

ions are located above as well as below each window which align the windows so that straight tunnels are formed running along the *a* axis (see Fig. 2).

Two neighbouring VO₆ octahedra form an edge-sharing bioctahedron with a V...V distance of 2.928 (1) Å. Each distorted VO₆ octahedron consists of two very short, two medium and two long V—O bond distances with one of the O atoms, O(4), not being shared with P and coordinated to the Ba²⁺ ions only. The two very short V—O bonds are *cis* to each other and the bonds *trans* to them have long V—O distances. Atoms O(1) and O(2), which are *trans* to each other, have two medium-length V—O bond distances. Each PO₄ group has two O atoms that bridge the nonbonding V...V contact within a bioctahedron, one O atom being coordinated to a neighbouring bioctahedron, and one O atom to the Ba²⁺ ions only. The P tetrahedron is slightly distorted as shown by three long P—O distances of ~1.55 Å and a short one of 1.507 Å. Atom O(6), which is not shared with V, shows the shortest P—O bond. The Ba²⁺ ions are nine coordinated by O atoms from adjacent layers. The coordination number is determined on the basis of the maximum gap in the Ba—O distances ranked in increasing order.

The Sr phase is isostructural with Ba(VO₂)PO₄. It should be noted that the Pb analogue can also be obtained under similar reaction conditions. Preliminary work on a crystal of Pb(VO₂)PO₄ of reasonable quality showed that it was isostructural with the Ba compound. The indexed cell dimensions of the Pb compound are *a* = 5.505 (2), *b* = 9.913 (3), *c* = 8.511 (3) Å, β = 90.50 (3)° and *V* = 464.4 (3) Å. However, the Ca and Mg analogues have not been obtained under similar reaction conditions.

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Structure of KYF₄

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Abstract. Potassium yttrium tetrafluoride, KYF₄, *M*_r = 204.0, trigonal, *P*3₁, *a* = 14.060 (10), *c* = 10.103 (10) Å, *V* = 1729 (5) Å³, *Z* = 18, *D*_x = 3.49 g cm⁻³, $\lambda(\text{Ag K}\alpha)$ = 0.5594 Å, μ = 87.93 cm⁻¹, *F*(000) = 753, room temperature, final *R* = 0.038 for 3045 independent reflections. KYF₄ is a fluorite-related superstructure. Cations are distributed on three layers perpendicular to the *c* axis. In each layer,

chains of YF₁₂ groups formed by two F pentagonal bipyramids surrounding the Y atoms alternate with chains of F edge-shared distorted cubes surrounding the K atoms.

Introduction. In an initial study (Le Fur, 1977; Aléonard, Le Fur, Pontonnier, Gorius & Roux, 1978), we have shown that the hexagonal lattices of the phases

KLnF_4 (Ln = Gd...Lu and Y) and the cubic face-centred lattice of the fluorite were related by $a_H = (a_F\sqrt{2})\sqrt{3}$, $c_H = a_F\sqrt{3}$. On the other hand, the stacking of hexagonal close-packed layers of cations perpendicular to the direction c and the fact that every F polyhedron centred by a cation is surrounded by six 'empty' F polyhedra, have been shown to be characteristic of the fluorite arrangement along the direction [111], the cations being distributed on three layers at the levels $z = 0, \frac{1}{3}, \frac{2}{3}$. But, to our knowledge, up to now, there has been no precise structural determination of these compounds. The results in this paper concern single crystals of KYF_4 suitable for X-ray analysis, supplied by Dr Khaidukov.

