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Intramolecular rearrangement of the bridging σ,π-acetylide ligand in the $Os_3H(CO)_9(L)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (L = CO, PMe_2Ph) clusters and crystal structure of $Os_3H(CO)_9(PMe_2Ph)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$

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Abstract

Clusters $Os_3H(Cl)(CO)_9(L)$ (L = CO, PMe_2Ph) react with lithium phenylacetylide to yield $Os_3H(CO)_9(L)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$, which has a bridging acetylide ligand. The $Os_3H(CO)_{10}(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ complex (II) is fluxional owing to rapid $\pi \rightarrow \sigma$, $\sigma \rightarrow \pi$ interchange of acetylide ligand between the bridged osmium atoms, whereas the phosphine-substituted derivative, $Os_3H(CO)_9(PMe_2Ph)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (III), is stereochemically rigid and exists at room temperature in two isomeric forms. These isomers have been isolated as solids and have been characterized by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy. According to the spectroscopic data, in the major (IIIa) and minor (IIIb) isomers the phosphine ligand is coordinated to the metal atom which is σ- or π-bonded to the bridging acetylide group, respectively. The isomerization of IIIb into IIIa occurs only at 80 °C. The structure of IIIa has been confirmed by an X-ray diffraction study.

Introduction

Whereas the stereochemical non-rigidity of transition metal clusters involving carbonyl scrambling is well known, the fluxionality of hydride and especially of the organic ligands has been given considerably less attention [1,2].

The first example of alkenyl fluxionality was observed by Shapley and coworkers [3] in 1975: variable-temperature ^{13}C NMR spectra of $Os_3H(CO)_{10}(\mu\text{-}\eta^2\text{-}CH{=}CH_2)$ (I) revealed that the bridging alkenyl ligand interchanges σ and π bonds between the bridged osmium atoms thus averaging their stereochemical environment. The same fluxionality of bridging alkenyl ligands has been reported for the complexes

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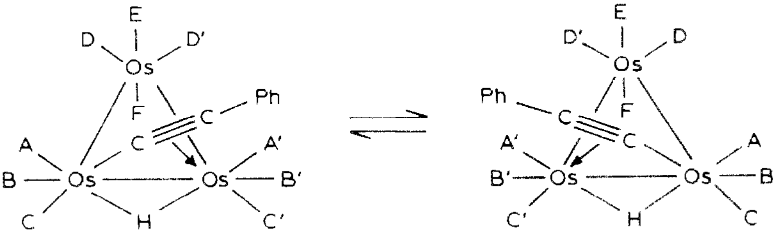
$Os_3H(CO)_{10}(\mu\text{-}\eta^2\text{-}CPh{=}CHPh)$ [4], $[M(C_5H_5)(CO)_2](\mu\text{-}\eta^2\text{-}CH{=}CH_2)^+$ (M = Fe [5], Ru [6]) and $Re_2H(CO)_8(\mu\text{-}\eta^2\text{-}CH{=}CH_2)$ [7].

In contrast to the μ-alkenyl ligand, the fluxionality of the μ-acetylide moiety remained elusive [8]. Only recently Brown's group [9] and ours [10] reported simultaneously σ,π-acetylide fluxionality in $Re_2H(CO)_8(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ and $Os_3H(CO)_{10}(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (II), respectively. In this paper we report the synthesis of phosphine-substituted derivatives of II and the effect of phosphine substitution on the mobility of the acetylide ligand. We also present the results of an X-ray structure analysis of complex IIIa, one of the isomers of $Os_3H(CO)_9(PMe_2Ph)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$. A preliminary account of this work has appeared [10].

Results and discussion

In accord with the proposed structure of cluster II suggested by Deeming [11], the bridged osmium atoms are nonequivalent, and in the ^1H NMR spectrum two sets of $^1J(^{187}Os\text{-}^1H)$ satellites for the hydride resonance should be expected. However, only one set of the satellites, with an $^{187}Os\text{-}^1H$ coupling constant of 33.5 Hz, is observed for the signal at δ −16.39 ppm ($CDCl_3$). This may be due to rapid $\pi \rightarrow \sigma$, $\sigma \rightarrow \pi$ exchange of the bridging acetylide ligand.



