Reactions of organotin chlorides R_2SnCl_2 (R = Et, Bu^t, or Ph) with lithium 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-amidophenolate. Synthesis and structures of tin(IV) *o*-iminoquinone complexes

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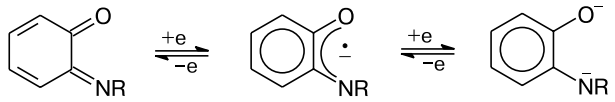
The exchange reactions of tin diorganohalides R_2SnCl_2 (R = Et, Bu^t, or Ph) with lithium amidophenolate $APLi_2$ (AP is the 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-iminobenzoquinone dianion) in tetrahydrofuran produced the new five-coordinate ($Et_2SnAP(THF)$ (**3**)) and four-coordinate (R_2SnAP (R = Bu^t, Ph)) tin(IV) complexes. The reaction of Ph_2SnCl_2 with $APLi_2$ in a nonpolar solvent (hexane or toluene) is accompanied by the additional redox process giving rise to the paramagnetic complex $Ph_2Sn(ImSQ)Cl$ (**6**) (ImSQ is the 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-iminobenzoquinone radical anion). The molecular structures of complexes **3** and **6** were established by X-ray diffraction.

Key words: *o*-iminobenzoquinone, tin, *o*-iminobenzosemiquinolate, amidophenolate, electron paramagnetic resonance, X-ray diffraction study.

o-Iminobenzoquinones are structural analogs of both *o*-quinones and *o*-diimines, being intermediate between these groups of compounds. Among these classes, the coordination chemistry of *o*-iminoquinones has been less well studied. Like *o*-quinones and *o*-diimines, *o*-iminobenzoquinones, being involved in complexes with metals, can undergo two-step one-electron reduction to form the *o*-iminosemiquinone radical anions and the amidophenolate dianion (Scheme 1).

Scheme 1



The introduction of various substituents at the nitrogen atom of *o*-iminobenzoquinone enables the variation of steric shielding of the metal center in complexes in a wide range.

For example, homoligand transition metal complexes with ligands based on *o*-iminobenzoquinone containing the phenyl substituent at the nitrogen atom can contain three *o*-iminosemiquinone ligands, which form a six-coordinate environment of metal.[1—3] The use of sterically more hindered *o*-iminoquinones, in particular, those containing the 2,6-diisopropylphenyl group at the nitrogen atom, allows the synthesis of iron, cobalt, and manganese complexes containing only two iminosemiquinone ligands, which corresponds to the lower-valence and low-coordination state of metal.[4—7] This is reflected in chemical activity of the resulting compounds. Since these compounds are valent- and coordinatively unsaturated, they can fix small molecules.[5] The presence of the redox-active *o*-amidophenolate ligand imparts new properties to main-group metal complexes, as exemplified by the reversible binding of molecular oxygen by antimony(V) complexes.[8]

Recently, we have synthesized the homoligand tin(II) and tin(IV) *o*-amidophenolate complexes[9] based on sterically hindered 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-iminobenzoquinone (**1**). In the present study, we describe the synthesis and structures of a series of tin(IV) amidophenolate complexes containing alkyl and aryl substituents at the metal atom.

Results and Discussion

The exchange reactions of alkali metal derivatives with metal halides are widely used for the synthesis of various coordination compounds. Dilithium salt **2** can easily be prepared by reduction of *o*-iminoquinone **1** with excess lithium both in polar (THF or Et_2O) and nonpolar (toluene or hexane) organic solvents (Scheme 2).

