Preparation and Structure of μ-Oxalato-bis{μ-oxo-μ-sulfido-bis-[(oxalato)oxomolybdate(V)]} Complex, $K_6[\{Mo_2O_3S(ox)_2\}_2(ox)]\cdot 10H_2O$

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The μ-oxo-μ-sulfido-(oxalato)molybdate(V) complex has been prepared and characterized by X-ray analysis. The potassium salt crystallizes in monoclinic system, space group C2/c, with cell dimensions $a=13.640(3)$, $b=20.296(3)$, $c=15.213(4)$ Å, $\beta=106.42(2)°$, and $Z=4$. The structure was solved by the heavy-atom technique and refined by the block-diagonal least-squares method to $R=0.048$ for 3552 independent reflections. The complex anion is tetrameric, two dimeric $Mo_2O_3S(ox)_2$ moieties being bridged by one oxalato ligand. Both the molybdenum atoms in the dimer are bridged by one sulfur and one oxygen atoms. Each molybdenum atom has distorted octahedral environment; two oxygen atoms of a bidentate oxalato ligand, bridging sulfur and oxygen atoms in the equatorial plane, a terminal oxygen and one of the bridging quadridentate oxalato ligand oxygens. The complex can be denoted as $K_6[\{Mo_2O_3S(ox)_2\}_2(ox)]\cdot 10H_2O$ and has an approximate C_{2h} symmetry, with a crystallographic center of symmetry. The Mo–O (quadridentate bridging oxalato) bonds (2.304(4), 2.302(4) Å) are evidently longer than the Mo–O (bidentate oxalato) bonds (2.112(4), 2.102(4), 2.093(4), 2.120(4) Å). The influence of the bridging oxalato ligand on the geometry of Mo_2O_3S core will be discussed.

The presence of sulfur in several molybdenum containing enzymes led us to the chemistry of μ-sulfur molybdenum compounds.[1]

The chemistry of di-μ-oxo and di-μ-sulfido molybdenum(V) compounds has been widely developed;[1] however, the reports of μ-oxo-μ-sulfido molybdenum(V) compounds are yet relatively rare.[2,3] Cotton and Morehouse[4] reported the structure of di-μ-oxo oxalato complex of $[Mo_2O_4(ox)_2(H_2O)_2]^{2-}$, and that of the di-μ-sulfido oxalato complex of $[Mo_2O_2S_2(ox)_2(H_2O)_2]^{2-}$ was determined by McDonald.[5]

Now we report here the preparation and X-ray structure of the potassium salt of μ-oxalato-(*O*,*O*′,*O*″,*O*‴)-bis{μ-oxo-μ-sulfido-bis[(oxalato)oxomolybdate(V)]}.

Experimental

Preparation of a Solution of μ-Oxo-μ-sulfido-bis[triaquaoxomolybdenum(V)], $[Mo_2O_3S(H_2O)_6]^{2+}$. The aqua complex was prepared by dissolving the corresponding cysteinato complex, $Na_2[Mo_2O_3S(cys)_2]\cdot 5H_2O$[2] in 1 M (1 M=1 mol dm^{-3}) perchloric acid followed by the Sephadex G-10 column separation, which gave three bands. The first and third effluents were proved to involve $[Mo_2O_4(H_2O)_6]^{2+}$ and $[Mo_2O_2S_2(H_2O)_6]^{2+}$, respectively, as evidenced by the UV and visible spectroscopy.

The second effluent was further purified by the use of cation exchange resin Dowex 50W-X2, on which the complex was sorbed and washed with 0.1 M $HClO_4$ and eluted with 2 M $HClO_4$. The analysis of the effluent (Mo: S=2.03: 1) suggests that this is $[Mo_2O_3S(H_2O)_6]^{2+}$.[6a]

