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Bis(triethylsulfonium) Tetraiodocadmate(II)

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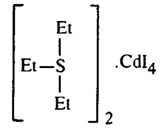
Abstract

The reaction of CdI_2 with an acetone solution of $(Et_3S)I$ (molar ratio 1:2) gives the title compound. The structure of $[(C_2H_5)_3S]_2[CdI_4]$ contains discrete pyramidal Et_3S^+ and tetrahedral CdI_4^{2-} ions. The bond distances of the two independent anions, Cd1—I1 and Cd2—I2, are 2.772 (1) and 2.784 (1) Å, respectively. The cation, with approximate threefold symmetry, is disordered such that each of the middle methylene groups takes two positions with a ratio of *ca* 2:1.

Comment

The title compound, $(Et_3S)_2[CdI_4](s)$, (I), consists of discrete pyramidal Et_3S^+ cations and tetrahedral CdI_4^{2-} moieties.



$$\left[\begin{array}{c}\mathrm{Et}\\|\\\mathrm{Et{-}S}\\|\\\mathrm{Et}\end{array}\right]_2\mathrm{.CdI_4}$$

(I)

Intra- and intermolecular distances and angles are listed in Table 2. The cations with approximate threefold symmetry are disordered over two distinct orientations with populations of 0.65 (2) and 0.35 (2) (Fig. 1). The S and methyl C atoms coincide approximately in the two orientations, but the methylene C atoms are displaced between 1.34 (4) and 1.50 (5) Å. The S—C distances and S—C—C angles in the tetraiodocadmate(II) compound, as calculated from the disordered atom positions, differ somewhat from those obtained in the ordered $(Et_3S)[Hg_2I_6]_{1/2}$ (Bengtsson, Norén & Stegemann, 1995). The cation–anion arrangement is related to the antifluorite-type structure, but the 'cubes' of cations are heavily distorted. The triethylsulfonium cations are positioned so that the pyramidal S atoms are pointing towards the faces of the $Cd2I_4^{2-}$ tetrahedra. The 'back side' of the cations are centred over the I atoms of $Cd1I_4^{2-}$. A similar relationship between the cations and anions was found in $(Et_3S)[Hg_2I_6]_{1/2}$. Also, the I···S distances are of the same magnitude and indicate that the cation–anion interaction is predominantly of electrostatic and van der Waals types (Bengtsson *et al.*, 1995).

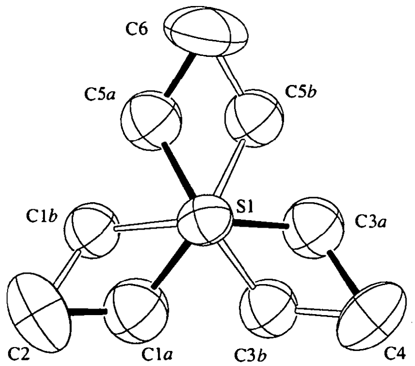


Fig. 1. The molecular structure of the Et_3S^+ cation showing 50% probability displacement ellipsoids. The major disorder components of the ethyl groups are shown with filled bonds.

The CdI_4^{2-} fragments have been observed both as isolated tetrahedra in solution and in the solid state (see Table 3), as edge-sharing tetrahedra in $Cd_2I_6^{2-}$ (Orioli & Ciampolini, 1972; Leemput, Cras & Willemse, 1977; Gritsenko, Konovalikhin, D'yachenko, Lyubovskaya & Zhilyaeva, 1990; Bu, Su & Coppens, 1991) and as corner-sharing chains of tetrahedra (Sjövall, Svensson & Lidin, 1996). The average Cd—I distances in compounds with isolated CdI_4^{2-} tetrahedra (Table 3) are typically similar to those observed in this study [2.772 (1) and 2.784 (1) Å], which are approximately equal to the sum of the covalent radii.

Experimental

Bis(triethylsulfonium) tetraiodocadmate(II), $(Et_3S)_2[CdI_4]$, was prepared by mixing stoichiometric amounts of an aqueous slurry of CdI_2 (Merck, p. A. grade) with an acetone solution of triethylsulfonium iodide to give a colourless solution. $(Et_3S)I$ was prepared by alkylation of Et_2S with EtI in chloroform and then purified by recrystallization from hot ethanol before use. Transparent single crystals were grown by slow evaporation.

