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Formation constants of some phosphine and phosphite adducts of (η^5-cyclopentadienyl) (substituted 1,2-ethylenedichalcogenolato)cobalt(III) complexes and their ^{1}H, ^{13}C and ^{31}P NMR spectra

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Abstract

The formation constants of the phosphine and phosphite adducts of several (η^5-cyclopentadienyl) (substituted 1,2-ethylenedithiolato)cobalt(III) complexes in acetonitrile were determined spectrophotometrically. The complexes with more electron-withdrawing substituents on the ditholate ligand form more stable adducts. The NMR chemical shifts (^{1}H, ^{13}C and ^{31}P) of the complexes, phosphines, phosphites and adducts were measured in CDCl$_3$. The shifts were changed on adduct formation. These changes indicate that the phosphines act as σ-donors and the phosphites as π-acceptors and that the cobaltadithiolene ring, which is aromatic in the unbound complex, becomes olefinic in the adduct.

1. Introduction

1,2-Ethylenedithiolato and -diselenolato ligands are often called "non-innocent" ligands, because the oxidation states of the central atoms of chelates they form cannot be determined uniquely. Since King [1] first reported a cyclopentadienyl complex, [Co(Cp)(S$_2$C$_2$-(CF$_3$)$_2$)], the electronic structures of the metalladichalcogenolenes have provoked a great deal of controversy. The actual structure can be best visualized as a resonance hybrid of the two extremes, olefinic and dithioketonic forms (Fig. 1). The metalladichalcogenolene ring, consisting of one metal cation, two ligating chalcogen atoms and a carbon–carbon double bond, is responsible for the interesting physicochemical properties and for the reactivity of the chelate.

Recently, we have noted that these chelates present

contradictory characters: the aromatic character of the ring and the double-bond character of the metal-chalcogen bonds. The former is demonstrated by such typical electrophilic substitution reactions on the ring as Friedel–Crafts acylation, bromination, nitration and H–D exchange [2] and the latter by the addition of dimethyl acetylenedicarboxylate [3], quadricyclane [4] and several alkylidenes generated from diazo compounds [5–7]. Another interesting feature is that the central metal atom is coordinatively unsaturated and can accept an additional ligand or ligands. Henderson et al. [8] isolated several adducts between [Co(Cp)(S$_2$-C$_2$(CN)$_2$)] and phosphines or phosphites.

Tertiary phosphines and phosphites are known to be useful reagents for examining the coordinative unsaturation of central metal atoms. Their coordination chemistry is well documented [9]. The bonding between the transition metal and the ligating phosphorus atom is regarded as having σ and π components, and hence the phosphorus atom behaves as both donor and ac-

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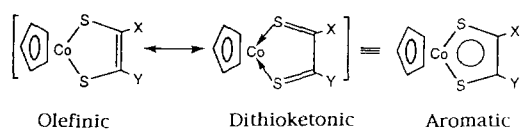Olefinic   Dithioketonic   Aromatic

Fig. 1. Electronic structure of dithiolene ring.

ceptor at the same time [10]. Quantitative evaluation of the σ-donating and π-accepting abilities have been made only recently [11–13].

Here, we report the formation constants in acetonitrile of adducts of η^5-cycopentadienyldithihilato-cobalt(III) complexes having a variety of substituents (X), [Co(Cp)(S$_2$C$_2$X$_2$)], with some phosphines or phosphites. The effect of X on the formation constant was examined in terms of the reversible half-wave potential for the reduction of the unbound complex, $E^r_{1/2}$. The relationships between NMR chemical shifts (^{1}H, ^{13}C and ^{31}P, in CDCl$_3$) and $E^r_{1/2}$ are discussed. The σ-donor and π-acceptor characters of phosphines and phosphites are discussed on the basis of the change of the chemical shifts brought about by the adduct formation. The electronic and structural changes in the adduct formation are also discussed mainly on this basis.

