

Cyclopentadienyl derivatives of octahedral carbonylcarbide hexaruthenium clusters: synthesis, NMR, IR and X-ray characterization

V.S. Kaganovich^{a,1}, M.I. Rybinskaya^a, Z.A. Kerzina^{a,*}, F.M. Dolgushin^a, A.I. Yanovsky^a,
Yu.T. Struchkov^{a,2}, P.V. Petrovskii^a, E. Kolehmainen^b, J. Kivikoski^b, J. Valkonen^b,
K. Laihia^b

^a Institute of Organoelement Compounds, Russian Academy of Sciences, 117813, Vavilova street 28, Moscow, Russia

^b Department of Chemistry, University of Jyväskylä PO Box 35, Jyväskylä, FIN-40351, Finland

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Abstract

The reaction of hexanuclear carbonylcarbide cluster $\text{Ru}_6\text{C}(\text{CO})_{17}$ (**1**) with pentamethylcyclopentadiene by reflux in heptane leads to a new chelate cluster $\text{Ru}_6\text{C}(\mu\text{-}\eta^1\text{-}\eta^5\text{-CH}_2\text{C}_5\text{Me}_4)(\text{CO})_{14}$ (**2**). The compound **2** contains a CH_2 group σ -bonded to one of the Ru atoms in the Ru_6 cluster and a C_5Me_4 -ligand η^5 -coordinated by the adjacent Ru atom of the Ru_6 cluster. Reflux of **1** in hexane with two molecular equivalents of nickelocene, $\text{Ni}(\text{C}_5\text{H}_5)_2 = \text{NiCp}_2$, used as a source of cyclopentadiene yields the $\text{Ru}_6\text{C}(\text{CO})_{12}\text{Cp}_2$ (**3**) cluster. The compounds **2** and **3** have been characterized by NMR and IR spectroscopy and their crystal structures determined by X-ray diffraction studies.

Keywords: Ruthenium; Cyclopentadienyl ligands; Semibridging carbonyl group; Crystal structure; CH_2 -Bridging ligand; Cluster

1. Introduction

In a preliminary communication [1] we reported the synthesis of cyclopentadienyl derivatives based on a hexanuclear carbide cluster $\text{Ru}_6\text{C}(\text{CO})_{17}$ (**1**). Now we communicate the details of the synthesis and the results of the spectroscopic (NMR and IR) as well as X-ray diffraction studies of these derivatives.

The π -arene and cyclopentadienyl ligands have always presented special interest in cluster chemistry. Thus the arene derivatives of cluster **1** have been synthesized, extensively studied and reported in numerous papers by authors from the UK and Italy [2–5].

The generation of η^6 -coordinated benzene derivatives of **1** was also described in several of our earlier works [6–9]. However, the cyclopentadienyl derivatives of **1** have received much less attention. In addition to

our preliminary communication [1], only one paper [10] has been published on this topic so far.

2. Results and discussion

The substitution of carbonyl groups by arene ligands in cluster **1** may be promoted either under mild conditions using Me_3NO [3–5] or by thermal excitation [6–9]. In the present work the treatment at elevated temperature was used. The pentamethylcyclopentadiene $\text{C}_5\text{Me}_5\text{H} = \text{Cp}^*\text{H}$, however, was chosen as a reagent for the substitution of CO ligands, since in the conditions of such a reaction the unsubstituted cyclopentadiene generally undergoes dimerization.

The reaction of compound **1** with the excess of Cp^*H was carried out by refluxing the starting materials in hydrocarbon solvents (Eq. (1)). The product was isolated by preparative thin layer chromatography (PTLC) on silica gel. As a result of the reaction, a previously unknown chelate complex **2** was obtained. A very interesting structural detail of **2** was that one of the methyl groups of the cyclopentadienyl ligand was trans-

* Corresponding author.

¹ Deceased on October 6, 1995.

² Deceased on August 16, 1995.

