



# Synthesis, structure and Hirshfeld surface analysis of a coordination compound of cadmium acetate with 2-aminobenzoxazole

Surayyo Razzoqova,<sup>a</sup> Aziz Ibragimov,<sup>b</sup> Batirbay Torambetov,<sup>a\*</sup> Shakhnoza Kadirova,<sup>a</sup> Tamas Holczbauer,<sup>c</sup> Jamshid Ashurov<sup>d</sup> and Bakhtiyar Ibragimov<sup>d</sup>

Received 8 August 2023

Accepted 22 August 2023

Edited by J. Reibenspies, Texas A & M University, USA

**Keywords:** crystal structure; molecular structure; cadmium complex; 2-aminobenzoxazole; Hirshfeld surface analysis.

**CCDC reference:** 2290113

**Supporting information:** this article has supporting information at journals.iucr.org/e

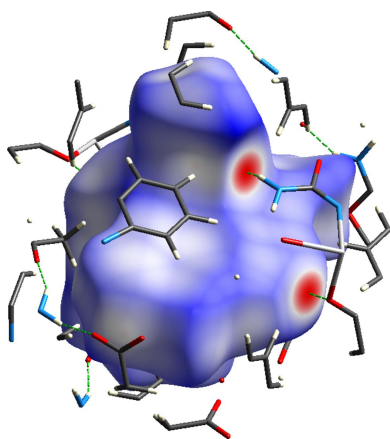
<sup>a</sup>National University of Uzbekistan named after Mirzo Ulugbek, 4 University St, Tashkent 100174, Uzbekistan, <sup>b</sup>Institute of General and Inorganic Chemistry, Academy of Sciences of Uzbekistan, M. Ulugbek Str 77a, Tashkent 100170, Uzbekistan, <sup>c</sup>Institute of Organic Chemistry, Research Centre for Natural Sciences, 2 Magyar tudosok korutja, H-1117 Budapest, Hungary, and <sup>d</sup>Institute of Bioorganic Chemistry, Academy of Sciences of Uzbekistan, M. Ulugbek Str 83, Tashkent 100125, Uzbekistan. \*Correspondence e-mail: torambetov\_b@mail.ru

A first coordination compound of 2-aminobenzoxazole (**2AB**), namely, bis(2-aminobenzoxazole- $\kappa N^3$ )bis(acetato- $\kappa^2 O, O'$ )cadmium(II),  $[\text{Cd}(\text{CH}_3\text{COO})_2(\mathbf{2AB})_2]$ , has been synthesized from ethanol solutions of  $\text{Cd}(\text{CH}_3(\text{COO})_2)$  and **2AB**. In the monoclinic crystals with the space group  $C2_1/c$ , the cadmium ions coordinate two neutral **2AB** molecules in a monodentate fashion through the oxazole N atom, while two acetate ligands are coordinated through the O atoms in a bidentate manner. The coordination polyhedron of the central ion is substantially distorted octahedral. There are two relatively strong intramolecular hydrogen bonds in the complex molecule. Additionally, two intermolecular hydrogen bonds associate complex molecules into columns running in the  $[1\bar{1}0]$  and  $[110]$  directions. The Hirshfeld surface analysis shows that 45.7% of the intermolecular interactions are from  $\text{H}\cdots\text{H}$  contacts, 24.7% are from  $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$  contacts and 18.8% are from  $\text{C}\cdots\text{H}/\text{H}\cdots\text{C}$  contacts, while other contributions are from  $\text{N}\cdots\text{H}/\text{H}\cdots\text{N}$  and  $\text{O}\cdots\text{O}$  contacts.

## 1. Chemical context

Benzoxazole is an aromatic organic compound with a benzene-fused oxazole ring structure and an odour similar to pyridine (Katritzky *et al.*, 2000; Clayden *et al.*, 2001). Although benzoxazole itself is of little practical interest, many benzoxazole derivatives are commercially important. They play an important role in medicinal and biological chemistry (Potashman *et al.*, 2007; Lachtova *et al.*, 2018; Razzoqova *et al.*, 2022), being described as potential therapeutic agents, including as various enzyme inhibitors (Chikhale *et al.*, 2018). Aminobenzoxazoles, in particular derivatives of 2-aminobenzoxazole (**2AB**), have anticancer and antibacterial properties (Khajondetchairit *et al.*, 2017; Ouyang *et al.*, 2012). The 2-amino-5-chlorobenzoxazole derivative is a muscle relaxant and it has been used as an antispasmodic and uricosurics drug (Lynch, 2004).

An analysis of the Cambridge Structural Database (CSD, Version 5.43, update of March 2022; Groom *et al.*, 2016) showed that there are no X-ray structures of **2AB** and its metal complexes in the database. However, recently, we reported the structure and intermolecular interactions of a **2AB**-fumaric acid organic salt in which the N atom of **2AB** is protonated by a fumaric acid H atom (Razzoqova *et al.*, 2022). Theoretically, metal complexes of **2AB** may involve coordination through the N or O atoms of the oxazole ring and the N atom of the amino substituent. In order to define which way

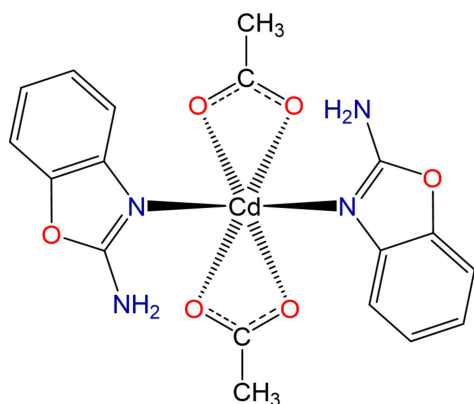


**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H2A\cdots O5$	0.86	1.95	2.762 (4)	157
$N2-H2B\cdots O4^i$	0.86	2.04	2.811 (3)	149
$N4-H4A\cdots O3$	0.86	1.99	2.790 (4)	155
$N4-H4B\cdots O6^{ii}$	0.86	2.04	2.803 (3)	148