2. Experimental

2.1. Materials

The complex [Co(Cp)(S$_2$C$_2$(CN)$_2$)] was prepared by the method reported by Locke and McCleverty [14]. The other complexes were prepared through a one-pot reaction of [Co(Cp)(CO)$_2$] or [Co(Cp)(1,5-cycloocta-diene)] with an acetylene derivative with suitable substituents and elemental sulphur or selenium according to the method developed by Boennemann et al. [15]. The starting complexes, [Co(Cp)(CO)$_2$] and [Co(Cp)-(1,5-cyclooctadiene)], were prepared as described by King [1] and Boennemann et al. [16]. Since the details of the one-pot synthesis and the characterization of the complexes prepared thereby were reported by the above authors, the preparation of [Co(Cp)(S$_2$C$_2$(COOMe)$_2$)] is described here as a typical example.

Elemental sulphur (10 mmol) was placed in a flask equipped with a reflux condenser, then the system was purged with argon. A 30 cm^3 portion of toluene which had been distilled under argon was introduced into the flask, and then 5 mmol (0.62 mm^3) of dimethyl acetylenedicarboxylate was added. After the mixture had been heated at 70–80°C for 10 min with stirring, 5 mmol (0.70 mm^3) of [Co(Cp)(CO)$_2$] was slowly added. The mixture was maintained at 80°C for 2 h, and then it was refluxed for 2 h. During this procedure the colour of the solution changed from red–brown to

violet. The solvent was then removed by evaporation under reduced pressure. The operations up to this point were carried out under an argon atmosphere. The residue was dissolved in a small quantity of hexane containing a minimal amount of dichloromethane and this was subjected to flash-column chromatography with gradient elution (hexane–dichloromethane) on silica gel (Wakogel C300). Violet fractions were collected. The complex was obtained as a violet powder with a yield of 85% when the solvent was evaporated under reduced pressure.

Four adducts, [Co(Cp)(S$_2$C$_2$(CN)$_2$)(PR$_3$)] with R = C$_4$H$_9$ (Bu), OCH$_3$ (OMe), OC$_2$H$_5$ (OEt), OC$_4$H$_9$ (OBu), were prepared according to the procedure reported by Henderson et al. [8]. The isolated adducts were identified on the basis of the spectral data and the elemental analyses.

The tertiary phosphines and phosphites used were purified by vacuum distillation or recrystallization. They were stored and used under an atmosphere of argon.

Acetonitrile and dichloromethane were spectral grade reagents (Dotite Spectrosol, Dojindo Laboratories). The other chemicals were of reagent grade.

2.2. Instruments

Elemental analyses of hydrogen and carbon were carried out with a Perkin-Elmer Model 240C apparatus.

UV–visible spectrophotometric measurements were performed by means of a Hitachi Model 220-20 spectrophotometer with stoppered 1 cm quartz cells; the cell compartment was thermostated at 25°C.

The NMR spectra were recorded with a JEOL JNM GX-270 spectrometer. The complexes and the isolated adducts were dissolved in deaerated CDCl$_3$ containing TMS; the spectra of the other adducts were measured in situ with equimolar mixtures of a complex and a phosphine or phosphite (each concentration was ca. 60 mmol dm^{-3}).

3. Results and discussion

3.1. Formation of adducts

All the complexes studied here reacted rapidly with phosphines or phosphites to form adducts in acetonitrile, dichloromethane, and other solvents, as was seen from an almost instantaneous colour change from blue or violet to yellow or brown which took place when a solution of a phosphine or phosphite was added to a complex.

Spectrophotometric titrations were carried out in acetonitrile. An example of the spectral change is presented in Fig. 2. The titration curves showed more
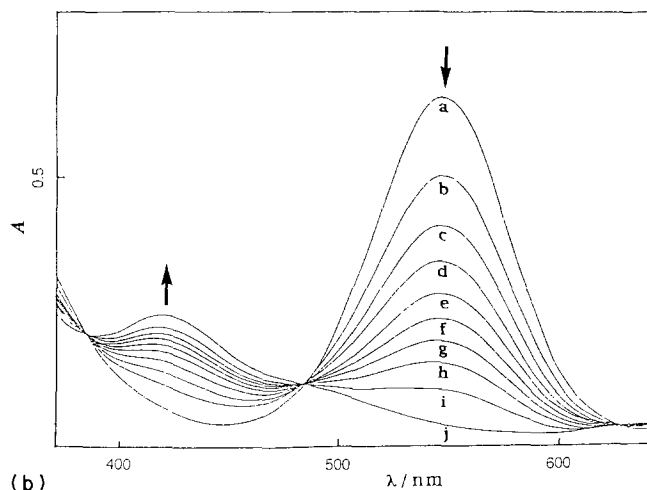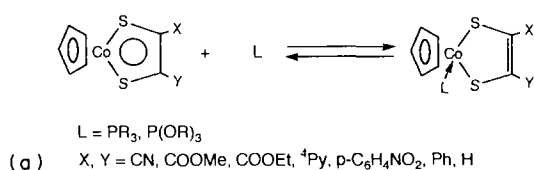
L = PR$_3$, P(OR)$_3$