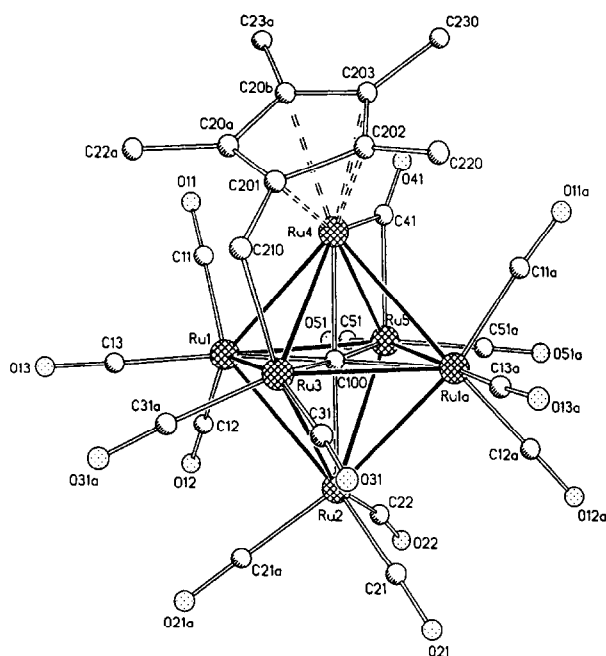
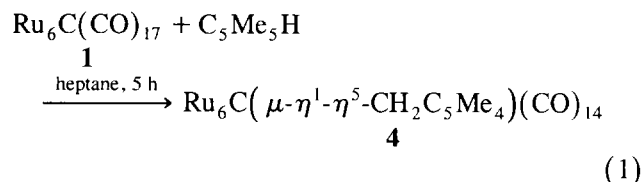


Fig. 1. The molecular structure of $[\text{Ru}_6\text{C}(\mu\text{-}\eta^1\text{-}\eta^5\text{-CH}_2\text{C}_5\text{Me}_4)(\text{CO})_{14}]$ **2** showing the atom labelling scheme. The C atoms of the CO groups bear the same number as the corresponding O atoms.

formed to a CH_2 group, which was σ -bonded to one of the Ru atoms of the Ru_6 cluster, while the C_5Me_4 ligand itself was η^5 -coordinated by the adjacent Ru atom of the Ru_6 cluster.



The isolated compound **2** was characterized by NMR, IR and mass spectra (see Experimental section) and its structure has been determined by X-ray diffraction study.

The single crystal of **2** was grown from a solution of CHCl_3 –heptane mixture by slow evaporation at 5°C . The X-ray crystal structure of compound **2** is shown in Fig. 1 and the bond lengths and angles are collected in Tables 1 and 2 respectively.

The structure of the chelate cluster **2** represents a Ru_6 -octahedron with an encapsulated carbide C(100) atom which is slightly displaced from the geometrical centre of the polyhedron towards the Ru(4) atom coordinated by the Cp^* ligand. The metal–metal bond lengths span the range 2.811–2.997 Å. The C(100)–Ru bond lengths range from 1.998 to 2.088 Å. One of the methyl groups of the Cp^* ligand is transformed by dehydrogenation to a CH_2 group, which forms a σ -bond with the Ru(3), thus producing a four-membered cycle containing the C(210) and C(201) carbon atoms of the Cp ring and the Ru(4) and Ru(3) atoms of the Ru_6 cluster. The Ru(4)–C(Cp) bond lengths span the range