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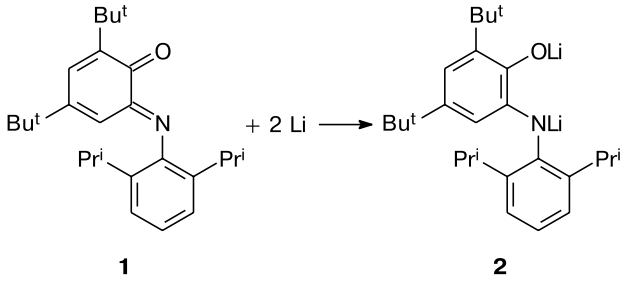
Scheme 2

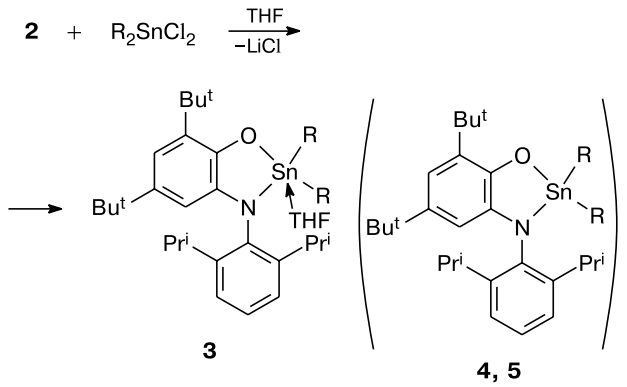


1 **2**

In the course of the reaction, the color of the mixture changes from red to dark-blue and then to pale-yellow, which is indicative of the reaction completion. After separation from excess metal, the solution of compound **2** can be used in subsequent syntheses.

The reaction of salt **2** with tin(IV) diorganohalides in THF proceeds already at −30 °C and is accompanied by the appearance of the intense yellow color (Scheme 3).

Scheme 3



3 **4, 5**

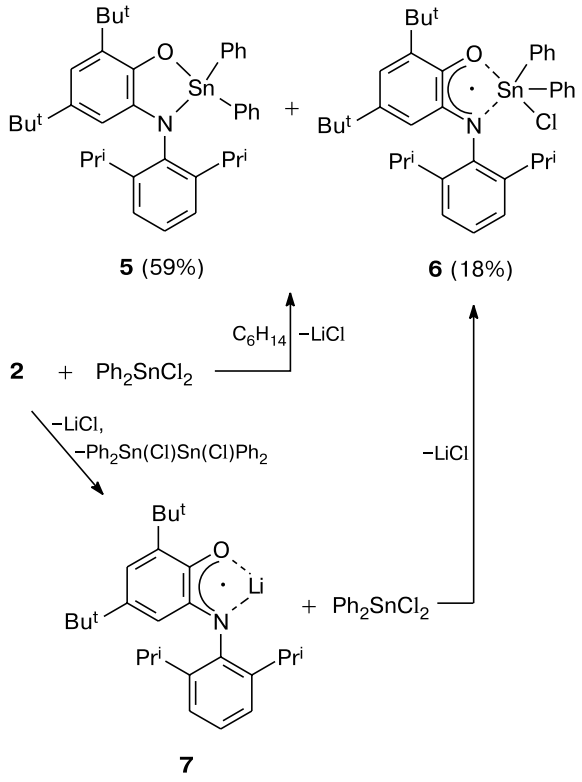
R = Et (**3**), Bu^t (**4**), Ph (**5**)

Compounds **3—5** were isolated in 60—80% yields and characterized by elemental analysis, IR spectroscopy, and ^{1}H NMR spectroscopy. The molecular structure of complex **3** was established by X-ray diffraction. Tin *o*-amidophenolate complexes **3—5** are sensitive to atmospheric oxygen and moisture both in solution and the crystalline state.

It should be noted that the amidophenolate ligand rather efficiently shields the coordination sphere of the tin atom. As a result, only complex **3** contains the coordinated THF molecule. The presence of the *tert*-butyl or phenyl substituents at the tin atom allows the isolation of four-coordinate metal derivatives.

