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## Structure Reports

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# Bis( $\mu$ -pyridine-2,4-dicarboxylato)- $\kappa^3N,O^2:O^2$ ; $\kappa^3O^2:N,O^2$ -bis[triaqua-magnesium(II)]

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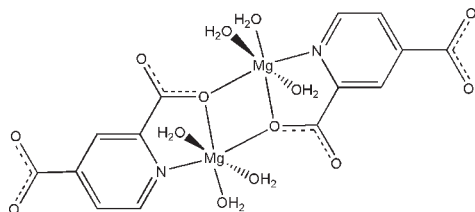
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(C-C) = 0.003$  Å;  $R$  factor = 0.040;  $wR$  factor = 0.100; data-to-parameter ratio = 11.9.

In the title centrosymmetric  $Mg^{II}$  complex,  $[Mg_2(C_7H_3NO_4)_2(H_2O)_6]$ , each Mg cation is  $N,O$ -chelated by a pyridine-2,4-dicarboxylate dianion and is coordinated by three water molecules. A carboxylate O atom from the neighboring pyridine-2,4-dicarboxylate dianion bridges the Mg cation to complete the  $MgNO_5$  distorted octahedral coordination geometry. The dinuclear complex molecules are linked by intermolecular  $O-H \cdots O$  hydrogen bonding, forming a three-dimensional supramolecular structure.

## Related literature

For the applications of Mg complexes, see: Davies *et al.* (2007); Dinca & Long (2005).



## Experimental

### Crystal data

$[Mg_2(C_7H_3NO_4)_2(H_2O)_6]$   
 $M_r = 486.92$   
 Orthorhombic,  $Pbca$   
 $a = 7.9221$  (8) Å  
 $b = 12.0951$  (12) Å  
 $c = 20.2989$  (18) Å

$V = 1945.0$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.21$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.25 \times 0.18 \times 0.15$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{min} = 0.951$ ,  $T_{max} = 0.970$   
 8884 measured reflections  
 1719 independent reflections  
 1289 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.042$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.100$   
 $S = 1.06$   
 1719 reflections  
 145 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{max} = 0.36$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.30$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Mg1—N1	2.2202 (19)	Mg1—O5	2.0953 (19)
Mg1—O1	2.1011 (19)	Mg1—O6	2.017 (2)
Mg1—O1 <sup>i</sup>	2.0613 (16)	Mg1—O7	2.033 (2)

Symmetry code: (i)  $-x, -y + 1, -z$ .

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O5—H5A $\cdots$ O4 <sup>ii</sup>	0.87	1.80	2.668 (3)	172
O5—H5B $\cdots$ O2 <sup>iii</sup>	0.86	2.12	2.834 (3)	140
O6—H6A $\cdots$ O3 <sup>iv</sup>	0.87	1.73	2.576 (3)	163
O6—H6B $\cdots$ O5 <sup>v</sup>	0.87	2.07	2.880 (2)	154
O7—H7A $\cdots$ O4 <sup>vi</sup>	0.86	1.89	2.745 (2)	174
O7—H7B $\cdots$ O4 <sup>vii</sup>	0.86	2.02	2.857 (3)	166

Symmetry codes: (ii)  $x - \frac{1}{2}, y, -z + \frac{1}{2}$ ; (iii)  $-x - \frac{1}{2}, y + \frac{1}{2}, z$ ; (iv)  $-x, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (v)  $x + \frac{1}{2}, -y + \frac{3}{2}, -z$ ; (vi)  $-x + \frac{1}{2}, -y + 1, z - \frac{1}{2}$ ; (vii)  $x + \frac{1}{2}, y, -z + \frac{1}{2}$ .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5007).

## References

- Davies, R. P., Less, R. J., Lickiss, P. D. & White, A. J. P. (2007). *Dalton Trans.* pp. 2528–2535.  
 Dinca, M. & Long, J. R. (2005). *J. Am. Chem. Soc.* **127**, 9376–9377.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Siemens. (1996). *SMART and SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

**supplementary materials**

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## Bis( $\mu$ -pyridine-2,4-dicarboxylato)- $\kappa^3 N, O^2: O^2; \kappa^3 O^2: N, O^2$ -bis[triaquamagnesium(II)]

Q.-F. Zhang, D.-D. Han, J.-D. Pang and N.-N. Meng

### Comment

Magnesium(II) complexes have been extensively studied in recent year due to their wide application in synthesis of inorganic materials, catalysis of organic reactions and storage of hydrogen (Dinca & Long, 2005). In order to explore the relationship between these applications and their structures, a series of magnesium(II) complexes have been prepared and structural characterized (Davies *et al.*, 2007). To expand research in this area, here we report a dinuclear magnesium(II) complex.

