quite constant (1.414 Å average). This value is significantly greater than that for N—O in [$ZnCl_2$-$(pyo)_2$] (1.338 Å), showing that the pyo bond retains considerable π character even in the coordinated molecule.

The structural results eliminate the suggestion by Herlocker (1969) that differences between the electronic spectra of [$CoX_2(Me_3NO)_2$] complexes run as mulls and in solution are a result of five coordination through ligand bridging in the crystals. Reinterpretation would suggest that, in solution, the polar solvent molecules (dichloromethane and acetonitrile) interact additionally with the metal centre to alter the field symmetry, but on crystallization these more weakly attracted solvent molecules are excluded. The earlier finding (Herlocker & Drago, 1968) that excess of the rather strongly bonding, but sterically demanding, amine oxide ligand displaces the spectral features towards those of [$Co(Me_3NO)_4$]-$(ClO)_2$ (with ease of displacement I > Br > Cl) is compatible with this view,

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Structure of Dibutyltin Bis(*p*-nitrobenzoate)

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Abstract. [$Sn(C_4H_9)_2(C_7H_4NO_4)_2$], $M_r = 565.15$, monoclinic, $C2/c$, $a = 15.694$ (7), $b = 7.042$ (5), $c = 22.950$ (11) Å, $\beta = 103.64$ (4)°, $V = 2465$ (2) Å^3, $Z = 4$, $D_x = 1.53$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.08$ mm^{-1}, $F(000) = 1144$, $T = 293$ K, $R = 0.054$ for 1023 observed reflexions. The compound is monomeric. The Sn atom lies on a crystallographic twofold axis amd displays skew-trapezoidal bipyramidal coordination. The basal plane is defined by the two asymmetrically chelating carboxylate groups

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[Sn—O(1) 2.089 (7) and Sn—O(2) 2.645 (7) Å]. The C—Sn—C angle is 137.1 (6)°.

Introduction. Carboxylate groups in organotin carboxylates can act as monodentate, bidentate and bridging ligands (Tiekink, 1991). Most crystal structure determinations of organotin carboxylates have been for triorganotin carboxylates; relatively few diorganotin dicarboxylate structures have been determined. We now report the crystal structure of dibutyltin bis(*p*-nitrobenzoate).

Experimental. The title compound was prepared in 60% yield by reaction of Bu_2SnO and *p*-$NO_2C_6H_4CO_2H$ in CH_2Cl_2* (Klebanov & Shologon, 1979). A colourless crystal, 0.04 × 0.18 × 0.6 mm, was used. The cell dimensions were obtained from setting angles of 14 independent reflexions with $2\theta \simeq 20°$ on a Nicolet *P*3 automated diffractometer using monochromated Mo *Kα* radiation. The intensities of 2117 unique reflexions with $2\theta \leq 50°$ were measured from *ω* scans with a fixed width of 0.6°, scan rates related to pre-scan intensity in the range 1.0 to 29.3° min^{-1} and stationary crystal–stationary counter background counts taken at ±1.0° in *ω* from the calculated position of the Bragg peak to yield 1023 $F_o > 6\sigma(F_o)$; *h* 0–20, *k* 0–9, *l* −29–29. The data were corrected for Lorentz and polarization effects; absorption was ignored. Two reference reflexions, monitored periodically, showed no significant variation in intensities.

The structure was determined by the heavy-atom method (Patterson function) which revealed the approximate position of the Sn atom. Due to the crystallographically imposed twofold symmetry, the Sn atom is at a 4(*e*) site. All the remaining atoms, corresponding to single representative *n*-butyl and *p*-nitrobenzoate groups, occupy 8(*f*) general sites and were located from successive difference syntheses using *SHELX*76 (Sheldrick, 1976). H atoms, with the exception of the methyl H atoms, were given ideal geometries with C—H = 1.00 (2) Å and allowed to ride on attached C atoms. Coordinates of methyl H atoms were calculated in idealized positions and subsequently the methyl group was treated as a rigid body.

Full-matrix least-squares calculations on *F* with anisotropic thermal parameters for the Sn, N, O and non-butyl C atoms and with isotropic thermal parameters for butyl C and H atoms (136 parameters) converged at $R = 0.054$, $wR = 0.055$. Atomic scattering factors and anomalous-dispersion terms from *SHELX*76. Final $w = 1.2766/[\sigma^2(F) + 0.001000F^2]$, maximum $\Delta/\sigma = 0.1$, final $\Delta\rho_{max} = 0.2$, $\Delta\rho_{min} = -0.2$ e Å^{-3}.

