# STUDIES ON SELECTED MONO- AND TETRANUCLEAR GROUP 8 CARBONYL COMPOUNDS

by

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of

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### ABSTRACT

M(CO)<sub>4</sub>L (M=Fe,Ru,Os, L=EPh<sub>3</sub>, E=P,As,Sb, The compounds L=PMe<sub>3</sub>, P(OCH<sub>2</sub>)<sub>3</sub>CMe; M=Ru,Os, L=SbMe<sub>3</sub>) have been synthesized from L and  $M(CO)_5$ . In  $Ru(CO)_4AsPh_3$  and  $Ru(CO)_4SbMe_3$ , crystal structures revealed the unique ligand is in an axial position of trigonal-bipyramidal coordination sphere, whereas, in the Os(CO)<sub>4</sub>SbPh<sub>3</sub>, SbPh<sub>3</sub> is in an equatorial site. Infrared spectroscopy showed the presence of both axial and equatorial isomers in solution for many of the complexes; interconversion rapid on the <sup>13</sup>C NMR time scale. The tendency to give the is less common equatorial isomer is Ru>Os>>Fe, Sb>As>P, Ph>Me, and  $P(OCH_2)_3CMe > PMe_3, PPh_3$ . The ordering of the group 15 elements is rationalized in terms of their  $\sigma$ -donor rather than  $\pi$ -acceptor ability ( $\pi$  bonding may be important in determining the other trends). This is the first time an axial-equatorial switchover has been observed in a closely related series of five-coordinate compounds.

The clusters  $Os_4(CO)_n PMe_3$  (n=15, 14, 13) have been synthesized and their crystal structures determined. This series constitutes the first example where a 64 electron cluster displays sequential ligand loss to a 62 and then to a 60 electron species. The  $Os_4(CO)_{15}PMe_3$  (1) cluster is the first reported with an unsupported donor-acceptor metal-metal bond. In  $Os_4(CO)_{14}PMe_3$  (2) the  $Os_4$  skeleton is a planar rhomboid with two long and two short Os-Os bonds. This asymmetry is discussed

iii

terms of a novel bondina model usina in three-centre-two-electron bonds. The structure of 2 is compared to that of  $(\mu-H)_2Os_4(CO)_{1,3}PMe_3$  (3) which has also been synthesized and characterized. Although 3 is isoelectronic with 2. it has an  $Os_4$  butterfly structure with a dihedral angle of 113°. The  $Os_4(CO)_{13}PMe_3$  (4) compound is the only reported group 8 tetrahedral cluster with 14 two-electron donor ligands.

The fluxional properties of the clusters have been investigated by variable temperature <sup>13</sup>C NMR spectroscopy. In solution 1 undergoes CO ligand exchange probably via several different processes. In contrast to 3 which is rigid, 2 exhibits remarkable nonrigidity which is accounted for by an unusual dynamic rearrangement of the metal skeleton. Cluster 4 is highly fluxional even at -120 °C.

This unique group of molecules will be of interest to chemists investigating structural and fluxional properties of clusters. For Nicole, for whom there has never been enough time.

"Concern for man himself and his fate must always form the chief interest of all technical endeavors, concern for the great unsolved problems of the organization of labor and the distribution of goods--in order that the creations of our mind shall be a blessing and not a curse to mankind. Never forget this in the midst of your diagrams and equations."

. Albert Einstein

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## TABLE OF CONTENTS

Appro	val	• • • • •		ii
Abstr	act	• • • • •	······································	ii
Dedic	ati	on		v
Quota	tio	n		vi
Ackno	wle	dgemer	nts v	ii
List	of	Tables	5	x
List	of	Figure	esxi	ii
Α.		AXIAI	L-EQUATORIAL ISOMERISM IN COMPLEXES M(CO) <sub>4</sub> L (M=Fe, Ru, Os; L=GROUP 15 LIGAND)	
8				1
1	•	Introd	luction	2
		1.1	Overview	2
		1.2	Literature Survey	5
		1.3	Project Description	18
2	2.	Result	ts	20
		2.1	Structural Studies	21
		2.2	Infrared Studies	32
		2.3	NMR Studies	38
. 3	3.	Discus	ssion	42
		3.1	Steric Considerations	42
		3.2	Electronic Properties of the Ligands	47
		3.3	Influence of the Central Metal	52
		3.4	Summary	55
		3.5	Future Directions	58
4	ł.	Exper	imental Procedures	60
		4.1	Materials and Instrumentation	60

		4.2	Synthesis Methods 61
		4.3	Crystal Structure Determinations
в		SYN' Osą(CO	THESIS, STRUCTURE AND CHARACTERIZATION OF $n_{n}PMe_{3}$ (n=15, 14, 13) AND ( $\mu$ -H) <sub>2</sub> Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub> 71
	1.	Intro	duction
		1.1	Theoretical Approaches 72
		1.2	Project Description 91
	2.	Resul	ts and Discussion 93
		2.1	$Os_4(CO)_{15}PMe_3$
		2.2	$Os_4(CO)_{14}PMe_3$ 108
		2.3	$(\mu - H)_{2}Os_{4}(CO)_{13}PMe_{3}$
		2.4	$Os_4(CO)_{13}PMe_3$
		2.5	Summary and Conclusions 139
	3.	Exper	imental Procedures 143
		3.1	Materials and Instrumentation
		3.2	Synthetic Methods 144
		3.3	Crystal Structure Determinations 148
App	endi	x	158
Ref	erer	nces	

ix

## LIST OF TABLES

Table		<i>,</i>		Page
				,
A.1.1 Previously Known	n M(CO) <sub>4</sub> L Deriv	atives		15
A.2.1 Selected Molecul	lar Dimensions	for Ru(CO) <sub>4</sub>	AsPh <sub>3</sub>	22
A.2.2 Selected Molecu	lar Dimensions	for Ru(CO) <sub>4</sub>	SbMe <sub>3</sub>	2 <sup>5</sup>
A.2.3 Selected Molecu	lar Dimensions	for Os(CO) <sub>4</sub>	SbPh <sub>3</sub>	27
A.2.4 M-C Distances in	n Fe(CO) $_5$ and M	(CO) <sub>4</sub> L Comp	ounds	29
A.2.5 Infrared, <sup>13</sup> C N Compounds .	MR and Melting	Point Data	for M(CO) <sub>a</sub> L	33
A.3.1 Ligand Bond Ang	les for M(CO) <sub>4</sub> E	R₃ Complexe	s S	55
A.4.1 Analytical and Derivatives	Mass Spectral I	Data for New	7 M(CO) <sub>4</sub> L	62
A.4.2 Diffractometer M(CO) <sub>4</sub> L Com	Collection and pounds	Refinement	Parameters	for 66
A.4.3 Crystallographi Ru(CO) <sub>4</sub> SbMe	c Data for Ru(( 3 and Os(CO)4As	CO)₄AsPh₃, C sPh₃	)s(CO) <sub>4</sub> SbPh <sub>3</sub>	, 69
B.1.1 Group 8 Tetranu	clear Clusters	with 60 Val	lence Electr	ons.86
B.1.2 Group 8 Butterf	ly Structures .			88
B.1.3 Tetranuclear Gr	oup 8 Clusters	with 64 Val	lence Electr	ons.90
B.2.1 Selected Molecu	lar Dimensions	for $Os_4(CO)$	) <sub>15</sub> PMe <sub>3</sub>	96
B.2.2 Selected Molecu	lar Dimensions	of Os <sub>1</sub> (CO)	₁₄PMe₃	109

B.2.3 Selected Molecular Dimensions for $(\mu-H)_2Os_4(CO)_{13}PMe_3$ .	121
B.2.4 Selected Molecular Dimensions for Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	131
B.3.1 Analytical and Mass Spectral Data for Os <sub>4</sub> Compounds	144
B.3.2 CO Stretching Frequencies, <sup>1</sup> H NMR Resonances and Melting Data for Os <sub>4</sub> Compounds	146
B.3.3 Crystallographic Data For Os <sub>4</sub> Compounds	149
B.3.4 Data Collection Parameters for Os <sub>4</sub> Compounds	152
S.1 Fractional Coordinates for Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	159
S.2 Thermal Parameters for Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	160
S.3 Bond Lengths and Angles for the Phenyl Groups of Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	161
S.4 Structure Factors for Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	162
S.5 Fractional Coordinates for Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	169
S.6 Thermal Parameters for Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	169
S.7 Structure Factors for Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	170
S.8 Fractional Coordinates for Os(CO) <sub>4</sub> SbPh <sub>3</sub>	174
S.9 Thermal Parameters for Os(CO) <sub>4</sub> SbPh <sub>3</sub>	175
S.10 Bond Lengths and Angles for the Phenyl Groups of Os(CO) <sub>4</sub> SbPh <sub>3</sub>	176
S.11 Structure Factors for Os(CO) <sub>4</sub> SbPh <sub>3</sub>	177
S.12 Fractional Coordinates for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> , Molecule 1	189
S.13 Fractional Coordinates for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> , Molecule 2	190
S.14 Thermal Parameters for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> , Molecule 1	191

xi

S.15 Thermal Parameters for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> , Molecule 2	192
S.16 Fractional Coordinates for Hydrogen Atoms of Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub>	192
S.17 Structure Factors for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub>	193
S.18 Fractional Coordinates for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub>	212
S.19 Thermal Parameters for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub>	213
S.20 Structure Factors for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub>	214
S.21 Fractional Coordinates for (µ-H) <sub>2</sub> Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	224
S.22 Thermal Parameters for $(\mu-H)_2OS_4(CO)_{13}PMe_3$	<b>22</b> 5
S.23 Structure Factors for $(\mu-H)_2OS_4(CO)_{13}PMe_3$	226
S.24 Fractional Coordinates for Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	236
S.25 Thermal Parameters for Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	237
S.26 Structure Factors for Os. (CO), PMe	238

xii

## LIST OF FIGURES

Fig	ure P	age
A.1	.1 Trigonal Bipyramidal Geometry	. 2
A.1	.2 Molecular Orbital Interaction Diagram for $\mathtt{ML}_5$ Complexes .	. 6
A.1	.3 Molecular Orbitals Filled by d Electrons of M(CO) <sub>5</sub> Complexes	. 8
A.1	.4 Molecular Orbital Diagram for $\pi$ -Interactions of an ML <sub>5</sub> Fragment with a $\pi$ -acceptor Ligand.	10
A.2	.1 Thermal Ellipsoid Diagram for Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	22
A.2	.2 Thermal Ellipsoid Diagram for Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	24
A.2	.3 Thermal Ellipsoid Diagram for Os(CO) <sub>4</sub> SbPh <sub>3</sub>	26
A.2	.4 Definition of Structural Angles for Five Coordinate Compounds	31
A.2	.5 Solution Infrared Spectra of a)Ru(CO) <sub>4</sub> SbPh <sub>3</sub> , b)Ru(CO) <sub>4</sub> AsPh <sub>3</sub> and c)Ru(CO) <sub>4</sub> PPh <sub>3</sub>	34
A.2	.6 Solution Infrared Spectra of a)Os(CO) <sub>4</sub> SbPh <sub>3</sub> and b)Fe(CO) <sub>4</sub> SbPh <sub>3</sub>	35
A.2	.7 Solution Infrared Spectra of a)Ru(CO) <sub>4</sub> SbMe <sub>3</sub> and b) Ru(CO) <sub>4</sub> P(OCH <sub>2</sub> ) <sub>3</sub> CMe	36
A.2	.8 Solid State Infrared Spectra of a)Ru(CO) <sub>4</sub> SbPh <sub>3</sub> and b)Os(CO) <sub>4</sub> SbPh <sub>3</sub>	37
A.2	Berry Pseudorotation Mechanism and Some Possible Reaction Coordinates	40

A.3.1 Newman Projections for M(CO) <sub>4</sub> ER <sub>3</sub> Compounds 44
B.1.1 Geometries of Tetranuclear Group 8 Clusters
B.1.2 Geometry of $HOs_3Re(CO)_{15}$
B.1.3 Frontier Orbitals for $[M(CO)_4]^{2+}$ and $M(CO)_3$
B.1.4 The Isolobal Analogy as Applied to Os(CO) <sub>4</sub> and Os(CO) <sub>3</sub> Fragments
B.2.1 Thermal Ellipsoid Diagram for Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> , Molecule 2 . 96
B.2.2 Os-Os Bond Lengths in Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> 99
B.2.3 Variable Temperature <sup>13</sup> C NMR Spectra of Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> 103
B.2.4 Fluxional Processes of Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub> 105
B.2.5 Fluxional Processes and <sup>13</sup> C NMR Spectrum of Os <sub>3</sub> (CO) <sub>11</sub> P(OMe) <sub>3</sub> 106
B.2.6 Thermal Ellipsoid Diagram for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub> 109
B.2.7 Bonding Model for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub> 112
B.2.8 Molecular Orbital Diagram for $[Os_4(CO)_{16}]^{2+}$ 114
B.2.9 Application of the Isolobal Analogy to Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub> 115
B.2.10 <sup>13</sup> C NMR Spectra of Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub> 115
B.2.11 Fluxional Mechanisms for Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub> 118
B.2.12 Thermal Ellipsoid Diagrams for $(\mu-H)_2Os_4(CO)_{13}PMe_3$ 121

B.2.13 <sup>1</sup> H NMR Spectrum and Hydride Ligand Positions for $(\mu-H)_2Os_4(CO)_{13}PMe_3$	124
B.2.14 Octahedral Geometry of the Hinge Vertices of Butterfly Structures	126
B.2.15 <sup>13</sup> C NMR Spectra for $(\mu-H)_2Os_4(CO)_{13}PMe_3$	129
B.2.16 Thermal Ellipsoid Diagram for Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	131
B.2.17 Possible Resonance Structures for Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	136
B.2.18 <sup>13</sup> C NMR Spectra of Os <sub>6</sub> (CO), 2PMe	138

## PART A

# AXIAL-EQUATORIAL ISOMERISM IN COMPLEXES $M(CO)_4L$

(M=Fe, Ru, Os; L=GROUP 15 LIGAND)

### CHAPTER 1

### INTRODUCTION

### 1.1 Overview

Five-coordinate, transition metal complexes of the type ML<sub>5</sub> (M=transition metal, L=two electron donor ligand) exist in two ideal geometries, the trigonal bipyramid (TBP) and the square In either geometry, a monosubstituted complex ML<sub>a</sub>L' pyramid. may take one of two isomeric forms. In a square pyramid, the unique ligand L' may appear at either an apical or basal position. In a TBP either axially or equatorially substituted geometries are possible (see Figure A.1.1). Most organometallic complexes of d 8 metals adopt a trigonal bipyramidal configuration and the site preference of the unique ligand in this form has been the subject of considerable theoretical and experimental interest. Several models describing the inherent



Figure A.1.1. Trigonal Bipyramidal Geometry. The unique ligand is in the a)axial and b)equatorial site.

differences of the M-L bonds in  $ML_5$  have also been proposed.<sup>1,2,3</sup> Interest has been expressed not only in the stable molecules<sup>4,5,6,7,8</sup> but also in intermediates of dissociative reactions of octahedral complexes<sup>9</sup> as well as of associative reactions of tetrahedral complexes.<sup>10</sup>

Nonrigidity involving axial-equatorial exchange has been observed for many ML<sub>1</sub>L' complexes, and the Berry TBP psuedorotation mechanism has often been proposed as the exchange mechanism.<sup>11</sup> Both theoretical<sup>12,13</sup> and experimental<sup>14</sup> studies indicate the barrier to nonrigidity is low so that which isomer is adopted by a given complex can be attributed to thermodynamic rather than kinetic constraints. Infrared spectroscopy reveals which isomer predominates in solution; an axial M(CO) L complex typically has three bands in the carbonyl stretching region while an equatorial M(CO)<sub>4</sub>L complex has four.<sup>15</sup> The short time scale of this technique  $(10^{-11}s)^{16}$  allows detection of both isomeric forms if they are present in solution.

A theme common to many studies is that a ligand possessing better  $\pi$ -acceptor properties is more likely to be found in the equatorial site.<sup>17</sup> Indeed, a search of the literature revealed numerous examples of strong  $\pi$ -acceptors such as CO, PF<sub>3</sub> or NO in the equatorial position of a TBP (see Table A.1.1, page 15). However, in most cases the unique ligand, a weak  $\pi$ -acceptor was found in the axial site. Also, the large majority of the known complexes contained a first row transition metal as the central atom. Other (theoretical) studies indicated that the  $\sigma$ -donor

ability<sup>1.18</sup> or the steric requirements<sup>2</sup> of the unique ligand might also be important in determining the site preference.

As the following literature survey illustrates there was little experimental evidence to support or refute these ideas. Also there was almost no discussion as to whether the period of the transition metal row could influence ligand site preference.

## 1.2 Literature Survey

## 1.2.1 Theoretical Papers

Without doubt, the most significant theoretical work on transition metal pentacoordination to appear in recent years is that of Rossi and Hoffmann.<sup>1</sup> As their work is so germane to this study, the appropriate sections are summarized here in some detail.

The authors cautioned that the extended Hückel calculations on which their model is based do not take into account steric or electrostatic effects. It might also be added that the calculations were performed on  $ML_5$  species where M was a third row transition metal (Pt). What effect changing M to a first or second row metal might have had on their conclusions was not discussed.

The consideration of the electronic effects began with a molecular orbital interaction diagram (reproduced in Figure A.1.2). for a trigonal bipyramidal  $ML_5$  species assuming  $\pi$  bonding ligands are absent. The low lying e',  $a_2$ " and  $a_1$ ' orbitals can be viewed as filled by ligand electrons; their nature is such that for d<sup>o</sup> systems the axial bonds are weaker than the equatorial ones. As metal d electrons are added the e" orbitals are filled first. Since this set is rigorously non-bonding, there is no effect on bond strength. The next orbitals to be filled, the e' set are weakly metal-equatorial antibonding. The antibonding nature arises from mixing of not



Figure A.1.2. Molecular Orbital Interaction Diagram for  $ML_5$ Complexes. The interactions shown here are for  $\sigma$  interactions only,  $\pi$ -bonding interactions are considered subsequently. (After Rossi and Hoffmann.<sup>1</sup>)

only ligand  $\sigma$  and metal nd orbitals but metal (n+1)p orbitals as The (n+1)p orbital mixing has the effect of hybridizing well. the orbitals away from the ligands thus weakening the orbital. interaction and stabilizing the antibonding Nevertheless, addition of metal d electrons to this e' set results in weakening of the metal-equatorial ligand bonds to the has d<sup>6</sup>-d<sup>8</sup> extent that for the cases where the central metal

configuration, they are calculated to be weaker than the metal-axial ligand bonds. The last orbital to be filled, having  $a_1$ ', symmetry, is strongly metal-axial ligand antibonding so that in a d<sup>10</sup> complex, the axial bonds are once again predicted to be weaker, (see Figure A.1.3).

It is assumed that a strong  $\sigma$ -donor would take up the position where it would form the most stable, lowest energy bond. That is, strong  $\sigma$ -donor ligands are more likely to be found at equatorial sites in complexes in which the central metal has  $d^{\circ}-d^{4}$  and  $d^{1\circ}$  electronic configuration and at axial sites if the central metal has  $d^{\circ}-d^{4}$  electronic configuration. Conversely, weak  $\sigma$ -donors are more likely found in an equatorial site in a complex having a central metal of  $d^{6}$  to  $d^{8}$  configuration.

Consideration of the effects of  $\pi$  bonding on the site preference of the ligands takes into account the change of symmetry of the molecule, and therefore of the orbitals, that results from substitution of only one ligand. A normal two electron donor,  $\pi$ -acceptor ligand, has two low-lying unoccupied orbitals of  $\pi$  symmetry. If the ligand occupies an axial site the molecular symmetry is changed from  $D_{3h}$  to  $C_{3v}$  and the  $\pi$ -acceptor orbitals on the ligand transform as  $e_x$  and  $e_y$ . If the ligand is equatorial the complex has  $C_{2v}$  symmetry and the ligand  $\pi$  orbitals transform as  $b_1$  and  $b_2$ . In this case these orbitals line up parallel and perpendicular to the original 3-fold axis of the trigonal bipyramid, repectively. The







C)



Figure A.1.3. Molecular Orbitals Filled by d Electrons of  $M(CO)_s$ Complexes. The e" orbitals shown in a) are filled first, then the e' orbitals shown in b) and finally the a' orbital shown in c). (After Rossi and Hoffmann.<sup>1</sup>)

orbitals of the ML<sub>4</sub> framework also transform differently with the change of molecular point group. From a consideration of the  $\pi$  interactions of the unique ligand orbitals and ML<sub>4</sub> framework metal d orbitals that are allowed by symmetry, Rossi and Hoffmann concluded that the equatorial perpendicular interaction is stronger than the equatorial parallel interaction which is about equal to the axial interaction. These features are displayed in the MO diagram, given in Figure A.1.4. It was concluded that when the central metal has d<sup>8</sup> electronic configuration a  $\pi$ -acceptor ligand will favour an equatorial site.

Prediction of the site preference of a real ligand must take into account both  $\sigma$  and  $\pi$  bonding. For example conclusions drawn regarding d<sup>8</sup> M(CO)<sub>a</sub>L complexes must attempt an evaluation of the relative importance of these factors since CO and many common ligands are poor  $\sigma$ -donors (preferring equatorial other sites) and good  $\pi$ -acceptors (also preferring equatorial sites). Any ligand which is both a poorer  $\sigma$ -donor and a better  $\pi$ -acceptor than CO would be predicted to occupy an equatorial site. But if only one of these conditions is satisfied the situation is less clear. For example, what would be the site preference of a very poor  $\sigma$ -donor ligand in the presence of very good  $\pi$ -acceptor ligands, i.e., are  $\sigma$  or  $\pi$  effects dominant? The structures analyzed by the authors showed longer crystal equatorial bonds for  $ML_5$  d<sup>8</sup> systems (i.e. dominance of the  $\sigma$ However there were no consistent trends for the ML\_L' effect).



axial equat

equatorial

Figure A.1.4. Molecular Orbital Diagram for  $\pi$ -Interactions of an ML<sub>4</sub> Fragment with a  $\pi$ -accectopr Ligand. (After Rossi and Hoffmann.<sup>1</sup>)

systems with d<sup>8</sup> configurations.

In summary, the work of Rossi and Hoffmann indicates that a weak  $\sigma$ -donor, or a strong  $\pi$ -acceptor ligand will occupy an equatorial site in the d<sup>8</sup> case; conversely a strong  $\sigma$ -donor, or weak  $\pi$ -acceptor will prefer the axial site. The prediction of the site preference of any particular ligand rests on a evaluation of the relative importance of these two types of bonding.

Another theoretical paper on pentacoordination is that of Burdett<sup>18</sup> who considered the angular overlap of the metal d and ligand  $\sigma$  orbitals only. He concluded that for a low-spin d<sup>8</sup> configuration the stronger  $\sigma$ -donor will prefer the axial position, and also that "within the d orbital manifold the axial bond strength was greater that the equatorial." Since there was no consideration of the effect of  $\pi$  bonding on site preference the author readily admitted "our scheme is not the whole story."

Shustorovich has written a number of papers based on perturbation theory, on substitution effects of the  $\sigma$  bonded components in molecules of various geometries.<sup>3,19,20</sup> He concluded that for d<sup>8</sup> metals, the axial site is favoured for a strong  $\sigma$ -donor. Once again, the effect of  $\pi$  bonding was not considered.

Since the question of site preference of the ligands cannot be separated from the inherently unequal nature of the axial and equatorial bonds of a TBP complex, several MO calculations on Fe(CO) and closely related molecules are also of interest.<sup>12,13,21,22</sup> The results underscore the difficulties of theoretical studies on pentacoordinate molecules. That is, the energy differences of the various conformations are small, and subject to error. The axial bonds of  $Fe(CO)_5$  have been calculated to be weaker,<sup>21</sup> stronger but much longer,<sup>13</sup> and marginally longer<sup>22</sup> than the equatorial bonds. Experimental lengths as determined in an electron diffraction study are 1.807(6) for the Fe-C<sub>ax</sub> distance and 1.827(6) for the Fe-C<sub>eq</sub>

distance.<sup>23</sup> The most recent calculations<sup>12</sup> (using an SCF method) show that a subtle change in the assumed 3d energy level brings the caculated lengths for the axial and equatorial Fe-C bonds (1.82Å and 1.83Å respectively) into close correspondence with the experimental values. This results from both a decrease in the  $p_x$  and  $p_y$  contribution to equatorial Fe-C bonds and an increased  $d_{z^2}$  bonding contribution to the a' orbital. Thus the symmetry overlap model of Rossi and Hoffmann in which the d orbitals make their main contribution to the e" and e' orbitals is only valid as a first approximation and the d orbital contribution to the lower lying bonding orbitals must also be taken into account for more precise predictions.

Unfortunately even this improved picture of the bonding in  $Fe(CO)_5$  does not provide a satisfactory explanation as to why a weak  $\sigma$ -donor ligand, such as SbPh<sub>3</sub> should be in an axial position in  $Fe(CO)_4SbPh_3^{24}$  but in an equatorial site in  $Ru(CO)_4SbPh_3$ .<sup>6</sup> Actually,  $Fe(CO)_4SbPh_3$  would be predicted to exist as the equatorial isomer on the basis of this model, since the equatorial bonds have the weaker  $\sigma$  component.

The situation with regard to steric factors is complicated since it is unclear whether axial or equatorial sites are more sterically hindered. Lichtenberger and Brown<sup>14</sup> have shown that for symmetric ER<sub>3</sub> ligands (the type used in this study) the equatorial position is more crowded. Thus ligands with large cone angles should prefer the axial position. Keiter<sup>25</sup> and Cowley<sup>26</sup> have pointed out that for large <u>asymmetric</u> ligands such

as  $PPh(PPh_2)_2$  and  $P_2(2,4,6,t-Bu_3C_6H_2)_2$  steric strain is actually eased in the equatorial position.

Favas and Kepert have developed more subtle steric reasoning . based on a mathematical summation of all possible ligand-ligand interactions.<sup>2</sup> They base their calculations on the ratio (R) of the effective bond length of the unique ligand to that of the other four ligands. The effective bond length is the distance from the metal atom to a theoretical point location of the bonding electron pair. When the one effective bond length associated with the unique ligand is shorter that the other four (e.q., R=0.8) the equatorial isomer is predicted to be stable. However, when R=1.2 the axial isomer is predicted. The crossover from the equatorial to axial form occurs at about For very low or high values of R the predicted R=1.00. stereochemistry approaches an apically substitued square pyramid a capped tetrahedron, respectively. The R values were or calculated for a number of different geometries, described in terms of the OC-M-CO and OC-M-L angles, and the results presented graphically. Thus if the structure of a complex is known its R value can be read off the appropriate graph.

Unfortunately nearly all the examples of  $M(CO)_{4}L$  type compounds cited by Favas and Kepert are axially substituted and have R values  $\approx 1.00$ . For example, the authors calculated the R value for Fe(CO)<sub>4</sub>L compounds to be  $\approx 1.00$  and thus explained the low barrier to axial-equatorial rearrangement found in <sup>13</sup>C NMR studies.<sup>27</sup> However, this does little to predict the axial

substitution of the cited examples. Nor can it explain the change in site preference for the SbPh<sub>3</sub> ligand when iron is replaced by ruthenium in  $M(CO)_4$ SbPh<sub>3</sub> (M=Fe,Ru).

In eq-Mn(CO)<sub>4</sub>NO<sup>28</sup> there is no significant distortion of the equatorial carbonyls and again an R value of  $\approx 1.0$  would be predicted. This is in contradiction to the statement of Favais and Kepert that the known geometry of this compound (equatorial substitution) implies "greater replusion (lower R values) from the nitrosyl ligand than from the other four unidentate ligands."<sup>2</sup>

In summary, it can be said that not enough work has been done to build up a sizeable set of self-consistent R values and in most cases, the R values are calculated from the structure, rather than the structure predicted on the basis of known R values. This has led to some contradictory results. The work of these authors is rarely cited by other workers in the field suggesting their method has not gained wide acceptance.

### 1.2.2 Experimental Studies

Table A.1.1 lists 46 known d<sup>8</sup> transition metal complexes of the type  $M(CO)_{4}L$ . Analysis of these data and the related literature points out that both experimental confirmation and a wide understanding of the theoretical predictions summarized above are lacking. Most of the complexes are of a first row transition metal and the unique ligand, a relatively poor  $\pi$ -acceptor, occupies an axial position. While this is

Table A.l.l. Previously Known M(CO)<sub>4</sub>L Derivatives.

Compound	Substitution Site	Ref.
$[Mn(CO)_4PPh_3]^-$	axial <sup>a</sup>	29
Mn (CO) 4 NO	equatorial <sup>a</sup>	28
$[Fe(CO)_4CN]^-$	axial <sup>a</sup>	8
Fe(CO) <sub>4</sub> L (L=PPh <sub>3</sub> , AsPh <sub>3</sub> , SbPh <sub>3</sub> , PPh <sub>2</sub> Me, PPhMe <sub>2</sub> , P(C <sub>6</sub> H <sub>11</sub> ) <sub>3</sub> P(n-Bu) <sub>3</sub> , P(OPh) <sub>3</sub> , P(OEt) <sub>3</sub> , P(OMe) <sub>3</sub> )	axial	30
Fe(CO) <sub>4</sub> L (L=PPh <sub>3</sub> , CNMe, CNEt, CNPh, CNBu <sup>t</sup> )	axial	31
<pre>Fe(CO)<sub>4</sub>P{(N(SiMe<sub>3</sub>)<sub>2</sub>} {C(SiMe<sub>3</sub>)<sub>2</sub>}</pre>	equatorial	32
$Fe(CO)_4SbMe_3$ , $Fe(CO)_4AsMe_3$	axial <sup>a</sup>	33
$Fe(CO)_4L$ (L=PEt <sub>3</sub> , Ph <sub>2</sub> P(CH <sub>2</sub> ) <sub>2</sub> PPh <sub>2</sub> , C <sub>2</sub> H <sub>5</sub> NC)	axial	34
$Fe(CO)_{4}L(L=C_{5}H_{5}N, C_{5}H_{4}N_{2})$	axial	35
Fe(CO) <sub>4</sub> PCl <sub>3</sub>	mixture	36
Fe(CO) <sub>4</sub> PF <sub>3</sub>	mixture	37
$Fe(CO)_{4}L$	mixture	7
$L=PF[(OC)(CF_{3})_{2}CN]_{2}$ $L=P[(OC)(CF_{3})_{2}CN]_{3}$ $L=PF_{2}OCNC_{2}(CF_{3})_{4}O$	mostly equatorial equatorial mixture	
$Fe(CO)_4 P(NMe_2)_3$	axiala	38
$Fe(CO)_{4}(\eta^{1}-PPh_{2}CH_{2}CH_{2}PPh_{2})$	axial <sup>a</sup>	25
$Fe(CO)_4SC_3Ph_2$	axial	2
[Fe(CO) <sub>4</sub> SnR <sub>2</sub> ] <sup>2-</sup>	axial	39

Ru(CO) <sub>4</sub> L (L=PPh <sub>3</sub> , PMePh <sub>2</sub> , PBu <sub>3</sub> )	axial	40
$Ru(CO)_4P(OCH_3)_3$	axial <sup>a</sup>	<b>4</b> 1
$Ru(CO)_{4}SbPh_{3}$	equatorial <sup>a</sup>	6
$Ru(CO)_4 PF_3$	mixture <sup>b</sup>	<b>42</b> .
$Os(CO)_4 PPh_3$	axial	43
$Co(CO)_{4}SiR_{3}$ (R=H, F, Cl)	axiala	14,44
Co(CO) <sub>4</sub> GeCl <sub>3</sub>	axial	2
Co(CO) AuPPh 3	axial	2

<sup>a</sup>Solid state crystal structure determined by X-ray crystallography.

<sup>b</sup>Very poorly characterized.

consistent with the theories outlined above it can only be taken as partial confirmation of them as there are so few equatorial isomers. Also very little attempt has been made to discuss both the  $\pi$  and  $\sigma$  properties of the unique ligands. Indeed,  $\pi$  bonding arguments alone have been used by a number of authors to explain site preference.<sup>8,29,38</sup> Several other studies dealt with other aspects of the chemistry of M(CO)<sub>4</sub>L complexes rather than the observed geometry.<sup>30,31,33,34,44</sup>

Reasoning based on  $\pi$ -bonding alone or in conjuction with steric factors was also used in a few cases where equatorial isomers were found.<sup>7,45</sup> In these examples, Fe(CO)<sub>4</sub>P[(OC)(CF<sub>3</sub>)<sub>2</sub>CN]<sub>3</sub> and Fe(CO)<sub>4</sub>PF<sub>3</sub>, the unusually high values of the carbonyl stretching frequencies were consistent

with the unique ligand having exceptional  $\pi$ -acceptor ability.

Steric arguments alone were used to explain why the unique ligand in both  $Fe(CO)_{4}P_{2}(2,4,6,t-Bu_{3}C_{6}H_{2})_{2}^{26}$  and  $Fe(CO)_{4}P\{N(SiMe_{3})_{2}\}\{C(SiMe_{3})_{2}\}^{32}$  adopts an equatorial position. In both cases the ligand is large and asymmetric.

The compound eq-Mn(CO)<sub>4</sub>NO is the subject of some controversy in the literature; the original authors<sup>28</sup> suggested the data were not consistent with NO being a better  $\pi$ -acceptor than CO while later workers<sup>8</sup> claimed the opposite.

A few authors did note that the relative  $\sigma$ -donor/ $\pi$ -acceptor ratio of the ligands is important in determining the geometry.<sup>25,35</sup> However in no case was there any attempt to rate the relative importance of these factors separately.

The situation for the nine listed compounds of the second and third row metals is no better. Of these, seven are axially substituted, and no explanation was given for the site preference.<sup>40.41.43</sup> Of the remaining two,  $Ru(CO)_4PF_3$  probably exists as a mixture of isomers in solution but it was very poorly characterized.<sup>42</sup> The other complex,  $Ru(CO)_4SbPh_3$ , was shown to exist as the equatorial form in the solid state, but the authors were unable to rationalize that obervation.<sup>6</sup> It will be shown later that their interpretation of the solution infrared spectrum of this compound was incorrect.

summary, it can be said that the preference of strong In  $\pi$ -acceptor ligands to occupy equatorial sites while weak  $\pi$ -acceptors are found in axial positions is widely accepted and illustrated by numerous examples. The role of  $\sigma$  bonding in influencing site preference is well documented in the theoretical literature but rarely cited in papers concerned with synthetic chemistry. Little effort has been made to rank the relative importance of the two types of bonding, i.e. to predict the site preference of a weak  $\sigma$ -donor in the presence of strong  $\pi$ -acceptors. The possibility of first, second, or third row transition metals having different tendencies to give equatorial or axial isomers has received little attention.

## 1.3 Project Description

It was decided to undertake a study of the site preference of the unique ligand in d<sup>8</sup> transition metal complexes of the iron triad of the type  $M(CO)_{4}L$  (M=Fe,Ru,Os; L=group 15 ligand). These complexes seemed a suitable target group since they exist as TBP's and from the one study on  $Ru(CO)_{4}SbPh_{3}$  were expected to exhibit axial-equatorial isomerism.

Interest in this project was stimulated by the knowledge that relatively few second and third row TBP complexes were known, and few of these were equatorially substituted. The structure of  $Ru(CO)_4SbPh_3$  was of special interest since it contained a second row transition metal and the unique ligand

(not known as a strong  $\pi$  acid) was equatorial.<sup>6</sup> In the Fe analogue it was axial.<sup>24</sup> Convenient synthetic routes to Ru(CO), and Os(CO), had been developed in this laboratory 46 and it was thought that this would allow facile synthesis of Ru(CO) L and Os(CO) L derivatives. It was hoped that new equatorial isomers could be synthesized and that this would lead to a better understanding of the question of site preference in general. The existence of eq-Ru(CO) SbPh, suggested that attempts to synthesize Os(CO) SbPh3 might be a useful starting point. λs the work proceeded it became apparent that compounds of the type  $M(CO)_{\mu}L$  (M=Fe, Ru, Os; L=ER<sub>3</sub>, E=P, As, Sb: R=Ph, Me:  $L=P(OCH_2)_3CMe)$  would be synthetically accessible and this would allow a systematic study of the influence of both the ligand and the central metal on the site preference.

### CHAPTER 2

### RESULTS

Several derivatives of the type M(CO) L (M=Fe,Ru,Os; L=group 15 ligand) have been prepared.<sup>47</sup> The ruthenium trimethylphosphine derivative, Ru(CO), PMe<sub>3</sub>, was prepared by the action of PMe<sub>3</sub> on  $Ru_3(CO)_{12}$  at 120 °C under 75 atmospheres of CO, a method used previously  $Ru(CO)_{4}P(OMe_{3})$ .<sup>41</sup> The osmium analogue was for synthesized in a similar manner only under more forcing conditions (280 °C, 200 atm CO). All other derivatives were prepared in hexane from  $M(CO)_5$  and the appropriate ligand. With  $Ru(CO)_5$  the reaction proceeded at a convenient rate at room temperature whereas for  $Os(CO)_5$  temperatures of 80 °C or above This illustrates the greater lability of were necessary. carbonyls when bonded to the second row transition metal, a property previously observed with this group of metals in M(CO)<sub>4</sub>(SiCl<sub>3</sub>)<sub>2</sub> derivatives.<sup>48</sup> For osmium, and to a lesser extent, ruthenium, there were competing reactions in the formation of the  $M(CO)_{4}L$  complexes which gave  $M_{3}(CO)_{12}$  and other clusters. The poor yields of Os(CO)<sub>4</sub>L were marginally improved by carrying out the reactions under carbon monoxide. The colour of the complexes ranged from the orange Ru(CO)<sub>4</sub>SbPh<sub>3</sub> to the white M(CO)<sub>4</sub>PMe<sub>3</sub> species. They could be handled in air for short periods but decomposed upon prolonged exposure.
## 2.1 Structural Studies

structures of ax-Ru(CO) AsPh<sub>3</sub>, ax-Ru(CO) SbMe<sub>3</sub> and The conventional determined by X-ray eg-Os(CO),SbPh, were diffraction techniques. Views of each molecule showing the thermal ellispoids and the atomic numbering scheme for each are given in Figures A.2.1, A.2.2 and A.2.3 respectively; selected bond lengths and angles are given in Tables A.2.1, A.2.2 and A.2.3. Although the structure was not determined, the compound  $Os(CO)_{4}AsPh_{3}$  was found to be isostructural with  $Ru(CO)_{4}AsPh_{3}$ . It can be seen that of the compounds studied here, Ru(CO)<sub>4</sub>AsPh<sub>3</sub>, Os(CO)<sub>4</sub>AsPh<sub>3</sub> and Ru(CO)<sub>4</sub>SbMe<sub>3</sub> adopt the axial form in the solid state while only Os(CO)<sub>4</sub>SbPh<sub>3</sub> (as determined in this study) and Ru(CO)<sub>4</sub>SbPh<sub>3</sub> (as determined by Forbes and coworkers<sup>6</sup>) adopt the equatorial form. This represents the first occasion where a site preference switchover for a closely related series of five-coordinate complexes has been observed. The reasons for this will be discussed subsequently, however there are several interest about the structures which are other points of considered now.

