metal-organic compounds

Tetraaquabis(1-hydroxy-2-naphthoato-κO^2)magnesium(II)

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In the title mononuclear complex, [Mg(C₁₁H₇O₃)₂(H₂O)₄], the Mg^{II} atom is located on a centre of inversion and is coordinated by two O atoms from two 1-hydroxy-2-naphthoate ligands and four water molecules in an octahedral geometry. The structure is consolidated by intermolecular O—H⋯O hydrogen bonding, as well as by π–π stacking interactions between adjacent naphthyl ring systems [centroid–centroid distance between parallel naphthoate rings is 3.768 (2) Å].

Related literature

For metal 1-hydroxy-2-naphthoates see: Ohki *et al.* (1986, 1987); Schmidt *et al.* (2005); Xue *et al.* (2005).
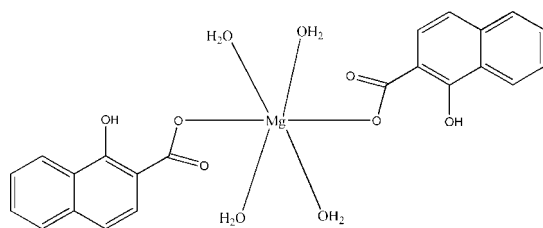
Experimental

Crystal data

[Mg(C₁₁H₇O₃)₂(H₂O)₄]
M_r = 470.71
Monoclinic, $P2_1/n$
a = 6.7462 (5) Å
b = 5.2285 (4) Å
c = 29.965 (2) Å
β = 94.280 (5)°
V = 1054.01 (14) Å³
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm⁻¹
T = 296 (2) K
0.30 × 0.26 × 0.25 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.965

8826 measured reflections
1936 independent reflections
1367 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.135
S = 1.06
1936 reflections
164 parameters
6 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å⁻³
$\Delta\rho_{min}$ = −0.19 e Å⁻³

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2W—H3W⋯O1[i]	0.814 (9)	2.074 (13)	2.819 (2)	152 (2)
O2W—H4W⋯O3[ii]	0.821 (9)	1.888 (10)	2.698 (2)	169 (2)
O1W—H2W⋯O2W[iii]	0.813 (10)	2.136 (10)	2.924 (2)	163 (2)
O1W—H1W⋯O3	0.821 (9)	2.000 (13)	2.734 (2)	149 (2)
O1—H1⋯O2	0.82	1.75	2.4858 (19)	148

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

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supporting information

Tetraaquabis(1-hydroxy-2-naphthoato-*κO²*)magnesium(II)

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S1. Comment

In the structural investigation of 1-hydroxy-2-naphthoate complexes, it has been found that the 1-hydroxy-2-naphthoate functions as a multidentate ligand (Ohki *et al.*, 1986; 1987; Schmidt *et al.* (2005); Xue *et al.* (2005), with versatile binding and coordination modes. In this paper, we report the crystal structure of the title compound, (I), a new Mg complex obtained by the reaction of 1-naphthol-2-carboxylic acid with magnesium chloride in alkaline aqueous solution.

As illustrated in Figure 1, the Mg^{II} atom, lies on a centre of inversion, has an octahedral geometry, which is defined by two O atoms from two 1-hydroxy-2-naphthoate ligands and four water molecules (Fig. 1). The structural components are governed by intermolecular O—H⋯O hydrogen bond (Table 1) involving the coordinated water molecules, the hydroxy and carboxyl O atoms of 1-hydroxy-2-naphthoate ligands, and *via π-π* stacking interaction. The centroid to centroid distance between parallel naphthoate rings of neighboring complexes (at *x*, 1 + *y*, *z*) is 3.768 (2) Å, thus indicating a weak *π-π* stacking interaction.

S2. Experimental

A mixture of magnesium chloride(1 mmol), 1-hydroxy-2-naphthoate (1 mmol) NaOH (1.5 mmol) and H$_2$O (12 ml) was placed in a 23 ml Teflon reactor, which was heated to 433 K for three days and then cooled to room temperature at a rate of 10 K h^{-1}. The crystals obtained were washed with water and dried in air.

