

Dibromobis[2-(*N,N*-dimethylaminomethyl)-phenyl]tin(IV)Zdeňka Padělková,^a Ivana Čísařová^b and Aleš Růžička^{a*}^aDepartment of General and Inorganic Chemistry, Faculty of Chemical Technology, University of Pardubice, Nám. Čs. legií 565, Pardubice 532 10, Czech Republic, and^bDepartment of Inorganic Chemistry, Charles University, Hlavova 2030, Prague 128 40, Czech Republic

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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$

R factor = 0.048

wR factor = 0.124

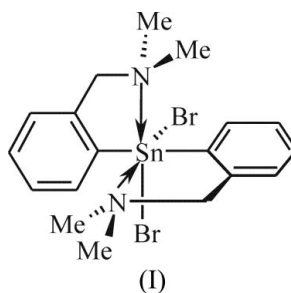
Data-to-parameter ratio = 21.3

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

In the crystal structure of the title compound, $[\text{SnBr}_2(\text{C}_9\text{H}_{12}\text{N})_2]$, the Sn atom is pseudo-octahedrally coordinated as a result of the *C,N*-chelation by the 2-(*N,N*-dimethylaminomethyl)phenyl groups and two *cis*-bonded bromides. The C atoms bonded to Sn are in *trans* positions [$\text{C}-\text{Sn}-\text{C} = 156.7(2)^\circ$] and the coordinated N atoms are bonded in a *cis* fashion [$\text{N}-\text{Sn} = 2.581(5)$ and $2.600(5) \text{ \AA}$; $\text{N}-\text{Sn}-\text{N} = 103.88(16)^\circ$].

Comment

The title compound, (I) (van Koten *et al.*, 1979), is one of the first of its type, *i.e.* containing a *C,N*-chelating ligand (and thus the related pincer ligands) and revealing a dynamic behavior in solution; this fact probably started the interest of many laboratories in such compounds (van Koten & Albrecht, 2001). Our interest is mainly focused on the family of organotin(IV) compounds containing 2-(dimethylaminomethyl)phenyl groups (L^{CN}) or related ligands which reveal interesting structural properties (Jastrzebski & van Koten, 1993; Davies, 1997) and potential utility (Růžička *et al.*, 2002). We have reported on such a class of diorganotin(IV) dihalides previously (Novák, Padělková *et al.*, 2005; Novák, Brus *et al.*, 2005). Although the title compound has been prepared and characterized, and the structure predicted from the NMR data more than thirty years ago, no single crystal suitable for X-ray measurements was obtained, even at the time of the study in which we described the structure of $(L^{\text{CN}})_2\text{SnX}_2$, with $X = \text{F}$, Cl , and I (Novák, Padělková *et al.*, 2005; Novák, Brus *et al.*, 2005). Therefore the structure of (I) was the last missing member of this series.



Relatively good crystals were finally obtained as described below. The Sn atom in (I) is pseudo-octahedrally coordinated as a result of the *C,N*-chelate bonding of two L^{CN} groups and two *cis* bonded halides. Atoms C11 and C21 are in *trans* positions and the coordinated nitrogen atoms are bonded in a *cis* fashion (Fig. 1 and Table 1). The molecular structure of (I) reveals some very close similarities to the previously described iodidobis[8-(dimethylamino)-1-naphthyl]methyltin(IV) (Jastrzebski *et al.*, 1992), dibromobis[8-(dimethylamino)-1-naph-

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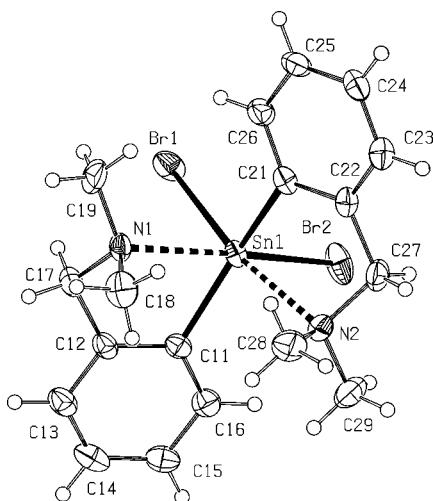


Figure 1
The molecular structure of (I) (ORTEP, 40% probability level). H atoms are drawn as spheres of arbitrary radius.

