Bis(1,3-diphenylpropane-1,3-dionato)tin(II)

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Abstract. $Sn(C_6H_5COCHCOC_6H_5)_2$, monoclinic, $P2_1/a$, $a = 12 \cdot 272$ (3), $b = 20 \cdot 483$ (3), $c = 10 \cdot 473$ (3) Å, $\beta = 107 \cdot 72$ (2)°, $D_m = 1 \cdot 51$ (by flotation), $D_x = 1 \cdot 49$ g cm^{-3}, $Z = 4$. The final $R = 0 \cdot 047$ for 5056 reflexions. The Sn atom is coordinated by two β-diketonate rings, which are not crystallographically equivalent, forming a distorted trigonal bipyramid with the following Sn—O distances: Sn—O(1) $2 \cdot 167$ (3), Sn—O(2) $2 \cdot 345$ (4), Sn—O(3) $2 \cdot 151$ (4), and Sn—O(4) $2 \cdot 263$ (4) Å. O(1) and O(3) occupy the equatorial sites, and O(2) and O(4) occupy the axial sites. The equatorial and axial O—Sn—O angles are $85 \cdot 8$ (1) and $146 \cdot 8$ (1)° respectively.

Introduction. The compound used was kindly supplied by Dr Ikuko Wakeshima of the Science University of Tokyo. A yellow prismatic crystal of dimensions $0 \cdot 3 \times 0 \cdot 4 \times 0 \cdot 8$ mm, crystallized from dichloromethane, was sealed in a glass capillary in a dry nitrogen atmosphere, and was mounted along [10Ī] on a Rigaku automated diffractometer (LiF-monochromatized Mo $K\alpha$ radiation, $\lambda = 0 \cdot 7107$ Å). Data were collected by the 2θ–ω scan method up to $2\theta = 60°$. Lorentz and polarization corrections were applied but no absorption correction was made ($\mu = 10 \cdot 89$ cm^{-1}). Of the 7269 independent reflexions measured, 5056 with $F_o > 3\sigma(F)$ were used in the analysis. Systematic absences: $0k0$, k odd, and $h0l$, h odd.

The structure was solved by the heavy-atom method and refined by the block-diagonal least-squares procedure. Since the discrepancy index R converged at about $0 \cdot 30$, the atomic scattering factors for Sn, O(2) and O(4) were changed from neutral to ionic ones: Sn^{2+} and O^- (*International Tables for X-ray Crystallography*, 1974). R then converged to $0 \cdot 056$ with anisotropic temperature factors for all non-hydrogen atoms. At this stage, the positions of the H atoms were calculated assuming a C—H bond distance of $1 \cdot 08$ Å. The final cycle was calculated including H atom parameters, and converged to $R = 0 \cdot 047$, nine F_o's having been omitted, some of which were as small as 80% of the F_c's, owing to extinction. The quantity minimized was $\Sigma w(|F_o| - k|F_c|)^2$. In the final cycle,

the average parameter shifts were less than the corresponding standard deviations. A final difference Fourier map showed no extra peaks. The atomic positional parameters, with their estimated standard deviations, are given in Table 1. The atom numbering is shown in Figs. 1 and 2. All the computations were performed at the Computer Centre of the University of Tokyo, with the *UNICS* (1967) program system.[†]
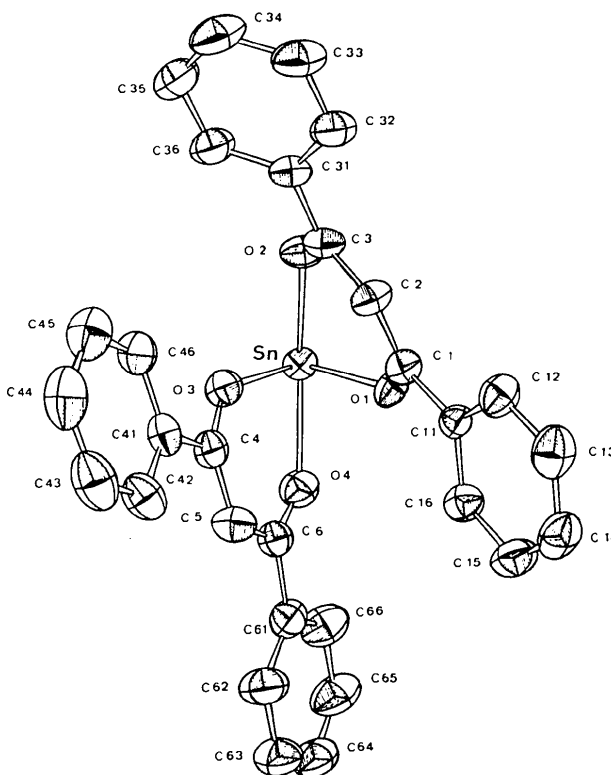
Fig. 1. Molecular structure of $Sn(C_6H_5COCHCOC_6H_5)_2$ viewed along the a axis, showing thermal ellipsoids with 50% probability (Johnson, 1965).

