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Reactivity of bis(alkene)tetracarbonyl complexes of tungsten: evidence for alkene to π-allyl hydride rearrangement

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Abstract

The bis(alkene)tetracarbonyl complexes of tungsten were identified as olefin isomerization and metathesis catalyst precursors whose activities are based on the formation of a π-allyl hydride intermediate. It has been observed that the β-hydrogen atom of coordinated olefin may migrate to the metal giving η^3-allylic hydrides which have been characterized by their ^{1}H NMR spectra. This is the first case where the rearrangement of tungsten(0) alkenes coordinated to a π-allylic hydrides intermediate, fundamental to many catalytic mechanisms, can be observed directly. In the presence of Lewis acid the same alkene complexes are catalytically active toward olefin metathesis. It seems reasonable that the catalytically active alkylidene compounds are formed from the coordinated alkene via π-allylic hydride and metallacyclobutane precursors.

Keywords: π-Allyl hydride intermediate; Isomerization; Metathesis; Tungsten; Bis(alkene) complexes

1. Introduction

Tungsten carbonyl compounds are known to catalyse several olefin reactions e.g., isomerization (*cis–trans* and double bond migration), metathesis, and polymerization [1,2] but mechanistic data are scarcely available. During our continuing study of the $W(CO)_6$ photocatalytic activity in reaction of alkenes we set out to prepare bis(alkene)tetracarbonyltungsten complexes, which can be the prototypical catalyst in the photocatalytic reaction of alkenes with $W(CO)_6$ [3]. We have, therefore, undertaken a study to establish the nature of the species produced from the reaction of bis(alkene)tetracarbonyltungsten complexes. Here we report our initial results on the thermal reaction of those compounds. These shed much light on the nature and method of formation of the catalytic species and provide some evidence on the mechanism of the isomerization and metathesis reaction. We have found that thermal reaction of bis(alkene) compounds leads to the formation of π-allylic hydride derivatives of tungsten. These studies are described below.

2. Results and discussion

2.1. Isomerization of alkenes

The thermal and photochemical isomerization of external olefins into the thermodynamically favored internal olefins in the presence of $M(CO)_6$ (M = W or Mo) has been extensively

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studied as a model process in homogeneous catalysis [1,2]. A key step in this isomerization of olefins under hydride-free conditions is believed to be the oxidative addition of a transition metal complex to an allylic carbon–hydrogen bond of olefinic substrate and formation of π-allyl hydride intermediate [4]. Reductive elimination of the olefin from this intermediate can then result in isomerization if the hydrogen moves to C_1 instead of returning to C_3 (in the case of terminal olefin). While a $[HM(CO)_n(\pi\text{-allyl})]$ intermediate has been invoked to account for the alkene isomerization, the only metalcarbonyl species observed in photochemical reaction of $M(CO)_6$ with alkene are $[M(CO)_5(\eta^2\text{-alkene})]$ and $M(CO)_4((\eta^2\text{-alkene})_2]$ complexes [1–3].

The synthesis of the bis(alkene)tetracarbonyl complexes of tungsten *trans*-$[W(CO)_4(\eta^2\text{-alkene})_2]$, where alkene = 1-pentene, 1-hexene, 2-hexene, 1-heptene, 1-octene, 1-decene, cyclopentene, cyclohexene, cycloheptene, and cyclooctene has been described recently [3]. These are soluble in aliphatic and aromatic hydrocarbons and the solutions are thermally stable at room temperature in oxygen free atmosphere. A

