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Dimethylbis(3-methylsulfanyl-1,2,4-thiadiazole-5-thiolato)tin(IV)

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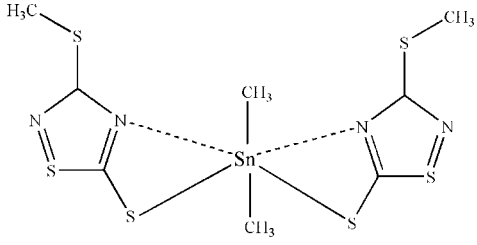
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Sn–C) = 0.009 Å; R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 18.6.

In the title compound, $[Sn(CH_3)_2(C_3H_3N_2S_3)_2]$, the Sn^{IV} atom is coordinated within a $C_2N_2S_2$ donor set that defines a skew-trapezoidal bipyramidal geometry in which the methyl groups lie over the weakly coordinated N atoms. Two independent molecules comprise the asymmetric unit, each of which lies on a mirror plane that passes through the C_2Sn unit.

Related literature

For related structures, see: Ma *et al.* (2005).



Experimental

Crystal data

$[Sn(CH_3)_2(C_3H_3N_2S_3)_2]$
M_r = 475.27
Orthorhombic, *Pnma*
a = 13.721 (9) Å
b = 16.383 (10) Å
c = 16.282 (10) Å
V = 3660 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.07 mm^{-1}
T = 293 K
0.48 × 0.37 × 0.25 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.436, T_{max} = 0.625
18306 measured reflections
3368 independent reflections
2269 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.09
3368 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: TK2508).

References

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supporting information

Acta Cryst. (2009). E**65**, m1030 [doi:10.1107/S1600536809030062]

Dimethylbis(3-methylsulfanyl-1,2,4-thiadiazole-5-thiolato)tin(IV)

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S1. Comment

Two independent molecules of $(CH_3)_2Sn(C_3H_3S_3N_2)_2$ (I) comprise the asymmetric unit, each of which has mirror symmetry so that the C_2Sn units lie on a plane (Fig. 1). The molecules have essentially equivalent bond distances and angles. Each tin atoms exists within a $C_2N_2S_2$ donor set that defines a skew-trapezoidal bipyramidal geometry where the methyl groups lie over the weakly coordinated N atoms. The structure of (I) resembles closely those reported for a series of diorganotin(IV) 2-mercapto-4-methylpyrimidine derivatives (Ma *et al.*, 2005).

S2. Experimental

3-Methylmercapto-5-mercapto-1,2,4-thiadiazole (2 mmol) was added to a solution of ethanol (20 ml) containing sodium ethoxide (2 mmol). The mixture was stirred for 30 minutes after which dimethyltin dichloride (1 mmol) was added. Stirring continued for 12 h at 318 K. After cooling to room temperature, the solution was filtered. The solvent was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ether-dichloromethane to yield coulorless crystals; m. p. 356 K. Analysis, calculated for $C_8H_{12}N_4S_6Sn$: C 20.22, H 2.54, N 11.79; Found: C 20.16, H 2.49, N 11.83%.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.96 Å, and with U_{iso}(H) = 1.5U_{eq}(C)].

