

## ❖ Vibrational Spectra of Metal Carbonyls for Bonding and Structure Elucidation

Vibrational spectroscopy is one of the most important methods used for the characterization of metal carbonyls. This technique provides very useful information not only about the structural prototype of different metal carbonyl compositions but also rationalizes the nature of bonding in them. Now, it is quite a well-known fact that the C–O vibration for free carbonyl group (CO gas) is typically denoted as  $\nu_{\text{CO}}$ , and absorbs at  $2143 \text{ cm}^{-1}$ . However, this C–O absorption shifts downward (sometimes upward) to cover a very wide range of wavenumber as the carbonyl ligand gets attached to a metal center. This is obviously due to the fact that the energies of the  $\nu_{\text{CO}}$  band for the metal carbonyls directly correlate with the strength of the carbon-oxygen bond, and are inversely correlated with the strength of the  $\pi$ -backbonding between the metal and the carbon. In other words, the molecular orbital diagram of carbonyl group suggests that the highest occupied molecular orbital, used for  $\sigma$ -donation is weakly bonding; but the lowest unoccupied molecular orbital, used for accepting  $d$ -electron density from metal center is strongly antibonding; therefore, the  $\sigma$ -donation does not affect the CO bond order very much but the acceptance of electron density in  $\pi^*$  orbital decreases the bond order and consequently the bond strength in a significant way. This effect reduces the force constant of C–O bond, while the magnitude of force constant for M–C will be increased by this backbonding. As a result, the enhancement of backbonding shifts the metal-carbon and carbon-oxygen stretching to higher and lower values, respectively. The main features about the bonding and structure of metal carbonyls which can be obtained from the vibrational spectra of metal carbonyls are discussed below:

### ➤ 1. $\pi$ -Basicity of the Metal Centre

The  $\pi$ -basicity of the metal center (and thus the C–O stretching frequency) depends upon a lot of factors like the nature and magnitude of the charge on metal center, and the  $\pi$ -accepting tendency of ligands attached other than the carbonyl. A negative charge on the metal center, or ligands with greater  $\sigma$ -donation and weaker  $\pi$ -acceptor strength, are expected to decrease the CO stretching frequency; while an accumulation of positive charge on metal center, or ligands with weaker  $\sigma$ -donation stronger  $\pi$ -acceptor strength are bound to increase the CO stretching frequency. For example, in the isoelectronic series of  $\text{Ti} \rightarrow \text{Fe}$ , the hexacarbonyls show decreasing  $\pi$ -backbonding as one increases (makes more positive) the charge on the metal.

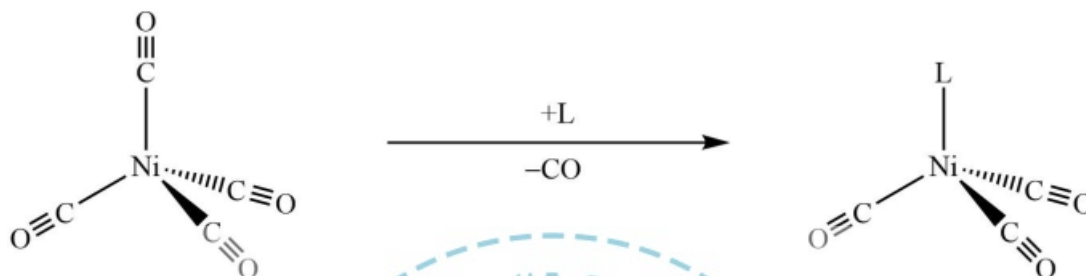
Compound	$[\text{Ti}(\text{CO})_6]^{2-}$	$[\text{V}(\text{CO})_6]^{1-}$	$[\text{Cr}(\text{CO})_6]$	$[\text{Mn}(\text{CO})_6]^{1+}$	$[\text{Fe}(\text{CO})_6]^{2+}$
$\nu_{\text{CO}} (\text{cm}^{-1})$	1748	1859	2000	2095	2204

Compound	$[\text{Hf}(\text{CO})_6]^{2-}$	$[\text{Ta}(\text{CO})_6]^{1-}$	$[\text{W}(\text{CO})_6]$	$[\text{Re}(\text{CO})_6]^{1+}$	$[\text{Os}(\text{CO})_6]^{2+}$
$\nu_{\text{CO}} (\text{cm}^{-1})$	1757	1850	1977	2085	2190

Hence,  $\pi$ -basic ligands increase  $\pi$ -electron density at the metal, and improved backbonding reduces  $\nu_{\text{CO}}$ .

