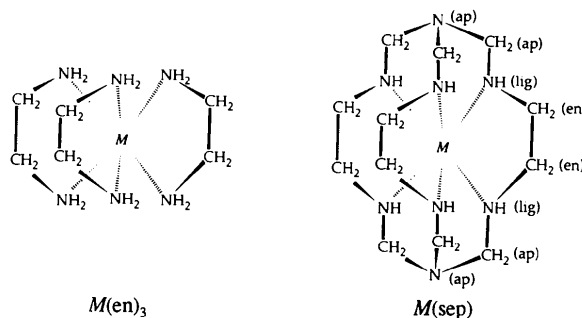


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Comment

Sepulchrate (1,3,6,8,10,13,16,19-octaazabicyclo[6.6.6]icosane) is a cage ligand with six secondary aminic N—H groups which can encapsulate a metal cation in an octahedral coordination arrangement. The complex molecule *M*(sep) can be thought of as derived from a trisethylendiamine metal complex *M*(en)₃, with two N(CH₂)₃ systems capping two opposite N—N—N faces of the octahedron.



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Conformation and Packing of Cobalt(III) Sepulchrate Dihydrogentriphosphate Hexahydrate

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Abstract

In cobalt(III) sepulchrate dihydrogentriphosphate hexahydrate (sepulchrate = 1,3,6,8,10,13,16,19-octaazabicyclo[6.6.6]icosane), [Co(C₁₂H₃₀N₈)](H₂P₃O₁₀).6H₂O, the cation adopts the *lel*₃ [$\Delta(\lambda\lambda\lambda)$] optically active conformation. The overall geometry of the cation is not significantly different from that found in other salts. A strong symmetric O··H··O hydrogen bond is responsible for the cyclic conformation of the anion, the geometry of which is described by an eight-membered puckered ring. The entire crystal packing is dominated by hydrogen bonds. The cations and the anions are linked together in a three-dimensional network with six-membered meshes; the water molecules are situated in the voids, and interact only with the anions.

The compound is optically active and presents potential *D*₃ point symmetry, with the *C*₃ axis passing through N(ap)—*M*—N(ap), and the three *C*₂ axes bisecting the three C(en)—C(en) bonds. The cobalt(III) sepulchrate cation has been studied in different crystalline environments (Creaser *et al.*, 1977, 1982; Bacchi, Ferranti & Pelizzi, 1993*a,b*; Bacchi, 1995) and its conformation has been shown to be practically invariant in all compounds. The preferred geometry for Co(sep)³⁺ is that which allows the formation of hydrogen-bonded Co(sep)ABC moieties (A, B, C = hydrogen-bond acceptors). Pairs of N—H groups belonging to one cation are bridged by hydrogen-bonding anions or water molecules. In order to make this possible, the six aminic N—H groups of a cation must point, in pairs, towards three different positions around the cation; this is accomplished by orienting the C(en)—C(en) bonds in directions almost parallel to the N(ap)—Co—N(ap) pseudo-threefold axis. Following the chirality independent *lellob* classification proposed by Corey & Baley (1959), the geometry of the cation is *lel*₃. A perspective view of the Co(sep)³⁺ cation and the H₂P₃O₁₀ anion is shown in Fig. 1. The angles α formed by the C(en)—C(en) bonds and the N(ap)—Co—N(ap) vector range from 4.0 (3) to 5.4 (3)° for the cation. The overall absolute configuration of the cation is Λ , while the conformations of the puckered Co—N(lig)—C(en)—C(en)—N(lig) rings are all δ ; a $\Lambda(\delta\delta\delta)$ cation results, in accord with the *lel*₃ geometry. The geometry of the cation was compared with that of cation *A* in (\pm)-Co(sep)Cl₃.H₂O (Bacchi *et al.*, 1993*a*) by performing a half-normal probability plot on all the interatomic distances between non-H atoms (Abrahams & Keve, 1971). The least-squares regression line through the data points is $y = -0.36(4) + 3.28(4)x$, with a correlation coefficient of 0.98. The

good agreement expressed by the correlation coefficient indicates the absence of relevant differences in the geometry of the two cations; the angular coefficient greater than 1 reveals a probable underestimation of the σ values [in agreement with the observations of Taylor & Kennard (1986)], while the non-zero intercept suggests some source of systematic error in the data. The conformational invariance of the $\text{Co}(\text{sep})^{3+}$ cation embedded in different crystalline fields is thus confirmed.

