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catena-Poly[[[3-(2-pyridyl)-1H-pyrazole]cadmium(II)]- μ -oxalato] dihydrate]

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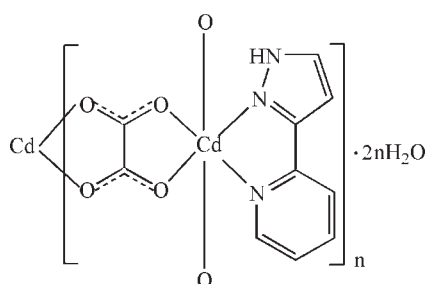
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.018; wR factor = 0.050; data-to-parameter ratio = 12.2.

In the title compound, $\{[\text{Cd}(\text{C}_2\text{O}_4)(\text{C}_8\text{H}_7\text{N}_3)] \cdot 2\text{H}_2\text{O}\}_m$, the Cd^{II} ion is chelated by two O, O' -bidentate oxalate ions and an N, N' -bidentate 3-(2-pyridyl)-1H-pyrazole molecule, thereby generating a distorted $\text{cis-CdN}_2\text{O}_4$ octahedral geometry. Adjacent pairs of Cd ions are bridged by oxalate ions, resulting in wave-like polymeric chains propagating in $[100]$. The packing is consolidated by $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For coordination compounds with pyridyl-pyrazolide ligands, see: Ward *et al.* (1998, 2001).



Experimental

Crystal data

$[\text{Cd}(\text{C}_2\text{O}_4)(\text{C}_8\text{H}_7\text{N}_3)] \cdot 2\text{H}_2\text{O}$
 $M_r = 381.63$
 Triclinic, $P\bar{1}$
 $a = 7.920$ (2) Å
 $b = 9.663$ (2) Å
 $c = 9.675$ (2) Å
 $\alpha = 92.940$ (4)°
 $\beta = 108.555$ (3)°

$\gamma = 106.164$ (4)°
 $V = 666.2$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.67$ mm⁻¹
 $T = 293$ K
 $0.12 \times 0.10 \times 0.08$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
 $T_{\text{min}} = 0.825$, $T_{\text{max}} = 0.878$

3416 measured reflections
 2346 independent reflections
 2247 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.008$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.050$
 $S = 1.00$
 2346 reflections
 193 parameters
 6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.38$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|---------------------|-------------|----------------------|-------------|
| Cd1—O1 | 2.2802 (16) | Cd1—O4 ⁱⁱ | 2.3010 (16) |
| Cd1—O2 ⁱ | 2.2850 (17) | Cd1—N1 | 2.365 (2) |
| Cd1—O3 | 2.3286 (17) | Cd1—N2 | 2.292 (2) |

Symmetry codes: (i) $-x + 1, -y + 2, -z + 2$; (ii) $-x + 1, -y + 1, -z + 2$.

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N3}-\text{H3A}\cdots\text{O5}^{\text{iii}}$ | 0.86 | 1.85 | 2.696 (3) | 169 |
| $\text{O5}-\text{H2W}\cdots\text{O2}^{\text{iv}}$ | 0.82 (2) | 2.20 (2) | 2.861 (3) | 138 (3) |
| $\text{O6}-\text{H3W}\cdots\text{O4}^{\text{v}}$ | 0.82 (4) | 2.34 (3) | 2.878 (3) | 124 (3) |
| $\text{O6}-\text{H4W}\cdots\text{O3}^{\text{vi}}$ | 0.82 (4) | 2.01 (4) | 2.832 (3) | 171 (4) |

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5161).

References

- Bruker (2003). *SMART*, *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Ward, M. D., Fleming, J. S., Psillakis, E., Jeffery, J. C. & McCleverty, J. A. (1998). *Acta Cryst.* **C54**, 609–612.
 Ward, M. D., McCleverty, J. A. & Jeffery, J. C. (2001). *Coord. Chem. Rev.* **222**, 251–272.

supplementary materials

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catena-Poly[[[3-(2-pyridyl)-1H-pyrazole]cadmium(II)]- μ -oxalato] dihydrate]

L. Zhu and Z. An

Comment

Tridentate ligand 3-(2-pyridyl)pyrazole and its derivatives have been used widely in the construction of supramolecular architectures by way of metal-organic coordination (Ward *et al.* 1998; 2001).

