Supplementary Material Available: Tables of observed and calculated structure factors for all three compounds (27 pages). Ordering information is given on any current masthead page.

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A Quadruply Bonded Dimolybdenum Compound with Bridging Pyrimidinethiol Ligands

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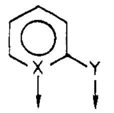
The reaction of dimolybdenum tetraacetate with the sodium salt of 4,6-dimethyl-2-mercaptopyrimidine (Hdmmp) affords $Mo_2(dmmp)_4$. Recrystallization from dichloromethane solution by slow addition of hexane gave mainly acicular crystals of $Mo_2(dmmp)_4 \cdot 2CH_2Cl_2$, but also some rectangular crystals of $Mo_2(dmmp)_4 \cdot CH_2Cl_2$. The structure of one of the latter crystals was determined. The space group was $P2_1/n$, and the unit cell had the following dimensions: $a = 10.927$ (2) Å, $b = 10.660$ (2) Å, $c = 28.149$ (5) Å, $\beta = 101.36$ (2)°, $V = 3214$ (1) Å^3. With $Z = 4$, the formula unit is the asymmetric unit. The structure is of the expected general type. It has a ligand arrangement with two trans S atoms and two trans N atoms on each molybdenum. The Mo–Mo bond length, 2.083 (2) Å, is slightly longer than those in the four other molecules containing quadruply bonded Mo_2^{4+} and ligands of the same general class. Other bond lengths and angles have values that are not surprising in comparison with those in comparable molecules.

Introduction

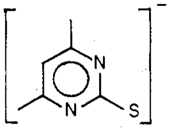
Over the past year and a half, we have discovered a series of new compounds containing $M\overset{4}{-}M'$ bonds with M and M′ being Cr, Mo, or W and including the mixed $Cr\overset{4}{-}Mo$ and $Mo\overset{4}{-}W$ species.[1-8] The ligands in these compounds are all of the type represented schematically in **1**, where the donor atoms X and Y may be C, N, or O, in various combinations. The common feature of all these ligands is the presence of an aromatic or heteroaromatic ring. We shall discuss the detailed nature of these ligands more fully later.



1

These compounds have been especially interesting for the following reasons. (1) The Cr_2 species all have supershort (<1.91 Å) metal-metal bonds. (2) Stable $W\overset{4}{-}W$ compounds became accessible for the first time with such ligands. (3) This type of ligand allows considerable variability in the identity of the donor atoms and the steric properties of the ligand.

We have now succeeded in preparing and structurally characterizing a $M\overset{4}{-}Mo$ compound containing a ligand, **2**, of type **1** in which one of the donor atoms is sulfur. The greater size and polarizability of the S atom, as compared to N and O atoms, aroused our interest in the detailed structural properties of the $M\overset{4}{-}M$ complexes, and we describe here, in full, the nature of the compound formed by this ligand, which is the anion 4,6-dimethyl-2-mercaptidopyrimidine, dmmp, with $Mo\overset{4}{-}Mo$, viz., $Mo_2(dmmp)_4$.



2

Experimental Section

Preparation. All operations were conducted in an atmosphere of nitrogen. 4,6-Dimethyl-2-mercaptopyrimidine (Hdmmp) was purchased from Aldrich Chemical Co. and used as received. The Hdmmp, 1.40 g (10 mmol), was dissolved in 100 mL of absolute ethanol and 0.54 g (10 mmol) of $NaOCH_3$ added. To this mixture was added 1.11 g (2.5 mmol) of $Mo_2(O_2CCH_3)_4$. The heterogeneous reaction mixture immediately turned red; it was stirred at room temperature for 6 h.

The product was separated by filtration, washed with 25 mL of ethanol, and dried under vacuum. It was then dissolved in 50 mL of dichloromethane, to produce a saturated solution, which was filtered through a medium glass frit into a tubular flask. A layer of hexane was gently introduced over the CH_2Cl_2 solution, and over a period of three days, as the two solvents mixed by diffusion, small acicular crystals were formed. They were collected on a filter and dried by brief pumping at room temperature. Anal. Calcd for $C_{26}H_{32}N_8S_4Cl_4Mo_2$: C, 34.06; H, 3.06; N, 12.22. Found: C, 34.2; H, 3.25; N, 12.0.

When the crystalline product was examined under a microscope it was found that in addition to the predominant acicular crystals there were also a few rectangular ones. One of the latter was selected for X-ray analysis, and it was found from the structure that the rectangular crystals differ in their solvent content from the acicular ones, having the composition $Mo_2(dmmp)_4 \cdot CH_2Cl_2$.

