Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Two *N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide coordination compounds

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Received 27 February 2018
Accepted 16 May 2018

Edited by A. M. Chippindale, University of Reading, England

Keywords: crystal structure; coordination compound; heterocyclic nitrogen ligand; Mn^{II} complex; Cd^{II} complex.

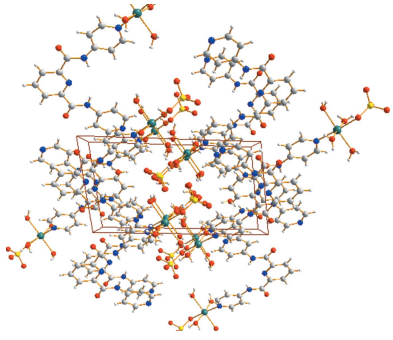
CCDC references: 1822492; 1822490

Supporting information: this article has supporting information at journals.iucr.org/e

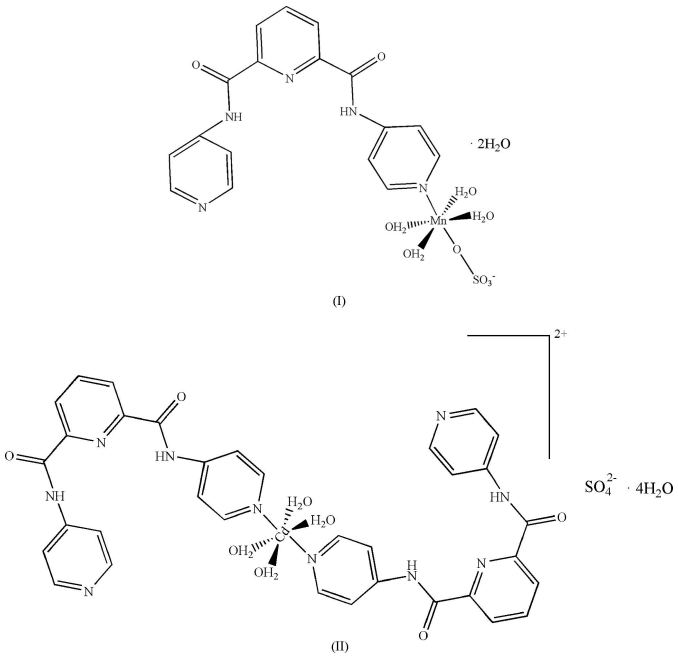
The molecular structures of tetraaqua[*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide]sulfatomanganese(II) dihydrate, $[Mn(SO_4)(C_{17}H_{13}N_5O_2)(H_2O)_4]\cdot 2H_2O$ or $[Mn(H_2L^1)(SO_4)(H_2O)_4]\cdot 2H_2O$, (I), and tetraaquabis[*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide]cadmium(II) sulfate tetrahydrate, $[Cd(C_{17}H_{13}N_5O_2)_2(H_2O)_4]SO_4\cdot 4H_2O$ or $[Cd(H_2O)_4(H_2L^1)_2]\cdot SO_4\cdot 4H_2O$, (II), both contain a central metal atom in a distorted octahedral geometry coordinated equatorially by four oxygen atoms from water molecules. In (I), the axial positions are occupied by a nitrogen atom from H_2L^1 and an oxygen atom from the sulfate anion, whereas in (II), the axial positions contain two nitrogen atoms from two different H_2L^1 ligands and the sulfate anion acts as the charge-balancing ion. π–π stacking between pyridine rings and a network of hydrogen bonds involving the water molecules and the sulfate anions play a crucial role in the molecular self-assembly of the two structures.

1. Chemical context

In recent years, the design of metal–organic complexes constructed from heterocyclic nitrogen-derivative ligands has witnessed an upsurge in interest due to their fascinating structures and potential applications in luminescence, catalysis, gas storage and separation (Perry *et al.*, 2004). The heterocyclic nitrogen-derivative ligands have σ-electron-donating ability and can form strong *M*—N covalent bonds with transition-metal ions. This bonding feature, when combined with the flexibility and length of molecular backbone within these ligands, can lead to the construction of porous coordination compounds (Gao *et al.*, 2003; Hagrman *et al.*, 1999; Li *et al.*, 2017). A variety of heterocyclic nitrogen-derivative complexes with interesting properties and topologies have been synthesized using ligands such as 4,4′-bipyridine (Fujita *et al.*, 1994), an asymmetric triazole dicarboxylate ligand (Hao *et al.*, 2018), 5-(pyridine-3-yl)pyrazole-3-carboxylic acid (Cheng *et al.*, 2016), 2-[4-(1*H*-imidazole-1-ylmethyl)-1*H*-1,2,3-triazol-1-yl] acetic acid (Yu *et al.*, 2016) and 2,2′-dihydroxy-[1,1′]binaphthalenyl-3,3′-dicarboxylate (Zheng *et al.*, 2004). Heterocyclic ligands containing aromatic systems continue to attract our interest because they can form various π–π stacking interactions between pyridine rings and direct the crystal packing and molecular assembly (Tomura & Yamashita, 2001; Li *et al.*, 2012). Here we report the synthesis and crystal structures of two new mononuclear complexes, $[Mn(H_2L^1)(SO_4)(H_2O)_4]\cdot 2H_2O$, (I), and $[Cd(H_2L^1)_2(H_2O)_4]SO_4\cdot 4H_2O$, (II), both of which contain



N,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide (H_2L^1) as the heterocyclic nitrogen ligand.



In complexes (I) and (II), the heterocyclic nitrogen ligand (H_2L^1) acts as a monodentate ligand. The results indicate that the rational design and selection of ligands with heterocyclic nitrogen systems is an effective synthetic strategy to construct complexes *via* self-assembly. The asymmetry of the [*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide ligand has resulted in some novel structures (Li *et al.*, 2012). Further study is ongoing.

2. Structural commentary

The mononuclear complex (I) crystallizes in the triclinic space group $P\overline{1}$. As shown in Fig. 1, the hexacoordinated Mn^{II} ion exhibits an octahedral geometry, arising from coordination to five water and one sulfate oxygen atoms (O3, O4, O5, O6, O10) and to one nitrogen (N1) atom of a pyridine group of the H_2L^1 ligand (Table 1). The Mn—O bond distances involving the water molecules coordinated by manganese(II) in equatorial positions lie in the range 2.1502 (17)–2.2333 (17) Å whilst the Mn—N1 and Mn—O10 distances in the axial positions are 2.2208 (17) and 2.1492 (15) Å, respectively. The bond angles around the Mn^{II} ion vary from 85.79 (7) to 177.74 (6)°. Intramolecular O4—H4···O8, N2—H20···N5 and N3—H24···N5 hydrogen bonds are also present (Table 3).

Table 1
Selected geometric parameters (Å, °) for (I).

Mn1—O10	2.1491 (15)	Mn1—N1	2.2208 (17)
Mn1—O6	2.1502 (17)	Mn1—O5	2.2298 (16)
Mn1—O4	2.1938 (17)	Mn1—O3	2.2333 (17)
O6—Mn1—O4	175.13 (6)	O6—Mn1—O3	85.79 (7)
O10—Mn1—N1	177.74 (6)	O4—Mn1—O3	90.90 (7)
O6—Mn1—O5	91.71 (6)	O5—Mn1—O3	175.51 (6)
O4—Mn1—O5	91.35 (7)		

Table 2
Selected geometric parameters (Å, °) for (II).

Cd1—N1	2.275 (3)	Cd1—O6	2.371 (3)
Cd1—O5	2.334 (2)		
$N1^i$—Cd1—N1	177.40 (15)	O5—Cd1—O6	175.09 (10)
N1—Cd1—$O5^i$	90.92 (10)	$N1^i$—Cd1—$O6^i$	90.88 (10)
N1—Cd1—O5	91.04 (10)	N1—Cd1—$O6^i$	87.31 (10)
$O5^i$—Cd1—O5	82.63 (14)	O5—Cd1—$O6^i$	92.82 (10)
N1—Cd1—O6	90.88 (10)	O6—Cd1—$O6^i$	91.79 (15)

Symmetry code: (i) $-x, y, -z+\frac{1}{2}$.

