

Crystal structure of the 1:1 adduct of 2,3-diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one with triphenyltin chloride

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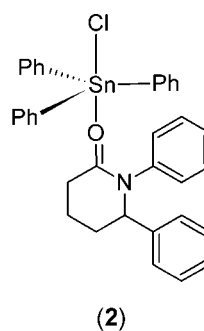
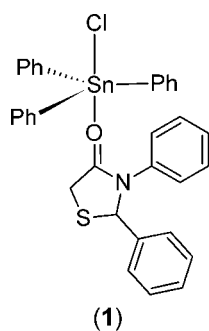
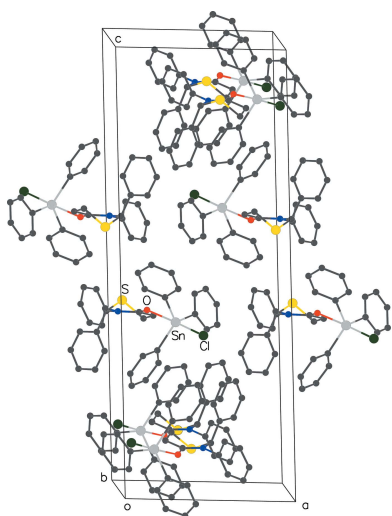
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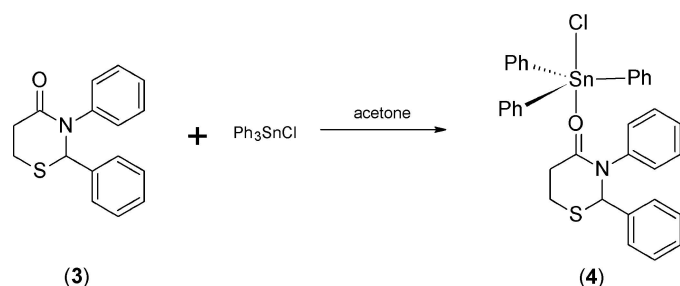
The title adduct, chlorido(2,3-diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one- κ O)triphenyltin, [Sn(C₆H₅)₃Cl(C₁₆H₁₅NOS)], resulted from reaction of 2,3-diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one with triphenyltin chloride. The thiazine ring has an envelope conformation with the S atom forming the flap. The molecule has five phenyl rings, two of them attached to the thiazine ring at positions 2 and 3, and three in coordination with the Sn^{IV} atom. The three rings of the triphenyltin group are involved in intramolecular interactions of different types, C–H···O, edge-to-face (or T-type) π – π interactions with the 3-phenyl ring of the thiazine, T-type interactions with both phenyl rings of the thiazine *etc.* On the other hand, all the phenyl rings participate in intermolecular π – π interactions. There is one instance of a ‘parallel-displaced’-type interaction extending continuously along the *a*-axis direction and seven instances of T-type interactions stabilizing the crystal lattice.

1. Chemical context

Eng and coworkers have reported the synthesis and fungicidal activity of 1:1 complexes of triphenyltin chloride complexes with five-membered 1,3-thiazolidin-4-ones (Smith *et al.*, 1995; Eng *et al.*, 1996, 1998), including a crystal structure of 2,3-diphenyl-1,3-thiazolidin-4-one (**1**) (Scheme 1) (Smith *et al.*, 1995). Tahara *et al.* have reported the preparation of similar 1:1 adducts of triphenyltin chloride with lactams, including the six-membered valerolactam (**2**) (Scheme 1) (Tahara *et al.*, 1987). They did not report a crystal structure of (**2**), but did report a crystal structure of the adduct of the seven-membered caprolactam. All of the complexes reported by Tahara and Eng bind through the carbonyl oxygen atom to the central tin atom and adopt a distorted trigonal–bipyramidal geometry around the tin atom, with the heterocycle and chlorine in axial positions.



We have recently reported a variety of six- and seven-membered 2,3-diaryl-1,3-thiazia-4-one heterocycles, including 2,3-diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one (**3**) (Yennawar & Silverberg, 2014; Silverberg, *et al.*, 2015). Herein, we report the synthesis and crystal structure of the 1:1 adduct (**4**) resulting from reaction of (**3**) with triphenyltin chloride (Scheme 2), which to the best of our knowledge is the first preparation of a tin complex of any 2,3-disubstituted-1,3-thiazin-4-one heterocycle [Eng *et al.* (1996) reported the adduct of 3-phenyl-1,3-thiazinane-2,4-dione]. Crystals for X-ray crystallographic analysis were grown by slow evaporation of the adduct solution in cyclohexane.



