

C4—C5—C6—C1	-4,0 (10)	C11'—C1'—C6'—C7'	72,2 (12)
C5—C6—C1—C2	7,1 (9)	C1'—C6'—C7'—C8'	-1,3 (11)
C1—C2—C3—C12	-179,7 (17)	C6'—C7'—C8'—C9'	-70,1 (13)
C11—C1—C6—C7	67,6 (10)	C7'—C8'—C9'—C10'	84,0 (17)
C1—C6—C7—C8	2,1 (9)	C8'—C9'—C10'—C11'	-68,1 (19)
C6—C7—C8—C9	-69,0 (11)	C9'—C10'—C11'—C1'	69,5 (19)
C7—C8—C9—C10	80,1 (12)	C10'—C11'—C1'—C6'	-83,7 (16)
C8—C9—C10—C11	-61,1 (12)	C7'—C8'—C9'—C10''	27,1 (16)
C9—C10—C11—C1	59,6 (12)	C8'—C9'—C10''—C11'	57,4 (21)
C10—C11—C1—C6	-79,2 (11)	C9'—C10''—C11'—C1'	-56,9 (21)
C5—C6—C7—C13	42,8 (10)	C10''—C11'—C1'—C6'	-29,0 (16)
C6'—C1'—C2'—C3'	-5,0 (11)	Pd1—C11—C12—Pd2	112,3 (2)
C1'—C2'—C3'—C4'	-4,4 (12)	C11—Pd1—Pd2—C11	119,4 (1)
C2'—C3'—C4'—C5'	12,2 (11)		

Cycle à sept chaînons du monomère lié à Pd2 désordonné (50%); conformation chaise ou bateau, C10' au dessus ou au dessous du plan des quatre atomes C1', C11', C8', C9' (C10', C14', C15' dédoublés en C10'', C14'' et C15''). Seuls les atomes de Pd et de Cl sont anisotropes. Pas d'atome d'hydrogène sur C9' et les atomes en désordre.

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope et des coordonnées des atomes d'hydrogène ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 55656: 11 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA1026]

Références

- Bäckvall, J.-E. & Björkman, E. E. (1982). *J. Chem. Soc. Chem. Commun.* pp. 693–694.
- Cambridge Structural Database (1989). Version 3.4. Cambridge Crystallographic Data Centre, Univ. Chemical Laboratory, Lensfield Road, Cambridge, Angleterre.
- El Firdoussi, L., Benharref, A., Karim, A., Castanet, Y., Mortreux, A. & Petit, F. (1992). *J. Mol. Catal.* Sous presse.
- Hansson, S., Heumann, A., Rein, T. & Åkermark, B. (1990). *J. Org. Chem.* **55**, 975–984.
- Hendrickson, J. B. (1961). *J. Am. Chem. Soc.* **83**, 4537–4547.
- Hütter, P., Butters, T., Winter, W., Handschuh, D. & Voelter, W. (1982). *Justus Liebigs Ann. Chem.* pp. 1111–1120.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, EU.
- Joseph, T. C. & Dev, S. (1968). *Tetrahedron*, **24**, 3841–3852.
- Larock, R. C., Song, H., Kim, S. & Jacobson, R. A. (1987). *J. Chem. Soc. Chem. Commun.* pp. 834–837.
- Minasyants, M. X. & Struchkov, Yu. T. (1971). *Arm. Khim. Zh.* **24**, 569–576.
- Riche, C. (1981). *PHIL. Program for Data Reduction for Philips PW1100 Diffractometer*. Institut de Chimie des Substances Naturelles du CNRS, France.
- Riche, C. (1983). *R3M*. Représentation et manipulation de modèles moléculaires. Institut de Chimie des Substances Naturelles du CNRS, France.
- Riche, C. (1992). *ACTACIF*. Logiciel de préparation des fichiers CIF. Institut de Chimie des Substances Naturelles du CNRS, France.
- Sheldrick, G. M. (1976). *SHELX76*. Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.
- Sheldrick, G. M. (1985). *SHELXS86*. Programme pour la solution des structures cristallines. Univ. de Göttingen, Allemagne.
- Soderberg, B. C., Åkermark, B. & Hall, S. S. (1988). *J. Org. Chem.* **53**, 2925–2937.
- Trost, B. M., Weber, L., Strege, P. E., Fullerton, T. J. & Dietsche, T. J. (1978). *J. Am. Chem. Soc.* **100**, 3416–3426.

