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Photochemical reaction of $W(CO)_6$ with $SnCl_4$

III. Synthesis of some novel seven-coordinate tungsten(II) complexes $[WCl(SnCl_3)(CO)_3L_2]$ (L≡N- and P-donor ligands)

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Abstract

The photochemical reaction of $W(CO)_6$ with $SnCl_4$ gives the oxidation addition product $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**). Compound **1** reacts with ligands L≡CH_3CN, bipy, PPh_3 or dppe to give the seven-coordinated species $[WCl_2(CO)_3L_2]$ and $[WCl(SnCl_3)(CO)_3L_2]$. These $W^{(II)}$ compounds can be prepared in a one-pot two-step manner, without isolating the intermediate $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**).

Keywords: Tungsten; Tin; Carbonyls; Seven-coordinate

1. Introduction

In recent years we have been investigating the chemistry of $W(CO)_6$–Lewis acid systems [1–4], in which are produced very active catalytic forms for metathesis of alkenes and polymerization of alkynes. The role of Lewis acids in catalytic system containing $M(CO)_6$ (M=W or Mo) is connected rather with the labilization effect of the CO group and its very fast substitution by alkene or alkyne [1–5]. However heterobimetallic compound may be also generated in this system.

In order to explain the role of the Lewis acid, investigations of the photochemical reaction of $W(CO)_6$ with $SnCl_4$ and the subsequent substitution of the CO group with different N- and P-donor ligands in the product $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**) were undertaken. The results of these studies are described below.

2. Results and discussion

The interaction of halotin(IV) derivatives with M^0 (M≡Mo or W) complexes to give seven-coordinated $M^{(II)}$ compounds containing M–Sn bonds has been studied for more than 20 years [6–15]. It has been found that $SnCl_{4-n}R_n$ reacts with a substituted M^0 compound such as $[M(CO)_4L\text{–}L]$ to give seven-coordinated $[MCl(SnCl_{3-n}R)(CO)_3(L\text{–}L)]$, where L–L denotes a bidentate chelating ligand containing nitrogen [6–11], phosphorus [6] or sulfur donors [8b]. Recently, Baker and coworkers [12,13] reported the oxidation addition of $SnCl_4$ to $[M(CO)_3(NCMe)_3]$ to give $[MCl(SnCl_3)(CO)_3(NCMe)_2]$, in which the labile acetonitrile ligands can be replaced by others.

The new method for the preparation of a family of seven-coordinated complexes of tungsten(II) of formula $[WCl_2(CO)_3L_2]$ (L≡PPh_3, dppe or bipy) [16,17] is the photochemical reaction of $W(CO)_6$ in cyclohexane in the presence of CCl_4 and the ligand L. Similar photochemical reaction with $SnCl_4$ as an oxidant gives a mixture of products $[WCl_2(CO)_3L_2]$ and $[WCl(SnCl_3)(CO)_3L_2]$ from which W–Sn compounds were separated by extraction with toluene.

2.1. Photochemical reaction of $W(CO)_6$ with $SnCl_4$ in the presence of N- or P-donor ligands

Irradiation of $W(CO)_6$ in cyclohexane with an equimolar amount of $SnCl_4$ results in the immediate formation of $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**), which is almost quantitatively a precipitate from the reaction mixture. Complex **1** was spectroscopically and crystallographically characterized [18]. The same photochemical

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reaction of $W(CO)_6$ with $SnCl_4$ but in the presence of the ligand L≡PPh_3, dppe or bipy gives seven-coordinated complexes of tungsten(II), $[WCl_2(CO)_3L_2]$ and $[WCl(SnCl_3)(CO)_3L_2]$, which precipitate from the reaction mixture. The IR spectrum of the reaction mixture witnessed a decay of the absorption at 1980 cm^{-1} due to W(CO) with the accompanying appearance of new absorption at a higher frequency (about 2030 cm^{-1}) and at a lower frequency (about 1930 cm^{-1}) owing to the W–Sn product. After irradiation for 2 h in solution only trace amounts of non-reacted $W(CO)_6$ are left. The orange compound isolated under a neutral atmosphere showed a number of carbonyl bands in the IR spectrum. This suggest that several compounds are present in solid. W–Sn compounds were separated by extraction with toluene. The residue was a yellow compound of $[WCl_2(CO)_3L_2]$ type.

