Journal of Organometallic Chemistry, 441 (1992) 277–284
Elsevier Sequoia S.A., Lausanne
JOM 22944

Reaction of $Ru_3(CO)_{12}$ with ferrocenylacetylene. Crystal and molecular structure of $Ru_2(CO)_6[C_4H_2(C_5H_4FeC_5H_5)_2]$

A.A. Koridze, A.I. Yanovsky and Yu.T. Struchkov

A.N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, Vavilov St. 28, 117813 Moscow (Russian Federation)

(Received April 17, 1992)

Abstract

The reaction of $Ru_3(CO)_{12}$ with ferrocenylacetylene FcC≡CH in refluxing hexane gives $Ru_3(\mu\text{-}H)(CO)_9(\mu_3\text{-}\eta^2\text{-}C{\equiv}CFc)$ (I), $Ru_2(CO)_6[C_4H_2(Fc)_2]$ (II) and $Ru_2(CO)_6[\mu\text{-}C(H){=}C(Fc)COC(H){=}C(Fc)]$ (III). Complexes I–III were characterized by IR and NMR spectroscopy, and II also by X-ray diffraction. Complex II crystallizes in the space group $Cmc2_1$ with $a = 20.072(4)$, $b = 14.322(2)$, $c = 9.703(2)$ Å, and $Z = 4$. The structure was solved by the heavy-atom method and refined by least-squares analysis to $R = 0.031$ and $R_w = 0.037$ for 1677 unique observed reflexions. Molecule II contains a ruthenacyclopentadiene ring, π-bonded to a $Ru(CO)_3$ group. The $Ru_2(CO)_6$ moiety adopts a "sawhorse" geometry, in contrast to the known structures of ruthenium complexes of this type, which have a "non-sawhorse" structure. Compound III possesses a nonsymmetrical diruthenacycloheptadienone moiety. The ^1H NMR spectroscopy of III did not reveal any fluxionality of the "flyover" bridge leading to the exchange of σ- and π-bonds at two ruthenium atoms.

Introduction

Previously, we obtained several osmium cluster complexes containing a ferrocene moiety, by interaction of the triosmium cluster $Os_3(\mu\text{-}H)_2(CO)_{10}$ with ferrocenylacetylene FcC≡CH, and studied some chemical properties of these compounds [1,2]. For a comparative study of the chemical and spectroscopic properties of ferrocene-containing osmium and ruthenium clusters, we have synthesized the corresponding ruthenium complexes by the thermal reaction of $Ru_3(CO)_{12}$ with ferrocenylacetylene. Some preliminary results of this work have already been published [3].

Experimental

$Ru_3(CO)_{12}$ and FcC≡CH were prepared according to published procedures [4,5].

Correspondence to: Dr. A.A. Koridze.

IR spectra were recorded on a Bruker IFS-113v spectrometer, and ^{1}H and ^{13}C NMR spectra on a Bruker WP-200SY instrument. TLC of reaction products was carried out on SiO_2 L 40/100μ (Chemapol, Czechoslovakia).

Synthesis

Reaction of $Ru_3(CO)_{12}$ with ferrocenylacetylene. A solution of 320 mg (0.5 mmol) of ruthenium carbonyl and 315 mg (1.5 mmol) of ferrocenylacetylene in 100 ml of hexane was refluxed for 0.5 h. After cooling, the solution was filtered and the solvent was removed under reduced pressure. The residue was dissolved in a minimum of benzene and loaded on a silica gel chromatography column. With hexane as eluting solvent, a yellow band containing $Ru_3(CO)_{12}$, red-orange $Ru_3(\mu\text{-}H)(CO)_9(\mu_3\text{-}\eta^2\text{-}C{\equiv}CFc)$ (I) ($\sim$ 15%), red-brown $Ru_2(CO)_6[C_4H_2(Fc)_2]$ (II) ($\sim$ 10%), and ferrocenylacetylene were eluted in that order. The remaining red-violet band was eluted with toluene. The toluene was evaporated at reduced pressure and the residue was dissolved in CH_2Cl_2 and separated by TLC. One of the fractions eluted with a petroleum ether/toluene (3:2) mixture, consisted of three unidentified complexes formed in very low yield. Subsequent elution gave a red fraction, containing the complex, $Ru_2(CO)_6[\mu\text{-}C(H){=}C(Fc)COC(H){=}C(Fc)]$ (III) ($\sim$ 5%).

