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# *catena*-Poly[[[diaquacadmium(II)]-$\mu$-2,2′-bipyridine-6,6′-dicarboxylato] dihydrate]

**James C. Knight,* Angelo J. Amoroso, Peter G. Edwards and Li-Ling Ooi**

Main Building, School of Chemistry, Cardiff University, Park Place, Cardiff CF10 3AT, Wales

Correspondence e-mail: knightjc@cardiff.ac.uk

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**Key indicators**

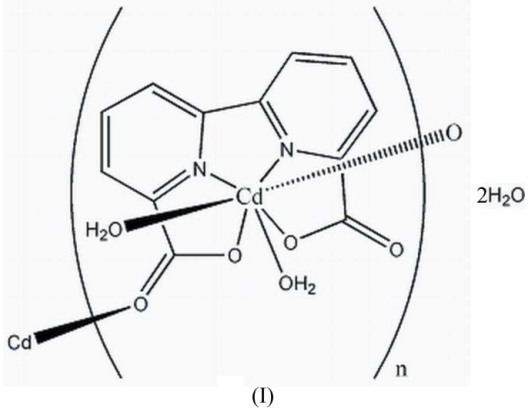
Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.008 Å
$R$ factor = 0.050
$wR$ factor = 0.131
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title hydrated 2,2′-bipyridine-6,6′-dicarboxylate–cadmium(II) complex, $\{[Cd(C_{12}H_2N_2O_4)(H_2O)_2]\cdot 2H_2O\}_n$, crystallizes with eight oxygen-bridged monomer units in the unit cell. The divalent cadmium ion is seven-coordinate, and the coordination polyhedron can best be described as slightly distorted pentagonal–bipyramidal.

## Comment

The title compound, (I), has been synthesized as part of a study investigating complex stability. Specifically, the research is part of initial investigations towards generating $Gd^{III}$-based contrast agents for applications in magnetic resonance imaging (MRI), where overall complex stability is crucial in preventing dissociation *in vivo*, due to the well documented interference of $Gd^{3+}$ in biological processes (Cacheris, 1990).




(I)

The research aims to elucidate structural features that contribute significantly to overall stability. A comprehensive series of transition metals has been considered, providing an insight into competition reactions which may compromise the effectiveness of the ability of the ligands to coordinate to the $Gd^{3+}$ ion and neutralize its potential toxicity (Caravan, 1999).

In this complex, the Cd atom is located at the centre of a distorted pentagonal bipyramid of seven coordinating atoms (five O atoms and two N atoms; Fig. 1). One of these donor atoms ($O4^{i}$) originates from another symmetry-related complex ($x-\frac{1}{2}, \frac{1}{2}-y, z$). The bond lengths are comparable to those of similar polymeric cadmium-based complexes (Deloume & Loiseleur, 1974). The bridging behaviour results in the formation of a polymer, which extends in a zigzag fashion (see Fig. 2).

The donor atoms $O4^{i}$ and O5 are located in the axial positions, with N1, N2, O1, O3 and O6 in the equatorial plane. The deviation of the equatorial angles N1—Cd1—N2, N2—

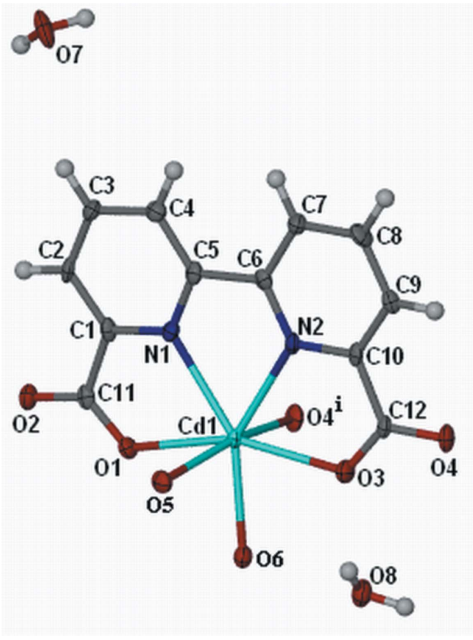



**Figure 1**
Perspective view of the asymmetric unit, expanded to complete the Cd coordination, showing the atom numbering. Displacement ellipsoids are shown at the 50% probability level. H atoms are represented by circles of arbitrary size. [Symmetry code: (i) $x-\frac{1}{2}, \frac{1}{2}-y, z$.]

