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# Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-$\kappa^2N^3,O^4$)cadmium(II) 3.5-hydrate

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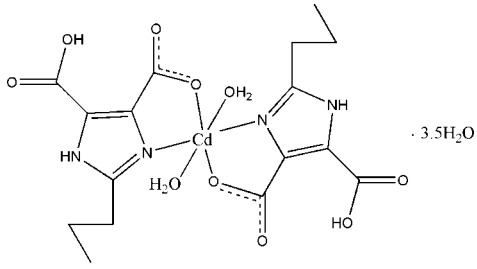
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.041; $wR$ factor = 0.107; data-to-parameter ratio = 13.0.

In the title complex, $[Cd(C_8H_9N_2O_4)_2(H_2O)_2]\cdot3.5H_2O$, the $Cd^{II}$ is coordinated by two water molecules and *N*,*O*-chelated by two 5-carboxy-2-propyl-1*H*-imidazole-4-carboxylate anions in a distorted octahedral geometry. The two imidazole rings are oriented to each other with a dihedral angle of 75.1 (2)°. Strong O—H···O hydrogen bonds between protonated and deprotonated carboxylate groups occur in the molecular structure. In the crystal structure extensive O—H···O and N—H···O hydrogen bonds help to stabilize the three-dimensional supramolecular framework. The propyl groups of anions are disordered over two sites with refined occupancies of 0.768 (6):0.232 (6) and 0.642 (8):0.358 (8).

## Related literature

For the potential uses and diverse structural types of metal complexes with the imidazole-4,5-dicarboxylate ligand, see: Zou *et al.* (2006); Li *et al.* (2006); Liu *et al.* (2004); Sun *et al.* (2005). For related structures, see: Yan *et al.* (2010); Li *et al.* (2010); Song *et al.* (2010); He *et al.* (2010); Fan *et al.* (2010).




## Experimental

### Crystal data

$[Cd(C_8H_9N_2O_4)_2(H_2O)_2]\cdot3.5H_2O$
$M_r$ = 605.83
Triclinic, $P\overline{1}$
$a$ = 10.6228 (12) Å
$b$ = 10.7000 (12) Å
$c$ = 11.3694 (13) Å
$\alpha$ = 83.690 (1)°
$\beta$ = 81.701 (1)°
$\gamma$ = 87.441 (1)°
$V$ = 1270.5 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.93 mm$^{-1}$
$T$ = 296 K
0.29 × 0.24 × 0.21 mm

### Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.775, $T_{max}$ = 0.829
6589 measured reflections
4494 independent reflections
3730 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
$S$ = 1.05
4494 reflections
346 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.59 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O4$W$ | 0.86 | 1.89 | 2.747 (5) | 172 |
| N4—H4···O6$W$ | 0.86 | 1.90 | 2.729 (5) | 161 |
| O1—H1···O4 | 0.82 (3) | 1.67 (3) | 2.490 (5) | 172 (7) |
| O7—H7···O6 | 0.82 (4) | 1.63 (4) | 2.440 (5) | 171 (8) |
| O1$W$—H1$W$···O5$W^{i}$ | 0.85 | 2.27 | 2.654 (5) | 108 |
| O1$W$—H2$W$···O8$^{ii}$ | 0.85 | 1.90 | 2.737 (5) | 168 |
| O2$W$—H3$W$···O8$^{iii}$ | 0.84 | 2.08 | 2.852 (5) | 152 |
| O2$W$—H4$W$···O2$^{iv}$ | 0.86 | 1.99 | 2.818 (5) | 163 |
| O3$W$—H5$W$···O3$^{iv}$ | 0.85 | 2.19 | 2.794 (7) | 128 |
| O3$W$—H6$W$···O3$^{v}$ | 0.85 | 1.96 | 2.770 (6) | 160 |
| O4$W$—H7$W$···O3$W$ | 0.85 | 1.78 | 2.620 (8) | 169 |
| O4$W$—H8$W$···O7$^{vi}$ | 0.85 | 2.05 | 2.904 (6) | 180 |
| O5$W$—H9$W$···O5$^{vi}$ | 0.87 | 2.01 | 2.841 (5) | 159 |
| O5$W$—H10$W$···O4$^{vii}$ | 0.85 | 1.95 | 2.778 (5) | 163 |
| O6$W$—H11$W$···O3$W^{vii}$ | 0.85 | 1.87 | 2.679 (8) | 160 |
| O6$W$—H12$W$···O5$W^{viii}$ | 0.85 | 2.22 | 2.850 (5) | 130 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: XU5001).

## References


Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fan, R.-Z., Li, S.-J., Song, W.-D., Miao, D.-L. & Hu, S.-W. (2010). *Acta Cryst.* E**66**, m897–m898.

He, L.-Z., Li, S.-J., Song, W.-D. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m896.

Li, C.-J., Hu, S., Li, W., Lam, C. K., Zheng, Y.-Z. & Tong, M.-L. (2006). *Eur. J. Inorg. Chem.* pp. 1931–1935.

Li, S.-J., Yan, J.-B., Song, W.-D., Wang, H. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m280.

Liu, Y.-L., Kravtsov, V., Walsh, R. D., Poddar, P., Srikanth, H. & Eddaoudi, M. (2004). *Chem. Commun.* pp. 2806–2807.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Song, W.-D., Yan, J.-B., Li, S.-J., Miao, D.-L. & Li, X.-F. (2010). *Acta Cryst.* E**66**, m53.

