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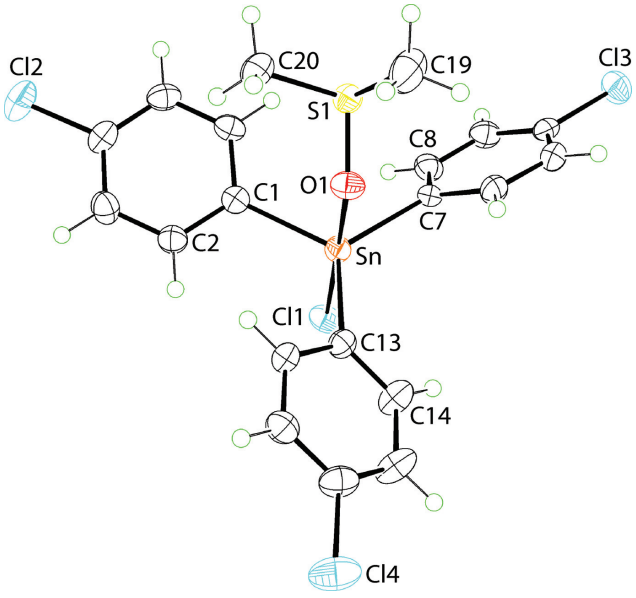
Crystal structure of chloridotris(4-chlorophenyl)(dimethyl sulfoxide-κ*O*)tin(IV), $C_{20}H_{18}Cl_4OSSn$



https://doi.org/10.1515/ncrs-2020-0291
Received June 15, 2020; accepted June 25, 2020; available online July 2, 2020

Abstract

$C_{20}H_{18}Cl_4OSSn$, orthorhombic, *Pbca* (no. 61), $a = 12.7348(2)$ Å, $b = 14.6361(3)$ Å, $c = 24.1580(6)$ Å, $V = 4502.76(16)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0337$, $wR_{ref}(F^2) = 0.0913$, $T = 100(2)$ K.

CCDC no.: 2012211

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.10 × 0.08 × 0.04 mm
Wavelength:	Cu *Kα* radiation (1.54184 Å)
μ:	14.3 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	29020, 4022, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3692
$N(param)_{refined}$:	267
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn	0.38309(2)	0.70357(2)	0.56618(2)	0.02520(10)
Cl1	0.26979(7)	0.56856(6)	0.54630(4)	0.0339(2)
Cl2	0.03535(8)	0.88698(8)	0.73813(4)	0.0493(3)
Cl3	0.39450(8)	0.83277(7)	0.30243(4)	0.0402(2)
Cl4	0.73419(9)	0.43891(8)	0.69073(6)	0.0558(3)
C1	0.2712(3)	0.7638(2)	0.62056(14)	0.0257(7)
C2	0.2540(3)	0.7258(3)	0.67251(15)	0.0310(8)
H2	0.291237	0.672310	0.683070	0.037*
C3	0.1828(3)	0.7651(3)	0.70935(15)	0.0347(8)
H3	0.172019	0.739497	0.745028	0.042*
C4	0.1280(3)	0.8422(3)	0.69297(16)	0.0329(8)
C5	0.1444(3)	0.8819(2)	0.64203(17)	0.0332(8)
H5	0.106905	0.935319	0.631709	0.040*
C6	0.2161(3)	0.8430(2)	0.60605(15)	0.0290(7)
H6	0.228140	0.870526	0.570995	0.035*
C7	0.3872(3)	0.7452(2)	0.48205(14)	0.0243(7)
C8	0.2948(3)	0.7516(2)	0.45106(16)	0.0313(8)
H8	0.229623	0.736943	0.468018	0.038*
C9	0.2962(3)	0.7787(2)	0.39638(16)	0.0331(8)
H9	0.232488	0.784476	0.376203	0.040*
C10	0.3911(3)	0.7974(2)	0.37137(16)	0.0277(8)
C11	0.4849(3)	0.7904(2)	0.40029(16)	0.0321(8)
H11	0.549969	0.802455	0.382507	0.039*
C12	0.4819(3)	0.7655(3)	0.45563(16)	0.0313(8)
H12	0.545544	0.762123	0.476012	0.038*
C13	0.5036(3)	0.6244(2)	0.60391(15)	0.0265(7)
C14	0.5535(3)	0.5546(3)	0.57537(17)	0.0366(9)
H14	0.537945	0.545409	0.537312	0.044*
C15	0.6257(3)	0.4979(3)	0.6014(2)	0.0440(10)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H15	0.660113	0.450875	0.581297	0.053*
C16	0.6466(3)	0.5109(3)	0.65672(18)	0.0371(9)
C17	0.5992(3)	0.5804(3)	0.68629(16)	0.0339(8)
H17	0.614982	0.589032	0.724371	0.041*
C18	0.5284(3)	0.6374(2)	0.65961(15)	0.0295(7)
H18	0.496327	0.685954	0.679541	0.035*
S1[a]	0.46367(9)	0.92169(8)	0.57549(5)	0.0273(4)
O1[a]	0.4970(3)	0.8234(3)	0.58971(19)	0.0301(10)
C19[a]	0.5886(4)	0.9765(4)	0.5638(2)	0.0644(16)
H19A[a]	0.632094	0.971074	0.597058	0.077*
H19B[a]	0.577281	1.041263	0.555283	0.077*
H19C[a]	0.624246	0.947029	0.532568	0.077*
C20[a]	0.4363(3)	0.9727(3)	0.64119(16)	0.0371(8)
H20A[a]	0.383314	0.936284	0.660662	0.044*
H20B[a]	0.409815	1.034870	0.635647	0.044*
H20C[a]	0.500813	0.974818	0.663281	0.044*
O1′[b]	0.4717(12)	0.8412(9)	0.5673(5)	0.030(3)
S1′[b]	0.5301(3)	0.8877(2)	0.61309(15)	0.0282(12)
C19′[b]	0.5886(4)	0.9765(4)	0.5638(2)	0.0644(16)
H19D[b]	0.611175	0.946366	0.529555	0.077*
H19E[b]	0.649112	1.005905	0.581393	0.077*
H19F[b]	0.535429	1.022769	0.555076	0.077*
C20′[b]	0.4363(3)	0.9727(3)	0.64119(16)	0.0371(8)
H20D[b]	0.387465	0.991443	0.611982	0.044*
H20E[b]	0.474978	1.026161	0.654617	0.044*
H20F[b]	0.396887	0.945354	0.671848	0.044*

