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Hexaaquamagnesium(II) benzene-1,3-dioxyacetate trihydrate

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Key indicators

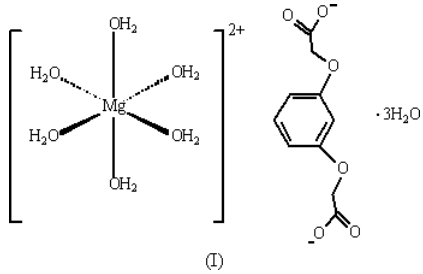
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.097
Data-to-parameter ratio = 14.7

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

The title complex, $[Mg(H_2O)_6](1,3\text{-BDOA})\cdot 3H_2O$ (1,3-$BDOA^{2-}$ = benzene-1,3-dioxyacetate, $C_{10}H_8O_6^{2-}$), consists of an $[Mg(H_2O)_6]^{2+}$ cation, a benzene-1,3-dioxyacetate dianion and three uncoordinated water molecules. The Mg^{II} atom is coordinated by six water molecules, forming a slightly distorted octahedral coordination. A two-dimensional supramolecular network structure is constructed by hydrogen bonds.

Comment

Phenylenedioxydiacetic acids, which have been known to show biological activities, are multidentate flexible ligands with versatile bonding modes. In contrast to metal complexes of benzene-1,2-dioxyacetic acid or benzene-1,4-dioxyacetic acid (Gao *et al.*, 2004; Liu *et al.*, 2004; McCann *et al.*, 1995, 1996; Kennard *et al.*, 1986), reports of structures of complexes with benzene-1,3-dioxyacetic acid are rare. Recently, we reported the crystal structure of a one-dimensional chain Zn^{II} polymer, $\{[Zn(C_{10}H_8O_6)(H_2O)_2]\cdot(H_2O)_2\}_n$ (Gao *et al.*, 2004), in which the zinc(II) ion displays a four-coordinate distorted tetrahedral geometry and benzene-1,3-dioxyacetate acts as the bridging ligand. In the present study, the title complex, $[Mg(H_2O)_6](1,3\text{-BDOA})\cdot 3H_2O$ (1,3-$BDOA^{2-}$ = benzene-1,3-dioxyacetate), (I), was obtained by the reaction of magnesium perchlorate hexahydrate, imidazole and sodium benzene-1,3-dioxyacetate in an aqueous solution. We report here the synthesis and structure of (I).



(I)

As shown in Fig. 1, the asymmetric unit of (I) consists of an $[Mg(H_2O)_6]^{2+}$ cation, a benzene-1,3-dioxyacetate dianion and three water molecules, which are linked by intermolecular hydrogen bonds. The Mg^{II} atom is coordinated by six water molecules, forming a distorted octahedral coordination [Mg—O = 2.048 (1)–2.117 (1) Å]. The oxyacetate groups and the benzene ring are essentially coplanar, with C7—O10—C9—C10 and C3—O9—C2—C1 torsion angles of −177.3 (1) and 165.0 (1)°, respectively. The cations and anions are linked by four O—H···O hydrogen bonds through the carboxylate O

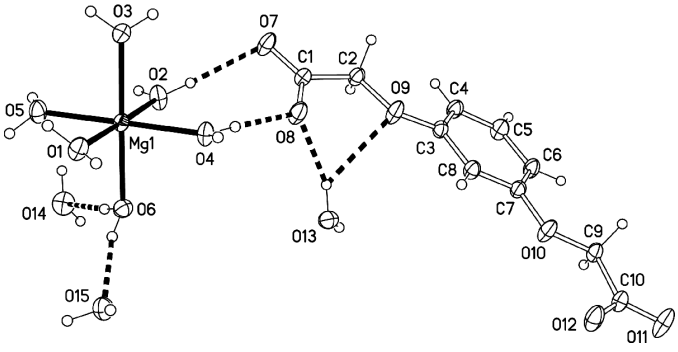


Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound, with 30% probability ellipsoids. Dashed lines represent hydrogen bonds.

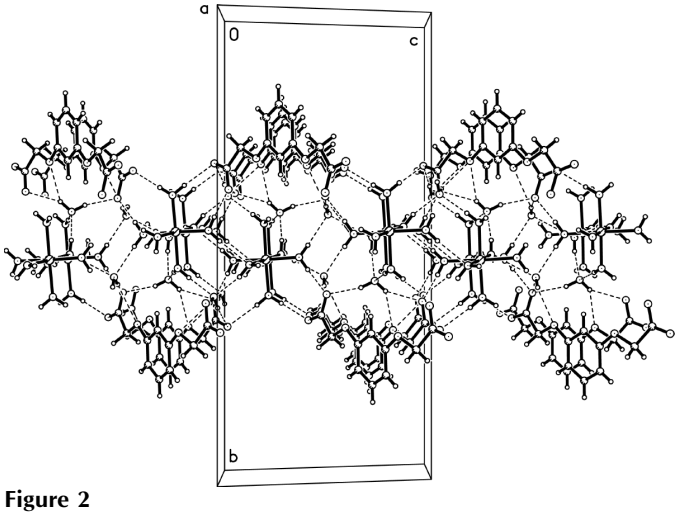


Figure 2
The hydrogen-bonded chain structure of the title complex.

atoms with coordinated water molecules, resulting in a one-dimensional chain along the *c* direction. A two-dimensional supermolecular network is constructed by hydrogen bonds in the *ac* plane (Fig. 2 and Table 2).

