Crystal and Molecular Structure of Di-μ-Sulphido-bis[(L-histidinato)oxomolybdenum(V)] Sesquihydrate

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The crystal and molecular structure of the title compound has been determined from single-crystal three-dimensional *X*-ray diffraction data collected by counter methods. Crystals are tetragonal, space group $P4_12_12$, with unit-cell dimensions: $a = b = 10{\cdot}549(14)$, $c = 36{\cdot}510(14)$ Å, $Z = 8$. The structure was solved by the heavy-atom method and refined by full-matrix least-squares to R 0·050 (for 3044 observed reflections). Each molybdenum atom is co-ordinated to two bridging sulphur atoms, one terminal oxygen atom, and a terdentate histidinato-ligand. Consideration of the Mo · · · Mo distance [2·848(1) Å] suggests the co-ordination geometry around each metal atom can best be described as a seven-co-ordinate distorted capped octahedron sharing a common edge determined by the two sulphur atoms. Factors influencing the co-ordination geometry in this and related complexes are described.

MOLYBDENUM in its higher oxidation states, *i.e.* +6, +5, and +4, is a necessary cofactor in several redox enzymes, and considerable evidence suggests that at the active site molybdenum is bound, at least in part, to sulphur donor atoms.[1] Thus, its chemistry in these oxidation states has become an area of active research, in particular the interaction of molybdenum with sulphur and with biologically significant ligands such as the α-amino-acids.[2]

As part of our study of the chemical and structural aspects of sulphido-bridged complexes of $\mathrm{Mo^V}$, we report here the structure of the title compound. A preliminary report has appeared.[3]

EXPERIMENTAL

Crystals suitable for *X*-ray structural analysis were obtained as described previously.[3]

Crystal Data.—$C_{12}H_{16}Mo_2N_6O_6S_2,1\frac{1}{2}H_2O$, $M = 623{\cdot}3$, Tetragonal, $a = b = 10{\cdot}549(4)$, $c = 36{\cdot}510(14)$ Å, $U = 4062{\cdot}8$ Å^3, $D_m = 2{\cdot}02$ (by flotation), $Z = 8$, $D_c = 2{\cdot}037$. Cu-K_α (graphite monochromator) radiation, $\lambda = 1{\cdot}5412$ Å; μ(Cu-K_α) = 132·52 cm^{-1}. Space group $P4_12_12$ (D_4^4, No. 92) or $P4_32_12$ (D_4^8, No. 96) from systematic absences; $00l$ $l \neq 4n$, and $h00$ $h \neq 2n$; shown to be the former for the subsequent successful analysis.

A rectangular plate of dimensions 0·24 × 0·24 × 0·08 mm was selected and mounted on an edge perpendicular to the flat face (100). Preliminary unit-cell and space-group data were obtained from precession and Weissenberg photographs. Unit-cell dimensions were obtained from back-reflection Weissenberg photographs with Cu-K_α radiation.[4] Owing to the length of the c axis and the overlapping of spots with Mo-K_α radiation, Cu-K_α radiation was used for data collection. By use of equi-inclination Weissenberg geometry, intensity data for the levels 0—$7kl$ were collected on a Stoe semi-automatic diffractometer by the stationary-background, ω—2θ scan, stationary-background counting sequence. Data were collected to sin θ/λ 0·49 and crystal stability was monitored by measurement of 2 standard

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[1] R. C. Bray and J. C. Swann, *Structure and Bonding*, 1972, **11**, 107.

[2] P. Kroneck and J. T. Spense, *Biochem.*, 1973, **12**, 5020, and refs. therein.

[3] B. Spivack, A. P. Gaughan, and Z. Dori, *J. Amer. Chem. Soc.*, 1971, **93**, 5265.

[4] F. H. Herbstein, *Acta Cryst.*, 1963, **16**, 255.

reflections on each level every 40 reflections; no significant variations were observed throughout data collection.

Raw data were processed as described previously [5] and corrections for absorption (transmission factors ranged from 0·150 to 0·450), Lorentz, and polarization effects were applied. F_o Values were brought to an approximate scale through a modification of Wilson's procedure.