[a]Occupancy: 0.762(3), [b]Occupancy: 0.238(3).

Source of material

Tetra(4-chlorophenyl)tin was synthesised from the reaction of stannic chloride (Fluka) with 4-chlorophenylmagnesium bromide (Fluka) in a 1:4 molar ratio. The subsequent tris(4-chlorophenyl)tin chloride was synthesised from the comproportionation reaction of tetra(4-chlorophenyl)tin with stannic chloride (Fluka) in a 3:1 molar ratio. Tri(4-chlorobenzyl) tin chloride (0.41 g, 1.0 mmol) was recrystallised in dimethyl sulfoxide and colourless crystals were obtained from the slow evaporation of the solvent.

Yield: 0.33 g (56%). **M.pt** (Mel-temp II digital melting point apparatus): 408–410 K. **IR** (Bruker Vertex 70v FTIR Spectrophotometer; cm^{-1}): 1475 (m) ν(C=C), 1063(s) ν(S=O), 506(m) ν(Sn–O). **^{1}H NMR** (Bruker Ascend 400 MHz NMR spectrometer, chemical shifts relative to Me_4Si, $CDCl_3$ solution at 40 °C; ppm): 2.59 (s, 6H, CH_3), 7.38–7.47 (m, 6H, Ph-H), 7.58–7.80 (m, 6H, Ph-H). **^{13}C{^{1}H} NMR** (as for ^{1}H NMR): 40.0 (CH_3), 128.6, 129.2, 136.2, 138.0 (Ph-C).

Experimental details

The C-bound H atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$. The DMSO molecule was disordered over two sites, with two positions for the S and O atoms but, common positions for the methyl groups. Each disorder component was refined independently. The major component refined to a site occupancy = 0.762(3).

