

# EPR Study of *o*-Semiquinone-Catecholate Cobalt Complexes with Bis(diphenylphosphanyl)ethane

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Three novel cobalt compounds with bis(diphenylphosphanyl)-ethane (dppe) and semiquinone and catecholate ligands were synthesized and investigated by EPR spectroscopy in solution. They are: [(dppe)Co(3,6-DBSQ)(Cat<sub>ox</sub><sup>Cl</sup>)] (**1**), [(dppe)Co(3,6-DBCat)(phen-SQ)] (**2**), and [(dppe)Co(3,6-DBSQ)<sub>2</sub>] (**3**), where 3,6-DBSQ and 3,6-DBCat are the radical anion and dianion of 3,6-di-*tert*-butyl-*o*-benzoquinone, respectively, Cat<sub>ox</sub><sup>Cl</sup> is the dianion of perchloroxanthrenequinone-2,3, and phen-SQ is the radical anion of phenanthrene-

quinone. According to the ratio of the redox potentials of the *o*-quinone fragments, **1** and **2** are mixed semiquinone-catecholate complexes, and the 3,6-di-*tert*-butyl-*o*-quinone fragment appears as a semiquinone in **1** and as a catecholate in **2**. Compound **3** exists as a mixture of redox isomers, namely semiquinone-catecholate and bis-semiquinone species.

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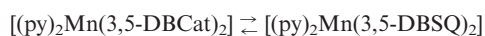
## Introduction

In 1980, Pierpont first observed a reversible intramolecular metal–ligand electron transfer of a cobalt bis-semiquinone complex in solution.<sup>[1]</sup>



3,5-SQ and 3,5-Cat are the anion-radical and dianion of 3,5-di-*tert*-butyl-*o*-benzoquinone.

A similar equilibrium involving a two-electron transfer was described later<sup>[2]</sup> for the manganese complex [(py)<sub>2</sub>Mn(3,5-DBCat)<sub>2</sub>].



Reversible electron transfer, taking place in the solid state, was first observed by our group<sup>[3,4]</sup> in 1990 and by Hendrickson<sup>[5]</sup> in 1993. Further development of the understanding of reversible metal–ligand electron transfer in semiquinone metal complexes was dedicated to investigation of solid-phase transformations.<sup>[6–14]</sup>

Number of mono-semiquinone complexes of Rh,<sup>[15–18]</sup> Ir,<sup>[19]</sup> Cu,<sup>[20]</sup> and Ni<sup>[21]</sup> of general formula [L<sub>n</sub>MSQ] demonstrate reversible electron transfer in solution (L<sub>n</sub> is a neutral ligand for Rh, Ir, and Cu, or monovalent one for Ni). It has been shown that complexes exist in solution as an equilibrium mixture of redox isomers that interconvert rapidly (according to the EPR time scale) between one another.

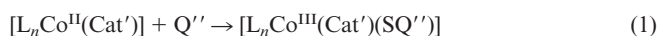
The equilibrium position and the rate of isomerization depend on the nature of the *o*-semiquinone, the neutral ligands, and the solvent. In all cases, decreasing the temperature shifts the equilibrium position towards the catecholate isomer.

All of the complexes [L<sub>n</sub>M(“Q”)<sub>2</sub>] that were investigated to date contain “Q” fragments that are derivatives of the same quinone and so their initial electronic ground state *a priori* is degenerate. The aim of this work was to obtain six-coordinate cobalt complexes containing different *o*-quinone fragments.

Most of publications listed above are dedicated to complexes containing nitrogen atom donors. We have not found in the literature any bis-quinone complexes with dppe. Moreover dppe contains two phosphorous atoms (<sup>31</sup>P, *I* = 1/2, 100%), the hyperfine splitting of which in the EPR spectrum provides information regarding the spin distribution in complex molecules.

## Results and Discussion

Cobalt compounds of types **1–3** can be obtained through oxidative addition reactions of the corresponding *o*-quinones to four-coordinate [(dppe)(3,6-di-*tert*-butylcatecholato)cobalt] [Equation (1)].

