

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

Li—O1	2.080 (5)	C12—O4	1.433 (3)
Li—O2	2.067 (5)	O4—C13	1.373 (3)
Li—O3	2.060 (6)	C13—C14	1.377 (4)
Li—O4	2.041 (5)	C13—C18	1.377 (4)
Li—O5	1.824 (6)	C14—C15	1.397 (4)
C1—O1	1.441 (4)	C15—C16	1.364 (5)
C1—C2	1.504 (5)	C16—C17	1.380 (5)
C2—C3	1.513 (5)	C17—C18	1.385 (4)
C3—O2	1.439 (4)	C18—O1	1.378 (3)
O2—C4	1.377 (3)	C19—C20	1.515 (4)
C4—C5	1.377 (4)	C20—O5	1.220 (4)
C4—C9	1.388 (4)	C20—O6A	1.24 (1)
C5—C6	1.384 (4)	C20—O6B	1.24 (1)
C6—C7	1.358 (4)	O7A—C21	1.27 (2)
C7—C8	1.392 (4)	O7B—C22	1.19 (2)
C8—C9	1.378 (4)	C21—C'21	1.49 (3)
C9—O3	1.376 (3)	C22—C'22	1.55 (3)
O3—C10	1.433 (3)	O6A···O7A	2.66 (2)
C10—C11	1.513 (4)	O6A···O7B	2.87 (2)
C11—C12	1.520 (4)	O6B···O7A	2.76 (4)
C11—C19	1.539 (4)	O6B···O7B	2.66 (2)
O1—C1—C2	108.1 (3)	O4—C13—C14	125.2 (3)
C1—C2—C3	115.1 (3)	C13—C14—C15	119.3 (3)
C2—C3—O2	108.4 (3)	C14—C13—C18	120.0 (2)
C3—O2—C4	117.8 (2)	C14—C15—C16	120.6 (3)
O2—C4—C9	114.5 (2)	C15—C16—C17	120.1 (3)
O2—C4—C5	125.5 (3)	C16—C17—C18	119.6 (3)
C4—C5—C6	119.6 (2)	C17—C18—C13	120.4 (3)
C5—C4—C9	120.1 (3)	C17—C18—O1	125.0 (3)
C5—C6—C7	120.4 (3)	C18—O1—C1	118.5 (2)
C6—C7—C8	120.6 (4)	C10—C11—C19	109.1 (2)
C7—C8—C9	119.3 (3)	C12—C11—C19	108.7 (2)
C8—C9—C4	120.0 (3)	C11—C19—C20	113.6 (2)
C8—C9—O3	125.1 (2)	C19—C20—O5	118.0 (3)
C9—O3—C10	118.4 (2)	C19—C20—O6A	113.5 (7)
O3—C10—C11	109.3 (2)	C19—C20—O6B	120.4 (8)
C10—C11—C12	113.5 (3)	O5—C20—O6A	126.0 (6)
C11—C12—O4	109.4 (2)	O5—C20—O6B	117 (1)
C12—O4—C13	117.5 (2)	O7A—C21—C'21	106 (2)
O4—C13—C18	114.8 (3)	O7B—C22—C'22	120 (1)

Primed atoms have been transformed by $1 - x, -y, -z$.

Sachleben, 1990). Of special interest is whether the side arm forms *intramolecular* bonds to cations complexed by the crown, because such bonding is assumed to be present to explain the solution behavior of similar molecules (Adamic, Eyring, Petrucci & Bartsch, 1985; Dutton, Fyles & McDermid, 1988).

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Structure of Dibutanidobis(2,4,6-trimethylbenzoato)tin

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Abstract. $[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_{10}\text{H}_{11}\text{O}_2)_2]$, $M_r = 559.3$, monoclinic, $P2_1/n$, $a = 12.07$ (2), $b = 9.976$ (10), $c = 22.76$ (4) Å, $\beta = 91.60$ (10)°, $V = 2739.5$ Å³, $Z = 4$, $D_x = 1.356$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 9.63$ cm⁻¹, $F(000) = 1160$, $T \approx 203$ K, $R = 0.0699$ for

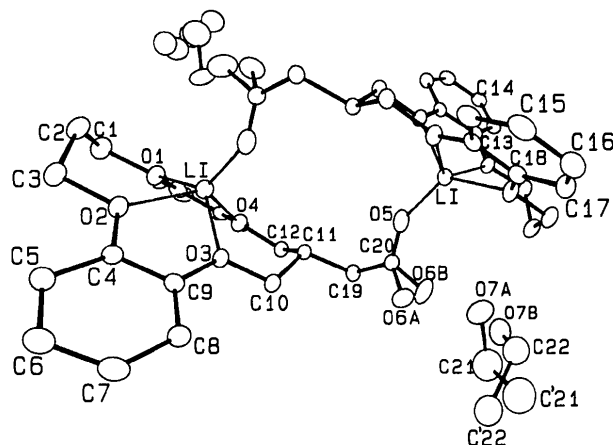


Fig. 1. One centrosymmetric dimer, 20% probability ellipsoids, numbered as in Table 1. Both sites shown for disordered ethanol.

