$V = 1306.9\,(2)$ Å^3
$Z = 4$
$D_x = 1.926$ Mg m^{-3}
$D_m = 1.94$ Mg m^{-3}
D_m measured by floatation
0.47 × 0.36 × 0.25 mm
Yellow

Data collection

Siemens *P*3*m*/*V* diffractometer
ω scans
Absorption correction: analytical
$T_{\min} = 0.606$, $T_{\max} = 0.701$
2080 measured reflections
1900 independent reflections
1524 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.0097$
$\theta_{\max} = 30°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 15$
$l = -13 \rightarrow 13$
4 standard reflections monitored every 96 reflections intensity decay: 1%

Refinement

Refinement on F
$R = 0.0456$
$wR = 0.0584$
$S = 2.06$
1524 reflections
113 parameters
H atoms riding
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.99$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ag	0	1/2	0	0.0738 (2)
O	0	0.4297 (3)	1/4	0.083 (2)
N	−0.0600 (2)	0.3156 (3)	0.0100 (2)	0.0538 (9)
C2	−0.1087 (4)	0.2609 (5)	−0.0997 (4)	0.0760 (15)
C3	−0.1541 (5)	0.1486 (6)	−0.0960 (6)	0.095 (2)
C4	−0.1519 (4)	0.0903 (5)	0.0238 (6)	0.088 (2)
C5	−0.1008 (4)	0.1424 (3)	0.1404 (4)	0.0665 (13)
C6	−0.0535 (3)	0.2546 (3)	0.1299 (3)	0.0454 (8)
C7	0	0.3201 (3)	1/4	0.0463 (12)
B	1/4	1/4	0	0.083 (3)
F1	0.3036 (9)	0.2937 (11)	−0.0843 (10)	0.159 (5)
F2	0.1471 (6)	0.1914 (6)	−0.0649 (7)	0.105 (3)
F3	0.2007 (5)	0.3287 (6)	0.0941 (6)	0.092 (2)
F4	0.2929 (6)	0.1536 (6)	0.0851 (6)	0.103 (2)

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

Ag—O	2.552 (1)	C2—C3	1.366 (8)
Ag—N	2.180 (3)	C3—C4	1.331 (8)
O—C7	1.217 (5)	C4—C5	1.381 (7)
N—C2	1.330 (5)	C5—C6	1.380 (5)
N—C6	1.345 (4)	C6—C7	1.486 (3)
O—Ag—N	69.35 (9)	C5—C4—C3	119.5
O—Ag—N$^{\rm i}$	110.65 (9)	C6—C5—C4	119.0 (4)
C7—O—Ag	107.85 (7)	C7—C6—N	115.4 (3)
C2—N—C6	117.4 (3)	C7—C6—C5	123.1 (3)
C2—N—Ag	121.7 (3)	N—C6—C5	121.3 (3)
C6—N—Ag	120.8 (2)	O—C7—C6	119.3 (2)
C3—C2—N	123.7 (4)	C6—C7—C6$^{\rm ii}$	121.3 (3)
C4—C3—C2	119.0 (5)		

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $-x, y, \frac{1}{2}-z$.

The H atoms were placed in calculated idealized positions (C—H 0.96 Å) and given fixed displacement parameters (0.08 Å^2).

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

This research was supported by a Cottrell College Science Award of the Research Corporation, Tucson, Arizona, USA.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1132). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Aoyama, T., Yamanaka, M., Ohba, S. & Saito, Y. (1990). *Acta Cryst.* C**46**, 762–766.

Colsman, M. R., Newbound, T. D., Marshall, L. J., Noirot, M. D., Miller, M. M., Wulfsberg, G. P., Frye, J. S., Anderson, O. P. & Strauss, S. H. (1990). *J. Am. Chem. Soc.* **112**, 2349–2362.

Housecroft, C. E. (1992). *Coord. Chem. Rev.* **115**, 141–161.

Nilsson, K. & Oskarsson, A. (1982). *Acta Chem. Scand. Ser. A*, **36**, 605–610.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.

Sommerer, S. O., Westcott, B. L. & Abboud, K. A. (1994). *Acta Cryst.* C**50**, 48–52.

