Crystal and Molecular Structure of Sodium Di-μ-sulphido-bis-[(L-cysteinato)oxomolybdate(V)] Dihydrate

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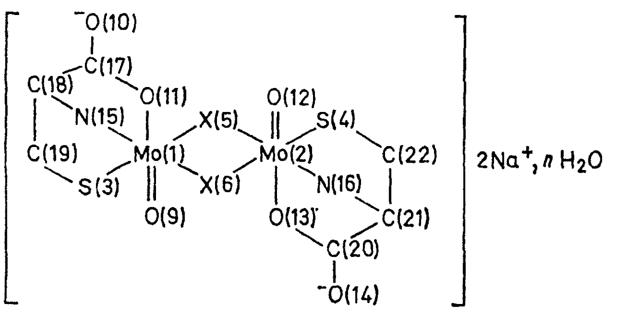
The crystal and molecular structure of the title compound have been determined from *X*-ray photographic data by Patterson and Fourier methods. Crystals are monoclinic prisms, space group $P2_1$, with $Z = 2$ in a cell with $a = 9{\cdot}65$, $b = 15{\cdot}03$, $c = 6{\cdot}76$ Å, $\beta = 92{\cdot}7°$. The structure was refined by least-squares techniques to R 0·151, based on 1808 reflections. The structure of the anion is similar to that of the corresponding compound with bridging oxygen atoms; the molybdenum–molybdenum distance (2·82 Å) is similar to that found in other compounds containing two molybdenum atoms linked by bridging sulphur atoms. A tetrahydrate also exists.

THE compound (Ia) sodium di-μ-sulphido-bis-[(L-cysteinato)oxomolybdate(V) dihydrate whose preparation has been reported previously[1,2] was synthesized here as part of a systematic study, both practical and theoretical, of molybdate complexes. The study includes a calculation of the electronic transition energies for which a set of atomic co-ordinates was needed. We describe the determination of the crystal structure, and compare the findings with those for compounds containing similar partial structures.



(I) a; X = S, $n = 2$
b; X = O, $n = 5$

The crystallographic numbering for (Ia) is shown. In order to illustrate the *cis,trans*-relationships at the molybdenum atoms, the Mo(1) ··· O(9) and Mo(2) ··· O(12) vectors are shown antiparallel instead of parallel

EXPERIMENTAL

The compound crystallized from water as orange prisms, elongated along c with {100} prominent and {110} present.

Crystal Data.—$C_6H_{10}Mo_2N_2Na_2O_6S_2,2H_2O$, $M = 608$, Monoclinic, $a = 9{\cdot}65$, $b = 15{\cdot}03$, $c = 6{\cdot}76$ Å, $\beta = 92{\cdot}7°$, $U = 979$ Å^3, $D_m =$ (*a*) $2{\cdot}03 \pm 0{\cdot}02$, (*b*) $2{\cdot}18 \pm 0{\cdot}01$, $Z = 2$, $D_c = 2{\cdot}06$, $F(000) = 596$. Space group $P2_1$ (No. 4) from systematic absences. Cu-K_α radiation, $\lambda = 1{\cdot}542$ Å; μ(Cu-K_α) = 168·5 cm^{-1}. Crystal dimensions 0·30 × 0·15 × 1·0 mm.

Crystallographic Measurements.—Unit-cell parameters were obtained from rotation and Weissenberg photographs about c, with Cu-K_α radiation; β was determined by the method of angular lag. The parameters accord with those reported in ref. 1, where the axial labels were assigned differently: $a = 6{\cdot}69$, $b = 14{\cdot}95$, $c = 9{\cdot}62$ Å, and $\beta = 92{\cdot}0°$. The space group for our compound cannot be $P2_1/m$ as the material is optically active. The density was determined by flotation in methyl iodide–*m*-dichlorobenzene, and is discussed later.

Intensity data for the layers *hk*0—6 were collected as equi-inclination multiple-film Weissenberg photographs and estimated visually. Accidentally absent reflections were assessed at one half of the locally observable minimum.[3] Intensities were corrected for time of exposure, polarization, and Lorentz factors, but not for absorption, and 1808 independent structure factors were evaluated, of which 140 were unobserved.