Experimental. Crystal size: sphere 0.308 mm in diameter. Density not measured. Nonius CAD-4 diffractometer, graphite monochromator. Systematic absences $00l$, $l \neq 3n$. 25 reflections ($10.0 < \theta < 18.0^\circ$) for refining unit-cell dimensions. ω scan, scan width 1.20° , scan speed $0.028^\circ \text{ s}^{-1}$, background measuring time 21 s. 21 348 reflections collected ($3 < \theta < 34^\circ$), $\pm h$, $\pm k$, $\pm l$, $h_{\text{max}} = 25$, $k_{\text{max}} = 25$, $l_{\text{max}} = 18$. Two orientation (666 and $0\bar{6}\bar{6}$) and two intensity ($\bar{7}81$ and $8\bar{7}1$) control reflections were measured every 2 h and showed no significant variations. 7433 independent reflections obtained after averaging Friedel pairs ($R_{\text{int}} = 0.04$). Lorentz and polarization corrections, spherical absorption correction (maximum/minimum transmission factors 14.5/17.8). Crystal structure solved by classical methods: three-dimensional Patterson function for locating the Y and K atoms, successive Fourier syntheses for defining the Y and K sites and, at the end, a difference Fourier synthesis in order to obtain the positions of the F atoms.

The first refinements, using the Enraf-Nonius (1977) *SDP* program, with 1286 data obtained after omission of all the 'fluorite' reflections [$(h + l)$, $(k + l)$ and $(h + k - l) = 3n$] and all the superstructure reflections with $I < 8\sigma(I)$ allowed the K and Y atoms to be shared on different cationic sites and led to an R factor of 0.03.

In the final refinement, the use of 3045 data corresponding to all the independent reflections with $I > 3.3\sigma(I)$ (including, therefore, the 'fluorite' reflections) did not modify the previous results and led to a final R (based on F) = 0.038, $wR = 0.044$ ($w = 1$), $S = 3.665$, maximum $\Delta/\sigma = 0.06$, for 325 refined parameters. Maximum peak height in final difference Fourier synthesis was $1.25 \text{ e } \text{\AA}^{-3}$, negative peak height in the final difference Fourier synthesis was $1.17 \text{ e } \text{\AA}^{-3}$. The extinction coefficient refined in least squares = 2.5956×10^{-7} . Scattering factors for neutral atoms and f' , f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). A MicroVAX II computer was used for all calculations.

Discussion. Table 1 reports the final atomic coordinates and Table 2 gives the interatomic distances and some angles in the polyhedra surrounding the K and Y atoms.* Fig. 1 shows the layer at the mean level $z = \frac{1}{3}$: the cationic subcell is very near that of the fluorite, but the distortions of the anionic subcell modify the cationic environments and chains of KF_8 and YF_7 polyhedra appear.

Environment of the cations. It is to be noticed that, when the ideal fluorite structure is projected along the direction [111], the F cube surrounding the cations may be described as involving four F atoms located in a plane $\{\bar{1}01\}$ of the ideal cube with two of them along the direction [111]. The four others are located in a perpendicular plane. The planes $\{\bar{1}01\}$ divide the cube into two trigonal prisms.

All the K atoms are located at the centres of distorted F cubes. Four F atoms are approximately found in a plane $\{\bar{1}01\}$ and are characterized by short distances (K—F 2.6 Å). The other four F atoms, approximately distributed in the perpendicular plane, are a little farther (K—F \approx 2.9 Å) from the K atom.

The polyhedra surrounding K(7), K(9) and K(11) are more distorted than those surrounding K(8), K(10) and K(12), but all of them retain three almost square faces of the ideal cube.

In Table 2 interatomic distances and angles are given for the $\text{K}(7)\text{F}_8$ and $\text{K}(8)\text{F}_8$ polyhedra schematized in Fig. 2.

The Y atoms are surrounded by only seven F atoms, with two types of environments: type I for Y(1), Y(3) and Y(5) and type II for Y(2), Y(4) and Y(6). The YF_7 polyhedra of type I are characterized by the presence of four F atoms in an 'almost ideal' plane $\{\bar{1}01\}$. Around Y(1), for instance (Fig. 2), they form the distorted rectangular face F(1)—F(7)—F(1A)—F(8) with F(1) and F(1A) along the direction [111]. These atoms and two 'ideal' others [F(10) and F(9)] define a distorted trigonal prism which is monocapped by the seventh F atom F(4), located at the middle of the edge of the other trigonal prism forming the 'ideal' cube. However, the trigonal prisms are very distorted (Table 2), and the observed distances and angles allow one to regard the YF_7 polyhedra rather as pentagonal bipyramids (Table 2 and Fig. 2). They are all characterized by angles $F_{\text{ax}}\text{—Y—}F_{\text{eq}} \approx 90$, $F_{\text{eq}}\text{—Y—}F_{\text{eq}} \approx 72$ and $F_{\text{ax}}\text{—Y—}F_{\text{ax}}$ observed between 171 and 174° .

