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Diaquabis[5-(5-carboxy-2-pyridyl)tetrazolato-κ^2N^1,N^5]cadmium(II) dihydrate

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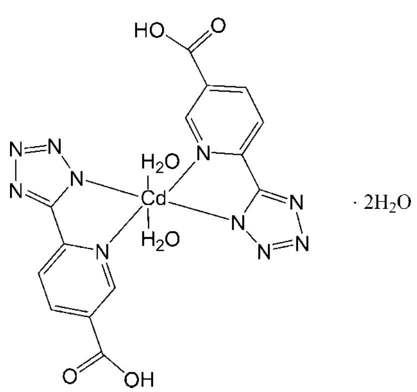
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.078; data-to-parameter ratio = 13.4.

In the title complex, $[Cd(C_7H_4N_5O_2)_2(H_2O)_2]\cdot 2H_2O$, the water-coordinated Cd^{II} atom ($\overline{1}$ symmetry) is coordinated by four N atoms from two symmetry-related 3-carboxypyidyl-6-tetrazolato ligands, forming a distorted octahedral complex. The uncoordinated water molecules connect the mononuclear units into a layer structure through O—H···N and O—H···O hydrogen bonds; similar hydrogen bonds between coordinated water molecules and anionic groups result in a three-dimensional structure.

Related literature

For background, see: Xiong *et al.* (2002)



Experimental

Crystal data

$[Cd(C_7H_4N_5O_2)_2(H_2O)_2]\cdot 2H_2O$
M_r = 564.77
Triclinic, $P\overline{1}$
a = 6.1018 (2) Å
b = 7.3805 (1) Å
c = 12.383 (2) Å
α = 84.17 (3)°
β = 88.91 (3)°
γ = 65.71 (2)°
V = 505.51 (8) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000) T_{min} = 0.773, T_{max} = 1.000 (expected range = 0.615–0.795)
3817 measured reflections
2286 independent reflections
2104 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.078
S = 1.10
2286 reflections
171 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cd1—N5	2.293 (2)	Cd1—N1	2.396 (2)
Cd1—O3	2.312 (3)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···N2	0.85 (3)	2.02 (3)	2.860 (4)	166 (4)
O4—H4A···O2^{ii}	0.85 (3)	1.92 (3)	2.767 (4)	170 (4)
O1—H1···O4^{iii}	0.89 (3)	1.68 (3)	2.566 (4)	170 (5)
O3—H3B···N4^{iv}	0.86 (2)	1.96 (3)	2.804 (4)	168 (4)
O3—H3A···N3^{v}	0.90 (2)	1.92 (2)	2.806 (4)	172 (3)

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

Data collection: *CrystalClear* (Rigaku, 2000); 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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2554).

References

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supplementary materials

Acta Cryst. (2009). E**65**, m378 [**doi:10.1107/S1600536809007399**]

Diaquabis[5-(5-carboxy-2-pyridyl)tetrazolato-κ^2N^1,N^5]cadmium(II) dihydrate

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Comment

Hydrothermal reactions involving *in situ* ligand synthesis have attracted great interests (Xiong *et al.*, 2002). In the contribution, we report the title mononuclear complex (I) based on tetrazol ligand obtained by *in situ* ligand synthesis.

In the structure of (I), the ligand chelates Cd(II) center through pyridyl N and tetrazol N to form a centrosymmetrical mononuclear complex. Two coordinated water molecules complete the octahedral geometry of Cd(II) center (Fig.1). Two solvent water molecules and carboxylic groups of the ligands form a synthon $R_4^4(12)$ which connects mononuclear unit into a two-dimensional layer structure through hydrogen bonds between solvent water and tetrazol groups (Table. 2). The hydrogen bonds between coordinated water molecules and tetrazol groups result in a three-dimensional structure (Fig.2).

Experimental

A mixture of $Cd(NO_3)_2.4H_2O$ (77 mg, 0.25 mmol), sodium azide(33 mg, 0.5 mmol) and 6-cyanopyridine-3-carboxylic acid (74 mg, 0.5 mmol) was suspended in water (10 ml) and heated in a teflon-lined steel bomb at 160 ° C for 3 days. The colorless crystals were obtained.

