

$$w = 1/\sigma^2(F_o)$$

$$(\Delta/\sigma)_{\max} = 0.0240$$

Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)*

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Zn	0	0.1304 (1)	1/4	0.0478 (5)
O1	-0.0828 (4)	-0.1128 (7)	0.1453 (4)	0.051 (1)
O2	0.1043 (3)	0.4319 (9)	0.2606 (3)	0.055 (1)
O3	0.0695 (3)	0.2158 (9)	0.0933 (3)	0.052 (1)
C1	0.1147 (4)	0.3940 (10)	0.1503 (6)	0.043 (2)
C2	0.1733 (5)	0.570 (1)	0.0880 (6)	0.054 (2)

Table 2. *Selected geometric parameters (\AA , $^\circ$)*

Zn—O1	1.987 (4)	O2—C1	1.260 (7)
Zn—O2	2.189 (5)	O3—C1	1.256 (7)
Zn—O3	2.179 (4)	C1—C2	1.503 (8)
O1—Zn—O1'	98.6 (3)	O2—Zn—O3	59.0 (1)
O1—Zn—O2	147.9 (2)	O3—Zn—O3'	155.9 (3)
O1—Zn—O2'	96.3 (2)	Zn—O2—C1	91.4 (4)
O1—Zn—O3	89.5 (2)	Zn—O3—C1	92.0 (3)
O1—Zn—O3'	106.3 (2)	O2—C1—O3	117.5 (5)
O2—Zn—O2'	85.5 (2)	O2—C1—C2	120.3 (6)
O2—Zn—O3'	102.0 (2)	O3—C1—C2	122.0 (5)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

H atoms were fixed at ideal positions with common isotropic displacement parameters ($U_{\text{iso}} = 0.0657 \text{\AA}^2$).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992a). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992b). Program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1020). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1031–1034

{Tris[*N*-(salicylidene)-2-aminoethyl]amine}tin(IV) Iodide Acetonitrile Solvate†

SUZANNE WATSON,^a WILLIAM ERRINGTON,^a DAVID FENN,^b PETER MOORE^a AND MALCOLM G. H. WALLBRIDGE^a

^aDepartment of Chemistry, University of Warwick, Coventry CV4 7AL, England, and ^bICI Paints, Slough, Berkshire SL2 5DS, England. E-mail: msrpq@csv.warwick.ac.uk

(Received 14 August 1996; accepted 7 March 1997)

Abstract

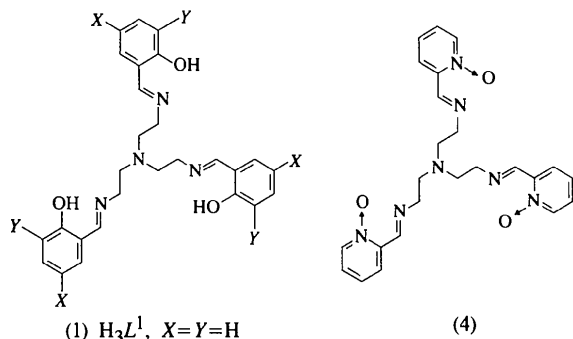
The crystal structure of the title compound, $[\text{Sn}(\text{C}_{27}\text{H}_{27}\text{N}_4\text{O}_3)]\text{I} \cdot 0.5\text{CH}_3\text{CN}$, involves two almost identical complex cations in the asymmetric unit. These cations contain Sn atoms in a pseudo-octahedral environment with facial coordination of the azomethine N and the phenolate O atoms. The average Sn—O and Sn—N bond lengths are 2.048 (4) and 2.192 (5) Å, respectively, while the apical N atom in the ligand is considered to be non-bonded at an average distance of 2.687 (5) Å from the Sn atom.

Comment

The crystal structure of the potentially heptadentate N_4O_3 Schiff base ligand derived from three moles of salicylaldehyde and tris(2-aminoethyl)amine (tren), known as (sal)₃tren [H_3L^1 , (1)], has been reported (Gunduz *et al.*, 1985). Metal complexes of the type $[\text{M}(\text{L}^1)]$ formed from the reaction of H_3L^1 with tripositive metal ions (M^{3+}) have been found to be either six coordinate with the apical N atom uncoordinated when M^{3+} is Co^{III} (Elerman, Kabak, Svoboda & Geselle, 1994) and Mn^{III} (Drew, Harding, McKee, Morgan & Nelson, 1995; Chandra & Chakravorty, 1991), or seven coordinate with all four N atoms bonded when M^{3+} is Bi^{III} (Bharadwaj, Lee, Mandal, Skelton & White,

† Alternative name: {2,2',2''-[nitriilotris(ethylenenitriolomethylidyn-κN)]triphenolato-κ³O}tin(IV) iodide 0.5-acetonitrile solvate.

