J. Am. Chem. Soc. 1986, 108, 2894-2897

The Relative Intensities of Vibrational Spectral Bands in the ν(CO) Region of $[Os_4O_4(CO)_{12}]$

Upali A. Jayasooriya* and Christopher E. Anson

Contribution from the School of Chemical Sciences, University of East Anglia, Norwich NR4 7TJ, United Kingdom. *Received November 27, 1985*

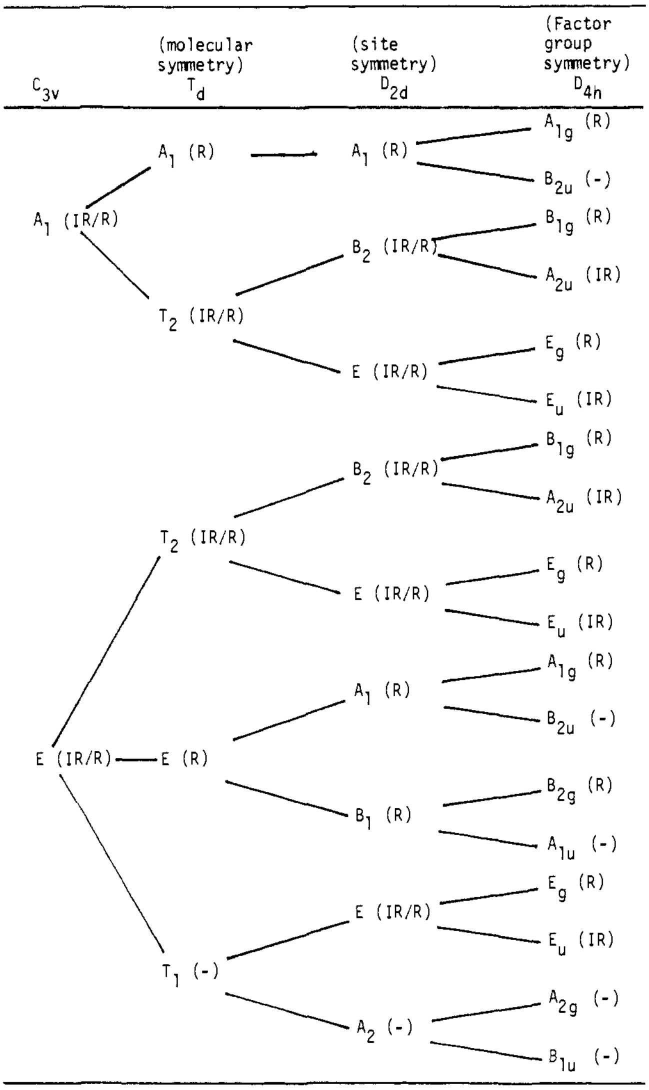
Abstract: The unusual vibrational intensities of the carbonyl stretching region of $[Os_4O_4(CO)_{12}]$ in the solid state are shown to be a consequence of the crystal and molecular structure. The derived polarizabilities perpendicular and parallel to the C–O bond are shown to be almost equal in magnitude but certainly opposite in sign.

It is normal practice to use a full factor group analysis in the interpretation of infrared and Raman spectroscopic data (i.e., **k** $\approx$ 0) of solids.[1] There are also, however, many cases in which the weakness of the intermolecular interactions present in the solid results in a site group model being a sufficiently good approximation. Most compounds probably lie between these two extremes, a factor group analysis being necessary to interpret some regions of the spectrum but a site group model being sufficient for other spectral regions.

