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Linear-Chain Structure of Bis(*O,O'*-dimethyldithiophosphato)cadmium(II)

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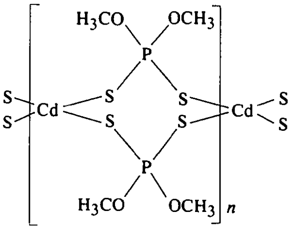
Abstract

The two ligands in the title compound, *catena*-poly[cadmium-μ-bis(O,O'-dimethyldithiophosphato-S:S')], $[\mathrm{Cd\{S_2P(OCH_3)_2\}_2}]$, bridge two adjacent Cd atoms forming a linear chain along the *c* axis. The chain has a twofold symmetry axis through the Cd atoms. The Cd atom is coordinated by four S atoms in a distorted tetrahedral arrangement. The Cd—S bond lengths are 2.565 (2) and 2.571 (2) Å.

Comment

Metal dialkyl dithiophosphates, $[M\{\mathrm{S_2P(OCH_3)_2}\}_n]$, are an interesting group of complexes which show various kinds of coordination around metals and various modes of molecular aggregation (Lawton & Kokotailo, 1969; Ito, Igarashi & Hagihara, 1969; Ito, 1972). We report here the crystal structure of $[\mathrm{Cd\{S_2P(OCH_3)_2\}_2}]$, (I), which displays a distorted tetrahedral coordination and a linear-chain structure.



(I)

An *ORTEP*II drawing (Johnson, 1976) of the structure of (I) with the atomic numbering scheme is shown in Fig. 1. The linear-chain structure is similar to that in zinc diethyl dithiophosphate (Ito *et al.*, 1969), but in the latter, one ligand is bridging and the other is chelating. On the other hand, zinc and cadmium diisopropyl dithiophosphates are both dimeric (Lawton & Kokotailo, 1969). The Cd atom lies on a twofold axis and has distorted tetrahedral coordination of four S atoms. The Cd—S1 and Cd—S2 bond lengths are 2.565 (2) and 2.571 (2) Å, respectively. The S—Cd—S angles range from 102.34 (4) to 124.23 (7)°.

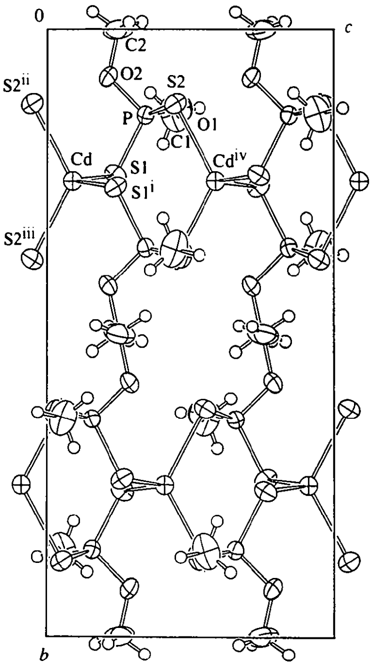


Fig. 1. The crystal structure viewed down the *a* axis (*ORTEP*II; Johnson, 1976). The displacement ellipsoids are at the 50% probability level. Symmetry codes: (i), (ii), (iii) as in Table 1; (iv) $x, \frac{1}{2}-y, z+\frac{1}{2}$.

Experimental

Powdered $\mathrm{P_2S_5}$ (0.05 mol) was added to 70 ml of $\mathrm{CH_3OH}$ and heated to 323–333 K. After ~1 h, when the generation of $\mathrm{H_2S}$ vapour ceased, powdered $\mathrm{CdSO_4}$ (0.05 mol) was added to the solution to give a white precipitate of $[\mathrm{Cd\{S_2P(OCH_3)_2\}_2}]$. Colourless crystals were obtained by recrystallization of the precipitate from an acetone solution.

