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catena-Poly[[chloridodimethyltin(IV)]-μ-thiophene-2-acetato-κ^2O:O′]

Hongyun Wang, Handong Yin* and Daqi Wang

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: handongyin@163.com

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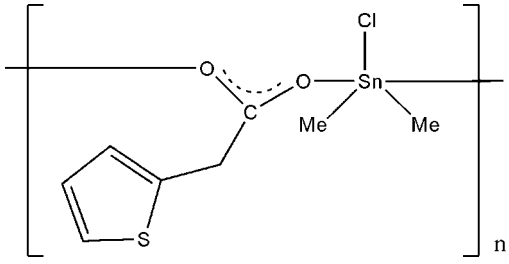
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.029; wR factor = 0.086; data-to-parameter ratio = 13.9.

The title compound, $[Sn(CH_3)_2(C_6H_5O_2S)Cl]_n$, forms an extended one-dimensional chain structure which propagates along the a-axis direction. The two independent SnO_2C_2Cl centres have distorted trigonal–bipyramidal environments with the coordinated O atoms in the axial positions. Both thiophene groups are disordered over two sites, with approximate occupancies of 0.5:0.5 and 0.78:0.22.

Related literature

For related literature, see: Ma *et al.* (2006).



Experimental

Crystal data

$[Sn(CH_3)_2(C_6H_5O_2S)Cl]$
M_r = 325.37
Triclinic, $P\overline{1}$
a = 9.2189 (18) Å
b = 10.591 (2) Å
c = 12.226 (2) Å
α = 97.898 (2)°
β = 93.287 (2)°
γ = 99.948 (2)°
V = 1160.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.58 mm^{-1}
T = 298 (2) K
0.53 × 0.42 × 0.38 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.342, T_{max} = 0.441
(expected range = 0.291–0.375)
6038 measured reflections
4020 independent reflections
3265 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.086
S = 1.00
4020 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2549).

References

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supplementary materials

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catena-Poly[[chloridodimethyltin(IV)]-μ-thiophene-2-acetato-κ^2O:O']

H. Wang, H. Yin and D. Wang

Comment

A series of new triorganotin(IV) pyridinedicarboxylates were synthesized by the reaction of trimethyltin(IV), triphenyltin(IV) or tribenzyltin(IV) chloride with 2,6(3,5 or 2,5)-H_2pdc (pdc = pyridinedicarboxylate) (Ma *et al.*,2006). The title compound, (I) (Fig. 1), has an extended one-dimensional chain structure formed from Sn—O bridges to the ligand with Sn—O distances close to those in reported organotin carboxylates (Ma *et al.*, 2006). The two independent Sn atoms are in distorted trigonal-bipyramidal coordination geometries, with the O atoms in axial positions and atoms C13/C14/Cl1 and C15/C16/Cl2 in equatorial positions. The sum of the equatorial angles at Sn1 and Sn2 of 357.2 and 356.2° indicate approximate coplanarity for the atoms involved.

Experimental

The reaction was carried out under N_2. 2-Thiopheneacetic acid (1 mmol) and sodium ethoxide (1.2 mmol) were added to the solution of benzene(30 ml) in a Schlenk flask and stirred for 0.5 h. Dimethyltin dichloride (1 mmol) was then added to the reactor and the reaction mixture was stirred for 12 h at 313 K. The resulting clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/methanol (1:1).(yield 80%; m.p. 472 K). Analysis calculated (%) for $C_{16}H_{22}Cl_2O_4S_2Sn_2$ (Mr = 650.74): C, 29.53; H, 3.41. found: C, 29.47; H, 3.52.

Refinement

During the refinement both thiophene groups were found to be disordered over two sites corresponding to an approximate 180° rotation parallel to the ring plane. The ratio of the occupancy factors refined to 0.502 (9):0.498 (2) and 0.783 (8):0.217 (8) for atoms S1/C3—C6:S1'/C3'-6' and atoms S2/C9—C12:S2'/C9'-C12', repectively. H atoms were positioned geometrically, with C—H = 0.91–0.97 Å and constrained to ride on their parent atoms, with U_{iso}(H) = U_{eq}(C) where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

Figures

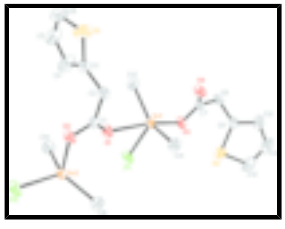

Fig. 1. The asymmetric unit showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. The disorder is not shown.

