Note

Chemistry of vinylidene complexes. XXII. Synthesis and physicochemical properties of the binuclear vinylidene complex [Cp(CO)$_2$ReCu(μ-C≡CHPh)(μ-Cl)]$_2$. Molecular structure of the new rheniumcopper complex ☆

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This article is dedicated to the memory of Alla B. Antonova and William A. Sokolenko.

ABSTRACT

The interaction between Cp(CO)$_2$Re=C=CHPh (**1**) and CuCl results in the new heterometallic μ-vinylidene complex [Cp(CO)$_2$ReCu(μ$_2$-C=CHPh)(μ$_2$-Cl)]$_2$ (**2**) characterized earlier by IR and ^{1}H, ^{13}C NMR spectra. The molecular structure of compound **2** has been determined by X-ray diffraction analysis, revealing a dimer structure with two binuclear fragments linked to each other by asymmetric chloride bridges. The electrochemical behavior of **2** in acetonitrile was studied by cyclic voltammetry at a Pt-electrode.

1. Introduction

Binuclear heterometallic complexes with bridging vinylidene ligands μ-C=CRR′ (R and R′ = H, alkyl, aryl, etc.) are of interest due to their potential use in heteronuclear clusters synthesis and catalysis [2–4] and the knowledge of their formation and properties is important for understanding chemistry of known complexes and designing new ones.

In the course of our investigations on the formation of heteronuclear vinylidene complexes we were interested in the possible formation of the μ-vinylidene compound containing ReCu core. Copper containing μ-vinylidene complexes are rare. The CuOs [5] and the CuRh complexes [6] with the μ-C=CHR (R = H, Me, Ph)

ligands have been reported, but their structures has not reliably been determined. A CuTi μ-vinylidene complex without metal–metal bond is known [7]. Earlier, we reported on the synthesis and molecular structure of the μ-vinylidene complex [Cp(CO)$_2$MnCu(μ-C=CHPh)(μ-Cl)]$_2$ (**3**) [8].

Prior to this article, spectroscopic characteristics of discussed compound **2** were reported [9]. However, the spectroscopic study of **2** didn't unambiguously define its molecular structure, and therefore a single-crystal X-ray diffraction study was carried out.

In this work we report the structure and electrochemical study of the new complex **2**.

2. Results and discussion

2.1. The synthesis, spectroscopic properties and reactivity

The reaction of Cp(CO)$_2$Re=C=CHPh (**1**) with CuCl in ether at RT for 4 h gave the complex [Cp(CO)$_2$ReCu(μ-C=CHPh)(μ-Cl)]$_2$ (**2**)

with nearly quantitative yield (Scheme 1). Compound **2** was isolated as a red powder, stable in the solid state.

The IR and NMR study of **2** [8] revealed the difference between the spectroscopic data of this complex and those of previously synthesized μ-vinylidene complexes of rhenium [1,10–12].

The presence of two high-frequency bands in the IR spectrum of **2** at 2004, 1937 cm^{-1} (Table 1), with their position being almost the same as that of the bands of the precursor **1** (1996, 1923 cm^{-1}), have indicated that both carbonyl groups at a rhenium atom are terminal. While, in the known binuclear complexes of the type $Cp(CO)_2ReM(\mu\text{-C}{=}CHPh)L$ (M = Pt, Pd; L = $(PPh_3)_2$, dppm, dppe, dppp), one of the carbonyl group is semibridging due to influence of the adjacent metal atom M [1,12].

In the IR spectrum of earlier investigated manganese analog $[Cp(CO)_2MnCu(\mu\text{-C}{=}CHPh)(\mu\text{-Cl})]_2$ (**3**) (Table 1), the $\nu(CO)$ stretching bands are also shifted to higher frequency as compared to the compound $Cp(CO)_2Mn{=}C{=}CHPh$ [8].

