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Kingstonite, $(Rh,Ir,Pt)_3S_4$, a new mineral species from Yubdo, Ethiopia

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ABSTRACT

Kingstonite, ideally Rh_3S_4, is a new mineral from the Bir Bir river, Yubdo District, Wallaga Province, Ethiopia. It occurs as subhedral, tabular elongate to anhedral inclusions in a Pt-Fe nugget with the associated minerals isoferroplatinum, tetraferroplatinum, a Cu-bearing Pt-Fe alloy, osmium, enriched oxide remnants of osmium, laurite, bowieite, ferrorhodsite and cuprorhodsite. It is opaque with a metallic lustre, has a black streak, is brittle and has a subconchoidal fracture and a good cleavage parallel to [001]. VHN_{25} is 871−920 kg/mm^2. In plane-polarized reflected light, kingstonite is a pale slightly brownish grey colour. It is weakly pleochroic and displays a weak bireflectance. It does not possess internal reflections. The anisotropy is weak to moderate in dull greys and browns. Reflectance data and colour values are tabulated. Average results of twenty electron microprobe analyses on four grains give Rh 46.5, Ir 16.4, Pt 11.2, S 25.6, total 99.7 wt.%. The empirical formula is $(Rh_{2.27}Ir_{0.43}Pt_{0.29})_{\Sigma 2.99}S_{4.01}$, based on 7 atoms per formula unit (a.p.f.u.). Kingstonite is monoclinic ($C2/m$) with a = 10.4616(5), b = 10.7527(5), c = 6.2648(3) Å, β = 109.000(5)º, V = 666.34(1) Å^3 (Z = 6). The calculated density is 7.52 g/cm^3 (on the basis of the empirical formula and unit-cell parameters refined from powder data). The seven strongest X-ray powder-diffraction lines [d in Å(I) (hkl)] are: 3.156 (100) (310), 3.081 (100) $(\bar{1}31)$, 2.957 (90) (002), 2.234 (60) (202), 1.941 (50) $(\bar{2}23)$, 1.871 (80) $(\bar{4}41)$ and 1.791 (90) (060, $\bar{1}33$). The structure of kingstonite was solved and refined to Rp = 3.8%. There are four distinct metal sites with Rh occupancies of 0.64−0.89. Two metal sites are regular RhS_6 octahedra that share edges to form a ribbon running parallel to c. The other two metal sites are coordinated by 4 S + 2 Rh and 5 S + 2 Rh and define a puckered Rh_6 ring. The ribbons of regular RhS_6 octahedra alternate with the columns of Rh_6 rings linked by S atoms. S−S bridges also connect the ribbons and columns. As such, the kingstonite structure is essentially that of synthetic Rh_3S_4. Minor differences in the unit-cell parameters, atom coordinates and displacement parameters of kingstonite and synthetic Rh_3S_4 arise from the considerable substitution of Ir for Rh. The mineral name honours Gordon Kingston (formerly of Cardiff University) in recognition of his contributions to platinum group element mineralogy and the geology of their mineral deposits.

Keywords: kingstonite, new mineral, platinum-group mineral, Rh-Ir-Pt sulphide, Yubdo, Ethiopia, electron microprobe data, reflectance data, X-ray diffraction data, crystal-structure analysis.

Introduction

Kingstonite, a rhodium sulphide with an ideal formula of Rh_3S_4, was discovered during re-investigation of a 1.5 cm-sized nugget that also happens to be the type specimen of 'prassoite' [the latter mineral was approved by the Commission on New Minerals and Mineral Names (CNMMN) in March 1971]. 'Prassoite' (assumed to be $Rh_{17}S_{15}$) has entered the mineralogical literature (e.g. Cabri, 1981, pp. 90, 132; Cabri and Laflamme, 1981, p. 155; Augé, 1988; Peckett, 1992; Augé and Maurizot, 1995; http://www.mindat.org/min-7250.html;

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Bowles, 2000; Strunz and Nickel, 2001) although it was never formally published, but was wrongly assumed not to have been approved by the CNMMN by Britvin *et al*. (2001) in their description of the mineral miassite $Rh_{17}S_{15}$ (see Jambor *et al*., 2002) and therefore promises confusion in the literature for years to come. In addition to this, it now seems after re-examination of analyses of the type specimen, that 'prassoite' is actually ferrorhodsite or cuprorhodsite (Cabri, pers. comm.).

