metal-organic compounds

Hexaaquamagnesium(II) bis[4-(3-pyridyl)pyrimidine-2-sulfonate] tetrahydrate

Yan-Ping Wang, Jian Li and Hua-Ze Dong*

Department of Chemistry and Chemical Engineering, Hefei Normal University, Hefei 230061, People's Republic of China
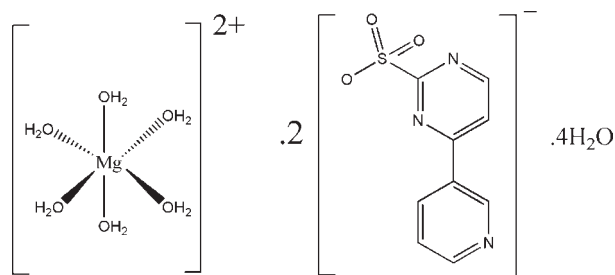Correspondence e-mail: dapdong@163.com

The asymmetric unit of the title compound, $[Mg(H_2O)_6]$- $(C_9H_6N_3O_3S)_2 \cdot 4H_2O$, contains half of a centrosymmetric cation, one 4-(3-pyridyl)pyrimidin-2-sulfonate anion and two solvent water molecules. Intermolecular O—H···O and O—H···N hydrogen bonds link the cations, anions and water molecules into a three-dimensional supramolecular structure. The crystal packing also exhibits intermolecular π–π interactions between the aromatic rings of the anions with a centroid–centroid distance of 3.604 (2) Å.

Related literature

For coordination complexes with pyridin-2-sulfonate ligands, see: Kimura et al. (1999); Lobana et al. (2004). For coordination complexes with 4-(pyridin-yl)pyrimidin-2-sulfonate, see: Zhu et al. (2007); Fang et al. (2009).

Experimental

Crystal data

$[Mg(H_2O)_6](C_9H_6N_3O_3S)_2 \cdot 4H_2O$	b = 13.3600 (3) Å
M_r = 676.95	c = 16.2565 (4) Å
Monoclinic, $P2_1/n$	β = 98.7240 (10)°
a = 6.9835 (2) Å	V = 1499.18 (7) Å^3

Z = 2	T = 291 K
Mo $K\alpha$ radiation	0.30 × 0.15 × 0.12 mm
μ = 0.28 mm^{-1}	

Data collection

Bruker SMART CCD area-detector diffractometer	14712 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	3438 independent reflections
T_{min} = 0.917, T_{max} = 0.966	2848 reflections with $I > 2\sigma(I)$
	R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.102	
S = 1.04	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
3438 reflections	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
236 parameters	
7 restraints	

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O1^i	0.83 (2)	1.94 (2)	2.7650 (19)	180 (3)
O4—H4B···O2	0.86 (2)	1.89 (2)	2.7475 (19)	176 (2)
O5—H5B···O3^i	0.83 (2)	2.04 (2)	2.8705 (19)	177.0 (18)
O5—H5A···O8^{ii}	0.86 (2)	1.91 (2)	2.755 (3)	167 (3)
O6—H6B···O3^{iii}	0.83 (2)	2.03 (2)	2.8601 (19)	178 (2)
O6—H6A···N3^{iv}	0.85 (3)	1.92 (3)	2.763 (2)	173 (2)
O7—H7B···O3^v	0.84 (3)	2.51 (3)	3.110 (2)	130 (3)
O7—H7B···N2^v	0.84 (3)	2.21 (3)	2.984 (2)	154 (3)
O7—H7A···O2	0.84 (3)	2.12 (3)	2.923 (2)	161 (3)
O8—H8B···O1^{vi}	0.85 (4)	2.43 (4)	3.064 (2)	132 (4)
O8—H8A···O7^{iii}	0.85 (3)	1.93 (3)	2.769 (3)	170 (3)

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2632).