In the ^1H NMR spectrum the signal of the vinylidene proton = C^2H is observed at 6.36 ppm (Table 1). The ^{13}C NMR spectrum of complex **2** shows the singlet signals of vinylidene μ-C^1 and C^2 atoms at 271.8 and 133.1 ppm, respectively. The low field position of the μ-C^1 signal at 271.8 ppm is unusual for the μ-vinylidene rhenium complexes of type $Cp(CO)_2ReM(\mu\text{-C}{=}CHPh)L$ (M = Pt, Pd), which ^{13}C NMR spectra show signals of bridging carbon atom in the range of 232–248 ppm [1,12]. Such position of the μ-C^1 atom signal of complex **2** in the ^{13}C NMR spectra indicates a weak interaction between copper and rhenium containing fragments as compared to μ-vinylidene rhenium complexes of type $Cp(CO)_2ReM(\mu\text{-C}{=}CHPh)L$ (M = Pt, Pd).

Due to the electron donor effect of rhenium the signal of the μ-C^1 atom of **2** is upfield shifted by 45 ppm as compared to corresponding complex **3**. For the same reason the $\nu(CO)$ frequencies of rhenium complexes **2** are lower than those of manganese analog **3**.

The chemical behavior of $[Cp(CO)_2ReCu(\mu\text{-C}{=}CHPh)(\mu\text{-Cl})]_2$ (**2**) has also shown a weak interaction between metal-containing fragments. The treatment of complex **2** with PPh_3 or $Fe_2(CO)_9$ resulted in cleavage of Re–Cu and Cu–C^1 bonds and formation of the precursor **1** or a transmetallation product *viz.* binuclear complex $Cp(CO)_2ReFe(\mu\text{-C}{=}CHPh)(CO)_4$ [9], respectively (Scheme 2). Similar results were previously obtained for the manganese complex $[Cp(CO)_2MnCu(\mu\text{-C}{=}CHPh)(\mu\text{-Cl})]_2$ (**3**) [8].

2.2. Molecular structure of $[Cp(CO)_2ReCu(\mu\text{-C}{=}CHPh)(\mu\text{-Cl})]_2$ (**2**)

Suitable (dark red) crystals of **2** for a X-ray diffraction analysis were obtained from the CH_2Cl_2–Et_2O (1:1) mixture. The molecular structure of complex **2** is shown in Fig. 1. The X-ray diffraction study established that compound **2** is a dimer, whose two binuclear fragments are linked to each other by asymmetrical chloride bridges (Cu(1)–Cl(1) 2.258(2), Cu(1)–Cl(1A) 2.284(2) Å). As is shown in Fig. 1, the dimer complex **2** contains a center of inversion,

which is the midpoint of the Cl(1)–Cl(1A) and Cu(1)–Cu(1A) vectors. In both binuclear fragments the copper, the rhenium and bridging carbon atoms form a triangle. The Re–Cu, Cu–C(1) and Re–C(1) bond lengths are 2.5442(6), 1.939(5) and 1.975(5) Å, respectively. The Re–C(1)–Cu, Cu–Re–C(1) and Re–Cu–C(1) bond angles of this triangle are 81.04(19)°, 48.85(13)°, 50.11(15)°, respectively.

The ^{13}C NMR measurements suggested the weak interaction between the copper [CuCl] and the rhenium [Re=C=C] fragments. The short Re–C(1) and C(1)=C(2) distances and the large Re–C(1)–C(2) angle confirm this assumption. The Re–C(1) distance of 1.976(5) Å is considerably shorter than that in μ-vinylidene compounds with the Re–Pt bonds (2.083–2.10 Å) [1,10,12]. The C(1)=C(2) bond length of 1.317(7) Å is also slightly shorter than that of the known μ-vinylidene complexes (its average value being 1.35 Å) [2]. The vector of the C(1)=C(2) bond is noticeably inclined toward the Cu atom, and the Re–C(1)–C(2) and Cu–C(1)–C(2) bond angles are 157.1(4)°, 121.7(4)°, respectively.

