

# Poly[ethane-1,2-diammonium tetra- $\mu$ -chlorido-cadmate(II)]

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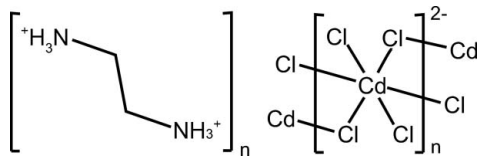
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 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.010;  $wR$  factor = 0.027; data-to-parameter ratio = 21.8.

The framework of the title compound,  $\{(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CdCl}_4]\}_n$ , is built upon layers parallel to (100) made up from corner-sharing  $[\text{CdCl}_6]$  octahedra.  $\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3^{2+}$  cations are situated between the layers and are linked to the layers via an  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen-bonding network. The Cd atom is located on an inversion centre and the coordination environment is described as highly distorted octahedral.

## Related literature

Isotypic structures have been reported by Berg & Sotofte (1976),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{PdCl}_4]$ ; Birrell & Zaslow (1972),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CuCl}_4]$ ; Tichý *et al.* (1978),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{MnCl}_4]$ ; Skaarup & Berg (1978),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{NiCl}_4]$ . For the structures of related compounds, see: Woode *et al.* (1987),  $\text{CdCl}_2\cdot\text{CH}_5\text{N}_2\text{S}\cdot\text{H}_2\text{O}$ ; Furmanova *et al.* (1996),  $\text{CdCl}_2\cdot\text{CO}(\text{NH}_2)_2$ ; Wang *et al.* (1993),  $\text{CdCl}_2\cdot\text{NH}_2\text{NHCONH}_2$ ; Cavalca *et al.* (1960),  $\text{CdCl}_2\cdot 2(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)$ . For crystallographic background, see: Becker & Coppens (1974).



## Experimental

### Crystal data

$(\text{C}_2\text{H}_{10}\text{N}_2)[\text{CdCl}_4]$   
 $M_r = 316.3$   
 Monoclinic,  $P2_1/c$   
 $a = 8.6205$  (5) Å  
 $b = 7.3425$  (8) Å  
 $c = 7.2937$  (7) Å  
 $\beta = 92.791$  (6)°

$V = 461.11$  (7) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 3.45$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.27 \times 0.13 \times 0.08$  mm

### Data collection

Oxford Diffraction Gemini (1995)  
 diffractometer with Atlas CCD  
 detector  
 $T_{\min} = 0.605$ ,  $T_{\max} = 0.841$   
 6603 measured reflections  
 Absorption correction: analytical  
 [implemented in *CrysAlis RED*  
 (Oxford Diffraction, 2008),  
 according to Clark & Reid  
 960 independent reflections  
 899 reflections with  $I > 3\sigma(I)$   
 $R_{\text{int}} = 0.023$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.010$   
 $wR(F^2) = 0.027$   
 $S = 1.08$   
 960 reflections  
 44 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.13$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.13$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Cd1—Cl1	2.6427 (5)	Cd1—Cl2	2.5585 (4)
Cd1—Cl1 <sup>i</sup>	2.6471 (5)		

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

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H3}\cdots\text{Cl1}^{\text{ii}}$	0.87	2.35	3.2123 (14)	173
$\text{N1}-\text{H4}\cdots\text{Cl2}^{\text{iii}}$	0.87	2.46	3.2824 (15)	157
$\text{N1}-\text{H5}\cdots\text{Cl2}$	0.87	2.34	3.2075 (12)	172

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2007); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2213).

## References

- Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* **A30**, 129–147.  
 Berg, R. W. & Sotofte, I. (1976). *Acta Chem. Scand. Ser. A*, **30**, 843–844.  
 Birrell, G. B. & Zaslow, B. (1972). *J. Inorg. Nucl. Chem.* **34**, 1751.  
 Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
 Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.  
 Cavalca, L., Nardelli, M. & Fava, G. (1960). *Acta Cryst.* **13**, 594–600.  
 Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* **A51**, 887–897.  
 Furmanova, N. G., Sulaimankulova, D. K., Resnyanskii, V. F. & Sulaimankulov, K. S. (1996). *Kristallografiya*, **41**, 669–672.  
 Oxford Diffraction (2005). *CrysAlis CCD*. Oxford Diffraction Ltd, Abingdon, England.  
 Oxford Diffraction (2008). *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.

Petříček, V., Dušek, M. & Palatinus, L. (2007). *JANA2006*. Institute of Physics, Praha, Czech Republic.  
Skaarup, S. & Berg, R. W. (1978). *J. Solid State Chem.* **26**, 59–67.  
Tichý, K., Beneš, J., Hälg, W. & Arend, H. (1978). *Acta Cryst.* **B34**, 2970–2981.

