National Library of Canada
Bibliothèque nationale du Canada

Canadian Theses Service
Service des thèses canadiennes

Ottawa, Canada
K1A 0N4

NOTICE

The quality of this microform is heavily dependent upon the quality of the original thesis submitted for microfilming. Every effort has been made to ensure the highest quality of reproduction possible.

If pages are missing, contact the university which granted the degree.

Some pages may have indistinct print especially if the original pages were typed with a poor typewriter ribbon or if the university sent us an inferior photocopy.

Reproduction in full or in part of this microform is governed by the Canadian Copyright Act, R.S.C. 1970, c. C-30, and subsequent amendments.

AVIS

La qualité de cette microforme dépend grandement de la qualité de la thèse soumise au microfilmage. Nous avons tout fait pour assurer une qualité supérieure de reproduction.

S'il manque des pages, veuillez communiquer avec l'université qui a conféré le grade.

La qualité d'impression de certaines pages peut laisser à désirer, surtout si les pages originales ont été dactylographiées à l'aide d'un ruban usé ou si l'université nous a fait parvenir une photocopie de qualité inférieure.

La reproduction, même partielle, de cette microforme est soumise à la Loi canadienne sur le droit d'auteur, SRC 1970, c. C-30, et ses amendements subséquents.

NL-339 (r. 88/04) c

Canada

THE TETRANUCLEAR CARBONYLS OF OSMIUM AND SOME RELATED DERIVATIVES

by

Victor J. Johnston

B.S., Central Washington University, 1985

THESIS SUBMITTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

in the Department

of

Chemistry

© Victor J. Johnston 1989

SIMON FRASER UNIVERSITY

September 1989

All rights reserved. This work may not be reproduced in whole or in part, by photocopy or other means, without permission of the author.

National Library of Canada
Bibliothèque nationale du Canada

Canadian Theses Service
Service des thèses canadiennes

Ottawa, Canada
K1A 0N4

The author has granted an irrevocable non-exclusive licence allowing the National Library of Canada to reproduce, loan, distribute or sell copies of his/her thesis by any means and in any form or format, making this thesis available to interested persons.

The author retains ownership of the copyright in his/her thesis. Neither the thesis nor substantial extracts from it may be printed or otherwise reproduced without his/her permission.

L'auteur a accordé une licence irrévocable et non exclusive permettant à la Bibliothèque nationale du Canada de reproduire, prêter, distribuer ou vendre des copies de sa thèse de quelque manière et sous quelque forme que ce soit pour mettre des exemplaires de cette thèse à la disposition des personnes intéressées.

L'auteur conserve la propriété du droit d'auteur qui protège sa thèse. Ni la thèse ni des extraits substantiels de celle-ci ne doivent être imprimés ou autrement reproduits sans son autorisation.

ISBN 0-315-59277-X

Canada

APPROVAL

Name: Victor J. Johnston

Degree: Doctor of Philosophy

Title of thesis: The Tetranuclear Carbonyls of Osmium and Some Related Derivatives

Examining Committee:

Chairman: Dr. P. W. Percival

Dr. F. W. B. Einstein
Co-Senior Supervisor

Dr. R. K. Pomeroy
Co-Senior Supervisor

Dr. B. M. Pinto
Committee Member

Dr. R. H. Hill
Internal Examiner

Dr. R. D. Adams
External Examiner
Department of Chemistry
University of South Carolina

Date Approved: September 8, 1969

PARTIAL COPYRIGHT LICENSE

I hereby grant to Simon Fraser University the right to lend my thesis, project or extended essay (the title of which is shown below) to users of the Simon Fraser University Library, and to make partial or single copies only for such users or in response to a request from the library of any other university, or other educational institution, on its own behalf or for one of its users. I further agree that permission for multiple copying of this work for scholarly purposes may be granted by me or the Dean of Graduate Studies. It is understood that copying or publication of this work for financial gain shall not be allowed without my written permission.

Title of Thesis/Project/Extended Essay

The Tetranuclear Carbonyls of Osmium and Some Related Derivatives

Author: [signature]
(signature)

Victor J. Johnston
(name)

12 September 1989
(date)

ABSTRACT

The reaction of $Os(CO)_5$ with $Os_3(CO)_{10}(cyclooctene)_2$ under mild conditions afforded the first tetraosmium binary carbonyl $Os_4(CO)_{15}$. The structure of this cluster consists of an unusual planar, "kite-like" arrangement of osmium atoms. The related compounds $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$, $Os_4(CO)_{14}(CNBu^t)$, and $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ were also prepared and found by X-ray crystallography to have planar metal skeletons. Each of these new clusters except $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ has two short and two long peripheral metal-metal bonds as found previously for $Os_4(CO)_{14}(PMe_3)$. Models are proposed to account for the unusual variation in the Os-Os (or Os-Ir) bond lengths in these clusters.

Addition of CO to $Os_4(CO)_{15}$ gave $Os_4(CO)_{16}$ in excellent yield. This cluster adopts an unprecedented cyclobutane-like structure ($Os(CO)_4$ is isolobal with CH_2). When stirred in solution at room temperature $Os_4(CO)_{16}$ decomposes to $Os_3(CO)_{12}$. It is concluded that the inherent stability of deltahedral clusters may result from the occupation of molecular orbitals that are centered in the metal triangles.

The compounds $Os_4(CO)_{15}(L)$ (L = $CNBu^t$, $SbPh_3$, PF_3, and $P(OCH_2)_3CMe$) were synthesized, and with the exception of $Os_4(CO)_{15}(SbPh_3)$ their structures determined by X-ray crystallography. When L = PF_3 (or CO), the cluster adopts a puckered-square structure, whereas the other derivatives adopt

the spiked-triangular geometry previously found for $Os_4(CO)_{15}(PMe_3)$. The results indicate that the donor properties of L dictate the structural preferences of these clusters.

Thermolysis of $Os_4(CO)_{15}$ in hexane gave $Os_4(CO)_{14}$ in excellent yield. This cluster adopts a pseudo-tetrahedral structure. (Tetrahedral metal clusters with 14 carbonyl ligands had been predicted to be unstable.) The ^{13}C NMR spectrum of $Os_4(CO)_{14}$ in $CHFCl_2/CD_2Cl_2$ consists of a single sharp resonance even at -130 °C, while the infrared spectrum (in hexane) shows only three broad absorptions in the CO-stretching region. This behavior is reminiscent of that of $Fe_3(CO)_{12}$. The possibility that there is CO-exchange on the infrared time scale in these molecules is discussed.

The stereochemical nonrigidity of several of the compounds was investigated by variable temperature ^{13}C NMR spectroscopy. Various mechanisms for CO-exchange in the clusters are proposed.

The binary carbonyls $Os_4(CO)_n$ (n = 14, 15, 16) are the missing links between $Os_3(CO)_{12}$ and the higher nuclearity clusters of osmium. Their novel structures and properties should be of interest to synthetic and theoretical chemists alike.

ACKNOWLEDGEMENTS

During the past four years I have learned much from my senior supervisors, Professors F. W. B. Einstein and R. K. Pomeroy. I thank them for their help and for their continued friendship. I also thank the other excellent teachers that I have studied under at Simon Fraser University and Central Washington University.

I have benefited from interaction with the other members of Dr. Pomeroy's research group. They have made my time here enjoyable, and I thank each of them for their help. In addition, I have had profitable collaborations with Messrs. H. B. Davis and C.-Y Lu, and I am grateful for this opportunity. I am particularly thankful for the help of Mr. A. K. Ma who has collaborated on some of the work reported in this thesis.

Drs. R. J. Batchelor and R. H. Jones are thanked for their assistance and for the many hours of enlightening (or at least entertaining) conversation during the refinement of the crystal structures reported on the following pages.

Finally, I thank my wife and family for their support and patience, particularly during the writing of this thesis.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

"Metal cluster chemistry presents a substantial scientific challenge, regardless of its immediate technological relevance. It has a structural elegance. And as a field of research, it is fun."[1]

E. L. Muetterties

One aspect of the challenge of which the late Professor Muetterties wrote is the rational synthesis of transition-metal carbonyl cluster compounds.[1] Many cluster compounds have been prepared by the thermolysis of lower nuclearity carbonyl species.[2] However, this method does not allow for the control of the nuclearity of the products. The controlled synthesis of specific cluster compounds is still far from routine,[3,4] but several strategies have been developed over recent years. Some of these methods are described below.

One approach to the rational synthesis of cluster compounds is the reaction of metal complexes with unsaturated metal clusters such as $Os_3(\mu\text{-}H)_2(CO)_{10}$ or $Fe_4(CO)_{11}(PPh)_2$ (eqn 1.1).[5-8]

$$Fe_4(CO)_{11}(PPh)_2 + (\eta^5\text{-}C_5H_5)Rh(CO)_2 \longrightarrow (\eta^5\text{-}C_5H_5)RhFe_4(CO)_{12}(PPh)_2 + CO \quad (1.1)$$

Stone and co-workers have used the isolobal analogy between ethylene and some transition-metal multiple bonded dimers in the synthesis of cluster compounds. Thus, for example, two equivalents of $(\eta^5\text{-}C_5H_5)Rh(\mu\text{-}CO)_2Rh(\eta^5\text{-}C_5H_5)$ will displace both ethylene ligands in $Pt(C_2H_4)_2$ to form a pentanuclear cluster with a "bow-tie" structure (eqn 1.2).[9]

$$(\eta^5\text{-}C_3H_5)Rh(\mu\text{-}CO)_2Rh(\eta^5\text{-}C_5H_5) + Pt(C_2H_4)_2 \longrightarrow Pt[(\eta^5\text{-}C_5H_5)Rh(\mu\text{-}CO)_2Rh(\eta^5\text{-}C_5H_5)]_2 \quad (1.2)$$

Metal carbene and metal carbyne complexes will also react with metal species that coordinate ethylene (such as $Pt(C_2H_4)_2$).[10]

Hieber and Schubert reported the first example of a redox condensation reaction in cluster synthesis in 1965.[11] The reaction of $Fe(CO)_5$ with $[Fe_3(CO)_{11}]^{2-}$ in solution at room temperature gave good yields of $[Fe_4(CO)_{13}]^{2-}$ (eqn 1.3).

$$[Fe_3(CO)_{11}]^{2-} + Fe(CO)_5 \longrightarrow [Fe_4(CO)_{13}]^{2-} \quad (1.3)$$

Chini and his co-workers at the University of Milan used this type of reaction to prepare a wide variety of higher nuclearity clusters. For example, the stepwise build up of rhodium clusters

was achieved by the reaction shown in eqn 1.4.[4]

$$[Rh_5(CO)_{15}]^- + [Rh(CO)_4]^- \longrightarrow [Rh_6(CO)_{15}]^{2-} + 4CO \quad (1.4)$$

Clusters that contain bridging ligands such as sulfur have been used in the synthesis of higher nuclearity clusters. Adams and co-workers have reported that when solutions of $Os_3(\mu_3\text{-}S)(CO)_{10}$ and $Os(CO)_5$ are irradiated the tetranuclear cluster $Os_4(\mu_3\text{-}S)(CO)_{13}$ is formed (eqn 1.5).[12]

$$Os_3(\mu_3\text{-}S)(CO)_{10} + Os(CO)_5 \xrightarrow{h\nu} Os_4(\mu_3\text{-}S)(CO)_{13} \quad (1.5)$$

The hexanuclear cluster $Os_6(\mu_3\text{-}S)_2(CO)_{17}$ can be prepared by the reaction of $Os_3(\mu_3\text{-}S)_2(CO)_9$ with $Os_3(CO)_{10}(MeCN)_2$ as shown in eqn 1.6.[12]

$$Os_3(\mu_3\text{-}S)_2(CO)_9 + Os_3(CO)_{10}(MeCN)_2 \longrightarrow Os_6(\mu_3\text{-}S)_2(CO)_{17} \quad (1.6)$$

Another successful approach to cluster synthesis involves the reaction of mono- or multinuclear compounds with clusters that contain easily displaced ligands.[13-17]

Recent work from this laboratory has shown that certain neutral 18-electron complexes can act as ligands to 16-electron metal carbonyl fragments.[18] Thus heterobimetallic complexes with

dative metal-metal bonds can be prepared by the reaction of $Os(CO)_4(PMe_3)$ with $M(CO)_5(THF)$ (M = Cr, Mo, or W) (eqn 1.7).[19]

$$Os(CO)_4(PMe_3) + M(CO)_5(THF) \longrightarrow (Me_3P)(OC)_4OsM(CO)_5 \quad (1.7)$$

M = Cr, Mo, W

This work has recently been extended to the synthesis of a tetranuclear cluster compound. As shown in eqn 1.8, $Os(CO)_4(PMe_3)$ reacts with $Os_3(CO)_{11}(MeCN)$ to give the 64-electron cluster $Os_4(CO)_{15}(PMe_3)$.[20]

$$Os(CO)_4(PMe_3) + Os_3(CO)_{11}(MeCN) \longrightarrow Os_4(CO)_{15}(PMe_3) \quad (1.8)$$

Treatment of $Os_4(CO)_{15}(PMe_3)$ with Me_3NO gave the 62-electron cluster $Os_4(CO)_{14}(PMe_3)$.[20] This compound adopts a planar, "kite-like" structure with unusually short and long Os-Os bonds. In solution the compound undergoes CO-exchange that is rapid on the NMR time scale to about -90 °C. The observation of an unusual pairwise collapse of the signals in the variable temperature ^{13}C NMR spectra (CO region) of this compound led to the proposal of a novel "windshield-wiper" mechanism for the CO-exchange.[21] The research described in this thesis started out as an effort to prepare other 62-electron clusters (related to $Os_4(CO)_{14}(PMe_3)$) in order to better understand the novel properties of this cluster.

The utility of our method of cluster synthesis is clearly demonstrated by the preparation of one of the long sought after tetranuclear binary carbonyls of osmium, $Os_4(CO)_{15}$, as well as the heteronuclear cluster $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$.[22] These 62-electron clusters were prepared by the reaction of the 18-electron complexes $Os(CO)_5$[23] and $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$[24] with $Os_3(CO)_{10}(COE)_2$ (COE = cyclooctene)[25] (eqn 1.9 and 1.10).

$$Os(CO)_5 + Os_3(CO)_{10}(COE)_2 \longrightarrow Os_4(CO)_{15} \qquad (1.9)$$

$$(\eta^5\text{-}C_5Me_5)Ir(CO)_2 + Os_3(CO)_{10}(COE)_2 \longrightarrow (\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12} \qquad (1.10)$$

In addition to being a worthy target molecule in its own right, $Os_4(CO)_{15}$ has allowed the preparation of two more tetraosmium carbonyl clusters, $Os_4(CO)_{16}$[26] and $Os_4(CO)_{14}$,[27] which are reported in Chapter 2. The clusters $Os_4(CO)_n$ (n = 14, 15, 16) represent the first time a series of three binary carbonyls of the same metal have been prepared that differ in the number of CO groups but have a constant number of metal atoms.

In general, the synthesis of neutral binary carbonyls has not followed a smooth course.[28] The discovery of nickel tetracarbonyl in 1890 by Mond had an almost immediate commercial application in the production of pure nickel (the Mond process).[28] The following year, Mond and Berthelot independently

reported the synthesis and characterization of iron pentacarbonyl.[28]

Progress was slow from this point, however, and no new examples of this novel type of metal complex were to be prepared for almost fifteen years. In 1905 Dewar and Jones described the preparation of $Fe_2(CO)_9$, and in 1907 they reported the first carbonyl cluster compound, $Fe_3(CO)_{12}$ (as polymeric $Fe(CO)_4$).[29] This was followed in 1910 by Mond and co-workers who reported the characterization of $Co_2(CO)_8$ and $Co_4(CO)_{12}$.[30] Once again, these successes were followed by a drought, which did not end until Job's synthesis of $Cr(CO)_6$ and $W(CO)_6$ in 1927 and 1928, respectively.[31]

Another fifteen years were to pass before Hieber and Stallmann prepared the first carbonyls of osmium. They reported the synthesis of pentacarbonylosmium by the dry reaction of OsO_4 with CO.[32] A recent electron diffraction investigation revealed $Os(CO)_5$ to have the expected trigonal bipyramidal (D_{3h}) configuration.[33]

Hieber and Stallmann prepared another osmium carbonyl which they formulated as enneacarbonyldiosmium.[32] Eighteen years later in 1961 Corey and Dahl showed by X-ray crystallography that this product was actually $Os_3(CO)_{12}$.[34] This is but one example of the important role of crystallography in the study of metal carbonyls. The trinuclear carbonyl of osmium, $Os_3(CO)_{12}$, is the common carbonyl of this metal. It has an extensive chemistry

Table 1.1. The Neutral Binary Carbonyls of Osmium Known Before 1987.

Compound	Date[a]	Structure	Ref[b]
$Os(CO)_5$	1943	trigonal bipyramid (D_{3h})	32,33
$Os_2(CO)_9$	1970	single carbonyl bridge (C_{2v})[c]	37
$Os_3(CO)_{12}$	1943	triangular Os_3	32,34
$Os_5(CO)_{16}$[d]	1972	trigonal bipyramidal Os_5	39,42
$Os_5(CO)_{19}$	1981	"bow-tie" Os_5	40
$Os_6(CO)_{18}$[d]	1972	capped trigonal bipyramidal Os_6	39,43
$Os_6(CO)_{21}$	1982	planar, "raft-like" Os_6[c]	41
$Os_7(CO)_{21}$[d]	1972	capped octahedral Os_7	39,44
$Os_8(CO)_{23}$[d]	1972	bicapped octahedral Os_8	39,e

[a]First report. [b]First report, structure report. [c]Probable structure. [d]From pyrolysis of $Os_3(CO)_{12}$. [e]B. F. G. Johnson, personal communication to R. K. Pomeroy, cited in ref 45.

which has been recently reviewed by Deeming.[35]

Genuine enneacarbonyldiosmium was first prepared by Moss and Graham in 1970 by the low temperature photolysis of pentacarbonylosmium.[36] The structure of $Os_2(CO)_9$ has not been determined (no doubt due in part to the low thermal stability of this compound), but it probably adopts a structure with a single bridging carbonyl.[37]

A big breakthrough in osmium cluster chemistry was reported in 1972 by Eady, Johnson, and Lewis.[38] This involved the controlled pyrolysis of $Os_3(CO)_{12}$ to yield four new carbonyl clusters, $Os_5(CO)_{16}$, $Os_6(CO)_{18}$, $Os_7(CO)_{21}$, and $Os_8(CO)_{23}$, as well as two carbido carbonyl clusters, $Os_5C(CO)_{15}$ and $Os_8C(CO)_{21}$. In their

original communication the authors identified $Os_4(CO)_{13}$ as one of the products, but this was later found to be incorrect.[39] Lewis, Johnson, and co-workers also reported the carbonylation of $Os_6(CO)_{18}$ to give $Os_5(CO)_{19}$[40] and $Os_6(CO)_{21}$[41] (originally thought to be $Os_6(CO)_{20}$).

The stable, neutral carbonyls of osmium known before 1987 are listed in Table 1.1. The gap in the table at Os_4 has been filled by the preparation of the three tetraosmium binary carbonyls $Os_4(CO)_n$ (n = 14, 15, 16) reported in this thesis. Together they are the missing links between $Os_3(CO)_{12}$ and the higher nuclearity carbonyls of osmium. The preparations and some aspects of the chemistry of these clusters are discussed in Chapter 2.

Like $Os_4(CO)_{14}(PMe_3)$, the 62-electron cluster $Os_4(CO)_{15}$ also has an unusual, planar Os_4 skeleton. As noted above, the carbonyls in $Os_4(CO)_{14}(PMe_3)$ undergo rapid intramolecular exchange in solution to about -90 °C.[20] The variable temperature ^{13}C NMR spectra of $Os_4(CO)_{15}$ indicate that this cluster is still fluxional on the NMR time scale at -120 °C. Several other derivatives of this type have also been prepared and characterized. These include the aforementioned $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$, and $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, $Os_4(CO)_{14}(CNBu^t)$, and $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.[46] The fluxional behavior of one of these clusters, $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, has been investigated. Unlike $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$, however, variable temperature ^{13}C NMR spectra indicate that

$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ is rigid in solution at -45 °C. Possible mechanisms for CO-exchange that can account for the observed changes in the ^{13}C NMR spectra of these clusters are discussed in Chapters 2 and 3. The structural chemistry of these derivatives is also discussed in Chapter 3.

The 64-electron clusters $Os_4(CO)_{16}$ and $Os_4(CO)_{15}(PMe_3)$ are constitutionally closely related, but they have dramatically different structures. The binary carbonyl $Os_4(CO)_{16}$ adopts a cyclobutane-like geometry previously unknown for cluster compounds, while $Os_4(CO)_{15}(PMe_3)$ adopts the more common spiked-triangular structure.[20] The final chapter of this thesis reports the results of an investigation into the factors that influence the structural preference of some 64-electron $Os_4(CO)_{15}(L)$ (L = donor ligand) clusters. Four new derivatives of this type have been prepared, namely $Os_4(CO)_{15}[P(OCH_2)_3CMe]$, $Os_4(CO)_{15}(SbPh_3)$, $Os_4(CO)_{15}(CNBu^t)$, and $Os_4(CO)_{15}(PF_3)$. The related cluster $Os_4(CO)_{14}(CNBu^t)_2$ has also been prepared. In addition, the fluxional behavior of $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$ was investigated by variable temperature ^{13}C NMR spectroscopy, and possible mechanisms for the CO-exchange in these clusters are discussed in Chapter 4.[47]

CHAPTER 2

THE TETRAOSMIUM BINARY CARBONYLS

2.1 $Os_4(CO)_{15}$: The First Tetraosmium Binary Carbonyl

Preparation

Reaction of $Os(CO)_5$[23] with $Os_3(CO)_{10}(COE)_2$ (COE = cyclooctene)[25] in hexane at -15 °C without stirring gave $Os_4(CO)_{15}$ in ≃ 70% yield after several days (eqn. 2.1). The product was isolated directly from the reaction vessel as dark red, air-stable crystals that were analytically pure. They are soluble in dichloromethane but insoluble in hexane at room temperature.

$$Os_3(CO)_{10}(COE)_2 + Os(CO)_5 \xrightarrow[\text{hexane}]{-15\ ^\circ C} Os_4(CO)_{15} \qquad (2.1)$$

The compound is extremely difficult to recrystallize; all attempts resulted in the precipitation of $Os_4(CO)_{15}$ as a dark green powder. Concentrated solutions of $Os_4(CO)_{15}$ in, for example, dichloromethane are dark red while more dilute solutions are green. Similar dichroic behavior is exhibited by solutions of the closely related clusters $Os_4(CO)_{14}(L)(L')$ (L = CO, L' = PMe_3,[20] $CNBu^t$; L = PMe_3, L' = $P(OMe)_3$) (Chapter 3) and of the formally unsaturated cluster $Re_4(\mu\text{-}H)_4(CO)_{12}$.[48] It arises from a combination of the absorption characteristics (in the visible region) of the solution and the different ability of the

human eye to detect different colors.[49]

Structural Considerations

The structure of $Os_4(CO)_{15}$ at -73 °C was determined by X-ray crystallography. The crystal was disordered (see experimental section for a description of the disorder). A view of the molecule is shown in Figure 2.1. The structure consists of an almost planar arrangement of osmium atoms (the dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(4) is 179.0°). There are adjacent pairs of short (≃ 2.77 Å) and long (≃ 3.00 Å) bonds around the periphery of the cluster (see Table 2.1). The hinge bond, Os(1)-Os(3), has a length of 2.945 Å. (As a result of the restraints applied to these bonds during the structure refinement the estimated standard deviations, esd's, are without significance.) These bond lengths can be compared with the average Os-Os bond length of ≃ 2.88 Å in $Os_3(CO)_{12}$.[50]

The gross features of this 62-electron cluster are consistent with the Polyhedral Skeletal Electron Pair Theory (PSEPT), which predicts a butterfly arrangement of osmium atoms with five metal-metal bonds.[51] However, PSEPT cannot be used to rationalize the observed osmium-osmium bond lengths in $Os_4(CO)_{15}$.

A similar pattern of short and long bonds was found in $Os_4(CO)_{14}(PMe_3)$.[20] The bonding in this cluster was rationalized in terms of a pair of three-center, two-electron metal-metal bonds, and similar arguments can be invoked for $Os_4(CO)_{15}$. In

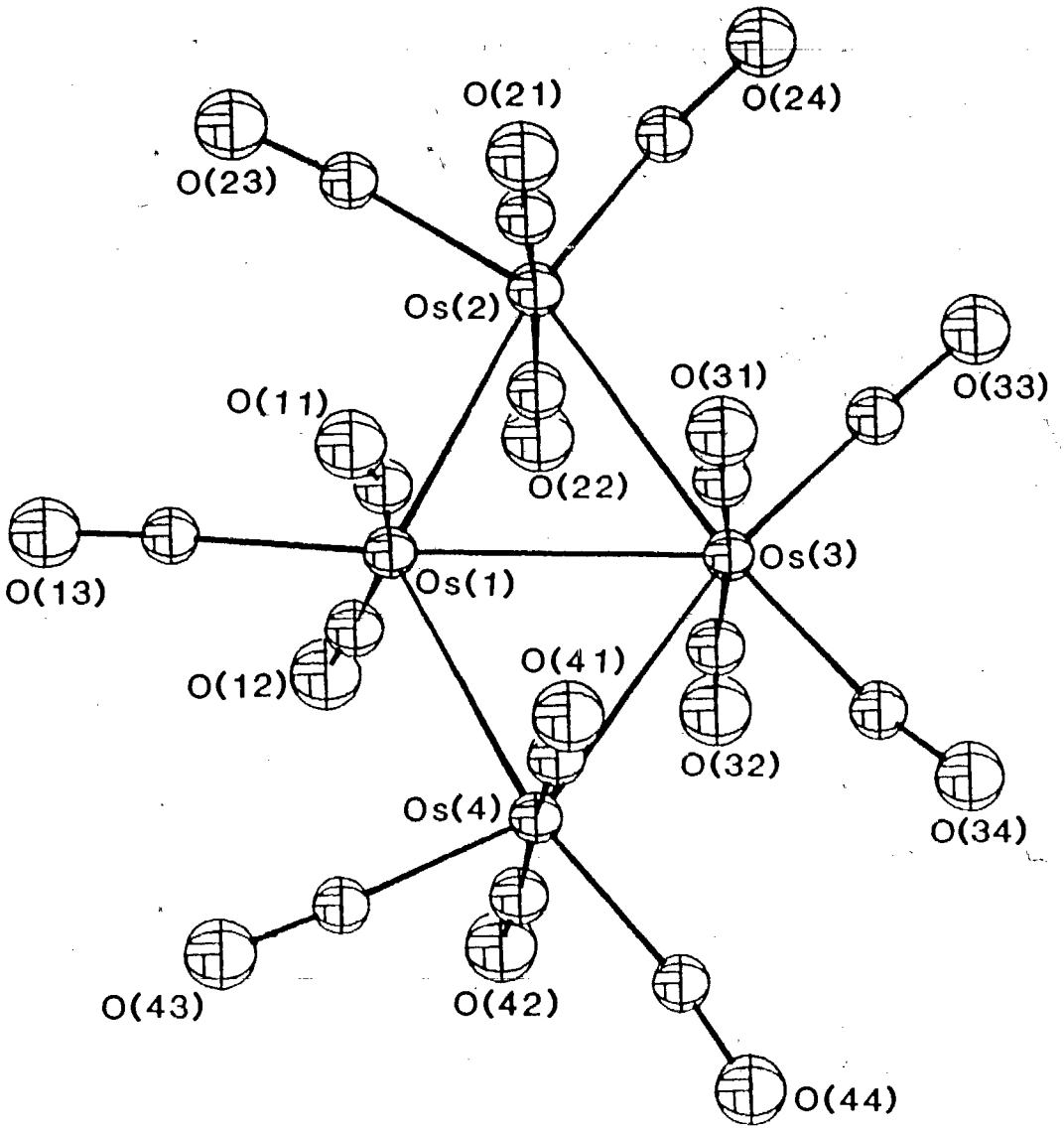


Figure 2.1. Molecular Structure of $Os_4(CO)_{15}$.

Table 2.1. Selected Molecular Dimensions of $Os_4(CO)_{15}$.

Bond Lengths (Å)

Os(1)-Os(2)	2.774 (1)[a]	Os(1)-Os(3)	2.948 (1)
Os(1)-Os(4)	2.775 (1)[a]	Os(2)-Os(3)	2.998 (1)[a]
Os(3)-Os(4)	2.998 (1)[a]	Os(1)-C(11)	1.96 (5)
Os(1)-C(12)	2.00 (5)	Os(1)-C(13)	1.90 (4)
Os(2)-C(21)	1.88 (5)	Os(2)-C(22)	2.08 (4)
Os(2)-C(23)	1.91 (5)	Os(2)-C(24)	1.91 (5)
Os(3)-C(31)	1.97 (6)	Os(3)-C(32)	1.88 (6)
Os(3)-C(33)	1.89 (6)	Os(3)-C(34)	1.95 (6)
Os(4)-C(41)	1.82 (4)	Os(4)-C(42)	1.94 (5)
Os(4)-C(43)	1.88 (4)	Os(4)-C(44)	1.94 (4)
C(11)-O(11)	1.15 (7)	C(12)-O(12)	1.07 (7)
C(13)-O(13)	1.11 (5)	C(21)-O(21)	1.18 (6)
C(22)-O(22)	1.00 (6)	C(23)-O(23)	1.13 (6)
C(24)-O(24)	1.20 (6)	C(31)-O(31)	1.13 (8)
C(32)-O(32)	1.19 (8)	C(33)-O(33)	1.18 (7)
C(34)-O(34)	1.14 (7)	C(41)-O(41)	1.30 (7)
C(42)-O(42)	1.16 (6)	C(43)-O(43)	1.16 (6)
C(44)-O(44)	1.16 (6)		

Selected Bond Angles (deg)

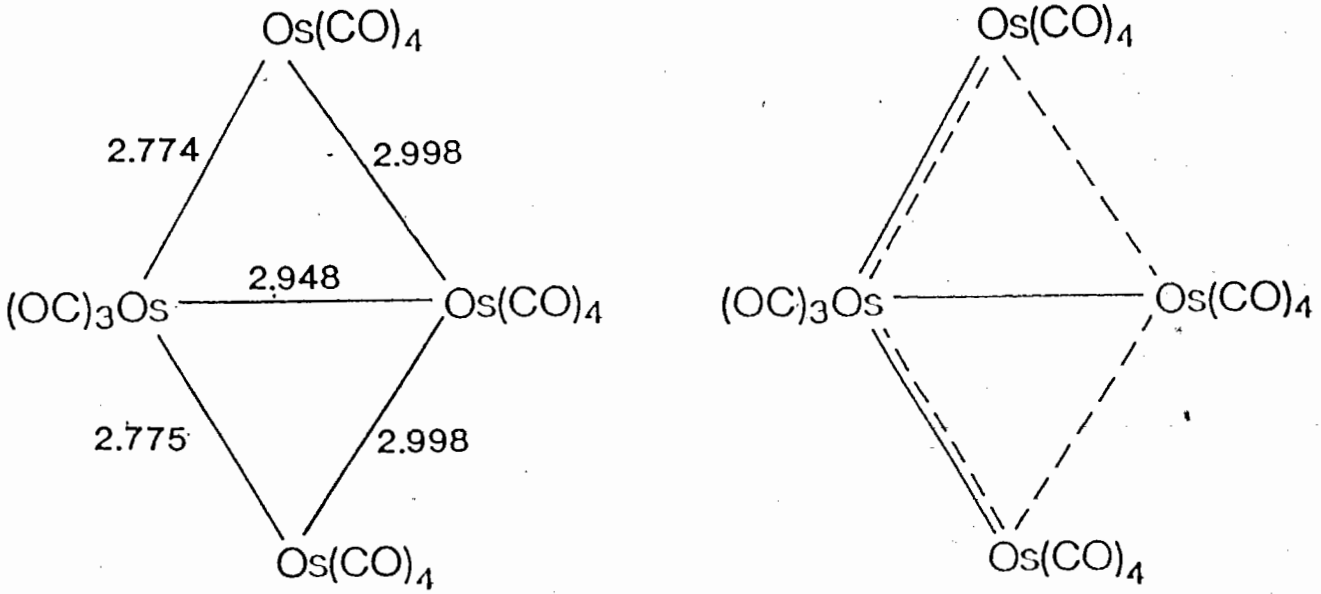
Os(2)-Os(1)-Os(3)	63.11 (3)	Os(3)-Os(1)-Os(4)	63.11 (3)
Os(1)-Os(2)-Os(3)	61.27 (4)	Os(1)-Os(3)-Os(2)	55.62 (3)
Os(1)-Os(3)-Os(4)	55.63 (3)	Os(1)-Os(4)-Os(3)	61.26 (4)
Os(2)-Os(1)-C(11)	93 (2)	Os(2)-Os(1)-C(13)	113 (1)
C(11)-Os(1)-C(12)	169 (2)	C(11)-Os(1)-C(13)	88 (2)
Os(3)-Os(2)-C(24)	109 (2)	C(21)-Os(2)-C(22)	174 (2)
C(21)-Os(2)-C(24)	94 (2)	Os(4)-Os(3)-C(31)	88 (2)
Os(4)-Os(3)-C(34)	77 (2)	C(31)-Os(3)-C(32)	172 (2)
C(31)-Os(3)-C(34)	90 (2)	C(33)-Os(3)-C(34)	95 (2)
Os(1)-Os(4)-C(41)	96 (2)	Os(1)-Os(4)-C(43)	90 (2)
C(41)-Os(4)-C(42)	176 (2)	C(41)-Os(4)-C(43)	92 (2)
C(43)-Os(4)-C(44)	104 (2)		

[a]Restraint was applied to this bond length.

this way the short bonds are assigned formal bond orders of 1.5, the long bonds orders of 0.5, and each osmium atom achieves an 18-electron configuration (Scheme 2.1). Further justification for this model comes from the bond length, bond order relationship developed by Pauling.[52] Use of the average Os-Os bond length in $Os_3(CO)_{12}$ (2.88 Å) as the length of an Os-Os single bond in Pauling's equation: $D(n) = D(1) - 0.60\log(n)$, where n is the bond order of a bond of length $D(n)$, leads to bond orders of 1.53 and 0.63 for the short and long bonds in $Os_4(CO)_{15}$, respectively.

The calculated bond orders are dependent on the value chosen for an Os-Os single bond. The average Os-Os bond length in $Os_3(CO)_{12}$ is typical of what are commonly considered single bonds in deltahedral osmium clusters, and so its use here is

Scheme 2.1



probably justified.[53] However, the empirical constant (0.60) in Paulings's equation was derived in conjunction with bulk metals and thus may not be appropriate for organometallic clusters.[54] Therefore, the calculated values should be interpreted with caution.

It is important to note that the model shown in Scheme 2.1 does account for the observed variation in the Os-Os bond lengths in $Os_4(CO)_{15}$. Significantly, it is also consistent with the structures of several derivatives of $Os_4(CO)_{15}$, namely $Os_4(CO)_{14}(PMe_3)$,[20] $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, $Os_4(CO)_{14}(CNBu^t)$, and $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ (the last three clusters are reported in Chapter 3). That so many clusters have this arrangement of metal atoms strongly suggests that the variation in bond lengths is an intrinsic property of the clusters and is not due to packing forces or to the trans influences of the ligands.

There are other examples of cluster compounds with unusually short or long metal-metal bonds in the literature. For example, in $Ru_4(CO)_{13}(\mu\text{-}PPh_2)_2$ and $Ru_4(CO)_{10}(\mu\text{-}PPh_2)_4$ there are long (2.91 - 3.18 Å) Ru-Ru bonds,[55] and in $Os_4(CO)_{12}(\mu\text{-}O_2CCF_3)_2$ there are short ($\simeq$ 2.78 Å) Os-Os bonds.[56] In $Os_4(CO)_{16}$ (Section 2.2) the Os-Os bond lengths are also long, and have an average value of $\simeq$ 2.99 Å. Also reported in this thesis is the hydrido cluster $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$, which is closely related to $Os_4(CO)_{15}$ and has one long Os-Os bond of length 3.050 (1) Å.[46] The application of the bonding concepts used in Scheme 2.1 to these clusters is not obvious, and therefore other possible explanations for the

observed bond lengths in $Os_4(CO)_{15}$ should be considered.

It may be possible to derive a qualitative molecular orbital picture of the bonding in $Os_4(CO)_{15}$ by replacing a hinge $Os(CO)_4$ group in the hypothetical cation $[Os_4(CO)_{16}]^{2+}$ with a T-shaped $Os(CO)_3$ fragment (T-shaped $Os(CO)_3$ is isolobal with C_{2v} $Os(CO)_4^{2+}$).[57] The bonding in $[Os_4(CO)_{16}]^{2+}$ has been investigated by Evans and Mingos using EHMO calculations, and their results are summarized in Figure 2.2.[58] The a_1 and b_2 frontier orbitals of the $Os(CO)_4^{2+}$ fragment interact in a stabilizing manner with all three bonding molecular orbitals of $Os_3(CO)_{12}$ without changing the number of Os-Os bonding interactions.[58] The $[Os_4(CO)_{16}]^{2+}$ cation is isoelectronic with $Os_4(CO)_{15}$ (both are 62-electron clusters), and from the isolobal analogy we can expect the bonding in $Os_4(CO)_{15}$ and $[Os_4(CO)_{16}]^{2+}$ to be qualitatively similar.[57]

The known cluster anion $[Re_4(CO)_{16}]^{2-}$ is also isoelectronic with $[Os_4(CO)_{16}]^{2+}$,[59] and can be used as experimental support for the conclusions reached by Evans and Mingos.[58] The structure of $[(n\text{-}C_4H_9)_4N]_2[Re_4(CO)_{16}]$ has been determined by Churchill and co-workers.[60] A view of the anion is shown in Figure 2.3. It consists of a planar arrangement of rhenium atoms, each coordinated by four terminal carbonyl ligands. As indicated in the figure, the Re-Re bond lengths around the periphery of the cluster are chemically equivalent, but they differ by $\simeq 0.04$ Å. Also evident from Figure 2.3 is that the four $Re(CO)_4$ groups are skewed with respect to the plane of the four rhenium atoms. The

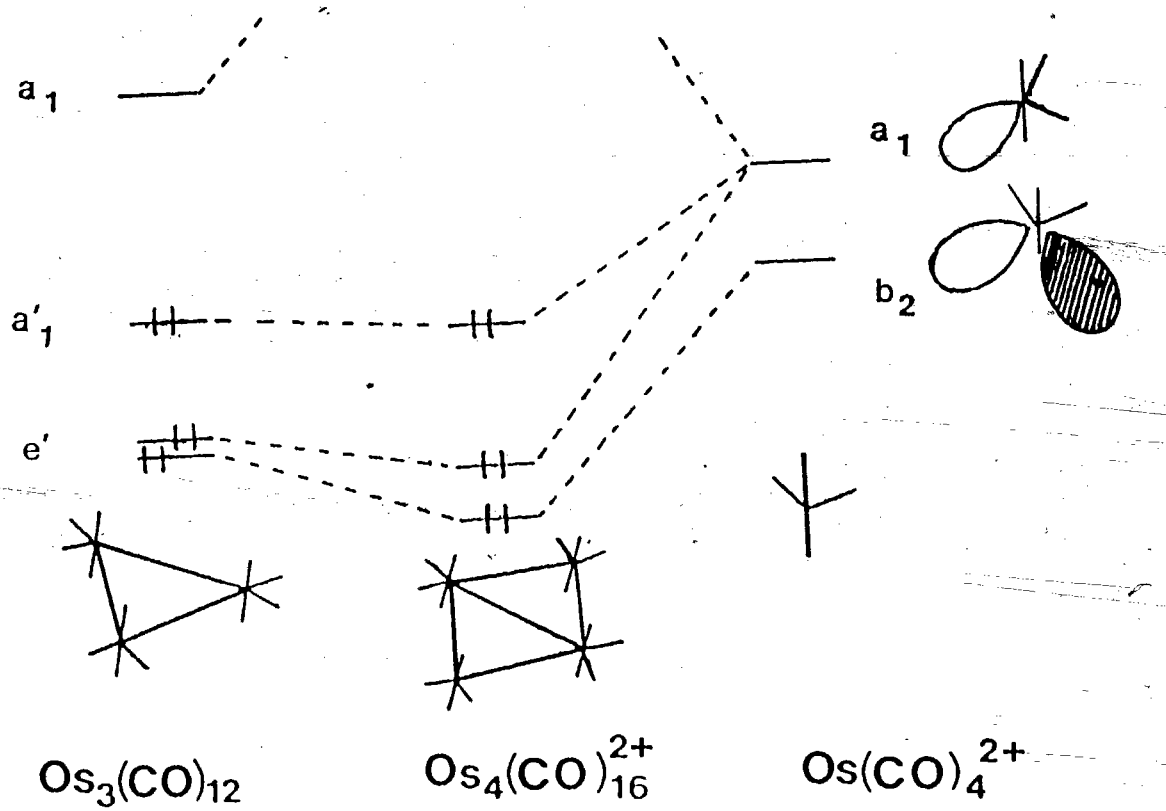
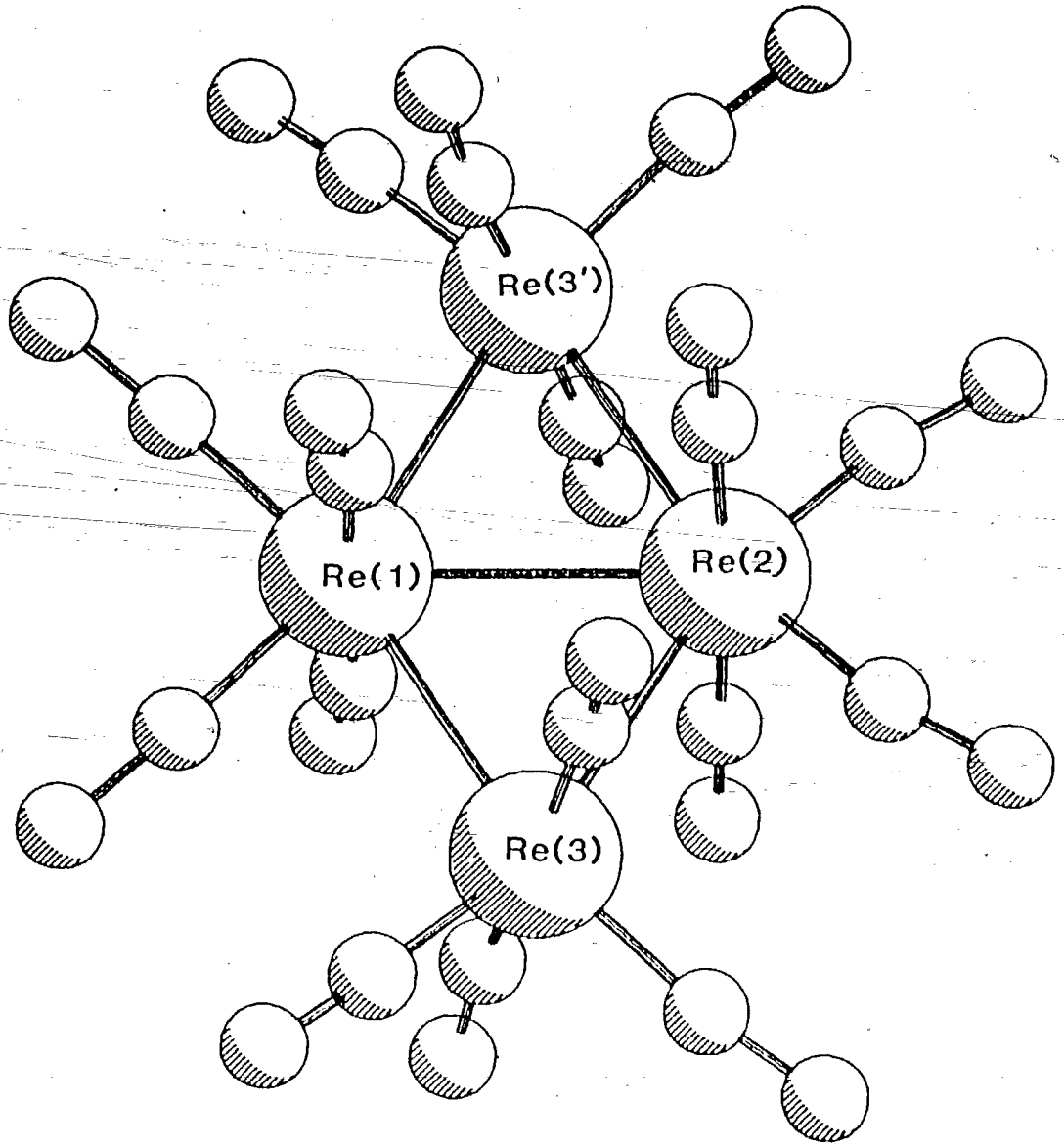


Figure 2.2. Bonding in $[Os_4(CO)_{16}]^{2-}$. (Adapted from ref 58.)

authors attribute the deformation of the structure of this anion from the idealized D_{2h} conformation to the minimization of the interactions between the carbonyl ligands.[60]

In $[Re_4(CO)_{16}]^{2-}$ the negative charge can formally be divided between the two wingtip $Re(CO)_4$ groups and thus allow each metal atom to achieve an 18-electron configuration.[60] For $[Os_4(CO)_{16}]^{2+}$, each osmium atom can achieve an 18-electron configuration if a formal charge of +1 is assigned to each of the hinge $Os(CO)_4$ groups. In a molecular orbital framework such formalisms are probably not accurate descriptions of these molecules, however, and the charge is expected to be delocalized over all the metal atoms.[60] In $Os_4(CO)_{15}$, on the other hand, the



Re(1)–Re(2) = 2.956 (7) Å
Re(1)–Re(3) = 2.982 (7) Å
Re(2)–Re(3) = 3.024 (7) Å

Figure 2.3. Structure of $[Re_4(CO)_{16}]^{2-}$.

imbalance between the isolated fragments does not result from ionic charge but rather from the number of ligands on the fragments. Thus although the T-shaped $Os(CO)_3$ and $Os(CO)_4^{2+}$ fragments are isolobal, they may interact with the three $Os(CO)_4$ groups in very different ways. It may be that the unusual Os-Os bond lengths found in $Os_4(CO)_{15}$ are a direct consequence of the nature of the T-shaped $Os(CO)_3$ fragment.

It has been argued that since the b_2 orbital of the $Os(CO)_3$ fragment is largely π^*_{CO} in character, the bonds in which it plays a major role will be long.[58] This is indeed the case in $Os_5(CO)_{19}$,[40] $Os_5(CO)_{18}(PMe_3)$,[61] and $Os_5(CO)_{16}[P(OMe)_3]$,[40] in which long bonds are found between the $Os(CO)_4$ (or $Os(CO)_3(PR_3)$) groups and the T-shaped $Os(CO)_3$ unit (these bond lengths range from 2.91 Å to 2.95 Å). In $Os_4(CO)_{15}$, however, the bonds involving this fragment are the shortest in the cluster with lengths of $\simeq$ 2.77 Å. The difference may result from the orientation of the $Os(CO)_3$ fragment. In the pentanuclear clusters the $2a_1$ orbital of the $Os(CO)_3$ group is pointed away from the the other osmium atoms, and is therefore unavailable for Os-Os bonding. In $Os_4(CO)_{15}$, on the other hand, both the $2a_1$ and $2b_2$ orbitals of the $Os(CO)_3$ group are well oriented for effective Os-Os bonding.

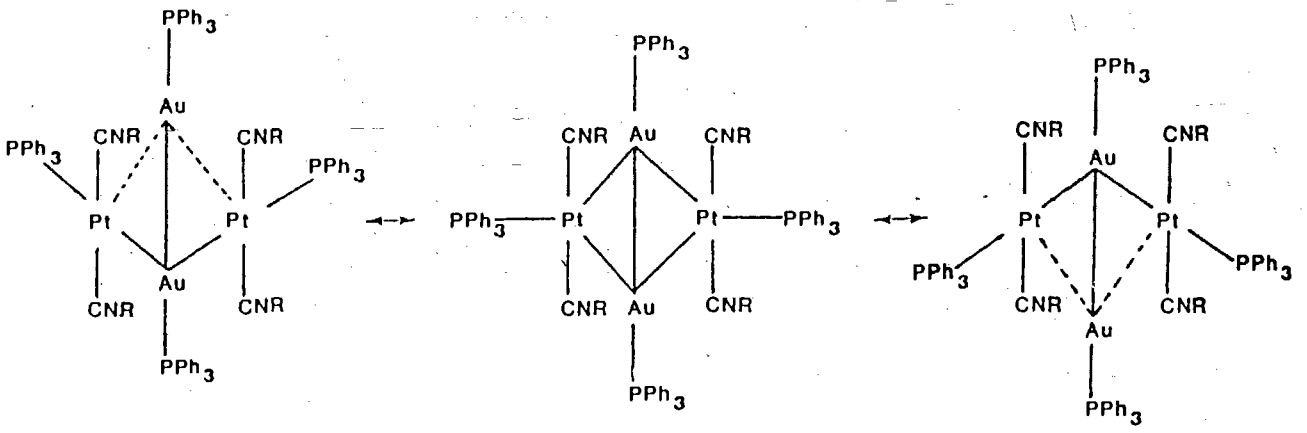
In a symmetric geometry (all Os-Os bond lengths $\simeq$ 2.88 Å) the repulsive interactions between the carbonyls along the Os(2)-Os(3)-Os(4) edge of $Os_4(CO)_{15}$ would be similar to those in D_{2h} $[Re_4(CO)_{16}]^{2-}$. As noted above, in the rhenium cluster these

interactions result in the skewing of the $Re(CO)_4$ groups.[60] In $Os_4(CO)_{15}$ this is not observed, rather the $Os_2(CO)_7$ hinge seems to have slid between the wingtip $Os(CO)_4$ groups. This results in a shortening of the Os(1)-Os(2) and Os(1)-Os(4) bonds and a lengthening of the Os(3)-Os(2) and Os(3)-Os(4) bonds. A similar situation was found by Mingos and co-workers for $[Pt_2Au_2(PPh_3)_4(CNC_6H_3Me_2)_4][PF_6]_2$.[62] This cation adopts a structure in the solid that is very similar to that of $Os_4(CO)_{15}$ (Scheme 2.2). The solution NMR data suggest that either the cation has a symmetric structure or it is fluxional. Regardless of which interpretation is correct, there must be significant changes in the Pt-Au bond lengths with only a small energy barrier. The authors report the results of EHMO calculations that are consistent with this conclusion.[62]

An example of steric influence on metal-metal bond length was reported by Shriver and co-workers.[63] They found the average Fe-Cr and Fe-Fe bond lengths in $[Fe_3Cr(CO)_{14}]^{2-}$ to be approximately 2.63 Å and 2.66 Å, respectively. These lengths may be compared to the average apical-basal and basal-basal Fe-Fe bond lengths in $[Fe_4(CO)_{13}]^{2-}$ of $\simeq$ 2.58 Å and $\simeq$ 2.50 Å, respectively.[64] The authors interpret the long Fe-Fe and Fe-Cr bonds in $[Fe_3Cr(CO)_{14}]^{2-}$ in terms of an isotropic expansion of the Fe_3Cr tetrahedron as a result of the steric congestion of the 14 carbonyl ligands.[63]

If the observed short and long peripheral bonds in $Os_4(CO)_{15}$ are due to steric factors, then the model shown in Scheme 2.3

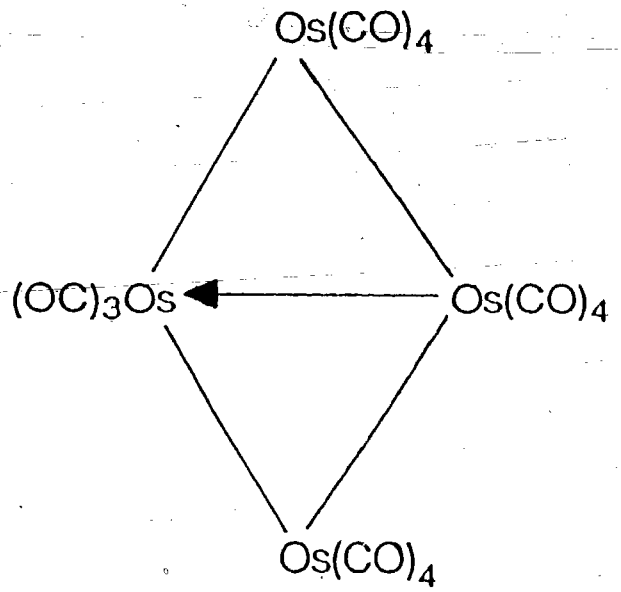
Scheme 2.2



would allow each osmium atom to achieve an 18-electron configuration. This model is not entirely satisfactory, however. The axial carbonyls along the Os(2)-Os(1)-Os(4) edge actually move closer together as the hinge slides between the wingtips. This strain is relieved somewhat by the tilting of the axial carbonyls on Os(1) (away from Os(3), see Figure 2.1), but the observed geometry must still be strained. This suggests that the structure of $Os_4(CO)_{15}$ must be governed at least in part by electronic factors. A similar argument has been used to account for the observation that the axial carbonyl ligands in $Os_3(CO)_{12}$ are eclipsed.[65]

Both the three-center, two-electron bond model (Scheme 2.1) and the model shown in Scheme 2.3 might be expected to leave Os(1) slightly electron rich and Os(3) slightly electron

Scheme 2.3



deficient. There is some spectroscopic evidence that this may be the case. In solution, the carbonyls of $Os_4(CO)_{15}$ are fluxional on the NMR time scale down to -118 °C (see below), but low temperature ^{13}C NMR spectra consistent with the solid state structures of $Os_4(CO)_{14}(PMe_3)$[20] and $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (Chapter 3) have been obtained. The resonances due to the carbonyls on Os(1) in these compounds come at an unusually low field, while those due to the axial carbonyls on Os(3) come at an unusually high field. The predicted polarization of the Os(1)-Os(3) bonds in these clusters, $Os(1)^{\delta-}$-$Os(3)^{\delta+}$, might be expected to cause a shielding of the carbonyls on Os(1) and a deshielding of the carbonyls on Os(3). It is known, however, that the change in chemical shifts of the ^{13}C NMR signals of metal carbonyls are opposite to those expected from simple

arguments based on electronic shielding.[66]

In closing, it is noted that neither of the simple models discussed above gives a completly satisfactory account of the observed structure of $Os_4(CO)_{15}$. It is apparent that molecular orbital calculations are required to more fully understand the unusual bonding in $Os_4(CO)_{15}$ and its derivatives.

Nonrigidity in $Os_4(CO)_{15}$[67]

The variable temperature ^{13}C NMR spectra of $Os_4(CO)_{15}$ in $CHFCl_2/CD_2Cl_2$ are shown in Figure 2.4. The spectrum obtained at -55 °C consists of three signals of approximate relative intensity 4:4:7. The pattern is invariant down to -118 °C (the lowest temperature investigated). Given the range in the chemical shifts found for the carbonyls of some derivatives of $Os_4(CO)_{15}$ (Chapter 3), the assignment of these signals is problematic. However, the two low field resonances (at δ 190.6 and 188.9) can be assigned to the axial carbonyls and the higher field resonance (at δ 171.1) to equatorial carbonyl ligands by comparison with the ^{13}C NMR spectra of $Os_3(CO)_{11}(L)$ clusters in the absence of CO-exchange.[68-70] Given this assignment, the pattern is consistent with a rapid, in plane merry-go-round process involving terminal-bridge carbonyl exchange as shown in Scheme 2.4. In this way all seven equatorial carbonyl ligands become equivalent, and further, the axial carbonyl ligands on Os(1) and Os(3) also become equivalent. The broadening of the resonances at δ 188.9 and 171.7 in the spectrum at -118 °C

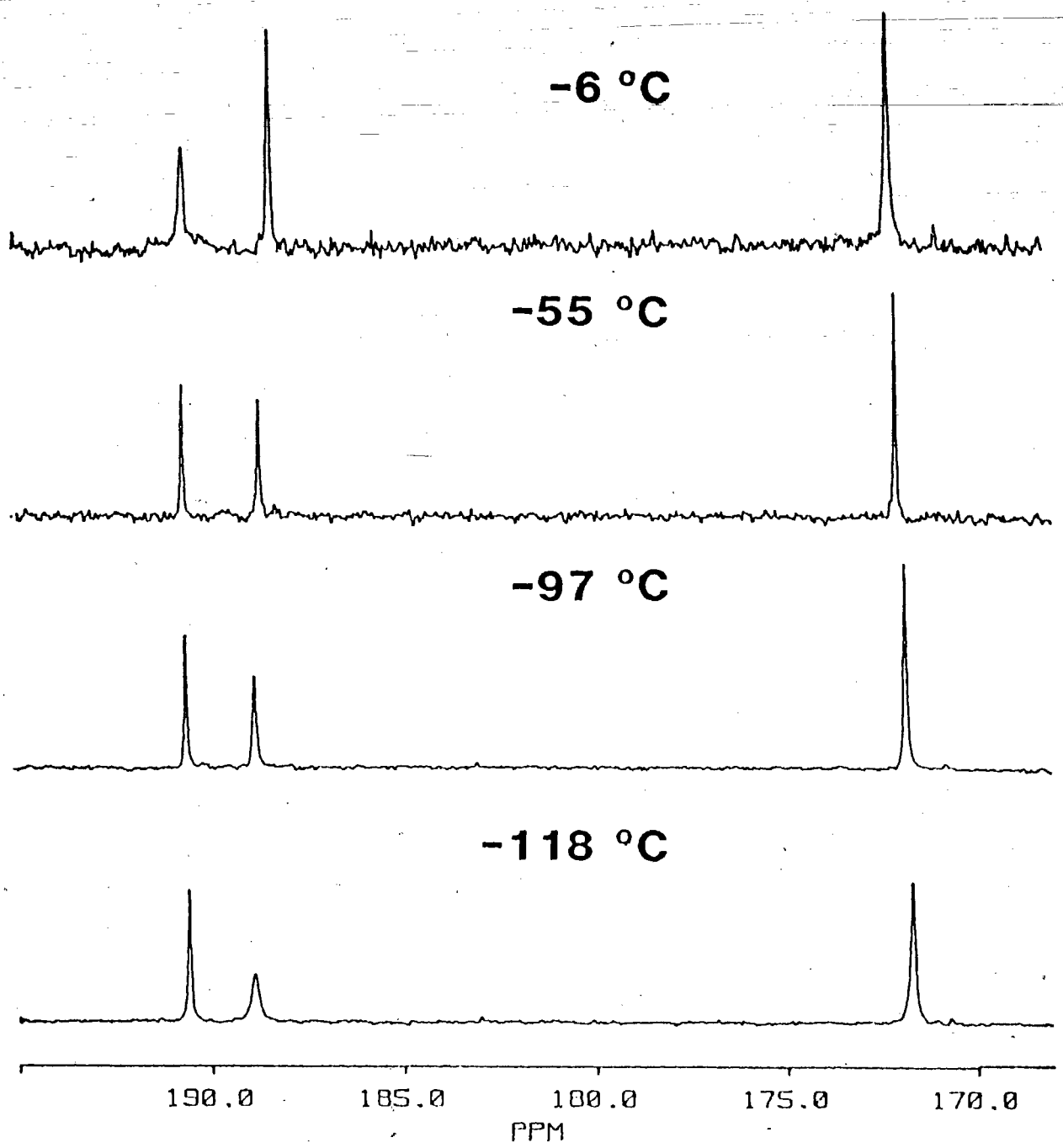
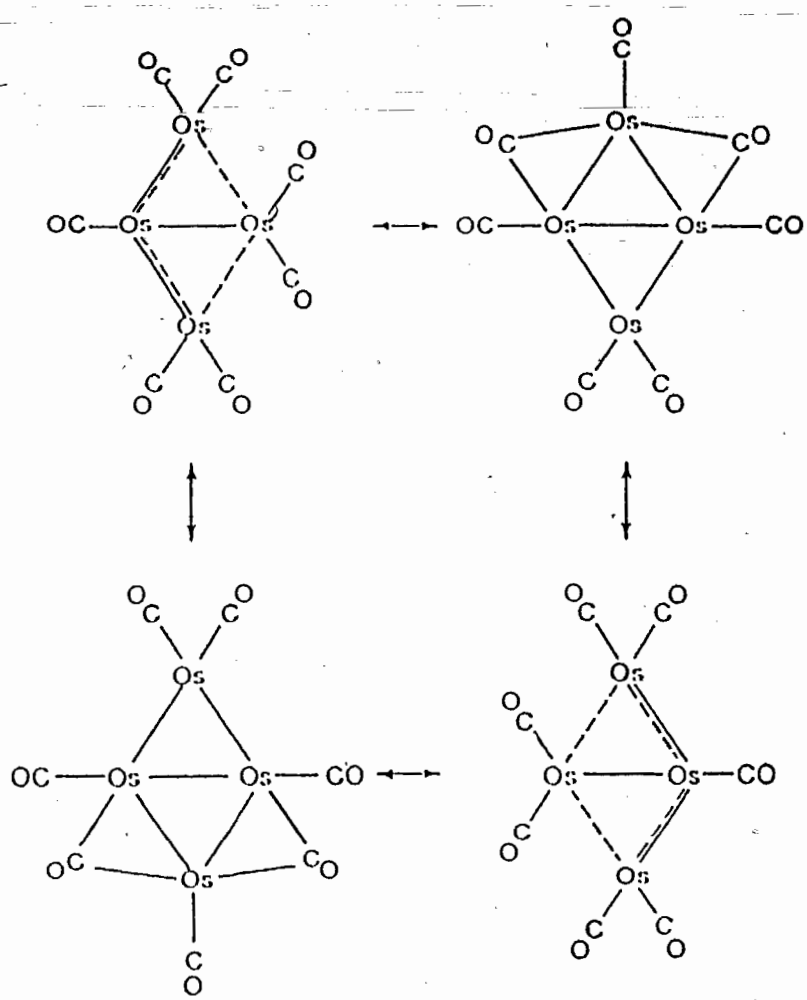


Figure 2.4. Variable Temperature ^{13}C NMR Spectra of $Os_4(CO)_{15}$.

Scheme 2.4



indicate that the CO-exchange is slowing on the NMR time scale. This observation also allows the further assignment of the resonances at δ 188.9 and 190.6 to the axial carbonyls on the hinge and wingtip osmium atoms, respectively.

If the mechanism shown in Scheme 2.4 is correct, an estimate for the barrier to exchange can be derived in the following way: The resonances due to the axial carbonyls on Os(1) and Os(3) in $Os_4(CO)_{15}$ in the absence of exchange are expected to have a chemical shift difference ($\Delta\nu$) of at least 35 ppm or ≃ 3500 Hz

at an operating frequency of 100.6 MHz. This value was chosen by analogy to the chemical shift difference for the resonances due to the corresponding carbonyls in the closely related derivatives $Os_4(CO)_{14}(PMe_3)$[20] and $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (in the absence of exchange these are 41.8 ppm and 39.9 ppm, respectively, see Chapter 3). The rate constant of an exchange process required to cause a detectable broadening ($\simeq$ 0.5 Hz) of a sharp signal is given by $\pi(\Delta\nu)^2$, which gives a value of $\simeq 10^7\ s^{-1}$ for the exchange at -55 °C.[71] Terminal-bridged CO-exchange in equatorial planes of clusters has been proposed before in the related compound $Os_4(CO)_{14}(PMe_3)$,[20] and in $[Os(CO)_3(PMe_3)]_2M(CO)_5$ (M = Cr,[72] Mo,[73] W[73]). However, exchange involving carbonyls in axial planes is much more common in cluster compounds.[68-70] Further discussion of this unusual fluxional process is postponed until Chapter 3.

When the sample is warmed to 0 °C, the resonances at δ 190.6 and 171.7 begin to broaden indicating the onset of a second fluxional process. This may be a three-fold twist mechanism taking place at the wingtip osmium atoms that exchanges the axial and equatorial carbonyls of these groups. A similar mechanism has been proposed to account for some aspects of the fluxional behavior of $Os_3(CO)_{12-x}[P(OMe)_3]_x$ (x = 1 - 5).[68] The ^{13}C NMR spectra of $Os_4(CO)_{15}$ at higher temperatures have not been recorded.

Reactivity

At room temperature, $Os_4(CO)_{15}$ is thermally stable both as a solid and in solution, but it is quite reactive. Addition of a donor ligand (*e.g.*, CO, PMe_3, $CNBu^t$, or $SbPh_3$) to a dichloromethane solution of $Os_4(CO)_{15}$ at room temperature or below leads to adduct formation (eqn 2.2). The 64-electron clusters so formed are discussed in Section 2.3 (L = CO) and in Chapter 4.

$$Os_4(CO)_{15} + L \longrightarrow Os_4(CO)_{15}(L) \qquad (2.2)$$

L = Two Electron Donor Ligand

It is interesting that no substitution reactions were observed. This is probably due to the ability of 62-electron species $Os_4(CO)_{15}$ to expand its metal core to accommodate the additional ligand, and to the stability of the 64-electron product to ligand loss. Other aspects of the reaction chemistry of $Os_4(CO)_{15}$ are discussed in the following sections.

2.2 $Os_4(CO)_{16}$: A Metal Carbonyl Analogue of Cyclobutane

Synthesis and Structure

Treatment of a dichloromethane solution of $Os_4(CO)_{15}$ with an atmosphere of CO at 0 °C caused essentially quantitative conversion to $Os_4(CO)_{16}$. The compound was isolated from the reaction mixture as air-stable, yellow-orange crystals in 86% yield.

$$Os_4(CO)_{15} \xrightarrow[0\ ^\circ C]{CO\ 1\ atm} Os_4(CO)_{16} \qquad (2.3)$$

The structure of this 64-electron cluster consists of a puckered-square arrangement of osmium atoms, each with four terminal carbonyl ligands (see Figures 2.5 and 2.6). The cluster is a metal carbonyl analogue of cyclobutane ($Os(CO)_4$ is isolobal with CH_2).[55] The dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(4)-Os(3) is 158.9° (cyclobutane has a dihedral angle of ≃ 145°[74]), and each osmium atom is in a pseudo-octahedral environment, see Table 2.2.

Most 64-electron clusters reported in the literature fall into three broad categories. The first group involves clusters which have distorted square geometries. Unlike $Os_4(CO)_{16}$, the metal framework in these clusters is supported by edge-bridging or face-capping ligands. Some examples of this type are $Pt_4(CH_3COO)_8$,[75] $Ir_4(CO)_8[(MeCO)_2C_2]_4$,[76] and

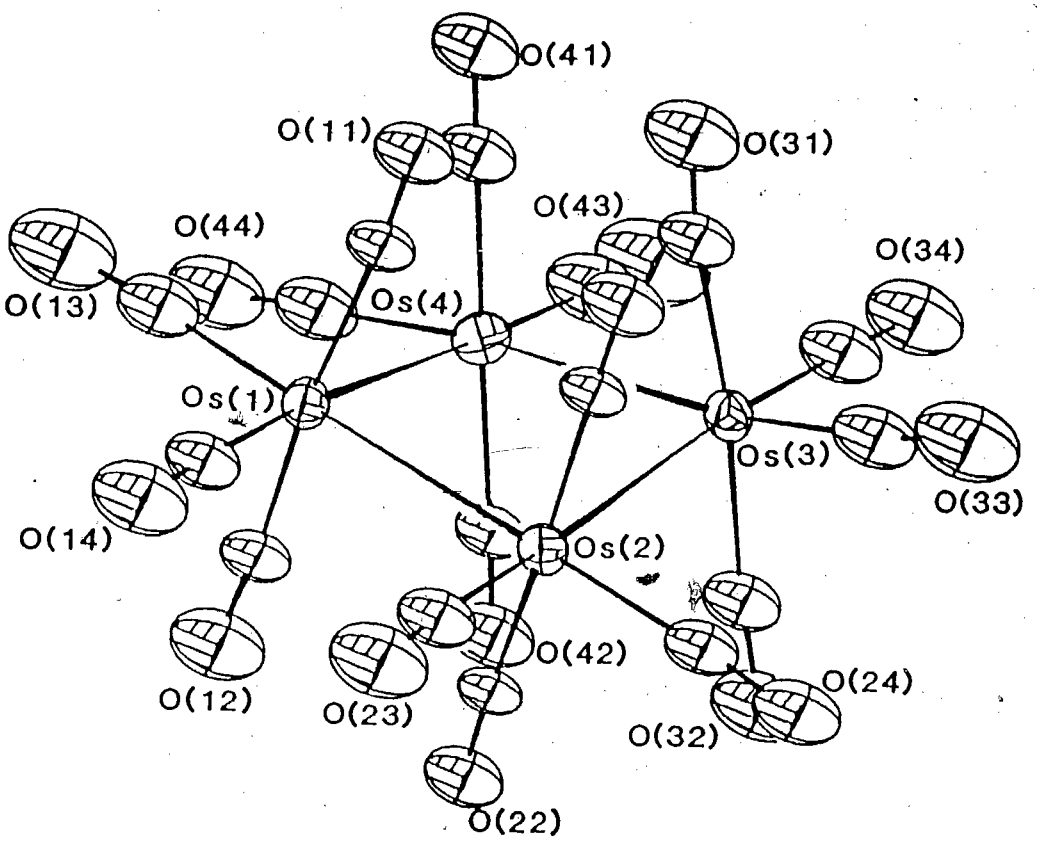


Figure 2.5. Molecular Structure of $Os_4(CO)_{16}$.

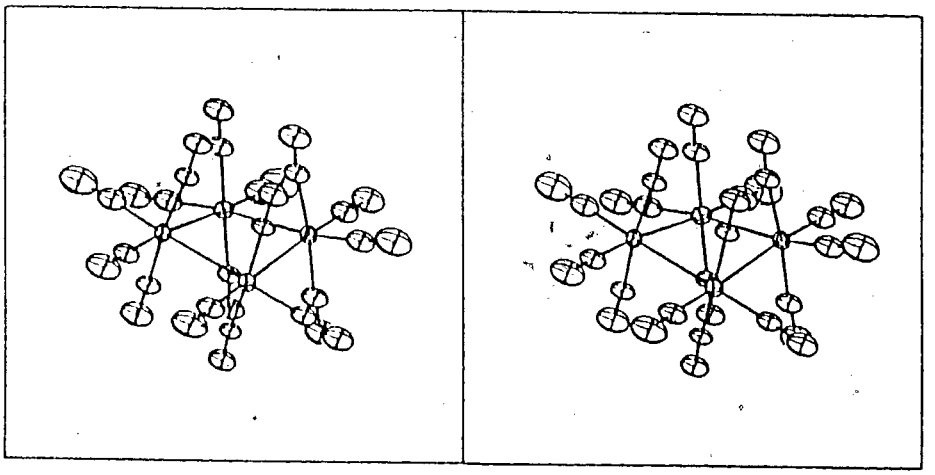


Figure 2.6. Stereoview of the Structure of $Os_4(CO)_{16}$.

$Fe_4(CO)_{12}(\mu_4\text{-}PPh)_2$.[77] Another common geometry is the spiked-triangle. Examples of this type are the clusters $Os_4(CO)_{15}(PMe_3)$,[20] $Os_4(\mu\text{-}H)_2(CO)_{15}$,[15] $Os_3Re(\mu\text{-}H)(CO)_{16}$,[78] and $Os_3Re(\mu\text{-}H)(CO)_{15}(NCMe)$[79] (other clusters of this type are reported in Chapter 4).

These two types of clusters have been termed *electron-precise*, since they possess the correct number of metal-metal bonds (four) to allow the cluster as a whole to obey the 18-electron rule.[80] A third type of 64-electron clusters consists of *electron-rich* butterfly clusters that possess five metal-metal bonds.[80] Some examples of this class are $Os_4(CO)_{12}(\mu_3\text{-}E)_2$ (E = S,[81] Se[82]), $Os_3W(CO)_{12}(PMe_2Ph)(\mu_3\text{-}S)_2$,[83] $Ru_4(CO)_{13}(PPh_2)_2$,[55] $Ru_4(CO)_{10}(PPh_2)_4$,[55] and $FeRu_3(CO)_{13}(PPh_2)_2$.[84] Note that as with the square geometry, the metal framework in these clusters is supported by bridging or capping ligands.

At the time of its preparation, the structure of $Os_4(CO)_{16}$ was therefore unique. It was the first example of a four membered ring of metal atoms held together exclusively by metal-metal bonds. Subsequently, it was found that $Os_4(CO)_{15}(PF_3)$ also adopts this unusual structure. Possible reasons why the $Os_4(CO)_{15}(L)$ clusters adopt the puckered-square arrangement when L = CO or PF_3 and the spiked-triangular geometry when L = PMe_3,[20] $CNBu^t$, $P(OCH_2)_3CMe$, or $SbPh_3$ are discussed in Chapter 4.

The osmium-osmium bonds in $Os_4(CO)_{16}$ (Table 2.2) are strikingly long compared to those in $Os_3(CO)_{12}$; the average

Table 2.2. Selected Molecular Dimensions of $Os_4(CO)_{16}$.

Bond Lengths (Å)

Os(1)-Os(2)	2.997 (1)	Os(1)-Os(4)	2.985 (1)
Os(2)-Os(3)	2.979 (1)	Os(3)-Os(4)	3.000 (1)
Os(1)-C(11)	1.97 (2)	Os(1)-C(12)	1.93 (2)
Os(1)-C(13)	1.87 (2)	Os(1)-C(14)	1.91 (2)
Os(2)-C(21)	1.94 (2)	Os(2)-C(22)	1.95 (2)
Os(2)-C(23)	1.89 (2)	Os(2)-C(24)	1.89 (2)
Os(3)-C(31)	1.92 (2)	Os(3)-C(32)	1.95 (2)
Os(3)-C(33)	1.86 (2)	Os(3)-C(34)	1.88 (2)
Os(4)-C(41)	1.94 (2)	Os(4)-C(42)	1.97 (2)
Os(4)-C(43)	1.84 (2)	Os(4)-C(44)	1.90 (2)
C(11)-O(11)	1.13 (2)	C(12)-O(12)	1.15 (2)
C(13)-O(13)	1.16 (3)	C(14)-O(14)	1.12 (3)
C(21)-O(21)	1.12 (2)	C(22)-O(22)	1.11 (2)
C(23)-O(23)	1.15 (3)	C(24)-O(24)	1.16 (3)
C(31)-O(31)	1.15 (2)	C(32)-O(32)	1.13 (2)
C(33)-O(33)	1.17 (3)	C(34)-O(34)	1.14 (3)
C(41)-O(41)	1.12 (2)	C(42)-O(42)	1.11 (2)
C(43)-O(43)	1.14 (4)	C(44)-O(44)	1.13 (3)

Os-Os bond length in $Os_4(CO)_{16}$ is 2.99 Å, whereas in $Os_3(CO)_{12}$ it is 2.88 Å.[50] The bonding in $Os_3(CO)_{12}$ is often considered in terms of two-center, two-electron bonds between the osmium atoms of the three $Os(CO)_4$ fragments.[53] If this were the case then it might be expected that the Os-Os bonds in $Os_4(CO)_{16}$ would be stronger than those in $Os_3(CO)_{12}$ because in the former cluster the angles about the osmium atoms more closely approach the 90° required for octahedral coordination.

The Os-Os bonds in $Os_4(CO)_{16}$ are also slightly longer than Os-Os single bonds that are not bridged by ligands or

Table 2.2. Continued.

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(4)	89.22 (3)	Os(1)-Os(2)-Os(3)	88.89 (3)
Os(2)-Os(3)-Os(4)	89.27 (3)	Os(3)-Os(4)-Os(1)	88.73 (3)
Os(2)-Os(1)-C(11)	84.1 (6)	Os(3)-Os(2)-C(21)	81.3 (6)
C(11)-Os(1)-C(12)	171.8 (9)	C(21)-Os(2)-C(22)	173.0 (8)
C(11)-Os(1)-C(13)	92.8 (8)	C(21)-Os(2)-C(23)	91.5 (8)
C(11)-Os(1)-C(14)	92.6 (8)	C(21)-Os(2)-C(24)	93.7 (8)
C(13)-Os(1)-Os(2)	176.6 (6)	C(23)-Os(2)-Os(3)	172.5 (6)
C(13)-Os(1)-C(14)	93.0 (10)	C(23)-Os(2)-C(24)	93.8 (8)
C(31)-Os(3)-Os(4)	81.6 (6)	C(41)-Os(4)-Os(1)	86.2 (7)
C(31)-Os(3)-C(32)	172.9 (9)	C(41)-Os(4)-C(42)	175.2 (8)
C(31)-Os(3)-C(33)	88.5 (9)	C(41)-Os(4)-C(43)	90.6 (10)
C(31)-Os(3)-C(34)	94.7 (9)	C(41)-Os(4)-C(44)	89.7 (9)
C(33)-Os(3)-Os(4)	169.9 (6)	C(43)-Os(4)-Os(1)	176.6 (7)
C(33)-Os(3)-C(34)	94.2 (10)	C(43)-Os(4)-C(44)	95.1 (10)

metal-ligand fragments. No structurally characterized binuclear complexes of osmium which contain an unbridged, single covalent Os-Os bond have been reported, but the structure of $(OC)_5OsOs(CO)_3(Cl)(GeCl_3)$ with an unbridged, dative osmium-osmium bond has been reported.[18] This compound is a member of a series of bimetallic complexes prepared in our laboratory that have an 18-electron complex acting as a ligand towards a 16-electron fragment.[19] Other examples of dative Os-Os single bonds are found in the spiked-triangular clusters, $Os_4(CO)_{15}(PMe_3)$,[20] $Os_4(CO)_{15}(CNBu^t)$, and $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ (the last two clusters are discussed in Chapter 4). The relevant bond lengths in these compounds are collected in Table 2.3 together with the Os-Os bond lengths in $Os_3(CO)_{12}(I)_2$[85] and

Table 2.3. Unbridged Os-Os Single Bond Lengths.

Compound	Bond Type	D(Os-Os) (Å)	Ref
$Os_2(CO)_8(GeCl_3)(Cl)$[a]	dative	2.931 (1)	18
	dative	2.927 (2)	
	dative	2.916 (2)	
$Os_4(CO)_{15}(PMe_3)$[b]	dative	2.939 (1)	20
	dative	2.937 (1)	
$Os_4(CO)_{15}(CNBu^t)$	dative	2.918 (2)	*c*
$Os_4(CO)_{15}[P(OCH_2)_3CMe]$	dative	2.926 (1)	*c*
$Os_3(CO)_{12}(I)_2$[d]	covalent	2.935 (2)	85
$Os_3(CO)_{12}(SiCl_3)_2$[d]	covalent	2.912 (2)	86

[a]Three crystallographically independent molecules. [b]Two crystallographically independent molecules. [c]This work, see Chapter 4. [d]The two Os-Os bond lengths are equivalent by symmetry.

$Os_3(CO)_{12}(SiCl_3)_2$[86] (both these compounds have a symmetry imposed linear Os_3 chain). As can be seen, there is no chemically significant difference between the lengths of the covalent and dative Os-Os bonds in these compounds. The ligands trans to the Os-Os bonds are expected to have some influence on the length of the bonds,[87] but from the values in Table 2.3 these effects seem to be small.

It has been found that a dative bond may in fact be slightly longer than a single covalent bond. In the closely related isomers $(OC)_5OsRe(CO)_4(Br)$ (dative Os-Re bond) and $(OC)_4(Br)OsRe(CO)_5$ (covalent Os-Re bond) the Os-Re bond lengths are 3.006 (1) Å and 2.977 (1) Å, respectively.[88] The difference of 0.029 Å has definite crystallographic significance, but it is

hardly significant in a chemical sense; it is roughly equal to the range of the known dative Os-Os bonds (0.025 Å). Therefore, the average value of the bond lengths in Table 2.3 (2.93 Å) is probably a reasonable estimate of an Os-Os single bond. The average Os-Os bond length in $Os_4(CO)_{16}$ (2.99 Å) is thus only slightly longer (by $\simeq$ 0.06 Å) than a single Os-Os bond, while that in $Os_3(CO)_{12}$ (2.88 Å) is slightly shorter (by $\simeq$ 0.05 Å). It is interesting to compare these differences to those between the carbon-carbon bonds in the organic analogues. These are 1.57 Å for cyclobutane, and 1.52 Å in cyclopropane.[89] A value of 1.54 Å is usually considered as typical for a C-C single bond, so the C-C bonds in cyclobutane are 0.03 Å longer, and those in cyclopropane are 0.02 Å shorter than a single bond. The similarity is quite intriguing. Not only is the pattern of bond length variation the same, but the lengthening (in $Os_4(CO)_{16}$) and shortening (in $Os_3(CO)_{12}$) is proportionally the same as that observed in the organic molecules.

The long C-C bonds observed in cyclobutane have been attributed to steric repulsion across the ring diagonal, and the short C-C bonds in cyclopropane have been explained in terms of bent bonds.[89] Similar arguments can be invoked for $Os_4(CO)_{16}$ and $Os_3(CO)_{12}$, but the similarity between these clusters and their organic counterparts should not be overstated. In spite of the relative C-C bond lengths, cyclobutane is thermodynamically more stable than cyclopropane.[90] On the other hand, when stirred in hexane solution at room temperature $Os_4(CO)_{16}$ decomposes

completely after 30 h to $Os_3(CO)_{12}$ plus a trace of $Os_4(CO)_{15}$. (Since $Os_4(CO)_{15}$ is stable under these conditions two different pathways must be involved in the decomposition.) A possible mechanism for the decomposition of $Os_4(CO)_{16}$ to $Os_3(CO)_{12}$ is discussed in Chapter 4. The important conclusion for this discussion is that *$Os_4(CO)_{16}$ is thermodynamically unstable with respect to $Os_3(CO)_{12}$*. Given that the vast majority of transition metal cluster compounds are based upon deltahedra this is not too surprising. Many clusters, particularly those of higher nuclearity, are prepared under extreme conditions. As noted in Chapter 1, for example, the pyrolysis of $Os_3(CO)_{12}$ at 210 °C affords $Os_6(CO)_{18}$ and smaller amounts of $Os_5(CO)_{16}$, $Os_5(CO)_{15}(C)$, $Os_6(CO)_{18}$ $Os_7(CO)_{21}$, $Os_8(CO)_{23}$, and $Os_8(CO)_{21}(C)$.[39] All of these clusters are comprised of triangular Os_3 building blocks.[42-45]

A reasonable explanation of the long Os-Os bonds in $Os_4(CO)_{16}$ and the low thermal stability of this cluster in solution can be found by considering the geometrical differences of square and triangular configurations. The bonding in $Os_3(CO)_{12}$ has been investigated by Trogler and co-workers using non-relativistic Xα calculations.[91] The authors report that the Os-Os bonding in $Os_3(CO)_{12}$ takes the form of a centrally directed molecular orbital of a_1 symmetry and an e' set directed along the edges of the Os_3 triangle (see Figure 2.7). Molecular orbital calculations on $Os_4(CO)_{16}$ are not yet available, but a qualitative picture of the Os-Os bonding can be derived from the

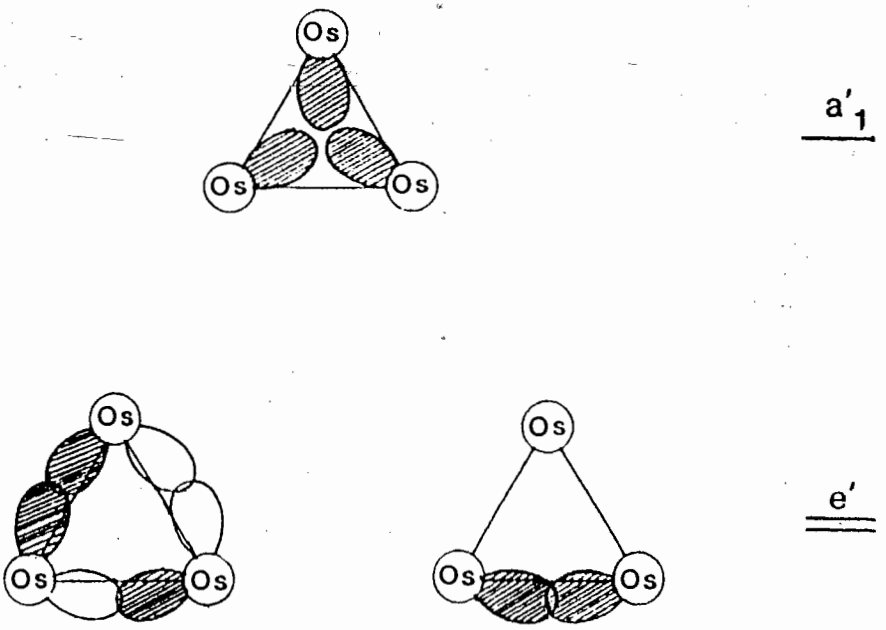


Figure 2.7. Os-Os Bonding in $Os_3(CO)_{12}$.

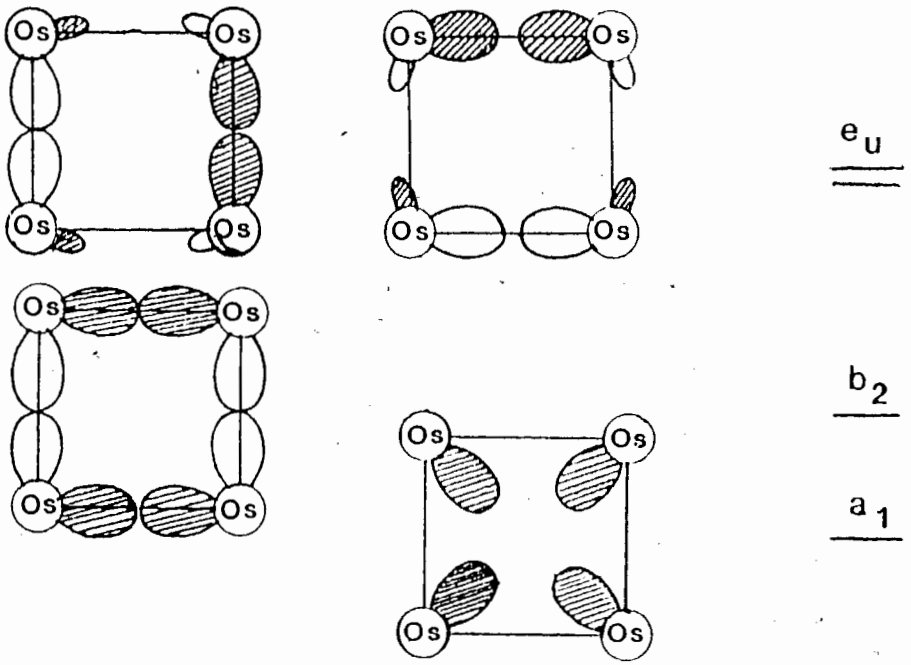


Figure 2.8. Proposed Os-Os Bonding in $Os_4(CO)_{16}$.

$Os(CO)_4$ fragment orbitals.[92] This is shown in Figure 2.8. Although little can be said about the energies of the orbitals, it is expected that the a_1 molecular orbital of $Os_4(CO)_{16}$ will be much higher in energy than its counterpart in $Os_3(CO)_{12}$. This is because the distance from an osmium atom to the centroid of the ring is $\simeq$ 2.1 Å in $Os_4(CO)_{16}$, while it is only $\simeq$ 1.2 Å in $Os_3(CO)_{12}$. Osmium-osmium bond lengths of $\simeq$ 1.8 Å would be required for osmium atoms of $Os_4(CO)_{16}$ to approach within 1.2 Å of the centroid. Because such Os-Os contacts are unobtainable, the overlap of atomic orbitals giving rise to the a_1 molecular orbital in $Os_4(CO)_{16}$ is poor and this orbital is destabilized.[93] It seems probable that this destabilization is a major cause of instability of the square cluster.

There may be other electronic factors that contribute to the instability of the square cluster. The bonding in $Os_2(CO)_8(C_2H_4)$ (an isolobal intermediate between cyclobutane and $Os_4(CO)_{16}$) and $Os_2(CO)_8(CH_2)$ (an intermediate between cyclopropane and $Os_3(CO)_{12}$) has recently been investigated.[94] The authors report that the diosmium compounds have framework bonding orbitals that correspond to the orbitals of the organic analogues, plus an additional orbital that originates from the remnants of the t_{2g} sets of the $Os(CO)_4$ groups. In both $Os_2(CO)_8(CH_2)$ and $Os_2(CO)_8(C_2H_4)$ this extra orbital is Os-C antibonding. In addition, the HOMO of the square cluster is also Os-C antibonding. This latter orbital is thought to account for the facile ejection of ethylene from $Os_2(CO)_8(C_2H_4)$.[94] Although

molecular orbital calculations are necessary to determine if a similar situation is present in $Os_4(CO)_{16}$, these results do at least suggest the possibility that an analogous Os-Os antibonding interaction exists. As will be discussed in Chapter 4, the puckered-square clusters may in fact dissociate into ethylene-like intermediates in solution.

If the Os-Os bond lengths in $Os_4(CO)_{16}$ were 2.88 Å as found in $Os_3(CO)_{12}$,[50] then there would be twelve C···C contacts of $\simeq$ 2.88 Å (assuming a D_{4h} conformation). In $Os_3(CO)_{12}$, there are only six C···C contacts of this length (the contacts between the carbon atoms of the equatorial carbonyls on adjacent osmium atoms of $Os_3(CO)_{12}$ are $\simeq$ 3.4 Å). It seems reasonable that the Os-Os bonds in $Os_4(CO)_{16}$ may lengthen to minimize these interactions, particularly since the Os-Os bonds in the square cluster are weak. That the Os-Os bonds in $Os_3(CO)_{12}$ are so strong attests to the importance of the centrally directed molecular orbital.

A method of quantifying the steric effects exerted on the structures of cluster compounds by the ligands has been put forward by Lauher.[65] In this model the metal atoms are used to define a surface about which the ligands are allowed to move. The movement of the ligands is determined by the minimization of non-bonded interactions between neighboring groups (within the constraints of metal-ligand bonding). The method allows the calculation of the relative steric energy ($E_{steric} = E_{tot}/n_{CO}$), which is a useful quantity for making comparisons between

different molecules.[65]

Lauher has reported the application of this model to $Os_3(CO)_{12}$ in both the observed D_{3h} and twisted D_3 conformations (Table 2.4).[95] The model predicts that the most sterically favored geometry is D_3 with a steric energy of 0.72 kcal per carbonyl, while the observed geometry, D_{3h}, has a steric energy of 0.98 kcal per carbonyl. Thus for $Os_3(CO)_{12}$, the sterically less favored geometry is observed, indicating that the structure of $Os_3(CO)_{12}$ is governed more by electronic considerations than by steric factors.[65,95]

For $Os_4(CO)_{16}$, Lauher has investigated three conformations.[95] He estimated the steric energy to be 1.08 kcal per carbonyl for a planar, symmetric (D_{4h}) conformation, 0.83 kcal per carbonyl for a planar, twisted (D_4) conformation, and 1.05 kcal per carbonyl for the observed puckered conformation, (D_2). Lauher concludes that since the lowest energy conformation is not observed, the instability of $Os_4(CO)_{16}$ cannot be due entirely to steric factors.[95] It must also be true, however, that the structure of $Os_4(CO)_{16}$ is not governed solely by electronic considerations, since the observed conformation is D_2 and not D_{4h}. The torsion angles around the Os-Os bonds are similar for opposite bond pairs: C(11)-Os(1)-Os(2)-C(21) = 6.4 (9)°, C(31)-Os(3)-Os(4)-C(41) = 8.4 (10)°, C(11)-Os(1)-Os(4)-C(41) = 25.6 (9)°, and C(21)-Os(2)-Os(3)-C(31) = 25.4 (9)°. The Os-Os bonds associated with the smaller torsion angles (Os(1)-Os(2) = 2.997 (1) Å and Os(3)-Os(4) = 3.000 (1) Å) are slightly longer

Table 2.4. Calculated Steric Energies in $Os_4(CO)_{16}$ and $Os_3(CO)_{12}$.[a]

Cluster	Point Group	Total Energy (kcal)	Steric energy (kcal per carbonyl)
$Os_4(CO)_{16}$[b]	D_{4h}	17.3	1.08
	D_4	13.3	0.83
	D_2	16.8	1.05
$Os_3(CO)_{12}$[c]	D_{3h}	11.7	0.98
	D_3	8.7	0.72

[a]Data are taken from ref 95. [b]Assumes Os-Os bond lengths of 2.99 Å. [c]Assumes Os-Os bond lengths of 2.85 Å.

than those associated with the larger torsion angles (Os(1)-Os(4) = 2.985 (1) Å and Os(2)-Os(3) = 2.979 (1) Å). (These differences are probably due to the interactions of the eclipsed carbonyls along the Os(1)-Os(2) and Os(3)-Os(4) bonds.) Thus the interactions of the carbonyls in $Os_4(CO)_{16}$ are minimized by both the puckering of the ring and by a slight pairwise skewing of the $Os(CO)_4$ groups. It is also important to note that the observed geometry of $Os_4(CO)_{16}$ is (by Lauher's method) 5 kcal more strained than $Os_3(CO)_{12}$ (in the observed geometry),[95] and if the Os-Os bond lengths in $Os_4(CO)_{16}$ were as short as those in $Os_3(CO)_{12}$ this difference would be even greater.

Solution Structure of $Os_4(CO)_{16}$

The carbonyl stretching region of the infrared spectra of $Os_4(CO)_{16}$ and $Os_3(CO)_{12}$ in hexane are shown in Figure 2.9. As can be seen they are remarkably similar. The observation of six carbonyl stretches in the IR spectrum of $Os_4(CO)_{16}$ in hexane is consistent with a puckered structure in solution. Group theory predicts four CO-stretches for a D_{4h} conformer and six for a D_{2d} conformer. The ^{13}C NMR spectra of $Os_4(CO)_{16}$ (^{13}CO-enriched) at 0 °C and -90 °C consist of two singlets of equal intensity in the carbonyl region, consistent with rapid ring inversion such that the "inner" and "outer" axial carbonyl ligands of the puckered structure are rendered equivalent. Such a process may involve the symmetric D_{4h} structure as an intermediate or transition state.

There may be axial-equatorial CO-exchange in $Os_4(CO)_{16}$ on the synthetic time scale. A solution of $Os_4(^{12}CO)_{15}$ in CD_2Cl_2 was treated with an atmosphere of ^{13}CO in an NMR tube, and the reaction (eqn 2.4) followed by ^{13}C NMR spectroscopy.

$$Os_4(CO)_{15} + {}^{13}CO \xrightarrow[CD_2Cl_2]{0\ ^\circ C} Os_4(CO)_{15}(^{13}CO) \qquad (2.4)$$

Figure 2.10 shows the spectrum (carbonyl region) recorded after 4 h (the large resonance at δ 184.5 is due to free ^{13}CO).[96] As can be seen, there is no evidence for selective enrichment of either the axial or equatorial sites. Thus either the addition occurs with no stereospecificity, or there is an exchange

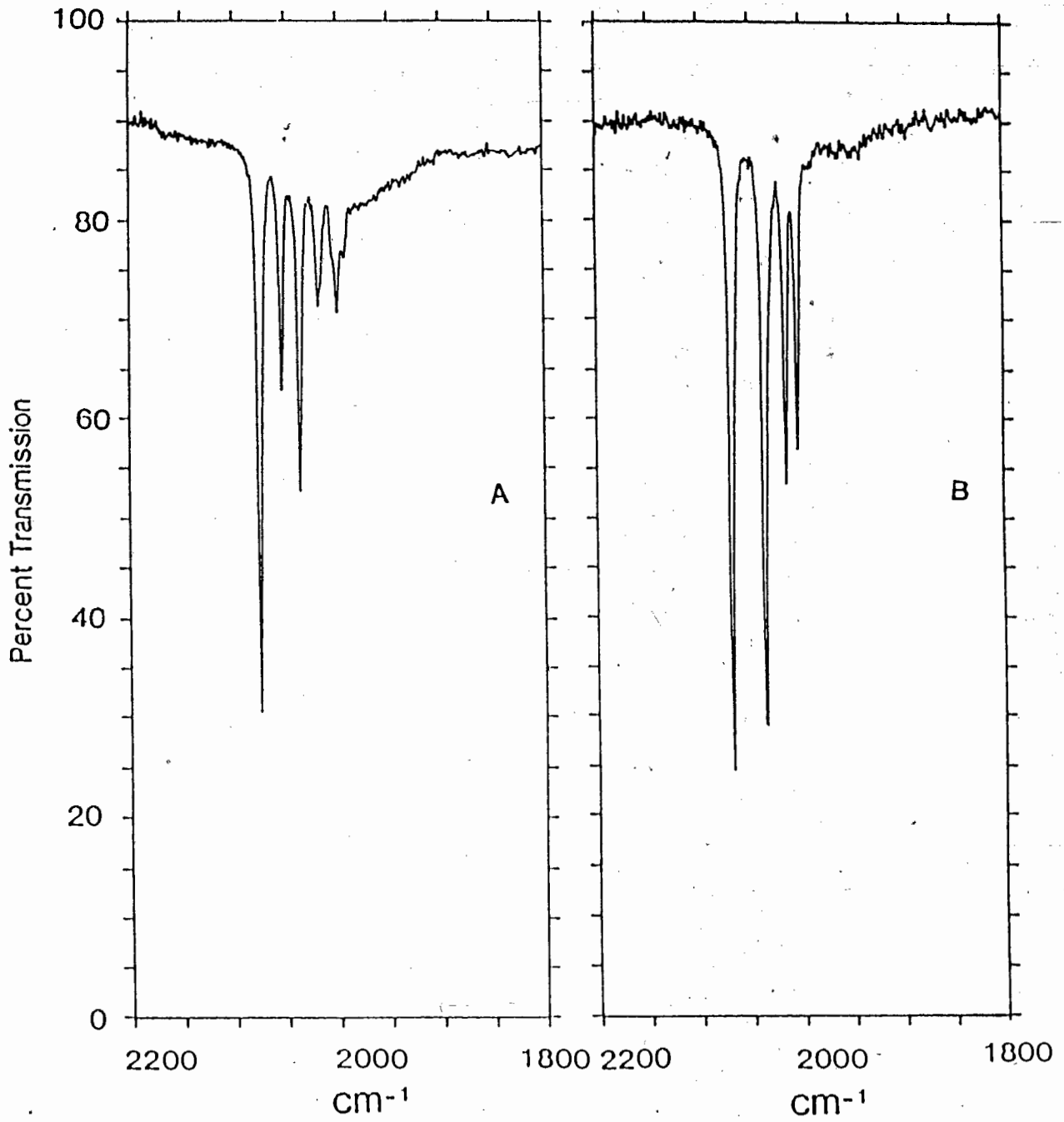


Figure 2.9. Infrared Spectra of $Os_4(CO)_{16}$ (A) and $Os_3(CO)_{12}$ (B) in Hexane (2200 - 1800 cm^{-1}).

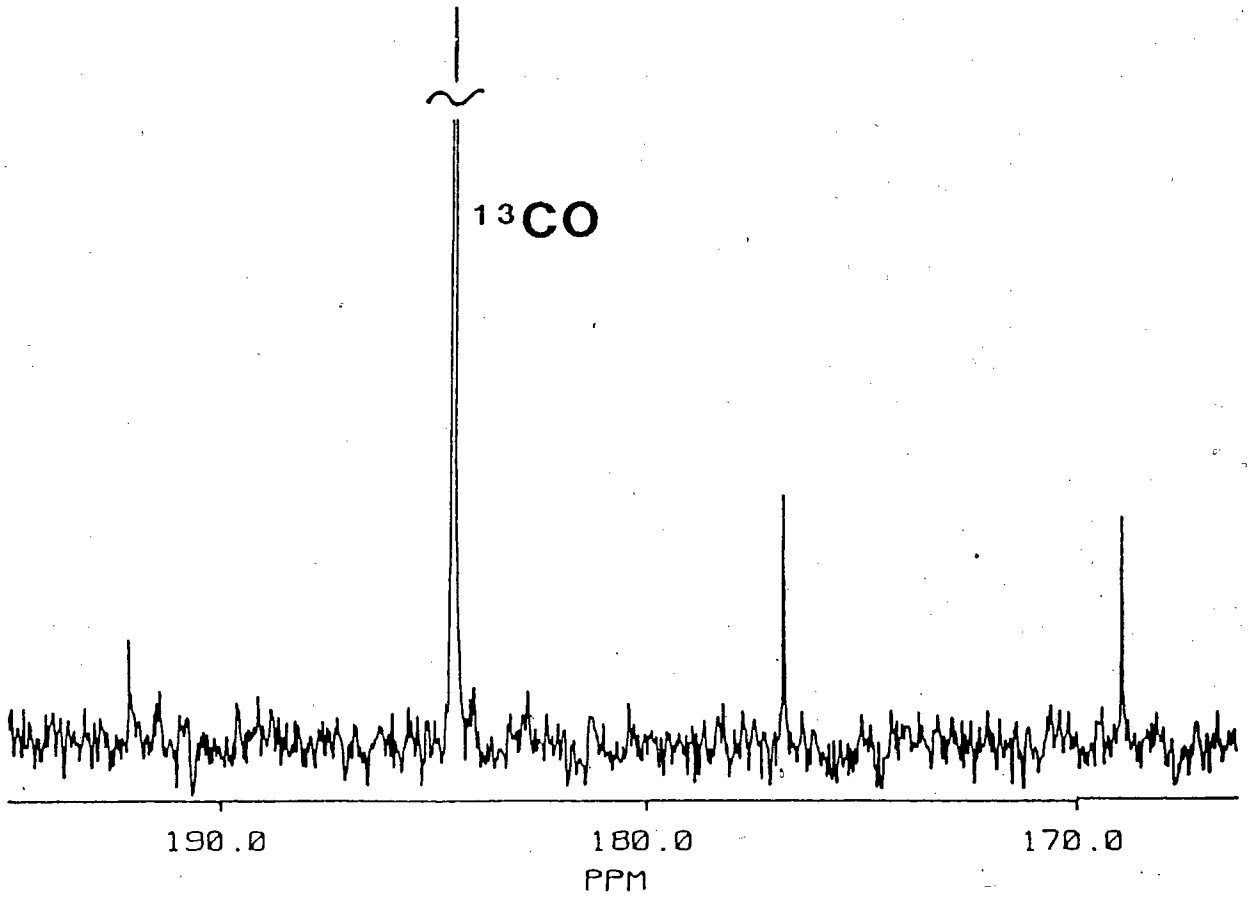


Figure 2.10. ^{13}C NMR Spectrum of $Os_4(CO)_{15}(^{13}CO)$ in CD_2Cl_2 at 0 °C.

process between the axial and equatorial carbonyl ligands of $Os_4(CO)_{16}$, which, although slow on the NMR time scale, is rapid on the synthetic time scale.

2.3 $Os_4(CO)_{14}$: A 60-electron Cluster that is Both Sterically Crowded and Coordinatively Unsaturated

Preparation of $Os_4(CO)_{14}$

The stability and therefore existence of tetrahedral transition metal clusters with 14 carbonyl ligands has been the subject of speculation. It was argued that a tetrahedral arrangement of transition metal atoms could not accommodate a shroud of 14 carbonyl ligands because of excessive steric interactions of the ligands.[64,97] Arguments based upon molecular orbital theory also led to the prediction that such a cluster would be unstable.[58]

More recent work suggested that these predictions were wrong. The trimethylphosphine derivative, $Os_4(CO)_{13}(PMe_3)$, was prepared and characterized by Dr. L. R. Martin of our laboratory,[20] and Shriver and co-workers reported the preparation of the tetrahedral cluster anions $[Fe_3M(\mu\text{-}CO)_2(CO)_{12}]^{2-}$ (M = Cr, Mo, W).[63] Additionally, the electron impact mass spectrum of $Os_4(CO)_{15}$ contained only a small envelope of peaks for the parent ion but an intense set of peaks corresponding to $[P\text{-}CO]^+$. These results indicated that $Os_4(CO)_{14}$ should be capable of existence and that it might be prepared by the decarbonylation of $Os_4(CO)_{15}$. Photolysis of $Os_4(CO)_{14}(PMe_3)$ in hexane has proven the most effective method for producing $Os_4(CO)_{13}(PMe_3)$.[20] However, because of the low solubility of $Os_4(CO)_{15}$ in hexane at room temperature this method could not be employed. Photolysis

of $Os_4(CO)_{15}$ in benzene or toluene was tried, but this led to a complex mixture of products. Somewhat suprisingly pyrolysis of $Os_4(CO)_{15}$ at 80 °C in hexane did produce $Os_4(CO)_{14}$ in ≃ 90% yield (pyrolysis of $Os_4(CO)_{14}(PMe_3)$ gave only poor yields of $Os_4(CO)_{13}(PMe_3)$[20]). The reaction is reversible; when the sealed reaction vessel was allowed to stand at room tempertature $Os_4(CO)_{15}$ was reformed quantitatively (by IR). For this reason the reaction solution had to be carefully degassed to drive the reaction to completion.

$$Os_4(CO)_{15} \xrightarrow[\text{hexane}]{80\ ^\circ C} Os_4(CO)_{14} + CO \qquad (2.5)$$

The reported yield is of the crude product; depending on the conditions, varying (but minor) amounts of impurities identified (by IR and ^1H NMR spectroscopy) as $Os_3(CO)_{12}$ and $Os_4(\mu\text{-}H)_4(CO)_{12}$[98] were also produced. The analytical sample of $Os_4(CO)_{14}$ was obtained by recrystallization of the sample twice from CH_2Cl_2/hexane. The source of the hydrogen atoms in $Os_4(\mu\text{-}H)_4(CO)_{12}$ was not investigated, but it might be from the carbon-hydrogen activation of the solvent (hexane).

Structure of $Os_4(CO)_{14}$

The structure of $Os_4(CO)_{14}$ was determined by X-ray crystallography. The cluster has a pseudo-tetrahedral arrangement of osmium atoms with 14, essentially terminal carbonyl ligands (Figure 2.11). There is a crystallographic C_2 axis bisecting the bonds Os(1)-Os(1B) and Os(2)-Os(2B). The

Table 2.5. Selected Molecular Dimensions of $Os_4(CO)_{14}$.

Bond Lengths (Å)

Os(1)-Os(2)	2.892 (1)	Os(1)-Os(1B)	2.834 (1)
Os(1)-Os(2B)	2.810 (1)	Os(2)-Os(2B)	2.763 (1)
Os(1)-C(11)	1.97 (2)	Os(1)-C(12)	1.94 (2)
Os(1)-C(13)	1.93 (2)	Os(1)-C(14)	1.94 (2)
Os(2)-C(21)	1.90 (2)	Os(2)-C(22)	1.87 (2)
Os(2)-C(23)	1.88 (2)	C(11)-O(11)	1.13 (2)
C(12)-O(12)	1.16 (2)	C(13)-O(13)	1.14 (2)
C(14)-O(14)	1.12 (2)	C(21)-O(21)	1.16 (2)
C(22)-O(22)	1.15 (2)	C(23)-O(23)	1.14 (2)

Selected Bond Angles (deg)

Os(1B)-Os(1)-Os(2)	58.77 (2)	Os(1B)-Os(1)-Os(2B)	61.64 (2)
Os(2B)-Os(1)-Os(2)	57.94 (2)	Os(1)-Os(2)-Os(1B)	59.60 (2)
Os(1)-Os(2)-Os(2B)	59.55 (2)	Os(1B)-Os(2)-Os(2B)	62.51 (2)
Os(2)-Os(1)-C(11)	121.6 (5)	C(12)-Os(1)-C(11)	174.4 (7)
C(13)-Os(1)-C(12)	90.0 (7)	C(14)-Os(1)-C(13)	101.6 (7)
Os(1)-Os(2)-C(21)	73.6 (3)	C(22)-Os(2)-C(21)	93.2 (8)
C(23)-Os(2)-C(22)	92.2 (8)	Os(1)-C(11)-O(11)	162.5 (15)
Os(1)-C(12)-O(12)	163.4 (16)	Os(1)-C(13)-O(13)	177.2 (14)
Os(1)-C(14)-O(14)	176.8 (16)	Os(2)-C(21)-O(21)	175.5 (16)
Os(2)-C(22)-O(22)	177.8 (17)	Os(2)-C(23)-O(23)	177.9 (16)

Os-Os bond lengths are Os(1)-Os(1B) = 2.834 (1) Å, Os(1)-Os(2) = 2.892 (1) Å, and Os(1)-Os(2B) = 2.810 (1) Å, and Os(2)-Os(2B) = 2.763 (1) Å (see Table 2.5 for additional molecular dimensions). There are four weakly semibridging contacts involving two carbonyl ligands on each of the two $Os(CO)_4$ groups and the osmium atoms of the two $Os(CO)_3$ groups. These contacts are Os(2)···C(12) = 2.68 (2) Å and Os(2B)···C(11) = 2.66 (2) Å (and their symmetry equivalents).

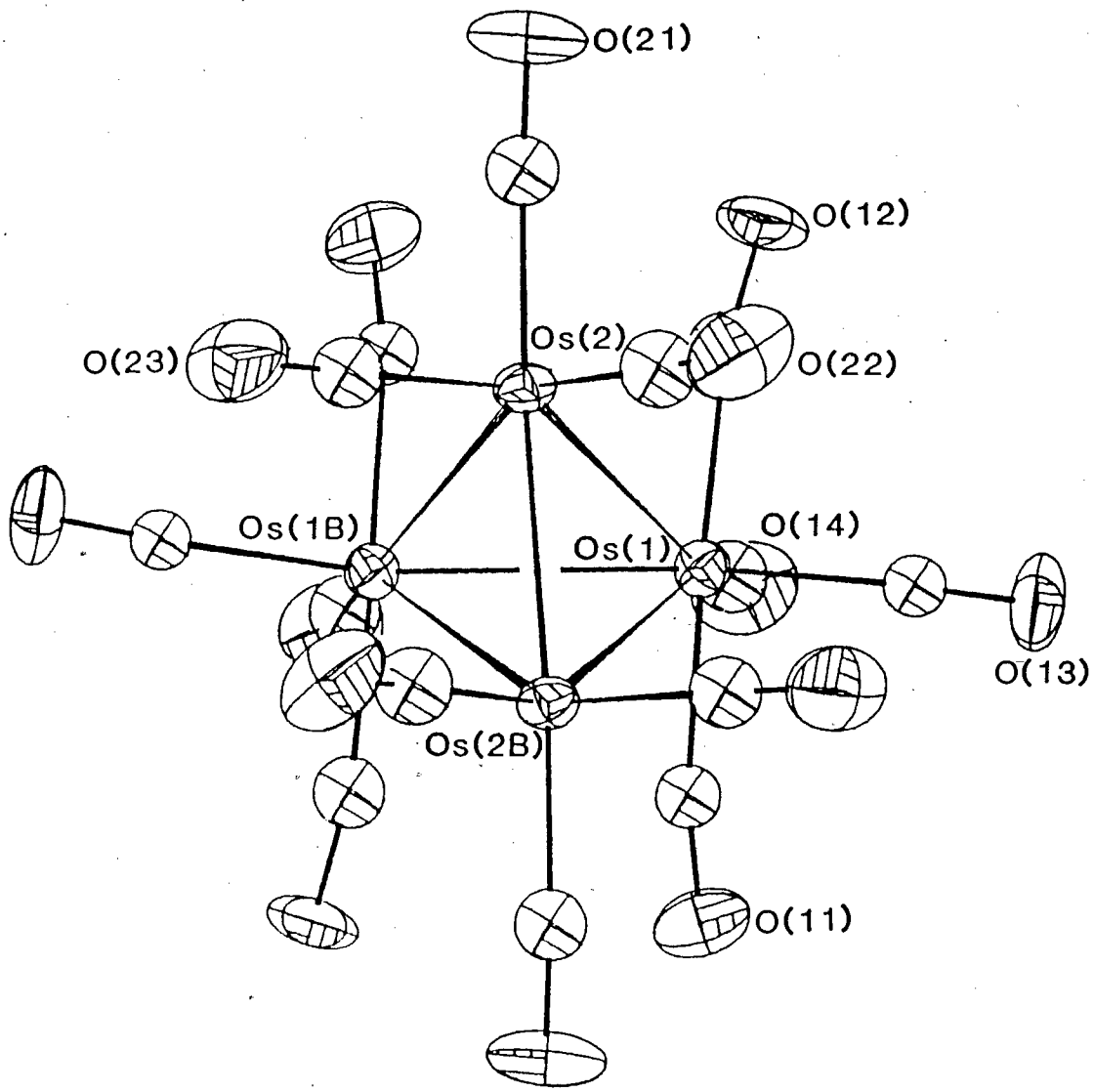
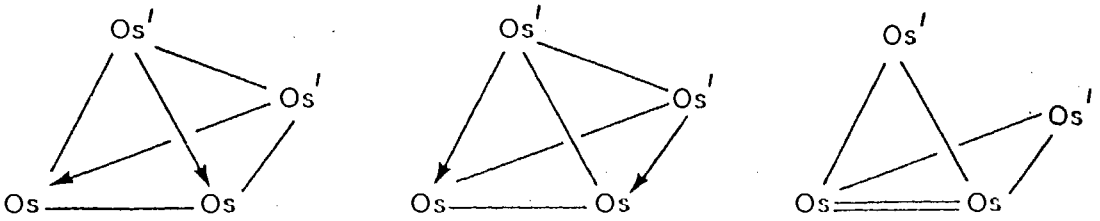


Figure 2.11. Molecular Structure of $Os_4(CO)_{14}$.