Kingstonite is so named in recognition of the contribution to platinum group element mineralogy of Dr Gordon Andrew Kingston (born 1939), formerly a senior lecturer at the Department of Geology, University of Wales, College of Cardiff, Wales, UK. The mineral and mineral name were approved by the CNMMN, International Mineralogical Association (1993-46). Type material is deposited at the Natural History Museum, London, UK as BM 2004, 56. A single-crystal mount and two powder mounts are housed within the Systematic Reference Series of the National Mineral Collection of Canada, Geological Survey of Canada, Ottawa.

Occurrence

The platinum deposits of Yubdo District, Wallaga Province, Ethiopia, occur over an area of ~30 km^2 within an overall prospecting area of >500 km^2. In addition to the primary occurrence of platinum group minerals in fresh and altered ultramafic rocks, placer deposits also occur. These placer deposits are of an alluvial-eluvial nature with the Alfe, Bir Bir and Deressa rivers cutting the Yubdo ultramafic rocks, and lateritic weathering having formed a silica-iron-oxide cap of 'birbirite' above the dunite and pyroxenites of the Yubdo ultramafic complex (Mogessie *et al*., 1999).

The mineralogy of the Yubdo deposit and particularly the platinum group mineralogy has been studied by numerous authors (Cabri *et al*., 1981; Evstigneeva *et al*., 1992; Mogessie *et al*., 1999) and can be summarized as dominated by isoferroplatinum with rather less tetraferroplatinum with inclusions of osmium laths. Other PGM reported are PGE alloys, antimonides, arsenides and sulphides.

Physical properties

Kingstonite occurs as subhedral (tabular, elongate) to anhedral inclusions between 15 and 40 μm in size in the Pt-Fe alloy host (Fig. 1). Most of the inclusions are single phase, although compound intergrowths with either bowieite, ferrorhodsite or cuprorhodsite are not uncommon. These intergrowths are simple and never include more than these two minerals. Additional associated minerals are isoferroplatinum, tetraferroplatinum, a Cu-bearing Pt-Fe alloy (not tulameenite as there is too little copper), osmium, enriched oxide remnants of osmium and laurite.

Kingstonite is opaque with a metallic lustre and black streak (from the powder extracted for X-ray powder diffraction). It is brittle with a subconchoidal fracture and one good cleavage parallel to [001]. Indentation hardness measurements gave VHN_{25} = 895 (range 871−920) kg/mm^2 which roughly equates to a Mohs hardness of 6. The density could not be measured because of the small grain size but, on the basis of the empirical formula and unit-cell parameters refined from powder data, the calculated density is 7.52 g/cm^3.

The difference in physical properties between kingstonite and the host Pt-Fe alloy was exploited to extract a grain for X-ray single-crystal studies, adapting the micro-indenter to loosen the brittle kingstonite grain from its more ductile Pt-Fe alloy matrix.

Optical properties

The sample containing kingstonite was prepared for optical investigation and measurement using standard diamond polishing techniques (e.g. Stanley and Laflamme, 1998).

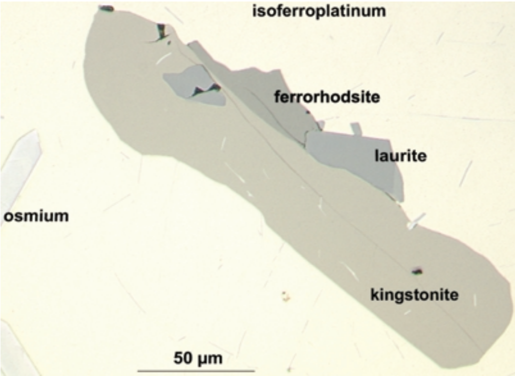

FIG. 1. Reflected light digital image in oil of kingstonite in association with laurite and ferrorhodsite in an isoferroplatinum matrix.

In plane-polarized reflected light (from an unfiltered quartz-halogen lamp at ~3100 K), kingstonite is a pale slightly brownish grey with weak bireflectance and pleochroism. It has no internal reflections. Kingstonite is weakly anisotropic, with weak to moderate rotation tints in dull greys and browns.