As a continuation of these studies, we now report the crystal structure of the title complex.

As shown in figure 1, the Cd^{II} ions are hexacoordinated, chelated by two oxalate and one 3-(2-pyridyl)pyrazole ligand (Table 1). While each oxalate ligand acts as one bridge to chelate two Cd ions, forming one wave-like line with Cd...Cd distance being 5.950 Å, shown in Figure 2. The structure is consolidated by N—H...O and O—H...O hydrogen bonds (Table 2, Figure 3).

Experimental

A mixture of Cd(CH₃COO)₂·2H₂O (1 mmol, 0.027 g), oxalic acid (1 mmol, 0.09 g), sodium hydroxide (0.04 g, 1 mmol) and 3-(2-pyridyl)pyrazole (1 mmol, 0.15 g) and water (12 ml) was stirred for 30 min in air. The mixture was then transferred to a 25 ml Teflon-lined hydrothermal bomb. The bomb was kept at 433 K for 72 h under autogenous pressure. Upon cooling, colorless prisms of (I) were obtained from the reaction mixture.

Refinement

All hydrogen atoms bound to carbon were refined using a riding model with C—H = 0.93 and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Two solvent water molecules are refined by using the 'DFIX' command with the hydrogen atoms were separated with 1.38 Å, and the lengths of bond H—O were constrained with 0.82 Å with error 0.02Å and $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{O})$.

Figures

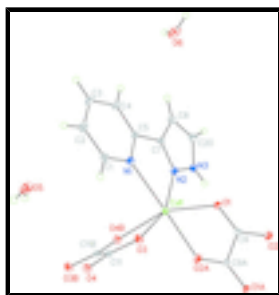


Fig. 1. , A view of the title compound with the unique atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms labeled with A are at the symmetry position (-x,-y + 2,-z + 2).

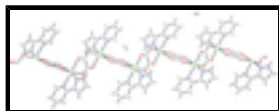


Fig. 2. , A view of the chain structure of (I).

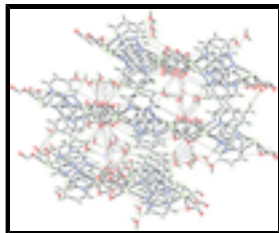


Fig. 3. , A view of the packing structure of (I).

catena-Poly[[[3-(2-pyridyl)-1H-pyrazole]cadmium(II)]-μ-oxalato] dihydrate]

Crystal data

[Cd(C₂O₄)(C₈H₇N₃)]·2H₂O

M_r = 381.63

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

a = 7.920 (2) Å

b = 9.663 (2) Å

c = 9.675 (2) Å

α = 92.940 (4)°

β = 108.555 (3)°

γ = 106.164 (4)°

V = 666.2 (3) Å³

Z = 2

*F*₀₀₀ = 376

D_x = 1.902 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3167 reflections

θ = 2.9–28.3°

μ = 1.67 mm⁻¹

T = 293 K

Block, colorless

0.12 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 293 K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2003)

*T*_{min} = 0.825, *T*_{max} = 0.878

3416 measured reflections

2346 independent reflections

2247 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.008

θ _{max} = 25.0°

θ _{min} = 2.3°

h = -8→9

k = -11→9

l = -11→10

Refinement

Refinement on *F*²

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.018$

$wR(F^2) = 0.050$

S = 1.00

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

$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 0.429P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.001