Acta Cryst. (1993). **C49**, 368–370

Structure of [N-(2-Mercaptophenyl)-4-oxo-2-pentylideneamino-N,O,S]diphenyltin(IV)

ROSALINE C. OKECHUKWU

School of Chemical Sciences, Universiti Sains Malaysia, Minden, 11800 Penang, Malaysia

HOONG-KUN FUN

School of Physics, Universiti Sains Malaysia, Minden, 11800 Penang, Malaysia

SIANG-GUAN TEOH AND SOON-BENG TEO*

School of Chemical Sciences, Universiti Sains Malaysia, Minden, 11800 Penang, Malaysia

K. CHINNAKALI†

School of Physics, Universiti Sains Malaysia, Minden, 11800 Penang, Malaysia

(Received 11 May 1992; accepted 19 August 1992)

Abstract

The Sn atom adopts a five-coordinate distorted trigonal bipyramidal geometry with the N and phenyl C atoms C(6) and C(7) occupying equatorial positions, and O and S in axial positions. The Sn—S, Sn—N and Sn—O distances are 2.502 (1), 2.146 (2) and 2.128 (2) Å, respectively, while the O—Sn—S angle is 161.1 (1)°.

Comment

The reaction of diphenyltin(IV) chloride with 4-(2-benzothiazoliny)-2-pentanone results in the formation of the title compound. In this compound, the Sn atom displays a five-coordinate distorted trigonal bipyramidal arrangement in which the tridentate dianion binds to the Sn atom through the imine N atom, the S atom of the benzothiazoline ring and the O atom of the carbonyl group. The N atom of the ligand and the phenyl C atoms C(6) and C(7) are located at equatorial positions whereas the S and O atoms are at axial positions with an S—Sn—O angle of 161.1 (1)°. The dihedral angle between the two phenyl rings is 37.2 (1)°. The widening of the angle C(6)—Sn—C(7) [128.6 (1)°] is due to steric interactions between the two phenyl rings.

* Post Doctoral Research Fellow. Permanent address: Physics Division, Department of Chemical Engineering, AC Tech. Campus, Anna University, Madras 600025, India.

The benzene ring of the ligand is planar and N—C(16) [1.339 (4) Å] shows partial double-bond character. These structural features and the Sn—S, Sn—O, Sn—C and Sn—C distances are comparable with those observed in other diorganotin(IV) complexes (Yeap, Fun, Teo & Teoh, 1992; Teo, Teoh, Okechukwu & Wei, 1992).

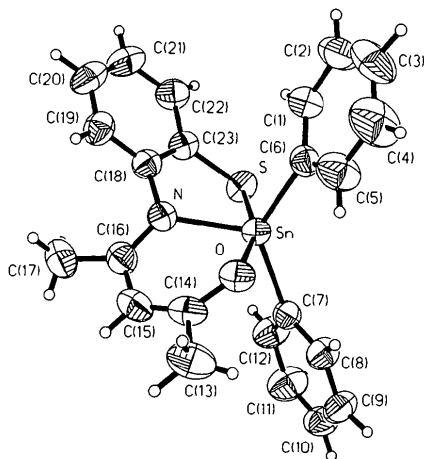


Fig. 1. View of the molecule showing the labelling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level.