The dihydrogenetriphosphate anion adopts a bent conformation with torsion angles $\text{P1—O4—P2—O7} = 82.3(3)$ and $\text{P3—O7—P2—O4} = 24.7(4)^\circ$. The folding of the P1—O4—P2—O7—P3 chain is forced by the strong hydrogen bond $\text{O2} \cdots \text{H2P} \cdots \text{O10}$ which keeps the two extremities of the anion together, giving an eight-membered highly puckered ring. This hydrogen-bonded cyclization occurs in all the five compounds containing the $\text{H}_2\text{P}_3\text{O}_{10}^{3-}$ anion retrieved by an ICSD search (Inorganic Crystal Structure Database, release January 1994) (Merritt & Sundaralingam, 1981; Lutsko & Johansson, 1984; Lutsko, Lyakhov, Tuchkovskii & Palkina, 1991; Lyakhov, Palkina, Lutsko, Maximova & Chibiskova, 1990; Lyakhov, Lutsko, Tuchkovskii & Palkina, 1991). For a sixth compound containing the anion, the hydrogen coordinates are not provided (Palkina, Maksimova & Kuznetsov, 1979). It must be stressed that in the present dihydrogenetriphosphate anion the intramolecular hydrogen bond is symmetric (there is some evidence for elongated density), with H2P situated halfway between O2 and O10 (see Table 3); this is not the case for the other above-mentioned compounds, for which the following geometries are found: $\text{O—H} = 0.817, 0.776, 0.932, 0.765, 0.876 \text{ \AA}$; $\text{H} \cdots \text{O} = 1.786, 1.983, 1.840, 2.037, 2.003 \text{ \AA}$; $\text{O—H} \cdots \text{O} = 161.6, 157.8, 155.5, 145.4, 137.0^\circ$. The puckering of the eight-membered hydrogen-bonded ring can be described by the parameters proposed by Cremer & Pople (1975): $Q_T = 1.12(1) \text{ \AA}^2$; $\theta_2 = 69(1)^\circ$; $\theta_3 = 77(3)^\circ$. The asymmetry parameters (Nardelli, 1983) indicate a distorted C_s conformation, with the pseudo-mirror plane bisecting P2—O7 and H2P—O2 [$\Delta_5(\text{P2}, \text{O7}) = 0.04(1)$].

The crystal packing is based on the hydrogen bonds between cations and anions, and between anions and water molecules. The cations and anions are linked together in a three-dimensional network with six-membered meshes; the water molecules are situated in the voids (Fig. 2). Each cation is hydrogen-bonded with three anions, at (x, y, z) , $(x, y, z - 1)$ and $(x - 1, y - 1, z - 1)$, and conversely each anion is hydrogen-bonded with cations at (x, y, z) , $(x, y, z + 1)$ and $(x + 1, y + 1, z + 1)$ (Table 3). The hydrogen bonds are of the type $\text{N—H} \cdots \text{O} \cdots \text{H}'\text{—N}'$, where both N—H and $\text{N}'\text{—H}'$ belong to the same cation, according to the $1el_3$ conformation of the sepulchrate cage, as described above. All the terminal O atoms of the anion are involved in intra- (O2 and O10) or intermolecular hydrogen bonds, either with cations (O1, O8 and O9) or with the water molecules

(O3, O5 and O6), which are also hydrogen-bonded to one another (Table 3). The entire crystal packing is dominated by hydrogen bonding due to the large number of hydrogen-bond acceptor sites on the anion; the high degree of hydration of the compound is probably required to satisfy the acceptor potentiality of $\text{H}_2\text{P}_3\text{O}_{10}$. A completely different situation has, in fact, been found for the cobalt(III) sepulchrate halogen/pseudo-halogen compounds (Bacchi, 1995), where the role of water molecules in the packing is minimal.