Solution and Refinement of Structure.—Positional parameters obtained from the previous study [3] were refined by a least-squares procedure in which the function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where $w = 1/6$ for $F_o \leqslant 36$ and $w = 1/(F_o)^2$ for $F_o > 36$. Of the 3215 reflections measured only those (3044) for which $(F_o)^2 > 3\sigma(F_o)^2$ were used in the refinement. Neutral atom scattering factors used in these calculations were from ref. 6; anomalous dispersion effects were included in the calculation, values of $\Delta f'$ and $\Delta f''$ for molybdenum and sulphur being taken from ref. 7. This refinement led to the discrepancy indices R 0·135 and the weighted factor R' 0·171, where $R' = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o{}^2]^{\frac{1}{2}}$.

A three-dimensional Fourier map based on this refinement revealed the presence of an additional water molecule located on a two-fold axis of the unit cell. Three cycles of full-matrix least-squares refinement of the complete structure, in which the water molecule located on the two-fold axis was assigned an occupancy factor of 0·5 in the calculation, including scale factors, positional parameters, and isotropic temperature factors led to R 0·083 and R' 0·104. An additional three cycles of anisotropic refinement of the entire model converged with R 0·050 and R' 0·067.

At convergence the largest parameter shifts were $< 0{\cdot}1\sigma$. The estimated standard deviation of an observation of unit weight of 0·51 e and the final difference-Fourier map showed peak heights $< 0{\cdot}1$ those found for a carbon atom in this structure. Final positional and thermal parameters are listed in Table 1, with their standard deviations. Final observed and calculated structure factors are listed in Supplementary Publication No. SUP 21291 (3 pp., 1 microfiche).*

TABLE 1

Final positional and thermal * parameters with estimated standard deviations in parentheses

Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	1624(1)	−3062(1)	1027(0)	49(1)	29(1)	5(0)	0(0)	−3(0)	−0(0)
Mo(2)	−696(1)	−2554(1)	1398(0)	43(1)	41(1)	7(0)	3(0)	−3(0)	−3(0)
S(1)	545(2)	−4372(2)	1433(1)	58(3)	54(2)	8(0)	2(2)	1(0)	3(0)
S(2)	154(2)	−1501(2)	894(1)	82(3)	51(2)	9(0)	10(2)	−4(0)	1(0)
O(Mo1)	2686(6)	−2343(6)	1303(2)	78(8)	63(7)	8(0)	−6(6)	−3(2)	−4(1)
O(Mo2)	−230(6)	−1670(7)	1749(2)	68(7)	84(8)	10(1)	−18(6)	−6(2)	−5(2)
O(1)	776(6)	−4280(6)	599(2)	56(6)	51(6)	9(0)	17(5)	−6(1)	−4(1)
O(2)	1082(7)	−5857(7)	202(2)	84(8)	108(9)	10(1)	−2(7)	−9(2)	−15(2)
O(1′)	−2047(6)	−3508(6)	1029(2)	64(7)	63(6)	8(0)	6(5)	−3(1)	−3(2)
O(2′)	−4056(8)	−3845(8)	891(2)	95(9)	113(10)	15(1)	−4(8)	−21(2)	−11(2)
W(1)	−1490(12)	−3210(14)	246(2)	228(18)	295(21)	9(1)	−25(17)	−4(3)	−4(3)
W(2)	1578(24)	1578(24)	0	69(28)	69(28)	40(3)	−5(18)	21(11)	−21(11)
N(1)	2967(7)	−4700(7)	940(2)	68(8)	51(7)	6(0)	−6(6)	−4(2)	−2(2)
N(2)	2661(7)	−2432(7)	522(2)	59(8)	60(8)	8(0)	−24(7)	−3(2)	4(2)
N(3)	3566(9)	−1106(9)	146(2)	96(12)	93(11)	10(1)	−30(9)	−6(2)	3(2)
N(1′)	−2417(9)	−1294(8)	1313(3)	72(9)	60(9)	17(1)	16(7)	−8(3)	−13(2)
N(2′)	−2014(8)	−3510(8)	1791(2)	60(8)	92(10)	10(1)	2(8)	1(2)	−3(2)
N(3′)	−2615(9)	−4690(10)	2264(2)	91(11)	103(11)	11(1)	−6(9)	7(2)	4(2)
C(1)	1442(9)	−5150(8)	451(2)	57(10)	51(9)	7(1)	−10(7)	−2(2)	−2(2)
C(2)	2827(10)	−5257(9)	568(2)	78(11)	61(10)	7(1)	−5(8)	−4(2)	0(2)
C(3)	3645(8)	−4512(10)	298(2)	47(9)	88(11)	8(1)	5(8)	−3(2)	0(2)
C(4)	3412(9)	−3122(9)	287(2)	77(10)	65(10)	6(1)	−11(8)	−5(2)	−1(2)
C(5)	2759(10)	−1221(9)	436(2)	94(12)	58(10)	8(1)	−19(9)	0(2)	4(2)
C(6)	3992(10)	−2326(11)	44(3)	79(12)	108(14)	10(1)	−26(10)	3(3)	10(3)
C(1′)	−3236(10)	−3235(10)	1036(2)	82(11)	84(11)	7(1)	6(9)	−1(2)	1(2)
C(2′)	−3606(11)	−2077(10)	1268(3)	94(14)	68(11)	14(1)	10(10)	−8(3)	−10(3)
C(3′)	−4125(12)	−2463(13)	1630(4)	85(14)	14(17)	15(1)	37(13)	−6(4)	−5(4)
C(4′)	−3339(12)	−3357(12)	1852(3)	103(14)	94(14)	10(1)	0(11)	0(3)	−16(3)
C(5′)	−1633(12)	−4308(12)	2045(3)	97(13)	97(13)	11(1)	−12(11)	−4(3)	1(3)
C(6′)	−3705(13)	−4095(12)	2142(4)	131(16)	84(13)	13(1)	−8(11)	7(4)	−6(3)

