



Note

Chemistry of vinylidene complexes. XVIII. Synthesis and molecular structure of the novel trinuclear μ_3 -vinylidene complex $\text{CpReFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6(\text{PPh}_3)^{\star, \star\star}$

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ABSTRACT

The interaction between $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C=CHPh})(\text{PPh}_3)_2$ (**1**) and $\text{Fe}_2(\text{CO})_9$ afforded the new heterometallic μ_3 -vinylidene cluster $\text{CpReFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6(\text{PPh}_3)$ (**2**). An X-ray diffraction study shows the complex **2** possesses a trimetallic Re–Fe–Pt chain core. The bond lengths are Re–Fe 2.8221(8), Fe–Pt 2.5813(8) Å; the Re...Pt distance is 3.3523(7) Å; the bond angle Re–Fe–Pt is 76.55(3)°. The $\mu_3\text{-C=CHPh}$ ligand is η^1 -bound to the Re and Pt atoms and η^2 -coordinated to the Fe atom. The C=C bond length is 1.412(4) Å. The Pt atom is coordinated by the PPh_3 and CO groups. Complex **2** is characterized by the IR and ^1H , ^{13}C and ^{31}P NMR spectra.

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1. Introduction

Heterometallic μ_3 -vinylidene clusters with the $\text{MM}'\text{M}'$ cores belong to the rarest type of vinylidene complexes [2–4]. μ_3 -Vinylidene clusters with the RuCo_2 and MCoM' cores ($\text{M} = \text{Fe}, \text{Ru}$; $\text{M}' = \text{Mo}, \text{W}, \text{Fe}, \text{Ni}$) were prepared earlier by rearrangement of μ_3 -alkyne isomers or by an exchange of the Co atom in the μ_3 -vinylidene FeCo_2 or RuCo_2 clusters for the $\text{M}' = \text{Mo}, \text{W}, \text{Fe}, \text{Ni}$ atom [5]. The majority of these reactions occurred at 60–110 °C.

We have described an original approach to the synthesis of the trimetallic $\text{MM}'\text{M}'$ μ_3 -vinylidene clusters by consecutive assembly of a metal core from mononuclear (M) and dinuclear (MM') vinylidene building blocks in mild conditions [2,4,6]. The first μ_3 -vinylidene MnFePt clusters were prepared by consecutive transformations of $\text{Cp}(\text{CO})_2\text{Mn=C=CHPh}$ into $\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C=CHPh})(\text{L})_2$ ($\text{L} = \text{PPh}_3, \text{P}(\text{OR})_3$; $(\text{L})_2 = \text{dppm}$) and the latter into $\text{CpMnFePt}(\mu_3\text{-C=CHPh})(\text{L})_n(\text{CO})_m$ ($n = 1$ and 2 ; $m = 5$ and 6) at 20 °C [6,7].

We continue to research the purposeful synthesis of the trimetallic $\text{MM}'\text{M}'$ μ_3 -vinylidene clusters using rhenium complexes, viz.

mononuclear $\text{Cp}(\text{CO})_2\text{Re=C=CHPh}$ and dinuclear $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C=CHPh})(\text{PPh}_3)_2$ (**1**) as building blocks. In this paper we describe the structure of the μ_3 -vinylidene cluster $\text{CpReFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6(\text{PPh}_3)$ (**2**) resolved by X-ray single crystal analysis. Synthesis, molecular structure and the IR and ^1H , ^{13}C and ^{31}P NMR data for complex **1** have been already published [1,8].

2. Results and discussion

The addition of the $[\text{Pt}(\text{PPh}_3)_2]$ group to $\text{Cp}(\text{CO})_2\text{Re=C=CHPh}$ resulted in the formation of bimetallic complex $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C=CHPh})(\text{PPh}_3)_2$ (**1**) [1]. The subsequent reaction between **1** and $\text{Fe}_2(\text{CO})_9$ (benzene, 20 °C, 6 h) has produced a new trimetallic cluster $\text{CpReFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6(\text{PPh}_3)$ (**2**) in 70% yield (Scheme 1). Red crystals **2** stable under usual conditions were obtained by crystallization from ether–hexane (1:4) mixture.

