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Molecular and Inner Complex Compounds of Dioxomolybdenum(VI) with Disubstituted Salicydenealcoholimines: Crystal Structure of 1 : 1 Dioxo(3,5-Dibromosalicylidenemonoethanoliminato)molybdenum(VI) Solvate with Methanol [$MoO_2(L^1) \cdot MeOH$] ($L^1 = C_9H_7Br_2NO_2$)

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Abstract—Complexes of dioxomolybdenum(VI) of the molecular ($MoO_2Cl_2 \cdot 2H_2L$) and inner complex ([$MoO_2L \cdot Solv$]) types are synthesized (H_2L are azomethines, derivatives of disubstituted R^1,R^2-salicylaldehydes (R^1, R^2 = 3.5-Br_2; R^1 = 3-MeO, R^2 = 5-Br) and monoethanolamine; Solv is a methanol, dimethylformamide, pyridine, or α-picoline molecule). The *cis*-octahedral structure of the complexes is concluded on the basis of the IR spectroscopic data. In the molecular compounds, the ligands are coordinated via the O atom of the carbonyl group of the H_2L tautomeric form. In the inner complex compounds, the ligands are coordinated in the deprotonated benzoid form. The structure of [$MoO_2(L^1) \cdot MeOH$] (**I**) (where L^1 is $C_9H_7Br_2NO_2$) is determined by X-ray diffraction analysis (CIF file CCDC no. 1898088). In the mononuclear molecule of compound **I**, the Mo atom has the octahedral coordination by two oxo ligands, two oxygen atoms, the nitrogen atom of the tridentate bis(chelate) two-charge ligand $(L^1)^{2-}$, and the O atom of the methanol molecule. The neutral N(1) and O(1) atoms of the L^1 and MeOH ligands, respectively, are arranged in the *trans* positions to the O(oxo) ligands. The Mo–N(1) (2.265 Å) and Mo–O(1) (2.372 Å) bonds are substantially elongated due to the structural manifestation of the *trans* effect of the multiply bonded oxo ligands. The intermolecular hydrogen bonds (MeOH)O–H···O(oxo) join the molecules into supramolecular 1D chains.

Keywords: synthesis, IR spectroscopy, X-ray diffraction analysis, dioxomolybdenum(VI) complexes, salicylidenealcoholiminate ligands

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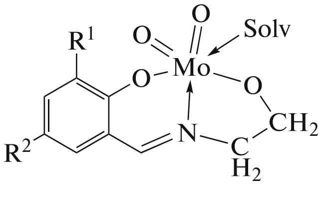
INTRODUCTION

Salicylidenealcoholimines (H_2L) are potential tridentate dibasic ligands, which react with the *d*-metal salts to form inner complex compounds (ICC) of several types with the monomeric ([ML_n], [$M(HL)_n$], and di-[ML_n]$_2$) or oligomeric ([ML_n]$_m$) structures and exhibit the bi- or tridentate function [1–5]. The monomeric structures of the complexes MoO_2L (coordination number 5) and $MoO_2L \cdot Solv$ (Solv is a solvent molecule) (coordination number 6) were concluded on the basis of the data of IR and NMR spectroscopy [6–9]. Later we found from the X-ray diffraction (XRD) data [1, 2] that the solvated dioxomolybdenum(VI) complexes with substituted salicylidenemonoethanolamines (R^1, R^2 = 3.5-Cl_2 and R^1 = 3-MeO, R^2 = H) have the *cis*-octahedral structure with the ligands coordinated via the azomethine nitrogen atom and two oxygen atoms of the deprotonated hydroxo groups of the aldehyde and alcohol fragments. The sixth vertex in the octahedron is occupied by the donor atom of the solvent molecule (Solv is MeOH).



The molecules in the ICC structures are joined in pairs into centrosymmetrical pseudodimers by nearly linear intermolecular hydrogen bonds. Similar conclusions were made [10, 11] on the basis of the XRD data for a series of the ICC of dioxomolybdenum(VI) with monosubstituted salicylidenealcoholimines.

