

New dioxygen-inert triphenylantimony(v) catecholate complexes based on *o*-quinones with electron-withdrawing groups*

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New triphenylantimony(v) catecholate complexes were synthesized by oxidative addition of sterically hindered *o*-benzoquinones containing electron-withdrawing substituents in different positions of the carbon ring to triphenylantimony. The complexes were characterized using IR spectroscopy, NMR spectroscopy, and cyclic voltammetry. The oxygen-inertness of the complexes is shown by NMR spectroscopy and electrochemical studies. The introduction of electron-withdrawing substituents to the catecholate ligand shifts the first oxidation potential of the complexes to the electropositive region and thus deactivates the triphenylantimony(v) catecholate complexes in the reaction with molecular oxygen.

Key words: antimony(v), *o*-benzoquinone, catecholate, NMR spectroscopy, cyclic voltammetry.

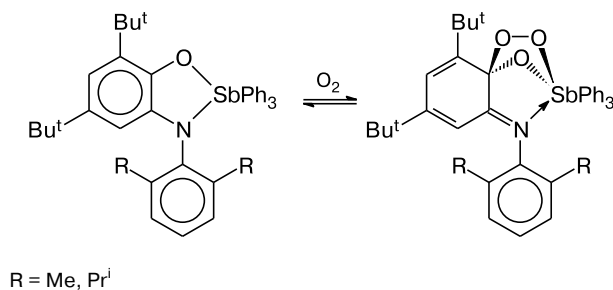
The chemistry of transition and nontransition metal complexes with sterically hindered *o*-semiquinone and catecholate ligands is one of the actively developing directions of modern organoelement chemistry.

The main results in this area of chemistry were obtained for the transition metal compounds.^{1–4} Much less information is available about molecular structures and physical and chemical properties of individual non-transition metal compounds^{5–8} compared to those of transition metals. This is valid for the antimony derivatives. Only few scanty data on the individual catecholate^{9–12} and *o*-amidophenolate^{13,14} organic antimony derivatives have been known to the recent time, whereas no data on their sterically hindered analogs were available. The first examples of the sterically hindered catecholate and *o*-amidophenolate triphenylantimony(v) complexes are described in Refs 15–19.

The reversible binding of molecular oxygen by the nontransition metal complexes has first been observed when *o*-iminobenzoquinone was used as the ligand in the triphenylantimony(v) complex. It was found that the antimony *o*-amidophenolate complexes can reversibly add and eliminate molecular oxygen under the mild conditions (Scheme 1).^{17,19}

* Dedicated to Academician I. I. Moiseev on his 80th birthday.

Scheme 1

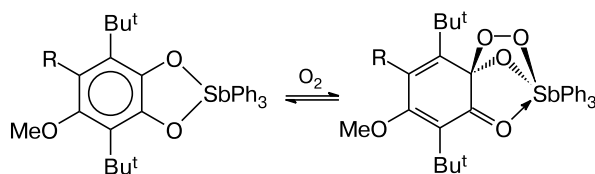


Contrary to this fact, the antimony catecholate complexes based on 3,6-di-*tert*-butyl-*o*-benzoquinone and related to *o*-aminophenolate complexes do not react with molecular oxygen under similar conditions.¹⁵

The mechanism proposed¹⁷ for the process assumes that one of the key steps is the one-electron oxidation of the dianionic ligand to the radical-anion one. The redox potential of the transformation of catecholate into semiquinolate (*o*-aminophenolate into *o*-iminobenzosemiquinolate) should play a critical role, allowing or forbidding the process itself to occur. Antimony complexes **1** and **2** with 4-methoxy-3,6-di-*tert*-butyl- and 4,5-dimethoxy-3,6-di-*tert*-butylcatecholate ligands,¹⁸ whose redox potentials are lower than those of 3,6-di-*tert*-

butylcatecholate,¹⁵ were synthesized and their reactivity was studied in the reaction with molecular oxygen. These complexes were shown to be able to add molecular oxygen with the formation of cyclic endoperoxide complexes also containing the five-membered trioxastibolane cycle (Scheme 2).

Scheme 2



R = H, OMe

The deactivation of the triphenylantimony catecholate complexes in the reaction with molecular oxygen should be expected when the redox potential of the catecholate complexes is shifted to the electropositive potential region. To confirm this fact, in the present work we have synthesized the triphenylantimony catecholate complexes containing the electron-withdrawing substituents in the ring of the catecholate ligand and studied them by electrochemical methods.

