

Discussion. The structure of $\text{La}_4\text{Mo}_2\text{O}_{11}$ is composed of isolated Mo_2O_{10} cluster units resulting from the edge sharing of two highly distorted MoO_6 octahedra related by an inversion centre (Fig. 1). These cluster units are held together by the La atoms to form the three-dimensional framework (Fig. 2). The distance between the two Mo atoms of the dimeric unit is 2.5905 (5) Å and corresponds approximately to a single bond. This is longer than the distance observed in the Mo_2 pairs occurring in $\text{La}_2\text{Mo}_2\text{O}_7$ [2.478 (4) Å], $\text{Y}_3\text{Mo}_2\text{O}_{12}$ [2.496 (1) Å] and MoO_2 [2.511 (1) Å] (Brant & Skapski, 1967) in which the formal Mo oxidation state is lower. In the last mentioned two compounds, the Mo_2 dimers form quasi-linear chains and are separated from each other by 3.221 (1) and 3.112 (1) Å respectively. In $\text{La}_2\text{Mo}_2\text{O}_7$, the Mo_2O_{10} units share corners in two dimensions to form layers which are separated from each other by the La atoms. The shortest distance between Mo_2 groups is 5.3684 (5) Å in $\text{La}_4\text{Mo}_2\text{O}_{11}$ and 3.888 Å in $\text{La}_2\text{Mo}_2\text{O}_7$. In $\text{Nd}_4\text{Re}_2\text{O}_{11}$, the Re atoms in the pairs are only 2.421 (1) Å apart, which reflects the presence of an $\text{Re}=\text{Re}$ double bond. The Mo—O distances spread over a wide range, 1.847 (2)–2.207 (2) Å, with a mean value of 1.989 Å. The La(1) atoms and their O-atom environment are in a somewhat deformed fluorite arrangement. The La(1)—O distances are in the range 2.3853 (2)–2.637 (2) Å, with a mean value of 2.524 Å. The coordination around La(2) is also eightfold and can be described as a trigonal prism whose two rectangular faces are capped. The La(2)—O distances are in the range 2.368 (2)–2.903 (2) Å, with a mean value of

2.558 Å. The shortest distance between La atoms is 3.8258 (3) Å [La(2)—La(2)].

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Structure of Potassium Hexarhenium Nonabromide Pentasulfide: $\text{KRe}_6\text{S}_5\text{Br}_9$

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Abstract. $\text{K}^+[\text{Re}_6(\text{S}_5\text{Br}_3)\text{Br}_6]^-$, $M_r = 2035.80$, monoclinic, $C2/c$, $a = 16.914$ (11), $b = 9.526$ (4), $c = 17.671$ (11) Å, $\beta = 127.41$ (2)°, $V = 2261.5$ Å³, $Z = 4$, $D_x = 5.979$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 488.14$ cm⁻¹, $F(000) = 3456$, $T = 293$ K, 101 parameters refined, $R = 0.034$ for 2077 reflections with $I \geq 3\sigma(I)$. The structure consists of a rock-salt-like ionic packing of K cations and complex anions; the latter

are built from an octahedral Re_6 cluster surrounded by a pseudo-cube of eight disordered ligands (5 S + 3 Br) and then an octahedron of six terminal Br atoms. In contrast to the parent, but not isostructural compound $\text{KRe}_6\text{Se}_5\text{Cl}_9$, the environment of the K^+ ion is prismatic, instead of being octahedral, the signature of steric constraints.

Introduction. A number of members of the series $M^+[\text{Re}_6\text{Y}_5\text{X}_9]^-$ have been previously synthesized

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(for instance: $M = Li, Na, K, Cu, X = Cl, Y = Se; M = Rb, X = Cl, Y = S, Se; M = Ag, X = Cl, Br, Y = S, Se$). All these compounds crystallize in the cubic system, space group $Pn\bar{3}$, as exemplified by the crystal structure of the representative compound $KRe_6Se_5Cl_9$ (Perrin, Leduc, Potel & Sergent, 1990) in which the environment of the K^+ ion is an almost perfect octahedron of six apical Cl belonging to six different units. From powder X-ray diffraction patterns, it appeared obvious that the thio-bromo potassium homologue was not isostructural with the others, and its crystal structure determination has been carried out in order to explain this discrepancy.