The reaction (see Scheme 3) can also be performed in a hydrocarbon solution. However, in this case the redox process resulting in oxidation of the amidophenolate ligands to *o*-iminobenzosemiquinone ligands occurs. For R = Ph, this leads to the isolation of paramagnetic complex **6** in ~18% yield based on starting compound **2** (Scheme 4). In a separate experiment, we found that complex **5** is not oxidized by diphenyltin dichloride in hexane. Hence, oxidation of complex **2** with diphenyltin dichloride seems to be the most probable reaction. This reaction produces 1,2-dichloro-1,1,2,2-tetraphenyldistannane[10] and lithium *o*-iminosemiquinolate **7**. The subsequent exchange reaction involving this compound affords complex **6**.

Scheme 4



5 (59%) **6** (18%)

7

Tin(IV) diorganohalides are known[11] to be efficiently solvated by *O*-donor ligands to form five- and six-coordinate complexes. The donation of the additional electron density to the tin atom leads to an increase in the polarity of the Sn—Cl bond and, as a consequence, to an increase in the rate of the heterolytic process, *viz.*, the exchange reaction.[12] All the above results for THF, where the starting organotin compound undergoes solvation, show that the reaction is not accompanied by oxidation. To the contrary, a nonpolar solvent facilitates the homolytic mechanism of the reaction[12] and, consequently, leads to an increase in the redox reaction rate.

Complex **6** is resistant to atmospheric oxygen and moisture both in the crystalline state and in solution for several hours. This complex is green colored and has a well-resolved ESR spectrum (Fig. 1). The spectrum is a

Fig. 1. Isotropic ESR spectrum of complex **6** (toluene, $T = 290$ K).

triplet (1 : 1 : 1) of doublets (1 : 1) due to interactions between the unpaired electron and the ^{14}N (99.63%, $I = 1$, $\mu_N = 0.4037$) and ^{1}H (99.98%, $I = 1/2$, $\mu_N = 2.7928$) nuclei[13] with satellite splitting on the magnetic tin isotopes ^{117}Sn (7.68%, $I = 1/2$, $\mu_N = 1.000$) and ^{119}Sn (8.58%, $I = 1/2$, $\mu_N = 1.046$)[13] characterized by the following parameters: $g_i = 2.0027$, $A_i(^1H) = 0.45$ mT, $A_i(^{14}N) = 0.56$ mT, $A_i(^{117,119}Sn) = 3.77$ mT. The hyperfine coupling constants for the nitrogen atom and the proton are similar to the corresponding values typical for *o*-iminoquinone **1** radical anion,[14] whereas the hyperfine coupling constants for the magnetic tin isotopes are substantially higher than the known values for $SQSnPh_2R$ [15] (SQ is the 3,6-di(*tert*-butyl)-*o*-benzoquinone radical anion). At the same time, paramagnetic diazabutadiene tin(II) complexes are characterized by substantially higher hyperfine coupling constants (12—16 mT).[16,17] The absence of splitting on the magnetic chlorine isotopes in the ESR spectrum of complex **6** indicates that the position of the Sn—Cl bond with respect to the plane of the paramagnetic ligand is nearly coplanar. This is also true for the crystalline state of complex **6**, as has been evidenced by the X-ray diffraction data for this compound.

The molecular structures of complexes **3** and **6** were established by X-ray diffraction. The crystallographic parameters and the X-ray diffraction data collection and refinement statistics are given in Table 1. Selected bond lengths and bond angles for complexes **3** and **6** are listed in Tables 2 and 3, respectively.