Experimental

Crystal data

[Sn(C₁₁H₁₁NOS)(C₆H₅)₂]

$M_r = 478.2$

Monoclinic

$P2_1/c$

$a = 11.137 (3) \text{ \AA}$

$b = 16.016 (3) \text{ \AA}$

$c = 11.765 (3) \text{ \AA}$

$\beta = 97.35 (2)^\circ$

$V = 2081.3 (9) \text{ \AA}^3$

$Z = 4$

$D_x = 1.526 \text{ Mg m}^{-3}$

$D_m = 1.532 \text{ Mg m}^{-3}$

Data collection

Siemens P4 diffractometer

$2\theta/\theta$ scans

Absorption correction:

empirical

$T_{\min} = 0.61$, $T_{\max} = 0.87$

5017 measured reflections

4782 independent reflections

3741 observed reflections

$[F > 4.0\sigma(F)]$

$R_{\text{int}} = 0.0203$

Refinement

Refinement on F

Final $R = 0.0229$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 50 reflections

$\theta = 7.5\text{--}17.5^\circ$

$\mu = 1.340 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Plate

$5 \times 0.2 \times 0.1 \text{ mm}$

Yellow

$\theta_{\max} = 55.0^\circ$

$h = 0 \rightarrow 14$

$k = 0 \rightarrow 20$

$l = -15 \rightarrow 15$

2 standard reflections

monitored every 100

reflections

intensity variation: insignificant

$w = 1.0/[\sigma^2(F)]$

$(\Delta/\sigma)_{\max} = 0.003$

$wR = 0.0216$

$S = 2.01$

3741 reflections

328 parameters

All H-atom parameters refined

$\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors from SHELXTL/PC (Sheldrick, 1990)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Sn	0.2398 (1)	-0.0041	0.2206 (1)	0.041 (1)
S	0.1640 (1)	-0.1387 (1)	0.2926 (1)	0.053 (1)
O	0.3615 (2)	0.0914 (1)	0.1732 (1)	0.051 (1)
N	0.4071 (2)	-0.0596 (1)	0.2948 (2)	0.045 (1)
C(1)	0.1359 (3)	0.0591 (2)	0.4353 (3)	0.060 (1)
C(2)	0.0945 (3)	0.1171 (3)	0.5093 (3)	0.078 (1)
C(3)	0.0907 (4)	0.1984 (3)	0.4823 (4)	0.092 (2)
C(4)	0.1270 (5)	0.2244 (3)	0.3816 (5)	0.105 (2)
C(5)	0.1723 (3)	0.1678 (2)	0.3092 (3)	0.077 (2)
C(6)	0.1760 (2)	0.0841 (2)	0.3340 (2)	0.047 (1)
C(7)	0.1813 (2)	-0.0229 (2)	0.0431 (2)	0.044 (1)
C(8)	0.1609 (3)	0.0437 (2)	-0.0324 (2)	0.055 (1)
C(9)	0.1261 (3)	0.0296 (2)	-0.1485 (3)	0.065 (1)
C(10)	0.1145 (3)	-0.0496 (2)	-0.1906 (3)	0.065 (1)
C(11)	0.1339 (3)	-0.1159 (2)	-0.1171 (3)	0.076 (1)
C(12)	0.1658 (3)	-0.1019 (2)	-0.0007 (3)	0.064 (1)
C(13)	0.5147 (4)	0.1438 (3)	0.0707 (4)	0.076 (2)
C(14)	0.4641 (3)	0.0752 (2)	0.1379 (2)	0.054 (1)
C(15)	0.5283 (3)	0.0027 (2)	0.1622 (3)	0.059 (1)
C(16)	0.5068 (3)	-0.0586 (2)	0.2421 (2)	0.054 (1)
C(17)	0.6017 (4)	-0.1261 (3)	0.2653 (4)	0.075 (2)
C(18)	0.3964 (2)	-0.1072 (2)	0.3946 (2)	0.046 (1)
C(19)	0.4884 (3)	-0.1113 (2)	0.4863 (3)	0.057 (1)
C(20)	0.4717 (3)	-0.1537 (2)	0.5845 (3)	0.068 (1)
C(21)	0.3614 (3)	-0.1894 (2)	0.5959 (3)	0.069 (1)
C(22)	0.2681 (3)	-0.1829 (2)	0.5081 (3)	0.058 (1)
C(23)	0.2841 (2)	-0.1436 (2)	0.4054 (2)	0.047 (1)