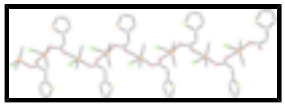

Fig. 2. The 1-D extended chain structure propagating along the *a* axis. H atoms have been omitted for clarity.

catena-Poly[[dichloridodimethyltin(IV)]-μ-thiophene-2-acetato-κ^2*O*:*O*^{1}]

Crystal data

$[Sn(CH_3)_2(C_6H_5O_2S)Cl]$	$Z = 4$
$M_r = 325.37$	$F_{000} = 632$
Triclinic, $P\overline{1}$	$D_x = 1.862$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.2189$ (18) Å	Cell parameters from 4001 reflections
$b = 10.591$ (2) Å	$\theta = 2.3$–28.1º
$c = 12.226$ (2) Å	$\mu = 2.58$ mm^{-1}
$\alpha = 97.898$ (2)º	$T = 298$ (2) K
$\beta = 93.287$ (2)º	Block, colourless
$\gamma = 99.948$ (2)º	0.53 × 0.42 × 0.38 mm
$V = 1160.6$ (4) Å^3	

Data collection

Bruker SMART CCD diffractometer	4020 independent reflections
Radiation source: fine-focus sealed tube	3265 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.017$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.7$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.342$, $T_{max} = 0.441$	$k = -10 \rightarrow 12$
6038 measured reflections	$l = -11 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 1.0989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
4020 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
289 parameters	$\Delta\rho_{min} = -0.87$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Sn1	0.62273 (3)	0.74816 (3)	0.10589 (2)	0.03419 (11)	
Sn2	1.14767 (3)	0.81755 (3)	0.23097 (3)	0.03814 (12)	
Cl1	0.79330 (14)	0.67290 (15)	−0.01443 (11)	0.0558 (4)	
Cl2	1.36682 (14)	0.88314 (17)	0.35067 (12)	0.0627 (4)	
O1	0.8452 (4)	0.8005 (4)	0.2392 (3)	0.0470 (8)	
O2	1.0249 (3)	0.8696 (4)	0.3698 (3)	0.0466 (8)	
O3	0.4573 (3)	0.6910 (3)	−0.0316 (3)	0.0456 (8)	
O4	0.3239 (4)	0.7682 (4)	0.0944 (3)	0.0525 (9)	
C1	0.8870 (5)	0.8430 (5)	0.3377 (4)	0.0392 (11)	
C2	0.7808 (6)	0.8625 (5)	0.4239 (4)	0.0496 (13)	
H2A	0.6871	0.8714	0.3883	0.060*	
H2B	0.8190	0.9420	0.4741	0.060*	
S1	0.6822 (9)	0.7729 (8)	0.6105 (6)	0.094 (2)	0.502 (9)
C3	0.7564 (6)	0.7512 (6)	0.4887 (4)	0.0546 (14)	0.502 (9)
C4	0.782 (6)	0.628 (3)	0.465 (3)	0.090 (2)	0.502 (9)
H4	0.8232	0.5987	0.4007	0.108*	0.502 (9)
C5	0.742 (19)	0.552 (14)	0.544 (14)	0.09 (2)	0.502 (9)
H5	0.7519	0.4659	0.5390	0.107*	0.502 (9)
C6	0.68 (2)	0.617 (18)	0.631 (17)	0.10 (2)	0.502 (9)
H6	0.6522	0.5811	0.6931	0.116*	0.502 (9)
S1'	0.7801 (14)	0.5963 (7)	0.4449 (7)	0.090 (2)	0.498 (9)
C3'	0.7564 (6)	0.7512 (6)	0.4887 (4)	0.0546 (14)	0.498 (9)
C4'	0.709 (4)	0.750 (3)	0.591 (3)	0.094 (2)	0.498 (9)
H4'	0.6883	0.8234	0.6328	0.113*	0.498 (9)
C5'	0.69 (2)	0.634 (17)	0.630 (16)	0.09 (2)	0.498 (9)
H5'	0.6633	0.6238	0.6993	0.111*	0.498 (9)
C6'	0.73 (2)	0.535 (15)	0.560 (15)	0.093 (16)	0.498 (9)
H6'	0.7239	0.4497	0.5729	0.111*	0.498 (9)
C7	0.3332 (5)	0.7131 (5)	−0.0002 (4)	0.0383 (10)	
C8	0.2003 (5)	0.6627 (6)	−0.0817 (4)	0.0523 (13)	
H8A	0.1862	0.5688	−0.0949	0.063*	
H8B	0.1139	0.6861	−0.0483	0.063*	
S2	0.3625 (3)	0.7310 (4)	−0.2599 (3)	0.0613 (9)	0.783 (8)
C9	0.2088 (6)	0.7109 (5)	−0.1905 (4)	0.0457 (12)	0.783 (8)
C10	0.094 (4)	0.747 (4)	−0.246 (3)	0.069 (4)	0.783 (8)
H10	0.0008	0.7441	−0.2196	0.083*	0.783 (8)
C11	0.129 (6)	0.787 (5)	−0.345 (3)	0.087 (10)	0.783 (8)
H11	0.0620	0.8143	−0.3930	0.104*	0.783 (8)
C12	0.272 (7)	0.783 (7)	−0.367 (6)	0.081 (9)	0.783 (8)
H12	0.3149	0.8060	−0.4300	0.097*	0.783 (8)
S2'	0.063 (4)	0.739 (4)	−0.267 (3)	0.069 (4)	0.217 (8)
C9'	0.2088 (6)	0.7109 (5)	−0.1905 (4)	0.0457 (12)	0.217 (8)
C10'	0.318 (6)	0.711 (6)	−0.260 (5)	0.0613 (9)	0.217 (8)
H10'	0.4020	0.6758	−0.2461	0.074*	0.217 (8)
C11'	0.30 (2)	0.77 (2)	−0.35 (2)	0.08 (4)	0.217 (8)