X-ray Crystallography.[9] Preliminary examination and systematic absences showed the space group to be $P2_1/n$ with unit cell dimensions $a = 10.927$ (2) Å, $b = 10.660$ (2) Å, $c = 28.149$ (5) Å, and $\beta = 101.36$ (2)°, $Z = 4$. With a unit cell volume of 3214 (1) Å, the asymmetric

Table I. Positional and Thermal Parameters and Their Estimated Standard Deviations[a]

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo(1)	0.7258 (1)	0.4230 (1)	0.40476 (5)	2.47 (6)	2.15 (6)	2.59 (6)	0.03 (6)	0.53 (5)	−0.18 (7)
Mo(2)	0.7321 (1)	0.4386 (1)	0.33153 (5)	2.00 (6)	2.34 (6)	2.52 (6)	−0.15 (6)	0.10 (5)	0.33 (6)
S(1)	0.5137 (4)	0.3383 (5)	0.3961 (2)	3.0 (2)	3.6 (2)	3.3 (2)	−0.4 (2)	1.1 (2)	−0.3 (2)
S(2)	0.6530 (5)	0.6530 (5)	0.3190 (2)	3.4 (2)	3.2 (2)	5.2 (3)	0.2 (2)	0.0 (2)	1.3 (2)
S(3)	0.8108 (4)	0.2271 (4)	0.3206 (2)	3.5 (2)	3.0 (2)	2.5 (2)	0.2 (2)	0.9 (2)	−0.2 (2)
S(4)	0.9359 (4)	0.5014 (5)	0.4370 (2)	2.7 (2)	3.7 (2)	2.9 (2)	−0.5 (2)	0.3 (2)	−0.6 (2)
N(1)	0.542 (1)	0.374 (1)	0.3036 (5)	2.1 (6)	2.5 (6)	5.3 (8)	−0.3 (6)	0.2 (6)	0.3 (6)
N(2)	0.650 (1)	0.614 (1)	0.4126 (5)	2.4 (6)	3.5 (7)	3.5 (7)	−0.0 (6)	0.3 (5)	−0.2 (6)
N(3)	0.793 (1)	0.230 (1)	0.4135 (4)	2.7 (6)	2.1 (6)	2.1 (5)	0.5 (5)	0.7 (5)	0.5 (5)
N(4)	0.926 (1)	0.506 (1)	0.3410 (4)	2.6 (6)	2.7 (6)	1.7 (5)	−0.0 (6)	0.2 (5)	0.7 (5)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
Cl(1)	0.1352 (7)	0.2113 (8)	0.4346 (3)	9.3 (2)	C(25)	0.663 (2)	0.561 (2)	0.4986 (7)	4.8 (5)
Cl(2)	0.1862 (8)	−0.0086 (9)	0.3862 (3)	10.8 (3)	C(26)	0.516 (2)	0.985 (3)	0.4229 (9)	7.5 (7)
N(12)	0.344 (1)	0.302 (1)	0.3190 (5)	3.0 (3)	C(31)	0.823 (1)	0.160 (1)	0.3762 (5)	2.2 (3)
N(22)	0.581 (2)	0.810 (2)	0.3781 (6)	4.8 (4)	C(32)	0.867 (2)	−0.018 (2)	0.4230 (6)	3.2 (4)
N(32)	0.856 (1)	0.040 (1)	0.3796 (5)	3.1 (3)	C(33)	0.845 (2)	0.044 (2)	0.4643 (6)	2.8 (4)
N(42)	0.115 (1)	0.567 (1)	0.3954 (5)	3.3 (3)	C(34)	0.805 (1)	0.173 (2)	0.4574 (6)	2.6 (3)
C(11)	0.461 (2)	0.336 (2)	0.3327 (6)	2.8 (4)	C(35)	0.775 (2)	0.244 (2)	0.4997 (7)	3.9 (4)
C(12)	0.299 (2)	0.303 (2)	0.2699 (6)	3.6 (4)	C(36)	0.909 (2)	-0.152 (2)	0.4253 (7)	3.8 (4)
C(13)	0.370 (2)	0.342 (2)	0.2354 (6)	3.6 (4)	C(41)	0.996 (1)	0.528 (2)	0.3869 (6)	2.4 (3)
C(14)	0.492 (2)	0.372 (2)	0.2565 (6)	3.2 (4)	C(42)	1.170 (2)	0.580 (2)	0.3566 (6)	3.2 (4)
C(15)	0.581 (2)	0.405 (2)	0.2236 (7)	4.1 (4)	C(43)	1.106 (2)	0.555 (2)	0.3089 (6)	3.1 (4)
C(16)	0.161 (2)	0.261 (2)	0.2555 (7)	4.6 (5)	C(44)	0.982 (2)	0.522 (2)	0.3023 (6)	2.9 (4)
C(21)	0.626 (2)	0.694 (2)	0.3733 (6)	3.0 (4)	C(45)	0.908 (2)	0.498 (2)	0.2531 (6)	3.7 (4)
C(22)	0.565 (2)	0.846 (2)	0.4205 (8)	5.8 (6)	C(46)	1.305 (2)	0.621 (2)	0.3672 (7)	4.5 (5)
C(23)	0.586 (2)	0.774 (2)	0.4608 (7)	5.0 (5)	C(100)	0.158 (3)	0.140 (3)	0.3725 (10)	9.3 (8)
C(24)	0.632 (2)	0.652 (2)	0.4580 (7)	3.6 (4)					

