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Reactions of $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ (R = Me or Ph) with carbon monoxide to give either $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ or $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$. Crystal structure of $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$

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Abstract

The complexes $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ (R = Me and Ph) react in CH_2Cl_2 with an excess of carbon monoxide to give initially the acetonitrile substituted products $[WI_2(CO)_2(\eta^2\text{-}RC_2R)_2]$. For R = Me, the complex $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ (**1**) was isolated and its structure determined by X-ray crystallography. However, for R = Ph, dimerisation occurs to give the iodide-bridged compound $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ (**2**) with loss of carbon monoxide. These reactions are reversible as **1** and **2** react with acetonitrile to give $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$. The ^{13}C NMR spectra of **1** and **2** indicate that the two alkyne ligands donate a total of six electrons to the tungsten in these complexes.

Introduction

There is considerable interest in alkyne complexes of molybdenum(II) and tungsten(II), mainly because they contain alkyne ligands which can donate 2 and 4 electrons to the metal by utilising their two filled perpendicular p_π orbitals. A large number of six-coordinate molybdenum(II) and tungsten(II) monoalkyne complexes have been reported [1–10], but far fewer bisalkyne compounds have been prepared. This type of reaction often results in the formation of π-bonded cyclobutadienes [11], metallocyclopentadienes [12], cyclotrimerised arenes [13], or polyalkynes [14].

In the past two years we have been studying the reactions of the bisalkyne complexes $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ (R = Me and Ph) [15], which are formed in quantitative yield by reaction of $[WI_2(CO)_3(NCMe)_2]$ [16] with two equivalents of RC_2R in CH_2Cl_2. We describe here the reactions of these complexes with carbon monoxide.

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Results and discussion

Carbon monoxide was bubbled through a solution of $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ (R = Me and Ph) to give $[WI_2(CO)_2(\eta^2\text{-}RC_2R)_2]$, which for R = Me (**1**) was fully characterised by elemental analysis and IR, ^1H and ^{13}C NMR spectroscopy and X-ray crystallography. For R = Ph the dicarbonyl compound $[WI_2(CO)_2(\eta^2\text{-}PhC_2Ph)_2]$ could not be isolated since it dimerised to give the iodide-bridged dimer $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ (**2**) with loss of carbon monoxide. The dimer **2** was also fully characterised, and molecular weight studies confirmed its dimeric nature. Complexes **1** and **2** reacted with acetonitrile to give the monoacetonitrile complexes $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$, i.e. the reactions with CO are reversible (see Experimental). Both complexes **1** and **2** are soluble in $CHCl_3$ or CH_2Cl_2, but only slightly soluble in Et_2O. Although they are air-sensitive in solution they can be stored for several days as solids at 0 °C under nitrogen.

The bis(but-2-yne) complex $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ (**1**) which is formed upon addition of CO to $[WI_2(CO)(NCMe)(\eta^2\text{-}MeC_2Me)_2]$, does not appear to dimerise even when its solution in CH_2Cl_2 is stirred at room temperature for 24 h. However, the bisdiphenylacetylene complex $[WI_2(CO)_2(\eta^2\text{-}PhC_2Ph)_2]$, which is formed upon addition of CO to $[WI_2(CO)(NCMe)(\eta^2\text{-}PhC_2Ph)_2]$, could not be isolated, since it dimerised to give the iodide-bridged dimer $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$. The dicarbonyl compound $[WI_2(CO)_2(\eta^2\text{-}PhC_2Ph)_2]$ was identified by IR spectroscopy (ν(CO) 2105 cm^{-1}) as an intermediate formed before dimerisation. This was expected, since the more electron rich but-2-yne ligand increases the electron density on the metal, and so increases the electron density available for backbonding into the empty $\pi^\star$-orbitals of CO and results in a stronger W–CO bond in **1** compared to that in $[WI_2(CO)_2(\eta^2\text{-}PhC_2Ph)_2]$. It is noteworthy that Davidson and co-workers [17] reported the preparation of a supposedly 5-coordinate bis(hexafluorobut-2-yne) complex $[WBr_2(CO)(\eta^2\text{-}CF_3C_2CF_3)_2]$ which is prepared by treating $[W(\mu\text{-}Br)Br(CO)_4]_2$ with $CF_3C_2CF_3$. It is possible that $[WBr_2(CO)(\eta^2\text{-}CF_3C_2CF_3)_2]$ is in fact a *trans*-dicarbonyl compound $[WBr_2(CO)_2(\eta^2\text{-}CF_3C_2CF_3)_2]$ in view of the structure of $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ described here. They also reported that the compound $[WBr_2(CO)(\eta^2\text{-}CF_3C_2CF_3)_2]$ had a single carbonyl stretching band at 2172 cm^{-1} (higher than "free" CO), which would be consistent with a *trans*-dicarbonyl structure. However, it is surprising that $[WBr_2(CO)(\eta^2\text{-}CF_3C_2CF_3)_2]$ does not dimerise to give $[W(\mu\text{-}Br)Br(CO)(\eta^2\text{-}CF_3C_2CF_3)_2]$ in view of its electron deficient nature, and since Davidson and Vasapollo [18] reported that reaction of the more electron rich alkynes RC_2R (R = Me, Et or Ph) with $[W(\mu\text{-}Br)Br(CO)_4]_2$ give the bromide-bridged dimers $[W(\mu\text{-}Br)Br(CO)(\eta^2\text{-}RC_2R)_2]_2$, which are analogous to the iodide-bridged dimer $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ (**2**) described in this paper.

