

Wen Bin Yan,^a Yuan Fu Deng,^a
Feng Zhang Zhang,^b Zhao Hui
Zhou,^a Hui Lin Wan^a and
Seik Weng Ng^{c*}^aDepartment of Chemistry, Xiamen University,
Xiamen 361005, People's Republic of China,
^bDepartment of Biology, Xiamen University,
Xiamen 361005, People's Republic of China,
and ^cInstitute of Postgraduate Studies, University
of Malaya, 50603 Kuala Lumpur, MalaysiaCorrespondence e-mail:
h1nswen@umcsd.um.edu.my

Key indicators

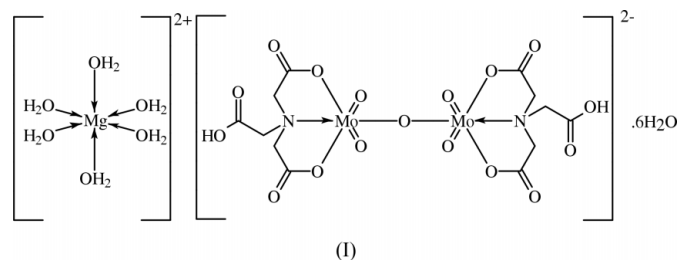
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.028
wR factor = 0.076
Data-to-parameter ratio = 14.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Hexaaquamagnesium bis{*trans*-[nitrilotriacetato(2-)- $\kappa^3\text{O}^1, \text{N}, \text{O}^2$]- μ -oxo-*cis*-dioxomolybdate(VI)] hexahydrateBoth the cation and anion in the title compound, $[\text{Mg}(\text{H}_2\text{O})_6][\text{Mo}_2\text{O}_5(\text{C}_6\text{H}_7\text{NO}_6)_2] \cdot 6\text{H}_2\text{O}$, lie on centers of symmetry, and their metals are both six-coordinate in octahedral environments.

Received 30 November 2001

Accepted 4 December 2001

Online 14 December 2001

Comment

Oxomolybdate(VI) complexes are of interest as possible models for molybdenum sites in metalloenzymes (Chan *et al.*, 1993; Hille, 1996; Stiefel, 1977). Among the complexes, those chelated by tridentate ligands derive their stability through the binding of the ligand to vacant coordination sites (Gebreyes *et al.*, 1985). The deprotonated nitrilotriacetato ligand, which functions as a tetradentate entity in a number of metal complexes, is only tridentate in the dipyridinium (Matsumoto *et al.*, 1984), monohydrated bis(tetrabutylammonium) (Liu *et al.*, 1990) and octahydrated disodium μ -oxobis(hydrogen-nitrilotriacetato-*cis*-dioxomolybdates) (Knobler *et al.*, 1980, 1983). In the peroxo complex, potassium nitrilotriacetato-(oxo)(peroxo)molybdate monohydrate, the ligand behaves as a tetradentate chelate (Won *et al.*, 1994). On the other hand, as the hexaaquamagnesium(II) cation has been used to balance the charges of a number of organic (Arranz Mascarós *et al.*, 2000; Castellari *et al.*, 1999; Kariuki *et al.*, 1994; Solans, Font-Altaba, Aguilo *et al.*, 1983) and inorganic (Coiro & Mazza, 1991; Kariuki & Jones, 1989; Maslen *et al.*, 1988; Solans, Font-Altaba, Oliva & Herrera, 1983) derivatives, we have used this dication as counter-ion in the title compound, (I).Both the cation and anion lies on centers of symmetry; for the anion, this symmetry requires the Mo—O—Mo unit to be linear. The Mo—O bond distance is similar to that [1.880 (1) Å] found in the sodium salt (Knobler *et al.*, 1983), which is also centrosymmetric. In the related dipotassium tetrasodium oxobis(citratodioxomolybdate), the Mo—O—Mo unit is bent [Mo—O—Mo = 144.7 (2)°; Zhou *et al.*, 1997]. In the title compound, the anions and the water-coordinated cations are linked by hydrogen bonds into a three-dimensional network motif.

