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The Intercalation Compound 1,4-Benzenediammonium Tetrachlorocadmate(II)

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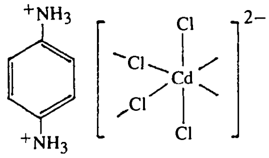
Abstract

Crystals of 1,4-benzenediammonium tetrachlorocadmate(II), $(C_6H_{10}N_2)[CdCl_4]$, contain two-dimensional anionic sheets in which each $CdCl_6$ octahedron shares four corners with adjacent octahedra {*i.e. catena*-poly[1,4-benzenediammonium dichlorocadmium-di-μ-chloro(2−)]}. Layers of organic diammonium cations are intercalated between the metal–halogen sheets.

Comment

Chemists have become increasingly involved in the synthesis and characterization of various low-dimensional solid-state chain or layer compounds because of their unusual physical properties, *e.g.* superconductivity. About twenty years ago many layer perovskites of general formulae $(C_nH_{2n+1}NH_3)_2MCl_4$ and $(NH_3C_nH_{2n}NH_3)MCl_4$ (M = Cd, Cu, Fe, Mn, Pt, Pd) were investigated in view of their interesting thermal and magnetic properties (Arend & Huber, 1978; Needham & Willett, 1984). The structures of these compounds are very similar to those of Langmuir–Blodgett films. In this paper, we report the crystal structure of such a compound, (I).



(I)

From Fig. 1 it can be seen that the title compound consists of two basic species: the $CdCl_6$ polyhedron and the *p*-phenylene cation. In the $CdCl_6$ layers (Fig. 2*a*), each Cd atom occupies the centre of a distorted but exactly centrosymmetric octahedron formed by four symmetry-related and almost coplanar Cl(1) atoms linking adjacent octahedra plus two axial Cl(2) ligands. In the $CdCl_6$ layers the bridging Cd—Cl(1)—Cd bond angles are 153.61 (4)°; the Cd atoms of the four closest octahedra and their associated bridging Cl^- ions form a pseudo-tetragonal arrangement which extends in two dimensions to form the $CdCl_4$ sheet (Fig. 2*b*). The axial Cl(2) ligands protrude above and below the sheets. *p*-Phenylene diammonium cations are trapped between two adjacent $CdCl_6$ layers and lie in the cavities formed by eight $CdCl_6$ octahedra (four octahedra form the base and four form the top of the cavity). The axial Cl(2) ligands protrude from the $CdCl_4$ sheets and thereby hinder the diffusion of the *p*-phenylene cations out of the cavities.

As can be seen from Fig. 2(*b*), in each *p*-phenylenediammonium layer adjacent aromatic rings are almost exactly normal to one another (dihedral angle 88.2°).

The structure of this layer perovskite is stabilized by N—H···Cl hydrogen bonds from both ends of the *p*-phenylenediammonium cations, which hold adjacent $CdCl_6$ layers together. They are arranged so that one hydrogen bond [N—H···Cl(1) 3.254 (6) Å] involves the corner-sharing Cl(1) atom whereas the other two from each —NH_3 group [N—H···Cl(2) 3.155 (6) and 3.210 (5) Å] are directed towards the axial Cl atoms of the $CdCl_6$ octahedra.

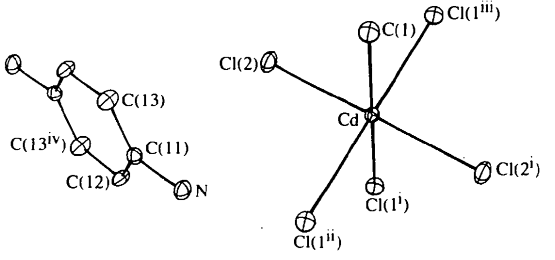


Fig. 1. The numbering of the atoms of the title compound with 30% probability displacement ellipsoids. H atoms are omitted for clarity.