A pseudo-tetrahedral geometry is predicted for a 60-electron cluster by PSEPT.[51] Consideration of the 18-electron rule leads to the prediction of six metal-metal bonds for $Os_4(CO)_{14}$, and can be used as shown in Scheme 2.5 to rationalize the observed variation in Os-Os bond lengths. Two of the resonance structures shown in the scheme involve dative Os-Os bonds linking an $Os(CO)_4$ vertex to an $Os(CO)_3$ group. These dative bonds correspond to those which are semibridged by carbonyl ligands. Semibridging carbonyls across dative metal-metal bonds are common and are thought to offset the polar nature of these bonds.[99]

The third resonance structure shown in the scheme involves a double-bond between the two $Os(CO)_3$ units, and no bond between the two $Os(CO)_4$ groups. Contribution from this structure would

Scheme 2.5



$Os \equiv Os(CO)_3$ $Os' \equiv Os(CO)_4$

account for the short Os(2)-Os(2B) bond length of 2.76 Å. This length is slightly longer than the Os-Os vectors found in the $Os(\mu\text{-}H)_2Os$ units of $Os_3(\mu\text{-}H)_2(CO)_{10}$,[100] $Os_3(\mu\text{-}H)_3(CO)_9(SiPh_3)$,[101] and the green[102] and red[103] isomers of $Os_3(\mu\text{-}H)_2(CO)_9(CNBu^t)$. For these compounds the relevent Os-Os bond lengths are 2.683 (1) Å, 2.7079 (4) Å, 2.680 (1) Å, and 2.690 (1) Å, respectively. The bonding in these units has been considered in terms of a formal osmium-osmium double bond,[104] although more recent theoretical studies suggest that the Os-Os bonding in these units should be considered mainly as three-center, two-electron $Os(\mu\text{-}H)Os$ bonds.[105]

On the other hand, the short Os(2)-Os(2B) vector in $Os_4(CO)_{14}$ may simply reflect the better bonding capabilities of the C_{3v} $Os(CO)_3$ fragment. Work by Mingos suggests that such fragments, as opposed to T-shaped $Os(CO)_3$, or $Os(CO)_4$ fragments, are better suited for forming strong metal-metal bonds in cluster compounds.[58]

One view of the structures of metal carbonyl clusters considers the relationship between the metal and ligand polyhedra. As proposed by Johnson, the ligands are thought to arrange themselves on the surface of a sphere around the metal atoms so as to minimize the non-bonded interactions between neighboring groups.[106] The lowest energy polyhedron (of carbonyls) with a sufficiently large cavity to accommodate the metal atoms will be favored. The model works reasonably well for clusters that are nearly spherical, but breaks down when planar

or irregular-shaped clusters are considered.[107]

Since $Os_4(CO)_{14}$ has a pseudo-tetrahedral core of osmium atoms it seems an ideal candidate for Johnson's model, especially since it has been suggested that the ligand polyhedra will only influence the structure in sterically congested species.[97] In an extension of Tolman's cone angle arguments, Mingos has proposed that the maximum number of carbonyl ligands that can be accommodated around a tetrahedron of third-row transition metal atoms is 13.2.[97] Thus $Os_4(CO)_{14}$ is predicted to be strained, and it is reasonable to expect the requirements of the ligands to influence the structure. However, the polyhedron formed by the carbonyl carbon atoms of $Os_4(CO)_{14}$ (Figure 2.12) does not resemble any of the lowest-energy 14-vertex polyhedra proposed by Johnson.[107] It should be noted at this point that contrary to arguments that $Os_4(CO)_{14}$ is highly strained, it is thermodynamically the most stable of the tetraosmium binary carbonyls (it is prepared in excellent yields from $Os_4(CO)_{15}$ at 80 °C).

As previously mentioned, the tetrahedral clusters $[Fe_3M(CO)_{14}]^{2-}$ (M = Cr, Mo, W) have been reported by Shriver and co-workers.[63] The crystal structure for M = Cr has been determined, and the ligand polyhedron (Figure 2.13) was discussed. As can be seen, the 14 carbonyl ligands of this cluster adopt a different geometry than that found for $Os_4(CO)_{14}$ (and different from the polyhedra predicted by Johnson's model[107]). The authors also report the results of strain energy

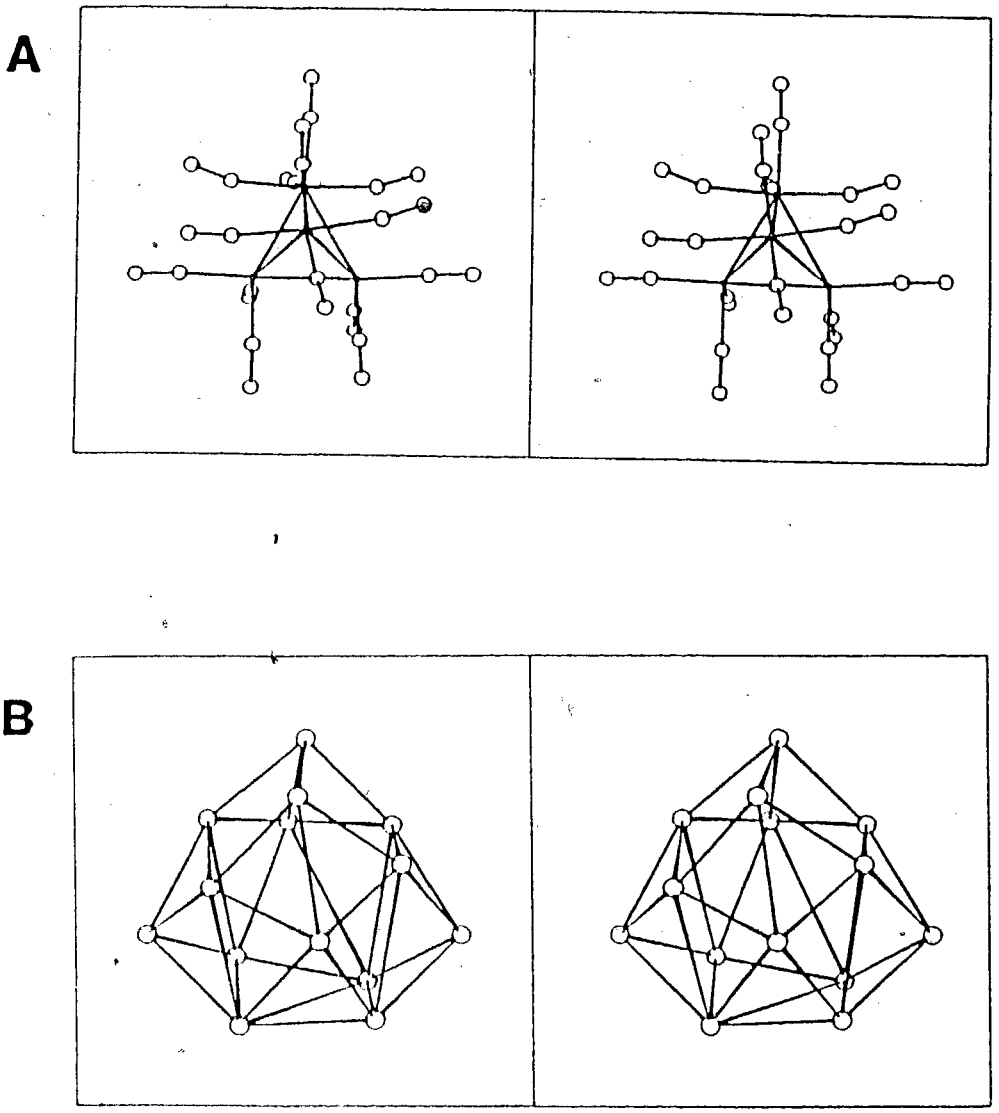


Figure 2.12. Stereoviews of the Structure of $Os_4(CO)_{14}$ (A) and the Polyhedron Formed by the Carbonyl Carbon Atoms (B).

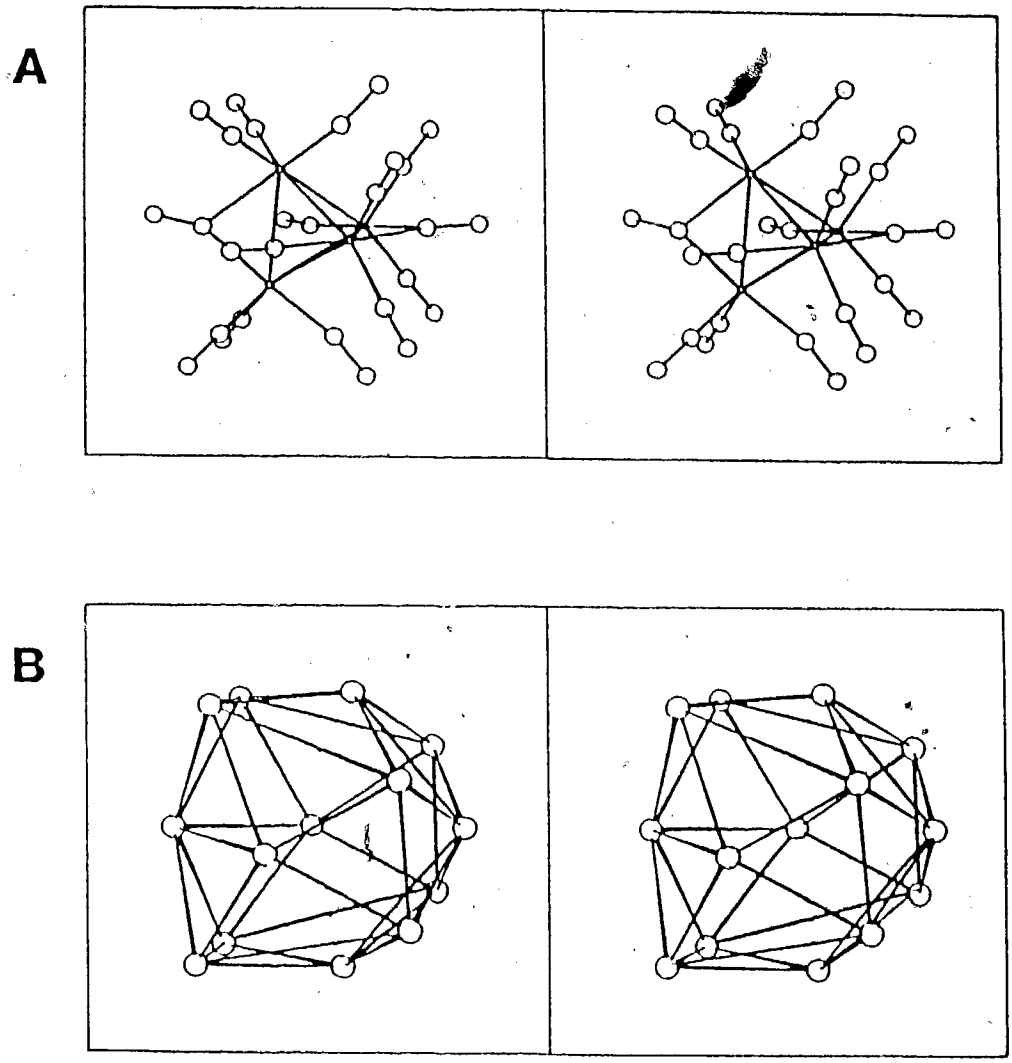


Figure 2.13. Stereoviews of the Structure of $[Fe_3Cr(CO)_{14}]^{2-}$ (A) and the Polyhedron Formed by the Carbonyl Carbon Atoms (B).

calculations on the anion. Consideration of the M-M and M-C bond lengths in the calculation led the authors to conclude that there is an isotropic expansion of the metal pseudo-tetrahedron as a consequence of the non-bonded C···C (and O···O) interactions. The observed structure is then a compromise of the competing factors involving the ligand-ligand interactions (repulsive) and the metal-metal and metal-ligand interactions (attractive).[63]

The $[Fe_3Cr(CO)_{14}]^{2-}$ cluster has two edge-bridging carbonyl ligands, whereas in $Os_4(CO)_{14}$ there are four weakly semibridging contacts. The difference is probably related to the increased ability of CO to bridge bonds between first-row transition metals relative to those between third-row transition metals.[108] Because of this difference, the carbonyl ligands of $Os_4(CO)_{14}$ are not as symmetrically disposed as are those of $[Fe_3Cr(CO)_{14}]^{2-}$, and the non-bonded interactions between the carbonyls in $Os_4(CO)_{14}$ are not symmetric. Closer examination of Figure 2.12 reveals that the deformation of the CO polyhedron (from a sphere) is in the direction of the short Os(2)-Os(2B) bond. Thus it is not possible to distinguish whether this bond distorts the CO polyhedron because it is short, or if the asymmetry resulting from the terminal CO ligands forces a contraction of this bond. However, the expansion of the Fe_3Cr core of $[Fe_3Cr(CO)_{14}]^{2-}$ as proposed by Shriver leads to average Fe-Fe and Fe-Cr bond lengths of $\simeq$ 2.66 Å and $\simeq$ 2.63 Å, respectively.[63] The Os-Os bonds in $Os_4(CO)_{14}$ are all

significantly longer than these, the shortest being Os(2)-Os(2B) = 2.763 (1) Å. Therefore, it is likely that the observed Os-Os bond lengths in $Os_4(CO)_{14}$ are governed by electronic considerations, and not by the steric congestion of the carbonyl ligands.

Nonrigidity in $Os_4(CO)_{14}$

The behavior of $Os_4(CO)_{14}$ in solution is extraordinary. The carbonyl region of the infrared spectrum of $Os_4(CO)_{14}$ in hexane consists of three broad absorptions at 2058(s), 2018(m), and 1938(vw) cm^{-1} (Figure 2.14). The last absorption is barely detectable above the base line. The broadness of the two stronger absorptions is remarkable. The peak width at half-height for the band at 2058 cm^{-1} is 14.5 cm^{-1}. For comparison, that for the most intense band in the spectrum of $Os_3(CO)_{12}$ under the same conditions is 5 cm^{-1}. Group theory predicts twelve infrared-active carbonyl stretches for $Os_4(CO)_{14}$ with C_{2v} symmetry (all CO ligands terminal). For the more symmetric D_{2d} geometry (four bridging carbonyl ligands), five infrared-active absorptions are expected in the terminal CO stretching region.

The ^{13}C NMR spectrum of $Os_4(CO)_{14}$ (in $CHFCl_2/CD_2Cl_2$) consists of a single sharp resonance at δ = 177.1 down to -130 °C. This behavior is reminiscent of that of $Fe_3(CO)_{12}$.[109] The ^{13}C NMR spectrum of this cluster consists of a singlet down to -150 °C. Furthermore, the infrared spectrum (CO-stretching region) of

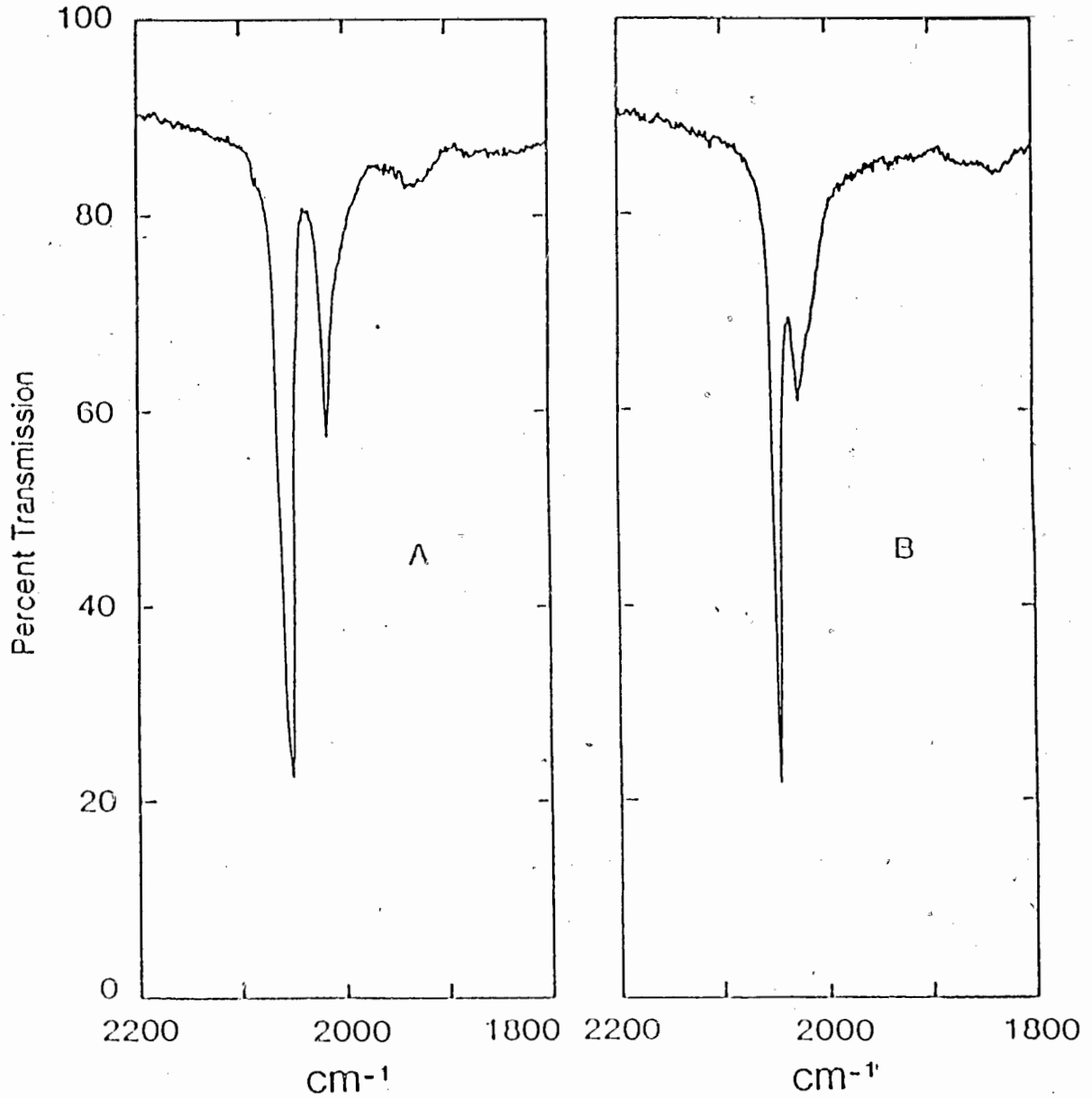


Figure 2.14. Infrared Spectra of $Os_4(CO)_{14}$ (A) and $Fe_3(CO)_{12}$ (B) in Hexane (2200 - 1800 cm^{-1}).

$Fe_3(CO)_{12}$ is remarkably similar to that of $Os_4(CO)_{14}$ (Figure 2.14). Cotton and Hunter have interpreted the spectral data of $Fe_3(CO)_{12}$ in terms of a continuum of isomers ranging from C_{2v}, as found in the solid state, to D_{3h}, as found for the ruthenium and osmium analogues.[109] Later, Johnson proposed that there is an equilibrium involving only three isomers: C_{2v}, D_3 (rather than D_{3h}), and one with two face capping CO groups.[110] In this model the ^{13}C NMR spectrum is interpreted in terms of the rapid interconversion of the three forms, with the D_3 isomer as the dominant species. The infrared spectrum results from the overlap of broad bands due to the dominant isomer plus minor contributions from the two other isomers.[110]

A recent EXAFS study on $Fe_3(CO)_{12}$ in light petroleum indicates that an isomer similar to the D_3 isomer proposed by Johnson is the major species in this solvent.[111] The authors further state that there is no clear evidence for a continuum of isomers in their results. Indeed, they state that there is only weak evidence for the presence of one minor isomer.[111] These results are most consistent with Johnson's proposal that the D_3 isomer is the major species in solution.[110]

Similar arguments could be invoked for $Os_4(CO)_{14}$, but it is difficult to envision an isomer that would give rise to the observed infrared spectrum. On the other hand, it is conceivable that CO-exchange in $Os_4(CO)_{14}$ is so fast as to cause broadening of the absorptions in the infrared spectrum. The difference in the chemical shifts ($\Delta\nu$) of chemically different carbonyls in

osmium clusters is usually at least 10 ppm or ≃ 1000 Hz at an operating frequency of 100.6 MHz. For example, the chemical shift separation of the resonances due to the axial and equatorial carbonyls of $Os_3(CO)_{12}$ is 11.9 ppm,[112] and the chemical shift separation of the resonances due to some of the carbonyls of $Os_4(CO)_{14}(PMe_3)$[20] and $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ is ≃ 40 ppm. The rate constant required to cause a detectable broadening (of ≃ 0.5 Hz) of two sites undergoing fast exchange is given by $\pi(\Delta\nu)^2$ (ref 71). If one uses a value of 1000 Hz for $\Delta\nu$, then the sharp singlet in the ^{13}C NMR spectrum of $Os_4(CO)_{14}$ at -130 °C suggests the carbonyls in this cluster are undergoing exchange with a rate constant of 3×10^6 s^{-1} at this temperature. If it is assumed that the rate constant doubles with every ten degree rise in temperature, then a minimum rate constant of ≃ 2×10^{11} s^{-1} is estimated for the CO-exchange in $Os_4(CO)_{14}$ at 30 °C. This rate is approaching that necessary to cause detectable broadening on the infrared time scale.[113]

Because of the possibility that rapid CO-exchange was the cause of the broad infrared spectrum of $Os_4(CO)_{14}$ at room temperature, variable temperature infrared spectra of $Os_4(CO)_{14}$ (in methylcyclohexane) were obtained (Figure 2.15).[114] As can be seen, at -90 °C there is indeed a splitting of the major bands in the spectrum, and other bands have grown in from the base line. This is consistent with the slowing of the CO-exchange on the infrared time scale. Dynamic rearrangement observable on the infrared time scale has recently been reported for some

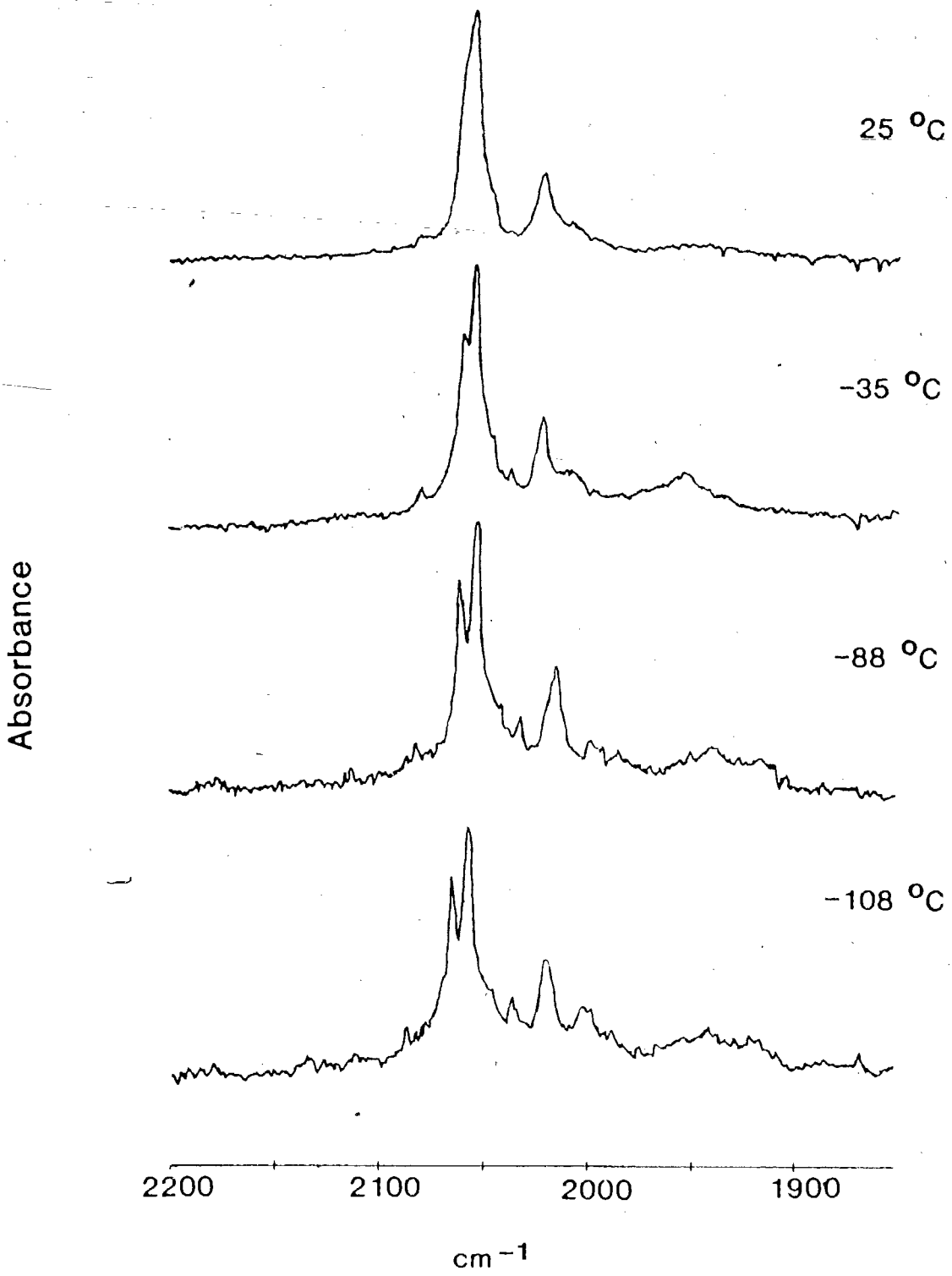


Figure 2.15. Variable Temperature Infrared Spectra of $Os_4(CO)_{14}$ (in Methylcyclohexane).

(η^4-diene)$Fe(CO)_3$ complexes.[115] It is also possible that rapid CO-exchange in $Fe_3(CO)_{12}$ gives rise to the observed infrared spectrum of this compound. Further studies are necessary to corroborate this proposal.

Reactivity of $Os_4(CO)_{14}$

As mentioned above, $Os_4(CO)_{14}$ readily adds CO to give $Os_4(CO)_{15}$. Since $Os_4(CO)_{15}$ also adds CO to give $Os_4(CO)_{16}$ and the latter cluster is thermally unstable in solution at room temperature, the reactions shown in eqn 2.6 may be responsible for the failure of other workers to isolated these clusters.

$$\begin{array}{ccc} Os_4(CO)_{14} & \xrightarrow{CO} & Os_4(CO)_{15} \\ & & \downarrow CO \\ Os_3(CO)_{12} & \longleftarrow & Os_4(CO)_{16} \end{array} \qquad (2.6)$$

The reaction of $Os_4(CO)_{14}$ with $CNBu^t$ (Chapter 4) follows a similar path as shown in eqn 2.6 ($Os_4(CO)_{14}(CNBu^t)_2$ was produced). The reactions of $Os_4(CO)_{14}$ with other donor ligands were not investigated.

The reactions of $Os_4(CO)_{14}$ with other small molecules such as H_2, HX, X_2, and HER_3 (X = halide; E = Si, Ge, Sn; R = alkyl, aryl, H) are currently being investigated by Mr. C.-Y. Lu of our laboratory. Preliminary results indicate that $Os_4(CO)_{14}$

oxidatively adds the H-E bond of a variety of HER_3 compounds (eqn 2.7).[46] The structure of the product from the reaction of $Os_4(CO)_{14}$ with $HSnMe_3$ is discussed in Chapter 3.

$$Os_4(CO)_{14} + HER_3 \xrightarrow[\text{hexane}]{\text{RT}} Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3) \quad (2.7)$$

One of the goals of this research was to use the ability of certain 18-electron complexes such as $Os(CO)_5$ to act as donor ligands in the development of rational synthetic routes to tetranuclear clusters. The synthesis of $Os_4(CO)_{15}$ (Section 2.2) and $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ (Chapter 3) demonstrate that this is clearly a viable route to cluster compounds. Preliminary work has shown that this approach can be extended to the synthesis of pentanuclear clusters. Thus $Os_4(CO)_{14}$ reacts with $Os(CO)_5$ in hexane at room temperature to give $Os_5(CO)_{19}$[40] (identified by IR). That the reaction (eqn 2.8) proceeds in only low yield is likely due to the poor donor ability of $Os(CO)_5$.

$$Os_4(CO)_{14} + Os(CO)_5 \xrightarrow[\text{hexane}]{\text{RT}} Os_5(CO)_{19} \quad (2.8)$$

It is known that $Os(CO)_4(PMe_3)$ is a much better donor than $Os(CO)_5$.[19] Reaction of $Os_4(CO)_{14}$ with this 18-electron complex proceeds smoothly over 20 h at room temperature to give $Os_5(CO)_{18}(PMe_3)$ (eqn 2.9).

$$Os_4(CO)_{14} + Os(CO)_4(PMe_3) \xrightarrow[CH_2Cl_2]{RT} Os_5(CO)_{18}(PMe_3) \qquad (2.9)$$

Mr C.-Y. Lu is continuing this work, and has obtained X-ray quality crystals of $Os_5(CO)_{18}(PMe_3)$. A recently completed crystallographic study revealed the compound to have a "bow-tie" configuration, similar to that found for $Os_5(CO)_{19}$.[61]

These examples illustrate that $Os_4(CO)_{14}$ is a highly reactive compound. It is expected that this novel cluster will prove to be a useful synthetic precursor to many new and interesting cluster compounds.

2.4 Experimental

2.4.1 Syntheses

General Comments

Unless otherwise stated, the following general procedures were followed for all syntheses reported in this thesis: All manipulations were carried out under a nitrogen atmosphere with standard Schlenk techniques. Hexane and toluene were refluxed over potassium, distilled, and stored under nitrogen prior to use. Dichloromethane and chloroform were treated similarly except P_2O_5 was used as the drying agent.

Starting materials were either purchased or prepared by the published methods (or minor variations thereof) that are cited below. ^{13}CO-enriched compounds were prepared from ^{13}CO-enriched $Os_3(CO)_{12}$ ($\simeq$ 35% ^{13}CO), which in turn was prepared by heating $Os_3(CO)_{12}$ in toluene at 125 °C under $\simeq$ 1.5 atm ^{13}CO (99% ^{13}C) for three days.

Infrared spectra were recorded with a Perkin-Elmer 983 spectrometer; the internal calibration of the instrument was checked periodically against the known absorption frequencies of gaseous CO. Electron impact (70 eV) and fast atom bombardment (FAB) mass spectra were recorded with a Kratos-MS 50 instrument (University of British Columbia, regional facility). The pattern of the envelope of ions at highest mass for each compound matched that simulated by computer for the species involved. NMR

spectra were recorded on a Bruker WM 400 or SY-100 spectrometer. The NMR line-shape simulations were obtained with a computer program written by Professor R. E. D. McClung of the University of Alberta. Melting points were determined on samples sealed in capillaries under air by use of a Gallenkamp or Mel-temp apparatus. Microanalyses were performed by Mr. M. K. Yang of the Microanalytical Laboratory of Simon Fraser University.

Preparation of $Os_4(CO)_{15}$

A Schlenk tube was placed in an ice-water bath and charged with $Os_3(CO)_{10}(COE)_2$[25] (60 mg, 0.057 mmol), CH_2Cl_2 (≃ 2mL), hexane (30 mL), and $Os(CO)_5$[23] (≃ 20 mg, ≃ 0.06 mmol). The vessel was taken out of the cold bath and placed in the freezer (-15 °C) for four days. Decanting of the mother liquor and washing with hexane afforded analytically pure, crystalline $Os_4(CO)_{15}$ (48 mg, 73%): mp 185 °C (dec); IR (CH_2Cl_2) ν(CO): 2086(s), 2073.5(m), 2045(vs), 2023(m), 2002(sh), 1939(m, br) cm^{-1}; ^{13}C NMR ($CHFCl_2/CD_2Cl_2$, 5/1, 0 °C) δ 190.6 (4C), 188.9 (4C), 171.7 (7C) (pattern invariant to -118 °C); MS (EI) m/z 1182(w) (M^+), 1154(s) ([M-CO]$^+$); Anal. Calcd for $C_{15}O_{15}Os_4$: C, 15.26; H, 0.0. Found: C, 15.51; H, 0.0.

Preparation of $Os_4(CO)_{16}$

A 125 mL round bottom flask fitted with a Teflon valve was charged with $Os_4(CO)_{15}$ (29 mg, 0.025 mmol) and CH_2Cl_2 (35 mL). The solution was degassed by three freeze-pump-thaw cycles. Approximately one atmosphere of CO was admitted over the cold

solution, and the reaction stirred at 0 °C for 4 h. The solvent and excess CO were removed on the vacuum line, and the yellow-orange residue dissolved in a minimum amount of CH_2Cl_2 and filtered through 1 cm of Celite. Hexane was layered on top of the filtrate, and the vessel stored at -15 °C whereupon $Os_4(CO)_{16}$ was obtained (26 mg, 86%): IR (hexane) ν(CO): 2075.5(vs), 2054(m), 2036.5(s), 2018.5(w), 2000(w), 1993(sh) cm^{-1}; ^{13}C NMR (CH_2Cl_2/CD_2Cl_2, 5/1, ambient temperature) δ 168.8 and 176.6 (pattern invariant to -95 °C); A satisfactory mass spectrum (EI, FAB) could not be obtained; peaks due to $[Os_3(CO)_{12}^+]$, $[Os_6(CO)_{18}^+]$, *etc.*, were observed in the EI spectrum; Anal. Calcd for $C_{16}O_{16}Os_4$: C, 15.90; H, 0.0. Found: C, 15.89; H, 0.0.

Preparation of $Os_4(CO)_{14}$

A thick-walled Pyrex tube was charged with $Os_4(CO)_{15}$ (38 mg, 0.033 mmol), and hexane (20 mL). The solution was degassed by three freeze-pump-thaw cycles, and the vessel placed in an oil bath at 80 °C and stirred for 4 h. The tube was removed from the oil bath and the solution degassed as above, and then the tube was returned to the oil bath for an additional 17 h. The tube was then removed from the oil bath, and the solution *immediately* degassed. The solvent was removed on the vacuum line, and the residue crystallized from hot hexane.

At this stage the isolated yield was 35 mg (92%) $Os_4(CO)_{14}$. However, the product did contain trace amounts of impurities

believed to be $Os_4(\mu\text{-}H)_4(CO)_{12}$[98] and $Os_3(CO)_{12}$ (as determined by IR and ^1H NMR spectroscopy) and had to be recrystallized twice from dichlorormethane/hexane to give the spectroscopically and analytically pure sample: mp 216 °C; IR (hexane) ν(CO) 2058(s), 2018(m), 1938(vw, br) cm^{-1}; ^{13}C NMR ($CHFCl_2/CD_2Cl_2$, 6/1, 0 °C) δ 117.1 (invarient to -130 °C); MS (EI) *m/z* 1154 (M^+); Anal. Calcd for $C_{14}O_{14}Os_4$: C, 14.58; H, 0.0. Found: C, 14.88; H, 0.0.

2.4.2 X-ray Crystallographic Studies

General Comments.

The general comments given here are relevant to all crystal structure determinations reported in this thesis. Additional information specific to a particular investigation is given in the appropriate sections and tables.

The crystals were fixed to glass fibers with epoxy (in air) and optically centered on an Enraf-Nonius CAD4F diffractometer. Unit cells were determined on the diffractometer with Mo *Kα* X-radiation (λ = 0.71069 Å). Intensity data were collected over a unique fraction of reciprocal space. The background was measured by extending the scan by 25% on both sides of the peak. Two strong reflections, approximately 90° apart in ϕ and with low values of χ were monitored after every hour of X-ray exposure time. Unless otherwise stated, these varied by less than ± 3% during the data collection. All data were scaled, and corrected for the effects of Lorentz, polarization, and absorption.

Neutral atom scattering factors with anomalous dispersion corrections were used.[116] The structures were solved as described below, and refined by full matrix least-squares. Unit weights were employed initially, but weighting schemes either based on counting statistics or of an empirical form were adopted during the latter stages of refinement. The choice of one scheme over the other was dictated by the computer programs used during the refinement. In the cases where counting statistics were used, the weight of a given reflection was given by $w = [\sigma^2(F) + K(F)^2]^{-1}$, and the value of K adjusted to remove trends in $\langle w(|F_o| - |F_c|)^2 \rangle$ in the error analysis. The empirical weighting schemes were of the Chebyshev type.[117] The specific form of the expressions were chosen to remove trends in $\langle w(|F_o| - |F_c|)^2 \rangle$ in the error analysis and are given in the appropriate sections. In the cases where extinction was included as a refinable parameter the corrected, calculated structure factor, F_c', was given by the expression: $F_c' = k|F_c|(1 + g|F_c|^2 \mathrm{Lp}/p_n)^{-1/2}$, where k is the scale, Lp is the Lorentz polarization factor, and $p_n = 1 + \cos^{2n}2\theta$. The values of g are given in the appropriate sections.

Computer programs used were from either *The NRC VAX 750/780 Crystal Structure System*[119] or the *CRYSTALS* program suite.[120] Thermal ellipsoid diagrams were drawn with the *SNOOPI* program,[121] and ball and stick models with *PLUTO*.[122] All programs were run on a MicroVAX II computer.

Table 2.6. Crystallographic Data for the Structure Determinations of $Os_4(CO)_n$ (n = 15, 16, 14).

Formula	$Os_4(CO)_{15}$	$Os_4(CO)_{16}$	$Os_4(CO)_{14}$
Crystal system	monoclinic	triclinic	monoclinic
Space group	$C2/c$	$P\bar{1}$	$C2/c$
temp, °C	-73±3	21	22
a (Å)	12.802(3)	9.436(1)	13.275(2)
b (Å)	10.217(3)	9.482(1)	13.033(1)
c (Å)	16.380(5)	14.082(2)	11.593(1)
α, (°)		87.67(1)	
β, (°)	91.39(2)	79.09(1)	90.435(9)
γ, (°)		69.69(1)	
V (Å^3)	2141.7	1159.9	2005.7
Z	4	2	4
FW	1180.8	1208.8	1152.8
ρ_c (g cm^{-3})	3.66	3.46	3.82
μ Mo K_α (cm^{-1})	237.56	219.42	253.61
Transmission	0.173-0.567	0.211-1.000[a]	0.480-1.000[a]
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$	$\omega - 2\theta$
Scan width (°)	0.6+0.35tanθ	1.3+0.35tanθ	0.9+0.35tanθ
Scan speed (°min^{-1})	0.7-2.8	1.65-2.75	0.9-2.8
Min-max 2θ (°)	0 - 50	0 - 50	0 - 50
Unique data	1887	4069	1762
Obs data, $I \geq 2.5\sigma(I)$	1204	3019	1301
Parameters	108	166	111
R_f [b]	0.044	0.045	0.033
R_{wf} [c]	0.060	0.054	0.040
Max |shift/error|	0.110	0.01	0.01
Max peak (e Å^{-3})	2.0(1)	2.8(4)	2.4(3)
$G.O.F.$ [d]	1.02	1.85	1.25

[a] normalized transmission coefficients. [b] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [c] $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$. [d] $G.O.F. = [\Sigma w(|F_o| - |F_c|)^2/N_{obs} - N_{var})]^{1/2}$.

$Os_4(CO)_{15}$

Crystals of $Os_4(CO)_{15}$ could only be grown by performing the reaction of $Os_3(CO)_{10}(COE)_2$ with $Os(CO)_5$ at -15 °C without stirring for a peroid of days. The crystals were dark red, thin plates. An initial data set was collected at room temperature on a crystal of dimensions 0.15 x 0.12 x 0.05 mm, and the structure solved by direct methods. After all the atoms were placed in the model, evidence was found that indicated the molecule was disordered in the unit cell. In order to better model the disorder, the data were recollected (on the same crystal) at -73 °C. Only the low temperature experiment will be described.

The unit cell was determined at -73 °C on the basis of 25 accurately centered reflections with $20° < 2\theta < 40°$. An analytical absorption correction was applied to the data. The space group was determined to be either $C2/c$ or Cc by the systematic absences. The final choice of $C2/c$ (disordered) over Cc (ordered) was based on the results of the refinement; the best ordered model in Cc had substantially worse agreement (R = 0.052, 120 variables) than the final disordered model in $C2/c$ (R = 0.044, 108 variables).

The asymmetric unit consists of two overlapping partial molecules of $Os_4(CO)_{15}$, each of half occupancy. The two-fold axis of space group $C2/c$ relates the two half molecules such that one of the half molecules is equivalent to the "missing" half of the other half molecule. The number of variables was

Table 2.7. Fractional Coordinates for $Os_4(CO)_{15}$.

Atom	x/a	y/b	z/c
Os(1)	0.4999(3)	0.3683(1)	0.7411(1)
Os(2)	0.5752(2)	0.2372(2)	0.6063(1)
Os(3)	0.4969(5)	0.0799(1)	0.7467(2)
Os(4)	0.4248(2)	0.2538(2)	0.8816(1)
O(11)	0.717 (5)	0.402 (5)	0.825 (4)
O(12)	0.282 (4)	0.423 (5)	0.665 (4)
O(13)	0.515 (3)	0.661 (3)	0.732 (2)
O(21)	0.798 (3)	0.248 (3)	0.677 (2)
O(22)	0.347 (4)	0.236 (5)	0.542 (2)
O(23)	0.622 (3)	0.488 (3)	0.515 (2)
O(24)	0.640 (3)	0.035 (4)	0.477 (2)
O(31)	0.721 (4)	0.086 (6)	0.824 (4)
O(32)	0.268 (4)	0.093 (6)	0.687 (4)
O(33)	0.560 (4)	-0.140 (4)	0.632 (3)
O(34)	0.455 (4)	-0.119 (4)	0.881 (3)
O(41)	0.641 (4)	0.227 (5)	0.971 (3)
O(42)	0.201 (3)	0.283 (4)	0.807 (2)
O(43)	0.397 (3)	0.524 (4)	0.956 (2)
O(44)	0.335 (3)	0.078 (4)	1.016 (3)
C(11)	0.637 (4)	0.374 (5)	0.796 (4)
C(12)	0.358 (4)	0.399 (5)	0.690 (4)
C(13)	0.508 (4)	0.553 (3)	0.731 (2)
C(21)	0.710 (4)	0.245 (5)	0.655 (3)
C(22)	0.421 (3)	0.237 (5)	0.564 (3)
C(23)	0.603 (4)	0.391 (4)	0.545 (3)
C(24)	0.606 (4)	0.121 (4)	0.519 (3)
C(31)	0.638 (5)	0.086 (5)	0.798 (3)
C(32)	0.358 (5)	0.092 (4)	0.709 (4)
C(33)	0.528 (4)	-0.050 (6)	0.669 (4)
C(34)	0.453 (4)	-0.043 (6)	0.830 (4)
C(41)	0.551 (3)	0.237 (5)	0.933 (2)
C(42)	0.285 (4)	0.268 (5)	0.833 (3)
C(43)	0.404 (4)	0.421 (4)	0.927 (3)
C(44)	0.364 (4)	0.134 (4)	0.959 (3)

limited by assigning one common isotropic temperature factor to the carbon atoms, another to the oxygen atoms, and one to each type of osmium atom. Bond length restraints were employed during the initial stages of the refinement on all Os-C and Os-O distances (these restraints were lifted in the final stages of the refinement). Restraints were also applied to Os-Os distances such that the chemically equivalent bond lengths were refined to their mean values.

After the refinement had converged, an empirical weighting scheme was adopted; the weight for a given reflection was given by $w = [4.35147t_0(X) + 5.61811t_1(X) + 1.65544t_2(X)]^{-1}$, where $X = |F_o|F_{max}^{-1}$ and t_n are polynomial functions of the Chebyshev series.[117] Additional details of data collection and refinement are listed in Table 2.6. Atomic positional parameters for one molecule of the disordered pair are listed in Table 2.7; bond lengths and selected bond angles are listed in Table 2.2. Thermal motion parameters, and observed and calculated structure factors are lised in Tables A.1 and A.2.

$Os_4(CO)_{16}$

Yellow, block-shaped crystals of $Os_4(CO)_{16}$ were grown from dichloromethane/hexane (1/1) at -15°C. A suitable specimen was cleaved to dimensions 0.24 x 0.21 x 0.16 mm. The unit cell was determined on the basis of 25 accurately centered reflections, widely spread through reciprocal space, with $31° < 2\theta < 47°$. The triclinic space group $P\bar{1}$ was confirmed as correct by the

Table 2.8. Fractional Coordinates for $Os_4(CO)_{16}$.

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.20396(8)	0.10379(9)	0.19292(5)
Os(2)	0.48344(8)	0.08064(9)	0.27312(5)
Os(3)	0.32955(8)	0.39914(9)	0.34398(5)
Os(4)	0.09870(8)	0.43798(9)	0.21605(5)
O(11)	0.0534 (16)	0.0797 (17)	0.4049 (10)
O(12)	0.3406 (19)	0.1642 (20)	-0.0140 (11)
O(13)	-0.0800 (23)	0.1096 (23)	0.1201 (13)
O(14)	0.3430 (20)	-0.2346 (21)	0.1629 (12)
O(21)	0.3386 (17)	0.0209 (18)	0.4766 (10)
O(22)	0.6043 (16)	0.1638 (17)	0.0692 (9)
O(23)	0.6382 (20)	-0.2555 (22)	0.2330 (12)
O(24)	0.7665 (18)	0.1052 (18)	0.3344 (10)
O(31)	0.0896 (18)	0.3278 (19)	0.4985 (11)
O(32)	0.5858 (19)	0.4463 (20)	0.1888 (12)
O(33)	0.5187 (22)	0.3381 (22)	0.5021 (13)
O(34)	0.2003 (20)	0.7327 (21)	0.3910 (12)
O(41)	-0.1560 (17)	0.4114 (18)	0.3792 (10)
O(42)	0.3615 (17)	0.4583 (17)	0.0555 (10)
O(43)	-0.0189 (25)	0.772 (3)	0.2418 (15)
O(44)	-0.1018 (19)	0.4442 (20)	0.0705 (11)
C(11)	0.1120 (21)	0.0912 (22)	0.3290 (13)
C(12)	0.2929 (22)	0.1446 (23)	0.0645 (13)
C(13)	0.026 (3)	0.112 (3)	0.1494 (15)
C(14)	0.2908 (23)	-0.109 (3)	0.1761 (13)
C(21)	0.3883 (21)	0.0453 (22)	0.4019 (13)
C(22)	0.5554 (20)	0.1354 (21)	0.1427 (12)
C(23)	0.5766 (24)	-0.128 (3)	0.2454 (14)
C(24)	0.6569 (23)	0.0968 (23)	0.3134 (13)
C(31)	0.1753 (23)	0.3513 (24)	0.4359 (13)
C(32)	0.4926 (24)	0.4231 (25)	0.2434 (14)
C(33)	0.451 (3)	0.360 (3)	0.4382 (15)
C(34)	0.2445 (25)	0.607 (3)	0.3720 (14)
C(41)	-0.0612 (24)	0.4188 (25)	0.3196 (14)
C(42)	0.2737 (22)	0.4438 (23)	0.1160 (13)
C(43)	0.028 (3)	0.643 (3)	0.2376 (17)
C(44)	-0.028 (3)	0.444 (3)	0.1250 (15)

successful refinement of the structure. The data suffered from severe absorption. Several absorption corrections were tried; an empirical correction based on ψ-scans gave the best agreement on an isotropic model and was therefore employed in the refinement.[123]

The osmium atoms were placed using a Patterson map. Subsequent refinement of the partial model followed by Fourier difference synthesis revealed the positions of the carbon and oxygen atoms. The osmium atoms were refined with anisotropic thermal motion parameters, while all other atoms were refined isotropically. A model with anisotropic oxygen atoms was tried, but these parameters refined to unreasonable values. Extinction was indicated in the strong low angle data and was added as a refinable parameter during the final cycles of refinement (g = 0.076 (7).[118] A weighting scheme based on counting statistics was employed ($K = 3 \times 10^{-4}$).

Additional details of data collection and structure refinement are collected in Table 2.6; atomic positional parameters are listed in Table 2.8. Bond lengths and selected bond angles are listed in Table 2.3, and thermal motion parameters, and observed and calculated structure factors are given in Tables B.1 and B.2, respectively.

$Os_4(CO)_{14}$

Deep-red irregular blocks of $Os_4(CO)_{14}$ were grown from a hexane/dichloromethane solution (2/1) by the slow cooling of the solution from room temperature to 0 °C. A crystal of approximate dimensions 0.21 x 0.19 x 0.11 mm was selected, and the unit cell determined on the basis of 25 accurately centered reflections with $26° < 2\theta < 45°$. The systematic absences were consistent with monoclinic space groups $C2/c$ or Cc; the choice of the centric group, $C2/c$, was shown to be correct by the successful solution and refinement of the the structure.

The irregular shape of the crystal could not be properly modeled; therefore, an empirical absorption correction based on ψ-scans was applied.[123] The osmium atoms were placed from a Patterson map. Refinement of the partial model followed by Fourier difference synthesis revealed the positions of the carbon and oxygen atoms. The asymmetric unit of the unit cell contains one $Os(CO)_3$ group and one $Os(CO)_4$ group (*i.e.*, half a molecule of $Os_4(CO)_{14}$), with all atoms on general positions. Refinement with all 16 atoms anisotropic converged with R = 0.034. However, the thermal motion parameters of some of the carbon atoms were unreasonable, so these atoms were refined with isotropic temperature factors. An analysis of the intense low angle reflections indicated the presence of extinction, and therefore this was added as a refinable parameter (g = 0.13 (1)).[118] After convergence of the model, a weighting scheme

Table 2.9. Fractional Coordinates for $Os_4(CO)_{14}$.

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.10418(4)	0.84889(5)	0.22419(5)
Os(2)	0.01665(4)	0.69141(5)	0.36776(5)
O(11)	0.0518 (10)	0.9009 (10)	-0.0272 (11)
O(12)	0.1942 (9)	0.8598 (12)	0.4687 (10)
O(13)	0.3092 (8)	0.7759 (11)	0.1358 (12)
O(14)	0.1332 (11)	1.0816 (11)	0.2306 (12)
O(21)	0.0707 (12)	0.7021 (12)	0.6242 (11)
O(22)	0.1530 (12)	0.5083 (12)	0.3372 (13)
O(23)	-0.1634 (11)	0.5579 (12)	0.4195 (12)
C(11)	0.0613 (11)	0.8684 (13)	0.0628 (14)
C(12)	0.1521 (13)	0.8429 (14)	0.3824 (16)
C(13)	0.2338 (11)	0.8025 (13)	0.1715 (13)
C(14)	0.1242 (12)	0.9964 (16)	0.2311 (15)
C(21)	0.0479 (12)	0.7016 (14)	0.5278 (15)
C(22)	0.1011 (14)	0.5787 (16)	0.3466 (16)
C(23)	-0.0946 (12)	0.6071 (15)	0.3984 (15)

based on counting statistics was adopted ($K = 4.5 \times 10^{-4}$).

Additional details of data collection and structure refinement are collected in Table 2.6. Atomic positional parameters are given in Table 2.9. Bond lengths and selected bond angles are listed in Table 2.5. Thermal motion parameters, and observed and calculated structure factors are listed in Tables C.1 and C.2, respectively.

CHAPTER 3

SOME DERIVATIVES OF $Os_4(CO)_{15}$

3.1 Introduction

The study reported in this Chapter was prompted by the unusual features of the 62-electon cluster $Os_4(CO)_{14}(PMe_3)$. As mentioned in Chapter 1, this cluster was prepared and characterized by Dr. L. R. Martin in our laboratory. It has an planar, "kite-like" Os_4 skeleton, with adjacent short and long peripheral Os-Os bonds.[20] In solution, $Os_4(CO)_{14}(PMe_3)$ is fluxional on the NMR time scale to low temperatures, and a ^{13}C NMR spectrum consistent with the solid-state structure of $Os_4(CO)_{14}(PMe_3)$ could only be obtained at -115 °C. A novel "windshield-wiper" mechanism was originally proposed to account for the unusual mode of collapse of the signals in the variable temperature ^{13}C NMR spectra of this cluster.[21] This mechanism was subsequently discarded in light of the results described in this thesis.

In order to better understand the unusual structure and fluxional behavior of $Os_4(CO)_{14}(PMe_3)$, five new 62-electron clusters have been prepared and characterized. The first, $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, was prepared by the addition of $P(OMe)_3$ to $Os_4(CO)_{13}(PMe_3)$[20] after an improved synthesis of the latter compound was developed. The binary carbonyl $Os_4(CO)_{15}$ (Chapter 2) and the heteronuclear cluster $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ were prepared by the reaction of $Os_3(CO)_{10}(COE)_2$ (COE =

cyclooctene)[25] with $Os(CO)_5$[23] and $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$,[24] respectively. Treatment of $Os_4(CO)_{15}(CNBu^t)$ (Chapter 4) with Me_3NO gave $Os_4(CO)_{14}(CNBu^t)$. The crystal structures of these compounds have been determined, as has that of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.[46] All but the hydrido cluster show a similar pattern of short and long Os-Os (or Os-Ir) bonds around a planar (or nearly planar) metal core. The significance of these observations is discussed below.

The stereochemical nonrigidity of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (in solution) has been investigated by variable temperature ^{13}C NMR spectroscopy. Somewhat surprisingly, a spectrum consistent with the solid-state structure of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ was obtained at -45 °C. This is in contrast to the behavior of $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$. The variable temperature ^{13}C NMR spectra of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ indicate that the carbonyls in this compound undergo a different type of exchange process than those in $Os_4(CO)_{15}$ or $Os_4(CO)_{14}(PMe_3)$. Mechanisms are proposed for the CO-exchange in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, and the difference in the fluxional behavior between $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, and $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$[20] are discussed.

3.2 Results

$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$

All attempts at the substitution of $P(OMe)_3$ for a carbonyl ligand in $Os_4(CO)_{14}(PMe_3)$ resulted in the fragmentation of the cluster. It was thought that the desired product, $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, might be prepared by the addition of $P(OMe)_3$ to the 60-electron cluster $Os_4(CO)_{13}(PMe_3)$. However, the reported preparation of $Os_4(CO)_{13}(PMe_3)$ (the thermolysis of $Os_4(CO)_{14}(PMe_3)$ in hexane) gave only minor amounts of this cluster.[20] Therefore, if $Os_4(CO)_{13}(PMe_3)$ was to be used as a synthetic starting material an improved preparation was necessary. It was subsequently found that UV photolysis of $Os_4(CO)_{14}(PMe_3)$ in hexane under an N_2 purge gave $Os_4(CO)_{13}(PMe_3)$ in 86% yield (eqn 3.1).

$$Os_4(CO)_{14}(PMe_3) \xrightarrow{h\nu} Os_4(CO)_{13}(PMe_3) \qquad (3.1)$$

Treatment of a hexane solution of $Os_4(CO)_{13}(PMe_3)$ at room temperature with $P(OMe)_3$ caused the instantaneous conversion of $Os_4(CO)_{13}(PMe_3)$ to $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (eqn 3.2).

$$Os_4(CO)_{13}(PMe_3) + P(OMe)_3 \xrightarrow[\text{hexane}]{\text{RT}} Os_4(CO)_{13}(PMe_3)[P(OMe)_3] \qquad (3.2)$$

The product was isolated in 92% yield after chromatography and recrystallization. It forms dark red, air-stable crystals that are soluble in both hexane and dichloromethane at room temperature. As observed for $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$, dilute solutions of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ are green, while concentrated solutions are red.

A single crystal X-ray diffraction investigation revealed the structure of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ to be analogous to those of $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$, with the PMe_3 group bonded to one wingtip osmium atom and the $P(OMe)_3$ bonded to the other (Figure 3.1). Selected molecular dimensions of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ are collected in Table 3.1. The four osmium atoms are essentially planar; the dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(4)-Os(3) is 178.59 (6)°. The osmium-osmium bond lengths around the periphery of the cluster show a similar pattern of variation as found for $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$: Os(1)-Os(2) = 2.783 (3) Å, Os(1)-Os(4) = 2.792 (2) Å, Os(2)-Os(3) = 2.978 (2) Å, Os(3)-Os(4) = 3.019 (2) Å. The hinge bond, Os(1)-Os(3), has a length of 2.937 (2) Å.

The ^1H NMR spectrum of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (in $CDCl_3$) at room temperature consists of two doublets at δ 3.65 (J_{P-H} = 12 Hz) and 1.83 (J_{P-H} = 11 Hz). These are assigned to the hydrogen atoms of the methyl groups of the $P(OMe)_3$ and PMe_3 ligands, respectively, on the basis of the chemical shifts and the coupling constants. The room temperature ^{13}C NMR spectrum of

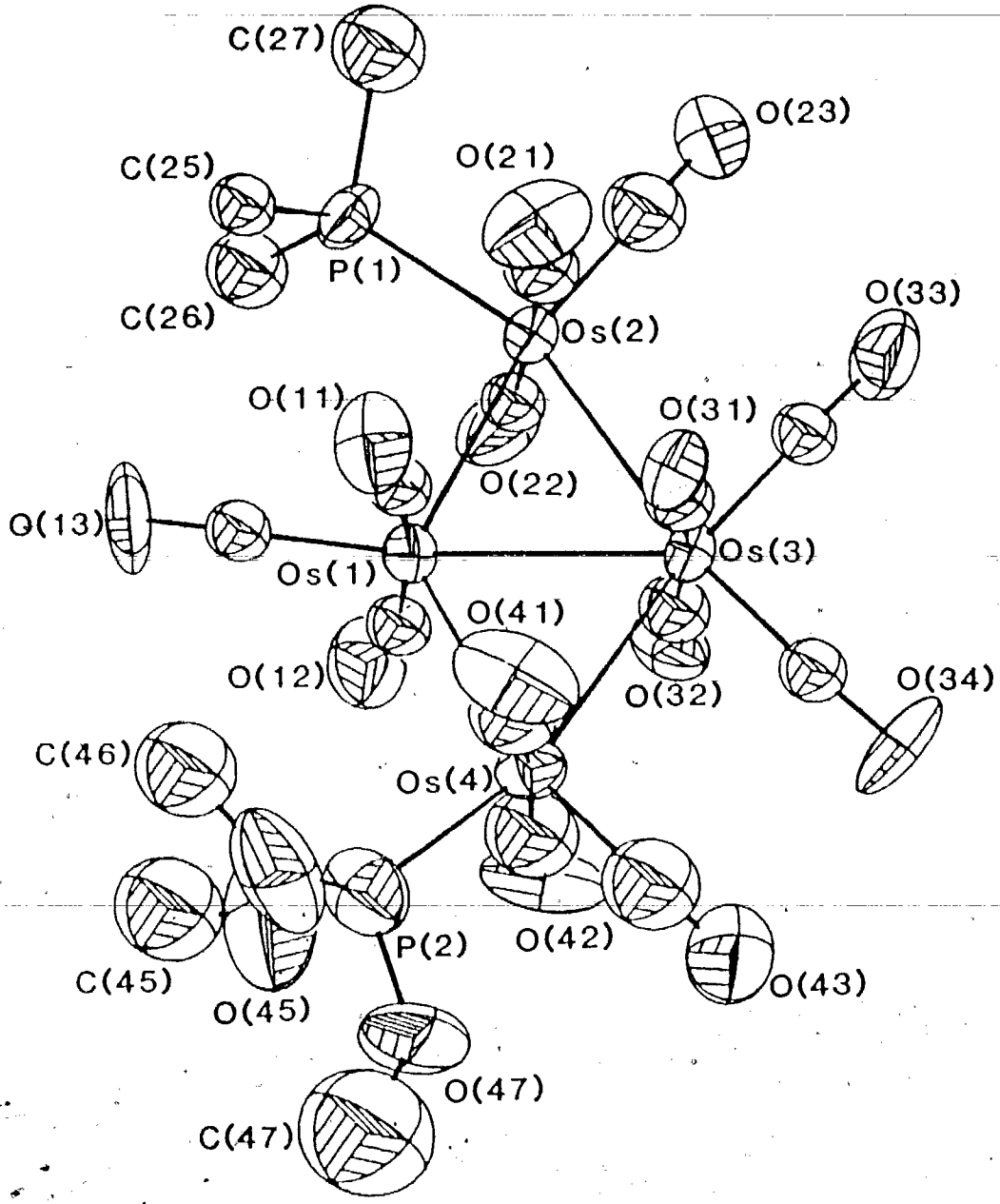


Figure 3.1. Molecular Structure of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Table 3.1. Selected Molecular Dimensions of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Bond Lengths (Å)

Os(1)-Os(2)	2.783 (3)	Os(1)-Os(3)	2.937 (2)
Os(1)-Os(4)	2.792 (2)	Os(2)-Os(3)	2.978 (2)
Os(3)-Os(4)	3.019 (2)	Os(1)-C(11)	1.89 (4)
Os(1)-C(12)	1.90 (4)	Os(1)-C(13)	1.83 (4)
Os(2)-P(1)	2.31 (1)	Os(2)-C(21)	1.90 (4)
Os(2)-C(22)	1.93 (4)	Os(2)-C(23)	1.86 (4)
Os(3)-C(31)	1.91 (4)	Os(3)-C(32)	1.88 (4)
Os(3)-C(33)	1.80 (5)	Os(3)-C(34)	1.85 (4)
Os(4)-P(2)	2.22 (1)	Os(4)-C(41)	1.79 (5)
Os(4)-C(42)	1.87 (5)	Os(4)-C(43)	1.88 (6)
C(11)-O(11)	1.22 (5)	C(12)-O(12)	1.17 (5)
C(13)-O(13)	1.19 (5)	C(21)-O(21)	1.18 (5)
C(22)-O(22)	1.12 (5)	C(23)-O(23)	1.16 (5)
C(31)-O(31)	1.15 (5)	C(32)-O(32)	1.19 (5)
C(33)-O(33)	1.23 (5)	C(34)-O(34)	1.22 (5)
C(41)-O(41)	1.23 (5)	C(42)-O(42)	1.15 (6)
C(43)-O(43)	1.13 (6)	P(1)-C(25)	1.84 (4)
P(1)-C(26)	1.82 (4)	P(1)-C(27)	1.86 (4)
P(2)-O(45)	1.62 (4)	P(2)-O(46)	1.54 (4)
P(2)-O(47)	1.67 (4)	O(45)-C(45)	1.19 (8)
O(46)-C(46)	1.46 (8)	O(47)-C(47)	1.29 (9)

$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (in CD_2Cl_2/CH_2Cl_2, 1/5) consists of a single resonance at δ 172.7, which indicates the molecule is fluxional. The spectrum obtained at -45 °C is consistent with the solid state structure of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (the assignment of the signals is discussed in Section 3.3). The ^{13}C NMR spectra obtained at temperatures above -45 °C indicate that

Table 3.1. Continued.

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(3)	62.69 (6)	Os(3)-Os(1)-Os(4)	63.54 (6)
Os(1)-Os(2)-Os(3)	61.20 (6)	Os(1)-Os(3)-Os(2)	56.11 (6)
Os(1)-Os(3)-Os(4)	55.89 (6)	Os(1)-Os(4)-Os(3)	60.57 (6)
Os(2)-Os(1)-C(11)	89 (1)	Os(2)-Os(1)-C(13)	110 (1)
C(11)-Os(1)-C(12)	172 (2)	C(11)-Os(1)-C(13)	88 (2)
Os(3)-Os(2)-C(23)	106 (1)	C(21)-Os(2)-C(22)	178 (2)
C(21)-Os(2)-C(23)	90 (2)	C(23)-Os(2)-P(1)	97 (1)
Os(4)-Os(3)-C(31)	85 (1)	Os(4)-Os(3)-C(34)	78 (1)
C(31)-Os(3)-C(32)	171 (2)	C(31)-Os(3)-C(34)	92 (2)
C(33)-Os(3)-C(34)	94 (2)	Os(1)-Os(4)-C(41)	89 (1)
Os(1)-Os(4)-P(2)	102.0 (4)	C(41)-Os(4)-C(42)	174 (2)
C(41)-Os(4)-P(2)	86 (1)	C(43)-Os(4)-P(2)	94 (1)

---

the carbonyls in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ undergo a different type of exchange process than those in $Os_4(CO)_{15}$ or $Os_4(CO)_{14}(PMe_3)$. Possible mechanisms for these rearrangements are discussed in Section 3.3.

$Os_4(CO)_{14}(CNBu^t)$

Dropwise addition of Me_3NO (in CH_2Cl_2) to a CH_2Cl_2 solution of $Os_4(CO)_{15}(CNBu^t)$ (Chapter 4) at room temperature resulted in the color of the solution changing from amber to dark green (eqn 3.3). The course of the reaction was followed by infrared spectroscopy, which showed the disappearance of bands due to $Os_4(CO)_{15}(CNBu^t)$ and the appearance of bands due to $Os_4(CO)_{14}(CNBu^t)$. The product was isolated from the reaction mixture in 62% yield as dark red, air-stable crystals.

$$Os_4(CO)_{15}(CNBu^t) \xrightarrow{Me_3NO} Os_4(CO)_{14}(CNBu^t) \qquad (3.3)$$

The structure of $Os_4(CO)_{14}(CNBu^t)$ has been determined by X-ray crystallography. A view of the molecule is shown in Figure 3.2. The overall structure consists of an almost planar Os_4 skeleton, with the $CNBu^t$ ligand in an axial position on the hinge osmium atom that has four terminal ligands (*i.e.*, Os(3)). The dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(4) is 178.35 (6)°. The now familiar pattern of short and long Os-Os bond lengths around the periphery of the cluster is observed. These are Os(1)-Os(2) = 2.793 (2) Å, Os(1)-Os(4) = 2.775 (2) Å, Os(3)-Os(2) = 2.985 (2) Å, and Os(3)-Os(4) = 2.983 (2) Å. The hinge bond, Os(1)-Os(3), has a length of 2.936 (2) Å (see Table 3.2 for other molecular dimensions of $Os_4(CO)_{14}(CNBu^t)$).

The ^1H NMR spectrum of $Os_4(CO)_{14}(CNBu^t)$ (in toluene-d_8, ambient temperature) consists of two resonances at δ 0.95 and 0.96. They are roughly the same intensity, but the higher field resonance is significantly broader than the other signal. The presence of two signals assignable to the hydrogen atoms of the $CNBu^t$ ligand of $Os_4(CO)_{14}(CNBu^t)$ indicates that the cluster exists as a mixture of (at least two) isomers in solution at room temperature. The ^{13}C NMR spectrum of $Os_4(CO)_{14}(CNBu^t)$ at -40 °C also consists of two signals of approximately equal intensity at δ 174.9 and 174.7.[67] This indicates that the carbonyls in the isomers of $Os_4(CO)_{14}(CNBu^t)$ are fluxional on

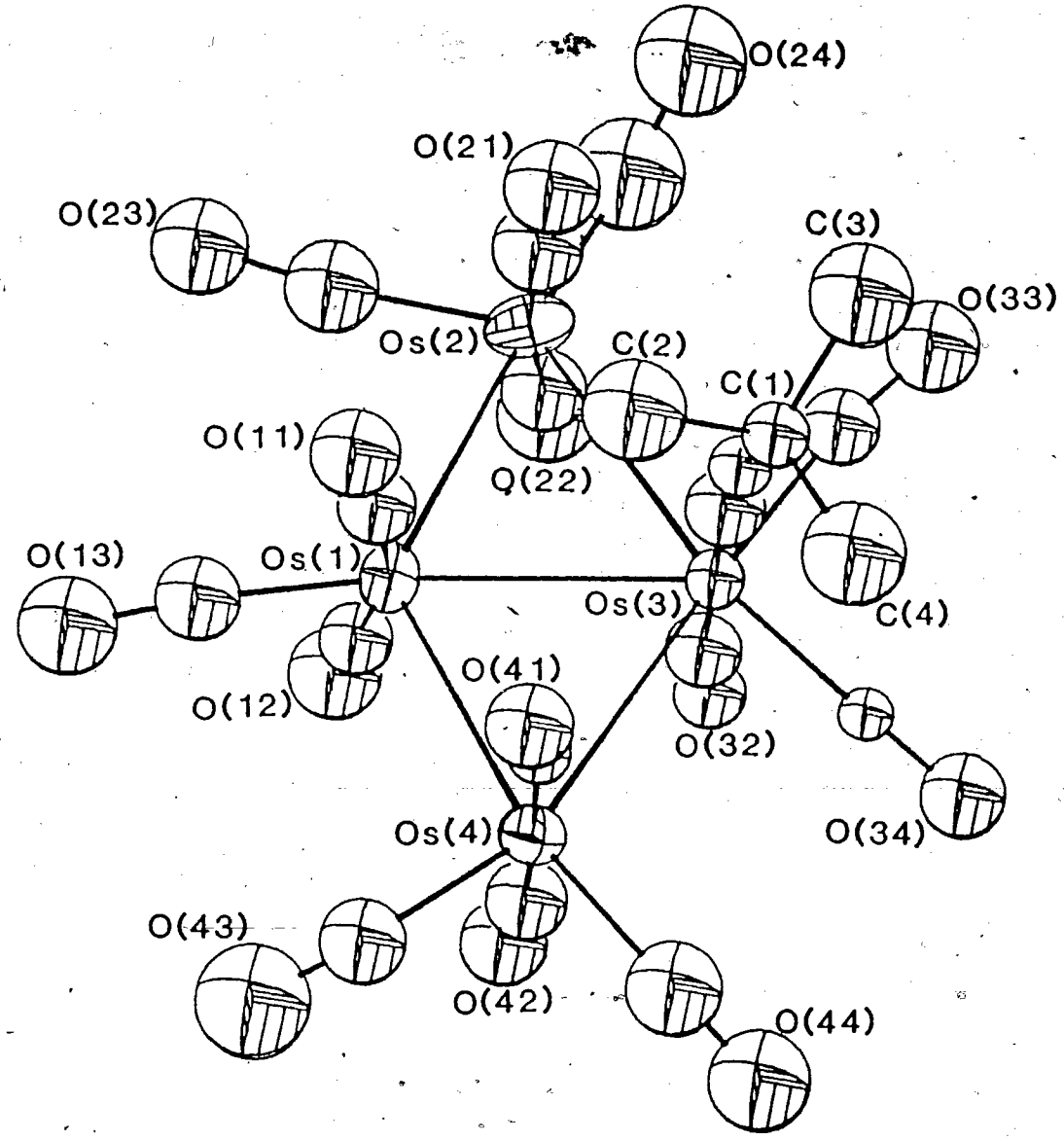


Figure 3.2. Molecular Structure of $Os_4(CO)_{14}(CNBu^t)$. Hydrogen atoms have been omitted for clarity.

Table 3.2. Selected Molecular Dimensions of $Os_4(CO)_{14}(CNBu^t)$.

Bond Lengths (Å)

Os(1)-Os(2)	2.793 (2)	Os(1)-Os(3)	2.936 (2)
Os(1)-Os(4)	2.775 (2)	Os(2)-Os(3)	2.985 (2)
Os(3)-Os(4)	2.983 (2)	Os(1)-C(11)	1.94 (4)
Os(1)-C(12)	1.88 (4)	Os(1)-C(13)	1.74 (4)
Os(2)-C(21)	1.94 (5)	Os(2)-C(22)	1.89 (5)
Os(2)-C(23)	1.83 (5)	Os(2)-C(24)	1.78 (6)
Os(3)-C(31)	2.01 (4)	Os(3)-C(32)	1.90 (4)
Os(3)-C(33)	1.90 (4)	Os(3)-C(34)	1.89 (3)
Os(4)-C(41)	1.88 (3)	Os(4)-C(42)	1.94 (4)
Os(4)-C(43)	1.88 (4)	Os(4)-C(44)	1.92 (4)
C(11)-O(11)	1.17 (4)	C(12)-O(12)	1.21 (4)
C(13)-O(13)	1.23 (4)	C(21)-O(21)	1.14 (4)
C(22)-O(22)	1.19 (5)	C(23)-O(23)	1.25 (5)
C(31)-N(31)	1.18 (4)	C(32)-O(32)	1.18 (4)
C(33)-O(33)	1.18 (4)	C(34)-O(34)	1.17 (4)
C(41)-O(41)	1.22 (4)	C(42)-O(42)	1.17 (4)
C(43)-O(43)	1.16 (5)	C(44)-O(44)	1.18 (5)
N(31)-C(1)	1.48 (4)	C(1)-C(2)	1.47 (5)
C(1)-C(3)	1.52 (5)	C(1)-C(4)	1.47 (5)

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(3)	62.76 (5)	Os(3)-Os(1)-Os(4)	62.91 (5)
Os(1)-Os(2)-Os(3)	60.97 (5)	Os(1)-Os(3)-Os(2)	56.27 (5)
Os(1)-Os(3)-Os(4)	55.90 (5)	Os(1)-Os(4)-Os(3)	61.19 (6)
Os(2)-Os(1)-C(11)	92 (1)	Os(2)-Os(1)-C(13)	125 (1)
C(11)-Os(1)-C(12)	167 (2)	C(11)-Os(1)-C(13)	86 (2)
Os(3)-Os(2)-C(24)	115 (2)	C(21)-Os(2)-C(22)	173 (2)
C(21)-Os(2)-C(24)	92 (2)	C(23)-Os(2)-C(24)	107 (2)
Os(4)-Os(3)-C(31)	92 (1)	Os(4)-Os(3)-C(34)	80 (1)
C(31)-Os(3)-C(32)	179 (2)	C(31)-Os(3)-C(34)	87 (2)
C(33)-Os(3)-C(34)	97 (2)	Os(1)-Os(4)-C(41)	87 (1)
Os(1)-Os(4)-C(43)	98 (1)	C(41)-Os(4)-C(42)	175 (2)
C(41)-Os(4)-C(43)	90 (2)	C(43)-Os(4)-C(44)	97 (2)

the NMR time scale. The nature of the CO-exchange (and the structure of the isomers) are currently being investigated by Mr. A. K. Ma of our laboratory. A detailed discussion must await the completion of these studies, but preliminary results suggest that the CO-exchange in $Os_4(CO)_{14}(CNBu^t)$ may be as rapid as that in $Os_4(CO)_{15}$.[124]

$(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$

Reaction of $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$[24] with $Os_3(CO)_{10}(COE)_2$[25] at room temperature in hexane gave $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ in about 60% yield after 3.5 h (eqn 3.4). Dark red, air-stable crystals of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ were obtained by recrystallization from hexane/toluene solution at -15 °C.

$$(\eta^5\text{-}C_5Me_5)Ir(CO)_2 + Os_3(CO)_{10}(COE)_2 \xrightarrow[\text{hexane}]{\text{RT}} (\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12} \qquad (3.4)$$

The structure of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ was determined by X-ray crystallography. The molecule adopts a "kite-like" arrangement of metal atoms, with an $(\eta^5\text{-}C_5Me_5)Ir(CO)$ group occupying a wingtip position (Figure 3.3). The lone carbonyl on the iridium atom is in an axial position. The metal framework of this 62-electron cluster is distorted slightly from planarity. The dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Ir is 169.9 (1)°. The bending is away from that side of the cluster with the four axial carbonyl groups and can

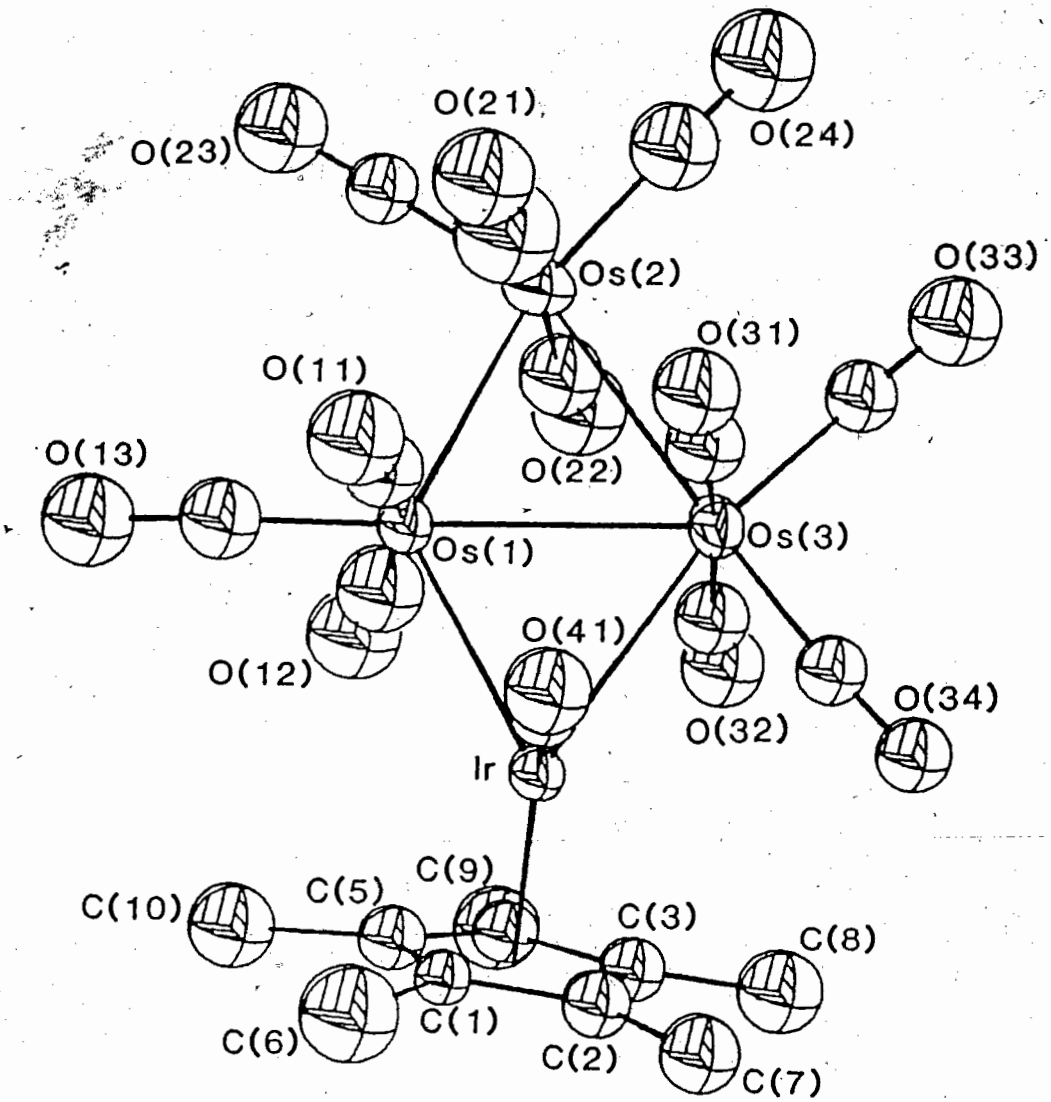


Figure 3.3. Molecular Structure of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$. Hydrogen atoms have been omitted for clarity.

Table 3.3. Selected Molecular Dimensions of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$.

Bond Lengths (Å)

Os(1)-Os(2)	2.794 (2)	Os(1)-Os(3)	2.908 (2)
Os(1)-Ir	2.703 (2)	Os(2)-Os(3)	2.994 (2)
Os(3)-Ir	2.939 (2)	Os(1)-C(11)	1.92 (3)
Os(1)-C(12)	1.90 (3)	Os(1)-C(13)	1.79 (3)
Os(2)-C(21)	1.86 (4)	Os(2)-C(22)	1.88 (3)
Os(2)-C(23)	1.87 (3)	Os(2)-C(24)	1.97 (4)
Os(3)-C(31)	2.01 (3)	Os(3)-C(32)	1.93 (3)
Os(3)-C(33)	1.93 (3)	Os(3)-C(34)	1.90 (3)
Ir-C(41)	1.86 (3)	C(11)-O(11)	1.15 (4)
C(12)-O(12)	1.16 (4)	C(13)-O(13)	1.19 (4)
C(21)-O(21)	1.20 (4)	C(22)-O(22)	1.19 (4)
C(23)-O(23)	1.17 (4)	C(24)-O(24)	1.15 (5)
C(31)-O(31)	1.07 (3)	C(32)-O(32)	1.15 (4)
C(33)-O(33)	1.13 (4)	C(34)-O(34)	1.14 (4)
C(41)-O(41)	1.12 (4)	Ir-C(1)	2.18 (3)
Ir-C(2)	2.28 (3)	Ir-C(3)	2.29 (3)
Ir-C(4)	2.32 (3)	Ir-C(5)	2.25 (3)
C(1)-C(6)	1.60 (5)	C(2)-C(7)	1.48 (4)
C(3)-C(8)	1.51 (4)	C(4)-C(9)	1.49 (4)
C(5)-C(10)	1.48 (4)	C(1)-C(2)	1.45 (4)
C(1)-C(5)	1.43 (4)	C(2)-C(3)	1.42 (4)
C(3)-C(4)	1.45 (4)	C(4)-C(5)	1.37 (4)

probably be attributed to the strain between these ligands (see Section 3.3).

The metal-metal bond lengths in $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ show a similar pattern of variation as found for the related tetraosmium derivatives: Os(1)-Os(2) = 2.794 (2) Å, Os(1)-Os(3)

Table 3.3. Continued.

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(3)	63.31 (4)	Os(3)-Os(1)-Ir	63.05 (4)
Os(1)-Os(2)-Os(3)	60.19 (4)	Os(1)-Os(3)-Os(2)	56.50 (4)
Os(1)-Os(3)-Ir	55.07 (4)	Os(1)-Ir-Os(3)	61.89 (4)
Os(2)-Os(1)-C(11)	101 (1)	Os(2)-Os(1)-C(13)	116 (1)
C(11)-Os(1)-C(12)	166 (1)	C(11)-Os(1)-C(13)	83 (1)
Os(3)-Os(2)-C(24)	104 (1)	C(21)-Os(2)-C(22)	170 (1)
C(21)-Os(2)-C(24)	97 (1)	C(23)-Os(2)-C(24)	97 (1)
Ir-Os(3)-C(31)	95 (1)	Ir-Os(3)-C(34)	71 (1)
C(31)-Os(3)-C(32)	174 (1)	C(31)-Os(3)-C(34)	88 (1)
C(33)-Os(3)-C(34)	99 (1)	Os(1)-Ir-C(41)	89 (1)
Os(1)-Os(4)-C(43)	98 (1)	C(41)-Os(4)-C(42)	175 (2)
C(41)-Os(4)-C(43)	90 (2)	C(43)-Os(4)-C(44)	97 (2)

= 2.908 (2) Å, Os(1)-Ir = 2.703 (2) Å, Os(3)-Os(2) = 2.994 (2) Å, and Os(3)-Ir = 2.939 (2) Å. The bonds between the osmium atoms and the iridium atom are slightly shorter than the analogous bonds in the tetraosmium clusters as a result of the smaller covalent radius of the iridium atom. The Os-Ir bond lengths in other cluster compounds reported in the literature range from 2.776 (5) Å to 2.881 (1) Å.[125]

The fluxional behavior of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ in solution has not been investigated.

$Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$[46]

X-ray quality crystals of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ were supplied by Mr. C.-Y. Lu. The compound was prepared by the reaction of $Os_4(CO)_{14}$ with Me_3SnH in hexane at room temperature. A view of the molecule is shown in Figure 3.4. The cluster adopts an almost planar arrangement in the solid that is similar to that found for $Os_4(CO)_{14}(PMe_3)$.[20] The Os-Os bond lengths in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ (Table 3.4) are unusual, but show a different pattern of variation than those in the other 62-electron clusters reported here. The peripheral Os-Os contacts are: Os(1)-Os(2) = 2.807 (1) Å, Os(1)-Os(4) = 2.810 (1) Å, Os(3)-Os(2) = 3.050 (1) Å, and Os(3)-Os(4) = 2.851 (1) Å. The hinge bond, Os(1)-Os(3), has a length of 2.875 (1) Å.

The hydride ligand was not located directly from the structure determination. Its position was calculated with the *HYDEX* program of Orpen.[126] The program clearly placed this ligand in a bridging position along the Os(1)-Os(2) bond, cis to the $SnMe_3$ group. This placement is consistent with the ^1H and ^{13}C NMR spectra of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ in solution.[46] In particular, irradiation of the signals due to the methyl hydrogen atoms of the $SnMe_3$ group led to a nuclear Overhauser enhancement of the signal due to the hydride. The magnitude of the enhancement corresponds to that expected if the distance between the nuclei is $\simeq$ 2.6 Å, in good agreement with the distance calculated using the coordinates given by the *HYDEX* program.

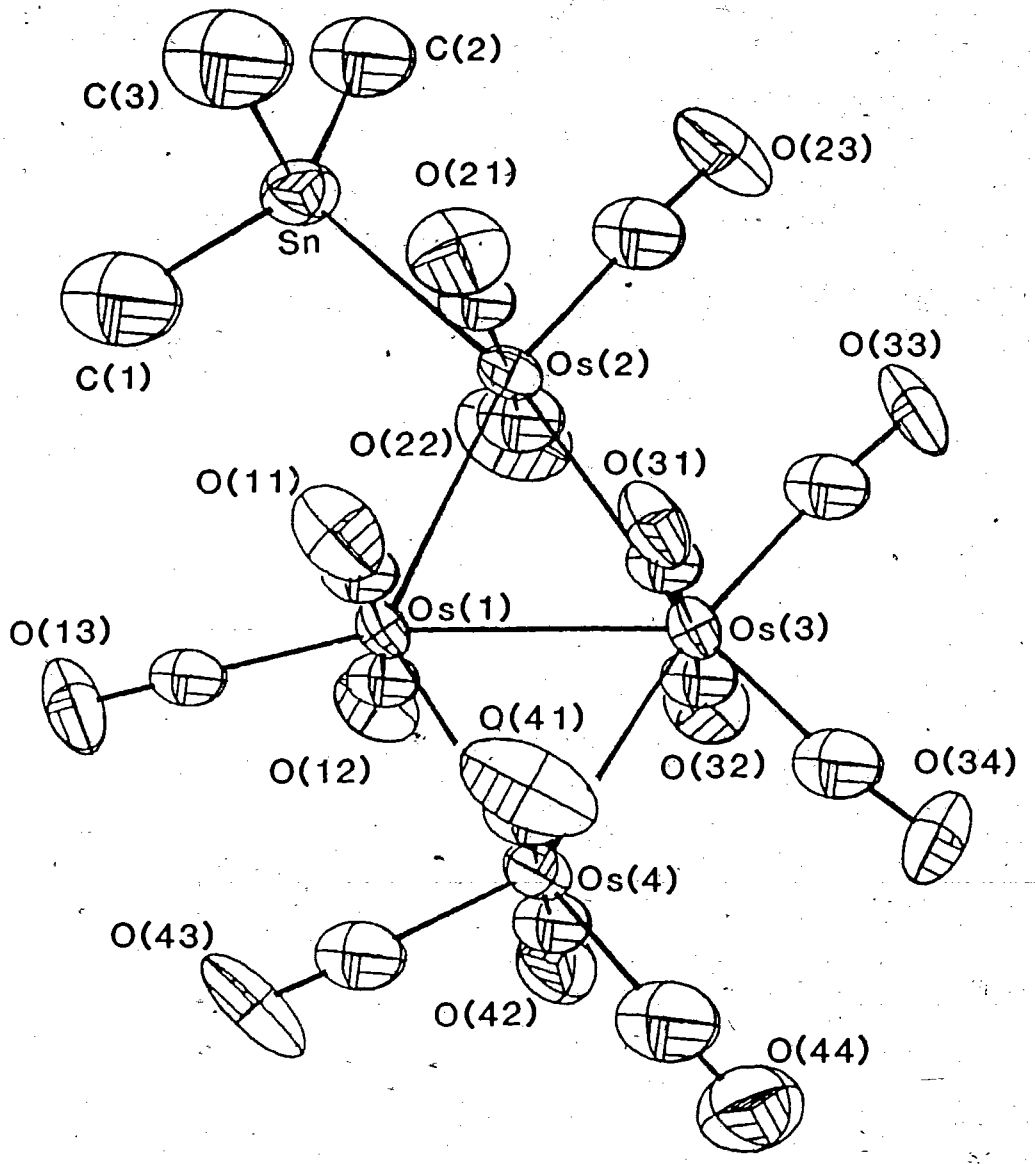


Figure 3.4.- Molecular Structure of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$. Hydrogen atoms have been omitted for clarity.

Table 3.4. Selected Molecular Dimensions of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

Bond Lengths (Å)

Os(1)-Os(2)	2.807 (1)	Os(1)-Os(3)	2.875 (1)
Os(1)-Os(4)	2.810 (1)	Os(2)-Os(3)	3.050 (1)
Os(3)-Os(4)	2.851 (1)	Os(1)-C(11)	1.82 (2)
Os(1)-C(12)	1.83 (2)	Os(1)-C(13)	1.85 (2)
Os(1)-H	1.82[a]	Os(2)-H	1.80[a]
Os(2)-Sn	2.697 (2)	Os(2)-C(21)	1.87 (2)
Os(2)-C(22)	1.86 (2)	Os(2)-C(23)	1.81 (2)
Os(3)-C(31)	1.89 (2)	Os(3)-C(32)	1.84 (2)
Os(3)-C(33)	1.84 (2)	Os(3)-C(34)	1.88 (2)
Os(4)-C(41)	1.86 (2)	Os(4)-C(42)	1.84 (2)
Os(4)-C(43)	1.84 (2)	Os(4)-C(44)	1.89 (3)
Sn-C(1)	2.00 (3)	Sn-C(2)	2.19 (3)
Sn-C(3)	2.04 (3)	C(11)-O(11)	1.13 (2)
C(12)-O(12)	1.07 (2)	C(13)-O(13)	1.10 (2)
C(21)-O(21)	1.06 (2)	C(22)-O(22)	1.07 (2)
C(23)-O(23)	1.11 (2)	C(31)-O(31)	1.05 (2)
C(32)-O(32)	1.10 (2)	C(33)-O(33)	1.10 (2)
C(34)-O(34)	1.18 (2)	C(41)-O(41)	1.11 (2)
C(42)-O(42)	1.07 (2)	C(43)-O(43)	1.10 (2)
C(44)-O(44)	1.14 (3)		

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(3)	64.93 (3)	Os(2)-Os(1)-Os(4)	125.12 (3)
Os(1)-Os(2)-Os(3)	58.62 (2)	Os(2)-Os(3)-Os(1)	56.45 (2)
Os(2)-Os(3)-Os(4)	115.21 (3)	Os(3)-Os(4)-Os(1)	61.04 (2)
Os(2)-Os(1)-C(13)	130.2 (6)	C(11)-Os(1)-C(12)	173 (1)
Os(1)-Os(2)-Sn	108.86 (4)	C(21)-Os(2)-C(22)	170 (1)
Os(2)-Os(3)-C(33)	75.6 (6)	C(33)-Os(3)-C(34)	94 (1)
C(31)-Os(3)-C(32)	172 (1)	Os(3)-Os(4)-C(44)	109.6 (7)
C(44)-Os(4)-C(43)	102 (1)	C(41)-Os(4)-C(42)	177 (1)
Os(1)-H-Os(2)	102[a]	Os(2)-Sn-C(1)	111 (1)
Os(2)-Sn-C(2)	113 (1)	Os(2)-Sn-C(3)	115 (1)

[a]Hydride is in a calculated position.

3.3 Discussion

Structural Considerations

As noted above the clusters $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, $Os_4(CO)_{14}(CNBu^t)$, $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$, (Figures 3.1, 3.2, and 3.3, respectively) and the previously known $Os_4(CO)_{14}(PMe_3)$[20] (Figure 3.5) adopt planar stuctures similar to that of $Os_4(CO)_{15}$ (Figure 3.6).

Until this study, planar 62-electron tetranuclear clusters that do not contain bridging ligands were rare. There were only three reported examples of such compounds, namely $Os_4(CO)_{14}(PMe_3)$,[20] $[Re_4(CO)_{16}]^{2-}$,[60] and $HOs_3Re(CO)_{15}$.[127] Planar 62-electron clusters that contain bridging ligands are more common. Two examples of this type of cluster are $Os_4(\mu\text{-}S)(CO)_{13}$[81] and the recently reported $Os_4(CO)_{12}(O_2CCF_3)_2$.[56] It has been suggested that 62-electron clusters that do not contain bridging ligands adopt a planar geometry when one or both of the hinge metal atoms has four terminal ligands.[129] The results of this study are consistent with this conclusion. That $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ deviates slightly from planarity is probably a result of the unbalanced steric interactions between the axial ligands (see below). A possible reason why the 62-electron clusters that have an $Os(CO)_4$ group in a hinge position are planar involves the frontier orbitals of this fragment. In C_{2v} $Os(CO)_4$, these orbitals are well orientated for effective bonding in a planar cluster, while in the C_{4v} geometry

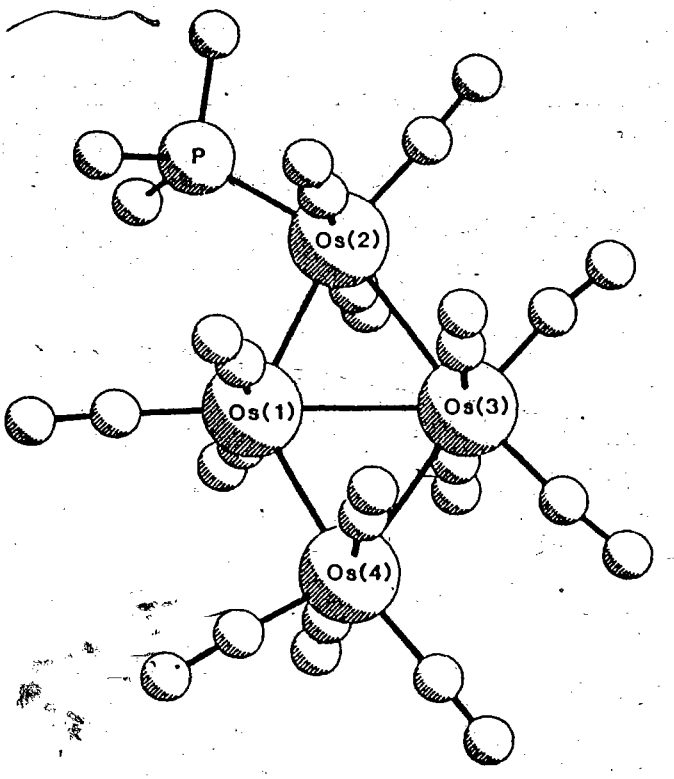


Figure 3.5. Molecular Structure of $Os_4(CO)_{14}(PMe_3)$.

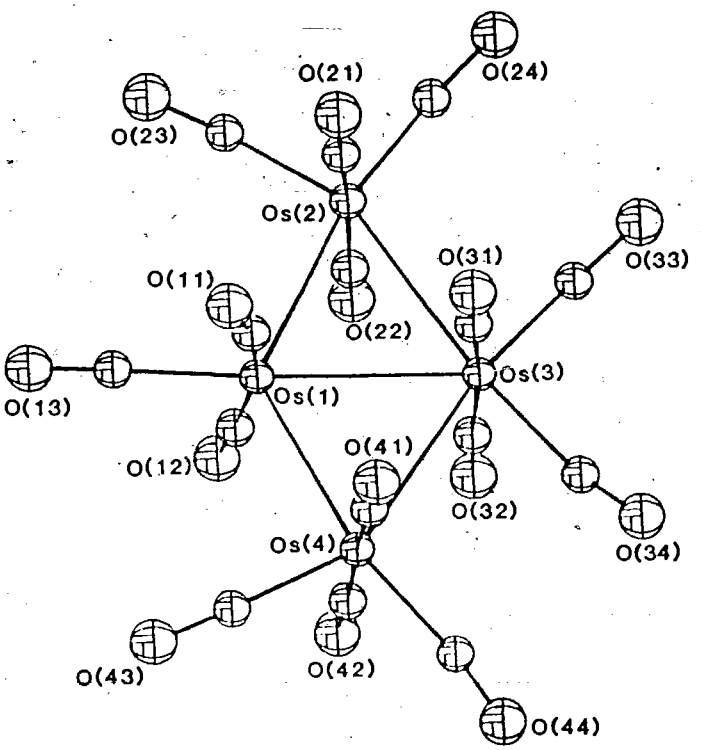


Figure 3.6. Molecular Structure of $Os_4(CO)_{15}$.

a bent structure is favored.[92] Evans and Mingos have estimated the latter geometry to be ≃ 2.6 eV higher in energy than the former,[58] and thus there may be a thermodynamic preference for the planar configuration.

Table 3.5 lists the Os-Os (and Os-Ir) bond lengths in the five 62-electron "kite" clusters. As can be seen, the structurally equivalent bonds are all at least chemically equivalent in length, except for those in $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ which involve the iridium atom. That these bonds are shorter than the coresponding Os-Os bonds is due to the smaller covalent radius of the iridium atom. The consistency of the bond lengths in Table 3.5 suggests that the pattern of short and long Os-Os (or Os-Ir) bond lengths in these clusters is an intrinsic property of the metal unit, and cannot be due to the trans influences of the ligands or to crystal packing forces. It also suggests that the bonding in these derivatives is similar. Therefore, the models discussed in Chapter 2 for the bonding in $Os_4(CO)_{15}$ also apply to these clusters. A few structural features of each of the new derivatives are discussed below.

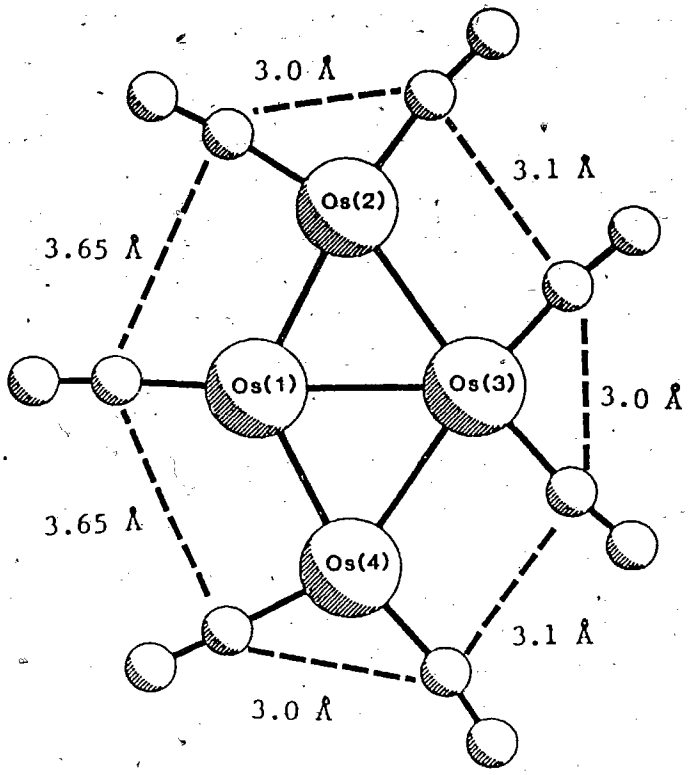
First, the site preference of the PMe_3 and $P(OMe)_3$ ligands in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (and the PMe_3 ligand in $Os_4(CO)_{14}(PMe_3)$[20]) probably results from steric considerations. This is illustrated in Scheme 3.1, which shows the Os_4 skeletons and equatorial ligands of $Os_4(CO)_{15}$ and $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$. As can be seen the phosphorus ligands in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ replace the carbonyls on the

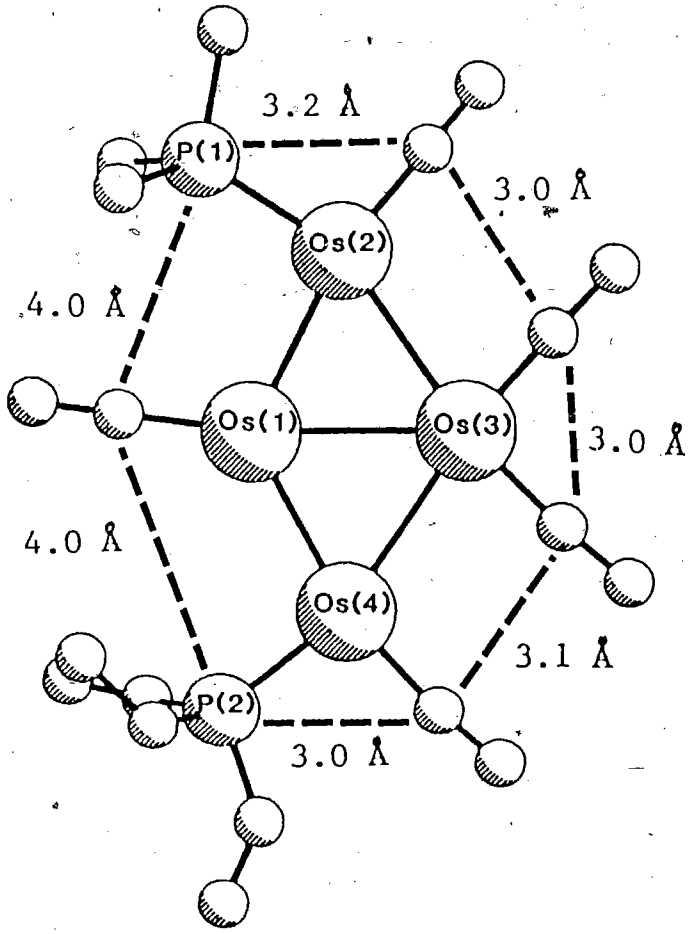
Table 3.5. Osmium-Osmium and Osmium-Iridium Bond Lengths in $Os_4(CO)_{15}$ and Some of Its Derivatives.

Compound	Os(1)-Os(2)	Os(1)-Os(3)	Os(1)-Os(4)	Os(2)-Os(3)	Os(3)-Os(4)
$Os_4(CO)_{15}$[a]	2.774	2.948	2.775	2.998	2.998
$Os_4(CO)_{14}(PMe_3)$[b]	2.779 (2)	2.935 (2)	2.784 (2)	3.013 (2)	2.982 (2)
$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$	2.783 (3)	2.937 (2)	2.792 (2)	2.978 (2)	3.019 (2)
$Os_4(CO)_{14}(CNBu^t)$	2.793 (2)	2.936 (2)	2.775 (2)	2.985 (2)	2.983 (2)
$(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$[c]	2.794 (2)	2.908 (2)	2.703 (2)	2.994 (2)	2.939 (2)

[a]Bond length restraints were applied. [b]Reference 20. [c]Ir ≡ Os(4).

Scheme 3.1


3.0 Å
3.1 Å
Os(2)
3.65 Å
Os(1)
Os(3)
3.0 Å
3.65 Å
Os(4)
3.1 Å
3.0 Å


3.2 Å
P(1)
3.0 Å
Os(2)
4.0 Å
Os(1)
Os(3)
3.0 Å
4.0 Å
Os(4)
3.1 Å
P(2)
3.0 Å

wingtips of $Os_4(CO)_{15}$ that are in the most open environments. It is also readily apparent from this scheme that the disposition of the equatorial carbonyls along the Os(2)-Os(3)-Os(4) edge of the cluster is not affected by the presence of the bulky phosphorus ligands (in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$).

In $Os_4(CO)_{14}(CNBu^t)$, the $CNBu^t$ group takes up an axial site on Os(3) (the hinge osmium atom with four terminal ligands, see Figure 3.2). The preference of the $CNBu^t$ group for an axial position in trinuclear osmium clusters is known,[130] and the same factors are probably involved in $Os_4(CO)_{14}(CNBu^t)$. That this ligand, which is a better σ-donor and poorer π-acceptor than CO, is bonded to Os(3) in the crystal stucture of $Os_4(CO)_{14}(CNBu^t)$ is consistent with the models discussed in Chapter 2 for the bonding in $Os_4(CO)_{15}$ (both models predict this atom will be slightly electron deficient). The phosphorus ligands, as mentioned, are probably prevented from binding to Os(3) in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ and $Os_4(CO)_{14}(PMe_3)$ for steric reasons. However, $Os_4(CO)_{14}(CNBu^t)$ exists as a mixture of isomers in solution and it is possible that the $CNBu^t$ group is bonded to one of the other osmium atoms in at least one of the isomers.[67] If this is the case, then there may not be a large electronic preference for this group to be bonded to Os(3).

The structure of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ is derived from that of $Os_4(CO)_{15}$ by replacing one wingtip $Os(CO)_4$ group with the isolobal fragment $(\eta^5\text{-}C_5Me_5)Ir(CO)$.[57] The lone carbonyl on the iridium atom is in an axial position while the C_5Me_5 group

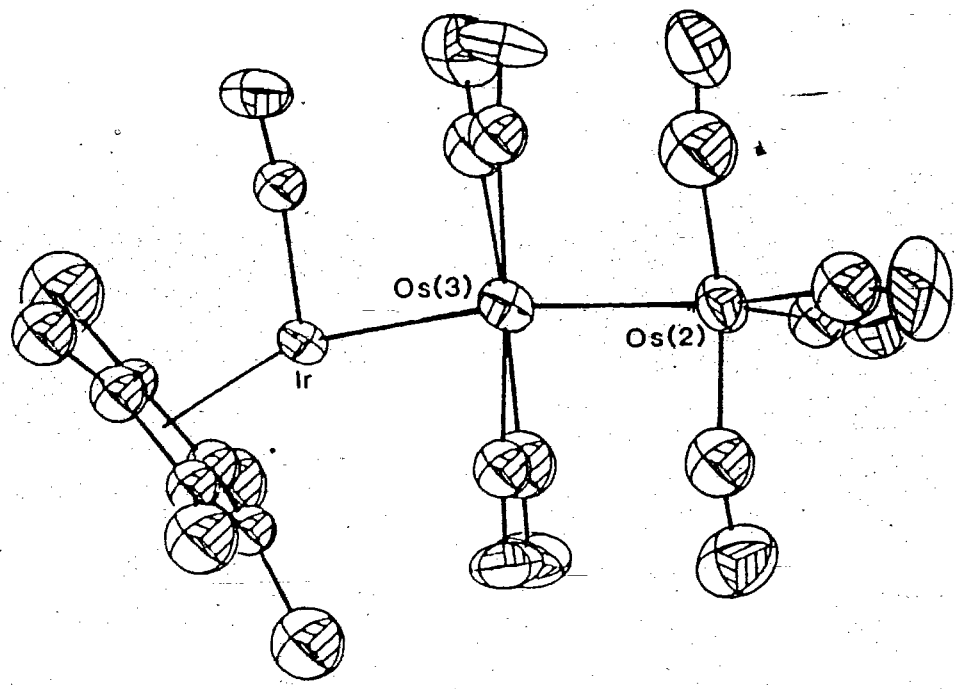


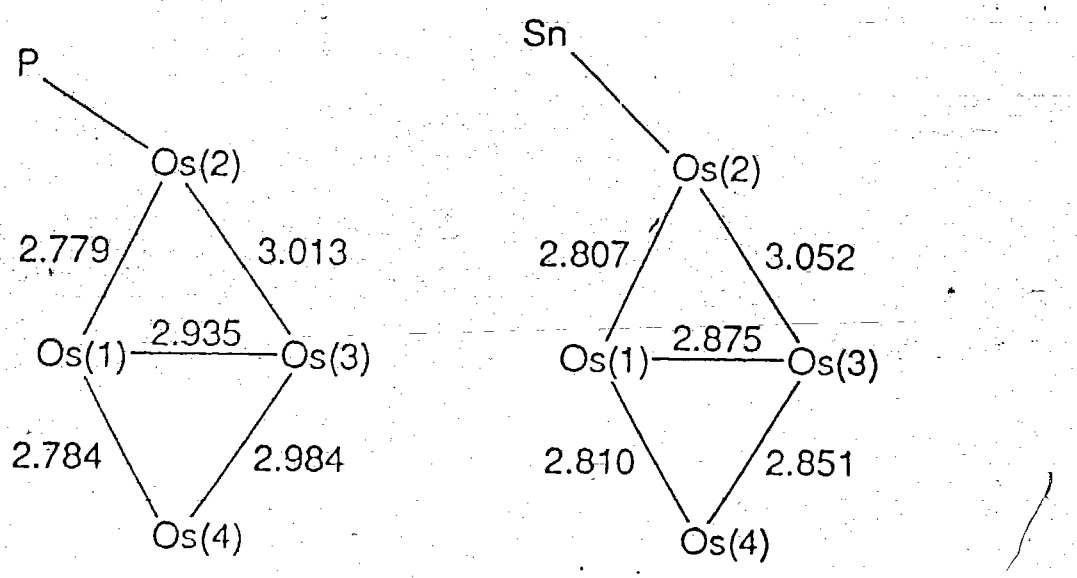
Figure 3.7. Structure of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ Viewed Down the Os(3)-Os(1) Bond Showing the Bending of the $IrOs_3$ Core. Hydrogen atoms and the equatorial carbonyl ligands on Os(1) and Os(3) have been omitted for clarity.

occupies the other axial site and both equatorial positions. Thus there are four axial carbonyls on one side of the cluster and only three on the other. The $IrOs_3$ core is slightly bent, with a dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Ir of $\simeq 170°$. A view of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ that shows the bending of the Os_3Ir framework more clearly is given in Figure 3.7. As can be seen, the bending is away from that side of the cluster with the four axial carbonyl ligands, and it is concluded that the deviation from planarity results from the

unbalanced interactions between the axial carbonyls. (Note that in each of the other kite-shaped clusters discussed here there are four axial ligands on each side of the plane of the metal atoms.) Incidentally, this is also consistent with the view that the structures of $Os_4(CO)_{15}$ and its derivatives are strained. It is also noteworthy that despite replacing the $Os(CO)_4$ fragment with the sterically very different $Ir(CO)(\eta^5\text{-}C_5Me_5)$ group and the consequent bending of the cluster, the metal skeleton retains the characteristic pattern of short and long peripheral bonds. This supports the conclusion that the unusual bond lengths in these clusters result from the electronic requirements of the metal core.

The structure of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ is shown in Figure 3.4. As can be seen, the gross features of this cluster are similar to those of the other 62-electron clusters described in this chapter. The Os_4 core of this 62-electron cluster is essentially planar; the dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(4) is 179.73 (3)°, with the tin atom 0.150 (2) Å from the best plane through the four osmium atoms. However, although the pattern of variation Os-Os bond lengths is unusual, it is different from that observed in the other derivatives discussed here (Scheme 3.2; since this hydrido cluster most closely resembles $Os_4(CO)_{14}(PMe_3)$ the dimensions of this compound[20] are also included in the scheme). The hydride ligand was not located in the crystallographic investigation. Given the lengths of the Os-Os bonds in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$,

Scheme 3.2



and that bridging hydrides are known to lengthen the bonds they are associated with,[131] it might be expected that this ligand bridges the Os(2)-Os(3) bond. However, because this bond is only slightly longer ($\simeq$ 0.04 Å) than the corresponding bonds in the $Os_4(CO)_{14}(L)$ derivatives, the hydride could not confidently be placed along this bond. Therefore, the *HYDEX* program written by Orpen was used to place the hydride ligand.[126] The program unequivocally preferred the position bridging the Os(1)-Os(2) bond (see Table 3.6). This bond has a length of 2.807 Å and is the second shortest Os-Os bond in the cluster. That this is the correct placement of the hydride ligand is graphically illustrated in Figure 3.8, which shows a superposition of the atoms in the equatorial planes of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ and $Os_4(CO)_{14}(PMe_3)$. Further support for this placement is found in the ^1H and ^{13}C NMR spectra of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ (described elsewhere[46]).

