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Journal of Organometallic Chemistry 648 (2002) 93–98

Journal of Organometallic Chemistry

www.elsevier.com/locate/jorganchem

Raman and infrared spectra and pressure tuning Raman and infrared spectra of multinuclear metal carbyne complexes☆

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Received 4 July 2001; received in revised form 16 August 2001; accepted 8 October 2001

Abstract

The Raman and infrared (IR) spectra of five multinuclear metal carbyne complexes of the type $[Cl(CO)_2(L)W{\equiv}CC_6H_4–(C{\equiv}CC_6H_4)_n–N{\equiv}C–]_m\boldsymbol{M}$ ($\boldsymbol{M} = PdI_2$ or $ReCl(CO)_3$) **I**–**V**, and the pressure tuning Raman and IR spectra of one representative complex **III**, were studied. The high pressure study on complex **III** reveals the presence of a pressure-induced phase transition near 20 kbar. For $\nu(W{\equiv}C)$, a high-pressure sensitivity ($d\nu/dp = 0.75$ cm^{-1} kbar^{-1}) was observed in the low-pressure phase, but a very low-pressure sensitivity ($d\nu/dp = 0.08$ cm^{-1} kbar^{-1}) in the high-pressure phase. The substitution of two CO ligands in $X(CO)_4W{\equiv}CPh(R)$ by amine (TMEDA) or phosphine (DPPE) donor ligands causes a slight decrease of $\nu(W{\equiv}C)$ and a strong decrease of $\nu(C{\equiv}O)$. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Raman; Infrared; High pressure; Carbyne; Isocyanide; Tungsten

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1. Introduction

Fischer's discovery of transition metal carbyne complexes initiated a new research field [1,2]. Many compounds containing a metal–carbon triple bond were synthesized and their properties were extensively studied [3–6]. Recently, several complexes of the novel type $[Cl(CO)_2(L)W{\equiv}CC_6H_4–(C{\equiv}CC_6H_4)_n–N{\equiv}C–]_m\boldsymbol{M}$ ($\boldsymbol{M} = PdI_2$ or $ReCl(CO)_3$) were synthesized [7,8]. On account of their special combination of electronic and photophysical molecular properties, these complexes are promising as building blocks for the design of molecular devices and materials [9]. Here we present a study of the vibrational spectra of five tungsten carbyne complexes of this type (**I**, **II**, **III**, **IV** and **V**), and a pressure tuning Raman and IR spectroscopic study for complex **III**. A pressure-induced phase transition was discovered.

$[Cl(CO)_2(TMEDA)W{\equiv}CC_6H_4NC]_2PdI_2$ (**I**);
$[Cl(CO)_2(TMEDA)W{\equiv}CC_6H_4NC]_2ReCl(CO)_3$ (**II**);
$[Cl(CO)_2(DPPE)W{\equiv}CC_6H_4NC]_2ReCl(CO)_3$ (**III**);
$[Cl(CO)_2(DPPE)W{\equiv}CC_6H_4C{\equiv}CC_6H_4NC]_2PdI_2$ (**IV**);
$[Cl(CO)_2(DPPE)W{\equiv}CC_6H_4C{\equiv}CC_6H_4NC]_2ReCl(CO)_3$ (**V**);
TMEDA = tetramethylethylenediamine, $(CH_3)_2NCH_2CH_2N(CH_3)_2$;
DPPE = bis(diphenylphosphine)ethane, $(Ph)_2PCH_2CH_2P(Ph)_2$.

2. Experimental

The samples are dark or light orange colored solids. They are stable in air for extended periods. The micro-IR spectra of crystals were measured on a Nicolet Magna 750 FTIR spectrometer equipped with a Nic-Plan microscope (4000–650 cm^{-1}, MCT-detector, 4 cm^{-1} resolution and 500 scans). The Raman spectra of crystals were measured on a Nicolet 910 FT-Raman spectrometer, using a $Nd{:}YVO_4$ solid laser (1064 nm and 600 mw power) as the excitation source. The

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☆ Present, in part, at The XVIIth International Conference on Raman Spectroscopy, August 20–25, 2000, Beijing, China

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PII: S0022-328X(01)01395-X

spectral range is 3400–100 cm^{-1}. The resolution is 4 cm^{-1} with 500 scans.