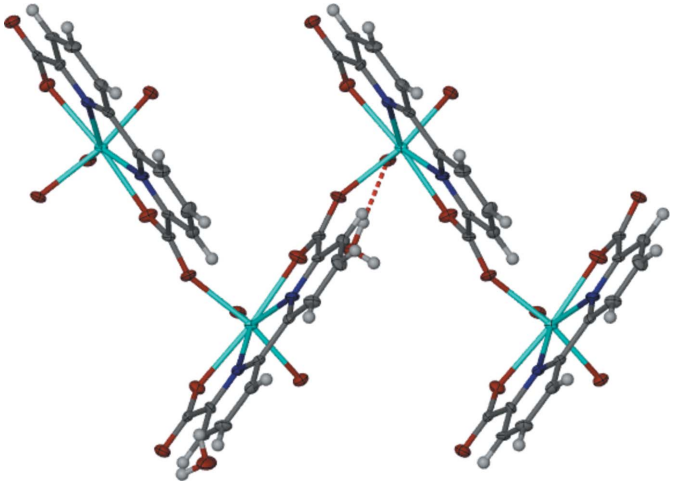


**Figure 2**
Fragment of the title structure, showing an oxygen-bridged chain extended in a zigzag fashion along the *c* axis. The dashed line indicates a hydrogen bond.

Cd1—O3, O3—Cd1—O6, O6—Cd1—O1, O1—Cd1—N1 (Table 1) from the theoretical average angle of 72° can be explained by the narrow bite angle resulting from the coordination of the rigid tetradentate ligand. While the bridging carboxylate group lies almost perpendicular to the tetradentate ligand at 88.75 (13)°, the axial water molecule appears to lean away from the more sterically demanding ligand, tending towards the equatorial water donor at a more acute angle of 82.08 (12)°.

The oxygen-bridged polymer is formed *via* coordination of one of the carbonyl O atoms (O4$^{i}$) to the cadmium in an axial orientation at an average angle of 90.10° to the pentagonal plane. The molecular packing is layered, with intermolecular $\pi$-stacking between bipyridine rings (N1/C1–C5 and N2/C6–C10) at a centroid–centroid distance of 3.267 Å. The equatorially coordinating water molecule (O6) is hydrogen bonded to a solvent water molecule (O8), which in turn is bonded to a second solvent water molecule (O7) which is hydrogen bonded to the axially coordinating water (O5) (see Fig. 3). All significant hydrogen bonds are listed in Table 2.

## Experimental

To an aqueous solution (5 ml water) of 2,2-bipyridine-6,6-dicarboxylate disodium salt (0.05 mol) was added an aqueous solution (5 ml water) of cadmium(II) perchlorate (0.05 mol) and the solution was stirred continuously at room temperature for 5 h. A white precipitate was collected *via* filtration and redissolved in the minimum amount of hot deionized water. Slow evaporation of the solution yielded well defined crystals suitable for X-ray diffraction. $^1$H NMR ($D_2O$): 8.48 (*d*, 2H, *J* = 9.63 Hz, py-H1), 8.16 (*t*, 2H, *J* = 9.75 Hz, py-H2), 8.08 (*d*, 2H, *J* = 7.60 Hz, py-H3).

