Quinone Adducts of Cobalt(II) and Iron(II) Complexes with a Quadridentate Schiff's Base

By **C. Floriani,* G. Fachinetti, and F. Calderazzo,** Istituto di Chimica Generale ed Inorganica, University of Pisa, 56100 Pisa, Italy

The reactions of some *p*- and *o*-quinones with *N*,*N'*-ethylenebis(salicylideneiminato)cobalt(II), Co(salen), and *N*,*N'*-ethylenebis(salicylideneiminato)iron(II), Fe(salen), have been studied. A low-spin 2:1 adduct of the cobalt complex with *p*-benzoquinone was isolated in pyridine: its formula is $[Co(salen)(py)]_2C_6H_4O_2$ and a dimeric structure with a bridging quinone and six-co-ordinate cobalt(III) is proposed for it. The iron complex in tetrahydrofuran suspension yielded 2:1 adducts with *p*-quinones (*p*-benzoquinone, tetramethyl-*p*-benzoquinone, and tetrachloro-*p*-benzoquinone) of formula $[Fe(salen)]_2$quinone: a dimeric structure with a bridging quinone is suggested for them mainly on the basis of magnetic properties. With *o*-quinones (9,10-phenanthrenequinone and 1,2-naphthoquinone), 1:1 adducts of formula Fe(salen)(quinone) were obtained.

p-BENZOQUINONE and substituted *p*-benzoquinones are known to interact with transition metals giving rise either to diolefin complexes [1-3] or to an electron-transfer type of complex.[4,5] The oxidative addition of *p*-benzoquinone to the $[Co(CN)_5]^{3-}$ anion yields the cobalt(III) derivative $[(NC)_5CoOC_6H_4OCo(CN)_5]^{6-}$, involving the co-ordination of the quinone moiety through the oxygen atoms to the cobalt(III) centres.

It is also worth mentioning that electron acceptors such as benzotrifuroxan, picryl azide, and 1,2,4,5-tetracyanobenzene have been used to form molecular complexes with 8-hydroxyquinolinato-complexes of copper, nickel, and palladium.[6] Most of these complexes are, however, of the charge-transfer type.[7]

We report *p*-quinone adducts obtained from cobalt(II) and iron(II) complexes of the quadridentate *N*,*N'*-ethylenebis(salicylideneiminate) ligand. No quinone adducts of Schiff's base metal complexes have been

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reported as yet.[8,9] However, Basolo and co-workers have reported the reactions of *N*,*N*′-ethylenebis(acetylacetoniminato)cobalt(II) with tetracyanoethylene[10] and with tetracyanoquinone dimethide.[11]

We also report adducts of the quadridentate Schiff's base–iron complex with the *o*-quinones, 9,10-phenanthrenequinone, and 1,2-naphthoquinone. The preparation and some of the properties of the adduct between 9,10-phenanthrenequinone and *N*,*N*′-ethylenebis(salicylideneiminato)iron(II), Fe(salen), have been reported in an earlier paper[12] dealing with the preparation and the properties of binary phenanthrenequinone complexes of iron, cobalt, and nickel.

TABLE 1

Magnetic properties of the quinone adducts

	Compound	T/K	$\chi_M \cdot 10^6$ [a] (c.g.s.u.)	Diamagnetic correction (c.g.s.u.)	μ_{eff}/B.M.
(I)	$[Co(salen)]_2$(*p*-quinone)$(py)_2$	287	235	219	0·74
(II)	$[Fe(salen)]_2$(*p*-quinone)	297	13 945	205	5·76
(VI)	Fe(salen)(9,10-phenanthrenequinone)	288	9 990	256	4·82

[a] Diamagnetic correction included. The values of χ_M were averaged over at least three different magnetic field strengths. The magnetic susceptibilities were found to be substantially independent of field.

