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Molecular and Intracomplex Dioxomolybdenum(VI) Compounds with Substituted R¹-Salycilidene-*N*-methylimines (HL): Crystal Structure of Three [MoO₂(L)₂] Complexes (R¹ = H, 5-Br, 5-Cl)

V. S. Sergienko^{*a*, *b*, *, V. L. Abramenko^{*c*}, and Yu. E. Gorbunova^{*a*}}

^aKurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Moscow, 119991 Russia ^bAll-Russia Institute of Scientific and Technical Information, Russian Academy of Sciences, Moscow, 125315 Russia ^cDal' East Ukrainian National University, Lugansk, 91034 Ukraine *e-mail: sergienko@igic.ras.ru

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Abstract—The synthesis and IR spectroscopic and X-ray diffraction studies of three $[MoO_2(L^n)_2]$ complexes with n = 1 (R = H, repeatedly) (I), n = 4 (R = Br) (II), and n = 3 (R = Cl) (III) have been performed. The molybdenum atoms in the structures of complexes I–III have a typical octahedral coordination with dioxo ligands in *cis*-positions, atoms N(Lⁿ) in *trans*-positions to O(oxo), and atoms O(Lⁿ) in *cis*-positions to oxo ligands. Ligands Lⁿ are bidentate chelate (N, O).

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Salicylideneimines (o-oxyazomethines) (Scheme 1) in the reaction with Lewis acids ML_n form complexes of two types, such as adducts or molecular complexes (MCs) containing neutral ligand molecules and chelates representing intracomplex compounds (ICCs), whose ligands are coordinated to the central atom in the form of deprotonated anions. For Group VIB oxo and dioxocations, the complexes with salicylidene-Naryl- and hetarylimines are best studied, but much less attention has been paid to their complexes with salicylidene-N-alkylimines. A major part of papers is devoted to the synthesis and structural study of intracomplex compounds [1]. The synthesis of intracomplex compounds was performed via the ligand exchange between dioxomolybdenum(VI) acetylacetonate and a corresponding azomethine in boiling alcohol. However, the reaction between molybdenum dioxodichloride and salicylideneimines in a medium of low-polarity solvents yields molecular complexes [2-4].

For the further study of conditions for the synthesis of complex compounds of different types and the broadening of information about the structure of complexes formed by Group VIB dioxocations with *o*-oxyazomethines, the synthesis of dioxomolybdenum(VI) complexes with imines, derivatives of substituted salicylic aldehydes and methylamine (**HL**^{*n*}) was performed, and their structures and properties were investigated. The structures of three intracomplex compounds, such as $[MoO_2(L^1)_2]$ (I), $[MoO_2(L^4)_2]$ (II), and $[MoO_2(L^3)_2]$ (III) were characterized by X-ray diffraction. The structure of compound I was determined anew (see below for details).



HLⁿ: $\mathbb{R}^1 = \mathbb{H}$ (n = 1); 3-OMe (n = 2); 5-Cl (n = 3); 5-Br (n = 4); 3-NO₂ (n = 5); 5-NO₂ (n = 6); 3-OMe-5-Br (n = 7); 3,5-Br₂ (n = 8); 5,6-cyclo-C₄H₄ (n = 9). Scheme 1.

EXPERIMENTAL

Synthesis. Salicylidene-*N*-methylimines were synthesized via the coupling of corresponding substituted salicylic aldehydes and methylamine in boiling ethanol. After recrystallization from ethanol, they represented crystalline lemon powders (HL^1 is a liquid at room temperature, and HL^2 is orange crystals).

Molecular complexes like $MoO_2Cl_2 \cdot 2HL^n$ were synthesized by the immediate reaction between MoO_2Cl_2 and *o*-oxyazomethines in a medium of lowpolarity solvents. In diethyl ether, these complexes are formed with a nearly quantitative yield. To a solution