Refinement

H atoms bonded to C were located geometrically (C—H = 0.95 Å) with $U_{iso}(H) = 1.2 U_{eq}(C)$. H atoms bonded to O were located by difference maps and refined with a distance restraint of O—H = 0.87 (3) Å. The displacement factors were freely refined.

Figures

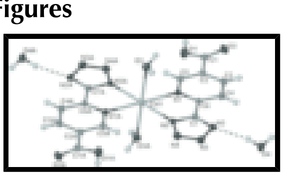


Fig. 1. *ORTEP* of complex (I) with 30% thermal ellipsoids. A = 1 - *x*, -*y*, 1 - *z*

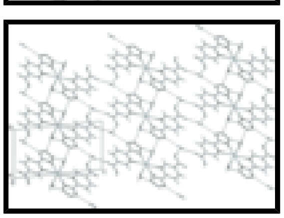


Fig. 2. The packing structure viewed along *a* axis.

Diaquabis[5-(5-carboxy-2-pyridyl)tetrazolato- κ$^2N^1$,N^5]cadmium(II) dihydrate

Crystal data

$[Cd(C_7H_4N_5O_2)_2(H_2O)_2]\cdot 2H_2O$	$Z = 1$
$M_r = 564.77$	$F_{000} = 282$
Triclinic, $P\overline{1}$	$D_x = 1.855$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.1018$ (2) Å	Cell parameters from 612 reflections
$b = 7.3805$ (1) Å	$\theta = 3.0$–27.5°
$c = 12.383$ (2) Å	$\mu = 1.15$ mm^{-1}
$\alpha = 84.17$ (3)°	$T = 293$ K
$\beta = 88.91$ (3)°	Prism, colorless
$\gamma = 65.71$ (2)°	0.20 × 0.20 × 0.20 mm
$V = 505.51$ (8) Å^3	

Data collection

Rigaku Mercury CCD diffractometer	2286 independent reflections
Radiation source: fine-focus sealed tube	2104 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
Detector resolution: 13.6612 pixels mm^{-1}	$\theta_{max} = 27.4$°
$T = 293$ K	$\theta_{min} = 3.0$°
CCD_Profile_fitting scans	$h = -7 \rightarrow 7$
Absorption correction: Multi-scan (CrystalClear; Rigaku, 2000)	$k = -7 \rightarrow 9$
$T_{min} = 0.773$, $T_{max} = 1.000$	$l = -15 \rightarrow 15$
3817 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.078$	$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.1909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
2286 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
171 parameters	$\Delta\rho_{min} = -0.70$ e Å^{-3}
5 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
Cd1	0.5000	0.0000	0.5000	0.03880 (13)
O1	−0.1058 (5)	−0.1234 (4)	0.1759 (2)	0.0614 (8)
N1	0.3050 (5)	0.1080 (4)	0.32445 (19)	0.0328 (6)
C1	−0.1196 (6)	0.0465 (5)	0.1283 (3)	0.0401 (7)
H1	−0.217 (7)	−0.160 (7)	0.152 (4)	0.098 (17)*
O2	−0.2500 (5)	0.1386 (4)	0.0504 (2)	0.0580 (7)
N2	0.5549 (5)	0.4825 (4)	0.2873 (2)	0.0404 (6)
C2	0.0443 (6)	0.1228 (4)	0.1777 (2)	0.0338 (7)
O3	0.1539 (5)	0.2229 (4)	0.5726 (2)	0.0512 (6)
N3	0.7080 (5)	0.4857 (4)	0.3617 (2)	0.0461 (7)
C3	0.0743 (6)	0.2851 (5)	0.1257 (3)	0.0425 (8)
H3	−0.0060	0.3468	0.0581	0.051*
H3A	0.195 (6)	0.321 (4)	0.587 (2)	0.031 (8)*
H3B	0.015 (5)	0.273 (5)	0.540 (3)	0.052 (11)*
O4	0.6141 (5)	0.7305 (4)	0.1064 (2)	0.0579 (7)
N4	0.7355 (5)	0.3463 (4)	0.4431 (2)	0.0450 (7)
C4	0.2210 (6)	0.3572 (5)	0.1722 (3)	0.0424 (8)
H4	0.2453	0.4676	0.1367	0.051*
H4A	0.492 (6)	0.781 (6)	0.063 (3)	0.062 (12)*
H4B	0.571 (7)	0.666 (6)	0.157 (3)	0.068 (13)*
N5	0.6029 (5)	0.2491 (4)	0.4230 (2)	0.0354 (6)
C5	0.1627 (6)	0.0381 (5)	0.2777 (2)	0.0366 (7)
H5	0.1418	−0.0732	0.3141	0.044*
C6	0.3338 (5)	0.2658 (4)	0.2722 (2)	0.0326 (6)
C7	0.4943 (6)	0.3343 (4)	0.3263 (2)	0.0340 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0443 (2)	0.0424 (2)	0.03052 (19)	−0.02092 (16)	−0.00857 (14)	0.00836 (13)
O1	0.075 (2)	0.0752 (19)	0.0503 (16)	−0.0506 (17)	−0.0210 (14)	0.0103 (13)
N1	0.0369 (15)	0.0352 (13)	0.0274 (13)	−0.0169 (12)	0.0003 (10)	0.0014 (10)

