

Bis(creatininium) tetrachlorido-cadmate(II)

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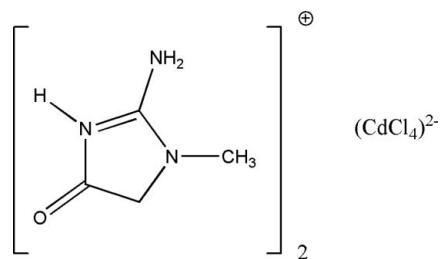
Received 22 June 2009; accepted 9 July 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.019; wR factor = 0.048; data-to-parameter ratio = 17.8.

In the title compound, $(\text{C}_4\text{H}_8\text{N}_3\text{O})_2[\text{CdCl}_4]$, the asymmetric unit comprises two creatininium cations and one tetrachloridocadmate anion. $\text{Cd}\cdots\text{O}$ secondary bonding links one of the two imidazole rings and the anion into ion pairs. The free and bound cations form layers between which the $[\text{CdCl}_4]^{2-}$ anions are sandwiched. The Cd^{II} atom adopts a distorted trigonal-bipyramidal geometry in which the $\text{Cd}\cdots\text{O}$ bond is axial. Intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds form a two-dimensional network parallel to (001) which ensures the junction between creatininium cations and $[\text{CdCl}_4]^{2-}$ anions.

Related literature

An abnormal level of creatinine in biological fluids is an indicator of various medical conditions, see: Narayanan & Appleton (1980). For interactions between creatinine and biologically important metal ions, see: Canty *et al.* (1979). Different complex species are formed depending on the reaction conditions, see: Nishida & Kida (1985). For bond lengths in the neutral creatinine molecule, see: Smith & White (2001) and in creatinium compounds, see: Wilkinson & Harrison (2005). For $\text{Cd}-\text{Cl}$ bond distances in dichlorido-bis(creatinine)cadmium(II), see: Okabe *et al.* (1995). For $\text{Cl}-\text{Cd}-\text{Cl}$ bond angles in bis(2,3,5-triphenyltetrazolium)tetrachloridocadmate(II), see: Zhang *et al.* (2007). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$(\text{C}_4\text{H}_8\text{N}_3\text{O})_2[\text{CdCl}_4]$
 $M_r = 482.48$
 Triclinic, $P\bar{1}$
 $a = 7.5203$ (4) Å
 $b = 7.6761$ (3) Å
 $c = 15.0757$ (7) Å
 $\alpha = 79.476$ (2)°
 $\beta = 85.438$ (5)°

$\gamma = 83.214$ (3)°
 $V = 848.14$ (7) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.93$ mm⁻¹
 $T = 100$ K
 $0.25 \times 0.15 \times 0.1$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.644$, $T_{\text{max}} = 0.831$

10950 measured reflections
 3843 independent reflections
 3711 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.048$
 $S = 1.11$
 3843 reflections
 216 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.77$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.54$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—Cl1	2.4571 (5)	Cd1—Cl3	2.5678 (4)
Cd1—Cl4	2.4596 (4)	Cd1—O1	2.6854 (13)
Cd1—Cl2	2.4627 (4)		
Cl1—Cd1—Cl4	116.512 (15)	Cl2—Cd1—Cl3	99.319 (15)
Cl1—Cd1—Cl2	120.122 (17)	Cl1—Cd1—O1	82.18 (3)
Cl4—Cd1—Cl2	117.621 (16)	Cl4—Cd1—O1	82.85 (3)
Cl1—Cd1—Cl3	96.649 (15)	Cl2—Cd1—O1	80.95 (3)
Cl4—Cd1—Cl3	98.074 (15)	Cl3—Cd1—O1	178.75 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N1—H1 ⁱ ···Cl3	0.79 (2)	2.41 (2)	3.1840 (16)	168 (2)
N2—H2 ⁱ ···Cl3 ⁱ	0.80 (3)	2.48 (3)	3.273 (2)	170 (3)
N4—H4 ⁱ ···Cl4	0.75 (2)	2.70 (2)	3.2967 (17)	139 (2)
N6—H6 ⁱ ···Cl2 ⁱⁱ	0.88 (3)	2.32 (3)	3.1673 (18)	161 (3)
N2—H22 ⁱ ···Cl1	0.90 (2)	2.34 (2)	3.2368 (18)	171 (2)
N6—H66 ⁱ ···Cl4 ⁱⁱⁱ	0.81 (3)	2.55 (3)	3.2117 (17)	140 (2)

