# Effects of solvation on charge distribution of 1,2-semiquinonato/catecholatocobalt complexes containing bis(3-pyridyl) phenylvinylsilane

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**Abstract** Recrystallization of  $[Co(3,5-dbbq)_2(L)_2]$  (3,5dbbq = 3,5-di-*tert*-butyl-1,2-benzoquinone; L = bis(3-pyridyl)phenylvinylsilane) from diethyl ether at -20 °C produces *trans*- $[Co(3,5-dbbq)_2(L)_2]$  while the recrystallization from toluene at -20 °C gives *trans*- $[Co(3,5-dbbq)_2(L)_2]\cdot2PhMe$ . The complex exists as *trans*- $[Co^{III}(3,5-dbsq)(3,5-dbcat)(L)_2]$ (3,5-dbsq = 3,5-di-*tert*-butyl-1,2-semiquinonato; 3,5-dbcat = 3, 5-di-*tert*-butylcatecholato) in the solid state at 173 K. Differences in charge distribution between *trans*- $[Co(3,5-dbbq)_2(L)_2]$ and *trans*- $[Co(3,5-dbbq)_2(L)_2]\cdot2PhMe$  have been observed based on the effective magnetic moments and IR spectra of the complexes along with their X-ray crystal structures.

# Introduction

Valence tautomers that exhibit different charge distributions via intramolecular electron transfer including spincrossover processes are a class of bistable molecules that can be switched by temperature, light, or pressure [1–3]. Much research has been directed toward controlling the cooperativity of the tautomerism [4, 5], studying photoswitching effects [6–13], and constructing new valence tautomeric systems [14–18] as potential building blocks for molecular electronic devices [19, 20]. Some metal complexes containing 1,2-semiquinonato (sq<sup>-</sup>, S = 1/2) and

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catecholato ( $cat^{2-}$ , S = 0) ligands have shown a specific bistability involving the intramolecular electron transfer between the metal and the ligand, even in the solid state around room temperature [5]. Their different valence states have significantly different charges and spin distributions and thus show characteristic optical, electrical, and magnetic properties [12, 21]. This phenomenon has been clearly revealed for the cobalt complexes, where equilibria between the [Co<sup>III</sup>] and [Co<sup>II</sup>] valence tautomeric species have been observed, both in the solution and in the solid state [2, 6]. Such electronic-labile complexes are known to be very sensitive to environmental factors such as temperature, solvent, state, and photo-energy [5]. Although solvent effects on cobalt valence tautomeric systems have been reported [5, 22], direct comparison of solvation effects on the same parent molecules remains unexplored. In this paper, solvation effects based on the charge distribution of solvated versus non-solvated 1,2-semiquinonato/ catecholatocobalt complexes containing bis(3-pyridyl) phenylvinylsilane (L, Scheme 1) are reported.

## Experimental

Materials and measurements

Co<sub>2</sub>(CO)<sub>8</sub>, 3,5-di-*tert*-butyl-1,2-benzoquinone (3,5-dbbq), and dichlorophenylvinylsilane were purchased from Aldrich and Gelest and used without further purification. Elemental microanalyses (C, H, N) were performed on crystalline samples by the Pusan Center, KBSI, using a Vario-EL III instrument. Infrared spectra were obtained on a Nicolet 380 FTIR spectrophotometer with samples prepared as KBr pellets. Electronic spectra were obtained on a UV–vis spectrophotometer S-3105. Temperature-

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## Scheme 1 Ligand L



dependent magnetic measurements were obtained using a Quantum Design PPMS-9T SQUID magnetometer at a field of strength 10 kG. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Varian Mercury Plus 300 spectrometer, and the chemical shifts are given relative to internal Me<sub>4</sub>Si.