Structural properties of such type, namely the asymmetrical chloride bridges, the short Mn–C(1) and C(1)=C(2) distances and inclination of the C(1)=C(2) bond toward the Cu atom, were previously observed for the corresponding manganese complex **3** [8].

The presence of terminal carbonyl groups in molecule of complex **2** was suggested by its IR spectrum. The X-ray diffraction study showed that the Re–C(3)–O(3) and Re–C(4)–O(4) bond angles were 179.1(6)° and 177.0(6)°, respectively and there was no interaction between the copper atom and the carbonyl groups in the molecule of **2**.

It should be noted that the Re–Cu distance of 2.544 Å found in complex **2** is significantly shorter than a sum (2.83 Å) of covalent radii for the Re (1.51 Å) and Cu (1.32 Å) atoms [13]. Moreover, this bond length is far shorter as compared to the Re–Cu bond lengths in other complexes containing the Re–Cu bond in which these bonds fall in the range of 2.59–2.93 Å [14]. However, in spite of this short Re–Cu distance, complex **2** shows the weak interaction between the copper and the rhenium fragments due to the reasons described above. So, the strength of the interaction between the copper [CuCl] and the rhenium [Re=C=C] fragments cannot be determined from the short Re⋯Cu distance in this complex.

Table 1
IR data, electrochemical properties and NMR data of complexes **2** and **3**.

Complex	IR spectra, $\nu(CO)$, cm^{-1} (CH_2Cl_2)	$E_{1/2}$, B (n)[a]	NMR, δ, ppm (CD_2Cl_2)		Ref.
			^1H	^{13}C	
[ReCu] (**2**)	2004, 1937	0.22 (2)	6.36c (=$C^2\underline{H}$)	271.8c (μ-C^1)	[9]
		0.85 (2)	5.85c ($C_5\underline{H}_5$)	133.1 (=C^2)	
[MnCu] (**3**)	2005, 1953	0.30 (2)	7.69c (=$C^2\underline{H}$)	317.1c (μ-C^1)	[8,16]
		0.90 (2)	5.27c ($C_5\underline{H}_5$)	136.3c (=C^2)	

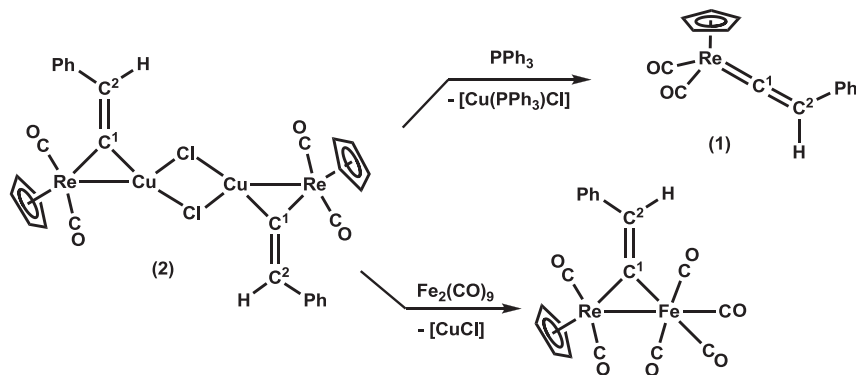[a] n – number of electrons; MeCN, 01 M Et_4NBF_4, $1 \cdot 10^{-3}$ M, Ag/0.1 M $AgNO_3$ in MeCN.
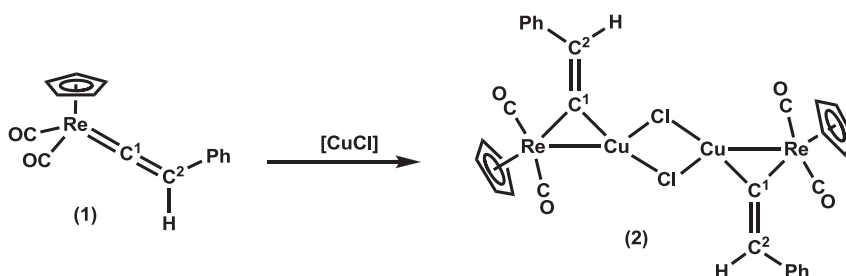
Scheme 1.