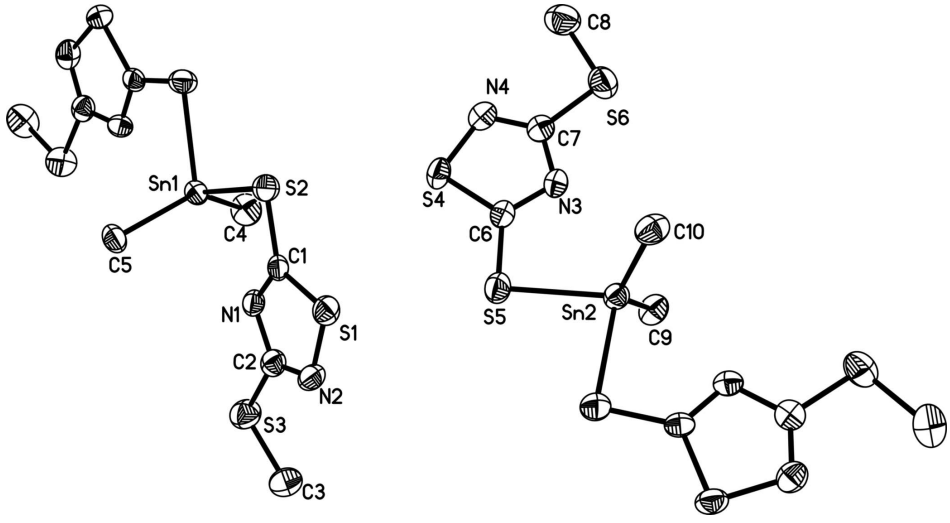

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and atom labelling.

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Crystal data

$[Sn(CH_3)_2(C_3H_3N_2S_3)_2]$
M_r = 475.27
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
a = 13.721 (9) Å
b = 16.383 (10) Å
c = 16.282 (10) Å
V = 3660 (4) Å^3
Z = 8
$F(000)$ = 1872
D_x = 1.725 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5224 reflections
θ = 3.0–24.2°
μ = 2.07 mm^{-1}
T = 293 K
Block, colourless
0.48 × 0.37 × 0.25 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.436, T_{max} = 0.625
18306 measured reflections
3368 independent reflections
2269 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 25.0°, θ_{min} = 1.9°
h = −10→16
k = −19→19
l = −17→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.09
3368 reflections
181 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
$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 5.554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Special details

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Sn1	0.82279 (4)	0.2500	0.19604 (3)	0.04949 (19)	
Sn2	0.43193 (4)	0.7500	0.09775 (3)	0.04987 (19)	

N1	0.8651 (4)	0.4226 (3)	0.1828 (3)	0.0512 (12)	
N2	0.8588 (4)	0.5637 (3)	0.1905 (3)	0.0652 (15)	
N3	0.3945 (4)	0.5731 (3)	0.0784 (3)	0.0523 (12)	
N4	0.4114 (4)	0.4321 (3)	0.0854 (3)	0.0636 (14)	
S1	0.75305 (13)	0.53359 (10)	0.23249 (11)	0.0635 (5)	
S2	0.70308 (12)	0.35277 (9)	0.24329 (11)	0.0622 (5)	
S3	1.02182 (15)	0.49793 (12)	0.12428 (12)	0.0799 (6)	
S4	0.51099 (13)	0.46584 (10)	0.13109 (10)	0.0637 (5)	
S5	0.54857 (14)	0.64762 (10)	0.14981 (11)	0.0685 (5)	
S6	0.25017 (14)	0.49009 (14)	0.00916 (12)	0.0833 (6)	
C1	0.7797 (4)	0.4321 (3)	0.2172 (3)	0.0487 (14)	
C2	0.9065 (5)	0.4983 (4)	0.1694 (4)	0.0568 (16)	
C3	1.0417 (6)	0.6055 (5)	0.1120 (5)	0.093 (3)	
H3A	1.1042	0.6144	0.0871	0.140*	
H3B	1.0398	0.6316	0.1647	0.140*	
H3C	0.9917	0.6279	0.0775	0.140*	
C4	0.8323 (8)	0.2500	0.0646 (5)	0.079 (3)	
H4A	0.7678	0.2500	0.0416	0.119*	
H4B	0.8666	0.2022	0.0467	0.119*	0.50
H4C	0.8666	0.2978	0.0467	0.119*	0.50
C5	0.9452 (7)	0.2500	0.2736 (6)	0.072 (3)	
H5A	0.9242	0.2500	0.3299	0.108*	
H5B	0.9837	0.2978	0.2632	0.108*	0.50
H5C	0.9837	0.2022	0.2632	0.108*	0.50
C6	0.4790 (4)	0.5664 (3)	0.1173 (3)	0.0486 (14)	
C7	0.3603 (5)	0.4953 (4)	0.0620 (3)	0.0525 (15)	
C8	0.2309 (6)	0.3820 (5)	0.0082 (5)	0.096 (3)	
H8A	0.1711	0.3700	−0.0198	0.144*	
H8B	0.2272	0.3623	0.0637	0.144*	
H8C	0.2841	0.3559	−0.0196	0.144*	
C9	0.4396 (8)	0.7500	−0.0320 (5)	0.066 (3)	
H9A	0.5066	0.7500	−0.0488	0.099*	
H9B	0.4078	0.7978	−0.0530	0.099*	0.50
H9C	0.4078	0.7022	−0.0530	0.099*	0.50
C10	0.3045 (7)	0.7500	0.1705 (6)	0.072 (3)	
H10A	0.3222	0.7500	0.2276	0.108*	
H10B	0.2667	0.7022	0.1586	0.108*	0.50
H10C	0.2667	0.7978	0.1586	0.108*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0439 (3)	0.0543 (3)	0.0502 (3)	0.000	−0.0037 (3)	0.000
Sn2	0.0534 (4)	0.0459 (3)	0.0503 (3)	0.000	−0.0019 (3)	0.000
N1	0.051 (3)	0.048 (3)	0.054 (3)	0.008 (2)	−0.004 (3)	0.006 (2)
N2	0.080 (4)	0.048 (3)	0.067 (3)	−0.010 (3)	−0.015 (3)	0.009 (3)
N3	0.058 (3)	0.049 (3)	0.050 (3)	0.010 (2)	0.000 (3)	0.002 (2)
N4	0.077 (4)	0.054 (3)	0.060 (3)	−0.001 (3)	0.001 (3)	0.003 (2)

