Journal of Organometallic Chemistry, 184 (1980) 113—124

AN ESR STUDY OF THE REACTIONS OF DECACARBONYLDIMANGANESE, TRIMETHYLTINPENTACARBONYLMANGANESE AND DECACARBONYLDIRHENIUM WITH QUINONES AND α-DIKETONES

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(Received June 22nd, 1979)

Summary

The thermal and photochemical reactions of $Mn_2(CO)_{10}$, $Me_3Sn—Mn(CO)_5$, and $Me_2Sn[Mn(CO)_5]_2$ with various quinones and α-diketones were studied by ESR. The results clearly show that with *p*-quinones the predominant manganese radical adducts are formed via complexation of the metal with the quinonoid π-electron systems. However, a different reaction with *o*-quinones and α-diketones leads to the formation of manganese radical adducts mainly via metal chelation to the carbonyl oxygens. The photolysis of $Re_2(CO)_{10}$ with *o*-quinones also yields the analogous rhenium radical adducts.

Introduction

While reports of the formation of substituted metal carbonyl radicals are numerous, these species have not, in general, been well characterized [1,2]. There have been relatively few direct ESR characterizations of metal carbonyl radicals produced by the photolysis of binuclear metal carbonyls [2,3]. One of the reasons may be that the CO ligands are very labile. For example, extended photolysis of $Mn_2(CO)_8(PBu_3)_2$ in the presence of excess PBu_3 in heptane with periodic removal of CO results in the formation of paramagnetic species, $Mn^{\cdot}(CO)_3(PBu_3)_2$ [2]:

$$Mn(CO)_8(PBu_3)_2 \overset{h\nu}{\rightleftharpoons} 2\ Mn^{\cdot}(CO)_4PBu_3 \xrightarrow[-CO]{+PBu_3} Mn^{\cdot}(CO)_3(PBu_3)_2 \qquad (1)$$

On the other hand, metal carbonyl radicals can be readily trapped to form spin adducts with nitroso compounds [4—7] and TCNE [8].

High resolution ESR spectra of manganese carbonyl radical adducts to *o*-quinones [9] and to α-diketones [10] also have been reported recently. It is generally expected that *o*-quinones and α-diketones will form manganese carbonyl radical adducts via chelation with the carbonyl oxygens of the organic compounds. However, with *p*-quinones complexation via the quinonoid π-electron systems is also possible [11]. Previous studies of the Group IVB organometallic spin adducts to phenanthrenequinone (an *o*-quinone) [9,10,12] and to 2,6-di-tert-butyl-*p*-benzoquinone (a *p*-quinone) [13] indicate that only the carbonyl oxygen(s) of the quinones are involved in complexation.

In this paper we report the ESR studies of the spin adducts derived from the reactions of various *o*- and *p*-quinones and α-diketones with binuclear metal carbonyls and mixed binuclear carbonyls. The results indicate that manganese carbonyl radicals may add to the quinones either via chelation of the two quinone carbonyl oxygens in *o*-quinones and α-diketones or via complexation directly to the quinonoid π-systems in the *p*-quinones.

Experimental

All quinones and α-diketones were available from Aldrich Chemicals and were either vacuum sublimed or distilled before use. Solvents were refluxed over molecular sieves and distilled. $Mn_2(CO)_{10}$ (Pressure Chemicals), $Re_2(CO)_{10}$ and $Co_2(CO)_8$ (Alfa Chemicals) were used as received. Trimethyltinpentacarbonylmanganese was a gift from Professor B.K. Hunter.

A typical sample containing 1 cm^3 of solvent and 5 mg each of the quinone and the organometallic compound to be studied was degassed and sealed off under high vacuum at liquid nitrogen temperature. The frozen sample was allowed to warm to the desired temperature (usually 20°C, unless otherwise specified) in an ESR cavity. The temperature was monitored by a potentiometer equipped with a copper-constantan. The spectra were recorded on a Bruker 420 X-band spectrometer with 100 kHz modulation and equipped with a Varian variable temperature controller. Magnetic field and microwave frequency were measured with a Bruker NMR oscillator and a Hewlett-Packard 5342 frequency counter, respectively. The light source was either a 200 W super mercury lamp using pyrex optics or a Molectron 1 megawatt N_2-pulsed laser. The time-resolved DC detection CIDEP observation system assembled similar to that reported by Kim and Weissman [14] was used to check the polarization of the observed paramagnetic species.