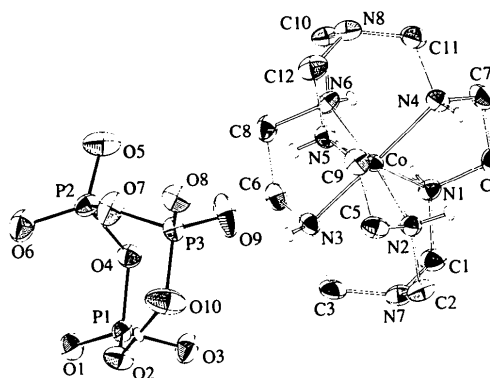


Fig. 1. ORTEP (Johnson, 1965) view of cation and anion; displacement ellipsoids are drawn at the 50% probability level.

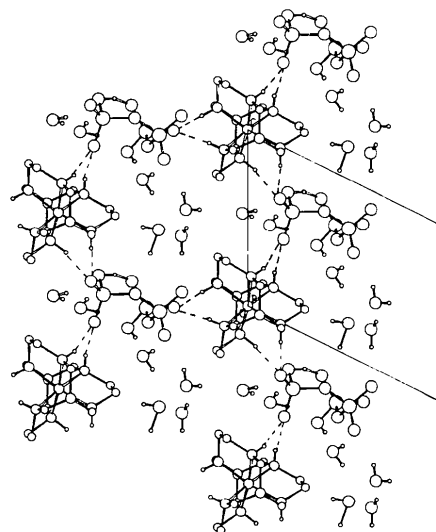


Fig. 2. Crystal packing showing the hydrogen-bonding network.

Experimental

The compound was prepared by mixing equivalent aqueous solutions of $\text{Co}(\text{sep})\text{Cl}_3$ (Bacchi *et al.*, 1993a) and $\text{Na}_3\text{H}_2\text{P}_3\text{O}_{10}$. The latter was obtained by ionic exchange of $\text{Na}_5\text{P}_3\text{O}_{10}$ on acidic resin.

Crystal data

$[\text{Co}(\text{C}_{12}\text{H}_{30}\text{N}_8)](\text{H}_2\text{P}_3\text{O}_{10}) \cdot 6\text{H}_2\text{O}$

Mo $K\alpha$ radiation
 $\lambda = 0.7107 \text{ \AA}$

$M_r = 708.38$
 Triclinic
 $P1$
 $a = 10.788 (6) \text{ \AA}$
 $b = 8.748 (4) \text{ \AA}$
 $c = 8.966 (4) \text{ \AA}$
 $\alpha = 103.91 (4)^\circ$
 $\beta = 111.14 (3)^\circ$
 $\gamma = 103.85 (5)^\circ$
 $V = 714.7 (7) \text{ \AA}^3$
 $Z = 1$
 $D_x = 1.6460 \text{ Mg m}^{-3}$
 $D_m = 1.63 \text{ Mg m}^{-3}$
 D_m measured by pycnometry

Cell parameters from 19 reflections
 $\theta = 7\text{--}18.17^\circ$
 $\mu = 0.8511 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Irregular prism
 $0.43 \times 0.26 \times 0.13 \text{ mm}$
 Orange

P2	0.5330 (1)	0.3364 (1)	0.7198 (2)	0.0268 (4)
P3	0.2364 (1)	0.1906 (1)	0.6592 (1)	0.0235 (4)
O1	0.6066 (3)	0.7675 (4)	0.7838 (5)	0.0363 (13)
O2	0.3864 (3)	0.6216 (4)	0.8122 (4)	0.0360 (15)
O3	0.3638 (3)	0.6514 (4)	0.5358 (4)	0.0371 (14)
O4	0.4717 (3)	0.4551 (4)	0.6246 (4)	0.0280 (12)
O5	0.5579 (4)	0.2167 (5)	0.5973 (6)	0.0474 (19)
O6	0.6502 (4)	0.4384 (5)	0.8914 (5)	0.0447 (16)
O7	0.4041 (3)	0.2386 (5)	0.7504 (5)	0.0392 (16)
O8	0.1706 (4)	0.0445 (4)	0.6985 (4)	0.0335 (14)
O9	0.1889 (4)	0.1649 (6)	0.4772 (5)	0.0544 (19)
O10	0.2129 (4)	0.3461 (5)	0.7555 (6)	0.0529 (19)
O11	0.4442 (5)	-0.1259 (6)	0.4208 (6)	0.050 (2)
O12	-0.0275 (5)	0.4069 (6)	0.5106 (6)	0.053 (2)
O13	0.1154 (4)	0.7166 (5)	0.5184 (5)	0.049 (2)
O14	0.6718 (6)	0.2793 (9)	0.3729 (7)	0.079 (3)
O15	0.6482 (10)	0.5142 (10)	0.2092 (9)	0.107 (5)
O16	0.5291 (8)	0.7492 (8)	0.1814 (7)	0.078 (3)