2.2. *Reaction of $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (1) with N- or P-donor ligands*

The halocarbonyls of the Group 6 metals in their formal oxidation states of II, $[MX_2(CO)_4]_2$ compounds with M=Mo or W and X=Cl, Br or I have been known since 1966 [19–21] as useful starting materials in synthesis of the $[MX_2(CO)_3L_2]$-type complexes where L=N- or P-donor ligands. Similar cleavage of the chloride bridges in the halocarbonyl compound of W–Sn (**1**) to give seven-coordinated $W^{(II)}$ compounds according to

$$[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3] + 4L \xrightarrow[\text{toluene or } CH_2Cl_2]{\text{room temperature}} [WCl_2(CO)_3L_2] + [WCl(SnCl_3)(CO)_3L_2] \qquad (1)$$

Complex **1** is air sensitive in the solid state and extremely air sensitive in solution, but it can be stored under nitrogen in the solid state for months. Complex **1** is only slightly soluble in aliphatic hydrocarbons but considerably more soluble in aromatic solvents and is readily soluble in CH_2Cl_2 or $CHCl_3$. The complex $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**) is highly soluble in acetonitrile, to give a bright-orange solution that after removal of the solvent in vacuo gives a bright-orange crystalline solid, probably a 1 : 1 mixture of $[WCl(SnCl_3)(CO)_3(NCMe)_2]$ (**2**) and $[WCl_2(CO)_3(NCMe)_2]$, which are considerably less stable than **1**; so far it has not been possible to separate and obtain satisfactory analytical data for them. However, the IR spectrum of this impure compound **2** shows three carbonyl bands at ν(CO) 2027vs, 1933sh and 1900vs cm^{-1} and nitrile stretching bands at 2326w and 2298w cm^{-1} (Fig. 1B). The same IR spectrum was observed for **2** quantitatively formed in reaction of $[W(CO)_4(NCMe)_2]$ with $SnCl_4$ in chlorobenzene. IR spectra are similar to those for **2** and the molybdenum–tin compounds ($[MoCl(SnCl_2R)(CO)_3(NCMe)_2]$, R = Bu, ν(CO) = 2014s, 1935sh and 1916s cm^{-1}; R = Ph, ν(CO) = 2017s, 1930sh and 1920s cm^{-1}) described by Riera and coworkers [14] and ($MoCl(SnCl_2R)(CO)_3(NCMe)_2]$, R = Me, ν(CO) = 2016vs, 1932sh and 1899vs cm^{-1}; R = Ph, ν(CO) = 2017vs, 1935sh and 1920vs cm^{-1}) described by Cano et al. [15], but qualitatively different from that reported by Baker et al. [12] for the tungsten compound ($[WCl(SnCl_3)(CO)_3(NCMe)_2]$, ν(CO) = 2017, 1978 and 1934 cm^{-1}). These results suggest than more than one isomer of **2** can be formed. It should be noted [12] that complex $[WCl(SnCl_3)(CO)_3(NCMe)_2)]$ obtained by Baker et al. could not be isolated in a pure state.