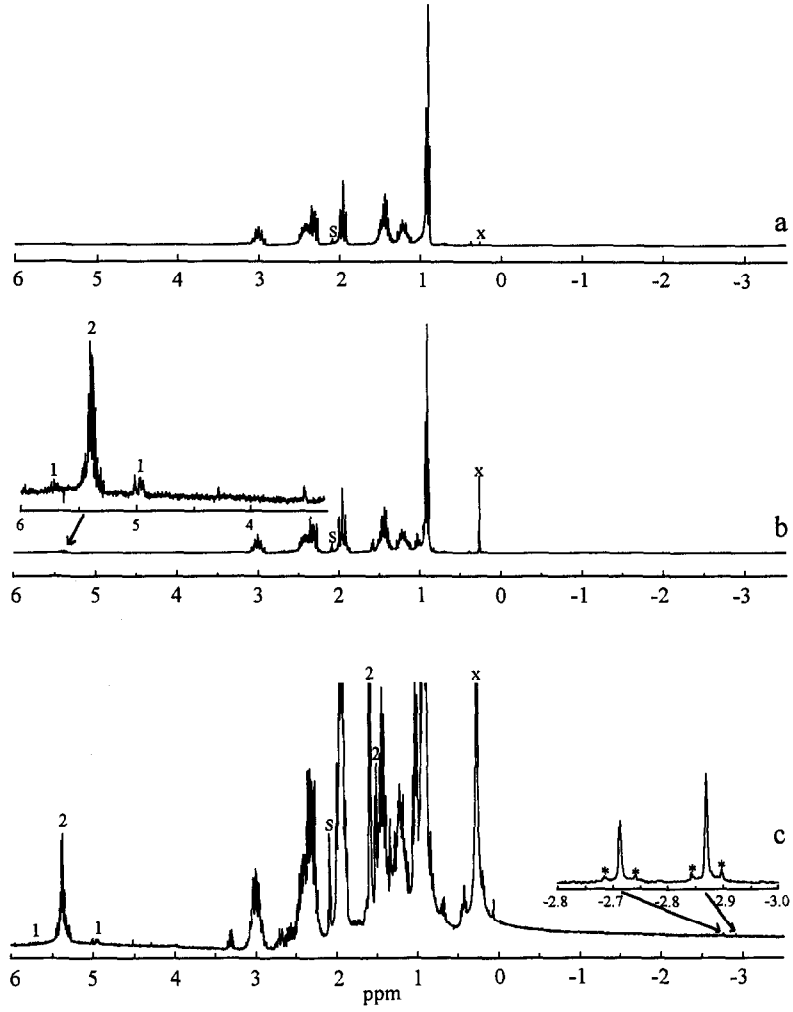
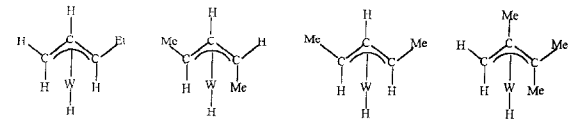


Fig. 1. Variable temperature ^1H NMR spectrum at 300 MHz of *trans*-$[W(CO)_4(1\text{-pentene})_2]$ (1) in toluene-d_8 showing the thermolysis products. (a) At the beginning at 293 K, (b) after 6 h heating at 348 K, (c) after 29 h heating at 348 K. The signals are labelled as follows: (S) proton impurities in the solvent; (1) protons of free 1-pentene; (2) protons of free 2-pentene; (×) grease; (*) ^{183}W satellites. The resonances in the range δ 6–3.5 ppm (b) and about δ −3.0 ppm (c) are shown with scale expansion in the insets.

trans structure has been assigned to these compounds on the basis of their IR and NMR spectra. Interestingly, the alkene molecules are staggered with respect to one another and eclipse the CO–W–CO vectors. Rapid rotation about the metal–olefin bond occurs at 293 K with an estimated barrier of 39–49 kJ mol^{-1} [3]. We used the compounds, *trans*-[$W(CO)_4$(1-pentene)$_2$] (**1**), *trans*-[$W(CO)_4$)(1-hexene)$_2$] (**2**), and *trans*-[$W(CO)_4$(2,2,dimethyl-2-butene)$_2$] (**3**) as the models in investigation of the mechanism of isomerization and metathesis reaction of olefins. We have studied the thermal decomposition of bis(alkene) derivatives by ^{1}H NMR measurement and GC–MS analysis.