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

Data collection: COLLECT (Nonius, 2002); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve

structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

This work was supported by the Université 20 Aout 1955 Skikda, Algérie. We would like to thank Thierry Roisnel from Renne 1 University for collecting the data and Olivier Mentré from UCCS, Equipe de Chimie du Solide, UMR CNRS 8181, ENSC Lille, France for his useful help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2978).

References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Canty, A. J., Chaichit, N. & Gatehouse, B. M. (1979). *Acta Cryst.* **B35**, 592–596.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- 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.
- Narayanan, S. & Appleton, H. D. (1980). *Clin. Chem.* **26**, 1119–1126.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Nishida, Y. & Kida, S. (1985). *Bull. Chem. Soc. Jpn.* **58**, 383–384.
- Nonius (2002). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Okabe, N., Ikeda, K., Kohyama, Y. & Sasaki, Y. (1995). *Acta Cryst.* **C51**, 224–226.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Smith, G. & White, J. M. (2001). *Aust. J. Chem.* **54**, 97–100.
- Wilkinson, H. S. & Harrison, W. T. A. (2005). *Acta Cryst.* **E61**, m1228–m1230.
- Zhang, S.-F., Yang, X.-G., Liu, Z., Li, W.-H. & Hou, B.-R. (2007). *Acta Cryst.* **E63**, m1583.

supplementary materials

Acta Cryst. (2009). E65, m930-m931 [doi:10.1107/S1600536809026944]

Bis(creatininium) tetrachloridocadmiate(II)

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Comment

Creatinine (2-amino- 1,5-dihydro- 1-methyl-4*H*-imidazol-4-one) is the waste product of protein metabolism that is found in the urine. It can be measured to assess overall kidney function. An abnormal level of creatinine in biological fluids is an indicator of various malady states (Narayanan & Appleton, 1980).

Over the last decade, there have been an increasing number of reports on compounds resulting from the combination of creatinine and biologically important metal ions, in order to obtain structural information on the mode of interaction between them. (Canty *et al.*, 1979), however, the different complex species are formed depending on the reaction conditions (Nishida & Kida, 1985). In the title compound, (I), we determine the crystal structure of a new cadmium complex showing the metal coordination and the structure cohesion. It is the first example of salt containing creatinium cation and $(\text{CdCl}_4)^{2-}$ anion.

The asymmetric unit, shown in Fig. 1, consists of the tetrachlorocadmiate anion and two cations of creatinine protonated. Hence, the complex investigated is better formulated as: $2(\text{C}_4\text{H}_8\text{ON}_3)^+, (\text{CdCl}_4)^{2-}$.

The cadmium is coordinated to four Cl atoms and one O atom forming a distorted trigonal bipyramid CdX_4O . As can be seen in Table 1, three of the four Cd1—Cl bond distances (2.4571 (5), 2.4596 (4) and 2.4627 (4) Å) are similar to those found in the dichlorobis(creatinine)cadmium(II) (Okabe *et al.*, 1995) and significantly shorter than the axial Cd \cdots Cl3 distance (2.5678 (4) Å) which is in *trans* position to the O atom ($d_{\text{Cd1—O}} = 2.6854$ (13) Å and 0.1 Å longer than the other chlorine atoms. In the trigonal bipyramid CdCl_4O , the displacement of Cd towards Cl3 from the equatorial plane defined by Cl1; Cl2 and Cl4 atoms is 0.2574 (2) Å. Furthermore, the Cl—Cd—Cl bond angles in the title complex fall in the range 96.649 (15) ° - 120.122 (17) ° which is very different to those of the free anionic CdCl_4 moiety of bis(2,3,5-triphenyltetrazolium) tetrachloridocadmiate(II) (Zhang *et al.*, 2007) where the Cl—Cd—Cl bond angles lie in the range 107.18 (3) ° - 117.232 (16) °.