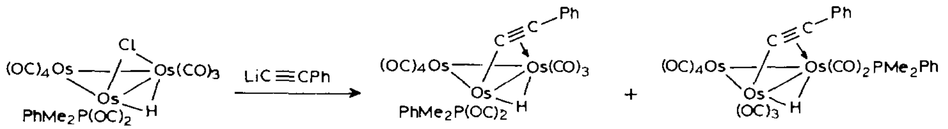
Indeed, the $^{13}C\{^1H\}$ NMR spectrum of II at room temperature displays six resonances due to the carbonyl ligands at δ 182.7, 182.3, 178.1, 175.0, 171.3 and 170.2 ppm of relative intensities 1/1/2/2/2/2. This spectral pattern reveals rapid exchange of acetylide ligand between the two osmium atoms, a process which equilibrates pairwise the carbonyl environments for A/A', B/B', C/C', D/D', but not for E and F. In the coupled spectrum, resonance at 171.3 ppm is observed as a doublet with $^2J(^{13}C\text{-}^1H)$ 10.5 Hz; this signal should be assigned to the carbonyls AA', at *trans*-position relative to the hydride ligand. Two of the remaining resonances, relative intensity 2, at 170.2 and 178.1 ppm, are somewhat broadened. At least one of them can be attributed to the ligands BB', but the other can be assigned to carbonyls CC' or DD'. Resonances of relative intensity 1 are assigned to carbonyls E and F. The most noteworthy chemical shifts of these ligands, at δ 182.7 and 182.3, are in fact in the same position as the axial carbonyl resonance of $Os_3(CO)_{12}$, at 182.3 ppm; resonance of the equatorial ligands of osmium carbonyl is observed at 170.4 ppm [12].

Unfortunately, our attempt to obtain a low-temperature limiting spectrum of II was unsuccessful: the carbonyl region of the spectrum was invariant between 30 and the lowest temperature used in our study, −90 °C. (The most significant temperature-dependent change in the spectrum was the upfield shift of the acetylide carbon atom signal from δ 97.3 at 30 °C to 91.9 ppm at −90 °C. For all other resonances the value of $\Delta\delta$ does not exceed 1.3 ppm.) So, rearrangement of the acetylide ligand of II has a lower activation energy than the rearrangement of alkenyl ligand in related triosmium clusters; thus, the low-temperature limiting spectrum for I is observed at −67 °C [3].

In an attempt to increase the energy barrier for rearrangement of the acetylide ligand we synthesized the complex in which one of the carbonyl ligands of $(OC)_3Os(\mu\text{-}H)Os(CO)_3$ moiety was replaced by tertiary phosphine. In a complex such as $Os_3H(CO)_9(PMe_2Ph)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (III), acetylide ligand rearrangement is no longer degenerate; moreover, it is known that analogous derivatives of I, $\mu\text{-}\eta^2$-vinyl complexes $Os_3H(CO)_9(PR_3)(\mu\text{-}\eta^2\text{-}CH{=}CH_2)$ [13] are stereochemically rigid even when heated to 95 °C.

Earlier we observed the formation of II under reaction of $Os_3H_2(CO)_{10}$ with $(OC)_5ReC{\equiv}CPh$. A similar reaction could have led to III. However, the reaction of $(OC)_5ReC{\equiv}CPh$ with phosphine-substituted dihydride $Os_3H_2(CO)_9(PMe_2Ph)$ proceeds differently, to give a mixture of three carbonyl-phosphine complexes all without hydride ligands. In the $^{31}P\{^1H\}$ NMR spectrum of this mixture three signals of relative intensity 4.6, 1.3 and 1.0 are observed at −44.30, −43.94 and −38.16 ppm, respectively. With the aid of fractional crystallization the main component was isolated in the form of yellow crystals. Elemental analysis of this material is consistent with the formula $Os_3(CO)_9(PMe_2Ph)\{(OC)_5ReC_2Ph\}$. Efforts are underway to establish the structure of this cluster.

