# Inner Complex Compounds of Dioxomolybdenum(VI) with *o*-Oxyazomethines, Derivatives of Substituted Salicylaldehydes and Tris(hydroxymethyl)aminomethane. Crystal Structures of Two Complexes [MoO(L)] $\cdot$ CH<sub>3</sub>OH; L = Z-Substituted Salicylalimines, Z = 3-NO<sub>2</sub> and 3-OCH<sub>3</sub>

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Abstract—Ten new dioxomolybdenum(VI) compounds with *o*-oxyazomethines, derivatives of substituted salicylaldehydes (I–X) and tris(hydroxymethyl)aminomethane, are synthesized. The structures of two of them,  $[MoO_2(L)] \cdot CH_3OH$ ; L = Z-substituted salicylalimines, Z = 3-NO<sub>2</sub> (IV) and 3-OCH<sub>3</sub> (V), are determined by X-ray diffraction analysis. Compounds IV and V have similar structures and geometric parameters. The Mo atoms are coordinated through the octahedral mode by two oxo ligands in the *cis* positions to each other, two O atoms, one N atom of the tridentate bis(chelating) ligand L, and the O atom of the MeOH molecule.

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## **INTRODUCTION**

Salicylalimines (*o*-oxyazomethines, Schiff bases) represent a classical example of prototropic tautometic systems for which two tautometrs can theoretically exist in the crystalline state and in solution (a, b,

c; scheme). One of them can be presented in two resonance forms (b, c; scheme), which makes it possible to obtain from them complexes of different types and structures: adducts or molecular complexes, and inner complex compounds [1, 2].



Phenol–imine tautomer (*a*)



Quinoid (b) and zwitterionic (c) resonance forms of keto-amine tautomer

#### Scheme.

The introduction of additional donor centers or functional groups into amine fragment R extends coordination possibilities of salicylalimines. In particular, salicylalcoholimines (H<sub>2</sub>L), where R is (CH<sub>2</sub>)<sub>n</sub>OH, form with transition metal salts several types of inner complex compounds of the monomeric [1-5] and di- and polymeric structures [6, 7]. In these structures, the ligand exhibits the bi- or tridentate function and is coordinated through the oxygen atom of the deprotonated phenolic OH group and the azomethine nitrogen atom or through two oxygen atoms of the phenolic and alcoholic hydroxy groups and the nitrogen atom of doubly deprotonated anion  $L^{2-}$ . The data of elemental analysis, IR and NMR spectroscopy [8–11], and X-ray diffraction analysis [12] showed that the solvated dioxomolybdenum(VI) complexes with substituted salicylalmonoethanolamines  $MoO_2L \cdot CH_3OH$  were monomeric. Continuing the systematic study of the complex formation of dioxomolybdenum(VI) with salicylalcoholimines and the direct proof of the complexes formed, we synthesized ten inner complex compounds of Mo(VI) with a series of *o*-oxyazomethines, derivatives of salicylaldehyde (Z = H (I), 5-Br (II), 5-NO<sub>2</sub> (III), 3-NO<sub>2</sub> (IV), 3-OCH<sub>3</sub> (V), 3,5-Cl<sub>2</sub> (VI), 3,5-Br<sub>2</sub> (VII), and 3-OCH<sub>3</sub>-5-NO<sub>2</sub> (VIII) and  $\beta$ -oxynaphthoic aldehyde (Z = 5,6-cyclo-C<sub>4</sub>H<sub>4</sub> (IX, X) and tris(hydroxymethyl)aminomethane ( $R = C(CH_2OH)_3$ ). The structures of compounds IV and V were determined by X-ray diffraction analysis.

### **EXPERIMENTAL**

Synthesis of I–X. Azomethines were synthesized by refluxing for 0.5 h of equimolar amounts of the corresponding aldehydes and amine  $NH_2C(CH_2OH)_3$  in alcohol followed by keeping the solution in a refrigerator for 24 h. The crystals formed were filtered off, washed with cold alcohol, and dried first on the filter and then over CaCl<sub>2</sub>. The purity of the substances was monitored chromatographically.