When $MnSO_4{\cdot}H_2O$ is replaced by $CdSO_4{\cdot}8/3H_2O$, complex (II) is obtained, which crystallizes in the monoclinic space group *C*2/*c*. As illustrated in Fig. 2, Cd^{II} also shows an octahedral environment coordinating four oxygen atoms from four water molecules and two axial nitrogen atoms from two symmetry-related H_2L^1 ligands (Table 2). In contrast to complex (I), the sulfate group does not coordinate to the cadmium(II) atom, but balances the compound charge as a

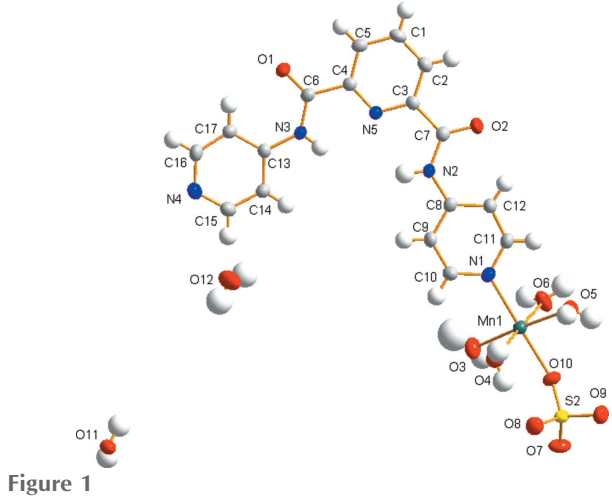


Figure 1
The molecular structure of the title complex (I) with displacement ellipsoids shown at the 50% probability level.

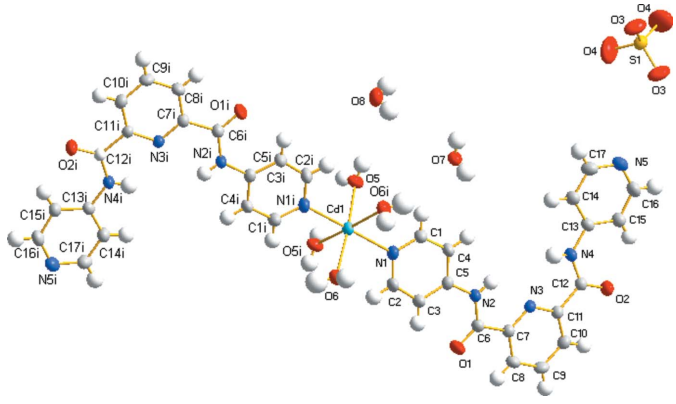


Figure 2
The molecular structure of the title complex (II) with displacement ellipsoids shown at the 50% probability level. Symmetry code: (i) $-x, y, -z+\frac{1}{2}$.

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O10^{i}	0.82	2.34	3.057 (3)	147
O4—H4···O8	0.82	1.98	2.754 (2)	158
O5—H5···O8^{ii}	0.82	1.98	2.774 (2)	163
O6—H6···O7^{i}	0.82	2.02	2.834 (2)	170
O12—H18···O9^{iii}	0.83 (3)	1.97 (3)	2.772 (3)	165 (4)
O12—H19···O7^{iv}	0.81 (4)	2.47 (4)	3.169 (3)	145 (3)
O12—H19···O9^{iv}	0.81 (4)	2.43 (4)	3.128 (3)	145 (3)
N2—H20···N5	0.85 (3)	2.34 (2)	2.733 (2)	109.0 (18)
N2—H20···O11^{v}	0.85 (3)	2.11 (3)	2.910 (3)	157 (2)
O6—H21···N4^{vi}	0.85 (3)	1.84 (3)	2.686 (3)	173 (3)
O5—H22···O12^{vi}	0.81 (3)	2.08 (3)	2.883 (3)	171 (3)
O4—H23···O9^{ii}	0.83 (3)	2.02 (4)	2.828 (2)	168 (3)
O3—H24···O12^{vii}	0.85 (4)	2.32 (4)	3.165 (3)	171 (4)
N3—H25···N5	0.85 (3)	2.28 (2)	2.709 (2)	111.4 (16)
N3—H25···O11^{v}	0.85 (3)	2.24 (2)	2.979 (2)	147 (3)
O11—H26···O7^{iii}	0.86 (3)	1.92 (3)	2.774 (2)	176 (3)
O11—H27···O1^{viii}	0.78 (3)	2.08 (3)	2.843 (2)	168 (3)

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

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O7^{ii}	0.84 (4)	2.17 (4)	2.958 (4)	156 (4)
N2—H2B···N3	0.84 (4)	2.35 (4)	2.736 (4)	109 (3)
N4—H4B···O7^{ii}	0.83 (4)	2.29 (4)	3.030 (4)	150 (4)
N4—H4B···N3	0.83 (4)	2.29 (4)	2.698 (4)	111 (3)
O5—H5A···N5^{iii}	0.80 (4)	1.91 (4)	2.704 (4)	174 (4)
O5—H5B···O3^{iii}	0.79 (4)	2.02 (4)	2.811 (4)	172 (4)
O6—H6A···O4^{iv}	0.82 (5)	1.98 (5)	2.775 (6)	161 (6)
O6—H6B···O8^{v}	0.84 (6)	2.04 (6)	2.872 (4)	173 (6)
O7—H7A···O3^{vi}	0.86 (5)	1.93 (5)	2.784 (4)	174 (4)
O7—H7B···O2^{vii}	0.82 (5)	2.13 (5)	2.912 (4)	159 (5)
O8—H8B···O5^{viii}	0.80	2.33	3.052 (4)	151
O8—H8C···O4^{vi}	0.80	2.53	3.233 (6)	147
O8—H8C···O3^{ix}	0.80	2.36	3.085 (4)	152

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

free anion. The Cd—O bond lengths lie in the range 2.334 (2)–2.371 (3) Å, the Cd—N bond length is 2.275 (3) Å, and the bond angles around the Cd^{II} cation lie in the range 82.63 (14) to 175.09 (10)°. Intramolecular N2—H2B···N3 and N4—H4B···N3 hydrogen bonds are also present (Table 4).

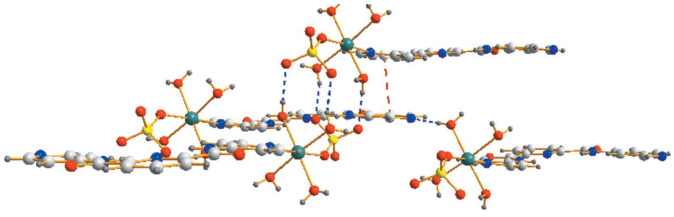


Figure 3
The weak interactions between molecules of complex (I). π–π interactions are shown as red dashed lines, hydrogen-bonding interactions as blue dashed lines.

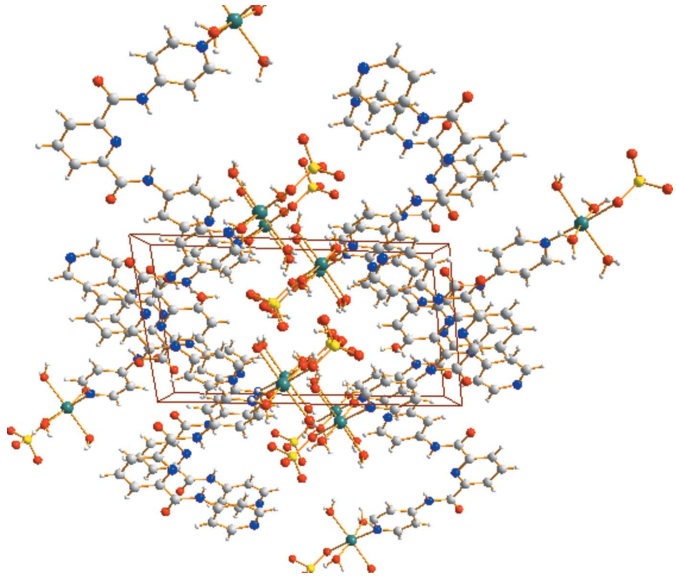


Figure 4
The molecular packing of (I) viewed along the *a* axis.

In complexes (I) and (II), the heterocyclic nitrogen ligand (H_2L^1) acts as a monodentate ligand. The results indicate that the rational design and selection of ligands with heterocyclic nitrogen systems is an effective synthetic strategy to construct complexes *via* self-assembly. The asymmetry of the [*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide ligand has resulted in some novel structures. Further study is ongoing.

