

Refinement

Refinement on F^2 $R(F) = 0.0288$ $wR(F^2) = 0.0787$ $S = 1.068$

3458 reflections

326 parameters

H atom parameters refined

 $w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.0113P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.787 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.628 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn1—C11	2.144 (4)	Sn1—C31	2.141 (4)
Sn1—C21	2.149 (4)	Sn1—C41	2.125 (4)
C11—Sn1—C21	107.9 (2)	C21—Sn1—C31	108.1 (1)
C11—Sn1—C31	107.4 (2)	C21—Sn1—C41	108.3 (2)
C11—Sn1—C41	111.2 (1)	C31—Sn1—C41	113.9 (2)

Data collection: XSCANS (Siemens, 1991). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ZORTEP (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: SHELXL93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[Bis(*N,N*-dimethylthiocarbamoylthio)-acetato-*O*]triphenyl(quinoline *N*-oxide-*O*)tin

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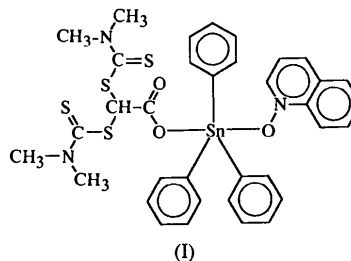
(Received 24 June 1996; accepted 5 November 1996)

Abstract

The Sn atom in the title compound, [Sn(C₆H₅)₃-(C₈H₁₃N₂O₂S₄)(C₉H₇NO)], shows *trans*-C₃SnO₂ trigonal bipyramidal coordination [$\Sigma\text{C—Sn—C}$ 357.5 (6), O—Sn—O 171.4 (2)° and Sn←O 2.446 (4) Å].

Comment

Triphenyltin carboxylates generally do not yield complexes with oxygen-donor ligands unless the anion possesses electron-withdrawing groups that can raise the Lewis acidity of tin. Bis(*N,N*-dimethyldithiocarbamyl)-acetate displays enhanced acceptor properties as it is able to form 1:1 complexes with hexamethylphosphoramide and triphenylphosphine oxide (Ng, 1995). The Sn atom in the title complex, (I), is also five-coordinate in a trigonal bipyramidal geometry, but the Sn←O bond [2.446 (4) Å] is much longer than those in the phosphorus donors, as N-atom donors are weaker Lewis bases than P-atom donors. The bond distance is also longer than that [2.319 (6) Å] found in the quinoline *N*-oxide complex of *N*-triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (Ng, 1994), but is similar to that [2.459 (5) Å] found in the quinoline *N*-oxide complex of tri(*p*-tolyl)tin bromide (Kumar Das, Yap, Ng, Chen & Mak, 1986).



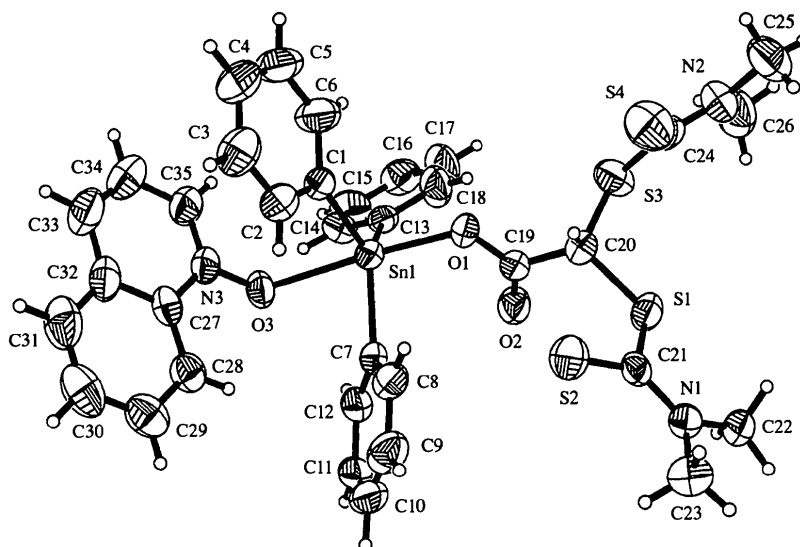


Fig. 1. A ZORTEP (Zsolnai & Pritzkow, 1996) plot of the title complex at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

The bond dimensions of the carboxylato anion are similar to those found in dicyclohexylammonium bis(*N,N*-dimethyldithiocarbamyl)acetate (Ng, 1996).

6492 measured reflections
6135 independent reflections

3 standard reflections
frequency: 60 min
intensity decay: 2%

Experimental

Equimolar quantities of bis(*N,N*-dimethyldithiocarbamyl)-acetic acid (Pluijgers & van der Kerk, 1961), triphenyltin hydroxide and quinoline *N*-oxide hydrate were dissolved in a small volume of hot ethanol. The complex separated on cooling the filtered solution.