The monoacetonitrile complexes $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ do not dimerise even when their solutions in $CHCl_3$ are refluxed for 24 h [15]. Hence it is highly likely that their reaction with CO involves an associative mechanism. The two alkyne ligands in these complexes donate a total of six electrons to the metal [15]. Hence they could rearrange their mode of bonding to accommodate a carbon monoxide ligand (with both alkynes acting as 2 electron donors), to give the seven-coordinate intermediates $[WI_2(CO)_2(NCMe)(\eta^2\text{-}RC_2R)_2]$, which would rapidly lose acetonitrile to give the initially observed products. This associative mechanism for substitution in bisalkyne complexes has been previously observed

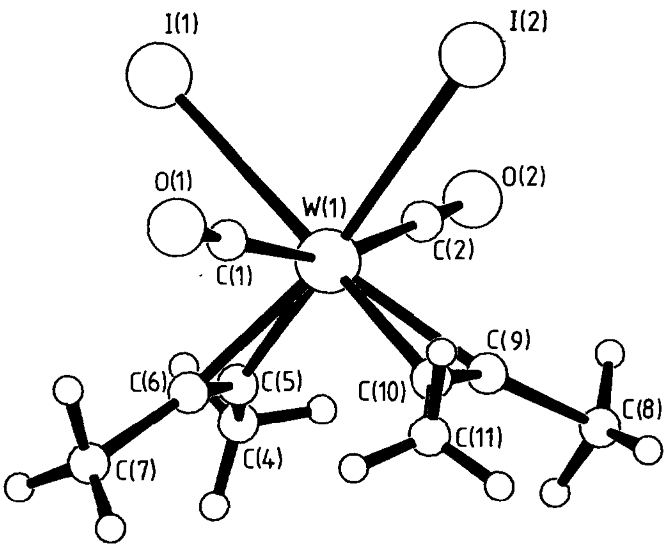


Fig. 1. X-Ray crystal structure of $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ (**1**).

[19] from the reactions of phosphines and phosphites with $[Mo(CO)(\eta^2\text{-}MeC_2Me)_2(\eta^5\text{-}C_5H_5)][BF_4]$.

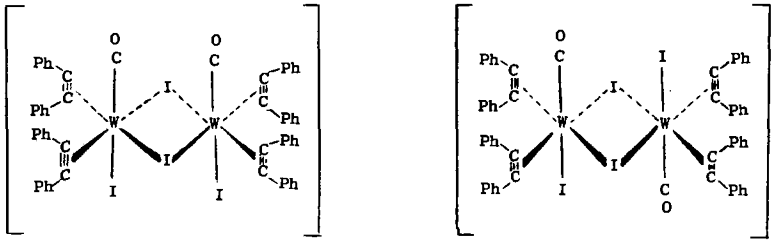
The structure of **1** is shown in Fig. 1 together with the atomic numbering scheme. The tungsten atom is bonded to two mutually *trans*-carbonyl groups (W–C(1) 2.089(18), W–C(2) 2.067(21) Å), two mutually *cis*-iodine atoms (W–I(1) 2.862(1), 2.851(1) Å and two dimethylalkynes (W–C(6) 2.062(16), W–C(5) 2.115(17), W–C(10) 2.123(15), W–C(9) 2.076(16) Å). The coordination geometry is probably best considered as octahedral, with each alkyne occupying one coordination site *trans*- to an iodide.

