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Di-*tert*-butylbis(*N*-isopropyl-*N*-methyldithiocarbamato-κ^2S,S')tin(IV)

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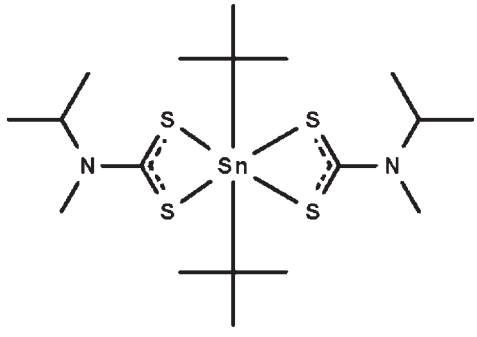
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; R factor = 0.062; wR factor = 0.186; data-to-parameter ratio = 23.5.

The dithiocarbamate anions in the title compound, $[Sn(C_4H_9)_2(C_5H_{10}NS_2)_2]$, chelate to the Sn^{IV} atom, which is six-coordinated in a skew-trapezoidal-bipyramidal geometry. The molecule lies across a twofold rotation axis. The crystal studied was a non-merohedral twin, the ratio of the twin components being 0.82 (1):0.18 (1).

Related literature

For the crystal structure of di(tert-butyl)bis(*N*,*N*-dimethyldithiocarbamato)tin(IV), see: Kim *et al.* (1987). For a discussion of the geometry of tin in diorganotin bischelates, see: Ng *et al.* (1987). For the treatment of non-merohedral twinning, see: Spek (2009).



Experimental

Crystal data

$[Sn(C_4H_9)_2(C_5H_{10}NS_2)_2]$ M_r = 529.43
Monoclinic, $P2/n$
a = 11.2934 (11) Å
b = 7.0175 (7) Å
c = 15.6894 (15) Å
β = 95.016 (1)°
V = 1238.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.37 mm^{-1}
T = 293 K
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.610, T_{max} = 0.875
7346 measured reflections
2838 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.186
S = 1.09
2838 reflections
121 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.76 e Å^{-3}
$\Delta\rho_{min}$ = −1.58 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.233 (7)	Sn1—S2	2.9911 (17)
Sn1—S1	2.5444 (18)		
C1^{i}—Sn1—C1	142.5 (4)		

Symmetry code: (i) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.

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: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5041).

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Acta Cryst. (2010). E**66**, m356 [doi:10.1107/S1600536810007439]

Di-*tert*-butylbis(*N*-isopropyl-*N*-methyldithiocarbamato-κ^2S,S')tin(IV)

Amirah Faizah Muthalib, Ibrahim Baba, Mohd Wahid Samsudin and Seik Weng Ng

S1. Experimental

Di-*t*-butyltin dichloride (10 mmol), isopropylmethylamine (10 mmol) and carbon disulfide (10 mmol) were reacted in ethanol (50 ml) at 277 K to produce a white solid. The mixture was stirred for 1 h. The solid was collected and recrystallized from ethanol.

S2. Refinement

H atoms were placed in calculated positions (C–H = 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2-1.5U_{eq}(C). The structure is a non-merohedral twin. The diffraction data were separated into two components by using *PLATON* (Spek, 2009). The final difference Fourier map had a peak near S2 and a hole near Sn1. The twin matrix is (0.293 0 0.707, 0 -1 0, 1.293 0 -0.293).