Table 3.6. Calculated Site Energies for the Hydride Ligand in Various Positions in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

Position	Site Energy[a]
Bridging	
Os(1)-Os(2)	2.4
Os(2)-Os(3)	29.9
Os(1)-Os(3)	45.5
Os(1)-Os(4)	33.9
Os(3)-Os(4)	53.8
terminal	
Os(1)	11.5
Os(2)	21.8

[a]Calculated with the *HYDEX* program; true hydride positions have been found to have site energies in the range 0.2 - 10.6, with an average value of 2.76, see ref. 126.

Perhaps the most interesting result of the substitution of the two-electron donor PMe_3 ligand in $Os_4(CO)_{14}(PMe_3)$ with $SnMe_3$ and a bridging hydride is the effect on the Os-Os bond lengths in the Os(1)-Os(3)-Os(4) triangle. Scheme 3.3 shows the relevant interatomic distances in the equatorial planes in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ and $Os_4(CO)_{14}(PMe_3)$. As can be seen, the Os-Os bond lengths in the Os_3 triangle not associated with the hydride bridge in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ are more nearly equal than those in $Os_4(CO)_{14}(PMe_3)$. This observation led the formulation of the bonding model shown in Scheme 3.4 for the bonding in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$. This model allows (as far as possible) each atom to achieve an 18-electron configuration. The

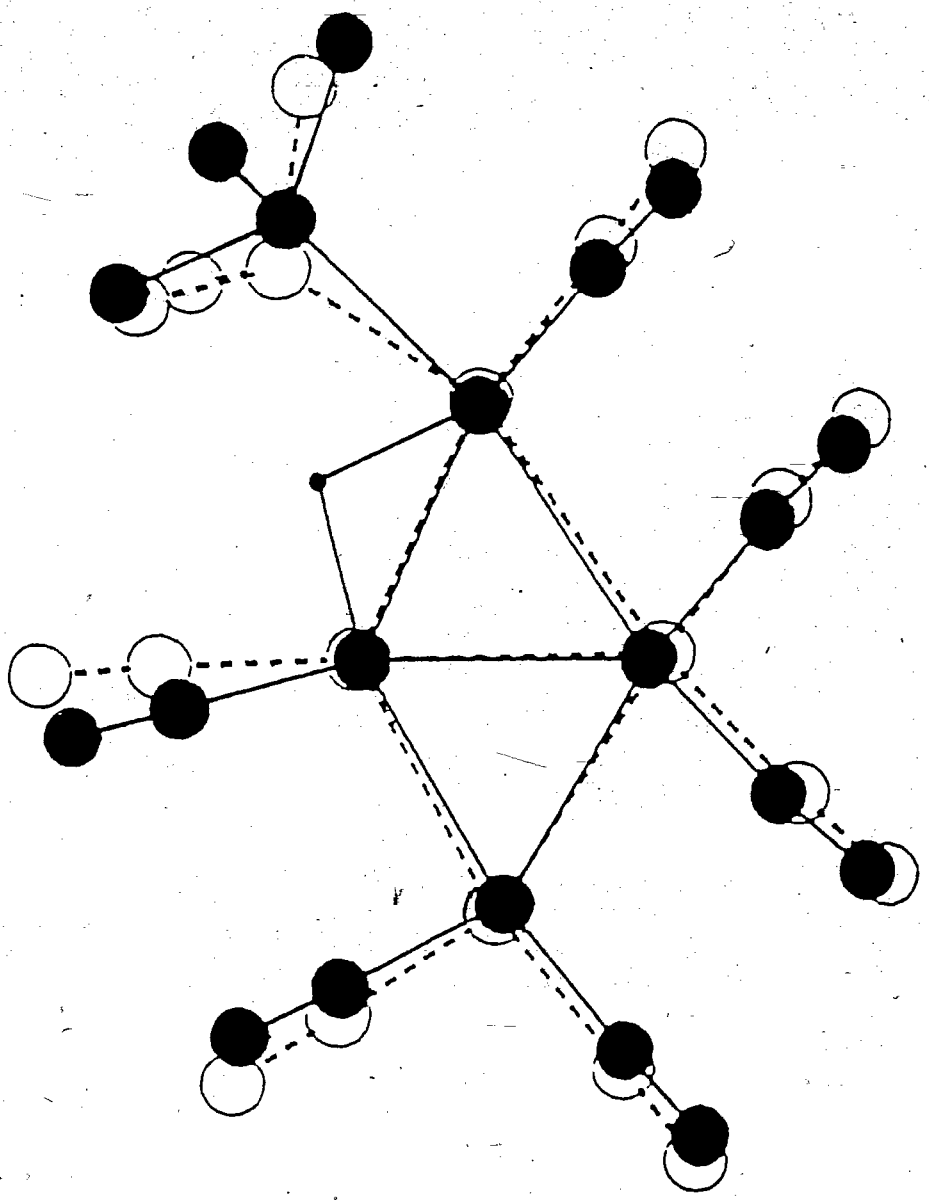
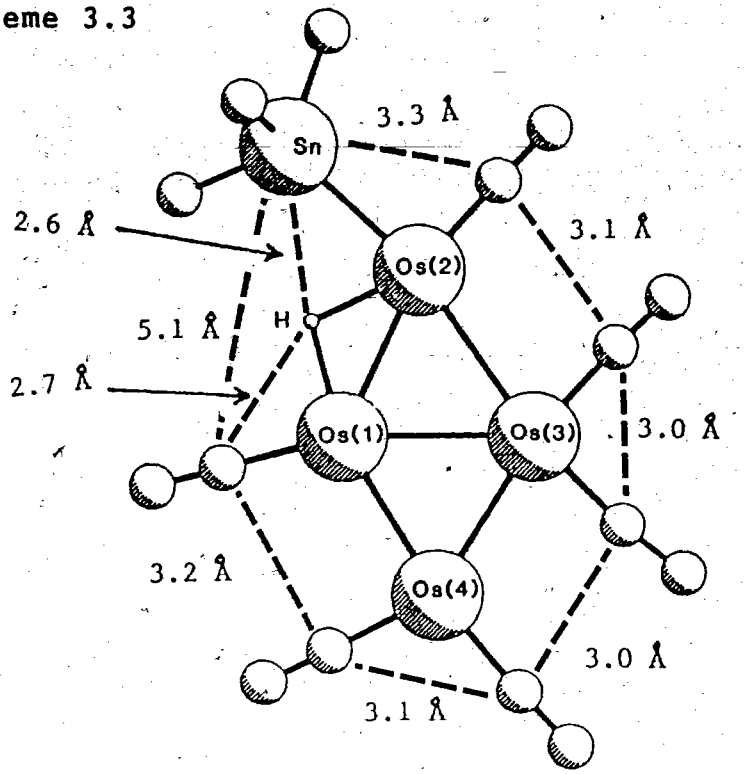
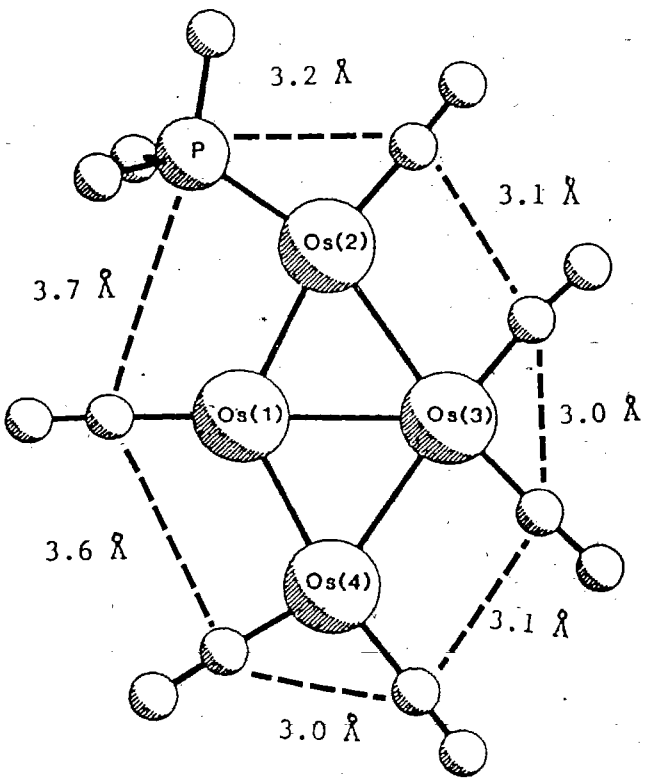


Figure 3.8. A Superposition of the Atoms in the Equatorial Planes of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ (Black Circles) and $Os_4(CO)_{14}(PMe_3)$ (White Circles).

Scheme 3.3

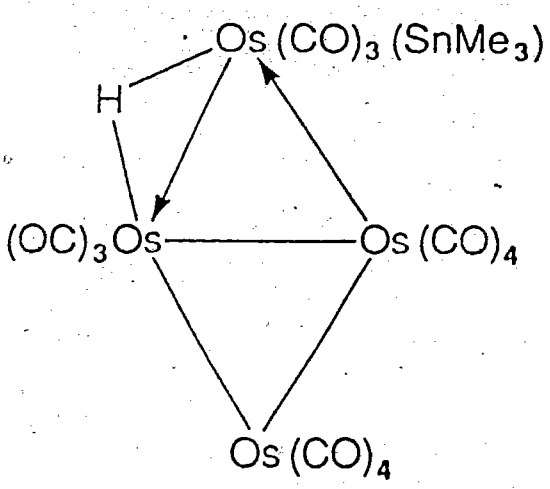


Os(1)-Os(2) = 2.81 Å
Os(1)-Os(3) = 2.88 Å
Os(1)-Os(4) = 2.81 Å
Os(2)-Os(3) = 3.05 Å
Os(3)-Os(4) = 2.85 Å



Os(1)-Os(2) = 2.78 Å
Os(1)-Os(3) = 2.94 Å
Os(1)-Os(4) = 2.78 Å
Os(2)-Os(3) = 3.01 Å
Os(3)-Os(4) = 2.98 Å

Scheme 3.4



model is also consistent with some of the spectroscopic properties of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ in solution.[86] A possible explanation for the relative Os-Os bond lengths in $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ may be found by considering the interactions between the equatorial ligands as shown in Scheme 3.3. The introduction of the hydride along the Os(1)-Os(2) bond results in a lengthening of this bond and also a lenthening of the Os(1)-Os(4) bond. The latter may be due to the trans influence of the hydride which is known to be large,[132] and is accompanied by the shortening of the Os(3)-Os(4) bond. The concerted lengthening of one bond and shortening of another has been proposed before in triangulated cluster compounds.[133] In addition, the lone equatorial carbonyl on Os(1) is displaced towards Os(4). From Scheme 3.3 it can be seen that Os(4) and its

ligands pivot in such a way (clockwise in the scheme) that the interactions between the ligands are roughly balanced along the Os(1)-Os(4) and Os(3)-Os(4) edges of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

In the Os(1)-Os(2)-Os(3) half of the molecule the wingtip group is prevented from pivoting by the presence of the hydride along the Os(1)-Os(2) bond, and the lengthening of the Os(1)-Os(2) bond (caused by the hydride bridge[131]) is now accompanied by a slight lengthening of the Os(2)-Os(3) bond. In this case the interactions between the adjacent equatorial carbonyls on Os(2) and Os(3) may prevent the shortening of the Os(2)-Os(3) bond.

Stereochemical Nonrigidity in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

The fluxional behavior of $Os_4(CO)_{15}$ was described in Chapter 2, and that of $Os_4(CO)_{14}(PMe_3)$ has been described elsewhere.[20] However, a quick review of the salient features is helpful at this point. Only three signals are observed in the CO region of the ^{13}C NMR spectra of $Os_4(CO)_{15}$ from 0 °C down to -120 °C (Figure 3.9). The two resonances (4C each) downfield of δ 180 are assigned to the axial carbonyls, and the highest field resonance at δ 171.7 (7C) is assigned to the seven equatorial carbonyls. The spectrum at -55 °C is interpreted in terms of an all-equatorial, merry-go-round CO-exchange process as shown in Scheme 3.5. The rate constant for this process was estimated to be on the order of 10^7 s^{-1} at -50 °C. At 0 °C there is evidence for the onset of a second exchange process, which may be a

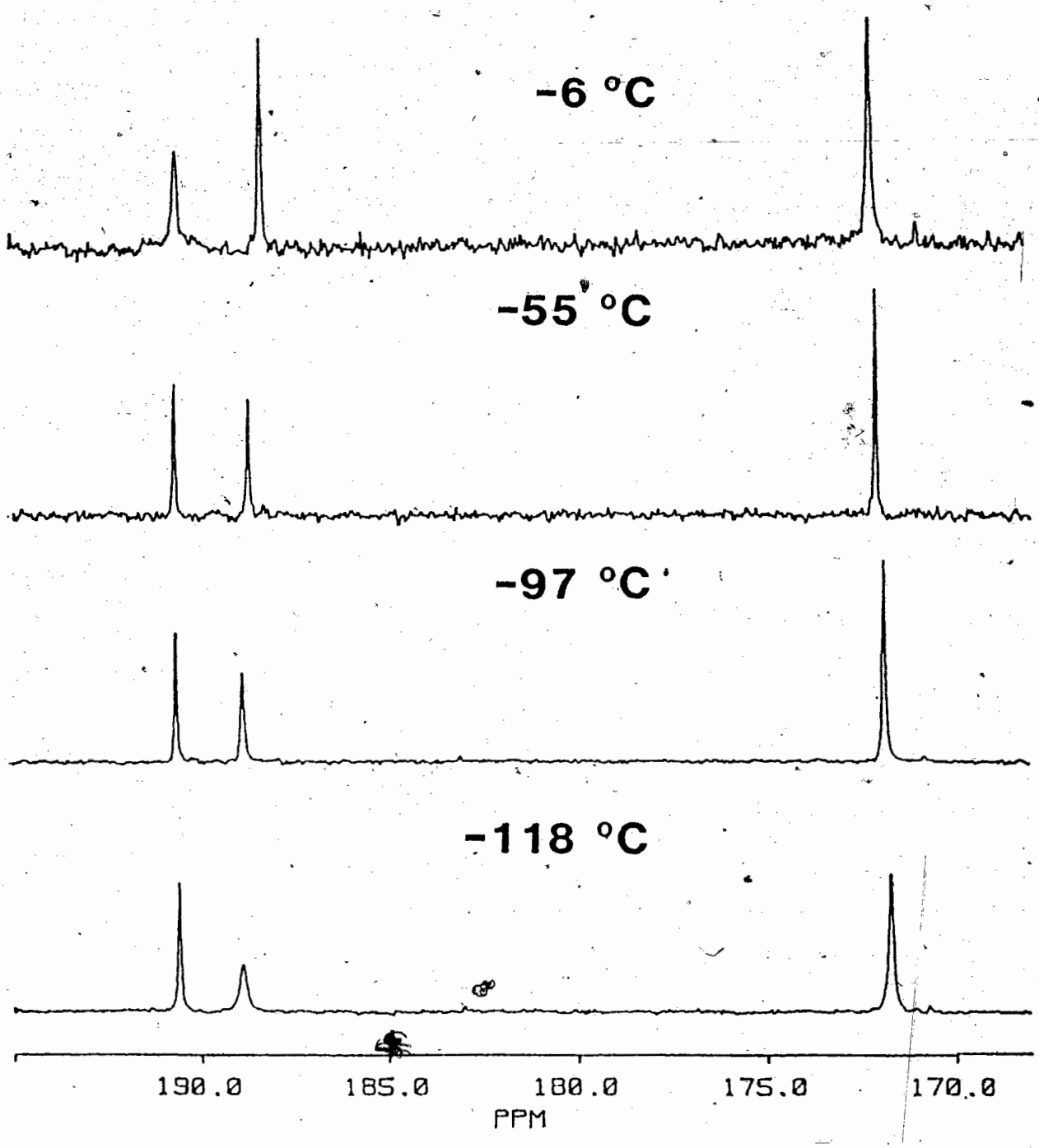
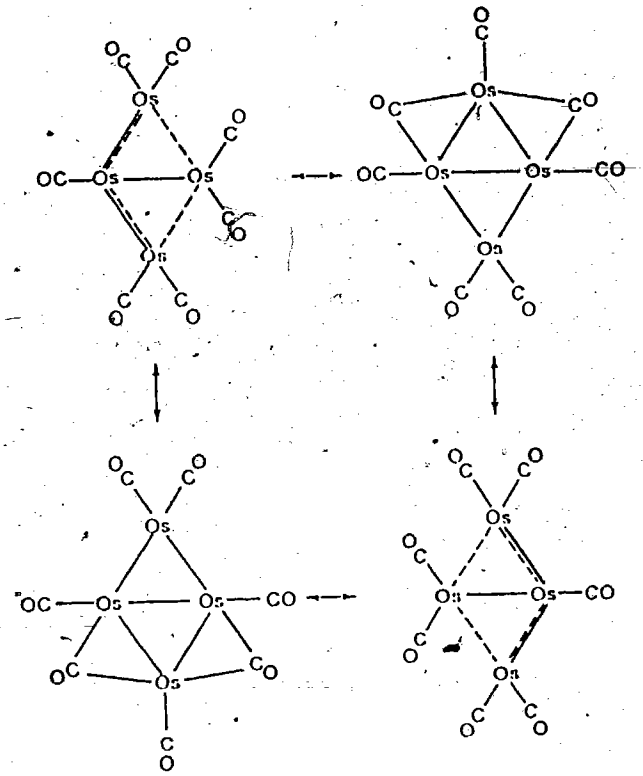
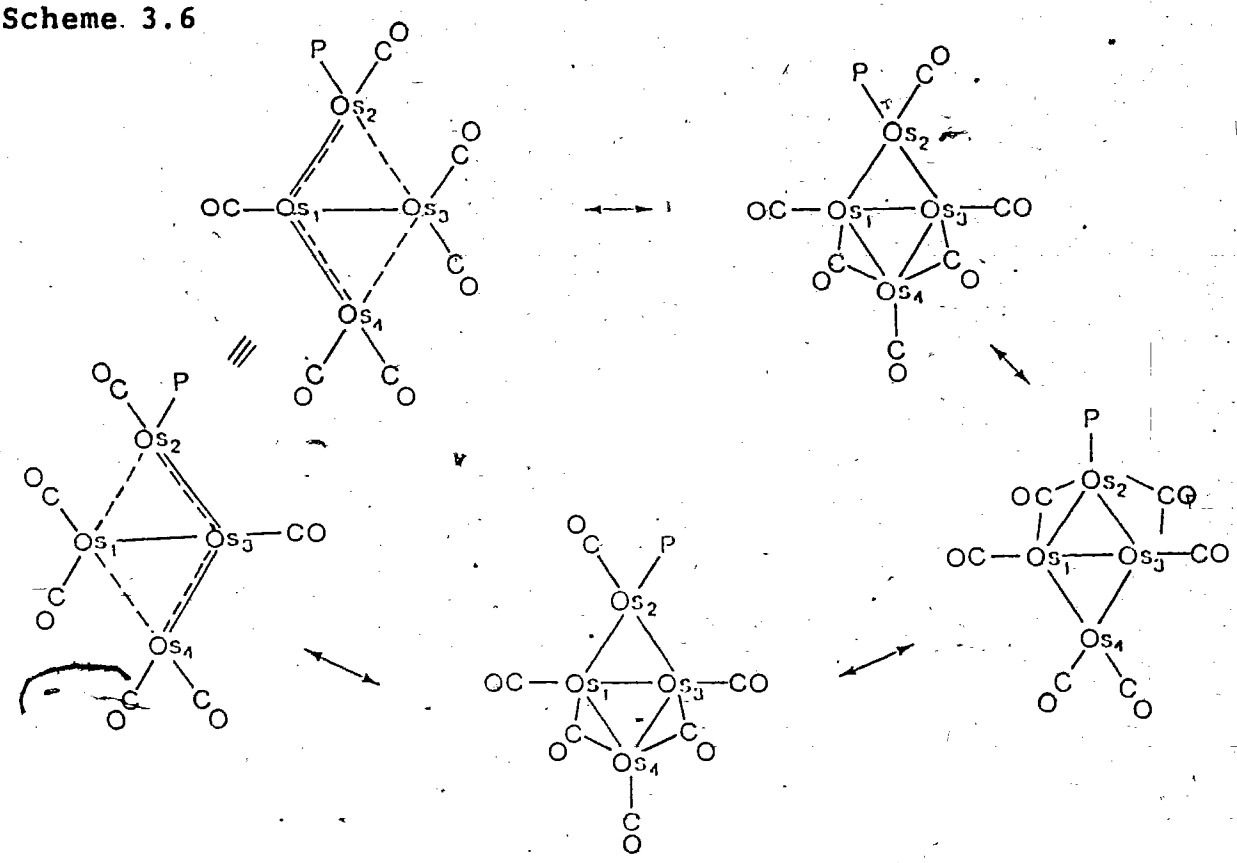


Figure 3.9. Variable Temperature ^{13}C NMR Spectra of $Os_4(CO)_{15}$.

Scheme 3.5



Scheme 3.6

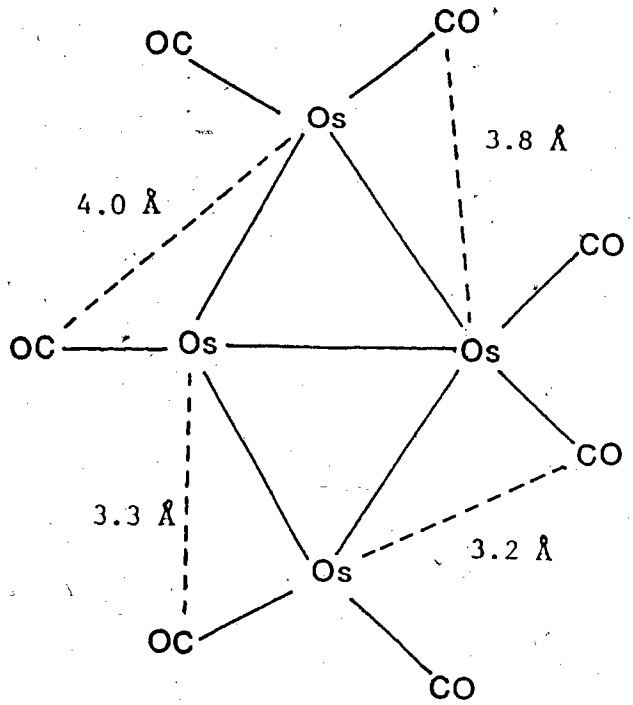


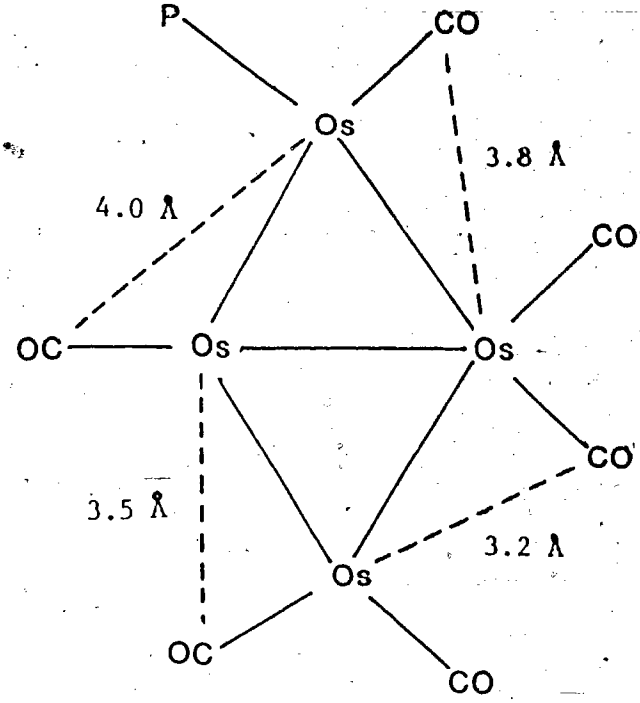
three-fold twist mechanism involving the equatorial carbonyls and axial carbonyls on the wingtip osmium atoms.

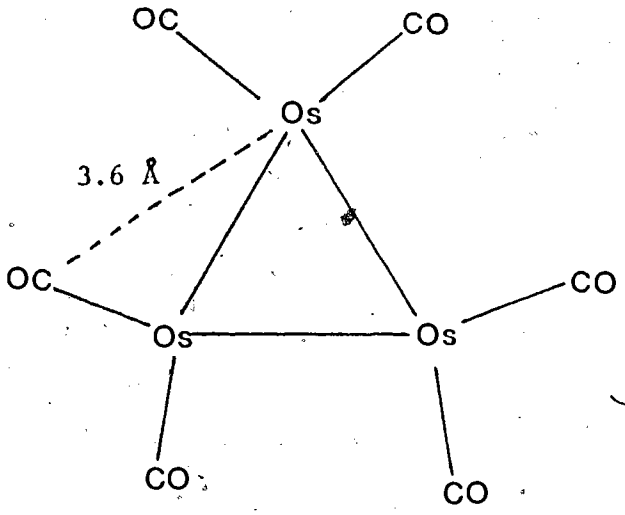
The nonrigidity in $Os_4(CO)_{14}(PMe_3)$ has been fully investigated. Two mechanisms were proposed to account for the observed variable temperature ^{13}C NMR spectra of this cluster. One of these also involves an all-equatorial, merry-go-round CO-exchange process (Scheme 4 in reference 20). A slightly modified version of this process is shown in Scheme 3.6; it involves some of the same intermediates, but it differs from the original in that the first step is now broken down into two steps. It has the advantage that all the intermediates with bridging carbonyls proposed for the nonrigidity in $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$ are now similar in that two carbonyls bridge edges of the same triangle. Another advantage of the new mechanism is discussed below.

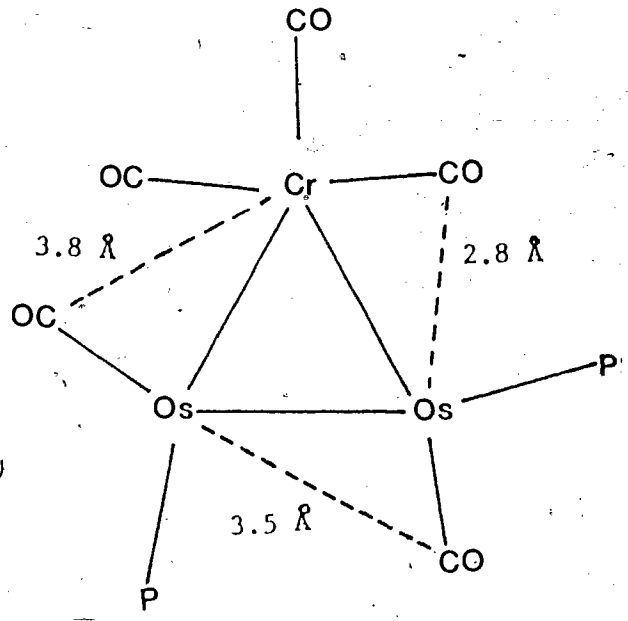
All-equatorial CO-exchange processes are rare in cluster chemistry but have been proposed for the CO-exchange heterotrimetallic clusters $[Os(CO)_3(PMe_3)]_2M(CO)_5$ (M = Cr,[72] Mo,[73] W[73]). A comparison of the equatorial planes of the clusters $Os_4(CO)_{15}$, $Os_4(CO)_{14}(PMe_3)$, and $[Os(CO)_3(PMe_3)]_2Cr(CO)_5$ is shown in Scheme 3.7. Also included for comparison is the equatorial plane of $Os_3(CO)_{12}$. Carbonyl exchange in $Os_3(CO)_{12}$ and $Os_3(CO)_{11}(L)$ derivatives is discussed in Chapter 4 in conjunction with the nonrigidity in $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$, but it is pointed out here that the low energy fluxional process in these trinuclear clusters involves

Scheme 3.7


OC
CO
Os
3.8 Å
4.0 Å
CO
OC
Os
Os
CO
3.3 Å
3.2 Å
Os
OC
CO


P
CO
Os
3.8 Å
4.0 Å
CO
OC
Os
Os
CO
3.5 Å
3.2 Å
Os
OC
CO


OC
CO
Os
3.6 Å
OC
CO
Os
Os
CO
CO


CO
OC
Cr
CO
3.8 Å
2.8 Å
OC
P
Os
Os
3.5 Å
P
CO

terminal-bridged CO-exchange in the axial planes of the clusters. As can be seen from Scheme 3.7, all-equatorial CO-exchange occurs in the clusters that have relatively short (2.8 - 3.4 Å) contacts between one metal atom and an equatorial carbonyl on an adjacent metal atom. This may be responsible for the observed low barrier to CO-exchange in these clusters. Also evident from Scheme 3.7 is that the shortest Os···C contacts in the tetranuclear clusters are between Os(1) and carbonyls on the wingtip groups, and carbonyls on Os(3) and the osmium atoms of the wingtip groups. This is consistent with the mechanism of bridge formation proposed for $Os_4(CO)_{15}$ in Scheme 3.3, and also with the modified mechanism for the CO-exchange in $Os_4(CO)_{14}(PMe_3)$ (Scheme 3.6). All-equatorial CO-exchange has also been proposed for the trinuclear clusters $Os_3(CO)_{10}(C_7H_8)$[134] and $Os_3(CO)_{10}(N_2C_4H_4)$.[135] The carbonyls in both clusters are fluxional on the NMR time scale, and low temperature limiting spectra were only obtained below -90 °C. Crystal structures have not been reported for either cluster, however, so it is not possible to determine the distances between the osmium atoms and the adjacent equatorial carbonyl ligands.

The variable temperature ^{13}C NMR spectra of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ are shown in Figure 3.10. The spectrum obtained at -45 °C is consistent with the crystal structure (Figure 3.11). This indicates that, unlike $Os_4(CO)_{15}$ and $Os_4(CO)_{14}(PMe_3)$, $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ is rigid on the NMR

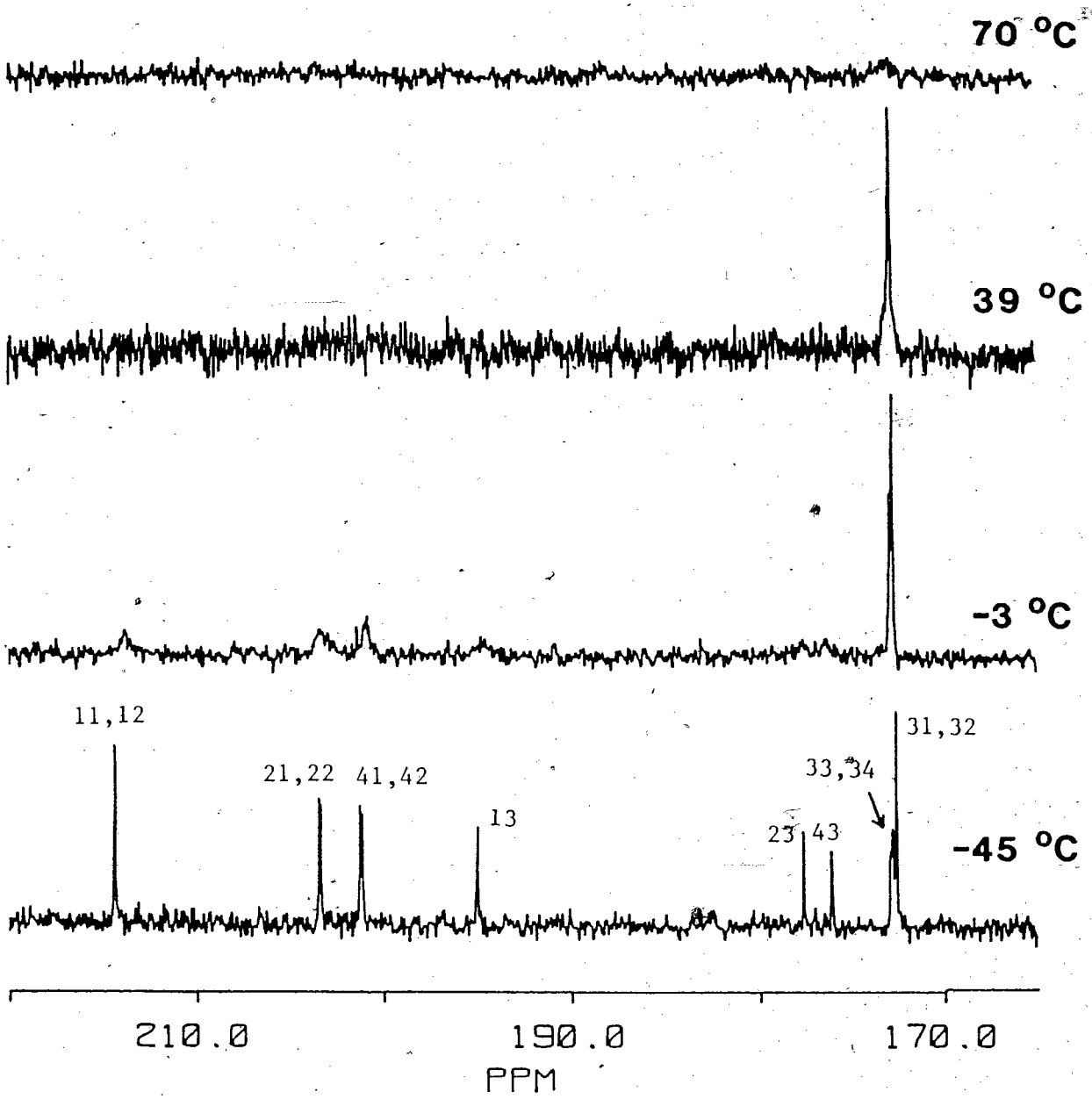


Figure 3.10. Variable Temperature ^{13}C NMR Spectra of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

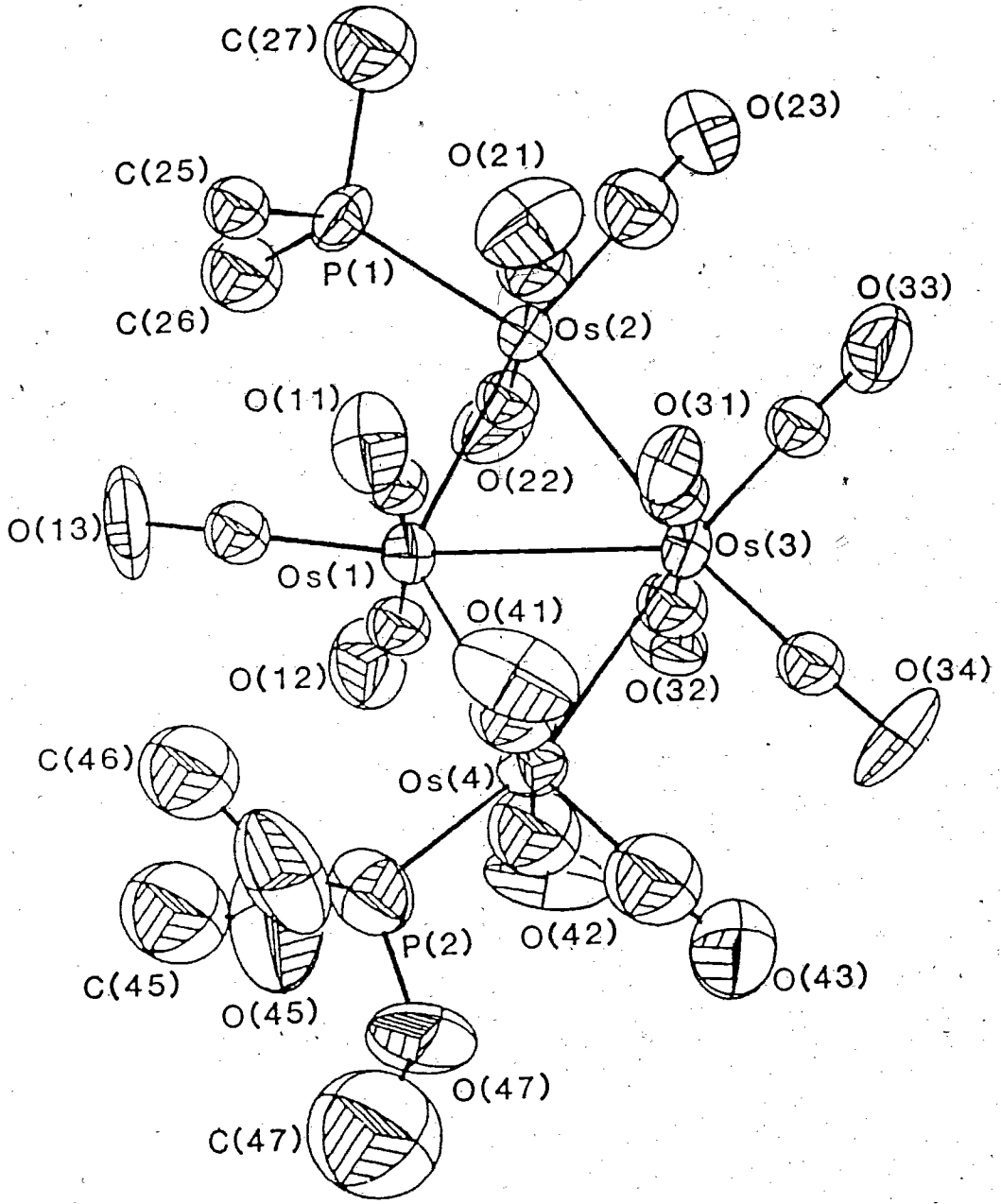


Figure 3.11. Molecular Structure of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

time scale in solution at this temperature. The labelling of the signals in Figure 3.10 follows the scheme used in Figure 3.11. The doublets at δ 203.5 (2C) and 201.3 (2C) are assigned to the axial carbonyls on Os(2) and Os(4) by virtue of their relative intensity and the observed coupling constants (they are coupled to the phosphorus atoms of PMe_3 and $P(OMe)_3$, respectively). The resonances at δ 214.6 (2C) and 195.2 are assigned to the axial and equatorial carbonyls on Os(1) on the basis of their unusual chemical shifts which suggest that they are due to carbonyls in unusual chemical environments. Similarly, the resonance at δ 172.8 (2C) is assigned to the axial carbonyls on Os(3). The assignment of the other resonances is less straightforward; it is based on the expected relative chemical shifts of the signals due to the equatorial carbonyls of the $Os(CO)_3(PMe_3)$ and $Os(CO)_3[P(OMe)_3]$ groups, and the mode of collapse of the signals in the spectra at higher temperatures (see below).

Upon warming the sample of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ to 0 °C, the six resonances to low field of δ 176 broadened and collapsed to the base line. Over the same temperature range the resonances at δ 173.1 and 172.9 (assigned to CO(33) and CO(34)) coalesced to a singlet. Because of the small chemical shift difference between these signals in the spectrum at -45 °C, it is likely that the coalescence is due to a shifting of the resonances with temperature rather than to an exchange process.

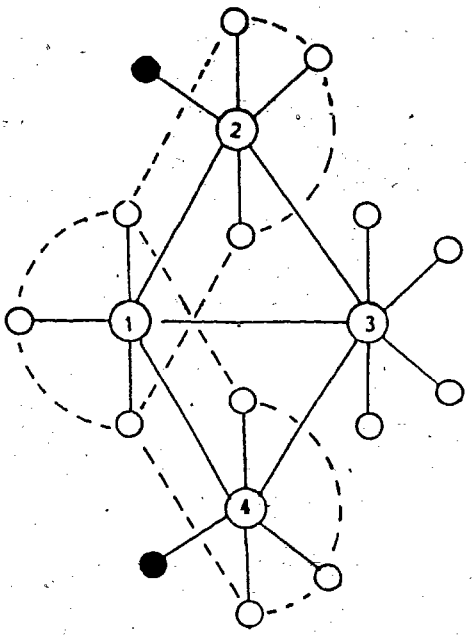
That the resonance assigned to CO(13) collapses when the sample is warmed to 0 °C suggests that the CO-exchange in

$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ does not involve an all-equatorial process because this would require one or both of the phosphorus ligands in $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ to move into a bridging position.

The collapse of the six lower field resonances is consistent with a pair of terminal-bridge CO-exchange processes taking place in the axial planes that contain Os(1)-Os(2) and Os(1)-Os(4) (Scheme 3.8). Simulation of the spectrum at 0 °C (Figure 3.12) gave a rate constant of 100 ± 10 s^{-1} for the exchange of carbonyls CO(11), CO(12), CO(13), CO(21), CO(22), and CO(23), and a rate constant of 70 ± 7 s^{-1} for the exchange of carbonyls CO(11), CO(12), CO(13), CO(41), CO(42), and CO(43). (As a result of the poor quality of the spectra the errors in the rate constants are probably underestimated.)

Warming the sample to room temperature caused the remaining two resonances (assigned to {CO(33), CO(34)} and {CO(31), CO(32)}) to coalesce to a singlet. This is consistent with a three-fold twist process taking place at Os(3) that scrambles the axial and equatorial carbonyls on this atom. Further warming of the sample caused this resonance to collapse. Terminal-bridged CO-exchange across the hinge would account for this observation, but it is not possible to confidently identify the process involved.

Scheme 3.8



≡ Os

≡ CO

≡ PMe$_3$ or P(OMe)$_3$

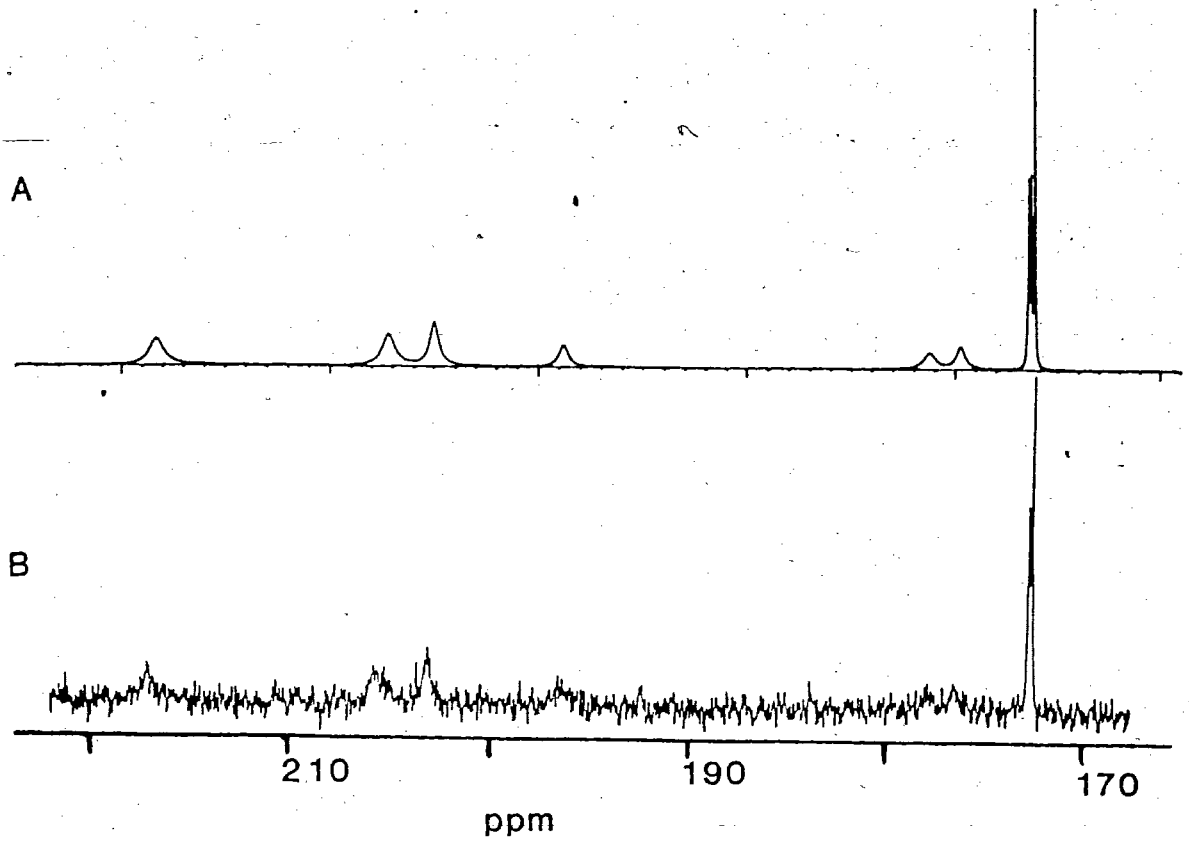


Figure 3.12. Simulated (A) and Experimental (B) ^{13}C NMR Spectra of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ at 0 °C.

3.4 Experimental

3.4.1 Syntheses

Improved Preparation of $Os_4(CO)_{13}(PMe_3)$

A Pyrex tube was charged with $Os_4(CO)_{14}(PMe_3)$[20] (98 mg, 0.08 mmol) and hexane (50 mL). The solution was stirred and subjected to UV irradiation for 2.5 h (≃ 3 cm between the waterjacket of the 200-W light source and the edge of the reaction vessel). The solution was purged with N_2 throughout the irradiation, and then for an additional 5 min after the lamp was turned off. The solution was then concentrated on the vacuum line, and stored at -15 °C overnight. The mother liquor was decanted, and the crystalline $Os_4(CO)_{13}(PMe_3)$ (82 mg, 86%) dried by vacuum. The product was pure as determined by infrared spectroscopy.

Preparation of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$

To a solution of $Os_4(CO)_{13}(PMe_3)$ (48 mg, 0.040 mmol) in hexane (60 mL) at room temperature $P(OMe)_3$ was slowly added dropwise until the color of the solution changed from red to dark green. The infrared spectrum of the solution at this point showed no bands due to $Os_4(CO)_{13}(PMe_3)$. The solvent and any excess $P(OMe)_3$ were removed on the vacuum line, and the brown residue was taken up in a minimum amount of CH_2Cl_2 and chromatographed on a silica-gel column (15 x 2.5 cm). A small amount of an unidentified yellow compound was eluted with hexane and discarded. The product, $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$, was eluted

with hexane/dichloromethane (1/1) as a red-green band. A yellowish compound was left on the column. The product-containing fraction was concentrated on the vacuum line and stored at -15 °C for 4 days. The mother liquor was decanted, and the crystals of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (49 mg, 92%) dried under vacuum: mp 127 °C; IR (hexane) ν(CO) 2102(m), 2075(w), 2044(m), 2036.5(s), 2024(vs), 2016(sh), 1997.5(m), 1977(sh), 1970(m), 1961(m), 1904.5(m) cm^{-1}; ^1H NMR ($CDCl_3$, ambient temperature) δ 3.65 d (J_{P-H} = 12 Hz), 1.83 d (J_{P-H} = 11 Hz); ^{13}C NMR (CD_2Cl_2/CH_2Cl_2, 5/1, -45 °C) δ 214.6 (2C), 203.5 (2C, d, J_{P-C} = 12 Hz), 201.3 (2C, d, J_{P-C} = 11 Hz), 195.2, 177.8, 176.3, 173.1, 172.9, 172.8 (2C); MS *m/z* 1304 ([M-CO]$^+$); Anal. Calcd for $H_{18}C_{19}O_{16}P_2Os_4$: C, 17.22; H, 1.37. Found: C, 17.66; H, 1.45.

Preparation of $Os_4(CO)_{14}(CNBu^t)$

A solution of $Os_4(CO)_{15}(CNBu^t)$[47] (76 mg, 0.050 mmol) in CH_2Cl_2 (30 mL) was stirred at room temperature. A slight stoichiometric excess of Me_3NO (5 mg, 0.066 mmol) slurried in CH_2Cl_2 was slowly added. The course of the reaction was followed by observing the the change in the color of the solution from amber to dark green and the disappearance of the ν(CN) absorption at 2222 cm^{-1} in the infrared spectrum of the reaction mixture. Upon completion of the reaction the solvent was removed on the vacuum line, and the green-brown residue dissolved in a minimum amount of CH_2Cl_2 and filtered through Celite. Hexane was layered on top of the filtrate, and the solution stored at -15 °C for two days. Decanting of the supernatant solution afforded $Os_4(CO)_{14}(CNBu^t)$

(46 mg, 62%): mp 124 °C; IR (hexane) ν(CN) 2185(m) cm^{-1}; ν(CO) 2110(m), 2074.5(s), 2066(s), 2054.5(s), 2045(vs), 2030(vs), 2015(vs), 2005(s), 1944.5(m), 1933.5(m) cm^{-1}; ^1H NMR (C_7D_8, ambient temperature) δ 0.96(s), 0.95(s); MS(EI) *m/z* 1209 ([M-CO]$^+$); Anal. Calcd for $H_9C_{19}NO_{14}Os_4$: C, 18.46; N, 1.13; H, 0.73. Found: C, 18.42; N, 1.28; H, 0.97.

Preparation of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$

A 125 mL round bottom flask (fitted with a Teflon valve) was placed in a cold bath at -196 °C and charged with $Os_3(CO)_{10}(COE)_2$[25] (60 mg, 0.057 mmol), $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$[24] (25 mg, 0.065 mmol), and hexane (30 mL). The vessel was placed under a slight vacuum and sealed. The orange solution was allowed to warm to room temperature and stirred until it had turned dark red (about 3.5 h). The solvent was removed on the vacuum line, and recrystalization of the residue from hexane/toluene (3/1) -15 °C afforded $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ (43 mg, 61% based on $Os_3(CO)_{10}(COE)_2$): mp 180 °C (dec); IR (hexane) ν(CO) 2107.5(m), 2075.5(w), 2062.5(s), 2038.5(sh), 2029(vs), 2021(sh), 2008(m), 1987(w), 1964(w), 1952.5(vw), 1934.5(w), 1927.5(w) cm^{-1}; ^1H NMR ($CDCl_3$, ambient temperature) δ 2.03 (Me); MS *m/z* 1206 ([M-CO]$^+$). Anal. Calcd for $H_{15}C_{22}O_{12}Os_3Ir$: C, 21.40; H, 1.22. Found C, 21.50; H. 1.17.

3.4.2 X-ray Crystallographic Studies

$Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$

Deep red, needle-shaped crystals of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ were grown from dichloromethane/hexane solution at -15 °C. A crystal with suitable cross section was cut across the long edge to dimensions 0.32 x 0.19 x 0.18 mm, and used for the data collection. The unit cell was determined on the basis of 25 accurately centered reflections, widely separated in reciprocal space, with $29° < 2\theta < 44°$. The space group, $P2_1/c$, was indicated by the systematic absences. An analytical absorption correction was applied to the data.

The structure was solved by direct methods which gave the positions of the four osmium atoms in the asymmetric unit. Fourier difference synthesis (after least-squares refinement of the partial model) revealed the positions of all non-hydrogen atoms. There was evidence for disorder of the methoxy groups of the trimethyl phosphite ligand, but this was not modeled (disorder is a common problem with the trimethyl phosphite ligand[136]). Six weak reflections that had very asymmetric backgrounds and the largest $\langle w(|F_o|-|F_c|)^2 \rangle$ values were excluded from the final cycles of refinement. These reflections were: (-8,4,6), (-9,2,6), (11,4,-4), (8,7,-5), (8,12,-6), and (8,3,-6). There were significant peaks ($\simeq$ 2 e Å^{-3}) near the osmium atoms (within 1 Å) in the final electron density difference map; these were most likely the result of improperly

Table 3.7. Crystallographic Data for the Structure Determinations of $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ (A) and $Os_4(CO)_{14}(CNBu^t)$ (B).

Compound	A	B
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/n$[a]
temp., °C	23	21
a (Å)	11.106(7)	11.747(3)
b (Å)	16.931(5)	18.009(5)
c (Å)	16.481(5)	12.448(2)
β, (°)	97.71(5)	92.65(2)
V (Å^3)	3071.0	2630.8
Z	4	4
FW	1324.8	1235.8
ρ_c (g cm^{-3})	2.87	3.12
μ Mo K_α (cm^{-1})	166.85	193.48
Transmission	0.126-0.195	0.51-1.00[b]
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$
Scan width (°)	1.2+0.35tanθ	1.0+0.35tanθ
Scan speed (°min^{-1})	1.7-5.5	1.0-2.8
Min-max 2θ (°)	0-45	0-50
Unique data	4003	4607
Obs data, I≥2.5σ(I)	2117	2131
Parameters	275	171
R_f [c]	0.051	0.054
R_{wf} [d]	0.053	0.050
Max |shift/error|	0.20	0.01
Max peak (e Å^{-3})	2.0(3)	2.5(6)
$G.O.F.$ [e]	1.32	1.04

[a]nonstandard setting of $P2_1/c$. [b]normalized transmission coefficients, see text. [c]$R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [d]$R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$. [e]$G.O.F. = [\Sigma w(|F_o| - |F_c|)^2/(N_{obs} - N_{var})]^{1/2}$.

Table 3.8. Fractional Coordinates for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.[a]

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.78038(13)	0.17035(8)	0.23725(8)
Os(2)	0.57949(13)	0.18132(8)	0.11720(8)
Os(3)	0.77657(13)	0.06787(8)	0.09311(8)
Os(4)	0.98272(14)	0.07342(9)	0.23248(9)
P(1)	0.4831 (9)	0.2823 (6)	0.1779 (6)
P(2)	1.0944 (14)	0.1014 (7)	0.3507 (8)
O(11)	0.899 (3)	0.3158 (14)	0.1657 (16)
O(12)	0.667 (3)	0.0564 (16)	0.3492 (16)
O(13)	0.749 (3)	0.2822 (18)	0.3747 (17)
O(21)	0.699 (3)	0.3111 (15)	0.0243 (19)
O(22)	0.449 (3)	0.0677 (14)	0.2188 (16)
O(23)	0.380 (3)	0.1556 (18)	-0.0234 (18)
O(31)	0.9184 (21)	0.1978 (13)	0.0186 (14)
O(32)	0.6444 (23)	-0.0519 (14)	0.1893 (15)
O(33)	0.5995 (25)	0.0486 (16)	-0.0617 (18)
O(34)	0.934 (3)	-0.0666 (14)	0.0424 (19)
O(41)	1.112 (3)	0.2045 (18)	0.1554 (18)
O(42)	0.870 (3)	-0.0612 (21)	0.3156 (20)
O(43)	1.159 (4)	-0.0420 (22)	0.1771 (22)
O(45)	1.030 (4)	0.0867 (20)	0.4323 (23)
O(46)	1.162 (3)	0.181 (3)	0.3665 (19)
O(47)	1.212 (4)	0.039 (3)	0.370 (3)
C(11)	0.853 (3)	0.2551 (20)	0.1876 (20)
C(12)	0.712 (3)	0.0922 (19)	0.3004 (21)
C(13)	0.765 (3)	0.2413 (20)	0.3188 (22)
C(21)	0.655 (4)	0.2585 (23)	0.0569 (24)
C(22)	0.503 (3)	0.1061 (20)	0.1824 (21)
C(23)	0.459 (4)	0.166 (3)	0.029 (3)

[a]Continued on following page.

Table 3.8. Continued.

Atom	*x/a*	*y/b*	*z/c*
C(31)	0.863 (3)	0.1501 (22)	0.0468 (22)
C(32)	0.696 (3)	-0.0040 (22)	0.1537 (22)
C(33)	0.672 (3)	0.0590 (20)	0.0005 (22)
C(34)	0.880 (3)	-0.0087 (21)	0.0626 (21)
C(41)	1.059 (4)	0.155 (3)	0.192 (3)
C(42)	0.909 (5)	-0.006 (3)	0.287 (3)
C(43)	1.102 (5)	0.008 (3)	0.197 (3)
C(25)	0.574 (3)	0.3725 (19)	0.2014 (20)
C(26)	0.428 (4)	0.2566 (23)	0.2736 (24)
C(27)	0.349 (4)	0.323 (3)	0.112 (3)
C(45)	0.993 (6)	0.113 (4)	0.491 (4)
C(46)	1.107 (5)	0.258 (3)	0.377 (3)
C(47)	1.316 (8)	0.042 (4)	0.415 (5)

modeled absorption. No peaks were observed which could be reasonably attributed to the methyl hydrogen atoms, and no attempt was made to include them in calculated positions. A weighting scheme based on counting statistics was adopted during the final cycles of refinement ($K = 4 \times 10^{-4}$).

Additional details of the data collection and structure refinement for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$ are given in Table 3.7. Bond length and angle data are listed in Table 3.1. Final atomic positional parameters are listed in Table 3.8. Final thermal motion parameters, and observed and calculated structure factors are given in Tables D.1 and D.2, respectively.

$Os_4(CO)_{14}(CNBu^t)$

Dark red, thin needles of $Os_4(CO)_{14}(CNBu^t)$ were grown by slowly cooling a dichloromethane/hexane solution from room temperature to 0 °C. A crystal with suitable cross section was cut across the long edge to dimensions 0.24 x 0.11 x 0.04 mm, and used for the data collection. The unit cell was determined on the basis of 25 accurately centered reflections, widely separated in reciprocal space, with $26° < 2\theta < 40°$. The monoclinic space group $P2_1/n$ was indicated by the systematic absences. An analytical absorption correction was applied to the data set.

The structure was solved by an inspection of a Patterson map which gave the positions of the osmium atoms. All other non-hydrogen atoms were placed from Fourier difference maps calculated after least-squares refinement of the partial model.

After refinement with anisotropic osmium atoms an electron density difference map revealed a pair of large peaks ($\simeq$ 4 e $Å^{-3}$) around each osmium atom. The peak-osmium-peak angles were approximately 180°, and the peak-peak vectors nearly parallel. Such a pattern is consistent with poorly modeled absorption, but could also result from crystal twinning. Disorder was ruled out as a possible cause because the peak positions were inconsistent with reasonable split osmium atom positions. An empirical absorption correction based on ψ-scans was also tried.[123] This gave essentially the same agreement as

Table 3.9. Fractional Coordinates for $Os_4(CO)_{14}(CNBu^t)$.[a]

Atom	x/a	y/b	z/c
Os(1)	0.1640(1)	0.23568(8)	0.2631(1)
Os(2)	0.1607(1)	0.1936(1)	0.0471(1)
Os(3)	0.1501(1)	0.07616(8)	0.2157(1)
Os(4)	0.1492(1)	0.14007(9)	0.4367(1)
O(11)	0.428(3)	0.243(2)	0.272(2)
O(12)	-0.092(3)	0.275(2)	0.246(2)
O(13)	0.190(3)	0.388(2)	0.352(3)
O(21)	0.419(3)	0.175(2)	0.037(2)
O(22)	-0.100(3)	0.198(2)	0.014(3)
O(23)	0.166(3)	0.364(2)	0.032(2)
O(24)	0.159(3)	0.141(2)	-0.178(3)
N(31)	0.419(2)	0.052(2)	0.228(2)
O(32)	-0.112(2)	0.089(1)	0.204(2)
O(33)	0.137(3)	-0.007(2)	-0.000(3)
O(34)	0.127(2)	-0.068(2)	0.341(2)
O(41)	0.412(2)	0.130(2)	0.454(2)
O(42)	-0.113(3)	0.165(2)	0.422(2)
O(43)	0.174(3)	0.258(2)	0.610(3)
O(44)	0.128(3)	0.004(2)	0.586(3)
C(11)	0.329(4)	0.235(2)	0.273(3)
C(12)	0.008(3)	0.259(2)	0.255(3)
C(13)	0.174(4)	0.327(2)	0.308(3)
C(21)	0.325(4)	0.183(3)	0.050(4)
C(22)	0.000(4)	0.193(2)	0.034(3)
C(23)	0.160(4)	0.295(3)	0.043(4)
C(24)	0.145(5)	0.162(3)	-0.088(5)
C(31)	0.320(4)	0.064(2)	0.225(3)
C(32)	-0.011(4)	0.088(2)	0.210(3)
C(33)	0.144(3)	0.030(2)	0.078(3)
C(34)	0.143(3)	-0.012(2)	0.297(2)

[a]Continued on the following page.

Table 3.9. Continued.

Atom	*x/a*	*y/b*	*z/c*
C(41)	0.309(3)	0.132(2)	0.435(3)
C(42)	-0.015(4)	0.150(2)	0.424(3)
C(43)	0.165(4)	0.211(3)	0.547(4)
C(44)	0.130(4)	0.056(3)	0.528(4)
C(1)	0.539(3)	0.028(2)	0.244(3)
C(2)	0.605(5)	0.095(3)	0.270(4)
C(3)	0.569(4)	-0.003(3)	0.135(4)
C(4)	0.556(5)	-0.025(3)	0.333(4)

---

the analytical correction for equivalent isotropic models, and a similar pattern of peaks near the osmium atoms was observed in the Fourier difference map.

Fourier refinement produced substantial shifts in the osmium atom positions, but these atoms returned to their original locations upon least-squares refinement. Because of these difficulties, the method of Walker and Stuart was used to model the absorption (R = 13%, no correction; R = 7%, after *DIFABS*).[137] After refinement of the model (anisotropic osmium atoms) with this absorption correction, the largest peaks in the electron density difference map were still near the osmium atoms, but were less than 2.5 e $Å^{-3}$ and showed a more random pattern.

At this point, the methyl hydrogen atoms were included at calculated positions (d(C-H) = 0.96 Å); the positions of these

atoms were not refined. The three methyl carbon atoms were assigned a common isotropic thermal motion parameter, as were the nine hydrogen atoms. An empirical weighting scheme was employed during the final cycles of refinement, with the weight, w, given by the expression $w = [26.5710t_0(X) + 40.5720t_1(X) + 24.4554t_2(X) + 8.03540t_3(X)]^{-1}$, where $X = |F_o|F_{max}^{-1}$ and t_n are polynomial functions of the Chebyshev series.[117]

Additional details of the data collection and structure refinement are given in Table 3.7. Final positional parameters for all non-hydrogen atoms of $Os_4(CO)_{14}(CNBu^t)$ are listed in Table 3.9. Bond lengths and selected bond angles are are given in Table 3.2. Hydrogen atom coordinates, thermal motion parameters for all atoms, and observed and calculated structure factors for $Os_4(CO)_{14}(CNBu^t)$ are listed in Tables E.1, E.2, and E.3, respectively.

$(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$

Dark red, prism-shaped crystals of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ were grown from dichloromethane/hexane solution at -15 °C. A crystal of dimensions 0.23 x 0.16 x 0.10 mm was selected for the data collection. The unit cell was determined on the basis of 25 accurately centered reflections, widely separated in reciprocal space, with $20° < 2\theta < 35°$. The monoclinic space group $P2_1/n$ was chosen after an examination of the systematic absences. An analytical absorption correction was applied to the data.

Table 3.10. Crystallographic Data for the Structure Determinations of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ (C) and $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ (D).

Compound	C	D
Crystal system	monoclinic	triclinic
Space group	$P2_1/n$	$P\bar{1}$
temp, °C	23	21
a (Å)	14.598(5)	7.929(1)
b (Å)	20.217(3)	12.072(2)
c (Å)	9.084(3)	14.739(2)
α, (°)		80.82(1)
β, (°)	91.07(3)	81.60(1)
γ, (°)		75.46(1)
V (Å^3)	2680.4	1338.0
Z	4	2
FW	1233.8	1317.7
ρ_c (g cm^{-3})	3.06	3.55
μ Mo K_α (cm^{-1})	191.84	216.45
Transmission	0.113-0.227	0.054-0.299
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$
Scan width (°)	$0.8+0.35\tan\theta$	$0.9+0.35\tan\theta$
Scan speed (°min^{-1})	0.8-2.8	0.9-3.3
Min-max 2θ (°)	0-45	0-50
Unique data	3483	4371
Obs data, $I \geq 2.5\sigma(I)$	2014	3113
Parameters	233	240
R_f [a]	0.040	0.041
R_{wf} [b]	0.048	0.046
Max |shift/error|	0.02	0.05
Max peak (e Å^{-3})	1.5(3)	2.1(3)
$G.O.F.$ [c]	1.01	1.23

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$.
[c] $G.O.F. = [\Sigma w(|F_o| - |F_c|)^2/(N_{obs} - N_{var})]^{1/2}$.

Table 3.11. Fractional Coordinates for $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$.[a]

Atom	x/a	y/b	z/c
Ir	0.24696(7)	0.11628(4)	0.73395(11)
Os(1)	0.16406(7)	0.19720(5)	0.53563(12)
Os(2)	0.25011(8)	0.29768(5)	0.37850(13)
Os(3)	0.34944(7)	0.23176(5)	0.62978(13)
O(11)	0.0585 (15)	0.2762 (12)	0.766 (3)
O(12)	0.2102 (16)	0.0989 (11)	0.2932 (22)
O(13)	-0.0272 (15)	0.1638 (10)	0.439 (3)
O(21)	0.1412 (20)	0.3841 (11)	0.589 (3)
O(22)	0.3497 (15)	0.2049 (12)	0.1691 (24)
O(23)	0.1047 (15)	0.3214 (10)	0.1409 (22)
O(24)	0.3855 (18)	0.4068 (11)	0.277 (3)
O(31)	0.2617 (14)	0.3322 (10)	0.840 (3)
O(32)	0.4300 (14)	0.1283 (9)	0.4211 (21)
O(33)	0.4918 (13)	0.3365 (11)	0.554 (3)
O(34)	0.4569 (12)	0.1746 (9)	0.8892 (19)
O(41)	0.1778 (15)	0.1982 (9)	0.9794 (23)
C(11)	0.1033 (22)	0.2472 (15)	0.685 (3)
C(12)	0.1958 (22)	0.1368 (16)	0.385 (4)
C(13)	0.0491 (23)	0.1772 (15)	0.478 (4)
C(21)	0.185 (3)	0.3467 (19)	0.515 (4)
C(22)	0.3102 (22)	0.2357 (16)	0.260 (4)
C(23)	0.1612 (19)	0.3116 (13)	0.231 (3)
C(24)	0.3338 (23)	0.3693 (16)	0.321 (4)
C(31)	0.2928 (20)	0.2957 (15)	0.771 (3)
C(32)	0.3944 (19)	0.1644 (14)	0.500 (3)
C(33)	0.4370 (19)	0.2998 (13)	0.583 (3)
C(34)	0.4129 (17)	0.1933 (12)	0.793 (3)
C(41)	0.2060 (18)	0.1712 (13)	0.883 (3)

[a]Continued on following page.

Table 3.11. Continued.

Atom	*x/a*	*y/b*	*z/c*
C(1)	0.1977 (16)	0.0239 (11)	0.827 (3)
C(2)	0.2918 (18)	0.0295 (13)	0.879 (3)
C(3)	0.3424 (17)	0.0267 (12)	0.747 (3)
C(4)	0.2820 (17)	0.0172 (12)	0.620 (3)
C(5)	0.1947 (17)	0.0153 (12)	0.672 (3)
C(6)	0.115 (3)	0.0199 (19)	0.939 (5)
C(7)	0.3331 (21)	0.0360 (15)	1.029 (3)
C(8)	0.4455 (23)	0.0250 (17)	0.740 (4)
C(9)	0.3103 (24)	-0.0067 (17)	0.473 (4)
C(10)	0.1076 (25)	-0.0018 (17)	0.595 (4)

The heavy atoms were placed by direct methods. All other non-hydrogen atoms were located in Fourier difference maps calculated after least-squares refinement of the partial model. The iridium atom was identified by its attachment to the η^5-C_5Me_5 ligand. After refinement with anisotropic osmium, iridium, and oxygen atoms, the methyl hyrdogen atoms were included at calculated positions (d(C-H) = 0.96 Å); the hydrogen atom positions were not refined, but their positions were recalculated after refinement of the rest of the model. This procedure was repeated until the C-H distances had stabilized. A weighting scheme based on counting statistics was adopted (K = 1 x 10^{-3}).

Additional details of the data collection and structure refinement are shown in Table 3.10. Final fractional coordinates

for all non-hydrogen atoms of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ are collected in Table 3.11; bond lengths and selected bond angles are given in Table 3.3. Hydrogen atom coordinates, thermal motion parameters for all atoms, and observed and calculated structure factors are listed in Tables F.1, F.2 and F.3, respectively.

$Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$

Crystals of $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ were supplied by Mr. C.-Y. Lu; they were grown from toluene solution at -15 °C as air-stable, orange-red plates. A sample with suitable thickness was cut across the plate to dimensions 0.23 x 0.18 x 0.07 mm and used for the data collection. The triclinic space group $P\bar{1}$ was confirmed as correct by the successful solution and refinement of the structure. The unit cell was determined on the basis of 23 reflections, widely spread through reciprocal space, with $32° < 2\theta < 45°$. An analytical absorption correction was applied to the data.

The osmium atoms were placed by direct methods. All other non-hydrogen atoms were located from Fourier electron density difference maps calculated after least-squares refinement of the partial model. After refinement with anisotropic osmium, tin, and oxygen atoms, the methyl hydrogen atoms were included as fixed atom contributions at calculated positions (d(C-H) = 0.96 Å). Upon convergence of the model, the lone bridging hydride ligand was placed at the position calculated by the

Table 3.12. Fractional Coordinates for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.15505(8)	0.26127(6)	0.68998(4)
Os(2)	0.40873(8)	0.07588(6)	0.77988(4)
Os(3)	0.29972(8)	0.31335(6)	0.84557(4)
Os(4)	0.04069(9)	0.48528(6)	0.74059(5)
Sn	0.43654(18)	-0.11488(12)	0.70034(10)
O(11)	0.4464 (18)	0.3276 (14)	0.5447 (9)
O(12)	-0.1424 (15)	0.1722 (11)	0.8098 (9)
O(13)	-0.0441 (17)	0.2727 (15)	0.5271 (9)
O(21)	0.7129 (18)	0.1189 (14)	0.6349 (10)
O(22)	0.1263 (18)	-0.0241 (15)	0.9074 (11)
O(23)	0.6845 (19)	-0.0602 (14)	0.9034 (11)
O(31)	0.5976 (16)	0.3746 (13)	0.7039 (8)
O(32)	0.0083 (17)	0.2247 (13)	0.9770 (9)
O(33)	0.5734 (16)	0.1795 (14)	0.9737 (8)
O(34)	0.2476 (25)	0.5249 (15)	0.9453 (12)
O(41)	0.3427 (20)	0.5685 (16)	0.6107 (13)
O(42)	-0.2568 (17)	0.4128 (14)	0.8726 (10)
O(43)	-0.1888 (19)	0.5468 (15)	0.5840 (11)
O(44)	-0.0477 (24)	0.7079 (18)	0.8261 (16)
C(11)	0.3352 (24)	0.3059 (16)	0.6021 (12)
C(12)	-0.0329 (23)	0.2081 (16)	0.7678 (12)
C(13)	0.0298 (23)	0.2684 (16)	0.5876 (12)
C(21)	0.6000 (24)	0.1076 (16)	0.6872 (12)
C(22)	0.229 (3)	0.0190 (18)	0.8643 (14)
C(23)	0.580 (3)	-0.0112 (20)	0.8557 (14)
C(31)	0.3874 (25)	0.3533 (16)	0.7527 (12)
C(32)	0.1127 (24)	0.2602 (16)	0.9261 (12)
C(33)	0.472 (3)	0.2253 (18)	0.9240 (13)
C(34)	0.258 (3)	0.4469 (19)	0.9037 (14)

[a]Continued on following page.

Table 3.12. Continued.

Atom	*x/a*	*y/b*	*z/c*
C(41)	0.234 (3)	0.5316 (19)	0.6582 (14)
C(42)	-0.141 (3)	0.4344 (17)	0.8263 (13)
C(43)	-0.102 (3)	0.5248 (19)	0.6423 (14)
C(44)	-0.017 (3)	0.6253 (24)	0.7928 (17)
C(1)	0.202 (4)	-0.104 (3)	0.6409 (20)
C(2)	0.475 (3)	-0.2736 (22)	0.7982 (16)
C(3)	0.652 (4)	-0.143 (3)	0.5957 (22)
H[b]	0.271	0.110	0.683

[b]Hydride is in a calculated position.

HYDEX program of Orpen.[126] This atom was not refined. A weighting scheme based on counting statistics was adopted during the final cycles of refinement ($K = 5 \times 10^{-4}$).

Additional details of the data collection and structure refinement are given in Table 3.10. Final fractional coordinates for all non-hydrogen atoms and the bridging hydride ligand are collected in Table 3.12. Bond length and selected bond angle data are listed in Table 3.4. Methyl hydrogen atom coordinates, thermal motion parameters for all atoms, and observed and calculated structure factors for $Os_4(\mu\text{-}H)(CO)_{14}SnMe_3$) are given in Tables G.1, G.2, and G.3, respectively.

CHAPTER 4

STRUCTURAL VARIATION IN SOME 64-ELECTRON CLUSTERS OF THE TYPE $Os_4(CO)_{15}(L)$

4.1 Introduction

In Chapter 2 it was noted that although the 64-electron clusters $Os_4(CO)_{16}$ and $Os_4(CO)_{15}(PMe_3)$ are constitutionally closely related, they adopt radically different structures (Figure 4.1). For $Os_4(CO)_{16}$, a novel cyclobutane-like structure is observed, whereas for $Os_4(CO)_{15}(PMe_3)$ the more common spiked-triangular geometry is found.[20] Both clusters can be prepared by the addition of L (L = CO or PMe_3) to $Os_4(CO)_{15}$ under mild conditions (eqn 4.1). This suggests that this dramatic difference in structure is a function of L. But is this influence steric or electronic in origin?

$$Os_4(CO)_{15} + L \longrightarrow Os_4(CO)_{15}(L) \qquad (4.1)$$
$$L = CO,\ PMe_3$$

The structural chemistry of $Os_4(CO)_{16}$ was discussed in Section 2.3, and that of $Os_4(CO)_{15}(PMe_3)$ was described elsewhere.[20] Of importance here is the difference between $Os_4(CO)_{15}(PMe_3)$ and other spiked-triangle clusters. In $Os_4(CO)_{15}(PMe_3)$, the "spike" is formed by the 18-electron complex $Os(CO)_4(PMe_3)$, which acts as a donor ligand to an $Os_3(CO)_{11}$ fragment via an unbridged,

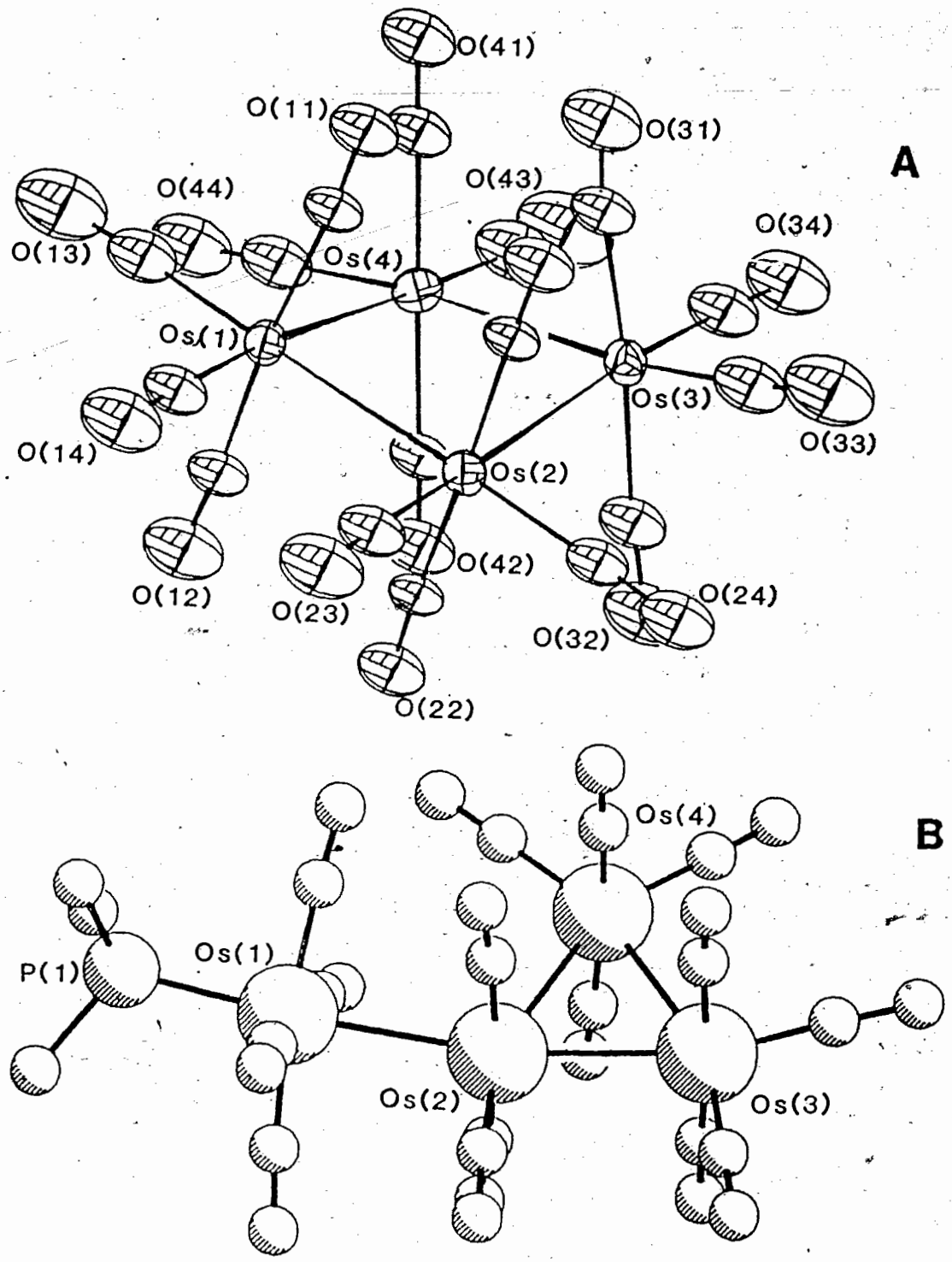


Figure 4.1. Molecular Structures of $Os_4(CO)_{16}$ (A) and $Os_4(CO)_{15}(PMe_3)$ (B).

dative Os-Os bond. The behavior of $Os_4(CO)_{15}(PMe_3)$ in solution has also been investigated; no evidence was found for a puckered-square isomer.[20]

Given the crowding of the equatorial CO groups on $Os_4(CO)_{16}$ (Chapter 2) and that PMe_3 is larger than CO, it is possible that the spiked-triangle form is favored for steric reasons. On the other hand, work from this laboratory has shown that $Os(CO)_4(PMe_3)$ is a much better ligand than $Os(CO)_5$.[20] This is probably because electron donation from the PMe_3 ligand makes the osmium atom in $Os(CO)_4(PMe_3)$ more electron rich than that in $Os(CO)_5$. Thus the difference in the electronic properties of CO and PMe_3 could be responsible for the different structures adopted by $Os_4(CO)_{16}$ and $Os_4(CO)_{15}(PMe_3)$.

In order to determine more precisely the factors which govern the structural preference of the $Os_4(CO)_{15}(L)$ (L = donor ligand) clusters, five new examples were prepared and characterized. The related cluster $Os_4(CO)_{14}(CNBu^t)_2$ was also prepared.[47] The choices of L were made in order that, as far as possible, the electronic and steric influences of L could be distinguished. Three of the new clusters, for L = $CNBu^t$, PF_3, and $P(OCH_2)_3CMe$, have been characterized by X-ray crystallography. The structures of $Os_4(CO)_{15}(SbPh_3)$ and $Os_4(CO)_{14}(CNBu^t)_2$ were inferred from spectroscopic data. The results strongly suggest that the structural preference of the $Os_4(CO)_{15}(L)$ clusters is governed by the electronic properties of L.

To determine if there was any isomerization from the spiked-triangle to the puckered-square geometry in solution, the variable temperature ^{13}C NMR spectra of $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$ were obtained. No evidence was found for a puckered-square isomer of either compound. Some of the fluxional processes in these clusters are characteristic of $Os_3(CO)_{11}(L)$ (L = donor ligand) clusters. In addition, the low temperature spectra of $Os_4(CO)_{15}(CNBu^t)$ are consistent with restricted rotation about the dative osmium-osmium bond.

4.2 Results

$Os_4(CO)_{15}(CNBu^t)$

Treatment of $Os_4(CO)_{15}$ in CH_2Cl_2 at 0 °C with $CNBu^t$ caused an immediate color change from dark-green to orange (eqn 4.2). The product, $Os_4(CO)_{15}(CNBu^t)$, was isolated in 88% yield as air-stable, yellow-orange crystals; it it decomposes slowly in solution at room temperature.

$$Os_4(CO)_{15} + CNBu^t \xrightarrow[CH_2Cl_2]{0\ ^\circ C} Os_4(CO)_{15}(CNBu^t) \qquad (4.2)$$

The infrared C≡N stretch of $Os_4(CO)_{15}(CNBu^t)$ (in hexane) occurs at 2200 cm^{-1} compared to 2190 cm^{-1} for $Os(CO)_4(CNBu^t)$. This shift to higher frequency is consistent with the expected decreased backbonding into the π^* orbitals of the C≡N group as a result of the removal of electron density from the osmium atom of $Os(CO)_4(CNBu^t)$ through donation to the $Os_3(CO)_{11}$ fragment.

The structure of $Os_4(CO)_{15}(CNBu^t)$ (Figure 4.2) consists of a spiked-triangular arrangement of metal atoms, with the 18-electron complex $Os(CO)_4(CNBu^t)$ acting as a donor ligand to the $Os_3(CO)_{11}$ triangle via an unbridged dative Os-Os bond. The $CNBu^t$ group is cis to this bond. In $Os_4(CO)_{15}(PMe_3)$, the PMe_3 group is located trans to the dative bond.[20] Possible reasons for the different site preferences of these ligands are discussed in Section 4.3.

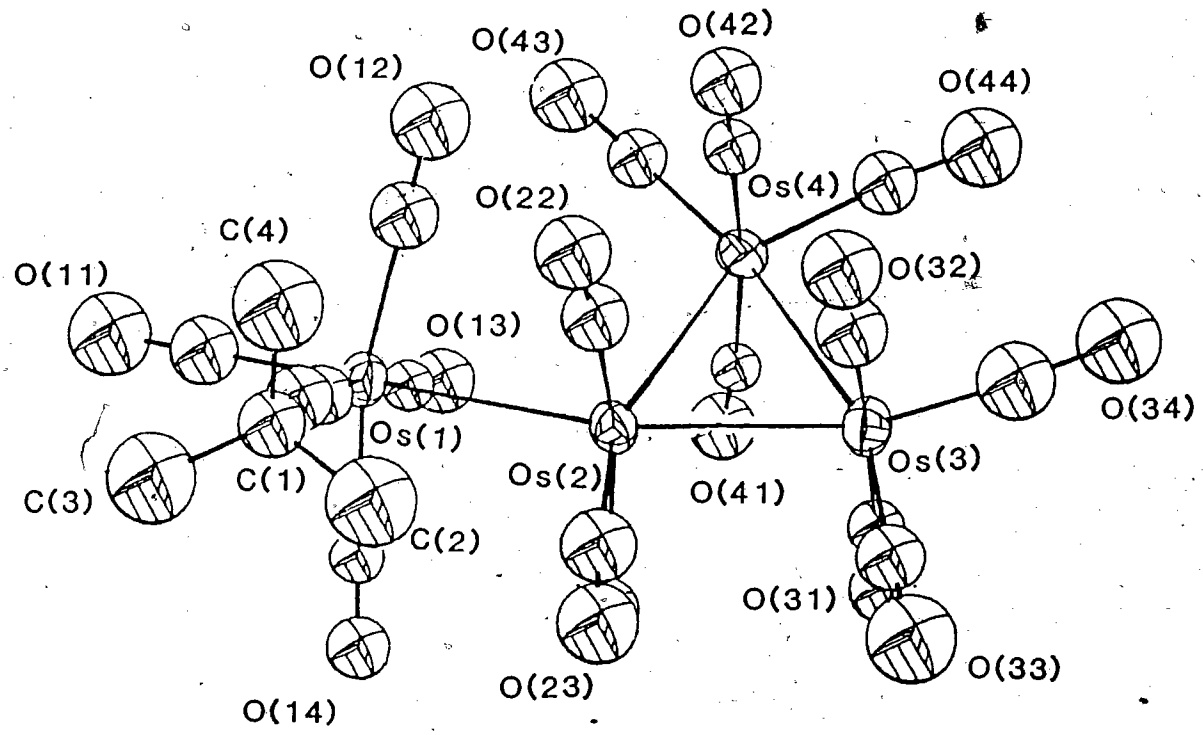


Figure 4.2. Molecular Structure of $Os_4(CO)_{15}(CNBu^t)$. Hydrogen atoms have been omitted for clarity.

Table 4.1. Selected Molecular Dimensions of $Os_4(CO)_{15}(CNBu^t)$.

Bond Lengths (Å)

Os(1)-Os(2)	2.918 (2)	Os(2)-Os(4)	2.929 (2)
Os(2)-Os(3)	2.853 (2)	Os(3)-Os(4)	2.890 (2)
Os(1)-C(11)	1.88 (4)	Os(1)-C(12)	1.92 (4)
Os(1)-C(13)	1.88 (3)	Os(1)-C(14)	2.01 (3)
Os(1)-C(15)	2.04 (4)	Os(2)-C(21)	1.83 (4)
Os(2)-C(22)	1.94 (3)	Os(2)-C(23)	1.80 (5)
Os(3)-C(31)	2.00 (4)	Os(3)-C(32)	1.81 (4)
Os(3)-C(33)	1.81 (4)	Os(3)-C(34)	1.76 (5)
Os(4)-C(41)	1.96 (3)	Os(4)-C(42)	1.95 (3)
Os(4)-C(43)	1.82 (4)	Os(4)-C(44)	1.89 (4)
C(11)-O(11)	1.15 (5)	C(12)-O(12)	1.13 (4)
C(13)-O(13)	1.18 (3)	C(14)-O(14)	1.03 (3)
C(15)-N(1)	1.17 (4)	C(21)-O(21)	1.14 (4)
C(22)-O(22)	1.22 (4)	C(23)-O(23)	1.23 (5)
C(31)-O(31)	1.07 (4)	C(32)-O(32)	1.24 (5)
C(33)-O(33)	1.19 (5)	C(34)-O(34)	1.26 (5)
C(41)-O(41)	1.14 (3)	C(42)-O(42)	1.12 (4)
C(43)-O(43)	1.21 (4)	C(44)-O(44)	1.18 (5)
N(1)-C(1)	1.44 (5)	C(1)-C(2)	1.50 (6)
C(1)-C(3)	1.50 (6)	C(1)-C(4)	1.48 (5)

The four osmium atoms are essentially planar; Os(1) is displaced from the plane of the Os_3 triangle by 0.100 (3) Å on the side of the plane with the $CNBu^t$ group. This small deviation from planarity may be due to packing forces or to intramolecular steric interactions. The Os-Os bond lengths in this cluster are listed Table 4.1. Of the three Os-Os bonds in the Os_3 unit, the bond that is cis to the $Os(CO)_4(PMe_3)$ group is the longest (Os(2)-Os(4) = 2.929 (2) Å), while the one that is trans to this group is the shortest (Os(2)-Os(3) = 2.853 (2) Å). The bond

Table 4.1. Continued.

Selected Bond Angles (deg)

Os(1)-Os(2)-Os(3)	164.02 (6)	Os(1)-Os(2)-Os(4)	104.17 (6)
Os(3)-Os(2)-Os(4)	59.96 (5)	Os(2)-Os(3)-Os(4)	58.72 (5)
Os(2)-Os(1)-C(11)	176 (1)	C(11)-Os(1)-C(12)	95 (2)
C(12)-Os(1)-Os(2)	86 (1)	C(12)-Os(1)-C(13)	93 (2)
C(12)-Os(1)-C(14)	165 (2)	C(13)-Os(1)-C(15)	173 (2)
C(21)-Os(2)-C(22)	172 (2)	C(21)-Os(2)-C(23)	90 (2)
C(31)-Os(3)-C(32)	177 (2)	C(31)-Os(3)-C(33)	89 (2)
C(33)-Os(3)-C(34)	100 (2)	C(41)-Os(4)-C(43)	90 (2)
C(43)-Os(4)-C(44)	98 (2)	C(15)-N(1)-C(1)	170 (4)
N(1)-C(1)-C(2)	108 (4)	N(1)-C(1)-C(3)	112 (4)
N(1)-C(1)-C(4)	103 (4)		

between Os(3) and Os(4) has an intermediate length of 2.890 (2) Å. A similar pattern of bond length variation is found for the other $Os_4(CO)_{15}(L)$ derivatives of this type, and is discussed in Section 4.3.

The ^{13}C NMR spectrum (CO region) of $Os_4(CO)_{15}(CNBu^t)$ (^{13}CO-enriched) in $CHFCl_2/CD_2Cl_2$ at -118 °C has 14 resonances each of intensity one, except for that at δ 184.6 which has an intensity of two. The spectrum obtained at -108 °C reveals that this signal is comprised of two resonances. Fifteen signals are consistent with the structure of $Os_4(CO)_{15}(CNBu^t)$ found in the solid. This indicates that there is restricted rotation about the dative Os-Os bond in solution at this temperature. Upon warming the solution of $Os_4(CO)_{15}(CNBu^t)$, sets of the signals in the spectrum collapse to the base line so that at 0 °C only the

resonance at δ 174.9 remains. The observed changes in the spectra are discussed, and various mechanisms for carbonyl exchange proposed in Section 4.3.

$Os_4(CO)_{15}[P(OCH_2)_3CMe]$

The $P(OCH_2)_3CMe$ ligand was included in this study because it is the next best π-acceptor after PF_3 that was readily available.[139] It has a much smaller cone angle than PMe_3 (θ = 101° compared to θ = 118°), and although it is a poorer donor than PMe_3 it is still better than CO.[140]

Dropwise addition of $P(OCH_2)_3CMe$ (in CH_2Cl_2) to a CH_2Cl_2 solution of $Os_4(CO)_{15}$ at 0 °C (eqn 4.3) led to the disappearance of the green color of $Os_4(CO)_{15}$ and the formation of an amber solution. The progress of the reaction was conveniently monitored by infrared spectroscopy. The product, $Os_4(CO)_{15}[P(OCH_2)_3CMe]$, rapidly decomposes in the presence of excess $P(OCH_2)_3CMe$, and was isolated in only 37% yield (after recrystallization). When pure, solid $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ is air-stable at room temperature.

$$Os_4(CO)_{15} + P(OCH_2)_3CMe \xrightarrow[CH_2Cl_2]{0\ ^\circ C} Os_4(CO)_{15}[P(OCH_2)_3CMe] \qquad (4.3)$$

A single crystal X-ray diffraction study revealed the cluster to have a spiked-triangular structure (Figure 4.3), with a dative Os-Os bond linking the 18-electron species

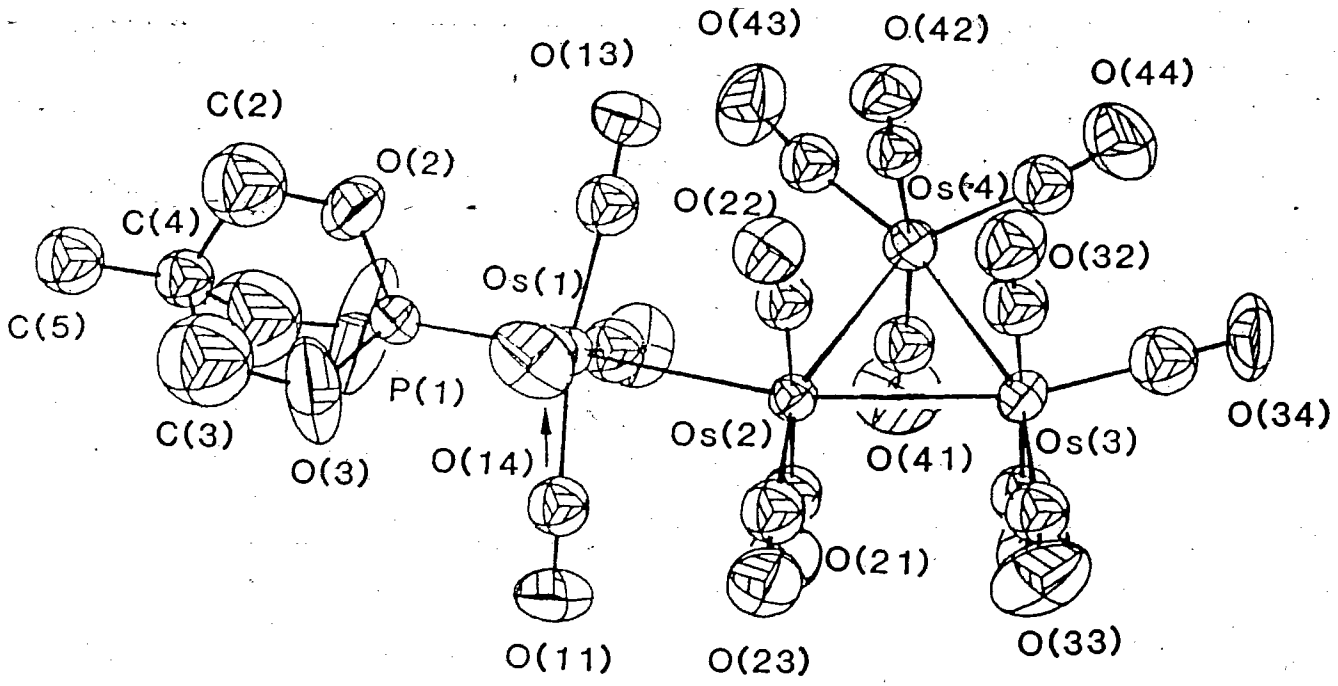


Figure 4.3. Molecular Structure of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$. Hydrogen atoms have been omitted for clarity.

Table 4.2. Selected Molecular Dimensions of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

Bond Lengths (Å)

Os(1)-Os(2)	2.926 (1)	Os(2)-Os(3)	2.849 (1)
Os(2)-Os(4)	2.936 (1)	Os(3)-Os(4)	2.882 (1)
Os(1)-P(1)	2.269 (6)	Os(1)-C(11)	1.96 (3)
Os(1)-C(12)	1.96 (3)	Os(1)-C(13)	1.94 (3)
Os(1)-C(14)	2.02 (3)	Os(2)-C(21)	1.92 (3)
Os(2)-C(22)	1.94 (3)	Os(2)-C(23)	1.85 (3)
Os(3)-C(31)	1.99 (3)	Os(3)-C(32)	1.88 (3)
Os(3)-C(33)	1.91 (3)	Os(3)-C(34)	1.86 (3)
Os(4)-C(41)	1.96 (3)	Os(4)-C(42)	1.96 (3)
Os(4)-C(43)	1.87 (3)	Os(4)-C(44)	1.89 (3)
C(11)-O(11)	1.13 (3)	C(12)-O(12)	1.13 (3)
C(13)-O(13)	1.12 (3)	C(14)-O(14)	1.06 (3)
C(21)-O(21)	1.16 (3)	C(22)-O(22)	1.14 (3)
C(23)-O(23)	1.15 (3)	C(31)-O(31)	1.12 (3)
C(31)-O(31)	1.12 (3)	C(32)-O(32)	1.19 (3)
C(33)-O(33)	1.13 (3)	C(34)-O(34)	1.15 (3)
C(41)-O(41)	1.13 (2)	C(42)-O(42)	1.10 (3)
C(43)-O(43)	1.15 (3)	C(44)-O(44)	1.16 (3)
P(1)-O(1)	1.52 (3)[a]	P(1)-O(2)	1.54 (3)[a]
P(1)-O(3)	1.53 (3)[a]	O(1)-C(1)	1.42 (2)[a]
O(2)-C(2)	1.43 (2)[a]	O(3)-C(3)	1.42 (2)[a]
C(4)-C(1)	1.53 (3)	C(4)-C(2)	1.50 (2)
C(4)-C(3)	1.52 (2)	C(4)-C(5)	1.49 (4)

[a]denotes restrained bond length

$Os(CO)_4[P(OCH_2)_3CMe]$ to an $Os_3(CO)_{11}$ fragment. As in $Os_4(CO)_{15}(PMe_3)$,[20] the phosphorus ligand is trans to the dative bond. The four osmium atoms are essentially planar, with Os(1) ≃ 0.06 Å from the plane of the Os_3 unit. The Os-Os bond lengths in $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ are listed along with selected bond

Table 4.2. Continued.

Selected Bond Angles (deg)

Os(4)-Os(3)-Os(2)	61.62 (3)	Os(1)-Os(2)-Os(3)	163.31 (4)
Os(1)-Os(2)-Os(4)	103.62 (4)	Os(3)-Os(2)-Os(4)	59.73 (3)
Os(2)-Os(4)-Os(3)	58.64 (3)	P(1)-Os(1)-Os(2)	178.8 (2)
P(1)-Os(1)-c(11)	95.9 (8)	C(11)-Os(1)-C(12)	89 (1)
C(11)-Os(1)-C(13)	169 (1)	C(12)-Os(1)-C(14)	171 (1)
C(21)-Os(2)-C(22)	176 (1)	C(21)-Os(2)-C(23)	89 (1)
C(31)-Os(3)-C(32)	177 (1)	C(31)-Os(3)-C(33)	92 (1)
C(33)-Os(3)-C(34)	102 (1)	C(41)-Os(4)-C(42)	171 (1)
C(41)-Os(4)-C(43)	87 (1)	C(43)-Os(4)-C(44)	103 (1)

angles in Table 4.2.

Disorder was evident in the $P(OCH_2)_3CMe$ ligand (see the experimental section for a description of the disorder). In the most reasonable restrained model, the $P(OCH_2)_3CMe$ group has local C_3 symmetry (not C_{3v}). In each of the two disordered halves of this ligand two of the C(4)-C-O-P(1) units have torsion angles of $\simeq 14°$, while the third has a torsion angle of $\simeq 5°$.

$Os_4(CO)_{15}(PF_3)$

Treatment of a degassed solution of $Os_4(CO)_{15}$ in CH_2Cl_2 at -20 °C with an atmosphere of PF_3 caused slow conversion to $Os_4(CO)_{15}(PF_3)$ (eqn 4.4). Again the distinctive green color of $Os_4(CO)_{15}$ gave way to an amber colored solution characteristic of the 64-electron $Os_4(CO)_{15}(L)$ clusters. Because the low thermal stability of $Os_4(CO)_{15}(PF_3)$ made monitoring the reaction

Table 4.3. Selected Molecular Dimensions of $Os_4(CO)_{15}(PF_3)$.

Bond Lengths (Å)

Os(1)-Os(2)	3.005 (2)	Os(1)-Os(4)	3.000 (2)
Os(2)-Os(3)	2.977 (2)	Os(3)-Os(4)	2.994 (2)
Os(1)-P(1)	2.192 (2)	Os(1)-C(11)	1.96 (3)
Os(1)-C(12)	1.90 (3)	Os(1)-C(13)	1.81 (3)
Os(2)-C(21)	1.89 (3)	Os(2)-C(22)	1.94 (3)
Os(2)-C(23)	1.93 (3)	Os(2)-C(24)	1.88 (3)
Os(3)-C(31)	1.83 (3)	Os(3)-C(32)	1.94 (3)
Os(3)-C(33)	1.96 (3)	Os(3)-C(34)	1.89 (3)
Os(4)-C(41)	1.98 (3)	Os(4)-C(42)	1.98 (3)
Os(4)-C(43)	1.89 (3)	Os(4)-C(44)	1.95 (3)
P(1)-F(1)	1.50 (2)	P(1)-F(2)	1.49 (2)
P(1)-F(3)	1.44 (2)	C(11)-O(11)	1.14 (4)
C(12)-O(12)	1.20 (4)	C(13)-O(13)	1.20 (4)
C(21)-O(21)	1.19 (4)	C(22)-O(22)	1.16 (4)
C(23)-O(23)	1.17 (4)	C(24)-O(24)	1.17 (4)
C(31)-O(31)	1.19 (4)	C(32)-O(32)	1.13 (4)
C(33)-O(33)	1.14 (4)	C(34)-O(34)	1.17 (4)
C(41)-O(41)	1.10 (4)	C(42)-O(42)	1.09 (4)
C(43)-O(43)	1.15 (4)	C(44)-O(44)	1.11 (4)

difficult, it was not possible to determine when the reaction had reached completion.

$$Os_4(CO)_{15} + PF_3 \xrightarrow[CH_2Cl_2]{-20\ ^\circ C} Os_4(CO)_{15}(PF_3) \qquad (4.4)$$

It is noteworthy that $Os_4(CO)_{15}(PF_3)$ is stable to the presence of excess PF_3 (at -20 °C). Other $Os_4(CO)_{15}(L)$ derivatives, for L = PMe_3, $SbPh_3$, $P(OCH_2)_3CMe$, or $CNBu^t$, decompose in the presence of excess L. The significance of this observation is addressed

Table 4.3. Continued.

Selected Bond Angles (deg)

Os(2)-Os(1)-Os(4)	86.94 (4)	Os(1)-Os(2)-Os(3)	89.38 (4)
Os(2)-Os(3)-Os(4)	87.59 (4)	Os(3)-Os(4)-Os(1)	89.15 (4)
Os(2)-Os(1)-C(11)	94.2 (8)	Os(3)-Os(2)-C(21)	93.6 (8)
C(11)-Os(1)-C(12)	171 (1)	C(21)-Os(2)-C(22)	174 (1)
C(11)-Os(1)-P(1)	92 (1)	C(21)-Os(2)-C(23)	92 (1)
C(11)-Os(1)-C(13)	94 (1)	C(21)-Os(2)-C(24)	93 (1)
P(1)-Os(1)-Os(2)	177.88 (5)	C(23)-Os(2)-Os(3)	88 (1)
C(13)-Os(1)-P(1)	90 (1)	C(23)-Os(2)-C(24)	93 (1)
C(31)-Os(3)-Os(4)	88 (1)	C(41)-Os(4)-Os(1)	93 (1)
C(31)-Os(3)-C(32)	174 (1)	C(41)-Os(4)-C(42)	175 (1)
C(31)-Os(3)-C(33)	95 (1)	C(41)-Os(4)-C(43)	91 (1)
C(31)-Os(3)-C(34)	94 (1)	C(41)-Os(4)-C(44)	90 (1)
C(33)-Os(3)-Os(4)	172 (1)	C(43)-Os(4)-Os(1)	175 (1)
C(33)-Os(3)-C(34)	95 (1)	C(43)-Os(4)-C(44)	91 (1)

in Section 4.3.

The decomposition of $Os_4(CO)_{15}(PF_3)$ in solution was also a problem during work up, and contributed to the low isolated yield (20%). The compound can be obtained as green-yellow crystals from $CHCl_3/C_6H_{14}$ at -50 °C. When dry, crystals of $Os_4(CO)_{15}(PF_3)$ are air stable at room temperature.

The structure of $Os_4(CO)_{15}(PF_3)$ was determined by X-ray crystallography. The cluster adopts a cyclobutane-like structure with three $Os(CO)_4$ groups and one $Os(CO)_3(PF_3)$ group forming the ring. All 16 ligands are terminal (Figure 4.4). The Os-Os bond lengths are similar to those found in $Os_4(CO)_{16}$, and range from

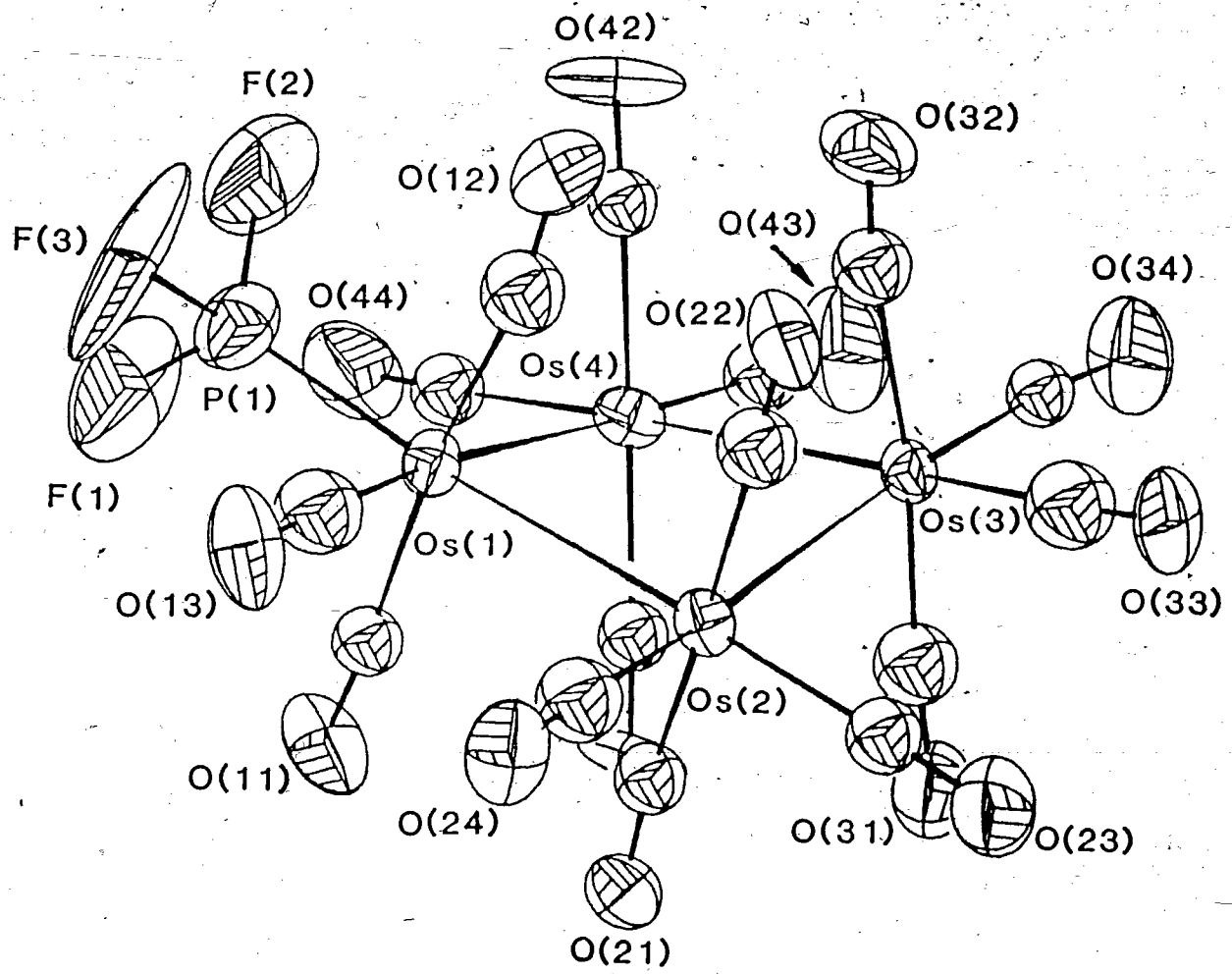


Figure 4.4. Molecular Structure of $Os_4(CO)_{15}(PF_3)$.

2.977 (2) Å to 3.005 (2) Å (see Table 4.3). The ring is distinctly puckered; the dihedral angle between the planes Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(4) is 151.68 (6)°, compared to 158.99 (4)° for the analogous planes in $Os_4(CO)_{16}$.

The ^{13}C NMR spectrum of $Os_4(CO)_{15}(PF_3)$ (^{13}CO-enriched, in $CHFCl_2/CD_2Cl_2$) at -55 °C (Figure 4.5) consists of seven resonances in the equatorial carbonyl region (1C each, and one showing small phosphorus coupling), and three resonances in the axial carbonyl region (one coupled to phosphorus).[67] The resonance at δ 176.5 has a shoulder and is significantly broader than the other signals in the spectrum. This pattern is consistent with the solid state structure of $Os_4(CO)_{15}(PF_3)$ if there is rapid ring inversion and if the resonances due to axial carbonyls on two different osmium atoms are nearly degenerate.

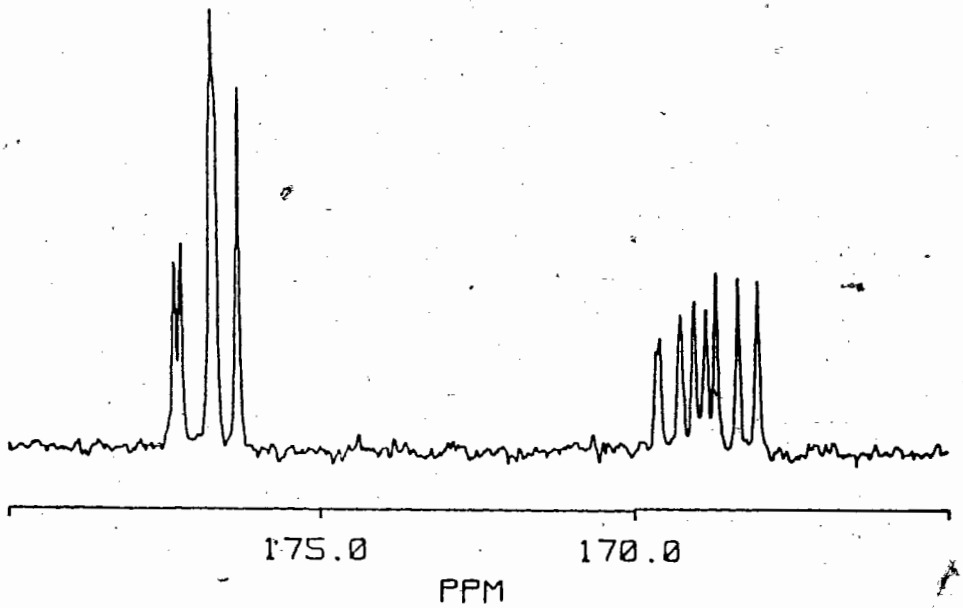


Figure 4.5. ^{13}C NMR Spectrum of $Os_4(CO)_{15}(PF_3)$ in $CHFCl_2/CD_2Cl_2$ at -55 °C.

There is no indication of line broadening in the spectrum obtained at -122 °C, which suggests that there is a low barrier to ring inversion. The spectra at warmer temperatures were not recorded due to the appreciable decomposition of $Os_4(CO)_{15}(PF_3)$ in solution above -20 °C. There was no evidence for a spiked-triangular isomer of $Os_4(CO)_{15}(PF_3)$ in solution at or below -55 °C.

$Os_4(CO)_{15}(SbPh_3)$

Slow addition of solid $SbPh_3$ to a CH_2Cl_2 solution of $Os_4(CO)_{15}$ at 0 °C gave $Os_4(CO)_{15}(SbPh_3)$ after 1.5 h (eqn 4.5). The color of the solution slowly changed from dark-green to amber, and the course of the reaction was followed by the disappearance of bands due to $Os_4(CO)_{15}$ in the infrared spectrum of the reaction mixture. The product was isolated in 37% yield as air-stable, yellow crystals from CH_2Cl_2/hexane.

$$Os_4(CO)_{15} + SbPh_3 \xrightarrow[CH_2Cl_2]{0\ ^\circ C} Os_4(CO)_{15}(SbPh_3) \quad (4.5)$$

Like the other derivatives, $Os_4(CO)_{15}(SbPh_3)$ decomposed in solution at room temperature over a period of 20 h. At 0 °C, this decomposition is sufficiently slow that it probably did not contribute to the low isolated yield. Instead, since both $Os(CO)_4(SbPh_3)$ and $Os_3(CO)_{11}(SbPh_3)$ were observed in the reaction mixture, it seems that $Os_4(CO)_{15}(SbPh_3)$ was reacting with excess $SbPh_3$ present in the solution (see below).

The carbonyl stretching region of the infrared spectrum of $Os_4(CO)_{15}(SbPh_3)$ is remarkably similar to that of $Os_4(CO)_{15}(PMe_3)$ (Figure 4.6). This strongly suggests that the two clusters have the same gross structure in solution (a spiked-triangle with $Os(CO)_4(L)$ acting as a ligand to $Os_3(CO)_{11}$ and with L trans to the dative bond). Therefore, it was decided not to perform an X-ray crystal structure investigation on $Os_4(CO)_{15}(SbPh_3)$.