### ➤ 2. Tolman Electronic Parameter

The electron-donating or withdrawing ability of a ligand is calculated in terms of the Tolman electronic parameter (TEP), named after the inventor, Chadwick A. Tolman. It is determined by measuring the frequency of the  $A_1$  vibrational mode of the carbonyl group in complex,  $Ni(CO)_3L$  by infrared spectroscopy, where L is the ligand being studied.  $Ni(CO)_3L$  was chosen as the model compound because such complexes can easily be synthesized from  $Ni(CO)_4$ .



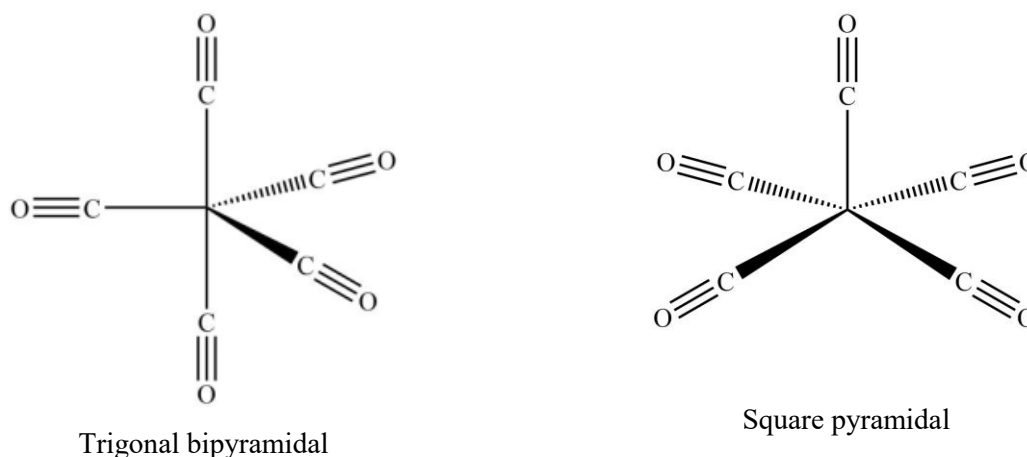
The CO band is pretty much unique and is rarely affected by other bands in the analyte's IR spectra. Moreover, owing to the small size of carbonyl ligand, steric factors do not muddle the analysis. The coordination of CO to a metal typically decreases  $\nu_{CO}$  value from  $2143\text{ cm}^{-1}$  (free CO). This can be rationalized by  $\pi$ -backbonding, resulting from the sidewise overlap of metal orbitals of  $\pi$ -symmetry with the empty anti-bonding  $\pi^*$  orbitals on CO ligand. This increases the strength of the metal-carbon bond, but also weakens the C–O bond. If some other ligand enhances the  $\pi$ -electrons-density on the metal, the CO bond strength decreases and so the  $\nu_{CO}$  value. On the other hand, if other ligands present compete with CO for  $\pi$ -backbonding,  $\nu_{CO}$  increases. TEPs for selected phosphines are given below.

L	P(t-bu) <sub>3</sub>	P(Me) <sub>3</sub>	P(Ph) <sub>3</sub>	P(OEt) <sub>3</sub>	PCl <sub>3</sub>	PF <sub>3</sub>
$\nu_{CO}\text{ (cm}^{-1}\text{)}$	2056.1	2064.1	2068.9	2076.3	2097.0	2110.8

The Tolman electronic parameter has been widely used to characterize the electronic properties of phosphine based ligands.

### ➤ 3. Structural Prototype of Metal Carbonyls

The symmetry behavior of different metal carbonyls can be used to determine the number of infrared active vibrational modes, which in turn enable us to comment on structural prototypes. In other words, it is well known from the group theory that only those vibrational modes will be observed which transform as the electric-dipole moment operator; therefore, the number of observable infrared transitions for a particular geometry can be predicted theoretically. These predictions are then matched with the experimentally observed infrared spectra to shortlist the various possible structural prototypes. For instance, consider the case of five-coordinated homoleptic metal carbonyl complexes. The two possible geometries are square-pyramidal and trigonal bipyramidal.

Figure 13. Two possible geometries of Iron pentacarbonyl i.e.  $\text{Fe}(\text{CO})_5$  complex.

For trigonal bipyramidal geometry, a reducible representation based on five C–O bonds is:

$D_{3h}$	E	$2C_3$	$3C_2$	$\sigma_h$	$2S_3$	$3\sigma_d$	Irreducible components
$\Gamma_\pi$	5	2	1	3	0	3	$2A_1' + A_2'' + E'$

Out of four irreducible representations,  $A_2''$  transforms as  $z$ -component while  $E'$  transforms with  $x$ - and  $y$ -components of the dipole moment. Now, owing to two doubly degenerate vibrational modes ( $E'$ ), only two peaks are expected in the experimental infrared spectrum. However, in the Raman infrared spectrum, three peaks are expected as only  $2A_1'$  and  $E'$  irreducible components transform alongside the polarizability tensors.

For square pyramidal geometry, a reducible representation based on five C–O bonds is:

$C_{4v}$	E	$2C_4$	$C_2$	$2\sigma_v$	$2\sigma_d$	Irreducible components
$\Gamma_\pi$	5	2	1	3	3	$2A_1 + B_1 + E$

Out of four irreducible representations,  $2A_1$  transforms as  $z$ -component while  $E$  transforms with  $x$ - and  $y$ -components of the dipole moment. Now, owing to two doubly degenerate vibrational modes ( $E$ ), only three peaks are expected in the experimental infrared spectrum. However, in the Raman infrared spectrum, four peaks are expected as all of the irreducible components transform alongside the polarizability tensors.

Similarly, the CO ligands of octahedral complexes, e.g.  $\text{Cr}(\text{CO})_6$ , transform as  $A_{1g}$ ,  $E_g$ , and  $T_{1u}$ , but only the  $T_{1u}$  mode (anti-symmetric stretch of the apical carbonyl ligands) is infrared-active; and therefore, only a single carbonyl stretching is observed in the IR-spectra of the octahedral metal hexacarbonyls. Spectra for complexes of lower symmetry are more complex. For example, the IR spectrum of  $\text{Fe}_2(\text{CO})_9$  displays CO bands at 2082, 2019, 1829  $\text{cm}^{-1}$ . The number of observable infrared-active vibrational modes for some metal carbonyls are listed in the following below.

Table 1. The number of IR-active vibrational modes of several prototypical metal carbonyl complexes.

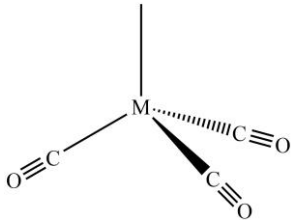
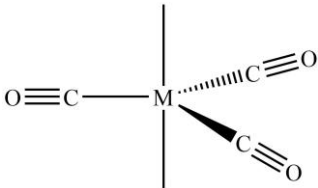
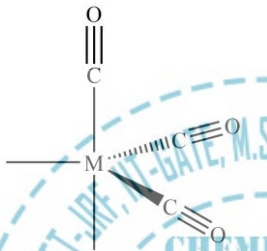
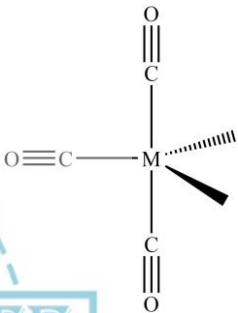
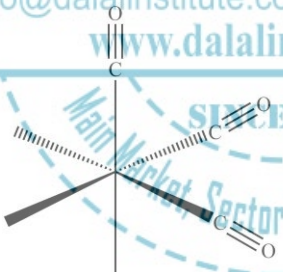
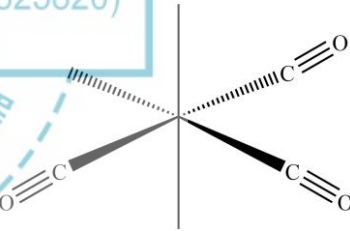
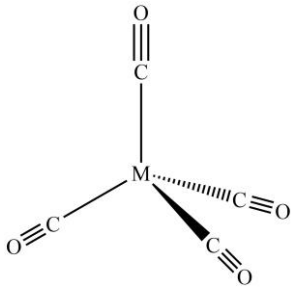
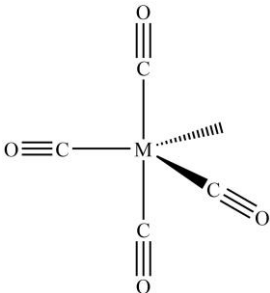
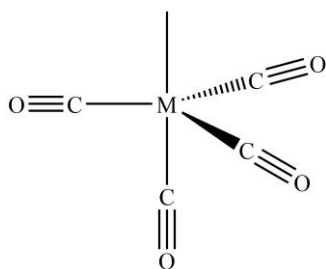
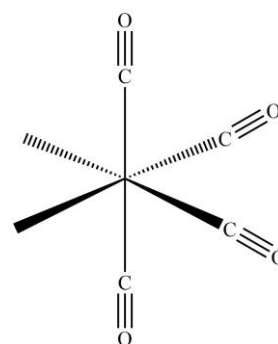
No. of carbonyls	Structural prototype (No. of IR-peaks)	Structural prototype (No. of IR-peaks)
Three		
	(2)	(1)
		