Two sharp bands appear at 2326 and 2298 cm^{-1} for

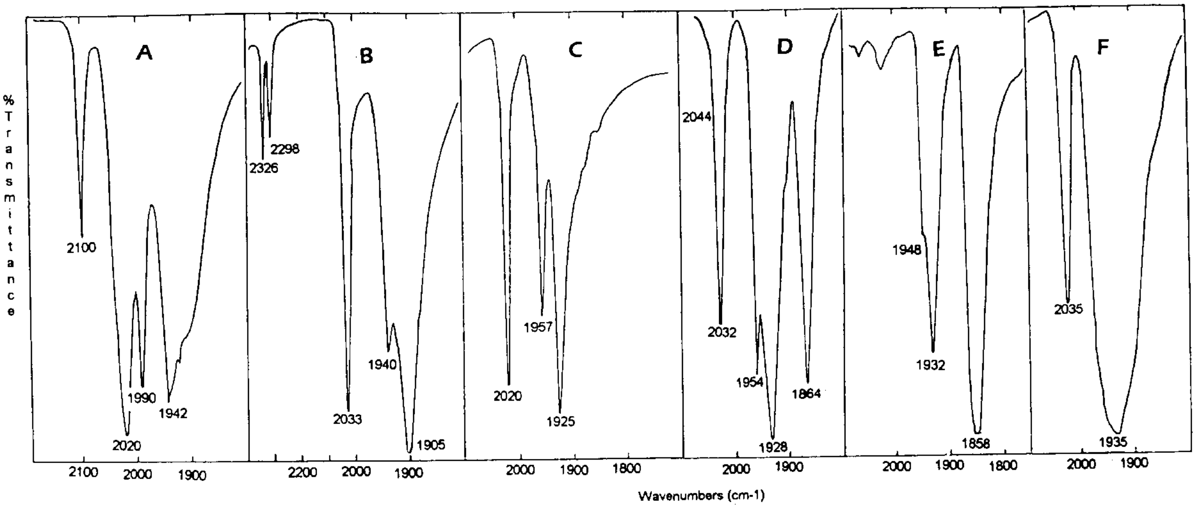

Fig. 1. ν(CO) region of the IR spectrum displayed by W–Sn complexes: A, $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**); B, $[WCl(SnCl_3)(CO)_3(NCMe)_2]$ (**2**); C, $[WCl(SnCl_3)(CO)_3(bipy)]$ (**5**); D, $[WCl(SnCl_3)(CO)_3(PPh_3)_2]$ (**3**) contaminated $[WCl(SnCl_3)(CO)_2(PPh_3)_2]$; E, $[WCl(SnCl_3)(CO)_2(PPh_3)_2]$; F, $[WCl(SnCl_3)(CO)_3(dppe)]$ (**4**).

Table 1
The most relevant IR frequencies ν of the compounds $[WCl(SnCl_3)(CO)_3L_2]$ (**1–5**) in Nujol mulls

Complex	L	ν(CO) (cm^{-1})	ν(MCl) (cm^{-1})	ν(CN) (cm^{-1})
1	CO	2100s, 2020vs, 1990sh, 1942s, 1924sh, 1910sh	348s, 310vw, 288w	
2	NCMe	2033vs, 1940s, 1905vs	349s 323s, 288w 264w	2326, 2298
3	PPh_3	2044sh, 2032s, 1954sh, 1928vs, 1864m	340sh, 330m, 302m	
4	dppe	2035s, 1965sh, 1933vs, 1895sh	344m	
5	bipy	2020vs, 1957s, 1925vs	345w, 328m, 290vw	

vs, very strong; s, strong; sh, shoulder.

2 (Fig. 1B). Each band has a similar intensity, consistent with *cis* nitrile groups [22].

The room-temperature ^1H NMR spectrum of **2** shows the expected single resonance at $\delta(CD_2Cl_2) =$ 2.54 and 2.59 ppm, consistent with two different nitrile groups. The ^{13}C NMR spectrum shows a number of carbonyl resonance at $\delta = 211.82, 208.00, 207.80, 201.77$ 200.87 and 196.76 ppm in the carbonyl region, and hence there must at least two different compounds in solution.

The addition of four molar equivalents of PPh_3 to the dichloromethane solution of **1**, at room temperature, leads to the facile destruction of three chloride bridges, displacement of one carbonyl ligand and the formation of monomeric tricarbonyl W–Sn compound $[WCl(SnCl_3)(CO)_3(PPh_3)_2]$ (**3**) together with the tricarbonyl compound $[WCl_2(CO)_3(PPh_3)_2]$ and small contamination with the dicarbonyl $[WCl(SnCl_3)(CO)_2(PPh_3)_2]$. The same reaction but in toluene solution gives only the tricarbonyl forms, which indicate the influence of dichloromethane on labilization of the CO group. Compound **3** has been isolated as a yellow–orange crystalline solid, and fully characterized by analytical and spectroscopic methods.