S1	0.0649 (11)	0.0480 (9)	0.0776 (11)	0.0094 (8)	−0.0071 (9)	−0.0073 (8)
S2	0.0522 (10)	0.0504 (9)	0.0841 (12)	0.0054 (8)	0.0027 (9)	0.0019 (8)
S3	0.0784 (13)	0.0800 (13)	0.0814 (13)	−0.0037 (11)	0.0119 (11)	0.0128 (10)
S4	0.0728 (11)	0.0481 (9)	0.0703 (10)	0.0163 (9)	−0.0074 (9)	0.0044 (8)
S5	0.0706 (12)	0.0535 (10)	0.0813 (12)	0.0090 (9)	−0.0135 (10)	0.0023 (8)
S6	0.0677 (12)	0.1003 (15)	0.0819 (13)	−0.0007 (11)	−0.0121 (11)	0.0127 (11)
C1	0.048 (4)	0.044 (3)	0.054 (3)	0.004 (3)	−0.014 (3)	0.003 (3)
C2	0.066 (4)	0.056 (4)	0.048 (3)	−0.001 (3)	−0.009 (3)	0.001 (3)
C3	0.086 (6)	0.091 (6)	0.102 (6)	−0.019 (5)	0.005 (5)	0.031 (5)
C4	0.086 (8)	0.110 (8)	0.043 (5)	0.000	0.011 (5)	0.000
C5	0.061 (6)	0.061 (6)	0.094 (7)	0.000	−0.032 (6)	0.000
C6	0.053 (4)	0.050 (3)	0.043 (3)	0.009 (3)	0.010 (3)	0.004 (3)
C7	0.059 (4)	0.066 (4)	0.033 (3)	−0.001 (3)	0.010 (3)	0.006 (3)
C8	0.092 (6)	0.108 (6)	0.088 (5)	−0.025 (5)	−0.009 (5)	0.006 (5)
C9	0.089 (7)	0.061 (6)	0.047 (5)	0.000	−0.004 (5)	0.000
C10	0.078 (7)	0.059 (6)	0.080 (6)	0.000	0.022 (6)	0.000

Geometric parameters (Å, º)

