Research Article

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Synthesis, structure, and cytotoxicity of some triorganotin(IV) complexes of 3-aminobenzoic acid-based Schiff bases

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Abstract: Six new triorganotin(IV) complexes of 3-aminobenzoic acid-based Schiff bases, 3-(R′-CH=N)C_6H_4COOSnR_3 (**1–6**) (R′, R = 5-Br-2-HOC_6H_3, Ph (**1**); 3,5-Br_2-2-HOC_6H_2, Ph (**2**); 4-NEt_2-2-HOC_6H_3, Cy (**3**); 3-OCH_3-2-HOC_6H_3, Cy (**4**); 2-HO$C_{10}H_6$, Ph (**5**); 2-HO$C_{10}H_6$, Cy (**6**)), have been synthesized by the one-pot reaction of equimolar 3-aminobenzoic acid, substituted 2-hydroxybenzaldehyde (or 2-hydroxy-1-naphthaldehyde) and triorganotin(IV) hydroxide, and characterized by elemental analysis, FT-IR, NMR spectroscopy, and X-ray single crystal diffraction. The NMR data ($^1J(^{119}Sn-^{13}C)$ and ^{119}Sn chemical shifts) suggested that these organotin(IV) complexes are all four-coordinated in $CDCl_3$ solution. In the crystalline state, the tin atoms in **1–4** and **6** are four-coordinated and possess a distorted tetrahedral geometry. Complex **5** with crystalline solvents (CH_3OH and $CHCl_3$) exhibits a zigzag chain, and the five coordination atoms on the tin atom are arranged in a trigonal bipyramidal geometry in which the carboxylate oxygen atom and the phenolic oxygen atom of the adjacent ligand occupy the axial positions. In all complexes, the 3-(arylmethyleneamino)benzoate ligands are coordinated with tin atoms in monodentate mode. Their cytotoxicity against two human cancer cell lines (A549 and HeLa), UV-Vis, and fluorescence have been determined, and the results reveal that complexes **1**–**6** have higher cytotoxicity than cisplatin and may be explored for potential blue luminescent materials.

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1 Introduction

Organotin(IV) compounds show a diverse range of applications, and are broadly used in the agriculture and industrial production (Davies et al., 2008). In recent years, there has been an increasing interest in organotin(IV) compounds in medicinal chemistry, and the literature shows that the bioactivity potency of organotin (IV) compounds increase in the following order: R_4Sn < $RSnL_3$ < R_2SnL_2 < R_3SnL (L = ligand) (Bantia et al., 2019; Pellerito and Nagy, 2002). Triorganotin(IV) carboxylates ($R_3SnOOCR'$) possess rich structural diversity including discrete, dimeric, chain, and macrocyclic structures and shows good application prospects as insecticides and fungicides, and, particularly, in cancer therapy (Bantia et al., 2019; Basu Baul et al., 2010; Davies et al., 2008; Hadi et al., 2021; Liang et al., 2014; Tiekink, 1991, 1994). The R groups on tin atom and carboxylic acid ligand have important effects on the biological activity of triorganotin (IV) carboxylates (Bantia et al., 2019; Basu Baul et al., 2017a). Therefore, to synthesize organotin(IV) complexes with different carboxylic acid ligands is an alternative good strategy to improve their biological activities. Schiff bases (C=N) prepared from the condensation of aldehyde or ketone and primary amine have some characteristic properties such as great synthesis flexibility and biological properties (Kaur et al., 2021; Nath and Saini, 2011). Introducing Schiff base functional groups into triorganotin(IV) carboxylates is expected to achieve the synergistic effect of their biological activities and obtain organotin(IV) complexes with novel structure and good activity. Some triorganotin(IV) complexes with the Schiff base carboxylate ligands have been synthesized and characterized. Basu Baul and co-workers synthesized a series of triorganotin (IV) complexes of amino acid Schiff bases, and found that

these complexes have good biological activities, especially anticancer activity (Basu Baul et al., 2007, 2009, 2010, 2017a, 2017b). In addition, they also synthesized a novel series of triphenyltin(IV) 2- or 4-((arylimino)methyl)benzoates, 2/4-(ArN=CH)$C_6H_4COOSnPh_3$, by the reactions between triphenyltin(IV) 2/4-formylbenzoate and aromatic amines, which revealed high cytotoxic activities against some human cancer cells (Basu Baul et al., 2018). Other groups (Dias et al., 2015; Goh et al., 1998; Saeed et al., 2017; Tzimopoulos et al., 2010; Yin et al., 2005, 2012; Yu et al., 2022) had also done good work on the triorganotin(IV) complexes. Recently, our team has synthesized the triorganotin(IV) complexes of *N*-(5-bromosalicylidene)-α-amino acid, 5-(salicylideneamino)salicylic acid, and 3-(salicylideneamino)benzoic acid, which exhibited strong cytotoxic efficacy (Chen et al., 2020; Liu et al., 2019; Yao et al., 2017). Organotin(IV) complexes of the carboxylate ligands containing Schiff base are generally synthesized by three different routes: (i) the reaction of organotin(IV) oxide or hydroxide or chloride with the Schiff base carboxylic acid ligand prepared in advance from amino acid and aldehyde (Basu Baul et al., 2017a, 2017b; Dias et al., 2015; Saeed et al., 2017; Tzimopoulos et al., 2010; Yin et al., 2005, 2012; Yu et al., 2022), (ii) the reaction of aldehyde with organotin(IV) aminocarboxylate first prepared from amino acid and organotin(IV) oxides or hydroxides or chlorides (Basu Baul et al., 2018; Tzimopoulos et al., 2010), and (iii) the one-pot reaction of organotin(IV) oxide or hydroxide, amino acid and aldehyde (Beltran et al., 2003; Chen et al., 2020; Yao et al., 2017). As a continuation of our previous works based on organotin(IV) complexes with Schiff base ligands (Chen et al., 2020; Liu et al., 2019; Yao et al., 2017), herein we report one-step preparation, and crystal structure and property of new triphenyltin(IV) and tricyclohexyltin(IV) 3-(arylmethyleneamino)benzoates (**1–6**), 3-(R′-CH=N)$C_6H_4COOSnR_3$ (**1–6**) (R′, R = 5-Br-2-HOC_6H_3, Ph (**1**); 3,5-Br_2-2-HOC_6H_2, Ph (**2**); 4-NEt_2-2-HOC_6H_3, Cy (**3**); 3-OCH_3-2-HOC_6H_3, Cy (**4**); 2-$HOC_{10}H_6$, Ph (**5**); 2-$HOC_{10}H_6$, Cy (**6**)) (Scheme 1).

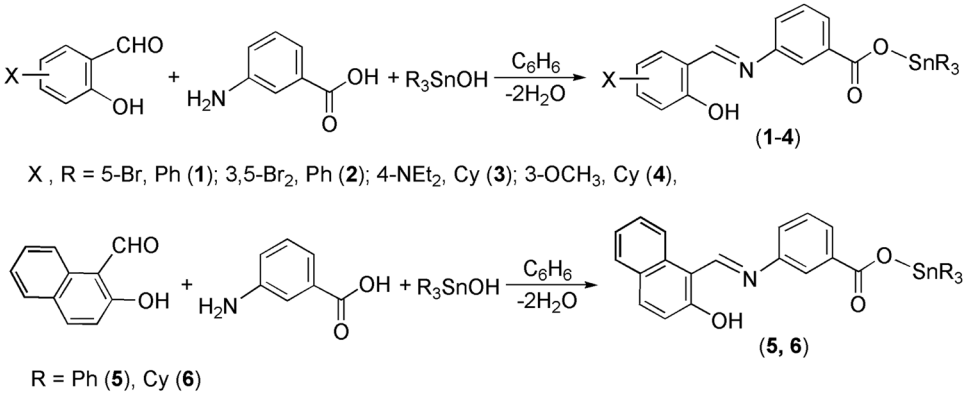
2 Results and discussion

2.1 Synthesis

Equimolar 3-aminobenzoic acid, substituted 2-hydroxybenzaldehyde (or 2-hydroxy-1-naphthaldehyde), and triorganotin(IV) hydroxide are refluxed in benzene for 6 h by azeotropical removal of water to give the products (**1–6**) 76% to 88% yield (Scheme 1). In this preparation, the Schiff base was formed *in situ*, and it is simpler than two-step method mentioned above. The complexes are yellow to orange red crystals that are stable in air and soluble in common organic solvents.