Crystal data

$[\mathrm{Cd(C_2H_6O_2PS_2)_2}]$
$M_r = 426.72$
Orthorhombic
Pccn
$a = 9.282\,(4)$ Å
$b = 17.837\,(5)$ Å
$c = 8.488\,(4)$ Å
$V = 1405.2\,(9)$ Å^3
$Z = 4$
$D_x = 2.017$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 24–25°$
$\mu = 2.336$ mm^{-1}
$T = 295$ K
Needle
$0.80 \times 0.20 \times 0.20$ mm
Colourless

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction:
  ψ scans (*TEXSAN*; Molecular Structure Corporation, 1985)
  $T_{\min} = 0.34$, $T_{\max} = 0.63$
3590 measured reflections
3515 independent reflections
1338 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.044$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 23$
$l = -11 \rightarrow 11$
3 standard reflections
  monitored every 150 reflections
  intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.0365$
$wR = 0.0545$
$S = 2.375$
1338 reflections
70 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.69$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A and 2.3.1)

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

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

	x	y	z	$B_{\rm eq}$
Cd	1/4	1/4	0.0893 (5)	2.42 (2)
S1	0.0100 (1)	0.2402 (7)	0.2377 (2)	2.96 (5)
S2	0.2256 (1)	0.1232 (8)	0.4477 (2)	2.95 (5)
P	0.0377 (1)	0.1418 (7)	0.3426 (1)	2.19 (5)
O1	−0.0814 (3)	0.1297 (2)	0.4739 (4)	3.2 (2)
O2	0.0083 (4)	0.0797 (2)	0.2123 (4)	3.0 (1)
C1	−0.2317 (6)	0.1401 (6)	0.4434 (9)	5.7 (4)
C2	0.0183 (8)	0.0009 (3)	0.2476 (8)	5.5 (3)

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

Cd—S1	2.565 (2)	S2—P	1.988 (2)
Cd—S1^{i}	2.565 (2)	P—O1	1.584 (3)
Cd—S2^{ii}	2.571 (2)	P—O2	1.588 (3)
Cd—S2^{iii}	2.571 (2)	O1—C1	1.433 (7)
S1—P	1.986 (2)	O2—C2	1.438 (6)
S1—Cd—S1^{i}	121.17 (7)	S1^{i}—Cd—S2^{ii}	102.34 (4)
S1—Cd—S2^{ii}	104.22 (4)	S1^{i}—Cd—S2^{iii}	104.22 (4)
S1—Cd—S2^{iii}	102.34 (4)	S2^{ii}—Cd—S2^{iii}	124.23 (7)

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

The Cd atom was located by an automatic Patterson map interpretation using *DIRDIF* (Beurskens, 1984) and the remaining non-H atoms were located by Fourier syntheses. H atoms were placed in geometrically calculated positions and were not included in the refinement. The structure was refined by full-matrix least-squares methods with anisotropic displacement parameters for all atoms. Calculations were performed on a VAX3200 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

Data collection: *CONTROL* (Molecular Structure Corporation, 1988). Cell refinement: *CONTROL*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *DIRDIF*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OA1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chlorotris(*m*-methoxyphenyl)tin(IV)

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Abstract

The title compound, [SnCl(C_7H_7O)$_3$], crystallizes in the trigonal space group *R*3, with completely symmetric molecules, the Sn—Cl bond lying along the threefold axis. In contrast to the methyl analogue, the *meta*-CH_3O groups are distal, *i.e.* they are towards the Sn—Cl bond, and coplanar with the phenyl rings, with the methyl group itself lying over the *ortho*-C atom of the ring.

Comment

The effect of phenyl-ring substituents on tetraaryltin (Ar_4Sn) structures has been thoroughly investigated, with *meta* substituents creating the greatest deviations from 'ideal' tetragonal crystal and molecular structures, compared with the effect of the same group in a *para* or *ortho* position (Wharf & Simard, 1995). In contrast, the opposite is the case for triaryltin halides (Ar_3SnX).

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