

Aziz-ur-Rehman,^a Saqib Ali,^{a*}
 Madeleine Helliwell^b and Saira
 Shahzadi^a

^aDepartment of Chemistry, Quaid-i-Azam
 University, Islamabad 45320, Pakistan, and

^bSchool of Chemistry, University of Manchester,
 Manchester, M13 9PL, England

Correspondence e-mail: drsa54@yahoo.com

Key indicators

Single-crystal X-ray study

$T = 100$ K

Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å

R factor = 0.015

wR factor = 0.038

Data-to-parameter ratio = 17.6

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

(3-Amino-4-chlorobenzoato)trimethyltin(IV)

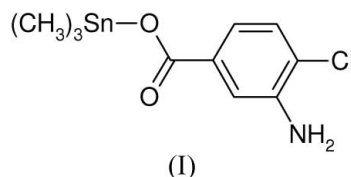
In the title compound, $[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_5\text{ClNO}_2)]$, the Sn atom is bonded to three methyl groups and one O atom in a distorted tetrahedral geometry, with Sn—C bond lengths of 2.118 (2)–2.119 (2) Å and an Sn—O bond length of 2.0804 (12) Å.

Received 23 June 2006

Accepted 3 July 2006

Comment

In view of our interest in the synthesis, characterization, biological applications and crystal structures of organotin carboxylates (Danish *et al.*, 1995; Parvez *et al.*, 2002; Sadiq-ur-Rehman *et al.*, 2006), we have synthesized a new organotin(IV) carboxylate of 4-chloro-3-aminobenzoic acid, the title compound, (I).



In compound (I) (Fig. 1), atom Sn1 is bonded to three methyl groups with essentially identical Sn—C distances, comparable with the values reported for the related structure $(\text{C}_{16}\text{H}_{13}\text{O}_3)\text{Sn}(\text{CH}_3)_3$ (Tahir *et al.*, 1997). The coordination geometry around Sn1 is distorted tetrahedral (Table 1).

The crystal structure of (I) contains centrosymmetric dimers formed *via* intermolecular N—H...O hydrogen bonds (Fig. 2, Table 2).

Experimental

The sodium salt of 4-chloro-3-aminobenzoic acid (0.194 g, 1 mmol) and trimethyltin chloride (0.199 g, 1 mmol) were suspended in dry toluene (150 ml) in a two-necked round-bottomed flask equipped with a water condenser. The mixture was refluxed for 8–10 h, the NaCl formed was filtered off, and the solvent was removed on a rotary evaporator under reduced pressure. The solid product was recrystallized from chloroform to obtain colourless crystals of (I) (yield 70%; m.p. 403–406 K).

Crystal data

$[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_5\text{ClNO}_2)]$

$M_r = 334.36$

Monoclinic, $P2_1/c$

$a = 11.9077$ (7) Å

$b = 9.1237$ (5) Å

$c = 12.6554$ (7) Å

$\beta = 113.086$ (1)°

$V = 1264.80$ (12) Å³

$Z = 4$

$D_x = 1.756$ Mg m⁻³

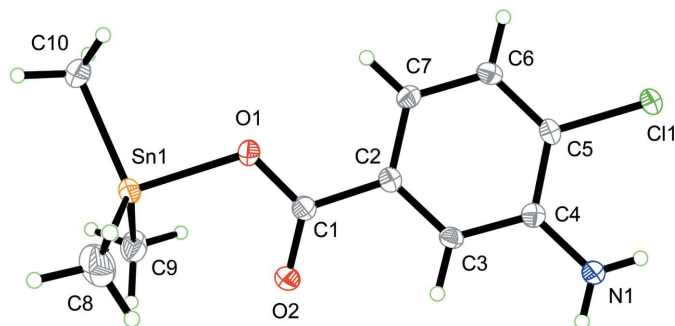
Mo $K\alpha$ radiation

$\mu = 2.21$ mm⁻¹

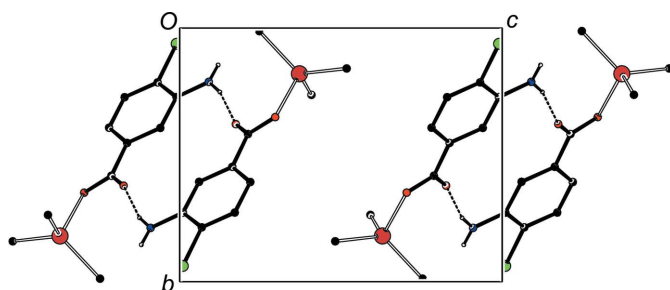
$T = 100$ (2) K

Block, colourless

0.40 × 0.30 × 0.30 mm

**Figure 1**

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

**Figure 2**

Centrosymmetric dimers formed through intermolecular hydrogen bonding (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.435$, $T_{\max} = 0.515$

