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Acta Cryst. (1995). C51, 617–619

Bis(*n*-heptylammonium) Tetrachlorozincate

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(Received 16 July 1993; accepted 8 August 1994)

Abstract

The crystal structure of $(C_7H_{18}N)_2[ZnCl_4]$ is composed of alternating layers of inorganic ions and paraffinic chains. It differs from the structures of analogous compounds that have a hydrocarbon chain containing an even number of C atoms. This may explain the odd–even effects observed in differential scanning calorimetry (DSC) measurements on these compounds.

Comment

Alkylammonium tetrahalometallates of general formula $(n-C_nH_{2n+1}NH_3)_2[MX_4]$ ($M = Mn, Cu, Cd, etc.$; $X = Cl, Br$) are known to form crystal structures in which

hydrocarbon layers are sandwiched between inorganic sheets (Phelps, Losee, Hatfield & Hodgson, 1976; Willet & Riedel, 1975; Peterson & Willet, 1972). DSC measurements indicate that the compounds of the series show a great variety of structural phases (Carla, Guiseppe & Guiseppe, 1986) and exhibit an odd–even effect as a function of the number of C atoms in the alkyl chain. This effect may depend on the structural differences between the odd and even compounds of the series. The structure of $(n-C_{12}H_{25}NH_3)_2[ZnCl_4]$ ($C_{12}Zn$) has been reported (Ciajalo, Corradini & Pavone, 1977). It is the aim of this paper to report the single-crystal room-temperature structure determination of the title compound (HAZn) in order to explain the odd–even effects observed in the DSC measurements.

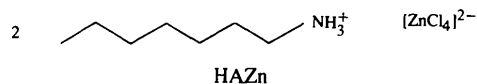


Fig. 1 shows the numbering scheme for the non-H atoms and a view of the unit cell is presented in Fig. 2. The structure consists of organic and inorganic layers. Each inorganic layer of tetrahedral $ZnCl_4^{2-}$ anions is sandwiched between two hydrocarbon layers formed by $C_7H_{15}NH_3^+$ cations (Fig. 2). Each tetrahedral $ZnCl_4^{2-}$ anion is surrounded by six NH_3 groups from adjacent sheets. The $ZnCl_4^{2-}$ tetrahedra show angles from 104.7 (4) to 113.3 (5)°, which indicate a small distortion from the normal value. The distortion probably results from the formation of hydrogen bonds between N and Cl atoms. The average Zn–Cl bond length is 2.26 \AA , which is slightly shorter than the value of 2.27 \AA in $[NH_3-(CH_2)_{10}-NH_3][ZnCl_4]$ (Guo, Lin, Zeng & Xi, 1992).

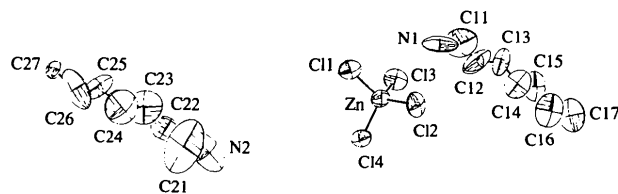


Fig. 1. Atomic numbering of the non-H atoms of the title compound shown with 50% probability ellipsoids.

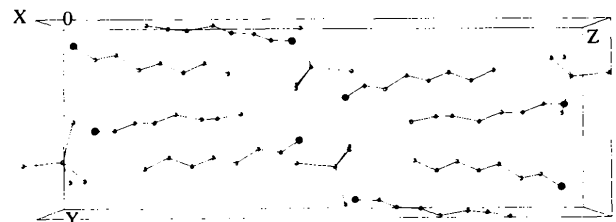


Fig. 2. View of the unit cell with N atoms shown as large filled circles.

Each C₇H₁₅NH₃⁺ group displays N—C—C—C and C—C—C—C torsion angles close to 180°, except for the *gauche* bond [C(22)—C(23)], near the NH₃ polar heads (see Table 2). A similar conformation is found in (*n*-C₁₀H₂₁NH₃)₂[MnCl₄] (Ciajolo, Corradini & Pavone, 1976). The chains show some evidence of disorder which may be related to the solid–solid phase transition which occurs below room temperature (Guo, Lin, Zeng & Xi, 1991). The atoms adjacent to the N atoms have larger anisotropic displacement parameters and consequently large positional standard deviations.

The packing of HAZn is different from that of C₁₂Zn. The aliphatic chains in different layers of C₁₂Zn are parallel, being related by centres of inversion, but in HAZn the aliphatic chains in different layers are not parallel, as was also found in (*n*-C₁₃H₂₇NH₃)₂[ZnBr₄] (Avitabile, Ciajolo, Napolitano & Tuzi, 1983).

Experimental

HAZn was prepared by mixing aqueous solutions of *n*-heptylammonium chloride and zinc chloride in stoichiometric amounts.