Complexes of the molecular type (adducts) $MX_n \cdot mH_2L$ (X^- is anion) in which the H_2L ligands are coordinated in the neutral form are less known. Continuing the concepts of the structures and properties of the dioxomolybdenum(VI) complexes with salicylidenealcoholimines, we synthesized the molecular compounds (MC) and ICC of dioxomolybdenum(VI) with disubstituted (R^1, R^2 = 3,5-Br_2 (H_2L^1) and R^1 = 3-MeO, R^2 = 5-Br (H_2L^2)) salicylidenemonoethanolimines and studied them by IR spectroscopy. According to the elemental analysis data, the synthesized compounds have the compositions $MoO_2Cl_2 \cdot 2H_2L^{1,2}$ and $[MoO_2L^{1,2} \cdot Solv]$ (Solv is a methanol, DMF, pyridine, and α-picoline (α-Pic) molecule).

The structure of the complex $[MoO_2(L^1) \cdot MeOH]$ (**I**) ($L^1 = C_9H_7Br_2NO_2$) was determined by the XRD method.

EXPERIMENTAL

The initial molybdenum dioxide was synthesized using the commonly known procedure by the reduction of molybdenum trioxide (reagent grade) with hydrogen at 400°C. The reagents and solvents purchased from Lancaster, Mark, and Fluka were used.

Synthesis of MoO_2Cl_2 was carried out by the chlorination of powdered MoO_2 at 350°C [12]. The MoO_2Cl_2 formed during the reaction was carried away from the hot zone and condensed in the cold part of the reactor as pale yellow flakes. An excess chlorine was removed by purging molybdenum dioxochloride with dry argon on weak heating (40–50°C) until the reaction to free chlorine became negative. Since the pressure of the initial molybdenum dioxide vapors is very low, the formed MoO_2Cl_2 needed no additional purification.

Molybdenyl acetylacetonate $MoO_2(Acac)_2$ was synthesized using the described procedure [13].

Syntheses of H_2L^1 and H_2L^2 were carried out by the condensation of 3,5-dibromo- and 3-methoxy-5-bromosalicylaldehyde and monoethanolamine in boiling ethanol. After recrystallization from ethanol, they represented lemon- (HL^1) or yellow-colored (HL^2) needles with mp = 147–148 and 124–125°C, respectively.

For $C_9H_9Br_2NO_2$ (HL^1)

Anal. calcd., %	C, 33.47	H, 2.81	N, 4.34
Found, %	C, 33.20	H, 2.98	N, 4.15

For $C_9H_9Br_2NO_2$ (HL^2)

Anal. calcd., %	C, 43.81	H, 4.41	N, 5.11
Found, %	C, 43.67	H, 4.64	N, 4.93

IR spectra for HL^1 (ν, cm^{-1}): 3310 br, 1652, 1610, 1512, 1350, 1270, 1215, 1158, 1090, 1070, 1030, 908, 870, 770, 748, 630, 580, 495, 447; for HL^2 (ν, cm^{-1}): 3400 br, 1650, 1598, 1520, 1412, 1344, 1240, 1210, 1100, 1065, 1040, 1017, 965, 898, 870, 850, 780, 763, 722, 665, 570, 552, 415.

Syntheses of MC $MoO_2Cl_2 \cdot 2H_2L^{1,2}$ were carried out by the direct reaction of MoO_2Cl_2 with salicylideneimines similarly to the described procedure [14] in low-polarity solvents. In diethyl ether the complexes were formed in an almost quantitative yield. A solution of the corresponding azomethine (0.002 mol) in benzene was added dropwise with magnetic stirring to a solution of molybdenum dioxodichloride (0.199 g, 0.001 mol) in anhydrous diethyl ether (20 mL). The formed precipitate of the complex was separated on the Schott filter equipped with the drying system, washed with ether, and dried in a dry argon flow.

