

Carbon-carbon and carbon-hydrogen bond activation at ditungsten centers supported by alkoxide ligands

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Abstract - Alkoxide ligands, which are π -donating ligands, complement the behavior of traditional organometallic ligands, which are π -acceptors. Steric control in the determination of coordination number and substrate binding can be introduced by the specific choice of alkoxide ligand. The compounds $W_2(OR)_6$ contain two important functionalities, the alkoxide group and the $(W=W)^{6+}$ unit, and form versatile templates for organometallic chemistry. The reactions between $W_2(OR)_6$ compounds and alkynes give rise to a variety of products derived from either metathesis of the $W=W$ and $C=C$ bonds, carbon-carbon or carbon-tungsten coupling reactions depending upon the alkoxide ligand and the substituents on the alkyne. The addition of ethylene to $W_2(OR)_6$ compounds proceeds via a sequence of reversible steps involving olefin adducts, metallacyclopentanes and ultimately alkyldiyne ligands by C-H activation and elimination of ethane. The compounds $1,2-R'_2W_2(OR)_4$ contain three functionalities of interest: (i) the ancillary alkoxide ligands, (ii) the central $(W=W)^{6+}$ moiety and (iii) metal-alkyl bonds. The reactions between $1,2-R'_2W_2(OR)_4$ compounds and alkynes give rise to a variety of products depending upon the steric properties of R and R', the lack or presence of β -H atoms in the R' group and the nature of the alkyne. Examples include (i) $(Pr^iO)_2(Me_3CCH_2)W=Ct$ by $W=W$ and $C=C$ bond metathesis, (ii) $W_2(\mu-CCMe_3)(H)(OPr^i)_4(\mu-C_4Me_2)$ by C-H activation and C-C bond formation, (iii) alkyne adduct formation, e.g. $W_2(OPr^i)_4(CH_2Ph)_2-(\eta^2-C_2R_2)_2$, where R = Me and Et, and (iv) $W_2(\mu-C_2Me_2)_2(OPr^i)_4$ by alkyl group disproportionation or M-C bond homolysis. Mechanistic studies of the above are discussed on the basis of results obtained from both isotopic labelling experiments and kinetic studies.

1. INTRODUCTION

Organometallic chemistry has developed largely through the use of soft π -acceptor ligands such as carbon monoxide, tertiary phosphines, π -olefins, -cyclopentadienes and -carbocyclic systems (ref. 1). The use of ancillary hard π -donor ligands such as oxo, imido and alkoxido ligands has only recently attracted attention (ref. 2). Soft π -acceptor and hard π -donor ligands complement each other. Both sets of ligands behave as π -buffers and whereas the π -acceptors stabilize d-electron rich metal centers in low oxidation states, the π -donor ligands stabilize d-electron poor metals in high oxidation states.

The alkoxide ligand is a particularly attractive spectator or ancillary ligand since the specific choice of R may greatly influence the coordination number of the metal and the binding of a substrate. Moreover, the electron releasing property of the RO^- ligand is dependent on the M-O-C angle (ref. 3). The maximum π -donation of 4 electrons correlates with a linear M-O-C moiety while a bent M-O-C group has two inequivalent lone pairs, one of π -donating character, the other, a mixture of oxygen 2s and 2p, is poised to form a metal-alkoxide bridge. The flexibility of the M-O-C angle, coupled with the ability of metal-alkoxide bridge formation affords the metal atom a unique degree of freedom to respond to the electronic changes that may occur upon substrate uptake or elimination.

The favorable properties of alkoxide ligands coupled with the inherent reactivity of the inorganic functional group $(W=W)^{6+}$ of M-M valence configuration $\sigma^2\pi^4$ make the compounds $W_2(OR)_6$ some of the most versatile templates for organometallic chemistry (ref. 4). In this short account I recall how this has been seen in the reactions between $W_2(OR)_6$ compounds and alkynes and ethylene and, furthermore, in the chemistry of $1,2-R'_2W_2(OR)_4$ compounds, where R' = an alkyl group (ref. 5).

2. $W_2(OR)_6 + R'C=CR'$

Three types of reactions have been observed.

