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(*Z*)-Methyl 2-(1,3-dithian-2-ylidene)-4-(triphenylstannyl)but-3-enoate

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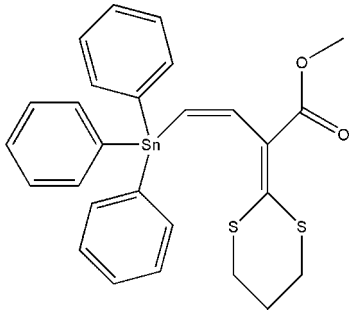
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.030; wR factor = 0.075; data-to-parameter ratio = 15.6.

In the title compound, $[Sn(C_6H_5)_3(C_9H_{11}O_2S_2)]$, the coordination geometry around the Sn atom is tetrahedral, involving three C atoms from three phenyl groups and one C atom from a methyl 2-(1,3-dithian-2-ylidene)but-3-enoate group. The conformation of the heterocyclic ring is twist-boat.

Related literature

For related literature, see: Marcel (2002); Zhu *et al.* (2003)



Experimental

Crystal data

$[Sn(C_6H_5)_3(C_9H_{11}O_2S_2)]$
M_r = 565.29
Monoclinic, $P2_1/c$
a = 8.170 (4) Å
b = 14.250 (7) Å
c = 21.171 (9) Å
β = 95.871 (9)°
V = 2452 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.23 mm^{-1}
T = 293 (2) K
0.34 × 0.32 × 0.29 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.661, T_{max} = 0.703
12531 measured reflections
4503 independent reflections
3086 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.075
S = 1.00
4503 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.113 (3)	Sn1—C31	2.129 (3)
Sn1—C11	2.128 (3)	Sn1—C21	2.144 (3)
C1—Sn1—C11	114.15 (13)	C1—Sn1—C21	105.21 (13)
C1—Sn1—C31	111.22 (13)	C11—Sn1—C21	110.69 (12)
C11—Sn1—C31	109.40 (13)	C31—Sn1—C21	105.80 (12)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2075).

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supplementary materials

Acta Cryst. (2007). E**63**, m2346 [**doi:10.1107/S160053680703961X**]

(*Z*)-Methyl 2-(1,3-dithian-2-ylidene)-4-(triphenylstannyl)but-3-enoate

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Comment

In recent years there has been considerable interest in organotin compounds. Many organotin compounds are biologically active (Marcel, 2002). In this paper, the structure of the title compound is described.

As shown in Fig. 1, the coordination geometry around the Sn atom is tetrahedral, involving three C atoms from three phenyl groups and one C atom from a methy 2-(1,3-dithian-2-ylidene)but-3-enoate group (Table 1). The bond distances and angles are normal (Zhu *et al.*, 2003).

Experimental

Methyl 2-(1,3-dithian-2-ylidene)but-3-enoate (10.70 g, 50 mmol) and dibenzoyl peroxide (0.100 g, 0.83 mmol) were added to dry ether (200 ml), and then triphenyltin hydride (21.94 g, 62.5 mmol) was added. The mixture was stirred for 30 h at room temperature. The ether was evaporated off and the residue was recrystallized three times from ethanol. Colorless single crystals were obtained by evaporation of ethanol at room temperature (17 g, yield 60%).

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93Å (CH), 0.97Å (CH_2) and U_{iso} = 1.2U_{eq}(C), and with C—H = 0.96Å (CH_3) and U_{iso} = 1.5U_{eq}(C).

Figures

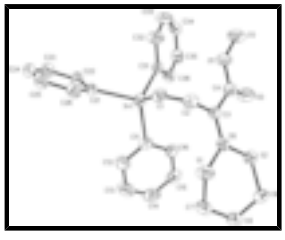


Fig. 1. The molecular structure of the title compound.