S3. Refinement

Carbon-bound and hydroxyl H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 Å, O—H = 0.82 Å and with U_{iso}(H) = 1.2 U_{eq}(C, O). Water H atoms were tentatively located in difference Fourier maps and were refined with distance restraints of O–H = 0.82 Å and H⋯H = 1.29 Å, each within a standard deviation of 0.01 Å, and with U_{iso}(H) = 1.5 U_{eq}(O).
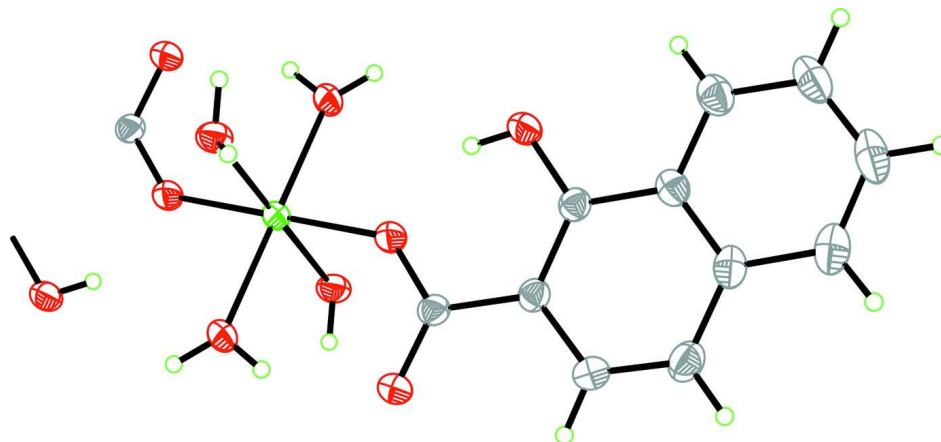
Figure 1

The structure of (I), showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids. Unlabeled atoms are related to the labelled atoms by the symmetry operator (1 - *x*, 1 - *y*, -*z*).
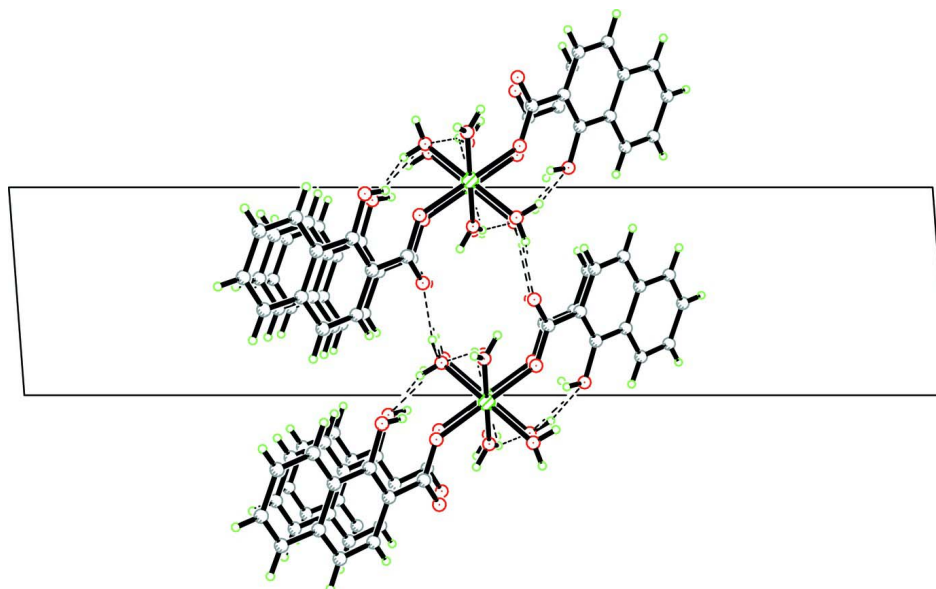
Figure 2

A packing view of the title compound. The intermolecluar hydrogen bonds are shown with dashed lines.

Tetraaquabis(1-hydroxy-2-naphthoato-*κO²*)magnesium(II)

Crystal data

$[Mg(C_{11}H_7O_3)_2(H_2O)_4]$	$F(000) = 492$
$M_r = 470.71$	$D_x = 1.483$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3500 reflections
$a = 6.7462$ (5) Å	$\theta = 1.3–26°$
$b = 5.2285$ (4) Å	$\mu = 0.14$ mm^{-1}
$c = 29.965$ (2) Å	$T = 296$ K
$\beta = 94.280$ (5)°	Block, colorless
$V = 1054.01$ (14) Å^3	$0.30 \times 0.26 \times 0.25$ mm
$Z = 2$	