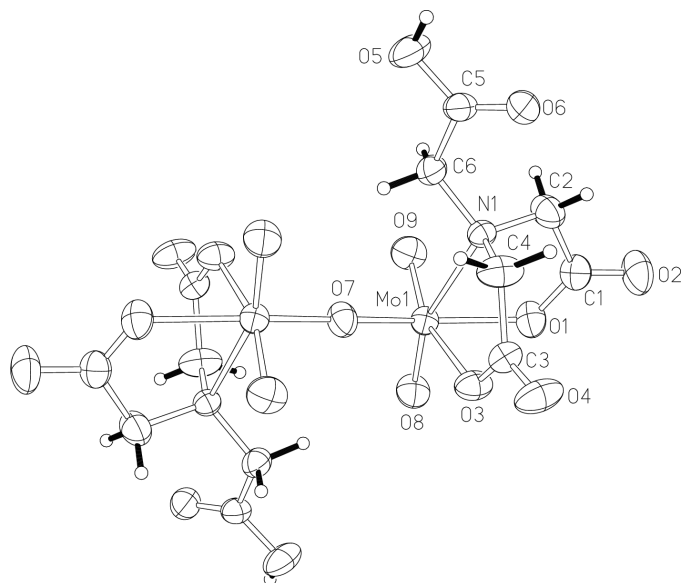


Figure 1
ORTEP (Johnson, 1976) plot of the anion of the title compound with ellipsoids at the 50% probability level.

Experimental

Magnesium molybdate (10 mmol) dissolved in water (10 ml) was added to nitrilotriacetic acid (20 mmol) dissolved in water (5 ml) and the mixture was stirred for several hours. The solution was concentrated to about 10 ml; colorless crystals of the title hydrate, (I), separated from the solution in 35% yield when it was set aside for several days.

Crystal data

$[\text{Mg}(\text{H}_2\text{O})_6][\text{Mo}_2\text{O}_5(\text{C}_6\text{H}_7\text{NO}_6)_2] \cdot 6\text{H}_2\text{O}$
 $M_r = 890.63$
 Triclinic, $P\bar{1}$
 $a = 6.4787(3) \text{ \AA}$
 $b = 9.2555(6) \text{ \AA}$
 $c = 13.9995(6) \text{ \AA}$
 $\alpha = 91.014(4)^\circ$
 $\beta = 101.315(3)^\circ$
 $\gamma = 106.592(4)^\circ$
 $V = 786.44(7) \text{ \AA}^3$

$Z = 1$
 $D_x = 1.881 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 12.5\text{--}13.0^\circ$
 $\mu = 0.93 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Irregular block, colorless
 $0.58 \times 0.58 \times 0.36 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: empirical via ψ scans (North *et al.*, 1968)
 $T_{\min} = 0.618$, $T_{\max} = 0.716$
 3339 measured reflections
 3079 independent reflections
 2877 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.010$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = 0 \rightarrow 7$
 $k = -11 \rightarrow 10$
 $l = -17 \rightarrow 16$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.076$
 $S = 1.11$
 3079 reflections
 215 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.3336P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.52 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Mo1—O1	2.076 (2)	Mo1—N1	2.418 (2)
Mo1—O3	2.172 (2)	Mg1—O1w	2.045 (2)
Mo1—O7	1.880 (1)	Mg1—O2w	2.053 (2)
Mo1—O8	1.695 (2)	Mg1—O3w	2.089 (3)
Mo1—O9	1.694 (2)		
O1w—Mg1—O2w	89.1 (1)	O2w—Mg1—O3w	89.3 (1)
O1w—Mg1—O3w	91.7 (1)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O5—H5 \cdots O5w	0.84 (1)	1.80 (2)	2.607 (3)	162 (4)
O1w—H1w1 \cdots O4	0.85	1.87	2.718 (3)	176
O1w—H1w2 \cdots O6 ⁱ	0.86	1.89	2.737 (3)	174
O2w—H2w1 \cdots O4w	0.84	1.86	2.699 (4)	174
O2w—H2w2 \cdots O2 ⁱⁱ	0.85	2.00	2.802 (4)	160
O3w—H3w1 \cdots O6w	0.83	1.88	2.701 (4)	174
O3w—H3w2 \cdots O3w ⁱⁱⁱ	0.83	2.18	2.989 (5)	165
O4w—H4w2 \cdots O2 ⁱ	0.85	2.13	2.945 (4)	160
O4w—H4w1 \cdots O2 ⁱⁱⁱ	0.84	2.28	3.039 (4)	151
O5w—H5w1 \cdots O5 ^{iv}	0.84	2.48	3.104 (4)	131
O5w—H5w1 \cdots O9 ^v	0.84	2.32	3.011 (3)	140
O5w—H5w2 \cdots O6w ^{vi}	0.85	2.34	3.178 (4)	170
O6w—H6w2 \cdots O3	0.85	2.12	2.937 (3)	164
O6w—H6w1 \cdots O4 ^{vii}	0.85	2.14	2.972 (4)	167