2. Structural commentary

The molecular structure obtained (Fig. 1) is similar to that reported for (**1**) (Smith *et al.*, 1995). It is a 1:1 complex, with

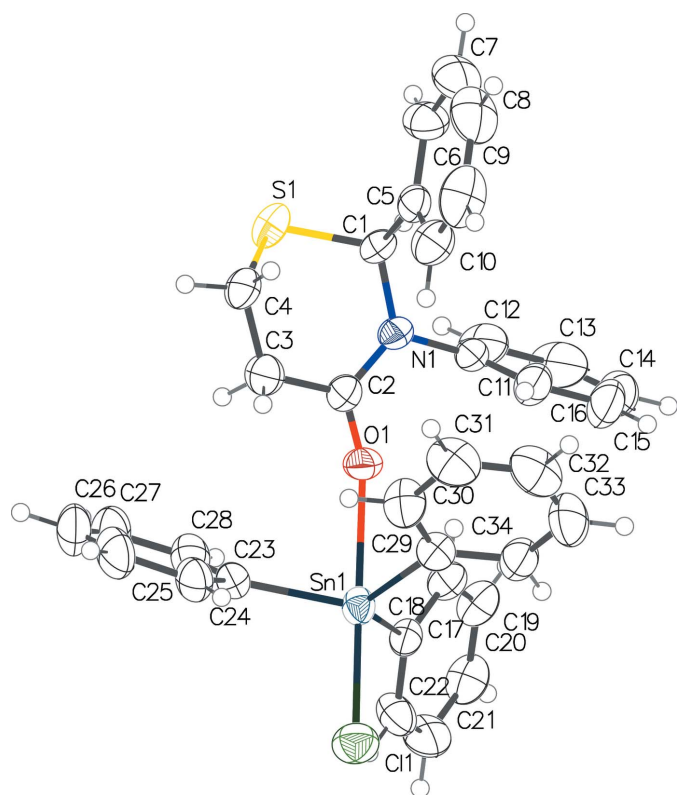


Figure 1
Ellipsoid plot (50% probability level for non-H atoms) of the title compound (**4**).

Table 1
Selected geometric parameters (Å, °).

C17—Sn1	2.140 (3)	Cl1—Sn1	2.4558 (10)
C23—Sn1	2.123 (3)	O1—Sn1	2.512 (2)
C29—Sn1	2.134 (4)		
C23—Sn1—C17	117.50 (12)	C29—Sn1—C17	119.48 (12)
C23—Sn1—C29	117.48 (12)	Cl1—Sn1—O1	178.00 (6)

Table 2
Intra- and intermolecular π - π interactions (Å, °).

*Cg*2, *Cg*3, *Cg*4, *Cg*5 and *Cg*6 are the centroids of the C5–C10, C11–C16, C17–C22, C23–C28, and C29–C34 rings, respectively.

<i>Cg</i> I... <i>Cg</i> J	<i>Cg</i> ... <i>Cg</i>	Dihedral angle	Comment
<i>Cg</i> 3... <i>Cg</i> 4	5.1455 (9)	85	Intra – T-type
<i>Cg</i> 6... <i>Cg</i> 2	5.9538 (10)	83	Intra – T-type
<i>Cg</i> 6... <i>Cg</i> 3	5.1126 (9)	50	Intra – T-type
<i>Cg</i> 2... <i>Cg</i> 5 ⁱ	5.3346 (9)	84	Inter – T-type
<i>Cg</i> 2... <i>Cg</i> 2 ⁱⁱ	5.8549 (10)	89	Inter – T-type
<i>Cg</i> 2... <i>Cg</i> 6 ⁱⁱⁱ	5.5685 (10)	83	Inter – T-type
<i>Cg</i> 3... <i>Cg</i> 5 ⁱ	3.8627 (7)	2	Inter – parallel-displaced
<i>Cg</i> 3... <i>Cg</i> 4 ^{iv}	5.7753 (10)	85	Inter – T-type
<i>Cg</i> 4... <i>Cg</i> 5 ^v	5.1688 (9)	86	Inter – T-type
<i>Cg</i> 5... <i>Cg</i> 6 ^{vi}	5.8599 (10)	89	Inter – T-type
<i>Cg</i> 6... <i>Cg</i> 6 ^{vii}	5.5050 (10)	69	Inter – T-type

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

the carbonyl oxygen in (**3**) bound to the tin atom. The tin atom is pentacoordinate with a distorted trigonal-bipyramidal geometry (Table 1), the apical axis being the O–Sn–Cl line. Chlorine and (**3**) are in the axial positions and the three phenyl groups are equatorial. The C–Sn, Cl–Sn, and C–O bond lengths are similar to those in (**1**).

The current crystal structure (**4**) exhibits an envelope conformation for the thiazine ring with the sulfur atom forming the flap, similar to (**3**) (Yennawar & Silverberg, 2014, 2015). The structure has a C–H...O type interaction between the only oxygen atom (O1) and a phenyl carbon C18 of the same molecule. Extensive intra- and intermolecular ring interactions influence the structure of the molecule as well as the crystal packing. Both parallel-displaced and T-shaped interactions, analyzed using *PLATON* (Spek, 2009) have been observed and are discussed below in Section 3.

3. Supramolecular Features

The adduct has a thiazine ring (ring-1) and five phenyl rings (rings-2 and ring-3 attached at positions 2 and 3 of the thiazine and rings 4, 5 and 6 of the triphenyltin moiety). The intramolecular interactions between all six rings influence orientation of the phenyl rings and the intermolecular interactions of the five phenyl rings stabilize the crystal lattice (Fig. 2).