Sun, Y.-Q., Zhang, J., Chen, Y.-M. & Yang, G.-Y. (2005). *Angew. Chem. Int. Ed.* **44**, 5814–5817.

Yan, J.-B., Li, S.-J., Song, W.-D., Wang, H. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m99.

Zou, R.-Q., Sakurai, H. & Xu, Q. (2006). *Angew. Chem. Int. Ed.* **45**, 2542–2546.

# supporting information

*Acta Cryst.* (2010). E**66**, m1175–m1176 [https://doi.org/10.1107/S1600536810031466]

## Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-$\kappa^2N^3,O^4$)cadmium(II) 3.5-hydrate

**Shi-Jie Li, Jing-Jing Dong, Wen-Dong Song, Jian-Bin Yan and Shi-Hong Li**

### S1. Comment

Imidazole-4,5-dicarboxylic acid ($H_3IDC$) has been widely used to coordinate with metal salts to obtain a series of MOFs with different structures and useful properties(Zou *et al.*, 2006; Li *et al.*, 2006; Liu *et al.*, 2004; Sun *et al.*, 2005), 2-propyl-1*H*-imidazole-4,5-carboxylate($H_3$pimda) ligand as one derivative of $H_3IDC$ with efficient N,*O*-donors has been used to obtain new metal-organic complexes by our research group, such as poly[diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato- k$^3$ N$^3$,*O*$^4$,*O*$^5$)calcium(II)] (Song *et al.*, 2010), [diaquabis (5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-k$^2$N$^3$,*O*$^4$) manganese(II)]*N*,*N*-dimethylformamide (Yan *et al.*, 2010), [Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-k$^2$ *N*$^3$,*O*$^4$)nickle(II)]*N*,*N*-dimethylformamide disolvate(Li *et al.*, 2010), Diaquabis(4-carboxy-2-propyl-1*H*-imidazole-5-carboxylato- k$^2$N$^3$,*O*$^4$)copper(II) *N*,*N*-dimethylformamide disolvate (He *et al.*, 2010) and Diaquabis(5-carboxy-2-propyl-1*H*-imidazole- 4-carboxylato-k$^2$N$^3$,*O*$^4$)nickle(II) tetrahedrate (Fan *et al.*, 2010). In this paper, we report the structure of a new Cd(II) complex obtained under hydrothermal conditions.

As illustrated in figure 1, the title complex molecule is similar to diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato- $\kappa^2N,O$)nickle(II) tetrahedrate (Fan *et al.* 2010), contains one $Cd^{II}$ ion, two mono-deprotonated $H_2pimda^-$ anions, two coordinated water molecules and 3.5 solvent water molecules. The $Cd^{II}$ is six-coordinated by two N,*O*-bidentate $H_3$pimda anions and two water molecules in a distorted octahedral geometry. the dihedral angle between the two imidazole rings is 75.0 (1) %A. In the crystal structure, the three-dimensional supramolecular framework is stabilized by extensive O—H···O and N—H···O hydrogen bonds involving the free water molecules, the coordinated water molecules, the carboxy O atoms and the protonated N atoms of $H_3$pimda. The propyl groups of $H_3$pimda are disordered over two sets of sites with refined occupiencies of 0.772 (6):0.228 (6) and 0.642 (8):0.358 (8).

### S2. Experimental

A mixture of $CdCl_2$ (0.5 mmol, 0.09 g) and 2-propyl-1*H*-imidazole-4,5-dicarboxylic acid (0.5 mmol, 0.99 g) in 15 ml water was sealed in an autoclave equipped with a Teflon liner (20 ml) and then heated at 433 K for 4 days. Crystals of the title compound were obtained by slow evaporation of the solvent at room temperature.

### S3. Refinement

Water H atoms were located in a difference Fourier map and were allowed to ride on the parent atom, with $U_{iso}$(H) = 1.5$U_{eq}$(O). Carboxyl H atoms were located in a difference map and refined with distance restraints, $U_{iso}$(H) = 1.5$U_{eq}$(O). Other H atoms were placed at calculated positions and were treated as riding on parent atoms with C—H = 0.96 (methyl), 0.97 (methylene) and N—H = 0.86 Å, $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C,*N*). The propyl groups of $H_3$pimda are disordered over two sites with refined occupancies of 0.768 (6):0.232 (6) and 0.642 (8):0.358 (8). C—C distance restraints of disordered components were applied. The O3W water molecule is located close to an inversion center, its occupancy factor was

refined to 0.49 (1) and was fixed as 0.5 at the final refinements.

