torsion angles for F and O atoms in *trans* positions are 175 (1), 178 (1) and 176 (1)°.

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Di-*tert*-butyl(glycylglycinato)tin(IV) Monohydrate

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Abstract. $[Sn(C_4H_6N_2O_3)(C_4H_9)_2].H_2O$, $M_r = 381 \cdot 0$, monoclinic, $P2_1/n$, $a = 8 \cdot 895$ (6), $b = 14 \cdot 092$ (7), $c = 13 \cdot 653$ (8) Å, $\beta = 93 \cdot 17$ (5)°, $V = 1709$ (2) Å^3, $Z = 4$, $D_x = 1 \cdot 481$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0 \cdot 71073$ Å, $\mu = 1 \cdot 5$ mm^{-1}, $F(000) = 776$, $T = 291$ (1) K, final $R = 0 \cdot 063$ for 1872 unique reflections $[F \geq 3 \cdot 0\sigma(F)]$. The polyhedron around Sn is a distorted trigonal bipyramid formed by two equatorial $C(CH_3)_3$ groups and the tridentate, nearly planar, glycylglycinate ligand. The equatorial Sn—N$_{peptide}$ bond is short: $2 \cdot 085$ (8) Å. The $(CH_3)_3C$—Sn—$C(CH_3)_3$ bond angle is $121 \cdot 7$ (4)°, the O—Sn—N$_{amino}$ bond angle is only $149 \cdot 6$ (3)°. The molecules are connected through a hydrogen-bond network involving hydrogen bonds between water and NH$_2$ and CO$_{peptide}$ and CO$_{carboxylate}$ groups as well as hydrogen bonds between NH$_2$ and CO$_{carboxylate}$ groups of neighbouring molecules.

Introduction. Previous structural studies of diorganotin derivatives of dipeptides R_2SnX (H$_2X$ = dipeptide),

some of which exhibit antileukaemic properties (Huber & Barbieri, 1986), demonstrated that Sn has a distorted trigonal-bipyramidal environment and that the bond angles in the polyhedron around Sn are more or less different depending on the type of R and X [$R = Ph = C_6H_5$, $X = GlyGly = $ glycylglycinate (Huber, Haupt, Preut, Barbieri & Lo Giudice, 1977); $R = Me = CH_3$, $X = GlyMet = $ glycylmethionate (Preut, Mundus, Huber & Barbieri, 1986); $R = Cy = C_6H_{11}$, $X = GlyAla = $ glycylalaninate, GlyGly (Vornefeld, Huber, Preut & von Angerer, 1989); $R = n$-Bu $= n$-C_4H_9, $X = GlyVal = $ glycylvalinate (Preut, Mundus, Huber & Barbieri, 1989)]. The molecules are linked by hydrogen bonds. In this context we found it worthwhile to study the function of the water molecule in the hydrogen-bonding system of the title compound with two voluminous R groups at Sn, which we synthesized recently as a first example of an R_2SnX hydrate. We also wanted to find out if the water molecule might even coordinate to Sn as we observed recently in an R_2Sn

compound with the alternative tridentate 2,6-pyridine-dicarboxylate ligand (Huber, Preut, Hoffmann & Gielen, 1989).

Experimental. Title compound prepared from (*tert*-$C_4H_9)_2SnCl_2$ and $Na_2GlyGly$ in methanol. Single crystals obtained from ethanol. Crystal size ~0·06 × 0·32 × 0·10 mm, $\omega/2\theta$ scan, scan speed 1·5–15·0° min^{-1} in θ, Nicolet $R3m$/V diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 22 reflections up to 2θ = 25·6°; six standard reflections recorded every 2·5 h, only random deviations; 6493 reflections measured, $1·5 \le \theta \le 25·0°$, $-11 \le h \le 11$, $-17 \le k \le 17$, $0 \le l \le 17$; after averaging ($R_{int} = 0·053$): 2494 unique reflections, 1872 with $F \ge 3·0\sigma(F)$; Lorentz–polarization correction, no absorption correction; systematic absences $(h0l)$ $h + l = 2n + 1$, $(0k0)$ $k = 2n + 1$ conform to space group $P2_1/n$; structure solution via Patterson function, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·95 Å);
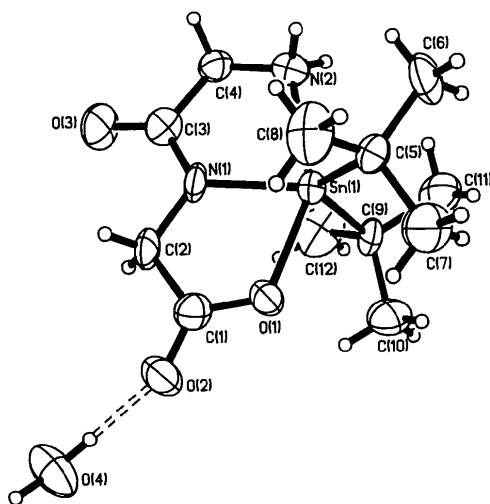

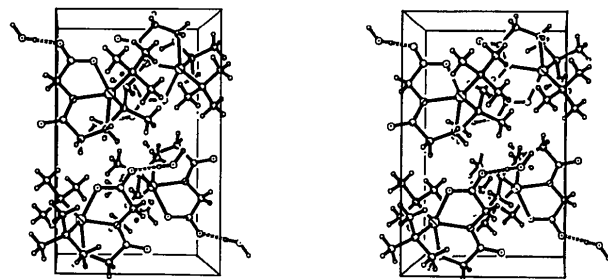Fig. 1. General view of the molecule, showing the atom-numbering scheme.