Experimental. $KRe_6S_5Br_9$ was prepared from a near stoichiometric amount of KBr and elemental Re, S and Br in evacuated silica tubes at 1118 K. Liquid bromine was added under an argon flow to the powdered mixture of the other components. This was then frozen at 77 K before sealing the tube under a residual atmosphere of about 1 Pa of argon. A slight excess of bromine, typically 10%, was used in order to take into account the formation of a small amount of volatile by-products like S_2Br_2 , $ReBr_3$ or $ReBr_5$. Red crystals were naturally grown by chemical transport during the synthesis process owing to the presence of these gaseous phases, and no specific crystal-growth experiment was needed for the purpose of structure determination. Crystals were analyzed by energy dispersive spectroscopy and their Gandolfi photographs were compared to the X-ray diffraction pattern of the powder for further identification. A translucent rhombic-shaped platelet-like single crystal ($0.204 \times 0.068 \times 0.023$ mm) was selected for the intensity data collection, carried out on an Enraf-Nonius CAD-4 diffractometer fitted with graphite-monochromated $Mo K\alpha$ radiation. The cell dimensions were obtained and refined by least squares from setting angles of 25 centred reflections ($6 \leq \theta \leq 12^\circ$). 3576 reflections were recorded by a θ - 2θ scan in the range $2 \leq 2\theta \leq 60^\circ$ and $0 \leq h \leq 23$, $0 \leq k \leq 13$, $-24 \leq l \leq 24$. The intensities were corrected for Lorentz-polarization and absorption factors, the latter by the use of the *DIFABS* (Walker & Stuart, 1983) procedure (correction factors 0.699–1.632). The structure was solved by direct methods (*MULTAN84*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1984) and successive difference Fourier syntheses. After averaging the symmetry related reflections ($R_{int} = 0.045$), 2077 independent reflections with $I \geq 3\sigma(I)$ were used in the full-matrix least-squares anisotropic (β_{ij}) refinements on F ; secondary extinction refined, $g = 5.64 \times 10^{-8}$; $R = 0.034$, $wR = 0.045$ $\{w = 4F_o^2/[\sigma^2(I^2) + (0.07F_o^2)^2]^{1/2}\}$, $S = 0.967$; $(\Delta/\sigma)_{max} = 0.0$, $\Delta\rho_{max} = 2.5 e \text{ \AA}^{-3}$ (for peaks very close to the Re positions). The scattering factors were taken from *International*

Tables for X-ray Crystallography (1974, Vol. IV). The statistical occupancy of the eight positions surrounding the metal cluster has been taken into account by the introduction of a pseudo atom $L = \frac{5}{8}S + \frac{3}{8}Br$, using the program *DISORDER* (B. A. Frenz & Associates, Inc., 1985), and the multiplicity of these positions has been refined. The structure illustrations were drawn by *ORTEP* (Johnson, 1965). All calculations were performed on a MicroVAX 3100 using the *SDP* programs (B. A. Frenz & Associates, Inc., 1985). Table 1* gives the atomic coordinates and the statistical occupancy of cluster-unit 'inner' positions, Table 2 the bond distances and a selection of bond angles. Fig. 1 displays the crystal-structure packing and Fig. 2 the K^+ -ion environment.

Discussion. $KRe_6S_5Br_9$ presents an unexpected symmetry as it is not isostructural with the previously reported members of the $M^+.[Re_6Y_5X_9]^-$ series. The structure of $KRe_6S_5Br_9$ is built, as in the case of the previous compounds, from the stacking of K^+ ions and anionic units arranged in a rock-salt-like packing. This is more easily seen when applying the matrix transformation $0 \ -1 \ -0.5 \ -1 \ 0 \ -0.5$
 $0 \ 10$ which gives a pseudo unit cell with $a = 12.992$, $b = 13.512$, $c = 12.992 \text{ \AA}$, $\alpha = 94.15$, $\beta = 94.21$, $\gamma = 94.15^\circ$, reminiscent of the unit cells of the actually cubic family $MRe_6Y_5X_9$.