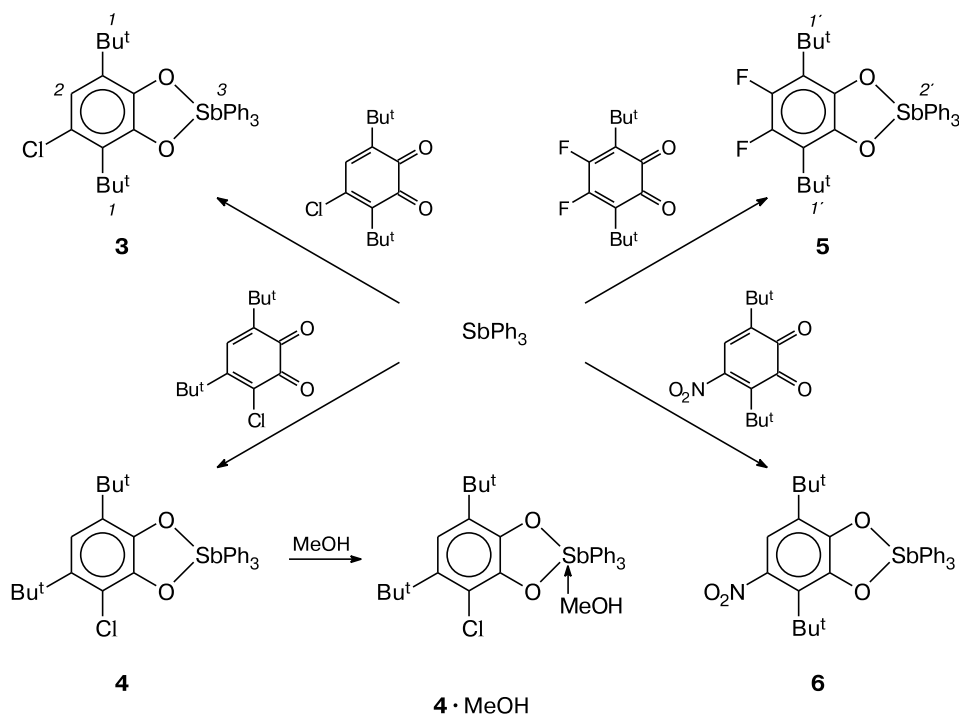
Results and Discussion

Five substituted catecholate complexes of triphenylantimony(v) were synthesized: (3,6-di-*tert*-butyl-4-chlorocatecholate)triphenylantimony(v) (**3**), (3,5-di-*tert*-butyl-6-chlorocatecholate)triphenylantimony(v) (**4**), (3,5-di-*tert*-butyl-6-chlorocatecholate)(methanol)triphenylantimony(v) (**4** · MeOH), (3,6-di-*tert*-butyl-4,5-difluorocatecholate)triphenylantimony(v) (**5**), and (3,6-di-*tert*-butyl-4-nitrocatecholate)triphenylantimony(v) (**6**).

The triphenylantimony complexes were synthesized by oxidative addition to the corresponding *o*-quinones at room temperature (Scheme 3). Compounds **3**–**6** and **4** · MeOH were characterized by the data of IR spectroscopy, ¹H NMR spectroscopy, elemental analysis, and electrochemical studies.

The IR spectra of compounds **3**–**6** and **4** · MeOH contain a set of bands in the region 1100–1300 cm^{−1}, which is characteristic of catecholate dianions, and also bands of vibrations of the functional groups of the complexes. Particularly, skeletal vibrations of the SbPh₃ fragment lie in the region from 680 to 750 cm^{−1}, stretching vibrations of the Sb–O bonds are observed at 580–650 cm^{−1}, and stretching vibrations of the Sb–C_{Ph} lie at 430–480 cm^{−1}. The vibration band of the C–F bond in complex **5** appears at 964 cm^{−1}, being very

Scheme 3



intense. The stretching vibrations of the nitro group in complex **6** manifest themselves as bands at 1515 and 1354 cm^{-1} .

