



Crystal structure of the salt bis(triethanolamine- κ^4N,O,O',O'')cadmium bis[2-(2-oxo-2,3-dihydro-1,3-benzothiazol-3-yl)acetate]

Jamshid Mengnorovich Ashurov*

Institute of Bioorganic Chemistry, Academy of Sciences of Uzbekistan, M. Ulugbek Str. 83, Tashkent 700125, Uzbekistan.

*Correspondence e-mail: atom.uz@mail.ru

Received 7 March 2016

Accepted 16 March 2016

Edited by M. Weil, Vienna University of
Technology, Austria

Keywords: crystal structure; cadmium(II) complex; triethanolamine; 2-(2-oxo-2,3-dihydro-1,3-benzothiazolin-3-yl)acetate; hydrogen bonding.

CCDC reference: 1468939

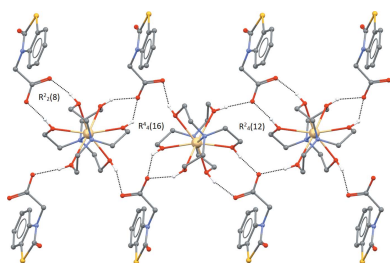
Supporting information: this article has supporting information at journals.iucr.org/e

The reaction of 2-(2-oxo-2,3-dihydro-1,3-benzothiazol-3-yl)acetic acid (NBTA) and triethanolamine (TEA) with $\text{Cd}(\text{CH}_3\text{OO})_2$ resulted in the formation of the title salt, $[\text{Cd}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_9\text{H}_6\text{NO}_3\text{S})_2$. In its crystal structure, the complex cation $[\text{Cd}(\text{TEA})_2]^{2+}$ and two independent NBTA[−] units with essentially similar geometries and conformations are present. In the complex cation, each TEA molecule behaves as an N,O,O',O'' -tetradentate ligand, giving rise to an eight-coordinate Cd^{II} ion with a bicapped trigonal-prismatic configuration. All ethanol groups of each TEA molecule form three five-membered chelate rings around the Cd^{II} ion. The Cd—O and Cd—N distances are in the ranges 2.392 (2)–2.478 (2) and 2.465 (2)–2.475 (3) Å, respectively. O—H \cdots O hydrogen bonds between the TEA hydroxy groups and carboxylate O atoms connect cationic and anionic moieties into chains parallel to [110]. Each NBTA[−] anion is additionally linked to a symmetry-related anion through π – π stacking interactions between the benzene and thiazoline rings [minimum centroid-to-centroid separation = 3.604 (2) Å]. Together with additional C—H \cdots O interactions, these establish a double-layer polymeric network parallel to (001).

1. Chemical context

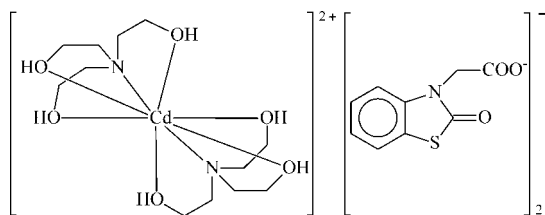
Triethanolamine (TEA) is a potential ligand for the incorporation of metals into metal-ion-containing supramolecular frameworks, and many compounds constructed from TEA have been reported in the last decade (Haukka *et al.*, 2005; Topcu *et al.*, 2001; Ucar *et al.*, 2004). TEA is also used as a corrosion inhibitor in metal-cutting fluids, as a curing agent for epoxy and rubber polymers, adhesives, antistatic agents or as a pharmaceutical intermediate and an ointment emulsifier. However, TEA has no specific physiological effects (Beyer *et al.*, 1983; Knaak *et al.*, 1997), with exception of its low antibacterial action. Benzothiazoles are bicyclic ring systems and their derivatives have been studied and found to have various chemical reactivities and biological activities. For example, benzothiazole is a precursor for rubber accelerators, a component of cyanine dyes, is used as a slimicide in the paper and pulp industry, or in the production of certain fungicides, herbicides, pharmaceuticals (Bellavia *et al.*, 2000; Seo *et al.*, 2000), anti-allergic (Musser *et al.*, 1984), antitumor (Yoshida *et al.*, 2005) or anti-diabetic (Pattan *et al.*, 2005) substances.

The interaction of metal ions with TEA results in the formation of complexes in which TEA demonstrates monodentate (Kumar *et al.*, 2014), bidentate (Kaptein *et al.*, 1997; Long *et al.*, 2004), tridentate (Gao *et al.*, 2004; Ucar *et al.*, 2004; Krabbes *et al.*, 1999; Haukka *et al.*, 2005; Yeşilel *et al.*, 2004; Mirskova *et al.*, 2013) or tetradentate binding modes (Zaitsev



OPEN ACCESS

et al., 2014; Kazak *et al.*, 2003; Yilmaz *et al.*, 2004; Rickard *et al.*, 1999; Maestri & Brown, 2004; Kovbasyuk *et al.*, 2001; Tudor *et al.*, 2001). In some complexes, TEA has bridging properties (Langley *et al.*, 2011; Atria *et al.*, 2015; Wittick *et al.*, 2006; Sharma *et al.*, 2014; Yang *et al.*, 2014; Funes *et al.*, 2014). In addition, there are metal complexes known in which TEA molecules are uncoordinating (Ilyukhin *et al.*, 2013; Manos *et al.*, 2012). As an ancillary ligand, TEA may enhance the physiological action of bioactive substances in mixed-ligand metal complexes (Boulsourani *et al.*, 2011). We have reported the synthesis of mixed-ligand complexes of Zn, Cd and Cu with TEA and *p*-nitrobenzoic acid (NBA) and determined the structures of $[\text{Cu}_2(\text{NBA})_2\text{TEA}](\text{NBA})(5\text{H}_2\text{O})$, $[\text{Zn}(\text{NBA})_2\text{TEA}]$ and $[\text{Cd}(\text{NBA})_2\text{TEA}]$ (Ashurov *et al.*, 2015). The cobalt(II) complex $[\text{Co}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_9\text{H}_6\text{NO}_3\text{S})_2$, obtained by the reaction of NBTA and TEA with $\text{Co}(\text{NO}_3)_2$, has been reported (Ashurov *et al.*, 2016). Here, the synthesis and structure of the related title compound, $[\text{Cd}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_9\text{H}_6\text{NO}_3\text{S})_2$, (I), is described.



2. Structural commentary

The structure of the molecular entities of (I) is shown in Fig. 1; these consist of a complex cation and two independent NBTA^- anions. In the cationic complex, the Cd^{II} ion is ligated by two neutral TEA molecules, which act as *N,O,O',O''*-tetradentate ligands, resulting in a bicapped trigonal-prismatic coordination polyhedron of the type CdN_2O_6 . In the complex, $\text{Cd}-\text{O}$ and $\text{Cd}-\text{N}$ distances are in the range 2.392 (2)–2.478 (2) and 2.465 (2)–2.475 (3) Å, respectively. The $\text{N}-\text{Cd}-\text{O}$ bond angles range from 68.58 (8) to 122.59 (10)° and the $\text{O}-\text{Cd}-\text{O}$ angles are in an interval of 72.54 (9) to 162.13 (11)°. Both thiazoline rings (C1/C6/N1/C7/S1 and C1A/C6A/N1A/C7A/S1A) and bicyclic benzothiazole units (N1/S1/C1–C7 and (N1A/S1A/C1A–C7A) are close to planar, the

