

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{eq}
Pt	0.1904 (1)	0.2710 (1)	0.1268 (1)	0.035 (1)
N(1)	0.1528 (5)	0.4263 (4)	0.1756 (4)	0.041 (1)
C(2)	0.0969 (7)	0.4241 (5)	0.2802 (5)	0.051 (1)
C(3)	0.0895 (9)	0.5356 (7)	0.2991 (6)	0.067 (2)
C(4)	0.1431 (8)	0.6501 (6)	0.2087 (6)	0.068 (2)
C(5)	0.2617 (7)	0.7737 (6)	-0.0071 (6)	0.061 (2)
C(6)	0.3176 (7)	0.7733 (6)	-0.1126 (6)	0.059 (2)
C(7)	0.3753 (7)	0.6537 (6)	-0.2370 (5)	0.056 (1)
C(8)	0.3759 (7)	0.5392 (6)	-0.2421 (5)	0.057 (2)
C(9)	0.3223 (7)	0.4287 (5)	-0.1409 (5)	0.048 (1)
N(10)	0.2677 (5)	0.4280 (4)	-0.0358 (3)	0.038 (1)
C(11)	0.2663 (6)	0.5437 (5)	-0.0295 (4)	0.038 (1)
C(12)	0.3202 (6)	0.6588 (5)	-0.1273 (5)	0.048 (1)
C(13)	0.2069 (6)	0.5427 (5)	0.0832 (5)	0.040 (1)
C(14)	0.2045 (7)	0.6580 (5)	0.0953 (5)	0.051 (1)
S	0.1108 (2)	0.1130 (1)	0.3065 (1)	0.040 (1)
O	-0.0412 (5)	0.1525 (4)	0.3630 (3)	0.054 (1)
C(15)	0.0656 (8)	-0.0374 (6)	0.3118 (6)	0.061 (2)
C(16)	0.2941 (7)	0.0580 (5)	0.4130 (5)	0.051 (1)
C(17)	0.2448 (9)	0.1388 (6)	0.0593 (5)	0.061 (2)
P	0.6253 (2)	-0.2360 (2)	0.3590 (2)	0.056 (1)
F(1)	0.6194 (6)	-0.0844 (4)	0.3180 (4)	0.103 (2)
F(2)	0.7604 (7)	-0.2150 (6)	0.2540 (5)	0.129 (2)
F(3)	0.4985 (10)	-0.2536 (6)	0.4650 (6)	0.177 (3)
F(4)	0.6396 (9)	-0.3861 (5)	0.4037 (6)	0.144 (2)
F(5)	0.7882 (9)	-0.2551 (6)	0.4455 (5)	0.153 (2)
F(6)	0.4729 (7)	-0.2170 (7)	0.2700 (6)	0.165 (3)