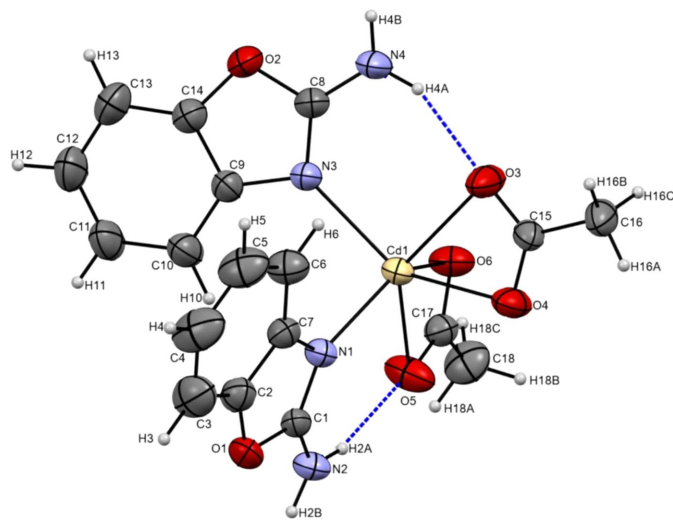
Symmetry codes: (i)  $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$ ; (ii)  $-x + 1, -y + 1, -z + 1$ .

these possibilities will be realized, we have prepared a coordination complex of **2AB** with cadmium and report here its molecular and crystal structure, as well as a Hirshfeld surface analysis.



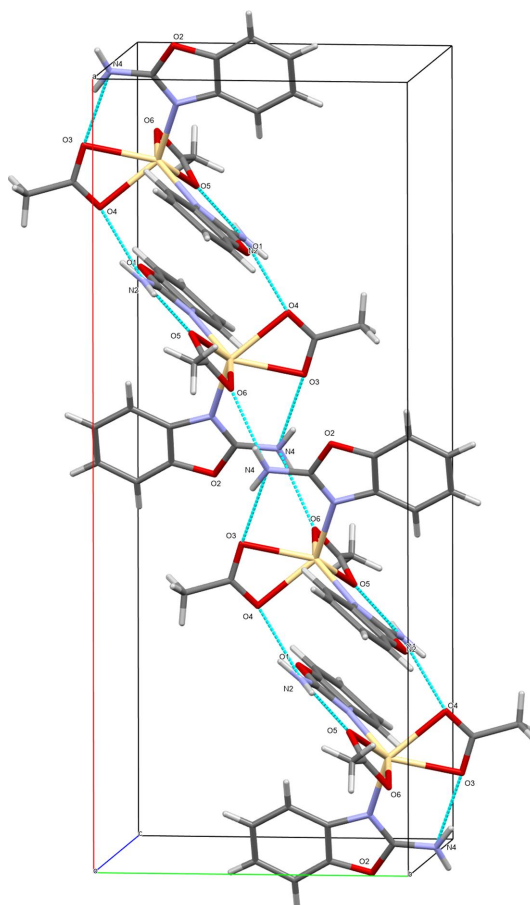
## 2. Structural commentary

The structure of  $[Cd^{2+}(CH_3COO^-)_2(2AB)_2]$  is shown in Fig. 1. The metal complex was obtained using the  $Cd(CH_3COO)_2$  salt for the synthesis. The  $Cd^{II}$  ion coordinates two **2AB** molecules through the oxazole N atom in a monodentate fashion. Furthermore, in order to compensate the positive charge of

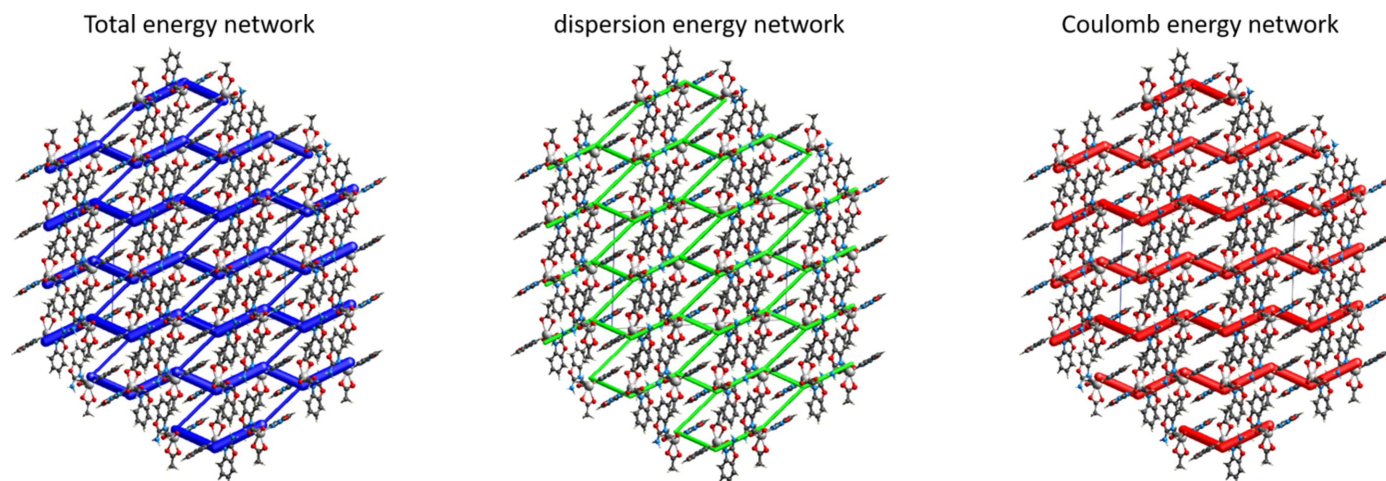


**Figure 1**  
The molecular structure of the title complex with the atom-numbering scheme. Intramolecular hydrogen bonds are indicated by dashed lines. Displacement ellipsoids are plotted at the 30% probability level.