(a)　X, Y = CN, COOMe, COOEt, ^{4}Py, p-C$_6$H$_4$NO$_2$, Ph, H

(b)

Fig. 2. UV–visible spectra of acetonitrile solutions containing [Co(Cp)(S$_2$C$_2$(COOMe)$_2$)] and P(OMe)$_3$. Initial concentration of the complex = 0.993 mol m^{-3}; concentration of the phosphite added = (a) 0, (b) 0.61, (c) 1.22, (d) 1.83, (e) 2.44, (f) 3.66, (g) 4.27, (h) 6.10, (i) 11.0 and (j) 22.0 mol m^{-3}; optical path length = 1 cm; temperature = 25°C; under an argon atmosphere.

or less distinct breaks at a binding ratio of 1:1, and isosbestic points were present in most cases. These observations indicate that the 1:1 adduct was formed:

$$[Co(Cp)(S_2C_2X_2)] + L = [Co(Cp)(S_2C_2X_2)L]$$
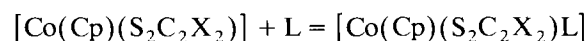
where L represents the ligand (phosphine or phosphite). The corresponding formation constant (concentration basis) of the adduct is defined by

$$K = [YL]/[Y][L] \tag{1}$$

where [L] is the concentration of the unbound ligand, [Y] that of the unbound [Co(Cp)(S$_2$C$_2$X$_2$)] and [YL] that of the adduct. The value of K was determined according to the following equation:

$$(A_0 - A)/(A - A^*)$$
$$= K[c_L - c(A_0 - A)/(A_0 - A^*)] \tag{2}$$

where A is the absorbance of the mixture containing L and the complex at total concentrations c_L and c, respectively, A_0 is the absorbance of a solution containing only the complex at c and A^* is the absorbance of the solution containing only the adduct at concentration c. Experimentally, A^* can be estimated from the absorbance of a solution containing a sufficiently large excess of L.
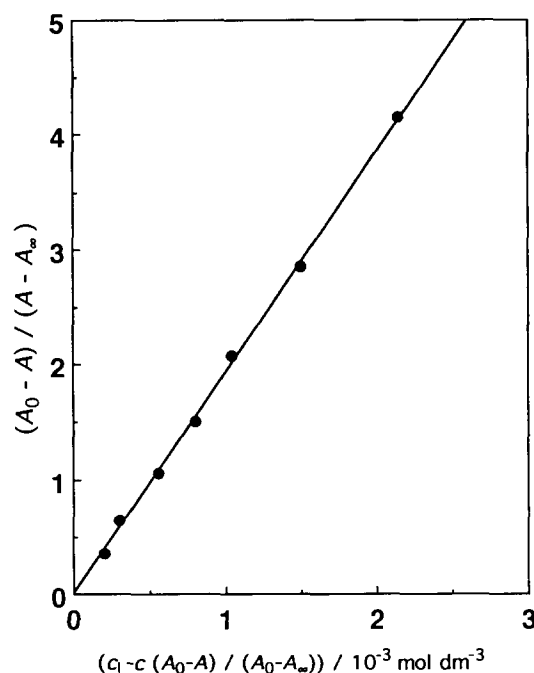

Fig. 3. Plot of Eq. (2). The experimental conditions as in Fig. 2; the absorbance was measured at 547 nm.

