# Synthesis and IR Spectral Study of MoO<sub>2</sub>Cl<sub>2</sub> Molecular Complexes with Acetoacetanilides. Crystal Structure of MoO<sub>2</sub>Cl<sub>2</sub> Complex with Acetoacet-2-Toluidine

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**Abstract**—A series of MoO<sub>2</sub>Cl<sub>2</sub> complexes with acetoacetanilide derivatives were synthesized and studied by IR spectroscopy. The crystal and molecular structures of the MoO<sub>2</sub>Cl<sub>2</sub> complex with acetoacet-2-toluidine (HL) were determined by X-ray diffraction analysis. The monoclinic crystals involved are a = 7.621(7) Å, b = 9.498(3) Å, c = 19.980(9) Å,  $\beta = 95.16(7)^{\circ}$ , V = 1440(2) Å<sup>3</sup>, Z = 4, and space group  $P2_1/n$ . The coordination polyhedron of the Mo atom is a distorted octahedron with two O oxoatoms in the *cis*-position relative to each other, two Cl atoms in the *trans*-positions, and two O atoms (HL) in the *trans*-positions to O(oxo).

Solutions of  $\beta$ -diketones and their nitrogen-containing derivatives ( $\beta$ -enamines, acetoacetanilides) represent equilibrium mixtures of various tautomers [1] and can be used to prepare complexes of different types: adducts (molecular complexes) and chelates [2, 3]. The type of compound formed is determined by the synthesis conditions and the nature of the reacting components. It was reported in [4] that the reaction of molybdenum dioxodichloride with  $\beta$ -diketones ( $\beta$ -DicH) in a benzene–petroleum ether medium at room temperature yields MoO<sub>2</sub>Cl<sub>2</sub> ·  $\beta$ -DicH adducts; however, their structures were not studied. Under more rigid conditions, MoO<sub>2</sub>( $\beta$ -Dic)<sub>2</sub> chelates are formed, whose structures were determined by various methods including X-ray diffraction [5–7].

With  $\beta$ -enamines (HL'), we obtained the molecular complexes MoO<sub>2</sub>Cl<sub>2</sub> · 2HL' [8, 9] only. The data obtained through X-ray diffraction and IR spectroscopy prove that, in molecular complexes, the neutral ligand molecules are coordinated by the molybdenum central atom in the aminoketone (enamine) tautomeric form through the oxygen atom of the carbonyl group. A similar coordination pattern was recently observed in the adducts of zirconium or titanium tetrachlorides with a number of  $\beta$ -aminovinylketones [10]. The results of IR spectral and conductometric studies showed that the sulfur-containing analogs of  $\beta$ -aminovinylketones are also coordinated by the central atom in the aminothione form through the sulfur atoms of the C=S group [11].

It was of interest to study the complexation of Lewis acids with acetanilides (HL) that formally combine the functions of  $\beta$ -diketones (**A**) and  $\beta$ -aminovinylketones (**B**) [12, 13], for which the following tautomeric forms

(A–D) can be suggested in a solution:



This paper reports the results of the study of the molybdenum dioxodichloride complexes with acetoacetanilide ( $\mathbf{R} = \mathbf{H}$ ,  $\mathbf{I}$ ) and its derivatives: acetoacet-2toluidine ( $\mathbf{R} = o$ -CH<sub>3</sub>,  $\mathbf{II}$ ), acetoacet-2-anisidine ( $\mathbf{R} = o$ -CH<sub>3</sub>O,  $\mathbf{III}$ ), and acetoacet-4-chloroanilide ( $\mathbf{R} = p$ -Cl,  $\mathbf{IV}$ ) in nonaqueous solvents. The structures of the isolated complexes are discussed in terms of IR spectral data. The structure of **II** was determined by X-ray diffraction.