As expected, creatinium cations are approximately planar. In the bound cation, the r.m.s. deviation for the non-H atoms = 0.0311 Å, the maximum deviation of N5 from the mean plane is 0.0592 (15) Å. However, the free cation has a r.m.s deviation for non-H atoms = 0.0488 Å with a maximum deviation from the mean plane = 0.0851 (0.11) Å (for O2).

In table 1, we notice that in each creatinium cation, the three C—N bond distances are clearly different, with C6—N4 and C3—N1 much longer than the other ones. This results in the localization of the exocyclic C3—N2 [1.325 (2) Å] & C3—N3 [1.321 (2) Å] and C6—N5 [1.319 (2) Å] & C6—N6 [1.322 (2) Å] double bonds in the free and bound creatinium respectively and the adjacent single bonds C3—N1 [1.371 (2) Å] and C6—N4 [1.380 (2) Å]. These values somewhat compared with the intermediate, delocalized values in the parent neutral creatinine molecule [1.320 (3) and 1.349 (3) Å], respectively (Smith & White, 2001). Such difference in C—N bonds has been reported in some creatinium compounds (Wilkinson *et al.*, 2005).

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In the crystal structure of (I), the anionic and cationic components are linked by N—H \cdots Cl, hydrogen bonds into a continuous two-dimensional network (Table 3). In which alternating $R^2_2(8)$, $R^2_4(12)$ and $R^6_6(28)$ (Bernstein, *et al.*, 1995) hydrogen-bonded rings are formed. Fig. 2 also shows a strong intramolecular hydrogen bond (N4—H4 \cdots Cl4) which can be described with an S(6) graph set motif. These rings form layers joined by means of the N6—H6 \cdots Cl2 hydrogen bond. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for this pattern is $R^4_4(12)$ ring (Fig. 3).

Experimental

The title compound was crystallized from a supersaturated hydrochloric acid solution (45%, 5 ml) prepared using doubly distilled water and a mixture of cadmium(II) chloride (1.83 g) and creatinine (2.26 g). Colourless plates-shaped single crystals of (I) were obtained at ambient temperature by slow evaporation of the solution.

Refinement

The iminium and ammino H atoms were located in a difference Fourier map and was refined isotropically. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C—C bonds. The methylene H atoms were placed in geometrically idealized positions (C—H = 0.97 Å) and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

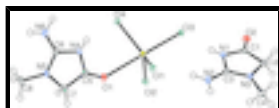


Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

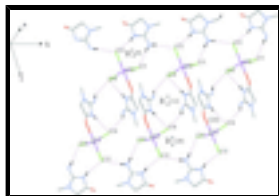


Fig. 2. Part of the crystal structure of (I), showing the formation of the $R^2_2(8)$, $R^2_4(12)$ and $R^6_6(28)$ rings. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in the motif have been omitted for clarity.

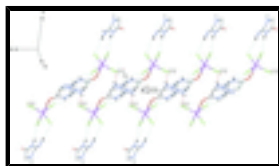


Fig. 3. Part of the crystal structure of (I), showing the $R^4_4(12)$ ring set motif

(I)

Crystal data

(C₄H₈N₃O)₂[CdCl₄]

$M_r = 482.48$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.5203(4)$ Å

$Z = 2$

$F_{000} = 476$

$D_x = 1.889$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8132 reflections

$b = 7.6761 (3) \text{ \AA}$
 $c = 15.0757 (7) \text{ \AA}$
 $\alpha = 79.476 (2)^\circ$
 $\beta = 85.438 (5)^\circ$
 $\gamma = 83.214 (3)^\circ$
 $V = 848.14 (7) \text{ \AA}^3$

$\theta = 2.7\text{--}27.4^\circ$
 $\mu = 1.93 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 Plates, colourless
 $0.25 \times 0.15 \times 0.1 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 Radiation source: fine-focus sealed X-ray tube
 Monochromator: graphite
 $T = 100 \text{ K}$
 φ scans, and ω scans with κ offsets
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.644$, $T_{\max} = 0.831$
 10950 measured reflections

3843 independent reflections
 3711 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$
 $\theta_{\max} = 27.4^\circ$
 $\theta_{\min} = 2.7^\circ$
 $h = -9 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.048$
 $S = 1.11$
 3843 reflections
 216 parameters
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0154P)^2 + 0.4426P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.77 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.54 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.08 (2)

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.