*Crystal data*

$[Cd(C_{12}H_2N_2O_4)(H_2O)_2]\cdot 2H_2O$
$M_r$ = 422.63
Orthorhombic, *Pcab*
*a* = 8.665 (5) Å
*b* = 17.392 (5) Å
*c* = 18.812 (5) Å
*V* = 2835 (2) Å$^3$
*Z* = 8
$D_x$ = 1.999 Mg m$^{-3}$
Mo *K*$\alpha$ radiation
$\mu$ = 1.59 mm$^{-1}$
*T* = 150 (2) K
Block, colourless
0.52 × 0.4 × 0.22 mm

*Data collection*

Bruker–Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min}$ = 0.492, $T_{max}$ = 0.722
13837 measured reflections
3234 independent reflections
2349 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.092
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.131
*S* = 1.04
3234 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 1.3489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.53 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| N1—Cd1 | 2.388 (4) | O4—Cd1 | 2.332 (4) |
| N2—Cd1 | 2.418 (4) | O5—Cd1 | 2.287 (3) |
| O1—Cd1 | 2.413 (3) | O6—Cd1 | 2.336 (4) |
| O3—Cd1 | 2.423 (4) | | |
| O5—Cd1—O4 | 164.37 (12) | O4—Cd1—N2 | 93.18 (13) |
| O5—Cd1—O6 | 82.08 (12) | O6—Cd1—N2 | 144.24 (13) |
| O4—Cd1—O6 | 85.52 (13) | N1—Cd1—N2 | 66.47 (14) |
| O5—Cd1—N1 | 91.00 (13) | O1—Cd1—N2 | 133.86 (12) |
| O4—Cd1—N1 | 94.87 (13) | O5—Cd1—O3 | 98.34 (13) |
| O6—Cd1—N1 | 149.28 (13) | O4—Cd1—O3 | 88.24 (13) |
| O5—Cd1—O1 | 80.18 (12) | O6—Cd1—O3 | 77.80 (12) |
| O4—Cd1—O1 | 88.70 (13) | N1—Cd1—O3 | 132.91 (13) |
| O6—Cd1—O1 | 81.88 (12) | O1—Cd1—O3 | 159.62 (12) |
| N1—Cd1—O1 | 67.44 (13) | N2—Cd1—O3 | 66.44 (12) |
| O5—Cd1—N2 | 102.44 (13) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O7—H7$A$···O4$^{i}$ | 0.85 | 2.26 | 3.055 (5) | 156 |
| O7—H7$B$···O5$^{ii}$ | 0.87 | 1.88 | 2.729 (5) | 167 |
| O8—H8$A$···O6$^{iii}$ | 0.88 | 2.02 | 2.814 (5) | 150 |
| O8—H8$B$···O7$^{iv}$ | 0.86 | 2.00 | 2.816 (6) | 158 |

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

The carbon bound H atoms were placed in calculated positions using a riding model with $U_{iso}$(H) = 1.2$U_{eq}$(C) and C—H = 0.95. The solvent water H atoms were located and refined with $U_{iso}$(H) = 1.2$U_{eq}$(O)]; however, the H atoms on the coordinating water molecules could not be found at geometrically sensible positions and were not included in the refinement.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This project was supported by the MRC (research grant No. G0300261).

## References

Beurskens, P. T., Beurskens, G., de Gelder, R., Garciía-Granda, S., Gould, R. O., Israel, R. & Smits, J. M. M. (1999). *The DIRDIF99 Program System.* Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

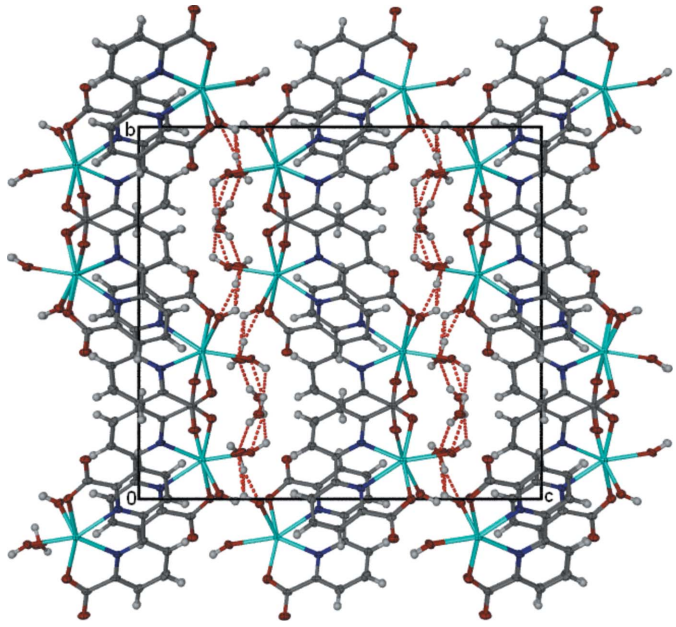



**Figure 3**
The molecular packing projected on the *bc* plane showing hydrogen bonds (dashed lines) holding the layered chains together in the crystal structure.