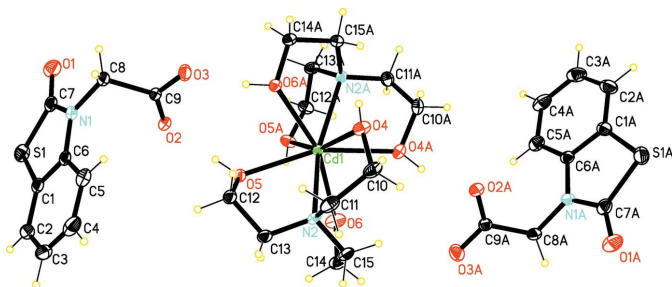


Figure 1

The structures of the molecular moieties in the title salt. Displacement ellipsoids are drawn at the 30% probability level.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4}\cdots\text{O3}^{\text{i}}$	0.87 (3)	1.82 (2)	2.676 (4)	170 (4)
$\text{O4A}-\text{H4A}\cdots\text{O2A}$	0.87 (3)	1.91 (3)	2.748 (4)	161 (3)
$\text{O5}-\text{H5}\cdots\text{O2}$	0.87 (3)	1.81 (3)	2.661 (3)	166 (3)
$\text{O5A}-\text{H5A}\cdots\text{O3A}^{\text{ii}}$	0.87 (2)	1.83 (3)	2.640 (4)	154 (2)
$\text{O6}-\text{H6}\cdots\text{O3A}^{\text{ii}}$	0.85 (2)	2.04 (2)	2.749 (5)	141 (3)
$\text{O6A}-\text{H6A}\cdots\text{O3}$	0.86 (2)	1.79 (2)	2.640 (4)	172 (2)
$\text{C11A}-\text{H11D}\cdots\text{O1}^{\text{iii}}$	0.97	2.47	3.282 (6)	142

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

largest deviations from the least-squares planes being 0.002 (2), 0.004 (2) and 0.008 (3), 0.005 (3) Å, respectively. The dihedral angles between the plane of the carboxylate group and the attached benzothiazole ring system are 77.895 (3) and 71.408 (3)° in the two anions.

3. Supramolecular features

In the crystal structure of (I), classical intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds are observed (Table 1) which link the complex cations and NBTA^- anions into a chain structure extending parallel to [110], whereby each cation is surrounded by four NBTA^- anions. The H atoms of all hydroxyl groups of the TEA ligands form a hydrogen bond with a carboxylate O atom of the NBTA^- ions. In addition, there is weak hydrogen bond between one $-\text{CH}_2$ group and the O1 atom of the NBTA^- anion, with a $\text{C}\cdots\text{O}$ distance of 3.282 (6) Å (Table 1). The above-mentioned hydrogen bonds give rise to $R_2^2(8)$, $R_4^4(12)$ and $R_4^4(16)$ graph-set motifs (Fig. 2). The NBTA^- anion layers are not linked by hydrogen bonds, but there are mutual $\pi-\pi$ stacking interactions between benzene rings (centroid Cg1) and thiazoline rings (centroid Cg2) of adjacent inversion-related molecules [$\text{Cg1}\cdots\text{Cg2}$ ($2-x, -y, 1-z$) = 3.604 (2) Å] (Fig. 3). Together, these supramolecular interactions generate a double-layer polymeric network parallel to (001).

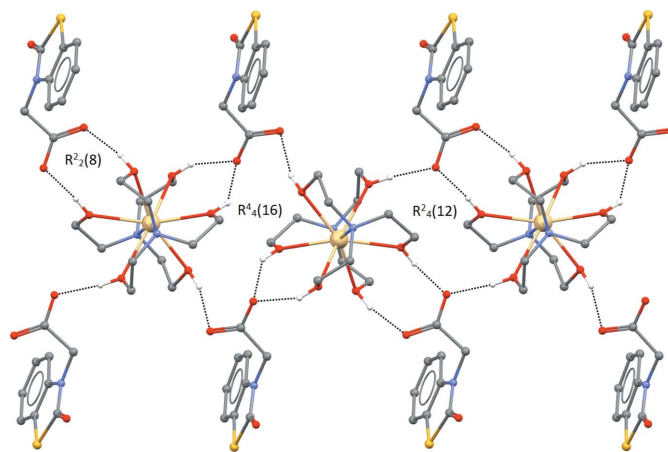


Figure 2

Part of the crystal structure with hydrogen bonds shown as dashed lines. For clarity, H atoms not involved in hydrogen bonding are omitted.

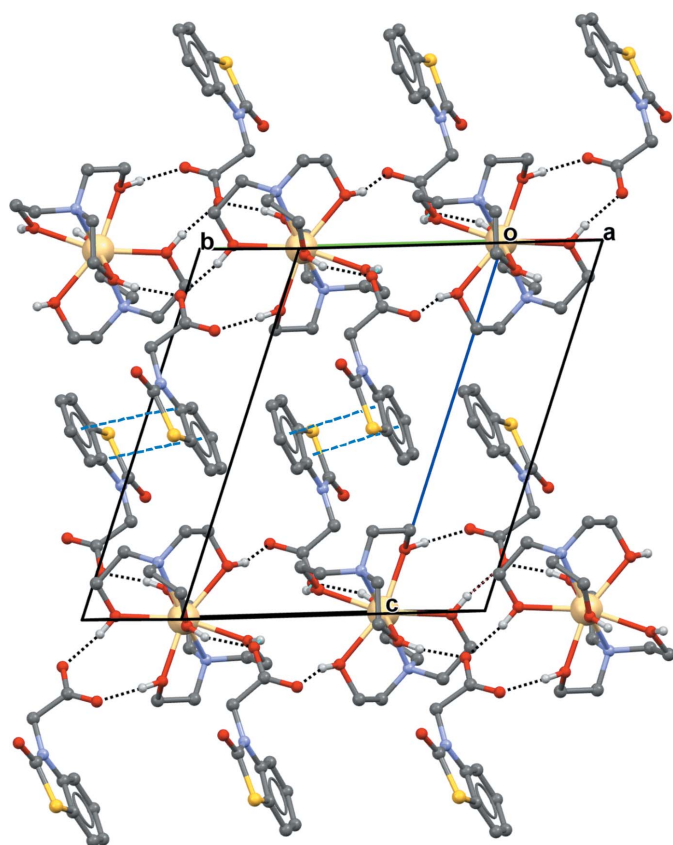


Figure 3
Packing of structural units in (I). Hydrogen bonds are indicated as black dashed lines and π - π stacking interactions as blue dashed lines.

4. Database survey

A survey of the Cambridge Structural Database (CSD; Groom & Allen, 2014) showed that coordination complexes of TEA with many metals including those of the *s*-, *d*-, *p*-, and *f*-block elements have been reported. Structures containing the $[\text{Cd}(\text{TEA})_2]^{2+}$ cation are deposited in the CSD with reference codes EYIPAD, MEVQIN and YOVBUI.

5. Synthesis and crystallization

To an aqueous solution (2.5 ml) of $\text{Cd}(\text{CH}_3\text{OO})_2$ (0.103 g, 0.5 mmol) was slowly added an ethanolic solution (5 ml) containing TEA (132 μl) and NBT (0.209 g, 1 mmol) under constant stirring. A bright-yellow crystalline product was obtained at room temperature by solvent evaporation after four weeks. Yield: 75%; calculated for $\text{C}_{30}\text{H}_{42}\text{CdN}_4\text{O}_{12}\text{S}_2$: C, 43.56; H, 5.12; N, 6.77, found: C, 43.61; H, 5.15; N, 6.69

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl H atoms of the TEA ligands were located in a difference-Fourier map and were refined with soft O—H distance restraints of 0.87 Å. The C-bound hydrogen atoms were placed in calculated positions

Table 2
Experimental details.