#### **EXPERIMENTAL**

Synthesis of the complexes. A solution of 0.199 g (1 mmol) of  $MoO_2Cl_2$  in ethyl acetate was added under the process of stirring to a warm (35–40°C) solution containing 1 mmol of the corresponding anilide in 15 ml of the same solvent, and the mixture was allowed to stay at room temperature for 1 h for crystallization. The precipitated fine crystals of a lemon color were separated by filtration, washed with dehydrated ether, and dried in a flow of dry argon.

| No. | R                  | T <sub>m</sub> , °C | Empirical formula   | Conte<br>(anal. cale | ent, %<br>cd./found) | ν, cm <sup>-1</sup>        |                            |                       |          | mol <sup>-1</sup>                  |
|-----|--------------------|---------------------|---|----------------------|----------------------|----------------------------|----------------------------|-----------------------|----------|------------------------------------|
|     |                    |                     |   | Мо                   | Cl                   | ν(N–H)*                    | v(C=O)*                    | v(Mo–O <sub>L</sub> ) | v(Mo=O)  | $\mu_{,\Omega^{-1}} \mathrm{cm}^2$ |
| Ι   | Н                  | 195–196             | C <sub>10</sub> H <sub>11</sub> Cl <sub>2</sub> MoNO <sub>4</sub> | 25.35/25.51          | 18.97/18.86          | 3315<br>(3300, 3250)       | 1690 (1725,<br>1710, 1660) | 442                   | 945, 915 | 155                                |
| II  | o-CH <sub>3</sub>  | 188–190             | C <sub>11</sub> H <sub>13</sub> Cl <sub>2</sub> MoNO <sub>4</sub> | 24.43/24.60          | 18.06/18.18          | 3340<br>(3270, 3240)       | 1690 (1720,<br>1710, 1670) | 458                   | 950, 920 | 162                                |
| III | o-OCH <sub>3</sub> | 185–187             | C <sub>11</sub> H <sub>13</sub> Cl <sub>2</sub> MoNO <sub>5</sub> | 23.71/23.63          | 17.32/17.46          | 3310<br>(3270, 3250)       | 1678 (1720,<br>1710, 1675) | 415                   | 953, 920 | 158                                |
| IV  | p-Cl               | 203–205             | C <sub>10</sub> H <sub>10</sub> Cl <sub>3</sub> MoNO <sub>4</sub> | 23.34/23.39          | 26.07/25.91          | 3300 (3290,<br>3250, 3200) | 1687 (1720,<br>1710, 1657) | 430                   | 947, 915 | 155                                |

Table 1. Summary of physicochemical and IR spectral studies of the MoO<sub>2</sub>Cl<sub>2</sub> molecular complexes of acetoacetanilides

\* Frequencies for noncoordinated ligands are given in parentheses.

The metal content of the filtrate was determined by calcining the weighed sample of the complex to produce molybdenum trioxide (weight form) [14]. The chlorine content was determined using the Volhard method after treatment of the complex with alkali, followed by the neutralization of the solution.

**IR spectra** were recorded on an IKS-29 spectrometer with samples prepared as mineral oil mull.

The **conductivity** of the methanol solutions of the complexes  $(10^{-3} \text{ mol/l})$  was measured at 25°C on a conductometer with an ac working frequency of 1000 Hz using a bridge scheme in a temperature-controlled cell with platinized electrodes.

The results of the physicochemical and IR spectral studies of the complexes are given in Table 1.

The X-ray diffraction study of complex II was carried out on an Enraf-Nonius CAD-4 automated diffractometer (Mo $K_{\alpha}$  radiation,  $\lambda = 0.71073$  Å) at 293 K. The experimental data arrays were corrected for the Lorentz and polarization factors [15]. The structure was solved using the direct method (SHELX86 [16]). All nonhydrogen atoms were refined by the full-matrix leastsquares method in anisotropic approximation for  $F^2$ (SHELXL93 [17]). All hydrogen atoms were placed in the calculated positions (d(C-H) = 0.96 Å) and then refined in isotropic approximation. Crystallographic data, details of the experiment, and structure II refinement are given in Table 2. Coordinates of the nonhydrogen atoms and their equivalent isotropic thermal parameters are listed in Table 3. Bond lengths and angles are listed in Table 4.

The unit cell parameters for the crystal of acetoacet-2-toluidine (**V**) were determined on an Enraf-Nonius CAD-4 automated diffractometer (Mo $K_{\alpha}$  radiation,  $\lambda =$ 0.71073 Å) at room temperature. Unfortunately, the low quality of the crystal did not allow a full X-ray diffraction study. The crystallographic data for **V** are given in Table 2.