Results and discussion

1. $Mn(CO)_5$ spin adduct to a p-quinone, 2,6-di-tert-butyl-p-benzoquinone

When a degassed frozen benzene solution of $Mn_2(CO)_{10}$ and 2,6-di-tert-butyl-*p*-benzoquinone (DTBQ) was allowed to thaw in the ESR cavity, a weak ESR signal was observed. With the incidence of radiation from a pulsed nitrogen laser, the intensity of the signal was greatly enhanced (Figure 1a). This complex ESR spectrum is the superposition of the spectra of the DTBQ radical ion [15], and of another species. A large excess of $Mn_2(CO)_{10}$ suppresses the $DTBQ^{-\cdot}$ lines yielding the undistorted spectrum of the second species (Figure 1b). The spectrum exhibits 6 groups of hyperfine lines due to manganese (^{55}Mn, $I = 5/2$) and the Mn sextet is further split into a large doublet and a small multiplet. The intensity ratio within the small multiplet can be well accounted for by the nine

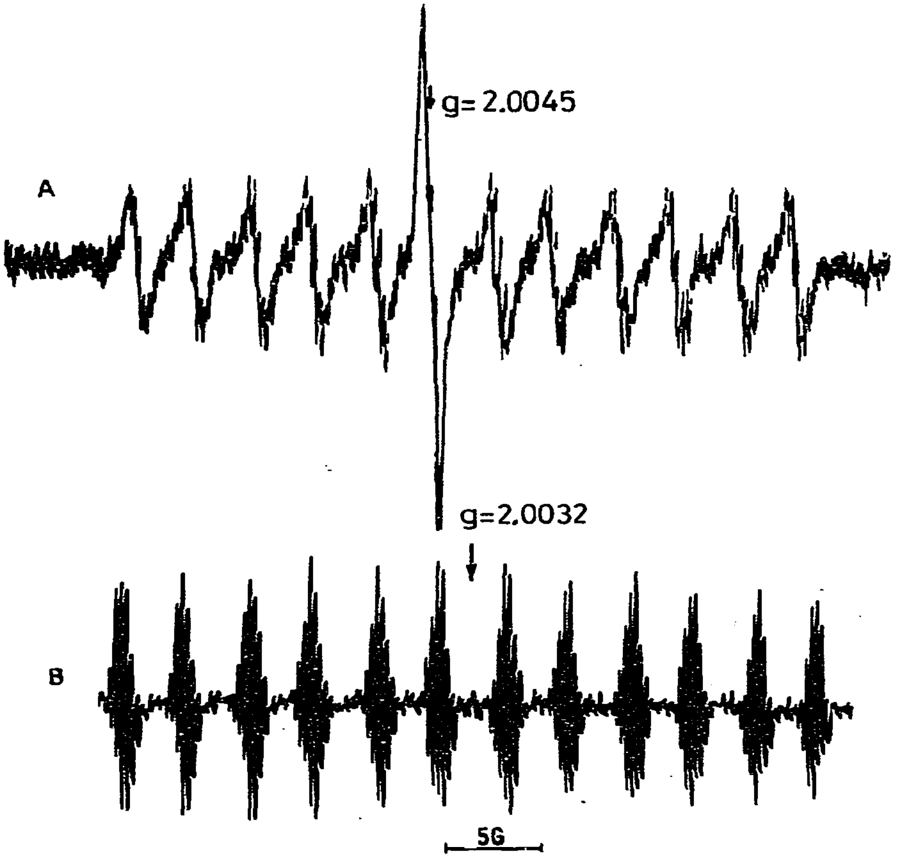
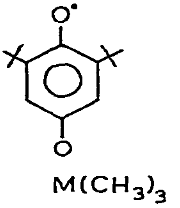


Fig. 1. The ESR spectra observed during the photolysis of $Mn_2(CO)_{10}$ and DTBQ in a benzene solution at 22°C, (a) [DTBQ] > $[Mn_2(CO)_{10}]$, (b) [DTBQ] << $[Mn_2(CO)_{10}]$

protons of one tert-butyl group. When other *p*-quinones including 2,6-di-methyl-*p*-benzoquinone (DMBQ), 2-methoxy-*p*-benzoquinone, and 2,5-di-tert-butyl-*p*-benzoquinone were used, similar phenomena were observed. The ESR parameters of the species with manganese splittings were given in Table 1.