The ^1H NMR spectra of the compounds synthesized were measured at room temperature in a CDCl_3 solution. Tetramethylsilane was used as standard. The ^1H NMR spectra confirm the proposed structures of the synthesized complexes. The ^1H NMR spectra of chloro- and difluoro-containing catecholates **3** and **5**, respectively, are shown in Fig. 1. Since catecholate **3** is unsymmetric, the protons of the *tert*-butyl groups are nonequivalent and appear in the NMR spectrum as two singlets, whereas the NMR spectrum of symmetric difluoro-containing catecholate **5** contains one singlet from two equivalent *tert*-butyl groups. The ^1H NMR spectra of catecholates **4**, **4**•MeOH, and **6** are analogous to the ^1H NMR spectrum of complex **3** and, hence, are not presented. The ^1H NMR spectrum of complex **4**•MeOH additionally exhibits a singlet with $\delta = 3.49$ from protons of the methyl group and a broad singlet at $\delta = 0.93$ from the proton of the hydroxy group of methanol coordinated to the antimony atom.

Catecholate complexes **3**, **4**, and **4**•MeOH containing chlorine in positions 4 or 6 of the phenyl ring of the catecholate ligand are inert toward air oxygen in both the solid state and solution. A similar situation is observed for complexes **5** (contains fluorine atoms in positions 4 and 5 of the phenyl ring of the catecholate ligand) and **6** (con-

Table 1. Electrochemical oxidation potentials of the triphenyl-antimony(v) catecholate complexes according to the CV method

Compound	I		II	
	$E_{1/2}^1/\text{V}$	I_c/I_a	E_{p2}^2/V	I_c/I_a
1	0.70	0.90	1.14 ^a	—
7	0.89	0.82	1.40 ^b	0.50
3	0.98	0.81	1.37 ^b	0.60
4	1.00	0.52	1.60 ^b	0.50
4 •MeOH	1.07	0.56	1.60 ^a	—
6	1.16	0.66	1.62 ^a	—

Note: GC electrode, CH_2Cl_2 , $V = 0.2 \text{ V s}^{-1}$, $0.1 \text{ M NBU}_4\text{ClO}_4$, $C = 3 \cdot 10^{-3} \text{ mol L}^{-1}$, Ar, vs Ag/AgCl/KCl (sat.).

I and II are the first and second anodic stages; $E_{1/2}^1$ is the half-wave potential of the first anodic process; I_c/I_a is the ratio of currents of the inverse cathodic and direct anodic peaks; E_p^2 is the potential of the second oxidation peak; the number of electrons of the first anodic stage relative to ferrocene as standard is $n = 1$.

^a Irreversible peak.

^b Quasi-reversible oxidation process.

tains the nitro group in position 4 of the phenyl ring of the catecholate ligand).

The electrochemical properties of synthesized antimony complexes **3**, **4**, **4**•MeOH, and **6** were studied by cyclic voltammetry (CV). The complexes are oxidized at the glassy-carbon electrode in dichloromethane in two stages (Table 1). For complex **3** and 3,6-di-*tert*-butylcatecholate)triphenylantimony (**7**), the first redox process is quasi-reversible one-electron (Scheme 4, Fig. 2). The reversibility coefficients show that the radical-anion form of the coordinated ligand that formed is rather stable. However, in the both cases, the inverse branch of the CV curve contains peaks of the fragmentation products of the cationic complexes ($E_{p1} = 0.03 \text{ V}$; $E_{p2} = -0.38 \text{ V}$), and

