

Kong Mun Lo, See Mun Lee and Edward R. T. Tiekink*

Crystal structure of dichlorido-bis(dimethyl sulfoxide- κO)bis(4-methylphenyl- κC^1)tin(IV), $C_{18}H_{26}Cl_2O_2S_2Sn$

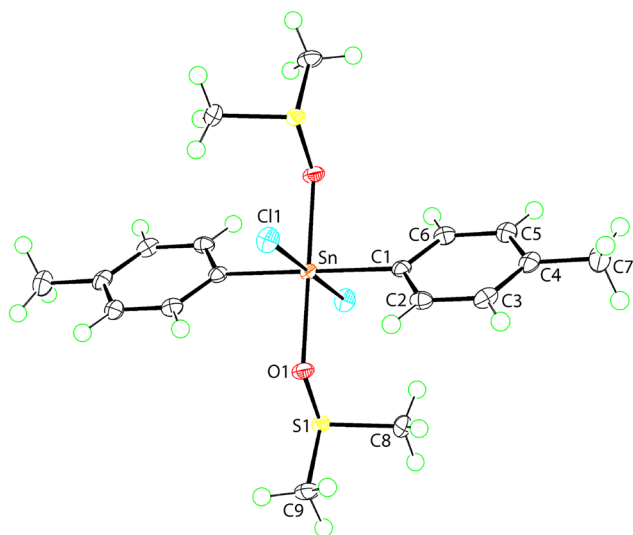


Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.13 × 0.08 × 0.06 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	14.0 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max} , completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	13,329, 1873, 0.030
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1823
$N(param)_{refined}$:	118
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

The (4-tolyl)₂SnCl₂ compound was prepared as per the literature procedure [5] and recrystallisation from its dimethyl sulphoxide (DMSO) solution gave the title compound (4-tolyl)₂SnCl₂(DMSO)₂, as colourless crystals. **M.pt** (Stuart SMP30 digital melting point apparatus; uncorrected): 421–423 K. **IR** (Bruker Vertex 70v FTIR Spectrometer; cm⁻¹): 1478 (m) ν (C=C), 950 (s) ν (S=O), 505 (m) ν (Sn–O). **¹H NMR** (Bruker Ascend 400 MHz NMR spectrometer; CDCl₃; ppm relative to Me₄Si): δ 2.45 (s, 6H, CH₃), 3.26 (s, 12H, CH₃), 7.29–7.45 (m, 4H, Ph–H), 7.56–7.67 (m, 4H, Ph–H). **¹³C{¹H} NMR** (as for ¹H NMR): 21.5 (CH₃), 40.5 (CH₃), 130.3, 133.7, 134.9, 142.4 (Ph–C).

Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$. Owing to poor agreement, one reflection, i.e. (2 1 1), was omitted from the final cycles of refinement.

Comment

Very recently, the crystal structures of (4-tolyl)₂SnCl₂ [5] and (4-ClC₆H₄)₂SnCl₂ [6] were described as a part of ongoing studies [7, 8] investigating putative examples of the chloro/methyl exchange [9]. Herein, the crystal and molecular structures of the solvated form of the former

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Abstract

$C_{18}H_{26}Cl_2O_2S_2Sn$, monoclinic, $P2_1/n$ (no. 14), $a = 8.3295(1)$ Å, $b = 10.5647(1)$ Å, $c = 12.0078(1)$ Å, $\beta = 96.612(1)^\circ$, $V = 1049.642(18)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0174$, $wR_{ref}(F^2) = 0.0455$, $T = 100$ K.

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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.