Intramolecular interactions – Carbon C18 of ring-4 has a C–H...O type interaction with the only oxygen O1 in the molecule [$\text{C18}\cdots\text{O1} = 3.017$ (4) Å; $\text{C18}-\text{H18}\cdots\text{O1} = 124^\circ$]. The same carbon C18 is at a distance of 3.8287 (7) Å from the

centroid of ring-3, resulting in a T-type π - π ring-4 \cdots ring-3 interaction. Ring-6 has T-type interactions with both (ring-2 and ring-3) phenyl rings of the thiazine with inter-centroid distances of 5.112 (1) with ring-3 and 5.954 (1) Å with ring-2. The C3 atom of the thiazine ring is 3.5235 (6) Å from the centroid of ring-5, resulting in a C-H $\cdots\pi$ interaction. Thus all six rings, aromatic and non-aromatic, participate in influencing the structure of the molecule.

Intermolecular interactions – The five phenyl rings interact extensively with the phenyl rings of the neighboring molecules in the lattice. Of the eight such π - π interactions, one belongs to the parallel-displaced type and seven are of the T-type. In the parallel-displaced interaction, ring-3 and ring-5 of a molecule interact respectively with ring-5 and ring-3 of molecules on opposite sides, forming a continuous chain along the *a*-axis direction. The distance between the centroids of these partially overlapping rings is 3.8627 (7) Å and the dihedral angle is 2° between the ring planes. Seven T-type interactions stabilize the lattice further with centroid distances ranging from 5.1688 (9) to 5.8599 (10) Å and the dihedral angles of 69° to 89°. Rings 2, 5 and 6 participate in three interactions each, ring-4 in two and ring-3 in one. The intra- and intermolecular π - π interactions are listed in Table 2.

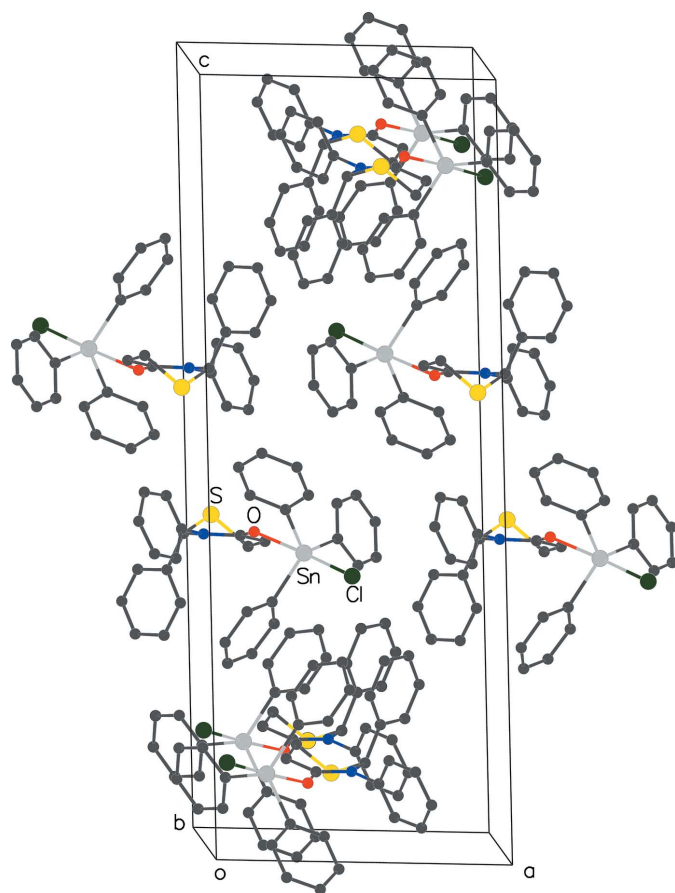


Figure 2
The packing of the title compound (4).

Table 3
Experimental details.

Crystal data	
Chemical formula	[Sn(C ₆ H ₅) ₃ Cl(C ₁₆ H ₁₅ NOS)]
<i>M_r</i>	654.79
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>n</i>
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.8454 (19), 9.5675 (16), 28.891 (5)
β (°)	92.886 (3)
<i>V</i> (Å ³)	2994.0 (9)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.04
Crystal size (mm)	0.21 × 0.18 × 0.17
Data collection	
Diffractometer	Bruker SMART APEX CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2001)
<i>T</i> _{min} , <i>T</i> _{max}	0.820, 1.0
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	27794, 7403, 6561
<i>R</i> _{int}	0.024
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.667
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.048, 0.144, 1.08
No. of reflections	7403
No. of parameters	352
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	1.14, -1.07

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

4. Database Survey

The crystal structure of triphenyltin chloride has also been reported (Tse *et al.*, 1986; Bokii *et al.*, 1970).

5. Synthesis and crystallization

Adduct (4) was prepared by reacting an equivalent each of triphenyltin chloride (Ph₃SnCl) and 2,3-diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one (3) (Yennawar & Silverberg, 2014) in acetone (Scheme 2) (Smith *et al.*, 1995; Cannon, 2015). The solvent was removed and the solid was recrystallized from ligroin.

