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Oxidative dehydrodimerization of manganese phenylvinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(H)Ph. X-ray structure of phenyl(trityl)vinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(CPh$_3$)Ph

Lyudmila N. Novikova, Mikhail G. Peterleitner, Karine A. Sevumyan, Oleg V. Semeikin, Dmitri A. Valyaev, Nikolai A. Ustynyuk *, Viktor N. Khrustalev, Lyudmila N. Kuleshova, Mikhail Yu. Antipin

A.N. Nesmeyanov Institute of Organoelement Compounds (INEOS), Russian Academy of Sciences, Vavilov Street 28, Moscow 119991, Russian Federation

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Abstract

The oxidatively induced dehydrodimerization of the manganese vinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(H)Ph (**I**) to the bis-vinylidene compound (η^5-C_5H_5)(CO)$_2$Mn=C=C(Ph)–C(Ph)=C=Mn(CO)$_2$(η^5-C_5H_5) (**II**) proceeds via C_β–H bond homolysis in the radical cation (**I**$^{+\bullet}$) and is thought to involve the formally 16-electron mononuclear σ-phenylethynyl cation [(η^5-C_5H_5)(CO)$_2$Mn–C≡CPh]$^+$ (**III**) and the bis-carbyne dication [(η^5-C_5H_5)(CO)$_2$Mn≡C–C(Ph)=C(Ph)–C≡Mn(CO)$_2$(η^5-C_5H_5)]$^{2+}$ (**II**$^{2+}$) as the key sequential intermediates. Compound **II**$^{2+}$ was characterized by IR and CV. The reduction of **II**$^{2+}$ with (C_6H_6)$_2$Cr gave **II** in an 80% yield. Compound **II** can be prepared more conveniently by treating **I** with (C_5H_5)$_2$FeBF$_4$ in the presence of Et$_3$N. The interaction of **I** with Ph$_3$CPF$_6$ yields the trityl(phenyl)vinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(CPh$_3$)Ph (**IV**) as a result of the electrophilic attack of the trityl cation on C_β followed by the loss of a proton. The structure of **IV** was established by an X-ray structural analysis. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Oxidative dehydrodimerization; Manganese phenylvinylidene complexes; X-ray structure

1. Introduction

Oxidatively induced ligand-to-ligand dimerization of transition metal complexes has become a common procedure for the formation of carbon–carbon bonds. Reactions of this type are under active study and a number of examples of dimerizations and dehydrodimerizations are now known in which complexes couple either through multicenter polyenyl [1] or through C_β (rarely C_γ) atoms of σ-vinyl [2], σ-ethynyl [3], σ-allyl [4], and vinylidene [5] ligands. Nevertheless, the general regularities and governing factors of these processes are still far from being completely understood and their systematic study therefore remains a critical task.

In this work, the oxidatively induced dehydrodimerization of the manganese phenylvinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(H)Ph (**I**) to the binuclear bis-vinylidene complex (η^5-C_5H_5)(CO)$_2$Mn=C=C(Ph)–C(Ph)=C=Mn(CO)$_2$(η^5-C_5H_5) (**II**) has been studied. Metallacumulene systems which are structurally similar to **II** have attracted considerable attention recently because rod-like conductors [6] as well as new materials with given non-linear optical [7a,b] and liquid-crystalline [8a,b] properties can be obtained on their basis.

1.1. Cyclic voltammetry study of oxidation of I and II

Electrochemical oxidation of **I** and **II** was studied by CV at a glassy carbon electrode in methylene chloride solution. The cyclic voltammogram of the bis-vinylidene complex **II** (Fig. 1a) showed two reversible one-electron oxidation peaks at $E_p^{a1} = +0.48$ V (peak A)

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* Corresponding author. Tel.: +7-95-135-5064; fax: +7-95-135-5085.

E-mail address: ustynyuk@ineos.ac.ru (N.A. Ustynyuk).

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and $E_p^{a2} = +0.72$ V (peak B) indicating stability of **II**$^+$ and **II**$^{2+}$ on the CV time scale.

