

**(Dimethyl sulfoxide- $\kappa$ O)[3-hydroxy-2-hydroxymethyl-2-(3-methoxy-2-oxido-benzylideneamino- $\kappa^2$ O<sup>2</sup>,N)propanolato- $\kappa$ O]dioxomolybdenum(VI). Corrigendum****Yan Sui, Xiao-Niu Fang,\* Qiu-Yan Luo, Hong-Mei Chen and Meng-Qiang Zhou**

JiangXi Province Key Laboratory of Coordination Chemistry, College of Chemistry &amp; Chemical Engineering, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China

Correspondence e-mail: ysui@163.com

Received 19 February 2010; accepted 26 February 2010

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.022;  $wR$  factor = 0.064; data-to-parameter ratio = 12.7.

The crystal structure of the title compound in the paper by Sui, Fang, Luo, Chen & Zhou [*Acta Cryst.* (2006), **E62**, m1994–m1996] has been re-refined to allow for identification of a disordered dimethyl sulfoxide ligand.

The structure reported by Sui *et al.* (2006) has been re-refined. The compound was originally determined by Rao *et al.* [*J. Chem. Soc. Dalton Trans.* (1998), 2383] and has been re-determined here to a significantly higher precision of the lattice parameters [ $a = 14.3130$  (7) Å,  $b = 9.2596$  (5) Å and  $c = 14.8563$  (7) Å here *versus*  $a = 14.305$  (3) Å,  $b = 9.249$  (2) Å and  $c = 14.860$  (3) Å reported by Rao *et al.*], bond lengths and s.u. values [*e.g.* Mo1–O6 = 1.6937 (17) Å here *versus* Mo1–O6 1.697 (4) Å reported by Rao *et al.*;  $R = 0.022$  here *versus*  $R = 0.050$  reported by Rao *et al.*]. The results of the current redetermination allow the identification of a disordered dimethyl sulfoxide ligand and a clarification of the nature of the intra- and intermolecular hydrogen bonding.

**Experimental***Crystal data*

[Mo(C <sub>12</sub> H <sub>15</sub> NO <sub>5</sub> )O <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> OS)]	$V = 1779.74$ (15) Å <sup>3</sup>
$M_r = 459.32$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 14.3130$ (7) Å	$\mu = 0.90$ mm <sup>-1</sup>
$b = 9.2596$ (5) Å	$T = 295$ K
$c = 14.8563$ (7) Å	$0.56 \times 0.39 \times 0.35$ mm
$\beta = 115.324$ (1)°	

*Data collection*

Bruker APEXII area-detector diffractometer	10668 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2004)	3278 independent reflections
$T_{\min} = 0.632$ , $T_{\max} = 0.746$	3056 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.024$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.022$	40 restraints
$wR(F^2) = 0.064$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.36$ e Å <sup>-3</sup>
3278 reflections	$\Delta\rho_{\text{min}} = -0.44$ e Å <sup>-3</sup>
259 parameters	

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1 $\cdots$ O5	0.93	2.22	2.874 (3)	127
C14–H14E $\cdots$ O7 <sup>i</sup>	0.96	2.45	3.343 (4)	154
C8–H8A $\cdots$ O6 <sup>ii</sup>	0.96	2.54	3.475 (4)	164
C11–H11A $\cdots$ O7 <sup>iii</sup>	0.97	2.57	3.534 (3)	170
O5–H5A $\cdots$ O1 <sup>iii</sup>	0.82	2.02	2.834 (2)	175
O5–H5A $\cdots$ O2 <sup>iii</sup>	0.82	2.53	2.970 (2)	115
O4–H4 $\cdots$ O3 <sup>iv</sup>	0.82	1.99	2.801 (2)	171

Symmetry codes: (i)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{5}{2}$ ; (ii)  $x, y - 1, z$ ; (iii)  $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (iv)  $-x + 1, -y + 2, -z + 2$ .

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* (Bruker, 2004); data reduction: *APEX2* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG9064).