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supporting information

Hexaaquamagnesium(II) bis[4-(3-pyridyl)pyrimidine-2-sulfonate] tetrahydrate

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S1. Comment

The rational design and synthesis of coordination complexes derived from heterocyclic sulfonate ligands have been of increasing interest recently in chemical research (Kimura *et al.*, 1999; Lobana *et al.*, 2004). In our previous work (Zhu *et al.*, 2007; Fang *et al.*, 2009), we have also studied transition metal coordination complexes with the heterocyclic sulfonate ligands, namely 4-(pyridin-2-yl)pyrimidin-2-sulfonate and 4-(pyridin-4-yl)pyrimidin-2-sulfonate. Herein, we report the magnesium(II) coordination complex with its analog, *viz.* 4-(pyridin-3-yl)pyrimidin-2-sulfonate.

The asymmetric unit of the title compound (Fig. 1) consists of a 4-(3-pyridyl)pyrimidin-2-sulfonate anion, one half of an $[Mg(H_2O)_6]^{2+}$ cation and two free water molecules. The averaged Mg—O coordinating bond length is 2.0664 (13) Å. In the crystal structure, intermolecular O—H···O and O—H···N hydrogen bonds (Table 1) link cations, anions and crystalline water molecules into three-dimensinal network. The crystal packing exhibits also intermolecular π—π interactions between the aromatic rings of the anions with the centroid-centroid distance of 3.604 (2) Å.

S2. Experimental

All solvents and chemicals were of analytical grade and were used without further purification. 4-(3-Pyridyl)pyrimidin-2-sulfonate (L) was prepared by similar procedure reported in the literature (Zhu *et al.*, 2007; Fang *et al.*, 2009). For the synthesis of title compoud, a solution of L (0.1 mmol), $MgSO_4$ (0.1 mmol) in 30 ml methanol was stirred for 1 h at room temperature. After filtration, the mother liguid was stood for one week to give the colourless crystals suitable for X-ray diffraction annalysis.

S3. Refinement

C-bound H atoms were placed in geometrically idealized positions (C—H 0.93 Å) and treated as riding on their parent atoms , with $U_{iso}(H)=1.2U_{eq}(C)$. O-bound H atoms were located on a difference map and refined isotropically with the bond restraint O—H = 0.84 (2) Å.
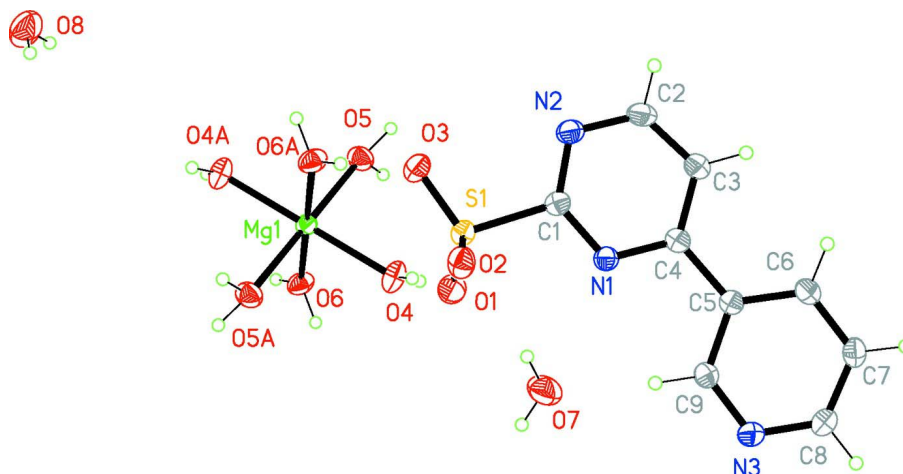
Figure 1

A portion of the crystal structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme [symmetry code: (A) -1 - x, 1 - y, 1 - z].

