Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[4-(Dimethylamino)pyridine-κN^1]trimethyl(thiocyanato-κN)tin(IV)

Ezzatollah Najafi,[a] Mostafa M. Amini[a] and Seik Weng Ng[b,c]*

[a]Department of Chemistry, General Campus, Shahid Beheshti University, Tehran 1983963113, Iran, [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and [c]Chemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia
Correspondence e-mail: seikweng@um.edu.my

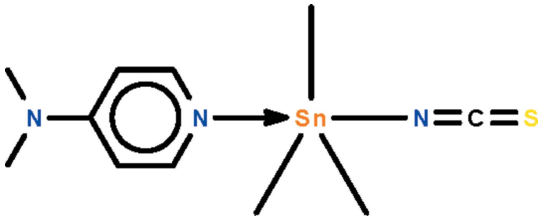
Received 7 May 2012; accepted 16 May 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.060; wR factor = 0.197; data-to-parameter ratio = 36.7.

In the title monomeric trimethyltin(IV) isothiocyanate–4,4-dimethylpyridine adduct, $[Sn(CH_3)_3(NCS)(C_7H_{10}N_2)]$, the Sn^{IV} atom shows a *trans*-C_3SnN_2 trigonal bipyramidal coordination. The Sn^{IV} atom lies out of the equatorial plane by 0.033 (4) Å in the direction of the donor N atom of the *N*-heterocycle. The crystal studied was a non-merohedral twin with a minor component of 48.8 (2)%.

Related literature

For trimethyltin isothiocyanate, see: Forder & Sheldrick (1970).



Experimental

Crystal data

$[Sn(CH_3)_3(NCS)(C_7H_{10}N_2)]$
M_r = 344.04
Monoclinic, $P2_1/c$
a = 7.2026 (4) Å
b = 13.4736 (8) Å
c = 14.9785 (8) Å
β = 93.792 (5)°
V = 1450.41 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.89 mm^{-1}
T = 100 K
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.558, T_{max} = 0.650
15682 measured reflections
5542 independent reflections
4916 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.197
S = 1.23
5542 reflections
151 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.61 e Å^{-3}
$\Delta\rho_{min}$ = −1.98 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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).

We thank Shahid Beheshti University and the Ministry of Higher Education of Malaysia (grant No. UM·C/HIR/MOHE/SC/12) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2162).

References

Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Forder, R. A. & Sheldrick, G. M. (1970). *J. Organomet. Chem.* **21**, 115–122.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, m790 [doi:10.1107/S1600536812022064]

[4-(Dimethylamino)pyridine-κN^1]trimethyl(thiocyanato-κN)tin(IV)

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

S1. Comment

Trimethyltin halides and pseudohalides are Lewis acids that form 1:1 complexes with aromatic amines. Trimethyltin isocyanate itself exists as a polymer in which the isocyanate anion bridges adjacent trimethyltin cations (Forder & Sheldrick, 1970). In the 4,4-dimethylpyridine adduct (Scheme I), the weaker tin–sulfur bond is disrupted, and the adduct is monomeric. The Sn^{IV} atom shows *trans*-C_3SnN_2 trigonal bipyramidal coordination. The tin atom lies out of the equatorial plane by 0.033 (4) Å in the direction of the donor N atom of the *N*-heterocycle.

S2. Experimental

Trimethyltin isothiocyanate (0.24 g, 1 mmol) and 4-(dimethylamino)pyridine (0.11 g, 1 mmol) were loaded into a convection tube and the tube was filled with methanol and kept at 333 K. Light yellow crystals were collected from the side arm after several days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The crystal is a non-merohedral twin having nearly equal components (minor component 48.8 (2) %). A 100% overlap gave the best refinement; however, an artifact of the twinning is the high weighting scheme, which was suggested by the refinement program. The twin law is (-1 0 0 / 0 - 1 0 / 0.2779 0 1).

The final difference Fourier map had a peak 1.08 Å from Sn1 and a hole 0.95 Å from H1a.

