Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Dimethyl sulfoxide-κO)[3-hydroxy-2-hydroxymethyl-2-(3-methoxy-2-oxidobenzylideneamino-κ^2O^2,N)propanolato-κO]dioxomolybdenum(VI). Corrigendum

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Received 19 February 2010; accepted 26 February 2010

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–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), E**62**, m1994–m1996] has been rerefined to allow for identification of a disordered dimethyl sulfoxide ligand.

The structure reported by Sui *et al.* (2006) has been rerefined. The compound was originally determined by Rao *et al.* [*J. Chem. Soc. Dalton Trans.* (1998), 2383] and has been redetermined 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

$[Mo(C_{12}H_{15}NO_5)O_2(C_2H_6OS)]$
M_r = 459.32
Monoclinic, $P2_1/n$
a = 14.3130 (7) Å
b = 9.2596 (5) Å
c = 14.8563 (7) Å
β = 115.324 (1)°
V = 1779.74 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.90 mm^{-1}
T = 295 K
0.56 × 0.39 × 0.35 mm

Data collection

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

Refinement

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

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O5	0.93	2.22	2.874 (3)	127
C14—H14E···O7^{i}	0.96	2.45	3.343 (4)	154
C8—H8A···O6^{ii}	0.96	2.54	3.475 (4)	164
C11—H11A···O7^{iii}	0.97	2.57	3.534 (3)	170
O5—H5A···O1^{iii}	0.82	2.02	2.834 (2)	175
O5—H5A···O2^{iii}	0.82	2.53	2.970 (2)	115
O4—H4···O3^{iv}	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).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sui, Y., Fang, X.-N., Luo, Q.-Y., Chen, H.-M. & Zhou, M.-Q. (2006). *Acta Cryst.* E**62**, m1994–m1996.

supporting information

Acta Cryst. (2010). E**66**, e23 [https://doi.org/10.1107/S1600536810007476]

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Hall symbol: -P 2yn
$a = 14.3130$ (7) Å
$b = 9.2596$ (5) Å
$c = 14.8563$ (7) Å
$\beta = 115.324$ (1)°
$V = 1779.74$ (15) Å^3
$Z = 4$
$F(000) = 936$
$D_x = 1.714$ Mg m^{-3}
Melting point = 454–456 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11097 reflections
$\theta = 2.6$–26.0°
$\mu = 0.90$ mm^{-1}
$T = 295$ K
Block, yellow
0.56 × 0.39 × 0.35 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
$T_{min} = 0.632$, $T_{max} = 0.746$
10668 measured reflections
3278 independent reflections
3056 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -17\rightarrow16$
$k = -11\rightarrow10$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.064$
$S = 1.07$
3278 reflections
259 parameters
40 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.9103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.61630 (15)	0.7171 (2)	0.82021 (14)	0.0306 (4)	
H1	0.5551	0.7275	0.7628	0.037*	
C2	0.67331 (15)	0.5841 (2)	0.83147 (14)	0.0296 (4)	
C3	0.76286 (15)	0.5507 (2)	0.91746 (14)	0.0290 (4)	
C4	0.81292 (15)	0.4178 (2)	0.92185 (15)	0.0320 (4)	
C5	0.77698 (17)	0.3257 (2)	0.84100 (16)	0.0353 (5)	
H5	0.8110	0.2390	0.8441	0.042*	
C6	0.69022 (17)	0.3620 (2)	0.75494 (16)	0.0382 (5)	
H6	0.6675	0.3005	0.7002	0.046*	
C7	0.63806 (16)	0.4877 (2)	0.75039 (15)	0.0358 (5)	
H7	0.5788	0.5097	0.6932	0.043*	
C8	0.9572 (2)	0.2667 (3)	1.0142 (2)	0.0606 (8)	
H8A	0.9176	0.1814	1.0104	0.091*	
H8B	1.0184	0.2670	1.0760	0.091*	
H8C	0.9764	0.2679	0.9597	0.091*	
C9	0.57775 (15)	0.9537 (2)	0.85896 (14)	0.0315 (4)	
C10	0.64007 (17)	1.0616 (2)	0.94075 (15)	0.0370 (5)	
H10A	0.6914	1.1084	0.9242	0.044*	
H10B	0.5944	1.1355	0.9457	0.044*	
C11	0.47619 (16)	0.9150 (2)	0.86463 (17)	0.0376 (5)	
H11A	0.4393	0.8422	0.8152	0.045*	
H11B	0.4904	0.8766	0.9300	0.045*	
C12	0.55921 (17)	1.0195 (2)	0.75826 (15)	0.0379 (5)	
H12A	0.5287	1.1146	0.7523	0.045*	
H12B	0.6247	1.0302	0.7542	0.045*	
Mo1	0.779330 (13)	0.829629 (19)	1.036557 (12)	0.03081 (8)	
N1	0.64298 (13)	0.82184 (17)	0.88262 (12)	0.0280 (3)	
O1	0.89683 (12)	0.39109 (19)	1.00918 (11)	0.0467 (4)	
O2	0.80380 (11)	0.63679 (17)	0.99656 (10)	0.0384 (3)	
O3	0.68969 (11)	0.98850 (16)	1.03313 (10)	0.0350 (3)	
O4	0.41554 (14)	1.0420 (2)	0.84657 (14)	0.0535 (4)	
H4	0.3791	1.0373	0.8766	0.080*	
O5	0.49290 (16)	0.9313 (2)	0.67962 (12)	0.0589 (5)	
H5A	0.4619	0.9810	0.6300	0.088*	
O6	0.85527 (13)	0.92141 (19)	0.99470 (13)	0.0498 (4)	

