Data collection: *MAD* (Allibon, 1995). Cell refinement: *MAD*. Data reduction: *KRYSTAL* (Hazell, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994) and *KRYSTAL*. Program(s) used to refine structure: modified *ORFLS* (Busing, Martin & Levy, 1962) and *KRYSTAL*. Molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *KRYSTAL*. Software used to prepare material for publication: *KRYSTAL*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1462). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allibon, J. (1995). *MAD* (Aarhus version). Institut Laue-Langevin, Grenoble, France.

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* A**30**, 129–147.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

De Munno, G., Nicolò, F. & Julve, M. (1993). *Acta Cryst.* C**49**, 1049–1052.

Hazell, A. (1995). *KRYSTAL. An Integrated System of Crystallographic Programs*. Aarhus University, Denmark.

Krämer, T. & Strähle, J. (1986). *Z. Naturforsch. Teil B*, **41**, 692–696.

Pawley, G. S. (1971). *Adv. Struct. Res. Diffr. Methods*, **4**, 1–64.

Su, C. C. & Huang, S. M. (1984). *Trans. Met. Chem.* **9**, 220–224.

Acta Cryst. (1997). C**53**, 725–726

Bis(propionato-*O*)[5,10,15,20-tetra(*p*-chlorophenyl)porphyrinato-κ^4N]tin(IV) Dichloromethane Solvate

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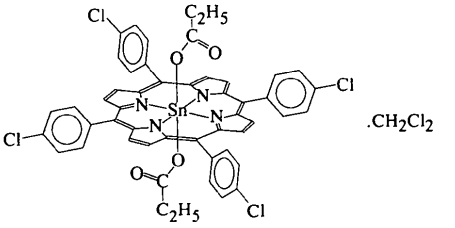
Abstract

As part of experiments directed towards the preparation of porphyrin derivatives of the main group elements, the title compound, $[Sn(C_{44}H_{24}Cl_4N_4)(C_3H_5O_2)_2].CH_2Cl_2$, crystallized as deep red crystals from dichloromethane.

Comment

The molecular structure of the title compound, (I), is shown in Fig. 1. The Sn atom lies on a centre of symmetry leading to a very symmetric structure. The geometry around the Sn atom is an almost regular octahedron, with Sn—N distances of 2.077 (4) and 2.091 (4) Å, and an Sn—O distance of 2.093 (4) Å. All angles around the Sn atom are close to 90°. The porphyrin ring system shows only minor deviations from planarity (mean deviation 0.024 Å) and bond distances are similar to those found in the closely related $[Sn(TPP)(OAc)_2]$ (Liu, Lin, Chen & Wang, 1996) and $[Sb(TPP)\{OCH(CH_3)_2\}]$ structures (Barbour, Belcher, Brothers, Rickard & Ware, 1992).



(I)

As in $[Sn(TPP)(OAc)_2]$, the interaction between the Sn atom and the carboxylic acid group is unidentate, the second O atom being 3.363 (5) Å from the Sn atom. There are no significant intermolecular interactions. The dichloromethane solvent lies on a twofold axis and in addition shows disorder of the Cl atom which has been modelled as two half-weighted atoms.

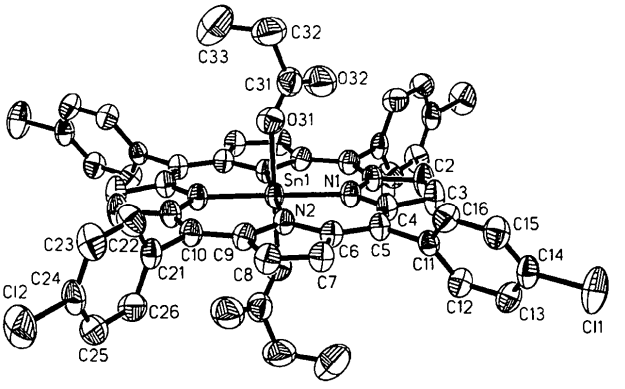


Fig. 1. The structure of $[Sn(TpClTPP)(OOCEt)_2]$. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

Experimental

$SnCl_4$ (2 ml, 17.1 mmol) and $H_2TpClPP$ (1.2 g, 1.6 mmol) were refluxed in chlorobenzene for 30 min. The solvent was removed under reduced pressure and the resulting crude solid chromatographed on basic alumina with dichloromethane as solvent. A single red band was removed, propionic acid (1 ml)

added and the solution slowly allowed to crystallize. The complex was isolated as small red prisms.

Crystal data

$[Sn(C_{44}H_{24}Cl_4N_4)(C_3H_5O_2)_2].CH_2Cl_2$
M_r = 1100.3
Monoclinic
$C2/c$
a = 25.310 (10) Å
b = 9.242 (2) Å
c = 23.006 (2) Å
β = 117.58 (2)°
V = 4770 (2) Å^3
Z = 4
D_x = 1.532 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 9.5–12.3°
μ = 0.922 mm^{-1}
T = 293 (2) K
Prism
0.27 × 0.20 × 0.15 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.845, T_{max} = 0.871
6009 measured reflections
5099 independent reflections

3294 reflections with $I > 2\sigma(I)$
R_{int} = 0.0231
θ_{max} = 27.58°
$h = 0 \rightarrow 32$
$k = 0 \rightarrow 11$
$l = -29 \rightarrow 26$
3 standard reflections
every 100 reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0482
$wR(F^2)$ = 0.1375
S = 1.063
5097 reflections
298 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 12.4112P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.048