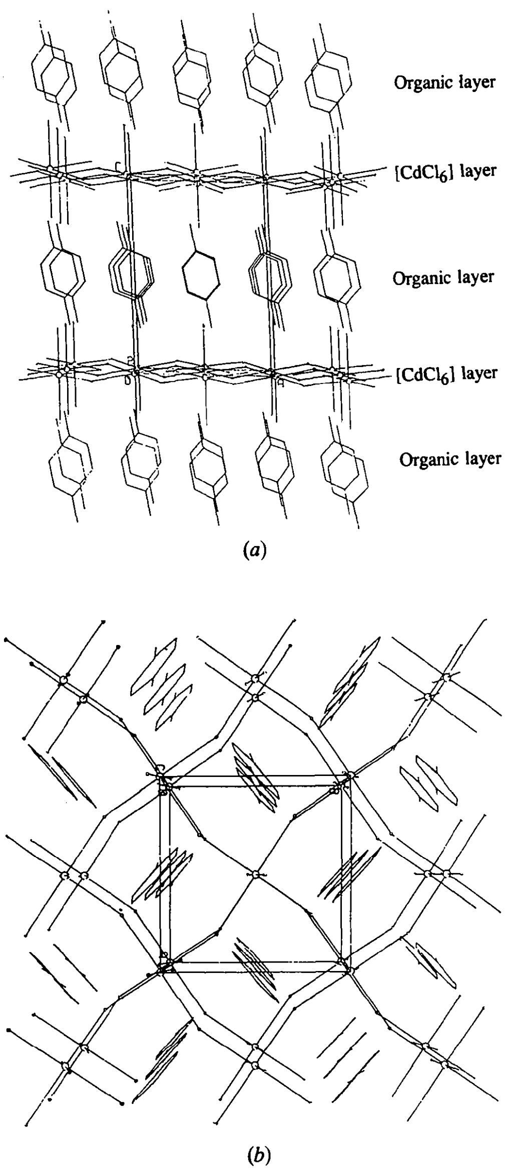


Fig. 2. (*a*) View of unit cell down the *b* axis. (*b*) View of unit cell down the *c* axis. H atoms are omitted for clarity.

Experimental

The compound was prepared by the reaction of stoichiometric amounts of $CdCl_2.H_2O$ and $NH_3C_6H_4NH_3$ in 2 *M* HCl solution. Elemental analysis of the C, H, N, Cd and Cl content corresponds to within experimental error with the composition $(NH_3C_6H_4NH_3)CdCl_4$. The crystal used for X-ray analysis was obtained by slow evaporation of an aqueous solution of $(NH_3C_6H_4NH_3)CdCl_4$ at room temperature.

Crystal data

$(C_6H_{10}N_2)[CdCl_4]$
$M_r = 364.38$
Monoclinic
$P2_1/a$
$a = 7.336\ (1)$ Å
$b = 7.401\ (2)$ Å
$c = 10.408\ (4)$ Å
$\beta = 93.34\ (2)°$
$V = 564.2\ (5)$ Å^3
$Z = 2$
$D_x = 2.145$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.95–14.93°$
$\mu = 2.84$ mm^{-1}
$T = 296$ K
Platelet
1.0 × 1.0 × 0.25 mm
Light pink

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.32$, $T_{max} = 1.00$
1174 measured reflections
1105 independent reflections

938 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.057$
$\theta_{max} = 25°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections frequency: 60 min intensity decay: 3.1%

Refinement

Refinement on F
$R = 0.055$
$wR = 0.064$
$S = 2.45$
938 reflections
82 parameters
H atoms: positions and U_{iso} refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.07$

$\Delta\rho_{max} = 1.23$ e Å^{-3}
$\Delta\rho_{min} = -1.11$ e Å^{-3}
Extinction correction: secondary (Zachariasen, 1967)
Extinction coefficient: 2.449×10^{-4}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cd	0	0	0	1.19 (4)
Cl(1)	0.2990 (1)	0.2043 (1)	0.03668 (9)	1.91 (5)
Cl(2)	−0.0001 (1)	−0.0492 (2)	0.2395 (1)	2.15 (5)
N	0.0627 (7)	−0.4709 (8)	0.2366 (5)	2.2 (2)
C(11)	0.0321 (9)	−0.4891 (5)	0.3727 (6)	1.6 (2)
C(12)	−0.1021 (6)	−0.6036 (6)	0.4105 (4)	2.0 (1)
C(13)	0.1333 (6)	−0.3836 (6)	0.4601 (4)	2.3 (2)