Data collection

Bruker APEXII area-detector diffractometer	8826 measured reflections
Radiation source: fine-focus sealed tube	1936 independent reflections
Graphite monochromator	1367 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.040$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.5°$, $\theta_{min} = 2.7°$
	$h = -7 \rightarrow 8$
$T_{min} = 0.958$, $T_{max} = 0.965$	$k = -6 \rightarrow 6$
	$l = -36 \rightarrow 28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	
$wR(F^2) = 0.135$	
$S = 1.07$	H atoms treated by a mixture of independent and constrained refinement
1936 reflections	
164 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
6 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.18$ e Å^{-3}
	$\Delta\rho_{min} = -0.19$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.8444 (3)	0.6423 (4)	0.06903 (7)	0.0358 (5)
C2	0.9063 (3)	0.8351 (4)	0.10386 (7)	0.0357 (5)
C3	0.7694 (3)	1.0058 (4)	0.11954 (7)	0.0364 (6)
C4	0.8258 (4)	1.1864 (4)	0.15346 (7)	0.0395 (6)
C5	0.6880 (4)	1.3630 (4)	0.16921 (8)	0.0509 (7)
H5	0.5572	1.3641	0.1570	0.061*
C6	0.7470 (5)	1.5322 (4)	0.20245 (9)	0.0608 (8)
H6	0.6556	1.6466	0.2129	0.073*
C7	0.9423 (5)	1.5343 (5)	0.22074 (9)	0.0666 (9)
H7	0.9804	1.6506	0.2432	0.080*
C8	1.0783 (5)	1.3682 (5)	0.20608 (9)	0.0613 (8)
H8	1.2083	1.3720	0.2188	0.074*
C9	1.0249 (4)	1.1889 (4)	0.17163 (7)	0.0463 (6)
C10	1.1625 (4)	1.0146 (4)	0.15507 (8)	0.0515 (7)
H10	1.2943	1.0166	0.1667	0.062*

C11	1.1040 (3)	0.8449 (4)	0.12247 (8)	0.0451 (6)
H11	1.1968	0.7320	0.1122	0.054*
Mg1	0.5000	0.5000	0.0000	0.0354 (3)
O1	0.5757 (2)	1.0040 (3)	0.10408 (5)	0.0463 (5)
H1	0.5581	0.8945	0.0846	0.069*
O2	0.6611 (2)	0.6510 (3)	0.05335 (5)	0.0439 (4)
O3	0.9626 (2)	0.4811 (3)	0.05591 (5)	0.0467 (5)
O1W	0.7019 (2)	0.2055 (3)	0.00065 (6)	0.0473 (5)
H1W	0.806 (2)	0.238 (4)	0.0157 (8)	0.071*
H2W	0.716 (4)	0.061 (3)	−0.0087 (8)	0.071*
O2W	0.3269 (2)	0.2933 (3)	0.04349 (5)	0.0437 (5)
H4W	0.221 (2)	0.351 (5)	0.0510 (7)	0.066*
H3W	0.387 (3)	0.249 (5)	0.0668 (5)	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0323 (14)	0.0336 (11)	0.0417 (14)	−0.0011 (10)	0.0052 (11)	0.0022 (10)
C2	0.0348 (14)	0.0336 (12)	0.0384 (13)	−0.0021 (10)	0.0009 (10)	0.0018 (9)
C3	0.0360 (14)	0.0334 (11)	0.0396 (13)	−0.0016 (10)	0.0016 (11)	0.0019 (10)
C4	0.0485 (15)	0.0329 (11)	0.0382 (14)	−0.0028 (11)	0.0095 (11)	0.0016 (10)
C5	0.0627 (18)	0.0401 (13)	0.0513 (16)	−0.0034 (12)	0.0129 (13)	−0.0026 (11)
C6	0.088 (2)	0.0398 (14)	0.0573 (18)	−0.0046 (14)	0.0244 (17)	−0.0095 (12)
C7	0.098 (3)	0.0509 (16)	0.0522 (18)	−0.0182 (16)	0.0125 (18)	−0.0174 (13)
C8	0.071 (2)	0.0593 (16)	0.0522 (17)	−0.0175 (16)	−0.0047 (15)	−0.0068 (13)
C9	0.0549 (17)	0.0440 (13)	0.0399 (14)	−0.0125 (12)	0.0033 (12)	−0.0035 (11)
C10	0.0436 (16)	0.0592 (16)	0.0502 (16)	−0.0065 (12)	−0.0060 (12)	−0.0050 (12)
C11	0.0354 (15)	0.0487 (14)	0.0512 (16)	0.0009 (11)	0.0037 (12)	−0.0032 (11)
Mg1	0.0318 (6)	0.0267 (5)	0.0476 (7)	0.0029 (4)	0.0022 (5)	−0.0050 (4)
O1	0.0387 (11)	0.0452 (10)	0.0543 (11)	0.0098 (7)	−0.0017 (8)	−0.0129 (7)
O2	0.0330 (10)	0.0410 (9)	0.0564 (10)	0.0061 (7)	−0.0047 (8)	−0.0123 (7)
O3	0.0366 (10)	0.0454 (10)	0.0581 (11)	0.0067 (7)	0.0035 (8)	−0.0127 (7)
O1W	0.0399 (11)	0.0299 (8)	0.0711 (13)	0.0059 (7)	−0.0019 (9)	−0.0112 (8)
O2W	0.0360 (10)	0.0402 (9)	0.0554 (11)	0.0071 (7)	0.0068 (8)	0.0016 (8)

