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Structure of 1,10-Diaminodecane Tetrachlorozincate

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Abstract. $[\text{NH}_3(\text{CH}_2)_{10}\text{NH}_3][\text{ZnCl}_4]$, $M_r = 381.51$, triclinic, $P\bar{1}$, $a = 7.296\,(1)$, $b = 10.110\,(3)$, $c = 12.814\,(4)$ Å, $\alpha = 90.84\,(2)$, $\beta = 101.17\,(2)$, $\gamma = 92.52\,(2)°$, $V = 926.13$ Å^3, $Z = 2$, $D_x = 1.37$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.71073$ Å, $\mu = 1.925$ mm^{-1}, $F(000) = 396$, $T = 298$ K, final $R = 0.070$ for 1237 unique reflections $[I > 3\sigma(I)]$. The structure is characterized by layers of inorganic ions sandwiched between layers formed by the paraffinic chains.

Introduction. The alkyldiammonium tetrahalometallates of general formula $[\text{NH}_3(\text{CH}_2)_n\text{NH}_3]$-

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Table 1. *Fractional coordinates and equivalent isotropic thermal displacement parameters* (Å^2 × 10^3)

$U_{eq} = (8\pi^2/3)\text{trace } \mathbf{U}$.

	x	*y*	*z*	U_{eq}
Zn	2549 (3)	2221 (2)	4651 (2)	60 (1)
Cl(1)	−135 (5)	3292 (4)	4275 (4)	71 (2)
Cl(2)	4608 (6)	3335 (4)	3808 (4)	76 (2)
Cl(3)	2038 (7)	94 (4)	4067 (4)	76 (2)
Cl(4)	3752 (6)	2342 (4)	6430 (3)	69 (2)
N(1)	7136 (17)	769 (12)	3460 (9)	66 (6)
N(2)	7656 (17)	3846 (12)	6195 (11)	70 (6)
C(1)	7435 (22)	876 (17)	2316 (13)	79 (8)
C(2)	5729 (20)	1247 (16)	1569 (13)	77 (8)
C(3)	4142 (19)	264 (16)	1412 (13)	72 (7)
C(4)	2466 (21)	769 (17)	641 (14)	88 (9)
C(5)	870 (21)	−238 (19)	399 (13)	89 (9)
C(6)	14504 (75)	4406 (32)	9893 (45)	312 (38)
C(7)	12908 (50)	4886 (28)	9195 (31)	228 (27)
C(8)	11219 (46)	3941 (26)	8796 (21)	203 (21)
C(9)	9590 (52)	4238 (29)	7879 (26)	294 (29)
C(10)	8951 (32)	3485 (22)	7098 (20)	128 (13)

Table 2. *Bond lengths* (Å) *and angles* (°)

E.s.d.'s in the least significant digits are given in parentheses.

Zn—Cl(1)	2.253 (4)	Zn—Cl(2)	2.282 (5)
Zn—Cl(3)	2.257 (5)	Zn—Cl(4)	2.278 (5)
N(1)—C(1)	1.528 (22)	N(2)—C(10)	1.409 (26)
C(1)—C(2)	1.482 (21)	C(2)—C(3)	1.473 (21)
C(3)—C(4)	1.527 (21)	C(4)—C(5)	1.495 (23)
C(5)—C(5^{i})	1.567 (30)	C(6)—C(7)	1.432 (59)
C(6)—C(6^{ii})	1.369 (77)	C(7)—C(8)	1.527 (43)
C(8)—C(9)	1.545 (43)	C(9)—C(10)	1.250 (38)
Cl(1)—Zn—Cl(2)	106.0 (2)	Cl(1)—Zn—Cl(3)	109.9 (2)
Cl(2)—Zn—Cl(3)	111.5 (2)	Cl(1)—Zn—Cl(4)	110.6 (2)
Cl(2)—Zn—Cl(4)	107.6 (2)	Cl(3)—Zn—Cl(4)	111.0 (2)
N(1)—C(1)—C(2)	112.6 (14)	C(1)—C(2)—C(3)	115.8 (14)
C(2)—C(3)—C(4)	110.8 (14)	C(3)—C(4)—C(5)	112.5 (14)
C(4)—C(5)—C(5^{i})	114.0 (19)	C(7)—C(6)—C(6^{ii})	97.6 (39)
C(6)—C(7)—C(8)	119.3 (27)	C(7)—C(8)—C(9)	124.4 (25)
C(8)—C(9)—C(10)	125.5 (28)	N(2)—C(10)—C(9)	124.6 (23)

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

[MX_4] (M = Mn, Cu, Cd, ...; X = Cl, Br) are known to crystallize in layer structures (Phelps, Losee, Hatfield & Hodgson, 1976; Willet & Riede, 1975; Peterson & Willet, 1972). These compounds show a large variety of phase transitions (Carla, Giuseppe & Guiseppe, 1986) which are governed by the dynamics of the alkylammonium groups and consequently they exhibit interesting electric and magnetic properties which may be dependent on the spacing between the metal ions. Depending on the length of the chains, different structures and chain packings are possible. Intercalation of the chains was found only for compounds with M = Co, Zn (Ciajolo, Corradini & Pavone, 1977). Up to now, no structure of a long-chain alkyldiammonium tetrahalometallate has been reported. We have therefore determined the crystal structure of 1,10-diaminodecane tetrachlorozincate (DDAZn) in order to compare it with those of other compounds.

