



2-, 3-, and 4-(1-Oxo-1*H*-2,3-dihydroisoindol-2-yl)benzoic acids and their corresponding organotin carboxylates: Synthesis, characterization, fluorescent, and biological activities

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ABSTRACT

Three novel organotin complexes with general formula $\text{Sn}(\text{OH})(\text{bz})_2\text{L}$ (bz = benzyl, HL = 2-, 3-, or 4-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)benzoic acid) and one of their ligands were prepared and characterized. In vitro antifungal and antibacterial activities of these complexes and ligands were investigated with the representative strains of *Candida albicans*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas aeruginosa*. Their fluorescence properties have also been discussed.

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Heterocyclic compounds containing the phthalimidine (isoindolin-1-one) skeleton are being paid more and more attention due to their fascinating properties and potential applications in many fields, especially in biology and medical science.¹ The recent elaboration has proven that the phthalimidino group plays a key role in the pharmacological activity of correlated synthetic and natural compounds.² The new phthalimidine derivative with unique biological activity might be a precursor of efficacious drug for therapeutic and prophylactic use, such as antipsychotics, hypolipidemic, and antimicrobial.³ In the last decades, many new *N*- and *C*-substituted phthalimidine derivatives have been synthesized and tested for their diverse biological activity.⁴ Although the majority of synthetic *N*-substituted derivatives are *N*-alkylphthalimidines, and most of them display no antifungal activity.^{1–4} The relatively less regarded *N*-arylphthalimidines might also be an alternative strategy in designing potent agents for medical use, in which the electronic delocalization in *N*-arylphthalimidines will be bound to affect the stability of the phthalimidino ring which is closely related to the bioactivity of this kind of compound.^{2,5} Moreover, many substituents such as carboxyl and hydroxyl them-

selves might be able to endow the compound with a broader spectrum of biological activity.⁶ Thus, a further study regarding the synthesis, bioeffect and structure–activity relationship of this kind of compound is essential. The combination of functionals from different kinds of compounds with extensive bioactivities could often be helpful for good selectivity and stronger activity as well as lower toxicity. Thus, an organotin complex containing phthalimidino group can improve the bioactivity of phthalimidines in view of the special bioactivity widely existed in a variety of organotin compound. However, to the best of our knowledge, no example of this kind of compound has been reported until now.

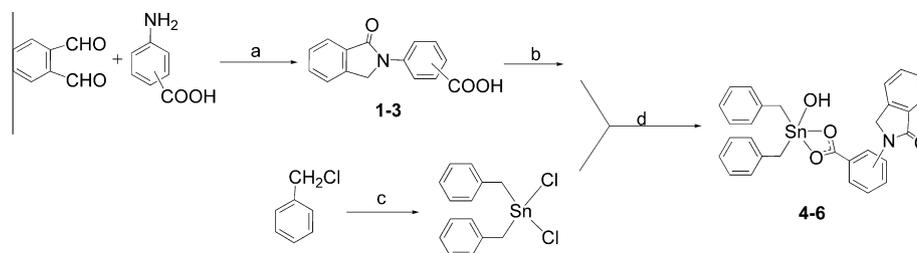
Herein, as a part of our ongoing research projects, we report the synthesis and characterization of 3-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)benzoic acid (**1**, mHpba, positional isomer of our previously reported 2- and 4-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)benzoic acid (oHpba **2** and pHpba **3**, respectively))⁷ and three correlated organotin carboxylates with general formula $\text{Sn}(\text{OH})(\text{bz})_2\text{L}$ (**4**, **5**, and **6**, bz = benzyl, HL = mHpba, oHpba, and pHpba, respectively). The latter are the first examples of organotin complex with phthalimidine derivative. The fluorescent and biological activities of all these six compounds have been investigated.