Scheme 2.

2.3. The electrochemical study

The redox properties of complex $[Cp(CO)_2ReCu(\mu$-C=$CHPh)(\mu$-$Cl)]_2$ (**2**) have been studied by cyclic voltammetry at a platinum stationary electrode and controlled-potential electrolyses in the acetonitrile solution with $[Et_4N][BF_4]$ as the supporting electrolyte. Potentials are given versus Ag/0.1 M $AgNO_3$ in MeCN (they can be converted to V versus SCE by adding 0.337 V).

The cyclic voltammograms of **2** show three two-electron oxidation at $E_{1/2}$ = 0.22, 0.85, 1.32 V (Fig. 2(a), (b)) and three reduction stages: two one-electron at $E_{1/2}$ = −0.83, −1.35 V and one at $E_{1/2}$ = −2.15 V (Fig. 2(b)).

A small oxidation wave at $E_{1/2}$ = 0.35 V is observed after a two-electron oxidation wave at $E_{1/2}$ = 0.22 V (Fig. 2(a), (b), peak A_1). On the reverse scanning of potential the height of reduction wave at $E_{1/2}$ = 0.35 V increases (Fig. 2(a), (b), peak A_1'). The half wave oxidation potential value (0.35 V) and the half wave reduction potential value (−2.15 V) of complex **2** (Fig. 2(b), peak A_2) coincide with the redox potential values of $Cp(CO)_2Re$=C=$CHPh$ (**1**). The redox properties of complex **1** in CH_2Cl_2 and MeCN were previously reported in Refs. [15,16], respectively.

The formation of **1** after the two-electron oxidation or reduction of the dimer complex **2** on the Pt-electrode was confirmed by controlled-potential electrolysis with the subsequent identification of electrolysis products by IR spectroscopy (ν(CO) 1996, 1923 cm^{-1}).

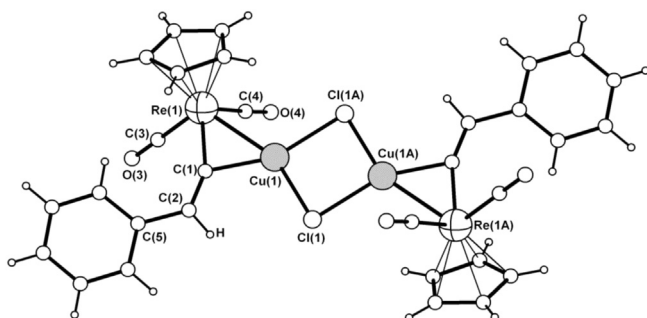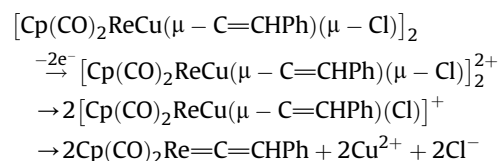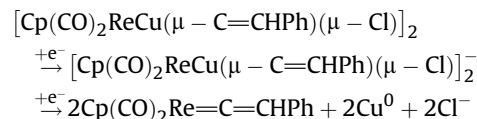On the reverse scanning of potential after a two-electron reduction of **2** (Fig. 2(b)) a large oxidation peak A_3 at $E_{1/2}$ = −0.63 V appears at the potential characteristic of the Cu^0 oxidation. After the reduction of compound **2** the Pt-electrode surface becomes coated with a film of metallic copper. Moreover, increasing the cathodic scan time leads to accumulation of copper at the electrode, and therefore the intensity of the peak A_3 increases as compared to other peaks. On the other hand, after the oxidation of complex **2** the peak A_3 is not observed in the anodic scan within the range from −0.70 to 2.00 V and in the reverse scanning of potential from 2.00 to −0.70 V (Fig. 2(a)). Thus, the addition of two electrons to the complex **2** results in the cleavage of the Cu–Cl, Re–Cu, Cu–C^1 bonds and formation of complex **1** and metallic copper.