Comment

Simple 1:1 diorganosulphoxide adducts of triorganotin halides are comparatively rare, being characterised crystallographically only for $Ph_3SnCl \cdot OS(Ph)CH_2Ph$ [5] and in trimorphic $Ph_3SnCl \cdot DMSO$, i.e. $P2_12_12_1$ & $Z' = 1$ [6], $P2_1$ & $Z' = 2$ [7] and $P2_1/c$ & $Z' = 2$ [8]. The common feature of the aforementioned structures is the adoption of trigonal-bipyramidal coordination geometries with the trigonal planes defined by the three ipso-C-donor atoms. In continuation of structural studies of closely related arsine adducts of triorganotin halides [9, 10], attention turned to DMSO adducts of R_3SnX resulting in the crystallographic characterisation of the title compound, $(4\text{-}ClC_6H_4)_3SnCl \cdot DMSO$ (I).

The molecular structure of (I) is shown in the figure (50% displacement ellipsoids; only the major component of the disordered DMSO molecule is shown). The Sn atom is in a distorted trigonal bipyramidal geometry defined by three ipso-C atoms of the 4-chlorophenyl groups [Sn—C1, C7 & C13 = 2.129(3), 2.122(3) & 2.128(3) Å] as well as Cl [Sn—Cl1 = 2.4934(9) Å] and DMSO-O [Sn—O1 = 2.346(4) Å] atoms. In this description, the Sn atom lies 0.1312(19) Å above the C_3 plane in the direction of the Cl1 atom. The Cl1—Sn—O1 axial angle = 175.54(11)°. The C1-, C7- and C13-phenyl rings form dihedral angles with the C_3 plane of 19.39(18), 40.10(10) and 28.97(15)°, respectively.

The key features of the molecular packing of (I) are phenyl- and methyl-C—H···π(phenyl) contacts with each of the phenyl rings accepting a contact. These contacts [C11—H11···Cg(C1–C6)^{i} = 2.64 Å with angle at H11 = 133°, C19—H19b···Cg(C7–C12)^{ii} = 2.81 Å, angle at H19b = 153° and C9—H9···Cg(C13–C18)^{iii} = 2.95 Å, angle at H9 = 132° for symmetry operations (i) $1/2 + x$, $3/2 - y$, $1 - z$, (ii) $1 - x$, $2 - y$, $1 - z$ and (iii) $-1/2 + x$, $3/2 - y$, $1 - z$] assemble molecules into a supramolecular layer in the *ab*-plane. The points of contact between layers include weak Cl···Cl, i.e. phenyl-Cl2···Cl4(phenyl)^{iv} contacts [3.4849(16) Å; (iv) $1 - x$, $1/2 + y$, $3/2 - z$] which are marginally less than the sum of the van der Waals radii of 3.50 Å [11].

References

1. Rigaku Oxford Diffraction: CrysAlis^{PRO}. Rigaku Corporation, Oxford, UK (2018).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.

3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Farrugia, L. J.: WinGX and ORTEP for Windows: an update. J. Appl. Crystallogr. **45** (2012) 849–854.
5. Tan, G.-X.; Zhang, C.-F.: (Benzyl phenyl sulfoxide-κ*O*) chloridotriphenyltin(IV). Acta Crystallogr. **E68** (2012) m304.
6. Kumar, S.; Shadab, S. M.; Idrees, M.: Chlorido(dimethyl sulfoxide-κ*O*)triphenyltin(IV). Acta Crystallogr. **E65** (2009) m1602–m1603.
7. Pouye, S. F.; Cissé, I.; Diop, L.; Ríos-Merino, F. J.; Bernès, S.: Monoclinic polymorph of chlorido(dimethyl sulfoxide-κ*O*) triphenyltin(IV). Acta Crystallogr. **E74** (2018) 163–166.
8. Owczarzak, A.; Kubicki, M.; Grześkiewicz, A. M.: Structural and thermal studies of new triphenyltin complexes. ACS Omega **4** (2019) 10311–10321.
9. Lo, K. M.; Lee, S. M.; Tiekink, E. R. T.: Crystal structure of iodido-triphenyl-(triphenylphosphine oxide)tin(IV), $C_{36}H_{30}IOPSn$. Z. Kristallogr. NCS **235** (2020) 179–181.
10. Lo, K. M.; Lee, S. M.; Tiekink, E. R. T.: Crystal structure of bromido-triphenyl-(triphenylarsine oxide-κ*O*) tin(IV), $C_{36}H_{30}AsBrOSn$. Z. Kristallogr. NCS **235** (2020) 981–983.
11. Spek, A. L.: checkCIF validation ALERTS: what they mean and how to respond. Acta Crystallogr. **E76** (2020) 1–11.