the central atom, two acetate ligands are coordinated in a bidentate manner through the O atoms. Despite the large ionic radius of the Cd atom, the coordination number of the central atom is six, in contrast to, for example, coordination numbers of four or eight in some mixed-ligand cadmium complexes (Kudiyarova *et al.*, 2021; Ibragimov *et al.*, 2017a). The two **2AB** ligands and the two acetate ions are coordinated to the Cd centre in a *cis* arrangement. The bond lengths of the Cd ion are in the range 2.269 (2)–2.400 (2) Å, while the bond angles vary from 53.35 (8) to 139.71 (8)°. Such a large difference in the valence distances and angles leads to a significant distortion of the octahedral coordination environment, caused by the acetate ligands acting as bidentate, with chelating angles of  $O3-Cd1-O4 = 53.57 (8)^\circ$  and  $O5-Cd1-O6 = 53.35 (8)^\circ$  of the cadmium polyhedron. The geometric parameters of the arene ring are similar to standard values and to those in other structures (Ibragimov *et al.*, 2017b; Ruzmetov *et al.*, 2022). In the **2AB** molecules, all the atoms are located on a plane, with the greatest r.m.s. deviations from the main planes seen for the amino atoms N2 (0.017 Å) and N4 (0.026 Å). The dihedral angle between the mean planes of the **2AB** molecules around the cadmium polyhedron is 65.59°. The positions of the ligands allow the formation of two relatively strong intramolecular hydrogen bonds in the complex mol-



**Figure 2**  
The formation of columns by hydrogen bonds in the crystal structure of the title complex. Generic atom labels without symmetry codes have been used.

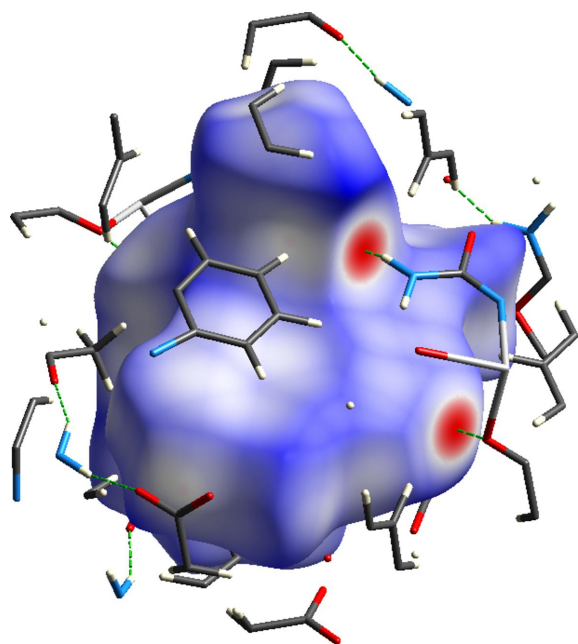

**Figure 3**

Interaction energy calculations within the molecules was performed using the HF method (HF/3-21G) in the *CrystalExplorer17* program. The thickness of the tube represents the value of the energy. The distribution of the interactions according to type shows strong interactions along the crystallographic *a* direction (the largest values are represented here). The total energy framework (in blue) and its two main components, dispersion (in green) and Coulombic energy (in red), are shown for a cluster around a reference molecule. also exhibit stronger interactions along the crystallographic *a* direction.

ecule: in particular, the amino groups  $\text{N2H}_2$  and  $\text{N4H}_2$  form hydrogen bonds with the nearest O atoms, O5 and O3, of the coordinated acetates, with distances of 2.762 (4) and 2.790 (4) Å, respectively (Table 1). These hydrogen bonds enclose six-membered rings with  $S(6)$  graph-set notations (Etter, 1990).

### 3. Supramolecular features

There are two proton-donor hydrogen-bonding groups in the complex molecule, *i.e.*  $\text{N2-H2}$  and  $\text{N4-H4}$ . Both of these


**Figure 4**

View of the three-dimensional Hirshfeld surface of the complex plotted over  $d_{\text{norm}}$  in the range from  $-0.6027$  to  $1.5939$  a.u.

groups realize their hydrogen-bonding capabilities by forming intramolecular  $\text{N2-H2A}\cdots\text{O5}$  and  $\text{N4-H4A}\cdots\text{O3}$  (first two hydrogen bonds in Table 1), and two intermolecular  $\text{N2-H2B}\cdots\text{O4}^i$  and  $\text{N4-H4B}\cdots\text{O6}^{ii}$  hydrogen bonds (the remaining two hydrogen bonds in Table 1). These intermolecular hydrogen bonds between the N atoms of the amino groups and the O atoms of the acetate carboxylate groups associate complex molecules into columns running in the  $[1\bar{1}0]$  and  $[110]$  directions (Fig. 2). The interaction energies of the hydrogen-bond system were calculated within the molecules using the HF method (HF/3-21G) in the *CrystalExplorer17* program (Fig. 3). The result shows the total energy ( $E_{\text{tot}}$ ), which is the sum of the Coulombic ( $E_{\text{ele}}$ ), polar ( $E_{\text{pol}}$ ), dispersion ( $E_{\text{dis}}$ ) and repulsive ( $E_{\text{rep}}$ ) contributions. The four energy components were scaled in the total energy ( $E_{\text{tot}} = 1.019E_{\text{ele}} + 0651E_{\text{pol}} + 0901E_{\text{dis}} + 0.811E_{\text{rep}}$ ). The interaction energies were investigated for a 3.8 Å cluster around the reference molecule. The calculation reveals two stronger interactions within the neighbouring molecules. The strongest interaction total energy ( $E_{\text{tot}}$ ) is  $-113.4$  kJ mol $^{-1}$  ( $\sim -27$  kcal mol $^{-1}$ ), with the highest polar ( $-32.5$  kJ mol $^{-1}$ ), dispersion ( $-51.3$  kJ mol $^{-1}$ ) and repulsive ( $68.1$  kJ mol $^{-1}$ ) energies (green–yellow). The second interaction among neighbouring molecules is similar to the first, with  $E_{\text{tot}} = -97.2$  kJ mol $^{-1}$ . The main attractive interactions (Coulombic, dispersion and the sum total energy) show a stronger bonding effect along the crystallographic *a* direction (Fig. 3).