The Group IVB organometallic centered radicals were added to the carbonyl oxygen of DTBQ, and a spin adduct of the following general structure was assigned from the observed triplet *meta*-proton splittings [13]. However, the



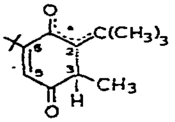
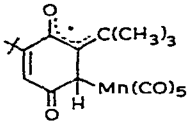
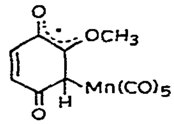
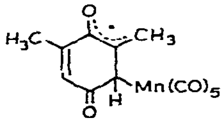
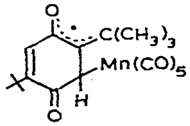
M = C, Si, Ge, Sn and Pb

(I)·

observation of proton hyperfine splittings only from one single proton and one tertiary butyl group or one methyl group in the reactions of $Mn_2(CO)_{10}$ with

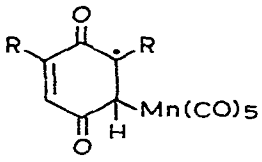
TABLE 1

ESR PARAMETERS OF Mn ADDUCTS TO *para*-QUINONES AT 20°C.

Quinone Adduct	g-factor	a_{Metal}	a_2^{H}	a_3^{H}	a_5^{H} (G)
	2.0039	—	0.36 [a]	0.36(methyl) 10.20(H)	
	2.0032	7.13	0.30 [a]	3.38	
		5.34	0.98 (quartet)	4.49	0.83
	4.98	5.52 (quartet)	1.51	0.30	
		6.49		3.79	

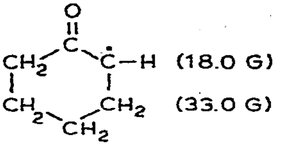
[a] Hfs from the t-butyl group.

DTBQ or DMBQ, respectively, led us to consider the "α-keto" type radicals as a possible structure for the manganese spin adducts:

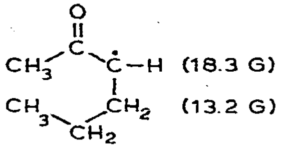


(II) R = t-Bu and Me

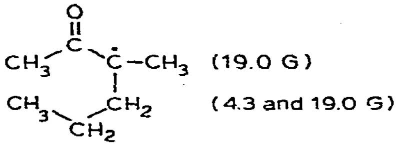
The α-keto radicals have been studied extensively by Pratt et al., [16]. Although a large hyperfine splitting from a pair of magnetically equivalent β-protons was observed for the 2-cyclohexanoyl radical (III), the magnitude of the β-protons splitting is dramatically reduced by "ring-opening" (IV). Substitution of an α-methyl group not only reduces the hyperfine splittings but also introduces magnetic inequivalence of β-protons (V):



(III)

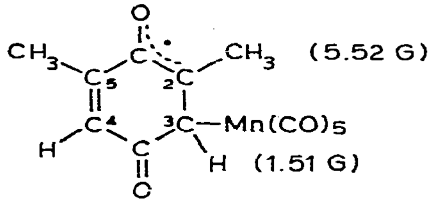


(IV)



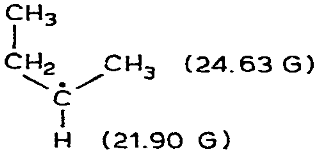
(V)

The α-proton or methyl proton splittings do not change in a similar fashion to that of β-protons with the "ring-opening" or α-methyl group substitution, and they are retained at the magnitude of 18—19 G. Since it has been shown that the β-substitution of Group IVB organometallic groups on the ethyl radical only alters the α-proton splitting slightly (22—19.7 G) [17], The observed much smaller α-methyl proton splittings (5.52 G) of the manganese adduct of DMBQ favor the "allylic" structure (VI) even though only 30% of allylic character of

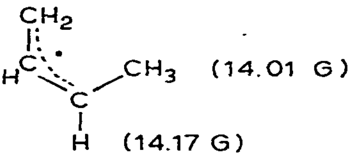


(VI)

2-cyclohexanoyl radical was determined from the observed oxygen-17 and carbon-13 hyperfine splittings [16]. The delocalization of the unpaired electron through the "allylic" structure can indeed reduce the methyl proton hyperfine splitting dramatically [18,19].