The anionic unit is the usual octahedral Re_6 cluster, surrounded by a cube of eight 'inner' ligands L (about $\frac{5}{8}S + \frac{3}{8}Br$) and then an octahedron of six 'terminal' Br atoms. The refinement of L -position multiplicities (Table 1) shows that they are unequally occupied: $L1$ and $L2$ are close to $\frac{1}{2}Br + \frac{1}{2}S$, whereas $L3$ and $L4$ are about $\frac{1}{3}Br + \frac{2}{3}S$. Such a feature has been commonly encountered in related compounds, such as $KRe_6Se_5Cl_9$ (Perrin, Leduc, Potel & Sergent, 1990) and $KRe_6Se_5Cl_7(OH)_2 \cdot H_2O$ (Perrin, 1990). The apical positions are fully occupied by six Br atoms, the multiplicity of these, when refined, being not significantly different from unity. So the determined stoichiometry does not differ significantly from the expected one, with integer indexes. This result is in good agreement with the optical and electrical properties of the material, which imply a fully filled valence band, with 24 valence electrons per Re_6 cluster (Potel, Perrin, Perrin & Sergent, 1986).

This conclusion is confirmed by the averaged Re—Re bond, 2.608 Å, characteristic of a two-electron metal-metal bond; this value is exactly identical to the Re—Re distance in the parent com-

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55280 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0337]

Table 1. *Positional parameters, equivalent isotropic thermal parameters (\AA^2) and refined occupancies of cluster-unit 'inner' positions*

$$B_{\text{eq}} = (4/3)[a^2 B(1,1) + b^2 B(2,2) + c^2 B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Re1	0.05305 (3)	0.07929 (6)	0.11710 (3)	1.080 (9)
Re2	0.03298 (3)	-0.17530 (6)	0.05552 (3)	1.110 (9)
Re3	-0.12232 (3)	-0.01296 (6)	-0.02419 (3)	1.111 (9)
Br1	0.13024 (8)	0.1817 (2)	0.27946 (8)	2.13 (3)
Br2	0.0788 (1)	0.4143 (2)	0.6327 (1)	2.62 (3)
Br3	-0.28604 (8)	0.0396 (2)	0.44887 (9)	2.48 (3)
L1	-0.1395 (1)	0.2603 (2)	0.4168 (1)	1.95 (4)
L2	0.1019 (1)	0.2362 (2)	0.4663 (1)	1.81 (4)
L3	0.1965 (2)	0.0787 (3)	0.6864 (2)	1.83 (5)
L4	-0.4649 (2)	0.3972 (3)	0.3613 (2)	1.90 (5)
K	0.500	-0.0330 (8)	0.250	3.5 (1)

Pseudo atom	Multiplicity	τ_{Br}	τ_{S}
$L = \frac{1}{3}\text{Br} + \frac{2}{3}\text{S}$	-	0.375	0.625
L1	1.165 (6)	0.576 (5)	0.424 (5)
L2	1.098 (6)	0.495 (5)	0.505 (5)
L3	0.866 (6)	0.212 (5)	0.788 (5)
L4	0.832 (6)	0.170 (2)	0.830 (5)
Average	0.990 (6)	0.363 (5)	0.637 (5)
Observed stoichiometry	-	2.90 (4)	5.09 (4)
Expected stoichiometry	-	3	5