### 4. Hirshfeld surface analysis

To further investigate the intermolecular interactions present in the title compound, a Hirshfeld surface analysis was performed, and the two-dimensional (2D) fingerprint plots were generated with *CrystalExplorer17* (Spackman *et al.*, 2021). Fig. 4 shows the three-dimensional (3D) Hirshfeld surface of the complex with  $d_{\text{norm}}$  (normalized contact

**Table 2**

Experimental details.

Crystal data	
Chemical formula	[Cd(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>2</sub> (C <sub>7</sub> H <sub>6</sub> N <sub>2</sub> O) <sub>2</sub> ]
<i>M</i> <sub>r</sub>	498.76
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	25.0497 (3), 9.8428 (1), 16.7577 (2)
$\beta$ (°)	94.534 (1)
<i>V</i> (Å <sup>3</sup> )	4118.83 (8)
<i>Z</i>	8
Radiation type	Cu <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	8.87
Crystal size (mm)	0.17 × 0.14 × 0.12
Data collection	
Diffractometer	Rigaku XtaLAB Synergy diffractometer with a HyPix3000 detector
Absorption correction	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2020)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.793, 1.000
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	11316, 3971, 3434
<i>R</i> <sub>int</sub>	0.030
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.614
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.030, 0.078, 1.05
No. of reflections	3971
No. of parameters	265
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.26, -0.59

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT* (Sheldrick, 2015a), *SHELXL2016* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

distance) plotted over the range from -0.6027 to 1.5939 a.u. The hydrogen-bond interactions given in Table 1 play a key

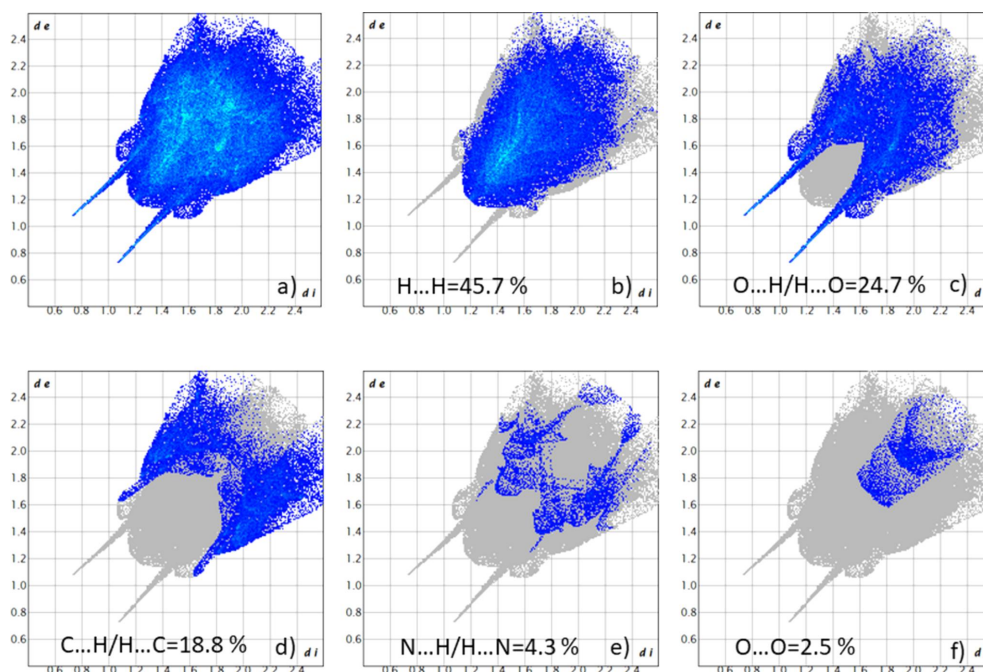
role in the molecular packing of the complex. The overall 2D fingerprint plot and those delineated into H···H, O···H/H···O, C···H/H···C, N···H/H···N and O···O interactions, are shown in Fig. 5. The percentage contributions to the Hirshfeld surfaces from the various interatomic contacts are as follows: H···H 45.7%, O···H/H···O 24.7%, C···H/H···C 18.8%, N···H/H···N 4.3% and O···O 2.5%. Other minor contributions to the Hirshfeld surface are: C···C 2.4% and O···C/C···O 1.6%.

## 5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43, update of March 2022; Groom *et al.*, 2016) for free **2AB** and its metal complexes gave no hits. Cadmium(II) acetate complexes of the general formula [Cd(OAc)<sub>2</sub>L<sub>2</sub>], where cadmium is hexacoordinated, the acetate ligand is attached to cadmium in a bidentate manner and *L* is a monodentate ligand with a ligator N atom, have been reported in the CSD with refcodes ODONEC (Ma *et al.*, 2012), ODONEC01 (Yan *et al.*, 2014), PIBMIX (Zhao *et al.*, 2007), TICDOY (Chotalia *et al.*, 1996), TICMID (Hei *et al.*, 2013) and UGOPOX (Liu *et al.*, 2015).

## 6. Synthesis and crystallization

Cd(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O (0.266 g, 1 mmol) and **2AB** (0.268 g, 2 mmol) were dissolved separately in ethanol (5 ml), mixed together and stirred for 1.5 h. The obtained colourless solution was filtered and left for crystallization. Single crystals of the


**Figure 5**

The full 2D fingerprint plots for the title complex, showing all interactions and delineated into separate interactions. The *d<sub>i</sub>* and *d<sub>e</sub>* values are the closest internal and external distances (Å) from given points on the Hirshfeld surface contacts.

title complex suitable for X-ray analysis were obtained by slow evaporation of the solution over a period of 10 d.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms of the acetate methyl groups were placed in calculated positions and refined in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  and  $\text{C}-\text{H} = 0.96 \text{ \AA}$ . The remaining H atoms were located experimentally and refined freely.