Parameter	Ι	II	III
Formula weight	396.25	554.05	465.13
Color, habit	Yellow, block	Yellow, block	Yellow, block
Crystal size	$0.15 \times 0.10 \times 0.05$	$0.18\times0.08\times0.05$	$0.18\times0.09\times0.05$
Symmetry system, space group	Orthorhombic, $P2_12_12_1$	Monoclinic, C2/c	Monoclinic, C2/c
a, Å	6.631(1)	19.312(1)	18.976(1)
b, Å	12.841(1)	9.398(1)	9.561(2)
<i>c</i> , Å	19.501(1)	13.595(1)	13.481(1)
β, deg	90	131.30(1)	132.24(3)
Ζ	4	4	4
$\rho_{calcd}, g/cm^3$	1.586	1.985	1.706
μ_{Mo}, mm^{-1}	9.810	5.041	1.042
<i>F</i> (000)	800	1072	926
Radiation (λ , Å)	MoK_{α}	(0.71073), graphite monochr	omator
<i>Т</i> , К		393	
Scanning type		ω	
Range of θ , deg	2.09-27.97	2.58-25.96	2.58-26.00
Index ranges	$-1 \ge h \ge 8, -1 \ge k \ge 16,$	$-1 \ge h \ge 23, -1 \ge k \ge 11,$	$-18 \ge h \ge 14, -9 \ge k \ge 9,$
	$-23 \ge l \ge 23$	$-16 \ge l \ge 13$	$-1 \ge l \ge 12$
Total number of reflections/ independent [<i>R</i> (int)]	5507/3945 [0.0690]	2247/1989 [0.0647]	1906/850 [0.0525]
Completeness over θ , %	98.7	100.0	100.0
Number of reflections with $I \ge 2\sigma(I)$	1713	851	688
Absorption correction		Semiempirical, by equivalents	S
T_{\min}/T_{\max}	0.8881/0.9606	0.4639/0.7867	0.8346/0.9497
Number of refined parameters	209	115	110
GOOF on F^2	0.920	0.849	1.104
$R\left[I \ge 2\sigma(I)\right]$	R1 = 0.0289, wR2 = 0.0655	R1 = 0.0319, wR2 = 0.0769	R1 = 0.0991, wR2 = 0.2125
R (all data)	R1 = 0.1930, wR2 = 0.0886	R1 = 0.1472, wR2 = 0.2095	R1 = 0.1148, wR2 = 0.2285
Residual electron density	0.493/-1.159	0.606/-0.852	2.430/-2.576
$(max/min), e/Å^3$			

Table 1. Crystallographic data and selected characteristics of X-ray diffraction experiment for the structures of complexesI-III

of molybdenum dioxodichloride (0.199 g, 0.001 mol) in absolute diethyl ether (20 mL), a solution of corresponding azomethine (0.002 mol) was added drop by drop under stirring on a magnetic agitator. The formed precipitates of complexes were separated out on a Schott glass filter equipped with a drying system, washed with ether, and dehumidified in a dry argon flow.

The intracomplex compounds $[MoO_2(L^n)_2]$ were synthesized via the ligand exchange between molybdenum acetylacetonate and corresponding azomethines. To a hot solution of $MoO_2(Acac)_2$ (0.326 g, 0.001 mol) in methanol (20 mL) a methanol solution of azomethine (0.002 mol) was added drop by drop, and the reaction mixture was boiled for 10-15 min and allowed to stand at room temperature for crystallization. The settled crystals of complexes were filtered out, washed on a filter with cold methanol, and dehumidified first in a dry air flow and then in vacuum desiccator over CaCl₂. The yield of complexes was 85-90%.

Elemental analysis for molybdenum, chlorine, and nitrogen was performed as described in [4] by standard methods.

IR spectra were recorded on an IKS-29 spectrometer as Nujol mulls.

X-ray diffraction analysis. Experimental data for crystals of complexes I-III were obtained on an

Enraf-Nonius CAD-4 automatic diffractometer. The structures were solved by direct methods (SHELXS-86) [5] and refined by the least-squares technique in the full-matrix anisotropic approximation for all nonhydrogen atoms (SHELXL-97) [6]. The positions of hydrogen atoms were calculated geometrically and included into refinement "as riding". It has been established that a crystal of complex I is a racemic twin (0.52(2): 0.48(6)). The low quality of a crystal of complex **III** (and its obvious propensity to twinning) leads to high *R*-factors. Unit cell parameters and selected characteristics of X-ray diffraction experiment for the structures of complexes I-III are given in Table 1, bond length and bond angles are listed in Table 2 for complex I and Table 3 for complexes II and III. Complete crystallographic data were deposited with the Cambridge Structure Database (nos. 1565270 (I), 1562990 (II), and 1565241 (III); http://www.ccdc. cam.ac.uk/deposit/.