	(3)	(3)
		
	(2)	(3)
Four		
	(1)	(4)

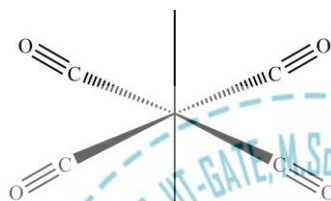
Table 1. Continued on the next page...



(3)



(4)

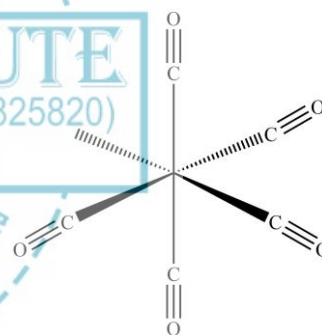


(3)

Five

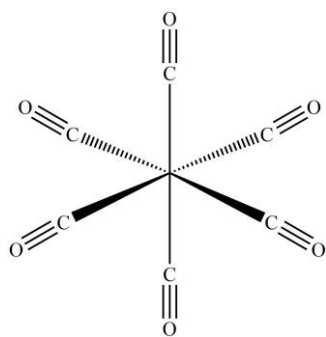


(2)



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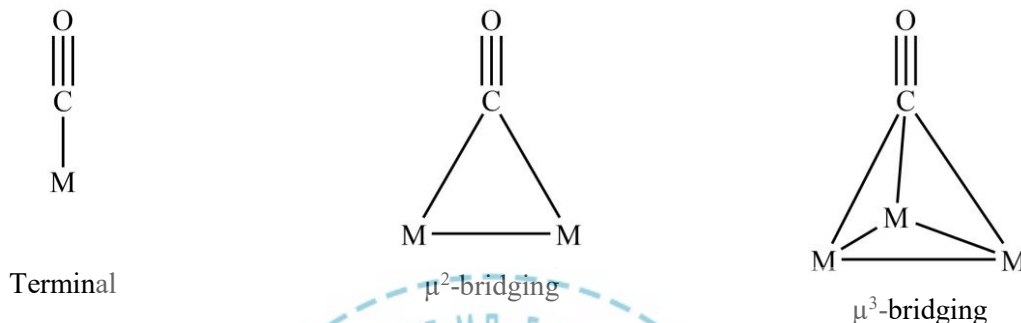


(1)

It is worthy to note that these rules apply to metal carbonyls in solution or the gas phase.

➤ **4. Differentiation of Terminal and Bridging Carbonyl Groups**

The mode of attachment of the carbonyl group to the metal center can also be determined by observed CO stretching frequencies. Terminal carbonyls absorb at the higher wavenumber in comparison to the bridging ones, which is obviously due to the fact that the extent of backbonding increases with the number of metal centers. Three main modes of attachment of the carbonyl group with the metal center are:



The  $\nu_{\text{CO}}$  follows the order: Terminal CO >  $\mu^2$ -bridging >  $\mu^3$ -bridging. The IR-range absorption for various types of carbonyl groups is listed below.

Table 2. The CO stretching frequencies in different metal carbonyl complexes.

Bonding mode of CO	$\nu_{\text{CO}}$ ( $\text{cm}^{-1}$ )
Free CO	2143
Free $\text{CO}^+$	2184
Terminal CO	2120 - 1850
Symmetric $\mu^2$ -CO	1860 - 1750
Symmetric $\mu^3$ -CO	1730 - 1600

For instance, consider the rhodium carbonyl complexes:

Compound	$\mu^1$ -CO, $\nu_{\text{CO}}$ ( $\text{cm}^{-1}$ )	$\mu^2$ -CO, $\nu_{\text{CO}}$ ( $\text{cm}^{-1}$ )	$\mu^3$ -CO, $\nu_{\text{CO}}$ ( $\text{cm}^{-1}$ )
$\text{Rh}_2(\text{CO})_8$	2060, 2084	1846, 1862	
$\text{Rh}_4(\text{CO})_{12}$	2044, 2070, 2074	1886	
$\text{Rh}_6(\text{CO})_{16}$	2045, 2075		1819

There is also a semi-bridging mode that lies in between bridging and terminal bonding profile and is usually labeled as asymmetric bridging carbonyls.