$Ru_3(\mu\text{-}H)(CO)_9(\mu_3\text{-}\eta^2\text{-}C{\equiv}CFc)$ (I). Red-orange crystals. Anal. Found: C, 33.29; H, 1.29. $C_{21}H_{10}FeO_9Ru_3$ calc.: C, 32.75; H, 1.32%. Infrared (hexane): ν(CO) 2100m, 2078vs, 2060vs, 2030vs, 1996m cm^{-1}. ^{1}H NMR ($CDCl_3$): δ -20.466 (s, 1 H); 4,198 (s, 5H); 4.429 (t, 2H); 4.331 (t, 2H).

$Ru_2(CO)_6[C_4H_2(C_5H_4FeC_5FeC_5H_5)_2]$ (II). Red-brown crystals. Anal. Found: C, 45.87; H, 2.76; Fe, 13.93. $C_{30}H_{20}Fe_2O_6Ru_2$ calc.: C, 45.59; H, 2.55; Fe, 14.13%. Infrared (hexane): ν(CO) 2070m, 2040vs, 1980s, 1975m,sh cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 4.034 (m, 1H); 4.108 (s, 5H); 4.175 (m, 1H); 4.231 (t, 2H); 7.198 (s, 2H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 68.22, 68.90 ($C_{\alpha,\alpha'}$); 69.42 (C_5H_5); 70.81, 71.02 ($C_{\beta,\beta'}$); 83.90 (C_i); 130.54 ($>\!C{=}$); 141.93 (CH); 195.85, 197.02 (CO).

$Ru_2(CO)_6[\mu\text{-}C(H){=}C(Fc)COC(H){=}C(Fc)]$ (III). Deep-red plates. Anal. Found: C, 46.00; H, 2.69. $C_{31}H_{20}Fe_2O_7Ru_2$ calc.: C, 45.50; H, 2.46%. Infrared (hexane): ν(CO) 2087m, 2061vs, 2022vs,sh, 2012m, 2002m, 1680m,br cm^{-1}. ^{1}H NMR (acetone-d_6): δ 4.110 (s, 5H); 4.167 (m, 1H); 4.282 (s, 5H); 4.286 (m, 1h); 4.376 (m, 1H); 4.533 (t, 2H); 4.178 (m, 1H); 4.784 (m, 1H); 4.904 (m, 1H); 5.359 (s, 1H); 9.100 (s, 1H).

Preparation of $Os_3(\mu\text{-}H)(CO)_9(\mu_3\text{-}\eta^2\text{-}C{\equiv}CFc)$ (IV). This complex was obtained by thermolysis of the known cluster $Os_3(CO)_9(\mu\text{-}CO)(\mu_3\text{-}\eta^2\text{-}HC_2Fc)$ [1] in refluxing octane. The yellow product was characterized by its ^{1}H NMR spectrum ($CDCl_3$): δ -22.870 (s, 1H); 4.192 (s, 5H); 4.316 (t, 2H); 4.413 (t, 2H).

X-Ray diffraction study of II

Crystals of **II** are orthorhombic, at +23°C, $a = 20.072(4)$, $b = 14.322(2)$, $c = 9.703(2)$ Å, $V = 2789.4(5)$ Å^3, $Z = 4$ ($C_{30}H_{20}Fe_2O_6Ru_2$), d_{calc} 1.822 mg m^{-3}, space group $Cmc2_1$. The unit cell dimensions and intensities of 1677 reflexions with $F^2 \geq 3\sigma(F^2)$ were measured with a Siemens P3/PC diffractometer (+23°C, λ(Mo-K_α), graphite monochromator, θ–2θ-scan, $\theta \leq 27°$, no absorption correction (μ(Mo-K_α) = 21.2 cm^{-1}) was applied). The structure was solved by means of a standard heavy-atom procedure. The coordinates of both independent Ru atoms