The complexes were synthesized by refluxing solutions of equimolar amounts of molybdenyl acetylacetonate and azomethines in methanol, distilling off a portion of the solvent, and cooling the solutions to 0°C. The yields of the complexes ranged from 80 to 90% of the theoretical value. Compounds **VIII** and **X** with dimethylformamide (DMF), MoO<sub>2</sub>L · DMF, were synthesized by the recrystallization of complexes MoO<sub>2</sub>L · CH<sub>3</sub>OH in methanol with the addition of 1– 2 mL of dimethylformamide.

On heating to  $140-150^{\circ}$ C complexes **I**-**X** are desolvated and on further heating melt with decomposition.

The elemental analysis data, melting points, and IR spectra of complexes I-X (IKS-29 spectrophotometer, suspension in Nujol) are given in Table 1.

**X-ray diffraction analysis.** The main crystallographic characteristics and refinement parameters for structures **IV** and **V** are listed in Table 2. The experimental data were obtained on Bruker SMART APEX II (for **IV**) and Enraf-Nonius CAD-4 (for **V**) diffractometers ( $\lambda Mo K_{\alpha}$  radiation,  $\lambda = 0.7073$  Å, graphite monochromator,  $\omega$  scan mode). An absorption correction was applied empirically by equivalent reflections.

Structures **IV** and **V** were determined by direct methods and refined by least squares for  $F^2$  in the fullmatrix anisotropic approximation for non-hydrogen atoms [13]. The hydrogen atoms in structure **IV** (except for the hydrogen atoms of the methyl group) and the H(1–3) atoms of the hydroxy groups in structure V were objectively localized from difference Fourier syntheses and refined isotropically. Other hydrogen atoms in structure V (and H(Me) atoms in IV) were specified in the calculated positions and refined by the riding model.

Selected interatomic distances and bond angles are given in Table 3. The hydrogen bond parameters are listed in Table 4. The full tables of atomic coordinates, bond lengths, and bond angles for compounds **IV** and **V** were deposited with the Cambridge Crystallographic Data Centre (nos. 917591 and 917592, respectively; deposit@ccdc.cam.ac.uk; http://www.ccdc. cam.ac.uk/data request/cif).

## **RESULTS AND DISCUSSION**

The X-ray diffraction results for free o-oxyazomethines and derivatives of salicylaldehydes and tris(hydroxymethyl)aminomethane show that in the crystalline state these azomethines exist predominantly in the quinoid form [14-20], whereas the zwitterionic form is rarely met [20]. For example, the N–H bond length is 1.01 Å and the H…O distance is 1.77 Å in an N-salicylideneamino-1-tris(hydroxymethyl)methane molecule, which unambiguously indicates the localization of the hydrogen atom on the nitrogen atom [14]. At the same time, the nature of substituents in both the aldehvde and amine fragments affects the stabilization of a specific tautomeric form of salicylalimines in the crystalline state. In this respect, the X-ray diffraction data are convincing for salicylalimines, condensation products of substituted Z-salicylaldehyde and tris(hydroxymethyl)aminomethane [20]. Zwitterionic form c is detected in the crystalline state for azomethine with the substituent Z = 3-OEt in the aldehvde fragment. In the case of Z = 5-NO<sub>2</sub>, the quinoid form b is observed as a result of a resonance between the zwitterionic form and nitro group. Unsubstituted (Z = H) salicylidene-2-(1hydroxybutyl)imine crystallizes in the phenol-imine form *a* [20].

The IR spectra of complexes I-X (Table 1) demonstrate the following features. Broad diffuse absorption bands of alcoholic groups are observed at 3000- $3600 \text{ cm}^{-1}$ , which (along with the elemental analysis data) suggests the participation of only one of three OH groups of the amine fragment in coordination. It is most likely that the ligands are transformed into the benzoid tautomeric form followed by the deprotonation of one of the alcohol groups and five-membered metallocycle closure. The stretching vibrations of the C=N azomethine bond appear as intense bands at 1620–1630 cm<sup>-1</sup> as in the spectra of the dioxomolybdenum(VI) complexes with salicylidenemonoethanolamines [12]. The absorption bands at  $500-590 \text{ cm}^{-1}$ can be assigned to stretching vibrations of the Mo–O<sub>I</sub> bond. The strong bands at 900–950 cm<sup>-1</sup> are attrib-





Structures of complex molecules (a) IV and (b) V. The intramolecular hydrogen bond is shown by dashed line.

uted to symmetric and antisymmetric stretching vibrations of the *cis*-MoO<sub>2</sub> group [13].