The tin atom in complexes **3** (Fig. 2) and **6** (Fig. 3) has a distorted trigonal-bipyramidal coordination environment. The symmetry plane in complex **6** passes through the *o*-iminoquinone fragment and the tin and halogen atoms. The equatorial plane is formed by the carbon atoms of the substituents (C(27) and C(29) in **3**; C(20) and C(20A) in **6**) and the N(1) atom of the *o*-iminoquinone ligand. The axial positions are occupied by the O(1) and O(1s) atoms in **3** and by the O(1) and Cl(1) atoms in **6**. The Sn(1) atom in complexes **3** and **6** deviates from the plane of the base of the bipyramid by 0.183 and 0.153 Å, respectively. According to the redox state of the *o*-iminoquinone ligand, the distributions of the bond lengths in the chelate metallocycle are substantially different. Complex **3** is characterized by longer C—O and C—N bond lengths compared to those in compound **6** (1.364(4) and 1.404(4) Å, respectively, in **3**; 1.298(4) and 1.334(4) Å in **6**). By contrast, the Sn—O and Sn—N bonds are substantially shorted in the *o*-amidophenolate derivative (2.047(2) and 2.044(3) Å, respectively, in **3**; 2.188(2) and 2.150(3) Å in **6**). The six-membered C(1)—C(6) ring in molecule **6** is characterized by a quinoid-type distortion, which is characteristic of *o*-semiquinones[18] and *o*-iminosemiquinones[1—7,19] and is manifested in alternation of the C—C bond lengths. The C(1)—C(2), C(1)—C(6), and C(5)—C(6) distances are 1.437(4), 1.460(4), and 1.402(4) Å, respectively. The C(2)—C(3) and C(4)—C(5) bonds are shorter (1.357(5) and 1.365(4) Å, respectively), whereas the C(3)—C(4) bond is longer (1.431(5) Å). To the contrary, the corresponding C—C bonds in molecule **3** are equalized and are close to 1.40 Å. It should be noted that the donor-acceptor interaction between the tin atom and the THF molecule in complex **3** is characterized by a rather long Sn(1)—O(1s) distance (2.617(3) Å), which is not typical of this type of interactions in five-coordinate tin(IV) complexes.[15,20] Evidently, this difference is attributed to a substantial steric hindrance in the coordination

Table 1. Crystallographic parameters and the X-ray diffraction data collection and refinement statistics for complexes **3** and **6**

Parameter	**3**	**6**
Molecular formula	$C_{34}H_{55}NO_2Sn$	$C_{38}H_{47}ClNOSn$
Molecular weight	628.48	687.91
T/K	100(2)	100(2)
Crystal system	Monoclinic	Orthorhombic
Space group	$P2(1)/n$	*Pnma*
a/Å	10.1211(6)	21.4041(11)
b/Å	14.7517(8)	14.8415(8)
c/Å	21.9797(12)	10.4797(6)
α/deg	90	90
β/deg	93.4680(10)	90
γ/deg	90	90
V/Å^3	3275.6(3)	3329.1(3)
Z	4	4
d_{calc}/g cm^{-3}	1.274	1.373
μ/mm^{-1}	0.808	0.878
F(000)	1328	1428
Crystal dimensions/mm	0.25×0.15×0.10	0.15×0.08×0.05
θ-Scan range/deg	1.86—26.00	1.90—26.01
Indices of measured reflections h, k, l	$-12 \le h \le 12$, $-18 \le k \le 10$, $-27 \le l \le 26$	$-26 \le h \le 26$, $-18 \le k \le 18$, $-12 \le l \le 10$
Number of observed reflections	18244	19095
Number of independent reflections	6430	3401
R_{int}	0.0277	0.0470
Goodness-of-fit on F^2	1.130	1.119
R_1/wR_2 ($I > 2\sigma(I)$)	0.0513/0.1221	0.0365/0.0747
R_1/wR_2 (based on all reflections)	0.0626/0.1265	0.0459/0.0778
Residual electron density (ρ_{max}/ρ_{min}) /e Å^{-3}	2.501/−0.912	0.765/−0.424