RESULTS AND DISCUSSION

Independently of the ratio between reacting components, the reaction of MoO_2Cl_2 with salicylidene-*N*-methylimines in a low-polarity solvents results in molecular complexes of the only composition $MoO_2Cl_2 \cdot 2HL^n$ (Table 4) representing amorphous powders, which are hydrolyzable in air and readily soluble in methanol with the formation of electrically conductive solutions.

In contrast to the molecular complexes, the slow crystallization of intracomplex compounds $[MoO_2(L^n)_2]$ from methanol solutions results in well-shaped crystals melting at a much higher temperature than the corresponding adducts. The studied intracomplex compounds are stable in air and poorly soluble in cold polar solvents, but well soluble in methanol under heating, dimethyl sulfoxide, and dimethyl sulfamide to form electrically unconducive solutions.

Certain information necessary to determine the type of formed complex and the position of coordination bonds between metals and azomethines can be acquired by studying their IR spectra in the region of $1500-1700 \text{ cm}^{-1}$, in which several absorption bands can be distinguished for non-coordinated salicylideneimines. Of the greatest interest is the high-frequency band at 1620-1635 cm⁻¹ assigned to the stretching vibrations of the C=N azomethine group [7]. When an intracomplex compound is formed, the v(C=N) absorption band shiftstoward lower frequencies, thus indicating that the nitrogen atom of the C=N group participates in donor-acceptor interaction. The same region in the spectra of molecular complexes contains an intense band at ~1650 cm⁻¹, which should be assigned with the highest probability to the downshifted band of quinoid (ketoamine) tautomer b instead of the complexation upshifted band of

Table 2. Selected bond lengths and bond angles in the structure of complex ${\bf I}$

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
Mo(1)–O(1)	1.698(4)	O(3)–C(1)	1.345(6)
Mo(1)–O(2)	1.700(4)	O(4)–C(7)	1.337(6)
Mo(1)–O(4)	1.940(4)	N(1)–C(13)	1.282(6)
Mo(1)–O(3)	1.963(3)	N(1)-C(15)	1.463(6)
Mo(1)-N(1)	2.322(5)	N(2)-C(14)	1.279(6)
Mo(1)-N(2)	2.341(4)	N(2)-C(16)	1.475(6)
Angle	ω, deg	Angle	ω, deg
O(1)Mo(1)O(2)	107.56(19)	N(2)C(14)C(8)	125.7(5)
O(1)Mo(1)O(4)	96.53(17)	C(1)O(3)Mo(1)	127.4(3)
N(1)Mo(1)N(2)	76.30(15)	C(13)N(1)Mo(1)	122.7(4)
O(3)Mo(1)N(2)	84.03(17)	C(7)O(4)Mo(1)	133.3(3)
O(2)Mo(1)O(4)	97.59(17)	C(13)N(1)C(15)	116.1(5)
O(1)Mo(1)O(3)	94.94(18)	C(15)N(1)Mo(1)	121.2(4)
O(2)Mo(1)O(3)	96.03(16)	C(14)N(2)C(16)	116.5(5)
O(4)Mo(1)O(3)	158.64(18)	C(14)N(2)Mo(1)	122.8(4)
O(1)Mo(1)N(1)	89.07(18)	C(16)N(2)Mo(1)	120.6(3)
O(2)Mo(1)N(1)	163.22(18)	O(3)C(1)C(2)	119.3(5)
O(4)Mo(1)N(1)	82.39(18)	O(3)C(1)C(6)	121.2(5)
O(3)Mo(1)N(1)	79.85(18)	O(4)C(7)C(8)	121.7(5)
O(1)Mo(1)N(2)	165.30(16)	O(4)C(7)C(12)	118.5(5)
O(2)Mo(1)N(2)	87.13(17)	N(1)C(13)C(6)	125.6(5)
O(4)Mo(1)N(2)	80.32(16)	N(2)C(14)C(8)	125.7(5)

the stretching vibrations of the C=N bond of benzenoid (enolimine) tautomer *a* (Scheme 2):



The available scanty literature data on the X-ray diffraction analysis of molecular complexes of some metals with *o*-oxyazomethines [8–11] also confirms the coordination of ligands in quinoid tautomeric form *b*.