According to the synthetic scheme described in Scheme 1, compounds **4**, **5**, and **6** were prepared by the one-step reaction of dibenzyltin chloride and the corresponding sodium salt of the phthalimidinobenzoic acids **1–3** in an ethanol/dimethylformamide (2:1) mixture with moderate yield.^{9,10} Both of the two kinds of

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Scheme 1. Synthetic method for compounds **1–6**: (a) EtOH, 90 °C; (b) NaOH; (c) Sn, H₂O, toluene, 123 °C; (d) EtOH/DMF, 50 °C.

reactants were prepared by the reported methods in the literature.^{11–13}

Although an attempt to obtain single crystals of compounds **1** and **4–6** for single-crystal X-ray diffraction was not successful, their molecular structure has been elucidated by elemental analyses, ¹H NMR, IR, and mass spectra.^{14–17} The results of elemental analyses are in good agreement with the proposed formulas. The molecular ion peak at *m/z* 253 and one set of fragments in the mass spectrum of **1** are fully consistent with its molecular structure as drawn. The IR spectrum of **1** showed a broad band at 3421 cm⁻¹ assigned to ν(O–H), and the medium band at 3062 cm⁻¹ ascribed to the C–H stretching of the aromatic ring. The bands of the antisymmetric and symmetric stretching vibrations of methylene C–H bond are observed at 2906 and 2827 cm⁻¹, respectively. The broad band around 2538 cm⁻¹ indicated the presence of strong hydrogen bond interaction.¹⁸ The characteristic strong absorption of the stretching vibration of lactam carbonyl was redshifted towards 1633 cm⁻¹ due to the conjugation between the benzene ring, carbonyl group, and an sp²-hybridized nitrogen.⁷ The free carboxyl group are clearly present at 1695 (vs, ν(C=O)), 1385 (s, O–H bend), 1257 (s, ν(C–O)) cm⁻¹.¹⁹ Bands at 1587, 1491, and 1444 cm⁻¹ are attributed to the skeleton vibration of aromatic ring, and the *meta*-disubstitution pattern on benzene ring was confirmed by the peaks at 758 (s) and 685 (m) cm⁻¹ (C–H out-of-plane bend).²⁰ The most significant change in IR spectra from the ligands **1–3** to their organotin carboxylates **4–6** mainly are embodied in the following three aspects: (1) the disappearance of strong absorption at ca. 1700 cm⁻¹ corresponding to ν(C=O) of carboxyl group; (2) the presence of obvious absorption at ca. 1545 for ν_{asym}(COO), 550 cm⁻¹ for Sn–C and 450 cm⁻¹ for Sn–O;²¹ (3) the appropriate blue shift (ca. 20 cm⁻¹) of ν(C=O) of the lactam carbonyl group in complex compared to that of **1**. These changes clearly indicated the deprotonation and participation of the carboxyl group in the complexation process with Sn(IV) cation. The Δν[ν_{asym}(COO) – ν_{sym}(COO)] in the range 160–210 cm⁻¹ suggested the chelating bidentate coordination mode of the carboxyl group in these three organotin complexes.^{21,22} As a result, the Sn(IV) center is coordinated by two C atoms from two benzyl groups and three O atoms from one carboxyl and one hydroxyl group, forming a distorted trigonal bipyramidal geometry. This mode is also proven by the ¹H NMR spectroscopic data, in which the most fascinating character is the coupling pattern and chemical shift related to the bridging methylene protons. Because of the lack of a plane of symmetry in the molecule, the methylenes of two benzyl groups as well as the relevant protons are stereochemically and magnetically non-equivalent. Large chemical-shift differences were observed between these methylene protons. Complex **5** gives typical two AX patterns for two groups of bridge C₆H₅CH₂Sn protons at δ 2.03–2.29 and 2.66–3.03, which arise as an AB system and an AX system in **4** (δ 2.58 and 2.68–2.86) and **6** (δ 2.61 and 2.79–2.99). The diagnostic signal of the resonance of bridging methylene protons of the phthalimidino group is represented by an AB system at ca. δ 6.54–6.72 the three complexes, which often

appear as a singlet in ¹H NMR of related free ligands at ca. δ 5 ppm.^{7,8} In addition, the existence of the hydroxyl hydrogen atom in the three complexes are confirmed by the singlet in the region of δ 1.24–3.27.

