Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(4-phenylpyridinium) tetrakis(nitrato-κ^2O,O')stannate(IV). Retraction

H. Zhong,[a]* X.-R. Zeng,[a] X.-M. Yang[b] and Q.-Y. Luo[a]

[a]College of Chemistry and Chemical Engineering, Provincial Key Laboratory of Coordination Chemistry, Jinggangshan University, Jian 343009, People's Republic of China, and [b]Institute of Applied Materials, Jiangxi University of Finance and Economics, Nanchang 330032, People's Republic of China
Correspondence e-mail: huazhong06@126.com

Received 25 September 2008; accepted 26 September 2008

The paper by Zhong, Zeng, Yang & Luo [*Acta Cryst.* (2007), E**63**, m1567] is retracted.

Due to problems with the structural model involving unrealistically short intermolecular contacts and an uncertain atomic composition, the paper by Zhong, Zeng, Yang & Luo [*Acta Cryst.* (2007), E**63**, m1567] is retracted.

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis(4-phenylpyridinium) tetrakis(nitrato-κ^2O,O')stannate(IV)

H. Zhong,[a]* X.-R. Zeng,[a] X.-M. Yang[b] and Q.-Y. Luo[a]

[a]College of Chemistry and Chemical Engineering, Provincial Key Laboratory of Coordination Chemistry, Jinggangshan University, Jian 343009, People's Republic of China, and [b]Institute of Applied Materials, Jiangxi University of Finance and Economics, Nanchang 330032, People's Republic of China
Correspondence e-mail: huazhong06@126.com

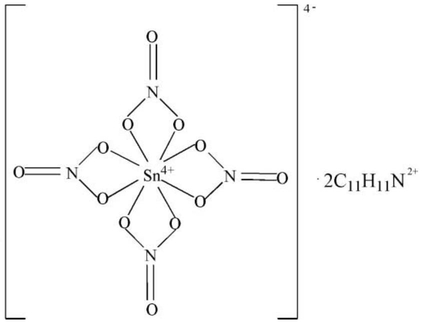
Received 14 April 2007; accepted 27 April 2007

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.011 Å; R factor = 0.035; wR factor = 0.123; data-to-parameter ratio = 13.3.

The asymmetric unit of the title compound, $(C_{11}H_9N)_2$-$[Sn(NO_3)_4]$, consists of a mononuclear complex anion and two non-coordinated 4-phenylpyridinium cations. The Sn^{IV} atom, lying on a twofold rotation axis, is coordinated by eight O atoms of four NO_3^- anions. This mononuclear complex is further extended into a supramolecular network structure *via* non-classical hydrogen bonds between CH groups of cations and O atoms of neighbouring anions.

Related literature

For related literature, see: Allen *et al.* (1987); Bandoli *et al.* (1992, 1993); Barone *et al.* (2002); Hencher *et al.* (1982); Jiang & Ozin (1998); Nair & Nair (1991); Valiukonis *et al.* (1986).



Experimental

Crystal data

$(C_{11}H_9N)_2[Sn(NO_3)_4]$
M_r = 677.11
Monoclinic, $C2/c$
a = 20.121 (5) Å
b = 7.8112 (12) Å
c = 18.307 (5) Å
β = 118.823 (9)°
V = 2520.8 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.09 mm^{-1}
T = 273 (2) K
0.40 × 0.33 × 0.21 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.656, T_{max} = 0.801
8171 measured reflections
2495 independent reflections
2450 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.123
S = 1.02
2495 reflections
187 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.466 (5)	Sn1—O4	2.552 (5)
Sn1—O3	2.514 (5)	Sn1—O6	2.485 (5)
O1—Sn1—O3	51.33 (15)	O6—Sn1—O4	50.34 (15)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O6	0.93	2.38	3.032 (8)	127
C1—H1···O1	0.93	2.74	3.100 (8)	104