3. Supramolecular features

In (I), intermolecular π–π interactions between the pyridine rings of the H_2L^1 ligands play a crucial role in molecular self-assembly, with centroid-to-centroid separations of 3.5808 (13) and 3.6269 (14) Å. In addition, a number of O—H···N and O—H···O hydrogen-bonding interactions (Table 3 and Fig. 3) connect separate mononuclear structures to produce a three-dimensional supramolecular framework (Fig. 4).

Complex (II) also extends into a three-dimensional supramolecular network (Figs. 5 and 6) *via* O—H···N and O—

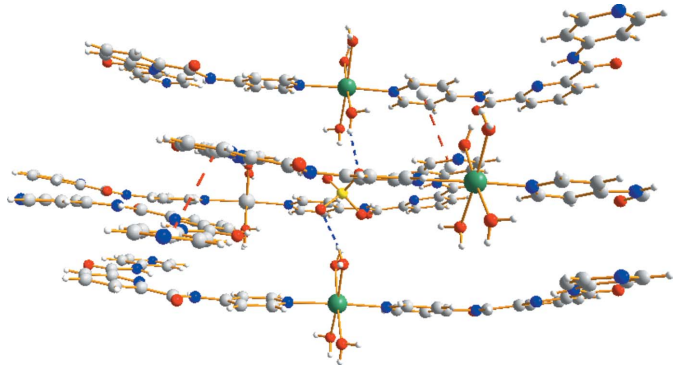


Figure 5
The weak interactions between molecules of complex (II) with hydrogen-bonding interactions and π–π interactions shown as blue and red dashed lines, respectively.

Table 5
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$[Mn(SO_4)(C_{17}H_{13}N_5O_2)(H_2O)_4\cdot 2H_2O$	$[Cd(C_{17}H_{13}N_5O_2)_2(H_2O)_4](SO_4)\cdot 4H_2O$
M_r	578.42	991.23
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $C2/c$
Temperature (K)	293	293
a, b, c (Å)	8.9333 (18), 8.9998 (18), 15.949 (3)	14.706 (3), 10.157 (2), 27.293 (6)
α, β, γ (°)	78.92 (3), 81.04 (3), 68.43 (3)	90, 99.52 (3), 90
V (Å^3)	1165.1 (5)	4020.6 (14)
Z	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.73	0.68
Crystal size (mm)	0.20 × 0.20 × 0.20	0.20 × 0.20 × 0.20
Data collection		
Diffractometer	Rigaku Saturn724	Rigaku Saturn724
Absorption correction	Multi-scan (*CrystalClear*; Rigaku/MSC, 2006)	Multi-scan (*CrystalClear*; Rigaku/MSC, 2006)
T_{min}, T_{max}	0.939, 1.000	0.780, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14157, 5299, 4440	22754, 4592, 4261
R_{int}	0.024	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.092, 1.07	0.051, 0.128, 1.14
No. of reflections	5299	4592
No. of parameters	369	313
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.45	0.81, −0.85

Computer programs: *CrystalClear* (Rigaku/MSC, 2006), *SHELXT2014/7* (Sheldrick, 2015*a*) and *SHELXL2014/7* (Sheldrick, 2015*b*).

H···O hydrogen-bonding interactions (Table 4). In (II), the centroid–centroid separations of the pyridine rings are 3.634 (2) and 3.768 (2) Å and indicating that intermolecular π–π stacking interactions of the H_2L^1 ligand are important in molecular self-assembly.

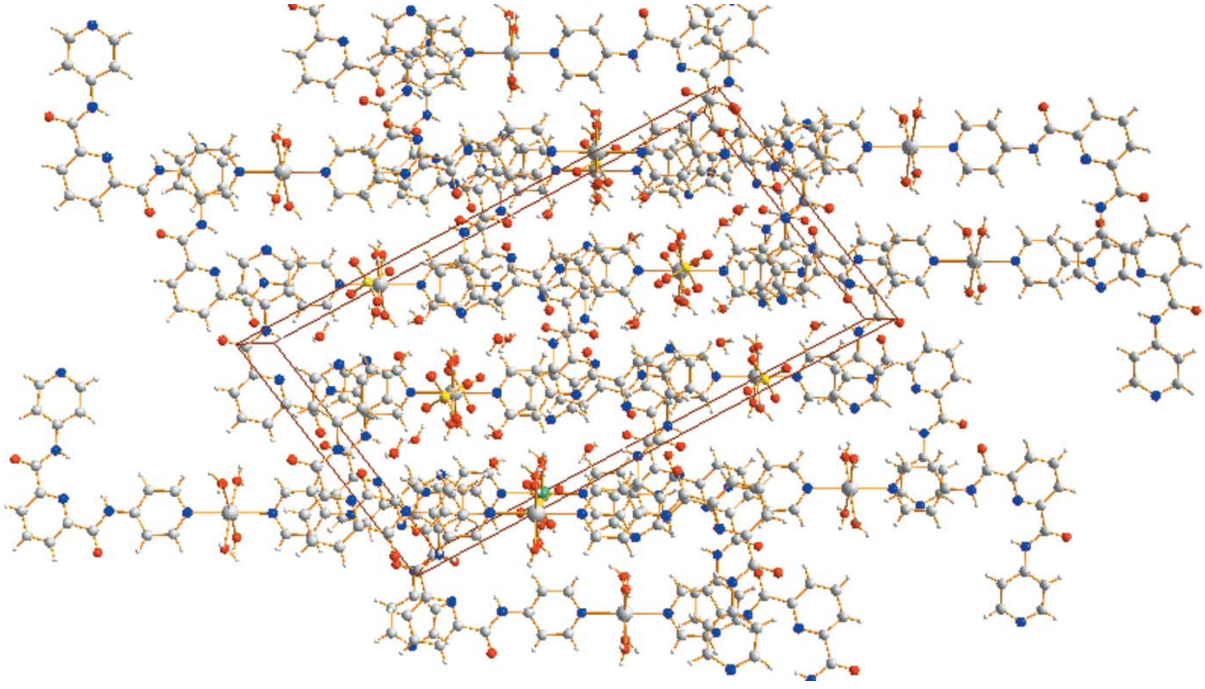


Figure 6
The molecular packing of (II) viewed along the *b* axis.

4. Database survey

A search of the Cambridge Crystallographic Database (CSD, version 5.39, update May 2018; Groom *et al.*, 2016) reveals eight structures with the H_2L^1 skeleton. These include the methyl pyridinium compound of H_2L^1 (Dorazco-González *et al.*, 2010), two Ru^+ compounds (Park *et al.*, 2006; Mishra *et al.*, 2012), two Pd^{2+} compounds (Qin *et al.*, 2002, 2003) and two Co^{2+} compounds (Singh *et al.*, 2010, 2011). There is only one Mn^{2+} coordination compound (Noveron *et al.*, 2003), with bis(hexafluoroacetylacetonato) as an ancillary ligand.

5. Synthesis and crystallization

The heterocyclic nitrogen ligand (H_2L^1) was prepared using a modified literature procedure (Qin *et al.*, 2003; Li *et al.*, 2012). In the preparation of complex (I), H_2L^1 (0.1 mmol, 0.032 g) in *N*,*N*′-dimethylformamide solution (4 mL) was gradually added to $MnSO_4{\cdot}H_2O$ (0.1 mmol, 0.017 g) in a mixed solution (3 mL, water–methanol *v*/*v* = 1/3). After standing for 5 min, the suspension was filtered and the filtrate was kept at room temperature in the dark. One week later, colourless single crystals suitable for X-ray diffraction were obtained. Complex (II) was prepared with the same procedure employed for (I) except that $CdSO_4{\cdot}8/3H_2O$ (0.1 mmol, 0.026 g) was used instead of $MnSO_4{\cdot}H_2O$.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. Water H atoms were located in a difference-Fourier map and freely refined. All other H atoms were positioned gemetrically and refined using a riding model with bond lengths of 0.93 Å (C—H, aromatic), 0.83 Å (N—H) and 0.85 Å (O—H), and with U_{iso}(H) = 1.2–1.5U_{eq}(C/N/O).

Funding information

This work was supported by the Hebei Province Science and Technology Support Program (China) (No. 16211233).

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supporting information

Acta Cryst. (2018). E**74**, 1049-1053 [https://doi.org/10.1107/S2056989018007351]

Two *N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide coordination compounds

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Computing details

For both structures, data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear* (Rigaku/MSC, 2006); data reduction: *CrystalClear* (Rigaku/MSC, 2006); program(s) used to solve structure: *SHELXT2014/7* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015b); molecular graphics: *SHELXL2014/7* (Sheldrick, 2015b); software used to prepare material for publication: *SHELXL2014/7* (Sheldrick, 2015b).