## Funding information

Funding for this research was provided by: Ministry of Higher Education, Science and Innovation of the Republic of Uzbekistan.

## References

- Chikhale, R., Thorat, S., Choudhary, R. K., Gadewal, N. & Khedekar, P. (2018). *Bioorg. Chem.* **77**, 84–100.
- Chotalia, R., Hambley, T. W., Ridley, D. D. & Turner, P. (1996). *Acta Cryst.* **C52**, 3043–3045.
- Clayden, J., Greeves, N., Warren, S. & Wothers, P. (2001). In *Organic Chemistry*. Oxford University Press.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hei, J.-H., Zhi, Y.-X., Zhen, Y., Li, J. & Zhang, F.-X. (2013). *J. Coord. Chem.* **66**, 1320–1329.
- Ibragimov, A. B., Ashurov, Zh. M., Ibragimov, A. B. & Zakirov, B. S. (2017b). *Russ. J. Inorg. Chem.* **62**, 439–445.
- Ibragimov, A. B., Ashurov, Zh. M. & Zakirov, B. S. (2017a). *J. Struct. Chem.* **58**, 588–590.
- Katritzky, A. R. & Pozharskii, A. F. (2000). In *Handbook of Heterocyclic Chemistry*, 2nd ed. New York: Academic Press.
- Khajondetchairit, P., Phuangasawai, O., Suphakun, P., Rattanabunyong, S., Choowongkamon, K. & Gleeson, M. P. (2017). *Chem. Biol. Drug Des.* **90**, 987–994.
- Kudiyarova, A. D., Ashurov, J. M., Ibragimov, A. B., Sabirov, V. K. & Ibragimov, B. T. (2021). *Chem. Data Collect.* **31**, 100633.
- Lachtova, V. & Brulikova, L. (2018). *ChemistrySelect*, **3**, 4653–4662.
- Liu, Y., Wang, D., Zheng, X.-J. & Jin, L.-P. (2015). *RSC Adv.* **5**, 36987–36992.
- Lynch, D. E. (2004). *Acta Cryst.* **E60**, 1715–1716.
- Ma, W.-X., Qian, B.-H. & Ge, H.-Y. (2012). *Asian J. Chem.* **24**, 3504–3506.
- Ouyang, L., Huang, Y., Zhao, Y., He, G., Xie, Y., Liu, J., He, J., Liu, B. & Wei, Y. (2012). *Bioorg. Med. Chem. Lett.* **22**, 3044–3049.
- Potashman, M. H., Bready, J., Coxon, A., DeMelfi, T. M., DiPietro, L., Doerr, N., Elbaum, D., Estrada, J., Gallant, P., Germain, J., Gu, Y., Harmange, J. C., Kaufman, S. A., Kendall, R., Kim, J. L., Kumar, G. N., Long, A. M., Neervannan, S., Patel, V. F., Polverino, A., Rose, P., van der Plas, S., Whittington, D., Zanon, R. & Zhao, H. (2007). *J. Med. Chem.* **50**, 4351–4373.
- Razzoqova, S., Torambetov, B., Amanova, M., Kadirova, S., Ibragimov, A. & Ashurov, J. (2022). *Acta Cryst.* **E78**, 1277–1283.
- Rigaku OD (2020). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
- Ruzmetov, A., Ibragimov, A., Ashurov, J., Boltaeva, Z., Ibragimov, B. & Usmanov, S. (2022). *Acta Cryst.* **E78**, 660–664.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Yan, X., Fan, Y., Bi, C., Zhang, X., Zuo, J., Zhang, P. & Zhang, Z. (2014). *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **44**, 603–610.
- Zhao, X.-J., Li, J., Ding, B., Wang, X.-G. & Yang, E.-C. (2007). *Inorg. Chem. Commun.* **10**, 605–609.

## supporting information

*Acta Cryst.* (2023). E79, 862-866 [https://doi.org/10.1107/S2056989023007399]

## Synthesis, structure and Hirshfeld surface analysis of a coordination compound of cadmium acetate with 2-aminobenzoxazole

**Surayyo Razzoqova, Aziz Ibragimov, Batirbay Torambetov, Shakhnoza Kadirova, Tamas Holczbauer, Jamshid Ashurov and Bakhtiyar Ibragimov**

### Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2020); cell refinement: *CrysAlis PRO* (Rigaku OD, 2020); data reduction: *CrysAlis PRO* (Rigaku OD, 2020); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

### Bis(2-aminobenzoxazole- $\kappa^3$ )bis(acetato- $\kappa^2$ O,O')cadmium(II)

#### Crystal data

[Cd(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>]

$M_r = 498.76$

Monoclinic, *C2/c*

$a = 25.0497$  (3) Å

$b = 9.8428$  (1) Å

$c = 16.7577$  (2) Å

$\beta = 94.534$  (1)°

$V = 4118.83$  (8) Å<sup>3</sup>

$Z = 8$

$F(000) = 2000$

$D_x = 1.609$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 6254 reflections

$\theta = 3.5\text{--}70.8^\circ$

$\mu = 8.87$  mm<sup>-1</sup>

$T = 293$  K

Block, colourless

0.17 × 0.14 × 0.12 mm

#### Data collection

Rigaku XtaLAB Synergy  
diffractometer with a HyPix3000 detector

Radiation source: micro-focus sealed X-ray  
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(*CrysAlis PRO*; Rigaku OD, 2020)

$T_{\min} = 0.793$ ,  $T_{\max} = 1.000$

11316 measured reflections

3971 independent reflections

3434 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 71.2^\circ$ ,  $\theta_{\min} = 3.5^\circ$

$h = -30 \rightarrow 30$

$k = -12 \rightarrow 11$

$l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.078$

$S = 1.05$

3971 reflections

265 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.4924P]$

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

$(\Delta/\sigma)_{\max} = 0.002$

$$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.59 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL2016  
(Sheldrick, 2015b),  
 $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
Extinction coefficient: 0.000082 (13)