* IR absorption frequencies (KBr disc); $\nu(NO_2)$ 1526 and 1343 cm^{-1}; $\nu(CO_2)$ asym. 1626 and 1586 cm^{-1}; $\nu(CO_2)$ sym. 1364 cm^{-1}; typical values for an uncomplexed conjugate NO_2 group (Williams & Fleming, 1989) and a chelating carboxylate group (Sandhu, Sharma & Tiekink, 1991; Vollano, Day, Rau, Chandrasekhar & Holmes, 1984).

Discussion. The structure of the title compound is shown in Fig. 1. Final atomic parameters are listed in Table 1 and bond lengths and angles in Table 2.* The carboxylate groups act as asymmetric chelating groups, which result in a six-coordinate Sn in the monomeric compound. The geometry about Sn is skew-trapezoidal bipyramidal with the O atoms of the chelating carboxylate forming the basal plane. The Sn—O(1) and Sn—O(2) bond lengths [2.089 (7) and 2.645 (7) Å respectively] are similar to those reported for other skew-trapezoidal bipyramidal $R_2Sn(O_2CR')_2$ compounds: *e.g.* for $R = R' =$ Me 2.106 (2) and 2.539 (2) Å (Lockhart, Calabrese & Davidson, 1987); $R =$ Me, $R' = C_6H_4NH_2$-*p* 2.077 (3)–2.097 (3) and 2.556 (3)–2.543 (3) Å (Chandrasekhar, Day, Holmes & Holmes, 1988); R = Bu, $R' = C_6H_4Br$-*p* 2.075 (3) and 2.635 (4) Å (Ng, Kumar Das, Skelton & White, 1989); R = Bu, $R' = CH_2SC_6H_5$ 2.134 (4) and 2.559 (5) Å (Sandhu, Sharma & Tiekink, 1989); R = Pr, $R' = CH_2SPh$ 2.114 (3) and 2.587 (4) Å (Sandhu, Sharma & Tiekink, 1991). In another diorganotin biscarboxylate, [$Bu_2Sn(OCOC_6H_4Br$-*o*$)_2$], having a skew-trapezoidal geometry, there are additional weak intermolecular Sn—O interactions [Sn···O 3.451 (5) Å] which provide weakly bridged dimers (Ng, Kumar Das, Yip, Wang & Mak, 1990). The O(1)—Sn—O(1′), O(2)—Sn—O(2′) and C—Sn—C

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54504 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0263]

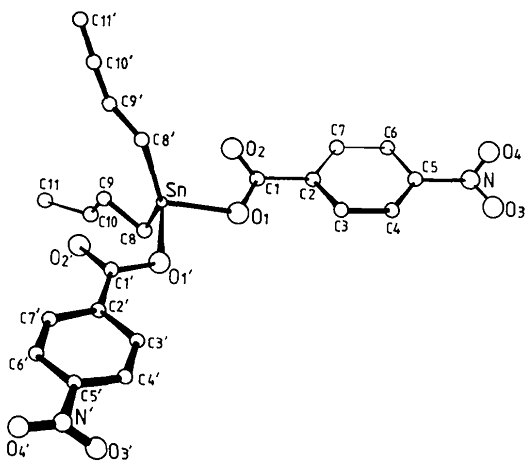

Fig. 1. The atomic arrangement in the molecule.

Table 1. *Coordinates* ($\times 10^4$) *for non-*H *atoms and* U_{eq} *or* U_{iso} *values* (Å$^2 \times 10^3$) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
Sn	5000†	−2968 (2)	7500†	71 (1)
N‡	6409 (6)	3321 (16)	4782 (4)	81 (4)
O(2)	5435 (5)	−3296 (9)	6459 (3)	87 (3)
O(1)	5239 (4)	−724 (10)	6965 (3)	73 (2)
O(3)	6330 (7)	5018 (13)	4836 (4)	116 (4)
O(4)	6698 (5)	2585 (11)	4390 (3)	97 (3)
C(1)‡	5444 (6)	−1566 (6)	6505 (4)	59 (4)
C(2)	5688 (6)	−293 (14)	6060 (4)	58 (3)
C(3)	5602 (7)	1680 (15)	6096 (4)	68 (4)
C(4)	5831 (7)	2847 (16)	5689 (4)	69 (3)
C(5)	6161 (6)	2055 (18)	5234 (4)	62 (3)
C(6)	6237 (7)	112 (16)	5177 (4)	66 (4)
C(7)	5991 (6)	−1022 (15)	5586 (4)	63 (3)
C(8)	6254 (9)	−4062 (21)	7893 (6)	116 (5)
C(9)	6214 (14)	−6213 (32)	7944 (9)	182 (7)
C(10)	7012 (14)	−7043 (35)	8182 (9)	194 (8)
C(11)	7034 (15)	−8784 (37)	8443 (11)	215 (9)

† Invariant by symmetry.
‡ Chemical identity of these atoms established from infrared spectroscopic data.