The required complex III was obtained as a mixture of isomers after the reaction of $Os_3H(Cl)(CO)_9(PMe_2Ph)$ [14] with lithium phenylacetylide, by the same method as that suggested for the synthesis of II [15]. The isomers were separated by TLC on silica gel.



(IIIa) (IIIb)

In the ^1H NMR spectrum of the major isomer (IIIa) the hydride resonance is observed at δ −16.17 ppm as a doublet with ^{31}P–^1H coupling constant of 7.4 Hz; the $^{31}P\{^1H\}$ NMR spectrum shows a signal at −29.2 ppm ($CDCl_3$). For the minor isomer (IIIb), $\mu\text{-}^1H$ and ^{31}P resonances in the corresponding NMR spectra are observed at −18.72 ppm (^{31}P–^1H coupling constant of 13.3 Hz) and −42.6 ppm, respectively. The values of $J(^{31}P\text{–}^1H)$ indicate that in both complexes the phosphine ligand is bonded to one of the osmium atoms of $Os(\mu\text{-}H)Os$ moiety; it is also evident that PMe_2Ph ligand occupies the sterically less-crowded equatorial position. The $^{31}P\{^1H\}$ NMR spectral data permit the structures IIIa and IIIb to be assigned to the major and minor isomers, respectively. This assignment is based on the data for ^{31}P shielding in the $^{31}P\{^1H\}$ NMR spectra of isomeric $Os_3H(Cl)(CO)_9(PMe_2Ph)$ complexes. It was confirmed by a single crystal X-ray structure analysis of the major isomer (IIIa).

The X-ray diffraction study of IIIa was desirable for the following reasons:

(1) Complex II had for the first time been synthesized by the reaction of $Os_3H_2(CO)_{10}$ with $PhC{\equiv}CH$ [11], and was the only known cluster of the composition $M_3H(CO)_{10}(C{\equiv}CR)$ among the Fe, Ru and Os derivatives, and it was of interest to determine the precise geometry of the bridging acetylide ligand in II.

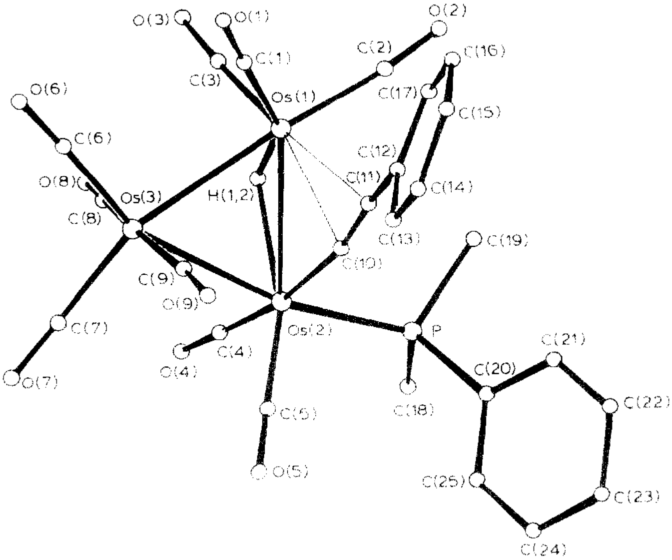


Fig. 1. Molecular structure of IIIa. Bond lengths (Å): Os(1)–Os(2) 2.869(1), Os(1)–Os(3) 2.859(1), Os(2)–Os(3) 2.864(1), Os(1)–C(10) 2.316(5), Os(1)–C(11) 2.530(5), Os(2)–C(10) 2.066(6), Os(2)–P 2.332(2), Os(1)–H(1,2) 1.84(7), Os(2)–H(1,2) 2.18(6), C(10)–C(11) 1.201(8), Os–C(CO) 1.876–1.953; bond angles (°): Os(2)C(10)C(11) 167.4(5), C(10)C(11)C(12) 164.3(6), Os(1)H(1,2)Os(2) 91(3).

However, our attempts to obtain a single crystal of II suitable for X-ray study were unsuccessful.

