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cis-Dibromo-*trans*-dimethyl-*cis*-bis(*N*,*N*-dimethylacetamide)tin(IV)

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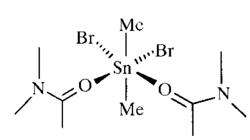
The synthesis and X-ray structure analysis of the title compound, $[SnBr_2(CH_3)_2(C_4H_9NO)_2]$, are described. The crystal contains molecules which are separated by normal van der Waals distances. Organotin(IV) compounds are found in a variety of structural types, in which the Sn atom can, for example, be hexacoordinated. In this case, the preferred solid-state molecular structure of the central atom is octahedral. The degree of distortion and the configuration depend on the ligands.

Comment

A comparative study of the title compound, $[SnBr_2(CH_3)_2(C_4H_9NO)_2]$, (I), and *cis*-dibromo-*trans*-dimethyl-*cis*-bis(*N*-methylpyrrolidinone)tin(IV) (König *et al.*, 2000), $[SnBr_2(CH_3)_2(C_5H_9NO)_2]$, may help to point out possible differences in the coordination of dibromodimethylstannane by cyclic and acyclic amides, particularly because the compounds chosen are of a similar constitution. This topic has not been dealt with in the literature and the available data seem to contain little information (Aslanov *et al.*, 1978). The Sn—C bond lengths of the title DMA complex (*N*,*N*-dimethylacetamide) are 2.107 (4) and 2.117 (5) Å. The differences from those in the analogous NMP (*N*-methylpyrrolidinone) compound [2.111 (5) and 2.115 (4) Å] are obviously small (König *et al.*, 2000), but both are in the range of reported values (Skinner & Sutton, 1944; Fujii & Kimura, 1971; Aslanov *et al.*, 1978). In the DMA adduct, the Sn—O bond lengths are longer (2.348 and 2.398 Å) than in the corresponding NMP compound [2.308 (3) and 2.352 (3) Å]. Although the differences between equivalent bonds in each compound are small, the Sn—O bonds in both cases are shorter than in related complexes (Yoshida *et al.*, 1968; Kimura *et al.*, 1969). However, the Sn—Br bond lengths in the DMA complex have values of 2.6385 (7) and 2.6589 (8) Å. As expected from other observations (Aslanov *et al.*, 1978), they are indeed shorter than those in the NMP complex with values of 2.6717 (14) and 2.6791 (10) Å, but the difference of 0.0204 Å between the two bonds in the DMA derivative is large compared with the NMP complex (0.0074 Å). Consequently, the Sn—Br bonds in both complexes are longer than in similarly configured compounds with coordination *via* oxygen (Yoshida *et al.*, 1968; Kimura *et al.*, 1969) and the uncomplexed Me_2SnBr_2 (Skinner & Sutton, 1944), but they are shorter than in compounds coordinated *via* nitrogen (Rivarola *et al.*, 1987) and in all-*trans* configured compounds (Aslanov *et al.*, 1978). The values of the C—O bond lengths in the DMA compound [1.236 (8) and 1.256 (8) Å] are comparable with those in the analogous NMP complex [1.246 (5) and 1.253 (4) Å]. The deviation from ideal geometry is demonstrated most clearly by the angles around the central atom. The value of the C1—Sn1—C2 angle in the DMA complex is 164.2 (2)° compared with 169.10 (18)° in the NMP complex. In the equatorial plane, the deviation from the ideal angle of 180° by the *trans* ligands in the DMA adduct becomes less obvious. The values found for the angles O1—Sn1—Br1 and O2—Sn1—Br2 are 176.46 (9) and 172.61 (8)°, respectively. The two methyl groups are distorted somewhat towards the two DMA ligands. This becomes obvious from the values of the angles between the methyl groups and the axial ligands: (*a*) O1—Sn1—C1 83.70 (16)°, O1—Sn1—C2 87.75 (16)°, O2—Sn1—C1 82.48 (16)°, O2—Sn1—C2 83.44 (16)°; (*b*) Br1—Sn1—C1 93.51 (14)°, Br1—Sn1—C2 94.45 (14)°, Br2—Sn1—C1 96.91 (14)°, Br2—Sn1—C2 96.12 (14)°. The orientation of the two DMA ligands is defined by some bond and torsion angles. The values of the angles C11—O1—Sn1 and C21—O2—Sn1 are 135.5 (3) and 137.9 (4)°, respectively. The difference in the NMP complex is more obviously demonstrated by the two appropriate angles 138.7 (2) and 133.8 (13)°. The torsion angles Sn1—O1—C11—C12, Sn1—O1—C11—N2, Sn1—O2—C21—C22 and Sn1—O2—C21—N2 which represent the orientation of the DMA ligands have the following values: 72.0 (7), −109.9 (9), −69.1 (7) and 115.1 (8)°. They differ considerably from the corresponding values in the NMP complex: 10.5 (7), −172.0 (3), 29.8 (6) and −151.8 (3)°.



