metal-organic papers

Hexaaquamagnesium bis{*trans*-[nitrilotriacetato(2–)-κ³O¹,N,O²]-μ-oxo-*cis*-dioxomolybdate(VI)} hexahydrate

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Both the cation and anion in the title compound, $[Mg(H_2O)_6][Mo_2O_5(C_6H_7NO_6)_2]\cdot 6H_2O$, lie on centers of symmetry, and their metals are both six-coordinate in octahedral environments.

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).

(I)
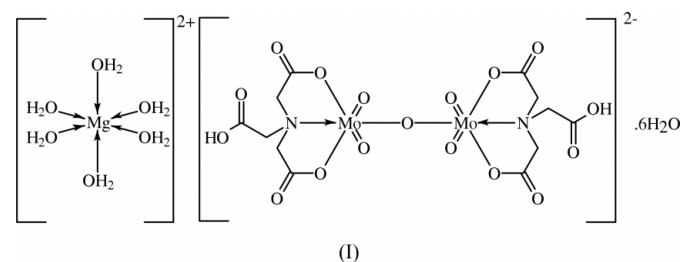
Both the cation and anion lies on centers of symmetry; for the anion, this symmetry requires the Mo—O—Mo unit to be linear. The Mo—O bond distance is similar to that [1.880 (1) Å] found in the sodium salt (Knobler *et al.*, 1983), which is also centrosymmetric. In the related dipotassium tetrasodium 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.
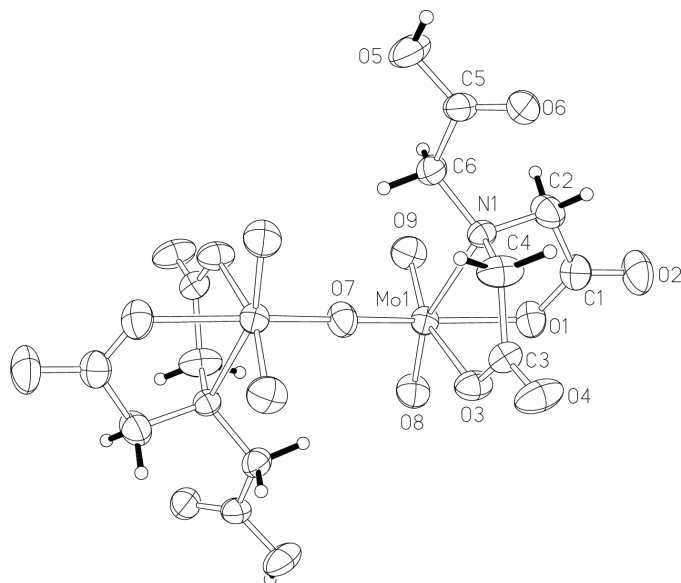
Figure 1
*ORTEP*II (Johnson, 1976) plot of the anion of the title compound with ellipsoids at the 50% probability level.

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

$[Mg(H_2O)_6][Mo_2O_5(C_6H_7NO_6)_2]\cdot$ $6H_2O$	$Z = 1$
	$D_x = 1.881$ Mg m^{-3}
$M_r = 890.63$	Mo $K\alpha$ radiation
Triclinic, $P\bar{1}$	Cell parameters from 25
$a = 6.4787$ (3) Å	reflections
$b = 9.2555$ (6) Å	$\theta = 12.5–13.0°$
$c = 13.9995$ (6) Å	$\mu = 0.93$ mm^{-1}
$\alpha = 91.014$ (4)°	$T = 293$ (2) K
$\beta = 101.315$ (3)°	Irregular block, colorless
$\gamma = 106.592$ (4)°	$0.58 \times 0.58 \times 0.36$ mm
$V = 786.44$ (7) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.010$
	$\theta_{max} = 26.0°$
ω scans	$h = 0 \rightarrow 7$
Absorption correction: empirical *via* ψ scans (North *et al.*, 1968)	$k = -11 \rightarrow 10$
	$l = -17 \rightarrow 16$
$T_{min} = 0.618$, $T_{max} = 0.716$	3 standard reflections
3339 measured reflections	frequency: 60 min
3079 independent reflections	intensity decay: none
2877 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.028$	$+ 0.3336P]$
$wR(F^2) = 0.076$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
3079 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
215 parameters	$\Delta\rho_{min} = -0.52$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

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 (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O5w	0.84 (1)	1.80 (2)	2.607 (3)	162 (4)
O1w—H1w1···O4	0.85	1.87	2.718 (3)	176
O1w—H1w2···O6[i]	0.86	1.89	2.737 (3)	174
O2w—H2w1···O4w	0.84	1.86	2.699 (4)	174
O2w—H2w2···O2[ii]	0.85	2.00	2.802 (4)	160
O3w—H3w1···O6w	0.83	1.88	2.701 (4)	174
O3w—H3w2···O3w[iii]	0.83	2.18	2.989 (5)	165
O4w—H4w2···O2[i]	0.85	2.13	2.945 (4)	160
O4w—H4w1···O2[iii]	0.84	2.28	3.039 (4)	151
O5w—H5w1···O5[iv]	0.84	2.48	3.104 (4)	131
O5w—H5w1···O9[v]	0.84	2.32	3.011 (3)	140
O5w—H5w2···O6w[vi]	0.85	2.34	3.178 (4)	170
O6w—H6w2···O3	0.85	2.12	2.937 (3)	164
O6w—H6w1···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: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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