The IR spectra of crystals were recorded at different high pressures (the highest pressure is about 50 kbar) with the aid of a diamond-anvil cell (DAC, High Pressure Diamond Optics, Tucson, AZ, USA) [10]. The sample and calibrant were placed in a 300 μm diameter hole drilled in the center of a 270 μm thick stainless-steel gasket (7 × 7 mm) located between the parallel faces of the pair of diamonds in the DAC. The DAC was mounted under the microscope of a Bruker IFS-48 spectrometer equipped with a MCT-detector. The pressure was calibrated with $NaNO_2$ (1279 cm^{-1} at ambient pressure) [11]. The spectra were recorded at 4 cm^{-1} resolution (1000 scans) over a wavenumber range in 4000–600 cm^{-1}. The pressure tuning Raman spectra of crystals were measured with the aid of a DAC mounted on a Bruker IFS-88 spectrometer equipped with a FRA 106 FT-Raman module connected to Nikon Optiphot-II optical microscope by two 1.00-m optical fibers. The samples were excited with a Nd:YAG laser emitting at 1064 nm with 200 MW power. The spectra were recorded (3600–50 cm^{-1}, resolution 2.6 cm^{-1}, 4000–5000 scans) using liquid N_2-cooled Ge detector at the different pressure. The pressure-induced shift of the Raman-active t_{2g} phonon mode of the diamond window of the DAC, located at 1332.5 cm^{-1} at ambient pressure, was used as an in situ calibrant for the FT-Raman micro-spectroscopic measurements at high external pressure [12,13]. The band positions were determined using a curve fitting program (from BRUKER OS/2 software) since the S/N (signal to noise) ratios of the spectra are very low at high external pressure.

3. Results and discussion

3.1. Raman and IR spectra of complexes **I**, **II**, **III**, **IV** and **V**

Selected spectroscopic data are listed in Table 1. Assignments are given only for the main vibrational modes [14–16].

3.1.1. $\nu(W{\equiv}C)$

Dao, Fischer, and coworkers studied the Raman and IR spectra of a series of metal carbyne complexes of the type $X(CO)_4W{\equiv}CR$ and observed characteristic bands in the region of 1300–1400 cm^{-1}. These bands were assigned to the $\nu(M{\equiv}C)$ stretches. Generally, the M≡C bonds give rise to strong Raman scattering, but weak IR absorptions. The complexes $Br(CO)_4W{\equiv}CPh$ and $I(CO)_4W{\equiv}CPh$ [17] exhibit Raman bands at 1373 and 1357 (vs), 1369 and 1345 (vs), cm^{-1}, respectively. For the ethylidyne complexes $X(CO)_4W{\equiv}CCH_3$, the W≡C stretches (X = Cl: 1361 and 1353 cm^{-1}, X=Br: 1355 and 1347 cm^{-1}, X = I: 1358 cm^{-1}) [18] were found at lower frequencies than for the benzylidyne complexes $X(CO)_4W{\equiv}CPh$.

These studies showed that the M≡C stretching modes in methyl- and phenylcarbyne metal complexes are different and strongly coupled with other vibrations, in particular the MC–R (R = CH_3 and Ph) stretching vibrations.

The dinuclear complex $Br(CO)_4W{\equiv}CC_6H_4C{\equiv}W(CO)_4Br$ [19] may be viewed as a model for a phenylcarbyne tungsten complex bearing a substituent at the 4-position of the phenyl ring. It features several Raman bands at 1386, 1379, 1372 (vs), 1352 (s), and 1332 (m)

Table 1
The part data of Raman and IR bands of complexes I, II, III, IV and V (cm^{-1})