## Synthesis of bis(3-pyridyl)phenylvinylsilane

*n*-Butyllithium (35.0 mmol, 2.5 *M* solution in *n*-hexane) was added to a solution of 3-bromopyridine (33.0 mmol) in dry diethyl ether (60 mL) under nitrogen gas at -78 °C, and the resulting mixture was stirred for 1.5 h. Dichlorophenylvinylsilane (16.0 mmol) was slowly added to the vellow suspension, and the mixture was stirred overnight at 0 °C. Distilled water (50 mL) was added into the reaction solution, and the organic layer was separated, washed with water, and then dried over MgSO<sub>4</sub>. The crude product was purified by column chromatography on silica gel with ethyl acetate as eluent. The solvent was evaporated to obtain the product L as a solid in a 63 % yield. Anal. Calcd. for C<sub>18</sub>H<sub>16</sub>N<sub>2</sub>Si: C, 75.0; H, 5.6; N, 9.7. Found: C, 74.9; H, 5.5; N, 9.5. IR (KBr, cm<sup>-1</sup>): 3068(w), 1571(s), 1468(m), 1394(s), 1331(m), 1192(m), 1120(s), 1030(s), 968(s), 805(s), 714(s), 557(s), 474(m). <sup>1</sup>H NMR (CDCl<sub>3</sub>, SiMe<sub>4</sub>):  $\delta$ 8.70 (s, 2H, Py), 8.67 (dd, J = 4.8 Hz, J = 1.5 Hz, 2H, Py), 7.78 (dt, J = 7.2 Hz, J = 1.8 Hz, 2H), 7.51 (dd, J = 7.8 Hz, J = 1.5 Hz, 2H, Py), 7.60 (d, J = 6.9 Hz, 1H), 7.42 (d, J = 6.9 Hz, 2H), 7.31 (dd, J = 7.8 Hz, J = 4.8 Hz, 2H, Py), 6.68 (dd, J = 20.1 Hz, J = 14.4 Hz, 1H), 6.42 (dd, J = 14.4 Hz, J = 3.3 Hz, 1H), 5.82 (dd, J = 20.1 Hz, J = 3.3 Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, SiMe<sub>4</sub>):  $\delta$  156.1, 151.1, 143.5, 138.7, 135.8, 131.7, 130.5, 128.4, 123.5, 77.6, 76.7.

## Synthesis of *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]

 $Co_2(CO)_8$  (43 mg, 0.12 mmol) and L (144 mg, 0.50 mmol) were combined in 30 mL of toluene. The mixture was stirred for 5 min, and a solution of 3,5-dbbq (110 mg, 0.50 mmol) in toluene (30 mL) was added into the solution, which was then stirred for 1 h at room temperature. Slow evaporation of the solvent gave a dark green microcrystalline product. Dark blue crystals suitable for crystallographic characterization were obtained by recrystallization from diethyl ether at -20 °C in 60 % yield. Anal. Calcd. for  $\begin{array}{l} C_{64}H_{72}N_4O_4Si_2Co:\ C,\ 71.4;\ H,\ 6.7;\ N,\ 5.2.\ Found:\ C,\ 71.4;\\ H,\ 6.6;\ N,\ 5.2.\ IR\ (KBr,\ cm^{-1}):\ 3650(br,\ s),\ 2956(s),\\ 1571(s),\ 1473(s),\ 1394(s),\ 1355(s),\ 1194(m),\ 1118(s),\ 985(s),\\ 897(m),\ 714(s),\ 559(w),\ 487(w). \end{array}$ 

## Synthesis of *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]·2PhMe

 $Co_2(CO)_8$  (43 mg, 0.12 mmol) and L (144 mg, 0.50 mmol) were combined in 30 mL of toluene. The mixture was stirred for 5 min, and a solution of 3,5-dbbq (110 mg, 0.50 mmol) in toluene (30 mL) was added. The solution was stirred for 1 h at room temperature. Dark blue crystals suitable for crystallographic characterization were grown from toluene solution at -20 °C in 55 % yield. Anal. Calcd. for  $C_{78}H_{88}N_4O_4Si_2Co$ : C, 74.3; H, 7.0; N, 4.4. Found: C, 74.3; H, 7.0; N, 4.3. IR (KBr, cm<sup>-1</sup>): 3660(br, s), 2954(s), 1571(s), 1473(s), 1396(s), 1355(s), 1193(m), 1106(s), 987(s), 896(m), 713(s), 559(w), 487(w).

## Crystal structure determination

X-ray data were collected on a Bruker SMART automatic diffractometer with graphite-monochromated Mo Ka radiation ( $\lambda = 0.71073$  Å) and a CCD detector at 170 K. Thirty-six frames of two dimensional diffraction images were collected and processed to obtain the cell parameters and orientation matrix. The data were corrected for Lorentz and polarization effects. The absorption effects were corrected using the multi-scan method (SADABS) [23]. The structures were solved using direct methods (SHELXS 97) and refined by full-matrix least square techniques (SHEL-XL 97) [24, 25]. The non-hydrogen atoms were refined anisotropically, and the hydrogen atoms were placed in calculated positions and refined only for the isotropic thermal factors. The crystal parameters and procedural information corresponding to the data collection and structure refinement are listed in Table 1.