The crystal structure of complex **2** is shown in Fig. 1. The Re...Pt distance (3.3523(7) Å) in complex **2** is longer than all known distances 2.65–3.13 Å [9] in complexes with the Re–Pt bonds. The Re–Pt bond length in complex **1** is 2.7360(3) Å [8]. The Re...Pt distances such as 3.228 Å in $[\text{Pt}_3\text{Re}(\text{CO})_3(\mu_3\text{-O})_2(\mu\text{-dppm})_3]^+$ [10a] and longer were not considered as bonded [10b]. Consequently, the metal core of complex **2** can be regarded as the Re–Fe–Pt chain. One PPh_3 ligand and the C(8)O(8) group are linked to the Pt atom.

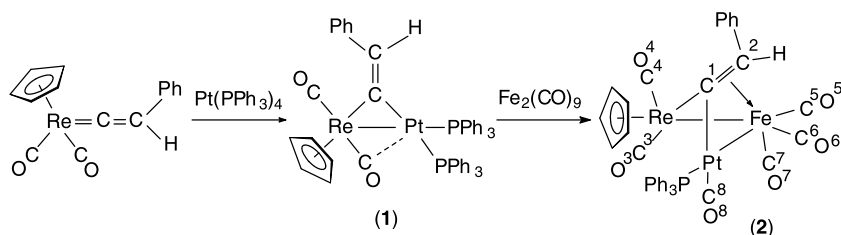
Three μ_3 -vinylidene complexes with heterometallic chain cores, viz. $(\eta^2\text{-dippe})\text{PdMn}(\mu_3\text{-C=CHPh})\text{PdMn}(\mu\text{-C=CHPh})(\text{CO})_4\text{Cp}_2$ (**3**)

* For Part XVII, see Ref. [1].

$\star\star$ $\text{Cp} = \eta^5\text{-C}_5\text{H}_5$; $\text{dppm} = \text{Ph}_2\text{P}(\text{CH}_2)\text{PPh}_2$; $\text{dppe} = \text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2$; d^+ , s^+ = doublet and singlet, respectively, with ^{195}Pt satellites.

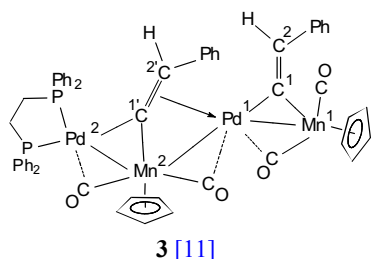
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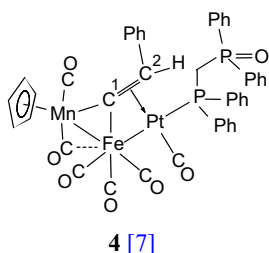


Scheme 1.

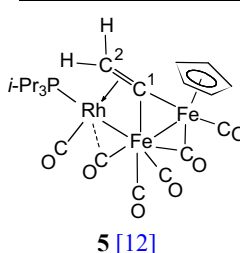
[11], $\text{CpMnFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6[\eta^1\text{-Ph}_2\text{PCH}_2\text{P(=O)Ph}_2]$ (**4**) [7] and $(i\text{-Pr}_3\text{P})\text{RhFe}_2(\mu_3\text{-C=CH}_2)(\mu\text{-CO})_2(\text{CO})_4\text{Cp}$ (**5**) [12] have been reported earlier.



3 [11]



4 [7]



5 [12]

Unlike complexes **3–5**, where vinylidene is η^1 -bound to two adjacent metal atoms and η^2 -coordinated to the terminal metal atom, complex **2** contains the $\mu_3\text{-C=CHPh}$ ligand η^1 -bound to the terminal Re and Pt atoms and η^2 -coordinated to the central Fe atom. The $\text{C}(1)=\text{C}(2)$ bond length of 1.412(4) Å in **2** is comparable with that in the known μ_3 -vinylidene complexes (1.25–1.43 Å) [4]. The $\mu_3\text{-C}^1=\text{C}^2\text{HPh}$ ligand in complex **2** is bound to the Fe atom

asymmetrically, since the Δd difference between the $\text{Fe}-\text{C}(2)$ and $\text{Fe}-\text{C}(1)$ distances is 0.231 Å and the $\text{Fe}-\text{C}(1)-\text{C}(2)$ and $\text{Fe}-\text{C}(2)-\text{C}(1)$ angles are 79.3(2)° and 61.6(2)°, respectively.