The Os-Sb bond length of 2.612(2)Å<sup>49</sup> appears to be the first reported in the literature for a molecular compound. It may be compared with the Os-Sb bond lengths in OsSb<sub>2</sub> (2.639(3)Å and 2.644(4)Å),<sup>50</sup> and the Ru-Sb distance of 2.623(4)Å in Ru(CO)<sub>4</sub>SbPh<sub>3</sub>.<sup>6,51</sup> The Ru-Sb distance of 2.619(1)Å found for Ru(CO)<sub>4</sub>SbMe<sub>3</sub> may likewise be compared to these lengths. The



Figure A.2.1. Thermal Ellipsoid Diagram for Ru(CO) AsPh3

**Table A.2.1** Selected Molecular Dimensions for Ru(CO)<sub>4</sub>AsPh<sub>3</sub>. a)Bond Lengths

Length (Å)	Corrected Length (Å)	
2.461(1)	2.507 <sup>a</sup>	
1.918(5)	1.930 <sup>b</sup>	
1.915(5)	1.921 <sup>b</sup>	
1.921(5)	1.932 <sup>b</sup>	
1.890(5)	1.906 <sup>b</sup>	
1.140(7)	1.153 <sup>C</sup>	
1.147(6)	1.160 <sup>C</sup>	· · · · ·
1.132(7)	1.146 <sup>C</sup>	
1.153(6)	1.168 <sup>C</sup>	
1.938(5)		
1.932(5)		× •
1.925(5)		
	Length (Å) 2.461(1) 1.918(5) 1.915(5) 1.921(5) 1.921(5) 1.140(7) 1.147(6) 1.147(6) 1.132(7) 1.153(6) 1.938(5) 1.932(5) 1.925(5)	Length (Å) Length (Å) 2.461(1) 2.507 <sup>a</sup> 1.918(5) 1.930 <sup>b</sup> 1.915(5) 1.921 <sup>b</sup> 1.921(5) 1.932 <sup>b</sup> 1.890(5) 1.906 <sup>b</sup> 1.140(7) 1.153 <sup>C</sup> 1.147(6) 1.160 <sup>C</sup> 1.132(7) 1.146 <sup>C</sup> 1.153(6) 1.168 <sup>C</sup> 1.938(5) 1.932(5) 1.925(5)

b)Bond Angles

Bond	Angle (°)	Bond	Angle (°)
C(1)-Ru-C(2)	117.9(2)	O(2)-C(2)-Ru	178.6(4)
C(1) - Ru - C(3)	118.5(2)	O(3)-C(3)-Ru	178.1(5)
C(1)-Ru-C(4)	92.5(2)	O(4)-C(4)-Ru	179.2(5)
C(2)-Ru-C(3)	123.4(2)	C(11)-As-C(21)	103.1(2)
C(2)-Ru-C(4)	90.0(2)	C(11)-As-C(31)	103.0(2)
C(3)-Ru-C(4)	91.5(2)	C(21)-As-C(31)	103.3(2)
As-Ru-C(1)	89.0(1)	$C_{ph}^{-As-C}_{ph}^{d}$	103.1
As-Ru-Č(2)	89.1(1)	Ru-As-C(11)	115.4(1)

As-Ru-C(3)	88.0(1)	Ru-As-C(21)	115.1(1)
As-Ru-C(4)	178.5(2)	Ru-As-C(31)	115.2(1)
O(1)-C(1)-Ru	177.9(5)	Ru-As-C <sup>d</sup> Ph	115.2
c)Torsion Angles Bonds		Angle (°)	
C(11)-As-Ru-C(3)		34.24	
C(21)-As-Ru-C(2)	`) ``	37.67	
C(31)-As-Ru-C(1)		35.57	

<sup>a</sup>Atoms assumed to move independently.

bSecond atom assumed to ride on the first.

<sup>c</sup>Both atoms assumed to ride on Ru.

d<sub>Mean</sub> value.



Figure A.2.2. Thermal Ellipsoid Diagram for Ru(CO)<sub>4</sub>SbMe<sub>3</sub>

**Table A.2.2.** Selected Molecular Dimensions for Ru(CO)<sub>4</sub>SbMe<sub>3</sub> a)Bond Lengths

Atoms	Length (Å)	Corrected Length (Å)
Ru-Sb	2.6187(9)	2.6578 <sup>a</sup>
Ru-C(1)	1.89(2)	1.90 <sup>b</sup>
Ru-C(2)	1.92(1)	1.94 <sup>b</sup>
Sb-C(3)	2.140(7)	
C(1)-O(1)	1.16(2)	1.17 <sup>C</sup>
C(2)-C(2)	1.13(1)	1.15 <sup>C</sup>
b)Bond Angles Bond	Angle (°)	
C(1)-Ru-C(2)	91.9(2)	
C(2)-Ru-C(2)	119.9(3)	·
Ru-Sb-C(1)	180.0(-)	
Ru-Sb-C(3)	115.7(2)	
C(3)-Sb-C(3)	102.6(3)	
Ru-C(2)-O(2)	178.3(9)	

<sup>a</sup>Atoms assumed to move independently.

<sup>b</sup>Second assumed to ride on first atom.

<sup>C</sup>Both atoms assumed to ride on Ru.





Table A.2.3. Selected Molecular Dimensions for Os(CO)<sub>4</sub>SbPh<sub>3</sub>. a)Bond Lengths

Atoms	Length (Å)	Corrected Length (Å)	
Os-Sb	2.612(2)	2.649 <sup>a</sup>	
Os-C(1)	1.926(5)	1.934 <sup>b</sup>	
Os-C(2)	1.911(7)	1.933 <sup>b</sup>	
Os-C(3)	1.952(6)	1.980 <sup>b</sup>	
Os-C(4)	1.939(6)	1.945 <sup>b</sup>	
C(1)-O(1)	1.134(7)	1.148 <sup>C</sup>	
C(2)-O(2)	1.148(8)	1.164 <sup>C</sup>	
C(3)-O(3)	1.117(8)	1.138 <sup>C</sup>	
C(4)-O(4)	1.134(7)	1.150 <sup>C</sup>	•
Sb-C(11)	2.131(5)		
Sb-C(21)	2.136(5)		~
Sb-C(31)	2.124(5)		
b)Bond Angles			
Bond	Angle (°)	Bond	Angle (°)
C(1)-Os-C(2)	136.4(3)	0(2)-C(2)-Os	175.6(6)
C(1)-Os-C(3)	90.7(3)	0(3)-C(3)-Os	178.6(8)
C(1)-Os-C(4)	88.8(2)	O(4)-C(4)-Os	177.1(5)
C(2)-Os-C(3)	88.6(3)	C(11)-Sb-Os	114.0(1)
C(2)-Os-C(4)	87.6(3)	C(21)-Sb-Os	118.4(1)
C(3)-Os-C(4)	173.8(3)	C(31)-Sb-Os	121.2(1)
Sb-Os-C(1)	107.0(2)	C <sub>Ph</sub> -Sb-Os <sup>d</sup>	117.9
Sb-Os-C(2)	116.6(2)	C(11)-Sb-C(21)	100.1(2)

Sb-Os-C(3)	90.7(2)	C(11)-Sb-C(31)	99.0(2)
Sb-Os-C(4)	95.4(2)	C(21)-Sb-C(31)	100.5(2)
O(1)-C(1)-Os	176.9(5)	C <sub>Ph</sub> -Sb-C <sub>Ph</sub> d	99.9
c)Torsion Angles Bonds		Angle (°)	
C(21)-Sb-Os-C(3)		22.97	
C(31)-Sb-Os-C(4)	Ϋ́	31.03	

<sup>a</sup>Atoms assumed to move independently.

<sup>b</sup>Second atom assumed to ride on the first.

<sup>C</sup>Both atoms assumed to ride on Ru.

d<sub>Mean</sub> value.

Ru-As bond length of 2.461Å is somewhat longer than the Ru-As bond literature which are in lengths in the the range 2.401(3)-2.445(2)Å.<sup>52.53</sup> However, the literature values are for bidentate arsenic ligands in cluster compounds so that it would be unwise to attach any significance to the differences. The C(1)-Os-Sb angle (107.0(2)°) in eq-Os(CO)<sub>4</sub>SbPh<sub>3</sub> is significantly smaller than the C(2)-Os-Sb angle (116.6(2)°). This difference difficult to rationalize. A similar inequality of seemingly is equivalent angles was also present in Co(PPh<sub>2</sub>Me)<sub>2</sub>Cl<sub>2</sub>NO.<sup>54</sup>

There has been considerable discussion<sup>8,28,29,38</sup> regarding differences in  $M-C_{eq}$  and  $M-C_{ax}$  bonds in  $Fe(CO)_{4}L$  and similar complexes. When L=phosphine the bonds are shortened as compared to those of the parent pentacarbonyl<sup>23</sup> with greater contraction of the

			· _	
Compound	M-C <sub>eq</sub> (Å)a	M-C <sub>ax</sub> (Å)	Ref.	
Fe(CO) <sub>5</sub> <sup>b</sup>	1.827(6)	1.807(6)	23	
Fe(CO) <sub>5</sub> <sup>C</sup>	1.795(20)	1.795(20)	55	
$Fe(CO)_4 PPh_3$	1.795(2)	1.795(2)	29	
$Fe(CO)_4P(NMe_2)_3$	1.787(5)	1.793(6)	38	
$Fe(CO)_4SbPh_3$	1.787(4)	1.765(4)	24	
$Fe(CO)_{4}Sb(Bu^{t})_{3}$	1.794(7)	1.748(8)	56	
$[Fe(CO)_4CN]^-$	1.768(8)	1.723(8)	8	
Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	1.918(5)	1.909(5)	this work	
Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	1.92(1)	1.89(2)	this work	
$Ru(CO)_{4}SbPh_{3}$	1.932(6)	1.929(6) <sup>a</sup>	6	
Os(CO) <sub>4</sub> SbPh <sub>3</sub>	1.919(6)	1.946(6) <sup>a</sup>	this work	

Table A.2.4. M-C Distances in Fe(CO)<sub>5</sub> and M(CO)<sub>4</sub>L Compounds

<sup>a</sup>Mean value

<sup>b</sup>Electron diffraction study

<sup>C</sup>X-ray diffraction study

 $Fe-C_{eq}$  bonds as compared to the  $Fe-C_{ax}$  bond (see Table A.2.4). The normal explanation for these observations is that replacement of the strongly  $\pi$ -accepting CO by a poorer  $\pi$ -bonding ligand reduces competition for the metal  $\pi$  electron density so that the other CO ligands, particularly the equatorial ones, may form stronger Fe-C  $\pi$  bonds. This can be seen in the structures of  $Fe(CO)_{4}PPh_{3}$  and  $Fe(CO)_{4}P(NMe_{2})_{3}$  both of which have nearly equal Fe-C<sub>eq</sub> and Fe-C<sub>ax</sub> bond lengths. When the ligand is SbPh<sub>3</sub>,

 $Sb(t-Bu)_3$  or  $CN^-$ , the overall shortening of the Fe-C bonds can be seen, but the axial and equatorial bonds are significantly different. Presumably any directional  $\pi$  bonding effect causing shortening of Fe-C bonds is accompanied by a reduced trans influence of the unique ligand causing shortening of the Fe-Car bond as well. For the compounds studied here comparisons with the parent carbonyls cannot be made since the structures of  $Ru(CO)_5$  and  $Os(CO)_5$  are not known. In the compounds  $Ru(CO)_{4}AsPh_{3}$  and  $Ru(CO)_{4}SbMe_{3}$  the mean M-C length is longer than the  $M-C_{ax}$  length, while in  $Os(CO)_4SbPh_3$  the opposite is true. However, in these compounds and in eq-Ru(CO)<sub>4</sub>SbPh<sub>3</sub> and in Fe(CO), PPh<sub>3</sub> noted above, the distances are not significantly different. In light of this, and the fact that the X-ray structure of Fe(CO)<sub>5</sub> had large estimated standard deviations and showed nearly equal Fe-C and Fe-C lengths<sup>55</sup> which were shorter than the distances found in the electron diffraction study, it seems hazardous to draw any firm conclusions on this question.

Finally it should be noted that although the distortion from TBP geometry in the equatorially substituted complexes is significant, assignment of square pyramid geometry would not be appropriate. According to criteria developed by Holmes,<sup>57</sup>  $Os(CO)_{4}SbPh_{3}$  has an axial angle,  $\theta_{15}$ , of 173.8°, and an equatorial angle,  $\theta_{24}$ , of 136.4° and a dihedral angle,  $\delta_{24}$ , of 36.6° (see Figure A.2.4). This places the complex considerably more than half-way along a Berry type coordinate along which the



Figure A.2.4. Definition of Structural Angles for Five Coordinate Compounds. The angle  $\delta_{24}$  is that between the normals of the 1,2,4 and 2,4,5 planes. (After Holmes.<sup>57</sup>)

 $\delta_{24}$  angles varies smoothly from 0° for a square pyramid to 53.1° for an ideal trigonal bipyramid.

# 2.2 Infrared Studies

Solution infrared spectra in the carbonyl stretching region are illustrated in Figures A.2.5, A.2.6 and A.2.7 and summarized in Table A.2.5. The assignment of peaks to either the axial or equatorial isomer was done on the following basis. When only three carbonyl stretches were present these were assigned to the axial isomer, in accord with the characteristic pattern for M(CO)<sub>4</sub>L complexes of C<sub>3v</sub> symmetry.<sup>15,58</sup> Similar peaks in solutions of compounds displaying more than three carbonyl stretches were also assigned to the axial isomer with the additional peaks being assigned to the equatorial isomer. No absorptions were assigned to possible isomers adopting a square pyramid geometry since few d<sup>8</sup> complexes of this type are known and theoretical predictions have been made to suggest this form is unlikely.<sup>59</sup> Futhermore, in the solid state M(CO)<sub>4</sub>SbPh<sub>3</sub> (M=Ru,Os) adopt the equatorial form; it is just these species which show the most intense absorptions of the second isomer in solution. The maximum number of peaks assigned to the equatorial isomer in any solution was three although four infrared active CO stretches are expected for an M(CO)<sub>4</sub>L molecule displaying  $C_{2v}$  symmetry. It is thought that, since the infrared spectrum of no solution indicated the presence of the equatorial isomer to the exclusion of the axial isomer, the unobserved peak, or peaks, of the equatorial form were degenerate with those due to the axial molecule. The solid state infrared spectra of both eq-Ru(CO)<sub>4</sub>SbPh<sub>3</sub> and

Compounds.
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M(CO)
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i∍CNMR<sup>b</sup> (ppm) m.p. °C<sup>C</sup>

Compound			v(co)o	61 - E			ijC NMR <sup>b</sup> (ppm)	т.р. •С <sup>С</sup>	
	edq	axe	еq	âX	bə	ax			
Fe(CO).PMe,		2051		1978		1937	214.2(20.8)		
Fe(CO) *P(OCH;);CMe		2070		1997		1970	212.2(25.5)		
Fe(CO).PPh,		2052		1979		1947	209.0(18.7)		
fe(CO).AsPh,		2051		1977		1947	213.7		
Fe(CO).SbPh,	•	2048		1976		1946	212.7		
Ru(CD).PMe,		2061		1985		1947	205.3(2.4)	84-86	
Ru(CO) + P(OCH + ) + CMe	2093	2076	2014	2006		1976	203.3(10.5)	95	
Ru(CO) • PPh •		2062		1988		1955	204.6(3.5)		
Ru(CO)48Ph;	2079	2061	1999	1986		1958	204.7	168	
ru(co).SbMe;	2071	2060	1988	1988	1965	1952	203.4	67-71	
Ru(CO).SbPh3	2078	2060	1999	1987	1967		207.4		
Os(CO).PMe,		2061		1980		1939	189.9(4.5)	1	1.1
0s(CO).P(0CH,),CMe	2092	2076	2009	2001		1967	186.7(12.5)	J	
0s(CO),PPh,		2061		1983		1946	189.4(6.0)		
0s(CO),AsPh;	2077	2061	1991	1981		1947	188.7	152-154	
Os(CO),SbMe,	2069	2058		1977	1955	1942	188.2	103-104	
0s(CO),SbPh,	2076	2060	1992	1978	1957	1950	190.1	104	
Ru(CO).SbPh,f	2078		1997		1980, 194	О			
0s(CO),SbPh, <sup>f</sup>	2076		1987		1973, 193	7			
									1

<sup>a</sup>All solution spectra recorded in hexane.

<sup>b</sup>Spectra recorded at room temperature in CD<sup>1</sup>Cl<sup>1</sup>/CH<sub>1</sub>Cl<sup>1</sup> (1:5) or CD<sub>1</sub>Cl<sup>1</sup> soln.; J<sub>PC</sub> (Hz) in parenthesis. <sup>C</sup>New compounds only.

I

d<sub>Equatorial isomer.</sub>

<sup>e</sup>Axial isomer.

<sup>f</sup>Solid state spectrum in KBr disk.



scale of spectrum c) is reduced by a factor of two compared to that of a) and b). and The b)Ru(CO) AsPh<sub>3</sub> Bands due to the equatorial isomer are marked with an asterisk. Solution Infrared Spectra of a)Ru(CO)<sub>4</sub>SbPh<sub>3</sub>, Figure A.2.5. c)Ru(CO)<sub>4</sub>PPh<sub>3</sub>. wavenumber











eq-Os(CO)<sub>4</sub>SbPh<sub>3</sub> did display four bands (Figure A.2.8.) These solid state spectra confirm that the structures determined by X-ray diffraction were representative of the bulk solid state structure and were not the chance result of single crystal analysis.

The short time scale<sup>16</sup> of infrared spectroscopy allowed the existence of two isomers in solution to be displayed for all the Ru and Os derivatives except when L was PMe<sub>3</sub>, see Table A.2.5. It was hoped that low temperature studies using the method of Hartman and co-workers<sup>60</sup> would reveal a shift in the equilibrium of the two isomers and hence allow the calculation of thermodynamic parameters. Accordingly the area of the absorbance peaks of Ru(CO)<sub>a</sub>SbPh<sub>3</sub> at 2078 cm<sup>-1</sup>(ax) and 2060 cm<sup>-1</sup>(eq) were measured at temperatures ranging from ambient to -90 °C. Similar attempts were made for Os(CO) SbPh, and Ru(CO)<sub>4</sub>SbMe<sub>3</sub> but no linear relationship between the band areas could be detected and this avenue of investigation was abandoned.

# 2.3 <u>NMR</u> <u>Studies</u>

While the solution infrared spectra clearly displayed the presence of two isomers in solution for many of the  $M(CO)_{a}L$  complexes, the <sup>13</sup>C NMR spectra revealed only a single resonance in all cases, except that <sup>31</sup>P coupling was observed when the complex contained a phosphorus donor ligand. (The  $J_{P-C}$  values

are noted in brackets in Table A.2.5.) The indication is clearly that not only are the axial and equatorial carbonyls exchanging rapidly in each isomer but also the isomeric forms must be rapidly interconverting. This would be the expected result if the exchange occurred by the Berry pseudorotation (BPR) mechanism.<sup>11</sup> Such a mechanism has been previously proposed many times to account for the nonrigidity of five coordinate molecules.<sup>45,61</sup> It seems likely to be occurring in the complexes studied here because the equatorial isomer can be detected for many of the species. If a BPR mechanism were operative the free energy change upon rearrangement could be represented as in It would be expected that the energy difference Figure A.2.9. between the equatorially substituted molecule (3) and the axial isomer (1 and 5) would be reflected in the ratio of isomers displayed in the infrared spectra. Where the ax/eq ratio is nearly one, the energy level of the equatorial form would be nearly equal to that of the axial form (case A). When only the axial form can be detected in solution the energy difference would be large, (case B) i.e., the equatorial form is best considered as an intermediate. Slowed exchange would be expected if the initial activation barrier were large (cases C and D), or if the equatorial isomer were highly unfavoured (case E).

Whereas it was previously noted that  $Fe(CO)_{4}PPh_{3}^{62}$  and several other  $Fe(CO)_{4}L^{7}$  complexes showed no signs of slowed exchange at low temperatures it was hoped that this would not be



Figure A.2.9. Berry Pseudorotation Mechanism and Some Possible Reaction Coordinates. (After Lichtenberger and Brown,<sup>14</sup>)

•

the case for the Ru and Os derivatives. Much higher barriers to nonrigidity had been observed for an octahedral system of M(CO), (EMe<sub>3</sub>)<sub>2</sub> (E=Si,Ge,Sn,Pb) complexes when M=Ru or Os than when M=Fe.<sup>63</sup> In this case both cis and trans isomers could be detected in the <sup>13</sup>C NMR spectra indicating a reaction diagram as in C would be applicable. A variable temperature <sup>13</sup>C NMR study of ax-Co(CO) EX; (E=C,Si,Ge,Sn,Pb; а series on X=F,Cl,CH<sub>3</sub>,CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>,C<sub>6</sub>H<sub>5</sub>) derivatives found slow exchange at low temperatures.<sup>14</sup> Coalescence temperatures varying from -10 °C to <-160 °C were observed. (In this case only the axial form was detected in the low temperature limit so that the diagrams D or It was found that higher barriers to might apply.) Е non-rigidity were associated with smaller group 14 donor atoms. For example, Co(CO)<sub>4</sub>CF<sub>3</sub> had a coalescence temperature of -10 °C.

The solution  ${}^{13}C$  NMR spectra of  $Ru(CO)_4P(OCH_2)_3CMe_3$ , Ru(CO),SbPh, and Os(CO),SbPh, were studied to very low temperatures (-100 °C, -110 °C, and -100 °C respectively), but all cases the carbonyl resonance remained a sharp singlet or in doublet, (the  ${}^{13}CO$  signal for  $Ru(CO)_{4}P(OCH_{2})_{3}CMe$  is coupled to P). Therefore kinetic parameters for axial-equatorial exchange could not be obtained. Valuable mechanistic information could have been obtained had the exchange been slowed on the NMR time scale because the Berry pseudorotation mechanism predicts simultaneous collapse of the signals of each isomer. This has yet to be observed for any system.

#### CHAPTER 3

#### DISCUSSION

It can be seen from Figures A.2.1 to A.2.3, A.2.5 to A.2.7 and Table A.2.5 that the tendency to give equatorial isomer is Ph<sub>3</sub>Sb>Ph<sub>3</sub>As>Ph<sub>3</sub>P and Ru>Os>>Fe. Also, as far the as ligand are concerned, (OCH<sub>2</sub>)<sub>3</sub>CMe must be substituents on the considered the most likely to give an equatorial isomer since it alone displays the existence of that form in solution when attached to phosphorus. The ordering for the ligand substituents is then SbPh<sub>3</sub>>SbMe<sub>3</sub> (As ligands would probably display similar behaviour) and P(OCH<sub>2</sub>)<sub>3</sub>CMe>PPh<sub>3</sub>,PMe<sub>3</sub>. The site preference switchover from axial to equatorial isomers in the solid state occurs for M=Ru, Os when the unique ligand is SbPh<sub>3</sub>. It was found that it was exactly those compounds which displayed the most intense solution IR bands for the equatorial isomer that adopted this form in the solid state.

It is clear that the tendency to give equatorial isomer depends both on the nature of the ligand and on the central metal. Each of these factors is discussed below, in terms of both steric and electronic ( $\sigma$  and  $\pi$  bonding) considerations.

### 3.1 Steric Considerations

It seems fairly easy to discount steric factors as the cause of the observed switchover of the solid state configurations and

the variation of the axial/equatorial isomer ratio of in solution. As mentioned earlier, Lichtenberger and Brown<sup>14</sup> have shown that ER<sub>3</sub> type ligands are more sterically crowded in the equatorial position than in the axial position, even though only two close encounters with CO groups at 90° are expected for the unique ligand when it is in an equatorial position. The reason is the inability of the three R groups to adopt a fully staggered conformation with respect to the two CO ligands. This is illustrated in the Newman projections in Figure A.3.1. We also find that the closest non-bonded contacts do indeed occur in the equatorial compound Os(CO) SbPh ,  $(C(1) \cdots H(12)=3.09Å)$ . Furthermore, calculations on a model of eq-Os(CO)<sub>4</sub>AsPh<sub>3</sub> using coordinates from ax-Ru(CO)<sub>4</sub>AsPh<sub>3</sub> and eq-Os(CO)<sub>4</sub>SbPh<sub>3</sub> indicate two contacts between the group 15 ligand and carbonyls that are slightly less than the sum of the van der Waals radii for the atoms involved (C(1)...H(12)=2.79Å:  $C(4) \cdots H(36) = 2.83$ Å).<sup>64</sup> Within  $ax - Ru(CO)_4$ AsPh<sub>3</sub> the comparable are at this sum  $(C(3) \cdots H(26)=3.16Å.)$  (The closest contacts nonbonded contacts between the hydrogens and oxygens were  $O(1) \cdots H(32) = 3.02$ Å for  $ax - Ru(CO)_{\mu}AsPh_{3}$  and  $O(1) \cdots H(12) = 3.04$ Å for eq-Os(CO) SbPha.)

Given that the equatorial site is more sterically crowded it is to be expected that a ligand having a larger cone angle would be more likely to occupy the axial position. In the case of  $Ru(CO)_4SbR_3$  (R=Me, Ph) the reverse is found. When R=Me (estimated cone angle=118°) the compound crystallizes as the



Figure A.3.1. Newman Projections for  $M(CO)_4 ER_3$  Compounds (After Lichtenberger and Brown.<sup>14</sup>)

axial isomer whereas when R=Ph (estimated cone angle=145°) the equatorial isomer is observed. (The cone angles of these ligands were considered to be equal to the corresponding phosphorus analogues.<sup>65</sup>) The solution IR spectra are consistent with there being more axial isomer in solution for R=Me than for Large ligands are known to occupy both equatorial sites, R=Ph.  $Fe(CO)_{a}P[OC(CF_{3})_{2}CN]_{3}$ , and axial sites as in in as  $Fe(CO)_{4}[P(t-Bu)_{3}]$ .<sup>66</sup> In the former case the cone angle is at least 160° while in the latter it is 182°.65 Ligands with small cone angles are also found in both sites. The compound Fe(CO), P(OMe), exists entirely in the axial form, while a

statistical distribution of isomers is found for  $Fe(CO)_{4}PF_{3}$ .<sup>67</sup> The cone angles are 107° for  $P(OMe_{3})^{66}$  and 104° for  $PF_{3}$ .<sup>67</sup> Also different site preferences are found for ligands which have approximately the same cone angle; i.e.  $PPh_{3}$  and  $AsPh_{3}$  prefer the axial site in  $M(CO)_{L}$  (M=Fe, Ru, Os) while SbPh<sub>3</sub> occupies an equatorial position in both  $Ru(CO)_{4}SbPh_{3}$  and  $Os(CO)_{4}SbPh_{3}$  in the solid state. The cone angle in all three cases is  $145^{\circ}$ .<sup>68</sup> We conclude that arguments based on the steric requirements of the ligands may be discounted as an explanation for axial-equatorial isomerism regardless of whether the axial or equatorial site is considered the more crowded.

As far as the central metal is concerned, it would be expected that iron (having the smallest covalent radius of the three metals) would be most affected by steric factors. And while it is true that all the iron compounds studied here displayed only the axial isomer in solution, the examples of other workers cited above show this is not always the case.

A more subtle steric argument based on calculating a minimum total repulsion energy summed over all the M-L bonds is the subject of the review by Favas and Kepert<sup>2</sup> noted in the introduction. It will be remembered that they base their calculations on the ratio (R) of the effective bond lengths of the unique ligand to the other four ligands and that for  $M(CO)_{4}L$ complexes the distortion of the C-M-C bond angles was calculated for different R values and the results were presented in graphical form. Thus the theoretical R values can be deduced

if structure is known. the In ax-Ru(CO) AsPh and ax-Ru(CO)<sub>4</sub>SbMe<sub>3</sub> there is only a slight bending of the equatorial carbonyls toward the non-carbonyl ligand. This would be in accord with  $R\simeq 1.00$  and no clear prediction of site preference. For the compounds eq-Ru(CO) SbPh, and eq-Os(CO) SbPh, the bending of the carbonyl ligands is guite pronounced and would lead to a predicted R value of ~0.85 in accord with the site preference found. However this R value is intuitively very unsatisfying in that it would be expected that the M-C bonding electrons would be closer to the metal nucleus than the M-Sb bonding electrons, leading to an R value >1.0. The distortion of the equatorial bond angles from the ideal 120° could then be the result of the greater replusion between seen as the electrons in the M-C bonds (which also have a large, diffuse,  $\pi$ component) than between these electrons and those in the M-Sb bond.

One final point is that it appears highly unlikely that crystal packing forces contribute to the switchover of isomeric forms in the solid state. If this were the case the close correlation with the solution infrared spectra would not be expected. Furthermore, the four species studied here,  $M(CO)_4AsPh_3$  and  $M(CO)_4SbPh_3$  (M=Ru,OS), as well as  $Fe(CO)_4EPh_3$  $E=P^{2.9}$  and  $Sb^{2.4}$  crystallize in the same space group (PT), regardless of the isomeric form adopted. Packing forces may be the determining factor in the compound  $Fe(CO)_4PPh(PPh_2)_2$  which crystallizes as the equatorial isomer but displays only an axial

carbonyl stretching pattern in solution.<sup>69</sup>

# 3.2 <u>Electronic</u> Properties of the Ligands

It is generally recognized<sup>70.71</sup> that while the  $\pi$ -acceptor properties of the group 15 ligands do not vary appreciably on descending the group, the  $\sigma$ -donor ability decreases in the order PR<sub>3</sub>>AsR<sub>3</sub>>SbR<sub>3</sub>.<sup>71.72</sup> Since some of the conclusions of this work are based on this premise it is worth exploring it in some detail.

It has been established that the carbonyl stretching frequencies of closely related species of the type  $M(CO)_{x}L$ increase with increasing  $\pi$ -acceptor properties of L but do not vary as much with the  $\sigma$ -donor properties.<sup>73</sup> From Table A.2.5 it can be seen that the CO stretching frequencies of  $M(CO)_{a}EPh_{3}$ (E=P,As,Sb) for the axial isomer are all very similar indicating no appreciable variation of the  $\pi$ -acidity of the ligands. This is consistent with earlier studies wherein it was taken to indicate near equality of the  $\pi$ -acceptor qualities of these ligands.<sup>74.75</sup>

Considerable experimental evidence 68.76.77.78 has been presented to show that the donor strength of group 15 ligands follows the order PR<sub>3</sub>>AsR<sub>3</sub>>SbR<sub>3</sub>. A quantitive understanding of the relative  $\sigma$ -donor strength of the ligands studied here can be gained from an analysis of the heats of formation of a number of LBX<sub>3</sub> adducts. Mente and Mills<sup>76</sup> found  $\Delta$ H values of -68.6,

-46.2 and -26.8 kcal/mol for the reaction of BCl<sub>3</sub> with Me<sub>3</sub>P. Me<sub>3</sub>As and Me<sub>3</sub>Sb respectively. Values for BH<sub>3</sub> were closely related at -79.9 and -49.6 kcal/mol for Me<sub>3</sub>P and Me<sub>3</sub>As. (The reaction between Me<sub>3</sub>Sb and BH<sub>3</sub> was anomalous.) Other investigators<sup>79</sup> have found the  $\Delta H$  of OCBH<sub>3</sub> to be -25.1 kcal/mol; a value approximately equal to that expected for Me<sub>3</sub>SbBH<sub>3</sub>. Since no  $\pi$  interaction is expected between the group 15 donor atom and the B atom we may take these measurements as an indication of donor strength. Because of the weaker inductive effect of the phenyl group compared th the methyl group, the SbPh<sub>3</sub> ligand would be expected to be a weaker donor than SbMe<sub>3</sub> and hence a weaker donor than CO. This would explain why a switchover of site preference in the solid state occurs when the unique ligand is changed from SbMe<sub>3</sub> to SbPh<sub>3</sub>. A rough estimate of the magnitude of the change of donor strength that occurs when Ph groups are substituted for Me groups can be seen from the values of the  $\Delta H$  values found for the reaction of ER, ligands with trans [CH<sub>3</sub>L'<sub>2</sub>(THF)Pt][PF<sub>6</sub>] (L'=P(CH<sub>3</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)).<sup>68</sup> The values for PMe<sub>3</sub> and PPh<sub>3</sub> were -26.2 and -19.5 kcal/mol respectively while those for AsMe<sub>3</sub> and AsPh<sub>3</sub> were -15.4 and -12.8 kcal/mol. (The value for SbPh<sub>3</sub> was -6.1 kcal/mol, no value was recorded for SbMe<sub>3</sub>.) Any strengthening of the  $\pi$ component in the L-Pt bond in the compounds studied above would offset the trend displayed. The fact that the trend so clearly exists must be taken as an indication of the weakening donor strength.

A more recent study<sup>80</sup> based on <sup>13</sup>C chemical shifts in NMR spectra concluded that the ordering of the donor/acceptor ratio of these ligands is SbR<sub>3</sub>>PR<sub>3</sub>>AsR<sub>3</sub>. However, this conclusion must be viewed with some caution since it is based only on the correlation between the <sup>13</sup>C NMR chemical shift ( $\delta$ CO) in LNi(CO)<sub>3</sub> complexes and the relative donor/acceptor character of L. The ordering is as above only when R=Ph, and differs when R=Me. From the <sup>13</sup>C NMR data obtained in this study (Table A.2.5) an order of AsPh<sub>3</sub>>SbPh<sub>3</sub>>PPh<sub>3</sub> is indicated, based on the Fe(CO)<sub>4</sub>L complexes which exist only in the axial form. Yet other orderings are possible if the data of Buchner and Schenk<sup>81</sup> are while such methods may be valid when used. It seems that applied to P donor ligands, they cannot be extended to other In 1965 it was noted<sup>82</sup> that <sup>13</sup>C chemical group 15 ligands. shifts of transition metal complexes were poorly understood. This comment was reiterated in 1974<sup>83</sup> and still seems valid in 1986.

A structural study of  $Cr(CO)_5EPh_3$  (E=P, As, Sb, Bi) complexes concluded from decreasing C-E-C angles and relatively shortened M-E bond lengths that the M-E bonds had more s character on descending the group.<sup>84</sup> From these results it was concluded that the bond order increased on going to the element lower in the group. (The distortion of the group 15 ligand from tetrahedral geometry can also be seen in the crystal structures determined here and by others.<sup>6,43</sup>) The explanation that the E-C bonds have enhanced p-character while the M-E bonds

have enhanced s-character is well accepted. It has been pointed out, however, that increased s character of the lone pair of such ligands makes it <u>less</u> available for bonding.<sup>81</sup> Futhermore, a recent theoretical study<sup>85</sup> indicated the lone pair on PF<sub>3</sub> was less diffuse and had more s character than that of PMe<sub>3</sub>. It is well known that PF<sub>3</sub> is less basic that PMe<sub>3</sub>.

While it is generally accepted that a shorter bond indicates a stronger bond it should be remembered that the Cr-E bonds of the heavier analogues are only shortened relative to the covalent radii of the donor atom. This would be the expected result of the lone pair having more s character and does not necessarily indicate increased bond order. Such an effect might be expected to be quite large when the donor atom is Sb or Bi since their covalent radii are considerably larger than that of P. The variation of bond length can thus be explained (at least in part) by the more compact nature of the orbital having the greater s character. Other workers have found that shorter bonds do not necessarily mean stronger bonds. Bryan and Kuchowski<sup>86</sup> concluded that Me<sub>3</sub>PBH<sub>3</sub> was a more stable adduct that  $F_3PBH_3$  even though the latter molecule had a significantly shorter P-B bond. Similarly, in PtCl<sub>2</sub>(PEt<sub>3</sub>)(PF<sub>3</sub>) the Pt-PEt<sub>3</sub> bond length (2.272(3)Å) was longer than the Pt-PF, length (2.141(3)Å) even though the Pt-PF<sub>3</sub> bond was thought to have lower intrinsic strength that the Pt-PEt<sub>3</sub> bond.<sup>87</sup>

We conclude that it is the  $\sigma$ -donor strength of most of the ligands studied here that determines their site preference in

 $M(CO)_{*}L$  complexes, i.e., the stronger  $\sigma$ -donors prefer the axial position while the weaker  $\sigma$ -donors favour the equatorial site <u>even in the presence of strong  $\pi$ -acceptor ligands (such as CO).</u> This is in accord with the theoretical studies discussed in the introduction but at variance with the usual explanation that it is the  $\pi$ -acceptor properties of the ligand that determines the site preference.

commonly held view that good  $\pi$ -acceptor ligands prefer The the equatorial site, is not disputed. However, if it were the determining factor in the molecules studied here it would be reflected in higher CO stretching frequencies for the axial of those complexes with a significant amount isomers of equatorial isomer in solution. As noted earlier the CO stretching frequencies of all the M(CO) EPh3 (E=P, As, Sb) complexes (axial isomer) are all very similar. Only for  $M(CO)_4 P(OCH_2)_3 CMe$  (M=Fe, Ru, Os) are the bands due to the axial isomer shifted to higher frequencies and in this case the occurrence of some equatorial isomer for M=Ru and Os may be rationalized accordingly, using  $\pi$  bonding arguments. The same can be said for other examples of M(CO) L compounds reported in the literature which have a large fraction of equatorial isomer in solution such as  $M(CO)_{\mu}PF_{3}$  $(M = Fe^{26})$  $Ru^{41}$ ) and  $Fe(CO)_{4}P[OC(CF_{3})_{2}CN]_{3}^{7}$ . That the carbonyl stretching frequencies of M(CO)<sub>4</sub>SbPh<sub>3</sub> complexes do not occur at high frequencies indicates that SbPh<sub>3</sub> is not a better  $\pi$ -acceptor than CO; therefore  $\pi$  bonding arguments cannot be used to explain why

 $Ru(CO)_4SbPh_3$  and  $Os(CO)_4SbPh_3$  exist predominantly as the equatorial form.

## 3.3 Influence of the Central Metal

As was stated earlier the tendency of the transition metals to give the equatorial isomer was Ru>Os>>Fe. In fact, there was no example for the group 15 ligands studied here of a  $Fe(CO)_{4}L$  derivative which gave detectable amounts of the equatorial isomer in solution. This trend is difficult to rationalize especially for the ruthenium and osmium complexes where the energy differences between the two isomers were small. Several possible explanations were considered and are now discussed. Unfortunately they do not account for the observations.

1)According to simple MO theory, ligand orbitals which are closest in energy to the symmetry matched metal orbitals form the strongest bonds. The ligand  $\sigma$ -donor orbitals are necessarily lower in energy than those of the metal while the opposite is true for the  $\pi$  orbitals. The increased energy of the valence metal orbitals on going from Fe, to Ru and/or Os should lead to stronger  $\pi$  bonds and weaker  $\sigma$  bonds. The energy levels of the valence orbitals are about equal for Ru and Os since the effect of increased atomic number is offset by the lanthanide contraction. Thus any ligand should have a greater tendency to occupy the equatorial position in ruthenium and

osmium complexes. However this effect should be approximately the same for CO as for the other ligand, so that no difference is site preference should result when the central metal is changed.

The available evidence suggests that the amount of  $\pi$  bonding to the CO ligands follows the order Fe-CO>Os-CO>Ru-CO, that is, the infrared CO stretching frequencies in complexes studied here and in the parent carbonyls follow the order, Ru-CO>Os-CO>Fe-CO. Also Ru(CO)<sub>5</sub> was the least stable of the three pentacarbonyls.<sup>46</sup> However, the differences in the infrared stretching frequencies are not great, and it is difficult to understand why the same order would not also be true for M-ER<sub>3</sub> bonds. In that case there would be no change in the difference between the M-CO and M-ER<sub>3</sub> bond strengths as M varied.

2)Other factors which have been used to explain differences in M-L bonding as the metal is replaced by heavier members of the triad are atomic size,<sup>88</sup> the lanthanide contraction<sup>89</sup> and relativity effects.<sup>90</sup> Each of these explain trends in M-L bonding as M is varied. But once again, a difference between M-CO and M-ER<sub>3</sub> bonding cannot be easily rationalized.

3)Arguments based on the hardness and softness of metals and ligands<sup>9</sup> might lead to the conclusion that the soft SbPh<sub>3</sub> ligand should form stronger bonds to ruthenium and osmium than to iron. This in turn would lead to the prediction that there would be less of the equatorial form for the M(CO)<sub>4</sub>SbPh<sub>3</sub>

complexes of the heavier metals, contrary to what is observed.