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	0.62410 (2)	0.36216 (2)	0.51980 (2)	0.05241 (10)
O1	0.73371 (8)	0.0598 (2)	0.64662 (14)	0.0683 (6)
O2	0.47350 (9)	0.2945 (3)	0.63959 (16)	0.0769 (7)
N1	0.67802 (9)	0.2252 (2)	0.60113 (14)	0.0537 (6)
O6	0.59362 (9)	0.3834 (3)	0.38471 (16)	0.0791 (7)
O4	0.68374 (9)	0.5447 (2)	0.52681 (17)	0.0789 (7)
N3	0.54967 (9)	0.2942 (3)	0.57764 (16)	0.0572 (6)
N2	0.72228 (10)	0.0877 (3)	0.51189 (17)	0.0642 (7)
H2A	0.707645	0.126500	0.469675	0.077*
H2B	0.744585	0.022233	0.507514	0.077*
O3	0.60454 (10)	0.5914 (3)	0.5565 (2)	0.0996 (10)
O5	0.66139 (11)	0.2548 (3)	0.40824 (15)	0.0948 (9)
C15	0.65104 (13)	0.6280 (3)	0.5472 (2)	0.0578 (7)
C1	0.71057 (11)	0.1285 (3)	0.5831 (2)	0.0528 (7)
N4	0.50825 (10)	0.5006 (3)	0.6106 (2)	0.0821 (9)
H4A	0.532476	0.550805	0.591987	0.099*
H4B	0.481505	0.537324	0.631454	0.099*
C17	0.62821 (12)	0.3087 (3)	0.36070 (19)	0.0600 (7)
C7	0.67858 (11)	0.2219 (3)	0.68508 (18)	0.0574 (7)
C9	0.53519 (12)	0.1592 (3)	0.5921 (2)	0.0613 (8)
C2	0.71305 (13)	0.1203 (4)	0.7127 (2)	0.0678 (9)
C8	0.51215 (13)	0.3665 (3)	0.6070 (2)	0.0636 (8)
C14	0.48834 (14)	0.1591 (4)	0.6302 (2)	0.0718 (9)
C16	0.66689 (16)	0.7730 (3)	0.5600 (2)	0.0842 (11)
H16A	0.704972	0.781517	0.558471	0.126*
H16B	0.656663	0.802761	0.611189	0.126*
H16C	0.649232	0.828000	0.518610	0.126*
C6	0.65278 (14)	0.3000 (4)	0.7383 (2)	0.0777 (10)
H6	0.629353	0.369251	0.721214	0.093*
C10	0.55866 (14)	0.0371 (4)	0.5755 (3)	0.0810 (11)
H10	0.590396	0.033844	0.550275	0.097*
C11	0.53352 (16)	-0.0804 (4)	0.5975 (3)	0.0931 (13)
H11	0.548254	-0.164167	0.585847	0.112*
C13	0.46251 (17)	0.0455 (5)	0.6528 (3)	0.0977 (13)
H13	0.430576	0.049423	0.677524	0.117*

C18	0.63098 (17)	0.2827 (5)	0.2729 (2)	0.0957 (13)
H18A	0.647214	0.195777	0.265397	0.144*
H18B	0.652023	0.352341	0.250357	0.144*
H18C	0.595468	0.283398	0.246754	0.144*
C3	0.72412 (17)	0.0889 (6)	0.7912 (3)	0.1035 (15)
H3	0.747541	0.019434	0.807937	0.124*
C5	0.66360 (17)	0.2699 (6)	0.8190 (2)	0.1001 (14)
H5	0.646981	0.320491	0.856801	0.120*
C12	0.48703 (18)	-0.0764 (5)	0.6365 (3)	0.1025 (15)
H12	0.471821	-0.157333	0.652136	0.123*
C4	0.6980 (2)	0.1678 (6)	0.8445 (3)	0.1133 (18)
H4	0.704006	0.151002	0.899045	0.136*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cd1	0.04847 (14)	0.04693 (14)	0.06296 (15)	0.00942 (8)	0.01144 (9)	-0.00134 (9)
O1	0.0585 (12)	0.0626 (14)	0.0837 (16)	0.0146 (10)	0.0057 (11)	0.0158 (12)
O2	0.0578 (13)	0.0723 (16)	0.1046 (19)	0.0008 (11)	0.0321 (12)	0.0026 (14)
N1	0.0502 (12)	0.0513 (14)	0.0604 (14)	0.0101 (11)	0.0094 (10)	0.0017 (12)
O6	0.0599 (13)	0.0937 (18)	0.0844 (17)	0.0208 (12)	0.0096 (12)	-0.0112 (14)
O4	0.0608 (13)	0.0584 (13)	0.119 (2)	0.0111 (11)	0.0163 (12)	-0.0188 (14)
N3	0.0455 (12)	0.0487 (13)	0.0788 (17)	0.0061 (11)	0.0131 (11)	0.0024 (13)
N2	0.0586 (14)	0.0593 (15)	0.0760 (18)	0.0178 (13)	0.0130 (12)	-0.0037 (14)
O3	0.0752 (16)	0.0610 (14)	0.170 (3)	-0.0079 (13)	0.0582 (17)	-0.0258 (17)
O5	0.1068 (19)	0.105 (2)	0.0710 (16)	0.0516 (17)	-0.0018 (14)	-0.0074 (15)
C15	0.0681 (19)	0.0468 (16)	0.0608 (18)	0.0067 (14)	0.0200 (15)	0.0019 (13)
C1	0.0418 (14)	0.0471 (15)	0.0697 (19)	0.0016 (12)	0.0069 (13)	0.0049 (14)
N4	0.0599 (16)	0.0578 (16)	0.133 (3)	0.0145 (13)	0.0356 (16)	-0.0006 (18)
C17	0.0547 (16)	0.0640 (19)	0.0621 (18)	0.0020 (15)	0.0092 (14)	0.0002 (16)
C7	0.0483 (15)	0.0618 (18)	0.0629 (18)	-0.0014 (14)	0.0087 (13)	0.0031 (16)
C9	0.0523 (17)	0.0525 (18)	0.079 (2)	0.0005 (13)	0.0049 (15)	0.0044 (16)
C2	0.0567 (18)	0.077 (2)	0.070 (2)	0.0027 (16)	0.0054 (15)	0.0133 (18)
C8	0.0509 (17)	0.062 (2)	0.080 (2)	0.0074 (14)	0.0151 (15)	0.0054 (16)
C14	0.0602 (19)	0.066 (2)	0.090 (3)	0.0000 (16)	0.0145 (17)	0.0017 (19)
C16	0.095 (3)	0.052 (2)	0.111 (3)	-0.0050 (18)	0.042 (2)	-0.005 (2)
C6	0.071 (2)	0.091 (3)	0.072 (2)	0.009 (2)	0.0092 (17)	-0.007 (2)
C10	0.062 (2)	0.058 (2)	0.122 (3)	0.0036 (16)	0.0091 (19)	-0.001 (2)
C11	0.079 (3)	0.055 (2)	0.144 (4)	-0.0059 (19)	0.005 (3)	0.004 (2)
C13	0.077 (2)	0.092 (3)	0.127 (4)	-0.021 (2)	0.027 (2)	0.011 (3)
C18	0.105 (3)	0.122 (4)	0.061 (2)	-0.002 (3)	0.016 (2)	-0.006 (2)
C3	0.089 (3)	0.132 (4)	0.088 (3)	0.018 (3)	0.001 (2)	0.037 (3)
C5	0.092 (3)	0.143 (4)	0.067 (2)	0.007 (3)	0.013 (2)	-0.010 (3)
C12	0.091 (3)	0.068 (3)	0.149 (5)	-0.020 (2)	0.016 (3)	0.016 (3)
C4	0.094 (3)	0.185 (6)	0.062 (2)	0.012 (3)	0.008 (2)	0.017 (3)



