metal-organic compounds

Diaquabis(2-methyl-1*H*-imidazol-3-ium-4,5-dicarboxylato-$\kappa^2 O,O'$)magnesium

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The title compound, $[Mg(C_6H_5N_2O_4)_2(H_2O)_2]$, was prepared by reaction of $Mg(NO_3)_2$ and 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid under hydrothermal conditions. The Mg^{II} atom lies on an inversion centre and displays a distorted octahedral coordination geometry. An extended three-dimensional network of intermolecular O—H$\cdots$O and N—H$\cdots$O hydrogen bonds stabilizes the crystal structure.

Related literature

For the crystal structures of metal complexes with *N*-heterocyclic carboxylic acids, see: Nie *et al.* (2007); Liang *et al.* (2002); Net *et al.* (1989); Zeng *et al.* (2008).
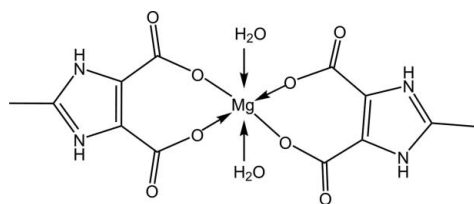
Experimental

Crystal data

$[Mg(C_6H_5N_2O_4)_2(H_2O)_2]$	a = 4.943 (2) Å
M_r = 398.58	b = 8.750 (6) Å
Triclinic, $P\bar{1}$	c = 9.621 (6) Å
α = 109.18 (3)°	Mo $K\alpha$ radiation
β = 95.142 (17)°	μ = 0.18 mm^{-1}
γ = 93.14 (2)°	T = 292 K
V = 389.9 (4) Å^3	0.30 × 0.25 × 0.20 mm
Z = 1	

Data collection

Rigaku SCXmini diffractometer	4002 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	1767 independent reflections
T_{min} = 0.948, T_{max} = 0.967	1308 reflections with $I > 2\sigma(I)$
	R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061	125 parameters
$wR(F^2)$ = 0.213	H-atom parameters constrained
S = 1.19	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
1767 reflections	$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O4^i	0.95	1.78	2.696 (4)	161
N2—H2$\cdots$O2^{ii}	0.95	1.81	2.727 (4)	162
O5—H5$B\cdots$O2^{iii}	0.93	2.23	3.155 (4)	172
O5—H5$B\cdots$O1^{iii}	0.93	2.36	2.961 (4)	122
O5—H5$A\cdots$O3^{iv}	0.87	1.98	2.841 (4)	170

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC*.

References

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supporting information

Diaquabis(2-methyl-1*H*-imidazol-3-ium-4,5-dicarboxylato-*κ²O,O′*)magnesium

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S1. Comment

Recently, the study of metal complexes with *N*-heterocyclic carboxylic acids has been given considerable attention (Nie *et al.*, 2007; Liang *et al.*, 2002; Net *et al.*, 1989; Zeng *et al.*, 2008). In this paper, we report on the synthesis and crystal structure of the title compound, which was obtained by the hydrothermal reaction of $Mg(NO_3)_2$ with 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid.

As shown in Fig. 1, the magnesium(II) atom, which lies on an inversion centre, adopts a distorted octahedral coordination, with the equatorial plane provided by four O atoms from two organic ligands [Mg1–O1 = 2.011 (2) Å; Mg1–O3 = 2.036 (2) Å] and the axial sites occupied by the O atoms of two water molecules [Mg1–O5 = 2.110 (3) Å]. The seven-membered chelate ring assumes an envelope-like conformation, with atom Mg1 displaced by 0.4353 (4) Å from the mean plane of the remaining atoms of the ring. The crystal structure is stabilized by intermolecular O—H⋯O and N—H⋯O hydrogen bonds (Table 1), forming an extended three-dimensional network (Fig. 2).

S2. Experimental

Colourless single crystals of title compound were obtained by hydrothermal treatment of $Mg(NO_3)_2$ (1 mmol), 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid (1 mmol) and water (5 ml) over 4 days at 368 K. Yield: 67% (based on $Mg(NO_3)_2$.

