and are 0·10–0·16 Å shorter than the Mn–C(S) or Mn–C(O) distances given above, suggesting that, for complexes of this type, Mn–C(S) $\simeq$ MnC(O) > Mn–N(O).

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Strontium Bis[tris(monochloroacetato)stannate(II)]

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Abstract. $Sr[Sn(CH_2ClCO_2)_3]_2$, $C_{12}H_{12}Cl_6O_{12}Sn_2Sr$, triclinic, $P\bar{1}$, $a = 10\cdot283$ (1), $b = 14\cdot878$ (1), $c = 9\cdot710$ (1) Å, $\alpha = 100\cdot196$ (8), $\beta = 115\cdot663$ (8), $\gamma = 99\cdot234$ (7)°, $M_r = 885\cdot94$, $V = 1269\cdot9$ Å³, $Z = 2$, $D_m = 2\cdot313$ (flotation, $CHBr_3/CHCl_3$), $D_c = 2\cdot317$ Mg m⁻³, $F(000) = 840$. Each asymmetric unit consists of one Sr^{2+} cation and two $[Sn(CH_2ClCO_2)_3]^-$ complex anions. Both trigonal-pyramidal Sn^{II} atoms are coordinated by three O atoms from three different unidentate chloroacetate ligands. The second O atom of each chloroacetate ligand, not bound to Sn, is involved in one contact to a Sr^{2+} cation, giving distorted octahedral coordination to Sr where the Sr–O distances range from 2·498 (7) to 2·611 (5) Å. The final R was 0·037.

Introduction. The number of crystal structures of Sn^{II} carboxylate compounds that are now known has grown considerably and among these are included $K[Sn(HCO_2)_3]$ (Jelen & Lindqvist, 1969), $Ca[Sn(CH_3CO_2)_3]_2$ (Dewan, Silver, Donaldson & Thomas, 1977), $K[Sn(CH_2ClCO_2)_3]$ (Clark, Donaldson, Dewan & Silver, 1979), $Na_2[Sn(C_2O_4)_2]$ (Donaldson, Donoghue & Smith, 1976), $Sn(O_2CCHCHCO_2).H_2O$ (Dewan, Silver, Andrews, Donaldson & Laughlin, 1977), $K_2[Sn(C_2O_4)_2].H_2O$ (Dewan, Silver, Henrick & Donaldson, 1976; Christie, Howie & Moser, 1979), and $Sn(C_2O_4)$ (Christie, Howie & Moser, 1979). Detailed reviews, concerned

entirely or in part with the structure and chemistry of Sn^{II}, are available (Donaldson, 1967; Harrison, 1976), the most recent and comprehensive of which is that by Zubieta & Zuckerman (1978). Donaldson & Jelen (1968a) have previously described the preparation of the title compound, and its structure is reported here.

The colourless crystal used in the diffraction study was bounded by ten faces: (11$\bar{1}$) and ($\bar{1}$$\bar{1}$1), 0·134 mm apart; (01$\bar{1}$) and (0$\bar{1}$1), 0·166 mm apart; and the three pairs (10$\bar{1}$), ($\bar{1}$01) and ($\bar{2}$10), (2$\bar{1}$0) and (010), (0$\bar{1}$0), each 0·20 mm apart. The quality of the data crystal was examined on the diffractometer, by taking ω scans of several strong low-angle reflections, and judged to be acceptable. The average width of the peaks at half-height was 0·13°. Intensity data, in the range $3 \leq 2\theta \leq 55°$, were collected at 298 K with the ω–2θ scan technique on an Enraf–Nonius CAD-4F κ-geometry diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda\alpha_1 = 0\cdot70926$ Å). Scan rates were automatically selected to make $I/\sigma(I) = 100$, and varied from 1 to 20° min⁻¹ in ω, with a maximum scan time of 50 s. Prescans were made at 20° min⁻¹ and reflections having $I/\sigma(I) < 1$ were considered weak and were not remeasured. Scan ranges were computed according to $\Delta\omega = (0\cdot6 + 0\cdot35 \tan \theta)°$ and were extended 25% on either side for the measurement of backgrounds. Three standard reflections, 3$\bar{6}$4, 60$\bar{3}$, and 046, measured after each 3600 s of X-ray exposure time (approximately after each 90 reflections), were

used to scale the data for a 10% isotropic decay during the course of the data collection. A total of 5882 unique reflections ($\pm h, \pm k, +l$) were scanned, the 3873 having $F_o > 4\sigma(F_o)$ being used in the structure solution and refinement. Unit-cell dimensions were obtained from a least-squares fit to the setting angles of 25 reflections with $2\theta > 30°$. Data were corrected for Lorentz and polarization effects as well as for absorption ($\mu = 4.507$ mm^{-1}), the maximum and minimum transmission factors being 0·552 and 0·417, respectively.