$Os_4(CO)_{14}(CNBu^t)_2$

In an effort to prepare $Os_4(CO)_{14}(CNBu^t)$ by the addition of $CNBu^t$ to $Os_4(CO)_{14}$, the only isolated product was found to be $Os_4(CO)_{14}(CNBu^t)_2$ (eqn 4.6). The novel fluxional properties found for $Os_4(CO)_{15}(CNBu^t)$ (see below) and the possibility of similar processes in $Os_4(CO)_{14}(CNBu^t)_2$ made this cluster a desirable target molecule.

$$Os_4(CO)_{14} + 2CNBu^t \xrightarrow[CH_2Cl_2]{RT} Os_4(CO)_{14}(CNBu^t)_2 \qquad (4.6)$$

This 64-electron cluster was isolated in 63% yield after recrystallization. It is more stable in solution than the other $Os_4(CO)_{15}(L)$ derivatives reported here. Figures 4.6 and 4.7 show the appropriate regions of the infrared spectra of $Os_4(CO)_{15}(PMe_3)$, $Os_4(CO)_{15}(SbPh_3)$, $Os_4(CO)_{15}(CNBu^t)$, and $Os_4(CO)_{14}(CNBu^t)_2$. As can be seen they are quite similar. In addition, the presence of two C≡N stretches in the spectrum of

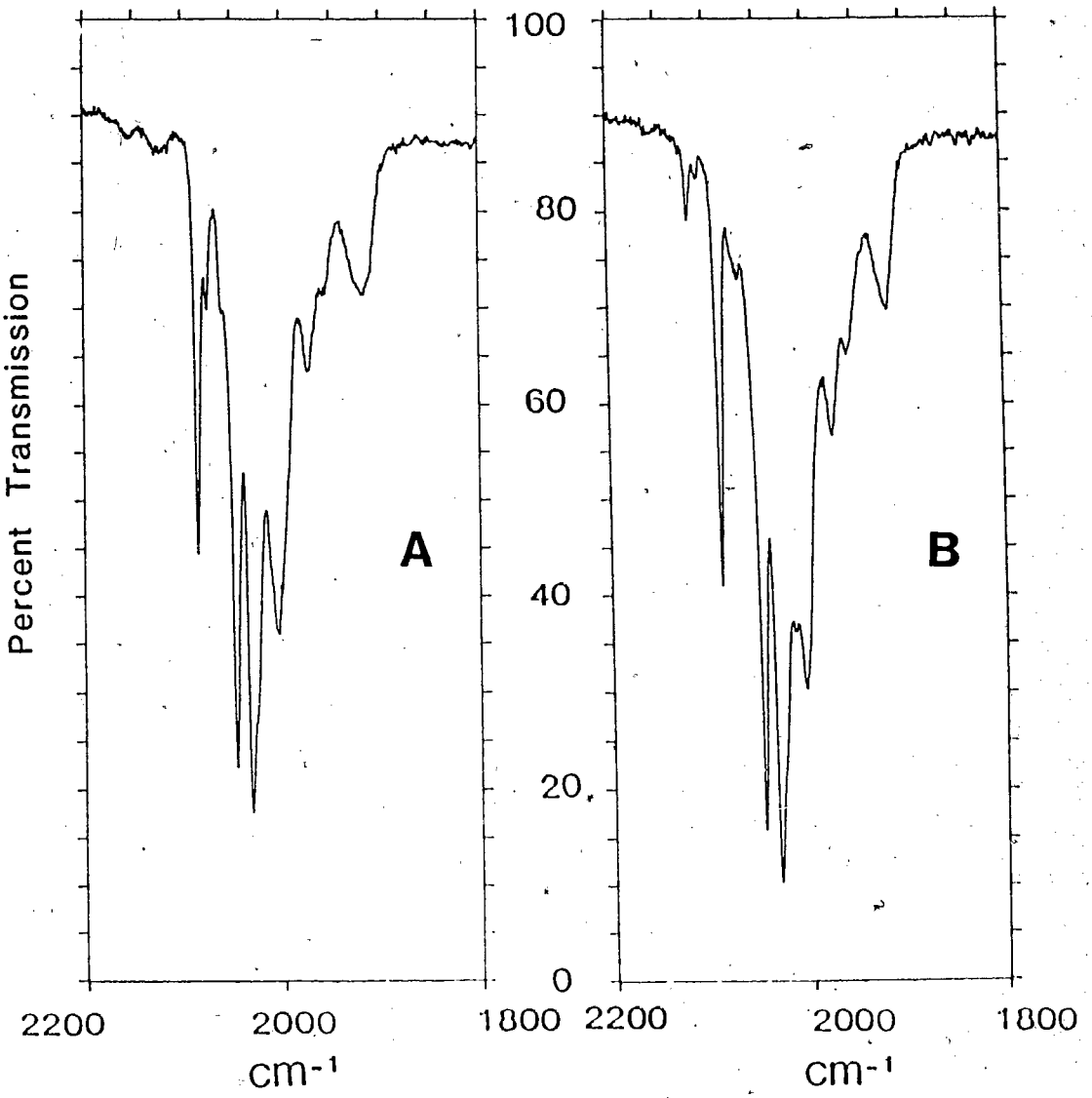


Figure 4.6. Infrared Spectra of $Os_4(CO)_{15}(PMe_3)$ (A) and $Os_4(CO)_{15}(SbPh_3)$ (B) in CH_2Cl_2 (2200 - 1800 cm^{-1}).

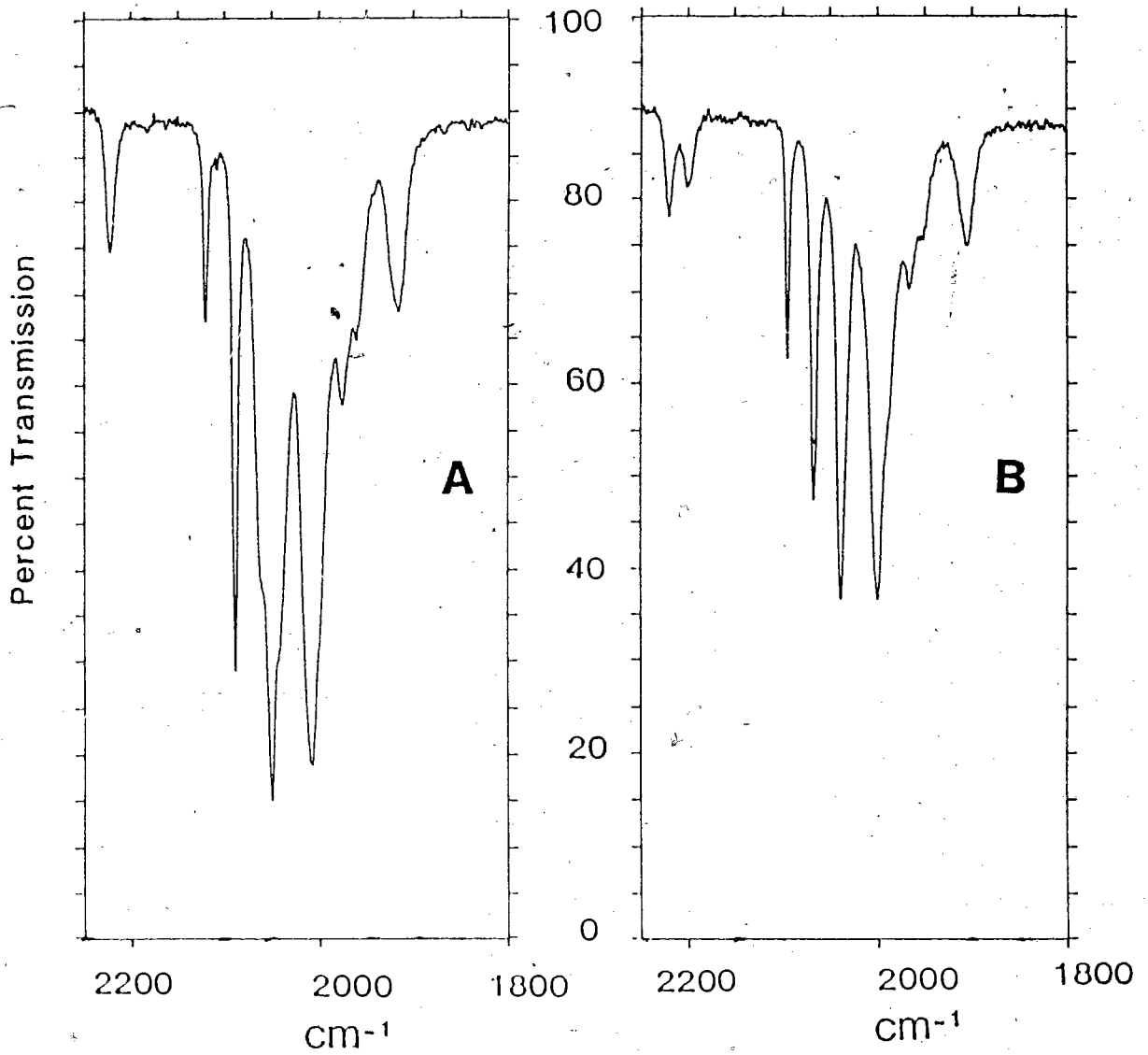


Figure 4.7. Infrared Spectra of $Os_4(CO)_{15}(CNBu^t)$ (A) and $Os_4(CO)_{14}(CNBu^t)_2$ (B) in CH_2Cl_2 (2250 - 1800 cm^{-1}).

$Os_4(CO)_{14}(CNBu^t)_2$ is consistent with a cis arrangement of the $CNBu^t$ ligands. The ^{13}C NMR spectrum of $Os_4(CO)_{14}(CNBu^t)_2$ (in CD_2Cl_2/CH_2Cl_2) at -101 °C indicates that this cluster has a spiked-triangular structure, with the 18-electron complex $Os(CO)_3(CNBu^t)_2$ acting as a ligand to the $Os_3(CO)_{11}$ fragment and with the two $CNBu^t$ groups mutually cis and cis to the dative bond. As expected, the spectra obtained at warmer temperatures show that the molecule is stereochemically nonrigid on the NMR time scale. Possible mechanisms for the carbonyl exchange in $Os_4(CO)_{14}(CNBu^t)_2$ are discussed in Section 4.3.

4.3 Discussion

The Influence of L on the Structure of $Os_4(CO)_{15}(L)$ Clusters

All of the $Os_4(CO)_{15}(L)$ clusters (L = CO, PMe_3, $CNBu^t$ $SbPh_3$, $P(OCH_2)_3CMe$, or PF_3) can be prepared by the addition of L to $Os_4(CO)_{15}$ (eqn 4.7). The PMe_3 derivative can also be prepared by the addition of the 18-electron complex $Os(CO)_4(PMe_3)$ to either $Os_3(CO)_{11}(MeCN)$ or $Os_3(\mu\text{-}H)_2(CO)_{10}$ under mild conditions (eqn 4.8 and 4.9).[20] Furthermore, for L = CO, $CNBu^t$, and PMe_3, the $Os_4(CO)_{15}(L)$ clusters can be prepared by the addition of CO to $Os_4(CO)_{14}(L)$ (eqn 4.10). The latter reaction is not a recommended route to the spiked-triangular $Os_4(CO)_{15}(L)$ clusters because these compounds react with CO to give $Os_3(CO)_{12}$ and $Os(CO)_4(L)$ (eqn 4.11). It is possible that this reaction is responsible for the failure to isolate $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{13}$ from the reaction of $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$ with CO, since only $Os_3(CO)_{12}$ and $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$ were observed. This would mean that the 64-electron cluster, if it is formed, adopts a spiked-triangular structure in solution (with $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$ acting as a ligand to $Os_3(CO)_{11}$). However, it was found that $(\eta^5\text{-}C_5Me_5)Ir(CO)_2$ does not displace the labile ligands in $Os_3(CO)_{11}(COE)$ (COE = cyclooctene) or $Os_3(CO)_{11}(MeCN)$,[14,1] which suggests that $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{13}$ may not be stable.

$$Os_4(CO)_{15} + L \longrightarrow Os_4(CO)_{15}(L) \qquad (4.7)$$

$L = CO,\ PMe_3,\ CNBu^t,\ SbPh_3,\ P(OCH_2)_3CMe,\ PF_3$

$$Os(CO)_4(PMe_3) + Os_3(CO)_{11}(MeCN) \longrightarrow Os_4(CO)_{15}(PMe_3) \qquad (4.8)$$

$$Os(CO)_4(PMe_3) + Os_3(\mu\text{-}H)_2(CO)_{10} \longrightarrow Os_4(CO)_{15}(PMe_3) \qquad (4.9)$$

$$Os_4(CO)_{14}(L) + CO \longrightarrow Os_4(CO)_{15}(L) \qquad (4.10)$$

$L = CO,\ PMe_3,\ CNBu^t$

$$Os_4(CO)_{15}(L) + CO \longrightarrow Os_3(CO)_{12} + Os(CO)_4(L) \qquad (4.11)$$

$L = PMe_3,\ CNBu^t$

The above evidence, together with the observation that the non-carbonyl ligand is always on the spike group in the spiked-triangular clusters, indicates that dramatic change from spiked-triangle to puckered-square on going from, for example, L = PMe_3 to L = CO must be a function of L.

Table 4.4 contains Tolman's cone angles and electronic parameters[140] for the ligands used in this study, along with the structure adopted by the $Os_4(CO)_{15}(L)$ clusters. Note that the spiked-triangular structure is observed for the non-carbonyl ligands with both the smallest ($CNBu^t$) and largest ($SbPh_3$) cone angles. The puckered-square, on the other hand, is found for PF_3, a ligand with a cone angle (θ = 104°) intermediate between

Table 4.4. Properties of Some Common Donor Ligands.

Ligand	θ (deg)[a]	ν (cm^{-1})[a]	Structure[b]
PMe_3	118	2064.1	spiked-triangle
$P(OCH_2)_3CMe$	101	2087.3	spiked-triangle
PF_3	104	2110.8	puckered-square
$SbPh_3$[c]	145	2068.0	spiked-triangle
CO	95	–	puckered-square

[a]Tolman's cone angle and electronic parameter, ref 140. [b]Structure adopted by $Os_4(CO)_{15}(L)$. [c]By analogy to PPh_3, ref 140.

that of PMe_3 (θ = 118°) and $P(OCH_2)_3CMe$ (θ = 101°).[140] This indicates that the gross structures of the $Os_4(CO)_{15}(L)$ clusters are probably independent of the size of L.

Also evident from Table 4.4 is the relationship between the structures of the $Os_4(CO)_{15}(L)$ clusters and the donor properties of L. No quantitative data is available for the electronic properties of $CNBu^t$, but this ligand is commonly thought to be a much better donor than CO.[142] Thus it is seen that when L is a superior donor than CO the spiked-triangular geometry adopted. The conclusion is that the donor properties of L dictate the structure of $Os_4(CO)_{15}(L)$.

However, a steric influence of L on the structure of the $Os_4(CO)_{15}(L)$ clusters cannot be ruled out. When L = $SbPh_3$ (the largest ligand in this study) a spiked-triangular structure is found for the $Os_4(CO)_{15}(L)$ derivative. Although the data for $SbPh_3$ in Table 4.4 suggests that this ligand is a good donor,

other evidence indicates that this may not be the case. For example, the observation that $Os(CO)_4(SbPh_3)$ exists as both axial and equatorial isomers has been interpreted in terms of this bulky ligand being a poor donor.[143] The variable temperature ^{13}C NMR spectra of $Os_3(CO)_{11}(SbPh_3)$ are also consistent with this conclusion.[69] Note that Tolman's electronic parameter is a measure of the net effect of the σ-donor and π-acceptor properties of the ligand on the metal, and thus is not a true measure of the σ-donor strength of the ligand. It should also be noted that the donor properties of a ligand may depend on the metal to which it is attached. Therefore, Tolman's data derived from $Ni(CO)_3(L)$ systems may not be transferable to osmium cluster compounds. If $SbPh_3$ is a poor donor ligand then the observed spiked-triangular structure may result from the steric bulk of this ligand.

Within the spiked-triangular clusters the non-carbonyl ligand occupies the position trans to the dative bond in every case studied except when L = $CNBu^t$. The same situation is found for binuclear species prepared in this laboratory that contain dative bonds between $Os(CO)_4(L)$ derivatives and 16-electron transition metal fragments. Thus in $(Me_3P)(OC)_4OsM(CO)_5$ (M = Cr, Mo, W) the PMe_3 ligand is found trans to the dative Os-M bond,[19] while in $(Bu^tNC)(OC)_4OsCr(CO)_5$ the $CNBu^t$ group is cis to the dative Os-Cr bond.[138] In addition, the variable temperature ^{13}C NMR spectra of $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$ give no evidence for the presence in solution of isomers with a $CNBu^t$

group trans to the dative bond (see below). In $Os_4(CO)_{15}(PMe_3)$, on the other hand, an isomer with the PMe_3 group cis to the dative Os-Os bond has been proposed to account for the nonrigidity of this cluster.[20] These observations suggest that the cis position may be electronically favored but sterically disfavored. The cause of the electronic site preference is not clear, but it may be due to the preference of the better π-acceptor CO groups to avoid (as far as possible) being trans to each other.

Another notable feature in the structures of both the spiked-triangular clusters and the binuclear compounds is that the radial ligands on the donor groups are displaced towards the acceptor fragments. This is best seen in the case of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ (Figure 4.8). That the radial carbonyls on the $M(CO)_5$ units of the series $(Me_3P)(OC)_4OsM(CO)_5$ (M = Cr, Mo, W) are not canted indicates that the leaning of the radial ligands on the donor moiety is due to electronic rather than steric factors.[19] There are several possible orbital interactions which could give rise to the observed geometry.[92] Perhaps the most important is the stabilization of the a_1 donor orbital through interaction with the π^*_{CO} orbitals on the radial CO ligands which is increased by the canting of the ligands.[92,144]

The lengths of the dative Os-Os bonds in the three structurally determined spiked-triangular $Os_4(CO)_{15}(L)$ derivatives are 2.938 (1) Å (L = PMe_3),[20] 2.926 (1) Å (L =

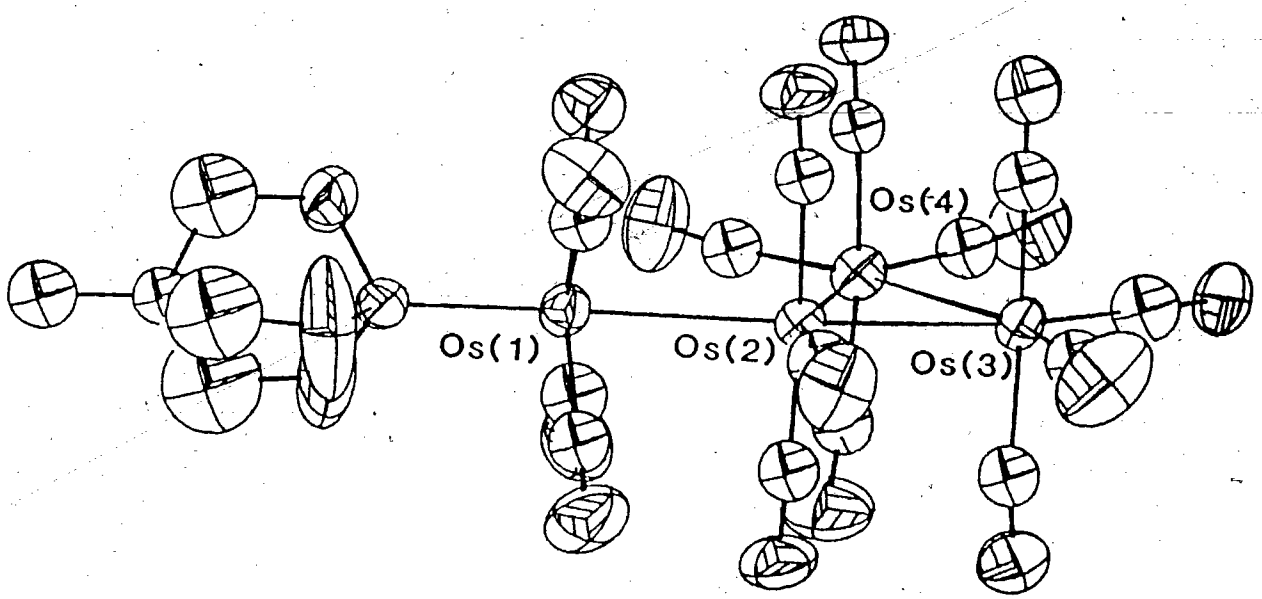


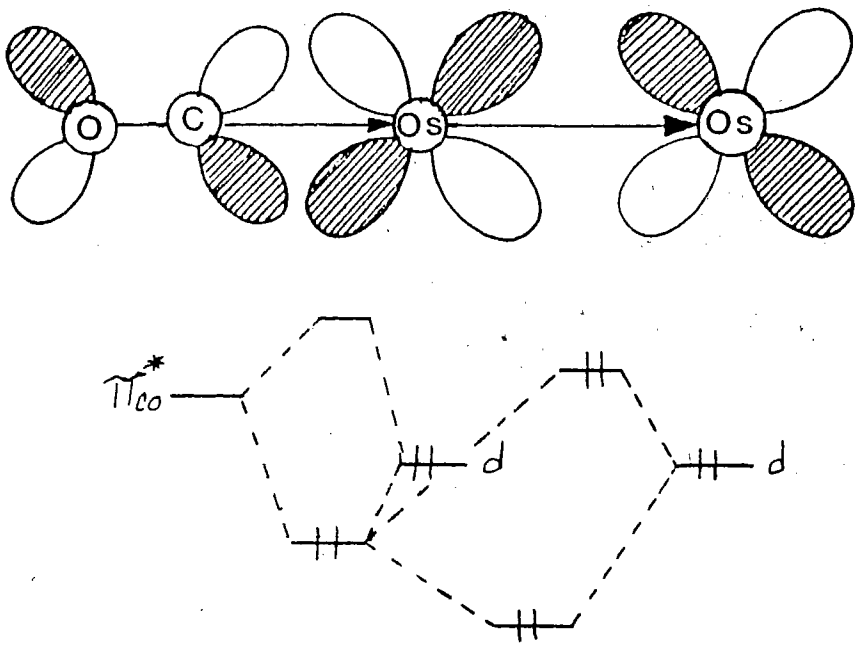
Figure 4.8. Molecular Structure of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ Showing the Canting of the Radial Carbonyls on Os(1).

$P(OCH_2)_3CMe$), and 2.918 (2) Å (L = $CNBu^t$). The differences are small, but the trend is opposite to that expected based on the relative donor strengths of the L groups (Table 4.4, and ref 69). However, the trend is consistent with the relative trans influences of the the ligands (CO is trans to the dative bond when L = $CNBu^t$).[87]

Another explanation for the observed bond lengths can be found by considering the nature of the dative Os-Os bond. This bond can be thought of as arising from the overlap of a filled orbital of a_1 symmetry on the $Os(CO)_4(L)$ group (the HOMO of an 18-electron, square pyramidal $M(CO)_5$ complex[92]) donating to a

vacant orbital of σ symmetry on the $Os_3(CO)_{11}$ fragment.[144] Note that both of the osmium atoms involved in the dative bond have filled d atomic orbitals of appropriate symmetry for overlap with each other (Scheme 4.1), but since this is a two-orbital, four-electron interaction it is destabilizing.[145] Formally the electronic configuration of the dative bond is thus $\sigma^2\pi^2\pi^{*2}$ (or $\sigma^2\pi^4\pi^{*4}$). If a good π-acceptor group is situated trans to the dative Os-Os bond, backbonding can occur between this ligand and the d orbitals on Os(1) that are involved in this destabilizing interaction. As shown in Scheme 4.1 (CO trans to the dative Os-Os bond), the d orbitals on the donor osmium atom are lowered in energy by this interaction. This in turn makes the d_π-d_π less destabilizing (the destabilization is proportional to the sum of the energies times the square of the overlap).[145] For the three $Os_4(CO)_{15}(L)$ clusters discussed here the longest dative bond is

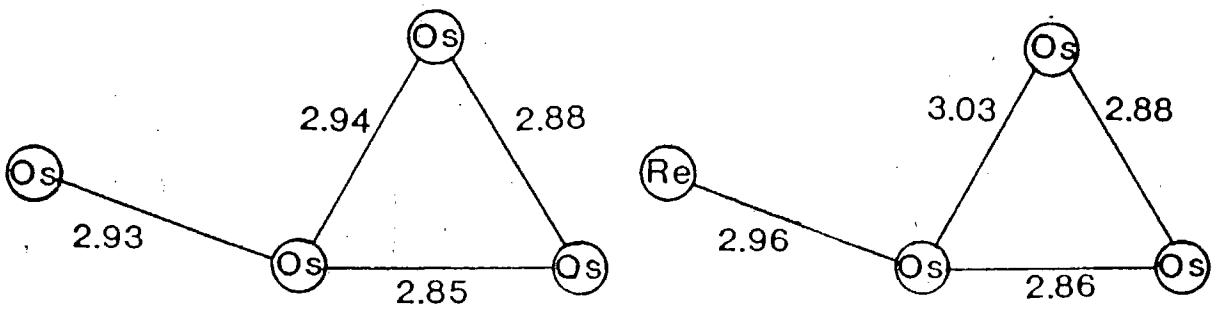
Scheme 4.1



found when the worst π-acceptor ligand (PMe_3) is in the trans position, while the shortest dative bond is found when the best π-acceptor (CO, for L = $CNBu^t$) is in this position.[139] These arguments can also be used to explain the electronic site preference of L: The better π-acceptor will want to go trans to the dative bond in order to take advantage of this π-backbonding.

As mentioned in Section 4.2, all the spiked-triangular clusters exhibit similar patterns of bond length variations in the $Os_3(CO)_{11}$ fragments. This is illustrated for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ on the left hand side of Scheme 4.2 For $Os_4(CO)_{15}(PMe_3)$ the bond lengths were rationalized as follows:[20] The Os(2)-Os(3) bond is short because it is trans to $Os(CO)_4(PMe_3)$ which is a weak donor ligand. The shortening of

Scheme 4.2



this bond causes a lengthening of the Os(2)-Os(4) bond, as observed previously in an Os_3 triangle.[133] The argument rests on the view that the 18-electron complexes are weak donor ligands. However, while it is true that they are weakly bound, this may be due to the repulsive π interaction mentioned above and thus may not reflect their σ-donor strengths. Recent work on $Os_3(CO)_{11}(L)$ derivatives in this laboratory has used ^{13}C NMR spectroscopy to quantify the σ-donor strengths of some common ligands.[69] The method correlates the donor properties of the ligands with $\Delta\delta$, defined as the difference in the chemical shifts of the resonances due to the axial carbonyls of the $Os(CO)_3(L)$ unit of $Os_3(CO)_{11}(L)$ and the resonance due to the axial carbonyls of $Os_3(CO)_{12}$ (*i.e.*, L = CO). This method has been used previously in mononuclear derivatives.[146] It has the advantage of directly measuring the electronic properties of a ligand in the molecule in question. The $\Delta\delta$ values for L = $Os(CO)_4(PMe_3)$ or $Os(CO)_4(CNBu^t)$ (15.5 ppm and 16.1 ppm, respectively) are significantly larger than those found for L = PMe_3 (9.99 ppm) PPh_3 (9.88 ppm), or $P(OMe)_3$ (6.91 ppm).[69] Therefore, if $\Delta\delta$ is truly a measure of the relative σ-donor strength of L, then these 18-electron complexes are actually stronger donors than the phosphorus ligands.

An argument based on steric considerations can also be used to rationalize the observed bond lengths. A comparison of the bond lengths in the clusters discussed here with those reported for $Os_3Re(\mu\text{-}H)(CO)_{15}(NCMe)$ (right hand side of Scheme 4.2) reveals

that the pattern of variation is the same. In the latter compound the bridging hydride was placed along the bond which is cis to the spike group ($Re(CO)_5$ in this case).[79] This probably accounts for the length of this bond (3.032 (1) Å) since it is well known that a bridging hydride causes a lengthening of a metal-metal vector.[131]

The pattern of Os-Os bond lengths in the spiked-triangular $Os_4(CO)_{15}(L)$ derivatives (and in the Os_3Re cluster) may result from interactions involving the radial ligands on the spike group. This conclusion is supported by the low temperature ^{13}C NMR spectra of $Os_4(CO)_{15}(CNBu^t)$, which suggests that there is a severe interaction between the radial ligands on Os(1) and the vicinal carbonyl on Os(4) (see below). Thus the bond cis to the spike group may be lengthened for steric reasons, which could then lead to the concomitant shortening of the bond trans to the spike group. However, in $Os_3(CO)_{11}(L')$ (L' = $[(\eta^5\text{-}C_5H_5)Mo(CO)_2]_2(\mu\text{-}Bu^tCP)$) there is a severe interaction between L' and the vicinal carbonyl on the adjacent osmium atom, but the Os-Os bond that is cis to L' (analogous to Os(2)-Os(4) in the spiked-triangular $Os_4(CO)_{15}(L)$ clusters) has a normal length.[147] This suggests that steric effects may not be responsible for the lengthening of the Os(2)-Os(4) bonds in the spiked-triangular clusters.

All of the spiked-triangular clusters discussed here (including $Os_4(CO)_{15}(PMe_3)$) are air-stable as solids but decompose in solution at room temperature. As noted above,

$Os_4(CO)_{14}(CNBu^t)_2$ seems to be more stable than the other clusters; this may be because the increased electron density placed on the donor osmium atom by the two good donor $CNBu^t$ ligands stabilizes the dative bond.

It has been reported that in solution $Os_4(CO)_{15}(PMe_3)$ decomposes to $Os_3(CO)_{12}$ and $Os_3(CO)_{11}(PMe_3)$ at room temperature after 24 h.[148] In contrast to this behavior, when CH_2Cl_2 solutions of $Os_4(CO)_{15}(L)$ (L = $CNBu^t$ or $SbPh_3$) are stirred under a nitrogen atmosphere at room temperature, the 62-electron clusters $Os_4(CO)_{14}(L)$ are observed as the major decomposition products after 20 h ($Os_4(CO)_{14}(CNBu^t)$ is known, see Chapter 3; $Os_4(CO)_{14}(SbPh_3)$ was identified by comparison of the infrared spectrum of the mixture with that of $Os_4(CO)_{14}(PMe_3)$.[20]) The carbonyl stretching region of the infrared spectra of the decomposition mixtures contained several absorption bands not assignable to $Os_4(CO)_{14}(L)$ clusters. These were possibly due to $Os_3(CO)_{11}(L)$ and $Os_3(CO)_{12}$. Since for L = PMe_3 and $CNBu^t$ the clusters $Os_4(CO)_{14}(L)$ are known to be stable under these conditions and can be produced from $Os_4(CO)_{15}(L)$ by decarbonylation, it seems that there are at least two different decomposition routes in operation. For $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ the relative intensity of the infrared bands expected for $Os_4(CO)_{14}[P(OCH_2)_3CMe]$ indicate that this is only a minor decomposition product. However, only a small scale preliminary study has been performed, and a more detailed mechanistic investigation is required to fully understand the different

processes involved. Recent work on the stability of binuclear complexes with unbridged dative metal-metal bonds indicates that in solution these complexes are sensitive to oxygen.[73] This suggests that the clusters described here may also be sensitive to oxygen.

The behavior of $Os_4(CO)_{15}(PF_3)$ in solution, as with that of $Os_4(CO)_{16}$, is best interpreted in terms of the rapid interconversion of the various puckered conformations of the ring. The ^{13}C NMR spectrum of $Os_4(CO)_{15}(PF_3)$ at -55 °C (Figure 4.9) consists of seven resonances to high field of δ 170 (the signal at δ 169.4 shows a small coupling to phosphorus), and three resonances to low field of δ 175. The resonance at δ 177.0 is a doublet due to phosphorus coupling, and the resonance at δ 176.5 is comprised of two overlapping signals. The latter resonance is probably due to the near accidental degeneracy of two resonances. From the chemical shifts of the signals the seven resonances of approximate relative intensity one can be assigned to the equatorial carbonyl ligands and the three other resonances to the axial carbonyl ligands. The equivalence of the signals due to the inner and outer axial carbonyls even in the spectrum at -122 °C suggests that ring inversion is rapid on the NMR time scale down to this temperature. Because of the rapid decomposition of the cluster in solution the spectra of $Os_4(CO)_{15}(PF_3)$ at temperatures above -55 ° were not recorded.

When stirred at room temperature in hexane (or CH_2Cl_2) under a nitrogen atmosphere $Os_4(CO)_{15}(PF_3)$ decomposed rapidly. The

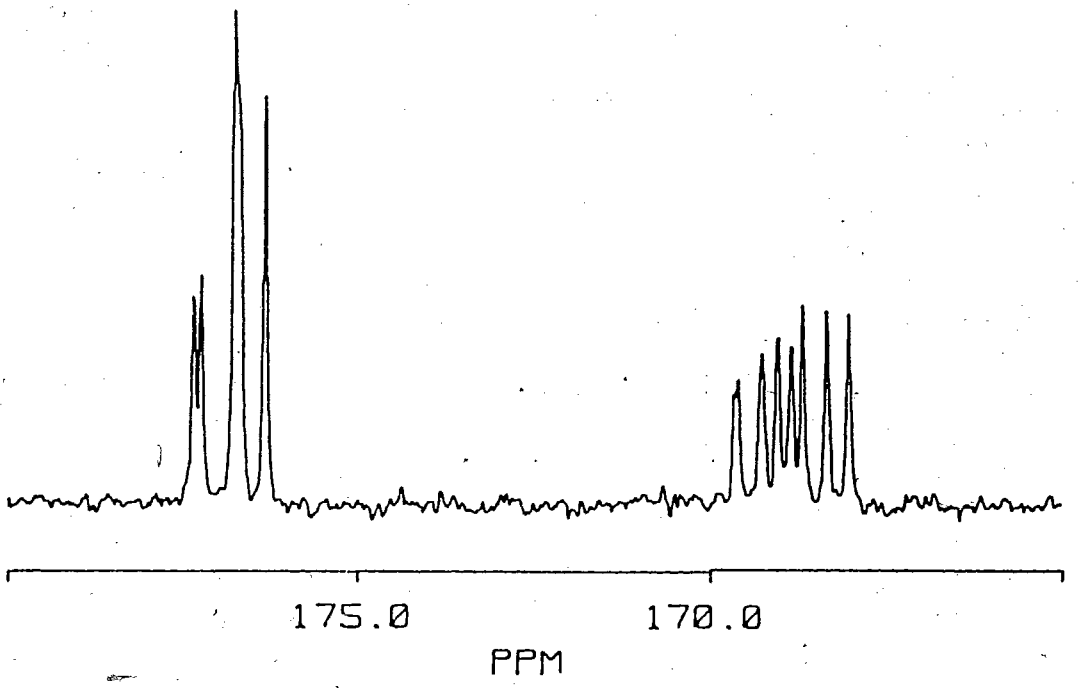


Figure 4.9. ^{13}C **NMR Spectrum of** $Os_4(CO)_{15}(PF_3)$ **in** $CHFCl_2/CD_2Cl_2$ **at -55 °C.**

infrared spectrum of the resulting solution was consistent with the presence of both $Os_3(CO)_{12}$ and $Os_3(CO)_{11}(PF_3)$,[69] but because of the overlapping of the carbonyl stretching bands this assignment was ambiguous. Therefore, a ^{13}CO-enriched sample of $Os_4(CO)_{15}(PF_3)$ in CD_2Cl_2/CH_2Cl_2 was allowed to stand for four hours at room temperature, and then the ^{13}C NMR spectrum was recorded at -55 °C. As expected, the spectrum (Figure 4.10) contains signals due to both $Os_3(CO)_{12}$ and $Os_3(CO)_{11}(PF_3)$ in approximately equal amounts.[149] Surprisingly, the most intense resonances in the spectrum, at δ 176.5 and δ 168.8, correspond to the signals of the carbonyls of $Os_4(CO)_{16}$. That this cluster is formed on the decomposition of $Os_4(CO)_{15}(PF_3)$ is truly

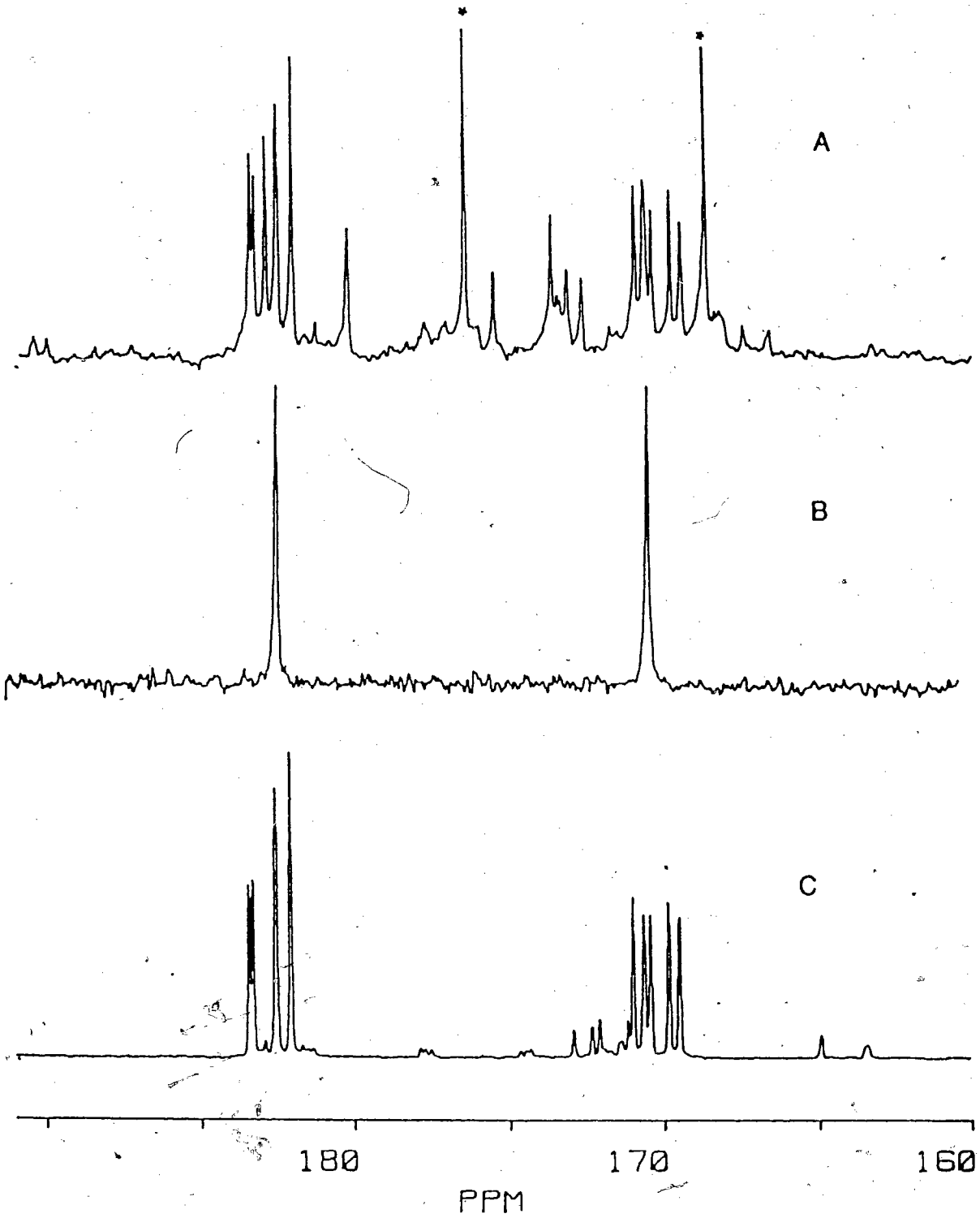
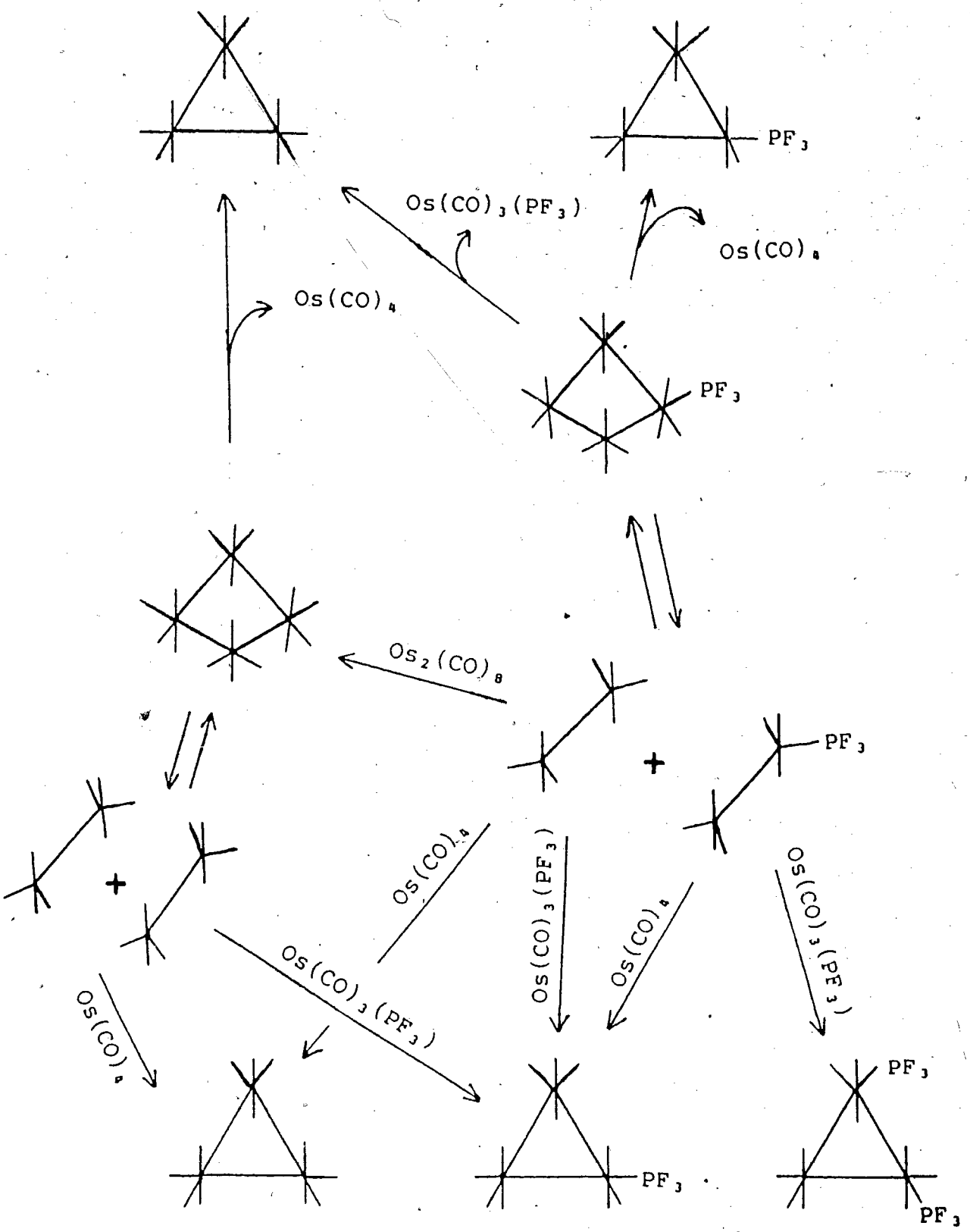


Figure 4.10. ^{13}C NMR Spectra of the Decomposition Products of $Os_4(CO)_{15}(PF_3)$ (A), $Os_3(CO)_{12}$ (B), and $Os_3(CO)_{11}(PF_3)$ (C) (all in CH_2Cl_2/CD_2Cl_2). *Signals due to $Os_4(CO)_{16}$.

remarkable. A possible mechanism for this process is shown is Scheme 4.3. A note of caution is called for here: the proposed scheme has no experimental support beyond that it accounts for the identified products. However, it does have precedent in the literature. Formal 2+2 cycloadditions involving $Os_2(CO)_8$ (isolobal with ethylene) are known to occur.[150] In addition, the decomposition of $Os_2(CO)_9$ to $Os_3(CO)_{12}$, originally thought by Graham to proceed via an $Os_2(CO)_8$ intermediate,[37] may in fact proceed through $Os_4(CO)_{16}$ since the infrared CO-stretches reported for the intermediate correspond to those of $Os_4(CO)_{16}$. Recent work supports this conclusion. The infrared spectrum of $Os_2(CO)_8$ in a matrix and by time resolved spectroscopy are inconsistent with the bands reported for Graham's intermediate.[151] The proposed dissociation of $Os_4(CO)_{15}(PF_3)$ (and $Os_4(CO)_{16}$) into two diosmium ethylene analogues also has precedent. Chisholm has reported that an equilibrium exists between two $(RO)_3W{\equiv}W(OR)_3$ ($R = Bu^t$) complexes and their dimer, $[W(OR)_3]_4$, which is an inorganic analog of cyclobutadiene.[152]

As shown in Scheme 4.3, the transient $Os_2(CO)_8$ or $Os_2(CO)_7(PF_3)$ species, can either dimerize, react with each other, or they may react with $Os(CO)_4$ or $Os(CO)_3(PF_3)$ The latter reactions are analogous to the reaction of ethylene with methylene, and also has precedent in cluster chemistry.[153] If such processes are occurring, one would also expect the formation of $Os_3(CO)_{10}(PF_3)_2$ from the decomposition of $Os_4(CO)_{15}(PF_3)$. There are extra signals in the ^{13}C NMR spectrum

Scheme 4.3


PF3
Os(CO)3(PF3)
Os(CO)4
Os(CO)4
PF3
Os2(CO)8
PF3
Os(CO)4
Os(CO)3(PF3)
Os(CO)4
Os(CO)3(PF3)
Os(CO)3(PF3)
Os(CO)4
PF3
PF3
PF3

of the decomposition mixture, but these cannot be confidently assigned to the carbonyls of $Os_3(CO)_{10}(PF_3)_2$.

Whatever the decomposition route of $Os_4(CO)_{16}$ and $Os_4(CO)_{15}(PF_3)$, there was no evidence for spiked-triangular isomers of these clusters in solution. Indeed, these compounds are prepared in the presence of excess CO or PF_3, and since the spiked-triangular $Os_4(CO)_{15}(L)$ clusters decompose in the presence of excess L, the puckered-square would seem to be the only isomer of $Os_4(CO)_{15}(L)$ (L = CO or PF_3) present in solution.

Fluxional Properties of $Os_4(CO)_{16-x}(CNBu^t)_x$ (x = 1, 2)

$Os_4(CO)_{15}(CNBu^t)$. The behavior of $Os_4(CO)_{15}(PMe_3)$ in solution has been thoroughly investigated.[20] No evidence was found for the isomerization of this spiked-triangle cluster to the puckered-square geometry. In order to determine if the same was true for $Os_4(CO)_{15}(CNBu^t)$, variable temperature ^{13}C NMR spectra were obtained on a ^{13}CO-enriched sample. These experiments were carried out in collaboration with Mr. A. K. Ma of our group. Therefore, details of the NMR experiments are presented elsewhere,[47] and only the results are discussed here.

The ^{13}C NMR spectrum of $Os_4(CO)_{15}(CNBu^t)$ in $CHFCl_2/CD_2Cl_2$ at -118 °C is shown in Figure 4.11. The carbonyl region consists of 14 resonances each of relative intensity one except for the resonance at δ 184.6 which has an intensity of two. The spectrum

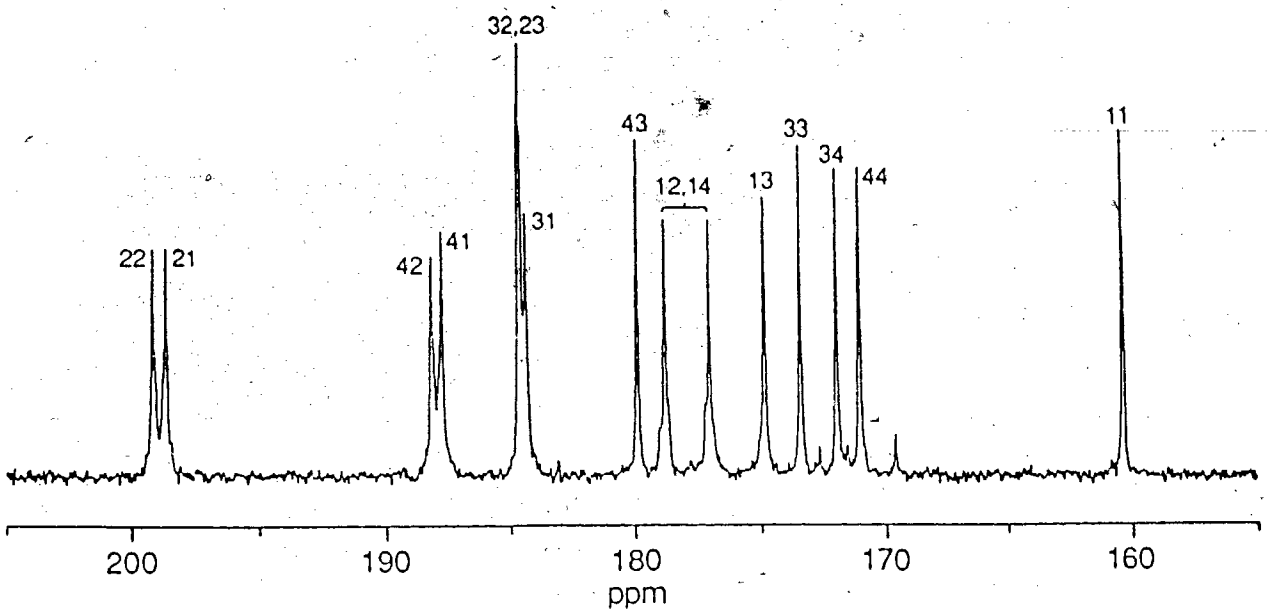


Figure 4.11. ^{13}C NMR Spectrum of $Os_4(CO)_{15}(CNBu^t)$ (in $CHFCl_2/CD_2Cl_2$) at -118 °C.

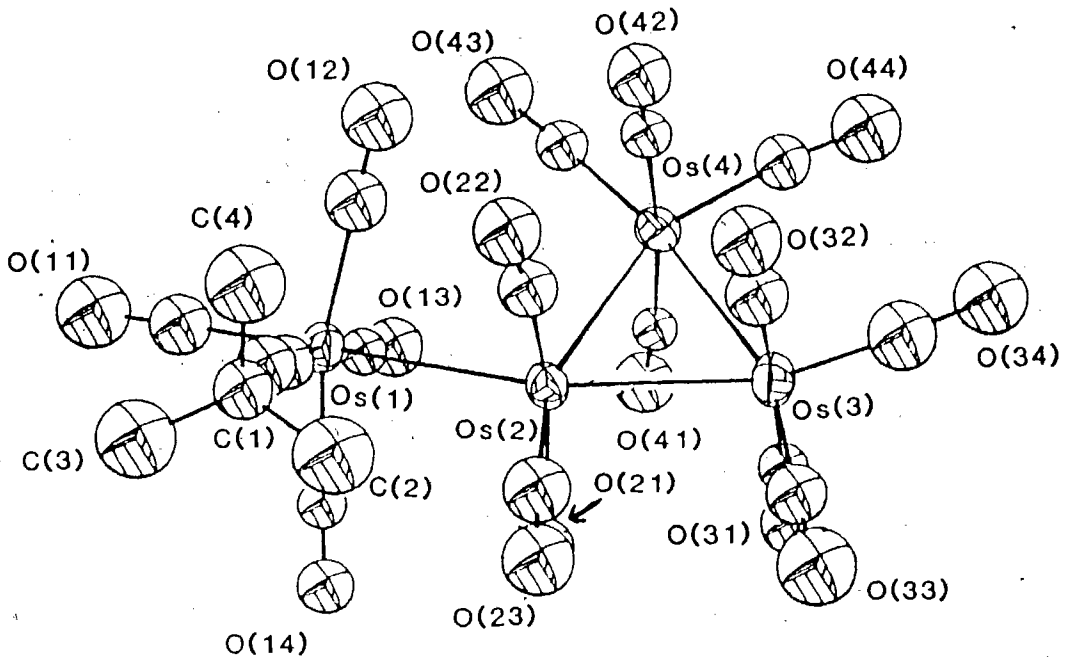


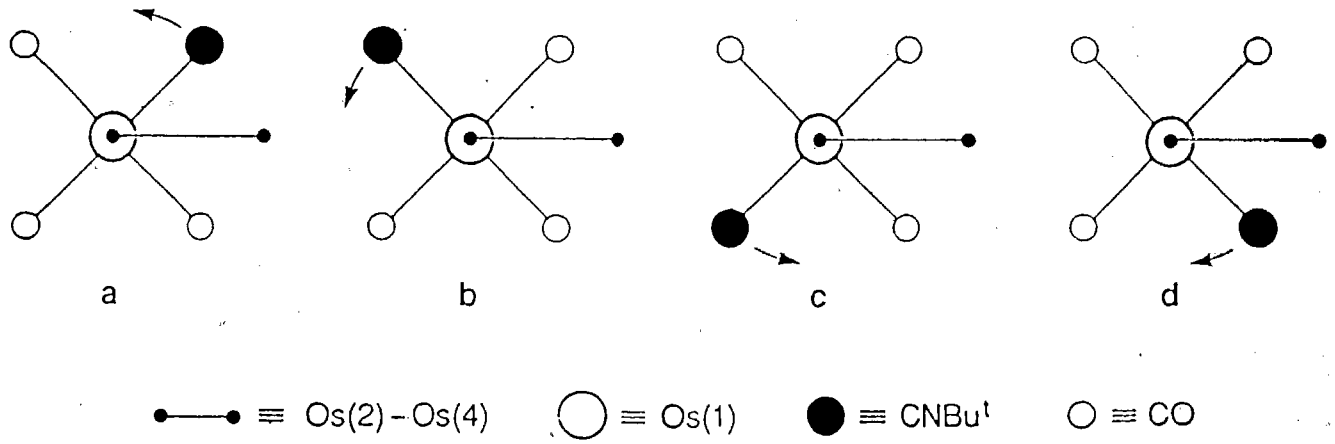
Figure 4.12. Molecular Structure of $Os_4(CO)_{15}(CNBu^t)$.

at -108 °C (not shown) reveals that this signal is comprised of two resonances. Fifteen equal intensity resonances are consistent with the solid state structure of $Os_4(CO)_{15}(CNBu^t)$ (Figure 4.12). The assignments of the signals in the spectrum at -118 °C is based upon the following: the resonances due to axial carbonyls come to lower field than those due to equatorial carbonyls on the same osmium atom;[68,154] the resonances due to carbonyls belonging to $Os(CO)_3(L)$ groups come to lower field than those on $Os(CO)_4$ groups as in triosmium clusters;[68,154] for axial carbonyls attached to the same osmium atom the one nearest to the $CNBu^t$ ligand gives rise to the resonance further downfield;[102] the resonance of a carbonyl with a chemically different carbonyl trans to it is expected to show C-C coupling;[134,155] the resonance of a CO which is trans to a dative Os-Os bond comes at a characteristically high field;[18,19,156] the mode of collapse of the signals at higher temperatures in conjunction with the proposed mechanisms for CO-exchange (see below). The labelling of the signals follows the scheme used in Figure 4.11.

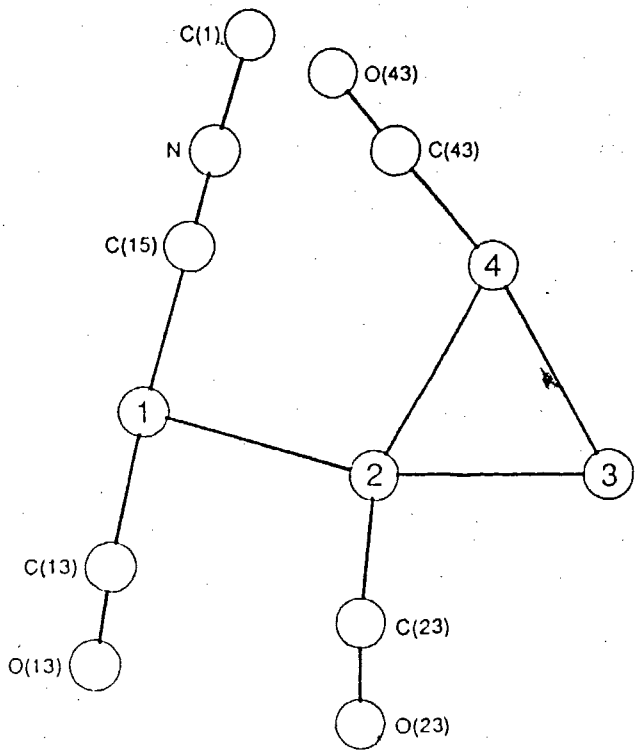
That the spectrum obtained at -118 °C is consistent with the crystal structure of $Os_4(CO)_{15}(CNBu^t)$ is remarkable. Fifteen signals can only be observed if there is no rotation (or oscillation) about the dative Os(1)-Os(2) bond. Scheme 4.4 illustrates the four possible staggered conformations that result from rotation about this bond. As shown in Scheme 4.5, there is a severe steric interaction between the isocyanide

ligand and the vicinal carbonyl group on Os(4) (*i.e.*, CO(43)) when the Os(1)-Os(2) bond is rotated. It is easy to see that this barrier will prevent the direct conversion of conformers **a** and **d** in Scheme 4.4, but this is insufficient to account for the observed low temperature spectrum of $Os_4(CO)_{15}(CNBu^t)$. Rapid oscillation about the Os(1)-Os(2) bond via conformers **b** and **c** would also result in pairs of carbonyls above and below the plane of the osmium atoms becoming equivalent (*e.g.*, in Figure 4.12 CO(21) is rendered equivalent to CO(22)). Therefore, if the barrier to oscillation is steric in origin, there must be a second point of severe interaction. Models show that the only interactions expected to cause a significant barrier are between CO(43) and any of the three carbonyls on Os(1) that are cis to the dative bond. This interaction is shown in Scheme 4.6 for the CO group that is trans to the $CNBu^t$ ligand.

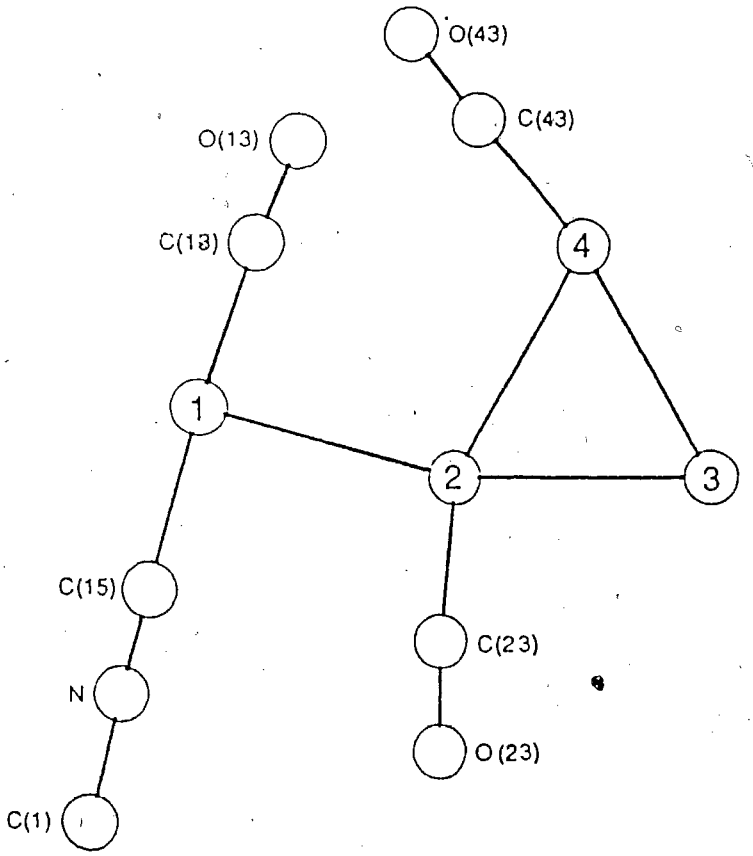
Scheme 4.4



Scheme 4.5



Scheme 4.6



If the interaction shown in Scheme 4.6 is responsible for the barrier to oscillation then it is expected that a similar barrier would also exist for any spiked-triangular $Os_4(CO)_{15}(L)$ cluster. This was not observed in the previous study of the nonrigidity in $Os_4(CO)_{15}(PMe_3)$, but the lowest temperature investigated for this compound was -67 °C.[20] Therefore, a fresh sample of ^{13}CO-enriched $Os_4(CO)_{15}(PMe_3)$ was prepared, and the ^{13}C NMR spectrum recorded at -90 °C. The results were inconclusive, but the signal assigned to the radial carbonyls of the $Os(CO)_4(PMe_3)$ unit was slightly broadened, possibly indicating restricted rotation about the dative Os-Os bond in this cluster. The spectrum of $Os_4(CO)_{15}(PMe_3)$ at lower temperatures could not be obtained due to the low solubility of this cluster. It might be expected that the barrier to rotation about the dative Os-Os bond would be lower in $Os_4(CO)_{15}(PMe_3)$ than in $Os_4(CO)_{15}(CNBu^t)$ because this bond is longer in the former compound.

Upon warming the sample of $Os_4(CO)_{15}(CNBu^t)$ from -110 °C to -50 °C four pairs of signals coalesce to singlets (Figure 4.13). This is consistent with the onset of oscillation about the Os(1)-Os(2) bond in the cluster. Simulation of the spectrum at -85 ± 2 °C (Figure 4.14) gave a rate constant of 350 ± 35 s^{-1} corresponding to a $\Delta G^{\ddagger}$ = 8.7 ± 0.2 kcal mol^{-1}. The only other examples of restricted rotation about transition-metal, transition-metal single bonds in the literature (that we are aware of) are those in molecules of the type $[(\eta^5-C_5H_5)M(CO)_3]_2$

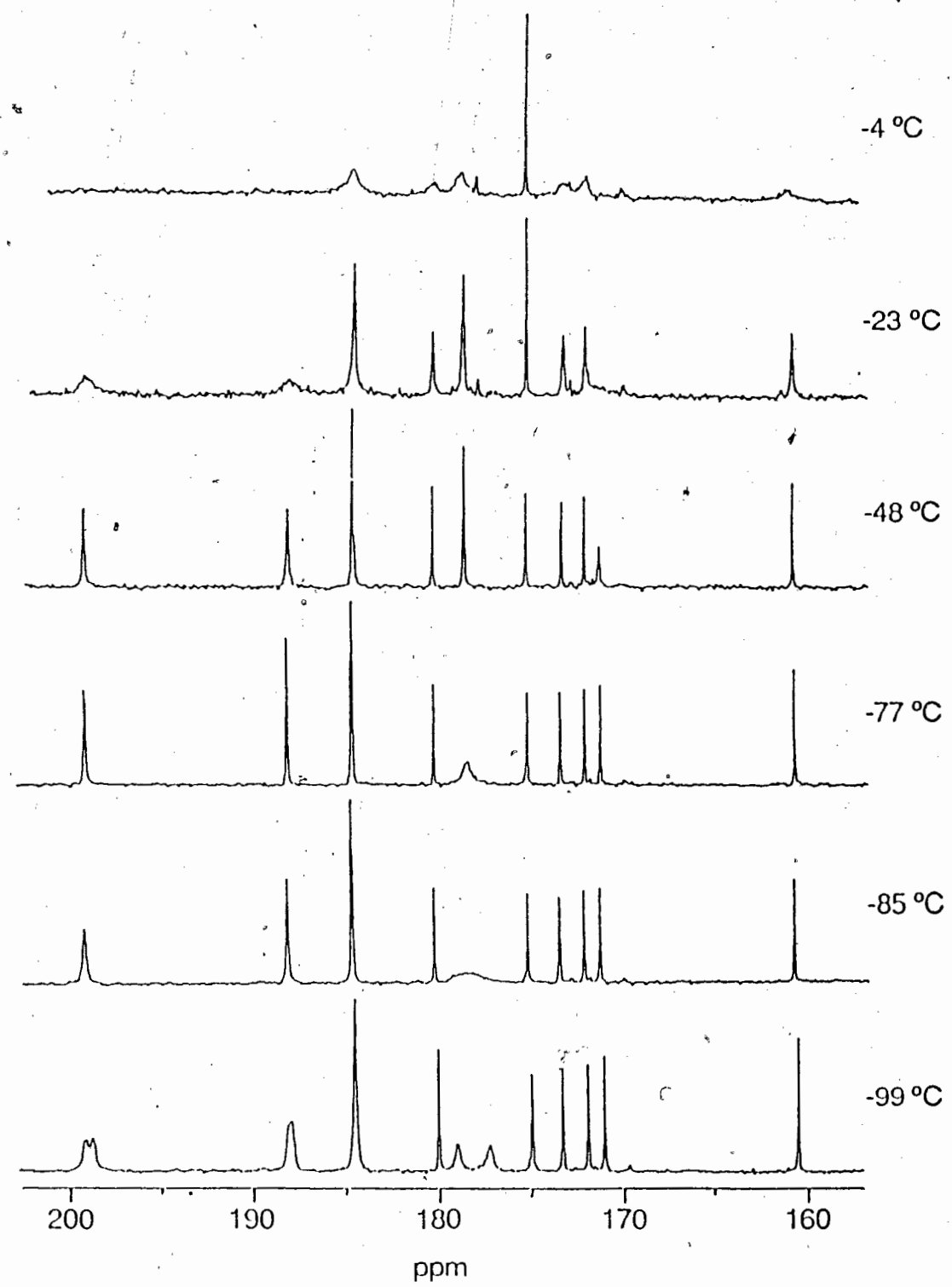


Figure 4.13. Variable Temperature ^{13}C NMR Spectra of $Os_4(CO)_{15}(CNBu^t)$.

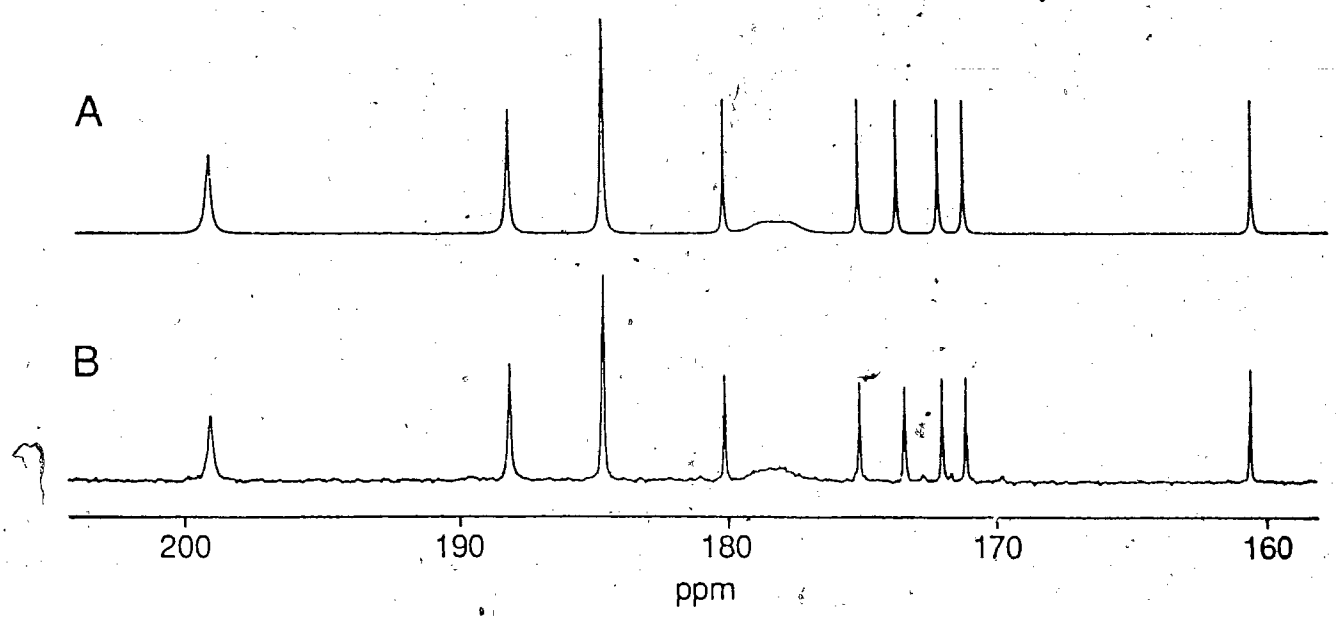


Figure 4.14. Simulated (A) and Experimental (B) ^{13}C NMR Spectra of $Os_4(CO)_{15}(CNBu^t)$ at -85 °C.

(M = Cr, Mo, W) and $(\eta^5\text{-}C_5H_5)_2Mo_2(CO)_5(CNMe)$ observed by Cotton and Adams.[157]

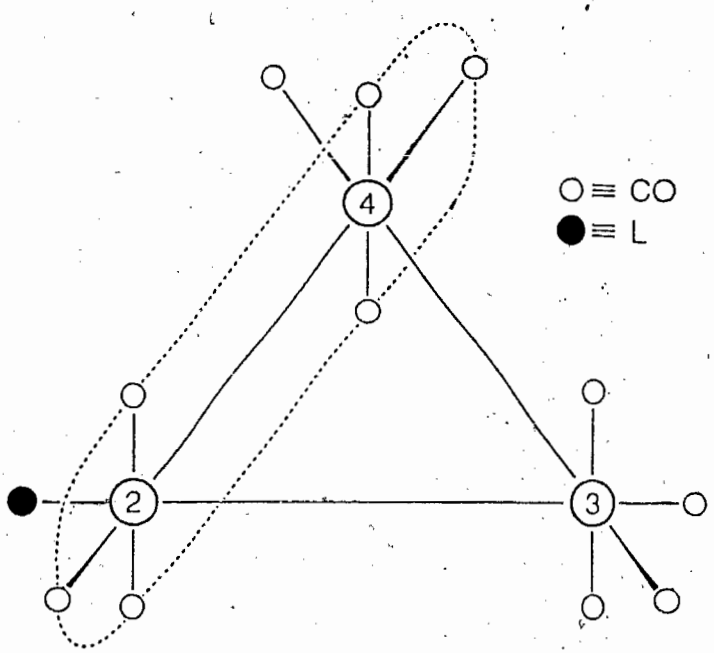
From about -50 °C to -20 °C two signals assigned to axial CO groups and two signals assigned to equatorial CO groups of the $Os_3(CO)_{11}$ fragment collapse to the base line (Figure 4.13) (all three carbonyl ligands on Os(2) take part in this exchange process). This pattern of collapse has been observed previously in $Os_4(CO)_{15}(PMe_3)$[20] and in $Os_3(CO)_{11}(L)$ derivatives (L = donor ligand).[68-70] It has been rationalized in terms of terminal-bridge CO-exchange in the axial plane that is cis-perpendicular to L as shown in Scheme 4.7 (L =

$Os(CO)_4(CNBu^t)$).

Simulation of the spectrum at -50 °C is complicated by the necessity of including the rate constant (k_1) for oscillation about the Os(1)-Os(2) bond along with the rate constant (k_2) for the CO-exchange shown in Scheme 4.7. Good agreement between the experimental and calculated spectra is obtained with k_1 = 37 000 ± 3 700 s^{-1} and k_2 = 11 ± 1 s^{-1}. This gives a value of 12.0 ± 0.3 kcal mol^{-1} for $\Delta G^{\ddagger}$ of the exchange process.

Between -20 °C and -4 °C all remaining signals except that at δ 174.9 collapsed to the base line (Figure 4.13). (Spectra at higher temperatures were not recorded because of the decomposition of $Os_4(CO)_{15}(CNBu^t)$ in solution at these temperatures.) Such behavior is inconsistent with any single exchange process, but can be rationalized in terms of two mechanisms if they have approximately the same activation energy. These mechanisms also involve terminal-bridge carbonyl exchange, this time in the two other axial planes that contain two osmium atoms of the $Os_3(CO)_{11}$ fragment. Three of the carbonyls on Os(1) (that which was initally trans to the dative bond and the two that were cis to the dative bond and cis to the $CNBu^t$ group) take part in the exchange in the axial plane that contains Os(2) and Os(3); this process is shown in Scheme 4.8. The second process occurring at this temperature is the exchange of the carbonyls in the axial plane containing Os(3) and Os(4) (Scheme 4.9, L = $Os(CO)_4(CNBu^t)$. Again, similar mechanisms have been proposed for other $Os_3(CO)_{11}(L)$ derivatives.[68,69] The

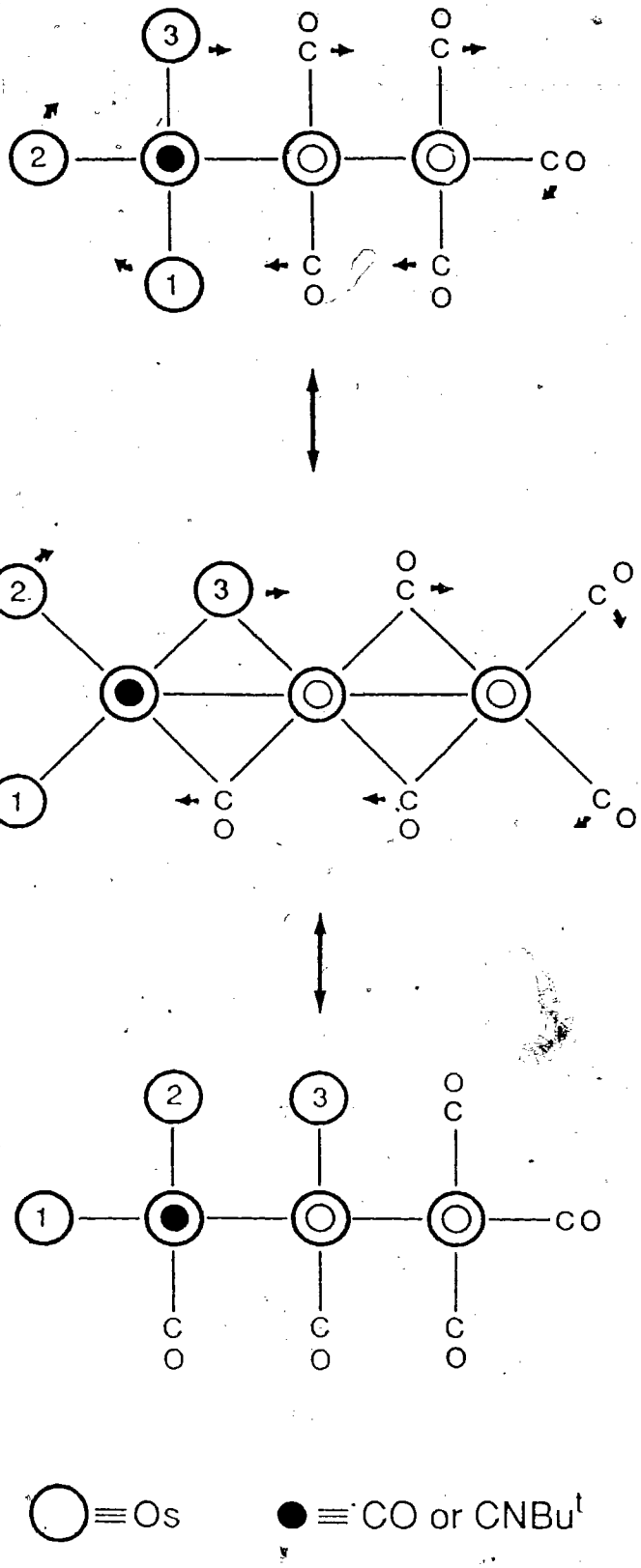
Scheme 4.7



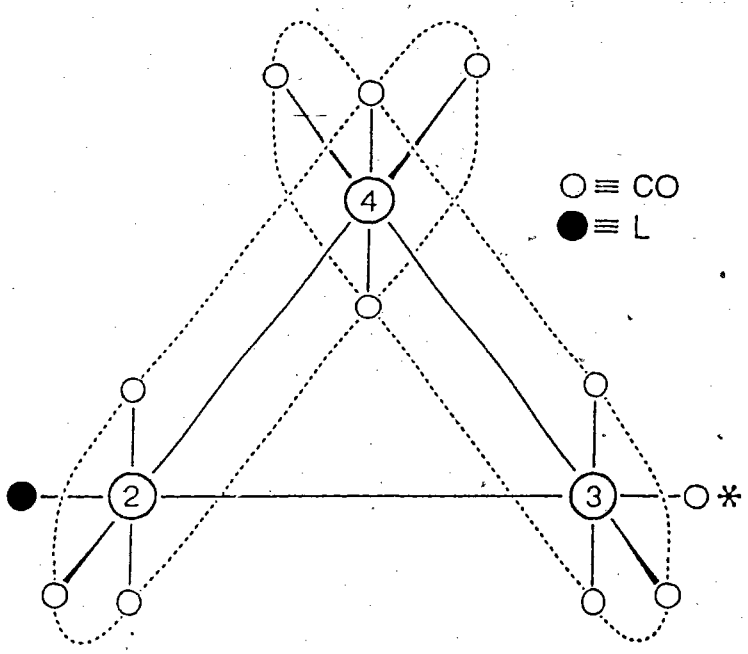
single sharp resonance in the spectrum at -4 °C is therefore assigned to CO(13), the carbonyl on Os(1) that is trans to the $CNBu^t$ ligand.

A similar pair of mechanisms was proposed for $Os_4(CO)_{15}(PMe_3)$, but since the PMe_3 ligand was originally trans to the dative Os-Os bond isomerization must occur along with the terminal-bridge CO exchange.[20] This isomerization allows all the carbonyls on the donor osmium atom to exchange, so that in the room temperature spectrum all the signals had collapsed to the base line.

Scheme 4.8

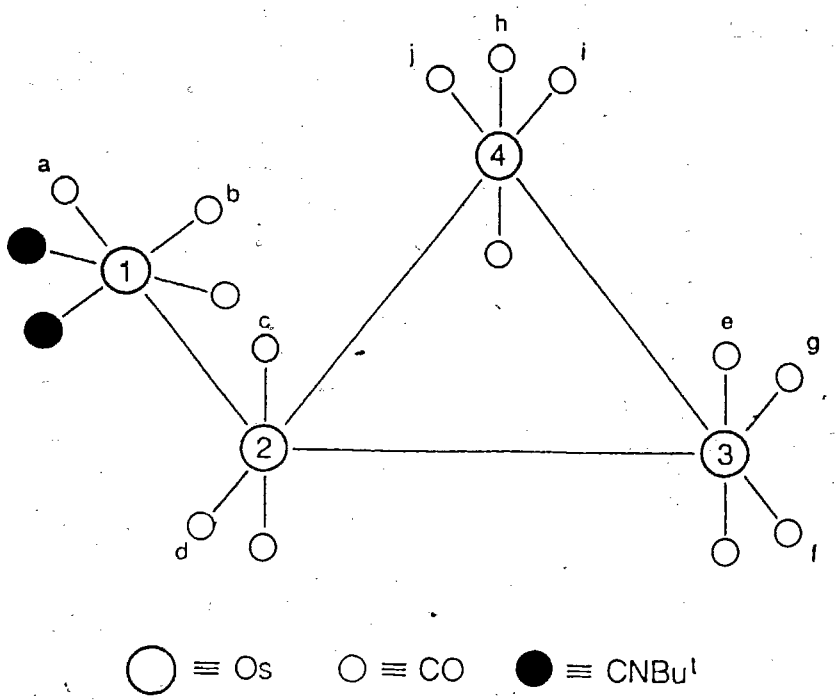


Scheme 4.9



$Os_4(CO)_{14}(CNBu^t)_2$. The cluster $Os_4(CO)_{14}(CNBu^t)_2$ was prepared by the addition of two equivalents of $CNBu^t$ to $Os_4(CO)_{14}$ in CH_2Cl_2. Although this compound does not have the general formula $Os_4(CO)_{15}(L)$, it is included here because of its close relationship to $Os_4(CO)_{15}(CNBu^t)$. The crystal structure of $Os_4(CO)_{14}(CNBu^t)_2$ has not been determined, but the structure shown in Scheme 4.10 is consistent with the spectroscopic evidence. In particular, strong support for the proposed structure is found in the low temperature ^{13}C NMR spectra of $Os_4(CO)_{14}(CNBu^t)_2$ discussed below. The cis arrangement of the $CNBu^t$ groups may be favored for steric reasons since it avoids the interaction between these ligands and the vicinal CO on Os(4) (carbonyl j in Scheme 4.10). It may also be favored for electronic reasons because it allows the better π-acceptor

Scheme 4.10



ligand (CO) to be in the position trans to the dative osmium-osmium bond (see above).

The ^{13}C NMR spectrum of $Os_4(CO)_{14}(CNBu^t)_2$ (^{13}CO-enriched) in CD_2Cl_2/CH_2Cl_2 at -101 °C is shown in Figure 4.15. The assignment of the signals follows that for $Os_4(CO)_{15}(CNBu^t)$, and the labelling is as in Scheme 4.10. The assignment of resonances labelled f, g, and j is not as definitive as the assignment of the other signals. It is based on the assumption that peaks due to carbonyls in similar chemical environments in $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$ should have similar chemical shifts.

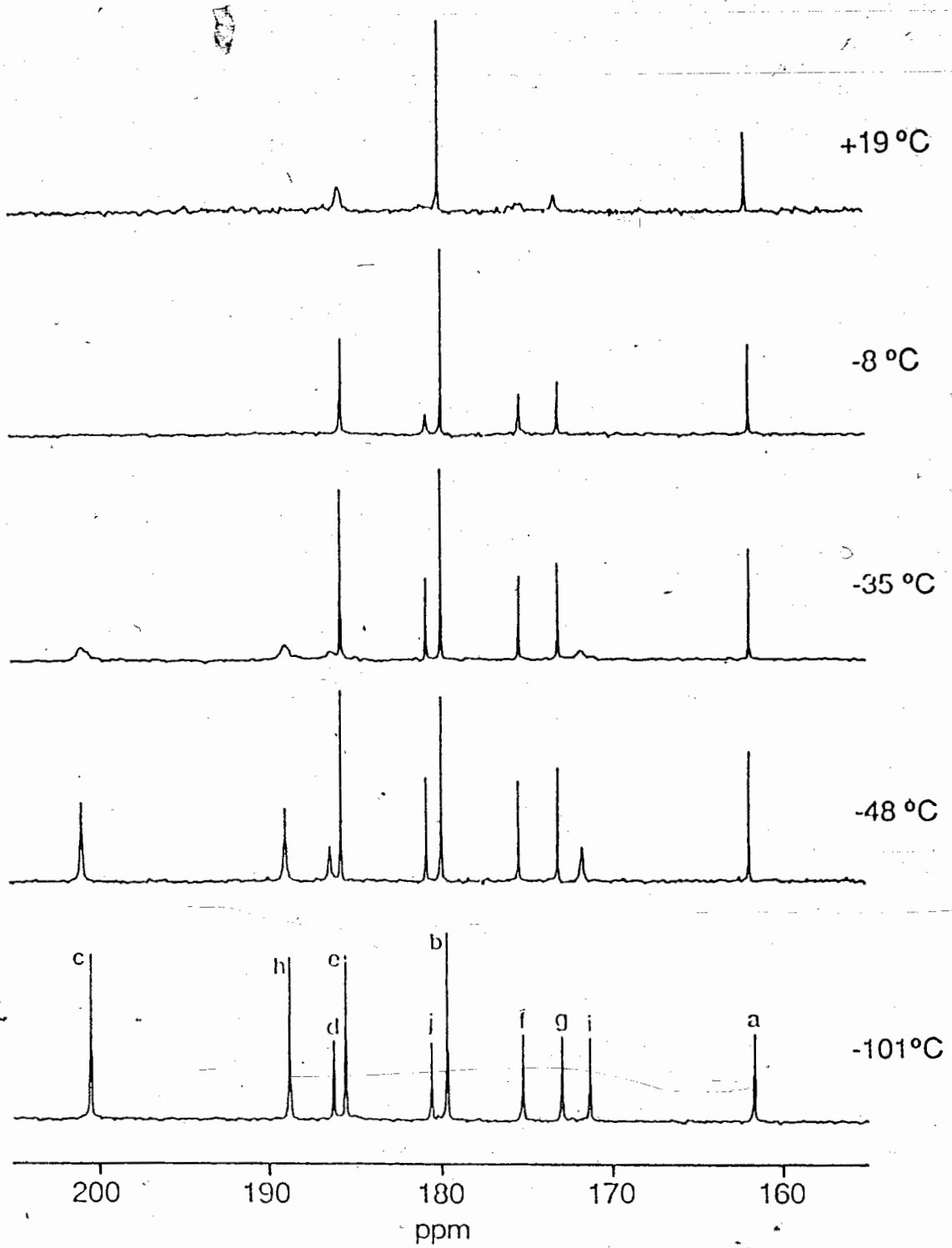


Figure 4.15. Variable Temperature ^{13}C NMR Spectra of $Os_4(CO)_{14}(CNBu^t)_2$.

Because of the cis arrangement of the $CNBu^t$ groups on the $Os(CO)_3(CNBu^t)_2$ unit, it is impossible to detect if there is restricted rotation (or oscillation) about the dative Os-Os bond. It is expected that the barrier to rotation in $Os_4(CO)_{14}(CNBu^t)_2$ would be at least as large as that in $Os_4(CO)_{15}(CNBu^t)$.

Upon warming the solution of $Os_4(CO)_{14}(CNBu^t)_2$ to above -60 °C, the signals labelled c, d, h, and i collapsed to the base line. As discussed above for $Os_4(CO)_{15}(CNBu^t)$, this behavior is common to many $Os_3(CO)_{11}(L)$ derivatives, and is interpreted in terms of terminal-bridge carbonyl exchange in the axial plane containing Os(2) and Os(4) (see Scheme 4.7, L = $Os(CO)_3(CNBu^t)_2$). Simulation of the spectrum at -48 ± 2 °C gave a first order rate constant of 14 ± 2 s^{-1} and hence $\Delta G^{\ddagger}$ = 11.9 ± 0.2 kcal mol^{-1}.

Warming the sample of $Os_4(CO)_{14}(CNBu^t)_2$ to above -10 °C caused four more of the signals to broaden and collapse (Figure 4.15). The two remaining resonances have relative intensity 2:1, and are confidently assigned to the three carbonyls on Os(1). That these signals remain sharp in the spectrum at room temperature indicates that the $CNBu^t$ groups do not migrate into the position trans to the dative Os-Os bond, nor do they bridge this bond. These results suggest that there is a strong electronic preference for the $CNBu^t$ groups to remain in the position cis to the dative Os-Os bond. This conclusion must also be true for the $CNBu^t$ group in $Os_4(CO)_{15}(CNBu^t)$, since the resonance due to the

carbonyl trans to this group remains sharp in the spectrum at 0 °C.

Finally, from Figure 4.15 it is apparent that all the carbonyls of the $Os_3(CO)_{11}$ unit in $Os_4(CO)_{14}(CNBu^t)_2$ undergo exchange at 19 °C. Terminal-bridge exchange in the perpendicular planes that contain Os(3) and Os(4) (Scheme 4.9, L = $Os(CO)_3(CNBu^t)_2$) can account for the collapse of signals e, f, and j, but there must be another process to account for the collapse of signal g (marked with an asterisk in Scheme 4.9). This may be a three-fold twist at Os(3) that exchanges carbonyls e, f, g. Alternately, this process could be occurring at Os(2), with the restriction that the $Os(CO)_3(CNBu^t)_2$ group not move into an axial position. This type of process has been previously proposed for $Os_3(CO)_{12-x}[P(OMe)_3]_x$ (x = 1 - 5).[68]

There is a strong similarity between the fluxional properties of $Os_4(CO)_{15}(CNBu^t)$ and $Os_4(CO)_{14}(CNBu^t)_2$ (and $Os_4(CO)_{15}(PMe_3)$ and those of $Os_3(CO)_{11}(L)$ derivatives (L = donor ligand). Certain aspects of the reaction chemistry of these 64-electron clusters are also reminiscent of the triosmium derivatives. This is particularly noticeable in the preparation of $Os_4(CO)_{15}(PMe_3)$ by the reaction of $Os(CO)_4(PMe_3)$ with $Os_3(CO)_{11}(MeCN)$,[20] and in the displacement of the $Os(CO)_4(L)$ groups by other ligands. The conclusion to be drawn is simply that these 18-electron complexes truly behave as ligands towards the $Os_3(CO)_{11}$ fragment in much the same way as, for example, PMe_3.

4.4 Experimental

4.4.1 Syntheses

Preparation of $Os_4(CO)_{15}(CNBu^t)$

In a Schlenk tube, $Os_4(CO)_{15}$ (73 mg, 0.062 mmol) in CH_2Cl_2 (25 mL) was stirred at 0 °C. A hexane solution of $CNBu^t$ ($\simeq$ 1 mg mL^{-1}) was slowly added dropwise until the solution had turned from dark green to bright orange. The infrared spectrum of the solution at this point showed no bands due to $Os_4(CO)_{15}$. The solvent and any excess $CNBu^t$ were removed on the vacuum line, and the yellow-orange residue was recrystallized from dichloromethane/hexane (1/1) at -15 °C; isolated yield 69 mg, 88%, $Os_4(CO)_{15}(CNBu^t)$: mp 118 °C; IR (CH_2Cl_2) ν(CN) 2222.5(m); ν(CO) 2120(m), 2088(s), 2058(sh), 2049(vs), 2041(sh), 2008(vs), 1974(sh), 1962(sh), 1918(m, br) cm^{-1}; ^1H NMR (C_6D_6, ambient temperature) δ 0.78 (Bu^t); ^{13}C NMR ($CHFCl_2/CD_2Cl_2$, 6/1, -118 °C) δ 199.2, 198.7, 188.2, 187.8, 184.6 (2C), 184.3, 179.9, 178.8, 177.0, 174.9, 173.4, 171.9, 171.0, 160.4; The compound did not give a satisfactory (EI) mass spectrum; Anal. Calcd for $H_9C_{20}NO_{15}Os_4$: C, 19.00; N, 1.11; H, 0.72. Found: C, 19.29; N, 1.09, H, 0.82.

Preparation of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$

A Schlenk tube was charged with $Os_4(CO)_{15}$ (51 mg, 0.043 mmol) and CH_2Cl_2 (20 mL) and the solution was stirred at 0 °C. A dichloromethane solution of $P(OCH_2)_3CMe$ was added dropwise at

about 5 min intervals. The progress of the reaction was monitored by observing the disappearance of bands due to $Os_4(CO)_{15}$ in the infrared spectrum. The solution turned from dark green to amber. Upon completion of the reaction (as judged by the infrared spectrum) the solvent was removed on the vacuum line at 0 °C, and the yellow residue recrystallized from dichloromethane/hexane at ≃ -50 °C (MeCN/CO_2 cold bath). Decanting of the supernatant solution and drying under vacuum afforded crystalline $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ (21 mg, 37%): mp 80 °C (blackens), 125 °C (melts); IR (CH_2Cl_2) ν(CO) 2116(w), 2090(s), 2066(vs), 2058(sh), 2051(s), 2039(s), 2027.5(s), 2007.5(s), 1985.5(sh), 1960(sh), 1917(m,br) cm^{-1}; The mass spectrum was not recorded; Anal. Calcd for $H_9C_{20}O_{18}POs_4$: C, 18.07; H, 0.68. Found: C, 18.32; H, 0.82.

Preparation of $Os_4(CO)_{15}(PF_3)$

A 125 mL round bottom flask fitted with a Teflon valve was charged with $Os_4(CO)_{15}$ (105 mg, 0.089 mmol), CH_2Cl_2 (25 mL), and a stir bar. The solution was degassed by three freeze-pump-thaw cycles. Approximately one atmosphere of PF_3 was admitted to the flask, and the solution stirred in a cold bath at -22 °C for 30 h. The temperature of the bath varied by approximately 5 °C over the course of the reaction, but at no time was it allowed to rise above -17 °C. The color of the solution slowly changed from dark green to amber. Upon completion of the reaction, the solvent and excess PF_3 were removed on the vacuum line at -20 °C, and the orange residue was recrystallized from

dichloromethane/hexane (1/1) at -50 °C to yield $Os_4(CO)_{15}(PF_3)$ (22 mg, 20%). The isolated yield was low due to the decomposition of the product during workup. IR (CH_2Cl_2) ν(CO) 2125(w), 2077(vs), 2067(s), 2048.5(s), 2033.5(s), 2020(m), 1998.5(m) cm^{-1} (due to the rapid decomposition of $Os_4(CO)_{15}(PF_3)$ in solution it is possible that some of the bands in the spectrum are due to decomposition products); ^{13}C NMR ($CHFCl_2/CD_2Cl_2$ 6/1, -55 °C) δ 177.0 (2C, d, J_{P-C} = 10 Hz), 176.5 (4C), 176.0 (2C), 169.4, 169.0, 168.8, 168.6, 168.4, 168.0, 167.8; the mass spectrum was not recorded; Anal. Calcd for $C_{15}O_{15}F_3POs_4$: C, 14.20; H, 0.0. Found: C, 14.25; H, 0.0.

Preparation of $Os_4(CO)_{14}(CNBu^t)_2$

A solution of $Os_4(CO)_{14}$ (11 mg, 0.010 mmol) in dichloromethane (15 mL) was stirred at room temperature under a nitrogen atmosphere. A hexane solution of $CNBu^t$ (approximately 1 mg mL^{-1}) was added slowly added until the solution turned from red-brown to yellow. The infrared spectrum of the solution at this point contained no bands due to $Os_4(CO)_{14}$, nor were there any bands due to $Os_4(CO)_{14}(CNBu^t)$. The solvent and any excess $CNBu^t$ were removed on the vacuum line, and the product, $Os_4(CO)_{14}(CNBu^t)_2$ (8 mg, 63%), recrystallized from dichloromethane/hexane at -15 °C: mp 148 °C (dec); IR (CH_2Cl_2) ν(CN) 2220.5(m); ν(CO) 2095.5(m), 2067(s), 2039(s), 2000.5(s), 1967.5(sh), 1956(sh), 1906(m, br) cm^{-1}; ^1H NMR (C_7D_9, 25 °C) δ 0.94 (Bu^t); ^{13}C NMR (CD_2Cl_2/CH_2Cl_2 1/5, -101 °C) δ 200.4 (2C), 188.7 (2C), 186.2, 185.5 (2C), 180.5, 179.6 (2C), 175.2, 172.9, 171.3, 161.7; A

satisfactory (EI) mass spectrum could not be obtained; Anal. Calcd for $H_{18}C_{24}N_2O_{14}Os_4$: C, 21.85; N, 2.12; H, 1.38. Found: C, 21.80; N, 1.95; H, 1.57.

Preparation of $Os_4(CO)_{15}(SbPh_3)$

A solution of $Os_4(CO)_{15}$ (46 mg, 0.040 mmol) in CH_2Cl_2 (≃ 25 mL) was stirred at 0 °C. Solid $SbPh_3$ was added slowly; approximately 10 min was allowed between additions of $SbPh_3$. The progress of the reaction was followed by observing the disappearance of bands due to $Os_4(CO)_{15}$ in the infrared spectrum of the reaction mixture. After 1.5 h the reaction was judged complete, and the color of the solution had changed from dark green to amber. The solvent was removed on the vacuum line, and the yellow residue washed with hexane (3 x ≃ 1 mL). The infrared spectrum of the initial washings indicated that the hexane soluble product was $Os(CO)_4(SbPh_3)$.[143] The residue was dissolved in a minumum amount of CH_2Cl_2 and filtered through a celite. Hexane was layered onto the filtrate, and storage at -15 °C overnight afforded $Os_4(CO)_{15}(SbPh_3)$ (22 mg, 37%): mp 135 °C (dec); IR (CH_2Cl_2) ν(CO) 2118(w), 2108(w), 2088(s), 2069(w), 2048(vs), 2032.5(vs), 2014(sh), 2004.5(s), 1975(m), 1959(m), 1917(m,br) cm^{-1}; The mass spectrum was not recorded; Anal. Calcd for $H_{15}C_{33}O_{15}SbOs_4$: C, 25.84; H, 0.99. Found: C, 25.63; H, 1.07.

Preparation of $Os_3(CO)_{11}(COE)$

Following the procedure of Johnson, Lewis, and Pippard for the preparation of $Os_3(CO)_{11}(MeCN)$,[141] $Os_3(CO)_{12}$ (100 mg,

0.110 mmol) in CH_2Cl_2/MeCN (20 mL, 3/1) was sirred under a nitrogen atmosphere in a 125 mL round bottom flask. Trimethylamine oxide in methanol was added dropwise, and the progress of the reaction followed by observing the disappearance of bands due to $Os_3(CO)_{12}$ in the infrared spectrum of the reaction mixture. After removal of the solvent on the vacuum line, approximately 20 mL cyclooctene (COE) was added, and the solution degassed by three freeze-pump-thaw cycles. The flask was placed in an oil bath at 40 °C and stirred for 12 h. The solvent was removed on the vacuum line at 40 °C, and the yellow $Os_3(CO)_{11}(COE)$ recrystallized from dichloromethane/hexane (1/1) at -15 °C in the presence of a small amount of cyclooctene. The yield was 54 mg, 50%, based on $Os_3(CO)_{12}$: mp 116 °C (dec); IR (hexane) ν(CO) 2114(w), 2062(s), 2042(s), 2026(vs), 2007(w), 2000(m), 1990(w), 1980(w), 1963(w) cm^{-1}; A satisfactory mass spectrum (EI) was not be obtained; Anal. Calcd for $H_{14}C_{19}O_{11}Os_3$: C, 23.08; H, 1.43. Found: C, 22.79; H, 1.62.

4.4.2 X-ray Crystallographic Studies

$Os_4(CO)_{15}(CNBu^t)$

Yellow-orange crystals of $Os_4(CO)_{15}(CNBu^t)$ were grown as thin plates from dichloromethane/hexane (1/2) solution at -20 °C. A crystal of dimensions 0.16 x 0.16 x 0.04 mm was selected for the data collection. The unit cell was determined from 25 accurately centered reflections, widely spread through reciprocal space, with $24° < 2\theta < 44°$. The systematic absences were consistent

Table 4.5. Crystallographic Data for the Structure Determination of $Os_4(CO)_{15}(CNBu^t)\cdot 0.4CH_2Cl_2$ (A), $Os_4(CO)_{15}[P(OCH_2)_3CMe]\cdot CH_2Cl_2$ (B), and $Os_4(CO)_{15}(PF_3)$ (C)

Compound	A	B	C
Crystal system	monoclinic	monoclinic	orthorhombic
Space group	$P2/a$	$P2_1/c$	$Pbca$
temp, °C	22	20	20
a (Å)	15.946 (2)	14.220 (2)	15.399 (1)
b (Å)	11.465 (2)	13.930 (2)	17.757 (2)
c (Å)	16.776 (2)	17.930 (2)	17.814 (3)
β, (°)	92.09 (1)	111.65 (1)	
V (Å^3)	3064.8	3308.9	4870.9
Z	4	4	8
FW	1297.4	1412.8	1268.8
ρ_c (g cm^{-3})	2.83	2.67	3.46
μ Mo K_α (cm^{-1})	167.00	154.45	209.83
Transmission	0.112-0.576	0.029-0.231	0.089-0.213
Scan mode	$\omega - 2\theta$	$\omega - 2\theta$	$\omega - 2\theta$
Scan width (°)	0.90+0.35tanθ	0.90+0.35tanθ	0.92+0.35tanθ
Scan speed (°min^{-1}) [d]	0.97-2.75	0.92-5.49	0.92-2.75
Min-max 2θ (°)	0 - 45	0 - 50	0 - 50
Unique data	3997	5817	4254
Obs data, $I \geq 2.5\sigma(I)$	2163	3298	2187
Parameters	189	315	268
R_f [a]	0.051	0.047	0.046
R_{wf} [b]	0.057	0.050	0.044
Max |shift/error|	0.08	0.20	0.05
Max peak (e Å^{-3})	2.2 (1)	1.5 (5)	1.6 (4)
$G.O.F.$ [c]	1.02	1.05	1.10

---

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$.
[c] $G.O.F. = [\Sigma w(F_o - F_c)^2]/(N_{obs} - N_{var})]^{1/2}$

with the monoclinic space groups *P2/a* and *Pa*. The final choice of the centric space group, *P2/a*, was based on the results of the refinement.

An analytical absorption correction was applied to the data. The intensity standards showed approximately 10% decay over the course of the data collection, and the data were therefore scaled appropriately. The osmium atoms were placed from a Patterson map. All other non-hydrogen atoms were located in Fourier difference maps calculated after least-squares refinement of the partial model. Methyl hydrogen atoms were placed in calculated positions (d(C-H) = 0.96 Å) and assigned a common isotropic temperature factor. The hydrogen atom positions were not refined. Another common isotropic temperature factor was assigned to the three methyl carbon atoms.

At this point (R = 0.059), several large peaks (2.0 - 4.5 e $Å^{-3}$) were observed in the electron density difference map. These were well removed from the osmium cluster, and were attributed to the presence of a molecule of solvation (most likely CH_2Cl_2) in the crystal. However, the peaks could not be modeled in a chemically reasonable way. This, together with the decay of the intensity standards indicated that the composition of the crystal may have changed during the data collection. However, the gross features of the $Os_4(CO)_{15}(CNBu^t)$ molecule are, in general, satisfactory.

Table 4.6. Fractional Coordinates for $Os_4(CO)_{15}(CNBu^t)\cdot 0.4CH_2Cl_2$.

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.36655(8)	0.0011(1)	0.16448(9)
Os(2)	0.48696(9)	0.1842(1)	0.20747(8)
Os(3)	0.57070(9)	0.4028(1)	0.22690(9)
Os(4)	0.40157(8)	0.4003(1)	0.15883(9)
N(1)	0.405 (2)	-0.124 (3)	0.333 (2)
O(11)	0.242 (2)	-0.182 (3)	0.107 (2)
O(12)	0.240 (2)	0.159 (3)	0.242 (2)
O(13)	0.349 (1)	0.128 (2)	0.004 (2)
O(14)	0.529 (2)	-0.119 (2)	0.122 (1)
O(21)	0.560 (2)	0.144 (2)	0.043 (2)
O(22)	0.405 (2)	0.175 (3)	0.369 (2)
O(23)	0.631 (2)	0.047 (3)	0.283 (2)
O(31)	0.637 (1)	0.397 (2)	0.058 (2)
O(32)	0.506 (2)	0.396 (3)	0.396 (2)
O(33)	0.739 (2)	0.316 (3)	0.286 (2)
O(34)	0.596 (2)	0.663 (3)	0.237 (2)
O(41)	0.461 (1)	0.360 (2)	-0.012 (2)
O(42)	0.329 (2)	0.411 (3)	0.325 (2)
O(43)	0.226 (2)	0.347 (3)	0.093 (2)
O(44)	0.404 (2)	0.667 (3)	0.151 (2)
C(11)	0.289 (2)	-0.113 (4)	0.130 (2)
C(12)	0.285 (2)	0.102 (4)	0.209 (2)
C(13)	0.357 (2)	0.074 (3)	0.064 (2)
C(14)	0.473 (2)	-0.080 (3)	0.135 (2)
C(15)	0.393 (2)	-0.080 (3)	0.270 (2)
C(21)	0.533 (2)	0.163 (3)	0.103 (2)
C(22)	0.436 (2)	0.186 (4)	0.303 (2)
C(23)	0.571 (3)	0.098 (4)	0.251 (3)
C(31)	0.609 (2)	0.402 (3)	0.115 (2)
C(32)	0.531 (3)	0.399 (4)	0.326 (3)
C(33)	0.672 (3)	0.347 (4)	0.260 (3)
C(34)	0.589 (3)	0.554 (4)	0.233 (3)
C(41)	0.444 (2)	0.378 (3)	0.052 (2)
C(42)	0.358 (2)	0.405 (3)	0.266 (2)
C(43)	0.296 (2)	0.368 (3)	0.120 (2)
C(44)	0.397 (2)	0.565 (4)	0.150 (2)
C(1)	0.432 (2)	-0.186 (4)	0.404 (2)
C(2)	0.519 (3)	-0.145 (5)	0.427 (3)
C(3)	0.426 (4)	-0.316 (5)	0.392 (3)
C(4)	0.373 (3)	-0.146 (5)	0.464 (3)
Cl(1)	0.172 (2)	0.439 (3)	0.460 (2)
Cl(2)	0.2500	0.54 (1)	0.5000
Cl(3)	0.2500	0.321 (8)	0.5000

After convergence of the model (anisotropic osmium atoms) an empirical weighting scheme was adopted, with the value of w given by $[0.90477t_0(X) + 0.770416t_1(X) + 0.376869t_2(X)]^{-1}$, where $X = |F_o|F_{max}^{-1}$ and t_n are polynomial functions of the Chebyshev series.[117]

Additional details of the data collection and structure refinement are given in Table 4.5. Final fractional coordinates for all non-hydrogen atoms of $Os_4(CO)_{15}(CNBu^t)$ are listed in Table 4.6. Bond length and selected bond angle data are listed in Table 4.1. Hydrogen atom coordinates, thermal motion parameters for all atoms, and observed and calculated structure factors are given in Tables H.1, H.2, and H.3, respectively.

$Os_4(CO)_{15}[P(OCH_2)_3CCH_3]$

Amber, hexagonal plates of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ were grown from dichloromethane/hexane at about -50 °C. A specimen of dimensions 0.43 x 0.40 x 0.10 mm was selected for the data collection. The unit cell was determined on the basis of 25 accurately centered reflections that have $32° < 2\theta < 43°$. The monoclinic space group $P2_1/c$ was indicated by the systematic absences. An analytical absorption correction was applied to the data. The (1,0,0) reflection was found to be in the beam-stop and was excluded from the data list.

The osmium atoms were placed from a Patterson map; all other non-hydrogen atoms were located from Fourier difference maps calculated after least-squares refinement of the partial model.

Table 4.7. Fractional Coordinates for

$Os_4(CO)_{15}[P(OCH_2)_3CMe]\cdot CH_2Cl_2$[a]

Atom	x/a	y/b	z/c
Os(1)	0.43359(7)	0.74928(8)	0.36556(6)
Os(2)	0.22836(7)	0.67547(7)	0.32638(5)
Os(3)	0.04413(7)	0.60974(7)	0.33719(6)
Os(4)	0.20013(7)	0.68093(7)	0.48046(5)
P(1)	0.5937 (5)	0.8036 (5)	0.3950 (4)
O(1)	0.676 (2)	0.748 (6)	0.460 (7)
O(2)	0.617 (4)	0.904 (5)	0.432 (9)
O(3)	0.630 (4)	0.81 (1)	0.325 (2)
O(10)	0.678 (2)	0.735 (5)	0.443 (8)
O(20)	0.620 (4)	0.900 (6)	0.439 (9)
O(30)	0.622 (4)	0.83 (1)	0.323 (2)
O(11)	0.450 (2)	0.580 (2)	0.260 (1)
O(12)	0.494 (2)	0.613 (2)	0.512 (1)
O(13)	0.378 (1)	0.905 (1)	0.462 (1)
O(14)	0.344 (2)	0.878 (2)	0.216 (1)
O(21)	0.323 (2)	0.475 (1)	0.360 (1)
O(22)	0.155 (1)	0.882 (1)	0.280 (1)
O(23)	0.176 (2)	0.633 (2)	0.152 (1)
O(31)	0.128 (2)	0.404 (1)	0.381 (1)
O(32)	-0.038 (1)	0.814 (2)	0.295 (1)
O(33)	-0.052 (2)	0.562 (2)	0.159 (1)
O(34)	-0.120 (2)	0.564 (1)	0.400 (2)
O(41)	0.304 (2)	0.484 (2)	0.524 (1)
O(42)	0.130 (1)	0.887 (1)	0.439 (1)
O(43)	0.387 (2)	0.765 (2)	0.606 (1)
O(44)	0.065 (2)	0.658 (2)	0.578 (1)
C(1)	0.777 (3)	0.774 (3)	0.472 (3)
C(2)	0.719 (2)	0.937 (2)	0.455 (2)
C(3)	0.727 (2)	0.850 (3)	0.344 (2)
C(4)	0.786 (2)	0.869 (2)	0.433 (1)
C(5)	0.892 (2)	0.902 (2)	0.451 (2)
C(11)	0.444 (2)	0.642 (2)	0.298 (2)
C(12)	0.471 (2)	0.661 (2)	0.456 (2)
C(13)	0.396 (2)	0.847 (2)	0.426 (2)
C(14)	0.373 (2)	0.835 (2)	0.269 (2)
C(21)	0.286 (2)	0.550 (2)	0.351 (2)
C(22)	0.178 (2)	0.805 (2)	0.300 (1)
C(23)	0.198 (2)	0.650 (2)	0.219 (2)
C(31)	0.104 (2)	0.480 (2)	0.365 (2)
C(32)	-0.007 (2)	0.735 (2)	0.311 (2)
C(33)	-0.018 (2)	0.577 (2)	0.226 (2)

[a]continued on following page.

Table 4.7. Continued.

Atom	*x/a*		*y/b*		*z/c*	
C(34)	-0.058	(2)	0.582	(2)	0.375	(2)
C(41)	0.262	(2)	0.553	(2)	0.504	(2)
C(42)	0.153	(2)	0.812	(2)	0.449	(1)
C(43)	0.319	(2)	0.729	(2)	0.558	(2)
C(44)	0.115	(2)	0.664	(2)	0.539	(2)
Cl(1)	0.257	(2)	0.216	(2)	0.325	(2)
Cl(2)	0.427	(2)	0.077	(2)	0.370	(2)
Cl(3)	0.304	(2)	0.082	(2)	0.282	(2)
Cl(4)	0.323	(2)	0.249	(3)	0.357	(2)
C(98)	0.372	(4)	0.166	(4)	0.333	(4)

After refinement of all non-hydrogen atoms in the molecule of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$, five peaks close to one another, but well removed from the osmium containing species were found in the difference map. The size and disposition of these peaks were consistent with a disordered molecule of solvent (CH_2Cl_2). The best model of the disorder of this group consisted of four half-chlorine sites and one full occupancy carbon site (the occupation factors of these atoms were not refined). Bond length restraints were employed, and one isotropic thermal motion parameter was refined for all four half-chlorine atoms.

There was also evidence for disorder of the $P(OCH_2)_3CCH_3$ ligand. The thermal motion parameters of the $-OCH_2-$ groups were unreasonably large. Several models of the disorder were tried. The best consisted of anisotropic split oxygen sites (*i.e.*, six oxygen atom sites each of half occupancy), with three full occupancy isotropic carbon sites. The methylene hydrogen atoms

were included at calculated positions such that there were two half-occupancy sites associated with each C(4)-C-O linkage, and were allowed to ride on the carbon to which they were bonded. Each pair of oxygen atoms of this group were assigned one set of anisotropic thermal motion parameters, and the group restrained (including vibrational restraints) to meet reasonable geometrical criteria.

Upon convergence of the model (with anisotropic osmium, phosphorus, and oxygen atoms) an empirical weighting scheme was adopted. The weight, w, was given by $w = [12.6216t_0(X) + 15.7944t_1(X) + 9.22089t_2(X) + 1.97702t_3(X)]^{-1}$, where $X = |F_o|F^{-1}_{max}$ and t_n are polynomial functions of the Chebyshev series.[117] Additional details of the data collection and structure refinement are given in Table 4.5. Final fractional coordinates for all non-hydrogen atoms of $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ are listed in Table 4.7. Bond lengths and selected bond angles are collected in Table 4.2. Hydrogen atom coordinates, thermal motion parameters for all atoms, and observed and calculated structure factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$ are given in Tables I.1, I.2, and I.3, respectively.