2346 reflections $\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$
 193 parameters $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$
 6 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|---------------|---------------|----------------------------------|
| Cd1 | 0.49223 (2) | 0.741369 (17) | 0.821503 (17) | 0.03309 (8) |
| C1 | 0.1758 (4) | 0.4289 (3) | 0.6244 (3) | 0.0458 (6) |
| H1 | 0.1605 | 0.4120 | 0.7141 | 0.055* |
| C2 | 0.0730 (4) | 0.3235 (3) | 0.5038 (4) | 0.0543 (7) |
| H2 | -0.0085 | 0.2366 | 0.5120 | 0.065* |
| C3 | 0.0924 (4) | 0.3483 (3) | 0.3708 (3) | 0.0531 (7) |
| H3 | 0.0232 | 0.2791 | 0.2869 | 0.064* |
| C4 | 0.2164 (4) | 0.4777 (3) | 0.3633 (3) | 0.0462 (6) |
| H4 | 0.2317 | 0.4967 | 0.2741 | 0.055* |
| C5 | 0.3179 (3) | 0.5791 (3) | 0.4901 (3) | 0.0339 (5) |
| C7 | 0.4569 (3) | 0.7167 (3) | 0.4902 (3) | 0.0342 (5) |
| C8 | 0.4998 (4) | 0.7780 (3) | 0.3728 (3) | 0.0475 (6) |
| H8 | 0.4419 | 0.7414 | 0.2727 | 0.057* |
| C9 | 0.4002 (3) | 0.9947 (2) | 0.9452 (2) | 0.0311 (5) |
| C11 | 0.4082 (3) | 0.5038 (2) | 1.0129 (2) | 0.0295 (5) |
| C20 | 0.6454 (4) | 0.9036 (3) | 0.4371 (3) | 0.0496 (7) |
| H20 | 0.7065 | 0.9693 | 0.3884 | 0.060* |
| N1 | 0.2973 (3) | 0.5554 (2) | 0.6200 (2) | 0.0365 (4) |
| N2 | 0.5697 (3) | 0.8005 (2) | 0.6184 (2) | 0.0351 (4) |
| N3 | 0.6837 (3) | 0.9143 (2) | 0.5838 (2) | 0.0421 (5) |
| H3A | 0.7701 | 0.9847 | 0.6474 | 0.050* |
| O1 | 0.3288 (2) | 0.89932 (18) | 0.83387 (18) | 0.0377 (4) |
| O2 | 0.3257 (2) | 1.0843 (2) | 0.9777 (2) | 0.0450 (4) |
| O3 | 0.3446 (2) | 0.60271 (19) | 0.9645 (2) | 0.0409 (4) |
| O4 | 0.3377 (2) | 0.41014 (18) | 1.07962 (18) | 0.0364 (4) |
| O5 | -0.0175 (3) | 0.1137 (3) | 0.7858 (3) | 0.0753 (7) |
| H1W | -0.063 (4) | 0.067 (5) | 0.841 (4) | 0.113* |