The intense absorption bands in the region of 900– 950 cm⁻¹ can be assigned to the symmetric and antisymmetric stretching vibrations of the Mo=O bond in the *cis*-MoO₂ group [12]. New absorption bands appear in the low-frequency region of the IR spectra of these complexes at 565–590 cm⁻¹ (MC spectra) and 520–540, 470–475 cm⁻¹ (ICC spectra) and should be

		II	
Bond	d, Å	Bond	$d, \mathrm{\AA}$
Mo(1)–O(2)	1.689(4)	Mo(1)–O(1)	1.950(4)
Mo(1) - N(1)	2.332(5)	Br(1)-C(4)	1.901(7)
O(1) - C(1)	1.342(7)	N(1) - C(7)	1.272(8)
N(1)–C(8)	1.481(8)		
Angle	ω, deg	Angle	ω, deg
O(1)#1Mo(1)O(2)	108.4(3)	C(1)O(1)Mo(1)	128.3(3)
O(1)Mo(1)N(1)	79.5 (2)	C(7)N(1)C(8)	117.5(5)
O(2)Mo(1)O(1)#1	95.7(2)	C(7)N(1)Mo(1)	121.9(4)
O(2)Mo(1)N(1)	86.8(2)	C(8)N(1)Mo(1)	120.3(4)
O(2)Mo(1)O(1)	96.6(2)	O(1)C(1)C(3)	118.0(5)
O(1)#1Mo(1)O(1)	158.9(2)	O(1)C(1)C(6)	122.6(5)
O(2)Mo(1)N(1)#1	164.6(2)	C(3)C(4)Br(1)	118.1(5)
O(1)Mo(1)N(1)#1	84.2(2)	C(5)C(4)Br(1)	120.4(5)
N(1)C(7)C(6)	125.9(6)		
		Ш	
Bond	d, Å	Bond	d, Å
Mo(1)–O(2)	1.687(9)	Mo(1)–O(1)	1.978(8)
Mo(1) - N(1)	2.329(11)	N(1)–C(7)	1.291(19)
N(1)–C(8)	1.465(19)	O(1)–C(6)	1.334(16)
Angle	w, deg	Angle	ω, deg
O(2)#1Mo(1)O(2)	108.7(6)	O(2)C(6)C(5)	119.3(12)
O(2)#1Mo(1)O(1)	96.9(4)	O(1)C(6)C(1)	119.3(12)
O(2)Mo(1)O(1)	95.9(4)	C(7)N(1)C(8)	117.5(11)
O(1)Mo(1)O(1)#1	157.9(5)	C(7)N(1)Mo(1)	121.0(8)
O(2)Mo(1)N(1)	86.8(4)	C(8)N(1)Mo(1)	121.2(10)
O(1)Mo(1)N(1)	79.4(4)	O(2)Mo(1)N(1)#1	164.5(4)
O(1)Mo(1)N(1)#1	83.4(4)	O(1)Mo(1)N(1)#1	79.4(4)
C(4)O(1)Mo(1)	129.5(7)	N(1)C(7)C(1)	125.6(12)

Table 3. Selected bond lengths (*d*) and bond angles (ω) in the structures of complexes II and III*

* Symmetry codes in the structures of complexes II and III: #1 - x, y, -z + 1/2.

assigned to the stretching vibrations of the $Mo-O_{HL}$, and $Mo-N_L$, $Mo-O_L$ bonds [4], respectively.

Based on the obtained experimental results and the literature data [13], it is possible to conclude that the studied dioxomolybdenum(VI) MCs and ICCs with substituted salicylidene-N-methylimines have an octahedral structure with two multiply bonded oxo ligands in cis-positions as typical for Group VIB oxoand dioxocations. In addition to the terminal oxygen atoms, the vertices of MC octahedra are occupied by two carbonyl oxygen atoms of the quinoide tautomeric form of oxoazomethines in trans-positions to oxo ligands according to the "self-consistency rule" [14] and two chlorine atoms in trans-positions to each other. The vertices of ICC octahedra are occupied by two multiply bonded terminal oxygen atoms in cispositions, two donating nitrogen atoms in trans-positions to oxo ligands, and two oxygen atoms of deprotonated hydroxo groups of salicylideneimines in *trans*-positions to each other.