Fig. 2. Stereoscopic view of the unit cell (a horizontal, b vertical).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$)

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
Sn(1)	0·16173 (8)	0·18894 (4)	0·29845 (5)	335
N(1)	0·3934 (9)	0·1673 (5)	0·3179 (6)	373
N(2)	0·186 (1)	0·0491 (6)	0·3837 (6)	449
O(1)	0·2637 (8)	0·3185 (5)	0·2423 (6)	510
O(2)	0·4772 (9)	0·3872 (5)	0·2024 (6)	584
O(3)	0·5899 (9)	0·0680 (6)	0·3551 (7)	642
O(4)	0·7260 (6)	0·4165 (4)	0·0922 (4)	787
C(1)	0·4098 (6)	0·3196 (4)	0·2386 (4)	440
C(2)	0·4957 (6)	0·2374 (4)	0·2807 (4)	434
C(3)	0·455 (1)	0·0860 (7)	0·3523 (8)	424
C(4)	0·343 (1)	0·0121 (7)	0·3818 (8)	401
C(5)	0·028 (1)	0·2685 (7)	0·3995 (8)	414
C(6)	−0·083 (1)	0·2009 (9)	0·4457 (9)	649
C(7)	−0·058 (2)	0·3445 (9)	0·340 (1)	717
C(8)	0·131 (1)	0·3135 (9)	0·4787 (9)	685
C(9)	0·065 (1)	0·1367 (8)	0·1586 (8)	432
C(10)	0·042 (1)	0·2199 (9)	0·0875 (8)	670
C(11)	−0·087 (1)	0·0903 (9)	0·1747 (9)	622
C(12)	0·175 (2)	0·064 (1)	0·1194 (9)	788

refinement on F with 1872 reflections and 167 refined parameters; $w = 1·0/[\sigma^2(F) + (0·0005F^2)]$; $S = 1·45$, $R = 0·063$, $wR = 0·049$, $(\Delta/\sigma)_{max} = 0·003$, no extinction correction; largest peak in final ΔF map $\pm 1·0$ (3) e Å^{-3}, atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: *PARST* (Nardelli, 1982), *SHELXTL-Plus* (Sheldrick, 1987).

Discussion. The structure of the title compound together with the numbering scheme is shown in Fig. 1 and a stereoscopic view in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* bond lengths and angles in Table 2. The trigonal-bipyramidal coordination sphere around Sn corresponds to those found in other R_2SnX compounds [Me$_2$SnGlyMet (Preut *et al.*, 1986), Ph$_2$SnGlyGly (Huber *et al.*, 1977), Cy$_2$SnX (X = GlyGly, GlyAla; Vornefeld *et al.*, 1989), *n*-Bu$_2$SnGlyVal (Preut *et al.*, 1989)]; the tridentate dipeptide ligand is nearly planar, and O(1) of the unidentate carboxylate group and N(2) of the amino group are in apical positions while N(1), being part of the short Sn—N$_{peptide}$ bond, C(5) and C(9) are in equatorial positions. The dihedral angle between the planes through Sn(1)—O(1)—C(1)—C(2)—N(1) (plane 1) and Sn(1)—N(1)—C(3)—C(4)—N(2) (plane 2) is 2·7 (2)°. The polyhedron around Sn is conspicuously distorted regarding the angle O(1)—Sn(1)—N(2) [149·6 (3)°], and an appropriate effect on other angles,

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Table 2. *Bond distances* (Å) *and angles* (°)

Sn(1)–N(1)	2·085 (8)	O(3)–C(3)	1·23 (1)	
Sn(1)–N(2)	2·293 (8)	C(1)–C(2)	1·485 (8)	
Sn(1)–O(1)	2·196 (7)	C(3)–C(4)	1·51 (1)	
Sn(1)–C(5)	2·18 (1)	C(5)–C(6)	1·54 (2)	
Sn(1)–C(9)	2·18 (1)	C(5)–C(7)	1·53 (2)	
N(1)–C(2)	1·45 (1)	C(5)–C(8)	1·52 (2)	
N(1)–C(3)	1·34 (1)	C(9)–C(10)	1·53 (2)	
N(2)–C(4)	1·49 (1)	C(9)–C(11)	1·53 (2)	
O(1)–C(1)	1·303 (9)	C(9)–C(12)	1·53 (2)	
O(2)–C(1)	1·24 (1)			