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	0.066723 (15)	0.225860 (16)	0.280951 (8)	0.01066 (5)

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Cl1	-0.04670 (6)	0.54257 (6)	0.24433 (3)	0.01616 (9)
Cl2	0.36325 (5)	0.13754 (7)	0.34349 (3)	0.01814 (10)
Cl3	0.14704 (6)	0.17937 (6)	0.11755 (3)	0.01524 (9)
Cl4	-0.15980 (5)	0.01360 (6)	0.32005 (3)	0.01418 (9)
O1	-0.01715 (17)	0.28215 (19)	0.45064 (9)	0.0176 (3)
N4	-0.3242 (2)	0.2622 (2)	0.47014 (11)	0.0130 (3)
N5	-0.38183 (19)	0.3218 (2)	0.60861 (10)	0.0134 (3)
N6	-0.6224 (2)	0.2535 (2)	0.53371 (12)	0.0179 (3)
C5	-0.1579 (2)	0.2895 (2)	0.49571 (12)	0.0127 (3)
C6	-0.4526 (2)	0.2762 (2)	0.53994 (12)	0.0126 (3)
C7	-0.1888 (2)	0.3305 (3)	0.59036 (12)	0.0144 (4)
H7A	-0.1208	0.2427	0.633	0.017*
H7B	-0.1565	0.4481	0.5929	0.017*
C8	-0.4703 (3)	0.3343 (3)	0.69777 (13)	0.0213 (4)
H8A	-0.4273	0.4292	0.7208	0.026*
H8B	-0.444	0.2239	0.7383	0.026*
H8C	-0.5976	0.358	0.6926	0.026*
O2	0.40943 (18)	0.36919 (18)	-0.10195 (9)	0.0191 (3)
N1	0.2474 (2)	0.5415 (2)	-0.00578 (11)	0.0139 (3)
C1	0.3552 (2)	0.5126 (2)	-0.08243 (12)	0.0138 (3)
C2	0.3922 (2)	0.6952 (2)	-0.13185 (12)	0.0127 (3)
H2A	0.52	0.7038	-0.1423	0.015*
H2B	0.3364	0.7239	-0.1893	0.015*
N2	0.1430 (2)	0.7800 (2)	0.07081 (11)	0.0176 (3)
N3	0.3113 (2)	0.8115 (2)	-0.06971 (10)	0.0129 (3)
C3	0.2295 (2)	0.7185 (2)	0.00098 (12)	0.0130 (3)
C4	0.3227 (3)	1.0033 (2)	-0.08903 (13)	0.0166 (4)
H4A	0.2574	1.0553	-0.1413	0.02*
H4B	0.4461	1.0256	-0.1004	0.02*
H4C	0.2722	1.0551	-0.0381	0.02*
H4	-0.340 (3)	0.224 (3)	0.4298 (17)	0.017 (6)*
H66	-0.689 (4)	0.238 (3)	0.5785 (19)	0.032 (7)*
H6	-0.653 (4)	0.215 (4)	0.486 (2)	0.038 (8)*
H1	0.212 (3)	0.462 (3)	0.0292 (17)	0.024 (6)*
H22	0.092 (3)	0.704 (3)	0.1156 (17)	0.024 (6)*
H2	0.142 (3)	0.884 (4)	0.0753 (18)	0.030 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.00992 (7)	0.01213 (8)	0.01001 (7)	-0.00125 (5)	-0.00016 (5)	-0.00229 (5)
Cl1	0.0200 (2)	0.0126 (2)	0.0151 (2)	0.00070 (16)	-0.00008 (16)	-0.00248 (16)
Cl2	0.01099 (18)	0.0271 (3)	0.0158 (2)	0.00115 (16)	-0.00235 (15)	-0.00372 (18)
Cl3	0.0220 (2)	0.0142 (2)	0.0106 (2)	-0.00484 (16)	0.00282 (16)	-0.00459 (16)
Cl4	0.01278 (18)	0.0140 (2)	0.0155 (2)	-0.00334 (15)	0.00140 (15)	-0.00161 (16)
O1	0.0146 (6)	0.0226 (7)	0.0159 (7)	-0.0034 (5)	0.0043 (5)	-0.0055 (5)
N4	0.0150 (7)	0.0162 (8)	0.0090 (8)	-0.0040 (6)	0.0002 (6)	-0.0044 (6)
N5	0.0122 (7)	0.0171 (8)	0.0110 (7)	-0.0009 (6)	0.0017 (5)	-0.0042 (6)

