Data collection

Nicolet $R3$ diffractometer
ω scans
Absorption correction:
  ψ scans (XEMP; Nicolet, 1987)
  $T_{min} = 0.608$, $T_{max} = 0.710$
3627 measured reflections
2185 independent reflections
2156 reflections with
  $I > 2\sigma(I)$

$R_{int} = 0.016$
$\theta_{max} = 27.56°$
$h = -21 \rightarrow 21$
$k = 0 \rightarrow 14$
$l = -13 \rightarrow 13$
3 standard reflections
  every 147 reflections
  intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.044$
$S = 1.123$
2185 reflections
103 parameters
Methyl H atoms rigid, other
  H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 1.2752P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.303$ e Å^{-3}
$\Delta\rho_{min} = -0.339$ e Å^{-3}
Extinction correction: none
Scattering factors from
  International Tables for
  Crystallography (Vol. C)
Absolute structure:
  Flack (1983), number of
  Friedel pairs = 1086
Flack parameter = -0.04 (3)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Mo	1/2	0.68595 (2)	0.50001 (2)	0.01559 (6)
Cl1	0.61002 (3)	0.63596 (5)	0.64051 (5)	0.02650 (11)
Cl2	1/2	0.87882 (6)	0.61846 (7)	0.02695 (15)
C1	1/2	0.8393 (3)	0.3298 (3)	0.0193 (5)
C2	0.56984 (11)	0.76648 (18)	0.31125 (18)	0.0196 (4)
C3	0.54265 (12)	0.64616 (18)	0.28640 (18)	0.0208 (4)
C4	0.65525 (12)	0.81059 (18)	0.3039 (2)	0.0238 (4)
C5	0.67372 (16)	0.9056 (2)	0.4109 (3)	0.0411 (6)
C6	0.66522 (14)	0.8704 (3)	0.1658 (2)	0.0402 (6)
C7	0.71309 (14)	0.7026 (2)	0.3160 (3)	0.0355 (5)
C8	1/2	0.3833 (3)	0.4639 (3)	0.0242 (6)
C9	1/2	0.2508 (3)	0.4382 (3)	0.0374 (8)
N	1/2	0.4857 (2)	0.4827 (3)	0.0230 (5)

Table 2. *Selected geometric parameters (Å, °)*

Mo—N	2.182 (2)	Mo—Cent	2.023
Mo—Cl1	2.3909 (6)	C8—N	1.127 (4)
Mo—Cl2	2.4084 (9)	C8—C9	1.463 (5)
N—Mo—Cl1	79.67 (4)	Cl2—Mo—Cent	105.0
Cl1—Mo—Cl1^i	101.59 (3)	N—Mo—Cent	100.0
N—Mo—Cl2	154.99 (7)	N—C8—C9	179.5 (3)
Cl1—Mo—Cl2	84.62 (2)	C8—N—Mo	174.9 (2)
Cl1—Mo—Cent	128.9		

Symmetry code: (i) $1 - x, y, z$.

The origin was fixed according to the method of Flack & Schwarzenbach (1988).

Data collection: *P3 Software* (Nicolet, 1987). Cell refinement: *P3 Software*. Data reduction: *XDISK* (Nicolet, 1987). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL93*.

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(C6H5CH2CH2NH3)2[CdCl4]

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## Abstract

The title compound, bis(β-phenylethylammonium) tetrachlorocadmate, $(C_8H_{12}N)_2[CdCl_4]$, crystallizes as a layer structure; the heavy atoms form a two-dimensional network of corner-connected octahedra, the organic cation being connected to the network through hydrogen bonding.

Comment

This crystal, (I), was investigated as part of a series of heavy-atom halides and organic cations (Zouari, Ben Salah, Daoud, Rothammel & Burzlaff, 1993) to obtain

information on the coordination environment of Cd^{II} and other elements of the same group in the periodic system (Knop, Cameron, Adhikesa Valu, Vincent & Jenkins, 1987).

(I)
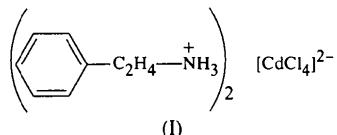
The asymmetric unit contains one Cd atom, two Cl atoms, Cl1 and Cl2, and one β-phenylethylamine group (βP). The Cd atom lies on the special position (000) with site symmetry $\bar{1}$ and is coordinated by Cl atoms in a nearly undistorted octahedral arrangement. Cl2 lies in a general position connecting two Cd atoms, thus resulting in a two-dimensional network of corner-connected centered squares close to the plane (0yz). Cl1 augments each centered square to an octahedron. Pairs of βP moieties arranged perpendicular to the plane of the connected octahedra center those squares not occupied by Cd atoms. The N atom is connected to the Cl atoms via hydrogen bonding (Fig. 1 and Table 3). The Cd—Cl distances are in good agreement with those reported in the literature.