$Os_4(CO)_{15}(PF_3)$

Crystals of $Os_4(CO)_{15}(PF_3)$ grew as hexagonal plates from chloroform/hexane solution at -50 °C. A sample of dimensions 0.21 x 0.18 x 0.11 mm was used for the data collection. The unit cell was determined on the basis of 25 accurately centered

Table 4.8. Fractional Coordinates for $Os_4(CO)_{15}(PF_3)$

Atom	*x/a*	*y/b*	*z/c*
Os(1)	0.27198(8)	0.39891(6)	0.01941(6)
Os(2)	0.44209(8)	0.32893(6)	0.06377(6)
Os(3)	0.37406(8)	0.30782(6)	0.21874(6)
Os(4)	0.24137(8)	0.42586(6)	0.18357(6)
P	0.1430 (7)	0.4377 (5)	-0.0155 (6)
F(1)	0.1219 (17)	0.5196 (12)	-0.0231 (16)
F(2)	0.0631 (15)	0.4148 (16)	0.0256 (18)
F(3)	0.1092 (20)	0.4128 (17)	-0.0865 (17)
O(11)	0.3401 (16)	0.5629 (10)	0.0266 (11)
O(12)	0.3238 (18)	0.3793 (13)	-0.1423 (12)
O(13)	0.1984 (14)	0.2373 (11)	0.0296 (11)
O(21)	0.5047 (14)	0.4911 (11)	0.0885 (10)
O(22)	0.6193 (17)	0.2734 (13)	0.1241 (13)
O(23)	0.3691 (15)	0.1674 (10)	0.0443 (10)
O(24)	0.5075 (14)	0.3318 (12)	-0.0977 (12)
O(31)	0.2404 (16)	0.1860 (11)	0.1788 (11)
O(32)	0.3236 (17)	0.2989 (13)	0.3848 (10)
O(33)	0.5080 (15)	0.1814 (10)	0.2335 (12)
O(34)	0.5032 (16)	0.4312 (11)	0.2517 (14)
O(41)	0.2079 (18)	0.4381 (13)	0.3515 (12)
O(42)	0.1020 (14)	0.3045 (11)	0.1630 (13)
O(43)	0.1088 (16)	0.5516 (14)	0.1563 (15)
O(44)	0.3826 (14)	0.5477 (10)	0.1949 (12)
C(11)	0.3158 (19)	0.5024 (15)	0.0257 (15)
C(12)	0.3017 (22)	0.3870 (17)	-0.0784 (20)
C(13)	0.2341 (22)	0.2976 (17)	0.0293 (17)
C(21)	0.4733 (19)	0.4303 (15)	0.0806 (15)
C(22)	0.5527 (22)	0.2934 (16)	0.1002 (17)
C(23)	0.3983 (21)	0.2272 (17)	0.0518 (17)
C(24)	0.4799 (23)	0.3319 (17)	-0.0366 (20)
C(31)	0.2859 (20)	0.2346 (17)	0.1912 (17)
C(32)	0.3378 (19)	0.3028 (14)	0.3202 (16)
C(33)	0.4595 (23)	0.2294 (19)	0.2268 (19)
C(34)	0.4514 (22)	0.3840 (17)	0.2352 (19)
C(41)	0.2202 (20)	0.4348 (15)	0.2879 (17)
C(42)	0.1542 (20)	0.3452 (15)	0.1692 (15)
C(43)	0.1577 (21)	0.5061 (16)	0.1618 (16)
C(44)	0.3345 (20)	0.5021 (15)	0.1902 (15)

reflections with $27° < 2\theta < 33°$. The orthorhombic space group *Pbca* was chosen after an examination of the systematic absences. An analytical absorption correction was applied to the data.

The osmium atoms were placed by direct methods; all other atoms were located in Fourier difference maps calculated after least-squares refinement of the partial model. After anisotropic refinement of the osmium, phosphorus, fluorine, and oxygen atoms, a weighting scheme based on counting statistics was adopted ($K = 3 \times 10^{-4}$). Additional details of the data collection and structure refinement are collected in Table 4.5. Final fractional coordinates for all atoms of $Os_4(CO)_{15}(PF_3)$ are listed in Table 4.8; bond lengths and selected bond angles are listed in Table 4.3. Thermal motion parameters, and observed and calculated structure factors for $Os_4(CO)_{15}(PF_3)$ are given in Tables J.1 and J.2, respectively.

REFERENCES

1. Muetterties, E. L. *Chem. Eng. News*. Aug. 30, **1982**, 28.

2. Lewis, J. *Chem. Br.* **1988**, 795.

3. Roberts, D. A.; Geoffroy, G. L. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon: New York, 1982; Chapter 40.

4. Chini, P. *J. Organomet. Chem.* **1980**, *200*, 37.

5. Hsu, L.-Y.; Hsu, W.-L.; Jan, D.-Y.; Shore, S. G. *Organometallics* **1986**, *5*, 1041, and references therein.

6. Farrugia, L. J.; Howard, J. A. K.; Mitrprachachon, P.; Stone, F. G. A. *J. Chem. Soc., Dalton Trans.* **1986**, 2105.

7. Park, J. T.; Shapley, J. R.; Churchill, M. R.; Bueno, C. *Inorg. Chem.* **1984**, *23*, 4476.

8. Jaeger, T. J.; Powell, A. K.; Vahrenkamp, H. *New. J. Chem.* **1988**, *12*, 405.

9. Stone, F. G. A. *Angew. Chem. Int. Ed. Engl.* **1984**, *23*, 89, and references therein.

10. (a) Elliott, G. P.; Howard, J. A. K.; Nunn, M. C.; Stone, F. G. A. *J. Chem. Soc., Chem. Commun.* **1986**, 431. (b) Delgado, E.; Jeffery, J. C.; Stone, F. G. A. *J. Chem. Soc., Dalton Trans.* **1986**, 2105, and references therein.

11. Hieber, W.; Schubert, E. H. *Z. Anorg. Allerg. Chem.* **1965**, *338*, 32.

12. Adams, R. D. *Polyhedron* **1985**, *4*, 2003, and references therein.

13. Shapley, J. R.; Pearson, G. A.; Tachikawa, M.; Schmidt, G. E.; Churchill, M. R.; Hollander, F. *J. Am. Chem. Soc.* **1977**, *99*, 8064.

14. (a) Ditzel, E. J.; Holden, H. D.; Johnson, B. F. G.; Lewis, J.; Saunders, A.; Taylor, M. *J. Chem. Soc., Chem. Commun.* **1982**, 1373. (b) Johnson, B. F. G.; Khuttar, R.; Lewis, J.; McPartlin, M.; Morris, J.; Powell, G. L. *J. Chem. Soc., Chem. Commun.* **1986**, 507. (c) Goudsmit, R. J.; Jeffery, J. G.; Johnson, B. F. G.; Lewis, J; McQueen, R. C. S.; Sanders, A. J.; Liu, J.-C. *J. Chem. Soc., Chem. Commun.* **1986**, 24.

15. Ditzel, E. J.; Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Taylor, M. J. *J. Chem. Soc., Dalton Trans.* **1985**, 555.

16. Couture, C.; Farrar, D. H. *J. Chem. Soc., Dalton Trans.* **1986**, 1395.

17. Beringhelli, T.; Ciani, G.; D'Alfonso, G.; De Maldè, V.; Sironi, A.; Freni, M. *J. Chem. Soc., Dalton Trans.* **1986**, 1051.

18. Einstein, F. W. B.; Pomeroy, R. K.; Rushman, P.; Willis, A. C. *J. Chem. Soc., Chem. Commun.* **1983**, 854.

19. Davis, H. B.; Einstein, F. W. B.; Glavina, P. G.; Jones, T.; Pomeroy, R. K.; Rushman, P. *Organometallics* **1989**, *8*, 1030.

20. Martin, L. R.; Einstein, F. W. B.; Pomeroy, R. K. *Organometallics* **1988**, *7*, 294.

21. Martin, L. R.; Einstein, F. W. B.; Pomeroy, R. K. *J. Am. Chem. Soc.* **1986**, *108*, 338.

22. Johnston, V. J.; Einstein, F. W. B.; Pomeroy, R. K. *J. Am. Chem. Soc.* **1987**, *109*, 7220.

23. Rushman, P.; van Buuren, G. N.; Shiralian, M.; Pomeroy, R. K. *Organometallics* **1983**, *2*, 693.

24. (a) Kang, J. W.; Mosely, K.; Maitlis, P. M. *J. Am. Chem. Soc.* **1969**, *91*, 5970. (b) Hoyano, J. K.; Graham, W. A. G. personal communication to R. K. Pomeroy.

25. Tachikawa, M.; Shapley, J. R. *J. Organomet. Chem.* **1977**, *124*, C19.

26. Johnston, V. J.; Einstein, F. W. B.; Pomeroy, R. K. *J. Am. Chem. Soc.* **1987**, *109*, 8111.

27. Johnston, V. J.; Einstein, F. W. B.; Pomeroy, R. K. *Organometallics* **1988**, *7*, 1867.

28. Calderazzo, F.; Ercoli, R.; Natta, G. In *Organic Syntheses via Metal Carbonyls*; Wender, I., Pino, P., Eds.; Wiley: New York, 1968; Vol. 1, Chapter 1, and references therein.

29. *ibid*, p 20.

30. *ibid*, p 14.

31. *ibid*, p 28.

32. Heiber, W.; Stallmann, H. *Z. Electrochem.* **1943**, *49*, 288.

33. Huang, J.; Hedberg, K.; Pomeroy, R. K. *Organometallics* **1988**, *7*, 2049.

34. Corey, E. R.; Dahl, L. F. *J. Am. Chem. Soc.* **1961**, *83*, 2203.

35. Deeming, A. J. *Adv. Organomet. Chem.* **1986**, *26*, 1. See also: Adams, R. D.; Selegue, J. P. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon: New York, 1982; Vol. 4, p 967.

36. Moss, J. R.; Graham, W. A. G. *J. Chem. Soc., Chem. Commun.* **1970**, 835.

37. Moss, J. R.; Graham, W. A. G. *J. Chem. Soc., Dalton Trans.* **1977**, 95.

38. Eady, C. R.; Johnson, B. F. G.; Lewis, J. *J. Organomet. Chem.* **1972**, *37*, C39.

39. Eady, C. R.; Johnson, B. F. G.; Lewis, J. *J. Chem. Soc., Dalton Trans.* **1975**, 2606.

40. (a) Farrar, D. H.; Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Rosales, M. J. *J. Chem. Soc., Dalton Trans.* **1982**, 2051. (b) Farrar, D. H.; Johnson, B. F. G.; Lewis, J.; Nicholls, J. N.; Raithby, P. R.; Rosales, M. J. *J. Chem. Soc., Chem. Commun.* **1981**, 273.

41. Nicholls, J. N.; Farrar, D. H.; Jackson, P. F.; Johnson. B. F. G.; Lewis, J. *J. Chem. Soc., Dalton Trans.* **1982**, 1395.

42. Eady, C. R.; Johnson, B. F. G.; Lewis, J.; Reichert, B. E.; Sheldrick, G. M. *J. Chem. Soc., Chem. Commun.* **1976**, 271.

43. Mason, R.; Thomas, K. M.; Mingos, D. M. P. *J. Am. Chem. Soc.* **1973**, *95*, 3802.

44. Eady, C. R.; Johnson, B. F. G.; Lewis, J.; Mason, R.; Hitchcock, P. B.; Thomas, K. M. *J. Chem. Soc., Chem. Commun.* **1977**, 385.

45. Pomeroy, R. K. *J. Organomet. Chem.*, accepted for publication.

46. Lu, C.-Y.; Einstein, F. W. B.; Johnston, V. J.; Pomeroy, R. K. *Inorg. Chem.*, in press.

47. Einstein, F. W. B.; Johnston, V. J.; Ma, A. K.; Pomeroy, R. K. *Organometallics*, in press.

48. Johnson, J. R.; Kaesz, H. D. *Inorg. Synth.* **1978**, *18*, 60.

49. Cartwright, H. *J. Chem. Educ.* **1986**, *63*, 984.

50. Churchill, M. R.; DeBoer, B. G. *Inorg. Chem.* **1977**, *16*, 878.

51. (a) Mingos, D. M. P. *Acc. Chem. Res.* **1984**, *17*, 311, and references therein. (b) Wade, K. *Inorg. Nucl. Chem. Lett.* **1972**, *8*, 559.

52. Pauling, L. *The Nature of the Chemical Bond*, 3rd. Ed.; Cornell University Press: Ithaca, NY, 1960; p 400.

53. (a) Cotton, F. A.; Wilkinson, G. *Advanced Inorganic Chemistry*, 4th Ed.; Wiley: New York, 1980; p 1083. (b) Wade, K. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: Chichester, England, 1980; p 211.

54. McCarley, R. E. *Polyhedron* **1986**, *5*, 51.

55. Hogarth, G.; Phillips, J. A.; Van Gastel, F.; Taylor, N. J.; Marder, T. B.; Carty, A. J. *J. Chem. Soc., Chem. Commun.* **1988**, 1570.

56. Diebold, M. P.; Drake, S. R.; Johnson, B. F. G.; Lewis, J.; McPartlin, M.; Powell, H. *J. Chem. Soc., Chem. Commun.* **1988**, 1358.

57. Hoffmann, R. *Angew. Chem. Int. Ed. Engl.* **1982**, *21*, 711.

58. Evans, D. G.; Mingos, D. M. P. *Organometallics* **1983**, *2*, 435.

59. (a) Bau R.; Fontal, B.; Kaesz, H. D., Churchill, M. R. *J. Am. Chem. Soc.* **1967**, *89*, 6374. (b) Hayward, C.-M. T.; Shapley, J. R. *Organometallics* **1988**, *7*, 448. (c) Ciani, G.; D'Alfonso, G.; Freni, M.; Romiti, P.; Sironi, A. *J. Organomet. Chem.* **1978**, *157*, 199.

60. Churchill, M. R.; Bau, R. *Inorg. Chem.* **1968**, *7*, 2606.

61. Einstein, F. W. B.; Johnston, V. J.; Pomeroy, R. K.; Lu, C.-Y., unpublished results.

62. Briant, C. E.; Gilmour, D. I.; Mingos, D. M. P. *J. Chem. Soc., Dalton Trans.* **1986**, 835.

63. Horwitz, C. P.; Holt, E. M.; Shriver, D. F. *Inorg. Chem.* **1984**, *23*, 2491.

64. Doedens, R. J.; Dahl, L. F. *J. Am. Chem. Soc.* **1966**, *88*, 4847.

65. Lauher, J. W. *J. Am. Chem. Soc.* **1986**, *108*, 1521.

66. (a) Bodner, G. M.; May, M. P.; McKinney, L. E. *Inorg. Chem.* **1980**, *19*, 1951. (b) Mann, B. E.; Taylor, B. F. *^{13}C NMR Data for Organometallic Compounds*; Academic: New York, 1981; p 6.

67. This work was performed in collaboration with Mr. A. K. Ma of our laboratory.

68. Alex, R. F.; Pomeroy, R. K. *Organometallics* **1987**, *6*, 2437, and *J. Organomet. Chem.* **1985**, *284*, 379.

69. Gilmour, B.; Ma, A. K.; Pomeroy, R. K.; Wong, E., to be published.

70. Johnson, B. F. G.; Lewis, J.; Reichert, B. E.; Schorpp, K. T. *J. Chem. Soc., Dalton Trans.* **1976**, 1403.

71. Sandström, J. *Dynamic NMR Spectroscopy*; Academic: New York, 1982; p 18.

72. Davis, H. B.; Einstein, F. W. B.; Johnston, V. J.; Pomeroy, R. K. *J. Am. Chem. Soc.* **1988**, *110*, 4457.

73. Davis, H. B.; Pomeroy, R. K., unpublished results.

74. Legon, A. C. *Chem. Rev.* **1980**, *80*, 231.

75. Carrondo, M. A. A. F. de C. T.; Skapski, A. C. *Acta Cryst.* **1978**, *B34*, 1857.

76. Heveldt, P. F.; Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Sheldrick, G. M. *J. Chem. Soc., Chem. Commun.* **1978**, 340.

77. Vahrenkamp, H.; Wolters, D. *Organometallics* **1982**, *1*, 874.

78. Churchill, M. R.; Hollander, F. J.; Lashewycz, R. A.; Pearson, G. A.; Shapley, J. R. *J. Am. Chem. Soc.* **1981**, *103*, 2430.

79. Churchill, M. R.; Hollander, F. J. *Inorg. Chem.* **1981**, *20*, 4124.

80. (a) Mingos, D. M. P. *Nature (London), Phys. Sci.* **1972**, *236*, 99. (b) Carty, A. J.; *Pure Appl. Chem.* **1982**, *54*, 113.

81. Adams, R. D.; Yang, L.-W. *J. Am. Chem. Soc.* **1983**, *105*, 235.

82. Adams, R. D.; Horvàth, I. T. *Inorg. Chem.* **1984**, *23*, 4718.

83. Adams, R. D.; Horvàth, I. T.; Mathur P. *J. Am. Chem. Soc.* **1984**, *106*, 6296.

84. Churchill, M. R.; Bueno, C.; Young, D. A. *J. Organomet. Chem.* **1981**, *213*, 139.

85. Cook, N.; Smart, L.; Woodward, P. *J. Chem. Soc., Dalton Trans.* **1977**, 1744.

86. Willis, A. C.; van Buuren, G. N.; Pomeroy, R. K.; Einstein, F. W. B. *Inorg. Chem.* **1983**, *22*, 1162.

87. Appleton, T. G.; Clark, H. C.; Manzer, L. E. *Coord. Chem. Rev.* **1979**, *10*, 335.

88. Einstein, F. W. B.; Jennings, M. C.; Krentz, R.; Pomeroy, R. K.; Rushman, P.; Willis, A. C. *Inorg. Chem.* **1987**, *26*, 1341.

89. Ref 52 p 223, and references therein.

90. Morrison, R. T.; Boyd, R. N. *Organic Chemistry*; Allyn and Bacon: Boston, 1976; p 290.

91. Delly, B.; Manning, M. C.; Ellis, D. E.; Berkowitz, J.; Trogler, W. C. *Inorg. Chem.* **1982**, *21*, 2247.

92. Elian, M.; Hoffmann, R. *Inorg. Chem.* **1975**, *14*, 1058.

93. Mealli, C. *J. Am. Chem. Soc.* **1985**, *107*, 2245.

94. Bender, B. R.; Bertoncello, R.; Burke, M. R.; Casarin, M.; Granozzi, G.; Norton, J. R.; Takats, J. *Organometallics* **1989**, *8*, 1777, and references therein.

95. Lauher, J. W. *Abstracts of Papers*, 28th Sanibel Symposia, Marineland, Florida, March 1988.

96. Ettinger, R.; Blume, P.; Lauterbur, P. C.; Patterson, A. *J. Chem. Phys.* **1960**, *33*, 1597.

97. Mingos, D. M. P. *Inorg. Chem.* **1982**, *21*, 466.

98. (a) Knox, S. A. R.; Koepke, J. W.; Andrews, M. A.; Kaesz, H. D. *J. Am. Chem. Soc.* **1975**, *97*, 3942. (b) Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Sheldrick, G. M.; Wong, K.; McPartlin, M. *J. Chem. Soc., Dalton Trans.* **1978**, 673.

99. Cotton, F. A. *Prog. Inorg. Chem.* **1976**, *21*, 1.

100. Broach, R. W.; Williams, J. M. *Inorg. Chem.* **1979**, *18*, 314.

101. Willis, A. C.; Einstein, F. W. B.; Ramadan, R. M.; Pomeroy, R. K. *Organometallics* **1983**, *2*, 935.

102. Ma, A. K.; Einstein, F. W. B.; Johnston, V. J.; Pomeroy, R. K. *Organometallics*, in press.

103. Adams, R. D.; Golembeski, N. M. *Inorg. Chem.* **1979**, *18*, 1909, and *J. Am. Chem. Soc.* **1979**, *101*, 2579.

104. Wade, K. In *Transition Metal Clusters*; Johnson, B. F. G., Ed.; Wiley: Chicester, England, 1980; p 212, and references therein.

105. Sherwood, D. E.; Hall, M. B. *Inorg. Chem.* **1982**, *21*, 3458.

106. Johnson, B. F. G.; Benfield, R. E. *J. Chem. Soc., Dalton Trans.* **1978**, 1554.

107. Benfield, R. E.; Johnson, B. F. G.; *J. Chem. Soc., Dalton Trans.* **1980**, 1743.

108. Collman, J. P.; Hegedus, L. S. *Principles and Applications of Organotransition Metal Chemistry*; University Science Books: Mill Valley, CA, 1980; p 83.

109. Cotton, F. A.; Hunter, D. L. *Inorg. Chim. Acta.* **1974**, *11*, L9.

110. Johnson, B. F. G. *J. Chem. Soc., Chem. Commun.* **1976**, 703.

111. Binsted, N.; Evans, J.; Creaves, N.; Price, R. J. *J. Chem. Soc., Chem. Commun.* **1987**, 1303.

112. Forster, A.; Johnson, B. F. G.; Lewis, J.; Matheson, T. W.; Robinson, B. H.; Jackson, W. A. G. *J. Chem. Soc., Chem. Commun.* **1974**, 1042.

113. (a) Muetterties, E. L. *Inorg. Chem.* **1965**, *4*, 769. (b) Benfield, R. E.; Braga, D.; Johnson, B. F. G. *Polyhedron* **1988**, *7*, 2549.

114. I am grateful to Professor R. H. Hill and Mr. B. J. Palmer for recording the variable temperature infrared spectra of $Os_4(CO)_{14}$.

115. Grevels, F.-W.; Jacke, J.; Klotzbücher, W. E.; Krüger, C.; Seevogel, K.; Tsay, Y.-H. *Angew. Chem. Int. Ed. Engl.* **1987**, *26*, 885.

116. *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, England, 1974; Vol IV, Tables 2.2B and 2.3.1.

117. Carruthers, J. R.; Watkin, D. J. *Acta Crystallogr.* **1979**, *A35*, 698.

118. Larson, A. C. In *Crystallographic Computing*; Ahmed, F. R., Hall, S. R., Huber, C. P., Eds.; Munksgaard: Copenhagen, 1970.

119. Gabe, E. J.; Larson, A. C.; Lee, F. L.; LePage, Y. *NRC VAX Crystal Structure System*; Chemistry Division, National Research Council, Ottawa, 1983.

120. Watkin, D. J.; Carruthers, J. R.; Betteridge, P. W. *CRYSTALS*; Chemical Crystallography Laboratory, University of Oxford, Oxford, 1985.

121. Davies, E. K. *SNOOPI Plot Program*; Chemical Crystallography Laboratory, University of Oxford, Oxford, 1985.

122. The version used was contained in the NRC package, ref 119.

123. North, A. C. T.; Phillips, D. C.; Mathews, F. *Acta Crystallogr., Sec. A: Cryst. Phys., Diff., Theo. Gen. Crystallogr.* **1968**, *A24*, 351.

124. Einstein, F. W. B.; Johnston, V. J.; Ma, A. K.; Pomeroy, R. K. unpublished results.

125. Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Azman, S. N.; Syed-Mustaffa, B.; Taylor, M. J.; Whitmire, K. H.; Clegg, W. *J. Chem. Soc., Dalton Trans.* **1984**, 2111.

126. Orpen, A. G. *J. Chem. Soc., Dalton Trans.* **1980**, 2509.

127. (a) Knight, J.; Mays, M. J. *J. Chem. Soc., Dalton Trans.* **1972**, 1022. (b) Churchill, M. R.; Hollander, F. J. *Inorg. Chem.* **1977**, *16*, 2493.

128. Adams, R. D.; Horvàth, I. T.; Segmüller, B. E.; Yang, L.-W. *Organometallics* **1983**, *2*, 1301.

129. Martin, L. R.; Einstein, F. W. B.; Pomeroy, R. K. *Inorg. Chem.* **1988**, *27*, 2986.

130. Mays, M. J.; Gavens, P. D. *J. Chem. Soc., Dalton Trans.* **1980**, 911.

131. (a) Churchill, M. R.; DeBoer, B. G.; Rotella, F. J. *Inorg. Chem.* **1976**, 1843. (b) See also papers in the series: Structural Studies on Polynuclear Osmium Hydrides, *e.g.*, Park, J. T.; Shapley, J. R.; Bueno, C.; Ziller, J. W.; Churchill, M. R. *Organometallics* **1988**, *7*, 2307.

132. Huheey, J. E. *Inorganic Chemistry: Principles of Structure and Reactivity*, 2nd Ed.; Harper & Row: New York, 1978; p 494, and references therein.

133. Adams, R. D.; Horvàth, I. T.; Segmüller, B. E.; Yang, L.-W. *Organometallics* **1983**, *2*, 144.

134. Tachikawa, M.; Richter, S. I.; Shapley, J. R. *J. Organomet. Chem.* **1977**, *128*, C9.

135. Cotton, F. A.; Hanson, B. E. *Inorg. Chem.* **1977**, *16*, 2820.

136. Cobbeldick, R. E.; Einstein, F. W. B.; Pomeroy, R. K.; Spetch, E. R. *J. Organomet. Chem.* **1980**, *195*, 77.

137. Walker, N.; Stuart, D. *Acta Cryst.* **1983**, *A39*, 158.

138. Batchelor, R. J.; Einstein, F. W. B.; Pomeroy, R. K.; Shipley, J. A., to be published.

139. Crabtree, R. H. *The Organometallic Chemistry of the Transition Metals*; Wiley: New York, 1988; p 72.

140. Tolman, C. A. *Chem. Rev.* **1977**, *77*, 313.

141. Johnson, B. F. G.; Lewis, J.; Pippard, D. A. *J. Chem. Soc., Dalton Trans.* **1981**, 407.

142. Singleton, E.; Oosthaizen, H. E. *Adv. Organomet. Chem.* **1983**, *22*, 209.

143. Martin, L. R.; Einstein, F. W. B.; Pomeroy, R. K. *Inorg. Chem.* **1983**, *22*, 1961.

144. Williams, I. D.; Marder, T. B. to be submitted for publication.

145. Albright, T. A.; Burdett, J. K.; Whangbo, M. H. *Orbital Interactions in Chemistry*; Wiley: New York, 1985; p 15.

146. (a) Bodner, G. M.; May, M. P.; McKinney, L. E. *Inorg. Chem.* **1980**, *19*, 1951. (b) Rushman, P.; Brown, T. L. *Inorg. Chem.* **1987**, *109*, 3632. (c) Hanckel, J. M.; Lee, K. W.; Rushman, P.; Brown, T. L. *Inorg. Chem.* **1986**, *25*, 1852.

147. Bartsch, R.; Hitchcock, P. B.; Meidine, M. F.; Nixon, J. F. *J. Organomet. Chem.* **1984**, *266*, C41.

148. Martin, L. R. *Ph. D. Thesis*, Simon Fraser University, Burnaby, B. C., Canada, 1986.

149. I thank Mr. A. K. Ma for supplying the ^{13}C NMR spectra of $Os_3(CO)_{12}$ and $Os_3(CO)_{11}(PF_3)$.

150. See for example: Hembre, R. T.; Scott, C. P.; Norton, J. R. *J. Am. Chem. Soc.* **1987**, *109*, 3468.

151. Takats, J. *Abstracts of Papers*, 72nd Canadian Chemical Conference and Exhibition, Victoria, B. C., Canada, June 1989.

152. Chisholm, M. H.; Folting, K.; Hammond, C. E.; Hampden-Smith, M. J. *J. Am. Chem. Soc.* **1988**, *110*, 3314.

153. Cirjak, L. M.; Huang, J.-S.; Zhu, Z.-H.; Dahl, L. F. *J. Am. Chem. Soc.* **1980**, *102*, 6626.

154. (a) Ref 66(b) p 176. (b) Aime, S.; Osella, D.; Milone, L.; Rosenberg, E. *J. Organomet. Chem.* **1981**, *213*, 207.

155. Aime, S.; Osella, D. *J. Chem. Soc., Chem. Commun.* **1981**, 300.

156. Fleming, M. M.; Pomeroy, R. K.; Rushman, P. *J. Organomet. Chem.* **1984**, *273*, C33.

157. Mann, B. E. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon: New York, 1982; Vol. 3, p 89, see also p 104, and references therein.

The Tetranuclear Carbonyls of Osmium and Some Related Derivatives

Appendix: Supplemental Tables

by

Victor J. Johnston

B.S., Central Washington University, 1985

THESIS SUBMITTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

in the Department

of

Chemistry

© Victor J. Johnston 1989

SIMON FRASER UNIVERSITY

September 1989

All rights reserved. This work may not be reproduced in whole or in part, by photocopy or other means, without permission of the author.

SUPPLEMENTAL TABLES

Table A.1 U_{iso} Values for $Os_4(CO)_{15}$.

Atom	U(iso)
Os(1)	0.0217(4)
Os(2)	0.0247(3)
Os(3)	0.0205(4)
Os(4)	0.0219(3)
O(11)	0.040(2)
O(12)	0.040(2)
O(13)	0.040(2)
O(21)	0.040(2)
O(22)	0.040(2)
O(23)	0.040(2)
O(24)	0.040(2)
O(31)	0.040(2)
O(32)	0.040(2)
O(33)	0.040(2)
O(34)	0.040(2)
O(41)	0.040(2)
O(42)	0.040(2)
O(43)	0.040(2)
O(44)	0.040(2)
C(11)	0.026(2)
C(12)	0.026(2)
C(13)	0.026(2)
C(21)	0.026(2)
C(22)	0.026(2)
C(23)	0.026(2)
C(24)	0.026(2)
C(31)	0.026(2)
C(32)	0.026(2)
C(33)	0.026(2)
C(34)	0.026(2)
C(41)	0.026(2)
C(42)	0.026(2)
C(43)	0.026(2)
C(44)	0.026(2)

* A single isotropic thermal parameter was refined for all 15 oxygen atoms, and another isotropic thermal parameter was refined for all 15 carbon atoms.

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	$/F_o/$	$/F_c/$	PHI
** K= 0 L= 0 **			
2	836	842	7
4	309	288	8
8	119	110	6
10	306	305	9
12	415	407	11
14	443	409	10
** K= 1 L= 0 **			
5	46	63	184
** K= 2 L= 0 **			
0	404	397	187
2	131	144	188
4	226	226	8
6	459	475	8
8	408	413	7
12	128	135	190
14	135	146	190
** K= 3 L= 0 **			
1	145	138	189
3	226	232	186
5	228	249	186
7	195	209	187
9	148	143	189
11	103	102	189
** K= 4 L= 0 **			
0	108	115	7
4	413	444	187
6	480	598	188
8	403	499	188
10	135	172	190
** K= 5 L= 0 **			
5	70	77	9
** K= 6 L= 0 **			
0	640	633	186
2	262	270	190
6	148	163	9
8	99	110	8
12	201	208	191
** K= 7 L= 0 **			
1	394	390	8
3	285	280	9
5	247	245	9
7	217	223	8
9	183	191	10
11	191	188	11
** K= 8 L= 0 **			
0	368	357	8
2	196	195	9
6	186	189	189
8	120	127	190
** K= 9 L= 0 **			
3	61	61	11
5	102	97	11
7	118	107	10
** K= 10 L= 0 **			
0	211	210	190
2	137	143	188
6	122	137	11
8	121	118	10
** K= 11 L= 0 **			
1	104	110	192
3	165	160	191
5	207	196	190
** K= 12 L= 0 **			
0	153	150	10
** K= 1 L= 1 **			
-15	117	92	191
-13	311	301	190
-11	377	380	190
-9	371	379	187
-7	71	61	185
-5	179	180	7
-3	75	72	2
-1	545	494	186
1	854	855	186
3	618	624	188
5	253	264	188
7	66	80	6
9	121	122	8
11	58	66	190
13	273	274	189
15	255	270	191
** K= 2 L= 1 **			
-2	66	58	185
0	68	59	182
4	72	70	182
** K= 3 L= 1 **			
-9	168	159	187
-7	497	484	188
-5	883	864	186
-3	493	473	190
-1	308	275	187
1	62	54	6
5	267	261	189
7	531	546	188
9	589	589	188
11	249	261	191
** K= 4 L= 1 **			
-14	110	98	10
-12	121	112	9
-10	121	133	9
-8	165	169	9
-6	254	243	8
-4	312	292	7
-2	214	193	10
0	234	217	7
2	189	194	7
4	160	172	9
6	200	200	8
8	194	223	8
12	78	112	11

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
** K= 5	L= 1 **		
-13	116	119	190
-11	181	170	191
-9	85	81	188
-7	236	227	7
-5	412	394	8
-3	286	277	8
-1	54	58	185
1	330	324	187
3	245	251	188
7	264	283	8
9	272	293	9
11	128	113	9
13	74	76	190
** K= 6	L= 1 **		
-12	138	135	11
-10	172	160	10
-8	176	165	9
-6	125	124	9
-4	125	121	10
-2	223	216	7
0	253	253	8
2	268	275	8
4	205	217	8
6	163	169	8
8	103	109	9
12	107	117	10
** K= 7	L= 1 **		
-11	207	220	12
-9	217	215	8
-5	147	140	189
-1	298	272	7
1	470	461	8
3	370	373	9
5	178	165	8
** K= 8	L= 1 **		
-10	130	123	192
-8	135	124	190
-6	91	81	192
-4	112	102	189
-2	174	176	188
0	209	204	189
2	211	201	190
4	173	174	190
6	124	138	189
8	90	87	190
** K= 9	L= 1 **		
-9	58	42	194
-7	86	89	12
-5	228	210	10
-3	127	124	12
1	186	188	190
3	144	157	191
7	145	151	10
9	154	158	11
** K= 10	L= 1 **		
-8	175	164	191
-6	237	219	191
-4	266	255	190
-2	238	231	190
0	212	208	189
2	170	169	190
4	192	183	190
6	239	221	190
8	205	218	190
** K= 11	L= 1 **		
-5	117	108	191
1	184	181	11
3	153	157	11
** K= 0	L= 2 **		
-14	136	133	189
-12	320	309	191
-10	648	623	188
-8	593	598	188
-6	401	390	187
-4	118	117	183
0	503	494	186
2	873	851	187
4	1250	1333	185
6	671	640	187
8	225	201	188
12	94	89	188
14	251	246	190
** K= 1	L= 2 **		
-9	56	48	8
-1	50	54	5
1	42	42	9
3	102	87	5
5	46	52	8
** K= 2	L= 2 **		
-14	159	143	190
-10	231	234	8
-8	186	183	7
-6	164	168	187
-4	509	500	187
-2	473	568	187
0	224	199	187
2	172	171	9
4	281	278	9
6	89	87	8
8	253	257	188
10	300	301	190
12	237	234	189
** K= 3	L= 2 **		
-13	90	81	12
-11	87	84	11
-9	126	123	8
-7	152	155	7
-5	174	167	9
-3	289	269	6
-1	349	324	5
1	272	263	5
3	115	123	9
5	133	128	9
7	169	179	7
9	141	159	8
11	109	126	10
** K= 4	L= 2 **		
-14	229	225	10
-6	329	321	8
-4	603	564	9

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	$/F_o/$	$/F_c/$	PHI
-2	728	687	7
0	492	475	6
6	121	117	7
8	408	416	8
10	377	388	11
12	298	302	10
** K=	5	L=	2 **
-5	84	68	189
-3	112	108	188
-1	79	70	190
1	73	71	185
** K=	6	L=	2 **
-12	121	114	14
-10	328	312	9
-8	296	295	9
-6	75	77	8
-4	179	168	188
-2	177	172	188
0	121	118	6
2	291	298	10
4	483	480	8
6	200	221	10
10	113	127	190
** K=	7	L=	2 **
-11	200	190	191
-9	268	259	190
-7	275	266	189
-5	273	254	189
-3	279	264	188
-1	309	291	188
1	307	290	189
3	340	339	189
5	333	321	189
7	226	247	189
9	176	187	190
11	138	147	190
** K=	8	L=	2 **
-10	190	186	191
-8	177	169	190
-4	182	166	10
-2	218	210	8
2	217	211	190
4	274	285	189
6	131	144	189
10	121	132	11
** K=	9	L=	2 **
-5	76	77	192
-3	132	120	190
-1	103	108	190
** K=	10	L=	2 **
-8	112	108	11
-4	137	129	192
-2	142	145	191
2	183	167	9
4	193	187	10
** K=	11	L=	2 **
-5	175	167	11
-3	223	205	11
-1	231	223	10
1	175	177	10
5	103	103	12
** K=	1	L=	3 **
-11	166	160	10
-9	498	491	8
-7	549	570	8
-5	399	395	7
-1	258	264	185
3	467	453	7
5	676	674	7
7	545	533	8
9	122	125	11
11	74	68	189
** K=	3	L=	3 **
-13	308	294	11
-11	181	178	10
-5	256	249	7
-3	627	591	8
-1	1200	1185	5
1	432	437	10
3	166	156	9
9	223	226	10
11	294	363	11
13	315	373	9
** K=	4	L=	3 **
-14	127	121	190
-12	129	129	190
-10	141	138	189
-8	172	173	187
-6	176	170	188
-4	196	192	189
-2	327	314	186
0	365	346	186
2	183	185	190
4	177	179	188
6	170	161	188
10	135	141	190
12	116	140	190
** K=	5	L=	3 **
-13	142	146	191
-9	216	214	8
-7	270	257	8
-3	305	302	187
-1	508	492	187
1	240	248	188
3	90	103	7
5	251	280	8
7	175	199	8
11	201	200	190
13	146	189	190
** K=	6	L=	3 **
-12	92	92	190
-10	144	144	189
-8	216	211	188
-6	231	220	188
-4	196	185	188
-2	143	137	189
0	117	123	189
2	178	178	188
4	213	213	188
6	209	232	188

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
8	155	181	189
10	116	110	190
** K= 7 L= 3 **			
-11	73	83	192
-9	299	273	190
-7	338	319	190
-5	218	206	188
-1	148	155	9
3	252	263	188
5	399	404	188
7	293	321	188
** K= 8 L= 3 **			
-10	124	120	10
-8	175	166	10
-6	175	168	10
-4	136	135	10
-2	101	86	12
0	101	102	10
2	160	156	8
4	177	179	9
6	180	177	10
8	145	153	10
10	83	95	10
** K= 9 L= 3 **			
-9	129	133	10
-7	161	148	11
-3	124	116	192
-1	271	268	188
1	89	97	192
5	173	174	10
** K= 10 L= 3 **			
-8	142	143	10
-6	148	144	11
-4	193	178	12
-2	274	258	10
0	298	291	9
2	225	221	10
4	156	173	10
6	162	150	10
8	149	139	11
** K= 11 L= 3 **			
-5	69	68	191
-3	71	70	9
-1	128	137	9
5	171	165	191
** K= 0 L= 4 **			
-10	245	236	8
-8	576	546	8
-6	853	870	7
-4	517	540	10
-2	439	460	5
0	110	109	2
4	437	424	8
6	805	809	7
8	821	808	7
10	352	335	10
12	118	110	8
** K= 1 L= 4 **			
-5	56	69	186
-3	91	98	356
** K= 2 L= 4 **			
-14	210	197	10
-12	247	246	11
-10	176	176	8
-8	127	128	187
-6	321	324	187
-4	175	178	186
-2	349	326	5
0	642	630	6
2	425	410	8
4	90	64	11
6	201	203	189
8	204	205	190
12	211	197	9
14	203	210	11
** K= 3 L= 4 **			
-13	106	102	190
-11	106	106	191
-9	107	109	191
-7	118	121	189
-5	150	148	187
-3	200	193	186
-1	212	200	188
1	264	276	186
3	253	257	186
5	191	187	187
7	104	111	189
9	85	89	190
11	85	111	189
** K= 4 L= 4 **			
-14	264	254	191
-12	318	316	191
-10	301	301	188
-8	72	56	187
-6	95	91	8
-4	54	57	187
-2	518	494	186
0	703	681	188
2	522	530	189
4	293	294	187
10	95	99	190
12	285	296	189
** K= 5 L= 4 **			
-11	59	53	11
-9	80	76	5
-1	103	96	7
1	115	109	8
** K= 6 L= 4 **			
-12	84	82	12
-8	227	219	190
-6	433	421	188
-4	260	258	190
0	196	199	7
2	137	135	8
4	132	135	188
6	292	336	189
8	313	339	189
10	87	108	193
** K= 7 L= 4 **			

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
-11	135	141	11
-9	194	193	10
-7	281	263	10
-5	331	312	9
-3	292	273	10
-1	324	298	8
1	262	261	8
3	242	242	9
5	247	270	9
7	252	280	9
9	208	229	10
11	145	161	11
** K=	8	L=	4 **
-8	171	165	9
-6	239	229	10
-4	160	153	10
0	210	204	189
2	166	170	188
6	203	214	9
8	189	209	10
** K=	9	L=	4 **
-9	69	57	11
-3	55	56	8
-1	90	95	10
1	125	119	10
3	95	89	12
5	87	59	10
** K=	10	L=	4 **
-8	97	96	190
-6	176	163	191
-4	117	110	190
-2	81	74	7
0	169	169	10
2	96	100	13
6	160	151	190
** K=	11	L=	4 **
-5	121	117	190
-3	156	156	189
-1	205	195	190
1	207	210	191
3	190	190	191
5	147	143	191
** K=	1	L=	5 **
-11	108	102	8
-7	305	291	188
-5	559	547	189
-3	629	653	187
-1	401	409	186
3	181	178	7
5	67	62	191
7	437	437	187
9	414	410	190
11	310	307	189
** K=	2	L=	5 **
0	71	58	357
** K=	3	L=	5 **
-13	226	220	192
-11	453	446	189
-9	420	415	189
-7	196	193	189
-1	425	396	186
1	626	595	188
3	838	841	186
5	387	383	189
7	106	100	188
13	161	197	191
** K=	4	L=	5 **
-12	104	120	11
-10	185	180	9
-8	175	179	9
-6	203	191	8
-4	208	204	6
-2	203	192	7
0	213	207	8
2	255	274	7
4	254	259	7
6	179	173	9
8	143	135	9
10	103	100	10
12	96	92	11
** K=	5	L=	5 **
-11	218	220	10
-9	195	195	9
-5	279	274	188
-3	295	292	187
1	300	305	8
3	332	373	8
5	168	173	7
7	139	131	188
9	165	179	191
** K=	6	L=	5 **
-10	91	100	8
-8	124	127	9
-6	163	170	9
-4	257	251	8
-2	261	253	7
0	205	203	7
2	111	126	9
4	100	113	9
6	127	144	9
8	146	151	10
10	145	147	10
** K=	7	L=	5 **
-11	71	82	191
-7	157	159	10
-5	301	295	11
-3	366	343	9
-1	211	202	6
1	81	73	190
3	116	130	189
7	237	257	8
9	224	260	11
11	182	192	10
** K=	8	L=	5 **
-8	85	97	190
-6	153	144	190
-4	190	183	190
-2	189	181	190
0	145	151	189
2	92	85	191
4	106	89	190

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
8	108	134	191
10	97	117	192
** K= 9 L= 5 **			
-9	111	116	10
-5	166	163	190
-3	159	148	190
1	138	137	12
3	194	208	10
** K= 10 L= 5 **			
-6	184	168	190
-4	159	159	190
-2	171	167	190
0	185	183	191
2	233	228	191
4	240	241	190
6	196	194	190
** K= 11 L= 5 **			
-5	157	154	11
-3	164	159	11
** K= 0 L= 6 **			
-14	297	284	192
-12	185	185	191
-6	337	348	187
-4	695	649	188
-2	1026	1154	185
0	539	506	189
2	281	297	186
6	75	73	192
8	344	329	189
10	495	494	189
12	497	479	188
14	190	190	191
** K= 1 L= 6 **			
-3	75	89	5
-1	74	85	6
** K= 2 L= 6 **			
-10	338	319	188
-8	370	354	188
-6	148	149	188
-4	182	183	8
-2	403	417	6
0	147	140	5
2	334	320	187
4	502	502	187
6	356	339	188
10	143	145	11
12	126	124	11
** K= 3 L= 6 **			
-13	72	66	11
-11	110	113	9
-9	152	157	8
-7	137	134	10
-5	110	112	11
-3	137	144	7
-1	145	148	7
1	152	152	8
3	166	170	8
5	213	218	7
7	194	183	7
9	110	112	9
11	69	77	10
** K= 4 L= 6 **			
-12	153	148	12
-10	390	390	9
-8	451	450	9
-6	312	306	8
-2	98	103	187
0	108	114	7
2	463	448	8
4	573	571	8
6	453	453	8
8	150	142	9
** K= 5 L= 6 **			
-7	101	90	188
-5	100	97	185
3	80	90	187
5	84	86	189
** K= 6 L= 6 **			
-10	85	81	191
-8	97	92	191
-6	70	56	12
-4	276	265	11
-2	556	532	7
0	223	216	10
4	162	174	188
8	129	123	10
10	235	229	11
12	237	224	10
** K= 7 L= 6 **			
-11	153	154	191
-9	161	168	190
-7	170	168	190
-5	221	215	190
-3	307	309	189
-1	367	350	188
1	261	257	190
3	240	247	188
5	187	216	188
7	169	175	190
9	182	184	191
11	177	185	191
** K= 8 L= 6 **			
-10	123	131	10
-8	140	131	10
-4	198	200	189
-2	281	273	189
0	128	128	190
2	64	73	10
4	184	182	10
6	94	123	8
10	124	146	192
** K= 9 L= 6 **			
-9	90	87	191
-7	107	91	191
5	89	105	190
** K= 10 L= 6 **			

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
-4	138	130	10
-2	171	171	10
0	95	85	10
4	151	156	190
6	81	90	191
** K= 11	L= 6 **		
-3	116	119	11
-1	121	117	10
1	137	135	11
3	158	171	11
** K= 1	L= 7 **		
-13	268	269	11
-11	219	223	9
-7	134	137	188
-3	462	441	7
-1	626	641	8
1	509	491	9
3	234	230	7
5	62	72	190
7	105	113	187
9	80	80	12
11	318	308	9
13	277	278	11
** K= 3	L= 7 **		
-11	134	135	10
-9	388	373	9
-7	596	583	8
-5	369	376	10
-3	177	183	7
3	376	359	7
5	603	594	8
7	562	552	8
9	208	203	12
** K= 4	L= 7 **		
-10	104	114	191
-8	170	171	189
-6	235	225	188
-4	177	175	190
-2	225	231	186
0	183	193	187
2	161	149	189
4	195	194	188
6	221	219	188
8	185	176	189
10	118	112	191
12	84	88	190
** K= 5	L= 7 **		
-9	173	175	189
-7	278	273	189
-5	186	184	188
-3	128	116	6
-1	364	336	7
1	207	224	8
5	288	287	188
7	263	261	189
11	112	109	10
** K= 6	L= 7 **		
-12	117	95	194
-10	90	107	191
-8	101	106	189
-6	89	101	189
-4	150	156	187
-2	207	202	188
0	249	248	188
2	203	211	188
4	159	164	188
6	91	104	189
8	89	72	192
10	85	97	190
** K= 7	L= 7 **		
-11	118	122	190
-7	104	101	10
-3	246	239	188
-1	370	347	190
1	256	260	191
3	99	113	187
11	189	193	190
** K= 8	L= 7 **		
-10	125	121	10
-8	85	79	11
-6	66	69	11
-4	112	114	9
-2	175	172	9
0	188	186	10
2	149	149	12
4	117	113	11
** K= 9	L= 7 **		
-7	204	187	190
-5	99	89	192
-1	179	176	10
1	118	115	11
5	155	161	190
** K= 10	L= 7 **		
-6	213	206	11
-4	201	191	11
-2	203	190	9
0	152	148	10
2	151	140	11
4	185	177	11
6	191	200	11
** K= 11	L= 7 **		
-1	172	172	191
1	140	134	191
** K= 0	L= 8 **		
-14	223	231	12
-12	421	424	10
-10	390	394	9
-8	204	196	9
-2	464	496	6
0	648	633	9
2	851	835	7
4	451	434	9
6	181	175	6
10	90	84	11
12	264	262	10
** K= 1	L= 8 **		
-1	64	64	186
1	84	87	186

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	$/F_o/$	$/F_c/$	PHI
** K= 2	L= 8 **		
-12	142	138	191
-10	84	75	190
-8	168	159	9
-6	372	365	9
-4	393	389	8
-2	100	109	7
0	202	195	188
2	296	303	187
6	310	305	7
8	292	297	10
10	195	185	10
** K= 3	L= 8 **		
-9	95	93	189
-7	149	142	188
-5	191	184	187
-3	149	150	189
-1	109	122	190
1	120	125	188
3	117	118	188
5	123	119	188
7	122	121	190
9	119	127	189
11	122	108	189
** K= 4	L= 8 **		
-8	241	239	190
-6	417	424	190
-4	481	494	188
-2	289	298	186
4	152	152	187
6	408	403	188
8	354	353	191
10	267	266	190
** K= 5	L= 8 **		
-5	56	68	12
-3	106	100	8
-1	117	118	4
** K= 6	L= 8 **		
-10	185	184	190
-6	107	103	11
-4	88	88	9
-2	139	124	189
0	284	278	191
2	394	404	188
4	174	167	190
8	114	122	9
** K= 7	L= 8 **		
-9	211	205	10
-7	205	197	10
-5	183	183	9
-3	190	190	9
-1	240	228	10
1	247	269	10
3	255	276	10
5	217	224	10
7	180	182	9
9	132	137	10
** K= 8	L= 8 **		
-6	174	166	189
-4	141	132	190
-2	68	56	5
0	202	196	10
2	194	211	10
8	118	124	192
** K= 9	L= 8 **		
-5	95	99	10
-3	110	104	10
** K= 10	L= 8 **		
-6	94	107	13
-4	107	105	11
0	140	142	190
2	142	144	191
** K= 11	L= 8 **		
-1	142	156	190
1	106	106	191
** K= 1	L= 9 **		
-13	124	128	192
-11	337	337	189
-9	369	371	189
-7	212	209	188
-3	138	163	6
-1	50	62	190
1	379	397	188
3	535	530	188
5	415	408	188
7	102	97	189
13	79	85	191
** K= 3	L= 9 **		
-7	190	192	190
-5	450	435	189
-3	727	717	187
-1	315	329	190
1	100	101	188
7	279	270	189
9	384	373	190
11	353	343	189
** K= 4	L= 9 **		
-12	101	97	11
-8	92	94	11
-6	121	128	10
-4	204	196	9
-2	271	254	7
0	179	172	10
2	192	186	7
4	138	147	7
6	110	111	10
8	130	131	10
10	151	136	10
12	115	118	10
** K= 5	L= 9 **		
-11	149	143	191
-9	142	131	192
-5	204	209	9
-3	327	324	8
-1	150	149	8
1	154	147	188
3	281	283	188
5	165	165	188

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
7	83	74	10
9	187	187	11
11	157	162	10
** K=	6	L= 9	**
-10	131	124	11
-8	127	117	13
-6	108	110	12
-4	103	113	8
-2	81	85	10
0	116	126	8
2	177	176	8
4	190	203	8
6	153	170	9
8	107	109	10
10	81	72	10
** K=	7	L= 9	**
-9	224	221	10
-7	99	103	10
-3	106	101	190
1	207	221	10
3	301	303	10
5	220	220	10
** K=	8	L= 9	**
-8	148	149	190
-6	135	134	190
-4	81	80	191
0	99	102	190
2	148	149	190
4	134	157	191
** K=	9	L= 9	**
-5	140	133	11
-3	231	222	8
-1	60	63	13
1	90	69	191
3	151	152	190
** K=	10	L= 9	**
-4	180	194	191
-2	233	231	190
0	179	183	191
2	147	157	190
4	108	123	190
** K=	0	L= 10	**
-12	161	157	190
-10	378	370	190
-8	533	534	189
-6	381	366	190
-4	162	182	188
0	112	113	187
2	438	434	187
4	624	616	188
6	581	556	188
8	239	234	191
10	84	83	187
** K=	1	L= 10	**
5	77	72	6
** K=	2	L= 10	**
-12	104	81	190
-8	171	165	10
-4	268	253	187
-2	392	418	188
0	294	294	189
4	212	201	8
6	191	196	8
10	229	218	189
12	196	196	191
** K=	3	L= 10	**
-11	98	87	10
-9	85	86	9
-5	112	117	7
-3	169	168	7
-1	160	178	7
1	128	126	10
3	111	106	10
5	91	92	9
7	70	68	10
** K=	4	L= 10	**
-12	156	149	10
-4	329	330	8
-2	517	476	9
0	385	381	9
2	175	179	7
8	127	121	10
10	285	283	9
** K=	5	L= 10	**
-1	74	79	191
1	90	86	189
** K=	6	L= 10	**
-10	201	192	11
-8	271	273	10
-6	146	156	12
-2	118	117	190
2	138	141	10
4	289	296	10
6	291	279	9
** K=	7	L= 10	**
-9	186	182	190
-7	235	227	190
-5	212	212	191
-3	214	220	189
-1	161	167	190
1	144	160	190
3	201	212	189
5	224	226	190
7	187	194	191
9	141	144	192
** K=	8	L= 10	**
-8	153	146	192
-6	65	79	191
-4	95	90	8
-2	204	191	9
0	107	100	11
4	195	187	190
6	154	151	191
** K=	9	L= 10	**

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	/F_o/	/F_c/	PHI
-1	107	101	190
** K= 10	L= 10 **		
-2	127	128	191
** K= 1	L= 11 **		
-9	195	204	10
-7	360	366	10
-5	403	397	8
-3	161	181	7
-1	63	62	190
1	114	109	188
3	109	102	9
5	360	359	8
7	323	322	11
9	242	231	10
** K= 3	L= 11 **		
-11	251	238	11
-3	298	277	8
-1	411	425	10
1	556	542	8
3	268	262	10
5	69	49	8
11	222	211	10
** K= 4	L= 11 **		
-10	134	119	191
-8	120	127	189
-2	134	139	190
0	170	170	190
2	199	194	189
4	154	156	189
6	131	134	188
8	92	88	190
10	87	78	191
** K= 5	L= 11 **		
-11	90	80	191
-7	171	163	11
-5	142	130	11
-1	226	204	190
1	241	249	189
3	83	85	187
5	153	156	8
7	175	173	10
** K= 6	L= 11 **		
-8	88	104	190
-6	140	133	191
-4	123	129	192
-2	119	110	191
4	112	101	189
6	125	122	190
8	118	124	191
** K= 7	L= 11 **		
-9	114	121	191
-7	218	223	191
-5	229	229	190
-3	102	90	187
-1	90	81	10
5	204	212	190
7	190	190	193
** K= 8	L= 11 **		
-6	133	129	11
-4	160	162	10
-2	149	148	8
0	68	69	12
6	110	117	10
** K= 9	L= 11 **		
-5	88	81	10
-1	138	141	192
1	165	169	190
** K= 10	L= 11 **		
0	170	166	12
** K= 0	L= 12 **		
-8	196	202	10
-6	424	423	10
-4	556	632	7
-2	304	319	10
0	116	114	9
4	127	121	8
6	308	308	9
8	388	385	10
10	349	347	9
** K= 1	L= 12 **		
-3	79	90	186
1	66	63	187
** K= 2	L= 12 **		
-10	203	189	11
-6	118	111	190
-4	193	189	188
0	251	247	8
2	358	348	9
4	228	229	8
8	140	135	190
10	104	108	189
** K= 3	L= 12 **		
-7	73	92	190
-5	88	90	188
-1	95	99	188
1	139	141	187
3	133	143	188
5	105	107	190
7	74	70	191
9	71	56	190
** K= 4	L= 12 **		
-10	262	252	190
-8	153	131	190
-4	79	67	8
0	290	295	190
2	409	403	189
4	300	303	189
6	64	75	190
** K= 6	L= 12 **		
-6	219	221	191
-4	309	321	188
-2	125	127	192

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	$/F_o/$	$/F_c/$	PHI
6	106	124	192
8	193	198	192
** K= 7 L= 12 **			
-7	118	138	11
-5	187	201	10
-3	256	254	9
-1	201	202	10
1	190	177	9
3	145	136	10
5	119	122	11
7	146	145	11
** K= 8 L= 12 **			
-6	124	119	11
-4	174	166	10
0	105	109	189
2	169	163	189
** K= 1 L= 13 **			
-5	266	277	189
-3	338	399	189
-1	292	299	190
1	92	98	188
3	86	83	9
5	55	64	9
7	90	89	192
9	261	255	190
11	214	217	192
** K= 3 L= 13 **			
-9	334	325	190
-7	215	213	191
1	263	263	189
3	418	415	189
5	388	368	189
7	131	133	193
** K= 4 L= 13 **			
-10	119	113	10
-8	144	137	10
-6	112	118	12
-4	128	143	8
0	78	80	12
2	121	124	10
4	139	145	10
6	133	136	10
8	88	100	11
** K= 5 L= 13 **			
-9	142	133	12
-5	110	113	188
-3	205	187	190
-1	78	94	193
1	68	69	8
3	207	198	9
5	168	170	9
9	113	121	190
** K= 6 L= 13 **			
-6	77	80	8
-2	129	128	11
0	108	109	13
2	90	87	12
** K= 7 L= 13 **			
-5	158	166	10
-3	240	245	10
-1	175	167	11
** K= 8 L= 13 **			
-4	92	103	191
-2	135	136	191
0	115	137	191
** K= 0 L= 14 **			
-10	91	80	192
-4	239	271	189
-2	385	403	190
0	470	481	188
2	249	250	190
4	73	67	188
8	120	101	190
10	236	228	190
** K= 1 L= 14 **			
1	66	66	8
** K= 2 L= 14 **			
-10	94	106	192
-8	213	202	191
-6	192	192	190
-2	96	114	11
0	125	134	10
4	234	231	189
6	217	211	191
8	131	124	190
** K= 3 L= 14 **			
-5	98	87	11
-3	82	87	9
1	55	54	9
3	88	77	8
5	89	94	9
7	86	87	10
9	76	72	10
** K= 4 L= 14 **			
-8	264	255	11
-6	262	257	10
-4	127	113	9
2	95	103	10
4	279	270	10
6	252	247	12
8	187	170	10
** K= 6 L= 14 **			
-4	120	126	9
-2	228	215	11
0	257	243	10
2	102	95	11
** K= 7 L= 14 **			
-3	113	140	191
-1	169	172	191
1	204	205	190
3	167	175	190
5	123	124	191

Table A.2. Observed and Calculated Structure Factors for $Os_4(CO)_{15}$.

H	$/F_o/$	$/F_c/$	PHI
** K= 8	L= 14 **		
-2	126	113	192
0	120	121	191
** K= 1	L= 15 **		
-9	65	80	11
-5	97	68	189
-1	230	249	10
1	337	331	9
3	236	228	9
7	62	61	191
** K= 3	L= 15 **		
-9	124	130	12
-7	276	275	11
-5	392	370	9
-3	156	180	11
5	203	199	11
7	275	266	11
** K= 4	L= 15 **		
-6	131	123	190
-4	161	156	189
-2	100	116	191
0	106	114	189
2	93	83	190
8	86	91	192
** K= 5	L= 15 **		
-7	118	111	192
-5	152	141	190
-1	115	120	10
1	171	162	11
7	137	129	191
** K= 6	L= 15 **		
0	103	100	189
2	115	112	191
4	91	92	192
** K= 7	L= 15 **		
-1	156	148	192
1	207	208	190
3	130	131	190
** K= 0	L= 16 **		
-8	156	158	14
-2	62	64	12
0	241	252	10
2	387	380	9
4	339	327	10
6	142	132	12
** K= 2	L= 16 **		
-6	143	149	10
-4	204	220	10
-2	146	140	11
2	131	120	190
4	99	84	190
** K= 3	L= 16 **		
1	74	66	190
** K= 4	L= 16 **		
-6	205	201	190
-4	286	268	190
-2	184	188	191
6	106	82	193
** K= 6	L= 16 **		
0	130	131	190
2	223	210	191
4	183	170	191
** K= 1	L= 17 **		
-7	183	178	190
1	76	83	190
3	223	224	190
5	211	197	192
** K= 3	L= 17 **		
-5	174	175	190
-3	231	258	191
-1	270	281	190
1	125	137	191
** K= 4	L= 17 **		
-2	100	109	11
0	114	124	11
2	104	106	11
4	93	83	10
** K= 5	L= 17 **		
-3	102	99	13
-1	104	93	12
3	125	118	190
** K= 0	L= 18 **		
-6	267	264	190
-4	112	130	194
2	62	72	192
4	199	187	192
** K= 2	L= 18 **		
-2	115	138	191
0	175	185	191
2	110	105	191
** K= 3	L= 18 **		
3	70	55	12
** K= 4	L= 18 **		
-2	161	172	11
0	226	218	11
2	159	143	10

Table B.1. U_{ij} or U_{iso} Values (*100) for $Os_4(CO)_{16}$.

	U11(U)	U22	U33	U12	U13	U23
Os(1)	2.61(4)	2.79(5)	2.98(4)	-0.94(3)	-0.75(3)	-0.20(3)
Os(2)	2.34(4)	2.24(4)	3.14(4)	-0.20(3)	-0.78(3)	-0.31(3)
Os(3)	2.73(4)	2.21(4)	3.14(4)	-0.52(3)	-0.64(3)	-0.48(3)
Os(4)	2.40(4)	2.60(5)	3.44(4)	-0.34(3)	-0.71(3)	0.38(3)
O(11)	4.4(3)					
O(12)	5.9(4)					
O(13)	7.8(5)					
O(14)	6.3(5)					
O(21)	4.8(4)					
O(22)	4.2(3)					
O(23)	6.6(5)					
O(24)	5.1(4)					
O(31)	5.8(4)					
O(32)	6.1(4)					
O(33)	7.5(5)					
O(34)	6.4(5)					
O(41)	5.1(4)					
O(42)	4.8(4)					
O(43)	9.0(6)					
O(44)	6.2(4)					
C(11)	3.2(4)					
C(12)	3.3(4)					
C(13)	4.6(5)					
C(14)	3.8(5)					
C(21)	3.1(4)					
C(22)	2.8(4)					
C(23)	4.1(5)					
C(24)	3.6(5)					
C(31)	3.7(5)					
C(32)	4.1(5)					
C(33)	4.7(5)					
C(34)	4.4(5)					
C(41)	4.0(5)					
C(42)	3.4(4)					
C(43)	5.9(6)					
C(44)	4.6(5)					

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-10,	0, L		-11	844	918	175	7	440	448	287
2	890	983	198	-10	362	369	335		-7,	2, L	
	-10,	1, L		-9	1107	1164	162	-12	1417	1502	167
-1	698	764	258	-7	649	693	232	-10	1659	1786	155
	-9,	0, L		-5	453	225	582	-8	823	883	158
1	366	405	335	-4	585	507	606	-4	743	588	420
2	714	788	204	-2	812	765	259	-3	451	442	534
4	433	460	294	-1	840	794	230	1	1056	1055	161
5	727	797	214	1	1135	1157	181	3	1086	1174	165
	-9,	1, L		2	496	563	320	4	639	696	225
-9	887	967	170	3	746	786	215	5	342	371	378
-8	516	510	243	4	580	633	267	6	806	883	202
-7	1548	1606	181		-8,	3, L		7	311	300	411
-6	594	536	368	-8	740	743	220	8	609	626	249
-5	1575	1158	329	-6	885	890	287		-7,	3, L	
-4	529	552	425	-2	949	919	299	-11	628	674	219
-2	828	874	215	0	1508	1503	215	-5	460	437	462
0	860	959	203	1	402	356	427	-3	1936	1483	277
2	394	400	334	2	1167	1230	195	-2	431	313	516
3	321	414	428	4	710	782	269	-1	1966	1856	226
4	382	322	341		-8,	4, L		0	776	760	257
	-9,	2, L		-7	1470	1460	218	1	1288	1308	208
-4	602	513	491	-5	1127	924	340	2	511	507	303
-3	377	418	571	-2	473	434	614	3	359	331	402
-1	533	643	349	-1	478	416	505	6	634	669	247
0	517	552	320	0	606	547	360		-7,	4, L	
2	897	978	211	1	328	348	610	-10	1025	1100	178
	-9,	3, L			-7,	0, L		-8	992	1034	193
-3	1027	865	329	1	651	697	165	-4	1248	1010	301
	-8,	0, L		2	423	466	227	-3	689	501	501
1	1597	1672	153	3	219	260	412	-2	2001	1642	268
3	951	1011	157	5	704	736	166	-1	667	589	384
4	804	856	175	6	451	492	246	0	1333	1280	226
5	451	473	267	7	542	581	231	2	828	866	260
6	1200	1218	169	8	449	457	272	3	637	658	288
7	317	237	378	9	315	276	381	4	619	642	295
8	788	807	199	10	707	737	217	5	665	802	286
	-8,	1, L			-7,	1, L			-7,	5, L	
-12	715	763	201	-13	574	555	228	-7	526	618	373
-10	1530	1599	160	-11	1079	1135	155	2	780	737	305
-9	345	354	322	-10	345	314	304		-6,	0, L	
-8	1706	1804	146	-9	1108	1203	157	1	961	985	138
-6	1166	1229	170	-7	301	342	277	3	1635	1672	132
-3	457	489	353	-5	1200	1250	185	4	1059	1042	139
-2	808	795	194	-4	427	380	729	5	1256	1313	149
-1	689	711	204	-3	2421	2340	199	6	892	917	134
0	695	737	202	-2	512	544	277	7	930	903	138
2	253	296	478	-1	1851	1894	161	8	251	226	391
3	964	1041	171	1	1219	1265	172	9	764	800	184
4	364	432	358	2	943	1008	150	10	736	746	195
5	1251	1366	181	3	668	689	181	11	817	826	196
6	594	597	242	4	1174	1275	165		-6,	1, L	
7	859	874	190	5	581	614	219	-11	561	591	200
	-8,	2, L		6	591	652	226	-9	373	453	229

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{12}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-6, 1, L				-6, 4, L			-4	1100	1121	143
-8	454	479	189	-10	455	488	277	-3	712	776	320
-7	379	351	203	-8	376	373	318	-2	396	318	335
-6	1718	1781	123	-7	559	537	252	-1	991	1026	152
-5	885	917	139	-6	506	449	308	0	1148	1116	146
-4	3205	3090	174	-5	1687	1651	222	1	1773	1869	131
-3	1458	1303	211	-4	1226	1058	239	2	1942	1993	127
-2	2190	2182	167	-3	2898	2240	261	3	1766	1860	131
0	646	628	171	-2	1580	1214	232	4	1346	1398	142
1	800	813	148	-1	1679	1451	234	5	1413	1455	143
2	357	335	260	0	344	361	544	7	1340	1398	156
3	1383	1405	153	1	537	475	325	8	791	822	159
4	430	473	231	2	664	655	269	9	1081	1150	172
5	574	635	191	4	932	954	210	10	635	664	218
6	589	623	190	6	320	382	469	11	695	702	209
7	453	422	231	7	407	383	382		-5, 2, L		
8	906	911	167		-6, 5, L			-14	480	494	260
9	931	967	173	-10	921	951	193	-13	593	607	207
10	1191	1237	186	-6	863	837	236	-12	370	393	282
11	671	720	237	-5	406	495	538	-11	583	615	172
	-6, 2, L			-4	1490	1262	245	-10	531	571	178
-13	1039	1128	159	-3	1060	778	310	-9	840	860	127
-11	1158	1204	162	-2	1333	1090	295	-8	2019	2149	122
-7	1034	1112	146	0	690	564	380	-7	1257	1292	136
-6	427	414	230	1	493	507	471	-6	2741	2798	118
-5	1984	1970	161	2	733	704	301	-5	1312	1336	145
-4	893	821	251	3	599	561	332	-4	1869	1823	161
-3	2399	1942	263	4	821	820	255	-3	730	625	303
-1	845	809	203	6	441	551	419	-2	677	604	328
0	750	794	200		-6, 6, L			-1	751	692	211
1	758	815	184	-2	636	505	487	1	1312	1332	166
2	1058	1072	156	-1	540	487	560	3	363	371	281
3	974	1043	154	0	821	710	379	4	358	351	280
5	713	760	180	2	406	383	586	5	656	694	178
6	785	839	171	3	708	641	343	6	923	990	150
8	782	764	192		-5, 0, L			7	960	1038	152
10	286	319	466	1	489	508	148	8	1156	1263	171
	-6, 3, L			2	1399	1435	115	9	277	289	440
-12	1038	1091	155	3	1490	1561	120	10	840	867	190
-11	466	487	252	4	1132	1151	133		-5, 3, L		
-10	519	571	220	5	1641	1710	131	-13	855	854	169
-9	517	551	215	7	366	364	245	-12	336	326	312
-4	480	407	427	9	640	620	169	-9	993	1065	152
-3	836	640	353	11	641	614	203	-8	358	385	250
-2	910	766	298		-5, 1, L			-7	1424	1510	146
-1	993	915	227	-15	622	605	223	-6	842	833	144
1	636	638	235	-14	667	665	200	-5	633	653	199
2	877	890	191	-12	886	907	151	-3	494	400	429
3	349	403	379	-11	261	250	329	-1	748	670	268
4	1123	1188	179	-10	749	783	139	0	390	307	378
5	1025	1057	175	-8	641	620	138	1	1335	1303	181
6	803	893	211	-7	1155	1196	129	2	757	772	196
7	689	691	219	-6	518	552	147	3	1248	1261	186
8	696	730	226	-5	1579	1591	113	4	1531	1598	176

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-5, 3, L			-5	865	782	294	-2	2103	1820	214
5	729	772	192	-4	815	746	339	-1	1967	1915	180
6	1231	1330	180	-3	1040	849	301	0	2545	2471	145
10	506	546	279	-2	952	809	338	1	1547	1506	143
	-5, 4, L			1	623	535	474	2	1461	1489	143
-13	676	687	208	2	985	840	320	3	1475	1460	140
-11	819	867	173	3	797	656	346	5	1474	1545	149
-10	302	265	346		-4, 0, L			6	849	863	141
-9	737	764	175	1	209	198	252	7	1634	1676	151
-7	641	614	187	2	1448	1418	100	9	738	746	165
-6	1166	1167	163	3	508	486	130	10	341	382	344
-5	766	771	210	4	1184	1176	116	12	471	471	290
-4	1459	1402	214	5	1385	1403	118	13	652	682	241
-3	1298	1060	223	7	1289	1290	136		-4, 3, L		
-2	587	470	438	8	907	879	124	-15	357	401	367
0	819	800	262	9	452	413	201	-14	415	403	288
1	1181	1106	193	10	861	861	145	-13	428	424	262
2	1164	1177	180	12	465	456	265	-12	505	499	211
3	1595	1649	216	13	516	520	260	-11	710	750	155
4	1127	1115	168		-4, 1, L			-10	1487	1536	138
5	1026	1031	176	-16	414	465	335	-9	1091	1132	154
6	899	959	201	-15	585	591	230	-8	1832	1889	126
8	920	1030	212	-13	606	649	199	-7	1610	1655	128
9	415	489	379	-12	1041	1044	149	-6	1094	1131	147
	-5, 5, L			-11	306	306	273	-5	972	990	149
-11	334	349	374	-10	836	858	124	-2	376	322	542
-10	429	476	300	-7	599	610	124	-1	353	363	550
-9	505	524	262	-6	592	633	115	0	360	292	381
-8	798	836	196	-3	695	725	144	1	287	281	436
-7	1539	1605	185	-2	1393	1272	208	3	1226	1264	170
-6	1195	1216	190	-1	1985	1963	131	4	574	570	210
-5	1846	1844	206	0	2414	2403	115	5	853	861	163
-4	1300	1220	209	1	2664	2646	110	6	1109	1175	160
-3	1381	1145	228	2	1598	1630	117	8	746	836	191
-2	764	547	342	3	2148	2215	115	9	852	889	186
-1	426	389	566	4	733	755	126	11	611	716	252
0	577	446	377	5	257	180	284		-4, 4, L		
2	841	781	244	6	423	444	200	-14	607	598	221
6	469	522	369	7	1265	1263	145	-12	467	508	255
7	733	750	252	8	367	353	244	-11	915	944	150
8	508	571	354	9	718	731	162	-9	605	639	169
	-5, 6, L			11	239	265	463	-8	232	231	377
-8	713	697	230	12	932	927	170	-6	457	438	227
-7	370	400	438		-4, 2, L			-5	558	598	215
-6	779	817	255	-14	1007	1018	158	-2	953	773	258
-5	549	581	378	-13	302	348	356	-1	1426	1208	214
-4	420	311	518	-12	1080	1124	159	0	2231	2025	220
0	619	533	436	-10	1122	1127	144	1	1752	1674	204
2	990	898	277	-9	1307	1310	129	2	2479	2376	179
3	814	746	291	-8	1043	1094	136	3	1006	985	176
4	716	772	324	-7	2153	2202	108	4	1452	1511	192
5	1265	1234	224	-6	994	1014	129	5	579	570	236
	-5, 7, L			-5	934	960	135	7	521	528	258
-6	489	506	424	-3	803	810	174	8	723	783	225

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-4,	4, L		1	488	509	599	1	1917	1856	127
9	393	421	371	2	1342	1114	264	2	1002	957	146
10	382	432	381	3	462	347	575	4	842	830	120
	-4,	5, L			-3,	0, L		5	1379	1409	135
-13	917	915	176	2	885	879	101	6	817	833	139
-12	344	380	349	3	513	532	110	7	1013	1000	139
-11	906	956	167	4	1729	1770	94	10	986	1006	146
-9	1004	1045	167	5	321	330	184	12	1462	1484	178
-8	1124	1199	169	6	1188	1161	117	14	1071	1088	183
-7	1117	1159	176	7	1295	1266	123		-3,	3, L	
-6	1510	1551	177	9	1222	1253	146	-15	335	430	384
-5	1016	1007	172	10	429	491	221	-14	846	875	170
-4	515	517	311	11	488	524	213	-13	285	295	364
-2	666	566	334	12	298	278	348	-12	913	929	136
-1	1774	1470	259		-3,	1, L		-11	1149	1207	151
0	1136	1025	240	-15	485	485	261	-10	1145	1185	147
1	1866	1708	240	-13	1186	1177	163	-9	1426	1473	129
2	748	744	265	-12	731	720	147	-8	1006	1033	141
3	960	980	215	-11	1273	1285	139	-7	356	372	207
4	823	827	221	-10	1288	1299	131	-5	884	914	132
6	1071	1143	198	-9	289	234	227	-4	1555	1554	126
7	345	377	458	-8	1007	1001	120	-3	1396	1409	154
8	1084	1144	203	-7	596	627	111	-2	2332	2352	172
10	435	487	387	-6	421	427	132	-1	1536	1398	218
	-4,	6, L		-3	445	447	128	0	968	921	186
-11	324	389	415	-2	1804	1796	141	1	644	590	200
-10	545	637	273	0	1708	1673	110	3	1055	1066	158
-9	1012	1055	173	1	402	421	166	4	398	332	245
-8	1030	1082	175	2	1085	1093	115	5	1534	1599	152
-7	1077	1132	183	3	901	883	124	6	569	569	191
-6	1270	1332	200	4	936	952	125	7	826	831	156
-5	481	499	333	5	950	982	130	8	1272	1322	174
-4	677	695	275	6	2152	2160	116	10	1035	1084	155
2	331	411	623	7	447	490	187	11	453	458	283
4	825	848	269	8	1925	1940	131	12	417	359	316
5	477	493	408	9	446	467	214		-3,	4, L	
6	556	502	340	10	599	607	181	-14	392	424	311
7	798	823	262	11	1043	1058	154	-12	935	956	150
	-4,	7, L		13	1409	1370	177	-11	825	847	147
-8	359	333	419		-3,	2, L		-10	832	903	141
-4	492	412	409	-16	439	463	310	-9	1228	1246	146
-1	857	619	317	-15	796	821	187	-7	870	865	125
0	925	737	316	-14	622	642	199	-6	602	621	158
1	1857	1527	276	-13	387	392	265	-5	447	468	207
2	1011	830	293	-12	262	249	315	-1	1862	1555	222
3	1908	1669	260	-10	632	636	140	1	1713	1667	204
4	391	426	623	-8	1032	1040	122	3	867	829	180
5	1033	973	263	-7	388	417	160	4	661	644	208
	-4,	8, L		-5	1353	1363	102	5	931	956	168
-6	941	943	266	-4	1695	1700	99	6	348	437	352
-5	923	848	278	-3	3198	3161	110	7	1713	1743	175
-4	836	760	322	-2	2330	2328	157	9	1316	1361	193
-1	502	379	531	-1	4344	3920	172	10	335	321	399
0	1237	1030	278	0	1646	1528	150	11	471	467	305

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-3, 4, L			-8	488	477	329	15	332	286	379
12	781	865	234	-6	605	613	306		-2, 2, L		
	-3, 5, L			-3	1114	938	246	-15	785	773	179
-13	539	590	249	-2	681	605	381	-14	419	426	266
-12	385	371	302	-1	2166	1787	251	-13	257	252	355
-9	641	657	173	0	647	510	420	-12	858	879	141
-7	914	916	141	1	2301	1864	254	-11	615	615	151
-6	263	297	384	2	367	287	693	-10	1194	1196	135
-4	1313	1315	191	3	1059	912	289	-9	1289	1295	119
-3	1174	1196	174		-3, 9, L			-8	867	884	124
-2	2982	2626	197	-3	451	402	533	-7	792	809	113
-1	1507	1240	219	-2	859	753	326	-6	1549	1530	92
0	3455	2837	209	-1	395	315	623	-5	396	405	131
1	854	782	265	0	951	844	308	-4	1938	1931	84
2	1424	1340	201		-2, 0, L			-3	2240	2191	88
3	711	674	243	1	737	729	90	-2	2771	2786	107
5	692	700	232	2	1165	1139	80	-1	392	459	390
6	748	776	220	3	1604	1584	78	0	1288	1275	169
7	658	664	233	4	571	534	98	2	1443	1389	118
8	442	440	348	6	558	561	116	3	234	227	306
11	701	787	258	7	1420	1364	109	4	3626	3588	109
	-3, 6, L			8	726	736	122	6	2300	2334	118
-12	279	268	432	9	1835	1800	123	7	794	809	133
-11	884	887	176	11	960	938	139	8	1235	1247	147
-10	842	884	177	12	580	581	182	9	1587	1617	145
-9	1128	1146	164	14	629	607	207	10	232	213	408
-8	865	869	169		-2, 1, L			11	1568	1568	160
-7	909	941	166	-15	655	634	201	13	868	833	180
-4	589	599	264	-14	894	925	160		-2, 3, L		
-3	1120	1112	199	-13	801	804	149	-15	537	569	245
-2	805	693	259	-12	862	875	134	-12	872	855	133
-1	1757	1467	241	-11	1076	1108	145	-10	1263	1281	134
0	584	479	358	-9	532	558	136	-9	454	454	169
1	656	587	330	-8	1420	1412	103	-8	319	291	209
2	390	350	503	-7	705	680	105	-7	1493	1504	110
4	856	828	232	-6	1683	1614	85	-6	989	1003	120
6	1024	1023	213	-5	1242	1237	83	-5	3116	3157	98
7	632	625	289	-4	558	547	87	-4	1644	1648	107
8	456	394	395	-3	970	974	84	-3	3584	3612	110
9	1005	1020	229	-2	259	228	206	-2	983	991	136
	-3, 7, L			-1	506	469	295	-1	2248	2111	169
-10	708	734	222	0	314	292	254	0	400	348	363
-9	473	408	279	2	1308	1297	95	1	306	271	352
-8	853	909	202	3	1613	1621	94	2	765	750	151
-6	561	598	289	4	1165	1140	106	3	955	915	141
-5	459	480	358	5	2771	2794	100	4	1040	1043	144
-2	362	333	520	7	2340	2384	112	5	300	299	291
0	1299	1098	221	8	338	368	241	6	223	278	411
2	1385	1198	231	9	834	807	128	7	486	516	213
4	469	473	473	10	254	239	342	8	1304	1321	161
5	454	458	467	11	424	422	228	9	325	356	326
6	735	711	308	12	672	652	174	10	1443	1492	171
8	1166	1165	238	13	340	372	355	11	296	334	379
	-3, 8, L			14	337	320	360	12	619	638	225

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{12}$.
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-2,	3, L		-6	1194	1190	172	9	514	591	172
13	527	539	270	-5	628	626	207	10	1045	1031	148
	-2,	4, L		-4	2295	2368	164	11	851	834	134
-14	677	683	196	-3	716	774	226	13	431	455	241
-13	706	707	177	-2	2564	2431	191	14	838	806	169
-12	886	888	150	-1	361	302	484	16	424	406	313
-11	599	629	172	0	1567	1341	205		-1,	1, L	
-10	1007	989	143	3	488	548	371	-15	619	640	212
-9	272	286	291	4	419	385	398	-13	952	978	149
-8	221	291	342	5	621	573	254	-10	1281	1237	123
-7	1160	1216	135	8	391	378	417	-9	1345	1387	115
-6	837	865	131	9	937	934	218	-8	2379	2409	97
-5	1003	1008	140	11	955	944	227	-7	1293	1316	97
-4	1248	1203	138		-2,	7, L		-6	2881	2796	80
-3	332	360	291	-11	818	824	194	-5	355	343	119
-2	490	502	251	-9	787	785	194	-4	1311	1281	70
0	419	390	382	-8	425	405	293	-3	311	366	127
1	472	449	319	-6	749	789	201	-2	287	278	117
2	380	391	338	-5	602	601	235	1	407	411	139
3	1035	1035	157	-4	405	383	353	2	911	856	96
4	1761	1753	161	-3	696	654	243	3	383	367	137
5	468	445	247	3	333	410	598	4	1591	1561	88
6	2558	2512	153	4	575	536	344	5	1477	1459	95
7	352	412	334	5	1344	1286	215	6	1093	1099	111
8	1766	1817	169	7	1698	1654	240	7	1248	1265	115
9	301	311	383	8	531	516	385	8	521	482	141
10	509	486	246	9	1107	1082	228	9	531	523	161
11	386	349	343		-2,	8, L		10	1584	1576	136
13	647	663	251	-9	632	664	258	11	287	346	326
	-2,	5, L		-8	489	483	311	12	1158	1137	167
-14	594	586	234	-7	869	917	214	13	425	424	274
-13	396	455	319	-6	379	423	429	14	690	675	204
-11	778	765	163	-5	422	427	390		-1,	2, L	
-10	777	766	149	-4	882	948	244	-16	312	353	413
-9	921	919	133	-3	395	287	480	-15	504	536	245
-8	1253	1274	148	-2	1766	1553	234	-13	947	973	144
-7	706	723	150	-1	455	394	491	-12	702	688	149
-6	637	677	165	0	1924	1604	242	-11	630	636	145
-5	1327	1379	156	2	459	366	522	-10	1563	1559	121
-4	545	540	200	4	997	906	281	-8	843	807	112
-3	2087	2104	163	6	1444	1372	226	-7	243	269	200
-2	1237	1258	185		-2,	9, L		-6	318	273	144
-1	2841	2481	189	-5	876	846	258	-5	217	266	199
1	874	778	233	-4	388	306	534	-4	580	564	87
3	1335	1278	200	-3	1764	1601	248	-3	398	394	120
5	2346	2294	179	-1	1840	1573	254	-2	456	436	124
7	1514	1542	192	1	891	799	320	-1	508	483	180
8	418	423	328		-1,	0, L		1	3458	3262	113
9	750	804	229	1	4048	4038	53	2	489	442	165
10	1011	1039	193	3	2602	2503	65	3	4182	4015	99
12	1036	1055	203	4	1679	1627	76	4	415	377	165
	-2,	6, L		5	1060	1065	94	5	3532	3455	104
-11	746	780	189	6	1759	1722	91	7	875	858	125
-9	929	945	156	8	1836	1835	109	8	848	878	136

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-1,	2, L		8	999	1011	154	-6	1522	1574	171
9	428	421	204	9	521	528	227	-4	1445	1581	192
10	1195	1211	161	11	1168	1177	172	-2	731	772	234
11	527	562	205	12	524	522	276	4	705	700	285
12	578	587	202	13	763	750	216	5	345	425	522
13	334	302	339	14	378	375	393	6	867	880	246
14	520	539	259		-1,	5, L		7	922	903	246
15	504	483	287	-14	604	548	221	8	392	338	442
	-1,	3, L		-12	816	818	164	9	738	682	277
-14	555	548	223	-11	783	761	161	10	452	447	422
-13	1066	1048	157	-10	371	377	265		-1,	8, L	
-12	823	797	136	-9	1314	1315	146	-10	695	673	232
-11	1386	1405	139	-7	593	606	158	-8	864	867	193
-10	1027	996	138	-5	255	252	315	-6	379	375	377
-9	907	885	134	-4	392	389	221	-3	403	381	368
-8	1706	1696	107	-3	599	578	179	-2	396	367	428
-7	626	624	112	-2	455	481	257	-1	341	381	526
-6	2571	2585	94	-1	393	365	335	1	578	501	360
-5	1009	1021	108	0	733	646	235	3	2134	1818	238
-4	2543	2501	89	2	3043	2831	184	4	517	438	473
-3	433	430	139	4	3364	3177	169	5	2192	1947	240
-2	1072	1073	126	5	714	663	218	6	750	668	321
0	2240	2059	162	6	2260	2185	170	7	1273	1207	237
1	343	287	354	8	574	567	234	9	380	350	566
2	3270	3130	130	9	704	770	225		-1,	9, L	
4	2204	2195	124	11	898	901	208	-8	448	403	363
5	775	765	133	13	395	353	389	-7	479	487	355
6	943	964	146		-1,	6, L		-6	1011	1013	214
7	1296	1317	150	-13	425	424	304	-4	1583	1538	213
8	330	323	278	-12	1095	1052	153	-2	1462	1280	215
9	1572	1561	153	-11	574	562	211	0	382	295	543
10	709	732	173	-10	1214	1257	172	2	1128	972	252
11	709	696	179	-9	721	726	164	4	1225	1148	257
12	773	800	196	-8	674	684	165	6	515	609	509
13	367	366	350	-7	1481	1495	149		-1,	10, L	
14	319	317	415	-6	248	374	415	-3	959	957	269
15	683	710	244	-5	2236	2283	146	-2	468	402	455
	-1,	4, L		-4	400	380	276	-1	415	390	551
-15	286	211	408	-3	1953	2001	169		0,	0, L	
-14	587	608	217	-1	639	646	267	1	384	304	47
-12	782	807	149	1	1686	1437	223	2	4227	4207	52
-9	1169	1166	137	3	2004	1914	213	3	402	412	94
-8	859	864	121	5	1205	1198	189	4	994	959	82
-7	2130	2168	111	6	288	287	517	5	1178	1170	86
-6	771	771	111	7	548	516	280	6	523	566	131
-5	2385	2415	106	8	903	900	201	7	1842	1826	95
-3	1191	1242	130	9	350	360	459	8	464	500	155
-2	317	333	286	10	916	962	220	9	423	443	170
1	373	296	369	11	678	652	267	10	263	238	288
3	863	831	160	12	296	279	573	11	642	608	150
4	448	491	242		-1,	7, L		13	1314	1290	162
5	1346	1349	164	-11	419	465	309	14	638	634	219
6	1287	1264	165	-8	799	824	184	15	975	936	169
7	723	733	177	-7	469	484	252	16	501	480	281

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$.
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o^2	F_c	σ	L	KF_o^2	F_c	σ	L	KF_o^2	F_c	σ
	0, 1, L			12	990	1003	142	6	411	419	246
-16	878	825	184	13	494	525	229	7	861	867	152
-15	703	721	208	14	783	813	202	8	1185	1248	164
-14	1284	1280	173	15	915	952	198	10	1333	1363	183
-13	885	862	143	16	739	748	233	11	912	923	170
-12	627	645	160		0, 3, L			12	938	915	190
-11	1235	1229	138	-15	422	448	299	13	741	737	220
-10	548	549	156	-14	1195	1136	176		0, 5, L		
-9	2374	2364	106	-13	570	558	* 203	-12	268	284	390
-8	977	987	117	-12	1318	1265	147	-11	294	281	307
-7	2790	2779	88	-11	330	296	247	-10	648	662	159
-5	888	865	87	-10	533	501	155	-9	1219	1204	143
-4	1046	1010	73	-9	477	475	156	-7	1370	1392	131
-3	1112	1058	63	-8	483	452	135	-6	812	878	135
-2	668	662	68	-7	854	850	118	-5	657	681	134
-1	3403	3364	51	-6	1466	1437	95	-4	1161	1222	137
0	408	127	160	-5	860	837	105	-2	894	976	144
1	2394	2284	73	-4	726	701	103	-1	635	714	188
2	293	309	158	-3	1023	967	98	0	627	521	221
3	212	221	218	-2	599	596	110	1	2066	2043	178
4	864	790	101	-1	3729	3867	104	2	646	698	247
5	221	207	239	0	697	764	188	3	2161	2352	181
6	1038	982	108	1	6497	6992	160	4	910	962	187
7	1381	1361	110	2	1080	1047	156	5	863	954	187
8	366	363	187	3	2596	2538	121	6	268	313	471
9	1587	1565	123	5	794	774	128	7	619	682	225
10	828	813	129	6	997	985	135	8	633	634	226
11	1220	1194	149	7	340	287	249	9	1312	1338	196
12	802	794	146	8	1412	1438	146	10	1072	1057	171
	0, 2, L			9	221	256	425	11	1087	1125	186
-13	367	356	255	10	357	354	280	12	462	422	318
-12	300	255	282	12	621	654	205	13	653	701	267
-11	837	847	124	13	327	316	377	14	376	410	422
-10	1309	1291	126	14	1142	1141	168		0, 6, L		
-9	538	526	136	15	674	678	240	-13	1118	1087	172
-8	1615	1584	102		0, 4, L			-12	383	387	315
-7	843	812	114	-15	899	869	175	-11	1161	1142	163
-6	1131	1103	92	-14	458	471	269	-10	272	260	372
-5	1223	1181	82	-13	1239	1220	169	-9	508	476	195
-3	912	883	82	-12	659	650	167	-8	478	491	204
-2	664	666	99	-11	509	515	182	-7	415	401	225
-1	460	492	123	-10	1219	1174	138	-6	916	935	151
0	2314	2660	117	-9	255	310	298	-5	920	909	135
1	420	425	252	-8	2251	2267	111	-3	467	443	233
2	3042	2934	99	-7	448	416	155	-2	969	981	157
3	885	862	126	-6	2337	2351	102	-1	559	594	248
4	1026	1005	116	-5	248	272	233	0	2766	2953	180
5	543	523	128	-4	416	432	154	1	963	993	207
6	880	879	131	-3	812	824	127	2	3715	3779	191
7	446	453	166	-2	1136	1193	130	3	978	1002	216
8	2130	2120	120	0	2567	2837	151	4	1533	1530	221
9	1198	1157	145	1	501	524	285	6	478	510	320
10	1795	1784	140	2	1452	1654	184	7	696	719	230
11	564	571	188	5	864	817	149	9	826	869	213

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
0, 6, L				2	734	752	324	-9	1696	1698	112
13	490	579	372	1, 0, L				-8	1026	1016	117
0, 7, L				0	659	636	61	-7	230	245	220
-12	984	970	177	1	2616	2539	44	-6	1597	1593	88
-11	458	452	281	2	234	155	125	-5	1355	1325	83
-10	406	352	285	3	1381	1341	64	-4	1491	1440	75
-9	1031	1022	154	4	980	972	82	-3	1233	1190	73
-7	1742	1798	156	5	2838	2763	76	-1	223	162	180
-5	1473	1554	168	6	1700	1694	89	0	347	345	179
-4	569	555	222	7	2584	2575	92	1	568	691	219
-2	471	563	305	8	703	762	121	3	243	158	286
-1	865	905	207	9	1748	1741	113	4	207	274	314
1	1407	1490	218	10	894	943	133	5	1384	1326	106
2	497	455	353	11	1207	1224	152	6	855	813	123
3	620	638	317	12	1377	1393	150	7	2573	2529	110
6	575	605	331	13	980	983	150	8	1573	1598	124
8	448	468	414	14	842	881	177	9	1982	1967	126
9	879	883	238	15	622	608	213	10	1382	1385	145
11	875	911	263	1, 1, L				11	924	906	146
0, 8, L				-16	474	479	324	14	843	848	182
-9	446	428	296	-15	1114	1121	184	16	509	489	284
-8	852	866	193	-14	478	487	263	1, 3, L			
-6	766	838	214	-13	675	623	165	-15	545	569	252
-5	690	736	219	-12	463	470	209	-13	805	812	159
-4	268	208	479	-11	687	706	145	-12	484	500	200
-3	809	877	206	-10	988	930	133	-10	652	636	141
-1	553	641	305	-9	1424	1422	118	-9	1266	1262	124
0	343	478	519	-8	657	602	110	-8	264	242	234
2	1178	1240	204	-7	1009	1008	106	-7	1884	1844	98
3	601	673	338	-6	1226	1246	91	-5	1757	1757	87
4	1449	1403	213	-5	287	301	151	-4	1037	1028	93
5	887	823	271	-4	4620	4517	69	-3	1570	1571	83
6	410	516	509	-3	364	327	99	-2	2443	2444	81
8	391	472	560	-2	4667	4555	56	-1	307	235	196
9	584	497	373	-1	1058	1028	54	0	3293	3453	110
10	747	766	312	0	3422	3412	57	1	853	885	171
0, 9, L				1	420	543	226	2	1652	1759	160
-8	363	347	404	2	407	389	130	3	374	358	266
-7	400	355	366	3	467	437	113	4	1045	994	145
-5	725	708	247	4	222	155	222	5	1068	1033	140
-4	472	422	362	5	650	623	104	6	2097	2048	122
-3	492	484	363	8	650	629	117	7	1644	1596	131
-1	695	673	299	9	1168	1168	130	8	1900	1880	133
0	442	513	478	11	1430	1427	142	9	731	736	154
1	1764	1794	240	12	699	681	161	10	1367	1352	158
2	852	851	288	13	393	383	256	11	634	623	186
3	2007	1990	240	14	763	771	183	12	1045	1085	159
4	876	815	301	16	429	371	302	13	1044	1052	164
5	962	903	279	1, 2, L				14	923	941	188
8	500	462	450	-15	406	429	311	15	518	576	296
0, 10, L				-14	332	320	337	1, 4, L			
-4	854	873	260	-13	843	816	155	-15	271	358	486
-3	479	500	412	-12	280	302	314	-14	1151	1107	174
0	586	652	387	-11	1423	1410	135	-13	408	379	269

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{12}$.
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	1, 4, L			2	1072	1067	191	2	1569	1554	220
-12	590	582	191	3	1035	1043	206	3	851	875	290
-11	601	583	161	4	426	281	394	4	526	444	423
-10	588	565	155	5	700	716	258	6	516	507	460
-9	1164	1113	132	6	913	941	209	7	708	686	357
-8	1040	1045	132	7	1159	1222	180	8	611	678	409
-7	624	599	121	8	1151	1200	180	9	789	834	333
-6	706	716	108	9	982	1045	199		1, 10, L		
-5	1090	1119	110	10	447	469	339	-5	395	334	421
-4	624	637	107	11	798	818	232	-3	557	566	350
-3	3421	3429	95	12	417	431	405	-1	1287	1327	211
-2	738	763	114	13	784	773	246	0	646	670	336
-1	3102	3274	112		1, 7, L			1	1175	1210	228
0	953	1014	135	-11	542	481	236	2	817	751	276
1	2149	2384	156	-10	549	560	226	3	598	588	377
2	654	686	216	-9	398	365	283		2, 0, L		
3	346	297	328	-8	1005	1009	143	0	274	243	139
4	380	358	281	-7	584	582	188	1	931	895	91
6	641	676	176	-6	483	484	223	2	2713	2671	60
9	512	465	219	-5	344	346	290	3	1830	1785	68
10	1150	1213	176	-4	865	859	156	4	4564	4473	74
12	1131	1132	174	-3	453	496	266	5	2707	2682	79
13	573	657	263	-2	2012	2190	172	6	3454	3409	86
15	501	521	315	-1	685	760	217	7	1523	1544	99
	1, 5, L			0	2008	2025	189	8	1604	1628	106
-13	283	310	406	1	885	910	213	9	391	392	178
-12	776	770	169	2	1159	1231	195	10	677	698	137
-10	1304	1298	147	3	522	541	349	11	1377	1377	138
-8	1308	1293	136	7	361	363	465	13	780	795	148
-7	980	974	144	11	839	908	258	16	657	624	216
-5	1213	1207	125		1, 8, L				2, 1, L		
-4	1215	1282	128	-11	569	583	257	-12	994	1007	161
-3	714	748	127	-9	919	952	181	-10	1960	1958	127
-2	1026	1085	148	-8	418	391	296	-8	2165	2185	108
2	584	580	259	-7	705	750	205	-7	1321	1291	107
6	1321	1366	178	-6	825	866	182	-6	1252	1276	98
7	1101	1088	167	-4	694	797	210	-5	2871	2851	82
8	1953	1963	172	-3	781	818	197	-4	700	690	104
9	1622	1642	183	-1	479	507	326	-3	4702	4570	69
10	1324	1339	184	3	393	391	447	-2	829	805	78
11	1069	1084	170	4	372	258	472	-1	1344	1294	60
12	598	600	268	7	909	938	282	0	192	159	171
	1, 6, L			8	894	916	279	1	1441	1470	102
-12	594	606	216	9	1099	1116	248	2	1191	1136	122
-11	480	442	237	10	1205	1250	244	3	2824	2651	83
-9	378	377	238	11	675	669	350	4	2611	2503	84
-8	1207	1207	148		1, 9, L			5	1712	1628	92
-6	1686	1711	135	-7	834	835	210	7	1415	1384	108
-4	1481	1545	141	-5	1097	1112	192	8	847	833	126
-3	825	844	142	-3	849	945	244	9	1020	969	134
-2	1054	1136	165	-2	522	512	326	10	1178	1167	141
-1	2082	2297	161	-1	755	740	252	11	1300	1273	143
0	724	711	207	0	1384	1470	227	12	246	283	374
1	2579	2711	179	1	790	793	282	13	864	831	149

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	2,	1, L		12	1053	1032	160	-9	1252	1213	154
14	455	499	270	13	313	322	369	-8	237	287	363
15	300	286	409	14	534	579	250	-7	1550	1528	137
16	819	804	192		2,	4, L		-6	935	955	139
	2,	2, L		-11	1109	1033	156	-5	880	923	136
-15	315	368	394	-10	261	279	311	-2	982	1059	135
-14	370	367	312	-9	1846	1817	123	-1	449	364	233
-13	330	328	320	-7	1908	1875	109	0	1140	1244	176
-12	509	515	197	-6	863	1027	137	3	1044	1063	192
-11	596	591	160	-5	1204	1205	108	4	1675	1846	214
-10	585	528	146	-4	2621	2601	94	5	1799	1891	204
-9	241	217	280	-3	1026	1019	110	6	2183	2202	196
-8	1146	1117	119	-2	3376	3412	95	7	2128	2121	193
-7	286	290	195	-1	1037	1073	124	8	1371	1343	194
-6	2564	2585	89	0	777	793	133	9	1012	1033	190
-5	232	281	197	2	893	941	178	10	837	788	204
-4	2691	2650	75	3	1109	1187	158	12	484	525	359
-3	1821	1732	72	4	1913	1909	160	13	685	642	268
-2	1092	1049	76	5	1854	1856	153		2,	7, L	
-1	1246	1213	75	6	1317	1283	161	-10	1018	985	155
0	366	380	143	7	418	370	246	-9	424	436	273
4	1686	1621	108	8	1028	1014	171	-8	1454	1460	159
5	358	319	189	9	584	565	203	-7	328	350	294
6	1306	1258	116	10	944	948	158	-6	1313	1340	162
7	1948	1932	114	11	850	871	171	-5	809	824	154
8	570	529	144	12	1130	1143	172	-4	1031	1051	158
9	1135	1149	146	14	716	678	224	-3	1998	2088	152
10	497	500	195	15	596	593	272	-2	1090	1158	181
12	791	840	158		2,	5, L		-1	1848	2043	173
13	959	946	152	-14	259	334	487	0	1069	1086	170
14	547	522	237	-13	384	377	306	1	378	409	386
15	891	893	186	-12	249	207	416	3	650	643	282
16	474	454	303	-11	571	555	193	4	816	856	253
	2,	3, L		-10	361	353	247	5	1029	1073	221
-13	642	627	189	-9	477	474	189	6	960	1048	226
-12	433	419	222	-8	221	186	340	7	583	603	315
-11	492	487	186	-7	1008	973	139	9	505	516	343
-10	1351	1310	135	-5	1997	2004	112	10	445	426	397
-8	1998	1939	108	-4	665	632	121	11	694	703	287
-7	1003	970	116	-3	2012	2033	112	12	533	468	353
-6	1274	1231	102	-2	1509	1547	123	13	779	776	285
-3	1150	1117	89	-1	691	688	140		2,	8, L	
-2	1108	1085	91	0	811	875	152	-10	464	465	273
-1	1313	1356	94	5	1095	1129	166	-8	346	300	334
2	1019	1127	163	6	814	850	190	-6	719	704	179
3	2358	2381	140	7	955	929	181	-4	1290	1311	184
4	1979	1950	130	8	1749	1743	178	-3	602	624	232
5	3629	3442	121	10	932	929	180	-2	1051	1114	170
6	2718	2598	122	11	409	411	324	-1	879	1036	200
7	2387	2314	126	13	378	495	412	1	500	513	315
8	1428	1437	144	14	853	868	233	6	655	638	310
9	1190	1189	158		2,	6, L		7	1075	806	275
10	297	326	317	-12	510	498	246	8	543	518	399
11	583	612	198	-11	546	515	214	9	1210	1196	235

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{12}$.
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	2, 8, L			-9	1019	1011	146	-14	311	295	383
11	612	615	377	-7	231	173	261	-11	437	411	213
	2, 9, L			-6	1432	1394	104	-10	652	637	151
-8	926	906	190	-5	873	893	115	-9	558	551	154
-7	393	366	341	-4	1965	1939	85	-8	1241	1215	124
-6	936	940	195	-3	989	962	91	-7	208	174	295
-5	623	646	242	-2	237	232	175	-6	933	950	135
-4	436	548	355	-1	1371	1221	74	-5	1384	1316	98
-1	614	713	276	0	2274	2220	66	-3	1609	1601	87
1	757	778	256	1	2744	2691	75	-2	760	747	106
4	790	722	285	2	3987	4573	159	-1	835	827	107
5	1087	1073	250	3	3854	3673	104	1	1096	1119	141
6	1358	1369	240	4	3288	3158	94	2	603	714	237
7	1150	1136	267	5	2045	2018	97	3	2310	2468	165
8	1510	1453	234	6	1200	1189	112	4	2286	2172	143
9	675	639	371	7	240	290	266	5	2009	1903	135
	2, 10, L			8	560	595	143	6	1251	1228	145
-7	869	875	233	9	1044	1031	141	7	1083	1115	157
-5	806	815	246	10	484	486	183	9	736	706	159
-4	538	495	320	11	441	420	209	10	636	623	187
-3	733	800	279	13	493	487	208	11	1716	1716	159
-2	1194	1251	208	14	377	410	309	12	543	576	222
-1	877	922	254	15	668	652	208	13	1192	1159	173
0	1009	1045	237	16	867	834	192	14	588	591	242
1	802	790	280		3, 2, L			16	864	878	215
4	503	469	445	-14	1025	1027	166		3, 4, L		
5	614	609	377	-12	1641	1615	150	-14	634	577	212
6	547	595	434	-10	1168	1174	145	-12	1512	1447	161
7	559	619	455	-9	675	656	136	-11	487	467	212
	2, 11, L			-8	685	707	128	-10	1548	1475	140
-1	494	557	414	-7	2290	2256	104	-9	366	347	223
0	539	650	393	-6	980	968	113	-8	1026	950	137
	3, 0, L			-5	2326	2328	89	-7	337	349	203
0	745	726	102	-4	1469	1470	87	-5	1513	1496	107
1	636	622	107	-3	764	735	99	-4	398	395	147
2	2245	2219	74	-2	1133	1120	83	-3	1760	1781	99
3	2632	2556	74	-1	1449	1408	77	-2	482	467	132
4	1859	1840	81	0	287	263	168	0	976	1001	141
5	1581	1588	88	1	1697	1691	101	1	1758	1833	140
6	1170	1223	102	2	286	224	483	2	2419	2679	158
7	189	167	294	3	1476	1417	157	3	2941	3095	175
8	605	594	124	4	357	322	244	4	3104	3182	166
9	979	976	137	5	485	499	164	5	1997	1953	163
10	1886	1918	122	6	489	494	155	6	1619	1543	162
11	978	981	136	7	315	287	230	7	743	726	178
12	1362	1387	147	8	553	542	156	9	492	500	239
13	829	832	153	9	1001	1004	145	10	796	811	177
14	349	303	306	10	556	576	185	11	509	460	234
15	1011	1017	164	11	1020	998	147	14	478	518	315
16	363	348	355	12	1464	1466	157	15	510	463	307
	3, 1, L			13	443	458	256		3, 5, L		
-13	1420	1351	166	14	1165	1139	174	-13	954	937	170
-11	1626	1589	147	16	449	413	308	-12	411	434	294
-10	382	366	224		3, 3, L			-11	1390	1323	157

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	3, 5, L			-4	1249	1290	162	4	1482	1476	220
-9	888	885	136	-2	1273	1326	172	5	799	812	313
-8	639	615	145	1	807	872	203	6	1414	1414	232
-7	801	797	123	2	1295	1370	208		3, 11, L		
-6	1878	1857	119	3	2081	2148	203	-2	409	477	478
-5	1162	1156	126	4	1620	1694	229	-1	744	736	278
-4	1729	1777	115	5	2100	2183	221	2	662	635	327
-3	1263	1305	123	6	934	972	226		4, 0, L		
-2	323	352	219	7	882	915	246	1	379	340	155
-1	778	773	123	8	375	388	466	2	380	360	182
0	1188	1274	150	10	403	384	445	3	515	516	103
1	398	426	269	11	441	455	418	4	216	206	231
2	1270	1453	179		3, 8, L			6	1031	1082	118
3	509	572	302	-10	897	902	188	7	1604	1611	111
4	842	878	201	-8	621	583	211	8	1584	1597	118
5	367	337	387	-7	504	540	252	9	2336	2368	117
6	330	315	416	-6	735	732	177	10	889	889	133
7	633	642	234	-5	1228	1260	181	11	1872	1888	133
9	389	379	330	-4	1025	1075	151	12	620	629	174
10	908	904	183	-3	931	1006	162	13	588	586	186
11	728	724	214	-2	1026	1076	162	14	491	491	250
12	612	623	262	0	432	498	340	15	391	415	333
13	1388	1377	199	1	838	931	210	16	554	566	260
15	935	963	229	2	297	357	522		4, 1, L		
	3, 6, L			3	720	761	265	-14	546	570	255
-13	321	297	394	4	412	457	434	-13	282	322	433
-10	373	345	285	8	553	538	370	-12	708	749	191
-9	595	574	175	9	392	346	509	-11	600	576	192
-8	390	380	229	11	585	584	395	-8	805	816	125
-7	991	965	146	12	645	629	367	-7	564	554	149
-6	363	337	227		3, 9, L			-6	1898	1905	110
-5	679	673	143	-9	358	363	400	-5	1271	1264	112
-4	1138	1176	146	-8	483	476	292	-4	1646	1637	98
-3	317	323	268	-6	649	622	231	-3	235	215	210
-2	1184	1246	153	-5	312	325	438	-2	2065	2020	85
-1	840	898	149	-3	735	767	227	-1	1522	1508	91
0	762	818	170	-2	360	419	380	0	2116	2096	78
2	1049	1146	173	-1	639	691	254	1	2857	2790	78
3	745	746	234	0	725	699	235	2	2674	2688	104
4	2073	2153	203	1	497	474	319	3	1796	1731	162
5	1478	1519	206	3	746	787	268	4	1064	1059	134
6	1915	1882	209	4	461	476	435	6	1296	1350	122
7	704	739	255	5	1437	1458	210	7	846	811	117
8	890	870	207	6	764	738	314	8	2544	2558	117
10	694	690	243	7	1411	1340	229	9	877	873	132
12	1262	1271	196	9	498	530	500	10	1268	1276	147
14	726	774	286		3, 10, L			11	1074	1068	153
	3, 7, L			-7	353	310	428	13	1161	1174	161
-11	1261	1210	178	-6	593	591	287	14	280	280	412
-10	656	597	202	-4	283	312	547	15	1071	1040	162
-9	1266	1231	169	-3	817	827	245		4, 2, L		
-8	407	363	247	-1	764	766	255	-13	1412	1376	176
-7	905	875	142	2	640	654	327	-12	277	286	404
-5	365	384	270	3	781	785	303	-11	1051	1031	149

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	4,	2, L		-6	961	914	136	3	453	510	353
-10	346	291	245	-5	1700	1656	110	4	380	380	457
-8	1009	998	141	-4	1336	1340	111	5	644	679	291
-6	1705	1666	109	-3	1340	1304	109	8	570	588	311
-5	378	362	156	-2	254	253	226	9	1598	1564	216
-3	1086	1069	100	-1	1999	1998	105	10	557	531	313
-2	2580	2552	82	0	1378	1353	121	11	1986	1926	212
-1	2386	2368	81	1	2094	2158	129	13	1276	1278	214
0	2474	2444	83	2	2006	2149	157		4,	7, L	
1	2814	2791	93	3	2296	2518	182	-11	497	480	273
2	1592	1604	137	4	965	982	197	-10	601	565	219
3	1149	1211	177	5	877	875	193	-9	575	563	226
4	511	532	229	7	1248	1247	174	-6	643	612	168
6	224	255	357	8	557	499	233	-5	1044	1034	145
7	488	482	186	9	2113	2074	162	-4	1206	1192	159
8	662	627	147	10	545	534	237	-3	1258	1280	164
9	447	461	211	11	1058	1050	161	-2	973	986	147
11	1153	1139	168	12	746	744	196	0	1521	1669	178
12	374	414	289	14	930	937	199	1	957	1051	183
13	948	962	158		4,	5, L		2	1862	2073	200
14	624	588	215	-13	468	441	282	3	1242	1298	186
	4,	3, L		-12	1299	1246	177	4	2026	2082	216
-14	770	728	196	-11	386	378	288	5	458	400	403
-12	502	536	235	-10	910	865	148	6	520	588	380
-11	510	490	211	-8	243	264	337	8	944	950	234
-10	230	284	387	-7	1076	1041	145	9	360	217	483
-9	898	863	126	-5	1393	1382	127	10	1340	1334	211
-8	647	656	141	-2	1028	1026	137	12	672	626	308
-7	714	687	123	-1	1784	1803	124	13	431	435	466
-6	1692	1646	111	0	2065	2154	132		4,	8, L	
-5	352	338	177	1	1552	1606	156	-10	486	431	280
-4	1498	1500	100	2	1992	2219	171	-9	625	624	233
-3	1602	1601	95	3	969	1011	191	-7	292	326	421
-2	488	471	116	4	1059	1084	202	-6	885	881	167
-1	1244	1164	101	8	454	399	308	-5	276	298	391
1	623	640	133	9	416	409	329	-4	782	815	176
2	276	288	384	10	555	511	259	-2	340	345	359
3	485	453	315	12	1060	1056	187	-1	683	728	218
4	805	776	192	13	478	512	336	0	1047	1138	178
7	888	862	146	14	720	719	258	1	1344	1496	201
8	1769	1734	149	15	558	529	311	2	816	887	227
9	1072	1085	166		4,	6, L		3	1349	1396	193
10	2310	2266	150	-11	463	430	263	4	375	386	452
11	409	474	288	-10	453	419	245	5	580	670	341
12	1682	1687	170	-9	324	320	312	11	472	473	479
13	449	382	286	-8	656	680	163		4,	9, L	
14	479	447	291	-7	718	715	149	-8	321	280	408
15	422	440	341	-6	459	435	197	-7	411	432	328
	4,	4, L		-5	1333	1358	141	-6	628	650	241
-13	775	719	188	-4	596	547	160	-4	908	927	189
-12	444	440	262	-3	970	994	150	-3	616	622	254
-11	768	759	170	-2	1443	1488	140	-2	552	547	278
-10	682	637	153	0	758	839	172	-1	919	1011	193
-7	772	749	124	2	352	461	384	1	392	427	388