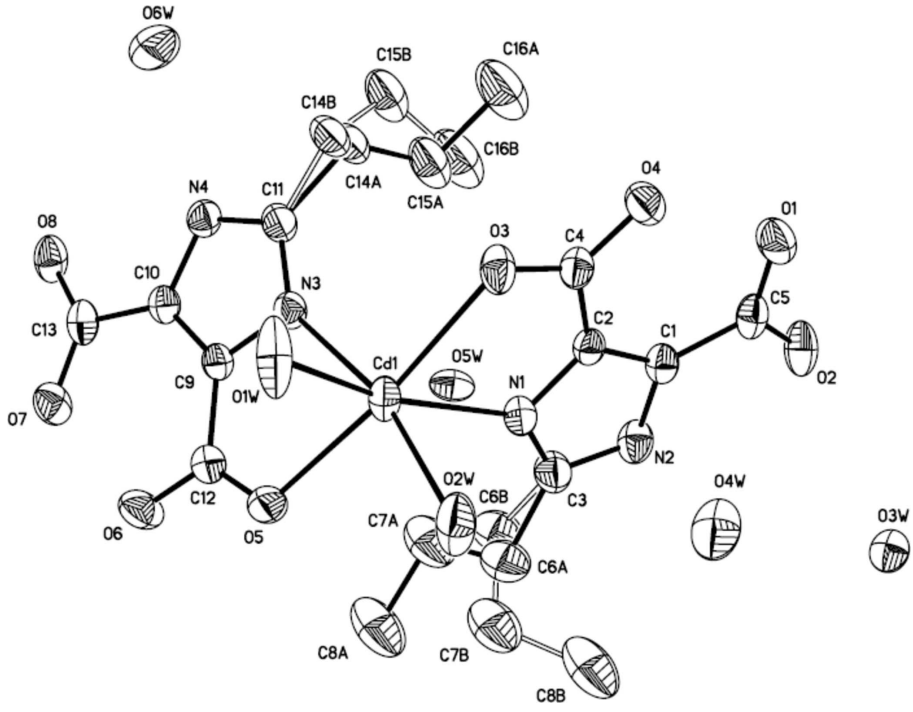


**Figure 1**

The structure of the title compound, showing the atomic-numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids.

## Diaquabis(5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-$\kappa^2N^3,O^4$)cadmium(II) 3.5-hydrate

*Crystal data*

$[Cd(C_8H_9N_2O_4)_2(H_2O)_2]\cdot 3.5H_2O$
$M_r = 605.83$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.6228\ (12)$ Å
$b = 10.7000\ (12)$ Å
$c = 11.3694\ (13)$ Å
$\alpha = 83.690\ (1)°$
$\beta = 81.701\ (1)°$
$\gamma = 87.441\ (1)°$
$V = 1270.5\ (2)$ Å$^3$
$Z = 2$
$F(000) = 618$
$D_x = 1.584$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3600 reflections
$\theta = 1.4–25.0°$
$\mu = 0.93$ mm$^{-1}$
$T = 296$ K
Block, colorless
0.29 × 0.24 × 0.21 mm

*Data collection*

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.775$, $T_{max} = 0.829$
6589 measured reflections
4494 independent reflections
3730 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.2°$, $\theta_{min} = 1.9°$
$h = -12\rightarrow12$
$k = -12\rightarrow8$
$l = -13\rightarrow13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.107$
$S = 1.05$
4494 reflections
346 parameters
7 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
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.1685P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å$^{-3}$
$\Delta\rho_{min} = -0.59$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cd1 | 0.84023 (3) | 0.29169 (3) | 0.19976 (2) | 0.04816 (13) | |
| O1 | 0.8778 (4) | −0.1913 (3) | 0.5186 (3) | 0.0698 (9) | |
| O2 | 0.8413 (3) | −0.1136 (3) | 0.6913 (3) | 0.0701 (9) | |
| O3 | 0.8810 (3) | 0.0754 (3) | 0.1940 (2) | 0.0565 (7) | |
| O4 | 0.8944 (3) | −0.1083 (3) | 0.3037 (3) | 0.0656 (8) | |
| O5 | 0.7595 (3) | 0.5000 (3) | 0.2111 (3) | 0.0553 (7) | |
| O6 | 0.5964 (3) | 0.6304 (3) | 0.1835 (3) | 0.0660 (8) | |
| O7 | 0.3809 (3) | 0.6208 (3) | 0.1350 (3) | 0.0604 (8) | |
| O8 | 0.2516 (3) | 0.4784 (3) | 0.0948 (3) | 0.0606 (8) | |
| N1 | 0.8262 (3) | 0.2015 (3) | 0.3933 (3) | 0.0454 (7) | |
| N2 | 0.8116 (3) | 0.1310 (3) | 0.5823 (3) | 0.0550 (9) | |
| H2 | 0.8009 | 0.1302 | 0.6588 | 0.066* | |
| N3 | 0.6294 (3) | 0.2956 (3) | 0.1734 (3) | 0.0438 (7) | |
| N4 | 0.4358 (3) | 0.2862 (3) | 0.1287 (3) | 0.0479 (8) | |
| H4 | 0.3681 | 0.2556 | 0.1112 | 0.058* | |
| C1 | 0.8362 (3) | 0.0280 (4) | 0.5209 (3) | 0.0447 (9) | |
| C2 | 0.8454 (3) | 0.0742 (3) | 0.4021 (3) | 0.0395 (8) | |
| C3 | 0.8069 (5) | 0.2337 (4) | 0.5040 (4) | 0.0591 (11) | |
| C4 | 0.8760 (4) | 0.0090 (4) | 0.2913 (3) | 0.0473 (9) | |
| C5 | 0.8512 (4) | −0.0992 (4) | 0.5834 (4) | 0.0517 (10) | |
| C6A | 0.7979 (9) | 0.3683 (10) | 0.5325 (10) | 0.085 (3) | 0.772 (6) |
| H6A | 0.8215 | 0.3717 | 0.6114 | 0.102* | 0.772 (6) |
| H6B | 0.8579 | 0.4174 | 0.4753 | 0.102* | 0.772 (6) |
| C7A | 0.6674 (9) | 0.4249 (7) | 0.5292 (8) | 0.107 (3) | 0.772 (6) |