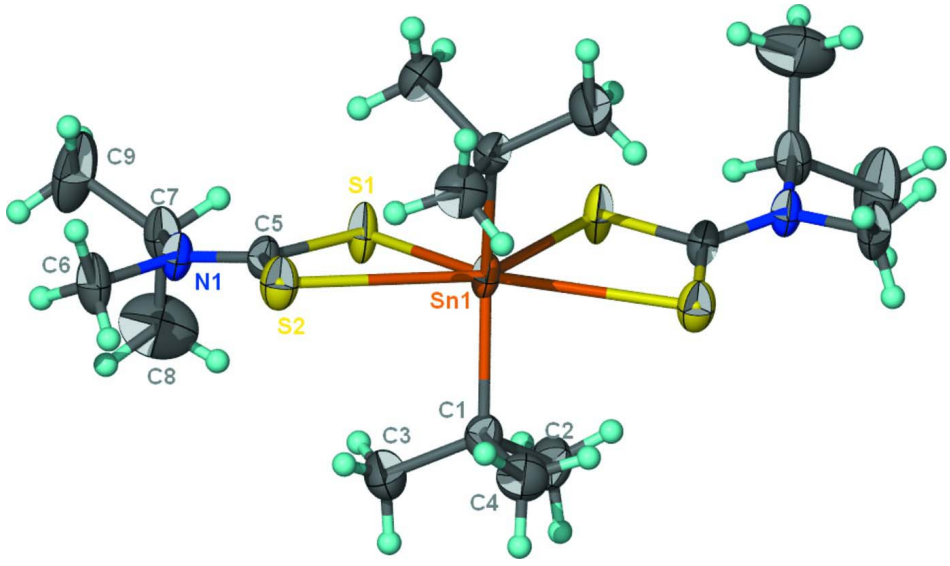

Figure 1

Displacement ellipsoid plot (Barbour, 2001) of $[Sn(C_6H_5)_2(C_5H_{11}NS_2)_2]$ at 50% probability level. H atoms are drawn as spheres of arbitrary radii. Unlabelled atoms are related to labelled atoms by the symmetry operation (3/2 - x, y, 3/2 - z).

Di-*tert*-butylbis(*N*-isopropyl-*N*-methyldithiocarbamato- κ^2S,S')tin(IV)

Crystal data

$[Sn(C_4H_9)_2(C_5H_{10}NS_2)_2]$
M_r = 529.43
Monoclinic, *P*2/*n*
Hall symbol: -P 2yac
a = 11.2934 (11) Å
b = 7.0175 (7) Å

c = 15.6894 (15) Å
β = 95.016 (1)°
V = 1238.6 (2) Å^3
Z = 2
F(000) = 548
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 4020 reflections
θ = 2.6–28.1°
μ = 1.37 mm^{-1}
T = 293 K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.610, T_{max} = 0.875

7346 measured reflections
2838 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −14→14
k = −8→9
l = −11→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.186
S = 1.09
2838 reflections
121 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
$w = 1/[\sigma^2(F_o^2) + (0.0968P)^2 + 1.7978P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.76 e Å^{-3}
$\Delta\rho_{min}$ = −1.58 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Sn1	0.7500	0.42189 (9)	0.7500	0.0369 (2)
S1	0.77107 (19)	0.1454 (3)	0.64749 (11)	0.0514 (5)
S2	0.82366 (17)	0.5223 (3)	0.57705 (11)	0.0469 (4)
N1	0.8073 (5)	0.2052 (8)	0.4857 (3)	0.0402 (12)
C1	0.5675 (6)	0.5241 (11)	0.7062 (4)	0.0426 (15)
C2	0.4811 (7)	0.3967 (12)	0.7504 (5)	0.059 (2)
H2A	0.4021	0.4474	0.7409	0.089*
H2B	0.5043	0.3925	0.8107	0.089*
H2C	0.4828	0.2702	0.7271	0.089*
C3	0.5406 (8)	0.5079 (17)	0.6112 (5)	0.070 (2)
H3A	0.4581	0.5351	0.5963	0.105*
H3B	0.5580	0.3809	0.5932	0.105*
H3C	0.5886	0.5972	0.5831	0.105*
C4	0.5556 (7)	0.7310 (12)	0.7355 (6)	0.060 (2)
H4A	0.4789	0.7793	0.7148	0.090*
H4B	0.6164	0.8071	0.7131	0.090*
H4C	0.5642	0.7362	0.7968	0.090*
C5	0.8030 (5)	0.2889 (9)	0.5620 (4)	0.0373 (13)
C6	0.8255 (8)	0.3238 (13)	0.4121 (5)	0.059 (2)