* In the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

RESULTS AND DISCUSSION

Figure 1 shows a drawing of the molecule, together with the atom labelling system used in the analysis; bond angles and lengths are listed in Table 2. Each independent histidinato-ligand co-ordinates to the molybdenum atom through the carboxylato-oxygen, the

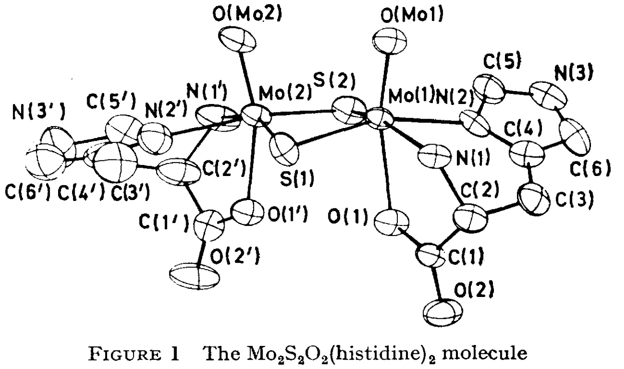

FIGURE 1 The $Mo_2S_2O_2$(histidine)$_2$ molecule

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1974, Index issue.

[5] B. Spivack and Z. Dori, *J.C.S. Dalton*, 1973, 1173, and refs. therein.

[6] D. T. Cromer and J. T. Waber, *Acta Cryst.*, 1965, **18**, 104.

[7] D. N. Templeton, 'International Tables for *X*-Ray Crystallography,' vol. 3, Kynoch Press, Birmingham, 1962, Table 3.3.2.

α-amino-nitrogen N(1), and N(2) of the imidazole ring. The remainder of the co-ordination sphere around each molybdenum atom consists of one terminal oxygen atom and two bridging sulphur atoms. Consideration of the two Mo–Mo distances (*vide infra*) leads to the conclusion that the co-ordination geometry about each molybdenum atom can best be described as a seven-co-ordinate distorted capped octahedron sharing a common edge defined by the two bridging sulphur atoms.