TABLE 2

			Analytical data, %					
			Found			Calc.		
	Compound	Colour	C	H	N	C	H	N
(I)	$[Co(salen)]_2$(*p*-quinone)$(py)_2$	Deep green	63·3	4·75	9·0	62·9	4·6	9·15
(II)	$[Fe(salen)])_2$(*p*-quinone) [a]	Deep violet	60·75	4·4	7·4	60·65	4·3	7·45
(III)	$[Fe(salen)]_2$(Me_4-*p*-quinone)	Deep brown	62·75	5·55	6·45	62·4	5·0	6·95
(IV)	$[Fe(salen)]_2$(Cl_4-*p*-quinone) [b]	Deep violet	51·4	3·2	6·4	51·25	3·15	6·3
(V)	Fe(salen)(9,10-phenanthrenequinone) [c]	Deep violet	67·5	4·2	5·35	67·95	4·2	5·3
(VI)	Fe(salen)(1,2-naphthoquinone)	Deep violet	64·5	4·3	5·9	65·0	4·2	5·85

[a] Iron: found 15·0 (calc. 14·85%). [b] Chlorine: found 16·05 (calc. 15·95%). [c] From ref. 12).

RESULTS

Cobalt Adducts.—*N*,*N*′-Ethylenebis(salicylideneiminato)cobalt(II), Co(salen), was shown to form a 2 : 1 adduct with *p*-benzoquinone, according to reaction (1), which took

$$2Co(salen) + C_6H_4O_2 + 2C_5H_5N \longrightarrow C_5H_5N{-}Co(salen){-}OC_6H_4O{-}Co(salen){-}NC_5H_5 \quad (1)$$

(I)

place at room temperature. No reaction occurred between Co(salen) and *p*-benzoquinone in dimethylformamide (DMF), dimethyl sulphoxide, or tetrahydrofuran. Even in pyridine, no reaction was observed between Co(salen) and tetrachloro-*p*-benzoquinone, tetramethyl-*p*-benzoquinone, and 9,10-phenanthrenequinone.

Formation of the 2 : 1 adduct (I) took place independently of the cobalt : quinone ratio used. Compound (I) is stable in air in the solid state and its solubility in all common organic solvents is very limited. In the i.r. spectrum of (I) no bands are observed above 1635 cm^{-1}, attributable to uncoordinated *p*-benzoquinone, which was reported[13] to have two CO stretching vibrations at 1674 and 1662 cm^{-1} in $CHCl_3$ solution. The band at 1635 cm^{-1} and some of those below can be easily recognised as vibrations of the Schiff's base ligand by comparison with Co(salen)(py). The strong reduction of the C–O bond order upon complexation can be attributed to electron transfer from cobalt to the ligand. This is confirmed by the magnetic susceptibility measurements (see Table 1). Compound (I) has a magnetic moment[14] μ_{eff} = 0·74 B.M. at 287 K, to be compared with the magnetic moment μ_{eff} = 0·50 B.M. for the 2 : 1 oxygen adduct[15] $[Co(salen)]_2O_2(py)_2$ and with μ_{eff} = 0·70 B.M. for $[Co(salen)]_2O_2(DMF)_2$.[15] The low value of magnetic moment should be attributed to temperature independent paramagnetism, although we did not establish this point by measurements at variable temperature. On the basis of the above results, compound (I) is believed to be best represented as a co-ordination compound of the $C_6H_4O_2{}^{2-}$ anion containing six-co-ordinate cobalt(III), similar to the proposed[15] structure of the oxygen adduct $[Co(salen)]_2O_2(DMF)_2$, confirmed by an *X*-ray investigation.[16]

The salt-like formulation $[(py)Co(salen)]_2{}^+C_6H_4O_2{}^{2-}$, similar to the one suggested[11] for the 1 : 1 adduct of *N*,*N*′-ethylenebis(acetylacetoneiminato)cobalt(II) and tetracyanoquinone is not regarded as a real possibility since it would not be in agreement with the diamagnetism of the compound, which is typical of six-co-ordinate cobalt(III).