H6A	0.7772	0.4361	0.4134	0.089*
H6B	0.8038	0.2539	0.3605	0.089*
H6C	0.9077	0.3598	0.4138	0.089*
C7	0.7887 (7)	0.0018 (11)	0.4715 (5)	0.0501 (17)
H7A	0.8045	−0.0620	0.5269	0.060*
C8	0.6609 (11)	−0.0364 (16)	0.4406 (10)	0.102 (4)
H8A	0.6102	0.0090	0.4822	0.153*
H8B	0.6494	−0.1710	0.4325	0.153*
H8C	0.6418	0.0283	0.3872	0.153*
C9	0.8748 (12)	−0.0792 (14)	0.4117 (7)	0.097 (4)
H9A	0.9505	−0.0174	0.4222	0.145*
H9B	0.8442	−0.0579	0.3534	0.145*
H9C	0.8841	−0.2136	0.4216	0.145*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0481 (4)	0.0382 (4)	0.0266 (3)	0.000	0.0163 (2)	0.000
S1	0.0883 (13)	0.0388 (9)	0.0311 (8)	−0.0034 (9)	0.0284 (8)	0.0010 (7)
S2	0.0678 (11)	0.0352 (9)	0.0400 (9)	−0.0041 (8)	0.0169 (8)	0.0007 (7)
N1	0.053 (3)	0.040 (3)	0.029 (3)	0.000 (2)	0.017 (2)	−0.001 (2)
C1	0.041 (3)	0.051 (4)	0.038 (3)	−0.004 (3)	0.010 (3)	0.004 (3)
C2	0.053 (4)	0.076 (6)	0.051 (4)	−0.014 (4)	0.015 (3)	0.001 (4)
C3	0.059 (5)	0.110 (8)	0.041 (4)	0.007 (5)	0.003 (4)	0.000 (5)
C4	0.064 (5)	0.052 (5)	0.064 (5)	0.009 (4)	0.005 (4)	0.004 (4)
C5	0.046 (3)	0.038 (3)	0.030 (3)	0.004 (3)	0.015 (2)	0.005 (3)
C6	0.087 (5)	0.060 (5)	0.033 (3)	−0.007 (4)	0.020 (4)	0.006 (3)
C7	0.080 (5)	0.037 (4)	0.035 (3)	−0.001 (4)	0.014 (3)	−0.003 (3)
C8	0.095 (8)	0.071 (7)	0.134 (12)	−0.026 (6)	−0.018 (8)	−0.007 (7)
C9	0.159 (12)	0.064 (7)	0.075 (7)	0.013 (6)	0.062 (7)	−0.014 (5)

Geometric parameters (Å, º)

Sn1—C1^{i}	2.233 (7)	C3—H3B	0.96
Sn1—C1	2.233 (7)	C3—H3C	0.96
Sn1—S1	2.5444 (18)	C4—H4A	0.96
Sn1—S1^{i}	2.5444 (18)	C4—H4B	0.96
Sn1—S2^{i}	2.9911 (17)	C4—H4C	0.96
Sn1—S2	2.9911 (17)	C6—H6A	0.96
S1—C5	1.739 (6)	C6—H6B	0.96
S2—C5	1.669 (7)	C6—H6C	0.96
N1—C5	1.338 (8)	C7—C8	1.506 (13)
N1—C6	1.453 (9)	C7—C9	1.520 (12)
N1—C7	1.457 (10)	C7—H7A	0.98
C1—C3	1.500 (10)	C8—H8A	0.96
C1—C2	1.533 (10)	C8—H8B	0.96
C1—C4	1.532 (11)	C8—H8C	0.96
C2—H2A	0.96	C9—H9A	0.96