TABLE 2

Significant molecular geometry

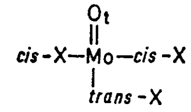
(*a*) Distances (Å)			
Mo(1) · · · Mo(2)	2·848(1)	S(1)–S(2)	3·636(3)
Mo(1)–S(1)	2·323(2)	Mo(2)–S(1)	2·342(3)
Mo(1)–S(2)	2·314(3)	Mo(2)–S(2)	1·331(3)
Mo(1)–O(Mo1)	1·686(6)	Mo(2)–O(Mo2)	1·666(7)
Mo(1)–O(1)	2·213(6)	Mo(2)–O(1′)	2·204(6)
Mo(1)–N(1)	2·257(7)	Mo(2)–N(1′)	2·272(9)
Mo(1)–N(2)	2·245(7)	Mo(2)–N(2′)	2·237(8)
O(1)–C(1)	1·28(1)	O(1′)–C(1′)	1·29(1)
O(2)–C(1)	1·23(1)	O(2′)–C(1′)	1·20(1)
C(1)–C(2)	1·53(1)	C(1′)–C(2′)	1·52(1)
C(2)–N(1)	1·49(1)	C(2′)–N(1′)	1·52(1)
C(2)–C(3)	1·53(1)	C(2′)–C(3′)	1·48(2)
C(3)–C(4)	1·49(1)	C(3′)–C(4′)	1·50(2)
C(4)–N(2)	1·38(1)	C(4′)–N(2′)	1·42(1)
N(2)–C(5)	1·32(1)	N(2′)–C(5′)	1·32(1)
C(5)–N(3)	1·36(1)	C(5′)–N(3′)	1·37(2)
N(3)–C(6)	1·41(1)	N(3′)–C(6′)	1·38(2)
C(6)–C(4)	1·37(1)	C(6′)–C(4′)	1·37(2)
(*b*) Angles (°)			
S(1)–Mo(1)–S(2)	103·2(1)	S(1)–Mo(2)–S(2)	102·7(1)
Mo(1)–S(1)–Mo(2)	75·6(1)	Mo(1)–S(2)–Mo(2)	75·6(1)
Mo(2)–Mo(1)–O(Mo1)	101·7(2)	Mo(1)–Mo(2)–O(Mo2)	101·7(2)
Mo(2)–Mo(1)–O(1)	95·6(2)	Mo(1)–Mo(2)–O(1′)	100·3(2)
S(1)–Mo(1)–O(Mo1)	102·3(2)	S(1)–Mo(2)–O(Mo2)	104·1(2)
S(1)–Mo(1)–O(1)	84·7(2)	S(1)–Mo(2)–O(1′)	91·2(2)
S(1)–Mo(1)–N(1)	86·7(2)	S(1)–Mo(2)–N(1′)	160·1(3)
S(1)–Mo(1)–N(2)	159·8(2)	S(1)–Mo(2)–N(2′)	86·8(2)
S(2)–Mo(1)–O(Mo1)	104·5(2)	S(2)–Mo(2)–O(Mo2)	102·7(2)
S(2)–Mo(1)–O(1)	89·6(2)	S(2)–Mo(2)–O(1′)	89·0(2)
S(2)–Mo(1)–N(1)	159·3(2)	S(2)–Mo(2)–N(1′)	85·4(2)
S(2)–Mo(1)–N(2)	86·7(2)	S(2)–Mo(2)–N(2′)	163·8(2)
O(Mo1)–Mo(1)–O(1)	162·1(3)	O(Mo2)–Mo(2)–O(1′)	157·9(3)
O(Mo1)–Mo(1)–N(1)	90·7(3)	O(Mo2)–Mo(2)–N(1′)	91·6(3)
O(Mo1)–Mo(1)–N(2)	91·8(3)	O(Mo2)–Mo(2)–N(2′)	87·4(3)
O(1)–Mo(1)–N(1)	73·1(2)	O(1′)–Mo(2)–N(1′)	70·5(3)
O(1)–Mo(1)–N(2)	77·8(2)	O(1′)–Mo(2)–N(2′)	77·5(3)
N(1)–Mo(1)–N(2)	78·7(3)	N(1′)–Mo(2)–N(2′)	81·7(3)
Mo(1)–O(1)–C(1)	119·6(5)	Mo(2)–O(1′)–C(1′)	121·1(6)
O(1)–C(1)–O(2)	125·2(8)	O(1′)–C(1′)–O(2′)	125·0(9)
O(1)–C(1)–C(2)	117·5(7)	O(1′)–C(1′)–C(2′)	115·8(9)
O(2)–C(1)–C(2)	117·1(8)	O(2′)–C(1′)–C(2′)	119·0(10)
C(1)–C(2)–N(1)	108·8(7)	C(1′)–C(2′)–N(1′)	106·6(9)
Mo(1)–N(1)–C(2)	111·6(6)	Mo(2)–N(1′)–C(2′)	110·3(6)
C(1)–C(2)–C(3)	108·7(8)	C(1′)–C(2′)–C(3′)	112·8(10)
N(1)–C(2)–C(3)	109·3(7)	N(1′)–C(2′)–C(3′)	111·1(10)
C(2)–C(3)–C(4)	115·5(8)	C(2′)–C(3′)–C(4′)	116·4(11)
C(3)–C(4)–N(2)	126·9(8)	C(3′)–C(4′)–N(2′)	121·9(10)
C(3)–C(4)–C(6)	123·3(8)	C(3′)–C(4′)–C(6′)	128·5(11)
Mo(1)–N(2)–C(4)	129·5(6)	Mo(2)–N(2′)–C(4′)	131·3(7)
Mo(1)–N(2)–C(5)	121·4(6)	Mo(2)–N(2′)–C(5′)	123·4(7)
C(4)–N(2)–C(5)	108·5(7)	C(4′)–N(2′)–C(5′)	105·1(9)
N(2)–C(5)–N(3)	108·7(9)	N(2′)–C(5′)–N(3′)	111·7(10)
C(5)–N(3)–C(6)	108·9(9)	C(5′)–N(3′)–C(6′)	107·8(9)
N(3)–C(6)–C(4)	108·6(9)	N(3′)–C(6′)–C(4′)	105·9(11)

