Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*TEXSAN*; Molecular Structure Corporation, 1985)
$T_{\min} = 0.34$, $T_{\max} = 0.63$
3590 measured reflections
3515 independent reflections
1338 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.044$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 23$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 150 reflections intensity decay: 0.5%

Refinement

Refinement on *F*
$R = 0.0365$
$wR = 0.0545$
$S = 2.375$
1338 reflections
70 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.69$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Cd	1/4	1/4	0.0893 (5)	2.42 (2)
S1	0.0100 (1)	0.2402 (7)	0.2377 (2)	2.96 (5)
S2	0.2256 (1)	0.1232 (8)	0.4477 (2)	2.95 (5)
P	0.0377 (1)	0.1418 (7)	0.3426 (1)	2.19 (5)
O1	−0.0814 (3)	0.1297 (2)	0.4739 (4)	3.2 (2)
O2	0.0083 (4)	0.0797 (2)	0.2123 (4)	3.0 (1)
C1	−0.2317 (6)	0.1401 (6)	0.4434 (9)	5.7 (4)
C2	0.0183 (8)	0.0009 (3)	0.2476 (8)	5.5 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cd—S1	2.565 (2)	S2—P	1.988 (2)
Cd—S1$^{\rm i}$	2.565 (2)	P—O1	1.584 (3)
Cd—S2$^{\rm ii}$	2.571 (2)	P—O2	1.588 (3)
Cd—S2$^{\rm iii}$	2.571 (2)	O1—C1	1.433 (7)
S1—P	1.986 (2)	O2—C2	1.438 (6)
S1—Cd—S1$^{\rm i}$	121.17 (7)	S1$^{\rm i}$—Cd—S2$^{\rm ii}$	102.34 (4)
S1—Cd—S2$^{\rm ii}$	104.22 (4)	S1$^{\rm i}$—Cd—S2$^{\rm iii}$	104.22 (4)
S1—Cd—S2$^{\rm iii}$	102.34 (4)	S2$^{\rm ii}$—Cd—S2$^{\rm iii}$	124.23 (7)

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}-y, z$; (ii) $\frac{1}{2}-x, y, z-\frac{1}{2}$; (iii) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

The Cd atom was located by an automatic Patterson map interpretation using *DIRDIF* (Beurskens, 1984) and the remaining non-H atoms were located by Fourier syntheses. H atoms were placed in geometrically calculated positions and were not included in the refinement. The structure was refined by full-matrix least-squares methods with anisotropic displacement parameters for all atoms. Calculations were performed on a VAX3200 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

Data collection: *CONTROL* (Molecular Structure Corporation, 1988). Cell refinement: *CONTROL*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *DIRDIF*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OA1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Ito, T. (1972). *Acta Cryst.* B**28**, 1034–1040.

Ito, T., Igarashi, T. & Hagihara, H. (1969). *Acta Cryst.* B**25**, 2303–2309.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lawton, S. L. & Kokotailo, G. T. (1969). *Inorg. Chem.* **8**, 2410–2412.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *CONTROL. An Automatic Package for Rigaku AFC Single-Crystal Diffractometers*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Acta Cryst. (1996). C**52**, 3025–3027

Chlorotris(*m*-methoxyphenyl)tin(IV)

Ivor Wharf and Anne-Marie Lebuis

Chemistry Department, McGill University, 801 Sherbrooke St. W., Montreal, Quebec, Canada H3A 2K6. E-mail: wharf@omc.lan.mcgill.ca

(*Received 23 July 1996; accepted 30 September 1996*)

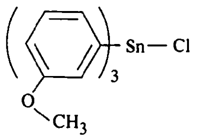
Abstract

The title compound, [SnCl(C_7H_7O)$_3$], crystallizes in the trigonal space group *R*3, with completely symmetric molecules, the Sn—Cl bond lying along the threefold axis. In contrast to the methyl analogue, the *meta*-CH_3O groups are distal, *i.e.* they are towards the Sn—Cl bond, and coplanar with the phenyl rings, with the methyl group itself lying over the *ortho*-C atom of the ring.

Comment

The effect of phenyl-ring substituents on tetraaryltin (Ar_4Sn) structures has been thoroughly investigated, with *meta* substituents creating the greatest deviations from 'ideal' tetragonal crystal and molecular structures, compared with the effect of the same group in a *para* or *ortho* position (Wharf & Simard, 1995). In contrast, the opposite is the case for triaryltin halides (Ar_3SnX).