A systematic search using *TRACER*-II (Lawton, 1967) failed to find any symmetry higher than triclinic and refinement, performed with *SHELX* 76 (Sheldrick, 1978), has been completed in $P\bar{1}$. The structure was solved by conventional heavy-atom methods using Patterson and Fourier maps. Cl(11) is disordered over two sites, approximately 1 Å apart, and the site-occupation factors of Cl(11) and Cl(11*) were refined such that their sum was constrained to be unity. The value of this parameter for Cl(11) converged at 0·53 (4). Refinement in the noncentrosymmetric space

Table 1. *Fractional atomic coordinates* ($\times 10^5$ *for* Sr *and* Sn; $\times 10^4$ *for* Cl, O, *and* C) *with estimated standard deviations in parentheses*

Atom Cl(11) is disordered over two sites.

	x	y	z
Sr	41769 (6)	25250 (4)	21858 (7)
Sn(1)	61593 (5)	59312 (3)	29304 (6)
Cl(11)	8497 (9)	3110 (9)	5790 (27)
Cl(11*)	8185 (18)	3425 (14)	6548 (25)
Cl(12)	8613 (3)	9249 (1)	7932 (2)
Cl(13)	7651 (2)	5866 (1)	−2160 (2)
O(111)	7843 (5)	5410 (3)	4535 (5)
O(121)	7726 (5)	7303 (3)	4160 (5)
O(131)	7392 (6)	5734 (4)	1667 (6)
O(112)	6189 (5)	4042 (3)	3878 (6)
O(122)	6962 (5)	7283 (3)	5969 (6)
O(132)	6326 (5)	6482 (3)	−133 (6)
C(111)	7458 (7)	4565 (4)	4596 (7)
C(121)	7661 (7)	7706 (4)	5409 (8)
C(131)	7148 (8)	5974 (5)	403 (8)
C(112)	8803 (8)	4282 (5)	5713 (9)
C(122)	8540 (8)	8732 (5)	6097 (8)
C(132)	7989 (9)	5553 (5)	−375 (8)
Sn(2)	60909 (5)	1039 (3)	20549 (5)
Cl(21)	9584 (2)	−2054 (1)	824 (2)
Cl(22)	8056 (3)	3675 (1)	1378 (3)
Cl(23)	5371 (3)	1899 (2)	7207 (2)
O(211)	7968 (5)	−484 (3)	2944 (5)
O(221)	7774 (5)	1204 (3)	2146 (7)
O(231)	6881 (5)	645 (3)	4557 (5)
O(212)	7134 (5)	−1191 (3)	405 (5)
O(222)	6223 (5)	2092 (4)	1703 (6)
O(232)	5163 (5)	1432 (3)	3994 (6)
C(211)	8033 (7)	−1031 (4)	1819 (7)
C(221)	7394 (7)	1951 (5)	1868 (8)
C(231)	6113 (7)	1136 (4)	4929 (8)
C(212)	9331 (8)	−1475 (5)	2430 (8)
C(222)	8572 (9)	2629 (5)	1747 (12)
C(232)	6502 (8)	1297 (5)	6654 (9)

Table 2. *Bond distances* (Å) *and angles* (°) *around the Sn atoms, with estimated standard deviations in parentheses*

Sn(1)–O(111)	2·133 (5)	Sn(2)–O(211)	2·145 (5)
Sn(1)–O(121)	2·145 (4)	Sn(2)–O(221)	2·142 (6)
Sn(1)–O(131)	2·130 (7)	Sn(2)–O(231)	2·145 (5)
Sn(1)···O(112)	3·110 (5)	Sn(2)···O(212)	2·914 (6)
Sn(1)···O(122)	2·948 (5)	Sn(2)···O(222)	3·026 (6)
Sn(1)···O(132)	3·287 (6)	Sn(2)···O(232)	3·034 (6)
O(111)–Sn(1)–O(121)	86·0 (2)	O(211)–Sn(2)–O(221)	80·4 (2)
O(111)–Sn(1)–O(131)	80·8 (2)	O(211)–Sn(2)–O(231)	80·7 (2)
O(121)–Sn(1)–O(131)	85·7 (2)	O(221)–Sn(2)–O(231)	90·9 (2)

group $P1$ did not resolve the disorder. Location of H atoms was not attempted. All atoms were refined anisotropically and the weighting scheme $w = 1.8007/[\sigma^2(F_o) + 0.00017F_o^2]$ gave a satisfactory analysis of the variation of $\sum w\Delta^2$ with data sectioned according to $|F_o|$ and $(\sin \theta)/\lambda$. At convergence, no parameter shift was >0·002 of its estimated standard deviation and a final difference-Fourier map showed no significant features. The final R was 0·037 and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$ was 0·047. Neutral-atom scattering factors, and anomalous-dispersion corrections, for Sr were taken from *International Tables for X-ray Crystallography* (1974); the values contained in *SHELX* 76 were used for the remainder of the atoms. Final atomic coordinates are given in Table 1, selected bond distances and angles in Table 2.*