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	4, 9, L			9	2377	2448	132		5, 4, L		
4	465	505	404	10	648	622	165	-11	323	330	344
6	424	447	502	11	561	572	192	-10	654	625	180
10	1096	1071	276	12	682	717	174	-8	1194	1141	148
	4, 10, L			13	341	341	334	-7	898	879	139
-5	289	342	530	14	757	758	193	-6	1246	1227	135
-4	737	795	247	15	309	351	415	-5	1536	1526	122
-3	742	589	250	16	327	267	391	-4	868	848	129
-2	850	883	234		5, 2, L			-3	543	521	140
-1	556	551	317	-10	711	701	176	-2	579	621	135
1	946	1059	241	-9	983	962	145	-1	606	609	130
2	567	554	338	-8	1125	1143	154	0	735	758	126
3	1471	1466	216	-7	1146	1162	139	1	1386	1451	141
4	585	543	367	-6	1667	1655	119	2	597	583	194
5	1346	1354	230	-5	307	290	222	3	616	675	261
	4, 11, L			-4	1804	1802	105	4	563	607	334
0	588	521	333	-3	1427	1415	105	5	391	401	427
1	631	660	335	-2	2440	2481	93	6	2392	2301	181
2	985	1015	245	-1	1969	1971	93	7	760	702	217
3	438	444	477	0	2453	2462	91	8	3178	3041	165
4	904	897	277	1	919	924	119	9	398	394	336
	5, 0, L			2	1114	1083	135	10	1920	1907	174
0	1365	1414	115	3	570	557	271	11	325	291	389
1	596	589	126	4	1042	1034	197	12	407	405	325
2	517	543	153	5	1009	1011	150	13	589	614	259
3	506	433	427	6	1652	1588	146	15	688	649	257
4	416	455	148	7	305	322	326		5, 5, L		
6	865	872	128	8	996	983	161	-10	305	298	341
7	854	852	121	9	584	543	190	-9	618	569	187
8	1826	1896	122	11	752	759	165	-8	1033	1010	154
10	1157	1188	149	13	871	849	176	-7	874	846	134
11	351	363	251	14	373	334	344	-6	1101	1136	150
14	1012	985	159	15	458	470	317	-5	1365	1333	134
	5, 1, L			16	666	656	245	-4	304	277	243
-12	378	387	325		5, 3, L			-3	1595	1597	124
-11	522	492	223	-13	455	455	289	-2	1146	1120	137
-9	1079	1048	151	-10	732	706	169	-1	2296	2330	122
-8	521	533	185	-8	567	567	164	0	1309	1306	144
-7	1305	1325	139	-6	624	593	137	1	2065	2137	146
-6	1374	1361	127	-5	367	361	184	2	453	455	273
-5	947	940	139	-4	1839	1815	106	3	638	713	260
-4	701	688	114	-3	1296	1304	111	4	503	532	356
-3	401	388	160	-2	1330	1309	105	5	988	1013	226
-2	468	475	133	-1	1412	1443	105	6	626	658	305
-1	414	408	138	1	1188	1199	126	7	1421	1374	205
0	1607	1635	94	2	881	867	136	9	752	762	231
1	242	252	212	4	518	542	347	12	668	684	258
2	845	891	122	5	661	627	239	14	743	733	254
3	725	697	232	7	1340	1309	180	15	342	394	515
4	517	514	222	8	578	564	211		5, 6, L		
5	2404	2360	124	9	2019	1985	156	-10	271	212	396
6	1170	1170	146	11	1070	1077	169	-9	608	590	202
7	3545	3529	121	12	305	316	366	-7	423	388	214
8	990	975	151	15	891	860	201	-5	542	511	174

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{12}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	5, 6, L			0	326	414	482	10	301	298	326
-4	442	462	207	1	759	843	231	12	1280	1279	169
-3	1298	1329	148	3	348	319	494	13	328	304	373
-2	1222	1188	150	4	543	559	361	14	1066	1100	171
-1	866	878	142	6	332	305	646		6, 2, L		
0	1311	1331	164	9	982	945	301	-12	339	313	372
2	791	834	204		5, 10, L			-11	873	873	173
3	745	812	239	-6	585	567	282	-10	1058	1056	174
5	372	504	521	-5	794	787	223	-9	1310	1264	162
8	1341	1303	205	-4	457	442	349	-8	1167	1163	162
10	1757	1670	222	-3	904	947	221	-7	864	872	141
11	325	382	556	-1	345	345	466	-6	498	468	174
12	893	864	238	0	326	437	516	-5	820	805	129
	5, 7, L			2	611	660	331	-4	739	729	122
-9	529	554	239	3	493	542	413	-3	844	814	132
-8	268	301	415	4	500	533	435	-2	1702	1686	110
-7	935	897	156	6	366	415	606	-1	572	583	128
-6	1011	964	154	8	1384	1328	240	0	888	885	127
-5	855	847	147		5, 11, L			1	775	767	113
-4	1404	1391	156	-1	890	901	246	2	393	358	198
-3	673	601	170	0	386	349	475	3	2842	2810	146
-2	553	495	196	1	1225	1261	223	4	1007	967	265
-1	568	613	212	2	324	203	574	5	3835	3571	181
0	530	535	239	3	983	948	262	6	568	498	235
1	795	850	195		6, 0, L			7	1761	1679	157
2	937	1037	200	0	883	933	143	9	413	405	264
3	672	708	263	1	348	367	234	10	471	515	243
4	345	390	513	2	930	946	139	12	671	628	203
5	536	557	368	3	809	827	148		6, 3, L		
7	1970	1972	231	4	671	688	135	-12	449	414	284
9	2263	2186	231	5	341	319	216	-11	729	740	195
11	1276	1217	220	10	484	542	206	-10	814	819	175
13	318	234	582	11	649	698	179	-9	1015	1014	164
	5, 8, L			12	355	358	317	-8	761	758	147
-9	359	335	358	13	1229	1231	178	-7	278	234	288
-8	480	429	268	15	949	986	181	-6	1032	1008	134
-7	981	944	167		6, 1, L			-5	940	927	126
-6	575	561	228	-10	410	407	286	-4	1639	1679	119
-5	984	977	161	-9	523	493	219	-2	1993	1934	108
-4	879	891	164	-8	291	302	317	-1	294	304	227
-2	1260	1312	178	-7	886	903	142	0	857	845	130
-1	738	751	200	-6	329	358	256	1	1252	1254	128
0	1741	1899	191	-5	784	799	131	2	441	455	220
1	600	623	258	-4	808	798	123	3	998	950	172
2	1404	1544	203	-3	578	562	145	4	765	769	298
5	442	447	419	-2	1008	1009	132	5	675	593	326
6	762	769	284	-1	1371	1367	115	8	515	479	249
8	865	837	288	0	706	702	114	11	446	450	273
	5, 9, L			1	895	917	124	12	722	718	211
-8	348	357	403	2	876	904	129	14	1156	1156	180
-4	462	497	306	4	2670	2391	221	15	352	306	434
-3	376	340	365	5	581	536	211		6, 4, L		
-2	805	874	227	6	2237	2210	137	-9	525	480	209
-1	807	843	224	8	1156	1208	158	-8	730	692	150

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	6,	4, L		3	566	553	279	2	777	868	276
-7	333	314	258	4	689	670	281	4	492	519	413
-6	1082	1052	148	5	834	794	270	5	531	513	406
-5	372	388	217	9	551	538	338	7	1176	1181	267
-4	622	588	139	12	422	372	427		7,	0, L	
-3	712	662	126	13	670	649	310	0	1708	1785	137
-2	909	882	134		6,	7, L		1	645	677	166
-1	679	642	130	-8	443	379	270	2	1960	2067	139
0	1465	1454	129	-7	697	662	190	4	1552	1487	221
1	456	451	204	-6	250	162	400	6	309	280	263
2	852	871	159	-5	929	917	147	9	215	154	423
3	1015	1014	185	-4	414	414	247	10	378	400	279
5	2312	2321	221	-3	335	300	297	12	744	815	193
7	2197	2004	194	-2	550	512	203	13	443	466	303
9	1059	1038	175	-1	878	883	162	14	566	581	251
11	290	300	444	0	341	278	342		7,	1, L	
13	1031	1044	200	1	1248	1323	167	-9	428	367	278
14	535	377	295	3	793	820	228	-8	421	453	270
15	863	840	231	4	818	848	251	-7	759	828	173
	6,	5, L		5	306	276	610	-5	902	938	133
-11	457	498	294	6	1801	1873	247	-4	639	682	158
-10	835	810	177	8	1754	1704	251	-3	609	645	157
-9	1324	1254	169	10	851	815	290	-2	1386	1438	135
-8	1158	1073	165		6,	8, L		0	1147	1174	135
-7	1208	1137	156	-8	1251	1200	184	3	308	289	249
-6	749	707	141	-7	764	738	203	4	572	538	257
-5	418	399	210	-6	1088	1036	171	5	810	808	229
-4	924	899	135	-5	534	508	245	9	961	1016	148
-3	648	634	146	-4	432	371	279	10	245	147	433
-2	1049	1045	151	-3	783	799	193	11	1996	1986	158
-1	1330	1319	141	-2	469	458	265	12	487	481	275
0	677	658	158	-1	1019	1001	161	13	1544	1550	179
1	569	516	195	0	780	796	206		7,	2, L	
2	779	734	187	1	544	560	275	-10	454	454	275
4	2364	2474	209	3	605	639	294	-9	829	826	176
5	382	399	531	5	1787	1805	235	-6	398	373	229
6	2739	2799	219	7	1764	1771	255	-5	822	860	137
8	1354	1217	194	9	722	701	344	-4	446	433	195
10	342	287	436		6,	9, L		-3	1533	1558	129
11	407	426	394	-7	636	606	247	-2	564	587	155
13	530	521	328	-6	302	305	446	-1	1318	1325	127
	6,	6, L		-4	726	732	221	0	1373	1374	124
-10	790	778	191	-3	408	349	334	1	238	226	316
-9	479	552	296	-2	1151	1200	178	2	2161	2154	119
-8	935	900	159	0	946	966	204	3	387	341	270
-7	522	506	217	1	468	552	368	4	2802	2714	197
-5	950	911	132	3	715	776	289	6	1317	1309	196
-4	637	643	159	4	419	457	460	8	407	420	298
-3	1594	1583	141	6	465	462	472	10	1175	1222	182
-2	333	355	284	10	375	436	671	11	490	500	291
-1	1580	1548	150		6,	10, L		12	1010	1020	179
0	463	453	236	-4	662	646	264	14	522	470	287
1	480	465	254	-3	327	315	466		7,	3, L	
2	1019	1072	176	0	547	643	332	-10	980	969	162

Table B.2. Observed and Calculated Structure Factors for $Cs_4(CO)_{12}$.
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	7, 3, L				7, 6, L			6	551	568	426
-9	776	777	179	-9	971	939	172		8, 0, L		
-8	349	342	307	-8	675	622	197	1	1831	1917	149
-7	552	544	197	-7	321	304	350	3	887	930	156
-6	640	654	160	-6	549	522	208	4	293	348	404
-5	661	664	148	-5	556	547	202	5	299	379	542
-4	746	742	134	-4	724	703	157	7	855	921	142
-3	345	286	223	-3	534	503	186	9	653	667	195
-2	257	229	284	-2	316	281	301	12	426	412	304
-1	640	642	140	0	657	629	184		8, 1, L		
0	689	695	135	1	477	447	262	-7	255	280	443
1	2021	2062	124	2	1657	1698	190	-6	340	376	332
2	462	425	219	4	1526	1573	219	-5	271	305	391
3	2049	2012	159	6	862	865	278	-4	266	278	373
5	1231	1239	253	12	538	516	386	-3	739	796	161
11	554	577	276		7, 7, L			-2	934	975	138
13	881	872	205	-7	417	404	289	-1	427	448	215
14	729	674	241	-5	725	707	188	0	1096	1160	155
	7, 4, L			-3	571	563	203	1	502	483	184
-10	372	293	317	-2	1015	987	146	2	619	620	154
-8	423	426	265	0	1385	1370	183	3	845	882	125
-7	443	413	231	1	311	299	401	5	682	537	440
-6	852	867	139	2	789	847	223	7	297	295	398
-4	829	823	126	5	776	743	274	8	763	801	185
-3	1027	1032	150	7	900	860	294	9	329	321	376
-2	458	457	188	11	925	866	291	10	1302	1276	187
-1	1662	1642	127	12	577	514	407	11	604	621	247
1	1125	1149	160		7, 8, L			12	894	919	188
4	725	693	275	-7	768	710	197	13	453	464	319
5	372	344	580	-3	874	891	185		8, 2, L		
6	1045	982	254	-1	1277	1261	184	-9	306	311	393
10	1035	997	186	1	853	908	211	-7	610	538	205
11	404	380	395	2	848	917	221	-6	1197	1155	168
12	1786	1717	204	4	1612	1686	223	-5	452	465	245
13	661	630	281	6	1708	1703	240	-4	1576	1606	147
14	1271	1230	194	7	437	444	526	-3	297	272	293
	7, 5, L			8	548	540	450	-2	1072	1088	153
-10	844	807	183		7, 9, L			-1	339	342	248
-9	341	383	356	-6	262	231	507	1	978	1012	142
-8	882	854	164	-5	441	423	322	2	732	718	138
-4	958	936	152	-4	460	376	302	3	1009	1038	158
-2	1505	1475	140	-3	628	611	248	4	431	454	310
-1	395	364	233	-2	305	214	462	7	1386	1345	189
0	1205	1178	157	1	542	530	313	8	583	558	264
1	1217	1185	172	2	413	321	394	9	2000	1918	180
2	280	246	422	3	1082	1162	217	10	716	671	233
3	1979	1983	194	5	937	977	272	11	1608	1604	190
5	2281	2321	234	7	387	389	582	12	370	342	384
7	1018	946	246		7, 10, L			13	704	713	236
9	426	407	409	-2	326	294	481		8, 3, L		
10	383	323	462	-1	766	793	255	-7	973	977	155
11	1059	980	221	1	852	899	254	-5	1449	1447	155
12	526	493	355	2	358	264	472	-4	337	346	280
13	743	765	282	3	477	478	409	-3	1068	1030	153

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{16}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	8,	3, L		-1	876	883	182	10	414	481	308
-2	1044	1037	150	1	1674	1660	175		9,	1, L	
-1	234	230	347	2	287	285	460	-6	465	437	266
0	1845	1894	131	3	1624	1631	216	-5	389	411	312
2	2047	2068	139	4	545	503	339	-3	1006	1088	165
3	406	423	289	5	489	501	427	-1	966	1047	153
4	855	795	214	7	500	534	532	0	464	520	240
5	416	416	571	9	817	737	331	1	470	462	228
6	575	531	465	11	454	376	477	2	752	796	160
8	904	873	228		8,	7, L		3	368	378	273
10	557	516	305	-6	377	376	327	4	446	446	227
13	483	486	343	-4	282	271	410	5	583	570	250
	8,	4, L		-3	469	426	261	6	394	323	599
-8	319	263	350	-2	558	515	223		9,	2, L	
-7	421	368	263	-1	620	620	228	-7	567	624	243
-5	358	372	294	0	874	858	187	-5	689	716	195
-4	493	478	195	2	712	730	242	-4	295	341	375
-3	471	487	202	3	607	567	298	-3	327	300	306
-2	846	853	136	5	617	609	364	-2	408	352	251
-1	988	1022	149	9	421	411	575	-1	422	463	245
0	313	309	283	10	796	729	356	1	989	1037	142
1	942	968	150	11	706	665	398	2	330	281	263
2	648	607	190		8,	8, L		3	854	901	146
3	327	275	388	-6	518	500	279	4	259	200	423
4	854	806	232	-4	1283	1182	179	5	800	733	283
6	486	495	508	-2	1305	1314	188	6	1492	1215	298
8	502	390	380	0	796	797	220	7	1014	858	269
9	936	875	257	1	445	446	368	8	1748	1654	216
10	622	555	306	3	1031	1019	227	9	756	747	246
11	1281	1182	200	5	896	901	280	10	878	899	217
12	806	763	260	9	978	985	319	11	342	341	441
13	814	754	246	10	685	621	405		9,	3, L	
	8,	5, L			8,	9, L		-6	1262	1279	178
-7	479	467	248	-3	931	879	202	-4	819	849	169
-6	359	408	316	-1	573	506	281	-3	502	515	219
-5	1404	1350	160	0	714	719	261	-1	955	956	140
-4	236	187	417	2	1198	1273	213	0	264	315	346
-3	1660	1611	147	3	403	388	455	1	982	986	146
-1	982	964	140	4	993	1042	250	4	277	246	481
0	503	488	206	5	374	494	602	5	1406	1285	210
2	1175	1162	178	8	431	470	578	6	678	591	500
3	442	430	322		8,	10, L		7	2114	1851	283
4	1081	1056	203	0	406	438	441	8	928	815	284
8	1296	1209	249	1	625	608	322	9	1182	1078	219
9	705	630	348	3	457	396	412	10	468	398	380
10	1744	1567	248	4	408	387	502		9,	4, L	
11	893	753	266		9,	0, L		-7	777	768	189
12	1244	1200	216	0	704	802	201	-5	455	428	249
13	475	370	404	1	283	334	419	-4	400	421	280
	8,	6, L		4	1080	1142	172	-2	1181	1176	161
-6	976	916	162	6	1744	1818	157	0	945	971	150
-5	274	178	376	7	577	563	205	1	742	712	164
-4	1268	1221	173	8	1175	1261	169	3	865	889	179
-2	784	792	164	9	305	398	400	4	672	589	245

Table B.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	9, 4, L			-1	364	375	448		10, 5, L		
5	460	467	499	1	770	778	267	-4	421	383	294
6	651	588	459	3	601	591	342	-3	393	351	307
7	469	365	602		10, 0, L			-2	297	288	399
10	338	416	614	0	804	913	203	-1	992	961	162
	9, 5, L			1	290	331	461	0	354	290	330
-6	796	759	181	2	650	680	235	1	1008	983	164
-4	828	793	172	3	997	1064	182	2	700	624	219
-3	416	383	267	4	669	668	242	3	788	760	220
-2	271	290	387	5	1344	1400	208	5	451	393	487
0	487	451	238	6	927	695	547	6	546	485	515
2	888	909	187	7	835	937	178	8	804	702	413
3	454	418	332		10, 1, L				10, 6, L		
4	770	753	266	-2	595	620	226	-2	551	489	245
5	385	172	557	2	988	1016	156	-1	619	577	225
6	792	792	385	4	1000	1079	153	0	840	794	196
7	1121	1095	314	5	508	577	253	1	465	450	302
8	1067	977	320	6	715	669	394	2	812	764	220
9	1534	1358	239	7	556	470	359	4	852	801	268
10	873	773	307		10, 2, L			5	926	904	291
11	644	653	384	-3	512	459	236	6	897	891	337
	9, 6, L			-2	882	885	167	7	1141	1092	314
-5	1234	1155	168	0	953	1020	156	8	888	871	400
-3	765	725	184	1	422	434	269		10, 7, L		
-2	521	494	233	2	767	806	174	-2	660	644	228
0	964	936	168	3	257	256	416	0	506	456	295
2	823	859	225	4	363	330	327	4	914	903	271
6	1157	1111	270	5	421	381	366	5	430	384	522
7	718	698	417	7	934	743	376	6	720	697	377
8	1514	1418	263		10, 3, L			7	579	532	495
9	870	788	348	-3	543	560	234		10, 8, L		
10	760	711	390	-2	433	463	277	1	478	431	365
	9, 7, L			-1	973	1002	157	2	732	729	285
-4	270	271	461	0	377	429	295	3	670	669	331
-3	414	396	306	1	940	956	158	4	508	549	431
-1	1022	999	171	2	328	261	326		11, 2, L		
0	375	326	359	3	882	853	178	3	287	296	424
1	740	724	225	4	1105	1065	174		11, 3, L		
2	697	670	266	5	947	902	243	2	470	508	271
4	732	708	294	6	1607	1391	263	3	533	491	245
5	646	644	356	7	1058	913	396	4	332	290	408
7	542	540	469	8	941	833	344	5	823	787	235
	9, 8, L				10, 4, L				11, 4, L		
-3	717	668	225	-3	695	696	199	1	641	617	215
-2	491	394	294	-1	665	629	191	2	817	812	188
1	441	394	370	1	356	310	318	3	711	710	219
2	368	394	472	3	1107	1117	176	4	792	776	230
3	665	689	302	4	383	350	419	5	816	758	273
4	441	486	480	5	1011	962	257		11, 5, L		
5	629	579	350	6	615	608	491	1	827	808	198
7	799	731	330	7	437	474	746	3	437	486	363
8	899	828	333	8	423	396	766	4	334	245	497
	9, 9, L										

Table C.1. U_{ij} or U_{iso} Values (*100) for $Os_4(CO)_{14}$.

	U11(U)	U22	U33	U12	U13	U23
Os(1)	2.44(3)	2.23(4)	2.56(4)	-0.306(24)	0.090(23)	-0.03(3)
Os(2)	3.10(3)	2.52(4)	2.07(3)	0.24 (3)	-0.073(23)	0.52(3)
O(11)	7.7 (9)	4.4 (8)	3.5 (7)	-0.4 (7)	0.5 (6)	0.9 (6)
O(12)	5.7 (8)	8.4 (12)	3.0 (7)	-1.8 (7)	-2.1 (6)	0.4 (7)
O(13)	3.0 (6)	6.7 (10)	7.3 (10)	0.9 (6)	2.0 (6)	1.2 (8)
O(14)	9.0 (11)	3.7 (9)	6.5 (10)	-1.0 (7)	0.4 (8)	-0.3 (7)
O(21)	10.4 (11)	6.6 (11)	3.0 (7)	0.0 (9)	-2.5 (7)	0.3 (7)
O(22)	9.8 (11)	5.8 (10)	6.7 (10)	4.7 (9)	2.3 (9)	1.5 (8)
O(23)	8.1 (10)	7.3 (12)	5.5 (9)	-3.4 (9)	0.1 (8)	1.0 (8)
C(11)	3.3(4)					
C(12)	4.2(4)					
C(13)	3.0(3)					
C(14)	4.1(4)					
C(21)	4.0(4)					
C(22)	4.8(5)					
C(23)	4.1(4)					

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-15, 1, L			4	1014	1033	550	3	1473	1421	349
2	1227	1256	553	6	740	859	804	5	2865	2791	420
	-15, 3, L			8	1206	1247	584	6	1364	1322	407
1	936	860	673		-12, 6, L			7	1192	1131	467
2	2612	2484	366	1	1347	1262	447	9	1076	1118	597
3	790	929	860	4	1331	1337	501	10	1115	1129	596
	-14, 0, L			5	1827	1728	401		-10, 4, L		
4	1645	1575	423	6	2220	2144	385	1	1088	988	442
6	2576	2509	384	7	1580	1539	470	2	1779	1778	337
	-14, 2, L				-12, 8, L			4	1365	1401	402
1	2651	2561	409	2	1723	1735	453	5	2454	2389	352
3	2801	2699	404	3	847	785	742	6	934	775	528
4	656	600	865	5	1360	1381	536	7	1358	1408	456
5	676	897	953		-12, 10, L			9	1477	1483	462
6	1443	1326	489	1	825	877	892		-10, 6, L		
	-14, 4, L				-11, 1, L			1	3063	3128	452
1	2476	2402	374	1	3174	3144	437	2	2877	2852	435
2	1565	1568	455	2	529	453	845	3	1898	1933	358
3	2261	2189	386	3	1347	1272	386	4	1080	1057	491
4	1265	1296	553	5	1678	1652	376	5	725	730	717
	-13, 1, L			7	1926	1861	371	7	680	691	814
2	1703	1694	386	8	810	813	707	9	973	936	654
4	1313	1277	478		-11, 3, L				-10, 8, L		
5	2841	2802	405	2	1400	1298	386	1	2388	2451	390
7	2518	2459	402	4	692	620	674	2	1333	1296	490
	-13, 3, L			6	1333	1312	436	3	1799	1771	413
1	1165	1097	503	8	1029	1084	600	4	1135	1119	548
2	3344	3267	460	9	660	818	956	6	1985	1934	403
3	957	1062	632		-11, 5, L				-10, 10, L		
4	2058	1945	362	1	1820	1743	354	1	770	914	886
	-13, 5, L			2	2295	2274	317	4	1736	1767	470
1	1158	1050	523	3	577	592	829	5	881	818	755
2	1894	1907	402	4	630	548	774		-9, 1, L		
3	1527	1510	446	5	623	715	833	1	5772	5782	349
5	2098	2078	401	6	1797	1753	396	2	1156	1120	384
6	1358	1265	497	7	1236	1190	498	3	3503	3528	390
	-13, 7, L				-11, 7, L			4	2094	2119	307
4	1540	1526	477	1	1709	1714	424	5	621	504	675
	-12, 0, L			2	1952	1929	387	6	2131	2126	351
2	1628	1593	367	3	760	837	785	8	538	565	951
4	1888	1907	358	5	1478	1468	479	9	2626	2621	382
6	3527	3491	467	6	1559	1490	445	11	1211	1163	580
8	2017	1978	402	7	1250	1207	543		-9, 3, L		
	-12, 2, L				-11, 9, L			1	741	680	549
1	1507	1441	385	1	837	796	772	2	928	843	461
2	1561	1605	384	5	976	966	701	3	677	620	604
3	1870	1828	356		-10, 0, L			4	3998	3927	399
6	1915	1888	388	2	5210	5103	380	5	1776	1768	331
7	809	764	704	4	569	398	781	6	3922	3894	437
8	735	677	811	6	1562	1497	387	7	1155	1161	470
	-12, 4, L			10	1033	1021	651	8	817	834	625
1	1987	1905	338		-10, 2, L			10	915	928	667
2	772	771	653	1	593	626	724		-9, 5, L		
3	1774	1722	378	2	2530	2556	417	1	3663	3586	424

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-9,	5, L		1	3144	3192	443	8	616	687	804
2	2255	2231	317	2	3511	3539	439	9	2650	2656	393
3	3194	3109	429	3	1618	1638	361	10	1635	1651	428
4	1019	964	482	4	1496	1496	375	11	1861	1947	438
5	1736	1699	343	7	776	688	629		-7,	7, L	
6	1857	1864	351	8	2102	2080	349	1	2900	2941	393
7	1378	1401	428	9	2400	2509	388	2	1598	1594	370
9	1199	1224	527	10	2028	2133	416	3	522	600	931
10	1437	1413	489		-8,	8, L		4	517	438	931
	-9,	7, L		1	2393	2457	360	5	1253	1247	440
1	2910	2872	392	2	1257	1271	478	6	709	645	709
2	3683	3649	468	3	2187	2180	358	8	2519	2499	396
3	1432	1436	426	4	1649	1680	395	9	2065	2037	375
4	1724	1749	382	5	1566	1505	402	10	2426	2464	376
5	859	846	620	6	2335	2431	416		-7,	9, L	
8	1601	1602	434	7	2083	2050	374	1	2122	2265	384
9	1230	1345	560	9	1909	2030	424	3	796	636	678
	-9,	9, L			-8,	10, L		4	1002	1014	615
2	1157	1091	552	1	653	767	998	5	3113	3116	406
3	1398	1407	477	4	2849	2805	408	6	1064	1129	604
4	1490	1474	480	5	1114	1058	603	7	1853	1825	412
5	3219	3173	474	6	3245	3167	435	8	1094	1084	596
6	1340	1332	518		-8,	12, L			-7,	11, L	
7	1841	1850	432	1	2622	2746	427	1	1317	1299	575
	-9,	11, L		3	1775	1744	492	2	1235	1204	592
2	2268	2247	427	4	826	914	906	5	2211	2115	403
3	1489	1426	523		-7,	1, L		6	763	722	900
5	2191	2190	430	1	3935	3844	310	7	1538	1500	518
	-8,	0, L		3	919	919	365		-7,	13, L	
2	6530	6592	330	4	1762	1756	260	2	1557	1678	589
4	1774	1758	290	5	1398	1362	317		-6,	0, L	
8	3297	3243	446	6	2738	2766	398	2	847	858	345
10	3540	3529	527	7	1491	1482	366	4	3325	3317	334
	-8,	2, L		8	1160	1168	473	6	1610	1665	307
2	2965	2824	366	9	4219	4104	459	8	3130	3099	426
3	2813	2778	380	11	2037	2068	413	10	3917	4000	514
5	4524	4548	382	12	1075	1051	663	12	898	879	780
6	1018	1006	474		-7,	3, L			-6,	2, L	
7	3060	3116	414	1	2196	2174	330	1	5663	5543	284
8	1318	1321	424	2	1570	1579	269	2	1461	1370	240
9	501	514	1001	4	2983	2979	373	3	1163	1152	287
10	1773	1763	412	5	1811	1759	310	4	1867	1869	261
	-8,	4, L		6	4384	4467	391	5	2210	2261	336
1	1943	1926	285	7	762	709	620	6	1921	1951	308
2	2034	1984	303	8	1107	1159	490	7	1248	1242	399
3	1675	1631	310	12	1751	1754	475	8	1679	1609	351
4	1839	1847	302		-7,	5, L		10	1815	1879	393
5	4303	4336	405	1	1267	1289	359	11	907	904	678
6	2210	2224	305	2	2682	2677	377		-6,	4, L	
7	2412	2376	335	3	1552	1571	334	1	5253	5185	325
8	1920	1927	351	4	2005	1988	291	2	1725	1708	255
9	735	745	737	5	894	932	525	3	739	728	467
10	1178	1150	542	6	2453	2442	341	5	3646	3556	363
	-8,	6, L		7	1906	1979	338	6	910	891	477

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ
	-6, 4, L		
7	1289	1323	403
8	681	644	696
9	1255	1329	460
10	1044	1094	577
12	1149	1120	628
	-6, 6, L		
1	1881	1863	297
3	1059	990	435
4	1720	1718	331
5	2182	2236	332
6	1044	978	466
8	1784	1823	365
9	2355	2394	338
10	2184	2228	357
11	1073	1077	602
	-6, 8, L		
1	1104	1087	534
2	2013	2032	355
3	994	1001	562
4	1584	1543	406
6	1635	1617	385
7	779	892	707
8	843	897	672
9	1558	1602	444
10	1101	1044	578
	-6, 10, L		
1	1241	1234	569
2	2412	2538	395
4	863	943	748
5	1012	1051	653
6	1670	1732	464
	-6, 12, L		
1	895	918	830
5	1755	1777	510
6	752	689	970
	-6, 14, L		
2	1039	1143	871
	-5, 1, L		
2	3582	3604	273
3	3333	3308	289
5	5168	5250	324
7	1275	1228	385
8	1092	1083	476
9	2555	2627	361
11	915	1026	742
12	857	792	794
	-5, 3, L		
1	3791	3679	288
2	6259	6271	283
3	586	496	484
5	949	942	376
6	753	748	506
8	1932	1900	318
9	1134	1179	501
10	1229	1246	493
12	1470	1507	519
	-5, 5, L		
1	3272	3222	360
2	1992	1938	286
3	3281	3287	360
4	2668	2653	360
5	2663	2678	371
6	2019	2041	305
7	1199	1210	430
8	1710	1760	348
9	941	861	541
10	1590	1684	423
11	701	815	876
12	878	978	783
	-5, 7, L		
1	2185	2197	333
2	2140	2178	341
3	1913	1844	323
4	3118	3097	447
5	3186	3210	442
6	2096	2167	325
8	688	598	695
9	1756	1863	400
10	917	871	628
11	772	713	765
	-5, 9, L		
1	4918	5195	497
2	2135	2175	378
3	1405	1472	496
4	743	797	799
5	1340	1349	475
9	1780	1729	415
	-5, 11, L		
1	3145	3449	509
3	1451	1471	538
4	1782	1852	486
6	1708	1754	500
8	772	604	868
	-5, 13, L		
2	659	704	1174
4	2310	2462	484
6	1793	1863	574
	-4, 0, L		
2	4527	4439	239
4	7354	7495	288
6	5749	5866	353
12	1447	1465	571
	-4, 2, L		
1	8488	8357	242
3	3097	3045	276
4	3436	3464	297
5	982	962	344
6	2652	2687	377
7	2596	2616	373
8	958	966	538
9	3322	3372	479
10	865	918	687
11	960	944	702
12	872	820	777
13	1192	1292	714
	-4, 4, L		
1	7549	7515	296
2	3384	3343	311
3	1775	1741	247
4	2139	2166	340
5	1648	1615	255
6	2610	2580	362
7	1779	1798	310
8	2178	2181	305
9	3545	3650	462
10	602	760	917
11	872	795	676
	-4, 6, L		
1	807	794	520
2	2375	2398	342
3	2365	2352	357
4	4387	4375	374
5	4752	4685	376
6	3654	3718	418
7	1082	1101	476
9	1258	1330	450
	-4, 8, L		
1	2511	2578	365
2	2727	2800	412
3	3215	3307	466
4	1668	1698	399
5	3023	3051	435
6	1043	1121	568
7	2127	2121	344
8	1904	2019	376
9	838	808	677
10	1475	1562	472
11	682	529	840
	-4, 10, L		
1	1253	1337	600
2	3349	3610	551
4	589	688	1146
5	987	1122	722
6	886	948	769
8	2393	2505	404
9	911	974	744
	-4, 12, L		
2	1155	1286	710
3	2498	2634	433
5	3350	3468	473
7	1295	1348	690

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ
	-4, 12, L		
8	952	968	832
	-4, 14, L		
2	1822	1868	562
3	1559	1552	626
4	890	759	974
	-3, 1, L		
1	2396	2328	237
2	3185	3167	236
3	3000	2965	266
4	2027	1966	294
5	7241	7355	302
6	1804	1839	290
7	2456	2453	365
8	2984	3042	434
10	1898	1926	405
11	1175	1225	630
13	2644	2648	441
	-3, 3, L		
1	4291	4128	259
2	3726	3593	265
3	865	879	334
4	538	503	521
5	988	982	335
6	1517	1554	279
7	1092	1075	403
8	4985	5081	404
9	2113	2126	348
10	3448	3550	501
	-3, 5, L		
1	1988	1890	267
3	3648	3613	343
4	3175	3204	351
5	4293	4357	347
6	1401	1396	322
7	3105	3157	406
8	2375	2421	351
9	1796	1876	365
10	1531	1629	427
11	1300	1408	530
12	1292	1285	550
	-3, 7, L		
1	2182	2234	316
2	1592	1635	371
3	1998	1992	327
4	4065	4120	430
5	3526	3673	440
6	3732	3776	440
7	1030	878	491
8	1219	1159	447
9	1210	1224	464
10	973	1081	620
12	1597	1706	486
	-3, 9, L		
1	4438	4566	508
2	1713	1783	465
3	1184	1208	601
4	928	956	725
6	1096	1036	606
7	2352	2337	371
8	1555	1544	467
9	3325	3387	464
10	1414	1481	492
	-3, 11, L		
1	2534	2574	406
3	1068	1037	691
4	2328	2494	447
6	1993	2026	496
7	1953	2033	509
9	2215	2315	465
	-3, 13, L		
2	794	835	1003
4	3372	3487	528
6	2718	2829	481
	-3, 15, L		
1	1974	1885	561
	-2, 0, L		
2	633	571	371
4	5306	5264	284
6	4601	4609	360
8	688	699	821
10	853	831	769
12	3568	3762	551
	-2, 2, L		
1	2818	2668	234
2	746	719	324
3	660	605	387
4	3974	3951	267
5	489	399	630
6	1792	1826	282
7	2523	2586	385
8	1705	1678	376
9	4295	4448	472
11	1981	1945	434
12	1783	1760	512
	-2, 4, L		
1	4450	4370	315
3	1268	1257	292
4	1348	1373	274
5	2840	2847	334
6	1588	1645	278
7	2755	2801	408
8	2516	2540	383
9	4104	4273	454
10	1486	1601	464
11	1186	1235	597
12	1233	1243	630
13	833	803	945
	-2, 6, L		
1	3212	3295	436
2	927	911	508
3	1452	1444	362
4	3385	3384	410
5	3906	3910	392
6	2574	2615	385
7	862	763	525
9	547	702	924
10	875	903	628
11	1104	1074	577
12	2087	2182	429
	-2, 8, L		
1	1313	1337	498
2	678	742	901
3	1277	1348	536
4	2511	2586	367
5	2212	2193	378
6	984	1020	638
7	1796	1902	408
8	2726	2823	399
9	1490	1584	457
10	1581	1712	452
11	1787	1839	419
	-2, 10, L		
1	1333	1305	586
4	993	1040	787
5	1140	1188	693
8	3369	3332	470
9	998	1097	734
10	2223	2279	438
	-2, 12, L		
1	2436	2346	430
3	1277	1371	689
5	2700	2794	456
7	830	1024	1108
	-2, 14, L		
1	1641	1591	600
3	994	875	872
5	2158	2117	538
	-1, 1, L		
1	7341	7314	155
2	1388	1322	207
3	1497	1446	214
4	2368	2370	350
5	3220	3227	351
6	609	633	686
7	680	628	696
8	1628	1682	410
9	1373	1414	505
10	1354	1384	549
11	1454	1518	554
13	3104	3211	449
	-1, 3, L		

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	-1, 3, L			2	5313	5280	221	5	1185	1192	657
2	2015	1946	290	4	1546	1585	300	6	2000	2065	479
3	1520	1440	223	6	1032	1043	478	8	1474	1270	596
4	4951	4934	261	8	4274	4494	472	9	1293	1065	698
5	1825	1812	252	10	1349	1350	589		0, 12, L		
6	1111	1116	361	12	2541	2722	463	1	3942	4026	563
7	929	990	495		0, 2, L			2	1115	1190	728
8	3974	4139	453	0	5820	5824	229	4	1458	1485	611
9	1783	1783	415	1	2246	2297	313	7	1690	1495	591
10	2496	2479	377	2	1265	1269	303		0, 14, L		
	-1, 5, L			3	5759	5743	294	0	932	924	946
1	4862	5044	382	4	2880	2950	384	1	2448	2435	499
2	749	819	668	5	5091	5181	358	2	1446	1374	637
3	1053	1008	452	8	2831	2879	429	4	1913	1839	577
4	3420	3423	379	9	1464	1477	528		1, 1, L		
6	1388	1448	329	10	984	791	701	0	978	749	193
7	504	533	845	12	1795	1837	555	1	7759	7489	222
8	2519	2571	379		0, 4, L			2	2994	3026	336
10	1202	1284	545	0	3933	3958	335	3	1171	1198	420
11	1518	1575	506	1	1450	1391	316	4	7132	7005	346
12	1661	1671	526	2	2535	2529	346	6	2733	2742	421
13	2013	2042	531	3	4236	4247	352	7	4516	4636	457
	-1, 7, L			4	3711	3727	385	9	3696	3704	514
1	3829	3932	464	5	5050	5151	394	12	1074	1137	895
2	2168	2232	350	6	1856	1907	357	13	1429	1467	712
3	835	918	730	7	582	721	981		1, 3, L		
4	1024	1080	625	9	2110	2166	434	0	667	553	614
5	2441	2543	367	13	1519	1375	647	2	3886	3918	353
6	758	606	776		0, 6, L			3	1251	1222	391
9	1610	1746	389	0	5879	6105	379	4	8452	8484	369
10	675	615	794	1	4495	4600	389	5	1764	1803	351
11	639	656	898	2	3707	3773	406	6	3898	3956	454
12	2172	2302	431	5	699	648	766	8	726	605	924
	-1, 9, L			6	669	742	936	11	744	696	1059
2	1393	1416	551	7	1964	1913	431	12	2423	2336	486
3	2407	2471	408	8	2126	2122	438		1, 5, L		
5	2930	3083	429	9	1883	1872	476	0	5356	5403	375
7	1365	1450	601	12	1177	1451	898	1	4443	4552	390
8	616	681	1167		0, 8, L			2	1247	1275	416
9	2653	2775	414	0	2568	2650	377	3	3551	3639	415
10	1181	1205	598	1	4029	4181	473	4	3882	4059	435
	-1, 11, L			2	617	442	856	5	3217	3276	444
2	1303	1244	630	3	2502	2528	381	6	886	981	703
3	1750	1773	535	4	3956	4006	525	7	3195	3262	489
4	1425	1532	654	5	2064	2093	416	8	3012	3015	423
5	1432	1465	644	6	1269	1276	609	9	2510	2537	422
7	1133	1086	839	7	1571	1525	530	10	1027	910	771
9	1555	1651	668	8	2242	2186	439	11	661	720	1304
	-1, 13, L			11	781	755	1154	12	1919	1872	590
2	1612	1569	591		0, 10, L				1, 7, L		
4	1383	1330	689	1	1198	1231	631	0	5670	5763	432
	-1, 15, L			2	2463	2500	390	1	3317	3315	468
3	1336	1306	748	3	1073	1007	666	2	2859	2920	399
	0, 0, L			4	4269	4467	570	3	682	642	790

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	1, 7, L			1	1742	1716	299	3	3838	3782	314
4	1042	1005	577	2	2474	2495	371	4	1954	1961	284
5	962	823	672	3	5229	5110	380	5	1437	1387	405
6	2802	2777	437	4	3241	3087	444	6	1243	1298	523
7	2151	2190	449	5	5192	4982	434	7	5071	4915	505
8	3673	3597	551	6	2443	2377	360	9	4094	3889	603
9	2492	2466	449	8	2167	2075	430	10	1297	1126	692
10	1458	1322	635	10	1671	1636	560	12	2344	2179	542
	1, 9, L			11	2069	2024	530		3, 3, L		
0	1248	1189	567	12	1634	1537	649	0	7203	6990	263
2	1688	1671	462	13	2512	2317	549	1	480	502	726
3	4528	4436	513		2, 6, L			3	2433	2485	386
4	2020	2064	456	0	3030	3097	443	4	4732	4761	396
5	4645	4577	558	1	2828	2797	431	5	1554	1567	414
6	1934	1890	490	2	1559	1579	387	6	2474	2372	396
8	852	925	959	3	876	793	601	10	1722	1633	572
11	1262	1193	806	6	2565	2551	462	11	1354	1317	740
	1, 11, L			7	3639	3517	497	12	3992	3646	629
0	3210	3251	480	8	3959	3747	574	13	878	954	1251
1	1072	972	712	9	2795	2640	438		3, 5, L		
2	1194	1221	674	10	2417	2258	471	0	4143	4203	363
3	3148	3080	451		2, 8, L			2	869	820	545
5	3074	2987	438	0	3083	3115	448	3	565	541	867
7	873	655	932	1	1739	1703	394	4	3433	3363	473
8	2394	2131	487	2	676	732	892	7	2757	2568	388
	1, 13, L			3	1858	1792	403	8	2778	2584	402
0	3886	3979	613	4	4001	3856	525	9	2741	2534	413
2	2231	2071	476	5	2290	2197	415	11	2263	2010	522
6	1565	1532	674	6	1148	1011	645	12	1948	1832	613
	1, 15, L			7	3318	3096	474		3, 7, L		
0	1277	1314	771	8	1928	1835	502	3	2185	2152	350
3	2526	2340	508	9	2549	2296	457	4	1127	996	553
	2, 0, L			11	1305	1152	762	5	1323	1272	552
0	6993	6837	155		2, 10, L			6	2031	1827	437
2	1719	1602	268	0	1881	1933	464	7	2327	2149	413
4	1168	1206	463	1	857	897	830	8	3313	2992	483
6	3936	3873	482	2	2170	2188	434	9	2411	2300	461
8	7545	7552	499	3	1258	1125	603	10	2014	1854	539
10	3016	3036	428	4	4661	4375	552		3, 9, L		
	2, 2, L			5	1252	1207	637	0	1550	1580	507
0	4461	4288	221	6	2488	2324	426	1	1880	1854	443
2	518	296	768	7	673	536	1179	3	2667	2758	450
3	5536	5619	341	10	1331	1192	779	4	1063	1006	699
4	1946	1925	322		2, 12, L			5	2800	2632	444
5	6219	5947	403	1	2209	2136	453	6	1253	1150	637
6	1151	1106	548	4	1387	1372	633	10	752	799	1232
7	1862	1858	410	7	3084	2769	443		3, 11, L		
8	2995	3055	472		2, 14, L			0	1606	1538	526
10	1166	1125	734	1	1489	1413	635	1	1213	1234	649
11	2752	2759	465	4	1936	1791	566	3	1748	1602	492
12	1206	1144	796		3, 1, L			4	1865	1691	480
13	2273	2189	561	0	2905	2720	222	5	1788	1647	530
	2, 4, L			1	1183	1115	237	6	1034	833	734
0	1156	1089	387	2	479	333	623	8	2420	2197	508

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	3, 13, L			5	799	877	895	7	966	1038	769
0	1280	1074	661	7	826	827	927	8	2779	2682	436
4	1392	1411	681	8	2164	2039	475	12	1740	1610	640
6	886	924	1027	9	1058	881	811		5, 7, L		
	3, 15, L				4, 10, L			0	1639	1619	359
0	1223	979	740	0	4387	4554	541	2	3915	4091	475
1	966	1044	932	1	676	767	1047	3	3477	3569	511
	4, 0, L			2	1001	933	703	4	4252	4218	504
0	1345	1315	214	3	1248	1190	608	5	2397	2360	387
2	4470	4380	248	4	1173	1116	652	6	1154	1142	638
4	5950	5910	310	5	831	784	902	7	1294	1188	593
6	551	533	937	7	791	808	1008	9	938	947	891
8	3427	3482	504	8	1365	1241	669	11	1113	1083	883
10	1918	1742	532		4, 12, L				5, 9, L		
	4, 2, L			0	1758	1684	500	0	2233	2286	383
0	3340	3249	262	1	675	697	1083	1	2885	3038	440
1	2739	2640	304	3	2788	2623	414	2	1349	1350	528
2	737	795	471	5	1725	1527	541	4	1073	1000	673
4	3984	3946	373	7	1569	1443	632	5	602	587	1186
5	889	866	550		4, 14, L			6	1010	1020	741
7	1705	1596	469	0	2129	2016	507	7	3044	2887	446
8	2457	2376	426	2	721	811	1198	8	1066	943	782
10	1004	787	844	3	2096	1944	517	9	1417	1362	681
11	2806	2588	470		5, 1, L				5, 11, L		
12	1121	954	905	0	4119	4062	260	1	2397	2501	426
13	2526	2293	539	1	2512	2427	301	2	1709	1726	496
	4, 4, L			2	2457	2371	317	4	2560	2524	421
0	3448	3421	331	3	6870	6899	300	5	908	895	870
1	4092	4040	334	4	1405	1412	312	7	2117	1927	488
2	2144	2145	308	5	3554	3613	399	8	1240	1110	755
3	2505	2524	366	6	2009	2081	344		5, 13, L		
4	978	1045	526	7	949	1135	711	1	769	684	1033
5	1282	1302	480	8	1412	1556	587	2	2874	2779	439
7	3053	2934	463	9	778	1055	1031	4	3529	3357	554
9	1176	1134	703	11	1672	1531	636	6	968	824	901
10	1321	1128	654	12	958	929	1103		6, 0, L		
11	2553	2313	482		5, 3, L			0	448	449	576
12	1375	1275	762	0	7179	7071	285	2	5843	5854	285
	4, 6, L			1	1240	1168	288	4	6846	7041	332
0	800	703	545	2	2369	2292	355	6	2309	2340	354
1	543	687	857	4	545	542	807	10	1939	2135	515
2	2363	2402	361	6	2715	2784	371	12	2631	2515	515
3	3875	3975	458	7	1960	1903	418		6, 2, L		
4	3387	3371	478	8	3538	3521	539	0	1195	1138	271
5	1497	1413	490	11	1195	973	776	1	3569	3505	314
7	2531	2324	395	12	2189	2107	570	2	2803	2802	350
8	2087	1846	466		5, 5, L			3	1191	1203	347
9	1760	1598	533	0	3697	3663	360	4	3574	3675	392
10	1358	1244	698	1	2980	3068	406	5	2428	2500	355
	4, 8, L			2	1163	1156	402	7	4099	4324	503
0	3597	3854	499	3	4343	4528	427	8	1326	1394	582
1	2773	2929	428	4	3169	3211	452	9	2494	2510	427
3	2562	2468	424	5	3449	3497	469	10	683	695	1183
4	2694	2579	400	6	841	752	794	12	1788	1634	629

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	6, 4, L			8	1898	1984	427	2	973	969	384
0	2822	2899	368	10	1240	1180	672	4	2901	3013	422
1	2714	2732	390	11	3014	3071	478	6	564	627	866
2	2412	2473	399		7, 3, L			10	2489	2549	422
3	1453	1436	361	0	2763	2765	353		8, 2, L		
4	2219	2213	340	1	633	632	546	0	1773	1776	295
5	709	649	802	3	882	841	481	1	1019	991	368
6	2814	2859	398	6	3685	3827	483	2	1695	1723	287
7	3927	4157	543	7	1950	2027	410	3	2077	2101	340
8	1551	1624	577	8	3891	3995	564	4	1820	1914	328
9	2557	2550	450	9	1088	1231	752	6	713	836	799
10	1133	1015	762	10	1617	1577	583	7	2879	3016	448
	6, 6, L				7, 5, L			8	740	774	882
1	893	875	530	0	2207	2221	306	9	2163	2311	451
2	3284	3281	455	1	1168	1198	397	10	1170	1137	712
3	4440	4497	456	2	1697	1701	336		8, 4, L		
4	4345	4472	478	3	2444	2501	398	0	892	899	456
5	2446	2648	376	4	2444	2553	368	1	833	805	504
6	1993	2029	439	5	2794	2838	429	2	1004	940	485
10	1220	1094	731	6	1172	1222	617	3	3367	3388	439
11	1962	1789	566	7	1507	1625	522	6	1076	1164	608
	6, 8, L			8	2185	2165	437	7	2965	3064	428
0	2872	2916	425	9	1704	1700	566	8	1212	1377	649
1	2472	2583	380	11	2107	1919	531	9	1862	1905	500
2	815	696	726		7, 7, L			10	1290	1220	664
3	2763	2941	435	2	2139	2136	361	11	830	779	1078
4	1603	1632	493	3	2975	3059	440		8, 6, L		
5	2716	2731	419	4	2868	3028	472	0	2689	2696	411
6	1831	1712	465	5	1404	1536	537	1	733	607	681
7	1695	1546	502	6	1261	1232	589	2	780	772	698
8	2442	2284	446	7	1294	1263	603	3	2161	2209	359
9	1398	1434	661	8	1028	996	791	4	1771	1824	419
	6, 10, L			10	2319	2195	507	5	891	972	715
0	3032	3232	456		7, 9, L			7	1026	1196	743
2	1216	1299	615	1	811	832	783	10	1466	1441	664
3	1004	1000	707	2	690	702	891		8, 8, L		
4	864	772	813	3	1159	1296	627	0	1446	1503	447
6	2746	2733	441	5	1064	1045	707	1	851	910	696
7	1088	1010	787	6	1194	1067	662	2	1611	1607	439
8	2867	2785	450	7	3205	3178	515	4	1580	1697	482
	6, 12, L			8	1668	1618	584	6	1406	1374	569
0	848	818	886	9	2090	2067	547	8	1887	1920	543
1	1300	1294	636		7, 11, L			9	1461	1430	653
3	3536	3515	517	0	887	918	779		8, 10, L		
5	2698	2613	450	2	1438	1375	544	2	1473	1701	547
6	1237	1176	725	4	2197	2206	458	3	889	857	782
	6, 14, L			5	1312	1181	648	4	1024	988	741
0	1448	1491	654	7	2413	2365	481	6	1784	1750	520
	7, 1, L				7, 13, L			7	786	782	1068
0	1499	1477	253	0	1127	1139	748		8, 12, L		
1	579	566	502	2	1713	1700	540	3	1564	1579	574
3	5164	5216	341	4	2465	2437	485	5	726	741	1129
5	3296	3436	426		8, 0, L				9, 1, L		
6	2073	2148	339	0	3694	3697	335	0	1181	1172	364

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	9, 1, L				10, 2, L			1	1505	1580	423
1	2086	2078	302	0	2066	2055	303	2	1776	1773	386
2	2542	2571	371	1	3174	3142	421	3	1621	1595	419
3	1306	1338	385	2	583	630	799	4	1163	1126	541
4	1771	1751	336	3	4454	4549	427	5	1928	1985	433
5	739	710	691	5	2207	2246	343	6	1276	1326	596
7	2071	2151	369	6	1805	1844	393	7	2195	2229	441
8	831	831	745	8	1433	1387	512	8	892	938	873
11	2212	2266	500		10, 4, L				11, 7, L		
	9, 3, L			0	2191	2147	342	0	1775	1848	410
0	1091	1086	413	1	2232	2204	311	3	945	933	704
1	986	944	455	2	2336	2327	342	4	1282	1377	588
2	4187	4251	408	3	4187	4245	458	5	823	801	839
3	1830	1842	344	4	1881	1946	384	6	2202	2328	450
4	3143	3145	436	5	1301	1267	490	7	1897	1928	501
6	981	981	609	6	899	1001	725		11, 9, L		
7	1206	1188	552	7	1223	1198	572	1	1661	1647	480
8	1960	1940	423		10, 6, L			2	1196	1212	630
	9, 5, L			0	2724	2640	376	3	3022	3108	427
0	820	844	600	1	785	827	710	4	1277	1303	640
1	2634	2557	395	2	986	979	589	5	830	990	932
2	2036	2053	341	4	1130	1182	577		11, 11, L		
3	961	1031	589	5	1164	1180	576	0	1238	1215	627
4	2024	2048	367	6	2022	2133	452	1	1256	1375	671
5	768	866	798	7	1801	1872	494		12, 0, L		
6	1381	1469	515	8	1521	1667	587	0	707	621	672
7	674	615	983		10, 8, L			2	1456	1460	392
8	1443	1563	585	0	554	358	1011	6	3032	3129	473
10	840	845	1041	1	1905	1934	409	8	2615	2817	401
	9, 7, L			2	2059	2140	419		12, 2, L		
0	2804	2836	450	3	1580	1599	477	1	837	743	594
1	774	764	719	4	1612	1710	495	2	1329	1289	439
2	799	981	774	5	1858	1851	472	3	2500	2605	353
3	1196	1148	517	6	838	892	868	5	1199	1074	499
6	1207	1215	620	7	1322	1372	664	6	1541	1711	480
7	1638	1707	537		10, 10, L			8	1318	1347	597
	9, 9, L			0	1394	1253	523		12, 4, L		
0	1185	1113	545	2	3042	3102	474	1	947	954	591
1	1659	1550	440	3	1110	1079	697	2	993	968	577
2	1069	1073	658	4	2469	2588	448	3	2261	2276	359
3	2911	3067	446		11, 1, L			4	1047	1053	603
4	1027	1120	723	1	754	769	618	6	852	948	806
5	661	707	1098	2	2050	2043	327	7	618	554	1120
7	1436	1481	625	4	1950	1991	346	8	1272	1324	642
	9, 11, L			5	2150	2262	392		12, 6, L		
0	1690	1719	499	7	3112	3288	465	5	1249	1262	591
1	1598	1602	531	9	1454	1459	558	6	1728	1744	505
3	2060	2123	482		11, 3, L			7	1788	1882	537
	10, 0, L			1	1086	1049	469		12, 8, L		
0	4256	4226	381	2	3944	3874	450	2	1789	1774	466
2	769	800	565	3	1585	1606	419	4	1250	1258	625
4	989	990	507	4	3673	3704	483	5	1286	1254	639
6	3480	3585	485		11, 5, L				13, 1, L		
8	2563	2603	365	0	888	777	562	0	1017	996	508

Table C.2. Observed and Calculated Structure Factors for $Os_4(CO)_{14}$. Columns Are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

L	KF_o	F_c	σ	L	KF_o	F_c	σ	L	KF_o	F_c	σ
	13,	1, L		1	941	945	710	3	598	562	1040
1	1604	1594	392	2	1365	1447	568	4	850	843	793
3	1893	1936	394	3	1513	1484	532	5	1172	1100	649
7	1856	1986	465		14,	0, L			14,	6, L	
	13,	3, L		0	1835	1788	390	0	1259	1150	533
0	1423	1423	441	2	3227	3185	484	1	1571	1577	506
2	966	1017	622	4	1564	1572	459	2	2133	2125	438
3	745	699	778		14,	2, L		3	1696	1732	499
6	697	731	939	0	560	524	985		15,	1, L	
	13,	5, L		1	1060	1041	590	0	716	812	862
1	1289	1143	493	2	1742	1670	423	1	2506	2454	380
2	1538	1523	455	5	1042	1078	640	3	2443	2484	396
6	1440	1446	553		14,	4, L		4	805	769	823
	13,	7, L		0	1367	1362	502		15,	3, L	
0	1054	1056	615	2	992	1056	669	0	1575	1550	465

TABLE D.1. U_{ij} or U_{iso} Values (*100) for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

	U11(U)	U22	U33	U12	U13	U23
Os(1)	4.63(10)	3.98(9)	4.01(9)	-0.13(7)	0.34(7)	-0.60(7)
Os(2)	4.47(10)	3.61(8)	4.15(9)	0.59(7)	0.38(7)	-0.53(7)
Os(3)	4.62(10)	3.31(8)	4.15(9)	0.13(7)	1.14(7)	-0.66(7)
Os(4)	4.23(11)	5.04(10)	6.13(12)	0.20(8)	0.34(8)	0.24(9)
P(1)	6.0 (7)	4.4 (6)	5.8 (7)	1.5 (5)	2.1 (6)	-0.3 (5)
P(2)	10.5 (12)	7.2 (9)	8.4 (10)	-2.4 (8)	-0.6 (8)	0.3 (7)
O(11)	11.5 (24)	6.0 (18)	7.9 (19)	-3.8 (18)	2.9 (17)	-0.4 (16)
O(12)	11.2 (26)	7.9 (21)	7.0 (20)	-0.5 (18)	2.8 (19)	0.1 (17)
O(13)	13.0 (29)	13.0 (26)	6.9 (20)	-0.5 (22)	0.9 (20)	-6.9 (20)
O(21)	11.0 (25)	6.3 (20)	12.4 (26)	-0.1 (18)	5.2 (20)	4.1 (18)
O(22)	10.3 (23)	5.3 (17)	8.7 (21)	1.8 (17)	3.0 (18)	1.4 (17)
O(23)	12.1 (29)	12.6 (27)	7.2 (22)	3.9 (23)	-1.2 (20)	-2.6 (20)
O(31)	6.0 (18)	4.3 (15)	7.4 (19)	-1.2 (13)	2.6 (15)	-0.9 (13)
O(32)	6.2 (18)	6.0 (18)	6.5 (18)	-1.7 (14)	-1.3 (14)	0.5 (14)
O(33)	8.3 (23)	7.3 (21)	11.2 (25)	0.5 (16)	0.0 (19)	-4.2 (18)
O(34)	11.3 (25)	2.9 (15)	15.9 (29)	1.3 (16)	6.5 (22)	-1.6 (17)
O(41)	12.1 (28)	11.1 (26)	10.5 (25)	-5.1 (21)	0.9 (21)	7.3 (21)
O(42)	6.1 (21)	13.5 (30)	12.6 (28)	0.6 (20)	-1.5 (19)	5.9 (25)
O(43)	13.8 (35)	14.0 (34)	13.0 (31)	3.4 (27)	-1.8 (26)	-4.9 (26)
O(45)	30.0 (58)	8.9 (28)	11.5 (31)	-6.1 (31)	6.2 (35)	-0.5 (24)
O(46)	10.0 (28)	21.5 (43)	9.7 (26)	-5.1 (29)	-0.7 (21)	-4.4 (28)
O(47)	14.0 (37)	19.9 (42)	19.3 (42)	9.4 (33)	-11.6 (33)	-6.2 (34)
C(11)	4.8(10)					
C(12)	4.7(10)					
C(13)	5.0(9)					
C(21)	6.5(12)					
C(22)	4.6(10)					
C(23)	8.0(13)					
C(31)	5.7(11)					
C(32)	6.0(11)					
C(33)	5.0(9)					
C(34)	5.4(10)					
C(41)	8.7(14)					
C(42)	10.0(17)					
C(43)	11.1(20)					
C(25)	5.0(9)					
C(26)	7.6(13)					
C(27)	9.8(15)					
C(45)	13.9(24)					
C(46)	11.9(19)					
C(47)	20.7(33)					

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ
h, 0, 0			
1	216	203	142
3	78	76	573
4	315	329	318
5	520	514	318
6	201	204	478
7	76	47	1134
9	306	309	565
10	112	113	1142
11	167	158	841
h, 1, 0			
1	533	539	125
2	71	77	493
3	147	140	384
4	300	300	312
5	297	300	361
6	229	233	440
7	60	73	978
9	95	77	1137
10	237	236	666
h, 2, 0			
1	391	378	148
3	215	220	290
4	155	163	407
6	175	178	510
8	106	105	878
h, 3, 0			
1	163	150	226
2	87	86	424
3	187	194	308
5	157	168	471
6	212	208	440
7	85	95	975
9	129	121	885
10	125	129	989
11	102	105	1196
h, 4, 0			
0	181	167	227
1	73	76	459
2	38	37	485
4	143	146	409
5	171	172	424
9	68	71	911
h, 5, 0			
2	81	79	526
3	144	146	372
4	172	175	372
6	135	124	562
7	76	76	1045
9	136	128	772
h, 6, 0			
0	66	46	572
1	363	349	238
2	157	148	335
3	224	229	308
4	243	248	342
6	225	232	442
8	152	160	687
11	76	58	1029
h, 7, 0			
1	314	314	271
2	60	60	751
3	58	54	563
4	273	268	339
5	212	215	415
6	95	95	772
7	97	92	841
9	111	121	938
10	137	136	893
h, 8, 0			
0	805	764	253
2	49	48	844
4	238	240	381
5	408	397	354
6	133	139	619
9	192	187	647
h, 9, 0			
1	239	240	352
4	108	105	640
5	212	206	458
6	147	149	612
10	144	140	911
h, 10, 0			
0	77	74	753
2	161	161	443
4	99	93	728
5	89	96	886
7	85	91	884
h, 11, 0			
1	280	286	387
2	126	130	583
3	190	191	460
4	117	106	665
5	160	166	625
6	202	201	540
8	82	91	1186
9	56	56	1006
h, 12, 0			
0	360	360	367
4	156	167	574
5	218	214	508
h, 13, 0			
2	88	76	916
4	126	122	747
h, 14, 0			
1	179	189	603
3	92	73	884
4	144	153	696
6	122	123	819
h, 15, 0			
2	79	87	1132
3	79	78	1112
4	115	109	830
h, 16, 0			
0	213	196	531
5	134	119	827
h, 1, 1			
-11	156	154	919
-10	177	166	805
-7	86	103	1015
-6	358	366	378
-4	143	146	414
-2	113	116	289
-1	399	398	127
0	378	381	124
1	276	267	182
2	91	102	457
3	211	204	334
5	389	388	346
6	97	109	805
9	158	154	797
10	186	191	779
h, 2, 1			
-11	92	88	1387
-10	184	195	815
-9	154	144	788
-7	98	89	838
-5	529	539	294
-4	133	138	404
-1	528	508	141
0	872	784	136
1	84	86	368
4	539	556	287
5	234	231	399
6	186	172	498
9	243	234	613
h, 3, 1			
-10	99	90	1095
-9	84	89	1247
-7	168	171	553
-6	221	228	425
-5	71	57	638
-4	225	230	309
-3	53	40	721
-2	268	264	210
-1	440	425	166
0	119	111	272
1	73	62	440
3	94	97	550
4	403	415	296
5	244	247	390
8	128	125	808
9	93	90	746
10	111	107	1050
h, 4, 1			
-11	129	133	986
-9	91	93	1108
-6	349	365	353
h, 5, 1			
-9	108	96	848
-7	219	223	470
-6	73	81	869
-4	60	55	775
-3	38	39	675
-2	357	335	232
-1	188	179	274
0	105	90	385
3	91	89	556
4	354	351	308
5	77	76	772
6	138	144	598
8	113	122	946
9	128	138	906
h, 6, 1			
-10	79	87	1008
-5	306	307	342
-4	64	60	487
-2	72	78	576
-1	196	198	285
0	197	183	293
1	154	144	334
4	240	238	357
5	90	94	724
6	122	132	646
7	81	82	981
9	94	105	1183
h, 7, 1			
-11	108	105	1101
-10	88	66	1190
-7	147	160	618
-6	206	206	461
-4	79	76	703
-3	71	80	688
-2	139	141	401
0	218	194	299
1	133	125	392
2	113	113	460
4	157	159	466
5	258	253	395
8	74	71	995
9	139	152	841
10	99	94	988
h, 8, 1			
-7	99	92	831

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are $1F_o$ $1F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 8, 1				3	263	264	436	-4	96	99	488	-7	239	246	449
-6	125	124	645	4	119	114	711	-3	80	71	499	-6	147	142	503
-3	130	131	460	8	150	140	755	-1	411	397	166	-5	77	76	675
-2	221	215	330	h, 13, 1				0	461	446	166	-4	64	48	696
-1	142	159	475	-7	124	117	796	1	87	72	416	-3	283	277	271
1	131	128	426	-6	81	69	912	2	226	223	278	-2	204	193	288
2	121	118	478	-4	65	56	927	3	270	262	298	-1	454	440	217
3	209	212	390	-3	62	56	955	4	70	66	791	0	78	59	509
4	64	53	799	-2	205	206	511	5	308	295	355	1	91	91	495
6	72	69	1030	1	164	168	567	7	73	76	758	2	85	90	563
7	101	102	846	3	89	98	984	8	86	83	1141	3	140	147	441
8	98	95	959	4	104	117	843	9	120	108	958	4	407	414	319
h, 9, 1				5	81	76	1068	10	112	119	1073	8	280	282	525
-10	163	150	782	6	107	102	893	h, 2, 2				9	113	107	938
-9	89	82	1125	h, 14, 1				-11	142	135	925	h, 6, 2			
-6	303	306	429	-4	82	80	803	-9	62	57	1018	-11	141	146	889
-4	220	217	403	1	70	90	958	-8	112	116	805	-8	81	75	1049
-2	70	65	775	2	72	55	1081	-7	206	206	487	-7	295	289	410
-1	386	374	304	h, 15, 1				-6	245	249	377	-6	319	323	376
0	304	284	326	-2	108	104	908	-3	59	57	642	-4	76	82	683
1	248	248	345	0	85	101	662	-2	465	471	181	-3	56	33	747
3	142	140	484	4	130	132	800	-1	181	182	214	-2	702	697	235
4	75	83	937	h, 16, 1				2	162	151	342	-1	91	84	491
5	310	313	398	-2	125	137	855	3	577	572	262	2	226	221	322
6	95	94	829	-1	89	103	1137	4	137	138	485	3	496	481	288
8	93	80	941	2	58	47	1011	5	84	88	792	4	148	158	496
10	164	161	827	3	159	162	704	7	152	153	700	7	250	252	513
h, 10, 1				h, 17, 1				8	151	157	737	8	176	179	692
-9	108	114	1009	-4	103	107	1070	9	142	127	808	9	145	158	789
-5	430	435	377	-1	151	159	789	h, 3, 2				h, 7, 2			
-4	95	103	729	1	67	74	965	-11	98	106	798	-11	96	87	1249
-1	248	253	405	h, 18, 1				-8	79	86	1058	-9	77	61	1105
0	566	548	323	0	128	163	1026	-7	236	241	441	-7	88	99	926
1	128	131	529	1	72	80	1144	-5	87	87	622	-6	317	310	380
4	407	413	373	h, 0, 2				-4	91	91	511	-4	74	71	710
5	189	192	529	-10	289	273	620	-3	290	278	242	-3	105	108	515
6	115	136	781	-8	87	84	742	-2	212	214	237	-1	224	249	385
9	175	190	746	-7	98	89	913	-1	216	214	215	0	307	292	273
h, 11, 1				-6	303	291	403	0	77	66	448	2	161	157	393
-9	84	64	1072	-5	473	479	307	1	62	53	639	3	189	186	396
-8	66	65	1203	-3	87	88	464	2	214	214	304	4	156	148	461
-7	86	78	1027	-2	89	88	374	3	74	81	730	5	192	187	472
-6	148	155	673	-1	721	785	156	4	406	402	310	6	87	92	914
-4	114	110	672	0	484	483	151	5	207	208	424	8	168	171	668
-2	124	120	597	1	211	193	217	8	198	205	652	10	93	71	1121
-1	257	264	419	2	152	144	327	9	162	162	786	h, 8, 2			
4	135	142	643	3	85	69	620	h, 4, 2				-10	179	174	683
5	193	192	523	4	620	594	283	-5	164	167	400	-7	78	41	947
h, 12, 1				7	85	100	1065	-3	69	65	605	-6	246	254	429
-6	130	139	742	8	117	109	853	-2	37	14	496	-5	366	361	351
-5	69	47	849	9	217	210	621	-1	147	145	295	-4	87	85	661
-4	119	133	720	10	117	127	1023	0	96	90	407	-3	86	90	624
-3	109	116	699	h, 1, 2				2	70	63	665	-1	454	446	274
-1	227	231	458	-11	164	147	903	4	170	160	437	0	157	145	389
1	116	105	640	-6	338	339	353	h, 5, 2				1	88	91	612
2	111	120	730	-5	80	83	701	-8	135	134	667	2	140	144	459

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Columns are $1F_o$ $1F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 8, 2				-6	185	181	625	h, 3, 3				3	110	118	613
3	79	92	767	-4	67	50	954	-11	114	115	1087	4	129	134	557
4	370	368	354	-2	336	335	435	-8	184	188	548	5	115	117	665
5	88	83	780	2	109	100	819	-7	70	81	1037	6	100	99	813
7	81	96	1174	3	316	320	474	-6	271	274	364	h, 7, 3			
8	111	82	838	7	120	111	939	-5	65	70	768	-8	172	185	616
9	120	117	937	h, 15, 2				-3	467	467	229	-7	112	92	696
10	89	104	1303	-1	110	113	839	-2	318	309	226	-6	76	102	992
h, 9, 2				0	97	82	866	-1	309	312	222	-5	194	197	413
-8	85	75	566	1	75	60	1123	0	92	97	472	-4	198	201	380
-6	169	180	564	4	112	95	844	1	107	107	458	-3	204	200	346
-5	136	132	579	h, 16, 2				2	282	268	294	-2	74	62	606
-3	80	88	743	-5	85	90	1070	3	466	445	290	-1	265	271	294
-1	57	67	827	-4	104	106	1002	5	152	153	531	0	65	57	643
0	314	300	322	-3	75	57	868	8	281	276	549	1	184	181	356
1	102	104	557	-1	161	151	664	9	80	82	763	3	302	299	361
4	136	124	551	1	89	91	1013	h, 4, 3				4	256	256	405
5	208	212	485	2	100	101	980	-11	146	143	824	6	92	80	908
7	72	77	951	5	84	81	1248	-7	458	464	360	7	165	158	637
9	105	117	1097	h, 17, 2				-6	155	155	460	8	194	206	654
h, 10, 2				-3	93	86	1100	-3	290	294	262	9	104	107	991
-9	62	61	1116	0	94	118	1106	-2	1115	1134	211	h, 8, 3			
-8	82	75	1067	h, 1, 3				-1	155	151	303	-7	209	208	501
-3	116	133	608	-11	112	100	1136	0	75	50	579	-6	78	83	917
-2	68	59	739	-8	104	101	862	1	46	33	878	-3	170	170	403
-1	70	61	809	-7	104	104	680	2	426	425	266	-2	269	274	319
2	71	72	920	-6	68	45	812	3	363	363	302	0	63	62	784
3	137	139	554	-5	184	190	368	4	330	328	343	2	230	226	375
h, 11, 2				-4	69	64	537	6	81	83	755	3	140	137	544
-7	114	110	785	-1	110	110	353	7	409	411	441	4	112	115	680
-6	142	140	647	0	128	121	314	9	164	183	802	6	77	90	1137
-4	81	91	897	1	98	93	428	h, 5, 3				7	150	153	708
-2	195	198	450	2	123	122	425	-8	170	169	581	9	71	78	1232
0	194	187	457	3	103	93	547	-7	123	120	666	h, 9, 3			
1	132	133	560	4	323	315	334	-6	228	241	404	-7	51	52	1000
2	74	60	916	5	126	129	565	-3	411	407	258	-6	132	134	662
4	133	144	666	7	156	158	644	-2	260	259	272	-5	125	122	597
5	189	187	572	8	97	101	998	-1	292	278	253	-2	65	75	866
9	94	123	1321	9	121	128	891	0	95	84	500	0	169	164	414
h, 12, 2				h, 2, 3				1	54	57	729	2	138	141	535
-5	190	196	553	-11	92	69	1225	2	68	74	677	3	124	124	592
-1	252	245	430	-10	128	115	806	3	384	385	310	4	118	120	714
0	188	186	492	-8	116	113	750	4	190	199	429	5	89	70	863
4	205	212	552	-6	269	270	352	8	272	286	563	9	101	94	1035
h, 13, 2				-5	175	176	379	h, 6, 3				h, 10, 3			
-7	136	133	732	-4	83	76	537	-11	90	62	1170	-8	101	109	923
-6	117	112	848	-3	243	244	259	-8	71	73	751	-6	234	231	469
-3	165	169	597	-2	68	60	520	-7	55	54	876	-5	118	116	698
-1	265	265	448	-1	442	447	201	-6	131	129	581	-4	59	51	711
2	133	136	675	1	123	119	393	-4	85	89	631	-3	180	177	449
3	99	102	904	2	82	86	642	-3	113	103	460	-1	345	341	341
4	183	186	585	3	141	132	461	-2	91	88	526	1	119	119	579
5	84	81	1032	4	347	345	336	-1	205	200	301	2	115	123	651
8	159	152	787	5	47	59	839	0	100	96	476	3	78	75	920
h, 14, 2				6	152	138	546	1	70	74	689	4	233	237	475
-7	141	133	783	8	90	71	1057	2	136	139	459	5	56	55	830

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 10, 3				0	82	74	1185	-1	162	161	328	-3	311	314	326
6	99	114	980	h, 0, 4				1	73	49	677	-2	262	264	321
h, 11, 3				-11	95	96	1293	2	232	227	344	1	131	125	491
-8	65	57	737	-9	90	83	686	3	410	400	318	2	134	127	507
-6	113	126	843	-8	212	210	526	4	83	76	752	3	321	331	368
-5	74	75	1044	-6	292	295	347	6	68	64	905	6	93	89	936
-3	177	171	487	-4	365	372	273	7	230	226	531	8	135	132	805
-2	113	118	676	-3	277	279	294	8	158	175	756	9	69	80	1332
-1	149	148	514	-2	60	57	782	h, 4, 4				h, 8, 4			
0	137	136	552	-1	188	179	294	-11	84	6	1210	-8	121	120	757
1	68	59	948	1	605	604	222	-5	87	95	644	-6	197	194	478
2	207	202	455	2	169	161	349	-4	125	128	436	-5	110	109	619
3	187	186	512	3	170	167	380	-3	63	56	485	-4	257	257	373
4	84	76	912	4	206	209	389	-1	117	114	428	-3	179	188	422
5	100	86	848	5	108	104	603	0	136	139	410	-2	75	63	719
8	88	97	1196	6	228	224	477	1	59	56	596	-1	178	178	386
h, 12, 3				7	131	128	748	2	103	99	550	0	55	51	568
-7	207	211	581	h, 1, 4				4	110	109	602	1	381	386	333
-3	76	76	974	-11	90	86	1286	h, 5, 4				3	85	83	843
-2	355	366	385	-9	135	119	717	-11	68	68	1041	4	65	66	991
2	208	204	503	-8	164	160	564	-9	73	86	926	6	189	188	559
3	209	202	522	-7	93	86	761	-8	251	247	468	7	81	86	1162
4	140	136	662	-6	58	53	598	-7	81	76	906	h, 9, 4			
7	135	142	871	-4	111	116	442	-6	217	230	418	-9	90	114	1142
h, 13, 3				-3	232	226	298	-4	213	209	338	-4	171	162	463
-8	94	95	1076	-2	188	186	325	-3	307	303	292	-3	105	103	617
-7	96	70	890	1	187	169	321	-2	472	478	256	-1	131	143	511
-3	107	106	783	2	470	457	262	-1	282	276	279	2	226	232	436
-1	137	123	590	3	265	259	334	0	80	77	639	4	194	190	476
1	60	59	1114	4	205	205	403	1	194	196	358	6	101	112	926
2	79	62	1026	6	192	193	519	2	118	122	514	7	108	115	932
3	167	165	619	8	83	80	1170	3	450	458	315	9	66	77	1122
4	66	63	740	9	85	89	1033	5	97	99	767	h, 10, 4			
h, 14, 3				11	112	95	1132	7	226	232	595	-6	62	54	806
-5	102	89	874	h, 2, 4				8	220	224	626	-4	54	40	1049
-4	60	70	1144	-10	62	21	900	h, 6, 4				-3	61	55	792
-2	77	75	1089	-8	101	89	768	-11	73	61	1247	-1	71	77	898
0	143	149	639	-7	320	329	382	-9	71	65	906	0	86	88	760
2	75	82	1072	-3	328	327	258	-8	108	123	831	2	94	100	805
5	99	92	970	-2	277	278	264	-7	411	420	382	h, 11, 4			
h, 15, 3				-1	173	174	315	-3	521	520	276	-9	95	72	1030
-6	114	116	958	0	66	60	701	-2	498	498	266	-8	85	74	764
-4	92	100	1043	1	134	126	421	-1	237	233	307	-4	86	78	846
-3	162	157	654	2	613	598	260	1	107	105	535	-3	74	89	842
-1	216	220	548	4	182	184	445	2	555	560	302	-2	88	100	807
1	161	157	657	6	84	75	908	3	51	53	768	-1	83	86	817
3	193	192	633	7	198	201	583	4	220	220	432	1	69	56	662
4	139	136	769	8	94	93	1111	5	104	107	723	2	138	144	634
5	65	58	848	h, 3, 4				6	162	162	604	3	93	93	930
h, 16, 3				-9	78	83	1137	7	283	299	526	6	105	101	940
-3	69	85	933	-8	208	208	497	8	159	164	763	7	129	121	786
-2	215	221	592	-7	59	57	875	9	93	76	851	h, 12, 4			
2	184	183	626	-6	77	89	799	h, 7, 4				-8	65	70	1175
3	101	129	1135	-4	147	152	376	-8	156	158	637	-7	78	27	1056
4	92	87	1149	-3	39	32	703	-7	74	77	1066	-6	77	67	832
h, 17, 3				-2	384	372	243	-6	143	138	549	-4	146	147	625

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 12, 4				-8	130	126	680	2	182	186	418	-9	188	175	642
-1	109	121	746	-6	89	91	705	3	261	267	386	-8	113	114	862
0	106	109	765	-5	73	80	722	5	90	82	841	-6	56	61	820
1	157	148	567	-4	489	523	275	7	211	212	615	-5	47	55	853
4	121	122	801	-2	96	95	545	h, 6, 5				-4	368	377	386
5	73	74	812	0	269	257	300	-8	94	66	859	-1	87	89	752
7	73	79	810	1	512	486	269	-5	79	69	745	0	195	201	477
h, 13, 4				2	260	255	329	-4	240	245	356	1	356	341	392
-8	176	155	686	3	75	69	684	-2	87	95	626	2	155	169	606
-6	140	140	744	4	115	115	611	-1	44	45	863	5	189	205	600
-3	191	187	553	5	264	264	425	0	65	66	821	7	140	145	774
-2	266	255	475	6	103	97	882	1	254	248	343	h, 11, 5			
-1	78	90	1062	7	174	172	642	2	68	59	876	-8	150	141	736
1	171	164	596	8	79	73	853	5	60	60	788	-7	106	102	877
3	272	268	499	10	98	73	1194	6	78	89	1131	-5	65	51	885
6	84	83	680	h, 3, 5				7	86	104	1219	-4	83	99	961
7	99	92	949	-8	227	215	478	h, 7, 5				-3	82	81	948
h, 14, 4				-7	176	176	515	-10	59	53	1096	-2	176	176	508
-7	249	230	549	-6	68	70	923	-9	111	107	854	1	191	194	534
-3	214	212	540	-4	131	144	447	-8	106	76	838	3	112	114	841
-2	207	209	549	-3	153	150	391	-7	114	105	713	4	90	119	1009
-1	138	131	673	-2	417	421	270	-6	86	87	833	6	142	149	781
1	83	78	1028	0	78	82	694	-5	146	139	500	7	85	78	640
2	337	328	464	1	405	391	291	-4	177	180	440	h, 12, 5			
4	132	138	785	3	290	293	359	-3	152	155	471	-8	150	153	800
h, 15, 4				4	100	93	677	-2	223	220	367	-7	126	111	774
-3	97	101	880	6	161	158	602	-1	111	106	519	-6	91	92	996
-2	142	145	734	7	208	217	597	0	148	156	474	-5	59	40	1046
3	152	154	729	8	62	74	1087	1	68	72	880	-3	240	241	481
h, 16, 4				h, 4, 5				2	308	318	367	0	121	125	780
-5	106	113	1050	-8	294	302	451	3	215	220	436	1	104	90	830
-3	106	95	725	-7	309	316	405	6	162	161	658	2	211	215	552
0	93	104	1069	-6	71	85	912	7	167	185	686	3	177	172	620
1	136	135	760	-5	83	79	650	h, 8, 5				h, 13, 5			
h, 17, 4				-4	67	71	753	-8	97	80	920	-8	87	55	1280
-1	155	144	678	-3	652	658	265	-7	149	152	621	-7	123	112	876
3	98	97	1113	-2	112	110	457	-3	284	285	359	-3	87	79	943
h, 1, 5				-1	107	118	479	-2	126	124	481	-2	164	158	575
-10	102	92	981	0	92	95	621	-1	80	83	703	-1	87	94	944
-9	139	136	700	1	212	210	371	1	66	56	954	2	157	153	662
-8	69	66	640	2	525	510	310	2	257	257	406	3	86	98	1149
-5	192	196	383	3	191	194	446	6	82	93	1179	4	84	74	1062
-4	42	45	804	5	145	148	621	h, 9, 5				5	93	91	1023
-3	271	267	311	6	202	207	550	-9	106	95	1002	6	89	72	1117
-2	65	62	769	7	168	168	693	-8	112	114	855	7	108	114	1066
-1	40	33	658	8	221	226	637	-5	151	154	544	h, 14, 5			
0	85	81	574	h, 5, 5				-4	57	62	1017	-7	123	43	893
1	300	293	288	-9	65	59	1131	-3	261	269	393	-5	116	102	819
2	486	465	278	-8	153	145	625	-2	68	69	877	-1	83	78	1001
3	92	91	686	-7	145	145	590	-1	48	44	651	0	74	87	1054
5	64	54	893	-4	108	112	562	0	60	57	587	4	85	102	1172
6	336	343	435	-3	60	62	856	1	212	216	475	h, 15, 5			
7	79	83	1172	-2	443	444	277	2	158	175	557	-6	80	77	848
10	74	81	843	-1	88	84	568	3	123	112	636	-5	86	72	1058
h, 2, 5				0	60	73	873	6	244	258	553	-4	101	107	1037
-9	228	220	521	1	168	166	399	h, 10, 5				-2	182	174	644

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 15, 5				-10	118	99	795	-8	109	100	852	h, 12, 6			
0	72	82	1134	-9	66	56	746	-7	100	86	810	-8	89	16	988
2	120	128	900	-8	106	51	805	-5	189	179	468	-5	143	128	675
3	154	154	753	-7	155	161	568	-4	309	310	363	-4	185	183	566
h, 16, 5				-6	134	147	541	-3	80	82	641	-3	99	98	838
-3	192	184	660	-5	68	77	764	-2	175	172	418	-1	69	85	722
-2	80	68	1165	-3	215	218	357	0	122	130	556	0	256	250	492
2	153	164	774	-2	86	94	651	1	328	335	361	1	78	57	941
h, 17, 5				-1	160	165	401	2	171	169	493	2	109	119	877
-3	100	95	1088	0	75	66	712	3	121	123	657	4	117	109	785
0	64	74	1125	1	239	239	363	6	179	186	634	5	124	128	869
1	86	67	1204	2	370	365	329	7	169	161	666	7	87	78	954
h, 0, 6				5	205	206	505	h, 8, 6				h, 13, 6			
-9	392	373	441	6	199	195	570	-9	239	233	550	-7	136	115	827
-5	405	423	345	7	107	115	974	-5	232	241	429	-4	91	89	1017
-4	600	643	311	9	84	73	1235	-4	464	468	352	-2	138	127	641
-3	188	189	385	10	122	123	1017	-3	140	146	551	1	103	113	953
-1	107	112	541	h, 4, 6				0	365	370	355	2	150	147	709
0	448	444	274	-9	84	79	1057	1	145	149	539	h, 14, 6			
1	213	210	346	-8	133	15	700	2	231	238	449	-5	88	86	1068
2	322	320	322	-5	89	100	692	3	63	82	832	-4	62	66	963
3	169	162	431	-4	120	126	507	5	265	278	496	-3	220	209	530
4	142	147	536	-3	64	73	845	6	121	119	863	-1	92	92	929
5	492	493	376	-1	65	70	561	7	120	123	852	0	62	67	795
6	188	193	574	0	177	162	411	h, 9, 6				1	118	124	844
7	156	168	716	2	109	104	623	-10	108	103	970	2	89	88	1073
8	89	79	1109	4	101	110	786	-8	87	73	1063	3	111	113	934
10	85	65	724	5	90	79	924	-5	145	146	604	4	101	82	981
h, 1, 6				h, 5, 6				-4	190	192	502	h, 15, 6			
-10	146	127	729	-9	83	93	1097	-3	103	111	716	0	87	99	1139
-8	192	178	541	-7	155	158	587	1	285	289	408	1	65	70	917
-7	90	99	853	-4	109	116	545	2	69	66	942	2	94	110	1130
-5	265	272	368	-3	131	130	494	5	83	75	634	h, 16, 6			
-4	320	344	335	-2	199	203	370	6	198	202	629	-4	129	137	916
-3	85	85	644	0	44	51	823	7	79	28	1141	0	96	103	1113
-2	152	150	417	1	66	51	537	h, 10, 6				h, 1, 7			
1	538	531	280	2	280	277	371	-8	91	61	1016	-11	90	71	1164
3	112	119	562	3	122	121	589	-7	84	78	1034	-10	158	142	740
5	94	90	819	5	91	72	876	-4	72	75	942	-9	121	105	828
6	309	302	463	7	174	179	705	-2	120	125	615	-8	123	128	793
7	112	126	906	h, 6, 6				-1	50	64	1000	-6	170	170	523
10	99	105	1277	-8	248	243	475	0	115	108	749	-4	311	365	461
h, 2, 6				-7	89	103	918	3	116	120	771	-3	106	93	578
-9	97	20	946	-5	115	120	590	4	95	93	899	-2	79	89	765
-8	226	216	513	-4	114	122	590	h, 11, 6				-1	62	74	865
-5	67	72	749	-3	444	442	316	-8	134	113	842	0	298	291	323
-4	126	134	525	-1	78	91	706	-6	114	120	852	1	439	444	315
-3	242	256	351	0	72	65	796	-5	101	99	853	3	76	60	811
-1	44	54	871	1	218	219	411	-4	116	116	711	5	328	343	447
0	135	130	457	2	260	273	390	-3	207	219	506	6	183	193	612
1	258	246	343	3	98	116	757	-1	151	148	586	7	72	70	982
2	225	218	383	4	106	115	768	1	278	279	462	9	90	90	1208
3	179	185	435	6	117	123	876	2	143	147	688	10	140	154	973
4	134	140	597	8	100	101	1085	3	92	85	915	h, 2, 7			
6	77	82	937	h, 7, 6				5	163	170	680	-10	147	146	755
h, 3, 6				-10	89	94	1173	6	203	210	629	-9	261	250	502

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Columns are h kF$_o$ F$_c$ 10σ, * for Insignificant

h	kF$_o$	F$_c$	10σ	h	kF$_o$	F$_c$	10σ	h	kF$_o$	F$_c$	10σ	h	kF$_o$	F$_c$	10σ
h, 2, 7				h, -6, 7				1	81	84	987	-6	148	171	674
-5	398	425	362	-10	64	63	1106	2	173	180	582	-4	278	307	431
-4	168	187	467	-9	88	77	1005	3	110	108	810	-3	73	72	849
-3	234	240	376	-5	158	166	515	4	156	153	623	-1	143	152	486
-1	153	155	422	-4	100	97	640	5	162	167	678	0	295	287	360
0	557	548	301	-3	109	104	649	6	208	224	661	1	222	218	404
1	109	116	580	-2	84	80	675	h, 11, 7				2	160	166	484
2	194	191	416	0	292	292	371	-5	85	89	1045	5	349	359	443
3	134	130	540	1	85	89	802	-4	190	188	546	h, 2, 8			
4	189	188	515	5	87	71	847	-3	88	81	901	-11	79	81	1051
5	232	240	506	6	99	94	975	1	215	220	537	-6	252	272	455
6	242	251	530	h, 7, 7				2	119	120	753	-4	114	136	703
8	71	64	911	-10	77	81	935	5	106	101	982	-2	158	158	485
9	58	70	1130	-6	78	78	841	h, 12, 7				-1	169	173	451
h, 3, 7				-4	162	164	518	-8	106	80	872	0	161	162	483
-10	94	100	1097	-3	108	99	652	-7	102	109	1027	1	71	70	906
-9	100	117	1028	0	172	183	504	-6	133	121	754	3	148	163	572
-7	76	47	1012	1	174	167	490	-5	78	63	1120	5	95	90	950
-5	144	155	540	2	81	75	865	-4	92	101	1001	9	76	78	1280
-4	238	256	386	4	106	111	813	-1	195	194	573	h, 3, 8			
-3	120	122	534	5	168	172	635	4	125	139	850	-11	78	62	833
-1	116	129	529	6	125	115	802	h, 13, 7				-9	83	60	1092
1	311	302	347	8	91	69	931	-6	122	105	843	-7	136	137	672
2	247	244	395	9	85	80	1030	-3	113	123	873	-5	133	165	708
4	125	126	658	h, 8, 7				-2	86	81	1008	-2	73	77	798
5	80	82	1060	-8	100	91	922	0	127	130	734	-1	182	191	443
6	68	79	787	-6	111	108	747	1	96	91	943	0	364	358	358
7	136	143	820	-3	133	123	576	2	87	111	1117	1	157	158	490
10	92	87	1000	-2	94	106	751	5	121	117	878	4	275	281	468
h, 4, 7				1	74	84	1005	6	71	73	1282	5	116	114	798
-8	196	194	550	2	141	141	593	h, 14, 7				8	92	85	822
-6	229	230	438	6	121	81	767	-6	88	51	1075	9	144	153	922
-5	88	105	775	h, 9, 7				h, 15, 7				h, 4, 8			
-4	108	117	636	-9	93	88	1135	0	58	58	1074	-5	125	120	642
-3	204	210	403	-8	116	121	899	h, 16, 7				-3	67	84	886
-1	367	379	324	-6	121	123	745	-3	96	90	1105	-1	72	65	896
1	257	248	379	-5	102	100	855	h, 0, 8				0	154	141	512
2	61	53	921	-4	335	343	410	-10	280	270	557	4	54	59	950
4	159	151	590	-3	74	90	1006	-9	178	166	649	h, 5, 8			
5	106	107	832	-2	127	122	603	-8	127	129	756	-10	58	59	1022
6	108	101	849	-1	161	166	520	-6	118	141	817	-7	103	97	833
h, 5, 7				0	160	162	558	-5	550	566	367	-6	75	78	1041
-11	67	57	1005	1	334	353	407	-4	126	124	584	-5	143	148	583
-9	82	76	1128	2	113	107	708	-3	120	130	596	-2	50	40	884
-7	64	8	877	3	81	96	1040	-1	323	315	344	-1	267	276	392
-6	67	67	812	5	267	269	518	0	363	366	335	0	148	137	555
-5	89	88	739	6	136	135	844	1	312	305	354	3	82	88	1023
-4	84	95	820	7	100	95	1004	3	190	193	466	4	289	285	481
-3	164	170	475	h, 10, 7				4	312	322	416	7	68	59	1254
-1	123	119	517	-9	228	207	603	5	70	63	838	8	85	96	1036
0	175	170	466	-8	90	69	1081	6	298	313	496	9	112	112	1158
1	67	45	936	-5	341	345	440	9	86	79	1243	h, 6, 8			
2	244	240	428	-4	138	145	671	h, 1, 8				-8	79	71	1088
4	173	167	556	-3	176	168	521	-11	79	72	1166	-7	83	89	1044
7	90	116	1184	-1	180	187	516	-10	85	80	1043	-6	250	255	458
9	116	113	1051	0	394	405	406	-9	182	172	647	-5	79	76	637

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are $1F_o$ $1F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 6, 8				h, 11, 8				-1	278	282	395	-2	114	111	679
-4	83	86	867	-7	108	116	995	0	143	147	515	-1	248	263	442
-3	96	97	736	-6	102	88	948	1	199	186	445	0	228	223	484
-2	200	207	446	-5	86	76	1079	2	122	123	620	1	102	95	782
-1	229	241	433	-4	195	200	586	4	133	133	717	3	200	197	582
0	218	219	456	-2	101	115	877	5	81	81	1142	4	209	206	561
1	148	154	573	0	231	239	521	6	120	115	830	7	86	88	1244
2	80	82	946	1	145	160	693	h, 3, 9				8	116	138	1135
3	199	200	542	4	138	143	770	-9	100	84	988	h, 8, 9			
5	142	137	714	5	183	183	706	-7	75	83	723	-8	68	66	913
6	87	86	845	6	66	54	882	-6	271	280	467	-7	99	84	936
9	109	105	1168	h, 12, 8				-4	120	132	652	-5	128	116	881
h, 7, 8				-6	108	76	922	-1	310	314	388	-3	84	99	995
-10	90	91	1263	-5	194	198	610	0	257	245	422	-2	179	182	519
-9	135	133	812	-3	91	86	989	3	145	143	658	0	95	97	910
-6	143	132	625	-1	103	80	869	4	160	155	628	1	98	97	865
-4	220	231	460	0	159	160	674	5	199	202	581	2	118	120	801
-3	81	97	951	1	101	106	947	8	126	115	861	3	79	66	1123
-1	270	280	404	4	107	116	1011	h, 4, 9				4	125	120	796
0	151	155	576	6	101	102	972	-7	268	269	491	6	86	60	1114
1	126	135	667	h, 13, 8				-6	238	242	493	h, 9, 9			
2	168	167	542	-5	66	80	811	-5	154	161	598	-9	126	117	942
4	92	105	993	-1	121	113	802	-4	80	70	525	-6	87	99	1120
5	207	223	598	4	140	141	826	-3	98	98	763	-5	136	130	743
h, 8, 8				h, 14, 8				-2	346	365	390	-4	126	116	684
-9	90	88	1139	-6	167	162	732	0	217	221	476	0	251	258	493
-8	85	81	1078	-2	101	86	779	1	160	167	561	2	62	48	1181
-6	99	99	899	-1	145	141	681	2	119	117	705	4	63	61	844
-5	321	328	428	0	105	102	914	3	307	311	460	5	184	187	639
-4	79	74	828	3	121	123	868	4	260	264	507	h, 10, 9			
-3	107	102	718	h, 15, 8				5	83	78	882	-7	82	64	1050
-2	100	100	752	-3	97	68	995	9	182	179	847	-6	126	127	861
-1	196	205	482	-1	112	108	879	h, 5, 9				-5	212	218	565
0	274	284	436	2	76	77	1111	-7	80	84	995	-4	138	130	694
1	260	268	459	h, 16, 8				-6	202	208	538	-1	203	212	525
3	81	101	1024	-2	74	63	860	-1	360	361	388	0	77	73	1094
4	144	150	707	1	123	121	916	0	79	74	509	1	155	159	635
6	191	202	630	h, 1, 9				3	169	170	633	2	90	95	991
h, 9, 8				-9	121	117	888	4	158	158	698	4	120	131	887
-9	89	109	1321	-5	206	209	507	5	90	102	1141	6	70	80	886
-6	90	61	949	-4	117	126	691	8	122	127	1093	h, 11, 9			
-5	95	92	803	-3	87	87	806	h, 6, 9				-6	139	134	801
-4	178	184	579	-2	72	73	855	-10	119	92	928	-5	72	70	850
-3	90	100	866	-1	131	134	542	-9	80	58	1160	-4	56	53	970
0	218	224	501	0	330	323	371	-4	95	92	823	-2	77	60	1019
1	130	128	678	3	132	140	679	-3	71	70	1019	-1	87	96	1006
3	110	102	759	4	185	183	576	-2	82	88	894	0	150	162	694
5	186	202	640	5	179	183	605	-1	120	119	665	5	162	162	748
h, 10, 8				8	93	95	1103	1	151	146	642	h, 12, 9			
-7	63	60	880	9	96	92	1195	2	105	111	861	-7	107	101	992
-6	79	80	977	h, 2, 9				5	75	77	833	-6	131	157	962
-4	74	76	881	-10	198	180	625	h, 7, 9				-5	95	67	1043
-3	93	98	916	-6	138	172	782	-8	96	80	976	-2	186	188	610
-1	107	112	822	-5	223	249	505	-6	70	101	1205	0	92	90	1018
2	79	94	1097	-4	123	142	672	-5	148	163	657	3	152	158	759
5	82	41	1127	-2	78	88	923	-3	123	115	656	4	89	101	1232

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.

Columns are kF_o F_c σ * for Insignificant

h	kF_o	F_c	σ
h, 13, 9			
-6	94	82	992
-5	70	58	740
-1	130	131	777
4	146	143	804
h, 14, 9			
-3	66	64	1058
0	64	61	1022
2	67	65	939
h, 15, 9			
-3	72	66	1264
-2	82	58	1167
-1	158	156	729
h, 0, 10			
-10	141	118	838
-8	140	126	780
-6	178	187	630
-5	111	110	796
-4	96	91	828
-3	222	225	475
-1	308	307	401
2	101	97	753
3	89	78	865
4	136	145	686
5	133	148	800
8	88	35	1102
h, 1, 10			
-6	142	146	716
-5	202	212	543
-3	133	129	626
0	226	220	459
2	93	91	810
5	148	150	722
h, 2, 10			
-7	211	221	579
-5	118	131	765
-4	89	75	847
-3	98	110	820
-2	268	275	440
-1	185	185	494
0	74	64	829
1	106	110	728
2	121	130	731
3	89	90	818
4	220	214	533
8	59	61	1135
h, 3, 10			
-8	99	79	933
-6	186	209	652
-2	248	249	444
-1	238	236	448
2	119	116	731
3	261	252	493
4	113	106	827
7	122	112	935
8	128	131	1004
h, 4, 10			
-1	79	102	1105
h, 5, 10			
-8	87	95	963
-6	253	260	536
-4	96	104	828
-2	273	274	462
-1	300	309	442
0	117	126	779
3	342	346	481
4	80	81	707
5	116	106	884
7	95	101	946
8	147	153	991
h, 6, 10			
-7	260	261	534
-5	135	143	713
-4	65	84	778
-3	207	221	515
-2	336	350	426
-1	220	215	493
0	116	114	749
1	170	164	639
2	207	206	586
3	90	85	703
4	308	293	519
6	84	83	1219
h, 7, 10			
-6	152	157	715
-5	104	120	927
-2	90	95	941
-1	151	165	642
0	221	224	549
3	130	145	833
5	105	108	999
h, 8, 10			
-8	112	112	968
-6	130	125	766
-3	163	170	614
-1	157	161	634
0	81	75	1091
2	124	107	792
4	72	75	939
5	92	97	1099
h, 9, 10			
-5	124	128	864
-2	95	106	883
-1	87	91	987
0	149	137	648
1	81	77	1034
2	144	139	708
3	62	70	1071
4	79	71	1058
h, 10, 10			
-2	96	76	847
h, 11, 10			
-5	68	68	852
-2	86	85	1006
-1	59	67	789
0	92	85	1005
2	67	67	738
3	93	94	1071
4	102	94	1042
h, 12, 10			
-5	87	88	1161
-1	89	94	1044
4	68	76	967
h, 13, 10			
-4	66	80	866
-2	91	108	1163
-1	137	154	805
0	150	131	664
3	168	171	779
h, 14, 10			
-3	105	96	878
-2	176	177	703
-1	127	131	879
1	94	84	1081
2	92	87	1198
h, 15, 10			
-1	78	82	1222
h, 1, 11			
-7	90	92	724
-3	194	192	548
-2	145	140	614
-1	89	77	601
2	224	229	498
6	98	95	1061
7	108	126	1122
h, 2, 11			
-8	129	133	854
-6	111	115	887
-4	105	114	883
-3	94	96	865
-2	76	76	911
-1	152	158	633
1	107	117	821
2	86	95	1053
7	119	109	900
h, 3, 11			
-8	106	110	1027
-7	102	111	993
-6	156	162	696
-5	96	109	969
-3	120	118	741
-2	271	270	486
-1	61	57	827
0	187	182	535
3	294	292	493
4	89	89	963
5	76	80	702
8	119	98	1012
h, 4, 11			
-8	124	129	916
-7	250	258	563
-6	149	154	716
-4	116	115	773
-3	272	276	496
-2	172	174	592
-1	323	333	453
0	80	79	1024
1	161	162	616
2	242	242	531
3	116	111	897
4	251	250	563
5	94	100	661
h, 5, 11			
-8	103	76	961
-7	120	135	897
-6	121	124	863
-5	75	71	957
-2	273	273	479
-1	104	90	857
0	128	125	719
3	278	274	538
4	72	79	1199
6	63	70	1117
7	91	86	1285
h, 6, 11			
-6	82	69	1092
-4	78	66	1046
-3	84	78	1098
0	92	79	998
1	107	110	960
h, 7, 11			
-9	114	104	999
-7	94	91	597
-6	139	134	733
-3	113	119	807
-2	243	262	521
-1	120	123	751
2	216	212	599
3	134	140	850
6	93	80	1127
7	122	131	1048
h, 8, 11			
-8	85	70	685
-7	122	104	885
-3	108	93	814
-2	100	85	828
-1	87	92	1070
1	94	89	842
2	90	88	1118
4	110	116	938
h, 9, 11			
-3	132	127	729
2	128	143	872
3	70	66	916

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ	h	kF_o	F_c	σ
h, 10, 11				-4	77	75	1047	1	162	150	695	3	189	185	646
-6	84	92	1048	-3	184	185	622	2	180	182	701	h, 5, 13			
-4	76	94	1077	-1	127	130	704	4	59	41	946	-7	126	132	902
-3	93	77	949	0	111	106	774	h, 9, 12				-5	69	65	779
-2	72	79	1098	3	126	132	787	-3	147	146	738	-3	87	75	774
-1	106	108	846	h, 3, 12				1	126	120	862	-2	126	120	859
1	132	114	734	-7	131	144	883	2	116	112	978	0	90	91	1150
4	79	71	767	-4	85	78	1086	h, 10, 12				2	135	126	856
h, 11, 11				-3	178	165	614	-5	58	66	980	5	88	86	981
-6	87	69	1175	-2	202	196	590	-2	81	65	1149	h, 6, 13			
-5	93	97	1150	-1	70	68	875	h, 11, 12				-4	71	77	1117
-3	129	135	815	1	154	148	665	-4	85	87	1211	-3	102	83	981
-2	110	116	891	2	187	183	609	-3	140	140	815	2	118	112	952
0	79	90	1177	3	71	75	802	-1	89	67	1029	h, 7, 13			
3	103	107	1027	5	101	90	1001	1	148	147	775	-4	115	122	897
4	89	80	1197	6	137	127	866	2	107	100	984	-3	127	104	769
h, 12, 11				h, 4, 12				h, 12, 12				0	91	63	1078
-6	95	91	853	-5	76	50	1118	-4	88	78	1216	1	172	154	732
-3	85	99	1239	-1	81	73	1073	-1	96	114	1084	2	102	94	1109
-1	167	163	665	2	98	88	967	2	116	108	988	h, 8, 13			
2	93	71	1118	h, 5, 12				h, 13, 12				-3	89	91	1155
4	105	107	1128	-7	149	166	786	-2	174	171	718	h, 9, 13			
h, 13, 11				-5	80	80	975	0	59	57	1087	-4	149	159	779
-2	128	128	857	-3	92	100	1079	h, 1, 13				-3	100	104	1105
1	77	86	891	-2	264	260	547	-8	155	144	840	-2	122	134	875
h, 14, 11				1	57	58	890	-4	226	224	604	1	193	204	703
-1	68	63	1088	2	161	157	722	-3	157	152	734	3	99	98	1091
0	85	71	1233	3	174	173	716	0	88	98	1038	h, 10, 13			
h, 0, 12				4	82	76	955	1	245	248	580	-5	112	108	986
-9	190	172	689	6	79	55	1094	5	120	126	978	-4	121	135	962
-8	81	85	858	7	81	101	1407	6	120	129	1050	-3	184	187	684
-7	116	110	907	h, 6, 12				h, 2, 13				-1	97	100	1063
-6	103	100	987	-8	166	141	732	-9	149	144	872	0	101	97	1066
-5	82	69	995	-7	93	107	1181	-6	82	76	899	2	190	181	687
-4	292	282	486	-6	122	133	838	-5	146	152	799	h, 11, 13			
-3	112	105	794	-5	84	69	1040	-4	159	162	714	-2	118	118	920
-2	81	66	979	-4	89	89	886	-3	195	198	624	-1	91	91	1147
-1	147	157	655	-3	200	210	607	-1	128	126	767	1	109	98	972
0	88	87	985	-1	172	168	667	0	122	115	789	2	56	61	1048
1	128	139	734	0	118	124	897	2	234	224	571	h, 12, 13			
2	286	286	493	1	66	55	998	3	69	82	1084	-2	113	119	1045
7	175	192	808	2	143	135	805	6	134	126	900	0	71	69	1063
h, 1, 12				3	177	173	736	h, 3, 13				h, 0, 14			
-8	148	150	829	4	87	81	1066	-7	135	147	905	-8	138	131	998
-4	74	67	732	5	73	63	1292	-4	100	91	884	-6	119	120	963
-3	204	207	579	h, 7, 12				-2	183	191	653	-5	227	214	622
-2	202	205	546	-8	107	110	1071	1	131	113	794	-3	305	293	529
-1	67	61	633	-7	89	94	1174	2	134	127	803	-2	135	127	752
1	174	171	620	-3	75	76	942	3	84	79	1163	-1	121	113	793
3	128	127	762	-2	182	178	606	h, 4, 13				0	158	155	662
4	107	110	868	1	105	91	954	-8	143	143	833	1	213	216	627
6	132	126	873	3	161	167	766	-5	115	114	893	2	178	166	656
h, 2, 12				4	84	77	1082	-3	162	156	701	3	122	136	898
-8	114	121	1001	h, 8, 12				-2	121	123	852	6	188	191	746
-7	96	73	1019	-8	83	81	910	0	88	86	1044	h, 1, 14			
-6	111	121	972	-4	164	176	674	2	84	70	1176	-7	90	83	1261

Table D.2. Obs and Calcd Structure factors for $Os_4(CO)_{13}(PMe_3)[P(OMe)_3]$.
Columns are lF_o lF_c 100σ, * for Insignificant

h	kF$_o$	F$_c$	σ	h	kF$_o$	F$_c$	σ	h	kF$_o$	F$_c$	σ	h	kF$_o$	F$_c$	σ
h, 1, 14				h, 8, 14				1	197	187	672	2	76	69	1228
-4	238	234	612	-6	66	58	980	3	92	75	1201	h, 1, 16			
-2	116	119	855	-5	136	131	837	h, 3, 15				-5	85	78	1165
0	96	89	938	-3	160	171	804	-4	125	121	922	-4	95	87	1225
1	208	208	639	-2	107	103	1007	-1	87	90	729	-3	83	84	861
2	119	128	921	-1	91	75	1114	1	98	92	1081	0	167	155	745
4	66	59	1030	0	92	96	1209	2	116	116	935	h, 2, 16			
5	90	91	1273	1	132	134	922	h, 4, 15				-5	119	93	980
h, 2, 14				2	125	110	946	-6	124	126	1000	0	107	113	1111
-3	62	58	1212	3	104	106	1155	-5	120	90	913	h, 3, 16			
-1	105	90	916	h, 9, 14				-1	84	61	1135	-6	114	115	1204
0	94	72	714	-4	145	142	852	0	201	176	638	-1	163	160	771
h, 3, 14				-1	82	70	1224	h, 5, 15				3	87	94	1020
-8	77	76	1295	0	90	89	1240	-5	94	73	1155	h, 5, 16			
-5	119	115	944	1	120	115	997	-1	124	111	889	-4	98	75	1163
-4	128	130	867	2	72	64	1295	2	106	100	1033	-2	108	82	1034
-3	90	67	1101	h, 10, 14				4	97	100	1334	-1	140	137	891
0	173	169	672	-1	97	86	947	h, 6, 15				h, 6, 16			
1	72	71	1163	h, 11, 14				-2	79	56	1121	-3	90	74	1254
4	109	93	1032	-2	98	87	1158	1	91	79	1239	-1	101	81	969
5	118	113	979	0	135	134	939	h, 7, 15				0	145	123	911
h, 4, 14				h, 1, 15				-5	110	102	1078	1	99	100	1210
1	63	47	1022	-5	128	128	961	-1	126	123	951	h, 7, 16			
h, 5, 14				-4	164	156	766	0	88	89	1021	-3	105	87	1165
-5	84	83	1054	-3	72	77	1232	3	97	87	1269	0	128	112	1029
-3	94	73	1055	-1	98	95	1056	h, 8, 15				h, 1, 17			
0	117	111	909	0	171	165	743	-3	94	80	1154	-2	88	89	944
2	85	89	1156	2	84	72	1175	0	98	81	1199	h, 2, 17			
4	70	49	1157	3	74	63	795	h, 9, 15				1	94	54	1125
h, 6, 14				4	78	91	1263	-3	94	88	1244	h, 3, 17			
-3	82	77	1171	h, 2, 15				0	135	125	940	-1	76	71	947
h, 7, 14				-6	65	75	1145	h, 0, 16				0	158	135	816
-4	137	147	833	-5	127	134	948	-6	75	65	1123	h, 4, 17			
-2	104	87	998	-4	130	97	843	-5	73	70	891	-1	171	171	816
1	122	128	984	-3	111	100	1001	-4	155	142	838	h, 5, 17			
2	116	134	1073	-2	94	102	1154	1	185	172	714	-2	118	90	1047

Table E.1. U_{ij} or U_{iso} Values for $Os_4(CO)_{11}(CNBu^t)$.

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Os(1)	0.0380(9)	0.0304(8)	0.0464(9)	0.0003(8)	0.0011(7)	-0.0018(7)
Os(2)	0.0398(9)	0.064(1)	0.0374(9)	0.0160(8)	-0.0013(7)	0.0031(8)
Os(3)	0.0311(8)	0.0287(8)	0.0340(8)	-0.0002(7)	0.0013(6)	0.0003(7)
Os(4)	0.0385(8)	0.0394(9)	0.0339(8)	-0.0012(7)	0.0064(6)	-0.0007(7)
O(11)	0.071(9)					
O(12)	0.075(9)					
O(13)	0.09(1)					
O(21)	0.078(9)					
O(22)	0.08(1)					
O(23)	0.081(9)					
O(24)	0.11(1)					
N(31)	0.035(7)					
O(32)	0.045(6)					
O(33)	0.077(9)					
O(34)	0.065(8)					
O(41)	0.067(8)					
O(42)	0.064(8)					
O(43)	0.11(1)					
O(44)	0.09(1)					
C(11)	0.05(1)					
C(12)	0.05(1)					
C(13)	0.06(1)					
C(21)	0.07(1)					
C(22)	0.06(1)					
C(23)	0.08(1)					
C(24)	0.11(2)					
C(31)	0.05(1)					
C(32)	0.05(1)					
C(33)	0.05(1)					
C(34)	0.026(7)					
C(41)	0.035(8)					
C(42)	0.06(1)					
C(43)	0.07(1)					
C(44)	0.08(1)					
C(1)	0.040(9)					
C(2)	0.10(1)					
C(3)	0.10(1)					
C(4)	0.10(1)					

Table E.2. Hydrogen Atom Coordinates for $Os_4(CO)_{14}(CNBu^t)$.