2.1. A metathesis of the $W=O$ and $C=C$ bonds

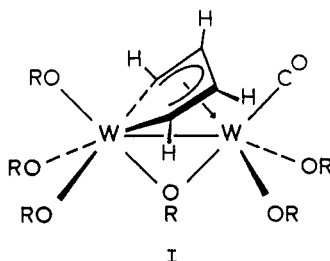
This was first noted by Schrock and coworkers in the reactions between $W_2(OBu^t)_6$ and $R'C=CR'$, where $R' = Me, Et$ and Pr , eq. (1) (ref. 6). Reaction (1) is rapid at room



temperature in hydrocarbon solvents but has steric limitations. For example, there is no reaction between $W_2(OBu^t)_6$ and $Bu^tC=CBu^t$ or $Me_3SiC=CSiMe_3$, though the alkylidyne compounds $(Bu^tO)_3W=CR'$ can be made from reactions between $W_2(OBu^t)_6$ and $R'C=CH$, where $R' = Me_3Si$ or Bu^t (ref. 7). Several of the $(Bu^tO)_3W=CR'$ compounds are of interest as alkyne metathesis catalysts (ref. 8 & 9).

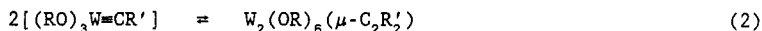
2.2 Carbon-carbon coupling reactions

With less sterically demanding alkoxide ligands such as Pr^iO and Me_3CCH_2O , alkyne-alkyne coupling reactions are generally favored over the $W=O$ and $C=C$ metathesis reaction. Products such as $W_2(OPr^i)_6(\mu-C_4Me_4)(\eta^2-C_2Me_2)_2$ have been isolated and fully characterized (ref. 10). Even in the reaction between $W_2(OBu^t)_6$ and ethyne (the least sterically encumbered alkyne), carbon-carbon bond formation is favored giving $W_2(OBu^t)_6(\mu-C_4H_4)$ which has been isolated and fully characterized as a carbonyl adduct, $W_2(OBu^t)_6(\mu-C_4H_4)(CO)$ (ref. 10 & 11). The structure of $W_2(OBu^t)_6(\mu-C_4H_4)(CO)$ is depicted by I below.



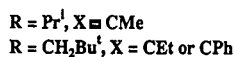
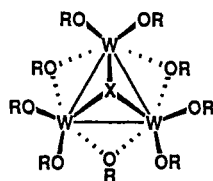
When C-C coupling occurs there is evidence for the initial formation of a $W_2(OR)_6(\mu-C_2R'_2)$ compound and several of these have been structurally characterized as pyridine adducts (ref 12). In each there is a μ -perpendicular alkyne ligand. The central $W_2(\mu-C_2R'_2)$ moiety, which may be termed a dimetallatetrahedrane, is supported by one or two alkoxy bridges. The C-C distances of the alkyne are the longest reported for $M_2(\mu-C_2)$ containing compounds, e.g. C-C = 1.44(1) Å in $W_2(OBu^t)_6(\mu-C_2H_2)(py)$, and have very small values for $^{13}C_{13}C_{13}C$ for the $\mu-C_2$ moiety, ca. 10-15 Hz. This contrasts with carbonyl supported $M_2(\mu-C_2H_2)$ containing compounds, e.g. $^{13}C_{13}C_{13}C = 56$ Hz in $Co_2(CO)_6(\mu-C_2H_2)$ (ref. 13 & 14).

There is evidence in a number of instances for the existence of an equilibrium between the $W_2(\mu-C_2R'_2)$ containing compound and the alkylidyne derivative, eq. (2) (ref. 12, 15 & 16).



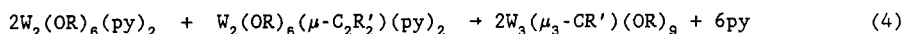
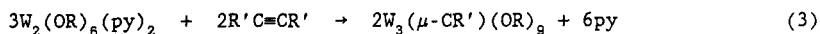
2.3. Carbon-to-tungsten bond formation

In certain instances the $C=C$ and $W=O$ metathesis reaction noted in 1 above gives rise to a highly reactive $(RO)_3W=CR'$ intermediate that reacts rapidly with the $W_2(OR)_6$ compound to give alkylidyne capped tritungsten complexes, $W_3(\mu_3-CR')(OR)_9$ (ref. 17). The latter adopt the structure depicted by II.



