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Some bioactive organotin(IV) derivatives with 3,4-dichlorophenylacetic acid: synthesis, spectroscopic properties, and X-ray structure of [Sn₄(C₄H₉)₈(OOCCH₂C₆H₃Cl₂)₄O₂]

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Some bioactive organotin(IV) derivatives with 3,4-dichlorophenylacetic acid: synthesis, spectroscopic properties, and X-ray structure of $[\text{Sn}_4(\text{C}_4\text{H}_9)_8(\text{OOCCH}_2\text{C}_6\text{H}_3\text{Cl}_2)_4\text{O}_2]$

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Some bioactive organotin(IV) derivatives of 3,4-dichlorophenylacetic acid (HL) were synthesized by reacting the ligand acid with various di- or tri-organotin compounds (in suitable mole ratios) in the presence of triethylamine. The structural investigation was carried out via multinuclear (¹H, ¹³C, and ¹¹⁹Sn) NMR, IR spectroscopy, and X-ray crystallography. The crystal structure of **4** comprises a central Sn₂O₂ core with O atoms bonded to 2 dibutylbis(3,4-dichlorophenylacetato)tin(IV) units. All the Sn atoms of **4** are essentially 5-coordinated in distorted trigonal-bipyramidal geometry. Some of the compounds exhibited significant antibacterial and antifungal activity.

Key Words: Organotin(IV), spectroscopy, distannoxane, X-ray structure, antibacterial, antifungal.

Introduction

Organotin compounds are a group of organometallics that have been intensely studied for many years due to their widespread industrial and biological applications.^{1,2} Organotin carboxylates exhibit considerable structural diversity, such as monomers, dimers, tetramers, and polymers.³ Organotin(IV) compounds are eco-toxicants

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CAD-4 diffractometer using M_oK_α radiation; LP and absorption corrections (semi-empirical from equivalents) were applied. Refinement of F^2 was accomplished by full-matrix-least squares (RAELS), using anisotropic thermal parameters for non-H atoms. H atoms were included in positions calculated for each cycle, and were assigned thermal parameters equal to their bonded atoms. A number of *n*-butyl C atoms, particularly terminal ones, were difficult to locate and refine, and were clearly affected by high thermal motion and disorder. Each of these was defined as being disordered over sites of equal occupancy using isotropic thermal parameters where appropriate. The distannoxane structure of **4** is given in Figures 2 and 3, and selected geometric data are presented in Table 2. Software used included SIR 92,¹⁰ RAELS 96,¹¹ ORTEP-3, and POV-3.6¹²

