New polydentate ligands based on sterically hindered *o*-benzoquinones (pyrocatechols) containing the 1,4-diazadiene group

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New bifunctional ligands containing the 1,4-diazadiene and 1,2-benzoquinone groups were synthesized by the reactions of 4-amino-3,6-di-*tert*-butylpyrocatechol with glyoxal and its derivatives. Their *o*-semiquinone complexes with transition and nontransition metals were studied in solution by the ESR method.

Key words: 1,4-diaza-1,3-diene, sterically hindered quinones and pyrocatechols, NMR spectroscopy, ESR spectroscopy.

1,4-Diazadienes form a large class of organic compounds used in coordination and organometallic chemistry as neutral ligands.[1—4] In recent three decades, sterically hindered *o*-quinones and pyrocatechols found wide use in the chemistry of *o*-semiquinone and catecholate metal complexes.[5—7] The *o*-quinone ligand in the *o*-semiquinone or catecholate form is valence-bonded in mixed-ligand metal complexes due to high differences in the values of reduction potentials of diazadienes and *o*-quinones.[8,9] To date there are no published data on bifunctional ligands containing simultaneously the diazadiene and *o*-quinone groups. In our opinion, these ligands are of doubtless interest for the chemistry of organometallic and coordination compounds, such as bridging ligands for the synthesis of bi- and polynuclear coordination compounds containing radical ligands along with magnetic metallocenters. In addition, they possess at least two electron-withdrawing groups and, hence, can act as "electron reservoirs."

The purpose of the present work is the synthesis of new bifunctional sterically hindered pyrocatechols and *o*-quinones with the 1,4-diazadiene group.

Results and Discussion

Diazadienes based on sterically hindered anilines and substituted diketones are stable and preparatively available compounds.[10] Diazadienes containing pyrocatechol substituents were synthesized by the reactions of 4-amino-3,6-di-*tert*-butylpyrocatechol (**1**) with α-iminoketone (**2**), glyoxal, and its substituted derivatives (Scheme 1).

Synthesized pyrocatecholodiazadienes **3**—**6** are yellow crystalline substances soluble in organic solvents and slowly oxidized in air. The compositions of compounds **3**—**6** were confirmed by the elemental analysis data, and their structures were determined by spectral methods (IR, NMR, and ESR).

The IR spectra of compounds **3**—**6** contain the characteristic absorption bands of stretching vibrations of the imine groups at 1640 and 1620 cm^{-1}, the broad bands corresponding to stretching vibrations of the O—H bond are observed in the region 3150—3300 cm^{-1}, and the line shape and the values of ν(OH) frequencies indicate the formation of intra- and intermolecular hydrogen bonds.

The ^1H NMR spectra of these compounds are also similar. They exhibit signals of the protons of the *tert*-butyl groups (δ 1.38—1.62), the protons of the diazadiene moiety of the molecule (δ 2.01—2.12 for R = Me, 8.07—8.13 ppm for R = H), the aromatic proton of the pyrocatechol fragment, and the protons of the hydroxyl groups (δ 5.27—6.40).

To obtain the corresponding quinone derivatives, compounds **3—6** were oxidized by lead dioxide in ether. However, bifunctional derivatives **7** and **8** were isolated in the individual state only in the case of compounds **5** and **6** (Scheme 2).

The IR spectra of compounds **7** and **8** contain the characteristic bands typical of *o*-quinones (1680—1690 cm^{-1}) and corresponding to stretching vibrations of the carbonyl groups and absorption bands (1640 cm^{-1}) of stretching vibrations of the C=N bond. The ^1H NMR spectra of these compounds are similar and completely correspond to the proposed structure.