Geometric parameters (Å, º)

C1—O3	1.244 (2)	C8—H8	0.9300
C1—O2	1.290 (2)	C9—C10	1.417 (3)
C1—C2	1.488 (3)	C10—C11	1.357 (3)
C2—C3	1.391 (3)	C10—H10	0.9300
C2—C11	1.407 (3)	C11—H11	0.9300
C3—O1	1.353 (3)	Mg1—O2i	2.0254 (15)
C3—C4	1.418 (3)	Mg1—O2	2.0254 (15)
C4—C9	1.411 (3)	Mg1—O1W	2.0551 (14)
C4—C5	1.416 (3)	Mg1—O1Wi	2.0551 (14)
C5—C6	1.369 (3)	Mg1—O2W	2.1112 (15)
C5—H5	0.9300	Mg1—O2Wi	2.1112 (15)

C6—C7	1.388 (4)	O1—H1	0.8200
C6—H6	0.9300	O1W—H1W	0.821 (9)
C7—C8	1.360 (4)	O1W—H2W	0.813 (10)
C7—H7	0.9300	O2W—H4W	0.821 (9)
C8—C9	1.421 (3)	O2W—H3W	0.814 (9)
O3—C1—O2	121.9 (2)	C11—C10—H10	119.7
O3—C1—C2	121.9 (2)	C9—C10—H10	119.7
O2—C1—C2	116.24 (19)	C10—C11—C2	121.7 (2)
C3—C2—C11	118.4 (2)	C10—C11—H11	119.2
C3—C2—C1	120.9 (2)	C2—C11—H11	119.2
C11—C2—C1	120.7 (2)	O2^{i}—Mg1—O2	180.00 (6)
O1—C3—C2	121.89 (19)	O2^{i}—Mg1—O1W	91.81 (6)
O1—C3—C4	116.78 (19)	O2—Mg1—O1W	88.19 (6)
C2—C3—C4	121.3 (2)	O2^{i}—Mg1—O1W^{i}	88.19 (6)
C9—C4—C5	119.7 (2)	O2—Mg1—O1W^{i}	91.81 (6)
C9—C4—C3	118.6 (2)	O1W—Mg1—O1W^{i}	180.00 (12)
C5—C4—C3	121.7 (2)	O2^{i}—Mg1—O2W	89.92 (6)
C6—C5—C4	120.1 (3)	O2—Mg1—O2W	90.08 (6)
C6—C5—H5	120.0	O1W—Mg1—O2W	90.42 (6)
C4—C5—H5	120.0	O1W^{i}—Mg1—O2W	89.58 (6)
C5—C6—C7	120.6 (3)	O2^{i}—Mg1—O2W^{i}	90.08 (6)
C5—C6—H6	119.7	O2—Mg1—O2W^{i}	89.92 (6)
C7—C6—H6	119.7	O1W—Mg1—O2W^{i}	89.58 (6)
C8—C7—C6	120.7 (2)	O1W^{i}—Mg1—O2W^{i}	90.42 (6)
C8—C7—H7	119.6	O2W—Mg1—O2W^{i}	180.00 (10)
C6—C7—H7	119.6	C3—O1—H1	109.5
C7—C8—C9	121.0 (3)	C1—O2—Mg1	135.80 (13)
C7—C8—H8	119.5	Mg1—O1W—H1W	112.7 (16)
C9—C8—H8	119.5	Mg1—O1W—H2W	142.1 (16)
C4—C9—C10	119.3 (2)	H1W—O1W—H2W	105.2 (15)
C4—C9—C8	118.0 (2)	Mg1—O2W—H4W	120.8 (18)
C10—C9—C8	122.8 (3)	Mg1—O2W—H3W	114.4 (18)
C11—C10—C9	120.6 (2)	H4W—O2W—H3W	104.8 (15)

Symmetry code: (i) −x+1, −y+1, −z.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O2W—H3W···O1^{ii}	0.81 (1)	2.07 (1)	2.819 (2)	152 (2)
O2W—H4W···O3^{iii}	0.82 (1)	1.89 (1)	2.698 (2)	169 (2)
O1W—H2W···O2W^{iv}	0.81 (1)	2.14 (1)	2.924 (2)	163 (2)
O1W—H1W···O3	0.82 (1)	2.00 (1)	2.734 (2)	149 (2)
O1—H1···O2	0.82	1.75	2.4858 (19)	148

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