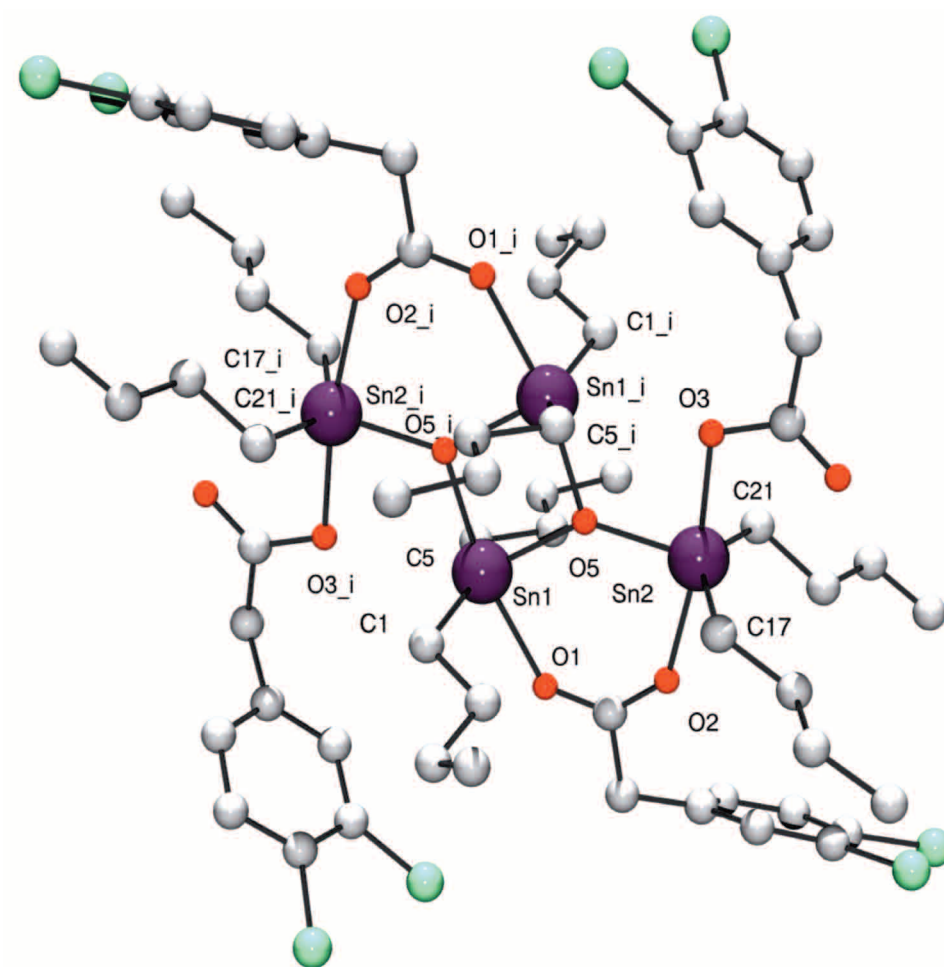


Figure 2. The ORTEP(Pluto)-POV molecular structure of **4** drawn at the 30% probability level. H atoms have been omitted. For clarity, only one component of the disordered *n*-butyl groups is shown.

Biological studies

Compounds **1-5** were bio-assayed to determine their possible antibacterial and antifungal activity via agar well diffusion and agar tube dilution methods, respectively.¹³⁻¹⁴ The antibacterial activity of these compounds

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against 4 different types of bacteria, namely *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus*, and *Micrococcus luteus*, were evaluated using roxithromycin as a reference drug. Compounds **1-5** were also screened for their antifungal activity against 3 types of fungi, *Aspergillus niger*, *Aspergillus flavus*, and *Mucor* spp.

Table 1. Crystallographic data for **4**.

Empirical formula	C ₆₄ H ₉₂ Cl ₈ O ₁₀ Sn ₄
Formula weight	1779.8
Crystal system	Monoclinic
Space group	C2/c
a(Å)	16.753(5)
b(Å)	19.625(3)
c(Å)	24.902(6)
$\beta(^{\circ})$	104.25(1)
V(Å ³)	7932(2)
Z (Calculated density) (mg m ⁻³)	4, 1.49
$\mu(M_o - K_{\alpha})$, mm ⁻¹	1.571
$\theta_{\max}(^{\circ})$	25
Reflections collected	7159
Independent reflections	6982 [$R_{int} = 0.055$]
Reflections observed ($> 2\delta$)	3559
Goodness-of-fit on F ²	1.55
Final R ₁ , wR ₂ indices [$I > 2\delta(I)$]	0.066, 0.086
$\Delta\delta_{\max}(e\text{Å}^{-3})$	1.33
$\Delta\delta_{\min}(e\text{Å}^{-3})$	-2.27

Synthesis

General procedure for the synthesis and characterization of organotin(IV) complexes (**1-5**)

3,4-Dichlorophenylacetic acid (HL) was suspended in dry toluene and treated with triethylamine in a 1:1 molar ratio at refluxing temperature for 3-4 h. To a stirred solution of triethylamine and ligand acid (HL), diorganotin(IV) dichloride or triorganotin(IV) chloride was added in appropriate mole ratios and then the reaction mixture was refluxed for 6-8 h to yield the product. The precipitate of Et₃NHCl was filtered off. The filtrate was evaporated on a rotary evaporator and the solid mass was recrystallized in a chloroform/*n*-hexane (3:1) mixture.

