metal-organic papers

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# Bis(2-amino-5-nitrobenzoato-$\kappa^2 O,O'$)di-n-butyltin(IV)

The title compound, $[Sn(C_4H_9)_2(C_7H_5N_2O_4)_2]$, has twofold rotation symmetry, and two 2-amino-5-nitrobenzoate ligands are chelated to the dibutyltin unit through both O atoms of the carboxylate group in a six-coordinate environment. There are intra- and intermolecular N—H···O hydrogen bonds, forming one-dimensional chains parallel to the b axis.

Comment

The reaction of dibutyltin oxide with substituted aromatic carboxylic acids such as 3-hydroxyphthalic acid and (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid leads to the formation of distannoxane-type complexes $[Sn_4(C_4H_9)_8(C_8H_5O_3)_4(\mu\text{-}O)_2]$ (Khoo & Hazell, 1999) and $[Sn_4(C_4H_9)_8(C_{17}H_{15}O_2)_4O_2]$ (Parvez *et al.*, 2004), respectively. However, the reaction of dibutyltin oxide with 2-amino-5-nitrobenzoic acid gave a normal monomeric condensation reaction product, (I).

(I)
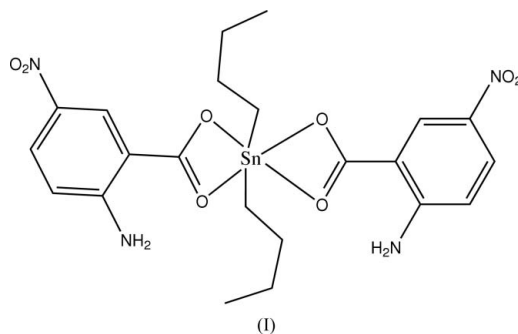
In (I), the molecule has a twofold rotation symmetry (Fig. 1) and is isostructural with di-*n*-butylbis(thiophene-3-carboxylato-*O,O'*)tin(IV), (II) (Yahyi *et al.*, 2001). The ligands are chelated to atom Sn1 asymmetrically *via* both O atoms of the carboxylate groups in a bidentate manner. The geometry of the Sn atom is highly distorted octahedral (Table 1). The Sn—O bond lengths are in agreement with the isostructural analogue (II) [Sn—O = 2.1233 (13) and 2.5641 (14) Å]. The chelated aminobenzoate fragment Sn1/O3/O4/N1/C1–C7 is planar, with a maximum deviation from the least-squares plane of 0.029 (2) Å for atom O3. The molecules are stabilized by intra- and intermolecular N—H···O hydrogen bonds (Table 2), forming one-dimensional chains parallel to the b axis (Fig. 2).

Experimental

A solution of di-*n*-butyltin(IV) oxide (0.75 g, 3 mmol) and (2-amino-5-nitro)benzoic acid (1.09 g, 6 mmol) in methanol (60 ml) was
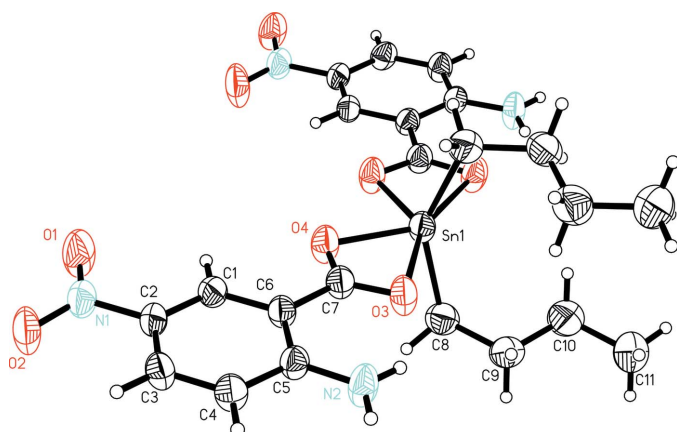
Figure 1
Molecular structure of (I), with 50% probability displacement ellipsoids. Unlabeled atoms are related to labeled atoms by $(1 - x, y, \frac{1}{2} - z)$.

refluxed for *ca* 4 h. The water formed during the reaction was removed by azeotropic dehydration using a Dean–Stark apparatus. The clear yellow solution was filtered and left for evaporation at room temperature. Yellow crystals were obtained after two weeks (yield 1.60 g, 90%; m.p. 482–483 K). Analysis found: C 43.91, H 4.46, N 8.85, Sn 19.08%; calculated: C 44.3, H 4.7, N 9.4, Sn 19.9%.