Syntheses of ICC $[MoO_2L^{1,2} \cdot MeOH]$ were carried out by the method of ligand exchange between molybdenyl acetylacetonate and azomethines [1, 2]. A methanol solution of azomethine (0.001 mol) was added dropwise to a hot solution of $MoO_2(Acac)_2$ (0.326 g, 0.001 mol) in methanol (20 mL), and the reaction mixture was refluxed for 10–15 min and left to stay at room temperature for crystallization. The precipitated crystals of the complexes were filtered off, washed with cold methanol, and dried first in a dry air flow and then in a vacuum desiccator over $CaCl_2$. The yields of the complexes were ~90%. The corresponding solvate complexes $[MoO_2L^{1,2} \cdot Py]$ and $[MoO\ L^{1,2} \cdot α\text{-Pic}]$ were formed after the recrystallization of the $[MoO_2L^{1,2} \cdot MeOH]$ complexes from pyridine or α-picoline. The $[MoO_2L^2 \cdot DMF]$ complex was obtained during the synthesis in methanol with the addition of DMF (2 mL) to the reaction mixture. In all cases, the yield of the complexes was at least 90%.

The used organic solvents were thoroughly purified and dehydrated using commonly accepted procedures [15].

Elemental analyses of the complexes to the contents of molybdenum and chlorine were conducted using standard procedures [14]. Analyses of the compounds to the contents of carbon, hydrogen, and nitrogen were carried out on a Carlo-Erba 1106 Elemental Analyzer CHN analyzer. The electric conductance of solutions of the complexes was measured on a conductometer with an alternating current frequency of 1000 Hz using the bridge scheme in a temperature-maintained cell with platinized electrodes.

Table 1. Bond lengths (*d*) and bond angles (ω) in the structure of compound **I**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Mo(1)–O(4)	1.691(5)	Mo(1)–O(2)	1.723(5)	Mo(1)–O(3)	1.899(5)	Mo(1)–O(5)	1.969(4)
Mo(1)–N(1)	2.265(5)	Mo(1)–O(1)	2.372(5)	Br(1)–C(5)	1.894(6)	Br(2)–C(3)	1.888(7)
O(1)–C(10)	1.428(9)	O(3)–C(9)	1.431(9)	N(1)–C(7)	1.273(8)	C(1)–C(7)	1.440(8)
O(1)–H(1)	0.74(10)	O(5)–C(2)	1.338(7)	N(1)–C(8)	1.465(8)	C(8)–C(9)	1.488(11)
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
O(4)Mo(1)O(2)	107.2(3)	O(2)Mo(1)O(5)	99.9(2)	O(3)Mo(1)N(1)	75.5(2)	O(3)Mo(1)O(1)	83.0(2)
O(4)Mo(1)O(3)	100.0(2)	O(3)Mo(1)O(5)	151.6(2)	O(5)Mo(1)N(1)	79.9(2)	O(5)Mo(1)O(1)	77.6(2)
O(2)Mo(1)O(3)	98.1(2)	O(4)Mo(1)N(1)	93.5(2)	O(4)Mo(1)O(1)	168.5(2)	N(1)Mo(1)O(1)	76.4(2)
O(4)Mo(1)O(5)	95.5(2)	O(2)Mo(1)N(1)	159.1(2)	O(2)Mo(1)O(1)	83.2(2)	N(1)C(8)C(9)	105.5(6)
C(10)O(1)Mo(1)	130.4(4)	C(9)O(3)Mo(1)	120.2(4)	C(7)N(1)Mo(1)	126.9(5)	O(3)C(9)C(8)	108.8(5)
C(10)O(1)H(1)	130(9)	C(2)O(5)Mo(1)	129.7(4)	C(8)N(1)Mo(1)	111.2(4)		
Mo(1)O(1)H(1)	99(9)	C(7)N(1)C(8)	121.8(6)	N(1)C(7)C(1)	124.6(6)		

The IR spectra of azomethines and the complexes were recorded on an IKS-29 spectrometer in a range of 3600–400 cm^{-1} (suspensions in Nujol).