Structure Determination.—A three-dimensional Patterson map yielded co-ordinates for the two molybdenum atoms, and successive rounds of structure factor calculations using only isotropic temperature parameters and Fourier syntheses revealed the positions of the remaining ligand atoms, the sodium ions, and two water molecules. In each such round the data were rescaled so that for each layer $\Sigma(\text{scaled}|F_o|) = \Sigma|F_c|$. It was easy to recognize the sites of the ligand atoms and the sodium ions, as their sites (x, y, z) in our compound are related to those (x', y', z') in the compound (Ib)[4] as $x \simeq \frac{1}{2} + 2y'$; $y \simeq \bar{x}' + 0{\cdot}09$; $z \simeq z'$; the additional term in the relation for y follows from a change in origin. Compound (Ib) crystallizes in space group $P2_12_12_1$, in which the origin is fixed not floating as in $P2_1$. At this stage the number of water molecules present was uncertain. A density measurement, on a single crystal from the same crop which yielded the crystal for the structure determination, gave $D_m = 2{\cdot}18 \pm 0{\cdot}01$, consistent with a tetrahydrate, $D_c = 2{\cdot}184$, as reported in ref. 1. The analytical figures fitted those for a trihydrate ($D_c = 2{\cdot}123$) as reported in ref. 2, but were not decisive. Thermal-balance records are consistent with a dihydrate becoming anhydrous in a well-defined process, with a maximum rate of weight loss at 105°, this dihydrate being formed by loss of water at temperatures below the instrument's lower accurate limit (*ca.* 80 °C). When a batch of crystals from the crop described were treated with methyl iodide–*m*-dichlorobenzene (4 : 1, v/v), D_m 2·05, *ca.* 25% of the batch floated and 75% sank. The heavier material had D_m $2{\cdot}18 \pm 0{\cdot}02$, corresponding to a tetrahydrate (assuming the lattice parameters to be the same as those measured), and the lighter material D_m $2{\cdot}03 \pm 0{\cdot}03$, corresponding to a dihydrate (D_c 2·06). We therefore conclude that the compound separates from water as either a di- or a tetra-hydrate, and that the crystal selected for the structure investigation was a dihydrate.

The Fourier maps revealed only two molecules of water. Repeated searches of maps based on F_o, or ($F_o - F_c$), and phased only on atoms definitely sited, or including putative water molecules, failed to reveal peaks of significant height

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[1] B. Spivak and Z. Dori, *Chem. Comm.*, 1970, 1716.

[2] A. Kay and P. C. H. Mitchell, *J. Chem. Soc. (A)*, 1970, 2421.

[3] W. C. Hamilton, *Acta Cryst.*, 1955, **8**, 185.

[4] J. R. Knox and C. K. Prout, *Acta Cryst.*, 1969, **B25**, 1857.

at chemically reasonable sites for any further water molecules. Block-diagonal least-squares refinement was then applied to the parameters, except the y co-ordinate of Mo(1) which was not refined, including anisotropic temperature parameters for molybdenum, and an overall scale-factor. Additionally, scattering factors were corrected for the real part of the anomalous dispersion. When, during the refinement, further oxygen atoms were added, as tests for possible water molecules, the temperature factors of these new atoms rose sharply on attempted refinement; they were then removed from the calculation. Refinement converged at R 0·151. The weighting scheme used was $\sqrt{w} = 1{\cdot}0$ if $|F_o| \leqslant F^*$; $\sqrt{w} = F^*/|F_o|$ if $F_o > F^*$. The value of F^* was adjusted during the refinement; during the last five rounds it was 30·0. In the final round the changes in parameters were all $< 0{\cdot}1\sigma$. The atomic scattering factors were taken from ref. 5, except for the sodium ions, where the values for neutral atoms were used. Observed and calculated structure factors are listed in Supplementary Publications No. SUP 20537 (9 pp., 1 microfiche).*

TABLE 1

Convergence of the refinement

Least-squares round No.	R overall	R' *
13	0·1538	0·0366
14	0·1528	0·0367

Mean values of $w\Delta^2$ in round (14)