[a] The form of the anisotropic thermal parameter is $\exp[-{}^1/_4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table II. Bond Distances (Å) for $Mo_2(dmmp)_4 \cdot CH_2Cl_2$

Mo(1)-Mo(2)	2.083 (2)	Mo(2)-S(2)	2.445 (4)
Mo(1)-S(1)	2.454 (4)	-S(3)	2.453 (4)
-S(4)	2.444 (4)	-N(1)	2.19 (1)
-N(2)	2.22 (1)	-N(4)	2.20 (1)
-N(3)	2.18 (1)	S(2)-C(21)	1.67 (1)
S(1)-C(11)	1.77 (1)	N(2)-C(21)	1.39 (2)
N(1)-C(11)	1.38 (1)	C(21)-N(22)	1.35 (2)
C(11)-N(12)	1.31 (2)	N(22)-C(22)	1.29 (2)
N(12)-C(12)	1.37 (2)	C(22)-C(23)	1.34 (2)
C(12)-C(13)	1.42 (2)	C(22)-C(26)	1.59 (3)
C(12)-C(16)	1.55 (2)	C(23)-C(24)	1.40 (2)
C(13)-C(14)	1.39 (2)	C(24)-N(4)	1.39 (2)
C(14)-N(1)	1.33 (2)	C(24)-C(25)	1.49 (2)
C(14)-C(15)	1.52 (2)	S(3)-C(31)	1.70 (1)
S(4)-C(41)	1.69 (1)	N(3)-C(31)	1.37 (2)
N(4)-C(41)	1.39 (2)	C(31)-N(32)	1.33 (2)
C(41)-N(42)	1.34 (2)	N(32)-C(32)	1.35 (2)
N(42)-C(42)	1.35 (2)	C(32)-C(33)	1.40 (2)
C(42)-C(43)	1.41 (2)	C(32)-C(36)	1.50 (2)
C(42)-C(46)	1.51 (2)	C(33)-C(34)	1.44 (2)
C(43)-C(44)	1.38 (2)	C(34)-N(3)	1.36 (1)
C(44)-N(4)	1.36 (2)	C(34)-C(35)	1.50 (2)
C(44)-C(45)	1.48 (2)	C(100)-Cl(2)	1.65 (3)
C(100)-Cl(1)	1.96 (2)		

unit contains one $Mo_2(dmmp)_4$ molecule and one dichloromethane molecule. By use of 2153 unique data with $I > 3\sigma(I)$, the structure was solved by direct methods and refined to convergence with anisotropic thermal parameters for the Mo, S, and coordinated N atoms and isotropic thermal parameters for the remaining nonhydrogen atoms. The final residuals were $R_1 = 0.065$ and $R_2 = 0.085$ with the esd of an observation of unit weight equal to 1.60. A table of structure factors is available as supplementary material.

Results

The atomic positional and thermal vibration parameters are listed in Table I. The structure of the $Mo_2(dmmp)_4$ molecule and the atom numbering are shown in Figure 1, and the bond distances and angles are listed in Tables II and III, respectively. The molecules are packed, along with the CH_2Cl_2 molecules, so as to make only van der Waals contacts with one another. There is, as in all other compounds of this class,[1-8] no axial coordination of the metal atoms.