Data collection

Siemens AED diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 3808 measured reflections
 3620 independent reflections
 2808 observed reflections
 $[I > 2\sigma(I)]$
 $R_{\text{int}} = 0.03$

$\theta_{\text{max}} = 26^\circ$
 $h = -13 \rightarrow 12$
 $k = -10 \rightarrow 10$
 $l = 0 \rightarrow 11$
 1 standard reflection monitored every 50 reflections
 intensity decay: none

Refinement

Refinement on F
 $R = 0.0278$
 $wR = 0.0380$
 2667 reflections
 530 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F) + 0.005F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.20$
 $\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$

Extinction correction: none
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
 Absolute configuration: Hamilton (1965) test applied to determine the absolute configuration

Table 2. Selected geometric parameters (\AA , $^\circ$)

Co—N1	1.963 (5)	N7—C3	1.431 (7)
Co—N2	1.979 (5)	N8—C10	1.420 (9)
Co—N3	1.993 (4)	N8—C11	1.436 (7)
Co—N4	1.968 (4)	N8—C12	1.44 (9)
Co—N5	1.989 (6)	C4—C7	1.488 (9)
Co—N6	1.984 (6)	C5—C9	1.490 (9)
N1—C1	1.511 (8)	C6—C8	1.510 (8)
N1—C4	1.495 (5)	P1—O1	1.477 (3)
N2—C2	1.513 (8)	P1—O2	1.507 (4)
N2—C5	1.479 (9)	P1—O3	1.557 (4)
N3—C3	1.498 (8)	P1—O4	1.601 (4)
N3—C6	1.480 (8)	P2—O4	1.610 (4)
N4—C7	1.479 (8)	P2—O5	1.473 (5)
N4—C11	1.509 (8)	P2—O6	1.469 (4)
N5—C9	1.484 (9)	P2—O7	1.600 (5)
N5—C12	1.500 (9)	P3—O7	1.588 (3)
N6—C8	1.484 (5)	P3—O8	1.491 (4)
N6—C10	1.501 (7)	P3—O9	1.463 (5)
N7—C1	1.420 (9)	P3—O10	1.555 (5)
N7—C2	1.439 (9)		
N5—Co—N6	90.9 (2)	C11—N8—C12	112.8 (5)
N4—Co—N6	91.9 (2)	C10—N8—C12	113.4 (5)
N4—Co—N5	89.6 (2)	C10—N8—C11	115.3 (5)
N3—Co—N6	85.1 (2)	N1—C1—N7	113.3 (5)
N3—Co—N5	93.1 (2)	N2—C2—N7	112.6 (5)
N3—Co—N4	176.0 (2)	N3—C3—N7	113.4 (5)
N2—Co—N6	173.9 (2)	N1—C4—C7	107.4 (4)
N2—Co—N5	84.6 (2)	N2—C5—C9	107.2 (5)
N2—Co—N4	92.2 (2)	N3—C6—C8	106.7 (5)
N2—Co—N3	90.9 (2)	N4—C7—C4	107.3 (4)
N1—Co—N6	94.2 (2)	N6—C8—C6	106.5 (4)
N1—Co—N5	173.6 (2)	N5—C9—C5	106.5 (5)
N1—Co—N4	86.3 (2)	N6—C10—N8	113.7 (5)
N1—Co—N3	91.2 (2)	N4—C11—N8	113.7 (5)
N1—Co—N2	90.6 (2)	N5—C12—N8	113.5 (5)
Co—N1—C4	107.8 (3)	O3—P1—O4	100.3 (2)
Co—N1—C1	115.5 (4)	O2—P1—O4	108.7 (2)
C1—N1—C4	112.6 (4)	O2—P1—O3	108.4 (2)
Co—N2—C5	109.4 (4)	O1—P1—O4	109.8 (2)
Co—N2—C2	115.6 (4)	O1—P1—O3	111.3 (2)
C2—N2—C5	112.7 (5)	O1—P1—O2	117.1 (2)
Co—N3—C6	108.5 (3)	O6—P2—O7	105.8 (3)
Co—N3—C3	115.1 (3)	O5—P2—O7	110.0 (3)
C3—N3—C6	112.9 (5)	O5—P2—O6	119.0 (3)
Co—N4—C11	115.2 (3)	O4—P2—O7	103.5 (2)
Co—N4—C7	108.1 (3)	O4—P2—O6	110.8 (2)
C7—N4—C11	113.1 (4)	O4—P2—O5	106.7 (2)
Co—N5—C12	115.0 (4)	O9—P3—O10	111.7 (3)
Co—N5—C9	108.2 (4)	O8—P3—O10	107.5 (2)
C9—N5—C12	112.9 (5)	O8—P3—O9	115.9 (2)
Co—N6—C10	115.2 (4)	O7—P3—O10	103.8 (3)
Co—N6—C8	108.4 (4)	O7—P3—O9	109.7 (2)
C8—N6—C10	112.5 (4)	O7—P3—O8	107.5 (2)
C2—N7—C3	113.6 (5)	P1—O4—P2	130.1 (3)
C1—N7—C3	114.8 (5)	P2—O7—P3	134.1 (3)
C1—N7—C2	114.7 (5)		