(2) The study of complex IIIa may give important information on the bonding geometry of acetylide ligand, and would establish unequivocally the site of coordination of PMe_2Ph ligand.

Crystals of IIIa suitable for an X-ray diffraction study were obtained by crystallization from n-hexane solution. The molecular structure of complex IIIa is shown in Fig. 1.

The molecular structure is consistent with the structure suggested on the basis of spectral data: phenylacetylide ligand forms a σ-bond with the Os(2) atom (Os(2)–C(10) 2.066(6) Å), and a π-bond with the Os(1) atom (Os(1)–C(11) 2.530(6) Å), with the phosphine ligand coordinated to Os(2) (Os(2)–P 2.332(2) Å). The directly located hydride ligand, H(1,2), in accord with NMR data, bridges the Os(1)–Os(2) edge.

The key feature of IIIa is the bridging acetylide group. In contrast to the complexes with σ,2π-acetylide ligand (e.g. $Os_3(CO)_9(PPh_2)(C{\equiv}CPr^i)$ [16] and $Ru_3H(CO)_9(C{\equiv}CBu^t)$ [17]) the π-bonding of acetylenic carbon atoms in IIIa is essentially asymmetric. The acetylene moiety itself is only very slightly distorted: the C(10)≡C(11) triple bond of 1.201(8) Å is in fact not elongated on coordination; the Os(2)C(10)C(11) 167.4(5)° and the C(10)C(11)C(12) 164.3(6)° bond angles indicate only a minor deviation of acetylide from linearity. At the same time in bi- and tetranuclear ruthenium complexes with σ,π-bound acetylides – $Ru_2(CO)_6(PPh_2)$-$(C{\equiv}CBu^t)$ and $Ru_4(CO)_{13}(PPh_2)(C{\equiv}CBu^t)$ [18] – the geometry of the coordination of acetylide group is similar to that found in IIIa (unequal M–C distances, no elongation of the triple C≡C bond, no deviation from linearity).

It is noteworthy that in contrast to the acetylide ligand, the alkenyl group exhibits much stronger distortion on σ,π-coordination. Thus, the σ,π-coordinated C=C double bond in I is elongated to 1.396 Å [19] compared with the standard C=C double bond length in olefins (1.34 Å [20]). The more pronounced changes which occur with σ,π-alkenyl coordination, compared with σ,π-acetylide ligands as well as the shorter π-bonds with the former ligand (Os–C 2.273 and 2.362 Å in I, 2.316(5) and 2.530(5) Å in IIIa), evidently may be related to the higher bonding energy of the σ,π-alkenyl groups and their smaller mobility in fluxional processes.

In contrast to cluster I and other triosmium clusters with bridging alkenyl groups [4,19], the longest Os–Os edge in IIIa is not between the $Os(CO)_4$ center and the Os atom π-coordinated to the bridging organic ligand, but along the edge linking the double bridged osmium atoms. It should be noted, however, that differences in Os–Os bond lengths in cluster IIIa are less than about 0.01 Å (Os(1)–Os(2) 2.869(1), Os(2)–Os(3) 2.864(1) and Os(1)–Os(3) 2.859(1) Å), and thus can hardly be considered chemically significant.

Complexes IIIa and IIIb are stereochemically rigid at room temperature, however, when IIIb is heated in toluene at 80 °C for 1.5 h, it is completely converted into the isomer IIIa, probably via $\pi \to \sigma$, $\sigma \to \pi$ rearrangement of acetylide ligand. Thus, comparison of the ability of alkenyl [3,4] and acetylide ligands to undergo intramolecular exchange shows that acetylide ligand is much more loosely bound into the cluster framework, and that its complexes are essentially non-rigid.

Results of the present study also show that the transfer of acetylide ligand from the σ-complex to the triosmium cluster by reaction with unsaturated dihydrides is by no means common to clusters of this type, since $Os_3H_2(CO)_{10}$ and $Os_3H_2(CO)_9(PMe_2Ph)$ react with $(OC)_5ReC{\equiv}CPh$ in a different way. As for the reaction of lithium phenylacetylide with $Os_3H(Cl)(CO)_9(L)$ (L = CO, PMe_2Ph), this method is a convenient route to triosmium clusters with σ,π-acetylide ligand.