➤ 5. Calculation of CO Bond Order

Owing to the well-known fact that the highest occupied molecular orbital (HOMO) set in carbonyl ligand is of antibonding nature, a close correlation between the magnitude of backbonding and carbon-oxygen bond order can easily be established. The vibrational frequency of a bond depends upon the strength of the bond (which is measured by its force constant), and is given as:

$$v_{CO}(\text{cm}^{-1}) = \frac{1}{2\pi c} \sqrt{\frac{k}{\mu}} \quad (1)$$

Where  $k$  is the force constant and  $\mu$  is the reduced mass. The physical significance of  $k$  is implied in the fact that it is proportional to the strength of the bond involved. Therefore, any factor that increases the electron density on CO group will decrease its bond order and its force constant; and the vice-versa is also true. In other words, the decrease in  $v_{CO}$  indicates a decrease in CO bond order while the increase in carbonyl stretching is associated with increasing CO bond order. For instance, the infrared absorption of free CO occurs at  $2143 \text{ cm}^{-1}$  while the metal coordinated CO absorbs generally in the range of  $2120\text{-}1800 \text{ cm}^{-1}$ . This clearly establishes the fact that metal to ligand back donation does occur which in turn reduces its bond order. Hence, the accumulation of positive charge or the deficiency of electron density will impart a larger CO bond order than neutral or ionic carbonyl complexes. Consider the following trend in isoelectronic-isostructural carbonyls.

Compound	$[\text{Ni}(\text{CO})_4]$	$[\text{Co}(\text{CO})_4]^{1-}$	$[\text{Fe}(\text{CO})_4]^{2-}$
$v_{CO} (\text{cm}^{-1})$	2046	1890	1730
M-C Bond order	1.33	1.89	2.16
C-O Bond order	2.64	2.14	1.85
M-C + C-O Bond order	3.97	4.03	4.01

A similar trend is observed in the case of mixed carbonyls i.e. metal carbonyls having some other ligands alongside CO group. If the other ligand is electron-withdrawing in nature, it would attract the electron density from the metal center, which in turn would oppose the metal-carbonyl back bonding, yielding somewhat higher carbon-oxygen bond order and higher carbonyl stretching frequency. On the other hand, If the other ligand is electron-donating in nature, it would donate the electron density to the metal center, which in turn would support the metal-carbonyl back bonding, yielding somewhat lower carbon-oxygen bond order and lower carbonyl stretching frequency.

Compound	$[\text{Ni}(\text{CO})_4]$	$[\text{Ni}(\text{PF}_3)(\text{CO})_3]$	$[\text{Ni}(\text{PMe}_3)(\text{CO})_3]$
$v_{CO} (\text{cm}^{-1})$	2046	1990	1980

Hence, the bond order of CO in  $[\text{Ni}(\text{PMe}_3)(\text{CO})_3]$  is definitely lower than in  $[\text{Ni}(\text{PF}_3)(\text{CO})_3]$  because  $\text{PMe}_3$  is a weaker  $\pi$ -acceptor than  $\text{PF}_3$  ligand.

➤ **6. Study of Reaction Kinetics in Metal Carbonyls**

Infrared spectroscopy is a very useful tool in the determination of reaction kinetics of metal carbonyl complexes. From Beer's Lambert Law, we know that the absorbance ( $A$ ) is related to concentration ( $c$ ) and path length ( $l$ ) of the sample as:

$$A = \epsilon cl = \log \frac{1}{T} \quad (1)$$

Where  $\epsilon$  is the molar extinction coefficient and  $T$  is the transmittance of the solution. Therefore, the rate of disappearance or appearance of a characteristic infrared absorption band can be used to estimate the rate of change of concentration for the corresponding reactant or product. In other words, the rate of intensity decrease in IR peaks of reactant, or the rate of intensity increase in IR peaks of product metal carbonyl is proportional to the rate of change of concentration; which in turn enable us to record various kinetic parameters from reaction order to activation energy or rate constants.



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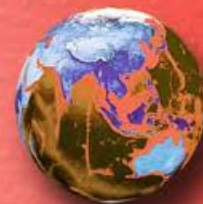
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**Volume I**

**MANDEEP DALAL**



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