3,4-Dichlorophenylacetatotrimethyltin(IV) (1)

Quantities used were 1.0 g (4.4 mmol) of (HL) and 0.90 g (4.4 mmol) of trimethyltin(IV) chloride in toluene. Yield = 86%; mp = 117-118 °C. Anal. Calc. for C₁₁H₁₄Cl₂O₂Sn (MW = 367.84): C, 35.92; H, 3.84. Found: C, 35.48; H, 3.17%. ¹H-NMR (300 MHz, CDCl₃, δ , ²J [¹¹⁹Sn-¹H] in Hz); 0.36 (s, 9H, H₃C-Sn [58]), 3.37

(s, 2H, H₂C-Ar), 6.92-7.18 (m, 3H, H₃C₆Cl₂). ¹³C-NMR (75.45 MHz, CDCl₃, δ, ⁿJ [¹¹⁹Sn-¹³C] in Hz); -4.8 [394] (H₃C-Sn), 135.7, 130.8, 132.2, 131.3, 130.3, 128.8 (C₆H₃Cl₂), 40.9 (H₂C-Ar), 175.8 (COO). ¹¹⁹Sn-NMR (CDCl₃, δ); 130. IR (KBr disc, cm⁻¹); $\nu_{asym}(\text{COO})$ 1560, $\nu_{sym}(\text{COO})$ 1386, $\Delta\nu = 174$, $\nu(\text{Sn-C})$ 586, $\nu(\text{Sn-O})$ 498, $\nu(\text{Ar-Cl})$ 1128.

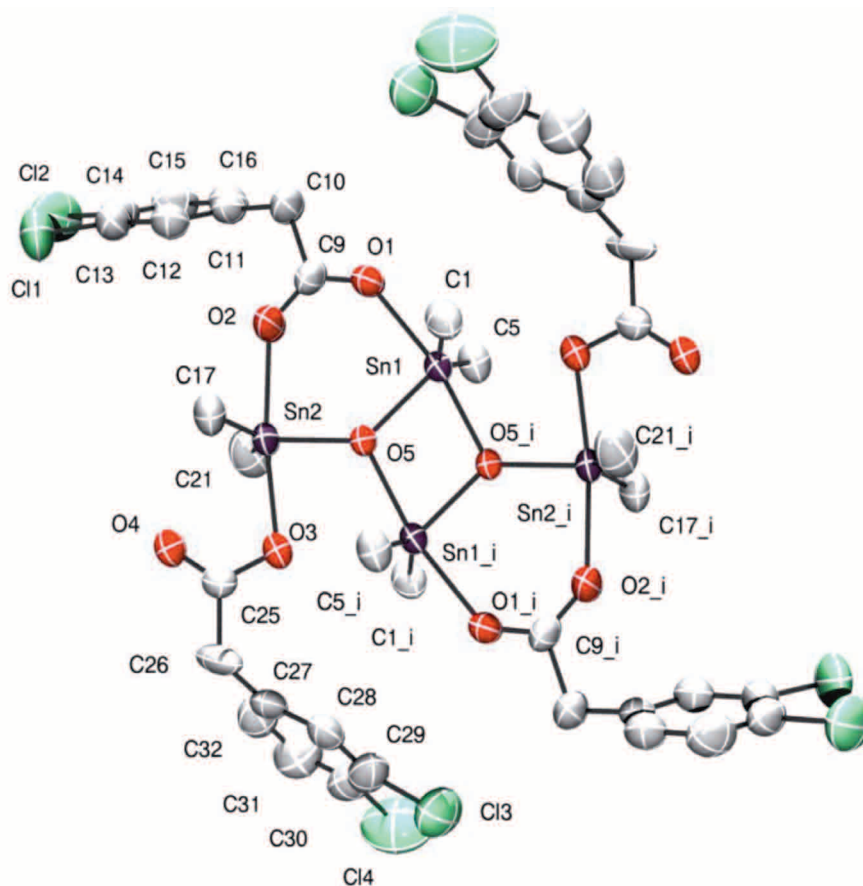


Figure 3. The ORTEP-POV molecular structure of **4** drawn at the 30% probability level. For clarity, only the connecting C atoms of the *n*-butyl groups are shown.