The polyhedra YF_7 of type II are characterized by the absence of F atoms in the direction [111] and can

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54888 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters (Å²) for KYF₄, with e.s.d.'s in parentheses*

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
Y(1)	0.12871 (6)	0.88764 (6)	0.333	0.62 (1)
Y(2)	0.28157 (7)	0.73104 (7)	0.32271 (8)	0.62 (1)
Y(3)	0.78003 (7)	0.20592 (6)	0.3391 (1)	0.52 (1)
Y(4)	0.94137 (7)	0.05770 (7)	0.3294 (2)	1.05 (1)
Y(5)	0.45256 (7)	0.54579 (7)	0.3250 (1)	0.99 (1)
Y(6)	0.60374 (7)	0.38662 (6)	0.34411 (7)	0.40 (1)
K(7)	0.7818 (2)	0.7190 (2)	0.3183 (2)	1.11 (4)
K(8)	0.2677 (2)	0.2208 (3)	0.3522 (3)	1.92 (5)
K(9)	0.1050 (2)	0.3945 (3)	0.3134 (4)	2.15 (6)
K(10)	0.6111 (2)	0.8778 (2)	0.3493 (3)	1.25 (4)
K(11)	0.9466 (3)	0.5591 (2)	0.3450 (3)	1.94 (5)
K(12)	0.4549 (3)	0.0652 (3)	0.3201 (4)	1.83 (6)
F(1)	0.1304 (7)	0.9342 (7)	0.1185 (9)	2.1 (2)
F(1A)	0.0692 (8)	0.8779 (8)	0.546 (1)	2.2 (2)
F(2)	0.2105 (8)	0.5934 (8)	0.472 (1)	2.5 (2)
F(2A)	0.334 (1)	0.7286 (7)	0.656 (1)	3.5 (3)
F(3)	0.7372 (5)	0.2023 (5)	0.5598 (8)	1.0 (1)
F(3A)	0.7901 (6)	0.2684 (6)	0.1305 (9)	1.4 (1)
F(4)	1.000 (1)	0.059 (1)	0.659 (2)	4.9 (4)
F(4A)	0.0489 (8)	0.1223 (8)	0.152 (2)	4.7 (2)
F(5)	0.3954 (6)	0.5413 (6)	0.5373 (8)	1.4 (1)
F(5A)	0.4618 (7)	0.5950 (6)	0.1110 (8)	1.5 (1)
F(6)	0.5440 (6)	0.2863 (6)	0.5317 (8)	1.4 (1)
F(6A)	0.6057 (7)	0.3368 (7)	0.009 (1)	2.0 (2)
F(7)	0.7727 (6)	0.7346 (6)	0.064 (1)	1.6 (1)
F(7A)	0.8133 (9)	0.7262 (6)	0.576 (1)	2.1 (2)
F(8)	0.2533 (6)	0.2289 (6)	0.606 (1)	1.8 (1)
F(8A)	0.8050 (6)	0.0719 (6)	0.4332 (8)	1.5 (1)
F(9)	0.1339 (6)	0.3916 (6)	0.5702 (8)	1.2 (1)
F(9A)	0.0941 (7)	0.4031 (8)	0.063 (1)	2.1 (2)
F(10)	0.1535 (6)	0.7561 (7)	0.4276 (9)	1.6 (1)
F(10A)	0.5874 (6)	0.8816 (7)	0.6044 (9)	1.4 (1)
F(11)	0.9390 (6)	0.5741 (7)	0.6029 (9)	1.5 (1)
F(11A)	0.9438 (6)	0.5316 (6)	0.0934 (8)	1.2 (1)
F(12)	0.4830 (7)	0.0692 (7)	0.5708 (9)	1.6 (2)
F(12A)	0.4469 (7)	0.0849 (6)	0.0572 (9)	1.7 (1)

Table 2 (cont.)