2.208–2.246 Å, whereas the C–C and C–C(Me) bonds in the $\text{C}_5\text{Me}_4\text{CH}_2$ ligand stay within the intervals 1.412–1.449 Å and 1.501–1.506 Å respectively. The carbon atoms of all Me groups are displaced from the average plane of the five-membered cycle (which is planar within 0.006 Å) in the direction opposite to the Ru(4) atom; the displacements of the C(220) and C(230) atoms being equal to 0.23 Å and 0.18 Å respectively. In contrast, the C(210) atom belonging to the methylene group σ -bonded to the Ru(3) atom is displaced from the Cp-ring plane by 0.09 Å in the direction of the Ru(4) atom. The inclination angle of the C(201)–C(210) vector to the Cp-ring plane in **2** is equal to 3.8° , whereas in an di-iridium complex $[(\text{CO})_2\text{IrCp-CH}_2\text{IrCp}(\text{CO})]^+$ [11] featuring a similar bridging CH_2 group the corresponding inclination angle is equal to 8.2° (this value is calculated from the coordinates given in Ref. [11]). The C(201)–C(210) bond length (1.446(12) Å) is significantly shorter than an ordinary σ -bond. It is, however, somewhat longer than the $\text{CH}_2\text{-C}(\text{Cp})$ bond (1.426(7) Å) in the mononuclear cationic chelate complex $[\text{C}_5\text{Me}_5\text{Ru}_5\text{C}_5\text{Me}_4\text{CH}_2]^+$ [12], wherein this bond has a partial double bond character.

It is noteworthy that both methylene and carbonyl bridging groups cause some shortening of the corresponding Ru(3)–Ru(4) and Ru(4)–Ru(5) edges in the metal cluster compared with other metal–metal bond lengths in the octahedron. However, there is no obvious explanation for the shortening of the Ru(1)–Ru(5) bond which does not have any bridging groups. The Ru–

Table 1
Bond lengths (Å) of **2** with standard deviations in parentheses

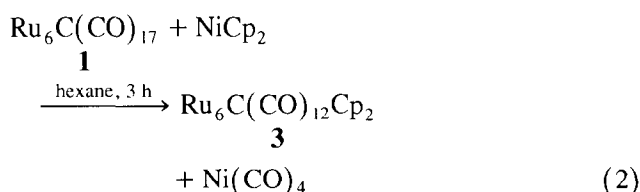
Ru1–Ru2	2.8712(6)	O11–C11	1.143(8)
Ru1–Ru3	2.9970(6)	O12–C12	1.138(9)
Ru1–Ru4	2.8905(6)	O13–C13	1.142(9)
Ru1–Ru5	2.8108(6)	O21–C21	1.133(10)
Ru2–Ru3	2.9270(9)	O22–C22	1.122(13)
Ru2–Ru5	2.9492(9)	O31–C31	1.147(9)
Ru3–Ru4	2.8404(9)	O41–C41	1.176(10)
Ru4–Ru(5)	2.8227(8)	O51–C51	1.142(8)
Ru1–C100	2.0535(7)	C201–C202	1.449(8)
Ru2–C100	2.049(7)	C202–C203	1.412(8)
Ru3–C100	2.025(7)	C201–C210	1.446(12)
Ru4–C100	1.998(7)	C202–C220	1.501(10)
Ru5–C100	2.088(7)	C203–C230	1.506(9)
Ru1–C11	1.894(7)		
Ru1–C12	1.895(7)		
Ru1–C13	1.892(7)		
Ru2–C21	1.892(8)		
Ru2–C22	1.908(11)		
Ru3–C31	1.846(7)		
Ru3–C210	2.240(9)		
Ru4–C41	2.051(8)		
Ru4–C201	2.220(8)		
Ru4–C202	2.208(6)		
Ru4–C203	2.246(5)		
Ru5–C41	2.077(8)		
Ru5–C51	1.879(6)		

Table 2
Selected bond angles (deg) of **2** with standard deviations in parentheses