According to the X-ray diffraction data, complexes IV and V (figure) have the monomeric octahedral structure. The structures of the complex molecules of both compounds and analogous geometric parameters are similar. The  $MoO_2^{2+}$  dioxo group is in the *cis* configuration typical of complexes of  $d^0$  metals of Groups V–VII in the highest oxidation state [21, 22]. The O(7) atoms of the solvate methanol molecules and the N(1) atoms of tridentate bis(chelate) ligands L<sup>2–</sup> are in the *trans* positions to the oxo ligands in structures IV and V. The deprotonated O(3) atoms of the oxyazomethine fragment and O(4)(ethanolamine) are arranged in the *trans* positions to each other and in the *cis* positions to the oxo ligands. For the coordination of the metal atom with ligand  $L^{2-}$  in structures IV and V, two condensed metallocycles are closed: the sixmembered cycle Mo, O(3), C(2), C(1), C(7), N(1) (A) and five-membered metallocycle Mo, O(4), C(9), C(8), N(1) (**B**). The conformation of chelate **A** in structure IV is a flattened sofa (the O(3) atom is declined by 0.153 Å from the plane of other five coplanar ( $\pm 0.003 - 0.011$  Å) atoms). The cycle in structure V has a substantially nonplanar conformation with the deviation of the Mo and O(3) atoms from the plane of other four coplanar ( $\pm 0.017 - 0.038$  Å) atoms by 0.317 and 0.199 Å, respectively. The five-membered metallocycles **B** in structures **IV** and **V** have the same skewed conformation with the deviation of the O(4) and C(9)atoms from the plane of the Mo, N(1), and C(8) atoms by -0.456, 0.127 Å in **IV** and -0.183, 0.443 Å in **V**,

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<b>Table 2.</b> Main crystallographic and experimental characteristics of compounds $IV$ and $V$					
Parameter	Value				
	IV	V			
Crystal system	Monoclinic	Monoclinic			
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	$P2_{1}/c$			
<i>a</i> , Å	6.9228(3)	10.883(2)			
b, Å	25.0045(11)	9.098(3)			
<i>c</i> , Å	8.4795(4)	15.801(4)			
β, deg	100.384(1)	101.30(2)			
<i>V</i> , Å <sup>3</sup>	1443.77(11)	1534.2(7)			
Temperature, K	150(2)	100(2)			
Ζ	4	4			

428.21

1.970

864

0.964

27.00

 $0.40 \times 0.10 \times 0.02$ 

0.0253

0.9810, 0.6991

11319

3142

2893

270

 $R_1 = 0.0221, wR_2 = 0.0541$ 

 $R_1 = 0.0251, wR_2 = 0.0556$ 

1.039

0.464, -0.446

Tab

М

 $\rho_{calcd}, g/cm^3$ 

*F*(000)

μ, mm

**R**<sub>int</sub>

 $\theta_{max}$ , deg

 $T_{\rm max}, T_{\rm min}$ 

 $R(I > 2\sigma(I))$ 

*R* (all data)