General: Triphenyltin chloride was purchased from Sigma-Aldrich (St. Louis, MO). Ligroin (363–383 K b.p. range) was purchased from Fisher Chemical (Pittsburgh, PA). Low-water acetone was purchased from J. T. Baker (Center Valley, PA). Melting points were determined with a Thomas Hoover Capillary Melting Point Apparatus (Arthur H. Thomas Co., Philadelphia, PA).

1:1 Adduct (4) of 2,3-Diphenyl-3,4,5,6-tetrahydro-2H-1,3-thiazin-4-one (3) with triphenyltin chloride: A two-neck 10 mL round-bottom flask and a 5 mL round-bottom flask with stir bars were oven-dried, fitted with septa, and cooled under N₂. Triphenyltin chloride (0.1427 g, 0.37 mmol) was added to the 10 mL flask. 2,3-Diphenyl-3,4,5,6-tetrahydro-2*H*-1,3-thiazin-4-one 3 (0.100 g, 0.37 mmol) was added to the 5 mL flask. 2.5 mL of low-water acetone was added to each flask and each solu-

tion was stirred. The contents of the 5 mL flask were transferred to the 10 mL flask dropwise by syringe over a period of 30 minutes. After two h of stirring, the stirrer was turned off. The solution was slightly hazy. After four days, the solution was transferred to a 50 mL round-bottom flask with acetone and concentrated under vacuum to a white solid. Recrystallization from ligroin produced (**4**) as a white powder (0.1086 g, 45%), m.p. 405–407 K. Crystals for X-ray crystallography were grown by slow evaporation from cyclohexane.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms were placed geometrically to ride on the carbon atoms during refinement with C–H distances of 0.97 Å (>CH₂) and 0.93 Å (–CH_{arom}) and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Acknowledgements

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supporting information

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Crystal structure of the 1:1 adduct of 2,3-diphenyl-3,4,5,6-tetrahydro-2H-1,3-thiazin-4-one with triphenyltin chloride

Hemant P. Yennawar, Ryan Fox and Lee J. Silverberg

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART* (Bruker, 2001); data reduction: *S SAINT* (Bruker, 2001); program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Chlorido(2,3-diphenyl-3,4,5,6-tetrahydro-2H-1,3-thiazin-4-one- κ O)triphenyltin

Crystal data

[Sn(C₆H₅)₃Cl(C₁₆H₁₅NOS)]

$M_r = 654.79$

Monoclinic, $P2_1/n$

$a = 10.8454$ (19) Å

$b = 9.5675$ (16) Å

$c = 28.891$ (5) Å

$\beta = 92.886$ (3)°

$V = 2994.0$ (9) Å³

$Z = 4$

$F(000) = 1328$

$D_x = 1.453$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5487 reflections

$\theta = 2.2$ – 28.2 °

$\mu = 1.04$ mm⁻¹

$T = 298$ K

Block, clear colourless

$0.21 \times 0.18 \times 0.17$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Parallel, graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.820$, $T_{\max} = 1.0$

27794 measured reflections

7403 independent reflections

6561 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.0$ °

$h = -14 \rightarrow 14$

$k = -11 \rightarrow 12$

$l = -38 \rightarrow 38$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.144$

$S = 1.08$

7403 reflections

352 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.14$ e Å⁻³

$\Delta\rho_{\min} = -1.07$ e Å⁻³

Special details

Experimental. The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (5 s exposure) covering -0.300° degrees in ω . The crystal to detector distance was 5.82 cm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. None

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.0293 (3)	0.7598 (3)	0.38394 (10)	0.0346 (6)
H1	-0.0976	0.7618	0.4049	0.042*
C2	0.1500 (3)	0.5935 (3)	0.38953 (11)	0.0405 (7)
C3	0.2413 (3)	0.7053 (4)	0.37798 (19)	0.0634 (12)
H3A	0.2994	0.7149	0.4044	0.076*
H3B	0.2874	0.6714	0.3524	0.076*
C4	0.1947 (3)	0.8490 (4)	0.36539 (14)	0.0487 (8)
H4A	0.1623	0.8498	0.3335	0.058*
H4B	0.2621	0.9156	0.3683	0.058*
C5	-0.0856 (3)	0.7891 (3)	0.33611 (12)	0.0390 (7)
C6	-0.1767 (3)	0.8910 (4)	0.33192 (15)	0.0560 (9)
H6	-0.2019	0.9362	0.3583	0.067*
C7	-0.2300 (4)	0.9260 (5)	0.28952 (18)	0.0762 (13)
H7	-0.2923	0.9929	0.2874	0.091*
C8	-0.1919 (5)	0.8631 (6)	0.25064 (19)	0.0848 (16)
H8	-0.2270	0.8884	0.2218	0.102*
C9	-0.1022 (5)	0.7628 (6)	0.25366 (15)	0.0750 (13)
H9	-0.0768	0.7202	0.2268	0.090*
C10	-0.0479 (4)	0.7232 (4)	0.29686 (14)	0.0543 (9)
H10	0.0123	0.6539	0.2989	0.065*
C11	-0.0549 (3)	0.5089 (3)	0.39995 (13)	0.0423 (7)
C12	-0.1050 (4)	0.5019 (4)	0.44224 (15)	0.0628 (10)
H12	-0.0850	0.5679	0.4650	0.075*
C13	-0.1872 (4)	0.3932 (6)	0.4506 (2)	0.095 (2)
H13	-0.2227	0.3869	0.4791	0.114*
C14	-0.2153 (5)	0.2971 (6)	0.4172 (3)	0.096 (2)
H14	-0.2706	0.2257	0.4230	0.115*
C15	-0.1640 (5)	0.3038 (5)	0.3759 (2)	0.087 (2)
H15	-0.1831	0.2360	0.3536	0.105*
C16	-0.0835 (4)	0.4096 (4)	0.36625 (16)	0.0620 (10)
H16	-0.0487	0.4144	0.3375	0.074*
C17	0.2724 (3)	0.1831 (3)	0.44010 (10)	0.0366 (6)
C18	0.1561 (3)	0.2141 (3)	0.45627 (12)	0.0430 (7)
H18	0.1068	0.2818	0.4415	0.052*
C19	0.1141 (4)	0.1431 (4)	0.49471 (12)	0.0505 (8)