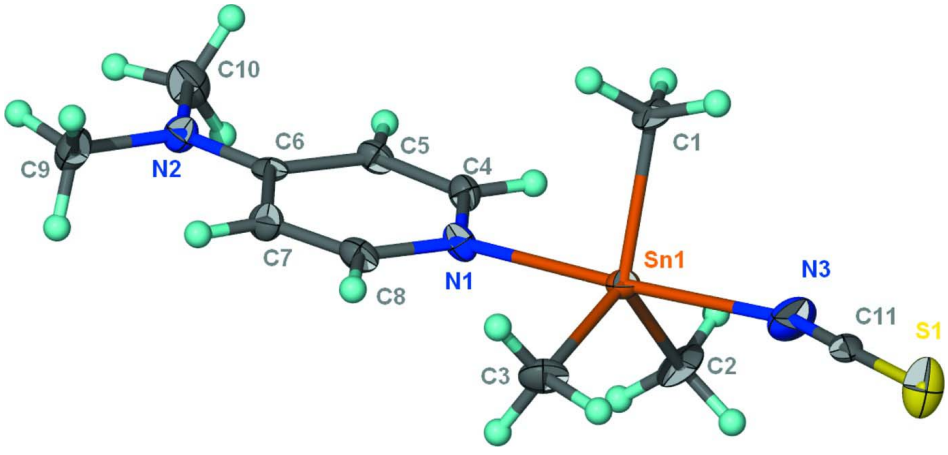


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $(CH_3)_3Sn(NCS)(C_7H_{10}N_2)$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

[4-(Dimethylamino)pyridine-κN^1]trimethyl(thiocyanato-κN)tin(IV)

Crystal data

$[Sn(CH_3)_3(NCS)(C_7H_{10}N_2)]$
$M_r = 344.04$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.2026$ (4) Å
$b = 13.4736$ (8) Å
$c = 14.9785$ (8) Å
$\beta = 93.792$ (5)°
$V = 1450.41$ (14) Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.576$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5713 reflections
$\theta = 2.7$–$27.5°$
$\mu = 1.89$ mm^{-1}
$T = 100$ K
Prism, light brown
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.558$, $T_{max} = 0.650$
15682 measured reflections
5542 independent reflections
4916 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 27.7°$, $\theta_{min} = 2.7°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.197$
$S = 1.23$
5542 reflections
151 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
$w = 1/[\sigma^2(F_o^2) + (0.1375P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.61$ e Å^{-3}
$\Delta\rho_{min} = -1.98$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.25215 (5)	0.41625 (3)	0.86069 (2)	0.01334 (16)
S1	0.3130 (2)	0.35874 (10)	1.18988 (10)	0.0267 (4)
N1	0.2370 (6)	0.4769 (3)	0.7145 (3)	0.0145 (9)
N2	0.2613 (7)	0.5696 (3)	0.4491 (3)	0.0160 (10)
N3	0.2743 (7)	0.3468 (4)	1.0044 (3)	0.0298 (12)
C1	0.5262 (7)	0.3660 (4)	0.8426 (4)	0.0176 (11)
H1A	0.6000	0.4208	0.8204	0.026*
H1B	0.5216	0.3116	0.7991	0.026*
H1C	0.5835	0.3425	0.8999	0.026*
C2	0.0214 (8)	0.3222 (5)	0.8288 (4)	0.0247 (13)
H2A	−0.0670	0.3561	0.7866	0.037*
H2B	−0.0398	0.3060	0.8835	0.037*
H2C	0.0644	0.2609	0.8015	0.037*