Reflectance measurements were made with a Zeiss microspectrophotometer relative to a WTiC reflectance standard (Zeiss 314) using the equipment and procedures reported by Criddle *et al*. (1983). The results are tabulated in Table 1.

TABLE 1. Reflectance data and colour values for kingstonite.

λ (nm)	R_1	R_2	${}^{im}R_1$	${}^{im}R_2$
400	46.7	47.6	32.7	33.8
420	46.7	47.9	32.8	34.0
440	46.9	48.3	32.9	34.3
460	47.1	48.7	33.1	34.5
480	47.4	49.2	33.3	34.9
500	47.7	49.5	33.6	35.3
520	48.0	49.9	33.9	35.7
540	48.4	50.2	34.2	36.0
560	48.7	50.5	34.5	36.3
580	49.0	50.7	34.9	36.4
600	49.3	50.8	35.2	36.6
620	49.5	50.9	35.4	36.6
640	49.7	50.9	35.6	36.7
660	49.8	51.0	35.6	36.7
680	49.9	51.0	35.7	36.6
700	50.0	51.0	35.7	36.6
470	47.2	48.9	33.2	34.7
546	48.4	50.3	34.3	36.1
589	49.1	50.7	35.0	36.5
650	49.8	51.0	35.6	36.7
Colour values for CIE illuminant A				
x	.451	.450	.453	.451
y	.409	.409	.409	.410
Y%	48.8	50.5	34.7	36.2
λ_d	587	583	588	584
P_e%	3.1	3.1	4.3	4.0
Colour values for CIE illuminant C				
x	.314	.314	.316	.315
y	.320	.321	.321	.322
Y%	48.6	50.3	34.5	36.1
λ_d	580	576	581	576
P_e%	2.1	2.2	2.8	2.8

Chemical composition

Chemical analyses were carried out at the Natural History Museum by means of a Cameca SX-50 electron microprobe operated in wavelength-dispersive mode with an accelerating voltage of 20 kV, and a beam current of 20 nA. The counting times on the peak and background varied, according to the count rate of the particular element, from 10 to 100 s in order to give detection limits of ~0.01 to 0.02 wt.%. ZAF correction was applied through a PAP correction procedure (Pouchou and Pichoir, 1985). The following standards were used: troilite (S) and pure elements (Rh, Ir,Pt). Lines measured were Pt *Lα*, Rh *Lα*, Ir *Lα* and S *Kα*. Interferences between these elements on peak and/or background were taken into account. Results are given in Table 2. The empirical formula for kingstonite calculated from the arithmetic mean in Table 2 is $(Rh_{2.27}Ir_{0.43}Pt_{0.29})_{\Sigma 2.99}S_{4.01}$ based on 7 atoms per formula unit. The simplified formula is $(Rh,Ir,Pt)_3S_4$, which requires: (with Rh:Ir:Pt = 2.28:0.43:0.29) Rh 46.73, Ir 16.46, Pt 11.27, S 25.54, total 100.00 wt.%.

X-ray diffraction and structure studies

X-ray powder diffraction

Fully-indexed 114.6 mm Debye-Scherrer camera X-ray powder data are presented in Table 3. The data are unique and do not bear resemblance to any other PGE-bearing compound or to any inorganic compound listed in the PDF up to and including Set 52. The unit-cell parameters refined from 23 reflections in the powder-diffraction pattern between 3.500 and 1.631 Å for which unambiguous indexing was possible are: *a* = 10.448(9), *b* = 10.749(7), *c* = 6.257(5) Å, β =

TABLE 2. Electron microprobe data for kingstonite.

	Wt.%	Ranges	Formula units
Rh	46.5	(46.5–46.9)	2.27
Pt	11.2	(10.8–11.3)	0.29
Ir	16.4	(15.6–16.9)	0.43
S	25.6	(25.2–25.7)	4.01
Total	99.7		7.00

Average results of twenty electron microprobe analyses on four grains.

108.98(6)° $V = 664.5(5)$ Å^3. These values agree well with those determined by single-crystal X-ray diffraction (see below).

