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Crystal structure of a one-dimensional coordination polymer of tin(IV) bromide with 1,4-dithiane

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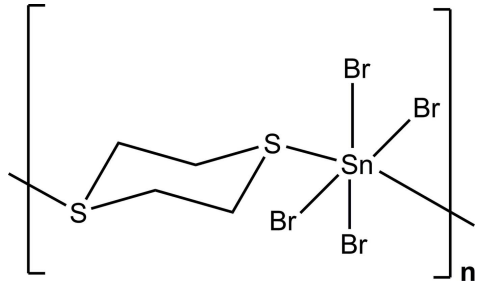
The title compound, $[SnBr_4(C_4H_8S_2)]$ {systematic name: *catena*-poly[[tetrabromidotin(IV)]-μ-1,4-dithiane-κ^2S:S']}, represents the first 1,4-dithiane complex with tin as coordination centre. The asymmetric unit consist of half a formula unit with the tin(IV) atom at the centre of symmetry at 0,0,1/2 (Wyckoff symbol *b*) and a centrosymmetric 1,4-dithiane molecule with the centre of symmetry in 1/2,0,1 (Wyckoff symbol *c*). The tin(IV) atom is coordinated in a distorted octahedral manner by the four bromine atoms and two sulfur atoms of two 1,4-dithiane molecules in a *trans*-position. Sn—Br [mean value: 2.561 (5) Å] and Sn—S distances [2.6546 (6) Å] are in the typical range for octahedrally coordinated tin(IV) atoms and the dithiane molecule adopts a chair conformation. The one-dimensional polymeric chains propagate along the [101] direction with weak intermolecular Br···Br [3.5724 (4) Å] between parallel chains and weak Br···H interactions [2.944–2.993 Å] within the chains.

Keywords: crystal structure; tin(IV) bromide; 1,4-dithiane; coordination polymer; Br···Br interactions; Br···H interactions.

CCDC reference: 1442283

1. Related literature

For the structural parameters in macrocyclic thioether complexes with $SnBr_4$, see: Levason *et al.* (2003), and for dithioether complexes with $SnBr_4$, see: Dann *et al.* (1996). For the oxidation of tin(II) to tin(IV), see: Deacon *et al.* (1997).



2. Experimental

2.1. Crystal data

$[SnBr_4(C_4H_8S_2)]$
M_r = 558.55
Monoclinic, $P2_1/n$
a = 7.1033 (4) Å
b = 12.0526 (8) Å
c = 7.4032 (5) Å
β = 112.144 (2)°
V = 587.06 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 16.09 mm^{-1}
T = 100 K
0.16 × 0.06 × 0.06 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.182, T_{max} = 0.450
22217 measured reflections
1426 independent reflections
1339 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.036
S = 1.14
1426 reflections
54 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected contacts (Å).

Br1···H11^{i}	2.965	Br1···H12^{iv}	3.078
Br1···H21^{ii}	2.993	Br1···H11^{v}	3.079
Br2···H22^{iii}	2.944	Br1···Br2^{vi}	3.5724 (4)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1, -y, -z+2$; (iii) $x-1, y, z-1$; (iv) $x, y, z-1$; (v) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (vi) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014*/7 (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: NR2064).

References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2009). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Dann, S. E., Genge, A. R. J., Levason, W. & Reid, G. (1996). *J. Chem. Soc. Dalton Trans.* pp. 4471–4478.

Deacon, P. R., Mahon, M. F., Molloy, K. C. & Waterfield, P. C. (1997). *J. Chem. Soc. Dalton Trans.* pp. 3705–3712.

Levason, W., Matthews, M. L., Patel, R., Reid, G. & Webster, M. (2003). *New J. Chem.* **27**, 1784–1788.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

supporting information

Acta Cryst. (2015). E**71**, m267–m268 [https://doi.org/10.1107/S2056989015023932]

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S1. Synthesis and crystallization

A mixture of 0.55 g (2 mmol) $SnBr_2$ and 0.24 g (2 mmol) 1,4-dithiane was heated in a closed ampule to 130 °C for 6 hours. No special care was taken to exclude oxygen or humidity. After cooling, the ampule was opened and its solid content inspected by optical microscopy. Only one fragment, a yellow needle-like crystal of the title compound proved to be suitable for single crystal X-ray diffraction. The presence of tin(IV) in the title compound instead of tin(II) demonstrates the complexity of reactions that must have taken place. Sensitivity of tin(II) compounds towards oxidation by air, however, is not unusual and well documented in literature (e.g. Deacon *et al*, 1997).