X-ray diffraction analysis of complex I was carried out on an Enraf-Nonius CAD4 automated diffractometer at room temperature (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). The crystals of complex **I** ($C_{10}H_{11}Br_2MoNO_5$, *FW* = 480.96) are orthorhombic, space group *Pbca*, a = 6.711(1), b = 13.278(1), c = 31.534(1) Å, V = 2810.0(5) Å^3, Z = 8, ρ_{calc} = 2.274 g/cm^3, μ(MoK_α) = 6.635 mm^{-1}, F(000) = 1840. The intensities of 12584 reflections (of which 2766 reflections were independent, R_{int} = 0.0948) were measured by the ω scan mode in the range $2.58° < \theta < 26.00°$ ($-1 \le h \le 8$, $-16 \le k \le 16$, $-38 \le l \le 38$). The structure was solved by a direct method and refined by full-matrix anisotropic least squares for F^2 for all nonhydrogen atoms (SHELXL-97 [16]). The hydroxyl hydrogen atom H(1) was found from the difference Fourier synthesis and refined isotropically, and other hydrogen atoms were placed in the calculated positions and refined by the riding model. The final *R* factors were R_1 = 0.0460, wR_2 = 0.1036 for 1856 reflections with $I > 2\sigma(I)$ and 178 refinement parameters, GOOF = 1.010, $\Delta\rho_{max}$, $\Delta\rho_{min}$ = 1.261, –1.564 e/Å^3. Selected bond lengths and bond angles are given in Table 1.

The coordinates of atoms and other parameters for the structure of compound **I** were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 1898088; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

It is experimentally found that the MC (rather than ICC) $MoO_2Cl_2 \cdot 2H_2L^{1,2}$ were formed by the reaction of MoO_2Cl_2 with $H_2L^{1,2}$ in low-polarity solvents (diethyl ether, dioxane, and tetrahydrofuran) in spite of the presence of two hydroxo groups in the salicylidenealcoholimine molecules (Table 2). These products are amorphous powders undergoing hydrolysis in air and are easily soluble in methanol to form conductive solutions. The molar electric conductance (λ_M) of MC solutions in methanol for concentrations of 1 × 10^{-3} mol/L is ~150 cm cm^2 mol^{-1}, which characterizes the MC as electrolytes of the 1 : 2 type [14].

Unlike the MC, the slow crystallization of the ICC [$MoOL^{1,2} \cdot$ Solv] from organic solvents produces structured crystals (Table 2). The obtained ICC are stable in air and poorly soluble in cold polar solvents (methanol, dimethyl sulfoxide, DMF, pyridine, and picolines), while they are soluble in them on heating to form solutions that do not conduct electric current. The storage of the complexes in air at 140–150°C for 2 h results in their complete desolvation.

The IR spectra of the obtained ICC (Table 2) exhibit the low-frequency shift of the stretching vibration band of the C=N bond by 10–14 cm^{-1} compared to its position in the spectra of free ligands, which is characteristic of inner complex compounds of metals with azomethine, and the disappearance of the stretching vibration bands of the hydroxo groups in a range of 3100–3600 cm^{-1}. The strong band at 1655 cm^{-1} in the spectrum of [$MoO_2L^2 \cdot$ DMF] is attributed to the decreased ν(C=O) frequency of the

Table 2. Results of physicochemical and IR spectroscopic studies of the dioxomolybdenum(VI) complexes with disubstituted salicylidenemonoethanolimines