I		II		III		IV		V		Assignments
Raman	IR	Raman	IR	Raman	IR	Raman	IR	Raman	IR	
						2274w,sh		2272w		$\nu(C{\equiv}C)$
						2217vs	2214w	2212vs	2211w	
2213vs	2194s	2182vs	2184m	2182s	2180m	2207vs	2181m	2183vs	2180m	$\nu(C{\equiv}N)$
		2140vs	2152m	2156s	2141s	2166m		2155s		
						2160m		2148vs	2141m	
		2038m	2039s	2030w	2032s			2035m	2035s	$\nu(C{\equiv}O)$
		1998w	1991s	2001w	1999s	2009w	2004s	2000w	1998s	
1985w	1986vs	1978w	1979s		1989s,sh					
		1968w	1973s							
		1926m	1925s	1936w	1925s	1936w	1925s	1934m	1931s	
							1912s			
		1896w	1896s							
1884w	1889vs	1881w	1881s							
1361vs	1359vw	1362vs	1356vw	1353vs	1363vw	1364vs	1362vw	1358vs	1359vw	$\nu(W{\equiv}C)$

cm^{-1}. For the deuterated analogue $\text{Br(CO)}_4\text{W}{\equiv}\text{CC}_6\text{D}_4\text{-C}{\equiv}\text{W(CO)}_4\text{Br}$, the number of bands is reduced to two at 1377 (vs) and 1345 (w) cm^{-1}. These two bands are probably representative of the vibrations associated with the W≡CC fragment. Moreover, they are very similar to the ν(W≡C) bands reported for the unsubstituted benzylidyne complex $\text{Br(CO)}_4\text{W}{\equiv}\text{CC}_6\text{H}_5$ [17].

The combined literature data [20–22] show that the frequencies of the vibrations most closely associated with the M≡C stretch depend strongly on the nature of the substituent on the carbyne carbon atom. The data also show that the M≡C stretches are very little affected by a change of the ligand in the position trans to the metal–carbon triple bond or by a change of the substituent on the phenyl ring of a phenylcarbyne ligand.

These general conclusions are confirmed by our results. The W≡C stretching frequencies of complexes **I**–**V** are found at 1361, 1362, 1353, 1364 and 1358 (vs) cm^{-1}, respectively (Table 1). This is within the range established for tungsten benzylidyne complexes. Since the major difference between the complexes **I**–**V** and the phenylcarbyne complexes studied by Dao and Fischer [17,19] is the presence of the donor ligands TMEDA and DPPE, it may be concluded that the ν(M≡C) stretch in metal carbyne complexes is little influenced by the nature of the ligands in the position cis to the metal–carbon triple bond. This finding is in agreement with theoretical calculations which indicate that the M≡C π orbitals are energetically well separated from the σ as well as π bonding orbitals of the four equatorial ligands [21,23,24]. Nevertheless, a careful comparison of the data for complexes **I**–**V** with those reported for the most closely related tetracarbonyltungsten carbyne complexes, $\text{Br(CO)}_4\text{W}{\equiv}\text{CC}_6\text{H}_5$ and $\text{Br(CO)}_4\text{W}{\equiv}\text{CC}_6\text{H}_4\text{C}{\equiv}\text{W(CO)}_4\text{Br}$, suggests that the substitution of two carbonyl ligands by the donor ligands TMEDA and DPPE causes a slight lowering of the W≡C stretching frequency by about 10–20 cm^{-1}. This influence of the donor ligands is possibly connected to the strengthening of the π back-bonding from the metal center to the π* orbital of remaining carbonyl ligands. It will cause the metal–carbon triple bond to weaken slightly. The fact that the ν(W≡C) stretching frequencies of complexes **III** and **V** are nearly identical confirms the previous finding that the modification of the substituent in the *para*-position of the phenyl ring (NC vs. CC) has practically no influence on the vibration of the metal–carbon triple bond [19].

3.1.2. ν(C≡N) and ν(C≡C)

The intensities of the Raman bands of ν(C≡N) and ν(C≡C) are much stronger than those of the IR bands, as is usually found (Table 1) [15,16]. For example, for complex **IV**, the Raman bands of ν(C≡N) at 2207 cm^{-1} and ν(C≡C) at 2217 cm^{-1} are very strong, but the IR bands of ν(C≡N) at 2181 cm^{-1} and ν(C≡C) at 2214 cm^{-1} have only medium or weak intensities (Table 1).