4)A careful study of the M-Sb-C<sub>Ph</sub> and  $C_{Ph}$ -Sb-C<sub>Ph</sub> bond angles for the three compounds M(CO)<sub>4</sub>SbPh<sub>3</sub> (M=Fe, Ru, Os) revealed that all the M-Sb-C<sub>ph</sub> angles are enlarged from the tetrahedral angle of 109.5° and follow the order Ru≥Os>Fe for M. Consequently all the C<sub>Ph</sub>-Sb-C<sub>Ph</sub> are reduced from the tetrahedral angle and follow the order Fe>Os≥Ru (see Table A.3.1.) In accord with the above discussion, this may be taken to mean that all the M-Sb bonds have enhanced s character while the Sb-C<sub>ph</sub> bonds have enhanced p character. The s character of the M-Sb bond would follow the order Ru-Sb>Os-Sb>Fe-Sb. The logical extension of the statement that increased s character of the lone pair on  $ER_3$ ligands makes them less available for bonding, <sup>81</sup> leads to the conclusion that M-ER<sub>3</sub>  $\sigma$  bonds with greater s character are weaker would be consistent with the observed trend for the site preference. This line of reasoning is weak however, in that it only correlates two sets of observations and explains neither. Also, it would lead one to predict that the Ru-Sb-C<sub>Me</sub> and Ru-As-C<sub>Ph</sub> angles of Ru(CO)<sub>4</sub>SbMe<sub>3</sub> and Ru(CO)<sub>4</sub>AsPh<sub>3</sub> would be larger than the Fe-Sb-C<sub>Ph</sub> angle in Fe(CO)<sub>4</sub>SbPh<sub>3</sub>. As can be seen from Table A.3.1 they are smaller.

It is concluded that a deeper understanding of the influence of the central metal on the site preference of a unique group 15 ligand awaits more detailed molecular orbital calculations. Simple symmetry overlap arguments do not suffice. Quite subtle yet significant differences could be expected from varying the

Compound	M-E-C <sub>ph</sub> Angle (°)a	C <sub>ph</sub> -E-C <sub>ph</sub> Angle(°)a	Ref.
$Fe(CO)_{4}SbPh_{3}$	116.0-116.8	101.6-102.0	24
Ru(CO) <sub>4</sub> SbPh <sub>3</sub>	113.8-121.7	98.7-99.9	6
$Os(CO)_4SbPh_3$	114.0-121.2	99.0-100.5	this work
Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	115.7(2)	102.6(3)	this work
Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	115.1-115.4	103.0-103.3	this work
aRange where a	ppropriate.	······································	

Table A.3.1. Ligand Bond Angles for M(CO)<sub>4</sub>ER<sub>3</sub> Complexes.

MO approach. This has been discussed by Blyholder and Springs in their study on  $Fe(CO)_5$ .<sup>12</sup>

### 3.4 Summary

It has been previously predicted that better  $\pi$ -acceptors and weaker  $\sigma$ -donor ligands have a greater tendency to occupy the equatorial site in a d<sup>8</sup> transition metal TBP complex of the type ML<sub>4</sub>L'.<sup>1</sup> Since good  $\pi$ -acceptor ligands are usually poor  $\sigma$ -donors and vice versa, changes in site preference have been difficult to attribute unambiguously to either the  $\sigma$  or  $\pi$  properties of the unique ligand. In many synthetic papers the site preferences were discussed only in terms of the  $\pi$  bonding ability of the unique ligand.<sup>17</sup>

The majority of known complexes of the type  $ML_4L'$  were of first row transition metals wherein the unique ligand occupied
an axial position. A few other examples existed which had a very strong  $\pi$ -acceptor ligand in the equatorial site. That the carbonyl stretching frequencies of these complexes were shifted to higher wave numbers was consistent with unusual  $\pi$  bonding ability of the ligands (i.e., PF<sub>3</sub> and P[OC(CF<sub>3</sub>)<sub>2</sub>CN]<sub>3</sub> in the iron tetracarbonyl compounds.)

The complex  $\operatorname{Ru}(\operatorname{CO})_4\operatorname{SbPh}_3$  was, therefore, unusual and interesting in that it contained both a second row transition metal and a unique ligand (SbPh<sub>3</sub>), not known as an exceptional  $\pi$ acid, in an equatorial position. This anomaly was the main impetus for the present study.

The new and known complexes synthesized and characterized here were all of the type M(CO) L (M=Fe, Ru, Os; L=group 15 This has allowed a systematic study of the influence ligand). of the central metal, the group 15 donor atom, and the ligand substituents on the site preference. Axial-equatorial isomerism was observed for a number of these complexes. The solution and solid state CO stretching frequencies and the crystal structures are all consistent with the tendency to give the equatorial isomer as Sb>As>P, Ph>Me, P(OCH<sub>2</sub>)<sub>3</sub>CMe>PPh<sub>3</sub>,PMe<sub>3</sub> and Ru>Os>>Fe. The switchover to the equatorial form in the solid state occurred between Ru(CO)<sub>4</sub>AsPh<sub>3</sub> and Ru(CO)<sub>4</sub>SbPh<sub>3</sub>, and Ru(CO)<sub>4</sub>SbMe<sub>3</sub> and Ru(CO)<sub>4</sub>SbPh<sub>3</sub>. The osmium analogues displayed similar behaviour.

Group 15 ligands with the same substituents are similar in size<sup>58</sup> and in their  $\pi$  bonding properties,<sup>70,71</sup> but their basicity decreases markedly on descending the period.<sup>70,75,75,77,78</sup> The observed site preference trend for the group 15 ligands with the same substituents has, therefore, been rationalized on the basis that weaker  $\sigma$ -donor ligands prefer the equatorial site even in the presence of strong  $\pi$ -acceptors such The same explanation was used with regard to EMe<sub>3</sub> and as CO.  $EPh_3$  ligands since the Me group is known to increase the  $\sigma$ basicity of any ligand to which it is attached. Reasoning based on  $\pi$  bonding is more relevant when applied to the  $P(OCH_2)_3CMe$  liqand. The  $M(CO)_4P(OCH_2)_3CMe$  (M=Ru, Os) complexes were the only ones with a phosphorus ligand to exhibit a detectable amount of the equatorial isomer in solution. It was noted that the CO stretching frequencies of these complexes were shifted to considerably higher wavenumbers compared to those found for M(CO) PMe\_. It thus seems reasonable that existence of a small amount of equatorial isomer when the unique ligand was  $P(OCH_2)_3CMe$  ligand may be rationalized in terms of the good  $\pi$  bonding ability of that ligand. However, it should be remembered that this phosphite ligand is also expected to be a weak  $\sigma$ -donor.

The influence of the central metal on the ligand site preference is certainly one of the most interesting and unexpected findings of this study. Unfortunately, it has been difficult to account for the observation in terms of  $\sigma$  bonding,

variation in valence orbital energies, or any other possible explanation. Arguments based on  $\pi$  bonding can be made in view of the lability of CO ligands attached to Ru and the CO stretching frequencies of Ru-CO>Os-CO>Fe-CO. But this rationalization is weak at best because it does not predict a different change in the nature of the M-CO bond as compared to the M-ER<sub>3</sub> bond.

Steric factors have been discussed and ruled out as a major contribution for the complexes studied here. They are probably important in other cases where the steric requirement of the unique ligand is extreme, e.g. in  $Fe(CO)_4P\{N(SiMe_3)_2\}\{C(SiMe_3)_2\}^{32}$  and  $Fe(CO)_4P\{N(SiMe_3)_2\}\{C(SiMe_3)_2\}^{32}$ 

The ''C NMR spectra displayed only one signal for each compound studied. This is indicative of rapid axial-equatorial exchange and rapid equilibrium between the isomers. The Berry psuedoratation mechanism accounts for both observations. Low temperature studies indicated the barrier to rearrangement is low.

#### 3.5 Future Directions

It is felt that the insight into the factors determining site perference in TBP derivatives of d<sup>8</sup> transition metals has been considerably enhanced by this study. However the nature of the influence of the central metal is still poorly

understood. Structural characterization of  $Ru(CO)_5$  and/or Os(CO)<sub>5</sub> would allow detailed MO calculations to be carried out on these molecules. This might point out interesting differences between their electronic structures which could help account for the changes in molecular geometry that occur when Ru or Os is substituted for Fe in the M(CO)<sub>4</sub>L complexes studied here.

Another approach would be to investigate whether the metals of other triads influence the geometry of  $ML_4L'$  complexes in a similar way. Numerous complexes of the type  $Co(CO)_4XR_3$ (X=group 14 atom; R=alkyl, aryl, H) are known. However, the rhodium and iridium analogues are unknown. Synthesis and characterization of such complexes might prove extremely interesting because axial-equatorial exchange in  $Co(CO)_4EX_3$ complexes can be slowed down on the <sup>13</sup>C NMR time-scale.<sup>14</sup>

#### CHAPTER 4

#### EXPERIMENTAL PROCEDURES

# 4.1 Materials and Instrumentation

Unless otherwise stated, manipulations of starting materials and products were carried out under a nitrogen atmosphere with use of standard Schlenk techniques. Reactions under moderate CO pressure were carried out in a 200-mL general-purpose bomb Instrument Co. Hexane was refluxed over potassium from Parr and THF over potassium benzophenone ketyl; both were distilled and stored under nitrogen before use. The pentacarbonyls of ruthenium and osmium were prepared by literature methods; 46 Fe(CO)<sub>5</sub> was commercially available. The group 15 ligand,  $P(OCH_2)_3CMe$ , was synthesized by the method of Heitsch and Verkade<sup>92</sup> by Mr. R. F. Alex; it was sublimed before use. Other ligands were commercially available. The iron complexes prepared by literature<sup>34</sup> methods or minor Fe(CO)<sub>4</sub>L were variations thereof. The method previously used to prepare  $Ru(CO)_4P(OMe)_3$  from  $Ru_3(CO)_{12}$  was used for  $Ru(CO)_4PMe_3$ .<sup>41</sup>

Infrared spectra were obtained with a Perkin-Elmer 983 spectrophotometer; NMR spectra with a Bruker 400-MHz instrument and mass spectra with a Hewlett Packard 5985 GC-MS system with an ionization voltage of 70eV. Microanalysis were performed by Mr. M. K. Yang of the Microanalytical Laboratory of Simon Fraser University. Analytical and mass spectral data for new

compounds are given in Table A.4.1; infrared, <sup>13</sup>C NMR and melting point data in Table A.2.5. Yields were calculated by comparing the weight of product (pure by infrared spectroscopy) to that expected for 100% yield.

Solutions of  $Ru(CO)_5$  and  $Os(CO)_5$  were handled with extreme caution in view of the well known toxic properties of  $Fe(CO)_5$ . The ligands PMe<sub>3</sub> and SbMe<sub>3</sub> were handled in the inert atmosphere of a dry box since they ignite spontaneously in air.

Because of the nature of the preparations of  $Ru(CO)_5$  and  $Os(CO)_5$ , the concentrations of the hexane solutions of these compounds could only be determined approximately. For most preparations described below the concentration of  $Ru(CO)_5$  in hexane was  $\approx 2x10^{-3}$  M, for  $Os(CO)_5$  it was  $\approx 7x10^{-3}$  M. Typical reactions using these starting materials involved 25mL of solution.

# 4.2 Synthesis Methods

# 4.2.1 Preparation of $Ru(CO)_{4}P(OCH_{2})_{3}CMe^{-1}$

A solution of  $\operatorname{Ru}(\operatorname{CO})_5$  and a slight excess of  $\operatorname{P}(\operatorname{OCH}_2)_3$ CMe was stirred in the dark at room temperature for 18 h during which time a pale yellow precipitate formed. The mother liquor was then removed and the precipitate washed with hexane (3x5mL). The precipitate was recrystallized from a large volume of hexane to give the product as pale yellow needles. The yield was 43%.

**Table A.4.1.** Analytical and Mass Spectral Data for New M(CO)<sub>4</sub>L Derivatives.

Compound	%Calcd.		%Found		Mass	
Compound	С	Н	С	Н		
$Ru(CO)_4 P(OCH_2)_3 CMe$	29.92	2.51	29.95	2.50	362 (P+)	
Ru(CO) <sub>4</sub> PMe <sub>3</sub>	29.07	3.14	29.01	3.16	290 (P+)	
Ru(CO) <sub>4</sub> AsPh <sub>3</sub>	50.89	2.91	50.92	2.89	dec.	
Ru(CO) <sub>4</sub> SbMe <sub>3</sub>	22.13	2.39	22.19	2.19	382 (P+)	
Os(CO) <sub>4</sub> P(OCH <sub>2</sub> ) <sub>3</sub> CMe	24.15	2.01	23.99	1.90	452 (P <sup>+</sup> )	
Os(CO) <sub>4</sub> PMe <sub>3</sub>	22.22	2.40	22.25	2.40	380 (P <sup>+</sup> )	
Os(CO) <sub>4</sub> AsPh <sub>3</sub>	43.43	2.48	43.51	2.57	610 (P <sup>+</sup> )	
Os(CO) <sub>4</sub> SbMe <sub>3</sub>	17.91	1.92	17.89	1.93	470 (P <sup>+</sup> )	
Os(CO) <sub>4</sub> SbPh <sub>3</sub>	40.32	2.31	40.43	2.58	656 (P+)	

<sup>a</sup>Most intense peak of parent ion envelope; in all cases the parent ion agreed with that simulated by computer.

4.2.2 Preparation of Ru(CO)<sub>4</sub>EPh<sub>3</sub> (E=P, As, Sb)

A hexane solution of  $Ru(CO)_5$  with an approximate two-fold molar excess of the ligand was stirred under CO pressure (25 atm) at room temperature for 18 h. After this period the gas was released and the solvent removed on the vacuum line to leave a yellow-orange powder. Recrystallization of the powder twice from hexane gave the pure  $Ru(CO)_4EPh_3$  product. The yield was 41% for  $Ru(CO)_4AsPh_3$  and 39% for  $Ru(CO)_4SbPh_3$ . The arsenic derivative was thermally unstable and slowly decomposed when

stored at room temperature. Preparation of  $Ru(CO)_{4}PPh_{3}$  from  $Ru(CO)_{5}$  and PPh<sub>3</sub> has been reported previously.<sup>43</sup>

# 4.2.3 Preparation of Ru(CO)<sub>4</sub>SbMe<sub>3</sub>

A solution of  $\operatorname{Ru}(\operatorname{CO})_5$  in hexane was placed in a Carius tube. The tube was cooled to -196 °C and evacuated; the solution was degassed with one freeze-thaw cycle. With the vessel at room temperature an approximately equimolar quantity of SbMe<sub>3</sub> was added and the tube pressurized with CO (2 atm). The solution was then stirred in the dark at room temperature for 5 h. After this period the gases were released, the solution transferred to a Schlenk tube and evaporated to dryness at 0 °C. The pure, pale yellow product was obtained by sublimation (static atmosphere, <0.02 mm) to a cold-water probe. The yield was 41%.

## 4.2.4 Preparation of Os(CO) <sub>4</sub>PMe<sub>3</sub>

METHOD 1. The compound was prepared in excellent yield by the reaction of equimolar quantities of  $PMe_3$  and  $Os(CO)_5$  in hexane at 120 °C under CO pressure (40 atm) for 16 h. The white compound was purified by sublimation at 35 °C, <0.02mm, to a probe at -78 °C.

METHOD 2. This derivative was also prepared (in excellent yield) by heating  $Os_3(CO)_{12}$  and  $PMe_3$  (1:3 molar ratio) in hexane at 280 °C under CO (200 atm) for 48 h. The compound was isolated as in Method 1. (This reaction was first carried out by Dr. P. Rushman.)

#### 4.2.5 Preparation of Os(CO)<sub>4</sub>SbMe<sub>3</sub>

Approximately equimolar quantities of  $Os(CO)_5$  and  $SbMe_3$  in hexane solution were heated at 80 °C under CO (33 atm) for 16 h. After the reaction vessel had been cooled and the CO released, the solution was transferred to a Schlenk tube and evaporated to dryness on the vacuum line. The pure, pale yellow  $Os(CO)_4SbMe_3$ (11% yield) was isolated by sublimation from the residue onto a cold water probe at static vacuum (<0.02 mm).

## 4.2.6 Preparation of Os(CO)<sub>4</sub>EPh<sub>3</sub> (E=As, Sb)

Approximately equimolar quantities of Os(CO)<sub>5</sub> and EPh<sub>3</sub> in hexane were heated at 90 °C under CO (25 atm). The resulting solution was evacuated to dryness. This procedure removed unreacted Os(CO)<sub>5</sub> as well as solvent. The remaining solid was dissolved in a minimum of hexane and chromatographed on a silica gel column (20 x 2.5 cm), with an eluent of hexane:THF (11:1). (Although occasional exposure of the solutions to air appeared not to affect the chromatography, the other conditions were found to be critical to the successful isolation of the desired During the chromatography three bands formed, the product.) first two were yellow, and the third very pale yellow to almost The first and third bands,  $Os_3(CO)_{12}$  and colourless. Os<sub>3</sub>(CO)<sub>11</sub>EPh<sub>3</sub> respectively, were discarded. The second band was the product, Os(CO)<sub>4</sub>EPh<sub>3</sub>, which was recrystallized from hexane to give pale yellow crystals . The yield was 15% for Os(CO)SbPh<sub>3</sub> and 7% for Os(CO)AsPh<sub>3</sub>. The triphenylphosphine

analogue,  $Os(CO)_4PPh_3$ , was prepared similarly, in much better yield.<sup>43</sup>

# 4.3 Crystal Structure Determinations

## 4.3.1 Ru(CO) AsPh<sub>3</sub> and Os(CO) SbPh<sub>3</sub>

Details are given here for the crystallographic analysis of  $Ru(CO)_4AsPh_3$  with the differences for  $Os(CO)_4SbPh_3$  noted in parentheses.

Pale yellow, plate-like crystals suitable for X-ray analysis were grown from hexane. Oscillation, Weissenberg and precession photographs were taken with Cu K radiation. These indicated the space group to be PI or P1 and gave approximate cell dimensions. The space group was subsequently determined to be PI by the structure solution. Accurate cell dimensions were determined by a least-squares refinement of 24 (22) reflections in the range  $2\theta$ =16.2-25.4° (30.9-42.4°) on a Picker FACS-I four circle automated diffractometer which employed graphite monochromated Mo K<sub>a</sub> radiation,  $\lambda$ =0.71069 Å.

Details of the data collection are given in Table A.4.2, other crystallographic data in Table A.4.3. The data were collected at  $20\pm1$  °C. Stationary-crystal stationary-counter background counts for 10% of the scan time were taken at each side of the scan. Peak-profile analysis was performed on all reflections to derive the intensity, I, and the associated error,  $\sigma(I)$ .<sup>93</sup> Two standards were measured after every 70

Table	A.4.2.	Diffractometer	<b>Collection</b> and	ıd Refinement	Parameters for
M(CO) <sup>4</sup> L	Compounds	• 20			
		Ru(CO)+AsPh+	0s(co)	4 SbPh 3	Ru(CO) 4 SbMe 3
scan method		u-2 <del>0</del>	u-20		u-28
scan range, 2	0, deg	3.5-40.0	3.5-40	0.	3.0-50.0
scan width, 2	0, deg	1.40	1.20		1.30
scan rate, 20	, deg/min	6	Ð		1.3-6.6
collection ra	nge	±h, +k, ±1	±h. +k	, ±1	±h, +k, +1
transmission	coefficient i	range 0.594-0.932	0.365-(	0.644	0.913-1.000
unique reflec	tions	2018	3754		582
observed refl	ections (I>2	. 3d) 1650	3115		523
variables		253	298		39
а. С		0.0203	0.0192		0.0233
R, C		0.0213	0.0205		0.0295
gof		1.0463	1.3989	1	0.9806
largest shift	σ	0.15	0.36		0.00

<sup>d</sup>Empirical absorption correction, see text. <sup>b</sup>R<sub>i</sub>=E||F<sub>O</sub>-|F<sub>C</sub>||/E|F<sub>O</sub>| <sup>C</sup>R<sub>i</sub>=[Eu(|F<sub>O</sub>|-|F<sub>C</sub>]):/Eu|F<sub>O</sub>|:|<sup>1/i</sup> <sup>d</sup>Largest shift/error in the final cycle of refinement.

reflections; they gave no indication of decomposition. The data were corrected for Lorentz and polarization effects and an analytical absorption correction<sup>94</sup> was also applied.

The structures were solved by Patterson and Fourier methods. All non-hydrogen atoms were located from successive electron density difference maps. The final refinement was by block-diagonal least-squares methods with anisotropic parameters for all non-hydrogen atoms. Hydrogen atoms were included at calculated (or previously refined) positions. Unit weights were used throughout for both structures. There were no significant trends in the average  $\Sigma \omega ||F_{c}| - |F_{c}||^{2}$  as a function of  $|\dot{\mathbf{F}}_{\lambda}|$  and  $\sin\theta/\lambda$  in the final error analysis. Atomic scattering factors including anomalous dispersion for non-hydrogen atoms were taken from reference 95.95 ORTEP views, with 50% thermal ellipsoids, of Ru(CO)<sub>4</sub>AsPh<sub>3</sub> and Os(CO)<sub>4</sub>SbPh<sub>3</sub> are given in Figures A.2.1 and A.2.3 respectively; these also give the labelling scheme employed for each molecule. Final coordinates for all atoms of both molecules, anisotropic thermal parameters for the non-hydrogen atoms, thermal parameters for the hydrogen atoms and structure factors are listed in the Selected bond lengths and angles for Ru(CO)<sub>4</sub>AsPh<sub>3</sub> appendix. are given in Table A.2.1; for Os(CO)<sub>4</sub>SbPh<sub>3</sub> in Table A.2.3. The computer programs used were those developed by Larson and Gabe.<sup>96</sup>

## 4.3.2 Os (CO) AsPh3

Space group and approximate cell dimensions were determined as for  $Ru(CO)_{4}AsPh_{3}$ . Accurate cell dimensions were obtained in a manner similar to that used for  $Ru(CO)_{4}SbMe_{3}$ ; the 20 scan range was 16.2-25.4° for 25 accurately centred reflections. The standard deviations of the cell dimensions were slightly higher than desirable for data collection; an examination of the peak profiles also indicated the crystal was not of sufficiently high quality for a structure determination. Cell dimensions (see Table A.4.3) and the space group revealed the compound is isostructural with  $Ru(CO)_{4}AsPh_{3}$ .

# 4.3.3 Ru(CO) SbMe 3

Pale yellow crystals suitable for X-ray analysis were obtained by slow sublimation in a sealed, evacuated tube. Weisenberg photographs (Cu,  $\lambda$ =1.5418 Å) were used to assign the space group The compound was then assumed to be isostructural as R3 or  $R\overline{3}$ . with Fe(CO)<sub>4</sub>SbMe<sub>3</sub><sup>33</sup> (space group R3) and the cell dimensions of this compound were used as a first approximation for the ruthenium analogue. Accurate cell dimensions were determined by least-squares refinement of 25 accurately centred reflections  $(2\theta=20-25^{\circ}, Mo K_{a1} \lambda=0.7093 \text{ Å})$ . Crystal data are given in Diffraction data were collected at 20±1 °C. on an Table A.4.2. CAD4-F diffractometer using graphite Enraf-Nonius а Background measurements were made by extending monochromator. the scan range by 25% at each side of the scan. Measurement of standard reflections every hour showed no significant two

Ru(CO)<sub>4</sub>AsPh<sub>3</sub>, Os(CO)<sub>4</sub>SbPh<sub>3</sub>, for Data Crystallographic Ru(CO)<sub>4</sub>SbMe<sub>3</sub> and Os(CO)<sub>4</sub>AsPh<sub>3</sub>. A.4.3. Table

	Ru(CO)4AsPh	0s(C0),SbPh,	Ru(CO) SbMe,	0s(CO),AsPh,
formula wt. space group	519.4 <b>9</b> 1	655.3 PT	380.0 R3	608.4 PT
crystal system	triclinic	triclinic	hexagonal	triclinic
в, А	10.605(3)	11.123(2)	10.378(1)	10.633(6)
b, À	11.068(4)	11.284(4)	ł	11.029(7)
c, A	9.979(3)	12.714(4)	9.632(1)	9,979(6)
α, deg	113.57(3)	129.29(2)	. 06	113.43(5)
þ, deg	93.14(3)	102.35(2)		93.24(5)
γ, deg	91.47(3)	102.45(2)	120	91.60(5)
Volume, Å <sup>3</sup>	1070.6	1068.5	898.3	1070.3
Z	0	0	Ċ.	2
d <sub>calcd</sub> , g/cm <sup>3</sup>	1.611	2.037	2.057	
dfound, g/cm <sup>1</sup>	1.50	1.99	2.10	
μ (Μο Κ <sub>α</sub> ), cm <sup>-1</sup>	22.70	71.81	33.99	•
crystal size, mm	0.24×0.22×0.030	0.076×0.11×0.22	0.61×0.37×0.14	

crystal decay, instability in the detection chain or variation in generator output. Lorentz polarization and a semi-empirical absorption<sup>97</sup> corrections were applied.

The non-hydrogen positional parameters from Fe(CO)<sub>4</sub>SbMe<sub>3</sub><sup>33</sup> were employed as the initial model. Refinement proceeded quickly by full matrix least-squares methods with anisotropic temperature factors used in the final cycles. Hydrogen atoms were not included in the calculations. When inverse coordinates were employed the residual R1 dropped from 0.0248 to 0.0233 therefore they were used for subsequent refinement. The weighting scheme  $\omega = [\sigma^2(F) + 0.0006F^2]^{-1}$  was shown to be correct on the basis of trends in  $\omega \Delta^2$  as a function of  $|F_{\alpha}|$  and  $\sin \theta / \lambda$ . The 1,-1,0 reflection was omitted from the refinement because it was judged to suffer from extinction (final KF = 229.43, F\_=260.88). Details of the data collection and final refinement parameters are given in Table A.4.2. Atomic scattering factors which included anomalous dispersion were taken from reference 95. An ORTEP view, with the atomic labelling scheme, of Ru(CO)<sub>4</sub>SbMe<sub>3</sub> is shown in Figure A.2.2. Selected bond lengths and angles are given in Table A.2.2. Final atomic coordinates, anisotropic thermal parameters and structure factor listings are listed in the appendix. The computer programs in this determination were from, "The Vax 750/780 Crystal Structure System".98

# PART B

SYNTHESIS, STRUCTURE AND CHARACTERIZATION OF Os<sub>4</sub>(CO)<sub>n</sub>PMe<sub>3</sub> (n=15, 14, 13) AND  $(\mu$ -H)<sub>2</sub>Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>3</sub>

#### CHAPTER 1

#### INTRODUCTION

#### 1.1 Theoretical Approaches

Chemists remain interested in transition metal clusters even though previously hoped for industrial catalytic applications have been slow in coming. The continued fascination stems from the large number of bonding modes, structures, fluxional processes and reactivities that clusters exhibit which present a challenge to both the theoretician and the experimentalist. The group 8 metals have proven a particularly fruitful area of study; over a thousand clusters are known with nuclearities ranging from three to ten<sup>99</sup> and geometries from chains<sup>100</sup> to very compact deltahedra.<sup>101</sup> Of special interest to this study are Group 8 tetranuclear clusters; these exhibit spiked triangle,<sup>102</sup> tetrahedral,<sup>103</sup>, butterfly,<sup>104</sup> planar rhomboidal<sup>105</sup> and other<sup>101,106</sup> geometries, see Figure B.1.1.

Several different approaches are used to rationalize or predict these structural variations but the discussion here will be limited to those most frequently encountered: the Effective Atomic Number (EAN) or 18-Electron Rule, Polyhedral Skeletal Electron Pair Theory (PSEPT), the isolobal analogy and some specific molecular orbital treatments. The first of these, the well known<sup>70.107</sup> EAN or 18-Electron Rule is often used to explain the structures of mono-, di- and trinuclear transition



metal complexes containing  $\pi$ -acceptor ligands. According to this approach valence electrons are assigned to the metal atoms, each of which is considered separately. Since a transition metal atom has nine valence orbitals, a stable structure is predicted only if a total of 18 electrons can be assigned to each metal atom. If there are not enough electrons available from the metal atom d orbitals and from the ligands then metal-metal bonding occurs. These bonds usually contribute one each metal atom involved. For tetranuclear electron to complexes the predicted number of cluster metal-metal bonds is given by the formula: nb=(72-ne)/2, where nb is the number of metal-metal bonds and ne is the number of electrons assignable to the complex from the ligands, charge and metal valence electrons. Thus a 64 electron tetranuclear complex is expected to have four metal-metal bonds and a square or spiked triangle geometry. The predicted number of bonds (and shapes) for 62 and 60 electron complexes are five (butterfly) and six (tetrahedron) respectively.

Churchill and Hollander used the 18-Electron Rule to explain the geometry of  $HOs_3Re(CO)_{15}$ , which has a planar rhomboidal structure.<sup>108</sup> The two  $Os(CO)_4$  moieties were viewed as 16 electron fragments needing two metal-metal bonds each and they thus bridge the  $HOs(CO)_3$  and  $Re(CO)_4$  fragments. The latter two fragments are 15 electron species needing three metal-metal bonds and hence the extra bond between them (see Figure B.1.2). The total number of valence electrons associated with the

cluster is 62.

Adams and co-workers also used the 18-Electron Rule in their discussion of the tetrahedral molecule  $Os_4(CO)_{12}(\mu_3-S)$ .<sup>109</sup> Each  $Os(CO)_3$  fragment was seen as having 14 electrons; an additional three are obtained from the three metal-metal bonds in which each osmium atom partakes. The sulphur ligand donates four electrons to the cluster, thus each osmium atom obtains an 18 electron configuration. The total number of electrons is 60 so that a tetrahedral structure is predicted, in agreement with experiment. However, it is difficult to understand on the basis of localized two-center, two-electron bonds how a  $\mu_3$ -S ligand, capping one face of a tetrahedron can contribute one electron to each of four osmium atoms.

A similar difficulty exists in describing the bonding in  $Os_4(CO)_{12}(\mu_3-S)_2$  which has a butterfly structure. Each osmium atom obtains an eighteen electron configuration only if each  $\mu_3$ -S ligand contributes three electrons to the cluster. This is in contradiction to the normal description of sulphur as a four electron donor. Adams and Yang view the sulphur ligands as contributing four electrons and refer to the cluster as 'electron rich'.<sup>110</sup> That is, it has 64 electrons but five metal-metal interactions. The authors believe the extra electrons cause weakening (and lengthening) of two of the Os-Os bonds which are consequently very reactive. It is difficult to view these elongated bonds as normal two-center-two-electron bonds.



Figure B.1.2. Geometry of HOs<sub>3</sub>Re(CO)<sub>15</sub>.

A large number of cluster compounds, particularly those of high nuclearity, cannot be adequately described using only the 18-Electron Rule and simple two-center-two-electron bonds. The tetrahedral cluster  $H_4Re_4(CO)_{12}$  is electron deficient and is more appropriately described as having four, four-center  $Re_3H$ bonds.<sup>111</sup> Each of the clusters,  $Rh_6(CO)_{16}$ ,  $[Co_6(CO)_{14}]^{4-}$ , and  $H_2Ru_6(CO)_{18}$  has two electrons too many to be accomodated by twelve two-center-two-electron bonds is its octahedral core.<sup>112</sup> A more suitable approach for these and other clusters is based on molecular orbital calculations which can be applied to transition metal clusters in a simplified version, namely the Polyhedral Skeletal Electron Pair Theory (PSEPT).

This theory was developed and applied to transition metal clusters almost simultaneously by Wade<sup>113</sup> and by Mingos.<sup>114</sup> According to these early versions of PSEPT each metal

contributes three atomic orbitals to the bonding molecular orbitals of the cluster. A cluster containing n metal atoms therefore has 3n atomic orbitals from which the cluster molecular orbitals can be formed. It is considered that one orbital from each vertex atom, that is n orbitals, point toward the center of the cluster to form one strongly bonding and n-1 non-bonding or antibonding molecular orbitals. The remaining 2n orbitals are dispersed radially and combine to form n bonding and n antibonding orbitals. The result is n+1 bonding molecular orbitals.

The number of electrons donated by a vertex varies with the metal and number and type of ligands. For group 8 metals,  $M(CO)_3$  vertices contribute two electrons and  $M(CO)_4$  vertices, Bridging carbonyl ligands contribute two four electrons. electrons and bridging or terminal hydrides one electron to cluster bonding. PSEPT predicts that when n+1 electron pairs are available to fill the n+1 bonding orbitals a stable compact deltahedron with n vertices i.e., a 'closo' structure, results. If n+2 electron pairs are present the appropriate deltahedron has n+1 vertices, one of which is unoccupied. This results in a more open structure, designated 'nido' which has one vertex Thus an  $M_5$  cluster with 7 electron pairs such as missing.  $Fe_5(CO)_{15}C$  is expected to, and does, adopt a square pyramidal geometry, i.e., an octahedron with one vertex missing.<sup>115</sup> This may be compared to  $Os_5(CO)_{16}$  which has 6 electron pairs (n+1) and a trigonal bipyramid (closo) geometry.<sup>116</sup> Complexes with n+3

electron pairs exhibit even more open 'arachno' geometries which are based on deltahedra with two vertices missing. For example, in  $H_3Os_4(CO)_{12}I$  which has seven electron pairs, the osmium atoms display a butterfly arrangement which is considered as an octahedron with two cis vertices missing.<sup>117</sup> A rarely used extension of this theory designates complexes with n+4 electron pairs as 'hypho' structures; deltahedra with three vertices missing.

Despite the advantage of PSEPT that it shifts the focus of attention from the individual metal atoms to the cluster as a whole, application of the theory to tetranuclear clusters is problematic. The view that the tetrahedral geometries of  $[H_3Ru_4(CO)_{12}]^{-}$ , <sup>118</sup>  $H_2FeRu_2Os(CO)_{13}$ , <sup>119</sup> and  $Os_4(\mu_3-NCH)(CO)_{12}$ , <sup>120</sup> (for example), which have six skeletal electron pairs apiece, are trigonal bipyramids with one vertex missing has been described as 'artifical'.<sup>121</sup> Certainly the idea that spiked triangles such as  $H_2OS_4(CO)_{1.5}$  or  $H_3OS_4Br(CO)_{1.3}^{10.2}$  which contain 8 skeletal pairs are hypho pentagonal bipyramids, is of little use. Mingos attempted to deal with part of the problem by defining an 'electron precise' cluster as one having the same number of bonding pairs as M-M vectors. This definition was then applied to tetrahedral clusters with six electron pairs.<sup>114</sup> Confusion is still evident, however, in that he later referred to such clusters as 'closo' structures.<sup>122</sup>

Another weakness of this theory is the rule that each metal atom makes available three atomic orbitals for cluster bonding

regardless of the number of terminal ligands bonded to it. Later calculations have shown that, in many cases, an  $Os(CO)_4$ fragment contributes only two orbitals to cluster bonding.<sup>122</sup>

Recent modifications of PSEPT have made some attempt to deal with these issues. Johnson and Benfield,<sup>107</sup> for example, suggested that an  $M(CO)_4$  (M=Fe, Ru, Os) fragment contributes two electrons and two atomic orbitals to cluster bonding. This concept is intuitively satisfying where the  $Os(CO)_4$  fragment is found in an edge bridging position as in  $H_2Os_5(CO)_{16}$ ,<sup>123</sup> but less so where the  $Os(CO)_4$  fragment is more highly coordinated. For example,  $Os_5(CO)_{16}$ , which was cited in the paper, has an  $Os(CO)_4$  fragment with a total connectivity of eight, but this was not discussed at all.

A far more comprehensive discussion is that of Evans and Mingos.<sup>122</sup> Their paper on the extension of PSEPT to nonconical fragments decribed the application of extended Hückel molecular orbital (EHMO) calculations to carbonyl clusters containing  $Os(CO)_4$  and T-shaped  $Os(CO)_3$  vertices. Specific calculations were performed for several tetranuclear configurations that bear a close relationship to those studied here. The authors showed that the isoelectronic  $C_{2v}$   $[Os(CO)_4]^{2+}$  and T-shaped  $Os(CO)_3$ fragments are also isolobal, i.e., they have frontier orbitals of similar, but not identical, energy and shape. The  $Os(CO)_4$ fragment is similar to the  $[Os(CO)_4]^{2+}$  fragment but has two more electrons. The frontier orbitals  $a_1$  and  $b_2$ , hybrid s-z and hybrid xy respectively, are illustrated in Figure B.1.3., which

shows that the order of the orbitals differs for the two fragments. The nature of the frontier orbitals also differs in that the  $b_2$  orbital in Os(CO)<sub>3</sub> is calculated to be 17% metal in character while in Os(CO), it is 53%. This led the authors to predict that T-shaped Os(CO)<sub>3</sub> fragments are "likely to be less effective in cluster bonding" and also "distorted from planar that the overlap with the a<sub>1</sub> orbital be geometry in order maximized". On the other hand, the Os(CO), fragments were calculated have to the correct symmetry and bonding characteristics to form planar systems. As an illustration, the performed calculations on the hypothetical planar authors cluster, [Os<sub>4</sub>(CO)<sub>16</sub>]<sup>2+</sup> while the planar structures, HOs<sub>3</sub>Re(CO)<sub>15</sub>  $[Re_{4}(CO)_{16}]^{2-}$  were cited as known examples. Taken together and these two concepts mean that 62 electron clusters with  $M(CO)_3$ groups at the hinges are more likely to be butterflies while those with M(CO), groups may be planar. It should also be noted that a cluster containing an excess of electrons for a closo polyhedron and having an  $M(CO)_4$  vertex is more likey to adopt an edge-bridged rather than a nido or arachno structure, as in  $H_2Os_5(CO)_{16}$ , cited above.

The authors considered polyhedra wherein the electron count for a closo structure was not exceeded and were led to several conclusions. First,  $Os(CO)_4$  fragments are likely to maintain their  $C_{2v}$  symmetry since deformation to  $C_{4v}$  is calculated to require 2.6eV. Second, the a<sub>1</sub> orbital of the  $M(CO)_4$  is only 28% localized on the metal meaning it is largely CO  $\pi^*$  in character.



Os(CO)<sub>3</sub>

Figure B.1.3. Frontier Orbitals for  $[M(CO)_4]^{2+}$  and  $M(CO)_3$ . (After Evans and Mingos.<sup>122</sup>)

In order to make up for a deficiency of electron density, the carbonyl ligands were predicted to take part in semi-bridging interactions with other metal atoms. Specific overlap population calculations for a tetrahedral cluster with one Os(CO), group gave 0.23 and 0.25 for M'-C overlap, depending on the conformer. Finally the authors predicted that even with additional metal-metal bonding arising from weak interaction of the cluster with the  $M(CO)_{\pm}$  b, orbital, an  $M(CO)_{\pm}$  fragment will be less effective in a nonplanar cluster enviroment than a conical M(CO)<sub>3</sub> fragment. That being the case, bonds involving Os(CO), fragments are expected to be longer than those involving only conical Os(CO), fragments. The authors go so far as to

predict that closed polyhedra with more than one M(CO)<sub>4</sub> vertex are "unlikely to be stable."

Although PSEPT is widely used to rationalize the structures of many medium-sized clusters, other approaches have also been employed. Hoffmann used EHMO calculations to develop an isolobal analogy betweed transition metal and organic fragments.<sup>124</sup> According to this analogy the number of electrons and orbitals a transition metal fragment has available depends on the coordination number of the transition metal. An  $Os(CO)_4$ , fragment, for example, can be isolobal to several different organic fragments depending on whether the osmium atom is 5, 6 or 7 coordinate, as in Figure B.1.4. This method has limited predictive powers but it does have flexibility and is therefore useful in rationalizing structures after the fact.