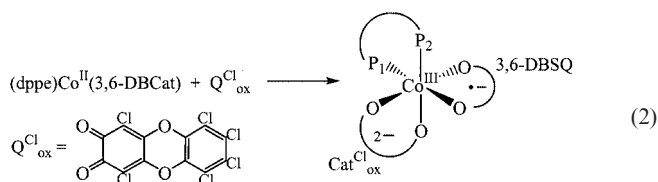


Stable paramagnetic adducts were studied by EPR spectroscopy in solution. The appearance of the EPR spectrum

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of a particular product depends on the redox potential ratio of the quinone fragments. We have observed three situations.

The first takes place when additional quinone is quite a stronger acceptor than is 3,6-DBQ, which has formed the initial catecholate. We have used perchlorooxanthrenequinone-2,3 [Equation (2)].



The presented EPR spectrum (Figure 1) displays one unpaired electron localized on the 3,6-DBSQ fragment:  $a_{\text{H1(SQ)}} = a_{\text{H2(SQ)}} = 0.30$  mT,  $g_i = 2.0016 \pm 0.0005$ . Perchlorooxanthrenequinone receives two electrons and becomes a catecholate dianion. The cobalt atom is oxidized from the oxidation state +2 to +3. The different hyperfine coupling constants on the  $^{31}\text{P}$  nuclei ( $a_{\text{P2}} = 1.30$  mT,  $a_{\text{P1}} = 0.30$  mT) reflect planar and apical positions of the corresponding nuclei with respect to the plane of the *o*-semiquinone.<sup>[22,23]</sup> The coupling constant on  $^{59}\text{Co}$  ( $a_{\text{Co}} = 1.00$  mT) are typical for octahedral *o*-semiquinone-catecholate complexes of cobalt(III) with one unpaired electron localized primarily (> 99% according to ref.<sup>[15]</sup>) on the semiquinone unit.<sup>[1]</sup> Thus, the result is a six-coordinate semiquinone-catecholate complex of low-spin cobalt(III).

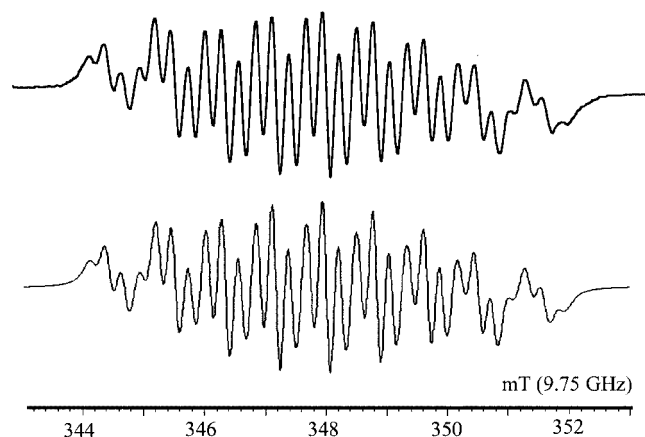
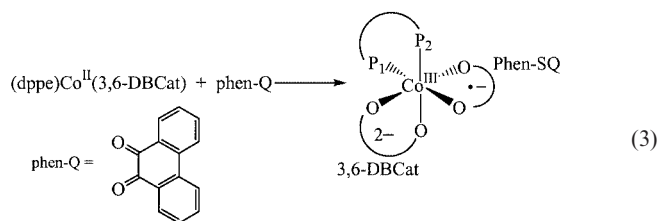


Figure 1. Room temperature x-band EPR spectrum (top) of  $[(\text{dppe})\text{Co}(3,6\text{-DBSQ})(\text{Cat}^{\text{Cl}}_{\text{ox}})]$  (1) (toluene, 290 K) and (bottom) its simulation (EPR line width = 0.2 mT)

The second situation occurs when the additional quinone is a quite-weaker acceptor than is quinone, which has formed the initial catecholate; e.g., when it is phenanthrenequinone [Equation (3)].