supplementary materials

| | | | | |
|-----|-------------|------------|------------|------------|
| H2W | 0.0949 (15) | 0.126 (5) | 0.805 (4) | 0.113* |
| O6 | 0.0648 (3) | 0.6774 (3) | 0.0467 (3) | 0.0863 (9) |
| H3W | -0.016 (4) | 0.703 (5) | -0.013 (4) | 0.130* |
| H4W | 0.143 (5) | 0.660 (5) | 0.015 (4) | 0.130* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|-------------|--------------|--------------|
| Cd1 | 0.04230 (12) | 0.02946 (11) | 0.02728 (11) | 0.01333 (8) | 0.01047 (8) | 0.00063 (7) |
| C1 | 0.0421 (14) | 0.0385 (14) | 0.0520 (16) | 0.0092 (11) | 0.0127 (12) | 0.0075 (12) |
| C2 | 0.0399 (14) | 0.0388 (14) | 0.071 (2) | 0.0056 (11) | 0.0093 (14) | -0.0040 (13) |
| C3 | 0.0412 (15) | 0.0483 (16) | 0.0574 (17) | 0.0102 (12) | 0.0071 (13) | -0.0147 (13) |
| C4 | 0.0400 (14) | 0.0520 (16) | 0.0393 (14) | 0.0122 (12) | 0.0092 (11) | -0.0124 (12) |
| C5 | 0.0319 (12) | 0.0368 (12) | 0.0333 (12) | 0.0144 (10) | 0.0095 (9) | -0.0023 (10) |
| C7 | 0.0338 (12) | 0.0372 (12) | 0.0312 (12) | 0.0138 (10) | 0.0093 (10) | 0.0004 (10) |
| C8 | 0.0505 (16) | 0.0583 (17) | 0.0315 (13) | 0.0162 (13) | 0.0121 (11) | 0.0055 (12) |
| C9 | 0.0329 (12) | 0.0288 (11) | 0.0286 (11) | 0.0096 (9) | 0.0070 (10) | 0.0024 (9) |
| C11 | 0.0301 (11) | 0.0357 (12) | 0.0242 (10) | 0.0137 (9) | 0.0091 (9) | 0.0009 (9) |
| C20 | 0.0571 (17) | 0.0508 (16) | 0.0447 (15) | 0.0159 (13) | 0.0220 (13) | 0.0172 (13) |
| N1 | 0.0355 (10) | 0.0348 (10) | 0.0362 (11) | 0.0109 (8) | 0.0090 (9) | 0.0011 (8) |
| N2 | 0.0384 (11) | 0.0332 (10) | 0.0336 (10) | 0.0108 (8) | 0.0132 (9) | 0.0027 (8) |
| N3 | 0.0458 (12) | 0.0328 (11) | 0.0432 (12) | 0.0079 (9) | 0.0138 (10) | 0.0036 (9) |
| O1 | 0.0402 (9) | 0.0347 (9) | 0.0311 (8) | 0.0152 (7) | 0.0014 (7) | -0.0049 (7) |
| O2 | 0.0393 (9) | 0.0438 (10) | 0.0444 (10) | 0.0208 (8) | 0.0005 (8) | -0.0119 (8) |
| O3 | 0.0448 (10) | 0.0459 (10) | 0.0487 (10) | 0.0268 (8) | 0.0260 (8) | 0.0158 (8) |
| O4 | 0.0361 (9) | 0.0426 (9) | 0.0392 (9) | 0.0170 (7) | 0.0197 (7) | 0.0123 (7) |
| O5 | 0.0519 (13) | 0.0818 (18) | 0.0707 (16) | 0.0203 (13) | -0.0022 (11) | -0.0180 (13) |
| O6 | 0.0464 (13) | 0.103 (2) | 0.0975 (19) | 0.0238 (13) | 0.0171 (13) | -0.0409 (16) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------------------|-------------|-----------------------|-------------|
| Cd1—O1 | 2.2802 (16) | C8—C20 | 1.370 (4) |
| Cd1—O2 ⁱ | 2.2850 (17) | C8—H8 | 0.9300 |
| Cd1—O3 | 2.3286 (17) | C9—O1 | 1.245 (3) |
| Cd1—O4 ⁱⁱ | 2.3010 (16) | C9—O2 | 1.253 (3) |
| Cd1—N1 | 2.365 (2) | C9—C9 ⁱ | 1.571 (4) |
| Cd1—N2 | 2.292 (2) | C11—O3 | 1.245 (3) |
| C1—N1 | 1.341 (3) | C11—O4 | 1.247 (3) |
| C1—C2 | 1.369 (4) | C11—C11 ⁱⁱ | 1.572 (4) |
| C1—H1 | 0.9300 | C20—N3 | 1.345 (4) |
| C2—C3 | 1.369 (5) | C20—H20 | 0.9300 |
| C2—H2 | 0.9300 | N2—N3 | 1.346 (3) |
| C3—C4 | 1.380 (4) | N3—H3A | 0.8600 |
| C3—H3 | 0.9300 | O2—Cd1 ⁱ | 2.2850 (17) |
| C4—C5 | 1.386 (3) | O4—Cd1 ⁱⁱ | 2.3010 (16) |
| C4—H4 | 0.9300 | O5—H1W | 0.82 (4) |
| C5—N1 | 1.341 (3) | O5—H2W | 0.82 (2) |