2.2 Spectroscopic characterization

In the complexes, IR spectra show a broad band at ~3,430 cm^{-1} assigned to ν(OH) of phenol hydroxyl with intramolecular O–H···N hydrogen bond. The strong bands attributable to the asymmetric ν_{as}(COO) and symmetric ν_s(COO) of carboxylate occur at 1,618–1,637 cm^{-1} and 1,324–1,343 cm^{-1}, respectively. The ν(C=N) absorption peak of Schiff base unit appears at about 1,615 cm^{-1}, which overlaps with the ν_s(COO) absorption in some cases (Vinayak and Nayek, 2019) (Supplementary material). The difference ($\Delta\nu$(COO)) between the ν_{as}(COO) and ν_s(COO) bands can provide useful information concerning the coordination behavior of the carboxylate ligand (Deacon and Phillips, 1980; Yin et al., 2005).



Scheme 1: Synthesis of the complexes.

Generally, the magnitude of $\Delta\nu$(COO) is above 200 cm^{-1} for a monodentate coordination and below 200 cm^{-1} for a bidentate coordination. The values (270–314 cm^{-1}) of $\Delta\nu$(COO) in complexes **1**–**6** indicate that the carboxylate ligand is bonded to the tin center in a monodentate mode.

The ^{1}H and ^{13}C chemical shift assignments of complexes **1**–**6** are from the multiplicity patterns and resonance intensities, as well as related literature (Chen et al., 2020; Tzimopoulos et al., 2010; Yin et al., 2012; Yu et al., 2022). The ^{1}H NMR integration values were consistent with the structures of **1**–**6** in Scheme 1. In the complexes, the phenolic O–H proton is observed in the range of 13.07–15.43 ppm as a broad signal due to the formation of intramolecular O–H···N hydrogen bond. The CH═O proton signal (~10 ppm) in the substituted 2-hydroxybenzaldehyde or 2-hydroxy-1-naphthaldehyde does not appear, and the imine CH═N proton signal is observed in the region of 8.50–9.43 ppm, indicating the formation of Schiff base ligand. The chemical shift value of CH═N proton in **1**–**4** (~8.6 ppm) is obviously smaller than that in **5** and **6** (~9.4 ppm) due to the different effects of benzene ring and naphthalene ring on CH═N proton. The COOH proton resonance signal of Schiff base carboxylic ligand is not observed, which confirms the deprotonation of carboxylic ligand and bonding to the tin atom.

In the ^{13}C NMR spectra, the resonances of carboxylate (COO) carbon appear in the range of 170.52–172.05 ppm. The chemical shifts of Schiff base imine carbon (CH═N) are observed in the range of 160.95–163.30 ppm for complexes **1**–**4** and 169.82–170.15 ppm for complexes **5** and **6**. The other carbon atoms of the ligand and SnR_3 skeletons display the expected resonance signals. The coupling constant, $^{1}J(^{13}C–^{119}Sn)$, is closely related to the coordination number of tin atom, and can provide structural information of organotin(IV) compounds in solution (Holecek et al., 1983, 1986). For four-coordinated triphenyltin(IV) and tricyclohexyltin(IV) compounds, $^{1}J(^{119}Sn–^{13}C)$ values lie in the range of 550–650 and 295–360 Hz, respectively (Holecek et al., 1983; Zhang et al., 1990). The $^{1}J(^{119}Sn–^{13}C)$ value of **1**–**6** is 640, 642, 336, 338, 648, and 336 Hz, respectively, clearly indicating that the tin atoms in these complexes are all four-coordinated in non-coordination solvent $CDCl_3$. This conclusion is further supported by the ^{119}Sn NMR chemical shifts of the complexes. Triphenyltin (IV) complexes **1**, **2**, and **5** exhibit a single sharp resonance at –102.7, –102.4, and –105.4 ppm, and tricyclohexyltin (IV) complexes **3**, **4,** and **6** display absorption peaks at 18.6, 19.4, and 17.3 ppm, suggesting that the central tin atom has a four-coordinate environment (Holecek et al., 1983; Zhang et al., 1990).

2.3 Crystal structures of the complexes

The molecular structures of these complexes are shown in Figures 1–6, respectively. The selected bond lengths and bond angles are listed in Table 1. The coordination environment of the tin atom in the triphenyltin(IV) complexes **1** and **2** is a distorted tetrahedron and four coordination atoms are from three carbon atoms of phenyl groups and one carboxyl oxygen atom of the (*E*)-3-[(2-hydroxybenzylidene)amino]benzoate ligand (Figures 1 and 2). The Sn–C bond lengths and the C–Sn–C bond angles around the tin atom are in the ranges of 2.117(4)–2.143(3) Å and 107.09(12)–119.13(12)°, respectively, which are similar to those observed in the analogous triphenyltin(IV) derivatives, such as 4-(2-HOC_6H_4CH═N)$C_6H_4COOSnPh_3$ (Yin et al., 2005), 4-$H_2NC_6H_4COOSnPh_3$ (Tzimopoulos et al., 2009), and 3-(4-$Me_2NC_6H_4CH$═N)$C_6H_4COOSnPh_3$ (Basu Baul et al., 2017a). The monodentate nature of the carboxylate ligand is further supported by the long separation of

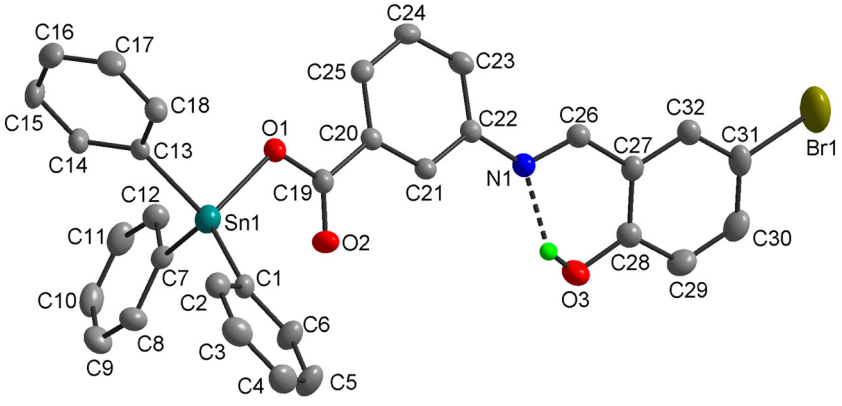


Figure 1: The molecular structure of **1.** Ellipsoids are drawn at 30% probability level. Hydrogen atoms are omitted for clarity except H_3 atom.

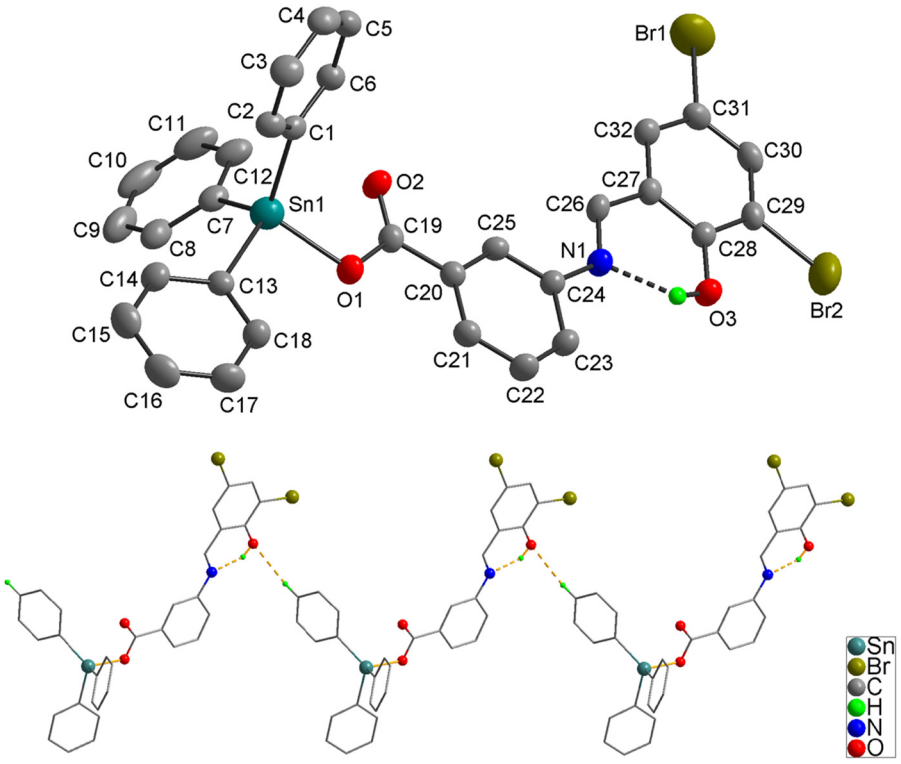


Figure 2: (Top) The molecular structure of **2.** Ellipsoids are drawn at 30% probability level. Hydrogen atoms are omitted for clarity except H_3 atoms. (Bottom) One-dimensional supramolecular chain formed by the intermolecular C–H···O hydrogen bonds.