Lauher also used extended Hückel calculations on bare metal clusters to investigate their bonding.<sup>125</sup> The four metal atoms of a tetranuclear cluster contribute nine orbitals each and generate 36 molecular orbitals. According to Lauher, six of these orbitals are antibonding in molecules of  $T_d$  symmetry, thus 30 orbitals are bonding and 60 valence electrons are necessary for a stable structure to be formed. Changing the symmetry to  $C_{2v}$  (butterfly geometry) results in one antibonding orbital being lowered in energy so that 62 cluster valence electrons are necessary. Square  $(D_{4h})$  symmetry is shown to require 64 electrons. No calculations were performed for a spiked triangle. While these conclusions are consistent with those of



7 coordinate

Figure B.1.4. The Isolobal Analogy as Applied to  $Os(CO)_4$  and  $Os(CO)_3$  Fragments. The 'O' character means 'is isolobal with'. (After Hoffmann<sup>124</sup>)

PSEPT and the 18-Electron Rule, it can be seen that this method cannot take into account any possible localized or unequal metal-metal bonds.

In an alternative scheme relating the electron count to structure, Teo emphasized the number of vertices, faces and 'missing' antibonding orbitals a cluster possesses.<sup>126.127</sup> The conclusions drawn regarding tetranuclear clusters are similar to those described above i.e., a four vertex, four face cluster (tetrahedron) needs 60 cluster electrons, and reduction of the number of faces (i.e., changing to butterfly or planar geometry) requires additional electrons. Like Lauher, Teo was concerned only with the total number of valence electrons of a cluster and not with the influence the ligands attached to a particular vertex have on localized bonding. Nor did he make any attempt to account for the variation in the structures of 62 or 64 electron species.

As can be seen there are several different approaches to understanding the structure and bonding in tetranuclear clusters, all of which give similar predictions. That is, tetranuclear clusters containing 60 valence electrons should be tetrahedra, while those containing 62 electrons should be butterflies or planar rhomboidal structures. To our knowledge no author has dealt extensively with 64 electron clusters, but it is accepted that only four metal-metal interactions are expected for these species.<sup>125</sup> That these general predictions are valid can be seen from the examples cited in Tables B.1.1,

B.1.2 and B.1.3. The most notable exceptions are 64 electron clusters which can be not only squares and spiked triangles, but distorted butterflies as well. There are also a few examples in the literature of 60 electron butterflies such as  $[Fe_4(CO)_{12}CCO_2Me]^{-128}$  and  $Os_4(CO)_{12}(C_2H_2)^{129}$  but they are not relevant to this study. Table B.1.1. Group 8 Tetrahedral Clusters with 60 Valence Electrons.

Compound	Comments	Ref.
[Fe <sub>4</sub> (CO) <sub>13</sub> ] <sup>2-</sup>	has one $\mu_3$ -CO ligand and three three weakly semi-bridging CO's	103
$H_2FeRu_3(CO)_{13}$	three terminal CO's on each Ru, two terminal CO's on Fe, two asymmetric CO bridges on Fe-Ru bonds	130
$[FeRu_3(CO)_{12}NO]^{-}$	NO is terminal on Fe, three CO's are $\mu_2$	131
$H_2FeRu_2Os(CO)_{13}$ $H_2FeRuOs_2(CO)_{13}$	isomeric forms in solution, NMR evidence that metal framework undergoes minor reorganization in solution at temperatures above -45 °C	119
$[HFeOs_{3}(CO)_{13}]^{-}$	two CO's bridge Fe and Os bonds, H bridges Os-Os bond	132
$H_2FeOs_3(CO)_{13}$	two bridging CO's on Fe-Os bonds	132
$H_2Ru_4(CO)_{13}$	H's probably bridge long Ru-Ru bonds	133
[H <sub>3</sub> Ru <sub>4</sub> (CO) <sub>12</sub> ] <sup>-</sup>	two isomers in solid state, rapid isomerism of H positions in solution	118, 134
$[H_2Ru_4(CO)_{12}]^2$	NMR evidence for CO bridges at ≃-80 °C	135
$H_4Ru_4(CO)_{12}$	H's probably bridge long Ru-Ru bonds	136
$H_{4}Ru_{4}(CO)_{10}PPh_{3}$	long Ru-Ru bonds bridged by H's	137
[HOs <sub>4</sub> (CO) <sub>13</sub> ] <sup>-</sup>	has a bridging CO which is rare for Os clusters	138
[H <sub>2</sub> Os <sub>4</sub> (CO) <sub>12</sub> I] <sup>-</sup>	I is terminal	139

$H_3Os_4(CO)_{11}NO$	NO is terminal, long Os-Os bonds and displacement of CO groups indicate position of bridging H's	140
$HOs_4AuPEt_3(CO)_{13}$	one bridging CO, AuPEt <sub>3</sub> group is $\mu_2$	141
$H_3Os_4AuPEt_3(CO)_{13}$	no bridging CO, AuPEt <sub>3</sub> group is $\mu_2$	141
$Os_4NCH_3(CO)_{13}$	$NCH_3$ caps one face and is 4 e donor	120
$H_{3}Os_{4}(CO)_{11}C_{6}H_{9}$	cyclohexene ring $\sigma$ -bonded to one Os and $\pi$ -bonded to another	142
$H_3Os_{k}(CO)_{11}CHCHPh$	H's located by neutron diffraction, one H bridges short Os-Os bond	143
$H_{4}Os_{4}(CO)_{11}P(OMe)_{3}$	the H's bridge long Os-Os bonds	144
$H_{4}Os_{4}(CO)_{11}NCMe$	as for $H_{4}OS_{4}(CO)_{11}P(OMe)_{3}$	145
$H_2OS_4(CO)_{12}CHCH_2Ph$	$CHCH_2Ph$ is $\mu_2$ only, 2 e donor	146

# Table B.1.2. Group 8 Butterfly Structures.

Compound	Comments	Ref.
[HFe <sub>4</sub> (CO) <sub>12</sub> ] <sup>-</sup>	one $\pi$ -CO, H bridges the hinge, $\phi$ =117°, converts to tetrahedron with all terminal CO's when warmed.	147,148
[Fe <sub>4</sub> C(CO) <sub>12</sub> ] <sup>2</sup> -	<sup>13</sup> C NMR evidence for butterfly structure	149
[Fe <sub>4</sub> N(CO) <sub>12</sub> ] <sup>-</sup>	N is $\mu_4$ to Fe's, Fe-N-Fe unit almost linear, $\phi=78.2^\circ$	150
$HFe_4N(CO)_{12}$	N is $\mu_4$ to Fe's, Fe-N-Fe unit almost linear, $\phi=101^\circ$	151
$Fe_4N(CO)_{11}NO$	<pre>spectroscopic evidence only for butterfly structure</pre>	150
$[HFe_4(CO)_{12}C]^-$	C is $\mu_{4}$ , $\phi = 104^{\circ}$	152
HFe <sub>4</sub> (CO) <sub>12</sub> ( $\eta^2$ -COCH <sub>3</sub> )	$\eta^2$ -COCH $_3$ group is 5 e donor	152
HFe <sub>4</sub> $(\eta^2 - CH) (CO)_{12}$	in $\eta^2$ -CH group the C is $\mu_4$ to all Fe's, the H is $\mu_2$ between C and one Fe, $\phi=110.6^\circ$	153
$Fe_4AuPEt_3(CO)_{12}COCH_3$	as for $HFe_4(CO)_{12}(\eta^2-COCH_3)$	154
$[Fe_4(CO)_{13}A]^- A=AuEt_3$ AuPh <sub>3</sub> , HgCH <sub>3</sub>	,two isomers as for $[HFe_4(CO)_{13}]^-$ , in tetrahedral form A is $\mu_3$ on a face	154,155
Ru <sub>4</sub> (CO) <sub>13</sub> PPh	$\mu_3$ PPh group bridges both wing tips and one hinge Ru $\phi=111.24^\circ$	156
[Ru <sub>4</sub> (CO) <sub>13</sub> Cl] <sup>-</sup>	Cl bridges wing tips, $\phi=91.0^{\circ}$	157
H₃Ru₄N(CO) <sub>11</sub>	one H bridges hinge, other two H's bridge hinge-wing tips, N is µ4 to Ru's, ¢=108.6(108.2)°b	158
$HRu_4N(CO)_{12-X}$ [P(OMe)_3]_x x=1,2.3	N is $\mu_4$ to Ru's, $\phi$ not given <sup>a</sup>	159

Os4(CO) <sub>13</sub> S	S is $\mu_3$ to one closed triangular face, i.e., no bridge across wing tips or hinge, nearly flat, $\phi=170.0(176.5)^{\circ b}$	160
$[Os_{4}(CO)_{12}N]^{-}$	isostructural with $[Fe_{4}(CO)_{12}N]^{-} \phi = 105.4^{\circ}$	158
H <sub>3</sub> Os <sub>4</sub> (CO) <sub>12</sub> I	I bridges the wing tips, H's located by neutron diffraction, $\phi$ not given <sup>a</sup>	117
$H_3OS_4(CO)_{12}NO$	NO bridges the wing tips, $\phi$ not given <sup>a</sup>	159
$[H_{3}Os_{4}(CO)_{12}(NCMe)_{2}]^{+}$	one H bridges the hinge, no bridge across the wing tips, $\phi=112.2(1)^\circ$	104
$H_3Os_4(CO)_{12}OH$	OH is 3 e donor, OH bridges the wing tips, $\phi=92.5^{\circ}$	161
$H_3Os_4(CO)_{12}OPO_3H$	wing tips bridged be $OPO_3H$ , $\phi=92.5(92.2)^{\circ}b$	161
$[H_4Os_4(CO)_{12}OH]^+$	hinge not bridged, OH bridges wing tips, $\phi=82.3^{\circ}$	161
<sup>a</sup> where the dihedral an	ngle, $\phi$ , is noted as 'not given'	the

crystal structure diagram clearly indicated a butterfly structure.

bTwo molecules in the asymmetric unit.

Table B.1.3. Tetranuclear Group 8 Clusters with 64 Valence Electrons.

Compound	Comment	Ref.
$Fe_4(CO)_{11}(NEt)(ONEt)$	square, NEt is $\mu_4$ , ONEt	162
$Fe_{4}(CO)_{12}(PR)_{2}$ $Fe_{4}(CO)_{11}P(OMe)_{3}(PR)_{2}$ $Fe_{4}(CO)_{10}(P(OMe)_{3})_{2}(PR)_{2}$	square, PR ligands are $\mu_4$ , first two easily lose CO	106
$FeRu_3(CO)_{13}(PPh_2)_2$	distorted planar rhomboid, all Ru-Ru bonds elongated, pheripheral Ru-Ru bonds bridged by PPh <sub>2</sub> ligands	163
$\operatorname{Ru}_{4}(\operatorname{CO})_{13}(\operatorname{C=CBu}^{t})(\operatorname{PPh})_{2}$	nearly planar rhomboid, $\phi=176.93^{\circ}$	164
$Ru_4(CO)_8(PPh_2)_2(C=CBu^t)_2$ (PPh_2C=CBu <sup>t</sup> )	butterfly, $\phi=167.04^{\circ}$	164
$Ru_4(CO)_{10}(PPh_2)(C=CHPr^{i})$	butterfly, $\phi=141.49^{\circ}$	164
$Ru_4(CO)_{10}(OEt)(PPh_2)$ $(C=CHPr^1)$	butterfly, $\phi=143.69^{\circ}$	165
$H_2OS_4(CO)_{15}$ , $H_3OS_4Br(CO)_{13}$	spiked triangles, all Os-Os bonds normal covalent bonds	102
Os <sub>4</sub> (CO) <sub>12</sub> (CS)S	distorted square or butterfly unit not bonded across the hinge, the S is $\mu_3$ , it caps one face, CS on other side of the framework	166
Os <sub>4</sub> (CO) <sub>12</sub> S <sub>2</sub>	butterfly, S ligands are $\mu_3$ and bridge both wing tips and one hinge atom apiece, two weak metal-metal interactions	110
Os <sub>4</sub> (CO) <sub>12</sub> Se <sub>2</sub>	see comments for $Os_4(CO)_{12}S_2$ , can be described as nido pentagonal bipyramid	167

# 1.2 Project Description

The study here described began as an attempt to synthesize a cluster containing an unsupported, donor-acceptor metal-metal bond. In such a bond one metal is considered as donating two electrons to the other metal in order that both metals achieve an 18-electron count. In most organometallic complexes with metal-metal bonds the bond is considered as a normal covalent bond, i.e., each metal contributes one electron to the bond. Bridged donor-acceptor metal-metal bonds had been proposed several times<sup>168</sup> but alternate electron counting schemes could be applied to many of the examples. Several dimeric compounds synthesized in our laboratory had metal-metal bonds that could be unambiguously assigned as donor-acceptor bonds, 169, 170, 171 but until this study no cluster containing an unsupported donor-acceptor metal-metal bond had been synthesized. It was thought that the synthesis of cluster compounds with one or more such bonds could provide a systematic route to larger clusters. Rational routes to high nuclearity clusters are known<sup>172</sup> but these are rare; for example, many large osmium clusters are still obtained, in poor yields, by pyrolysis of Os<sub>3</sub>(CO)<sub>12</sub> or its derivatives.<sup>99,173,174</sup> The mechanisms of these building up reactions are still not well understood.

Until the present work, there was no reported fully characterized example of a 64 electron cluster displaying sequential ligand loss to give a 62 and then a 60 electron
species.<sup>102,175</sup> Here we report just such a series of compounds in the synthesis and characterization of the three clusters  $Os_4(CO)_n PMe_3$  (n=15, 14, 13). The 62 electron butterfly structure ( $\mu$ -H)<sub>2</sub>Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>3</sub> has also been synthesized and characterized. All four clusters have gross frameworks in accord with the theories outlined in the previous discussion. However, each has unique and interesting aspects not predicted by theory. The fluxional properties of these molecules were also investigated by variable temperature <sup>13</sup>C NMR spectroscopy. For two of the compounds novel exchange mechanisms are proposed. We believe the mechanism put forward for  $Os_4(CO)_{14}PMe_3$  has not been previously proposed.

#### **CHAPTER 2**

### RESULTS AND DISCUSSION

The reaction of  $Os_3(CO)_{11}MeCN$  with  $Os(CO)_4PMe_3$  proceeded at a convenient rate in hexane at 60 °C to produce  $Os_4(CO)_{15}PMe_3$ , a brilliant orange compound. Subsequent structural studies revealed it to be a spiked triangle in which  $Os(CO)_4PMe_3$ , an 18 electron moiety acts as a two electron donor to the  $Os_3(CO)_{11}$ species.<sup>176</sup>

This initial success led to a similar attempt with  $H_2Os_3(CO)_{10}$  and  $Os(CO)_4PMe_3$ . The unsaturated cluster  $H_2Os_3(CO)_{10}$  was known to be very reactive and many ligand addition reactions with it had been reported.<sup>177</sup> Surprisingly, the product of this reaction was not  $H_2Os_4(CO)_{14}PMe_3$  but rather  $Os_4(CO)_{15}PMe_3$  as before. Indeed, the reaction in hexane was complete at room temperature in one hour. A 57% yield could be obtained if excess  $Os(CO)_4PMe_3$  was present.

A further attempt to obtain a new cluster with а donor-acceptor bond was made by adding Me<sub>3</sub>NO to Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub> in  $CH_2Cl_2$  in the presence of  $Os(CO)_4PMe_3$ . It was hoped that substitution of a CO ligand by an Os(CO) PMe would occur producing a doubly spiked triangle,  $Os_5(CO)_{18}(PMe_3)_2$ . Instead, ligand simple CO loss occurred and the product was  $Os_4(CO)_{14}PMe_3$ . This compound has the interesting property that it appears deep red in CH<sub>2</sub>Cl<sub>2</sub> solution and distinctly green in hexane. Novel features were displayed in the variable

temperature <sup>13</sup>C NMR spectra of this compound so the structure was determined by X-ray analysis.

Results of mass spectra of both these compounds showed the most abundant peak in the highest mass envelope to be at 1202  $m^+/e$ , i.e.,  $[P-2CO]^+$  and  $[P-CO]^+$  respectively suggesting the compound  $Os_4(CO)_{1,3}PMe_3$  might be synthetically accessible. Indeed, this deep burgundy compound was obtained in 34% yield when a hexane solution of  $Os_4(CO)_{1,4}PMe_3$  was heated at 90 °C for 24 h.

During the investigation of the reaction of  $H_2OS_3(CO)_{10}$  and  $OS(CO)_4PMe_3$  it was noted that addition of  $Me_3NO$  to the reaction mixture produced several products. Isolation of bright red  $H_2OS_4(CO)_{13}PMe_3$  was possible using chromatographic techniques. Although this complex is isoelectronic with  $OS_4(CO)_{14}PMe_3$  its structure and fluxional properties are guite different.

These differences along with other aspects of the chemistry of these four tetranuclear osmium clusters are the basis of this discussion.

# 2.1 Os4(CO)15PMe3

### 2.1.1 Structure and Bonding

The structure of  $Os_4(CO)_{15}PMe_3$  (1) as determined by X-ray crystallography shows there are two molecules in the asymmetric unit but the differences between them are not chemically significant (see Figure B.2.1 and Table B.2.1). The metal atom framework of 1 is best described as a 'spiked triangle' in which the 18 electron moiety  $Os(CO)_4PMe_3$  donates two electrons to the  $Os_3(CO)_{11}$  fragment. There are no unusual bond lengths or angles; all carbonyl ligands are terminal and the the closest non-bonded Os-C contact is  $Os(12) \cdot C(113)$  at 3.32(3)Å. This is consistent with the IR solution spectrum in  $CH_2Cl_2$  which exhibits only terminal carbonyl stretches (see Table B.3.2 page 146). According to the 18-Electron Rule each osmium atom has its required complement of 18 electrons.

Similar spiked triangle structures have been observed for  $(\mu-H)Os_3Re(CO)_{15}NCMe^{175}$  and  $(\mu-H)HOs_4(CO)_{15}^{102}$ , but this is the first example containing a dative, or donor-acceptor, metal-metal bond. The dative metal-metal bond in 1 has no supporting metal-metal bonds nor is there evidence of even weak interaction between the Os(11) (or Os(21)) carbonyl ligands and Os(12) (or Os(22)).

While this type of unsupported dative bond is previously unknown for cluster compounds it has been reported for a number of dimeric complexes synthesized in this laboratory.<sup>169,170,171</sup>



Thermal Ellipsoid Diagram for Os4 (CO) 15 PMe3. Figure B.2.1. **Table B.2.1.** Selected Molecular Dimensions for Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub> a)Bond Lengths--Molecule 1.

Atoms	Bond Length (Å)	Atoms	Bond Length (Å)
Os(11)-Os(12)	.2.939(1)	Os(14)-C(118)	1.91(2)
Os(11)-P(1)	2.354(5)	₽(1)-C(11)	1.83(2)
Os(11)-C(14)	1.92(2)	P(1)-C(12)	1.79(2)
Os(11)-C(15)	1.92(2)	P(1)-C(13)	1.83(2)
Os(11)-C(16)	1.92(2)	C(14)-O(14)	1.15(2)
Os(11)-C(17)	1.91(2)	C(15)-O(15)	1.16(2)
Os(12)-Os(13)	2.849(1)	C(16)-O(16)	1.15(2)
Os(12)-Os(14)	2.923(1)	C(17)-O(17)	1.15(2)
Os(12)-C(18)	1.91(2)	C(18)-O(18)	1.16(2)
Os(12)-C(19)	1.89(2)	C(19)-O(19)	1.17(2)
Os(12)-C(110)	1.81(2)	C(110)-O(110)	1.18(2)
Os(13)-Os(14)	2.894(1)	C(111)-O(111)	1.13(2)
Os(13)-C(111)	1.93(2)	C(112)-O(112)	1.17(3)
Os(13)-C(112)	1.85(2)	C(113)-O(113)	1.15(2)
Os(13)-C(113)	1.92(2)	C(114)-O(114)	1.19(3)
Os(13)-C(114)	1.84(2)	C(115)-O(115)	1.14(2)
Os(14)-C(115)	1.89(2)	C(116)-O(116)	1.16(3)
Os(14)-C(116)	1.87(2)	C(117)-O(117)	1.15(3)
Os(14)-C(117)	1.88(2)	C(118)-O(118)	1.16(2)

b)Bond Angles--Molecule 1.

Bonds	Angle (°)	Bonds	Angle (°)
Os(11)-Os(12)-Os(13)	165.32(3)	Os(14)-Os(12)-C(110)	157.3(6)
Os(13)-Os(12)-Os(14)	60.17(3)	C(18)-Os(12)-C(19)	173.1(8)
Os(12)-Os(13)-Os(14)	61.19(3)	Os(14)-Os(13)-C(112)	157.4(7)
Os(12)-Os(14)-Os(13)	58.64(3)	Os(14)-Os(13)-C(114)	99.7(7)
Os(12)-Os(11)-P(1)	174.0(1)	C(111)-Os(13)-C(113)	175.6(9)
Os(12)-Os(11)-C(14)	88.3(6)	Os(13)-Os(14)-C(115)	162.5(6)
Os(12)-Os(11)-C(15)	84.1(6)	Os(12)-Os(14)-C(118)	89.2(6)
Os(12)-Os(11)-C(16)	88.0(5)	C(116)-Os(14)-C(117)	91.8(9)
Os(12)-Os(11)-C(17)	84.9(5)	Os(11)-P(1)-C(11)	113.4(7)
C(14)-Os(11)-C(15)	171.3(8)	Os(11)-P(1)-C(12)	114.3(7)
C(16)-Os(11)-C(17)	172.8(8)	Os(11)-P(1)-C(13)	114.9(7)

c)Bond Lengths--Molecule 2.

Atoms	Bond Length (Å	) Atoms	Bond Length (Å)
Os(21)-Os(22)	2.937(1)	Os(24)-C(218)	1.88(2)
Os(21)-P(2)	2.355(5)	P(2)-C(21)	1.81(2)
Os(21)-C(24)	1.94(2)	P(2)-C(22)	1.79(2)
Os(21)-C(25)	1.93(2)	P(2)-C(23)	1.82(2)
Os(21)-C(26)	1.93(2)	C(24)-O(24)	1.13(2)
Os(21)-C(27)	1,94(2)	C(25)-O(25)	1.14(2)
Os(22)-Os(23)	2.855(1)	C(26)-O(26)	1.16(2)
Os(22)-Os(24)	2.930(1)	C(27)-O(27)	1.13(2)
Os(22)-C(28)	1.93(2)	C(28)-O(28)	1.13(2)
Os(22)-C(29)	1.90(2)	C(29)-O(29)	1.17(2)

Os(22)-C(210)	1.83(2)	C(210)-O(210)	1.17(2)
Os(23)-Os(24)	2.894(1)	C(211)-O(211)	1.16(2)
Os(23)-C(211)	1.91(2)	C(212)-O(212)	1.18(2)
Os(23)-C(212)	1.84(2)	C(213)-O(213)	1.15(2)
Os(23)-C(213)	1.92(2)	C(214)-O(214)	1.14(2)
Os(23)-C(214)	1.90(2)	C(215)-O(215)	1.18(2)
Os(24)-C(215)	1.85(2)	C(216)-O(216)	1.17(2)
Os(24)-C(216)	1.92(2)	C(217)-O(217)	1.14(2)
Os(24)-C(217)	1.91(2)	C(218)-O(218)	1.17(2)

d)Bond Angles--Molecule 2.

Bonds	Angle (°)	Bond	Angle (°)
Os(21)-Os(22)-Os(23)	163.05(3)	Os(24)-Os(22)-C(210)	159.3(6)
Os(23)-Os(22)-Os(24)	60.02(2)	C(28)-Os(22)-C(29)	173.8(7)
Os(22)-Os(23)-Os(24)	61.28(3)	Os(24)-Os(23)-C(212)	163.2(6)
Os(22)-Os(24)-Os(23)	58.70(2)	Os(22)-Os(23)-C(214)	156.9(6)
Os(22)-Os(21)-P(2)	177.3(1)	C(211)-Os(23)-C(213)	173.9(8)
Os(22)-Os(21)-C(24)	90.6(5)	Os(23)-Os(24)-C(215)	162.8(6)
Os(22)-Os(21)-C(25)	84.5(5)	Os(22)-Os(24)-C(218)	154.6(6)
Os(22)-Os(21)-C(26)	82.21(6)	C(216)-Os(24)-C(217)	174.5(8)
Os(22)-Os(21)-C(27)	87.0(5)	Os(21)-P(2)-C(21)	113.6(7)
C(24)-Os(21)-C(25)	175.1(7)	Os(21)-P(2)-C(22)	113.4(7)
C(26)-Os(21)-C(27)	169.2(7)	Os(21)-P(2)-C(23)	114.4(6)

Invesigation of the structure, reactivity, and fluxional properties of these compounds has established the following: 1)Dative metal-metal bonds are slightly longer than normal covalent bonds between the same metals.

2) The Os(CO), PMe<sub>3</sub> complex is a weak donor ligand, comparable in strength to CO.

3)These complexes are either sparingly soluble or insoluble in hexane but all dissolve readily in CH<sub>2</sub>Cl<sub>2</sub>. This is probably a consequence of the polar nature of the donor-acceptor bond. 4)Compounds with donor-acceptor bonds are fluxional; they exhibit intramolecular carbonyl exchange. A mechanism consistent with the variable temperature <sup>13</sup>C NMR spectra is bridge-terminal carbonyl exchange across the dative bond.

Complex 1 exhibits all the above characteristics. From Figure B.2.2 which shows the Os-Os bond lengths in A within each of the two independent molecules it can be seen that the





donor-acceptor bond in each molecule is  $\simeq 0.06$ Å longer than the average Os-Os bond length of 2.877Å in Os<sub>3</sub>(CO)<sub>12</sub>.<sup>178</sup> (The standard deviation in each length is 0.001Å). While unbridged Os-Os bond lengths in osmium clusters vary, they rarely exceed 2.91Å, especially when the osmium atoms are six-coordinate.<sup>179</sup>

The Os(12)-Os(13) (or Os(22)-Os(23)) bond which is trans to the dative metal-metal bond is short compared to those in  $Os_3(CO)_{12}$ , indicating  $Os(CO)_{\mu}PMe_3$  is a weak donor ligand.<sup>180</sup> It is known that strong  $\sigma$ -donors lengthen the M-L bond in the position trans to the donor ligand in monomeric complexes<sup>181</sup> and Adams has extended this idea in his proposal that the trans influence of ligands affects the length of metal-metal bonds in osmium clusters.<sup>180</sup> It was observed that the Os-Os bonds trans to PMe<sub>2</sub>Ph and CS were 2.856(1)Å and 2.830(2)Å in Os<sub>3</sub>( $\mu_3$ -S)(CO)<sub>8</sub>L complexes compared to a length of 2.814(1)Å when L=CO. It follows that a comparatively short Os-Os bond indicates the ligand in the trans position is a weak  $\sigma$ -donor.

The Os(13)-Os(14) (or Os(23)-Os(24)) bond is of comparable length to those in  $Os_3(CO)_{12}$ , while the bond cis to the dative bond, Os(12)-Os(14) (or Os(22)-Os(24)), is somewhat lengthened. This may be a result of the shortening of the Os(12)-Os(13) (or Os(22)-Os(23)) bond. This type of reciprocal relationship between bond lengths suggested in was regard to  $Os_3(\mu_3-S)_2(CO)_8PMe_2Ph$  by Adams and coworkers who speculated that the weakening of one bond is accompanied by a decrease in metal-metal overlap.<sup>180</sup> This in turn permits increased overlap

between one of the atoms involved in the weak bond and a third metal atom. Presumably the reverse is equally reasonable, i.e., strengthening of one bond, with increased orbital overlap could lead to a longer neighbouring bond.

Further evidence that  $Os(CO)_4 PMe_3$  is a weak donor ligand comes from thermal decomposition and ligand, substitution studies. When a solution of 1 was stirred overnight at room temperature (25 °C) it decomposed. Both  $OS_{3}(CO)_{12}$ and  $Os_3(CO)_{11}PMe_3$  were identified as decomposition products by IR spectroscopy.<sup>182</sup> (Decomposition of 1 during synthesis was avoided by carrying out the reaction in hexane, in which 1 precipitates.) Reaction of 1 with PPh<sub>3</sub> in solution quickly produced  $Os_3(CO)_{1,1}PPh_3$  and  $Os(CO)_{\mu}PMe_3$ .

We have described the bonding in 1 as that of a triangular cluster having one unusual ligand, Os(CO)<sub>4</sub>PMe<sub>3</sub>. However, 1 may also be considered a tetranuclear cluster with 64 electrons that is predicted to have four metal-metal bonds. Both a spiked triangle and a square fulfill this requirement and both configurations are known. As mentioned in the introduction, there are also a number of 64 electron butterfly structures; these have two or three elongated metal-metal bonds.

Neither the 18-Electron Rule nor PSEPT is useful in predicting the specific geometry of a particular 64 electron compound. An alternative proposal is that the bridging characteristics of the ligands strongly influence the geometry

of the metal atom framework.<sup>102</sup> For example, the square clusters cited in Table B.1.1 and elsewhere<sup>183.184</sup> all have two face bridging ligands, perhaps this  $\mu_4$  bonding is able to overcome diagonal interactions that could produce strain in square complexes. Butterfly clusters which have 64 electrons also have ligands that are edge or face bridging and may consequently prevent the complete rupture of metal-metal bonds. The known spiked triangles have ligands which do not constrain the structure in this way even though some have bridging hydrides or halogens. It therefore seems that the spiked triangle is the preferred, or lowest energy configuration and other geometries are adopted when there are ligands present that prevent the molecule from opening up to the fullest extent possible.

## 2.1.2 Fluxional Properties of Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub>

The <sup>13</sup>C NMR spectrum at -67 °C of a <sup>13</sup>CO enriched sample of 1 is consistent with the solid state structure. It has five resonances of relative intensity one at  $\delta$ =183.9, 180.1, 173.0, 171.6 and 171.0 ppm; three resonances of relative intensity two at  $\delta$ =198.4, 187.7 and 184.0 ppm; and one resonance of relative intensity four at  $\delta$ =181.6 ppm (see Figure B.2.3). The resonance at  $\delta$ =181.6 ppm is assigned to the carbonyl groups on the Os(CO)<sub>4</sub>PMe<sub>3</sub> ligand which rotates about the Os(11)-Os(12) (or Os(21)-Os(22)) axis in solution; those of relative intensity one and two are assigned to the equatorial and axial carbonyls respectively of the Os<sub>3</sub>(CO)<sub>11</sub> fragment. When the solution was warmed to -26 °C two axial signals and two equatorial signals



Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub>. 'NS' means 'number of scans'.

broadened and collapsed into the baseline. Further warming resulted in collapse of the remaining signals. At ambient temperature (20 °C) the only peaks present in the spectrum were the very weak signals due to the decomposition products  $Os(CO)_4PMe_3$  and  $Os_3(CO)_{12}$ . Decomposition was more extensive on further warming and precluded investigation of the <sup>13</sup>C NMR spectrum of 1 at a higher temperature. Apart from the decomposition, the collapse process was reversible.

Mechanistic proposals which account for these observations are as follows:

1)At temperatures between -67 °C and -26 °C the process A shown in Figure B.2.4 occurs. In the intermediate, two carbonyls the Os-Os bond cis to the  $Os(CO)_{\sharp}PMe_{3}$  ligand. bridge Bridge-terminal exchange causes collapse of carbonyl signals A, D. This mechanism has been proposed for  $Os_3(CO)_{11}L$ B. C and  $(L=PEt_3^{185}, P(OMe_3)^{186})$ . It appears that the extra electron density on the osmium atom donated from L, or left there because of weak back-bonding, facilitates carbonyl exchange with the osmium atom cis to the L--->Os bond. Ligand exchange along the trans Os-Os bond might also be expected, but would this bring L into an axial position, which appears to an energetically unfavourable process. Investigations in this laboratory of the <sup>13</sup>C NMR spectra of  $Os_3(CO)_{1,1}P(OMe)_3$  have confirmed that at -10 °C there is only one sharp equatorial carbonyl signal, all others are broadened (see Figure B.2.5).<sup>186</sup> This observation is accounted for if bridge-terminal exchange does not take place







Figure B.2.5. Fluxional Processes and <sup>13</sup>C NMR Spectrum of  $Os_3(CO)_{1,1}P(OMe)_3$ .

along the Os-Os bond trans to  $P(OMe)_3$  but does occur along the other two Os-Os bonds. Such processes always leave the  $P(OMe)_3$  ligand in an equatorial position. Therefore the CO ligand in the trans three bond position to  $P(OMe)_3$  also does not exchange with, or become equivalent to, any other CO group, and the peak assigned to it does not broaden.

2)At temperatures above -26 °C, two processes, B and C, illustrated in Figure B.2.4 occur. The first, B, involves simultaneous bridge terminal exchange across the dative Os-Os bond and across the Os-Os bond trans to the dative bond. Such a mechanism accounts for the collapse of signals due to carbonyl ligands B, F, G and I. In order to account for collapse of the other signals mechanism C is proposed. That this exchange occurs more slowly than A is consistent with the observations made for  $Os_3(CO)_{11}P(OMe)_3$ .

An alternative mechanism which accounts for the spectra observed at -26 °C and at 0 °C is exchange across the dative Os-Os bond involving carbonyls A and I and a change of position of the  $Os(CO)_{4}PMe_{3}$  group (mechanism D in Figure B.2.4). However, when 1 was prepared from  $Os_{3}(CO)_{1,1}MeCN$  and <sup>13</sup>CO enriched  $Os(CO)_{4}PMe_{3}$  the <sup>13</sup>C NMR spectrum revealed that the <sup>13</sup>C label was equally distributed over all carbonyl sites within the molecule. This cannot occur unless mechanism C is also operative. Without a high temperature limiting spectrum it is not possible to distinguish between the A,B,C and A,C,D possibilities.

It is interesting to note that both B and D mechanisms give rise to a second isomer of 1 with the PMe<sub>3</sub> ligand in an axial position. There are a number of weak peaks in the <sup>13</sup>C NMR spectrum of 1 taken at -67 °C, suggesting that such an isomer is present. The <sup>13</sup>C NMR spectra of two other compounds which have donor-acceptor metal-metal bonds,  $(Me_3P_x(CO)_{5-x}OsW(CO)_5 (x=1,2),$ also indicated the presence of two isomers in solution.<sup>169,187</sup>

# 2.2 $Os_4(CO)_{14} PMe_3$

### 2.2.1 Structure and Bonding

According to the bonding models discussed in the introduction, a 62 electron tetranuclear complex such as  $Os_{4}(CO)_{14}PMe_{3}$  (2) is expected to adopt a butterfly arrangement of metal atoms. Such a geometry accomodates five nearly equal metal-metal bonds and allows variation of the dihedral angle between the metal atom triangles depending upon the number and bonding characteristics of the ligands. The crystal structure of 2 revealed it is not a butterfly. Rather, as can be seen from Figure B.2.6 and Table B.2.2, molecules of 2 are very nearly planar in the solid state; the dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(4) is 177.10(3)°. The solution <sup>13</sup>C NMR spectrum at low temperature (see Figure B.2.10, page 115) exhibits four signals of relative intensity two which are assigned to the four different pairs of axial carbonyl ligands. The axial carbonyls on any given osmium atom can be equivalent only if the molecule is planar. It is



Figure B.2.6. Thermal Ellipsoid Diagram for Os<sub>4</sub>(CO)<sub>14</sub>PMe<sub>3</sub>.

Table B.2.2 Selected Molecular Dimensions for Os<sub>4</sub>(CO)<sub>14</sub>PMe<sub>3</sub>.

a)Bond Lengths

Atoms	Bond Length (	Å) Atoms	Bond Length (Å)
Os(1)-Os(2)	2.779(2)	Os(4)-C(44)	1.93(2)
Os(1)-Os(3)	2.935(2)	P(1)-C(1)	1.79(2)
Os(1)-Os(4)	2.784(2)	P(1)-C(2)	1.78(2)
Os(2)-Os(3)	3.013(2)	P(1)-C(3)	1.80(3)
Os(3) - Os(4)	2.982(2)	C(11)-O(11)	1.13(3)
Os(2)-P(1)	2.320(6)	C(12)-O(12)	1.16(3)
Os(1)-C(11)	1.92(2)	C(13)-O(13)	1.16(2)
Os(1)-C(12)	1.88(2)	C(21)-O(21)	1.14(2)
Os(1)-C(13)	1.86(2)	C(22)-O(22)	1.12(3)
Os(2)-C(21)	1.95(2)	C(23)-O(23)	1.16(3)
Os(2)-C(22)	1.92(2)	C(31)-O(31)	1.13(3)
Os(2)-C(23)	1.89(2)	C(32)-O(32)	1.08(3)
Os(3)-C(31)	1.93(2)	C(33)-O(33)	1.14(3)
Os(3)-C(32)	2.02(2)	C(34)-O(34)	1.18(3)
Os(3)-C(33)	1.88(2)	C(41)-O(41)	1.13(3)
Os(3)-C(34)	1.87(2)	C(42)-O(42)	1.14(3)
Os(4)-C(41)	1.97(3)	C(43)-O(43)	1.19(3)
Os(4)-C(42)	1.93(3)	C(44)-O(44)	1.12(3)
$O_{5}(4) - C(43)$	1.82(3)		

b	) B	on	d	An	q.	16	es.
					_		

Bonds	Angle (°)	Bonds	Angle (°)
Os(2)-Os(1)-Os(3)	63.57(3)	Os(1)-Os(2)-P(1)	97.7(2)
Os(2)-Os(1)-Os(4)	126.28(5)	Os(1)-Os(2)-C(23)	165.7(7)
Os(3) - Os(1) - Os(4)	62.79(3)	Os(2)-Os(3)-C(33)	74.1(8)
Os(1) - Os(2) - Os(3)	60.74(5)	Os(2)-Os(3)-C(34)	170.9(8)
Os(2) - Os(3) - Os(1)	55.69(3)	Os(3)-Os(4)-C(43)	155.5(7)
Os(2) - Os(3) - Os(4)	111.76(5)	Os(1)-Os(4)-C(44)	168.0(8)
Os(1) - Os(3) - Os(4)	56.12(3)	Os(4)-Os(1)-C(13)	116.0(7)
Os(3) - Os(4) - Os(1)	61.09(4)	Os(2)-Os(1)-C(13)	117.7(7)
C(11)-Os(1)-C(12)	170.0(8)	Os(2)-P(1)-C(1)	114.8(9)
C(21)-Os(2)-C(22)	177.8(8)	Os(2)-P(1)-C(2)	117.0(7)
C(31)-Os(3)-C(32)	172.4(8)	Os(2)-P(1)-C(3)	114.0(8)
C(41)-Os(4)-C(42)	174.2(9)		•

concluded that 2 is planar from a chemical point of view and the small deviation of the dihedral angle from  $180^{\circ}$  arises from solid state effects. This structure exists despite the presence of a T-shaped Os(CO)<sub>3</sub> fragment, in conflict with the prediction of Evans and Mingos discussed earlier.<sup>122</sup>

We believe the planar geometry is related to the lack of bridging ligands. There is no indication in the crystal structure of any weakly semi-bridging interaction between a carbonyl ligand and a neighbouring non-bonded Os atom. The closest non-bonded Os-C contact is Os(2)...C(33) at 3.08(2)Å. Furthermore, the solution infrared spectrum displayed only

terminal carbonyl stretches, see Table B.3.2. Because 2 is isoelectronic with  $(\mu-H)_2Os_4(CO)_{13}PMe_3$  (3), a butterfly with two bridging ligands, further discussion of this point is postponed until the structure of 3 has been described.

interesting aspect of the structure of 2 is its Another asymmetry. The fifteen terminal ligands are not evenlv distributed among the four osmium atoms; one of the hinge atoms, Os(1), has only three carbonyl ligands while each of the other three metal atoms four has ligands apiece. Contrary to the prediction of Evans and Mingos, the peripheral bonds involving the T-shaped  $Os(CO)_3$  fragment are much shorter than the other The Os(1)-Os(2), Os(1)-Os(4) distances Os-Os vectors. are 2.779(2)Å and 2.784(2)Å respectively, while the other two peripheral bonds are very long; i.e., Os(2)-Os(3)=3.013(2)Å and Os(3)-Os(4)=2.982(2)Å. The short diagonal Os(1)-Os(3) vector is of more typical length, 2.935(2)Å. These values can be compared to the average Os-Os distance in Os<sub>3</sub>(CO)<sub>12</sub> i.e., 2.877(3)Å.<sup>178</sup> Both  $Os_3(CO)_{12}$  and 2 are planar clusters and three of the four in 2, like those in  $Os_3(CO)_{12}$ , have four terminal vertices ligands.