The geometry is comparable to that found for two compounds previously studied in this programme, namely $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ with R = Me and with R = Ph [15]. In these compounds the acetonitrile and the carbonyl groups are mutually *trans*- but the relative positions of the iodine and alkyne groups are comparable.

It is of interest that both alkyne groups are asymmetrically bonded, with one W–C bond longer than the other (viz. W–C(6) 2.062(16), W–C(5) 2.115(17) Å and W–C(9) 2.076(16), W–C(10) 2.123(15) Å). The structures of $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ also show this asymmetry, but in these species the two shorter bonds are both closer to the acetonitrile than to the carbonyl. In the present molecule, the asymmetry is different in that of the two shorter bonds, C(6) is adjacent to one carbonyl and C(9) to the other. We have no explanation for this difference.

Remaining dimensions in the structure are as expected. The two alkyne groups are both planar, and intersect each other at an angle of 84.9°. In the alkynes the central C–C bonds retain double bond character, with bond lengths of 1.22(2), 1.27(2) Å. The C–C–C angles are 145(2), 148(2), 146(2), 147(2)°. There are no intramolecular distances of less than the sum of Van der Waals radii.

In view of the structure of **1**, the dinuclear complex **2** is likely to have bridging iodide ligands, with *cis*-diphenylacetylenes which are *trans*- to the bridging ligands.



(a) (b)

Fig. 2. Proposed structures for the dimeric complex $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ (**2**).

The two possible isomers are shown in Fig. 2(a) and (b). However, the most likely structure of **2** shown in Fig. 2(a), has *cis*-carbonyl ligands, since the IR spectrum of **2** shows two equally intense carbonyl bands indicating *cis*-carbonyl ligands with a small interaction between the carbonyl groups. This structure was also postulated by Davidson and Vasapollo [18] for the analogous bromide-bridged compounds $[W(\mu\text{-}Br)Br(CO)(\eta^2\text{-}RC_2R)_2]_2$ (R = Me, Et or Ph) and PhC_2Me.

The IR carbonyl bands for $[WI_2(CO)_2(\eta^2\text{-}RC_2R)_2]$ at 2095 cm^{-1} for R = Me and 2105 for R = Ph are among the highest known for metal carbonyl compounds (free (CO) 2143 cm^{-1}). This indicates that little electron density is being back donated into empty π^*-orbitals of the CO ligands. The alkyne ($C{\equiv}C$) stretching band for **1** at 1643 cm^{-1} is lower than that for uncoordinated but-2-yne since electron density is back donated into π^*-orbitals of the but-2-yne ligand.

The ^1H NMR spectra (see Experimental) of **1** and **2** are consistent with the static structures shown in Figs. 1 and 2(a). Templeton and Ward reported [20] that the number of electrons donated by an alkyne ligand can be correlated with the ^{13}C NMR chemical shifts of the coordinated alkyne carbon atoms. The alkyne contact ^{13}C chemical shifts for **1** and **2** indicates that the alkyne ligands donate an average of three electrons each to the tungsten atoms. It is highly likely that one alkyne donates four electrons and the other two in these complexes, with rapid interchange between these resonance hybrids.

Experimental

All reactions were carried out under nitrogen. Distilled deoxygenated dichloromethane was used. The complexes $[WI_2(CO)(NCMe)(\eta^2\text{-}RC_2R)_2]$ were prepared by published methods [15], and all other chemicals were from commercial sources. IR spectra were recorded as $CHCl_3$ films between NaCl plates on a Perkin–Elmer 197 IR spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on a JEOL FX 60 MHz NMR spectrometer and shifts are relative to tetramethylsilane. The molecular weight of $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ was determined by Rast's method [21] with camphor as solvent.

Preparation of $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ (1)

$[WI_2(CO)(NCMe)(\eta^2\text{-}MeC_2Me)_2]$ (0.500 g, 0.813 mmol) was dissolved in CH_2Cl_2 (10 cm^3). CO (generated by the action of concentrated H_2SO_4 on HCOOH and dried over $CaCl_2$) was bubbled vigorously through the solution for 90 min. Filtration followed by removal of the solvent in vacuo gave a yellow powder, which was recrystallised from CH_2Cl_2/Et_2O at −18 °C to give small yellow crystals of $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$. Yield 0.37 g, 76%. Analysis. Found: C, 19.3; H, 2.0; $C_{10}H_{12}I_2O_2W$ calc: C, 19.9; H, 2.0%. IR $\nu(C{\equiv}O)$ 2095vs, $\nu(C{\equiv}C)$ 1643w cm^{-1}. NMR ($CDCl_3$, +25 °C) ^1H; δ 3.0 (s, *Me*). ^{13}C; δ 192.84 (s, *CO*); 151.9 (s, *C≡C*); 19.1 (s, CH_3).

