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Key indicators

Single-crystal X-ray study
 $T = 100\text{ K}$
 Mean $\sigma(\text{N}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.017
 wR factor = 0.041
 Data-to-parameter ratio = 19.7

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

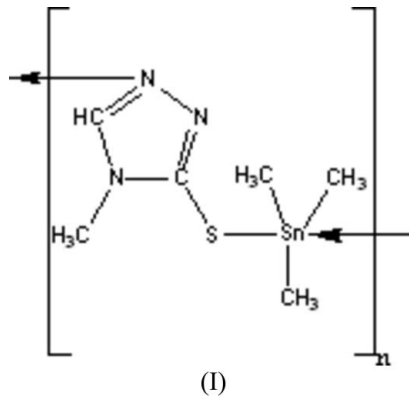
catena-Poly[[trimethyltin(IV)]- μ -4-methyl- 4*H*-1,2,4-triazole-3-thiolato- κ^2 S:N¹]

The crystal structure of the title compound, $[\text{Sn}(\text{CH}_3)_3(\text{C}_3\text{H}_4\text{N}_3\text{S})]_n$, consists of a linear chain in which adjacent trimethyltin groups are bridged by the 4-methyl-4*H*-1,2,4-triazole-3-thiolate anion through its N and S atoms.

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Comment

The synthesis and structural chemistry of organotin compounds is still a fertile area of research because of their extensive biological applications. However, there is relatively little information available on organotin compounds as anti-cancer agents *in vivo*. Diorganotins represent the largest group of tin compounds to have been extensively examined for cytotoxicity *in vitro*; they have been found to be less toxic than platinum complexes (Narayan, 1983). We report here the structure of the title compound, (I), in a continuation of our work on the synthesis and structural characterization of organotin complexes of sulfur donor ligands (Shahzadi, Ali, Bhatti *et al.*, 2006, Shahzadi, Ali & Fettouhi, 2006).



In the crystal structure of (I) (Fig. 1), the Sn atom is bonded to three methyl groups in equatorial positions. The axial positions are occupied by N and S atoms of a 4-methyl-4*H*-1,2,4-triazole-3-thiolate anion, with an almost linear S—Sn—N angle; the Sn atom has a distorted trigonal-bipyramidal coordination geometry. The Sn—S bond length is 2.7116 (7) Å, which is shorter than the Sn—S bond distance reported earlier (Shahzadi, Ali, Bhatti *et al.*, 2006, Shahzadi, Ali & Fettouhi, 2006).

Experimental

3-Mercapto-4-methyl-4*H*-1,2,4-triazole (0.15 g, 1 mmol) and triethylamine (0.1 g, 1 mmol) were suspended in dry toluene (150 ml) in a two-necked round-bottomed flask equipped with a water condenser. The mixture was stirred for 25 min at room temperature

and then trimethyltin chloride (0.2 g, 1 mmol) was added. The reaction mixture was refluxed for 4–5 h. After cooling at room temperature, triethylammonium chloride formed, was filtered off and the solvent was removed on a rotary evaporator under reduced pressure. The solid product was recrystallized from chloroform to obtain crystals suitable for X-ray analysis (yield 80%; m.p. 433 K).

Crystal data

[Sn(CH ₃) ₃ (C ₃ H ₄ N ₃ S)]	Z = 4
$M_r = 277.94$	$D_x = 1.822 \text{ Mg m}^{-3}$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 13.7254 (11) \text{ \AA}$	$\mu = 2.68 \text{ mm}^{-1}$
$b = 11.0183 (9) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 6.6998 (5) \text{ \AA}$	Plate, colourless
$V = 1013.21 (14) \text{ \AA}^3$	$0.40 \times 0.30 \times 0.05 \text{ mm}$

Data collection

Bruker APEX CCD diffractometer	7581 measured reflections
φ and ω scans	2051 independent reflections
Absorption correction: multi-scan	2032 reflections with $I > 2\sigma(I)$
(<i>SADABS</i> ; Bruker, 2001)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.414$, $T_{\text{max}} = 0.878$	$\theta_{\text{max}} = 26.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 0.5609P]$
$R[F^2 > 2\sigma(F^2)] = 0.017$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.042$	$(\Delta/\sigma)_{\text{max}} = 0.003$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.65 \text{ e \AA}^{-3}$
2051 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
104 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	928 Friedel pairs
	Flack parameter: 0.06 (2)

H atoms were included in calculated positions using the riding method, with C–H = 0.95–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