(*Z*)-Methyl 2-(1,3-dithian-2-ylidene)-4-(triphenylstannyl)but-3-enoate

Crystal data

$[Sn(C_6H_5)_3(C_9H_{11}O_2S_2)]$	F_{000} = 1144
M_r = 565.29	D_x = 1.531 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 3517 reflections
a = 8.170 (4) Å	θ = 1.7–25.4º
b = 14.250 (7) Å	μ = 1.23 mm^{-1}
c = 21.171 (9) Å	T = 293 (2) K
β = 95.871 (9)º	Block, colorless
V = 2452 (2) $Å^3$	0.34 × 0.32 × 0.29 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4503 independent reflections
Radiation source: fine-focus sealed tube	3086 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 293(2) K	θ_{max} = 25.4º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
T_{min} = 0.661, T_{max} = 0.703	$k = -15 \rightarrow 17$
12531 measured reflections	$l = -20 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.030	H-atom parameters constrained
$wR(F^2)$ = 0.075	$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 0.6236P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
4503 reflections	$\Delta\rho_{max}$ = 0.37 e $Å^{-3}$
289 parameters	$\Delta\rho_{min}$ = −0.26 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}
Sn1	0.14620 (3)	0.750137 (16)	0.024269 (10)	0.05035 (9)
S1	−0.33618 (14)	0.63882 (7)	0.09192 (5)	0.0722 (3)
S2	−0.20634 (13)	0.70399 (7)	0.22197 (4)	0.0599 (2)
O1	−0.0865 (3)	0.95267 (16)	0.11336 (11)	0.0649 (7)
O2	−0.0514 (4)	0.87350 (17)	0.20459 (12)	0.0790 (8)
C1	−0.0983 (4)	0.7962 (2)	0.00111 (15)	0.0531 (8)
H1A	−0.1334	0.8059	−0.0416	0.064*
C2	−0.2048 (4)	0.8123 (2)	0.04241 (15)	0.0546 (9)
H2A	−0.3084	0.8338	0.0266	0.066*
C3	−0.1736 (4)	0.7991 (2)	0.11178 (14)	0.0436 (7)