The hyperfine coupling with protons of 3,6-DBSQ is absent ( $g_{\text{iso}} = 2.0016 \pm 0.0005$ , Figure 2). Only hyperfine coupling is observed with  $^{59}\text{Co}$  and one  $^{31}\text{P}$  ( $a_{\text{Co}} \approx a_{\text{P2}} \approx 0.85$  mT). The interaction with the protons of phen-SQ and  $\text{P}_1$  is unresolved because of a relatively large line width. The spectrum view changes slightly with temperature. Optimal resolution was achieved at 250 K. An acceptable simulation indicates an essentially larger line width than was observed in the case 1 (0.20 mT for 1 and 0.73 mT for 2). Diminution of the  $^{59}\text{Co}$  and  $^{31}\text{P}$  coupling constants is expected. This feature is explained by lower acceptor properties of phen-Q relative to 3,6-DBQ.<sup>[24,25]</sup>

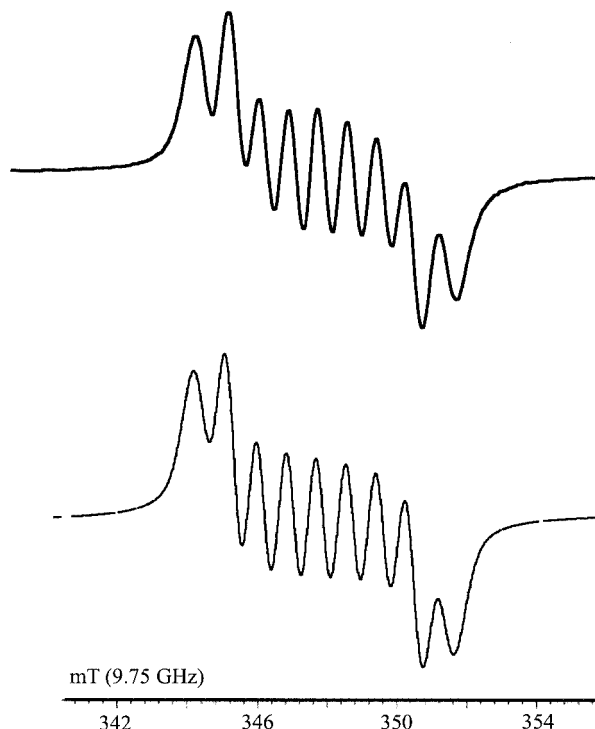
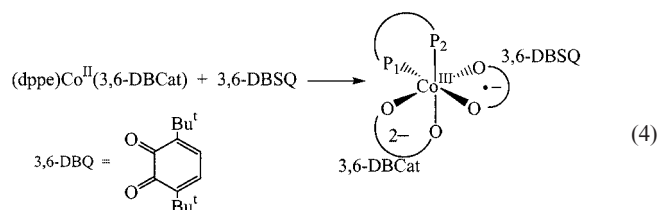


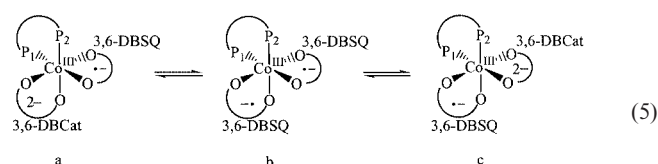
Figure 2. X-Band EPR spectrum (top) of  $[(\text{dppe})\text{Co}(3,6\text{-DBCat})(\text{phen-SQ})]$  (2) (toluene, 250 K) and (bottom) its simulation (EPR line width = 0.73 mT)

Thus, additional phenanthrenequinone accepts one electron and becomes a semiquinone, the cobalt(II) ion loses one electron and becomes a cobalt(III) ion, and the 3,6-DBSQ unit remains a catecholate. The resulting six-coordinate complex of cobalt(III) contains one unpaired electron localized on the phenanthrenesemiquinone and the 3,6-DBQ fragment in the catecholate form.

The third situation takes place when both the quinone fragments are the same [Equation (4)].