Preparation of Potassium μ-oxalato-(O, O′, O″, O‴)-bis{μ-oxo-{μ-sulfido-bis[(oxalato)oxomolybdate(V)]} Decahydrate, $K_6[\{Mo_2O_3S(ox)_2\}_2(ox)]\cdot 10H_2O$. The preparative method of the sample is similar to that of the di-μ-sulfido-bis[aqua(oxalato)-oxomolybdate(V)];[6a] the addition of oxalic acid (H_2ox/Mo =1.2) to *e.g.* 250 ml of the $Mo_2O_3S^{2+}$ aqua dimer solution (0.0264 M in 1 M $HClO_4$)[6b] and neutralization to pH *ca.* 3 by the addition of *ca.* 10 M KOH solution. The solution was cooled, filtered, and rotary-evaporated to the volume of *ca.* 70 ml. After one gram of KCl was added, the solution was cooled for some time in an ice bath and then filtered. The filtrate was made turbid by the addition of ethanol and kept in a refrigerator overnight. The resulting orange crystals were filtered and washed successively with ethanol and diethyl ether. Yield 2.6 g. Found: C, 8.67; H, 1.20%. Calcd for $C_{10}H_{20}K_6Mo_4O_{36}S_2$: C, 8.56; H, 1.44%.

Data Collection. The crystal employed for data collection was approximately 0.20×0.15×0.15 mm in dimension with well defined faces. Crystal data: $C_{10}H_{20}K_6Mo_4O_{36}S_2$, $M=1398.72$, monoclinic, $a=13.640(3)$, $b=20.296(3)$, $c=15.213(4)$ Å, $\beta=106.42(2)°$, $V=4039.8$ Å^3, $D_m=2.29$ g cm^{-3}, $Z=4$, $D_c=2.30$ g cm^{-3}, space group C2/c, λ(Mo *Kα*)=0.7107 Å, and μ(Mo *Kα*)=20.1 cm^{-1}. Intensity data ($2\theta\leqslant 55°$) were collected on a Phillips 1100 four-circle diffractometer using graphite-monochromatized Mo *Kα* radiation with ω-scan technique. The scan range was 1.20°, and the scan speed, 0.033° s^{-1}; the background was counted for half of the scan time at each end of the scan. The three standard reflections 600, 080, and 008, monitored every 360 min showed no appreciable decay. A total of 4793 reflections were collected, of which 3552 with $F_o^2\geqslant 3\sigma(F_o^2)$ were used for the structure analysis. No correction was made for absorption.

Structure Determination and Refinement. The two molybdenum atoms were located from a Patterson synthesis and the remaining non-hydrogen atom positions were obtained from successive difference-Fourier calculations. The electron density of one potassium ion indicated disorder. It was possible, however, to resolve the ion into two with an ocucpancy factor of 0.5 (K(1) and K(2)). The block-diagonal least-squares refinement with anisotropic temperature factors for all non-hydrogen atoms gave $R=0.048$ and $R'=[\sum w\Delta F^2/\sum wF_o^2]^{1/2}=0.060$. The function minimized was $\sum w(F_o-F_c)^2$, where $w=\sigma^{-2}$ (F_o) was used. All the parameter shifts were less than 0.3 σ except for O(1) (0.8 σ) and O(4) (0.4 σ). A final difference Fourier map revealed no peaks greater than 0.9 e Å^{-3}. The positions of hydrogen atoms of water of crystallization could not be found unequivocally. The atomic scattering factors, with correction for anomalous dispersion of Mo^0, K^{+1}, and S were taken from Ref. 7. The F_o-F_c table and the anisotropic thermal parameters are preserved by the Chemical Society of Japan (Document No 8251). Most of the computation were carried out by the Melcom Cosmo 700 III computer at Okayama University of Science. The programs used included a local version of the UNICS.[8]

Table 1. Atomic coordinates (×10⁵ for Mo and ×10⁴ for others) and equivalent isotropic temperature factors[a] with their standard deviations