The Raman ν(C≡N) frequencies of **I** and **IV** are higher [2213 cm^{-1} (**I**) and 2207 cm^{-1} (**IV**)] than those of **II**, **III** and **V** [2182 cm^{-1} (**II**), 2182 cm^{-1} (**III**) and 2183 cm^{-1} (**V**)]. These data show that the PdI_2 fragment is more strongly electron withdrawing than the ReCl(CO)_3 group [14]. There are more ν(C≡N) bands (Raman and IR) for the complexes **II**, **III** and **V** than for complexes **I** and **IV** (Table 1), in agreement with the expected *trans*-arrangement of the two isocyanide groups at the PdI_2 fragment and the *cis*-arrangement at the ReCl(CO)_3 fragment [25,26].

Although there are different central metal complex fragments in complexes **IV** and **V** (**IV**: PdI_2 and **V**: ReCl(CO)_3), we found that the data for ν(C≡C) (number of bands and frequencies, Table 1) are almost the same for **IV** and **V**. These results show that the electronic environment of the central C≡C bond is little affected by the nature of the central metal centers.

3.1.3. ν(C≡O)

The behavior of the C≡O stretches differs from that of the C≡N and C≡C stretches. They give rise to strong bands in the IR spectra, but relatively weak bands in the Raman spectra (Table 1). Here we discuss the IR spectra. For solid W(CO)_6, the IR band of maximum intensity is near 1970 cm^{-1} [27]. The complexes $\text{X(CO)}_4\text{W}{\equiv}\text{CC}_6\text{H}_5$ (X = halogen) feature a maximum intensity IR band near 2040 cm^{-1} (in hexane) [28]. After substitution of two carbonyl ligands, two strong IR absorptions are observed, e.g. at 1984 and 1897 cm^{-1} for $\text{Br(CO)}_2\text{(pyridine)}_2\text{W}{\equiv}\text{CC}_6\text{H}_5$ and at 2004 and 1934 cm^{-1} for $\text{Br(CO)}_2\text{(PPh}_3)_2\text{W}{\equiv}\text{CC}_6\text{H}_5$ [29]. These two complexes, containing two nitrogen and two phosphorus donor ligands, serve as good models for the metal carbyne complex fragments of complexes **I** and **II**, and **III**–**V**, respectively. As in these complexes, the presence of the amine (TMEDA) and phosphine (DPPE) donor ligands in complexes **I**–**V** increases π back-bonding and is responsible for the relatively low frequencies of the two carbonyl ligands. In complexes **I** and **IV**, the central metal complex fragment does not bear carbonyl ligands. Therefore, only the absorptions for the W(CO)_2 fragment are observed. The IR spectra of solid **II** feature more IR bands than expected. We assign the bands at 2039, 1991 and 1925 cm^{-1} to the ReCl(CO)_3 fragment and the bands at 1979, 1973, 1881 and 1896 cm^{-1} mainly to the $\text{Cl(TMEDA)(CO)}_2\text{W}{\equiv}\text{C}$ group [30,31]. A possible reason for the appearance of multiple absorptions may be the solid-state packing of the molecules. The IR spectra of **III** and **V** feature fewer carbonyl bands. The absorptions near 2030 cm^{-1} may be assigned to the ReCl(CO)_3 fragment and the bands near 2000 cm^{-1} to the $\text{Cl(DPPE)(CO)}_2\text{W}{\equiv}\text{C}$ group. The absorptions near 1930 cm^{-1} may be overlapping bands of both types of metal complex fragment.