The molybdenum atoms are displaced 0·28 and 0·30 Å towards the oxomolybdenum oxygen atom away from the bonding plane defined by the six atoms: N(1), N(2), S(1), S(2), N(1′), N(2′). This is a consequence of the multiply bonded terminal oxygen atoms (O_t), as manifested by the short Mo–O_t bond length of 1·67 Å and as observed in a variety of doubly bridged Mo^V complexes having terminal oxygen atoms.[5] The mean molybdenum carboxylato-oxygen bond length (2·210 Å) is identical to that found in the corresponding oxo-bridged complex [8] but is significantly longer than that (2·11 Å) in $Cs_2Mo_2S_2O_2$(edta),$2H_2O$ and $BaMo_2O_4(C_2O_4)_2(H_2O)_2$.[9] The differences in the mean Mo–N(amino) (2·263 Å) and Mo–N(imidazole) (2·241 Å) distances are not significant, and further illustrate the observations of Freeman [10] that the imidazole group of histidine is not involved in d_π–p_π bonding to metal atoms. Furthermore, the metal–nitrogen bond lengths found here are similar to those for other Mo^V complexes having σ-bonded nitrogen atoms.[5] The mean Mo–S distance (2·323 Å) is representative of other such sulphido-bridged Mo^V structures.[11,12]

The bond lengths and angles of the two crystallographically independent histidine molecules are similar (Table 2) and compare favourably with those of other histidinato-complexes in both dimension and conformation.[8,10] The bonding of the histidinato-ligand in this and in the analogous oxo-bridged complex is not significantly different, as shown by the bond lengths and angles subtended at molybdenum. Similar behaviour has been observed in two other ligand systems of Mo^V in which the bridging atoms were respectively oxygen and sulphur.[11,12] Thus, replacement of the bridging atom by sulphur has no significant effect on the bonding between molybdenum and non-bridging atoms, but only on the dimension and conformation of the bridge itself.