Goodness-of-fit

 $\rho_{max}, \rho_{min}, e/Å^3$ 

Sample size, mm

Total number of reflections

Number of independent reflections

Number of reflections,  $I > 2\sigma(I)$ 

Number of refined parameters

413.23

1.789

840

0.897

25.98

 $0.40 \times 0.30 \times 0.10$ 

0.0313

0.9156, 0.7155

11590

3006

2723

222

 $R_1 = 0.0181, wR_2 = 0.0460$ 

 $R_1 = 0.0226, wR_2 = 0.0474$ 

1.048

0.393, -0.280

Dand	IV	V	
Bond	d. Å		
$M_{0}(1) = O(1)$	1 7025(15)	1 6997(13)	
$M_0(1) = O(1)$	1.7023(15) 1.7053(15)	1.077(13) 1.7055(13)	
$M_0(1) = O(2)$ $M_0(1) = O(4)$	1.7055(15) 1.0250(13)	1.7055(15) 1.9550(13)	
$M_0(1) = O(4)$ $M_0(1) = O(3)$	1.9239(13) 1.0780(14)	1.9550(15) 1.0640(13)	
$M_0(1) = O(3)$ $M_0(1) = N(1)$	1.3700(14) 2.2705(16)	1.9049(13) 2.2085(15)	
$M_0(1) = N(1)$ $M_2(1) = O(7)$	2.2793(10) 2.2021(16)	2.2903(13) 2.2054(14)	
MO(1) = O(7)	2.3021(10) 1.200(2)	2.3034(14)	
N(1) - C(7)	1.280(2) 1.407(2)	1.288(2) 1.491(2)	
N(1) - C(8) N(2) - O(0)	1.497(2)	1.481(2) 1.220(2)	
N(2) = O(9)	1.223(2) 1.220(2)	1.339(2)	
N(2) = O(8)	1.230(2)	1.419(2)	
N(2) - C(3)	1.465(3)	1.424(2)	
O(3) - C(2)	1.313(2)	1.422(2)	
O(4) - C(9)	1.431(2)	1.42/(2)	
O(5) - C(11)	1.425(2)	1.366(2)	
O(6) - C(10)	1.427(3)	1.436(2)	
O(7) - C(12)	1.420(3)	1.403(2)	
Angle	IV	V	
	ω, deg		
O(1)Mo(1)O(2)	106.27(8)	105.65(6)	
O(1)Mo(1)O(4)	99.38(7)	98.04(6)	
O(2)Mo(1)O(4)	97.41(6)	97.35(6)	
O(1)Mo(1)O(3)	97.35(7)	97.91(6)	
O(2)Mo(1)O(3)	100.05(7)	101.78(5)	
O(4)Mo(1)O(3)	151.31(6)	150.69(5)	
O(1)Mo(1)N(1)	93.57(7)	89.76(6)	
O(2)Mo(1)N(1)	159.69(7)	163.60(6)	
O(4)Mo(1)N(1)	74.57(6)	74.50(5)	
O(3)Mo(1)N(1)	81.31(6)	81.15(5)	
O(1)Mo(1)O(7)	168.67(7)	170.27(5)	
O(2)Mo(1)O(7)	85.02(7)	84.03(6)	
O(4)Mo(1)O(7)	79.76(6)	81.41(5)	
O(3)Mo(1)O(7)	79.29(6)	78.76(5)	
N(1)Mo(1)O(7)	75.27(6)	80.71(5)	
C(7)N(1)C(8)	118.49(16)	119.74(14)	
C(7)N(1)Mo(1)	127.74(14)	126.68(12)	
C(8)N(1)Mo(1)	113.53(11)	113.32(10)	
O(9)N(2)O(8)	123.42(19)	133.21(11)	
O(9)N(2)C(3)	119.03(17)	117.11(10)	
O(8)N(2)C(3)	117.50(18)	133.28(12)	
C(2)O(3)Mo(1)	138.07(13)	116.45(14)	
C(9)O(4)Mo(1)	118.78(11)	119.69(16)	
C(12)O(7)Mo(1)	127.48(14)	123.07(16)	
O(3)C(2)C(3)	120.95(18)	119.09(15)	
O(3)C(2)C(1)	122.93(18)	124.66(16)	
C(4)C(3)N(2)	116.87(18)	119.99(16)	
C(2)C(3)N(2)	120.20(18)	120.43(16)	
N(1)C(7)C(1)	125.72(18)	104.42(13)	
N(1)C(8)C(9)	106.02(15)	106.38(14)	
N(1)C(8)C(10)	109.53(15)	110.25(14)	
N(1)C(8)C(11)	111.87(15)	110.02(14)	
O(4)C(9)C(8)	111.11(16)	110.00(15)	
O(6)C(10)C(8)	108.61(16)	110(2)	
O(5)C(11)C(8)	112.40(16)	103.3(18)	
C(11)O(5)H(1)	105(2)	114(2)	
C(10)O(6)H(2)	109(3)	109(2)	
C(12)O(7)H(3)	116(2)	120	
$M_0(1)O(7)H(3)$	117(2)	120	

**Table 3.** Selected bond lengths (*d*) and bond angles ( $\omega$ ) for compounds IV and V

respectively. The phenylene ring C(1–6) (C) conjugated with the six-membered chelate A is planar with an accuracy of  $\pm 0.004-0.016$  and  $\pm 0.005-0.016$  Å in structures IV and V, respectively. The inflection angles along the C(1)–C(2) line are 4.0° and 11.9° in structures IV and V, respectively.

The lengths of the Mo=O(1,2) multiple bonds  $(1.704 \pm 0.001 \text{ Å in IV}, 1.703 \pm 0.003 \text{ Å in V})$  fall in the typical range of values from 1.66 to 1.77 Å (Table 5 in [23, 24]).