The oxidation of compounds **3** and **4** under milder conditions with air oxygen gave dark blue crystalline products, which were identified by the data of elemental analysis, IR, NMR spectroscopy (^{13}C, DEPT), and correlation procedures (CHCOR, COSY) as substituted indolo[3,2-*b*]-indolediones (**9**, **10**) (Scheme 3). The oxidation of pyro-

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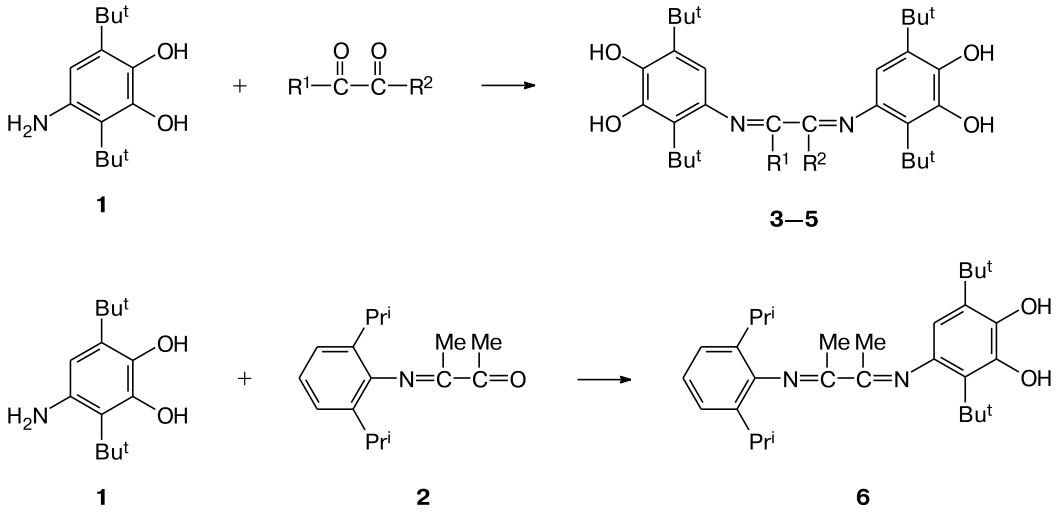
Scheme 1



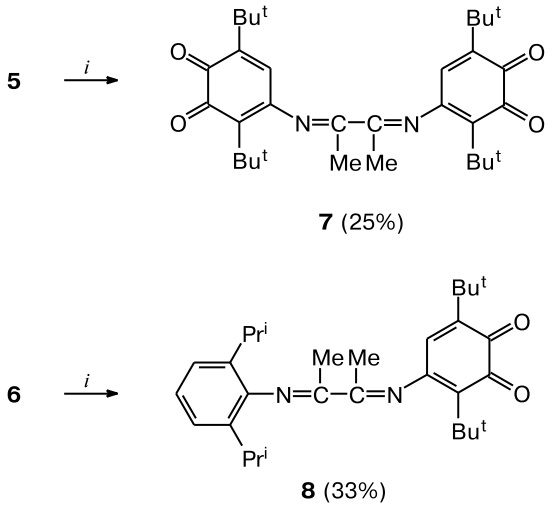
$R^1 = R^2 = H$ (**3**); $R^1 = H$, $R^2 = Me$ (**4**); $R^1 = R^2 = Me$ (**5**)

catechols **3** and **4** by lead dioxide produces a mixture of colored products, which we failed to separate and identify.

The IR spectrum of compound **9** in the region 3700—3000 cm^{-1} exhibit absorption bands at 3550 cm^{-1} (ν_{O-H}) and 3290 cm^{-1} (ν_{N-H}). In addition, the spectrum contains intense absorption bands (1610, 1585, 1565, and 1555 cm^{-1}) corresponding to the stretching vibrations of the C=O, C=N, C—O, and C—N bonds. The NMR spectra of compounds **9** and **10** differ from both the spectra of the corresponding pyrocatechols **3** and **4** and the spectra of quinones **7** and **8**. The most substantial changes are obtained in the region 5.0—8.2 ppm: the signals from the aromatic protons of the pyrocatechol fragment and the protons at the imine bond disappeared.