S3. Refinement

The water H atoms and H atoms connected to N were located from a difference Fourier map but not refined [U_{iso}(H)=1.5U_{eq}(O, N)]. The methyl H atoms were placed at calculated positions and refined as riding, with C—H = 0.96 Å, and with U_{iso}(H) = 1.5U_{eq}(C).
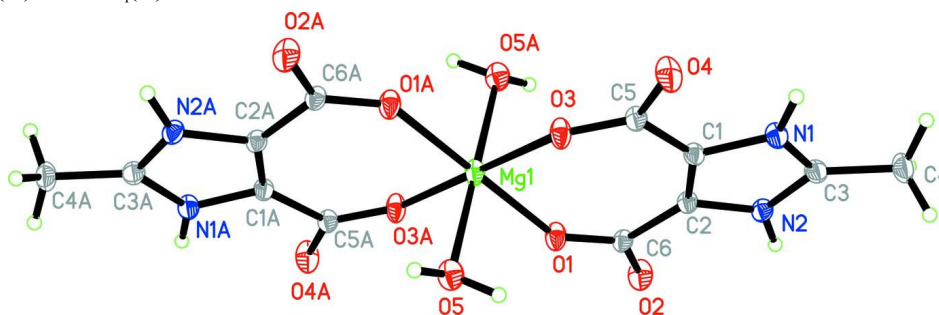
Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms with suffix A are related to atoms with no suffix by 1-x, 2-y, 2-z.
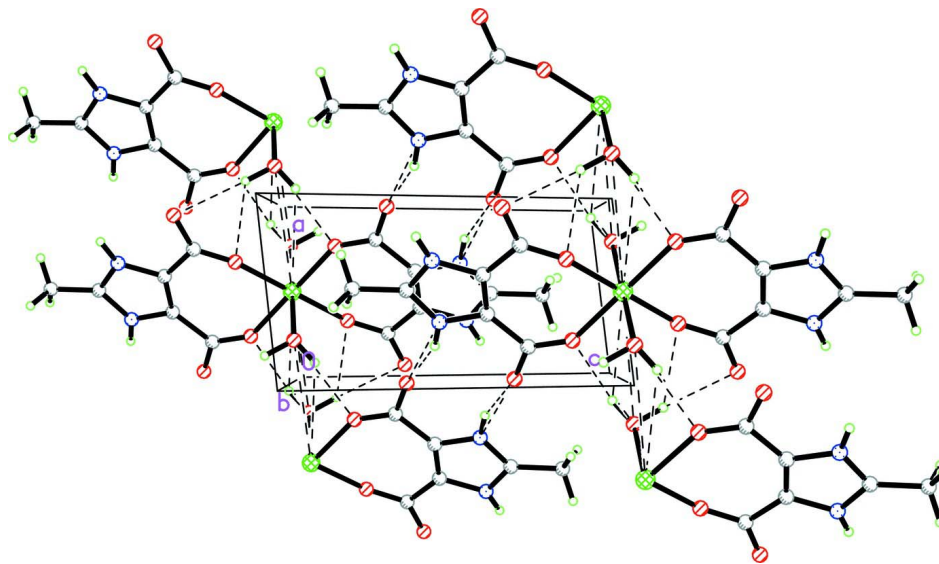
Figure 2

Packing diagram of the title compound viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Diaquabis(2-methyl-1*H*-imidazol-3-ium-4,5-dicarboxylato- *κ²O,O′*)magnesium

Crystal data

[Mg(C$_6$H$_5$N$_2$O$_4$)$_2$(H$_2$O)$_2$]	$Z = 1$
$M_r = 398.58$	$F(000) = 206$
Triclinic, $P\overline{1}$	$D_x = 1.698$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 4.943$ (2) Å	Cell parameters from 1023 reflections
$b = 8.750$ (6) Å	$\theta = 3.9–27.4°$
$c = 9.621$ (6) Å	$\mu = 0.18$ mm^{-1}
$\alpha = 109.18$ (3)°	$T = 292$ K
$\beta = 95.142$ (17)°	Block, colourless
$\gamma = 93.14$ (2)°	$0.30 \times 0.25 \times 0.20$ mm
$V = 389.9$ (4) Å^3	

Data collection

Rigaku SCXmini diffractometer	4002 measured reflections
Radiation source: fine-focus sealed tube	1767 independent reflections
Graphite monochromator	1308 reflections with $I > 2\sigma(I)$
Detector resolution: 13.6612 pixels mm^{-1}	$R_{int} = 0.046$
ω scans	$\theta_{max} = 27.4°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$h = -6 \rightarrow 6$
	$k = -11 \rightarrow 11$
	$l = -12 \rightarrow 12$
$T_{min} = 0.948$, $T_{max} = 0.967$	

Refinement

Refinement on F^2	1767 reflections
Least-squares matrix: full	125 parameters
$R[F^2 > 2\sigma(F^2)] = 0.061$	0 restraints
$wR(F^2) = 0.213$	Primary atom site location: structure-invariant direct methods
$S = 1.19$	