Table 2. Selected bond lengths (*d*) and bond angles (ω) in complex **3**

Bond	*d*/Å	Angle	ω/deg
Sn(1)—N(1)	2.044(3)	N(1)—Sn(1)—O(1)	81.52(9)
Sn(1)—O(1)	2.047(2)	N(1)—Sn(1)—C(27)	116.08(14)
Sn(1)—O(1S)	2.617(3)	O(1)—Sn(1)—C(27)	102.84(12)
Sn(1)—C(27)	2.131(4)	N(1)—Sn(1)—C(29)	116.58(13)
Sn(1)—C(29)	2.141(4)	O(1)—Sn(1)—C(29)	99.10(13)
O(1)—C(1)	1.364(4)	C(27)—Sn(1)—C(29)	125.08(15)
N(1)—C(2)	1.404(4)	O(1)—Sn(1)—O(1s)	170.21(17)
N(1)—C(15)	1.438(3)	C(1)—O(1)—Sn(1)	112.90(19)
C(1)—C(6)	1.398(4)	C(2)—N(1)—C(15)	118.3(2)
C(1)—C(2)	1.414(4)	C(2)—N(1)—Sn(1)	111.4(2)
C(2)—C(3)	1.399(4)	C(15)—N(1)—Sn(1)	130.18(17)
C(3)—C(4)	1.391(4)	C(28)—C(27)—Sn(1)	123.8(5)
C(4)—C(5)	1.387(4)	C(30)—C(29)—Sn(1)	115.8(3)
C(5)—C(6)	1.407(4)		

Table 3. Selected bond lengths (*d*) and bond angles (ω) in complex **6**

Bond	*d*/Å	Angle	ω/deg
Sn(1)—C(20)	2.141(2)	C(20)—Sn(1)—C(20A)	118.68(12)
Sn(1)—N(1)	2.150(3)	C(20)—Sn(1)—N(1)	119.91(6)
Sn(1)—O(1)	2.188(2)	C(20)—Sn(1)—O(1)	91.75(7)
Sn(1)—Cl(1)	2.4476(8)	N(1)—Sn(1)—O(1)	74.47(9)
O(1)—C(1)	1.298(4)	C(20)—Sn(1)—Cl(1)	96.76(7)
N(1)—C(6)	1.334(4)	N(1)—Sn(1)—Cl(1)	88.75(7)
N(1)—C(13)	1.436(4)	O(1)—Sn(1)—Cl(1)	163.22(6)
C(1)—C(2)	1.437(4)	C(1)—O(1)—Sn(1)	116.04(19)
C(1)—C(6)	1.460(4)	C(6)—N(1)—C(13)	121.1(3)
C(2)—C(3)	1.357(5)	C(6)—N(1)—Sn(1)	117.0(2)
C(3)—C(4)	1.431(5)	C(13)—N(1)—Sn(1)	121.93(19)
C(4)—C(5)	1.365(4)		
C(5)—C(6)	1.416(4)		

sphere of tin due to the presence of the *o*-amidophenolate ligand. The shortest distance between the C(1s) atom of the THF molecule and the C(15) atom of the *o*-amidophenolate ligand is comparable with the sum of the van der Waals radii of these atoms (3.366(5) Å). As demonstrated above, the introduction of bulkier (*tert*-butyl or phenyl) hydrocarbon substituents at the tin atom facilitates the displacement of the tetrahydrofuran ligand from the coordination sphere of tin to form four-coordinate *o*-amidophenolate derivatives **4** and **5**.

To summarize, we found that four- and five-coordinate tin(IV) *o*-amidophenolate complexes can be synthesized by the exchange reactions of tin diorganohalides with the dilithium derivative of 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-iminobenzoquinone in THF. In hexane, this reaction is accompanied by the redox process as a side reaction. In the case of the reaction of lithium *o*-amidophenolate with diphenyltin dichloride in hexane,

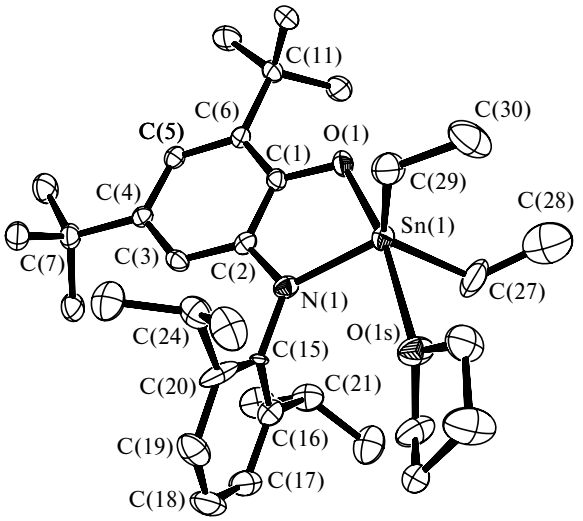


Fig. 2. Molecular structure of complex **3**. Atoms are represented by displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are omitted.

