_6 octahedron in which all edges are bridged by chlorines, and six other ligands are in apical positions (Fig. 1 and Table 1). The Nb—Cl bond lengths [Nb—Cl^{*i*} = 2.4497 (11)–2.4636 (11) Å and Nb—Cl^{*a*} = 2.5982 (11) Å] are typical for $\text{Nb}_6\text{Cl}_{18}$ clusters. The Nb—Nb bond lengths [2.9237 (6)–2.9309 (7) Å] indicate that the VEC (valence electrons per cluster) of the cluster core is 16. The three-dimensional structure of the title compound is based on the cluster units interlinked through K^+ and Sr^{2+} cations to form cluster layers. The cluster layers are arranged according to face-centered cubic stacking along the *c* axis (Fig. 2). The K^+ ions occupy tetrahedral sites between the units and are bonded to 12 Cl ligands, with K—Cl distances in

the range 3.2648 (12)–3.6182 (11) Å. The Sr^{2+} ions are located in octahedral sites between the units and are bonded to six Cl ligands in a perfect octahedral geometry, with Sr—Cl distances of 2.8954 (11) Å.

The quaternary chloride compound $\text{K}_2\text{SrNb}_6\text{Cl}_{18}$ is isostructural with $\text{Cs}_2\text{PbNb}_6\text{Cl}_{18}$ (Gulo *et al.*, 2001), $\text{Cs}_2\text{PbTa}_6\text{Cl}_{18}$ (Cordier *et al.*, 1999) and $\text{KGdNb}_6\text{Cl}_{18}$ (Ihmaine *et al.*, 1987). The alkali site, only half occupied in $\text{KGdNb}_6\text{Cl}_{18}$, is, in contrast, fully occupied in the title compound. In order to obtain 16 VEC per cluster, the substitution of trivalent gadolinium by divalent strontium is compensated by the presence of two K atoms, as is the case in $\text{Cs}_2\text{PbNb}_6\text{Cl}_{18}$.

**Figure 1**

The chloride cluster anion $[\text{Nb}_6\text{Cl}_{12}^i\text{Cl}_6^a]^{4-}$ present in $\text{K}_2\text{SrNb}_6\text{Cl}_{18}$ (*i* and *a* denote 'inner' and 'outer' ligands, respectively).

**Figure 2**

Projection of the crystal structure of $\text{K}_2\text{SrNb}_6\text{Cl}_{18}$ in the [110] direction.

Experimental

The title compound, $K_2SrNb_6Cl_{18}$, was initially obtained as shiny black gem-like crystals from a reaction proposed to yield an oxychloride compound of composition $KSr_3Nb_6Cl_{10}O_6$. Subsequently, the compound was prepared quantitatively from a stoichiometric mixture containing $NbCl_5$ (Alfa, 99.8%), Nb powder (Alfa 99.8%), $SrCl_2$ (Alfa 99.8%) and KCl (Alfa, 99.99%). The mixture was handled under an argon atmosphere and the reaction was performed in a sealed quartz tube at 1023 K over a period of 7 d. The heating and cooling rates were 20 and 10 K h^{-1} , respectively. The crystals obtained were between 0.1 and 0.4 mm in size, stable in air, and dark green in color when ground.

Crystal data

$K_2SrNb_6Cl_{18}$	Mo $K\alpha$ radiation
$M_r = 1361.4$	Cell parameters from 34 reflections
Trigonal, $R\bar{3}$	$\theta = 2.7\text{--}12.5^\circ$
$a = 9.3025$ (8) Å	$\mu = 6.77$ mm $^{-1}$
$c = 25.9412$ (18) Å	$T = 273$ (2) K
$V = 1944.1$ (3) Å 3	Truncated cuboctahedron, black
$Z = 3$	$0.25 \times 0.22 \times 0.20$ mm
$D_x = 3.488$ Mg m $^{-3}$	

Data collection

Bruker P4 diffractometer	$R_{int} = 0.035$
ω scans	$\theta_{max} = 30.2^\circ$
Absorption correction: empirical via ψ scan (North <i>et al.</i> , 1968)	$h = -12 \rightarrow 12$
$T_{min} = 0.554$, $T_{max} = 0.923$	$k = -13 \rightarrow 13$
2837 measured reflections	$l = -36 \rightarrow 36$
1288 independent reflections	3 standard reflections
1072 reflections with $I > 2\sigma(I)$	every 297 reflections
	intensity decay: none

Table 1

Selected interatomic distances (Å).

Nb—Cl ⁱ	2.4497 (11)	Nb—Nb ⁱⁱ	2.9309 (7)
Nb—Cl ⁱⁱ	2.4505 (11)	Sr—Cl ⁱⁱⁱ	2.8954 (11)
Nb—Cl ⁱⁱⁱ	2.4631 (11)	K—Cl ^{iv}	3.2648 (12)
Nb—Cl ^{iv}	2.4635 (11)	K—Cl ^v	3.533 (2)
Nb—Cl ^v	2.5982 (11)	K—Cl ^{vi}	3.539 (2)
Nb—Nb ⁱ	2.9237 (6)	K—Cl ^{vii}	3.6182 (11)

Symmetry codes: (i) $x - y, x, -z$; (ii) $-y, x - y, z$; (iii) $-x + y, 1 - x, z$; (iv) $1 - x + y, 1 - x, z$; (v) $\frac{2}{3} + y, \frac{1}{3} - x + y, \frac{1}{3} - z$; (vi) $1 - y, x - y, z$.

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R(F) = 0.031$	$\Delta\rho_{max} = 0.76$ e Å $^{-3}$
$wR(F^2) = 0.073$	$\Delta\rho_{min} = -0.75$ e Å $^{-3}$
$S = 0.90$	Extinction correction: <i>SHELXL</i>
1288 reflections	(Sheldrick, 1997a)
43 parameters	Extinction coefficient: 0.00170 (11)
$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997a); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1367). Services for accessing these data are described at the back of the journal.

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