Hexaaquamagnesium(II) bis[4-(3-pyridyl)pyrimidine-2-sulfonate] tetrahydrate

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[Mg(H$_2$O)$_6$](C$_9$H$_6$N$_3$O$_3$S)$_2$·4H$_2$O
M_r = 676.95
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.9835 (2) Å
b = 13.3600 (3) Å
c = 16.2565 (4) Å
β = 98.724 (1)°
V = 1499.18 (7) Å^3
Z = 2

$F(000)$ = 708
D_x = 1.500 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 15164 reflections
θ = 2.0–27.5°
μ = 0.28 mm^{-1}
T = 291 K
Block, colourless
0.30 × 0.15 × 0.12 mm

Data collection

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Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan
 (*SADABS*; Bruker, 2000)
T_{min} = 0.917, T_{max} = 0.966

14712 measured reflections
3438 independent reflections
2848 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −9→9
k = −17→15
l = −19→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.04
3438 reflections
236 parameters
7 restraints

Primary atom site location: structure-invariant
 direct methods
Secondary atom site location: difference Fourier
 map
Hydrogen site location: inferred from
 neighbouring sites
H atoms treated by a mixture of independent
 and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.508P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Experimental. The structure was solved by direct methods (Bruker, 2000) and successive difference Fourier syntheses.

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*	$U_{iso}*/U_{eq}$
Mg1	0.5000	0.5000	0.5000	0.03117 (19)
S1	0.05652 (6)	0.48762 (3)	0.71033 (2)	0.03373 (13)
N1	0.1709 (2)	0.47749 (10)	0.87143 (9)	0.0339 (3)
O1	−0.12580 (18)	0.43964 (10)	0.71840 (8)	0.0441 (3)
O2	0.20815 (19)	0.41651 (10)	0.70020 (8)	0.0484 (3)
N3	0.3137 (2)	0.26720 (11)	1.05433 (9)	0.0425 (4)
N2	0.1324 (2)	0.64241 (10)	0.81545 (9)	0.0416 (3)
C4	0.2220 (2)	0.51444 (12)	0.94866 (10)	0.0320 (3)
C1	0.1302 (2)	0.54365 (12)	0.81052 (10)	0.0324 (3)
O3	0.03921 (19)	0.56801 (9)	0.64992 (8)	0.0443 (3)
C9	0.2743 (3)	0.33887 (12)	0.99732 (11)	0.0380 (4)
H9	0.2487	0.3200	0.9417	0.046*
C5	0.2691 (2)	0.44024 (12)	1.01627 (10)	0.0319 (3)
C6	0.3095 (3)	0.46696 (14)	1.09979 (10)	0.0411 (4)
H6	0.3084	0.5339	1.1155	0.049*
C8	0.3510 (3)	0.29500 (14)	1.13397 (11)	0.0448 (4)
H8	0.3782	0.2456	1.1744	0.054*
C3	0.2265 (3)	0.61758 (13)	0.96124 (11)	0.0412 (4)
H3	0.2593	0.6445	1.0142	0.049*
C7	0.3512 (3)	0.39317 (14)	1.15898 (11)	0.0466 (4)
H7	0.3792	0.4096	1.2151	0.056*
C2	0.1811 (3)	0.67811 (13)	0.89291 (12)	0.0465 (4)
H2	0.1843	0.7471	0.9006	0.056*
O7	0.4762 (3)	0.29673 (14)	0.81603 (13)	0.0812 (6)
H7A	0.407 (5)	0.342 (2)	0.791 (2)	0.137 (15)*
H7B	0.438 (5)	0.2425 (19)	0.793 (2)	0.133 (14)*
O6	0.7440 (2)	0.42088 (10)	0.48561 (9)	0.0445 (3)
O4	0.5193 (2)	0.43705 (11)	0.61644 (8)	0.0477 (3)
O5	0.6808 (2)	0.61790 (10)	0.54696 (9)	0.0467 (3)
O8	0.1251 (4)	0.72373 (16)	0.14928 (13)	0.0869 (6)
H6A	0.773 (3)	0.3654 (19)	0.5094 (15)	0.064 (7)*