Sn(1)···O(2) (2.758(2) Å for **1** and 2.785(3) Å for **2** and short C(19)–O(2) bond length (1.222(4) Å for **1** and 1.217(5) Å for **2**). However, the Sn(1)···O(2) interaction leads to the distortion from a regular tetrahedron, which expands the C(1)–Sn(1)–C(7) bond angle (119.13(12)° for **1** and 117.52(15)° for **2**) and reduces the O(1)–Sn(1)–C(13) bond angle (96.51(10)° for **1** and 94.77(14)° for **2**).

The other triphenyltin(IV) complex **5** with crystalline solvents (CH_3OH and $CHCl_3$) is a coordination polymer, and displays a one-dimensional zigzag chain with the Sn···Sn distances of 12.269(3) and 12.419(3) Å (Figure 3). The asymmetric unit contains two molecules of complex **5** with similar geometric parameters. In the chain, the (*E*)-3-[(2-hydroxy-1-naphthalenyl)methyleneamino]benzoate ligand bridges two tin atoms by a carboxylate oxygen atom and a phenolic oxygen atom, and each tin atom has a distorted trigonal bipyramidal geometry. Three phenyl groups are in the equatorial positions with the three C–Sn–C bond angles of 118.85(11)–120.00(11)° for Sn(1) and 116.73(11)–122.27(12)° for Sn(2), and the axial positions are occupied by a carboxylate oxygen and the phenolic oxygen of an adjacent ligand with the O–Sn–O bond angle of 174.18(7)° for Sn(1) and 175.07(7)° for Sn(2). The Sn–O (carboxylate) bonds (Sn(1)–O(1) 2.175(2) Å and Sn(2)–O(6) 2.167(2) Å) are shorter than the Sn–O(phenol) bonds (Sn(1)–O(1) 2.378(2) Å and Sn(2)–O(4) 2.391(2) Å), which are consistent with that observed in analogue triphenyltin (IV) 4-[(2-hydroxy-1-naphthyl)methyleneamino]benzoate, 4-(2-$HOC_{10}H_6CH{=}N)C_6H_4COOSnPh_3$ (Yin et al., 2012). The long distance between the carbonyl oxygen of carboxylate and tin atom (Sn(1)···O(2) 3.015(3) Å) indicates that the carbonyl oxygen does not coordinate to tin atom and the carboxylate is monodentate.

The three tricyclohexyltin(IV) complexes **3**, **4**, and **6** crystallize in $P2_1/n$, $C2/c$, and P-1 space groups, respectively, and the coordination environment of tin atom is a distorted tetrahedral geometry in which the four vertices are occupied by three carbon atoms of the cyclohexyl groups and the oxygen atom of the aminobenzoate (Figures 3, 4, and 6). The C–Sn–C bond angles around tin atoms are in the range of 111.30(13)–124.89(9)°, and the cyclohexyl groups bound to the tin atom adopt chair conformations with the Sn–C bond lengths of 2.149(4)–2.176(3) Å. The structural parameters are similar to those found in other reported tricyclohexyltin(IV) substituted-benzoates, such as 3-(2-$HOC_6H_4CH{=}N)C_6H_4COOSnCy_3$ (Chen et al., 2020), 5-(2-$HOC_6H_4CH{=}N$)-2-$HOC_6H_3COOSnCy_3$ (Liu et al., 2019), and 2-(5-Me-2-$HOC_6H_3N{=}N)C_6H_4COOSnCy_3$ (Willem et al., 1998).

In these complexes, the C=N bond lengths (C(26)–N(1)) of the Schiff base carboxylate ligands are in the range of

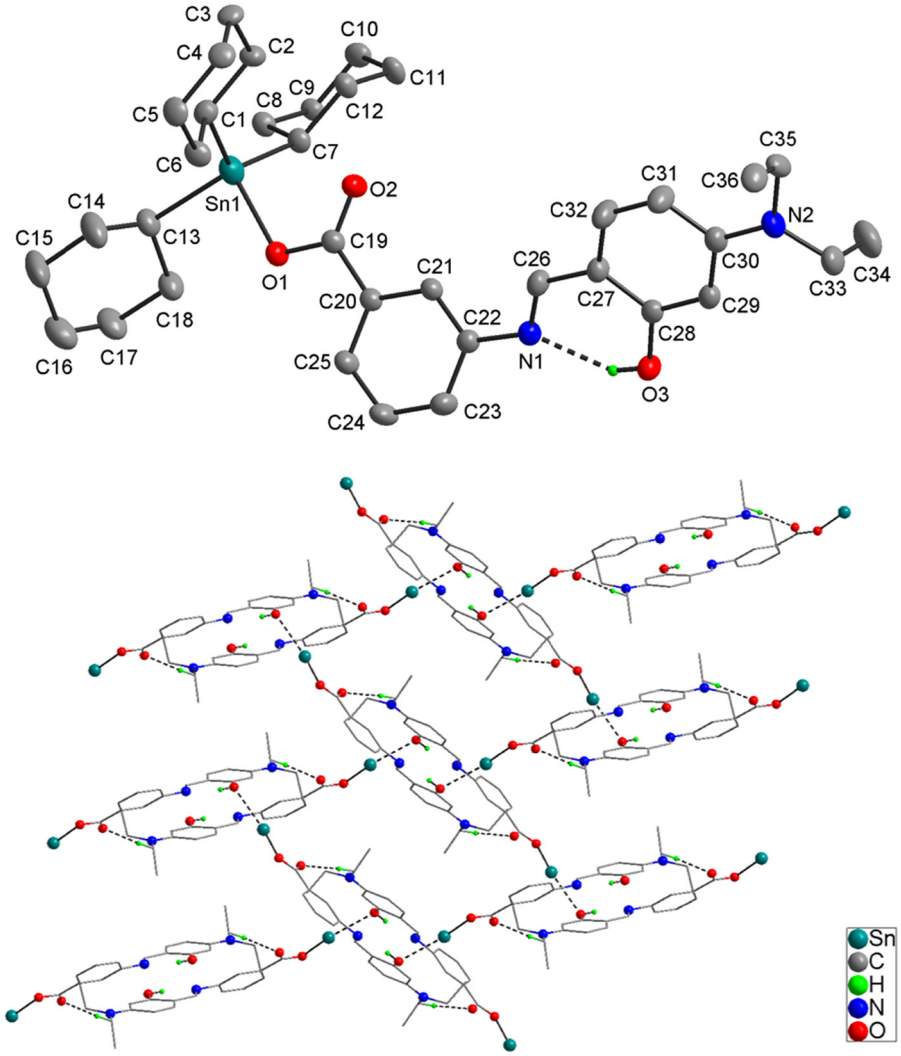


Figure 3: (Top) The molecular structure of **3**. Ellipsoids are drawn at 30% probability level. Hydrogen atoms are omitted for clarity except H_3 atoms. (Bottom) Two-dimensional supramolecular network formed by the intermolecular Sn···O interactions and C–H···O hydrogen bonds. The Cy groups bonded to Sn are omitted for clarity.