Tetraaqua[*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-dicarboxamide]sulfatomanganese(II) dihydrate (I)

Crystal data

$[Mn(SO_4)(C_{17}H_{13}N_5O_2)(H_2O)_4]\cdot 2H_2O$
M_r = 578.42
Triclinic, $P\overline{1}$
a = 8.9333 (18) Å
b = 8.9998 (18) Å
c = 15.949 (3) Å
α = 78.92 (3)°
β = 81.04 (3)°
γ = 68.43 (3)°
V = 1165.1 (5) Å^3
Z = 2
$F(000)$ = 598
D_x = 1.649 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3219 reflections
θ = 3.3–27.5°
μ = 0.73 mm^{-1}
T = 293 K
Prism, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn724 diffractometer
Detector resolution: 28.5714 pixels mm^{-1}
dtprofit.ref scans
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2006)
T_{min} = 0.939, T_{max} = 1.000
14157 measured reflections
5299 independent reflections
4440 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −11→11
k = −11→11
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.092
S = 1.07
5299 reflections
369 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.1434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2351 (2)	0.4933 (2)	−0.18307 (12)	0.0332 (4)
H1	0.2377	0.5031	−0.2424	0.040*
C2	0.1757 (2)	0.3842 (2)	−0.13011 (12)	0.0323 (4)
H2	0.1351	0.3207	−0.1530	0.039*
C3	0.1769 (2)	0.3699 (2)	−0.04191 (11)	0.0254 (4)
C4	0.2824 (2)	0.5697 (2)	−0.05814 (11)	0.0251 (4)
C5	0.2907 (2)	0.5875 (2)	−0.14691 (12)	0.0297 (4)
H5A	0.3329	0.6616	−0.1811	0.036*
C6	0.3359 (2)	0.6775 (2)	−0.01818 (12)	0.0271 (4)
C7	0.1178 (2)	0.2448 (2)	0.01474 (12)	0.0290 (4)
C8	0.0623 (2)	0.1421 (2)	0.16695 (12)	0.0263 (4)
C9	0.0734 (2)	0.1556 (2)	0.25115 (12)	0.0321 (4)
H9	0.1269	0.2202	0.2620	0.039*
C10	0.0053 (3)	0.0734 (3)	0.31810 (13)	0.0356 (5)
H10	0.0158	0.0829	0.3737	0.043*
C11	−0.0834 (2)	−0.0329 (2)	0.22587 (12)	0.0331 (4)
H11	−0.1387	−0.0971	0.2168	0.040*
C12	−0.0160 (2)	0.0415 (2)	0.15486 (12)	0.0310 (4)
H12	−0.0228	0.0248	0.1000	0.037*
C13	0.3595 (2)	0.7410 (2)	0.12187 (11)	0.0255 (4)
C14	0.3298 (2)	0.7027 (2)	0.21023 (12)	0.0315 (4)
H14	0.2766	0.6302	0.2325	0.038*
C15	0.3801 (3)	0.7733 (2)	0.26411 (13)	0.0354 (5)
H15	0.3585	0.7473	0.3230	0.042*
C16	0.4822 (2)	0.9162 (2)	0.15173 (13)	0.0345 (4)
H16	0.5342	0.9904	0.1314	0.041*
C17	0.4350 (2)	0.8534 (2)	0.09234 (12)	0.0314 (4)
H17	0.4535	0.8856	0.0339	0.038*
H18	0.663 (4)	0.456 (4)	0.455 (2)	0.092 (12)*
H19	0.587 (4)	0.454 (4)	0.389 (2)	0.086 (12)*
H20	0.167 (3)	0.295 (3)	0.1157 (15)	0.048 (7)*
H21	−0.463 (4)	−0.051 (3)	0.3149 (18)	0.070 (9)*
H22	−0.202 (4)	−0.400 (3)	0.3595 (18)	0.065 (9)*
H23	0.090 (4)	−0.300 (4)	0.4613 (18)	0.067 (9)*
H24	−0.293 (5)	0.175 (5)	0.462 (3)	0.138 (17)*
H25	0.278 (3)	0.588 (3)	0.0925 (13)	0.037 (6)*
H26	0.645 (3)	0.661 (3)	0.7711 (19)	0.065 (9)*
H27	0.593 (4)	0.694 (3)	0.8471 (19)	0.062 (10)*
Mn1	−0.21144 (3)	−0.13074 (3)	0.41352 (2)	0.02613 (9)

N1	−0.0756 (2)	−0.0201 (2)	0.30737 (10)	0.0328 (4)
N2	0.1244 (2)	0.23492 (19)	0.10064 (10)	0.0279 (3)
N3	0.3141 (2)	0.66182 (19)	0.06822 (10)	0.0286 (3)
N4	0.4582 (2)	0.8772 (2)	0.23662 (11)	0.0356 (4)
N5	0.22775 (18)	0.46192 (17)	−0.00561 (9)	0.0248 (3)
O1	0.39470 (19)	0.77213 (17)	−0.06388 (9)	0.0391 (3)
O2	0.0668 (2)	0.16229 (19)	−0.01660 (9)	0.0472 (4)
O3	−0.2975 (2)	0.0822 (2)	0.48362 (12)	0.0453 (4)
H3	−0.3920	0.0995	0.5027	0.068*
O4	0.00523 (19)	−0.23999 (19)	0.48356 (10)	0.0390 (3)
H4	−0.0150	−0.2930	0.5288	0.059*
O5	−0.14334 (18)	−0.34701 (17)	0.34781 (10)	0.0352 (3)
H5	−0.0516	−0.4073	0.3579	0.053*
O6	−0.43556 (19)	−0.01988 (18)	0.35520 (11)	0.0434 (4)
H6	−0.4679	0.0780	0.3552	0.065*
O7	−0.42592 (18)	−0.32142 (18)	0.66102 (9)	0.0429 (4)
O8	−0.14065 (18)	−0.39726 (18)	0.61734 (10)	0.0418 (4)
O9	−0.29838 (19)	−0.52446 (17)	0.56986 (10)	0.0423 (4)
O10	−0.33597 (17)	−0.24703 (16)	0.51556 (9)	0.0354 (3)
O11	0.6732 (2)	0.6493 (2)	0.82167 (11)	0.0396 (4)
O12	0.6731 (2)	0.4413 (3)	0.40463 (14)	0.0622 (5)
S2	−0.29885 (5)	−0.37332 (5)	0.59117 (3)	0.02647 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0400 (11)	0.0386 (11)	0.0209 (9)	−0.0124 (9)	−0.0036 (8)	−0.0061 (8)
C2	0.0382 (11)	0.0331 (10)	0.0289 (10)	−0.0133 (9)	−0.0051 (8)	−0.0085 (8)
C3	0.0249 (9)	0.0264 (9)	0.0261 (9)	−0.0091 (7)	−0.0029 (7)	−0.0056 (7)
C4	0.0240 (9)	0.0262 (9)	0.0249 (9)	−0.0090 (7)	−0.0022 (7)	−0.0031 (7)
C5	0.0322 (10)	0.0312 (10)	0.0245 (9)	−0.0121 (8)	−0.0010 (8)	−0.0006 (7)
C6	0.0284 (9)	0.0265 (9)	0.0268 (9)	−0.0116 (7)	−0.0020 (7)	−0.0011 (7)
C7	0.0322 (10)	0.0277 (9)	0.0288 (10)	−0.0125 (8)	−0.0012 (8)	−0.0053 (8)
C8	0.0267 (9)	0.0232 (9)	0.0286 (9)	−0.0092 (7)	−0.0031 (7)	−0.0018 (7)
C9	0.0368 (11)	0.0374 (11)	0.0308 (10)	−0.0223 (9)	−0.0039 (8)	−0.0055 (8)
C10	0.0436 (12)	0.0434 (12)	0.0268 (10)	−0.0241 (10)	−0.0015 (8)	−0.0048 (8)
C11	0.0409 (11)	0.0317 (10)	0.0333 (11)	−0.0212 (9)	−0.0024 (9)	−0.0038 (8)
C12	0.0391 (11)	0.0316 (10)	0.0279 (10)	−0.0186 (9)	−0.0028 (8)	−0.0048 (8)
C13	0.0279 (9)	0.0236 (9)	0.0259 (9)	−0.0094 (7)	−0.0044 (7)	−0.0035 (7)
C14	0.0400 (11)	0.0300 (10)	0.0277 (10)	−0.0178 (9)	0.0001 (8)	−0.0032 (8)
C15	0.0480 (12)	0.0325 (11)	0.0270 (10)	−0.0152 (9)	−0.0044 (9)	−0.0045 (8)
C16	0.0409 (11)	0.0296 (10)	0.0388 (11)	−0.0186 (9)	−0.0038 (9)	−0.0058 (8)
C17	0.0398 (11)	0.0304 (10)	0.0263 (10)	−0.0167 (9)	−0.0012 (8)	−0.0021 (8)
Mn1	0.02740 (16)	0.02532 (16)	0.02546 (16)	−0.01036 (12)	−0.00155 (11)	−0.00162 (11)
N1	0.0377 (9)	0.0340 (9)	0.0318 (9)	−0.0209 (7)	0.0006 (7)	−0.0028 (7)
N2	0.0348 (9)	0.0297 (8)	0.0259 (8)	−0.0191 (7)	−0.0026 (7)	−0.0038 (6)
N3	0.0365 (9)	0.0295 (9)	0.0255 (8)	−0.0197 (7)	−0.0016 (7)	−0.0019 (6)
N4	0.0431 (10)	0.0317 (9)	0.0358 (9)	−0.0143 (8)	−0.0077 (8)	−0.0083 (7)