The Mo_2S_2 bridge is not planar, the dihedral angle between the planes defined by Mo(1), S(1), S(2) and Mo(2), S(1), S(2) being 159·7°. This behaviour is common in Mo_2X_2 (X = O or S) bridged complexes and is a consequence of the tetragonal distortion brought about by the Mo–O_t bond.

As previously mentioned, the molybdenum carboxylato-oxygen distance (2·210 Å) is larger than in other Mo^V complexes. This lengthening of bonds *trans* to O_t is generally assumed to result from the *trans*-influence of this atom. However, steric effects may also be partially responsible; for similar geometric arguments concerning osmium–nitrido-complexes see ref. 13. Assuming perfect octahedral geometry around the molybdenum atom and using observed bond lengths, the non-bonded distances of neighbouring atoms were calculated by use of the following



$$\begin{array}{c} O_t \\ \| \\ \textit{cis}\text{-X—Mo—}\textit{cis}\text{-X} \\ | \\ \textit{trans}\text{-X} \end{array}$$

where *cis*-X represents atoms *cis* and *trans*-X *trans* to O_t. The results of calculations for several complexes (Table 3)

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[8] L. T. J. Delbaere and C. K. Prout, *Chem. Comm.*, 1971, 162; C. K. Prout, personal communication.

[9] F. A. Cotton and S. M. Morehouse, *Inorg. Chem.*, 1966, **4**, 1377.

[10] H. C. Freeman, *Adv. Protein Chem.*, 1967, **22**, 257.

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

[12] D. H. Brown and J. A. D. Jeffereys, *J.C.S. Dalton*, 1973, 732.

[13] D. Bright and J. A. Ibers, *Inorg. Chem.*, 1969, **8**, 709.

indicate that complexes with large *trans*-influences usually have short $O_t \cdots C$ non-bonded distances in this geometry. Consequently, the bending of *cis*-X atoms away from the Mo–O_t bond relieves most of the non-bonded repulsions, but necessitates the lengthening of the Mo–X(*trans*) bond to maintain reasonable non-bonded distances between the *cis*- and *trans*-X atoms. These effects, however, are not exclusively responsible for lengthening of the Mo–X(*trans*) bond; *e.g.* histidine, which forms five- and six-membered rings on complex formation (Figure 1), is more flexible than cysteine which forms two five-membered rings. Thus, the added length of the molybdenum–carboxylato-oxygen bond in the cysteine complexes of the $[Mo_2X_2O_2]^{2+}$ (X = O or S) ions [14] is due to the conformation of the cysteinato-ligand as described by Knox and Prout.[15] In the mono-oxygen-bridged complexes, $Mo_2O_3\{S_2P(OEt)_2\}_4$ and $Mo_2O_3(S_2COEt)_4$,[16,17] the larger size of the co-ordinated sulphur atoms undoubtedly contributes to the lengthening of the Mo–X(*trans*) bonds by the foregoing mechanism.

TABLE 3

Comparison of *cis*-X ⋯ O distances (Å) in several Mo^V complexes

Complex	Mo–O_t	Mo–X(*cis*) [a]	*cis*-X ⋯ O Obs.	*cis*-X ⋯ O Calc.
$Mo_2S_2O_2(histidine)_2,1\frac{1}{2}H_2O$ [b]	1·67	2·25 (N) [c]	2·82	2·80
$Mo_2O_4(histidine)_2,3H_2O$ [d]	1·71	2·24 (N) [c]	2·84	2·82
$Cs_2Mo_2O_2(edta),2H_2O$ [e]	1·67	2·11 (O) [c]	2·80	2·70
$Na_2Mo_2O_4(cysteine)_2,5H_2O$ [f]	1·71	2·23 (N) [g]	2·88	2·81
		2·49 (S) [g]	3·12	3·02
$Mo_2O_3\{S_2P(OEt)_2\}_4$ [h]	1·64	2·47 (S) [g]	3·19	2·96
		2·54 (S) [c]	3·12	3·02
$Mo_2O_3\{S_2COEt\}_4$ [i]	1·64	2·51 (S) [c]	3·02	3·00
		2·46 (S) [g]	3·20	2·96