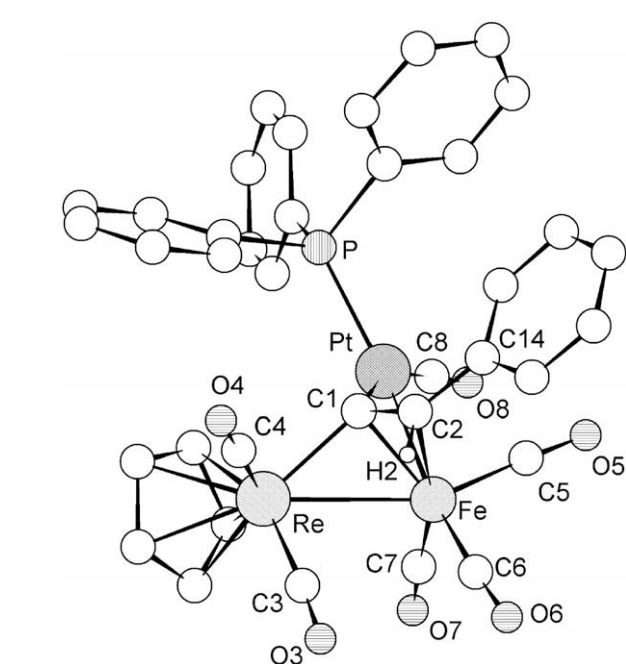


Fig. 1. Molecular structure of $\text{CpReFePt}(\mu_3\text{-C=CHPh})(\text{CO})_6(\text{PPh}_3)_2$ (**2**). The H atoms in Cp and Ph groups are omitted for clarity. Selected interatomic distances (Å) and bond angles (°): Re–Fe 2.8221(8); Fe–Pt 2.5813(8); Re–C(1) 2.122(3); Pt–C(1) 2.006(3); Fe–C(1) 1.967(3); Fe–C(2) 2.198(3); C(1)=C(2) 1.412(4); Pt–P 2.295(1); Pt–C(8) 1.905(3); C(8)–O(8) 1.128(4); Re–Fe–Pt 76.55(3); Fe–C(1)–Re 87.2(1); Fe–C(1)–Pt 81.0(1); Re–C(1)–Pt 108.6(1); Re–C(1)–C(2) 120.9(2); Pt–C(1)–C(2) 125.1(2); P–Pt–Fe 153.67(2).

Significant asymmetry of π -bonding of the M atom with the $\mu_3\text{-C}^1=\text{C}^2\text{HR}$ ligand is a characteristic feature of the well known μ_3 -vinylidene clusters with triangular metal cores, where the Δd difference between the M–C² and M–C¹ bond lengths averages 0.25 Å [4]. Similarly, the $\mu_3\text{-C}(1')=\text{C}(2')\text{HPh}$ ligand in **3** is η^2 -coordinated to the Pd(1) atom asymmetrically, since the Δd difference between the Pd(1)–C(2') and Pd(1)–C(1') distances is equal to 0.227 Å; the angles are Pd(1)–C(1')–C(2') 81.4(2)° and Pd(1)–C(2')–C(1') 63.2(2)° [11].

On the contrary, an almost symmetrical η^2 -bonding of vinylidene with the Pt or Rh atom is observed in complexes **4** and **5**. The Δd values are 0.00 and 0.05 Å for two independent molecules of complex **4** [7]. For complex **5** containing the Fe–Fe–Rh chain, the Δd difference between the Rh–C² and Rh–C¹ bond lengths is 0.04 Å [12].

Thus, the study of the complex **2** structure indicates that an iron-carbonyl fragment addition to $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C=CHPh})(\text{PPh}_3)_2$ (**1**) is accompanied by the replacement of one of PPh_3 ligands by CO group, a cleavage of the Re–Pt bond and formation of the $[\text{Re}-\text{Fe}-\text{Pt}](\mu_3\text{-C=CHPh})$ system with an unusual geometry.

The IR and NMR spectra of complex **2** indicate the presence of a mixture of isomers **2a** and **2b** in solutions. The IR spectrum of a cyclohexane solution of complex **2** exhibits 10 $\nu(\text{CO})$ bands. The band at 2054–2039 cm^{-1} belongs to the C(8)O(8) group at the Pt atom and the bands in an interval of 2000–1870 cm^{-1} belong to the CO groups at the Fe and Re atoms.

The ^1H NMR spectrum of **2** in CD_2Cl_2 measured at -40°C contains the Cp singlet at δ 5.39 and the signal at δ 6.35 [J(PtH) 66.4 Hz] corresponding to the proton of the $\mu_3\text{-C=CHPh}$ ligand. Two singlets of the Cp protons at δ 5.44 and 5.07 with intensity ratio of 3.7:1 and two signals of vinylidene protons at δ 6.42 [J(PtH) 66.4 Hz] and 5.95 [J(PtH) 54.4 Hz] appeared on warming up to $+20^\circ\text{C}$.