Table 1

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{Å}^2 \times 10^3$) in molecule **II**

	x	y	z	U_{eq} [a]
Ru(1)	5000	8329(1)	10000	48(1)
Ru(2)	5000	7397(1)	7557(1)	42(1)
Fe	6875(1)	6421(1)	11052(1)	39(1)
O(1)	5000	8166(10)	13096(12)	143(7)
O(2)	6084(3)	9799(4)	9462(9)	107(3)
O(3)	5000	5979(7)	5223(10)	99(4)
O(4)	6122(3)	8575(6)	6404(10)	120(3)
C(1)	5000	8244(10)	11946(15)	84(5)
C(2)	5693(4)	9262(4)	9711(10)	71(3)
C(3)	5000	6495(8)	6109(12)	61(3)
C(4)	5697(3)	8135(6)	6790(9)	71(3)
C(5)	5656(3)	7247(3)	9582(5)	38(1)
C(6)	5348(3)	6413(4)	9146(6)	40(1)
C(7)	6384(3)	7268(4)	9702(5)	41(2)
C(8)	6852(3)	6683(4)	8998(6)	45(2)
C(9)	7495(3)	6854(4)	9510(6)	50(2)
C(10)	7439(3)	7546(4)	10543(6)	48(2)
C(11)	6767(3)	7807(4)	10667(7)	48(2)
C(12)	6186(3)	5666(5)	12097(7)	56(2)
C(13)	6589(3)	5070(5)	11325(6)	58(2)
C(14)	7250(4)	5195(5)	11784(8)	65(2)
C(15)	7244(4)	5866(5)	12827(6)	60(2)
C(16)	6599(4)	6172(5)	13033(7)	59(2)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

(occupying special positions on the mirror plane) and an Fe atom in general position were derived from the Patterson synthesis. All other non-hydrogen atoms were located in the subsequent approximations of electron density synthesis and

