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Crystal structure of tetrakis(*n*-butyl)-(μ_2 -1,2-bis(2-oxidobenzoyl)hydrazine-1,2-diido- $\kappa^6 N, O, O': N', O'', O'''$)ditin(IV), $C_{30}H_{44}N_2O_4Sn_2$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.26 × 0.16 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.61 mm ⁻¹
Diffractometer, scan mode:	SMART CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7943, 2782, 0.069
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1637
$N(\text{param})_{\text{refined}}$:	174
Programs:	Bruker [1], SHELX [2, 3]

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

N-(2-hydroxybenzoyl)-2-hydroxybenzoyl hydrazide was synthesized according to the reference [4]. The other reagents were commercially available and used as purchased [2, 3, 5].

Experimental details

A mixture of bis(salicyl)hydrazide (88.88 mg, 0.4 mmol), tetrabutylammonium hydroxide (103.79 mg, 0.4 mmol), dibutyltin dichloride (208.42 mg, 0.8) and methanol (10 mL) heated at 60 °C for 10 h, and then cooled to room temperature. The resulting block crystals of the compound were obtained and washed with distilled water. The yield is ca. 25%. IR(KBr): 3217(s), 1518(s), 1519(s), 1416(s), 1405(s), 1319(s), 1216(s), 1116(s), 1105(s), 1012(s), 1001(s), 915(s), 819(s), 804(m), 761(m), 721(m), 600(m), 444(m) cm⁻¹.

Comment

Acyl hydrazide complexes have been an important research subject for their extensive biological and pharmacological activity [4]. The activity of its derivatives increased significantly when it formed a stable complex with metal cations in biological cells. In the ligand structure of *o*-carboxyl, hydroxyl benzoylhydrazide, which is located in the *ortho* carboxyl

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Abstract

$C_{30}H_{44}N_2O_4Sn_2$, monoclinic, $P2_1/c$, $a = 8.8810(8)$ Å, $b = 16.8999(13)$ Å, $c = 10.5705(10)$ Å, $\beta = 92.0460(10)^\circ$, $V = 1585.5(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0660$, $wR_{\text{ref}}(F^2) = 0.1528$, $T = 298(2)$ K.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Sn1	0.72189(7)	−0.01162(4)	0.43518(5)	0.0693(3)
N1	0.9354(7)	0.0048(3)	0.5363(6)	0.0582(17)
O1	1.1006(7)	0.0310(4)	0.6964(5)	0.0813(18)
O2	0.6280(7)	0.0155(4)	0.6044(5)	0.0804(18)
C1	0.9675(11)	0.0221(5)	0.6546(8)	0.064(2)
C2	0.8425(9)	0.0318(5)	0.7442(7)	0.0604(19)
C3	0.8943(10)	0.0495(5)	0.8696(7)	0.067(2)
H3	0.9972	0.0533	0.8881	0.081*
C4	0.7939(10)	0.0612(5)	0.9641(8)	0.075(2)
H4	0.8294	0.0688	1.0470	0.090*
C5	0.6442(11)	0.0617(5)	0.9367(8)	0.074(2)
H5	0.5768	0.0727	0.9997	0.089*
C6	0.5925(10)	0.0462(5)	0.8166(8)	0.076(2)
H6	0.4891	0.0460	0.7992	0.091*
C7	0.6888(10)	0.0307(5)	0.7195(7)	0.063(2)
C8	0.763(2)	−0.3159(10)	0.4022(18)	0.248(8)
H8A	0.6814	−0.3104	0.3405	0.372*
H8B	0.7785	−0.3709	0.4209	0.372*
H8C	0.8528	−0.2938	0.3693	0.372*
C9	0.7244(17)	−0.2733(7)	0.5200(16)	0.188(5)
H9A	0.7783	−0.2968	0.5921	0.226*
H9B	0.6173	−0.2778	0.5333	0.226*
C10	0.7681(14)	−0.1853(7)	0.5085(15)	0.165(5)
H10A	0.7737	−0.1629	0.5930	0.198*
H10B	0.8685	−0.1828	0.4755	0.198*
C11	0.6661(12)	−0.1330(6)	0.4267(11)	0.119(4)
H11A	0.5632	−0.1395	0.4527	0.143*
H11B	0.6703	−0.1505	0.3395	0.143*
C12	0.775(2)	0.2489(9)	0.1736(15)	0.230(7)
H12A	0.8786	0.2369	0.1606	0.346*
H12B	0.7546	0.3029	0.1505	0.346*
H12C	0.7119	0.2145	0.1221	0.346*
C13	0.7411(17)	0.2367(7)	0.3107(15)	0.174(5)
H13A	0.6339	0.2439	0.3216	0.209*
H13B	0.7940	0.2766	0.3611	0.209*
C14	0.7875(14)	0.1546(6)	0.3589(13)	0.151(4)
H14A	0.8747	0.1368	0.3140	0.181*
H14B	0.8165	0.1581	0.4481	0.181*
C15	0.6617(11)	0.0941(5)	0.3414(10)	0.100(3)
H15A	0.6435	0.0837	0.2519	0.119*
H15B	0.5696	0.1149	0.3753	0.119*

group, the hydroxyl group and the amide group have a variety of structure and properties when they coordinate with metal [4–6]. The hydrazide complexes containing tin(IV) are rare.

The compound crystallizes in the monoclinic space group *P*2₁/*c* with two formula units in the unit cell. As shown in Figure 1, one formula unit consists of one acyl hydrazide ligands, two Sn atoms and four butyl groups. Each Sn ion is five-fold coordinated with one nitrogen atoms, two oxygen atoms from the acyl hydrazide ligand and two carbon atoms from butyl groups. The bond length of Sn–N is 2.161 Å, while the

bond length of Sn–O range from 2.052 Å to 2.164 Å, the bond length of Sn–C are 2.103 Å and 2.111 Å.

Figure 1: The molecule structure of the compound.

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References

1. Bruker. SMART and SAINT for Windows NT Software Reference Manuals, Version 5.0, Bruker Analytical X-Ray Systems, Madison, WI (1997).
2. Sheldrick, G. M.: SADABS, a Software for Empirical Absorption Correction. University of Göttingen, Göttingen, Germany (1997).
3. SHELXL Reference Manual, version 5.1. Bruker Analytical X-Ray Systems, Madison, WI (1997).
4. Moon, D.; Lah, M. S.: Size and shape selectivity of host networks built based on tunable secondary building units. *Inorg. Chem.* **44** (2005) 1934–1940.
5. Mondal, S.; Schwederski, B.; Frey, W.; Fiedler, J.; Zálíš, S.; Kaim, W.: At the borderline between metal–metal mixed valency and a radical bridge situation: four charge states of a diruthenium complex with a redox-active bis(mer-tridentate) ligand. *Inorg. Chem.* **57** (2018) 3983–3992.
6. Cheaib, K.; Martel, D.; Clément, N.; Eckes, F.; Kouaho, S.; Rogez, G.; Dagorne, S.; Kurmoo, M.; Choua, S.; Welter, R.: Structural, magnetic and optical properties of an FeII dimer bridged by the meridional planar divergent N,N'-bis(salicyl)hydrazide and its photo- and electro-chemistry in solution. *Dalton Trans.* **42** (2013) 1406–1416.