Ru2–Ru1–Ru3	59.79(2)	C11–Ru1–C12	97.2(3)
Ru2–Ru1–Ru4	89.21(2)	C11–Ru1–C13	92.6(3)
Ru2–Ru1–Ru5	62.53(2)	C11–Ru1–C100	116.3(3)
Ru3–Ru1–Ru4	57.66(2)	C12–Ru1–C13	91.1(3)
Ru3–Ru1–Ru5	90.08(2)	C12–Ru1–C100	123.3(3)
Ru4–Ru1–Ru5	59.33(2)	C13–Ru1–C100	128.4(3)
Ru1–Ru2–Ru1	90.93(2)	C21–Ru2–C21	92.3(3)
Ru1–Ru2–Ru3	62.24(2)	C21–Ru2–C22	97.3(3)
Ru1–Ru2–Ru5	57.73(2)	C21–Ru2–C100	124.7(2)
Ru3–Ru2–Ru5	88.81(2)	C22–Ru2–C100	114.1(4)
Ru1–Ru3–Ru1	86.14(2)	C31–Ru3–C31	86.7(3)
Ru1–Ru3–Ru2	57.97(2)	C31–Ru3–C100	128.9(2)
Ru1–Ru3–Ru4	59.29(2)	C31–Ru3–C210	89.3(3)
Ru2–Ru3–Ru4	89.09(2)	C100–Ru3–C210	121.1(3)
Ru1–Ru4–Ru1	90.16(2)	C41–Ru4–C100	94.9(3)
Ru1–Ru4–Ru3	63.05(2)	C41–Ru4–C201	152.9(3)
Ru1–Ru4–Ru5	58.93(1)	C41–Ru4–C202	123.3(2)
Ru3–Ru4–Ru5	93.12(2)	C41–Ru4–C203	91.5(3)
Ru1–Ru5–Ru1	93.46(2)	C100–Ru4–C201	112.2(3)
Ru1–Ru5–Ru2	59.74(2)	C100–Ru4–C202	127.1(2)
Ru1–Ru5–Ru4	61.74(2)	C100–Ru4–C203	160.4(2)
Ru2–Ru5–Ru4	88.98(2)	C201–Ru4–C202	38.2(2)
		C201–Ru4–C203	62.9(2)
		C202–Ru4–C203	36.9(2)
		C41–Ru5–C51	96.3(3)
		C41–Ru5–C100	91.5(3)
		C51–Ru5–C51	89.3(3)
		C51–Ru5–C100	134.4(2)
Ru1–C100–Ru1	170.6(4)	Ru1–C11–O11	176.0(6)
Ru1–C100–Ru2	88.8(2)	Ru1–C12–O12	179.6(9)
Ru1–C100–Ru3	94.6(2)	Ru1–C13–O13	173.5(6)
Ru1–C100–Ru4	91.0(2)	Ru2–C21–O21	178.5(7)
Ru1–C100–Ru5	85.5(2)	Ru2–C22–O22	170.4(9)
Ru2–C100–Ru3	91.9(3)	Ru3–C31–O31	177.2(7)
Ru2–C100–Ru4	178.3(4)	Ru4–C41–Ru5	86.3(3)
Ru2–C100–Ru5	90.9(3)	Ru4–C41–O41	138.4(7)
Ru3–C100–Ru4	89.8(3)	Ru5–C41–O41	135.4(7)
Ru3–C100–Ru5	177.2(4)	Ru5–C51–O51	176.9(5)
Ru4–C100–Ru5	87.4(3)		
Ru4–C201–C202	70.4(4)		
Ru4–C201–C203	59.1(2)		
Ru4–C201–C210	118.7(6)		
Ru4–C202–C201	71.4(4)		
Ru4–C202–C203	73.0(3)		
Ru4–C202–C220	132.0(4)		
Ru4–C203–C201	58.0(2)		
Ru4–C203–C202	70.1(3)		
Ru4–C203–C230	130.5(4)		
C202–C201–C202	105.5(6)		
C202–C201–C210	127.1(4)		
C201–C202–C203	109.1(5)		
C201–C202–C220	125.1(5)		
C203–C202–C220	124.6(6)		
C202–C203–C230	126.3(6)		
Ru3–C210–C201	98.1(5)		

C(CO) bond lengths in **2** are not exceptional; those involving bridging CO groups (2.051–2.077 Å) are naturally significantly longer than the corresponding bonds in the terminal carbonyls (1.841–1.908 Å).