Cacheris, W. P. (1990). *Magn. Reson. Imaging*, **8**, 467–481.
Caravan, P. (1999). *Chem. Rev.* **99**, 2293–2352.
Deloume, J.-P. & Loiseleur, H. (1974). *Acta Cryst.* B**30**, 607–609.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

# supporting information

*Acta Cryst.* (2006). E**62**, m3306–m3308 [https://doi.org/10.1107/S1600536806046423]

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**James C. Knight, Angelo J. Amoroso, Peter G. Edwards and Li-Ling Ooi**

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*Crystal data*

$[Cd(C_{12}H_2N_2O_4)(H_2O)_2]\cdot 2H_2O$
$M_r = 422.63$
Orthorhombic, *Pcab*
Hall symbol: -P 2bc 2ac
$a = 8.665$ (5) Å
$b = 17.392$ (5) Å
$c = 18.812$ (5) Å
$V = 2835$ (2) Å$^3$
$Z = 8$
$F(000) = 1664.0$
$D_x = 1.999$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 24536 reflections
$\theta = 2.9$–$27.5°$
$\mu = 1.59$ mm$^{-1}$
$T = 150$ K
Block, colourless
0.52 × 0.4 × 0.22 mm

*Data collection*

Bruker–Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.492$, $T_{max} = 0.722$
13837 measured reflections
3234 independent reflections
2349 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 22$
$l = -24 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.131$
$S = 1.04$
3234 reflections
208 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 1.3489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.49$ e Å$^{-3}$
$\Delta\rho_{min} = -0.53$ e Å$^{-3}$