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

Sn—S	2.502 (1)	Sn—O	2.165 (2)
Sn—N	2.146 (2)	Sn—C(6)	2.127 (3)
Sn—C(7)	2.128 (2)	S—C(23)	1.761 (3)
O—C(14)	1.290 (4)	N—C(16)	1.339 (4)
N—C(18)	1.418 (3)	C(1)—C(2)	1.392 (5)
C(1)—C(6)	1.385 (4)	C(2)—C(3)	1.340 (6)
C(3)—C(4)	1.364 (7)	C(4)—C(5)	1.383 (6)
C(5)—C(6)	1.370 (4)	C(7)—C(8)	1.387 (4)
C(7)—C(12)	1.370 (4)	C(8)—C(9)	1.390 (4)
C(9)—C(10)	1.363 (5)	C(10)—C(11)	1.369 (5)
C(11)—C(12)	1.388 (5)	C(13)—C(14)	1.504 (5)
C(14)—C(15)	1.374 (4)	C(15)—C(16)	1.401 (4)
C(16)—C(17)	1.513 (5)	C(18)—C(19)	1.391 (4)
C(18)—C(23)	1.400 (4)	C(19)—C(20)	1.373 (4)
C(20)—C(21)	1.378 (5)	C(21)—C(22)	1.372 (4)
C(22)—C(23)	1.394 (4)		
S—Sn—O	161.1 (1)	S—Sn—N	79.5 (1)
O—Sn—N	81.8 (1)	S—Sn—C(6)	101.9 (1)
O—Sn—C(6)	87.9 (1)	N—Sn—C(6)	111.3 (1)
S—Sn—C(7)	97.8 (1)	O—Sn—C(7)	88.3 (1)
N—Sn—C(7)	118.8 (1)	C(6)—Sn—C(7)	128.6 (1)
Sn—S—C(23)	92.1 (1)	Sn—O—C(14)	123.4 (2)
Sn—N—C(16)	122.3 (2)	Sn—N—C(18)	113.5 (2)
C(16)—N—C(18)	123.5 (2)	C(2)—C(1)—C(6)	120.9 (3)
C(1)—C(2)—C(3)	120.2 (4)	C(2)—C(3)—C(4)	119.9 (4)
C(3)—C(4)—C(5)	120.4 (4)	C(4)—C(5)—C(6)	120.9 (4)
Sn—C(6)—C(1)	121.2 (2)	Sn—C(6)—C(5)	121.3 (2)
C(1)—C(6)—C(5)	117.6 (3)	Sn—C(7)—C(8)	121.6 (2)
Sn—C(7)—C(12)	120.5 (2)	C(8)—C(7)—C(12)	117.8 (2)
C(7)—C(8)—C(9)	120.5 (3)	C(8)—C(9)—C(10)	120.6 (3)
C(9)—C(10)—C(11)	119.5 (3)	C(10)—C(11)—C(12)	119.9 (3)
C(7)—C(12)—C(11)	121.6 (3)	O—C(14)—C(13)	115.4 (3)
O—C(14)—C(15)	124.2 (3)	C(13)—C(14)—C(15)	120.4 (3)

C(14)—C(15)—C(16)	127.0 (3)	N—C(16)—C(15)	122.8 (3)
N—C(16)—C(17)	120.6 (3)	C(15)—C(16)—C(17)	116.5 (3)
N—C(18)—C(19)	122.4 (2)	N—C(18)—C(23)	118.0 (2)
C(19)—C(18)—C(23)	119.2 (2)	C(18)—C(19)—C(20)	120.7 (3)
C(19)—C(20)—C(21)	120.4 (3)	C(20)—C(21)—C(22)	119.6 (3)
C(21)—C(22)—C(23)	121.2 (3)	S—C(23)—C(18)	120.9 (2)
S—C(23)—C(22)	120.3 (2)	C(18)—C(23)—C(22)	118.8 (2)

The density was measured by flotation in a mixture of chloroform and bromoform. The data were collected using a variable scan speed of $5.33\text{--}29.3^\circ \text{ min}^{-1}$ in ω . The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* was used for all calculations.