1.272(5)–1.314(3) Å, and all imine C=N bonds exhibit *E*-type configuration. Except for complex **2**, the two aromatic rings at both ends of the C=N double bond in **1** and **3–6** are not in the same plane, and the dihedral angles between the two aryl rings are 23.58(9)°, 35.43(6)°, 51.11(3)°, 14.81(5)°, and 29.29(5)°, respectively. In **1–4** and **6**, there is the intramolecular O–H···N hydrogen bond involving hydroxyl O(3)–H(3) and imino N(1) atom, while in **5**, this proton transfers from hydroxyl oxygen O(3) to nitrogen N(1) to form the intramolecular N–H···O hydrogen bond (Table 2).

The crystal structure is stabilized by weak intermolecular interactions. In **2** and **4**, the molecules are connected to one-dimensional supramolecular chain with Sn···Sn distance of 13.74(3) and 12.47(2) Å, respectively, by the intermolecular C–H···O hydrogen bonds (Figures 2 and 4, Table 2). In the crystal of **3**, a pair of intermolecular C(35)–H(35)···O(2) hydrogen bonds connect two molecules into a centrosymmetric $R_2^2(28)$ macrocycle, and the adjacent $R_2^2(28)$ macrocycles are further linked by a pair of Sn(1)···O(3) (3.417(3) Å) interactions to form two-dimensional supramolecular network (Figure 4, Table 2).

2.4 UV-Vis absorption and fluorescence of the complexes

UV-Vis absorption and emission spectra of complexes **1–6** were recorded in dichloromethane solution (3.0×10^{-5} M) at

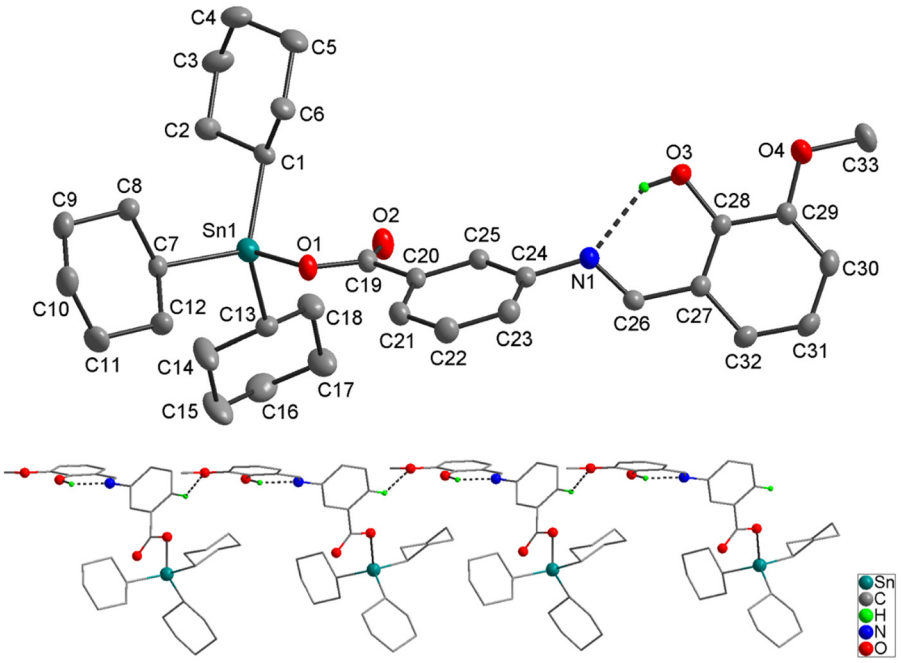


Figure 4: (Top) The molecular structure of **4.** Ellipsoids are drawn at 30% probability level. Hydrogen atoms are omitted for clarity except H_3 atoms. (Bottom) One-dimensional supramolecular chain formed by the intermolecular C–H···O hydrogen bonds.

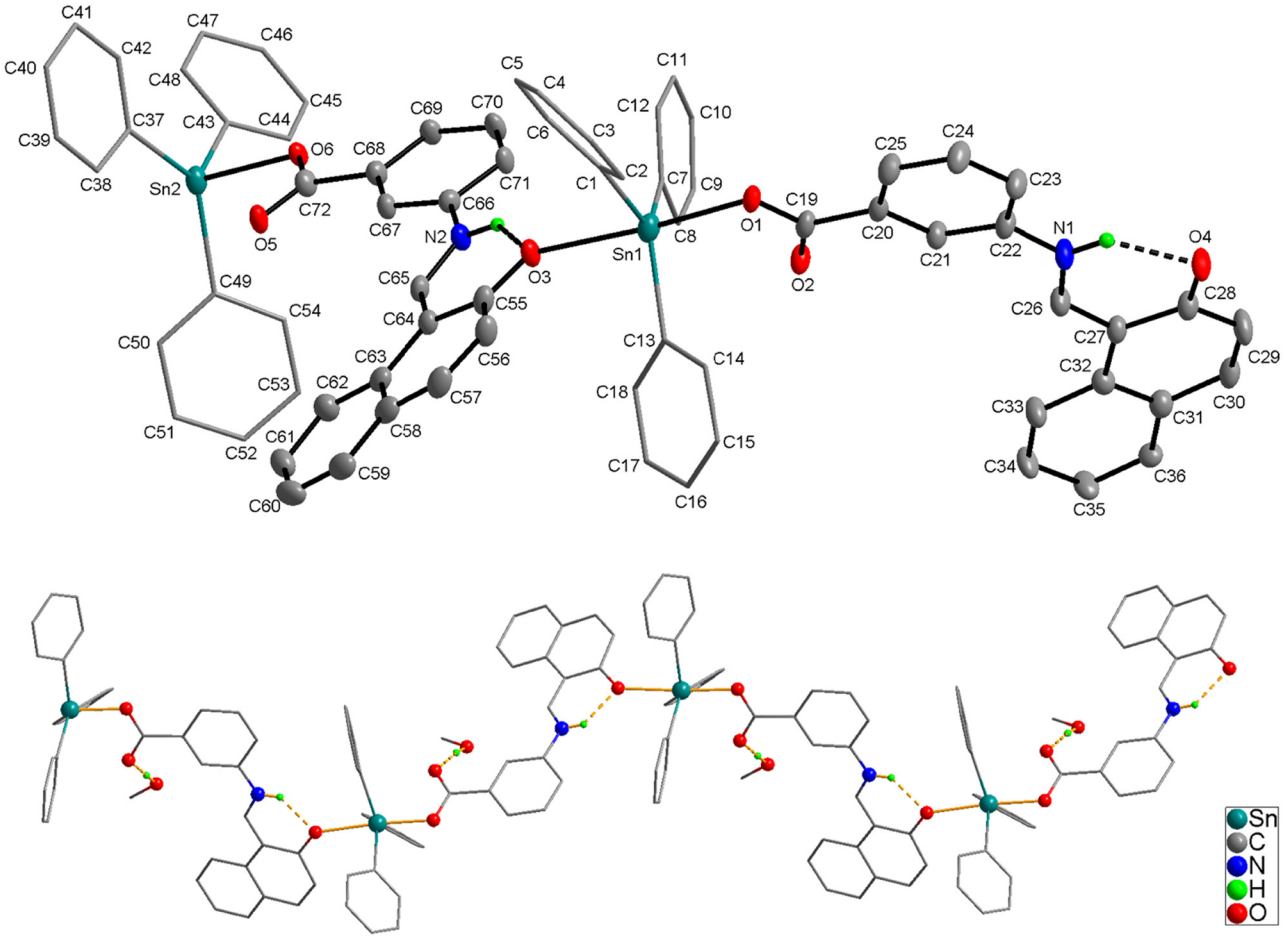


Figure 5: (Top) The molecular structure of **5.** Ellipsoids are drawn at 30% probability level. For clarity, hydrogen atoms except H1 and H2 atoms and solvent molecules are omitted. (Bottom) One-dimensional chain formed by the intermolecular phenolic O→Sn coordination. The solvent molecule $CHCl_3$ is omitted for clarity.