O7	0.84674 (14)	0.82180 (19)	1.16266 (12)	0.0506 (4)	
O8	0.65402 (13)	0.69377 (18)	1.06088 (12)	0.0463 (4)	
C13	0.7091 (3)	0.4309 (4)	1.1317 (3)	0.0645 (9)	0.9402 (18)
H13D	0.6536	0.3982	1.0710	0.077*	0.9402 (18)
H13E	0.7147	0.3685	1.1854	0.077*	0.9402 (18)
H13F	0.7727	0.4291	1.1247	0.077*	0.9402 (18)
C14	0.5638 (3)	0.5787 (5)	1.1629 (3)	0.0811 (10)	0.9402 (18)
H14D	0.5338	0.6697	1.1671	0.097*	0.9402 (18)
H14E	0.5749	0.5216	1.2205	0.097*	0.9402 (18)
H14F	0.5179	0.5285	1.1040	0.097*	0.9402 (18)
C13A	0.662 (5)	0.419 (7)	1.110 (4)	0.056 (6)	0.0598 (18)
H13A	0.6969	0.4216	1.0676	0.068*	0.0598 (18)
H13B	0.6114	0.3433	1.0892	0.068*	0.0598 (18)
H13C	0.7112	0.4022	1.1776	0.068*	0.0598 (18)
C14A	0.630 (4)	0.631 (6)	1.219 (4)	0.059 (4)	0.0598 (18)
H14A	0.6970	0.5990	1.2657	0.071*	0.0598 (18)
H14B	0.5784	0.5938	1.2383	0.071*	0.0598 (18)
H14C	0.6276	0.7345	1.2184	0.071*	0.0598 (18)
S1	0.68357 (6)	0.60903 (8)	1.15748 (5)	0.0488 (2)	0.9402 (18)
S1A	0.6059 (10)	0.5697 (14)	1.1039 (9)	0.060 (2)	0.0598 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0303 (10)	0.0313 (11)	0.0266 (9)	−0.0003 (8)	0.0087 (8)	0.0030 (8)
C2	0.0318 (10)	0.0281 (10)	0.0271 (9)	−0.0020 (8)	0.0109 (8)	−0.0002 (8)
C3	0.0321 (9)	0.0269 (10)	0.0269 (9)	−0.0024 (8)	0.0117 (8)	−0.0043 (8)
C4	0.0308 (10)	0.0318 (11)	0.0305 (10)	0.0006 (8)	0.0103 (8)	−0.0035 (8)
C5	0.0393 (11)	0.0292 (12)	0.0380 (11)	0.0002 (8)	0.0172 (9)	−0.0064 (8)
C6	0.0452 (12)	0.0334 (12)	0.0323 (11)	−0.0064 (9)	0.0132 (9)	−0.0115 (9)
C7	0.0364 (11)	0.0349 (12)	0.0287 (10)	−0.0050 (9)	0.0069 (8)	−0.0028 (9)
C8	0.0528 (15)	0.0461 (16)	0.0591 (16)	0.0222 (12)	0.0011 (12)	−0.0087 (13)
C9	0.0351 (10)	0.0262 (10)	0.0321 (10)	0.0046 (8)	0.0133 (8)	0.0039 (8)
C10	0.0439 (12)	0.0265 (11)	0.0379 (11)	0.0037 (9)	0.0150 (9)	0.0025 (9)
C11	0.0373 (11)	0.0373 (12)	0.0394 (11)	0.0064 (9)	0.0176 (9)	0.0053 (9)
C12	0.0444 (12)	0.0319 (12)	0.0356 (11)	0.0045 (9)	0.0154 (9)	0.0078 (9)
Mo1	0.03081 (12)	0.02818 (12)	0.02761 (12)	0.00253 (6)	0.00691 (8)	−0.00596 (6)
N1	0.0285 (8)	0.0256 (9)	0.0286 (8)	0.0023 (6)	0.0110 (7)	0.0026 (6)
O1	0.0446 (9)	0.0392 (9)	0.0382 (8)	0.0160 (7)	0.0006 (7)	−0.0099 (7)
O2	0.0379 (8)	0.0342 (8)	0.0292 (7)	0.0085 (6)	0.0012 (6)	−0.0102 (6)
O3	0.0410 (8)	0.0293 (8)	0.0324 (7)	0.0061 (6)	0.0135 (6)	−0.0027 (6)
O4	0.0528 (10)	0.0543 (11)	0.0618 (11)	0.0231 (8)	0.0326 (9)	0.0136 (9)
O5	0.0757 (12)	0.0455 (11)	0.0326 (8)	−0.0047 (9)	0.0014 (8)	0.0095 (7)
O6	0.0439 (9)	0.0449 (10)	0.0640 (11)	−0.0054 (7)	0.0264 (8)	−0.0097 (8)
O7	0.0563 (10)	0.0460 (11)	0.0318 (8)	0.0119 (7)	0.0021 (7)	−0.0098 (7)
O8	0.0544 (9)	0.0405 (9)	0.0445 (9)	0.0027 (7)	0.0215 (8)	0.0132 (7)
C13	0.077 (2)	0.0439 (13)	0.071 (2)	0.0125 (18)	0.0298 (19)	0.0151 (15)
C14	0.0865 (18)	0.083 (2)	0.095 (2)	0.0244 (18)	0.059 (2)	0.043 (2)