## **Results and discussion**

## Synthesis

The synthetic method described in earlier studies was used to prepare the present complex *trans*- $[Co(3,5-dbbq)_2(L)_2]$ [22]. Thus, the reaction between  $Co_2(CO)_8$  and 3,5-dbbq in the presence of L as coligand afforded the present complex in toluene at room temperature. *trans*- $[Co(3,5-dbbq)_2(L)_2]$ containing two monodentate L coligands was formed instead of the species containing a bidentate L, presumably owing to the steric hindrance resulting from chelation of L. An important feature is that recrystallization from diethyl ether at -20 °C produces *trans*- $[Co(3,5-dbbq)_2(L)_2]$ .

Table 1 Crystal data and structure refinement for trans-[Co(3, 5-dbbq)<sub>2</sub>(L)<sub>2</sub>] and trans-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]·2PhMe

	<i>trans</i> -[Co(3, 5-dbbq) <sub>2</sub> (L) <sub>2</sub> ]	trans-[Co(3, 5-dbbq) <sub>2</sub> (L) <sub>2</sub> ] $\cdot$ 2PhMe
Empirical formula	$C_{64}H_{72}N_4O_4Si_2Co$	C <sub>78</sub> H <sub>88</sub> N <sub>4</sub> O <sub>4</sub> Si <sub>2</sub> Co
Weight	1076.37	1260.63
Crystal system	Triclinic	Triclinic
Space group	$P\overline{1}$	$P\overline{1}$
a (Å)	10.2316(2)	10.3115(5)
<i>b</i> (Å)	11.2622(2)	12.6999(6)
<i>c</i> (Å)	13.4227(3)	13.3212(6)
α (°)	100.301(1)	81.848(3)
β (°)	91.233(1)	85.502(3)
γ (°)	105.349(1)	81.660(3)
Volume (Å <sup>3</sup> )	1463.62(5)	1705.6(1)
Density (mg/m <sup>3</sup> )	1.221	1.227
Ζ	1	1
Absorption coefficient (mm <sup>-1</sup> )	0.384	0.339
F(000)	571	671
<i>R</i> <sub>int</sub>	0.0572	0.1303
Completeness (%)	99.7	96.9
GoF on $F^2$	1.032	1.043
$R_1 \left[ I > 2\sigma(I) \right]^{\rm a}$	0.0609	0.0800
$wR_2$ (all data) <sup>b</sup>	0.1693	0.2286

 $\overline{ a } R_1 = \sum ||F_0| - |F_c|| / \sum |F_0|$   $b wR_2 = (\sum w (F_0^2 - F_c^2)^2 / \sum w F_0^2)^{1/2}$ 

whereas crystallization from toluene at -20 °C gives *trans*- $[Co(3,5-dbbq)_2(L)_2]$ ·2PhMe. The crystalline products gave satisfactory chemical analyses and were soluble in

Fig. 1 ORTEP drawings of trans-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>] (left) and trans-[Co(3,5dbbq)2(L)2]·2PhMe (right) showing thermal ellipsoids at 20 % probability. Hydrogen atoms are omitted for clarity

common organic solvents such as toluene, benzene, tetrahydrofuran, acetone, dimethyl sulfoxide, and N,N-dimethylformamide. However, the products were easily dissociated in dimethyl sulfoxide, alcohols, and N,N-dimethylformamide. The solubility characteristics indicate that the complexes are discrete molecules instead of polymeric species. The complexes were characterized by spectral, thermal, and magnetic properties along with the crystal structures. The same products were formed upon varying the mole ratio of reactants, indicating that the discrete trans- products are favorable species. Formation of the discrete molecular species instead of polymers may be attributed to the small and rigid L coligand.

#### Crystal structures

The crystal structures of *trans*- $[Co(3,5-dbbq)_2(L)_2]$  and trans-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]·2PhMe are depicted in Fig. 1, and selected bond lengths and angles are listed in Table 2. The L coligand is monodentate, resulting in discrete centrosymmetric mononuclear complexes with two free pyridyl groups. The molecule approximates to the charge distribution of *trans*-[Co<sup>III</sup>(3,5-dbsq)(3,5-dbcat)(L)<sub>2</sub>] (3,5 $dbsq^{-} = 3,5$ -di-*tert*-butyl-1,2-semiquinonate; 3,5-dbcat<sup>2-</sup> = 3,5-di-*tert*-butylcatecholate) rather than *trans*-[Co<sup>II</sup>(3,5 $dbsq_{2}(L)_{2}$  in the solid state at 173 K; this assignment is consistent with the IR data and effective magnetic moments as well as the Co-O bond lengths. The Co-O bond lengths  $(1.877(2) \text{ and } 1.887(2) \text{ Å for } trans-[Co(3,5-dbbq)_2(L)_2];$ 1.878(3) and 1.874(3) Å for *trans*- $[Co(3,5-dbbq)_2(L)_2]$ . 2PhMe) are also shorter than generally observed for Co<sup>II</sup>-O bonds ( $\geq 2.00$  Å). The radius of low-spin Co<sup>III</sup> is roughly 0.2 Å smaller than that of high-spin Co<sup>II</sup> [5]. The low-spin Co<sup>III</sup>



