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Synthesis and structure of the heteronuclear alkyne cluster $RuOs_3(\mu_4\text{-}HC_2Me)(CO)_{12}$

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Abstract

The acetylide cluster $Os_3(\mu\text{-}H)(\mu_3\text{-}C{\equiv}CMe)(CO)_9$ reacts with $Ru_3(CO)_{12}$ in refluxing hexane to give $RuOs_3(\mu_4\text{-}HC_2Me)(CO)_{12}$ (**2**), a tetranuclear alkyne cluster with a butterfly metal core. Compound **2** was characterized by IR, NMR and single-crystal X-ray diffraction studies. © 1997 Published by Elsevier Science S.A.

Keywords: Ruthenium; Carbonyl; Osmium; Cluster; Alkyne; Crystal structure

1. Introduction

Recently we reported the reactions of triruthenium and triosmium acetylide complexes with $Ru_3(CO)_{12}$ [1]. It was found that the structure of the products formed depended on the nature of the substituent on the acetylide ligand. Thus, the reactions of ferrocenylacetylide derivatives $M_3(\mu\text{-}H)(\mu_3\text{-}C{\equiv}CFc)(CO)_9$ (M = Ru, Os; Fc = ferrocenyl) afforded green tetranuclear butterfly clusters with μ_4-acetylide and μ-hydrido ligands, whereas the reaction of cluster $Os_3(\mu\text{-}H)(\mu_3\text{-}C{\equiv}CMe)(CO)_9$ (**1**) with $Ru_3(CO)_{12}$ yielded the red tetranuclear μ_4-alkyne cluster with a butterfly metal core $RuOs_3(\mu_4\text{-}HC_2Me)(CO)_{12}$ (**2**). An assumption on the structure of cluster **2** was made on the basis of spectroscopic (IR, NMR) data.

Several μ_4-alkyne derivatives of ruthenium and osmium clusters with a butterfly metal core have been characterized by single-crystal X-ray diffraction studies, viz. $Os_4(HC_2H)(CO)_{12}$ (**3**) and $Os_4(HC_2Et)(CO)_{12}$ (**4**) [2], $Ru_4(PhC_2Ph)(CO)_{12}$ (**5**) [3,4], $Ru_4(HC_2Ph)(CO)_{12}$ (**6**) [5], $Ru_4(C_8H_{10})(CO)_{11}$ (**7**) [6], $Ru_4(C_{12}H_{16})(CO)_{10}$ (**8**) [7], and $Ru_4(C_6H_6)(C_6H_3)(CO)_9$ (**9**) [8]. It is remarkable, that complexes **3**, **4**, and **6** are formed in the reactions of metal carbonyls with olefins, ethylene and styrene respectively, rather than with terminal alkynes. Among the mixed-metal tetranuclear Fe, Ru or Os alkyne clusters with a butterfly metal core, only iron–ruthenium complexes $FeRu_3(RC_2R')(CO)_{12}$ (R = R' = Ph; R = R' = Me; R = Me, R' = Ph) are known; they are formed in thermal reaction of $FeRu_3(\mu\text{-}H)_2(CO)_{13}$ with alkynes $RC{\equiv}CR'$. When terminal alkynes $HC{\equiv}CH$, $HC{\equiv}CBu^t$ or $HC{\equiv}CPh$ were allowed to react with $FeRu_3(\mu\text{-}H)_2(CO)_{13}$, no evidence for formation of $FeRu_3(HC_2R')(CO)_{12}$ clusters was obtained [9].

Thus, complex **2** is the first mixed ruthenium–osmium alkyne cluster with a butterfly metal core. In this report we present details of the synthesis and single-crystal X-ray diffraction study of **2**.

2. Experimental

2.1. General

IR spectra were obtained on a Bruker IFS-113v spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker AMX-400 instrument. Thin-layer chromatography (TLC) was carried out on glass plates (20 × 30 cm^2) coated with silica gel.