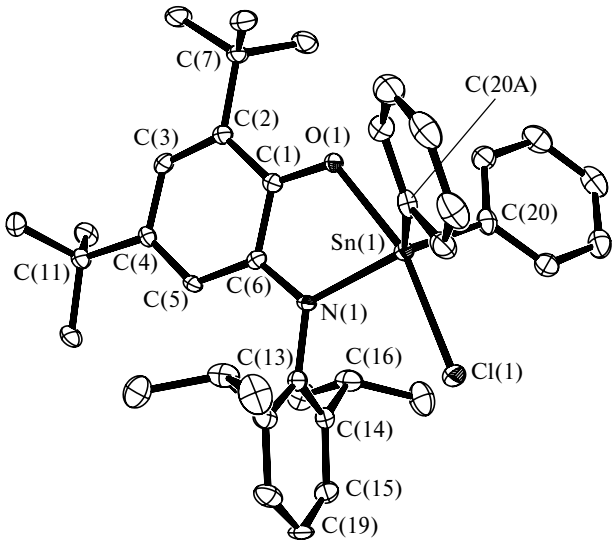


Fig. 3. Molecular structure of complex **6**. Atoms are represented by displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are omitted.

a stable *o*-iminosemiquinone-containing tin(IV) complex was isolated.

Experimental

All experiments associated with the synthesis of tin(IV) *o*-iminoquinone complexes were performed under air- and moisture-excluding conditions. The IR spectra were recorded on a FSM-1201 Fourier-transform spectrometer in Nujol mulls in KBr cells. The ESR spectra were measured on a Bruker ER 200 D-SRC spectrometer equipped with an ER 4105 DR double resonator and an ER 4111 VT temperature controller.

The ^{1}H NMR spectra were recorded on a Bruker DPX-200 Fourier-transform NMR spectrometer. The solvents were purified and dried according to recommendations given in the study.[21] *o*-Iminoquinone **1** [22] and organotin compounds R_2SnCl_2 (R = Et, Bu^t, and Ph)[23] were synthesized according to known procedures.

Lithium 4,6-di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-amidophenolate (2). A suspension of *o*-iminoquinone **1** (1 g, 2.6 mmol) in hexane (30 mL) was vigorously stirred with an excess of granular lithium (0.18 g, 26.0 mol) at ~20 °C until a pale-yellow solution was obtained. Complex **2** precipitated from the solution due to poor solubility in cold hexane. The reaction mixture was separated from the unconsumed metal on a Schott glass filter No. 1. The filter was washed with hot hexane (3×20 mL) until complex **2** was completely dissolved. The combined hexane fractions were concentrated to 20 mL and cooled to ~20 °C. After cooling, the product again precipitated from the solution as a white finely crystalline compound. Analytically pure complex **2** was obtained in a yield of 0.93 g (90%), m.p. 185 °C. Found (%): C, 79.08; H, 9.40; Li, 3.71. $C_{26}H_{37}Li_2NO$. Calculated (%): C, 79.37; H, 9.48; Li, 3.53. ^{1}H NMR (200 MHz, $CDCl_3$, 20 °C), δ: 0.65, 0.84, and 0.92 (all d, 3 H each, CH_3 (Pr^i), J = 6.7 Hz); 1.03 (s, 9 H, Bu^t); 1.08 (d, 3 H, CH_3 (Pr^i), J = 6.7 Hz); 1.26 (s, 9 H, Bu^t); 2.96 (sept, 2 H, CH (Pr^i), J = 6.7 Hz); 6.24 and 6.96 (both d, 1 H each, H arom., J = 2.4 Hz); 7.03 (m, 3 H, H arom.). IR, ν/cm^{-1}: 1585, 1422, 1405, 1359, 1302, 1266, 1211, 1182, 1123, 1035, 970, 927, 875, 858, 841, 795, 764, 739, 695, 655, 627, 530, 462, 436.