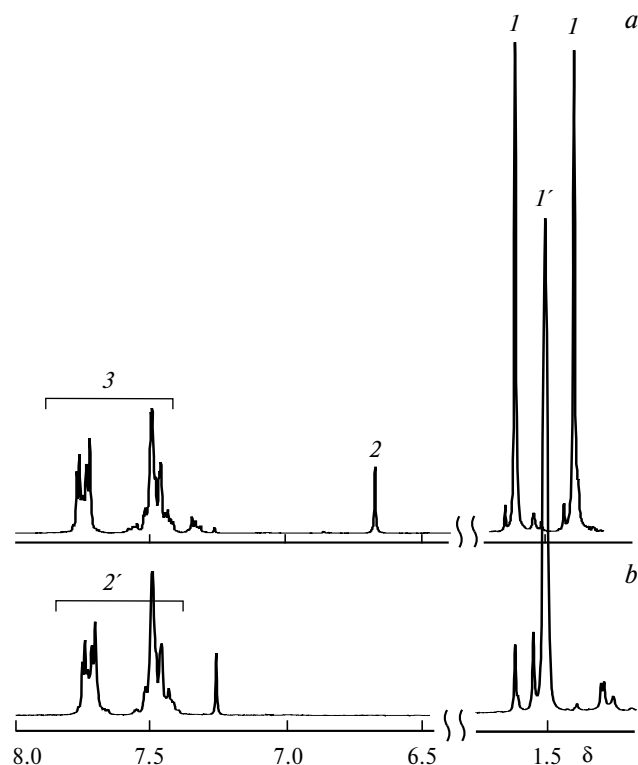


Fig. 1. ^1H NMR spectra of catecholates **3** (a) and **5** (b) (CDCl_3 , $\sim 20^\circ\text{C}$). The groups are designated in Scheme 1.

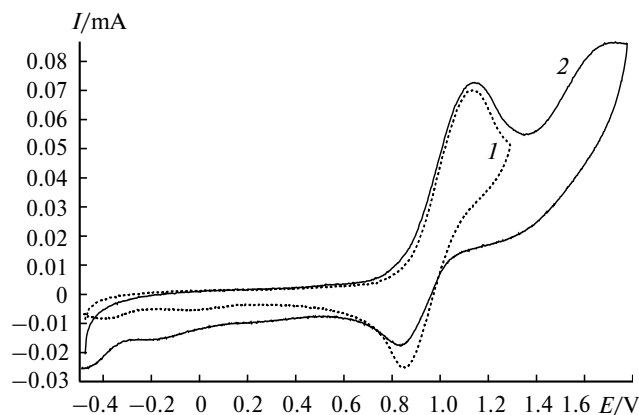
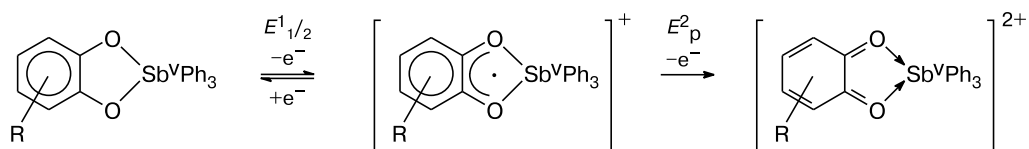


Fig. 2. Cyclic voltammograms of the oxidation of complex **3** (CH_2Cl_2 , GC anode, Ag/AgCl/KCl, $0.1 \text{ M NBU}_4\text{ClO}_4$, $C = 3 \cdot 10^{-3} \text{ mol L}^{-1}$, argon): **1**, potential sweep to 1.3 V; **2**, potential sweep to 1.78 V.

Scheme 4



the second cathodic process corresponds to the reduction of the free *o*-quinoid ligand. The extension of the potential sweep range to the anodic region allows one to detect the second irreversible oxidation process corresponding to the transition of the semiquinone form of the ligand into the quinoid one with the formation of the dicationic complex.

The values of the cathodic peaks of the products of complex decomposition increase, indicating the decoordination of neutral *o*-quinone. The acceptor character of the chlorine atom affects the value of only the first redox transition, while the second anodic peaks are almost identical for the both complexes.

The change in the mutual arrangement of substituents (chlorine atom and *tert*-butyl groups) in the carbon ring of complex **4** (with respect to **3**) exerts no considerable effect on the first oxidation potential. However, the I_c/I_a values (ratio of currents of the inverse cathodic and anodic peaks) show that the stability of the intermediates formed decreases (see Table 1). Complex **4** is also characterized by the appearance of the decomposition products during backward scan. The reversibility factor for the hexacoordinated analog of **4**•MeOH, which contains solvated methanol in the coordination sphere of the antimony atom, at the first oxidation stage (Fig. 3) differs insignificantly from the data obtained for pentacoor-

ordinated complex **4**. Coordination of a methanol molecule shifts the half-wave oxidation potential of complex **4**•MeOH compared to that of **4** to the anodic region by 0.07 V. The second anodic peaks for the both compounds are similar. The observed effects of shifting the oxidation potentials suggests that the redox properties of these compounds can be controlled due to coordination of solvent molecules of different nature.