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Science and Technology Bureau of Jian, Jiangxi Province of China (grant No. 20052817).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2015).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bandoli, G., Dolmella, A., Peruzzo, V. & Plazzogna, G. (1992). *Inorg. Chim. Acta*, **193**, 185–190.
Bandoli, G., Dolmella, A., Peruzzo, V. & Plazzogna, G. (1993). *Inorg. Chim. Acta*, **209**, 231–234.
Barone, G., Chaplin, T., Hibbert, T. G., Kana, A. T., Mahon, M. F., Molloy, K. C., Worsley, I. D., Parkin, I. P. & Price, L. S. (2002). *J. Chem. Soc. Dalton Trans.* pp. 1085–1093.
Hencher, J. L., Khan, M., Said, F. F., Sieler, R. & Tuck, D. G. (1982). *Inorg. Chem.* **21**, 2787–2791.
Jiang, T. & Ozin, G. A. (1998). *J. Mater. Chem.* **8**, 1099–1109.
Nair, P. K. & Nair, M. T. S. (1991). *J. Phys. D Appl. Phys.* **24**, 83–87.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SMART*, *SAINT* and *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Valiukonis, G., Guseinvoa, D., Krivate, A. G. & Silecia, A. (1986). *Phys. Status Solidi B*, **135**, 299–307.

doi:10.1107/S1600536807021174

supplementary materials

Acta Cryst. (2007). E**63**, m1567 [**doi:10.1107/S1600536807021174**]

Bis(4-phenylpyridinium) tetrakis(nitrato-κ^2O,O')stannate(IV)

H. Zhong, X.-R. Zeng, X.-M. Yang and Q.-Y. Luo

Comment

In recent years, the researches on tin complexes draw increasing attention owning to their potential applications as photovoltaic materials, holographic recording system and biological activities (Jiang & Ozin, 1998; Valiukonis *et al.*, 1986; Hencher *et al.*, 1982; Bandoli *et al.*, 1992, 1993), solar control devices (Nair & Nair, 1991) and semiconductor materials. Mononuclear or binuclear tin materials are important candidates as molecular precursors to prepare tin film materials by chemical vapor deposition (CVD)(Barone *et al.*, 2002). We report herein the crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The eight-coordinate environment of the Sn atom is completed by the eight O atoms of four NO_3^- (Table 1). The Sn—O bond lengths are in the range 2.466 (5) to 2.552 (5) Å. Hydrogen bonds

between C—H groups of free 4-phenylpyridinium and O atoms of neighboring molecules, with C···O distances of 3.032 (8) and 3.100 (8), generate a layered hydrogen-bonded network (Fig. 2 and Table 2). The non-classical hydrogen-bonding interactions link the mononuclear complex into a supramolecular network structure.

Experimental

Crystals of the title compound were synthesized using hydrothermal method in a 23 ml Teflon-lined Parr bomb, which was then sealed. Tin dioxide (30.1 mg, 0.2 mmol), 4-phenylpyridinium (62.8 mg, 0.4 mmol), nitric acid (0.2 mol/l, 4 ml) and ethanol (5 ml) were placed into the bomb and sealed. The bomb was then heated under autogenous pressure for 7 d at 413 K and allowed to cool at room temperature for 24 h. Upon opening the bomb, a clear colorless solution was decanted from small colorless crystals. These crystals were washed with distilled water followed by ethanol, and allowed to air-dry at room temperature. Powder X-ray diffraction was conducted on the sample.

Refinement

H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

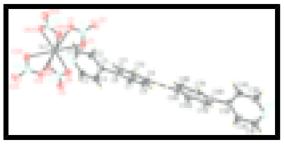


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms labeled with the suffix A and B are generated by the symmetry operation ($-x + 2$, y, $-z + 3/2$) and ($-x + 3/2$, $-y + 5/2$,$-z + 2$), respectively.

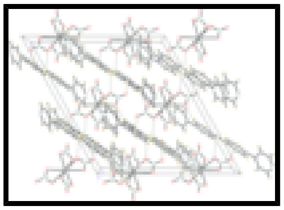


Fig. 2. A packing diagram of (I), H-bonds with dashed lines.