H19	0.0362	0.1626	0.5051	0.061*
C20	0.1861 (4)	0.0461 (4)	0.51693 (12)	0.0556 (9)
H20	0.1580	0.0006	0.5428	0.067*
C21	0.3013 (4)	0.0143 (5)	0.50130 (14)	0.0627 (10)
H21	0.3502	-0.0532	0.5164	0.075*
C22	0.3434 (3)	0.0834 (4)	0.46308 (12)	0.0521 (9)
H22	0.4210	0.0621	0.4528	0.062*
C23	0.4673 (3)	0.4534 (3)	0.39319 (12)	0.0410 (7)
C24	0.5381 (3)	0.5026 (4)	0.35781 (14)	0.0521 (8)
H24	0.5375	0.4546	0.3298	0.063*
C25	0.6092 (4)	0.6217 (5)	0.36365 (17)	0.0679 (12)
H25	0.6536	0.6560	0.3394	0.082*
C26	0.6136 (4)	0.6895 (5)	0.4061 (2)	0.0752 (14)
H26	0.6620	0.7691	0.4104	0.090*
C27	0.5473 (4)	0.6402 (5)	0.44153 (17)	0.0718 (12)
H27	0.5520	0.6856	0.4700	0.086*
C28	0.4730 (3)	0.5232 (4)	0.43537 (13)	0.0540 (9)
H28	0.4269	0.4913	0.4595	0.065*
C29	0.2290 (3)	0.2921 (3)	0.31778 (12)	0.0425 (7)
C30	0.2426 (4)	0.3976 (4)	0.28580 (12)	0.0564 (9)
H30	0.3056	0.4626	0.2909	0.068*
C31	0.1660 (4)	0.4095 (5)	0.24657 (14)	0.0675 (11)
H31	0.1755	0.4835	0.2262	0.081*
C32	0.0757 (4)	0.3115 (6)	0.23774 (15)	0.0703 (12)
H32	0.0247	0.3175	0.2110	0.084*
C33	0.0607 (4)	0.2040 (5)	0.26873 (19)	0.0747 (14)
H33	-0.0004	0.1373	0.2628	0.090*
C34	0.1362 (4)	0.1950 (4)	0.30866 (15)	0.0548 (9)
H34	0.1246	0.1230	0.3296	0.066*
Cl1	0.48733 (9)	0.10626 (10)	0.36005 (4)	0.0572 (2)
N1	0.0277 (2)	0.6213 (3)	0.38945 (9)	0.0364 (5)
O1	0.18753 (19)	0.4749 (2)	0.39935 (9)	0.0467 (5)
S1	0.07553 (9)	0.89817 (9)	0.40313 (3)	0.0494 (2)
Sn1	0.33979 (2)	0.28758 (2)	0.38094 (2)	0.03769 (10)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0426 (15)	0.0265 (13)	0.0354 (14)	-0.0004 (11)	0.0088 (12)	0.0011 (12)
C2	0.0404 (15)	0.0348 (16)	0.0460 (17)	-0.0032 (12)	0.0003 (13)	0.0035 (13)
C3	0.0407 (18)	0.048 (2)	0.102 (3)	-0.0064 (14)	0.010 (2)	0.025 (2)
C4	0.0472 (17)	0.0364 (17)	0.063 (2)	-0.0092 (14)	0.0081 (16)	0.0078 (16)
C5	0.0382 (15)	0.0347 (17)	0.0442 (17)	-0.0067 (11)	0.0012 (13)	0.0046 (12)
C6	0.0513 (19)	0.050 (2)	0.067 (2)	0.0065 (15)	0.0062 (17)	0.0169 (18)
C7	0.061 (2)	0.074 (3)	0.092 (4)	0.000 (2)	-0.016 (2)	0.035 (3)
C8	0.086 (3)	0.094 (4)	0.070 (3)	-0.020 (3)	-0.034 (3)	0.030 (3)
C9	0.100 (4)	0.082 (3)	0.043 (2)	-0.026 (3)	0.000 (2)	-0.004 (2)
C10	0.064 (2)	0.050 (2)	0.049 (2)	-0.0045 (16)	0.0046 (18)	-0.0028 (16)