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

Pt—C(17)	2.056 (5)	C(7)—C(12)	1.414 (8)
Pt—N(10)	2.075 (4)	C(8)—C(9)	1.379 (7)
Pt—N(1)	2.135 (4)	C(9)—N(10)	1.332 (6)
Pt—S	2.203 (1)	N(10)—C(11)	1.374 (6)
N(1)—C(2)	1.321 (6)	C(11)—C(12)	1.392 (7)
N(1)—C(13)	1.376 (6)	C(11)—C(13)	1.427 (7)
C(2)—C(3)	1.408 (8)	C(13)—C(14)	1.407 (7)
C(3)—C(4)	1.354 (9)	S—O	1.472 (3)
C(4)—C(14)	1.409 (8)	S—C(16)	1.772 (5)
C(5)—C(6)	1.343 (8)	S—C(15)	1.774 (5)
C(5)—C(14)	1.428 (8)	C(7)—C(8)	1.354 (8)
C(6)—C(12)	1.416 (8)		
C(17)—Pt—N(10)	93.1 (2)	C(11)—C(12)—C(6)	119.1 (5)
C(17)—Pt—N(1)	172.2 (2)	C(7)—C(12)—C(6)	123.6 (5)
N(10)—Pt—N(1)	79.3 (2)	N(1)—C(13)—C(14)	123.1 (5)
C(17)—Pt—S	90.9 (2)	N(1)—C(13)—C(11)	117.2 (4)
N(10)—Pt—S	175.7 (1)	C(14)—C(13)—C(11)	119.7 (5)
N(1)—Pt—S	96.6 (1)	C(13)—C(14)—C(4)	116.7 (5)
C(2)—N(1)—C(13)	117.8 (4)	C(13)—C(14)—C(5)	118.4 (5)
C(2)—N(1)—Pt	130.0 (4)	C(4)—C(14)—C(5)	124.9 (5)
C(13)—N(1)—Pt	112.0 (3)	O—S—C(16)	108.0 (2)
N(1)—C(2)—C(3)	122.5 (5)	O—S—C(15)	106.4 (3)
C(4)—C(3)—C(2)	120.0 (6)	C(16)—S—C(15)	100.7 (3)
C(9)—N(10)—C(11)	117.5 (4)	C(11)—N(10)—Pt	114.0 (3)
C(9)—N(10)—Pt	128.4 (3)		

The intensity data were evaluated by a peak-profile procedure (Diamond, 1969) and then corrected for Lp and absorption effects. The structure was solved by standard Patterson methods and subsequently completed by a combination of a least-squares technique and Fourier syntheses (SHELXL93; Sheldrick, 1993), and refined by a least-squares technique based on F^2 . Neutral-atom scattering factors and anomalous-dispersion corrections were used (Stewart, 1970). The SHELXTL-Plus package (Sheldrick, 1991) was used for data reduction and producing the graphics, while the final geometrical calculations were carried out with a locally modified version of PARST (Nardelli, 1983) on a DEC MicroVAX/3400

computer of the Centro Interdipartimentale per la Diffrazione a Raggi-X.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and hydrogen-bonding parameters have been deposited with the IUCr (Reference: NA1193). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 829–832

Poly[*trans*-bis{trimethyltin(IV)}- μ -(2,2'-bipyridyl-4,4'-dicarboxylato-O:O':O'':O''')]. A Layered Structure Composed of Sheets Interconnected by Organometallic Moieties

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(Received 12 May 1995; accepted 29 September 1995)

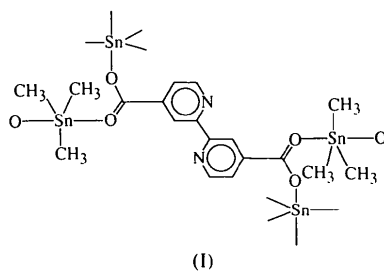
Abstract

In the solid state, the title compound, $[\text{Sn}_2(\text{C}_{12}\text{H}_6\text{N}_2\text{O}_4)(\text{CH}_3)_6]_n$, forms a two-dimensional polymer, which is developed parallel to the crystallographic 2_1 screw axis

of the $P2_1/n$ space group. The Sn atom shows normal *trans* pentagonal-bipyramidal coordination geometry and forms two bonds of different length, 2.145(2) and 2.519(2) Å, with the two *trans* O atoms. The carboxylate group bonds two organometallic moieties in a bridging bidentate fashion with a *syn-anti* configuration.