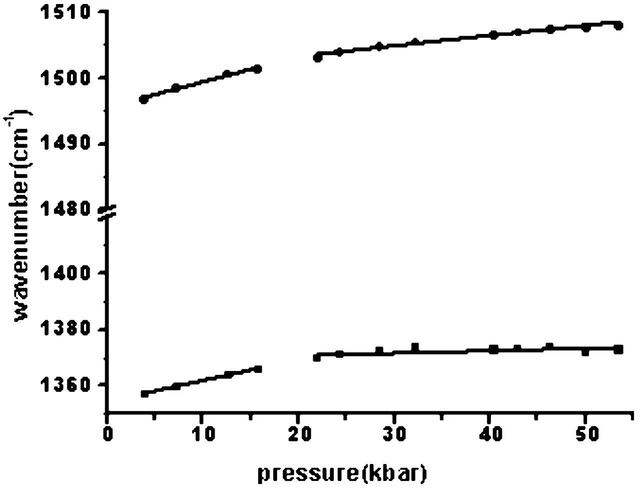

Fig. 1. Pressure dependences of selected Raman bands of complex III.

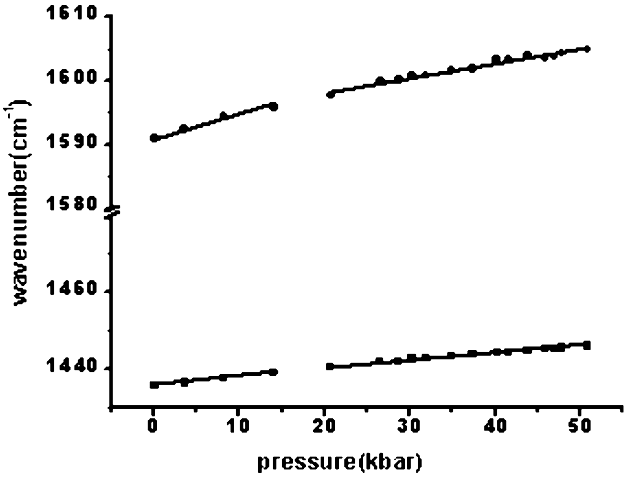

Fig. 2. Pressure dependences of selected IR bands of complex III.

3.2. Spectra of complex ***III*** at high external pressure

The plots of wavenumber shifts observed with change in pressure for selected bands are shown in Fig. 1 (Raman) and Fig. 2. (IR).

1. The discontinuities occurring in the pressure to wavenumber plots indicate the presence of a phase transition near 20 kbar (Figs. 1 and 2). The values for the pressure sensitivity, dv/dp in the two phases are given in Tables 2 and 3. Assignments are given only for main vibrational modes [15,16].
2. The pressure sensitivity of the Raman v(W≡C) mode has a high value ($dv/dp = 0.75$ cm^{-1} kbar^{-1}) in the low-pressure phase and a very low value ($dv/dp =$ 0.08 cm^{-1} kbar^{-1}) in the high-pressure phase (Table 2). The dramatic change of the pressure dependence over a narrow pressure region is attributed to a phase transition. The origin of the change from a high to a low pressure sensitivity is less certain. Below 20 kbar, the normal compression shortening of the W≡C bond will increase the wavenumber of v(W≡C), explaining the high dv/dp value. Above 20 kbar, other effects apparently work in the opposite direction. If, as concluded above, the introduction of donor ligands at the metal center causes a slight decrease of v(W≡C), then compression of the substituted carbyne metal complex may further weaken the metal–carbon triple bond. At high-pressure, this effect would be amplified by the compression strengthening π back-bonding from W to the π^* of carbonyl ligands [32,33]. These combined influences would thus lower the pressure sensitivity of v(W≡C).
3. The pressure sensitivities of the other modes from substituted benzene have higher values in the low-pressure phase than in the high-pressure phase as usually [10,34].
    - 3.1. The dv/dp values for the ring stretching modes of the substituted benzene fragment are normal [IR: 1591 cm^{-1} (0.36 and 0.22 cm^{-1} kbar^{-1}); Raman: 1495 (0.39 and 0.16 cm^{-1} kbar^{-1}) and 1585 cm^{-1} (0.49 and 0.19 cm^{-1} kbar^{-1})]. But there is a low dv/dp value for the absorption at 1436 cm^{-1} (IR: 0.23 and 0.18 cm^{-1} kbar^{-1}) which changed a little in different phase (Tables 2 and 3).