C11	0.0329 (13)	0.0317 (15)	0.062 (2)	-0.0016 (11)	-0.0009 (13)	0.0102 (14)
C12	0.064 (2)	0.054 (2)	0.073 (3)	0.0030 (18)	0.023 (2)	0.021 (2)
C13	0.068 (3)	0.077 (4)	0.145 (5)	0.010 (3)	0.046 (3)	0.056 (4)
C14	0.056 (3)	0.058 (3)	0.171 (7)	-0.019 (2)	-0.021 (3)	0.057 (4)
C15	0.087 (4)	0.044 (3)	0.126 (6)	-0.023 (2)	-0.044 (4)	0.020 (3)
C16	0.065 (2)	0.0397 (19)	0.079 (3)	-0.0082 (17)	-0.021 (2)	0.0052 (19)
C17	0.0402 (14)	0.0336 (14)	0.0356 (14)	-0.0059 (12)	-0.0020 (12)	0.0004 (12)
C18	0.0505 (18)	0.0370 (18)	0.0420 (17)	-0.0018 (12)	0.0069 (14)	0.0021 (13)
C19	0.064 (2)	0.0436 (19)	0.0446 (17)	-0.0069 (16)	0.0139 (16)	-0.0022 (15)
C20	0.074 (2)	0.054 (2)	0.0391 (17)	-0.0142 (18)	0.0062 (16)	0.0088 (16)
C21	0.061 (2)	0.064 (3)	0.062 (2)	0.0017 (18)	-0.0089 (18)	0.030 (2)
C22	0.0382 (15)	0.063 (2)	0.054 (2)	-0.0041 (15)	-0.0047 (14)	0.0191 (17)
C23	0.0327 (14)	0.0399 (17)	0.0499 (17)	-0.0021 (12)	-0.0033 (12)	0.0022 (14)
C24	0.0392 (16)	0.055 (2)	0.063 (2)	-0.0088 (15)	0.0067 (15)	0.0061 (17)
C25	0.047 (2)	0.074 (3)	0.083 (3)	-0.0201 (19)	0.000 (2)	0.026 (2)
C26	0.057 (2)	0.061 (3)	0.105 (4)	-0.025 (2)	-0.019 (3)	0.008 (3)
C27	0.073 (3)	0.067 (3)	0.073 (3)	-0.013 (2)	-0.019 (2)	-0.012 (2)
C28	0.0516 (19)	0.059 (2)	0.0501 (19)	-0.0090 (16)	-0.0078 (15)	0.0051 (17)
C29	0.0433 (16)	0.0439 (19)	0.0405 (16)	0.0002 (12)	0.0022 (14)	0.0007 (13)
C30	0.062 (2)	0.062 (2)	0.0444 (18)	-0.0131 (17)	0.0004 (16)	0.0128 (17)
C31	0.072 (3)	0.083 (3)	0.048 (2)	0.005 (2)	0.0019 (19)	0.019 (2)
C32	0.070 (3)	0.097 (4)	0.042 (2)	0.011 (2)	-0.0127 (19)	-0.006 (2)
C33	0.062 (3)	0.079 (3)	0.081 (3)	-0.011 (2)	-0.018 (2)	-0.019 (2)
C34	0.059 (2)	0.043 (2)	0.061 (2)	-0.0078 (15)	-0.0093 (18)	0.0049 (16)
Cl1	0.0561 (5)	0.0514 (5)	0.0649 (6)	0.0070 (4)	0.0117 (4)	-0.0012 (4)
N1	0.0383 (12)	0.0287 (13)	0.0422 (13)	0.0004 (10)	0.0021 (10)	0.0055 (10)
O1	0.0378 (11)	0.0341 (12)	0.0684 (15)	0.0050 (9)	0.0044 (10)	0.0107 (11)
S1	0.0655 (5)	0.0362 (4)	0.0466 (4)	-0.0109 (4)	0.0033 (4)	-0.0088 (3)
Sn1	0.03675 (14)	0.03780 (16)	0.03835 (15)	-0.00725 (7)	0.00030 (9)	0.00637 (8)

Geometric parameters (Å, °)