N6	0.0132 (7)	0.0249 (9)	0.0151 (8)	-0.0055 (6)	-0.0003 (6)	-0.0005 (7)
C5	0.0147 (8)	0.0111 (9)	0.0121 (9)	-0.0020 (6)	0.0001 (6)	-0.0013 (7)
C6	0.0144 (8)	0.0101 (8)	0.0120 (8)	-0.0014 (6)	0.0008 (6)	0.0009 (7)
C7	0.0130 (8)	0.0181 (9)	0.0129 (9)	-0.0014 (6)	0.0001 (6)	-0.0052 (7)
C8	0.0223 (9)	0.0278 (11)	0.0132 (9)	-0.0003 (8)	0.0066 (7)	-0.0072 (8)
O2	0.0242 (7)	0.0130 (7)	0.0206 (7)	0.0005 (5)	-0.0015 (5)	-0.0059 (5)
N1	0.0178 (7)	0.0111 (8)	0.0125 (8)	-0.0033 (6)	0.0006 (6)	-0.0009 (6)
C1	0.0137 (8)	0.0155 (9)	0.0130 (9)	-0.0013 (6)	-0.0040 (7)	-0.0032 (7)
C2	0.0148 (8)	0.0135 (9)	0.0102 (8)	-0.0009 (6)	0.0008 (6)	-0.0039 (7)
N2	0.0241 (8)	0.0143 (9)	0.0143 (8)	-0.0038 (6)	0.0052 (6)	-0.0039 (7)
N3	0.0170 (7)	0.0105 (8)	0.0116 (7)	-0.0018 (6)	0.0014 (6)	-0.0034 (6)
C3	0.0140 (8)	0.0125 (9)	0.0129 (9)	-0.0022 (6)	-0.0019 (6)	-0.0021 (7)
C4	0.0226 (9)	0.0115 (9)	0.0158 (9)	-0.0026 (7)	0.0017 (7)	-0.0031 (7)

Geometric parameters (Å, °)

Cd1—Cl1	2.4571 (5)	C8—H8B	0.96
Cd1—Cl4	2.4596 (4)	C8—H8C	0.96
Cd1—Cl2	2.4627 (4)	O2—C1	1.208 (2)
Cd1—Cl3	2.5678 (4)	N1—C3	1.371 (2)
Cd1—O1	2.6854 (13)	N1—C1	1.394 (2)
O1—C5	1.213 (2)	N1—H1	0.79 (3)
N4—C6	1.380 (2)	C1—C2	1.508 (3)
N4—C5	1.387 (2)	C2—N3	1.460 (2)
N4—H4	0.75 (2)	C2—H2A	0.97
N5—C6	1.319 (2)	C2—H2B	0.97
N5—C7	1.463 (2)	N2—C3	1.325 (2)
N5—C8	1.466 (2)	N2—H22	0.90 (3)
N6—C6	1.322 (2)	N2—H2	0.81 (3)
N6—H66	0.81 (3)	N3—C3	1.321 (2)
N6—H6	0.87 (3)	N3—C4	1.459 (2)
C5—C7	1.511 (2)	C4—H4A	0.96
C7—H7A	0.97	C4—H4B	0.96
C7—H7B	0.97	C4—H4C	0.96
C8—H8A	0.96		
Cl1—Cd1—Cl4	116.512 (15)	N5—C8—H8B	109.5
Cl1—Cd1—Cl2	120.122 (17)	H8A—C8—H8B	109.5
Cl4—Cd1—Cl2	117.621 (16)	N5—C8—H8C	109.5
Cl1—Cd1—Cl3	96.649 (15)	H8A—C8—H8C	109.5
Cl4—Cd1—Cl3	98.074 (15)	H8B—C8—H8C	109.5
Cl2—Cd1—Cl3	99.319 (15)	C3—N1—C1	110.39 (15)
Cl1—Cd1—O1	82.18 (3)	C3—N1—H1	127.6 (18)
Cl4—Cd1—O1	82.85 (3)	C1—N1—H1	121.8 (18)
Cl2—Cd1—O1	80.95 (3)	O2—C1—N1	125.86 (18)
Cl3—Cd1—O1	178.75 (3)	O2—C1—C2	128.57 (17)
C5—O1—Cd1	132.55 (13)	N1—C1—C2	105.55 (15)
C6—N4—C5	110.28 (15)	N3—C2—C1	102.80 (14)
C6—N4—H4	122.8 (18)	N3—C2—H2A	111.2
C5—N4—H4	125.3 (18)	C1—C2—H2A	111.2