| | | | | | |
|---|---|---|---|---|---|
| H7A | 0.6091 | 0.3854 | 0.5949 | 0.128* | 0.772 (6) |
| H7B | 0.6379 | 0.4108 | 0.4550 | 0.128* | 0.772 (6) |
| C8A | 0.6699 (11) | 0.5651 (7) | 0.5390 (9) | 0.129 (4) | 0.772 (6) |
| H8A | 0.5848 | 0.5970 | 0.5604 | 0.193* | 0.772 (6) |
| H8B | 0.7059 | 0.6073 | 0.4635 | 0.193* | 0.772 (6) |
| H8C | 0.7207 | 0.5796 | 0.5992 | 0.193* | 0.772 (6) |
| C6B | 0.740 (4) | 0.350 (4) | 0.549 (4) | 0.085 (3) | 0.228 (6) |
| H6C | 0.6751 | 0.3816 | 0.5002 | 0.102* | 0.228 (6) |
| H6D | 0.6998 | 0.3328 | 0.6313 | 0.102* | 0.228 (6) |
| C7B | 0.844 (3) | 0.442 (3) | 0.540 (3) | 0.107 (3) | 0.228 (6) |
| H7C | 0.8078 | 0.5259 | 0.5500 | 0.128* | 0.228 (6) |
| H7D | 0.8951 | 0.4445 | 0.4616 | 0.128* | 0.228 (6) |
| C8B | 0.924 (3) | 0.399 (3) | 0.637 (3) | 0.129 (4) | 0.228 (6) |
| H8D | 0.9722 | 0.4679 | 0.6515 | 0.193* | 0.228 (6) |
| H8E | 0.9808 | 0.3317 | 0.6117 | 0.193* | 0.228 (6) |
| H8F | 0.8695 | 0.3699 | 0.7086 | 0.193* | 0.228 (6) |
| C9 | 0.5736 (3) | 0.4138 (3) | 0.1688 (3) | 0.0397 (8) | |
| C10 | 0.4529 (3) | 0.4099 (4) | 0.1405 (3) | 0.0413 (8) | |
| C11 | 0.5431 (4) | 0.2209 (4) | 0.1490 (4) | 0.0509 (10) | |
| C12 | 0.6485 (4) | 0.5201 (4) | 0.1898 (3) | 0.0470 (9) | |
| C13 | 0.3529 (4) | 0.5090 (4) | 0.1221 (3) | 0.0481 (9) | |
| C14A | 0.5509 (17) | 0.0797 (5) | 0.1655 (9) | 0.061 (3) | 0.642 (8) |
| H14A | 0.6361 | 0.0508 | 0.1353 | 0.073* | 0.642 (8) |
| H14B | 0.4918 | 0.0464 | 0.1201 | 0.073* | 0.642 (8) |
| C15A | 0.5189 (11) | 0.0302 (8) | 0.2979 (9) | 0.081 (3) | 0.642 (8) |
| H15A | 0.5740 | 0.0680 | 0.3443 | 0.098* | 0.642 (8) |
| H15B | 0.4315 | 0.0537 | 0.3267 | 0.098* | 0.642 (8) |
| C16A | 0.5361 (10) | −0.1097 (8) | 0.3138 (11) | 0.108 (4) | 0.642 (8) |
| H16A | 0.5605 | −0.1354 | 0.3912 | 0.161* | 0.642 (8) |
| H16B | 0.6013 | −0.1358 | 0.2531 | 0.161* | 0.642 (8) |
| H16C | 0.4576 | −0.1479 | 0.3075 | 0.161* | 0.642 (8) |
| C14B | 0.572 (3) | 0.0878 (9) | 0.119 (2) | 0.061 (3) | 0.358 (8) |
| H14C | 0.6611 | 0.0664 | 0.1216 | 0.073* | 0.358 (8) |
| H14D | 0.5526 | 0.0790 | 0.0400 | 0.073* | 0.358 (8) |
| C15B | 0.4893 (14) | 0.0002 (15) | 0.2123 (14) | 0.081 (3) | 0.358 (8) |
| H15C | 0.4005 | 0.0244 | 0.2101 | 0.098* | 0.358 (8) |
| H15D | 0.5017 | −0.0851 | 0.1905 | 0.098* | 0.358 (8) |
| C16B | 0.518 (3) | 0.002 (2) | 0.3363 (17) | 0.108 (4) | 0.358 (8) |
| H16D | 0.4796 | −0.0688 | 0.3859 | 0.161* | 0.358 (8) |
| H16E | 0.4832 | 0.0784 | 0.3669 | 0.161* | 0.358 (8) |
| H16F | 0.6080 | −0.0025 | 0.3363 | 0.161* | 0.358 (8) |
| O1W | 0.8927 (3) | 0.3292 (4) | 0.0015 (3) | 0.0955 (13) | |
| H1W | 0.9095 | 0.2617 | −0.0314 | 0.143* | |
| H2W | 0.8569 | 0.3905 | −0.0363 | 0.143* | |
| O2W | 1.0419 (3) | 0.3400 (4) | 0.2218 (3) | 0.0897 (12) | |
| H3W | 1.0847 | 0.4010 | 0.1869 | 0.135* | |
| H4W | 1.0911 | 0.2778 | 0.2405 | 0.135* | |
| O3W | 0.9149 (6) | 0.0145 (6) | 0.9607 (5) | 0.0625 (15) | 0.50 |