Range of $\|F_o\|$	0	10	20	30	40	50	60	70	80	90	100	>100
$\langle w\Delta^2 \rangle$	23·61	14·38	19·88	25·60	32·15	29·07	35·67	27·31	37·17	39·76		44·81
No. of values	192	310	425	272	213	169	77	43	45	24		38

Final structure-factor calculation with output from round (14)

Layer l	0	1	2	3	4	5	6	Overall
R	0·175	0·144	0·132	0·158	0·135	0·151	0·207	0·151
No. of data	164	343	322	303	272	231	173	1808

* $R' = \Sigma w\Delta^2/\Sigma w(KF_o^2)$

TABLE 2

Final co-ordinates and isotropic temperature parameters and, in parentheses, their standard deviations

Atom	x/a	y/b	z/c	U/Å^2
Mo(1)	0·8873(2)	0·5000	0·8080(4)	*
Mo(2)	0·9248(2)	0·6819(2)	0·7201(4)	*
S(3)	0·8068(9)	0·4073(6)	1·0882(13)	0·0261(17)
S(4)	0·8890(9)	0·7814(6)	0·4298(14)	0·0274(17)
S(5)	0·8730(9)	0·5685(6)	0·4966(13)	0·0261(17)
S(6)	0·8758(9)	0·6162(6)	1·0271(14)	0·0262(17)
Na(7)	0·5550(21)	0·6150(16)	0·9878(32)	0·0553(48)
Na(8)	0·5419(25)	0·6168(18)	0·4969(39)	0·0719(65)
O(9)	1·0523(23)	0·4627(15)	0·8196(35)	0·0193(44)
O(10)	0·4616(32)	0·4070(21)	0·7778(47)	0·0405(67)
O(11)	0·6416(23)	0·5014(18)	0·7603(35)	0·0234(46)
O(12)	1·0852(28)	0·6854(23)	0·7316(42)	0·0368(59)
O(13)	0·6858(23)	0·7121(15)	0·7261(35)	0·0179(45)
O(14)	0·5386(31)	0·8274(21)	0·7130(45)	0·0386(65)
N(15)	0·8061(30)	0·3770(21)	0·6351(47)	0·0258(62)
N(16)	0·8906(29)	0·8103(19)	0·8741(42)	0·0216(56)
C(17)	0·5922(41)	0·4227(28)	0·7664(61)	0·0332(83)
C(18)	0·6821(42)	0·3472(28)	0·7557(64)	0·0351(86)
C(19)	0·7252(39)	0·3162(27)	0·9538(59)	0·0305(77)
C(20)	0·6556(36)	0·7963(23)	0·7293(54)	0·0244(69)
C(21)	0·7762(47)	0·8598(33)	0·7645(73)	0·0449(102)
C(22)	0·8182(55)	0·8817(38)	0·5438(83)	0·0530(120)
O(W; 23)	0·5576(42)	0·5109(31)	0·2533(62)	0·0641(100)
O(W; 24)	0·6010(33)	0·7214(23)	0·2241(50)	0·0444(75)

* For these atoms an anisotropic temperature factor (T) was used, given by $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

Final values for the components of the tensor ($\times 10^4$)

Atom	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{13}$	$2U_{23}$
Mo(1)	143(12)	230(11)	192(14)	27(19)	52(18)	−14(20)
Mo(2)	94(11)	223(11)	225(14)	9(18)	78(17)	−20(21)

RESULTS

Table 1 shows the final weighting scheme analysis and convergence of the refinement. Table 2 gives the final co-ordinates of the atoms, their temperature factors and standard deviations derived from the final least-squares matrix. (Ia) shows the anion, and the crystallographic numbering. Figure 1 shows the asymmetric unit in projection on the ab plane. Table 3 lists interatomic distances, valency angles, and some nonbonded interactions. Table 4 gives the mean values for the lengths and angles of selected types of bond, together with the means of some previously measured values, and the mean estimated standard deviations for bond lengths and angles.