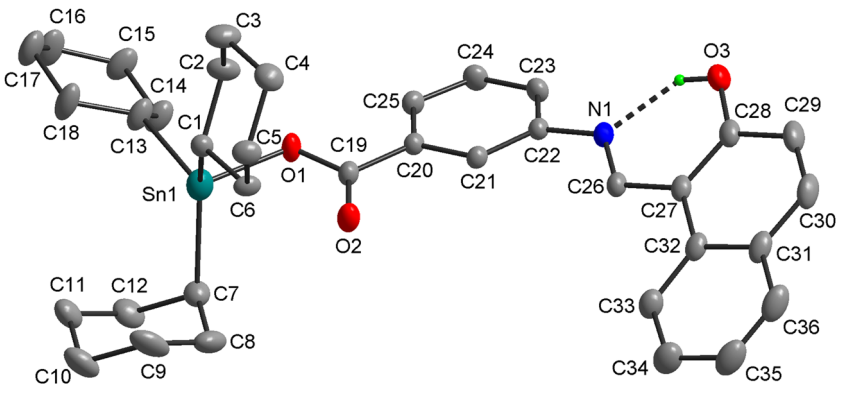


Figure 6: The molecular structure of **6.** Ellipsoids are drawn at 30% probability level. Hydrogen atoms are omitted for clarity except H_3 atoms.

room temperature (Figures 7 and 8, Table 3). In the complexes, the absorption bands at ~235 nm are assigned to the π→π* electronic transitions of the aromatic rings, and the bands at 270–459 nm are assigned to the π→π* and n→π* transitions of the conjugated imine (C=N) in the ligand moiety (Gonzalez-Hernandez et al., 2021). The naphthyls (**5** and **6**) and substituents bonded to the benzene ring (**1**–**4**) affected the position of maximum absorption wavelength by the electronic delocalization. The complexes displayed similar emission at ~440 nm (438 nm for **1**, **2**, and **4**–**6**, 444 nm for **3**) when excited at 310 nm (Figure 8a). When excited at their absorption maxima (Figure 8b), their maximum emission wavelengths did not change essentially, but their fluorescence intensity increased. The complexes may be explored for potential blue luminescent materials.

2.5 *In vitro* cytotoxicity of the complexes

The cytotoxic activity of complexes **1**–**6** against two human cancer cells A549 and HeLa were evaluated using the MTT assay, and the results are listed in Table 4 (a dose-dependent antiproliferative effect was shown in ESI). The IC_{50} values of exposure to the complexes after 24 h showed that the activity of the complexes against A549 and HeLa cells was higher than that of the reference drug cisplatin, indicating that the compounds are all potent anti-cancer agents. Their cytotoxic activities were similar to those of the reported triorganotin(IV) complexes of aminobenzoic acid-based Schiff bases, such as triphenyltin [4-(dimethylamino)phenylmethyleneamino]benzoates ($(4\text{-}Me_2NC_6H_4CH{=}N)C_6H_4COOSnPh_3$ (IC_{50} = 0.88–6.18 μM against HeLa) (Basu Baul et al., 2017a) and triorganotin

Table 1: Selected bond lengths (Å) and angles (°) for the complexes

	1	**2**	**3**	**4**	**$[5{\cdot}CH_3OH{\cdot}CHCl_3]_2$**	**6**
Sn(1)–C(1)	2.127(3)	2.132(4)	2.155(3)	2.149(4)	2.131(3)	2.162(2)
Sn(1)–C(7)	2.125(3)	2.117(4)	2.165(3)	2.157(4)	2.128(2)	2.152(2)
Sn(1)–C(13)	2.143(3)	2.132(4)	2.176(3)	2.158(4)	2.127(3)	2.158(2)
Sn(1)–O(1)	2.064(2)	2.059(3)	2.095(2)	2.081(3)	2.175(2)	2.0712(15)
Sn(1)–O(3)	—	—	—	—	2.378(2)	—
C(19)–O(1)	1.307(4)	1.312(5)	1.298(3)	1.310(4)	1.284(3)	1.302(2)
C(19)–O(2)	1.222(4)	1.217(5)	1.224(3)	1.210(5)	1.229(3)	1.217(3)
O(1)–Sn(1)–C(1)	115.27(11)	108.62(13)	105.48(10)	108.52(13)	87.41(8)	104.67(7)
O(1)–Sn(1)–C(7)	104.90(11)	111.99(14)	102.22(10)	105.55(13)	99.00(8)	104.45(9)
O(1)–Sn(1)–C(13)	96.51(10)	94.77(14)	94.92(11)	98.21(12)	94.72(9)	90.00(9)
C(1)–Sn(1)–C(7)	119.13(12)	117.52(15)	124.60(12)	115.89(14)	119.91(11)	124.89(9)
C(1)–Sn(1)–C(13)	111.32(12)	109.07(15)	112.91(12)	116.64(15)	120.00(11)	109.44(3)
C(7)–Sn(1)–C(13)	107.09(12)	112.49(17)	111.30(13)	109.87(14)	118.85(11)	116.26(11)
O(1)–Sn(1)–O(3)	—	—	—	—	174.18(7)	—

Table 2: H-bonding geometry parameters (Å, °) for the complexes

Complex	D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)	Code[#]
1	O(3)–H(3)···N(1)	0.85	1.89	2.650(4)	148	
2	O(3)–H(3)···N(1)	0.85	1.77	2.552(5)	151	
	C(10)–H(10)···O(3)[#]	0.93	2.58	3.455(7)	157	$x, 1+y, z$
3	O(3)–H(3)···N(1)	0.85	1.85	2.626(3)	151	
	C(35)–H(35)···O(2)[#]	0.97	2.54	3.508(5)	173	$-x, 1-y, 1-z$
4	O(3)–H(3)···N(1)	0.85	1.90	2.645(5)	146	
	C(21)–H(21)···O(4)[#]	0.93	2.49	3.246(6)	138	$x, 2-y, 1/2+z$
[**5**·CH_3OH·$CHCl_3$]$_2$	N(1)–H(1)···O(4)	0.86	1.91	2.597(3)	136	
	N(2)–H(2)···O(3)	0.86	1.91	2.583(3)	135	
	O(7)–H(7)···O(5)	0.85	1.97	2.805(3)	168	
	O(8)–H(8)···O(2)	0.85	1.91	2.751(3)	173	
6	O(3)–H(3)···N(1)	0.85	1.80	2.549(2)	146	

5-(salicylideneamino)salicylate, 5-(2-$HOC_6H_4CH{=}N$)-2-$HOC_6H_3COOSnR_3$ (IC_{50} = 0.9–6.6 μM against A549 and HeLa) (Liu et al., 2019).

3 Conclusion

Six new triorganotin(IV) 3-(arylmethyleneamino)benzoates (**1**–**6**) have been synthesized by the one-pot three-component reaction. In chloroform solution, the tin atoms in these complexes have a four coordinated environment. In the crystalline state, the compounds adopt a four- or five-coordination mode. Complex **5** exhibits a zigzag chain in which tin atom has a distorted *trans*-[C_3SnO_2] trigonal bipyramidal geometry, and other complexes (**1–4** and **6**) are discrete molecules, and tin atoms possess distorted [C_3SnO] tetrahedral configuration. These complexes display fluorescence with an emission at ~440 nm in CH_2Cl_2 solution at room temperature, and may be explored for potential blue luminescent materials. Compounds **1**–**6** have potent *in vitro* cytotoxic activity against A549 and HeLa tumor cell lines (IC_{50} value of 0.89–2.59 μM), indicating their potential as potent anticancer agents, and can be further studied.

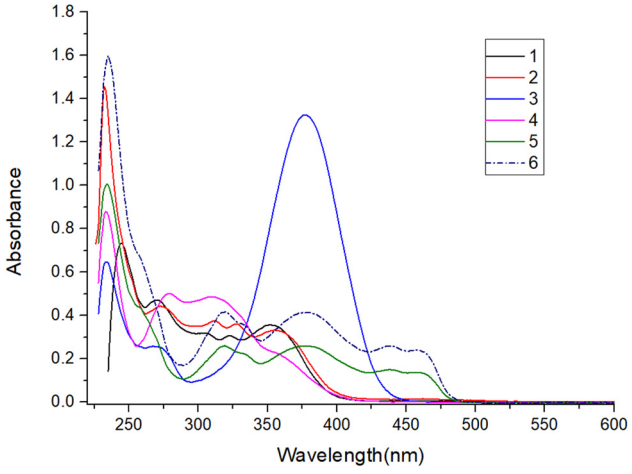


Figure 7: The UV-Vis spectra of complexes **1**–**6** in CH_2Cl_2 (3.0×10^{-5} M).