For complex **6** containing the nitro group, according to the acceptor influence of the latter, the oxidation potentials are shifted to the anodic region (see Table 1). Rather stable cationic complex is formed upon the primary oxidation process (Fig. 4), and no decomposition products are observed in the inverse branch of the CV curve. The stability of this complex can be due to the ability of the nitro group to participate in the delocalization of the unpaired electron of the *o*-benzosemiquinone form of the oxidized complex (Scheme 5).

Scheme 5

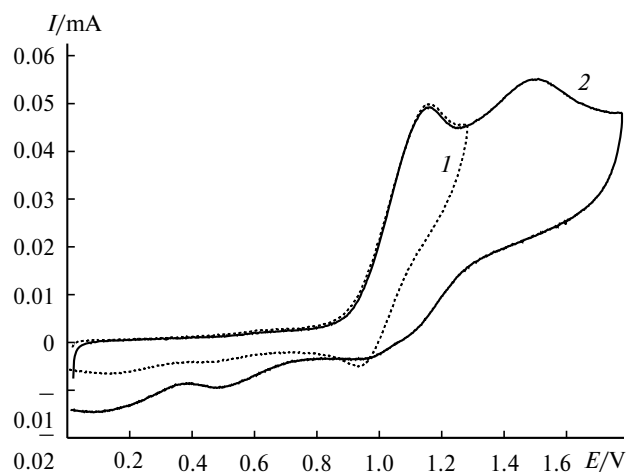
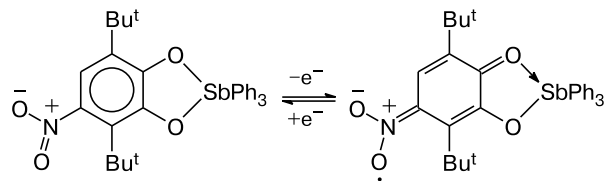


Fig. 3. Cyclic voltammograms of the oxidation of complex **4**•MeOH (CH_2Cl_2 , GC anode, Ag/AgCl/KCl, 0.1 M NBu_4ClO_4 , $C = 3 \cdot 10^{-3} \text{ mol L}^{-1}$, argon): 1, potential sweep to 1.3 V; 2, potential sweep to 1.78 V.

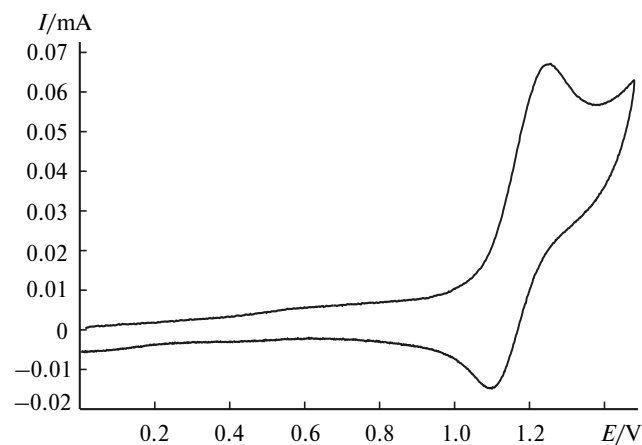


Fig. 4. Cyclic voltammogram of the oxidation of complex **6** (CH_2Cl_2 , GC anode, Ag/AgCl/KCl, 0.1 M NBu_4ClO_4 , $C = 3 \cdot 10^{-3} \text{ mol L}^{-1}$, argon).

It has previously¹⁸ been found that (4-methoxy-3,6-di-*tert*-butylcatecholate)triphenylantimony (**1**) can reversibly bind molecular oxygen. The voltammetric data show that the introduction of an electron-donor substituent considerably decreases the potential of the first anodic process, which assumes the reaction with molecular oxygen to occur. The complex is electrochemically oxidized in one-electron reversible stage to form the stable cation (see Table 1). The voltammogram contains no secondary peaks of fragmentation product reduction. The second redox process is irreversible.

Thus, compounds **3**, **4**, **4**•MeOH, and **6** undergo two-stage electrochemical oxidation. The first electron transfer is the quasi-reversible one-electron process, due to which the catecholate ligand is transformed into the *o*-semi-quinone form. The subsequent oxidation of the complexes is irreversible and results in the decoordination of neutral *o*-quinone. The electrochemical potentials obtained for the compounds with electron-withdrawing substituents differ considerably from a threshold value of +0.7 V typical of (4-methoxy-3,6-di-*tert*-butylcatecholate)triphenylantimony(v).