Based on the data obtained the following schemes of the redox reactions of complex **2** at the platinum electrode are proposed:

Oxidation

$$[Cp(CO)_2ReCu(\mu - C{=}CHPh)(\mu - Cl)]_2$$
$$\xrightarrow{-2e^-} [Cp(CO)_2ReCu(\mu - C{=}CHPh)(\mu - Cl)]_2^{2+}$$
$$\rightarrow 2[Cp(CO)_2ReCu(\mu - C{=}CHPh)(Cl)]^+$$
$$\rightarrow 2Cp(CO)_2Re{=}C{=}CHPh + 2Cu^{2+} + 2Cl^-$$

Reduction

$$[Cp(CO)_2ReCu(\mu - C{=}CHPh)(\mu - Cl)]_2$$
$$\xrightarrow{+e^-} [Cp(CO)_2ReCu(\mu - C{=}CHPh)(\mu - Cl)]_2^-$$
$$\xrightarrow{+e^-} 2Cp(CO)_2Re{=}C{=}CHPh + 2Cu^0 + 2Cl^-$$

The comparison of the electrochemical behavior of complex $[Cp(CO)_2ReCu(\mu$-C=$CHPh)(\mu$-$Cl)]_2$ (**2**) with **3** shows that the scheme of the redox reactions of complex **2** and its Mn-containing analog **3** at a Pt-electrode in acetonitrile are quite similar [17].

3. Conclusions

The present study of $[Cp(CO)_2ReCu(\mu$-C=$CHPh)(\mu$-$Cl)]_2$ (**2**) allows one to interpret the previously obtained chemical and spectroscopic data. Crystallographically determined molecular structure and electrochemical studies of **2** confirm the hypothesis on the weak interaction between the copper and rhenium fragments, which was suggested based on the reaction and spectroscopic data.

Fig. 1. Molecular structure of complex $[Cp(CO)_2ReCu(\mu$-C=$CHPh)(\mu$-$Cl)]_2$ (**2**). The selected interatomic distances (Å) and angles (°): Re(1)–Cu(1) = 2.5442(6), Re(1)–C(1) = 1.975(5), Cu(1)–C(1) = 1.939(5), Cu(1)–Cl(1) = 2.258(2), Cu(1)–Cl(1A) = 2.284(2), C(1)–C(2) = 1.317(7), C(3)–O(3) = 1.136(6), C(4)–O(4) = 1.156(7), Re(1)–C(3) = 1.913(6), Re(1)–C(4) = 1.907(6); Re(1)–C(1)–C(2) = 157.1(4), Cu(1)–C(1)–C(2) = 121.7(4), Re(1)–C(3)–O(3) = 179.1(6), Re(1)–C(4)–O(4) = 177.0(6), C(1)–Cu(1)–Cl(1) = 105.53(16), Cl(1)–Cu(1)–Cl(1A) = 93.68(5), Re(1)–C(1)–Cu(1) = 81.04(19), C(1)–Re(1)–Cu(1) = 48.85(13), C(1)–Cu(1)–Re(1) = 50.11(15), C(1)–C(3)–(5) = 128.6(5).

Fig. 2. The cyclic voltammogram of oxidation (a), reduction and oxidation (b) of [Cp(CO)$_2$ReCu(μ-C=CHPh)(μ-Cl)]$_2$ (**2**) at Pt-electrode in MeCN (0.1 M Et$_4$NBF$_4$, C = 2 mM, scan rate 25 V s^{-1}, potentials versus Ag/0.1 M AgNO$_3$ in MeCN).