Table 2. *Bond lengths* (Å) *and valency angles* (°) *with e.s.d.'s in parentheses*

Sn—C(8)	2.11 (1)	C(1)—O(1)	1.32 (1)
Sn—O(1)	2.089 (7)	C(1)—O(2)	1.22 (1)
Sn—O(2)	2.645 (7)	C(2)—C(7)	1.38 (1)
C(1)—C(2)	1.48 (1)	C(2)—C(3)	1.40 (1)
C(3)—C(4)	1.35 (1)	C(4)—C(5)	1.39 (1)
C(5)—C(6)	1.38 (1)	C(6)—C(7)	1.36 (1)
N—O(3)	1.21 (1)	N—O(4)	1.22 (1)
N—C(5)	1.49 (1)	C(8)—C(9)	1.52 (2)
C(9)—C(10)	1.38 (2)	C(10)—C(11)	1.36 (3)
C(8)—Sn—C(8′)	137.1 (6)	O(1)—Sn—C(8)	104.4 (4)
O(1)—Sn—O(2)	54.2 (2)	O(1)—Sn—C(8′)	107.7 (4)
O(1)—Sn—O(2′)	135.9 (2)	O(2)—Sn—C(8)	86.6 (4)
O(2)—Sn—O(2′)	170.0 (2)	O(2)—Sn—C(8′)	89.7 (2)
O(1)—Sn—O(1′)	81.7 (3)	O(1)—C(1)—O(2)	121.2 (9)
Sn—O(1)—C(1)	104.0 (6)	C(1)—C(2)—C(3)	121.2 (9)
Sn—O(2)—C(1)	80.5 (6)	C(1)—C(2)—C(7)	120.8 (10)
O(1)—C(1)—C(2)	115.7 (9)	C(2)—C(3)—C(4)	121.1 (9)
O(2)—C(1)—C(2)	123.0 (9)	C(4)—C(5)—C(6)	121.6 (10)
C(3)—C(4)—C(5)	118.8 (11)	C(6)—C(7)—C(2)	122.1 (10)
C(5)—C(6)—C(7)	118.2 (9)	O(3)—N—O(4)	124.2 (10)
C(5)—N—O(3)	118.1 (10)	C(5)—N—O(4)	117.7 (10)
C(6)—C(5)—N	119.1 (10)	Sn—C(8)—C(9)	110.2 (12)
C(8)—C(9)—C(10)	113.8 (21)	C(9)—C(10)—C(11)	119.0 (24)
C(4)—C(5)—N	119.3 (11)	C(3)—C(2)—C(7)	118.1 (9)

bond angles in $Bu_2Sn(O_2CC_6H_4NO_2\text{-}p)_2$ [81.7 (3), 170.0 (2) and 137.1 (6)° respectively] are also similar to those in the related $R_2Sn(O_2CR')_2$ structures [*viz.* 79.5 (1)–81.8 (1), 168.0–171.1 (1) and 130.6 (2)–140.7 (1)° respectively]. The nitro groups in $Bu_2Sn(O_2CC_6H_4NO_2\text{-}p)$ are not involved in coordination to Sn, the shortest Sn—O(nitro) being 4.532 (8) Å.

Unlike the $R_2Sn(O_2CR')_2$ compounds mentioned above, the *o*-carboxylatopyridine complex, $Me_2Sn(O_2CC_5H_4N\text{-}o)_2$, has been reported to be polymeric with the Sn atom seven coordinate (Lockhart & Davidson, 1987), while the formate, $Me_2Sn(O_2CH)_2$ (with six-coordinate Sn), is a sheet polymer with linear Me_2Sn moieties nearly symmetrically bridged by formate anions (Mistry, Rettig, Trotter & Aubke, 1990).

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Structure of 7-Amino-4-trifluoromethylcoumarin

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Abstract. $C_{10}H_6F_3NO_2$, $M_r = 229.1$, triclinic, $P\bar{1}$, $a = 5.190$ (3), $b = 6.883$ (4), $c = 14.165$ (4) Å, $\alpha = 82.72$ (6), $\beta = 85.54$ (6), $\gamma = 72{\cdot}67$ (4)°, $V = 478.7$ (4) Å^3, $Z = 2$, $D_m = 1.59$, $D_x = 1.60$ g cm^{-3}, filtered Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, $\mu = 13.0$ cm^{-1}, $F(000) = 232$, $T = 296$ K, $R = 0.05$, $wR =$

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