*Special details*

**Experimental**. 1H NMR (D2O): 8.48 (d, 2H, J = 9.63?Hz, py-H1), 8.16 (t, 2H, J = 9.75?Hz, py-H2), 8.08 (d, 2H, J = 7.60?Hz, py-H3). IR (KBr) cm-1: 1617.02 (–C=O), 1591.95, 1574.59, 1421.28, 1384.64, 1267.97, 1144.55, 1111.76, 1087.66, 1018.23, 909.272, 770.423.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.8416 (5) | −0.0338 (3) | 0.0556 (3) | 0.0178 (11) |
| C2 | 0.8675 (6) | −0.0807 (3) | −0.0025 (3) | 0.0199 (11) |
| H2 | 0.9326 | −0.1244 | 0.0017 | 0.024* |
| C3 | 0.7986 (6) | −0.0636 (3) | −0.0662 (3) | 0.0224 (12) |
| H3 | 0.8159 | −0.0951 | −0.1067 | 0.027* |
| C4 | 0.7035 (5) | 0.0003 (3) | −0.0706 (3) | 0.0200 (11) |
| H4 | 0.6551 | 0.0135 | −0.1143 | 0.024* |
| C5 | 0.6802 (5) | 0.0447 (3) | −0.0106 (3) | 0.0179 (11) |
| C6 | 0.5794 (5) | 0.1142 (3) | −0.0097 (3) | 0.0168 (11) |
| C7 | 0.5030 (6) | 0.1419 (3) | −0.0701 (3) | 0.0221 (11) |
| H5 | 0.5132 | 0.1164 | −0.1145 | 0.027* |
| C8 | 0.4132 (6) | 0.2067 (3) | −0.0641 (3) | 0.0251 (12) |
| H8 | 0.3603 | 0.2264 | −0.1043 | 0.030* |
| C9 | 0.4004 (6) | 0.2431 (3) | 0.0014 (3) | 0.0201 (11) |
| H7 | 0.3364 | 0.2870 | 0.0068 | 0.024* |
| C10 | 0.4827 (5) | 0.2142 (3) | 0.0590 (3) | 0.0174 (11) |
| C11 | 0.9164 (5) | −0.0482 (3) | 0.1272 (3) | 0.0181 (11) |
| C12 | 0.4806 (6) | 0.2505 (3) | 0.1310 (3) | 0.0174 (11) |
| N1 | 0.7503 (5) | 0.0284 (2) | 0.0518 (2) | 0.0166 (9) |
| N2 | 0.5693 (5) | 0.1500 (2) | 0.0531 (2) | 0.0177 (9) |
| O1 | 0.8860 (4) | −0.0007 (2) | 0.17608 (17) | 0.0217 (8) |
| O2 | 1.0017 (4) | −0.10573 (19) | 0.1324 (2) | 0.0240 (8) |
| O3 | 0.5496 (4) | 0.2156 (2) | 0.18009 (18) | 0.0259 (8) |
| O4 | 0.9154 (4) | 0.1849 (2) | 0.13790 (19) | 0.0245 (8) |
| O5 | 0.5412 (4) | 0.01180 (19) | 0.19987 (17) | 0.0212 (8) |
| O6 | 0.7508 (4) | 0.1238 (2) | 0.27733 (19) | 0.0225 (8) |
| O7 | 0.8528 (5) | −0.1242 (2) | −0.2466 (2) | 0.0403 (11) |
| H7A | 0.9337 | −0.1427 | −0.2277 | 0.048* |
| H7B | 0.8762 | −0.0776 | −0.2593 | 0.048* |
| O8 | 0.3706 (4) | 0.2275 (2) | 0.29834 (19) | 0.0292 (9) |
| H8A | 0.3340 | 0.2731 | 0.3089 | 0.035* |
| H8B | 0.2974 | 0.2057 | 0.2748 | 0.035* |
| Cd1 | 0.70612 (4) | 0.103047 (19) | 0.156370 (18) | 0.01591 (15) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.013 (2) | 0.013 (3) | 0.028 (3) | 0.003 (2) | 0.002 (2) | 0.003 (2) |
| C2 | 0.021 (3) | 0.008 (2) | 0.031 (3) | 0.000 (2) | 0.003 (2) | −0.003 (2) |
| C3 | 0.032 (3) | 0.014 (3) | 0.022 (3) | 0.000 (2) | 0.001 (2) | −0.004 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.022 (3) | 0.018 (3) | 0.020 (3) | −0.005 (2) | −0.002 (2) | −0.001 (2) |
| C5 | 0.017 (2) | 0.012 (3) | 0.025 (3) | −0.004 (2) | 0.000 (2) | 0.000 (2) |
| C6 | 0.016 (2) | 0.014 (3) | 0.021 (3) | −0.0046 (19) | 0.002 (2) | 0.004 (2) |
| C7 | 0.030 (3) | 0.017 (3) | 0.020 (3) | 0.001 (2) | −0.002 (2) | −0.001 (2) |
| C8 | 0.025 (3) | 0.027 (3) | 0.024 (3) | 0.006 (2) | −0.005 (2) | 0.008 (2) |
| C9 | 0.019 (2) | 0.016 (3) | 0.025 (3) | 0.001 (2) | −0.001 (2) | 0.005 (2) |
| C10 | 0.017 (2) | 0.008 (2) | 0.026 (3) | −0.002 (2) | 0.002 (2) | 0.000 (2) |
| C11 | 0.013 (2) | 0.014 (3) | 0.028 (3) | −0.004 (2) | 0.000 (2) | 0.003 (2) |
| C12 | 0.019 (2) | 0.009 (2) | 0.024 (3) | −0.001 (2) | 0.002 (2) | −0.001 (2) |
| N1 | 0.017 (2) | 0.013 (2) | 0.020 (2) | −0.0026 (18) | −0.0001 (16) | −0.0021 (18) |
| N2 | 0.021 (2) | 0.009 (2) | 0.023 (2) | −0.0003 (18) | 0.0023 (17) | 0.0006 (17) |
| O1 | 0.0254 (19) | 0.0185 (19) | 0.0211 (17) | 0.0056 (17) | −0.0025 (15) | 0.0029 (15) |
| O2 | 0.026 (2) | 0.016 (2) | 0.030 (2) | 0.0057 (16) | −0.0009 (16) | −0.0006 (16) |
| O3 | 0.035 (2) | 0.019 (2) | 0.0242 (19) | 0.0089 (17) | −0.0019 (16) | −0.0007 (16) |
| O4 | 0.0254 (19) | 0.0131 (19) | 0.035 (2) | −0.0065 (16) | 0.0008 (16) | −0.0008 (16) |
| O5 | 0.0236 (18) | 0.0172 (18) | 0.0228 (18) | −0.0057 (15) | −0.0001 (15) | 0.0017 (15) |
| O6 | 0.0289 (19) | 0.0156 (18) | 0.023 (2) | 0.0027 (16) | −0.0033 (15) | −0.0012 (16) |
| O7 | 0.040 (2) | 0.019 (2) | 0.061 (3) | −0.002 (2) | −0.016 (2) | 0.017 (2) |
| O8 | 0.035 (2) | 0.018 (2) | 0.035 (2) | 0.0018 (18) | 0.0004 (18) | −0.0021 (17) |
| Cd1 | 0.0193 (2) | 0.0107 (2) | 0.0177 (2) | −0.00014 (14) | −0.00005 (14) | −0.00005 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.342 (6) | C10—N2 | 1.350 (6) |
| C1—C2 | 1.381 (7) | C10—C12 | 1.494 (7) |
| C1—C11 | 1.515 (7) | C11—O2 | 1.247 (6) |
| C2—C3 | 1.371 (7) | C11—O1 | 1.264 (6) |
| C2—H2 | 0.9500 | C12—O3 | 1.256 (6) |
| C3—C4 | 1.386 (7) | C12—O4$^{i}$ | 1.265 (6) |
| C3—H3 | 0.9500 | N1—Cd1 | 2.388 (4) |
| C4—C5 | 1.383 (7) | N2—Cd1 | 2.418 (4) |
| C4—H4 | 0.9500 | O1—Cd1 | 2.413 (3) |
| C5—N1 | 1.351 (6) | O3—Cd1 | 2.423 (4) |
| C5—C6 | 1.492 (7) | O4—C12$^{ii}$ | 1.265 (6) |
| C6—N2 | 1.340 (6) | O4—Cd1 | 2.332 (4) |
| C6—C7 | 1.400 (7) | O5—Cd1 | 2.287 (3) |
| C7—C8 | 1.373 (7) | O6—Cd1 | 2.336 (4) |
| C7—H5 | 0.9500 | O7—H7A | 0.8490 |
| C8—C9 | 1.389 (7) | O7—H7B | 0.8691 |
| C8—H8 | 0.9500 | O8—H8A | 0.8762 |
| C9—C10 | 1.390 (7) | O8—H8B | 0.8617 |
| C9—H7 | 0.9500 | | |
| | | | |
| N1—C1—C2 | 122.0 (4) | O3—C12—C10 | 117.2 (4) |
| N1—C1—C11 | 115.6 (4) | O4$^{i}$—C12—C10 | 118.3 (5) |
| C2—C1—C11 | 122.4 (4) | C1—N1—C5 | 118.7 (4) |
| C3—C2—C1 | 119.5 (5) | C1—N1—Cd1 | 119.3 (3) |
| C3—C2—H2 | 120.3 | C5—N1—Cd1 | 122.0 (3) |