Crystal data

$(C_6H_{15}S)_2[CdI_4]$
$M_r = 858.53$
Tetragonal
$I4_1/a$
$a = 13.885 (1)$ Å
$c = 26.207 (3)$ Å
$V = 5052 (1)$ Å^3
$Z = 8$
$D_x = 2.257$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 49 reflections
$\theta = 5$–$38°$
$\mu = 5.854$ mm^{-1}
$T = 295$ K
Tetragonal bipyramid
$0.22 \times 0.20 \times 0.17$ mm
Colourless

Data collection

Huber diffractometer
ω scans with profile analysis
Absorption correction: by integration from crystal shape
$T_{min} = 0.357$, $T_{max} = 0.438$
2503 measured reflections
2303 independent reflections

1329 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.036$
$\theta_{max} = 25°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 31$
2 standard reflections frequency: 60 min intensity decay: 1.6%

Refinement

Refinement on F
$R = 0.0486$
$wR = 0.0525$
$S = 1.702$
1329 reflections
85 parameters
H atoms not located
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.08$

$\Delta\rho_{max} = 0.94$ e Å^{-3}
$\Delta\rho_{min} = -0.85$ e Å^{-3}
Extinction correction: Zachariasen type 2, Gaussian isotropic
Extinction coefficient: $0.22 (9) \times 10^{-7}$
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)*

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

	x	y	z	U_{eq}
Cd1	0	1/4	5/8	0.0469 (6)
Cd2	0	1/4	1/8	0.0464 (6)
I1	0.01258 (7)	0.08422 (7)	0.56641 (4)	0.0727 (6)
I2	0.05048 (7)	0.09488 (6)	0.06326 (4)	0.0639 (6)
S1	0.2742 (2)	0.1789 (2)	0.4656 (1)	0.060 (3)
C1*a*†	0.304 (2)	0.139 (2)	0.5298 (10)	0.092 (8)
C1*b*‡	0.257 (3)	0.223 (3)	0.534 (2)	0.069 (12)
C2	0.363 (1)	0.214 (1)	0.5557 (6)	0.108 (13)
C3*a*†	0.199 (2)	0.082 (2)	0.4416 (9)	0.084 (8)
C3*b*‡	0.264 (3)	0.053 (3)	0.476 (2)	0.075 (12)
C4	0.271 (1)	−0.005 (1)	0.4261 (7)	0.093 (16)
C5*a*†	0.185 (2)	0.270 (2)	0.4813 (9)	0.081 (8)
C5*b*‡	0.146 (3)	0.202 (3)	0.442 (2)	0.077 (13)
C6	0.141 (1)	0.310 (1)	0.4301 (6)	0.117 (18)

† Site occupancy = 0.655 (19). ‡ Site occupancy = 0.345 (19).

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

Cd1—I1	2.772 (1)	S1—C3*a*	1.82 (2)
Cd1—I1^{i}	2.772 (1)	S1—C3*b*	1.77 (4)
Cd1—I1^{ii}	2.772 (1)	S1—C5*a*	1.83 (2)
Cd1—I1^{iii}	2.772 (1)	S1—C5*b*	1.91 (4)
Cd2—I2	2.784 (1)	C1*a*—C2	1.49 (3)
Cd2—I2^{i}	2.784 (1)	C1*b*—C2	1.58 (4)
Cd2—I2^{iv}	2.784 (1)	C3*a*—C4	1.61 (3)
Cd2—I2^{v}	2.784 (1)	C3*b*—C4	1.55 (4)
S1—C1*a*	1.82 (3)	C5*a*—C6	1.57 (3)
S1—C1*b*	1.91 (4)	C5*b*—C6	1.53 (5)
I1—Cd1—I1^{i}	112.74 (4)	C1*a*—S1—C3*a*	103 (1)
I1—Cd1—I1^{ii}	107.86 (2)	C1*a*—S1—C5*a*	99 (1)
I1—Cd1—I1^{iii}	107.86 (2)	C1*b*—S1—C3*b*	99 (1)
I1^{i}—Cd1—I1^{ii}	107.86 (2)	C1*b*—S1—C5*b*	97 (1)
I1^{i}—Cd1—I1^{iii}	107.86 (2)	C3*a*—S1—C5*a*	101 (1)
I1^{ii}—Cd1—I1^{iii}	112.74 (4)	C3*b*—S1—C5*b*	98 (1)
I2—Cd2—I2^{i}	108.92 (4)	S1—C1*a*—C2	109 (1)
I2—Cd2—I2^{iv}	109.75 (2)	S1—C1*b*—C2	101 (2)
I2—Cd2—I2^{v}	109.75 (2)	S1—C3*a*—C4	106 (1)
I2^{i}—Cd2—I2^{iv}	109.75 (2)	S1—C3*b*—C4	111 (2)
I2^{i}—Cd2—I2^{v}	109.75 (2)	S1—C5*a*—C6	108 (1)
I2^{iv}—Cd2—I2^{v}	108.92 (4)	S1—C5*b*—C6	105 (2)

Symmetry codes: (i) $-x, \frac{1}{2}-y, z$; (ii) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{5}{4}-z$; (iii) $\frac{1}{4}-y, \frac{1}{4}+x, \frac{5}{4}-z$; (iv) $\frac{1}{4}-y, \frac{1}{4}+x, \frac{1}{4}-z$; (v) $y-\frac{1}{4}, \frac{1}{4}-x, \frac{1}{4}-z$.