(VII)



(VIII)

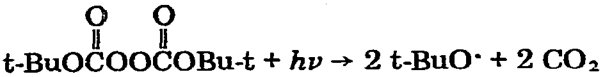
Based on the "allylic" structure VI, one can estimate the dihedral angle θ between the C_3—H bond and the odd electron orbital at carbon 2 from the general expression (2) [20,21] with the assumption that the substitution of the

$$\theta = \cos^{-1}\left(\frac{Q_{3,\mathrm{H}}Q}{Q_{2,\mathrm{H}}B}\right)^{1/2}, \text{ where } \left|\frac{B}{Q}\right| = 2.401 \qquad (2)$$

methyl group on the allyl radical does not change the spin distribution significantly [19]. The determined θ = 70° indicates that the carbon—manganese bond is essentially eclipsed with the π-orbital on carbon-2 (which contains the odd electron). The comparable manganese splittings observed in the manganese adducts of p-quinones with respect to that of $Mn^{\cdot}(CO)_3(PBu_3)_2$ (7 G at 298 K) [2] may indicate the involvement of p—d homoconjugation with the preferred eclipsed conformation. The observed unusually large metal splittings of β-Group

IVB organometallic substituted ethyl radicals have been ascribed to this effect [17].

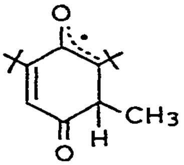
The ability of DTBQ to trap free radicals via its quinonoid π-system has also been demonstrated by the photochemical reaction of di-t-butylperoxyoxalate with DTBQ, which led to the formation of a radical adduct IX [22]:



$$\text{t-BuO}\overset{\overset{O}{\|}}{C}\text{OO}\overset{\overset{O}{\|}}{C}\text{OBu-t} + h\nu \rightarrow 2\ \text{t-BuO}^{\cdot} + 2\ CO_2 \qquad (3)$$

$$\text{t-BuO}^{\cdot} \rightarrow (CH_3)_2CO + \dot{C}H_3 \qquad (4)$$

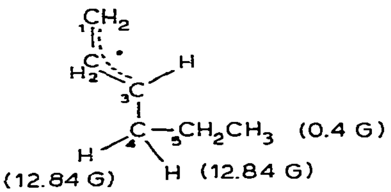
$\dot{C}H_3$ + DTBQ → (5)



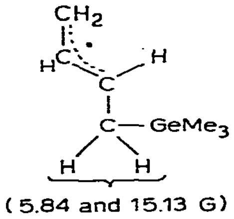
(IX)

We have further carried out a series of photolyses of other peroxides including t-BuOOBu-t, $\text{Ph}\overset{\overset{O}{\|}}{C}\text{OO}\overset{\overset{O}{\|}}{C}\text{Ph}$, and $\text{Ph}\overset{\overset{O}{\|}}{C}\text{OOBu-t}$ in the presence of DTBQ. In each case, a radical adduct similar to IX was observed.

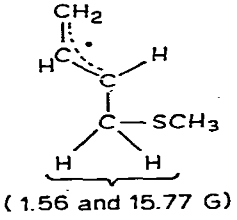
The assignment of the "allylic" structure of the manganese adducts of *p*-quinones is further supported by the comparison of the observed hyperfine splittings of both π-quinonoid spin adducts and the allyl radicals derived from the addition of Group IVB organometallic centered radicals to butadiene [23].