Crystal data	
Chemical formula	$[\text{Cd}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_9\text{H}_6\text{NO}_3\text{S})_2$
M_r	827.20
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	10.7061 (5), 12.2157 (5), 14.6159 (8)
α, β, γ (°)	65.520 (5), 79.600 (4), 82.417 (4)
V (Å ³)	1707.59 (14)
Z	2
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	6.85
Crystal size (mm)	0.6 × 0.3 × 0.2
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Ruby
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2009)
$T_{\text{min}}, T_{\text{max}}$	0.127, 0.254
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14752, 7001, 6533
R_{int}	0.047
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.629
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.040, 0.107, 1.06
No. of reflections	7001
No. of parameters	461
No. of restraints	18
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	1.20, -0.67

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2009), *SHELXS97*, *SHELXL97* and *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006).

and refined as riding atoms with C—H = 0.93 and 0.97 Å for aromatic and methylene hydrogen atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Acknowledgements

This work was supported by a Grant for Fundamental Research from the Center of Science and Technology, Uzbekistan (No. FPMI T.2–16).

References

- Ashurov, J. M., Ibragimov, A. B. & Ibragimov, B. T. (2015). *Polyhedron*, **102**, 441–446.
- Ashurov, J. M., Obidova, N. J., Abdireymov, H. B. & Ibragimov, B. T. (2016). *Acta Cryst.* **E72**, 420–423.
- Atria, A. M., Parada, J., Garland, M. T. & Baggio, R. (2015). *J. Chil. Chem. Soc.* **60**, 3059–3062.
- Bellavia, V., Natangelo, M., Fanelli, R. & Rotilio, D. (2000). *J. Agric. Food Chem.* **48**, 1239–1242.
- Beyer, K. H., Bergfeld, W. F., Berndt, W. O., Boutwell, R. K., Carlton, W. W., Hoffmann, D. K. & Schroeder, A. L. (1983). *J. Am. Coll. Toxicol.* **2**, 183–235.
- Boulsourani, Z., Geromichalos, G. D., Repana, K., Yiannaki, E., Psycharis, V., Raptopoulou, C. P., Hadjipavlou-Litina, D., Pontiki, E. & Dendrinou-Samara, C. (2011). *J. Inorg. Biochem.* **105**, 839–849.
- Funes, A. V., Carrella, L., Rentschler, E. & Alborés, P. (2014). *Dalton Trans.* **43**, 2361–2364.

- Gao, S., Liu, J.-W., Huo, L.-H. & Ng, S. W. (2004). *Acta Cryst.* **E60**, m462–m464.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
- Haukka, M., Kirillov, A. M., Kopylovich, M. N. & Pombeiro, A. J. L. (2005). *Acta Cryst.* **E61**, m2746–m2748.
- Ilyukhin, A. B., Koroteev, P. S., Kiskin, M. A., Dobrokhotova, Z. V. & Novotortsev, V. M. (2013). *J. Mol. Struct.* **1033**, 187–199.
- Kaptein, G. M., Baesjou, P. J., Alsters, P. L., Grove, D. M., Koten, G. V., Smeets, W. J. J., Kooijman, H. & Spek, A. L. (1997). *Chem. Ber. Recl.* **130**, 35–44.
- Kazak, C., Hamamci, S., Topcu, Y. & Yilmaz, V. T. (2003). *J. Mol. Struct.* **657**, 351–356.
- Knaak, J. B., Leung, H. W., Stott, W. T., Busch, J. & Bilsky, J. (1997). *Rev. Environ. Contam. Toxicol.* **149**, 1–86.
- Kovbasyuk, L. A., Vassilyeva, O. Yu., Kokozay, V. N., Chun, H., Bernal, I., Reedijk, J., Albada, G. V. & Skelton, B. W. (2001). *Cryst. Eng.* **4**, 201–213.
- Krabbes, I., Seichter, W., Breuning, T., Otschik, P. & Gloe, K. (1999). *Z. Anorg. Allg. Chem.* **625**, 1562–1565.
- Kumar, R., Obrai, S., Kaur, A., Hundal, M. S., Meehnian, H. & Jana, A. K. (2014). *New J. Chem.* **38**, 1186–1198.
- Langley, S. K., Ungur, L., Chilton, N. F., Moubaraki, B., Chibotaru, L. F. & Murray, K. S. (2011). *Chem. Eur. J.* **17**, 9209–9218.
- Long, D.-L., Abbas, H., Kögerler, P. & Cronin, L. (2004). *J. Am. Chem. Soc.* **126**, 13880–13881.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Maestri, A. G. & Brown, S. N. (2004). *Inorg. Chem.* **43**, 6995–7004.
- Manos, M. J., Moushi, E. E., Papaefstathiou, G. S. & Tasiopoulos, A. J. (2012). *Cryst. Growth Des.* **12**, 5471–5480.
- Mirskova, A. N., Adamovich, S. N., Mirskov, R. G. & Schilde, U. (2013). *Chem. Cent. J.* **7**, 34–38.
- Musser, J. H., Brown, R. E., Loev, B., Bailey, K., Jones, H., Kahen, R., Huang, F. C., Khandwala, A., Leibowitz, M., Sonnino-Goldman, P. & Donigi-Ruzza, D. (1984). *J. Med. Chem.* **27**, 121–125.
- Oxford Diffraction (2009). *CrysAlis PRO CCD*. Oxford Diffraction Ltd, Yarnton, England.
- Pattan, S. R., Suresh, C., Pujar, V. D., Reddy, V. V. K., Rasal, V. P. & Kotti, B. C. (2005). *Indian J. Chem.* **4B**, 2404–2408.
- Rickard, C. E. F., Roper, W. R., Woodman, T. J. & Wright, L. J. (1999). *Chem. Commun.* pp. 837–838.
- Seo, K. W., Park, M., Kim, J. G., Kim, T. W. & Kim, H. J. (2000). *J. Appl. Toxicol.* **20**, 427–30.
- Sharma, R. P., Saini, A., Venugopalan, P., Ferretti, V., Spizzo, F., Angeli, C. & Calzado, C. J. (2014). *New J. Chem.* **38**, 574–583.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Topcu, Y., Andac, O., Yilmaz, V. T. & Harrison, W. T. A. (2001). *Acta Cryst.* **E57**, m82–m84.
- Tudor, V., Kravtsov, V., Julve, M., Lloret, F., Simonov, Y. A., Lipkowski, J., Buculei, V. & Andruh, M. (2001). *Polyhedron*, **20**, 3033–3037.
- Ucar, I., Yesilel, O. Z., Bulut, A., Icbudak, H., Olmez, H. & Kazak, C. (2004). *Acta Cryst.* **E60**, m322–m324.
- Wittick, L. M., Jones, L. F., Jensen, P., Moubaraki, B., Spiccia, L., Berry, K. J. & Murray, K. S. (2006). *Dalton Trans.* pp. 1534–1543.
- Yang, D., Liang, Y., Ma, P., Li, S., Wang, J. & Niu, J. (2014). *CrystEngComm*, **16**, 8041–8046.
- Yeşilel, O. Z., Bulut, A., Uçar, İ., Içbudak, H., Ölmez, H. & Büyükgüngör, O. (2004). *Acta Cryst.* **E60**, m228–m230.
- Yilmaz, V. T., Senel, E. & Thöne, C. (2004). *Transition Met. Chem.* **29**, 336–342.
- Yoshida, M., Hayakawa, I., Hayashi, N., Agatsuma, T., Oda, Y., Tanzawa, F., Iwasaki, S., Koyama, K., Furukawa, H., Kurakata, S. & Sugano, Y. (2005). *Bioorg. Med. Chem. Lett.* **15**, 3328–3332.
- Zaitsev, K. V., Churakov, A. V., Poleschchuk, O. Kh., Oprunenko, Y. F., Zaitseva, G. S. & Karlov, S. S. (2014). *Dalton Trans.* **43**, 6605–6609.

supporting information

Acta Cryst. (2016). E72, 526-529 [doi:10.1107/S2056989016004515]