C4	−0.0978 (4)	0.8761 (2)	0.14920 (17)	0.0526 (8)
C5	−0.0064 (6)	1.0320 (3)	0.1450 (2)	0.0967 (15)
H5A	−0.0034	1.0830	0.1155	0.145*
H5B	0.1038	1.0151	0.1610	0.145*
H5C	−0.0662	1.0508	0.1797	0.145*
C6	−0.2290 (4)	0.7211 (2)	0.13986 (15)	0.0470 (8)
C7	−0.2896 (6)	0.5317 (3)	0.13250 (19)	0.0846 (13)
H7A	−0.3470	0.4817	0.1083	0.102*
H7B	−0.1727	0.5201	0.1323	0.102*
C8	−0.3302 (6)	0.5244 (3)	0.1989 (2)	0.0867 (13)
H8A	−0.4291	0.4869	0.1994	0.104*
H8B	−0.2418	0.4911	0.2235	0.104*
C9	−0.3562 (5)	0.6145 (3)	0.23040 (19)	0.0770 (12)
H9A	−0.3598	0.6027	0.2754	0.092*
H9B	−0.4631	0.6386	0.2139	0.092*
C11	0.1669 (4)	0.6234 (2)	0.07770 (15)	0.0514 (8)
C12	0.1930 (5)	0.5402 (3)	0.04814 (19)	0.0772 (12)
H12A	0.2003	0.5394	0.0046	0.093*
C13	0.2086 (6)	0.4572 (3)	0.0819 (2)	0.0954 (15)
H13A	0.2221	0.4010	0.0609	0.115*
C14	0.2042 (5)	0.4576 (3)	0.1457 (2)	0.0854 (13)
H14A	0.2185	0.4020	0.1686	0.102*
C15	0.1791 (5)	0.5390 (3)	0.17601 (19)	0.0706 (11)
H15A	0.1746	0.5392	0.2197	0.085*
C16	0.1602 (4)	0.6215 (3)	0.14231 (16)	0.0594 (9)
H16A	0.1424	0.6771	0.1636	0.071*
C21	0.2445 (4)	0.7326 (2)	−0.06506 (15)	0.0489 (8)
C22	0.4102 (5)	0.7381 (2)	−0.06899 (17)	0.0629 (10)
H22A	0.4811	0.7445	−0.0320	0.075*
C23	0.4741 (5)	0.7344 (3)	−0.1265 (2)	0.0734 (12)
H23A	0.5873	0.7384	−0.1279	0.088*
C24	0.3745 (6)	0.7250 (3)	−0.18070 (19)	0.0714 (12)
H24A	0.4186	0.7238	−0.2195	0.086*
C25	0.2125 (6)	0.7174 (3)	−0.17882 (17)	0.0731 (12)
H25A	0.1441	0.7093	−0.2163	0.088*
C26	0.1451 (5)	0.7214 (2)	−0.12110 (17)	0.0626 (10)
H26A	0.0317	0.7166	−0.1204	0.075*
C31	0.2921 (4)	0.8562 (2)	0.07341 (14)	0.0486 (8)
C32	0.3052 (5)	0.9437 (3)	0.04708 (17)	0.0722 (11)
H32A	0.2424	0.9577	0.0091	0.087*
C33	0.4084 (6)	1.0109 (3)	0.0754 (2)	0.0796 (12)
H33A	0.4167	1.0689	0.0559	0.095*
C34	0.4985 (4)	0.9933 (3)	0.13154 (18)	0.0621 (9)
H34A	0.5670	1.0392	0.1511	0.075*
C35	0.4872 (4)	0.9076 (3)	0.15873 (16)	0.0620 (9)
H35A	0.5488	0.8946	0.1971	0.074*
C36	0.3853 (4)	0.8396 (2)	0.13000 (15)	0.0566 (9)
H36A	0.3795	0.7813	0.1494	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.05945 (16)	0.05002 (14)	0.04181 (13)	−0.00129 (12)	0.00632 (10)	0.00262 (11)