Table 2

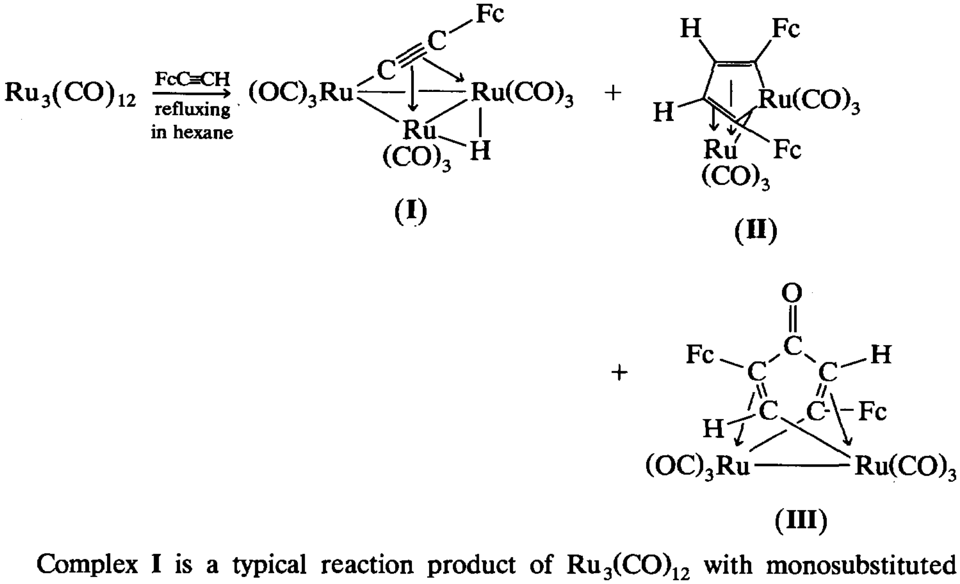
Bond lengths (Å) in molecule **II**

Ru(1)–Ru(2)	2.721(1)	Ru(1)–C(1)	1.892(14)
Ru(1)–C(2)	1.949(7)	Ru(1)–C(5)	2.074(5)
Ru(2)–C(3)	1.909(11)	Ru(2)–C(4)	1.906(8)
Ru(2)–C(5)	2.375(5)	Ru(2)–C(6)	2.203(5)
Fe–C(7)	2.040(6)	Fe–C(8)	2.029(6)
Fe–C(9)	2.042(6)	Fe–C(10)	2.029(7)
Fe–C(11)	2.032(6)	Fe–C(12)	2.029(7)
Fe–C(13)	2.036(7)	Fe–C(14)	2.038(7)
Fe–C(15)	2.036(7)	Fe–C(16)	2.032(7)
O(1)–C(1)	1.121(19)	O(2)–C(2)	1.125(9)
O(3)–C(3)	1.134(15)	O(4)–C(4)	1.124(11)
C(5)–C(6)	1.411(7)	C(5)–C(7)	1.465(8)
C(6)–C(6a)	1.398(11)	C(7)–C(8)	1.433(8)
C(7)–C(11)	1.437(8)	C(8)–C(9)	1.404(9)
C(9)–C(10)	1.414(9)	C(10)–C(11)	1.404(9)
C(12)–C(13)	1.395(10)	C(12)–C(16)	1.428(10)
C(13)–C(14)	1.411(10)	C(14)–C(15)	1.396(10)
C(15)–C(16)	1.382(11)		

refined by full-matrix least-squares technique, first in the isotropic and then in the anisotropic approximation. The H atoms placed in the calculated positions were included in the final cycles of refinement in the riding model approximation with the common isotropic temperature factor $U_{iso} = 0.08$ Å^2. The final discrepancy factors are $R = 0.031$, $R_w = 0.037$. All calculations were performed with the SHELXTL (PC version) program package [6]. Atomic coordinates are listed in Table 1, bond lengths are given in Table 2.

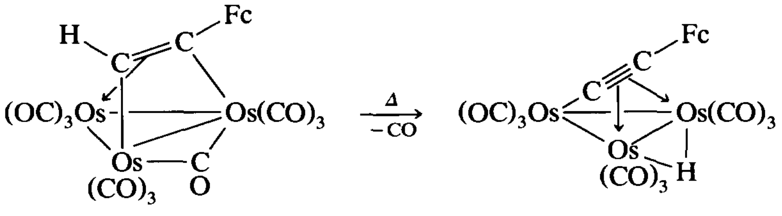
Results and discussion

Reaction of $Ru_3(CO)_{12}$ with ferrocenylacetylene in refluxing hexane gives a complex mixture of products, from which three organometallic compounds can be isolated by chromatography: $Ru_3(\mu\text{-H})(CO)_9(\mu_3\text{-}\eta^2\text{-C≡CFc})$ (I), $Ru_2(CO)_6[C_4H_2(Fc)_2]$ (II) and $Ru_2(CO)_6[\mu\text{-C(H)=C(Fc)COC(H)=C(Fc)}]$ (III). These were indentified on the basis of elemental analysis and spectroscopic (IR, NMR) data. Complex **II** was also characterized by an X-ray diffraction study.



Complex **I** is a typical reaction product of $Ru_3(CO)_{12}$ with monosubstituted alkynes; clusters of the same structure were previously obtained by interaction of ruthenium carbonyl with phenyl-, tert-butyl- [7] and trimethylsilylacetylene [8]. The structure of the $Ru_3(\mu\text{-H})(CO)_9(\mu_3\text{-}\eta^2\text{-C≡C}^t\text{Bu})$ complex has been unequivocally established by diffraction methods [9,10]. It is believed that formation of the complexes of the type **I** proceeds *via* the intermediates with the $2\sigma,\pi$-coordinated alkyne ligand, $Ru_3(CO)_9(\mu_3\text{-}\eta^2\text{-HC}_2R)$ complexes. Recently this belief received additional support in a study of the reaction of $Ru_3(CO)_{10}(NCMe)_2$ with alkynes RC≡CH. Complexes $Ru_3(CO)_9(\mu\text{-CO})(\mu_3\text{-}\eta^2\text{-HC}_2R)$, obtained in this reaction in high yield, readily decarbonylated on heating to give $Ru_3(\mu\text{-H})(CO)_9(\mu_3\text{-}\eta^2\text{-C≡CR})$ clusters as a result of the oxidative addition of a C–H bond to the trimetallic core [11].