Discussion. The structure consists of discrete [Sn(CH$_2$ClCO$_2$)$_3$]$^-$ complex anions and simple Sr^{2+} cations in a ratio of 2:1. Each of the two crystallographically independent Sn^{II} atoms is coordinated by three O atoms from three different unidentate chloroacetate groups (Fig. 1a,b). Each three-coordinate Sn atom is at the apex of a trigonal pyramid with a stereochemically active lone pair of non-bonding electrons presumed to occupy the Sn coordination sphere opposite to the ligand O atoms. This geometry had been suggested by an earlier Mössbauer-effect study (Donaldson & Jelen, 1968b). The Sn—O bond lengths range from 2·130 (7) to 2·145 (5) Å and are not significantly shorter than any others that have been determined in similar compounds, but are all within the short range of such lengths. A list of Sn^{II}—O distances is given in Table 3 of Dewan, Silver, Donaldson & Thomas (1977). The O—Sn—O angles of the structure range from 80·4 (2) to 90·9 (2)° and, again, are not unusual when compared to those of similar compounds.

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* Lists of structure factors, bond distances and angles, and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35219 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
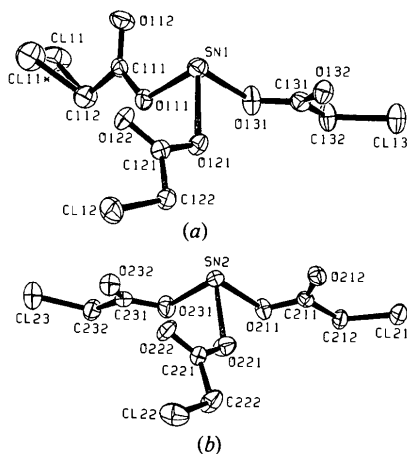
Fig. 1. Geometry and atomic numbering scheme around (a) Sn(1) and (b) Sn(2). Thermal ellipsoids are shown at the 40% probability level. Cl(11) is disordered over two sites. The diagrams were produced with ORTEP II (Johnson, 1976).

The disposition of the ligands about each of the Sn atoms in the present complex, and in K[Sn(HCO₂)₃] and K[Sn(CH₂ClCO₂)₃], is irregular. This finding is in marked contrast to the highly regular arrangement of ligands about the Sn atom in Ca[Sn(CH₃CO₂)₃]₂, which has the same stoichiometry as the present complex, but crystallizes in the trigonal crystal system. Despite the variability in the arrangement of ligands around Sn in the [Sn(ligand)₃]⁻ complex carboxylate anions, the *intramolecular* non-bonding Sn···O distances are all very similar and average 3·053 Å in the present complex, 3·02 Å in K[Sn(HCO₂)₃], 2·930 Å in Ca[Sn(CH₃CO₂)₃]₂ and 2·98 Å in K[Sn(CH₂ClCO₂)₃]. The closest *intermolecular* approach of any non-H atom to Sn is 3·450 Å in the present complex, 3·73 Å in K[Sn(CH₂ClCO₂)₃] and 4·139 Å in Ca[Sn(CH₃CO₂)₃]₂. The situation is different in K[Sn(HCO₂)₃], where there is a close 3·004 Å intermolecular contact to an O atom.

The bond distances and angles of the chloroacetate groups are similar to those in K[Sn(HCO₂)₃], Ca[Sn(CH₃CO₂)₃]₂ and K[Sn(CH₂ClCO₂)₃], and the C—Cl distances [av. 1·774 (9) Å] agree with those determined in K[Sn(CH₂ClCO₂)₃]. As expected, the C—O bond lengths for O bound to Sn [av. 1·276 (3) Å]

are significantly longer than those of the other C—O bonds [av. 1·223 (5) Å].

The shortest Sn···Sn distance in the structure is 3·555 (1) Å, being somewhat shorter than the 4·15 Å in K[Sn(HCO₂)₃], and these are both considerably shorter than the 5·073 Å in Ca[Sn(CH₃CO₂)₃]₂ and the 6·43 Å in K[Sn(CH₂ClCO₂)₃]. The closest Sr···Sn distance is 4·381 (1) Å.

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