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Sn	3784 (1)	6653 (1)	399 (1)	31 (1)
O(1)	3008 (7)	6822 (9)	1381 (3)	35 (3)
O(2)	2745 (6)	8205 (8)	659 (3)	34 (3)
O(3)	4819 (7)	5990 (9)	-571 (3)	36 (3)
O(4)	3755 (7)	7737 (8)	-400 (3)	42 (3)
C(1)	2570 (10)	7858 (12)	1192 (6)	30 (5)
C(2)	1839 (9)	8680 (10)	1563 (4)	23 (4)
C(3)	2040 (10)	8801 (11)	2165 (5)	27 (4)
C(4)	1287 (11)	9533 (12)	2499 (6)	39 (5)
C(5)	360 (11)	10104 (14)	2245 (6)	40 (5)
C(6)	187 (10)	10026 (13)	1642 (6)	40 (5)
C(7)	913 (10)	9288 (12)	1291 (5)	36 (5)
C(8)	3014 (10)	8177 (14)	2475 (5)	43 (5)
C(9)	-433 (11)	10868 (14)	2622 (6)	54 (6)
C(10)	627 (10)	9237 (15)	643 (5)	55 (6)
C(11)	4346 (10)	7015 (14)	-751 (5)	34 (5)
C(12)	4482 (10)	7536 (12)	-1351 (5)	32 (5)
C(13)	5483 (10)	8169 (13)	-1502 (5)	36 (5)
C(14)	5561 (11)	8662 (12)	-2066 (6)	39 (5)
C(15)	4727 (12)	8524 (14)	-2485 (5)	42 (5)
C(16)	3740 (12)	7882 (13)	-2335 (5)	49 (6)
C(17)	3631 (10)	7408 (13)	-1755 (6)	41 (5)
C(18)	6418 (9)	8301 (15)	-1074 (5)	51 (5)
C(19)	4830 (12)	9020 (14)	-3104 (5)	64 (6)
C(20)	2556 (10)	6717 (17)	-1606 (5)	65 (6)
C(21)	2706 (9)	5054 (12)	160 (5)	35 (4)
C(22)	1734 (9)	4802 (12)	562 (5)	32 (4)
C(23)	1032 (9)	3619 (12)	326 (5)	32 (4)
C(24)	65 (9)	3334 (16)	716 (5)	51 (5)
C(25)	5342 (9)	7168 (12)	754 (6)	41 (5)
C(26)	5479 (13)	8576 (16)	956 (9)	106 (9)
C(27)	6627 (16)	8817 (20)	1363 (9)	115 (11)
C(28)	7282 (17)	8875 (20)	886 (9)	135 (12)

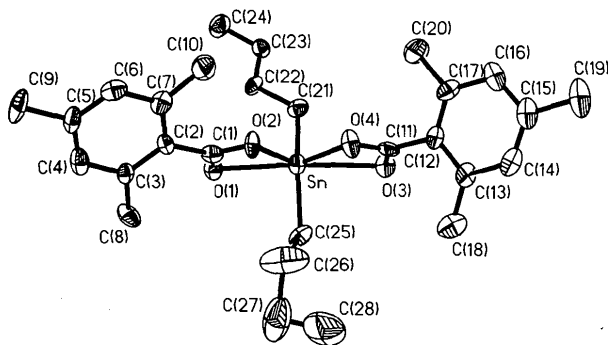


Fig. 1. Thermal ellipsoid (50% probability) plot containing the numbering scheme employed. H atoms have been omitted for clarity.

SnO_4 plane of 0.007 \AA . The carboxylate ligands are anisobidentate with short Sn—O bond lengths of 2.116 (9) and 2.088 (9) \AA and long Sn—O bond lengths of 2.451 (6) and 2.651 (14) \AA . The butyl groups are approximately *trans*, with a Bu—Sn—Bu angle of 145.4 (5)°.