Fig. 1 shows a comparison of the ^{1}H NMR spectral changes accompanying a thermal reaction of *trans*-[$W(CO)_4$(1-pentene)$_2$] (**1** in the NMR tube. The ^{1}H NMR of **1** in toluene-d_8 at 293 K (Fig. 1a) shows resonances of coordinated 1-pentene at δ 2.99 (=C*H*), ~2.4 (C*H*H), 2.31 (=C*H*H), 1.96 (=C*H*H), 1.42 (CH_2), 1.19 (C*H*H) and 0.90 (CH_3). Two complex signals of equal intensity at δ 2.4 and 1.2 ppm are assigned to the nonequivalent methylene hydrogen atoms of C_3 carbon. On heating the toluene-d_8 solution of **1** in NMR tube no significant reaction occurs up to 323 K. At 323 K new resonances grow in at δ 5.68 (=C*H*), 4.92 (=C*H*H and 4.86 ppm (=CH*H*), due to olefinic protons of free 1-pentene (Fig. 1b). Much more intense signals of free 2-pentene appear at δ 5.39 (=C*H*), 1.95 (CH_2), 1.55 and 1.48 (=CHCH_3) due to methyl protons of *trans* and *cis*-2-pentene respectively, 0.85 and 0.84 due to methyl protons in =CHCH_2CH_3 group of *trans* and *cis*-2-pentene respectively. New signals at δ 4.48, 4.28, 3.70, 3.34, 2.75, 1.03, 0.42, 0.18, −0.10, −0.45 ppm are most probably due to protons of coordinated to tungsten π-allyl moieties in **1a**–**1c** (Fig. 1b). After 29 h of heating at 348 K a broad resonances appear at δ ca. −3 ppm. After cooling the sample to 309 K this broad signal gives two sharp resonances flanked by tungsten satellites (^{183}W, I = 1/2, 14.5% natural abundance); signal at δ −2.814 ppm, with tungsten–hydride coupling constant $^1J^{183}{}_{W-^1H}$ = 17.3 Hz and at δ −2.966 ppm with $^1J^{183}{}_{W-^1H}$ = 16.7 Hz; both resonances in the ratio 1:1.6 respectively (Fig. 1c). Clearlythese latter resonances result from a W–H bond formation.

The reaction of **1** can lead to a mixture of three isomeric form of η^3-allylic ligand, **1a**, **1b** and **1c**. Compounds **1b** and **1c** are products of 1,3 hydrogen shifts in **1a**, and differ in the configuration of the methyl groups: in **1c** these both occupy a *syn* position while in **1b** one methyl group is *syn* and the other *anti*. Presence of two hydride resonances indicate that π-allyl hydrides **1a**–**1c** can exist in isomeric forms of different relative stabilities which convert at higher temperatures to the most stable two forms, most probably **1b** and **1c**.



1a 1b 1c 3a

The new resonances which appear in the ^{1}H NMR spectra of **1** in the range 4.5 to −0.45 ppm (Fig. 1b, c) are readily assigned to η^3-allyl metal hydrides **1a**, **1b**, and **1c** which are formed from coordinated 1-pentene.

The allyl complexes formed are too dilute for accurate integration and specific coupling constant calculation but resonances were observed for most of protons in the molecule **1a**, **1b** and **1c**. The most characteristic feature in the ^{1}NMR spectra of thermolysis products of **1** are signals of π-allyl ligands which appear in the range 4.5–3.3 ppm. This region does not overlap the resonances of **1** and of the free 1-pentene and 2-pentene. The signal at 3.70 ppm appears as a quintet with coupling constant J = 5.8 Hz. The signal at 3.35 ppm is a well resolved doublet of doublets with J = 6.5 Hz. These spectra are closely analogous to those of other π-allylic moieties, for example, some [$W(\eta^3\text{-}C_3H_5)_3(\eta^3\text{-}1,3\text{-}Me_2C_3H_3)$] complexes (for η^3-1,3-$Me_2C_3H_3$ isomer *anti* signals at δ 4.33, 3.53,

1.52, 1.45, 0.63 ppm, and for isomer *syn*, signals at δ 4.28, 3.25, 1.92, 1.51, −0.13 ppm were observed [5]) and are comparable to the spectrum of $[W(\eta^3\text{-}C_3H_5)_4]$ [6] with signals at δ 3.89, 2.82, 1.66, −0.19 ppm. However, problems of overlap with the resonances of other species hampered identification and precluded definitive characterization of the allyl fragments in **1a–1c**. Signals due to methyl of **1a–1c** were less easy to detect. With position close to those of the coordinated and free alkene, these were most probably obscured by other more intense signals.