The spectral properties of **3** are qualitatively different from that those by Baker and Bury [12] for the analogous $[MoCl(SnCl_3)(CO)_3(PPh_3)_2]$ with ν(CO) bands at 1961s, 1943s and 1881s cm^{-1} but very similar to the other W–Sn compounds of type $[WCl(SnCl_3)(CO)_3L_2]$ (Figs. 1B–1F and Table 1), where L≡CH_3CN, dppe or bipy prepared with this method and characterized by IR spectroscopy. The IR spectra of **2–5** suggest the same *fac* distribution of the three carbonyl groups and the presence of only one isomer with the geometry shown in Fig. 3.

The analogous reaction of **1** with two equivalent of L_2≡dppe or 2,2′-bipyridyl gave the new compounds $[WCl(SnCl_3)(CO)_3L_2]$ (**4** and **5**) together with $[WCl_2(CO)_3L_2]$ and dicarbonyl forms. The separation of **4** and **5** was possible by the extraction of toluene.

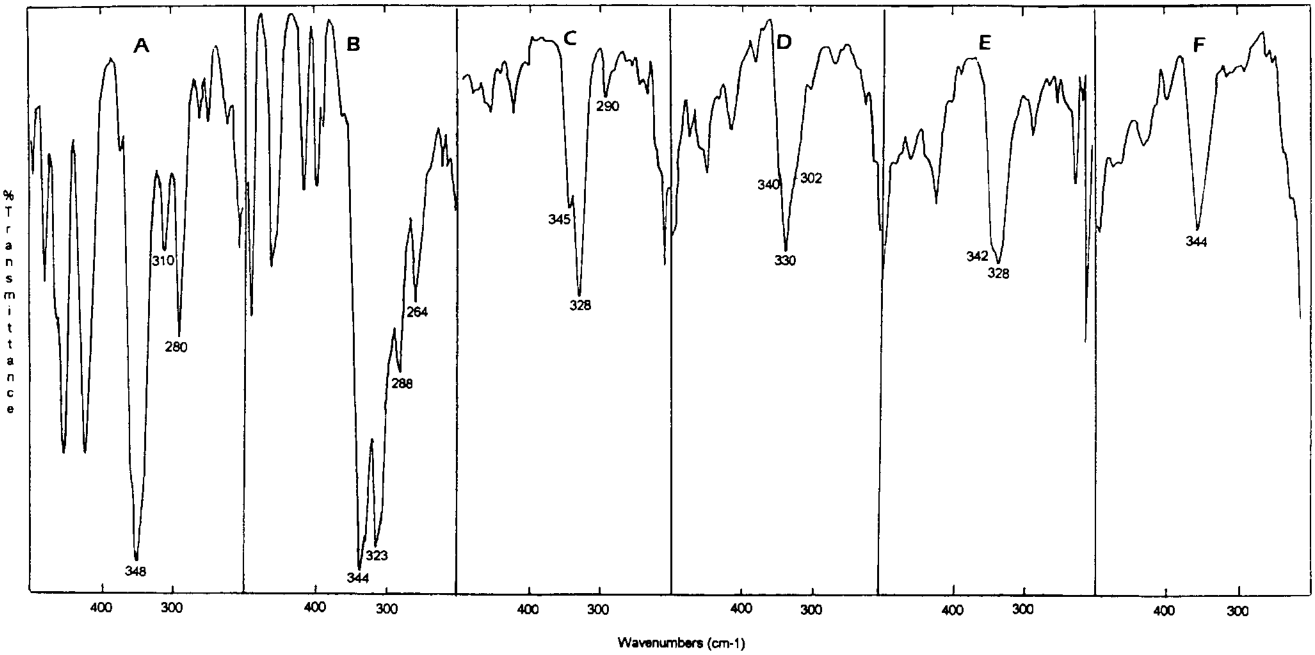

Fig. 2. ν(SnCl) region of the IR spectrum in Nujol mulls displayed by W–Sn complexes; A, $[(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3]$ (**1**); B, $[WCl(SnCl_3)(CO)_3(NCMe)_2]$ (**2**); C, $[WCl(SnCl_3)(CO)_3(bipy)]$ (**5**); D, $[WCl(SnCl_3)(CO)_3(PPh_3)_2]$ (**3**), contaminated $[WCl(SnCl_3)(CO)_2(PPh_3)_2]$; *E*, $[WCl(SnCl_3)(CO)_2(PPh_3)_2]$; F, $[WCl(SnCl_3)(CO)_3(dppe)]$ (**4**).

The IR pattern in the carbonyl stretching region of these complexes is very similar to that obtained with the triphenylphosphine (Table 1 and Fig. 1).

2.3. IR spectra of [$WCl(SnCl_3)(CO)_3L_2$]

The typical IR spectra of the compounds [$WCl(SnCl_3)(CO)_3L_2$] in the carbonyl stretching region are shown in Fig. 1, with the band positions for all the compounds given in Table 1. Three bands are observed in some cases and in others only two but in the latter case the lower band is broad or has a shoulder. The supernumerary bands present as the shoulder on the higher energy band, e.g. in