II

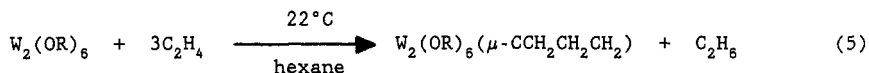
The successful synthesis of $W_3(\mu_3-CR')(OR)_9$ compounds may be achieved by reactions (3) and (4), where $R = Pr^i$ and CH_2CMe_3 and $R' = Me, Et$ and Ph (ref 15).



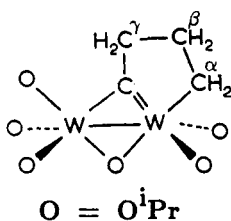
In certain instances all three types of reactions (1 to 3 above) are competitive and different products are obtained under different experimental conditions. Such is the case in the reactions between $W_2(OPr^i)_6(HNMe_2)_2$ and $EtC=Ct$ which can lead to (a) $[(Me_2NH)(Pr^iO)_3W=Ct]_2$, (b) $W_3(\mu_3-Ct)(OPr^i)_9$ and (c) $W_2(OPr^i)_6(\mu-C_4Et_4)(\eta^2-C_2Et_2)$ (ref. 16 & 18).

3. $W_2(OR)_6 + C_2H_4$

A general reaction between $W_2(OR)_6$ compounds and ethylene occurs according to the stoichiometry shown in eq. (5), where $R = Pr^i, CH_2CMe_3, cy-C_3H_9$ and $cy-C_8H_{11}$ (*cy* is *cyclo*).



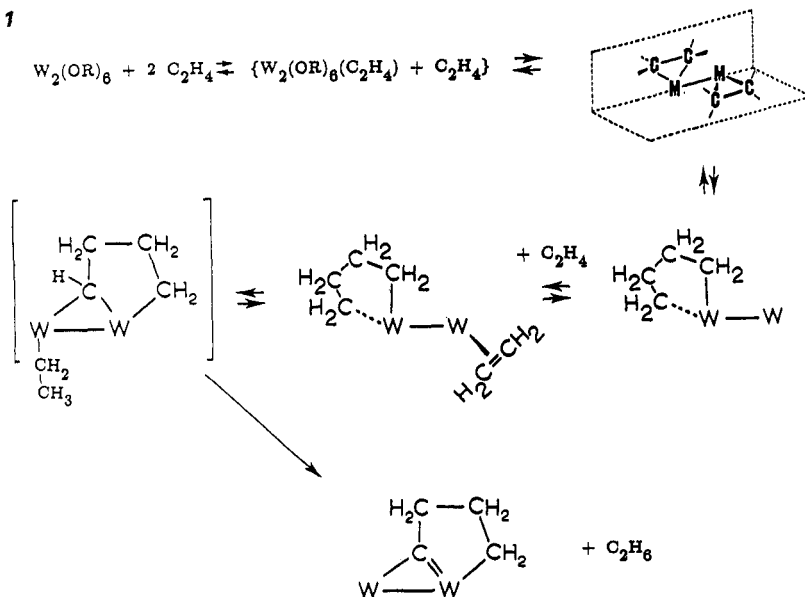
There is no reaction between $W_2(OBu^t)_6$ and ethylene under comparable conditions. The compounds $W_2(OR)_6(\mu-CCH_2CH_2CH_2)$ are similar (based on spectroscopic studies) and adopt the structure depicted by III below. A pyridine adduct has been structurally characterized by an X-ray study. The latter contains a weakly coordinated py molecule, as evidenced by the long W-N bond distance, 2.41 Å, attached to W(1) of the novel 1,6-ditungstacyclo[3.1.0]hex-1(5)-ene moiety.



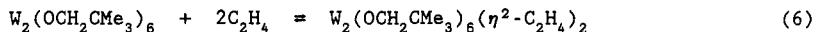
III

A general reaction pathway for the formation of the alkylidyne bridged complexes can be formulated and this is shown in Scheme 1.