Crystal structure of the salt bis(triethanolamine- κ^4N,O,O',O'')cadmium bis-[2-(2-oxo-2,3-dihydro-1,3-benzothiazol-3-yl)acetate]

Jamshid Mengnorovich Ashurov

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

Bis(triethanolamine- κ^4N,O,O',O'')cadmium bis[2-(2-oxo-2,3-dihydro-1,3-benzothiazol-3-yl)acetate]

Crystal data

[Cd(C₆H₁₅NO₃)₂](C₉H₆NO₃S)₂

$M_r = 827.20$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 10.7061$ (5) Å

$b = 12.2157$ (5) Å

$c = 14.6159$ (8) Å

$\alpha = 65.520$ (5)°

$\beta = 79.600$ (4)°

$\gamma = 82.417$ (4)°

$V = 1707.59$ (14) Å³

$Z = 2$

$F(000) = 852$

$D_x = 1.609$ Mg m⁻³

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

Cell parameters from 10769 reflections

$\theta = 3.4\text{--}75.7^\circ$

$\mu = 6.85$ mm⁻¹

$T = 293$ K

Block, bright yellow

$0.6 \times 0.3 \times 0.2$ mm

Data collection

Oxford Diffraction Xcalibur Ruby diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.2576 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.127$, $T_{\max} = 0.254$

14752 measured reflections

7001 independent reflections

6533 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 75.9^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -13 \rightarrow 12$

$k = -14 \rightarrow 15$

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.107$

$S = 1.06$

7001 reflections

461 parameters

18 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
 $w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.3219P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00078 (13)

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	0.712574 (17)	0.241527 (14)	0.997601 (13)	0.02746 (9)
O6A	0.8300 (3)	0.0513 (2)	1.00868 (17)	0.0434 (5)
H6A	0.855 (4)	0.0269 (19)	0.9608 (12)	0.065*
O5A	0.5116 (2)	0.1940 (2)	0.97875 (18)	0.0444 (5)
H5A	0.4541 (16)	0.247 (2)	0.947 (2)	0.067*
O5	0.7418 (2)	0.24576 (19)	0.82842 (16)	0.0368 (5)
H5	0.736 (3)	0.186 (2)	0.8118 (19)	0.055*
O4	0.9043 (2)	0.2228 (2)	1.0772 (2)	0.0457 (6)
H4	0.960 (2)	0.1633 (14)	1.100 (3)	0.069*
O4A	0.6459 (3)	0.3096 (2)	1.13739 (19)	0.0462 (6)
H4A	0.668 (4)	0.3705 (16)	1.1455 (16)	0.069*
N2A	0.6329 (2)	0.0713 (2)	1.15330 (18)	0.0310 (5)
N2	0.8509 (2)	0.4053 (2)	0.8822 (2)	0.0333 (5)
O6	0.5915 (3)	0.4392 (3)	0.9324 (3)	0.0594 (7)
H6	0.5116 (9)	0.453 (2)	0.934 (4)	0.089*
C13A	0.5318 (3)	0.0196 (3)	1.1293 (3)	0.0405 (7)
H13A	0.4851	-0.0337	1.1921	0.049*
H13B	0.5707	-0.0279	1.0912	0.049*
C15A	0.7417 (3)	-0.0170 (3)	1.1835 (2)	0.0383 (7)
H15A	0.7114	-0.0886	1.2408	0.046*
H15B	0.8014	0.0170	1.2056	0.046*
C14	0.6569 (4)	0.5438 (3)	0.8689 (3)	0.0474 (8)
H14A	0.6188	0.6117	0.8845	0.057*
H14B	0.6518	0.5628	0.7983	0.057*
C12A	0.4410 (3)	0.1157 (3)	1.0688 (3)	0.0431 (7)
H12A	0.3780	0.0794	1.0521	0.052*
H12B	0.3973	0.1604	1.1079	0.052*
C14A	0.8105 (3)	-0.0530 (3)	1.0995 (2)	0.0404 (7)
H14C	0.8918	-0.0948	1.1175	0.048*

H14D	0.7605	-0.1072	1.0900	0.048*
C15	0.7929 (3)	0.5198 (3)	0.8870 (3)	0.0430 (7)
H15C	0.8415	0.5856	0.8366	0.052*
H15D	0.7979	0.5178	0.9533	0.052*
C13	0.8646 (4)	0.4119 (3)	0.7768 (3)	0.0445 (8)
H13C	0.9443	0.4465	0.7394	0.053*
H13D	0.7960	0.4645	0.7429	0.053*
C10	0.9712 (4)	0.3297 (3)	1.0288 (3)	0.0514 (9)
H10A	1.0567	0.3125	1.0467	0.062*
H10B	0.9289	0.3898	1.0531	0.062*
C11	0.9783 (3)	0.3794 (3)	0.9157 (3)	0.0501 (9)
H11A	1.0229	0.4531	0.8859	0.060*
H11B	1.0270	0.3219	0.8909	0.060*
C12	0.8618 (3)	0.2906 (3)	0.7759 (3)	0.0405 (7)
H12C	0.8722	0.2968	0.7066	0.049*
H12D	0.9302	0.2369	0.8094	0.049*
C11A	0.5800 (4)	0.1123 (3)	1.2352 (3)	0.0456 (8)
H11C	0.5818	0.0443	1.3002	0.055*
H11D	0.4918	0.1409	1.2286	0.055*
C10A	0.6504 (4)	0.2095 (3)	1.2338 (3)	0.0480 (8)
H10C	0.6118	0.2341	1.2883	0.058*
H10D	0.7381	0.1812	1.2433	0.058*
S1	0.71637 (9)	0.01677 (10)	0.49722 (8)	0.0535 (2)
O2	0.7640 (2)	0.05730 (19)	0.77831 (17)	0.0375 (5)
O3	0.9143 (3)	-0.0435 (2)	0.87446 (19)	0.0475 (6)
N1	0.8544 (3)	-0.0361 (2)	0.6378 (2)	0.0384 (6)
O1	0.7093 (3)	-0.1763 (3)	0.6719 (3)	0.0660 (8)
C6	0.8954 (3)	0.0728 (3)	0.5643 (2)	0.0362 (6)
C1	0.8305 (3)	0.1163 (3)	0.4801 (3)	0.0408 (7)
C9	0.8546 (3)	-0.0180 (3)	0.8009 (2)	0.0333 (6)
C8	0.8983 (4)	-0.0917 (3)	0.7365 (3)	0.0463 (8)
H8A	0.9906	-0.1007	0.7269	0.056*
H8B	0.8667	-0.1715	0.7727	0.056*
C7	0.7584 (3)	-0.0826 (3)	0.6175 (3)	0.0438 (8)
C5	0.9898 (4)	0.1373 (4)	0.5677 (3)	0.0493 (8)
H5B	1.0324	0.1099	0.6240	0.059*
C4	1.0196 (4)	0.2437 (4)	0.4851 (4)	0.0646 (12)
H4B	1.0839	0.2877	0.4856	0.078*
C2	0.8590 (5)	0.2232 (4)	0.3992 (3)	0.0555 (10)
H2	0.8145	0.2527	0.3438	0.067*
C3	0.9553 (5)	0.2852 (4)	0.4026 (4)	0.0656 (12)
H3	0.9772	0.3567	0.3478	0.079*
S1A	0.79635 (10)	0.49886 (10)	1.49702 (8)	0.0570 (2)
N1A	0.6853 (3)	0.5582 (2)	1.3394 (2)	0.0411 (6)
O2A	0.7500 (3)	0.4620 (2)	1.19404 (19)	0.0455 (5)
C2A	0.6322 (5)	0.3091 (4)	1.5830 (3)	0.0633 (12)
H2A	0.6654	0.2787	1.6440	0.076*
O3A	0.6440 (3)	0.6114 (3)	1.0843 (2)	0.0602 (7)