Experimental. Dibutanidobis(2,4,6-trimethylbenzoato)tin was synthesized by reacting two equivalents

Table 2. Bond lengths (\AA) and angles (°)

Sn—O(1)	2.451 (9)	Sn—O(2)	2.088 (9)
Sn—O(3)	2.651 (9)	Sn—O(4)	2.116 (9)
Sn—C(21)	2.119 (12)	Sn—C(25)	2.089 (12)
O(1)—C(1)	1.233 (15)	O(2)—C(1)	1.283 (14)
O(3)—C(11)	1.235 (16)	O(4)—C(11)	1.303 (15)
C(1)—C(2)	1.485 (16)	C(2)—C(3)	1.391 (15)
C(2)—C(7)	1.401 (16)	C(3)—C(4)	1.405 (17)
C(3)—C(8)	1.489 (16)	C(4)—C(5)	1.369 (19)
C(5)—C(6)	1.384 (18)	C(5)—C(9)	1.509 (19)
C(6)—C(7)	1.409 (18)	C(7)—C(10)	1.505 (17)
C(11)—C(12)	1.475 (17)	C(12)—C(13)	1.414 (17)
C(12)—C(17)	1.365 (17)	C(13)—C(14)	1.379 (17)
C(13)—C(18)	1.475 (17)	C(14)—C(15)	1.374 (19)
C(15)—C(16)	1.402 (20)	C(15)—C(19)	1.502 (18)
C(16)—C(17)	1.412 (18)	C(17)—C(20)	1.515 (18)
C(21)—C(22)	1.528 (16)	C(22)—C(23)	1.541 (16)
C(23)—C(24)	1.511 (16)	C(25)—C(26)	1.486 (21)
C(26)—C(27)	1.662 (26)	C(27)—C(28)	1.361 (29)
O(1)—Sn—O(2)	56.2 (3)	O(1)—Sn—O(3)	167.7 (3)
O(2)—Sn—O(3)	136.2 (3)	O(1)—Sn—O(4)	138.7 (3)
O(2)—Sn—O(4)	82.5 (3)	O(3)—Sn—O(4)	53.7 (3)
O(1)—Sn—C(21)	92.2 (4)	O(2)—Sn—C(21)	105.2 (4)
O(3)—Sn—C(21)	84.2 (4)	O(4)—Sn—C(21)	99.7 (4)
O(1)—Sn—C(25)	89.7 (4)	O(2)—Sn—C(25)	104.4 (4)
O(3)—Sn—C(25)	86.8 (4)	O(4)—Sn—C(25)	101.5 (4)
C(21)—Sn—C(25)	145.4 (5)	Sn—O(1)—C(1)	84.9 (7)
Sn—O(2)—C(1)	100.6 (7)	Sn—O(4)—C(11)	104.4 (7)
O(1)—C(1)—O(2)	118.4 (11)	O(1)—C(1)—C(2)	121.4 (11)
O(2)—C(1)—C(2)	120.3 (10)	C(1)—C(2)—C(3)	121.2 (10)
C(1)—C(2)—C(7)	117.9 (10)	C(3)—C(2)—C(7)	120.8 (10)
C(2)—C(3)—C(4)	118.7 (11)	C(2)—C(3)—C(8)	123.0 (10)
C(4)—C(3)—C(8)	118.3 (10)	C(3)—C(4)—C(5)	121.5 (12)
C(4)—C(5)—C(6)	119.6 (12)	C(4)—C(5)—C(9)	119.6 (12)
C(6)—C(5)—C(9)	120.7 (12)	C(5)—C(6)—C(7)	120.8 (12)
C(2)—C(7)—C(6)	118.5 (11)	C(2)—C(7)—C(10)	125.1 (11)
C(6)—C(7)—C(10)	116.4 (11)	O(3)—C(11)—O(4)	120.6 (11)
O(3)—C(11)—C(12)	122.5 (11)	O(4)—C(11)—C(12)	116.7 (11)
C(11)—C(12)—C(13)	120.0 (10)	C(11)—C(12)—C(17)	119.3 (11)
C(13)—C(12)—C(17)	120.7 (11)	C(12)—C(13)—C(14)	117.8 (11)
C(12)—C(13)—C(18)	121.5 (10)	C(14)—C(13)—C(18)	120.8 (11)
C(13)—C(14)—C(15)	122.8 (12)	C(14)—C(15)—C(16)	119.2 (12)
C(14)—C(15)—C(19)	122.7 (13)	C(16)—C(15)—C(19)	118.1 (12)
C(15)—C(16)—C(17)	118.8 (12)	C(12)—C(17)—C(16)	120.6 (12)
C(12)—C(17)—C(20)	121.6 (11)	C(16)—C(17)—C(20)	117.7 (11)
Sn—C(21)—C(22)	116.5 (8)	C(21)—C(22)—C(23)	110.0 (9)
C(22)—C(23)—C(24)	111.5 (10)	Sn—C(25)—C(26)	116.3 (9)
C(25)—C(26)—C(27)	113.1 (13)	C(26)—C(27)—C(28)	93.2 (16)