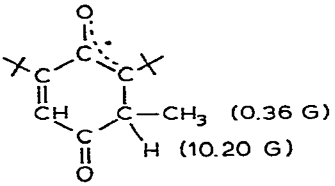
(X)



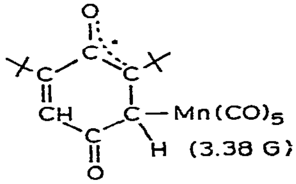
(XI)



(XII)



(IX)



(XIII)

For the ethyl adduct of butadiene (X), both proton hyperfine splittings from carbons 4 and 5 (12.84 G, 0.4 G) are comparable with those of adduct IX (10.20 G, 0.36 G). The inequivalence of the two protons on carbon 4 and the dramatical decrease of one proton splitting from carbon 4 can indeed be induced by introducing the heteroatom into the carbon 4. With the methylthiol group, one proton splitting does go down to a very small value of 1.56 G (XII).

With the assignment of the manganese adducts of *p*-quinones together with the observation of $DTBQ^{-\cdot}$, a charge-transfer process, both thermally and photochemically accessible, followed the trapping of $Mn^{\cdot}(CO)_5$ by a *p*-quinone, can account for the formation of manganese π-quinonoid adducts:

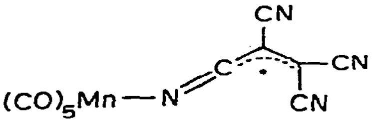
$$Q + (CO)_5MnMn(CO)_5 \overset{h\nu \text{ or } \Delta}{\rightleftharpoons} [Q^{-\cdot}(CO)_5\overset{+\cdot}{Mn}Mn(CO)_5] \quad (6)$$

$$[Q^{-\cdot}(CO)_5\overset{+\cdot}{Mn}Mn(CO)_5] \to \underset{\text{Observed}}{Q^{-\cdot}} + (CO)_5\overset{+\cdot}{Mn}Mn(CO)_5 \quad (7)$$

$$(CO)_5Mn\overset{+\cdot}{Mn}(CO)_5 \to \dot{Mn}(CO)_5 + Mn(CO)_5^+ \quad (8)$$

$$\dot{Mn}(CO)_5 + DTBQ \to (XIII) \quad (9)$$

A similar mechanism, except thermally accessible, has been suggested for the reaction of $Mn_2(CO)_{10}$ with TCNE to form an iminotricyanoallyl radical (XIV) [8].



(XIV)

Since a strong absorption at 340 nm was assigned to the excitation of a Mn—Mn σ-bonding electron into the corresponding antibonding orbital of $Mn_2(CO)_{10}$ [24], the formation of $\dot{Mn}(CO)_5$ through this excitation by the nitrogen laser can-not be completely excluded. Time-resolved CIDEP studies did not give any indication of polarized $DTBQ^-$ or manganese adduct.

The addition of $Mn^{\cdot}(CO)_5$ to the quinonoid π-system of DTBQ is not in itself surprising. However, it is interesting to note that a previous study [13] showed that Group IVB metal centered radicals add to DTBQ exclusively via the carbonyl oxygen of the quinone to yield radical I. This bifunctional behaviour of DTBQ afforded an interesting study when the mixed binuclear compound $Me_3SnMn(CO)_5$ was photolyzed in the presence of DTBQ. Both adducts I (with M = Sn) and XIII were observed, but the intensity of the Sn adduct I was much stronger than that of the Mn adduct XIII at room temperature. These results can be accounted for by a similar chargetransfer mechanism:

$$R_3SnMn(CO)_5 + DTBQ \overset{h\nu}{\to} [R_3Sn\overset{+\cdot}{Mn}(CO)_5\, DTBQ^{-\cdot}] \quad (10)$$

$$[R_3Sn\overset{+\cdot}{Mn}(CO)_5 + DTBQ\overset{+\cdot}{}] \to R_3Sn\overset{+\cdot}{Mn}(CO)_5 + DTBQ^{-\cdot} \quad (11)$$

$$R_3Sn\overset{+\cdot}{Mn}(CO)_5 \begin{cases} \to R_3Sn^{\cdot} + Mn(CO)_5^{+} & (12a) \\ \to R_3Sn^{+} + \dot{Mn}(CO)_5 & (12b) \end{cases}$$

$$R_3Sn^{\cdot} + DTBQ \to (I) \quad (13)$$

$$(CO)_5Mn^{\cdot} + DTBQ \to (XIII) \quad (14)$$

The competition in the fission of the radical cation $R_3Sn\overset{+\cdot}{Mn}(CO)_5$ into either