However, the situation is not always quite as simple. In many cases intermolecular vibrational coupling can be seen to be of sufficient magnitude for a site group approach to be inadequate, but at the same time the predictions from a full factor group analysis are not realized. In these systems a certain amount of success has been achieved by the recognition of approximate symmetries present in the solid state[2-4] which appear to have a profound effect on spectral intensities. A more detailed understanding of vibrational spectral intensities in the solid state might well facilitate crystal structure determinations and in particular enable the correct choice of space group to be made where X-ray crystallographic techniques cannot distinguish between two or more possibilites.[5]

Detailed vibrational analysis of metal carbonyl clusters is confined to a very few compounds[6-10] compared to the simple mononuclear transition metal carbonyls where the ability qualitatively to interpret the ν(CO) vibrational spectra has been of considerable utility.[11] Therefore, there is a need to investigate the validity of the extension of treatments developed with the simple carbonyls to the cluster carbonyls, especially in the solid state since, although solution infrared spectra are routinely obtained, the majority of clusters are insufficiently soluble for accurate Raman data to be obtained in solution. Some of the problems encountered with such studies are discussed in the literature by Kettle, Rossetti, and Stanghellini.[8] Here we report an investigation of the ν(CO) region of $[Os_4O_4(CO)_{12}]$ in the solid state which shows an unexpectedly simple spectral pattern in the Raman.

Table I



$[Os_4O_4(CO)_{12}]$ was first reported by Johnson, Lewis, Williams, and Wilson.[12] An X-ray crystal structure determination by Bright[13] has shown it to crystallize in the tetragonal system, space group $I4_1/amd$, with a unit cell content of four molecules. The primitive unit cell, with which we are concerned in vibrational studies, contains two molecules. The molecular structure is shown schematically in Figure 1, with the crystallographic S_4 axis. It should be noted that, to a close approximation, the carbonyl ligands are all mutually either parallel or perpendicular. The vibrational spectra in the carbonyl stretching region are shown in Figure 2. A factor group analysis for the ν(CO) vibrations involving a

---

(1) Decius, J. C.; Hester, R. M. *Molecular Vibrations in Crystals*; McGraw-Hill: New York, 1977.

(2) Ismail, M.; Jayasooriya, U. A.; Kettle, S. F. A. *J. Chem. Phys.* **1983**, *79*, 4459.

(3) Kettle, S. F. A.; Jayasooriya, U. A.; Norrby, L.-J. *J. Phys. Chem.* **1984**, *88*, 5971.

(4) Jayasooriya, U. A.; Kettle, S. F. A. *Phys. Rev. B* **1984**, *29*, 2227.

(5) Durman, R.; Jayasooriya, U. A.; Kettle, S. F. A. *J. Chem. Soc., Chem. Commun.* **1985**, 916.

(6) Quicksall, C. O.; Spiro, T. G. *Inorg. Chem.* **1968**, *7*, 2365.

(7) Battiston, G. A.; Bor, G.; Dietler, U.; Kettle, S. F. A.; Rossetti, R.; Sbrignadello, G.; Stanghellini, P. L. *Inorg. Chem.* **1980**, *19*, 1961.

(8) Kettle, S. F. A.; Rossetti, R.; Stanghellini, P. L. *Inorg. Chem.* **1983**, *22*, 661.

(9) Bor, G.; Sbrignadello, G.; Marcati, F. *J. Organomet. Chem.* **1972**, *46*, 357.

(10) Bor, G.; Sbrignadello, G.; Noack, K. *Helv. Chim. Acta* **1975**, *55*, 815.

(11) Braterman, P. S. *Metal Carbonyl Spectra*; Academic Press: New York, 1975.

(12) Johnson, B. F. G.; Lewis, J.; Williams, I. G.; Wilson, J. *J. Chem. Soc., Chem. Commun.* **1966**, 391.

(13) Bright, D. *J. Chem. Soc., Chem. Commun.* **1970**, 1169.