(I)

Experimental

The title compound was prepared by the reaction of *N*,*N*-dimethylacetamide (1.31 g, 1.39 ml, 15.0 mmol) with freshly sublimed dibromodimethylstannane (2.27 g, 7.5 mmol) derived from the reaction of dimethyltin oxide with HBr (Pfeiffer, 1902) in 15 ml dry diethyl ether. The reaction mixture was stirred for 30 min and afterwards stored in a refrigerator at 278 K. Colourless crystals were obtained in quantitative yield after filtration and drying *in vacuo*. A solution of the complex (50 mg) in $CDCl_3$ (820 mg) gives the following values for the structure-relevant NMR parameters: $^2J(^{119}Sn–^1H)$ = 79 Hz, $^1J(^{119}Sn–^{13}C)$ = 589 Hz and $\delta(^{119}Sn)$ = −68.6 p.p.m. These values represent an equilibrium, which is as expected shifted when a solution of the complex (70 mg) dissolved in DMA (460 mg) is studied: ^2J(Sn–C–H) = 105 Hz, ^1J(Sn–C) = 855 Hz and $\delta(^{119}Sn) = -206.9$ p.p.m. It is obvious that the relevant NMR parameters of both samples correlate well with the values for *cis*-dibromo-*trans*-dimethyl-*cis*-bis(*N*-methylpyrrolidinone)tin(IV) (König *et al.*, 2000).

Crystal data

$[SnBr_2(CH_3)_2(C_4H_9NO)_2]$	$D_x = 1.901$ Mg m^{-3}
$M_r = 482.83$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4990 reflections
$a = 13.798$ (3) Å	$\theta = 2.85$–25.01°
$b = 9.3350$ (19) Å	$\mu = 6.245$ mm^{-1}
$c = 13.729$ (3) Å	$T = 173$ (2) K
$\beta = 107.42$ (3)°	Parallelepiped, colourless
$V = 1687.3$ (6) Å^3	0.14 × 0.13 × 0.13 mm
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	2307 reflections with $I > 2\sigma(I)$
Method: 294 frames *via* ω rotation ($\Delta\omega = 1°$) at different κ values and two times 80 s per frame	$R_{int} = 0.022$
	$\theta_{max} = 25.01°$
	$h = -15 \rightarrow 15$
4990 measured reflections	$k = -11 \rightarrow 9$
2849 independent reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 1.0314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	
$wR(F^2) = 0.078$	
$S = 1.093$	$(\Delta/\sigma)_{max} < 0.001$
2849 reflections	$\Delta\rho_{max} = 1.33$ e Å^{-3}
162 parameters	$\Delta\rho_{min} = -1.07$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.107 (4)	Sn1—O2	2.398 (3)
Sn1—C2	2.117 (5)	Sn1—Br2	2.6385 (7)
Sn1—O1	2.348 (3)	Sn1—Br1	2.6589 (8)
C1—Sn1—C2	164.2 (2)	O1—Sn1—Br2	88.15 (9)
C1—Sn1—O1	83.70 (16)	O2—Sn1—Br2	172.61 (8)
C2—Sn1—O1	87.75 (16)	C1—Sn1—Br1	93.51 (14)
C1—Sn1—O2	82.48 (16)	C2—Sn1—Br1	94.45 (14)
C2—Sn1—O2	83.44 (16)	O1—Sn1—Br1	176.46 (9)
O1—Sn1—O2	84.46 (11)	O2—Sn1—Br1	93.02 (8)
C1—Sn1—Br2	96.91 (14)	Br2—Sn1—Br1	94.37 (2)
C2—Sn1—Br2	96.12 (14)		

H atoms were placed in calculated positions with U_{iso} constrained to be $1.2U_{eq}$ of the carrier atom. The largest features in the final difference synthesis are close to N1; this, together with relatively high displacement parameters, indicates possible unresolved disorder.

Data collection: *KappaCCD Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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