In complexes, the Δν value of 160–210 cm⁻¹ indicated the difference of the two O atoms of the carboxyl group with chelating bidentate coordination mode. Meanwhile, the big unsymmetrical phthalimidino carboxylate group⁷ tend to hamper the rotation of the two benzyl groups, and it inevitably lead to the large chemical-shift difference between the methylene protons with magnetically non-equivalent as above mentioned. And vice versa, the IR and NMR data suggested that there is no symmetry plane (σ) in these organotin molecules. Obviously, the inversion center (*i*) and 4n-fold improper axis (S_{4n}) of rotation cannot exist in these molecules. Therefore, the organotin carboxylate molecules have no type of symmetry whatever, and are chiral molecules. Whereas, it would be beyond the scope of this study to assign the chiral Sn(IV) centers as *R* or *S*, since the stereoisomers have not been separated into enantiomers.

In order to study the bioactivity of this kind of ligand and their organotin carboxylates, their *in vitro* antifungal and antibacterial activities have been evaluated by the minimum inhibition concentration (MIC) test using the doubling dilution technique against fungus *Candida albicans*, Gram-positive bacteria *Staphylococcus aureus*, and Gram-negative bacteria *Escherichia coli* and *Pseudomonas aeruginosa*. The results are summarized in Table 1. As revealed from these data, all the above mentioned six compounds were found to be not only active against these bacteria corresponds well with that of the reported N-substituted phthalimidines, but also to be active against fungi which is infrequent for this kind of compound.⁶ Moreover, the organotin carboxylates **4–6** display a great improvement in antibacterial and antifungal activity upon their corresponding phthalimidine derivatives **1–3**. It is also worth mentioning that the inhibition efficiency, in cases both of phthalimidine ligands and of organotin carboxylates, is dependent somewhat on the substitution position of the carboxylate group on its N-connected benzene ring. Among which, the compounds **1** and **4** with the substituent carboxylate group on the *meta*-posi-

Table 1

In vitro antifungal and antibacterial activities as the minimum inhibition concentration (MIC) values (mg/mL)^a for compounds **1–6**

Compounds	E.c. ^b	P.a. ^c	S.a. ^d	C.a. ^e
1	0.625	0.625	1.25	0.625
2	2.5	1.25	2.5	1.25
3	1.25	0.625	1.25	1.25
4	0.0625	0.125	0.0625	0.125
5	0.125	0.125	0.0625	0.125
6	0.125	0.125	0.0625	0.125

^a Values are means of three experiments.

^b E.c. = *Escherichia coli*.

^c P.a. = *Pseudomonas aeruginosa*.

^d S.a. = *Staphylococcus aureus*.

^e C.a. = *Candida albicans*.

tion are found to be more active than the others, while the ligand **2** with the carboxylate group on the *ortho*-position exhibit lowest activity in three ligands, and its organotin carboxylate **5** display the same activity as **6** whose carboxylate group is on the *para*-position.

The substituent position can also strongly influence the fluorescence of the phthalimidine derivatives. Distinctly unlike its position isomers **2** and **3** which emits intense fluorescence around 408 nm (in DMF), ligand **1** exhibits only a very weak emission at 455 nm with a large redshift of 47 nm.⁸ While the emission spectra of its organotin carboxylate **4** (in the solid state at room temperature) shows this ligand-centered (LC, $n-\pi^*$ and/or $\pi-\pi^*$) emission at 451 nm as a very strong peak with a shoulder peak at 425 nm. Simultaneously, it also exhibits two new strong emissions at 471 and 526 nm which may be assigned as ligand-to-metal charge transfer (LMCT) or metal-to-ligand charge transfer (MLCT).²³ The emission spectra of organotin carboxylate **6** is quite similar to that of **4** except the significant enhancement of the yellow-green emission at 525 nm which may come from a higher conjugation of the ligand **3** than that of **4**.⁷ However, complex **5** displays the only fluorescence maximum at 409 nm which is attributed to its ligand **2** just with an obvious enhancement. It could be envisioned that these remarkable and unique fluorescent property of phthalimidine derivatives might be useful in exploiting some sensory and diagnostic applications.