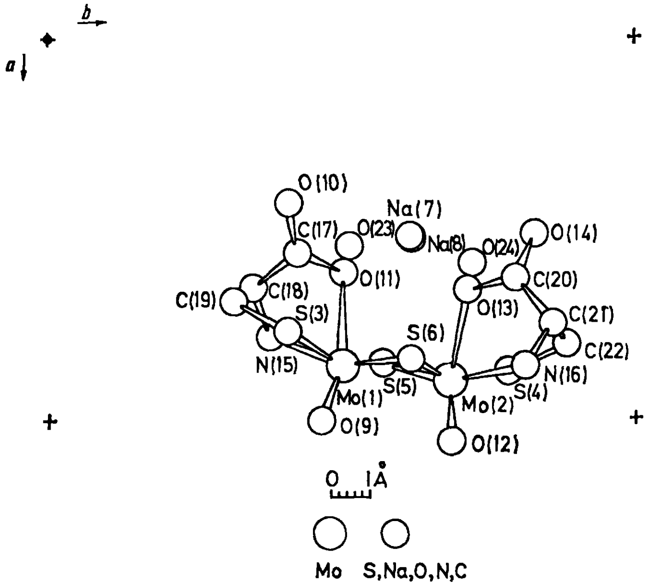

FIGURE 1 The asymmetric unit projected on the ab plane

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* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. are sent as full size copies).

[5] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962, p. 202.

The crystal structure and the conformation of the anion are very similar to those for the oxygen-bridged compound (Ib).[4] The epimer we chose is the same as that on which the calculations for (Ib) were based, and represents cysteine in its true chirality.[6] There is no evidence of disorder in the crystal; the temperature factors are comparable with those for similar compounds. A difference-Fourier map, calculated on the parameters from the final least-squares round showed both positive and negative peaks. These we ascribe to absorption errors; the highest peaks, both positive and negative, are grouped symmetrically about each of the two molybdenum atoms.

TABLE 3

Selected valency parameters

(*a*) Distances (Å)

Mo(1) · · · Mo(2) 2·82

Mo(1)–S(3)	2·51	Mo(2)–S(4)	2·48
Mo(1)–S(5)	2·34	Mo(2)–S(5)	2·32
Mo(1)–S(6)	2·30	Mo(2)–S(6)	2·37
Mo(1)–O(9)	1·69	Mo(2)–O(12)	1·55
Mo(1)–O(11)	2·38	Mo(2)–O(13)	2·35
Mo(1)–N(15)	2·31	Mo(2)–N(16)	2·22

(*b*) Bond angles (°)

Mo(2)–Mo(1)–S(3)	138	Mo(1)–Mo(2)–S(4)	137
Mo(2)–Mo(1)–S(5)	52	Mo(1)–Mo(2)–S(5)	53
Mo(2)–Mo(1)–S(6)	54	Mo(1)–Mo(2)–S(6)	52
Mo(2)–Mo(1)–O(9)	102	Mo(1)–Mo(2)–O(12)	99
Mo(2)–Mo(1)–O(11)	96	Mo(1)–Mo(2)–O(13)	93
Mo(2)–Mo(1)–N(15)	136	Mo(1)–Mo(2)–N(16)	136
S(3)–Mo(1)–S(5)	157	S(4)–Mo(2)–S(5)	85
S(3)–Mo(1)–S(6)	85	S(4)–Mo(2)–S(6)	157
S(3)–Mo(1)–O(9)	96	S(4)–Mo(2)–O(12)	97
S(3)–Mo(1)–O(11)	76	S(4)–Mo(2)–O(13)	78
S(3)–Mo(1)–N(15)	80	S(4)–Mo(2)–N(16)	80
S(5)–Mo(1)–S(6)	104	S(5)–Mo(2)–S(6)	103
S(5)–Mo(1)–O(9)	102	S(5)–Mo(2)–O(12)	104
S(5)–Mo(1)–O(11)	82	S(5)–Mo(2)–O(13)	88
S(5)–Mo(1)–N(15)	84	S(5)–Mo(2)–N(16)	156
S(6)–Mo(1)–O(9)	107	S(6)–Mo(2)–O(12)	102
S(6)–Mo(1)–O(11)	90	S(6)–Mo(2)–O(13)	80
S(6)–Mo(1)–N(15)	156	S(6)–Mo(2)–N(16)	85
O(9)–Mo(1)–O(11)	160	O(12)–Mo(2)–O(13)	166
O(9)–Mo(1)–N(15)	93	O(12)–Mo(2)–N(16)	97
O(11)–Mo(1)–N(15)	68	O(13)–Mo(2)–N(16)	70
Mo(1)–S(3)–C(19)	101	Mo(2)–S(4)–C(22)	102
Mo(1)–S(5)–Mo(2)	75	Mo(2)–S(6)–Mo(1)	75