*Corresponding author: Edward R. T. Tiekink, Research Centre for Crystalline Materials, School of Medical and Life Sciences, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia, E-mail: edwardt@sunway.edu.my. <https://orcid.org/0000-0003-1401-1520>

Kong Mun Lo and See Mun Lee, Research Centre for Crystalline Materials, School of Medical and Life Sciences, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
Sn	0.500000	0.500000	0.500000	0.00827 (8)
Cl1	0.62113 (5)	0.71549 (4)	0.55534 (3)	0.01438 (11)
S1	0.11176 (5)	0.52966 (4)	0.38629 (3)	0.01040 (10)
O1	0.26659 (14)	0.59435 (11)	0.44089 (10)	0.0128 (2)
C1	0.5729 (2)	0.52221 (17)	0.33674 (16)	0.0104 (3)
C2	0.5374 (2)	0.63238 (16)	0.27488 (14)	0.0119 (3)
H2	0.479231	0.698698	0.305553	0.014*
C3	0.5865 (2)	0.64607 (17)	0.16861 (14)	0.0134 (3)
H3	0.559477	0.721190	0.127137	0.016*
C4	0.6748 (2)	0.55122 (18)	0.12170 (14)	0.0129 (4)
C5	0.7084 (2)	0.44063 (17)	0.18386 (15)	0.0126 (3)
H5	0.766491	0.374195	0.153305	0.015*
C6	0.65824 (19)	0.42608 (16)	0.28976 (14)	0.0116 (3)
H6	0.682316	0.349977	0.330432	0.014*
C7	0.7302 (2)	0.56695 (19)	0.00772 (15)	0.0180 (4)
H7A	0.804631	0.498174	-0.005631	0.027*
H7B	0.785737	0.648386	0.004102	0.027*
H7C	0.636519	0.564537	-0.049624	0.027*
C8	0.1476 (3)	0.49425 (16)	0.24576 (18)	0.0157 (4)
H8A	0.182651	0.571114	0.209801	0.024*
H8B	0.047733	0.462708	0.203753	0.024*
H8C	0.231954	0.429498	0.246658	0.024*
C9	-0.0214 (2)	0.66035 (18)	0.35802 (15)	0.0152 (4)
H9A	-0.051041	0.694421	0.428770	0.023*
H9B	-0.119076	0.632347	0.311168	0.023*
H9C	0.032512	0.726275	0.318559	0.023*

compound, (4-tolyl)₂SnCl₂, namely (4-tolyl)₂SnCl₂(DMSO)₂, hereafter (I), are described.

The molecular structure of (I) is shown in the figure (70% displacement ellipsoids). The tin atom in (I) is located on a crystallographic centre of inversion and is coordinated by two chloride atoms [Sn–Cl1 = 2.5471(4) Å], two DMSO-oxygen atoms [Sn–O1 = 2.2270(12) Å] and two ipso-carbon atoms [Sn–C1 = 2.1311(19) Å] of the 4-tolyl groups. The *all-trans* C₂Cl₂O₂ donor set approximates an octahedron.

There are several literature precedents for (I), each conforming to the general formula R₂SnCl₂(DMSO)₂. The *all-trans* isomer, with the tin atom located at a centre of inversion, as for (I), are found in two species, namely when R = CH₂C₆H₄Me-4 [10] and R = CH₂C₆H₄F-4 [11]. By contrast, for the R = Me [12] and R = Ph [13] structures, the isomer is observed whereby the chloride atoms are *cis* as are the two DMSO-oxygen atoms.

Based on the distance criteria assumed in PLATON [14], DMSO-C–H···π(tolyl) interactions [C9–H9b···Cg(C1–C6)ⁱ: H9b···Cg(C1–C6)ⁱ = 2.47 Å, C9···Cg(C1–C6)ⁱ = 3.4382(19) Å with angle at H9b = 168° for symmetry operation (i): –1+x, y, z] are the only directional intermolecular contacts in the crystal of (I). These occur within linear chains of molecules

aligned along the *a*-axis. Weak S1···S1ⁱⁱ secondary-bonding contacts of 3.5337(5) Å are noted within the chain; (ii): –x, 1–y, 1–z].

Finally, the calculation of the Hirshfeld surfaces and two-dimensional fingerprint plots (full and delineated into individual contacts) were also performed to gain additional information on the molecular packing. This was achieved using Crystal Explorer 17 [15] following literature protocols [16]. This analysis revealed that with the exception of S···S contacts [1.0%], all surface contacts involved H, with a clear dominance of H···H [59.4%] contacts. Other contributions were from Cl···H/H···Cl [16.5%], C···H/H···C [16.1%], S···H/H···S [4.3%] and O···H/H···O [2.7%] contacts.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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