Apart from the magnetic properties, the *p*-benzoquinone adducts of Co(salen) are similar to the corresponding oxygen adducts in their chemical behaviour. Reversion to Co(salen), pyridine, and *p*-benzoquinone was observed either by heating the compound *in vacuo* at about 80 °C or

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[11] S. G. Clarkson, B. C. Lane, and F. Basolo, *Inorg. Chem.*, 1972, **11**, 662.

[12] C. Floriani, R. Henzi, and F. Calderazzo, *J.C.S. Dalton*, 1972, 2640.

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[14] We are indebted to Professor C. Ercolani of the Istituto Chimico, Rome, for this measurement.

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by room-temperature treatment with chloroform. The oxygen adducts with dimethylformamide and dimethyl sulphoxide had a similar behaviour.[15]

Iron Adducts.—2 : 1 and 1 : 1 Adducts were obtained with Fe(salen), with *p*- and *o*-quinones, respectively (see Table 2). The reactions were all carried out in tetrahydrofuran suspension and took place rapidly at room temperature and in good yields:

$$2\mathrm{Fe(salen)} + \mathrm{C_6H_4O_2} \xrightarrow{\mathrm{THF}} \mathrm{Fe(salen)}\text{–}\mathrm{C_6H_4O_2}\text{–}\mathrm{Fe(salen)} \quad (2)$$

(II)

$$2\mathrm{Fe(salen)} + \mathrm{C_6Me_4O_2} \xrightarrow{\mathrm{THF}} \mathrm{Fe(salen)}\text{–}\mathrm{C_6Me_4O_2}\text{–}\mathrm{Fe(salen)} \quad (3)$$

(III)

$$2\mathrm{Fe(salen)} + \mathrm{C_6Cl_4O_2} \xrightarrow{\mathrm{THF}} \mathrm{Fe(salen)}\text{–}\mathrm{C_6Cl_4O_2}\text{–}\mathrm{Fe(salen)} \quad (4)$$

(IV)

Compounds (II)—(IV) are black microcrystalline solids; (II) and (IV) are stable in air in the solid state, whereas (III) is sensitive to air oxidation, the products of the reaction being tetramethyl-*p*-benzoquinone and $[\mathrm{Fe(salen)}]_2\mathrm{O}$. This reaction can be used to establish that no alterations of the starting materials have taken place in the course of reaction (3). As with the cobalt adducts, the i.r. spectra of iron 2 : 1 adducts (II) and (IV) show no bands attributable to unco-ordinated quinones [tetrachloro-*p*-benzoquinone: ν(CO) at 1681 $\mathrm{cm^{-1}}$ as Nujol mull [17]]. The C–O stretching vibration of tetramethyl-*p*-benzoquinone would fall together with those of the organic chelate ligand [ν(CO) at 1639 $\mathrm{cm^{-1}}$ as Nujol mull [17]] but there are no reasons to believe that the absorption due to unco-ordinated quinone should be present in this case.

Of particular interest are the magnetic susceptibility measurements on compound (II), see Table 3. These data show that the *p*-benzoquinone adduct of Fe(salen) has a substantially normal Curie–Weiss behaviour, although the Weiss temperature is somewhat high (−52°). The magnetic moment at room temperature is 5·76 B.M., thus quite close to the spin-only value for five unpaired electrons. These results suggest that we are dealing, for compound (II), with high-spin iron(III) of d^5 configuration. In agreement with this and with the i.r. results, we suggest for (II) a structure similar to that of the corresponding cobalt(III) derivative discussed above, *i.e.* a dimeric structure with a quinone bridging unit.

TABLE 3

Temperature-dependence of magnetic susceptibility for $[\mathrm{Fe(salen)}]_2$(*p*-quinone) (II)

T/K	$\chi_{\mathrm{Fe}} \cdot 10^6$ (c.g.s.u.)	μ_{eff}/B.M.[a]
297	13 945	5·76
240	15 560	5·49
180	19 730	5·35
150	22 510	5·22
120	27 140	5·13
90	32 720	4·87

[a] Weiss temperature = −52°.