Crystal data

(C₇H₁₈N)₂[ZnCl₄]

M_r = 439.64

Orthorhombic

*P*2₁*nb*

a = 7.447 (1) Å

b = 10.387 (2) Å

c = 30.082 (10) Å

V = 2326.9 (9) Å³

Z = 4

D_x = 1.255 Mg m⁻³

D_m = 1.23 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25

reflections

θ = 1.5–17.5°

μ = 1.546 mm⁻¹

T = 298 K

Plate

0.30 × 0.28 × 0.12 mm

Colourless

Data collection

Nicolet R3m/E diffractometer

ω scans

Absorption correction:

empirical (Sheldrick, 1983)

T_{min} = 0.530, *T_{max}* =

0.650

2451 measured reflections

1304 independent reflections

503 observed reflections

[*I* > 3σ(*I*)]

R_{int} = 0.0370

θ_{max} = 17.5°

h = 0 → 9

k = 0 → 13

l = 0 → 36

1 standard reflection

monitored every 100

reflections

intensity decay: <2%

Refinement

Refinement on *F*

R = 0.053

wR = 0.045

S = 1.181

503 reflections

190 parameters

w = 1/[σ²(*F*) + 0.00026*F*²]

(Δ/σ)_{max} = 0.088

Δρ_{max} = 0.28 e Å⁻³

Δρ_{min} = -0.23 e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Zn	0.0207 (10)	-0.2290 (2)	0.5225 (1)	0.091 (1)
Cl(1)	-0.0071 (15)	-0.2488 (5)	0.4481 (2)	0.132 (2)
Cl(2)	0.2712 (8)	-0.3261 (6)	0.5478 (2)	0.114 (2)
Cl(3)	-0.2192 (10)	-0.3371 (6)	0.5508 (2)	0.133 (2)
Cl(4)	0.026 (2)	-0.0206 (5)	0.5416 (2)	0.129 (2)
N(1)	0.017 (4)	-0.614 (1)	0.5429 (5)	0.179 (3)
C(11)	-0.048 (4)	-0.684 (3)	0.5795 (9)	0.253 (3)
C(12)	-0.005 (4)	-0.663 (2)	0.6195 (7)	0.217 (3)
C(13)	-0.024 (3)	-0.738 (2)	0.6604 (7)	0.190 (3)
C(14)	0.057 (3)	-0.708 (2)	0.7016 (7)	0.190 (3)
C(15)	-0.026 (3)	-0.754 (2)	0.7419 (9)	0.187 (3)
C(16)	0.066 (3)	-0.708 (3)	0.7823 (8)	0.221 (3)
C(17)	0.057 (4)	-0.763 (2)	0.8257 (7)	0.211 (3)
N(2)	0.009 (3)	0.478 (2)	0.3401 (7)	0.316 (3)
C(21)	0.095 (3)	0.504 (3)	0.2966 (9)	0.474 (3)
C(22)	-0.037 (3)	0.506 (2)	0.2640 (8)	0.162 (3)
C(23)	-0.029 (3)	0.479 (3)	0.2139 (7)	0.221 (3)
C(24)	0.063 (4)	0.524 (3)	0.1765 (11)	0.308 (3)
C(25)	-0.036 (3)	0.525 (2)	0.1342 (7)	0.174 (3)
C(26)	0.091 (3)	0.568 (2)	0.1012 (10)	0.215 (3)
C(27)	0.011 (4)	0.568 (2)	0.0600 (5)	0.104 (3)

Table 2. Selected geometric parameters (Å, °)