We have shown recently that [SnCl(m-$CH_3C_6H_4$)$_3$], (A), and [SnCl{3,5-$(CH_3)_2C_6H_4$}$_3$], (B), have very symmetrical trigonal crystal and molecular structures (Wharf & Simard, 1996), unlike practically all other Ar_3SnX structures, which usually crystallize in one of the ubiquitous monoclinic space groups ($P2_1/a$, $P2_1/c$ or $P2_1/n$), with distorted trigonal molecular geometries, although with Ar = p-$CH_3S(O_2)C_6H_4$– and X = Cl, space group $Pna2_1$ is adopted (Wharf, Lebuis & Lamparski, 1996). Since increasing substituent size causes greater steric distortions in Ar_4Sn systems, the crystal structure of the title compound, (1), was determined in order to assess the effect of the same trend on *meta*-substituted Ar_3SnX structures.



(1)

The title compound, (1), crystallizes in trigonal space group $R3$, with the molecules having threefold crystallographic symmetry. The packing diagram (Fig. 1) shows completely regular three-bladed propeller molecules stacking on top of one another, with Cl—Sn···Cl′ columns along the crystal threefold axes, as in the two previously reported *meta*-substituted Ar_3SnCl structures (A) and (B) (Wharf & Simard, 1996). The resulting columns of molecules then interlock like 'cogs in gears'. Unlike (A), however, which has the methyl groups vicinal, *i.e.* away from the chloride, compound (1) has the methoxy groups distal, *i.e.* on the same side as the Cl atom (Fig. 2), directed towards the *ortho*-C atom and effectively coplanar with the phenyl ring (Table 2). This arrangement is possible because of the smaller van der Waals radius of the O atom compared with the CH_3 group (Pauling, 1960; Bondi, 1964). The result is a compact symmetric molecule which can pack even more efficiently than (A) or (B), as shown by the Sn···Cl′ distance of 4.3719 (15) Å, which is less than the same distance in (A) of 4.969 (1) Å and in (B) of 5.439 (2) Å. Distal groups also appear to flatten the molecule, as shown by the dihedral angle between the phenyl ring and the Cl—Sn—C1 planes [32.14 (9) in (1), 22.63 (8) in (A) and 25.3(2)° in (B)]. In contrast, having vicinal CH_3O groups in (1) with the same *ortho* orientation would hinder close stacking, as in (A), while placing methoxy groups vicinal or distal over the *para* position would interfere with phenyl-group atoms in neighbouring chains. In fact, all intermolecular contacts in (1) are greater than the sum of the van der Waals radii. We note that although rotation of the CH_3O group out of the plane might enable even better packing, the planar geometry is the electronically most favoured conformation (Schaeffer *et al.*, 1984). It is unusual, however, to find it so completely achieved in the solid state where crystal-packing requirements often prevail. Thus, for compound (1), the molecule in its symmetric lowest-energy conformation is able to pack closely without the molecular distortion usually required for this to happen (Brock & Dunitz, 1994).

The bromide analogue of (1), for which crystal data are reported, is isomorphous with (1) and is thus presumably isostructural, having the same close-packed highly symmetric structure.

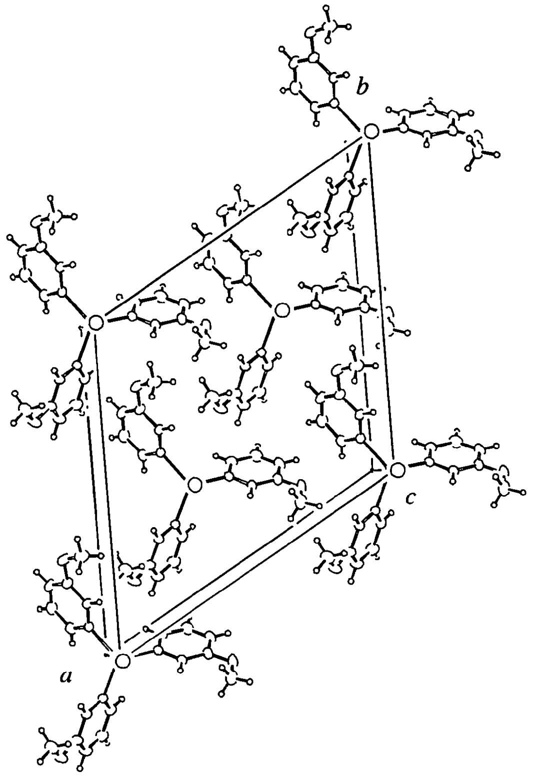


Fig. 1. *ORTEP*II plot (Johnson, 1976) of the unit cell viewed down the *c* axis, showing the interlocking of 'cogs in gears'. Ellipsoids are shown at the 30% probability level.

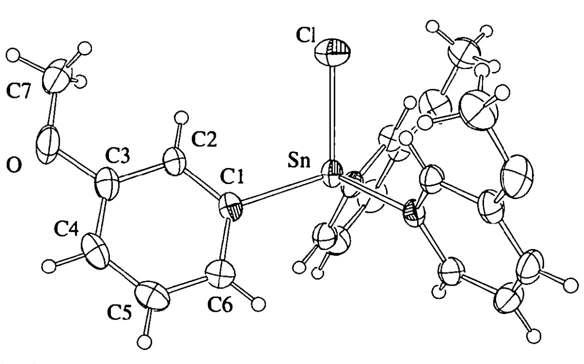


Fig. 2. *ORTEP*II plot (Johnson, 1976) of the molecular unit showing the atomic numbering scheme. Ellipsoids are shown at the 50% probability level.