Unlike **II**, the mononuclear complex **I** displays an irreversible one-electron oxidation peak at more positive potential $E_p^a = +0.80$ V (Fig. 1b, peak C). Reverse scanning of potential revealed new cathodic peaks B′ (+0.64 V vs. SCE) and A′ (+0.40 V vs. SCE) that coincide completely with reduction peaks of binuclear complexes **II**$^{2+}$ and **II**$^+$. Thus, the radical cation **I**$^{+\bullet}$ undergoes conversion to the dication **II**$^{2+}$ even on the CV time scale. We propose that this conversion proceeds (Scheme 1) through C_β–H bond homolysis followed by dimerization of the formally 16-electron complex **III**$^+$. Precedents for the dimerization of 16-electron σ-ethynyl transition metal complexes have been reported [3c].

The cyclic voltammogram of oxidation of **I** in the presence of triethylamine shows the oxidation peak of the latter (D) at +1.02 V as well as the reduction peaks of **II**$^{2+}$ (B′, +0.64 V vs. SCE) and **II**$^+$ (A′, +0.40 V vs. SCE), that are somewhat increased in their intensity compared to the same peaks observed in the absence of amine (Fig. 1c). The oxidation potentials of **I** (+0.80 V vs. SCE) and Et_3N (+1.02 V vs. SCE) are rather close so that joint generation of **I**$^{+\bullet}$ and $\{Et_3N\}^{+\bullet}$ on the electrode surface seems quite possible. As $\{Et_3N\}^{+\bullet}$ is known [9] to abstract hydrogen radicals from organic solvents it should accelerate the hydrogen abstraction from **I**$^{+\bullet}$ in CH_2Cl_2 (see Eq. (1)).

$$\mathrm{I}^{+\bullet} + \{\mathrm{NEt_3}\}^{+\bullet} \rightarrow \underset{\mathrm{III}^+}{[(\eta^5\text{-}C_5H_5)(CO)_2Mn - C \equiv CPh]^+} + [Et_3NH]^+ \quad (1)$$

1.2. Preparative oxidation of *I*. Synthesis of bis-vinylidene complex *II*

The results of our CV study of oxidation of **I** described above indicate that in methylene chloride, the major pathway of decomposition of **I**$^{+\bullet}$ is the homolysis of C_β–H bond followed by the dimerization of the 16-electron cation **III**$^+$ to the bis-carbyne dication **II**$^{2+}$ (Scheme 1).

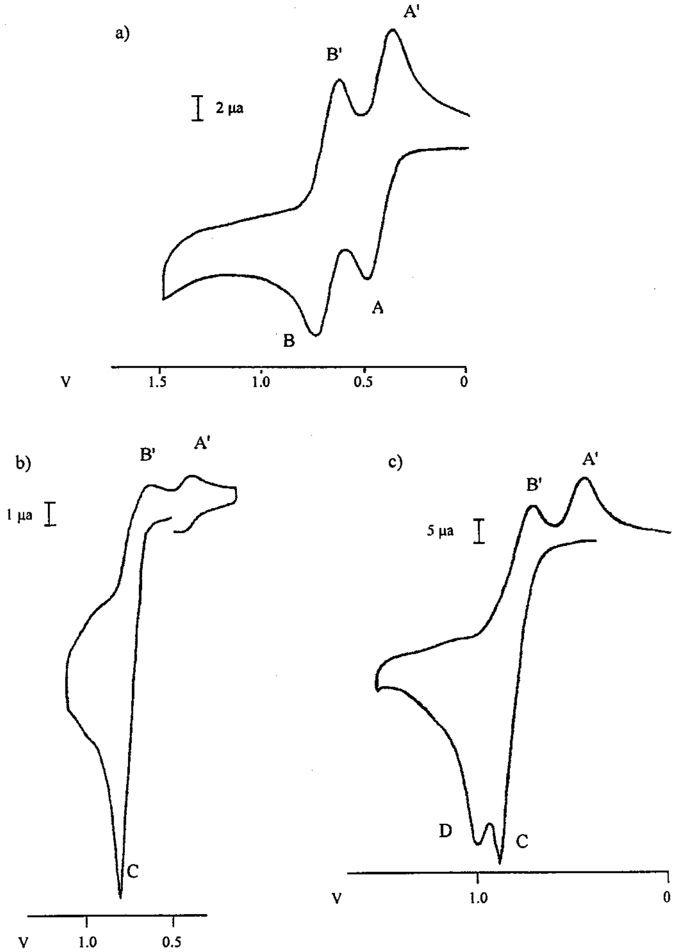
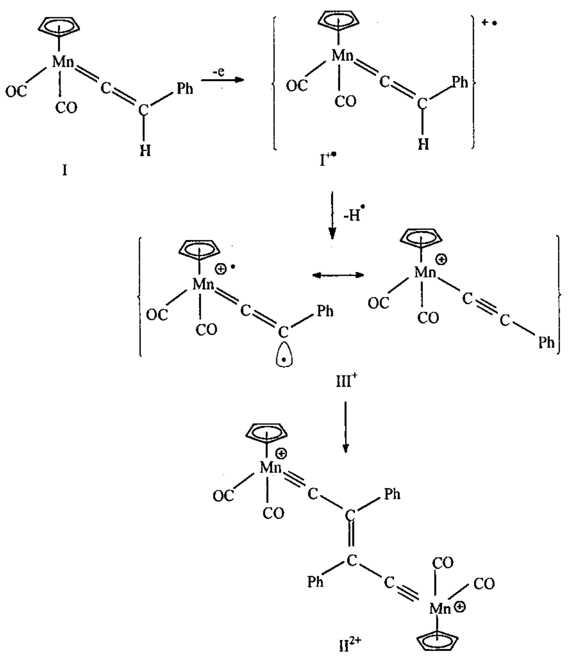