In order to account for the long and short bonds in 2 and ensure that each osmium atom obeys the 18-Electron Rule we propose the bonding model illustrated in Figure B.2.7. It has a single bond between the  $Os(CO)_3$  fragment and each of the other three vertices. In addition there are two three-centre-two-electron bonds, each encompassing the wing tips



Figure B.2.7. Bonding Model for Os<sub>4</sub>(CO)<sub>14</sub>PMe<sub>3</sub>.

and one hinge vertex. The short Os-Os vectors are thereby assigned a bond order of 1.5 and the long Os-Os vectors an order of 0.5, while the hinge vertices are connected by a normal single bond.

We would be interested to see if detailed molecular orbital calculations would support or refute this proposed bonding model, as simpler approaches have not proven helpful. For example we have attempted to apply PSEPT to 2, but the results are unsatisfactory. If, in accord with the view of Evans and Mingos, the T-shaped Os(CO)<sub>3</sub> fragments donates two orbitals and no electrons to cluster bonding while the  $C_{2v}$  OsL<sub>4</sub> fragments donate two orbitals and one electron pair, there are only three bonding interactions. This is shown in the scheme developed for

 $[Os_4(CO)_{16}]^2$ ; (Figure B.2.8) a hypothetical molecule isoelectronic with 2.<sup>122</sup> Three bonds hardly seems enough to hold so stable a tetranuclear species together. Also, the  $[Os_4(CO)_{16}]^{2+}$  molecule is not predicted to be asymmetric, no localized bonding is anticipated.

A rather liberal application of the isolobal analogy<sup>124</sup> with the T-shaped  $Os(CO)_3$  fragment deemed to provide three orbitals and two electrons in a manner similar to a conical  $Os(CO)_3$ fragment (see page 83) does result in a structure with five bonding interactions including a dative covalent bond across the hinge (see Figure B.2.9). The model also satisfies the 18-Electron Rule for all the osmium atoms but all the bonds are normal single bonds. Like PSEPT, this model fails to predict the observed asymmetry in 2.

### 2.2.2 Fluxional Properties

A <sup>13</sup>CO enriched sample of 2 in CHFCl<sub>2</sub>/CD<sub>2</sub>Cl<sub>2</sub> solution at -115 °C exhibited a <sup>13</sup>C NMR spectrum in the carbonyl region consistent with the structure found in the solid state, i.e., four resonances of relative intensity two (at  $\delta$ =211.9, 202.0 ( $J_{P-C}$ =8.6Hz), 192.5 and 171.9 ppm), assigned to the axial carbonyls; and six resonances of relative intensity one (at  $\delta$ =192.2, 176.9, 174.7, 172.7, 171.0 and 166.4 ppm) assigned to equatorial carbonyls, see Figure B.2.10. The axial signal at  $\delta$ =202.0 ppm can be assigned to the carbonyls bonded to Os(2) by virtue of the carbon-phosphorus coupling, but no phosphorus



Figure B.2.8.Molecular Orbital Diagram for  $[Os_4(CO)_{16}]^{2+}$ . (After Evans and Mingos<sup>123</sup>)



Figure B.2.9. Application of the Isolobal Analogy to Os<sub>4</sub>(CO)<sub>14</sub>PMe<sub>3</sub>.





coupling was observed for any of the equatorial resonances. This lack of equatorial carbon-phosphorus coupling has been noted for other osmium clusters.<sup>185,186</sup>

When the solution was warmed to -87 °C, eight of the signals broadened and collapsed into the baseline; the two remaining signals were due to axial carbonyls including those bonded to The spectrum obtained with the sample at -26 °C has Os(2). three additional peaks at  $\delta$ =183.6, 173.1 and 169.0 ppm, as well as a broad resonance just upfield of the signal at  $\delta$ =191.6 ppm.<sup>188</sup> These four signals are at the average value of the chemical shifts of the original signals taken in pairs. The broad signal at approximately  $\delta = 190$  ppm arises from the coalescence of the two axial peaks, while the other three new peaks come from the pair-wise collapse of the original six equatorial signals.

Various fluxional mechanisms have been proposed for osmium clusters, 101.189 but none of these can explain the variable temperature <sup>13</sup>C NMR spectra of 2. For example, three-fold twist and bridge-terminal carbonyl exchange mechanisms cause axial and equatorial signals to average. simple merry-go-round A rearrangement (as in step I of Mechanism B, Figure B.2.11) produces a second isomer of 2 with the PMe<sub>3</sub> ligand trans to the  $Os(CO)_3$  fragment rather than to an  $Os(CO)_4$  fragment. There is no evidence for such a second isomer in the spectrum at -115 °C. Furthermore, this mechanism would not cause pair-wise coalescence of the original signals, rather signals would appear

at values averaging those assignable to the individual isomers. Mechanism A, shown in Figure B.2.11, correctly accounts for the observed spectra, it predicts the following exchanges: C(13) with C(23), C(33) with C(43), C(34) with C(44), and C(31),C(32) with C(41), C(42). The axial carbons of  $Os(1) \{C(11), C(12)\}$ and Os(2) {C(21),C(22)} remain unaffected by the process. The double bond between Os(1) and Os(2) in the intermediate is proposed in order to fulfill the requirements of the 18-Electron Rule for both Os atoms. Similar configurations have been reported elsewhere.<sup>190</sup> It is probable that the extreme weakness of the Os(2)-Os(3) bond lowers the activation barrier to non-rigidity and allows exchange to continue at temperatures down to approximately -100 °C. It would be interesting to see what effect substitution of the PMe<sub>3</sub> with weaker  $\sigma$ -donors might have on the exchange rate.

This novel 'windshield wiper' mechanism involves a significant rearrangement of the metal skeleton. Skeletal reorganization has been previously proposed for metal clusters but was either unspecified<sup>191.192</sup> or included only subtle changes in bond lengths.<sup>193</sup> A recent study has concluded, however, that Pt-Os bond breaking and making probably occurs in PtOs<sub>3</sub>(CO)<sub>9</sub>(PMe<sub>2</sub>Ph)<sub>2</sub>( $\mu_3$ -S)<sub>2</sub>.<sup>194</sup> Recently, Johnson has suggested that such mechanisms are more common than previously thought.<sup>195</sup>

Because the mechanism A is so unusual, an exhausive search was made for other explanations of the  $^{13}$ C NMR spectra, but only one emerged. Mechanism B (Figure B.2.11) shows a two step



Mechanism A





Step I



Mechanism B

Figure B.2.11. Fluxional Mechanisms for  $Os_4(CO)_{14}PMe_3$ . (Axial carbonyls have been omitted for clarity.)

merry-go-round rearrangement wherein only equatorial carbonyls take part in bridging but the axial carbons C(11), C(12)exchange with C(31), C(32) by virtue of the change in the number of ligands on Os(1) and Os(3). Equatorial carbonyl signal averaging predicted by this mechanism is C(13) with C(23), C(34)with C(44), and C(33) with C(43). This scheme is consistent with the observed spectra but is thought unlikely to be correct for the following reasons:

1)The mechanism has two steps, each of which must occur at the same rate, otherwise signal averaging would occur between the structure found in the solid state and the intermediate II. It is unlikely that step I which involves four bridging carbonyls would occur at the same speed as step II which involves only two.

2) The mechanism involves carbonyls bridging the extremely long Os(2)-Os(3) and Os(3)-Os(4) bonds.

3)The intermediates of both steps do not obey the 18-Electron Rule.

4) If such merry-go-round mechanisms have low activation barriers, one would be expected to operate in  $Os_3(CO)_{11}PEt_3$  but this is not observed.<sup>185</sup>

The <sup>13</sup>C spectrum of 2 with the sample at 0 °C showed the peaks at  $\delta$ =201.6, 183.6, 173.1 and 169.0 ppm had broadened. An attempt was made to investigate the second fluxional process operating at higher temperatures but a spectrum for the fast exchange limit could not be obtained as the sample underwent

some sort of reaction (perhaps with the solvent). A sharp peak at  $\delta$ =189.9 ppm appeared in the room temperature spectrum which was assigned to the decomposition product Os(CO)<sub>4</sub>PMe<sub>3</sub>.

## 2.3 $(\mu-H)_2OS_4(CO)_{13}PMe_3$

### 2.3.1 Structure and Bonding

Like 2,  $(\mu-H)_2OS_4(CO)_{13}PMe_3$  (3) is a 62 electron species, but the X-ray structure determination revealed it has a butterfly framework with a dihedral angle of 112.74(3)° between the OS<sub>3</sub> triangles (Figure B.2.12 and Table B.2.3.) The PMe<sub>3</sub> group is attached to one of the wing-tips, OS(2), and all the carbonyl ligands are terminal. The closest non-bonded OS-C contact is OS(2)···O(12) 3.17Å.

As a consequence of the bend at the hinge the terminal ligands at the wing-tips are forced into close contact. In .3 the affected carbonyl ligands are skewed to alleviate the resulting non-bonded interactions, (Figure B.2.12.) The dihedral angle between the Os(2)-Os(4)-C(41) and Os(4)-Os(2)-C(21) planes is  $51.1(9)^{\circ}$  and the  $C(21)\cdots C(41)$  distance is 2.87(2)Å. This distance is considered a 'close contact' since the van der Waals radius of carbon, if taken as half the interplanar distance in graphite, is 1.67Å.<sup>196</sup>

The hydride ligands in 3 were not located in the X-ray diffraction investigation, however, the <sup>1</sup>H NMR spectrum had two signals in the bridging hydride region, a doublet of doublets at



 $(\mu^{-H})_{2}Os_{4}(CO)_{13}PMe_{3}$ .

for

Diagrams

Ellipsoid

Thermal

B.2.12.

Figure



121a

Table B.2.3. Selected Molecular Dimensions for

 $(\mu - H)_{2}Os_{4}(CO)_{13}PMe_{3}$ 

a)Bond Lengths

Bond	Length (Å)	Bond	Length (Å)
Os(1)-Os(2)	2.868(1)	Os(4)-C(43)	1.90(2)
Os(1)-Os(3)	2.886(1)	Os(4)-C(44)	1.94(2)
Os(1)-Os(4)	2.939(1)	P(1)-C(1)	1.82(2)
Os(2)-Os(3)	3.115(1)	P(1)-C(2)	1.79(3)
Os(3)-Os(4)	2.850(1)	P(1)-C(3)	1.80(3)
Os(2)-P(1)	2.342(4)	C(11)-O(11)	1.17(3)
Os(1)-C(11)	1.86(2)	C(12)-O(12)	1.15(2)
Os(1)-C(12)	1.87(1)	C(13)-O(13)	1.12(2)
Os(1)-C(13)	1.91(2)	C(21)-O(21)	1.14(2)
Os(2)-C(21)	1.94(2)	C(22)-O(22)	1.13(3)
Os(2)-C(22)	1.93(2)	C(23)-O(23)	1.16(2)
Os(2)-C(23)	1,91(2)	C(31)-O(31)	1.15(2)
Os(3)-C(31)	1.89(2)	C(32)-O(32)	1.13(2)
Os(3)-C(32)	1.93(2)	C(33)-O(33)	1.13(3)
Os(3)-C(33)	1.89(2)	C(41)-O(41)	1.15(2)
Os(4) - C(41)	1.92(2)	C(42)-O(42)	1.15(2)
Os(4)-C(42)	1.89(2)	C(43)-O(43)	1.13(2)
		C(44)-O(44)	1.16(2)

b)Bond Angles (°)

Bond	Angle (°)	Bond	Angle (°)
Os(2)-Os(1)-Os(3)	65.56(2)	Os(2)-Os(1)-C(13)	170.1(5)
Os(2) - Os(1) - Os(4)	94.86(3)	Os(1)-Os(2)-C(22)	100.6(5)
Os(3) - Os(1) - Os(4)	58.59(3)	Os(3)-Os(2)-P(1)	106.4(1)
Os(1)-Os(2)-Os(3)	57.48(2)	Os(2)-Os(3)-C(31)	110.1(6)
Os(2) - Os(3) - Os(1)	56.96(2)	Os(2)-Os(3)-C(32)	89.4(5)
Os(2) - Os(3) - Os(4)	91.50(3)	Os(2)-Os(3)-C(33)	158.8(6)
Os(1)-Os(3)-Os(4)	61.64(3)	Os(3)-Os(4)-C(43)	107.8(5)
Os(3)-Os(4)-Os(1)	59.77(2)	Os(1)-Os(4)-C(42)	91.9(5)
C(21)-Os(2)-C(23)	169.1(7)	Os(2)-P(1)-C(1)	113.3(9)
C(41)-Os(4)-C(44)	168.4(7)	Os(2)-P(1)-C(2)	114.6(7)
Os(2)-Os(1)-C(11)	93.7(6)	Os(2)-P(1)-C(3)	116.2(8)
Os(2)-Os(1)-C(12)	81.0(5)		х

 $\delta = -20.4 \text{ ppm}, H_{A}, (J_{H-H} = 1.23 \text{ Hz and } J_{P-H} = 10.2 \text{ Hz})$ and an unresolved doublet at  $\delta$ =-18.3 ppm, H<sub>B</sub>. The small J<sub>P-H</sub> value is consistent with  $H_{\lambda}$  bridging the Os(2)-Os(3) vector, i.e., cis to the PMe<sub>3</sub> ligand. Trans  $J_{P-H}$  values are of the order of 150 Hz.<sup>197,198,199</sup> Consistent with this positioning is the Os(2)-Os(3) distance which is extremely long, 3.115(1)Å. Long Os-Os vectors have been previously associated with  $Os(\mu-H)Os$ configurations as in  $H_2Os_3(CO)_{11}$  which has an H-bridged Os-Os vector of 2.9886(9)Å.179 Other long Os-Os vectors that are H-bridged have been noted for the butterfly clusters  $H_3Os_4(CO)_{12}(NCMe)_2$  in which the bridged Os-Os distances are

3.130(2)Å, 3.145(2)Å and 2.937(2)Å,<sup>104</sup> and  $H_3Os_4(CO)_{12}I$  in which the bridged Os-Os distances are 3.055(1)Å and 2.927(2)Å<sup>117</sup>

The location of the second bridging hydrogen cannot be determined unambiguously. There are no other Os-Os vectors which, by being conspicuously long, would indicate the presence of an Os( $\mu$ -H)Os linkage. Since the two hydrogens are coupled H<sub>B</sub> probably bridges either the Os(1)-Os(3) or the Os(3)-Os(4) vector. The Os(1)-Os(3) distance is significantly longer, i.e., 2.886(1)Å cf. 2.850(1)Å, and therefore this position is favoured, i.e., H<sub>B</sub> bridges the hinge. It should be noted however, that the Os-Os distance assigned to this Os( $\mu$ -H)Os grouping is very short. Further evidence in support of the positioning of this hydride is presented in the section on the <sup>13</sup> C NMR spectra of this complex.

The position of bridging hydride ligands can sometimes be inferred from the distortion of M-M-CO angles cis to the hydride bridge.<sup>178</sup> For this compound, however, it was thought that such distortion could be a result of the skewing of the CO(21) and CO(41) ligands noted earlier. If this were the case the displacement of the CO ligands could not be relied upon to indicate the position of the hydride ligands.

Comparison of the isolectronic structures 2 and 3 leads to the question of why 2 is planar but 3 is bent, or more generally, 'What determines the dihedral angle of such compounds?' As indicated earlier, we believe the answer lies in



**B.2.13.** <sup>1</sup>H NMR Spectrum and Hydride Ligand Positions for  $(\mu^-H)_2Os_4(CO)_{13}PMe_3$ . Figure
the presence and position of bridging ligands. Two other flat 62 electron clusters are known,  $\operatorname{ReOs_3H(CO)_{15}}$  and  $[\operatorname{Re}_4(CO)_{16}]^{2-}$ ; both lack bridging ligands.<sup>108.200</sup> The same is true for the larger planar structures  $\operatorname{Os}_5(\operatorname{CO})_{19}^{201}$  and  $\operatorname{Os}_5(\operatorname{CO})_{17}[\operatorname{P(OMe)_3}]_4$ .<sup>202</sup> On the other hand the twenty-three butterfly clusters listed in Table B.1.2 all have a bridging ligand across the hinge, across the wing-tips, or both. Some flat 62 or 64 electron structures with bridging ligands are known, but these bridges are either on a face or along a peripheral edge.

It is easy to understand that the wing-tips must be drawn closer together when bridged, otherwise the distance between them would be too long for bonding with the bridging ligand to take place. The OS(2)-OS(4) distance in 2, for example, is 4.962(3)Å, while typical wing-tip spans in  $[OS_4(CO)_{12}(\mu_4-N)]^-$  and  $[(\mu-H)_{4}OS_4(CO)_{12}(\mu-OH)]^+$  are 3.903Å<sup>158</sup> and 3.537Å,<sup>161</sup> respectively. Bending at the hinge allows the wing-tip osmiums to maintain pseudo-octahedral geometry in that the bridge atom-wing-tip osmium-terminal carbonyl angle is close to  $180^\circ$ . Such angles in  $(\mu-H)_3OS_4I(CO)_{13}$  and  $[(\mu-H)_4OS_4(CO)_{12}OH]^+$  are  $172.5(1)^\circ$  and  $174.2(4)^\circ$  respectively.<sup>117.161</sup> (The geometry is only pseudo-octahedral because the angles of the osmium triangles must be approximately  $60^\circ$ .)

Similar maintenance of nearly octahedral geometry occurs for the hinge osmium atoms if they are bridged by a hydride ligand, as is often the case. Such  $M-\mu H-M'$  configurations are often





Figure B.2.14. Octahedral Geometry of the Hinge Vertices of Butterfly Structures.

represented as three-centre-two-electron bonds with little or no direct metal-metal interaction.<sup>203,204</sup> Octahedral geometry can maintained at the hinge vertices if the terminal ligands are be bonded trans and cis to the bridging hydride. Only by bending a butterfly achieve this, otherwise the terminal ligands can must make very small angles with each other or the peripheral  $(\mu-H)_{3}Os_{4}I(CO)_{13}$ , the hydride ligands were M-M vectors. In located by neutron diffraction, and the H(2)-Os(2)-C(22) i.e., angle is 172.5(1)°.117 The octahedral geometry  $\mu$ -H-M-CO<sub>trans</sub> must be energetically favoured otherwise the bending of the Os, framework in 3 would be prevented by the steric interaction between C(21) and C(41), noted earlier.

We conclude that it is the wing-tip or hinge bridging ligands that force the metal framework to adopt a butterfly geometry; if neither is present the molecule will be flat. The electronic and steric properties of the hinge or wing-tip bridge ligands may determine the exact dihedral angle. In this regard, it is interesting to note that the cluster cation  $[(\mu-H)_3Os_4(CO)_{12}(NCMe)_2];$  which has a similar structure to 3 in that the hinge is hydrogen bridged but the wing-tips are not connected, has a dihedral angle of 112.2°, very close to that of 3. 104

# 2.3.2 <sup>13</sup>C NMR Studies

Unlike the other complexes in this study, 3 does not appear to undergo rapid ligand exchange at temperatures close to <sup>13</sup>C{<sup>1</sup>H} NMR spectrum run on a <sup>13</sup>CO enriched sample ambient. Α 0 °C revealed thirteen sharp resonances, of 3 held at as expected from the solid state structure (Figure B.2.15). It is possible that the bridging hydrogens block the carbonyl ligand exchange. The peaks of low intensity at  $\delta = 190.3$  and  $\delta = 188.0$  ppm can be assigned to the pseudo-axial carbonyls on Os(2), consistent with previous assignments of low field signals to axial carbonyls in planar osmium clusters.<sup>102,185,186</sup> The low intensity of these signals is due to unresolved coupling to the phosphorus atom. The low field signals at  $\delta$ =191.3 and 186.7 ppm were tentatively assigned to the pseudo-axial carbonyls on Os(4), but the origin of the other peaks could not be unambiguously determined.

In order to help determine the position of the bridging hydrogens, gated and selectively hydrogen decoupled <sup>13</sup>C NMR spectra were obtained on 3 with the sample at 0 °C. The gated spectrum showed that the three carbon peaks at  $\delta$ =175.5, 174.4 and 171.4 ppm exhibited coupling to hydrogen. The peak at  $\delta$ =176.8 ppm was reduced in intensity from that in the <sup>13</sup>C{<sup>1</sup>H} spectrum of 3. Selective decoupling of H<sub>A</sub> at -20.4 ppm gave a singlet signal at  $\delta$ =174.4 ppm, while the signal at  $\delta$ =176.8 ppm sharpened marginally. The same procedure applied to H<sub>B</sub> at -18.3 ppm resulted in singlets for the  $\delta$ =175.5 and 171.4 ppm





resonances, see Figure B.2.15. The data are consistent with the presence of the bridging hydrides along the Os(2)-Os(3) ( $H_A$ ) and Os(1)-Os(3) ( $H_B$ ) vectors, except that the weakness of the coupling of  $H_A$  to one of the carbonyl signals is not explained. Placement of  $H_A$  along the Os(1)-Os(2) vector would explain why it shows strong coupling to only one carbonyl but then it would be expected to exhibit strong trans coupling to the phosphorus atom, which is not observed.

An alternative position of  $H_B$ , i.e., along the Os(3)-Os(4) vector, is also in accord with with the observed H-C couplings, but this location is not consistent with the length of the Os(3)-Os(4) vector which is only 2.850(1)Å. Placement of  $H_B$  along the much longer Os(1)-Os(4) vector (2.939(1)Å) is ruled out because the two hydrogen signals are coupled in the <sup>1</sup>H NMR spectrum.

# 2.4 $Os_4(CO)_{13}PMe_3$

#### 2.4.1 Structure and Bonding

The major stimulus for investigating the structure of  $Os_4(CO)_{1,3}PMe_3$  (4) was the desire to examine the validity of the scheme of structural changes that occur with sequential ligand loss. It can be seen, however, from Figure B.2.16 and Table B.2.4 that this complex has several interesting aspects in its own right. As expected, the metal atom framework consists of a tetrahedron, but the vertices are more unusual: one  $Os(CO)_3PMe_3$ 



Figure B.2.16. Thermal Ellipsoid Diagram for Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>3</sub>.

Table B.2.4. Selected Molecular Dimensions for  $Os_4(CO)_{13}PMe_3$ . a)Bond Lengths.

Atoms	Bond Length ()	A) Atoms	Bond Length (Å)
Os(1)-Os(2)	2.861(2)	Os(4)-C(42)	1.88(3)
Os(1) - Os(3)	2.831(1)	Os(4)-C(43)	1.89(3)
Os(1)-Os(4)	2.842(1)	P(1)-C(1)	1.73(3)
Os(2)-Os(3)	2.857(2)	P(1)-C(2)	1.83(3)
Os(2) - Os(4)	2.869(1)	P(1)-C(3)	1.85(3)
Os(3) - Os(4)	2.765(1)	C(11)-O(11)	1.18(3)
Os(1)-P(1)	2.361(6)	C(12)-O(12)	1.15(3)
Os(1)-C(11)	1.91(2)	C(13)-O(13)	1.20(3)
Os(1)-C(12)	1.92(3)	C(21)-O(21)	1.17(4)
Os(1)-C(13)	1.87(2)	C(22)-O(22)	1.17(5)
Os(2)-C(21)	1.90(3)	C(23)-O(23)	1.13(4)
Os(2)-C(22)	1.87(4)	C(24)-O(24)	1.17(4)
Os(2)-C(23)	1.92(3)	C(31)-O(31)	1.15(3)
Os(2)-C(24)	1.94(3)	C(32)-O(32)	1.17(4)
Os(3)-C(31)	1.89(3)	C(33)-O(33)	1.12(4)
Os(3)-C(32)	1.85(3)	C(41)-O(41)	1.07(4)
Os(3)-C(33)	1.90(3)	C(42)-O(42)	1.18(4)
Os(4)-C(41)	1.90(3)	C(43)-O(43)	1.16(4)

# b)Weak Interactions

Atoms	Distance Å		·
Os(3)C(11)	2.73(2)		
Os(3)C(24)	2.57(3)		• .
Os(4)C(12)	2.68(2)	· ·	
Os(4)C(22)	2.80(4)		
c)Bond Angles			
Bonds	Angle (°)	Bonds	Angle (°)
Os(2)-Os(1)-Os(3)	60.24(4)	Os(4)-Os(2)-C(21)	94.4(8)
Os(2)-Os(1)-Os(4)	60.41(4)	Os(4) - Os(2) - C(22)	69(1)
Os(3)-Os(1)-Os(4)	58.34(3)	Os(3)-Os(2)-C(24)	61.2(9)
Os(1)-Os(2)-Os(3)	59.35(4)	Os(1)-Os(2)-C(23)	108(1)
Os(1)-Os(2)-Os(4)	59.45(4)	Os(4)-Os(1)-C(12)	65.3(7)
Os(3) - Os(2) - Os(4)	57,75(4)	Os(2)-Os(3)-C(32)	148.3(8)
Os(1)-Os(3)-Os(2)	60.41(4)	Os(4)-Os(3)-C(33)	92.1(9)
Os(1)-Os(3)-Os(4)	61.02(3)	Os(1)-Os(3)-C(31)	112.5(8)
Os(2)-Os(3)-Os(4)	61,35(4)	Os(2)-Os(4)-C(43)	150(1)
Os(1) - Os(4) - Os(2)	60.14(4)	Os(3)-Os(4)-C(41)	93.7(9)
Os(1)-Os(4)-Os(3)	60.64(3)	Os(1)-Os(4)-C(42)	115(1)
Os(2)-Os(4)-Os(3)	60.90(4)	Os(1)-P(1)-C(1)	117(1)
Os(2)-Os(1)-P(1)	111.8(2)	Os(1)-P(1)-C(2)	113(1)
Os(3)-Os(1)-C(11)	66.9(7)	Os(1)-P(1)-C(3)	115(1)
Os(2)-Os(1)-C(13)	152.4(7)		

fragment, one Os(CO)<sub>4</sub> fragment and two Os(CO)<sub>3</sub> fragments. This appears to be the first recorded example of a closed polyhedron having two vertices with four terminal two-electron donor ligands. In contrast to the prediction of Mingos and Evans<sup>122</sup> neither steric nor electronic effects appear to make this compound unstable. It can be stored under  $N_2$ , at room temperature, for several months without decomposition. Indeed, the other compounds 1 and 2 showed loss of CO under the ionization conditions of mass spectroscopy while 4 did not, indicating it may be the most stable of the three. Complex 4 has the highest melting point of the series, 183 °C (see Table B.2.5.) Further evidence of stability comes from the Os-Os bond shortest Os-Os bond [Os(3)-Os(4), 2.765(1)Å] is lengths. The between the two Os(CO)<sub>3</sub> fragments, while the other five bonds are all within 0.02Å of their mean value of 2.852Å. Churchill has pointed out that such values should be compared to similar unbridged bond lengths in other tetrahedra.<sup>205</sup> The complex  $H_2OS_3CO(CO)_{16}(\eta^5-C_5H_5)^{205}$  for example, has an unbridged Os-Os bond between two Os(CO)<sub>3</sub> fragments, of 2.778(1)A°, similar to the Os(3)-Os(4) bond length determined here. The cluster [H<sub>2</sub>Os<sub>4</sub>(CO)<sub>12</sub>]<sup>2-</sup> has unbridged Os-Os bonds of 2.797(2), 2.811(2), 2.792(2) and 2.792(2)Å, 206 while those in [HOs<sub>4</sub>(CO)<sub>13</sub>] are 2.817(2), 2.807(2), 2.773(2) and 2.774(2)Å.<sup>138</sup> The unique inequivalent unbridged Os-Os distance in the neutral complex  $H_2OS_4(CO)_{11}CNMe$  is 2.822(1)Å.<sup>145</sup> The bonds involving the OS(CO)<sub>4</sub> and Os(CO)<sub>3</sub>PMe<sub>3</sub> fragments in 4 are significantly longer than any of these lengths, perhaps reflecting their diminished

cluster bonding capacity, relative to that of an  $Os(CO)_3$ fragment. However, the differences are not so great as to imply any of the Os-Os bonds in 4 are particularly weak. The crystallographically inequivalent bonds in  $Os_5(CO)_{16}$  which involve the  $Os(CO)_4$  fragment are significantly longer at 2.889(3)Å and 2.866(4)Å.<sup>116</sup> One might predict the Os(1)-Os(2)bond in 4 to be especially lengthened since both osmium atoms have four ligands, but it is not particularly long at 2.861(2)Å.

In agreement with Mingos and Evans' calculations, 122 the OsL, fragments maintain nearly C<sub>2</sub>, symmetry and there exists evidence for weakly semi-bridging interactions between four of the carbon atoms bonded to Os(1) and Os(2) and neighbouring osmium atoms  $[Os(3) \cdots C(11)=2.73(2)Å, Os(3) \cdots C(24)=2.57(3)Å,$  $Os(4) \cdots C(12) = 2.68(2)$  and  $Os(4) \cdots C(22) = 2.80(4)$  he the corresponding Os-C-O angles are listed in Table B.2.4.] These semi-bridging interactions, however weak, add to the overall stability of the complex. As Mingos noted, such interactions have been previously observed in  $OS_5(CO)_{16}$ , <sup>116</sup>  $(\mu-H)_2 Ru_1 (CO)_{13}^{133}$  and  $(\mu-H)_2 FeRu_3 (CO)_{13}^{130}$  It is of interest that other similar structures such as  $[(\mu-H)Os_{\mu}(CO)_{13}]^{-138}$ and  $(\mu-H)_2$  FeOs<sub>3</sub> (CO)<sub>13</sub><sup>207</sup> have fully bridging CO ligands rather than M(CO), vertices, but the reasons for such variation in structure are not understood.

It can be seen that PSEPT in its more sophisticated form is applicable to this complex, in spite of the fact that its ability to exist was questioned. Predictions regarding the

symmetry of  $OsL_4$  vertices, their weakly semi-bridging interactions and the relative strength of the metal framework bonds are all in accord with the results determined here.<sup>122</sup> While a bonding scheme of the osmium carbonyl cluster  $Os_4(CO)_{14}$ , which would be analogous to 4 was not developed by Mingos and Evans, there can be little doubt such calculations would result in the prediction of six cluster bonding orbitals containing twelve electrons.

We have used the isolobal analogy to develop a simple localized bonding picture for 4.<sup>124</sup> According to this approach, the Os(CO)<sub>3</sub> fragments, which have a total coordination of six, contribute three orbitals containing two electrons to the cluster, while the other two fragments, Os(CO), and Os(CO), PMe, which are seven coordinate contribute three orbitals containing four electrons. Thus the resonance structures shown in Figure B.2.16 may be proposed for 4, in which each osmium atom achieves an 18 electron configuration. The model shows donor-acceptor bonds from the vertices having four ligands to those having three. The expectation that these bonds would be slightly longer than normal covalent bonds is in accord with the lengths determined here. The electron density built up on Os(3) and Os(4) is relieved by the semi-bridging action of the carbonyl ligands.

The main difference between this approach and PSEPT is the assumption that the frontier orbitals are almost degenerate rather that separated by an energy gap large enough to maintain

the electrons in pairs. This difference is minor especially in view of the fact that the EHMO method, on which both the isolobal analogy and PSEPT are based, is well known to be subject to error regarding the computed energy levels of the orbitals. Hence, there is no strong reason to prefer one model over the other.

### 2.4.2 Fluxional Properties

Variable temperature <sup>13</sup>C NMR spectra revealed that the barrier to internuclear carbonyl exchange is very low in 4. The room temperature spectrum had only one resonance, a sharp singlet at  $\delta$ =181.1 ppm. This signal shifted slightly upfield as the sample was cooled; at -125 °C, the lowest temperature



Figure B.2.17. Possible Resonance Structures for Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>3</sub>.

attained, the peak was at  $\delta$ =179 ppm, and was considerably broadened (Figure B.2.17). These data are consistent with rapid exchange of all carbonyl ligands on the four osmium atoms probably via a bridge-terminal mechanism. Local exchange at separate vertices is not ruled out, but must occur in conjunction with internuclear exchange if the existence of only one carbonyl signal is to be explained. It is probable that the weak semi-bridging interactions between non-bonded Os and C atoms facilitate bridge-terminal carbonyl exchange.

When the sample was cooled to -91 °C, two other small peaks at  $\delta$ =202.0 and 195.2 ppm appeared in the spectrum. The spectrum obtained with the sample at -125 °C had a total of ten additional peaks, all of which were attributable to complex 2. Compound 4 is almost certainly not reverting back to 2 at this temperature. Rather it is probable that the two complexes were both present in solution because they were not completely separated during synthesis. Pure samples of 4 were obtained by chromatography on a silica gel column, a procedure which required that the column eluent be monitored by IR spectroscopy because the bands due to 2 and 4 overlapped somewhat. This was very difficult with samples that had been enriched with <sup>13</sup>CO because of the large number of CO stretches. The reason that signals due to 2 did not appear in the higher temperature the <sup>13</sup>C spectrum is that this complex was present in only trace amounts in the sample. The presence of sharp resonances due to 2 in the spectrum taken with the sample at -125 °C confirms that



the broadening of the signal due to 4 was indeed due to slowed exchange and not to loss of field homogeneity of the spectrometer or solvent viscosity.

## 2.5 Summary and Conclusions

The  $Os_4(CO)_n PMe_3$  (n=15,14,13) series constitutes the first fully characterized example where a 64 electron complex displays sequential ligand loss to 62 and then to a 60 electron species. The gross skeletal features of each of the three clusters conforms to that predicted by the current bonding theories. Nevertheless, many of the details of the structures are unexpected. The 64 electron species  $Os_4(CO)_{15}PMe_3$  (1) has a dative covalent metal-metal bond, i.e.,  $Os(CO)_4PMe_3$  acts as a two electron donor ligand in the cluster. The bonding is best described in terms of a triangular cluster with twelve two electron donor ligands, one of which is unusual.

Unlike most 62 electron species,  $Os_4(CO)_{14}PMe_3$  (2) is flat, and its perpheral framework is asymmetric. A novel bonding model involving Os-Os bond orders of 1.5 and 0.5 has been proposed to account for the unusual bond lengths. Similar non-integral bond orders may be applicable to other osmium clusters that have Os-Os bonds of unusual lengths.

The structure of 2 has been compared to that of  $(\mu_2-H)_2OS_4(CO)_{13}PMe_3$  (3) which has the more common butterfly framework with a dihedral angle of 112.2°. A hydrogen ligand

bridging the hinge has been proposed as the reason 3 is bent.

The 60 electron tetrahedral complex  $Os_4(CO)_{13}PMe_3$  (4) is stable despite a forecast to the contrary. We believe it is the first reported closed cluster with two ML<sub>4</sub> vertices.

As was noted earlier 4 is the most stable of the series. The rapid decomposition of 1 in solution at room temperature shows it to be the least stable. A temperature of 90 °C is necessary to cause ligand loss from 2 at a reasonable rate. The mass spectra are consistent with this ranking in that 1 and 2 show as the peaks of highest mass, ions due to the parent ion of 4<sup>+</sup>. It may be that increased metal-metal interactions inhibit decomposition; the 'spike' osmium in 1 takes part in only one metal-metal bond while each osmium in 4 is bonded to three others. Also, complex 4 may resist further ligand loss as there is no facile framework reorganization available that can make up for the loss of two more electrons. Further CO ligand loss from 4 was not studied although it did appear that it melted without decomposition at 183 °C.

The <sup>13</sup>C NMR spectra reveal that while 3 is nearly rigid at room temperature all members of the series  $Os_4(CO)_n PMe_3$  (n=15, 14, 13) undergo rapid ligand exchange. Furthermore the barrier to non-rigidity is lowered as the complexes become more compact. The low temperature limiting spectra for 1 was reached at -67 °C, for 2 at -115 °C while 4 was still fluxional at the latter temperature, which is about the lowest accessible to us.

The well known bridge-terminal carbonyl exchange mechanism accounts for the observed spectra of 1 provided that exchange involving the carbonyls of the Os(CO) PMe, liqand is assumed to occur concurrently with carbonyl exchange on the osmium triangle temperatures above -26 °C. We believe the remarkable at fluxional behaviour of 2 involves not only ligand exchange but skeletal rearrangement as well. The 'windshield wiper' mechanism proposed here is unique. The extremely low barrier to the non-rigidity exhibited by the tetrahedral complex 4 may be a consequence of the weak semi-bridging C-Os interactions. The lack of a low temperature limiting spectra for 4 prohibited development of a definite proposal for the ligand exchange mechanism.

There are several reasons why the bonding theories discussed in the introduction cannot predict the fine detail of these structures. Neither Lauher nor Teo take into account any ligand influence on localized bonding. The values calculated for the orbital energy levels using the EHMO method are a qualitative guide at best. Wooley has pointed out that the fundamental assumption of the EHMO method, i.e., that the resonance integral is proportional to the overlap integral, is invalid.<sup>208</sup> As a result, models based on EHMO calculations greatly underestimate the d orbital contribution to cluster bonding.

If accurate bonding models for osmium clusters are to be developed, relativistic effects must be considered. Drs. Malli and Arratia-Perez of this department and others have shown that

the d orbitals of third row transition metals are destabilized while the s orbitals are stabilized by relativity effects.<sup>209,210,211</sup> As a result, there is considerable s-d orbital mixing. It is unfortunate that full relativistic calculations are currently possible only on molecules of high symmetry, but such calculations are planned for the Os(CO)<sub>4</sub> and Os(CO)<sub>3</sub> fragments.<sup>212</sup>

#### CHAPTER 3

## EXPERIMENTAL PROCEDURES

# 3.1 Materials and Instrumentation

Unless otherwise stated, manipulations of starting materials and products were carried out under a nitrogen atmosphere using standard Schlenk techniques. Hexane was refluxed over potassium and methylene chloride over phosphorus pentoxide; they were distilled and stored under nitrogen before use. The complexes  $H_2OS_3(CO)_{10}$ ,  $OS_3(CO)_{11}MeCN$  and  $OS_3(CO)_{10}(MeCN)_2$  were prepared by literature methods,  $2^{13} \cdot 2^{14} \cdot 2^{15}$  and  $OS(CO)_4PMe_3$  was prepared as in Part A. The reagent  $Me_3NO$  was sublimed, then dissolved in MeOH before use.

Infrared spectra were obtained with a Perkin-Elmer 983 spectrophotometer; NMR spectra with a Bruker 400-MHz instrument and mass spectra with a Kratos MS-50 using a direct insertion probe at 220-250 °C and electron impact with an ionization voltage of 70eV. Microanalyses were performed by M. K. Yang of the Microanalytical Laboratory of Simon Fraser University. Analytical and mass spectral data are given in Table B.3.1. The infrared stretching frequencies in the carbonyl region, melting points and <sup>1</sup>H NMR resonances are listed in Table B.3.2.

Table B.3.1.	Analytical	and	Mass	Spectral	Data	for	Os <sub>4</sub>
Compounds.							

Compound	%Calcd.		۶Fo	und	Mass Spectrum <sup>a</sup>	
Compound	С	Н	С	Н	- Spectrum	
Os <sub>4</sub> (CO) <sub>15</sub> PMe <sub>3</sub>	17.20	0.72	17.09	0.76	1202 (P-2CO)+	
Os <sub>4</sub> (CO) <sub>14</sub> PMe <sub>3</sub>	16.60	0.74	16.84	0.67	1202 (P-CO)+	
$(\mu - H)_{2}Os_{4}(CO)_{13}PMe_{3}$	15.98	0.92	16.12	0.82	1176 (P-CO)+	
Os <sub>4</sub> (CO) <sub>13</sub> PMe <sub>3</sub>	16.00	0.76	16.11	0.79	1202 (P <sup>+</sup> )	

Most intense peak of parent ion envelope; in all cases the parent ion agreed with that simulated by computer.