The proposed structure is strongly supported by the results of the magnetic measurements suggesting the presence of high-spin iron(III) centres with no important magnetic interactions between them at least in the temperature range studied. The magnetic moment at room temperature of the *p*-benzoquinone adduct is very similar to that of the chloro-derivative $\mathrm{Fe(salen)Cl,CH_3NO_2}$,[18] which was shown [19] to be monomeric in the solid state with unco-ordinated nitromethane (see Discussion section). The proposed structure implies the presence of iron(III) with square-pyramidal structure. This is a quite common stereochemistry for iron(III), especially with the ethylenebis(salicylideneiminate)-type of ligand. It may be enough to recall that, as mentioned above, the nitromethane of the chloro-derivative adduct is not co-ordinated to iron and that the μ-oxo-derivative $[\mathrm{Fe(salen)}]_2\mathrm{O}$ contains five-co-ordinate iron(III). It is interesting to note that no pyridine was retained in the co-ordination sphere of the metal when the μ-oxo-compound was prepared [20] by reaction of Fe(salen) with oxygen in pyridine as solvent.

The 2 : 1 adducts obtained by reactions (3) and (4) are assumed to have structures similar to the one suggested for (II).

1 : 1 Adducts (V) and (VI) were obtained with Fe(salen) by the reactions of the iron(II) chelate with 9,10-phenanthrenequinone or 1,2-naphthoquinone in tetrahydrofuran. The solids obtained by these reactions are deep violet, they are decomposed by oxygen to the μ-oxo-iron(III) compound and the starting quinone. No bands due to the unco-ordinared quinones were observed in the i.r. spectra of compounds (V) and (VI). According to magnetic susceptibility measurements carried out [14] on compound (V), the magnetic moment is $\mu_{\mathrm{eff}} = 4{\cdot}82$ B.M. at room temperature. This corresponds to the theoretical spin-only value for four unpaired electrons, and, to a first approximation, may be interpreted as being due to spin coupling between a d^5 high-spin iron centre and a semiquinone moiety, which amounts to attributing a formal oxidation state III to the metal. Another bonding possibility would be that of regarding the central metal as iron(IV) of high-spin d^4 configuration bonded to the quinol dianion. This is considered as a less likely possibility, first, because of the low stability of iron(IV) and secondly, because the i.r. spectrum of compound (V) shows a strong i.r. (polychlorotrifluoroethylene mull) absorption *ca.* 1470 $\mathrm{cm^{-1}}$ which is absent in both Fe(salen) and phenanthrenequinone and which is attributed, on the basis of its intensity to a C–O stretching vibration of the co-ordinated quinone. This corresponds to a *ca.* 210 $\mathrm{cm^{-1}}$ shift with respect to unco-ordinated phenanthrenequinone (1684 $\mathrm{cm^{-1}}$ in $\mathrm{CCl_4}$ solution [21]), which is in the range expected for a semiquinone type of ligand.[12] The i.r. results exclude the alternative possibility of high-spin iron(II) bonded to neutral phenanthrenequinone. No molecular weight measurements could be carried out because of very limited solubility in hydrocarbon solvents or other poorly co-ordinating solvents.

