

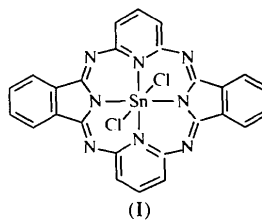
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### *trans*-Dichloro(hemiporphyrinato)tin(IV)†

PHILIP J. FARRIS,<sup>a</sup> JUDE T. JACOBS,<sup>a</sup> MICHAEL P. OKONCZAC,<sup>a</sup> WILLIAM S. DURFEE<sup>a</sup> AND BRUCE C. NOLL<sup>b</sup>

<sup>a</sup>Department of Chemistry, Buffalo State College, 1300 Elmwood Avenue, Buffalo, NY 14222-1095, USA, and

<sup>b</sup>Department of Chemistry and Biochemistry, University of Colorado, Boulder, CO 80309-0215, USA. E-mail: [durfeews@buffalostate.edu](mailto:durfeews@buffalostate.edu)

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#### Abstract

*trans*-Dichloro(hemiporphyrinato)tin(IV), [SnCl<sub>2</sub>(C<sub>26</sub>H<sub>14</sub>N<sub>8</sub>)], is a planar macrocyclic complex with six-coordinate tin in an N<sub>4</sub> coordination site bound to axial Cl ligands. The molecule has *D*<sub>2h</sub> symmetry and is characterized by two short Sn—N(isoindole) bonds [2.041 (3) Å] and two long Sn—N(pyridine) bonds [2.213 (4) Å].

#### Comment

Transition metal, main-group and lanthanide complexes of the phthalocyanine macrocyclic ligand have been extensively studied. Complexes of the related hemiporphyrin ligand (Elvidge & Linstead, 1952), however, have received much less attention. We have started to examine the chemistry of the hemiporphyrin (hp) macrocycle, especially complexes of the main-group and lanthanide elements. In an attempt to form eight-coordinate Sn<sup>IV</sup>-bis(hp) dimers similar to those reported for the phthalocyanine (pc) ligand (Bennett *et al.*, 1973), we first synthesized and structurally characterized the complex *trans*-(Cl)<sub>2</sub>(hp)Sn<sup>IV</sup>, (I) (Meyer *et al.*, 1975). This represents the second structure determination of a main-group hemiporphyrin complex (Hecht & Luger, 1974).

† Alternative name: dichloro(5,26:13,18-diimino-7,11:20,24-dinitrilo-benzoc[*c,n*][1,6,12,17]tetrazacyclodocosinato)tin(IV).

Tin(IV) forms a six-coordinate complex with the hp ligand (Fig. 1). The asymmetric unit consists of half of the macrocycle, with tin located on an inversion center. The tin cation is thus located exactly in the plane of the N<sub>4</sub> coordination sphere; chloride ligands bond to tin in the axial positions. Two different Sn—N distances result from the *D*<sub>2h</sub> symmetry of the complex. The Sn—N(isoindole) distance is 2.041 (3) Å, while the Sn—N(pyridine) distance is considerably longer at 2.213 (4) Å. The Sn—Cl distance is 2.421 (1) Å. Unusually short intermolecular contacts are observed between the Cl<sup>−</sup> ion and H3A from the molecule at ( $\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} - z$ ) and H4A from the molecule at ( $1 - x, -y, -z$ ); these distances are 2.7 and 2.9 Å, respectively. All other Cl<sup>−</sup>⋯H intermolecular contacts are greater than 3 Å. The molecule packs in a herringbone motif (see figure in supplementary material).

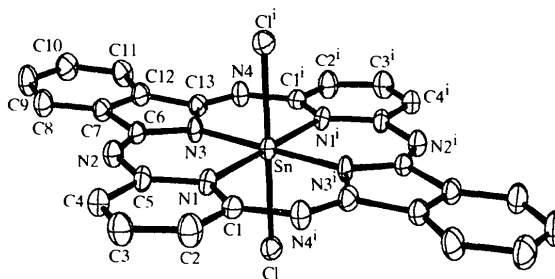


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

The planarity of the hp macrocycle of (I) is in marked contrast with the related *trans*-dichloro-(phthalocyaninato)tin(IV) complex. In the pc complex, the macrocycle assumes a ruffled non-planar geometry to accommodate the large radius of the Sn<sup>IV</sup> ion (Rogers & Osborn, 1971). The Sn—N distances are 2.051 Å. The larger coordination site in the hp macrocycle, resulting from the replacement of two of the four isoindole groups found in phthalocyanine with pyridines, is readily seen in the long Sn—N(pyridine) distances, although the Sn—N(isoindole) distance of 2.041 (3) Å is shorter than the Sn—N distance in the pc complex. Two different metal–nitrogen bond distances have also been observed for transition metal–hp complexes (Agostinelli *et al.*, 1984; Collamati *et al.*, 1986).