Complex	Color	Mp, °C	Empirical formula	Content (found/calculated), %				IR spectrum, cm^{-1}			
								ν(C=N)	ν(C=N)	ν(C=O)	ν(Mo=O)
				Mo	C	H	N	of ligand	of complex		
$MoO_2L^1 \cdot MeOH$	Lemon	295–297	$C_{10}H_{11}Br_2MoNO_5$	20.18/19.95	24.79/24.97	2.46/2.30	2.80/2.91	1652	1638		930, 915, 900 sh
$MoO_2L^1 \cdot Py$	Lemon	300–302	$C_{14}H_{12}Br_2MoN_2O_4$	18.10/18.17	31.78/31.85	2.40/2.29	5.42/5.31	1652	1642		920, 890
$MoO_2L^1 \cdot \alpha$-Pic	Lemon	296–298	$C_{15}H_{14}Br_2MoN_2O_4$	17.58/17.70	33.05/33.24	2.88/2.60	5.25/5.17	1652	1642		922, 905
$MoO_2L^2 \cdot MeOH$	Lemon	278–280	$C_{11}H_{14}BrMoNO_6$	22.35/22.20	30.44/30.58	3.42/3.27	3.10/3.24	1650	1637		928, 902
$MoO_2L^2 \cdot DMF$	Lemon	275–277	$C_{13}H_{17}BrMoN_2O_6$	20.16/20.28	32.85/33.00	3.76/3.62	5.85/5.92	1650	1638		925, 900
$MoO_2L^2 \cdot Py$	Lemon	283–285	$C_{15}H_{15}BrMoN_2O_5$	19.96/20.02	37.46/37.60	3.27/3.16	5.70/5.84	1650	1640		922, 897
$MoO_2L^2 \cdot \alpha$-Pic	Lemon	285–287	$C_{16}H_{17}BrMoN_2O_5$	19.34/19.45	38.82/38.97	3.60/3.47	5.45/5.68	1650	1640		925, 903
$MoO_2Cl_2 \cdot 2HL^1$ *	Yellow	224–226	$C_{18}H_{18}Br_4Cl_2MoN_2O_6$	11.30/11.36	25.43/25.59	2.27/2.15	3.45/3.32	1652		1662	930, 905
$MoO_2Cl_2 \cdot 2HL^2$ **	Orange	218–220	$C_{20}H_{24}Br_2Cl_2MoN_2O_8$	12.87/12.84	32.06/32.15	3.42/3.24	3.90/3.75	1650		1658	932, 905

* Content of Cl (found/calculated), %: 8.45/8.39.

** Content of Cl (found/calculated), %: 9.40/9.49.

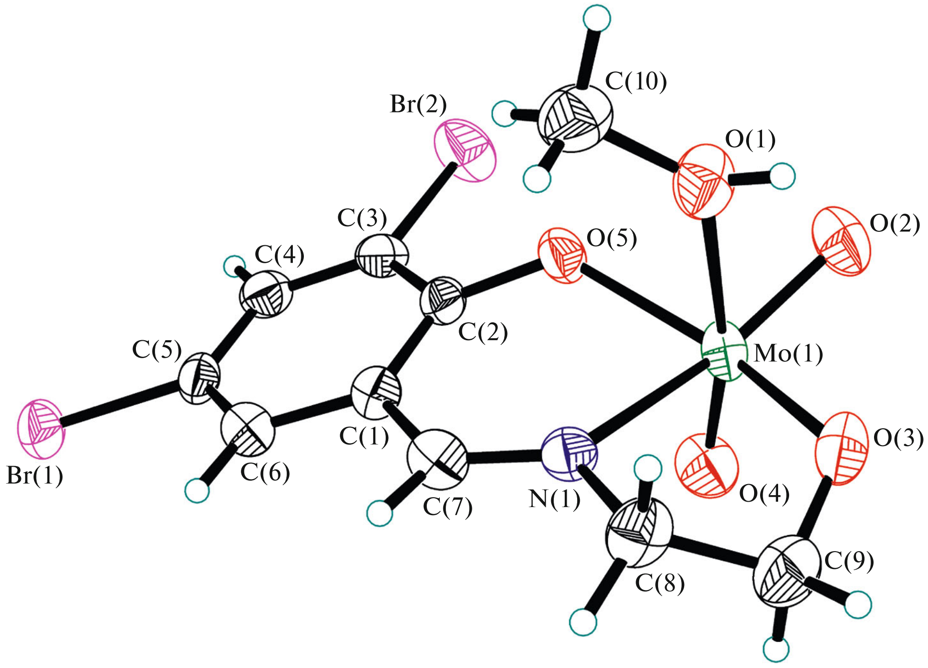


Fig. 1. Structure of the molecule of compound **I**. Thermal ellipsoids are given with 50% probability.