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| C1—C2—H2 | 120.3 | C6—N2—C10 | 119.6 (4) |
| C2—C3—C4 | 119.0 (5) | C6—N2—Cd1 | 121.3 (3) |
| C2—C3—H3 | 120.5 | C10—N2—Cd1 | 119.1 (3) |
| C4—C3—H3 | 120.5 | C11—O1—Cd1 | 120.8 (3) |
| C3—C4—C5 | 119.0 (5) | C12—O3—Cd1 | 121.4 (3) |
| C3—C4—H4 | 120.5 | C12$^{ii}$—O4—Cd1 | 154.9 (3) |
| C5—C4—H4 | 120.5 | H7A—O7—H7B | 106.1 |
| N1—C5—C4 | 121.8 (5) | H8A—O8—H8B | 104.4 |
| N1—C5—C6 | 115.1 (4) | O5—Cd1—O4 | 164.37 (12) |
| C4—C5—C6 | 123.1 (5) | O5—Cd1—O6 | 82.08 (12) |
| N2—C6—C7 | 121.7 (4) | O4—Cd1—O6 | 85.52 (13) |
| N2—C6—C5 | 115.1 (4) | O5—Cd1—N1 | 91.00 (13) |
| C7—C6—C5 | 123.2 (5) | O4—Cd1—N1 | 94.87 (13) |
| C8—C7—C6 | 118.9 (5) | O6—Cd1—N1 | 149.28 (13) |
| C8—C7—H5 | 120.5 | O5—Cd1—O1 | 80.18 (12) |
| C6—C7—H5 | 120.5 | O4—Cd1—O1 | 88.70 (13) |
| C7—C8—C9 | 119.5 (5) | O6—Cd1—O1 | 81.88 (12) |
| C7—C8—H8 | 120.3 | N1—Cd1—O1 | 67.44 (13) |
| C9—C8—H8 | 120.3 | O5—Cd1—N2 | 102.44 (13) |
| C8—C9—C10 | 119.0 (5) | O4—Cd1—N2 | 93.18 (13) |
| C8—C9—H7 | 120.5 | O6—Cd1—N2 | 144.24 (13) |
| C10—C9—H7 | 120.5 | N1—Cd1—N2 | 66.47 (14) |
| N2—C10—C9 | 121.3 (4) | O1—Cd1—N2 | 133.86 (12) |
| N2—C10—C12 | 115.5 (4) | O5—Cd1—O3 | 98.34 (13) |
| C9—C10—C12 | 123.2 (5) | O4—Cd1—O3 | 88.24 (13) |
| O2—C11—O1 | 126.1 (5) | O6—Cd1—O3 | 77.80 (12) |
| O2—C11—C1 | 117.2 (4) | N1—Cd1—O3 | 132.91 (13) |
| O1—C11—C1 | 116.7 (4) | O1—Cd1—O3 | 159.62 (12) |
| O3—C12—O4$^{i}$ | 124.5 (5) | N2—Cd1—O3 | 66.44 (12) |

