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Bis[μ-furan-2-carbaldehyde (1-carboxyethylidene)hydrazonato(2−)]bis[methanoldimethyltin(IV)]

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In the title complex, $[Sn_2(CH_3)_4(C_8H_6N_2O_4)_2(CH_4O)_2]$, each Sn^{IV} ion exists in a distorted pentagonal–bipyramidal coordination environment, coordinated by three O atoms and one N atom from the pyruvic acid 2-furoic acid hydrazone ligands, one O atom from a methanol molecule and two axial C atoms from *trans* methyl groups, thus forming a dimeric molecule, which has crystallographic $\overline{1}$ symmetry. In the dimeric structure there are also intramolecular hydrogen bonds, which contribute to the crystal stability and compactness.

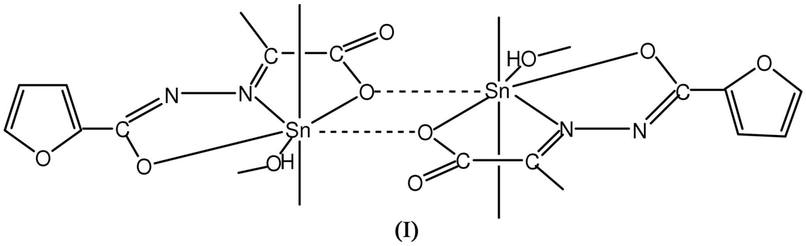
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.037
wR factor = 0.091
Data-to-parameter ratio = 14.1

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

Comment

Fig. 1 demonstrates that, in this complex, the Sn atom exists in a distorted pentagonal–bipyramidal coordination environment in which one methanol molecule, two tridentate pyruvic acid 2-furoic acid hydrazone ligands, and two *trans* methyl groups coordinate to each Sn center. The atoms O1, O5, O1^{i}, O3 and N1 are coplanar to within 0.0302 Å [symmetry code: $-x, -y+2, -z$], and form the equatorial plane. The inter-axial angle C9—Sn1—C10 is 163.0 (2)°, which deviates from the ideal value of 180°. Atom O1 of the carboxylate residue also binds another Sn atom, Sn1^{i}, generating an Sn_2O_2 four-membered ring. Thus, the structure of this complex can be described as a dimer, with crystallographically imposed $\overline{1}$ symmetry.



(I)

Each Sn atom is also coordinated by a methanol molecule. The Sn—O methanol bond distance [2.409 (4) Å] is longer than those in some analogues (Yin *et al.*, 2003; Parulekar *et al.*, 1989), owing to the formation of intramolecular hydrogen bonds (as in Hong *et al.*, 2005). These hydrogen bonds (Fig. 2) contribute to the crystal stability and compactness.

Experimental

Pyruvic acid 2-furoic acid hydrazone (1 mmol) and sodium ethoxide (1 mmol) were added to dry benzene (20 ml) in a Schlenk flask and stirred for 0.5 h. Dimethyltin dichloride (1 mmol) was added to the reactor; the reaction mixture was stirred for 12 h at 313 K and then filtered. The solvent was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from methanol and colourless crystals suitable for X-ray diffraction were obtained (m.p. 381.5 K). Elemental analysis calculated for $C_{11}H_{16}N_2O_5Sn$: C 35.24, H 4.30, N 7.47; found: C 35.11, H 4.23, N 7.60%.