N5	0.0262 (8)	0.0253 (8)	0.0242 (8)	−0.0106 (6)	−0.0022 (6)	−0.0036 (6)
O1	0.0562 (10)	0.0421 (8)	0.0287 (7)	−0.0321 (7)	0.0009 (6)	−0.0014 (6)
O2	0.0780 (12)	0.0517 (9)	0.0327 (8)	−0.0456 (9)	−0.0039 (8)	−0.0092 (7)
O3	0.0421 (9)	0.0416 (9)	0.0570 (11)	−0.0162 (7)	0.0034 (8)	−0.0218 (8)
O4	0.0335 (8)	0.0401 (9)	0.0411 (9)	−0.0111 (7)	−0.0072 (7)	−0.0007 (7)
O5	0.0352 (8)	0.0310 (8)	0.0403 (8)	−0.0115 (6)	−0.0031 (6)	−0.0079 (6)
O6	0.0481 (9)	0.0311 (8)	0.0518 (10)	−0.0030 (7)	−0.0267 (8)	−0.0118 (7)
O7	0.0436 (9)	0.0448 (9)	0.0263 (7)	−0.0043 (7)	0.0058 (6)	−0.0022 (6)
O8	0.0380 (8)	0.0427 (9)	0.0447 (9)	−0.0136 (7)	−0.0124 (7)	−0.0007 (7)
O9	0.0523 (9)	0.0318 (8)	0.0468 (9)	−0.0186 (7)	−0.0056 (7)	−0.0062 (6)
O10	0.0348 (8)	0.0363 (8)	0.0307 (7)	−0.0134 (6)	−0.0024 (6)	0.0072 (6)
O11	0.0480 (10)	0.0516 (10)	0.0280 (8)	−0.0280 (8)	−0.0045 (7)	−0.0044 (7)
O12	0.0493 (11)	0.1032 (17)	0.0448 (11)	−0.0374 (11)	−0.0040 (9)	−0.0148 (11)
S2	0.0293 (2)	0.0244 (2)	0.0232 (2)	−0.00813 (19)	−0.00086 (18)	−0.00150 (17)

Geometric parameters (Å, º)

C1—C2	1.372 (3)	C15—N4	1.334 (3)
C1—C5	1.373 (3)	C15—H15	0.9300
C1—H1	0.9300	C16—N4	1.336 (3)
C2—C3	1.389 (3)	C16—C17	1.378 (3)
C2—H2	0.9300	C16—H16	0.9300
C3—N5	1.331 (2)	C17—H17	0.9300
C3—C7	1.503 (3)	Mn1—O10	2.1491 (15)
C4—N5	1.338 (2)	Mn1—O6	2.1502 (17)
C4—C5	1.387 (2)	Mn1—O4	2.1938 (17)
C4—C6	1.500 (2)	Mn1—N1	2.2208 (17)
C5—H5A	0.9300	Mn1—O5	2.2298 (16)
C6—O1	1.226 (2)	Mn1—O3	2.2333 (17)
C6—N3	1.348 (2)	N2—H20	0.85 (2)
C7—O2	1.213 (2)	N3—H25	0.84 (2)
C7—N2	1.365 (2)	O3—H24	0.85 (4)
C8—C12	1.386 (3)	O3—H3	0.8200
C8—C9	1.393 (3)	O4—H23	0.83 (3)
C8—N2	1.394 (2)	O4—H4	0.8200
C9—C10	1.372 (3)	O5—H22	0.82 (3)
C9—H9	0.9300	O5—H5	0.8200
C10—N1	1.342 (2)	O6—H21	0.85 (3)
C10—H10	0.9300	O6—H6	0.8200
C11—N1	1.341 (3)	O7—S2	1.4711 (15)
C11—C12	1.373 (3)	O8—S2	1.4640 (15)
C11—H11	0.9300	O9—S2	1.4624 (14)
C12—H12	0.9300	O10—S2	1.4753 (14)
C13—C17	1.386 (2)	O11—H26	0.86 (3)
C13—C14	1.392 (3)	O11—H27	0.77 (3)
C13—N3	1.399 (2)	O12—H18	0.83 (4)
C14—C15	1.372 (3)	O12—H19	0.81 (4)
C14—H14	0.9300		

C2—C1—C5	118.78 (17)	C16—C17—H17	120.8
C2—C1—H1	120.6	C13—C17—H17	120.8
C5—C1—H1	120.6	O10—Mn1—O6	87.71 (7)
C1—C2—C3	119.17 (18)	O10—Mn1—O4	88.59 (6)
C1—C2—H2	120.4	O6—Mn1—O4	175.13 (6)
C3—C2—H2	120.4	O10—Mn1—N1	177.74 (6)
N5—C3—C2	122.91 (17)	O6—Mn1—N1	93.60 (7)
N5—C3—C7	118.79 (16)	O4—Mn1—N1	90.19 (7)
C2—C3—C7	118.31 (16)	O10—Mn1—O5	88.16 (6)
N5—C4—C5	123.57 (16)	O6—Mn1—O5	91.71 (6)
N5—C4—C6	117.74 (15)	O4—Mn1—O5	91.35 (7)
C5—C4—C6	118.68 (16)	N1—Mn1—O5	89.96 (6)
C1—C5—C4	118.43 (18)	O10—Mn1—O3	88.01 (7)
C1—C5—H5A	120.8	O6—Mn1—O3	85.79 (7)
C4—C5—H5A	120.8	O4—Mn1—O3	90.90 (7)
O1—C6—N3	124.43 (17)	N1—Mn1—O3	93.91 (7)
O1—C6—C4	119.88 (16)	O5—Mn1—O3	175.51 (6)
N3—C6—C4	115.69 (16)	C11—N1—C10	115.82 (17)
O2—C7—N2	124.42 (18)	C11—N1—Mn1	119.92 (13)
O2—C7—C3	120.07 (17)	C10—N1—Mn1	124.00 (13)
N2—C7—C3	115.50 (16)	C7—N2—C8	126.96 (16)
C12—C8—C9	117.44 (17)	C7—N2—H20	116.9 (16)
C12—C8—N2	124.29 (17)	C8—N2—H20	116.0 (16)
C9—C8—N2	118.22 (16)	C6—N3—C13	127.71 (16)
C10—C9—C8	119.91 (17)	C6—N3—H25	115.3 (14)
C10—C9—H9	120.0	C13—N3—H25	116.6 (14)
C8—C9—H9	120.0	C15—N4—C16	116.70 (17)
N1—C10—C9	123.33 (18)	C3—N5—C4	117.08 (15)
N1—C10—H10	118.3	Mn1—O3—H24	125 (3)
C9—C10—H10	118.3	Mn1—O3—H3	109.5
N1—C11—C12	125.09 (18)	H24—O3—H3	105.1
N1—C11—H11	117.5	Mn1—O4—H23	120 (2)
C12—C11—H11	117.5	Mn1—O4—H4	109.5
C11—C12—C8	118.35 (18)	H23—O4—H4	105.3
C11—C12—H12	120.8	Mn1—O5—H22	117 (2)
C8—C12—H12	120.8	Mn1—O5—H5	109.5
C17—C13—C14	117.86 (17)	H22—O5—H5	107.8
C17—C13—N3	123.86 (17)	Mn1—O6—H21	126.4 (19)
C14—C13—N3	118.27 (16)	Mn1—O6—H6	109.5
C15—C14—C13	119.23 (18)	H21—O6—H6	116.0
C15—C14—H14	120.4	S2—O10—Mn1	139.12 (9)
C13—C14—H14	120.4	H26—O11—H27	103 (3)
N4—C15—C14	123.52 (19)	H18—O12—H19	111 (3)
N4—C15—H15	118.2	O9—S2—O8	109.64 (9)
C14—C15—H15	118.2	O9—S2—O7	109.31 (10)
N4—C16—C17	124.19 (18)	O8—S2—O7	110.22 (9)
N4—C16—H16	117.9	O9—S2—O10	109.28 (9)

