

## {2,6-Bis[(dimethylamino)methyl]-phenyl- $N^2,C^1,N^6$ }diphenyltin(II) bromide monohydrate

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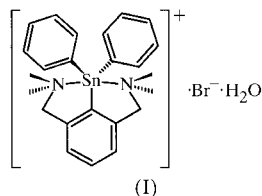
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In the title compound,  $[\text{Sn}(\text{C}_6\text{H}_5)_2(\text{C}_{12}\text{H}_{19}\text{N}_2)]\text{Br}\cdot\text{H}_2\text{O}$ , the  $\text{Sn}^{\text{IV}}$  atom lies on a twofold axis and is coordinated by a C and two N atoms from the 2,6-bis[(dimethylamino)methyl]phenyl ligand in a tridentate fashion and by two phenyl groups. The resulting geometry is intermediate between square pyramidal and trigonal bipyramidal, with three C atoms in equatorial and the two N atoms in axial positions. The main deformation from ideal trigonal-bipyramidal geometry is seen for the  $\text{N}-\text{Sn}-\text{N}$  angle [ $152.18(7)^\circ$ ]. The  $\text{Br}^-$  anion and the water solvate molecule are on an inversion centre and twofold axis, respectively. They form an infinite chain of  $\text{Br}\cdots\text{H}-\text{O}-\text{H}\cdots\text{Br}$  hydrogen bonds [ $\text{Br}\cdots\text{O}$  3.529 (2) Å] without contributing to the primary coordination sphere of the Sn atom.

### Comment

Organotin(IV) compounds have great potential applications in different fields of life science (Gielen, 1996), especially biocidal applications, utilizing the antitumor and anticancer activities of such compounds. The title compound, (I), is of interest because of its relatively good solubility in water and other polar solvents, which is the most important attribute in the study of biological properties of organotin compounds.



Although the 2,6-bis[(dimethylamino)methyl]phenyl ( $L1$ ) moiety is a widely used ligand, being part of more than 80 organometallic crystal structures documented in the Cambridge Structural Database (Allen & Kennard, 1993),

there are only two organotin(IV) complexes with this ligand [(II) (Amini *et al.*, 1993) and (III) (Jastrzebski *et al.*, 1991)].

The molecular structure and atomic numbering scheme of (I) are shown in Fig. 1. The coordination polyhedron of the Sn atom does not belong either to a square pyramid or a trigonal bipyramid, as  $\tau$  is 0.45 (Alvarez & Llunel, 2000). The latter was chosen for further description because some of its symmetry properties are preserved in the structure. The pseudo-trigonal bipyramid is formed by three C atoms in the equatorial plane and two *trans*-N atoms. Since both N atoms are part of one tridentate ligand ( $L1$ ), the geometry of the coordination sphere is distorted, with an  $\text{N}-\text{Sn}-\text{N}$  angle of  $152.18(7)^\circ$ , however, the whole moiety follows the twofold axial symmetry of space group  $C2/c$ , which passes through Sn1, C1 and C4. Because of the symmetry, the  $\text{C}_3\text{Sn}$  girdle is perfectly planar and the  $\text{Sn}-\text{N}$  distances are equivalent. The bond distances and angles, as well as the planarity of the aromatic rings in the organic part of the title compound, are unexceptional.

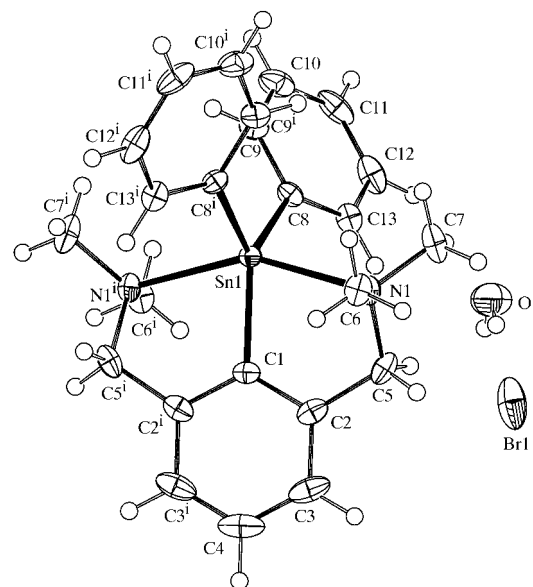


Figure 1

A view of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (i)  $-x, y, \frac{3}{2} - z$ ].

In two comparable structures, (II) and (III), the coordination polyhedra are trigonal bipyramid and octahedron, respectively, and the ranges of  $\text{Sn}-L1$  distances ( $\text{Sn}-\text{C}$  2.082–2.109 Å,  $\text{Sn}-\text{N}$  2.355–2.501 Å and  $\text{N}-\text{Sn}-\text{N}$  150.7–152.7°) suggest a similarity in the coordination of  $L1$  in both configurations. As expected, the parameters of the primary sphere of the Sn atom in the title compound and (II), namely [2,6-bis(dimethylamino)methyl]phenyldimethyltin, are almost identical, the differences in the parameters mentioned in Table 1 being within four times the s.u.'s of (II), with the exception of the  $\text{C8}-\text{Sn}-\text{C8}^i$  angle, which has values of  $110.09(9)$  and  $114.2(9)^\circ$  in (I) and (II), respectively. Unfortunately, a more detailed discussion is hindered by the rather large s.u.'s associated with parameters of structure (II).