(*c*) Contacts, not involving σ-bonds, < 3·5 Å

(i) Bonding contacts

$\mathrm{N}(7^{\mathrm{I}}) \cdots \mathrm{O}(11^{\mathrm{I}})$	2·47	$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{I}})$	2·51
$\mathrm{Na}(7^{\mathrm{I}}) \cdots \mathrm{O}(13^{\mathrm{I}})$	2·66		
$\mathrm{Na}(7^{\mathrm{I}}) \cdots \mathrm{W}(23^{\mathrm{II}})$	2·38	$\mathrm{O}(10^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{III}})$	2·85
$\mathrm{Na}(7^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{II}})$	2·29	$\mathrm{O}(14^{\mathrm{I}}) \cdots \mathrm{W}(23^{\mathrm{IV}})$	2·92
$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{O}(11^{\mathrm{I}})$	2·64	$\mathrm{W}(23^{\mathrm{I}}) \cdots \mathrm{O}(14^{\mathrm{III}})$	2·92
$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{O}(13^{\mathrm{I}})$	2·49	$\mathrm{W}(24^{\mathrm{I}}) \cdots \mathrm{O}(10^{\mathrm{IV}})$	2·85
$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{W}(23^{\mathrm{I}})$	2·30		

(ii) Non-bonded contacts

$\mathrm{W}(23^{\mathrm{I}}) \cdots \mathrm{S}(3^{\mathrm{II}})$	3·10	$\mathrm{W}(24^{\mathrm{I}}) \cdots \mathrm{S}(6^{\mathrm{II}})$	3·41
$\mathrm{W}(23^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{I}})$	3·19	$\mathrm{O}(10^{\mathrm{I}}) \cdots \mathrm{C}(22^{\mathrm{III}})$	2·43
$\mathrm{S}(5^{\mathrm{I}}) \cdots \mathrm{S}(6^{\mathrm{II}})$	3·25	$\mathrm{O}(13^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{I}})$	3·45
$\mathrm{Na}(7^{\mathrm{I}}) \cdots \mathrm{C}(17^{\mathrm{I}})$	3·27	$\mathrm{W}(23^{\mathrm{I}}) \cdots \mathrm{O}(11^{\mathrm{II}})$	2·45
$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{C}(20^{\mathrm{I}})$	3·29	$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{Na}(7^{\mathrm{II}})$	3·46
$\mathrm{C}(18^{\mathrm{I}}) \cdots \mathrm{W}(24^{\mathrm{III}})$	3·34	$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{C}(17^{\mathrm{I}})$	3·46
$\mathrm{Na}(7^{\mathrm{I}}) \cdots \mathrm{C}(20^{\mathrm{I}})$	3·40	$\mathrm{Na}(8^{\mathrm{I}}) \cdots \mathrm{O}(14^{\mathrm{I}})$	3·49

Roman numerals as superscripts denote the following equivalent positions:

I x, y, z	III $1 - x, -\frac{1}{2} + y, 1 - z$
II $x, y, -1 + z$	IV $1 - x, \frac{1}{2} + y, 1 - z$

TABLE 4

Means of selected valency parameters; values in parentheses are the means of those previously published

(*a*) Bond lengths (Å)

Mo–Mo	2·82	(2·80,^{a} 2·89 ^{b})
Mo–S(b), *trans* to S	2·35	(2·32 ^{a})
trans to N	2·31	(2·30 ^{a})
Mean	2·33	(2·31,^{a} 2·31 ^{b})
Mo–S(c)	2·50	(2·49,^{c} 2·38,^{a} 2·39 ^{d})
Mo=O	1·62	(1·71,^{a} 1·66,^{d} 1·68,^{b} 1·71 ^{c})
Mo–O	2·37	(2·30 ^{c})
Mo–N	2·27	(2·23,^{c} 2·22,^{d} 2·23 ^{c})
S–C	1·82	
C–O(Mo)	1·29	
C–O	1·26	
C–N	1·53	
C–C	1·50	