1994). Six-coordinate complexes are reported to form with the closely related ligands tris[*N*-(5-chlorosalicylidene)-2-aminoethyl]amine [H₃L², (2)] and tris[*N*-(3-methoxysalicylidene)-2-aminoethyl]amine [H₃L³, (3)]. Three structures of [M(L²)] were found to be isomorphous when M³⁺ is Fe^{III}, Cr^{III} and Mn^{III} (Bailey, Cook, Cummins & McKenzie, 1975; Cook, Cummins & McKenzie, 1976; Alcock, Cook, McKenzie & Worthington, 1980; Chandra, Chakraborty & Chakravorty, 1993), and the structure of [Mn(L³)] (Aguari *et al.*, 1994) is very similar to those of [Mn(L¹)] and [Mn(L²)]. In all cases, except the complex of (1) formed with Bi^{III}, the apical N atom of compounds (1)–(3) has been found to be non-coordinating and six-coordinate *fac* complexes are formed by coordination of the three azomethine N atoms and the three deprotonated phenolate groups. This is in contrast to the closely analogous heptadentate N₄O₃ ligand, (4), which is derived from the reaction of three moles of 2-pyridinecarboxaldehyde *N*-oxide with one mole of tren. Compound (4) has been found to form seven-coordinate isostructural complexes with Mn^{II} and Co^{II}, with bonding to all four of the ligand N atoms and the three O atoms of the pyridine *N*-oxide groups (Gou, You, Yu & Lu, 1993). The different coordination modes of (1) and (4) are reported to arise from (4) having the azomethine N atoms spread further apart, allowing easier coordination of the apical N atom, although the cause of this effect is not obvious.



In an attempt to isolate cationic Sn^{IV} complexes, (1) was refluxed with an equimolar amount of SnL₄ in ethanol containing three mole equivalents of triethylamine. The product, [Sn(L¹)]I, was recrystallized from acetonitrile and characterized by ¹H and ¹³C NMR, CHN analysis and a FAB mass spectrum. Its X-ray structure was determined to provide definitive information about the Sn^{IV} coordination sites.

The asymmetric unit contains two almost identical cationic Sn^{IV} complexes, one of which is given in Fig. 1, while the root-mean overlay of the two complexes is shown in Fig. 2. In one of the two complexes, the cal-

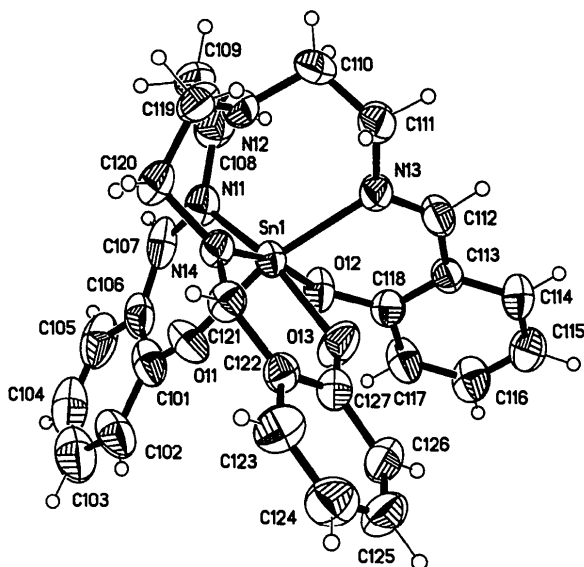


Fig. 1. View of the molecule showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

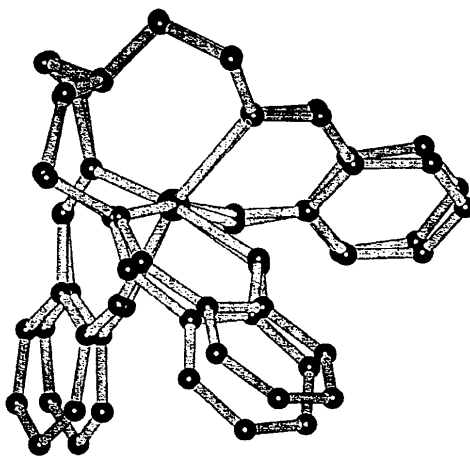


Fig. 2. Root-mean-square overlay of the two [Sn(L¹)]⁺ ions found in the asymmetric unit.