Synthesis of complexes 3—5. A solution of complex **2** (1.03 g, 2.6 mmol) in THF was added to a solution of an equimolar amount of R_2SnCl_2 (0.64 g for R = Et, 0.79 g for R = Bu^t, or 0.89 g for R = Ph) in THF (10 mL) at −30 °C. The reaction mixture was slowly warmed to 20 °C and kept at this temperature for 1 h. The solvent was removed under reduced pressure.

Complexes **3** and **4**. The residue was treated with hexane. The hexane solution was filtered from the precipitate of lithium chloride on a glass filter No. 4. The filtrate was concentrated until crystals formed and then cooled to −20 °C. The crystals of complexes **3** and **4** were separated by decantation. The mother liquor was concentrated to one-half of the initial volume, after which an additional portion of crystals was obtained.

Complex **5**. The residue was treated with dichloromethane and filtered from the precipitate of lithium chloride on a glass filter No. 4. An equal volume of hexane was added to the resulting solution, and the solution was slowly concentrated until crystals formed and then cooled to −20 °C. The crystals of complex **5** were separated by decantation.

[4,6-Di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-amidophenolato]diethyltin(IV) tetrahydrofuranate (3). Analytically pure complex **3** was obtained in a yield of 1.01 g (69%) as pale-yellow crystals, m.p. 160 °C (decomp.). Found (%): C, 65.12; H, 8.97; Sn, 18.77. $C_{34}H_{55}NO_2Sn$. Calculated (%): C, 64.97; H, 8.82; Sn, 18.89. ^{1}H NMR (200 MHz, $CDCl_3$, 20 °C), δ: 1.17 and 1.19 (both d, 6 H each, CH_3 (Pr^i), J = 6.8 Hz); 1.26 (t, 6 H, CH_3 ($SnCH_2CH_3$), J = 7.5 Hz); 1.31 (s, 9 H, Bu^t); 1.47 (m, 4 H, β-CH_2 (THF)); 1.68 (q, 4 H, CH_2 ($SnCH_2CH_3$), J = 7.5 Hz, $J_{H,^{119}Sn}$ = 106.4 Hz); 1.92 (s, 9 H, Bu^t); 3.31 (sept, 2 H, CH (Pr^i), J = 6.8 Hz); 3.60 (m, 4 H, α-CH_2 (THF)); 6.52 (d, 1 H, H arom., J = 2.3 Hz); 7.23 (br.s, 3 H, H arom.); 7.34 (d, 1 H, H arom., J = 2.3 Hz). IR, ν/cm^{-1}: 1563, 1441, 1414, 1362, 1331, 1279, 1240, 1203, 1121, 1102, 1031, 984, 924, 906, 872, 855, 843, 821, 800, 770, 758, 716, 670, 583, 545, 522, 495, 428.

[4,6-Di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-amidophenolato]di-*tert*-butyltin(IV) (4). Analytically pure complex **4** was obtained in a yield of 0.98 g (62%) as yellow-brown crystals, m.p. 183 °C. Found (%): C, 66.78; H, 9.13; Sn, 19.12. $C_{34}H_{55}NOSn$. Calculated (%): C, 66.67; H, 9.05; Sn, 19.38. ^{1}H NMR (200 MHz, C_6D_6, 20 °C), δ: 1.16 (d, 6 H, CH_3 (Pr^i), J = 6.8 Hz); 1.17 (s, 18 H, Bu^t—Sn, $J_{H,^{119}Sn}$ = 94.30 Hz); 1.19 (d, 6 H, CH_3 (Pr^i), J = 6.8 Hz); 1.24 and 1.80 (both s, 9 H each, Bu^t); 3.39 (sept, 2 H, CH (Pr^i), J = 6.8 Hz); 6.30 and 6.97 (both d, 1 H each, H arom., J = 2.2 Hz); 7.13 (m, 3 H, H arom.). IR, ν/cm^{-1}: 1564, 1442, 1411, 1377, 1365, 1316, 1273, 1250, 1235, 1205, 1159, 1118, 1106, 1051, 1015, 977, 938, 922, 900, 849, 816, 800, 762, 682, 652, 588, 551, 523, 503.