of trimethylbenzoic acid with $\text{Bu}_2\text{Sn}(\text{O})$ in refluxing toluene. Clear, colourless, blocky crystals were obtained by crystallization from dry pentane at 233 K. The crystal was sealed in a glass capillary for the analysis. Data were collected on a Nicolet P3/F automated diffractometer at 203 K with graphite-monochromatized $\text{Mo K}\alpha$ radiation. The crystal dimensions were approximately 0.2 \times 0.2 \times 0.2 mm. Lattice parameters were determined from 12 reflections from 5–12° (2θ). Data were collected from $h = 0$ to 13, $k = 0$ to 11, $l = -25$ to 25, with maximum $(\sin\theta)/\lambda = 0.538 \text{ \AA}^{-1}$. Two standard reflections (111 and 103) were monitored with a standard deviation in observed intensity of 1% in both cases. 4038 reflections were collected using an $\omega/2\theta$ scan. 3575 unique reflections ($R_{int} = 0.0469$) and 2286 unique observed reflections with $F \geq 3\sigma(F)$. No absorption correction was applied. Structure was solved by

direct methods using *SHELX* (Sheldrick, 1981). Full-matrix refinement was carried out, minimizing $\sum w(F_o - F_c)^2$.

H-atom positions were located by difference Fourier synthesis. In the final stages of refinement, C—H bond lengths and H—C—H angles were fixed at 0.96 Å and 109.5°. A fixed isotropic thermal parameter of 0.06 Å² was used for all H atoms. All non-H atoms were refined anisotropically. 298 parameters were refined. Final $wR = 0.0595$, $R = 0.0699$, with $w^{-1} = \sigma^2(F) + 0.0003F^2$. In the final difference Fourier map, the maximum and minimum peaks were 0.83 and -0.92 e \AA^{-3} , respectively. Max. $\Delta/\sigma = 0.006$. Mean $\Delta/\sigma = 0.001$. Scattering factors and f' , f'' were supplied by the computer programs.

Final atomic coordinates for all non-H atoms are presented in Table 1, with derived bond lengths and angles in Table 2.* A thermal ellipsoid (50%)

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54063 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

drawing containing the numbering scheme employed is shown in Fig. 1.

Related literature. Diorganotin carboxylates are industrially important as PVC stabilizers (Davis & Smith, 1982), and as catalysts for transesterification reactions, polyurethane polymerizations (Evans & Karpel, 1985), and moisture-curable silicone sealants (Gross, 1989). The compound described here has more sterically demanding carboxylate ligands than the previously reported $\text{Me}_2\text{Sn}(\text{OAc})_2$ (Lockhart, Calabrese & Davidson, 1987).

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Structure of Bis(2,3-butanedione dioximato)[(*R,S*)-1-cyanoethyl](dimethylphenylphosphine)cobalt(III)

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Abstract. $[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2(\text{C}_3\text{H}_4\text{N})\{\text{P}(\text{CH}_3)_2(\text{C}_6\text{H}_5)\}]$, $M_r = 499.39$, monoclinic, $P2_1/a$, $a = 12.972$ (4), $b = 16.019$ (7), $c = 11.438$ (5) Å, $\beta = 97.19$ (3)°, $V = 2358$ (1) Å³, $Z = 4$, $D_x = 1.407 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 8.61 \text{ cm}^{-1}$, $F(000) = 1048$, $T = 298 \text{ K}$, $R = 0.081$ for 1489 observed reflections. The *R* and *S* cyanoethyl groups have a disordered structure at the same position and a solvent water molecule links the neighboring molecules. This structure is different from the X-ray-induced racemic crystal in the crystalline state reaction.

Experimental. Recrystallized from an aqueous methanol solution. Red brown crystal, $0.35 \times 0.30 \times$

0.20 mm . Rigaku AFC5R diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters using 25 reflections in the range $20 < 2\theta < 30^\circ$, intensity measurement $2\theta \leq 45^\circ$; range of hkl : -16 to 16 , -20 to 0 , 0 to 14 ; ω - 2θ scan, scan speed $16^\circ \text{ min}^{-1}$ (2θ), scan range $(1.2 + 0.35\tan\theta)^\circ$; intensities of three standard reflections monitored every 150 reflections with only small random variations, 3385 reflections measured, 1489 observed with $|F_o| < 5\sigma(F_o)$. Intensity data corrected for Lorentz and polarization, empirical absorption correction based on ψ scans, normalized transmission factors 1.00 – 0.88 . Structure solved by direct methods with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson,