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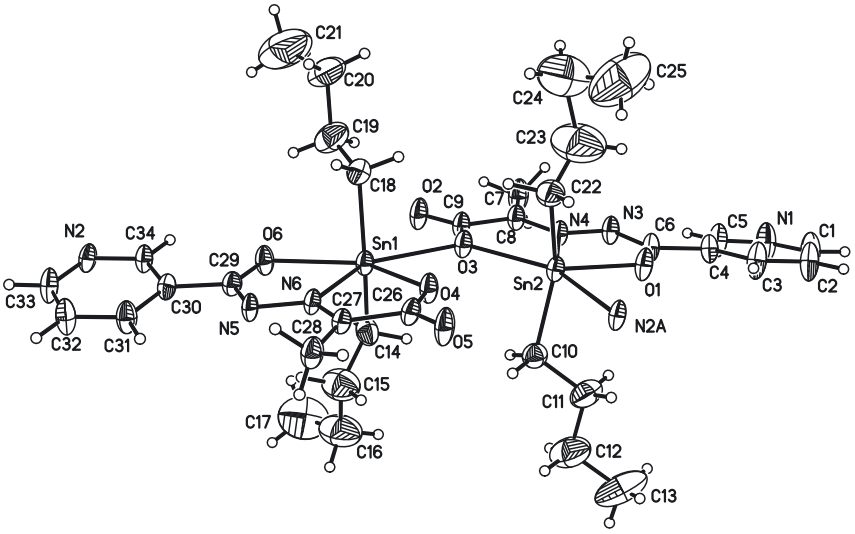
Crystal structure of *catena*-poly[tetrakis(butyl)-μ_2-2-((oxido(phenyl)methylene)hydrazineylidene) propanoato-κ^4*O:O,O′*,N-μ_2-2-((oxido(phenyl) methylene)hydrazineylidene)propanoato-κ^4*O,N,O′:N′*-ditin(IV)], $C_{34}H_{50}N_6O_6Sn_2$



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Abstract

$C_{34}H_{50}N_6O_6Sn_2$, triclinic, $P\bar{1}$, a = 12.110(2) Å, b = 12.406(2) Å, c = 13.744(3) Å, α = 79.547(2)°, β = 82.072(2)°, γ = 87.257(2)°, V = 2010.7(7) Å^3, Z = 2, $R_{gt}(F)$ = 0.0471, $wR_{ref}(F^2)$ = 0.1392, T = 298 K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.29 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,055, 6951, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5574
$N(param)_{refined}$:	439
Programs:	Bruker [1], SHELX [2], Olex2 [3]

1 Source of material

A mixture of dibutyltin oxide (0.4979 g, 2.0 mmol) and pyruvic acid nicotinyl hydrazone (0.4144 g, 2.0 mmol), in methanol (30 mL) was heated under reflux for 5 h. The obtained clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/ethanol (1:1). Yield 0.6659 g, 76 %, m.p. 467 K.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	Y	z	U_{iso}*/U_{eq}
Sn1	0.29540 (3)	0.11744 (3)	0.18751 (3)	0.04148 (14)
Sn2	0.26282 (3)	0.44821 (3)	0.25646 (3)	0.04228 (14)
O1	0.2271 (4)	0.5870 (3)	0.3297 (3)	0.0568 (12)
O2	0.2117 (5)	0.1128 (3)	0.4189 (4)	0.0656 (13)
O3	0.2600 (4)	0.2549 (3)	0.2984 (3)	0.0482 (10)
O4	0.3340 (4)	0.2779 (3)	0.1026 (3)	0.0509 (11)
O5	0.3960 (5)	0.3734 (3)	−0.0453 (4)	0.0689 (14)
O6	0.2960 (4)	−0.0657 (3)	0.1673 (3)	0.0530 (11)
N1	0.0557 (7)	0.7378 (5)	0.6083 (5)	0.089 (2)
N2	0.3044 (5)	−0.3857 (4)	0.1177 (4)	0.0516 (13)
N3	0.1661 (5)	0.4705 (4)	0.4762 (4)	0.0493 (13)
N4	0.1989 (4)	0.3922 (4)	0.4181 (4)	0.0455 (12)
N5	0.3467 (4)	−0.0022 (4)	−0.0009 (4)	0.0443 (12)
N6	0.3457 (4)	0.0982 (4)	0.0292 (4)	0.0423 (11)
C1	0.0861 (7)	0.8376 (6)	0.5609 (6)	0.0794 (19)
H1	0.065043	0.896823	0.592808	0.095*
C2	0.1465 (7)	0.8580 (6)	0.4680 (6)	0.0756 (18)
H2	0.165236	0.929078	0.437398	0.091*
C3	0.1788 (6)	0.7705 (5)	0.4210 (6)	0.0695 (17)
H3	0.218221	0.781959	0.357097	0.083*
C4	0.1524 (6)	0.6666 (5)	0.4688 (5)	0.0555 (14)
C5	0.0908 (6)	0.6555 (6)	0.5614 (6)	0.0671 (16)
H5	0.072216	0.584928	0.593719	0.080*
C6	0.1842 (6)	0.5686 (5)	0.4211 (5)	0.0521 (14)
C7	0.1488 (7)	0.2503 (6)	0.5654 (5)	0.070 (2)
H7A	0.193598	0.279672	0.606374	0.106*
H7B	0.153936	0.171626	0.579306	0.106*
H7C	0.072549	0.273737	0.579669	0.106*
C8	0.1892 (5)	0.2896 (5)	0.4597 (5)	0.0483 (14)
C9	0.2222 (5)	0.2111 (5)	0.3882 (5)	0.0497 (14)
C10	0.1154 (6)	0.4407 (6)	0.1920 (6)	0.0626 (17)
H10A	0.135727	0.426690	0.124585	0.075*
H10B	0.072654	0.379105	0.229745	0.075*
C11	0.0419 (7)	0.5431 (8)	0.1882 (8)	0.090 (2)
H11A	0.025121	0.560089	0.254944	0.108*
H11B	0.082469	0.604006	0.146505	0.108*
C12	−0.0654 (9)	0.5318 (10)	0.1482 (9)	0.115 (3)
H12A	−0.049520	0.508070	0.084150	0.138*
H12B	−0.110274	0.476558	0.193602	0.138*
C13	−0.1287 (12)	0.6380 (13)	0.1363 (12)	0.169 (5)
H13A	−0.132751	0.667944	0.196414	0.253*
H13B	−0.202764	0.626311	0.123396	0.253*
H13C	−0.091612	0.688191	0.081481	0.253*
C14	0.1196 (6)	0.1127 (7)	0.2013 (6)	0.0674 (16)
H14A	0.095077	0.050395	0.252011	0.081*
H14B	0.088888	0.178314	0.225016	0.081*
C15	0.0730 (8)	0.1051 (10)	0.1114 (8)	0.100 (2)
H15A	0.106308	0.041350	0.085918	0.120*
H15B	0.094567	0.169151	0.061669	0.120*
C16	−0.0553 (9)	0.0963 (11)	0.1228 (10)	0.120 (3)
H16A	−0.088981	0.154984	0.155715	0.144*
H16B	−0.078431	0.107351	0.056792	0.144*
C17	−0.0973 (12)	−0.0063 (13)	0.1782 (11)	0.159 (4)
H17A	−0.036868	−0.050993	0.202996	0.239*
H17B	−0.132127	−0.043449	0.135361	0.239*
H17C	−0.151094	0.006987	0.233166	0.239*
C18	0.4460 (6)	0.0776 (6)	0.2496 (6)	0.0665 (17)
H18A	0.503081	0.062897	0.196551	0.080*
H18B	0.468085	0.141900	0.272465	0.080*
C19	0.4456 (8)	−0.0154 (9)	0.3325 (7)	0.097 (2)
H19A	0.420544	−0.079566	0.311442	0.117*
H19B	0.392133	0.000521	0.387744	0.117*
C20	0.5599 (9)	−0.0426 (10)	0.3698 (8)	0.109 (3)
H20A	0.578791	0.012189	0.406496	0.130*
H20B	0.618072	−0.046037	0.314216	0.130*
C21	0.5449 (13)	−0.1632 (13)	0.4430 (12)	0.180 (5)
H21A	0.496835	−0.155524	0.503119	0.270*
H21B	0.616455	−0.191709	0.459325	0.270*
H21C	0.512669	−0.212735	0.409209	0.270*
C22	0.4376 (7)	0.4339 (10)	0.2513 (9)	0.103 (3)
H22A	0.461850	0.362441	0.235996	0.124*
H22B	0.471322	0.488831	0.197352	0.124*
C23	0.4761 (10)	0.4461 (15)	0.3389 (13)	0.169 (4)
H23A	0.434471	0.397477	0.393523	0.203*
H23B	0.458399	0.520512	0.349512	0.203*
C24	0.6050 (12)	0.4231 (15)	0.3461 (14)	0.170 (4)
H24A	0.617310	0.378218	0.409437	0.204*
H24B	0.640985	0.388180	0.291972	0.204*
C25	0.6407 (12)	0.5263 (17)	0.3382 (15)	0.212 (6)
H25A	0.593666	0.576675	0.299861	0.318*
H25B	0.716078	0.531228	0.305560	0.318*
H25C	0.637547	0.544299	0.403603	0.318*
C26	0.3682 (5)	0.2887 (4)	0.0082 (5)	0.0466 (14)
C27	0.3723 (5)	0.1844 (4)	−0.0347 (4)	0.0428 (12)
C28	0.4048 (6)	0.1871 (5)	−0.1431 (5)	0.0574 (16)
H28A	0.456243	0.127495	−0.153022	0.086*
H28B	0.439643	0.255334	−0.172565	0.086*
H28C	0.339671	0.180124	−0.173954	0.086*
C29	0.3190 (5)	−0.0799 (5)	0.0780 (5)	0.0466 (12)
C30	0.3185 (5)	−0.1923 (5)	0.0540 (5)	0.0456 (12)
C31	0.3328 (6)	−0.2126 (5)	−0.0413 (5)	0.0563 (14)
H31	0.342047	−0.155207	−0.095364	0.068*
C32	0.3333 (7)	−0.3204 (5)	−0.0563 (5)	0.0637 (15)
H32	0.342249	−0.336027	−0.120593	0.076*
C33	0.3206 (6)	−0.4032 (5)	0.0244 (5)	0.0617 (16)
H33	0.323489	−0.475173	0.013474	0.074*
C34	0.3040 (5)	−0.2819 (5)	0.1311 (5)	0.0495 (13)
H34	0.293343	−0.268583	0.196277	0.059*

2 Experimental details

H atoms were included in the riding model approximation with C–H 0.93–0.97 Å, and with $U_{iso}(H)$ = 1.2U_{eq}(C) or 1.5U_{eq} (methyl C).

3 Comment

The studies of organotin(IV) compounds are of current interest owing to their wide range of applications as biocides and homogeneous catalysts in industry. Recently organotin(IV) compounds with Schiff bases have attracted considerable attention owing to their biological activity and diverting structure [4–8]. We are exploring whether or not the hetero-atoms (N, O or S) of the carboxylic acid are to influence the coordination mode and extension of diaryltin(IV) compounds with heteroatom carboxylate ligands. We have synthesized and structurally characterized a new dibutyltin(IV) compound with pyruvic acid nicotinyl hydrazone.

In this compound, every tin atom is six-coordination in a distorted tetragonal bipyramidal geometry and center tin atom is surrounded equatorially by four O or N atoms from the nicotinyl hydrazone ligand, and axis position were occupied by two C atoms from the trans butyl groups (see the figure). It is quit clear that the identical hydrazone ligands coordinate in a different way (see name in the title and figure) [9].

The whole structure consists of molecular units connected by intermolecular Sn–N interactions and formed a one-dimensional chain structure. The bond length of Sn2–N2A 2.558(5) Å, Sn1–N6 2.229(4) Å and Sn2–N4 2.245(5) Å, which is greater than the sum of the covalent radii of Sn and N (2.15 Å), but is considerably less than the sum of the van der Waals radii (3.75 Å), and should be considered as bonding interactions [10].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. BRUKER. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
4. Gao Z. J., Zhang H. F. Crystal structure of catena-poly[tribenzyl-κ^1C-(μ_2-6-oxidopyridin-1-ium-3-carboxylato-κ^2O:O')tin(IV)–dichloromethane–methanol (1/1/1), $C_{29}H_{31}Cl_2NO_4Sn$. *Z. Kristallogr. N. Cryst. Struct.* 2022, *237*, 663–665.
5. Zuo D. S., Jiang T., Guan H. S., Wang K. Q., Qi X., Shi Z. Synthesis, structure and antitumor activity of dibutyltin oxide complexes with 5-fluorouracil derivatives. Crystal structure of [(5-fluorouracil)-1–$CH_2CH_2COOSn(n\text{–}Bu)_2]_4O_2$. *Molecules* 2001, *6*, 647–654.
6. Lee S. M., Lo K. M., Tiekink E. R. T. Crystal structure of 4-(dimethylamino)pyridinium dibromido-tris(4-chlorophenyl-κC^1) stannate(IV), $C_{25}H_{23}Br_2Cl_3N_2Sn$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1267–1269.
7. Ma C. L., Zhang J. H., Tian G. R., Zhang R. F. Syntheses, crystal structures and coordination modes of tri- and di-organotin derivatives with 2-mercapto-4-methylpyrimidine. *J. Organomet. Chem.* 2005, *690*, 519–533.
8. Ashfaq M. Synthesis of novel bioactive phthalimido-4-methyl pentanoateorganotin(IV) esters with spectroscopic investigation. *J. Organomet. Chem.* 2006, *691*, 1803–1808.
9. Yin H. D., Hong M., Li G., Wang D. Q. Synthesis, characterization and structural studies of diorganotin(IV) complexes with Schiff base ligand salicylaldehyde isonicotinylhydrazone. *J. Organomet. Chem.* 2005, *690*, 3714–3719.
10. Yin H. D., Chen S. W., Li L. W., Wang D. Q. Synthesis, characterization and structural of the organotin(IV) compounds with Schiff base ligands of pyruvic acid thiophene-2-carboxylic hydrazone and salicylaldehyde thiophene-2-carboxylic hydrazone. *Inorg. Chim. Acta* 2007, *360*, 2215–2223.