Fig. 1. Cyclic voltammograms of: (a) **II**; (b) **I**; and (c) **I** in the presence of Et_3N (CH_2Cl_2, working electrolyte, 0.1 M $^nBu_4NPF_6$; sweep rate, 200 mV s^{-1}; glassy-carbon electrode, potential vs. SCE).

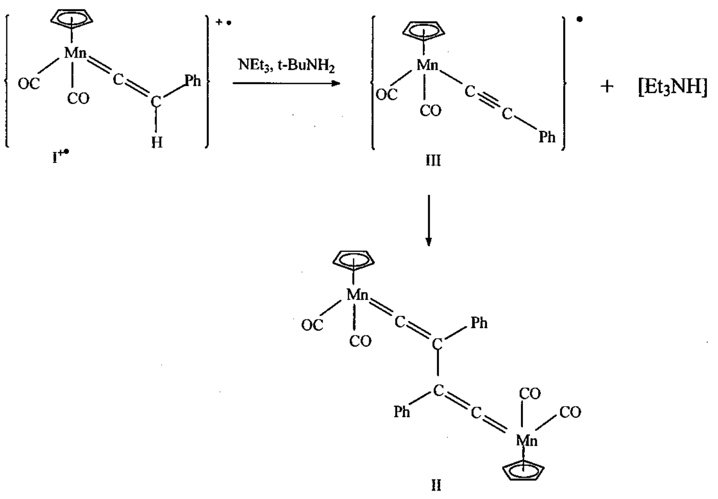


Scheme 1.

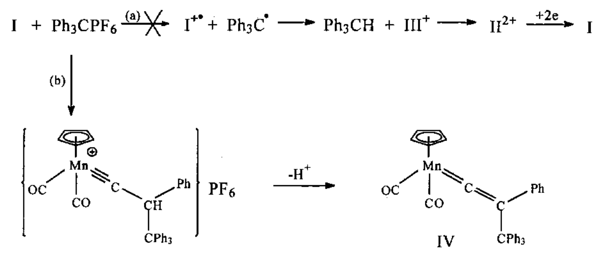
The oxidation of **I** to $\mathbf{I}^{+\bullet}$ by $AgBF_4$ starts at − 65 °C as evidenced by the silver mirror appearance. The optimal temperature for generating $\mathbf{I}^{+\bullet}$ is − 50 °C. At this temperature $\mathbf{I}^{+\bullet}$ is stable in solution for at least 10–15 min. The radical cation $\mathbf{I}^{+\bullet}$ transforms to the dication $\mathbf{II}^{2+}$ according to Scheme 1, the rate of this process becoming noticeable even at − 30 °C. The dimeric bis-carbyne complex $\mathbf{II}^{2+}$ is rather long-lived in solution below 0 °C but not readily isolable; therefore, it was characterized only in solution by IR (ν_{CO} 2000, 2052 cm^{-1}) and CV (vide supra). Reduction of $\mathbf{II}^{2+}$ is necessary to obtain **II** and can be accomplished by the use of $(C_6H_6)_2Cr$ ($E_{ox} = -0.7$ V vs. SCE) as the reductant to give **II** in a reasonable yield. We suggest that this reduction process should proceed with high rate as its key step, i.e. electron transfer from $(C_6H_6)_2Cr$ to $\mathbf{II}^{2+}$ is exergonic by ≈ 1.4 eV. This provides support for the mechanism in Scheme 1.