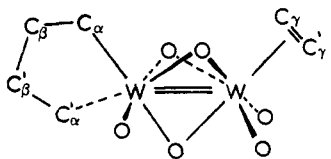
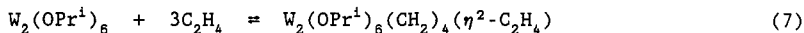
Scheme 1



There is an initial reversible binding of two equivalents of ethylene and this can be monitored for the neopentoxide, eq. (6) (ref 20).



Studies of eq. (6) reveal no evidence for the intermediacy of a monoethylene adduct $W_2(OCH_2CMe_3)_6(C_2H_4)$, and the use of labelled ethylenes, cis, trans, and gem- $C_2H_2D_2$ shows that no isotopic scrambling occurs. This rules out C-H bond activation and olefin metathesis reactions competing with (6). However in the presence of excess ethylene $W_2(OCH_2CMe_3)_6(\eta^2-C_2H_4)_2$ reacts further to give $W_2(OCH_2CMe_3)_6(\mu-CCH_2CH_2CH_2)$ via the intermediacy of $W_2(OCH_2CMe_3)_6(CH_2)_4(\eta^2-C_2H_4)$. Though the latter compound has not been isolated for the neopentoxide, the reaction between $W_2(OPr^i)_6$ and ethylene leads to the reversible formation of $W_2(OPr^i)_6(CH_2)_4(\eta^2-C_2H_4)$, eq. (7), which has been characterized by an X-ray study (ref. 19). Diagrammatically the structure is shown in IV.



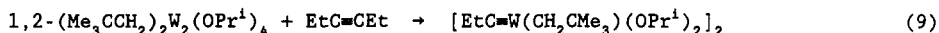
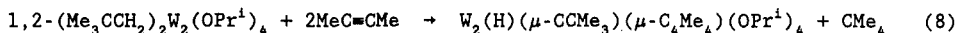
IV

There are a number of interesting questions that are raised in Scheme 1 concerning how the carbon-carbon bonds are formed and broken and how carbon-hydrogen bond activation occurs at the dinuclear metal center. These questions also are raised in our studies of the reactions involving $1,2-(R')_2W_2(OR)_4$ compounds outlined below.

4. $1,2-(R')_2W_2(OR)_4$ + ALKYNES

4.1 General comments

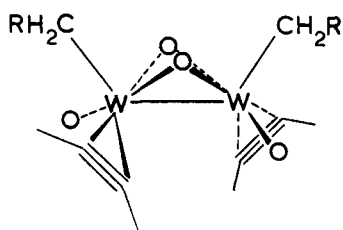
These reactions reveal the greatest sensitivity to steric and electronic factors that we have seen. For example, ethyne, diphenylacetylene, di-*t*-butylacetylene and terminal acetylenes do not react (except to form polyacetylenes) yet $MeC=CMe$, $MeC=CET$ and $EtC=CET$ all show reactivity that is dependent upon the nature of R and R' (ref. 21). The extreme sensitivity to steric factors is seen in the stoichiometry of the reactions between $1,2-(Me_3CCH_2)_2W_2(OPr^i)_4$ and $MeC=CMe$ and $EtC=CET$ which proceed according to eqs. (8) and (9), respectively. In each case the ditungsten compound is isolated in greater than 60% yield and by 1H NMR studies each product is formed rapidly. [The use of only one equivalent of alkyne in (8) lead to a mixture of starting material and product (ref. 21).]



The W=W and C=C metathesis reaction (9) parallels those described in the reactions between certain $W_2(OR)_6$ compounds and alkynes in section 2.1.

4.2. Alkyne adducts and studies of their thermolysis

The reaction (8) requires a number of fundamental steps and the order in which these steps occur can not be inferred from literature precedent. However, with less sterically demanding alkyl ligands, R', e.g. Ph, $PhCH_2$ and Pr and Et, it is possible to isolate intermediates of formula $W_2(R')_2(OPr^i)_4(alkyne)_2$ and structure type V (ref. 21 & 22).