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Table 1
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2**

	x	y	z	U_{eq} [a]
Os(1)	8352(1)	2484(1)	7597(1)	32(1)
Os(2)	8748(1)	2181(1)	9381(1)	34(1)
M(3)	9824(1)	1092(1)	8254(1)	37(1)
M(4)	10531(1)	3383(1)	8676(1)	36(1)
O(1)	9057(12)	3092(6)	5844(5)	91(3)
O(2)	5767(11)	1315(5)	6794(6)	81(3)
O(3)	5731(10)	3701(5)	7971(5)	68(2)
O(4)	10100(11)	1368(5)	11038(4)	73(2)
O(5)	5348(10)	1347(6)	9177(5)	77(3)
O(6)	7313(16)	3562(6)	10329(6)	108(4)
O(7)	6948(11)	−64(5)	8275(6)	83(3)
O(8)	12121(12)	87(5)	9490(5)	76(2)
O(9)	10783(12)	367(4)	6593(5)	73(2)
O(10)	8408(14)	4783(5)	9061(7)	100(3)
O(11)	13333(14)	3804(6)	10056(6)	106(4)
O(12)	11833(12)	4313(6)	7222(5)	87(3)
C(1)	8799(14)	2865(8)	6493(6)	61(3)
C(2)	6763(12)	1743(6)	7100(6)	49(2)
C(3)	6695(12)	3249(5)	7825(5)	44(2)
C(4)	9621(13)	1697(6)	10432(6)	46(2)
C(5)	6595(14)	1655(7)	9259(6)	56(3)
C(6)	7907(16)	3053(7)	9955(6)	62(3)
C(7)	8036(14)	366(6)	8268(7)	54(2)
C(8)	11275(14)	425(5)	8991(7)	52(2)
C(9)	10424(14)	617(5)	7197(6)	50(2)
C(10)	9244(16)	4256(6)	8943(7)	63(3)
C(11)	12306(16)	3662(6)	9530(7)	63(3)
C(12)	11350(14)	3994(6)	7810(7)	57(3)
C(13)	10928(11)	2256(5)	7945(5)	37(2)
C(14)	11141(10)	2129(5)	8891(5)	36(2)
C(15)	12413(15)	2242(8)	7428(8)	56(3)
H(14)	12273(99)	2001(43)	9219(46)	23(19)
H(15A)	13311(139)	1771(62)	7446(61)	61(32)
H(15B)	12097(133)	2400(57)	6960(66)	37(29)
H(15C)	13076(168)	2766(74)	7373(82)	88(41)

[a] U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

2.2. Starting compounds

Complex $Os_3(\mu\text{-H})(\mu_3\text{-C}{\equiv}\text{CMe})(CO)_9$ (**1**) was obtained by desilylation–decarbonylation of alkyne complex $Os_3(\mu_3\text{-Me}_3SiC_2Me)(\mu\text{-CO})(CO)_9$ [10]. ^{13}CO-enriched dodecacarbonyltriruthenium was prepared by stirring a benzene solution of $Ru_3(CO)_{12}$ with an excess of ^{13}CO (85% enrichment) at 40°C for 10 days.

2.3. $RuOs_3(\mu_4\text{-HC}_2Me)(CO)_{12}$ (2)

A hexane solution (40 ml) of $Os_3(\mu\text{-H})(\mu_3\text{-C}{\equiv}\text{CMe})(CO)_9$ (50 mg, 58 mmol) and $Ru_3(CO)_{12}$ (50 mg, 78 mmol) was refluxed under argon atmosphere for 10 h. Solvent was removed under reduced pressure, and the residue was chromatographed. The preparative TLC (light petroleum–toluene 10:1 v/v) yielded four bands. The first yellow band contained a mixture of $Ru_3(CO)_{12}$ and $Ru_4(\mu\text{-H})_4(CO)_{12}$, the second gave almost colourless unreacted **1**, and the third yielded red $RuOs_3(\mu_4\text{-HC}_2Me)(CO)_{12}$ (**2**), (11 mg, 11%); the fourth band contained uncharacterizable decomposition products.

$RuOs_3(\mu_4\text{-HC}_2Me)(CO)_{12}$ (**2**). ^{1}H NMR ($CDCl_3$, 25°C): δ 10.68 (s, 1H), 3.25 (s, 3H), ^{13}C NMR (CO region, $CDCl_3$, 25°C): δ 187.73 (s, 3CO), 180.60 (d, $J_{CH} = 4$ Hz, 3CO), 180.31 (s, 3CO), 175.5 (s, 3CO). IR (hexane): ν_{CO} 2099w, 2072vw, 2045s, 2039vs, 2019m, 2012m (sh), 2001m, 1980w, 1972w cm^{-1}.