Crystal data

$[Sn(C_4H_9)_2(C_7H_5N_2O_4)_2]$	$D_x = 1.581$ Mg m^{-3}
$M_r = 595.17$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 5761
$a = 14.356$ (4) Å	reflections
$b = 9.006$ (2) Å	$\theta = 2.1$–25.5°
$c = 20.155$ (5) Å	$\mu = 1.08$ mm^{-1}
$\beta = 106.305$ (4)°	$T = 298$ (2) K
$V = 2501.0$ (11) Å^3	Plate, light yellow
$Z = 4$	$0.48 \times 0.44 \times 0.18$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2328 independent reflections
	2257 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.015$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 25.5°$
	$h = -17 \rightarrow 17$
$T_{min} = 0.626$, $T_{max} = 0.830$	$k = -10 \rightarrow 10$
6494 measured reflections	$l = -19 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$+ 2.0841P]$
$wR(F^2) = 0.060$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{max} < 0.001$
2328 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
160 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—O4	2.1238 (15)	Sn1—O3	2.4665 (16)
Sn1—C8	2.127 (2)		
O4—Sn1—O4^i	85.68 (8)	C8^i—Sn1—O3	89.31 (8)
C8^i—Sn1—C8	143.56 (14)	C8—Sn1—O3	84.95 (8)
O4—Sn1—O3	56.37 (6)	O3—Sn1—O3^i	161.57 (7)
O4^i—Sn1—O3	142.05 (5)		

Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$.
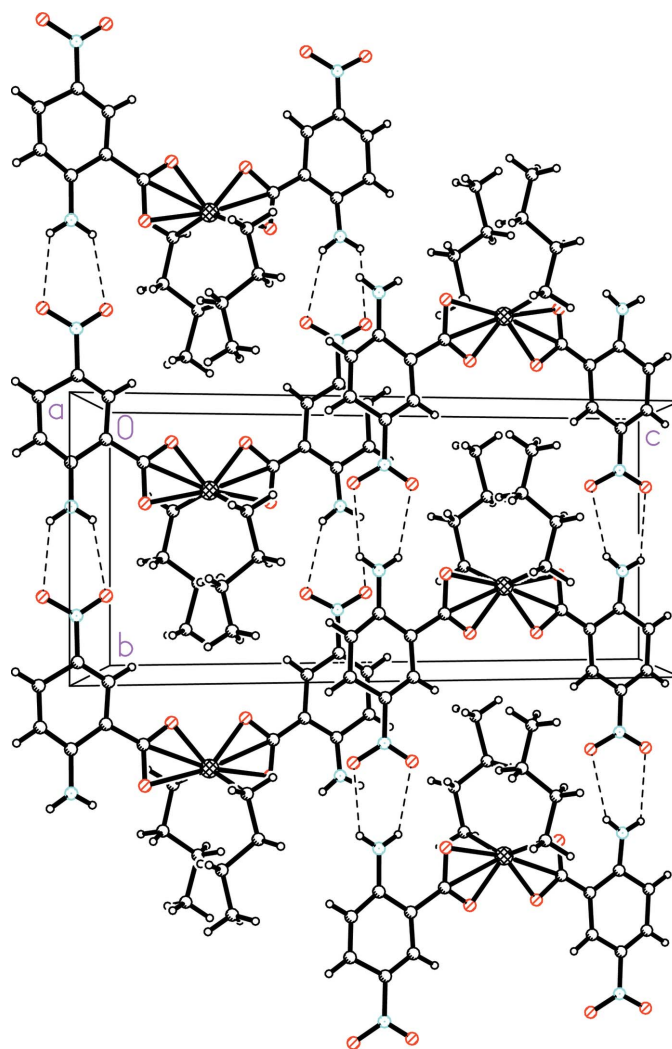
Figure 2
A packing diagram of (I). The dashed lines denote hydrogen bonds.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2B$\cdots$O3	0.86	2.02	2.665 (3)	132
N2—H2A$\cdots$O2^{ii}	0.86	2.47	3.036 (3)	124
N2—H2B$\cdots$O1^{ii}	0.86	2.45	3.079 (3)	130

Symmetry code: (ii) $x, y + 1, z$.

H atoms were located in difference-density maps and repositioned geometrically, with N—H = 0.86 Å and C—H = 0.93–0.97 Å. They were constrained to ride on their parent atoms, with U_{iso}(H) values of 1.2 (1.5 for methyl) times U_{eq}(C,N).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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