S1	0.0930 (8)	0.0571 (6)	0.0637 (6)	−0.0188 (5)	−0.0064 (5)	0.0009 (5)
S2	0.0757 (6)	0.0560 (5)	0.0489 (5)	−0.0053 (5)	0.0109 (4)	0.0064 (4)
O1	0.0839 (18)	0.0426 (14)	0.0678 (16)	−0.0125 (12)	0.0061 (13)	0.0067 (12)
O2	0.115 (2)	0.0654 (18)	0.0524 (15)	−0.0256 (16)	−0.0129 (15)	0.0019 (13)
C1	0.059 (2)	0.057 (2)	0.0416 (18)	−0.0067 (18)	−0.0023 (16)	0.0089 (16)
C2	0.049 (2)	0.057 (2)	0.056 (2)	−0.0015 (17)	−0.0036 (17)	0.0101 (17)
C3	0.0457 (19)	0.0410 (18)	0.0441 (18)	0.0045 (15)	0.0039 (14)	0.0035 (15)
C4	0.053 (2)	0.046 (2)	0.058 (2)	−0.0027 (16)	0.0064 (17)	0.0038 (17)
C5	0.140 (4)	0.052 (3)	0.101 (3)	−0.036 (3)	0.025 (3)	−0.008 (2)
C6	0.049 (2)	0.0465 (19)	0.0458 (18)	0.0038 (14)	0.0045 (15)	0.0005 (14)
C7	0.120 (4)	0.048 (2)	0.088 (3)	−0.005 (2)	0.022 (3)	−0.006 (2)
C8	0.125 (4)	0.052 (3)	0.087 (3)	−0.011 (2)	0.034 (3)	0.002 (2)
C9	0.099 (3)	0.063 (3)	0.075 (3)	−0.007 (2)	0.037 (2)	0.011 (2)
C11	0.052 (2)	0.049 (2)	0.053 (2)	−0.0044 (16)	0.0048 (16)	0.0063 (16)
C12	0.109 (3)	0.062 (3)	0.060 (2)	−0.003 (2)	0.003 (2)	0.001 (2)
C13	0.138 (4)	0.051 (3)	0.096 (4)	0.003 (3)	0.004 (3)	0.002 (2)
C14	0.100 (4)	0.066 (3)	0.088 (3)	−0.003 (2)	−0.001 (3)	0.026 (2)
C15	0.068 (3)	0.081 (3)	0.065 (2)	−0.001 (2)	0.011 (2)	0.020 (2)
C16	0.060 (2)	0.063 (2)	0.057 (2)	0.0037 (18)	0.0154 (18)	0.0087 (18)
C21	0.062 (2)	0.040 (2)	0.0447 (18)	−0.0042 (15)	0.0044 (16)	0.0015 (13)
C22	0.064 (2)	0.073 (3)	0.051 (2)	−0.002 (2)	0.0013 (17)	−0.0087 (18)
C23	0.072 (3)	0.072 (3)	0.080 (3)	−0.008 (2)	0.025 (2)	−0.011 (2)
C24	0.107 (4)	0.056 (3)	0.055 (2)	0.000 (2)	0.028 (2)	−0.0018 (18)
C25	0.108 (4)	0.066 (3)	0.042 (2)	0.011 (2)	−0.007 (2)	−0.0076 (17)
C26	0.066 (2)	0.061 (2)	0.059 (2)	0.0005 (18)	−0.0020 (19)	−0.0070 (17)
C31	0.056 (2)	0.049 (2)	0.0412 (18)	0.0033 (16)	0.0067 (15)	−0.0031 (15)
C32	0.096 (3)	0.055 (2)	0.060 (2)	−0.004 (2)	−0.016 (2)	0.0128 (19)
C33	0.108 (3)	0.047 (2)	0.081 (3)	−0.007 (2)	−0.005 (3)	0.012 (2)
C34	0.063 (2)	0.055 (2)	0.069 (2)	0.0017 (18)	0.008 (2)	−0.0118 (19)
C35	0.060 (2)	0.072 (3)	0.053 (2)	0.008 (2)	−0.0007 (17)	−0.0062 (19)
C36	0.065 (2)	0.051 (2)	0.053 (2)	0.0050 (18)	0.0047 (17)	0.0018 (17)