C17—C16—H16	117.9	O8—S2—O10	110.20 (9)
C16—C17—C13	118.42 (18)	O7—S2—O10	108.16 (9)
C5—C1—C2—C3	−1.4 (3)	C14—C13—C17—C16	2.5 (3)
C1—C2—C3—N5	2.6 (3)	N3—C13—C17—C16	−176.26 (18)
C1—C2—C3—C7	−177.44 (18)	C12—C11—N1—C10	−0.1 (3)
C2—C1—C5—C4	−0.8 (3)	C12—C11—N1—Mn1	174.26 (16)
N5—C4—C5—C1	2.1 (3)	C9—C10—N1—C11	1.7 (3)
C6—C4—C5—C1	−177.26 (17)	C9—C10—N1—Mn1	−172.42 (16)
N5—C4—C6—O1	176.21 (17)	O2—C7—N2—C8	−6.2 (3)
C5—C4—C6—O1	−4.4 (3)	C3—C7—N2—C8	172.84 (17)
N5—C4—C6—N3	−3.9 (2)	C12—C8—N2—C7	0.8 (3)
C5—C4—C6—N3	175.47 (17)	C9—C8—N2—C7	−176.46 (18)
N5—C3—C7—O2	178.03 (18)	O1—C6—N3—C13	−3.4 (3)
C2—C3—C7—O2	−1.9 (3)	C4—C6—N3—C13	176.79 (17)
N5—C3—C7—N2	−1.1 (3)	C17—C13—N3—C6	0.8 (3)
C2—C3—C7—N2	179.01 (17)	C14—C13—N3—C6	−177.94 (19)
C12—C8—C9—C10	−1.3 (3)	C14—C15—N4—C16	2.2 (3)
N2—C8—C9—C10	176.22 (18)	C17—C16—N4—C15	−1.5 (3)
C8—C9—C10—N1	−1.0 (3)	C2—C3—N5—C4	−1.4 (3)
N1—C11—C12—C8	−2.1 (3)	C7—C3—N5—C4	178.70 (16)
C9—C8—C12—C11	2.7 (3)	C5—C4—N5—C3	−1.0 (3)
N2—C8—C12—C11	−174.63 (18)	C6—C4—N5—C3	178.35 (15)
C17—C13—C14—C15	−1.9 (3)	Mn1—O10—S2—O9	−100.05 (14)
N3—C13—C14—C15	176.98 (18)	Mn1—O10—S2—O8	20.49 (16)
C13—C14—C15—N4	−0.6 (3)	Mn1—O10—S2—O7	141.04 (13)
N4—C16—C17—C13	−0.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O10^{i}	0.82	2.34	3.057 (3)	147
O4—H4···O8	0.82	1.98	2.754 (2)	158
O5—H5···O8^{ii}	0.82	1.98	2.774 (2)	163
O6—H6···O7^{i}	0.82	2.02	2.834 (2)	170
O12—H18···O9^{iii}	0.83 (3)	1.97 (3)	2.772 (3)	165 (4)
O12—H19···O7^{iv}	0.81 (4)	2.47 (4)	3.169 (3)	145 (3)
O12—H19···O9^{iv}	0.81 (4)	2.43 (4)	3.128 (3)	145 (3)
N2—H20···N5	0.85 (3)	2.34 (2)	2.733 (2)	109.0 (18)
N2—H20···O11^{v}	0.85 (3)	2.11 (3)	2.910 (3)	157 (2)
O6—H21···N4^{vi}	0.85 (3)	1.84 (3)	2.686 (3)	173 (3)
O5—H22···O12^{vi}	0.81 (3)	2.08 (3)	2.883 (3)	171 (3)
O4—H23···O9^{ii}	0.83 (3)	2.02 (4)	2.828 (2)	168 (3)
O3—H24···O12^{vii}	0.85 (4)	2.32 (4)	3.165 (3)	171 (4)
N3—H25···N5	0.85 (3)	2.28 (2)	2.709 (2)	111.4 (16)
N3—H25···O11^{v}	0.85 (3)	2.24 (2)	2.979 (2)	147 (3)

O11—H26···O7^{iii}	0.86 (3)	1.92 (3)	2.774 (2)	176 (3)
O11—H27···O1^{viii}	0.78 (3)	2.08 (3)	2.843 (2)	168 (3)

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

Tetraaquabis[*N*,*N*′-bis(pyridin-4-yl)pyridine-2,6-\ dicarboxamide]cadmium(II) sulfate tetrahydrate (II)

Crystal data

$[Cd(C_{17}H_{13}N_5O_2)_2(H_2O)_4]SO_4·4H_2O$
M_r = 991.23
Monoclinic, *C*2/*c*
a = 14.706 (3) Å
b = 10.157 (2) Å
c = 27.293 (6) Å
β = 99.52 (3)°
V = 4020.6 (14) Å^3
Z = 4
F(000) = 2032
D_x = 1.638 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5365 reflections
θ = 3.0–27.5°
μ = 0.68 mm^{-1}
T = 293 K
Prism, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn724 diffractometer
Detector resolution: 28.5714 pixels mm^{-1}
dtprofit.ref scans
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2006)
T_{min} = 0.780, T_{max} = 1.000
22754 measured reflections
4592 independent reflections
4261 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −19→19
k = −13→13
l = −35→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
S = 1.14
4592 reflections
313 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 3.9753P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.85 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1669 (2)	0.0681 (3)	0.18881 (12)	0.0366 (7)
H1A	0.2000	0.0777	0.2207	0.044*
C2	0.0297 (2)	0.0417 (4)	0.13698 (13)	0.0385 (7)
H2A	−0.0341	0.0331	0.1326	0.046*
C3	0.0710 (2)	0.0386 (3)	0.09526 (12)	0.0355 (7)
H3A	0.0362	0.0267	0.0639	0.043*