Crystal data

[Sn(C₆H₅)₃(C₈H₁₃N₂O₂S₄)-
(C₉H₇NO)]

$M_r = 792.59$

Triclinic

$P\bar{1}$

$a = 10.9126 (5) \text{ \AA}$

$b = 11.5192 (6) \text{ \AA}$

$c = 14.182 (1) \text{ \AA}$

$\alpha = 94.715 (5)^\circ$

$\beta = 97.083 (5)^\circ$

$\gamma = 96.918 (4)^\circ$

$V = 1747.8 (2) \text{ \AA}^3$

$Z = 2$

$D_x = 1.506 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25
reflections

$\theta = 12\text{--}13^\circ$

$\mu = 1.009 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Block

$0.57 \times 0.50 \times 0.43 \text{ mm}$

Colorless

Refinement

Refinement on F^2

$R(F) = 0.053$

$wR(F^2) = 0.1540$

$S = 1.03$

6135 reflections

415 parameters

H atoms: riding model;

$U(\text{H}) = 1.5U_{\text{eq}}(\text{C})$

$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2 + 4.5654P]$

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

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 2.312 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.740 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

*International Tables for
Crystallography* (Vol. C)

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

Sn1—C1	2.120 (5)	Sn1—O1	2.147 (4)
Sn1—C7	2.124 (5)	Sn1—O3	2.446 (4)
Sn1—C13	2.135 (5)		
C1—Sn1—C7	120.1 (2)	C13—Sn1—O1	99.0 (2)
C1—Sn1—C13	112.4 (2)	C1—Sn1—O3	85.6 (2)
C7—Sn1—C13	125.0 (2)	C7—Sn1—O3	79.5 (2)
C1—Sn1—O1	91.6 (2)	C13—Sn1—O3	89.6 (2)
C7—Sn1—O1	95.0 (2)	O1—Sn1—O3	171.4 (2)

Data collection: CAD-4-VAX/PC (Enraf-Nonius, 1988). Cell refinement: CAD-4-VAX/PC. Data reduction: NRCVAX (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: DIRDIF94 (Beurskens *et al.*, 1994). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ZORTEP (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: SHELXL93.

The author thanks the University of Malaya (F102/96 and F677/96) for supporting this work.

Data collection

Enraf-Nonius CAD-4
diffractometer

ω scans

Absorption correction:

ψ scans (North, Phillips
& Mathews, 1968)

$T_{\text{min}} = 0.535$, $T_{\text{max}} = 0.648$

5379 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.0115$

$\theta_{\text{max}} = 24.97^\circ$

$h = 0 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -16 \rightarrow 16$

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Diethylenetriamine)(imidazole)(thiocyanato)copper(II) Perchlorate

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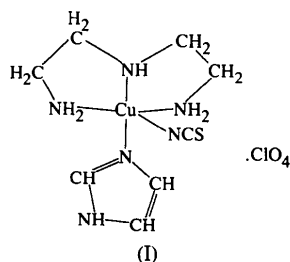
Abstract

The Cu atom in the title complex, [*N*-(2-aminoethyl)-1,2-ethylenediamine-*N,N'*](imidazole-*N*³)(thiocyanato-*N*)copper(II) perchlorate, [Cu(NCS)(C₄H₁₃N₃)(C₃H₄N₂)]ClO₄, is coordinated in a trigonal bipyramidal arrangement by two primary N atoms of *N*-(2-aminoethyl)-1,2-ethylenediamine (diethylenetriamine) and one N atom of thiocyanate in equatorial positions, and by

the 'pyridine' N atom of imidazole and one secondary N atom of diethylenetriamine in axial positions. The Cu—N(imidazole) and Cu—N(thiocyanate) bond lengths are 1.978 (3) and 2.245 (4) Å, respectively.

Comment

Imidazolate-bridged dinuclear complexes have been synthesized and studied extensively (O'Young, Dewan, Lilienthal & Lippard, 1978; Kolks, Frihart, Coughlin & Lippard, 1981; Coughlin & Lippard, 1984). Polynuclear imidazolate-bridged structures with additional imidazole neutral ligands in the solid state have been reported (Ivarsson, Lundberg & Ingri, 1972; Lundberg, 1972; Sato, Nagae, Uehara & Nakaya, 1984; Sato *et al.*, 1986). Although dinuclear and polynuclear imidazolate-bridged copper(II) complexes have been reported, the known structures and properties of mononuclear complexes with mixed ligands are few. In this paper, we describe the preparation and crystal structure of the monocopper(II) complex, [Cu(dien)(imH)(NCS)]ClO₄, (I), where dien is diethylenetriamine and imH is imidazole.



The geometry around the Cu atom is trigonal bipyramidal, comprising the tridentate diethylenetriamine, the imidazole and the thiocyanate ligands (Fig. 1). The two primary N atoms of dien and the N atom of thiocyanate are coordinated in the trigonal plane, and the 'pyridine' N atom of imidazole and the secondary N atom of dien occupy the apical sites. The Cu and imidazole-ring atoms are coplanar, with a maximum

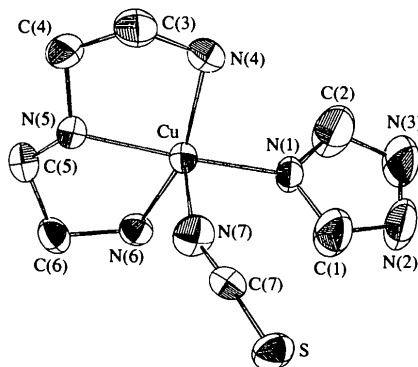


Fig. 1. The cation structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. Atoms N(2) and N(3) are both disordered (50:50% N:C).