supplementary materials

C6—N5—C7	110.56 (14)	N3—C2—H2B	111.2
C6—N5—C8	126.70 (15)	C1—C2—H2B	111.2
C7—N5—C8	122.02 (15)	H2A—C2—H2B	109.1
C6—N6—H66	120.4 (19)	C3—N2—H22	119.4 (15)
C6—N6—H6	119.0 (19)	C3—N2—H2	120.6 (19)
H66—N6—H6	117 (3)	H22—N2—H2	120 (2)
O1—C5—N4	126.58 (18)	C3—N3—C4	127.85 (15)
O1—C5—C7	127.43 (17)	C3—N3—C2	110.50 (15)
N4—C5—C7	105.99 (14)	C4—N3—C2	121.64 (15)
N5—C6—N6	126.98 (17)	N3—C3—N2	127.02 (17)
N5—C6—N4	110.34 (15)	N3—C3—N1	110.42 (15)
N6—C6—N4	122.61 (17)	N2—C3—N1	122.56 (17)
N5—C7—C5	102.69 (14)	N3—C4—H4A	109.5
N5—C7—H7A	111.2	N3—C4—H4B	109.5
C5—C7—H7A	111.2	H4A—C4—H4B	109.5
N5—C7—H7B	111.2	N3—C4—H4C	109.5
C5—C7—H7B	111.2	H4A—C4—H4C	109.5
H7A—C7—H7B	109.1	H4B—C4—H4C	109.5
N5—C8—H8A	109.5		
Cd1—Cd1—O1—C5	79.99 (16)	O1—C5—C7—N5	179.62 (18)
Cd1—Cd1—O1—C5	-38.13 (16)	N4—C5—C7—N5	-0.20 (19)
Cd1—Cd1—O1—C5	-157.67 (17)	C3—N1—C1—O2	172.47 (17)
Cd1—O1—C5—N4	2.7 (3)	C3—N1—C1—C2	-5.74 (19)
Cd1—O1—C5—C7	-177.09 (13)	O2—C1—C2—N3	-172.51 (18)
C6—N4—C5—O1	178.19 (18)	N1—C1—C2—N3	5.63 (18)
C6—N4—C5—C7	-2.0 (2)	C1—C2—N3—C3	-3.83 (19)
C7—N5—C6—N6	179.08 (18)	C1—C2—N3—C4	177.23 (15)
C8—N5—C6—N6	8.7 (3)	C4—N3—C3—N2	-1.6 (3)
C7—N5—C6—N4	-3.8 (2)	C2—N3—C3—N2	179.59 (18)
C8—N5—C6—N4	-174.16 (17)	C4—N3—C3—N1	179.35 (16)
C5—N4—C6—N5	3.7 (2)	C2—N3—C3—N1	0.5 (2)
C5—N4—C6—N6	-179.05 (17)	C1—N1—C3—N3	3.5 (2)
C6—N5—C7—C5	2.43 (19)	C1—N1—C3—N2	-175.66 (17)
C8—N5—C7—C5	173.30 (16)		