Bis(4-phenylpyridinium) tetrakis(nitrato-κ^2*O,O*')tin(IV)

Crystal data

$(C_{11}H_9N)_2[Sn(NO_3)_4]$	F_{000} = 1352
M_r = 677.11	D_x = 1.784 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 8235 reflections
a = 20.121 (5) Å	θ = 2.5–29.5°
b = 7.8112 (12) Å	μ = 1.09 mm^{-1}
c = 18.307 (5) Å	*T* = 273 (2) K
β = 118.823 (9)°	Plane, colourless
V = 2520.8 (10) Å^3	0.40 × 0.33 × 0.21 mm
Z = 4	

Data collection

Bruker APEXII area-detector diffractometer	2495 independent reflections
Radiation source: fine-focus sealed tube	2450 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\rm int}$ = 0.014
T = 273(2) K	$\theta_{\rm max}$ = 26.4°
φ and ω scans	$\theta_{\rm min}$ = 2.3°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -24 \rightarrow 25$
$T_{\rm min}$ = 0.656, $T_{\rm max}$ = 0.801	$k = -9 \rightarrow 9$
8171 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 16.0815P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.123	$(\Delta/\sigma)_{\rm max} < 0.001$
S = 1.02	$\Delta\rho_{\rm max}$ = 0.88 e Å^{-3}
2495 reflections	$\Delta\rho_{\rm min}$ = −0.61 e Å^{-3}

187 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0034 (4)
Secondary atom site location: difference Fourier map	

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}
Sn1	1.0000	0.33855 (4)	0.7500	0.04796 (19)
O1	1.0921 (3)	0.4420 (6)	0.8900 (3)	0.0671 (12)
O2	1.0826 (4)	0.4129 (8)	1.0028 (3)	0.0948 (18)
O3	0.9970 (3)	0.3046 (6)	0.8849 (3)	0.0675 (12)
O4	0.9294 (3)	0.0724 (7)	0.6688 (4)	0.0773 (14)
O5	0.8183 (3)	−0.0046 (7)	0.6531 (4)	0.0850 (15)
O6	0.8782 (3)	0.2146 (6)	0.7289 (3)	0.0673 (12)
N1	1.0582 (3)	0.3864 (7)	0.9290 (3)	0.0609 (13)
N2	0.8730 (3)	0.0901 (7)	0.6818 (3)	0.0572 (12)
N3	0.9363 (3)	0.6016 (7)	0.7803 (3)	0.0571 (12)
C1	0.9745 (4)	0.7370 (8)	0.8257 (4)	0.0579 (14)
H1	1.0235	0.7541	0.8343	0.069*
C2	0.9464 (4)	0.8528 (7)	0.8605 (4)	0.0557 (14)
H2	0.9758	0.9452	0.8910	0.067*
C3	0.8639 (4)	0.5857 (9)	0.7678 (4)	0.0625 (15)
H3	0.8347	0.4953	0.7349	0.075*
C4	0.8316 (4)	0.6929 (8)	0.8002 (4)	0.0582 (14)
H4	0.7821	0.6751	0.7898	0.070*
C5	0.8742 (4)	0.8307 (7)	0.8497 (4)	0.0509 (13)
C6	0.8427 (3)	0.9443 (7)	0.8910 (4)	0.0511 (12)
C7	0.7945 (4)	0.8780 (8)	0.9188 (4)	0.0560 (14)
H7	0.7821	0.7622	0.9127	0.067*
C8	0.7654 (4)	0.9871 (9)	0.9554 (4)	0.0624 (15)
H8	0.7331	0.9433	0.9738	0.075*
C9	0.7820 (4)	1.1524 (7)	0.9653 (4)	0.0508 (13)
H9	0.7616	1.2226	0.9905	0.061*
C10	0.8271 (5)	1.2168 (10)	0.9397 (6)	0.078 (2)
H10	0.8371	1.3336	0.9460	0.093*