Similar spectral changes occur upon heating the other alkene complexes, including the tetramethylethylene compound **3**. The ^{1}H NMR of **3** at 313 K in toluene-d_8 shows the metal–hydride resonance as a triplet ($^1J^{183}{}_{W-^1H} = 16.3$ Hz) at δ −3.05 ppm which suggests formation of π-allyl complex type **3a**.

In addition to the expected *trans*-$[W(CO)_4(1\text{-pentene})_2]$ and signals of the π-allylic moieties, the NMR spectrum of the reaction mixture exhibits very low intensity signals of free 1-pentene (Fig. 1b) and much more intense signals of free 2-pentenes (Fig. 1c). This assignment is supported by the almost quantitative formation of 2-pentenes (identified by gas chromatography and mass spectrometry) upon thermolysis of *trans*-$[W(CO)_4(1\text{-pentene})_2]$ in a sealed ampule (Table 1).

There is a marked change in the *trans* to *cis* ratio of 2-pentenes with variation of temperature and reaction time of **1**. In the initial stages of thermolysis of **1**, the major product is *cis*-2-pentene. But as time progresses, the thermodynamically more stable isomer *trans*-2-pentene gradually accumulates at the expense of *cis*-pentene. The initial ratio of *trans*- to *cis*-pentene obtained at 315 K was lower than 1 ($\approx$ 0.6–0.9). At higher temperature (348 K) *trans*-2-pentene increased and after 4 h reaction time ratio of 2-pentenes reached 3.4 (Table 1). The experiments described so far, clearly illustrates that the kinetically controlled product distribution is enriched in the thermodynamically less stable isomer, *cis*-2-pentene, compared to what the ratio (4.4) would be at thermodynamic equilibrium [7] (2.3% 1-pentene, 79.6% *trans*-2-pentene and 18.3% *cis*-2-pentene). The ratio of the *trans*- to *cis*-2-pentenes is readily obtained also from ^{1}H NMR integration of CH_3 signals at 1.55 and 1.48 ppm of *trans*- and *cis*-2-pentene respectively (Fig. 1c).

The intermediate thermally generated from **1** in the presence of 1-pentene at 348 K leads to

Table 1
Comparison of 1-alkene isomerization using $[W(CO)_4(1\text{-pentene})_2]$ (**1**) and $[W(CO)_4(1\text{-hexene})_2]$ (**2**) catalyst precursor

Alkene complex	Molecular ratio free 1-alkene/alkene complex	T (K)	Time (h)	Conversion [a] (%)	*trans*/*cis* [b] 2-alkene
$W(CO)_4(1\text{-pentene})_2$	[c]	315	1	35.5	0.8
	[c]	315	4	78.0	0.9
	[c]	348	1	74.0	1.9
	[c]	348	4	95.5	3.4
	10	315	4	1.8	1.4
	10	348	1	37.0	0.6
	10	348	4	90.6	1.7
$W(CO)_4(1\text{-hexene})_2$	[c]	348	1	74.9	2.4
	[c]	348	4	95.1	3.1
	10	315	4	1.0	1.8
	20	348	4	16.5	0.6
	40	348	4	8.8	0.8
	560	348	4	3.5	2.0