We employed pentamethylcyclopentadiene in the above-mentioned reaction with **1** in order to avoid the dimerization process, which could have occurred in the case of the unsubstituted cyclopentadiene. As the purpose of our research was to study the synthesis of cluster **1** substituted by η^5 -cyclopentadienyl ligand, we decided to use nickelocene as a possible source of the Cp ligand. Nickelocene may also act as the acceptor of carbonyl groups, which are thus bound in the form of Ni(CO)₄. Reaction of **1** with nickelocene was carried out by reflux in hexane for 3 h. The bis(cyclopentadienyl) cluster Ru₆C(CO)₁₂Cp₂ (**3**) was isolated by means of PTLC on silica gel.



An X-ray diffraction study confirmed the structure of **3**. Details of this study are not given since, following completion, the structure was reported by Johnson and coworkers [10]. The metal cluster **3** consists of six octahedrally arranged Ru atoms and contains an interstitial carbide atom; such a cage is typical for derivatives of cluster **1** [13–15]. Johnson and coworkers synthesized cluster **3** by a different route, involving reaction of compound **1** with unsubstituted cyclopentadiene under mild conditions in the presence of trimethylamine oxide [10].

3. Experimental section

3.1. Syntheses

All reactions were carried out in Ar atmosphere using absolute dry solvents. Hexaruthenium carbonyl-carbide complex **1** was prepared according to a previously known method [16].

3.1.1. Ru₆C(μ-η¹-η⁵-CH₂C₅Me₄)(CO)₁₄ (**2**)

The mixture of 120 mg (0.1 mmol) of **1** and 400 mg (0.3 mmol) of C₅Me₅H in 50 ml of heptane was refluxed for 5 h. The solvent was evaporated under reduced pressure. The residue was isolated by TLC on silica gel. From the first brown zone, 4.2 mg (3.4%) of complex **2** was separated. Spectral data of **2**. (1) IR spectrum, ν(CO) stretching: 2072m, 2042s, 2024s, 2016s, 2004w, 1986w, 1978w, 1820m cm⁻¹ (in heptane). (2) Mass spectrum, [m/e]⁺-values: complex pattern of peaks from 1146 to 747 (with intensity greater than 15% when the highest peak at 747 is 100%), molecular ion [M]⁺ = 1146 and further successive loss of 14 carbonyl groups were observed. ¹H NMR spec-

Table 3
Crystallographic data and details of measurements for $[\text{Ru}_6\text{C}(\mu\text{-}\eta^1\text{-}\eta^5\text{-CH}_2\text{C}_5\text{Me}_4\text{XCO})_{14}] \mathbf{2}$

Crystal data	2
Molecular formula	$\text{C}_{25}\text{H}_{16}\text{O}_{14}\text{Ru}_6$
Formula weight	1146.82
Crystal system	Orthorhombic
Space group	<i>Pnma</i>
<i>a</i> (Å)	12.698(5)
<i>b</i> (Å)	15.928(4)
<i>c</i> (Å)	15.681(5)
α (deg)	90
β (deg)	90
γ (deg)	90
<i>V</i> (Å ³)	3171(2)
<i>Z</i>	4
<i>d</i> _{calc.} (g cm ⁻³)	2.40
μ (cm ⁻¹)	28.0
Data collection	
$2\theta_{\text{max}}$ (deg)	56
Collected data	5275
Unique data	4274
Observed data, for $I > 3\sigma(I)$	2729
Absorption correction	max 1.000 min 0.8296
Refinement	
Number of parameters	221
Final <i>R</i> (%)	2.7
<i>R</i> _w (%)	3.1

trum, chemical shifts (δ ppm) in CDCl_3 from TMS: 1.52s (Me), 2.10s (CH_2) with intensity ratio of 6:1. ¹³C NMR spectrum of **2** showed some weak signals but owing to a low S/N ratio these values are not reported here.

