Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Redetermination of L-(–)-dichloro(*N*,*N*-diethyldithiocarbamato)(*β*-menthoxycarbonylethyl)tin(IV)

Seik Weng Ng

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 18 April 2005
Accepted 22 April 2005
Online 7 May 2005

Key indicators

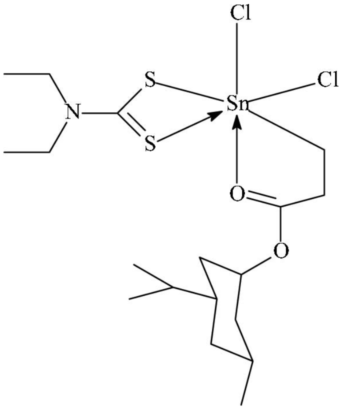
Single-crystal X-ray study
T = 295 K
Mean *σ*(C–C) = 0.007 Å
R factor = 0.035
wR factor = 0.079
Data-to-parameter ratio = 24.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the chiral title compound, $[Sn(C_5H_{10}NS_2)(C_{13}H_{23}O_2)Cl_2]$, has been determined in space group $P2_12_12_1$. The Sn atom is in a distorted octahedral geometry within a CCl_2OS_2 donor set.

Comment

The title compound, (I), which has an optically active menthyl group, has been reported previously with two independent molecules in the monoclinic space group $P2_1$ (Tian *et al.*, 2005). New data are presented for this compound here, with very similar unit-cell dimensions to the published structure. In space group $P2_12_12_1$, there is only one independent molecule. The Sn atom is chelated by a dithiocarbamate group, as well as by the organyl substituent and two Cl atoms, the donor set defining an octahedral environment (Fig. 1 and Table 1).



(I)

Experimental

The title compound was synthesized as described by Tian *et al.* (2005).

Crystal data

$[Sn(C_5H_{10}NS_2)(C_{13}H_{23}O_2)Cl_2]$
M_r = 549.16
Orthorhombic, $P2_12_12_1$
a = 10.305 (1) Å
b = 12.088 (1) Å
c = 20.341 (2) Å
V = 2533.8 (4) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 5645 reflections
θ = 2.6–24.7°
μ = 1.40 mm^{-1}
T = 295 (2) K
Irregular block, colourless
0.27 × 0.12 × 0.09 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.675, T_{max} = 0.885
15 820 measured reflections

5665 independent reflections
5005 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.5°
$h = -13 \rightarrow 12$
$k = -15 \rightarrow 14$
$l = -23 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.079$
$S = 1.00$
5665 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.52$ e Å^{-3}
$\Delta\rho_{\min} = -0.63$ e Å^{-3}
Absolute structure: Flack (1983), with 2417 Friedel pairs
Flack parameter = −0.02 (2)

Table 1
Selected geometric parameters (Å, °).

Sn1—C6	2.135 (4)	Sn1—S2	2.693 (1)
Sn1—O1	2.374 (3)	Sn1—Cl1	2.399 (1)
Sn1—S1	2.462 (1)	Sn1—Cl2	2.405 (1)
C6—Sn1—O1	75.6 (1)	O1—Sn1—Cl2	84.63 (8)
C6—Sn1—S1	152.4 (1)	S1—Sn1—S2	69.50 (3)
C6—Sn1—S2	91.2 (1)	S1—Sn1—Cl1	98.98 (4)
C6—Sn1—Cl1	102.1 (1)	S1—Sn1—Cl2	90.83 (4)
C6—Sn1—Cl2	105.3 (1)	S2—Sn1—Cl1	94.43 (4)
O1—Sn1—S1	84.02 (7)	S2—Sn1—Cl2	159.72 (4)
O1—Sn1—S2	88.32 (8)	Cl1—Sn1—Cl2	93.47 (4)
O1—Sn1—Cl1	176.49 (8)		

H atoms were placed in calculated positions (C—H = 0.98 Å for the methine H atoms, 0.97 Å for the methylene H atoms and 0.96 Å for the methyl H atoms) and were included in the refinement in the riding-model approximation, with $U_{\rm iso}$(H) values set at $1.2U_{\rm eq}$(C) for the methine and methylene H atoms, and at $1.5U_{\rm eq}$(C) for the methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; method used to solve structure: averaging the two molecules described in $P2_1$ (Tian *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The author thanks Professor Lai-Jin Tian of Qufu Normal University for synthesizing the crystal used in the diffraction measurements, as well as for collecting the diffraction data, and the University of Malaya for supporting this study.

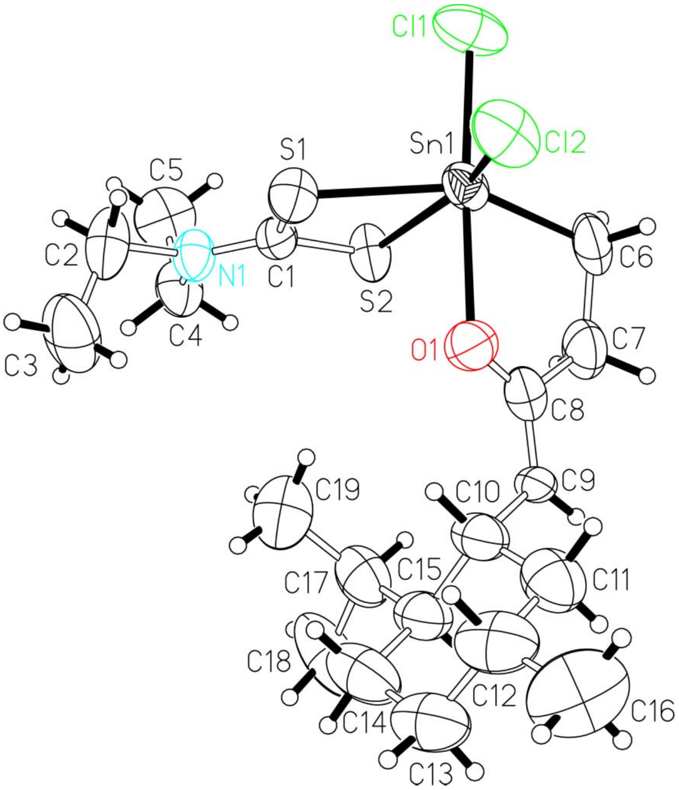


Figure 1
Plot of (I). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

References

Bruker (2002). *SADABS, SAINT* and *SMART*. Bruker AXS Inc, Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Tian, L.-J., Shang, Z.-C., Yu, Q.-S. & Zhang, L.-P. (2005). *Appl. Organomet. Chem.* **19**, 179–180.