Experimental

Benzene-1,3-dioxyacetic acid was prepared following the method described for the synthesis of benzene-1,2-dioxyacetic acid by Mirci (1990). The title complex was prepared by the addition of magnesium perchlorate hexahydrate (20 mmol) and imidazole (20 mmol) to an aqueous solution of benzene-1,3-dioxyacetic acid (20 mmol), and the pH was adjusted to 7 with 0.1 *M* sodium hydroxide. Colorless crystals were separated from the filtered solution after several days. Analysis calculated for $C_{10}H_{26}MgO_{15}$: C 29.25, H 6.38%; found: C 29.01, H 6.49%.

Crystal data

$[Mg(H_2O)_6](C_{10}H_8O_6)\cdot 3H_2O$
M_r = 410.62
Monoclinic, $P2_1/n$
a = 6.134 (1) Å
b = 26.020 (5) Å
c = 12.028 (2) Å
β = 102.64 (3)°
V = 1873.3 (6) Å^3
Z = 4
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12616 reflections
θ = 3.5–27.4°
μ = 0.17 mm^{-1}
T = 293 (2) K
Prism, colorless
0.39 × 0.26 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.937, T_{max} = 0.971
17782 measured reflections
4240 independent reflections
3547 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.4°
$h = -7 \rightarrow 7$
$k = -33 \rightarrow 33$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.097
S = 1.09
4240 reflections
289 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.4786P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mg1—O1	2.051 (1)	Mg1—O4	2.048 (1)
Mg1—O2	2.059 (1)	Mg1—O5	2.055 (1)
Mg1—O3	2.114 (1)	Mg1—O6	2.117 (1)
O1—Mg1—O2	178.36 (5)	O4—Mg1—O2	88.69 (4)
O1—Mg1—O3	88.20 (5)	O4—Mg1—O3	88.81 (5)
O1—Mg1—O5	85.25 (5)	O4—Mg1—O5	178.18 (5)
O1—Mg1—O6	91.18 (5)	O4—Mg1—O6	89.74 (5)
O2—Mg1—O3	91.61 (5)	O5—Mg1—O2	93.12 (5)
O2—Mg1—O6	89.05 (5)	O5—Mg1—O3	90.91 (5)
O3—Mg1—O6	178.39 (5)	O5—Mg1—O6	90.52 (5)
O4—Mg1—O1	92.94 (5)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O13^{i}	0.853 (9)	1.876 (9)	2.728 (2)	177 (2)
O1—H12···O15^{ii}	0.844 (9)	1.962 (9)	2.803 (2)	174 (2)
O2—H13···O11^{iii}	0.855 (9)	1.839 (9)	2.694 (2)	179 (2)
O2—H14···O7	0.849 (9)	2.067 (9)	2.913 (2)	175 (2)
O3—H15···O6^{ii}	0.851 (9)	2.04 (1)	2.854 (2)	161 (2)
O3—H16···O13^{ii}	0.859 (9)	1.813 (9)	2.648 (2)	163 (2)
O4—H17···O8	0.853 (9)	1.797 (9)	2.649 (2)	176 (2)
O4—H18···O3^{iv}	0.845 (9)	2.08 (1)	2.926 (2)	176 (2)
O5—H19···O12^{iii}	0.851 (9)	1.81 (1)	2.657 (2)	174 (2)
O5—H20···O14^{v}	0.843 (9)	2.057 (9)	2.897 (2)	173 (2)
O6—H21···O14	0.848 (9)	1.941 (9)	2.787 (2)	175 (2)
O6—H22···O15	0.847 (9)	1.90 (1)	2.738 (2)	168 (2)
O13—H23···O8	0.848 (9)	1.95 (1)	2.745 (2)	156 (2)
O13—H23···O9	0.848 (9)	2.42 (2)	3.047 (2)	132 (2)
O13—H24···O7^{vi}	0.846 (9)	1.919 (9)	2.751 (2)	167 (2)
O14—H25···O10^{vii}	0.849 (9)	2.53 (2)	3.138 (2)	129 (2)
O14—H25···O12^{vii}	0.849 (9)	2.02 (1)	2.837 (2)	162 (2)
O14—H26···O11^{iii}	0.845 (9)	2.59 (1)	3.381 (2)	156 (2)
O15—H27···O10^{viii}	0.846 (9)	2.65 (2)	3.242 (2)	128 (2)
O15—H27···O12^{viii}	0.846 (9)	1.955 (9)	2.779 (2)	165 (2)
O15—H28···O8^{i}	0.847 (9)	2.11 (1)	2.856 (2)	147 (2)
O15—H28···O9^{i}	0.847 (9)	2.44 (1)	3.174 (2)	145 (2)

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

Water H atoms were located in a difference map and refined as riding, with O—H and H···H distance restraints of 0.85 (1) and 1.39 (1) Å, respectively, and U_{iso}(H) = 1.5U_{eq}(O). All other H atoms were placed in calculated positions [C—H = 0.93 (aromatic) or 0.97 Å (aliphatic)] and refined using a riding model [U_{iso}(H) = 1.2U_{eq}(C)].

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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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