When the left-hand side of Eq. (2) is plotted against the quantity in the square brackets on the right-hand side, a straight line passing the origin should result, the slope of which gives K.

Some of the systems examined here gave reasonably linear plots over fairly wide ranges of the titration curves, as shown by the example of Fig. 3. The results for these systems are listed in Table 1 (the values given to three digits). However, the plots for the other systems tended to deviate upwards. Such an upward deviation suggests the formation of some species with more than one L. Attempts were made to explain the curved titration curves by means of curve-fitting methods based on the assumption of the formation of a 1:2 adduct. The results were inconclusive because of the limited experimental accuracy: the titration data did not allow us to determine two adjustable parameters, the forma-

Table 1
Logarithm of formation constants, log(K/mol^{-1} dm^3), of the phosphine and phosphite adducts, [Co(Cp)(E$_2$C$_2$X$_2$)L], in acetonitrile at 25°C

XnL	PMe$_3$	PEt$_3$	PBu$_3$	P(OMe)$_3$	P(OEt)$_3$	P(OBu)$_3$
CN	$-^a$	(5)^c	(5)^c	5.3$_7^b$	5.1$_1^b$	6.0$_7^b$
COOMe	$-^a$	(5)^c	5.2$_9^b$	2.9^c	3.3^c	3.3^c
^{4}Py	(5)^c	4.3$_4^b$	4.0$_7^b$	1.7^c	1.9$_0^b$	1.$_9^c$
Ph	5.3^c	2.$_9^c$	3.2^c	0.55$_9^b$	0.94$_7^b$	0.74$_5^b$

^{a} Not determined.
^{b} Only the 1:1 adduct was detected.
^{c} Estimated from the data for lower c_L; the figures in parentheses are less reliable (see text).

tion constants of the 1:1 and 1:2 adducts, with meaningful certainty. However, they indicated that the K value of the 1:1 adducts could be estimated with reasonable reliability from the linear portion of the titration curve at lower c_L. When the linear portion is too short, however, only a rough estimation is possible. These estimated K values are also included in Table 1.

The K values of the phosphine adducts in most systems are larger than those of the corresponding phosphite adducts. This suggests that the stability of the adduct depends principally on the basicity of the phosphorus ligand rather than on steric factors; for example, the pK_a values (K_a is the acid dissociation constant) of $[HPEt_3]^+$ and $[HPMe_3]^+$ are 8.69 and 8.65 and those of $[HP(OEt)_3]^+$ and $[HP(OMe)_3]^+$ are 3.35 and 2.60 [11], whereas the cone angles of PEt_3 and PMe_3 are 132° and 118° and those of $P(OEt)_3$ and $P(OMe)_3$ are 109° and 107°, respectively [10].

When the logarithm of the formation constant, $\log(K/\text{mol}^{-1}\ \text{dm}^3)$, is plotted against the reversible half-wave potential of the reduction of the cobalt atom of the complex, $E^r_{1/2}(\text{red})$ [17,18], the points lie along an almost straight line for a given phosphorus ligand

(Fig. 4). We have reported previously that the presence of more electron-attracting substituents makes $E^r_{1/2}$ (red) more positive by lowering the energy level of the vacant Co d-orbital [17]. The correlation in Fig. 4 can be interpreted in the same way.

The formation constants of the PMe_3 adducts of the complexes with X = CN and COOMe, which we failed to determine, are expected to be large, in view of the above-mentioned correlation. In fact, trialkylphosphine adducts of $[Co(Cp)(S_2C_2(CN)_2)]$ can be isolated in the crystalline state, as reported by Henderson et al. [8]. Those adducts with smaller formation constants will be very hard, if not impossible, to isolate. Their existence in solutions, however, was indicated in some cases by the presence of ^1H, ^{13}C and ^{31}P NMR signals assignable to the adducts.

