

Crystal data

[SnCl₂(C₄H₉)₂(C₁₀H₈N₂)]
M_r = 460.00
 Monoclinic
*P*2₁/*n*
a = 9.529 (2) Å
b = 16.354 (5) Å
c = 13.719 (5) Å
 β = 105.130 (10)°
V = 2063.8 (11) Å³
Z = 4
D_x = 1.480 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 56 reflections
 θ = 12.38–19.62°
 μ = 1.498 mm⁻¹
T = 293 (2) K
 Parallelepiped
 0.8 × 0.2 × 0.2 mm
 Colourless

Data collection

Philips PW1100 diffractometer (updated by Stoe)
 ω -2 θ scans
 Absorption correction:
 ψ scan (North *et al.*, 1968)
T_{min} = 0.469, *T_{max}* = 0.741
 3575 measured reflections
 3456 independent reflections

2873 reflections with *I* > 2 σ (*I*)
 R_{int} = 0.064
 θ_{max} = 25.08°
 h = -10 → 8
 k = 0 → 17
 l = -8 → 16
 3 standard reflections
 frequency: 120 min
 intensity decay: 6.1%

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.158$
S = 1.119
 3456 reflections
 207 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0680P)^2 + 9.9655P]$
 where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.001
 $\Delta\rho_{\text{max}}$ = 1.21 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -1.09 e Å⁻³
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 5 (5) × 10⁻⁴
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn—C11	2.493 (2)	Sn—C15	2.142 (9)
Sn—C12	2.566 (2)	Sn—N1	2.412 (6)
Sn—C11	2.127 (9)	Sn—N2	2.405 (6)
C11—Sn—C11	96.7 (3)	C15—Sn—C12	85.3 (3)
C11—Sn—C12	89.3 (3)	N1—Sn—C11	159.73 (16)
C11—Sn—C15	169.3 (4)	N1—Sn—C12	95.96 (16)
C11—Sn—N1	84.5 (3)	N1—Sn—N2	67.8 (2)
C11—Sn—N2	88.8 (3)	N2—Sn—C11	91.98 (17)
C15—Sn—N1	86.8 (3)	N2—Sn—C12	163.74 (17)
C15—Sn—N2	93.8 (3)	C11—Sn—C12	104.28 (8)
C15—Sn—C11	93.6 (3)		

The rather high values of the isotropic displacement parameters of atoms C14, C17 and C18 of the two butyl groups were indicative of unmodelled disorder (Warda, 1998). The assumption of 'similar' *U^{ij}* values (*SIMU*) for the adjacent atoms and the application of distance restraints to target values of 1.52 (1) Å proved to be very effective for all pairs of atoms separated by less than 0.8 Å on the two butyl chains. This type of refinement led to occupancies of 0.76 (3) and 0.24 (3) for C14 and C14', respectively, and values of 0.71 (3) for C17 and C18, and of 0.29 (2) for C17' and C18'. The final difference electron-density map revealed a highest peak of 1.21 e Å⁻³

0.97 Å from Sn and a deepest hole of -1.09 e Å⁻³ 0.83 Å⁻³ from Sn. The data have been corrected for absorption effects. Five low-resolution reflections suffering from extinction were omitted from the refinement. An extinction correction was applied. H atoms were placed in calculated positions and thereafter allowed to ride on their parent atoms, with *U_{iso}*(H) values of 1.2*U_{eq}*(C).

Data collection: *DIF4* (Stoe & Cie, 1987a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1987b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCR electronic archives (Reference: BM1319). Services for accessing these data are described at the back of the journal.

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Bis(2-methyl-8-quinolinolato-*N,O*)tin(II)

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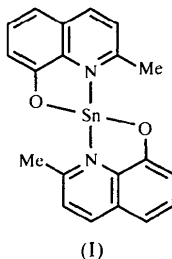
Abstract

The title compound, [Sn(C₁₀H₈NO)₂], displays a unique geometry, looking like a two-bladed screw. The geometry about the Sn center is distorted pseudo-trigonal

bipyramidal, with an equatorial Sn^{II} lone pair and two *N,O*-bidentate 8-quinolinolate ligands. The angle between the two quinoline planes is 91.6(1)°. The molecule has approximate *C*₂ symmetry. The presence of a near-*C*₂ axis shows that the molecule has an axial chirality.