the IR spectrum of [$WCl(SnCl_3)(CO)_3(PPh_3)_2$], are best accounted for on the assumption that more than one isomer is present for this compound. Another reason is the decarbonylation process of tricarbonyl species leads to the dicarbonyl form with the ν(CO) band at lower energy (see Figs. 1D and 1E).

The pattern of the ν(CO) bands, one sharp, and the other broad and split, suggests a *fac* distribution of the three carbonyl groups [23] in **2–5**.

The IR spectra of **2–5** are in good agreement with molecular structure observed for this type of complex [8a,b,24,25]. Most of the seven-coordinated complexes of $Mo^{(II)}$ and $W^{(II)}$ containing the fragment "$M(CO)_3(SnCl_{3-n}R_n)$" have been described in the literature as having a capped octahedron of ligands around the metal. The $SnCl_{3-n}R_n$ group occupies the capping position, and the unique chlorine atom a position in the capped face. In this geometry three CO groups can be mutually *cis* as is schematically drawn in Fig. 3.

In the IR spectra of **1–5** the fundamentals of the $SnCl_3^-$ group are observed at about 340 and 320 cm^{-1} (Fig. 2 and Table 1) and are attributed to the asymmetrical and symmetrical stretching respectively of the Sn–Cl bond.

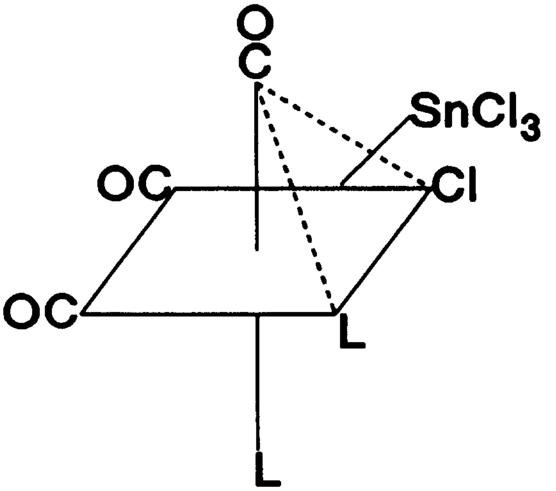

Fig. 3. Proposed molecular geometry of [$WCl(SnCl_3)(CO)_3L_2$] (**2–5**). The $SnCl_3$ group capping a face of the octahedron is occupied by three different ligands. Three CO groups are mutually *cis*.

3. Experimental details

All reactions were carried out under nitrogen by the standard Schlenk line techniques. The solvents applied were dried, distilled and deoxygenated.

$W(CO)_6$, $SnCl_4$, PPh_3, 1,2-bis(diphenylphosphino) ethane (dppe) and 2,2′-bipyridyl (bipy) were commercial products, used without purification. [$W(CO)_4(NCMe)_2$] was prepared according to the literature procedure [26].