Experimental methods

Materials and physical measurements

The chemicals were of reagent grade and were purchased from Sinopharm Chemical Reagent Co. Ltd (Shanghai, China) and Energy Chemical Reagent Co. Ltd (Shanghai, China). The melting points were measured with a WRS-1A digital melting-point apparatus (Shanghai Precision Scientific Instrument Co. Ltd, Shanghai, China). C, H, and N analyses were performed using a Perkin Elmer 2400 Series II elemental analyzer (Perkin Elmer, Waltham, USA). IR spectra were recorded on a Nicolet Nexus 470 FT-IR spectrophotometer (Thermo Nicolet Corporation, USA) using KBr discs in the range of 4,000–400 cm^{-1}. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance III HD500 NMR spectrometer (Bruker Corporation, Switzerland) with $CDCl_3$ as solvent and Me_4Si (TMS) as internal standard, and the chemical shifts are reported in δ units (ppm). ^{119}Sn NMR spectra were recorded in $CDCl_3$ on a Varian Mercury Vx300 spectrometer using Me_4Sn external reference (Varian Corporation, USA). The UV-visible spectra were obtained on an Agilent 8453 spectrophotometer (Agilent Technologies Inc., USA). Fluorescence spectra were

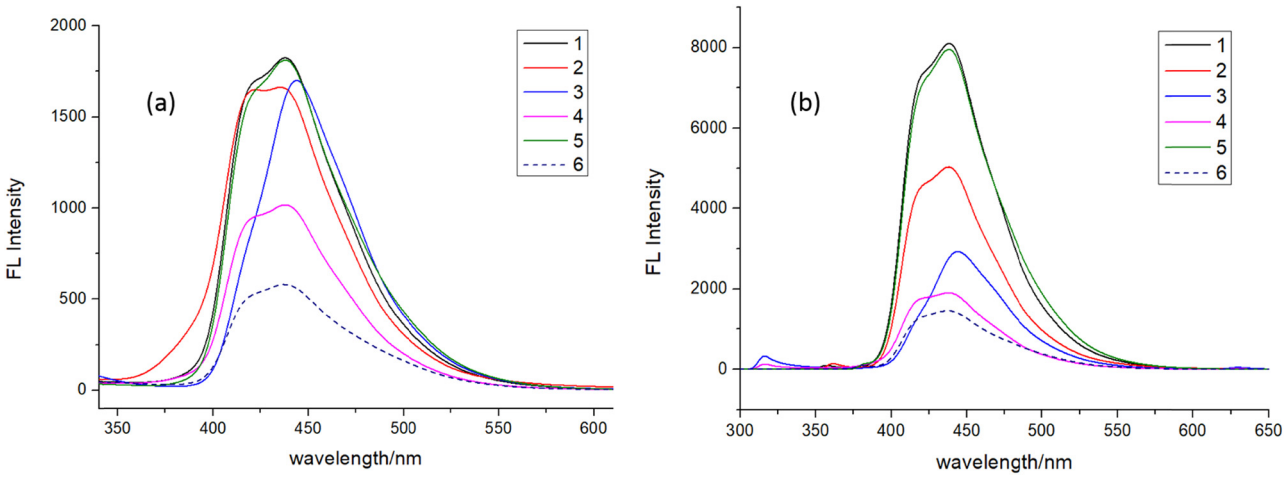


Figure 8: Fluorescence spectra of the complexes in CH_2Cl_2 (3.0×10^{-5} M). (a) At λ_{ex} = 310 nm and (b) at λ_{ex} = 352 nm (**1**), 356 nm (**2**), 377 nm (**3**), 356 nm (**4**), 378 nm (**5**), and 376 nm (**6**).

carried out by using a Hitachi F-4600 fluorescence spectrophotometer (HITACHI, Japan).

Synthesis of the complexes (1–6)

3-Aminobenzoic acid (0.137 g, 1 mmol), aldehyde (1 mmol) (for 5-bromo-2-hydroxybenzaldehyde 0.201 g, for 3,5-dibromo-2-hydroxybenzaldehyde 0.280 g, for 4-diethylamino-2-hydroxybenzaldehyde 0.193 g, for 2-hydroxy-3-methoxybenzaldehyde 0.152 g, for 2-hydroxy-1-naphthaldehyde 0.172 g), and triorganotin(IV) hydroxide (1 mmol) (for triphenyltin(IV) hydroxide 0.367 g, for tricyclohexyltin(IV) hydroxide 0.375 g) were mixed in 30 mL of benzene. The mixtures were refluxed under stirring, and the water formed during the reaction was removed using a Dean-Stark water separator. After the reaction is completed (about 6 h), the solution is cooled to room temperature and then filtered to remove the insoluble matter. The filtrate was evaporated by a rotary evaporator, and the residue was recrystallized from chloroform–methanol (1:3, v/v) or dichloromethane-hexane (1:2, v/v). The physical data of the compounds are as follows (for the NMR assignments, refer to the numbering in Scheme 1).

Table 4: IC_{50} (μM) of complexes recorded over a period of 24 h[a]

Complex	A549	Hela
1	1.89 ± 0.13	1.59 ± 0.06
2	1.39 ± 0.09	1.49 ± 0.11
3	2.49 ± 0.12	1.59 ± 0.11
4	1.79 ± 0.05	0.89 ± 0.03
5	2.59 ± 0.10	1.19 ± 0.06
6	1.19 ± 0.07	0.89 ± 0.06
Cisplatin	21.3 ± 1.7	7.8 ± 0.3

[a]Data represent mean value ± SD.

Triphenyltin(IV) 3-(5-bromo-2-hydroxybenzylideneamino)benzoate (1)

Light yellow product of 0.562 g (84%) was obtained. M.p. 149–150°C. Anal. Calcd for $C_{32}H_{24}BrNO_3Sn$: C 57.44,

Table 3: UV-Vis absorption data of the complexes[a]

Complex	λ_{max} (nm) (ε_{max} ($M^{-1}\cdot cm^{-1}$))				
1	245 (24,433)	270 (15,733)	306 (10,600)	352 (11,900)	
2	233 (48,233)	273 (14,766)	311 (12,500)	356 (11,066)	
3	234 (21,600)	270 (8,533)		377 (44,167)	
4	234 (29,233)	278 (16,700)	310 (16,200)	356 (7,600)	
5	235 (33,366)	319 (8,700)	377 (8,666)	438 (5,000)	459 (4,566)
6	235 (53,266)	319 (13,966)	377 (13,800)	438 (8,666)	457 (8,000)

[a]Measured in CH_2Cl_2 (3.0×10^{-5} M).