Table 2. *Bond distances (\AA) and bond angles ($^\circ$)*

Re1—Re2	2.5946 (8)	Re2—L3 ⁱⁱⁱ	2.464 (2)
Re1—Re2'	2.6203 (7)	Re2—L4 ⁱⁱⁱ	2.453 (4)
Re1—Re3	2.6090 (5)	Re3—Br3 ⁱⁱⁱ	2.521 (2)
Re1—Re3'	2.6086 (9)	Re3—L1 ⁱⁱⁱ	2.521 (2)
Re1—Br1	2.517 (1)	Re3—L2 ⁱⁱ	2.523 (2)
Re1—L1 ⁱ	2.556 (2)	Re3—L3 ⁱⁱ	2.486 (3)
Re1—L2 ⁱⁱ	2.566 (2)	Re3—L4 ⁱⁱ	2.456 (3)
Re1—L3 ⁱⁱⁱ	2.458 (3)	Br1 ⁱⁱⁱ —K	3.334 (6)
Re1—L4 ⁱⁱⁱ	2.463 (3)	Br2 ⁱⁱⁱ —K	3.273 (3)
Re2—Re3	2.6038 (7)	Br2 ⁱⁱⁱ —K	3.273 (3)
Re2—Re3'	2.6120 (8)	Br3—K	3.236 (2)
Re2—Br2 ⁱⁱⁱ	2.522 (2)	Br3 ⁱⁱⁱ —K	3.236 (2)
Re2—L1 ⁱⁱⁱ	2.547 (1)		
Re2—L2 ⁱⁱⁱ	2.533 (3)		
Re2—Re1—Re2	89.96 (2)	L1—Re2—L3	176.58 (8)
Re2—Re1—Re3	60.05 (2)	L1—Re2—L4	89.21 (7)
Re2—Re1—Re3	60.27 (2)	L2—Re2—L3	89.48 (9)
Re2—Re1—Re3	59.93 (2)	L2—Re2—L4	176.79 (9)
Re2—Re1—Re3	59.73 (2)	L3—Re2—L4	90.02 (9)
Re3—Re1—Re3	90.02 (2)	L1—Re3—L2	178.39 (8)
Re1—Re2—Re1	90.04 (2)	L1—Re3—L3	90.02 (8)
Re1—Re2—Re3	60.25 (2)	L1—Re3—L4	89.74 (9)
Re1—Re2—Re3	60.13 (2)	L2—Re3—L3	89.20 (8)
Re1—Re2—Re3	59.91 (2)	L2—Re3—L4	90.91 (8)
Re1—Re2—Re3	59.82 (2)	L3—Re3—L4	175.1 (1)
Re3—Re2—Re3	90.06 (2)	Br1—K—Br1	70.8 (1)
Re1—Re3—Re1	89.98 (2)	Br1—K—Br2	131.9 (1)
Re1—Re3—Re2	59.70 (2)	Br1—K—Br2	84.01 (7)
Re1—Re3—Re2	60.25 (2)	Br1—K—Br3	78.92 (7)
Re1—Re3—Re2	60.36 (2)	Br1—K—Br3	122.7 (1)
Re1—Re3—Re2	59.60 (2)	Br2—K—Br2	139.6 (2)
L1—Re1—L2	90.16 (7)	Br2—K—Br3	81.69 (7)
L1—Re1—L3	89.85 (9)	Br2—K—Br3	89.82 (8)
L1—Re1—L4	175.91 (8)	Br3—K—Br3	155.3 (3)
L2—Re1—L3	176.06 (8)	Re1—Br1—K	107.57 (4)
L2—Re1—L4	89.77 (8)	Re2—Br2—K	123.0 (1)
L3—Re1—L4	89.9 (1)	Re3—Br3—K	123.98 (7)
L1—Re2—L2	91.11 (7)		

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

pound $\text{KRe}_6\text{Se}_5\text{Cl}_9$. Moreover, the distances and angles (Table 2) show that the cluster is hardly distorted. The Re—L1 and Re—L2 distances are systematically slightly larger than the Re—L3 and Re—L4 ones, in relation to the difference in anionic occupation of the two types of L sites. The Re—Br

distances (average 2.520 \AA) are, as expected, slightly shorter than the corresponding values previously determined for Re—Br—Re bridges: 2.604 (2)–2.618 (3) \AA (Perrin, Leduc & Sergent, 1991).

The structure cohesion is achieved by the ionic interaction of anionic cluster units and K cations. Owing to rock-salt-like packing, each cluster is octahedrally surrounded by six K^+ ions and *vice versa*. However, the first shell of coordination of the K^+ ion is a prism of six terminal Br atoms (average K—Br distance = 3.281 \AA , to be compared to the sum of ionic radii, 3.34 \AA), in contrast to $\text{KRe}_6\text{Se}_5\text{Cl}_9$ where the K^+ ion lies at the centre of a perfect octahedron of six terminal Cl atoms. This essential difference between the two structures is a result of steric constraints. In fact, from the unit cells of $\text{AgRe}_6\text{Se}_5\text{Cl}_9$ ($a = 12.75 \text{\AA}$) and $\text{AgRe}_6\text{S}_5\text{Br}_9$ ($a = 12.96 \text{\AA}$), one can approximate a molecular volume

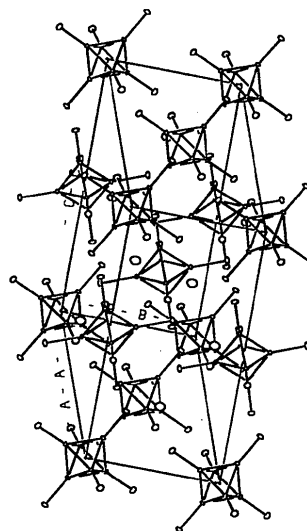


Fig. 1. The unit cell of $\text{KRe}_6\text{S}_5\text{Br}_9$; ligands L omitted and K^+ -ion probability increased for clarity.

