Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Bis[*N*,*N*-bis(2-hydroxyethyl)dithiocarbamato]dimethyltin(IV)

A. Aziz Yang Farina,[a] Abdul Hamid Othman,[a]* Ibrahim Baba,[a] K. Sivakumar,[b] Hoong-Kun Fun[b] and Seik Weng Ng[c]

[a]School of Chemical Science and Food Technology, Universiti Kebangsaan Malaysia, 46300 Bangi, Malaysia, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 Penang, Malaysia, and [c]Institute of Postgraduate Studies and Research, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: hamie@ukm.edu.my

Received 31 January 2000
Accepted 9 February 2000

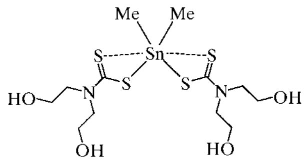
Data validation number: IUC0000042

The title compound, $[Sn(CH_3)_2(C_5H_{10}NO_2S_2)_2]$, has crystallographic mirror symmetry (C—Sn—C on mirror plane) and the coordination polyhedron around the Sn atom is a tetrahedron [C—Sn—C 139.3 (2)° and S—Sn—S 82.3 (1)°] distorted towards a skew-trapezoidal bipyramid owing to an intramolecular Sn···S contact [3.0427 (6) Å]. The molecules are linked into a linear chain by intermolecular O—H···O hydrogen bonds [O···O 2.646 (3) Å].

Comment

The structures of a number of diorganotin(IV) bis(*N*,*N*-dithiocarbamates) have been reported. Among the dimethyltin derivatives, the dimethyldithiocarbamate (Kimura *et al.*, 1972), the three modifications of the diethyldithiocarbamate (Lockhart *et al.*, 1986; Morris & Schlemper, 1979) and the tetramethylenedithiocarbamate (Lockhart *et al.*, 1985) have a dimethyltin skeleton is opened up to about 135°, so that the geometry is not a strict tetrahedron. The geometries of these and other bis(chelated) diorganotin compounds having such a bent skeleton have been described in terms of a skew-trapezoidal bipyramid (Ng *et al.*, 1987). The dithiocarbamates display an intramolecular tin–sulfur interaction of about 3 Å, which is only about 0.5 Å longer than the covalent tin–sulfur distances. On the other hand, for the trimethyltin derivatives, as the interaction is much longer, the dithiocarbamate group is essentially monodentate (Lokaj *et al.*, 1986).

The introduction of two hydroxy substituents in the diethyldithiocarbamato ligand results in the title compound, (I), whose molecules are stacked as a linear chain along the *a* axis. The component molecules are held together by strong hydrogen bonds; the intermolecular hydrogen bond [O···O 2.646 (3) Å and O—H···O 170 (3)°] is shorter than the intramolecular hydrogen bond [O···O 2.683 (3) Å and O—H···O 172 (4)°]. In the tellurium(II) and diiodotellurium(IV) derivatives, the two hydroxy groups of each dithiocarbamate ligand are not linked; instead, the four hydroxy groups of one molecule are linked to the the hydroxy groups of adjacent molecules, but the hydrogen bonds are much weaker (Appa Rao *et al.*, 1983; Rout *et al.*, 1983). Similarly, only intermolecular molecular hydrogen bonds are found in the nickel [O···O 2.707 (5) and 2.711 (5) Å; Ramalingam *et al.*, 1984] and copper [O···O 2.412 (2) Å; Radha *et al.*, 1985] derivatives.



(I)

Experimental

A solution of carbon disulfide (1.5 ml) in methanol (5 ml) was added to a methanol solution (100 ml) of a mixture of dimethyltin dichloride (1.1 g, 5 mmol) and ethanolamine (3 ml, 31 mmol) in methanol (100 ml). Both solutions were initially cooled to 273 K and the carbon disulfide solution was added at a rate such that the temperature of the reaction mixture did not rise above 273 K. The yellow product that separated from the stirred mixture after several hours of stirring was collected and recrystallized from methanol to afford yellow crystals of the dimethyltin complex. The crystal used in the study was the only one suitable for the diffraction measurements. Because of the relatively large size, it was likely that the crystal would not be bathed in the X-ray radiation during a part of the measurements. However, the error was deemed to be minor as the structure was well behaved in the refinements.

Crystal data

$[Sn(CH_3)_2(C_5H_{10}NO_2S_2)_2]$
$M_r = 509.28$
Orthorhombic, *Pnma*
$a = 7.251$ (1) Å
$b = 28.761$ (3) Å
$c = 9.635$ (1) Å
$V = 2009.3$ (4) Å^3
$Z = 4$
$D_x = 1.683$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 46 reflections
$\theta = 5.4$–12.5°
$\mu = 1.704$ mm^{-1}
$T = 298$ (2) K
Irregular block, colorless
0.70 × 0.56 × 0.48 mm

Data collection

Siemens *P*4 diffractometer
ω–2θ scans
Absorption correction: empirical ψ scan (North *et al.*, 1968)
$T_{min} = 0.580$, $T_{max} = 0.606$
3859 measured reflections
2964 independent reflections
2457 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$
$\theta_{max} = 30°$
$h = -1 \rightarrow 10$
$k = -40 \rightarrow 1$
$l = -1 \rightarrow 13$
3 standard reflections every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.049$
2964 reflections
118 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0360P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0134 (5)

The hydroxyl H atoms were located and refined.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

We thank the National Science Council for R&D and Universiti Sains Malaysia for supporting this work (R&D 305/pfizik/622004 and IRPA 09–02–02–0010).

References

Appa Rao, G. V. N., Seshasayee, M., Aravanudan, G. & Radha, K. (1983). *Acta Cryst.* C**39**, 1018–1020.

Kimura, T., Yasuoka, N., Kasai, N. & Kakudo, M. (1972). *Bull. Chem. Soc. Jpn*, **45**, 1649–1654.

Lockhart, T. P., Manders, W. F. & Schlemper, E. O. (1985). *J. Am. Chem. Soc.* **107**, 7451–7453.

Lockhart, T. P., Manders, W. F., Schlemper, E. O. & Zuckerman, J. J. (1986). *J. Am. Chem. Soc.* **108**, 4074–4078.

Lokaj, J., Kellö, E., Kettmann, V., Vrabel, V. & Rattay, V. (1986). *Collect. Czech. Chem. Commun.* **51**, 2521–2527.

Morris, J. S. & Schlemper, E. O. (1979). *J. Cryst. Mol. Struct.* **9**, 13–31.

Ng, S. W., Chen, W., Kumar Das, V. G. & Mak, T. C. W. (1987). *J. Organomet. Chem.* **334**, 295–305.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Radha, A., Seshasayee, M., Radha, K., Aravamudan, G. & Subramanyam, ch. (1985). *Acta Cryst.* C**41**, 1166–1169.

Ramalingam, K., Radha, K., Aravamudan, G., Mahadevan, C., Subramanyam, ch. & Seshasayee, M. (1984). *Acta Cryst.* C**40**, 1838–1839.

Rout, G. C., Seshasayee, M., Radha, K. & Aravamudan, G. (1983). *Acta Cryst.* C39, 1021–1023.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Siemens (1994). *XSCANS User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (2000). C**56**, e84–e85 A. Aziz Yang Farina *et al.* • $[Sn(CH_3)_2(C_5H_{10}NO_2S_2)_2]$ **e85**