Experimental

Complex $Os_3H(CO)_{10}(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (II) was prepared by reaction of $Os_3H(Cl)(CO)_{10}$ with $LiC{\equiv}CPh$ as reported recently [15]. Solvents used were dried by standard procedures, and reactions were carried out under purified argon.

NMR spectra were obtained on a Bruker WP-200SY spectrometer. The solvent was $CDCl_3$. ^1H NMR spectra were internally referenced to tetramethylsilane. $^{31}P\{^1H\}$ NMR spectra were externally referenced to 85% H_3PO_4 with upfield chemical shifts reported as negative.

Preparation of $Os_3H(CO)_9(PMe_2Ph)(\mu\text{-}\eta^2\text{-}C{\equiv}CPh)$ (IIIa and IIIb)

A solution of lithium phenylacetylide was prepared from $PhC{\equiv}CH$ (0.23 ml, 0.2 mmol) and n-butyllithium (1.3 *N* hexane solution, 0.16 ml, 0.2 mmol) in 15 ml of freshly distilled THF at −10 °C. This solution was added dropwise to a solution of $Os_3H(Cl)(CO)_9(PMe_2Ph)$ (isomer with PMe_2Ph ligand at H- and Cl-bridged osmium atom) (90 mg, 0.1 mmol) in 40 ml of hexane with stirring, and the mixture refluxed for ~ 1 h. The reaction mixture was then cooled, water was added, and extracted with benzene. The organic phase was dried over anhydrous Na_2SO_4. The solution was concentrated and then subjected to TLC, eluent: hexane/benzene 1/1. The first yellow band yielded isomer IIIa (60 mg, 63%); ^1H NMR: δ (ppm): −16.17 (d,

$J(^1H-^{31}P)$ 7.4 Hz, μ-H), 2.03 (d, $J(^1H-^{31}P)$ 9.8 Hz, PCH_3), 2.08 (d, $J(^1H-^{31}P)$ 9.8 Hz, PCH_3) and 6.86–7.89 (m, $2C_6H_5$); $^{31}P\{^1H\}$ NMR: δ (ppm): −29.2 (s). The second yellow band yielded isomer IIIb (10 mg 11%); ^1H NMR: δ (ppm): −18.72 (d, $J(^1H-^{31}P)$ 13.3 Hz, μ-H), 2.12 (d, $J(^1H-^{31}P)$ 9.8 Hz, PCH_3), 2.15 (d, $J(^1H-^{31}P)$ 9.8 Hz, PCH_3), and 6.80–7.85 (m, $2C_6H_5$); $^{31}P\{^1H\}$ NMR: δ (ppm): −42.6 (s).

Conversion of IIIb into IIIa

A solution of isomer IIIb (10 mg) in toluene-d_8 was heated at 80 °C in a sealed NMR ampoule. The ^1H NMR spectrum of solution after 1.5 h showed complete conversion of IIIb into IIIa.

Table 1

Atomic coordinates and their equivalent isotropic (for hydride H atom – isotropic) temperature factors for IIIa