| | | | | | |
|---|---|---|---|---|---|
| H5W | 0.9951 | 0.0020 | 0.9528 | 0.094* | 0.50 |
| H6W | 0.9090 | 0.0145 | 1.0360 | 0.094* | 0.50 |
| O4W | 0.7737 (5) | 0.1534 (4) | 0.8233 (3) | 0.1211 (17) | |
| H7W | 0.8269 | 0.1083 | 0.8598 | 0.182* | |
| H8W | 0.7288 | 0.2197 | 0.8353 | 0.182* | |
| O5W | 0.1097 (3) | 0.2735 (3) | 0.8678 (3) | 0.0718 (9) | |
| H9W | 0.1608 | 0.3312 | 0.8299 | 0.108* | |
| H10W | 0.1151 | 0.2123 | 0.8255 | 0.108* | |
| O6W | 0.2598 (4) | 0.1582 (4) | 0.0406 (4) | 0.1044 (13) | |
| H11W | 0.2130 | 0.0949 | 0.0534 | 0.157* | |
| H12W | 0.2045 | 0.2161 | 0.0283 | 0.157* | |
| H1 | 0.876 (8) | −0.162 (7) | 0.449 (2) | 0.157* | |
| H7 | 0.453 (3) | 0.632 (7) | 0.149 (7) | 0.157* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cd1 | 0.04170 (18) | 0.0518 (2) | 0.0479 (2) | 0.00347 (13) | −0.00891 (12) | 0.01025 (13) |
| O1 | 0.091 (2) | 0.0495 (19) | 0.067 (2) | −0.0007 (16) | −0.0173 (19) | 0.0084 (16) |
| O2 | 0.070 (2) | 0.079 (2) | 0.0510 (19) | 0.0116 (17) | −0.0047 (15) | 0.0254 (15) |
| O3 | 0.0718 (19) | 0.0587 (18) | 0.0362 (15) | 0.0191 (14) | −0.0077 (13) | −0.0008 (13) |
| O4 | 0.096 (2) | 0.0453 (18) | 0.0579 (18) | 0.0127 (16) | −0.0199 (16) | −0.0092 (14) |
| O5 | 0.0471 (16) | 0.0495 (17) | 0.0723 (19) | −0.0052 (13) | −0.0187 (14) | −0.0049 (14) |
| O6 | 0.0647 (19) | 0.0398 (17) | 0.096 (2) | 0.0045 (14) | −0.0164 (17) | −0.0127 (16) |
| O7 | 0.0518 (17) | 0.0510 (18) | 0.077 (2) | 0.0153 (14) | −0.0098 (15) | −0.0072 (15) |
| O8 | 0.0370 (15) | 0.069 (2) | 0.071 (2) | 0.0022 (13) | −0.0108 (13) | 0.0172 (15) |
| N1 | 0.0490 (18) | 0.0434 (19) | 0.0429 (18) | 0.0051 (14) | −0.0080 (14) | −0.0005 (14) |
| N2 | 0.063 (2) | 0.064 (2) | 0.0355 (17) | 0.0098 (17) | −0.0058 (15) | −0.0041 (16) |
| N3 | 0.0387 (17) | 0.0403 (18) | 0.0517 (19) | 0.0014 (14) | −0.0099 (14) | 0.0012 (14) |
| N4 | 0.0384 (17) | 0.050 (2) | 0.055 (2) | −0.0057 (14) | −0.0133 (14) | 0.0032 (15) |
| C1 | 0.038 (2) | 0.049 (2) | 0.044 (2) | 0.0000 (16) | −0.0035 (16) | 0.0043 (18) |
| C2 | 0.0334 (18) | 0.044 (2) | 0.041 (2) | 0.0010 (15) | −0.0074 (15) | −0.0007 (16) |
| C3 | 0.076 (3) | 0.053 (3) | 0.050 (3) | 0.009 (2) | −0.014 (2) | −0.006 (2) |
| C4 | 0.047 (2) | 0.050 (2) | 0.045 (2) | 0.0074 (17) | −0.0120 (17) | −0.0020 (18) |
| C5 | 0.043 (2) | 0.061 (3) | 0.047 (2) | 0.0004 (18) | −0.0083 (18) | 0.014 (2) |
| C6A | 0.109 (9) | 0.068 (5) | 0.078 (5) | 0.011 (6) | −0.009 (6) | −0.021 (4) |
| C7A | 0.133 (8) | 0.069 (5) | 0.115 (7) | 0.011 (5) | −0.004 (5) | −0.017 (5) |
| C8A | 0.174 (10) | 0.067 (5) | 0.136 (8) | 0.020 (5) | 0.006 (7) | −0.012 (5) |
| C6B | 0.109 (9) | 0.068 (5) | 0.078 (5) | 0.011 (6) | −0.009 (6) | −0.021 (4) |
| C7B | 0.133 (8) | 0.069 (5) | 0.115 (7) | 0.011 (5) | −0.004 (5) | −0.017 (5) |
| C8B | 0.174 (10) | 0.067 (5) | 0.136 (8) | 0.020 (5) | 0.006 (7) | −0.012 (5) |
| C9 | 0.0391 (19) | 0.039 (2) | 0.0383 (19) | 0.0003 (15) | −0.0029 (15) | 0.0024 (15) |