C4	0.2149 (2)	0.0669 (3)	0.14971 (12)	0.0343 (7)
H4A	0.2788	0.0747	0.1553	0.041*
C5	0.1666 (2)	0.0538 (3)	0.10146 (11)	0.0296 (6)
C6	0.1819 (2)	0.0643 (3)	0.01320 (11)	0.0319 (6)
C7	0.2508 (2)	0.0910 (3)	−0.02056 (11)	0.0292 (6)
C8	0.2192 (2)	0.0944 (3)	−0.07171 (11)	0.0356 (7)
H8A	0.1570	0.0823	−0.0842	0.043*
C9	0.2815 (2)	0.1161 (4)	−0.10331 (11)	0.0375 (7)
H9A	0.2625	0.1169	−0.1375	0.045*
C10	0.3732 (2)	0.1366 (3)	−0.08306 (11)	0.0340 (6)
H10A	0.4170	0.1505	−0.1035	0.041*
C11	0.39861 (19)	0.1362 (3)	−0.03202 (10)	0.0285 (6)
C12	0.49709 (19)	0.1659 (3)	−0.00939 (11)	0.0308 (6)
C13	0.59738 (19)	0.1960 (3)	0.07196 (11)	0.0305 (6)
C14	0.5979 (2)	0.1960 (3)	0.12313 (12)	0.0377 (7)
H14A	0.5433	0.1866	0.1357	0.045*
C15	0.6807 (2)	0.2147 (3)	0.05531 (12)	0.0360 (7)
H15A	0.6831	0.2191	0.0215	0.043*
C16	0.7602 (2)	0.2269 (4)	0.09021 (13)	0.0412 (7)
H16A	0.8158	0.2380	0.0787	0.049*
C17	0.6805 (2)	0.2102 (4)	0.15466 (12)	0.0424 (8)
H17A	0.6798	0.2106	0.1887	0.051*
Cd1	0.0000	0.06136 (3)	0.2500	0.03132 (12)
H5A	0.133 (3)	0.229 (5)	0.3090 (16)	0.053 (12)*
H6A	−0.088 (4)	−0.177 (5)	0.2195 (19)	0.068 (16)*
H7A	0.441 (3)	1.003 (5)	0.1380 (18)	0.058 (13)*
H2B	0.274 (3)	0.069 (4)	0.0709 (14)	0.039 (10)*
H4B	0.470 (3)	0.153 (4)	0.0549 (15)	0.050 (11)*
H5B	0.071 (3)	0.305 (4)	0.2858 (15)	0.044 (12)*
H6B	−0.160 (4)	−0.097 (6)	0.218 (2)	0.09 (2)*
H7B	0.415 (3)	0.948 (5)	0.0934 (19)	0.066 (16)*
N1	0.07517 (18)	0.0563 (3)	0.18357 (10)	0.0358 (6)
N2	0.21691 (18)	0.0606 (3)	0.06249 (9)	0.0306 (5)
N3	0.33872 (16)	0.1131 (3)	−0.00068 (9)	0.0280 (5)
N4	0.51311 (17)	0.1742 (3)	0.04068 (10)	0.0333 (6)
N5	0.76221 (19)	0.2236 (3)	0.13927 (11)	0.0415 (6)
O1	0.10075 (16)	0.0500 (3)	−0.00425 (9)	0.0533 (7)
O2	0.55554 (15)	0.1802 (3)	−0.03618 (8)	0.0429 (6)
O3	0.9969 (2)	0.4750 (3)	0.20625 (10)	0.0606 (8)
O4	0.9169 (3)	0.6394 (5)	0.24461 (16)	0.1008 (14)
O5	0.09316 (17)	0.2339 (3)	0.28544 (10)	0.0404 (5)
O6	−0.10537 (19)	−0.1011 (3)	0.21310 (10)	0.0440 (6)
O7	0.41102 (16)	1.0167 (3)	0.10871 (10)	0.0425 (6)
O8	0.29730 (18)	0.8984 (3)	0.27805 (11)	0.0568 (7)
H8B	0.3077	0.8417	0.2594	0.068*
H8C	0.3420	0.9441	0.2797	0.068*
S1	1.0000	0.56069 (11)	0.2500	0.0352 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0307 (15)	0.0508 (19)	0.0287 (15)	0.0013 (13)	0.0059 (12)	0.0002 (13)
C2	0.0257 (14)	0.053 (2)	0.0381 (17)	−0.0061 (13)	0.0093 (12)	−0.0052 (14)
C3	0.0271 (14)	0.0473 (19)	0.0322 (16)	−0.0053 (13)	0.0052 (12)	−0.0024 (13)
C4	0.0229 (13)	0.0478 (18)	0.0324 (15)	0.0003 (12)	0.0053 (11)	0.0005 (13)
C5	0.0283 (14)	0.0314 (15)	0.0306 (15)	0.0002 (11)	0.0096 (11)	−0.0007 (11)
C6	0.0272 (14)	0.0381 (16)	0.0308 (15)	−0.0031 (12)	0.0059 (11)	−0.0019 (12)
C7	0.0278 (14)	0.0311 (14)	0.0294 (14)	−0.0009 (11)	0.0065 (11)	−0.0013 (11)
C8	0.0295 (15)	0.0455 (18)	0.0300 (15)	−0.0024 (13)	−0.0002 (12)	−0.0002 (13)
C9	0.0408 (17)	0.0463 (18)	0.0237 (14)	−0.0014 (14)	0.0004 (12)	0.0002 (13)
C10	0.0355 (15)	0.0386 (16)	0.0298 (15)	−0.0006 (13)	0.0108 (12)	0.0005 (13)
C11	0.0267 (13)	0.0321 (15)	0.0274 (13)	0.0008 (11)	0.0070 (11)	0.0032 (11)
C12	0.0259 (13)	0.0360 (15)	0.0311 (14)	0.0010 (12)	0.0066 (11)	0.0010 (12)
C13	0.0259 (13)	0.0319 (15)	0.0332 (15)	−0.0029 (11)	0.0030 (11)	0.0016 (12)
C14	0.0323 (15)	0.0462 (19)	0.0353 (16)	−0.0061 (13)	0.0074 (12)	0.0005 (14)
C15	0.0309 (15)	0.0424 (17)	0.0351 (16)	−0.0044 (13)	0.0072 (12)	0.0025 (14)
C16	0.0288 (15)	0.0464 (19)	0.0484 (19)	−0.0065 (14)	0.0061 (13)	0.0000 (15)
C17	0.0440 (18)	0.049 (2)	0.0331 (16)	−0.0090 (15)	0.0018 (14)	−0.0002 (15)
Cd1	0.02672 (17)	0.0394 (2)	0.02952 (19)	0.000	0.00968 (12)	0.000
N1	0.0284 (13)	0.0469 (16)	0.0347 (14)	−0.0021 (11)	0.0129 (11)	−0.0017 (11)
N2	0.0226 (12)	0.0429 (15)	0.0273 (12)	−0.0019 (10)	0.0069 (10)	0.0009 (10)
N3	0.0258 (11)	0.0339 (13)	0.0252 (11)	0.0010 (10)	0.0071 (9)	0.0003 (10)
N4	0.0247 (12)	0.0453 (16)	0.0306 (13)	−0.0045 (11)	0.0071 (10)	0.0017 (11)
N5	0.0343 (14)	0.0443 (16)	0.0436 (15)	−0.0072 (12)	−0.0007 (12)	0.0000 (13)
O1	0.0270 (12)	0.096 (2)	0.0367 (13)	−0.0126 (12)	0.0036 (10)	−0.0016 (13)
O2	0.0326 (11)	0.0639 (17)	0.0348 (12)	−0.0061 (11)	0.0131 (9)	−0.0010 (11)
O3	0.096 (2)	0.0527 (17)	0.0318 (13)	−0.0022 (16)	0.0057 (14)	−0.0026 (12)
O4	0.093 (3)	0.111 (3)	0.104 (3)	0.059 (3)	0.032 (2)	0.014 (3)
O5	0.0357 (12)	0.0384 (13)	0.0426 (14)	0.0004 (11)	−0.0071 (10)	−0.0025 (11)
O6	0.0364 (13)	0.0438 (15)	0.0518 (15)	−0.0051 (11)	0.0077 (11)	−0.0037 (12)
O7	0.0332 (12)	0.0613 (17)	0.0326 (13)	0.0018 (11)	0.0038 (10)	0.0011 (12)
O8	0.0448 (14)	0.0694 (19)	0.0610 (17)	0.0013 (13)	0.0230 (13)	−0.0069 (14)
S1	0.0403 (6)	0.0326 (5)	0.0341 (6)	0.000	0.0102 (4)	0.000

Geometric parameters (Å, º)

C1—N1	1.338 (4)	C14—C17	1.375 (4)
C1—C4	1.373 (4)	C14—H14A	0.9300
C1—H1A	0.9300	C15—C16	1.386 (4)
C2—N1	1.344 (4)	C15—H15A	0.9300
C2—C3	1.377 (5)	C16—N5	1.335 (4)
C2—H2A	0.9300	C16—H16A	0.9300
C3—C5	1.397 (4)	C17—N5	1.344 (4)
C3—H3A	0.9300	C17—H17A	0.9300
C4—C5	1.395 (4)	Cd1—N1^{i}	2.275 (3)
C4—H4A	0.9300	Cd1—N1	2.275 (3)