Experimental

All experiments on synthesis and investigation of the complexes were carried out in evacuated ampules without oxygen and water. In all cases, the yields of the target products were higher than 90%. The solvents used were purified and dehydrated according to standard procedures.²⁰ *o*-Benzoquinones, viz., 3,6-di-*tert*-butyl-4-chloro-*o*-benzoquinone, 3,5-di-*tert*-butyl-6-chloro-*o*-benzoquinone, 3,6-di-*tert*-butyl-4,5-difluoro-*o*-benzoquinone, and 3,6-di-*tert*-butyl-4-nitro-*o*-benzoquinone, were synthesized earlier at the Laboratory of Chemistry of Organoelemental Compounds of the G. A. Razuvaev Institute of Organometallic Chemistry of the Russian Academy of Sciences.

IR spectra were recorded on an FSM 1201 FTIR spectrometer in Nujol. ¹H NMR spectra were measured on a Bruker AVANCE DPX-200 instrument using tetramethylsilane as internal standard and CDCl₃ as solvent. The oxidation potentials were measured by cyclic voltammetry in a three-electrode cell with an IPC-pro potentiostat in argon. The working electrode was a stationary glassy-carbon (GC) electrode with a diameter of 2 mm, and a platinum wire (*S* = 18 mm²) served as the auxiliary electrode. The reference electrode (Ag/AgCl/KCl) was equipped with a water-proof membrane. The potential sweep rate was 0.2 V s⁻¹. The supporting electrolyte 0.1 M Bu₄NClO₄ (99%, Acros) was two times recrystallized from aqueous EtOH and dried *in vacuo* (48 h at 50 °C). Dichloromethane was dried and purified by known procedures.²¹ The concentration of the antimony complexes was 0.003 mol L⁻¹.

(3,6-Di-*tert*-butyl-4-chlorocatecholate)triphenylantimony(v) (3). A solution of 3,6-di-*tert*-butyl-4-chloro-*o*-benzoquinone (0.255 g, 1 mmol) in toluene was added with stirring to a solution of triphenylantimony (0.353 g, 1 mmol) in toluene. The color of the solution changed from green to orange. After hexane was added, a finely dispersed light yellow precipitate of product **3** was formed, filtered off, and dried *in vacuo*. M.p. 160–164 °C

(with decomp.). Found (%): C, 63.36; H, 5.97; Sb, 18.91; Cl, 5.87. C₃₂H₃₄ClO₂Sb. Calculated (%): C, 63.23; H, 5.64; Sb, 20.03; Cl, 5.83. IR (Nujol), v/cm⁻¹: 1478 w, 1432 w, 1387 m, 1367 m, 1332 w, 1294 m, 1244 s, 1202 w, 1076 m, 1069 m, 1056 m, 1025 w, 996 m, 985 s, 950 s, 854 w, 843 m, 813 m, 766 s, 736 s, 693 s, 673 w, 637 w, 615 w, 598 w, 481 w, 469 m, 449 s. ¹H NMR (CDCl₃), δ: 1.39 and 1.61 (both s, 9 H each, Bu^t), 6.67 (s, 1 H, C₆H), 7.40–7.53 (m, 9 H, SbPh₃), 7.70–7.79 (m, 6 H, SbPh₃).

(3,5-Di-*tert*-butyl-6-chlorocatecholate)triphenylantimony(v) (4). Complex **4** was synthesized by the reaction of triphenylantimony (0.251 g, 0.71 mmol) and 3,5-di-*tert*-butyl-6-chloro-*o*-benzoquinone (0.181 g, 0.71 mmol) using a method similar to that for complex **3**. The complex isolated from hexane represents light yellow fine crystals. M.p. 145–147 °C (decomposes at *t* > 180 °C). Found (%): C, 63.52; H, 5.49; Sb, 19.20; Cl, 5.74. C₃₂H₃₄ClO₂Sb. Calculated (%): C, 63.23; H, 5.64; Sb, 20.03; Cl, 5.83. IR (Nujol), v/cm⁻¹: 1478 w, 1437 w, 1416 m, 1360 m, 1310 w, 1270 m, 1258 m, 1243 m, 1178 w, 1157 w, 1070 m, 1024 w, 990 s, 955 m, 871 s, 856 w, 757 w, 738 s, 730 s, 692 s, 642 w, 634 w, 615 w, 593 w, 453 s. ¹H NMR (CDCl₃), δ: 1.42 and 1.46 (both s, 9 H each, Bu^t), 6.73 (s, 1 H, C₆H), 7.40–7.56 (m, 9 H, SbPh₃), 7.74–7.88 (m, 6 H, SbPh₃).