Wang, B.-G., Cao, Y., Zhang, H.-F. & Ye, C. (1993). *Rengong Jingti Xuebao* **22**, 341–344.  
Woode, M. K., Bryan, R. F. & Bekoe, D. A. (1987). *Acta Cryst.* **C43**, 2324–2327.

**supplementary materials**

*Acta Cryst.* (2009). E65, m215-m216 [ doi:10.1107/S1600536809002025 ]

## Poly[ethane-1,2-diammonium tetra- $\mu$ -chlorido-cadmate(II)]

A. Lamhamdi, E. Mejdoubi, K. Fejfarová, M. Dusek and B. El Bali

### Comment

Crystals of the new title compound  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CdCl}_4]$  were obtained as a side product during the preparation of a phosphate in solution. We report here on its crystal structure. Compounds including cadmium chloride and an organic moiety are frequently found in the form  $\text{CdCl}_2 \cdot X$ , where  $X$  is the organic moiety, for example:  $\text{CdCl}_2 \cdot \text{CH}_5\text{N}_2\text{S} \cdot \text{H}_2\text{O}$  (Woode *et al.*, 1987),  $\text{CdCl}_2 \cdot \text{CO}(\text{NH}_2)_2$  (Furmanova *et al.*, 1996),  $\text{CdCl}_2 \cdot \text{NH}_2\text{NHCONH}_2$  (Wang *et al.*, 1993), or  $\text{CdCl}_2 \cdot 2(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)$  (Cavalca *et al.*, 1960). The title compound, however, contains cadmium in the anionic part of the crystal structure and is isotopic with  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{PdCl}_4]$  (Berg & Sotofte, 1976),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CuCl}_4]$  (Birrell & Zaslow, 1972),  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{MnCl}_4]$  (Tichý *et al.*, 1978) and  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{NiCl}_4]$  (Skaarup & Berg, 1978).

Fig. 1 shows  $[\text{CdCl}_6]$  octahedra and the 1,2-ethanediammonium cation connected via hydrogen bonds  $\text{N1-H3} \cdots \text{Cl1}$ ,  $\text{N1-H4} \cdots \text{Cl2}$  and  $\text{N1-H5} \cdots \text{Cl2}$ . All chloride ligands of the  $\text{CdCl}_6$  octahedron participate in hydrogen bonding, as well as all hydrogens that are attached to N1.

Packing of  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CdCl}_4]$  viewed along  $a$  (Fig. 2) shows a layer of corner sharing  $[\text{CdCl}_6]$  octahedra and the neighbouring layer of 1,2-ethanediammonium cations. The minimal Cd—Cd distance within a layer is 5.1747 (8) Å.

The interlayer space is large enough to allow minimal distortions of the 1,2-ethanediammonium cation molecule, the angles and distances of which have usual values as reported in known compounds containing this cation.

### Experimental

Crystals of the title compound were obtained by mixing solutions of  $\text{K}_4\text{P}_2\text{O}_7$  (10 ml, 0.1M),  $\text{CdCl}_2$  (10 ml, 0.1M) and three drops of isopropylamine,  $(\text{CH}_3)_2(\text{CH})\text{NH}_2$ . The pH of the resulting solution was controlled with hydrochloric acid (pH = 2.5), stirred for 30 min, and then left to stand at ambient temperatures. After 5 d, colourless crystals appeared that were filtered off and washed with a solution of ethanol-water (80/20). Under the given reaction conditions isopropylamine will not convert into ethylenediamine (en), as evidenced by the structure analysis. Therefore it is most likely that the two supply bottles with isopropylamine and ethylenediamine were confused for synthesis.

### Refinement

All hydrogen atoms were discernible from difference Fourier maps and could be refined to a reasonable geometry. In the last refinement cycles they were nevertheless kept in ideal positions with N—H and C—H distances restrained to 0.87 Å and 0.96 Å, respectively, and with  $U_{\text{iso}}(\text{H}) = 1.2 \times U_{\text{eq}}$  of the respective parent atom.

## Figures

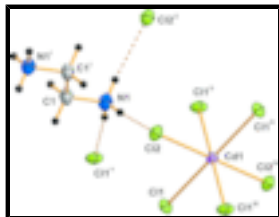


Fig. 1. Part of the structure of  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CdCl}_4]$ . Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are represented by dashed lines. [Symmetry codes: (i)  $1 - x, 1 - y, 2 - z$ ; (ii)  $x, 0.5 - y, 0.5 + z$ ; (iii)  $2 - x, -0.5 + y, 1.5 - z$ ; (iv)  $2 - x, -y, 2 - z$ ; (v)  $2 - x, 0.5 + y, 1.5 - z$ ]

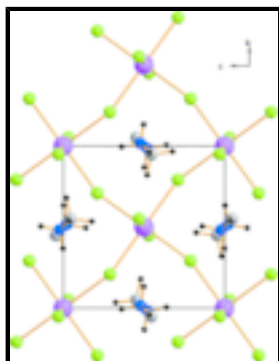


Fig. 2. Packing of  $(\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3)[\text{CdCl}_4]$  viewed along  $a$ . Color code: Pink balls (Cd), green balls (Cl), grey balls (C), blue balls (N), black balls (H).