O1A	0.8392 (3)	0.6849 (3)	1.3194 (3)	0.0691 (8)
C6A	0.6268 (3)	0.4562 (3)	1.4111 (2)	0.0378 (7)
C7A	0.7774 (4)	0.5974 (3)	1.3696 (3)	0.0474 (8)
C8A	0.6497 (4)	0.6233 (3)	1.2389 (3)	0.0456 (8)
H8AA	0.6891	0.6998	1.2081	0.055*
H8AB	0.5583	0.6406	1.2455	0.055*
C1A	0.6771 (4)	0.4107 (3)	1.5032 (3)	0.0456 (8)
C5A	0.5312 (4)	0.4003 (4)	1.3989 (3)	0.0508 (9)
H5AA	0.4984	0.4301	1.3378	0.061*
C9A	0.6860 (3)	0.5590 (3)	1.1673 (2)	0.0353 (6)
C3A	0.5362 (5)	0.2531 (4)	1.5704 (4)	0.0703 (14)
H3A	0.5055	0.1838	1.6234	0.084*
C4A	0.4852 (4)	0.2984 (4)	1.4801 (4)	0.0645 (12)
H4AA	0.4195	0.2603	1.4739	0.077*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.03033 (13)	0.02172 (12)	0.02883 (12)	-0.00431 (7)	-0.00164 (8)	-0.00887 (8)
O6A	0.0632 (15)	0.0304 (11)	0.0320 (11)	0.0038 (10)	0.0008 (10)	-0.0131 (9)
O5A	0.0400 (12)	0.0439 (13)	0.0370 (11)	-0.0092 (10)	-0.0066 (10)	-0.0017 (10)
O5	0.0450 (12)	0.0319 (10)	0.0331 (10)	-0.0079 (9)	0.0024 (9)	-0.0143 (9)
O4	0.0386 (12)	0.0331 (11)	0.0648 (16)	-0.0003 (9)	-0.0148 (11)	-0.0165 (11)
O4A	0.0587 (15)	0.0375 (12)	0.0485 (13)	0.0007 (10)	-0.0069 (11)	-0.0247 (11)
N2A	0.0339 (12)	0.0293 (11)	0.0300 (11)	-0.0041 (9)	-0.0015 (10)	-0.0125 (10)
N2	0.0343 (13)	0.0246 (11)	0.0431 (14)	-0.0049 (9)	-0.0023 (10)	-0.0159 (10)
O6	0.0388 (13)	0.0431 (14)	0.085 (2)	-0.0025 (11)	-0.0041 (14)	-0.0170 (14)
C13A	0.0410 (17)	0.0305 (14)	0.0459 (17)	-0.0117 (12)	-0.0052 (14)	-0.0089 (13)
C15A	0.0471 (18)	0.0315 (14)	0.0336 (15)	0.0026 (12)	-0.0101 (13)	-0.0100 (12)
C14	0.050 (2)	0.0336 (16)	0.053 (2)	0.0060 (14)	-0.0059 (16)	-0.0144 (15)
C12A	0.0325 (15)	0.0476 (18)	0.0395 (16)	-0.0104 (13)	-0.0009 (13)	-0.0072 (15)
C14A	0.0499 (18)	0.0283 (14)	0.0394 (16)	0.0076 (13)	-0.0077 (14)	-0.0123 (13)
C15	0.0494 (19)	0.0256 (14)	0.0540 (19)	-0.0066 (12)	-0.0001 (15)	-0.0176 (14)
C13	0.057 (2)	0.0291 (15)	0.0405 (17)	-0.0121 (13)	0.0104 (15)	-0.0116 (13)
C10	0.0445 (19)	0.0366 (17)	0.080 (3)	-0.0048 (14)	-0.0231 (18)	-0.0240 (18)
C11	0.0341 (17)	0.0399 (18)	0.079 (3)	-0.0053 (13)	-0.0051 (17)	-0.0273 (18)
C12	0.0480 (18)	0.0344 (15)	0.0383 (16)	-0.0075 (13)	0.0055 (14)	-0.0171 (13)
C11A	0.054 (2)	0.0467 (19)	0.0336 (16)	-0.0024 (15)	0.0053 (14)	-0.0181 (14)
C10A	0.061 (2)	0.051 (2)	0.0403 (17)	-0.0004 (16)	-0.0052 (16)	-0.0282 (16)
S1	0.0509 (5)	0.0601 (5)	0.0625 (6)	0.0047 (4)	-0.0210 (4)	-0.0347 (5)
O2	0.0419 (12)	0.0328 (10)	0.0408 (11)	0.0018 (9)	-0.0047 (9)	-0.0193 (9)
O3	0.0560 (15)	0.0518 (14)	0.0432 (13)	0.0144 (11)	-0.0160 (11)	-0.0289 (12)
N1	0.0504 (16)	0.0363 (13)	0.0308 (12)	0.0019 (11)	-0.0050 (11)	-0.0173 (11)
O1	0.0657 (18)	0.0528 (17)	0.075 (2)	-0.0178 (14)	0.0162 (16)	-0.0277 (15)
C6	0.0397 (16)	0.0364 (15)	0.0368 (15)	0.0057 (12)	-0.0037 (13)	-0.0219 (13)
C1	0.0475 (18)	0.0391 (16)	0.0410 (16)	0.0087 (13)	-0.0073 (14)	-0.0240 (14)
C9	0.0407 (16)	0.0286 (13)	0.0306 (14)	-0.0007 (11)	-0.0009 (12)	-0.0139 (11)
C8	0.067 (2)	0.0382 (17)	0.0354 (16)	0.0153 (15)	-0.0129 (16)	-0.0194 (14)

C7	0.0441 (18)	0.0454 (18)	0.0467 (18)	-0.0006 (14)	0.0033 (14)	-0.0277 (16)
C5	0.0456 (19)	0.052 (2)	0.062 (2)	0.0000 (15)	-0.0057 (17)	-0.0362 (19)
C4	0.060 (2)	0.055 (2)	0.089 (3)	-0.0164 (19)	0.013 (2)	-0.044 (2)
C2	0.077 (3)	0.0425 (19)	0.0419 (19)	0.0118 (18)	-0.0065 (18)	-0.0170 (16)
C3	0.090 (3)	0.0377 (19)	0.058 (2)	-0.005 (2)	0.015 (2)	-0.0182 (18)
S1A	0.0626 (6)	0.0600 (6)	0.0567 (5)	0.0088 (4)	-0.0195 (5)	-0.0307 (5)
N1A	0.0558 (17)	0.0300 (13)	0.0342 (13)	-0.0024 (11)	-0.0031 (12)	-0.0109 (11)
O2A	0.0572 (15)	0.0322 (11)	0.0461 (13)	0.0045 (10)	-0.0083 (11)	-0.0164 (10)
C2A	0.087 (3)	0.048 (2)	0.0381 (19)	0.008 (2)	0.002 (2)	-0.0083 (17)
O3A	0.0721 (19)	0.0615 (17)	0.0503 (15)	0.0130 (14)	-0.0267 (14)	-0.0235 (14)
O1A	0.0694 (19)	0.0522 (17)	0.085 (2)	-0.0193 (14)	0.0006 (17)	-0.0263 (16)
C6A	0.0432 (17)	0.0291 (14)	0.0382 (15)	0.0040 (12)	0.0009 (13)	-0.0152 (13)
C7A	0.0486 (19)	0.0440 (18)	0.053 (2)	-0.0005 (15)	-0.0009 (16)	-0.0268 (17)
C8A	0.065 (2)	0.0266 (14)	0.0405 (17)	0.0064 (14)	-0.0084 (16)	-0.0107 (13)
C1A	0.055 (2)	0.0383 (17)	0.0373 (16)	0.0094 (15)	-0.0017 (15)	-0.0150 (14)
C5A	0.050 (2)	0.0458 (19)	0.058 (2)	-0.0030 (15)	0.0010 (17)	-0.0256 (18)
C9A	0.0370 (16)	0.0314 (14)	0.0358 (15)	-0.0042 (11)	-0.0051 (12)	-0.0114 (12)
C3A	0.087 (3)	0.044 (2)	0.057 (3)	-0.008 (2)	0.023 (2)	-0.0100 (19)
C4A	0.059 (2)	0.048 (2)	0.084 (3)	-0.0146 (18)	0.019 (2)	-0.033 (2)