**References**

- Bruker (2004). *APEX2* (Version 1.22) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA
- Rao, C. P., Sreedhara, A., Rao, P. V., Verghese, M. B., Rissanen, K., Kolehmainen, E., Lokanath, N. K., Sridhar, M. A. & Prasad, J. S. (1998). *J. Chem. Soc. Dalton Trans.* pp. 2383.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sui, Y., Fang, X.-N., Luo, Q.-Y., Chen, H.-M. & Zhou, M.-Q. (2006). *Acta Cryst.* **E62**, m1994–m1996.

Yan Sui, Xiao-Niu Fang,\*  
 Qiu-Yan Luo, Hong-Mei Chen  
 and Meng-Qiang Zhou

JiangXi Province Key Laboratory of Coordination  
 Chemistry, College of Chemistry & Chemical  
 Engineering, JingGangShan University, 343009  
 Ji'an, JiangXi, People's Republic of China

Correspondence e-mail: ysui@163.com

Key indicators

Single-crystal X-ray study  
 T = 296 K  
 Mean  $\sigma(C-C)$  = 0.003 Å  
 R factor = 0.027  
 wR factor = 0.086  
 Data-to-parameter ratio = 15.0

For details of how these key indicators were  
 automatically derived from the article, see  
<http://journals.iucr.org/e>.

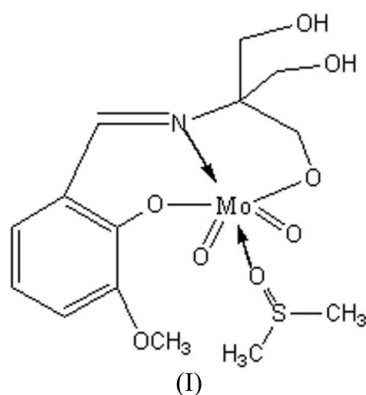
(Dimethyl sulfoxide- $\kappa O$ )[3-hydroxy-2-hydroxy-  
 methyl-2-(3-methoxy-2-oxidobenzylideneamino-  
 $\kappa^2 O^2, N$ )propanolato- $\kappa O$ ]dioxomolybdenum(VI)

Received 11 July 2006  
 Accepted 25 July 2006

The title complex,  $[MoO_2(C_{12}H_{15}NO_5)(C_2H_6OS)]$ , adopts a distorted octahedral geometry made up of one coordinated dimethyl sulfoxide molecule and a Schiff base dianion which acts in a tridentate manner through the alkoxo O, phenolate O and azomethine N atoms. The crystal structure is stabilized by strong intermolecular  $O-H \cdots O$  and weak  $C-H \cdots O$  interactions.

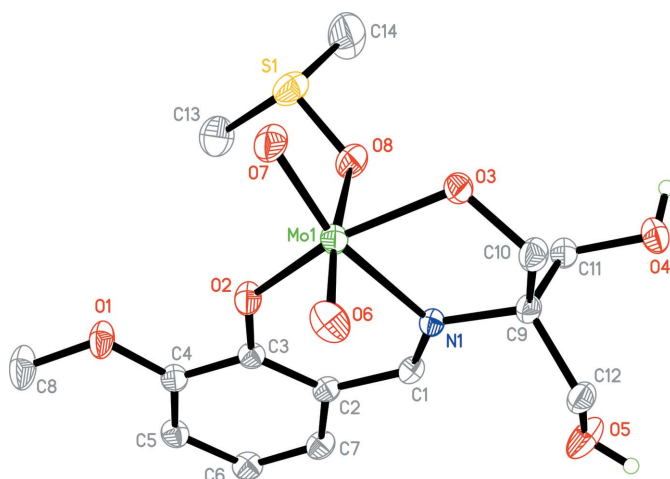
Comment

The chemistry of transition metal ion complexes of polyhydroxy molecules containing an imine/amine group is important in the biomimetic study of metalloproteins (Dey *et al.*, 2002). The coordination and biomimetic chemistry of complexes of Schiff bases derived from [tris(hydroxymethyl)amino]methane (THAM) have gained attention in the case of metal ions such as vanadium, manganese, iron and copper (Asgedom *et al.*, 1995; Cornman *et al.*, 1992; Crans *et al.*, 1993). However, not addressed in the literature is the case of molybdenum, an important oxometal species in biological systems (Hille, 1996). We report here the synthesis and X-ray crystal structure analysis of the title complex, (I), a new dioxomolybdenum(VI) complex of a Schiff base derived from THAM.