Atom	x	y	z	B_{eq}/Å²	Atom	x	y	z	B_{eq}/Å²
Mo(1)	932(4)	18637(2)	−2133(3)	1.74	O(22)	−552(3)	4013(2)	−694(3)	2.4
Mo(2)	1514(4)	31692(2)	−70(3)	1.74	O(23)	1377(3)	3365(2)	99(4)	3.2
K(1)[b]	0	409(3)	2500	9.9	O(24)	−1527(3)	3057(2)	−3(3)	2.1
K(2)[b]	0	2721(2)	2500	4.8	O(25)	−1347(4)	4942(2)	−532(3)	3.2
K(3)	2383(1)	4373(1)	−498(1)	3.41	O(26)	−909(4)	4649(3)	1318(3)	3.9
K(4)	1726(1)	4504(1)	2222(1)	3.88	O(1)	1718(10)	3964(5)	−2372(6)	13.8
Sb	−384(1)	2622(1)	−1406(1)	2.47	O(2)	−2013(5)	3202(3)	2131(4)	5.3
Ob	292(3)	2434(2)	828(3)	2.3	O(3)	1431(6)	915(4)	−1910(5)	7.8
O(11)	−123(3)	1084(2)	633(3)	2.4	O(4)	−1375(8)	1477(5)	1979(7)	11.4
O(12)	−677(3)	1151(2)	−1160(3)	2.1	O(5)	2233(7)	2623(4)	−1556(5)	7.9
O(13)	1303(3)	1663(2)	−187(4)	3.1	C(11)	−672(5)	600(3)	230(4)	2.4
O(14)	−1583(3)	1962(2)	−194(3)	2.1	C(12)	−1022(4)	647(3)	−831(4)	2.2
O(15)	−1579(4)	229(2)	−1290(3)	3.4	C(21)	−609(4)	4289(3)	811(4)	2.2
O(16)	−924(4)	126(2)	604(3)	3.3	C(22)	−875(4)	4439(3)	−223(4)	2.1
O(21)	−36(3)	3774(2)	1070(3)	2.3	C(1)	−1952(4)	2505(3)	−54(4)	1.7

a) W. C. Hamilton, *Acta Crystallogr.*, **12**, 609 (1959). b) Site occupation factor 0.5.

Table 2. Interatomic distances and bond angles with estimated standard deviations in parentheses[a]

Bond distance	l/Å	Bond distance	l/Å
Mo(1)–Sb	2.325(2)	Mo(2)–Sb	2.326(2)
Mo(1)–Ob	1.918(5)	Mo(2)–Ob	1.934(5)
Mo(1)–O(11)	2.112(4)	Mo(2)–O(21)	2.120(4)
Mo(1)–O(12)	2.102(4)	Mo(2)–O(22)	2.093(4)
Mo(1)–O(13)	1.689(5)	Mo(2)–O(23)	1.681(5)
Mo(1)–O(14)	2.304(4)	Mo(2)–O(24)	2.302(4)
Mo(1)–Mo(2)	2.667(1)		
C(11)–O(11)	1.282(8)	C(21)–O(21)	1.279(7)
C(11)–O(16)	1.215(8)	C(21)–O(26)	1.213(8)
C(11)–C(12)	1.553(9)	C(21)–C(22)	1.542(9)
C(12)–O(12)	1.284(8)	C(22)–O(22)	1.279(7)
C(12)–O(15)	1.219(8)	C(22)–O(25)	1.227(8)
C(1)–O(14)	1.254(7)	C(1)–O(24)	1.254(7)
C(1)–C(1′)	1.548(16)		
Short contact	l/Å	Short contact	l/Å
K(1)···O(16)	2.861(7)	K(2)···O(2)	2.817(7)
K(1)···O(16)^{III}	2.861(7)	K(2)···O(2)^{III}	2.817(7)
K(1)···O(4)	2.828(12)	K(2)···Ob	2.747(5)
K(1)···O(4)^{III}	2.828(12)	K(2)···Ob^{III}	2.747(5)
K(3)···O(13)^{V}	2.769(6)	K(4)···O(15)^{VI}	2.793(5)
K(3)···O(15)^{IV}	2.728(5)	K(4)···O(25)^{I}	2.719(5)
K(3)···O(16)^{IV}	2.880(5)	K(4)···O(26)^{III}	2.769(6)
K(3)···O(23)	2.755(6)	K(4)···O(2)^{III}	2.807(7)
K(3)···O(25)^{I}	2.763(5)	K(4)···O(3)^{V}	2.818(9)
K(3)···O(26)^{I}	2.849(6)		
K(3)···O(1)	2.859(13)		
O(14)···O(24′)	2.679(12)	O(1)···O(5)	2.992(16)
O(1)···O(22)^{II}	2.924(14)		