(*b*) Bond angles (°)

S(5)–Mo–S(6)	103	(102,^{a} 103 ^{d})
Mo(1)–S(b)–Mo(2)	75	(75,^{a} 77 ^{d})

(*c*) Mean estimated standard deviations

Bond lengths (Å)		Bond angles (°)	
Mo–Mo	0·003	Mo–Mo–S	0·7
Mo–S	0·009	Mo–Mo–(O,N,C)	1·7
Mo–(O,N,C)	0·025	S–Mo–S	0·8
S–(O,N,C)	0·048	(L)–Mo–(L)	2·5
(L)–(L) light atom	0·054	3 light atoms	4·9

(b) Denotes a bridging atom, (c) an atom in the cysteine ligand, (L) a light atom.

^{a} Ref. 7. ^{b} Ref. 8. ^{c} Ref. 4. ^{d} Ref. 9.

TABLE 5

Out-of-plane distances (Å)

Plane (1): S(3), S(5), S(6), N(15)

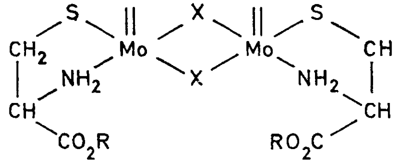
S(3) 0·00, S(5) 0·00, S(6) 0·00, N(15) 0·01, Mo(1) −0·42, O(9) −2·09, O(11) 1·91

Plane (2): S(4)—(6), N(16)

S(4) 0·00, S(5) 0·00, S(6) 0·00, N(16) 0·00, Mo(2) −0·42, O(12) −1·96, O(13) 1·90

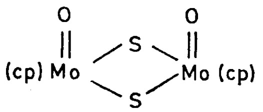
DISCUSSION

The molybdenum–molybdenum distance (2·82 Å) is similar to that found in the analogous compound (IIa) (2·80 Å),[7] and in the sulphur-bridged cyclopentadienyl molybdenum compound (III) (2·894 Å).[8] Within com-



(II)

a; R = Me, X = S
b; R = Et, X = O



(III)

cp = cyclopentadienyl

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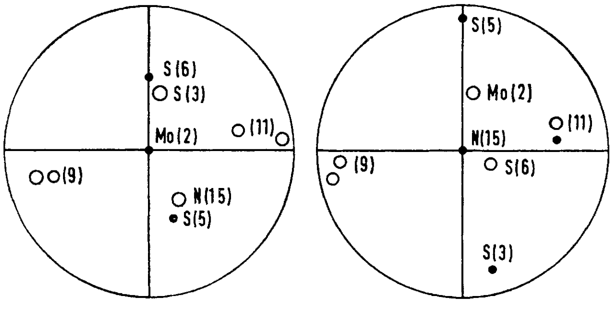
[6] M. M. Harding and H. A. Long, *Acta Cryst.*, 1968, **B24**, 1096.

[7] M. G. B. Drew and A. Kay, *J. Chem. Soc.* (*A*), 1971, 1851.

[8] D. L. Stevenson and L. F. Dahl, *J. Amer. Chem. Soc.*, 1967, **89**, 3721.

[9] M. G. B. Drew and A. Kay, *J. Chem. Soc.* (*A*), 1971, 1846.

pound (Ia) corresponding bond angles at the two molybdenum atoms are very similar, and the molybdenum atoms have the ligand atoms in distorted octahedral co-ordination. Figure 2(a) shows a stereographic projection of the bond vectors round Mo(1) with the Mo(1) ··· Mo(2) vector at the N pole; Figure 2(b) shows the projection with the Mo(1) ··· N(15) vector at the N pole. The bond angles at the molybdenum atoms all lie within 3° of that for the corresponding bonds in the oxygen-bridged analogue (Ib).[4] Round each molybdenum atom the three sulphur and one nitrogen atoms are coplanar, and the molybdenum atoms are each displaced by 0·42 Å from this plane toward the oxo-oxygen atom; this value is not significantly different from that (0·38 Å) found for compound (Ib).[4] The bridging sulphur atoms are not symmetrically related to the two molybdenum atoms, and the difference between the two lengths [0·04 for Mo(1), 0·05 Å for Mo(2)] is *ca.* 5σ for this type of bond. The molybdenum–sulphur bond *trans* to a sulphur atom is longer than a molybdenum–sulphur bond *trans* to a nitrogen atom. A similar difference in length is reported for compound (IIa);[7] an analogous difference in bond lengths for bridging oxygen atoms has been observed in compound (Ia),[4] but not in compound (IIb).[9] Bond angles involving the molybdenum and bridging sulphur atoms are similar to previously-measured values (Table 4). The two halves of the anion show slight relative twisting; the plane containing Mo(1), Mo(2), and O(9), and that containing Mo(1), Mo(2), and O(12) are inclined at 5°. The plane containing Mo(1), S(5), and S(6) is inclined to that containing Mo(2), S(5), and S(6) at 156°. The corresponding figure for the oxygen-bridged compound is 151°.