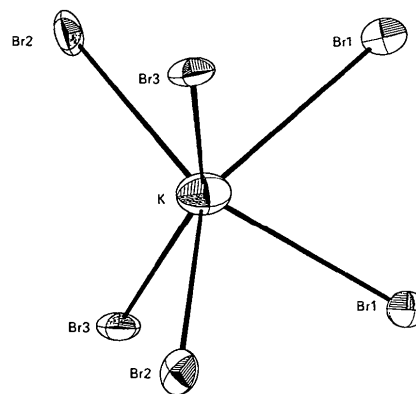


Fig. 2. The prismatic environment of the K^+ ion in the structure of $\text{KRe}_6\text{S}_5\text{Br}_9$; this contrasts with the octahedral environment in the structure of the parent compound $\text{KRe}_6\text{Se}_5\text{Cl}_9$.

increase of 26 Å³ when going from the Se—Cl to the S—Br systems. For KRe₆S₅Br₃, this would lead to a hypothetical cubic cell of about 13.25 Å (*i.e.* a cell volume of 2326.2 Å³), a value above those we have observed for any cubic material in this family. The passage to the monoclinic symmetry is in fact accompanied by a reduction in the cell volume to the actual value of 2261.5 Å³ at the expense of a distortion of the K⁺ site. The latter results in a clear reduction of one pair of Re—Br—K angles (107° instead of 123° for the other), while in KRe₆Se₅Cl₃, all the Re—Cl—K angles are equal (125.35°).

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Structures of Some Quasi-Two-Dimensional Molecular Conductors. I. Structure of κ -Tetrakis(3,4-methylenedithio-2,2',5,5'-tetrathiafulvalen)ium Tetracyanoplatinate Dihydrate: κ -(MDTTTF)₄Pt(CN)₄(H₂O)₂

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Abstract. Tetrakis[2-(1,3-dithiol-2-ylidene)-[1,3]dithiolo[4,5-*d*][1,3]dithiol]ium tetracyanoplatinate dihydrate, [C₇H₄S₆]₄[Pt(CN)₄].2H₂O, *M_r* = 1457.2, triclinic, *P*1, *a* = 8.569 (9), *b* = 9.991 (5), *c* = 14.811 (5) Å, α = 108.84 (3), β = 95.10 (4), γ = 90.37 (7)°, *V* = 1194.5 Å³, *Z* = 1, *D_x* = 2.03 g cm⁻³, *Mo K*α, λ = 0.71073 Å, μ = 40.16 cm⁻¹, *F*(000) = 718, *T* = 293 K, *R* = 0.054 for 3234 reflections with *I* ≥ 3σ(*I*). The crystal structure contains alternating layers of MDTTTF molecules and Pt(CN)₄²⁻ anions. The MDTTTF packing presents a κ mode characterized by face-to-face dimers of MDTTTF molecules which are orthogonal to adjacent dimers. The anionic sheet contains water molecules located at cavities generated by the organic dimers and the Pt(CN)₄ units. The latter form a one-dimensional polymer through hydrogen bonding as indicated by

C—N···O1*W* of 2.848 (14) and 3.121 (14) Å. Strong intra- and interdimer S···S interactions (3.38–3.58 Å) are observed.

Introduction. In previous papers (Ouahab, Padiou, Grandjean, Garrigou-Lagrange, Delhaes & Bencharif, 1989; Garrigou-Lagrange, Ouahab, Grandjean & Delhaes, 1990; Ouahab, Triki, Grandjean, Bencharif, Garrigou-Lagrange & Delhaes, 1991; Garrigou-Lagrange, Amiéll, Dupart, Delhaes, Ouahab, Fettouhi, Triki & Grandjean 1991) we reported the synthesis and X-ray and physical characterizations of conducting salts based on symmetrical TTF (tetrathiafulvalene) derivatives and tetracyanometalate dianions, which are, in our opinion, of great interest, especially as a recent investigation revealed the first example of superconductivity in this kind of polyanion material (Mori, Hirabayashi, Tanaka, Mori, Maruyama & Inokuchi, 1991). We report here,

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