C(5)–Sn(1)–C(9)	121·7 (4)	N(1)–C(2)–C(1)	110·3 (5)
O(1)–Sn(1)–C(9)	97·2 (4)	N(1)–C(3)–O(3)	124·4 (9)
O(1)–Sn(1)–C(5)	92·5 (3)	O(3)–C(3)–C(4)	121 (1)
N(2)–Sn(1)–C(9)	100·2 (4)	N(1)–C(3)–C(4)	115·0 (9)
N(2)–Sn(1)–C(5)	99·2 (4)	N(2)–C(4)–C(3)	113·2 (8)
N(2)–Sn(1)–O(1)	149·6 (3)	Sn(1)–C(5)–C(8)	109·7 (7)
N(1)–Sn(1)–C(9)	113·7 (4)	Sn(1)–C(5)–C(7)	107·4 (7)
N(1)–Sn(1)–C(5)	124·3 (4)	Sn(1)–C(5)–C(6)	109·1 (7)
N(1)–Sn(1)–O(1)	75·0 (3)	C(7)–C(5)–C(8)	110·7 (9)
N(1)–Sn(1)–N(2)	75·2 (3)	C(6)–C(5)–C(8)	110·2 (9)
Sn(1)–N(1)–C(3)	123·3 (7)	C(6)–C(5)–C(7)	109·7 (9)
Sn(1)–N(1)–C(2)	119·3 (5)	Sn(1)–C(9)–C(12)	107·7 (8)
C(2)–N(1)–C(3)	116·9 (8)	Sn(1)–C(9)–C(11)	109·2 (7)
Sn(1)–N(2)–C(4)	110·7 (6)	Sn(1)–C(9)–C(10)	109·3 (7)
Sn(1)–O(1)–C(1)	117·1 (5)	C(11)–C(9)–C(12)	111 (1)
O(1)–C(1)–O(2)	121·9 (7)	C(10)–C(9)–C(12)	110·9 (9)
O(2)–C(1)–C(2)	120·2 (6)	C(10)–C(9)–C(11)	109 (1)
O(1)–C(1)–C(2)	117·9 (6)		

N(2)···O(2)(0·5–x, y–0·5, 0·5–z)	2·92 (1)
N(2)···O(4)(x–0·5, 0·5–y, 0·5+z)	2·89 (1)
O(4)···O(2)(x, y, z)	2·77 (1)
O(4)···O(3)(1·5–x, –0·5+y, 0·5–z)	2·76 (1)

e.g. O(1)–Sn(1)–C(9) [97·2 (4)°], is evident. The distortion appears to be caused mainly by the rigidity of the GlyGly ligand, while the two voluminous *tert*-butyl ligands at Sn seem to be of minor influence. The dihedral angles between planes (1) and (2) and the plane through N(1)–C(5)–C(9), from which Sn(1) deviates by only 0·065 (1) Å, are 92·2 (2) and 90·2 (2)°, respectively, and correspond, like the angle C(5)–Sn–C(9) of 121·7 (4)°, to nearly ideal values. This last angle is larger than the appropriate angle in Ph₂SnGlyGly [117·5 (3)° (Huber *et al.*, 1977)], but smaller than in Cy₂SnGlyGly [123·6 (2)° (Vornefeld *et al.*, 1989)].

The molecules are linked by a three-dimensional network of hydrogen bonds, involving both H atoms of the water molecule and the NH₂ group, O(2) of the carboxylate group and O(3) of the peptide link. Considering that in the title compound the C(1)–O(1) and C(1)–O(2) bonds are longer and that the C(1)–C(2) bond is shorter than the appropriate bonds in the R_2SnGlyGly compounds which contain no water [R = Ph/Cy/*tert*-Bu: C(1)–O(1) 1·293 (14)/1·287 (6)/1·303 (9); C(1)–O(2) 1·200 (13)/1·216 (6)/1·24 (1); C(1)–C(2) 1·549 (18)/1·522 (7)/1·485 (8) Å], it is inferred that these changes in bond lengths are a consequence of the formation of the extended hydrogen-bond system.

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Structures of Tetrakis(μ-acetato)bis(2-amino-4,6-dimethylpyrimidine)dirhodium(II) and its Monohydrate

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Abstract. [Rh₂(C₂H₃O₂)₄(C₆H₉N₃)₂], $M_r = 688·30$, triclinic, $P\bar{1}$, $a = 11·342$ (2), $b = 7·996$ (2), $c = 8·032$ (1) Å, $\alpha = 89·74$ (2), $\beta = 106·55$ (1), $\gamma = 102·96$ (2)°, $V = 679·1$ (2) Å³, $Z = 1$, $D_x = 1·683$ g cm⁻³, λ(Mo *K*α) = 0·71073 Å, $\mu = 12·46$ cm⁻¹, $F(000) = 346$, $T = 296$ K, $R = 0·023$ for

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