9722 measured reflections
 2586 independent reflections
 2528 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 26.4^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.015$
 $wR(F^2) = 0.038$
 $S = 1.11$
 2586 reflections
 147 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0146P)^2 + 0.9615P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.005$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Sn1—O1	2.0804 (12)	Sn1—C9	2.119 (2)
Sn1—C8	2.118 (2)	Sn1—C10	2.119 (2)
O1—Sn1—C8	102.26 (7)	O1—Sn1—C9	106.60 (6)
O1—Sn1—C10	96.02 (6)	C8—Sn1—C9	118.66 (9)
C8—Sn1—C10	115.55 (9)	C10—Sn1—C9	113.69 (8)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H2N \cdots O2 ⁱ	0.83 (2)	2.14 (2)	2.947 (2)	166 (2)

Symmetry code: (i) $-x + 2, -y + 2, -z + 1$.

H atoms bound to C atoms were included in calculated positions and allowed to ride during subsequent refinement, with $C-H = 0.95 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ for Csp^2 , and $C-H = 0.98 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ for the methyl groups. The methyl groups were allowed to rotate about their local threefold axes. H atoms bound to N1 were located in a difference Fourier map and refined isotropically, with final N—H distances of 0.80 (2) and 0.83 (2) \AA .

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

AR is grateful to the HEC (Higher Education Commission, Islamabad, Pakistan) for financial support under the PhD Fellowship Scheme Batch-II (PIN Code 042-111621-PS2-179).

References

- Bruker (2001). SMART (Version 5.625) and SHELXTL (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2002). SAINT. Version 6.36a. Bruker AXS Inc., Madison, Wisconsin, USA.
- Danish, M., Ali, S., Mazhar, M., Badshah, A., Masood, T. & Tiekink, E. R. T. (1995). *Main Group Met. Chem.* **18**, 27–34.
- Parvez, M., Ali, S., Ahmad, S., Bhatti, M. H. & Mazhar, M. (2002). *Acta Cryst.* **C58**, m334–m335.
- Sadiq-ur-Rehman, Ali, S., Shahzadi, S. & Parvez, M. (2006). *Acta Cryst.* **E62**, m910–m911.
- Sheldrick, G. M. (2003). SADABS. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Tahir, M. N., Ülkü, D., Ali, S., Masood, T., Danish, M. & Mazhar, M. (1997). *Acta Cryst.* **C53**, 1574–1576.

supporting information

Acta Cryst. (2006). E62, m1778–m1779 [https://doi.org/10.1107/S1600536806025517]

(3-Amino-4-chlorobenzoato)trimethyltin(IV)

Aziz-ur-Rehman, Saqib Ali, Madeleine Helliwell and Saira Shahzadi

(3-Amino-4-chlorobenzoato)trimethyltin(IV)

Crystal data

[Sn(CH₃)₃(C₇H₅ClNO₂)]

$M_r = 334.36$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.9077$ (7) Å

$b = 9.1237$ (5) Å

$c = 12.6554$ (7) Å

$\beta = 113.086$ (1)°

$V = 1264.80$ (12) Å³

$Z = 4$

$F(000) = 656$

$D_x = 1.756$ Mg m⁻³

Melting point = 403–406 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8154 reflections

$\theta = 2.8$ – 26.4 °

$\mu = 2.21$ mm⁻¹

$T = 100$ K

Block, colourless

0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)

$T_{\min} = 0.435$, $T_{\max} = 0.515$

9722 measured reflections

2586 independent reflections

2528 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 1.9$ °

$h = -14 \rightarrow 14$

$k = -11 \rightarrow 11$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.015$

$wR(F^2) = 0.038$

$S = 1.11$

2586 reflections

147 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0146P)^2 + 0.9615P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.005$