[4,6-Di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-amidophenolato]diphenyltin(IV) (5). Analytically pure complex **5** was obtained in a yield of 1.40 g (83%) as yellow finely crystalline compound, m.p. 218 °C (decomp.). Found (%): C, 70.12; H, 7.34; Sn, 19.12. $C_{38}H_{47}NOSn$. Calculated (%): C, 69.95; H, 7.26; Sn, 18.19. ^{1}H NMR (200 MHz, $CDCl_3$, 20 °C), δ: 0.51 and 0.97 (both d, 6 H each, CH_3 (Pr^i), J = 6.8 Hz); 1.14 and 1.59 (both s, 9 H each, Bu^t); 3.17 (sept, 2 H, CH (Pr^i), J = 6.8 Hz); 5.87 and 6.64 (both d, 1 H each, H arom., J = 2.3 Hz); 7.13 (m, 3 H, H arom.); 7.47 (m, 6 H, Ph—Sn); 7.71 (m, 4 H, Ph—Sn). IR, ν/cm^{-1}: 1567, 1443, 1431, 1411, 1359, 1323, 1281, 1256, 1231, 1212, 1158, 1120, 1072, 995, 985, 933, 924, 904, 862, 847, 817, 798, 770, 728, 670, 654, 553, 444.

Reaction of complex 2 with Ph_2SnCl_2 in hexane. A suspension of complex **2** (1.03 g, 2.6 mmol) in hexane (30 mL) was added to a solution of Ph_2SnCl_2 (0.89 g, 2.6 mmol) in hexane (10 mL) at −30 °C. The reaction mixture was slowly warmed to 20 °C; at the same time, the color of the solution changed from yellow to intense green. The reaction mixture was stirred at this temperature for 1 h. The liquid phase was filtered on a Schott glass filter No. 4. After recrystallization from acetone, complex **6** was isolated from the filtrate in a yield of 0.32 g (0.46 mmol, 18%). The residue was washed on a filter with dichloromethane. Complex **5** was isolated from the solution according to the above-described procedure in a yield of 1.01 g (1.55 mmol, 59%).

[4,6-Di(*tert*-butyl)-*N*-(2,6-diisopropylphenyl)-*o*-iminosemiquinone]diphenylchlorotin(IV) (6). Green crystals, m.p. 233 °C. Found (%): C, 66.44; H, 6.93; Cl, 5.20; Sn, 17.34. $C_{38}H_{47}ClNOSn$. Calculated (%): C, 66.34; H, 6.89; Cl, 5.15; Sn, 17.26. IR, ν/cm^{-1}: 1582, 1435, 1358, 1332, 1318, 1254, 1199, 1192, 1167, 1109, 1098, 1068, 1054, 1029, 995, 935, 915, 888, 877, 865, 821, 798, 766, 735, 698, 665, 650, 630, 612, 528, 498, 451, 437.

X-ray diffraction study. Crystals of complexes **3** and **6** suitable for X-ray diffraction study were grown from hexane and acetone, respectively. X-ray data were collected on a Smart Apex diffractometer (Mo-Kα radiation, graphite monochromator). The structures of complexes **3** and **6** were solved by direct methods followed by the refinement by the full-matrix least-squares method against F^2 with the use of the SHELXTL program package.[24] Absorption corrections were applied using the SADABS program.[25] All nonhydrogen atoms were refined anisotropically. The hydrogen atoms in complex **3** were positioned geometrically and refined using a riding model. The positions of

the hydrogen atoms in complex **6** were located in difference electron density maps and refined isotropically.

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