Geometric parameters (Å, °)

Cd1—O5A	2.392 (2)	C12—H12C	0.9700
Cd1—O5	2.415 (2)	C12—H12D	0.9700
Cd1—O6A	2.449 (2)	C11A—C10A	1.481 (5)
Cd1—N2A	2.465 (2)	C11A—H11C	0.9700
Cd1—O4A	2.470 (2)	C11A—H11D	0.9700
Cd1—N2	2.475 (3)	C10A—H10C	0.9700
Cd1—O6	2.476 (3)	C10A—H10D	0.9700
Cd1—O4	2.478 (2)	S1—C1	1.750 (4)
O6A—C14A	1.413 (4)	S1—C7	1.772 (4)
O6A—H6A	0.856 (9)	O2—C9	1.234 (4)
O5A—C12A	1.416 (4)	O3—C9	1.256 (4)
O5A—H5A	0.871 (10)	N1—C7	1.368 (5)
O5—C12	1.422 (4)	N1—C6	1.384 (4)
O5—H5	0.872 (10)	N1—C8	1.453 (4)
O4—C10	1.420 (4)	O1—C7	1.213 (5)
O4—H4	0.866 (9)	C6—C5	1.381 (5)
O4A—C10A	1.436 (5)	C6—C1	1.396 (5)
O4A—H4A	0.870 (10)	C1—C2	1.375 (5)
N2A—C15A	1.475 (4)	C9—C8	1.533 (4)
N2A—C11A	1.477 (4)	C8—H8A	0.9700
N2A—C13A	1.480 (4)	C8—H8B	0.9700
N2—C15	1.478 (4)	C5—C4	1.385 (6)
N2—C11	1.482 (4)	C5—H5B	0.9300
N2—C13	1.490 (4)	C4—C3	1.374 (7)
O6—C14	1.415 (5)	C4—H4B	0.9300
O6—H6	0.848 (9)	C2—C3	1.376 (7)

C13A—C12A	1.502 (5)	C2—H2	0.9300
C13A—H13A	0.9700	C3—H3	0.9300
C13A—H13B	0.9700	S1A—C1A	1.743 (4)
C15A—C14A	1.509 (4)	S1A—C7A	1.781 (4)
C15A—H15A	0.9700	N1A—C7A	1.362 (5)
C15A—H15B	0.9700	N1A—C6A	1.389 (4)
C14—C15	1.495 (5)	N1A—C8A	1.446 (4)
C14—H14A	0.9700	O2A—C9A	1.235 (4)
C14—H14B	0.9700	C2A—C1A	1.373 (6)
C12A—H12A	0.9700	C2A—C3A	1.384 (8)
C12A—H12B	0.9700	C2A—H2A	0.9300
C14A—H14C	0.9700	O3A—C9A	1.249 (4)
C14A—H14D	0.9700	O1A—C7A	1.216 (5)
C15—H15C	0.9700	C6A—C5A	1.376 (5)
C15—H15D	0.9700	C6A—C1A	1.406 (5)
C13—C12	1.492 (4)	C8A—C9A	1.522 (4)
C13—H13C	0.9700	C8A—H8AA	0.9700
C13—H13D	0.9700	C8A—H8AB	0.9700
C10—C11	1.498 (6)	C5A—C4A	1.388 (6)
C10—H10A	0.9700	C5A—H5AA	0.9300
C10—H10B	0.9700	C3A—C4A	1.386 (8)
C11—H11A	0.9700	C3A—H3A	0.9300
C11—H11B	0.9700	C4A—H4AA	0.9300
O5A—Cd1—O5	75.23 (8)	H13C—C13—H13D	108.0
O5A—Cd1—O6A	97.23 (9)	O4—C10—C11	111.8 (3)
O5—Cd1—O6A	74.12 (7)	O4—C10—H10A	109.2
O5A—Cd1—N2A	70.99 (8)	C11—C10—H10A	109.2
O5—Cd1—N2A	124.88 (8)	O4—C10—H10B	109.2
O6A—Cd1—N2A	68.58 (8)	C11—C10—H10B	109.2
O5A—Cd1—O4A	100.09 (9)	H10A—C10—H10B	107.9
O5—Cd1—O4A	159.22 (8)	N2—C11—C10	112.5 (3)
O6A—Cd1—O4A	126.65 (8)	N2—C11—H11A	109.1
N2A—Cd1—O4A	70.34 (8)	C10—C11—H11A	109.1
O5A—Cd1—N2	130.03 (8)	N2—C11—H11B	109.1
O5—Cd1—N2	70.57 (8)	C10—C11—H11B	109.1
O6A—Cd1—N2	106.90 (8)	H11A—C11—H11B	107.8
N2A—Cd1—N2	158.75 (9)	O5—C12—C13	107.1 (3)
O4A—Cd1—N2	99.33 (9)	O5—C12—H12C	110.3
O5A—Cd1—O6	75.81 (9)	C13—C12—H12C	110.3
O5—Cd1—O6	88.12 (10)	O5—C12—H12D	110.3
O6A—Cd1—O6	162.13 (10)	C13—C12—H12D	110.3
N2A—Cd1—O6	122.59 (9)	H12C—C12—H12D	108.6
O4A—Cd1—O6	71.14 (10)	N2A—C11A—C10A	113.0 (3)
N2—Cd1—O6	67.86 (9)	N2A—C11A—H11C	109.0
O5A—Cd1—O4	158.32 (8)	C10A—C11A—H11C	109.0
O5—Cd1—O4	118.25 (9)	N2A—C11A—H11D	109.0
O6A—Cd1—O4	72.54 (9)	C10A—C11A—H11D	109.0

