Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Aqua[4-(1*H*-benzimidazol-2-yl)benzoato]-triphenyltin(IV) dioxane solvate

Xiao-Niu Fang,[a]* Yi-An Xiao,[b] Yan Sui,[a] Hong-Mei Chen[a] and Chuan-Peng Zuo[a]

[a]JiangXi Province Key Laboratory of Coordination Chemistry, College of Chemistry and Chemical Engineering, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China, and [b]College of Life Science, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China

Correspondence e-mail: fangxiaoniu@163.com

Received 24 August 2006
Accepted 1 September 2006

The title complex, $[Sn(C_6H_5)_3(C_{14}H_9N_2O_2)(H_2O)]\cdot C_4H_8O_2$, is composed of a triphenyltin carboxylate with a coordinated water molecule and a free dioxane molecule. The Sn^{IV} atom is five-coordinate and has a *trans*-C_3SnO_2 trigonal–bipyramidal geometry, with the three phenyl groups occupying the equatorial plane, the 4-(1*H*-benzimidazol-2-yl)benzoate ligand coordinating in a monodentate manner and a water molecule in the axial positions. Strong intermolecular O—H···O and N—H···O hydrogen bonds link the molecules into an infinite two-dimensional network, with these networks held together by van der Waals forces to generate a layer structure. π–π Stacking interactions also exist in the crystal structure.

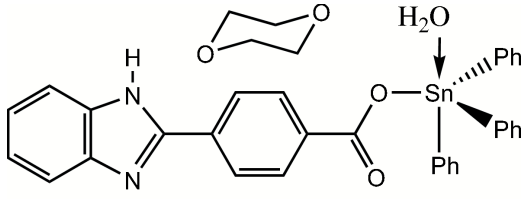
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.011 Å
Disorder in solvent or counterion
R factor = 0.055
wR factor = 0.177
Data-to-parameter ratio = 23.0

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

Comment

Benzimidazole and its derivatives are potential complexing agents, and have been found to have a broad scope for spin crossover and biological activities (Lin *et al.*, 2004), as well as antitumour activity (Schulz & Skibo, 2000) and anti-amoebic activity (Bharti *et al.*, 2000). The structural chemistry of organotin carboxylate complexes has attracted considerable attention, owing to their good antitumour activities (Barbieri *et al.*, 2001; Zhou *et al.*, 2005), their versatile molecular structures, and the supramolecular architectures exhibited by these complexes (Ma *et al.*, 2005). In the context of our continued interest in the structural and biological properties of organotin complexes (Fang *et al.*, 2001, 2006), we report here the synthesis and crystal structure of the title complex, (I).



(I)

Compound (I) crystallizes in the space group $P2_1/c$, with a triphenyltin carboxylate, a coordinated water molecule and a free dioxane molecule in the asymmetric unit, as shown in Fig. 1. The Sn^{IV} atom is five-coordinate and has a *trans*-C_3SnO_2 trigonal–bipyramidal geometry. The three phenyl groups occupy the equatorial plane, with the Sn^{IV} atom lying only 0.169 (2) Å out of the trigonal plane (in the direction of atom O2) defined by the three *ipso*-C atoms of the phenyl rings. The three phenyl rings are not coplanar, and the interplanar angles between them are 28.4 (1) (between the C15- and C27-rings),

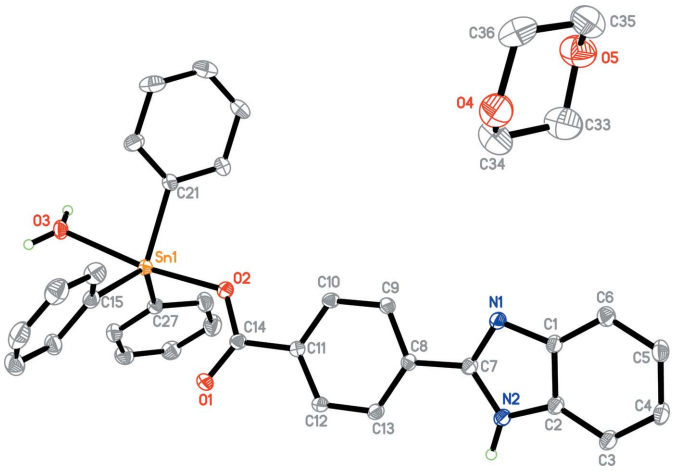


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme and 10% probability displacement ellipsoids. All carbon-bound H atoms have been omitted for clarity.

