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Structure of Tetrakis(*p*-ethoxyphenyl)tin(IV)

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Abstract. $[Sn(C_2H_5OC_6H_4)_4]$, $M_r = 630{\cdot}3$, monoclinic, $P2_1/c$, $a = 12{\cdot}964\,(2)$, $b = 13{\cdot}953\,(2)$, $c = 19{\cdot}630\,(4)$ Å, $\beta = 123{\cdot}41\,(2)^\circ$, $V = 2964{\cdot}0\,(12)$ Å^3, $Z = 4$, $D_x = 1{\cdot}352$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å (Zr filter), $\mu = 9{\cdot}0$ cm^{-1}, $F(000) = 1240$, $T \simeq 295$ K, $R = 0{\cdot}040$ for 2986 observed reflections $[I > 3\sigma(I)]$. The coordination polyhedron around Sn is approximately tetrahedral with d(Sn—C) = 2·130 (6) Å and average C—Sn—C = 109·4 (2)°. Three p-C_2H_5O groups are coplanar with the attached phenyl ring while the fourth is 10° out of the ring plane. Thus, the steric requirements of the p-C_2H_5O groups are accommodated by both the loss of $\bar{4}$ symmetry at Sn and the twisting of one of the p-$C_2H_5OC_6H_4$ units from planarity. This work was carried out to confirm the crystal data of Zhdanov & Ismailzade [*Dokl. Akad. Nauk SSSR* (1949), **68**, 95–98; *Chem. Abstr.* **43**, 8764f] showing the title compound to be the first reported tetra(organo)tin without fourfold symmetry.

Experimental. The title compound was obtained by reacting excess Grignard reagent (from p-bromophenetole) with tin tetrachloride in the usual manner (Wharf & Simard, 1987); crystals from methanol, m.p. 384–385 K, lit. 377 K (Talalaeva & Kocheshkov, 1942). Enraf–Nonius CAD-4 diffractometer. Cell parameters from crystal 0·09 (100, $\bar{1}00$) × 0·26 (112, $\bar{1}\bar{1}2$) × 0·38 ($03\bar{1}$, 031) mm using 25 well centred reflections in the range $9 < \theta < 12^\circ$, $\omega/2\theta$-scan technique. Data collection over two octants ($-15 < h < 12$, $0 < k < 16$, $0 < l < 23$), ω scan, $\Delta\omega = (1{\cdot}00 + 0{\cdot}35\tan\theta)^\circ$, scan rate 4° min^{-1}, $2\theta_{\max} = 50{\cdot}0^\circ$. Six standard reflections checked every hour, largest fluctuation < ± 1·2%. Space group from analysis. A total of 5961 measured reflections which gave 5209 unique reflections, 2986 with $I > 3\sigma(I)$. Lorentz–polarization correction; no absorption correction. Structure solution was by direct methods, ΔF syntheses, and full-matrix least-squares refinement on F with all non-H atoms having anisotropic thermal parameters and H atoms being assigned isotropic thermal parameters and placed in fixed positions [C—H = 1·08 Å; $U_{\rm iso} = 0{\cdot}08$ Å^2 (phenyl) or 0·12 Å^2 (ethyl)], giving $R = 0{\cdot}040$, $wR = 0{\cdot}038$, $w = [\sigma^2(F) + 0{\cdot}0001F^2]^{-1}$, $S = 1{\cdot}47$ for 334 parameters refined. $(\Delta/\sigma)_{\max} = 0{\cdot}06$, $(\Delta/\sigma)_{\rm av} = 0{\cdot}01$, $(\Delta\rho)_{\max} = 0{\cdot}48$, $(\Delta\rho)_{\min} = -0{\cdot}33$ e Å^{-3} and two largest peaks, 0·48–0·43 e Å^{-3}, in final ΔF map at 1·07–1·17 Å from Sn, background < 0·33 e Å^{-3}. Scattering curves for C and O from Cromer & Mann (1968), for Sn from Doyle & Turner (1968) and for H from Stewart, Davidson & Simpson (1965) with anomalous-dispersion corrections from Cromer & Liberman (1970). Programs: *SHELX*76 (Sheldrick, 1976), modified versions of *NRC*-2, *NRC*-10 and *NRC*-22 (Ahmed, Hall, Pippy & Huber, 1973). Atomic parameters (non-H atoms) are given in Table 1, selected bond lengths, bond angles, and torsion angles are in Table 2. The numbering scheme is shown in Fig. 1.*