$\Delta\rho_{\max} = 0.35$ e Å⁻³

$\Delta\rho_{\min} = -0.26$ e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.758545 (10)	0.680159 (12)	0.129024 (9)	0.01696 (5)
Cl1	0.63628 (4)	1.43820 (4)	0.49036 (3)	0.02033 (9)
O1	0.70022 (11)	0.85101 (13)	0.20397 (10)	0.0173 (2)
O2	0.89305 (11)	0.87859 (15)	0.32613 (11)	0.0269 (3)
N1	0.87650 (15)	1.28807 (19)	0.59015 (14)	0.0252 (3)
C1	0.78575 (15)	0.91776 (18)	0.28834 (14)	0.0164 (3)
C2	0.74554 (14)	1.04716 (18)	0.33707 (14)	0.0150 (3)
C3	0.82684 (14)	1.10631 (18)	0.43995 (14)	0.0156 (3)
H3	0.9053	1.0633	0.4770	0.019*
C4	0.79528 (15)	1.22799 (19)	0.48994 (14)	0.0163 (3)
C5	0.67799 (15)	1.28591 (18)	0.43147 (14)	0.0155 (3)
C6	0.59600 (15)	1.22758 (19)	0.32933 (14)	0.0169 (3)
H6	0.5173	1.2698	0.2924	0.020*
C7	0.62928 (15)	1.10714 (18)	0.28108 (14)	0.0161 (3)
H7	0.5738	1.0662	0.2111	0.019*
C8	0.7946 (2)	0.5117 (2)	0.25317 (19)	0.0388 (5)
H8A	0.8668	0.5374	0.3216	0.058*
H8B	0.8095	0.4194	0.2211	0.058*
H8C	0.7242	0.5001	0.2742	0.058*
C9	0.90513 (18)	0.7631 (2)	0.09166 (17)	0.0291 (4)
H9A	0.9051	0.8704	0.0949	0.044*
H9B	0.8956	0.7315	0.0146	0.044*
H9C	0.9825	0.7257	0.1482	0.044*
C10	0.59313 (17)	0.6591 (2)	-0.01744 (17)	0.0269 (4)
H10A	0.5240	0.6761	0.0046	0.040*
H10B	0.5875	0.5601	-0.0492	0.040*
H10C	0.5914	0.7312	-0.0754	0.040*
H1N	0.855 (2)	1.355 (3)	0.6190 (18)	0.021 (5)*
H2N	0.943 (2)	1.248 (3)	0.6245 (18)	0.025 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01860 (7)	0.01495 (7)	0.01740 (7)	0.00233 (4)	0.00713 (5)	-0.00167 (4)
Cl1	0.01977 (19)	0.01832 (19)	0.0220 (2)	0.00442 (15)	0.00727 (16)	-0.00461 (15)
O1	0.0180 (6)	0.0170 (6)	0.0170 (6)	-0.0001 (5)	0.0069 (5)	-0.0036 (5)
O2	0.0193 (6)	0.0336 (7)	0.0231 (7)	0.0098 (5)	0.0031 (5)	-0.0086 (6)
N1	0.0176 (8)	0.0267 (8)	0.0245 (8)	0.0061 (7)	0.0007 (7)	-0.0108 (7)
C1	0.0188 (8)	0.0173 (8)	0.0138 (7)	0.0020 (6)	0.0071 (6)	0.0016 (6)
C2	0.0168 (8)	0.0147 (8)	0.0153 (7)	0.0000 (6)	0.0084 (6)	0.0010 (6)

C3	0.0122 (7)	0.0169 (8)	0.0176 (8)	0.0012 (6)	0.0058 (6)	0.0000 (6)
C4	0.0161 (8)	0.0168 (8)	0.0165 (8)	-0.0003 (6)	0.0070 (6)	0.0004 (6)
C5	0.0183 (8)	0.0132 (7)	0.0175 (8)	0.0013 (6)	0.0098 (7)	-0.0005 (6)
C6	0.0145 (7)	0.0184 (8)	0.0174 (8)	0.0031 (6)	0.0058 (6)	0.0026 (6)
C7	0.0155 (8)	0.0181 (8)	0.0137 (7)	-0.0009 (6)	0.0048 (6)	-0.0003 (6)
C8	0.0566 (14)	0.0229 (10)	0.0363 (12)	0.0075 (10)	0.0177 (11)	0.0083 (9)
C9	0.0251 (9)	0.0346 (11)	0.0324 (10)	-0.0037 (8)	0.0163 (8)	-0.0112 (8)
C10	0.0232 (9)	0.0283 (10)	0.0253 (9)	0.0000 (7)	0.0054 (8)	-0.0097 (8)