Solvate with methanol (3,5-di-*tert*-butyl-6-chlorocatecholate)triphenylantimony(v), (4•MeOH). A finely crystalline sample of complex **4**•MeOH was isolated by the recrystallization of complex **4** from methanol. M.p. 122–126 °C. Found (%): C, 61.46; H, 5.63; Sb, 18.70; Cl, 6.06. C₃₃H₃₈ClO₂Sb. Calculated (%): C, 61.94; H, 5.99; Sb, 19.03; Cl, 5.54. IR (Nujol), v/cm⁻¹: 3330 s, 1480 w, 1432 s, 1409 s, 1359 s, 1312 m, 1279 w, 1262 s, 1243 s, 1184 m, 1174 w, 1158 w, 1110 w, 1076 m, 1064 m, 1024 w, 997 s, 985 s, 953 s, 868 s, 858 w, 754 m, 738 s, 730 s, 695 s, 660 m, 634 w, 618 w, 588 w, 456 s. ¹H NMR (CDCl₃), δ: 0.93 (br.s, 1 H, OH) 1.42 and 1.46 (both s, 9 H each, Bu^t), 3.49 (s, 3 H, CH₃ of methanol), 6.73 (s, 1 H, C₆H), 7.40–7.60 (m, 9 H, SbPh₃), 7.76–7.86 (m, 6 H, SbPh₃).

(3,6-Di-*tert*-butyl-4,5-difluorocatecholate)triphenylantimony(v) (5). The complex was synthesized by the reaction of triphenylantimony (0.353 g, 1.0 mmol) and 3,6-di-*tert*-butyl-4,5-difluoro-*o*-benzoquinone (0.256 g, 1.0 mmol) using a method similar to that for the synthesis of complex **3**. The complex isolated from toluene represents yellow-orange crystals. Found (%): C, 63.20; H, 5.70; Sb, 19.67. C₃₂H₃₃F₂O₂Sb. Calculated (%): C, 63.07; H, 5.46; Sb, 19.98. IR (Nujol), v/cm⁻¹: 1477 s, 1432 s, 1410 s, 1357 s, 1333 w, 1305 w, 1280 s, 1246 m, 1203 m, 1181 w, 1158 w, 1075 m, 1070 m, 1059 m, 1046 s, 1023 w, 997 m, 964 s, 929 w, 891 m, 849 w, 800 w, 773 w, 737 s, 732 s, 692 s, 673 w, 666 w, 656 w, 624 m, 582 w, 523 m, 447 s. ¹H NMR (CDCl₃), δ: 1.50 (s, 18 H, 2 Bu^t), 7.40–7.56 (m, 9 H, Ph), 7.68–7.76 (m, 6 H, Ph).

(3,6-Di-*tert*-butyl-4-nitrocatecholate)triphenylantimony(v) (6). Complex **6** was synthesized by the reaction of triphenylantimony (0.296 g, 0.837 mmol) and 3,6-di-*tert*-butyl-4-nitro-*o*-benzoquinone (0.222 g, 0.837 mmol) using a method analogous to that for the synthesis complex **3**. The complex isolated from hexane represents fine yellow crystals. IR (Nujol), v/cm⁻¹: 1515 m, 1438 m, 1371 m, 1354 s, 1255 s, 1217 m, 1160 w, 1070 w, 1059 m, 1038 w, 984 s, 890 w, 855 w, 812 m, 788 w, 774 w, 737 s, 790 m, 619 w, 529 w, 456 m, 447 s. ¹H NMR (CDCl₃), δ: 1.38 and 1.47 (both s, 9 H each, 2 Bu^t), 6.66 (s, 1 H, arom. C₆H), 7.40–7.60 (m, 9 H, Ph), 7.66–7.82 (m, 6 H, Ph).

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