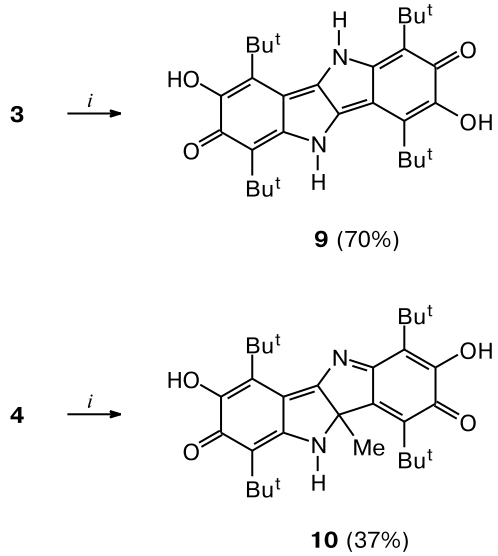
The ^1H NMR spectrum of compound **10** exhibits signals of four *tert*-butyl and one methyl groups, as well as broadened signals from the protons of two nonequivalent OH groups and one NH group. The data of the ^{13}C NMR and DEPT spectra indicate the absence of CH groups in a molecule of compound **10**. Similar pattern is also observed for compound **9** with the distinction that the structure of this compound is symmetric, which is reflected in the NMR spectra. Thus, the oxidation of compounds **3** and **4**

Scheme 2



i. PbO_2, Et_2O

Scheme 3



i. Air, MeOH.

Table 1. ESR spectral parameters of the radical anions of compounds **7** and **8** (THF, potassium mirror, 290 K)

Radical anion	g_i	a_H (1 H)	a_N (1 N)	a_H (4 H, $C\underline{H}_3$)
		mT		
$[\mathbf{7a}]^-$	2.0041	0.340	0.15	0.065
$[\mathbf{7a}]^{3-}$	2.0040	0.350	0.18	0.045
$\mathbf{8a}^-$	2.0046	0.347	0.16	0.047

containing the proton at the carbon atom of the imine bond leads to ring closure to form the indolo[3,2-*b*]indoledione derivatives.

The presence of the *o*-quinone fragments in compounds **7** and **8** causes their ability to form radical anions of *o*-semiquinones upon the one-electron reduction by alkaline metals.[11] The parameters of the ESR spectra of the paramagnetic derivatives obtained by the reduction of solutions of quinones **7** and **8** with metallic potassium are given in Table 1.

The diquinone nature of compound **7** is manifested by ESR monitoring of the course of its chemical reduction (Scheme 4). The reduction of **7** by metallic potassium in THF first generates paramagnetic radical monoanion $[\mathbf{7a}]^-$ (Fig. 1, spectrum *1*). The subsequent reduction of $[\mathbf{7a}]^-$ affords diamagnetic dianion $[\mathbf{7a}]^{2-}$, which exists as a conjugated bis-*p*-iminoquinone structure. Radical trianion