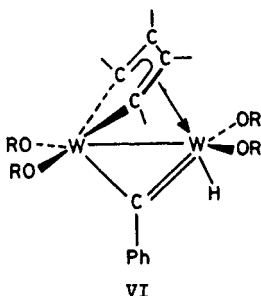


[O = OPrⁱ]

V

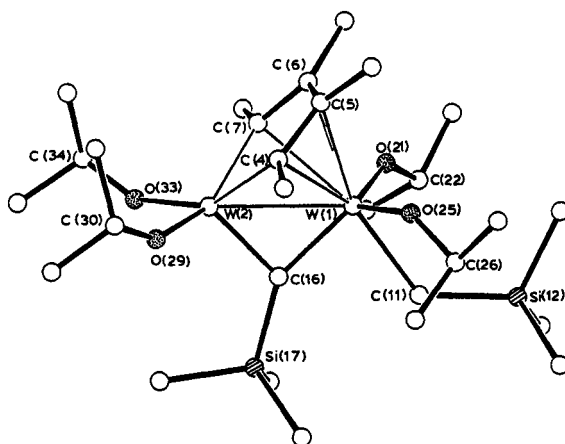
In compounds of formula $W_2(R')_2(OPr^i)_4(alkyne)_2$ the alkyne ligand may be considered a metallacyclopropene moiety having relatively short W-C distances, 2.05 Å, and C-C distances of 1.31 Å for the η^2-C_2 carbons. The W-W distances of 2.67(1) Å are effectively single bond distances consistent with the oxidation of the $(W=W)^{6+}$ unit to $(W-W)^{10+}$ upon coordination and reduction of two alkyne ligands, one at each metal center (ref. 3). In solution there is rotation about the W- η^2-C_2 axis that can be monitored on the NMR time-scale.

The compounds $W_2(CH_2R)_2(OPr^i)_4(alkyne)_2$ react in solution by competitive double α -H activation pathways. In all cases the major product is the alkylidyne bridged hydrido compound VI formed by elimination of RCH_3 and a coupling of the two alkyne units to give a μ -metallacyclopentadiene ligand (ref. 21 & 22).



The rates of thermolysis of the compounds of formula $W_2(CH_2R)_2(OPr^i)_4(alkyne)_2$ followed the order $k_{Bu^t} > k_{SiMe_3} > k_{Ph}$ and for the alkyne $k_{MeC\equiv CMe} > k_{EtC\equiv CEt}$. In all cases the thermolysis was shown to be 1st order in $W_2(CH_2R)_2(OPr^i)_4(alkyne)_2$. A small kinetic isotope effect $k_H/k_D = 1.4$ was observed when $W_2(CH_2Ph)_2(OPr^i)_4(MeCCMe)_2$ and $W_2(CD_2Ph)_2(OPr^i)_4(MeCCMe)_2$ were compared. The activation parameters for $W_2(CH_2Ph)_2(OPr^i)_4(MeCCMe)_2$ ($\Delta H^\ddagger = 21$ kcal mol $^{-1}$, $\Delta S^\ddagger = -9$ eu) and $W_2(CH_2SiMe_3)_2(OPr^i)_4(MeCCMe)_2$ ($\Delta H^\ddagger = 23$ kcal mol $^{-1}$, $\Delta S^\ddagger = -9$ eu) indicate that the differences in rates of thermolyses are enthalpic in origin. This observation, together with the rate dependence on alkyne, $k_{MeCCMe} > k_{EtCCEt}$, lead us to believe that the rate determining step is alkyne-alkyne coupling and that this is followed by a series of rapid steps some of which involve C-H bond activation.