C11	0.8606 (5)	1.1178 (9)	0.9033 (5)	0.0704 (19)
H11	0.8941	1.1661	0.8876	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0310 (3)	0.0253 (2)	0.0368 (3)	0.000	0.02356 (19)	0.000
O1	0.062 (3)	0.070 (3)	0.076 (3)	−0.012 (2)	0.038 (2)	0.001 (2)
O2	0.123 (5)	0.095 (4)	0.052 (3)	−0.010 (4)	0.031 (3)	−0.005 (3)
O3	0.070 (3)	0.073 (3)	0.071 (3)	−0.017 (2)	0.043 (2)	−0.005 (2)
O4	0.082 (3)	0.066 (3)	0.104 (4)	−0.002 (3)	0.060 (3)	−0.016 (3)
O5	0.077 (3)	0.076 (3)	0.093 (4)	−0.033 (3)	0.033 (3)	−0.018 (3)
O6	0.074 (3)	0.058 (2)	0.084 (3)	−0.011 (2)	0.051 (3)	−0.019 (2)
N1	0.068 (3)	0.057 (3)	0.058 (3)	−0.001 (3)	0.030 (3)	0.002 (2)
N2	0.061 (3)	0.050 (3)	0.063 (3)	−0.006 (2)	0.032 (2)	−0.005 (2)
N3	0.063 (3)	0.056 (3)	0.065 (3)	0.001 (2)	0.041 (3)	−0.003 (2)
C1	0.062 (4)	0.050 (3)	0.076 (4)	−0.006 (3)	0.046 (3)	−0.006 (3)
C2	0.060 (4)	0.051 (3)	0.069 (4)	−0.007 (2)	0.041 (3)	−0.007 (3)
C3	0.059 (4)	0.063 (4)	0.068 (4)	−0.002 (3)	0.033 (3)	−0.016 (3)
C4	0.053 (3)	0.062 (3)	0.063 (3)	0.000 (3)	0.030 (3)	−0.012 (3)
C5	0.056 (3)	0.051 (3)	0.055 (3)	0.004 (2)	0.034 (3)	0.001 (2)
C6	0.054 (3)	0.051 (3)	0.055 (3)	0.002 (2)	0.032 (3)	−0.002 (2)
C7	0.058 (3)	0.054 (3)	0.066 (3)	−0.003 (3)	0.038 (3)	−0.008 (3)
C8	0.060 (4)	0.073 (4)	0.070 (4)	−0.002 (3)	0.043 (3)	−0.005 (3)
C9	0.056 (3)	0.051 (3)	0.061 (3)	0.007 (2)	0.040 (3)	−0.011 (2)
C10	0.101 (6)	0.053 (4)	0.109 (6)	−0.005 (4)	0.074 (5)	−0.015 (4)
C11	0.093 (5)	0.052 (3)	0.100 (5)	−0.008 (3)	0.073 (5)	−0.013 (3)

Geometric parameters (Å, °)

Sn1—O1	2.466 (5)	C2—C5	1.379 (9)
Sn1—O3	2.514 (5)	C2—H2	0.9300
Sn1—O4	2.552 (5)	C3—C4	1.359 (9)
Sn1—O6	2.485 (5)	C3—H3	0.9300
Sn1—O1^{i}	2.466 (5)	C4—C5	1.402 (9)
Sn1—O6^{i}	2.485 (5)	C4—H4	0.9300
Sn1—O3^{i}	2.514 (5)	C5—C6	1.490 (8)
Sn1—O4^{i}	2.552 (5)	C6—C11	1.393 (9)
O1—N1	1.278 (7)	C6—C7	1.395 (8)
O2—N1	1.213 (7)	C7—C8	1.377 (8)
O3—N1	1.271 (7)	C7—H7	0.9300
O4—N2	1.276 (7)	C8—C9	1.324 (9)
O5—N2	1.215 (7)	C8—H8	0.9300
O6—N2	1.269 (7)	C9—C10	1.306 (10)
N3—C1	1.336 (8)	C9—H9	0.9300
N3—C3	1.367 (8)	C10—C11	1.390 (9)
C1—C2	1.374 (8)	C10—H10	0.9300
C1—H1	0.9300	C11—H11	0.9300