a) Roman numeral superscripts refer to atoms in the positions:
I $-x, 1-y, -z$; II $-x, y, -1/2-z$; III $-x, y, 1/2-z$; IV $1/2+x, 1/2+y, z$;
V $1/2-x, 1/2-y, -z$; VI $1/2+x, 1/2-y, 1/2+z$.

Bond angle	φ/°	Bond angle	φ/°
O(13)–Mo(1)–Mo(2)	103.9(2)	O(23)–Mo(2)–Mo(1)	104.0(2)
O(13)–Mo(1)–Ob	102.5(2)	O(23)–Mo(2)–Ob	102.1(2)
O(13)–Mo(1)–Sb	102.3(2)	O(23)–Mo(2)–Sb	103.1(2)
O(13)–Mo(1)–O(11)	96.3(2)	O(23)–Mo(2)–O(21)	97.0(2)
O(13)–Mo(1)–O(12)	98.4(2)	O(23)–Mo(2)–O(22)	99.0(2)

TABLE 2. (Continued)

Bond angle	ϕ/°	Bond angle	ϕ/°
O(13)–Mo(1)–O(14)	170.8(2)	O(23)–Mo(2)–O(24)	170.3(2)
O(14)–Mo(1)–Mo(2)	84.8(1)	O(24)–Mo(2)–Mo(1)	84.5(1)
O(14)–Mo(1)–OB	81.2(2)	O(24)–Mo(2)–OB	80.6(2)
O(14)–Mo(1)–SB	85.1(1)	O(24)–Mo(2)–SB	85.5(1)
O(14)–Mo(1)–O(11)	75.3(2)	O(24)–Mo(2)–O(21)	73.7(2)
O(14)–Mo(1)–O(12)	76.3(2)	O(24)–Mo(2)–O(22)	76.5(2)
OB–Mo(1)–Mo(2)	46.4(1)	OB–Mo(2)–Mo(1)	45.9(1)
OB–Mo(1)–SB	101.0(1)	OB–Mo(2)–SB	100.5(1)
OB–Mo(1)–O(11)	87.7(2)	OB–Mo(2)–O(21)	87.2(2)
OB–Mo(1)–O(12)	155.3(2)	OB–Mo(2)–O(22)	155.0(2)
SB–Mo(1)–Mo(2)	55.1(1)	SB–Mo(2)–Mo(1)	55.0(1)
SB–Mo(1)–O(11)	157.1(1)	SB–Mo(2)–O(21)	156.4(1)
SB–Mo(1)–O(12)	87.2(1)	SB–Mo(2)–O(22)	87.7(1)
O(11)–Mo(1)–Mo(2)	132.6(1)	O(21)–Mo(2)–Mo(1)	131.4(1)
O(11)–Mo(1)–O(12)	76.9(2)	O(21)–Mo(2)–O(22)	76.9(2)
O(12)–Mo(1)–Mo(2)	139.2(1)	O(22)–Mo(2)–Mo(1)	139.5(1)
Mo(1)–OB–Mo(2)	87.7(2)	Mo(1)–SB–Mo(2)	70.0(1)
Mo(1)–O(11)–C(11)	116.5(4)	Mo(2)–O(21)–C(21)	115.3(4)
Mo(1)–O(12)–C(12)	116.7(4)	Mo(2)–O(22)–C(22)	117.0(4)
O(11)–C(11)–C(12)	114.6(5)	O(21)–C(21)–C(22)	114.5(5)
O(11)–C(11)–O(16)	126.0(6)	O(21)–C(21)–O(26)	125.2(6)
O(16)–C(11)–C(12)	119.5(6)	O(26)–C(21)–C(22)	120.3(6)
O(12)–C(12)–C(11)	114.6(5)	O(22)–C(22)–C(21)	114.9(5)
O(12)–C(12)–O(15)	124.7(6)	O(22)–C(22)–O(25)	125.0(6)
O(15)–C(12)–C(11)	120.7(6)	O(25)–C(22)–C(21)	120.1(6)
Mo(1)–O(14)–C(1)	121.8(4)	Mo(2)–O(24)–C(1)	122.2(4)
O(13)–Mol(1)–O(14)	170.8(2)	O(23)–Mo(2)–O(24)	170.3(2)
O(14)–C(1)–C(1′)	116.9(5)	O(24)–C(1)–C(1′)	116.7(5)
O(1)–C(1)–O(24)	126.4(6)		

