are 1·394 (10) Å for N(3)—C(16), 1·328 (8) Å for N(2)—N(3) and 1·274 (9) Å for C(15)=N(2).

The molecular packing is mainly determined by an intermolecular hydrogen bond between Cl(1) $(1-x, -y, -z)$ and N(3) [3·409 (8) Å] of two molecules related by an inversion centre. There is also a contact (< 3·65 Å) between the C=N—N fragment and the pyridine ring $(1-x, -y, -z)$ [C(12)···C(15) 3·49 (1), C(13)···C(15) 3·53 (1), C(10)···N(2) 3·62 (1) Å] and such an arrangement, depicted in Fig. 2, may be responsible for the pyridine twist.

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Structure of Di-μ-methoxo-bis[dichlorodimethoxomolybdenum(V)]*

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Abstract. $[Mo_2Cl_4(OCH_3)_6]$, $M_r = 519·9$, monoclinic, $P2_1/n$, $a = 12·029$ (2), $b = 9·615$ (2), $c = 7·240$ (1) Å, $\beta = 95·60$ (1)°, $V = 833·38$ Å^3, $Z = 2$, $D_x = 2·074$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 17·77$ mm^{-1}, $F(000) = 508$, final $R = 0·056$ for 1118 reflections. The dimeric molecules, located on crystallographic inversion centres, consist of two edge-sharing octahedra. Each Mo atom is coordinated by a pair of *cis* terminal Cl atoms [2·391 (2) and 2·393 (2) Å], a pair of *trans* terminal methoxo O atoms [1·811 (5) and 1·801 (6) Å], and a pair of *cis* bridging methoxo O atoms [2·027 (5) and 2·029 (5) Å]. The Mo–Mo distance is 2·733 (1) Å.

Introduction. The crystal structure analysis of the title complex forms part of our research on Mo^V complexes with O- and N-donor ligands. The attempts to prepare such complexes with different ligands, *e.g.* methionine or morpholine in methanol as solvent, always resulted in the binuclear chloro(methoxo) complex $Mo_2Cl_4(OCH_3)_6$ (Kamenar, Korpar-Čolig, Penavić & Cindrić, 1988*b*). It was of interest to determine the crystal structure of this complex in order to establish the type of bridging between the two Mo atoms, the strength of the Mo^V–Mo^V interaction, as well as to compare our findings with earlier works (Cotton, 1987).

Experimental. In the attempt to prepare the complex of Mo^V with methionine, by mixing $MoCl_5$ with methionine in dry methanol, the title compound was isolated in low yield as a first-reaction product. Deep-red-brown crystals sensitive to air and moisture were separated by filtration in a dry box under dry nitrogen. Alternatively, the same complex can be obtained by the procedure given by Funk, Hesselbarth & Schmeil (1962).

* Reported at the 11th European Crystallographic Meeting, Vienna 1988, but with the space group assigned as $P2_1/c$ (Kamenar, Korpar-Čolig, Penavić & Cindrić, 1988*a*).

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Table 1. *Fractional positional parameters* ($\times 10^4$; $\times 10^5$ *for* Mo) *and equivalent isotropic temperature factors* ($\times 10^3$; $\times 10^4$ *for* Mo) *for non*-H *atoms*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq} (Å^2)
Mo	2740 (5)	12705 (6)	7576 (8)	306 (2)
Cl(1)	1873 (2)	2757 (2)	887 (4)	52 (0)
Cl(2)	−648 (2)	2940 (2)	2528 (4)	53 (1)
O(1)	−1137 (4)	110 (5)	671 (8)	34 (1)
O(2)	852 (5)	558 (6)	2960 (8)	35 (1)
O(3)	−233 (5)	2250 (6)	−1272 (8)	39 (1)
C(1)	−2288 (7)	456 (11)	674 (16)	50 (2)
C(2)	1361 (10)	781 (13)	4801 (15)	60 (3)
C(3)	−313 (11)	3573 (9)	−2138 (16)	57 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

Primed atoms are related to unprimed atoms by a centre of symmetry at (0,0,0).