3.2. The σ-donor character of phosphine and the π-acceptor character of phosphite

The NMR chemical shifts of the cyclopentadienyl hydrogen and carbon atoms, $\delta(^1H)$ and $\delta(^{13}C)$, of the unbound complexes and the adducts are listed in Table

Table 2
Reversible reduction half-wave potentials [a] and selected ^1H, ^{13}C and ^{31}P NMR shifts in $CDCl_3$ of some $[Co(Cp)(E_2C_2X,X')]$ complexes and their adducts with phosphine or phosphite

No.	X, X'	$E^r_{1/2}(\text{red})$ [a] V	L	Chemical shift/ppm		
				^1H, Cp	^{13}C, Cp	^{31}P [b]
1	CN, CN	−0.59		5.66	81.98	–
1a	CN, CN		PBu_3	5.16	89.19	35.22
1d	CN, CN		PPh_3	5.02		
1e	CN, CN		$P(OMe)_3$	5.28	90.39	120.1
2	COOMe, COOMe	−0.91		5.47	80.68	–
2a	COOMe, COOMe		PBu_3	5.09	88.78	31.03
2b	COOMe, COOMe		$P(Oct)_3$	5.09		
2c	COOMe, COOMe		$PMePh_2$	4.94		
2e	COOMe, COOMe		$P(OMe)_3$	5.28	89.93	123.07
2f	COOMe, COOMe		$P(OEt)_3$	5.17		
2g	COOMe, COOMe		$P(OBu)_3$	5.18		
3	H, COOEt	−1.02		5.36	79.98	–
3a	H, COOEt		PBu_3	5.07	88.59	30.92
3e	H, COOEt		$P(OMe)_3$	5.20	89.95	125.57
4	$^4Py, ^4Py$	−1.03		5.48	80.29	–
4a	$^4Py, ^4Py$		PBu_3	5.11	89.05	31.70
4e	$^4Py, ^4Py$		$P(OMe)_3$	5.24	90.19	126.57
5	Ph, COOEt	−1.04		5.41	80.04	–
5a	Ph, COOEt		PBu_3	5.07	88.53	31.03
5e	Ph, COOEt		$P(OMe)_3$	5.20	89.04	126.13
6	Ph, $p-C_6H_4NO_2$	−1.07		5.38	79.95	–
6a	Ph, $p-C_6H_4NO_2$		PBu_3	5.07	88.89	31.25
6e	Ph, $p-C_6H_4NO_2$		$P(OMe)_3$	5.23	90.15	$-^c$
7	Ph, Ph	−1.16		5.39	79.62	–
7a	Ph, Ph		PBu_3	5.08	$-^c$	$-^c$
7e	Ph, Ph		$P(OMe)_3$	$-^c$	$-^c$	$-^c$
–	–		PBu_3			−30.92
–	–		$P(OMe)_3$			141.31

[a] Corresponding to the reduction of the cobalt atom in the unbound complex; in 0.1 mol dm^{-3} $(C_2H_5)_4NClO_4$–CH_3CN, vs. Ag|0.1 mol dm^{-3} $AgClO_4$, 25°C [17].
[b] External reference, 1% H_3PO_4 in H_2O.
[c] Signal was too weak.

2 together with those of phosphorus atoms, $\delta(^{31}P)$, of the unbound and bound PBu_3 and $P(OMe)_3$.

It is noteworthy that the $\delta(^{31}P)$ values of the phosphine and the phosphite changed in opposite directions: $\delta(^{31}P)$ of PBu_3 was shifted downfield whereas that of $P(OMe)_3$ was shifted upfield. This can be interpreted as reflecting the σ-donor character of phosphines and the π-acceptor character of phosphites.

For a given ligand, the magnitude of the shift of $\delta(^{31}P)$ depends on the electron density at the cobalt atom. An electron-poor cobalt atom attracts the electrons of a σ-donor more strongly, and it gives away electrons to a π-acceptor less easily. As pointed out previously [18], $E^r_{1/2}(red)$, which corresponds to the reduction of the central atom of the unbound complex, is a useful measure of the electron density at the metal site: a more positive $E^r_{1/2}(red)$ indicates a smaller electron density. Hence, a complex with a more positive $E^r_{1/2}(red)$ will bring about a downfield shift of $\delta(^{31}P)$ of PBu_3. The same will be true for $\delta(^{31}P)$ of $P(OMe)_3$ except that the shift is upfield.