3.1.2. $\text{Ru}_6\text{C}(\eta^5\text{-C}_5\text{H}_5)_2(\text{CO})_{12}$ (**3**)

The mixture of 120 mg (0.1 mmol) of **1** and 50 mg (0.26 mmol) of nickelocene was refluxed in 60 ml of hexane for 3 h. The solvent was evaporated under reduced pressure. The residue was chromatographed by the same procedure as above. The second of the two brown zones gave 6.5 mg (yield 6.0%) of complex **3**. IR spectrum, $\nu(\text{CO})$ stretching: 2058m, 2026s, 1978m, 1962m, 1876w cm⁻¹ (in heptane).

3.2. Spectroscopy

The mass spectrum of **2** was obtained with a high-resolution VG Autospec mass spectrometer using direct inlet and 35 eV electron impact ionization. ¹H and ¹³C NMR spectra were recorded on a Jeol JNM GSX 270 FT NMR spectrometer working at 270.17 (¹H) and 67.94 (¹³C) MHz. Preliminary experiments were done with a Bruker WP-200 SY spectrometer. IR spectra

Table 4
Fractional atomic coordinates and equivalent isotropic temperature factors U_{iso} of **2** with standard deviations in parentheses

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
Ru1	0.78261(3)	0.12145(3)	0.52484(3)	0.0328(2)
Ru2	0.79864(5)	0.2500	0.65262(4)	0.0360(3)
Ru3	0.61175(5)	0.2500	0.54337(4)	0.0352(3)
Ru4	0.74550(5)	0.2500	0.39817(4)	0.0295(2)
Ru5	0.93060(4)	0.2500	0.49783(4)	0.0306(3)
O11	0.8593(4)	0.0379(3)	0.3623(3)	0.077(3)
O12	0.9287(4)	0.0264(3)	0.6436(3)	0.086(3)
O13	0.6180(4)	-0.0140(3)	0.5500(5)	0.096(4)
O21	0.7298(6)	0.3857(4)	0.7755(4)	0.119(5)
O22	1.0258(6)	0.2500	0.7097(5)	0.087(6)
O31	0.4768(5)	0.3766(4)	0.6327(4)	0.105(4)
O41	0.9552(5)	0.2500	0.3057(4)	0.063(4)
O51	1.1031(4)	0.1194(3)	0.4959(4)	0.079(3)
C11	0.8279(5)	0.0709(4)	0.4218(4)	0.051(3)
C12	0.8742(5)	0.0622(4)	0.5989(4)	0.056(4)
C13	0.6755(5)	0.0405(4)	0.5414(5)	0.059(4)
C21	0.7563(6)	0.3357(5)	0.7288(4)	0.068(4)
C22	0.9446(8)	0.2500	0.6814(6)	0.065(6)
C31	0.5294(5)	0.3296(5)	0.5971(5)	0.060(4)
C41	0.9024(6)	0.2500	0.3673(5)	0.042(4)
C51	1.0358(4)	0.1670(4)	0.4962(4)	0.048(3)
C100	0.7694(5)	0.2500	0.5241(4)	0.028(3)
C201	0.5755(6)	0.2500	0.3651(5)	0.043(4)
C202	0.6260(4)	0.3225(4)	0.3270(4)	0.045(3)
C203	0.7015(5)	0.2948(4)	0.2671(3)	0.044(3)
C210	0.4990(6)	0.2500	0.4335(6)	0.049(5)
C220	0.5884(5)	0.4115(5)	0.3352(4)	0.065(4)
C230	0.7610(6)	0.3492(5)	0.2051(4)	0.067(4)

were recorded on a Specord 75 IR spectrometer in heptane solutions.

3.3. Crystal structure determination

The crystals of clusters **2** and **3** were grown from their solutions in the mixtures of CH₂Cl₂–heptane or CHCl₃–heptane by slow evaporation of the solvents in refrigerator at +5°C. X-ray diffraction measurements of **2** and **3** were made on an Enraf–Nonius CAD-4 and Siemens P3/PC diffractometers respectively (graphite monochromator, MoK α radiation, $\lambda = 0.71073$ Å, θ – 2θ scan mode, $T = 295$ K). Crystallographic data and details of data collection and structure refinement for **2** are summarized in Table 3. The atomic coordinates for compound **2** are listed in Table 4. The structure of **2** was solved by direct methods and refined in the anisotropic approximation using the MolEN [17] program. Empirical (Ψ -scan) absorption correction for **2** was applied using five reflections. The hydrogen atoms in **2** bonded to C(220) and C(230) were placed geometrically (C–H distance 0.95 Å) and refined in the riding model approximation with the common fixed temperature factor $U_{\text{iso}} = 0.063$ Å. Hydrogen atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited at the Cambridge Crystallographic Data Centre.