In conclusion, this Letter describes the preparation and characterization of a new N-substituted phthalimidine and three organotin carboxylates as the first examples of organotin complex of phthalimidine derivatives with broad spectrum resistance and great improved antibacterial and antifungal activity upon their corresponding phthalimidine derivatives. The substitution position of the carboxylate group on their N-connected benzene ring strongly influenced the bioactivity and fluorescent activities of both the ligands and the organotin carboxylates.

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- Chemicals and reagents were purchased commercially and used as received. Thin-layer chromatography (TLC) analyses were conducted with silica gel-coated plate (from Qingdao Ocean Chemicals). C, H, and N-microanalyses were carried out with a Vario EL III elemental analyzer. Melting point of compound **1** was determined using a Beijing Taike XT-4 microscopy melting point apparatus and was reported uncorrected. The IR spectra were recorded as KBr discs on a Nicolet Magna 750 FT-IR spectrometer. ¹H NMR spectrum was obtained at room temperature on Varian INOVA-400 spectrometer, and the chemical shift scale (ppm) is based on internal standard tetramethylsilane. LC–MS analyses were performed using a Waters Micromass ZQ-4000 spectrometer. Fluorescent analyses were carried out with an F-2500 fluorescence spectrophotometer with the slit widths of excitation and emission at 10.0 nm.
- Synthesis of **4**, **5**, and **6**: Related ligand **1**, **2**, or **3** (0.0405 g, 0.16 mmol) and sodium hydroxide (0.040 g, 1.0 mmol) were successively added to a mixture of dibenzyltin chloride (0.2976 g, 0.80 mmol) in EtOH/DMF solution (4:1, 30 mL). The reactant mixture was violently stirred for 2 h at 60–80 °C. Then, the resulting mixture was cooled down to room temperature followed by filtration. The residue for **4** and **6** were washed with EtOAc and acetone for four times and dried for 1 h under vacuum at about 60 °C to give the compounds **4** (0.043 g) and **6** as pale yellow solid (0.035 g) with the yield of 47% and 38%, respectively. On the other hand, the filtrate for **5** was concentrated by rotary evaporation followed by standing at room temperature for 3 days to produce compound **5** as white crystal (0.027 g, 30%). Mp: >300 °C. Anal. Calcd for C₂₉H₂₅NO₄Sn: C, 60.94; N, 2.45; H, 4.41. Found for **4**: C, 69.75; N, 2.40; H, 4.31. Found for **5**: C, 70.22; N, 2.38; H, 4.27. Found for **6**: C, 70.31; N, 2.34; H, 4.25.