C3	0.2037 (8)	0.5581 (4)	0.9169 (4)	0.0219 (12)
H3A	0.0925	0.5878	0.8865	0.033*
H3B	0.3113	0.6011	0.9092	0.033*
H3C	0.1851	0.5509	0.9807	0.033*
C4	0.2356 (7)	0.4114 (4)	0.6457 (3)	0.0146 (11)
H4	0.2297	0.3427	0.6592	0.018*
C5	0.2421 (8)	0.4383 (4)	0.5584 (4)	0.0151 (11)
H5	0.2408	0.3884	0.5135	0.018*
C6	0.2507 (7)	0.5397 (3)	0.5334 (3)	0.0118 (10)
C7	0.2515 (8)	0.6078 (4)	0.6063 (4)	0.0159 (11)
H7	0.2566	0.6771	0.5952	0.019*
C8	0.2448 (8)	0.5743 (3)	0.6913 (4)	0.0152 (11)
H8	0.2457	0.6221	0.7379	0.018*
C9	0.2689 (9)	0.6754 (4)	0.4273 (4)	0.0232 (12)
H9A	0.3785	0.7053	0.4590	0.035*
H9B	0.1563	0.7083	0.4458	0.035*
H9C	0.2771	0.6834	0.3627	0.035*
C10	0.2531 (8)	0.4977 (5)	0.3755 (4)	0.0224 (12)
H10A	0.3642	0.4556	0.3805	0.034*
H10B	0.2477	0.5331	0.3183	0.034*
H10C	0.1418	0.4563	0.3786	0.034*
C11	0.2906 (7)	0.3530 (4)	1.0821 (4)	0.0171 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0109 (2)	0.0157 (2)	0.0135 (2)	0.00108 (13)	0.00189 (17)	0.00061 (12)
S1	0.0435 (9)	0.0206 (7)	0.0158 (7)	−0.0071 (7)	−0.0002 (7)	−0.0019 (5)
N1	0.017 (2)	0.0109 (19)	0.015 (2)	0.0021 (17)	−0.0028 (19)	−0.0013 (16)
N2	0.020 (3)	0.014 (2)	0.014 (2)	−0.0016 (18)	0.001 (2)	0.0002 (16)
N3	0.031 (3)	0.042 (3)	0.017 (3)	0.004 (3)	0.009 (2)	0.003 (2)
C1	0.004 (2)	0.028 (3)	0.020 (3)	−0.003 (2)	−0.001 (2)	0.006 (2)
C2	0.014 (3)	0.030 (3)	0.030 (3)	−0.015 (2)	0.003 (2)	0.004 (3)
C3	0.021 (3)	0.024 (3)	0.022 (3)	0.002 (2)	0.008 (3)	−0.006 (2)
C4	0.016 (3)	0.011 (2)	0.017 (3)	0.0000 (18)	0.000 (3)	0.0000 (18)
C5	0.015 (3)	0.013 (2)	0.017 (3)	0.002 (2)	0.001 (2)	−0.0027 (19)
C6	0.006 (2)	0.013 (2)	0.016 (2)	−0.0007 (18)	0.001 (2)	−0.0023 (19)
C7	0.016 (3)	0.012 (2)	0.019 (3)	−0.003 (2)	0.002 (2)	0.000 (2)
C8	0.015 (3)	0.012 (2)	0.019 (3)	0.0017 (19)	0.000 (2)	−0.0031 (18)
C9	0.032 (3)	0.020 (3)	0.019 (3)	−0.003 (2)	0.008 (3)	0.004 (2)
C10	0.026 (3)	0.027 (3)	0.013 (2)	0.004 (2)	0.000 (2)	−0.001 (2)
C11	0.007 (2)	0.018 (2)	0.027 (3)	0.000 (2)	0.004 (2)	0.008 (2)

Geometric parameters (Å, º)

Sn1—C2	2.120 (5)	C3—H3A	0.9800
Sn1—C1	2.121 (5)	C3—H3B	0.9800
Sn1—C3	2.126 (5)	C3—H3C	0.9800

Sn1—N1	2.333 (4)	C4—C5	1.360 (7)
Sn1—N3	2.344 (5)	C4—H4	0.9500
S1—C11	1.614 (6)	C5—C6	1.419 (6)
N1—C4	1.357 (6)	C5—H5	0.9500
N1—C8	1.359 (6)	C6—C7	1.426 (7)
N2—C6	1.333 (6)	C7—C8	1.354 (8)
N2—C9	1.464 (6)	C7—H7	0.9500
N2—C10	1.466 (7)	C8—H8	0.9500
N3—C11	1.164 (7)	C9—H9A	0.9800
C1—H1A	0.9800	C9—H9B	0.9800
C1—H1B	0.9800	C9—H9C	0.9800
C1—H1C	0.9800	C10—H10A	0.9800
C2—H2A	0.9800	C10—H10B	0.9800
C2—H2B	0.9800	C10—H10C	0.9800
C2—H2C	0.9800		