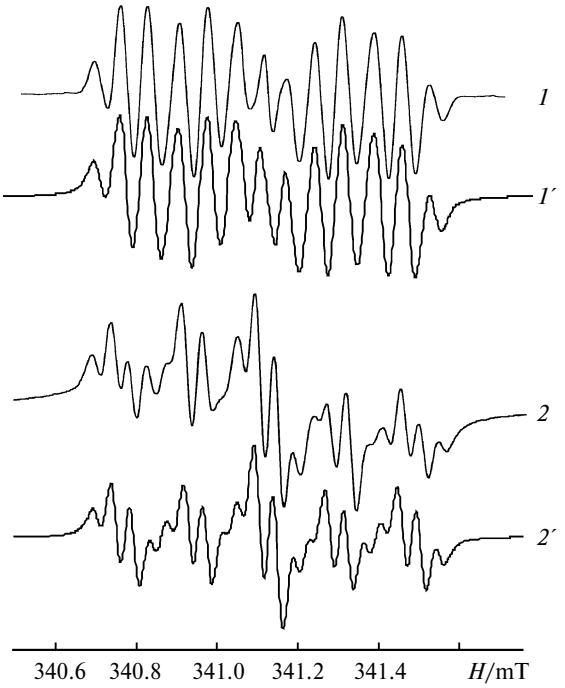
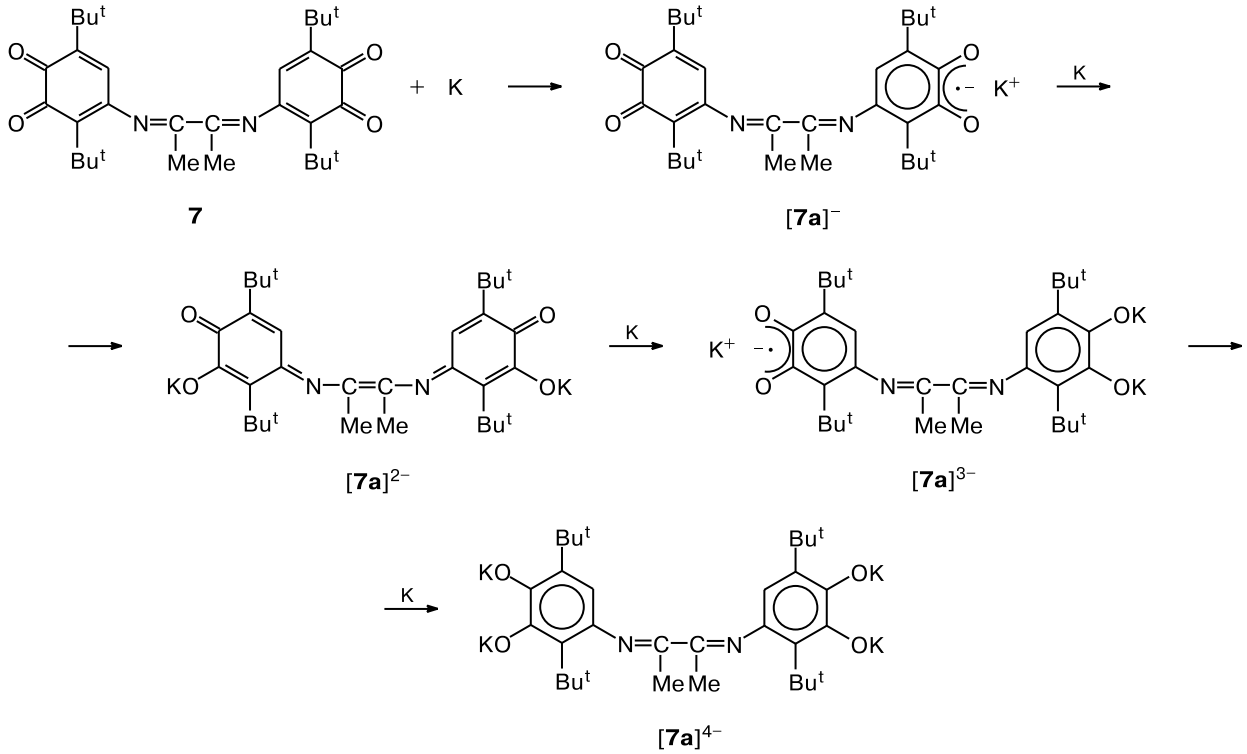


Fig. 1. ESR spectra of radical anions $[\mathbf{7a}]^-$ (*1*, *1′*) and $[\mathbf{7a}]^{3-}$ (*2*, *2′*): *1* and *2*, experimental spectra (THF, 290 K); *1′* and *2′*, simulated spectra.

$[\mathbf{7a}]^{3-}$ formed upon the deeper reduction is also paramagnetic, and its ESR spectrum (Fig. 1, spectrum *2*) differs

Scheme 4



from that of $[\mathbf{7a}]^-$, indicating a substantial difference in the spin density distribution in mono- and trianionic radicals of diquinone **7**. The further reduction of $[\mathbf{7a}]^{3-}$ results in the formation of diamagnetic tetraanion $[\mathbf{7a}]^{4-}$ and, correspondingly, the disappearance of the ESR signal.