trans-[Co(3,5-dbbq) <sub>2</sub> (L) <sub>2</sub> ]		<i>trans</i> -[Co $(3,5-dbbq)_2(L)_2$ ]·2	trans-[Co(3,5-dbbq) <sub>2</sub> (L) <sub>2</sub> ]·2PhMe	
Co(1)–O(1)	1.877(2)	Co(1)–O(1)	1.878(3)	
Co(1)–O(2)	1.887(2)	Co(1)–O(2)	1.874(3)	
Co(1)–N(1)	1.953(2)	Co(1)–N(1)	1.946(4)	
C(1)–O(1)	1.327(3)	C(1)–O(1)	1.321(6)	
C(2)–O(2)	1.334(3)	C(2)–O(2)	1.340(6)	
O(1)–Co(1)–O(2)	93.20(8)	O(1)–Co(1)–O(2)	87.0(1)	
O(1)–Co(1)–N(1)	90.1(1)	O(1)-Co(1)-N(1)	90.7(2)	
O(2)–Co(1)–N(1)	90.80(9)	O(2)–Co(1)–N(1)	90.0(2)	
$O(1)-Co(1)-O(1)^{\#1}$	180.0	$O(1)-Co(1)-O(1)^{#2}$	180.00(1)	
$O(1)-Co(1)-O(2)^{\#1}$	86.80(8)	O(1)-Co(1)-O(2) <sup>#2</sup>	93.00(1)	
	$rans-[Co(3,5-dbbq)_2(L)_2]$ $Co(1)-O(1)$ $Co(1)-O(2)$ $Co(1)-N(1)$ $C(1)-O(1)$ $C(2)-O(2)$ $D(1)-Co(1)-O(2)$ $D(1)-Co(1)-N(1)$ $D(2)-Co(1)-N(1)$ $O(1)-Co(1)-O(1)^{\#1}$ $O(1)-Co(1)-O(2)^{\#1}$	rans-[Co(3,5-dbbq)_2(L)_2]Co(1)-O(1)1.877(2)Co(1)-O(2)1.887(2)Co(1)-N(1)1.953(2)C(1)-O(1)1.327(3)C(2)-O(2)1.334(3)O(1)-Co(1)-O(2)93.20(8)O(1)-Co(1)-N(1)90.1(1)O(2)-Co(1)-N(1)90.80(9)O(1)-Co(1)-O(1) <sup>#1</sup> 180.0O(1)-Co(1)-O(2) <sup>#1</sup> 86.80(8)	$\begin{array}{llllllllllllllllllllllllllllllllllll$	

would be expected to be in a strictly octahedral geometry, in contrast to a few known examples of the high-spin Co<sup>II</sup> trigonal prismatic geometry [27]. The centrosymmetric molecules result in similar C – O bonds: C(1)-O(1) = 1.327(3)Å, C(2)-O(2) = 1.334(3) Å for *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]; C(1)-O(1) = 1.321(6) Å, C(2)-O(2) = 1.340(6) Å for *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>]·2PhMe. For *trans*-[Co(3,5-dbbq)<sub>2</sub> (L)<sub>2</sub>]·2PhMe, the two solvate toluene molecules are located in the vacancies within the unit cell (Fig. 2).