2.4. X-ray diffraction study of 2

An X-ray diffraction study of single crystal of **2** was carried out with a four-circle diffractometer (293 K, CAD4 Enraf Nonius, Mo Kα radiation, graphite monochromator, θ–$5/3\theta$ scan technique $\theta \leq 32°$). Crystals of **2** are monoclinic, at 293 K $a = 8.049(2)$, $b = 16.847(3)$, $c = 15.687(3)$ Å, $\beta = 95.98(3)°$, $V = 2115.6(7)$ Å^3, $d_{calc} = 3.290$ g cm^{-3}, $Z = 4$, space group $P2_1/n$.

The structure was solved by a direct method and refined in the anisotropic approximation. The absorption correction (μ(Mo Kα) = 187.19 cm^{-1}) was applied using the ψ-scan technique ($T_{max} = 0.9886$, $T_{min} = 0.4944$). The Ru atom is disordered over positions M(3) and M(4) in the metal core. The refinement of the various models yielded best results for equal distribution of Ru over these two sites, i.e. both M(3) and M(4) positions are occupied by the atoms made up of Ru and Os with site occupancy factors equal to 0.5 for each. H atoms were located in the difference Fourier synthesis and included in the refinement in the isotropic approximation. The refinement converged to $R1 = 0.0353$ (on F for 4118 observed reflections with $I > 2\sigma(I)$) and $wR2 = 0.1065$ (on F^2 for all 6555 reflections used in

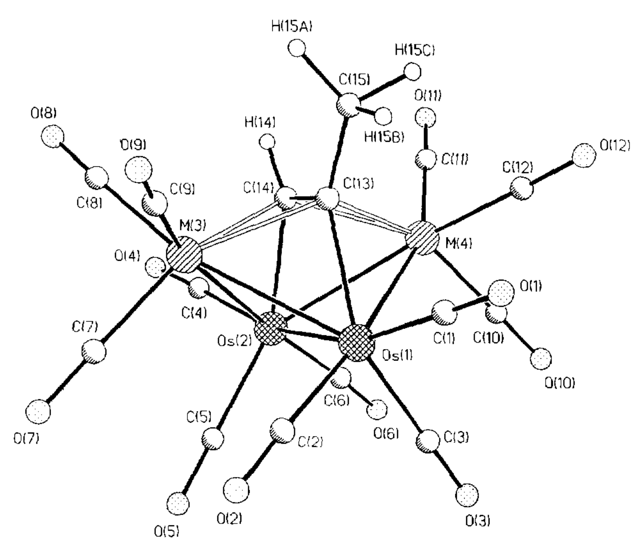


Fig. 1. Molecular structure of **2**. Each of the M(3) and M(4) positions is half-occupied by an Ru and half-occupied by an Os atom.

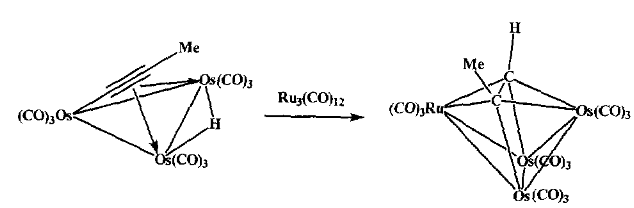
Table 2
Bond lengths (Å) and angles (deg) for **2**