Similarly to other sterically hindered *o*-quinones,[12] the photolytic interaction of compounds **7** and **8** with $Mn_2(CO)_{10}$ in solution affords the *o*-semiquinone derivatives, whose ESR spectral parameters are listed in Table 2 (Scheme 5).

Scheme 5

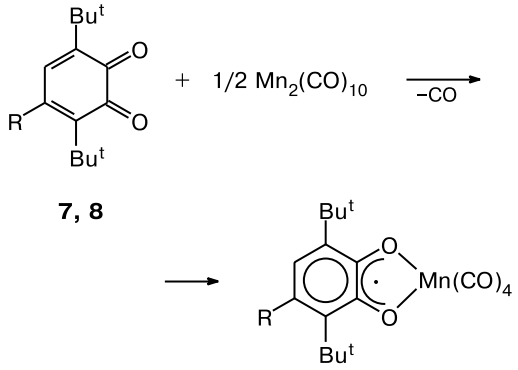
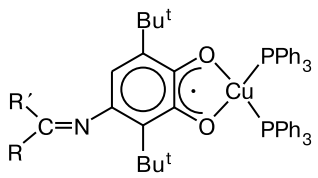


Table 2. ESR spectral parameters of the *o*-semiquinone complexes formed upon the reactions of compounds **7** and **8** with decacarbonyldimanganese (toluene, 290 K)

Radical anion	g_i	a_H (1 H)	a_N (1 N)	a_H (4 H, CH_3)	a_{Mn} (^{55}Mn)
		mT			
$[\mathbf{7a}]^-$	2.0029	0.335	0.25	0.03	0.655
$[\mathbf{8a}]^-$	2.0031	0.350	0.28	—	0.640

The interaction of an unpaired electron with the protons of the methyl substituent is observed only in the case of compound **7**. Somewhat higher value of the hyperfine coupling (HFC) constant with the ^{14}N nucleus compared to that of a solvate-separated pair in the case of potassium is a consequence of chelate ring formation.

One of the characteristic properties of pyrocatechols is their ability to form chelate *o*-semiquinone complexes with transition metals.[13] The copper *o*-semiquinone complexes, which are derivatives of pyrocatechols **4**—**6**, were studied in solution by the ESR method. Ethereal solutions of the complexes were obtained by the reaction of the corresponding pyrocatechols with copper(II) chloride and triphenylphosphine in the presence of NaOH (see Ref. 14).



The hyperfine structure of the ESR complexes of all *o*-semiquinone derivatives of pyrocatechols **4**—**6** is caused by the HFC of an unpaired electron with the magnetic isotopes of copper (^{63}Cu, $I = 3/2$, 69.09% (see Ref. 15), $a_{Cu}(1\ ^{63}Cu) = 1.06$ mT and ^{65}Cu, $I = 3/2$, 30.91% (see Ref. 15), $a_{Cu}(1\ ^{65}Cu) = 1.13$ mT), two phosphorus atoms of the phosphine ligands (^{31}P, $I = 1/2$, 100%, $a_p(2\ ^{31}P) = 1.54$ mT), one aromatic proton of the semiquinone fragment ($a_H(1H) = 0.35$ mT), and one nitrogen atom of the diazadiene fragment (^{14}N, $I = 1$, 100%, $a_N(1N) = 0.21$ mT).

Experimental

All procedures were carried out in evacuated tubes in the absence of oxygen and water (except for the synthesis of compound **2**). Solvents used in the work were purified and dehydrated using standard procedures.[16]

Suspensions of the synthesized compound in Nujol were prepared for IR spectra recording. IR spectra were recorded on a Specord M-80 spectrometer. NMR spectra were measured on a Bruker DRX-200 instrument in $CDCl_3$ at 20 °C, and chemical shifts are given in ppm (^1H, 200 MHz and ^{13}C, 50 MHz) using $SiMe_4$ as an internal standard. ESR spectra were detected on a Bruker ER 200 D-SRC spectrometer equipped with an ER 4105 DR double resonator and an ER 4111 VT thermocontroller. When determining *g* factor, DPPH was used as a standard.