Comment

2,2'-Bipyridine has been used extensively as a ligand in both analytical and synthetic coordination chemistry (Reedijk, 1987) and has been incorporated in macrocycles. Polypyridine ligands display a variety of interesting complexation properties and have been used extensively in the synthesis of photosensitive and electroactive metal ion (particularly ruthenium) complexes (Juris, Balzani, Barigelletti, Campagna, Belser & Von Zelewsky, 1988). Monomeric complexes of the general formula *cis*-di(X)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II) (where X is chloro, bromo, iodo, cyanato, thiocyanato) have been reported which have been shown to act as efficient charge-transfer sensitizers for nanocrystalline TiO₂ films (Nazeeruddin, Kay, Rodicio, Humphry-Baker, Muller, Liska, Vlachopoulos & Gratzel, 1993). A pendant polymer complex of ruthenium(II) attached to a vinyl-2,2'-bipyridyl network has been described and its luminescence and redox properties investigated (Sun & De Armond, 1994). The title compound, in which trimethyltin(IV) moieties interconnect 2,2'-bipyridyl-4,4'-dicarboxylate units in a two-dimensional polymer, (1), offers a promising substrate for the synthesis of metal complex polymers possessing catalytic, redox and photoredox properties.



A drawing of the monomeric unit with the atomic numbering scheme is shown in Fig. 1, while Fig. 2 shows the polymer layer constituting the three-dimensional crystal packing of the solid. The central pentacoordinated Sn^{IV} atom has distorted trigonal bipyramidal geometry, with the three methyl groups in the equatorial plane; the Sn atom is displaced by 0.155(1) Å from this plane towards one of the carboxylate O atoms [O(1)] in apical positions. The main features of the polymer are: (a) each trimethyltin(IV) moiety bridges two adjacent 2,2'-bipyridyl-4,4'-dicarboxylate ligands and (b) each carboxylate group bonds to

two organometallic moieties in a bridging bidentate fashion with a *syn-anti* configuration. IR spectra of the ligand show a $\nu_{\text{asym}}(\text{COOH})$ stretching vibration at 1720 cm⁻¹, while in the polymer it appears to be shifted as $\nu_{\text{asym}}(\text{COO}^-)$ to 1603 cm⁻¹.

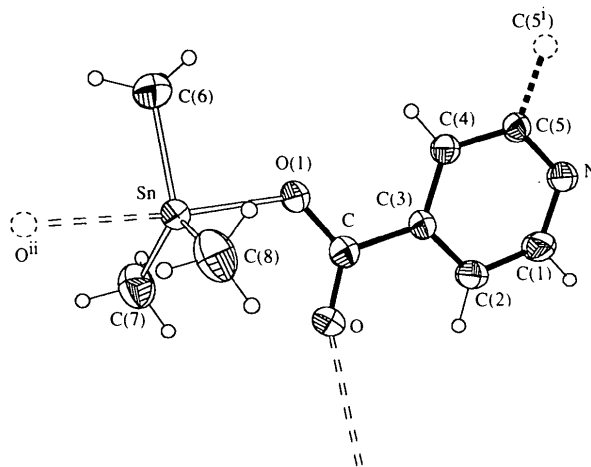


Fig. 1. A view of the monomeric unit of the title compound showing the crystallographic asymmetric unit with its numbering scheme (symmetry codes are as in Table 2). Atoms are represented by displacement ellipsoids at the 40% probability level. The H-atom size is arbitrary.

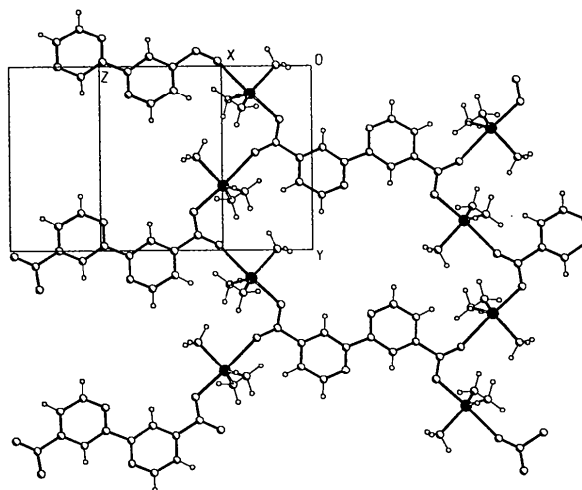


Fig. 2. A view of the structure of the polymer layer.