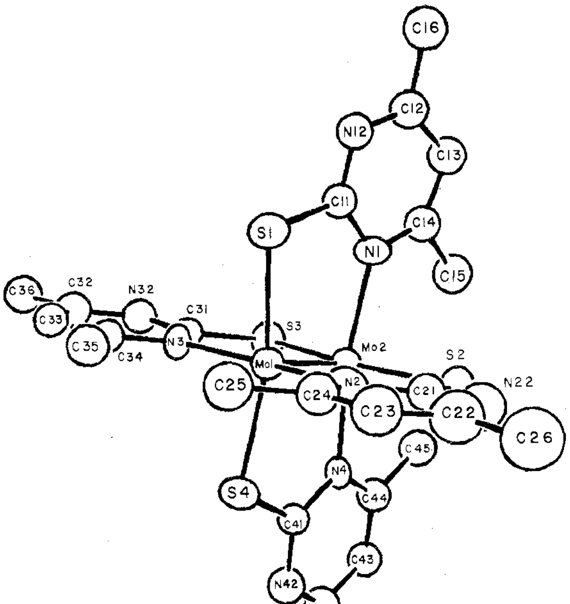


Figure 1. ORTEP projection of the molecule showing the atomic numbering scheme. Atomic sizes are proportional to their thermal vibration amplitudes.

The arrangement of the ligands around the Mo_2 unit is such that on each metal atom the S atoms are trans to each other and so, of course, are the N atoms. The entire molecular structure, therefore, has virtual D_{2d} symmetry. The Mo–Mo distance, 2.083 (2) Å, is typical for structures of this type, and the Mo–S bond lengths, averaging 2.449 (5) Å, are within the

Table III. Bond Angles (deg) for $Mo_2(dmmp)_4 \cdot CH_2Cl_2$

Mo(2)–Mo(1)–S(1)	98.4 (1)	Mo(1)–Mo(2)–S(2)	97.9 (1)
–S(4)	97.4 (1)	–S(3)	97.6 (1)
–N(2)	96.4 (3)	–N(1)	96.6 (3)
–N(3)	96.3 (3)	–N(4)	97.2 (3)
S(1)–Mo(1)–S(4)	164.2 (1)	S(2)–Mo(2)–S(3)	164.5 (1)
–N(2)	89.0 (3)	–N(1)	87.5 (3)
–N(3)	87.5 (3)	–N(4)	91.1 (3)
S(4)–Mo(1)–N(2)	89.6 (3)	S(3)–Mo(2)–N(1)	90.0 (3)
–N(3)	90.4 (3)	–N(4)	87.8 (3)
N(2)–Mo(1)–N(3)	167.2 (4)	N(1)–Mo(2)–N(4)	166.1 (4)
Mo(1)–S(1)–C(11)	102.7 (5)	Mo(2)–S(2)–C(21)	103.6 (5)
Mo(2)–N(1)–C(11)	124 (1)	Mo(1)–N(2)–C(21)	120.1 (9)
–C(14)	122 (1)	–C(24)	119.1 (9)
C(11)–N(1)–C(14)	114 (1)	C(21)–N(2)–C(24)	121 (1)
S(1)–C(11)–N(1)	118 (1)	S(2)–C(21)–N(2)	122 (1)
–N(12)	114 (1)	–N(22)	118 (1)
N(1)–C(11)–N(12)	128 (1)	N(2)–C(21)–N(22)	120 (1)
C(11)–N(12)–C(12)	115 (1)	C(21)–N(22)–C(22)	119 (2)
N(12)–C(12)–C(13)	124 (1)	N(22)–C(22)–C(23)	125 (2)
–C(16)	113 (1)	–C(26)	115 (2)
C(13)–C(12)–C(16)	123 (1)	C(23)–C(22)–C(26)	120 (2)
C(12)–C(13)–C(14)	112 (1)	C(22)–C(23)–C(24)	119 (2)
C(13)–C(14)–N(1)	127 (1)	C(23)–C(24)–N(2)	116 (1)
–C(15)	118 (1)	–C(25)	126 (1)
N(1)–C(14)–C(15)	115 (1)	N(2)–C(24)–C(25)	117 (1)
Mo(1)–S(4)–C(41)	103.9 (5)	Mo(2)–S(3)–C(31)	103.5 (5)
Mo(2)–N(4)–C(41)	120.8 (8)	Mo(2)–N(3)–C(31)	123.3 (8)
–C(44)	121.2 (9)	–C(34)	119.5 (8)
C(41)–N(4)–C(44)	118 (1)	C(31)–N(3)–C(34)	117 (1)
S(4)–C(41)–N(4)	121 (1)	S(3)–C(31)–N(3)	119 (1)
–N(42)	115 (1)	–N(32)	116 (1)
N(4)–C(41)–N(42)	124 (1)	N(3)–C(31)–N(32)	124 (1)
C(41)–N(42)–C(42)	117 (1)	C(31)–N(32)–C(32)	119 (1)
N(42)–C(42)–C(43)	122 (1)	N(32)–C(32)–C(33)	122 (1)
–C(46)	116 (1)	–C(36)	116 (1)
C(43)–C(42)–C(46)	121 (1)	C(33)–C(32)–C(36)	121 (1)
C(42)–C(43)–C(44)	118 (1)	C(32)–C(33)–C(34)	116 (1)
C(43)–C(44)–N(4)	121 (1)	C(33)–C(34)–N(3)	121 (1)
–C(45)	121 (1)	–C(35)	119 (1)
N(4)–C(44)–C(45)	118 (1)	N(3)–C(34)–C(35)	120 (1)
Cl(1)–C(100)–Cl(2)	102 (1)		