Bis(3,4-dichlorophenylacetato)dimethyltin(IV)(2)

Quantities used were 1.0 g (4.40 mmol) of (HL) and 0.483 g (2.20 mmol) of dimethyltin(IV) chloride in toluene. Yield = 74%; mp = 118-120 °C. Anal. Calc. for C₁₈H₁₆O₄Cl₄Sn (MW = 556.84): C, 38.83; H, 2.90. Found: C, 38.17; H, 2.82%. ¹H-NMR (300 MHz, CDCl₃, δ, ²J [¹¹⁹Sn-¹H] in Hz); 1.01 (s, 6H, H₃C-Sn [76]), 3.66 (s, 4H, H₂C-Ar), 7.15-7.42 (m, 6H, C₆H₃Cl₂). ¹³C-NMR (75.45 MHz, CDCl₃, δ, ⁿJ [¹¹⁹Sn-¹³C] in Hz); -4.7 [651] (H₃C-Sn), 134.1, 131.3, 132.6, 131.4, 130.5, 128.7 (C₆H₃Cl₂), 40.9 (H₂C-Ar), 178.4 (COO). ¹¹⁹Sn-NMR (CDCl₃, δ); -140. IR (KBr disc, cm⁻¹); $\nu_{asym}(\text{COO})$ 1558, $\nu_{sym}(\text{COO})$ 1354, $\Delta\nu = 204$, $\nu(\text{Sn-C})$ 567, $\nu(\text{Sn-O})$ 486, $\nu(\text{Ar-Cl})$ 1127.

Table 2. Selected geometric parameters (Å, °) for **4**.

Sn(1)–O(1)	2.257 (9)	Sn(2)–O(2)	2.235 (11)
Sn(1)–O(5)	2.054 (6)	Sn(2)–O(3)	2.167 (8)
Sn(1)–O(5)	2.165 (6)	Sn(2)–O(5)	2.012 (5)
Sn(1)–C(1)	2.112 (13)	Sn(2)–C(17)	2.181 (14)
Sn(1)–C(5)	2.089 (11)	Sn(2)–C(21)	2.139 (15)
O(1)–Sn(1)–C(1)	89.3 (5)	O(2)–Sn(2)–O(3)	171.6 (3)
O(1)–Sn(1)–C(5)	86.1 (5)	O(2)–Sn(2)–C(17)	85.9 (6)
O(1)–Sn(1)–O(5)	92.0 (3)	O(2)–Sn(2)–C(21)	88.8 (6)
O(1)–Sn(1)–C ⁱ (5)	168.3 (3)	O(2)–Sn(2)–O(5)	90.9 (3)
C(1)–Sn(1)–C(5)	143.8 (5)	O(3)–Sn(2)–C(17)	96.2 (5)
C(1)–Sn(1)–O(5)	107.8 (4)	O(3)–Sn(2)–C(21)	94.7 (5)
C(1)–Sn(1)–O ⁱ (5)	95.3 (4)	O(3)–Sn(2)–O(5)	80.8 (3)
C(5)–Sn(1)–O(5)	108.3 (4)	C(17)–Sn(2)–C(21)	139.9 (5)
C(5)–Sn(1)–O ⁱ (5)	96.3 (4)	C(17)–Sn(2)–O(5)	108.8 (4)
O(5)–Sn(1)–O ⁱ (5)	76.4 (2)	C(21)–Sn(2)–O(5)	110.9 (4)

⁽ⁱ⁾x,y,z**3,4-Dichlorophenylacetatotributyltin(IV) (3)**

Quantities used were 1.0 g (4.4 mmol) of (HL) and 1.20 g (4.4 mmol) of tributyltin chloride in toluene. Yield = 81%; mp = 49–51 °C. Anal. Calc. for C₂₀H₃₂O₂Cl₂Sn (MW = 494.08): C, 48.62; H, 6.53. Found: C, 48.71; H, 6.58%. ¹H-NMR (300 MHz, CDCl₃, δ, ⁿJ [¹H-¹H] in Hz); 0.91 (t, 9H, H₃C-Sn [7]), 1.29–1.57 (m, 18H, (H₂C)₃-Sn), 3.58 (s, 2H, H₂C-Ar), 7.14–7.41 (m, 3H, C₆H₃Cl₂). ¹³C-NMR (75.45 MHz, CDCl₃, δ, ⁿJ [¹¹⁹Sn-¹³C] in Hz); 27.4[350], 136.1, 132.2, 130.6, 131.3, 130.2, 128.8 (C₆H₃Cl₂), 41.3 (H₂C-Ar), 178.7 (COO). ¹¹⁹Sn-NMR (CDCl₃, δ); 116. IR (KBr disc, cm⁻¹); ν_{asym}(COO) 1575, ν_{sym}(COO) 1382, Δν = 193, ν(Sn-C) 551, ν(Sn-O) 445, ν(Ar-Cl) 1128.