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

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

	x	y	z	U_{eq}
Co	0.0000	0.0000	0.0000	0.0159 (2)
N1	-0.0414 (5)	0.0923 (5)	-0.1839 (5)	0.0234 (17)
N2	-0.1604 (4)	0.0408 (5)	0.0390 (5)	0.0243 (15)
N3	0.1342 (4)	0.2270 (5)	0.1736 (5)	0.0230 (15)
N4	-0.1252 (4)	-0.2218 (5)	-0.1810 (5)	0.0244 (15)
N5	0.0187 (5)	-0.1064 (5)	0.1757 (6)	0.0247 (16)
N6	0.1723 (5)	-0.0330 (5)	-0.0120 (6)	0.0234 (15)
N7	-0.0584 (5)	0.3161 (5)	0.0236 (6)	0.0287 (18)
N8	0.0615 (5)	-0.3153 (5)	-0.0193 (6)	0.030 (2)
C1	-0.0615 (7)	0.2597 (7)	-0.1405 (7)	0.032 (3)
C2	-0.1724 (6)	0.2102 (7)	0.0418 (9)	0.033 (3)
C3	0.0785 (6)	0.3675 (6)	0.1651 (8)	0.032 (2)
C4	-0.1660 (6)	-0.0407 (7)	-0.3403 (6)	0.032 (2)
C5	-0.1582 (6)	0.0061 (7)	0.1933 (7)	0.030 (2)
C6	0.2718 (5)	0.2607 (6)	0.1641 (7)	0.0309 (19)
C7	-0.1479 (6)	-0.2054 (7)	-0.3484 (6)	0.032 (2)
C8	0.2998 (5)	0.0976 (6)	0.1400 (7)	0.031 (2)
C9	-0.1162 (6)	-0.1443 (6)	0.1925 (7)	0.029 (2)
C10	0.1742 (6)	-0.2080 (6)	-0.0347 (8)	0.031 (2)
C11	-0.0776 (6)	-0.3668 (7)	-0.1581 (7)	0.033 (2)
C12	0.0639 (7)	-0.2559 (7)	0.1469 (8)	0.034 (2)
P1	0.4634 (1)	0.6353 (1)	0.7025 (1)	0.0243 (4)