The X-ray diffraction analysis of **2** revealed the shortest Re–C1 and C(1)=C(2) distances and the largest Re–C1–C2 angle among the known binuclear μ-vinylidene rhenium complexes. The electrochemical study showed that the two-electron reduction or two-electron oxidation of complex [Cp(CO)$_2$ReCu(μ-C=CHPh)(μ-Cl)]$_2$ (**2**) at the Pt-electrode resulted in the cleavage of the Cu–Cl, Re–Cu, Cu–C^1 bonds and formation of complex Cp(CO)$_2$Re=C^1=C^{2}HPh (**1**).

The comparison of XRD, IR and NMR data of complex **2** with ones of earlier synthesized binuclear μ-vinylidene complexes of rhenium revealed their structural and spectroscopical differences. On the other hand, there is similarity of chemical and electrochemical behavior, structural and spectroscopic properties between **2** and the manganese complex [Cp(CO)$_2$MnCu(μ-C=CHPh)(μ-Cl)]$_2$ (**3**).

4. Experimental

All operations and manipulations were carried out under an argon atmosphere. Solvents were purified by distillation from appropriate drying agents and stored under argon. The course of reaction was followed by means of TLC on Silufol plates and IR spectroscopy. Neutral alumina was used for the chromatography. Physico-chemical characteristics were obtained in the Krasnoyarsk Regional Centre of Research Equipment, Siberian Branch of the Russian Academy of Sciences. The IR spectra were recorded on the Vector 22 Infrared Fourier spectrometer (Bruker, Germany). The ^{1}H, ^{13}C{^{1}H} NMR spectra were obtained using NMR spectrometer AVANCE III 600 DPX (Bruker, Germany). The X-ray data were obtained with the SMART APEX II diffractometer (Bruker AXS, Germany). Complex Cp(CO)$_2$Re=C=CHPh (**1**) was prepared as described in Ref. [15].

4.1. Synthesis of [Cp(CO)$_2$ReCu(μ-C=CHPh)(μ-Cl)]$_2$ (**2**)

A mixture of 100 mg (0.244 mmol) of **1** and 30 mg (0.3 mmol) of CuCl in 20 mL of diethyl ether was stirred for 4 h. The solvent was

evaporated *in vacuo* and the obtained residue was dissolved in 10 mL of dichloromethane. After filtration, the solution was reduced to 3 mL, and then 3 mL of diethyl ether was added. After 2 h at room temperature and 18 h at 5°C the dark-red crystals formed were collected by removing the mother liquor, washing with diethyl ether and drying under argon atmosphere at 20 °C. Yield [Cp(CO)$_2$ReCu(μ-C=CHPh)(μ-Cl)]$_2$ (**2**): 110 mg (89%).

Anal. Calc. for C$_{30}$H$_{22}$Cl$_2$Cu$_2$O$_4$Re$_2$ (1016.91): C, 35.43; H, 2.18%. Found: C, 35.47; H, 2.22%.

IR (CH$_2$Cl$_2$, cm^{-1}): 2004s, 1937m, br (ν_{CO}); (KBr, cm^{-1}): 1993s, 1933m, br (ν_{CO}).

^{1}H NMR (CD$_2$Cl$_2$) δ: 5.85 (s, 5H, C$_5$H$_5$); 6.36 (s, 1H, =C^{2}HPh); 7.17 (t, 1H, H$_{para}$ of =C^{2}HC$_6$H$_5$); 7.25 (d, 2H, H$_{ortho}$ of =C^{2}HC$_6$H$_5$); 7.34 (t, 2H, H$_{meta}$ of =C^{2}HC$_6$H$_5$).

^{13}C{^{1}H} NMR (CD$_2$Cl$_2$) δ: 89.8 (s, C$_5$H$_5$); 125.9 (s, C$_{meta}$ of =C^{2}HC$_6$H$_5$); 126.5 (s, C$_{para}$ of =C^{2}HC$_6$H$_5$); 128.5 (s, C$_{ortho}$ of =C^{2}HC$_6$H$_5$); 131.7 (s, C$_{ipso}$ of =C^{2}HC$_6$H$_5$); 133.1 (s, =C^{2}HPh); 195.6 (s, br, 2Re–CO); 271.8 (s, br, μ-C^1).