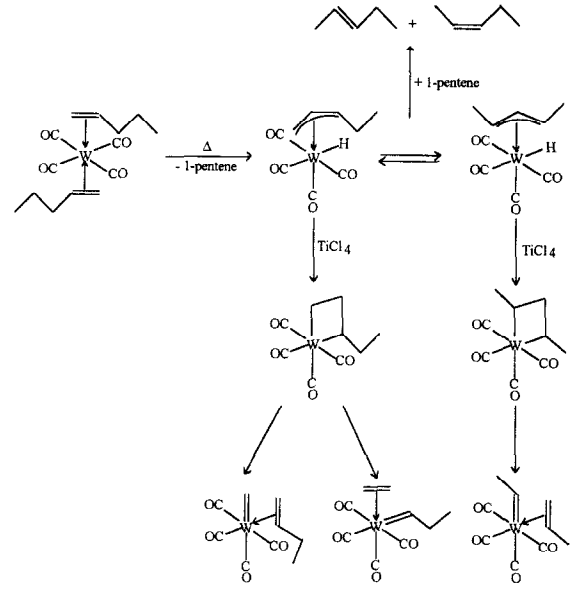
[a] % of 1-alkene isomerized to internal alkenes.
[b] Ratio of *trans* to *cis* isomers of 2-alkene.
[c] 1-Alkene originating only from decomposition of started alkene complexes.

significant isomerization of the uncomplexed 1-pentene (TON = 5).

A similar isomerization reaction is observed with *trans*-[W(CO)$_4$(1-hexene)$_2$] (**2**). Thermal decomposition at 348 K of **2** leads to a mixture of 1-hexene, *cis*- and *trans*-2-hexene and 3-hexene. Thermally formed intermediates initiate isomerization of 1-hexene added in an excess. The ratio of *trans*- to *cis*-2-hexene obtained from decomposition of **2** is similar to that of 2-pentene (Table 1). Further, it is to be noted here that 3-hexene is formed in a substantial amount from 1-hexene.

It is concluded that olefin bond-migration takes place in an η^3-allylmetal hydride intermediate produced from a *trans*-[W(CO)$_4$(1-alkene)$_2$]. Noticeable changes of in the colour of the solution from pale yellow to dark yellow occurred and the reactions often become heterogeneous after a number of hours. In addition, the ^{1}H NMR spectra of the decomposition products, which precipitated from solution after dissolving in acetone-d_6, indicate the presence of other, unidentified species.

These data, taken together, imply that the bis(alkene) complexes of tungsten can be used as catalyst precursor. However, it must be realized that [W(CO)$_4$(alkene)$_2$] itself must be at least one step away from the active alkene isomerization catalyst. We believe that loss of a second alkene must occur in order to actually effect catalysis. There is some evidence in ^{1}H NMR (Fig. 1b) and GC–MS measurement (Table 1) for loss of alkene from bis(alkene) complexes. W–alkene bond fission creates the vacant coordination site. The resulting 16-valence electron species can reversibly form either a *syn*- or an *anti*-π-allyl hydride as a precursor to the *cis*- and *trans*-2-pentene complexes, respectively (Scheme 1). Formation of the π-allyl tungsten hydride can proceed by an internal oxidative addition of an allylic carbon–hydrogen bond of complexed alkene to the five coordinated d^6, W$^{(0)}$ complex [W(CO)$_4$(1-alkene)] to give six coordinate d^4 W$^{(II)}$ complex [WH(η^3-allyl)(CO)$_4$]. Reductive elimination of the olefin



Scheme 1.

from this intermediate can then result in isomerization if the hydrogen moves to C_1 instead of returning to C_3.

2.2. Metathesis of alkenes

Whereas it has become established that the olefin metathesis reaction is initiated by coordinated carbenes [8], there is no evidence on the formation of the first carbene from the olefin. According to the work on metallacyclobutanes reported by Green and coworkers [9] and supporting evidence by Sherman and Schreiner [10] initial carbene can come from the coordinated alkene via π-allylic hydride and metallacyclobutane precursors (Scheme 1). Thus this mechanism predicts the first-formed olefins from 1- and 2-pentene to be ethylene, propylene, and 1-butene (Scheme 1).