The structures of intracomplex compounds I-III were established by X-ray diffraction. The crystal structure of compound I was characterized earlier by a photographic method (Ia) [15] at a low precision (*R*-factor = 7%). Our study is more precise (R = 3.2%) despite the twin nature of a single crystal of compound I. Crystals of compounds II and III are isostructural. Since the structure of a twin crystal of compound III was determined at a low precision, we shall operate with the data on the structure of compound II in the discussion of results. The molybdenum atoms in compounds I-III have an octahedral coordination. The structures of complexes I and III are shown in Fig. 1 and Fig. 2, respectively. Similarly to all the other dioxo complexes of Group V–VII metals d^0 –M^{5+x} (x = 0-2; $M = V^{5+}$, Nb^{5+} , Mo^{6+} , W^{6+} , Tc^{7+} , Re^{7+}), the O(oxo) ligands in compounds I-III are in *cis*-positions to

Table 4.	Results of the phy	sicochem	ical and IR	c-spectroscopic studies o	f dioxomolyb	denum(VI) N	MCs and ICC	s with substit	uted salicylide	ene-N-methy	limines
					Content (found/calcu	lated) %		IR spectru	ım, cm ⁻¹	
Ligand	Complex	Color	$T_{\mathrm{m}}, ^{\circ}\mathrm{C}$	Bulk formula		יזטמוומ/ כמוכמ	acu), /o	v(C=N)	v(C=N)	v(C=0)	v(Mo=O)
					Мо	z	CI	ligand		complex	
HL^{1}	$MoO_2Cl_2 \cdot 2HL^1$	Yellow	141-143	$C_{16}H_{18}Cl_2MoN_2O_4$	20.58/20.45		15.23/15.11	1630		1660	928, 903
HL^2	$MoO_2Cl_2 \cdot 2HL^2$	Orange	136-138	$\mathrm{C}_{\mathrm{18}}\mathrm{H}_{22}\mathrm{Cl}_{6}\mathrm{MoN}_{2}\mathrm{O}_{6}$	18.25/18.13		13.35/13.40	1632		1658	930, 903
HL^3	$MoO_2Cl_2 \cdot 2HL^3$	Yellow	155–157	$C_{16}H_{16}Cl_4MoN_2O_4$	17.87/17.83		13.07/13.18*	1633		1662	933, 902
HL^4	$MoO_2Cl_2 \cdot 2HL^4$	Yellow	150-152	$C_{16}H_{16}Br_2Cl_2MoN_2O_4$	15.42/15.30		11.35/11.31	1633		1660	932, 900
HL ⁵	$MoO_2Cl_2 \cdot 2HL^5$	Yellow	143-145	$C_{16}H_{16}Cl_2MoN_4O_8$	17.07/17.16		12.60/12.68	1632		1658	928, 904
HL^{6}	$MoO_2Cl_2 \cdot 2HL^6$	Lemon	156–158	$C_{16}H_{16}Cl_2MoN_4O_8$	17.20/17.16		12.55/12.68	1635		1662	930, 905
HL^7	$MoO_2Cl_2 \cdot 2HL^7$	Orange	145-147	$\mathrm{C}_{\mathrm{18}}\mathrm{H}_{\mathrm{20}}\mathrm{Br}_{2}\mathrm{Cl}_{2}\mathrm{MoN}_{2}\mathrm{O}_{6}$	13.87/13.96		10.37/10.32	1627		1657	932, 904
HL^{8}	$MoO_2Cl_2 \cdot 2HL^8$	Yellow	138-140	$C_{16}H_{14}Br_4Cl_2MoN_2O_4$	12.15/12.23		9.15/9.04	1628		1660	933, 905
HL^9	$MoO_2Cl_2 \cdot 2HL^9$	Brown	142144	$C_{24}H_{22}Cl_2MoN_2 O_4$	16.92/16.85		12.38/12.45	1632		1658	930, 904
HL^{1}	$MoO_2L^1_2$	Lemon	237-238	$\mathrm{C}_{16}\mathrm{H}_{16}\mathrm{MoN}_{2}\mathrm{O}_{4}$	24.28/24.21	7.15/7.07		1630	1624		922, 895
HL^2	$MoO_2L^2_2$	Orange	234-236	$\mathrm{C}_{18}\mathrm{H}_{20}\mathrm{MoN}_{2}\mathrm{O}_{4}$	21.10/21.03	6.05/6.14		1632	1624		920, 897
HL^3	$MoO_2L^3_2$	Brown	272-274	$C_{16}H_{14}Cl_2MoN_2O_4$	20.58/20.63	5.95/6.02		1633	1623		930, 902
HL^4	$MoO_2L^4_2$	Brown	278–280	$C_{16}H_{14}Br_2MoN_2O_4$	17.37/17.32	5.10/5.06		1633	1626		928, 902
γTH	$MoO_2L^5_2$	Yellow	245-247	$C_{16}H_{14}MoN_4O_8$	19.65/19.73	11.35/11.52		1632	1625		925, 900
HL ⁶	$MoO_2L^6_2$	Lemon	253-255	$C_{16}H_{14}MoN_4O_8$	19.86/19.73	11.40/11.52		1635	1624		922, 897
HL^7	$MoO_2L^7_2$	Yellow	240-242	$\mathrm{C}_{\mathrm{18}}\mathrm{H}_{\mathrm{18}}\mathrm{Br}_{2}\mathrm{MoN}_{2}\mathrm{O}_{6}$	15.74/15.62	4.45/4.56		1627	1615		925, 903
HL^{8}	$MoO_2L^8_2$	Yellow	270-272	$C_{16}H_{12}Br_4MoN_2O_4$	13.57/13.48	3.85/3.94		1628	1617		923, 895
HL^9	$MoO_2L_2^9$	Yellow	285-287	$\mathrm{C}_{24}\mathrm{H}_{20}\mathrm{MoN}_{2}\mathrm{O}_{4}$	19.40/19.33	5.53/5.64		1632	1625		924, 900
* Conten	t of the Cl ⁻ ion.										