F(7A)—F(3A)	2.752	F(9A)—F(7A)—F(4A)	77
F(7A)—F(9A)	3.360	F(3A)—F(7A)—F(9A)	75
F(7A)—F(4A)	2.656	F(4A)—F(7A)—F(3A)	135
		F(7A)—F(3A)—F(12)	105
		F(12)—F(3A)—F(8)	87
		F(8)—F(3A)—F(7A)	66
F(9A)—F(1A)	3.693	F(3A)—F(12)—F(9A)	79
F(9A)—F(12)	2.868	F(9A)—F(12)—F(7)	80
F(12)—F(3A)	3.045	F(7)—F(12)—F(3A)	99
		F(7A)—F(9A)—F(1A)	83
		F(1A)—F(9A)—F(12)	100
		F(12)—F(9A)—F(7A)	95
F(8)—F(3A)	3.83	F(8)—F(4A)—F(7A)	65
F(8)—F(4A)	4.05	F(7A)—F(4A)—F(1A)	114
F(4A)—F(1A)	2.925	F(1A)—F(4A)—F(8)	75
		F(3A)—F(8)—F(4A)	80
		F(4A)—F(8)—F(7)	80
		F(7)—F(8)—F(3A)	84
K(8)F₈ polyhedron			
Edges			
F(8)—F(1)	2.893	F(1)—F(8)—F(7)	87
F(8)—F(7)	3.482	F(7)—F(8)—F(11A)	84
F(8)—F(11A)	3.357	F(11A)—F(8)—F(1)	103
		F(4)—F(1)—F(8)	89
		F(8)—F(1)—F(10A)	78
		F(10A)—F(1)—F(4)	99
F(8A)—F(4)	3.641	F(3)—F(8A)—F(4)	105
F(8A)—F(10A)	3.365	F(4)—F(8A)—F(10A)	93
F(8A)—F(3)	2.767	F(10A)—F(8A)—F(3)	79
		F(7)—F(3)—F(11A)	91
		F(11A)—F(3)—F(8A)	103
		F(8A)—F(3)—F(7)	73
F(7)—F(3)	3.460	F(3)—F(7)—F(4)	110
F(7)—F(4)	2.773	F(4)—F(7)—F(8)	82
F(4)—F(1)	2.998	F(8)—F(7)—F(3)	86
		F(1)—F(4)—F(7)	99
		F(7)—F(4)—F(8A)	69
		F(8A)—F(4)—F(1)	86
F(1)—F(10A)	3.624	F(1)—F(10A)—F(11A)	97
F(10A)—F(11A)	2.913	F(11A)—F(10A)—F(8A)	89
F(3)—F(11A)	2.900	F(8A)—F(10A)—F(1)	81
		F(3)—F(11A)—F(8)	98
		F(8)—F(11A)—F(10A)	82
		F(10A)—F(11A)—F(3)	85

Table 2. *Principal interatomic distances (Å) and bond angles (°) in the atomic arrangement of KYF₄, with e.s.d.'s in parentheses*