S1.1. Refinement

All hydrogen atoms could be localized in difference Fourier syntheses but were refined in geometric positions riding on the carbon atoms with C—H distances of 0.99 Å (-CH_2-) and one common, free refined isotropic displacement factor.

S2. Results and discussion

Only some few coordination compounds of tin(IV) bromide with Lewis-bases containing two or more S-atoms as Lewis-base centers have been structurally characterized. The main structural features are one-dimensional chain structures in case of macrocyclic thioether complexes (Levason *et al.*, 2003) with the Lewis-base molecules in a *cis*- , and *trans*-position, respectiveley, and the formation of monomeric complexes as a result of chelatization in case of open chain di-thioether molecules (Dann *et al.*, 1996). In all cases, the tin atoms are octahedrally coordinated with similar Sn—Br and Sn—S bond lengths.

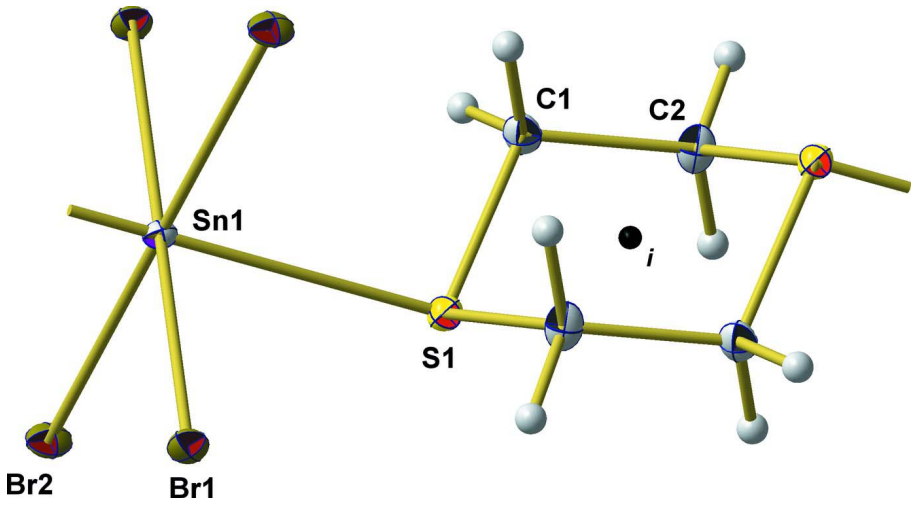

Figure 1

Ball-and-stick model of the asymmetric unit of the title compound with the atomic numbering scheme used. For a better understanding the asymmetric unit of the 1,4-dithiane molecule has been extended by its symmetry-related atoms generated by the centre of symmetry *i* (black dot) at 1/2,0,1. With exception of the H atoms, which are shown as spheres of arbitrary radius, all atoms are drawn as displacement ellipsoids at the 50% probability level.

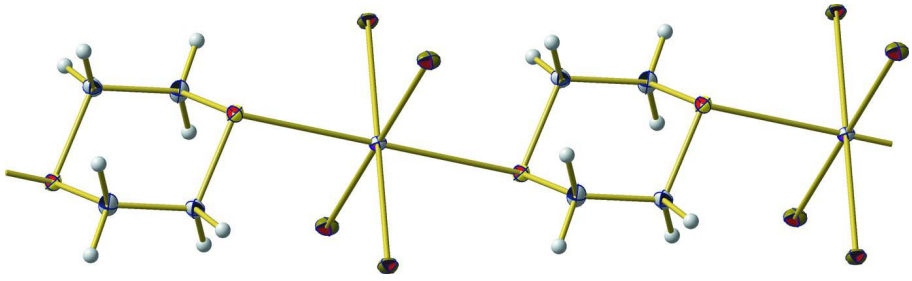

Figure 2

Part of the one-dimensional coordination polymer showing two complete building units.

