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

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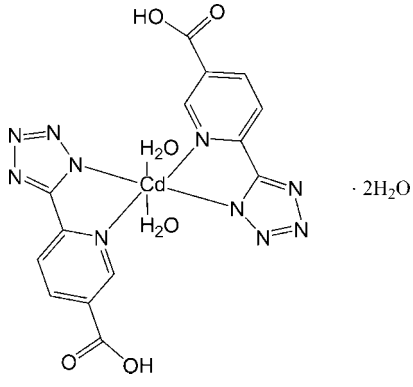
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.078; data-to-parameter ratio = 13.4.

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).

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Xiong, R. G., Xue, X., Zhao, H., You, X. Z., Abrahams, B. F. & Xue, Z. (2002). *Angew. Chem. Int. Ed.* **41**, 3800–3803.

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

Haoyong Yin, Ling Wang and Qiulin Nie

S1. 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).

S2. 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.

S3. 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.

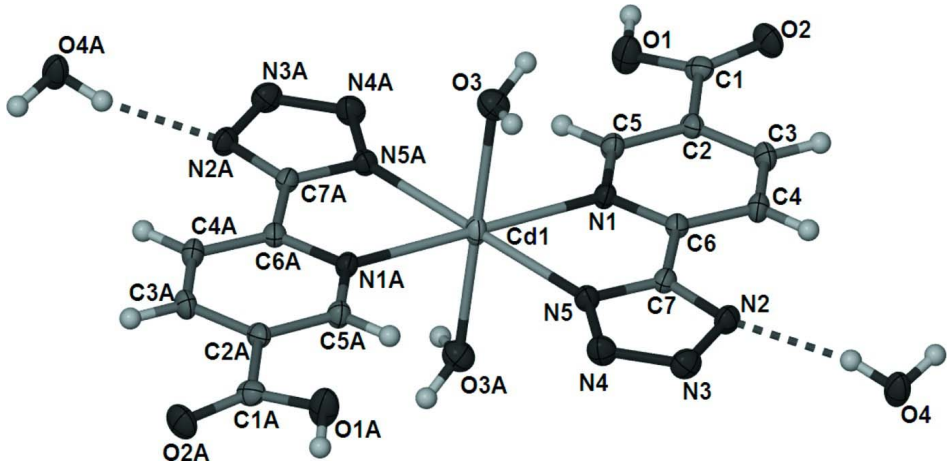

Figure 1

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

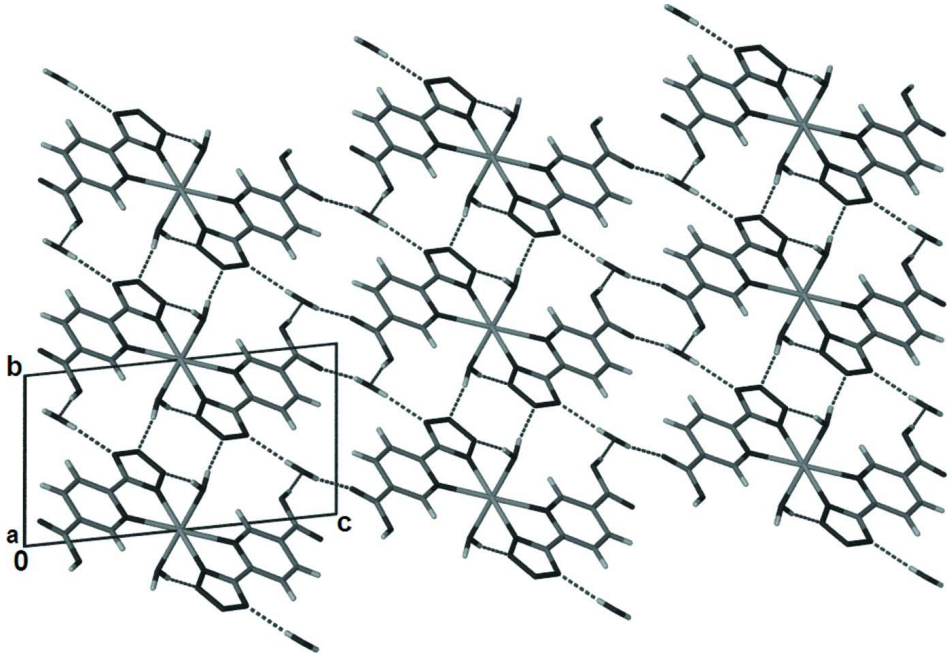

Figure 2

The packing structure viewed along *a* axis.

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

Crystal data

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

Data collection

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

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.078$
$S = 1.10$
2286 reflections
171 parameters
5 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
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.1909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.70$ 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 (Å²)

	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 (Å²)

	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, º)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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