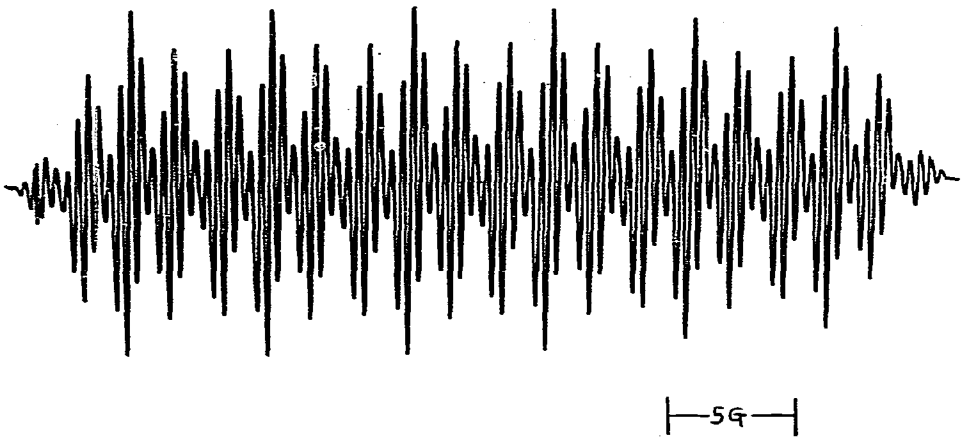


Fig. 2. The ESR spectra of a radical adduct formed by the photolysis of $Mn_2(CO)_{10}$ and PQ in benzene solution at 22°C.

$R_3Sn^{\cdot}$ or $(CO)_5Mn^{\cdot}$ together with the rate of formation and stability of adducts will determine the signal intensity ratio of the adducts I and XIII.

2. *Phenanthrenequinone spin adducts of Group VII metal carbonyl radicals*

When a degassed and frozen benzene solution containing 9,10-phenanthrenequinone (PQ) and $Mn_2(CO)_{10}$ was allowed to warm slowly within the ESR cavity, a typical ESR spectrum as shown in Figure 2 was observed. The spectrum exhibits 6 groups of hyperfine lines due to manganese and each group was resolved into two binomial quintets by the interactions with 2 pairs of 4-equivalent protons. An identical spectrum was also obtained when the solution was irradiated by light. The radical is assigned to the spin adduct of $Mn^{\cdot}(CO)_4$ to PQ probably via the following mechanism:

$$\dot{Mn}(CO)_5 + PQ \xrightarrow{-CO} P\dot{Q}\text{—}Mn(CO)_4 \quad (15)$$

The formation of $\dot{Mn}(CO)_5$ could be either a charge-transfer process followed by a homolysis of the resulting cation radical (Equations 6, 7 and 8) or a direct dissociation of Mn—Mn bond by the light [24].

The ESR parameters (Table 2) suggest a structure for the adduct in which Mn

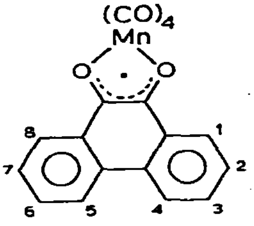
TABLE 2

ESR PARAMETERS OF METAL CARBONYL RADICAL ADDUCTS TO PQ AT 22°C. [a]

Metal	Solvent	g-factor	a_{Metal}	$a^{H}_{1,8}$	$a^{H}_{2,7}$	$a^{H}_{3,6}$	$a^{H}_{4,5}$ (Gr)
$Mn(CO)_4$	C_6H_6	2.0038 (±0.0001)	5.50 g (±0.02)	1.72	0.40	1.72	0.40
$Re(CO)_4$	C_6H_6	2.0031	22.23	—	—	—	—
Na^{+} [b]	THF	2.0046	0.48	1.43	0.28	1.72	0.43
None	DMF	2.0049	—	1.34	0.21	1.67	0.42