thyl]tin(IV) (Jastrzebski *et al.*, 1991), quinoline-substituted dialkyltin dihalides (Leung *et al.*, 2003) and $(L^{CN})_2SnX_2$, where $X = F, Cl$, and (I) (Novák, Padělková *et al.*, 2005; Novák, Brus *et al.*, 2005). The main deviations from the ideal octahedral geometry are the C—Sn—C (ideal 180°) and N—Sn—N angles (ideal 90°). The Sn—N distances are somewhat longer than found for triorganotin C,N -chelated compounds (around 2.5 \AA) (Cambridge Structural Database; Conquest Version 1.7, update of 20 August 2005; Allen, 2002) but in good agreement with those for $(L^{CN})_2SnX_2$. All of the reported values for Sn—halide distances as well as the appropriate bond angles match perfectly well with the theoretical curves deduced from crystallographic evidence for $SnC_2X_2Y_2$ ensembles having a C,C -transoid arrangement (Britton & Dunitz, 1981). In addition, the values of the torsion angles indicating the deviations of the nitrogen donor atoms from the planes of their parent aromatic rings as well as the angles between the planes of these aromatic rings are in line with previous findings [34.1 (C11—C12—C17—N1), 34.4 (C21—C22—C27—N2) and 40.4° ; Novák, Padělková *et al.*, 2005; Novák, Brus *et al.*, 2005].

Experimental

Compound (I) was prepared by methods published elsewhere (van Koten *et al.*, 1979; Novák, Padělková *et al.*, 2005). Single crystals suitable for X-ray analysis were obtained by slow vapour diffusion of hexane into a dichloromethane (5%) solution of (I) under vacuum at 213 K .

Crystal data

$[SnBr_2(C_9H_7N)_2]$	$D_x = 1.851 \text{ Mg m}^{-3}$
$M_r = 546.90$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 23900 reflections
$a = 9.8370$ (2) \AA	$\theta = 1-27.5^\circ$
$b = 13.3470$ (3) \AA	$\mu = 5.38 \text{ mm}^{-1}$
$c = 15.0230$ (3) \AA	$T = 150$ (2) K
$\beta = 95.8090$ (13) $^\circ$	Prism, colourless
$V = 1962.31$ (7) \AA^3	$0.25 \times 0.2 \times 0.12 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD area-detector diffractometer	4507 independent reflections
φ and ω scans	3812 reflections with $I > 2\sigma(I)$
Absorption correction: integration (Gaussian; Coppens, 1970)	$R_{int} = 0.069$
$T_{min} = 0.288, T_{max} = 0.659$	$\theta_{max} = 27.5^\circ$
29364 measured reflections	$h = -12 \rightarrow 12$
	$k = -17 \rightarrow 17$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 11.5726P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.10$	$\Delta\rho_{max} = 3.02 \text{ e \AA}^{-3}$
4507 reflections	$\Delta\rho_{min} = -1.41 \text{ e \AA}^{-3}$
212 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

Sn1—C21	2.114 (5)	Sn1—N2	2.600 (5)
Sn1—C11	2.128 (5)	Sn1—Br1	2.6164 (7)
Sn1—N1	2.581 (5)	Sn1—Br2	2.6201 (7)
C21—Sn1—C11	156.7 (2)	N1—Sn1—Br1	83.30 (12)
C21—Sn1—N1	91.02 (19)	N2—Sn1—Br1	170.43 (11)
C11—Sn1—N1	74.87 (19)	C21—Sn1—Br2	97.02 (15)
C21—Sn1—N2	74.25 (19)	C11—Sn1—Br2	99.02 (16)
C11—Sn1—N2	91.02 (19)	N1—Sn1—Br2	170.52 (12)
N1—Sn1—N2	103.88 (16)	N2—Sn1—Br2	83.23 (12)
C21—Sn1—Br1	99.60 (16)	Br1—Sn1—Br2	90.37 (3)
C11—Sn1—Br1	97.03 (16)		

All H atoms were positioned geometrically and refined as riding on their parent C atoms, with C—H = 0.93 \AA and $U_{iso}(H) = 1.2U_{eq}(C)$, and C—H = 0.96 \AA and $U_{iso}(H) = 1.5U_{eq}(C)$ for phenyl and methyl H atoms, respectively. The highest density peak and deepest hole are located 0.9 \AA from atom Br2.

Data collection: COLLECT (Hooft, 1998) and DENZO (Otwinowski & Minor, 1997); cell refinement: COLLECT and DENZO; data reduction: COLLECT and DENZO; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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