## Experimental

The metal-free hemiporphyrzine ligand (hpH2) was synthesized according to a published procedure (Dirk & Marks, 1984). *trans*-Dichloro(hemiporphyrzinato)tin(IV) was synthesized by refluxing hpH<sub>2</sub> (1.0 g, 2.3 mmol) and SnCl<sub>4</sub>·5H<sub>2</sub>O (1.0 g, 2.9 mmol) in 10 ml of 1-chloronaphthalene for 45 min. The hot reaction vessel was then suspended in 463 K silicon oil in a dewar and slowly cooled to room temperature. Crystals suitable for X-ray diffraction formed over the course of several hours.

### Crystal data

[SnCl<sub>2</sub>(C<sub>26</sub>H<sub>14</sub>N<sub>8</sub>)]

*M<sub>r</sub>* = 628.04

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 7.6544 (12) Å

*b* = 15.351 (3) Å

*c* = 10.287 (2) Å

β = 106.544 (8)°

*V* = 1158.69 (2) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.800 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

### Data collection

Siemens SMART diffractometer

0.3° ω scans

Absorption correction: multi-scan (Blessing, 1995)

*T<sub>min</sub>* = 0.73, *T<sub>max</sub>* = 0.96

8149 measured reflections

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 4116 reflections

θ = 0.00–28.29°

μ = 1.37 mm<sup>-1</sup>

*T* = 153 (2) K

Needle

0.27 × 0.04 × 0.04 mm

Dark green

2842 independent reflections

2011 reflections with

*I* > 2σ(*I*)

*R<sub>int</sub>* = 0.053

θ<sub>max</sub> = 28.29°

*h* = -10 → 10

*k* = -20 → 12

*l* = -13 → 13

### Refinement

Refinement on *F*<sup>2</sup>

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.046

*wR*(*F*<sup>2</sup>) = 0.120

*S* = 0.999

2842 reflections

169 parameters

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0689*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 1.10 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -2.06 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn—N3 (isoindole)	2.041 (3)	Sn—Cl	2.4214 (11)
Sn—N1 (pyridine)	2.213 (4)		
N3—Sn—N1	90.27 (13)	N1'—Sn—Cl	88.86 (10)
N3'—Sn—N1	89.73 (13)	N3—Sn—Cl	90.82 (11)
N1—Sn—Cl	91.13 (10)	N3'—Sn—Cl	89.17 (11)
N1—Sn—Cl'	88.87 (10)	N3—Sn—Cl'	89.18 (11)

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

Crystals were handled under a light hydrocarbon oil. The datum crystal was affixed with a small amount of silicone vacuum grease to a thin glass fiber on a tapered copper mounting pin and mounted on the goniometer head. An arbitrary hemisphere of space was collected at 30 s per 0.3° ω scan. Coverage to 0.75 Å was 92%, and 99% to 0.80 Å. No decay was observed in 50 frames remeasured on completion of

the data collection. The largest features in the final difference map were 1.10 e Å<sup>-3</sup> at 0.87 Å from Sn and -2.06 e Å<sup>-3</sup> at 0.88 Å from Sn.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1399). Services for accessing these data are described at the back of the journal.

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## 1,1'-Diiodo-3,3'-bis(trimethylsilyl)ferrocene

DANIEL A. FOUCHER,<sup>a</sup> ALAN J. LOUGH<sup>b</sup> AND PETER PARK<sup>b</sup>

<sup>a</sup>Xerox Research Centre of Canada, 2660 Speakman Drive, Mississauga, Ontario, Canada L5K 2L1, and <sup>b</sup>Department of Chemistry, University of Toronto, Toronto, Ontario, Canada M5S 3H6. E-mail: [alough@chem.utoronto.ca](mailto:alough@chem.utoronto.ca)

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## Abstract

In the structure of the title compound, [Fe(C<sub>8</sub>H<sub>12</sub>ISi)<sub>2</sub>], the cyclopentadienyl rings are fully eclipsed. The trimethylsilyl groups are staggered by 141.0(4)° and the iodo groups by 69.6(3)°.