N2A—Cd1—O4	87.39 (8)	H11C—C11A—H11D	107.8
O4A—Cd1—O4	73.06 (9)	O4A—C10A—C11A	108.1 (3)
N2—Cd1—O4	71.65 (9)	O4A—C10A—H10C	110.1
O6—Cd1—O4	119.25 (9)	C11A—C10A—H10C	110.1
C14A—O6A—Cd1	119.37 (18)	O4A—C10A—H10D	110.1
C14A—O6A—H6A	106.6 (15)	C11A—C10A—H10D	110.1
Cd1—O6A—H6A	127.9 (16)	H10C—C10A—H10D	108.4
C12A—O5A—Cd1	115.7 (2)	C1—S1—C7	92.04 (17)
C12A—O5A—H5A	103.9 (15)	C7—N1—C6	116.2 (3)
Cd1—O5A—H5A	124.9 (17)	C7—N1—C8	120.8 (3)
C12—O5—Cd1	109.85 (18)	C6—N1—C8	122.6 (3)
C12—O5—H5	103.7 (15)	C5—C6—N1	126.8 (3)
Cd1—O5—H5	127.1 (17)	C5—C6—C1	120.1 (3)
C10—O4—Cd1	111.1 (2)	N1—C6—C1	113.0 (3)
C10—O4—H4	107.2 (16)	C2—C1—C6	121.1 (4)
Cd1—O4—H4	131.4 (17)	C2—C1—S1	128.9 (3)
C10A—O4A—Cd1	110.41 (18)	C6—C1—S1	110.0 (3)
C10A—O4A—H4A	105.2 (16)	O2—C9—O3	126.3 (3)
Cd1—O4A—H4A	130.4 (18)	O2—C9—C8	118.3 (3)
C15A—N2A—C11A	110.2 (3)	O3—C9—C8	115.4 (3)
C15A—N2A—C13A	111.4 (2)	N1—C8—C9	112.6 (3)
C11A—N2A—C13A	109.5 (3)	N1—C8—H8A	109.1
C15A—N2A—Cd1	107.07 (18)	C9—C8—H8A	109.1
C11A—N2A—Cd1	110.73 (19)	N1—C8—H8B	109.1
C13A—N2A—Cd1	107.92 (18)	C9—C8—H8B	109.1
C15—N2—C11	109.8 (3)	H8A—C8—H8B	107.8
C15—N2—C13	110.8 (3)	O1—C7—N1	126.4 (4)
C11—N2—C13	109.0 (3)	O1—C7—S1	124.9 (3)
C15—N2—Cd1	108.45 (19)	N1—C7—S1	108.7 (3)
C11—N2—Cd1	109.2 (2)	C6—C5—C4	118.4 (4)
C13—N2—Cd1	109.68 (18)	C6—C5—H5B	120.8
C14—O6—Cd1	119.6 (2)	C4—C5—H5B	120.8
C14—O6—H6	111.6 (15)	C3—C4—C5	120.8 (4)
Cd1—O6—H6	127.8 (15)	C3—C4—H4B	119.6
N2A—C13A—C12A	111.9 (3)	C5—C4—H4B	119.6
N2A—C13A—H13A	109.2	C1—C2—C3	118.1 (4)
C12A—C13A—H13A	109.2	C1—C2—H2	120.9
N2A—C13A—H13B	109.2	C3—C2—H2	120.9
C12A—C13A—H13B	109.2	C4—C3—C2	121.4 (4)
H13A—C13A—H13B	107.9	C4—C3—H3	119.3
N2A—C15A—C14A	113.5 (3)	C2—C3—H3	119.3
N2A—C15A—H15A	108.9	C1A—S1A—C7A	91.44 (18)
C14A—C15A—H15A	108.9	C7A—N1A—C6A	116.3 (3)
N2A—C15A—H15B	108.9	C7A—N1A—C8A	120.6 (3)
C14A—C15A—H15B	108.9	C6A—N1A—C8A	123.1 (3)
H15A—C15A—H15B	107.7	C1A—C2A—C3A	118.4 (4)
O6—C14—C15	108.2 (3)	C1A—C2A—H2A	120.8
O6—C14—H14A	110.1	C3A—C2A—H2A	120.8

C15—C14—H14A	110.1	C5A—C6A—N1A	127.0 (3)
O6—C14—H14B	110.1	C5A—C6A—C1A	120.8 (3)
C15—C14—H14B	110.1	N1A—C6A—C1A	112.2 (3)
H14A—C14—H14B	108.4	O1A—C7A—N1A	126.9 (4)
O5A—C12A—C13A	108.3 (3)	O1A—C7A—S1A	123.9 (3)
O5A—C12A—H12A	110.0	N1A—C7A—S1A	109.2 (3)
C13A—C12A—H12A	110.0	N1A—C8A—C9A	115.5 (3)
O5A—C12A—H12B	110.0	N1A—C8A—H8AA	108.4
C13A—C12A—H12B	110.0	C9A—C8A—H8AA	108.4
H12A—C12A—H12B	108.4	N1A—C8A—H8AB	108.4
O6A—C14A—C15A	109.3 (2)	C9A—C8A—H8AB	108.4
O6A—C14A—H14C	109.8	H8AA—C8A—H8AB	107.5
C15A—C14A—H14C	109.8	C2A—C1A—C6A	120.6 (4)
O6A—C14A—H14D	109.8	C2A—C1A—S1A	128.5 (3)
C15A—C14A—H14D	109.8	C6A—C1A—S1A	110.9 (3)
H14C—C14A—H14D	108.3	C6A—C5A—C4A	118.4 (4)
N2—C15—C14	113.1 (3)	C6A—C5A—H5AA	120.8
N2—C15—H15C	108.9	C4A—C5A—H5AA	120.8
C14—C15—H15C	108.9	O2A—C9A—O3A	125.5 (3)
N2—C15—H15D	108.9	O2A—C9A—C8A	120.0 (3)
C14—C15—H15D	108.9	O3A—C9A—C8A	114.5 (3)
H15C—C15—H15D	107.8	C2A—C3A—C4A	121.2 (4)
N2—C13—C12	111.6 (3)	C2A—C3A—H3A	119.4
N2—C13—H13C	109.3	C4A—C3A—H3A	119.4
C12—C13—H13C	109.3	C3A—C4A—C5A	120.6 (5)
N2—C13—H13D	109.3	C3A—C4A—H4AA	119.7
C12—C13—H13D	109.3	C5A—C4A—H4AA	119.7
O5A—Cd1—O6A—C14A	-75.5 (3)	C15A—N2A—C13A—C12A	-162.3 (3)
O5—Cd1—O6A—C14A	-147.9 (3)	C11A—N2A—C13A—C12A	75.6 (3)
N2A—Cd1—O6A—C14A	-9.3 (2)	Cd1—N2A—C13A—C12A	-45.0 (3)
O4A—Cd1—O6A—C14A	32.8 (3)	C11A—N2A—C15A—C14A	-173.5 (3)
N2—Cd1—O6A—C14A	148.7 (2)	C13A—N2A—C15A—C14A	64.7 (4)
O6—Cd1—O6A—C14A	-141.2 (3)	Cd1—N2A—C15A—C14A	-53.0 (3)
O4—Cd1—O6A—C14A	84.9 (3)	Cd1—O6—C14—C15	-22.1 (4)
O5—Cd1—O5A—C12A	148.5 (2)	Cd1—O5A—C12A—C13A	-40.0 (4)
O6A—Cd1—O5A—C12A	77.1 (2)	N2A—C13A—C12A—O5A	57.6 (4)
N2A—Cd1—O5A—C12A	12.7 (2)	Cd1—O6A—C14A—C15A	-14.3 (4)
O4A—Cd1—O5A—C12A	-52.3 (2)	N2A—C15A—C14A—O6A	45.6 (4)
N2—Cd1—O5A—C12A	-163.6 (2)	C11—N2—C15—C14	-171.7 (3)
O6—Cd1—O5A—C12A	-119.7 (3)	C13—N2—C15—C14	67.9 (4)
O4—Cd1—O5A—C12A	17.0 (4)	Cd1—N2—C15—C14	-52.5 (3)
O5A—Cd1—O5—C12	172.1 (2)	O6—C14—C15—N2	49.7 (4)
O6A—Cd1—O5—C12	-85.8 (2)	C15—N2—C13—C12	-152.6 (3)
N2A—Cd1—O5—C12	-134.39 (19)	C11—N2—C13—C12	86.5 (3)
O4A—Cd1—O5—C12	92.7 (3)	Cd1—N2—C13—C12	-32.9 (3)
N2—Cd1—O5—C12	29.11 (19)	Cd1—O4—C10—C11	-42.0 (3)
O6—Cd1—O5—C12	96.3 (2)	C15—N2—C11—C10	78.3 (3)