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### Crystal data

$(\text{C}_2\text{H}_{10}\text{N}_2)[\text{CdCl}_4]$

$M_r = 316.3$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2ybc$

$a = 8.6205\ (5)\ \text{\AA}$

$b = 7.3425\ (8)\ \text{\AA}$

$c = 7.2937\ (7)\ \text{\AA}$

$\beta = 92.791\ (6)^\circ$

$V = 461.11\ (7)\ \text{\AA}^3$

$Z = 2$

$F_{000} = 304$

$D_x = 2.278\ (1)\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5814 reflections

$\theta = 2.8\text{--}26.5^\circ$

$\mu = 3.45\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Irregular shape, colourless

$0.27 \times 0.13 \times 0.08\ \text{mm}$

### Data collection

Oxford Diffraction Gemini diffractometer with Atlas CCD detector

Radiation source: X-ray tube

Monochromator: graphite

Detector resolution:  $20.7491\ \text{pixels mm}^{-1}$

$T = 298\ \text{K}$

Rotation method data acquisition using  $\omega$  scans

Absorption correction: analytical

[implemented in CrysAlis RED (Oxford Diffraction, 2008), according to Clark & Reid (1995)]

960 independent reflections

899 reflections with  $I > 3\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 26.5^\circ$

$\theta_{\text{min}} = 3.6^\circ$

$h = -10 \rightarrow 10$

$T_{\min} = 0.605$ ,  $T_{\max} = 0.841$   
6603 measured reflections

$l = -9 \rightarrow 9$

### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.010$	Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0004I^2]$
$wR(F^2) = 0.027$	$(\Delta/\sigma)_{\max} = 0.014$
$S = 1.08$	$\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$
960 reflections	$\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$
44 parameters	Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
20 constraints	Extinction coefficient: 1620 (80)

### Special details

**Refinement.** The refinement was carried out against all reflections. The conventional  $R$ -factor is always based on  $F$ . The goodness of fit as well as the weighted  $R$ -factor are based on  $F$  and  $F^2$  for refinement carried out on  $F$  and  $F^2$ , respectively. The threshold expression is used only for calculating  $R$ -factors *etc.* and it is not relevant to the choice of reflections for refinement.

The program used for refinement, Jana2006, uses the weighting scheme based on the experimental expectations, see `_refine_ls_weighting_details`, that does not force  $S$  to be one. Therefore the values of  $S$  are usually larger than the ones from the *SHELX* program.

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	1	0	1	0.01781 (5)
Cl1	1.03989 (4)	0.20870 (4)	0.71088 (4)	0.02864 (10)
Cl2	0.70621 (4)	0.05039 (5)	0.96946 (5)	0.02895 (10)
N1	0.71742 (15)	0.48402 (15)	1.0216 (2)	0.0310 (4)
C1	0.56378 (15)	0.55139 (19)	0.95400 (19)	0.0286 (4)
H3	0.789703	0.53995	0.964335	0.0372*
H4	0.729665	0.504914	1.138852	0.0372*
H5	0.723446	0.367498	1.001561	0.0372*
H1	0.55253	0.534368	0.823527	0.0344*
H2	0.555534	0.678959	0.980677	0.0344*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cd1	0.01855 (9)	0.01728 (9)	0.01754 (9)	0.00042 (4)	0.00020 (5)	0.00048 (4)
Cl1	0.03678 (18)	0.02466 (16)	0.02474 (16)	0.00197 (13)	0.00416 (13)	0.00987 (12)
Cl2	0.01873 (15)	0.03278 (18)	0.03532 (19)	0.00157 (13)	0.00114 (13)	-0.00058 (15)
N1	0.0225 (7)	0.0334 (7)	0.0369 (7)	-0.0008 (4)	-0.0004 (5)	0.0011 (5)
C1	0.0237 (7)	0.0291 (6)	0.0331 (7)	0.0005 (6)	0.0017 (6)	0.0080 (6)