Table 5: Crystallographic and refinement data for the complexes

Complex	1	2	3	4	$[5\cdot CH_3OH\cdot CHCl_3]_2$	6
Formula	$C_{32}H_{24}BrNO_3Sn$	$C_{32}H_{23}Br_2NO_3Sn$	$C_{36}H_{52}N_2O_3Sn$	$C_{33}H_{45}NO_4Sn$	$C_{76}H_{64}Cl_6N_2O_8Sn_2$	$C_{36}H_{45}NO_3Sn$
Formula weight	669.12	748.02	679.48	638.39	1,583.37	658.42
Crystal system	Monoclinic	Triclinic	Monoclinic	Monoclinic	Triclinic	Triclinic
Space group	*C*2/*c*	*P*-1	$P2_1/n$	*C*2/*c*	*P*-1	*P*-1
a (Å)	52.125(7)	7.6169(15)	18.529(2)	43.516(4)	10.0543(14)	8.9941(13)
b (Å)	6.6015(8)	13.743(3)	9.6937(12)	7.8719(7)	16.823(2)	10.6435(15)
c (Å)	17.029(2)	15.096(3)	20.779(3)	19.3847(19)	22.907(3)	18.484(3)
α (°)	90	100.394(4)	90	90	89.664(4)	77.027(4)
β (°)	108.791(4)	100.809(4)	110.565(4)	109.226(3)	78.352(4)	77.660(4)
γ (°)	90	103.453(4)	90	90	73.409(3)	70.385(4)
Volume (Å^3)	5,547.4(12)	1,467.6(5)	3,494.4(8)	6,270.0(10)	3,631.2(9)	1,605.7(4)
Z	8	2	4	8	2	2
D_c (g·cm^{-3})	1.602	1.693	1.292	1.353	1.448	1.362
μ (mm^{-1})	2.395	3.626	0.766	0.851	0.964	0.831
F(000)	2,656	732	1,424	2,656	1,600	684
θ range (°)	3.1–26.0	1.8–26.0	2.3–26.0	2.1–26.0	2.2–26.0	2.5–26.0
Tot. reflections	32,075	9,466	40,142	34,750	75,403	31,932
Uniq. reflections	5,401	5,737	6,807	6,090	14,172	6,232
R_{int}	0.028	0.023	0.025	0.028	0.017	0.018
GOF on F^2	1.05	1.07	1.05	1.08	1.03	1.05
R_1 indices	0.031	0.041	0.035	0.042	0.032	0.023
wR_2 indices	0.072	0.101	0.102	0.119	0.091	0.064
$\Delta\rho_{min}$ (e·Å^{-3})	−1.346	−0.462	−0.523	−0.874	−0.725	−0.505
$\Delta\rho_{max}$ (e·Å^{-3})	1.311	1.000	0.874	1.549	1.167	0.683

H 3.62, N 2.09; found C 57.24, H 3.36, N 2.12%. IR (KBr, *ν*): 3,433 (O–H), 1,625 [$(COO)_{as}$ + C═N], 1,328 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, *δ*): 13.07 (bs, 1H, OH), 8.59 (s, 1H, CH═N), 8.08 (d, ^3J = 7.5 Hz, 1H, H-6 of C_6H_4), 8.03 (d, ^4J = 1.5 Hz, 1H, H-2 of C_6H_4), 7.82–7.80 (m, 6H, $^3J(^{119}Sn-^1H)$ = 60 Hz, *o*-H of Ph), 7.51–7.42 (m, 13H, Ar-H), 6.92 (t, ^3J = 9.0 Hz, 1H, H-3 of C_6H_3) ppm. ^{13}C NMR ($CDCl_3$, *δ*): 172.05 (C═O), 162.07 (C═N), 160.22, 148.02, 136.00, 134.44, 132.25, 129.42, 126.13, 122.59, 120.48, 119.36, 110.63 (aromatic carbons), 138.19 ($^1J(^{117/119}Sn-^{13}C)$ = 614/640 Hz, *i*-C of Ph), 136.95 ($^2J(^{119}Sn-^{13}C)$ = 48 Hz, *o*-C of Ph), 130.33 ($^4J(^{119}Sn-^{13}C)$ = 13 Hz, *p*-C of Ph), 129.05 ($^3J(^{119}Sn-^{13}C)$ = 63 Hz, *m*-C of Ph) ppm. ^{119}Sn NMR ($CDCl_3$) *δ*: −102.7 ppm.

Triphenyltin(IV) 3-(3,5-dibromo-2-hydroxybenzylideneamino)benzoate (2)

The orange crystals (0.605 g, 81%) are obtained. M.p. 169–171°C. Anal. Calcd for $C_{32}H_{23}Br_2NO_3Sn$: C 51.38, H 3.10, N 1.87; found C 51.44, H 3.08, N 1.83%. IR (KBr, *ν*): 3,432 (O–H), 1,637 [$(COO)_{as}$], 1,612 (C═N), 1,324 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, *δ*): 14.20 (bs, 1H, OH), 8.68 (s, 1H, CH═N), 8.10 (d, ^3J = 7.5 Hz, 1H, H-6 of C_6H_4), 8.05 (s, 1H, H-2 of C_6H_4), 7.82–7.80 (m, 6H, $^3J(^{119}Sn-^1H)$ = 60 Hz, *o*-H of Ph), 7.75 (d, ^4J = 2.5 Hz, 1H, H-6 of C_6H_2), 7.52–7.44 (m, 12H, Ar-H) ppm. ^{13}C NMR ($CDCl_3$, *δ*): 171.87 (C═O), 161.02 (C═N), 157.33, 146.86, 138.39, 133.65, 132.42, 129.92, 129.58, 126.36, 122.33, 120.61, 112.25, 110.42 (aromatic carbons), 138.06 ($^1J(^{117/119}Sn-^{13}C)$ = 614/642 Hz, *i*-C of Ph), 136.98 ($^2J(^{119}Sn-^{13}C)$ = 49 Hz, *o*-C of Ph), 130.37 ($^4J(^{119}Sn-^{13}C)$ = 13 Hz, *p*-C of Ph), 129.07 ($^3J(^{119}Sn-^{13}C)$ = 64 Hz, *m*-C of Ph) ppm. ^{119}Sn NMR ($CDCl_3$) *δ*: −102.4 ppm.

Tricyclohexyltin(IV) 3-(4-diethylamino-2-hydroxybenzylideneamino)benzoate (3)

The brown crystal of **3** was obtained in 76% (0.516 g) yield. M.p. 136–138°C. Anal. Calcd for $C_{36}H_{52}N_2O_3Sn$: C 63.63, H 7.71, N 4.12; found C 63.44, H 7.68, N 4.16%. IR (KBr, *ν*): 3,440 (O–H), 1,634 [$(COO)_{as}$ + C═N], 1,329 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, *δ*): 13.61 (vbs, 1H, OH···N), 8.50 (s, 1H, CH═N), 7.95 (s, 1H, H-2 of C_6H_4), 7.90 (d, ^3J = 7.5 Hz, 1H, H-6 of C_6H_4), 7.41 (t, ^3J = 7.5 Hz, 1H, H-5 of C_6H_4), 7.37 (d, ^3J = 8.5 Hz, 1H, H-4 of C_6H_4), 7.16 (d, ^3J = 9.0 Hz, 1H, H-6 of C_6H_3), 6.25 (dd, ^4J = 2.0 Hz, ^3J = 9.0 Hz, 1H, H-5 of C_6H_3), 6.19 (d, ^4J = 2.0 Hz, 1H, H-3 of C_6H_3), 3.40 (q, ^3J = 7.0 Hz, 4H, $2NCH_2$), 2.03–1.33 (m, 33H, Cy), 1.21 (t, ^3J = 7.0 Hz, 6H, $2CH_3$) ppm. ^{13}C NMR ($CDCl_3$, *δ*): 171.06 (COO),

160.95 (C═N), 164.21, 151.93, 148.80, 133.90, 133.54, 128.96, 127.18, 125.11, 121.67, 109.11, 103.87, 97.74 (aromatic carbons), 44.59 ($2NCH_2$), 12.71 ($2CH_3$), 33.97 ($^1J(^{117/119}Sn–^{13}C)$ = 322/336 Hz, α-C), 31.14 ($^2J(^{119}Sn–^{13}C)$ = 15 Hz, β-C), 28.94 ($^3J(^{119}Sn–^{13}C)$ = 65 Hz, γ-C), 26.92 (δ-C) (Cy) ppm. ^{119}Sn NMR ($CDCl_3$) δ: 18.6 ppm.