The coordination environment about the Sn atom is of the *trans*-O₂SnC₃ type, with a structural motif which is common for polymeric tin(IV) carboxylates (Tiekink, 1991). The *trans* Sn—O bond distances are different: 2.145(2) and 2.519(2) Å for Sn—O(1) and Sn—Oⁱⁱ, respectively [symmetry code as in Table 2]. Corresponding asymmetry is observed in the C—

O bonds of the carboxylate groups, the shorter one [1.230(4) Å] being *trans* to the longer Sn—Oⁱⁱ bond, while the longer bond [C—O(1) 1.281(4) Å] is opposite the shorter Sn—O bond. This suggests that partial double-bond character is preserved in the C—O bond coordinatively bonded to tin (longer Sn—O distance), while the O atom formally singly bonded to the C atom of the RCOO group seems to be covalently bonded to tin (shorter Sn—O distance). The former acyl O atom may be involved in a secondary intramolecular interaction with the Sn atom at a distance of 3.244(3) Å, similar to that reported for tributylstannyl-3-indolyl acetate (Molloy, Purcell, Mahon & Minshall, 1987). The *trans*-O(1)—Sn—Oⁱⁱ angle is 175.14(8)°. Catenated polymers are formed when just one carboxylate group, attached to an indole ring (Molloy, Purcell, Mahon & Minshall, 1987), is bridging bidentate or when a three-dimensional rather than a one-dimensional network is created by hydrogen bonds, as in aqua(2-pyridyl)carboxylatotrimethyltin (Harrison & Philips, 1979).

The sum of the valency angles around the metal atom in the equatorial plane is 358.3(2)°, the three C—Sn—C bond angles being C(7)—Sn—C(8) 125.8(2), C(6)—Sn—C(8) 115.8(2) and C(7)—Sn—C(6) 116.7(2)°. Such asymmetry is also observed in bis(2,6-dimethylphenoxy)trimethylstannate (Suzuki, Son, Noyori & Masuda, 1990). The three Sn—C bond lengths are nearly equal, ranging from 2.101(5) to 2.108(4) Å [mean value 2.104(4) Å], and are in good agreement with the corresponding distances reported in the literature (see Molloy, Quil, Cunningham, McArdle & Higgins, 1989).

In the 2,2'-bipyridyl-4,4'-dicarboxylate ligand, the central 2,2'-bipyridyl fragment is perfectly planar and in a *transoid* conformation, as can be seen from the N—C(5)—C(5')—Nⁱ torsion angle of 180.0(1)°, creating, *via* the carboxylate—tin bond, a two-dimensional molecular network. The carboxylate fragment makes an interplanar angle of 15.8(1)° with the 2,2'-bipyridyl plane. Bond distances and angles within the 2,2'-bipyridyl moiety are as expected. The layers of the polymeric structure are parallel to the crystallographic *b* axis and interconnected by weak van der Waals interactions. In the crystal packing, the 2,2'-bipyridyl rings of adjacent layers are parallel (but with an exactly stacked disposition), showing an interplanar distance of 3.566 Å.

As for the potential applications of this two-dimensional polymer, it has been mentioned recently (Chang, West, Fowler & Lauher, 1993) in the context of strategies for designing molecular solids depending on the particular anisotropic properties of their crystal packing constituted by rigid layers, with very strong interactions, namely covalent bonds, along the planes which are interconnected by weak interactions in the third dimension. The established coordination ability of 2,2'-bipyridine and other heterocyclic N donors suggests that ligands containing two carboxylate groups in suitable positions may be ideal for the assembly of mol-

ecules organized in layered structures which may possess properties of intramolecular energy transfer, even without active metal ions.