Environment of the K atoms

K(7)—F(9A)	2.552 (8)	K(10)—F(11)	2.555 (10)
K(7)—F(7)	2.587 (10)	K(10)—F(9A)	2.577 (12)
K(7)—F(8)	2.595 (10)	K(10)—F(10A)	2.603 (9)
K(7)—F(7A)	2.635 (10)	K(10)—F(10)	2.606 (9)
K(7)—F(3A)	2.898 (10)	K(10)—F(1A)	2.846 (13)
K(7)—F(12)	2.912 (11)	K(10)—F(8A)	2.856 (6)
K(7)—F(1A)	3.002 (9)	K(10)—F(5A)	2.982 (7)
K(7)—F(4A)	3.039 (15)	K(10)—F(2A)	3.022 (16)
K(8)—F(8A)	2.574 (9)	K(11)—F(8)	2.566 (7)
K(8)—F(8)	2.579 (10)	K(11)—F(11A)	2.568 (9)
K(8)—F(7)	2.593 (10)	K(11)—F(12A)	2.619 (10)
K(8)—F(10A)	2.660 (8)	K(11)—F(11)	2.620 (9)
K(8)—F(3)	2.833 (9)	K(11)—F(6)	2.896 (10)
K(8)—F(11A)	2.860 (9)	K(11)—F(5)	2.914 (10)
K(8)—F(1)	2.865 (8)	K(11)—F(10)	2.961 (7)
K(8)—F(4)	3.038 (20)	K(11)—F(3A)	3.044 (7)
K(9)—F(9A)	2.539 (11)	K(12)—F(12)	2.560 (9)
K(9)—F(10A)	2.578 (11)	K(12)—F(7)	2.615 (7)
K(9)—F(12A)	2.593 (10)	K(12)—F(11)	2.615 (11)
K(9)—F(9)	2.629 (8)	K(12)—F(12A)	2.678 (10)
K(9)—F(2)	2.905 (10)	K(12)—F(5A)	2.809 (11)
K(9)—F(7A)	2.908 (13)	K(12)—F(3)	2.869 (7)
K(9)—F(1)	2.915 (12)	K(12)—F(6A)	2.906 (11)
K(9)—F(5)	2.929 (7)	K(12)—F(9)	2.911 (9)

K(7)F₈ polyhedron

Edges			
F(7)—F(1A)	2.955	F(8)—F(7)—F(1A)	84
F(7)—F(8)	3.482	F(12)—F(7)—F(8)	89
F(7)—F(12)	3.359	F(1A)—F(7)—F(12)	106
		F(9A)—F(1A)—F(4A)	69
		F(4A)—F(1A)—F(7)	111
		F(7)—F(1A)—F(9A)	74
Angles			
F(8)—F(7)—F(1A)	84		
F(12)—F(7)—F(8)	89		
F(1A)—F(7)—F(12)	106		
F(9A)—F(1A)—F(4A)	69		
F(4A)—F(1A)—F(7)	111		
F(7)—F(1A)—F(9A)	74		

YF₇ polyhedra of type I

Y(1)—F(7)	2.127 (9)	Y(3)—F(12A)	2.127 (10)	Y(5)—F(10A)	2.142 (10)
Y(1)—F(8)	2.161 (9)	Y(3)—F(11)	2.155 (10)	Y(5)—F(9A)	2.160 (12)
Y(1)—F(9)	2.209 (9)	Y(3)—F(7A)	2.239 (10)	Y(5)—F(2A)	2.214 (8)
Y(1)—F(4)	2.238 (10)	Y(3)—F(6A)	2.222 (7)	Y(5)—F(11A)	2.248 (8)
Y(1)—F(10)	2.257 (10)	Y(3)—F(3A)	2.260 (8)	Y(5)—F(5A)	2.253 (8)
Y(1)—F(1)	2.263 (9)	Y(3)—F(8A)	2.289 (9)	Y(5)—F(5)	2.281 (8)
Y(1)—F(1A)	2.288 (10)	Y(3)—F(3)	2.303 (8)	Y(5)—F(12)	2.289 (10)

Monocapped trigonal prism Y(1)F₇

F(1A)—F(8)	3.217	F(10)—F(1A)—F(8)	57
F(8)—F(10)	2.896	F(1A)—F(8)—F(10)	54
F(10)—F(1A)	2.793	F(8)—F(10)—F(1A)	69
F(7)—F(1)	3.302	F(9)—F(7)—F(1)	51
F(1)—F(9)	2.695	F(7)—F(1)—F(9)	56
F(9)—F(7)	2.869	F(1)—F(9)—F(7)	73

Rectangular faces

F(1A)—F(7)	2.955	F(8)—F(1A)—F(7)	88
F(7)—F(1)	3.302	F(1A)—F(7)—F(1)	89
F(1)—F(8)	2.893	F(7)—F(1)—F(8)	87
F(8)—F(1A)	3.217	F(1)—F(8)—F(1A)	92
F(1A)—F(7)	2.955	F(10)—F(1A)—F(7)	74
F(7)—F(9)	2.869	F(1A)—F(7)—F(9)	91
F(9)—F(10)	2.447	F(7)—F(9)—F(10)	81
F(10)—F(1A)	2.792	F(9)—F(10)—F(1A)	104
F(8)—F(1)	2.893	F(10)—F(8)—F(1)	90
F(1)—F(9)	2.695	F(8)—F(1)—F(9)	77
F(9)—F(10)	2.447	F(1)—F(9)—F(10)	106
F(10)—F(8)	2.896	F(9)—F(10)—F(8)	81

Table 2 (cont.)