## 3.2 Synthetic Methods

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#### 3.2.1 Preparation of $Os_4(CO)_{15}PMe_3$

METHOD I. A solution of of  $H_2Os_3(CO)_{10}$  in hexane (200 mg, .27 mmol in 25 mL) was stirred at room temperature with a large excess of  $Os(CO)_4PMe_3$  (230 mg, .60 mmol) for approximately 2 h. Evolved gases were allowed to escape through an oil bubbler. During this time  $Os_4(CO)_{15}PMe_3$  precipitated from solution as an orange powder. The supernatant liquid was decanted and the product washed with hexane ( $\approx 3x5$  mL), dried on a vacuum line and then dissolved in a minimum of hot  $CH_2Cl_2$  ( $\approx 10$  mL). Hexane was added to precipitate the product. The yield was 194 mg (.15 mmol, 57%) yield. The analytical sample was recrystallized from  $CH_2Cl_2$ . METHOD II. A hexane solution of  $Os_3(CO)_{11}MeCN$  (130 mg) and excess  $Os(CO)_4PMe_3$  (55 mg), was stirred at 60 °C for 2 h. The supernatant liquid was decanted and the orange precipitate,  $Os_4(CO)_{15}PMe_3$ , dried on a vacuum line. The yield was about 40% and was not improved by longer reaction times.

#### 3.2.2 Preparation of $Os_4(CO)_{14}PMe_{3}$

METHOD I. To a solution of  $Os_4(CO)_{15}PMe_3$  in  $CH_2Cl_2$ (typically 100 mg, .079 mmol in 25 mL), Me<sub>3</sub>NO in MeOH was added until the infrared band at 2086 cm<sup>-1</sup> had just disappeared. During this time the colour of the solution changed from orange to deep red. The solution was filtered through a short silica column ( $\approx$ 10x2.5 cm) and evacuated to dryness. The product was then extracted into hot hexane (3x20 mL). Removal of solvent gave the dark red crystalline product  $Os_4(CO)_{14}PMe_3$ ; the yield was 80 mg (0.065 mmol, 80%). The analytical sample was recrystallized from hot hexane.

METHOD II. A toluene solution of  $Os_3(CO)_{10}(MeCN)_2$  (~30 mg in 10 mL) and excess  $Os(CO)_4PMe_3$  (150 mg) was degassed with two freeze-thaw cycles and stirred at room temperature overnight. The solvent was removed on the vacuum line and the excess  $Os(CO)_4PMe_3$  sublimed at 40 °C onto a cold water probe to leave the crude product  $Os_4(CO)_{14}PMe_3$ , which was purified as in Method I.

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Table	Data

Compound		د CD	(cm - 1)				н 9	NMR (ppm) <sup>a</sup>	(), d m	
0s.(CO)PMe.	2115vw, 1974w,	2086m, 1961w,	2048s, 1916w(t	2033s, or)	2005m(	asym),	0.57,	(10.4)	148	
0s.(C0)PMe,	2116w. 2014m.	2069s, 2004w,	2046m, 1986vw,	2035vs, 1978m,	2028 ( 1951vw	sh). , 1927w	0.95,	(10.2)	128	
("-H);Ös.(CO),PMe,	2101w, 1985m,	2069s, 1976w	2052s,	2030s,	2016s,	2003s,	1.03,	(10.2)	204	
0s.(c0).PMe.	2091m,	2048vs,	2016s,	1992m			1.02,	(10.6)	183	

<sup>a</sup>J<sub>P-H</sub> values are in parenthesis. <sup>b</sup>Infrared spectrum in CH<sup>1</sup>Cl<sup>1</sup> solution. <sup>C</sup>Infrared spectrum in hexane solution.

## 3.2.3 Preparation of $(\mu - H)_2Os_4(CO)_{13}PMe_3$

A  $CH_2Cl_2$  solution of  $H_2Os_3(CO)_{10}$  (100 mg, .12mmol in 15 mL) was stirred with a slight excess of Os(CO) PMe<sub>3</sub> (25 mg, .13 A solution of Me<sub>3</sub>NO in MeOH was added dropwise until mmol). the purple colour was discharged and the infrared bands at 2094, 2074 and 2060 cm<sup>-1</sup> had disappeared. The resulting red solution was then filtered through a short silica gel column (10x2.5 cm) and evacuated to dryness. The product was extracted with cold hexane (4x5 mL) and chromatographed on a silica gel column (30x2.5 cm) with 1:2 CH<sub>2</sub>Cl<sub>2</sub>/hexane as the eluent. The first band, a faint yellow, was not indentified. The second band, which was orange, was the product and was followed by а broad dark brown band identified as  $Os_4(CO)_{14}PMe_3$ . Further bands were discarded without identification. The yield was 31 mq (.025 mmol, 26%) and the analytical sample was recrystallized from a 3:4  $CH_2Cl_2$ /hexane mixture.

# 3.2.4 Preparation of Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>3</sub>

A hexane solution of  $Os_4(CO)_{14}PMe_3$  (85 mg, .071 mmol in 20 mL) was placed in a sealed tube and degassed with two freeze-thaw cycles. It was then stirred at 90 °C for approximately 24h with frequent degassing (every 2h. when possible). The reaction was followed by infrared spectroscopy and when it appeared no further changes in the relative intensities of the CO bands were taking place the sample was

cooled and chromatographed on a silica gel column (30x2.5 cm) with 9:1 hexane/CH<sub>2</sub>Cl<sub>2</sub> as the eluent. The first band, (light yellow) was identified by infrared spectroscopy as  $Os_3(CO)_{11}PMe_3^{182}$ ; the second, brown-purple band was the product, and was collected in fractions as it was not well separated from the third band,  $Os_4(CO)_{14}PMe_3$ . Two further bands, light orange and purple, were not identified. The yield was 29 mg (.024 mmol, 34%) and the analytical sample was recrystallized from hexane.

## 3.3 Crystal Structure Determinations

#### 3.3.1 General

The following procedure was followed for each of the four compounds. Details for the individual complexes are given subsequently. A crystal of suitable size (see Table B.3.3) was mounted on an Enraf-Nonius CAD4-F diffractometer. Use of the diffractometer 'Search', 'Index' and 'Transformation' routines allowed approximate cell dimensions to be assigned on the basis of 25 high  $\theta$  angle reflections. The NRC 'Creduc' routine was used to check for possible alternate Bravais lattices.98 Subsequent examination of the systematic absences and Laue symmetry allowed these particular monoclinic space groups to be determined unambiguously. The triclinic space group was confirmed during structure refinement. Diffraction data were collected at  $20\pm1$  °C using graphite monochromated Mo-K  $a_{1}$  $(\lambda=0.7930\text{\AA})$  radiation. Background measurements were made by

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:	0s4(CO)18PMe1	0s4(C0)14PMe	(μ-H):Os.(CO),PMe,	0s4(CO),,PMe,	
Formula Wt.	1258	1230	1204	1202	
Colour	orange	deep red	red	burgandy	
Solvent	CHrClr	hexane	hexane/CH <sup>1</sup> Cl1	hexane	
Space Group	P.1	P2:/a	P2,/n <sup>C</sup>	P2./c	
Crystal System	triclinic	monoclinic	monoclinic	monclinic	
a, À.	12.473(2)	13.525(5)	15.541(6)	15.470(2)	
ь, А	13.325(2)	12.969(6)	11.013(3)	10.193(1)	
с, Х	17.547(2)	14.825(5)	16.280(4)	16.527(2)	
a, deg	109.05(1)	8	ł	1	
þ, deg	90.44(1)	99.14(3)	114.10(3)	113.452(9)	
γ, deg	94.60(1)	1	4 1		
۷, گ	2745	2567	2560	2390	
2	4	4	4	4	
d <sub>cald</sub> , g/cm <sup>1</sup>	3.036	3.185	3.153	3.337	
µ(Mo K <sub>d</sub> ) cm⁻¹	186	199 <sup>b</sup>	199	213	
Crystal Size, mm	0.11×0.29×0.28	0.34×0.16×0.11	0.11×0.18×0.27	0.24×0.19×0.08	
<sup>8</sup> Alternate P2./c no.1					1

-alternate P2:/C no.14. <sup>D</sup>An emperimentally based absorption correction was applied, see text. <sup>C</sup>P2:/n, non-standard setting for P2:/C; equivalent positions are: x,y,z; 1/2-x,1/2+y,1/2-z; -x,-y,-z; 1/2+x,1/2-y,1/2+z.

extending the scan range by 25% at each side of the scan. Monitoring of two standard relections every one or two hours revealed no significant crystal decay. A Lorentz polarization correction was applied. Accurate cell dimensions were assigned the basis on of 25 [24 for  $Os_{4}(CO)_{15}PMe_{3}$  and 19 for  $(\mu-H)_2Os_4(CO)_{13}PMe_3$ ] carefully centered high angle  $(2\theta>21^\circ)$ reflections widely scattered in reciprocal space. Psi scans were run for a number of reflections (typically about six) throughout the  $2\theta$  range of data collection and having chi near 90°. Crystal faces were measured and indexed using a locally modified optical telescope with a filar eyepiece and the diffractometer 'Micros' and 'Micror' routines.

The osmium atoms were located by electron density methods and other non-hydrogen atoms by alternating Fourier difference maps with least squares refinement. Since the linear absortion coefficients were so large,  $\approx 200 \text{ cm}^{-1}$ , great care was taken with the absorption correction. Analytical corrections were applied to those reflections for which psi scans had been obtained and the results were compared to those of the empirical method. Small adjustments in the values of the crystal dimensions were made when necessary, to ensure that the intensity variation as determined by the analytical method matched that of the empirical method. In general, the analytical correction was preferred to the empirical one; see the following individual structure determinations for details. The strong inner data

showed evidence for extinction in all four structures as |KF values were less than  $|F_c|$  values. Extinction was added as a refinable parameter if its size, significance and the improvement in the agreement so warranted, otherwise those reflections suffering most noticeably from extinction were removed from the refinement process (see Table B.3.3).216 Unit weights were used during the initial stages of refinement for all four structures. After anisotropic refinement the weight,  $\omega$ , for each reflection was set to be  $\omega = [\sigma^2(F) + KF^2]^{-1}$  and the value of K was adjusted to remove trends in the average  $\omega\Delta^2$ values as a function of  $|F_{0}|$  and  $\sin\theta/\lambda$  in the error analysis. There were no significant trends in the final error analyses. In each case the largest peaks in the final Fourier difference map were in the vicinity of the osmium atoms. Crystallographic data are given in Table B.3.3 and details of data collection in Atomic scattering factors which included Table B.3.4. anomalous dispersion were taken from reference 95. Diagrams of the molecules as computed by the SNOOPI program<sup>217</sup> are shown in Figures B.2.1, B.2.6, B.2.10 and B.2.16. Bond lengths and angles are given in Tables B.2.1 to B.2.4. Final atomic coordinates, anisotropic thermal parameters and structure factors are listed in the appendix. The computer programs used were from "The Vax 750/780 Crystal Structure System."98

Compounds.
Os 4
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B.3.4.
Table

	0s*(CO)'*PMe;	0s4(CO)14PMe1	(J-4),0S4(CO),PMe,	Os.(CO).,PMe,
scan method	u-20	u-20	u-20	u-20
scan range, 20, deg	0.0-45.0	0.0-45.0	0.0-45.0	0.0-45.0
scan width, 20, deg	1.3	1.3	1.3	1.3
scan rate, 20, deg/min	1.4-6.6	1.4-6.6	1.4-6.6	1.4-6.6
collection range	±h,±k,+1	±h,+k,+1	±h,+k,+1	±h,+k,+1
transmission coeffectent range	0.120-1.00 <sup>8</sup>	0.0555-0.146	0.0141-0.164	0.0249-0.422
unique relections	7167	3331	3544	3104
observed reflections <sup>b</sup>	5172	2424	2503	2060
variables in final model	386	325	308	163
RIG	0.0367	0.0385	0.0325	0.0470
R d	0.0407	0.0479	0.0380	0.0601
K factor in wt. scheme	0.0004	0.0008	0.0004	0.0006
gof	1.3508	1.3112	1.4231	1.6416
largest shift <sup>e</sup>	0.03	0.00	0.00	0.02
map ø e/Å <sup>1 f</sup>	0.25	0.27	0.23	0.39
largest peak e/Å <sup>1</sup> <sup>g</sup>	1.55	1.59	1.79	2.18
extinction parameter(X10-•) <sup>h</sup>	22(1.3)	:	6.7(1.3)	23(2.6)

<sup>a</sup>Empirical absorption correction, see text.

 ${}^{D}I>2.3d(I)$  except for ( $\mu$ -H),OS,(CO), PMe, for which I>3.0 d(I).  ${}^{C}R_1=E|F_O|-|F_O|/E|F_O$   $d_{R_1}=[Lu(|F_O|-|F_O)^2/Lu|F_O|^2|^{1/2}$ 

fStandard deviation of the final Fourier difference map. <sup>9</sup>Largest peak in the final Fourier difference map.

<sup>h</sup>Parameter is g in the formula  $F_{c}' = k |F_{c}| (1+g |F_{c}|^{2} L_{p_{1}}/p_{1})^{-1/4}$ where  $F_{c}'$  is the corrected, calculated structure factor, k is a scale factor, Lp is the Lorentz polarization factor

and  $P_n = 1 + \cos^{1} 2\theta$ .

## 3. 3. 2 Os 4 (CO) 1 5 PMe 3

After all the atoms had been located, refinement proceeded quickly with only the osmium atoms having anisotropic thermal parameters  $(R_1=0.082)$ . Since the crystal had been cut to appropriate size it was necessary to define some 16 crystal faces with indexes as high as 25 in order that an analytical absorption correction<sup>97</sup> could be applied. Extinction was added as a refinable parameter.96 (It was noted, in particular, that 1,0,2 and 2,0,0 reflections had much better agreement after the this proceedure)<sup>218</sup> ( $R_1=0.041$ ). Initial positions of hydrogen atoms on the methyl carbons in each of the two molecules in the asymmetric unit were located in a Fourier difference map using inner data only. These were used to calculate the positions of the second and third hydrogen atoms of the methyl groups. This procedure was applied to five of the six methyl groups; hydrogen atoms on the sixth methyl group could not be located. In view of the difficulty in defining crystal faces it was decided to try an empirical absorption correction, 94 which proved advantageous (R1=0.038). At this point the phosphorus and methyl carbon atoms were allowed to vary anisotropically and the weighting adjusted  $(R_1=0.037)$ . Finally the scheme was structure was refined to completion using block diagonal methods and the positions of the hydrogen atoms were recalculated.

# 3. 3. 3 Os 4 (CO) 1 4 PMe 3

Isotropic refinement of all non-hydrogen atoms proceeded quickly  $(R_1=0.088)$ , however correction for absorption proved difficult. The variations of intensity as measured by the psi scans were not consistent for the different reflections, nor was there any match with the analytical correction. It was not surprising, therefore, that neither the empirical<sup>94</sup> nor the analytical<sup>97</sup> correction improved the value of the residual, even though the measurement of crystal dimensions was carefully checked. Since the transmission factors for the analytical correction appeared to be too small (0.00529-0.06219) it was decided to decrease the absorption coefficient from the calculated value of 199 cm<sup>-1</sup> in a systematic way. It was found that a  $\mu$  value of 100 cm<sup>-1</sup> gave the best results (R<sub>1</sub>=0.064). The reason for this difficulty was very probably that the crystal had re-entrant angles making it impossible for the computer program to handle the absorption correction properly. All non-hydrogen atoms were allowed to vary anisotropically  $(R_1=0.0393)$ , and then the weighting scheme was adjusted  $(R_1=0.039)$ . Hydrogen atoms were included at calculated positions<sup>219</sup> with a single isotropic temperature ( $R_1=0.039$ ). Addition of extinction as a refinable parameter did not improve agreement and gave an unreasonably small extinction the coefficient (2.4(.26)x10<sup>-8</sup>), but four reflections were removed from the refinement as they suffered from extinction.<sup>220</sup> The data were then refined to completion using full matrix least

squares.

# 3.3.4 $(\mu - H)_2 Os_4 (CO)_{13} PMe_3$

Full-matrix least-squares refinement with only the osmium and phosphorus atoms located and with these atoms assigned isotropic thermal parameters gave a residual  $(R_1)$  value of 0.19. Application of an analytical absorption correction greatly improved the agreement  $(R_1=0.11)$  as did location of the other non-hydrogen atoms (R<sub>1</sub>=0.052). Anisotropic temperature factors were then added and the weighting scheme was adjusted. Methyl hydrogen atom positions were generated from initial positions located on a Fourier difference map generated using inner data only  $(R_1 = 0.036)$ . The -2,2,2 reflection was omitted from structure refinement as it had an anomalously large  $\omega\Delta^2$  value i.e., 441. (The next highest  $\omega\Delta^2$  value was 74.) Because the error analysis showed the weak data had a large average  $\omega\Delta^2$  it was decided to flag unobserved those reflections for which  $I \leq 3\sigma(I)$ . This procedure reduced the number of observed reflections from 2597 to 2503 and the residual  $(R_1)$  value to 0.033. Attempts to locate the bridging hydrogens usinq difference Fourier synthesis on inner data only were not successful. Final convergence was achieved using full matrix least-squares refinement.

## 3.3.5 Os 4 (CO) 13 PMe 3

Initial phasing difficulties were overcome by application of analytical absorption correction. This was necessary an because the transmission coefficients ranged from 0.0249 to 0.422. The heavy and light atoms were then easily located, and refined with anisotropic thermal parameters assigned to the osmium atoms only  $(R_1=0.051)$ . At this point the phosphorus and oxygen atoms were allowed anisotropic thermal parameters and attempts were made to locate the hydrogen atoms using Fourier difference synthesis on inner data only. This proved to be a non-trivial matter. Initially only one hydrogen atom could be located. The methyl carbon atoms were allowed anisotropic parameters and two hydrogen atoms were added at calculated positions. Full matrix refinement reduced the R1 value to 0.047. The weighting scheme was then adjusted and the -5,3,8 reflection which had an anomalously high  $\omega\Delta^2$  value of 401 was eliminated from the refinement process. (No other reflection had an  $\omega \Delta^2$  value greater than 150.) At this point it was possible to locate two more hydrogen positions from the difference synthesis and consequently calculate the position of the remaining four  $(R_1=0.046)$ . Analysis of low angle strong data revealed several reflections having large  $\omega \Delta^2$  values for which the observed structure factors were smaller than the calculated values suggesting the data might suffer from extinction. This, combined with the fact that not all thermal parameters for the oxygen and carbon atoms were well behaved led

to the decision to return to isotropic parameters for these Full matrix refinement produced an R, value of 0.047. atoms. Addition of extinction as a refinable parameter reduced the residual  $R_1$  to 0.045. Further investigation of the weighting scheme led to a K value of 0.0006 being assigned. A second attempt at allowing the oxygen atoms anisotropic thermal parameters was made but the results were no better than those of the first trial. It was decided to leave all oxygen and carbon The hydrogen atom positions were atoms isotropic. then recalculated and the structure was refined to completion by full matrix least squares. One large peak  $(2.2e/Å^3, 5.6\sigma)$  in the final Fourier difference map was located inside the tetrahedron of osmium atoms at a distance of 1.319(1)Å from Os(4), but there was no evidence to suggest it had any chemical significance.

# Supplemental Tables

Table S.1. Fractional Coordinates for Ru(CO), AsPh3

ATOM	x	Y	Z	BISO
RU	0.35373( 4)	0.19970( 4)	0.23603( 5)	4.57(2)
AS	0.20818( 5)	0.31855( 5)	0.13740( 5)	4.18( 2)
C1	0.2193( 5)	0.0705(5)	0.2070( 6)	6.2(3)
01	0.1387( 4)	-0.0043( 4)	0.1941( 5)	9.0( 3)
C2	0.3699( 5)	0.3512( 5)	0.4186( 6)	5.5( 3)
02	0.3770( 4)	0.4417( 4)	0.5281( 4)	7.0(2)
C3	0.4571( 5)	0,1771(5)	0.0764( 6)	6.1(3)
03	0.5152( 4)	0.1628( 5)	-0.0202( 5)	8.7(3)
C4	0.4686( 5)	0.1122( 5)	0.3134( 6)	6.4(3)
04	0.5392( 4)	0.0582( 4)	0.3591( 5)	9.7(3)
C11	0.1925( 4)	0.2555( 5)	-0,0744( 5)	4.6(3)
C12	0.1768( 5)	0.1237( 6)	-0.1561( 6)	6.3(3)
C13	0.1618( 6)	0.0733('6)	-0.3085( 6)	7.7( 4)
C14	0.1657( 6)	0.1601( 8)	-0.3739( 6)	8.9( 6)
C15	0.1809( 6)	\0.2903( 7)	-0.2955( 7)	9.3( 5)
C16	0.1940( 5)	0.3399( 6)	-0,1438( 6)	6.7( 4)
C21	0.2510( 4)	0.5048( 5)	0.2005( 5)	4.8(3)
C22	0.1593( 5)	0.5956( 5)	0.2319( 5)	5.4( 3)
C23	0.1908( 6)	0.7290( 5)	0,2790( 6)	6.7( 4)
C24	0.3150( 7)	0.7693( 6)	0,2932( 7)	8.2( 5)
C25	0.4056( 6)	0.6821( 7)	0.2627( 8)	9.4(5)
C26	0.3747(5)	0.5474( 6)	0.2164(7)	7.3(4)
C31	0.0355(4)	0.3147(4)	0.1864( 5)	4.2(2)
C32	-0.0632( 5)	0.2969( 6)	0.0869( 6)	6.2( 4)
C33	-0.1869( 5)	0.2937(7)	0.1231( 7)	7.5( 5)
C34	-0.2120( 5)	0.3095( 6)	0.2592(7)	6.8( 4)
C35	-0.1163( 6)	0.3277(7)	0.3604( 6)	8.0( 5)
C36	0.0083( 5)	0.3305( 6)	0.3245( 6)	6.7( 4)
H12	0.171( 4)	0.066( 4)	-0,118( 5)	8.(0)
H13	0.158( 5)	-0.018( 5)	-0.365( 5)	8.( 0)
H14	0.152( 4)	0.126( 5)	-0.461( 5)	8.( 0)
H15	0.178( 5)	0.359( 5)	-0.335( 5)	8.( 0)
H16	0.207(4)	0.432(4)	-0.089( 5)	8.( 0)
H22	0.080( 4)	0.570(4)	0.221(4)	8.(0)
H23	0.128( 4)	0.791(4)	0.305(5)	8.( 0)
H24	0.328(4)	0.85/( 5)	0.332(5)	8.( 0)
H23	0.479(3)	0.681( 5)	0.24/( 5)	8.(0)
H20	0.43/( 4)	0.486(4)	0.189( 5)	8.(0)
H32	-0.051(4)	0.286(4)	-0.001(4)	8.(0)
H33	-0.251(4)	0.289(5)	0.060(5)	8.(0)
H.54	-0.274(4)	0.308(4)	0,285( 5)	8.(0)
H35	-0.128( 4)	0.334( 4)	0.437( 5)	8.( 0)
H36	0.067( 4)	0.347( 4)	0.387(5)	8.(0)
ì

Atom	U11	U22	Ū33	U12	U <sub>13</sub>	U23
Ru	5.53(2) 5.21(3)	5.80(3) 5.55(3)	6.03(3) 5.12(3)	0.27(2) - $0.02(2)$	-0.13(2) 0.00(2)	2.77(2) 2.6(3)
CI	8.4(4)	6.6(4)	8.7(4)	-0.2(3)	-1.2(3)	4.2(3)
C2	5.9(3)	7.5(4)	7.5(4)	0.0(3)	-0.5(3)	3.2(3)
C3	6.1(3)	8.7(4)	8.6(4)	1.2(3)	0.4(3)	3.9(3)
C4	8.2(3)	7.6(3)	8.3(4)	0.4(3)	-1.4(3)	2.8(3)
01	10.9(3)	8.3(3)	14.8(4)	-2.8(2)	-1.2(3)	6.0(3)
02	10.2(3)	8.8(3)	7.9(3)	-0.4(2)	-0.1(2)	-1.0(2)
03	8.5(3)	14.5(4)	10.0(3)	1.5(3)	3.3(2)	5.2(3)
04	12.5(4)	11.6(4)	12.8(4)	3.3(3)	-3.2(3)	5.5(3)
C11	4.8(3)	6.7(3)	5.6(3)	0.3(2)	0.5(2)	2.6(3)
C12	9.5(4)	8.5(4)	5.9(4)	-0.4(3)	-0.3(3)	2.3(3)
C13	12.2(5)	10.2(5)	6.8(4)	0.7(4)	0.3(4)	0.7(4)
C14	11.4(5)	16.8(7)	5.6(4)	2.3(5)	1.6(4)	3.4(4)
C15	11.2(4)	16.9(3)	8.2(5)	1.0(5)	1.8(4)	7.5(5)
C16	7.7(4)	10.4(3)	7.2(4)	0.6(3)	1.0(3)	5.0(4)
C21	6.3(3)	6.3(3)	5.6(3)	-0.4(3)	-0.8(2)	3.4(3)
C22	8.0(4)	6.2(3)	6.3(3)	-0.5(3)	01(3)	3.2(3)
C23	11.0(5)	6.7(4)	7.5(4)	0.2(3)	-0.8(3)	3.4(3)
	14.2(6)	6.4(4)	10.5(5)	-2.5(4)	-4.2(4)	4.5(4)
	8.4(5)	10.4(5)	17.8(5)	-2.9(4)	-3.2(4)	8.3(5)
	/.4(4)	7.4(4)	13.0(4)	-1.1(3)	-1.5(4)	5.8(4)
	6.1(3)	5.4(3)	4.4(3)	0.4(2)	0.1(2)	1.9(2)
L32	6.0(3)	12 6(4)	0.3(3)	-0.8(3)	0.4(3)	3.3(3)
C33	$0 \circ   (4)$	10 2(5)	0./(4/	-0.0(4)	0.5(3)	3.0(4)
C34 725	3.3(4) 0.0(5)	10.3(5)	7 1(4)	-0.4(3)	2.2(3)	2.9(4)
	3.3(3)	13.3(6)	/ . I (4)	0.3(4)	3.4(3)	4.4(4)
	/.3(4)	12.2(3)	3.3(4)	0.4(3)	0.5(3)	3.0(4)

U<sub>ij</sub> values have been multiplyed be 100.

Ru(CO) AsPh3

a) Bond Lengths (Å).

C(11)-C(12)	1.357(7)
C(12)-C(13)	1.392(8)
C(13)-C(14)	1.361(10)
C(14)-C(15)	1.337(10)
C(15)-C(16)	1.386(8)
C(16)-C(11)	1.368(7)
C(21)-C(22)	1.374(7)
C(22)-C(23)	1.383(7)
C(23)-C(24)	1.362(9)
C(24)-C(25)	1.339(10)
C(25)-C(26)	1.397(8)
Ç(26)-C(21)	1.362(7)
C(31)-C(32)	1.356(7)
C(32)-C(33)	1.383(7)
C(33)-C(34)	1.341(9)
C(34)-C(35)	1.344(9)
C(35)-C(36)	1.391(8)
C(36)-C(31)	1.366(7)

b) Bond Angles (\*)

C()	11)-C(12)-C(13)	121.3(5)
C(	12)-C(13)-C(14)	118.0(6)
¢()	13)-c(14)-c(15)	121.6(6)
C(	14)-C(15)-C(16)	120.0(6)
c	15)-C(16)-C(11)	119.9(6)
C(	16)-C(11)-C(12)	119.0(5)
c	21)-C(22)-C(23)	121.1(5)
c(	22)-C(23)-C(24)	118.8(5)
C(	23)-C(24)-C(25)	120.9(5)
C(	24)-C(25)-C(26)	120.6(6)
C(	26)-C(21)-C(22)	119.1(5)
C(	31)-C(32)-C(33)	121.4(5)
C(	32)-c(33)-c(34)	120.3(5)
C()	33)-c(34)-c(35)	119.7(5)
C(	34)-C(35)-C(36)	120.3(5)
C(	35)-c(36)-c(31)	120.8(5)
C()	36)-C(31)-C(32)	117.6(5)

Table S.4. Structure Factors for Ru(CO)<sub>4</sub>AsPh<sub>3</sub>

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Table S.5. Fractional Coordinates for Ru(CO), SbMe3

atoa	. X	у	z
Ru	0.(-)	0.(-)	0.(-)
Sb	0.(-)	0.(-)	-0.27183(9)
C(1)	0.(-)	0.(-)	0.196(2)
C(2)	-0.206(1)	-0.052(1)	-0.0066(8)
C(3)	-0.176(1)	-0.194(1)	-0.3682(9)
0(1)	0.0(-)	0.0(-)	0.317(1)
0(2)	-0.326(8)	-0.080(1)	-0,0091(9)

## Table S.6. Thermal Parameters for Ru(CO)<sub>4</sub>SbMe<sub>3</sub>

ATOM	U11	022	U33	U12	U13	U23
RU	4.91(4)	4.91(0)	3.81( 9)	2,45( 0)	0.00( 0)	0.00( 0)
SB	5,34(4)	5.34( 0)	3.48(7)	2.67(0)	0.00( 0)	0.00( 0)
C1	7.6( 5)	7.6( 0)	4.6( 6)	3.8( 0)	0.0( 0)	0.0( 0)
C2	6.3( 4)	8,8( 5)	4.8(3)	4.0( 4)	0.1(3)	0.6(3)
C3	6.8( 4)	6.6( 4)	6.7(5)	2.2(3)	-0.9(3)	-1.7(3)
01	11.3( 6)	11.3( 0)	3.9( 4)	5.6( 0)	0.0( 0)	0.0( 0)
02	6.4(4)	15.2( 6)	10.7( 5)	5.2(4)	0.8(3)	1.4( 5)

TEMP=-2(PI)\*\*2(U11\*H\*H\*ASTAR\*ASTAR+---+'2\*U12\*H\*K\*ASTAR\*BSTAR+---)

THE UIJ VALUES HAVE BEEN MULTIPLIED BY 100.