Table 3. *Average bond distances of some isolated CdI_4^{2-} ions*

Ion	Distance (Å)	Reference
$CdI_4{}^{2-}$(aq)	2.79	(*a*)
$CdI_4{}^{2-}$(dmso)	2.79	(*b*)
$Cs_2[CdI_4]$(s)	2.77	(*c*)
$(C_4H_{12}N)_2[CdI_4]$(s)	2.79	(*d*)
$(C_4H_{12}N)_2[CdI_4]$(s)	2.77	(*e*)
$(C_6H_{15}S)_2[CdI_4]$(s)	2.78	(*f*)
$Ca[CdI_4].8H_2O$(s)	2.80	(*g*)
$Sr[CdI_4].8H_2O$(s)	2.78	(*g*)
$[C_8H_{12}N_2(H_2)]_2[CdI_4]$(s)	2.79	(*h*)
$[C_8H_{12}N_2(H_2)]_2[CdI_4].H_2O$(s)	2.79	(*h*)
$[Cd(pyo)_6][CdI_4]$(s)	2.78	(*i*)
$[Cd(dmso)_6][CdI_4]$(s)	2.79	(*j*)
$[N(CH_3)_4]_2[CdI_4]$(s)	2.76	(*k*)

References: (*a*) Ohtaki, Maeda & Ito (1974); (*b*) Pocev, Triolo & Johansson (1979); (*c*) Sjövall (1989); (*d*) Kallel, Bats & Daoud (1981); (*e*) Kallel, Borg, Fuess & Boutiba (1991); (*f*) this work; (*g*) Thiele & Putzas (1988); (*h*) Bailey & Pennington (1995); (*i*) Nieuwenhuyzen, Robinson & Wilkins (1991); (*j*) Nieuwenhuyzen, Wen & Wilkins (1992); (*k*) Rogers, Bond, Aguinaga & Reyes (1993).

During a first data collection, the intensities of the two standards increased by a total of about 100%. This was taken to indicate a transformation of the crystal in the X-ray beam towards the ideally imperfect state. When the intensities seemed stable, a second data collection was started. This second data set, with only 1.6% intensity decrease, was used for the structure determination. The Laue group is $4/m$ and systematic extinctions (hkl: $h + k + l \neq 2n$; $hk0$: $h \neq 2n$; $00l$: $l \neq 4n$) are consistent with the space group $I4_1/a$ (No. 88), which was verified by the statistical analysis of intensity distribution, packing considerations and the successful refinement of the structure.

Data collection: local diffractometer control program. Cell refinement: *LATCON* in *XRAY76* (Stewart *et al.*, 1976). Data reduction: *TEXSAN PREPROCESS* and *PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *TEXSAN MITHRIL*. Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: JZ1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Triple-Decker Pentalene Complex of Iron and Cobalt

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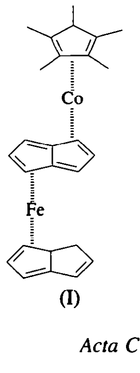
Abstract

In [1(1,2,3,3a,6a-η)-1,4-dihydropentalenyl][μ-1(1,2,3,3a,6a-η):2(3a,4,5,6,6a-η)-pentalene][2(η^5)-pentamethylcyclopentadienyl]cobaltiron, [CoFe(C_8H_7)Cp*(C_8H_6)] (Cp* = $C_{10}H_{15}$), the Cp*–Co and (C_8H_7)–Fe moieties reside on opposite sides of the fused bridging pentalene ring system.

Comment

Model sandwich complexes having two or more metals, which may be considered as the smallest segments of one-dimensional conducting materials, are excellent candidates for the detailed study of metal–metal interactions. We have recently developed a systematic controlled route for the preparation of triple- and quadruple-decker molecular-sandwich derivatives of pentalene (Oelckers, Chávez, Manríquez & Román, 1993). As a part of this general study, we present here the structure of the title compound, (I), which may serve as a potential building block in the preparation of larger polymetallic systems.



(I)