Pentagonal bipyramid Y(1)F₇					
Pentagonal base					
F _{eq} —F _{eq} (av.)	2.694	F _{ax} —Y(1)—F _{eq} (av.)	73.5		
F(1)—F(9)	2.695	F(1)—Y(1)—F(9)	74.1		
F(9)—F(10)	2.447	F(9)—Y(1)—F(10)	66.4		
F(10)—F(1A)	2.793	F(10)—Y(1)—F(1A)	75.8		
F(1A)—F(4)	2.861	F(1A)—Y(1)—F(4)	78.4		
F(4)—F(1)	2.677	F(4)—Y(1)—F(1)	73.0		
Pyramids					
F _{ax} —F _{eq} (av.)	3.132	F _{ax} —Y(1)—F _{eq} (av.)	91		
F(8)—F(1)	2.893	F(8)—Y(1)—F(1)	82		
F(8)—F(9)	3.47	F(8)—Y(1)—F(9)	105		
F(8)—F(10)	2.896	F(8)—Y(1)—F(10)	82		
F(8)—F(1A)	3.217	F(8)—Y(1)—F(1A)	93		
F(8)—F(4)	3.187	F(8)—Y(1)—F(4)	93		
F_{ax}—F_{eq} (av.)					
F(7)—F(1)	3.302	F(7)—Y(1)—F(1)	97		
F(7)—F(9)	2.869	F(7)—Y(1)—F(9)	83		
F(7)—F(10)	3.47	F(7)—Y(1)—F(10)	104		
F(7)—F(1A)	2.955	F(7)—Y(1)—F(1A)	84		
F(7)—F(4)	2.773	F(7)—Y(1)—F(4)	79		
F_{ax}—Y(1)—F_{ax}					
F(8)—Y(1)—F(7)	171.5 (3)				
YF₇ polyhedra of type II					
Y(2)—F(2A)	2.194 (9)	Y(4)—F(1A)	2.175 (13)	Y(6)—F(6A)	2.196 (9)
Y(2)—F(5)	2.202 (9)	Y(4)—F(4)	2.175 (13)	Y(6)—F(3)	2.202 (8)
Y(2)—F(5A)	2.203 (9)	Y(3)—F(7A)	2.223 (7)	Y(6)—F(3A)	2.210 (9)
Y(2)—F(2)	2.234 (11)	Y(4)—F(4A)	2.227 (13)	Y(6)—F(6)	2.210 (9)
Y(2)—F(2)	2.255 (10)	Y(4)—F(1)	2.231 (10)	Y(6)—F(12)	2.243 (7)
Y(2)—F(9)	2.260 (6)	Y(4)—F(4A)	2.240 (15)	Y(6)—F(6)	2.259 (8)
Y(2)—F(10)	2.264 (10)	Y(4)—F(8A)	2.279 (9)	Y(6)—F(11A)	2.265 (6)
Pentagonal bipyramid Y(2)F₇					
Pentagonal base					
F _{eq} —F _{eq} (av.)	2.650	Average angle	72.5		
F(2A)—F(2)	2.718	F(2A)—Y(2)—F(2)	75.3		
F(2)—F(10)	2.813	F(2)—Y(2)—F(10)	77.0		
F(10)—F(9)	2.447	F(10)—Y(2)—F(9)	65.5		
F(9)—F(2)	2.671	F(9)—Y(2)—F(2)	72.9		
F(2)—F(2A)	2.603	F(2)—Y(2)—F(2A)	72.0		
Pyramids					
F _{ax} —F _{eq} (av.)	3.130	F _{ax} —Y(2)—F _{eq} (av.)	89.9		
F(5)—F(2A)	3.376	F(5)—Y(2)—F(2A)	100		
F(5)—F(2)	2.885	F(5)—Y(2)—F(2)	81		
F(5)—F(10)	3.070	F(5)—Y(2)—F(10)	87		
F(5)—F(9)	3.212	F(5)—Y(2)—F(9)	92		
F(5)—F(2)	3.105	F(5)—Y(2)—F(2)	89		
F_{ax}—F_{eq} (av.)					
F(5A)—F(2A)	3.142	F _{ax} —Y(2)—F _{eq} (av.)	90.4		
F(5A)—F(2)	3.015	F(5A)—Y(2)—F(2A)	87		
F(5A)—F(2)	3.44	F(5A)—Y(2)—F(2)	101		
F(5A)—F(10)	3.075	F(5A)—Y(2)—F(10)	87		
F(5A)—F(9)	2.944	F(5A)—Y(2)—F(9)	83		
F(5A)—F(2)	3.237	F(5A)—Y(2)—F(2A)	94		
F_{ax}—Y(2)—F_{ax}					
F(5)—Y(2)—F(5A)	173.1 (3)				