Zn—Cl(1)	2.258 (5)	Zn—Cl(2)	2.252 (9)
Zn—Cl(3)	2.275 (9)	Zn—Cl(4)	2.240 (5)
N(1)—C(11)	1.40 (3)	C(11)—C(12)	1.28 (4)
C(12)—C(13)	1.48 (3)	C(13)—C(14)	1.41 (3)
C(14)—C(15)	1.44 (3)	C(15)—C(16)	1.48 (4)
C(16)—C(17)	1.43 (3)	N(2)—C(21)	1.48 (3)
C(21)—C(22)	1.39 (3)	C(22)—C(23)	1.54 (3)
C(23)—C(24)	1.40 (4)	C(24)—C(25)	1.47 (4)
C(25)—C(26)	1.44 (3)	C(26)—C(27)	1.37 (3)
Cl(1)—Zn—Cl(2)	111.7 (4)	Cl(1)—Zn—Cl(3)	104.7 (4)
Cl(2)—Zn—Cl(3)	107.7 (2)	Cl(1)—Zn—Cl(4)	110.1 (2)
Cl(2)—Zn—Cl(4)	109.3 (5)	Cl(3)—Zn—Cl(4)	113.3 (5)
N(1)—C(11)—C(12)	123 (2)	C(11)—C(12)—C(13)	130 (2)
C(12)—C(13)—C(14)	123 (2)	C(13)—C(14)—C(15)	119 (2)
C(14)—C(15)—C(16)	113 (2)	C(15)—C(16)—C(17)	127 (2)
N(2)—C(21)—C(22)	109 (2)	C(21)—C(22)—C(23)	132 (2)
C(22)—C(23)—C(24)	138 (2)	C(23)—C(24)—C(25)	117 (2)
C(24)—C(25)—C(26)	106 (2)	C(25)—C(26)—C(27)	110 (2)
N(1)—C(11)—C(12)—C(13)	-165 (3)		
C(11)—C(12)—C(13)—C(14)	170 (3)		
C(12)—C(13)—C(14)—C(15)	155 (2)		
C(13)—C(14)—C(15)—C(16)	-176 (2)		
C(14)—C(15)—C(16)—C(17)	-159 (3)		
N(2)—C(21)—C(22)—C(23)	-154 (3)		
C(21)—C(22)—C(23)—C(24)	-57 (5)		
C(22)—C(23)—C(24)—C(25)	-146 (3)		
C(23)—C(24)—C(25)—C(26)	-177 (3)		
C(24)—C(25)—C(26)—C(27)	178 (2)		

The structure was solved by Patterson methods. Successive Fourier syntheses gave the coordinates of all non-H atoms. The structure was refined using a full-matrix least-squares technique with anisotropic displacement parameters for all non-H atoms. H atoms were placed in calculated positions and were assigned isotropic displacement parameters (*U*_{iso} = 0.05 Å²). All calculations were performed on an Eclipse S/140 computer using the *SHELXTL* (Sheldrick, 1983) program package.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 619–621

***cis*-[1,2-Bis(propylsulfinyl)ethane-*S,S'*]-dichloroplatinum(II)**

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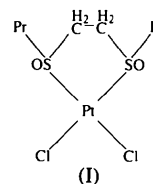
(Received 4 October 1993; accepted 25 August 1994)

Abstract

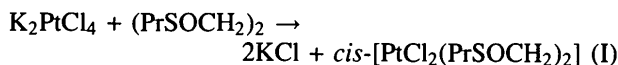
The coordination geometry about Pt in the title complex, [PtCl₂(C₈H₁₈O₂S₂)], deviates slightly from square planar. The Pt ion is bonded to two Cl atoms, arranged in a *cis* configuration, and two S atoms of the bidentate disulfoxide ligand.

Comment

The title complex, (I), was synthesized as part of a research program on the organometallic chemistry of



tin and the behaviour of these compounds towards Pt and Pd complexes. The synthesis was carried out in H₂O/MeOH solution, with a yield of 85%, as follows:



The average Pt—Cl bond length of 2.317 Å is in the normal range, according to the compilation of Orpen *et al.* (1989), which gives a value of 2.323 (38) Å for 293 examples of four-coordinate Pt. The disulfoxide ligand is bonded to the Pt atom at two coordination sites through the S atoms. The average Pt—S distance of 2.209 Å is not significantly different from the values found in *cis*-[PtCl₂(*meso*-PhSOCH₂CH₂SOPh)] [2.217 (2) and 2.209 (2) Å] and *cis*-[PtCl₂(*rac*-PhSOCH₂CH₂SOPh)] [2.192 (4) and 2.188 (4) Å] (Cattalini, Michelon, Marangoni & Pelizzi, 1979), and *cis*-[PtCl₂(*rac-cis*-PhSO-

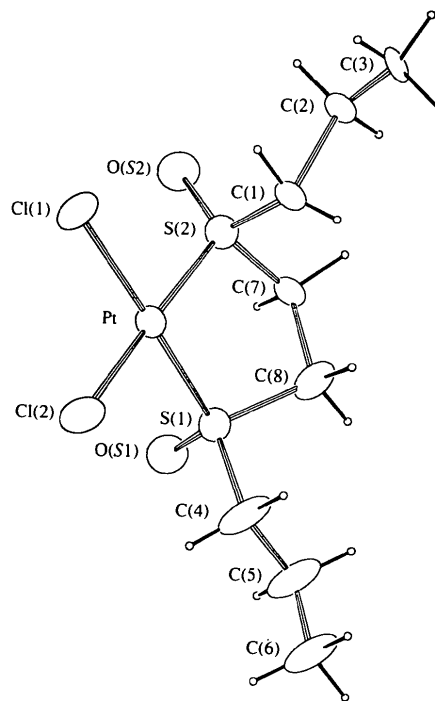


Fig. 1. A perspective view of the title molecule with atom numbering. Displacement ellipsoids are drawn at the 50% probability level.