The EPR spectrum consists of one broad, unresolved line, the form and intensity of which depends on the temperature ( $g_{\text{iso}} = 2.0020 \pm 0.0005$ , Figure 3). No hyperfine structure is observed. A bearable simulation of the spectrum at 210 K indicates a line width of 5.0 mT. Based on literature data,<sup>[1,26]</sup> we propose that line broadening takes place because of the following equilibrium [Equation (5)]:



This equilibrium is a tautomeric interconversion of isomers **a** and **c** followed by a reversible electron transfer. It

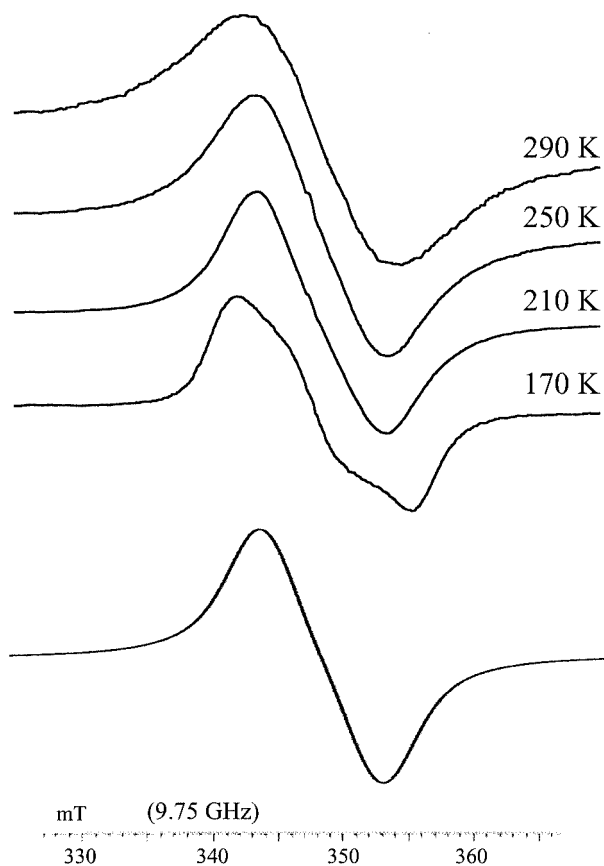


Figure 3. Temperature dependence of the x-band EPR spectrum (top) of  $[(dppe)Co(3,6\text{-DBCat})(3,6\text{-DBSQ})]$  (**3**) ( $g = 2.002 \pm 0.0005$ ) and (bottom) its simulation at  $T = 210$  K ( $a_{Co} = a_{P2} = 1.3$  mT; line width = 5.0 mT).

can occur though the intermediacy of a bis-semiquinone adduct of cobalt(II) (**b**). The presence of this intermediate containing at least three unpaired electrons could be the reason for the broadening and temperature dependence of the EPR spectrum.

One can imagine the EPR spectrum in the absence of this intermediate. In the case of slow interconversion (relative to the timescale of EPR spectroscopy) of **a** and **c**, the EPR spectrum should reflect, as was observed for **1**, a hyperfine interaction with the cobalt nucleus of two different phosphorus atoms (one planar and one apical with respect to the plane of the semiquinone unit) and two protons of one semiquinone ring.

In the case of fast exchange, the EPR spectrum would show an interaction of the unpaired electron with the cobalt nucleus, the two different phosphorus atoms with equal constants, and the four protons of 3,6-DBSQ with one half-constant ( $\approx 0.15$  mT).<sup>[27]</sup>

In the case of a medium rate of interconversion, i.e., when the lifetime of this intermediate is close to the characteristic time of EPR spectroscopy, an EPR spectrum presenting slow exchange phenomena will transform to depict fast exchange with an increase in temperature, according to known rules.<sup>[28]</sup> Only some components of the spectrum can undergo broadening.

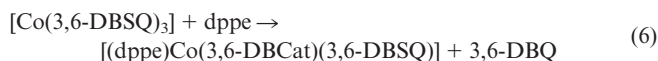
Total EPR spectroscopic line broadening can take place in the case where one of the exchanging particles has a very broad (maybe infinite) line width and/or a very short lifetime. The particles **a** and **c** presented above have equal lifetimes because of their total identity; the EPR spectra corresponding to fast and slow exchange between them have already been described. A complex of cobalt(II) with two semiquinone units (particle **b**) having at least three unpaired electrons should display a very broad line. Interconversion of **a** to **c** passing through form **b** leads to lines broadening. However, the molar fraction of this cobalt(II) adduct appears to be relatively low because it is possible to observe the spectrum of **3** and its  $g$  factor is the same as it is in the cases of **1** and **2**.