C1—H1	0.9800	C17—C22	1.375 (5)
C1—C5	1.509 (4)	C17—Sn1	2.140 (3)
C1—N1	1.467 (4)	C18—H18	0.9300
C1—S1	1.814 (3)	C18—C19	1.398 (5)
C2—C3	1.507 (4)	C19—H19	0.9300
C2—N1	1.352 (4)	C19—C20	1.354 (5)
C2—O1	1.234 (4)	C20—H20	0.9300
C3—H3A	0.9700	C20—C21	1.383 (6)
C3—H3B	0.9700	C21—H21	0.9300
C3—C4	1.503 (5)	C21—C22	1.384 (5)
C4—H4A	0.9700	C22—H22	0.9300
C4—H4B	0.9700	C23—C24	1.391 (5)
C4—S1	1.795 (4)	C23—C28	1.388 (5)
C5—C6	1.389 (5)	C23—Sn1	2.123 (3)
C5—C10	1.378 (5)	C24—H24	0.9300
C6—H6	0.9300	C24—C25	1.382 (5)

C6—C7	1.370 (6)	C25—H25	0.9300
C7—H7	0.9300	C25—C26	1.385 (7)
C7—C8	1.357 (8)	C26—H26	0.9300
C8—H8	0.9300	C26—C27	1.365 (7)
C8—C9	1.367 (8)	C27—H27	0.9300
C9—H9	0.9300	C27—C28	1.386 (6)
C9—C10	1.405 (6)	C28—H28	0.9300
C10—H10	0.9300	C29—C30	1.381 (5)
C11—C12	1.364 (5)	C29—C34	1.384 (5)
C11—C16	1.384 (5)	C29—Sn1	2.134 (4)
C11—N1	1.442 (4)	C30—H30	0.9300
C12—H12	0.9300	C30—C31	1.376 (5)
C12—C13	1.398 (6)	C31—H31	0.9300
C13—H13	0.9300	C31—C32	1.370 (6)
C13—C14	1.355 (9)	C32—H32	0.9300
C14—H14	0.9300	C32—C33	1.378 (7)
C14—C15	1.344 (9)	C33—H33	0.9300
C15—H15	0.9300	C33—C34	1.383 (6)
C15—C16	1.375 (6)	C34—H34	0.9300
C16—H16	0.9300	Cl1—Sn1	2.4558 (10)
C17—C18	1.399 (4)	O1—Sn1	2.512 (2)
C5—C1—H1	106.2	C18—C19—H19	119.8
C5—C1—S1	111.3 (2)	C20—C19—C18	120.5 (3)
N1—C1—H1	106.2	C20—C19—H19	119.8
N1—C1—C5	114.6 (2)	C19—C20—H20	119.9
N1—C1—S1	111.8 (2)	C19—C20—C21	120.3 (3)
S1—C1—H1	106.2	C21—C20—H20	119.9
N1—C2—C3	121.0 (3)	C20—C21—H21	120.2
O1—C2—C3	119.4 (3)	C20—C21—C22	119.7 (3)
O1—C2—N1	119.6 (3)	C22—C21—H21	120.2
C2—C3—H3A	107.5	C17—C22—C21	121.1 (3)
C2—C3—H3B	107.5	C17—C22—H22	119.4
H3A—C3—H3B	107.0	C21—C22—H22	119.4
C4—C3—C2	119.1 (3)	C24—C23—Sn1	120.6 (3)
C4—C3—H3A	107.5	C28—C23—C24	118.8 (3)
C4—C3—H3B	107.5	C28—C23—Sn1	120.3 (2)
C3—C4—H4A	109.7	C23—C24—H24	119.5
C3—C4—H4B	109.7	C25—C24—C23	121.0 (4)
C3—C4—S1	109.6 (3)	C25—C24—H24	119.5
H4A—C4—H4B	108.2	C24—C25—H25	120.4
S1—C4—H4A	109.7	C24—C25—C26	119.2 (4)
S1—C4—H4B	109.7	C26—C25—H25	120.4
C6—C5—C1	117.6 (3)	C25—C26—H26	119.8
C10—C5—C1	123.0 (3)	C27—C26—C25	120.4 (4)
C10—C5—C6	119.3 (4)	C27—C26—H26	119.8
C5—C6—H6	119.5	C26—C27—H27	119.7
C7—C6—C5	121.0 (4)	C26—C27—C28	120.5 (4)