Os(1)-C(2)	1.896(10)	Os(1)-C(1)	1.916(9)
Os(1)-C(3)	1.916(9)	Os(1)-C(13)	2.123(8)
Os(1)-M(4)	2.7597(8)	Os(1)-M(3)	2.7768(7)
Os(1)-Os(2)	2.8293(7)	Os(2)-C(6)	1.886(11)
Os(2)-C(4)	1.907(9)	Os(2)-C(5)	1.937(12)
Os(2)-C(14)	2.148(8)	Os(2)-M(3)	2.7493(7)
Os(2)-M(4)	2.7770(7)	M(3)-C(7)	1.890(11)
M(3)-C(8)	1.919(10)	M(3)-C(9)	1.946(10)
M(3)-C(14)	2.227(8)	M(3)-C(13)	2.227(8)
M(4)-C(10)	1.871(11)	M(4)-C(12)	1.878(12)
M(4)-C(11)	1.912(10)	M(4)-C(14)	2.186(9)
M(4)-C(13)	2.258(8)	O(1)-C(1)	1.127(11)
O(2)-C(2)	1.145(12)	O(3)-C(3)	1.127(11)
O(4)-C(4)	1.133(10)	O(5)-C(5)	1.126(13)
O(6)-C(6)	1.168(13)	O(7)-C(7)	1.138(12)
O(8)-C(8)	1.136(12)	O(9)-C(9)	1.103(11)
O(10)-C(10)	1.141(13)	O(11)-C(11)	1.132(12)
O(12)-C(12)	1.170(13)	C(13)-C(14)	1.492(11)
C(13)-C(15)	1.513(13)		
C(2)-Os(1)-C(1)	91.8(5)	C(2)-Os(1)-C(3)	93.8(4)
C(1)-Os(1)-C(3)	98.2(5)	C(2)-Os(1)-C(13)	125.8(4)
C(1)-Os(1)-C(13)	91.3(4)	C(3)-Os(1)-C(13)	139.0(4)
C(2)-Os(1)-M(4)	166.4(3)	C(1)-Os(1)-M(4)	101.7(3)
C(3)-Os(1)-M(4)	85.8(3)	C(13)-Os(1)-M(4)	53.2(2)
C(2)-Os(1)-M(3)	80.8(3)	C(1)-Os(1)-M(3)	120.4(4)
C(3)-Os(1)-M(3)	141.1(3)	C(13)-Os(1)-M(3)	52.0(2)
M(4)-Os(1)-M(3)	90.95(3)	C(2)-Os(1)-Os(2)	106.8(3)
C(1)-Os(1)-Os(2)	160.4(3)	C(3)-Os(1)-Os(2)	86.9(2)
C(13)-Os(1)-Os(2)	73.0(2)	M(4)-Os(1)-Os(2)	59.57(2)
M(3)-Os(1)-Os(2)	58.73(2)	C(6)-Os(2)-C(4)	92.2(4)
C(6)-Os(2)-C(5)	92.3(5)	C(4)-Os(2)-C(5)	97.8(4)
C(6)-Os(2)-C(14)	125.6(5)	C(4)-Os(2)-C(14)	91.6(4)
C(5)-Os(2)-C(14)	140.6(4)	C(6)-Os(2)-M(3)	168.5(3)
C(4)-Os(2)-M(3)	99.1(3)	C(5)-Os(2)-M(3)	88.3(3)
C(14)-Os(2)-M(3)	52.4(2)	C(6)-Os(2)-M(4)	81.3(3)
C(4)-Os(2)-M(4)	119.8(3)	C(5)-Os(2)-M(4)	142.0(3)
C(14)-Os(2)-M(4)	50.8(2)	M(3)-Os(2)-M(4)	91.16(3)
C(6)-Os(2)-Os(1)	108.8(3)	C(4)-Os(2)-Os(1)	157.8(3)
C(5)-Os(2)-Os(1)	88.7(3)	C(14)-Os(2)-Os(1)	70.5(2)