C1	0.0366 (19)	0.054 (2)	0.0307 (16)	−0.0186 (16)	0.0024 (13)	−0.0080 (14)
O2	0.0524 (17)	0.0714 (17)	0.0467 (15)	−0.0237 (14)	−0.0215 (12)	0.0051 (12)
N2	0.0446 (17)	0.0393 (15)	0.0422 (16)	−0.0237 (13)	−0.0003 (12)	0.0031 (11)
C2	0.0324 (17)	0.0403 (16)	0.0252 (15)	−0.0118 (13)	−0.0002 (12)	−0.0016 (12)
O3	0.0495 (18)	0.0497 (15)	0.0547 (16)	−0.0199 (14)	−0.0028 (13)	−0.0076 (12)
N3	0.0491 (19)	0.0470 (16)	0.0503 (18)	−0.0276 (15)	0.0015 (14)	−0.0062 (13)
C3	0.042 (2)	0.0482 (19)	0.0329 (17)	−0.0168 (16)	−0.0089 (14)	0.0099 (14)
O4	0.0610 (19)	0.0764 (19)	0.0474 (16)	−0.0446 (16)	−0.0197 (14)	0.0201 (14)
N4	0.0446 (18)	0.0470 (16)	0.0478 (17)	−0.0231 (14)	−0.0064 (13)	−0.0032 (13)
C4	0.046 (2)	0.0419 (18)	0.0370 (18)	−0.0190 (16)	−0.0024 (14)	0.0105 (13)
N5	0.0352 (15)	0.0353 (13)	0.0381 (14)	−0.0178 (12)	−0.0034 (11)	0.0014 (11)
C5	0.0393 (19)	0.0405 (17)	0.0315 (16)	−0.0194 (15)	−0.0029 (13)	0.0032 (12)
C6	0.0321 (17)	0.0321 (15)	0.0312 (16)	−0.0114 (13)	0.0011 (12)	0.0000 (12)
C7	0.0345 (17)	0.0319 (15)	0.0336 (16)	−0.0128 (13)	0.0034 (12)	0.0017 (12)

Geometric parameters (Å, °)

Cd1—N5	2.293 (2)	C2—C5	1.398 (4)
Cd1—N5^{i}	2.293 (2)	O3—H3A	0.90 (2)
Cd1—O3^{i}	2.312 (3)	O3—H3B	0.86 (2)
Cd1—O3	2.312 (3)	N3—N4	1.325 (4)
Cd1—N1	2.396 (2)	C3—C4	1.374 (5)
Cd1—N1^{i}	2.396 (2)	C3—H3	0.9500
O1—C1	1.301 (4)	O4—H4A	0.85 (3)
O1—H1	0.89 (3)	O4—H4B	0.85 (3)
N1—C5	1.342 (4)	N4—N5	1.324 (4)
N1—C6	1.348 (4)	C4—C6	1.394 (4)
C1—O2	1.215 (4)	C4—H4	0.9500
C1—C2	1.499 (4)	N5—C7	1.343 (4)
N2—N3	1.332 (4)	C5—H5	0.9500
N2—C7	1.336 (4)	C6—C7	1.472 (4)
C2—C3	1.379 (4)		
N5—Cd1—N5^{i}	180.0	C5—C2—C1	121.8 (3)
N5—Cd1—O3^{i}	87.03 (10)	Cd1—O3—H3A	103 (2)
N5^{i}—Cd1—O3^{i}	92.97 (10)	Cd1—O3—H3B	124 (3)
N5—Cd1—O3	92.97 (10)	H3A—O3—H3B	109 (3)
N5^{i}—Cd1—O3	87.03 (10)	N4—N3—N2	109.7 (3)
O3^{i}—Cd1—O3	180.0	C4—C3—C2	119.7 (3)
N5—Cd1—N1	72.97 (9)	C4—C3—H3	120.2
N5^{i}—Cd1—N1	107.03 (9)	C2—C3—H3	120.2
O3^{i}—Cd1—N1	91.55 (9)	H4A—O4—H4B	103 (4)
O3—Cd1—N1	88.45 (9)	N5—N4—N3	109.3 (3)
N5—Cd1—N1^{i}	107.03 (9)	C3—C4—C6	119.0 (3)
N5^{i}—Cd1—N1^{i}	72.97 (9)	C3—C4—H4	120.5
O3^{i}—Cd1—N1^{i}	88.45 (9)	C6—C4—H4	120.5
O3—Cd1—N1^{i}	91.55 (9)	N4—N5—C7	105.1 (2)