Atom	x	y	z	B (Å^2)
Os(1)	0.21362(2)	0.34251(2)	0.10236(2)	2.663(5)
Os(2)	0.08828(2)	0.39935(2)	0.27841(2)	2.607(5)
Os(3)	0.35058(3)	0.55430(2)	0.25651(2)	2.903(5)
P	−0.1241(2)	0.2519(2)	0.2606(1)	3.06(3)
O(1)	0.4956(5)	0.3272(6)	0.0590(4)	5.7(1)
O(2)	0.0456(6)	0.1219(5)	−0.0596(4)	5.8(1)
O(3)	0.1919(6)	0.5190(5)	−0.0593(4)	6.1(1)
O(4)	−0.0349(6)	0.6285(5)	0.2815(5)	6.4(1)
O(5)	0.1466(7)	0.4254(7)	0.5121(4)	7.4(2)
O(6)	0.6191(5)	0.6429(6)	0.1728(4)	5.9(1)
O(7)	0.4036(7)	0.7558(5)	0.4460(4)	6.4(2)
O(8)	0.1786(6)	0.7046(5)	0.1308(4)	5.6(1)
O(9)	0.4661(6)	0.3800(5)	0.3950(4)	5.7(1)
C(1)	0.3915(7)	0.3341(6)	0.0761(5)	3.4(1)
C(2)	0.1036(7)	0.1989(7)	0.0030(5)	4.1(2)
C(3)	0.2024(7)	0.4553(7)	0.0035(5)	3.9(2)
C(4)	0.0118(7)	0.5418(7)	0.2812(5)	3.9(2)
C(5)	0.1229(7)	0.4162(7)	0.4240(5)	4.3(2)
C(6)	0.5185(7)	0.6081(7)	0.2020(5)	4.1(2)
C(7)	0.3847(7)	0.6791(6)	0.3770(5)	4.0(2)
C(8)	0.2425(7)	0.6489(7)	0.1764(6)	4.2(2)
C(9)	0.4214(7)	0.4388(6)	0.3408(5)	3.9(2)
C(10)	0.1812(6)	0.2527(6)	0.2529(4)	3.1(1)
C(11)	0.2421(7)	0.1788(6)	0.2219(5)	3.4(1)
C(12)	0.3287(7)	0.0915(6)	0.2147(5)	3.3(1)
C(13)	0.4033(8)	0.0722(7)	0.3059(6)	4.3(2)
C(14)	0.4912(8)	−0.0081(7)	0.3016(7)	5.2(2)
C(15)	0.5030(8)	−0.0682(7)	0.2067(7)	5.4(2)
C(16)	0.4289(8)	−0.0514(7)	0.1192(7)	5.4(2)
C(17)	0.3415(8)	0.0281(7)	0.1223(6)	4.7(2)
C(18)	−0.2664(8)	0.3189(8)	0.2780(6)	5.1(2)
C(19)	−0.1808(9)	0.1613(8)	0.1322(6)	5.3(2)
C(20)	−0.1374(6)	0.1283(6)	0.3469(5)	3.5(1)
C(21)	−0.1365(8)	0.0054(7)	0.3142(6)	4.7(2)
C(22)	−0.1542(9)	−0.0864(7)	0.3781(8)	6.3(2)
C(23)	−0.1775(9)	−0.0568(8)	0.4789(7)	6.2(2)
C(24)	−0.1744(9)	0.0668(9)	0.5134(6)	5.9(2)
C(25)	−0.1578(8)	0.1569(8)	0.4474(6)	4.8(2)
H(1,2)	0.088(7)	0.436(7)	0.118(5)	6(2)

X-Ray diffraction study of IIIa

Crystals of IIIa are triclinic, at 20 °C: a 10.155(2), b 10.921(2), c 13.111(3) Å, α 95.53(2), β 97.80(2), γ 103.75(2)°, V 1386.9 Å^3, d 2.541 g/cm^3, $Z = 2$, space group $P\bar{1}$. The unit cell parameters and intensities of 4223 reflections with $F^2 \geqslant 5\sigma$ were measured with an automatic four-circle CAD-4 diffractometer (+20 °C, λ Mo-K_α, graphite monochromator, $\omega{:}\theta$ scan ratio 1.2/1, $\theta \leqslant 26°$, absorption correction applied according to DIFABS procedure [21], μ(Mo-K_α) 138.3 cm^{-1}).

The structure was solved by the standard heavy-atom method (coordinates of the three Os atoms were found from the Patterson synthesis) and refined anisotropically. All H atoms including the μ_2-bridging hydride atom were located in the difference Fourier synthesis; their contributions to F_{calc} were taken into account in the subsequent refinement ($B_{iso} = 5$ Å^2), although only positional and isotropic thermal parameters of the hydride atom were actually refined. Final discrepancy factors are $R = 0.024$, $R_w = 0.034$. All calculations were carried out with a PDP-11/23PLUS computer using the SDP-PLUS [22] program package. Atomic coordinates are listed in Table 1.

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