coordinated DMF molecule. The shift of a series of bands to a range of ~1000–1610 cm^{-1} corresponding to the vibrations of the bonds of the heterocycle is observed in the spectra of [MoO$_2$L^{1,2} · Solv], where Solv is pyridine or α-picoline [17].

The 1600–1700 cm^{-1} range of the spectra of the MC contains the intense band at ~1660 cm^{-1}, which should be ascribed (with the highest probability) to the stretching vibration band of the C=O quinoid (ketoamine) tautomer increased due to complex formation. The stabilization of the quinoid tautomeric form of *o*-oxyazomethines during the formation of complexes of the molecular type was repeatedly discussed [18–20]. Scarce literature data on the XRD of the molecular complexes of a series of metals with *o*-oxyazomethines [21–24] also confirm the coordination of the ligands in the quinoid tautomeric form.

The strong bands at 890–930 cm^{-1} are attributed to the symmetric and antisymmetric stretching vibrations of the Mo=O bond of the *cis*-MoO$_2$ group [1, 2]. New absorption bands appear in the IR spectra of the complexes at low frequencies: at 540–580 cm^{-1} (in the spectra of the MC) and at 520–540 and 470–475 cm^{-1} (in the spectra of the ICC), which should be assigned to the stretching vibrations of the $\mathrm{Mo{-}O_{HL}}$ or $\mathrm{Mo{-}N_L}$ and $\mathrm{Mo{-}O_L}$ bonds, respectively [1, 2, 14].

Based on the obtained experimental results and taking into account literature data, we may conclude that the studied MC and ICC of dioxomolybdenum(VI) with substituted salicylidenemonoethanolimines have the octahedral structure typical of oxo and dioxo cations of VIB Group with two multiply bonded oxo ligands in the *cis* position. In the MC, the vertices of the octahedron contain the terminal oxygen atoms, two carbonyl O atoms of the quinoid tautomeric form of oxoazomethines in the *trans* positions to the oxo ligands according to the principle of self-consistency [25], and two chlorine atoms in the *trans* positions to each other. The vertices of the octahedron in the ICC are occupied by two multiply bonded O atoms: the donor N atom of the azomethine group (*trans* to the oxo ligands) and two O atoms of the deprotonated hydroxo groups of the aldehyde and alcohol fragments in the *cis* positions to the oxo ligands. The sixth coordination site (*trans* to the oxo ligand) is occupied by the donor atoms of the coordinated solvent molecules.

The structure of ICC **I** was determined by the XRD method. In the mononuclear molecule of complex **I** (Fig. 1), the molybdenum atom has the octahedral coordination mode by two oxo ligands O(2) and O(4), two oxygen atoms O(3) and O(5) and the N(1) nitrogen atom of the tridentate bis(chelate) two-charge ligand (L^1)$^{2-}$, and the O(1) atom of the methanol molecule. According to the principle of self-consistency [25], the neutral N(1) and O(1) atoms of the L^1 and MeOH ligands, respectively, are arranged in the *trans* positions to the O(oxo) ligands. The Mo–N(1) (2.265 Å) and Mo–O(1) (2.372 Å) bonds are substantially elongated due to the structural manifestation of the *trans* effect of the multiply bonded oxo ligands. These bonds are also appreciably longer than the standard distances Mo–O(ST) (2.04 Å) and Mo–N(ST)