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.1078P)^2 + 0.1465P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$ $\Delta\rho_{max} = 0.39$ e Å^{-3}
H-atom parameters constrained	$\Delta\rho_{min} = -0.42$ 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}
Mg1	0.5000	1.0000	1.0000	0.0250 (4)
C1	0.3793 (6)	0.7562 (4)	0.6015 (3)	0.0229 (7)
C2	0.5912 (6)	0.8628 (4)	0.6010 (3)	0.0223 (7)
C3	0.5089 (7)	0.6811 (4)	0.3730 (4)	0.0267 (7)
C4	0.5167 (9)	0.5941 (5)	0.2137 (4)	0.0385 (9)
H4A	0.4178	0.6492	0.1569	0.058*
H4B	0.4347	0.4851	0.1885	0.058*
H4C	0.7026	0.5915	0.1922	0.058*
C5	0.1991 (7)	0.7356 (4)	0.7122 (3)	0.0240 (7)
C6	0.7466 (6)	1.0094 (4)	0.7140 (3)	0.0233 (7)
N1	0.3343 (6)	0.6454 (3)	0.4585 (3)	0.0268 (6)
H1	0.2007	0.5554	0.4365	0.040*
N2	0.6676 (6)	0.8120 (3)	0.4581 (3)	0.0258 (6)
H2	0.7914	0.8719	0.4213	0.039*
O1	0.6722 (5)	1.0584 (3)	0.8405 (2)	0.0319 (6)
O2	0.9380 (5)	1.0749 (3)	0.6734 (3)	0.0380 (7)
O3	0.2358 (5)	0.8310 (3)	0.8427 (2)	0.0312 (6)
O4	0.0166 (6)	0.6225 (3)	0.6638 (3)	0.0410 (7)
O5	0.2236 (5)	1.1757 (3)	1.0012 (3)	0.0336 (6)
H5B	0.1354	1.1572	0.9070	0.050*
H5A	0.0940	1.1836	1.0574	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0230 (8)	0.0287 (8)	0.0195 (8)	−0.0077 (6)	0.0050 (6)	0.0042 (6)
C1	0.0240 (15)	0.0222 (15)	0.0209 (15)	−0.0021 (12)	0.0033 (12)	0.0055 (12)
C2	0.0213 (14)	0.0243 (15)	0.0216 (15)	−0.0001 (12)	0.0050 (12)	0.0078 (12)
C3	0.0294 (17)	0.0240 (16)	0.0261 (16)	−0.0019 (13)	0.0057 (13)	0.0076 (13)
C4	0.054 (2)	0.0332 (19)	0.0227 (18)	−0.0042 (17)	0.0105 (16)	0.0014 (15)
C5	0.0232 (15)	0.0254 (15)	0.0224 (15)	−0.0035 (12)	0.0057 (12)	0.0067 (12)

C6	0.0230 (15)	0.0259 (16)	0.0206 (15)	−0.0033 (12)	0.0031 (12)	0.0081 (13)
N1	0.0277 (14)	0.0253 (14)	0.0248 (14)	−0.0064 (11)	0.0068 (11)	0.0052 (11)
N2	0.0284 (14)	0.0268 (14)	0.0228 (14)	−0.0031 (11)	0.0060 (11)	0.0092 (11)
O1	0.0353 (13)	0.0336 (14)	0.0227 (12)	−0.0103 (11)	0.0076 (10)	0.0049 (10)
O2	0.0346 (14)	0.0432 (16)	0.0330 (14)	−0.0165 (12)	0.0112 (11)	0.0098 (12)
O3	0.0319 (13)	0.0347 (14)	0.0208 (12)	−0.0114 (10)	0.0069 (10)	0.0022 (10)
O4	0.0408 (15)	0.0392 (15)	0.0323 (14)	−0.0239 (12)	0.0092 (12)	0.0008 (11)
O5	0.0280 (13)	0.0385 (14)	0.0326 (13)	−0.0015 (10)	0.0091 (10)	0.0090 (11)

Geometric parameters (Å, º)