| | | | |
|---------------------------------------|------------|--------------------------|-------------|
| C5—C7 | 1.471 (3) | O6—H3W | 0.82 (4) |
| C7—N2 | 1.334 (3) | O6—H4W | 0.82 (4) |
| C7—C8 | 1.400 (4) | | |
| O1—Cd1—O2 ⁱ | 73.10 (6) | N2—C7—C8 | 110.4 (2) |
| O1—Cd1—N2 | 99.85 (7) | N2—C7—C5 | 119.4 (2) |
| O2 ⁱ —Cd1—N2 | 110.73 (7) | C8—C7—C5 | 130.2 (2) |
| O1—Cd1—O4 ⁱⁱ | 153.29 (6) | C20—C8—C7 | 105.1 (2) |
| O2 ⁱ —Cd1—O4 ⁱⁱ | 89.10 (6) | C20—C8—H8 | 127.5 |
| N2—Cd1—O4 ⁱⁱ | 105.11 (6) | C7—C8—H8 | 127.5 |
| O1—Cd1—O3 | 88.25 (6) | O1—C9—O2 | 125.3 (2) |
| O2 ⁱ —Cd1—O3 | 90.72 (7) | O1—C9—C9 ⁱ | 118.1 (2) |
| N2—Cd1—O3 | 158.43 (7) | O2—C9—C9 ⁱ | 116.6 (2) |
| O4 ⁱⁱ —Cd1—O3 | 71.86 (6) | O3—C11—O4 | 125.3 (2) |
| O1—Cd1—N1 | 106.71 (6) | O3—C11—C11 ⁱⁱ | 117.3 (2) |
| O2 ⁱ —Cd1—N1 | 177.56 (7) | O4—C11—C11 ⁱⁱ | 117.4 (2) |
| N2—Cd1—N1 | 71.72 (7) | N3—C20—C8 | 107.4 (2) |
| O4 ⁱⁱ —Cd1—N1 | 90.22 (7) | N3—C20—H20 | 126.3 |
| O3—Cd1—N1 | 86.84 (7) | C8—C20—H20 | 126.3 |
| N1—C1—C2 | 123.5 (3) | C1—N1—C5 | 117.8 (2) |
| N1—C1—H1 | 118.3 | C1—N1—Cd1 | 126.77 (18) |
| C2—C1—H1 | 118.3 | C5—N1—Cd1 | 115.34 (15) |
| C3—C2—C1 | 118.7 (3) | C7—N2—N3 | 105.76 (19) |
| C3—C2—H2 | 120.7 | C7—N2—Cd1 | 116.09 (15) |
| C1—C2—H2 | 120.7 | N3—N2—Cd1 | 137.04 (15) |
| C2—C3—C4 | 119.0 (3) | C20—N3—N2 | 111.3 (2) |
| C2—C3—H3 | 120.5 | C20—N3—H3A | 124.3 |
| C4—C3—H3 | 120.5 | N2—N3—H3A | 124.3 |
| C3—C4—C5 | 119.4 (3) | C9—O1—Cd1 | 115.63 (14) |
| C3—C4—H4 | 120.3 | C9—O2—Cd1 ⁱ | 115.99 (14) |
| C5—C4—H4 | 120.3 | C11—O3—Cd1 | 115.95 (14) |
| N1—C5—C4 | 121.6 (2) | C11—O4—Cd1 ⁱⁱ | 116.68 (14) |
| N1—C5—C7 | 116.4 (2) | H1W—O5—H2W | 115 (4) |
| C4—C5—C7 | 121.9 (2) | H3W—O6—H4W | 115 (4) |

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+1, -y+1, -z+2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N3—H3A \cdots O5 ⁱⁱⁱ | 0.86 | 1.85 | 2.696 (3) | 169 |
| O5—H2W \cdots O2 ^{iv} | 0.82 (2) | 2.20 (2) | 2.861 (3) | 138 (3) |
| O6—H3W \cdots O4 ^v | 0.82 (4) | 2.34 (3) | 2.878 (3) | 124 (3) |
| O6—H4W \cdots O3 ^{vi} | 0.82 (4) | 2.01 (4) | 2.832 (3) | 171 (4) |