Results and Discussion

Peak positions of UV-visible spectra of $[Mo_2O_3S(H_2O)_6]^{2+}$ and $[\{Mo_2O_3S(ox)_2\}_2(ox)]^{6-}$ are as follows: $[Mo_2O_3S(H_2O)_6]^{2+}$ (per dimer),[9] 220_{sh}(ε=5600), 278 (3950), 314(3250), 450_{sh}(57) nm; $[\{Mo_2O_3S(ox)_2\}_2(ox)]^{6-}$ (per tetramer), 280(ε=13600), 320_{sh}(10400), 470_{sh}(220) nm. Addition of cysteine or $Na_2H_2edta\cdot 2H_2O$ to the $[Mo_2O_3S(H_2O)_6]^{2+}$ solution and neutralization to pH *ca.* 6 gave $[Mo_2O_3S(cys)_2]^{2-}$ and $[Mo_2O_3S(edta)]^{2-}$, respectively, and this can confirm the existence of μ-oxo-μ-sulfido molybdenum(V) dimer. The $[Mo_2O_3S(H_2O)_6]^{2+}$ aqua dimer is not so stable as the $[Mo_2O_2S_2(H_2O)_6]^{2+}$ is; the solution of the former gives yellow precipitates in a week even in a refrigerator.

It is interesting that the order of elution from Sephadex G-10 column depends on the number of the sulfur atoms in the bridge, possibly being due to difference in molecular size which will give three bands in the separation column (see Experimental).

The atomic coordinates, and bond lengths and bond angles are listed in Tables 1 and 2, respectively.

The complex anion contains two $Mo_2O_3S(ox)_2$ moieties bridged by an oxalato ligand as shown in Fig. 1. The midpoint of C–C bond of the bridging oxalato ligand lies on the crystallographic center of symmetry at (−1/4, 1/4, 0) and the complex anion has an approximate mirror plane which passes through six atoms, *viz*, two μ-S's, two μ-O's, and two carbon atoms of the bridging oxalato ligand; thus the tetramer has an approximate C_{2h} symmetry.

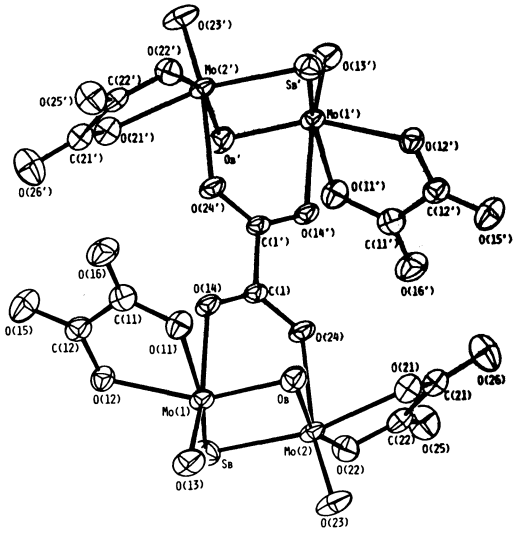
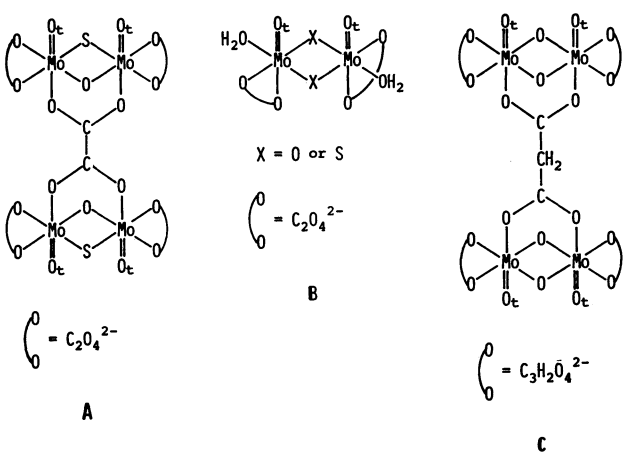