$\Delta\rho_{max}$ = 0.674 e Å^{-3}
$\Delta\rho_{min}$ = −0.688 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00043 (12)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

$U_{eq} = (1/3)\sum_i\sum_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Sn1	0	0	0	0.03444 (15)
Cl1	0.27471 (8)	−0.7738 (2)	0.15003 (9)	0.0800 (5)
Cl2	−0.18457 (8)	−0.3648 (2)	−0.43123 (7)	0.0810 (6)
N1	0.0721 (2)	−0.0644 (4)	0.0871 (2)	0.0377 (9)
N2	0.0080 (2)	−0.1897 (4)	−0.0448 (2)	0.0355 (8)
C1	0.0935 (2)	0.0104 (6)	0.1450 (2)	0.0404 (10)
C2	0.1409 (3)	−0.0730 (6)	0.1933 (2)	0.0546 (15)
C3	0.1471 (3)	−0.1940 (6)	0.1642 (2)	0.057 (2)
C4	0.1040 (2)	−0.1889 (5)	0.0965 (2)	0.0426 (11)
C5	0.0955 (2)	−0.2943 (5)	0.0491 (2)	0.0381 (10)
C6	0.0514 (2)	−0.2944 (5)	−0.0160 (2)	0.0379 (10)
C7	0.0432 (2)	−0.3996 (6)	−0.0655 (2)	0.0440 (11)
C8	−0.0028 (2)	−0.3575 (6)	−0.1219 (2)	0.0453 (12)
C9	−0.0254 (2)	−0.2246 (5)	−0.1098 (2)	0.0398 (11)
C10	−0.0722 (2)	−0.1428 (5)	−0.1559 (2)	0.0406 (11)
C11	0.1391 (2)	−0.4158 (5)	0.0707 (2)	0.0355 (11)
C12	0.1990 (2)	−0.3883 (6)	0.0924 (2)	0.0444 (12)
C13	0.2406 (2)	−0.4970 (7)	0.1166 (2)	0.0507 (11)
C14	0.2217 (2)	−0.6367 (6)	0.1179 (2)	0.0466 (12)
C15	0.1629 (2)	−0.6693 (6)	0.0943 (3)	0.0496 (13)
C16	0.1215 (2)	−0.5581 (6)	0.0713 (3)	0.0449 (12)
C21	−0.1010 (2)	−0.1992 (5)	−0.2245 (2)	0.0402 (11)
C22	−0.1468 (3)	−0.2991 (6)	−0.2457 (3)	0.0529 (14)
C23	−0.1722 (3)	−0.3505 (6)	−0.3093 (3)	0.0563 (15)
C24	−0.1517 (2)	−0.3014 (6)	−0.3511 (2)	0.0501 (13)
C25	−0.1071 (3)	−0.2010 (7)	−0.3324 (3)	0.0563 (14)
C26	−0.0814 (2)	−0.1487 (7)	−0.2686 (3)	0.0529 (14)
O31	−0.0627 (2)	−0.1085 (4)	0.0195 (2)	0.0532 (9)
O32	−0.0343 (2)	−0.0583 (6)	0.1229 (2)	0.0858 (15)
C31	−0.0698 (3)	−0.1106 (7)	0.0689 (3)	0.0581 (15)
C32	−0.1283 (3)	−0.1687 (10)	0.0614 (4)	0.083 (2)
C33	−0.1795 (4)	−0.0710 (12)	0.0174 (4)	0.108 (3)
Cl3†	0.9371 (4)	0.6581 (9)	0.2435 (4)	0.164 (3)
Cl3′†	0.9392 (2)	0.6026 (6)	0.2182 (2)	0.0934 (13)
C34	1	0.7373 (15)	0.25	0.100 (4)

† Site occupancy = 0.50.

Table 2. *Selected geometric parameters (Å, °)*

Sn1—N1	2.077 (4)	C3—C4	1.430 (7)
Sn1—N2	2.091 (4)	C4—C5	1.403 (7)
Sn1—O31	2.093 (4)	C5—C6	1.394 (6)
N1—C4	1.365 (6)	C5—C11	1.489 (6)
N1—C1	1.371 (6)	C6—C7	1.436 (6)
N2—C9	1.373 (6)	C7—C8	1.338 (7)
N2—C6	1.382 (6)	C8—C9	1.435 (7)
C1—C2	1.426 (7)	C9—C10	1.391 (7)
C2—C3	1.349 (7)	C10—C21	1.494 (6)
N1—Sn1—N2^i	90.4 (2)	N1—Sn1—O31	94.0 (2)
N1—Sn1—N2	89.6 (2)	N2^i—Sn1—O31	92.3 (2)
N1^i—Sn1—O31	86.0 (2)	N2—Sn1—O31	87.7 (2)

Symmetry code: (i) $-x, -y, -z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: NA1286). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Barbour, T., Belcher, W. J., Brothers, P. J., Rickard, C. E. F. & Ware, D. C. (1992). *Inorg. Chem.* **31**, 746–754.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Liu, I.-C., Lin, C.-C., Chen, J.-H. & Wang, S.-S. (1996). *Polyhedron*, **15**, 459–463.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.