The dependence of the changes on $E^r_{1/2}(red)$ was linear, as seen in Fig. 5, where the difference, $\Delta\delta = \delta(^{31}P$ of unbound phosphorus ligand$) - \delta(^{31}P$ of adduct$)$, is plotted against $E^r_{1/2}(red)$ for $P(Bu)_3$ (a) and $P(OMe)_3$ (b).

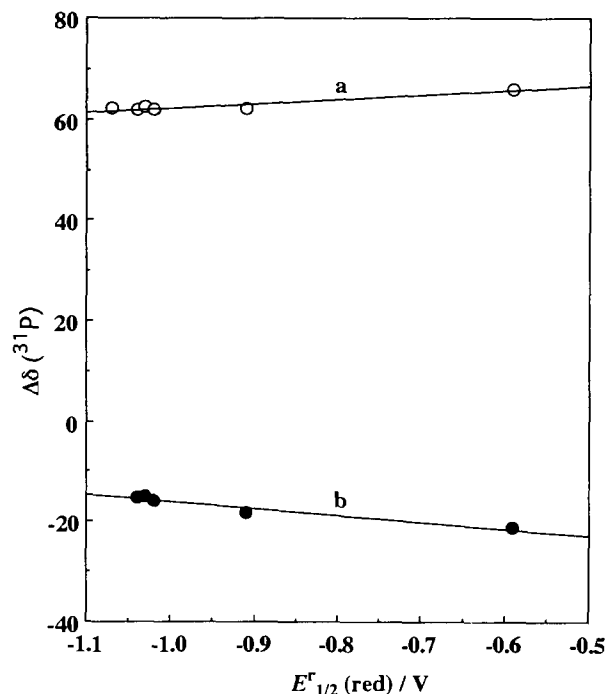The adduct formation increases the electron density of the complex moiety. This is reflected by the changes

Fig. 5. Dependence of the chemical shift displacement ($\Delta\delta$) of ^{31}P on $E^r_{1/2}(red)$ of the unbound complex. (a) PBu_3; (b) $P(OMe)_3$.

in the chemical shift of the cyclopentadienyl protons, $\delta(^1H)$, i.e the adduct formation brings about an upfield shift as seen from Table 2. That the shift was larger in the $P(Bu)_3$ systems than in the $P(OMe)_3$ systems is interpreted as another indication of the π-acceptor character of the phosphite.

3.3. Electronic structural change in adduct formation

In contradistinction to the $C_5^1H_5$ signal, the $^{13}C_5H_5$ signal showed a large downfield shift on adduct formation (Table 2). This shift is attributable to an extensive structural change brought about by the coordination of a phosphorus ligand to the central atom: in an unbound complex, in which the central atom is five-coordinated, the cyclopentadienyl ring is perpendicular to the cobaltaditholene ring [19], but the central atom in the adduct is coordinated in a piano-stool geometry [3,5,7].

Such a structural change affects the environments of the carbon atoms in the cobaltadithiolene ring and that of the proton bound to one of the carbon atoms. The large upfield shifts of the chemical shifts of these atoms (Table 3) can be explained only by essential alterations in the structure of the ring, i.e. from aromatic to olefinic. The chemical shifts of these atoms in the adducts fall in the range of values for olefinic carbon and hydrogen thereon. The olefinic structure of the cobaltadithiolene ring has been inferred by Henderson et al. [8] from the stretching frequency of the carbon–carbon bond in $[Co(Cp)(S_2S_2(CN)_2)(PBu_3)]$.
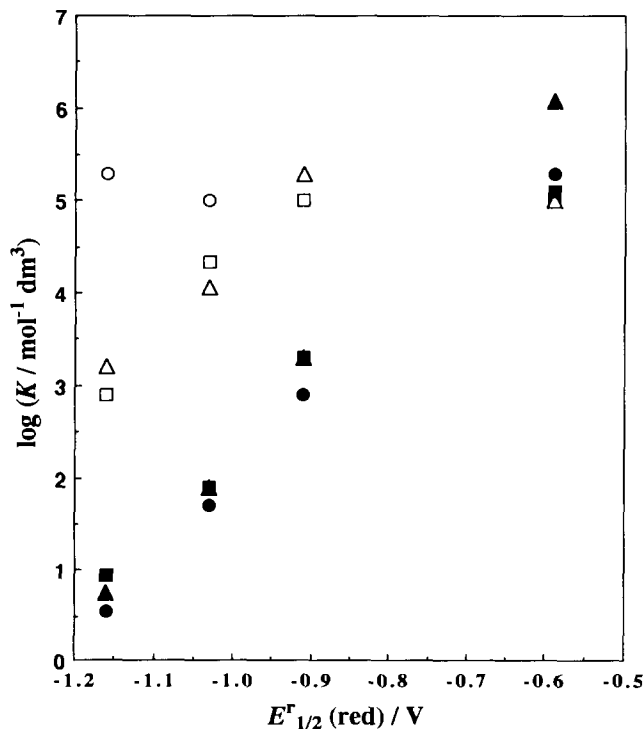