| C10 | 0.039 (2) | 0.044 (2) | 0.0378 (19) | −0.0004 (16) | 0.0002 (15) | 0.0032 (16) |
| C11 | 0.048 (2) | 0.043 (2) | 0.061 (3) | −0.0025 (18) | −0.0127 (19) | 0.0029 (19) |
| C12 | 0.048 (2) | 0.046 (2) | 0.047 (2) | 0.0007 (18) | −0.0077 (17) | −0.0012 (17) |
| C13 | 0.041 (2) | 0.056 (3) | 0.040 (2) | 0.0088 (18) | 0.0033 (16) | 0.0075 (18) |
| C14A | 0.061 (7) | 0.036 (3) | 0.087 (9) | −0.006 (3) | −0.022 (7) | 0.007 (4) |
| C15A | 0.062 (5) | 0.058 (5) | 0.121 (8) | −0.003 (4) | −0.028 (5) | 0.023 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16A | 0.101 (7) | 0.059 (5) | 0.157 (10) | −0.007 (5) | −0.023 (6) | 0.023 (6) |
| C14B | 0.061 (7) | 0.036 (3) | 0.087 (9) | −0.006 (3) | −0.022 (7) | 0.007 (4) |
| C15B | 0.062 (5) | 0.058 (5) | 0.121 (8) | −0.003 (4) | −0.028 (5) | 0.023 (5) |
| C16B | 0.101 (7) | 0.059 (5) | 0.157 (10) | −0.007 (5) | −0.023 (6) | 0.023 (6) |
| O1W | 0.085 (3) | 0.121 (3) | 0.059 (2) | 0.052 (2) | 0.0107 (18) | 0.035 (2) |
| O2W | 0.0499 (19) | 0.100 (3) | 0.110 (3) | −0.0224 (18) | −0.0251 (18) | 0.057 (2) |
| O3W | 0.065 (4) | 0.075 (4) | 0.048 (3) | 0.008 (3) | −0.007 (3) | −0.013 (3) |
| O4W | 0.156 (4) | 0.142 (4) | 0.072 (2) | 0.058 (3) | −0.030 (3) | −0.048 (3) |
| O5W | 0.0623 (19) | 0.069 (2) | 0.084 (2) | −0.0175 (16) | 0.0083 (16) | −0.0255 (17) |
| O6W | 0.101 (3) | 0.098 (3) | 0.131 (4) | −0.001 (2) | −0.064 (3) | −0.025 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cd1—O1W | 2.238 (3) | C6B—H6D | 0.9700 |
| Cd1—O2W | 2.281 (3) | C7B—C8B | 1.512 (14) |
| Cd1—N1 | 2.290 (3) | C7B—H7C | 0.9700 |
| Cd1—N3 | 2.300 (3) | C7B—H7D | 0.9700 |
| Cd1—O3 | 2.341 (3) | C8B—H8D | 0.9600 |
| Cd1—O5 | 2.362 (3) | C8B—H8E | 0.9600 |
| O1—C5 | 1.291 (5) | C8B—H8F | 0.9600 |
| O1—H1 | 0.82 (3) | C9—C10 | 1.370 (5) |
| O2—C5 | 1.209 (5) | C9—C12 | 1.476 (5) |
| O3—C4 | 1.243 (5) | C10—C13 | 1.487 (5) |
| O4—C4 | 1.257 (5) | C11—C14A | 1.502 (7) |
| O5—C12 | 1.243 (5) | C11—C14B | 1.507 (9) |
| O6—C12 | 1.280 (5) | C14A—C15A | 1.534 (12) |
| O7—C13 | 1.275 (5) | C14A—H14A | 0.9700 |
| O7—H7 | 0.82 (4) | C14A—H14B | 0.9700 |
| O8—C13 | 1.229 (5) | C15A—C16A | 1.495 (10) |
| N1—C3 | 1.327 (5) | C15A—H15A | 0.9700 |
| N1—C2 | 1.363 (5) | C15A—H15B | 0.9700 |
| N2—C3 | 1.341 (5) | C16A—H16A | 0.9600 |
| N2—C1 | 1.364 (5) | C16A—H16B | 0.9600 |
| N2—H2 | 0.8600 | C16A—H16C | 0.9600 |
| N3—C11 | 1.320 (5) | C14B—C15B | 1.532 (14) |
| N3—C9 | 1.371 (5) | C14B—H14C | 0.9700 |
| N4—C11 | 1.346 (5) | C14B—H14D | 0.9700 |
| N4—C10 | 1.368 (5) | C15B—C16B | 1.486 (13) |
| N4—H4 | 0.8600 | C15B—H15C | 0.9700 |
| C1—C2 | 1.377 (5) | C15B—H15D | 0.9700 |
| C1—C5 | 1.478 (6) | C16B—H16D | 0.9600 |
| C2—C4 | 1.495 (5) | C16B—H16E | 0.9600 |
| C3—C6A | 1.506 (11) | C16B—H16F | 0.9600 |
| C3—C6B | 1.51 (4) | O1W—H1W | 0.8499 |
| C6A—C7A | 1.492 (12) | O1W—H2W | 0.8500 |
| C6A—H6A | 0.9700 | O2W—H3W | 0.8405 |
| C6A—H6B | 0.9700 | O2W—H4W | 0.8547 |
| C7A—C8A | 1.518 (10) | O3W—H5W | 0.8500 |