C2—H2B	0.96	C9—H9B	0.96
C2—H2C	0.96	C9—H9C	0.96
C3—H3A	0.96		

C1^{i}—Sn1—C1	142.5 (4)	H3A—C3—H3C	109.5
C1^{i}—Sn1—S1	107.74 (18)	H3B—C3—H3C	109.5
C1—Sn1—S1	100.7 (2)	C1—C4—H4A	109.5
C1^{i}—Sn1—S1^{i}	100.7 (2)	C1—C4—H4B	109.5
C1—Sn1—S1^{i}	107.74 (19)	H4A—C4—H4B	109.5
S1—Sn1—S1^{i}	80.64 (8)	C1—C4—H4C	109.5
S1—Sn1—S2	63.73 (6)	H4A—C4—H4C	109.5
S1—Sn1—S2^{i}	143.31 (6)	H4B—C4—H4C	109.5
C1—Sn1—S2	88.14 (17)	N1—C5—S2	122.8 (5)
C1^{i}—Sn1—S2^{i}	88.14 (17)	N1—C5—S1	117.5 (5)
S1^{i}—Sn1—S2	143.31 (6)	S2—C5—S1	119.7 (4)
S2^{i}—Sn1—S2	152.75 (6)	N1—C6—H6A	109.5
C1—Sn1—S2^{i}	83.17 (17)	N1—C6—H6B	109.5
C1^{i}—Sn1—S2	83.17 (17)	H6A—C6—H6B	109.5
S1^{i}—Sn1—S2^{i}	63.73 (6)	N1—C6—H6C	109.5
C5—S1—Sn1	94.8 (2)	H6A—C6—H6C	109.5
C5—N1—C6	118.6 (6)	H6B—C6—H6C	109.5
C5—N1—C7	123.4 (5)	N1—C7—C8	110.2 (7)
C6—N1—C7	117.9 (6)	N1—C7—C9	111.7 (7)
C3—C1—C2	108.9 (7)	C8—C7—C9	112.4 (9)
C3—C1—C4	110.8 (7)	N1—C7—H7A	107.4
C2—C1—C4	110.0 (6)	C8—C7—H7A	107.4
C3—C1—Sn1	112.5 (5)	C9—C7—H7A	107.4
C2—C1—Sn1	106.3 (5)	C7—C8—H8A	109.5
C4—C1—Sn1	108.3 (5)	C7—C8—H8B	109.5
C1—C2—H2A	109.5	H8A—C8—H8B	109.5
C1—C2—H2B	109.5	C7—C8—H8C	109.5
H2A—C2—H2B	109.5	H8A—C8—H8C	109.5
C1—C2—H2C	109.5	H8B—C8—H8C	109.5
H2A—C2—H2C	109.5	C7—C9—H9A	109.5
H2B—C2—H2C	109.5	C7—C9—H9B	109.5
C1—C3—H3A	109.5	H9A—C9—H9B	109.5
C1—C3—H3B	109.5	C7—C9—H9C	109.5
H3A—C3—H3B	109.5	H9A—C9—H9C	109.5
C1—C3—H3C	109.5	H9B—C9—H9C	109.5

C1^{i}—Sn1—S1—C5	−76.5 (3)	S1^{i}—Sn1—C1—C4	117.8 (5)
C1—Sn1—S1—C5	78.8 (3)	C6—N1—C5—S2	−2.8 (9)
S1^{i}—Sn1—S1—C5	−174.8 (2)	C7—N1—C5—S2	−179.6 (5)
C1^{i}—Sn1—C1—C3	103.1 (6)	C6—N1—C5—S1	175.8 (5)
S1—Sn1—C1—C3	−35.9 (6)	C7—N1—C5—S1	−1.0 (8)
S1^{i}—Sn1—C1—C3	−119.4 (6)	Sn1—S1—C5—N1	−171.6 (5)
C1^{i}—Sn1—C1—C2	−137.8 (5)	Sn1—S1—C5—S2	7.0 (4)
S1—Sn1—C1—C2	83.1 (5)	C5—N1—C7—C8	95.0 (9)

S1^{i}—Sn1—C1—C2	−0.3 (5)	C6—N1—C7—C8	−81.9 (9)
C1^{i}—Sn1—C1—C4	−19.7 (4)	C5—N1—C7—C9	−139.4 (8)
S1—Sn1—C1—C4	−158.7 (5)	C6—N1—C7—C9	43.8 (10)

Symmetry code: (i) −*x*+3/2, *y*, −*z*+3/2.