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

Fig. 1

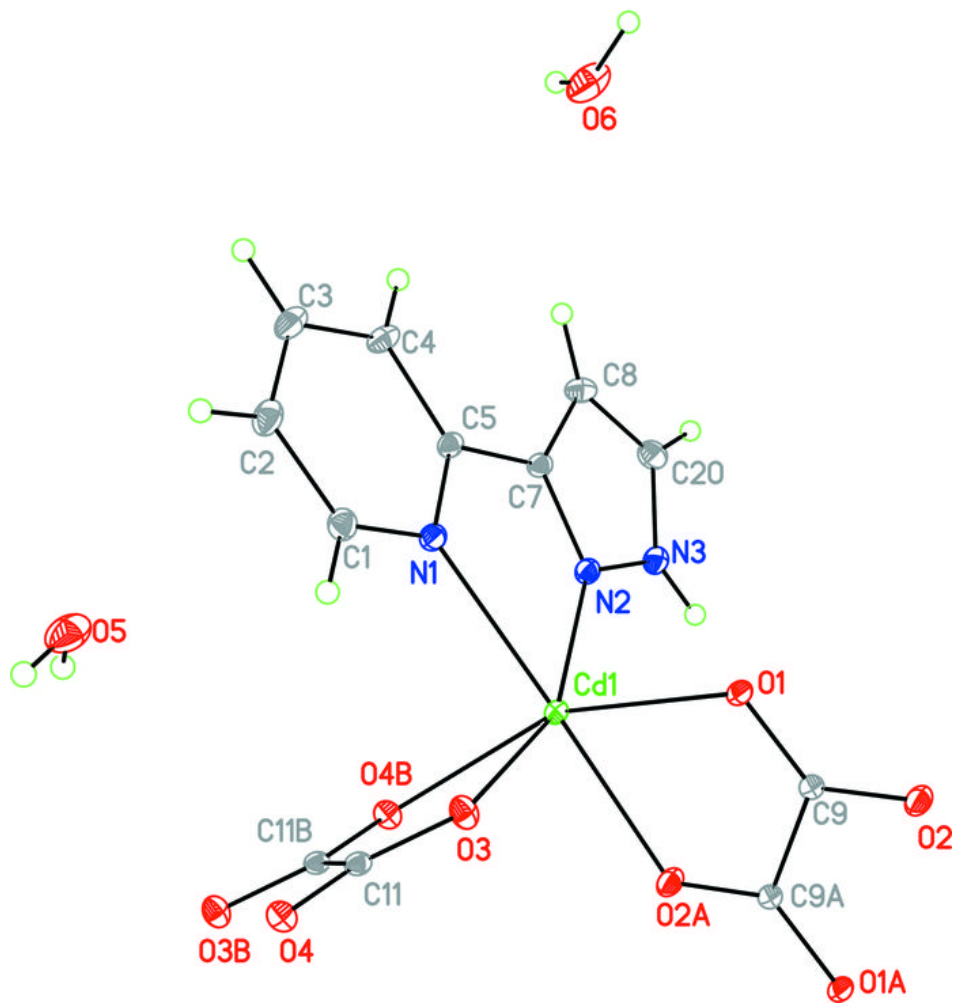


Fig. 2

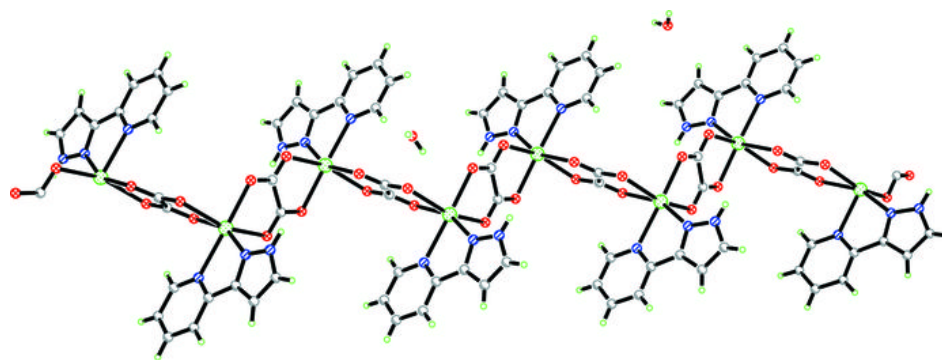


Fig. 3