no longer be described in terms of the trigonal prisms of the ideal cube, but, like the polyhedra of the type I, they correspond to pentagonal bipyramids (Table 2). Around Y(2) (Fig. 1), the pentagonal base is formed by the atoms F(2), F(2A), F(10), F(9) and F(2A); F(5) and F(5A) are the two axial atoms.

In the two types of environments, the axial bonds Y—F_{ax} are generally a little shorter than the equatorial bonds Y—F_{eq} (approximately 2.17 and 2.25 Å, respectively).

Characteristics of the structure. Fig. 1 shows that in the cationic layers, two polyhedra of the types I and II share one of their equatorial edges to form

Y₂F₁₂ groups. These groups share one of their equatorial corners to form chains running along one of the three trigonal directions (respectively [210], [110] and [120] at the levels 0, $\frac{1}{3}$ and $\frac{2}{3}$).

In the cationic planes (Fig. 1), the bipyramid chains are connected by the K atoms. The polyhedra KF₈ also form chains. Each of their two shared edges bonds the axial F atoms of two bipyramids YF₇, on either side of the chains.

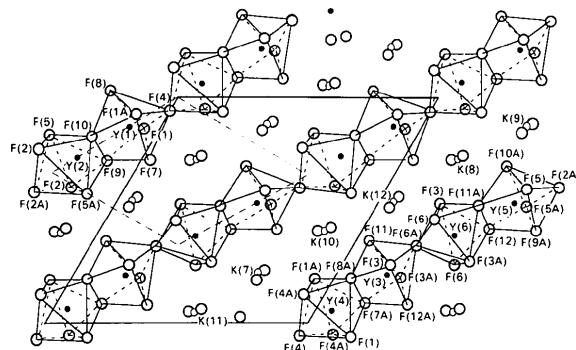


Fig. 1. Projection along the *c* axis of the cationic layer of KYF₄ at the level $z = \frac{1}{3}$ (broken lines: KTIF₄ lattice).

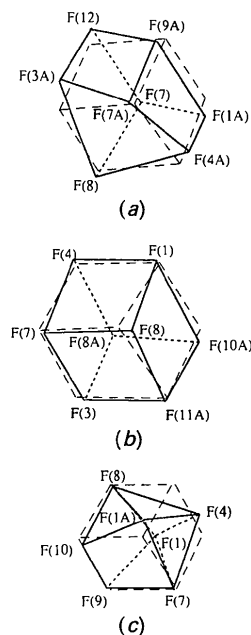


Fig. 2. Schematic representation of the environments of the cations showing their deformation from the 'ideal fluorite cube' (in thin dotted lines): (a) K(7)F₈ environment, with F(7), F(8), F(7A), F(9A) approximately in the ideal plane (011); (b) K(8)F₈ environment, with F(8), F(7), F(8A), F(10A) approximately in the ideal plane (101); (c) Y(1)F₇ environment, described as either a trigonal prism bounded by the almost ideal plane (110) [F(1), F(8), F(1A), F(7)] and mon capped by F(4), or a pentagonal bipyramid with F_{eq} = F(4), F(1A), F(10), F(9), F(1) and F_{ax} = F(7), F(8).