The tungsten carbonyl compounds are well-known as olefin metathesis catalysts; [2,11,12] however, their activity depends upon additives, namely, electron withdrawing compounds (Lewis acids). Additives affect the catalytic activity of tungsten carbonyl compounds in one of two ways. A basic requirement for activity of a transition-metal complex toward catalytic alkene reaction is the presence of an open coordination

site for the reversible binding of alkene. Photolysis of tungsten carbonyls or Lewis acid interaction with ligands as CH_3CN, Cl^- etc. creates the free coordination site at tungsten to the incoming alkene [12]. The interaction of the Lewis acid via a CO or other ligand creates an electron deficiency at the tungsten. This facilitates reductive α-elimination processes of ($W^{II} \rightarrow W^0$) causing alkene loss and carbene formation and easier electrophilic atack of the carbene complex on the reactant olefin. However, beyond this, relatively little is known about the nature of catalytic sites, and little information is available concerning the mechanism except to say, that the metathesis products have been identified.

Thermolysis of **1** in neat 1-pentene gives rise to the isomerization products *cis* and *trans* 2-pentene. In contrast, reaction of a solution of **1** and $TiCl_4$ (1:4) causes smooth evolution of ethylene, propylene and 1-butene e.g., olefins which have fewer carbon atoms than were present in the original alkene. We have also observed the appearence of isomerization products, internal pentenes and metathesis products: 2-butene, *cis* and *trans*-2-hexene, 3-hexene, *cis* and *trans*-3-heptene and 4-octene (Table 2). In view of the above it seems reasonable that catalytic active alkylidene complexes ($W{=}CH_2$, W=CHMe, W=CHEt, W=CHPr) are formed from the coordinated alkenes via π-allyl hydride and metallacyclobutane intermediates (Scheme 1).

The formation of metallacyclobutane from π-allyl hydride intermediate can proceed via hydride-ion migration to the central carbon C_2 of the η^3-allylic group while H^- addition to the terminal carbon (C_1 or C_3) would give rise to the alkene complex. It is of interest to find out which factors may control the observed regiospecificity of H^- addition. In fact, from the literature [13], the possition of H^- migration may depends on electron density on the metal centre.

In previously noted catalytic system containing tungsten(0) carbonyl compounds and Lewis acid, (AX_n = metal or non-metal halides) the role of Lewis acids is to remove electron density from the metal [2,11,12].

Table 2
GC–MS analysis of products formed in the reaction of *trans*-[$W(CO)_4$(1-pentene)$_2$]

Components [a]	Area (%)
ethene	6.41
propene	8.16
i	0.24
1-butene	10.11
trans-2-butene	2.95
cis-2-butene	2.15
i	1.04
1-pentene	19.87
i	2.37
trans-2-pentene	12.64
cis-2-pentene	4.84
i	0.47
trans / *cis*-3-hexene	0.40
trans-2-hexene	2.64
cis-2-hexene	0.86
cyclohexane	21.76
trans-3-heptene	1.11
i	0.40
cis-3-heptene	0.76
trans / *cis*-4-octene	0.62
i	0.16

Conditions: *trans*-[$W(CO)_4$(1-pentene)$_2$] (0.04 mmol), $TiCl_4$ (0.16 mmol), 1-pentene (0.4 mmol) and cyclohexane (0.1 mmol) after 4 h reaction time at 348 K.
[a] i = unidentified products, impurities.

We suggest the following general explanation concerning the position of hydride-ion addition to allylic ligand. When the metal centre is electron rich the hydride-ion addition proceeds on the terminal carbon of the η^3-allylic group. But in the presence of Lewis acid, which removes electron density from the metal centre, the hydride-ion addition proceeds on the central carbon of the η^3-allylic group.

We propose a mechanism for the formation of metalacyclobutane from π-allylic hydrides intermediate which proceeds via attack of the H^- on the central carbon of the η^3-allylic group, as shown in Scheme 1. Strong support for this proposal arises from the observation that as predicted, the first-formed olefins from coordinated 1-pentene are ethylene, propylene, and 1-butene (Scheme 1, Table 2).

This mechanism may be relevant as an initiation mechanism for olefin metathesis reactions, especially those catalysed by tungsten carbonyl–Lewis acid systems.