| | | | |
|---|---|---|---|
| C7A—H7A | 0.9700 | O3W—H6W | 0.8501 |
| C7A—H7B | 0.9700 | O4W—H7W | 0.8503 |
| C8A—H8A | 0.9600 | O4W—H8W | 0.8498 |
| C8A—H8B | 0.9600 | O5W—H9W | 0.8741 |
| C8A—H8C | 0.9600 | O5W—H10W | 0.8500 |
| C6B—C7B | 1.498 (15) | O6W—H11W | 0.8452 |
| C6B—H6C | 0.9700 | O6W—H12W | 0.8490 |

| | | | |
|---|---|---|---|
| O1W—Cd1—O2W | 88.94 (14) | C8B—C7B—H7C | 110.3 |
| O1W—Cd1—N1 | 162.70 (12) | C6B—C7B—H7D | 110.3 |
| O2W—Cd1—N1 | 85.50 (12) | C8B—C7B—H7D | 110.3 |
| O1W—Cd1—N3 | 89.03 (13) | H7C—C7B—H7D | 108.5 |
| O2W—Cd1—N3 | 165.95 (12) | C7B—C8B—H8D | 109.5 |
| N1—Cd1—N3 | 100.21 (11) | C7B—C8B—H8E | 109.5 |
| O1W—Cd1—O3 | 91.83 (12) | H8D—C8B—H8E | 109.5 |
| O2W—Cd1—O3 | 96.11 (12) | C7B—C8B—H8F | 109.5 |
| N1—Cd1—O3 | 72.55 (10) | H8D—C8B—H8F | 109.5 |
| N3—Cd1—O3 | 97.85 (10) | H8E—C8B—H8F | 109.5 |
| O1W—Cd1—O5 | 90.99 (12) | C10—C9—N3 | 110.1 (3) |
| O2W—Cd1—O5 | 94.05 (12) | C10—C9—C12 | 131.2 (3) |
| N1—Cd1—O5 | 105.72 (10) | N3—C9—C12 | 118.6 (3) |
| N3—Cd1—O5 | 72.08 (10) | N4—C10—C9 | 105.1 (3) |
| O3—Cd1—O5 | 169.50 (10) | N4—C10—C13 | 122.2 (3) |
| C5—O1—H1 | 107 (6) | C9—C10—C13 | 132.6 (4) |
| C4—O3—Cd1 | 117.3 (2) | N3—C11—N4 | 111.0 (3) |
| C12—O5—Cd1 | 115.9 (2) | N3—C11—C14A | 125.1 (8) |
| C13—O7—H7 | 118 (6) | N4—C11—C14A | 122.8 (8) |
| C3—N1—C2 | 106.7 (3) | N3—C11—C14B | 123.8 (15) |
| C3—N1—Cd1 | 140.1 (3) | N4—C11—C14B | 123.8 (16) |
| C2—N1—Cd1 | 113.2 (2) | O5—C12—O6 | 122.5 (4) |
| C3—N2—C1 | 108.9 (3) | O5—C12—C9 | 119.3 (3) |
| C3—N2—H2 | 125.6 | O6—C12—C9 | 118.2 (4) |
| C1—N2—H2 | 125.6 | O8—C13—O7 | 125.1 (4) |
| C11—N3—C9 | 105.6 (3) | O8—C13—C10 | 118.7 (4) |
| C11—N3—Cd1 | 140.3 (3) | O7—C13—C10 | 116.2 (4) |
| C9—N3—Cd1 | 113.7 (2) | C11—C14A—C15A | 110.9 (6) |
| C11—N4—C10 | 108.1 (3) | C11—C14A—H14A | 109.5 |
| C11—N4—H4 | 125.9 | C15A—C14A—H14A | 109.5 |
| C10—N4—H4 | 125.9 | C11—C14A—H14B | 109.5 |
| N2—C1—C2 | 105.1 (3) | C15A—C14A—H14B | 109.5 |
| N2—C1—C5 | 121.4 (4) | H14A—C14A—H14B | 108.0 |
| C2—C1—C5 | 133.4 (4) | C16A—C15A—C14A | 110.3 (8) |
| N1—C2—C1 | 109.3 (3) | C16A—C15A—H15A | 109.6 |
| N1—C2—C4 | 119.8 (3) | C14A—C15A—H15A | 109.6 |
| C1—C2—C4 | 130.8 (4) | C16A—C15A—H15B | 109.6 |
| N1—C3—N2 | 110.0 (4) | C14A—C15A—H15B | 109.6 |
| N1—C3—C6A | 123.2 (6) | H15A—C15A—H15B | 108.1 |
| N2—C3—C6A | 126.4 (6) | C11—C14B—C15B | 108.1 (13) |