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References

- [1] V.S. Kaganovich, Z.A. Kerzina, M.I. Rybinskaya and E. Kolehmainen, *Izv. Akad. Nauk Ser. Khim.*, **6** (1995) 1171.
- [2] M.P. Gomez-Sal, B.F.G. Johnson, J. Lewis, P.R. Raithby and A.H. Wright, *J. Chem. Soc. Chem. Commun.*, (1985) 1682.
- [3] P.J. Dyson, B.F.G. Johnson, J. Lewis, M. Martinelli, D. Braga and F. Grepioni, *J. Am. Chem. Soc.*, **115** (1993) 9062.
- [4] D. Braga, F. Grepioni, P. Sabatino, P.J. Dyson, B.F.G. Johnson, J. Lewis, P.J. Bailey, P.R. Raithby and D. Stalke, *J. Chem. Soc. Dalton Trans.*, (1993) 985.
- [5] D. Braga, P. Sabatino, P.J. Dyson, A.J. Blake and B.F.G. Johnson, *J. Chem. Soc. Dalton Trans.*, (1994) 393.
- [6] V.S. Kaganovich, Z.A. Kerzina, T. Asunta, K. Wickström and M.I. Rybinskaya, *J. Organomet. Chem.*, **421** (1991) 117.
- [7] E. Kolehmainen, K. Laihia, M.I. Rybinskaya, V.S. Kaganovich and Z.A. Kerzina, *J. Organomet. Chem.*, **453** (1993) 273.
- [8] E. Kolehmainen, K. Laihia, J. Korvola, V.S. Kaganovich, M.I. Rybinskaya and Z.A. Kerzina, *J. Organomet. Chem.*, **485** (1995) 109.
- [9] E. Kolehmainen, K. Laihia, J. Korvola, V.S. Kaganovich, M.I. Rybinskaya and Z.A. Kerzina, *J. Organomet. Chem.*, **487** (1995) 215.
- [10] A.J. Blake, P.J. Dyson, R.C. Gash, B.F.G. Johnson and P.J. Trickey, *J. Chem. Soc. Dalton Trans.*, (1994) 1105.
- [11] F.W.B. Einstein, R.H. Jones, X. Zhang, X. Yan, R. Nagelkerke and D. Sutton, *J. Chem. Soc. Chem. Commun.*, (1989) 1424.
- [12] A.J. Yanovsky, Yu.T. Struchkov, A.Z. Kreindlin and M.I. Rybinskaya, *J. Organomet. Chem.*, **369** (1989) 125.
- [13] D. Braga, F. Grepioni, P.Y. Dyson, B.F.G. Johnson, P. Fnediani, M. Bianchi and F. Piacenti, *J. Chem. Soc. Dalton Trans.*, (1992) 2565.
- [14] R. Mason and W.R. Robinson, *J. Chem. Soc. Chem. Commun.*, (1968) 468.
- [15] L.J. Farrugia, *Acta Crystallogr. Sect. C*, **44** (1988) 997.
- [16] V.S. Kaganovich, V.A. Petrakova and M.I. Rybinskaya, *Izv. Akad. Nauk Ser. Khim.*, (1990) 1893 (*Bull. Acad. Sci. USSR Div. Chem. Sci.*, **39** (1990) 1720) and references cited therein.
- [17] C.K. Fair, *MolEN, An interactive intelligent system for crystal structure analysis*, Enraf–Nonius, Delft, Netherlands, 1990.