The $Os_3(\mu\text{-H})(CO)_9(\mu_3\text{-}\eta^2\text{-C≡CFc})$ (IV) cluster, the osmium analogue of complex I, was obtained by thermolysis of $Os_3(CO)_9(\mu\text{-CO})(\mu_3\text{-}\eta^2\text{-HC}_2Fc)$, one of the products of the reaction of $Os_3(\mu\text{-}H)_2(CO)_{10}$ dihydride with ferrocenylacetylene [1].



(IV)

The presence of a ferrocenyl group in isostructural clusters I and IV allows the use of the ferrocene moiety as an indicator for estimating the electronic effect of the triruthenium and triosmium core on the μ_3-η^2-(5e)-coordinated acetylide ligand.

In the ^1H NMR spectrum of the ruthenium cluster I, the resonance of the ferrocene unsubstituted ring appears at δ 4.198, whereas corresponding resonance for the osmium analogue IV is observed at 4.192 ppm. There is a somewhat more marked difference in the shielding of the protons of substituted cyclopentadienyl rings in complexes I and IV: δ 4.331 and 4.429 in ruthenium, and 4.316 and 4.413 ppm in the osmium complex. Hence, the protons of the ferrocenyl moiety are somewhat more deshielded in complex I, which may promote the conclusion that the triruthenium cluster more effectively accepts electron density from the μ_3-η^2-acetylide ligand than its triosmium analogue. Although the results of the electrochemical oxidation of the ferrocene nucleus in complexes I and IV [12] are not in contradiction with this conclusion, the differences in proton shielding of the Fc-moiety in I and IV are too small for any definite conclusion.

Complex II was identified as a ferrole-type derivative with a ruthenacyclopentadiene ring π-coordinated to the $Ru(CO)_3$ group. This compound was characterized by ^1H and ^{13}C NMR spectra. In the ^{13}C NMR spectrum, resonances of two $Ru(CO)_3$ groups are observed as two signals at 195.85 and 197.05 ppm, implying fast localized exchange of carbonyl ligands, just as has been found for the related iron derivatives [13].

Complexes of the type II are quite frequently formed in reactions of iron and ruthenium carbonyls with mono-, as well as disubstituted acetylenes [14,15]. Until now, only $Ru_2(CO)_6L_2$ complexes, formed in the reactions of $Ru_3(CO)_{12}$ with disubstituted acetylenes $MeOOCC{\equiv}CCOOMe$, $HOCH_2C{\equiv}CCH_2OH$ and $EtC{\equiv}CCH_2CH_2OH$, were characterized in detail, including X-ray diffraction studies [16,17]. Formation of the isomeric mixture of such ruthenium species $Ru_2(CO)_6L_2$ ($L{=}HC{\equiv}CCMe_2OH$) was reported [18]. The isomerism of such complexes is associated with possible coupling modes of alkyne ligands, *viz.* "head-to-head", "head-to-tail" and "tail-to-tail" coupling. As no X-ray diffraction studies of the complexes of the $Ru_2(CO)_6L_2$ type formed in the reaction with monosubstituted acetylenes have as yet been published, it was of interest to study complex II by this method.

The X-ray diffraction study of complex II confirmed its structure as a molecule of ferrole-type (Fig. 1). Molecule II occupies a special position on the mirror plane which passes through the Ru(1), Ru(2), C(1), C(3) and O(3) atoms. The ruthenacy-

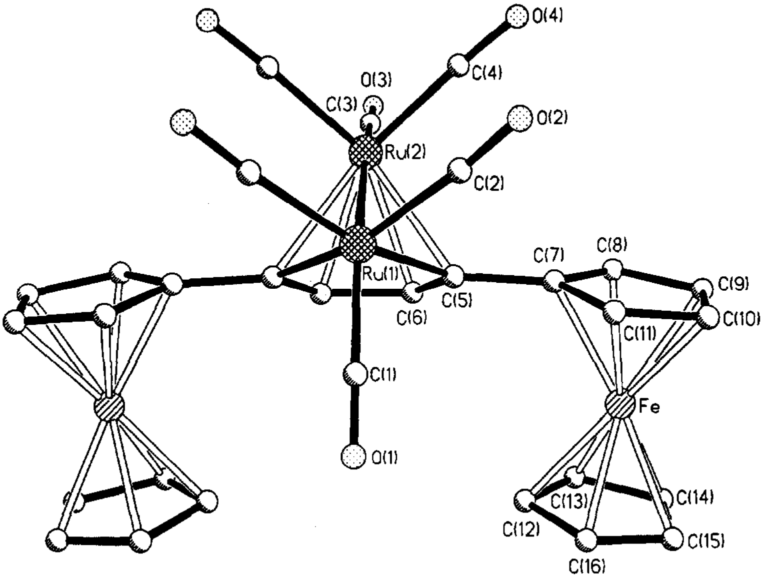
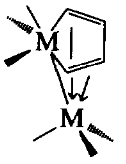


Fig. 1. Molecular structure of complex **II**.