In a publication describing a similar situation for complex  $[(\alpha, \alpha'\text{-bpy})Rh(3,6\text{-DBCat})(3,6\text{-DBSQ})]$ , EPR spectroscopic line broadening occurs upon warming it in solution as well as in the solid state at a very small high-spin fraction that cannot be detected by magnetic susceptibility.<sup>[26]</sup>

A very small impurity of a  $Co^{II}$  species seems to be present in the cases of the complexes **1** and **2**. This impurity might be the reason for the relatively large line widths. By comparing the EPR spectroscopic line widths of complexes **1** and **2** with the values of  $E_{1/2}$  of the corresponding quinones [ $E_{1/2}(\text{phen-Q}) = -0.66$  V,  $E_{1/2}(3,6\text{-DBQ}) = -0.39$  V,  $E_{1/2}(Q_{ox}^{Cl}) = -0.03$  V] it becomes clear why the EPR spectrum of **2** is poorly resolved in comparison with **1**. The difference between the values of  $E_{1/2}$  of  $Q_{ox}^{Cl}$  and 3,6-DBQ ( $\Delta \approx 0.36$  V) is larger than between 3,6-DBQ and phen-Q ( $\Delta \approx 0.27$  V). Based on these general considerations it becomes clear that the energy separation between the  $Co^{III}(\text{Cat}')(SQ'')$  and  $Co^{II}(SQ')(SQ'')$  states is larger in the  $Q_{ox}^{Cl}$ –3,6-DBQ pair than in the 3,6-DBQ–phen-Q pair, and

so the amount of an impurity of a  $\text{Co}^{\text{II}}$  species is, respectively, smaller. As a consequence, the line widths in the EPR spectrum of **1** are smaller than they are in the case of **2**. Of course, these comments are qualitative and approximate because they are based on the assumption that the first values of  $E_{1/2}$  of quinones correlate with the second ones.

Complex **3** was isolated and characterized in the solid state. Moreover, it was obtained in another way [Equation (6)].



A magnetic susceptibility investigation indicates a magnetic moment of ca.  $1.76 \mu_{\text{B}}$ , which corresponds to one unpaired electron per complex. The magnetic moment as a function of temperature remains constant down to ca. 10 K.

The EPR spectrum of **3** (powder), which displays one broad, unresolved line, is quite similar to those reported<sup>[26]</sup> to have been observed for  $[(\alpha, \alpha' \text{-bpy})\text{Rh}(\text{3,6-DBCat})(\text{3,6-DBSQ})]$ , and no temperature changes were detected. Thus, complex **3** appears to be a “ $\text{Co}^{\text{III}}(\text{SQ})(\text{Cat})$ ” species in the solid state that lacks the “ $\text{Co}^{\text{II}}(\text{SQ})_2$ ” impurity.

## Conclusion

Naturally, the charge on the quinone fragment – “3,6-DBQ” in complex  $[(\text{dppe})\text{Co}(\text{“3,6-DBQ”})(\text{“Q”})]$  – depends on the redox potential of the neighboring quinone fragment “Q”. If “Q” is a stronger acceptor, such as “ $\text{Q}_{\text{ox}}^{\text{Cl}}$ ”, then “3,6-DBQ” becomes a semiquinone unit, but if “Q” is a weaker acceptor (“phen-Q”) then it remains a catecholate unit. When “Q” ligands are identical, the complex remains in the  $\text{Co}^{\text{III}}(\text{Cat})(\text{SQ})$  state, in general, but a very small impurity of a  $\text{Co}^{\text{II}}$  species essentially causes lines to broaden in the EPR spectrum. Complex  $[(\text{dppe})\text{Co}(\text{3,6-DBCat})(\text{3,6-DBSQ})]$  in the solid state exists as a “ $\text{Co}^{\text{III}}(\text{Cat})(\text{SQ})$ ” species without any “ $\text{Co}^{\text{II}}(\text{SQ})_2$ ” impurity.

## Experimental Section

**General Remarks:** EPR spectra were recorded on a Bruker ER 200D-SRC (X-band) spectrometer with an ER 4111 VT variable temperature unit. Coupling constants were determined with the help of a simulator (WINEPR SimFonia 1.25). All the operations with semiquinone and catecholate compounds were carried out under vacuum. Solvents were dried by standard procedures and degassed before use.