The photochemical reactions were carried out in a glass reactor with a quartz window. An HBO 200 mercury lamp was used as the light source. IR spectra were recorded with Specord 80 IR and Nicolet Fourier transform IR model 400 instruments. ^1H and ^{13}C NMR spectra were recorded on an AMX 300 Brucker spectrometer. The UV-visible absorption spectra were recorded with a Hewlett–Packard 8452A spectrophotometer. The molecular weight of **3** was determine by osmometry using toluene as the solvent.

3.1. [$(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3$] (1)

[$(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3$] (**1**) was prepared by the photochemical reaction of $W(CO)_6$ and $SnCl_4$ according to the literature method [18].

3.2. [$WCl(SnCl_3)(CO)_3(NCMe)_2$] (2)

Compound **2** can be prepared as a mixture with [$WCl_2(CO)_3(NCMe)_2$] in the reaction of **1** in acetonitrile (method a) and in the reaction of [$W(CO)_4(NCMe)_2$] with $SnCl_4$ (method b).

3.2.1. Method a

[$(CO)_4W(\mu\text{-}Cl)_3W(SnCl_3)(CO)_3$] (**1**) (0.1 g, 0.11 mmol) dissolves in CH_3CN (20 cm^3) with CO evolution and formation of a deep-yellow clear solution. Evaporation of solvent in vacuo gives a mixture of **2** and [$WCl_2(CO)_3(NCMe)_2$].

IR(KBr): ν(CO) 2027vs, 1933sh and 1900vs; (Nujol): ν(CO) 2033vs, 1940s and 1905vs; ν(CN) 2326 and 2298; ν(MCl) 344s, 323 and 288w cm^{-1}. ^1H NMR (CD_2Cl_2); δ 2.59, 2.54 ppm (NC*Me*). ^{13}C NMR: δ 211.82, 208.00, 207.80, 201.77, 200.86 and 196.75 (CO), 126.86 and 126.10 (*N*CMe), 3.18 and 2.49 (NC*Me*) ppm. Satisfactory analyses could not be obtained for **2** owing to its instability and difficulty in separation from [$WCl_2(CO)_3(NCMe)_2$].

3.2.2. Method b

A mixture of [W(CO)$_4$(NCMe)$_2$] and SnCl$_4$ in chlorobenzene was stirred under a stream of nitrogen. The amorphous orange precipitate that formed was filtered off, washed with hexane and dried in vacuo to give pure **2**.

3.3. [WCl(SnCl$_3$)(CO)$_3$(PPh$_3$)$_2$] (3)

3.3.1. Method a

Tin(IV) chloride (0.7 g, 2.8 mmol) was added under nitrogen to a stirred solution of W(CO)$_6$ (1.0 g, 2.8 mmol) and PPh$_3$ (1.6 g, 6.1 mmol) in cyclohexane (100 cm^3). The starting white [SnCl$_4$(PPh$_3$)$_2$] formed after irradiation for a few minutes dissolved and there was very quickly precipitation of an orange–yellow amorphous solid. The IR spectrum of the reaction mixture witnessed a decay in the absorption due to W(CO)$_6$ with the accompanying appearance and growth of new absorption at 2032s, 1940vs and 1862s cm^{-1}. After irradiation for 2 h the precipitate was removed by filtration, washed with three 10 cm^3 portions of cyclohexane and dried in vacuo. The crude product was a mixture of [WCl$_2$(CO)$_3$(PPh$_3$)$_2$] (ν(CO) = 2014s, 1936vs and 1894s) and [WCl(SnCl$_3$)(CO)$_3$(PPh$_3$)$_2$] (**3**), from which **3** was separated by extraction using toluene. The addition of heptane to toluene solution gave **3** as an orange solid (yield, 1.6 g (75%)).

Anal. Found (molecular weight, 1030); C, 44.40; H. 3.00; Cl, 13.55; P, 6.04; Sn, 11.55. $C_{39}H_{30}Cl_4O_3P_2SnW$ Calc. (molecular weight, 1052.93); C. 44.48; H, 2.87; Cl, 13.47; P, 5.88; Sn, 11.27%.