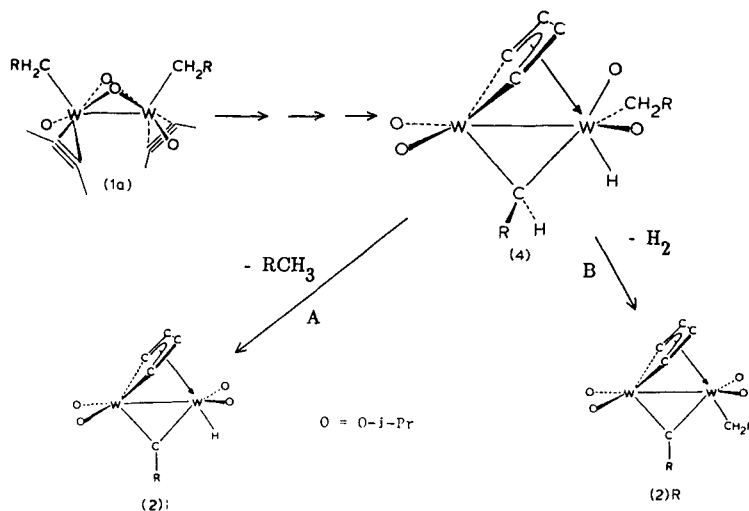
The minor product in the thermolysis of $W_2(CH_2R)_2(OPr^i)_4(alkyne)_2$ compounds is VII, formed by elimination of H_2 . Only in the case of $W_2(CH_2SiMe_3)_2(OPr^i)_4(MeCCMe)_2$ does this reaction pathway contribute significantly and the compound $W_2(\mu-C_5SiMe_3)(\mu-C_4Me_4)(OPr^i)_4$ is even formed slowly (ca. 150 days) in the solid-state at room temperature (ref. 21).



The position of the terminal hydride ligand in VI was not ascertained from crystallographic studies but was established by NOE difference spectra. In the single crystal X-ray structural determination of $W_2(\mu-C_5SiMe_3)(\mu-C_4Me_4)(CH_2SiMe_3)(OPr^i)_4$ we see the remarkable similarity between VI and VII wherein the CH_2SiMe_3 ligand substitutes for H. All the other structural parameters are virtually identical.

The competitive elimination of Me_4Si and H_2 are intramolecular processes which leads us to suspect that a common reactive intermediate must be involved since both processes require α -CH activation (at least as formally defined in mononuclear chemistry). It is conceivable that the partitioning of the reaction occurs from an alkylidene-hydride. Furthermore there must be transference of groups from one metal atom to the other. Scheme 2 presents a plausible pathway for the reaction. The coupling of the alkynes occurs prior to C-H bond activation. Elimination of alkane versus H_2 are competitive from a hydrido-alkylidene-alkyl complex and, as in mononuclear chemistry, elimination by C-H bond formation is favored to elimination by H-H or C-C bond formation (ref. 23).

Scheme 2



Although there is no evidence for any intermediate in the reaction between 1,2- $(\text{Me}_2\text{CCH}_2)_2\text{W}_2(\text{OPr}^i)_4$ and $\text{MeC}\equiv\text{CMe}$ (2 equiv) that gives $\text{W}_2(\text{H})(\mu\text{-CCMe}_3)(\mu\text{-C}_2\text{Me}_4)(\text{OPr}^i)_4$ even at -78°C , eq. (8), it seems likely that this proceeds via a reactive bis-alkyne adduct. Presumably in the reaction with $\text{EtC}\equiv\text{CEt}$ a bis acetylene adduct is not formed because its precursor, the monoalkyne adduct $\text{W}_2(\text{CH}_2\text{CMe}_3)_2(\text{OPr}^i)_4(\text{EtCCEt})$ is sterically congested and is kinetically labile toward the $\text{W}=\text{W}$ and $\text{C}=\text{C}$ metathesis reaction, eq. (9).

Clearly very subtle factors are at work in shunting the reaction pathways in different directions. An example of a 1,2-migration at the ditungsten center was seen in the reaction between 1,2- $(\text{Me})_2\text{W}_2(\text{OBu}^t)_4(\text{py})_2$ and $\text{MeC}\equiv\text{CMe}$ that gave a blue crystalline compound at low temperatures ($< -20^\circ\text{C}$). This compound was crystallographically characterized and shown to be $(\text{py})(\text{Bu}^t\text{O})_2\text{W}(\mu\text{-C}_2\text{Me}_2)(\mu\text{-OBu}^t)\text{W}(\text{Me})_2(\text{OBu}^t)$ with a μ -perpendicular alkyne bridge (ref. 21). Above -20°C in hydrocarbon solvents this compound decomposed to give, amongst others, the well characterized $(\text{Bu}^t\text{O})_2\text{W}=\text{CMe}$ (ref. 7 & 24), again implying that the monoalkyne adduct is the precursor to $\text{W}=\text{W}$ and $\text{C}=\text{C}$ bond metathesis.