Experimental. DDAZn was prepared by mixing aqueous solutions of *n*-decyldiammonium chloride and zinc chloride in stoichiometric amounts. A single crystal of dimensions 0.04 × 0.38 × 0.40 mm was selected for structure determination. Intensity data were collected using a Nicolet *R3M/E* diffractometer. Cell parameters were obtained by a least-squares method using 25 centred reflections with $6.83 < 2\theta < 23.33°$. Data were collected within $3 < 2\theta < 48°$ using the ω-scan method and were corrected for Lorentz–polarization and absorption effects (transmission coefficients minimum 0.227, maximum 0.337). The range for h was 0 to 9, for k −12 to 12 and for l −15 to 15. The intensity variation of a standard reflection ($1\bar{2}\bar{1}$) was ±2% about the mean value. The main computer program used was *SHELXTL* (Sheldrick, 1983). Of the 3242 reflections measured, 2899 were independent (R_{int} = 0.030) of which 1237 were observed [$I > 3\sigma(I)$] and were used in the refinement. The structure was solved by Patterson methods. Full-matrix least-squares refinements on F of positional and anisotropic

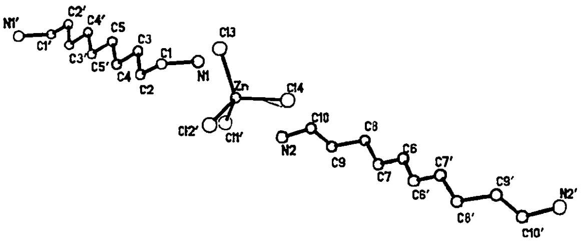

Fig. 1. Atomic numbering scheme for the non-hydrogen atoms of DDAZn.

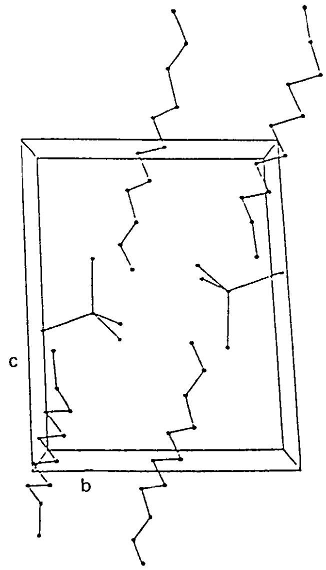

Fig. 2. View of the unit cell for DDAZn.

thermal parameters. Hydrogen atoms were placed in calculated positions and were assigned isotropic thermal parameters $U = 0.08$ Å^2. Final $R = 0.070$, $wR = 0.054$, maximum $\Delta/\sigma = 0.001$, $w = [\sigma^2(F)]^{-1}$. Maximum, minimum $\Delta\rho$ values in final difference synthesis 1.01, -0.52 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Atomic fractional coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are listed in Table 1.* Fig. 1. shows the atomic numbering scheme of the non-hydrogen atoms. Bond lengths and angles are given in Table 2. A view of the unit-cell contents is shown in Fig. 2. The structure contains two independent cations, each with crystallographically imposed $\bar{1}$ symmetry. Like $(C_{12}H_{25}NH_3)_2[ZnCl_4]$ (Ciajolo, Corradini & Pavone, 1977), the structure of DDAZn differs from that of $[NH_3(CH_2)_3NH_3][SnCl_4]$ (Kallet, Fall & Daoud, 1980) at room temperature in several ways. Crystals of $[NH_3(CH_2)_3NH_3][ZnCl_4]$ are monoclinic, space group $P2_1/n$ with $Z = 4$, whereas those of the title compound are triclinic, space group $P\bar{1}$ with $Z = 2$. As shown in Fig. 1 the cation containing C(6)—C(10) displays anomalous C—C bond lengths and U_{eq} values for its carbon atoms are physically unreasonable, probably as a result of disorder.

The tetrahedral $ZnCl_4^{2-}$ anions which form two-dimensional sheets parallel to 001 are sandwiched between hydrocarbon layers which consist of $[NH_3(CH_2)_{10}NH_3]^{2+}$ cations, as shown in Fig. 2. The average Zn—Cl bond length is 2.27 Å which accords with the corresponding value for $(n\text{-}C_{12}H_{25}NH_3)_2$-$[ZnCl_4]$. All the Cl—Zn—Cl angles deviate by less than 4° fom 109.5° indicating a small distortion from tetrahedral symmetry. Since the shorter N···Cl distances range from 3.19 to 3.60 Å the $ZnCl_4$ tetrahedron may be connected to the 1,10-decanediammonium cation by medium to very weak N—H···Cl hydrogen bonds. However, the hydrogen bonds appear stronger in DDAZn than in $[NH_3(CH_2)_3NH_3][ZnCl_4]$ judging from the minimum N···Cl distance in each structure.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54515 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0179]

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Structure of a Copper Complex of an α-Hydroxylated Acid: $[Cu(C_{14}H_8O_3)(C_{12}H_8N_2)]_2.2H_2O$

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Abstract. Bis[μ-(9-hydroxy-9H-fluorene-9-carboxylato-O,μ-O')]-bis(1,10-phenanthroline)copper(II)], $M_r = 971{\cdot}97$, monoclinic, $P2_1/n$, $a = 9{\cdot}101$ (2), $b = 20{\cdot}681$ (3), $c = 11{\cdot}101$ (1) Å, $\beta = 93{\cdot}55$ (2)°, $V = 2085$ Å^3, $Z = 2$, $D_x = 1{\cdot}55$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11{\cdot}29$ cm^{-1}, $F(000) = 996$, $T = 297$ K, $R = 0{\cdot}039$ for 3064 observed reflections. Every Cu atom is square pyramidally coordinated by three O atoms and two N atoms. The two Cu atoms are connected to two bridging hydroxyl O atoms to

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