Comment

8-Quinolinol (oxine) is well known as an analytical reagent (Phillips, 1956). It forms chelate complexes with many metal ions as an *N,O*-bidentate ligand, is used for the determination of metals and has found a recent application as a component molecule for molecular electronic devices (Tang & VanSlyke, 1987). Studies on the molecular structure of its complexes, however, have not been reported satisfactorily. With respect to bivalent metal complexes, it was reported that Cu (Hoy & Morriss, 1967), Zn (Palenik, 1964) and Pd (Kamenar *et al.*, 1966) complexes have *trans*-planar configurations, as determined by X-ray analysis. The structure of the bivalent Sn complex, (I), was not elucidated, although a few studies (Stevens, 1959; Morrison & Haendler, 1967; Doskey & Curran, 1969) on its analysis and synthesis have been reported.



The molecular structure of (I) is shown in Fig. 1. The geometry of the Sn^{II} atom is *trans*, with two almost equivalent Sn—N and Sn—O bonds from two bidentate ligands. It is of interest that the molecule has a unique geometry, looking like a two-bladed screw. The dihedral angle between the two quinoline planes is 91.6(1)°. Further, the molecule has a chemical *C*₂ axis lying on the bisectors of the N—Sn—N and O—Sn—O bond angles. These unusual structural characteristics can be ascribed to valence electrons of the Sn^{II} atom, which has one lone pair of electrons. The geometry about the Sn^{II} atom can be described as distorted pseudo-trigonal bipyramidal, with the Sn^{II} lone pair occupying an equatorial site. Similar observations were noted for other Sn^{II} complexes (Ewings, Harrison & Fenton, 1975; Ewings, Harrison & King, 1975; Ewings *et al.*, 1976). The Sn—O [2.099(2) and 2.102(2) Å] and Sn—N [2.424(2) and 2.419(2) Å] bonds are characteristic of covalent (Ewings, Harrison & King, 1975) and coordinate (Shields *et al.*, 1973) interactions, respectively. The O—Sn—O and N—Sn—N

bond angles are 97.43(7) and 137.32(6)°, respectively, showing a high degree of distortion. The presence of an approximate *C*₂ axis leads to the appearance of Δ or Λ chirality around the Sn atom, although the crystals obtained are racemic. Some Sn^{II} complexes are sensitive to air and moisture, however, the title compound has been stable for several months. The stability would be due mainly to the electronic contribution of the N-donor in quinoline and partly to protection for Sn^{II} by two side-chain methyl groups against oxygen.

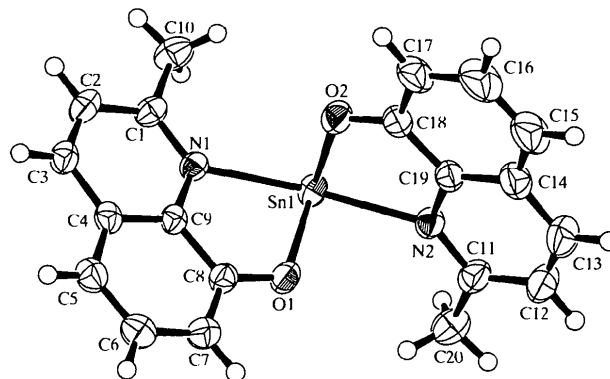


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids.

Experimental

The reported procedure of Morrison & Haendler (1967) was modified for the preparation of (I). To a freshly prepared solution of sodium methoxide (4.2 mmol) in methanol, 2-methyl-8-quinolinol (0.65 g, 4.0 mmol) in methanol (20 ml) was added. After 5 min, tin(II) chloride (0.38 g, 2.0 mmol) in methanol (10 ml) was added and the mixture was stirred for 30 min at room temperature. Filtration afforded a yellow solid, which was washed with methanol and dried *in vacuo* [yield 0.57 g, 66%; m.p. 534–537 K (decomposition)]. Analysis: C 55.22, H 3.71, N 6.44%; calculated for C₂₀H₁₆N₂O₂Sn: C 55.38, H 3.82, N 6.55%. MS (FAB): *m/z* 436 (*M*⁺). Crystals were obtained by slow evaporation from an EtOH–THF solution.