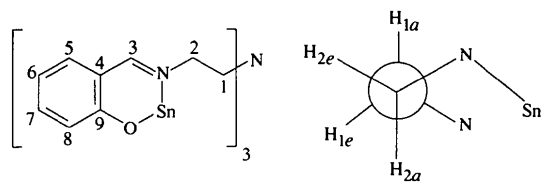


Fig. 3. Atomic numbering scheme used for the assignment of the NMR data.

culated dihedral angles H_{1a}—C—C—H_{2a} (Fig. 3) of the three ligating arms are very similar and average 177.6(6)°; in the second complex, two of these angles are very similar to those found in the first complex [average 177.1(6)°], but the third angle is somewhat

smaller [174.0 (6)°]). Application of the Karplus equation to the coupling constant ${}^3J_{\text{H}1a,\text{H}2a}$ found in solution gives a calculated $\text{H}_{1a}-\text{C}-\text{C}-\text{H}_{2a}$ dihedral angle of 179°. It appears, therefore, that the conformation in solution is very similar to that observed in the solid state.

The apical N atom is essentially non-bonding in both cations, with Sn1—N12 and Sn2—N22 distances of 2.661 (5) and 2.712 (5) Å, respectively. These values compare with the much shorter Sn1—N and Sn2—N distances to the three azomethine N atoms which average 2.192 (5) Å. The bond lengths for the ligand L^1 are unexceptional and are very similar to those reported previously in its complexes with Co^{III} , Mn^{III} and Bi^{III} .

Experimental

The ligand (sal)₃tren (H_3L^1) was prepared according to the published method (Broomhead & Robinson, 1968). To a solution of SnI₄ (0.750 g, 1.2 mmol) in ethanol (100 ml) was added H_3L^1 (0.565 g, 1.2 mmol) and triethylamine (0.354 g, 3.6 mmol). The solution was heated under reflux for 24 h and upon cooling the product separated as a bright yellow solid (yield 0.768 g, 91%). Needle-like single crystals of formula $\text{C}_{27}\text{H}_{27}\text{N}_4\text{O}_3\text{SnI}_4 \cdot 0.5\text{CH}_3\text{CN}$ were grown in a 5 mm NMR tube at the interface between an acetonitrile solution with an upper layer of diethyl ether. Analysis found: C 46.0, H 3.9, N 8.4%; calculated for $\text{C}_{28}\text{H}_{28.5}\text{N}_{4.5}\text{O}_3\text{SnI}_4$: C 46.5, H 4.0, N 8.7%. FAB MS (in NBA): m/z 703 (calculated for $[\text{C}_{27}\text{H}_{28}\text{N}_4\text{O}_3\text{SnI}]^+$ 703) and 576 (calculated for $[\text{C}_{27}\text{H}_{27}\text{N}_4\text{O}_3\text{Sn}]^+$ 576). NMR in $[\text{H}^1]_6$ -DMSO (with assignments as shown in Fig. 3); values in square parentheses refer to uncomplexed H_3L^1 in $[\text{H}^2]_4$ -methanol (Gunduz *et al.*, 1985); ${}^1\text{H}$ (400.1 MHz): δ (p.p.m.) 3.10 (H_{1a} , ${}^2J_{\text{H}1a,\text{H}1e} = -13.2$, ${}^3J_{\text{H}1a,\text{H}2a} = 12.6$, ${}^3J_{\text{H}1a,\text{H}2e} = 2.9$ Hz), 3.19 (H_{1e} , ${}^3J_{\text{H}1e,\text{H}2a} = 2.7$ Hz) [2.87], 3.67 (H_{2e} , ${}^2J_{\text{H}2a,\text{H}2e} = -12.9$, ${}^3J_{\text{H}2e,\text{H}1e} < 1$, ${}^3J_{\text{Sn}-\text{H}2e} = 47.8$, ${}^3J_{\text{Sn}-\text{H}2a} < 1$ Hz), 3.85 (H_{2a}) [3.57], 6.40 (H_8 , d , 3 H, ${}^3J_{\text{H}-\text{H}} = 8.4$ Hz) [6.23], 6.83 (H_6 , ddd , 3 H, ${}^3J_{\text{H}-\text{H}} = 7.4$, ${}^4J_{\text{H}-\text{H}} = 0.8$ Hz) [6.54], 7.39 (H_7 , ddd , 3 H, ${}^3J_{\text{H}-\text{H}} = 7.8$, ${}^4J_{\text{H}-\text{H}} = 1.7$, ${}^5J_{\text{Sn}-\text{H}} = 12.3$ Hz) [6.83], 7.44 (H_5 , dd , 3 H, ${}^3J_{\text{H}-\text{H}} = 7.8$, ${}^4J_{\text{H}-\text{H}} = 1.7$ Hz) [7.27], 8.86 [H_3 , s , 3 H, ${}^3J(^{117}\text{Sn}-\text{H}) = 58.4$, ${}^3J(^{119}\text{Sn}-\text{H}) = 60.8$ Hz] [7.88]; ${}^{13}\text{C}$ (100.6 MHz): δ (p.p.m.) 55.58 (C2) [56.8], 55.77 (C1, ${}^3J_{\text{Sn}-\text{C}} = 25.5$ Hz) [57.8], 117.37 (C4, ${}^3J_{\text{Sn}-\text{C}} = 65.5$ Hz) [119.5], 117.44 (C6) [134.0], 121.41 (C8, ${}^3J_{\text{Sn}-\text{C}} = 35.6$ Hz) [133.5], 135.82 (C5) [118.6], 136.58 (C7) [119.1], 167.34 (C9, ${}^2J_{\text{Sn}-\text{C}} = 40.1$ Hz) [164.9], 174.97 (C3) [167.6].