This study shows that these phases and the compound KTlF₄ described by Hebecker (1975) have the same structural characteristics. But according to Hebecker, KTlF₄ would crystallize with a unit cell three times smaller ($a' = a/\sqrt{3}$, $c' = c$) (see Fig. 1) involving only one Tl₂F₁₂ group per layer.

The larger unit cell of KYF₄ which contains three Y₂F₁₂ groups per layer is to be attributed to the small extent of distortion observed between two adjacent Ln₂F₁₂ groups along the three trigonal directions; as is probably the case with the compounds NaScF₄ (Thomas & Karraker, 1966) and NaInF₄ (Grannec, Champarnaud & Portier, 1970).

To our knowledge, up to now, bipyramidal surroundings have never been reported for Y. Such surroundings have been mentioned only for the lanthanides with the smallest ionic radii: Lu, Yb and Tm, for instance in the compounds β -KYb₂F₇ (Le Fur, Aléonard, Gorius & Roux, 1980), CsYb₃F₁₀ (Aléonard, Roux & Lambert, 1982) and β -RbLu₃F₁₀

(Arbus, Fournier, Cousseins, Védérine & Chevalier, 1982). In these compounds, the bipyramids are always characterized by bond lengths Ln—F_{ax} a little shorter than Ln—F_{eq}, as observed in KYF₄.

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Na₂[Ru(NO₂)₄(NO)(OH)].2H₂O – a Redetermination

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Abstract. Disodium hydroxotetranitro(nitroso)ruthenate(III) dihydrate, $M_r = 414.1$, monoclinic, $C2/m$, $a = 12.8765$ (6), $b = 14.5867$ (9), $c = 7.4478$ (4) Å, $\beta = 121.521$ (3)°, $V = 1192.5$ Å³, $Z = 4$, $D_x = 2.306$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.430$ mm⁻¹, $F(000) = 808$, $T = 298$ K, $R = 0.0251$ for 1760 unique observed reflections. We have re-determined the structure of the title compound by single-crystal X-ray diffraction. After recalculation of the bond lengths derived in the neutron diffraction study, the distances involving the non-H atoms show no significant differences between the two determinations.

Introduction. As part of ongoing research related to the geometrical aspects of tetranitro- and hexanitrometallate species, single crystals of Na₂[Ru(NO₂)₄(NO)(OH)].2H₂O were prepared. A crystal structure analysis using neutron diffraction had previously been published (Simonsen & Mueller, 1965), but both the R value of 0.098 and the uncertainties in

the bond lengths of up to 0.007 Å were rather high. In order to improve the accuracy of the structure, a dataset was collected using Mo $K\alpha$ X-radiation. In addition to the expected random errors between the two determinations, there appear to be marked systematic errors.

Experimental. Compound prepared by dissolution of excess NaNO₂ in an aqueous solution of RuCl₃ until colour change observed, crystals obtained by slow evaporation from resulting solution. Yellow columnar crystal, 0.78 × 0.39 × 0.39 mm, Stoe STADI-4 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ X-radiation, cell parameters from 2θ values of 44 reflections measured at $\pm \omega$ ($30 \leq 2\theta \leq 32^\circ$). For data collection $T = 298$ K, ω - 2θ scans using the learnt-profile method (Clegg, 1981), $2\theta_{\text{max}} 60^\circ$, $h -18 \rightarrow 15$, $k 0 \rightarrow 20$, $l 0 \rightarrow 10$, 3 standard reflections, no significant crystal movement or decay, absorption correction using ψ scans (min. and max. transmission factors 0.470 and 0.561, respectively), 1811 unique reflections, giving 1760 with $F \geq 4\sigma(F)$ for structure refinement [using full-matrix least

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