Symmetry codes: (i) $x, 1 + y, z$; (ii) $1 + x, 1 + y, z$; (iii) $1 - x, 2 - y, 2 - z$; (iv) $2 - x, -y, 1 - z$; (v) $1 - x, -y, 1 - z$; (vi) $1 + x, y - 1, z$; (vii) $x - 1, y, z$.

The acid H atom was located and refined. The H atoms of the water molecules were located in difference maps, but these were not refined. For the O3w water, one of its H atoms is disordered over two positions.

Data collection: CAD-4 VAX/PC Fortran System (Enraf–Nonius, 1988); cell refinement: CAD-4 VAX/PC Fortran System; data reduction: XCAD4 (Harms, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

We thank the Ministry of Science and Technology of China, the National Science Foundation of China (Nos. 29933040, 20021002 and 39970176) and the University of Malaya (No. F0758/2001A) for supporting this work. The last author thanks the Third World Academy of Sciences for a travel grant to China.

References

- Arranz Mascarós, P., Cobo Domingo, J., Godino Salido, M., Gutiérrez Valero, M. D., López Garzón, R. & Low, J. N. (2000). *Acta Cryst.* **C56**, e4–e5.
 Castellari, C., Comelli, F. & Ottani, S. (1999). *Acta Cryst.* **C55**, 1054–1056.
 Chan, M. K., Kim, J. & Rees, D. C. (1993). *Science*, **260**, 792–794.
 Coiro, V. M. & Mazza, F. (1991). *Acta Cryst.* **C47**, 1169–1173.
 Enraf–Nonius (1988). *CAD-4 VAX/PC Fortran System*. Enraf–Nonius, Scientific Instruments Division, PO Box 483, 2600 AL Delft, The Netherlands.
 Gebreyes, K., Shaikh, S. N. & Zubieta, J. (1985). *Acta Cryst.* **C41**, 871–873.
 Harms, K. (1997). *XCAD4*. University of Marburg, Germany.

- Hille, R. (1996). *Chem. Rev.* **96**, 2757–2816.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kariuki, B. M. & Jones, W. (1989). *Acta Cryst.* **C45**, 1297–1299.
- Kariuki, B. M., Valim, J. B., Jones, W. & King, J. (1994). *Acta Cryst.* **C50**, 1665–1667.
- Knobler, C.B., Penfold, B. R., Robinson, W. T., Wilkins, C. J. & Yong, S. H. (1980). *J. Chem. Soc. Dalton Trans.* pp. 248–252.
- Knobler, C. B., Robinson, W. T., Wilkins, C. J. & Wilson, A. J. (1983). *Acta Cryst.* **C39**, 443–445.
- Liu, S. C., Ma, L. D., McGowty, D. & Zubieta, J. (1990). *Polyhedron*, **9**, 1541–1553.
- Maslen, E. N., Ridout, S. C., Watson, K. J. & Moore, F. H. (1988). *Acta Cryst.* **C44**, 409–412.
- Matsumoto, K., Marutani, Y. & Ooi, S.-I. (1984). *Bull. Chem. Soc. Jpn*, **57**, 2671–2672.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Solans, X., Font-Altaba, M., Aguiló, M., Solans, J. & Domenech, V. (1983). *Acta Cryst.* **C39**, 1488–1490.
- Solans, X., Galí, S. Font-Altaba, M., Oliva, J. & Herrera, J. (1983). *Acta Cryst.* **C39**, 438–440.
- Stiefel, E. I. (1977). *Prog. Inorg. Chem.* **22**, 1–223.
- Won, T.-J., Sudam, B. M. & Thompson, R. C. (1994). *Inorg. Chem.* **33**, 3804–3810.
- Zhou, Z. H., Wan, H. L. & Tsai, K. R. (1997). *Polyhedron*, **16**, 75–79.