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grants R&D Nos. 123-3203-2504 and 123-3417-2201.

Lists of structure factors, anisotropic thermal parameters, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55478 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1015]

References

- Sheldrick, G. M. (1990). *SHELXTL/PC*. Release 4.1. Siemens Crystallographic Research Systems, Madison, Wisconsin, USA.
 Teo, S. B., Teoh, S. G., Okechukwu, R. C. & Wei, C. (1992). *J. Coord. Chem.* In the press.
 Yeap, G. Y., Fun, H. K., Teo, S. B. & Teoh, S. G. (1992). *Acta Cryst.* **C48**, 1109-1110.

Acta Cryst. (1993). **C49**, 370-372

Structure of [Cu(CN)(py)₂]

M. M. OLMSTEAD*

Department of Chemistry, University of California, Davis, California 95616, USA

G. SPEIER AND L. SZABÓ

Department of Organic Chemistry, University of Veszprém, 8201 Veszprém, Schönherz u.8, Hungary

(Received 12 June 1992; accepted 2 September 1992)

Abstract

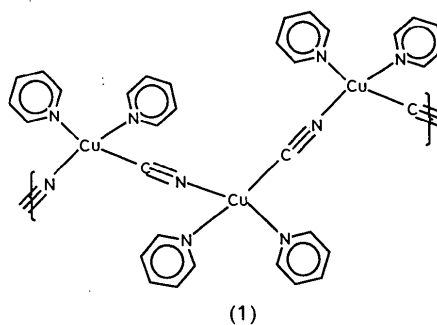
catena-Poly[$\{\text{bis}(\text{pyridine-}N\text{)copper(I)}\}\text{-}\mu\text{-cyano-N:C}$] crystallizes with two independent polymeric chains in the structure. The chains are propagated by —CN— groups that are N-bound and C-bound to Cu^I centres with no apparent disorder. Each Cu

bound to two pyridines, the N atom of one CN and the C atom of another. The coordination geometry is distorted tetrahedral with particularly wide angles involving the cyanide groups.

Comment

Although the compound [Cu(CN)(py)₂] probably has been known since before the turn of the century, its structure never appears to have been reported. Werner (1897) understood that it was probably not monomeric, based on solubility considerations. The polymeric nature of a related structure, [Cu(CN)(bpy)], was deduced by Cooper & Plane (1966) based on solubility and IR considerations. However, the considerable variety found in the structures and stoichiometries of copper(I) cyanides (Roof, Larson & Cromer, 1968; Cromer & Larson, 1962; Cromer, 1957) and of their Lewis-base adducts (Cromer, Larson & Roof, 1965, 1966; Williams, Cromer & Larson 1971; Goher & Drátovski, 1976) is ample proof that a crystal structure determination is necessary to know the structure with certainty.

Recently, the compound [Cu(CN)(py)₂] (1) was reported to be a product in the reaction between metallic copper, oxygen and nitromethane in pyridine (Gargano, Ravasio, Rossi, Tiripicchio & Camellini, 1989), as well as from heating a mixture of copper(II) acetate, acetic anhydride and formamide in pyridine (Connor, Gibson & Price, 1986, 1987). Evidently its synthesis can be approached from any of the common oxidation states of copper. In the present case it was formed from the decomposition of tetracyanoethylene during the reaction of metallic copper and tetracyanoethylene in pyridine.



The structure of [Cu(CN)(py)₂] consists of two separate polymeric spiral chains of $[-\text{C}-\text{N}-\text{Cu}(\text{py})_2-\text{C}-\text{N}-\text{Cu}(\text{py})_2-]$. A portion of these chains is depicted in Fig. 1. Although the geometry at copper can best be described as tetrahedral, there is a significant widening of the angle involving the cyanides. At Cu(1) this N—Cu—C angle is $131.3(2)^\circ$ while at Cu(2) it is $128.3(2)^\circ$. The spiral chain motif with wide N—Cu—C angles of *ca* 133° is common to