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Fig. 1. Structure of complex I.



Fig. 2. Structure of complex II.

each other. According to the "self-consistency rule" [14], trans-positions to oxo ligands (and cis-positions to each other) are occupied by the neutral nitrogen atoms of ligands L^n (n = 1, 3, 4), the bonds with which can be more easily weakened, and cis-positions to oxo ligands (and *trans*-positions to each other) are occupied by acido atoms $O(L^n)$. The Mo-N bonds in complexes I and II (2.331(4) \pm 0.010 Å in I, 2.341(9) \pm 0.004 Å in Ia, and 2.332(5) Å in II) are elongated by 0.23 Å due to the structural *trans*-effect of the multiply bonded ligand in comparison with the standard distance Mo-N(ST) of 2.11 Å [14]. The bonds Mo-O(0x0) (1.699(4) ± 0.001 Å in I, 1.685(8) ± 003 Å in Ia, and 1.689(4) Å in II) and Mo–O(L¹) (1.952(3) \pm 0.012 Å in I, $1.965(8) \pm 0.015$ Å in Ia, and 1.950(5) Å in II) have a standard length. The coordination of ligands L^n to metal atoms closes two chelate rings of

different antisymmetric conformation. In a crystal of compound I, the neighboring Mo(1) and N(1) atoms of the Mo(1)-N(1)-C(1)-C(2)-C(3)-O(3) chelate ring deviate at 1.136 and 0.304 Å, respectively, in the same direction from the plane of nearly coplanar $(\pm 0.003 - 0.063 \text{ Å}) \text{ O}(3)$ and C(1) - C(3) atoms, while the neighboring Mo(1) and O(4) atoms of the Mo(1)-N(2)-C(11)-C(12)-C(17)-O(4) chelate ring deviate at -0.506 and +0.148 Å, respectively, in different directions from the plane of nearly coplanar (± 0.015 – 0.033 Å) N(2), C(11), C(12), and C(17) atoms. The similar Mo(1)-N(1)-C(7)-C(6)-C(1)-O(1) chelate ring in the structure of complex II has a "sofa" conformation, where the Mo(1) atom deviates at 0.896 Å from the plane of the other five nearly coplanar $(\pm 0.022 - 0.077 \text{ Å})$ atoms.

The structures of compounds **I**–**III** contain no short intermolecular contacts that would correspond to hydrogen bonds.

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