As shown in Fig.1, each magnesium(II) ion in the title complex is coordinated by two carboxyl O atoms, one pyridyl N atom and three water molecules, forming a distorted octahedral geometry (axial angle, O7—Mg1—O5 = 173.33 (9)°). Interestingly, the two adjacent magnesium(II) ions are linked by two  $\mu_2$ -carboxylate O atoms to form a dinuclear structure.

In the crystal structure, these dimeric molecules are linked by intermolecular O—H...O H-bonding interactions into a three-dimensional framework (Fig.2).

### Experimental

A mixture of pyridine-2,4-dicarboxylic acid (16.7 mg, 0.10 mmol) and magnesium chloride hexahydrate (20.3 mg, 0.10 mmol) in 5 ml dimethyl acetamide (DMA) was heated to 373 K in a sealed 10 ml Teflon-lined reactor for 72 h. The mixture was allowed to cool to room temperature and the resulting block-shaped colorless crystals filtered from the reaction mixture. Yield, 72%.

### Refinement

The H atoms on water were located in a difference Fourier map and refined in riding mode with the fixed  $U_{\text{iso}}(\text{H}) = 0.105 \text{ \AA}^2$ . The aromatic H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

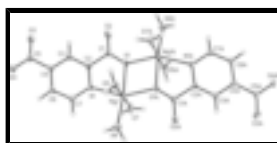


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids.



Fig. 2. The packing diagram of the title complex.

# supplementary materials

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## Bis( $\mu$ -pyridine-2,4-dicarboxylato)- $\kappa^3N,O^2:O^2$ ; $\kappa^3O^2:N,O^2$ - bis[triaquamagnesium(II)]

### Crystal data

[Mg <sub>2</sub> (C <sub>7</sub> H <sub>3</sub> NO <sub>4</sub> ) <sub>2</sub> (H <sub>2</sub> O) <sub>6</sub> ]	$F(000) = 1008$
$M_r = 486.92$	$D_x = 1.663 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ac 2ab	Cell parameters from 2705 reflections
$a = 7.9221 (8) \text{ \AA}$	$\theta = 3.2\text{--}26.8^\circ$
$b = 12.0951 (12) \text{ \AA}$	$\mu = 0.21 \text{ mm}^{-1}$
$c = 20.2989 (18) \text{ \AA}$	$T = 293 \text{ K}$
$V = 1945.0 (3) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.25 \times 0.18 \times 0.15 \text{ mm}$

### Data collection

Bruker SMART CCD area-detector diffractometer	1719 independent reflections
Radiation source: fine-focus sealed tube graphite	1289 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.042$
Absorption correction: multi-scan ( <i>SADABS</i> ; Sheldrick, 1996)	$\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 2.0^\circ$
$T_{\text{min}} = 0.951$ , $T_{\text{max}} = 0.970$	$h = -5 \rightarrow 9$
8884 measured reflections	$k = -14 \rightarrow 14$
	$l = -24 \rightarrow 23$

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.100$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 1.7679P]$
1719 reflections	where $P = (F_o^2 + 2F_c^2)/3$
145 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$