Acta Cryst. (1996). C**52**, 1428–1430

Polymeric [*p*-(2-Hydroxy-5-methylphenylazo)benzoato]trimethyltin

TUSHAR S. BASU BAUL† AND EDWARD R. T. TIEKINK*

Department of Chemistry, The University of Adelaide, South Australia 5005, Australia. E-mail: etiekink@chemistry.adelaide.edu.au

(*Received 2 January 1996; accepted 9 February 1996*)

Abstract

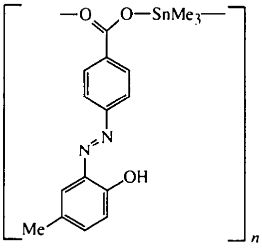
The presence of bidentate bridging carboxylate ligands leads to the formation of a polymeric structure in the title compound, *catena*-poly[trimethyltin-μ-[*p*-(2-hydroxy-5-methylphenylazo)benzoato-*O*:*O*′]], [Sn(C$_{14}$H$_{11}$N$_2$O$_3$)(CH$_3$)$_3$]$_n$. The Sn atom is five-coordinate and has distorted trigonal bipyramidal geometry with the three methyl groups defining the trigonal plane.

---

† Permanent address: Chemical Laboratory, Regional Sophisticated Instrumentation Centre, North-Eastern Hill University, Bijni Complex, Shillong 793 003, India.

Comment

The structure of the title compound, $[\mathrm{Me_3Sn\{O_2CC_6H_4(N{=}NC_6H_3\text{-}2\text{-}OH\text{-}5\text{-}Me)\text{-}}p\}]_n$, (I), is polymeric owing to the presence of bidentate-bridging carboxylate ligands. The intramolecular separation of 2.139 (3) Å is significantly shorter than the intermolecular Sn—O(2^{i}) distance of 2.497 (3) Å [symmetry code: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$]. The asymmetric Sn—O separations are reflected in the associated C(1)—O(1) and C(1)—O(2) distances of 1.283 (5) and 1.241 (4) Å, respectively. The geometry about the Sn atom is distorted trigonal bipyramidal with the three methyl groups occupying equatorial positions; the O(1)—Sn—O(2^{i}) angle is 174.3 (1)° and the Sn atom lies 0.1766 (3) Å out of the plane in the direction of the O(1) atom.



(I)

The carboxylate ligand is essentially planar, as evidenced by the O(1)—C(1)—C(2)—C(3) and C(5)—N(1)—N(2)—C(8) torsion angles of −176.4 (4) and −178.6 (3)°, respectively. Within the ligand, there is a close N(1)···H(O3) contact of 1.59 Å.

The structure found here for (I) is one of the two major motifs found for structures having the general formula $[R_3\mathrm{Sn(O_2C}R')]$, *i.e.* the *trans*-$R_3\mathrm{SnO_2}$ motif (Tiekink, 1994). The other motif features a monomeric structure with an essentially four-coordinate Sn atom, depending on the magnitude of the second Sn···O interaction.

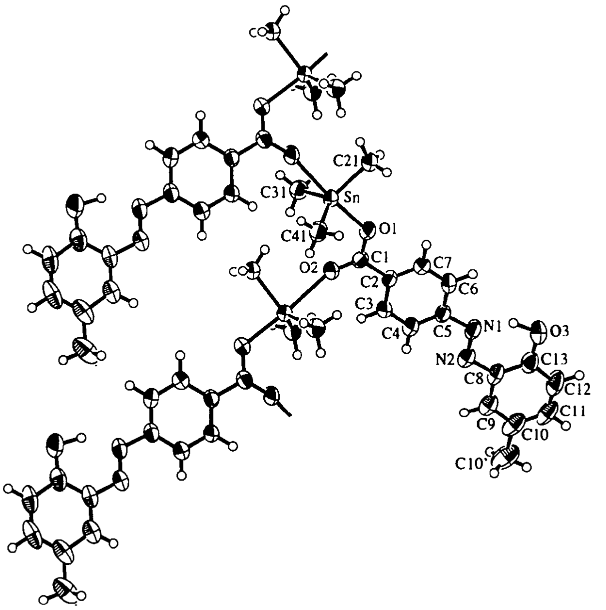


Fig. 1. The polymeric structure of $[\mathrm{Me_3Sn\{O_2CC_6H_4(N{=}NC_6H_3\text{-}2\text{-}OH\text{-}5\text{-}Me)\text{-}}p\}]_n$ shown with 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

Experimental

Crystals of (I) were prepared from the equimolar reaction of sodium *p*-(2-hydroxy-5-methylphenylazo)benzoate and $\mathrm{Me_3SnCl}$ in methanol under reflux conditions for 2 h. The solid obtained after removal of the solvent was washed with petroleum ether (313–333 K) and recrystallized from chloroform solution. Crystals for the X-ray study were grown by the slow evaporation of a chloroform/ethanol solution (1/1); 79% yield of red crystals, m.p. 465–466 K.