C7—C6—H6	119.5	C28—C27—H27	119.7
C6—C7—H7	120.0	C23—C28—H28	120.0
C8—C7—C6	120.0 (4)	C27—C28—C23	120.1 (4)
C8—C7—H7	120.0	C27—C28—H28	120.0
C7—C8—H8	119.9	C30—C29—C34	117.6 (3)
C7—C8—C9	120.3 (4)	C30—C29—Sn1	120.8 (3)
C9—C8—H8	119.9	C34—C29—Sn1	121.4 (3)
C8—C9—H9	119.6	C29—C30—H30	119.0
C8—C9—C10	120.8 (5)	C31—C30—C29	122.1 (4)
C10—C9—H9	119.6	C31—C30—H30	119.0
C5—C10—C9	118.6 (4)	C30—C31—H31	120.2
C5—C10—H10	120.7	C32—C31—C30	119.6 (4)
C9—C10—H10	120.7	C32—C31—H31	120.2
C12—C11—C16	120.7 (3)	C31—C32—H32	120.1
C12—C11—N1	120.4 (3)	C31—C32—C33	119.7 (4)
C16—C11—N1	118.9 (3)	C33—C32—H32	120.1
C11—C12—H12	120.7	C32—C33—H33	119.9
C11—C12—C13	118.5 (5)	C32—C33—C34	120.2 (4)
C13—C12—H12	120.7	C34—C33—H33	119.9
C12—C13—H13	119.8	C29—C34—H34	119.6
C14—C13—C12	120.3 (5)	C33—C34—C29	120.7 (4)
C14—C13—H13	119.8	C33—C34—H34	119.6
C13—C14—H14	119.7	C2—N1—C1	125.9 (2)
C15—C14—C13	120.7 (4)	C2—N1—C11	118.2 (2)
C15—C14—H14	119.7	C11—N1—C1	115.7 (2)
C14—C15—H15	119.6	C2—O1—Sn1	145.1 (2)
C14—C15—C16	120.7 (5)	C4—S1—C1	94.74 (15)
C16—C15—H15	119.6	C17—Sn1—Cl1	96.85 (9)
C11—C16—H16	120.5	C17—Sn1—O1	84.81 (10)
C15—C16—C11	119.0 (5)	C23—Sn1—C17	117.50 (12)
C15—C16—H16	120.5	C23—Sn1—C29	117.48 (12)
C18—C17—Sn1	121.2 (2)	C23—Sn1—Cl1	98.15 (9)
C22—C17—C18	118.6 (3)	C23—Sn1—O1	82.03 (10)
C22—C17—Sn1	120.3 (2)	C29—Sn1—C17	119.48 (12)
C17—C18—H18	120.1	C29—Sn1—Cl1	98.63 (9)
C19—C18—C17	119.8 (3)	C29—Sn1—O1	79.56 (10)
C19—C18—H18	120.1	Cl1—Sn1—O1	178.00 (6)
C1—C5—C6—C7	178.3 (3)	C23—C24—C25—C26	2.6 (6)
C1—C5—C10—C9	-177.0 (3)	C24—C23—C28—C27	0.5 (5)
C2—C3—C4—S1	41.8 (5)	C24—C25—C26—C27	-0.8 (7)
C3—C2—N1—C1	-7.4 (5)	C25—C26—C27—C28	-1.1 (7)
C3—C2—N1—C11	178.8 (4)	C26—C27—C28—C23	1.2 (6)
C3—C2—O1—Sn1	-38.6 (6)	C28—C23—C24—C25	-2.5 (5)
C3—C4—S1—C1	-63.2 (3)	C29—C30—C31—C32	-2.3 (7)
C5—C1—N1—C2	101.4 (3)	C30—C29—C34—C33	0.3 (6)
C5—C1—N1—Cl1	-84.7 (3)	C30—C31—C32—C33	1.5 (7)
C5—C1—S1—C4	-73.3 (2)	C31—C32—C33—C34	0.2 (7)

C5—C6—C7—C8	-1.6 (7)	C32—C33—C34—C29	-1.0 (7)
C6—C5—C10—C9	0.7 (5)	C34—C29—C30—C31	1.4 (6)
C6—C7—C8—C9	1.3 (8)	N1—C1—C5—C6	157.5 (3)
C7—C8—C9—C10	-0.1 (8)	N1—C1—C5—C10	-24.8 (4)
C8—C9—C10—C5	-0.9 (7)	N1—C1—S1—C4	56.3 (2)
C10—C5—C6—C7	0.5 (5)	N1—C2—C3—C4	-1.1 (6)
C11—C12—C13—C14	0.4 (7)	N1—C2—O1—Sn1	141.6 (3)
C12—C11—C16—C15	0.4 (5)	N1—C11—C12—C13	178.4 (3)
C12—C11—N1—C1	-70.3 (4)	N1—C11—C16—C15	-178.8 (3)
C12—C11—N1—C2	104.1 (4)	O1—C2—C3—C4	179.1 (4)
C12—C13—C14—C15	0.6 (8)	O1—C2—N1—C1	172.4 (3)
C13—C14—C15—C16	-1.1 (8)	O1—C2—N1—C11	-1.4 (5)
C14—C15—C16—C11	0.6 (7)	S1—C1—C5—C6	-74.4 (3)
C16—C11—C12—C13	-0.9 (6)	S1—C1—C5—C10	103.3 (3)
C16—C11—N1—C1	108.9 (3)	S1—C1—N1—C2	-26.4 (4)
C16—C11—N1—C2	-76.7 (4)	S1—C1—N1—C11	147.5 (2)
C17—C18—C19—C20	1.1 (5)	Sn1—C17—C18—C19	179.3 (3)
C18—C17—C22—C21	0.2 (6)	Sn1—C17—C22—C21	-179.7 (3)
C18—C19—C20—C21	-1.2 (6)	Sn1—C23—C24—C25	171.5 (3)
C19—C20—C21—C22	0.8 (6)	Sn1—C23—C28—C27	-173.4 (3)
C20—C21—C22—C17	-0.3 (6)	Sn1—C29—C30—C31	-174.7 (3)
C22—C17—C18—C19	-0.6 (5)	Sn1—C29—C34—C33	176.4 (3)