### 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Mg1	0.07954 (11)	0.60169 (7)	0.04323 (3)	0.0240 (2)
O1	-0.0075 (2)	0.43818 (14)	0.05141 (7)	0.0272 (4)
O2	-0.0923 (4)	0.30663 (19)	0.12021 (10)	0.0760 (9)
O3	-0.0102 (4)	0.3649 (2)	0.35781 (9)	0.0875 (11)
H6A	0.1178	0.8068	0.0765	0.105*
H5B	-0.1892	0.7246	0.0848	0.105*
H5A	-0.2377	0.6193	0.0826	0.105*
H7A	0.3503	0.5401	-0.0194	0.105*
H7B	0.4054	0.5600	0.0457	0.105*
H6B	0.2174	0.8016	0.0211	0.105*
O4	0.1026 (2)	0.51899 (15)	0.39638 (8)	0.0364 (5)
O5	-0.1604 (2)	0.66298 (14)	0.06670 (8)	0.0323 (5)
O6	0.1539 (3)	0.76106 (16)	0.04659 (9)	0.0491 (6)
O7	0.3209 (2)	0.56088 (17)	0.01937 (8)	0.0430 (5)
N1	0.1126 (3)	0.56101 (17)	0.14911 (9)	0.0257 (5)
C1	-0.0232 (4)	0.3941 (2)	0.10852 (11)	0.0325 (6)
C2	0.0461 (3)	0.4620 (2)	0.16485 (11)	0.0261 (6)
C3	0.0314 (3)	0.4258 (2)	0.22895 (11)	0.0302 (6)
H3	-0.0135	0.3564	0.2378	0.036*
C4	0.0838 (3)	0.4934 (2)	0.28011 (11)	0.0281 (6)
C5	0.0576 (4)	0.4556 (2)	0.35037 (12)	0.0361 (7)
C6	0.1530 (4)	0.5952 (2)	0.26430 (11)	0.0318 (6)
H6	0.1902	0.6426	0.2973	0.038*
C7	0.1660 (4)	0.6255 (2)	0.19836 (11)	0.0325 (6)
H7	0.2139	0.6935	0.1881	0.039*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Mg1	0.0328 (5)	0.0234 (5)	0.0157 (4)	-0.0004 (4)	0.0015 (3)	-0.0008 (3)
O1	0.0415 (11)	0.0250 (10)	0.0151 (8)	0.0001 (8)	-0.0028 (8)	0.0000 (7)
O2	0.151 (3)	0.0485 (15)	0.0289 (11)	-0.0543 (16)	-0.0205 (13)	0.0080 (10)
O3	0.176 (3)	0.0627 (17)	0.0241 (11)	-0.0682 (19)	0.0088 (14)	0.0028 (10)
O4	0.0497 (12)	0.0419 (12)	0.0176 (8)	-0.0064 (9)	-0.0043 (8)	-0.0016 (8)
O5	0.0379 (11)	0.0271 (10)	0.0320 (9)	0.0057 (8)	0.0049 (8)	-0.0031 (8)
O6	0.0683 (15)	0.0308 (11)	0.0482 (12)	-0.0158 (10)	0.0282 (11)	-0.0089 (9)
O7	0.0339 (11)	0.0693 (15)	0.0260 (9)	0.0104 (10)	-0.0014 (8)	-0.0103 (9)
N1	0.0348 (13)	0.0239 (12)	0.0185 (10)	-0.0021 (9)	-0.0003 (9)	0.0002 (8)
C1	0.0538 (18)	0.0229 (14)	0.0208 (13)	-0.0060 (13)	-0.0040 (12)	-0.0003 (11)

## supplementary materials

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C2	0.0327 (15)	0.0238 (14)	0.0220 (12)	-0.0006 (11)	-0.0008 (11)	0.0010 (10)
C3	0.0445 (17)	0.0256 (14)	0.0205 (12)	-0.0074 (12)	-0.0023 (12)	0.0028 (10)
C4	0.0349 (15)	0.0301 (15)	0.0194 (12)	-0.0011 (12)	-0.0010 (11)	0.0007 (10)
C5	0.0526 (19)	0.0362 (17)	0.0193 (13)	-0.0074 (14)	-0.0014 (12)	0.0027 (12)
C6	0.0449 (16)	0.0308 (15)	0.0196 (12)	-0.0068 (13)	-0.0052 (12)	-0.0026 (10)
C7	0.0466 (17)	0.0275 (15)	0.0236 (13)	-0.0102 (13)	-0.0025 (12)	0.0009 (11)

### *Geometric parameters (Å, °)*

Mg1—N1	2.2202 (19)	O7—H7A	0.8581
Mg1—O1	2.1011 (19)	O7—H7B	0.8563
Mg1—O1 <sup>i</sup>	2.0613 (16)	N1—C7	1.336 (3)
Mg1—O5	2.0953 (19)	N1—C2	1.347 (3)
Mg1—O6	2.017 (2)	C1—C2	1.511 (3)
Mg1—O7	2.033 (2)	C2—C3	1.378 (3)
O1—C1	1.282 (3)	C3—C4	1.385 (3)
O2—C1	1.215 (3)	C3—H3	0.9300
O3—C5	1.231 (3)	C4—C6	1.385 (4)
O4—C5	1.260 (3)	C4—C5	1.512 (3)
O5—H5B	0.8616	C6—C7	1.392 (3)
O5—H5A	0.8701	C6—H6	0.9300
O6—H6A	0.8688	C7—H7	0.9300
O6—H6B	0.8725		
O6—Mg1—O7	88.02 (9)	H6A—O6—H6B	104.2
O6—Mg1—O1 <sup>i</sup>	109.63 (8)	Mg1—O7—H7A	123.0
O7—Mg1—O1 <sup>i</sup>	88.93 (7)	Mg1—O7—H7B	126.1
O6—Mg1—O5	85.36 (8)	H7A—O7—H7B	110.8
O7—Mg1—O5	173.33 (9)	C7—N1—C2	117.7 (2)
O1 <sup>i</sup> —Mg1—O5	92.50 (7)	C7—N1—Mg1	129.22 (17)
O6—Mg1—O1	173.16 (8)	C2—N1—Mg1	112.37 (15)
O7—Mg1—O1	95.68 (8)	O2—C1—O1	125.6 (2)
O1 <sup>i</sup> —Mg1—O1	76.26 (7)	O2—C1—C2	119.3 (2)
O5—Mg1—O1	90.98 (8)	O1—C1—C2	115.0 (2)
O6—Mg1—N1	98.32 (8)	N1—C2—C3	122.7 (2)
O7—Mg1—N1	93.77 (8)	N1—C2—C1	116.5 (2)
O1 <sup>i</sup> —Mg1—N1	152.00 (8)	C3—C2—C1	120.7 (2)
O5—Mg1—N1	88.02 (8)	C2—C3—C4	119.7 (2)
O1—Mg1—N1	75.74 (7)	C2—C3—H3	120.2
O6—Mg1—Mg1 <sup>i</sup>	148.06 (7)	C4—C3—H3	120.2
O7—Mg1—Mg1 <sup>i</sup>	92.97 (6)	C6—C4—C3	118.0 (2)
O1 <sup>i</sup> —Mg1—Mg1 <sup>i</sup>	38.56 (5)	C6—C4—C5	122.8 (2)
O5—Mg1—Mg1 <sup>i</sup>	92.20 (6)	C3—C4—C5	119.2 (2)
O1—Mg1—Mg1 <sup>i</sup>	37.70 (4)	O3—C5—O4	125.1 (2)
N1—Mg1—Mg1 <sup>i</sup>	113.44 (7)	O3—C5—C4	116.5 (2)
C1—O1—Mg1 <sup>i</sup>	135.94 (16)	O4—C5—C4	118.4 (2)
C1—O1—Mg1	119.66 (15)	C4—C6—C7	119.1 (2)