The structure is a severe superstructure nearly showing the translational symmetry of an oF cell (cf Table 4). The peak-hunting routine observed only the (212) re-

flection in the range $5 < \theta < 15°$ with a counting rate larger than 5σ. Thus, the structure determination was started with the wrong space group $Bmab$. The solution was not compatible, however, with the presence of an m mirror plane, also other space groups with a B cell led to too short distances for atoms between different organic molecules. Several atoms showed unreasonable behavior in their displacement ellipsoids, in agreement with this observation. A reasonable model demanded a glide plane c; a control of the integral extinctions led to the proper oP cell and to the space group No. 61 $Pcab$, a subgroup of $Bmab$. This space group is compatible with the distribution of the βP group in the unit cell. An additional measurement of the weak non-F reflections, which are mainly caused by parts of the organic cation, confirmed the space group $Pcab$ and led to the final solution of the structure.

There is a strong structural relation to the analogous copper halide compound (Willet, 1990), in fact, they are isostructural. The local neighborhood, however, differs significantly in two properties: (a) the layer of corner-connected octahedra is an antiferrodistortive layer with one short and one long distance [2.286 (1) and 2.899 (1) Å] in the case of CuCl$_4^{2-}$ within the plane and a short distance [2.296 (1) Å] perpendicular to it, whereas the CdCl$_4^{2-}$ layer contains nearly equivalent distances in the plane and perpendicular to it (cf Table 2), and (b) as a consequence there is a change with respect to the hydrogen bondings in the CuCl$_4^{2-}$ octahedral layer, there are two hydrogen bonds to bridging halide ions and one to a terminal halide ion. In the CdCl$_4^{2-}$ octahedral layer, it is the opposite way around. Fig. 1 shows the displacement ellipsoids and Table 4 presents the distribution of $|F|^2(hkl)$ on parity classes.

Experimental

Fig. 1. Displacement ellipsoids of 50% probability and the hydrogen bonding between the N and Cl atoms

Crystal data

(C$_8$H$_{12}$N)$_2$[CdCl$_4$]	Mo $K\alpha_1$ radiation
$M_r = 498.59$	$\lambda = 0.70926$ Å
Orthorhombic	Cell parameters from 28
$Pcab$	reflections
$a = 38.846$ (2) Å	$\theta = 12.5–15.7°$
$b = 7.434$ (1) Å	$\mu = 1.530$ mm^{-1}
$c = 7.367$ (1) Å	$T = 293$ K
$V = 2127.5$ (4) Å^3	Truncated pyramid
$Z = 4$	0.194 (5) × 0.168 (5) ×
$D_x = 1.557$ Mg m^{-3}	0.130 (5) mm
D_m not measured	Colorless
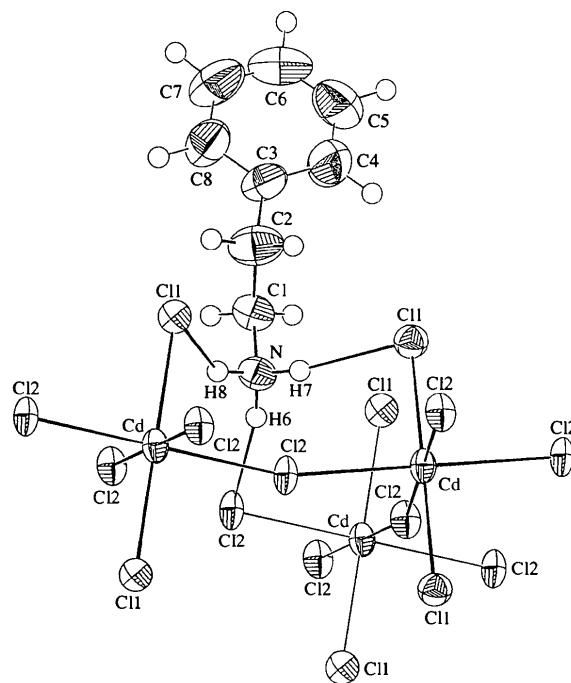
Data collection