H11'	0.3732	0.7960	−0.3941	0.091*	0.217 (8)
C12'	0.16 (2)	0.777 (19)	−0.378 (12)	0.08 (3)	0.217 (8)
H12'	0.1162	0.8001	−0.4428	0.100*	0.217 (8)
C13	0.5429 (6)	0.6015 (5)	0.1980 (4)	0.0505 (13)	
H13A	0.5026	0.6387	0.2629	0.076*	
H13B	0.6224	0.5600	0.2197	0.076*	
H13C	0.4672	0.5387	0.1535	0.076*	
C14	0.6487 (6)	0.9496 (5)	0.1136 (5)	0.0549 (14)	
H14A	0.5993	0.9844	0.1746	0.082*	
H14B	0.6068	0.9696	0.0458	0.082*	
H14C	0.7520	0.9870	0.1239	0.082*	
C15	1.0898 (7)	0.6159 (5)	0.2174 (5)	0.0594 (15)	
H15A	1.0252	0.5940	0.2735	0.089*	
H15B	1.1774	0.5797	0.2270	0.089*	
H15C	1.0403	0.5814	0.1456	0.089*	
C16	1.1208 (7)	0.9686 (6)	0.1402 (5)	0.0617 (15)	
H16A	1.0683	0.9327	0.0696	0.093*	
H16B	1.2160	1.0160	0.1291	0.093*	
H16C	1.0658	1.0259	0.1805	0.093*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.03134 (18)	0.03700 (19)	0.03505 (19)	0.00966 (13)	0.00292 (13)	0.00393 (13)
Sn2	0.03394 (19)	0.0430 (2)	0.0377 (2)	0.00999 (14)	0.00003 (13)	0.00405 (14)
Cl1	0.0422 (7)	0.0801 (10)	0.0466 (7)	0.0228 (7)	0.0089 (6)	−0.0023 (6)
Cl2	0.0373 (7)	0.0929 (11)	0.0523 (8)	0.0124 (7)	−0.0067 (6)	−0.0057 (7)
O1	0.0382 (18)	0.062 (2)	0.038 (2)	0.0098 (16)	−0.0044 (15)	0.0012 (16)
O2	0.0342 (18)	0.067 (2)	0.0360 (18)	0.0096 (16)	0.0004 (14)	−0.0022 (16)
O3	0.0340 (17)	0.063 (2)	0.0393 (19)	0.0175 (16)	−0.0030 (14)	−0.0019 (16)
O4	0.043 (2)	0.067 (2)	0.047 (2)	0.0147 (18)	0.0086 (16)	−0.0021 (18)
C1	0.038 (3)	0.043 (3)	0.039 (3)	0.012 (2)	0.000 (2)	0.010 (2)
C2	0.044 (3)	0.065 (4)	0.046 (3)	0.024 (3)	0.009 (2)	0.010 (3)
S1	0.119 (4)	0.097 (4)	0.086 (4)	0.038 (3)	0.063 (2)	0.035 (3)
C3	0.048 (3)	0.069 (4)	0.049 (3)	0.014 (3)	0.007 (2)	0.014 (3)
C4	0.146 (3)	0.074 (5)	0.057 (4)	0.034 (4)	0.014 (3)	0.014 (3)
C5	0.12 (5)	0.08 (3)	0.08 (4)	0.02 (3)	0.00 (3)	0.03 (2)
C6	0.11 (5)	0.10 (4)	0.10 (4)	0.02 (3)	0.04 (3)	0.05 (3)
S1'	0.146 (3)	0.074 (5)	0.057 (4)	0.034 (4)	0.014 (3)	0.014 (3)
C3'	0.048 (3)	0.069 (4)	0.049 (3)	0.014 (3)	0.007 (2)	0.014 (3)
C4'	0.119 (4)	0.097 (4)	0.086 (4)	0.038 (3)	0.063 (2)	0.035 (3)
C5'	0.10 (3)	0.11 (5)	0.09 (3)	0.03 (4)	0.04 (2)	0.05 (4)
C6'	0.12 (3)	0.08 (4)	0.08 (4)	0.01 (3)	0.01 (2)	0.03 (2)
C7	0.033 (2)	0.040 (3)	0.042 (3)	0.007 (2)	0.002 (2)	0.008 (2)
C8	0.031 (3)	0.071 (4)	0.055 (3)	0.010 (2)	0.002 (2)	0.009 (3)
S2	0.072 (2)	0.0661 (18)	0.0522 (13)	0.0244 (15)	0.0142 (15)	0.0144 (11)
C9	0.046 (3)	0.042 (3)	0.047 (3)	0.011 (2)	−0.006 (2)	0.000 (2)
C10	0.059 (12)	0.072 (5)	0.075 (12)	0.010 (7)	−0.025 (7)	0.015 (7)