0002-7863/86/1508-2894$01.50/0

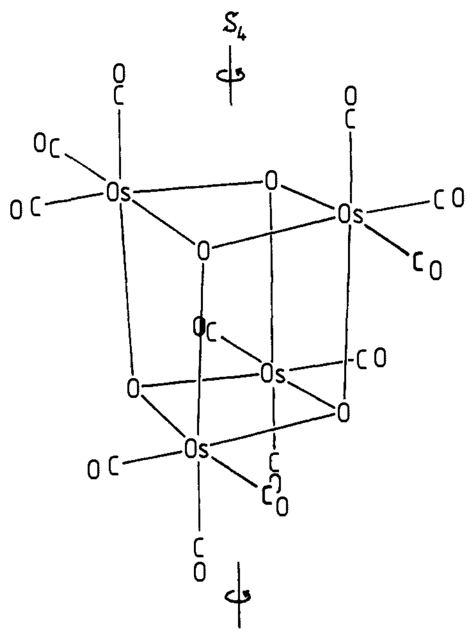


Figure 1. Molecular structure of $[Os_4O_4(CO)_{12}]$ in the crystalline state.[13]

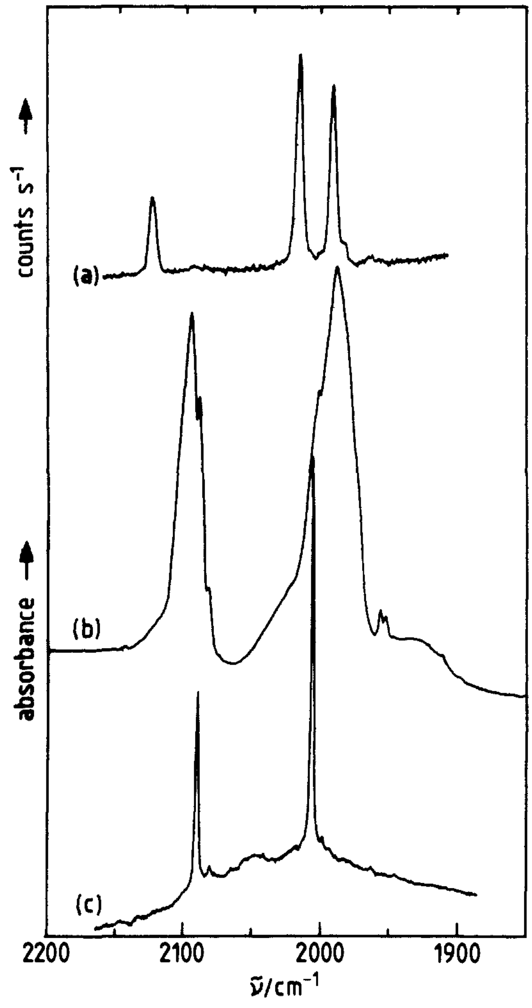


Figure 2. (a) Raman spectrum of polycrystalline $[Os_4O_4(CO)_{12}]$ at ca. 100 K. (b) Infrared spectrum of solid $[Os_4O_4(CO)_{12}]$ in CsI disk at ca. 100 K. (c) Infrared spectrum of $[Os_4O_4(CO)_{12}]$ in dilute cyclohexane solution.

symmetry correlation from the three CO groups on one metal atom with C_{3v} symmetry via the T_d symmetry of the four Os atoms in a regular Os_4O_4 molecular skeleton and the D_{2d} site symmetry to the factor group D_{4h} is given in Table I.

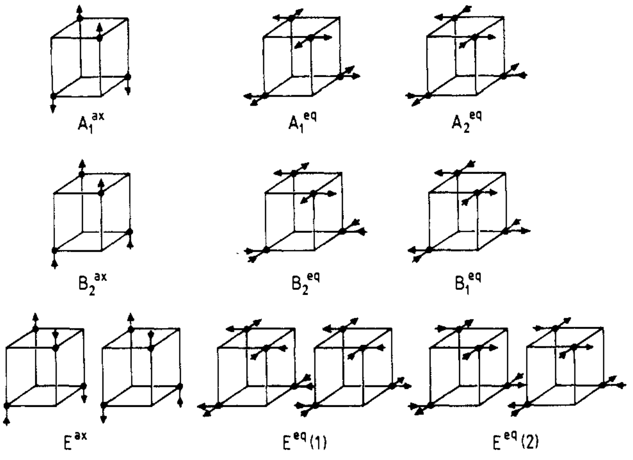


Figure 3. Symmetry coordinates for the ν(CO) vibrations of $[Os_4O_4(CO)_{12}]$ under D_{2d} site symmetry.