Fig. 1. Molecular structure of $[\{MO_2O_3S(ox)_2\}_2(ox)]^{6-}$ showing 50% probability ellipsoids of thermal vibration. The primed atoms are centrosymmetric of the unprimed ones.

The mode of ligation of the oxalato ligand in this complex (**A**) is different from those of the di-μ-oxo and di-μ-sulfido oxalato complex anions (**B**), but rather similar to that of the di-μ-oxo malonato complex, $K_6[\{Mo_2O_4(mal)_2\}_2(ox)]\cdot 4H_2O$ (**C**), where two types of malonato ligands are existent; one is bidentate and the other is quadridentate.[10] The reason why the oxalato ligand has such a coordination mode in the



μ-oxo-μ-sulfido complex is not clear at present; the use of less amount of oxalic acid ($H_2ox/Mo=1.0$, see Experimental) also gives the same type of complex, though the yield is low.

Each molybdenum atom in the tetramer has distorted octahedral environment, disregarding any Mo–Mo bonding included in the coordination sphere: two oxygen atoms of a bidentate oxalato ligand and bridging sulfur and oxygen atoms in the equatorial plane, a terminal oxygen and one of the bridging quadridentate oxalato ligand oxygens. In the $Mo_2O_3S(ox)_2$ moiety, two terminal oxygen atoms are *cis* to each other.

The present Mo–Mo bond distance 2.667(1) Å is close to those of $[Mo_2O_3S(p_rdtc)_2]$ (2.673(3) Å)[11] and $[Mo_2O_3S(pdta)]^{2-}$ (2.656(1) Å)[12] and also to the mean value (2.681 Å) of the di-μ-oxo and di-μ-sulfido oxalato complexes.

While no trans influence due to the multiply-bonded terminal oxo ligand was observed for both the di-μ-oxo and di-μ-sulfido exalato complexes, evident lengthening of the Mo–O bond trans to the terminal oxo ligand is observed as seen in Table 3. It seems that, if one oxalato ligand occupies both the trans and cis positions to the multiply-bonded terminal oxygen as in **B**, no trans influence is observed, whereas, in case the trans and cis positions are occupied by the two respective oxalato ligands as in **A**, trans influence can be found.

The angle O_t–Mo–O (trans to O_t, 170.8(2) and 170.3(2)°) and the dihedral angle (168.5°) defined by O_B, Mo(1), S_B and O_B, Mo(2), S_B planes are considerably larger than those of di-μ-oxo and di-μ-sulfido oxalato complexes (Table 3). These enlargement may be caused by the coordination of the bridging oxalato anion as a quadridentate ligand.[10] This fact indicates that the present way of occupation of the sixth coordination position trans to O_t will give a remarkable influence to the coordination geometry, the existence of trans influence of O_t being overcome.

There is slight trans influence of the bridging sulfur; bond lengths of Mo–O (trans to μ-S) are 2.112(4) and 2.120(4) Å, and those of Mo–O (trans to μ-O) are 2.102(4) and 2.093(4) Å.