Mo—Mo	2·733 (1)	Mo—O(2)	1·811 (5)
Mo—Cl(1)	2·391 (2)	Mo—O(3)	1·801 (6)
Mo—Cl(2)	2·393 (2)	O(1)—C(1)	1·424 (10)
Mo—O(1)	2·027 (5)	O(2)—C(2)	1·428 (11)
Mo—O(1′)	2·029 (5)	O(3)—C(3)	1·417 (10)
Cl(1)—Mo—Cl(2)	89·5 (1)	O(1)—Mo—O(3)	92·7 (3)
Cl(1)—Mo—O(1)	176·6 (2)	O(2)—Mo—O(3)	170·7 (2)
Cl(1)—Mo—O(2)	87·4 (2)	O(1)—Mo—O(1′)	95·3 (3)
Cl(1)—Mo—O(3)	85·8 (2)	Mo—O(1)—Mo′	84·7 (3)
Cl(2)—Mo—O(1)	87·4 (2)	Mo—O(1)—C(1)	133·1 (5)
Cl(2)—Mo—O(2)	86·6 (2)	Mo—O(2)—C(2)	149·1 (6)
Cl(2)—Mo—O(3)	87·1 (2)	Mo—O(3)—C(3)	146·4 (6)
O(1)—Mo—O(2)	93·8 (2)		

Crystal of dimensions 0·15 × 0·17 × 0·10 mm protected from decomposition by a thin layer of epoxy resin was used for data collection on a Philips PW 1100 automatic diffractometer with Cu $K\alpha$ radiation. Cell dimensions and their e.s.d.'s obtained by least-squares refinement of 16 reflections with θ between 13 and 26°. Total of 1289 reflections measured using ω–2θ scan mode, $5 < 2\theta < 70°$, in the range h: −14 to 14, k: 0 to 11, l: 0 to 8. Intensities of three standard reflections ($\bar{3}41$, $\bar{3}20$, 012) measured every 2 h showed no significant intensity decay. Corrections for Lorentz and polarization effects but not for absorption were applied. 1118 independent reflections with $I > 5\sigma(I)$ were used for structure solution and refinement. Position of Mo atom located from Patterson and all non-H atoms from successive Fourier syntheses. H atoms were positioned on geometrical grounds (C—H 1·08 Å) and included in the structure-factor calculations. Final full-matrix least-squares refinement included anisotropic thermal parameters for non-H atoms, one common thermal parameter for H atoms, U = 0·135 (22) Å^2 (final value); 83 refined parameters. Final $R = 0{\cdot}056$, $wR = 0{\cdot}054$, function minimized $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2|F_o| + 0{\cdot}0056|F_o|^2]$, max. shift/e.s.d. in final cycles = 0·002. Max. and min. heights in final difference Fourier map: 2·19 (close to Mo atom) and −1·43 e Å^{-3}. Scattering factors and anomalous-scattering corrections for Mo were taken from *International Tables for X-ray Crystallography* (1974), computations performed with *SHELX*76 (Sheldrick, 1976) and figure drawn with *ORTEP* (Johnson, 1971). All calculations were performed on the Univac 1110 computer of the Zagreb University Computing Centre, SRCE.

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond lengths and angles in Table 2.

The crystal structure of the dimeric $Mo_2Cl_4(OCH_3)_6$ contains isolated complex molecules situated on crystallographic inversion centres. Each individual molecule consists of two edge-sharing distorted octahedra as presented in Fig. 1. The octahedral coordination about each Mo atom consists of two *cis* Cl atoms and two *cis* bridging methoxo O atoms in the equatorial plane, and two *trans* terminal methoxo O atoms in the axial positions. The observed coordination is similar to that established for the structure of the $W_2Cl_4(OCH_3)_4(HOCH_3)_2$ complex with W in the oxidation state +4 and therefore with two methanol molecules instead of two methoxo ligands (Anderson, Cotton, DeMarco, Fang, Ilsley, Kolthammer & Walton, 1981), and to that in $W_2Cl_4(OCH_2CH_3)_6$ (Cotton, DeMarco, Kolthammer & Walton, 1981) and in $Mo_2Cl_4(O\text{-}i\text{-}Pr)_6$ or $Mo_2Br_4(O\text{-}i\text{-}Pr)_6$ (Chisholm, Kirkpatrick & Huffman, 1981). The octahedral coordination about Mo is slightly distorted; the angles at Mo range from 85·8 (2) to 93·8 (2)°. The greatest deviation from linearity for a *trans* angle is between the *trans* pair of terminal methoxo ligands (9·3°). This is certainly the result of steric hindrance

* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52253 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

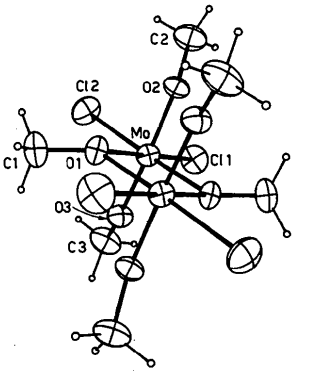

Fig. 1. A perspective view of the molecule showing atom numbering.

between the ligands coordinated to two centrosymmetrically related Mo atoms. A similar but consequently greater deviation (mean value 12°) was found in the structure of the analogous *i*-propoxo complex (Chisholm *et al.*, 1981).

The Mo_2O_2 bridging system is almost square planar, the internal angles O(1)—Mo—O(1′) and Mo—O(1)—Mo′ being 95·3 (3) and 84·7 (3)°, respectively. The lateral distances Mo—O(1) and Mo—O(1′) are equal within experimental error [2·027 (5) and 2·029 (5) Å]. The bond lengths Mo—Cl(1) of 2·391 (2) and Mo—Cl(2) of 2·393 (2) Å agree with the values of 2·317 (3) and 2·360 (4) Å found in $Mo_2Cl_4(OPh)_6$ (Kamenar & Penavić, 1977) and agree even better with those of 2·421 (2) and 2·416 (1) Å in the above mentioned $Mo_2Cl_4(O\text{-}i\text{-}Pr)_6$. However, all these Mo—Cl bond lengths are considerably longer than molybdenum—terminal-chlorine bond lengths of 2·26 (2) Å in $MoCl_3O$ (Drew & Tomkins, 1970), and of 2·24 (3) Å in Mo_2Cl_{10} (Sands & Zalkin, 1959). This bond lengthening may indicate the *trans* influence of the alkoxo ligands. The bond lengths between Mo and terminal methoxo O atoms [Mo—O(2) of 1·811 (5) and Mo—O(3) of 1·801 (5) Å] are short and suggest Mo=O double bonds (Schröder, 1975; Manojlović-Muir & Muir, 1972), indicating the existence of additional π bonding between the metal and alkoxo ligands (Huffman, Molloy, Marsella & Caulton, 1980). The same shortening of the Mo—O*R* bonds was found in the Mo—phenoxo (Kamenar & Penavić, 1977) and Mo—propoxo complexes (Chisholm *et al.*, 1981); in both structures such Mo—O bond lengths have a mean value of 1·81 Å. The Mo—Mo bond is also of interest because in the Mo complexes it varies with the metal oxidation state, the nature of the bridging ligands, as well as with the steric and electronic properties of the terminal ligands. In this structure the Mo—Mo bond length of 2·733 (1) Å is of the d^1—d^1 type and corresponds to a single bond (Cotton, 1977). It is the same as already found in the above mentioned analogous Mo(chloro)(propoxo) [2·731 (1) Å] and Mo(bromo)-(propoxo) [2·739 (1) Å] complexes.

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Structure of Tris(thioacetyltrifluoroacetonato)rhodium(III)

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Abstract. $[Rh(C_5H_4F_3OS)_3]$, $M_r = 610{\cdot}340$, monoclinic, $P2_1/n$, $a = 9{\cdot}065$ (2), $b = 13{\cdot}302$ (2), $c = 18{\cdot}360$ (2) Å, $\beta = 93{\cdot}08$ (2)°, $V = 2210{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}833$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 22{\cdot}2$ cm^{-1}, $F(000) = 1200$, $T = 294$ K, $R = 0{\cdot}043$ for 2206 unique observed reflections. The octahedral

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