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

D—H...A	D—H	H...A	D...A	D—H...A
N3—H3N...O9	0.77 (10)	2.785 (7)	2.09 (10)	152 (8)
N5—H5N...N3	0.76 (7)	2.892 (7)	2.61 (10)	104 (8)
N5—H5N...O9	0.76 (7)	2.750 (6)	2.03 (7)	158 (9)
O2—H2P...O7	1.27 (7)	3.326 (6)	2.74 (9)	106 (4)
O10—H2P...O4	1.23 (7)	3.422 (7)	2.90 (9)	104 (4)
O11—H1O1...O5	0.73 (7)	2.748 (6)	2.07 (8)	154 (9)
O12—H1O2...O13	0.96 (8)	2.748 (7)	1.79 (8)	176 (7)
O12—H2O2...O9	0.84 (8)	3.548 (8)	2.88 (9)	138 (7)
O12—H2O2...O10	0.84 (8)	2.991 (7)	2.18 (8)	164 (8)
O13—H1O3...O2	0.92 (9)	3.583 (6)	2.93 (8)	129 (6)
O13—H1O3...O3	0.92 (9)	2.829 (6)	1.95 (9)	161 (8)
O14—H2O4...O5	0.81 (9)	2.80 (1)	2.03 (9)	160 (7)
O15—H2O5...O14	0.80 (9)	2.81 (1)	2.05 (9)	159 (8)
O13—H2O3...O8 ⁱ	0.84 (7)	2.730 (6)	1.91 (8)	165 (8)
O3—H1P...O11 ⁱ	0.93 (8)	2.532 (7)	1.61 (9)	171 (8)
N1—H1N...O8 ⁱⁱ	0.95 (10)	2.916 (8)	2.02 (10)	155 (9)
N6—H6N...O8 ⁱⁱ	0.96 (10)	2.831 (7)	1.88 (10)	172 (8)
N1—H1N...O10 ⁱⁱ	0.95 (10)	3.390 (7)	2.59 (10)	141 (8)
O15—H1O5...O6 ⁱⁱ	0.88 (9)	2.78 (1)	1.92 (9)	166 (7)
O16—H1O6...O2 ⁱⁱ	1.24 (7)	2.881 (7)	1.66 (6)	165 (5)
N2—H2N...O1 ⁱⁱⁱ	0.89 (6)	2.759 (5)	1.89 (6)	164 (8)
N4—H4N...O1 ⁱⁱⁱ	0.80 (10)	2.771 (6)	2.00 (10)	163 (9)
O11—H2O1...O16 ^{iv}	0.82 (10)	2.72 (1)	1.97 (9)	152 (8)
O14—H1O4...O12 ^v	0.76 (9)	2.830 (8)	2.18 (8)	144 (9)

Symmetry codes: (i) $x, 1 + y, z$; (ii) $x, y, z - 1$; (iii) $x - 1, y - 1, z - 1$; (iv) $x, y - 1, z$; (v) $1 + x, y, z$.

Program(s) used to solve structure: *SHELX76* (Sheldrick, 1976). Program(s) used to refine structure: *SHELX76*. Molecular graphics: *ORTEP* (Johnson, 1965), *PLUTO* (Motherwell & Clegg, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *ABRAHAMS* (Gilli, 1977).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1022). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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catena-Poly[*cis*-dichloro-*trans*-dimethyltin(IV)- μ -*meso*-1,2-bis(propylsulfinyl)ethane-O:O']

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Abstract

The structure of the title compound, $[\text{SnCl}_2(\text{CH}_3)_2(\text{C}_8\text{H}_{18}\text{O}_2\text{S}_2)]_n$, has been determined and revealed the occurrence of infinite chains of tin complexes bridged by disulfoxide derivatives. The Sn^{IV} atom is hexacoordinate in a distorted octahedral environment, with Cl atoms at distances of 2.521 (1) and 2.515 (1) Å, and methyl carbanions at distances of 2.110 (4) and 2.111 (4) Å. Two different disulfoxide groups, with Sn—O distances of 2.316 (3) and 2.320 (3) Å, complete the coordination. NMR spectroscopy indicated the occurrence of discrete binuclear molecules in solution.

Comment

Dihalide-organotin compounds form adducts with a number of ligands and exhibit an important variety of structural features depending on the binding mode of the ligand. A number of these adducts of organotin and sulfoxide derivatives have been studied due to their potential application in biological systems (de Sousa, Filgueiras, Darenbourg & Reibenspies, 1992).