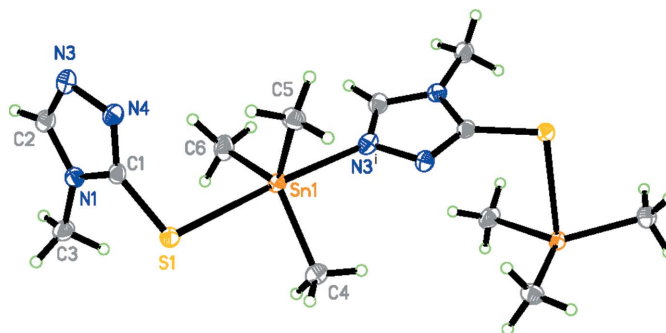


Figure 1

The structure of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$]

SHELXTL (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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supporting information

Acta Cryst. (2006). E62, m2328–m2329 [https://doi.org/10.1107/S1600536806033654]

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[Sn(CH₃)₃(C₃H₄N₃S)]

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Orthorhombic, *Pna*2₁

$a = 13.7254$ (11) Å

$b = 11.0183$ (9) Å

$c = 6.6998$ (5) Å

$V = 1013.21$ (14) Å³

$Z = 4$

$F(000) = 544$

$D_x = 1.822$ Mg m⁻³

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

Cell parameters from 6370 reflections

$\theta = 2.4$ – 26.4°

$\mu = 2.68$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.40 \times 0.30 \times 0.05$ mm

Data collection

Bruker APEX CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

$T_{\min} = 0.414$, $T_{\max} = 0.878$

7581 measured reflections

2051 independent reflections

2032 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 26.3^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -17 \rightarrow 17$

$k = -13 \rightarrow 13$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.042$

$S = 1.07$

2051 reflections

104 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 0.5609P]$

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

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.65$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Absolute structure: Flack (1983), 928 Friedel
pairs

Absolute structure parameter: 0.06 (2)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.307904 (10)	0.849930 (13)	0.50000 (4)	0.01597 (6)
S1	0.45004 (5)	0.68083 (6)	0.45904 (9)	0.01853 (14)
N1	0.43802 (16)	0.6160 (2)	0.0649 (3)	0.0171 (4)
N3	0.31104 (16)	0.4998 (2)	0.0555 (4)	0.0202 (5)
N4	0.32403 (17)	0.5347 (2)	0.2536 (4)	0.0182 (5)
C1	0.40084 (19)	0.6056 (2)	0.2553 (4)	0.0155 (5)
C2	0.3798 (2)	0.5489 (2)	-0.0520 (4)	0.0197 (6)
H2	0.3875	0.5388	-0.1919	0.024*
C3	0.52432 (17)	0.6837 (2)	0.0019 (6)	0.0242 (5)
H3A	0.5830	0.6379	0.0373	0.036*
H3B	0.5255	0.7627	0.0692	0.036*
H3C	0.5223	0.6959	-0.1429	0.036*
C4	0.4086 (2)	0.9616 (3)	0.6575 (5)	0.0250 (6)
H4A	0.3730	1.0240	0.7318	0.037*
H4B	0.4529	1.0005	0.5623	0.037*
H4C	0.4462	0.9116	0.7507	0.037*
C5	0.2120 (2)	0.7243 (3)	0.6375 (5)	0.0219 (6)
H5A	0.1450	0.7407	0.5943	0.033*
H5B	0.2163	0.7326	0.7829	0.033*
H5C	0.2302	0.6416	0.5989	0.033*
C6	0.2961 (2)	0.8696 (3)	0.1844 (5)	0.0261 (7)
H6A	0.2774	0.7916	0.1251	0.039*
H6B	0.3589	0.8955	0.1298	0.039*
H6C	0.2464	0.9306	0.1534	0.039*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01775 (9)	0.01547 (9)	0.01468 (9)	0.00015 (5)	-0.00075 (9)	-0.00140 (9)
S1	0.0168 (3)	0.0211 (3)	0.0177 (4)	0.0030 (2)	-0.0027 (2)	-0.0036 (2)
N1	0.0174 (11)	0.0145 (9)	0.0194 (11)	-0.0007 (9)	0.0019 (8)	0.0001 (8)
N3	0.0257 (13)	0.0171 (11)	0.0179 (14)	0.0021 (8)	0.0004 (8)	-0.0016 (9)
N4	0.0212 (11)	0.0174 (11)	0.0159 (12)	-0.0007 (9)	-0.0002 (9)	-0.0004 (9)
C1	0.0172 (12)	0.0133 (12)	0.0160 (12)	0.0032 (10)	-0.0016 (10)	-0.0012 (9)
C2	0.0223 (13)	0.0161 (12)	0.0205 (16)	0.0006 (10)	-0.0005 (9)	-0.0005 (10)
C3	0.0216 (11)	0.0267 (12)	0.0242 (13)	-0.0071 (9)	0.0043 (15)	-0.0060 (19)
C4	0.0235 (14)	0.0236 (14)	0.0278 (16)	-0.0035 (12)	-0.0016 (12)	-0.0064 (12)
C5	0.0195 (14)	0.0204 (15)	0.0258 (16)	-0.0002 (11)	0.0010 (11)	0.0015 (12)
C6	0.0320 (17)	0.0289 (17)	0.0173 (16)	0.0078 (12)	-0.0013 (12)	-0.0020 (12)