Treatment of $\mathbf{I}^{+\bullet}$ with a base (triethylamine or *tert*-butylamine) gives **II** in a 70% yield. However, it is more convenient to prepare **II** by addition of the oxidant $(C_5H_5)_2FeBF_4$ ($E_{red} = +0.45$ V vs. SCE) to a mixture of **I** and excess triethylamine in methylene chloride. The yield of **II** remains high (~ 70–80%). The results obtained enable us to propose that the dehydrodimerization process **I** → **II** can proceed via two competing paths differing in whether $\mathbf{I}^{+\bullet}$ undergoes initially the homolysis (Scheme 1) or the deprotonation of C_β–H bond (Scheme 2). In the former case triethylamine participates as the reductant of $\mathbf{II}^{2+}$ in the final step. A separate experiment demonstrated that this is plausible. The addition of triethylamine to the dicationic complex $\mathbf{II}^{2+}$ (the latter was generated by oxidation of **II** by $(C_5H_5)_2FeBF_4$ in CH_2Cl_2 at − 30 °C) did result in the regeneration of **II** in a nearly quantitative yield. This finding is rather unexpected as in this case the electron transfer from Et_3N to $\mathbf{II}^{2+}$ should be endergonic by ~ 0.3 eV. We argue that the first electron-transfer step is followed by other non-redox steps, so that the overall process becomes thermodynamically feasible. We will not comment further on the strict phenomenology of this work.

Thus, the oxidative dehydrodimerization of **I** to **II** can proceed via two different reaction paths implying either homolysis or deprotonation of C_β–H bond in $\mathbf{I}^{+\bullet}$ (Schemes 1 and 2, respectively). Treatment of the mixture of **I** and excess Et_3N with $(C_5H_5)_2FeBF_4$ as the oxidant is the procedure of choice from the preparative point of view.



Scheme 2.



Scheme 3.

The oxidatively induced conversion of **I** to the key intermediate (**III**) can be considered formally as a hydride abstraction process. The classic reagent for this process is the trityl cation. There is some evidence [10] that suggests that this process proceeds via initial one-electron oxidation followed by hydrogen atom loss. For this reason, we turned to the reaction of **I** with the trityl cation, expecting that this reaction to proceed according to Scheme 3a.

The reaction carried out in CH_2Cl_2 solution, however, gave the mononuclear vinylidene complex (η^5-C_5H_5)$(CO)_2Mn{=}C{=}C(CPh_3)Ph$ (**IV**) rather than the dimeric complex $\mathbf{II}^{2+}$. The formation of **IV** is believed to be the result of an electrophilic attack of the trityl cation on C_β of the vinylidene ligand in **I** followed by proton loss (Scheme 3, path b). The structure of **IV** was established by X-ray structural analysis.

The general view of the molecule **IV** is given in Fig. 2a. The main feature of the structure is the planar moiety involving Mn(1), C(8), C(9), C(10) and C(11) atoms: the mean square deviation from average plane is 0.009 Å. The projection of the molecule on this plane is presented in Fig. 2b. The sum of the valence angles at C(9) atom is 360.0°. The valence angle Mn(1)–C(8)–C(9) is practically linear — 176.3(2)°. The C(8)–C(9) bond distance 1.326(3) Å is close to the standard value for a carbon–carbon double bond [11]. The Mn(1)–C(8) bond length 1.778(2) Å falls in the typical range for Mn=C double bonds [11]. Unlike previously studied (η^5-C_5H_5)$(CO)_2Mn{=}C{=}C(H)Ph$, in which the phenyl group lies practically in the plane defined by central $Mn{=}C_\alpha{=}C_\beta(H)C_{ipso}$ moiety [12], the phenyl ring in **IV** is rotated from the corresponding plane by 32° at C(11). This is probably due to the presence of the highly sterically demanding trityl moiety in **IV**. At the same time, there are no shortened intramolecular contacts in **IV**. The phenyl rings of the trityl group also have typical C–C bond distances varying from 1.377(3) to 1.400(3) Å; the mean value is 1.388(3) Å.