Crystal data

[Sn(C₁₀H₈NO)₂]
M_r = 435.05
 Triclinic
P $\bar{1}$
a = 9.713(2) Å
b = 12.079(3) Å
c = 7.716(2) Å
 α = 93.11(2)°
 β = 106.85(2)°
 γ = 81.92(2)°
V = 857.7(3) Å³
Z = 2
D_x = 1.684 Mg m⁻³
D_m not measured

Mo *K* α radiation
 λ = 0.7107 Å
 Cell parameters from 25 reflections
 θ = 14.9–15.0°
 μ = 1.505 mm⁻¹
T = 224.4 K
 Prism
 0.5 × 0.3 × 0.1 mm
 Yellow

Data collection

Rigaku AFC-7R diffractometer
 ω -2 θ scans
 Absorption correction:
 ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.529$, $T_{\max} = 0.860$
 5276 measured reflections
 4999 independent reflections

4710 reflections with $I > 0$
 $R_{\text{int}} = 0.016$
 $\theta_{\max} = 30^\circ$
 $h = 0 \rightarrow 13$
 $k = -16 \rightarrow 16$
 $l = -10 \rightarrow 10$
 3 standard reflections every 150 reflections
 intensity decay: 2.1%

Refinement

Refinement on F^2
 $R(F) = 0.031$
 $wR(F^2) = 0.089$
 $S = 1.551$
 4999 reflections
 227 parameters
 H atoms not refined
 $w = 1/[\sigma^2(F_o^2) + 0.0025(F_o^2)^2]$
 $(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.52 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.33 \text{ e } \text{\AA}^{-3}$
 Extinction correction: Zachariasen (1967), type 2, Gaussian isotropic
 Extinction coefficient: 0.042 (3)
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Sn1—O1	2.099 (2)	Sn1—N1	2.424 (2)
Sn1—O2	2.102 (2)	Sn1—N2	2.419 (2)
O1—Sn1—O2	97.43 (7)	O2—Sn1—N1	79.19 (6)
O1—Sn1—N1	73.13 (6)	O2—Sn1—N2	72.90 (7)
O1—Sn1—N2	79.13 (6)	N1—Sn1—N2	137.32 (6)

The diffractometer was equipped with a Rigaku low-temperature device. H atoms were placed in calculated positions and were not refined.

Data collection: *MSCI/AFD Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCI/AFD Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). 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*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1007). Services for accessing these data are described at the back of the journal.

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[CuCl(C₁₂H₉N₂O₅)(C₁₂H₁₀N₂O₅)(H₂O)]

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Abstract

In the title compound, aquachloro(1-ethyl-1,4-dihydro-4-oxo-1,3-dioxolo[4,5-g]cinnoline-3-carboxylato-*O*³,*O*⁴)-(1-ethyl-1,4-dihydro-4-oxo-1,3-dioxolo[4,5-g]cinnoline-3-carboxylic acid-*O*³,*O*⁴)copper(II), the Cu^{II} ion adopts a tetragonally distorted octahedral geometry. In the equatorial plane, the Cu^{II} ion is coordinated to two O atoms (O_{keto} and O_{carboxylate}) of one cinoxacin ligand, and to two O atoms (O_{keto} and O_{carboxylic}) of one cinoxacin molecule. The apical positions are occupied by one O atom from the water molecule and one Cl⁻ ion, resulting in a CuClO₅ chromophore.

Comment

Cinoxacin, HCx (1-ethyl-1,4-dihydro-4-oxo-1,3-dioxolo[4,5-g]cinnoline-3-carboxylic acid; Cx is the deprotonated form, 1-ethyl-1,4-dihydro-4-oxo-1,3-dioxolo[4,5-g]cinnoline-3-carboxylate), is a quinolone antimicrobial