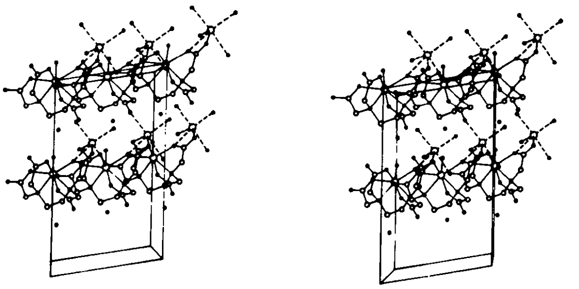

Fig. 2. Packing diagram for $Na[Fe(C_{10}H_{12}N_2O_8)(H_2O)].2H_2O$ as viewed down the *b* axis. The origin lies at the top back left corner with the *a* axis pointing to the right, the *b* axis projecting out of the plane, and the *c* axis pointing down. The dashed lines show the coordination about the sodium ions.

was fixed in the earlier study as the polar space group necessitates. The e.s.d.'s of the atom positions reported here are lower by an average factor of approximately 8×10^{-2}. With the more precise data, the apparent difference in C—C bond distances in the two types of glycinate rings (those containing a coordinating carboxyl oxygen in the equatorial plane of the pseudo pentagonal bipyramid and those containing a coordinated carboxyl oxygen axial to this plane) indicated in the earlier study disappears. On the other hand, these more precise data do indicate the same trend in Fe—O bond lengths and carboxyl C—O bond lengths observed by Hamor, Hamor & Hoard (1964) for $Li[Fe(C_{10}H_{12}N_2O_8)(H_2O)].H_2O$. In that structure it was observed that as the Fe—O bond length increased, the C—O (coordinated) bond length decreased and the C—O (uncoordinated) bond length increased in the carboxylate groups. Fig. 2 presents a packing diagram for the structure.

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Bis{μ-[4-chloro-1,3-benzenedithiolato(2—)-*S*,*S*′]}-bis[diphenyltin(IV)], $[Sn_2(C_6H_3ClS_2)_2(C_6H_5)_4]$

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Abstract. $M_r = 895\cdot2$, monoclinic, $C2/c$, $a = 15\cdot886\,(6)$, $b = 7\cdot560\,(3)$, $c = 28\cdot883\,(9)$ Å, $\beta = 100\cdot93\,(5)°$, $U = 3406$ Å^3, $Z = 4$, $D_x = 1\cdot746$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot89$ mm^{-1}, $F(000) = 1760$, $T = 294\,(1)$ K, final $R = 0\cdot062$ for 1805 unique X-ray diffractometer data ($F_o > 3\sigma$) and 200 refined parameters. The title compound has been found to be dimeric. A twelve-membered ring is formed by two tetrahedrally bonded Sn atoms and two 4-chloro-1,3-benzenedithiolato moieties. The bond lengths conform with those of similar compounds; there are some differences in bond angles which may be ascribed to packing effects and intramolecular strains.

Introduction. Bis(1,2-dithiolato)tin(IV) and (1,2-dithiolato)diorganotin(IV) compounds have been known for several years, while the appropriate 1,3-dithiolates have been prepared by us only recently. For the diorganotin derivatives, *e.g.* for R_2Sn(tdt) ($R = CH_3$, C_6H_5; H_2tdt = 3,4-toluenedithiol) a monomeric structure was proposed (Epstein & Straub, 1965; Poller & Spillman, 1966), but it was also shown that tin

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can add donor ligands (Poller & Ruddick, 1973). X-ray structure determinations of $[(C_2H_5)_4N][(C_6H_5)_2Sn(tdt)Cl]$ and $[(CH_3)_4N][Sn(tdt)_2Cl]$ revealed monomeric anionic moieties with pentacoordinated tin (Sau, Day & Holmes, 1981), while a polymeric structure with hexacoordinated tin was inferred from Mössbauer data (Poller & Ruddick, 1972). The (1,3-dithiolato)diorganotin(IV) compounds in contrast to the 1,2-dithiolates proved to be dimeric in solution. Therefore, it seemed worth while to find out if there are also principal differences between these two types of compounds in the solid state and so we determined the structure of (4-chloro-1,3-benzenedithiolato)diphenyltin(IV) as a representative example of (1,3-dithiolato)diorganotin(IV) compounds.