Experimental

The title polymer was synthesized by adding Me₃SnOH (0.434 g, 2.4 mmol) dissolved in 15 ml of anhydrous methanol to a suspension of 2,2'-bipyridyl-4,4'-dicarboxylic acid (0.293 g, 1.2 mmol) in 15 ml of anhydrous methanol (2:1 molar ratio). 2,2-Dimethoxypropane was added to the reaction mixture to remove water resulting from the neutralization reaction. The reaction mixture was refluxed with stirring for 36 h. The solids dissolved almost completely and a small amount of white solid (identified by IR as unreacted 2,2'-bipyridyl-4,4'-dicarboxylic acid) was filtered off and the remaining clear solution was evaporated in a vacuum desiccator at room temperature until colourless crystals of the title compound separated. The polymer appears to be stable on exposure to air and is soluble in most organic solvents. Analysis calculated (found) for C₁₈H₂₄N₂O₄Sn₂: C 37.94 (37.67), H 4.25 (4.30), N 4.92% (5.04%). Suitable yellow crystals were obtained from anhydrous methanol solution by slow evaporation.

Crystal data

[Sn₂(C₁₂H₆N₂O₄)(CH₃)₆]

M_r = 569.8

Monoclinic

*P*2₁/*n*

a = 6.595(2) Å

b = 9.964(2) Å

c = 17.821(2) Å

β = 94.76(2)°

V = 1167.0(3) Å³

Z = 2

D_x = 1.621 Mg m⁻³

D_m = 1.60 Mg m⁻³

D_m measured by flotation

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 50 reflections

θ = 6.5–15°

μ = 2.161 mm⁻¹

T = 298 K

Prismatic

0.35 × 0.31 × 0.20 mm

Yellow

Data collection

Siemens *R3m/V* diffractometer

ω–2θ scans

Absorption correction:

ψ scan (Kopffmann & Huber, 1968)

T_{min} = 0.200, *T_{max}* =

0.271

4143 measured reflections

2698 independent reflections

1934 observed reflections

[*F* ≥ 7σ(*F*)]

R_{int} = 0.041

θ_{max} = 27.5°

h = –1 → 9

k = –1 → 13

l = –24 → 24

3 standard reflections

monitored every 97

reflections

intensity decay: <3%

Refinement

Refinement on *F*

R = 0.0219

wR = 0.0295

S = 1.09

1934 reflections

121 parameters

H atoms in calculated

positions (*U* = 0.06 Å²)

w = 1/[σ²(*F_o*) + 0.0004*F_o*²]

(Δ/σ)_{max} = 0.005

Δρ_{max} = 0.45 e Å⁻³

Δρ_{min} = –0.33 e Å⁻³

Extinction correction: none

Atomic scattering factors

from Cromer & Mann

(1968) for non-H atoms

and Stewart (1970) for H

atoms

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U _{eq}
Sn	0.1633 (1)	0.1513 (1)	0.2226 (1)	0.042 (1)
C	0.0474 (5)	0.4195 (3)	0.1497 (2)	0.041 (1)
O	0.1844 (4)	0.4714 (3)	0.1908 (2)	0.058 (1)
O(1)	0.0128 (4)	0.2930 (2)	0.1463 (1)	0.050 (1)
N	-0.3560 (4)	0.6542 (3)	0.0001 (2)	0.044 (1)
C(1)	-0.1817 (5)	0.7039 (3)	0.0311 (2)	0.050 (1)
C(2)	-0.0471 (5)	0.6340 (3)	0.0805 (2)	0.045 (1)
C(3)	-0.0933 (4)	0.5015 (3)	0.0975 (2)	0.036 (1)
C(4)	-0.2726 (4)	0.4475 (3)	0.0647 (2)	0.036 (1)
C(5)	-0.4015 (4)	0.5260 (3)	0.0172 (2)	0.035 (1)
C(6)	0.0030 (8)	-0.0053 (4)	0.1654 (3)	0.092 (2)
C(7)	0.4641 (6)	0.1836 (4)	0.1960 (3)	0.068 (1)
C(8)	0.0313 (6)	0.2288 (5)	0.3173 (2)	0.067 (1)

Table 2. Selected geometric parameters (Å, °)