[a] Atom *cis*-X is given in parentheses. [b] This work. [c] Mean of 4 atoms. [d] Ref. 8. [e] Ref. 5. [f] Ref. 15. [g] Mean of 2 atoms. [h] Ref. 17. [i] Ref. 16.

The Mo–Mo distance [2·848(1) Å] is indicative of a metal–metal interaction as manifested by the observed diamagnetism of this complex, and as was similarly found in other Mo_2X_2 (X = O or S) complexes,[5] the Mo–Mo–O_t and Mo–Mo–X(*trans*) angles are greater than 90° as predicted by the Nyholm–Gillespie model.[16] This distribution of donor atoms around the molybdenum is not unique to doubly bridged complexes but is also observed in the mono-oxygen-bridged Mo^V complexes of ethylxanthate and diethyl dithiophosphate.[17,18]

Unit Cell.—A stereoscopic view of half the unit cell is presented in Figure 2. The molecules which are related by the 4_1 screw axis are arranged so that the adjacent imidazole rings are nearly parallel to one another at an average distance of *ca.* 3·7 Å. This packing is very efficient and may indicate some van der Waals interaction between rings.[19]

The water of crystallization is hydrogen bonded to both carboxylato-oxygen atoms and free imidazole nitrogen atoms. Water molecule, W(1), forms two equal hydrogen bonds of 2·94 Å to the bonded carboxylato-oxygen atoms of one molecule and another of 2·73 Å to the free imidazole nitrogen, N(3′), on a neighbouring molecule. Water molecule, W(2), is hydrogen bonded to the free carboxylato-oxygen atoms (2·85 Å) of two adjacent molecules related by the two-fold axis of the unit cell. One additional hydrogen bond of 2·71 Å is found between a free carboxylato-oxygen and the nitrogen of the imidazole ring of a neighbouring molecule.

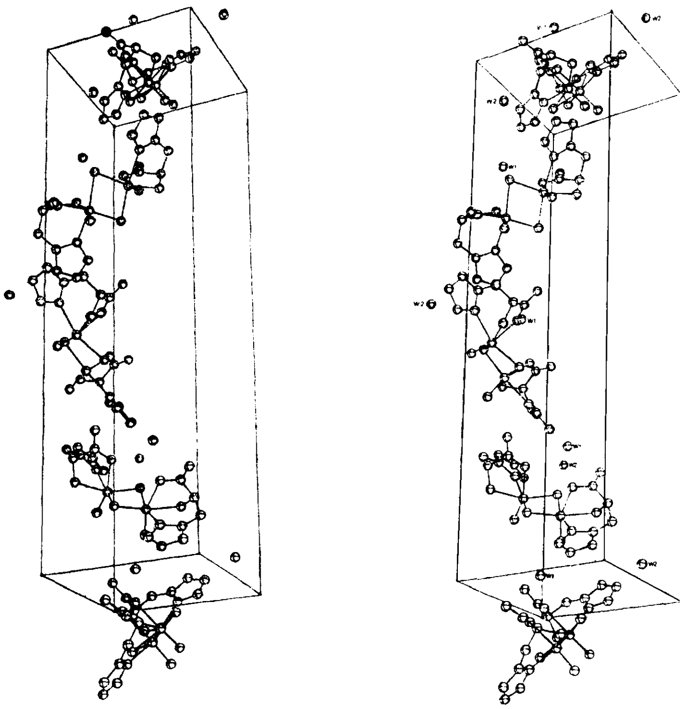

FIGURE 2 Stereoscopic view of the packing of $Mo_2S_2O_2(histidine)_2,1\frac{1}{2}H_2O$ in half the unit cell

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