Crystal data

$[\mathrm{Sn(C_{14}H_{11}N_2O_3)(CH_3)_3}]$
$M_r = 419.05$
Monoclinic
$C2/c$
$a = 13.165\ (3)$ Å
$b = 9.530\ (5)$ Å
$c = 28.924\ (2)$ Å
$\beta = 98.27\ (1)°$
$V = 3591\ (1)$ Å^3
$Z = 8$
$D_x = 1.55$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 21.6$–$23.9°$
$\mu = 1.438$ mm^{-1}
$T = 293$ K
Block
0.39 × 0.16 × 0.14 mm
Red

Data collection

Rigaku AFC-6*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical (De Meulenaer & Tompa, 1965)
$T_{\min} = 0.627$, $T_{\max} = 0.825$
4605 measured reflections
4160 independent reflections

4160 observed reflections [all reflections used]
$R_{\mathrm{int}} = 0.0356$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 12$
$l = -37 \rightarrow 37$
3 standard reflections monitored every 400 reflections intensity decay: 0.58%

Refinement

Refinement on F^2
$R(F) = 0.0639$
$wR(F^2) = 0.0848$
$S = 1.572$
4160 reflections
208 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{\max} = 0.0007$
$\Delta\rho_{\max} = 0.95$ e Å^{-3}
$\Delta\rho_{\min} = -0.64$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn	0.29093 (2)	0.29113 (3)	0.23552 (1)	0.04819 (8)
O(1)	0.3224 (2)	0.1550 (3)	0.18053 (9)	0.0563 (9)
O(2)	0.2442 (2)	−0.0300 (3)	0.20570 (9)	0.0581 (9)

O(3)	0.4773 (3)	−0.2629 (4)	−0.0576 (1)	0.083 (1)
N(1)	0.3901 (3)	−0.2640 (4)	0.0168 (1)	0.053 (1)
N(2)	0.3595 (3)	−0.3885 (4)	0.0111 (1)	0.053 (1)
C(1)	0.2939 (3)	0.0262 (4)	0.1774 (1)	0.046 (1)
C(2)	0.3226 (3)	−0.0526 (4)	0.1364 (1)	0.044 (1)
C(3)	0.2904 (3)	−0.1907 (4)	0.1282 (1)	0.054 (1)
C(4)	0.3123 (4)	−0.2635 (5)	0.0898 (1)	0.057 (1)
C(5)	0.3655 (3)	−0.1980 (5)	0.0582 (1)	0.049 (1)
C(6)	0.3989 (3)	−0.0614 (5)	0.0661 (1)	0.058 (1)
C(7)	0.3771 (3)	0.0111 (4)	0.1050 (1)	0.054 (1)
C(8)	0.3854 (3)	−0.4552 (5)	−0.0289 (1)	0.054 (1)
C(9)	0.3512 (3)	−0.5928 (5)	−0.0348 (1)	0.063 (1)
C(10)	0.3707 (4)	−0.6756 (6)	−0.0720 (2)	0.073 (2)
C(10′)	0.3313 (5)	−0.8228 (7)	−0.0783 (2)	0.100 (2)
C(11)	0.4277 (4)	−0.6137 (7)	−0.1033 (2)	0.078 (2)
C(12)	0.4631 (4)	−0.4783 (7)	−0.0983 (2)	0.078 (2)
C(13)	0.4427 (4)	−0.3952 (6)	−0.0612 (1)	0.063 (1)
C(21)	0.3601 (4)	0.4560 (5)	0.2023 (2)	0.065 (1)
C(31)	0.1277 (3)	0.2779 (5)	0.2245 (2)	0.059 (1)
C(41)	0.3866 (3)	0.1874 (5)	0.2902 (1)	0.063 (1)