Crystal data

$[Sn_2(CH_3)_4(C_8H_6N_2O_4)_2(CH_4O)_2]$
$M_r = 374.95$
Monoclinic, $C2/c$
$a = 20.794\ (19)$ Å
$b = 9.911\ (9)$ Å
$c = 14.142\ (13)$ Å
$\beta = 102.658\ (15)°$
$V = 2844\ (5)$ Å^3
$Z = 8$
$D_x = 1.752$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.81$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
0.23 × 0.15 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.680$, $T_{\max} = 0.812$
7350 measured reflections
2538 independent reflections
1798 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 25.1°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.091$
$S = 1.00$
2538 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 1.0171P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C9	2.082 (6)	Sn1—O1	2.316 (4)
Sn1—C10	2.098 (6)	Sn1—O5	2.409 (4)
Sn1—O3	2.178 (4)	Sn1—O1^i	2.747 (4)
Sn1—N1	2.248 (4)		
C9—Sn1—C10	163.0 (2)	C9—Sn1—O5	86.8 (2)
C9—Sn1—O3	95.7 (2)	C9—Sn1—O1^i	82.67 (19)
C9—Sn1—N1	94.3 (2)	O3—Sn1—O1^i	153.79 (11)
O3—Sn1—N1	70.06 (15)	N1—Sn1—O1^i	136.11 (13)
C9—Sn1—O1	89.70 (19)	O1—Sn1—O1^i	66.51 (12)
O3—Sn1—O1	139.69 (12)	O5—Sn1—O1^i	76.88 (13)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1···O2^i	0.84 (6)	1.82 (6)	2.643 (6)	166 (6)

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

H atoms attached to C atoms were all positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å and methyl C—H distances of 0.96 Å. The $U_{\rm iso}$(H) values were set at $1.5U_{\rm eq}$(C) for the methyl H atoms and at $1.2U_{\rm eq}$(C) for the other C-bound H atoms. The coordinates of the H atom bonded to O were refined, giving an O—H distance of 0.84 (6) Å; the $U_{\rm iso}$(H) value was 70 (20) Å^2 × 10^3.

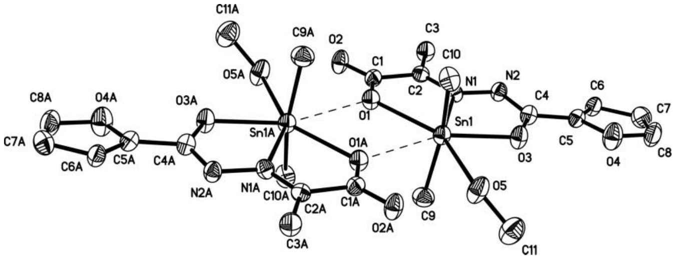


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. The long Sn—O bonds are indicated by dashed lines. The suffix A corresponds to symmetry code i in Table 1.

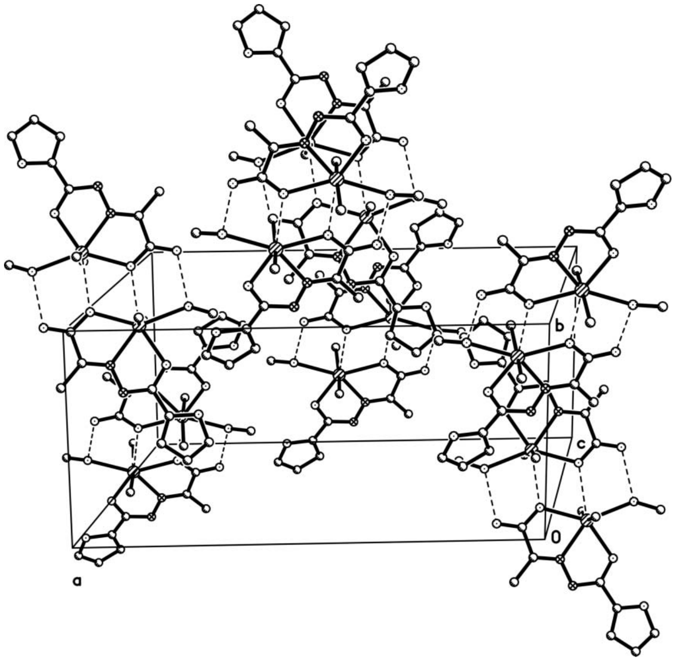


Figure 2
The packing of the title complex. Hydrogen bonds and the long Sn—O bonds are indicated by dashed lines. H atoms have been omitted for clarity.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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