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| N1—C1—C2—C3 | 0.1 (8) | C10—C12—O3—Cd1 | 7.5 (6) |
| C11—C1—C2—C3 | −178.7 (4) | C12$^{ii}$—O4—Cd1—O5 | −122.0 (8) |
| C1—C2—C3—C4 | −0.4 (7) | C12$^{ii}$—O4—Cd1—O6 | −84.5 (8) |
| C2—C3—C4—C5 | −0.4 (7) | C12$^{ii}$—O4—Cd1—N1 | 126.3 (8) |
| C3—C4—C5—N1 | 1.5 (7) | C12$^{ii}$—O4—Cd1—O1 | −166.4 (8) |
| C3—C4—C5—C6 | −179.6 (4) | C12$^{ii}$—O4—Cd1—N2 | 59.7 (8) |
| N1—C5—C6—N2 | −1.4 (6) | C12$^{ii}$—O4—Cd1—O3 | −6.6 (8) |
| C4—C5—C6—N2 | 179.6 (4) | C1—N1—Cd1—O5 | −75.5 (4) |
| N1—C5—C6—C7 | 177.1 (4) | C5—N1—Cd1—O5 | 101.3 (4) |
| C4—C5—C6—C7 | −1.9 (7) | C1—N1—Cd1—O4 | 90.0 (4) |
| N2—C6—C7—C8 | −1.3 (7) | C5—N1—Cd1—O4 | −93.2 (4) |
| C5—C6—C7—C8 | −179.7 (5) | C1—N1—Cd1—O6 | 0.6 (5) |
| C6—C7—C8—C9 | 0.1 (8) | C5—N1—Cd1—O6 | 177.4 (3) |
| C7—C8—C9—C10 | 1.9 (8) | C1—N1—Cd1—O1 | 3.4 (3) |
| C8—C9—C10—N2 | −2.8 (7) | C5—N1—Cd1—O1 | −179.7 (4) |
| C8—C9—C10—C12 | 178.1 (5) | C1—N1—Cd1—N2 | −178.7 (4) |
| N1—C1—C11—O2 | 179.8 (4) | C5—N1—Cd1—N2 | −1.8 (3) |
| C2—C1—C11—O2 | −1.3 (7) | C1—N1—Cd1—O3 | −177.9 (3) |