C11	0.099 (18)	0.083 (13)	0.08 (2)	0.012 (11)	−0.033 (15)	0.027 (17)
C12	0.12 (2)	0.076 (18)	0.049 (13)	0.016 (15)	−0.006 (17)	0.017 (10)
S2'	0.059 (12)	0.072 (5)	0.075 (12)	0.010 (7)	−0.025 (7)	0.015 (7)
C9'	0.046 (3)	0.042 (3)	0.047 (3)	0.011 (2)	−0.006 (2)	0.000 (2)
C10'	0.072 (2)	0.0661 (18)	0.0522 (13)	0.0244 (15)	0.0142 (15)	0.0144 (11)
C11'	0.10 (6)	0.07 (6)	0.06 (7)	0.01 (4)	0.00 (5)	0.02 (4)
C12'	0.10 (7)	0.08 (4)	0.06 (6)	0.00 (4)	−0.03 (5)	0.02 (5)
C13	0.055 (3)	0.048 (3)	0.050 (3)	0.009 (3)	0.005 (2)	0.011 (2)
C14	0.058 (3)	0.043 (3)	0.063 (4)	0.009 (3)	0.000 (3)	0.012 (3)
C15	0.061 (4)	0.046 (3)	0.073 (4)	0.015 (3)	0.006 (3)	0.010 (3)
C16	0.065 (4)	0.063 (4)	0.066 (4)	0.022 (3)	0.015 (3)	0.027 (3)

Geometric parameters (Å, °)