4.2. Reaction of **2** with triphenylphosphine

To 5 mg (0.012 mmol) of **2** dissolved in 3 mL of CH$_2$Cl$_2$ was added 10 mg (0.038 mmol) of PPh$_3$ at RT. The formation of Cp(CO)$_2$Re=C=CHPh (**1**) in reaction mixture was detected by TLC. After stirring this reaction solution for 40 min the solvent was evaporated *in vacuo* and residue was extracted by hexane and filtered through 0.2 cm of SiO$_2$. The filtrate was concentrate to 2 mL under reduced atmosphere and after crystallization for 16 h at −18°C complex **1** was obtained (3 mg, 75%), identified by IR spectroscopy.

IR (C$_6$H$_{14}$, cm^{-1}): 2000, 1932 (ν_{CO}).

4.3. Reaction of **2** with Fe$_2$(CO)$_9$

A mixture of 32 mg (0.031 mmol) of complex **2** and 23 mg (0.063 mmol) of Fe$_2$(CO)$_9$ in 5 mL of CH$_2$Cl$_2$ was stirred at RT for 3 h.

Table 2
Crystallographic data and refinement details for complex **2**.

Empirical formula	$C_{15}H_{11}ClCuO_2Re$
Formula weight	952
T (K)	296
Space group	$\boldsymbol{P2_1/n}$
a (Å)	11.618(1)
b (Å)	8.1025(9)
c (Å)	17.017(2)
β (°)	105.042(1)
V (Å^3)/Z	1547/4
d_c (g/cm^3)	2.183
μ (mm^{-1})	9.36
N_{tot}/N_{uniq} (R_{int})	13,667/3729 (0.0434)
$2\theta_{max}$ (°)	56
Refined parameters	173
R1	0.030
wR2	0.064
Extinction parameter	0.0014(1)
GooF	1.049
$\Delta\rho_{min}/\Delta\rho_{max}$ (e/Å^3)	−0.99/1.08

The solvent was evaporated *in vacuo* and the obtained residue was extracted with hexane. The extract was chromatographed on a SiO$_2$ column, and 30 mg (85%) of amorphous orange complex Cp(CO)$_2$ReFe(μ-C=CHPh)(CO)$_4$ was obtained, identified by IR spectroscopy.

IR (C$_6$H$_{14}$, cm^{-1}): 2087, 2034, 2007, 2001, 1967, 1921 (ν_{CO}).

4.4. X-ray diffraction studies

A crystal of 0.12 × 0.18 × 0.38 mm dimensions has been chosen for the structure **2** search. The intensities of reflections were collected using graphite monochromated Mo K$_\alpha$-radiation. Absorption corrections were applied using the SADABS program [18] via multi-scan method. The structure was solved by direct methods and refined in anisotropic approach for non-hydrogen atoms using the SHELXTL program [19]. The hydrogen atoms were located by difference electron density maps. The phenyl cycle and hydrogen atoms, besides H2, were refined in a constrained mode. The experimental data and the refinement parameters of **2** structure are listed in Table 2.

4.5. Electrochemical studies

Electrochemical measurements were carried out in acetonitrile solutions with 0.1 M [Et$_4$N][BF$_4$] as supporting electrolyte under an argon atmosphere at room temperature. Cyclic voltammograms were recorded on an IPC-Pro M potentiostat with computer software using a three-electrode system. The working electrode was a platinum stationary electrode (1 mm diameter). The reference electrode was Ag/0.1 M AgNO$_3$ in MeCN, and the auxiliary electrode was platinum wire. Controlled-potential electrolyses were carried out with the IPC-Pro potentiostat using three-electrode system with a working platinum electrode (4 mm diameter).

Acknowledgments

Appendix A. Supplementary material

CCDC 962721 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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