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Table 1. *Positional parameters* ($\times 10^4$ *for* Sn, O *and* C; $\times 10^3$ *for* H) *with their e.s.d.'s*

	x	y	z		x	y	z
Sn	5450 (0)	−295 (0)	7017 (0)	C(46)	2342 (5)	728 (3)	9550 (6)
O(1)	3902 (3)	−78 (2)	5408 (3)	C(61)	5118 (5)	1920 (3)	7294 (5)
O(2)	4386 (3)	−1253 (2)	6936 (4)	C(62)	4585 (6)	2439 (3)	7728 (7)
O(3)	4453 (3)	−96 (2)	8343 (4)	C(63)	4882 (8)	3087 (3)	7488 (8)
O(4)	5458 (3)	810 (2)	7062 (4)	C(64)	5687 (7)	3211 (3)	6863 (8)
C(1)	2868 (4)	−256 (2)	5262 (4)	C(65)	6199 (7)	2702 (4)	6440 (9)
C(2)	2556 (4)	−824 (2)	5803 (5)	C(66)	5924 (6)	2058 (3)	6647 (8)
C(3)	3308 (4)	−1292 (2)	6593 (5)	H(2)	181 (5)	−87 (3)	559 (6)
C(4)	3904 (4)	400 (2)	8549 (4)	H(5)	367 (5)	137 (3)	848 (6)
C(5)	4101 (5)	1043 (3)	8195 (6)	H(12)	62 (6)	−40 (3)	393 (7)
C(6)	4881 (4)	1216 (2)	7515 (5)	H(13)	−78 (6)	17 (3)	274 (6)
C(11)	1951 (4)	183 (2)	4454 (4)	H(14)	−33 (5)	136 (3)	236 (5)
C(12)	831 (4)	−31 (3)	3822 (5)	H(15)	160 (7)	174 (4)	339 (8)
C(13)	0 (5)	398 (3)	3072 (6)	H(16)	300 (5)	98 (3)	471 (6)
C(14)	267 (5)	1042 (3)	2945 (6)	H(32)	134 (6)	−200 (3)	545 (7)
C(15)	1380 (5)	1268 (3)	3560 (6)	H(33)	56 (6)	−285 (3)	637 (7)
C(16)	2210 (4)	844 (2)	4305 (5)	H(34)	145 (6)	−315 (3)	850 (7)
C(31)	2810 (4)	−1848 (2)	7129 (5)	H(35)	339 (5)	−276 (3)	988 (6)
C(32)	1768 (5)	−2133 (2)	6405 (6)	H(36)	403 (6)	−188 (4)	882 (7)
C(33)	1291 (5)	−2633 (2)	6960 (7)	H(42)	349 (5)	−70 (3)	953 (5)
C(34)	1828 (6)	−2841 (3)	8241 (7)	H(43)	215 (7)	−102 (4)	1062 (8)
C(35)	2867 (6)	−2569 (3)	8972 (6)	H(44)	108 (6)	−14 (3)	1115 (7)
C(36)	3355 (5)	−2076 (3)	8418 (6)	H(45)	112 (6)	86 (4)	1038 (8)
C(41)	3052 (4)	260 (2)	9275 (4)	H(46)	232 (6)	116 (4)	926 (7)
C(42)	2963 (4)	−387 (2)	9673 (5)	H(62)	402 (5)	235 (3)	812 (6)
C(43)	2184 (5)	−544 (3)	10352 (6)	H(63)	457 (6)	335 (4)	786 (6)
C(44)	1490 (5)	−61 (4)	10619 (6)	H(64)	593 (7)	368 (4)	680 (7)
C(45)	1581 (5)	569 (4)	10230 (6)	H(65)	682 (7)	281 (4)	596 (9)
				H(66)	642 (9)	176 (5)	649 (10)
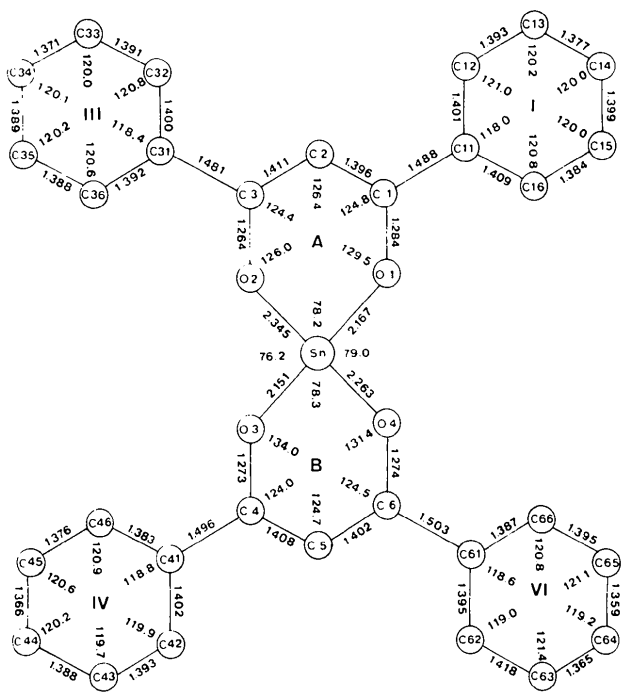
Fig. 2. Bond distances (Å) and angles (°).