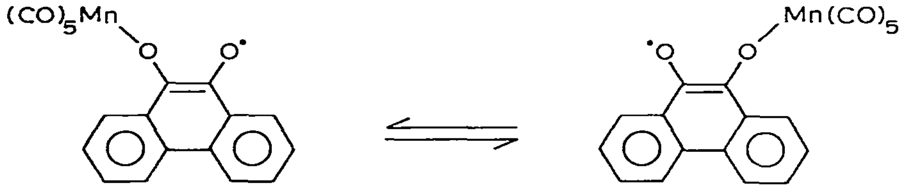
[a] The numbering of protons in PQ is according to the system in structure XV. [b] Taken from the ref. 27.

is chelated to both carbonyl oxygens of the quinone as represented by XV:



(XV)

The assignment was made taking into account the facile dissociative loss of CO from the primary $\dot{\mathrm{M}}\mathrm{n(CO)_5}$ radical [2] and the preferred 6-coordination of Mn [25]. The alternate assignment of structure XVI for the $\dot{\mathrm{M}}\mathrm{n(CO)_5}$ adduct to PQ would require a rapid equilibrium between the two unsymmetrical configurations:



(XVI) (XVI')

There is no experimental evidence for such an equilibrium as linewidth alternation in the ESR spectrum was not observed even over a relatively wide range of temperature (between —60 to +40°C) in toluene.

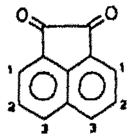
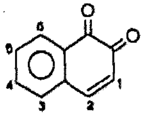
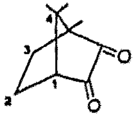
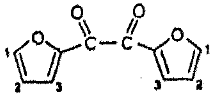
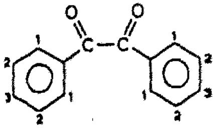
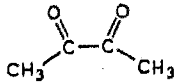
In another series of experiments $Mn_2(CO)_{10}$ was replaced by $(CH_3)_3SnMn(CO)_5$. The photolysis led to the observation of an interesting ESR spectrum which is the superposition of the two individual spectra of the tin and the manganese adducts. The tin—PQ adduct has previously been generated by using $Sn_2(CH_3)_6$ [9,12]. This unique feature of PQ being able to trap simultaneously both the Mn and Sn radicals makes PQ a potential spin trap for radical reactions of mixed binuclear metallic compounds. However, when a trinuclear compound, $(CH_3)_2Sn[Mn(CO)_5]_2$, was irradiated in the presence of PQ, only the Mn adduct was observed.

The photolysis of $Re_2(CO)_{10}$ in the presence of PQ led to the observation of an ESR spectrum showing a broad sextet due to intereaction with the two isotopes ^{185}Re (37.07%, $I = 5/2$) and ^{187}Re (62.93%, $I = 5/2$). Since the magnetic moments of these isotopes differ only by about 1%, they were not resolved in the broad spectrum (line width = 6.5 Gr). Indeed, the proton hyperfine splittings due to the PQ moiety were also unresolved. This ESR spectrum is assigned to the adduct $(CO)_4Re$—PQ and the spectral parameters are given in Table 2. Irradiation of $Co_2(CO)_8$ in the presence of PQ did not give any ESR signal and the solution turned to a green color with the formation of a black precipitate.

Several different reaction mechanisms, including a charge-transfer process, have been proposed [9,10] to account for the observations of the Mn adducts to PQ and α-diketones. In the case of $(CH_3)_3SnMn(CO)_5$, which is relatively

TABLE 3

ESR PARAMETERS OF Mn, Ge, Sn, AND Tl RADICAL ADDUCTS OF ORTHOQUINONES AND α-DIKETONES.