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DISCUSSION

The analogy between 2 : 1 quinone and oxygen cobalt adducts is substantial. The quinone adduct can be decomposed to its components in the presence of $CHCl_3$ or thermally, behaviour which is common to some of the oxygen adducts.[15] Moreover, as was the case for the oxygen adducts, the solvent is a very critical factor for the formation of the quinone adducts. Formation of the cobalt–*p*-benzoquinone 2 : 1 adduct was only observed in pyridine, which is also a suitable solvent for oxygenation.[15] The influence of the *trans*-ligand upon the reactivity [15,22] and the electronic configuration [23] of cobalt(II) complexes with an equatorial planar arrangement of the ligands has been studied. It has been pointed out that a considerable enhancement of rates was observed [22] in the presence of strong axial ligands in the course of the alkylation of bis(dioximato)cobalt(II) complexes; also it has been suggested by e.s.r. measurements [23] that certain nitrogen and oxygen donor ligands may alter the relative energies of d_{xy} and d_{z^2} orbitals such as to favour the electronic configuration d_{yz}^2, d_{xz}^2, d_{xy}^2, $d_{z^2}^1$, $d_{x^2-y^2}^0$, which is believed to be essential for an electron transfer to take place in the axial position, *i.e.* along the z axis. We believe that a similar type of reasoning applies also to the 2 : 1 quinone adducts. We may therefore suggest that the lifting of energy of the singly occupied d_{z^2} orbital promoted by pyridine makes the electron transfer to the organic ligand thermodynamically more favourable. However, since dimethyl sulphoxide is also known [23] to induce the $d_{z^2}^1$ configuration of Co(salen), our failure to isolate the *p*-benzoquinone adduct in this solvent should not be taken as evidence against the formation of the adduct. It may be that in this solvent the total energy balance is such that the equilibrium is not considerably shifted towards the formation of the adduct. Electronic spectra and e.s.r. measurements should be able to establish this point.

Reasonable agreement seems to have been reached concerning the oxygenation of cobalt(II) chelates in assuming that a 1 : 1 adduct is the primary product of the reaction.[15,24,25] We assume that also in the case of the *p*-benzoquinone adducts the formation of the 1 : 1 adducts is the slow, primary step of the reaction. Of course, e.s.r. measurements will be helpful in detecting the 1 : 1 adduct before it further reacts with the excess of complex present in solution.

In the case of the *o*-quinones, the radical anion arising from the primary electron-transfer from the metal may become stabilised by a chelating effect, if the structure of the 1 : 1 adducts is symmetric. On the other hand, if the structure is asymmetric, the approach of a second chelate molecule may be hindered for steric reasons. One of the two is almost certainly the reason for the successful isolation of 1 : 1 adducts with 9,10-phenanthrenequinone and with 1,2-naphthoquinone.

It is interesting to note that the formation of the iron–*p*-benzoquinone adducts is in some cases reversible, if the reaction with oxygen to give $[Fe(salen)]_2O$ and the quinone is taken as a good criterion of reversibility. The reaction took place with compounds (III), (V), and (VI), *i.e.* with the adducts of tetramethyl-*p*-benzoquinone, 9,10-phenanthrenequinone, and 1,2-naphthoquinone, respectively. No reaction with oxygen to $[Fe(salen)]_2O$ and the quinone was observed with compounds (II) and (IV), *i.e.* with *p*-benzoquinone and tetrachloro-*p*-benzoquinone adducts. This can be tentatively correlated with the standard reduction potentials of the quinones which decrease in the order:[21] $C_6Cl_4O_2 > C_6H_4O_2 >$ 1,2-naphthoquinone > 9,10-phenanthrenequinone. There are no data available in the literature for tetramethyl-*p*-benzoquinone but this can be accommodated in between *p*-benzoquinone and 1,2-naphthoquinone in the series of the reduction potentials. It therefore appears that the reversibility of the quinone adducts' formation is related to a lower stability of the reduced quinone species.

Finally it is worth commenting on the results of the magnetic susceptibility measurements for the 2 : 1 adduct of *p*-benzoquinone with Fe(salen), compound (II). It has been mentioned earlier that the compound has a substantially normal Curie–Weiss behaviour from room temperature to liquid nitrogen temperature. However, the Weiss temperature (−52°) is somewhat high and the values of the effective magnetic moment decrease considerably at lower temperatures (see Table 3). This may be because of the beginning of a coupling phenomenon between the d^5 iron(III) centres through the bridging $C_6H_4O_2$ ligand which could be revealed by a study at temperatures lower than that of liquid nitrogen. It is interesting to note that recently spin–spin coupling has been found to occur [26] between copper(II) centres through pyrazine ligands in the temperature range 2·9—65 K.