*Geometric parameters (Å, °)*

Cd1—N1	2.282 (2)	C17—C18	1.501 (5)
Cd1—O6	2.341 (3)	C7—C2	1.377 (4)
Cd1—O4	2.333 (2)	C7—C6	1.377 (4)
Cd1—N3	2.269 (2)	C9—C14	1.380 (5)
Cd1—O3	2.399 (3)	C9—C10	1.376 (5)
Cd1—O5	2.400 (2)	C2—C3	1.358 (5)
Cd1—C15	2.732 (3)	C14—C13	1.360 (5)
Cd1—C17	2.727 (3)	C16—H16A	0.9600
O1—C1	1.353 (4)	C16—H16B	0.9600
O1—C2	1.392 (4)	C16—H16C	0.9600
O2—C8	1.349 (4)	C6—H6	0.9300
O2—C14	1.396 (4)	C6—C5	1.389 (5)
N1—C1	1.304 (4)	C10—H10	0.9300
N1—C7	1.406 (4)	C10—C11	1.381 (5)
O6—C17	1.228 (4)	C11—H11	0.9300
O4—C15	1.227 (4)	C11—C12	1.380 (6)
N3—C9	1.404 (4)	C13—H13	0.9300
N3—C8	1.306 (4)	C13—C12	1.385 (6)
N2—H2A	0.8600	C18—H18A	0.9600
N2—H2B	0.8600	C18—H18B	0.9600
N2—C1	1.314 (4)	C18—H18C	0.9600
O3—C15	1.241 (4)	C3—H3	0.9300
O5—C17	1.226 (4)	C3—C4	1.386 (7)
C15—C16	1.492 (4)	C5—H5	0.9300
N4—H4A	0.8600	C5—C4	1.369 (7)
N4—H4B	0.8600	C12—H12	0.9300
N4—C8	1.325 (4)	C4—H4	0.9300
N1—Cd1—O6	139.71 (8)	O6—C17—C18	120.8 (3)
N1—Cd1—O4	94.49 (8)	O5—C17—Cd1	61.59 (18)
N1—Cd1—O3	121.70 (11)	O5—C17—O6	120.4 (3)
N1—Cd1—O5	87.52 (8)	O5—C17—C18	118.8 (3)
N1—Cd1—C15	109.79 (9)	C18—C17—Cd1	178.6 (3)
N1—Cd1—C17	113.80 (9)	C2—C7—N1	108.0 (3)
O6—Cd1—O3	96.12 (10)	C6—C7—N1	131.9 (3)
O6—Cd1—O5	53.35 (8)	C6—C7—C2	120.1 (3)
O6—Cd1—C15	97.73 (9)	C14—C9—N3	108.8 (3)
O6—Cd1—C17	26.66 (8)	C10—C9—N3	132.2 (3)
O4—Cd1—O6	97.96 (10)	C10—C9—C14	119.1 (3)
O4—Cd1—O3	53.57 (8)	C7—C2—O1	107.8 (3)
O4—Cd1—O5	95.22 (10)	C3—C2—O1	127.8 (3)
O4—Cd1—C15	26.57 (8)	C3—C2—C7	124.4 (4)
O4—Cd1—C17	97.13 (10)	N3—C8—O2	115.3 (3)
N3—Cd1—N1	92.19 (9)	N3—C8—N4	128.1 (3)
N3—Cd1—O6	102.90 (9)	N4—C8—O2	116.5 (3)
N3—Cd1—O4	138.59 (9)	C9—C14—O2	107.2 (3)