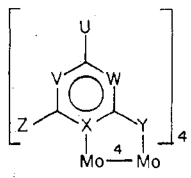
range of those reported for several other Mo_2L_4 compounds containing sulfur ligands.[10,11]

Discussion

It is of interest to compare the key structural features of the $Mo_2(dmmp)_4$ molecule with those of other dimolybdenum molecules with ligands of type **1**. The pertinent results for five molecules, including the present one, are listed in Table IV.

It can be seen that this molecule resembles all others having N in the X position in having the D_{2d} arrangement of the ligands. It also differs from all of the others in certain ways. First, it has the longest Mo–Mo distance, although it is still shorter than those, ≥2.09 Å, found in carboxylato-bridged molecules.[12] It resembles one of the other compounds but differs from three of them in having nearly equal Mo–Mo–X and Mo–Mo–Y angles and differs from all of the other four in having the sum of these angles considerably greater than in any previous case. These larger angles are clearly the result of the larger X···Y distance here than in any other case, which is, in turn, attributable to the C–S distance being considerably longer than C–N or C–O distances. It is possible that the slightly longer Mo–Mo distance can also be attributed to the greater X···Y separation since the Mo–Mo–X(Y) angles would have to be even greater to accommodate a shorter Mo–Mo bond, and each of these angles is already larger than any single such angle previously seen in molecules of this type with Mo$\overset{4}{-}$Mo bonds. However, larger Mo–Mo–X angles are found in species such as $Mo_2Cl_8^{4-}$ (where they exceed 100°), and even in molecules with ligands of type **1**, if M = Cr, angles of 98–100° are found. Thus, it is doubtful if a simple steric explanation for the longer Mo–Mo distance will suffice.

Table IV. Mean Distances (Å) and Angles (deg) in Quadruply Bonded Dimolybdenum Complexes with Aromatic Ligands[a]



	compd				
	1	2	3	4	5
U	H	H	H	Me	Me
V	HC	HC	HC	N	HC
W	HC	HC	HC	HC	N
X	C	N	N	N	N
Y	OMe	O	NH	O	S
Z	OMe	Me	Me	Me	Me
symmetry	C_{2h}	D_{2d}	D_{2d}	D_{2d}	D_{2d}
Mo–Mo	2.064 (1)	2.065 (1)	2.070 (1)	2.072 (1)	2.083 (2)
Mo–C	2.16 (2)				
Mo–N(X)		2.167 (12)	2.180 (8)	2.157 (4)	2.20 (2)
Mo–N(Y)			2.136 (9)		
Mo–O	2.27 (2)	2.086 (6)		2.077 (4)	
Mo–S					2.449 (5)
Mo–Mo–X	90.7 (5)	90.2 (4)	92.9 (4)	90.0 (2)	96.6 (3)
Mo–Mo–Y	96.1 (4)	95.3 (1)	93.1 (2)	95.5 (2)	97.8 (3)
ref	1	5	6	7	this work

[a] Average values are given for all except the Mo–Mo distance. Figures in parentheses are mean deviations of individual values from the average value.

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Registry No. $Mo_2(dmmp)_4 \cdot CH_2Cl_2$, 69401-63-4; $Mo_2(O_2CCH_3)_4$, 14221-06-8.

Supplementary Material Available: A table of observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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