## supplementary materials

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Cd1—C11	2.6427 (5)	N1—H3	0.87
Cd1—C11 <sup>i</sup>	2.6471 (5)	N1—H4	0.87
Cd1—C11 <sup>ii</sup>	2.6427 (5)	N1—H5	0.87
Cd1—C11 <sup>iii</sup>	2.6471 (5)	C1—C1 <sup>iv</sup>	1.5169 (19)
Cd1—C12	2.5585 (4)	C1—H1	0.96
Cd1—C12 <sup>ii</sup>	2.5585 (4)	C1—H2	0.96
N1—C1	1.4760 (18)		
C11—Cd1—C11 <sup>i</sup>	91.326 (12)	C12—Cd1—C12 <sup>ii</sup>	180
C11—Cd1—C11 <sup>ii</sup>	180	Cd1—C11—Cd1 <sup>v</sup>	156.050 (14)
C11—Cd1—C11 <sup>iii</sup>	88.674 (12)	C1—N1—H3	109.471
C11—Cd1—C12	90.766 (12)	C1—N1—H4	109.4712
C11—Cd1—C12 <sup>ii</sup>	89.234 (12)	C1—N1—H5	109.4713
C11 <sup>i</sup> —Cd1—C11 <sup>ii</sup>	88.674 (12)	H3—N1—H4	109.4717
C11 <sup>i</sup> —Cd1—C11 <sup>iii</sup>	180	H3—N1—H5	109.471
C11 <sup>i</sup> —Cd1—C12	88.066 (11)	H4—N1—H5	109.4711
C11 <sup>i</sup> —Cd1—C12 <sup>ii</sup>	91.934 (11)	N1—C1—C1 <sup>iv</sup>	110.09 (11)
C11 <sup>ii</sup> —Cd1—C11 <sup>iii</sup>	91.326 (12)	N1—C1—H1	109.4717
C11 <sup>ii</sup> —Cd1—C12	89.234 (12)	N1—C1—H2	109.4714
C11 <sup>ii</sup> —Cd1—C12 <sup>ii</sup>	90.766 (12)	C1 <sup>iv</sup> —C1—H1	109.4709
C11 <sup>iii</sup> —Cd1—C12	91.934 (11)	C1 <sup>iv</sup> —C1—H2	109.4709
C11 <sup>iii</sup> —Cd1—C12 <sup>ii</sup>	88.066 (11)	H1—C1—H2	108.8487

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

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H3 $\cdots$ C11 <sup>v</sup>	0.87	2.35	3.2123 (14)	173
N1—H4 $\cdots$ C12 <sup>iii</sup>	0.87	2.46	3.2824 (15)	157
N1—H5 $\cdots$ C12	0.87	2.34	3.2075 (12)	172

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

Fig. 1

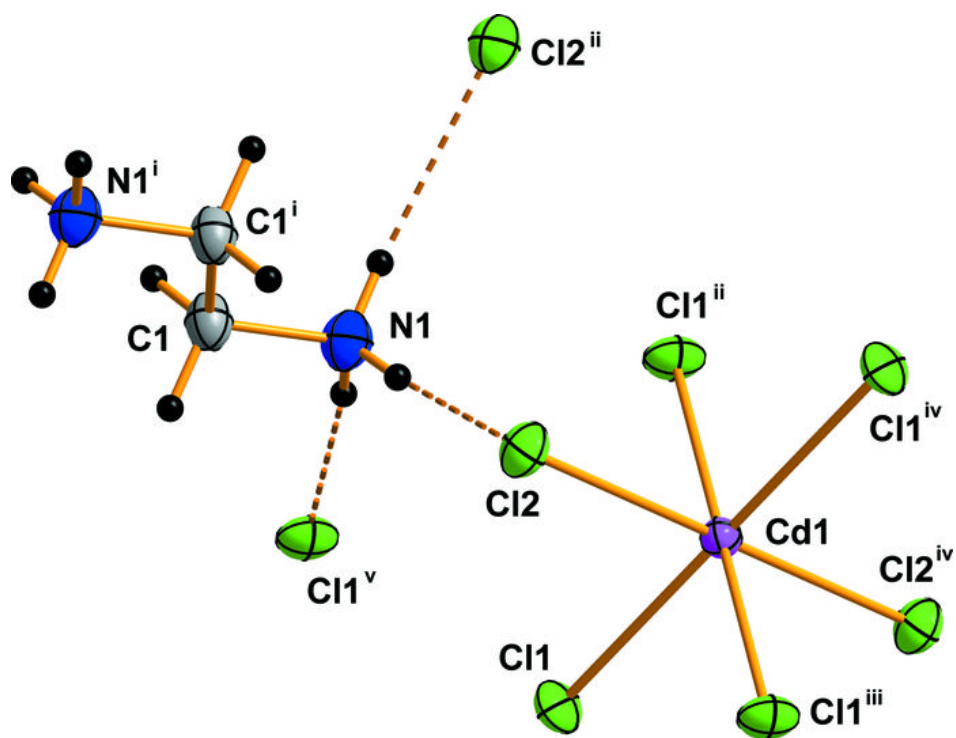


Fig. 2