Tetrabutylbis(3,4-dichlorophenylacetato)distannoxane dimer (4)

Quantities used were 2.0 g (4.4 mmol) of (HL) and 1.34 g (4.4 mmol) of dibutyltin(IV)oxide in toluene. Yield = 76%; mp = 57–58 °C. Anal. Calc. for C₆₄H₉₂O₁₀Cl₈Sn₄ (MW = 1779.87): C, 43.19; H, 5.21. Found: C, 43.21; H, 5.20%. ¹H-NMR (300 MHz, CDCl₃, δ, ⁿJ [¹H-¹H] in Hz); 0.86–0.99 (t, 6H, [7.1]), 1.31–1.85 (m, 12H, (CH₂)₃-Sn), 3.58 (s, 2H, CH₂COOH), 7.14–7.48 (m, 3H, Cl₂C₆H₃). ¹³C-NMR (75.45 MHz, CDCl₃, δ, ⁿJ [¹¹⁹Sn-¹³C] in Hz); 27.2 [696], 25.6 [586.0], 27.4 [36.5], 26.6 [31.2], 26.5 [100], 26.3 [67.0], 13.5, 13.6 (6.7) (Sn-CH₂-CH₂-CH₂-CH₃), 128.8, 130.2, 130.6, 131.3, 132.2, 136.1 (C₆H₃Cl₂), 41.8 (CH₂-Ar), 176.8 (COO). ¹¹⁹Sn-NMR (CDCl₃, δ); -206, -213. IR (KBr disc, cm⁻¹); ν_{asym}(COO) 1599, ν_{sym}(COO) 1412, Δν = 187, ν_{asym}(COO) 1724, ν_{sym}(COO) 1420, Δν = 254, ν(Sn-C) 519, 559, ν(Sn-O) 440, ν(Ar-Cl) 1130.

3,4-Dichlorophenylacetatotriphenyltin(IV)(5)

Quantities used were 1.0 g (4.40 mmol) of (HL) and 1.69 g (4.40 mmol) of triphenyltin(IV) chloride in toluene. Yield = 80%; mp = 130 °C. Anal. Calc. for C₂₆H₂₀O₂Cl₂Sn (MW = 554.05): C, 56.36; H, 3.64. Found: C, 56.58; H, 3.67%. ¹H-NMR (300 MHz, CDCl₃, δ); 7.04-7.10 (m, 15H, C₆H₅Sn), 3.30 (s, 2H, H₂C-Ar), 7.28-7.61 (m, 6H, Cl₂C₆H₃). ¹³C-NMR (75.45 MHz, CDCl₃, δ, ⁿJ [¹¹⁹Sn-¹³C] in Hz); 135.7, 132.5, 134.7, 133.5 [648.8] (C₆H₅Sn), 128.7, 142.9, 135.6, 127.8, 136.06, 132.1 (C₆H₃Cl₂), 41.8 (H₂C-Ar), 177.5 (COO). ¹¹⁹Sn-NMR (CDCl₃, δ); -112. IR (KBr disc, cm⁻¹); ν_{asym} (COO) 1555, ν_{sym} (COO) 1382, $\Delta\nu$ = 173, ν (Sn-C) 556, ν (Sn-O) 460, ν (Ar-Cl) 1128.

Results and discussion

Compounds **1-5** were synthesized by reacting the acid (HL) and the di- or tri-organotin(IV) chlorides in toluene in the presence of triethylamine. The colorless compounds thus obtained were quite stable in moist air. They were characterized by multinuclear (¹H, ¹³C, and ¹¹⁹Sn) NMR, IR spectroscopy, and single crystal X-ray crystallography, in combination with melting point and elemental analyses.