Discussion. Fig. 1 shows a perspective view of the molecular structure. Bond distances and angles are shown in Fig. 2. The estimated standard deviations are within 0·004 Å and 0·1° including Sn, and 0·01 Å and 0·8° excluding Sn. Each of the four Sn—O distances lies within the range of distances found in various tin complexes (Jelen & Lindqvist, 1969; Kamenar & Grdenić, 1961; Domingos & Sheldrick, 1974a,b; Kiriyama, Kitahama, Nakamura & Kiriyama, 1973; Miller & Schlemper, 1973; van Remoortere, Flynn, Boer & North, 1971; Bancroft, Davies, Payne & Sham, 1975). Sn—O(2) and Sn—O(4) are slightly longer than Sn—O(1) and Sn—O(3), and this could be attributed to some ionic character of the bonds. Fig. 3 shows the atomic arrangement about Sn, in the form of a distorted trigonal bipyramid in which O(1), O(3) and the lone-pair electrons of Sn occupy the equatorial sites, and O(2) and O(4) occupy the axial sites. Similar arrangements are found in numerous Sn^{II} complexes (Ewings, Harrison & King, 1975; Furue, Kimura, Yasuoka, Kasai & Kakudo, 1970; Potenza & Mastropaolo, 1973; Donaldson, Nicholson & Puxley, 1973). Many five-coordinated Sn^{II} complexes are found to possess a trigonal bipyramidal configuration (Clark, O'Brien & Trotter, 1964; Forder & Sheldrick, 1970, 1971; King & Harrison, 1972; Domingos & Sheldrick, 1974a,b;

Schlemper & Britton, 1966), in which axial and equatorial angles are close to 180 and 120° respectively. In the case of the present complex and the other four-coordinated Sn^{II} complexes with a distorted trigonal bipyramidal configuration, 'lone-pair–bond-pair repulsion' gives rise to smaller bond angles than those of the typical five-coordinated Sn^{II} complexes.

The four phenyl rings, defined in Fig. 2 as I, III, IV and VI, are planar within experimental error (maximum deviation 0·009 Å). The two bidentate ligands form planes A and B, excluding Sn, which is located 0·74 Å from A and 0·35 Å from B. The C—C distances in these six planes, which range from 1·359 to 1·418 Å, indicate the existence of conjugation. The C—O distances are almost the same as those of other tin-coordinated β-diketonate rings (Ewings, Harrison & King, 1975; Miller & Schlemper, 1973; Bancroft, Davies, Payne & Sham, 1975). Planes B, IV and VI form an almost complete plane, as shown in Table 2. However, planes I and III are twisted from plane A possibly by crystal-packing effects. The closest atomic distance between two adjacent molecules is 2·980 Å for Sn—O(1'), which can be considered as a van der Waals contact (Jelen & Lindqvist, 1969). The other intermolecular atomic distances all exceed 3·0 Å.
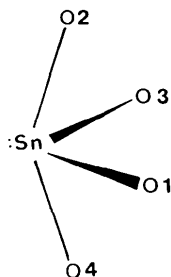

Fig. 3. Geometry of the trigonal bipyramid around Sn atom.

Table 2. *Dihedral angles between the planes*

β-Diketonate ring	Phenyl ring	Dihedral angle
A	I	22·2°
A	III	36·0
B	IV	4·6
B	VI	7·5

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