Related literature. All full Ar_4Sn crystal structures reported to date have tetragonal unit cells with $\bar{4}$ molecular symmetry (Wharf & Simard, 1987) except for $Sn[p\text{-}CH_3S(O_2)C_6H_4]_4$, space group $C2/c$, 2 molecular symmetry (Wharf, Simard & Lamparski, 1990). The structure of the title compound is best compared with that of $Sn(p\text{-}CH_3OC_6H_4)_4$ which retains $\bar{4}$ molecular symmetry and has less deviation from 'ideal' tetrahedral values of bond angles at Sn but which has the four p-CH_3O groups each 10° out of the planes of the attached phenyl rings. Kitaigorodskii (1961) has shown that an increase in

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* Lists of anisotropic temperature parameters, H-atom coordinates, least-squares planes, phenyl-ring bond lengths and angles, shorter interatomic distances, structure factors, and a stereoview of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53821 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$; Sn $\times 10^5$) *and equivalent isotropic temperature factors* (Å^2 $\times$ 10)

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

	x	y	z	U_{eq}
Sn	46597 (4)	20008 (3)	56814 (3)	53
O(1)	1214 (4)	− 641 (3)	6205 (2)	71
O(2)	1685 (4)	1824 (3)	1842 (2)	73
O(3)	3641 (4)	6228 (3)	6272 (2)	59
O(4)	10115 (5)	451 (3)	7859 (3)	74
C(11)	3568 (5)	1093 (4)	5912 (3)	50
C(12)	3895 (6)	817 (5)	6681 (4)	73
C(13)	3134 (6)	253 (5)	6807 (4)	69
C(14)	2028 (5)	− 70 (4)	6156 (3)	53
C(15)	1705 (6)	174 (5)	5382 (4)	71
C(16)	2474 (5)	737 (5)	5272 (4)	64
C(17)	1509 (6)	− 996 (5)	6984 (4)	70
C(18)	441 (6)	− 1546 (5)	6848 (4)	75
C(21)	3783 (5)	1977 (4)	4391 (3)	49
C(22)	2960 (5)	2697 (4)	3940 (4)	58
C(23)	2235 (5)	2678 (4)	3087 (4)	61
C(24)	2356 (5)	1916 (5)	2680 (4)	57
C(25)	3181 (5)	1201 (4)	3133 (4)	60
C(26)	3884 (5)	1228 (4)	3965 (3)	56
C(27)	834 (6)	2565 (5)	1350 (4)	86
C(28)	257 (7)	2294 (6)	469 (4)	110
C(31)	4458 (5)	3433 (4)	5974 (3)	50
C(32)	5050 (5)	4197 (4)	5874 (3)	51
C(33)	4828 (5)	5155 (4)	5974 (3)	50
C(34)	3968 (5)	5329 (4)	6173 (3)	47
C(35)	3379 (5)	4590 (4)	6287 (3)	57
C(36)	3626 (5)	3652 (4)	6185 (4)	56
C(37)	4160 (6)	7050 (4)	6124 (4)	67
C(38)	3651 (7)	7917 (5)	6290 (5)	86
C(41)	6535 (5)	1540 (4)	6390 (4)	53
C(42)	7099 (6)	1462 (4)	7220 (4)	59
C(43)	8279 (5)	1107 (4)	7739 (4)	62
C(44)	8958 (6)	828 (4)	7412 (4)	58
C(45)	8423 (6)	931 (4)	6586 (4)	63
C(46)	7238 (6)	1286 (4)	6081 (4)	62
C(47)	10606 (6)	191 (5)	8685 (4)	85
C(48)	11758 (7)	− 364 (6)	8990 (4)	110

Table 2. *Selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

Sn—C(11)	2·126 (7)	O(3)—C(34)	1·372 (7)
Sn—C(21)	2·130 (5)	O(3)—C(37)	1·438 (8)
Sn—C(31)	2·135 (6)	O(4)—C(44)	1·359 (9)
Sn—C(41)	2·130 (7)	O(4)—C(47)	1·424 (8)
O(1)—C(14)	1·367 (9)	C(17)—C(18)	1·495 (11)
O(1)—C(17)	1·430 (7)	C(27)—C(28)	1·506 (10)
O(2)—C(24)	1·380 (7)	C(37)—C(38)	1·497 (10)
O(2)—C(27)	1·430 (8)	C(47)—C(48)	1·484 (12)

C(11)—Sn—C(21)	105·2 (2)	C(24)—O(2)—C(27)	118·9 (5)
C(11)—Sn—C(31)	107·8 (2)	C(34)—O(3)—C(37)	119·0 (5)
C(11)—Sn—C(41)	109·6 (2)	C(44)—O(4)—C(47)	118·4 (5)
C(21)—Sn—C(31)	105·7 (2)	C(13)—C(14)—O(1)	125·3 (6)
C(21)—Sn—C(41)	116·2 (2)	C(15)—C(14)—O(1)	116·4 (6)
C(31)—Sn—C(41)	111·8 (2)	O(1)—C(17)—C(18)	107·7 (6)
Sn—C(11)—C(12)	124·1 (5)	C(23)—C(24)—O(2)	124·1 (6)
Sn—C(11)—C(16)	119·7 (5)	C(25)—C(24)—O(2)	117·4 (6)
Sn—C(21)—C(22)	118·1 (5)	O(2)—C(27)—C(28)	107·7 (6)
Sn—C(21)—C(26)	124·2 (5)	C(33)—C(34)—O(3)	123·9 (6)
Sn—C(31)—C(32)	121·4 (4)	C(35)—C(34)—O(3)	114·9 (5)
Sn—C(31)—C(36)	121·2 (5)	O(3)—C(37)—C(38)	106·8 (6)
Sn—C(41)—C(42)	118·0 (5)	C(43)—C(44)—O(4)	124·1 (6)
Sn—C(41)—C(46)	125·4 (5)	C(45)—C(44)—O(4)	117·5 (6)
C(14)—O(1)—C(17)	119·8 (5)	O(4)—C(47)—C(48)	108·2 (6)

C(13)—C(14)—O(1)—C(17)	− 1·5 (9)	C(33)—C(34)—O(3)—C(37)	3·3 (9)
C(15)—C(14)—O(1)—C(17)	176·9 (6)	C(35)—C(34)—O(3)—C(37)	− 176·8 (6)
C(14)—O(1)—C(17)—C(18)	178·4 (5)	C(34)—O(3)—C(37)—C(38)	− 179·3 (6)
C(23)—C(24)—O(2)—C(27)	2·0 (9)	C(43)—C(44)—O(4)—C(47)	9·6 (9)
C(25)—C(24)—O(2)—C(27)	− 178·6 (6)	C(45)—C(44)—O(4)—C(47)	− 169·8 (6)
C(24)—O(2)—C(27)—C(28)	178·7 (6)	C(44)—O(4)—C(47)—C(48)	169·4 (6)

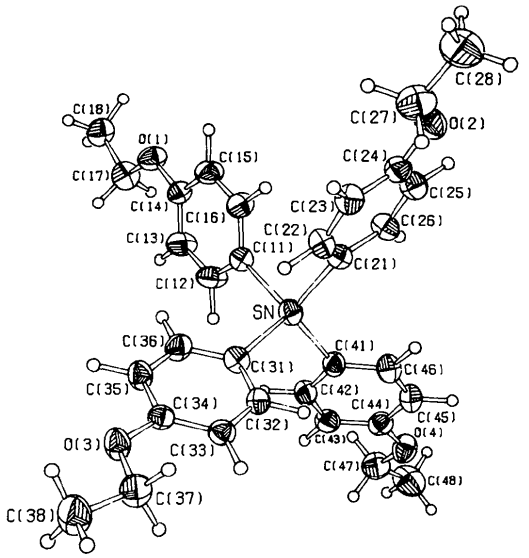

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule with the numbering scheme used.

para-substituent size would reduce the ability of Ar_4Sn to crystallize in tetragonal space groups, with lower symmetries being required to provide more efficient crystal packing.

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