### **RESULTS AND DISCUSSION**

Unlike  $\beta$ -enamines [8, 9], acetoacetanilides react with MoO<sub>2</sub>Cl<sub>2</sub> in the ethyl acetate medium to form complexes of equimolecular compositions independent of the initial ratio of the reagents. The obtained complexes are lemon-colored crystals stable in dry air; they melt at high temperature without decomposition (Table 1). They form electrolyte solutions in polar solvents; the molar conductivity of the 10<sup>-3</sup> M methanol solutions is 150– 160  $\Omega^{-1}$  cm<sup>2</sup> mol<sup>-1</sup>, which is characteristic of three-ion electrolytes. Dissociation of the complexes in a methanol solution may be presented by the following scheme [11]:

$$MoO_2Cl_2 \cdot HL + (x + y)CH_3OH$$
$$\implies [MoO_2(HL)(CH_3OH)_x]^{2+} + Cl^{-}(CH_3OH)_y.$$

This is confirmed by the ready precipitation by silver nitrate of chloride ions from the methanol solutions of the complexes studied.

Electronic, vibrational, and NMR spectroscopy are widely used in determining the structures of metal complexes with  $\beta$ -diketones and their nitrogen-containing analogs. However, as was noted in [3, 18], the frequencies of vibrations of  $\beta$ -diketones and  $\beta$ -enamines are the complex functions of not only the force constants but also of the molecular geometry and steric features of the substituents, thus significantly complicating the interpretation of the data.

As is seen from Fig. 1, the IR spectrum of separate acetanilide is rather complicated; this is typical of prototropic equilibrium systems, in particular, of  $\beta$ -diketones and  $\beta$ -enamines [19]. The complexation is accompanied by the following changes in the IR spectra. A wide band of the stretching vibrations of the N–H bond of the ligand molecule (3300–3150 cm<sup>-1</sup>) remains unchanged upon complexation but is slightly shifted to higher frequencies by ~60 cm<sup>-1</sup>. The absorption bands of the carbonyl groups of different acetanilide tau-

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| Table 2. | Crystallographic | data and detail | s of the exp | periment and | structure | refinement | for <b>II</b> a | nd V |
|----------|------------------|-----------------|--------------|--------------|-----------|------------|-----------------|------|
|          |                  |                 |              |              |           |            |                 |      |

| Parameter                                     | II  | V                           |
|---|---|-----------------------------|
| Empirical formula                             | C <sub>11</sub> H <sub>13</sub> Cl <sub>2</sub> MoNO <sub>4</sub> | $C_{11}H_{13}NO_2$          |
| Molecular weight                              | 390.06  | 191.23                      |
| Crystal system                                | Monoclinic  | Monoclinic                  |
| Space group                                   | $P2_1/n$  | $P2_{1}/c$                  |
| a, Å  | 7.621(7)  | 7.494(7)                    |
| b, Å  | 9.498(3)  | 12.16(2)                    |
| <i>c</i> , Å                                  | 19.980(9)   | 10.816(8)                   |
| β, deg  | 95.16(7)  | 100.50(7)                   |
| <i>V</i> , Å <sup>3</sup>                     | 1440(2)   | 962(2)                      |
| Ζ   | 4   | 4                           |
| $\rho$ (calcd.), g/cm <sup>3</sup>            | 1.799   | 1.311                       |
| <i>F</i> (000)                                | 776   | 408                         |
| $\mu(MoK_{\alpha}), mm^{-1}$                  | 1.289   | 0.090                       |
| Crystal size, mm                              | 0.5 	imes 0.4 	imes 0.2   | $0.3 \times 0.2 \times 0.2$ |
| Scanning type/region for $\theta$ , deg       | ω/2.05–24.97  |                             |
| Intervals of reflection indexes               | $-9 \leq h \leq 9$  |                             |
|   | $-2 \leq k \leq 11$   |                             |
|   | $-3 \leq l \leq 23$   |                             |
| Number of measured reflections                | 3865  |                             |
| Number of unique reflections                  | 2536 ( $R_{\rm int} = 0.0171$ )                                   |                             |
| Reflections with $I > 2\sigma(I)$             | 2178  |                             |
| Absorption correction                         | ψ scan mode   |                             |
| Transmission, min/max                         | 0.4312/0.6480   |                             |
| Refined parameters                            | 225   |                             |
| <i>R</i> factors for $I > 2\sigma(I)$         | $R_1 = 0.0258, wR_2 = 0.0676$                                     |                             |
| for all reflections                           | $R_1 = 0.0342, wR_2 = 0.0718$                                     |                             |
| Quality for $F^2$                             | 1.088   |                             |
| Extinction coefficient                        | 0.0066(6)   |                             |
| Residual electronic density, min/max, $e/Å^3$ | -0.882/0.676  |                             |