The infrared spectrum of $[Os_4O_4(CO)_{12}]$ in cyclohexane solution is easily interpreted as due to four $Os(CO)_3$ groups coupled over a molecule of undistorted T_d symmetry, and the vibrational bands at 2090.1 and 2006.2 cm^{-1} are assigned to the two T_2 modes expected for such a symmetry (Table I). The bands in the infrared of the solid are broad and overlapping as is usually expected and observed in this region for such strongly interacting vibrators.[1] However, five bands can be observed within the two broad features at 2096, 2090, ca. 2035, 2002, and 1988 cm^{-1}, assignable to the ν(CO) vibrations in agreement with a site or a factor group analysis (Table I). The weak features at 2087, 1957, and 1953 cm^{-1} are probably due to the natural abundance of ^{13}CO. The relative intensities of the two broad features are ca. 1:2, thus revealing their parentage in the $Os(CO)_3$ group vibrations of A_1 and E symmetry under C_{3v} point group (Table I).

The primitive unit cell of this compound has two molecules related by a center of symmetry, and therefore either the infrared or the Raman spectrum taken in isolation should be explicable in terms of a molecular model under the site symmetry. This is broadly what is observed in the infrared. Any intermolecular coupling effect should in principle be manifested as a separation between the infrared and Raman frequencies corresponding to these molecular vibrations.

According to Table I, eight Raman active modes are predicted under either a factor group or a site group analysis, four modes under molecular T_d symmetry and only two modes for an isolated $Os(CO)_3$ model. However, three modes are observed experimentally (Figure 2a) in disagreement with each of these predictions. The inaccuracy of the possible assignment of these three bands to an A_1 mode and a split E mode under an isolated $Os(CO)_3$ group of near C_{3v} symmetry is shown by the following observations. First, the infrared data clearly show the need for at least a molecular site group analysis, and the presence of strong intermolecular vibrational interactions in the CO stretching region of carbonyl compounds is without dispute.[11] Second, the intensity ratio between these modes is very different to the 1:2 value expected for an $I_{A_1}:I_E$ ratio under C_{3v} symmetry, indicating strong mixing between these modes in the Raman under site and factor group symmetries. An explanation of the observed Raman spectrum is clearly not to be found in the above analyses, and therefore we must look elsewhere. The following explanation is based on the bond polarizability approach introduced by Wolenstein.[14-17]

The molecular structure of this compound in the solid state is such that the CO bonds about a metal atom are mutually perpendicular. These bonds are divided into two symmetry-related sets, axial and equatorial with respect to the crystallographic S_4

---

(14) Wolkenstein, M. *J. Exp. Theor. Phys.* **1941**, *11*, 642.
(15) Wolkenstein, M. *J. Phys.* **1943**, *5*, 185.
(16) Eliashevich, M.; Wolkenstein, M. *J. Phys.* **1945**, *9*, 101.
(17) Eliashevich, M.; Wolkenstein, M. *J. Phys.* **1945**, *9*, 326.

Table II

vibrational assignment				calculated values of a/b ratio			
2125 cm^{-1}	2015 cm^{-1}	1992 cm^{-1}	δ				
A_1^{ax} A_1^{eq}	A_1^{eq} A_1^{ax}	B_1^{eq} B_1^{eq}	1.57	−0.19	$\underline{-1.04}$	−0.32	−0.60
A_1^{ax} A_1^{eq}	B_1^{eq} B_1^{eq}	A_1^{eq} A_1^{ax}	1.40	−0.21	$\underline{-0.96}$	−0.35	−0.60
B_1^{eq} B_1^{eq}	A_1^{eq} A_1^{ax}	A_1^{ax} A_1^{eq}	5.39	0.07	−2.89	0.30	−0.69

axis under D_{2d} site symmetry (see Figure 1). The irreducible representations spanned by the axial set are $A_1 + B_2 + E$ while those spanned by the equatorial set are $A_1 + A_2 + B_1 + B_2 + 2E$. The symmetry coordinates corresponding to these vibrations are given in Figure 3. The favorable molecular geometry of this compound together with the relatively high symmetry of its crystal structure facilitates the application of the Wolkenstein approach[14-17] for the estimation of polarizability derivatives which in turn provides predictions for Raman intensities.