Fig. 4. Dependence of log K for the phosphine and phosphite adducts on $E^r_{1/2}(red)$ of the unbound complex. ○, PMe_3; □, PEt_3; △, PBu_3; ●, $P(OMe)_3$; ■, $P(OEt)_3$; ▲, $P(OBu)_3$.

Table 3
Selected NMR chemical shifts in CDCl$_3$ of ^{13}C and ^{1}H NMR on the dithiolene rings in typical [Co(Cp)(E$_2$C$_2$X,X')] complexes and their adducts with phosphine or phosphite

No.	X, X'	L	Chemical shift/ppm	
			^{13}C	^{1}H
1	CN, CN	–	143.11	–
1a	CN, CN	PBu$_3$	126.32[a]	–
1e	CN, CN	P(OMe)$_3$	126.63[b]	–
2	COOMe, COOMe	–	162.00	–
2a	COOMe, COOMe	PBu$_3$	139.34[c]	–
2e	COOMe, COOMe	P(OMe)$_3$	139.44	–
3	COOEt, H	–	166.95[d], 157.71	9.42
3a	COOEt, H	PBu$_3$	145.12[c], 132.38	6.69[i]
3e	COOEt, H	P(OMe)$_3$	145.56[g], 132.38	6.77[h]

[a] $J(PC) = 2.5$ Hz.
[b] $J(PC) = 2.4$ Hz.
[c] $J(PC) = 1.8$ Hz.
[d] $J(CH) = 176.2$ Hz.
[e] $J(CH) = 171.4$ Hz.
[f] $J(PH) = 6.0$ Hz.
[g] $J(CH) = 168.5$ Hz.
[h] $J(PH) = 5.4$ Hz.

The characteristic UV–visible spectral behaviour on adduct formation is a result of this structural change.

The signal of the dithiolene proton in adduct 3a split into a doublet with $J = 6.0$ Hz; a comparable splitting was observed in the case of 3e. The most plausible cause of the splitting is the coupling with ^{31}P. A similar H-P coupling has been reported in the case of [Fe(CO)(P(OMe)$_3$)$_2$(S$_2$C$_2$(H, COOEt))] by Carty et al. [20].

The structural change in the cobaltadithiolene ring
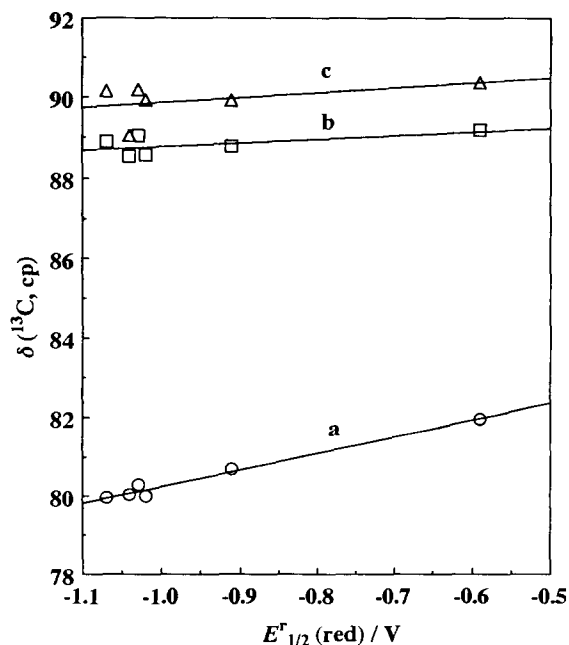