O1—Sn1—O3	51.33 (15)	O5—N2—O4	123.7 (6)
O1—Sn1—O4	143.24 (17)	O6—N2—O4	114.8 (5)
O1—Sn1—O6	118.72 (15)	C1—N3—C3	115.0 (5)
O3—Sn1—O4	102.50 (16)	N3—C1—C2	124.6 (6)
O6—Sn1—O3	67.87 (16)	N3—C1—H1	117.7
O6—Sn1—O4	50.34 (15)	C2—C1—H1	117.7
O1^{i}—Sn1—O6	77.00 (17)	C1—C2—C5	119.4 (6)
O6^{i}—Sn1—O6	134.1 (2)	C1—C2—H2	120.3
O1^{i}—Sn1—O3	133.95 (16)	C5—C2—H2	120.3
O6^{i}—Sn1—O3	107.13 (17)	C4—C3—N3	124.5 (6)
O1—Sn1—O3^{i}	133.95 (16)	C4—C3—H3	117.8
O1^{i}—Sn1—O3^{i}	51.33 (15)	N3—C3—H3	117.8
O6^{i}—Sn1—O3^{i}	67.86 (16)	C3—C4—C5	118.9 (6)
O6—Sn1—O3^{i}	107.13 (17)	C3—C4—H4	120.6
O3—Sn1—O3^{i}	167.9 (2)	C5—C4—H4	120.6
O1—Sn1—O4^{i}	74.51 (18)	C2—C5—C4	117.5 (5)
O1^{i}—Sn1—O4^{i}	143.24 (18)	C2—C5—C6	122.0 (5)
O6^{i}—Sn1—O4^{i}	50.34 (15)	C4—C5—C6	120.5 (6)
O6—Sn1—O4^{i}	90.19 (17)	C11—C6—C7	118.3 (6)
O3—Sn1—O4^{i}	67.15 (17)	C11—C6—C5	121.4 (5)
O3^{i}—Sn1—O4^{i}	102.50 (16)	C7—C6—C5	120.3 (5)
O1^{i}—Sn1—O4	74.51 (18)	C8—C7—C6	118.8 (6)
O6^{i}—Sn1—O4	90.19 (17)	C8—C7—H7	120.6
O1—Sn1—O1^{i}	141.7 (2)	C6—C7—H7	120.6
O1—Sn1—O6^{i}	77.00 (17)	C9—C8—C7	122.1 (6)
O1^{i}—Sn1—O6^{i}	118.72 (15)	C9—C8—H8	118.9
O3^{i}—Sn1—O4	67.15 (17)	C7—C8—H8	118.9
O4^{i}—Sn1—O4	70.9 (3)	C10—C9—C8	119.9 (6)
N1—O1—Sn1	97.6 (4)	C10—C9—H9	120.0
N1—O3—Sn1	95.5 (3)	C8—C9—H9	120.0
N2—O4—Sn1	95.6 (3)	C9—C10—C11	122.7 (7)
N2—O6—Sn1	99.1 (3)	C9—C10—H10	118.6
O2—N1—O3	122.7 (6)	C11—C10—H10	118.6
O2—N1—O1	121.7 (6)	C10—C11—C6	118.1 (6)
O3—N1—O1	115.6 (5)	C10—C11—H11	120.9
O5—N2—O6	121.5 (6)	C6—C11—H11	120.9

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O6	0.93	2.38	3.032 (8)	127
C1—H1···O1	0.93	2.74	3.100 (8)	104

Fig. 1

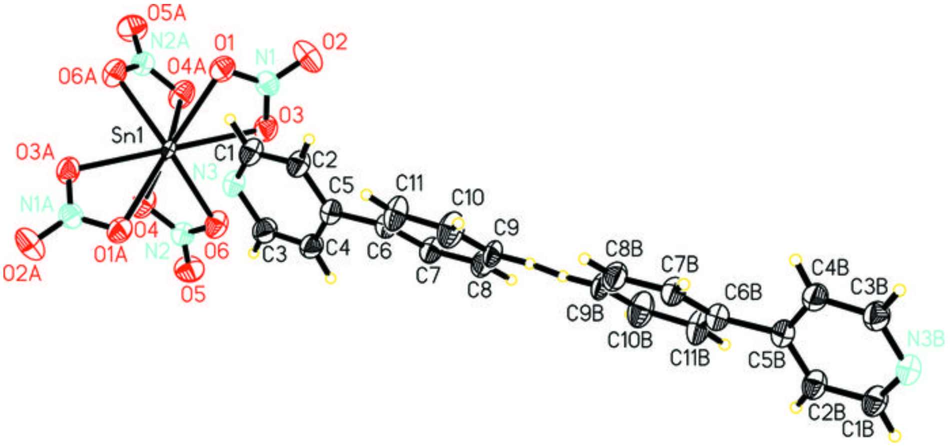

O5A
N2A
O1
N1
O2
O6A
O4A
O3
Sn1
O3A
C1
C2
N3
N1A
C5
C11
C10
C9
O4
O1A
N2
O6
C3
C4
C6
C7
C8
O2A
O5
C8B
C7B
C4B
C3B
C6B
N3B
C9B
C10B
C11B
C5B
C2B
C1B

Fig. 2