Geometric parameters (Å, °)

Sn1—O1	2.0804 (12)	C4—C5	1.402 (2)
Sn1—C8	2.118 (2)	C5—C6	1.385 (2)
Sn1—C9	2.119 (2)	C6—C7	1.388 (2)
Sn1—C10	2.119 (2)	C6—H6	0.950
C11—C5	1.7387 (16)	C7—H7	0.950
O1—C1	1.302 (2)	C8—H8A	0.980
O2—C1	1.229 (2)	C8—H8B	0.980
N1—C4	1.372 (2)	C8—H8C	0.980
N1—H1N	0.80 (2)	C9—H9A	0.980
N1—H2N	0.83 (2)	C9—H9B	0.980
C1—C2	1.495 (2)	C9—H9C	0.980
C2—C3	1.391 (2)	C10—H10A	0.980
C2—C7	1.396 (2)	C10—H10B	0.980
C3—C4	1.400 (2)	C10—H10C	0.980
C3—H3	0.950		
O1—Sn1—C8	102.26 (7)	C5—C6—C7	119.77 (15)
O1—Sn1—C10	96.02 (6)	C5—C6—H6	120.1
C8—Sn1—C10	115.55 (9)	C7—C6—H6	120.1
O1—Sn1—C9	106.60 (6)	C6—C7—C2	118.97 (15)
C8—Sn1—C9	118.66 (9)	C6—C7—H7	120.5
C10—Sn1—C9	113.69 (8)	C2—C7—H7	120.5
C1—O1—Sn1	115.51 (10)	Sn1—C8—H8A	109.5
C4—N1—H1N	119.1 (16)	Sn1—C8—H8B	109.5
C4—N1—H2N	119.6 (16)	H8A—C8—H8B	109.5
H1N—N1—H2N	121 (2)	Sn1—C8—H8C	109.5
O2—C1—O1	122.87 (15)	H8A—C8—H8C	109.5
O2—C1—C2	121.43 (15)	H8B—C8—H8C	109.5
O1—C1—C2	115.70 (14)	Sn1—C9—H9A	109.5
C3—C2—C7	120.71 (15)	Sn1—C9—H9B	109.5
C3—C2—C1	118.33 (14)	H9A—C9—H9B	109.5
C7—C2—C1	120.96 (15)	Sn1—C9—H9C	109.5
C2—C3—C4	121.26 (15)	H9A—C9—H9C	109.5
C2—C3—H3	119.4	H9B—C9—H9C	109.5
C4—C3—H3	119.4	Sn1—C10—H10A	109.5
N1—C4—C3	121.18 (15)	Sn1—C10—H10B	109.5
N1—C4—C5	122.16 (16)	H10A—C10—H10B	109.5

C3—C4—C5	116.65 (15)	Sn1—C10—H10C	109.5
C6—C5—C4	122.64 (15)	H10A—C10—H10C	109.5
C6—C5—C11	119.47 (13)	H10B—C10—H10C	109.5
C4—C5—C11	117.89 (13)		
C8—Sn1—O1—C1	75.15 (13)	C2—C3—C4—N1	-178.61 (16)
C10—Sn1—O1—C1	-167.03 (12)	C2—C3—C4—C5	0.4 (2)
C9—Sn1—O1—C1	-50.09 (13)	N1—C4—C5—C6	178.94 (17)
Sn1—O1—C1—O2	-3.8 (2)	C3—C4—C5—C6	0.0 (2)
Sn1—O1—C1—C2	175.90 (10)	N1—C4—C5—C11	-0.4 (2)
O2—C1—C2—C3	-12.0 (2)	C3—C4—C5—C11	-179.37 (12)
O1—C1—C2—C3	168.30 (14)	C4—C5—C6—C7	-0.2 (3)
O2—C1—C2—C7	168.20 (16)	C11—C5—C6—C7	179.18 (13)
O1—C1—C2—C7	-11.5 (2)	C5—C6—C7—C2	0.0 (2)
C7—C2—C3—C4	-0.5 (2)	C3—C2—C7—C6	0.4 (2)
C1—C2—C3—C4	179.62 (15)	C1—C2—C7—C6	-179.81 (15)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H2N \cdots O2 ⁱ	0.83 (2)	2.14 (2)	2.947 (2)	166 (2)

Symmetry code: (i) $-x+2, -y+2, -z+1$.