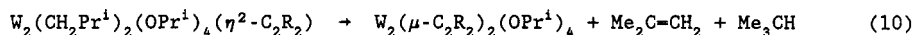
4.3. Photolysis of 1,2- $(\text{PhCH}_2)_2\text{W}_2(\text{OPr}^i)_4(\text{RCCR})_2$

The bis alkyne adducts of the benzyl compounds are photolytically sensitive and eliminate toluene and bibenzyl. The former elimination generates the benzylidyne hydride, VI, while the latter generates $\text{W}_2(\text{OPr}^i)_4(\mu\text{-C}_2\text{R}_2)_2$, where $\text{R} = \text{Me}$ and Et . The latter are only formed in up to 40% by photolysis of the benzyl compounds and a better synthesis is found from 1,2-dialkyl ditungsten compounds that contain β -hydrogen atoms as described below.

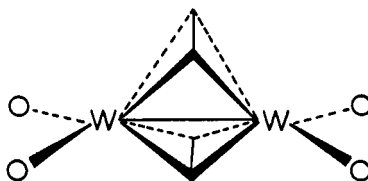
4.4 Thermolysis of 1,2- $(\text{RCH}_2)_2\text{W}_2(\text{OPr}^i)_4(\text{C}_2\text{R}'_2)_2$ compounds, where $\text{R} = \text{Me}, \text{Et}$ and Pr^i and $\text{R}' = \text{Me}$ and Et

The addition of either $\text{MeC}\equiv\text{CMe}$ or $\text{EtC}\equiv\text{CEt}$ to 1,2- $(\text{RCH}_2)_2\text{W}_2(\text{OPr}^i)_4$ compounds, where $\text{R} = \text{Me}, \text{Et}$ and Pr^i , gives bis-alkyl bis-alkyne adducts that are spectroscopically analogous to V and the structure has been established crystallographically for the compound $\text{W}_2(\text{Pr}^i)_2(\text{OPr}^i)_4(\text{C}_2\text{Me}_2)_2$ (ref. 25). However, these compounds are thermally unstable and slowly decompose at room temperature in hydrocarbon solvents (ref. 21). The compounds $\text{W}_2(\text{CH}_2\text{R})_2(\text{OPr}^i)_4(\text{MeCCMe})_2$, where $\text{R} = \text{Me}$ and Et , react thermally to give 2:1 mixtures of $\text{W}_4(\mu\text{-CR})_2(\mu\text{-C}_2\text{Me}_2)_2(\eta^2\text{-C}_2\text{Me}_2)_2(\text{OPr}^i)_6$ and $\text{W}_2(\mu\text{-C}_2\text{Me}_2)_2(\text{OPr}^i)_4$ via competitive α - and β -hydrogen elimination pathways, respectively (ref. 21 & 25). The formation of the tetragonal compound is accompanied by elimination of 1:1 alkane and Pr^iOH , and provides a rare example of where a W-O bond is sacrificed to

form a W-C bond. In the latter reaction 1:1 alkane and alkene are eliminated. The thermolysis of $W_2(CH_2Pr^i)_2(OPr^i)_4(C_2R_2)_2$ gives exclusively $W_2(\mu-C_2R_2)_2(OPr^i)_4$ with the formation of iso-butylene and iso-butane, according to eq. (10).



The structure of the bis-dimethylacetylene bridged compound was determined and is diagrammatically depicted by VIII below (ref. 21 & 26).



VIII

5. CONCLUDING REMARKS

From the above it is evident that the $(W=W)^{6+}$ moiety supported by alkoxide ligands is a most versatile template upon which to carry out organometallic reactions. The mere addition of ethylene or alkynes to $W_2(OR)_6$ compounds generates W-C bonds in a variety of ways that are influenced by the nature of the alkoxide. If alkyl groups are also introduced to the $(W=W)^{6+}$ center, as in the chemistry described for 1,2- $(R)_2W_2(OPr^i)_4$ compounds, then the chemistry becomes even more complex and fascinating as a plethora of C-H bond activation, and C-C coupling reactions become possible. Undoubtedly this is a fertile area of organometallic chemistry for the future and provides tremendous challenges for mechanistic studies.

Acknowledgements

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