| | | | |
|---|---|---|---|
| N1—C3—C6B | 129 (2) | C11—C14B—H14C | 110.1 |
| N2—C3—C6B | 117.6 (18) | C15B—C14B—H14C | 110.1 |
| O3—C4—O4 | 125.2 (4) | C11—C14B—H14D | 110.1 |
| O3—C4—C2 | 117.2 (4) | C15B—C14B—H14D | 110.1 |
| O4—C4—C2 | 117.6 (3) | H14C—C14B—H14D | 108.4 |
| O2—C5—O1 | 122.4 (4) | C16B—C15B—C14B | 113.8 (19) |
| O2—C5—C1 | 119.9 (4) | C16B—C15B—H15C | 108.8 |
| O1—C5—C1 | 117.6 (4) | C14B—C15B—H15C | 108.8 |
| C7A—C6A—C3 | 112.3 (8) | C16B—C15B—H15D | 108.8 |
| C7A—C6A—H6A | 109.2 | C14B—C15B—H15D | 108.8 |
| C3—C6A—H6A | 109.2 | H15C—C15B—H15D | 107.7 |
| C7A—C6A—H6B | 109.2 | C15B—C16B—H16D | 109.5 |
| C3—C6A—H6B | 109.2 | C15B—C16B—H16E | 109.5 |
| H6A—C6A—H6B | 107.9 | H16D—C16B—H16E | 109.5 |
| C6A—C7A—C8A | 109.4 (8) | C15B—C16B—H16F | 109.5 |
| C6A—C7A—H7A | 109.8 | H16D—C16B—H16F | 109.5 |
| C8A—C7A—H7A | 109.8 | H16E—C16B—H16F | 109.5 |
| C6A—C7A—H7B | 109.8 | Cd1—O1W—H1W | 112.0 |
| C8A—C7A—H7B | 109.8 | Cd1—O1W—H2W | 119.5 |
| H7A—C7A—H7B | 108.2 | H1W—O1W—H2W | 118.7 |
| C7B—C6B—C3 | 104 (3) | Cd1—O2W—H3W | 127.7 |
| C7B—C6B—H6C | 111.0 | Cd1—O2W—H4W | 116.0 |
| C3—C6B—H6C | 111.0 | H3W—O2W—H4W | 110.5 |
| C7B—C6B—H6D | 111.0 | H5W—O3W—H6W | 92.8 |
| C3—C6B—H6D | 111.0 | H7W—O4W—H8W | 136.0 |
| H6C—C6B—H6D | 109.0 | H9W—O5W—H10W | 107.5 |
| C6B—C7B—C8B | 107 (3) | H11W—O6W—H12W | 100.2 |
| C6B—C7B—H7C | 110.3 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O4*W* | 0.86 | 1.89 | 2.747 (5) | 172 |
| N4—H4···O6*W* | 0.86 | 1.90 | 2.729 (5) | 161 |
| O1—H1···O4 | 0.82 (3) | 1.67 (3) | 2.490 (5) | 172 (7) |
| O7—H7···O6 | 0.82 (4) | 1.63 (4) | 2.440 (5) | 171 (8) |
| O1*W*—H1*W*···O5*W*$^{i}$ | 0.85 | 2.27 | 2.654 (5) | 108 |
| O1*W*—H2*W*···O8$^{ii}$ | 0.85 | 1.90 | 2.737 (5) | 168 |
| O2*W*—H3*W*···O8$^{iii}$ | 0.84 | 2.08 | 2.852 (5) | 152 |
| O2*W*—H4*W*···O2$^{iv}$ | 0.86 | 1.99 | 2.818 (5) | 163 |
| O3*W*—H5*W*···O3$^{iv}$ | 0.85 | 2.19 | 2.794 (7) | 128 |
| O3*W*—H6*W*···O3$^{v}$ | 0.85 | 1.96 | 2.770 (6) | 160 |
| O4*W*—H7*W*···O3*W* | 0.85 | 1.78 | 2.620 (8) | 169 |
| O4*W*—H8*W*···O7$^{vi}$ | 0.85 | 2.05 | 2.904 (6) | 180 |
| O5*W*—H9*W*···O5$^{vi}$ | 0.87 | 2.01 | 2.841 (5) | 159 |
| O5*W*—H10*W*···O4$^{vii}$ | 0.85 | 1.95 | 2.778 (5) | 163 |

| | | | | |
|---|---|---|---|---|
| O6*W*—H11*W*···O3*W*$^{vii}$ | 0.85 | 1.87 | 2.679 (8) | 160 |
| O6*W*—H12*W*···O5*W*$^{viii}$ | 0.85 | 2.22 | 2.850 (5) | 130 |

Symmetry codes: (i) *x*+1, *y*, *z*−1; (ii) −*x*+1, −*y*+1, −*z*; (iii) *x*+1, *y*, *z*; (iv) −*x*+2, −*y*, −*z*+1; (v) *x*, *y*, *z*+1; (vi) −*x*+1, −*y*+1, −*z*+1; (vii) −*x*+1, −*y*, −*z*+1; (viii) *x*, *y*, *z*−1.