We conclude that our observations on the decomposition of the bis(alkene)tetracarbonyl compounds provide strong support for the feasibility of this mechanism.

3. Experimental

Operations were performed under nitrogen. All the solvents and reagents were dried and distilled before use.

The bis(alkene)teracarbonyl tungsten complexes *trans*-[$W(CO)_4$(1-pentene)$_2$] (**1**), *trans*-[$W(CO)_4$(1-hexene)$_2$] (**2**), *trans*-[$W(CO)_4$(tetramethylethylene)$_2$] (**3**) were synthesized according to the literature procedure [3]. Tetramethylethylene complex **3** was obtained for the first time and characterized only by ^{1}H and ^{13}C NMR spectroscopy. The ^{1}H NMR spectrum of **3** at room temperature consists of one signal at δ 1.968 ppm due to the 12 protons of four methyl groups. The ^{13}C NMR spectrum of **3** at 223 K shows single resonances for CO groups at δ 205.81, olefin carbons at δ 68.40 and methyl carbons at 28.47 ppm.

The analysis of the alkene reaction for starting materials or products was performed on a Hewlett-Packard GC–MS system. A hydrogen flame detector was employed in all cases. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AMX 300 instrument operating at 300 and 75.5 MHz respectively. All spectra were measured in toluene-d_8 and calibrated by using the methyl resonance of the toluene at 2.09 ppm in ^{1}H NMR as an internal standard.

3.1. Typical procedure

Thermal reaction of bis(alkene) complexes **1–3** were run under nitrogen in hermetically sealed ampules. The samples were solutions or suspensions of bis(alkene) complex in neat 1-alkene. In a typical experiment, a solid sample of bis(alkene) complex ($\approx$ 5 mg) and alkene ($\approx$ 10 μl) in molar ratio 1:10 were placed into glass tubes and sealed. The ampules were then placed in an oil bath and heated to effect olefin isomerization. The reaction tubes were opened, and the samples were removed and subjected to GC–MS analysis. The metathesis reaction was carried out in a similar manner except that a fourfold excess of $TiCl_4$ to bis(alkene) complex was added.

The isomerization reaction of coordinated alkene was conveniently monitored in a sealed-tube NMR experiment. The bis(alkene) complex was taken up in 0.5 cm^3 of toluene-d_8 and sealed. ^{1}H NMR showed that the sample remains unchanged for several days at room temperature. The sealed tube was immersed in an oil bath (348 K) and periodically monitored by ^{1}H NMR spectroscopy. After some time, new signals appeared at the expense of bis(alkene) complex.

4. Conclusion

The results described above show the reactivity of bis(alkene)tetracarbonyltungsten complexes as catalyst precursors toward the isomerization and metathesis of olefins.

The catalytic activity of the bis(alkene)tetracarbonyltungsten complexes in the isomerization of alkenes is based on the formation of π-allyl metal hydride.

The *trans*-[$W(CO)_4$(1-alkene)$_2$] complexes, by forming the allyl hydride intermediates, [$HW(\pi$-allyl$)(CO)_4$], yield *cis* and *trans* internal alkenes.

The key isomerization intermediate implies that [$WH(\eta^3$-allyl$)(CO)_4$] can be formed by thermal dissociation of an alkene from *trans*-[$W(CO)_4$(1-alkene)$_2$] as was observed by ^{1}H NMR spectra.

This is the first direct indication for the so-called 'π-allyl mechanism' of olefin isomerization by group 6 metal carbonyls. Hence, this

provides the direct observation of the π-allyl hydride formation from coordinated alkene proposal in many catalyzed olefin reactions.

In the presence of the Lewis acid, $TiCl_4$, alkene coordinated to tungsten rearranges to give new alkenes, products of metathesis and cometathesis reaction. This indicates that the alkylidene ligand initially formed must come from the olefin through a π-allyl metal hydride and a metallacyclobutane complex.

These results are in support of the mechanism involving the formation of the initiating alkylidene from coordinated alkene via a π-allylic hydride and a metallacyclobutane derivatives.

Acknowledgements

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