C13A	0.069 (9)	0.049 (9)	0.050 (9)	0.002 (9)	0.025 (8)	0.014 (8)
C14A	0.071 (5)	0.057 (5)	0.058 (5)	0.007 (5)	0.036 (5)	0.024 (5)
S1	0.0633 (4)	0.0416 (4)	0.0375 (3)	−0.0005 (3)	0.0177 (3)	0.0088 (3)
S1A	0.068 (3)	0.053 (3)	0.054 (3)	0.003 (3)	0.022 (3)	0.015 (3)

Geometric parameters (Å, º)

C1—N1	1.282 (3)	C12—H12A	0.9700
C1—C2	1.448 (3)	C12—H12B	0.9700
C1—H1	0.9300	Mo1—O6	1.6937 (17)
C2—C3	1.403 (3)	Mo1—O7	1.7028 (16)
C2—C7	1.407 (3)	Mo1—O3	1.9383 (14)
C3—O2	1.331 (2)	Mo1—O2	1.9602 (15)
C3—C4	1.411 (3)	Mo1—N1	2.2861 (17)
C4—O1	1.362 (2)	Mo1—O8	2.3419 (17)
C4—C5	1.381 (3)	O4—H4	0.8200
C5—C6	1.390 (3)	O5—H5A	0.8200
C5—H5	0.9300	O8—S1	1.5280 (17)
C6—C7	1.369 (3)	O8—S1A	1.606 (12)
C6—H6	0.9300	C13—S1	1.767 (4)
C7—H7	0.9300	C13—H13D	0.9600
C8—O1	1.422 (3)	C13—H13E	0.9600
C8—H8A	0.9600	C13—H13F	0.9600
C8—H8B	0.9600	C14—S1	1.772 (4)
C8—H8C	0.9600	C14—H14D	0.9600
C9—N1	1.485 (2)	C14—H14E	0.9600
C9—C12	1.530 (3)	C14—H14F	0.9600
C9—C10	1.531 (3)	C13A—S1A	1.59 (6)
C9—C11	1.534 (3)	C13A—H13A	0.9600
C10—O3	1.419 (2)	C13A—H13B	0.9600
C10—H10A	0.9700	C13A—H13C	0.9600
C10—H10B	0.9700	C14A—S1A	1.69 (5)
C11—O4	1.417 (3)	C14A—H14A	0.9600
C11—H11A	0.9700	C14A—H14B	0.9600
C11—H11B	0.9700	C14A—H14C	0.9600
C12—O5	1.409 (3)		