Sn1—C5	2.102 (9)	S5—C6	1.721 (6)
Sn1—C4	2.144 (8)	S6—C7	1.741 (7)
Sn1—S2^{i}	2.475 (2)	S6—C8	1.790 (8)
Sn1—S2	2.475 (2)	C3—H3A	0.9600
Sn1—N1	2.894 (5)	C3—H3B	0.9600
Sn2—C10	2.112 (9)	C3—H3C	0.9600
Sn2—C9	2.115 (8)	C4—H4A	0.9600
Sn2—S5^{ii}	2.468 (2)	C4—H4B	0.9600
Sn2—S5	2.468 (2)	C4—H4C	0.9600
N1—C1	1.308 (7)	C5—H5A	0.9600
N1—C2	1.382 (7)	C5—H5B	0.9600
N2—C2	1.301 (8)	C5—H5C	0.9600
N2—S1	1.678 (6)	C8—H8A	0.9600
N3—C6	1.325 (7)	C8—H8B	0.9600
N3—C7	1.384 (7)	C8—H8C	0.9600
N4—C7	1.306 (7)	C9—H9A	0.9600
N4—S4	1.651 (6)	C9—H9B	0.9600
S1—C1	1.721 (6)	C9—H9C	0.9600
S2—C1	1.725 (6)	C10—H10A	0.9600
S3—C2	1.744 (7)	C10—H10B	0.9600
S3—C3	1.794 (8)	C10—H10C	0.9600
S4—C6	1.719 (6)		
C5—Sn1—C4	123.5 (4)	H3A—C3—H3C	109.5
C5—Sn1—S2^{i}	110.1 (2)	H3B—C3—H3C	109.5
C4—Sn1—S2^{i}	110.5 (2)	Sn1—C4—H4A	109.5
C5—Sn1—S2	110.1 (2)	Sn1—C4—H4B	109.5
C4—Sn1—S2	110.5 (2)	H4A—C4—H4B	109.5
S2^{i}—Sn1—S2	85.74 (9)	Sn1—C4—H4C	109.5

C5—Sn1—N1	83.37 (11)	H4A—C4—H4C	109.5
C4—Sn1—N1	85.03 (11)	H4B—C4—H4C	109.5
S2^{i}—Sn1—N1	145.17 (11)	Sn1—C5—H5A	109.5
S2—Sn1—N1	59.45 (11)	Sn1—C5—H5B	109.5
C10—Sn2—C9	127.0 (4)	H5A—C5—H5B	109.5
C10—Sn2—S5^{ii}	110.1 (2)	Sn1—C5—H5C	109.5
C9—Sn2—S5^{ii}	108.1 (2)	H5A—C5—H5C	109.5
C10—Sn2—S5	110.1 (2)	H5B—C5—H5C	109.5
C9—Sn2—S5	108.1 (2)	N3—C6—S4	111.4 (4)
S5^{ii}—Sn2—S5	85.61 (10)	N3—C6—S5	124.6 (4)
C1—N1—C2	109.2 (5)	S4—C6—S5	124.0 (4)
C1—N1—Sn1	84.5 (3)	N4—C7—N3	119.4 (6)
C2—N1—Sn1	166.2 (4)	N4—C7—S6	124.8 (5)
C2—N2—S1	107.5 (4)	N3—C7—S6	115.8 (5)
C6—N3—C7	108.2 (5)	S6—C8—H8A	109.5
C7—N4—S4	108.1 (4)	S6—C8—H8B	109.5
N2—S1—C1	92.3 (3)	H8A—C8—H8B	109.5
C1—S2—Sn1	91.8 (2)	S6—C8—H8C	109.5
C2—S3—C3	100.4 (4)	H8A—C8—H8C	109.5
N4—S4—C6	92.9 (3)	H8B—C8—H8C	109.5
C6—S5—Sn2	93.5 (2)	Sn2—C9—H9A	109.5
C7—S6—C8	100.4 (4)	Sn2—C9—H9B	109.5
N1—C1—S1	111.6 (4)	H9A—C9—H9B	109.5
N1—C1—S2	124.2 (4)	Sn2—C9—H9C	109.5
S1—C1—S2	124.2 (4)	H9A—C9—H9C	109.5
N2—C2—N1	119.4 (6)	H9B—C9—H9C	109.5
N2—C2—S3	124.8 (5)	Sn2—C10—H10A	109.5
N1—C2—S3	115.9 (5)	Sn2—C10—H10B	109.5
S3—C3—H3A	109.5	H10A—C10—H10B	109.5
S3—C3—H3B	109.5	Sn2—C10—H10C	109.5
H3A—C3—H3B	109.5	H10A—C10—H10C	109.5
S3—C3—H3C	109.5	H10B—C10—H10C	109.5

Symmetry codes: (i) x, $-y+1/2$, z; (ii) x, $-y+3/2$, z.