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

Cd—Cl(2)	2.519 (1)	N—C(11)	1.454 (8)
Cd—Cl(1)	2.673 (1)	C(11)—C(12)	1.373 (7)
C(11)—C(13)	1.382 (7)	Cd—Cl(1^{ii})	2.679 (1)
C(12)—C(13^{iv})	1.383 (6)		
Cl(1)···N^{v}	3.254 (6)	Cl(2)···N	3.155 (6)
Cl(1)···N^{vi}	3.337 (5)	Cl(2)···N^{vii}	3.210 (5)
Cl(2)—Cd—Cl(1)	89.31 (4)	Cl(2)—Cd—Cl(1^{ii})	93.13 (5)
Cd—Cl(1)—Cd^{vi}	153.61 (4)	C(12)—C(11)—C(13)	121.8 (5)
C(12)—C(11)—N	119.4 (5)	C(13)—C(11)—N	118.8 (5)
C(11)—C(12)—C(13^{iv})	118.9 (5)	C(11)—C(13)—C(12^{iv})	119.3 (4)
Cl(1)—Cd—Cl(1^{ii})	91.64 (2)		

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

The structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares methods, with anisotropic displacement parameters for all non-H atoms.

Data collection: Enraf–Nonius (1982) CAD-4 software. Structure solution and refinement: *TEXSAN* (Molecular Structure Corporation, 1988). Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell, 1974).

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1132). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Adduct of $AlMe_3$ with Benzo[*f*]quinoline

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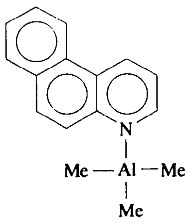
Abstract

The reaction of $AlMe_3$ with benzo[*f*]quinoline gave the Lewis acid–base adduct Me_3Al–BQ, (BQ = benzo[*f*]quinoline) {(benzo[*f*]quinoline-*N*)trimethylaluminium, [$Al(CH_3)_3(C_{13}H_9N)$]}, with an Al—N bond length of 2.057 (2) Å. This adduct is much less air and water sensitive than the trialkyl metal. The complex was found to be a discrete 1:1 molecular adduct of trimethylaluminium bonded to BQ. The compound contains a tetracoordinate Al atom with distorted tetrahedral geometry.

Comment

The growth of epitaxial layers of Group III–V and Group II–VI compound semiconductors by metal-organic chemical vapour deposition (MOCVD) is being studied extensively and is progressing towards the production phase for *e.g.* gallium arsenide. The electrical properties of these layers depend critically upon the purity of the precursors (metal alkyls and hydrides) employed in the semiconductor growth. The development of purification techniques for the removal of metal-containing impurities in these precursors to levels ≤ 1 p.p.m. has provided an exciting challenge for the organometallic chemist (Beachley & Coates, 1965; Bradley, Dawes, Frigo, Hursthouse & Smith, 1990). In this paper we report the synthesis, characterization and X-ray structure determination of a novel intermolecular-stabilized organoaluminium complex, (I), as a MOCVD precursor.



(I)

The structure consists of a discrete 1:1 molecular adduct of trimethylaluminium bonded to BQ. The compound contains tetracoordinate Al atoms within distorted tetrahedra, with the distortion being towards trigonal pyramidal geometry. The base of the pyramid is defined by the three methyl C atoms. The Al atom is almost coplanar with the plane of the coordinated benzo[*f*]quinoline (Bradford, Bradley, Hursthouse & Motevalli, 1992). The C—Al—N bond angles [103.9 (1)–105.5 (1)°] are consistently *ca* 4–5° smaller than the tetrahedral angle. The Al—C bond lengths [1.965 (4)–1.987 (3) Å] are within the expected range for metal–carbon single bonds. Thus, it is noteworthy that whereas the Al—C and Ga—C [1.972 (5)–1.984 (6) Å] (Sun, Wang, Huang & You, 1995) bond distances are very similar to each other, the Al—N bond [2.057 (2) Å] is significantly shorter than the Ga—N bond [2.152 (3) Å], implying stronger bonding of BQ to $AlMe_3$ than to $GaMe_3$.

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