- Synthesis of **1**: *Ortho*-phthalaldehyde (OPA) (0.685 g, 5.0 mmol) was added to a stirred solution of *meta*-aminobenzoic acid (0.670 g, 5.0 mmol) in the mixture of acetone (10 mL) and deionized water (50 mL). The reactant mixture was refluxed for 3 h at about 110 °C. After the completion of the reaction (TLC, 4:1 EtOAc/MeOH), the resulting mixture was cooled down to room temperature followed by filtration, and the residue was washed with water, extracted with EtOAc. Then, recrystallized from EtOH and dried under vacuum to give the compound **1** as brownish black solid (0.7080 g, 56%). Mp: 182 °C. Anal. Calcd for C₁₅H₁₁NO₃: C, 71.13; N, 5.53; H, 4.38. Found: C, 70.39; N, 5.45; H, 4.29.
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- Compound **1**: IR (KBr pellets): 3421 (m, br), 3062 (m), 2906 (m), 2827 (m), 2619 (m), 2538 (m, br), 1695 (vs), 1633 (s), 1587 (s), 1491 (m), 1444 (s), 1385 (s), 1296 (s), 1257 (s), 1225 (s), 1159 (m), 1120 (m), 1080 (m), 997 (w), 945 (w), 912 (w), 816 (w), 758 (s), 685 (m), 567 (w) cm⁻¹; ¹H NMR (DMSO-*d*₆, 400 MHz): δ 5.10 (s, 2H, ArCH₂N), 7.69–7.71 (m, 2H, Ar-H₄, 6), 7.55–7.60 (m, 2H, Ar-H₇), 7.81 (d, *J* = 7.6 Hz, 2H, Ar-H_{5,13}), 8.16 (d, *J* = 8.0 Hz, 1H, Ar-H₁₄), 8.53 (s, 1H, Ar-H₁₀), 12.94 (s, br, 1H, COOH). ES-MS (70 eV, *m/z*): 253 ([M]⁺), 236, 225, 207, 178, 148, 132, 117, 105, 89, 76, 65, 44.
- Compound **4**: IR (KBr pellets): 3575 (w, br), 3055 (w), 3022 (m), 2920 (w), 2864 (w), 2000–1700 (vw), 1653 (s), 1595 (s), 1545 (s), 1489 (s), 1448 (s), 1385 (s), 1257 (m), 1205 (m), 1107 (m), 1053 (m), 1028 (m), 1001 (w), 947 (w), 904 (m), 800 (m), 758 (vs), 696 (vs), 627 (s), 584 (m), 553 (s), 449 (m) cm⁻¹; ¹H NMR (CDCl₃, 400 MHz): δ 2.58 (AB system, *J* = 12.8 Hz, 2H, ArCH₂Sn), 2.68 and 2.86 (AX system, *J* = 11.6 Hz, 2H, ArCH₂Sn), 1.24 (s, 1H, Sn(OH)), 6.72 (AB system, *J* = 8.0 Hz, 2H, ArCH₂N), 6.82–7.40 (m, 18H, Ar-H).
- Compound **5**: IR (KBr pellets): 3616 (w), 3431 (m, br), 3057 (m), 3022 (m), 2924 (w), 2878 (w), 2000–1700 (vw), 1662 (s), 1599 (s), 1545 (s), 1491 (s), 1450 (m), 1414 (s), 1358 (vs), 1254 (w), 1209 (m), 1180 (w), 1155 (w), 1107 (m), 1055 (m), 1030 (w), 999 (w), 972 (w), 904 (w), 839 (w), 758 (vs), 698 (vs), 611 (s), 555 (m), 517 (s), 453 (m), 400 (m) cm⁻¹; ¹H NMR (CDCl₃, 400 MHz): δ 2.03 and 2.29 (AX system, *J* = 12.4 Hz, 2H, ArCH₂Sn), 2.66 and 3.03 (AX system, *J* = 11.6 Hz, 2H, ArCH₂Sn), 3.27 (s, 1H, Sn(OH)), 6.54 (AB system, *J* = 7.2 Hz, 2H, ArCH₂N), 6.96–7.42 (m, 18H, Ar-H).
- Compound **6**: IR (KBr pellets): 3425 (m, br), 3059 (m), 3026 (m), 2927 (w), 2864 (w), 2000–1700 (vw), 1651 (s), 1593 (vs), 1512 (s), 1452 (m), 1377 (s), 1333 (s), 1308 (s), 1265 (m), 1201 (m), 1174 (m), 1138 (m), 1107 (m), 1059 (m), 1026 (w), 895 (w), 858 (m), 760 (s), 727 (m), 698 (s), 660 (m), 604 (m), 557 (m), 503 (m), 479 (m) cm⁻¹; ¹H NMR (CDCl₃, 400 MHz): δ 2.61 (AB system, *J* = 12.4 Hz, 2H, ArCH₂Sn), 2.79 and 2.99 (AX system, *J* = 11.6 Hz, 2H, ArCH₂Sn), 2.17 (s, 1H, Sn(OH)), 6.69 (AB system, *J* = 8.0 Hz, 2H, ArCH₂N), 6.8–7.4 (m, 18H, Ar-H).
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