H6B	0.804 (4)	0.425 (2)	0.4457 (14)	0.081 (8)*
H4B	0.422 (3)	0.4341 (17)	0.6427 (14)	0.056 (6)*
H5A	0.647 (4)	0.6623 (18)	0.5805 (15)	0.088 (9)*
H5B	0.786 (3)	0.602 (2)	0.5755 (16)	0.085 (9)*
H4A	0.626 (3)	0.438 (2)	0.6468 (15)	0.074 (8)*
H8A	0.245 (4)	0.717 (2)	0.1660 (17)	0.072 (9)*
H8B	0.055 (6)	0.681 (3)	0.169 (3)	0.168 (19)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0328 (4)	0.0308 (4)	0.0295 (4)	0.0031 (3)	0.0035 (3)	0.0025 (3)
S1	0.0337 (2)	0.0357 (2)	0.0314 (2)	−0.00007 (16)	0.00366 (16)	0.00238 (16)
N1	0.0345 (7)	0.0317 (7)	0.0342 (7)	−0.0004 (6)	0.0008 (6)	0.0002 (6)
O1	0.0399 (7)	0.0520 (7)	0.0390 (7)	−0.0115 (6)	0.0012 (5)	−0.0002 (6)
O2	0.0504 (8)	0.0518 (7)	0.0445 (7)	0.0138 (6)	0.0115 (6)	0.0012 (6)
N3	0.0540 (9)	0.0327 (7)	0.0396 (8)	−0.0044 (7)	0.0038 (7)	0.0016 (6)
N2	0.0472 (9)	0.0315 (7)	0.0440 (8)	−0.0003 (6)	−0.0003 (7)	0.0036 (6)
C4	0.0270 (8)	0.0330 (8)	0.0357 (8)	−0.0004 (6)	0.0034 (6)	−0.0023 (7)
C1	0.0271 (8)	0.0328 (8)	0.0364 (8)	−0.0010 (6)	0.0018 (6)	0.0027 (7)
O3	0.0493 (7)	0.0459 (7)	0.0370 (7)	0.0006 (6)	0.0044 (5)	0.0092 (5)
C9	0.0433 (10)	0.0354 (8)	0.0337 (8)	−0.0031 (7)	0.0011 (7)	−0.0024 (7)
C5	0.0287 (8)	0.0324 (8)	0.0343 (8)	−0.0014 (6)	0.0037 (6)	−0.0014 (6)
C6	0.0487 (10)	0.0363 (9)	0.0379 (9)	0.0024 (8)	0.0057 (8)	−0.0060 (7)
C8	0.0546 (11)	0.0429 (9)	0.0369 (9)	0.0013 (8)	0.0067 (8)	0.0076 (8)
C3	0.0481 (10)	0.0342 (8)	0.0403 (9)	−0.0014 (8)	0.0038 (8)	−0.0056 (7)
C7	0.0585 (12)	0.0504 (10)	0.0303 (9)	0.0041 (9)	0.0051 (8)	−0.0025 (8)
C2	0.0575 (12)	0.0285 (8)	0.0518 (11)	0.0014 (8)	0.0026 (9)	−0.0022 (8)
O7	0.1034 (15)	0.0525 (10)	0.0792 (12)	0.0227 (11)	−0.0132 (11)	−0.0133 (9)
O6	0.0488 (8)	0.0411 (7)	0.0463 (8)	0.0148 (6)	0.0164 (6)	0.0104 (6)
O4	0.0383 (8)	0.0696 (9)	0.0344 (7)	0.0010 (7)	0.0025 (6)	0.0124 (6)
O5	0.0434 (8)	0.0399 (7)	0.0532 (8)	0.0009 (6)	−0.0044 (7)	−0.0073 (6)
O8	0.1058 (18)	0.0780 (13)	0.0705 (12)	−0.0248 (13)	−0.0067 (12)	0.0268 (10)

Geometric parameters (Å, º)

Mg1—O6	2.0487 (13)	C5—C6	1.391 (2)
Mg1—O6^i	2.0487 (13)	C6—C7	1.378 (3)
Mg1—O4	2.0570 (13)	C6—H6	0.9300
Mg1—O4^i	2.0570 (13)	C8—C7	1.373 (3)
Mg1—O5^i	2.0893 (13)	C8—H8	0.9300
Mg1—O5	2.0893 (13)	C3—C2	1.372 (3)
S1—O3	1.4480 (13)	C3—H3	0.9300
S1—O1	1.4491 (13)	C7—H7	0.9300
S1—O2	1.4504 (13)	C2—H2	0.9300
S1—C1	1.7954 (17)	O7—H7A	0.838 (18)
N1—C1	1.326 (2)	O7—H7B	0.837 (19)
N1—C4	1.346 (2)	O6—H6A	0.85 (3)