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

Sn—O(1)	2.139 (3)	O(2)—C(1)	1.241 (4)
Sn—O(2^i)	2.497 (3)	O(3)—C(13)	1.340 (6)
Sn—C(21)	2.115 (4)	N(1)—N(2)	1.255 (5)
Sn—C(31)	2.131 (4)	N(1)—C(5)	1.430 (5)
Sn—C(41)	2.119 (4)	N(2)—C(8)	1.405 (5)
O(1)—C(1)	1.283 (5)	C(1)—C(2)	1.497 (5)
O(1)—Sn—O(2^i)	174.3 (1)	C(31)—Sn—C(41)	124.2 (2)
O(1)—Sn—C(21)	88.3 (1)	Sn—O(1)—C(1)	123.2 (2)
O(1)—Sn—C(31)	98.7 (1)	Sn—O(2)—C(1)	155.6 (3)
O(1)—Sn—C(41)	96.5 (1)	N(2)—N(1)—C(5)	114.8 (3)
O(2)—Sn—C(21)	86.3 (1)	N(1)—N(2)—C(8)	115.1 (4)
O(2)—Sn—C(31)	82.0 (1)	O(1)—C(1)—O(2)	122.8 (4)
O(2)—Sn—C(41)	87.7 (1)	O(1)—C(1)—C(2)	115.6 (3)
C(21)—Sn—C(31)	117.9 (2)	O(2)—C(1)—C(2)	121.6 (4)
C(21)—Sn—C(41)	115.9 (2)		

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to refine structure: *TEXSAN* (all reflection data used). Software used to prepare material for publication: *TEXSAN*.

The authors thank the Department of Science and Technology (India) for a BOYSCAST fellowship and the North-Eastern Hill University, Shillong, for support (TSBB). The Australian Research Council is thanked for support of the crystallographic facility.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1087). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

De Meulenaer, J. & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1018.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Tiekink, E. R. T. (1994). *Trends Organomet. Chem.* **1**, 71–116.

Acta Cryst. (1996). C**52**, 1430–1436

Les Composés de la Metformine avec les Ions Co^{II}, Cu^{II} et Ni^{II}

PASCALE LEMOINE,[a] MOHAMED CHIADMI,[a] VÉRONIQUE BISSERY,[a] ALAIN TOMAS[a] ET BERNARD VIOSSAT[b]

[a]*Laboratoire de Physique, Faculté des Sciences Pharmaceutiques et Biologiques de Paris V, 4 avenue de l'Observatoire, 75270 Paris CEDEX 06, France, et* [b]*Laboratoire de Chimie Générale, UFR de Médecine et de Pharmacie, 34 rue du Jardin des Plantes, BP199, 86005 Poitiers CEDEX, France*

(*Reçu le 24 mars 1995, accepté le 21 juin 1995*)

Abstract

The structures of trichloro(*N*,*N*-dimethylbiguanidium)cobalt(II), $[Co(C_4H_{12}N_5)Cl_3]$, (I), and di-μ-chlorobis[chloro-(*N*,*N*-dimethylbiguanide)copper(II)], $[CuCl_2(C_4H_{11}N_5)]_2$, (II), consist of the metal atom bound to three Cl atoms and to one or two N atoms, respectively, belonging to one metformine ligand. The structures of bis(*N*,*N*-dimethylbiguanide)copper(II) dichloride dihydrate $[Cu(C_4H_{11}N_5)_2]Cl_2.2H_2O$, (III), and bis(*N*,*N*-dimethylbiguanide)nickel(II) monohydroxide chloride, $[Ni(C_4H_{11}N_5)_2](Cl)(OH)$, (IV), are formed of $[M(\text{metformine})_2]^{2+}$ (M = Cu or Ni) and anions (Cl^- or OH^-). In these compounds, the metal is on a centre of symmetry and forms a square-planar structure comprising four N atoms of two *N*,*N*-dimethylbiguanidine ligands. The metformine ligand is planar in (II), (III) and (IV), allowing delocalization of the π-electron system in the metal–ligand ring, but not in (I), where the dihedral angle between the two guanidine groups is 68.4 (2)°. The four structures are stabilized by hydrogen bonding and van der Waals interactions.

Commentaire

La metformine (*N*,*N*-diméthylbiguanide) présente comme plusieurs dérivés de la biguanide, une importante activité biologique. Elle est utilisée comme antidiabétique, analgésique et dans la lutte contre la malaria (Pignard, 1962). Par ailleurs, elle se comporte comme un antimétabolite spécifique pour les microorganismes qui inhibent le métabolisme de l'acide folique (Siest, Roos & Gabou, 1963).

Comme la biguanide et ses dérivés N-substitués, la metformine est un ligand susceptible de constituer des composés de coordination avec des ions de transition (Ray, 1961). Suivant le mode de préparation, en particulier selon l'acidité du milieu, différents complexes peuvent être synthétisés. Nous avons précédemment décrit le tétrachlorocuprate de metformine (Lemoine, Tomas, Viossat & Nguyen-Huy Dung, 1994) obtenu