(a) (b)

FIGURE 2 Stereographic projections of the bond vectors round Mo(1), with (a) the Mo(1) ··· Mo(2), and (b) the Mo ··· N(15) vector at the N pole

Within each cysteine ligand the mean bond lengths and angles do not differ significantly from the values found for compound (Ib).[4]

Figure 3 shows the packing projected down *a*; this is very different from that in the oxygen-bridged analogue.[4] The two sodium ions are in similar environments, each associated with those oxygen atoms of the carboxylate groups which are bonded to molybdenum, the other oxygen atoms of these carboxylate groups being associated with water molecules. The sodium–oxygen, sodium–water, and sodium–sodium distances are unexceptional. There is a possible hydrogen bond (3·10 Å) between W(23) and S(3),[10] that is not shown in the Figure; there is no indication of N–H ··· S bonding. The contact $S(5^{I}) \cdots S(6^{II})$ is close, but the implied value (1·62 Å) for the van der Waals radius of sulphur agrees with Donohue's value, 1·60 Å,[11] and a shorter intramolecular S ··· S contact (3·19 Å), has recently been reported.[12]

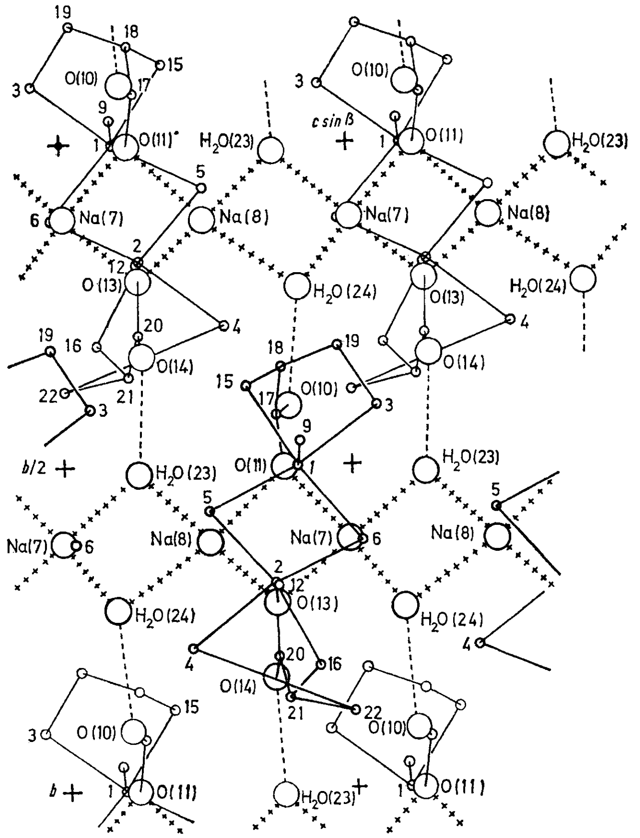


FIGURE 3 The crystal packing viewed down *a*. Atom sites (small circles) are identified by the crystallographic number. Large circles indicate sodium ions, or oxygen atoms involved in intermolecular interactions, and are labelled appropriately. Dashes indicate hydrogen bonds, crosses, electrostatic interactions, and thicker lines indicate proximity to the observer

Calculations were carried out on an ICI 1905 computer, at the University of Strathclyde. We thank Mrs. Metters (née Tuladhar) for the drawings.

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