Phenanthrenequinone, dppe,  $\text{CoCl}_2$ , and solvents were obtained commercially. 3,6-DBQ,<sup>[29,30]</sup> perchloroxanthrenequinone-2,3<sup>[31]</sup> and  $[\text{Co}(\text{3,6-DBSQ})_3]$ <sup>[32]</sup> were obtained according to known procedures.

**[Bis(diphenylphosphanyl)ethane](3,6-di-*tert*-butylcatecholato)cobalt:** An evacuated ampoule containing 3,6-di-*tert*-butyl-*o*-quinone (0.22 g, 1.0 mmol) in THF (30 mL) and a large excess of thallium

amalgam was shaken until the color of the suspension became bright yellow. The light suspension of thallium catecholate was carefully decanted to another evacuated ampoule. Thallium amalgam was carefully washed several times with THF ( $5 \times 10 \text{ mL}$ ) until the solvent became colorless. (Attention! Thallium catecholate is extremely air sensitive.) Thallium catecholate was added to a mixture of dppe (0.4 g, 1 mmol) and  $\text{CoCl}_2$  (0.13 g, 1.0 mmol) in THF (70 mL). The reaction mixture was heated under reflux for 1 h, and then the solvent was changed to toluene. The brown solution was filtered, partly evaporated, diluted with *n*-hexane, and then kept at  $-10^\circ \text{C}$  overnight. The resulting air-stable, brown, crystalline solid was filtered, washed with cold light petroleum, and then dried under vacuum. Yield 0.2 g (33%). IR (Nujol):  $\tilde{\nu} = 515 \text{ s}, 525 \text{ (C}_3\text{P skeleton vibrations); } 735, 695 \text{ v.s. } \delta_{\perp} \text{ C-H (Ph); } 1105 \text{ (v}_{\text{P-C}}\text{); } 1260, 1250 \text{ (v}_{\text{C-O, Cat}}\text{); } 940, 980, 1395, 1375, 1440 \text{ (3,6-DBCat) cm}^{-1}$ .  $\text{C}_{40}\text{H}_{44}\text{CoO}_2\text{P}_2$  (677.67) calcd. C 70.90, H 6.50, Co 8.71; found C 70.95, H 6.49, Co 8.75.

**Sample Preparation:** Equimolar amounts of  $[(\text{dppe})\text{Co}(\text{3,6-DBCat})]$  and the corresponding quinone were placed into a special ampoule for EPR spectroscopic investigations. The ampoule was evacuated and purified toluene was condensed inside.

**[Bis(diphenylphosphanyl)ethane](3,6-di-*tert*-butyl-*o*-benzosemiquinono)(3,6-di-*tert*-butylcatecholato)cobalt (**3**):** Dppe (0.4 g, 1 mmol) in THF (50 mL) was added to  $[\text{Co}(\text{3,6-DBSQ})_3]$  (0.716 g, 1.0 mmol) in THF (80 mL) and then the mixture was carefully warmed. The solvent was evaporated and the residue was washed with *n*-hexane ( $3 \times 50 \text{ mL}$ ). The solid was dissolved in  $\text{CH}_2\text{Cl}_2$ , and then diluted with *n*-hexane. Slow evaporation led to a dark-blue microcrystalline solid, which was filtered, washed by cold light petroleum, and dried under vacuum. Yield: 0.5 g (ca. 50%). An additional crop of crystals was obtained after cooling the mother solution. IR (Nujol):  $\tilde{\nu} = 480, 495, 535 \text{ (C}_3\text{P skeleton vibrations); } 695 \text{ v.s., } 740 \text{ m } \delta_{\perp} \text{ C-H (Ph); } 1105 \text{ (v}_{\text{P-C}}\text{); } 1270, 1280 \text{ (v}_{\text{C-O, Cat}}\text{); } 1410 \text{ s (v}_{\text{C-O, SQ}}\text{) cm}^{-1}$ .  $\text{C}_{54}\text{H}_{64}\text{CoO}_4\text{P}_2$  (897.98) calcd. C 72.24, H 7.13, Co 6.58; found C 72.32, H 7.18, Co 6.60.

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