Geometric parameters (Å, °)

Sn1—C5	2.121 (3)	C2—H2	0.9500
Sn1—C4	2.130 (3)	C3—H3A	0.9800
Sn1—C6	2.131 (3)	C3—H3B	0.9800
Sn1—N3 ⁱ	2.351 (2)	C3—H3C	0.9800
Sn1—S1	2.7116 (7)	C4—H4A	0.9800
S1—C1	1.734 (3)	C4—H4B	0.9800
N1—C2	1.341 (3)	C4—H4C	0.9800
N1—C1	1.378 (3)	C5—H5A	0.9800
N1—C3	1.462 (3)	C5—H5B	0.9800
N3—C2	1.306 (3)	C5—H5C	0.9800
N3—N4	1.393 (3)	C6—H6A	0.9800
N3—Sn1 ⁱⁱ	2.351 (2)	C6—H6B	0.9800
N4—C1	1.312 (4)	C6—H6C	0.9800
C5—Sn1—C4	124.37 (12)	N1—C3—H3A	109.5
C5—Sn1—C6	116.73 (13)	N1—C3—H3B	109.5
C4—Sn1—C6	118.82 (12)	H3A—C3—H3B	109.5
C5—Sn1—N3 ⁱ	87.61 (10)	N1—C3—H3C	109.5
C4—Sn1—N3 ⁱ	88.09 (10)	H3A—C3—H3C	109.5
C6—Sn1—N3 ⁱ	91.88 (11)	H3B—C3—H3C	109.5
C5—Sn1—S1	92.42 (8)	Sn1—C4—H4A	109.5
C4—Sn1—S1	88.86 (8)	Sn1—C4—H4B	109.5
C6—Sn1—S1	91.38 (9)	H4A—C4—H4B	109.5
N3 ⁱ —Sn1—S1	176.33 (6)	Sn1—C4—H4C	109.5
C1—S1—Sn1	97.35 (9)	H4A—C4—H4C	109.5
C2—N1—C1	105.9 (2)	H4B—C4—H4C	109.5
C2—N1—C3	126.5 (2)	Sn1—C5—H5A	109.5
C1—N1—C3	127.5 (2)	Sn1—C5—H5B	109.5
C2—N3—N4	108.6 (2)	H5A—C5—H5B	109.5
C2—N3—Sn1 ⁱⁱ	134.93 (19)	Sn1—C5—H5C	109.5
N4—N3—Sn1 ⁱⁱ	115.70 (17)	H5A—C5—H5C	109.5
C1—N4—N3	106.0 (2)	H5B—C5—H5C	109.5
N4—C1—N1	109.8 (2)	Sn1—C6—H6A	109.5
N4—C1—S1	127.2 (2)	Sn1—C6—H6B	109.5
N1—C1—S1	123.0 (2)	H6A—C6—H6B	109.5
N3—C2—N1	109.7 (2)	Sn1—C6—H6C	109.5
N3—C2—H2	125.2	H6A—C6—H6C	109.5
N1—C2—H2	125.2	H6B—C6—H6C	109.5
C5—Sn1—S1—C1	77.35 (13)	C3—N1—C1—N4	-178.4 (2)
C4—Sn1—S1—C1	-158.29 (12)	C2—N1—C1—S1	-178.03 (19)
C6—Sn1—S1—C1	-39.48 (13)	C3—N1—C1—S1	3.1 (4)
N3 ⁱ —Sn1—S1—C1	167.8 (9)	Sn1—S1—C1—N4	-71.9 (2)
C2—N3—N4—C1	0.7 (3)	Sn1—S1—C1—N1	106.3 (2)
Sn1 ⁱⁱ —N3—N4—C1	171.96 (17)	N4—N3—C2—N1	-0.4 (3)
N3—N4—C1—N1	-0.7 (3)	Sn1 ⁱⁱ —N3—C2—N1	-169.27 (18)

N3—N4—C1—S1	177.73 (19)	C1—N1—C2—N3	0.0 (3)
C2—N1—C1—N4	0.5 (3)	C3—N1—C2—N3	178.8 (2)

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