Organic Moiety	Metal	Solvent	a_{Metal}	a_1^H	a_2^H	a_3^H	a_4^H	a_5^H	a_6^H (Gr)	Notes
	$Mn(CO)_4$	Toluene	3.92	1.06	0.26	1.28				
	$TlPh_2$	Dioxane	5.15	1.15	0.29	1.32				*a*
	$GePh_3$			1.92	0.55	1.82				*b*
	$Sn(CH_3)_3$		3.12	1.47	0.47	1.55				*b*
	$Mn(CO)_4$	Toluene	5.97	0.69	4.22	—	1.55	—	1.55	
	$TlPh_2$	Dioxane	11.78	0.53	4.31	0.17	1.50	0.10	1.45	*a*
	$Mn(CO)_4$	Toluene	3.85	2.49	3.09	3.09	0.69			
	$Mn(CO)_4$	Toluene	5.20	1.78	0.38	2.11				
	$TlPh_2$	Dioxane	6.08	1.48	0.40	1.97				*a*
	$SnPh_3$	THF	5.62	2.26	0.39	2.65				
	$Mn(CO)_4$	Toluene	5.81	0.91	0.41	1.36				
	$TlPh_2$	Pyridine	9.05	0.87	0.40	0.95				*a*
	$GePh_3$			2.52	1.00	2.71				
	$Mn(CO)_4$	Toluene	5.22	7.60 (Septet)						

[a] Taken from ref. 28. [b] Taken from ref. 12. [c] Taken from ref. 29.

stable to pyrolysis up to 130°C, it is conceivable that the photochemical reaction with PQ involves the optical excitation to a charge-transfer complex which subsequently disproportionates into Sn and Mn adducts simultaneously:

$$R_3SnMn(CO)_5 + PQ \xrightarrow{h\nu\ (CT)} [R_3SnMn(CO)_5^{+\cdot} PQ^{-\cdot}] \qquad (16)$$

$$[R_3SnMn(CO)_5^{+\cdot} PQ^{-\cdot}] \rightarrow R_3Sn\text{—}\dot{P}Q + \dot{M}n(CO)_5 \qquad (17)$$

$$\dot{M}n(CO)_5 + PQ \xrightarrow{-CO} P\dot{Q}\text{—}Mn(CO)_4 \qquad (15)$$

The ESR parameters; the small metal splittings, the lower *g*-factors, the narrow line-width (0.17 G) and the absence of appreciable magnetic anisotropies all indicate that the unpaired electron in the Sn and Mn adducts is mainly in the PQ moiety. This can be accounted for by the above charge-transfer complex mechanism. However, it must be stressed that chemical evidence for the charge-transfer mechanism is still lacking. Nevertheless, it has been reported [26] that $(CH_3)_3SnMn(CO)_5$ is stable to heat and photolysis and it does not react with in C_2F_4 in the same manner as $Sn_2(CH_3)_6$. For example, Clark and Tsai [25] have shown that the reaction of C_2F_4 with $Sn_2(CH_3)_6$ proceeds by way of a free radical mechanism involving the $R_3\dot{S}n$ radical, whereas a concerted four-centre insertion reaction is found for $R_3SnMn(CO)_5$.

3. Mn(CO)$_4$ spin adducts to other o-quinones and α-diketones.

$Mn_2(CO)_{10}$ was photolysed in a benzene solution and in the presence of a number of *o*-quinones and *α*-diketones, including biacetyl, benzil, furil, and camphoquinone. In each case the corresponding Mn adduct was observed and their ESR parameters are given in Table 3. All of the adducts have a relatively long half-life of a few minutes when the photolyses were terminated. In all of the adducts the ESR parameters are consistent with a chelate structure XV. Most of the Mn a adducts have smaller proton splittings compared to those of the Ge and Sn adducts (Table 3). However, the proton splittings in the Mn adducts are larger than those of Tl adducts (Table 3). Since both Mn and Ge are in the same row of the periodic table, the abrupt drop in the proton splittings in the Mn adducts may indicate some involvement of the empty *d*-orbitals of Mn in delocalization of the unpaired electron.

Conclusion

Manganese carbonyl radicals add to *ortho*-quinones and *α*-diketones via the formation of a chelate with the two carbonyl oxygens of the ketones or quinones. The reaction is markedly different when *para*-quinones are present. Manganese carbonyl radicals add directly to the quinonoid π-system of the *para*-quinones by forming an aryl-manganese bond.

Acknowledgements

This research is supported by a grant from the Natural Sciences and Engineering Research Council of Canada. The authors are most grateful to the referees for their constructive comments.

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