M(3)-Os(2)-Os(1)	59.68(2)	M(4)-Os(2)-Os(1)	58.97(2)
C(7)-M(3)-C(8)	91.8(4)	C(7)-M(3)-C(9)	90.1(5)
C(8)-M(3)-C(9)	94.8(4)	C(7)-M(3)-C(14)	146.1(4)
C(8)-M(3)-C(14)	87.6(4)	C(9)-M(3)-C(14)	123.8(4)
C(7)-M(3)-C(13)	153.3(4)	C(8)-M(3)-C(13)	114.5(4)
C(9)-M(3)-C(13)	92.1(4)	C(14)-M(3)-C(13)	39.2(3)
C(7)-M(3)-Os(2)	97.6(3)	C(8)-M(3)-Os(2)	102.5(3)
C(9)-M(3)-Os(2)	160.9(3)	C(14)-M(3)-Os(2)	49.8(2)
C(13)-M(3)-Os(2)	73.3(2)	C(7)-M(3)-Os(1)	104.7(3)
C(8)-M(3)-Os(1)	158.0(3)	C(9)-M(3)-Os(1)	99.5(3)
C(14)-M(3)-Os(1)	70.6(2)	C(13)-M(3)-Os(1)	48.7(2)
Os(2)-M(3)-Os(1)	61.59(2)	C(10)-M(4)-C(12)	88.6(5)
C(10)-M(4)-C(11)	92.3(5)	C(12)-M(4)-C(11)	94.5(5)
C(10)-M(4)-C(14)	147.7(4)	C(12)-M(4)-C(14)	123.4(4)
C(11)-M(4)-C(14)	89.4(4)	C(10)-M(4)-C(13)	152.3(4)
C(12)-M(4)-C(13)	90.8(4)	C(11)-M(4)-C(13)	115.3(4)
C(14)-M(4)-C(13)	39.2(3)	C(10)-M(4)-Os(1)	103.7(4)
C(12)-M(4)-Os(1)	96.1(3)	C(11)-M(4)-Os(1)	160.9(3)
C(14)-M(4)-Os(1)	71.5(2)	C(13)-M(4)-Os(1)	48.8(2)
C(10)-M(4)-Os(2)	99.4(4)	C(12)-M(4)-Os(2)	157.3(3)
C(11)-M(4)-Os(2)	106.3(4)	C(14)-M(4)-Os(2)	49.5(2)
C(13)-M(4)-Os(2)	72.3(2)	Os(1)-M(4)-Os(2)	61.46(2)
O(1)-C(1)-Os(1)	179.7(10)	O(2)-C(2)-Os(1)	177.8(9)
O(3)-C(3)-Os(1)	178.9(7)	O(4)-C(4)-Os(2)	175.9(9)
O(5)-C(5)-Os(2)	179.0(9)	O(6)-C(6)-Os(2)	175.8(12)
O(7)-C(7)-M(3)	179.3(10)	O(8)-C(8)-M(3)	173.0(9)
O(9)-C(9)-M(3)	178.1(9)	O(10)-C(10)-M(4)	176.1(12)
O(11)-C(11)-M(4)	177.2(12)	O(12)-C(12)-M(4)	173.7(10)
C(14)-C(13)-C(15)	121.1(8)	C(14)-C(13)-Os(1)	106.8(5)
C(15)-C(13)-Os(1)	132.1(7)	C(14)-C(13)-M(3)	70.4(5)
C(15)-C(13)-M(3)	117.3(7)	Os(1)-C(13)-M(3)	79.3(3)
C(14)-C(13)-M(4)	67.8(5)	C(15)-C(13)-M(4)	116.5(7)
Os(1)-C(13)-M(4)	78.0(3)	M(3)-C(13)-M(4)	123.3(4)
C(13)-C(14)-Os(2)	109.6(5)	C(13)-C(14)-M(4)	73.0(5)
Os(2)-C(14)-M(4)	79.7(3)	C(13)-C(14)-M(3)	70.4(5)
Os(2)-C(14)-M(3)	77.9(3)	M(4)-C(14)-M(3)	126.9(4)