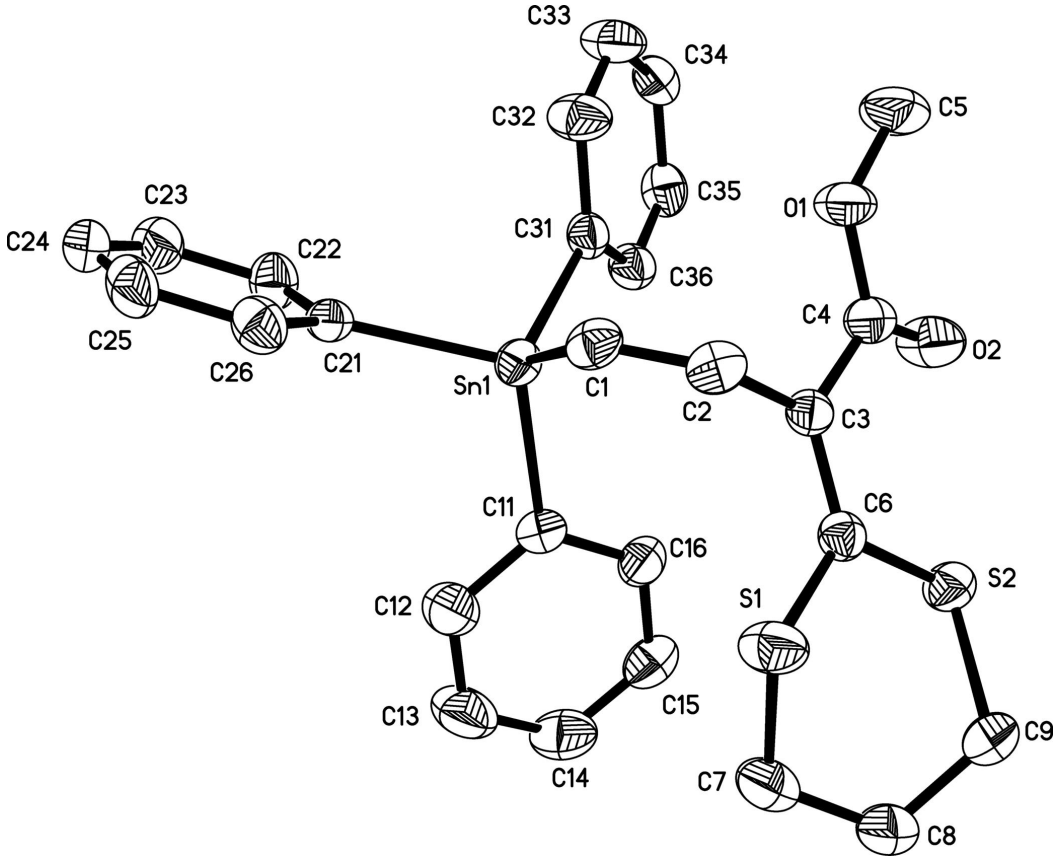
Geometric parameters (Å, °)