Table S.7. Structure Factors for Ru(CO)<sub>4</sub>SbMe<sub>3</sub>

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16	F0 FC SIG K KF0 FC SIG 7 58 182* 3 240 238 A	3, K, 9 5, K, 10		11 123 7 4 143 137 7 6 70 184* 6/K 10	4, K, 9 2 181 176 8	67 166 6 77 10 45 64 24x 0 215 199 7	5, K, 9 -6, K, 11	06 110 10 7 260 246 6	27 70 45# -5,K,11 6,K,9 8 236 227 7	17 126 9 -4, K, 11	71 68 16* 6 326 310 5	7, N, 9 -3, N, 11 71 53 '16# 4 394 392 4	B, K, 9 7 286 288 6	7 46 1904	49 37 23* 8 223 221 7	5, N, 10 -1, N, 11 32 173 7 3 411 428 4	5, K, 10 6 339 335 5 47 742 4 6 884 11	75 162 8 1 513 521 4	4, K, 10 4 382 390 5 10 200 4 7 234 230 2	3, K, 10 1, K, 11	24 314 3 415 4 30 194 7 5 327 316 5	2, K, 10 2, K, 11	20 437 4 0 4/2 4/0 4 76 273 5 3 363 363 5	51 142 9 6 220 219 7	, K, 10 3, K, 11 44 349 4 1 410 417 5	17 219 7 4 280 274 6	0, K, 10 4, K, 11 54 379 4 2 330 341 5	35 292 5 5, K, 11	24 129 10 0 366 357 5 0 1 × 10		0 308 5 1 269 267 6	33 226 6 -4, K, 12	29 Ki 10 - 5 211 195 6 51 354 4 - 5 211 195 6		13 132 9 -2, K, 12	5, K, 10 4 224 215 6 21 298 5 -1, K, 12	33         198         7         2         265         259         5	1, K, 10 5 214 215 7 17 350 5 0, K, 12	
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• 10SIG	K KFO FC SIG K KFO FC SIG 8 7 58 182* 3 240 238 A	3, K, 9 5, K, 10		5 111 123 7 4 143 137 7 6 6 70 184* 67 K 10	4, K, 9 2 181 176 8	1 167 166 6 77 Nº 10 4 45 64 24x 0 215 199 7	5, K, 9 -6, K, 11	2 106 110 10 7 260 246 6	5 27 70 45# -5, K, 11 6, K, 9 8 236 227 7	0 117 126 9 -4, K, 11	3 71 68 16# 6 326 310 5 2 6 6	1 71 53 '16* 4 394 392 4	8, K, 9 7 286 288 6	2 / 46 190#	0 49 37 23* 8 223 221 7	-6/ N 10 -1/ N 11 -6/ N 12 -6/	-5, K, 10 6 339 335 5 2 22 22 2 6 6 4 1	9 175 162 8 1 513 521 4	-4, K, 10 4 382 390 5 7 210 200 4 7 234 230 5	-3, K, 10 1, K, 11	0     322     314     0     2     370     412     4       8     200     194     7     5     327     316     5	-2, K, 10 2, K, 11	6 276 273 5 3 363 363 5	<u>9</u> 151 142 <u>9</u> <u>6</u> 220 219 <u>7</u>	1, K, 10 3, K, 11 4 344 349 4 1 410 417 5	7 217 219 7 4 280 274 6	0, K, 10 4, K, 11 2 354 379 4 2 330 341 5	5 285 292 5 5, K, 11	8 124 129 10 0 366 357 5 0 12 K-10 7 7 212 222 0	0 462 459 4 6, K, 11	3 300 308 5 1 269 267 6	6 233 226 64, K, 12	2, K, 10 , 5 211 195 6 1 751 754 4 - 71 K, 12	4 294 290 5 6 188 195 8	7 143 132 9 -2, K, 12	3, K, 10 4 224 215 6 2 291 298 5 -1, K, 12	5 203 198 7 2 265 259 5	4, K, 10 5 5 214 215 7 0 347 350 5 0, K, 12	
DFC+ 10SIG	K KFO FC SIG K KFO FC SIG 8 7 58 182* 3 240 238 A	3, K, 9 5, K, 10		5 111 123 7 4 143 137 7 6 6 70 184 <b>*</b> 67 K3 10	4, K, 9 2 181 176 8	1 167 166 6 77 Nº 10 4 45 64 24x 0 215 199 7	5, K, 9 -6, K, 11	2 106 110 10 7 260 246 6	5 27 70 45# ~5, K, 11 6, K, 9 8 236 227 7	0 117 126 9 -4, K, 11	3 71 68 16# 6 326 310 5 2 6 6 2 6 326 310 5	7, N, 9 -3, N, 11 1 71 53 '16# 4 394 392 4	8, K, 9 7 286 288 6	2 7 46 190427 11 1 9, 13, 9 5 379 378 5	0 49 37 23* 8 223 221 7	-6/ N, 10 -17 N, 11 B 182 173 7 3 411 428 4	-5, K, 10 6 339 335 5 227 242 4 0. K, 11	9 175 162 8 1 513 521 4	-4, K, 10 4 382 390 5 7 210 200 4 7 234 230 5	-3, K, 10 1, K, 11	B 200 194 7 5 327 316 5	-2, K, 10 2, K, 11	6 276 273 5 3 363 363 5	9 151 142 9 6 220 219 7	1, K, 10 3, K, 11 4 344 347 4 1 410 417 5	7 217 219 7 4 280 274 6	0, K, 10 4, K, 11 2 354 379 4 2 330 341 5	5 285 292 5 5, K, 11	8 124 129 10 0 366 357 5 0 1 8 10 10 2 10 366 357 5 0	0 462 459 4 6, K, 11	3 300 308 5 1 269 267 6	6 233 226 6 -4, K, 12	2, K, 10 - 5 211 195 6 1 251 254 4 - 21 K, 12	4 294 290 5 6 188 195 8	7 143 132 9 -2, K, 12	3, K, 10 4 224 215 6 2 291 298 5 -1, K, 12	5 203 198 7 2 265 259 5	0 347 350 5 214 215 7 0 347 350 5	
1,10FC, 10SIG	) K KFO FC SIG K KFO FC SIG B 7 58 182# 3 240 238 A	5 . 3, K, 9 5, K, 10		5 6 6 70 184¥ 67 K10	4, K, 9 2 181 176 8	1 1 167 166 6 77 17 10 1 4 45 64 24x 0 215 195 7	5, K, 9 -6, K, 11	2 106 110 10 7 260 246 6	0 5 27 70 45# -5, K, 11 6, K, 9 8 236 227 7	0 117 126 9 -4, K, 11	• 3 71 68 16* 6 326 310 5 2 6 6 15* 5 326 310 5	i 1 71 53 164 4 394 392 4	8 Ki 9 7 286 288 6	2 7 46 190¥21 N 11 3 91 K9 9 5 379 378 5	0 49 37 23* 8 223 221 7	14 – 6, N, 10 – 1, N, 11 8 182 173 7 3 413 428 4	14 -5, K, 10 6 337 335 5 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	-4, K, 10 4 382 390 5 - 7 210 200 6 7 234 230 7	× -3, K, 10 - 1, K, 11	8 200 194 7 5 327 316 5	-2, K, 10 2, K, 11	3     433     434     4     4     4     4       1     6     276     273     5     3     363     5	9 151 142 9 6 220 219 7	1, K, 10 3, K, 11 4 344 349 4 1 410 417 5	7 217 219 7 4 260 274 6	* 0, K, 10 4, K, 11 2 354 379 4 2 330 341 5	5 285 292 5 5, K, 11		0 462 459 4 6, K, 11	3 300 308 5 1 269 267 6	6 233 226 6 -4, K, 12	* 1 251 X 10 - 5 211 195 6 * 1 251 754 4 - 72 K 12		7 143 132 9 -2, K, 12	3, K, 10 4 224 215 6 2 291 298 5 -1, K, 12	5 203 198 7 2 265 259 5	* 0 347 350 5 214 215 7 * 0 347 350 5 0 <i>Ki</i> 12	
OF0,10FC, 10SIG	SIG K KFO FC SIG K KFO FC SIG 8 7 58 182# 3 240 238 A	6 . 3, K, 9 5, K, 10		5 6 6 70 184 <b>4</b> 64 K 10	4, K, 9 2 181 176 8	4 1 167 166 6 77 N 10 5 4 45 64 24x 0 215 199 7	7 5, K, 9 -6, K, 11	2 106 110 10 7 260 246 6	4 5 27 70 45# -5, K, 11 7 6, K, 9 8 236 227 7	0 117 126 9 -4, K, 11	6 3 71 68 16# 6 326 310 5 2 6 6 3 26 310 5	5 1 71 53 164 4 394 392 4	B B, K, 9 7 286 288 6	8 2 7 46 190#27 N 11 8 9 1/1 9 5 379 378 5	0 49 37 23* 8 223 221 7	45# -6/ K7 10 -1/ K7 11 8 182 173 7 3 411 428 4	18# -5, K, 10 6 339 335 5 2 22 22 22 2	175* 9 175 162 8 1 513 521 4	-4, K, 10 4 382 390 5 9 7 210 200 4 7 234 230 7		7 8 200 194 7 5 327 316 5	13 -2, K, 10 2, K, 11	8 6 276 273 5 3 363 363 5	14 9 151 142 9 6 220 219 7		8 7 217 219 7 4 280 274 6	34× 0, K, 10 4, K, 11 2 354 379 4 2 330 341 5	6 5 285 292 5 5, K, 11		0 462 459 4 67 K1 11	4 3 300 308 5 1 269 267 6	5 6 233 226 6 -4, K, 12	9 2, K, 10 , 5 211 195 6 1914 1 751 754 4 -72 K, 12	171* 1 201 201 1 201 201 1 201 201 1 201 201	5 7 143 132 9 -2, K, 12	7 3, K, 10 4 224 215 6 14 2 291 298 5 -1, K, 12	5 203 198 7 2 265 259 5	6 4, K, 10 5 2 2 4 2 15 7 171* 0 347 350 5 0, K, 12	
<pre>xE 10F0,10FC, 10SIG</pre>	) SIG K KFO FC SIG K KFO FC SIG 1 B 7 58 182# 3 240 238 A	0 6 . 3, K, 9 5, K, 10		0 5 6 6 70 184× 61 K 10	4, K, 9 2 181 176 8	0 4 1 167 166 6 77 N 10 5 4 45 64 24x 0 215 199 7	7 5, K, 9 -6, K, 11		7 5 27 70 45# -5, K, 11 7 6, K, 9 8 236 227 7	0 117 126 9 -4, K, 11	1 6 3 71 68 16* 6 326 310 5 2 6 3 2 71 68 16* 7 326 310 5	5 1 71 53 164 4 394 392 4	B B K 9 7 286 288 6	1 2 7 46 190¥27 Kr 11 8 9, Kr 9 5 379 378 5	0 49 37 23* 8 223 221 7	45 <b>x</b> -6/N/10 -1/N/11 - 45x -	184 -57 Kr 10 6 339 335 5 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	175* 9 175 162 8 1 513 521 4	4, K, 10 4 382 390 5 9 7 210 200 4 7 234 230 7		7 8 200 194 7 5 327 316 5	13 -2, K, 10 2, K, 112, K, 102, K,	8 6 276 273 5 3 363 363 5	14 9 151 142 9 6 220 219 7	· -1, K, 10 3, K, 11 7 4 344 349 4 1 410 417 5	8 7 217 219 7 4 280 274 6	· 34# 0º K' 10 4 4º K' 11 2 354 379 4 2 330 341 5	6 5 285 292 5 5, K, 11		0 462 459 4 6, K+ 11	4 3 300 308 5 1 269 267 6	5 6 233 226 6 -4, K, 12	9 2, K, 10 5 211 195 6 6 214 195 6		5 7 143 132 9 -2, K, 12	7 3, K, 10 4 224 215 6 14 2 291 298 5 -1, K, 12	5 203 198 7 2 265 259 5	6 4, K, 10 5 214 215 7 121* 0 347 350 5 0, K, 12	
i ARE 10FD,10FC, 10SIG	FC SIG K KFO FC SIG K KFO FC SIG B 8 7 58 182* 3 240 238 A	239 6 . 3, K, 9 5, K, 10		23/ 4 3 111 123 7 4 143 137 7 279 5 6 6 70 184 <b>4</b> 6/ K 10	B 4, K, 9 2 181 176 B	343 5 4 1 169 166 6 77 17 10 343 5 4 45 64 24% 0 215 199 7	199 7 5, K, 9 -6, K, 11	B 2 106 110 10 7 260 246 6	410 4 5 27 70 45¥ -5, K, 11 223 7 6, K, 9 8 236 227 7	B 0 117 126 9 -4, K, 11	238 6 3 71 68 16# 6 326 310 5 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	8	182 8 8, K, 9 7 286 288 6	8 2 7 46 1904	9 0 49 37 23* 8 223 221 7	46 45# -6/N,10 -1/N,11 9 8 182 173 7 3 411 428 4	63 18# -5, K, 10 6 339 335 5 e 2, 2, 2, 2, 2, 2, 1	() 175* 9 175 162 8 1 513 521 4	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	47 182* -3, K, 10 1, K, 11	7 7 8 200 194 7 5 327 316 5 139 7 8 200 194 7 5 327 316 5	B6 13 -2, K, 10 2, K, 11	7 3 433 437 45 7 4 0 4/2 4/0 4 119 8 6 276 273 5 3 363 363 5	46 14 9 151 142 9 6 220 219 7	9	116 8 7 217 219 7 4 280 274 6	45 34# 0, K, 10 4, K, 11 9 2 354 379 4 2 330 341 5	163 6 5 285 292 5 5 Kr 11	166 6 8 124 129 10 0 366 357 5 0 72 14 125 10 7 126 357 5 0	7 0 462 459 4 67 Kr 11	303 4 3 300 308 5 1 269 267 6	194 5 6 233 226 6 -4, K, 12	114 9 2, K, 10 , 5 211 195 6 20 1014 1 751 754 4 -7. K, 12	9 ITTA I 294 290 5 6 188 195 8	237 5 7 143 132 9 -2, K, 12	147 7 3, K, 10 4 224 215 6 44 14 2 291 298 5 -1, K, 12	9 5 203 198 7 2 265 259 5	187 6 4, K, 10 5 214 215 7 63 121* 0 347 350 5 0, K, 12	
NANS ARE 10F0,10FC, 10SIG	I FC SIG K KFO FC SIG K KFO FC SIG K, B 8 7 58 182* 3 240 238 A	239 6 . 3, K, 9 5, K, 10		279 5 6 6 70 184 <b>x</b> 67 10	K, B 4, K, 9 2 181 176 B	336 4 1 167 166 6 77 17 10 343 5 4 45 64 24¥ 0 215 199 7	199 7 5, K, 9 -6, K, 11	K, 8 2 106 110 10 7 260 246 6	: 410 4 5 27 70 45¥ -5, K+11 223 7 6, K+9 8 236 227 7	K, B 0 117 126 9 -4, K, 11	238 6 3 71 68 16 <b>*</b> 6 326 310 5	N, 8 -3, N, 9 -3, N, 11 266 5 1 71 53 164 4 394 392 4	, 182 8 8, K, 9 7 286 288 6	N/ 8 2 7 46 190427 N/ 11 186 8 9, N/ 9 5 379 378 5	K+ 9 0 49 37 23# 8 223 221 7	46 45# -6, N 10 -1, N 11 N 1 K, 9 B 182 173 7 3 411 42B 4	63 18# -5, K, 10 6 339 335 5 K, e 2, 247 242 4 0, K, 11	63 175* 9 175 162 8 1 513 521 4	Ki         9         -4i         Ki         10         4         382         390         5           121         8         7         210         200         4         7         230         5	47 182* -3, k, 10 1, K, 11	139 7 8 200 194 7 5 327 316 5 139 7 8 200 194 7 5 327 316 5	86 13 -2, K, 10 2, K, 11	119 8 6 276 273 5 3 363 363 5	46 14 9 151 142 9 6 220 219 7	K, P1, K, 10 3, K, 11 125 7 4 344 349 4 1 410 417 5	116 8 7 217 219 7 4 260 274 6	45 34# 0, K, 10 4, K, 11 5 K, 9 2 354 379 4 2 330 341 5	163 6 5 285 292 5 5, K, 11	166 6 8 124 129 10 0 366 357 5 0 72 14 125 10 7 11 71 772 8	K, 9 17 0 462 459 4 6, K, 11	303 4 3 300 308 5 1 269 267 6	194 5 6 233 226 6 -4, K, 12	114 9 29 Ka 10 5 211 195 6 4 20 1014 1 25 211 195 6	Kr 9 1714 1 331 334 4 290 5 6 188 195 8	237 5 7 143 132 9 -2, K, 12	147 7 3, K, 10 4 224 215 6 64 14 2 261 298 5 -1, K, 12	K, 9 5 203 198 7 2 265 259 5	187 6 4, K, 10 5 2 214 215 7 63 171* 0 347 350 5 0, K, 12	
CLUMNS ARE 10FD+10FC+ 10SIG	NFO FC SIG N NFO FC SIG N NFO FC SIG 3, N, B 8 7 58 182# 3 240 238 A	256 239 6 . 3, K, 9 5, K, 10	4• K 8 0 125 131 8 1 270 273 6	278 279 5 6 6 70 184¥ 61 K3 10 278 279 5 6 6 70 184¥ 61 K3 10	5, K, B 4, K, 9 2 181 176 B	241 336 4 1 167 166 6 77 K 10 350 343 5 4 45 64 24x 0 215 199 7	202 199 7 5, K, 9 -6, K, 11	61 K1 B 2 106 110 10 7 260 246 6	408 410 4 5 27 70 45# -5, K, 11 226 223 7 6, K, 9 8 236 227 7	7, K, B 0 117 126 9 -4, K, 11	229 238 6 3 71 68 16 <b>*</b> 6 326 310 5	8, K, B	185 182 8 81 K1 9 7 286 288 6	9, N, B 2 7 46 190¥2, N, 11 189 186 8 9, K, 9 5 379 378 5	-8, K, 9 0 49 37 23* 8 223 221 7	27 46 45# -6/ 1/ 10 -1/ 1/ 11 -7/ 1/ 9 8 182 173 7 3 411 428 4	60 63 18# -5, K, 10 6 339 335 5 -4, K, 6 2 7 747 747 4 6, 544 1	6 63 175* 9 175 162 8 1 513 521 4	-5, K, 9 -4, K, 10 4 382 390 5 120 121 9 7 210 200 4 7 234 230 7	7 47 182* -3, K, 10 1, K, 11	-41 V1 Y 7 5 522 514 5 2 573 412 4 141 139 7 8 200 194 7 5 327 316 5	80 86 13 -2, K, 10 2, K, 11 2 7 7 7 7 7 7 7 7 7 7 7	-31 N1 Y 3 433 437 4 0 472 470 4 122 119 8 6 276 273 5 3 363 363 5	74 46 14 9 151 142 9 6 220 219 7	-2, K, 9 -1, K, 10 3, K, 11 127 125 7 4 344 349 4 1 410 417 5	126 118 8 7 217 219 7 4 260 274 6	34 45 34× 0, K, 10 4, K, 11 -1, K, 9 2 354 379 4 2 330 341 5	167 163 6 5 285 292 5 5, K, 11	165 166 6 8 124 129 10 0 366 357 5 0 20 72 14 15 10 7 212 776 9	0, K, 9 0 462 459 4 6, K, 11	308 303 4 3 300 308 5 1 269 267 6	192 194 5 6 233 226 6 -4, K, 12	109 114 9 2, K, 10 , 5 211 195 6 7 40 4014 1 751 754 A -72 K, 12	1 k 9 171 1 331 331 3 1 2 2 2 2 1 31 1 2 2 2 2 2	234 237 5 7 143 132 9 -2, K, 12	145 147 7 3, K, 10 4 224 215 6 76 64 14 2 291 298 5 -1.K, 12	2, K, 9 5 203 198 7 2 265 259 5	190 187 6 4, K, 10 5 214 215 7 6 63 171* 0 347 350 5 0, K, 12	
COLUMNS ARE 10FD,10FC, 10SIG	. KFO FC SIG K KFO FC SIG K KFO FC SIG 3, K, 8 8 7 58 182* 3 240 238 A	256 239 6 . 3, K, 9 5, K, 10	4, K, 8 0 125 131 8 1 270 273 6	243 337 4 3 111 123 7 4 143 137 7 278 279 5 6 6 70 184 <b>4</b> 6 <b>1</b> K1 10	5, K, 8 4, K, 9 2 181 176 8	241 236 4 1 167 166 6 77 17 10 350 343 5 4 45 64 24x 0 215 199 7	202 199 7 5, K, 9 -6, K, 11	61 K1 B 2 106 110 10 7 260 246 6	408 410 4 5 27 70 45# -5, K, 11 226 223 7 6, K, 9 8 236 227 7	7, K, B 0 117 126 9 -4, K, 11	229 238 6 3 71 68 16 <b>*</b> 6 326 310 5	Bi Ni B 7; Ni 9 -3; Ni 11 272 266 5 1 71 53 164 4 394 392 4	185 , 182 B B, K, 9 7 286 288 6	9, N, 8     2     7     46     190#    2/N, 11       189     186     8     9, K, 9     5     379     378     5	-8, K, 9 0 49 37 23* 8 223 221 7	27 46 45# -6, N, 10 -1, N, 11 -7, K, 9 B 182 173 7 3 411 428 4	60 63 18# -5, K, 10 6 339 335 5 -2, K, 6 2 247 242 2 6 8 4 1	6 63 175* 9 175 162 8 1 513 521 4	-5, K, 9 -4, K, 10 4 382 390 5 120 121 8 7 210 200 4 7 234 230 7	7 47 182x -3, K, 10 1, K, 11	-47 Nf Y 5 322 314 3 2 57 412 4 141 139 7 8 200 194 7 5 327 316 5	80 86 13 -2, K, 10 2, K, 11 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	-31 N1 Y 3 433 437 4 0 472 470 4 122 119 8 6 276 273 5 3 363 363 5	74 46 14 9 151 142 9 6 220 219 7	-2, K, 9 -1, K, 10 3, K, 11 127 125 7 4 344 349 4 1 410 417 5	126 118 8 7 217 219 7 4 280 274 6	34 45 34× 0, K, 10 4, K, 11 -1, K, 9 2 354 379 4 2 330 341 5	167 163 6 5 285 292 5 5, K, 11	165 166 6 8 124 129 10 0 366 357 5 0 00 72 14 15 15 10 7 316 357 5 0	0, K, 9 0 462 459 4 6, K, 11	308 303 4 3 300 308 5 1 269 267 6	192 194 5 6 233 226 6 -4, K, 12	109 114 9 2, K, 10 5 211 195 6 7 20 3014 1 751 754 4 -72 K, 12	1, K, 9 1, 1, 2, 2, 2, 2, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,	234 237 5 7 143 132 9 -2, K, 12	145 147 7 3, K, 10 4 224 215 6 76 64 14 2 201 298 5 -1, K, 12	2, K, 9 5 203 198 7 2 265 259 5	190 187 6 4, K, 10 5 2 214 215 7 6 63 171* 0 347 350 5 0, K, 12	

ATOM	x	Y	z	BISO
DS	0.67903(2)	0.44175(3)	0.21107(2)	3.35(1)
SB .	0.82775(3)	0.69625(4)	0.50892(3)	3.13(1)
C1	0.5342(6)	0.4872(8)	0.1543(7)	5.3(3)
Č2	0.7344(8)	0.2647(8)	0.1069(7)	6.8(5)
C3	0.5592(7)	0.3104( 9)	0.2312(8)	6.5(4)
C4	0.7885( 6)	0.5495(7)	0.1677(6)	4.3(3)
D1	0.4473(5)	0.5078( 7)-	0.1140( 6)	8.3(3)
02	0.7634(7)	0.1558(7)	0.0360( 6)	11.7(5)
03	0.4920( 6)	0.2333(9)	0.2420(8)	12.0(5)
D4	0.8507(5)	0.6056( 6)	0.1364(5)	7.2(3)
C11	0.7652( 5)	0.8940( 6)	0.6142(5)	3.9(2)
C12	0.6238( 6)	0.8461(8)	0.5595( 6)	5.0(3)
C13	0.5798( 6)	0.9708( 9)	0.6204(7)	6.2(4)
C14	0.6749( 7)	1.1420( 9)	0.7331( 8)	7.8(4)
C15	0.8158( 6)	1.1920( 7)	0,7904( 7)	5.6( 3)
C16	0.8601( 5)	1.0679( 7)	0,7306( 6)	4.3(2)
Ç21	0.8263( 5)	0.6416( 6)	0,6418( 5)	3.8(2)
C22	0.8276( 7)	0.7558( 7)	0.7808( 6)	5.9(4)
223	0.8282( 8)	0.7170( 9)	0.8642( 8)	8.0(5)
C24	0.8277( 8)	0.5698( 9)	0.8124( 8)	7.7( 4)
C25	0.8271( 8)	0.4556( 9)	0.6753( 8)	7,7(5)
26	0.8266( 7)	0.4895( 7)	0.5878( 7)	5.8(4)
231	1.0440( 5)	0.8531( 6)	0.6123( 5)	3.1(2)
232	1.1363( 5)	0.9006( 7)	0.7390( 6)	3.8(2)
C33	1.2762( 5)	1.0091( 8)	0.8101( 6)	4.4(3)
234	1.3246( 5)	1.0726( 7)	0.7557( 6)	4.3(3)
235	1.2343( 6)	1.0236( 8)	0.6285(7)	5.2(3)
C36	1.0945( 5)	0.9146( 7)	0,5565( 6)	4.5(3)
H12	0.551( 5)	-0.717( 6)	0.471( 5)	5.(0)
413	0.483( 6)	0.929( 7)	0.577( 6)	6.( 0)
41.4	0.638( 6)	1.225( 8)	0.771( 7)	8.( 0)
415	0.887( 6)	1.312( 7)	0,868( 6)	6.( 0)
416	0.963( 5)	1.108( 6)	0.773( 5)	5.( 0)
122	0.828( 5)	0.852( 7)	0.813( 6)	6.( 0)
423	0.834( 6)	0,798(8)	0,955( 7)	8.( 0)
124	0.826( 6)	0.547(8)	0.868( 7)	8.( 0)
425	0.838( 6)	0.357( 8)	0.640( 7)	8.( 0)
H26	0.828( 5)	0,406( 7)	0.488( 6)	6.( 0)
H32	1.100( 5)	0.851( 6)	0,769( 5)	4.( 0)
H33	1.344( 5)	1.041( 6)	0.897( 5)	5.( 0)
134	1.407( 5)	1.143( 6)	0.798( 5)	5.( 0)
H35	1,266( 5)	1.067( 6)	0,593( 5)	5.( 0)
174	4 A77/ E1	A 005/ /\	A 471/ EL	

Table S.9. Thermal Parameters for Os(CO)<sub>4</sub>SbPh<sub>3</sub>

Atom	U <sub>11</sub>	U 2 2	033	Uiz	Ū13	Ū23
Os	4.41(1)	4.59(1)	3.20(1)	1.10(1)	1.11(1)	2.78(1)
Sb	3.38(2)	4.35(2)	3.66(2)	1.65(1)	1.60(1)	2.77(2)
C1	6.2(3)	7.6(4)	6.4(4)	2.6(3)	3.1(3)	5.3(3)
C2	12.2(6)	7.2(4)	6.6(4)	4.9(4)	4.6(4)	5.4(4)
C3	5.9(4)	9.6(5)	9.2(5)	0.8(4)	1.5(4)	7.3(5)
C4	5.9(3)	5.4(3)	4.8(3)	1.9(3)	2.0(3)	3.2(3)
C4	5.9(3)	13.3(4)	11.0(4)	6.0(3)	4.1(3)	9.6(4)
02	22.4(7)	11.6(4)	10.7(4)	11.8(5)	10.0(5)	8.7(4)
03	9.1(4)	17.3	19.4(7)	0.6(4)	4.1(4)	15.8(6)
04	9.5(3)	8.8(3)	8.8(3)	2.2(3)	5.1(3)	6.5(3)
C11	4.8(3)	5.4(3)	4.6(3)	2.9(2)	2.8(2)	3.9(2)
C12	5.2(3)	7.2(4)	6.6(4)	3.3(3)	3.2(3)	4.9(3)
C13	5.7(3)	9.3(5)	8.3(4)	4.7(3)	4.2(3)	6.4(3)
C14	9.3(5)	9.7(5)	10.7(5)	7.3(4)	7.2(4)	8.3(5)
C15	7.7(4)	5.5(3)	7.9(4)	3.6(3)	4.3(3)	4.7(3)
C16	5.3(3)	5.3(3)	5.8(3)	2.8(3)	2.8(3)	3.9(3)
C21	5.1(3)	4.8(3)	4.2(3)	2.5(2)	2.5(3)	3.3(2)
C22	10.9(5)	5.5(3)	6.0(3)	4.6(3)	5.1(4)	4.3(3)
C23	14.1(7)	8.8(5)	7.4(4)	7.0(5)	7.3(0)	6.4(4)
C24	11.4(5)	9.5(5)	8.1(4)	5.7(4)	6.1(4)	7.5(4)
C25	12.5(6)	7.5(4)	9.1(5)	5.8(4)	5.5(4)	6.7(4)
C26	9.9(5)	6.0(3)	6.2(4)	5.0(4)	4.7(3)	4.4(3)
C31	4.0(2)	4.1(2)	3.8(2)	1.9(2)	1.9(2)	2.5(2)
C32	4.4(3)	5.5(3)	4.6(3)	1.4(2)	1.5(2)	3.4(3)
C33	4.7(3)	7.4(4)	4.7(3)	1.9(3)	1.1(2)	4.0(3)
C34	4.1(3)	6.7(4)	5.7(3)	1.5(3)	2.1(3)	3.6(3)
C35	6.0(3)	7.0(4)	7.0(4)	2.6(3)	3.7(3)	5.2(3)
C36	4.9(3)	7.0(4)	5.3(3)	2.5(3)	2.2(3)	4.6(3)

U<sub>ij</sub> values have been multiplyed be 100.

Table S.10. Bond Lengths and Angles for the Phenyl Groups of Os(CO)<sub>4</sub>SbPh<sub>3</sub>

a) Bond Lengths (Å).

C(11)-C(12)	1.392(7)
C(12)-C(13)	1.379(8)
C(13)-C(14)	1.367(10)
C(14)-C(15)	1.384(9)
C(15)-C(16)	1.375(8)
C(16)-C(11)	1.384(7)
C(21)-C(22)	1.371(7)
C(22)-C(23)	1.380(8)
C(23)-C(24)	1.343(9)
C(24)-C(25)	1.359(9)
C(25)-C(26)	1.385(8)
C(26)-C(21)	1.388(7)
C(31)-C(32)	1.379(7)
C(32)-C(33)	1.383(7)
C(33)-C(34)	1.378(8)
C(34)-C(35)	1.365(8)
C(35)-C(36)	1.388(7)

b) Bond Angles (°).

C(11)-C(12)-C(13)	120.1(5)
C(12)-C(13)-C(14)	120.0(5)
c(13)-c(14)-c(15)	120.6(5)
C(14)-C(15)-C(16)	119.5(5)
C(15)-C(16)-C(11)	120.6(5)
C(16)-C(11)-C(12)	119.1(5)
C(21)-C(22)-C(23)	119.4(5)
C(22)-C(23)-C(24)	121.4(6)
C(23)-C(24)-C(25)	119.9(5)
C(24)-C(25)-C(26)	120.6(6)
C(25)-C(26)-C(21)	119.1(5)
C(26)-C(21)-C(22)	119.6(5)
C(31)-C(32)-C(33)	120.5(5)
<b>C(32)-C(33)-</b> C(34)	120.3(5)
<b>C(33)-C(34)</b> -C(35)	119.6(5)
<b>C(34)-C(3</b> 5)-C(36)	120.5(5)
C(35)-C(36)-C(31)	120.4(5)
c(36)-c(31)-c(32)	118.7 4)

Table S.11. Structure Factors for Os(CO)<sub>4</sub>SbPh<sub>3</sub>

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	KF0	104 104	198	10,	156	124	72F	91	88	405	194	170	10,1	389	203	67	292 292	421	0 / 4 7 / 0	106	83	10,	125	281	167		298	10,	231	208 775	11,	135	11,	202	159	178	270	128	168	411 65	347	409		104 114 114	11,	354
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10516	KFO	8/5	218	420	9.	357	191	313	404	157	56 56	373	50	242	162	138	168	479	444	209	141	221	6	141		545	483	<b>6</b>	234	127	10,	370	10,		4 7 6	128	574	124		1490	240	406	516	100	386	496
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COLUM	KF0	48 477	273 273	156	293	297	0 U U	223	388	498	178	121	278	291	111	330	6	109	181	6	176	440	567	135	4 4	100	255	511	429	201 748		210	101	141	402	281	350	380	264	4 0 4 0 4	389	30E	171	101 101 101	178	533
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Molecule 1

ATOM	v	<b>v</b>	7	DICO
MIUM OC(11)	A 207/7/ 5)	1 057404 41	A 110107 AV	2 50/ 7)
05(11)	0.28363( 3/		0.11710( 47	2.37(3)
05(12)		1.18187( 0)		2.00(3)
05(13)	0.24004( 0)	1,40407( 8)	0.24823( 4/	3.20(3)
US(14)	0.20324(5)	1.27036(-6)		2.93(4)
P(1)	0.2/43(3)	0.7686(4)		3.12(22)
	0.1433(15)	0.7001(15)	0.03/2(13)	4./(12)
	0.2737(13)	0./320(18)		4.4(11) E 7/14)
	0.3/12(13)	0.67/3(16)	0.0888(13)	3.7(14) 7.9(-4)
C(15)	0.3437(13)	0.9747(15)	0.2149(11)	3.4(4)
C(14)	0.4244(13)	0.7387(137 0.9499(15)	0.1648(10)	3.5( 4)
C(17)	0.1412(12)	0.9574(14)	0.0796(10)	3.3(3)
C(18)	0.1233(13)	1.1504(15)	0.2239(10)	3.6(4)
C(19)	0.4172(14)	1.1057(14)	0.1804(12)	4.6(4)
C(110)	0.3111(13)	1.1919(15)	0.3048(11)	4.0(4)
C(111)	0.0978(14)	1.7757(14)	0.2498(12)	4.6(4)
C(112)	0.2915(15)	1.4777(17)	0.3570(12)	5.0(4)
C(113)	0.3951(15)	1.4207(18)	0.2224(12)	5.2(5)
C(114)	0.2219(14)	1.5347(18)	0.2395(13)	5.7(5)
C(115)	0.1927(14)	1.1509(14)	-0.0170(11)	3.1(4)
C(114)	0.1755(17)	1, 3764(19)	0.0385(14)	4.0(5)
6(117)	0.3513(16)	1.7844(19)	0.0636(13)	5.8(5)
C(118)	0.0571(14)	1.2478(16)	0.1087(12)	4.5(4)
0(14)	0.3865(10)	1.0141(11)	-0.0197(8)	5.3(3)
0(15)	0.2024(11)	0.9256(12)	0.2767(9)	6.3(3)
0(16)	0.5126(10)	0.9853(12)	0.1930( 8)	5.5(3)
0(17)	0.0560( 9)	0.9634(10)	0.0577( 8)	4.6(3)
0(18)	0.0351(11)	1.1262(12)	0.2355( 9)	6.2(3)
J(19)	0.3100(11)	1.2016(13)	0.1711( 9)	6.7( 4)
0(110)	0.3378(11)	1.1867(13)	0.3704( 9)	6.8( 4)
0(111)	0.0114(11)	1.3645(13)	0.2857( 9)	6.7( 4)
0(112)	0.3004(13)	1.4580(14)	0.4263(10)	8.0( 4)
0(113)	0.4845(12)	1.4352(13).	0.2115(10)	7.1( 4)
0(114)	0.2055(11)	1.6207(13)	0.2382( 9)	6.9(4)
G(115)	0.1722(11)	1.0781(12)	-0.0687( 9)	6.0(3)
0(116)	0.1635(14)	1.4396(16)	0.0071(11)	9.3(5)
0(117)	0.4385(13)	1.2901(15)	0.0442(11)	8.5(5)
0(118)	-0.0333(11)	1.2290(12)	0.1170( 9)	6.0(3)

\_ BIS0=8PI\*\*2(U11+U22+U33)/3

Molecule 2

ATQM	x	Y	Z	BISO
Ū\$(21)	0.77334( 5)	0.03464( 5)	0.37813( 4)	2.40( 3)
OS(22)	0.81459( 5)	-0.18932( 5)	0.30054( 4)	2.28(3)
GS(23)	0.80617( 5)	-0.41600( 6)	0.25594( 4)	2.79(3)
OS(24)	0.66744( 5)	-0.30249( 6)*	0.38046( 4)	2.65(3)
F(2)	0.7459( 4)	0.2145( 4)	0.4453( 3)	3.23(23)
C(21)	0.8585(16)	0.3065(16)	0.4408(14)	5.3(14)
C(22)	0.6284(16)	0.2564(16)	0.4069(13)	5.3(12)
C(23)	0.7250(16)	0.2469(14)	0.5530(12)	4.4(11)
C(24)	0.7665(13)	0.0554(15)	0.2738(10)	3.5( 4)
C(25)	0.7813(12)	0.0013(14)	0.4769(10)	3.1(3)
C(26)	0.9282(13)	0.0545(15)	0.3819(10)	3.ó(4)
C(27)	0.6212(12)	-0.0130(14)	0.3627(10)	3.3(3)
C(28)	0.7083(13)	-0.1934(14)	0.2187(10)	3.4(3)
C(29)	0.9227(12)	-0.1695(13)	0.3825(10)	2.9(3)
C(210)	0.9167(14)	-0.1682(16)	0.2320(11)	4.3(4)
C(211)	0.9182(14)	-0.3966(17)	0.3358(12)	4.6(4)
C(212)	0.9092(14)	-0.4482(16)	0.1789(12)	4.6(4)
C(213)	0.7013(14)	-0.4227(16)	0.1737(11)	4.2(4)
C(214)	0.7612(14)	-0.5534(16)	0.2602(11)	4.3(4)
C(215)	0.5786(13)	-0.1955(14)	0.4531(10)	3,4(3)
C(216).	0.7814(12)	-0.2872(14)	0.4589(10)	3.2(3)
C(217)	0.5625(13)	-0.3083(15)	0.2990(11)	3.9( 4)
C(218)	0.6005(14)	-0.4240(16)	0.3979(11)	'4.3( 4)
0(24)	0.7657(10)	0.0755(11)	0.2159( 8)	5.2(3)
ū(25)	0.7891( 9)	-0.0126(10)	0.5375(8)	4.7(3)
0(26)	1.0214( 9)	0.0687(10)	0.3826( 7)	4.4(3)
0(27)	0.5330( 9)	-0.0430(10)	0.3527( 7)	4.2(3)
0(28)	0.6469( 9)	-0.1910(11)	0,1716( 9)	4.7(3)
0(29)	0.9958( 9)	-0.1491(10)	0.4296( 8)	4.6(3)
6(210)	0.9814(11)	-0.1600(12)	0.1863( 9)	6.0(3)
ű(211)	0.9921(11)	-0.3910(13)	0.3781( 9)	6.5(4)
0(212)	0.9733(11)	-0.4719(13)	0.1283( 9)	6.8( 4)
0(213)	0.6366(10)	-0.4324(11)	0.1242( 8)	5.5(3)
0(214)	0.7368(11)	-0.6369(12)	0.2622( 9)	6.2(3)
0(215)	0.5510(10)	-0.1268(11)	0.4954(8)	4.9(3)
0(216)	0.8455(10)	-0.2780(11)	0.5101( 8)	5.2(3)
0(217)	0.4940(10)	-0.3062(11)	0.2566( 8)	5.3(3)
0(218)	0.5552(11)	-0.4990(13)	0.4082( 9)	6.7(4)

BIS0=8PI\*\*2(U11+U22+U33)/3

Table S.14. Thermal Parameters for Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub>, Molecule 1

U12 0.45( 3)

0.45(3) 0.13(3) -0.22(3) 0.21(3) 0.9(2) -0.7(10) 1.5(11) 2.5(11)

U13 -0.10(3) -0.06(3) 0.00(3) -0.08(3) 0.1(2) 0.8(10)

-0.1(10) -1.3(12)

U23

U23 1.04( 3) 0.95( 3) 0.59( 4) 1.40( 4) 1.1( 2) 2.7(12) -1.3(11) 4.5(14)

ATUM	011
OS(11)	3.40( 3)
OS(12)	3.67( 4)
OS(13)	5.38( 4)
OS(14)	4.02( 4)
P(1)	4.7(3)
C(11)	6.2(12)
C(12)	6.7(13)
C(13)	6.2(13)
C(14)	4.9( 5)
C(15)	4.6( 5)
C(16)	4.5( 4)
C(17)	4.2(4)
C(18)	4.5( 5)
C(19)	5.8( 5)
C(110)	5.1( 5)
C(111)	5.8( 5)
C(112)	6.3( 6)
C(113)	6.5( 6)
C(114)	7.2( 6)
C(115)	5.1( 5)
C(116)	7.7(7)
C(117)	7.3( 6)
C(118)	5.6( 5)
0(14)	6.8( 4)
0(15)	8.0( 4)
0(16)	6.9( 4)
0(17)	5.8( 3)
0(18)	7.8(4)
0(19)	8.4( 5)
D(110)	8.6( 5)
0(111)	8.5( 5)
0(112)	10.1( 5)
0(113)	9.0( 5)
0(114)	8.7( 5)
0(115)	7.5( 4)
0(116)	11.8( 6)
0(117)	10.7( 6)
0(118)	7.6( 4)

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U22

3.30( 4) 3.09( 4)

2.88(4) 3.86(5) 3.2(3) 3.8(13)

4.6(14) 5.2(14)

U33

3.14( 4) 2.94( 4) 3.90( 4) 3.24( 4) 4.0( 3)

8.0(15)

5.8(14) 10.3(18)

ATOM OS(21)	U11 3.80( 4)	U22 2,56(4)	U33 2.75(4)	U12 0.34(3)	U13 0.05(3)	U23 0.55(3)
OS(22)	3.28(3)	2.37(4)	3.01(4)	0.26(3)	0.11(3)	0,55(3)
05(23)	3.72( 4)	3.20( 4)	3.16( 4)	-0.09( 3)	0.01(3)	1.05(3)
OS(24)	4.52(4)	2.48(4)	3.58( 4)	0.47(3)	-0.25(3)	0.28(3)
P(2)	5.4( 3)	2.7(3)	4.2(3)	0.5(2)	0.3(2)	0.6(2)
C(21)	6.9(14)	4,4(14)	9.1(17)	-0.8(11)	1.0(12)	1.8(13)
C(22)	7.6(14)	4.7(14)	7.9(16)	0.5(11)	0.4(11)	3.5(13)
C(23)	9.4(15)	2.4(11)	5.4(13)	2.0(10)	0.6(11)	0.3(10) /
C(24)	4.5( 4)					
C(25)	3.9(4)					
C(26)	4.5(5)					
C(27)	4.2(4)					
C(28)	4.3(4)					
C(29)	3.6( 4)	•				
C(210)	5.5( 5)	8 				
C(211)	4.3(4)					
C(212)	4.1(4)					
C(213)	4.9( 5)					
C(214)	5.4( 5)					
C(215)	5.8( 5)					
C(216)	5,8( 5)					
C(217)	5.3( 5)					
C(218)	5.4( 5)					
0(24)	6.6(4)					
0(25)	5.9( 4)					
0(26)	5.6(3)					
0(27)	5.3(3)					
0(28)	5.9( 4)					
0(29)	5.8( 3)					
0(210)	7.7(4)					
0(211)	6.2(4)					
0(212)	6.6( 4)					
0(213)	6.7( 4)					
0(214)	8.5( 5)					
0(215)	8.2(4)					
0(216)	8.6( 5)					
0(217)	6.9( 4)					
0(218)	7.9( 4)					

## TEMP=-2(PI)\*\*2(U11\*H\*H\*ASTAR\*ASTAR+---+'2\*U12\*H\*K\*ASTAR\*BSTAR+---)

THE UIJ VALUES HAVE BEEN MULTIFLIED BY 100.

	Table	S.16.	Fractional	Coordinates	for	Hydroaen	Atoms	0	Ε
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Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub>

ATGM	x	Y	Z	в
H(111)	0.1433	0.6230	0.0367	5.
H(112)	0.1186	0.7221	0.1132	5.
H(113)	0.0976	0.7268	0.0238	5.
H(121)	0.2991	0.6551	-0.0810	5.
H(122)	0.2303	0.7563	-0.0738	5.
H(123)	0.3610	0.7693	-0.0660	5.
H(211)	0.8794	0,2964	0.3849	5.
H(212)	0.9228	0.3017	0.4710	5.
H(213)	0.8292	0.3769	0.4652	5.
H(221)	0.5631	0.2115	0.4110	5.
H(222)	0.6267	0.3306	0.4463	5.
H(223)	0.6353	0.2537	0.3528	5.
H(231)	0.6977	0.3189	0.5734	5.
H(232)	0.7951	0.2527	0.5805	5.
H(233)	0.6743	0.1892	0.5550	5.

Table S.17. Structure Factors for Os<sub>4</sub>(CO)<sub>15</sub>PMe<sub>3</sub>

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KF0 584	597	1692	18/1	650	652 652	700	440	-10,	728	440	122	834 878	2770	2106	865	366	200	767	202	-10,	797	716	698 225	4 10 14	200	481	471 408	439	680	878 7 A E	620	930	594	538	10	576	848	472	412	200 865	464	379	281	
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15(1)	0.30601( 5)	0.46157( 6)	0.31561( 5)
15(2)	0.24561( 6)	0.64841( 6)	0.23286( 5)
15(3)	0.20186( 5)	0.45139( 7)	0.12628( 5)
<b>15(4)</b>	0.28547( 6)	0.26800( 6)	0.23360( 6)
2(1)	0.2911( 4)	0.7534( 4)	0.3594( 4)
-(1)	0.217(2)	0.738( 2)	0.448( 2)
-(2)	0.416(2)	0.740( 2)	0.420( 1)
C(3)	0.285(2)	0.889(-2)	0.334( 2)
C(11)	0.437( 2)	0.475( 2)	0.282( 1)
C(12)	0.188( 1)	0.459(2)	0.368( 1)
C(13)	0.368( 2)	0.465( 2)	0.437( 1)
C(21)	0.380( 2)	0.661( 2)	0.202( 1)
C(22)	0.114( 2)	0.630( 2)	0.264(1)
C(23)	0.196( 2)	0.755(2)	0.150(2)
C(31)	0.083(2)	0.432( 2)	0.181(1)
C(32)	0,334(2)	0.480(2)	0.085(1)
C(33)	0.141( 2)	0.557(2)	0.050(1)
C(34)	0.167( 2)	0.342(2)	0.044(2)
C(41)	0+405( 2)	0.293(2)	0.1/6( 2)
C(42)	0.172( 2)	0.256(2)	0.297(1)
C(43)	0.362(2)	0.201(2)	0.328( 2)
C(44)	0.248( 2)	0.147( 2)	0.161(2)
0(11)	0.519( 1)	0.482(1)	0.2//(1)
0(12)	0.123(1)	0.458(1)	0.411(1)
0(13)	0.402(1)	0.463(2)	0.514(1)
0(21)	0.460(1)	0.676(1)	0.187(1)
0(22)	0.035(1)	0.629(1)	0.2/9(1)
0(23)	0.169( 1)	0.823(2)	0.102(1)
0(31)	0.009(1)	0.420(1)	0.207(1)
0(32)	0.403(1)	0.496(1)	0.001(1)
0(33)	0.093(1)	0.012(2)	-0.011(1)
0(34)	0.142(1)	0.278( 27	0.142(1)
0(41)	0.4/4(1)	0.300(1)	0.333(1)
0(42)	0.104(1)	0.243(1)	0.391(1)
0(43)	0.408(1)	0.133(1)	0.121(2)
0(44)	0.224(2)	0.070( 2) 0.707( 0)	0.493(0)
H(11)	0.229( 0)	0.793(0)	0.424(0)
H(12)	0.145( 0)	0./42(0)	0.479(0)
H(13)	0.228( 0)	0.8/4(0)	0.467(0)
H(21)	0.431( 0)	0.798(0)	0.379(0)
H(22)	0.464(0)	0./43(0)	0.454(0)
H(23)	0.424(0)	0.6/7(0)	0.387(0)
H(31)	0.304(0)	0.933(0)	A 29A( A)
H(32)	0.327( 0)	0.910( 0)	0.270(0)
4723	0.217(0)	0.413(0)	V+3V/\ V/

ATCH	U11	U22	U33	U12	. U13	U23
OS(1)	3.41(4)	3.34(5)	2.81(4)	-0.21( 3)	0.10( 3)	-0.06( 4)
05(2)	3.54(4)	3,78(5)	3.38(5)	0.44( 3)	0.93(3)	0.53( 4)
OS(3)	3.54(4)	5.43( 6)	2.53( 4)	-0.11( 4)	0.23( 3)	-0.27( 4)
OS(4)	4.06(5)	3.79( 5)	4.27(5)	-0.06( 4)	0.83(3)	-0.89('4)
P(1)	4.7(3)	4.1(3)	4.7(3)	-0.2( 3)	1.0(2)	-0.2(3)
C(1)	11.9(23)	8.8(21)	6.3(17)	-2.8(17)	4.8(16)	-3.1(16)
C(2)	6.4(14)	5.8(15)	4.3(12)	-0.9(11)	1.1(10)	-0.2(12)
C(3)	10.8(21)	5.2(17)	6.1(16)	- 0.1(14)	1.7(15)	-0.7(13)
C(11)	6.3(15)	3.6(13)	4.3(12)	0.2(11)	2.5(11)	1.3(10)
C(12)	4.2(11)	4.3(13)	3.5(11)	-0.3(10)	0.4(9)	1.1(10)
C(13)	6.7(14)	6.0(15)	2.8(12)	-2.8(12)	0.4(10)	0.9(11)
C.(21)	6.9(16)	4.6(14)	3.0(11)	-0.7(11)	3.0(10)	1.5(10)
C(22)	5.0(14)	3.7(12)	4.2(12)	1.5(10)	1.4(10)	1.1(10)
C(23)	4.7(13)	6.5(16)	6.2(15)	1.2(11)	1.8(11)	1.6(14)
C(31)	3.9(12)	5.4(15)	4.3(12)	0.2(10)	0.4(10)	-2.2(11)
C(32)	6.2(15)	6.3(16)	2.5(11)	1.2(12)	0.8(11)	-0.2(11)
C(33)	5.4(14)	9.7(21)	4,4(13)	2.4(14)	1.6(11)	3.7(15)
C(34)	5.5(15)	12.1(24)	4.2(13)	-1.1(15)	0.7(11)	-4.0(16)
C(41)	6.2(15)	4.3(14)	5.5(14)	1.5(12)	-0.7(12)	-0.2(12)
C(42)	5.3(14)	8.2(19)	3.0(12)	1.5(13)	-1.7(10)	-1.3(12)
C(43)	4.6(13)	6.8(17)	5.8(15)	0.4(12)	2.0(11)	-1.7(13)
C(44)	5.8(15)	5.9(17)	9.4(20)	-1.8(12)	2.7(14)	-3,2(16)
0(11)	5.1(10)	8.6(13)	6.9(11)	-0.9(9)	1.6(8)	-0.1( 9)
0(12)	5.9(10)	6.8(12)	6.5(10)	-0.3(8)	1.4(9)	-0.8( 9)
0(13)	8.6(12)	13.8(18)	3.1(9)	-3.2(11)	-0.4(8)	2.9(11)
0(21)	5.7(9)	6.7(11)	7.7(11)	-1.3(8)	4.4(9)	0.6(9)
0(22)	4.3(9)	6.9(12)	12.0(15)	1.2(8)	4.2(10)	-0.3(10)
0(23)	9.6(14)	9.7(15)	7.6(13)	2.6(12)	0.3(10)	4.4(12)
0(31)	4.6(9)	6.9(11)	5.3(9)	-0.9(8)	1.1(7)	-1.2(8)
0(32)	5.1(9)	8.5(12)	5.1(10)	0.0(8)	3.1(8)	0.6(8)
0(33)	7.0(11)	11.5(16)	6.4(11)	2.4(11)	-1.8(9)	3.8(11)
D(34)	9.0(13)	11.4(16)	6.6(12)	-2,6(12)	1.7(10)	-3.4(12)
0(41)	6.5(11)	8.5(13)	6.7(11)	0.6( 9)	3.7(9)	1.5(10)
0(42)	4.6( 9)	6.4(11)	8.7(12)	0.5(8)	2.1( 9)	0.0(10)
0(43)	7.8(12)	10.7(16)	7.8(12)	3.9(11)	2.6(10)	3.1(12)
0(44)	18,0(22)	7.6(14)	13.0(19)	-4.2(15)	3.4(16)	-8.1(15)

TEMP=-2(PI)\*\*2(U11\*H\*H\*ASTAR\*ASTAR+---+'2\*U12\*H\*K\*ASTAR\*BSTAR+---)

THE UIJ VALUES HAVE BEEN MULTIPLIED BY 100.