The geometry around Mn(1) is pseudo octahedral (Fig. 2c). The angles between the linear central Mn(1)=C(8)=C(9) moiety and the carbonyl groups C(1)O(1) and C(2)O(2) are equal to 90.0(1) and 92.5(1)°, respectively. The Mn–C_5H_5 (centroid) distance is 1.788 Å. The distances from Mn(1) to individual carbon atoms of the cyclopentadienyl ring vary from 2.139(2) to 2.164(2) Å. The cyclopentadienyl ring plane (0.003 Å) forms an angle of 37.5° with the plane of the central Mn(1)–C(8)–C(9)–C(10) [C(11)] moiety.

The ^{1}H- and ^{13}C-NMR spectra reveal that **IV** exists in solution as a mixture of two isomers in the ratio of 5:1. The latter can be tentatively assigned to conformers that differ in the orientation of phenyl and trityl groups relative to the cyclopentadienyl ring plane. The structure of the isomers is under study.

The CV behavior of complex **IV** was also studied. It displayed a quasi reversible oxidation peak ($E_p^a = +1.02$ V, $i_p^c/i_p^a = 0.8$) and irreversible two-electron reduction peak $E_p^c = -1.93$ V.

2. Experimental

Reagent grade C_6H_6, heptane, and amines (Et_3N and *tert*-butylamine) were dried and distilled under a purified Ar atmosphere prior to use. Methylene chloride was distilled first from P_4O_{10} and, just prior to use, from $LiAlH_4$. Literature procedure was used to prepare (η^5-C_5H_5)$(CO)_2Mn{=}C{=}C(H)Ph$ [13]. All manipulations were carried out under a purified Ar atmosphere using Schlenk techniques.

Cyclic voltammogramms were registered on 'ПИ-50-1' (Gomel', Belarus) with glassy carbon ($S = 2$ mm^2) as a working electrode, platinum plate as an auxiliary electrode and SCE as a reference electrode. The number of elecrons consumed was estimated by comparison of the currents of the peaks observed with those of the one-electron decamethylferrocene/decamethylferrocenium couple at the same concentrations.

IR spectra were registered on Specord 75 IR instrument. NMR spectra were obtained on Bruker DPX-300 and Bruker AMX 400 specrometers.

2.1. *Oxidation of **I** in a preparative scale*

To the dark-red solution of 0.07 g (0.136 mmol) of **I** in 10 ml of CH_2Cl_2 cooled to −30 °C, 0.07 g (0.152 mmol) of $AgBF_4{\cdot}3$(dioxane) was added. After stirring at this temperature for 1 h 0.06 g (0.21 mmol) of solid $(C_6H_6)_2Cr$ was added in one portion and the mixture was allowed to reach room temperature (r.t.). The mixture was filtered, filtrate was concentrated in vacuum to one-fourth of the initial volume and rose crystals of complex **II** were precipitated by addition of heptane. After repetitive precipitations from C_6H_6–heptane, 0.055 g (80%) was obtained. IR (CH_2Cl_2, cm^{-1}): 2010, 1996, 1942 (ν_{CO}). M.p. (dec.): 210 °C. Anal. Found: C, 62.93; H, 3.62; Mn, 19.53. Calc. for $C_{30}H_{20}Mn_2O_4$: C, 64.98; H, 3.61; Mn, 19.86%. ^{1}H-NMR ($CDCl_3$): δ 5.0 (s, 10H, C_5H_5), 7.0, 7.25, 7.4 (m, 10H, Ph); (THF-d_8): δ 5.26 (s, 10H, C_5H_5), 7.08 (t, 2H, $J = 6.8$ Hz, Ph), 7.30 (t, 4H, $J = 6.8$ Hz, Ph), 7.47 (d, 4H, $J = 6.8$ Hz, Ph). ^{13}C-NMR ($CDCl_3$): δ 88.02 (C_5H_5), 124.52, 128.69 (Ph, *ortho*-, *meta*-), 125.32 (Ph, *para*-), 127.76 (Ph, *ipso*-), 135.13 (C_β), 226.62 (CO), 374.88 (C_α); (THF-d_8): 89.22 (C_5H_5), 125.74, 129.51 (Ph, *ortho*, *meta*), 126.13 (Ph, *para*), 136.48 (C_β), 227.96 (CO), 376.17 (C_α). MS; m/z: 554 [M^+], 498 [$M^+ - 2CO$].