Each of the five membered rings defined by Mo(1), O(11), O(12), C(11), and C(12), and, Mo(2), O(21),

Table 3. Relevant structural parameters for oxalato and malonato molybdenum(V) complexes

	$K_6[\{Mo_2O_3S(ox)_2\}_2(ox)]\cdot 10H_2O$	$Ba[Mo_2O_4(ox)_2(H_2O)_2]\cdot 3H_2O$	$Cs_2[Mo_2O_2S_2(ox)_2(H_2O)_2]\cdot 2H_2O$	$K_6[\{Mo_2O_4(mal)_2\}_2(mal)]\cdot 4H_2O$[h]
Mo–Mo l/Å	2.667(1)	2.541	2.882(1)	2.555
Mo–O_t l/Å	1.689(5)	1.70(3)	1.702(6)	1.684
	1.681(5)		1.692(6)	
Mo–O_B l/Å	1.918(5)	1.93(3)		1.934
	1.934(5)	1.88(3)		
Mo–S_B l/Å	2.325(2)		2.310(2)	
	2.326(2)		2.318(2)	
			2.308(2)	
			2.320(2)	
Mo–O l/Å	2.112(4)[a,b]	2.14(4)[a,c]	2.159(5)[a,b]	2.081[i]
	2.102(4)[a,c]		2.166(5)[a,b]	
	2.120(4)[a,b]			
	2.093(4)[a,c]			
Mo–O l/Å[d]	2.304(4)[e]	2.11(3)[a]	2.155(5)[a]	2.321[j]
	2.302(4)[e]		2.164(6)[a]	
Mo–O l/Å[f]		2.22(4)	2.229(6)	
			2.196(7)	
Mo–O_B–Mo ϕ/°	87.7(2)	83.5(12)		82.7
Mo–S_B–Mo ϕ/°	70.0(1)		75.4(1)	
			75.0(1)	
O_t–Mo–O ϕ/°[d]	170.8(2)	150.9(15)	158.1[m]	171
	170.3(2)		158.6[m]	
Dihedral angle ϕ/°[g]	168.5	150.6[k]	155.6	161
Ref.	This work	4	5	10

a) Non-bridging oxalato ligand. b) Trans to S_B. c) Trans to O_B. d) Trans to O_t. e) Bridging oxalato ligand. f) Coordinated water. g) Formed about X···Y in the Mo_2XY ring. h) Average value. i) Non-bridging malonato ligand. j) Bridging malonato ligand. k) Calculated using the atomic parameters from Ref. 4. m) Calculated using the atomic parameters from Ref. 5.

TABLE 4. DEVIATIONS (l/Å) OF ATOMS FROM MEAN PLANES

(A) Plane through Mo(1), O(11), O(12), C(11), and C(12):
Mo(1) −0.045, O(11) 0.047, O(12) 0.060, C(11) −0.015, C(12) −0.040
(B) Plane through Mo(2), O(21), O(22), C(21), and C(22):
Mo(2) 0.067, O(21) −0.075, O(22) −0.056, C(21) 0.055, C(22) 0.013
(C) Plane through SB, OB, O(11), and O(12):
Mo(1) 0.371, SB 0.005, OB −0.006, O(11) 0.007, O(12) −0.007
(D) Plane through SB, OB, O(21), and O(22):
Mo(2) −0.387, SB 0.005, OB −0.005, O(21) 0.006, O(22) −0.006
Equation of planes (Orthogonalized coordinates)
(A) $0.8937x-0.4481y-0.0226z+1.4469=0$
(B) $-0.8711x-0.4774y-0.1152z+3\ 3196=0$
(C) $0.9839x-0.1785y-0.0023z+0\ 8313=0$
(D) $-0\ 9808x-0\ 1881y-0\ 0514z+1\ 0281=0$

O(22), C(21), and C(22) are coplanar, the largest deviation from the plane being 0.075 Å. The molybdenum atoms are displaced (0.371 and 0.387 Å) toward terminal oxygens from the basal planes (Table 4).

The potassium ions are coordinated by four to seven oxygen atoms within the range of 2.7—2.9 Å. All water oxygens (from O(1) through O(4)) except O(5) coordinate to potassium ions, and O(5) is close to O(1) (2.992(16) Å). An additional contact is observed between O(1) and O(22) (2.924(14) Å) indicating formation of a hydrogen bond (Table 2).

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