Table S.20. Structure Factors for  $Os_4(CO)_{1,4}$  FMe<sub>3</sub>

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10FD,10FC, 10SIG	SIG H KFO FC SIG H KFO FC SI -0 A54 513 71 -10 1327 1372 26	31 -8 920 976 39 -8 672 738 4	39 -7 714 672 46 -7 649 619 4	24 -6 408 221 /8 -5 1276 1281 2 34 -5 687 748 54 -4 964 1000 3	25 -4 438 465 82 -3 1392 1360 2	22 -3 588 643 59 -2 1443 1444 2 28 -2 1006 1091 37 -1 1093 1096 2	24 -1 403 593 78 0 1360 1339 2	41 0 954 964 36 1 592 599 4 7 7 7 814 867 39 7 620 645 4	42 4 698 706 48 7 525 530 5		66 -7 1413 1394 30 -9 1118 1113 3 28 -4 713 727 53 -7 936 915 3	42 -5 1220 1204 35 -5 420 351 6	37 -3 546 497 68 0 695 733 4	56 3 673 696 48 1 723 775 4	48 H 7, 11 2 1020 1104 31 2 1020 1104 31 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2		20 -4 1180 1163 39 5 1721 1796 1	32 -2 786 803 51 6 1347 1372 7 21 1 556 621 63 7 1627 1658 3	35 H 10, 11 H 4, 12	45 -3 788 824 57 -10 506 628 0 31 -2 1008 1948 20 -9 1233 1249 3		H 0, 12 -7 599 575 49	40 -10 810 833 32 -5 384 249 7	42 -4 1745 1707 20 -4 1546 1571 2	48 -2 1293 1242 24 -3 945 943	43 2 718 672 43 - 2 873 877 3 45 4 1376 1312 24 -1 685 781 4	48 6 1851 1920 23 1 434 477 6	54 H 1, 12 2 413 382 67	11 363 339 7 97 97 97 97 97 97 97 97 97 97 97 97	33 -8 728 702 39 H 5, 12	38 -7 754 736 38 -8 583 684 5 33 -5 2208 2137 20 -6 2323 2190 2	55 -4 700 690 40 -4 3586 3451 2	-3 2927 2948 17 -3 865 799 39	43 -2 929 887 30 -2 3667 3652 1	73 -1 2528 2562 181 804 826 4 26 2 2 252 2562 181 804 826 4	2 0 / / 0 / 0 / 0 / 0 0 0 0 0 0 0 0 0 0		21 4 907 923 35 6 971 988	26 5 1034 1053 31 H 64 12 50 / 1075 1205 20 20 401 521	42 7 123 1205 29 446 443 75 130 1215 31 -6 446 443 33 45 45 45 45 45 45 45 45 45 45 45 45 45	
S ARE 10FD,10FC, 10SIG	FC SIG H KFD FC SIG H KFD FC SI 	1067 31 -8 920 976 39 -8 672 738 4	631 39 -7 714 672 46 -7 649 619 4	1389 24 -6 408 221 /8 -5 1276 1281 2 757 34 -5 687 748 54 -4 964 1000 3	1243 25 -4 438 465 82 -3 1392 1360 2 	1509 22 -3 588 645 59 -2 1445 1537 2 1101 28 -2 1006 1091 37 -1 1093 1096 2	1433 24 -1 403 593 78 0 1360 1339 2	648 41 0 954 964 36 1 392 399 4 1157 727 2 814 842 39 2 620 645 4			504 66 -7 1413 1394 30 -7 1118 1113 3 1300 78 -6 713 727 53 -7 936 915 3		916 37 -3 546 497 68 0 695 733 4	456 56 3 673 696 48 1 723 775 4	598 48 H 7, 11 2 1025 1104 31 222 23 23 24 058 007 47 7 1570 1505 2		2256 20 -4 1180 1163 39 5 1721 1796 2	925 32 -2 786 803 51 6 134/ 1372 1 1875 21 1 556 621 63 7 1627 1658 3	842 35 H 10, 11 H 4, 12	746 45 -3 788 824 57 -10 506 628 0 1124 31 _2 1008 1948 20 -0 1233 1249 3		11 H Q+ 12 -7 599 575 49	851 40 -10 810 833 32 -5 1362 1308 34 44 70 -6 890 823 32 -5 384 249 7	668 42 -4 1745 1707 20 -4 1546 1571 2	662 48 -2 1293 1242 24 -3 945 943 3	858 43 2 /18 672 43 -2 873 877 5 690 45 4 1376 1312 24 -1 685 781 4	677 48 6 1851 1920 23 1 434 477 6	677 54 H 1, 12 2 413 382 67	554 52 -11 385 556 76 4 613 665 5 632 48 -10 1066 1079 31 6 997 972 5	1006 33 -8 728 702 39 H 5, 12	946 38 -7 754 736 38 -8 583 684 5 1947 33 -5 2208 2137 20 -6 2323 2190 2	699 55 -4 700 690 40 -4 3586 3451 2	11 -3 2927 2948 17 -3 865 799 39	695 43 -2 929 887 30 -2 3667 3652 1	277 73 -1 2528 2562 18 -1 804 826 4 26 2 27 70 77 7748 7	1440 20 0 /02 /10 3/ 0 2/12 2/13 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/ 2/	3128 20 2 377 219 72 2 1749 1757 2	2678 21 4 907 923 35 6 971 988	1674 26 5 1034 1053 31 H 6* 12 /0/ 5/ / 075 17/5 70 -0 /01 5/1	999 42 7 1131 1215 21 -6 446 443 1 199 42 7 1131 1215 31 -6 446 443 33 11 H 2, 12 -5 1309 1306 33	
DLUMNS ARE 10FD,10FC, 10SIG	KFO FC SIG H KFO FC SIG H KFO FC SI	064 1067 31 -8 920 976 39 -8 672 738 4	723 631 39 -7 714 672 46 -7 649 619 4	376 1389 24 -6 408 221 /8 -5 1296 1281 2 772 757 34 -5 687 748 54 -4 964 1000 3	234 1243 25 -4 438 465 82 -3 1392 1360 2 234 1243 25 -4 438 75 75 50 -3 1375 73 2	477 1509 22 -3 588 633 59 -2 1433 1337 2 040 1101 28 -2 1006 1091 37 -1 1093 1096 2	406 1433 24 -1 403 593 78 0 1360 1339 2	688 648 41 0 954 964 36 1 592 599 4 304 1557 7 7 814 872 39 7 2620 645 4	717 709 42 4 698 706 48 7 525 530 5		456 504 66 -7 1413 1394 30 -7 1118 1113 3 278 1300 28 -4 713 727 53 -7 936 915 3	Z/7 1300 ZB -5 720 1204 35 -5 420 351 6	812 916 37 -3 546 497 68 0 695 733 4	534 456 56 3 673 696 48 1 723 775 4	645 598 48 H 9, 11 2 1023 1104 31	8/3 1/0/ 23 -8 /32 /37 /35 55 4 1330 1330 2 008 918 30 -5 /37 /35 55 4 1330 1330 2	192 2256 20 -4 1180 1163 39 5 1721 1796 2	928 925 32 -2 786 803 51 6 1347 1372 1 851 1875 21 1 556 621 63 7 1627 1658 2	876 842 35 H 10, 11 H 4, 12	709 746 45 -3 788 824 57 -10 506 628 ( **7 **** 31 -2 ***********************************	701 712 45 -1 998 991 42 -8 972 985	H 5, 11 H 0, 12 -7 599 575 49	825 851 40 -10 810 835 35 -6 156 1508 414 444 70 -6 890 823 32 -5 384 249 7	703 668 42 -4 1745 1707 20 -4 1546 1571 2	631 662 48 -2 1293 1242 24 -3 945 943 3	818 858 43 2 /18 672 43 -2 873 877 5 726 690 45 4 1376 1312 24 -1 685 781 4	651 677 48 6 1851 1920 23 1 434 477 6	602 677 54 H 1, 12 2 413 382 67	565 554 52 -11 365 556 76 4 613 665 554 675 565 565 565 572 55	038 1006 33 -8 728 702 39 H 5, 12	919 946 38 -7 754 736 38 -8 583 684 5 201 1247 33 -5 2208 2137 20 -6 2323 2190 2	603 699 55 -4 700 690 40 -4 3586 3451 2	H 6, 11 -3 2927 2948 17 -3 865 799 39	711 695 43 -2 929 887 30 -2 3667 3652 1	396 277 73 -1 2528 2562 18 -1 804 826 4 coc 26 . 272 700 37 0 771 7748 7	377 1440 28 0 /82 /70 3/ 2 2/10 2/10 2/10 2/10 2/10 2/10 2/10 2	098 3128 20 2 377 219 72 2 1749 1757 2	535 2678 21 4 907 923 35 6 971 988	541 1674 26 5 1034 1053 31 H 67 12 220 201 520 7 2755 20 20 20 521 1	953 999 42 7 1131 1215 31 -6 446 443 7 H 7, 11 H 2, 12 -5 1309 1306 33	

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COLUMNS ARE 10FD, 10FC, 10SIG

COLUMNS AKE 10FD, 10FC, 10SIG

SIG	35	28	38	29		20	52	37	41	36	
5	942	1520	923	1536	15	429	661	957	950	1130	
KFO	991	1522	991	1593	Ŧ	467	641	972	907	1101	
X	7	•	-	2		မာ ၊	ų	7	•	1	
SIG	38	6E	F	61	27	47		41	43	4F	32
л Г	941	760	1132	576	1700	808	, 15	867	889	966	1130
KFO	885	662	1129	537	1705	839	H N	833	756	1012	1126
I	Ϋ́	4	7	•	-	N		ιΩ Ϊ	4	m 1	4
516		65	40		29	27	26	6E	60		32
5 C	14	545	1012	15	1195	1377	1395	866	765	15	982
KFO	H 6	553	677	о́ н	1228	1406	1434	924	644	H 1,	1071
I		2 1	-		<b>9</b> -	4	ч Г	•	~		<b>9</b> -

ATOM	X	Y Y	z
OS(1)	0.33486( 4)	0.10071( 5	0.05491(4)
05(2)	0.30382( 4)	-0.14812(5)	0.08784(4)
OS(3)	0.26492( 4)	-0.05045( 5)	-0.10381(4)
OS(4)	0.14666( 4)	0.13670( 5)	-0.08784(4)
P(1)	0.2452( 4)	-0.3469( 4)	0.0484( 4)
C(1)	0.320(2)	-0.452(2)	0.154( 2)
C(2)	0.136(2)	-0.363(2)	0.080(2)
C(3)	0.227(2)	-0.419(2)	-0.036(2)
C(11)	0.291( 1)	0.161(2)	0.137(1)
0(11)	0.269( 1)	0.206(1)	0.191(1)
C(12)	0.454( 1)	0.065(1)	0.144(1)
0(12)	0.526( 1)	0.046(1)	0.201(1)
C(13)	0.374( 1)	0.256(2)	0.031(1)
0(13)	0.403( 1)	0.346(1)	0.022(1)
C(21)	0.204( 1)	-0.081( 2)	0.115(1)
0(21)	0.150( 1)	-0.047(1)	0.141(1)
C(22)	0.376( 1)	-0.157(2)	0.216( 2)
0(22)	0.418( 1)	-0.160(2)	0.292(1)
C(23)	0.416(2)	-0.189(2)	0.075(1)
0(23)	0.488(1)	-0.217(1)	0.076(1)
C(32)	0.344(1)	-0.176(1)	-0.117(1)
0(32)	0.386( 1)	-0.249( 1)	-0.132(1)
C(31)	0.153(1)	-0.119( 2)	-0.190( 1)
0(31)	0.085( 1)	-0.160( 1)	-0.243(1)
C(33)	0.279( 1)	0.046( 2)	-0.194( 1)
0(33)	0.289( 1)	0.101( 1)	-0.248( 1)
C(41)	0.086( 1)	-0.001( 2)	-0.063( 1)
0(41)	0.042(1)	-0.082( 1)	-0.055( 1)
C(42)	0.116( 1)	0.252(2)	-0.018( 1)
0(42)	0.097( 1)	0.320( 1)	0.025( 1)
C(43)	0.042( 1)	0.151( 1)	-0.201( 1)
0(43)	-0.024( 1)	0.161( 1)	-0.264(1)
C(44)	0.211( 1)	0.254( 2)	-0.131( 1)
0(44)	0.246( 1)	0:324( 1)	-0.161( 1)
H(11)	0.305(0)	-0.529( 0)	0.121( 0)
H(12)	0.308( 0)	-0.459( 0)	0.208(0)
H(13)	0.388( 0)	-0.437(0)	0.175(0)
H(21)	0.093( 0)	-0.322( 0)	0.036( 0)
H(22)	0.151( 0)	-0.340( 0)	0.146( 0)
H(23)	0.127( 0)	-0.457( 0)	0.083( 0)
H(31)	0.282(0)	-0.437( 0)	-0.034( 0)
H(32)	0.188( 0)	-0.357( 0)	-0.087( 0)
H(33)	0.179( 0)	-0.486( 0)	-0.048( 0)

ATOM	U11	U22	<b>U33</b>	U12	U13	U23
OS(1)	3.33( 4)	3.97( 4)	3.18( 4)	-0.03( 3)	1.34( 3)	-0.24(3)
OS(2)	4.20( 4)	4.03( 4)	3.52( 4)	0.68( 3)	2.00( 3)	0.42(3)
OS(3)	3.81(4)	3.62(3)	2.99( 4)	0.39( 3)	1.54( 3)	-0.03(3)
OS(4)	3.45(4)	3.71( 4)	3.73(4)	0.41(3)	1.36( 3)	0.03(3)
F(1)	7.3(3)	3.5(2)	5.9( 3)	- 0.4(2)	3.6(3)	0.8(2)
C(1)	13.5(22)	6.9(14)	9.2(19)	3.4(14)	5.2(17)	3.6(13)
C(2)	9.5(18)	5.2(12)	15.0(26)	-0.6(11)	5.1(19)	-1.4(14)
C(3)	22.0(34)	5.4(13)	10.2(21)	-2.5(16)	8.4(23)	-1.8(14)
C(11)	4.6(11)	6.8(12)	5.9(13)	1.1( 9)	1.9(10)	1.1(11)
0(11)	9.1(11)	11.1(11)	5.6( 9)	0.3(9)	4.1( 9)	-3.1( 9)
C(12)	1.7( 9)	5.6(10)	4.7(11)	0.7(7)	-0.5(8)	1.4( 9)
0(12)	4.8(8)	8.3( 9)	7.8(10)	0.0( 7)	0.5( 8)	1.0( 8)
C(13)	4.1( 9)	5.3(10)	3.6( 9)	0.5(8)	1.8( 8)	-0.2(8)
0(13)	7.2(9)	5,1( 8)	8.1(11)	-2.2(7)	2.3(8)	0.0(7)
C(21)	3.2(9)	5.5(10)	4.9(11)	-0.9( 8)	0.8( 9)	-0,9( 9)
0(21)	6.6( 9)	8.3( 9)	5.8(9)	2.3(7)	3.1(8)	0,3(7)
C(22)	5.0(11)	6.9(12)	8.3(16)	0.0( 9)	5.6(12)	0.4(12)
0(22)	10.1(13)	17.0(17)	2.9(8)	-0.1(11)	1.7(8)	1.4(10)
C(23)	8.2(15)	6.5(12)	4.2(11)	0.2(10)	4.4(11)	-1.2(9)
0(23)	6.6( 9)	13.3(13)	7.2(10)	4.0(9)	4.3(8)	0.8( 9)
C(32)	4.4(9)	4.4(.9)	4.3(10)	0.3(8)	2.3( 9)	0.4(8)
0(32)	7.2(8)	5.6( 7)	7.2( 9)	2.5( 7)	4.4(8)	-0.2(7)
C(31)	4.9(11)	5.9(11)	4.7(11)	0.6(9)	1.6(10)	-0.9(10)
0(31)	6.8(10)	10.8(11)	6.5(10)	-2.2(8)	1.0( 8)	-4.3( 9)
C(33)	5.3(12)	5.4(11)	5.4(13)	1.2( 9)	1.6(10)	-0.4(10)
0(33)	10.7(12)	7.8(9)	6.2(10)	1.8( 8)	5.1( 9)	3,0( 8)
C(41)	5.2(11)	6.3(11)	2.8( 9)	2.3(9)	1.6( 9)	0.4(9)
0(41)	6.3(8)	5.6( 8)	7.9(10)	-0.5( 6)	4.6(8)	0.9(7)
C(42)	5.4(11)	3.8(10)	5.8(12)	0.5(8)	-0.1(10)	0.6( 9)
0(42)	9.5(11)	7.5( 9)	10.1(13)	-0.1( 8)	6.1(10)	-2.5( 9)
C(43)	2.7(9)	5.7(10)	4.8(11)	1.0(8)	-0.9( 9)	1.0( 9)
0(43)	5.8(10)	13.7(13)	7.4(11)	-1.3( 9)	-2.1( 9)	4.0(10)
C(44)	4.3(10)	5.7(10)	1.7(8)	1,1(8)	-0.5( 8)	-0.2(8)
0(44)	7.9(10)	7.0( 8)	5.5( 9)	-0.8(7)	.2.8( 8)	2.5(7)

TEMP=-2(PI)\*\*2(U11\*H\*H\*ASTAR\*ASTAR+---+'2\*U12\*H\*K\*ASTAR\*BSTAR+---)

THE UIJ VALUES HAVE BEEN MULTIPLIED BY 100.

Table S.23. Structure Factors for  $(\mu-H)_2OS_4(CO)_{13}PMe_3$ 

	SIG	40	5 M	42	5 Q	9 00 1 01		515	5 00	ЗЧ С	1ó	8	2 9 1 9	50	00	P N r N	37	38	Li C	)     	19	14	11	34	18	9 M	5 6 7 6	52	48	5	14	11		17	80	21	55	4 0 0 0	10 10	E i	10	15
	С,	0, L 844	1340	815 400	478 1388	1297	1, L	2717	1775	105	1260	1154 7720	5322 968	2028	858 1020	2090	772	883	2+ ·L	3718	722	697	3874	880	4494 1272	879	1722	2474	006	3, L 2018	2054	2395	838	1040	1295	2399	560B	732	1639	042	1.721	467
	KF0	11,	336	779	010	277	11,	651 51	147	140	384	171	46 197	959	796	967	B40	841	11,	584	736	738	1040	886	371 181	100	720	4124	794		949	325		24	. 98	383	200	878 1	81	116		173
	_	ı M		۰;		- 11 - 11	1	- i - i	- M V	) 4 • _	ີ ມ	-i⊷ •i	ν ν αο	6		i M M H	4		، •	- N	i M	41	סא נו א אי	~	∞ o	10	1:	4 Ci 4 T	5	÷ ۲	ій (М	<b>4</b>	י הי הי	- M 0 N	8	6 6	01		14	11 21	9 9	1
					_																																				•	
	, SIG	0 4 0 0	2 8 M		0 9 1 -	20	53		1 4	9 4 9 4		ដូដ	431	18	0- 1/ () ()	38	29	88	5	19	18	50	N 8	ទី	4 1	4 4	40	31	0E M	6 C H H	1 N 1 N	S E	м г 4 г	\$	32	23	20	4 14	2	55	2 G	4 Ci
	0	155	1021	3, 1	11634	548	767	1092	205	1184	4, 4,	863	316	1088	609 1 7 5 0	1107	1441	905	655	J122	1035	769	960 887	497	1243	771	1042	580	631	1291	427	612	764	1.08	621	926	749	1061	8, L	920	673 974	1195
0516	KFO	100	1070	112.	12/3	580	299	1090	470	1231	-12,	855	1×04 360	1079	613	1183	1429	980	607	1101	1077	203	575 915	482	1338	787	1057	570	621	1325	407	710	783	- 04	603	943	815	1065	-12,	626	1000	1326
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AKE 10F0,10FC, 10516	FC SIG L KFO FC SIG . 1 A 455 370 48	613 40 10 507 470 54	509 43 5, 7, L		809 6 2 2312 2318 14 10 267 15 7 107 107 70 12	270 13 3 110/ 10// 20 12 856 15 4 864 874 25	545 15 5 962 959 25 0	7/7 1/ 0 1207 1230 23 1 834 28 8 1276 1251 25 2	174 18 9 1222 1204 26 3	028 28 5, 8, L 5	700 38 1 1141 1201 18 0 776 25 2 1324 1315 18 7	519 46 3 1581 1548 17 8 3	7 L 4 1033 1043 1/ 11 . 818 & K 727 710 32	<u>932 11 7 1470 1514 22 0 3</u>	177 13 8 1064 1130 29 1 2	121 20 5, 9, L 2 2) 779 19 0 446 473 26 4 1(	553 17 2 506 542 39 5 10	376 23 3 1305 1307 20 6 20	597 23 5, 10, L 8 12	・ L 0 553 583 23 11 8	501 12 1 5, 11, L 33		752 29 1 384 302 31 3 752 29 6, 0, L 7 1	880 19 0 2160 2133 19 9	247 23 2 1664 1662 18 10 704 18 6 1385 1354 20		292 25 10 1165 1128 21 3 3 667 42 6, 1, L 4	, L 0 363 389 23 5	948 10 1 1031 1036 21 6 108 15 2 818 817 24 7	922 16 3 657 625 34		382 50 6 881 878 30 1 1 475 22 7 747 740 31 2 0	B99         32         B         1462         1471         20         4         16	141 28 9 1316 1285 20 5 1	432 62 10 493 538 42 6	7 L 11 BI5 BU5 29 7 677 13 12 791 B49 30 9	491 14 6, 2, L		91 1921 9191 7 16 610
JLUMNS AKE 10FU,10FC, 10516	KF0         FC         SIG         L         KF0         FC         SIG           5.         1.         1         4         45         770         48	37 613 40 10 507 470 54	180 509 43 5, 7, L	5, 2, L 3 1 531 503 29 5	765 4809 6 2 2312 2318 14 10	130 1270 13 3 110/ 10// 20 12 143 1856 15 4 864 874 25	561 2545 15 5 962 959 25 0	24/ 1/// 1/ 0 1207 1230 23 1 146 834 28 8 1276 1251 25 2	163 2174 18 9 1222 1204 26 3	146 1028 28 5, 8, L 5	15. 976 25 2 1324 1315 18 7	77 519 46 3 1581 1548 17 8 3 5. 7. 1		60 3932 11 7 1470 1514 22 0 3	11         3177         13         8         1064         1130         29         1	[70 1121 20 5; 9; L 2 2] (86 1779 19 0 446 473 26 4 10	41 2553 17 2 506 542 39 5 10	26 1376 23 3 1305 1307 20 6 20 26 2014 10 5 1303 1307 20 6 20	73 1597 23 5, 10, L 2 8 12	5,4,L 0 553 583 23 11 8 ,0 107 / 1 //E //E 72 7	77 2501 12 1 5, 11, L 0 0		73 752 29 I 384 302 JI 3	229 1880 19 0 2160 2133 19 9	./2 1247 23 2 1664 1662 19 10 06 2704 18 6 1385 1354 20	03 1155 27 8 1543 1517 18 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	00 1292 25 10 1165 1128 21 3 185 667 42 6, 1, L	5, 5, L 0 363 389 23 5	46 948 10 1 1031 1036 21 6 44 1108 15 2 818 817 24 7	31 1922 16 3 657 625 34	12 763 26 4 812 816 31 0 11 2 2 2 4 812 816 31 0 11	15 382 50 6 881 878 30 1 1 47 1475 22 7 747 740 31 2 0	07 899 32 8 1462 1471 20 4 16	07 1141 28 9 1316 1285 20 5 1	22 432 62 10 493 538 42 6	51 67 13 12 791 849 30 9	24 1491 14 6, 2, L 0. E4E 70 0 700 17 0		0/ 101 101 7 14 +TC /0
COLUMNS ARE 10F0+10FC+ 10SIG	L KFO FC SIG L KFO FC SIG 5. 1. 1 A 455 370 48	9 539 613 40 10 507 470 54	11 480 509 43 5, 7, L	5, 2, L 1 531 503 29 5	0 4765 4809 6 2 2312 2318 14 10	2 1943 1856 15 4 864 874 25	3 2661 2545 15 5 962 959 25 0	5 846 834 28 8 1276 1251 25 2	<u>6 2263 2174 18 9 1222 1204 26 3</u>	7 1046 1028 28 5, 8, L 5	0 1015 976 25 2 1324 1315 18 0	1 477 519 46 3 1581 1548 17 8 3 5. 7. 1	0 1743 1818 6 6 707 710 30	1 4160 3932 11 7 1470 1514 22 0 3	2 3211 3177 13 8 1064 1130 29 1	3 11/0 1121 20 55 97 L 2 23 5 1886 1779 19 0 446 473 226 4 10	6 2641 2553 17 2 506 542 39 5 10	7 1326 1376 23 3 1305 1307 20 6 20 6 2028 2010 40 5 1307 1307 20 6 20	2 1573 1597 23 5, 10, L 8 12	5, 4, L 0 553 583 23 11 8	1 2577 2501 12 5, 11, L 0	2 3216 3101 12 0 701 662 21 1 1	5 1047 773 252 29 1 384 302 JI 3 4 773 752 29 6, 0, L 7 1	5 1929 1880 19 0 2160 2133 19 9	6 11/2 1247 23 2 1664 1662 19 10 7 2706 2704 18 6 1385 1354 20	8 1103 1155 27 8 1543 1517 18 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	9 1300 1292 25 10 1165 1128 21 3 1 1 685 667 42 6, 1, L 4	5, 5, L 0 363 389 23 5	0 896 948 10 1 1031 1036 21 6 1 1146 1108 15 2 818 817 26 7	3 1931 1922 16 3 657 625 34	4 812 763 26 4 812 816 31 0 11	5 415 382 50 6 881 878 30 1 1 4 1447 1475 22 7 747 740 31 2 0	7 907 899 32 8 1462 1471 20 4 16	B 1107 1141 28 9 1316 1285 20 5 1	9 422 432 62 10 493 538 42 6	0 693 677 13 12 791 849 30 9	1 1524 1491 14 6, 2, L 5 500 515 70 5 70 705 17 0		81 1801 8191 7 18 810 /AC C

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NOTA	X	· Y	7.	BISO
JS(1)	0.72624( 6)	0.06807( 9)	0.16526( 7)	2.11(5)
JS(2)	0.81211( 7)	0.08846(10)	0.35340( 7)	2.91( 5)
JS(3)	0.84245( 7)	0.28599(10)	0.24489( 7)	2.76( 6)
JS(4)	0.66698( 7)	0.26834(10)	0.25142(8)	2.92(6)
>	0.7071( 4)	-0.1526( 6)	0.1181( 5)	2.8( 4)
2(1)	0.726( 2)	-0.272( 2)	0.197(2)	3.3(5)
2(2)	0.591( 2)	-0.187( 3)	0.032( 3)	6.1(8)
2(3)	0.783(2)	-0.207(3).	0.062(2)	3.6( 6)
2(11)	0.851( 2)	0.051(2)	0.169( 2)	2,8(5)
3(11)	0.921(1)	0.019(2)	0.162(1)	3.2(3)
2(12)	0.605( 2)	0.040( 2)	0.167( 2)	2.8( 5)
J(12)	0.534( 1)	-0.003( 2)	0.164(1)	4.2(4)
C(13)	0.676(1)	0.141(2)	0.052(2)	2.1( 4)
D(13)	0.642( 1)	0.193(2)	-0.019( 1)	4.6( 4)
C(21)	0.849( 2)	0.188( 3)	0.459(2)	3.9( 6)
D(21)	0.875( 1)	0.254(2)	0.522(2)	5.9( 5)
C(22)	0.697( 2)	0.049(3)	0.361( 3)	5.6( 8)
0(22)	0.637( 2)	-0.006(3)	0.374( 2)	7.9(7)
C(23)	0.857(2)	-0.083( 3)	0.400(2)	4.8(7)
0(23)	0.885(2)	-0.183( 3)	0.425(2)	8.4(7)
C(24)	9.936( 2)	0.104(3)	0.351(2)	4.3( 6)
0(24)	1.014( 1)	0.079(2)	0,365(2)	5.6( 5)
C(31)	0.958(2)	0.282(2)	0,232(2)	3.3(5)
0(31)	1.026( 1)	0.276( 2)	0.219( 1)	4.4( 4)
C(32)	0.792( 2)	0.398( 2)	0.150( 2)	3.3(5)
0(32)	0.757(1)	0.462(2)	0.086(2)	5.8( 5)
C(33)	.0.879(2)	0.425( 3)	0.328( 2)	4.2(6)
0(33)	0.905(2)	0.508( 2)	0.377(2)	6.0( 5)
C(41)	0.690(2)	0.411( 3)	0.332( 2)	4.1(6)
0(41)	0.707(1)	0.500(2)	0.369( 2)	5.1(5)
C(42)	0.556(2)	0.233(3)	0.268(2)	4.8(7)
0(42)	0.487(2)	0.207(2)	0.278(2)	7.2( 6)
C(43)	0.806(2)	0.3/0(-3)	0.148(2)	4.3(0)
0(43)	0.561(2)	0.441(2)	0.093( 2)	6.2(5)
R(11)	0.8/3(0)	-0.366(0)	0.155( 0)	5.(0)
H(12)	0.705(0)	-0.238( 0)	0.248(0)	4.(0)
M(I3)	0.798(0)	-0.302(0)	0.226(0)	4.(0)
R(21)	V.366( ())	-0.098( 0)	-0.008( 0)	7.(0)
R(22)	0.591( 0)	-0.269( 0)	-0.013( 0)	5.(0)
H(23)	0.542(0)	-0,209( 0)	0.062( 0)	7.(0)
H(31)	0.789(0)	-0.128( 0)	0.025( 0)	5.(0)
H(32)	0./45( 0)	-0.287(0)	0.020( 0)	5.(0)
H(33)	V.854( ())	-0.245( 0)	0.103( 0)	5.( 0)

arameters	for	OSA	(CO)	) <sub>13</sub> PMe <sub>4</sub>
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ATOM	U11 2.59( 5)	U22	U33 2.66( A)	U12 0.01(4)	U13	U23
05(1)	7.07(4)	4.58(7)	2.66( 7)	-0.40(5)	1.44(5)	-0.08(5)
08(2)	7 20/ 41	7.55( 4)	3.45(7)	~0.83(4)	1.94(5)	-0.67( 5)
	3,20( 0)	3,33(6)	A 10( 7)	-0.04(5)	2,12(5)	-0.82( 5)
03(4/	3,20( 6)	3.72( 4)	3.9(5)	0.1(3)	1.3(3)	-0.5(3)
r C(1)	4.2(7)	3.3( 4)		••••••		0.01 0/
	7.8(10)					
	4.6(7)			-		
C(11)	3.5( 6)					
0(11)	4.1(4)					
C(12)	3.6( 6)					
0(12)	5.3(5)					
C(13)	2.7(6)					
0(13)	5.8( 5)					
C(21)	5.0(8)					
0(21)	7.5( 6)		·			
C(22)	7.1(10)		1			
0(22)	10.1( 9)					
C(23)	6.1(9)				,	
0(23)	10.6( 9)					
C(24)	5.5(8)					
0(24)	7.1( 6)					
C(31)	4.2(7)					
0(31)	5.6( 5)					
C(32)	4.2(7)					
0(32)	7.3( 6)					
C(33)	5.4(8)					
0(33)	7.6( 7)					
C(41)	5.3(8)					
0(41)	6.5( 6)					
C(42)	6.1( 9)					
0(42)	9.1( 8)					
C(43)	5.5(8)					
0(43)	7.8( 7)			•		

TEMP=-2(PI)\*\*2(U11\*H\*H\*ASTAR\*ASTAR+---+'2\*U12\*H\*K\*ASTAR\*BSTAR+---)

THE UIJ VALUES HAVE BEEN MULTIPLIED BY 100.

Table S.26. Structure Factors for Os<sub>4</sub>(CO)<sub>13</sub>PMe<sub>4</sub>

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	KF0	668	9605	1158 606	1026	-11-	941	1158	2921	1582	1589	1156	1140	1910	1497	2551	1886	2236	4 C 7 F 7 F	1020	1421	111	761	1860	1323	1238	111	0601	505	010	326	060	0 C C C C	11,	720	599 107		428	341	306	646
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	- KFO FC SIG	1 842 751 66	-13, 2, L	1 1875 1861 41	446 477 53	1051 1085 27		0 657 739 54	1640 1028 41	1261 1137 47	1041 975 64	-13; 3; L	1131 1130 29	1 850 850 32		0 526 51/ 53	1 686 691 59	0 1854 1816 56 0 2114 1666 47	-13, 4, L	1129 1172 36	2 656 673 42 1949 2014 47	854 800 56	2146 2048 51	974 744 113	-13, 5, L	973 935 33	1463 1373 60 1084 984 96	1146 1036 113	1513 1221 108 -13- 4- 1	893 880 49	1162 1124 30	1228 1187 4/	-12, 0, 1.	1878 1993 35	3187 3301 31	3227 3415 32	10/7 2030 41 274 554 55	2097 2011 45	2763 2712 70	-12, 1, L 1925 2012 37	581 605 39
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	SIG L KFO FC SIG 44 13 433 444 75	35 14 842 751 66	43 -13, 2, L	55 1 1875 1861 41 2 1011 1077 28	39 3 446 477 53	32 5 1051 1085 27 74 4 477 574 55		42 9 657 739 54	45 10 1069 1028 41 31 11 1440 1597 47	26 12 1261 1137 47	28 13 1041 975 64	-39 -13, 3, L	70 I 734 728 37 38 2 1131 1130 29	55 3 850 850 32		44 7 609 617 58	54 8 686 691 59		38	47 1 1129 1172 36	34 2 656 673 42 2 47 5 1949 2014 47	73 6 854 800 56		55 11 974 744 113	61 -13, 5, L		79 4 1463 1373 60 5 1084 984 96	33 6 1146 1036 113	61 7 1513 1221 108 71 -13- 6- 1	1 893 880 49		29 3 818 82/ 4/ 34 4 1228 1187 84	26 -12, 0, L	41 2 1878 1993 35	40 4 3187 3301 31		34 64 16/7 2030 41 75 10 572 557 55		41 16 2763 2712 70	54 - 121 17 L 67 1 1925 2012 37	36 2 581 605 39
	FC SIG L KFO FC SIG 817 44 13 433 444 75	1217 35 14 842 751 66	1600 43 -13, 2, L	961 55 1 1875 1861 41 5. 1 2 1011 1077 28	620 39 3 446 477 53	-838 32 5 1051 1085 27 1087 34 4 477 574 55		LR41 42 9 657 739 54	556 45 10 1069 1028 41 878 31 11 1440 1587 43	1139 26 12 1261 1137 47	1030 28 13 1041 975 64	1632 39 -13, 3, L	386 /0 I /34 /28 37 1515 38 2 1131 1130 29	803 55 3 850 850 32		24 L 5 526 517 53 702 44 7 609 617 58	795 54 B 686 691 59		1569 38 +131 41 L	1996 47 1 1129 1172 36	1292 34 2 656 673 42 444 47 5 1949 2014 47	638 73 6 854 800 56		1, 1, 10 1127 660 68 370 55 11 974 744 113	387 61 +13, 5, L		1047 79 4 1468 1373 60 5, L 5 1084 984 96	1347 33 6 1146 1036 113	200 61 7 1513 1221 108 430 71 -13 6. 1	), l. 1 893 880 49	1056 26 2 1162 1124 30	[217 29 3 818 82/ 4/ 0800 34 4 1008 1187 84		720 41 2 1878 1993 35	843 40 4 3187 3301 31	1, L 6 3227 3415 32	104 34 61 10/7 2030 41 047 75 10 574 554 55		747 41 16 2763 2712 70	.3/7 34 -12/1/L 653 67 1 1925 2012 37	173 36 2 581 605 39
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1,0,2 2,0,0	kF <sub>0</sub> 656 766	F <sub>C</sub> 779 944	ωΔ² 88 135
With Extinc 1,0,2 2,0,0	tion 790 952	779 944	0.48 0.16

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