4-Amino-3,6-di-*tert*-butyl-*o*-benzoquinone was synthesized according to the known procedure.[17]

4-Amino-3,6-di-*tert*-butylbenzo-1,2-diol (1). Aqueous 50% hydrazine hydrate (0.42 g, 0.13 mol) was poured to a solution of 4-amino-3,6-di-*tert*-butyl-*o*-benzoquinone (3.1 g, 0.13 mol) in methanol (75 mL). The reaction mixture was periodically heated, and nitrogen evolved during the reaction was pumped out. After the solution was decolorized and gas evolution stopped, the solvent was changed by toluene. Reaction water was distilled off by refluxing the toluene solution. Then the remained solution was recondensed to a particular ampule. The dry residue was dissolved in methanol, and toluene was added (1 : 1). Milky white crystals participated after the solution was evaporated. The yield was 2.42 g (78%), m.p. 164 °C. Found (%): C, 70.80; H, 9.67. $C_{14}H_{23}NO_2$. Calculated (%): C, 70.88; H, 9.70. IR, ν/cm^{-1}: 3410, 3360 (O—H, N—H); 1110 (C—N). ^1H NMR, δ: 1.37, 1.56 (both s, 9 H each, Bu^t); 3.6 (s, 2 H, OH); 4.52 (s, 2 H, NH_2); 6.13 (s, H, Ar).

***N*-(2,6-Diisopropylphenyl)-3-iminobutan-2-one (2).** Diacetyl (4.6 mL, 52 mmol) and 3—5 drops of HCOOH were added dropwise to a solution of 2,6-diisopropylaniline (9.4 g, 52 mmol) in hexane (50 mL). The reaction was carried out in the presence of $MgSO_4$ to bind water. The reaction mixture was magnetically stirred for 12 h. After $MgSO_4$ was separated and the solvent was removed under reduced pressure, a oily yellow liquid was isolated in a yield of 8.6 g (75.3%). IR, ν/cm^{-1}: 1710 (C=O), 1645 (C=N). ^1H NMR, δ: 1.13, 1.14 (both d, 6 H each, $CH(CH_3)_2$, $J = 6.9$ Hz); 1.82, 2.59 (both s, 3 H each, CH_3); 2.72 (sept, 2 H, $CH(CH_3)_2$, $J = 6.9$ Hz); 7.06—7.20 (m, 3 H, H_{Ar}).

1,4-Bis(2,5-di-*tert*-butyl-3,4-dihydroxyphenyl)-1,4-diazabuta-1,3-diene (3). A solution of 40% aqueous glyoxal (0.24 mL, 2.1 mmol) in methanol (20 mL) was added to a solution of compound **1** (1 g, 4.2 mmol) in methanol (50 mL). The reaction

mixture was heated for 10 min in a water bath at 80 °C. A yellow compound **3** (0.27 g, 27%), m.p. 176 °C (with decomp.), precipitated from the solution on cooling to room temperature. Found (%): C, 72.83; H, 9.01. $C_{30}H_{44}N_2O_4$. Calculated (%): C, 72.58; H, 8.87. IR, ν/cm^{-1}: 3550 (O—H); 1640 (C=N); 1125 (C—N); 1235 (C—O). ^{1}H NMR, δ: 1.41, 1.60 (both s, 18 H each, Bu^t); 5.77, 5.86 (both s, 2 H each, OH); 6.35 (s, 2 H, H(5)); 8.13 (s, 2 H, HC=N).