Preparation of $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ *(2)*

$[WI_2(CO)(NCMe)(\eta^2\text{-}PhC_2Ph)_2]$, (0.500 g, 0.579 mmol) was dissolved in CH_2Cl_2 (25 cm^3). CO (generated as before) was bubbled through the solution vigorously for 6 h. Filtration followed by removal of the solvent in vacuo gave the deep yellow $[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ as a crystalline powder. Yield 0.28 g, 58%. Analysis. Found: C, 42.4; H, 2.5; $C_{58}H_{40}I_4O_2W_2$ calc: C, 42.1; H, 2.5%. IR $\nu(C{\equiv}O)$ 2050vs, 1981vs, $\nu(C{\equiv}C)$ 1640w cm^{-1}. NMR ($CDCl_3$, +25 °C), ^1H, δ 7.45 (m, *Ph*). ^{13}C, δ 199.34 (s, *CO*); 189.46, 160.49 (s, *C≡C*), 135.28 (s, *Ph*); 130.34 (s, *o*-Ph); 129.17 (s, *m*-Ph); 128.13 (s, *p*-Ph). Molecular weight determination: Found, 1409; calcd. 1644.

Reaction of 1 with acetonitrile

$[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$ (0.500 g, 0.831 mmol) was dissolved in NCMe (15 cm^3) and the solution stirred for 60 min. Filtration, followed by removal of the solvent in vacuo gave the yellow $[WI_2(CO)(NCMe)(\eta^2\text{-}MeC_2Me)_2]$ which was recrystallized from CH_2Cl_2. Yield 0.28 g, 55%. Analysis. Found: C, 21.3; H, 2.4; N, 2.4; $C_{11}H_{15}NI_2OW$ calc: C, 21.5; H, 2.5; N, 2.3%. IR $\nu(C{\equiv}N)$ 2325 and 2298w; $\nu(C{\equiv}O)$ 2050s, $\nu(C{\equiv}C)$, 1642w cm^{-1}. Other spectral data agreed with the complex $[WI_2(CO)(NCMe)(\eta^2\text{-}MeC_2Me)_2]$ discussed elsewhere [15].

Reaction of 2 with acetonitrile

$[W(\mu\text{-}I)I(CO)(\eta^2\text{-}PhC_2Ph)_2]_2$ (0.500 g, 0.304 mmol) was dissolved in NCMe (15 cm^3) and the solution stirred for 4 h. Filtration followed by removal of solvent in vacuo gave the yellow $[WI_2(CO)(NCMe)(\eta^2\text{-}PhC_2Ph)_2]$, which was recrystallised from CH_2Cl_2. Yield 0.40 g, 76%. Analysis. Found: C, 43.5; H, 2.5; N, 1.8. $C_{31}H_{23}NI_2OW$ calc: C, 43.1; H, 2.7; N, 1.6%. IR $\nu(C{\equiv}N)$ 2320 and 2300w, $\nu(C{\equiv}O)$ 2090s, $\nu(C{\equiv}C)$ 1618vw cm^{-1}. Other spectral data agreed with the complex $[WI_2(CO)(NCMe)(\eta^2\text{-}PhC_2Ph)_2]$ discussed elsewhere [15].

Crystal data of 1

Crystals were prepared as described above; $[WI_2(CO)_2(\eta^2\text{-}MeC_2Me)_2]$, $WI_2\text{-}C_{10}H_{12}O_2$, $M = 601.9$, monoclinic, *a* 7.317(7), *b* 13.417(11), *c* 15.445(12) Å, β 93.7(1)°, *U* 1513.11 Å^3, D_{calc} 2.62, D_{meas} 2.67 g cm^{-3}, $F(000) = 1074$, $Z = 4$, λ 0.7107 Å, μ 121.0 cm^{-1}, space group $P2_1/n$.