A sole signal at δ 24.90 [J(PtP) 3409 Hz] was found in the ^{31}P NMR spectrum of **2** measured at -40°C , and two signals at δ 24.64 [J(PtP) 3424 Hz] and 22.33 [J(PtP) 3490 Hz] in the ratio of 3.7:1 were observed at $+20^\circ\text{C}$.

A mixture of **2a** and **2b** (3.7:1) is formed and the equilibrium settled immediately after solution of crystals **2** in CD_2Cl_2 at $+20^\circ\text{C}$. This ratio remains constant during 50 h.

The ^{13}C NMR spectrum at +20 °C contains two sets of signals for **2a** and **2b** isomers. The C^1 and C^2 resonances (δ 270.94 and 98.79 for **2a** and δ 267.32 and 93.57 for **2b**, respectively) are located in the δ regions typical for the known clusters containing the $\mu_3\text{-C}^1=\text{C}^2\text{HR}$ (R = H, Ph) ligands [4].

No additional signals in the ^1H , ^{31}P and ^{13}C spectra, besides those attributed to **2a** and **2b**, were observed over a period of more than 50 h. Attempts to isolate isomer **2b** were unsuccessful and crystallization afforded solely complex **2**. Therefore, the complex **2** in solution undergoes isomerization rather than decomposition. It is possible that isomer **2b** has a structure similar to that of the MnFePt complex **4** [7], and **2b** is formed perhaps by means of the change of the $\mu_3\text{-C}=\text{CHPh}$ ligand position about the ReFePt framework.

3. Conclusions

Complex $\text{CpReFePt}(\mu_3\text{-C}=\text{CHPh})(\text{CO})_6(\text{PPh}_3)$ (**2**) results from the consecutive metalation reactions, namely the addition of $[\text{Pt}(\text{PPh}_3)_2]$ to $\text{Cp}(\text{CO})_2\text{Re}=\text{C}=\text{CHPh}$ followed by addition of $[\text{Fe}(\text{CO})_4]$ to $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C}=\text{CHPh})(\text{PPh}_3)_2$ (**1**). Compound **2** is the first structurally characterized μ_3 -vinylidene cluster with the ReFePt core, and along with **3**, **4** and **5**, it belongs to the special type of vinylidene complexes with the central fragment incorporating a chain of several different metal atoms.

4. Experimental

All operations were carried out in an argon atmosphere. Absolute solvents saturated with argon were used. 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 FTIR spectrometer Tensor 27 (Bruker, Germany). The ^1H , $^{13}\text{C}\{^1\text{H}\}$ and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra (CD_2Cl_2 solutions) were obtained using NMR spectrometer AVANCE 200 (Bruker, Germany). The X-ray data were obtained with the SMART APEX II autodiffractometer (Bruker, Germany). Complex $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C}=\text{CHPh})(\text{PPh}_3)_2$ (**1**) was prepared as described in Ref. [1].

4.1. Reaction of $\text{Cp}(\text{CO})_2\text{RePt}(\mu\text{-C}=\text{CHPh})(\text{PPh}_3)_2$ (**1**) with $\text{Fe}_2(\text{CO})_9$

A mixture of **1** (100 mg, 0.089 mmol) and $\text{Fe}_2(\text{CO})_9$ (161 mg, 0.443 mmol) in benzene (20 ml) was stirred at 20 °C for 6 h. The reaction mixture was filtered through ~1 cm alumina pad and the solvent was evaporated in vacuum. Dark brown residue was dissolved in hexane–benzene (4:1) mixture and chromatographed on an alumina column. Two zones were eluted with hexane–benzene (1:4) and (1:3) mixtures sequentially. The first colorless zone gave 33 mg of $\text{Fe}(\text{CO})_4(\text{PPh}_3)$. The second light pink zone contained 2 mg of a mixture of $\text{Fe}(\text{CO})_3(\text{PPh}_3)_2$ and an unidentified compound. A third dark green–brown zone was eluted with hexane–benzene (1:2) mixture. After removal of solvent and crystallization of the residue from hexane–ether (4:1) mixture, 65 mg (70%) of dark red crystals $\text{CpReFePt}(\mu_3\text{-C}=\text{CHPh})(\text{CO})_6(\text{PPh}_3)$ (**2**) were isolated. Anal. Found: C, 42.89; H, 2.45%. $\text{C}_{37}\text{H}_{26}\text{O}_6\text{PFePtRe}$. Anal. Calc: C, 42.95; H, 2.53%.