Spectroscopic data

Spectroscopic data were mentioned in the synthesis section. The ¹H-NMR spectra were recorded for the free ligand and the synthesized compounds in non-coordinating solvent (CDCl₃), and the characteristic chemical shifts were identified by their intensity and multiplicity patterns. The total number of protons, calculated from the integration curve, is in agreement with the expected molecular composition of the compounds. The methylene protons of the acetate moiety appeared at about 3.60 ppm, whereas aromatic protons of the ligand and the phenyltin moieties resonate as multiplets in the range of 7.04-7.61 ppm.¹⁵ The proton chemical shift assignment of the triorganotin moiety is straightforward from the multiplicity pattern and the ²J [¹¹⁹Sn-¹H] coupling constants that conform to the tetrahedral environment around the Sn atom. The dimethyltin derivative coupling constant, ²J [¹¹⁹Sn-¹H], was calculated to be about 76.5 Hz;¹⁶ however, the coupling constant, ⁿJ [¹¹⁹Sn-¹H], for *n*-butyl and phenyltin derivatives could not be calculated due to the complexity of the multiplet pattern.

¹³C-NMR data (in CDCl₃) explicitly resolved the resonances of all the distinct carbon atoms present in the compounds. The aromatic carbon resonances of the triphenyltin moieties are easily assigned on the basis of both the aromatic ⁿJ [¹³C-¹¹⁹Sn] coupling constant and signal intensities. It has already been reported that the ¹J [¹³C-¹¹⁹Sn] coupling constant can be used to assess the coordination number of the tin atom in organotin compounds.^{17,18} The coupling constants were determined to be in the order of 392.8 Hz for trimethyltin, 344 Hz for tributyltin, and 646 Hz for triphenyltin compounds, which are the characteristic values for tetrahedral compounds; that is, the solid state polymeric structure of each of these compounds breaks down into its monomeric 4-coordinated tetrahedral structure in solution. Another characteristic feature of the triphenyltin derivatives is the observance of a ¹³C chemical shift of the *ipso*-carbon at about 138 ppm, which is attributed to a tetrahedral tin atom.¹⁷ The coupling constant values for diorganotin compounds described a distorted octahedral environment around the Sn atom.¹⁹ The chemical shift of the carboxylic carbon in these organotins

is observed in the range of 175-177 ppm, in comparison to 180.7 ppm in the ligand, describing coordination of the ligand through carboxylic oxygen to the organotin(IV) moiety.

^{119}Sn -NMR data (in CDCl_3) show a single resonance at 130 ppm for the trimethyltin compound (**1**), 116 ppm for the tributyltin compound (**3**), and -112 ppm for the triphenyltin compound (**5**). These values are in conformity to 4 coordination around the Sn atom, as reported earlier.^{17,19} Diorganotin(IV) compounds also exhibit a single tin chemical shift around -140 ppm for **2**, which is the characteristic value for a skew trapezoidal bipyramidal geometry around the Sn atom.

^1H - and ^{13}C -NMR spectral data for compound **4** exhibited 2 sets of signals due to 2 different environments around the alkyl groups attached to the endo- and exocyclic Sn atoms. Compound **4** also shows two ^{119}Sn NMR signals at -206 and -213 ppm due to the 2 non-equivalent tin sites in the molecule.¹⁹

The characteristic IR bands of compounds **1-5** were identified by comparing our data with previously reported values.^{15,16} The vibrational absorption of interest includes $\nu(\text{Sn-C})$, 550-586 cm^{-1} ; $\nu(\text{Sn-O})$, 440-486 cm^{-1} ; $\nu(\text{COO})_{\text{asym}}$, 1549-1599 cm^{-1} ; and $\nu(\text{COO})_{\text{sym}}$, 1372-1394 cm^{-1} . The mode of coordination of the carboxylate ligand to tin(IV) was attributed to the parameter, $\Delta\nu[\nu_{\text{asym}}(\text{COO}) - \nu_{\text{sym}}(\text{COO})]$. The magnitude of $\Delta\nu$ values for **1**, **3**, and **5** lie in the range of 173-193, which describes the monodentate nature of the carboxylate group in these compounds, whereas for compound **2** the value 204 is indicative of the bidentate nature of the carboxylate group in solid state. Data for distannoxane **4** explicitly show 2 tin atoms with different modes of coordination. The presence of an absorption band around 450 cm^{-1} clearly demonstrates the existence of a Sn-O-C unit in these compounds.