A crystal of approximate size 0.3 × 0.3 × 0.3 mm was set up to rotate about the *a* axis on a Stoe Stadi2 diffractometer and data was collected via variable width ω scan. Background counts were for 20 s and a scan rate of 0.0333°/s was applied to a width of $(1.5 + \sin \mu/\tan \theta)°$. 2475 independent reflections were measured of which 1782 with $I > 3\sigma(I)$ were used in subsequent refinement. An empirical absorption correction [22] was applied. The structure was determined by the heavy-atom method. All non-hydrogen atoms were refined anisotropically. Hydro-

Table 1

Atomic coordinates ($\times 10^4$) with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
W(1)	5568(1)	2451.4(5)	3723.6(4)
I(1)	3536(2)	4095(1)	2974(1)
I(2)	2087(2)	1727(1)	4169(1)
C(1)	4916(22)	1773(13)	2526(12)
O(1)	4526(26)	1411(11)	1906(9)
C(2)	5371(25)	3270(16)	4848(13)
O(2)	5258(24)	3720(11)	5453(9)
C(4)	8856(28)	4178(16)	4171(14)
C(5)	7902(22)	3376(12)	3682(13)
C(6)	7756(22)	2863(11)	3026(12)
C(7)	8658(34)	2580(14)	2188(11)
C(8)	7596(35)	1430(18)	5584(13)
C(9)	6817(20)	1501(13)	4642(11)
C(10)	6644(21)	998(12)	3947(12)
C(11)	7031(31)	45(14)	3510(16)

Table 2

Molecular dimensions; distances (Å) and angles (°)

W(1)-I(1)	2.862(1)	C(1)-W(1)-C(5)	111.6(6)
W(1)-I(2)	2.851(1)	C(2)-W(1)-C(5)	79.3(7)
W(1)-C(1)	2.089(18)	C(6)-W(1)-C(5)	34.0(6)
W(1)-C(2)	2.067(21)	I(1)-W(1)-C(10)	162.0(5)
W(1)-C(5)	2.115(17)	I(2)-W(1)-C(10)	88.6(4)
W(1)-C(6)	2.062(16)	C(1)-W(1)-C(10)	78.7(6)
W(1)-C(9)	2.076(16)	C(2)-W(1)-C(10)	113.4(7)
W(1)-C(10)	2.123(15)	C(6)-W(1)-C(10)	92.2(5)
C(1)-O(1)	1.095(20)	C(5)-W(1)-C(10)	104.6(6)
C(2)-O(2)	1.120(22)	I(1)-W(1)-C(9)	160.8(5)
C(4)-C(5)	1.465(24)	I(2)-W(1)-C(9)	89.2(4)
C(5)-C(6)	1.224(24)	C(1)-W(1)-C(9)	113.8(6)
C(6)-C(7)	1.539(24)	C(2)-W(1)-C(9)	78.7(7)
C(8)-C(9)	1.530(26)	C(6)-W(1)-C(9)	101.5(6)
C(9)-C(10)	1.268(23)	C(5)-W(1)-C(9)	93.5(6)
C(10)-C(11)	1.481(25)	C(10)-W(1)-C(9)	35.1(6)
		W(1)-C(1)-O(1)	178.0(16)
I(1)-W(1)-I(2)	84.91(4)	W(1)-C(2)-O(2)	179.4(18)
I(1)-W(1)-C(1)	83.9(5)	W(1)-C(6)-C(7)	139.8(12)
I(2)-W(1)-C(1)	84.8(5)	W(1)-C(6)-C(5)	75.3(11)
I(1)-W(1)-C(2)	82.4(5)	C(7)-C(6)-C(5)	144.7(17)
I(2)-W(1)-C(2)	82.3(5)	W(1)-C(5)-C(6)	70.6(10)
C(1)-W(1)-C(2)	162.0(7)	W(1)-C(5)-C(4)	140.7(13)
I(1)-W(1)-C(6)	89.0(4)	C(6)-C(5)-C(4)	148.5(17)
I(2)-W(1)-C(6)	162.4(5)	W(1)-C(10)-C(11)	143.0(14)
C(1)-W(1)-C(6)	78.2(6)	W(1)-C(10)-C(9)	70.4(9)
C(2)-W(1)-C(6)	113.3(7)	C(11)-C(10)-C(9)	146.5(17)
I(1)-W(1)-C(5)	86.3(4)	W(1)-C(9)-C(10)	74.5(10)
I(2)-W(1)-C(5)	160.5(5)	W(1)-C(9)-C(8)	143.9(14)
		C(10)-C(9)-C(8)	141.5(17)

gen atoms were included in calculated positions although the methyl hydrogen atoms were refined as rigid groups.

The structure was given a weighting scheme in the form $w = 1/[\sigma^2(F) + 0.003F^2]$. The final R value was 0.044 ($R_w = 0.052$). Calculations were carried out using Shelx 76 [23] and some of our own programs on the Amdahl V7 at the University of Reading. Positional parameters are given in Table 1 and molecular dimensions in Table 2.

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