the refinement). All calculations were carried out using the SHELXTL PLUS 5 (gamma version) programs on an IBM PC computer. The atomic coordinates for the structure of **2** are given in Table 1.

3. Results and discussion

The reaction of $Os_3(\mu\text{-H})(\mu_3\text{-C}{\equiv}CMe)(CO)_9$ (**1**) with $Ru_3(CO)_{12}$ in hexane at 68 °C gives the compound $RuOs_3(\mu_4\text{-HC}_2Me)(CO)_{12}$ (**2**) in 11% yield. ^1H NMR spectrum of **2** (in C_6D_6) consists of two singlet resonances at δ 9.88 and 2.78 with relative intensities of 1:3, and the IR spectrum reveals the close similarity with that of the structurally characterized tetranuclear butterfly clusters $Os_4(\mu_4\text{-HC}_2H)(CO)_{12}$ (**3**) and $Os_4(\mu_4\text{-HC}_2Et)(CO)_{12}$ (**4**) [2]. Therefore, on the basis of the spectroscopic data, we suggested for **2** the butterfly structure with the ruthenium atom at the wing-tip position.



1 2

In some experiments we observed formation of the inseparable mixture of **2** and another complex with ^1H NMR (C_6D_6) parameters δ 9.71 (s, 1H) and 2.72 (s, 3H). At present we do not know the structure of this complex, but we observed that in solution it undergoes transformation into cluster **2**.

The structure of **2** was confirmed by a single-crystal X-ray diffraction study (Fig. 1). Bond distances and angles are listed in Table 2. The molecule involves the

$RuOs_3$ butterfly-shaped metal core. The same butterfly core has previously been reported for tetranuclear alkyne complexes **3**–**9** [2–9]. The ruthenium atom in molecule **2** occupies a wing-tip position and each metal atom has three terminal carbonyl ligands. Owing to the degeneracy of positions M(3) and M(4), the Ru atom is equally distributed between these two wing-tip sites of the cluster (see Section 2); the alkyne ligand is μ_4-η^1-η^1-η^2-η^2-coordinated by the metal core.

The interatomic distance Os(1)–Os(2) 2.8293(3) Å in molecule **2** is somewhat shorter than those observed for the hinge bonds in **3** and **4** (2.847(2) Å and 2.849(2) Å respectively [2]). The metal–metal bonds formed by the atoms in the hinge and wing-tip positions for **2** are in the range 2.7493–2.7770 Å, whereas in clusters **3** and **4** the analogous bonds are in the range 2.791–2.799 Å and 2.740–2.764 Å respectively.

The dihedral angle between the butterfly wings in **2** is equal to 112.3°, which is comparable with the corresponding dihedral angle (115.5°) in the butterfly metal core of the $Ru_4(\mu_4\text{-}PhC_2Ph)(CO)_{12}$ (**5**) cluster [4]. The four-membered Os(1)Os(2)C(1 4)C(13) cycle is planar within 0.02 Å; its mean plane coincides with the bisecting plane of the dihedral angle formed by the wings of the butterfly (the dihedral angles Os(1)Os(2)C(13)C(14)/Os(1)Os(2)M(3) and Os(1)Os(2)C(13)C(14)/Os(1)Os(2)M(4) are equal to 56.3 and 56.0°).

The C(13)–C(14) and Os(1)–Os(2) bond vectors are almost parallel to each other; the angle formed by these vectors is equal to 2.2°. The C(13)–C(14) bond length in **2** is 1.492(11) Å, whereas the corresponding bond lengths in **3** and **4** are equal to 1.55(4) Å and 1.54(3) Å respectively.

The ^{13}C NMR spectrum of the ^{13}CO-enriched sample of **2** in CD_2Cl_2–$CDCl_3$ solution exhibits four sharp resonances of equal intensity at δ 188.30, 181.43, 181.15 and 176.43 ppm, indicating fast localized scrambling of CO ligands at individual metal atoms. In the undecoupled spectrum the resonance at δ 184.43 is H-coupled with $J_{CH} = 4$ Hz, which allowed us to assign this resonance to the carbonyl groups at the osmium atom in the hinge position σ-bonded to the alkyne carbon with a terminal hydrogen; the neighbouring signal at 181.15 ppm should be assigned to another osmium atom in the hinge position. Two other resonances, at lowest and highest field, evidently correspond to the carbonyl groups in the wing-tip positions at the Ru and Os atoms respectively.

As the temperature is decreased to approximately −50 °C, the resonance at 176.43 ppm becomes gradually broader, but even at −90 °C, the lowest temperature reached, it remains broadened, and the scrambling process is still not frozen. At this temperature the resonance of the $Ru(CO)_3$ group also becomes slightly broadened. Similar observations were reported earlier for clusters **5** [4] and **6** [5], wherein the exchange of carbonyl groups at the wing-tip metal atoms is characterized by a higher barrier compared with that at the metal atoms in the hinge positions.

4. Supplementary material

Tables of anisotropic thermal parameters, complete bond lengths and angles, and observed and calculated structural factors can be obtained from the authors upon request.

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