We follow the analysis given by Kettle, Paul, and Stamper[18] for the case of the group 6B metal hexacarbonyls and assume cylindrical symmetry for the derived polarizability tensor for a C–O bond and use two parameters a and b to define the C–O bond polarizability derivatives respectively perpendicular and parallel to the CO bond. This analysis results in the following derived polarizability tensors for the symmetry coordinates given in Figure 3.

$$A_1^{ax}: \quad 2\begin{pmatrix} a & 0 & 0 \\ 0 & a & 0 \\ 0 & 0 & b \end{pmatrix}$$

$$A_1^{eq}: \quad 2\begin{pmatrix} (a+b) & 0 & 0 \\ 0 & (a+b) & 0 \\ 0 & 0 & 2a \end{pmatrix}$$

$$B_1^{eq}: \quad 2\begin{pmatrix} (a-b) & 0 & 0 \\ 0 & -(a-b) & 0 \\ 0 & 0 & 0 \end{pmatrix}$$

All the other coordinates of symmetry B_2^{ax}, E^{ax}, A_2^{eq}, B_2^{eq}, and $2E^{eq}$ give zero derived polarizability tensors. Now an inspection of Table I shows that the factor group modes derived from the molecular A_1^{ax}, A_1^{eq}, and B_1^{eq} modes are "segregated" by symmetry from the factor group modes. This immediately explains the presence of only three bands in the solid-state Raman spectrum as it is the in-phase coupling of the molecular vibrations of the two centrosymmetrically related molecules of the unit cell which are Raman active in the crystal. The expressions given above for the molecularly derived polarizability tensor elements are appropriate for the unit cell vibrations when these are held fixed in a set of Cartesian coordinate axes. Before comparison with experimental intensities we must allow for the random orientations of the unit cells in the polycrystalline samples which were investigated. By using the method given for this by Wilson, Decius, and Cross[19] and following a similar line of argument to Kettle, Paul, and Stamper,[18] the following expressions for the intensities are obtained.

$$I_T^{A_1^{ax}}(\text{obsd} \perp) = \frac{16\pi^4 \nu_{A_1^{ax}}{}^4}{45c^4} N I_0[90(2a+b)^2 + 28(a-b)^2]$$

$$I_T^{A_1^{eq}}(\text{obsd} \perp) = \frac{16\pi^4 \nu_{A_1^{eq}}{}^4}{45c^4} N I_0[40(2a+b)^2 + 2(a-b)^2]$$

$$I_T^{B_1^{eq}}(\text{obsd} \perp) = \frac{16\pi^4 \nu_{B_1^{ax}}{}^4}{45c^4} N I_0[42(a-b)^2]$$

Assuming $\nu_{A_1^{ax}} = \nu_{A_1^{eq}} = \nu_{B_1^{eq}}$, since these refer to the frequency of the scattered light (17 310, 17 470, and 17 413 cm^{-1} for a laser frequency of 19 435 cm^{-1}) not to the frequency of the vibrations, it is possible to obtain much simpler expressions in terms of ratios of intensities. The likelihood of coupling between these A_1 modes should be taken into account in calculating these ratios since strong coupling between modes of A_1 symmetry has been shown to be present in many of the better understood compounds such as the trinuclear metal clusters $[M_3(CO)_{12}]$.[7] Thus the following expression is derived:

$$\frac{I_T^{A_1^{ax}} + I_T^{A_1^{eq}}}{I_T^{B_1^{eq}}} = \frac{5}{7} + \frac{65}{21}\left(\frac{2a+b}{a-b}\right)^2 = \delta(\text{say})$$

$$\therefore \frac{a}{b} = \frac{\sqrt{(\delta - 5/7)21/65} + 1}{\sqrt{(\delta - 5/7)21/65} - 2}$$

Therefore for each value of δ, one would obtain four possible values of a/b. The possible assignments of the observed bands together with the values of δ and the a/b ratios are given in Table II.