1,4-Bis(2,5-di-*tert*-butyl-3,4-dihydroxyphenyl)-2-methyl-1,4-diazabuta-1,3-diene (4). A solution of 40% methylglyoxal (0.32 mL, 2.1 mmol) in methanol (25 mL) was poured to a solution of compound **1** (1 g, 4.2 mmol) in methanol (50 mL). The reaction mixture was heated for 1 h in a water bath at 80 °C, and the color of the solution changed from light yellow to red-orange. Dark yellow crystals of compound **4** (0.68 g, 68%), m.p. 168 °C (with decomp.), precipitated from the solution on cooling to room temperature. Found (%): C, 72.59; H, 9.14. $C_{31}H_{46}N_2O_4$. Calculated (%): C, 72.90; H, 9.02. IR, ν/cm^{-1}: 1640 (C=N), 1125 (C—N), 1235 (C—O), 3550 (O—H). ^{1}H NMR, δ: 1.38, 1.41, 1.46, 1.62 (all s, 9 H each, Bu^t); 2.12 (s, 3 H, CH_3); 5.25, 5.78, 5.86, 5.89 (all s, 1 H each, OH); 5.91, 6.40 (both s, 1 H each, H_{Ar}); 8.07 (s, 1 H, HC=N).

1,4-Bis(2,5-di-*tert*-butyl-3,4-dihydroxyphenyl)-2,3-dimethyl-1,4-diazabuta-1,3-diene (5). A solution of diacetyl (0.15 g, 1.7 mmol) in methanol (30 mL) was poured to a solution of compound **1** (0.83 g, 3.4 mmol) in methanol (50 mL). The reaction mixture was heated in a water bath for 40 h at 80 °C. Yellow crystalline compound **5** precipitated from the solution during the reaction. The yield was 0.56 g (67%), m.p. 185 °C (with decomp.). Found (%): C, 73.00; H, 8.98. $C_{32}H_{48}N_2O_4$. Calculated (%): C, 73.28; H, 9.16. IR, ν/cm^{-1}: 1620 (C=N), 1150 (C—N), 1205 (C—O), 3500 (O—H). ^{1}H NMR, δ: 1.39 (s, 18 H, Bu^t); 1.47 (s, 18 H, Bu^t); 2.12 (s, 6 H, CH_3); 5.27 (s, 2 H, H_{Ar}); 5.90 (s, 4 H, OH).

1-(2,6-Diisopropylphenyl)-4-(2,5-di-*tert*-butyl-3,4-dihydroxyphenyl)-2,3-dimethyl-1,4-diazabuta-1,3-diene (6). A solution of compound **2** (2.3 g, 9.4 mmol) in methanol (50 mL) was poured to a solution of compound **1** (1.6 g, 6.7 mmol) in methanol (50 mL). The reaction mixture was heated for 8 h in a water bath at 80 °C. Yellow crystals of compound **6**, m.p. 117 °C, precipitated on slow evaporation of the solution. The yield was 35.4%. Found (%): C, 77.97; H, 10.01. $C_{30}H_{44}N_2O_2$. Calculated (%): C, 77.58; H, 9.48. IR, ν/cm^{-1}: 1640 (C=N), 1120, 1055, 1025 (C—O, C—N); 3540, 3150 (O—H). ^{1}H NMR, δ: 1.16 (d, 12 H, $CH(C\underline{H}_3)_2$, J = 6.9 Hz); 1.41, 1.47 (both s, 9 H each, Bu^t); 2.01, 2.18 (both s, 3 H each, CH_3); 2.70 (sept, 1 H, $C\underline{H}(CH_3)_2$, J = 6.9 Hz); 5.24, 5.90 (both s, 1 H each, OH); 5.94 (s, 1 H, H_{Ar}(5)); 7.02—7.20 (m, 3 H, Ar).

1,4-Bis(2,5-di-*tert*-butylcyclohexadiene-1,5-dion-3,4-yl)-2,3-dimethyl-1,4-diazabuta-1,3-diene (7). A solution of compound **5** (0.55 g, 1.05 mmol) in diethyl ether (40 mL) was oxidized with an excess of PbO_2 in air. The reaction course was monitored by TLC (Silufol UV-254, hexane—ethyl acetate (100 : 1)). After the end of the reaction, lead oxides were filtered off, and the solution was concentrated. Dark red crystals of compound **7** (0.25 g, 25%) decomposing on heating above 100 °C were isolated on cooling. Found (%): C, 73.42; H, 8.65. $C_{32}H_{44}N_2O_4$. Calculated (%): C, 73.81; H, 8.52. IR, ν/cm^{-1}: 1640 (C=N); 1690 (C=O); 1125 (C—N). ^{1}H NMR, δ: 1.24, 1.26 (both s, 18 H each, Bu^t); 2.23 (s, 6 H, CH_3); 6.12 (s, 2 H, H_{Ar}).