Single-crystal X-ray diffraction

A single fragment, dug out of a polished section and measuring 35 × 10 × 10 μm, was mounted and studied by single-crystal precession methods employing Zr-filtered Mo radiation. The fragment was initially orientated with 101* parallel to the dial axis, then reorientated with c^* parallel to the dial axis. The reciprocal lattice levels collected were: $h0l \rightarrow h4l$, $101^*\wedge b^*$, $0kl \rightarrow 3kl$ and $c^*\wedge 110^*$ and consistent with space groups *C*2/*m* (#12), *C*2 (#5) or *Cm* (#8).

For the structure determination study, the same crystal was mounted on a Nonius CAD4 four-circle diffractometer equipped with graphite-monochromated Mo-$K\alpha$ radiation. The orientation matrix was refined from the setting angles of 24 reflections using four-position centering ($\pm\theta,\pm\chi$) and gave unit-cell parameters: $a = 10.4616(5)$, $b = 10.7527(5)$, $c = 6.2648(3)$ Å, $\beta = 109.000(5)$° $V = 666.34(1)$ Å^3 ($Z = 6$). A whole sphere of reflections to 65°2θ was collected using peak profile analysis. Data reduction (including an absorption correction), structure solution by Direct Methods and structure refinement were performed using the program NRCVAX of Gabe *et al*. (1989). A total of 840 out of 1253 unique reflections were "observed" at the 2.5σ level. Reflection merging indicated space group *C*2/*m* to be correct. The structure of kingstonite was solved and refined to $R_p = 3.78\%$ in space group *C*2/*m*. Observed and calculated structure factors are given in Table 4 (deposited with the Editor and deposited in the Mineralogical Society website: www.minersoc.org/pages/e_journals/dep_mat.html). Atom coordinates and

TABLE 3. X-ray powder-diffraction data for kingstonite.

I_{est}	$d_{meas.}$ (Å)	$d_{calc.}$ (Å)	$h\ k\ l$
20	5.23	5.25	$\bar{1}11$
20	4.97	4.94	200
5	4.60	4.60	$\bar{2}01$
5	4.13	4.13	111
*20	3.500	3.494	$\bar{2}21$
*10	3.305	3.301	201
*100	3.156	3.149	310
*100	3.081	3.078	$\bar{1}31$
*90	2.957	2.958	002
40	2.805	2.812	221
		2.797	131
3	2.673	2.687	040
		2.623	$\bar{2}22$
40	2.601	2.600	$\bar{4}01$
		2.596	$\bar{3}12$
*5	2.536	2.537	112
*20	2.471	2.470	400
*30	2.449	2.447	041
30	2.353	2.356	$\bar{1}32$
		2.340	$\bar{4}21$
*10	2.318	2.320	$\bar{2}41$
*60	2.234	2.237	202
30	2.081	2.084	241
		2.079	$\bar{2}03$
5	2.056	2.054	401
		2.051	$\bar{5}11$
*10	2.029	2.030	$\bar{1}13$
*20	2.000	2.003	$\bar{2}42$
3	1.971	1.970	$\bar{3}13$
*50	1.941	1.939	$\bar{2}23$
*40	1.918	1.919	421
*40	1.885	1.884	312
*80	1.871	1.868	$\bar{4}41$
3	1.820	1.819	440
		1.814	$\bar{3}51$
90	1.791	1.791	060
		1.791	$\bar{1}33$
10	1.769	1.772	$\bar{1}52$
		1.762	$\bar{4}23$
*10	1.745	1.747	$\bar{4}42$
*30	1.722	1.723	$\bar{5}32$
*30	1.692	1.692	$\bar{6}02$
*20	1.678	1.676	$\bar{3}52$
*20	1.656	1.656	203
*10	1.647	1.647	600
*40	1.631	1.632	441
5	1.546	1.546	531
		1.533	$\bar{6}03$
70	1.532	1.532	062
		1.532	$\bar{4}43$

114.6 mm Debye-Scherrer powder camera, Cu radiation, Ni filter (λ Cu-$K\alpha$ = 1.54178 Å).
Intensities estimated visually. Not corrected for shrinkage and no internal standard. Indexed on 23 lines (marked with *) with $a = 10.448$, $b = 10.749$, $c = 6.257$ Å, ß = 108.98°.

Table 5. Atom coordinates and displacement parameters (Å^2) of kingstonite.