Mg1—O1[i]	2.010 (2)	C3—C4	1.475 (5)
Mg1—O1	2.010 (2)	C4—H4A	0.9600
Mg1—O3[i]	2.036 (2)	C4—H4B	0.9600
Mg1—O3	2.036 (2)	C4—H4C	0.9600
Mg1—O5[i]	2.110 (3)	C5—O4	1.238 (4)
Mg1—O5	2.110 (3)	C5—O3	1.249 (4)
C1—C2	1.365 (4)	C6—O2	1.237 (4)
C1—N1	1.388 (4)	C6—O1	1.247 (4)
C1—C5	1.496 (4)	N1—H1	0.9534
C2—N2	1.393 (4)	N2—H2	0.9489
C2—C6	1.498 (5)	O5—H5B	0.9297
C3—N2	1.330 (4)	O5—H5A	0.8656
C3—N1	1.336 (4)		
O1[i]—Mg1—O1	180.000 (1)	C3—C4—H4A	109.5
O1[i]—Mg1—O3[i]	89.92 (10)	C3—C4—H4B	109.5
O1—Mg1—O3[i]	90.08 (10)	H4A—C4—H4B	109.5
O1[i]—Mg1—O3	90.08 (10)	C3—C4—H4C	109.5
O1—Mg1—O3	89.92 (10)	H4A—C4—H4C	109.5
O3[i]—Mg1—O3	180.000 (1)	H4B—C4—H4C	109.5
O1[i]—Mg1—O5[i]	87.90 (11)	O4—C5—O3	124.6 (3)
O1—Mg1—O5[i]	92.10 (11)	O4—C5—C1	115.5 (3)
O3[i]—Mg1—O5[i]	88.95 (11)	O3—C5—C1	119.9 (3)
O3—Mg1—O5[i]	91.05 (11)	O2—C6—O1	124.7 (3)
O1[i]—Mg1—O5	92.10 (11)	O2—C6—C2	117.0 (3)
O1—Mg1—O5	87.90 (11)	O1—C6—C2	118.3 (3)
O3[i]—Mg1—O5	91.05 (11)	C3—N1—C1	110.7 (3)
O3—Mg1—O5	88.95 (11)	C3—N1—H1	129.8
O5[i]—Mg1—O5	180.000 (1)	C1—N1—H1	119.3
C2—C1—N1	105.8 (3)	C3—N2—C2	110.1 (3)
C2—C1—C5	136.3 (3)	C3—N2—H2	123.8
N1—C1—C5	117.9 (3)	C2—N2—H2	125.2
C1—C2—N2	106.6 (3)	C6—O1—Mg1	147.0 (2)
C1—C2—C6	135.2 (3)	C5—O3—Mg1	145.7 (2)
N2—C2—C6	118.2 (3)	Mg1—O5—H5B	111.2
N2—C3—N1	106.8 (3)	Mg1—O5—H5A	117.4
N2—C3—C4	127.0 (3)	H5B—O5—H5A	104.9

N1—C3—C4	126.2 (3)		
N1—C1—C2—N2	−0.3 (4)	N1—C3—N2—C2	−0.7 (4)
C5—C1—C2—N2	−179.6 (4)	C4—C3—N2—C2	177.5 (4)
N1—C1—C2—C6	−179.5 (3)	C1—C2—N2—C3	0.7 (4)
C5—C1—C2—C6	1.2 (7)	C6—C2—N2—C3	−180.0 (3)
C2—C1—C5—O4	176.8 (4)	O2—C6—O1—Mg1	−150.7 (3)
N1—C1—C5—O4	−2.4 (5)	C2—C6—O1—Mg1	30.6 (6)
C2—C1—C5—O3	−2.0 (6)	O3ⁱ—Mg1—O1—C6	142.4 (4)
N1—C1—C5—O3	178.8 (3)	O3—Mg1—O1—C6	−37.6 (4)
C1—C2—C6—O2	177.8 (4)	O5ⁱ—Mg1—O1—C6	53.4 (4)
N2—C2—C6—O2	−1.3 (5)	O5—Mg1—O1—C6	−126.6 (4)
C1—C2—C6—O1	−3.4 (6)	O4—C5—O3—Mg1	163.9 (3)
N2—C2—C6—O1	177.5 (3)	C1—C5—O3—Mg1	−17.4 (6)
N2—C3—N1—C1	0.5 (4)	O1ⁱ—Mg1—O3—C5	−151.8 (4)
C4—C3—N1—C1	−177.8 (3)	O1—Mg1—O3—C5	28.2 (4)
C2—C1—N1—C3	−0.1 (4)	O5ⁱ—Mg1—O3—C5	−63.9 (4)
C5—C1—N1—C3	179.3 (3)	O5—Mg1—O3—C5	116.1 (4)

Symmetry code: (i) −x+1, −y+2, −z+2.

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O4ⁱⁱ	0.95	1.78	2.696 (4)	161
N2—H2⋯O2ⁱⁱⁱ	0.95	1.81	2.727 (4)	162
O5—H5*B*⋯O2^{iv}	0.93	2.23	3.155 (4)	172
O5—H5*B*⋯O1^{iv}	0.93	2.36	2.961 (4)	122
O5—H5*A*⋯O3^v	0.87	1.98	2.841 (4)	170

Symmetry codes: (ii) −x, −y+1, −z+1; (iii) −x+2, −y+2, −z+1; (iv) x−1, y, z; (v) −x, −y+2, −z+2.