Tricyclohexyltin(IV) 3-(3-methoxy-2-hydroxybenzylideneamino)benzoate (4)

The reaction gave an orange product with 88% (0.562 g) yield. M.p. 86–88°C. Anal. Calcd for $C_{33}H_{45}NO_4Sn$: C 62.08, H 7.10, N 2.19; found C 62.22, H 6.96, N 2.21%. IR (KBr, ν): 3,433 (O–H), 1,641 [$(COO)_{as}$], 1,617 (C═N), 1,327 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, δ): 13.50 (bs, 1H, OH), 8.71 (s, 1H, CH═N), 8.01 (s, 1H, H-2 of C_6H_4), 8.00 (d, ^3J = 7.5 Hz, 1H, H-6 of C_6H_4), 7.47 (t, ^3J = 7.5 Hz, 1H, H-5 of C_6H_4), 7.43 (d, ^3J = 7.5 Hz, 1H, H-4 of C_6H_4), 7.03 (d, ^3J = 8.0 Hz, 1H, H-6 of C_6H_3), 7.00 (d, ^3J = 8.0 Hz, 1H, H-4 of C_6H_3), 6.89 (t, ^3J = 7.5 Hz, 1H, H-5 of C_6H_3), 3.94 (s, 3H, OCH_3), 1.33–2.04 (m, 33H, Cy) ppm. ^{13}C NMR ($CDCl_3$, δ): 170.72 (COO), 163.30 (C═N), 151.47, 148.50, 148.17, 133.83, 129.18, 128.70, 125.69, 123.93, 121.86, 119.11, 118.65, 114.93 (aromatic carbons), 56.23 (OCH_3), 34.04 ($^1J(^{117/119}Sn–^{13}C)$ = 323/338 Hz, α-C), 31.15 ($^2J(^{119}Sn–^{13}C)$ = 14 Hz, β-C), 28.94 ($^3J(^{119}Sn–^{13}C)$ = 64 Hz, γ-C), 26.91 (δ-C) (Cy) ppm. ^{119}Sn NMR ($CDCl_3$) δ: 19.4 ppm.

Triphenyltin(IV) 3-((2-hydroxy-1-naphthalenyl)methyleneamino)benzoate (5)

Orange-yellow crystals, yield 0.512 (80%). M.p. 168–171°C. $C_{36}H_{27}NO_3Sn$: C 67.53, H 4.25, N 2.19; found C 67.59, H 4.24, N 2.15%. IR (KBr, ν): 3,431 (O–H), 1,623 [$(COO)_{as}$ + C═N], 1,353 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, δ): 15.33 (bs, 1H, O–H···N), 9.39 (s, 1H, CH═N), 8.13 (d, ^3J = 8.5 Hz, 1H, H-6 of C_6H_4), 8.13 (s, 1H, H-2 of C_6H_4), 8.07–8.05 (m, 1H, H of $C_{10}H_6$), 7.82 (dd, ^4J = 1.0 Hz, ^3J = 7.0 Hz, $^3J(^{119}Sn–^1H)$ = 64 Hz, 6H, *o*-H of Ph), 7.80 (d, ^3J = 9.0 Hz, 1H, H of $C_{10}H_6$), 7.71 (d, ^3J = 8.0 Hz, 1H, H of $C_{10}H_6$), 7.54–7.48 (m, 12H, (*m*,*p*)-H of Ph + H of $C_{10}H_6$), 7.34 (t, ^3J = 8.0 Hz, 1H, H-5 of C_6H_4), 7.09 (d, ^3J = 9.0 Hz, 1H, H-4 of C_6H_4) ppm. ^{13}C NMR ($CDCl_3$, δ): 171.93 (COO), 169.82 (CH═N), 155.44, 145.52, 136.86, 133.13, 132.42, 129.49, 129.33, 128.51, 128.15, 127.33, 125.17, 123.65, 121.92, 121.55, 119.08, 108.91 (aromatic carbons), 138.15 ($^1J(^{117/119}Sn–^{13}C)$ = 620/648 Hz, *i*-C of Ph), 136.92 ($^2J(^{119}Sn–^{13}C)$ = 50 Hz, *o*-C of Ph), 130.27 ($^4J(^{119}Sn–^{13}C)$ = 13 Hz, *p*-C of Ph), 129.00 ($^3J(^{119}Sn–^{13}C)$ = 62 Hz, *m*-C of Ph) ppm. ^{119}Sn NMR ($CDCl_3$) δ: –105.4 ppm.

Tricyclohexyltin(IV) 3-((2-hydroxy-1-naphthalenyl)methyleneamino)benzoate (6)

Orange-yellow crystals, yield 0.533 g (81%). M.p. 117–119°C. Anal. Calcd for $C_{36}H_{45}NO_3Sn$: C 65.67, H 6.89, N 2.13; found C 65.44, H 6.86, N 2.19%. IR (KBr, ν): 3,435 (O–H), 1,618 [$(COO)_{as}$ + C═N], 1,327 [$(COO)_s$] cm^{-1}. ^{1}H NMR ($CDCl_3$, δ): 15.43 (bs, 1H, OH···N), 9.42(s, 1H, CH═N), 8.13 (d, ^3J = 9.0 Hz, 1H, H-6 of C_6H_4), 8.11 (s, 1H, H-2 of C_6H_4), 8.00–7.98 (m, 1H, H of $C_{10}H_6$), 7.80 (d, ^3J = 9.0 Hz, 1H, H of $C_{10}H_6$), 7.71 (d, ^3J = 8.0 Hz, 1H H of $C_{10}H_6$), 7.54–7.49 (m, 3H, H of $C_{10}H_6$), 7.34 (t, ^3J = 7.5 Hz, 1H, H-5 of C_6H_4), 7.09 (d, ^3J = 9.0 Hz, 1H, H-4 of C_6H_4), 2.06–1.32 (m, 33H, Cy) ppm. ^{13}C NMR ($CDCl_3$, δ): 170.52 (COO), 170.15 (CH═N), 154.97, 145.24, 136.79, 134.09, 133.21, 129.41, 129.34, 128.17, 128.11, 127.32, 124.63, 123.60, 122.11, 120.88, 119.01, 108.88 (aromatic carbons), 34.06 ($^1J(^{117/119}Sn–^{13}C)$ = 322/336 Hz, α-C), 31.16 ($^2J(^{119}Sn–^{13}C)$ = 15 Hz, β-C), 29.83 ($^3J(^{119}Sn–^{13}C)$ = 63 Hz, γ-C), 26.91 ($^4J(^{119}Sn–^{13}C)$ = 7 Hz, δ-C) (Cy) ppm. ^{119}Sn NMR ($CDCl_3$) δ: 17.3 ppm.

X-ray crystallography

The yellow to orange single crystals of **1**–**6** were obtained by slow evaporation of $CHCl_3$-MeOH (1:2, v/v) solution, respectively. Compound **5** crystallizes with two molecules of each MeOH and $CHCl_3$ (**5**·CH_3OH·$CHCl_3$). Diffractions data were collected at 295(2) K on a Bruker D8 Quest CCD fitted with graphite monochromatized Mo-Kα radiation (0.71073 Å). The structure solution and refinement were completed using SHELXS Version 2014/5 (Sheldrick, 2015a) and SHELXL-2018 (Sheldrick, 2015b), respectively. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms bonded to C were included in their calculated positions and refined in the riding-model, and hydrogen atoms of the hydroxyl and amino groups were freely refined. Crystal data and refinement details are listed in Table 5. Crystallographic data have been deposited with the Cambridge Crystallographic Data Center as supplementary publication numbers CCDC 2167238–2167243.

3.1 *In vitro* screening

The human tumor cell lines A549 (lung tumor cells) and HeLa (cervix tumor cells) were obtained from the Cell Bank of Type Culture Collection of the Chinese Academy of

Sciences, Shanghai, China. The cells were cultured in Dulbecco's modified Eagle medium supplemented with 10% heat-inactivated new-born calf serum and 1% penicillin–streptomycin solution at 310 K in a humidified 5% CO_2 incubator.

After plating 5,000 tumor cells per well in 96-well plates, the cells were pre-incubated in drug-free media at 310 K for 24 h before adding various concentrations of the tin complexes to be tested. In order to prepare stock solutions, each complex was dissolved in DMSO and cisplatin was dissolved in phosphate-buffered saline of pH 7.2. This stock was further diluted using cell culture medium until working concentrations were achieved. The drug exposure period was 24 h. Subsequently, 15 µL of 5 $mg{\cdot}mL^{-1}$ MTT solution was added to form purple formazan. Afterwards, 100 µL of DMSO was transferred into each well to dissolve the purple formazan. Each well was triplicated and each experiment repeated at least three times. The dose causing 50% inhibition of cell growth (IC_{50}) was calculated and the data were expressed as the mean values ± standard deviation.

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Author contributions: Ruili Wang: experimental work and writing – original draft; Jing Zhang: experimental work; Daoyu Cui: experimental work; Laijin Tian: methodology and writing – review and editing.

Conflict of interest: Authors state no conflict of interest.

Data availability statement: Crystallographic data (CCDC 2167238–2167243) for this article can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

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