Crystal data

$[\text{Sn}(\text{C}_{27}\text{H}_{27}\text{N}_4\text{O}_3)] \cdot 0.5\text{CH}_3\text{CN}$

$M_r = 721.64$

Monoclinic

$P2_1/c$

$a = 21.45870$ (10) Å

$b = 14.9163$ (3) Å

$c = 17.8510$ (3) Å

$\beta = 95.1180$ (10)°

$V = 5691.0$ (2) Å³

$Z = 8$

$D_x = 1.684$ Mg m⁻³

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 7070 reflections

$\theta = 0.95-23.21$ °

$\mu = 2.019$ mm⁻¹

$T = 220$ (2) K

Needle

$0.35 \times 0.22 \times 0.18$ mm

Yellow

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction:

SADABS (Sheldrick, 1995a; Blessing, 1995)

$T_{\text{min}} = 0.59$, $T_{\text{max}} = 0.70$

24 313 measured reflections

8089 independent reflections

6599 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\text{max}} = 23.21$ °

$h = -23 \rightarrow 22$

$k = -15 \rightarrow 16$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.098$

$S = 1.140$

8072 reflections

662 parameters

H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2 + 11.1015P]$

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

$(\Delta/\sigma)_{\text{max}} = 0.006$

$\Delta\rho_{\text{max}} = 0.681$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.550$ e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn1—O13	2.036 (4)	Sn2—O21	2.046 (4)
Sn1—O12	2.045 (4)	Sn2—O22	2.051 (3)
Sn1—O11	2.055 (4)	Sn2—O23	2.052 (4)
Sn1—N14	2.177 (5)	Sn2—N24	2.191 (4)
Sn1—N11	2.191 (5)	Sn2—N21	2.193 (4)
Sn1—N13	2.196 (5)	Sn2—N23	2.204 (4)
O13—Sn1—O12	84.5 (2)	O21—Sn2—O22	87.63 (14)
O13—Sn1—O11	86.3 (2)	O21—Sn2—O23	88.1 (2)
O12—Sn1—O11	94.5 (2)	O22—Sn2—O23	87.13 (15)
O13—Sn1—N14	85.3 (2)	O21—Sn2—N24	165.6 (2)
O12—Sn1—N14	168.2 (2)	O22—Sn2—N24	80.32 (15)
O11—Sn1—N14	78.9 (2)	O23—Sn2—N24	83.6 (2)
O13—Sn1—N11	157.8 (2)	O21—Sn2—N21	83.8 (2)
O12—Sn1—N11	77.5 (2)	O22—Sn2—N21	164.0 (2)
O11—Sn1—N11	82.2 (2)	O23—Sn2—N21	79.2 (2)
N14—Sn1—N11	110.9 (2)	N24—Sn2—N21	106.0 (2)
O13—Sn1—N13	80.4 (2)	O21—Sn2—N23	79.6 (2)
O12—Sn1—N13	82.7 (2)	O22—Sn2—N23	83.70 (15)
O11—Sn1—N13	166.6 (2)	O23—Sn2—N23	164.9 (2)
N14—Sn1—N13	101.5 (2)	N24—Sn2—N23	106.6 (2)
N11—Sn1—N13	109.8 (2)	N21—Sn2—N23	107.8 (2)
Sn1—O11—C101—C102	159.8 (4)		
Sn1—O11—C101—C106	-22.7 (9)		
Sn1—N11—C107—C106	16.8 (9)		
Sn1—N11—C108—C109	-62.6 (6)		
Sn1—N13—C111—C110	-59.9 (6)		
Sn1—N13—C112—C113	12.5 (10)		
Sn1—O12—C118—C113	-36.6 (8)		
Sn1—O12—C118—C117	146.7 (5)		
Sn1—N14—C120—C119	-63.9 (5)		
Sn1—N14—C121—C122	6.1 (8)		
Sn1—O13—C127—C126	166.3 (5)		
Sn1—O13—C127—C122	-14.0 (9)		
Sn2—O21—C201—C206	-33.5 (7)		
Sn2—O21—C201—C202	150.3 (4)		
Sn2—N21—C208—C209	-59.6 (6)		
Sn2—N23—C211—C210	-62.0 (5)		
Sn2—N23—C212—C213	9.7 (7)		