IR (C_6H_{12}) $\nu(\text{CO})$ 2054m, 2039s, 2012s, 1972m, 1959s, 1950m, 1936m, 1923w, 1987m, 1873w cm^{-1} .

^1H NMR (–40 °C) δ : 6.60–7.70 (m, 20H, C_6H_5); 6.35 [d^{*}, 1H, $^3\text{J}(\text{PtH})$ 66.4 Hz, $^4\text{J}(\text{PH})$ 1.5 Hz, =CHPh]; 5.39 (s, 5H, C_5H_5). (+20 °C) Isomer **2a**: δ 6.70–7.70 (m, C_6H_5); 6.42 [d^{*}, $^3\text{J}(\text{PtH})$ 66.4 Hz, $^4\text{J}(\text{PH})$ 1.5 Hz, =CHPh]; 5.44 (s, C_5H_5); isomer **2b**: δ 6.70–7.70 (m, C_6H_5); 5.95 [s^{*}, $^3\text{J}(\text{PtH})$ 54.4 Hz, =CHPh]; 5.07 (s, C_5H_5); **2a:2b** ratio is 3.7:1.

$^{31}\text{P}\{^1\text{H}\}$ NMR (–40 °C) δ : 24.90 (s^{*}, $^1\text{J}(\text{PtP})$ 3409 Hz, P). (+20 °C) Isomer **2a**: δ 24.64 [s^{*}, $^1\text{J}(\text{PtP})$ 3424 Hz, P]; isomer **2b**: δ 22.33 [s^{*}, $^1\text{J}(\text{PtP})$ 3490 Hz, P]; **2a:2b** ratio is 3.7:1.

$^{13}\text{C}\{^1\text{H}\}$ NMR (+20 °C) Isomer **2a**: δ 270.94 [s^{*}, $^1\text{J}(\text{PtC}^1)$ 691.6 Hz, $\text{C}^1=\text{CHPh}$]; 201.87 (s, Re–CO); 204.29 (s, Re–CO); 214.67 [s^{*}, $^2\text{J}(\text{PtC})$ 29.3 Hz, Fe–CO]; 213.65 [d^{*}, $^2\text{J}(\text{PtC})$ 12.9 Hz, $^3\text{J}(\text{PC})$ 1.3 Hz, Fe–CO]; 187.79 [d^{*}, $^2\text{J}(\text{PC})$ 6.6 Hz, $^1\text{J}(\text{PtC})$ 1289.8 Hz, Pt–CO]; 147.44 [s^{*}, $^3\text{J}(\text{PtC})$ 16.3 Hz, C_{ipso} of =CHC $_6\text{H}_5$]; 124.5–134.5 (m, C_6H_5); 98.79 [s^{*}, $^2\text{J}(\text{PtC}^2)$ 26.2 Hz, $\text{C}=\text{C}^2\text{HPh}$]; 88.30 (s, C_5H_5); isomer **2b**: δ 267.32 (s, $\text{C}^1=\text{CHPh}$); 206.16 (s, Re–CO); 208.02 (s, Re–CO); 187.36 [d^{*}, $^2\text{J}(\text{PtC})$ 6.6 Hz, Pt–CO]; 143.57 [d, $^4\text{J}(\text{PC})$ 4.5 Hz, C_{ipso} of =CHC $_6\text{H}_5$]; 124.5–134.5 (m, C_6H_5); 93.57 [s, $\text{C}=\text{C}^2\text{HPh}$]; 89.10 (s, C_5H_5).