Fig. 6. Dependence of $\delta(^{13}C, cp)$ on $E^r_{1/2}$(red) of the unbound complex. (a) Unbound complexes; (b) PBu$_3$ adducts; (c) P(OMe)$_3$ adducts.

is reflected also be the effect of the subsituents X on the environment of the cyclopentadienyl carbons. In Fig. 6, $\delta(^{13}C, Cp)$ is plotted against $E^r_{1/2}$(red) for a series of complexes. The slopes of the linear regression lines for the phosphine and phosphite adducts (b and c) are appreciably smaller than that for the unbound complexes, indicating again the loss of aromaticity in the adducts.

References

[1] R.B. King, J. Am. Chem. Soc., 86 (1963) 1587.
[2] G. Hagino, M. Sakurada, T. Fujita, M. Tamada, T. Fukuzumi, M. Kajitani, T. Akiyama and A. Sugimori, submitted for publication.
[3] M. Kajitani, T. Suetsugu, R. Wakabayashi, A. Igarashi, T. Akiyama and A. Sugimori, J. Organomet. Chem., 293 (1985) C15.
[4] M. Kajitani, H. Hatano, F. Fujita, T. Okumachi, H. Nagao, T. Akiyama and A. Sugimori, J. Organomet. Chem., 430 (1992) C64.
[5] M. Kajitani, M. Sakurada, K. Dohki, T. Suetsugu, T. Akiyama and A. Sugimori, J. Chem. Soc., Chem. Commun., (1990) 19.
[6] M. Sakurada, J. Okubo, M. Kajitani, T. Akiyama and A. Sugimori, Chem. Lett., (1990) 1837.
[7] M. Sakurada, M. Kajitani, K. Dohki, T. Akiyama and A. Sugimori, J. Organomet. Chem., 423 (1992) 141.
[8] S.D. Henderson, T.A. Stephens and E.J. Wharton, J. Organomet. Chem., 179 (1984) 231.
[9] C.A. McAulife, in G. Wilkinson, R. Gillard and J.A. McCleverty (eds.), Comprehensive Coordination Chemistry, Vol. 2, Pergamon Press, Oxford, 1987, pp. 986–1066.
[10] C.A. Tolman, J. Am. Chem. Soc., 92 (1970) 2953.
[11] M.N. Golovin, M.N. Rahman, J.E. Belmonte and W.P. Giering, Organometallics, 4 (1985) 1981.
[12] A.G. Orpen and N.G. Connelly, Organometallics, 9 (1990) 1206.
[13] H. Liu, E. Eriks, A. Prock and W. Giering, Organometallics, 9 (1990) 1758.
[14] J. Locke and J.A. McCleverty, Inorg. Chem., 5 (1966) 1157.
[15] H. Boennemann, B. Bogdanovic, W. Brijoux, R. Brinkmann, M. Kajitani, R. Mynott, G.S. Natarajan and M.G. Samson, Transition-metal-catalyzed synthesis of heterocyclic compounds in J.R. Kosak (ed.), Catalysis in Organic Reactions, Marcel Dekker, New York, 1984, pp. 31–62.
[16] H. Boennemann, W. Brijoux, R. Brinkmann, W. Meurers and R. Mynott, J. Organomet. Chem., 272 (1984) 231.
[17] M. Kajitani, T. Akiyama, A. Sugimori, K. Hirakata, Y. Hoshino, Y. Satsu, G.P. Satô, K. Shimizu, M. Kaise and C. Nishihara, J. Electroanal. Chem., 251 (1988) 421.
[18] H. Ushijima, M. Kajitani, K. Shimizu, G.P. Satô, T. Akiyama and A. Sugimori, J. Electroanal. Chem., 303 (1991) 199.
[19] M.R. Churchill and J.P. Fennessey, Inorg. Chem., 7 (1968) 1123.
[20] A.J. Carty, P.H. Dixneuf, A. Gorgus, F. Hartstock, H. LeBozee and N.J. Taylar, Inorg. Chem., 20 (1981) 3929.