The assignment of the highest frequency carbonyl band in the Raman spectra of metal carbonyl compounds to a totally symmetric mode is now undisputed.[6-11,18,20] Therefore the third assignment given in Table II can be disregarded. It is not possible, with the data available at present, to distinguish between the other two assignments, although studies by Gilson[21] suggest the first of these assignments as the more likely. It has not proved possible to grow sufficiently large single crystals for a single crystal Raman experiment which would provide such a distinction. The sets of a/b ratios calculated for these two assignments are very close indeed (Table II) and therefore the following conclusions are independent of the validity of either assignment.

First, all a/b ratios are negative, indicating the derived polarizability parallel and perpendicular to the C–O bond to be opposite in sign. This is in agreement with the results of such an analysis for the group 6B metal hexacarbonyls in the solution state.[18] In the latter study, Kettle, Paul, and Stamper[18] find two possible values for this ratio of −1.1 and −4.5 and opt for the first value on chemical grounds. The present study provides a distinction between these two values on the reasonable assumption that the CO bonds are not very different between these compounds. It is a value close to −1 which is common to both these studies and is thus assigned to the a/b ratio.

Experimental Section

A sample of $[Os_4O_4(CO)_{12}]$ was kindly provided by Dr. B. F. G. Johnson, University of Cambridge. Infrared spectra were run on a Digilab FTS-20V Fourier transform infrared spectrometer, while the Raman spectra were run with a Spex 1401 Raman spectrophotometer and the 514 nm exciting line of a Spex 164 argon ion laser.

Conclusion

The unusual Raman spectrum of $[Os_4O_4(CO)_{12}]$ in the carbonyl stretching region of the solid state is explained by using the Wolkenstein bond polarizability approach,[14-17] which predicts zero intensity for five out of the eight vibrational modes expected to

---

(18) Kettle, S. F. A.; Paul, I.; Stamper, P. J. *Inorg. Chim. Acta* **1973**, *7*, 11.

(19) Wilson, E. B.; Decius, J. C.; Cross, P. C. "Molecular Vibrations"; McGraw-Hill: New York, 1955.

(20) Jayasooriya, U. A.; Skinner, P. *Inorg. Chem.*, submitted for publication.

(21) Gilson, T. R. *J. Chem. Soc., Dalton Trans.* **1984**, 149.

be spectrally active under a site or a factor group model. Therefore this is an interesting example of a *non*-centrosymmetric molecule showing a centrosymmetric infrared and Raman mutually exclusive spectral pattern (Table II). Thus, the criterion for the detection of the factor group effects in this compound, where one expects it to be manifested as a separation between the infrared and Raman frequencies of the infrared–Raman coincident site group vibrational modes, cannot be used.

This investigation also confirms an earlier observation on the group 6B metal hexacarbonyls[18] by indicating the values of the derived polarizabilities perpendicular and parallel to the CO bond to be almost equal in magnitude but certainly opposite in sign.

Registry No. $Os_4O_4(CO)_{12}$, 29994-93-2.