Table 2
Pressure dependences of selected Raman bands of complex III [a]

Low-pressure phase		High-pressure phase		Assignments
v (cm^{-1})	dv/dp (cm^{-1}/kbar)	v (cm^{-1})	dv/dp (cm^{-1}/kbar)	
1001	0.32	1004	0.21	CH in plane bending of substituted benzene
1165	0.21	1167	0.14	CH in plane bending of substituted benzene
1354	0.75	1369	0.08	W≡C stretching
1495	0.39	1500	0.16	ring stretching of substituted benzene
1585	0.49	1591	0.19	ring stretching of substituted benzene
3060	1.06	3071	0.51	CH stretching of substituted benzene

[a] These wavenumbers are the intercepts of the plots of pressure against wavenumber at low-pressure phase or high-pressure phase. There are a 1–3 cm^{-1} differences to the frequencies measured at ambient pressure for some modes at the low-pressure phase.

Table 3
Pressure dependences of selected IR bands of complex III [a]

Low-pressure phase		High-pressure phase		Assignments
ν (cm^{-1})	dν/dp (cm^{-1}/kbar)	ν (cm^{-1})	dν/dp (cm^{-1}/kbar)	
741	0.30	746	0.17	CH wagging of monosubstituted benzene
1029	0.33	1030	0.21	CH in plane bending of substituted benzene
1100	0.44	1102	0.31	CH in plane bending of substituted benzene
1160	0.24	1161	0.19	CH in plane bending of substituted benzene
1188	0.26	1189	0.13	CH in plane bending of substituted benzene
1436	0.23	1437	0.18	ring stretching of substituted benzene and CH_2 deformation
1591	0.36	1594	0.22	ring stretching of substituted benzene

[a] These wavenumbers are the intercepts of the plots of pressure against wavenumber at low-pressure phase or high-pressure phase. There are a 1–3 cm^{-1} differences to the frequencies measured at ambient pressure for some modes at the low-pressure phase.

3.2. The CH in plane bending modes of the substituted benzene have relatively low dν/dp values [IR: 1029 (0.33 and 0.21 cm^{-1} kbar^{-1}), 1160 (0.24 and 0.19 cm^{-1} kbar^{-1}) and 1188 cm^{-1} (0.26 and 0.13 cm^{-1} kbar^{-1}); Raman: 1001 (0.32 and 0.21 cm^{-1} kbar^{-1}) and 1165 cm^{-1} (0.21 and 0.14 cm^{-1} kbar^{-1})]. But the IR band at 1100 cm^{-1} has a high value (0.44 and 0.31 cm^{-1} kbar^{-1}).

3.3. For the CH stretching mode of the substituted benzene, there is the highest dν/dp value [Raman 3060 cm^{-1} (1.06 cm^{-1} kbar^{-1} in the low-pressure phase and 0.51 cm^{-1} kbar^{-1} in the high-pressure phase)] as is usually observed (Table 2). The CH wagging mode of the substituted benzene at 744 cm^{-1} has a relatively high dν/dp value, 0.30 and 0.17 cm^{-1} kbar^{-1}, respectively (Table 3).

4. Conclusions

1. There is a pressure-induced phase transition near 20 kbar with increasing pressure on tungsten carbyne complex **III**.
2. There are two effects on ν(W≡C) of complex **III** with increasing pressure: a normal increase of the ν(W≡C) wavenumber due to the pressure-induced shortening of the W≡C bond, and an opposing decrease of ν(W≡C), which may be mainly caused by the compression strengthening W–CO π back-bonding from M to the π*orbitals of carbonyl ligands due to the compression of M–CO and M–L.
3. The substitution of two CO ligands in $X(CO)_4W{\equiv}CPh(R)$ by amine (TMEDA) or phosphine (DPPE) donor ligands causes a slight decrease of ν(W≡C) and a strong decrease of ν(C≡O).

Acknowledgements

The Milton Long Fund (McGill University) is acknowledged for its financial support of Z.X. (Peking University). The NSERC (Canada) is thanked for providing operating and equipment grants to I.S.B. A.M. acknowledges gratefully the Committee on Research and Conference Grants of The University of Hong Kong for supporting the research project.

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