IR(KBr): ν(CO) 2044sh, 2030s, 1954sh, 1928vs and 1864m; (Nujol): ν(MCl) 340sh, 330m, 302m cm^{-1}.

Absorption maximum (toluene): λ, nm (ε, M^{-1} cm^{-1}): 386 (1660), 330 (2760).

3.3.2. Method b

Compound **1** (0.5 g, 0.5 mmol) was dissolved in toluene (50 cm^3) with continuous stirring under a stream of dry nitrogen. To this was added PPh$_3$ (0.6 g, 2.23 mmol). The colour of the solution immediately changed to bright orange. The mixture was left stirring for 1 h. The IR spectrum of the reaction mixture witnessed a decay of the absorption at 2100 cm^{-1} due to **1** with the accompanying appearance of new absorption at 2030 and 1928 cm^{-1}. An amorphous yellow, precipitate settled. This was filtered off, washed twice with 20 cm^3 toluene and dried in vacuo to give 0.24 g (0.28 mmol) of analytically pure (WCl$_2$ (CO)$_3$(PPh$_3$)$_2$). The solvent was evaporated in vacuo to give an orange oil, which was recrystallized from toluene–heptane to produce an orange microcrystalline solid of **3** (yield, 0.3 g (50%)).

Reaction in CH$_2$Cl$_2$ gives a mixture of **3** and [WCl(SnCl$_3$)(CO)$_2$(PPh$_3$)$_2$] with ν(CO) 1932s and 1858vs cm^{-1} (Fig. 1E).

3.4. [WCl(SnCl$_3$)(CO)$_3$(dppe)] (4)

The procedure was similar to that used for **3** (method a) starting from W(CO)$_6$ (1.0 g, 2.8 mmol), dppe (1.1 g, 2.8 mmol) and SnCl$_4$ (0.7 g, 2.8 mmol) in cyclohexane (100 cm^3). At the beginning, white [SnCl$_4$(dppe)] is formed but after irradiation for a few minutes it disappeared and there was very quickly precipitation of yellow amorphous solid. Extraction using toluene and recrystallization from toluene–heptane gives a yellow solid of **4** (yield, 2.3 g (87%)).

Anal. Found: C, 37.40; H, 2.80; P, 7.05; Sn, 11.50. $C_{29}H_{24}Cl_4O_3P_2SnW$ Calc. (molecular weight, 926.8): C, 37.58; H, 2.61; P, 6.68; Sn, 12.81%.

IR(KBr): ν(CO) 2035s, 1933vs; (Nujol): 2035s, 1965sh, 1933vs and 1895sh; ν(MCl) 344cm^{-1}.

3.5. [WCl(SnCl$_3$)(bipy)(CO)$_3$] (5)

3.5.1. Method a

Compound **5** was prepared as described above for **3** (method a), starting from W(CO)$_6$ (1.0 g, 2.8 mmol), bipy (0.4 g, 2.8 mmol) and SnCl$_4$ (0.7 g, 2.8 mmol) in cyclohexane (100 cm^3). The crude product, besides showing bands typical for **5**, shows the additional absorption bands at 2040vs, 1954vs and 1906s cm^{-1} which could be attributed to a [WCl$_2$(bipy)(CO)$_3$] [17]. Compound **5** was washed out from the crude product by toluene. Addition of heptane to the filtrate gave **5** as an orange solid with a good yield (1.0 g (52%)).

Anal. Found: C, 22.4; H, 1.05; N, 4.00; Cl, 20.95; Sn, 16.84. $C_{13}H_8Cl_4N_2O_3SnW$ Calc. (molecular weight, 684.6): C, 22.80; H, 1.18; N, 4.09; Cl, 20.71; Sn, 17.34%.

IR(KBr): ν(CO) 2020vs, 1950sh and 1925vs; (Nujol): ν(CO) 2020vs, 1957s and 1925vs; ν(MCl) 345w, 328m and 290vw cm^{-1}.