Mg1 <sup>i</sup> —O1—Mg1	103.74 (7)	C4—C6—H6	120.5
Mg1—O5—H5B	130.0	C7—C6—H6	120.5
Mg1—O5—H5A	120.5	N1—C7—C6	122.9 (2)
H5B—O5—H5A	100.5	N1—C7—H7	118.6
Mg1—O6—H6A	122.4	C6—C7—H7	118.6
Mg1—O6—H6B	133.3		

Symmetry codes: (i)  $-x, -y+1, -z$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A $\cdots$ O4 <sup>ii</sup>	0.87	1.80	2.668 (3)	172
O5—H5B $\cdots$ O2 <sup>iii</sup>	0.86	2.12	2.834 (3)	140
O6—H6A $\cdots$ O3 <sup>iv</sup>	0.87	1.73	2.576 (3)	163
O6—H6B $\cdots$ O5 <sup>v</sup>	0.87	2.07	2.880 (2)	154
O7—H7A $\cdots$ O4 <sup>vi</sup>	0.86	1.89	2.745 (2)	174
O7—H7B $\cdots$ O4 <sup>vii</sup>	0.86	2.02	2.857 (3)	166

Symmetry codes: (ii)  $x-1/2, y, -z+1/2$ ; (iii)  $-x-1/2, y+1/2, z$ ; (iv)  $-x, y+1/2, -z+1/2$ ; (v)  $x+1/2, -y+3/2, -z$ ; (vi)  $-x+1/2, -y+1, z-1/2$ ; (vii)  $x+1/2, y, -z+1/2$ .

Fig. 1

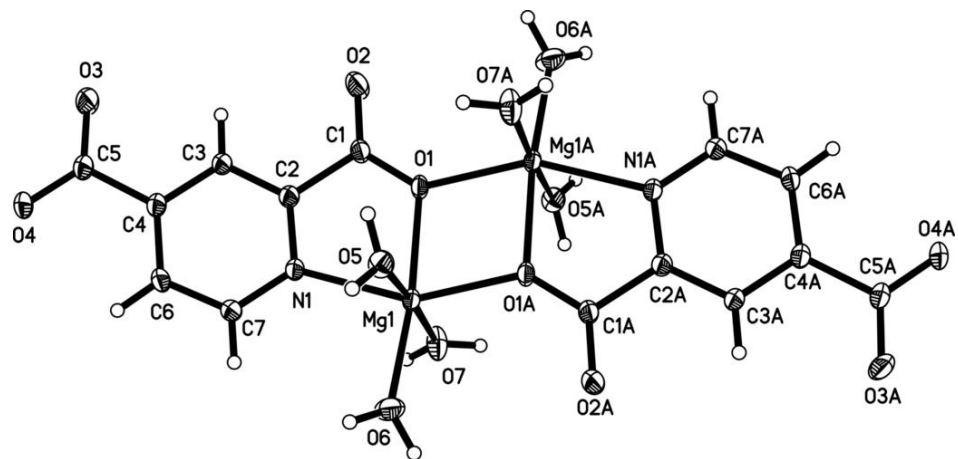




Fig. 2