N1—Cd1—N1^{i}	180.00 (5)	N4—N5—Cd1	140.8 (2)
C1—O1—H1	113 (3)	C7—N5—Cd1	114.08 (19)
C5—N1—C6	118.3 (2)	N1—C5—C2	122.5 (3)
C5—N1—Cd1	127.62 (19)	N1—C5—H5	118.7
C6—N1—Cd1	113.95 (18)	C2—C5—H5	118.7
O2—C1—O1	124.7 (3)	N1—C6—C4	122.1 (3)
O2—C1—C2	121.4 (3)	N1—C6—C7	116.0 (2)
O1—C1—C2	113.9 (3)	C4—C6—C7	121.9 (3)
N3—N2—C7	104.7 (3)	N2—C7—N5	111.3 (3)
C3—C2—C5	118.4 (3)	N2—C7—C6	126.0 (3)
C3—C2—C1	119.8 (3)	N5—C7—C6	122.7 (3)

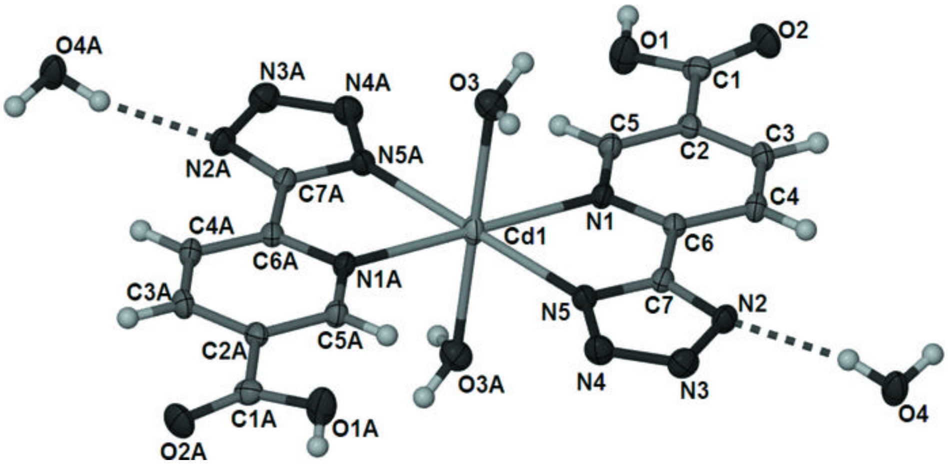
Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4B···N2	0.85 (3)	2.02 (3)	2.860 (4)	166 (4)
O4—H4A···O2^{ii}	0.85 (3)	1.92 (3)	2.767 (4)	170 (4)
O1—H1···O4^{iii}	0.89 (3)	1.68 (3)	2.566 (4)	170 (5)
O3—H3B···N4^{iv}	0.86 (2)	1.96 (3)	2.804 (4)	168 (4)
O3—H3A···N3^{v}	0.90 (2)	1.92 (2)	2.806 (4)	172 (3)

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

Fig. 1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N4A
O3
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O2
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C5
C2
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N1
C4
Cd1
C6A
C6
N1A
C3A
N5
C7
N2
C2A
C5A
O3A
N4
N3
O4
C1A
O1A
O2A

Fig. 2