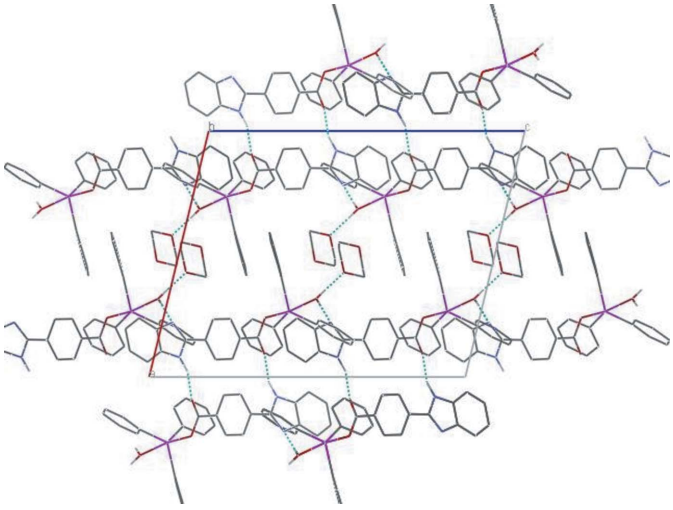


Figure 2
A packing diagram of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. All carbon-bound H atoms have been omitted for clarity.

44.34 (9) (between the C21- and C27-rings) and 49.23 (9)° (between the C15- and C21-rings), respectively. The 4-(1*H*-benzo[*d*]imidazol-2-yl)benzoate ligand coordinates in monodentate mode and occupies an axial position, with the other axial ligand being a coordinated water molecule. The axial ligands are not coordinated exactly linearly [O3—Sn1—O2 = 172.2 (2)°] and, although the Sn1—O2 bond length is normal, the Sn1—O3 bond is much shorter than the value of 2.471 (6) Å found by Kemmer *et al.* (1999). This is due to the coplanarity of the atoms Sn1, C15, C21 and C27, as mentioned above. The benzimidazole ring system makes a dihedral angle of 15.66 (8)° with the benzene ring of the benzoate group.

There are three classical intermolecular hydrogen bonds in the packing of (I), as shown in Table 2 and Fig. 2. These strong hydrogen bonds link the molecules into an infinite two-dimensional network, as shown in Fig. 2, while these networks are held together by van der Waals forces to generate a layer structure. A π–π stacking interaction exists between the benzene ring of the benzimidazole and one of the phenyl rings attached to the Sn atom [the C6$\cdots$C17^{iv} distance in the offset face-to-face interaction is 3.477 Å; symmetry code: (iv) x, $-\frac{1}{2}-y$, $\frac{1}{2}+z$].

Experimental

The title complex was prepared by the following procedure. A solution of 1,2-benzenediamine (0.108 g, 1 mmol) and 4-formylbenzoic acid (0.150 g, 1 mmol) in ethanol (20 ml) was heated for 3 h under reflux. Triphenylstannanol (0.625 g, 1 mmol) dissolved in acetonitrile (10 ml) was then added, and the mixture was heated for another 3 h under reflux. The reaction mixture was then cooled and the pale-yellow precipitate which had formed was filtered off. The reaction yield is 75% and no melting point could be measured since it was higher than 573 K. Recrystallization of the crude product from the solvent mixture benzene–toluene–dioxane (1:1:0.25) resulted in single crystals of (I) suitable for X-ray diffraction analysis after several days.