N1—C1—C2	125.56 (18)	O5—C12—H12B	109.5
N1—C1—H1	117.2	C9—C12—H12B	109.5
C2—C1—H1	117.2	H12A—C12—H12B	108.1
C3—C2—C7	119.53 (19)	O6—Mo1—O7	105.95 (9)
C3—C2—C1	122.88 (18)	O6—Mo1—O3	97.53 (7)
C7—C2—C1	117.55 (18)	O7—Mo1—O3	96.80 (7)
O2—C3—C2	123.88 (18)	O6—Mo1—O2	96.88 (8)
O2—C3—C4	117.29 (17)	O7—Mo1—O2	101.78 (7)
C2—C3—C4	118.83 (18)	O3—Mo1—O2	152.32 (6)
O1—C4—C5	124.42 (19)	O6—Mo1—N1	93.20 (7)
O1—C4—C3	115.21 (17)	O7—Mo1—N1	159.85 (8)

C5—C4—C3	120.36 (19)	O3—Mo1—N1	74.06 (6)
C4—C5—C6	120.3 (2)	O2—Mo1—N1	81.65 (6)
C4—C5—H5	119.8	O6—Mo1—O8	168.61 (7)
C6—C5—H5	119.8	O7—Mo1—O8	85.31 (8)
C7—C6—C5	120.31 (19)	O3—Mo1—O8	82.51 (6)
C7—C6—H6	119.8	O2—Mo1—O8	78.78 (7)
C5—C6—H6	119.8	N1—Mo1—O8	75.82 (6)
C6—C7—C2	120.55 (19)	C1—N1—C9	119.23 (17)
C6—C7—H7	119.7	C1—N1—Mo1	127.61 (14)
C2—C7—H7	119.7	C9—N1—Mo1	113.08 (12)
O1—C8—H8A	109.5	C4—O1—C8	117.75 (18)
O1—C8—H8B	109.5	C3—O2—Mo1	138.18 (13)
H8A—C8—H8B	109.5	C10—O3—Mo1	116.08 (12)
O1—C8—H8C	109.5	C11—O4—H4	109.5
H8A—C8—H8C	109.5	C12—O5—H5A	109.5
H8B—C8—H8C	109.5	S1—O8—S1A	41.6 (5)
N1—C9—C12	112.79 (16)	S1—O8—Mo1	119.88 (10)
N1—C9—C10	104.78 (15)	S1A—O8—Mo1	158.9 (5)
C12—C9—C10	108.35 (17)	S1A—C13A—H13A	109.5
N1—C9—C11	108.02 (16)	S1A—C13A—H13B	109.5
C12—C9—C11	112.00 (17)	H13A—C13A—H13B	109.5
C10—C9—C11	110.68 (17)	S1A—C13A—H13C	109.5
O3—C10—C9	109.68 (17)	H13A—C13A—H13C	109.5
O3—C10—H10A	109.7	H13B—C13A—H13C	109.5
C9—C10—H10A	109.7	S1A—C14A—H14A	109.5
O3—C10—H10B	109.7	S1A—C14A—H14B	109.5
C9—C10—H10B	109.7	H14A—C14A—H14B	109.5
H10A—C10—H10B	108.2	S1A—C14A—H14C	109.5
O4—C11—C9	108.29 (18)	H14A—C14A—H14C	109.5
O4—C11—H11A	110.0	H14B—C14A—H14C	109.5
C9—C11—H11A	110.0	O8—S1—C13	106.01 (15)
O4—C11—H11B	110.0	O8—S1—C14	103.85 (14)
C9—C11—H11B	110.0	C13—S1—C14	98.9 (2)
H11A—C11—H11B	108.4	C13A—S1A—O8	111 (2)
O5—C12—C9	110.72 (18)	C13A—S1A—C14A	111 (3)
O5—C12—H12A	109.5	O8—S1A—C14A	103 (2)
C9—C12—H12A	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O4	0.97	2.50	2.911 (3)	105
C1—H1···O5	0.93	2.22	2.874 (3)	127
C14—H14*E*···O7^{i}	0.96	2.45	3.343 (4)	154
C8—H8*A*···O6^{ii}	0.96	2.54	3.475 (4)	164
C11—H11*A*···O7^{iii}	0.97	2.57	3.534 (3)	170
O5—H5*A*···O1^{iii}	0.82	2.02	2.834 (2)	175

O5—H5*A*···O2^{iii}	0.82	2.53	2.970 (2)	115
O4—H4···O3^{iv}	0.82	1.99	2.801 (2)	171

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+5/2; (ii) *x*, *y*−1, *z*; (iii) *x*−1/2, −*y*+3/2, *z*−1/2; (iv) −*x*+1, −*y*+2, −*z*+2.