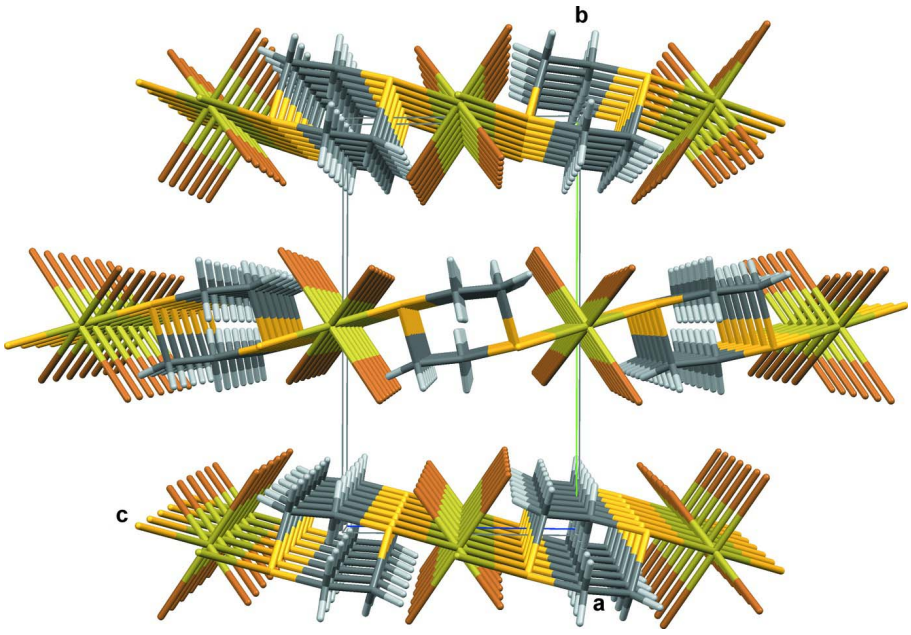

Figure 3

Perspective view of the crystal structure looking down the *a* axis.

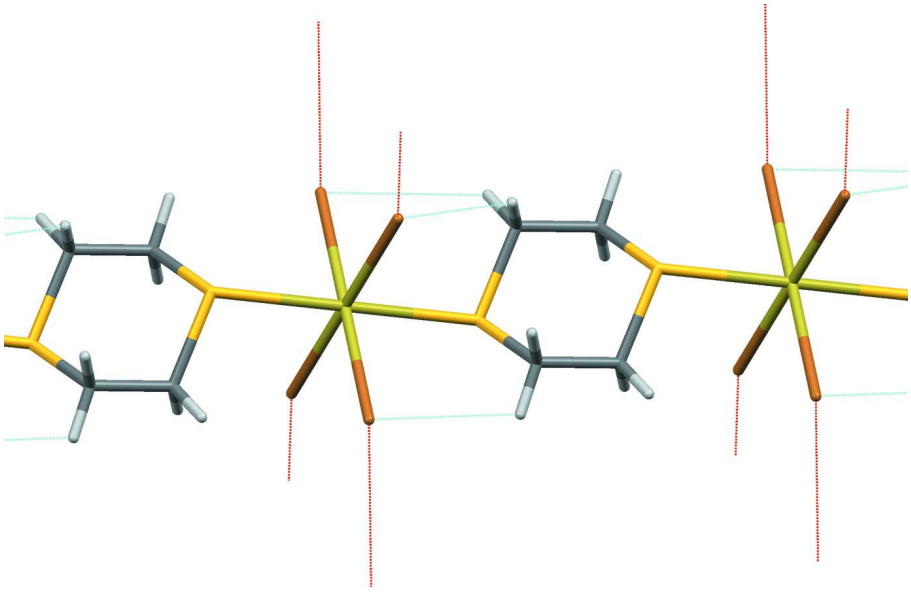

Figure 4

Shortest intrachain H···Br (blue) and interchain Br···Br (red) interactions.