C2—Sn1—C1	120.2 (2)	Sn1—C3—H3C	109.5
C2—Sn1—C3	118.7 (2)	H3A—C3—H3C	109.5
C1—Sn1—C3	121.1 (2)	H3B—C3—H3C	109.5
C2—Sn1—N1	90.6 (2)	N1—C4—C5	124.0 (4)
C1—Sn1—N1	88.74 (18)	N1—C4—H4	118.0
C3—Sn1—N1	93.30 (18)	C5—C4—H4	118.0
C2—Sn1—N3	88.5 (2)	C4—C5—C6	120.9 (5)
C1—Sn1—N3	89.0 (2)	C4—C5—H5	119.5
C3—Sn1—N3	89.9 (2)	C6—C5—H5	119.5
N1—Sn1—N3	176.70 (16)	N2—C6—C5	123.2 (4)
C4—N1—C8	115.5 (5)	N2—C6—C7	122.2 (5)
C4—N1—Sn1	118.9 (3)	C5—C6—C7	114.6 (5)
C8—N1—Sn1	125.4 (3)	C8—C7—C6	120.4 (5)
C6—N2—C9	120.7 (4)	C8—C7—H7	119.8
C6—N2—C10	120.7 (4)	C6—C7—H7	119.8
C9—N2—C10	118.4 (5)	C7—C8—N1	124.6 (5)
C11—N3—Sn1	152.3 (5)	C7—C8—H8	117.7
Sn1—C1—H1A	109.5	N1—C8—H8	117.7
Sn1—C1—H1B	109.5	N2—C9—H9A	109.5
H1A—C1—H1B	109.5	N2—C9—H9B	109.5
Sn1—C1—H1C	109.5	H9A—C9—H9B	109.5
H1A—C1—H1C	109.5	N2—C9—H9C	109.5
H1B—C1—H1C	109.5	H9A—C9—H9C	109.5
Sn1—C2—H2A	109.5	H9B—C9—H9C	109.5
Sn1—C2—H2B	109.5	N2—C10—H10A	109.5
H2A—C2—H2B	109.5	N2—C10—H10B	109.5
Sn1—C2—H2C	109.5	H10A—C10—H10B	109.5
H2A—C2—H2C	109.5	N2—C10—H10C	109.5
H2B—C2—H2C	109.5	H10A—C10—H10C	109.5
Sn1—C3—H3A	109.5	H10B—C10—H10C	109.5
Sn1—C3—H3B	109.5	N3—C11—S1	178.7 (5)
H3A—C3—H3B	109.5		

C2—Sn1—N1—C4	−52.6 (4)	C9—N2—C6—C5	179.7 (5)
C1—Sn1—N1—C4	67.6 (4)	C10—N2—C6—C5	3.8 (8)
C3—Sn1—N1—C4	−171.4 (4)	C9—N2—C6—C7	−1.8 (8)
C2—Sn1—N1—C8	133.2 (5)	C10—N2—C6—C7	−177.7 (5)
C1—Sn1—N1—C8	−106.6 (5)	C4—C5—C6—N2	178.5 (5)
C3—Sn1—N1—C8	14.4 (5)	C4—C5—C6—C7	−0.1 (8)
C2—Sn1—N3—C11	−133.1 (10)	N2—C6—C7—C8	−178.4 (5)
C1—Sn1—N3—C11	106.7 (10)	C5—C6—C7—C8	0.2 (8)
C3—Sn1—N3—C11	−14.4 (10)	C6—C7—C8—N1	−0.1 (9)
C8—N1—C4—C5	0.3 (8)	C4—N1—C8—C7	−0.2 (8)
Sn1—N1—C4—C5	−174.5 (4)	Sn1—N1—C8—C7	174.2 (5)
N1—C4—C5—C6	−0.1 (9)		