O4—Cd1—O5—C12	-26.2 (2)	C13—N2—C11—C10	-160.2 (3)
O5A—Cd1—O4—C10	-165.5 (3)	Cd1—N2—C11—C10	-40.4 (3)
O5—Cd1—O4—C10	69.8 (2)	O4—C10—C11—N2	57.5 (4)
O6A—Cd1—O4—C10	130.2 (3)	Cd1—O5—C12—C13	-56.8 (3)
N2A—Cd1—O4—C10	-161.5 (2)	N2—C13—C12—O5	60.9 (4)
O4A—Cd1—O4—C10	-91.2 (2)	C15A—N2A—C11A—C10A	82.7 (4)
N2—Cd1—O4—C10	15.0 (2)	C13A—N2A—C11A—C10A	-154.5 (3)
O6—Cd1—O4—C10	-35.1 (3)	Cd1—N2A—C11A—C10A	-35.6 (4)
O5A—Cd1—O4A—C10A	89.6 (2)	Cd1—O4A—C10A—C11A	-50.9 (3)
O5—Cd1—O4A—C10A	164.5 (2)	N2A—C11A—C10A—O4A	59.1 (4)
O6A—Cd1—O4A—C10A	-17.3 (3)	C7—N1—C6—C5	179.6 (3)
N2A—Cd1—O4A—C10A	24.1 (2)	C8—N1—C6—C5	7.1 (5)
N2—Cd1—O4A—C10A	-136.6 (2)	C7—N1—C6—C1	-0.5 (4)
O6—Cd1—O4A—C10A	160.7 (3)	C8—N1—C6—C1	-173.1 (3)
O4—Cd1—O4A—C10A	-69.2 (2)	C5—C6—C1—C2	0.2 (5)
O5A—Cd1—N2A—C15A	137.1 (2)	N1—C6—C1—C2	-179.7 (3)
O5—Cd1—N2A—C15A	81.8 (2)	C5—C6—C1—S1	-179.6 (2)
O6A—Cd1—N2A—C15A	30.95 (18)	N1—C6—C1—S1	0.5 (3)
O4A—Cd1—N2A—C15A	-114.2 (2)	C7—S1—C1—C2	179.9 (3)
N2—Cd1—N2A—C15A	-50.6 (3)	C7—S1—C1—C6	-0.3 (2)
O6—Cd1—N2A—C15A	-164.77 (18)	C7—N1—C8—C9	-106.3 (4)
O4—Cd1—N2A—C15A	-41.31 (19)	C6—N1—C8—C9	66.0 (4)
O5A—Cd1—N2A—C11A	-102.8 (2)	O2—C9—C8—N1	19.0 (5)
O5—Cd1—N2A—C11A	-158.1 (2)	O3—C9—C8—N1	-162.7 (3)
O6A—Cd1—N2A—C11A	151.1 (2)	C6—N1—C7—O1	-179.9 (3)
O4A—Cd1—N2A—C11A	5.9 (2)	C8—N1—C7—O1	-7.2 (5)
N2—Cd1—N2A—C11A	69.6 (3)	C6—N1—C7—S1	0.3 (3)
O6—Cd1—N2A—C11A	-44.6 (2)	C8—N1—C7—S1	173.0 (2)
O4—Cd1—N2A—C11A	78.8 (2)	C1—S1—C7—O1	-179.8 (3)
O5A—Cd1—N2A—C13A	17.12 (19)	C1—S1—C7—N1	0.0 (2)
O5—Cd1—N2A—C13A	-38.2 (2)	N1—C6—C5—C4	178.5 (3)
O6A—Cd1—N2A—C13A	-89.0 (2)	C1—C6—C5—C4	-1.3 (5)
O4A—Cd1—N2A—C13A	125.8 (2)	C6—C5—C4—C3	1.1 (6)
N2—Cd1—N2A—C13A	-170.6 (2)	C6—C1—C2—C3	1.3 (5)
O6—Cd1—N2A—C13A	75.2 (2)	S1—C1—C2—C3	-179.0 (3)
O4—Cd1—N2A—C13A	-161.29 (19)	C5—C4—C3—C2	0.4 (6)
O5A—Cd1—N2—C15	74.1 (2)	C1—C2—C3—C4	-1.6 (6)
O5—Cd1—N2—C15	123.6 (2)	C7A—N1A—C6A—C5A	179.6 (3)
O6A—Cd1—N2—C15	-170.6 (2)	C8A—N1A—C6A—C5A	1.5 (5)
N2A—Cd1—N2—C15	-96.4 (3)	C7A—N1A—C6A—C1A	-1.0 (4)
O4A—Cd1—N2—C15	-37.6 (2)	C8A—N1A—C6A—C1A	-179.1 (3)
O6—Cd1—N2—C15	27.5 (2)	C6A—N1A—C7A—O1A	-179.2 (4)
O4—Cd1—N2—C15	-106.2 (2)	C8A—N1A—C7A—O1A	-1.1 (6)
O5A—Cd1—N2—C11	-166.3 (2)	C6A—N1A—C7A—S1A	1.1 (4)
O5—Cd1—N2—C11	-116.8 (2)	C8A—N1A—C7A—S1A	179.2 (2)
O6A—Cd1—N2—C11	-51.1 (2)	C1A—S1A—C7A—O1A	179.6 (4)
N2A—Cd1—N2—C11	23.2 (3)	C1A—S1A—C7A—N1A	-0.7 (3)
O4A—Cd1—N2—C11	81.9 (2)	C7A—N1A—C8A—C9A	111.3 (4)

O6—Cd1—N2—C11	147.1 (2)	C6A—N1A—C8A—C9A	-70.7 (4)
O4—Cd1—N2—C11	13.4 (2)	C3A—C2A—C1A—C6A	-0.2 (6)
O5A—Cd1—N2—C13	-47.0 (2)	C3A—C2A—C1A—S1A	-179.7 (3)
O5—Cd1—N2—C13	2.5 (2)	C5A—C6A—C1A—C2A	0.3 (5)
O6A—Cd1—N2—C13	68.3 (2)	N1A—C6A—C1A—C2A	-179.2 (3)
N2A—Cd1—N2—C13	142.5 (2)	C5A—C6A—C1A—S1A	179.9 (3)
O4A—Cd1—N2—C13	-158.7 (2)	N1A—C6A—C1A—S1A	0.4 (3)
O6—Cd1—N2—C13	-93.5 (2)	C7A—S1A—C1A—C2A	179.7 (4)
O4—Cd1—N2—C13	132.8 (2)	C7A—S1A—C1A—C6A	0.1 (3)
O5A—Cd1—O6—C14	-147.9 (3)	N1A—C6A—C5A—C4A	179.8 (3)
O5—Cd1—O6—C14	-72.6 (3)	C1A—C6A—C5A—C4A	0.4 (5)
O6A—Cd1—O6—C14	-79.0 (4)	N1A—C8A—C9A—O2A	-5.2 (5)
N2A—Cd1—O6—C14	156.2 (3)	N1A—C8A—C9A—O3A	173.6 (3)
O4A—Cd1—O6—C14	106.0 (3)	C1A—C2A—C3A—C4A	-0.6 (7)
N2—Cd1—O6—C14	-2.9 (3)	C2A—C3A—C4A—C5A	1.4 (7)
O4—Cd1—O6—C14	49.0 (3)	C6A—C5A—C4A—C3A	-1.3 (6)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4 \cdots O3 ⁱ	0.87 (3)	1.82 (2)	2.676 (4)	170 (4)
O4A—H4A \cdots O2A	0.87 (3)	1.91 (3)	2.748 (4)	161 (3)
O5—H5 \cdots O2	0.87 (3)	1.81 (3)	2.661 (3)	166 (3)
O5A—H5A \cdots O3A ⁱⁱ	0.87 (2)	1.83 (3)	2.640 (4)	154 (2)
O6—H6 \cdots O3A ⁱⁱ	0.85 (2)	2.04 (2)	2.749 (5)	141 (3)
O6A—H6A \cdots O3	0.86 (2)	1.79 (2)	2.640 (4)	172 (2)
C11A—H11D \cdots O1 ⁱⁱⁱ	0.97	2.47	3.282 (6)	142

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