C5—N2	1.394 (4)	Cd1—O5^{i}	2.334 (2)
C6—O1	1.218 (4)	Cd1—O5	2.334 (2)
C6—N2	1.359 (4)	Cd1—O6	2.371 (3)
C6—C7	1.503 (4)	Cd1—O6^{i}	2.371 (3)
C7—N3	1.336 (4)	N2—H2B	0.84 (4)
C7—C8	1.397 (4)	N4—H4B	0.83 (4)
C8—C9	1.376 (5)	O3—S1	1.472 (3)
C8—H8A	0.9300	O4—S1	1.447 (3)
C9—C10	1.386 (4)	O5—H5A	0.79 (4)
C9—H9A	0.9300	O5—H5B	0.79 (4)
C10—C11	1.381 (4)	O6—H6A	0.82 (5)
C10—H10A	0.9300	O6—H6B	0.85 (6)
C11—N3	1.346 (4)	O7—H7A	0.86 (5)
C11—C12	1.508 (4)	O7—H7B	0.82 (5)
C12—O2	1.226 (4)	O8—H8B	0.8001
C12—N4	1.350 (4)	O8—H8C	0.8000
C13—C15	1.389 (4)	S1—O4^{ii}	1.447 (3)
C13—C14	1.395 (4)	S1—O3^{ii}	1.472 (3)
C13—N4	1.401 (4)		

N1—C1—C4	123.7 (3)	N5—C16—H16A	117.9
N1—C1—H1A	118.2	C15—C16—H16A	117.9
C4—C1—H1A	118.2	N5—C17—C14	123.9 (3)
N1—C2—C3	124.5 (3)	N5—C17—H17A	118.0
N1—C2—H2A	117.7	C14—C17—H17A	118.0
C3—C2—H2A	117.7	N1^{i}—Cd1—N1	177.40 (15)
C2—C3—C5	118.1 (3)	N1^{i}—Cd1—O5^{i}	91.04 (10)
C2—C3—H3A	121.0	N1—Cd1—O5^{i}	90.92 (10)
C5—C3—H3A	121.0	N1^{i}—Cd1—O5	90.92 (10)
C1—C4—C5	119.2 (3)	N1—Cd1—O5	91.04 (10)
C1—C4—H4A	120.4	O5^{i}—Cd1—O5	82.63 (14)
C5—C4—H4A	120.4	N1^{i}—Cd1—O6	87.31 (10)
N2—C5—C4	117.6 (3)	N1—Cd1—O6	90.88 (10)
N2—C5—C3	124.3 (3)	O5^{i}—Cd1—O6	92.82 (10)
C4—C5—C3	118.0 (3)	O5—Cd1—O6	175.09 (10)
O1—C6—N2	124.8 (3)	N1^{i}—Cd1—O6^{i}	90.88 (10)
O1—C6—C7	119.9 (3)	N1—Cd1—O6^{i}	87.31 (10)
N2—C6—C7	115.3 (2)	O5^{i}—Cd1—O6^{i}	175.09 (10)
N3—C7—C8	122.8 (3)	O5—Cd1—O6^{i}	92.82 (10)
N3—C7—C6	119.2 (3)	O6—Cd1—O6^{i}	91.79 (15)
C8—C7—C6	118.0 (3)	C1—N1—C2	116.5 (3)
C9—C8—C7	118.9 (3)	C1—N1—Cd1	121.8 (2)
C9—C8—H8A	120.5	C2—N1—Cd1	121.7 (2)
C7—C8—H8A	120.5	C6—N2—C5	126.5 (3)
C8—C9—C10	118.6 (3)	C6—N2—H2B	118 (3)
C8—C9—H9A	120.7	C5—N2—H2B	116 (3)
C10—C9—H9A	120.7	C7—N3—C11	117.5 (2)
C11—C10—C9	119.0 (3)	C12—N4—C13	128.0 (3)

C11—C10—H10A	120.5	C12—N4—H4B	116 (3)
C9—C10—H10A	120.5	C13—N4—H4B	115 (3)
N3—C11—C10	123.1 (3)	C16—N5—C17	116.3 (3)
N3—C11—C12	117.4 (2)	Cd1—O5—H5A	126 (3)
C10—C11—C12	119.6 (3)	Cd1—O5—H5B	119 (3)
O2—C12—N4	125.0 (3)	H5A—O5—H5B	107 (4)
O2—C12—C11	120.0 (3)	Cd1—O6—H6A	114 (4)
N4—C12—C11	115.0 (2)	Cd1—O6—H6B	118 (4)
C15—C13—C14	117.9 (3)	H6A—O6—H6B	106 (5)
C15—C13—N4	124.2 (3)	H7A—O7—H7B	106 (5)
C14—C13—N4	117.9 (3)	H8B—O8—H8C	102.2
C17—C14—C13	119.0 (3)	O4^{ii}—S1—O4	112.9 (4)
C17—C14—H14A	120.5	O4^{ii}—S1—O3	108.8 (2)
C13—C14—H14A	120.5	O4—S1—O3	109.4 (2)
C16—C15—C13	118.5 (3)	O4^{ii}—S1—O3^{ii}	109.4 (2)
C16—C15—H15A	120.7	O4—S1—O3^{ii}	108.8 (2)
C13—C15—H15A	120.7	O3—S1—O3^{ii}	107.5 (2)
N5—C16—C15	124.3 (3)		

N1—C2—C3—C5	−1.2 (5)	C14—C13—C15—C16	2.7 (5)
N1—C1—C4—C5	0.6 (5)	N4—C13—C15—C16	−175.9 (3)
C1—C4—C5—N2	176.3 (3)	C13—C15—C16—N5	−1.0 (6)
C1—C4—C5—C3	−1.9 (5)	C13—C14—C17—N5	−0.4 (6)
C2—C3—C5—N2	−176.0 (3)	C4—C1—N1—C2	0.4 (5)
C2—C3—C5—C4	2.1 (5)	C4—C1—N1—Cd1	−179.3 (3)
O1—C6—C7—N3	176.6 (3)	C3—C2—N1—C1	−0.1 (5)
N2—C6—C7—N3	−2.2 (4)	C3—C2—N1—Cd1	179.6 (3)
O1—C6—C7—C8	−1.9 (5)	O1—C6—N2—C5	−7.0 (5)
N2—C6—C7—C8	179.3 (3)	C7—C6—N2—C5	171.6 (3)
N3—C7—C8—C9	2.8 (5)	C4—C5—N2—C6	−172.5 (3)
C6—C7—C8—C9	−178.7 (3)	C3—C5—N2—C6	5.6 (5)
C7—C8—C9—C10	−1.5 (5)	C8—C7—N3—C11	−1.8 (4)
C8—C9—C10—C11	−0.7 (5)	C6—C7—N3—C11	179.8 (3)
C9—C10—C11—N3	1.9 (5)	C10—C11—N3—C7	−0.6 (5)
C9—C10—C11—C12	−176.6 (3)	C12—C11—N3—C7	177.9 (3)
N3—C11—C12—O2	175.8 (3)	O2—C12—N4—C13	−2.2 (5)
C10—C11—C12—O2	−5.6 (5)	C11—C12—N4—C13	177.7 (3)
N3—C11—C12—N4	−4.0 (4)	C15—C13—N4—C12	0.9 (5)
C10—C11—C12—N4	174.6 (3)	C14—C13—N4—C12	−177.7 (3)
C15—C13—C14—C17	−2.1 (5)	C15—C16—N5—C17	−1.4 (6)
N4—C13—C14—C17	176.6 (3)	C14—C17—N5—C16	2.1 (6)

Symmetry codes: (i) −*x*, *y*, −*z*+1/2; (ii) −*x*+2, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O7^{iii}	0.84 (4)	2.17 (4)	2.958 (4)	156 (4)

N2—H2*B*···N3	0.84 (4)	2.35 (4)	2.736 (4)	109 (3)
N4—H4*B*···O7^{iii}	0.83 (4)	2.29 (4)	3.030 (4)	150 (4)
N4—H4*B*···N3	0.83 (4)	2.29 (4)	2.698 (4)	111 (3)
O5—H5*A*···N5^{iv}	0.80 (4)	1.91 (4)	2.704 (4)	174 (4)
O5—H5*B*···O3^{iv}	0.79 (4)	2.02 (4)	2.811 (4)	172 (4)
O6—H6*A*···O4^{v}	0.82 (5)	1.98 (5)	2.775 (6)	161 (6)
O6—H6*B*···O8^{vi}	0.84 (6)	2.04 (6)	2.872 (4)	173 (6)
O7—H7*A*···O3^{vii}	0.86 (5)	1.93 (5)	2.784 (4)	174 (4)
O7—H7*B*···O2^{viii}	0.82 (5)	2.13 (5)	2.912 (4)	159 (5)
O8—H8*B*···O5^{ix}	0.80	2.33	3.052 (4)	151
O8—H8*C*···O4^{vii}	0.80	2.53	3.233 (6)	147
O8—H8*C*···O3^{x}	0.80	2.36	3.085 (4)	152

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