Atom	x	y	z	occ*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}	U_{eq}
Rh1	0.36578(10)	0.14279(12)	0.95211(16)	0.75(2)	0.0055(5)	0.0064(4)	0.0066(4)	−0.0004(3)	0.0024(3)	−0.0005(3)	0.0061(5)
Rh2	0.64989(16)	0	0.44360(27)	0.90(2)	0.0049(7)	0.0061(7)	0.0070(8)	–	0.0021(5)	–	0.0059(8)
Rh3	0	0	0	0.64(3)	0.0039(9)	0.0057(8)	0.0042(8)	–	0.0015(6)	–	0.0045(9)
Rh4	0	0.16031(14)	½	0.69(3)	0.0048(6)	0.0047(6)	0.0050(6)	–	0.0013(4)	–	0.0048(6)
S1	0.41625(56)	0	0.23612(90)	1	0.0055(23)	0.0084(22)	0.0051(21)	–	−0.0006(17)	–	0.0062(23)
S2	0.87342(37)	0.15705(36)	0.11257(61)	1	0.0048(15)	0.0062(16)	0.0075(15)	0.0018(12)	0.0019(11)	0.0006(12)	0.0061(16)
S3	0.11639(62)	0	0.39114(94)	1	0.0119(26)	0.0085(23)	0.0056(22)	–	0.0046(18)	–	0.0083(24)
S4	0.14923(40)	0.28955(37)	0.40093(62)	1	0.0071(16)	0.0078(16)	0.0056(15)	−0.0033(12)	0.0023(12)	0.0002(12)	0.0068(16)

* occ = fractional Rh content of site Rh; remainder is Ir + Pt.

Table 6. Bond distances (Å) and angles (º) of Rh polyhedra in kingstonite.

Rh1		Rh2		Rh3		Rh4	
S(1)	2.278(4)	S(1)	2.365(6)	S(2) × 4	2.390(4)	S(2) × 2	2.357(4)
S(2)	2.366(4)	S(1)	2.326(6)	S(3) × 2	2.355(6)	S(3) × 2	2.320(4)
S(2)	2.406(4)	S(3)	2.324(6)			S(4) × 2	2.285(4)
S(4)	2.285(4)	S(4) × 2	2.278(4)				
Rh(1)	2.674(2)	Rh(1) × 2	2.875(2)				
Rh(2)	2.875(2)						
S(1)–S(2)	97.95(17)	S(1)–S(1)	85.96(10)	S(2)–S(2)	89.93(13)	S(2)–S(3)	81.95(17)
	108.31(13)	S(1)–S(3)	100.43(20)		90.07(13)		96.79(17)
S(1)–S(4)	153.65(16)		173.61(21)	S(2)–S(3)	80.87(13)	S(2)–S(4)	86.26(13)
S(2)–S(2)	84.43(13)	S(1)–S(4)	88.42(11)		99.13(13)		94.76(13)
S(2)–S(4)	94.40(14)		96.36(10)			S(3)–S(3)	84.93(15)
	95.96(14)	S(3)–S(4)	90.85(11)			S(3)–S(4)	84.48(15)
S(1)–Rh(1)	82.26(14)	S(4)–S(4)	166.64(16)				168.46(16)
S(2)–Rh(1)	90.97(10)					S(4)–S(4)	106.42(15)
	175.23(11)						
S(4)–Rh(1)	87.33(11)						