Sn—O(1)	2.145 (2)	Sn—O(1 ⁱ)	2.519 (2)
Sn—C(6)	2.101 (5)	Sn—C(7)	2.103 (4)
Sn—C(8)	2.108 (4)	N—C(5)	1.353 (4)
C(1)—C(2)	1.385 (5)	C(2)—C(3)	1.393 (4)
C—O	1.230 (4)	C(3)—C(4)	1.383 (4)
C—O(1)	1.281 (4)	C(4)—C(5)	1.391 (4)
C—C(3)	1.501 (4)	C(5)—C(5 ⁱⁱ)	1.483 (5)
N—C(1)	1.329 (4)		
O(1)—Sn—C(6)	89.6 (2)	O(1)—Sn—C(7)	98.7 (1)
C(6)—Sn—C(7)	116.7 (2)	O(1)—Sn—C(8)	93.8 (1)
C(6)—Sn—C(8)	115.8 (2)	C(7)—Sn—C(8)	125.8 (2)
O(1)—Sn—O ⁱ	175.1 (1)	C(6)—Sn—O ⁱ	85.8 (1)
C(7)—Sn—O ⁱ	84.9 (1)	C(8)—Sn—O ⁱ	86.6 (1)
C—C(3)—C(2)	121.0 (3)	C—C(3)—C(4)	120.8 (3)
O—C—O(1)	124.1 (3)	C(2)—C(3)—C(4)	118.1 (3)
O—C—C(3)	121.8 (3)	C(3)—C(4)—C(5)	119.7 (3)
O(1)—C—C(3)	114.1 (3)	N—C(5)—C(4)	122.2 (3)
C(1)—N—C(5)	117.3 (3)	N—C(5)—C(5 ⁱⁱ)	116.1 (3)
N—C(1)—C(2)	124.3 (3)	C(4)—C(5)—C(5 ⁱⁱ)	121.7 (3)
C(1)—C(2)—C(3)	118.3 (3)		

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

The intensity data were evaluated by a peak-profile procedure (Diamond, 1969) and then corrected for Lp and absorption effects. The structure was solved by standard Patterson methods, subsequently completed by a combination of a least-squares technique and Fourier syntheses (SHELXTL-Plus; Sheldrick, 1991), and refined by a full-matrix least-squares technique based on *F*. Neutral-atom scattering factors were used and an anomalous-dispersion correction was applied (Stewart, 1970). The SHELXTL-Plus package was used for data reduction and producing the graphics, while the final geometrical calculations were carried out with a locally modified version of PARST (Nardelli, 1983) on a DEC MicroVAX/3400 computer of the Centro Interdipartimentale per la Diffrazione a Raggi-X.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 832–835

μ -Oxo-bis[(5,10,15,20-tetraphenylporphyrinato)oxomolybdenum(V)]

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(Received 6 July 1995; accepted 23 October 1995)

Abstract

The crystal structure of a new solvated crystal form of the title complex, $[\{\text{Mo}(\text{O})(\text{TPP})\}_2\text{O}].1.5\text{H}_2\text{O}.0.5\text{CH}_2\text{Cl}_2$ (TPP = C₄₄H₂₈N₄), has been determined. The dimeric molecule is in a general position, and the overall structural features are extremely similar to those of the previously reported non-solvated form.

Comment

The μ -oxo form of Mo^V porphyrinates, $[\text{Mo}(\text{O})(\text{Porph})]_2\text{O}$, is now well known, since the structural characterization of the TPP (Johnson & Scheidt, 1978) and OEP (Kim, Sparapan & Ibers, 1987) derivatives. However, the behavior of this dimeric molecule in solution is rather subtle. Ledon, Bonnet, Brigandat & Varescon (1980) suggested an equilibrium in solution between the μ -oxo dimer and its dissociated monomer Mo(O)(Porph)(OH). They further pointed out that the isolation of solid material would depend upon the sol-