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...Cl3	0.79 (2)	2.41 (2)	3.1840 (16)	168 (2)
N2—H2...Cl3 ⁱ	0.80 (3)	2.48 (3)	3.273 (2)	170 (3)
N4—H4...Cl4	0.75 (2)	2.70 (2)	3.2967 (17)	139 (2)
N6—H6...Cl2 ⁱⁱ	0.88 (3)	2.32 (3)	3.1673 (18)	161 (3)
N2—H22...Cl1	0.90 (2)	2.34 (2)	3.2368 (18)	171 (2)
N6—H66...Cl4 ⁱⁱⁱ	0.81 (3)	2.55 (3)	3.2117 (17)	140 (2)

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

Fig. 1

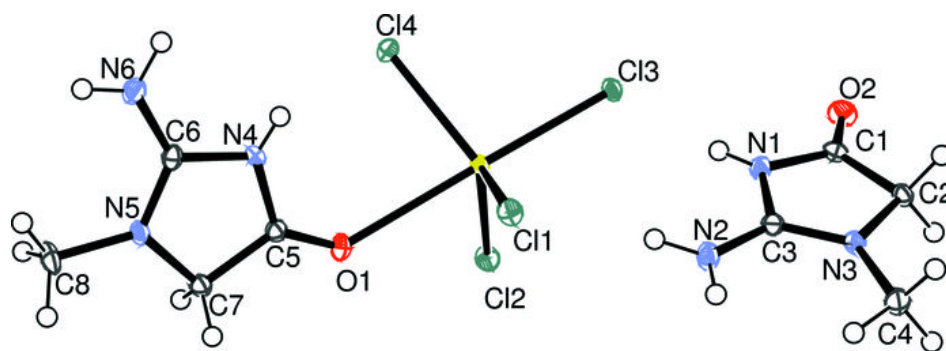


Fig. 2

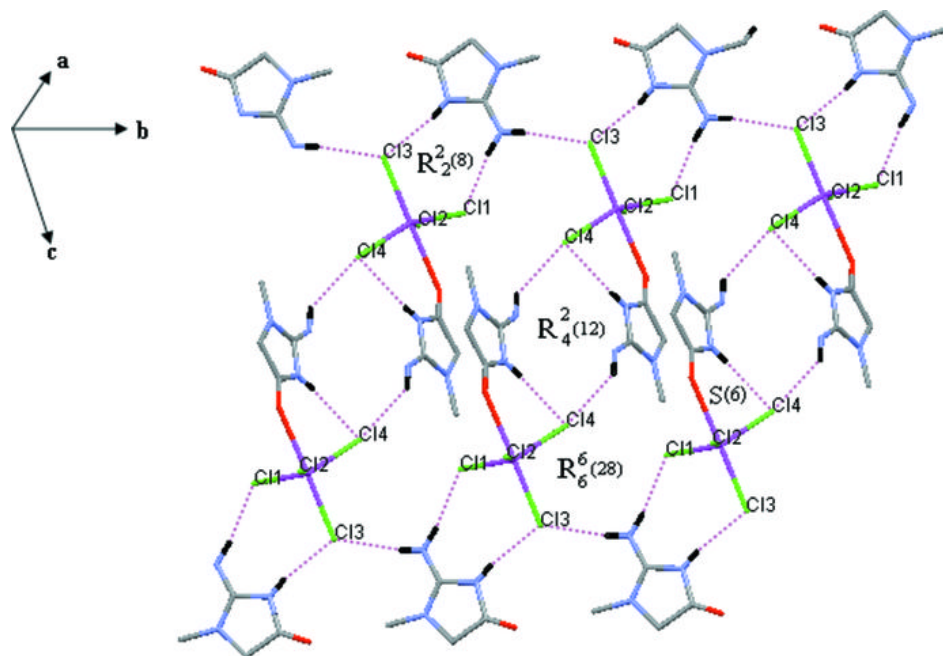


Fig. 3