EXPERIMENTAL

Unless otherwise stated, all the reactions described here were carried out under an atmosphere of purified nitrogen. Solvents were purified by standard methods.

N,N'-Ethylenebis(salicylideneiminato)cobalt(II) [15] and the corresponding iron(II) [20] compounds were prepared as previously described. All the commercially available quinones were purified by sublimation or crystallisation before use.

I.r. spectra were measured with Perkin-Elmer spectrometers 521 and 337.

Magnetic susceptibility measurements were carried out with a Gouy balance and in all cases the χ_M is quoted in c.g.s.u.

Some of the typical reactions are described in detail, the

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preparations of the other compounds being carried out under similar conditions.

Reaction of N,N′-*Ethylenebis*(*salicylideneiminato*)*cobalt*(II) *with* p-*Benzoquinone, Compound* (I).—Co(salen) (2·16 g; 6·64 mmol) was suspended in pyridine (30 ml) and treated at room temperature with *p*-benzoquinone (0·36 g; 3·33 mmol). Upon addition of the quinone the colour of the suspension changed immediately from red-brown to maroon-green. After two days stirring at room temperature, the solid was collected by filtration, washed several times with toluene, and dried *in vacuo* (2·56 g; 84% yield). The *compound* was analysed without further purification. The *p*-benzoquinone adduct (I) (0·25 g) was treated with $CHCl_3$ (20 ml). Immediately after the addition of chloroform, the solid dissolved giving a red solution from which Co(salen) crystallised out as the chloroform adduct Co(salen),$\frac{1}{2}$($CHCl_3$) (Found: C, 51·05; H, 3·9; Cl, 13·3. $C_{16\cdot5}H_{14\cdot5}Cl_{1\cdot5}CoN_2O_2$ requires C, 51·5; H, 3·8; Cl, 13·8%).

Reaction of N,N′-*Ethylenebis*(*salicylideneiminato*)*iron*(II) *with* p-*Benzoquinone, Compound* (II).—Fe(salen) (1·95 g; 6·05 mmol) was added to a solution of *p*-benzoquinone (0·26 g; 3·33 mmol) in tetrahydrofuran (100 ml). The immediate formation of a green-maroon suspension was noticed, whose colour turned to deep violet over two days. The solid was filtered off, washed with tetrahydrofuran, dried *in vacuo* (1·8 g; 79% yield), and analysed without further purification. The *compound* has a very limited solubility in all polar organic solvents, but can be recrystallised at 100 °C from tetrahydrofuran in a steel autoclave.

Reaction of N,N′-*Ethylenebis*(*salicylideneiminato*)*iron*(II) *with* 1,2-*Naphthoquinone, Compound* (VI).—Fe(salen) (0·205 g; 0·64 mmol) was added to a solution of 0·12 g (0·76 mmol) of the quinone in tetrahydrofuran (20 ml). A deep violet suspension was immediately obtained which was stirred at room temperature for two days. Toluene (50 ml) was added and the solid was then filtered off, washed with toluene, dried *in vacuo* (0·10 g, 33% yield), and analysed without further purification.

I.r. spectra (Nujol mulls) of the compounds reported in this paper are deposited in Supplementary Publication No. SUP 20643 (4 pp.).*

Reactions of the Iron–Quinone Adducts with Oxygen.—The reaction of compound (V) with oxygen is described as a typical example. The iron–phenanthrenequinone adduct (V) (0·30 g) was suspended in toluene (10 ml) and exposed to dry air. The oxidation in this solvent was very slow. Tetrahydrofuran was then added and a very rapid oxidation was observed. The orange solid formed was collected by filtration and recognised as pure [Fe(salen)]$_2$O by its i.r. spectrum. The filtered solution was evaporated to dryness and the resulting yellow solid identified as 9,10-phenanthrenequinone by i.r. spectrum and melting point.

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