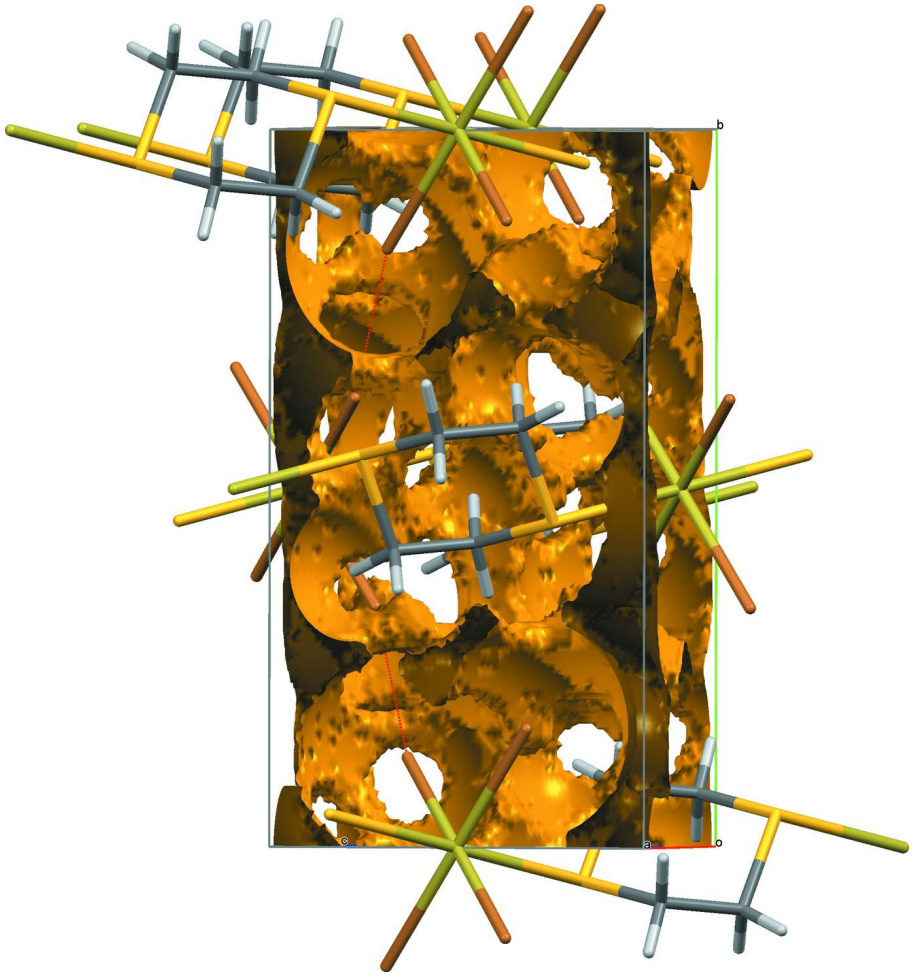

Figure 5

Three-dimensional representation of the contact surface (probe radius = 0.2 Å, outside color = yellow, inside color = brown) within the unit cell visualizing Br···Br interactions (red) between neighboring chains through holes in the surface.

catena-Poly[[tetrabromidotin(IV)]-μ-1,4-dithiane-κ²S:S′]

Crystal data

$[SnBr_4(C_4H_8S_2)]$
M_r = 558.55
Monoclinic, $P2_1/n$
a = 7.1033 (4) Å
b = 12.0526 (8) Å
c = 7.4032 (5) Å
β = 112.144 (2)°
V = 587.06 (7) Å^3
Z = 2
F(000) = 508
D_x = 3.160 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9935 reflections
θ = 3.4–28.7°
μ = 16.09 mm^{-1}
T = 100 K
Needle, yellow
0.16 × 0.06 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.182, T_{max} = 0.450
22217 measured reflections

1426 independent reflections
1339 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 28.0°$, $\theta_{min} = 3.4°$

$h = -9 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.036$
$S = 1.14$
1426 reflections
54 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0043P)^2 + 0.5753P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Extinction correction: *SHELXL2014*/7 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0042 (3)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.0000	0.0000	0.5000	0.00740 (7)
Br1	0.17963 (4)	−0.12497 (2)	0.33464 (3)	0.01109 (8)
Br2	0.04504 (4)	0.16589 (2)	0.30464 (4)	0.01267 (8)
S1	0.36282 (9)	0.04681 (5)	0.76832 (9)	0.01033 (13)
C1	0.3132 (4)	0.0756 (2)	0.9867 (3)	0.0119 (5)
H11	0.2349	0.1455	0.9686	0.015 (4)*
H12	0.2295	0.0151	1.0078	0.015 (4)*
C2	0.5088 (4)	0.0860 (2)	1.1657 (4)	0.0129 (5)
H21	0.4771	0.1146	1.2764	0.015 (4)*
H22	0.6002	0.1399	1.1388	0.015 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.00749 (13)	0.00663 (12)	0.00903 (13)	0.00013 (8)	0.00420 (9)	0.00008 (8)
Br1	0.01171 (14)	0.01121 (13)	0.01229 (14)	0.00266 (9)	0.00672 (10)	−0.00103 (9)
Br2	0.01522 (15)	0.00948 (13)	0.01585 (14)	0.00006 (9)	0.00873 (11)	0.00347 (9)
S1	0.0096 (3)	0.0106 (3)	0.0110 (3)	0.0001 (2)	0.0041 (2)	0.0002 (2)
C1	0.0122 (12)	0.0131 (12)	0.0102 (12)	0.0027 (10)	0.0041 (10)	−0.0012 (10)
C2	0.0121 (12)	0.0117 (12)	0.0124 (12)	0.0029 (10)	0.0017 (10)	−0.0038 (10)