| Atom  | x         | У       | z        | $U_{ m equiv}, { m \AA}^2$ | Atom  | x        | У       | z        | $U_{ m equiv}, { m \AA}^2$ |
|-------|-----------|---------|----------|----------------------------|-------|----------|---------|----------|----------------------------|
| Mo    | -10163(1) | 5883(1) | -2205(1) | 28(1)                      | C(3)  | -5379(4) | 5229(3) | -2339(2) | 34(1)                      |
| Cl(1) | -10598(1) | 4107(1) | -3011(1) | 47(1)                      | C(4)  | -6303(3) | 4810(3) | -1778(1) | 27(1)                      |
| Cl(2) | -8662(1)  | 7555(1) | -1487(1) | 51(1)                      | C(5)  | -5881(4) | 3784(3) | -654(1)  | 32(1)                      |
| O(1)  | -8016(2)  | 6422(2) | -2887(1) | 37(1)                      | C(6)  | -7392(4) | 4291(4) | -399(2)  | 41(1)                      |
| O(2)  | -7930(2)  | 4613(2) | -1823(1) | 31(1)                      | C(7)  | -7993(4) | 3638(4) | 156(2)   | 50(1)                      |
| O(3)  | -11472(2) | 5186(2) | -1654(1) | 41(1)                      | C(8)  | -7080(5) | 2548(5) | 457(2)   | 55(1)                      |
| O(4)  | -11441(3) | 7085(2) | -2631(1) | 44(1)                      | C(9)  | -5537(5) | 2096(4) | 216(2)   | 50(1)                      |
| N(1)  | -5294(3)  | 4431(3) | -1241(1) | 34(1)                      | C(10) | -4908(4) | 2695(3) | -345(1)  | 38(1)                      |
| C(1)  | -5539(5)  | 6436(4) | -3510(2) | 46(1)                      | C(11) | -3242(5) | 2176(5) | -605(2)  | 54(1)                      |
| C(2)  | -6478(4)  | 6103(3) | -2905(1) | 31(1)                      |       |          |         |          |                            |

| Table 3.       | Coordinates of non-hydrogen atoms ( $\times 10^4$ ) and their isotropic equivalent thermal parameters $U_{\text{equiv}}$ ( $\times$ | $10^3$ ) for com- |
|----------------|---|-------------------|
| plex <b>II</b> |   |                   |