Sn1—C14	2.093 (5)	C6'—H6'	0.9300
Sn1—C13	2.098 (5)	C7—C8	1.506 (7)
Sn1—O3	2.151 (3)	C8—C9	1.490 (7)
Sn1—Cl1	2.3766 (13)	C8—H8A	0.9700
Sn1—O1	2.479 (3)	C8—H8B	0.9700
Sn2—C15	2.091 (5)	S2—C9	1.690 (6)
Sn2—C16	2.109 (5)	S2—C12	1.71 (6)
Sn2—O2	2.155 (3)	C9—C10	1.36 (3)
Sn2—Cl2	2.3670 (14)	C10—C11	1.38 (5)
Sn2—O4^{i}	2.463 (3)	C10—H10	0.9300
O1—C1	1.241 (5)	C11—C12	1.37 (8)
O2—C1	1.280 (6)	C11—H11	0.9300
O3—C7	1.276 (5)	C12—H12	0.9300
O4—C7	1.236 (6)	S2'—C12'	1.70 (16)
O4—Sn2^{ii}	2.463 (3)	C10'—C11'	1.4 (2)
C1—C2	1.497 (7)	C10'—H10'	0.9300
C2—C3	1.500 (7)	C11'—C12'	1.3 (3)
C2—H2A	0.9700	C11'—H11'	0.9200
C2—H2B	0.9700	C12'—H12'	0.9500
S1—C3	1.678 (7)	C13—H13A	0.9600
S1—C6	1.7 (2)	C13—H13B	0.9600
C3—C4	1.36 (3)	C13—H13C	0.9600
C4—C5	1.37 (12)	C14—H14A	0.9600
C4—H4	0.9300	C14—H14B	0.9600
C5—C6	1.4 (2)	C14—H14C	0.9600
C5—H5	0.9300	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
S1'—C6'	1.69 (13)	C15—H15C	0.9600
C4'—C5'	1.36 (19)	C16—H16A	0.9600
C4'—H4'	0.9300	C16—H16B	0.9600
C5'—C6'	1.3 (2)	C16—H16C	0.9600
C5'—H5'	0.9100		
C14—Sn1—C13	140.1 (2)	S1'—C6'—H6'	126.9
C14—Sn1—O3	98.89 (18)	O4—C7—O3	120.8 (4)

C13—Sn1—O3	97.05 (18)	O4—C7—C8	122.5 (4)
C14—Sn1—Cl1	108.22 (17)	O3—C7—C8	116.6 (4)
C13—Sn1—Cl1	108.83 (15)	C9—C8—C7	115.2 (4)
O3—Sn1—Cl1	87.44 (9)	C9—C8—H8A	108.5
C14—Sn1—O1	84.14 (18)	C7—C8—H8A	108.5
C13—Sn1—O1	86.87 (17)	C9—C8—H8B	108.5
O3—Sn1—O1	169.29 (12)	C7—C8—H8B	108.5
Cl1—Sn1—O1	81.85 (9)	H8A—C8—H8B	107.5
C15—Sn2—C16	139.9 (3)	C9—S2—C12	92 (2)
C15—Sn2—O2	98.55 (19)	C10—C9—C8	124 (2)
C16—Sn2—O2	99.14 (19)	C10—C9—S2	111 (2)
C15—Sn2—Cl2	107.45 (17)	C8—C9—S2	124.6 (4)
C16—Sn2—Cl2	108.80 (19)	C9—C10—C11	113 (4)
O2—Sn2—Cl2	88.31 (9)	C9—C10—H10	123.4
C15—Sn2—O4^{i}	83.98 (19)	C11—C10—H10	123.4
C16—Sn2—O4^{i}	84.37 (19)	C12—C11—C10	113 (4)
O2—Sn2—O4^{i}	170.58 (12)	C12—C11—H11	123.6
Cl2—Sn2—O4^{i}	82.28 (9)	C10—C11—H11	123.6
C1—O1—Sn1	142.4 (3)	C11—C12—S2	110 (4)
C1—O2—Sn2	108.5 (3)	C11—C12—H12	124.8
C7—O3—Sn1	109.3 (3)	S2—C12—H12	124.8
C7—O4—Sn2^{ii}	143.5 (3)	C11'—C10'—H10'	122.6
O1—C1—O2	120.4 (4)	C12'—C11'—C10'	115 (10)
O1—C1—C2	122.3 (4)	C11'—C12'—S2'	105 (10)
O2—C1—C2	117.3 (4)	Sn1—C13—H13A	109.5
C1—C2—C3	111.6 (4)	Sn1—C13—H13B	109.5
C1—C2—H2A	109.3	H13A—C13—H13B	109.5
C3—C2—H2A	109.3	Sn1—C13—H13C	109.5
C1—C2—H2B	109.3	H13A—C13—H13C	109.5
C3—C2—H2B	109.3	H13B—C13—H13C	109.5
H2A—C2—H2B	108.0	Sn1—C14—H14A	109.5
C3—S1—C6	93 (6)	Sn1—C14—H14B	109.5
C4—C3—C2	130.6 (19)	H14A—C14—H14B	109.5
C4—C3—S1	110.9 (19)	Sn1—C14—H14C	109.5
C2—C3—S1	118.4 (5)	H14A—C14—H14C	109.5
C3—C4—C5	114 (7)	H14B—C14—H14C	109.5
C3—C4—H4	123.1	Sn2—C15—H15A	109.5
C5—C4—H4	123.1	Sn2—C15—H15B	109.5
C6—C5—C4	113 (10)	H15A—C15—H15B	109.5
C6—C5—H5	123.5	Sn2—C15—H15C	109.5
C4—C5—H5	123.5	H15A—C15—H15C	109.5
C5—C6—S1	110 (10)	H15B—C15—H15C	109.5
S1—C6—H6	127.0	Sn2—C16—H16A	109.5
C5'—C4'—H4'	121.9	Sn2—C16—H16B	109.5
C6'—C5'—C4'	116 (10)	H16A—C16—H16B	109.5
C6'—C5'—H5'	122.2	Sn2—C16—H16C	109.5
C4'—C5'—H5'	124.00	H16A—C16—H16C	109.5
C5'—C6'—S1'	106 (10)	H16B—C16—H16C	109.5