The bonds in the *trans* positions to Mo=O(oxo) are usually elongated strongly due to the random transeffect of the multiply bonded oxo ligands. Nevertheless, the Mo-N(1) bonds in IV and V (2.280 and 2.299 Å, respectively), as a similar bond in the close in composition structure  $[MoO_2(L)] \cdot CH_3OH$  (XI), where  $L^2$  is 3.5-dichlorosalicylidene monoethanoliminate; R,R is 3,5-Cl<sub>2</sub> (Mo–N 2.277 Å), are among the shortest (in other words, least weakened) bonds according to literature data (for example, Mo-N<sub>trans</sub> 2.308–2.486 Å for 12 structures of the monomeric octahedral Mo(VI) dioxo complexes with n-dentate ligands (n = 2-4) [21]). In particular, the Mo-N bonds in structures IV, V, and XI are shorter than similar bonds in the structures of the salicylaliminate complexes  $[MoO_2{R-N=CH-C_6H_4-O_2}], R = Ph$ (XII) [23] and Me (XIII) [25]: Mo-N<sub>trans</sub> 2.382 and 2.341 Å, respectively. The presence of the third donor center (the oxygen atom of the ethanolamine substituent at the nitrogen atom) enhances the dentate character of oxyazomethine ligand L on going from XII and XIII to III, V, and XI. The nitrogen atom migrates from the periphery of the bidentate ligand to the central position of the bicyclic system in structures III, V, and XI, which results in some fixation and mobility restriction of the Mo–N bond. Note that the  $Mo-N_{trans}$  distances in structures III, V, and XI are elongated by 0.160, 0.179, and 0.157 Å, respectively, compared to the standard ("expected") value of the Mo–NR<sub>3</sub> bond (2.12 Å [26]).

The Mo–O(7)(CH<sub>3</sub>OH) bonds, which are *trans*coordinated to O(1)<sub>oxo</sub> (2.302 and 2.305 Å in **IV** and **V**, respectively) as Mo–O(CH<sub>3</sub>OH) 2.336 Å in **XI**, are elongated due to the random trans-effect of the multiply bonded oxo ligand. The lengths of these bonds in three above mentioned structures are elongated by 0.350, 0.345, and 0.380 Å, on the average, in **IV**, **V**, and **XI**, respectively, compared to the Mo–O(3,4)<sub>cis</sub> distances (1.952, 1.960, and 1.956 Å). The Mo–O<sub>cis</sub> bonds in structures **IV**, **V**, and **XI** are comparable in length with analogous values in complexes **XII** and **XIII** containing two bidentate (O, N) ligands (Schiff bases) (Mo–O<sub>cis</sub> 1.942 and 1.964 Å, respectively). The Mo–O(CH<sub>3</sub>OH)<sub>trans</sub> bonds in structures **IV**, **V**, and **XI** 

Contact D. H. A	Distance, Å		Angle DHA deg			
Contact D=11A	D-H	Н…А	D···A	Aligie DHA, deg		
IV						
O(7)-H(3)····O(6)	0.72(3)	2.06(3)	2.734(2)	156(3)		
$O(5)-H(1)\cdots O(4)^{\#1}$	0.68(3)	2.05(3)	2.721(2)	171(3)		
O(6)-H(2)···O(5) <sup>#2</sup>	0.69(3)	2.23(3)	2.879(2)	156(3)		
		V				
O(5)-H(1)···O(8) <sup>#3</sup>	0.80(3)	2.09(3)	2.863(2)	162(3)		
O(6)-H(2)···O(5)	0.77(3)	2.05(3)	2.703(2)	143(2)		
$O(7)-H(3)\cdots O(4)^{#4}$	0.70(3)	1.94(3)	2.6323(19)	169(3)		

Table 4. Geometric parameters of hydrogen bonds for compounds IV and V\*

\* Symmetry transforms of equivalent atoms:  ${}^{\#1}x, -y + 3/2, z + 1/2; {}^{\#2}-x, -y + 1, -z + 1; {}^{\#3}x + 1/2, -y + 1/2, z + 1/2; {}^{\#4}-1, y, z.$ 

are 0.212, 0.215, and 0.246 Å longer than the standard  $Mo-OR_2$  bond (2.09 Å [26]).

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