| Bond        | <i>d</i> , Å | Bond         | <i>d</i> , Å | Bond           | d, Å     |
|-------------|--------------|--------------|--------------|----------------|----------|
| Mo-O(4)     | 1.682(2)     | O(2)–C(4)    | 1.249(3)     | C(5)–C(6)      | 1.387(4) |
| Mo-O(3)     | 1.685(2)     | N(1)–C(4)    | 1.313(4)     | C(6)–C(7)      | 1.384(5) |
| Mo-O(2)     | 2.168(2)     | N(1)–C(5)    | 1.432(4)     | C(7)–C(8)      | 1.357(5) |
| Mo-O(1)     | 2.281(2)     | C(1)–C(2)    | 1.492(4)     | C(8)–C(9)      | 1.379(5) |
| Mo–Cl(1)    | 2.336(1)     | C(2)–C(3)    | 1.490(4)     | C(9)–C(10)     | 1.382(4) |
| Mo-Cl(2)    | 2.365(1)     | C(3)–C(4)    | 1.496(4)     | C(10)–C(11)    | 1.498(5) |
| O(1)–C(2)   | 1.215(3)     | C(5)–C(10)   | 1.385(4)     |                |          |
| Angle       | ω, deg       | Angle        | ω, deg       | Angle          | ω, deg   |
| O(4)MoO(3)  | 104.3(1)     | O(2)MoCl(2)  | 80.70(8)     | N(1)C(4)C(3)   | 116.1(2) |
| O(4)MoO(2)  | 163.03(9)    | O(1)MoCl(2)  | 82.99(7)     | C(10)C(5)C(6)  | 121.7(3) |
| O(3)MoO(2)  | 92.5(1)      | Cl(1)MoCl(2) | 159.11(4)    | C(10)C(5)N(1)  | 119.4(3) |
| O(4)MoO(1)  | 87.6(1)      | C(2)O(1)Mo   | 135.1(2)     | C(6)C(5)N(1)   | 118.9(3) |
| O(3)MoO(1)  | 167.71(9)    | C(4)O(2)Mo   | 133.2(2)     | C(7)C(6)C(5)   | 118.9(3) |
| O(2)MoO(1)  | 75.69(8)     | C(4)N(1)C(5) | 125.7(2)     | C(8)C(7)C(6)   | 120.3(3) |
| O(4)MoCl(1) | 95.81(9)     | O(1)C(2)C(3) | 124.2(3)     | C(7)C(8)C(9)   | 120.2(3) |
| O(3)MoCl(1) | 96.39(8)     | O(1)C(2)C(1) | 120.3(3)     | C(8)C(9)C(10)  | 121.6(3) |
| O(2)MoCl(1) | 84.24(7)     | C(3)C(2)C(1) | 115.4(3)     | C(9)C(10)C(5)  | 117.2(3) |
| O(1)MoCl(1) | 79.30(7)     | C(2)C(3)C(4) | 117.9(2)     | C(9)C(10)C(11) | 120.8(3) |
| O(4)MoCl(2) | 94.4(1)      | O(2)C(4)N(1) | 121.4(3)     | C(5)C(10)C(11) | 122.0(3) |
| O(3)MoCl(2) | 98.64(8)     | O(2)C(4)C(3) | 122.4(2)     |                |          |

Table 4. Bond lengths and angles in structure II

tomers (~1725, 1710, and 1670–1660 cm<sup>-1</sup>) are shifted to the 1690–1680 cm<sup>-1</sup> range upon complexation. The intensities and positions of the absorption bands of skeletal vibrations of the conjugated bond system of the coordinated acetanilide molecules (1550–1100 cm<sup>-1</sup> range) become changed also. uted to the symmetric and asymmetric stretching vibrations of the Mo=O bonds of the *cis*-MoO<sub>2</sub> group [20], as well as a new intensive band in the 458–415 cm<sup>-1</sup> range, which can be attributed to the stretching vibrations of the Mo  $\leftarrow$  O=C (ligand) bond.

One should notice, in the low-frequency region, two intensive bands at 950–945 and 920–915  $cm^{-1}$  attrib-

Therefore, with a view to the results of the elemental analysis and IR spectral study and the data obtained [1, 3, 18–20], one can suggest that acetoacetanilides react



Fig. 1. IR spectra of (1) acetoacet-2-toluidine and (2) its complex with  $MoO_2Cl_2$  (absorption bands of mineral oil are marked with asterisks).

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Fig. 2. Molecular structure of complex II.

with molybdenum dioxodichloride like  $\beta$ -diketones [4] to yield equimolecular adducts with octahedral structure, in which the neutral ligand molecules are coordinated by two carbonyl oxygen atoms of the diketone tautomeric form A.

This suggestion is confirmed by the results of the Xray diffraction analysis of complex **II**. In this complex (Fig. 2), the Mo atom is coordinated by two O(3,4) oxoatoms in *cis*-position relative to each other, two Cl(1,2) chlorine atoms in *trans*-position, and two O(1,2) oxygen atoms in *trans*-positions to the O(3,4) atoms. This geometry of the metal coordination polyhedron conforms to the self-consistency rule [21], which states that *trans*-positions to the multiple-bonded ligands are preferably occupied by the neutral donor ligands (or ligand atoms) rather than by the acido ligands.

