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Redetermination of bis(*N*,*N*-diethyldithiocarbamato-κ*S*)dimethyltin(IV) at low temperature

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.026
wR factor = 0.052
Data-to-parameter ratio = 22.3

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

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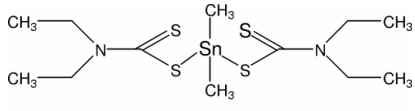
The title compound, $[Sn(CH_3)_2(C_5H_{10}NS_2)_2]$, previously reported by Morris & Schlemper [*J. Cryst. Mol. Struct.* (1979), **9**, 13–31], has been rerefined against new intensity data. Geometric parameters agree quite well. However, the positions of the hydroxyl H atoms could be determined employing the new data. Furthermore, the results of the present structure determination are of significantly higher precision. There are one and a half molecules in the asymmetric unit. The Sn atom of one molecule is located on a twofold rotation axis, whereas all other atoms are located in general positions.

Comment

Perspective views of the title compound, (I), are shown in Figs. 1 and 2. The original structure was reported by Morris & Schlemper (1979), who also reported a triclinic polymorph. Lockhart *et al.* (1986), on the other hand, determined the structure of an orthorhombic polymorph. The geometric parameters of both determinations agree quite well. A least-squares fit between all non-H atoms gives an r.m.s. deviation of 0.044 Å. However, the present work is of significantly improved precision and we were able to determine the positions of the H atoms. There are one and a half molecules in the asymmetric unit. The Sn atom of one molecule is located in a twofold rotation axis, whereas all other atoms are located on general positions.



(I)

Experimental

In an effort to synthesize a macrocyclic tin complex, we added diethyltin dichloride, 2,4-pentanedione, ethylenediamine and sodium diethyl dithiocarbamate trihydrate to DMSO as solvent medium. From the product mixture we isolated suitable single crystals. However, the resulting structure was totally unexpected.

Crystal data

$[Sn(CH_3)_2(C_5H_{10}NS_2)_2]$
M_r = 445.28
Monoclinic, *C*2/*c*
a = 27.735 (2) Å
b = 12.3703 (7) Å
c = 17.8281 (12) Å
β = 100.829 (6)°
V = 6007.7 (7) Å^3
Z = 12
D_x = 1.477 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 22309 reflections
θ = 3.5–25.2°
μ = 1.68 mm^{-1}
T = 293 (2) K
Block, colourless
0.22 × 0.21 × 0.12 mm

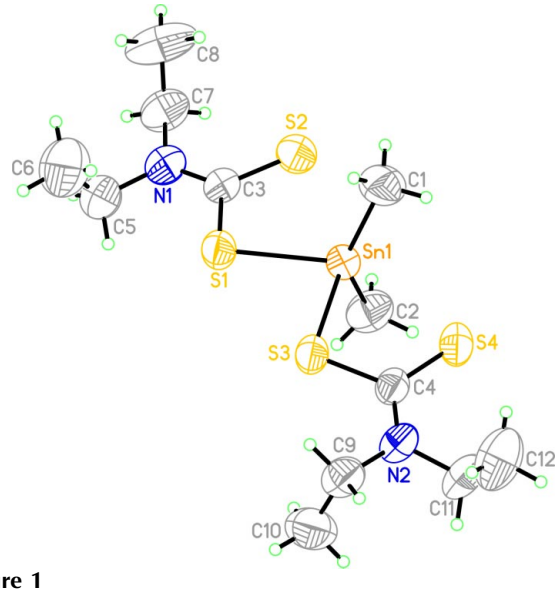


Figure 1
Perspective view of molecule 1 of the title compound, showing the atom numbering and displacement ellipsoids at the 50% probability level.

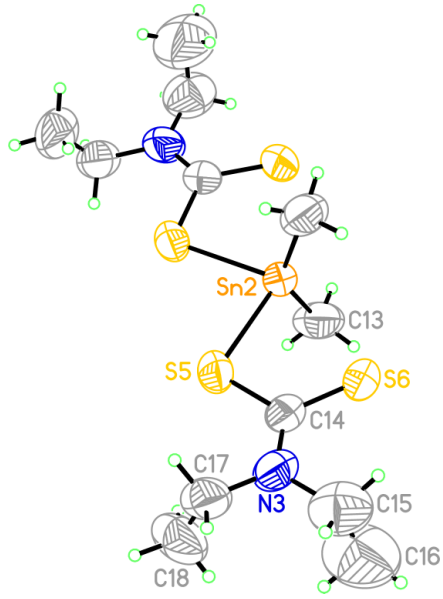


Figure 2
Perspective view of molecule 2 of the title compound, showing the atom numbering and displacement ellipsoids at the 50% probability level.

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.708, T_{max} = 0.824
41948 measured reflections
5755 independent reflections
3910 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.8°
$h = -33 \rightarrow 33$
$k = -15 \rightarrow 15$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.052
S = 0.97
5755 reflections
258 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected geometric parameters (Å).

Sn1—C1	2.113 (3)	S3—C4	1.750 (3)
Sn1—C2	2.116 (3)	S4—C4	1.688 (3)
Sn1—S1	2.5207 (8)	Sn2—C13	2.114 (4)
Sn1—S3	2.5308 (8)	S5—C14	1.748 (3)
S1—C3	1.746 (3)	S6—C14	1.688 (4)
S2—C3	1.680 (3)		

All H atoms could be located unequivocally in a difference Fourier synthesis and were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl})], using a riding model, with C—H = 0.99 Å or methyl C—H = 0.98 Å. One curious feature of the structure is the very short C15—C16 bond, which at 1.356 (7) Å is much shorter than a normal CH_2—CH_3 bond. We attribute this to a slight disorder of these atoms. It is interesting to note that the structure of Morris & Schlemper (1979) shows the same feature.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

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