4.2. X-ray diffraction studies of (1,1,2,2,2,3-hexacarbonyl)-(1- η^5 -cyclopentadienyl)- μ_3 -[1,3- η^1 , η^1 ,2- η^2 -(phenyl)ethenylidene]-(3-triphenylphosphine)-rhenium-iron-platinum(Fe–Re, Fe–Pt) (**2**)

$\text{C}_{37}\text{H}_{26}\text{O}_6\text{PReFePt}$, monoclinic, $\text{C}2/c$, $a = 38.159(15)$, $b = 11.984(5)$, $c = 16.948(7)$ Å, $\beta = 116.171(4)^\circ$, $V = 6956(5)$ Å 3 , $Z = 8$. The experimental data were collected using dark-red fragment of a crystal, dimensions of 0.29 × 0.27 × 0.19 mm, with a SMART Apex II diffractometer (Bruker SMART 4K CCD area detector, graphite monochromator, Mo K α radiation, $\lambda = 0.71073$ Å, ϕ and ω scans, $2\theta \leq 56^\circ$); 33640 reflections were obtained at 293 K, 8386 unique. The experimental completeness is 99.7% in $2\theta \leq 56^\circ$ limits. Absorption corrections ($\mu_{\text{Mo}} = 7.99 \text{ mm}^{-1}$) have been applied using multi-scan procedure [13], $R_{\text{int}} = 0.0216$. The structure was solved by direct methods and refined by full-matrix least squares on F^2 , using SHELXTL program [14]. All phenyl rings were idealized and refined as rigid groups; hydrogen atoms have been placed in calculated positions and taken into account in the final stages of refinement in the “riding model” approximation. Refinement converged at a final $R_1 = 0.0198$ for 7241 reflections with $F_o > 4\sigma_{F_o}$ and 0.0262 for all 8386 data; $wR_2 = 0.0489$, $\text{GoF} = 1.018$, 368 refined parameters.

The supplementary crystallographic data for compound **2** have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 678748. The data can be obtained free of charge via <http://www.ccdc.cam.ac.uk> or e-mail: deposit@ccdc.cam.ac.uk.

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References

- [1] O.S. Chudin, A.B. Antonova, N.I. Pavlenko, W.A. Sokolenko, A.I. Rubaylo, A.D. Vasiliev, O.V. Semeikin, J. Siberian Federal University. Chemistry 1 (2008) 60–70.
- [2] A.B. Antonova, A.A. Ioganson, Usp. Khim. 58 (1989) 1197–1229; Russ. Chem. Rev. 58 (1989) 693–710 (Engl. Transl.).
- [3] M.I. Bruce, Chem. Rev. 91 (1991) 197–257.
- [4] A.B. Antonova, Coord. Chem. Rev. 251 (2007) 1521–1560.
- [5] (a) E. Roland, H. Vahrenkamp, J. Mol. Catal. 21 (1983) 233–237; (b) E. Roland, W. Bernhardt, H. Vahrenkamp, Chem. Ber. 118 (1985) 2858–2873; (c) T. Albiez, W. Bernhardt, C. von Schnering, E. Roland, H. Bantel, H. Vahrenkamp, Chem. Ber. 120 (1987) 141–151.
- [6] A.B. Antonova, A.A. Johansson, N.A. Deykhina, A.G. Ginzburg, E.D. Korniyets, S.V. Kovalenko, N.I. Pavlenko, P.V. Petrovskii, A.I. Rubaylo, I.A. Sukhina, Inorg. Chim. Acta 230 (1995) 97–104.
- [7] A.B. Antonova, A.A. Johansson, N.A. Deykhina, D.A. Pogrebnikov, N.I. Pavlenko, A.I. Rubaylo, F.M. Dolgushin, P.V. Petrovskii, A.G. Ginzburg, J. Organomet. Chem. 577 (1999) 238–242.
- [8] A.D. Vasiliev, O.S. Chudin, A.B. Antonova, Acta Crystallograph. E63 (2007) m2272.
- [9] Cambridge Structural Database System, CSD version 5.29, 2007.

- [10] (a) J. Xiao, J.J. Vittal, R.J. Puddephatt, L. Manojlovic-Muir, K.W. Muir, *J. Am. Chem. Soc.* 115 (1993) 7882;
(b) J. Xiao, R.J. Puddephatt, *Coord. Chem. Rev.* 143 (1995) 457–500.
- [11] A.B. Antonova, Z.A. Starikova, N.A. Deykhina, D.A. Pogrebnyakov, A.I. Rubaylo, *J. Organomet. Chem.* 692 (2007) 1641–1647.
- [12] H. Werner, F.J. Garcia Alonso, H. Otto, K. Peters, H.G. von Schnering, *Chem. Ber.* 121 (1988) 1565–1573.
- [13] G.M. Sheldrick, *SADABS*, Bruker AXS Inc., Madison, Wisconsin, USA, 2004.
- [14] G.M. Sheldrick, *SHELXTL*, Bruker AXS Inc., Madison, Wisconsin, USA, 2004. Version 5.10.