The crystal structure of (I) is ionic, where, in addition to coulombic attraction between  $\{[(\text{CH}_3)_2\text{NCH}_2]_2(\text{C}_6\text{H}_5)\}\text{Sn}^+$  and  $\text{Br}^-$ , two  $\text{Br}^-$  anions are bridged by water molecules as a result of two  $\text{H}^i\text{—O—H}$  symmetrically related hydrogen bonds  $[\text{O}1\cdots\text{Br}1^- 3.529(2) \text{ \AA}$  and  $\text{O}1\text{—H}1\cdots\text{Br}^- 178(3)^\circ]$ , forming an infinite chain along the  $[101]$  direction [symmetry code: (i)  $-x, y, \frac{3}{2} - z$ ].

## Experimental

The synthesis was carried out under anaerobic conditions. The final compound was prepared from 2,6-bis[(dimethylamino)methyl]-benzene *via* ortholithiation with *n*-butyllithium in hexane/diethyl ether. The organolithium salt formed was reacted with diphenyltin dibromide in an equimolar ratio and the product recrystallized from boiling toluene. Suitable crystals were obtained by vapor diffusion of *n*-hexane into a wet dichloromethane solution of the title compound.

### Crystal data

$[\text{Sn}(\text{C}_6\text{H}_5)_2(\text{C}_{12}\text{H}_{19}\text{N}_2)]\text{Br}\cdot\text{H}_2\text{O}$	$D_x = 1.566 \text{ Mg m}^{-3}$
$M_r = 562.11$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 15656 reflections
$a = 13.8520(3) \text{ \AA}$	$\theta = 0.998\text{--}27.485^\circ$
$b = 20.0760(4) \text{ \AA}$	$\mu = 2.765 \text{ mm}^{-1}$
$c = 10.4740(2) \text{ \AA}$	$T = 150(2) \text{ K}$
$\beta = 125.0660(12)^\circ$	Prism, colourless
$V = 2384.05(8) \text{ \AA}^3$	$0.2 \times 0.2 \times 0.1 \text{ mm}$
$Z = 4$	

### Data collection

Nonius KappaCCD area-detector diffractometer	2688 independent reflections
$\varphi$ and $\omega$ scans to fill the Ewald sphere	2612 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (SORTAV; Blessing 1997)	$R_{\text{int}} = 0.046$
$T_{\text{min}} = 0.521, T_{\text{max}} = 0.659$	$\theta_{\text{max}} = 27.49^\circ$
23135 measured reflections	$h = -17 \rightarrow 17$
	$k = -25 \rightarrow 26$
	$l = -13 \rightarrow 13$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 2.3842P]$
$R[F^2 > 2\sigma(F^2)] = 0.020$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.050$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.114$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2688 reflections	$\Delta\rho_{\text{min}} = -0.80 \text{ e \AA}^{-3}$
154 parameters	
H atoms treated by a mixture of independent and constrained refinement	

All H atoms were found in difference Fourier maps. The unique H1 atom of the water molecule was refined isotropically; all other H atoms were allowed for as riding atoms with C—H distances in the range 0.93–0.97 Å. Four strong reflections, 020, 110,  $-111$  and 021, were omitted from the final refinement due to measurement inaccuracy.

**Table 1**

Selected geometric parameters (Å, °).

Sn1—C1	2.093(2)	Sn1—N1	2.4398(14)
Sn1—C8	2.1408(15)		
C1—Sn1—C8	124.95(4)	C8 <sup>i</sup> —Sn1—N1 <sup>i</sup>	99.48(5)
C1—Sn1—C8 <sup>i</sup>	124.95(4)	C1—Sn1—N1	76.09(3)
C8—Sn1—C8 <sup>i</sup>	110.09(9)	C8—Sn1—N1	99.48(5)
C1—Sn1—N1 <sup>i</sup>	76.09(3)	C8 <sup>i</sup> —Sn1—N1	96.36(5)
C8—Sn1—N1 <sup>i</sup>	96.36(5)	N1 <sup>i</sup> —Sn1—N1	152.18(7)

Symmetry code: (i)  $-x, y, \frac{3}{2} - z$ .

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\text{—H}\cdots A$	$D\text{—H}$	$H\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
O1—H1 $\cdots$ Br1	0.80(3)	2.73(3)	3.5293(16)	176(3)

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, 1999); software used to prepare material for publication: SHELXL97 and PLATON.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1427). Services for accessing these data are described at the back of the journal.

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