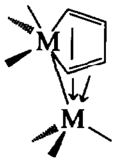
clopentadiene cycle is obviously the product of a "head-to-head" alkyne coupling which brings the ferrocenyl substituents to the α-carbons of the heterocyclic ring.

The C(5)–C(6) and C(6)–C(6a) (C(Na) atoms are related to corresponding C(N) atoms by the mirror plane) bond distances (1.411(7) and 1.398(11) Å) are in good agreement with the corresponding distances observed earlier in $Ru_2(CO)_6$-$[C_4(COOMe)_4]$ (**V**) (average 1.42 Å) [16], $Ru_2(CO)_6[C_4(CH_2OH)_4]$ (**VI**), and $Ru_2(CO)_6[C_4(CH_2CH_2OH)(C_2H_5)_2]$ (**VII**) (average 1.43 Å in both **VI** and **VII**) [17], and indicate effective π-electron delocalization in the diene group of the metallocycle. The Ru–Ru bond length (2.721(1) Å) in complex **II** is within the range of 2.717–2.753 Å found for the metal–metal bonds in **V**–**VII**.

It is known that ferrole-type complexes usually have a "sawhorse" geometry **a**, with staggered carbonyls of two $M(CO)_3$ groups, and a semi-bridging CO ligand. The alternative "non-sawhorse" structure **b** occurs quite rarely. Structure **b** with the eclipsed CO groups may be derived from **a** by rotation of the π-coordinated $Ru(CO)_3$ group by 60°.



(a)



(b)

In contrast to complexes **V**–**VII** with the **a** type conformation complex **II** has a **b** type structure. The absence of a semi-bridging carbonyl group in **II** confirms the suggestion [16,17] that the energy difference between these two structures is rather small.

It is also noteworthy that in contrast to complexes V–VII, wherein the endocyclic Ru atom is displaced from the butadiene plane in the direction opposite to the second Ru atom, in molecule **II** both ruthenium atoms are on the same side of the C(5)C(6)C(5a)C(6a) plane; the displacement of the Ru(1) atom from this plane is 0.134(1) Å.

Ferrocenyl groups have unexceptional geometry. Cyclopentadienyl ligands have almost ideally eclipsed orientation; the dihedral angle between the planes of the substituted cyclopentadienyl ring (planar within 0.002 Å) and the butadiene moiety is 17.3°.

We have identified complex **III** as a diruthenacycloheptadienone derivative, $Ru_2(CO)_6[\mu\text{-}C(H)=C(Fc)COC(H)=C(Fc)]$. The infrared spectrum of **III** shows absorption of the keto-group at 1680 cm^{-1}. In the ^1H NMR spectrum, the resonances of two non-equivalent ferrocenyl groups, and two singlets of ethylene protons at δ 5.59 and 9.10 ppm are observed at room temperature. The downfield peak at 9.10 ppm can be assigned to the hydrogen atom at the σ,π-coordinated carbon atom, by analogy with the known complexes with σ,π-bridged alkenyl ligands.

Formation of complex **III** is quite remarkable, as dimetallacycloheptadienone derivatives are usually obtained in the reactions of ruthenium carbonyl with di-, rather than monosubstituted acetylenes [19].