Rebuilt Philips PW1100 (Gomm, 1993) diffractometer	1076 reflections with $F^2 > 2.5\sigma(F^2)$
$\theta/2\theta$ scans	$R_{int} = 0.105$
Absorption correction:	$\theta_{max} = 25.0°$
Gaussian by integration	$h = 0 \rightarrow 46$
(Busing & Levy, 1957)	$k = -8 \rightarrow 8$
$T_{min} = 0.813$, $T_{max} = 0.833$	$l = -8 \rightarrow 8$
7648 measured reflections	6 standard reflections
1885 independent reflections	every 50 reflections intensity decay: 3.8%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.61$ e Å^{-3}
$wR(F^2) = 0.039$	$\Delta\rho_{min} = -0.73$ e Å^{-3}
$S = 0.95$	Extinction correction:
1076 reflections	Larson (1970)
108 parameters	Extinction coefficient:
H atoms not refined	33. (2) $\times 10^2$
$w = 1/[\sigma^2(F^2{}_{obs}) + 0.001F^2{}_{obs} + 10]$	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
$(\Delta/\sigma)_{max} < 0.001$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cd	0	0	0	0.0322 (1)
Cl1	0.06491 (3)	−0.0134 (3)	0.0477 (2)	0.0444 (4)
Cl2	−0.00802 (4)	0.2256 (2)	0.2753 (2)	0.0387 (5)
N	0.0589 (1)	0.5290 (8)	0.0111 (7)	0.047 (2)
C1	0.0942 (1)	0.4657 (9)	−0.0422 (9)	0.058 (3)
C2	0.1206 (2)	0.5815 (9)	0.036 (1)	0.081 (3)
C3	0.1569 (1)	0.505 (2)	0.012 (1)	0.070 (2)
C4	0.1691 (2)	0.376 (1)	0.129 (2)	0.104 (5)
C5	0.2022 (3)	0.311 (1)	0.110 (2)	0.115 (5)
C6	0.2223 (2)	0.374 (1)	−0.025 (2)	0.101 (5)
C7	0.2110 (2)	0.495 (2)	−0.142 (1)	0.103 (4)
C8	0.1777 (2)	0.559 (1)	−0.128 (1)	0.083 (4)

Table 2. *Selected geometric parameters (Å, °)*

Cd—Cl1	2.548 (1)	Cd—Cl2	2.650 (1)
Cd—Cl2^i	2.645 (1)		
Cl2^i—Cd—Cl1	90.00 (5)	Cl2—Cd—Cl2^i	89.73 (4)
Cl2—Cd—Cl1	92.03 (5)	Cd—Cl2—Cd^{ii}	162.36 (6)

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

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	H···A	D···A	D—H···A
N—H6···Cl2^i	2.30	3.241 (5)	166
N—H7···Cl1^{ii}	2.60	3.421 (5)	143
N—H8···Cl1^{iii}	2.34	3.273 (4)	165

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

Table 4. *Statistics of F^2 values*

	All	$3\sigma \le F^2$	$5\sigma \le F^2$	$10\sigma \le F^2$	$20\sigma \le F^2$
F-lat. ref.	566	548	526	421	223
non-F ref.	1319	406	204	78	17
non-F (%)	70.0	42.6	27.9	15.6	7.1
All ref.	1885	954	730	499	240

Notes: F reflections: *ggg, uuu*; non-F reflections: *ggu, gug, guu, ugg, ugu, uug*.

Data collection: local software (Gomm, 1993). Cell refinement: local software. Data reduction: local software. Program(s) used to solve structure: *CRYSTAN* (Burzlaff & Rothammel, 1988). Program(s) used to refine structure: *CRYSTAN* (Spengler & Zimmermann, 1996). Molecular graphics: *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1289). Services for accessing these data are described at the back of the journal.

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Dimethylammonium Trichloropalladate

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Abstract

The title compound, [NH$_2$(CH$_3$)$_2$][PdCl$_3$], was produced as a minor product of the reaction involving 1,3-thiazolidine-2-thione and K$_2$PdCl$_4$ in aqueous HCl. The structure contains [NH$_2$(CH$_3$)$_2$]$^+$ cations and [PdCl$_3$]$^-$ anions. The anion consists of Pd^{II} atoms bonded to a combination of bridging and terminal Cl$^-$ ions in the form of an infinite chain of general formula [Pd(μ-Cl)(Cl)$_2$]$_2$. Each Pd^{II} atom is bonded in a four-coordinate square-planar configuration. The cations are hydrogen bonded to the [PdCl$_3$]$^-$ chain.