Experimental. Preparation by reaction of $(C_6H_5)_2SnCl_2$ and the K^+ salt of 4-chloro-1,3-benzenedithiol in methanol at room temperature, clear colorless crystals obtained by slow evaporation of a solution in chloroform (m.p. 411 K). $\omega/2\theta$ scan, scan speed 0·6–2·5° $\min^{-1}$ in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, crystal 0·28 × 0·19 × 0·06 mm, lattice parameters from least-squares fit with 19 equally distributed reciprocal-lattice reflections in the range $8{\cdot}2 \le \theta \le 12{\cdot}8°$; 3 standard reflections every 4 h, only random deviations; 3294 reflections, $4° \le 2\theta \le 50°$, $\pm h$, $+k$, $+l$, 1805 with $F_o > 3\sigma(F_o)$; Lorentz–polarization correction, absorption correction *via* ψ scans (max., min. transmission factors 1·0, 0·68); systematic absences: hkl, $h+k=2n+1$ and $h0l$, $h=2n+1$ and $l=2n+1$, space group $C2/c$ or Cc; structure solution in $C2/c$ *via* Patterson function, ΔF syntheses and full-matrix least-squares refinements on F, non-H atoms anisotropic; before last refinement cycles H atoms placed in geometrically calculated positions (C—H 0·97 Å, C—C—H 120°) with a common temperature factor $\{B[H(2)] = 3{\cdot}1\,(8)\,Å^2\}$; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); unit weights, max. Δ/σ 0·03, no significant features in final ΔF synthesis; programs: *Enraf–Nonius Structure Determination Package* (Frenz, 1981); *ORTEP* (Johnson, 1976); *POP*1 (van de Waal, 1976).

Discussion. The structure of the title compound is shown in Fig. 1 (*ORTEP*) and in a stereoview in Fig. 2 (*POP*1). Positional parameters and the equivalent values of the anisotropic temperature factors β_{ij} are given in Table 1,* bond lengths and angles in Table 2. Each of the two Sn atoms of the molecule is tetrahedrally bonded by two C(phenyl) and two S atoms. The Sn atoms are connected *via* two 4-chloro-1,3-benzenedithiolato moieties so that a twelve-membered ring, consisting of two Sn, four S and six C atoms, is formed. The molecule has a center of symmetry.

The Sn—C, Sn—S and S—C bond lengths with the exception of one Sn—C bond are in good accordance with the sums of the corresponding covalent radii (Pauling, 1970) and are in the range of the corresponding distances in comparable organotin compounds containing tetrahedral tin: $\{(C_6H_5)_2SnS\}_3$ (Schumann, 1967), $(C_6H_5)_3SnSC_6H_2(CH_3)_3$ (Bokii, Struchkov, Kravtsov & Rokhlina, 1973), $(C_6H_5)_3SnBr$ (Preut & Huber, 1979), $(C_6H_5)_6Sn_2$ (Preut, Haupt & Huber, 1973) and $\{(CH_3)_2SnS\}_3$ (Menzebach & Bleckmann, 1975). The difference between the two Sn—C bond lengths could be caused by packing effects. The maximum deviation of