Electron-Transfer Catalysis of Ligand Substitution in Triiron Clusters. The Role of the Bridging Ligand in Anion Radical Intermediates

H. H. Ohst and J. K. Kochi*

Contribution from the Department of Chemistry, University of Houston, University Park, Houston, Texas 77004. *Received October 10, 1985*

Abstract: The polynuclear cluster $Fe_3(CO)_9(\mu_3\text{-}PPh)_2$ (I) undergoes rapid ligand substitution by electron-transfer catalysis (ETC) under conditions in which the thermal process is nonexistent. X-ray crystallography and ^{31}P NMR spectroscopy establish the stepwise substitution of the CO ligands by trimethyl phosphite to take place selectively at three separate iron centers. The high selectivity to the mono-substitution product II is achieved by tuning the reduction potential specifically to generate catalytic amounts of the anion radical $Fe_3(CO)_9(PPh)_2^{-}\cdot$ ($I^{-}\cdot$) in either acetonitrile or tetrahydrofuran. Transient ESR spectroscopy of $I^{-}\cdot$ and three related paramagnetic intermediates establish the sequential transformation of anion radicals as they evolve in the ETC mechanism. The rate-limiting rearrangement of $I^{-}\cdot$ by the slippage of a phosphinidene cap from $\mu_3 \rightarrow \mu_2$ coordination underscores the key role of the bridging ligand in the substitution process. The importance of this critical transformation relates to the formation of a 17-electron, coordinatively unsaturated iron center in the otherwise intact cluster. As such, it emphasizes the key role that the bridging ligand can play in cluster activation.

Active interest in transition-metal clusters as catalysts derives from their conceptual relationship to catalytically active metal surfaces.[1,2] Indeed their delocalized electronic structure,[3] characteristic of metals, is reflected in their multiple redox behavior[4] as well as in their multimetal reactivity.[5] These two important aspects of catalytic activity may be coupled. Thus, if one considers that metal–metal bonding may not be stronger than metal–ligand bonding (except with the heaviest transition metals),[6] the polymetallic core should participate directly in the reactions of the clusters, especially under reducing conditions. For example, ESR studies have shown that the metal frameworks of trinuclear clusters with one and two μ_3-bridging ligands [i.e., $L_nM_3(\mu_3\text{-}E)$ and $L_mM_3(\mu_3\text{-}E)_2$ where E = CR, PR, or S] are weakened upon reduction, in accord with the metal–metal antibonding character of the LUMOs.[7-10] In these clusters, the bridging ligands E help to preserve the integrity of the cluster, since polynuclear metal carbonyls are known to otherwise undergo ready cleavage to species of lower nuclearity under reducing conditions.[11] Activation of metal clusters by reduction also relates to recent evidence that ligand substitutions of $L_nM_3(\mu_3\text{-}E)$[12] and $L_mM_3(\mu_3\text{-}E)_2$[13] are both strongly induced by electron attachment. Such studies of electron-transfer catalysis suggest that the chemical reactivity of metal clusters may generally be enhanced by deliberate reduction.[11-14]

We believe that electron-transfer catalysis (ETC) represents an important new method for cluster activation as it has proved to be for their mononuclear counterparts.[15] In this study, we examine the activation of the bicapped triiron cluster I to ligand substitution via labile radical anion intermediates. Most importantly, the ESR detection and characterization of a distinctive series of transient paramagnetic species provide an unusual opportunity to delineate the individual steps of the catalytic cycle involved in ETC.

---

(1) Moskovitz, M. *Acc. Chem. Res.* **1979**, *12*, 229.

(2) Muetterties, E. L.; Rhodin, R. N.; Band, E.; Brucker, C. F.; Pretzer, W. R. *Chem. Rev.* **1979**, *79*, 91.

(3) Wooley, R. G. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: New York, 1980; p 607.

(4) See, e.g.: Vahrenkamp, H. *Adv. Organomet. Chem.* **1983**, *22*, 167.

(5) See: Deeming, A. J. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: New York, 1980; p 391.

(6) Connor, J. A. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: New York, 1980; p 345.