3.5.2. Method b

To a stirred solution of **1** (0.5 g, 0.56 mmol) in CH$_2$Cl (20 cm^{-3}) under a stream of dry nitrogen was added 2,2′-bipyridyl (0.2 g, 1.12 mmol). Filtration of [WCl$_2$(bipy)(CO)$_3$], followed by the removal of solvent, in vacuo gave the orange complex **5**.

4. Conclusions

An oxidative–addition reaction of SnCl$_4$ to W(CO)$_6$ is utilized to convert the W$^{(0)}$ to the W$^{(II)}$ compound. This is followed by a substitution step using of N- or P-donor ligands to generate seven-coordinated compounds [WCl(SnCl$_3$)(CO)$_3$L$_2$] and [WCl$_2$(CO)$_3$L$_2$].

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References

[1] D. Borowczak, T. Szymańska-Buzar and J.J. Ziółkowski, *J. Mol. Catal., 27* (1984) 355.
[2] T. Szymańska-Buzar and J.J. Ziółkowski, *J. Mol. Catal., 43* (1987) 161.
[3] T. Szymańska-Buzar, *J. Mol. Catal., 68* (1991) 177.
[4] T. Szymańska-Buzar, *J. Mol. Catal., 93* (1994) 137.
[5] T. Szymańska-Buzar, to be published.
[6] R. Kumer and W.A.G. Graham, *Inorg. Chem., 7* (1968) 310.
[7] K. Edgar, B.F. Johnson, J. Lewis and S.B. Wild, *J. Chem. Soc. A,* (1968) 2851.
[8] (a) M. Elder and D. Hall, *Inorg. Chem., 8* (1969) 1268; (b) M. Elder and D. Hall, *Inorg. Chem., 8* (1969) 1273.
[9] M. Iglesias, A. Lorente, C. del Pino and A. Santos, *J. Organomet. Chem., 256* (1983) 75.
[10] M. Panizo and M. Cano, *J. Organomet. Chem., 287* (1985) 221.
[11] A. Bell and R. Walton, *J. Organomet. Chem., 290* (1985) 341.
[12] P.K. Baker and A. Bury, *J. Organomet. Chem., 359* (1989) 189.
[13] P.K. Baker and A.J. Quinlan, *Inorg. Chim. Acta, 162* (1989) 1.
[14] D. Miguel, J.A. Perez-Martinez and V. Riera, *J. Organomet. Chem., 455* (1993) 121.
[15] M. Cano, M. Panizo, J.A. Campo, J. Tornero and N. Menèndez, *J. Organomet. Chem., 463* (1993) 121.
[16] T. Szymańska-Buzar, *Inorg. Chim. Acta, 145* (1988) 231.
[17] T. Szymańska-Buzar, *J. Organomet. Chem., 375* (1989) 85.
[18] T. Szymańska-Buzar and T. Glowiak, *J. Organomet. Chem., 489* (1995) 207.
[19] (a) R. Colton and I.B. Tomkins, *Aust. J. Chem., 19* (1966) 1143; (b) R. Colton and I.B. Tomkins, *Aust. J. Chem., 19* (1966) 1519; (c) R. Colton and I.B. Tomkins, *Aust. J. Chem., 20* (1967) 13.
[20] M.W. Anker, R. Colton, and I.B. Tomkins, *Aust. J. Chem., 20* (1967) 9.
[21] A.D. Westland and N. Muriithi, *Inorg. Chem., 12* (1973) 2356.
[22] Y. Kawano, Y. Hasse and O. Sala, *J. Mol. Struct., 30* (1976) 45.
[23] E.E. Isaacs and W.A.G. Graham, *Inorg. Chem., 14* (1975) 2560).
[24] R.A. Anderson and F.W.B. Einstein, *Acta Crystallogr., Sect., B, 32* (1976) 966.
[25] F.W.B. Einstein and J.S. Field, *J. Chem. Soc., Dalton Trans.,* (1975) 1628.
[26] G.R. Dobson, M.F. Amr El Sayed, I.W. Stolz and R.K. Sheline, *Inorg. Chem., 1* (1961) 526.