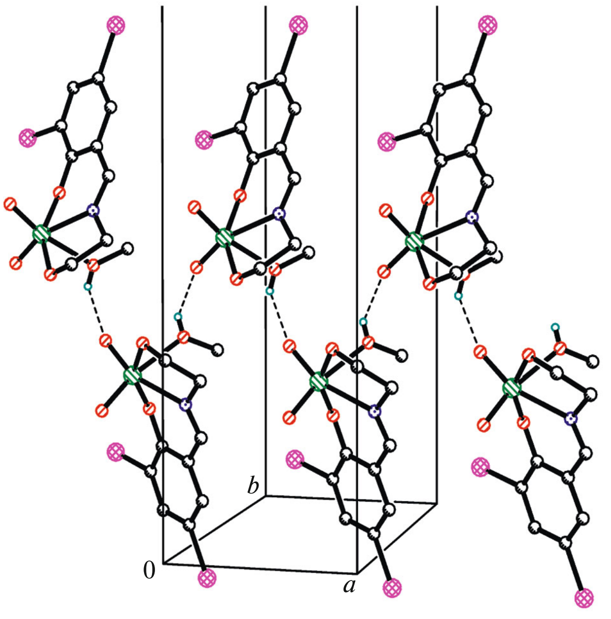


Fig. 2. Chains joined by hydrogen bonds in the structure of compound **I**.

(2.10 Å) [25]. The Mo–O(oxo) bonds (average 1.707 ± 0.016 Å) are typical in length and correspond to the increased multiplicity. Fairly short Mo–O(3)$_{cis}$ and Mo–O(5)(L^1)$_{cis}$ bonds differ substantially in length (1.899 and 1.969 Å, respectively). The Mo atom is shifted from the center of the MoO_5N octahedron to the O(2)(oxo)–O(4)(oxo) edge: the O(oxo)Mo(O,N)$_{cis}$ bond angles with the only exception for O(2)MoO(1) (83.2°) are larger than an ideal value of 90° (93.5°–100.0°), whereas the opposite (O,N)Mo(O,N) angles are noticeably smaller than 90° (75.5°–83.0°). The largest bond angle in the structure of complex **I** is O(2)MoO(4) (107.2°) because of the repulsion of the multiply bonded oxo ligands. The angles between the oppositely lying donor atoms of the ligands (O(3)MoO(5) 151.6°, O(2)MoN(1) 159.1°, and O(4)MoO(1) 168.5°) deviate from an ideal value of 180° to different extents. Two chelate cycles conjugated at the Mo–N(1) bond are closed upon the coordination of the tridentate ligand with the metal atom: six-membered $MoNC_3O$ (A) and five-membered $MoNC_2O$ (B). Metallocycle A has a sofa conformation with the deviation from four coplanar atoms NC_3 (±0.020–0.046 Å) to one direction of the Mo atoms by 0.430 Å and by 0.266 Å for O(5). Chelate cycle B has the asymmetric *twist* conformation with the deviation of the C(8) atoms by 0.129 Å and of C(9) by –0.469 Å from the plane of the Mo, N(1), and O(3) atoms. The dihedral angle between the A and B planes is 11.6°, and the average deviation of the atoms from the mean planes is 0.151 and 0.153 Å, respectively.

The molecules of compound **I** are joined in pairs by the hydrogen bond O(1)–H(1)···O(2) (0.5 + *x, y,* 0.5 – *z*) between the methanol molecule and one of the oxo ligands (O(1)–H(1) 0.74(10), H(1)···O(2) 2.07(10), O(1)···O(2) 2.695(6) Å, angle O(1)H(1)O(2) 143(13)°). The 1D chains along the *c* axis of the crystal are formed due to these hydrogen bonds (Fig. 2). Evidently, the participation of the oxo ligand O(2) in the hydrogen bond is a reason for the Mo–O(2) bond to be somewhat longer (1.723 Å) than Mo–O(4) (1.691 Å).

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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