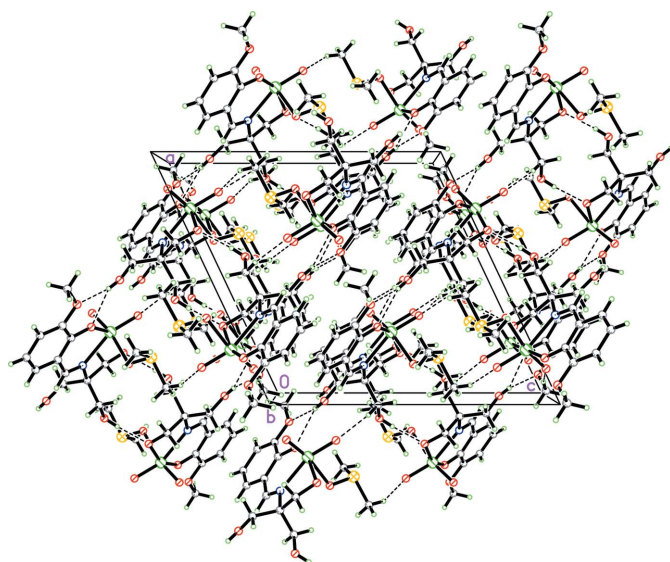


The title complex, (I), adopts a distorted octahedral geometry. The azomethine N atom, phenolate O atom ( $O_{ph}$ ) and one of the alkoxo O atoms ( $O_{alk}$ ) of the Schiff base bind to the metal. In the coordination polyhedron, the O3 (alkoxo), O2 (phenolate), N1 (imino) and O7 (from *cis*- $MoO_2$ ) are nearly coplanar, with a mean deviation of 0.022 (2) Å. The distance of the Mo atom to the plane is 0.302 (2) Å. Atoms O6 (from *cis*- $MoO_2$ ) and O8 (from DMSO), coordinating with Mo, lie above and below the plane.

Both the  $Mo=O$  distances and the  $O=Mo=O$  angle are in the usual range for *cis*- $MoO_2$  complexes (Holm, 1987). The



**Figure 1**  
The molecular structure of (I), showing 30% probability displacement ellipsoids. All H atoms on carbon have been omitted for clarity.



**Figure 2**  
A packing diagram of (I), viewed along the *b* axis; hydrogen bonds are shown as dashed lines.

imine N atom is *trans* to one Mo=O. The Mo—O<sub>alk</sub> and Mo—O<sub>ph</sub> distance are also in the usual ranges.

Adjacent molecules are held together by classical intermolecular hydrogen bonds (Table 2) and weak intermolecular C—H...O hydrogen bonding. These link the molecules into an infinite layer (Fig. 2).

## Experimental

To the Schiff base 3-hydroxy-2-(2-hydroxy-3-methoxybenzylideneamino)-2-hydroxymethylpropanol (3.205 g, 12.57 mmol) derived from THAM and 3-methoxysalicylaldehyde in methanol (75 ml) was added MoO<sub>2</sub>(acac)<sub>2</sub> (acac is acetylacetonate; 4.120 g, 12.57 mmol). The reagents were allowed to react at room temperature for 1 d. The volume was reduced to about 10 ml, and diethyl ether (20 ml) was added to precipitate the compound as a yellow solid. The solid was washed twice with diethyl ether and dried under

vacuum to obtain the title complex, (I) (m.p. 454–456 K). Analysis calculated for C<sub>12</sub>H<sub>17</sub>MoNO<sub>8</sub>: C 36.10, H 4.29, Mo 24.03, N 3.51%; found: C 36.70, H 4.40, Mo 23.37, N 3.41%. Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature of a DMSO–methanol (1:1) solution.