(7) (a) Strouse, C. E.; Dahl, L. F. *Discuss. Faraday Soc.* **1969**, *47*, 93. Stevenson, D. L.; Wei, C. H.; Dahl, L. F. *J. Am. Chem. Soc.* **1971**, *93*, 6027. Strouse, C. E.; Dahl, L. F. *J. Am. Chem. Soc.* **1971**, *93*, 6032. Frisch, P. D.; Dahl, L. F. *J. Am. Chem. Soc.* **1972**, *94*, 5082. (b) Beurich, H.; Madach, T.; Richter, F.; Vahrenkamp, H. *Agnew. Chem., Int. Ed. Engl.* **1979**, *18*, 690. (c) Bond, A. M.; Dawson, P. A.; Peake, B. M.; Rieger, P. H.; Robinson, B. H.; Simpson, J. *Inorg. Chem.* **1979**, *18*, 1413. (d) Peake, B. M.; Rieger, P. H.; Robinson, B. H.; Simpson, J. *Inorg. Chem.* **1981**, *20*, 2540. (e) Enoki, S.; Kawamura, T.; Yonezawa, T. *Inorg. Chem.* **1983**, *22*, 3821. (f) Kotz, J. C.; Petersen, J. V.; Reed, R. C. *J. Organomet. Chem.* **1976**, *120*, 433. (g) Bond, A. M.; Peake, B. M.; Robinson, B. H.; Simpson, J.; Watson, D. J. *Inorg. Chem.* **1977**, *16*, 410. (g) Lindsey, P. N.; Peake, B. M.; Robinson, B. H.; Simpson, J.; Honrath, U.; Vahrenkamp, H.; Bond, A. M. *Organometallics* **1984**, *3*, 413. (h) See also ref 17.

(8) Lindsey, P. N.; Peake, B. M.; Robinson, B. H.; Simpson, J.; Honrath, U.; Vahrenkamp, H.; Bond, A. M. *Organometallics* **1984**, *3*, 413.

(9) (a) Muller, M.; Vahrenkamp, H. *Chem. Ber.* **1983**, *116*, 2765. (b) Vahrenkamp, H. *Angew. Chem., Int. Ed. Engl.* **1979**, *18*, 690. (c) Honrath, U.; Shu-Tang, L.; Vahrenkamp, H. *Chem. Ber.* **1985**, *118*, 132.

(10) (a) Schilling, B. E. R.; Hoffmann, R. *J. Am. Chem. Soc.* **1979**, *101*, 3456. (b) Rives, A. B.; Xiao-Zeng, Y.; Fenske, R. F. *Inorg. Chem.* **1982**, *21*, 2286. (c) Chesky, P. T.; Hall, M. B. *Inorg. Chem.* **1983**, *22*, 2102. (d) Chesky, P. T.; Hall, M. B. *Inorg. Chem.* **1983**, *22*, 2998.

(11) (a) Vahrenkamp, H. *Struct. Bonding (Berlin)* **1977**, *32*, 1. (b) Deeming, A. J. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: New York, 1980; Chapter 6.

(12) (a) Bezems, G. J.; Rieger, P. H.; Visco, S. *J. Chem. Soc., Chem. Commun.* **1981**, 265. (b) Bruce, M. I.; Kehoe, D. C.; Matisons, J. G.; Nicholson, B. K.; Rieger, P. H.; Williams, M. L. *J. Chem. Soc., Chem. Commun.* **1982**, 442.

(13) (a) Darchen, A.; Mahe, C.; Patin, H. *J. Chem. Soc., Chem. Commun.* **1982**, 243. (b) Darchen, A.; Mahe, C.; Patin, H. *Nouv. J. Chim.* **1982**, *6*, 539. (c) See also: Lhadi, E. K.; Mahe, C.; Patin, H.; Darchen, A. *J. Organomet. Chem.* **1983**, *246*, C61. And: Darchen, A.; Mahe, C.; Patin, H. *Nouv. J. Chim.* **1983**, *7*, 453.

(14) Arewgoda, M.; Robinson, B. H.; Simpson, J. *J. Am. Chem. Soc.* **1983**, *105*, 1893.

(15) Hershberger, J. W.; Kochi, J. K. *J. Chem. Soc., Chem. Commun.* **1982**, 212.

0002-7863/86/1508-2897$01.50/0