1-(2,6-Diisopropylphenyl)-4-(2,5-di-*tert*-butylcyclohexadiene-1,5-dion-3,4-yl)-2,3-dimethyl-1,4-diazabuta-1,3-diene (8). The synthesis is analogous to the synthesis of compound **7**. Red crystals of compound **8** (0.12 g, 33%), which decompose at 117 °C, were isolated. Found (%): C, 77.81; H, 8.89. $C_{30}H_{42}N_2O_2$. Calculated (%): C, 77.92; H, 9.09. IR, ν/cm^{-1}: 1640 (C=N); 1690 (C=O); 1125 (C—N). ^{1}H NMR, δ: 1.16 (d, 12 H, $CH(C\underline{H}_3)_2$, J = 6.9 Hz); 1.27, 1.28 (both s, 9 H each, Bu^t); 1.97, 2.32 (both s, 3 H each, CH_3); 2.61 (sept, 2 H, $C\underline{H}(CH_3)_2$, J = 6.9 Hz); 6.22 (s, 1 H, H(5)); 7.07—7.21 (m, 3 H, Ar).

4,9-Dihydroxy-2,5,7,10-tetra-*tert*-butylindolo[3,2-*b*]indole-3,8-dione (9). Air was bubbled for 12 h through a methanolic solution containing compound **3** (1.19 g, 2.4 mmol). The color of the solution changed from light yellow to violet. The evaporation of the solution gave 0.83 g (69.7%) of blue-violet crystals of compound **9**, having no distinct melting temperature. Found (%): C, 73.01; H, 7.98. $C_{30}H_{40}N_2O_4$. Calculated (%): C, 73.17; H, 8.13. IR, ν/cm^{-1}: 3550 (O—H); 3290 (N—H); 1610 (C=O); 1585 (C=N). ^{1}H NMR, δ: 1.43, 1.47 (both s, 18 H each, CH_3); 7.33, 7.99 (both br.s, 2 H each, OH, NH). ^{13}C NMR, DEPT (200 MHz), δ: 30.7, 31.5 ($C(\underline{C}H_3)_3$); 34.6, 36.5 ($\underline{C}(CH_3)_3$); 107.5 (C); 118.3 (C); 121.3 (C); 143.2 (C); 144.8 (C); 154.6 (C); 180.2 (C=O).

3,9-Dihydroxy-5b-methyl-2,5,7,10-tetra-*tert*-butyl-indolo[3,2-*b*]indole-4,8-dione (10). The synthesis was analogous to that of compound **9**. Dark blue crystals without distinct melting temperature were obtained in a yield of 0.47 g (37%). Found (%): C, 73.45; H, 8.19. $C_{31}H_{42}N_2O_4$. Calculated (%): C, 73.51; H, 8.30. IR, ν/cm^{-1}: 3475—3190 (O—H, N—H); 1630 (C=O); 1570 (C=N). ^{1}H NMR, δ: 1.49, 1.50, 1.52, 1.63 (all s, 9 H each, CH_3); 1.73 (s, 3 H, CH_3); 5.93 (br.s, 1 H, NH); 8.08, 8.89 (both br.s, 1 H each, OH). ^{13}C NMR, DEPT (200 MHz), δ: 30.6, 30.90, 30.94, 30.95 ($C(\underline{C}H_3)_3$); 31.2 (CH_3); 35.1, 35.8, 35.9, 37.0 ($\underline{C}(CH_3)_3$); 74.4 (C(5b)); 114.3 (C), 117.8 (C), 117.9 (C), 125.4 (C), 139.0 (C), 145.7 (C), 147.8 (C), 150.0 (C), 162.0 (C), 172.7 (C); 172.8 (C); 179.0, 183.4 (C=O).

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