## Crystal data

[Mo(C<sub>12</sub>H<sub>15</sub>NO<sub>5</sub>)O<sub>2</sub>(C<sub>2</sub>H<sub>6</sub>OS)]  
*M<sub>r</sub>* = 459.32  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 14.3130 (7) Å  
*b* = 9.2596 (5) Å  
*c* = 14.8563 (7) Å  
 $\beta$  = 115.324 (1)°  
*V* = 1779.74 (15) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.714 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.90 mm<sup>-1</sup>  
*T* = 296 (2) K  
 Block, yellow  
 0.37 × 0.27 × 0.14 mm

## Data collection

Bruker APEX-II area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 2004)  
*T<sub>min</sub>* = 0.753, *T<sub>max</sub>* = 0.882

11097 measured reflections  
 3461 independent reflections  
 3214 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.024  
 $\theta_{\max}$  = 26.0°

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.027  
*wR* (*F*<sup>2</sup>) = 0.086  
*S* = 1.00  
 3461 reflections  
 231 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.031P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.003$   
 $\Delta\rho_{\max} = 1.20 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.49 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

Mo1—N1	2.289 (2)	Mo1—O6	1.6958 (19)
Mo1—O2	1.9619 (16)	Mo1—O7	1.7028 (18)
Mo1—O3	1.9380 (15)	Mo1—O8	2.345 (2)
O7—Mo1—O8	85.34 (9)	O6—Mo1—N1	93.20 (8)
O7—Mo1—O3	96.81 (8)	O3—Mo1—O8	82.51 (7)
O7—Mo1—O2	101.69 (8)	O3—Mo1—O2	152.29 (7)
O7—Mo1—N1	159.84 (10)	O2—Mo1—O8	78.67 (8)
O6—Mo1—O8	168.57 (8)	O2—Mo1—N1	81.72 (6)
O6—Mo1—O7	105.96 (11)	N1—Mo1—O8	75.78 (7)
O6—Mo1—O3	97.58 (8)	C1—N1—Mo1	127.44 (15)
O6—Mo1—O2	96.94 (9)	O3—Mo1—N1	74.03 (6)

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4...O3 <sup>i</sup>	0.82	1.99	2.801 (2)	173
O5—H5A...O1 <sup>ii</sup>	0.82	2.02	2.835 (2)	170
O5—H5A...O2 <sup>ii</sup>	0.82	2.51	2.966 (2)	116

Symmetry codes: (i)  $-x + 1, -y + 2, -z + 2$ ; (ii)  $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$ .

H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.97 (methylene) and 0.96 Å (methyl), and *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C) for methyl H atoms and 1.2*U*<sub>eq</sub>(C) for other H atoms. The maximum electron-density peak lies 1.14 Å from atom S1, which may be due to unresolved disorder of this atom.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *APEX2*; program(s) used to refine structure: *APEX2*; molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2*.

We gratefully acknowledge financial support from the Natural Science Foundation of JiangXi Province (grant No. 0620029), the Science and Technology Bureau of Ji'an City (grant No. 200528), and Jinggangshan University.

## References

- Asgedom, G., Sreedhara, A. & Rao, C. P. (1995). *Polyhedron*, **14**, 1873–1879.
- Bruker (2004). *APEX2*. Version 1.22. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cornman, C. R., Colpas, G. J., Hoeschele, J. D., Kampf, J. & Pecoraro, V. L. (1992). *J. Am. Chem. Soc.* **114**, 9925–9933.
- Crans, D. C., Chen, H., Anderson, O. P. & Miller, M. M. (1993). *J. Am. Chem. Soc.* **115**, 6769–6776.
- Dey, M., Rao, C. P., Saarenketo, P. K. & Rissanen, K. (2002). *Inorg. Chem. Commun.* **5**, 380–383.
- Hille, R. (1996). *Chem. Rev.* **96**, 2757–2816.
- Holm, H. R. (1987). *Chem. Rev.* **87**, 1401–1449.