N3—C9	1.332 (2)	O6—H6B	0.827 (17)
N3—C8	1.334 (2)	O4—H4B	0.85 (2)
N2—C1	1.322 (2)	O4—H4A	0.827 (17)
N2—C2	1.341 (2)	O5—H5A	0.862 (17)
C4—C3	1.393 (2)	O5—H5B	0.837 (17)
C4—C5	1.480 (2)	O8—H8A	0.85 (3)
C9—C5	1.391 (2)	O8—H8B	0.844 (19)
C9—H9	0.9300		
O6—Mg1—O6ⁱ	180.00 (8)	N3—C9—H9	118.1
O6—Mg1—O4	87.40 (6)	C5—C9—H9	118.1
O6ⁱ—Mg1—O4	92.60 (6)	C9—C5—C6	117.26 (16)
O6—Mg1—O4ⁱ	92.60 (6)	C9—C5—C4	119.88 (15)
O6ⁱ—Mg1—O4ⁱ	87.40 (6)	C6—C5—C4	122.86 (15)
O4—Mg1—O4ⁱ	180.0	C7—C6—C5	119.23 (16)
O6—Mg1—O5ⁱ	92.08 (6)	C7—C6—H6	120.4
O6ⁱ—Mg1—O5ⁱ	87.92 (6)	C5—C6—H6	120.4
O4—Mg1—O5ⁱ	88.83 (6)	N3—C8—C7	122.99 (17)
O4ⁱ—Mg1—O5ⁱ	91.17 (6)	N3—C8—H8	118.5
O6—Mg1—O5	87.92 (6)	C7—C8—H8	118.5
O6ⁱ—Mg1—O5	92.08 (6)	C2—C3—C4	117.84 (16)
O4—Mg1—O5	91.17 (6)	C2—C3—H3	121.1
O4ⁱ—Mg1—O5	88.83 (6)	C4—C3—H3	121.1
O5ⁱ—Mg1—O5	180.00 (5)	C8—C7—C6	119.09 (17)
O3—S1—O1	113.91 (8)	C8—C7—H7	120.5
O3—S1—O2	113.33 (8)	C6—C7—H7	120.5
O1—S1—O2	112.78 (8)	N2—C2—C3	123.04 (16)
O3—S1—C1	106.80 (8)	N2—C2—H2	118.5
O1—S1—C1	103.71 (7)	C3—C2—H2	118.5
O2—S1—C1	105.22 (8)	H7A—O7—H7B	106 (4)
C1—N1—C4	116.66 (14)	Mg1—O6—H6A	122.8 (16)
C9—N3—C8	117.68 (15)	Mg1—O6—H6B	125.8 (19)
C1—N2—C2	114.26 (15)	H6A—O6—H6B	108 (2)
N1—C4—C3	119.79 (15)	Mg1—O4—H4B	122.2 (15)
N1—C4—C5	116.42 (14)	Mg1—O4—H4A	118.0 (19)
C3—C4—C5	123.79 (15)	H4B—O4—H4A	114 (2)
N2—C1—N1	128.39 (16)	Mg1—O5—H5A	122.9 (19)
N2—C1—S1	118.06 (13)	Mg1—O5—H5B	116 (2)
N1—C1—S1	113.52 (12)	H5A—O5—H5B	97 (3)
N3—C9—C5	123.75 (16)	H8A—O8—H8B	114 (4)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O4—H4*A*⋯O1ⁱⁱ	0.83 (2)	1.94 (2)	2.7650 (19)	180 (3)
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O7—H7*A*···O2	0.84 (3)	2.12 (3)	2.923 (2)	161 (3)
O8—H8*B*···O1ᵛⁱ	0.85 (4)	2.43 (4)	3.064 (2)	132 (4)
O8—H8*A*···O7ⁱ	0.85 (3)	1.93 (3)	2.769 (3)	170 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) *x*+1/2, −*y*+3/2, *z*+1/2; (iv) *x*+1/2, −*y*+1/2, *z*−1/2; (v) −*x*+1/2, *y*−1/2, −*z*+3/2; (vi) −*x*, −*y*+1, −*z*+1.