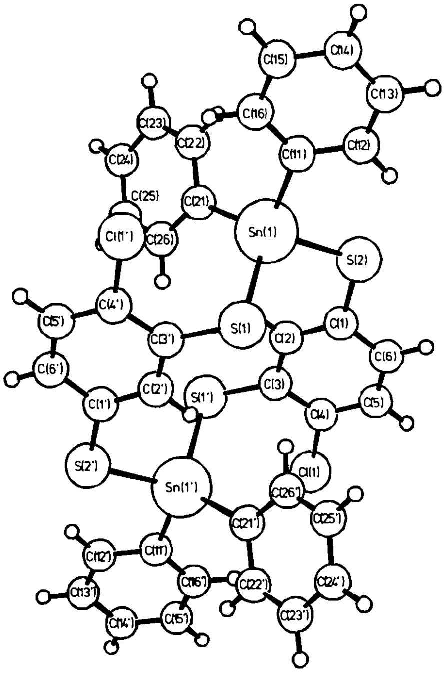

Fig. 1. General view of the molecule.

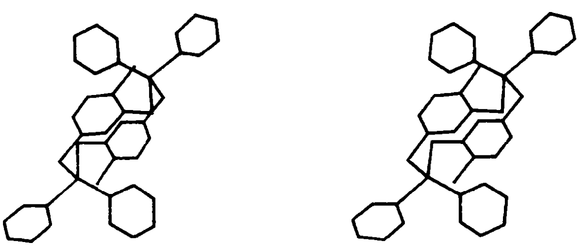

Fig. 2. Stereoscopic view of the molecule.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39257 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and temperature factors* (Å²)

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i \sum_j \beta_{ij}\mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
Sn(1)	0·12932 (7)	0·0966 (1)	0·10155 (3)	2·59 (2)
Cl(1)	−0·0813 (3)	0·3541 (6)	−0·1400 (1)	5·2 (1)
S(1)	0·1401 (3)	−0·1556 (5)	0·0515 (1)	3·23 (8)
S(2)	0·1700 (3)	0·3380 (5)	0·0554 (1)	3·64 (9)
C(1)	0·0962 (9)	0·335 (2)	0·0006 (4)	2·5 (3)
C(2)	0·0184 (9)	0·255 (2)	−0·0052 (5)	2·7 (3)
C(3)	−0·0408 (9)	0·257 (2)	−0·0485 (4)	2·4 (3)
C(4)	−0·0122 (9)	0·347 (2)	−0·0844 (5)	2·9 (3)
C(5)	0·065 (1)	0·432 (2)	−0·0791 (5)	3·7 (4)
C(6)	0·1205 (9)	0·425 (2)	−0·0364 (5)	3·1 (3)
C(11)	0·2322 (8)	0·108 (2)	0·1620 (4)	2·6 (3)
C(12)	0·3027 (9)	0·203 (2)	0·1594 (5)	3·3 (3)
C(13)	0·370 (1)	0·210 (2)	0·1976 (6)	4·6 (4)
C(14)	0·364 (1)	0·119 (2)	0·2376 (5)	5·0 (4)
C(15)	0·293 (1)	0·025 (2)	0·2412 (5)	4·3 (4)
C(16)	0·224 (1)	0·019 (2)	0·2036 (5)	3·6 (3)
C(21)	0·0119 (9)	0·122 (2)	0·1222 (5)	3·2 (3)
C(22)	0·009 (1)	0·193 (2)	0·1664 (5)	3·6 (4)
C(23)	−0·066 (1)	0·222 (2)	0·1820 (6)	4·7 (4)
C(24)	−0·144 (1)	0·187 (2)	0·1535 (6)	4·7 (4)
C(25)	−0·1446 (9)	0·115 (2)	0·1098 (6)	3·9 (3)
C(26)	−0·0700 (9)	0·083 (2)	0·0946 (5)	3·4 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