N3—Cd1—O3	88.65 (9)	C13—C14—O2	128.1 (4)
N3—Cd1—O5	125.90 (11)	C13—C14—C9	124.8 (4)
N3—Cd1—C15	114.27 (9)	C15—C16—H16A	109.5
N3—Cd1—C17	117.16 (9)	C15—C16—H16B	109.5
O3—Cd1—O5	135.54 (11)	C15—C16—H16C	109.5
O3—Cd1—C15	27.00 (9)	H16A—C16—H16B	109.5
O3—Cd1—C17	117.15 (11)	H16A—C16—H16C	109.5
O5—Cd1—C15	116.52 (11)	H16B—C16—H16C	109.5
O5—Cd1—C17	26.69 (8)	C7—C6—H6	121.8
C17—Cd1—C15	108.73 (10)	C7—C6—C5	116.5 (4)
C1—O1—C2	104.4 (2)	C5—C6—H6	121.8
C8—O2—C14	104.5 (2)	C9—C10—H10	121.1
C1—N1—Cd1	130.1 (2)	C9—C10—C11	117.8 (4)
C1—N1—C7	105.0 (2)	C11—C10—H10	121.1
C7—N1—Cd1	124.74 (18)	C10—C11—H11	119.3
C17—O6—Cd1	94.5 (2)	C12—C11—C10	121.4 (4)
C15—O4—Cd1	95.2 (2)	C12—C11—H11	119.3
C9—N3—Cd1	125.8 (2)	C14—C13—H13	122.3
C8—N3—Cd1	129.8 (2)	C14—C13—C12	115.4 (4)
C8—N3—C9	104.3 (3)	C12—C13—H13	122.3
H2A—N2—H2B	120.0	C17—C18—H18A	109.5
C1—N2—H2A	120.0	C17—C18—H18B	109.5
C1—N2—H2B	120.0	C17—C18—H18C	109.5
C15—O3—Cd1	91.61 (19)	H18A—C18—H18B	109.5
C17—O5—Cd1	91.7 (2)	H18A—C18—H18C	109.5
O4—C15—Cd1	58.28 (16)	H18B—C18—H18C	109.5
O4—C15—O3	119.7 (3)	C2—C3—H3	122.4
O4—C15—C16	120.2 (3)	C2—C3—C4	115.2 (4)
O3—C15—Cd1	61.39 (17)	C4—C3—H3	122.4
O3—C15—C16	120.1 (3)	C6—C5—H5	119.0
C16—C15—Cd1	178.1 (3)	C4—C5—C6	122.0 (4)
N1—C1—O1	114.8 (3)	C4—C5—H5	119.0
N1—C1—N2	128.5 (3)	C11—C12—C13	121.5 (4)
N2—C1—O1	116.7 (3)	C11—C12—H12	119.2
H4A—N4—H4B	120.0	C13—C12—H12	119.2
C8—N4—H4A	120.0	C3—C4—H4	119.1
C8—N4—H4B	120.0	C5—C4—C3	121.8 (4)
O6—C17—Cd1	58.82 (18)	C5—C4—H4	119.1
Cd1—N1—C1—O1	-175.14 (18)	C1—N1—C7—C6	179.2 (3)
Cd1—N1—C1—N2	2.7 (5)	C7—N1—C1—O1	-0.4 (3)
Cd1—N1—C7—C2	175.5 (2)	C7—N1—C1—N2	177.4 (3)
Cd1—N1—C7—C6	-5.7 (5)	C7—C2—C3—C4	-0.2 (7)
Cd1—O6—C17—O5	1.0 (4)	C7—C6—C5—C4	-0.1 (7)
Cd1—O6—C17—C18	-178.5 (3)	C9—N3—C8—O2	-0.4 (4)
Cd1—O4—C15—O3	0.7 (4)	C9—N3—C8—N4	177.7 (4)
Cd1—O4—C15—C16	-178.7 (3)	C9—C14—C13—C12	1.3 (7)
Cd1—N3—C9—C14	178.2 (2)	C9—C10—C11—C12	-1.4 (7)

Cd1—N3—C9—C10	-1.8 (6)	C2—O1—C1—N1	0.2 (3)
Cd1—N3—C8—O2	-178.2 (2)	C2—O1—C1—N2	-177.9 (3)
Cd1—N3—C8—N4	-0.2 (6)	C2—C7—C6—C5	-0.1 (5)
Cd1—O3—C15—O4	-0.6 (4)	C2—C3—C4—C5	0.0 (8)
Cd1—O3—C15—C16	178.7 (3)	C8—O2—C14—C9	-0.2 (4)
Cd1—O5—C17—O6	-1.0 (4)	C8—O2—C14—C13	-179.6 (4)
Cd1—O5—C17—C18	178.5 (3)	C8—N3—C9—C14	0.2 (4)
O1—C2—C3—C4	179.2 (4)	C8—N3—C9—C10	-179.8 (4)
O2—C14—C13—C12	-179.4 (4)	C14—O2—C8—N3	0.4 (4)
N1—C7—C2—O1	-0.3 (4)	C14—O2—C8—N4	-177.9 (4)
N1—C7—C2—C3	179.1 (4)	C14—C9—C10—C11	0.5 (6)
N1—C7—C6—C5	-178.7 (4)	C14—C13—C12—C11	-2.1 (8)
N3—C9—C14—O2	0.0 (4)	C6—C7—C2—O1	-179.2 (3)
N3—C9—C14—C13	179.5 (4)	C6—C7—C2—C3	0.2 (6)
N3—C9—C10—C11	-179.5 (4)	C6—C5—C4—C3	0.2 (8)
C1—O1—C2—C7	0.1 (3)	C10—C9—C14—O2	180.0 (3)
C1—O1—C2—C3	-179.4 (4)	C10—C9—C14—C13	-0.5 (6)
C1—N1—C7—C2	0.5 (3)	C10—C11—C12—C13	2.2 (8)

*Hydrogen-bond geometry (Å, °)*

<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>
N2—H2 <i>A</i> $\cdots$ O5	0.86	1.95	2.762 (4)	157
N2—H2 <i>B</i> $\cdots$ O4 <sup>i</sup>	0.86	2.04	2.811 (3)	149
N4—H4 <i>A</i> $\cdots$ O3	0.86	1.99	2.790 (4)	155
N4—H4 <i>B</i> $\cdots$ O6 <sup>ii</sup>	0.86	2.04	2.803 (3)	148

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