X-ray diffraction analysis

The crystal structure of **1** has previously been reported²¹ and describes the polymeric chains involving both O atoms of the carboxylate group in the solid state. The X-ray structure of **4** is delineated in Figures 2 and 3, and selected bond lengths and bond angles are given in Table 2. The structure is composed of a centrosymmetric dimer of an oxoditin(IV) complex comprising 2 dibutylbis[3,4-dichlorophenylacetato]tin(IV) units with monodentate and bridging bidentate carboxylate ligands. The central Sn_2O_2 core is fused with 2 six-membered rings Sn(1)/O(1)/C(9)/O(2)/Sn(2)/O(5) incorporating O and C atoms of the ligand that show different modes of coordination with Sn. The endocyclic Sn atoms of the Sn_2O_2 core are 5-coordinated in a C_2SnO_3 distorted trigonal-bipyramidal environment in which Sn is coordinated to 2 α -C atoms of the *n*-butyl groups at axial positions, and 2 bridging O-atoms and 1 O-atom of the ligand form the basal plane. The endocyclic Sn-O distances in the Sn_2O_2 core are 2.165(6) and 2.054(6) Å, and in exocyclic 6-membered rings Sn(1)/O(1)/C(9)/O(2)/Sn(2)/O(5) they are 2.235(11) and 2.257(9) Å, which is in accordance with the reported structures.^{22,23} Thus, both the Sn atoms assume distorted trigonal-bipyramidal geometry with Sn-C distances lying in the range of 2.089(11)-2.181(14) Å, while Sn-O distances are in the range of 2.054(6)-2.257(9) Å.

Biological studies

Antibacterial activity

Compounds **1-5** were tested against 4 bacterial strains using the agar well diffusion method and the results are presented in Table 3. Compound **4** exhibited highly significant activity against all the tested bacteria, while

compounds **3** and **5** exhibited good activity against both *Escherichia coli* and *Micrococcus luteus*; however, compound **2** exhibited good activity only against *Micrococcus luteus*. These results indicate that compound **4** could be a potent antibacterial agent.

Table 3. Antibacterial activity data^{a,b} for compounds **1-5**.

Name of Bacteria	Zone of Inhibition (mm)						Standard Drug*
	Ligand (HL)	1	2	3	4	5	
<i>Escherichia coli</i>	8	10	10	14	26	18	28
<i>Bacillus subtilis</i>	13	13	13	16	22	14	38
<i>Staphylococcus aureus</i>	10	12	11	10	24	12	38
<i>Micrococcus luteus</i>	12	12	18	16	26	16	28

*Standard drug = roxithromycin

^a(In vitro) concentration = 100 μ g mL⁻¹.

^bSize of well = 4 mm (diameter).

Antifungal activity

The agar tube dilution method was employed to test the antifungal activity of the synthesized compounds (Table 4). The overall results show that these compounds exhibited moderate activity. Compound **3** was active against *Aspergillus niger* and compounds **2** and **4** were active against *Aspergillus flavus*; however, these compounds were the least active against the *Mucor* spp.

Table 4. Antifungal activity data^{a,b,c} for compounds **1-5**.

Name of fungus	Percentage Inhibition					
	Ligand (HL)	1	2	3	4	5
<i>Aspergillus niger</i>	12	14	9	49	26	17
<i>Aspergillus flavus</i>	15	19	45	18	45	34
<i>Mucor</i> spp.	2	0	26	6	2	28

*Standard drug = miconazole (100% inhibition).

^aIncubation period = 5-7 days.

^bIncubation temperature = 25 °C.

^cConcentration = 100 μ g mL⁻¹ of medium.

Supplementary data

Crystallographic data (excluding structure factors) for the structures reported in this paper were deposited at the Cambridge Crystallographic Data Centre, as supplementary publication no. CCDC-610168 for **4**. Copies of the available materials can be obtained, free of charge, upon application to the director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-(0)-1223-336033, or e-mail: deposit@ccdc.cam.ac.uk).

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