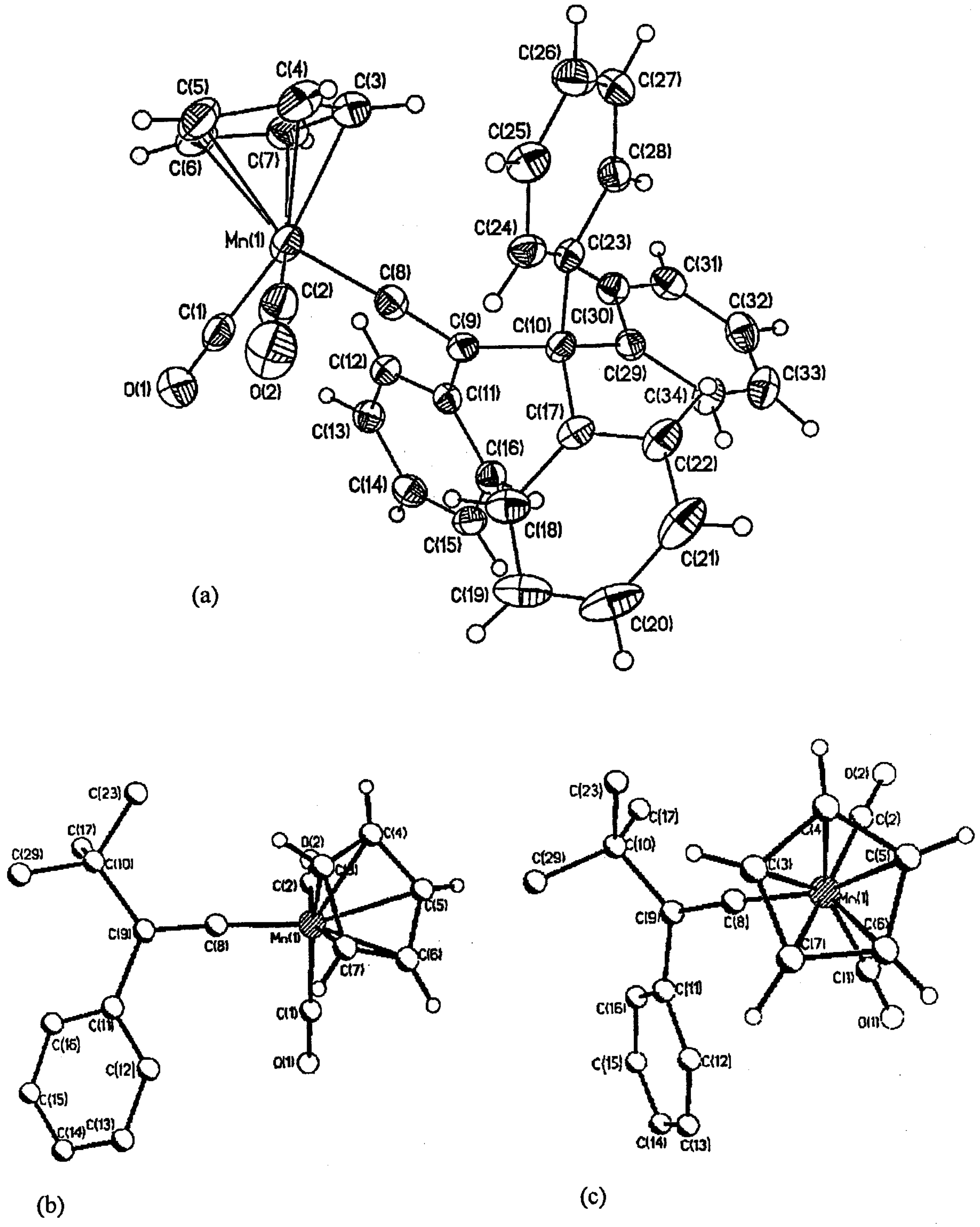


Fig. 2. (a) The general view of the molecule **IV**; (b) the projection of the molecule on the plane Mn(1), C(8), C(9), C(11), C(11); and (c) the geometry around Mn(1) atom.

*2.2. Preparation of **II***

To the solution of 0.20 g (0.72 mmol) of **I** and 0.227 g (0.3 ml, 2.25 mmol) of Et_3N in 25 ml of CH_2Cl_2 0.196 g (0.72 mmol) of ferricenium tetrafluoroborate was added at r.t. The mixture was magnetically stirred at this temperature for about 30 min. A rose-colored amorphous precipitate was filtered and washed with petroleum ether to remove residual ferrocene. The filtrate was further concentrated to yield the second crop of **II**. Crude **II** was reprecipitated from CH_2Cl_2 with heptane. The yield of complex **II** is 0.12 g (80%).