It is known that complexes with σ,π-bridged unsaturated hydrocarbon ligands can exhibit stereochemical non-rigidity owing to fast $\sigma \rightarrow \pi$, $\pi \rightarrow \sigma$ exchange of these ligands between two bridged metal atoms. Such fluxionality of the bridging ligand has been observed earlier for the complexes with alkenyl, $Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}\eta^2\text{-}CH=CH_2)$ [20], acetylide, $Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}\eta^2\text{-}C\equiv CPh)$ [21], ligands, as well as for the known "flyover" complexes $M_2(CO)_6[\mu\text{-}C(R)=C(R')CO]$ [19]. However, variable temperature ^1H NMR studies of **III** did not reveal any fluxionality of the "flyover" bridge leading to the exchange of σ- and π-bonds at two ruthenium atoms; the spectral pattern observed in the temperature range from 100 to −70°C may be accounted for merely by the localized exchange of CO ligands at two ruthenium atoms. Perhaps the reason for stereochemical rigidity is the presence of bulky ferrocenyl substituents in the seven-membered heterocycle.

An X-ray diffraction study of complex **III** could probably make it possible to elucidate its structural peculiarities to account for the stereochemical rigidity of the hydrocarbon ligand. However, until now we have failed to grow single crystals of **III**, suitable for an X-ray study.

References

1 A.A. Koridze, O.A. Kizas, P.V. Petrovskii, N.E. Kolobova and G.M. Mikheeva, Dokl. Akad. Nauk. SSSR, 293 (1987) 117.

2 A.A. Koridze, O.A. Kizas, P.V. Petrovskii, M.G. Ezernitskaya and B.V. Lokshin, Metalloorg. Khim., 1 (1988) 1237.

3 A.A. Koridze, M.G. Ezernitskaya and P.V. Petrovskii, Metalloorg. Khim., 5 (1992) 235.

4 A. Mantovani and S. Cenini, Inorg. Synth., 16 (1976) 47.

5 K. Schlögl and W. Steyer, Monatsh. Chem., 96 (1965) 1520.

6 W. Robinson and G.M. Sheldrick, SHELX, in N.W. Isdaas and M.R. Taylor (Eds.), Crystallographic Computing-Techniques and New Technologies, Oxford University Press, Oxford, 1988, p. 366.

7 E. Sappa, O. Gambino, L. Milone and G. Cetini, J. Organomet. Chem., 39 (1972) 169.

8 A.I. Yanovsky, Yu.T. Struchkov, N.M. Astakhova and A.A. Koridze, Metalloorg. Khim., 3 (1990) 704.

9 G. Gervasio and G. Ferraris, Cryst. Struct. Commun., 2 (1973) 447.

10 M. Catti, G. Gervasio and S.A. Mason, J. Chem. Soc., Dalton Trans., (1977) 2260.

11 S. Aime, R. Gobetto, L. Milone, D. Osella, L. Violano, A.J. Arce and Y. De Sanctis, Organometallics, 10 (1991) 2854.

12 M.G. Peterleitner, L.I. Denisovich, O.A. Kizas, N.M. Astakhova and A.A. Koridze, unpublished results.

13 S. Aime, L. Milone and E. Sappa, J. Chem. Soc., Dalton Trans., (1976) 838.

14 W.B. Faklhammer and H. Stolzenberg, in G. Wilkinson, F.G.A. Stone and E. Abel (Eds.), Comprehensive Organometallic Chemistry, Pergamon, Oxford, 1982, Chap. 31.4.

15 W. Hübel, in I. Wender and P. Pino (Eds.), Organic Synthesis via Metal Carbonyls, Vol. 1, Wiley-Interscience, New York, 1968, p. 273.

16 M.I. Bruce, J.G. Matisons, B.W. Skelton and A.H. White, J. Organomet. Chem., 251 (1983) 249.

17 H. Astier, J.C. Daran, Y. Jeanin and C. Rigault, J. Organomet. Chem., 241 (1983) 53.

18 S. Aime, A. Tiripicchio, M. Tiripicchio Camellini and A.J. Deeming, Inorg. Chem., 20 (1981) 2027.

19 S. Aime, R. Gobetto, G. Nicola, D. Osella, L. Milone and E. Rosenberg, Organometallics, 5 (1986) 1829.

20 J.R. Shapley, S.I. Richter, M. Tachikawa and J.B. Keister, J. Organomet. Chem., 94 (1975) C43.

21 A.A. Koridze, O.A. Kizas, N.E. Kolobova and P.V. Petrovskii, Izv. Akad. Nauk SSSR., Ser. Khim., (1984) 472.