## Spectroscopic data

The temperature-dependent magnetic moments [26] recorded on solid samples of the two complexes are depicted in Fig. 3. For *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>], the effective magnetic moment remains at approximately 2.3  $\mu_{\rm B}$  at 5 K and, finally, approaches 5.0  $\mu_{\rm B}$  at 360 K, indicating that *trans*-[Co<sup>III</sup>(3,5-dbsq)(3,5-dbcat)(L)<sub>2</sub>] shifts to the tautomeric species *trans*-[Co<sup>II</sup>(3,5-dbsq)<sub>2</sub>(L)<sub>2</sub>] with increasing temperature in the solid state. At high temperatures, the value of 5.0  $\mu_{\rm B}$  is indicative of weak antiferromagnetic exchange between the S = 3/2 metal center and the two S = 1/2semiquinonato (3,5-dbsq) ligands. For *trans*-[Co(3,5dbbq)<sub>2</sub>(L)<sub>2</sub>]·2PhMe,  $\mu_{\rm eff}$  is 1.7  $\mu_{\rm B}$  at 5 K, rising to 3.6  $\mu_{\rm B}$  at 360 K, indicating that valence tautomerism occurs in the solid state in this case also. The magnetic moment value



**Fig. 2** Packing diagrams of *trans*- $[Co(3,5-dbbq)_2(L)_2]$  (*left*) and *trans*- $[Co(3,5-dbbq)_2(L)_2]$ ·2PhMe in *a*-axis view. All hydrogen atoms are omitted for clarity

should converge to 1.7  $\mu_{\rm B}$  at the lowest temperature, resulting from the 1,2-semiquinonato (sq<sup>-</sup>, *S* = 1/2) moiety of low-spin *trans*-[Co<sup>III</sup>(3,5-dbsq)(3,5-dbcat)(L)<sub>2</sub>]. Electron transfer from the 3,5-dbcat ligand to the Co<sup>III</sup> center controls the ratio of [Co<sup>III</sup>]/[Co<sup>II</sup>] species. Thus, the ratio of [Co<sup>III</sup>]/[Co<sup>II</sup>] can be controlled by the donor effects of the coligand L, with great sensitivity.

The characteristic IR band around 4,000 cm<sup>-1</sup> (Fig. 4) indicates the presence of  $[Co^{III}]$  species in the solid state at room temperature [22], which is consistent with the X-ray crystal structures and effective magnetic moments discussed above. A more important result is that the IR band of the solvated crystal, *trans*- $[Co(3,5-dbbq)_2(L)_2]$ ·2PhMe, is stronger than that of *trans*- $[Co(3,5-dbbq)_2(L)_2]$ , indicating that the  $[Co^{III}]/[Co^{II}]$  ratio of the solvated crystal is larger than that of the non-solvated crystal, which is coincident with the above magnetic susceptibility measurements.

The electronic spectrum ( $\sim 10^{-4}$  M) of *trans*-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>] in the range of 400–900 nm was measured in tetrahydrofuran (Fig. 5). The two bands observed at 640 and 740 nm are the characteristic of the [Co<sup>III</sup>] and [Co<sup>II</sup>] tautomers, respectively [22]. Hence, the electronic



**Fig. 3** Temperature-dependent effective magnetic moments for *trans*- $[Co(3,5-dbbq)_2(L)_2]$  (*solid line*) and *trans*- $[Co(3,5-dbbq)_2(L)_2]$ · 2PhMe (*dashed line*) in the solid state



**Fig. 4** IR spectra of *trans*- $[Co(3,5-dbbq)_2(L)_2]$  (solid line) and *trans*- $[Co(3,5-dbbq)_2(L)_2]$ ·2PhMe (*dashed line*)



Fig. 5 Electronic spectrum of trans-[Co(3,5-dbbq)<sub>2</sub>(L)<sub>2</sub>] in tetrahydrofuran

spectrum provides further evidence that the compound exists as an equilibrium between  $[Co^{III}]$  and  $[Co^{II}]$  species in solution. The ratio of  $[Co^{III}]/[Co^{II}]$  in solution is, however, small compared to that in the solid state, which is a general trend of such systems [22]. The distinctive physicochemical properties of the present system, including the transition temperature  $T_c$ , may be attributed to the bulky monodentate coligand L. Both *trans*- $[Co(3,5-dbbq)_2(L)_2]$ and *trans*- $[Co(3,5-dbbq)_2(L)_2]$ ·2PhMe, of course, show the same electronic spectrum in solution.

#### Conclusion

Mononuclear cobalt complexes with two free nitrogen donors may act as sensitive metalloligands. In particular, direct evidence for differences in charge distribution between solvated and non-solvated crystals has been observed in this work. Further experiments on such bistable molecular systems will provide more detailed information on the notable solvation effects.

### Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Center, under deposition number CCDC 871339 and 871340 for the complex *trans*- $[Co(3,5-dbbq)_2(L)_2]$  and *trans*- $[Co(3,5-dbbq)_2(L)_2]\cdot 2C_6$  H<sub>5</sub>CH<sub>3</sub>, respectively. Copy of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2, 1EZ, UK (fax: +44-1223-336033); E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam. ac.uk).

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