Sn2—O22—C218—C213	-34.2 (7)		
Sn2—O22—C218—C217	148.3 (4)		
Sn2—N24—C221—C222	10.4 (8)		
Sn2—O23—C227—C222	-26.3 (7)		
Sn2—O23—C227—C226	156.0 (4)		

The data collection nominally covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle for the crystal and each

exposure of 10 s covered 0.3° in ω . The crystal-to-detector distance was 5 cm. Coverage of the unique set is over 95% complete out to at least 23° in θ . Crystal decay was found to be negligible by comparison of the intensities of repeated reflections at the end of the data collection. The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the parent atom.

Data collection: *SMART* (Siemens, 1994). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1995b). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

The authors wish to acknowledge the use of the EP-SRC's Chemical Database Service at Daresbury (Allen *et al.*, 1991). We also wish to thank the EPSRC and ICI Paints for a Case award (to SW), and EPSRC and Siemens for provision of X-ray facilities.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1015). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1034–1036

Tetramethylammonium Bis(coumarin-3-carboxylato)triphenylstannate Ethanol Solvate

SEIK WENG NG^a AND V. G. KUMAR DAS^b

^a*Institute of Advanced Studies, University of Malaya, 50603 Kuala Lumpur, Malaysia, and* ^b*Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia.*
E-mail: h1nswen@cc.um.edu.my

(Received 15 July 1996; accepted 17 March 1997)

Abstract

The title compound, [N(CH₃)₄][Sn(C₆H₅)₃(C₁₀H₅O₄)₂].·C₂H₅OH, crystallizes as non-interacting ammonium cations and stannate anions. The Sn atom which shows *trans*-trigonal bipyramidal coordination is linked axially to two monodentate carboxylato groups [Sn—O 2.232 (2) Å and O—Sn—O 167.1 (1)°].

Comment

Triphenyltin trifluoroacetate forms a stannate derivative with tetramethylammonium trifluoroacetate as a result of the electron-withdrawing ability of the CF₃CO₂ group. The formation of the coumarin-3-carboxylato analog implies an electron-withdrawing ability of the coumarin group, but this is not apparent from the structure of the parent acid. The coordination environment of Sn in the title compound, (I), differs from that in tetramethylammonium bis(trifluoroacetato)triphenylstannate (Ng & Kumar Das, 1997) in that the metal atom lies on a twofold rotation axis, so that the two Sn—O bonds are equivalent [Sn—O 2.232 (2) Å]. The O—Sn—O skeleton is bent [O—Sn—O 167.1 (1)°], whereas that in the trifluoroacetato derivative is almost linear [O—Sn—O 175.1 (1)°].