Sn1—C1	2.113 (3)	C12—H12A	0.9300
Sn1—C11	2.128 (3)	C13—C14	1.354 (5)
Sn1—C31	2.129 (3)	C13—H13A	0.9300
Sn1—C21	2.144 (3)	C14—C15	1.351 (6)
S1—C6	1.729 (3)	C14—H14A	0.9300
S1—C7	1.774 (4)	C15—C16	1.376 (5)
S2—C6	1.746 (3)	C15—H15A	0.9300
S2—C9	1.789 (4)	C16—H16A	0.9300
O1—C4	1.337 (4)	C21—C22	1.367 (5)
O1—C5	1.437 (4)	C21—C26	1.377 (5)

O2—C4	1.196 (4)	C22—C23	1.373 (5)
C1—C2	1.315 (4)	C22—H22A	0.9300
C1—H1A	0.9300	C23—C24	1.344 (6)
C2—C3	1.477 (4)	C23—H23A	0.9300
C2—H2A	0.9300	C24—C25	1.333 (6)
C3—C6	1.360 (4)	C24—H24A	0.9300
C3—C4	1.455 (4)	C25—C26	1.392 (5)
C5—H5A	0.9600	C25—H25A	0.9300
C5—H5B	0.9600	C26—H26A	0.9300
C5—H5C	0.9600	C31—C36	1.372 (4)
C7—C8	1.481 (5)	C31—C32	1.374 (4)
C7—H7A	0.9700	C32—C33	1.373 (5)
C7—H7B	0.9700	C32—H32A	0.9300
C8—C9	1.473 (5)	C33—C34	1.356 (5)
C8—H8A	0.9700	C33—H33A	0.9300
C8—H8B	0.9700	C34—C35	1.358 (5)
C9—H9A	0.9700	C34—H34A	0.9300
C9—H9B	0.9700	C35—C36	1.377 (5)
C11—C12	1.368 (5)	C35—H35A	0.9300
C11—C16	1.375 (4)	C36—H36A	0.9300
C12—C13	1.381 (5)		
C1—Sn1—C11	114.15 (13)	C11—C12—C13	121.0 (4)
C1—Sn1—C31	111.22 (13)	C11—C12—H12A	119.5
C11—Sn1—C31	109.40 (13)	C13—C12—H12A	119.5
C1—Sn1—C21	105.21 (13)	C14—C13—C12	120.2 (4)
C11—Sn1—C21	110.69 (12)	C14—C13—H13A	119.9
C31—Sn1—C21	105.80 (12)	C12—C13—H13A	119.9
C6—S1—C7	103.22 (18)	C15—C14—C13	119.9 (4)
C6—S2—C9	101.23 (18)	C15—C14—H14A	120.1
C4—O1—C5	115.6 (3)	C13—C14—H14A	120.1
C2—C1—Sn1	125.0 (3)	C14—C15—C16	120.1 (4)
C2—C1—H1A	117.5	C14—C15—H15A	119.9
Sn1—C1—H1A	117.5	C16—C15—H15A	119.9
C1—C2—C3	125.5 (3)	C11—C16—C15	121.2 (4)
C1—C2—H2A	117.3	C11—C16—H16A	119.4
C3—C2—H2A	117.3	C15—C16—H16A	119.4
C6—C3—C4	121.4 (3)	C22—C21—C26	117.1 (3)
C6—C3—C2	120.5 (3)	C22—C21—Sn1	120.6 (2)
C4—C3—C2	117.8 (3)	C26—C21—Sn1	122.2 (3)
O2—C4—O1	123.3 (3)	C21—C22—C23	121.3 (4)
O2—C4—C3	125.8 (3)	C21—C22—H22A	119.3
O1—C4—C3	110.9 (3)	C23—C22—H22A	119.3
O1—C5—H5A	109.5	C24—C23—C22	120.6 (4)
O1—C5—H5B	109.5	C24—C23—H23A	119.7
H5A—C5—H5B	109.5	C22—C23—H23A	119.7
O1—C5—H5C	109.5	C25—C24—C23	119.9 (4)
H5A—C5—H5C	109.5	C25—C24—H24A	120.0
H5B—C5—H5C	109.5	C23—C24—H24A	120.0
C3—C6—S1	118.0 (2)	C24—C25—C26	120.4 (4)

C3—C6—S2	122.9 (2)	C24—C25—H25A	119.8
S1—C6—S2	119.04 (19)	C26—C25—H25A	119.8
C8—C7—S1	117.6 (3)	C21—C26—C25	120.6 (4)
C8—C7—H7A	107.9	C21—C26—H26A	119.7
S1—C7—H7A	107.9	C25—C26—H26A	119.7
C8—C7—H7B	107.9	C36—C31—C32	116.8 (3)
S1—C7—H7B	107.9	C36—C31—Sn1	122.5 (2)
H7A—C7—H7B	107.2	C32—C31—Sn1	120.6 (3)
C9—C8—C7	115.1 (3)	C33—C32—C31	121.7 (4)
C9—C8—H8A	108.5	C33—C32—H32A	119.2
C7—C8—H8A	108.5	C31—C32—H32A	119.2
C9—C8—H8B	108.5	C34—C33—C32	120.6 (4)
C7—C8—H8B	108.5	C34—C33—H33A	119.7
H8A—C8—H8B	107.5	C32—C33—H33A	119.7
C8—C9—S2	116.4 (3)	C33—C34—C35	118.9 (4)
C8—C9—H9A	108.2	C33—C34—H34A	120.6
S2—C9—H9A	108.2	C35—C34—H34A	120.6
C8—C9—H9B	108.2	C34—C35—C36	120.6 (3)
S2—C9—H9B	108.2	C34—C35—H35A	119.7
H9A—C9—H9B	107.3	C36—C35—H35A	119.7
C12—C11—C16	117.5 (3)	C31—C36—C35	121.4 (3)
C12—C11—Sn1	120.0 (3)	C31—C36—H36A	119.3
C16—C11—Sn1	122.4 (3)	C35—C36—H36A	119.3

Fig. 1


C33
C34
C32
C5
C35
C23
O1
C24
C31
C22
C36
C4
C25
O2
C26
C21
Sn1
C1
C2
C3
C11
C6
C16
S2
C12
S1
C15
C13
C9
C14
C7
C8