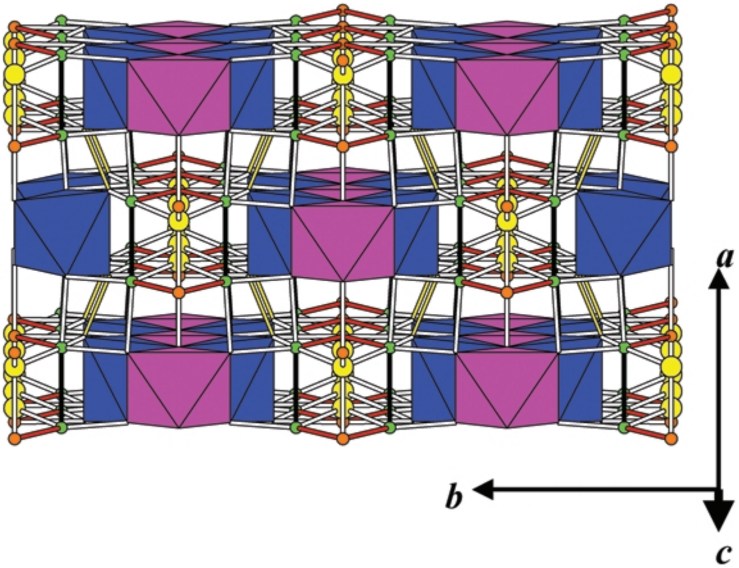

FIG. 2. Structure of kingstonite viewed nearly parallel to the c axis. Ribbons of edge-sharing Rh3 (blue) and Rh4 (magenta) RhS_6 octahedra run parallel to the *c* axis in checker-board formation. These ribbons alternate with chains of Rh1 and Rh2 Rh_6 rings. Rh1 atoms are shown in green and Rh2 atoms in orange. The shortest Rh1–Rh1 and Rh1–Rh2 distances are shown in black and red, respectively. S–S bonds linking columns and ribbons are shown in yellow.

displacement parameters are given in Table 5 and bond distances are shown in Table 6.

The kingstonite structure

The structure of kingstonite, shown in Figs 2 and 3, is essentially that of synthetic Rh_3S_4 (Beck and Hilbert, 2000), but showing minor differences in cell parameters, atom coordinates and displacement parameters due to the presence of Ir and Pt. However, kingstonite has no obvious structural relationship to any naturally occurring species. There are four unique metal sites and four unique sulphur sites, but the site multiplicities are such that the unit cell contains 36 metal atoms and 48 sulphur atoms, thus giving a stoichiometric ratio of $(Rh,Ir,Pt)_3S_4$. The metal composition was simplified for refinement purposes as Rh/Ir, with Pt being included with Ir (Pt and Ir are adjacent in the Periodic Table). The Rh population parameter was refined for each site, with its difference from unity being treated as Ir. The four distinct metal sites have Rh population parameters of 0.64(3), 0.69(2), 0.90(2) and 0.75(2), which gives a ratio for Rh/Ir refined by least-squares of 2.27(3)/0.73 for the formula; this is in excellent agreement with the microprobe values of 2.28/0.72 if Pt is not differentiated from Ir. The metal sites fall into two categories: metal atoms Rh3 and Rh4 have regular octahedral coordination by six S atoms at

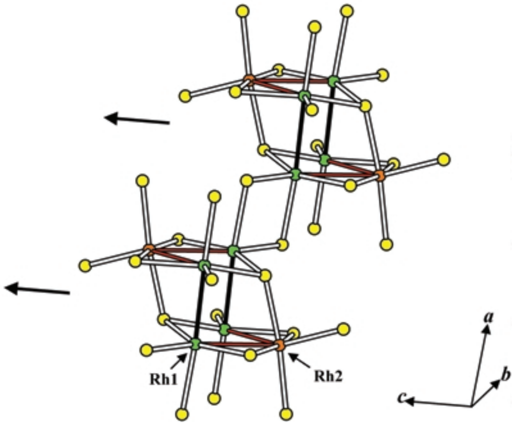

FIG. 3. Clinographic view of the chains of Rh1 and Rh2 polyhedra in kingstonite (Rh3-Rh4 octahedral ribbons omitted for clarity). Parts of two parallel chains are shown and an Rh_6 ring is evident in both columns. Rh1–Rh1 bonds are shown in black, Rh1–Rh2 bonds are shown in red as in Fig. 2. Arrows indicate direction of chains.

distances between 2.320(4) and 2.390(4) Å; metal atoms Rh1 and Rh2 are combined into a cage in the form of a six-membered Rh_6 puckered ring −Rh1−Rh1−Rh2−Rh1−Rh1−Rh2− lying astride a mirror plane, having Rh1−Rh1 = 2.674(2) Å, Rh1−Rh2 = 2.875(2) Å. This metal Rh_6 ring is surrounded by S atoms so that Rh1 is coordinated to Rh1, Rh2 and four S atoms, and Rh2 is coordinated to two Rh1 atoms and five S atoms. Three of the S atoms (S1, S2, S3) are coordinated approximately tetrahedrally by metal atoms. The S4 atom is coordinated to three metal atoms [2.278(4), 2.285(4), 2.320(4) Å] and another S4 atom at a S−S distance of 2.237(8) Å.

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