*2.3. Oxidation of dimer **II** to II^{2+} followed by reverse reduction of II^{2+} with Et_3N*

A solution of 0.05 g (0.090 mmol) of dimer **II** in 5 ml CH_2Cl_2 was cooled to −30 °C and 0.025 g (0.090 mmol) of ferricenium tetrafluoroborate was added. After 30 min the reaction mixture turned dark violet and bands of carbonyl absorption of dimer **II** in IR spectrum transformed into new bands 2000 and 2052 cm^{-1}. After additional 30 min of stirring at −30 °C an excess of Et_3N (0.2 ml) was added. The mixture was warmed slowly to r.t. The resulting solution according to IR data contained only dimer **II**. Compound **II** was reprecipitated twice from CH_2Cl_2 with heptane to give 0.04 g (80%).

*2.4. Reaction of (η^5-C_5H_5)$(CO)_2$Mn=C=CHPh (**I**) with Ph_3CPF_6*

To the solution of 0.1 g (0.36 mmol) of **I** in 30 ml of CH_2Cl_2, 0.14 g (0.37 mmol) of Ph_3CPF_6 was added at −5 °C. The mixture was magnetically stirred at −5 to 10 °C for 1.5 h, the solvent was evaporated in vacuum, and the residue was chromatographed on SiO_2 column (Aldrich, 70–230 mesh, 60 Å). The main crimson zone was eluted by petroleum ether. The eluate was evaporated and the residue was reprecipitated twice from C_6H_6–heptane. The yield of complex **IV** is 0.70 g (70%). M.p. 149–150 °C. IR (KBr pellet, cm^{-1}): 1930, 1987 (ν_{CO}); 1615, 1567 ($\nu_{C=C}$). Anal. Found: C, 77.91; H 4.99; Mn, 10.95. Calc. for $C_{34}H_{25}MnO_2$ (520): C, 78.46; H, 4.81; Mn, 10.58%. ^1H-NMR (C_6D_6): cyclopentadienyl protons: two singlets δ 4.32 (major), 4.37 (minor); phenyl protons 7.7 (d, $J = 6.6$ Hz), 7.47 (d, minor), 7.39 (d, minor, $J = 6.6$ Hz), 7.17 (d, 6.6 Hz), multiplet 7.132–7.117, 6.99 (t, minor). The ratio of the total integral intensity of the Cp protons to the total integral intensity of the Ph protons is 1:4. ^{13}C-NMR (C_6D_6): δ CO, 227.4; cyclopentadienyl C-atoms, 87.97; other C-atoms, 146.19 (int.); 129.76; 129.58; 128.49; 128.19 (int.), 128.07; 127.71 (int.); 128.48; 126.44; 125.97. According to ^1H- and ^{13}C-NMR spectra in C_6D_6 solution complex **IV** exists as a mixture of two isomers.

2.5. X-ray crystallographic data

The crystal of **IV** ($C_{34}H_{25}MnO_2$, $M = 520.48$) is triclinic, space group $P\bar{1}$ at $T = 110$ K: $a = 8.6453(10)$, $b = 12.5716(14)$, $c = 12.7821(14)$ Å, $\alpha = 102.388(2)$, $\beta = 102.933(2)$, $\gamma = 104.261(2)°$, $V = 1257.6(2)$ Å^3, $Z = 2$, $D_{calc} = 1.374$ Mg m^{-3}, $F(000) = 540$, $\mu = 0.555$ mm^{-1}.

Total of 15 060 reflections were measured on a SMART CCD 1000 diffractometer (λ (Mo–K_α), ω-scans with 0.3° step and 30 s per frame exposure, $\theta_{max} = 30°$) and correction for Lorentz and polarization effects and for absorption [14]. The structure was determined by direct methods and refined by the full-matrix least-squares technique with anisotropic thermal parameters for non-hydrogen atoms. Hydrogen atoms were objectively located in the difference Fourier map and refined in isotropic approximation. The final divergence factors were $R_1 = 0.0583$ for 4886 independent reflections with $I > 2\sigma(I)$ and $wR_2 = 0.1514$ for all 7275 independent reflections. All calculations were carried out using the SHELXTL (PC Version 5.10) programs [15].

3. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 161915 for compound **IV**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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