Geometric parameters (Å, º)

Sn1—Br2^{i}	2.5574 (3)	Br1—H11^{v}	3.0788
Sn1—Br2	2.5574 (3)	Br1—Br2^{vi}	3.5724 (4)
Sn1—Br1	2.5638 (2)	S1—C1	1.813 (2)
Sn1—Br1^{i}	2.5638 (3)	S1—C2^{ii}	1.816 (3)
Sn1—S1^{i}	2.6546 (6)	C1—C2	1.521 (3)
Sn1—S1	2.6546 (6)	C1—H11	0.9900
Br1—H11^{i}	2.9646	C1—H12	0.9900
Br1—H21^{ii}	2.9932	C2—S1^{ii}	1.816 (3)
Br2—H22^{iii}	2.9438	C2—H21	0.9900
Br1—H12^{iv}	3.0783	C2—H22	0.9900
Br2^{i}—Sn1—Br2	180.0	H21^{ii}—Br1—H11^{v}	68.8
Br2^{i}—Sn1—Br1	90.092 (9)	H12^{iv}—Br1—H11^{v}	143.5
Br2—Sn1—Br1	89.908 (9)	Sn1—Br1—Br2^{vi}	167.821 (10)
Br2^{i}—Sn1—Br1^{i}	89.908 (9)	H11^{i}—Br1—Br2^{vi}	102.6
Br2—Sn1—Br1^{i}	90.092 (9)	H21^{ii}—Br1—Br2^{vi}	88.3
Br1—Sn1—Br1^{i}	180.0	H12^{iv}—Br1—Br2^{vi}	85.5
Br2^{i}—Sn1—S1^{i}	87.931 (15)	H11^{v}—Br1—Br2^{vi}	58.0
Br2—Sn1—S1^{i}	92.069 (15)	Sn1—Br2—H22^{iii}	79.0
Br1—Sn1—S1^{i}	92.027 (15)	C1—S1—C2^{ii}	100.11 (12)
Br1^{i}—Sn1—S1^{i}	87.973 (15)	C1—S1—Sn1	104.35 (8)
Br2^{i}—Sn1—S1	92.069 (15)	C2^{ii}—S1—Sn1	105.15 (8)
Br2—Sn1—S1	87.931 (15)	C2—C1—S1	111.83 (17)
Br1—Sn1—S1	87.973 (15)	C2—C1—H11	109.2
Br1^{i}—Sn1—S1	92.027 (15)	S1—C1—H11	109.2
S1^{i}—Sn1—S1	180.00 (3)	C2—C1—H12	109.2
Sn1—Br1—H11^{i}	83.1	S1—C1—H12	109.2
Sn1—Br1—H21^{ii}	83.6	H11—C1—H12	107.9
H11^{i}—Br1—H21^{ii}	161.5	C1—C2—S1^{ii}	111.25 (17)
Sn1—Br1—H12^{iv}	106.2	C1—C2—H21	109.4
H11^{i}—Br1—H12^{iv}	80.0	S1^{ii}—C2—H21	109.4
H21^{ii}—Br1—H12^{iv}	116.1	C1—C2—H22	109.4
Sn1—Br1—H11^{v}	110.3	S1^{ii}—C2—H22	109.4
H11^{i}—Br1—H11^{v}	104.2	H21—C2—H22	108.0
Br2^{i}—Sn1—S1—C1	−60.87 (9)	Br1—Sn1—S1—C2^{ii}	−46.00 (9)
Br2—Sn1—S1—C1	119.13 (9)	Br1^{i}—Sn1—S1—C2^{ii}	134.00 (9)
Br1—Sn1—S1—C1	−150.89 (9)	Sn1—S1—C1—C2	170.73 (16)
Br1^{i}—Sn1—S1—C1	29.11 (9)	S1—C1—C2—S1^{ii}	−69.0 (2)
Br2^{i}—Sn1—S1—C2^{ii}	44.02 (9)	C1—C2—S1^{ii}—C1^{ii}	61.7 (2)
Br2—Sn1—S1—C2^{ii}	−135.98 (9)	C2^{ii}—S1—C1—C2	62.1 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*+2; (iii) *x*−1, *y*, *z*−1; (iv) *x*, *y*, *z*−1; (v) −*x*+1/2, *y*−1/2, −*z*+3/2; (vi) −*x*+1/2, *y*−1/2, −*z*+1/2.