C5'—C6'—H6'	125.8

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*−1, *y*, *z*.

Fig. 1

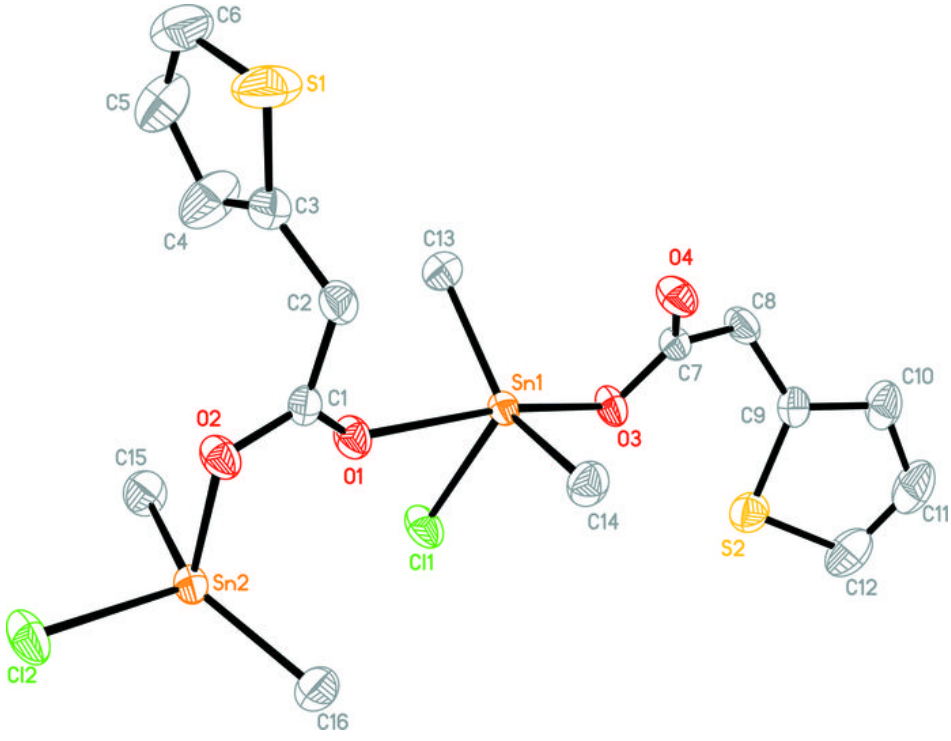

Fig. 2