Experimental

Crystals of the title compound (Wharf & Simard, 1996) were obtained by slow evaporation of an ethanol solution. The bromide analogue, also prepared at the same time, is trigonal ($R3$), with $a = 19.994\,(5)$, $c = 9.916\,(8)$ Å, $V = 1532.1\,(18)$ Å^3, $Z = 3$ and $D_x = 1.619$ Mg m^{-3}.

Crystal data

$[SnCl(C_7H_7O)_3]$
$M_r = 475.52$
Trigonal
$R3$
$a = 16.0940\,(10)$ Å
$c = 6.7170\,(10)$ Å
$V = 1506.7\,(3)$ Å^3
$Z = 3$
$D_x = 1.572$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 16–17°$
$\mu = 1.422$ mm^{-1}
$T = 293\,(2)$ K
Triangular prism
$0.20 \times 0.20 \times 0.20$ mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*NRCVAX*; Gabe, Le Page, Charland, Lee & White, 1989)
$T_{\min} = 0.68$, $T_{\max} = 0.75$
5910 measured reflections
1978 independent reflections

1971 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.054$
$\theta_{\max} = 29.94°$
$h = -22 \rightarrow 22$
$k = -22 \rightarrow 22$
$l = -9 \rightarrow 9$
3 standard reflections monitored every 200 reflections intensity decay: 1.5%

Refinement

Refinement on F^2
$R(F) = 0.0244$
$wR(F^2) = 0.0522$
$S = 1.049$
1978 reflections
82 parameters
H atoms riding (C—H = 0.93–0.96 Å)
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.524$ e Å^{-3}
$\Delta\rho_{\min} = -0.263$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0140 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.05 (2)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn	1/3	2/3	0.854	0.03155 (10)
Cl	1/3	2/3	0.5048 (2)	0.0589 (4)
O	0.7249 (2)	0.8951 (2)	0.7576 (4)	0.0611 (6)
C1	0.4802 (2)	0.7246 (2)	0.9333 (4)	0.0336 (5)
C2	0.5525 (2)	0.7927 (2)	0.8111 (4)	0.0362 (5)
C3	0.6481 (2)	0.8280 (2)	0.8648 (5)	0.0433 (6)
C4	0.6705 (2)	0.7953 (3)	1.0357 (5)	0.0528 (7)
C5	0.5983 (3)	0.7284 (3)	1.1552 (5)	0.0532 (7)
C6	0.5032 (2)	0.6933 (2)	1.1054 (4)	0.0427 (6)
C7	0.7059 (3)	0.9295 (3)	0.5791 (6)	0.0638 (9)

Table 2. *Selected geometric parameters (Å, °)*

Sn—C1	2.129 (2)	O—C3	1.371 (4)
Sn—Cl	2.3451 (14)	O—C7	1.417 (5)
Sn···Cl$^{\rm i}$	4.3719 (15)		
C1$^{\rm ii}$···Sn—C1	113.96 (6)	O—C3—C4	115.5 (3)
C1—Sn—Cl	104.49 (7)	O—C3—C2	124.2 (3)
C3—O—C7	117.8 (2)		
Cl—Sn—C1—C6	−147.6 (2)	C7—O—C3—C4	178.8 (3)
Cl—Sn—C1—C2	31.5 (2)	C7—O—C3—C2	−1.2 (5)

Symmetry codes: (i) $x, y, 1 + z$; (ii) $1 - y, 1 + x - y, z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *DATRD*2 in *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *NRCVAX*. Software used to prepare material for publication: *SHELXL*93.

The financial assistance of the Fonds FCAR of the Gouvernement du Québec is gratefully acknowledged, as well as the administration of Dawson College for supporting this research (IW) with release time. We thank Dr M. G. Simard, Université de Montréal, for providing the crystal data for the bromide analogue of the title compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1223). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Brock, C. P. & Dunitz, J. D. (1994). *Chem. Mater.* **6**, 1118–1127.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1985). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Pauling, L. (1960). In *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell University Press.

Schaeffer, T., Laatikanen, R., Wildman, T. A., Peeling, J., Penner, G. H., Baleja, J. & Marat, K. (1984). *Can. J. Chem.* **62**, 1592–1597.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Wharf, I., Lebuis, A.-M. & Lamparski, H. (1996). *Acta Cryst.* C**52**, 2477–2480, and references therein.

Wharf, I. & Simard, M. G. (1995). *Acta Cryst.* B**51**, 973–980, and references therein.

Wharf, I. & Simard, M. G. (1996). *J. Organomet. Chem.* In the press.