The average Mo=O(oxo) bond length in structure **II**  $(1.684(2) \pm 0.002 \text{ Å})$  is comparable to the average Mo=O bond length for 162 structures of the Mo(VI) dioxo complexes  $(1.704 \pm 0.028 \text{ Å})$  [22]. The Mo-O(HL) bond lengths in II (Mo-O(2) 2.168(2) Å and Mo–O(1) 2.281(2) Å) vary within the standard range of the Mo-O<sub>trans</sub> distances (2.11–2.38 Å [23]). These bonds are notably elongated in comparison with standard values (Mo-O 1.94-2.08 Å [23]), due to the transeffect of the multiple-bonded oxoligand. It is noteworthy that the Mo– $\hat{O}_{trans}$  bond lengths in structure **II** are different: the Mo–O(1) bond, adjacent to the CMe group, is 0.113 Å longer than the Mo-O(2) bond joining the  $CN(H)C_6H_4Me$  fragment. The C=O bond lengths are also unequal. Due to partial delocalization of the electronic density over the amide fragment, the O(2)-C(4) bond (1.249(3) Å) adjacent to the N(1)-C(4) multiple bond (1.313(4) Å) is 0.034 Å shorter than

the O(1)–C(2) bond (1.215(3) Å). The average Mo– Cl(1, 2) bond length is  $2.350(1) \pm 0.001$  Å.

The coordination polyhedron of the Mo atom in structure **II** exhibits distortions typical of the dioxo complexes of  $d^0$ -metals of Groups V–VII. As a result of the repulsion of the electronic clouds of the multiple-bonded oxoligands, the O(3)MoO(4) angle increases to 104.3(1)° as compared to the standard value of 90° and the opposite O(1)MoO(2) angle decreases to 75.69(8)°. The *trans*-angles Cl(1)MoCl(2) 159.11(4)° and O(oxo)MoO(HL) 165.37(9)  $\pm$  2.34° (average) also deviate notably from the value of 180° in a standard octahedron. The Mo atom is shifted from the center of the MoO<sub>4</sub>Cl<sub>2</sub> octahedron toward the O(3)–O(4) edge by 0.390 Å.

Upon coordination of the metal by the HL bidentate ligand, the six-membered ring Mo-O(1)-C(2)-C(3)-C(4)-O(2) closes in the sofa conformation with a deviation of 0.415 Å of the O(2) atom from the plane of the other five atoms, coplanar within ±(0.001-0.064) Å (average 0.036 Å).

As was mentioned above, the low quality of the crystals of acetoacet-2-toluidine V did not allow a full X-ray diffraction study. Earlier [24], the crystal structure of the unsubstituted analog of V, i.e., the *N*-phenyl amide of the acetoacetic acid (VI), was determined. If we assume that the conformations of molecules V and VI are similar, then one can conclude that the configuration of the initial HL molecule somewhat changes during coordination to metal and closure of the sixmembered metal cycle. The angles between the planar fragments C(5)–C(10) and N(1), C(3,4), O(2) in complex II are only slightly changed in comparison with a free molecule (41.8° for II and 33.2°, 39.0° for two

independent molecules **VI**, respectively), whereas the angles between the fragments N(1), C(3,4), O(2) and C(1,3), O(1) in structures **II** and **VI** are quite different (21.8° and 97.6°, 84.1°, respectively). The characteristic torsion angles C(4)N(1)C(5)C(10) (138.8° in **II** and 31.4°, 39.7° in **VI**), C(4)N(1)C(5)C(6) ( $-42.7^{\circ}$  in **II** and 156.5°,  $-145.1^{\circ}$  in **VI**), and N(1)C(4)C(3)C(2) ( $-169.4^{\circ}$  in **II** and 101.2°,  $-95.0^{\circ}$  in **VI**) also differ notably.

Structure **II** contains no intermolecular contacts that are less than the sums of the van der Waals radii of the corresponding atoms.

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