	$n = 1$	$n = 2$
Sn(1)–C(*n*1)	2·16 (1)	2·07 (1)
Sn(1)–S(*n*)	2·419 (4)	2·420 (4)
C(*n*1)–C(*n*2)	1·34 (2)	1·40 (2)
C(*n*2)–C(*n*3)	1·39 (2)	1·36 (2)
C(*n*3)–C(*n*4)	1·37 (2)	1·38 (2)
C(*n*4)–C(*n*5)	1·35 (2)	1·37 (2)
C(*n*5)–C(*n*6)	1·39 (2)	1·36 (2)
C(*n*6)–C(*n*1)	1·40 (2)	1·42 (2)

S(1)–C(3)	1·74 (1)	C(3)–C(4)	1·39 (2)
S(2)–C(1)	1·78 (1)	C(4)–C(5)	1·37 (2)
Cl(1)–C(4)	1·76 (1)	C(5)–C(6)	1·38 (2)
C(1)–C(2)	1·36 (2)	C(6)–C(1)	1·38 (2)
C(2)–C(3)	1·42 (2)		

C(11)–Sn(1)–C(21)	110·5 (5)	Sn(1)–S(2)–C(1)	106·3 (4)
C(11)–Sn(1)–S(1)	112·7 (4)	S(1)–C(3)–C(2)	118·9 (9)
C(11)–Sn(1)–S(2)	100·1 (4)	S(1)–C(3)–C(4)	127 (1)
C(21)–Sn(1)–S(1)	114·5 (4)	C(3)–C(4)–Cl(1)	117·3 (9)
C(21)–Sn(1)–S(2)	116·0 (4)	C(5)–C(4)–Cl(1)	119 (1)
S(1)–Sn(1)–S(2)	102·0 (1)	C(6)–C(1)–S(2)	116·6 (9)
Sn(1)–S(1)–C(3)	102·0 (4)	C(2)–C(1)–S(2)	123 (1)

C–C–C in the range 114 (1) to 124 (1)° [mean 120 (1)°]

	$n = 1$	$n = 2$
Sn(1)–C(*n*1)–C(*n*2)	119 (1)	119·4 (9)
Sn(1)–C(*n*1)–C(*n*6)	119·8 (9)	127 (1)

the bond angles around the Sn atom from the ideal tetrahedral angle (109·47°) amounts to 9·37 (4)°. Packing effects and intramolecular strains are probably the reason for these deformations and the significant difference of 4·3 (4)° between the two Sn–S–C angles. The Sn–S–C angles are comparable with the Sn–S–C angles in $\{(C_6H_5)_2SnS\}_3$ [104 (2)°], $(C_6H_5)_3SnSC_6H_2(CH_3)_3$ [100·2 (6)°] and $\{(CH_3)_2SnS\}_3$ [103·0 (3)°]. The S–Sn–S angle of 102·0 (1)° in the 1,3-dithiolato compound studied here is appreciably larger than the S–Sn–S angle of 84·0 (1)° in the pentacoordinated 1,2-dithiolato compound $[(C_2H_5)_4N][(C_6H_5)_2Sn(tdt)Cl]$ (Sau, Day & Holmes, 1981). Differences in some other angles of the rings containing tin can be correlated with the different sizes of the rings of these two compounds.

Intramolecular distances between Sn and the two S atoms bonded to the second Sn atom of the molecule are 5·845 (4) and 5·950 (4) Å, respectively, excluding intramolecular coordination. The intramolecular distance Sn(1)···Cl(1′) of 3·708 (5) Å is appreciably longer than the sum of the covalent radii (Cl = 0·99, Sn = 1·41 Å) but shorter than the sum of the van der Waals radii [Cl = 1·75; Sn = 2·16 Å (estimated), Bondi (1964)]. The bond angles S–Sn–S, S–Sn–C and C–Sn–C, however, exclude bonding between Sn and Cl, which exceeds the van der Waals interaction. Since intermolecular distances also do not indicate interactions exceeding van der Waals forces it can be ruled out that Sn has a coordination number higher than 4. It also seems reasonable to assume that the dimeric unit found by this structure determination is retained in solution where the presence of dimers had been shown by molecular-weight measurements.

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