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| N1—C1—C11—O1 | 0.0 (6) | C5—N1—Cd1—O3 | −1.0 (4) |
| C2—C1—C11—O1 | 178.9 (4) | C11—O1—Cd1—O5 | 91.6 (3) |
| N2—C10—C12—O3 | −4.8 (6) | C11—O1—Cd1—O4 | −99.4 (4) |
| C9—C10—C12—O3 | 174.4 (5) | C11—O1—Cd1—O6 | 174.9 (4) |
| N2—C10—C12—O4$^{i}$ | 172.5 (4) | C11—O1—Cd1—N1 | −3.6 (3) |
| C9—C10—C12—O4$^{i}$ | −8.3 (7) | C11—O1—Cd1—N2 | −6.2 (4) |
| C2—C1—N1—C5 | 0.9 (7) | C11—O1—Cd1—O3 | 179.2 (4) |
| C11—C1—N1—C5 | 179.9 (4) | C6—N2—Cd1—O5 | −84.6 (4) |
| C2—C1—N1—Cd1 | 177.9 (4) | C10—N2—Cd1—O5 | 96.3 (3) |
| C11—C1—N1—Cd1 | −3.2 (5) | C6—N2—Cd1—O4 | 94.9 (4) |
| C4—C5—N1—C1 | −1.7 (7) | C10—N2—Cd1—O4 | −84.2 (3) |
| C6—C5—N1—C1 | 179.3 (4) | C6—N2—Cd1—O6 | −178.3 (3) |
| C4—C5—N1—Cd1 | −178.6 (3) | C10—N2—Cd1—O6 | 2.5 (5) |
| C6—C5—N1—Cd1 | 2.4 (5) | C6—N2—Cd1—N1 | 1.0 (3) |
| C7—C6—N2—C10 | 0.4 (7) | C10—N2—Cd1—N1 | −178.1 (4) |
| C5—C6—N2—C10 | 178.9 (4) | C6—N2—Cd1—O1 | 3.6 (4) |
| C7—C6—N2—Cd1 | −178.7 (4) | C10—N2—Cd1—O1 | −175.5 (3) |
| C5—C6—N2—Cd1 | −0.2 (5) | C6—N2—Cd1—O3 | −178.4 (4) |
| C9—C10—N2—C6 | 1.6 (7) | C10—N2—Cd1—O3 | 2.5 (3) |
| C12—C10—N2—C6 | −179.2 (4) | C12—O3—Cd1—O5 | −105.5 (4) |
| C9—C10—N2—Cd1 | −179.2 (4) | C12—O3—Cd1—O4 | 88.8 (4) |
| C12—C10—N2—Cd1 | −0.1 (5) | C12—O3—Cd1—O6 | 174.6 (4) |
| O2—C11—O1—Cd1 | −176.6 (4) | C12—O3—Cd1—N1 | −6.3 (4) |
| C1—C11—O1—Cd1 | 3.3 (5) | C12—O3—Cd1—O1 | 170.3 (4) |
| O4$^{i}$—C12—O3—Cd1 | −169.6 (4) | C12—O3—Cd1—N2 | −5.5 (3) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O7—H7*A*···O4$^{iii}$ | 0.85 | 2.26 | 3.055 (5) | 156 |
| O7—H7*B*···O5$^{iv}$ | 0.87 | 1.88 | 2.729 (5) | 167 |
| O8—H8*A*···O6$^{i}$ | 0.88 | 2.02 | 2.814 (5) | 150 |
| O8—H8*B*···O7$^{v}$ | 0.86 | 2.00 | 2.816 (6) | 158 |

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