Crystal data

$[Sn(C_6H_5)_3(C_{14}H_9N_2O_2)(H_2O)]\cdot C_4H_8O_2$
M_r = 693.34
Monoclinic, $P2_1/c$
a = 15.233 (3) Å
b = 11.580 (2) Å
c = 19.047 (4) Å
β = 103.617 (2)°
V = 3265 (1) Å^3
Z = 4
D_x = 1.410 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 296 (2) K
Block, pale yellow
0.34 × 0.32 × 0.22 mm

Data collection

Bruker APEXII area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.75, T_{max} = 0.83
27888 measured reflections
7464 independent reflections
4923 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.177
S = 1.01
7464 reflections
325 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 3.11P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C15	2.113 (2)	Sn1—C21	2.123 (2)
Sn1—C27	2.114 (2)	Sn1—O3	2.434 (4)
Sn1—O2	2.121 (3)		
C15—Sn1—O2	98.7 (2)	O2—Sn1—O3	172.1 (2)
C27—Sn1—O2	97.0 (2)	O1—C14—C11	121.4 (4)
O2—Sn1—C21	87.4 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···O4^{i}	0.85 (5)	2.00 (5)	2.814 (7)	162 (6)
N2—H2···O1^{ii}	0.97 (4)	1.83 (3)	2.738 (5)	155 (6)
O3—H3A···N1^{iii}	0.85 (5)	2.00 (6)	2.842 (6)	176 (8)

Symmetry codes: (i) $x, y, z-1$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Disorder in the dioxane was identified from a difference map. Disordered atoms were refined by applying restraints to the bond lengths [1.54 (2) Å for C—C and 1.44 (2) Å for C—O] and the displacement parameters (similarity, rigid bond and approximate isotropic restraints) in the skeleton of the dioxane. H atoms on O and N atoms were located and refined with distance restraints of O—H = 0.85 (2) and N—H = 0.97 (2) Å. All other H atoms were positioned in idealized locations and refined as riding on their carrier atoms, with C—H distances of 0.93 (aryl) or 0.97 Å (methylene), and with U_{iso}(H) = 1.2U_{eq}(C). The highest peak is loacted 0.98 Å from atom C28.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *APEX2*; program(s) used to refine structure: *APEX2*; molecular graphics: *APEX2* and *MERCURY* (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: *APEX2*.

The authors gratefully acknowledge financial support from the Natural Science Foundation of JiangXi Province (grant No. 0620029) and from the Science and Technology Bureau of Ji'an City (grant No. 200528).

References

Barbieri, F., Sparatore, F., Cagnoli, M., Bruzzo, C., Novelli, F. & Alama, A. (2001). *Chem. Biol. Interact.* **134**, 27–39.
Bharti, N., Maurya, M. R., Naqvi, F. & Azam, A. (2000). *Bioorg. Med. Chem. Lett.* **10**, 2243–2245.
Bruker (2004). *APEX2* (Version 1.22) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fang, X.-N., Song, X.-Q. & Xie, Q. L. (2001). *J. Organomet. Chem.* **619**, 43–48.
Fang, X.-N., Sui, Y., Ying, S.-M., Xu, Y.-P. & Guo, X.-F. (2006). *Acta Cryst.* E**62**, m2008–m2010.
Kemmer, M., Ghys, L., Gielen, M., Biesemans, M., Tiekink, E. R. T. & Willem, R. (1999). *J. Organomet. Chem.* **582**, 195–203.
Lin, X.-J., Li, Y.-Z., Xu, H.-J., Liu, S.-G., Xu, L., Shen, Z. & You, X.-Z. (2004). *Acta Cryst.* E**60**, o77–o78.
Ma, C.-L., Zhang, Q.-F., Zhang, R.-F. & Qiu, L.-L. (2005). *J. Organomet. Chem.* **690**, 3033–3043.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Schulz, W. G. & Skibo, E. B. (2000). *J. Med. Chem.* **43**, 629–638.
Zhou, Y.-Z., Jiang, T., Ren, S.-M., Yu, J.-S. & Xia, Z.-C. (2005). *J. Organomet. Chem.* **690**, 2186–2190.