Atom	*x/a*	*y/b*	*z/c*	U(iso)
H(1)	0.684(5)	0.082(3)	0.281(4)	0.1000
H(2)	0.578(5)	0.116(3)	0.335(4)	0.1000
H(3)	0.597(5)	0.130(3)	0.213(4)	0.1000
H(4)	0.647(4)	-0.020(3)	0.141(4)	0.1000
H(5)	0.561(4)	0.035(3)	0.081(4)	0.1000
H(6)	0.520(4)	-0.044(3)	0.117(4)	0.1000
H(7)	0.635(5)	-0.039(3)	0.340(4)	0.1000
H(8)	0.534(5)	-0.002(3)	0.399(4)	0.1000
H(9)	0.510(5)	-0.068(3)	0.319(4)	0.1000

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_O/$	$/F_C/$	PHI
** K= 0 L= 0 **			
2	3619	3577	187
4	9099	8985	185
6	7253	7495	7
10	3795	3765	189
12	2289	2100	9
** K= 1 L= 0 **			
1	3345	3242	6
2	7419	7452	187
3	5382	5450	186
4	6999	6795	5
6	2010	1921	8
7	3943	3965	6
8	5037	4993	187
9	1244	1366	191
10	1467	1420	9
11	880	1080	188
12	1690	1533	10
13	1317	1286	10
** K= 2 L= 0 **			
0	4104	3958	186
1	6048	6087	186
2	2003	2035	6
3	1578	1587	187
4	1320	1273	9
5	5246	5328	7
6	2721	2738	187
7	1941	2020	187
8	1225	1238	7
9	2057	2051	190
11	2399	2307	9
12	1364	1334	189
** K= 3 L= 0 **			
1	2664	2843	186
2	551	342	186
3	3124	3082	10
4	837	821	186
5	783	842	187
6	1218	1254	8
7	2316	2310	188
9	1601	1706	9
10	1118	1073	190
12	1059	963	10
13	1098	951	191
** K= 4 L= 0 **			
1	1896	1850	6
4	664	760	9
5	1247	1244	187
7	1213	1181	8
8	1139	1133	189
13	796	588	11
** K= 5 L= 0 **			
3	1617	1500	184
4	772	856	9
5	915	957	8
8	586	613	189
9	703	715	190
11	840	791	10
** K= 6 L= 0 **			
0	1681	1528	189
1	2091	1961	185
4	822	776	10
5	1268	1249	7
6	1293	1217	189
7	1387	1331	188
11	927	780	9
** K= 7 L= 0 **			
1	1228	1317	187
2	890	1062	6
3	2174	2162	8
4	542	651	187
5	793	864	188
7	1023	955	190
9	1364	1292	10
** K= 8 L= 0 **			
1	1348	1349	9
5	1411	1332	188
7	1138	1199	8
** K= 10 L= 0 **			
0	1326	1171	187
** K= 11 L= 0 **			
2	1623	1531	9
3	1170	949	9
4	867	1027	188
6	858	879	188
8	1316	1093	10
9	946	552	8
** K= 12 L= 0 **			
0	1320	1283	7
1	2553	2395	7
5	2179	2084	189
6	744	703	9
** K= 13 L= 0 **			
1	1160	1536	8
3	2490	2348	188
7	1743	1537	9
** K= 14 L= 0 **			
0	1881	1930	8
4	1637	1577	188
6	916	1121	9
10	972	988	191
** K= 15 L= 0 **			
1	1160	858	8
3	1162	946	190
7	1098	1006	10
** K= 16 L= 0 **			
0	1126	1132	8
5	1494	1248	10
9	902	940	191
** K= 17 L= 0 **			
2	2084	1974	190

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
4	1282	1199	11
8	1451	1375	191
** K= 18 L= 0 **			
0	2054	1987	190
1	1248	1388	190
4	1209	1253	11
6	1522	1322	190
** K= 19 L= 0 **			
3	2072	2032	11
** K= 20 L= 0 **			
1	1450	1490	10
** K= 0 L= 1 **			
-11	1961	1917	189
-9	1053	1122	10
-7	2687	2674	7
-5	5162	5113	187
-1	7394	6820	6
1	6050	6325	186
3	963	1120	191
5	6465	6354	7
7	2703	2676	187
9	2138	2226	189
11	3050	2892	9
** K= 1 L= 1 **			
-12	987	919	189
-9	1647	1763	9
-8	1662	1674	8
-7	1991	2041	187
-6	1303	1340	188
-5	1636	1644	186
-4	1023	1018	191
-3	4450	4286	7
-2	3215	3024	7
-1	2689	2576	186
0	723	325	184
1	3535	3724	186
2	3191	3159	187
3	4658	4544	8
4	1801	1854	8
5	734	699	187
6	1396	1407	7
7	3458	3392	188
8	2410	2378	188
9	2029	2057	10
12	1321	1274	9
13	1535	1469	191
** K= 2 L= 1 **			
-13	879	833	190
-10	1160	1131	10
-9	933	683	10
-8	751	668	187
-7	1746	1765	187
-6	1548	1568	189
-5	613	540	11
-4	2683	2531	7
-3	623	640	11
-2	563	422	15
-1	2253	2314	186
0	4834	4836	185
1	1087	1099	7
2	1158	1068	11
3	858	786	6
4	2905	2830	7
5	1074	1079	191
6	2806	2811	188
7	1219	1213	7
10	1808	1864	10
11	857	824	190
12	944	920	190
** K= 3 L= 1 **			
-11	1575	1431	10
-9	758	715	190
-7	1355	1236	188
-5	1662	1692	9
-4	559	484	11
-1	1326	1307	188
1	1143	1141	6
2	884	753	17
4	1478	1437	188
6	605	582	188
8	1391	1330	10
10	761	608	189
11	821	582	9
12	567	601	191
** K= 4 L= 1 **			
-13	1249	1101	11
-11	828	811	189
-10	875	894	11
-9	1042	927	191
-8	825	1006	189
-7	2071	2142	8
-5	579	608	185
-4	1578	1480	7
-3	1719	1671	189
-2	601	588	189
-1	1871	1883	7
0	853	1042	182
3	1163	1244	188
5	623	575	5
7	688	470	5
** K= 5 L= 1 **			
-13	1173	943	190
-12	1109	1128	10
-10	1037	1220	190
-9	1980	1977	10
-7	1223	1273	187
-6	2286	2367	8
-5	2093	2059	188
-4	1514	1515	187
-3	2563	2646	9
-2	759	854	191
0	3210	3160	5
1	2154	2284	189
3	1673	1862	7
4	1469	1441	186
7	1271	1475	188
** K= 6 L= 1 **			
-12	1428	1392	190
-11	1701	1603	10
-9	1080	1017	189
-8	2605	2591	8
-7	1656	1720	188
-6	2404	2439	187
-5	3061	3031	8

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-4	1389	1394	187
-3	772	814	183
-2	3721	3679	8
-1	3388	3432	186
0	1705	1724	187
1	2589	2687	7
2	1570	1636	191
4	2515	2741	8
5	1840	1948	189
7	1280	1111	6
8	1563	1440	189
10	951	846	10
11	888	830	189
** K=	7	L=	1 **
-11	770	569	190
-10	1739	1814	11
-8	2010	1967	189
-7	1822	1759	8
-6	1126	1262	189
-5	886	931	187
-4	4187	4253	8
-3	1105	1187	189
-2	1753	1839	186
-1	3242	3236	6
0	3962	3902	186
1	835	904	184
2	3757	3709	7
3	2009	1955	187
5	2085	2008	7
6	2867	2926	188
8	1385	1322	9
9	1082	1015	191
12	1153	1084	191
** K=	8	L=	1 **
-12	1150	1167	10
-9	959	901	10
-8	1220	1223	190
-7	1514	1608	188
-6	2436	2443	8
-3	2479	2451	9
-2	2602	2632	188
-1	1523	1843	187
0	2458	2598	7
1	1773	1695	189
3	3167	3229	8
4	2379	2563	187
6	1186	1111	7
7	2187	2317	188
8	974	847	9
9	1667	1654	10
10	1115	972	189
** K=	9	L=	1 **
-11	895	876	10
-9	1153	1065	190
-7	856	861	188
-5	2582	2656	8
-3	979	1083	187
-2	662	873	5
-1	2885	2833	188
1	3736	3743	7
5	3207	3280	188
7	1696	1605	9
9	1030	1105	11
11	1648	1671	190
** K=	10	L=	1 **
-9	1041	1084	192
-7	1261	1226	8
-6	799	763	8
-5	1085	1048	10
-4	767	763	8
-3	2810	2856	187
-2	1392	1482	189
1	2782	2885	7
2	1988	2005	8
3	1942	1869	190
4	1748	1711	187
6	963	957	187
7	1918	1962	9
8	1888	1899	9
** K=	11	L=	1 **
-11	749	789	190
-7	1895	1994	8
-6	1619	1745	8
-4	1107	1140	188
-3	1861	1899	188
-2	1628	1583	188
-1	1822	1915	9
0	3236	3208	7
3	1646	1715	190
4	2536	2487	188
5	1171	1092	8
6	1766	1767	9
9	1005	992	190
10	1502	1402	191
** K=	12	L=	1 **
-11	969	1006	190
-10	1337	1269	190
-8	917	1132	11
-7	1078	1028	8
-6	906	1035	9
-5	1466	1488	189
-4	2875	2875	188
-1	1429	1288	10
0	1455	2480	8
1	1537	1380	187
2	2970	2919	188
5	1240	1240	9
6	2301	2350	9
8	1403	1448	189
** K=	13	L=	1 **
-8	1427	1605	11
-6	1700	1824	189
-5	1119	992	189
-4	1027	945	189
-2	2952	2957	9
2	1914	2023	188
4	2479	2466	9
8	1289	1496	190
10	846	879	11
** K=	14	L=	1 **
-10	881	812	11
-9	990	1060	10
-6	1058	1189	189
-5	1270	1092	189
-4	1339	1229	9
-3	1176	1239	10
0	1181	1515	189
1	1484	1529	189

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
5	1322	1123	9
7	949	839	189
** K= 15 L= 1 **			
-7	879	803	191
-5	920	833	9
-3	638	517	10
-1	1269	1278	188
** K= 17 L= 1 **			
-3	1094	1095	11
-1	684	630	190
3	1438	1290	10
7	1366	1181	191
** K= 18 L= 1 **			
-6	1397	1299	191
0	1771	1515	191
4	1016	1039	10
5	1194	1051	191
** K= 19 L= 1 **			
-4	1191	1095	11
-2	1705	1715	190
2	1756	1656	10
4	1410	1152	191
** K= 20 L= 1 **			
-4	912	866	190
0	1338	1280	10
** K= 0 L= 2 **			
-8	614	531	6
0	1166	1141	185
** K= 1 L= 2 **			
-6	599	521	186
-1	647	689	360
** K= 2 L= 2 **			
-10	760	750	9
-6	814	695	187
-5	720	815	187
-4	1020	919	6
-1	1094	1076	5
0	1432	1466	184
1	900	955	184
4	710	747	8
8	830	687	189
** K= 3 L= 2 **			
-13	1014	1041	190
-12	711	543	9
-9	1411	1394	10
-7	1843	1977	188
-6	749	621	8
-5	1051	1057	185
-3	2591	2709	8
-2	606	655	182
-1	713	719	192
1	2036	2084	187
3	1814	1850	8
7	1587	1608	188
** K= 4 L= 2 **			
-12	1089	1091	189
-11	1795	1851	9
-10	849	900	10
-9	1430	1382	189
-8	1139	1175	6
-7	2005	1947	186
-6	2307	2368	188
-5	3482	3445	7
-2	2480	2500	6
-1	3927	3928	186
0	3602	3566	185
1	1966	2036	9
2	1139	1094	180
3	1400	1380	7
4	2012	2201	7
5	2389	2388	189
6	943	1054	190
9	1065	1061	11
11	975	917	189
** K= 5 L= 2 **			
-12	1140	1172	10
-10	1431	1409	11
-8	3759	3812	188
-7	1541	1587	8
-5	739	799	185
-4	5388	5362	6
-3	1195	1409	188
-2	3118	3142	190
-1	3174	3129	4
0	3229	3223	184
1	985	1094	2
2	4266	4252	9
3	2081	2176	186
4	1782	1710	188
6	2529	2572	188
7	873	984	6
8	2424	2527	8
9	832	691	190
12	1081	1165	190
** K= 6 L= 2 **			
-12	1720	1744	10
-11	975	885	9
-10	2144	2138	191
-8	589	480	189
-7	1755	1787	188
-6	4133	4205	8
-5	935	911	9
-4	3431	3429	187
-3	1407	1414	9
-2	2339	2375	189
-1	3060	2978	186
0	7881	7659	5
2	1069	1065	188
3	2991	3074	6
4	4411	4415	186
5	1188	1135	189
6	3081	3180	8
7	1586	1573	186
8	875	722	6
9	965	1144	11
10	1717	1814	190
13	701	695	191
** K= 7 L= 2 **			
-13	1059	1088	12

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$

H	$/F_o/$	$/F_c/$	PHI
-12	893	753	190
-9	2152	2168	190
-8	2023	2069	8
-7	1508	1619	8
-6	1312	1272	188
-5	2515	2516	8
-4	1823	1801	187
-3	3647	3713	188
-2	3103	3051	8
0	1435	1479	185
1	4106	4226	8
2	2164	2117	188
3	2430	2405	188
4	3100	3032	7
5	2033	2132	188
7	3018	3154	8
8	1404	1537	189
10	1135	1249	10
11	1590	1775	189

** K= 8 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-11	1413	1430	190
-8	943	745	188
-7	2038	2097	8
-5	2370	2440	188
-4	1420	1418	8
-3	861	805	188
-2	1906	1881	188
-1	4045	4033	6
0	684	711	185
1	1201	1190	190
2	2512	2467	10
3	2007	2174	187
4	1879	2014	187
5	2326	2315	8
6	1375	1561	188
8	2407	2534	9
9	1332	1368	190
11	873	731	9
12	1299	1343	190

** K= 9 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	570	455	9
-6	850	1042	9
-4	1697	1777	188
0	3620	3635	7
2	1426	1487	188
3	998	1091	6
4	2392	2486	188
6	2893	3070	8
9	676	613	10
10	1900	1932	191
12	1284	1281	10

** K= 10 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-7	889	821	8
-4	1617	1564	188
-3	1233	1221	188
-2	1213	1182	8
-1	1144	1173	9
0	1170	1422	6
1	967	958	7
2	2375	2336	188
3	2040	2054	189
6	1763	1648	8
7	1825	1658	9
8	1679	1821	189
9	1473	1535	190
12	1059	1126	10

** K= 11 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-8	1745	1789	9
-6	1013	1102	188
-5	927	1070	188
-4	1565	1660	188
-2	2441	2414	9
-1	1738	1768	8
1	2283	2155	187
2	2110	2176	188
3	1048	956	187
4	1864	2049	8
5	2515	2564	8
7	908	943	189
8	1366	1544	189
9	1159	1202	190
11	1178	1361	11

** K= 12 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-11	925	768	190
-10	1273	1325	11
-6	2435	2409	189
-5	1816	1859	188
-4	1733	1722	8
-3	1607	1571	7
-2	1429	1469	9
0	3240	3147	187
1	2903	2928	187
3	1186	1080	9
4	1821	1916	9
5	1464	1327	9
6	1796	1637	188
7	2141	2047	189
10	981	981	11
11	1176	963	11

** K= 13 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	1408	1426	11
-8	1114	1050	190
-7	2037	1963	189
-4	1351	1334	9
-3	2918	2761	9
-2	1302	1252	188
-1	2171	2310	188
2	1562	1449	8
3	2898	2847	9
5	1330	1175	189
7	1247	1257	190
9	1538	1458	11

** K= 14 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	649	692	192
-7	1282	1232	190
-5	2373	2317	9
-1	1803	2085	189
1	2467	2362	9
5	1694	1847	190
7	1245	1209	10

** K= 15 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	896	846	192
-7	749	888	10
-3	1581	1531	189
3	1252	1286	190
9	861	759	191

** K= 16 L= 2 **

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
-5	998	810	191
1	1287	1094	189
** K= 17	L= 2	**	
-3	1226	1229	10
3	1174	1016	11
** K= 18	L= 2	**	
-7	946	874	191
-5	1495	1328	10
-1	1483	1473	190
1	1224	1103	11
5	1054	1163	191
** K= 19	L= 2	**	
-4	998	922	11
-3	1209	1164	190
-2	781	922	190
2	1204	1017	11
3	1321	1125	190
** K= 20	L= 2	**	
0	1121	1208	11
4	958	886	191
** K= 21	L= 2	**	
1	1243	820	10
** K= 0	L= 3	**	
-11	947	926	9
-7	1052	950	188
-5	941	953	5
1	691	454	358
** K= 1	L= 3	**	
-7	457	453	4
-6	746	735	188
-4	1058	907	7
-1	731	712	3
0	1853	1728	185
1	1219	1135	185
2	1018	941	1
4	1123	956	8
5	829	831	7
6	880	896	186
7	1145	1315	187
** K= 2	L= 3	**	
-8	752	879	187
-7	814	727	190
-4	1742	1686	5
-3	1267	1248	10
-2	1563	1509	182
-1	1720	1655	187
0	1439	1508	186
1	792	765	187
2	2066	2047	5
3	2537	2464	8
5	1200	1330	187
6	1270	1473	188
7	1619	1557	187
9	1601	1525	10
** K= 3	L= 3	**	
-11	784	643	9
-9	532	528	190
-8	1577	1570	188
-7	1002	909	188
-6	961	951	4
-5	2101	1998	7
-4	1834	1818	8
-2	2111	2184	188
-1	3413	3464	185
1	2606	2579	6
2	2059	2039	11
3	1509	1471	5
4	1765	1755	185
5	3182	3196	187
6	1378	1483	190
7	1012	1082	8
8	2088	2121	8
9	1275	1293	9
11	1519	1519	189
12	1091	1227	190
** K= 4	L= 3	**	
-12	1199	1109	10
-11	751	747	10
-10	1017	1060	190
-9	1240	1219	191
-8	978	1018	189
-6	2749	2767	8
-5	2687	2662	8
-4	933	947	186
-3	2718	2676	186
-2	2577	2553	190
-1	2237	2199	185
0	4925	4944	5
1	4874	4940	5
2	1183	1274	7
3	787	1027	189
4	4189	4130	187
5	2518	2458	187
6	1172	1235	9
7	2402	2476	7
8	1999	1987	8
10	1519	1622	191
11	1122	1221	189
** K= 5	L= 3	**	
-13	1335	1354	11
-10	1142	1203	190
-9	1812	1977	190
-8	745	872	8
-7	3394	3374	8
-6	1415	1601	7
-5	1138	1025	5
-4	2840	2864	187
-3	4758	4889	187
-1	2946	2830	8
0	4006	3908	5
1	3295	3254	7
2	1725	1783	189
3	4583	4614	188
4	1032	1029	187
6	2063	2190	9
7	2910	3066	8
9	1530	1594	191
12	901	1007	10
13	886	1100	11
** K= 6	L= 3	**	

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
-13	736	551	10
-11	2089	2185	189
-10	1393	1237	189
-9	889	1024	10
-8	1168	1126	10
-7	2960	3022	7
-6	845	936	7
-5	4087	4121	188
-4	2653	2641	187
-2	539	466	14
-1	5610	5471	7
0	2072	2110	6
1	3393	3441	188
2	1582	1517	189
3	1753	1814	188
5	3917	3919	8
6	1145	1146	9
7	1388	1216	187
9	1637	1688	190
11	1641	1588	10
** K= 7 L= 3 **			
-12	1478	1404	190
-9	1494	1502	10
-8	2366	2442	9
-7	975	1013	188
-6	1778	1871	188
-5	1566	1449	189
-4	1647	1851	188
-3	2489	2617	8
-2	3255	3278	8
-1	1391	1362	184
0	720	687	185
1	2403	2487	187
2	2707	2711	189
3	2310	2325	8
4	1838	2069	8
6	1151	1168	8
7	1814	1722	188
8	1521	1651	190
9	944	969	10
12	744	874	10
** K= 8 L= 3 **			
-10	1397	1368	10
-9	741	799	11
-7	815	909	188
-6	1977	2088	188
-4	2285	2388	7
-3	713	571	11
-2	748	753	8
0	3469	3487	187
2	1250	1486	6
4	1814	1925	7
6	1850	1953	188
10	929	1017	10
** K= 9 L= 3 **			
-11	1099	1105	10
-7	1107	1178	189
-5	975	1062	8
-2	1078	1073	187
-1	1003	1007	186
4	1421	1216	187
11	721	904	10
** K= 10 L= 3 **			
-9	934	771	189
-7	1351	1382	9
-3	1021	1012	189
-1	1415	1518	6
0	707	731	187
3	1035	834	186
4	1040	1078	9
8	1150	1116	190
** K= 11 L= 3 **			
-10	945	778	190
-9	1565	1632	10
-7	1139	985	188
-6	1035	1080	8
-5	1709	1679	188
-4	655	643	187
-3	2432	2447	9
0	722	640	4
1	2245	2388	188
3	1956	1951	8
7	1626	1734	189
** K= 12 L= 3 **			
-11	1167	1091	12
-9	755	725	189
-8	1337	1386	9
-7	1420	1360	190
-6	1896	1862	189
-5	2376	2330	8
-2	2086	2091	8
-1	3024	3035	187
0	1571	1525	188
1	2054	2013	8
3	1098	1122	9
4	1375	1523	8
5	2348	2255	189
6	758	665	190
9	1115	1054	11
11	999	900	190
** K= 13 L= 3 **			
-10	1246	1300	11
-8	1667	1752	190
-7	1167	1159	10
-4	2698	2628	9
-3	1204	1155	190
-2	1793	1979	188
-1	1515	1663	8
0	1356	1388	189
2	2641	2661	9
3	1809	1777	188
4	826	855	189
6	1189	1483	189
8	1191	1324	10
** K= 14 L= 3 **			
-10	947	843	192
-7	876	843	190
-6	1819	1945	10
-4	1105	1148	190
-3	1337	1306	10
-2	1383	1411	189
0	2535	2540	9
1	1108	1007	190
3	1292	1221	10
4	1653	1624	190
6	1504	1704	10
** K= 15 L= 3 **			

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
-6	699	658	191
-5	1233	1004	10
-2	1810	1731	10
-1	936	924	189
1	1173	1184	9
2	1285	1244	190
4	1658	1640	10
5	1503	1117	188
** K= 16	L= 3 **		
-4	1308	1311	10
0	1499	1371	190
2	1475	1676	9
6	1304	1501	190
8	976	1024	10
** K= 17	L= 3 **		
-8	949	735	192
-2	1438	1485	190
2	1138	1064	10
4	1684	1430	190
** K= 18	L= 3 **		
-6	1232	1260	11
-4	971	884	191
0	1589	1427	11
** K= 19	L= 3 **		
-4	876	918	191
-3	1166	1078	191
-2	1003	816	11
1	804	824	10
2	908	904	191
** K= 20	L= 3 **		
-1	1109	1177	11
** K= 0	L= 4 **		
-12	2277	2290	9
-10	2224	2292	190
-8	1079	969	190
-6	4144	4145	9
-4	3315	3245	186
-2	1011	917	205
0	5938	6143	5
2	1744	1699	182
4	2637	2495	188
6	1793	1868	7
** K= 1	L= 4 **		
-13	871	856	11
-12	963	1087	189
-10	980	1032	192
-9	1555	1567	191
-8	3363	3320	8
-7	742	701	10
-6	1353	1321	186
-5	1945	1892	8
-4	3063	3024	187
-3	2049	2059	189
-2	3491	3360	7
-1	691	702	187
0	607	681	181
1	2751	2524	8
2	1819	1749	191
3	1040	1127	185
4	2443	2326	7
5	1416	1246	190
7	1325	1536	7
8	964	1015	189
11	1025	973	189
** K= 2	L= 4 **		
-13	763	741	11
-12	515	389	187
-11	1788	1747	190
-10	989	998	10
-8	947	898	189
-7	2669	2659	8
-6	980	922	187
-5	2178	2128	188
-4	3048	2920	7
-3	1705	1728	186
-2	1331	1325	189
-1	3967	3860	6
0	2427	2468	185
2	2623	2552	10
3	2570	2516	186
4	1472	1392	186
5	1183	1145	8
6	1629	1650	189
7	917	1038	7
8	2267	2354	9
9	828	777	191
12	1001	1179	190
** K= 3	L= 4 **		
-10	966	994	190
-9	1089	1040	9
-7	887	902	189
-6	2487	2423	8
-5	841	892	183
-4	2029	1963	187
-3	983	1062	9
-2	1707	1604	190
0	5901	5730	6
2	991	1056	186
4	4156	4101	187
6	3472	3377	8
8	856	648	9
10	2276	2390	190
12	1038	1133	10
** K= 4	L= 4 **		
-12	600	502	188
-9	764	741	191
-8	1628	1650	8
-7	1149	1085	8
-4	2670	2713	187
-3	2232	2308	188
-2	2346	2381	8
-1	1293	1331	8
0	1886	1969	5
1	1478	1549	8
2	3617	3617	188
3	2756	2699	188
4	1626	1394	7
5	784	752	6
6	2606	2530	7
7	2071	2065	8
8	2422	2465	189
9	1462	1554	190
12	1465	1472	10

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_O/$	$/F_C/$	PHI
** K= 5	L= 4 **		
-11	945	1050	189
-9	593	648	10
-8	1292	1314	9
-7	1508	1512	8
-6	1225	1238	188
-5	2786	2903	187
-4	783	796	188
-2	2238	2324	8
-1	4061	4043	6
0	1102	1199	187
1	3308	3431	186
2	1575	1656	188
3	1467	1439	187
4	2233	2238	8
5	3744	3582	8
7	1417	1400	188
8	1565	1525	188
9	1319	1478	190
10	804	684	12
11	1710	1759	10
** K= 6	L= 4 **		
-12	824	917	189
-10	733	813	11
-9	1598	1455	10
-8	771	625	8
-7	945	922	187
-6	1861	1887	189
-5	2008	2121	187
-4	964	963	9
-3	2938	3020	8
-2	1360	1381	9
0	2844	2873	186
1	3564	3711	187
3	2359	2414	8
4	1942	2031	7
5	1077	1013	7
6	1090	1205	189
7	2409	2453	189
9	904	935	9
10	1040	911	11
11	971	855	10
** K= 7	L= 4 **		
-10	886	859	10
-9	806	791	9
-7	2262	2269	188
-6	706	652	189
-5	798	871	9
-4	1635	1660	8
-3	2001	2156	8
-1	3061	3058	187
0	1525	1461	187
2	1279	1305	8
3	2418	2565	8
5	1806	1871	188
6	1185	1171	187
7	965	722	188
9	1032	1264	10
** K= 8	L= 4 **		
-11	764	780	10
-9	869	1017	189
-8	1008	944	190
-5	1893	1968	8
-4	1094	1027	9
-3	1204	1342	188
-2	911	895	190
-1	1367	1363	186
1	2362	2449	7
2	959	936	8
5	1248	1297	188
7	1635	1513	8
11	905	692	190
** K= 9	L= 4 **		
-12	874	730	10
-10	682	694	191
-6	1593	1531	8
-4	660	547	192
-3	539	625	185
-2	1191	1120	188
-1	1179	1270	7
0	1648	1522	8
3	808	856	187
4	1116	1011	188
5	1125	1255	8
** K= 10	L= 4 **		
-8	1560	1507	8
-6	943	1052	189
-4	1041	1105	188
-2	1966	2026	8
2	1640	1644	188
3	948	846	6
4	1269	1240	9
8	1088	853	191
9	1074	709	9
** K= 11	L= 4 **		
-11	827	594	192
-10	1110	998	12
-8	1359	1128	189
-6	691	784	190
-5	1117	1281	189
-4	2193	2317	8
-3	855	771	6
-1	1361	1364	5
0	2217	2187	188
1	811	824	190
2	1848	1936	8
6	1933	1914	188
** K= 12	L= 4 **		
-7	2078	2083	190
-6	909	1109	10
-5	890	906	9
-3	1521	1481	11
-2	1014	1159	188
-1	2586	2563	188
0	1422	1355	9
3	2135	2238	8
4	1373	1277	189
6	909	874	8
7	1013	947	189
9	1022	945	11
** K= 13	L= 4 **		
-9	1540	1657	190
-5	2061	1996	10
-3	1753	1892	190
0	1125	1246	189
1	2308	2335	10
3	1217	1060	188

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
5	1202	1384	189
7	1454	1400	10
** K= 14 L= 4 **			
-8	1152	1140	191
-7	902	877	10
-6	1070	1117	9
-5	860	854	189
-2	2147	2053	189
-1	1454	1457	8
2	1771	1754	11
4	1507	1423	189
5	1121	916	10
6	873	841	191
8	1535	1470	11
** K= 15 L= 4 **			
-6	1042	1088	9
-4	1386	1155	190
-1	861	1046	190
0	1835	1895	9
3	708	853	8
4	1136	1232	190
5	1291	1250	190
6	1197	1240	10
** K= 16 L= 4 **			
-3	1481	1429	189
1	1537	1328	9
3	1367	1466	191
7	1499	1491	10
** K= 17 L= 4 **			
-4	1285	1174	190
-1	997	1078	11
2	974	1013	190
** K= 18 L= 4 **			
-6	1191	880	190
-5	1146	1179	190
-2	906	867	11
-1	1163	1250	10
1	961	1079	190
** K= 19 L= 4 **			
-3	1372	1477	11
1	1324	1130	191
3	1344	1018	11
** K= 0 L= 5 **			
-13	837	1059	11
-11	3315	3357	190
-9	1196	1250	10
-7	4683	4592	8
-5	6000	5960	188
-3	699	719	187
-1	8783	8515	6
1	4220	4161	188
3	3263	3167	187
5	5043	4885	8
7	1086	1164	187
9	1763	1859	191
11	1788	1754	9
** K= 1 L= 5 **			
-13	1518	1672	191
-12	836	603	190
-10	1064	1125	191
-9	2950	2956	10
-8	2402	2367	8
-7	3249	3233	188
-5	2191	2077	187
-4	3767	3690	187
-3	5388	5306	8
-2	1630	1626	10
-1	2797	2788	187
0	2827	2834	6
1	4102	3931	187
2	2797	2702	189
3	5149	5037	7
6	2392	2277	9
7	3264	3195	187
8	1320	1266	189
9	1448	1650	10
10	831	724	191
12	910	1023	10
** K= 2 L= 5 **			
-12	1611	1677	190
-11	839	961	11
-10	1658	1701	11
-9	950	998	189
-8	1218	1250	8
-7	831	875	187
-6	3860	3850	188
-5	2778	2743	8
-4	1839	1734	7
-3	1537	1481	188
-2	3293	3145	9
-1	2257	2249	186
0	6087	5777	186
1	3782	3692	7
3	1080	1167	188
4	4331	4313	7
5	2273	2189	188
6	1742	1756	188
7	2639	2629	8
8	1491	1605	189
10	1599	1588	10
11	1114	1220	190
** K= 3 L= 5 **			
-12	824	796	10
-10	722	786	10
-8	2334	2259	189
-7	706	689	10
-6	759	794	8
-5	1542	1515	188
-4	2717	2718	8
-2	3300	3195	188
-1	3359	3348	6
0	892	863	184
1	1998	1955	188
2	3105	3071	8
3	1645	1541	188
4	1913	1919	188
5	3281	3260	8
6	1220	1270	188
7	1246	1073	187
8	1743	1791	10
9	1590	1632	190
11	1697	1822	9
** K= 4 L= 5 **			

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-8	608	492	9
-7	854	701	187
-6	1012	1014	9
-4	1813	1801	189
-3	1270	1330	8
-1	1345	1226	187
0	2219	2131	8
1	1279	1300	187
2	2212	2282	188
3	2429	2343	8
6	2243	2241	8
7	2005	1995	188
8	1511	1260	189
9	1318	1313	10
12	1014	1182	11
** K= 5 L= 5 **			
-11	625	377	190
-6	1037	979	187
-2	1446	1427	9
0	1710	1710	188
4	2093	2147	9
6	1338	1306	188
8	919	933	190
10	1446	1475	10
11	648	541	190
** K= 6 L= 5 **			
-11	650	583	11
-7	841	902	188
-6	818	759	190
-4	693	575	9
-3	432	544	5
0	1108	1163	188
2	884	1065	8
6	1258	1189	188
8	719	816	10
** K= 7 L= 5 **			
-10	912	998	10
-8	1009	774	191
-4	1044	1157	10
-2	851	951	187
-1	612	520	8
2	918	924	9
** K= 8 L= 5 **			
-7	681	784	188
-6	891	987	8
-5	539	512	187
-3	1182	1241	9
-2	774	657	186
0	786	602	13
1	854	877	188
7	764	775	189
** K= 9 L= 5 **			
-7	859	888	188
-5	832	879	11
-2	1283	1172	7
-1	1062	987	190
0	1537	1455	187
1	947	912	8
4	1219	1253	9
5	971	962	189
6	937	938	190
10	1049	878	11
** K= 10 L= 5 **			
-8	827	775	190
-4	1261	1384	8
-2	1060	1062	190
1	933	920	5
2	1594	1733	9
4	993	1041	187
8	1466	1360	9
** K= 11 L= 5 **			
-8	649	721	191
-6	1126	1025	9
-5	860	892	9
-3	913	1047	189
-2	1383	1328	189
0	913	1105	6
1	1327	1340	8
4	1132	1161	189
** K= 12 L= 5 **			
-10	1017	948	191
-7	787	675	9
-6	1572	1535	9
-4	1018	1024	188
-2	1126	1116	188
0	1428	1431	9
3	732	651	189
4	993	1076	189
** K= 13 L= 5 **			
-8	1151	1142	10
-4	1352	1420	189
-3	720	819	188
-2	954	907	9
1	1065	1008	7
2	976	1148	190
6	811	868	9
** K= 14 L= 5 **			
-7	1311	1118	10
-5	815	858	190
-3	910	934	189
-1	1525	1598	9
3	1521	1487	190
** K= 15 L= 5 **			
-5	793	676	190
-4	1037	1090	190
0	1262	1338	9
1	1032	1048	189
2	879	982	191
5	1151	1025	9
6	1302	1341	10
** K= 16 L= 5 **			
-6	1017	1053	190
-2	1490	1484	10
-1	699	797	9
2	1416	1337	189
4	1647	1416	11
** K= 17 L= 5 **			
-5	1238	1111	190
-3	1565	1574	10

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
1	1841	1723	190
3	1226	989	10
** K= 18	L= 5 **		
-6	1164	1018	190
-5	1312	1154	10
-1	1786	1681	190
0	1186	1046	190
5	1071	1135	190
** K= 19	L= 5 **		
-4	1263	1343	12
-2	1241	1161	191
2	1419	1439	11
** K= 20	L= 5 **		
0	1356	1355	11
1	744	186	194
** K= 0	L= 6 **		
-12	1266	1316	190
-10	1143	1272	10
-8	1134	1041	8
-6	3192	3157	188
-4	1285	1319	9
-2	2585	2546	9
0	4560	4640	187
4	3531	3531	8
6	2252	2136	188
8	1097	1030	189
10	1707	1618	10
** K= 1	L= 6 **		
-10	819	819	11
-9	844	997	10
-8	1941	1969	188
-7	678	630	188
-5	1127	1200	188
-4	2583	2591	8
-3	1782	1775	8
-2	2676	2571	188
0	1042	1046	185
1	2063	1933	188
2	3213	3018	8
3	1059	1036	9
4	1615	1543	188
5	1057	944	10
6	1574	1531	188
7	1611	1448	189
8	1857	1822	9
12	951	853	190
** K= 2	L= 6 **		
-11	945	838	10
-7	1491	1386	188
-6	1116	1023	8
-5	1852	1932	7
-4	1511	1499	187
-3	525	475	9
-1	3120	3130	186
0	2383	2351	6
1	1468	1382	6
2	1437	1307	189
3	1742	1668	8
4	887	659	187
5	2150	2144	188
6	1976	1795	8
9	1132	1230	11
12	841	860	11
** K= 3	L= 6 **		
-7	970	833	8
-3	1374	1389	188
-2	753	694	9
-1	1153	1266	6
0	1655	1614	186
1	1090	1069	8
3	1951	1959	188
4	1704	1768	7
6	1344	1293	187
7	1371	1441	8
9	1119	843	191
10	1148	1189	10
** K= 4	L= 6 **		
-8	919	778	8
-6	690	686	187
-3	662	765	7
0	557	558	183
1	1280	1285	188
5	911	1044	8
7	1203	1244	188
8	712	685	9
11	984	893	10
** K= 5	L= 6 **		
-10	1234	1238	11
-8	1136	1150	188
-6	954	981	189
-4	1875	2012	7
0	1324	1429	187
1	755	643	185
2	735	832	6
** K= 6	L= 6 **		
-12	1207	1228	9
-9	1440	1251	10
-8	853	1038	188
-7	1899	1903	189
-6	1546	1620	9
-3	2219	2299	8
-2	1109	1348	188
-1	1669	1711	187
0	1078	1147	7
1	901	966	189
3	2192	2227	7
7	1208	1215	187
** K= 7	L= 6 **		
-11	1497	1479	11
-9	1644	1824	190
-8	906	1057	9
-7	804	894	188
-6	1415	1392	189
-5	3191	3424	8
-3	1100	1221	189
-2	1365	1528	10
-1	2942	3107	186
0	1993	2067	186
1	2452	2589	8
4	1700	1810	8
5	1915	2112	189
6	1033	949	190

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	F_o	F_c	PHI
7	848	824	9
11	893	829	190
** K= 8 L= 6 **			
-10	804	832	13
-8	2280	2381	189
-7	1569	1698	9
-4	3020	3169	8
-3	1209	1359	190
-2	2949	2919	188
-1	1937	1908	7
0	1429	1463	187
1	704	580	5
2	3539	3635	9
3	1338	1440	188
4	1681	1683	188
6	2000	2103	188
8	2233	2336	10
** K= 9 L= 6 **			
-10	1595	1552	191
-8	777	646	188
-7	696	673	190
-6	3122	3161	9
-5	746	592	8
-4	1966	1899	189
-2	2492	2438	189
-1	1546	1557	187
0	4507	4571	8
1	1121	967	5
3	1123	1166	7
4	3380	3359	189
5	1440	1386	188
6	2434	2433	9
8	915	896	9
9	1006	812	10
10	1664	1748	191
** K= 10 L= 6 **			
-10	1012	985	190
-9	1214	1241	191
-8	1578	1429	10
-7	1254	1338	9
-5	1286	1334	8
-4	2317	2378	189
-3	2734	2742	189
-2	1573	1580	9
-1	648	504	11
0	1543	1565	7
1	2575	2714	8
2	2141	2201	190
3	2277	2425	189
6	1095	1265	10
7	2321	2279	10
8	1198	1273	190
9	1090	995	191
** K= 11 L= 6 **			
-8	777	870	11
-7	1910	1989	9
-5	1935	1941	189
-2	819	899	10
-1	3093	3142	9
1	1421	1613	188
3	1804	1932	189
5	2301	2500	9
9	1318	1417	191
** K= 12 L= 6 **			
-9	1151	1133	12
-5	1809	1751	190
-3	1770	1735	9
1	2254	2339	189
3	717	917	10
5	1217	932	10
7	1404	1348	190
** K= 13 L= 6 **			
-7	1259	1206	191
-6	853	527	190
-3	1338	1337	10
-1	1251	1213	189
3	1125	1081	10
** K= 14 L= 6 **			
-6	651	607	190
-5	1051	903	9
0	829	747	191
8	701	388	192
** K= 15 L= 6 **			
1	1096	917	191
5	1120	1037	11
** K= 16 L= 6 **			
-2	865	596	9
3	1101	1205	10
** K= 17 L= 6 **			
-4	1314	891	11
0	998	905	190
2	1054	1005	10
6	1137	1038	190
** K= 0 L= 7 **			
-5	642	673	187
-3	449	479	4
-1	700	850	5
1	807	925	185
** K= 1 L= 7 **			
-6	721	731	188
-3	715	643	7
-2	604	606	12
0	751	652	188
1	645	722	188
7	845	609	188
** K= 2 L= 7 **			
-7	797	811	189
-6	689	717	191
-4	849	840	9
-3	1208	1211	9
-1	876	927	187
0	1040	1172	189
1	728	793	189
3	1435	1401	8
7	1063	1097	189
** K= 3 L= 7 **			
-8	1207	1253	190

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-7	867	919	188
-5	763	800	9
-4	1772	1776	9
-2	667	601	194
-1	1572	1736	187
0	1467	1469	187
1	791	745	7
2	1071	1070	12
5	1252	1347	188
6	1016	922	190
9	989	749	11
** K= 4 L= 7 **			
-12	1191	1091	10
-11	1079	964	10
-8	1167	1190	189
-7	965	1080	189
-6	1663	1608	10
-5	1418	1498	9
-2	1493	1518	191
-1	1460	1549	188
0	1678	1654	6
1	1451	1350	7
2	836	1008	9
4	1728	1754	188
5	998	924	189
8	1192	1064	9
** K= 5 L= 7 **			
-11	633	632	10
-9	2151	2032	190
-8	734	652	7
-7	1127	1106	8
-5	2154	2245	8
-4	814	998	186
-3	2555	2657	188
1	2372	2439	8
3	1268	1337	188
5	1041	996	189
7	1504	1296	8
** K= 6 L= 7 **			
-11	1473	1601	191
-8	1248	1185	189
-7	2427	2457	8
-6	959	1007	8
-5	2146	2184	188
-4	749	825	9
-3	832	1060	188
-2	1709	1860	188
-1	3449	3440	7
1	719	715	191
2	1703	1778	8
3	1657	1780	187
4	1206	1155	188
5	1102	1259	9
6	979	917	188
8	1282	1125	10
** K= 7 L= 7 **			
-10	1567	1539	191
-9	959	899	10
-8	1029	1160	9
-7	1428	1298	190
-6	1557	1610	9
-4	2720	2771	188
-3	1199	1266	12
-1	2086	2112	187
0	3490	3474	7
2	1384	1414	189
3	1853	1887	8
4	1784	1905	188
5	1189	1369	188
6	2125	2104	9
9	1044	1016	10
10	1491	1329	191
** K= 8 L= 7 **			
-9	1114	1294	191
-8	1210	1121	10
-7	1207	1271	8
-6	1532	1479	190
-5	1285	1354	9
-3	2826	2870	189
-2	1824	1866	9
1	2869	2952	9
2	777	847	193
3	2800	2825	189
4	1307	1270	9
7	2967	2949	9
9	1032	1003	191
** K= 9 L= 7 **			
-11	1104	1039	191
-8	657	564	189
-7	1638	1640	9
-6	780	753	8
-5	2271	2428	189
-2	1417	1320	187
-1	3531	3665	8
0	1119	1229	6
1	2150	2299	189
3	1854	1915	189
4	1311	1314	188
5	3117	3309	9
9	1687	1782	192
** K= 10 L= 7 **			
-9	731	826	11
-8	1281	1222	11
-5	1003	1128	189
-4	1775	1846	189
-3	1725	1767	10
-2	1708	1723	10
0	1217	1019	8
1	2113	2172	189
2	2446	2588	190
3	1518	1607	10
4	870	995	9
6	1770	1718	9
7	1645	1725	190
8	1639	1761	190
9	757	669	12
** K= 11 L= 7 **			
-10	895	622	12
-9	887	742	12
-7	980	719	190
-6	1791	1724	189
-4	682	687	12
-3	941	1000	11
-2	1785	1755	9
0	2276	2450	189
4	2239	2334	10
6	1360	1556	190
** K= 12 L= 7 **			

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	F_o	F_c	PHI
-7	1128	1121	191
-5	877	909	10
-4	1448	1380	9
-1	971	1263	189
0	1306	1323	189
2	1513	1637	9
3	705	645	12
6	1182	1279	189
8	1014	933	11
** K= 13 L= 7 **			
-8	1147	1060	191
-4	946	997	11
-2	1262	1262	190
2	1035	998	10
4	807	970	190
** K= 14 L= 7 **			
-6	1428	1297	10
-4	972	974	191
0	1361	1432	10
6	1082	875	11
** K= 15 L= 7 **			
-2	1450	1489	10
2	797	760	190
4	1320	1256	11
** K= 16 L= 7 **			
-6	716	650	191
-5	685	355	188
-4	1203	1189	11
0	1209	1043	191
2	1234	1228	10
** K= 17 L= 7 **			
-2	989	827	191
** K= 0 L= 8 **			
-6	918	899	187
-4	921	977	8
-2	980	943	7
0	2071	2020	187
4	2171	2110	8
6	1634	1540	188
10	1490	1461	10
** K= 1 L= 8 **			
-11	823	763	10
-7	1179	1126	189
-5	1020	894	8
-4	1290	1222	8
-3	901	909	9
-2	743	713	187
-1	2026	1968	187
0	1548	1622	186
2	1712	1789	8
3	1731	1755	8
6	1747	1751	188
7	1200	925	188
8	1213	1148	10
9	946	931	11
** K= 2 L= 8 **			
-9	1009	973	190
-8	1519	1499	189
-5	1688	1742	8
-4	1714	1772	8
-3	930	859	189
-2	2139	2107	189
-1	1863	1778	187
1	2159	2075	8
2	2257	2171	9
4	1747	1749	188
5	1910	1949	189
6	698	706	189
7	1056	1006	9
8	1742	1618	10
9	897	761	11
11	1100	1046	191
** K= 3 L= 8 **			
-10	996	948	191
-9	889	891	191
-8	992	1001	188
-7	832	705	8
-6	2416	2439	9
-5	1364	1226	9
-4	875	756	190
-3	1985	1898	189
-2	2231	2110	189
0	3232	3223	7
1	2478	2313	8
3	1495	1460	189
4	2614	2665	188
5	1131	1026	189
6	1481	1498	9
7	1930	1805	9
8	988	774	10
10	1174	1282	191
11	809	844	191
** K= 4 L= 8 **			
-11	783	681	190
-10	1299	1251	191
-9	849	777	191
-8	766	910	10
-7	2085	2009	9
-6	1431	1331	9
-4	2543	2522	188
-3	2235	2180	189
-1	2480	2440	8
0	2908	2815	7
1	875	741	9
2	1676	1641	190
3	2544	2576	189
5	978	1167	9
6	1710	1624	9
7	1452	1364	9
8	805	682	189
9	1329	1276	191
** K= 5 L= 8 **			
-11	1098	1088	190
-9	563	730	10
-8	1861	1872	9
-7	1103	1142	9
-5	2073	2036	189
-4	2045	2104	188
-2	1913	1891	9
-1	2126	2065	8
0	823	788	7
1	1840	1953	188

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
2	1964	1995	189
4	959	793	9
5	1628	1754	9
6	1008	980	9
** K= 6 L= 8 **			
-9	853	771	12
-8	997	1053	9
-6	1955	2113	189
-5	1128	1116	189
-3	953	975	9
-2	1924	1949	9
0	2111	2149	187
1	1201	1138	187
4	1796	1848	8
** K= 7 L= 8 **			
-10	921	649	11
-9	918	977	10
-8	840	833	190
-7	930	994	189
-4	1330	1338	8
-3	1250	1299	9
-2	895	1025	188
2	1174	1212	8
** K= 8 L= 8 **			
-5	1064	1098	9
-1	1056	1195	188
1	606	551	9
2	1216	1070	190
6	1139	1155	9
8	764	807	191
** K= 9 L= 8 **			
-7	848	696	10
-2	615	491	9
-1	1192	1099	8
0	963	895	190
4	1102	1103	10
** K= 10 L= 8 **			
-3	807	965	11
1	1122	902	191
3	995	1174	10
7	882	1011	192
** K= 11 L= 8 **			
-4	1065	1102	10
2	792	793	9
** K= 12 L= 8 **			
-6	1253	1192	10
-2	1134	1224	189
0	1082	1004	10
** K= 13 L= 8 **			
-8	1113	943	10
-5	1081	1219	10
-2	1070	1354	10
2	1092	1103	189
4	951	863	11
** K= 14 L= 8 **			
-7	990	858	11
-5	1122	1111	190
-4	1362	1465	11
-1	1426	1441	8
0	1176	1201	190
2	1377	1344	11
6	1149	1191	190
** K= 15 L= 8 **			
-6	1284	1040	11
-3	1297	1337	11
-2	916	866	191
-1	1475	1515	190
0	1100	1115	10
3	1631	1697	10
4	1071	1038	190
5	1058	908	191
** K= 16 L= 8 **			
-5	1634	1463	11
-3	1394	1369	190
-1	844	780	190
1	2048	2015	10
5	1495	1169	191
** K= 17 L= 8 **			
-3	1082	1053	191
-1	1232	1181	11
3	1054	1112	191
** K= 0 L= 9 **			
-11	1037	1089	191
-7	1600	1577	9
-5	883	993	188
-3	726	683	189
-1	1074	1116	7
** K= 1 L= 9 **			
-10	1032	935	192
-9	1051	1061	11
-7	844	857	189
-6	1232	1331	9
-4	938	1007	189
-3	1029	971	10
-2	802	806	190
-1	714	853	188
0	1872	1878	7
3	632	593	7
4	1469	1498	188
5	901	879	188
8	760	722	10
** K= 2 L= 9 **			
-9	1281	1344	190
-8	1439	1453	9
-6	714	711	191
-5	1712	1798	9
-4	1055	1162	188
-3	2242	2292	189
-2	977	1117	11
1	2478	2442	9
2	1293	1174	190
3	1925	1915	189
6	1147	1030	9
7	2154	2045	9

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
** K= 3	L= 9 **		
-11	1553	1404	191
-8	746	661	189
-7	2190	2341	9
-5	2426	2450	189
-3	962	898	190
-1	4177	4235	8
1	1632	1640	189
3	2236	2290	189
5	2584	2810	10
9	1524	1558	192
** K= 4	L= 9 **		
-10	839	803	192
-9	1413	1416	11
-8	1166	1202	10
-7	1105	1010	189
-5	1331	1530	189
-4	2026	2083	189
-3	2316	2379	10
-2	1085	1174	11
0	1313	1299	8
1	2436	2488	189
2	2175	2170	190
3	1980	2114	9
5	725	749	10
6	1477	1406	10
7	1903	1984	190
8	1320	1378	191
** K= 5	L= 9 **		
-10	752	830	12
-8	717	794	10
-7	925	753	190
-6	2119	2199	190
-5	670	569	8
-4	920	811	9
-2	2173	2215	9
-1	1007	1190	188
0	2924	2818	188
4	2322	2532	9
6	1471	1581	189
8	912	803	190
10	1397	1370	11
** K= 6	L= 9 **		
-10	870	797	11
-8	904	1026	191
-4	1812	1767	10
-2	1214	1278	189
0	1212	1236	188
2	1913	2078	9
6	1198	1274	189
8	1182	1161	10
** K= 7	L= 9 **		
-6	833	929	9
-2	905	1075	189
0	1087	992	10
4	968	906	190
6	901	551	11
** K= 8	L= 9 **		
2	934	911	190
** K= 9	L= 9 **		
-6	944	796	190
-2	832	839	10
0	1183	1079	189
4	891	885	10
** K= 10	L= 9 **		
-3	929	931	11
3	698	791	10
** K= 11	L= 9 **		
-5	989	1190	10
-1	1058	1206	189
5	1023	930	191
** K= 12	L= 9 **		
-8	855	1053	191
-4	955	933	10
-2	1087	1133	190
2	1078	1081	11
** K= 13	L= 9 **		
-6	969	1047	11
-4	809	824	191
-1	1131	1061	190
0	1225	1237	10
4	1122	975	190
** K= 14	L= 9 **		
-3	1666	1630	191
1	1570	1692	10
3	1306	1111	191
** K= 15	L= 9 **		
-5	925	935	191
-2	1257	1135	190
-1	1417	1445	10
0	1071	1091	10
3	1041	911	192
4	1091	1174	191
** K= 16	L= 9 **		
-4	1436	1399	191
-2	962	857	11
2	1672	1572	191
** K= 0	L= 10 **		
-10	1102	952	12
-8	1492	1460	9
-6	2396	2453	190
-2	2169	2154	10
0	2104	2264	188
4	1643	1636	9
6	686	616	190
** K= 1	L= 10 **		
-10	1021	968	12
-9	949	914	11
-8	1707	1815	190
-5	1554	1555	189
-4	2081	2190	9
-3	907	708	11
-2	1748	1805	189

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-1	1431	1359	7
0	864	850	186
1	1267	1199	191
2	1893	1896	9
5	982	1087	10
6	865	683	189
8	923	841	10
** K= 2 L= 10 **			
-10	985	1043	192
-7	1862	1842	190
-6	1216	1428	9
-5	919	977	9
-4	1990	1970	189
-3	1402	1278	11
-1	2265	2213	188
0	2381	2365	8
2	1682	1698	190
3	1776	1730	9
6	1769	1820	9
** K= 3 L= 10 **			
-10	839	560	12
-9	1256	1317	190
-8	886	837	9
-7	1096	1085	9
-6	1837	1832	189
-5	999	967	9
-3	1994	2041	189
-2	2095	2062	9
0	2442	2347	188
1	1649	1630	9
3	1487	1586	189
4	2267	2425	9
6	948	1149	190
7	1280	1244	9
8	1080	987	191
** K= 4 L= 10 **			
-9	1060	926	11
-8	846	948	190
-5	1884	1911	189
-4	1077	1167	10
-3	1047	1017	9
-2	1581	1695	189
-1	1714	1702	8
1	2192	2222	189
2	1780	1794	10
4	1382	1212	188
5	1571	1713	10
6	977	990	189
7	1301	1306	190
8	1261	1331	11
** K= 5 L= 10 **			
-7	951	1088	190
-4	905	927	189
-3	876	1055	10
-1	1920	1849	189
0	1155	1195	8
2	1184	1072	190
3	1663	1756	9
5	1417	1419	189
6	1364	1264	9
9	1090	1175	11
** K= 6 L= 10 **			
0	920	891	190
1	937	891	8
3	692	763	189
4	990	988	10
6	754	677	190
7	855	811	11
** K= 7 L= 10 **			
2	598	427	11
** K= 8 L= 10 **			
-4	1011	1086	10
7	752	402	191
** K= 9 L= 10 **			
-7	722	701	191
-6	961	933	11
-2	989	910	191
-1	1161	1171	190
3	699	714	10
5	938	1018	190
** K= 10 L= 10 **			
-5	1251	1217	10
-3	1267	1319	190
1	1527	1511	10
3	668	807	190
** K= 11 L= 10 **			
-7	1093	1001	10
-3	942	880	189
2	785	473	8
** K= 12 L= 10 **			
-7	930	486	10
-4	795	720	189
0	1179	890	8
** K= 14 L= 10 **			
-2	860	924	10
** K= 15 L= 10 **			
1	807	1053	190
** K= 0 L= 11 **			
-7	913	875	190
-5	869	798	9
-1	1137	1229	189
5	695	724	189
** K= 1 L= 11 **			
-7	671	583	10
-3	1001	1055	190
0	692	800	189
1	876	854	10
3	946	858	190
7	614	621	11
** K= 2 L= 11 **			
-5	1023	1120	190
-3	735	583	11
-1	1003	966	8

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
0	842	490	10
1	1306	1186	191
4	860	759	190
5	981	952	10
** K=	3	L= 11 **	
-8	739	684	10
-7	842	789	190
-4	815	710	190
-3	731	666	10
-2	911	998	9
-1	1377	1310	188
2	1201	1207	190
3	949	1071	9
5	829	823	190
6	811	797	9
8	674	698	192
** K=	4	L= 11 **	
-6	591	712	190
-4	1145	1032	9
0	1464	1406	189
4	879	934	10
6	1054	1079	190
** K=	5	L= 11 **	
7	895	896	190
** K=	6	L= 11 **	
-8	875	889	191
-4	999	1014	10
-1	630	424	193
5	772	866	190
** K=	7	L= 11 **	
-7	793	759	191
-6	1535	1411	11
-5	610	609	12
-2	1041	1256	191
-1	1391	1206	189
0	1103	1118	10
1	953	843	9
4	1193	1166	190
5	1004	1024	190
** K=	8	L= 11 **	
-5	1535	1634	10
-4	868	734	190
-3	1992	2031	191
1	1943	2127	10
3	1449	1437	190
5	682	680	192
7	1315	1524	10
** K=	9	L= 11 **	
-7	2003	2029	10
-5	1672	1607	190
-3	1123	1082	191
-1	2780	2774	9
1	796	696	191
3	1830	1761	190
4	793	886	191
5	1525	1609	11
** K=	10	L= 11 **	
-6	874	638	11
-4	2011	2010	190
-3	1275	1288	11
-2	792	709	12
0	1689	1628	10
1	1422	1274	190
2	1597	1746	191
3	1024	907	11
6	1342	1468	11
** K=	11	L= 11 **	
-6	1854	1635	191
-2	1880	1879	10
0	1350	1474	190
4	1785	1648	11
** K=	12	L= 11 **	
-4	1229	1042	11
-2	838	495	191
1	858	770	190
2	1019	992	11
** K=	0	L= 12 **	
-6	916	1075	190
-2	995	1078	10
0	1028	1190	190
4	1170	1210	10
6	911	643	191
** K=	1	L= 12 **	
-4	920	971	10
-3	931	800	11
0	809	906	189
2	890	893	10
6	777	726	190
** K=	2	L= 12 **	
-7	772	743	191
-5	889	921	10
-4	680	740	10
-1	1261	1186	190
1	910	608	10
2	734	637	12
** K=	3	L= 12 **	
-6	645	634	11
-5	1103	913	10
-2	896	810	192
1	982	779	11
** K=	4	L= 12 **	
-6	810	709	11
** K=	5	L= 12 **	
0	852	742	9
** K=	6	L= 12 **	
-3	1068	875	191
** K=	7	L= 12 **	
-7	918	886	11
-6	699	560	10

Table E.3. Observed and Calculated Structure Factors for $Os_4(CO)_{14}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-5	1059	1072	191
-2	769	897	190
-1	1749	1622	9
0	758	725	8
3	1142	1153	191
4	993	831	191
** K= 8 L= 12 **			
-6	670	525	10
-4	1808	1786	191
0	1508	1492	10
2	1725	1775	191
** K= 9 L= 12 **			
-6	1824	1740	191
-2	1866	2002	10
0	1768	1793	190
4	1806	1994	11
** K= 10 L= 12 **			
-5	1036	1101	191
-4	1068	1140	11
-3	1144	1210	11
-2	1017	840	190
1	1832	1579	191
2	1130	1333	11
3	961	654	12
** K= 11 L= 12 **			
-1	1751	1604	191
3	1200	1150	11
** K= 12 L= 12 **			
1	739	803	12
** K= 0 L= 13 **			
-7	1029	1054	191
-5	1171	1213	10
-1	2103	2185	190
1	885	768	10
3	1403	1513	11
5	1744	1710	191
** K= 1 L= 13 **			
-5	973	967	11
-4	793	856	11
-3	1093	1112	191
-2	1161	1090	190
1	1759	1622	11
2	1357	1216	11
3	822	830	190
5	1009	919	191
** K= 2 L= 13 **			
-7	769	595	10
-6	1152	1261	11
-4	833	813	190
-3	1145	1134	191
-2	1052	1013	191
0	1684	1683	10
1	929	750	11
3	1173	989	191
4	1456	1448	191
** K= 3 L= 13 **			
-4	1064	1113	191
-3	907	817	192
-1	1471	1514	9
0	1041	1151	9
2	981	1048	191
3	1205	1070	190
** K= 4 L= 13 **			
-5	1198	1149	191
-4	940	880	190
-3	750	420	14
-2	1096	971	11
1	1045	849	192
2	1107	916	191
** K= 5 L= 13 **			
-6	1057	858	192
0	903	795	191
** K= 0 L= 14 **			
-2	703	505	190
0	795	714	10
** K= 1 L= 14 **			
0	887	610	10
3	871	798	190
** K= 2 L= 14 **			
-4	1115	1111	190
0	783	822	9
2	1067	1012	191
** K= 3 L= 14 **			
-2	1541	1522	11
0	1255	1110	190
** K= 4 L= 14 **			
-4	1132	1184	12
0	811	881	190
2	883	1042	12
** K= 5 L= 14 **			
-3	773	743	12
-1	1271	1299	191
** K= 6 L= 14 **			
1	834	950	11

Table F.1. U_{ij} or U_{iso} Values (*100) for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

	U11(U)	U22	U33	U12	U13	U23
Ir	2.81(5)	2.69(5)	3.99(7)	0.14(4)	0.08(5)	-0.12(5)
Os(1)	2.79(6)	2.98(6)	4.64(7)	-0.24(5)	-0.59(5)	0.11(5)
Os(2)	5.08(7)	3.26(6)	4.94(7)	-0.46(5)	0.01(6)	0.33(6)
Os(3)	2.72(6)	3.28(6)	4.98(7)	-0.50(5)	-0.04(5)	-0.40(5)
O(11)	7.5 (16)	8.9 (17)	8.7 (18)	3.3 (13)	-2.1 (14)	0.0 (14)
O(12)	10.9 (19)	7.9 (15)	5.4 (15)	1.7 (13)	-1.9 (13)	-3.3 (12)
O(13)	5.1 (14)	5.7 (13)	16.8 (26)	-0.4 (11)	-5.5 (15)	1.8 (15)
O(21)	16.3 (27)	5.3 (14)	8.3 (19)	0.8 (15)	-0.7 (18)	1.0 (13)
O(22)	8.2 (17)	11.3 (19)	6.1 (15)	2.1 (14)	-0.9 (13)	-0.4 (14)
O(23)	6.8 (15)	8.4 (16)	5.8 (14)	1.8 (12)	-0.6 (12)	0.2 (12)
O(24)	12.1 (22)	7.3 (16)	11.1 (21)	-6.8 (16)	0.4 (17)	2.9 (15)
O(31)	6.8 (15)	5.6 (13)	9.8 (18)	1.7 (11)	0.6 (13)	-3.7 (13)
O(32)	7.5 (15)	5.9 (13)	5.6 (13)	0.1 (11)	1.9 (11)	-2.2 (11)
O(33)	3.6 (12)	7.1 (14)	14.0 (23)	-2.4 (11)	0.8 (13)	3.1 (14)
O(34)	4.4 (12)	6.7 (12)	3.9 (11)	0.9 (9)	-1.9 (10)	-0.2 (10)
O(41)	8.5 (15)	5.4 (13)	7.2 (15)	1.4 (11)	3.0 (12)	-1.2 (12)
C(11)	6.1 (9)					
C(12)	6.5 (9)					
C(13)	6.5 (9)					
C(21)	8.5 (11)					
C(22)	6.7 (9)					
C(23)	4.7 (7)					
C(24)	6.8 (9)					
C(31)	5.0 (7)					
C(32)	4.8 (7)					
C(33)	4.7 (7)					
C(34)	3.9 (6)					
C(41)	3.9 (7)					
C(1)	3.2 (6)					
C(2)	4.7 (7)					
C(3)	4.1 (7)					
C(4)	3.9 (7)					
C(5)	4.2 (7)					
C(6)	9.9 (13)					
C(7)	6.1 (8)					
C(8)	7.6 (10)					
C(9)	7.5 (10)					
C(10)	7.7 (10)					

Table F.2. Hydrogen Atom Coordinates for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Atom	*x/a*	*y/b*	*z/c*	U(iso)
H1	0.128	0.043	1.028	10.9
H2	0.100	-0.025	0.962	10.9
H3	0.066	0.041	0.885	10.9
H4	0.389	0.060	1.025	7.1
H5	0.345	-0.006	1.070	7.1
H6	0.288	0.059	1.082	7.1
H7	0.467	0.045	0.653	8.6
H8	0.464	0.050	0.825	8.6
H9	0.468	-0.019	0.744	8.6
H10	0.319	-0.053	0.487	8.5
H11	0.365	0.015	0.442	8.5
H12	0.264	0.002	0.400	8.5
H13	0.109	-0.049	0.589	8.7
H14	0.104	0.018	0.500	8.7
H15	0.056	0.013	0.649	8.7

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF$_o$	F$_c$	σ
-15,	0,	1	
3	1388	1307	590
-15,	2,	1	
2	1166	1153	645
-15,	4,	1	
1	1477	1510	535
-14,	1,	1	
4	963	1051	793
-14,	2,	1	
1	1689	1766	472
-14,	3,	1	
4	996	940	710
-14,	4,	1	
1	667	780	1014
2	1527	1595	499
3	1240	1372	590
4	645	653	1033
-14,	5,	1	
3	712	541	918
-14,	6,	1	
3	1842	1881	460
-14,	7,	1	
3	705	760	986
-13,	0,	1	
5	2714	2760	447
-13,	1,	1	
1	1641	1713	464
5	940	920	826
-13,	2,	1	
1	933	873	699
2	879	725	748
3	1018	1004	639
4	1317	1379	561
5	1377	1342	564
-13,	3,	1	
1	1370	1431	542
2	1334	1373	524
4	866	961	799
5	935	989	758
-13,	4,	1	
1	1868	1996	442
2	1023	991	646
3	1401	1635	551
-13,	5,	1	
2	1675	1702	452
4	1410	1509	535
-13,	6,	1	
2	2053	2028	406
-13,	7,	1	
2	978	1059	735
4	1266	1232	569
-13,	8,	1	
3	820	777	834
4	928	803	748
-13,	10,	1	
-13,	11,	1	
2	864	707	845
-13,	12,	1	
1	1082	868	681
-12,	0,	1	
2	971	989	704
6	1447	1482	553
-12,	1,	1	
4	1829	1757	434
6	1070	957	668
-12,	2,	1	
1	1829	1874	415
2	1888	2017	418
4	973	988	702
5	1298	1389	596
6	658	502	1021
-12,	3,	1	
3	975	1050	675
4	1432	1493	512
-12,	4,	1	
2	2502	2606	374
3	1981	2070	410
4	1597	1562	476
-12,	5,	1	
1	1252	1297	578
3	1365	1415	536
4	623	666	1060
-12,	6,	1	
3	2825	2860	442
-12,	7,	1	
1	1040	1007	663
3	1140	1184	628
-12,	8,	1	
1	998	952	700
2	1211	1239	587
3	1083	1028	639
-12,	12,	1	
1	1287	1382	594
3	684	715	1037
-11,	0,	1	
1	837	931	768
5	1913	1921	438
-11,	1,	1	
1	3377	3504	506
2	555	626	1073
5	817	812	839
-11,	2,	1	
3	1133	1245	590
4	1970	2063	405
-11,	3,	1	
1	2522	2615	414
2	1087	1265	600
5	1106	1096	593
-11,	4,	1	
1	1673	1592	410
3	2165	2248	371
4	795	864	806
5	747	764	861
-11,	5,	1	
1	1196	1119	533
2	1745	1806	426
4	1116	1127	589
5	938	943	705
-11,	6,	1	
1	913	920	684
2	2729	2723	437
6	1061	1014	677
-11,	7,	1	
2	1588	1534	473
3	795	767	795
4	874	804	742
6	833	815	865
-11,	8,	1	
1	1878	1822	422
2	581	483	1133
-11,	9,	1	
1	1077	1067	619
2	1221	1164	582
3	934	881	712
-11,	10,	1	
3	1115	1094	672
5	1622	1548	518
-11,	11,	1	
1	1267	1198	537
-11,	12,	1	
3	1189	1155	630
4	2296	2167	433
-11,	14,	1	
1	1112	1084	628
-10,	1,	1	
3	594	482	929
4	627	608	935
5	603	742	1091
-10,	2,	1	
1	1687	1716	394
2	1017	1071	576
3	839	886	697
4	1406	1487	471
5	795	804	810
6	761	910	904
7	981	896	691
-10,	3,	1	
3	1247	1307	497
5	1476	1526	487
-10,	4,	1	
2	1529	1591	427
3	1349	1402	476
4	2144	2302	379
5	707	860	938
6	1201	1081	575
1	829	898	729
2	1406	1389	455
3	1570	1620	422
5	1045	975	639
6	837	822	817
7	814	841	886
-10,	6,	1	
1	982	925	627
2	971	946	639
3	2187	2149	362
5	1535	1599	502
-10,	7,	1	
1	945	929	640
2	2044	1978	381
3	839	914	793
-10,	8,	1	
1	1682	1669	407
2	2250	2267	372
6	1311	1135	560
-10,	9,	1	
2	2096	2044	388
6	1371	1239	586
-10,	10,	1	
1	2262	2217	352
4	964	958	738
-10,	11,	1	
2	1364	1407	501
3	1283	1287	561
5	810	913	944
-10,	12,	1	
3	1375	1236	520
4	761	793	941
5	1458	1251	579
-10,	13,	1	
3	1778	1862	476
-10,	14,	1	
2	769	785	808
3	708	882	988
4	1028	930	725
-10,	15,	1	
3	2042	1975	440
-10,	16,	1	
1	1269	1280	574
-9,	0,	1	
1	663	659	827
3	3319	3498	500
-9,	1,	1	
1	1641	1730	382
3	1428	1375	436
7	1710	1634	456
-9,	2,	1	
2	1261	1290	450
3	1149	1261	521
4	2397	2529	371
5	949	1065	708

1 1132 1090 671 2 1816 1861 397 -10, 5, 1 8 790 683 896

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ	l	kF_o	F_c	σ	l	kF_o	F_c	σ	l	kF_o	F_c	σ
	-9,	3, 1			-9,	16, 1		1	1208	1175	450	4	1544	1593	406
1	1308	1278	438	2	1806	1776	433	2	2742	2748	401	5	1571	1635	433
3	1328	1348	442	3	924	980	746	5	1179	1159	608	6	1023	1048	668
4	1136	1208	544		-8,	0, 1			-8,	10, 1		7	1560	1590	521
7	1641	1717	492	2	3283	3455	485	1	3026	2919	449		-7,	5, 1	
	-9,	4, 1		6	605	605	1091	3	1459	1368	420	1	1145	1191	427
1	1214	1135	473	8	1811	1796	481		-8,	11, 1		4	1495	1516	429
3	819	839	668		-8,	1, 1		2	2387	2321	337	5	895	906	682
4	1170	1263	536	1	721	796	688	3	949	916	636	6	1992	2064	415
5	1669	1823	449	2	2323	2384	347		-8,	12, 1		7	602	457	1150
6	1348	1360	543	4	3078	3244	471	1	1196	1111	457		-7,	6, 1	
7	1114	987	635	5	839	932	745	2	666	772	874	1	2098	2004	306
	-9,	5, 1		6	639	665	989	5	674	736	970	3	878	804	609
3	1259	1248	486		-8,	2, 1			-8,	13, 1		6	1935	1830	435
4	683	686	831	1	1668	1658	345	2	1090	1078	558	8	950	981	785
6	1662	1615	469	2	1472	1540	388	3	1379	1252	477		-7,	7, 1	
7	831	806	857	3	2178	2243	315	5	905	787	740	1	1167	1160	404
	-9,	6, 1		4	833	857	697		-8,	14, 1		4	1802	1732	389
1	2873	2877	442	6	1756	1817	444	2	606	756	961	5	1725	1575	423
2	1766	1689	383	7	1264	1328	593	4	1203	1089	562	6	1427	1282	538
6	2067	1949	408	8	847	845	841		-8,	15, 1		7	849	844	870
	-9,	7, 1			-8,	3, 1		3	1670	1592	436		-7,	8, 1	
1	908	896	624	1	1277	1341	427		-8,	16, 1		1	3969	3852	411
2	747	760	761	2	1522	1541	370	1	1879	1863	404	2	2713	2676	398
3	1346	1444	503	4	1998	2064	356	3	1295	1272	551	4	1685	1666	413
4	857	840	763	5	2077	2154	359	4	826	717	827	5	1380	1340	558
6	1319	1174	577	8	920	1038	763		-8,	17, 1		6	646	471	1053
	-9,	8, 1			-8,	4, 1		2	1225	1155	563		-7,	9, 1	
1	4553	4388	450	4	1984	2002	354	3	1668	1654	485	1	961	990	514
2	1037	995	554	5	2134	2166	363		-8,	18, 1		3	897	844	588
4	755	832	885	6	1965	1991	425	2	1223	1172	574	4	1445	1475	459
6	729	696	965		-8,	5, 1			-7,	0, 1		5	1110	1145	655
7	1059	1072	722	1	1651	1652	359	3	6405	6502	418	7	900	876	850
	-9,	9, 1		3	759	793	727	5	628	729	948		-7,	10, 1	
1	1417	1316	437	4	810	750	711		-7,	1, 1		1	1748	1719	327
2	1157	1123	518	5	1497	1440	451	1	1599	1627	314	2	2549	2518	399
3	910	842	689	7	1194	1094	599	3	1977	2153	329	3	2348	2349	367
4	654	800	1044	8	851	766	822	4	688	818	823	4	1469	1495	449
	-9,	10, 1			-8,	6, 1		5	909	888	658	7	944	793	821
1	1923	1911	365	1	1429	1360	380	7	962	845	681		-7,	11, 1	
2	2061	2039	347	2	1898	1908	348		-7,	2, 1		3	735	934	765
3	1968	1985	391	3	784	578	702	1	1434	1396	356	6	986	992	730
5	1094	1105	672	5	2996	3029	485	2	2762	2904	409		-7,	12, 1	
	-9,	11, 1			-8,	7, 1		3	4170	4102	240	1	675	579	736
1	1208	1235	516	1	1356	1339	399	4	2538	2636	369	3	2289	2261	360
2	1310	1393	507	2	2081	2004	323	5	905	914	679	4	623	555	911
	-9,	12, 1		5	1177	1330	660	7	837	963	817	6	1075	982	692
3	1855	1853	412	7	775	789	919		-7,	3, 1			-7,	13, 1	
4	2372	2277	376		-8,	8, 1		1	1671	1629	336	6	1228	1177	624
5	1167	1119	638	1	2368	2339	362	3	1155	1149	448		-7,	14, 1	
	-9,	13, 1		2	2288	2351	366	4	1821	1885	363	1	1348	1327	460
1	957	885	614	3	1365	1361	465	6	1090	1071	618	2	956	952	590
2	1044	1017	601	4	1316	1320	519	7	1291	1287	568	4	922	955	705
	-9,	14, 1		5	990	935	712		-7,	4, 1		6	668	815	1098
1	1122	1110	560	6	766	801	930	1	2218	2221	334		-7,	16, 1	
4	2846	2665	379		-8,	9, 1		2	1531	1505	363	2	1343	1278	489

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
	-7, 16, 1		
3	1058	1118	617
	-7, 18, 1		
1	1980	2036	412
2	1188	1197	589
	-6, 0, 1		
2	4749	4916	384
6	1107	1061	590
8	826	834	872
	-6, 1, 1		
1	826	871	502
2	4522	4643	388
4	2136	2222	324
6	1584	1546	432
7	657	717	997
	-6, 2, 1		
1	2594	2555	394
2	2242	2343	330
3	2048	2142	307
7	1646	1695	468
	-6, 3, 1		
1	3245	3135	409
2	3086	3082	433
4	981	1012	537
5	1133	1141	534
6	685	596	877
	-6, 4, 1		
4	1110	1171	503
5	2120	2199	353
6	1430	1437	491
7	1003	932	692
8	997	971	721
	-6, 5, 1		
1	4098	4066	361
2	1469	1517	340
5	860	790	704
	-6, 6, 1		
1	1512	1488	303
3	857	906	580
4	2341	2323	339
5	3058	3048	488
	-6, 7, 1		
1	4031	3931	366
3	633	556	711
5	1080	1068	655
6	1472	1341	508
	-6, 8, 1		
1	1208	1151	363
2	739	750	586
3	2086	2112	319
4	3706	3766	484
	-6, 9, 1		
1	2345	2320	365
2	1605	1559	333
5	1528	1558	456
6	747	790	922
7	1055	1067	754
	-6, 10, 1		
1	1483	1415	338
2	1702	1727	330
3	2488	2456	370
4	1608	1601	393
5	1270	1212	540
6	1106	1003	628
	-6, 11, 1		
2	1771	1690	326
4	997	1069	578
5	1226	1151	538
	-6, 12, 1		
1	1429	1364	385
2	1796	1826	344
6	1080	1003	641
7	2008	1850	466
	-6, 13, 1		
2	890	947	604
3	953	863	592
	-6, 14, 1		
1	1627	1566	371
2	768	791	714
4	1025	1072	607
5	991	1060	699
	-6, 15, 1		
1	728	818	771
	-6, 16, 1		
2	960	937	631
3	1817	1788	407
5	2112	2108	416
	-6, 18, 1		
2	597	502	1040
3	1411	1415	514
4	2014	1990	436
	-6, 19, 1		
2	714	741	918
	-5, 0, 1		
1	948	919	468
3	5387	5529	382
5	2437	2508	324
9	926	905	824
	-5, 1, 1		
1	1889	1896	312
3	1520	1484	327
7	1191	1198	602
8	772	869	930
	-5, 2, 1		
1	2303	2226	313
2	3226	3370	402
3	2483	2624	362
4	1964	1983	314
5	957	858	600
6	1579	1658	462
7	591	526	1108
9	1309	1353	614
	-5, 3, 1		
1	1986	1958	292
2	1365	1359	336
4	885	924	568
5	1097	1089	528
6	905	923	717
8	1036	969	677
	-5, 4, 1		
1	4558	4449	323
2	1334	1326	324
7	1372	1351	557
8	1350	1344	556
9	1411	1279	559
	-5, 5, 1		
1	694	691	490
3	1362	1302	358
6	1620	1620	465
	-5, 6, 1		
1	868	835	391
3	1240	1263	391
5	1693	1614	421
8	1891	1821	488
	-5, 7, 1		
3	2024	2018	313
5	2058	2043	367
6	1079	1004	652
7	1107	884	669
	-5, 8, 1		
1	1085	1012	356
2	1483	1513	313
3	976	997	487
4	2214	2279	308
5	1886	1943	392
6	783	777	891
8	879	810	918
	-5, 9, 1		
2	953	861	441
3	3459	3362	437
5	2152	2161	353
6	773	818	906
7	719	690	1060
8	866	867	973
	-5, 10, 1		
1	665	591	600
2	740	768	595
3	2249	2230	336
4	2617	2664	373
6	878	866	776
7	901	860	826
	-5, 11, 1		
2	1717	1712	323
3	2788	2867	441
5	1232	1149	514
6	1414	1325	519
	-5, 12, 1		
1	832	774	554
2	2386	2428	353
3	2063	2186	346
5	645	619	923
6	808	660	830
	-5, 13, 1		
2	2263	2306	312
3	1369	1320	419
4	799	763	712
5	1044	969	573
6	1562	1494	504
	-5, 14, 1		
1	1570	1549	375
2	2474	2517	399
	-5, 15, 1		
2	2105	2074	348
6	2020	1983	450
	-5, 16, 1		
1	1084	1040	542
3	926	845	639
4	1377	1432	500
	-5, 17, 1		
2	1634	1576	411
3	1124	1101	578
5	1019	962	688
	-5, 18, 1		
1	782	719	773
	-5, 19, 1		
2	1144	1186	611
3	1494	1454	508
	-4, 0, 1		
2	2085	2086	244
4	652	648	702
6	3558	3746	513
	-4, 1, 1		
1	939	950	411
2	2459	2494	291
4	910	933	564
6	2067	2105	383
7	1395	1350	513
8	1428	1498	560
	-4, 2, 1		
1	3406	3165	265
2	961	870	409
3	540	711	758
4	1552	1632	368
5	1067	1120	543
6	2072	2166	384
7	1780	1952	454
	-4, 3, 1		
1	3236	3243	258
2	2309	2296	336
4	1712	1740	334
6	763	801	848
7	1725	1776	488
	-4, 4, 1		
1	477	393	642

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
	-4, 4, 1		
2	2641	2668	349
3	2124	2093	287
4	1866	1788	324
7	1233	1158	642
8	898	794	792
	-4, 5, 1		
1	4660	4556	297
2	1718	1758	254
3	1204	1208	364
4	1315	1321	406
5	1103	1007	561
7	1460	1446	553
9	1224	1276	697
	-4, 6, 1		
1	1063	1027	314
2	753	798	443
3	3714	3806	381
4	2527	2597	436
5	1655	1641	404
9	1051	1023	770
	-4, 7, 1		
1	2974	2992	350
3	1290	1251	346
6	671	688	967
7	1623	1361	523
	-4, 8, 1		
1	752	719	437
4	4736	4808	420
5	875	918	647
7	1082	1013	728
	-4, 9, 1		
1	711	728	506
3	1522	1560	326
7	1755	1733	506
	-4, 10, 1		
1	718	601	512
2	2803	2807	392
3	1345	1322	378
4	2474	2477	366
5	1808	1781	366
6	1179	1177	572
8	892	742	907
	-4, 11, 1		
1	886	851	474
3	2199	2194	328
6	897	939	732
	-4, 12, 1		
1	3729	3676	424
2	2595	2591	361
4	984	947	532
6	1104	1109	607
7	1138	1023	638
	-4, 13, 1		
1	1389	1446	397
3	2166	2126	306
7	679	676	1057
	-4, 14, 1		
1	4445	4377	450
2	715	752	711
4	1258	1240	481
5	806	878	790
7	1232	1208	633
	-4, 15, 1		
1	1537	1569	408
3	1466	1397	434
	-4, 16, 1		
1	1728	1740	396
2	1349	1419	475
3	1865	1880	384
5	2240	2245	387
	-4, 17, 1		
1	984	1076	626
	-4, 18, 1		
1	987	1005	644
3	1122	1092	594
4	2469	2426	372
	-4, 20, 1		
1	1531	1522	480
	-3, 0, 1		
1	942	912	291
3	989	1102	446
5	3747	4044	449
7	944	887	660
	-3, 1, 1		
1	1753	1711	226
2	1045	1027	309
3	2216	2178	322
5	3073	3093	441
7	1934	1878	406
	-3, 2, 1		
1	2449	2426	200
2	1971	1931	242
3	1295	1355	356
4	1837	1885	310
5	2484	2550	368
6	2186	2246	361
9	1084	1068	719
	-3, 3, 1		
1	1902	1875	212
2	4313	4252	205
4	1183	1217	446
5	2930	3021	477
8	769	868	926
	-3, 4, 1		
1	5449	5226	171
2	1023	1064	266
3	2539	2733	393
6	1592	1618	454
8	1767	1692	485
9	1060	1130	757
	-3, 5, 1		
1	1337	1314	211
2	4017	4107	314
3	2196	2220	329
4	2323	2277	334
5	984	961	589
6	1578	1516	481
	-3, 6, 1		
1	1365	1374	253
2	2666	2695	347
4	1270	1340	386
5	1191	1272	493
7	1199	1014	664
8	2111	2036	487
	-3, 7, 1		
2	2848	2917	358
3	2528	2664	394
4	2546	2645	414
5	1669	1672	372
6	1158	1132	570
9	1027	873	833
	-3, 8, 1		
3	1197	1187	356
4	1507	1558	349
5	606	779	908
6	1695	1832	438
7	1743	1653	494
	-3, 9, 1		
2	863	840	427
3	3128	3179	410
4	1358	1410	387
5	1841	1853	349
8	1369	1275	638
	-3, 10, 1		
1	2160	2094	317
3	1464	1641	370
4	1908	1963	321
5	1419	1401	415
6	1543	1489	450
	-3, 11, 1		
2	1408	1413	339
3	2883	2910	421
4	756	778	657
5	705	801	803
	-3, 12, 1		
1	1942	1882	298
2	3024	3051	442
3	1437	1537	389
5	1552	1564	428
	-3, 13, 1		
1	536	573	877
2	3147	3175	459
3	1072	1145	483
4	680	666	735
	-3, 14, 1		
1	1583	1560	368
2	2897	2933	438
4	754	722	714
7	723	658	946
	-3, 15, 1		
2	2914	2921	402
	-3, 16, 1		
1	1691	1690	400
6	955	894	716
	-3, 17, 1		
2	1551	1573	432
3	987	985	624
	-3, 18, 1		
3	967	897	667
	-3, 19, 1		
3	1205	1153	584
4	730	711	914
	-3, 20, 1		
2	1375	1302	512
	-2, 0, 1		
2	1232	1260	307
4	1446	1549	328
6	3225	3368	469
8	1324	1409	558
	-2, 1, 1		
2	1201	1173	348
3	987	1043	494
4	2984	3030	434
6	2060	2136	373
7	1332	1351	534
8	732	785	992
	-2, 2, 1		
1	4783	4718	140
2	2435	2337	350
4	2786	2813	446
5	2402	2377	339
6	2048	2103	399
7	1352	1377	565
9	655	724	1192
	-2, 3, 1		
4	3042	3048	403
7	1764	1781	475
	-2, 4, 1		
1	2940	2825	212
2	5278	5298	277
3	4370	4476	339
4	3310	3290	423
	-2, 5, 1		
1	907	912	248
3	1429	1491	288
4	1373	1465	359
5	1395	1353	422
6	887	800	762
7	1223	1219	651
9	1475	1337	596
	-2, 6, 1		
1	1420	1375	201
2	1206	1213	285

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
	-2, 6, 1		
3	6261	6406	321
4	774	787	560
5	959	1028	565
6	1044	1030	633
	-2, 7, 1		
3	1659	1627	263
5	812	935	648
6	965	1075	679
7	1024	1101	765
8	1410	1253	633
9	839	722	1080
	-2, 8, 1		
1	2064	2014	313
2	1062	1064	323
3	2035	2125	327
4	2522	2531	406
5	1004	982	540
6	759	738	787
7	1555	1461	513
8	795	873	1085
	-2, 9, 1		
1	1486	1434	273
6	1603	1644	427
7	1277	1252	599
8	938	985	871
	-2, 10, 1		
1	1007	1022	382
2	1507	1500	292
4	966	952	508
5	993	925	536
6	1140	1076	582
7	1442	1454	546
	-2, 11, 1		
1	1474	1492	251
3	716	728	623
5	874	931	621
6	2381	2334	344
	-2, 12, 1		
1	3697	3578	402
2	1253	1251	381
3	574	644	788
5	945	836	577
6	949	1107	725
	-2, 13, 1		
1	1889	1820	314
3	524	529	897
5	1581	1551	425
6	718	836	901
7	1426	1364	533
	-2, 14, 1		
1	4191	4039	432
2	1290	1284	425
4	1378	1494	460
5	722	813	838
6	589	510	1019

l	kF_o	E_c	σ
	-2, 15, 1		
1	1949	1929	336
2	821	737	635
5	832	898	750
	-2, 16, 1		
1	1603	1542	375
2	1185	1157	519
3	1594	1689	424
5	929	997	703
6	795	805	845
	-2, 17, 1		
1	1574	1609	420
	-2, 18, 1		
2	695	714	835
3	776	677	781
4	1371	1292	506
5	875	924	796
	-2, 19, 1		
1	1106	1128	572
	-2, 20, 1		
1	1080	1016	583
3	1470	1415	494
	-1, 0, 1		
1	3962	3978	205
3	601	638	668
5	3376	3678	439
9	944	837	747
	-1, 1, 1		
1	6712	6757	194
2	2057	2000	300
3	885	889	547
5	2474	2500	386
6	563	604	1034
9	939	1017	838
	-1, 2, 1		
1	445	497	487
2	993	954	350
3	2567	2577	397
4	2495	2533	391
5	1921	1949	369
6	1446	1442	519
7	935	715	749
	-1, 3, 1		
1	4360	4185	120
2	4578	4638	261
4	1652	1676	336
5	3133	3117	460
7	1128	1185	693
	-1, 4, 1		
1	1410	1332	218
2	2742	2661	309
3	3468	3605	342
4	703	804	611
5	1247	1223	478
8	1115	1003	737
	-1, 5, 1		

l	kF_o	F_c	σ
1	996	983	290
2	6378	6421	264
3	966	970	360
4	2724	2764	390
5	1795	1767	346
6	1498	1458	493
	-1, 6, 1		
2	4238	4212	297
3	528	544	608
4	638	626	626
6	854	819	749
7	1572	1537	544
8	1417	1381	685
	-1, 7, 1		
2	4773	4768	304
3	1453	1433	277
4	2398	2453	395
6	1374	1385	483
9	1200	1042	805
	-1, 8, 1		
1	2161	2070	352
3	1161	1163	344
4	895	831	485
5	1112	1065	470
6	781	856	764
7	2573	2556	383
	-1, 9, 1		
1	1775	1715	280
2	2874	2791	374
5	655	663	800
6	666	587	879
8	858	706	896
	-1, 10, 1		
1	797	789	468
2	1190	1183	346
3	1148	1173	379
5	2814	2936	487
6	1267	1279	537
7	1321	1325	574
8	1095	1077	747
	-1, 11, 1		
1	2034	1984	305
3	1508	1494	343
6	995	894	641
	-1, 12, 1		
1	1025	978	436
2	1253	1227	384
3	1739	1714	326
4	2690	2755	426
5	2602	2666	403
8	943	848	840
	-1, 13, 1		
1	1153	1153	417
2	1087	1094	450
3	814	806	602
5	746	763	810

l	kF_o	F_c	σ
6	1693	1685	436
	-1, 14, 1		
1	1851	1755	311
2	975	998	529
4	3088	3118	461
7	693	806	1059
	-1, 15, 1		
2	1394	1345	416
	-1, 16, 1		
1	1173	1179	466
2	870	885	650
3	656	581	893
4	956	954	643
5	1208	1257	567
6	888	870	776
	-1, 18, 1		
1	928	875	611
4	758	847	898
	-1, 19, 1		
1	704	721	828
	-1, 20, 1		
1	732	661	790
2	1350	1406	525
	0, 0, 1		
2	550	525	547
4	659	627	572
6	887	985	630
8	2258	2392	396
	0, 1, 1		
1	691	679	284
2	1164	1039	358
5	1266	1241	457
6	1700	1759	413
	0, 2, 1		
0	8313	7573	106
1	4868	4769	118
2	1722	1736	232
3	803	758	562
4	2646	2597	374
5	1290	1266	505
7	1033	981	720
8	1407	1336	611
9	1224	1319	724
	0, 3, 1		
1	1285	1230	188
2	1535	1529	226
3	1710	1700	259
4	753	814	594
5	1956	2000	373
8	1402	1327	627
	0, 4, 1		
0	2234	1993	211
1	1887	1850	276
2	2271	2265	331
3	3989	4144	322
4	3498	3675	397

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	k	$10F_o$	$10F_c$	100σ
		0, 4, 1		
6	1	1519	1463	514
9	1	1114	1072	778
		0, 5, 1		
1	1	1348	1277	224
2	1	2156	2071	299
3	1	2663	2648	367
5	1	792	814	639
6	1	1596	1568	462
9	1	1302	1168	732
		0, 6, 1		
0	1	1439	1306	228
1	1	1609	1549	224
2	1	2754	2773	330
3	1	5917	6015	311
4	1	571	542	693
5	1	1761	1837	336
		0, 7, 1		
1	1	609	633	507
2	1	2641	2646	352
3	1	1294	1452	326
4	1	1389	1455	327
6	1	1764	1788	395
8	1	1253	1151	672
		0, 8, 1		
1	2	2716	2697	348
2	2	3820	3935	337
3	1	1944	1941	315
6	1	1098	1163	572
7	1	708	602	955
9	1	1152	1105	833
		0, 9, 1		
1	2	1246	1193	293
2	2	1890	1957	301
3	2	958	1005	432
4	2	1407	1480	360
6	2	2309	2301	342
8	2	982	1008	792
		0, 10, 1		
0	4	4911	4770	328
1	3	3343	3157	364
4	2	958	984	510
7	1	1200	1101	571
		0, 11, 1		
2	1	1042	1052	408
3	2	2097	2075	329
5	1	1103	1166	532
6	2	2865	2721	428
		0, 12, 1		
0	3	3901	3748	262
1	1	1438	1333	312
3	2	698	731	682
4	2	863	768	593
5	2	2139	2175	350
8	2	662	693	1140
		0, 13, 1		
1	1	641	638	663
2	1	901	868	526
3	1	2416	2376	372
5	1	2285	2275	355
6	1	1230	1153	545
7	1	1573	1501	497
		0, 14, 1		
0	2	696	661	624
1	2	1110	1168	446
2	2	1121	1185	473
4	2	1388	1431	475
5	2	2018	2037	398
6	2	622	623	1021
		0, 15, 1		
3	2	2650	2691	416
5	2	1409	1494	509
7	2	1053	911	690
		0, 16, 1		
0	3	1299	1381	424
3	3	1622	1635	430
4	3	1235	1140	528
		0, 17, 1		
1	3	1192	1160	504
3	3	2165	2230	365
5	3	729	720	905
6	3	1092	1002	667
		0, 18, 1		
1	4	917	875	635
2	4	1762	1829	399
3	4	960	765	618
		0, 19, 1		
1	4	976	963	620
2	4	1155	1110	555
3	4	769	782	830
4	4	1123	1096	617
		0, 20, 1		
2	4	1369	1345	508
3	4	1109	1183	640
		0, 21, 1		
1	5	898	856	701
2	5	852	895	775
		1, 0, 1		
1	5	3784	3515	158
3	5	2687	2838	371
5	5	1695	1808	318
7	5	824	836	743
9	5	1410	1490	593
		1, 1, 1		
0	6	675	196	113
1	6	5233	4991	174
2	6	917	828	397
3	6	2448	2250	355
4	6	673	701	718
7	6	1577	1736	486
9	6	856	929	931
		1, 2, 1		
0	7	3930	3639	108
1	7	448	444	451
2	7	1406	1370	227
3	7	613	566	680
4	7	4167	4041	446
7	7	1846	1819	480
9	7	931	829	882
		1, 3, 1		
0	8	572	565	223
1	8	2647	2411	243
2	8	1994	1984	316
3	8	1271	1251	309
4	8	1055	1028	423
5	8	1000	968	601
7	8	2699	2528	401
		1, 4, 1		
0	9	620	579	248
2	9	2895	2817	301
3	9	1732	1746	281
4	9	2686	2652	386
5	9	2489	2484	373
6	9	2044	1919	402
7	9	1669	1408	566
		1, 5, 1		
2	10	1811	1715	232
3	10	2099	2068	295
4	10	871	831	457
5	10	584	643	839
6	10	1411	1491	505
7	10	1322	1339	628
8	10	1839	1731	576
		1, 6, 1		
0	11	666	652	301
1	11	2670	2477	315
2	11	3455	3412	315
4	11	704	714	568
5	11	1241	1267	420
6	11	2884	2857	410
7	11	930	846	796
8	11	1170	936	739
		1, 7, 1		
2	12	2515	2583	370
3	12	3062	3103	371
5	12	744	707	644
6	12	1225	1246	541
7	12	911	855	775
8	12	1226	1154	683
		1, 8, 1		
0	13	1396	1333	180
1	13	5356	5141	298
2	13	949	964	380
3	13	597	724	654
4	13	1795	1784	295
5	13	1118	1085	465
6	13	809	946	737
7	13	2061	2099	418
		1, 9, 1		
0	14	1009	1005	244
1	14	2591	2554	380
2	14	2775	2759	395
3	14	1803	1831	296
4	14	1478	1480	353
		1, 10, 1		
0	15	3378	3272	250
1	15	1715	1713	278
2	15	3385	3332	388
3	15	1769	1731	297
5	15	2778	2762	424
7	15	1022	1057	685
8	15	831	770	896
		1, 11, 1		
0	16	1871	1821	193
1	16	2994	2986	385
2	16	1257	1269	361
4	16	1225	1234	438
6	16	885	695	696
		1, 12, 1		
2	17	1099	1079	418
3	17	2324	2356	356
4	17	3313	3386	456
5	17	2236	2304	387
		1, 13, 1		
0	18	1559	1547	226
1	18	1708	1703	312
6	18	1261	1204	542
		1, 14, 1		
1	19	1545	1611	348
2	19	1556	1481	359
4	19	3880	4006	520
6	19	607	348	1030
7	19	950	822	749
		1, 15, 1		
0	20	1440	1542	296
5	20	708	880	921
		1, 16, 1		
0	21	2327	2332	224
2	21	2788	2795	406
4	21	1693	1659	434
5	21	943	1039	737
		1, 17, 1		
0	22	898	1125	621
1	22	1210	1241	508
3	22	999	1070	647
4	22	1272	1384	554
		1, 18, 1		
0	23	2163	2129	246
1	23	2232	2273	347
2	23	1593	1557	425
4	23	1088	1065	611
		1, 19, 1		
1	24	1275	1373	541
3	24	1158	1248	599

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$, $10F_c$, 100σ, * for Insignificant

l	kF_o	F_c	σ
	1, 20, 1		
-1	2018	2061	393
	1, 21, 1		
1	1295	1275	542
	2, 0, 1		
0	8852	8797	136
2	2980	2807	251
4	1188	1291	346
6	1494	1634	410
8	2168	2234	399
	2, 1, 1		
2	3281	3262	278
3	676	755	835
4	2817	2826	428
5	1684	1756	384
6	1924	2004	388
8	996	994	767
	2, 2, 1		
0	3138	2985	144
1	3175	2955	218
2	722	727	415
3	839	897	466
6	2490	2425	404
8	1240	1225	714
9	1243	1124	687
	2, 3, 1		
0	913	881	174
2	2054	2028	276
3	994	1016	371
4	1902	1959	306
5	3091	3120	453
8	1919	1733	538
	2, 4, 1		
2	900	896	378
3	1359	1393	285
4	2480	2606	382
5	1189	1159	462
6	2414	2516	367
9	1073	976	896
	2, 5, 1		
0	2585	2438	159
3	3104	3240	369
4	1129	1141	387
5	2281	2336	334
7	1498	1536	578
8	1331	1180	715
9	992	902	1046
	2, 6, 1		
0	362	398	609
1	1420	1345	199
2	3045	3032	352
3	2099	2040	290
5	2840	2877	426
7	980	919	765
9	961	709	1020
	2, 7, 1		
0	2703	2684	226
1	1343	1268	264
2	3017	2956	350
3	1558	1510	289
4	1484	1490	328
5	2266	2364	347
7	899	856	778
	2, 8, 1		
1	2279	2217	310
2	4231	4229	344
3	1017	944	399
4	1048	1013	433
5	1070	1063	511
6	959	1070	641
	2, 9, 1		
0	2964	2898	249
1	1063	1086	340
2	3452	3486	376
3	883	848	490
5	2190	2154	318
6	1217	1247	516
	2, 10, 1		
0	4075	3962	246
1	4226	4147	350
2	1264	1324	352
3	2034	2052	313
4	1109	1093	474
8	1336	1379	613
	2, 11, 1		
0	1985	2019	184
1	1424	1472	337
2	2578	2585	400
3	2196	2173	344
4	671	666	764
6	2019	1941	387
	2, 12, 1		
0	3758	3594	220
1	870	887	502
4	1050	1021	526
5	1126	1162	576
7	925	868	764
8	1408	1198	566
	2, 13, 1		
1	969	1027	476
2	1550	1537	341
3	2516	2508	388
5	1594	1629	455
6	985	1031	672
7	700	792	1032
	2, 14, 1		
1	566	573	826
2	1068	1070	468
4	1123	1117	544
5	895	1030	751
6	901	724	725
7	1059	1000	699
	2, 15, 1		
0	791	747	431
3	2985	2962	435
5	1229	1238	567
	2, 16, 1		
0	1173	1223	330
1	2064	2013	317
3	1941	2005	388
4	1165	1157	560
5	659	544	962
	2, 17, 1		
0	1378	1385	316
2	1122	1086	554
3	2503	2558	396
	2, 18, 1		
1	1367	1312	457
2	2138	2289	378
3	1405	1452	500
	2, 19, 1		
0	1468	1377	312
2	1789	1846	438
3	928	840	674
	2, 20, 1		
1	748	852	845
2	1788	1779	432
	2, 21, 1		
0	1151	1273	443
2	1142	1207	614
	3, 0, 1		
1	800	880	405
3	7479	7245	264
5	722	721	651
7	926	1182	689
9	780	679	894
	3, 1, 1		
0	572	527	323
2	1285	1308	230
3	1128	1103	469
4	818	856	642
5	2289	2397	350
7	1432	1544	510
9	798	962	987
	3, 2, 1		
0	1108	1020	154
1	2812	2681	185
2	4026	3819	183
3	4046	4182	340
4	3841	3866	411
5	677	556	1047
7	1828	1819	511
	3, 3, 1		
0	1203	1196	145
1	1188	1100	190
2	830	783	295
4	1849	1910	281
5	546	601	999
7	2000	1905	572
	3, 4, 1		
0	3912	3820	140
1	2995	2873	192
4	3117	3078	401
5	1968	2019	324
6	2148	2146	378
7	2066	1959	500
	3, 5, 1		
0	604	553	284
1	1828	1776	228
2	800	776	414
3	697	694	515
4	1264	1275	361
6	1829	1903	410
8	870	951	1074
9	941	849	1123
	3, 6, 1		
0	5252	5149	149
1	2081	2117	290
2	1692	1682	261
3	860	849	439
4	1627	1643	309
6	2754	2864	432
8	943	865	887
	3, 7, 1		
1	2153	2170	344
2	978	945	368
3	1689	1596	282
4	1645	1720	334
5	1443	1483	400
6	1195	1108	539
7	1529	1559	492
9	666	454	1287
	3, 8, 1		
0	603	635	347
1	4805	4923	321
2	2138	2110	305
4	3350	3352	453
5	1015	964	532
6	731	827	831
	3, 9, 1		
0	852	827	272
1	2359	2393	356
4	2036	2058	346
7	1203	1276	615
	3, 10, 1		
0	1173	1123	237
1	2331	2328	318
2	3413	3323	385
3	2665	2644	383
4	1604	1663	384
5	1105	972	519
	3, 11, 1		
0	2633	2527	254
1	2139	2135	333

Table F.3. Obs and Calcd Structure Factors for $(\eta^3\text{-}C_5Me_5)IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ	l	kF_o	F_c	σ	l	kF_o	F_c	σ	l	kF_o	F_c	σ
	3, 11, 1			2	7315	7476	215		4, 9, 1				5, 0, 1		
3	551	579	843	3	490	576	869	0	2424	2461	248	1	1597	1538	348
4	742	807	725	4	2835	3005	423	1	1745	1756	255	3	6249	6562	365
6	947	932	697	5	1488	1636	418	2	2802	2873	392	5	1757	1713	318
7	860	688	786	6	1963	2211	382	4	1016	1053	544	7	635	536	962
	3, 12, 1				4, 2, 1			5	2347	2346	337	9	1075	906	696
0	863	932	361	0	1221	1189	199	7	914	958	790		5, 1, 1		
1	1255	1253	373	1	1424	1402	246		4, 10, 1			0	761	683	392
2	777	773	580	2	1018	981	346	0	2070	1997	213	1	1758	1830	286
3	2566	2615	398	3	2467	2545	342	1	1683	1671	281	3	878	835	599
4	1367	1381	440	6	1212	1192	783	2	482	594	878	4	1059	1086	566
5	1453	1405	472	7	1528	1451	611	3	1981	2017	306	8	872	719	810
	3, 13, 1				4, 3, 1			5	1398	1454	478	9	1109	1056	710
0	3057	3079	305	0	1219	1182	185	8	1844	1781	520		5, 2, 1		
1	942	908	506	1	2639	2554	253		4, 11, 1			1	3723	3741	339
2	565	609	823	2	5673	5681	283	0	1893	1887	199	2	3305	3379	354
7	816	571	848	4	932	879	471	1	1150	1161	387	3	3905	3904	361
	3, 14, 1			5	2624	2720	386	2	1946	1967	300	4	2563	2604	373
1	1700	1749	342		4, 4, 1			4	1797	1778	360	5	605	648	934
2	866	900	595	0	4040	3925	175	5	705	697	905	6	1022	1063	988
4	2216	2212	356	1	1057	936	264	6	636	635	1039	9	1083	1210	875
	3, 15, 1			2	1120	1117	259		4, 12, 1				5, 3, 1		
0	2826	2850	319	3	1287	1351	289	0	2163	2126	238	0	2739	2641	258
5	880	820	746	4	602	669	717	1	682	749	662	1	1488	1479	262
6	1323	1228	580	5	2089	2242	327	2	1066	1087	465	2	910	915	379
	3, 16, 1			6	1704	1733	429	5	1037	962	638	3	893	791	415
0	1915	1985	248	7	878	811	936	7	1701	1646	507	4	1618	1675	323
2	2390	2495	395		4, 5, 1				4, 13, 1			6	887	879	793
3	999	998	606	0	2581	2535	233	0	427	449	756	8	1252	1282	936
	3, 17, 1			1	3115	3191	313	2	1576	1597	368		5, 4, 1		
0	1830	1798	267	2	1266	1286	277		4, 14, 1			0	6008	5970	205
1	624	851	975	3	2132	2164	324	1	939	892	537	1	3864	3848	312
4	782	862	816	5	2071	2129	333	2	941	1064	572	2	901	909	361
	3, 18, 1			7	825	930	913	4	1156	1172	554	4	1447	1527	367
0	1215	1214	357	8	769	789	1169	5	821	876	798	7	1140	1217	749
1	2697	2697	393		4, 6, 1				4, 15, 1			9	1107	998	1021
2	1603	1629	446	0	1060	1050	211	4	912	825	637		5, 5, 1		
5	650	595	1025	1	2401	2461	358		4, 16, 1			0	978	909	239
	3, 19, 1			2	1163	1227	307	0	676	696	591	1	894	895	355
0	653	768	813	4	2026	2079	310	1	863	965	694	2	1220	1211	293
2	984	883	655	5	2993	3101	460	2	753	797	746	3	1580	1561	288
3	711	555	861	8	755	876	1082	3	1739	1830	416	4	981	946	503
	3, 20, 1				4, 7, 1			5	1978	1964	435	6	2429	2476	345
0	811	845	545	0	2297	2355	229		4, 17, 1				5, 6, 1		
1	2124	2256	415	1	2904	2994	346	0	1100	1096	394	0	7811	7886	212
	3, 21, 1			2	1791	1850	244	2	862	852	684	2	708	740	505
1	1196	1333	629	3	1791	1701	288	4	585	644	1059	3	791	831	509
	4, 0, 1			5	2211	2324	337		4, 18, 1			5	1758	1742	399
0	1127	1051	235	6	1319	1401	517	2	1014	1017	626	6	945	1024	696
2	4449	4392	250		4, 8, 1			3	1559	1548	471	8	931	1081	914
4	1866	1837	263	1	676	698	476	4	1304	1312	561		5, 7, 1		
6	523	385	1031	2	2330	2351	351		4, 19, 1			0	630	519	365
8	1468	1546	518	3	1697	1690	304	0	999	1077	451	1	709	665	468
	4, 1, 1			4	2758	2784	396	2	1397	1429	505	2	1256	1329	329
0	1280	1257	212	6	1104	1037	586		4, 20, 1			3	1211	1300	386
1	461	445	790	8	931	940	845	1	1405	1470	515	5	1732	1843	421

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
5, 7, 1			
6	1294	1259	544
7	1742	1803	483
5, 8, 1			
0	2974	2932	261
1	2570	2694	363
2	1180	1214	358
4	2506	2657	383
5	2101	2239	385
5, 9, 1			
3	1570	1642	368
5	1447	1434	464
6	970	921	703
7	1124	1228	698
5, 10, 1			
1	541	574	695
2	1110	1074	414
3	2676	2644	377
4	2238	2376	344
6	784	886	870
5, 11, 1			
0	532	434	534
1	922	950	487
2	989	1061	502
3	2205	2311	332
4	532	702	1046
5	619	487	1001
6	1652	1710	511
5, 12, 1			
0	987	1000	336
2	1715	1785	349
3	2536	2676	363
5, 13, 1			
0	842	826	407
2	1635	1669	383
3	762	902	750
4	1757	1825	418
5	612	633	1015
6	1563	1604	532
5, 14, 1			
1	1405	1457	406
2	1910	2003	357
5, 15, 1			
2	1325	1364	486
4	967	1008	660
5	710	810	958
6	2175	2126	445
5, 16, 1			
0	1954	1925	261
1	618	629	922
3	1083	1273	600
4	824	882	789
5, 17, 1			
2	1055	1093	610
3	923	919	683
5, 18, 1			
0	884	947	509
1	1548	1589	461
4	578	589	1121
5, 19, 1			
2	1271	1337	557
3	774	825	857
5, 20, 1			
0	1440	1474	362
1	1002	1028	679
6, 0, 1			
2	3170	3098	429
6	2551	2475	374
8	947	866	763
6, 1, 1			
0	1072	1063	292
2	3868	3838	420
3	708	692	830
4	1114	1012	672
5	690	741	806
6	1014	977	608
7	760	682	877
8	1584	1695	511
6, 2, 1			
0	1963	1935	199
1	2136	2173	329
3	1533	1617	346
4	1181	1231	445
6	1229	1111	737
7	1607	1764	645
6, 3, 1			
0	608	637	431
1	2180	2204	301
2	3749	3822	371
4	1237	1266	402
5	966	953	608
7	1258	1250	744
6, 4, 1			
0	3554	3537	250
2	2673	2695	364
4	1329	1354	377
5	1596	1637	424
7	1328	1496	629
6, 5, 1			
0	919	966	278
1	3828	3889	349
2	1480	1477	289
3	883	920	479
4	1377	1429	391
6	632	526	1038
6, 6, 1			
1	2154	2134	325
2	964	940	405
3	1580	1640	321
4	2247	2376	323
5	1971	2088	396
6	817	809	831
6, 7, 1			
0	1325	1319	221
1	2881	3023	391
2	971	1047	427
3	1296	1332	384
6	1717	1815	479
7	903	897	815
6, 8, 1			
0	1152	1174	251
2	508	513	761
3	565	477	774
4	3497	3876	500
7	1155	1133	678
6, 9, 1			
1	825	755	470
2	1434	1431	343
3	874	814	557
4	1025	1070	602
6	816	829	877
7	1487	1604	594
6, 10, 1			
0	1473	1482	235
2	2575	2681	346
3	872	886	600
4	1942	2020	387
5	1910	1985	427
6, 11, 1			
1	653	727	690
2	790	829	629
3	1632	1710	398
4	1563	1679	448
7	795	830	989
6, 12, 1			
0	1654	1670	240
1	2450	2578	371
2	1890	1991	342
3	920	914	628
4	935	997	690
5	1408	1430	522
6	928	936	776
7	965	1066	867
6, 13, 1			
1	1356	1370	427
2	889	941	629
3	2113	2154	370
6, 14, 1			
1	2719	2802	370
2	825	986	727
3	662	746	934
4	1264	1285	529
5	1505	1590	517
6, 15, 1			
1	1873	1858	388
3	1340	1339	505
4	856	918	773
6, 16, 1			
0	589	485	662
2	1424	1569	496
3	1046	1074	633
5	2208	2357	447
6, 17, 1			
1	1241	1355	563
4	676	719	1000
6, 18, 1			
0	1008	1011	459
1	793	767	785
3	912	970	766
7, 0, 1			
3	2079	2049	367
5	2073	2115	353
7	1353	1230	525
7, 1, 1			
1	618	699	788
2	1469	1435	413
3	1498	1623	479
5	2118	2152	448
7	1464	1429	509
8	772	853	970
7, 2, 1			
0	543	435	554
1	2143	2177	316
2	2328	2356	355
3	600	630	862
4	1539	1591	411
5	878	873	710
6	1665	1635	430
7, 3, 1			
1	497	531	871
2	2013	2023	311
5	2554	2542	368
6	1041	899	677
8	1364	1469	895
7, 4, 1			
0	4005	3892	271
1	3786	3661	371
2	690	800	633
3	1955	1998	360
6	1186	1029	631
8	1026	1114	879
7, 5, 1			
1	1013	931	408
2	1317	1326	351
3	1303	1255	370
4	1279	1364	441
5	838	836	677
6	2589	2449	370
7, 6, 1			
0	5609	5540	252
2	906	950	477
4	1315	1272	409
5	1165	1212	538
8	1478	1344	604

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
	7, 7, 1		
0	631	571	565
1	1331	1376	348
2	828	845	519
3	1784	1815	315
4	1181	1180	474
5	1636	1530	417
6	1542	1429	498
7	992	993	797
8	877	789	960
	7, 8, 1		
0	1310	1329	257
1	1002	1026	440
2	504	371	845
4	692	671	773
5	1641	1553	447
6	1490	1437	513
	7, 9, 1		
0	1505	1562	241
1	1163	1166	400
2	832	777	551
3	2597	2635	380
5	1804	1756	436
	7, 10, 1		
0	958	1007	345
1	1047	1173	476
3	1805	1757	353
4	1504	1419	445
6	1308	1266	579
	7, 11, 1		
0	1736	1743	236
2	787	743	636
3	3021	3053	421
4	1190	1118	536
5	865	803	762
6	649	581	1080
	7, 12, 1		
1	1277	1238	412
2	3103	3068	430
3	1654	1633	428
	7, 13, 1		
0	1636	1657	260
2	1699	1734	425
3	1107	1074	568
4	1938	1868	414
5	810	770	842
6	612	452	1149
	7, 14, 1		
0	584	585	667
1	1070	1051	565
2	3306	3349	497
	7, 15, 1		
0	1819	1815	272
2	1332	1385	538
4	938	882	707
	7, 16, 1		

l	kF_o	F_c	σ
0	2133	2185	263
1	1302	1270	514
2	1036	1043	639
3	946	919	696
4	981	888	709
	7, 17, 1		
0	1367	1339	367
1	995	943	657
2	931	879	741
3	1210	1178	584
	7, 18, 1		
0	1120	1065	430
	7, 19, 1		
1	953	951	750
	8, 0, 1		
0	2808	2792	302
2	1096	1170	584
4	2319	2392	407
6	2882	2879	425
8	820	748	935
	8, 1, 1		
0	509	512	835
2	890	979	728
4	1464	1431	619
7	931	926	857
8	1353	1355	614
	8, 2, 1		
0	1079	1078	341
1	3428	3347	468
2	889	905	646
4	3085	3267	477
5	985	870	718
6	1514	1525	553
7	1043	1313	661
	8, 3, 1		
1	1071	915	458
4	2142	2176	350
5	605	608	972
7	1721	1572	528
	8, 4, 1		
0	1271	1215	285
1	1914	1880	306
2	3288	3258	449
3	2561	2486	386
4	2793	2713	404
7	1349	1132	617
	8, 5, 1		
0	1442	1419	259
1	1276	1327	389
4	1078	958	514
5	1936	1937	372
7	1025	821	779
	8, 6, 1		
0	987	1005	343
2	1352	1311	380
3	3815	3782	437

l	kF_o	F_c	σ
4	969	888	559
5	1253	1139	516
7	924	852	869
	8, 7, 1		
0	2344	2304	239
1	732	721	619
2	917	906	525
3	662	568	745
5	1303	1361	514
7	992	875	815
	8, 8, 1		
0	1131	1153	317
1	1624	1657	338
3	1373	1389	418
4	2840	2799	430
7	1510	1374	585
	8, 9, 1		
0	2485	2434	243
1	669	771	730
2	672	676	732
7	978	975	869
	8, 10, 1		
0	1252	1278	301
2	2203	2269	324
4	1656	1566	423
5	1164	1095	594
6	1105	1043	711
	8, 11, 1		
0	1503	1531	276
1	1750	1775	345
4	863	944	743
6	1227	1113	645
	8, 12, 1		
0	1510	1536	279
1	2401	2385	375
2	1771	1733	386
3	775	900	797
4	823	742	742
6	1029	947	748
	8, 13, 1		
0	683	759	586
1	1970	1990	370
3	1383	1375	497
5	923	915	799
	8, 14, 1		
1	2954	2973	426
4	1204	1131	588
	8, 15, 1		
1	2377	2350	360
2	682	616	924
	8, 16, 1		
1	896	962	740
2	1180	1350	607
3	968	930	716
	8, 17, 1		
0	1120	1056	422

l	kF_o	F_c	σ
1	1841	1886	437
	8, 18, 1		
0	954	982	513
	9, 0, 1		
1	2811	2896	418
3	940	934	764
5	2004	2019	481
	9, 1, 1		
1	2688	2615	373
2	1859	1856	431
3	2215	2241	430
5	2163	2297	532
	9, 2, 1		
0	1010	1008	396
1	802	708	728
2	1967	1930	395
3	2072	2064	399
4	905	989	826
6	1034	1257	884
	9, 3, 1		
0	939	905	427
1	1783	1789	385
2	3536	3623	519
4	866	899	743
5	2655	2735	373
	9, 4, 1		
0	879	869	453
1	1756	1688	370
3	2291	2348	338
6	656	678	1027
	9, 5, 1		
0	1340	1287	298
1	1590	1577	374
2	3362	3359	483
4	2003	1970	357
5	1424	1279	491
6	1405	1384	528
	9, 6, 1		
0	1711	1696	250
2	1376	1401	430
4	1557	1598	428
7	932	940	864
	9, 7, 1		
0	491	583	724
1	1109	1081	469
2	2975	3059	431
4	1368	1308	476
5	783	694	799
6	1308	1253	565
	9, 8, 1		
0	1028	1118	369
1	700	683	698
3	738	749	721
6	961	991	776
7	1429	1413	656
	9, 9, 1		

Table F.3. Obs and Calcd Structure Factors for (η^5-C_5Me_5)$IrOs_3(CO)_{12}$.

Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF_o	F_c	σ
	9, 9, 1		
0	859	883	437
2	2116	2197	345
3	936	900	611
5	1139	1076	593
	9, 10, 1		
0	2046	2024	234
1	1014	1004	518
3	828	855	730
5	1479	1405	509
	9, 11, 1		
0	1471	1470	301
1	747	750	752
3	1657	1754	423
6	765	665	1012
	9, 12, 1		
1	979	994	609
2	1635	1615	406
3	988	987	631
4	1125	1175	614
5	1403	1266	549
	9, 13, 1		
0	1701	1699	283
2	1252	1220	531
3	606	590	988
5	730	549	985
	9, 14, 1		
1	1051	1027	591
2	1640	1653	444
4	1287	1148	561
	9, 15, 1		
0	1829	1841	294
2	956	843	668
	9, 16, 1		
0	2109	2143	282
1	819	866	798
	9, 17, 1		
0	1069	1198	481
	10, 0, 1		
0	5285	5340	341
2	1071	1014	665
4	1197	1294	743
6	1328	1456	690
	10, 1, 1		
0	1082	1088	435
2	2210	2249	420
	10, 2, 1		
0	3512	3448	359
1	3300	3180	496
2	602	631	1201
4	2493	2480	434
5	1039	1111	871
7	734	612	1397
	10, 3, 1		
0	978	926	447
1	577	673	1128

l	kF_o	F_c	σ
2	1024	953	670
4	1286	1220	598
7	868	938	915
	10, 4, 1		
0	553	525	926
1	2274	2270	348
2	1415	1445	494
3	2643	2704	399
4	2363	2361	369
6	647	535	1064
	10, 5, 1		
2	802	740	742
6	1151	1045	638
	10, 6, 1		
0	1076	1110	384
1	1343	1315	455
3	3437	3484	494
5	773	694	870
	10, 7, 1		
0	1291	1322	337
2	1000	1034	591
4	1336	1264	493
6	1011	861	711
	10, 8, 1		
1	2387	2335	375
2	1517	1490	433
3	1341	1274	496
4	1134	1036	556
	10, 9, 1		
0	1241	1232	339
1	616	714	926
3	1104	1138	567
4	1147	1194	581
6	1619	1515	530
	10, 10, 1		
0	1983	1962	262
1	1747	1716	403
	10, 11, 1		
3	1149	1042	551
	10, 12, 1		
0	2245	2220	250
1	991	953	618
2	565	669	1077
5	1540	1521	537
	10, 14, 1		
1	1432	1466	489
	10, 15, 1		
1	887	889	713
3	1035	1073	721
	10, 16, 1		
0	754	767	618
	11, 0, 1		
1	2466	2528	375
3	893	881	903
5	1442	1435	688
	11, 1, 1		

l	kF_o	F_c	σ
1	2625	2536	410
2	1123	1194	734
3	1719	1674	567
	11, 2, 1		
0	1899	1903	296
1	1188	1103	644
2	950	792	820
3	719	690	1181
4	1585	1581	628
	11, 3, 1		
1	1151	1102	622
2	2561	2510	385
5	1016	937	774
	11, 4, 1		
3	1100	1097	667
5	1005	1053	744
	11, 5, 1		
0	1240	1240	387
2	2044	2060	409
3	705	741	937
4	1212	1244	606
	11, 6, 1		
1	1597	1625	442
2	1336	1344	537
5	928	951	787
	11, 7, 1		
0	1004	856	451
2	2120	2038	371
3	1230	1224	569
4	589	646	1130
5	846	741	852
	11, 8, 1		
0	1357	1278	348
1	1945	1841	381
3	1271	1298	540
5	1047	1016	694
	11, 9, 1		
1	1523	1499	463
2	1592	1587	440
4	988	904	677
5	626	513	1108
	11, 10, 1		
0	1527	1501	331
2	1615	1702	443
5	1928	1922	460
	11, 11, 1		
1	1677	1668	423
4	993	1053	730
	11, 12, 1		
4	1617	1662	515
	11, 14, 1		
1	691	727	928
	12, 0, 1		
0	3785	3962	389
2	1198	1173	683
	12, 1, 1		

l	kF_o	F_c	σ
0	927	873	558
4	969	928	942
6	1444	1603	815
	12, 2, 1		
0	2330	2280	282
1	1913	1875	504
3	685	713	1322
5	1035	827	925
6	1176	1035	903
	12, 3, 1		
0	1262	1249	425
1	776	683	1003
3	751	597	1045
5	1179	1279	764
	12, 4, 1		
1	936	816	815
3	880	845	861
4	1077	1043	734
	12, 5, 1		
0	1308	1248	402
3	1571	1458	505
	12, 6, 1		
2	1595	1502	489
3	1396	1455	559
	12, 7, 1		
0	687	549	831
2	932	917	747
3	800	835	865
4	1679	1651	506
5	884	932	865
	12, 8, 1		
1	1747	1750	446
2	2433	2422	371
	12, 9, 1		
0	866	792	669
2	777	660	826
3	652	636	1005
4	1155	1091	628
	12, 10, 1		
0	2180	2128	277
1	1799	1819	445
2	843	827	768
3	1125	1117	625
	12, 11, 1		
3	978	1128	764
	12, 12, 1		
0	1980	2006	303
	12, 13, 1		
1	1374	1341	543
	13, 0, 1		
3	2294	2478	461
5	882	968	1123
	13, 1, 1		
5	1460	1519	736
	13, 2, 1		
0	1024	982	514

Table F.3. Obs and Calcd Structure Factors for $(\eta^5\text{-}C_5Me_5)IrOs_3(CO)_{12}$.
Columns are $10F_o$ $10F_c$ 100σ, * for Insignificant

l	kF$_o$	F$_c$	σ
	13, 2, 1		
3	1328	1204	732
4	2078	2015	568
	13, 3, 1		
4	679	280	1275
	13, 4, 1		
1	1010	1050	833
4	1816	1706	510
	13, 5, 1		
1	1218	1172	651
2	650	342	1103
	13, 6, 1		
0	1257	1189	435
1	1145	1110	703
2	1229	1153	616
3	946	847	793
4	949	1113	858
	13, 7, 1		
1	1217	1108	615
3	1439	1293	561
	13, 8, 1		
1	2172	2088	401
3	768	875	964
	13, 9, 1		
0	695	537	681
1	1601	1657	513
3	1004	964	731
	13, 10, 1		
1	725	791	990
2	1691	1703	473
	13, 11, 1		
0	934	1040	554
1	1518	1564	533
	13, 12, 1		
0	992	961	518
	14, 0, 1		
0	1244	1255	443
4	1698	1726	600
	14, 1, 1		
2	1874	1910	531
4	1249	1269	796
	14, 2, 1		
3	1202	1071	825
4	763	824	1371
	14, 3, 1		
2	2133	1988	533
3	1020	882	941
	14, 4, 1		
0	1454	1436	409
	14, 5, 1		
0	641	736	988
1	758	810	1094
2	782	860	1085
3	1684	1745	577
	14, 6, 1		
1	1515	1403	584
2	1204	1095	700
	14, 7, 1		
0	772	752	701
1	949	873	851
2	914	699	863
	14, 8, 1		
2	1856	1748	497
	14, 9, 1		
0	1317	1310	449
	15, 0, 1		
1	1315	1290	640
	15, 2, 1		
1	1881	1841	573
	15, 3, 1		
0	769	884	743
1	756	682	1222
	15, 4, 1		
0	1332	1305	470
1	1930	1760	579
	15, 6, 1		
0	2234	2185	332

Table G.1. U_{ij} U_{iso} Values (*100) for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

	U11(U)	U22	U33	U12	U13	U23
Os(1)	2.27(3)	3.34(4)	2.98(4)	-0.92(3)	-0.684(25)	-0.02(3)
Os(2)	2.22(3)	3.21(4)	3.81(4)	-0.67(3)	-0.38 (3)	0.14(3)
Os(3)	2.80(3)	3.75(4)	3.11(4)	-1.06(3)	-0.89 (3)	-0.16(3)
Os(4)	3.07(4)	3.24(4)	4.98(4)	-0.50(3)	-1.18 (3)	-0.16(3)
Sn	4.73(7)	3.83(8)	6.58(9)	-0.87(6)	-0.94 (6)	-0.85(7)
O(11)	5.3 (8)	9.2 (13)	4.7 (8)	-2.8 (8)	-0.1 (6)	2.1 (8)
O(12)	3.0 (6)	5.3 (9)	6.3 (8)	-2.0 (6)	-0.4 (6)	0.8 (7)
O(13)	5.3 (8)	10.7 (14)	4.2 (8)	-2.2 (8)	-2.0 (6)	-0.7 (8)
O(21)	4.0 (7)	9.0 (13)	7.6 (10)	-2.3 (8)	2.4 (7)	-1.2 (9)
O(22)	4.4 (8)	9.1 (13)	8.7 (11)	-2.6 (8)	1.1 (7)	2.3 (10)
O(23)	5.9 (9)	7.1 (12)	9.6 (12)	-1.3 (8)	-4.3 (8)	2.7 (10)
O(31)	3.6 (7)	8.6 (12)	4.6 (8)	-2.3 (7)	-1.3 (6)	0.4 (7)
O(32)	4.8 (8)	7.5 (11)	4.8 (8)	-1.6 (7)	0.1 (6)	0.1 (8)
O(33)	3.7 (7)	11.3 (14)	3.9 (7)	-1.1 (7)	-2.0 (6)	0.4 (8)
O(34)	12.8 (14)	8.3 (14)	8.7 (12)	0.2 (11)	-4.0 (10)	-5.3 (11)
O(41)	5.2 (9)	9.3 (14)	12.6 (15)	-3.0 (9)	-0.3 (9)	5.9 (12)
O(42)	4.0 (7)	8.3 (12)	6.3 (9)	-1.1 (7)	-0.7 (6)	-1.4 (8)
O(43)	5.5 (8)	10.1 (14)	9.0 (11)	-2.6 (9)	-4.5 (8)	4.9 (10)
O(44)	8.5 (12)	9.2 (16)	17.8 (21)	0.1 (11)	-3.7 (12)	-6.3 (15)
C(11)	4.0 (4)					
C(12)	3.6 (4)					
C(13)	3.8 (4)					
C(21)	4.1 (4)					
C(22)	5.1 (5)					
C(23)	5.8 (5)					
C(31)	4.0 (4)					
C(32)	4.0 (4)					
C(33)	5.0 (5)					
C(34)	5.5 (5)					
C(41)	5.6 (5)					
C(42)	4.2 (4)					
C(43)	5.4 (5)					
C(44)	7.2 (7)					
C(1)	9.9 (9)					
C(2)	7.4 (7)					
C(3)	11.1 (10)					
H	5.0					

Table G.2. Methyl Hydrogen Atom Coordinates for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$.

Atom	*x/a*	*y/b*	*z/c*	U(iso)
H(1)	0.233	-0.177	0.617	10.9
H(2)	0.185	-0.038	0.595	10.9
H(3)	0.094	-0.094	0.687	10.9
H(4)	0.480	-0.329	0.757	8.4
H(5)	0.370	-0.267	0.846	8.4
H(6)	0.589	-0.283	0.826	8.4
H(7)	0.635	-0.211	0.578	11.8
H(8)	0.770	-0.150	0.620	11.8
H(9)	0.635	-0.077	0.550	11.8

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 1,-17			-2	215	212	425	0	409	224	585
-1	611	644	372	-1	1127	1158	249		H, 7,-13		
	H, 1,-16			0	873	883	307	-2	1048	1093	337
-3	327	224	294		H, 4,-14			-1	379	482	591
-2	360	356	336	-1	372	386	501	2	744	651	541
-1	211	109	406	0	1164	1187	310		H, 1,-12		
0	352	316	484	1	493	528	683	-5	713	707	261
2	1002	959	294		H, 5,-14			-3	1316	1399	200
	H, 2,-16			-3	717	659	301	-2	991	1034	202
-3	515	472	388	0	408	448	525	-1	330	333	376
	H, 3,-16			1	996	947	479	0	238	253	282
-1	370	397	368		H, 6,-14			2	803	812	236
0	546	540	447	2	557	619	727	3	342	336	415
	H, 1,-15				H, 1,-13			4	897	870	255
-4	606	570	313	-3	577	566	269		H, 2,-12		
-2	592	541	308	-2	991	990	220	-6	456	392	375
0	535	505	313	-1	498	493	281	-5	346	316	441
1	536	570	509	0	382	319	341	-2	1721	1738	189
3	1158	1148	264	1	841	840	268	-1	1031	1025	216
	H, 2,-15			3	674	657	282	0	218	166	325
-4	453	447	436		H, 2,-13			2	262	239	382
-3	442	432	372	-5	836	805	270	5	807	785	295
-1	682	727	319	-3	638	587	259		H, 3,-12		
0	346	361	433	-2	750	739	245	-4	556	557	306
2	422	511	464	-1	1185	1216	210	-3	414	414	371
	H, 3,-15			0	742	736	257	-1	1714	1783	208
-1	437	477	454		H, 3,-13			0	557	542	336
0	769	723	327	-4	434	362	378	1	405	368	599
1	509	589	679	-3	388	383	391	2	443	441	409
	H, 4,-15			-2	954	962	230	3	468	432	331
-1	393	396	465	-1	551	606	321	4	466	375	356
0	427	476	563	0	1266	1272	258		H, 4,-12		
1	831	775	492	1	401	436	567	-5	730	734	313
2	525	512	562	2	517	423	360	-4	722	727	308
	H, 5,-15				H, 4,-13			-3	376	372	382
-1	388	375	429	-4	276	252	468	-1	400	398	430
0	471	396	526	-2	568	527	303	0	1649	1664	260
1	511	496	748	-1	984	1051	272	2	757	684	336
	H, 1,-14			0	447	404	457	3	726	746	308
-5	1150	1171	250	1	767	815	456	4	758	746	297
-4	261	295	447	3	760	815	343		H, 5,-12		
-3	681	703	273	4	569	544	378	-4	820	795	290
-2	466	415	301		H, 5,-13			-3	1175	1176	248
0	314	314	288	-3	446	434	410	1	776	771	481
2	1195	1211	236	-1	471	415	386	3	949	890	280
	H, 2,-14			0	549	582	473	4	807	845	317
-4	1015	977	241	1	433	225	606		H, 6,-12		
-2	976	952	244	2	646	539	445	-3	816	863	327
-1	888	890	254	3	529	464	387	-2	1075	1040	277
0	356	395	467	4	912	932	319	0	626	633	433
2	278	259	421		H, 6,-13			3	319	240	401
3	773	781	289	-3	1146	1192	296	4	578	662	455
	H, 3,-14			-2	487	524	497		H, 7,-12		
-3	452	456	406	-1	391	395	577	-3	580	528	394

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 7,-12			-1	283	278	435	-4	715	740	281
-2	785	790	361	0	422	382	601	-3	263	260	455
-1	582	675	453	1	1030	912	538	-2	336	299	388
1	1016	876	567	4	522	531	435	0	803	840	326
3	422	384	624	5	797	810	320	1	586	622	608
	H, 8,-12				H, 7,-11			2	1829	1802	235
-1	575	629	521	-3	648	701	363	3	719	750	273
2	1263	1131	446	-2	558	614	447	4	708	700	271
	H, 1,-11			0	544	604	543	5	272	189	364
-6	766	785	277	1	580	442	634	6	399	480	483
-4	1241	1231	193	2	963	958	458		H, 5,-10		
-3	843	833	202		H, 8,-11			-5	876	873	292
-2	1556	1593	172	-2	443	383	532	-4	1035	1075	260
-1	514	531	249	1	782	805	779	-3	722	715	266
0	510	550	253	2	656	603	623	-1	1258	1301	241
2	274	270	419	3	947	881	394	0	353	241	305
5	1653	1645	230		H, 9,-11			2	421	424	366
	H, 2,-11			2	926	899	583	3	1188	1195	241
-4	261	278	255		H, 1,-10			4	621	673	324
-3	1416	1419	185	-6	447	485	351	5	508	481	352
-2	513	523	278	-5	617	672	268		H, 6,-10		
-1	1090	1104	194	-3	1886	1915	165	-4	515	546	406
0	222	212	368	-2	980	967	176	-3	932	915	259
1	938	911	349	-1	412	406	245	-2	438	426	386
2	584	610	267	0	651	685	208	-1	569	534	372
4	368	404	419	1	315	350	335	0	1735	1791	290
	H, 3,-11			4	1140	1153	210	4	638	644	351
-6	649	593	312	6	614	563	331	5	397	355	447
-3	475	465	317		H, 2,-10				H, 7,-10		
-2	1559	1617	195	-6	285	280	428	-2	612	587	333
0	480	511	375	-5	678	651	255	0	884	928	374
2	1208	1184	237	-4	885	881	212	1	1684	1539	475
3	718	701	250	-2	2128	2194	164		H, 8,-10		
5	290	332	415	-1	389	367	295	-3	359	350	560
	H, 4,-11			0	1242	1274	192	0	466	471	584
-5	1230	1235	241	1	1028	934	267	1	1009	883	574
-4	369	411	429	2	565	568	258	2	1044	1024	474
-2	554	529	284	3	499	502	275	4	402	375	570
-1	897	917	248	5	886	874	256		H, 9,-10		
2	651	655	343		H, 3,-10			-2	857	936	398
3	1526	1511	224	-6	924	977	282	2	683	588	634
4	646	620	297	-5	757	750	265	3	961	912	411
	H, 5,-11			-4	450	505	324		H, 1, -9		
-5	536	615	407	-3	537	556	264	-4	2316	2286	162
-4	1409	1406	242	-1	1628	1712	190	-3	460	463	248
-3	665	712	308	0	380	380	424	-2	386	376	252
-1	387	454	479	1	2017	1988	324	0	1217	1250	154
3	828	788	293	2	936	903	232	1	1027	1070	162
4	1587	1604	239	3	498	501	303	3	584	607	232
5	313	315	343	4	294	263	371	5	1124	1148	222
	H, 6,-11			6	300	291	480		H, 2, -9		
-4	626	661	379		H, 4,-10			-7	600	659	344
-3	1205	1209	260	-6	864	877	297	-5	332	317	383
-2	451	491	411	-5	1287	1266	235	-3	1801	1818	165

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 2, -9			1	1062	986	538	1	3360	3323	242
-2	362	356	298	2	672	559	426	2	657	624	231
-1	522	542	232	3	269	217	483	4	388	422	329
0	463	451	266	4	660	664	360	5	837	818	235
1	1969	1922	218		H, 8, -9				H, 4, -8		
2	1006	1063	187	-4	671	638	367	-6	907	903	272
3	200	201	293	1	1297	1257	454	-5	1415	1375	212
4	277	306	288	2	945	821	397	-4	277	241	279
5	305	310	445	4	393	411	467	-3	739	750	234
6	404	406	439	5	912	886	309	-2	1720	1740	180
	H, 3, -9				H, 9, -9			-1	262	292	283
-6	1174	1208	239	-3	831	972	391	1	576	513	482
-5	421	449	384	-1	467	591	621	2	2530	2503	199
-3	566	588	253	2	860	746	515	3	340	373	394
-2	1161	1225	189	3	587	559	544	5	946	996	231
-1	606	634	243		H, 10, -9			6	1237	1299	243
0	866	897	244	0	857	734	490		H, 5, -8		
1	906	865	363	2	615	325	685	-6	441	473	455
2	2569	2543	181		H, 1, -8			-5	612	598	322
3	380	381	357	-7	405	344	351	-4	732	744	272
4	510	506	295	-6	498	495	302	-2	1008	1029	221
	H, 4, -9			-5	1230	1225	182	-1	2189	2309	205
-6	1051	1088	268	-4	501	566	257	0	401	407	499
-5	1541	1514	215	-3	1784	1781	149	3	694	686	269
-4	595	554	265	-2	375	379	234	5	373	344	359
-3	494	520	304	-1	2004	2075	133	6	916	959	271
-2	686	711	247	0	1214	1277	147		H, 6, -8		
0	356	334	503	1	529	537	188	-2	349	374	453
1	850	873	467	2	516	552	203	-1	1467	1515	237
2	912	877	266	3	300	269	210	0	1647	1708	280
3	1679	1698	196	4	730	794	224	2	406	368	505
5	826	801	237	6	373	387	384	6	496	520	383
6	539	547	346		H, 2, -8				H, 7, -8		
	H, 5, -9			-7	906	935	259	-4	421	371	433
-5	1191	1162	248	-6	351	371	403	-1	811	778	307
-4	939	967	248	-5	555	566	275	0	1621	1658	304
-2	989	983	227	-4	863	864	201	1	1369	1259	429
-1	687	720	307	-3	597	579	213	3	890	815	299
0	932	1013	317	-2	860	883	170	5	482	497	416
3	495	469	343	-1	460	450	226		H, 8, -8		
4	1122	1120	226	0	3138	3284	164	-3	1138	1119	275
6	744	796	318	1	943	909	228	0	717	688	446
	H, 6, -9			2	310	260	317	1	1308	1218	415
-4	916	864	257	3	309	258	269	2	685	554	451
-3	374	386	434	7	571	620	359	4	1361	1394	285
-2	391	327	376		H, 3, -8			5	517	544	440
-1	1232	1250	253	-7	921	931	271		H, 9, -8		
0	816	816	357	-6	1193	1245	232	-2	1588	1588	293
1	1452	1270	470	-5	427	378	323	5	1317	1313	296
5	445	385	374	-4	623	645	248		H, 10, -8		
	H, 7, -9			-3	528	471	239	-1	1395	1362	343
-3	520	522	392	-2	251	231	198	1	531	487	685
-1	546	589	415	-1	514	529	243	3	407	224	605
0	1462	1466	311	0	734	743	256		H, 11, -8		

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 11, -8			7	318	306	328	2	754	696	552
0	991	910	490		H, 5, -7				H, 1, -6		
2	714	706	610	-5	975	962	240	-6	579	592	254
3	459	377	631	-4	316	294	342	-5	724	720	215
	H, 1, -7			-3	903	924	224	-4	1102	1125	159
-6	415	416	335	-2	1684	1739	191	-3	184	193	282
-4	2197	2219	153	-1	1098	1191	225	-1	3286	3428	112
-3	495	491	200	0	751	827	318	0	923	951	139
-2	1774	1786	131	2	467	519	414	1	625	622	159
-1	377	412	217	5	361	394	433	6	1015	1040	229
0	2327	2455	127	6	1600	1660	220		H, 2, -6		
1	1129	1152	138	7	610	647	337	-7	1141	1137	241
2	280	265	261		H, 6, -7			-5	1047	1038	193
3	268	273	337	-6	364	378	521	-4	520	488	226
4	552	532	238	-4	440	465	378	-3	636	602	183
7	1423	1458	241	-2	1281	1292	220	-1	488	499	193
	H, 2, -7			-1	1688	1708	232	0	4555	4879	153
-7	896	893	264	0	957	979	304	2	1073	1096	144
-4	375	400	320	2	455	523	517	3	742	789	181
-3	1324	1339	154	3	943	866	243	4	1078	1068	176
-2	972	979	154		H, 7, -7			7	924	963	274
-1	2164	2253	137	-5	1018	1070	291		H, 3, -6		
0	501	561	287	-2	412	351	385	-7	754	748	293
1	2298	2239	175	-1	1052	1075	268	-6	791	845	265
2	238	244	276	0	1174	1227	339	-5	308	298	250
3	1179	1213	160	1	980	869	458	-4	1499	1467	175
4	193	197	361	2	610	650	478	-3	2028	2024	155
6	306	308	417	3	808	870	329	-2	376	391	261
	H, 3, -7			4	997	977	249	0	396	348	353
-7	1290	1344	248		H, 8, -7			1	3056	2983	201
-6	822	816	255	-4	1525	1517	259	3	1297	1336	158
-4	1214	1213	185	-2	304	305	395	4	1239	1249	173
-3	900	921	187	1	990	943	413	5	1352	1349	191
-1	521	502	224	2	650	584	482	7	389	321	303
0	1819	1876	197	3	983	964	313		H, 4, -6		
1	637	589	327	4	965	990	302	-7	608	619	343
2	2159	2163	163	5	1346	1320	259	-6	330	299	437
3	513	547	232		H, 9, -7			-4	471	541	315
4	1505	1529	174	-3	1459	1508	288	-3	1720	1730	173
5	592	596	260	0	483	403	598	-2	1830	1854	164
7	290	187	496	1	629	604	564	-1	408	438	295
	H, 4, -7			2	466	395	630	0	612	665	321
-6	1101	1129	262	4	835	807	359	2	921	922	212
-5	686	683	263	5	715	741	387	3	256	182	375
-4	372	373	351		H, 10, -7			4	1117	1132	185
-3	1708	1752	180	-3	366	345	411	5	1060	1133	210
-2	995	992	192	-2	1201	1263	336	6	1433	1464	208
-1	880	883	205	1	475	389	629		H, 5, -6		
1	524	497	495	2	1018	908	459	-4	296	206	408
2	442	454	338	5	397	413	333	-3	716	733	247
3	1189	1206	188		H, 11, -7			-2	1718	1792	191
4	517	581	278	-1	991	852	393	-1	1569	1656	199
5	1774	1807	193	3	958	914	411	0	232	224	401
6	901	927	251		H, 12, -7			1	1184	1122	338

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 5, -6			-8	431	391	412	0	575	550	348
3	351	273	351	-7	317	309	405	1	847	828	368
5	1042	1081	218	-6	316	256	225	2	1189	1130	209
6	786	827	280	-4	603	625	186	4	468	477	279
7	770	760	290	-3	728	739	161	6	1151	1211	221
	H, 6, -6			-2	4319	4409	111	7	237	275	386
-5	374	388	498	0	1065	1084	135		H, 6, -5		
-2	869	914	242	2	1685	1717	119	-6	1090	1107	277
-1	2117	2202	216	3	465	468	200	-3	371	401	382
0	815	813	327	4	276	245	344	-2	1620	1639	205
1	919	857	444	5	731	710	206	-1	861	892	256
2	1865	1789	239	6	475	409	300	0	255	260	412
3	450	426	372	7	1232	1216	227	1	1668	1567	319
4	497	426	298		H, 2, -5			2	1800	1725	224
6	755	780	275	-7	416	364	381	3	1413	1389	207
7	368	308	464	-6	503	526	282	7	485	485	370
	H, 7, -6			-5	1294	1326	179		H, 7, -5		
-5	394	285	488	-4	923	938	181	-5	1770	1783	246
-4	1172	1198	265	-3	254	216	312	-4	438	274	382
-1	745	835	341	-2	582	589	164	-1	575	553	335
0	1557	1592	291	-1	2556	2598	119	0	702	646	368
2	1070	1055	317	0	341	373	373	2	2024	2000	251
3	1817	1783	229	1	1422	1326	155	3	1883	1860	223
4	623	672	316	2	991	1024	139	4	893	834	238
5	235	246	366	3	3247	3280	132	6	677	710	300
	H, 8, -6			4	723	698	189		H, 8, -5		
-5	863	803	307	6	246	284	319	-5	447	389	438
-4	396	289	490		H, 3, -5			-4	1896	1944	256
-3	1379	1357	254	-7	1057	1093	251	-2	394	324	437
-1	792	764	325	-6	405	355	255	-1	404	377	498
1	729	629	423	-5	912	899	211	0	727	731	451
3	775	737	343	-4	2271	2238	163	1	605	582	466
4	1595	1566	244	-3	1664	1654	155	3	1369	1363	256
5	720	730	309	0	890	901	233	4	1201	1161	252
	H, 9, -6			2	812	765	176	5	759	736	284
-4	745	784	381	3	1181	1182	155	7	952	1024	275
-3	372	332	508	4	3508	3581	151		H, 9, -5		
-2	1222	1263	294	5	502	511	270	-4	605	571	379
0	1232	1186	374	7	332	312	465	-3	1170	1221	299
4	498	524	486		H, 4, -5			-1	662	635	353
5	1399	1403	276	-6	564	574	297	0	436	435	550
	H, 10, -6			-4	1675	1677	181	1	1273	1269	329
-3	611	606	419	-3	2414	2405	163	2	421	310	510
-2	482	386	483	-2	883	883	180	4	642	587	347
-1	713	647	449	1	925	894	282	5	588	543	370
1	1496	1491	353	2	382	385	307	6	388	394	299
2	411	424	476	4	586	610	238		H, 10, -5		
5	264	205	444	5	1929	1930	173	-3	471	502	529
	H, 11, -6			6	508	517	298	-2	653	720	447
-2	425	385	511		H, 5, -5			0	1190	1163	365
2	1413	1398	395	-4	324	268	378	2	1269	1192	412
	H, 12, -6			-3	2175	2225	179		H, 11, -5		
3	1315	1238	417	-2	1131	1168	195	-1	368	404	672
	H, 1, -5			-1	402	414	327	1	1436	1428	334

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 11, -5			-1	677	716	208	4	1390	1390	234
3	681	625	449	0	1863	1944	200	5	347	325	472
5	616	542	415	1	381	288	399	6	1251	1265	237
	H, 12, -5			2	479	462	243	7	500	539	413
2	1094	1041	397	3	481	495	225		H, 9, -4		
	H, 1, -4			4	2157	2176	156	-4	669	727	394
-8	576	593	343	5	700	732	217	-3	384	289	484
-7	314	383	433	6	536	498	257	0	2101	2031	294
-6	1250	1244	190	7	585	644	312	2	375	399	644
-5	318	294	321	8	1022	1018	266	3	342	302	325
-4	681	673	173		H, 5, -4			5	716	763	334
-3	1224	1182	132	-6	326	271	309		H, 10, -4		
-2	574	582	151	-5	341	313	361	-3	443	389	369
-1	4070	4251	94	-4	756	731	231	-1	416	394	384
0	237	200	301	-3	1000	999	199	0	638	600	433
1	2533	2528	98	-2	2040	2101	174	1	1787	1805	304
2	905	891	123	0	1730	1816	231	6	276	221	538
3	830	859	150	1	2114	2010	248		H, 11, -4		
4	422	371	228	2	418	367	318	-3	609	557	489
6	1028	1035	197	3	606	591	224	0	446	486	638
7	457	458	345	5	817	800	211	1	528	580	541
	H, 2, -4				H, 6, -4			2	1545	1555	351
-7	366	325	378	-6	587	545	368	4	649	757	486
-6	736	783	250	-5	671	645	291		H, 12, -4		
-5	1684	1730	177	-2	653	726	276	3	953	951	457
-4	942	959	171	-1	1454	1526	214		H, 1, -3		
-3	223	221	227	0	608	556	373	-7	1051	1086	229
0	2401	2487	147	1	2364	2260	288	-6	1261	1219	191
1	691	678	182	2	2025	1991	213	-5	738	753	201
2	3253	3291	114	3	710	743	243	-4	209	179	250
3	1304	1340	136	4	211	231	364	-3	464	442	173
4	1565	1584	147	5	242	224	298	-2	4167	4234	99
6	416	418	313	6	244	236	450	-1	1148	1145	100
7	458	494	346		H, 7, -4			0	413	395	259
8	282	226	399	-6	1146	1085	278	1	1152	1142	101
	H, 3, -4			-5	696	588	310	2	5006	5161	98
-6	270	256	319	-4	1107	1099	253	3	228	273	323
-5	838	840	221	-3	285	280	436	4	607	613	183
-4	1883	1846	160	-2	1032	1081	243	5	563	561	215
-3	1874	1838	144	-1	389	417	448	6	504	514	291
-2	256	230	324	0	686	730	408	7	446	493	340
-1	1356	1380	142	1	587	527	444		H, 2, -3		
0	380	328	374	2	1959	1929	243	-6	1494	1498	193
1	1147	1133	203	3	2391	2342	209	-5	1956	1973	167
3	2987	3075	133	4	692	684	263	-4	1169	1134	155
4	1373	1387	160	5	950	965	233	-3	469	458	200
5	1159	1164	178	6	512	512	335	-2	400	386	200
6	512	561	280	7	363	419	513	-1	1184	1181	119
7	887	835	238		H, 8, -4			0	363	334	398
	H, 4, -4			-5	1043	986	290	1	449	391	233
-5	699	703	247	-4	489	415	419	2	1280	1233	116
-4	869	851	209	-3	845	827	292	3	4172	4193	118
-3	2487	2501	158	-1	1754	1833	257	5	799	822	188
-2	1225	1242	162	3	1050	1028	271	6	431	362	282

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 3, -3			1	682	598	400	0	802	810	167
-5	1915	1928	177	2	1827	1800	237	1	5116	5250	76
-4	1482	1487	162	3	1799	1781	210	2	1235	1171	103
-3	693	675	176	5	556	561	306	3	592	603	156
-2	1007	961	144	6	1267	1336	228	4	476	482	204
-1	513	510	210	7	452	496	393	5	598	610	204
0	800	827	240		H, 8, -3			6	570	651	263
1	766	729	223	-5	659	640	372	8	636	731	352
2	830	803	153	-4	1224	1226	264		H, 2, -2		
3	506	497	194	-2	1455	1471	246	-7	761	742	265
4	2958	3006	139	0	1274	1329	312	-6	1043	1015	209
6	754	758	233	1	810	733	309	-5	2120	2080	163
7	807	846	260	2	374	270	546	-4	683	673	183
8	703	642	295	3	951	959	277	-3	379	385	229
	H, 4, -3			4	597	594	321	-2	1712	1701	113
-4	2187	2219	169	6	638	646	323	-1	506	522	181
-3	1154	1142	167	7	1548	1584	239	0	963	1031	202
-2	437	441	243		H, 9, -3			2	5436	5543	98
-1	1163	1176	169	-4	499	575	464	3	884	894	135
0	783	787	276	-3	564	556	397	4	507	540	203
1	1770	1692	200	-2	483	484	441	5	1281	1278	165
2	292	303	346	-1	1711	1696	287	6	1397	1371	179
3	726	718	180	1	950	996	355		H, 3, -2		
5	1054	1116	185	2	430	369	586	-6	1169	1148	215
7	1157	1176	226	3	426	342	471	-5	771	775	230
8	899	929	277	7	569	618	415	-4	1388	1387	162
	H, 5, -3				H, 10, -3			-3	526	535	195
-7	1030	1046	285	-2	271	204	473	-2	1553	1531	130
-5	344	274	349	-1	532	492	496	-1	2495	2515	122
-4	398	427	339	0	1850	1815	287	0	894	882	236
-3	2299	2293	171	1	426	411	543	1	220	206	380
-2	527	491	233	2	311	323	542	2	691	665	153
-1	891	940	214	4	748	756	377	3	2437	2434	121
0	1894	1963	228		H, 11, -3			4	713	734	172
1	2343	2311	228	-3	477	358	524	6	971	1000	202
2	1044	995	195	1	1513	1515	293	7	1313	1325	209
3	385	360	289	2	447	349	476		H, 4, -2		
8	1194	1284	262	5	1040	1167	348	-6	321	274	424
	H, 6, -3				H, 12, -3			-5	1092	1102	206
-6	1548	1531	241	-2	685	558	475	-4	276	261	404
-4	699	679	254	-1	641	578	456	-3	1239	1235	166
-2	759	755	245	0	435	481	612	-2	455	483	250
0	873	871	310	2	756	791	423	-1	2588	2695	147
1	2690	2540	261		H, 13, -3			0	979	1034	267
2	2550	2481	196	0	513	509	563	2	329	266	298
3	556	549	277	1	559	505	452	3	503	500	193
4	663	634	227	4	378	304	483	4	976	964	162
5	696	713	246		H, 1, -2			5	375	388	283
	H, 7, -3			-7	931	929	235	6	699	712	229
-6	603	585	392	-6	1763	1725	178	7	543	563	332
-5	1464	1440	240	-5	499	446	235	8	1322	1345	243
-3	1193	1205	230	-4	288	312	287		H, 5, -2		
-1	488	494	376	-2	607	567	141	-7	652	662	370
0	664	646	377	-1	2618	2626	79	-5	255	176	400

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 5, -2			-4	575	601	477	1	577	524	230
-4	188	187	366	-1	947	855	358	2	951	892	132
-3	445	429	295	0	1010	986	315	4	695	716	169
-2	1172	1171	179	1	1146	1198	297	6	2289	2274	171
-1	1359	1389	183	3	1644	1634	292	7	742	779	257
0	3297	3451	214	5	354	357	377	8	520	545	361
1	2093	2001	217	7	430	430	476		H, 4, -1		
3	945	946	173		H, 11, -2			-5	337	268	384
4	322	270	275	-3	967	1036	398	-4	1729	1726	168
7	907	965	245	2	791	852	413	-3	215	229	364
8	500	432	382	4	1732	1703	287	-2	2400	2450	143
	H, 6, -2			6	523	428	412	-1	2506	2579	146
-6	577	618	379		H, 12, -2			0	1527	1565	216
-3	1384	1363	198	-2	1078	999	384	1	1017	952	203
-2	542	508	272	3	476	462	438	2	474	458	225
-1	583	598	289	5	1352	1404	326	7	1555	1593	200
0	1107	1078	293		H, 13, -2			8	570	575	338
1	3261	3220	243	-1	792	744	404		H, 5, -1		
2	1977	1926	199	2	615	651	468	-7	1057	1005	283
4	2302	2265	171	3	484	489	445	-5	931	971	247
5	1011	998	199		H, 1, -1			-4	504	459	275
8	595	609	348	-7	1822	1810	206	-3	912	906	197
	H, 7, -2			-6	1113	1104	200	-1	2300	2420	172
-6	1002	983	288	-5	649	657	201	0	1654	1659	236
-5	326	337	300	-4	1044	1005	146	1	2345	2289	202
-2	2221	2287	208	-3	545	547	165	2	569	520	237
-1	479	486	369	-2	678	655	132	3	1320	1295	159
1	362	194	535	0	2557	2847	98	4	786	791	186
2	1513	1467	233	1	728	692	101	8	1287	1344	244
3	1594	1570	202	2	3934	3900	85		H, 6, -1		
5	2006	2023	191	3	740	724	136	-6	1119	1070	257
6	971	1008	227	4	1774	1752	131	-4	1636	1645	207
7	458	479	383	5	357	319	270	-3	302	315	353
	H, 8, -2				H, 2, -1			-2	435	420	234
-5	557	545	394	-6	2020	1967	183	-1	622	653	281
-3	428	419	415	-5	1205	1175	175	0	1027	1060	268
-2	475	419	408	-3	1360	1319	129	1	2203	2139	239
-1	1870	1897	252	-2	1042	1006	119	2	1873	1785	188
3	298	292	426	-1	690	704	145	3	1483	1472	176
4	880	856	248	0	990	1042	206	4	1774	1750	171
5	438	479	394	1	1301	1196	108	5	1601	1647	180
6	1333	1423	237	3	2519	2509	110	6	445	516	310
7	710	737	311	4	501	465	177		H, 7, -1		
	H, 9, -2			5	2366	2343	149	-6	521	591	456
-2	881	892	303	6	1030	992	195	-5	769	695	282
-1	986	980	337	7	606	583	261	-4	366	351	457
0	1761	1741	261		H, 3, -1			-3	2220	2205	208
2	777	774	406	-7	273	206	413	-1	835	770	260
3	548	472	364	-5	2227	2184	173	0	932	891	269
4	415	356	410	-4	555	597	226	2	1208	1192	242
5	291	291	300	-3	1292	1275	144	3	486	466	314
6	729	748	316	-2	2183	2155	124	4	1174	1143	211
7	994	1024	271	-1	1968	1976	125	5	1381	1381	201
	H, 10, -2			0	1382	1475	204	6	1265	1345	214

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 7, -1			1	253	258	204	1	1075	997	194
7	469	502	339	2	846	790	111	2	1901	1776	131
8	565	563	342	3	659	627	142	3	384	378	241
	H, 8, -1			4	509	479	189	5	260	275	357
-5	461	416	438	5	582	570	199	6	1808	1825	175
-4	245	192	442	7	1430	1439	209	7	247	214	331
-3	932	915	266	8	627	608	329	8	699	686	270
-2	2296	2319	230		H, 1, 0				H, 5, 0		
-1	293	197	404	-8	783	896	327	-7	519	508	403
0	446	465	408	-7	841	803	246	-6	345	403	505
1	500	441	397	-6	1452	1390	187	-5	264	234	339
2	680	625	331	-4	925	923	153	-4	964	962	205
3	282	342	453	-3	1224	1164	124	-3	566	558	251
4	406	487	418	-2	671	650	128	-1	1410	1453	186
5	523	495	324	-1	733	672	107	0	3597	3738	136
6	973	952	240	0	643	417	126	1	874	819	255
7	1130	1192	250	1	5292	5475	61	2	1174	1112	171
	H, 9, -1			2	203	123	301	3	2770	2687	143
-2	875	970	337	3	972	896	123	4	1328	1313	159
-1	1803	1755	281	4	1682	1645	130	7	856	907	242
0	355	355	403	5	1846	1786	149		H, 6, 0		
1	485	463	420	6	305	297	302	-5	579	623	332
3	1173	1138	264	8	723	743	288	-4	322	274	245
5	518	512	353		H, 2, 0			-3	1679	1705	194
7	623	656	363	-7	1302	1298	226	-2	836	850	227
	H, 10, -1			-6	455	427	331	-1	287	258	458
-1	482	491	491	-5	823	781	198	0	579	574	225
0	1465	1513	282	-4	274	194	310	1	1806	1686	230
1	389	351	508	-3	1481	1474	130	2	990	976	208
2	1339	1229	298	-2	1115	1119	122	3	1201	1146	179
4	1467	1424	259	-1	1023	993	119	4	2765	2744	163
5	370	267	436	1	972	910	114	5	1529	1577	180
	H, 11, -1			2	2046	1967	94		H, 7, 0		
-3	463	468	578	3	693	666	140	-6	425	477	500
-2	446	363	496	4	1170	1167	135	-4	874	865	253
-1	636	603	390	5	1395	1373	152	-3	748	697	282
1	909	971	328	6	2148	2102	168	-2	2169	2219	210
3	1184	1178	354		H, 3, 0			-1	452	491	386
5	1189	1188	295	-6	1194	1156	213	0	554	540	227
	H, 12, -1			-3	735	698	166	1	348	357	476
-2	532	467	509	-2	2902	2904	123	3	716	710	243
0	744	749	377	-1	2537	2540	122	4	1333	1345	197
1	523	569	450	2	499	479	189	5	2274	2265	185
2	340	325	489	3	702	701	152	6	936	930	228
3	490	388	538	4	650	646	182		H, 8, 0		
4	627	603	453	5	1949	1929	151	-4	389	282	434
	H, 13, -1			6	816	800	213	-3	1249	1201	247
-1	433	381	583	7	1236	1250	213	-2	1105	1141	274
1	602	656	414		H, 4, 0			-1	1260	1243	274
2	636	689	435	-6	275	275	481	0	264	261	397
4	498	410	573	-5	354	330	387	1	1289	1354	255
	H, 14, -1			-2	1349	1330	153	2	623	606	347
2	442	516	622	-1	4691	5089	144	3	516	468	305
	H, 0, 0			0	1424	1406	157	4	459	449	341

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ
	H, 8, 0		
5	1153	1157	229
6	1002	1011	233
7	357	288	425
8	373	377	482
	H, 9, 0		
-5	449	440	498
-4	397	276	423
-2	1185	1164	288
-1	1140	1144	316
0	441	438	301
1	608	634	360
2	2351	2216	261
4	393	424	418
6	664	640	320
7	591	561	332
	H, 10, 0		
-4	864	859	378
-3	424	373	511
-1	670	629	381
0	437	488	342
1	255	235	344
2	539	513	458
3	2577	2483	263
4	297	238	333
	H, 11, 0		
-3	994	1001	382
-2	478	420	563
-1	520	465	441
1	292	327	426
2	308	281	531
3	395	372	438
4	2053	1992	271
5	306	325	393
6	410	351	384
	H, 12, 0		
-2	621	620	470
0	686	677	256
1	561	602	418
2	663	661	372
5	1065	1048	336
	H, 13, 0		
1	813	949	376
2	506	605	462
3	435	425	304
4	399	275	476
	H, 14, 0		
2	1125	1142	332
3	378	367	370
	H, 0, 1		
-8	1319	1332	242
-7	456	466	347
-5	966	959	173
-4	744	710	153
-3	1092	1037	123
-1	3765	3845	63
0	364	397	77
1	3050	2968	60
2	1105	1067	100
3	2733	2687	107
6	404	397	313
7	849	825	231
8	1152	1223	247
	H, 1, 1		
-7	1599	1614	211
-6	570	574	256
-5	748	720	197
-4	1703	1678	140
-3	1389	1332	120
-1	929	928	102
0	2922	3187	115
1	220	232	254
2	1339	1318	94
3	946	907	120
4	4090	4032	122
5	586	557	205
6	431	400	277
	H, 2, 1		
-6	1519	1473	198
-4	1984	1992	147
-3	1641	1648	131
-2	1052	1005	123
-1	1096	1084	122
0	826	851	275
1	647	565	141
2	246	216	235
3	450	344	170
5	3001	2919	143
6	769	805	222
7	320	238	395
8	470	510	351
	H, 3, 1		
-5	1148	1102	186
-4	287	305	353
-3	2958	2997	138
-2	842	850	157
-1	1587	1597	134
1	633	625	211
4	905	894	150
5	505	521	223
6	2454	2389	166
7	446	436	321
8	275	281	511
	H, 4, 1		
-6	972	926	242
-5	444	448	325
-4	687	715	219
-3	387	382	278
-2	3051	3113	144
-1	2002	2079	154
0	1637	1646	194
1	2184	2111	152
2	1957	1890	129
3	800	811	159
5	732	709	192
6	186	179	361
7	1350	1336	205
	H, 5, 1		
-7	444	491	470
-5	1894	1852	204
-4	563	567	274
-3	303	270	329
-1	2094	2173	178
0	1890	2002	194
1	933	894	239
2	2854	2735	149
3	3002	2941	140
4	1008	997	165
5	386	374	289
6	270	272	305
8	693	700	298
	H, 6, 1		
-5	604	580	332
-4	2152	2101	200
-3	377	430	380
-1	748	802	260
1	1434	1405	239
3	2101	2013	164
4	2385	2351	164
5	1512	1510	181
6	333	375	406
7	830	849	245
8	394	411	446
	H, 7, 1		
-4	1158	1106	249
-3	1662	1681	227
-1	223	228	442
0	402	417	388
1	749	765	302
2	708	683	282
3	838	842	225
4	791	786	234
5	1436	1415	197
6	914	926	227
8	1156	1141	245
	H, 8, 1		
-3	1290	1301	252
-2	1766	1767	242
0	1379	1406	226
2	1243	1197	248
3	377	373	429
4	845	829	251

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
H, 8, 1				3	1017	989	121	-6	491	539	375
5	339	293	412	4	1830	1769	131	-5	449	425	320
6	610	571	293	5	533	523	194	-4	453	447	264
7	353	316	427	7	1136	1149	210	-3	503	488	240
8	457	515	423	8	514	422	303	-2	714	768	206
H, 9, 1				H, 1, 2				-1	3209	3368	155
-2	796	752	321	-8	952	980	269	1	2609	2500	149
-1	1271	1310	286	-5	605	583	224	2	1712	1620	130
1	2129	2149	230	-4	1772	1712	142	3	1234	1223	139
2	667	587	373	-3	1500	1430	126	4	311	319	287
3	1124	1116	270	-2	821	795	126	6	1422	1401	178
4	355	352	382	-1	318	321	237	8	281	325	519
H, 10, 1				0	831	827	270	H, 5, 2			
-2	616	553	397	1	2474	2388	72	-6	1125	1107	260
-1	231	224	441	2	587	558	133	-5	767	676	264
0	893	960	291	3	2646	2612	105	-4	1319	1254	197
2	1860	1907	267	4	1399	1362	135	-3	827	847	219
3	607	604	432	5	1960	1932	148	-2	351	378	353
4	1103	1095	286	6	607	615	240	-1	796	862	231
5	520	418	371	7	380	371	364	0	1773	1856	189
6	905	861	284	8	369	355	442	2	2700	2578	148
H, 11, 1				H, 2, 2				3	3032	2935	141
-1	778	794	343	-7	859	872	256	4	1619	1585	154
0	751	804	336	-5	787	758	208	5	926	952	176
1	509	519	367	-4	714	689	193	6	331	348	351
2	409	435	559	-3	3049	3078	129	8	269	300	452
3	1223	1155	325	-2	1255	1244	125	H, 6, 2			
5	654	623	380	-1	429	442	234	-6	410	408	509
H, 12, 1				0	786	787	259	-5	1205	1162	242
-2	347	339	561	1	695	653	138	-4	746	713	275
-1	593	588	432	2	246	97	291	-3	1089	1072	220
0	724	694	338	3	1316	1260	115	-2	458	458	302
1	785	802	311	4	3168	3113	125	-1	1437	1485	219
3	512	465	450	5	620	610	195	2	475	419	278
4	338	375	528	6	1216	1193	178	3	2245	2181	162
H, 13, 1				7	532	506	287	4	2291	2247	162
0	975	1026	322	8	717	699	271	5	899	912	203
1	501	491	441	H, 3, 2				6	1173	1167	202
2	528	592	464	-7	397	332	414	7	507	515	333
H, 14, 1				-6	314	324	437	8	431	397	376
1	1102	1080	297	-4	1001	973	184	H, 7, 2			
3	289	337	499	-3	985	955	163	-5	532	598	434
H, 0, 2				-2	3087	3159	130	-4	1135	1106	255
-8	361	327	504	-1	761	791	191	-3	984	984	256
-7	597	584	291	0	2100	2170	166	-2	512	521	350
-6	447	498	315	1	1796	1710	126	-1	506	469	363
-5	1180	1208	173	2	218	126	219	0	1790	1890	204
-4	474	510	202	3	336	296	238	1	248	265	495
-3	660	651	150	4	662	632	168	2	776	735	249
-2	261	201	244	5	2279	2251	146	3	609	636	267
-1	425	415	152	7	272	240	279	4	1423	1447	198
0	4773	4917	61	8	325	293	462	5	1422	1406	194
1	573	509	111	H, 4, 2				7	911	907	241
2	2977	2912	87	-7	707	735	339	8	568	590	372

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 8, 2			-4	1094	1091	149	-7	447	456	425
-3	1016	1010	292	-3	235	219	323	-6	1543	1483	228
-2	1007	988	280	-2	459	435	159	-5	718	699	253
0	1159	1191	239	-1	2016	2008	88	-4	330	342	298
1	1927	2001	216	1	165	104	211	-2	1750	1775	162
4	354	300	364	2	862	846	113	-1	992	1032	200
5	807	771	244	3	3896	3888	109	0	527	555	314
6	564	545	299	7	324	279	431	1	3744	3688	148
7	435	532	436	8	556	643	339	2	3380	3297	127
8	776	767	309		H, 1, 3			3	346	348	244
	H, 9, 2			-7	567	558	322	7	486	553	329
-5	447	545	563	-5	2256	2218	164		H, 5, 3		
-4	505	487	442	-4	909	872	161	-6	761	771	321
-3	253	286	402	-3	934	931	144	-5	1686	1638	220
-2	828	794	336	-2	167	81	253	-4	615	582	261
-1	377	380	521	-1	524	564	178	-3	467	483	279
0	494	539	385	1	696	684	117	-1	196	165	372
1	1059	1126	260	2	508	482	150	0	1432	1525	192
2	2616	2560	256	3	190	137	204	1	1039	989	217
3	596	617	369	4	3399	3400	125	2	1719	1683	155
4	860	805	275	5	316	276	286	3	2859	2814	142
	H, 10, 2			6	422	409	307	4	546	522	230
-4	641	536	427	7	634	658	274	6	1291	1283	190
-3	610	581	420	8	366	387	438	7	673	665	277
-2	824	803	345		H, 2, 3				H, 6, 3		
-1	285	214	551	-6	474	423	301	-5	1216	1174	253
2	569	594	421	-5	279	269	412	-4	1459	1463	230
3	2192	2127	253	-4	3121	3148	151	-2	943	961	238
4	458	469	449	-3	903	937	163	-1	313	327	347
5	1244	1255	269	-1	566	571	211	0	1080	1157	214
7	451	555	486	0	793	771	265	1	1200	1160	243
	H, 11, 2			1	268	270	317	2	1210	1115	188
-2	457	445	510	2	570	529	149	3	1141	1099	179
-1	1023	1067	311	3	1292	1295	119	4	1400	1387	172
1	555	549	353	4	766	731	153	5	298	272	382
4	1397	1317	288	5	2160	2095	148	6	670	682	256
6	1094	1073	299	6	287	330	395	7	1593	1619	203
	H, 12, 2			7	1192	1163	207	8	667	691	320
0	1017	1109	307	8	611	664	317		H, 7, 3		
1	447	395	405		H, 3, 3			-5	397	338	465
3	422	428	540	-7	651	649	332	-4	1285	1264	251
5	436	435	532	-6	438	360	345	-3	839	852	286
6	401	427	572	-5	386	357	334	-2	553	525	321
	H, 13, 2			-4	291	305	359	-1	2137	2129	231
-1	464	468	437	-3	3323	3392	147	1	671	758	291
1	1068	1104	297	-2	688	719	184	2	643	643	281
4	507	454	524	-1	433	439	281	3	910	877	214
5	261	238	492	0	3134	3408	158	4	373	328	331
	H, 14, 2			1	3351	3245	121	5	419	368	317
2	888	942	357	4	1281	1242	141	7	962	964	247
	H, 0, 3			5	489	487	231	8	1122	1132	263
-8	918	940	271	6	1193	1174	184		H, 8, 3		
-6	1077	1072	202	8	1466	1476	217	-3	709	693	324
-5	1265	1280	173		H, 4, 3			-2	606	677	385

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 8, 3			1	998	1006	104	-1	1011	1045	214
-1	799	777	301	2	5564	5761	98	0	868	917	223
0	2591	2805	206	3	569	568	164	1	4520	4488	153
1	1014	1058	260	4	1195	1182	144	2	1923	1883	137
2	492	468	385	5	1061	1053	160	3	955	956	154
3	313	267	455	6	672	638	227	4	1871	1836	143
8	968	1005	288	7	467	520	325	5	741	755	196
	H, 9, 3				H, 1, 4			6	517	498	258
-4	389	393	550	-6	1114	1120	213	7	345	316	397
-1	763	769	294	-5	405	436	337	8	938	961	260
0	428	512	408	-4	2034	2028	151		H, 5, 4		
1	2133	2213	223	-3	767	772	161	-6	1133	1094	268
2	1066	964	289	-2	492	483	174	-5	819	746	260
5	1315	1298	242	-1	789	780	151	-4	374	380	381
6	399	350	398	2	1566	1543	102	-2	1300	1285	196
	H, 10, 3			3	2714	2705	112	-1	590	624	313
-3	677	636	426	4	508	506	189	0	286	261	260
-2	613	618	452	5	373	442	285	2	2494	2409	154
-1	854	896	282	6	780	794	213	3	1980	1963	148
0	749	820	291	7	1101	1095	214	4	460	432	234
1	411	399	340		H, 2, 4			5	1943	1934	164
2	1434	1441	261	-7	313	294	428	6	1062	1086	204
6	1612	1627	250	-5	1560	1536	188	7	354	358	399
7	468	492	431	-4	296	285	204	8	485	514	371
	H, 11, 3			-3	2270	2314	143		H, 6, 4		
-2	1121	1051	312	-1	2048	2105	139	-6	728	716	329
-1	832	803	303	0	529	495	380	-5	993	904	263
0	758	771	297	1	378	352	237	-4	661	639	301
1	489	556	405	2	688	694	140	-2	733	724	274
2	349	351	503	3	1173	1177	126	-1	1665	1673	230
3	604	559	416	4	2140	2129	133	1	726	698	310
4	477	392	445	6	664	648	226	2	559	557	269
7	1189	1267	303	7	334	354	436	3	1340	1305	178
	H, 12, 3			8	1256	1256	228	4	1329	1314	178
-1	1296	1322	283		H, 3, 4			5	631	620	228
0	389	344	482	-7	614	618	359	6	1580	1572	196
2	410	399	453	-5	225	258	404	7	971	969	231
5	646	575	378	-4	893	898	202	8	343	377	462
6	466	278	463	-3	704	709	210		H, 7, 4		
	H, 13, 3			-2	2030	2059	149	-5	385	440	545
0	1202	1226	289	-1	1390	1459	173	-4	835	810	295
3	441	442	418	0	3700	4067	162	-3	392	440	471
4	583	582	521	1	2587	2475	132	-2	945	995	280
	H, 14, 3			2	817	799	146	-1	1700	1652	229
1	806	815	325	3	360	392	232	0	2280	2450	195
4	476	510	610	5	1466	1418	160	1	425	475	373
	H, 0, 4			7	1289	1256	205	3	416	409	340
-7	486	497	356		H, 4, 4			4	530	561	294
-6	441	424	321	-7	929	939	303	5	563	556	286
-5	1874	1828	166	-6	809	764	281	6	1243	1253	211
-4	706	663	176	-5	424	415	369	7	1204	1201	231
-3	472	429	188	-4	458	478	319	8	655	695	335
-2	241	265	294	-3	400	383	321		H, 8, 4		
0	919	881	114	-2	909	911	195	-5	384	377	508

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 8, 4			-4	439	421	237	-5	484	437	333
-3	696	749	369	-3	483	493	203	-4	899	885	237
-1	1159	1198	257	-1	355	353	204	-1	939	995	225
0	1538	1600	217	0	848	823	125	0	1612	1728	186
1	1771	1886	217	1	1541	1553	101	1	2486	2423	164
2	562	539	365	2	373	371	204	2	2835	2770	139
3	1530	1471	216	3	3155	3144	119	3	994	968	155
6	297	331	333	4	215	233	345	4	1139	1139	161
7	964	1024	265	5	1321	1317	166	5	1254	1269	172
8	651	696	344	6	559	538	251	6	834	839	216
	H, 9, 4			7	454	475	337		H, 5, 5		
-4	447	519	561	8	256	242	323	-6	1021	942	291
-3	874	875	346		H, 1, 5			-5	883	860	267
-2	652	594	372	-5	2519	2531	175	-4	329	205	430
-1	343	245	496	-4	526	500	229	-3	1520	1533	206
0	536	582	336	-3	674	664	173	-1	555	536	332
1	773	836	281	-2	1252	1247	135	0	1198	1275	201
2	1635	1592	261	-1	654	685	189	1	1711	1701	198
4	2239	2147	219	1	889	864	122	2	1340	1306	173
5	381	367	460	2	2408	2420	106	3	927	904	172
6	527	547	375	3	771	749	147	4	479	476	243
7	389	422	448	4	1362	1365	139	5	1440	1438	176
	H, 10, 4			5	213	220	332	6	1598	1588	187
-3	490	366	443	6	1939	1938	174	7	921	906	235
-2	1154	1191	314	7	511	544	312		H, 6, 5		
0	444	460	400	8	556	589	338	-6	330	308	531
3	1164	1103	292		H, 2, 5			-5	985	942	277
5	1991	1933	237	-4	2514	2581	165	-4	455	379	355
7	519	488	397	-2	1716	1731	146	-3	990	971	259
	H, 11, 4			-1	1706	1770	159	-2	1833	1900	224
-3	559	535	508	0	1840	1814	203	-1	348	373	469
-1	1080	1182	300	1	352	331	260	0	684	728	251
2	385	405	450	2	315	299	221	1	918	910	264
4	441	464	488	3	1492	1463	126	2	845	828	225
5	706	670	353	4	659	654	167	3	364	372	320
6	1294	1287	289	5	534	507	204	4	296	286	356
	H, 12, 4			6	337	285	337	5	401	353	317
-2	785	782	365	7	1950	1951	192	6	1547	1567	197
0	852	866	298	8	404	387	422	7	1397	1416	216
2	583	528	343		H, 3, 5			8	526	504	358
3	507	452	458	-7	654	662	354		H, 7, 5		
4	470	459	345	-6	453	507	405	-5	438	316	401
6	631	607	408	-5	424	382	317	-4	599	542	346
	H, 13, 4			-3	1567	1584	170	-2	1230	1219	263
-1	494	531	450	-2	371	402	301	-1	2062	2119	225
1	588	605	390	-1	1813	1846	179	0	873	945	237
3	1048	1085	330	0	1531	1602	190	3	211	124	304
5	480	443	543	1	3609	3570	138	5	621	657	262
	H, 14, 4			2	583	575	178	6	386	322	387
2	301	366	425	3	686	660	161	7	1314	1362	233
4	1169	1188	351	4	250	207	306	8	539	609	373
	H, 0, 5			8	1751	1795	219		H, 8, 5		
-6	1570	1551	196		H, 4, 5			-5	708	628	373
-5	631	639	237	-6	980	995	269	-2	450	405	412

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 8, 5				H, 14, 5			7	1400	1406	210
-1	930	912	268	1	326	365	545		H, 4, 6		
0	2328	2517	206	2	377	394	532	-6	540	473	373
1	965	1034	253	3	617	665	428	-3	1007	1012	213
2	1106	1097	260		H, 0, 6			-2	842	864	223
3	505	482	346	-7	1268	1243	244	-1	267	314	520
4	1258	1266	227	-5	950	982	210	1	2892	2785	170
5	673	661	270	-3	831	829	169	2	1588	1530	155
6	454	354	331	-2	450	443	193	3	428	430	241
7	356	289	492	-1	387	360	206	4	2031	2048	151
8	736	785	320	0	303	343	252	5	1471	1511	172
	H, 9, 5			1	1347	1384	113	7	464	431	336
-4	998	946	356	2	3175	3151	113	8	576	613	342
-2	641	556	368	3	450	453	196		H, 5, 6		
-1	716	769	321	4	639	628	181	-6	545	536	408
0	362	426	418	5	694	680	193	-4	709	750	300
1	1555	1687	227	6	1287	1304	189	-3	935	885	243
2	380	338	483	8	335	327	258	-2	1457	1506	224
3	1121	1056	267		H, 1, 6			-1	590	652	360
4	342	367	426	-6	1274	1291	221	0	436	490	322
5	1693	1707	231	-4	949	929	189	1	418	399	375
6	627	665	325	-3	710	689	187	2	1151	1143	186
	H, 10, 5			-2	2397	2455	137	3	998	973	175
-3	1227	1106	338	-1	706	680	193	4	1349	1353	171
-2	322	330	581	0	232	270	334	5	2032	2041	172
-1	525	563	392	1	528	530	169	6	1268	1238	200
0	777	810	283	2	1387	1390	119	8	266	271	424
2	725	742	332	3	2115	2087	126		H, 6, 6		
3	567	566	411	4	313	343	291	-6	416	374	467
4	758	721	313	5	1652	1627	162	-5	568	548	369
5	452	321	410	6	240	293	443	-3	1272	1203	246
6	1477	1501	255	7	1198	1200	212	-2	1491	1438	241
7	448	451	452		H, 2, 6			-1	893	925	263
	H, 11, 5			-5	880	901	237	0	300	323	434
-3	404	317	362	-4	428	403	289	1	688	640	301
-2	1329	1264	287	-3	483	518	248	2	343	307	386
-1	331	324	304	-2	1373	1403	163	3	272	279	330
1	518	558	362	-1	4035	4203	164	4	476	505	286
2	279	254	350	2	715	737	157	5	1881	1883	186
4	927	873	311	4	1142	1133	152	6	1259	1246	212
5	590	533	393	5	344	353	314	7	626	706	303
7	678	722	376	6	2081	2073	174	8	241	225	445
	H, 12, 5			8	1061	1036	236		H, 7, 6		
-1	1203	1238	281		H, 3, 6			-4	606	613	365
1	629	618	343	-7	588	630	379	-2	1300	1251	273
2	424	383	464	-3	762	756	222	-1	1642	1674	231
3	616	681	423	-2	330	243	317	0	964	1013	233
5	606	557	418	-1	1143	1156	209	2	1875	1868	213
6	389	328	568	0	3331	3575	180	3	473	479	326
	H, 13, 5			1	1148	1104	169	5	325	356	430
0	857	831	294	3	1966	1983	136	6	1491	1468	211
2	784	824	330	4	978	999	163	7	817	814	263
3	428	426	532	5	514	498	229	8	242	241	476
4	998	998	394	6	378	407	324		H, 8, 6		

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ
	H, 8, 6		
-5	282	284	437
-4	821	815	354
-3	612	599	398
-1	848	848	270
0	973	992	238
1	823	883	265
2	284	293	537
3	2468	2356	213
4	735	696	272
6	379	405	423
7	461	550	409
8	471	527	429
	H, 9, 6		
-3	1061	1009	343
-2	571	532	389
0	302	211	442
2	630	631	333
3	607	580	350
4	2315	2301	228
5	580	585	316
7	422	466	458
	H, 10, 6		
-2	781	723	317
2	559	592	369
3	370	353	491
4	764	770	341
5	1627	1581	253
	H, 11, 6		
-3	847	791	359
-1	361	401	534
1	761	745	267
2	363	225	461
3	476	548	533
5	723	708	361
6	810	860	361
	H, 12, 6		
-2	636	574	375
2	1323	1339	267
	H, 13, 6		
0	412	343	303
3	1359	1405	299
	H, 0, 7		
-6	1466	1467	215
-4	1043	1059	188
-3	901	921	181
-2	656	663	175
-1	251	271	350
0	1165	1147	136
1	1448	1487	123
2	554	558	171
3	664	674	173
4	272	234	329
5	1897	1923	167
6	376	373	353
7	519	495	308
8	366	341	424
	H, 1, 7		
-5	1326	1324	202
-3	2197	2258	159
-2	630	635	193
-1	1505	1603	176
1	288	323	276
2	1211	1239	134
3	509	503	197
4	539	550	207
5	594	608	225
6	1990	2041	180
	H, 2, 7		
-5	317	315	337
-4	1057	1035	203
-2	2614	2710	163
-1	1218	1263	217
0	1831	1797	216
1	1135	1114	155
2	1222	1188	140
4	248	250	361
5	671	677	217
6	450	419	300
7	1751	1750	198
	H, 3, 7		
-5	835	880	274
-4	387	435	380
-3	343	325	362
-2	586	602	259
-1	2126	2201	212
0	1207	1239	212
1	1417	1348	172
2	1923	1883	142
3	2157	2169	142
4	496	477	219
5	757	772	211
8	1227	1258	242
	H, 4, 7		
-5	505	500	373
-4	1485	1448	219
-3	395	423	386
-2	457	415	330
-1	1112	1131	258
0	1014	1122	224
1	1311	1276	197
2	419	400	261
3	1254	1263	162
4	1970	1973	157
5	1175	1190	181
6	874	871	216
7	485	461	317
	H, 5, 7		
-6	511	508	461
-4	1150	1172	262
-3	1552	1526	226
-2	484	473	361
0	539	572	282
1	502	505	345
2	780	756	215
3	915	944	200
4	432	348	282
5	1762	1725	177
6	904	892	221
7	342	309	426
8	742	694	274
	H, 6, 7		
-5	292	254	389
-4	263	171	485
-3	1475	1455	250
-2	1697	1711	247
-1	828	805	254
0	963	1063	231
2	446	376	314
3	478	514	298
4	1200	1178	189
5	443	367	314
6	1283	1294	215
7	356	338	426
8	380	365	440
	H, 7, 7		
-2	1047	1017	303
-1	1540	1559	224
0	596	637	296
1	1895	1972	216
2	755	790	302
3	768	776	254
4	723	705	248
5	505	504	309
6	338	329	470
7	784	780	278
	H, 8, 7		
-2	403	472	518
-1	325	294	449
0	1324	1435	226
2	1824	1832	248
3	1029	986	254
4	1284	1252	237
5	739	784	288
6	415	424	431
7	360	319	470
	H, 9, 7		
-4	1145	1111	362
-1	613	625	320
1	829	861	269
2	675	684	325
3	1092	1069	287

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$ Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 9, 7			5	2030	2100	167	-4	449	484	498
4	439	480	458	6	314	230	363	-3	1107	1138	294
5	1044	1028	265	7	612	590	280	-2	1378	1375	274
6	402	493	464		H, 2, 8			0	702	725	253
7	918	926	297	-4	532	498	278	1	1547	1583	241
	H, 10, 7			-3	604	657	275	2	394	436	382
-3	983	981	349	-2	506	540	291	3	246	201	376
-1	369	395	497	-1	2996	3103	201	4	346	323	362
0	617	602	293	1	1062	1066	168	5	1526	1566	202
1	563	565	313	2	1303	1311	149	6	473	528	357
2	427	427	428	3	1218	1228	156	8	644	599	328
3	969	940	328	4	514	524	226		H, 7, 8		
4	707	673	344	5	439	439	279	-5	594	518	456
6	472	491	446	6	1583	1614	186	-4	615	563	411
	H, 11, 7				H, 3, 8			-2	931	942	327
-2	778	791	349	-6	503	497	406	-1	734	727	298
0	1078	1093	273	-5	488	474	368	1	1068	1100	249
1	345	399	502	-4	657	696	291	2	2623	2614	220
2	612	701	352	-3	900	917	239	3	1201	1146	227
3	375	287	538	-2	487	462	307	4	481	429	322
4	642	600	380	-1	416	342	395	6	614	606	289
5	501	374	439	0	1807	1877	208	7	459	507	384
	H, 12, 7			2	1858	1854	150		H, 8, 8		
-1	693	658	350	3	2534	2498	147	-4	639	664	461
1	1235	1262	261	4	1622	1638	165	-3	655	606	406
3	705	689	347	5	727	697	206	-1	512	485	319
4	513	456	480	6	219	255	317	2	943	892	298
	H, 13, 7			7	726	749	259	3	2176	2118	220
0	388	363	489		H, 4, 8			4	700	734	320
1	375	369	266	-5	735	682	293	5	696	736	305
2	873	912	313	-4	819	768	270	6	208	229	396
3	305	256	548	-3	797	820	260		H, 9, 8		
4	461	520	587	-2	709	760	282	-3	440	409	550
5	530	501	500	1	571	580	280	-2	432	389	430
	H, 0, 8			2	689	689	216	-1	371	351	415
-4	462	428	280	3	2278	2288	159	1	747	765	258
-3	2125	2200	163	4	1694	1711	167	2	268	151	440
0	477	477	215	5	1103	1148	198	3	723	755	332
1	1061	1089	140	6	659	700	251	4	1447	1421	243
2	1016	1011	142		H, 5, 8			6	817	833	311
3	788	785	164	-5	458	489	466		H, 10, 8		
4	2652	2675	153	-4	1119	1075	270	0	844	935	284
6	970	964	214	-3	1333	1261	239	2	671	708	333
7	303	346	346	-2	431	465	491	4	604	575	372
	H, 1, 8			0	480	478	297	5	723	704	346
-6	743	730	299	2	356	350	355	6	415	402	508
-4	453	477	316	3	610	571	234	7	720	851	413
-3	759	778	214	4	2061	2025	171		H, 11, 8		
-2	2916	3021	158	5	1067	1086	202	-1	375	343	490
0	413	400	358	6	382	405	371	1	1322	1418	254
1	288	247	246	7	589	591	303	6	390	320	492
2	208	210	233	8	365	403	405		H, 12, 8		
3	672	676	182		H, 6, 8			-1	264	334	429
4	674	654	200	-5	387	345	522	0	312	301	431

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o^*$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 12, 8			3	2482	2481	157	1	788	835	273
2	1289	1348	271	4	379	350	304	2	1667	1612	254
4	475	467	491	5	462	464	303	3	515	568	409
5	394	336	469	6	416	433	331	4	280	266	379
	H, 13, 8			7	318	273	296	5	271	305	452
0	542	586	398	8	473	388	360	6	1163	1156	255
3	866	888	330		H, 4, 9			7	652	651	332
	H, 0, 9			-5	1120	1028	273		H, 9, 9		
-6	661	689	330	-4	1000	981	272	-2	691	612	325
-5	268	233	305	-3	512	500	352	-1	364	324	442
-4	1826	1829	189	-2	379	358	471	0	572	602	308
-3	274	232	302	-1	323	324	366	1	500	504	343
-2	763	808	203	1	898	848	238	2	925	993	298
-1	328	342	367	2	1453	1422	181	3	838	818	318
1	782	800	166	3	956	973	193	7	1129	1199	289
3	1331	1347	158	4	1636	1677	178		H, 10, 9		
4	804	828	206	7	789	813	266	-3	464	439	513
5	1754	1759	178	8	535	543	365	-1	1078	1099	276
7	394	379	399		H, 5, 9			1	563	586	316
	H, 1, 9			-5	355	268	373	2	421	441	439
-6	280	275	425	-4	1301	1257	279	3	432	491	523
-5	439	400	334	-3	983	949	277	4	450	380	418
-3	2538	2573	175	-2	303	280	361	5	377	324	469
-2	411	414	316	-1	1283	1300	241		H, 11, 9		
-1	686	685	272	0	393	427	351	-2	388	354	369
0	1378	1370	206	2	674	695	243	0	1292	1377	260
1	1244	1268	158	3	1653	1645	182	2	276	275	353
2	612	609	193	4	321	361	327	3	486	487	465
3	430	391	226	5	918	879	214	6	417	464	556
4	1350	1371	175	7	792	793	276		H, 12, 9		
5	666	688	236	8	827	826	296	0	286	283	378
6	1315	1365	199		H, 6, 9			1	1044	1147	285
	H, 2, 9			-3	985	978	322	4	515	487	511
-6	635	635	339	-2	983	939	314	5	738	783	425
-5	463	469	377	0	1738	1871	209		H, 13, 9		
-3	315	287	425	1	1280	1337	259	1	337	353	395
-2	2003	2066	198	2	593	558	304	2	565	528	361
-1	735	781	318	3	697	734	245	3	437	378	287
0	303	279	319	4	556	558	287		H, 0, 10		
1	1592	1590	168	5	285	279	382	-5	903	906	257
2	2189	2197	150	6	395	344	386	-3	1646	1702	185
3	265	259	310		H, 7, 9			-2	287	331	416
4	750	746	197	-1	894	917	264	-1	752	790	259
5	345	287	319	0	327	402	454	3	911	953	186
7	981	966	229	1	1795	1858	226	4	2148	2176	169
	H, 3, 9			2	1212	1194	263	5	306	275	316
-6	615	571	363	3	519	519	370		H, 1, 10		
-5	735	737	307	4	568	595	308	-5	338	301	398
-4	526	545	338	5	587	535	297	-4	811	772	243
-2	550	567	315	6	515	510	332	-3	299	253	444
-1	1038	1037	277		H, 8, 9			-2	1834	1876	197
0	940	974	252	-2	481	389	399	-1	566	578	367
1	498	490	286	-1	408	344	388	0	1911	1854	206
2	1065	1057	180	0	770	772	248	1	771	772	206

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 1, 10				H, 6, 10			4	1000	988	386
2	855	892	179	-3	849	856	355		H, 0, 11		
3	387	384	282	-2	423	418	523	-4	1644	1658	214
4	539	558	256	-1	640	697	320	-2	319	265	312
5	1621	1651	187	0	1249	1298	227	-1	831	829	293
6	193	162	349	1	1288	1297	261	0	1171	1126	190
7	565	587	302	2	912	864	260	1	395	401	307
	H, 2, 10			3	694	728	289	2	515	541	247
-6	524	531	415	5	478	523	362	3	1098	1112	183
-4	552	545	316	6	407	394	370	4	749	780	232
-3	502	469	331	7	1017	1030	264	5	936	909	216
-2	407	393	375		H, 7, 10			7	1102	1136	248
-1	1024	1073	300	-4	449	418	588		H, 1, 11		
0	235	245	426	-2	593	626	395	-3	1292	1289	219
1	2315	2281	173	0	269	338	492	-2	322	298	470
2	1040	1027	179	1	1013	1069	275	-1	747	734	352
3	1260	1246	170	2	1819	1746	244	0	977	1025	277
4	962	957	194	3	676	611	297	1	2107	2070	168
6	877	864	230	4	1283	1263	231	2	515	554	257
7	321	302	470	5	235	296	442	3	312	277	338
	H, 3, 10			6	262	210	402	4	484	472	272
-5	670	664	329	7	664	638	324	5	231	203	373
-4	249	226	461		H, 8, 10			6	345	400	439
-3	500	494	332	-3	400	315	515		H, 2, 11		
1	364	313	376	-2	648	590	333	-5	521	484	374
2	2123	2072	171	-1	284	256	403	-2	706	693	297
3	1474	1414	173	0	697	667	268	-1	575	638	395
4	829	835	218	2	667	673	397	0	1233	1227	273
5	954	993	221	3	1258	1225	268	1	1182	1176	208
6	383	355	366	5	1248	1192	256	2	1812	1792	171
	H, 4, 10			6	299	287	368	3	481	489	278
-5	569	568	439	7	537	542	389	5	389	329	328
-4	889	794	276		H, 9, 10			6	443	498	372
-1	558	593	364	-3	449	365	328		H, 3, 11		
0	285	268	472	-1	854	842	283	-5	403	405	501
1	614	587	276	0	270	307	506	-3	491	489	404
2	637	611	262	1	499	523	360	0	808	842	306
3	2125	2102	177	3	379	306	456	1	1571	1605	216
4	833	805	217	4	464	476	447	2	559	558	273
6	661	701	280	5	576	584	370	3	1144	1122	194
7	595	641	323	6	1122	1083	277	4	333	334	395
	H, 5, 10				H, 10, 10			5	590	596	285
-5	472	477	525	-2	658	613	356	6	595	617	287
-4	738	730	368	0	884	968	283	7	699	723	304
-3	829	801	325	1	322	328	479		H, 4, 11		
-2	596	635	423	6	803	799	347	-5	888	866	332
-1	1074	1112	259		H, 11, 10			-3	289	248	469
0	1027	1148	238	-1	603	487	329	-2	1139	1082	290
1	745	695	306	1	933	975	285	-1	603	666	318
3	373	368	374	2	307	327	353	0	284	230	313
4	1381	1350	200	3	832	797	326	1	763	757	296
5	326	289	231	5	422	367	322	2	1356	1339	213
6	821	834	272		H, 12, 10			3	252	269	381
7	737	740	293	2	476	652	490	4	454	411	293

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 4, 11			4	356	412	521	5	946	970	247
5	284	214	423	5	586	600	419	6	775	859	283
6	1069	1094	242	6	548	465	430		H, 4, 12		
7	704	728	303		H, 11, 11			-3	676	626	376
	H, 5, 11			0	883	912	289	-2	1049	1073	349
-4	708	674	382	2	534	533	377	-1	545	603	355
-3	382	297	499	3	414	393	523	0	529	626	367
-2	637	632	432	4	887	814	362	3	823	807	237
-1	1428	1479	236	5	539	480	397	4	253	289	422
0	1053	1129	256		H, 12, 11			5	1394	1386	220
2	388	377	400	1	511	527	403	6	579	613	341
3	526	505	301	2	363	383	515	7	756	766	307
4	365	334	405	3	501	515	466		H, 5, 12		
7	1313	1332	243	4	268	233	500	-4	554	478	455
	H, 6, 11				H, 0, 12			-2	809	817	365
-2	421	474	523	-5	840	843	305	-1	1268	1367	259
-1	710	784	313	-3	568	621	322	0	713	765	286
0	1586	1647	225	-2	454	482	364	1	498	521	431
1	1187	1152	265	-1	1891	1872	271	2	760	711	287
2	483	441	378	1	565	563	245	3	242	261	379
4	575	543	300	3	517	532	271	4	260	241	321
5	773	769	259	4	1158	1187	209	5	433	433	388
6	251	273	439	6	1168	1223	237	6	1245	1293	247
	H, 7, 11				H, 1, 12			7	529	533	406
-2	396	408	334	-5	348	314	495		H, 6, 12		
-1	571	572	338	-4	373	413	479	-3	626	519	380
0	782	770	266	-3	219	248	417	-2	410	361	481
1	1217	1267	251	-2	256	251	325	-1	599	596	330
2	540	482	426	-1	323	341	632	0	923	1032	276
3	501	469	344	0	2261	2213	227	1	716	709	353
5	1047	1014	262	1	421	457	385	2	265	192	405
6	908	862	270	2	642	659	230	3	1433	1358	232
	H, 8, 11			3	714	710	225	4	581	597	341
-3	696	697	416	5	759	750	242	6	540	548	346
-1	559	551	332	6	387	409	432	7	801	823	302
0	466	508	373		H, 2, 12				H, 7, 12		
1	661	689	303	-2	466	425	413	-3	567	548	467
2	772	787	351	-1	396	369	441	-2	495	486	438
4	478	475	413	1	1996	1968	202	-1	471	530	383
5	378	392	518	2	471	462	297	1	357	412	495
6	1109	1094	279	4	935	912	209	2	671	569	329
	H, 9, 11			5	701	695	256	3	371	277	454
-2	1061	1069	296	6	329	249	433	4	1581	1543	241
1	358	372	425	7	608	587	318	5	705	755	344
2	362	303	482		H, 3, 12				H, 8, 12		
3	293	358	520	-4	365	352	322	-2	620	677	370
4	609	606	381	-3	548	502	364	-1	378	392	434
5	389	462	574	-2	569	448	356	0	264	322	483
6	559	587	415	-1	562	585	356	3	273	251	420
	H, 10, 11			0	624	594	357	4	777	717	308
-2	307	261	457	1	364	376	469	5	1115	1099	282
-1	1151	1138	268	2	1359	1300	208	6	412	423	486
1	262	264	445	3	320	266	394		H, 9, 12		
2	431	375	425	4	743	722	241	-2	356	275	542

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 9, 12			3	364	375	411	0	1546	1418	251
-1	554	579	402	4	712	680	255	2	452	452	342
0	418	342	368	5	359	224	420	3	669	637	259
1	273	330	452	6	1289	1293	244	4	785	772	261
5	899	899	320		H, 5, 13			5	290	311	470
6	772	773	361	-2	869	885	352		H, 2, 14		
	H, 10, 12			-1	1063	1136	267	-4	440	492	546
0	457	362	379	0	731	748	299	-2	463	481	475
2	1006	996	283	1	867	823	337	-1	453	505	443
4	476	430	403	2	291	253	386	1	874	801	259
	H, 11, 12			4	851	785	252	3	1021	991	240
3	1125	1157	320	5	628	583	300	4	521	559	346
	H, 0, 13				H, 6, 13			5	886	881	257
-4	882	855	277	-2	416	313	468		H, 3, 14		
-2	1106	1061	232	-1	722	712	289	-3	958	879	334
-1	468	516	567	0	931	1015	267	-1	352	386	522
0	1419	1362	205	2	801	819	340	3	329	320	471
1	689	692	235	3	583	575	366	4	1429	1408	223
3	549	532	270	4	902	842	272	5	576	572	338
6	357	367	531	5	901	912	290	6	634	632	313
	H, 1, 13				H, 7, 13				H, 4, 14		
-3	368	345	437	-1	401	388	462	-3	595	505	476
-2	449	423	400	0	374	394	461	-2	1040	1003	345
-1	1389	1470	285	1	579	590	368	1	555	603	445
0	559	623	412	2	406	384	547	2	442	449	407
1	1258	1217	205	3	748	704	335	4	420	420	409
2	865	817	213	4	635	619	354	5	1449	1446	241
3	510	473	281	5	867	797	300	6	327	331	491
5	546	590	326	6	641	580	326		H, 5, 14		
	H, 2, 13				H, 8, 13			-2	464	516	512
-4	483	395	400	3	807	827	366	-1	741	753	323
-3	259	284	401	4	723	683	360	2	1044	1034	288
-1	685	724	344	5	616	592	394	3	936	929	280
0	1479	1387	270	6	405	401	461	5	353	384	518
1	370	434	452		H, 9, 13			6	841	796	290
2	467	514	345	0	521	546	376		H, 6, 14		
3	475	457	304	2	377	374	509	-2	420	348	483
4	942	922	223	3	450	411	499	-1	411	387	312
5	540	527	323	4	804	781	354	2	507	457	432
6	815	824	283	5	439	461	545	3	1116	1063	282
	H, 3, 13				H, 10, 13			4	855	790	285
-4	492	437	453	1	788	764	301		H, 7, 14		
-3	547	506	412	3	666	693	431	-2	285	324	456
-2	562	581	443	4	510	577	521	3	874	833	325
0	534	579	377		H, 11, 13			4	1091	1061	295
1	997	1012	274	2	676	735	371	5	459	392	472
5	1326	1337	231		H, 0, 14				H, 8, 14		
6	352	411	510	-1	1470	1569	296	0	388	410	464
	H, 4, 13			2	439	402	321	2	368	426	455
-3	782	803	401	4	446	460	388	4	899	884	338
-2	1000	942	351	5	421	490	446	5	676	642	397
-1	845	885	304		H, 1, 14				H, 9, 14		
0	298	392	355	-3	477	433	422	1	766	794	344
2	370	386	459	-2	491	425	405	2	399	463	564

Table G.3. Obs and Calcd Structure Factors for $Os_4(\mu\text{-}H)(CO)_{14}(SnMe_3)$
Columns are $10F_o$, $10F_c$, 100σ. * Denotes an Insignificant Reflection.

H	KF_o	F_c	σ	H	KF_o	F_c	σ	H	KF_o	F_c	σ
	H, 9, 14			-1	507	551	426	1	599	565	409
3	303	320	491	1	868	890	392	3	315	302	466
	H, 10, 14			2	799	766	344	4	1042	1035	277
2	915	936	319	4	657	632	343		H, 4, 16		
3	394	376	458		H, 6, 15			0	543	467	406
	H, 0, 15			0	430	463	423	1	706	645	395
-2	1144	1067	283	1	478	504	483	2	779	812	360
0	481	454	387	2	801	744	362		H, 5, 16		
1	591	612	320	3	794	736	321	1	726	686	436
2	718	675	267	4	374	293	296	2	900	775	333
3	253	168	403	5	391	410	493	3	682	658	361
4	574	589	318		H, 7, 15			4	318	313	384
	H, 1, 15			1	313	362	467		H, 6, 16		
-2	460	423	538	2	834	787	383	0	324	377	470
-1	762	868	355	3	528	501	491	2	778	772	409
2	333	370	400	4	532	446	375	3	563	541	438
3	1036	1035	244		H, 8, 15				H, 7, 16		
5	606	580	317	1	319	327	374	1	488	444	437
	H, 2, 15			2	440	428	505	3	652	606	416
-3	484	480	485	3	637	641	457		H, 0, 17		
-1	367	307	521	4	439	410	481	0	401	385	496
0	529	601	524		H, 9, 15			2	916	911	300
2	639	653	319	2	305	347	448		H, 1, 17		
4	1118	1064	242	3	483	470	588	1	808	800	331
	H, 3, 15				H, 0, 16			3	843	826	288
-3	432	381	603	-1	583	629	426		H, 2, 17		
-2	477	451	320	1	814	750	274	1	483	480	475
-1	380	410	450	2	273	268	452	2	920	855	303
1	269	264	533	3	765	711	267		H, 3, 17		
2	409	449	443		H, 1, 16			0	526	614	526
3	893	921	277	-2	456	324	562	2	543	480	398
5	1000	962	267	2	1182	1119	237	3	537	555	418
	H, 4, 15			3	372	364	326		H, 4, 17		
-2	509	460	467	4	673	658	306	0	660	675	412
0	753	820	347		H, 2, 16			1	843	815	397
1	644	585	380	2	395	354	365	3	500	347	392
3	728	687	304	3	1285	1278	251		H, 5, 17		
4	618	600	326		H, 3, 16			1	684	619	427
	H, 5, 15			0	265	328	521	2	659	605	420
-2	457	477	448								

Table H.1. U_{ij} or U_{iso} Values (*10^4) for $Os_4(CO)_{13}(CNBu^t)$

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Os(1)	370(7)	323(8)	497(10)	53(8)	-12(7)	-32(7)
Os(2)	368(8)	286(8)	411(9)	13(7)	-19(7)	7(7)
Os(3)	418(9)	387(10)	553(10)	-28(9)	-32(7)	-67(8)
Os(4)	376(8)	297(9)	460(9)	32(8)	32(7)	40(8)
N(1)	601(95)					
O(11)	925(107)					
O(12)	847(100)					
O(13)	576(74)					
O(14)	527(70)					
O(21)	692(86)					
O(22)	773(93)					
O(23)	881(105)					
O(31)	605(76)					
O(32)	849(96)					
O(33)	1071(124)					
O(34)	950(114)					
O(41)	591(79)					
O(42)	675(81)					
O(43)	778(95)					
O(44)	849(100)					
C(11)	639(121)					
C(12)	630(116)					
C(13)	359(85)					
C(14)	394(89)					
C(15)	482(98)					
C(21)	341(85)					
C(22)	607(117)					
C(23)	777(140)					
C(31)	446(93)					
C(32)	694(126)					
C(33)	637(124)					
C(34)	836(166)					
C(41)	328(84)					
C(42)	428(91)					
C(43)	456(102)					
C(44)	532(113)					
C(1)	615(116)					
C(2)	1105(116)					
C(3)	1105(116)					
C(4)	1105(116)					
Cl(1)	2322(136)					
Cl(2)	2322(136)					
Cl(3)	2322(136)					

Table H.2. Hydrogen Atom Coordinates for $Os_4(CO)_{15}(CNBu^t)$.

Atom	x/a	y/b	z/c	U(iso)
H(1)	0.5387	-0.1855	0.4738	0.14(7)
H(2)	0.5556	-0.1608	0.3839	0.14(7)
H(3)	0.5176	-0.0629	0.4369	0.14(7)
H(4)	0.4449	-0.3542	0.4404	0.14(7)
H(5)	0.4608	-0.3385	0.3494	0.14(7)
H(6)	0.3689	-0.3365	0.3797	0.14(7)
H(7)	0.3867	-0.1832	0.5140	0.14(7)
H(8)	0.3781	-0.0631	0.4699	0.14(7)
H(9)	0.3170	-0.1658	0.4467	0.14(7)

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{11}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
** K= 0 L= 0 **			
2	574	585	5
4	299	304	186
6	205	201	184
8	243	238	6
10	137	128	9
12	99	93	9
14	128	133	10
** K= 1 L= 0 **			
0	144	110	184
1	77	54	186
2	91	88	189
4	80	70	5
6	442	440	7
8	360	361	7
9	118	126	187
10	147	149	187
11	224	225	188
12	201	194	188
13	191	185	189
14	88	77	8
15	80	77	189
16	88	76	11
** K= 2 L= 0 **			
0	291	270	6
1	73	83	2
2	115	122	186
4	558	554	186
5	191	191	188
6	105	104	192
7	238	243	188
9	120	111	185
10	118	114	189
11	107	111	9
12	108	125	188
13	85	68	11
15	90	93	189
** K= 3 L= 0 **			
0	124	122	8
1	75	76	183
2	220	232	186
3	100	104	1
4	554	530	187
5	206	205	7
6	191	192	188
7	166	157	10
10	126	121	189
11	121	123	191
12	149	140	188
13	125	112	190
** K= 4 L= 0 **			
0	563	506	182
1	80	75	3
2	277	264	186
3	96	78	4
4	58	65	186
6	306	311	7
8	226	220	8
9	99	85	10
10	168	163	189
11	208	209	9
12	176	178	190
13	205	208	9
15	82	74	10
16	91	80	9
** K= 5 L= 0 **			
0	870	873	7
2	387	383	7
4	236	236	187
5	97	85	5
6	97	89	189
8	168	156	8
9	67	62	6
10	95	105	10
12	78	74	9
14	98	109	10
** K= 6 L= 0 **			
1	75	43	187
4	140	146	6
6	338	343	9
8	287	296	9
10	76	72	188
11	143	134	189
12	95	109	190
13	106	101	191
** K= 7 L= 0 **			
0	264	268	8
4	257	253	188
5	159	152	188
6	86	74	189
7	189	180	190
9	88	69	191
** K= 8 L= 0 **			
2	148	133	189
4	303	309	189
5	116	103	8
6	105	107	191
7	107	104	9
10	90	87	189
11	116	121	191
12	102	106	189
13	133	122	190
** K= 9 L= 0 **			
0	129	150	188
2	196	187	188
4	96	88	189
6	115	116	10
8	77	74	11
10	114	130	191
11	98	122	11
** K= 10 L= 0 **			
0	429	413	9
2	154	158	9
4	163	158	189
5	77	62	9
6	92	81	190
8	99	76	10
** K= 11 L= 0 **			
4	107	89	9
6	230	222	11

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
** K= 12	L= 0 **		
0	187	183	10
** K= 0	L= 1 **		
-16	54	61	187
-12	277	277	187
-10	309	311	188
-8	74	70	3
-6	248	253	7
-4	92	78	189
-2	203	205	185
2	469	471	6
4	251	250	187
6	395	379	186
8	221	205	8
10	381	378	9
12	222	217	10
14	97	109	10
** K= 1	L= 1 **		
-16	97	100	190
-15	67	63	8
-14	150	146	10
-13	152	154	9
-12	113	110	9
-11	207	205	9
-10	171	181	187
-9	99	107	9
-8	62	50	191
-6	205	204	5
-5	63	55	1
-3	298	287	7
-2	360	373	185
-1	554	565	6
0	84	67	2
1	467	464	6
2	192	188	7
3	109	109	9
4	141	144	3
6	264	259	5
8	240	248	9
9	69	79	11
11	77	84	9
12	248	247	189
16	204	203	10
** K= 2	L= 1 **		
-14	110	121	9
-11	110	100	188
-10	130	131	187
-9	156	159	7
-8	99	93	8
-7	416	401	9
-6	232	234	8
-5	241	243	14
-4	68	46	189
-3	144	128	5
-2	180	170	189
-1	262	259	186
0	165	148	6
1	214	214	184
2	179	176	6
3	230	234	7
4	415	419	185
5	519	518	6
6	345	356	187
7	239	241	8
10	76	65	6
12	129	123	189
14	71	69	191
** K= 3	L= 1 **		
-14	152	152	9
-13	115	114	9
-11	123	114	10
-10	73	75	190
-9	101	93	188
-8	85	80	11
-7	358	359	188
-6	214	222	9
-5	343	339	188
-4	128	122	184
-2	182	187	189
-1	384	385	6
0	159	165	5
1	268	273	6
2	100	99	9
3	195	201	185
4	378	368	186
5	350	347	187
6	355	349	187
7	158	160	189
12	171	173	189
14	114	121	190
** K= 4	L= 1 **		
-14	152	157	10
-13	138	150	190
-12	149	150	8
-11	211	218	188
-9	90	98	189
-6	134	135	8
-4	100	90	184
-3	280	290	187
-2	217	218	188
-1	506	473	187
0	134	122	181
1	593	557	185
2	108	98	8
3	207	208	185
6	110	115	6
8	226	217	7
9	121	116	187
10	75	61	193
11	89	93	188
12	254	261	190
14	68	54	190
16	162	156	10
** K= 5	L= 1 **		
-14	84	57	189
-12	172	181	190
-11	63	69	188
-10	206	214	190
-7	95	102	187
-6	166	157	10
-5	112	105	187
-4	88	95	187
-3	110	107	187
-2	151	156	186
0	296	288	8
2	297	298	8
3	95	94	186
4	221	213	187
5	86	92	186
6	295	298	187
8	191	192	7
10	312	321	9

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	$/F_O/$	$/F_C/$	PHI
12	175	174	10
14	92	78	11
** K= 6 L= 1 **			
-14	92	96	10
-13	93	88	11
-11	148	145	9
-10	145	133	190
-9	68	71	9
-8	88	84	188
-6	115	101	7
-4	80	71	187
-3	137	131	8
-2	182	190	188
-1	410	373	6
1	322	300	6
2	160	150	9
4	168	165	4
5	80	81	184
6	185	184	8
8	255	254	9
9	81	92	7
11	83	61	9
12	175	171	190
** K= 7 L= 1 **			
-13	61	44	188
-10	95	101	190
-9	111	118	9
-7	347	357	9
-6	184	183	8
-5	343	351	9
-3	101	103	8
-2	118	131	188
-1	137	134	187
0	105	95	10
1	56	51	186
2	143	141	7
3	221	228	7
4	219	216	187
5	341	341	8
6	222	225	188
7	193	200	8
** K= 8 L= 1 **			
-13	104	98	11
-11	125	116	10
-8	81	68	10
-7	176	175	189
-6	162	159	9
-5	201	196	188
-2	138	145	189
-1	337	316	8
0	105	99	6
1	279	276	7
2	84	76	9
4	239	222	188
5	169	163	188
6	209	221	189
7	78	58	191
12	150	138	189
** K= 9 L= 1 **			
-11	113	117	191
-9	87	43	191
-6	82	109	9
-3	142	147	189
-2	148	130	189
-1	243	253	189
1	253	248	188
3	85	80	189
8	63	63	10
** K= 10 L= 1 **			
-9	103	81	191
-6	125	110	10
-5	81	97	190
-3	109	100	189
-2	125	101	188
0	172	148	10
1	82	83	188
2	168	167	9
3	87	82	189
4	154	140	188
5	79	71	190
6	165	165	190
7	80	61	191
8	101	81	9
9	78	69	189
** K= 11 L= 1 **			
-2	122	98	189
-1	138	118	9
1	94	80	9
2	100	96	9
4	93	74	8
6	139	122	10
** K= 12 L= 1 **			
2	103	87	10
3	132	107	11
** K= 0 L= 2 **			
-16	125	113	189
-14	170	164	190
-12	248	258	188
-10	339	348	187
-8	78	75	185
-6	406	404	6
-4	95	95	358
-2	759	766	187
0	849	790	186
4	79	67	10
6	106	117	192
8	95	87	187
10	94	101	10
14	99	83	190
** K= 1 L= 2 **			
-16	148	136	189
-14	94	109	9
-13	99	100	189
-12	287	285	8
-11	81	77	187
-10	120	116	187
-8	477	484	187
-6	221	214	189
-5	53	47	183
-4	58	50	2
-3	83	82	9
-1	329	324	6
1	270	277	6
2	198	207	6
3	71	64	6
4	268	263	184
5	87	89	186

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
6	377	368	187
9	214	217	9
10	152	148	8
11	276	276	9
13	155	159	10
14	104	98	189
16	92	92	10
** K= 2 L= 2 **			
-12	133	137	8
-8	65	75	7
-6	407	403	7
-4	323	328	7
-3	81	80	358
-2	259	270	184
-1	201	202	187
0	118	105	185
2	380	377	6
3	231	232	8
4	293	284	5
5	657	652	6
6	73	66	190
7	352	362	7
10	185	186	8
11	165	152	189
14	102	104	190
15	111	124	12
** K= 3 L= 2 **			
-14	79	80	10
-12	150	138	10
-8	87	95	8
-6	397	397	8
-5	124	129	184
-4	374	367	7
-3	62	46	7
-2	146	134	184
-1	225	236	7
0	69	60	181
1	115	112	9
2	485	484	6
3	335	337	184
4	398	384	5
5	512	492	186
6	94	87	186
7	268	270	187
9	72	92	8
10	165	173	8
11	201	197	9
14	112	103	190
15	96	87	191
** K= 4 L= 2 **			
-15	98	79	10
-14	99	111	11
-13	113	112	9
-12	280	289	9
-11	80	71	8
-8	321	322	188
-6	129	123	188
-4	110	100	8
-3	102	109	188
-1	189	190	190
0	133	127	8
1	361	347	185
2	228	226	7
3	91	88	185
6	300	310	186
9	247	255	187
10	179	174	7
11	253	256	190
13	167	165	190
14	110	97	190
16	86	61	12
** K= 5 L= 2 **			
-14	140	144	190
-12	190	191	189
-10	205	212	189
-6	230	239	9
-2	583	586	188
0	523	525	188
1	86	85	184
3	114	113	185
4	143	130	6
5	103	86	188
6	180	175	187
7	61	62	191
9	81	62	190
10	96	117	8
** K= 6 L= 2 **			
-13	79	67	188
-12	136	148	10
-10	125	118	190
-8	420	419	188
-6	213	228	189
-1	183	158	8
1	174	167	8
2	90	83	7
4	179	182	187
5	102	104	186
6	330	331	187
8	62	64	187
9	163	159	9
10	89	86	10
11	166	165	10
13	91	88	10
14	94	79	190
** K= 7 L= 2 **			
-6	246	240	8
-5	93	101	7
-4	153	140	9
-2	146	153	190
-1	96	98	189
0	132	125	188
2	145	149	9
3	264	274	6
4	74	80	11
5	437	440	7
6	94	84	189
7	263	265	8
10	119	128	9
11	101	94	189
** K= 8 L= 2 **			
-12	105	114	10
-8	71	67	8
-6	244	254	10
-4	235	235	9
-3	87	39	9
-1	159	164	9
1	200	186	7
2	280	280	8
3	82	64	190
4	166	163	9
5	221	224	189

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
7	97	101	189
9	108	124	8
10	95	96	11
11	139	164	11
** K= 9	L= 2 **		
-8	98	116	191
-4	127	138	9
-1	141	131	190
0	104	116	10
1	120	125	189
2	168	168	9
6	121	118	189
9	124	128	189
10	118	99	11
11	165	168	191
** K= 10	L= 2 **		
-6	164	168	11
-2	316	303	190
0	272	267	189
1	91	58	188
3	63	65	192
5	100	103	189
7	100	84	190
9	93	76	190
** K= 11	L= 2 **		
-6	165	155	190
4	109	102	190
5	104	89	188
6	176	167	190
** K= 12	L= 2 **		
-2	133	130	190
0	139	117	189
3	114	101	10
** K= 0	L= 3 **		
-12	128	134	8
-10	72	64	9
-8	63	54	191
-6	124	117	4
-2	493	487	187
0	862	830	186
4	438	437	7
8	478	489	186
10	423	423	187
12	188	178	189
14	132	128	190
** K= 1	L= 3 **		
-15	132	126	189
-14	77	70	190
-13	239	233	189
-12	160	157	7
-11	203	202	188
-10	86	84	8
-8	297	296	187
-6	240	239	187
-5	176	173	184
-4	182	179	8
-3	254	255	188
-2	190	181	9
-1	367	366	186
1	216	227	185
2	95	93	187
3	107	116	184
4	392	390	184
5	70	74	186
6	535	532	186
8	167	166	189
10	268	274	7
11	84	94	8
12	179	171	9
13	55	45	11
14	133	134	190
16	154	141	191
** K= 2	L= 3 **		
-14	79	75	190
-13	136	126	9
-12	89	90	8
-9	284	282	188
-8	94	107	188
-7	470	474	187
-6	60	51	6
-5	316	312	186
-4	261	266	7
-3	91	95	178
-1	86	94	8
0	292	281	186
1	150	153	182
2	157	158	8
3	159	157	187
4	506	514	7
5	61	54	191
6	198	199	6
9	89	88	185
10	122	111	8
11	86	101	188
12	117	121	9
15	89	84	13
** K= 3	L= 3 **		
-16	88	82	190
-14	84	90	189
-13	162	159	189
-11	71	70	188
-9	221	221	8
-8	107	115	187
-7	376	381	7
-6	79	65	7
-5	155	147	10
-4	358	350	6
-3	119	109	187
-1	181	181	187
0	157	156	187
2	260	270	6
3	123	123	6
4	624	607	6
6	258	255	6
9	72	61	8
10	125	122	10
11	110	117	8
12	163	166	9
15	98	77	191
** K= 4	L= 3 **		
-15	145	136	9
-14	84	87	189
-13	214	220	10
-12	85	93	10
-11	201	205	8
-10	83	75	9

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
-8	236	236	188
-6	192	193	187
-5	91	91	9
-4	226	231	7
-3	285	279	7
-2	228	224	8
-1	234	235	9
1	213	213	6
3	61	61	10
6	279	282	187
9	59	49	185
10	326	324	7
11	89	83	189
12	187	192	10
13	74	57	190
14	83	88	190
** K= 5 L= 3 **			
-12	87	78	11
-11	76	72	8
-10	77	54	8
-9	82	71	9
-8	70	67	188
-7	83	87	8
-6	54	56	8
-5	119	123	6
-4	118	108	7
-3	62	57	10
-2	323	313	189
0	573	585	188
2	66	75	187
3	71	62	5
4	404	408	7
6	93	88	5
8	351	347	188
10	254	277	188
12	143	138	189
14	111	104	190
** K= 6 L= 3 **			
-14	70	51	190
-13	132	141	190
-12	90	93	11
-11	125	127	189
-8	210	213	189
-7	56	53	7
-6	175	180	189
-4	138	145	7
-3	143	158	189
-2	131	123	9
-1	206	181	188
0	75	74	188
1	73	64	190
2	70	80	192
4	223	224	188
6	416	427	187
8	230	231	188
10	132	132	9
12	121	106	9
14	142	132	190
** K= 7 L= 3 **			
-12	93	79	9
-9	231	233	189
-8	76	59	192
-7	357	363	188
-5	193	188	189
-4	169	159	8
0	216	218	188
1	83	75	188
3	143	138	188
4	278	276	9
5	97	82	188
6	86	70	9
8	94	105	187
11	83	75	188
** K= 8 L= 3 **			
-11	97	87	189
-9	77	74	10
-7	165	158	10
-5	80	73	8
-4	213	220	8
-3	119	131	189
-2	102	83	6
-1	177	153	190
0	92	77	190
2	156	151	9
4	301	302	9
6	106	121	8
10	97	95	11
11	85	72	10
12	129	137	10
** K= 9 L= 3 **			
-11	102	108	11
-8	120	122	190
-4	138	139	9
-3	133	158	9
-2	149	162	10
-1	181	163	9
1	110	116	8
6	84	99	189
10	210	207	10
** K= 10 L= 3 **			
-9	64	70	12
-7	74	84	11
-5	86	81	12
-3	112	78	10
-2	175	165	190
0	310	309	190
4	246	237	9
6	88	70	8
8	167	158	190
** K= 11 L= 3 **			
-6	106	117	190
2	73	65	193
4	119	133	190
6	241	232	190
** K= 12 L= 3 **			
0	141	147	190
** K= 0 L= 4 **			
-16	142	138	9
-14	192	191	9
-12	281	276	9
-10	217	216	9
-8	133	136	187
-6	173	174	188
-4	282	275	8
-2	447	453	8
2	202	199	183

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
4	208	210	10
6	225	221	7
8	236	236	188
10	400	398	186
** K=	1	L=	4 **
-14	197	202	190
-12	85	86	189
-10	256	263	8
-8	278	279	7
-7	53	57	9
-3	364	341	187
-2	153	158	7
-1	299	288	190
0	130	133	186
1	199	199	189
2	194	196	186
3	51	57	180
4	89	90	8
7	150	146	188
8	80	100	188
9	314	311	187
10	113	103	6
11	211	218	188
12	183	191	9
13	93	93	189
16	217	204	191
** K=	2	L=	4 **
-14	113	100	190
-13	61	58	9
-9	146	152	188
-8	222	223	188
-7	312	299	188
-6	394	398	187
-5	155	153	188
-2	135	133	8
-1	294	286	6
0	163	156	188
1	122	123	185
2	223	211	187
3	240	242	194
4	109	113	10
5	485	487	187
6	142	144	8
7	190	195	187
8	154	154	188
10	100	104	188
12	106	111	9
14	83	92	9
15	106	99	190
** K=	3	L=	4 **
-14	138	142	189
-12	88	73	189
-9	121	121	9
-8	243	247	188
-7	295	302	7
-6	378	393	187
-5	108	105	8
-4	56	49	187
-3	277	274	186
-2	174	172	4
-1	279	261	189
0	200	205	188
2	237	247	187
3	329	326	8
4	157	157	6
5	425	417	7
6	148	149	7
7	137	137	6
8	131	122	189
9	153	160	187
10	83	79	187
11	121	129	187
12	127	129	10
14	75	79	12
** K=	4	L=	4 **
-14	230	223	190
-12	119	131	189
-11	62	52	7
-10	187	186	8
-8	143	147	8
-6	77	88	187
-5	64	67	9
-3	377	370	7
-2	84	90	6
-1	344	329	9
0	128	130	188
1	256	255	7
2	142	138	188
4	115	116	5
6	66	61	6
7	116	135	8
8	75	75	189
9	275	277	8
10	127	125	6
11	207	205	9
12	163	172	10
13	105	99	9
** K=	5	L=	4 **
-14	138	145	10
-12	213	211	10
-10	166	175	9
-8	108	114	188
-6	189	177	187
-5	49	43	8
-4	271	266	7
-2	417	417	7
1	81	71	8
2	83	83	188
3	93	82	9
4	221	230	8
5	110	123	7
6	221	232	6
7	98	97	8
8	208	208	188
9	97	95	6
10	265	272	188
12	65	53	187
** K=	6	L=	4 **
-14	152	138	190
-10	241	244	9
-8	249	248	8
-7	62	78	7
-6	69	81	6
-4	132	129	7
-3	189	187	188
-2	173	182	7
-1	266	291	187
0	77	54	188
1	105	96	191
2	110	112	187
4	101	112	6
7	90	111	187

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
8	98	89	189
9	197	193	188
10	67	47	8
11	121	126	189
12	124	135	9
14	94	69	189
** K= 7	L= 4 **		
-9	121	106	190
-8	87	106	189
-7	249	256	188
-6	196	209	188
-5	122	132	189
-4	54	59	5
-3	97	96	7
-2	132	124	10
-1	145	140	8
0	82	93	188
1	87	85	190
2	143	157	186
3	357	362	188
4	126	115	9
5	409	405	188
6	107	110	8
7	156	169	188
8	112	115	189
10	98	100	189
13	91	78	190
** K= 8	L= 4 **		
-9	71	78	9
-8	151	150	190
-7	120	134	9
-6	241	242	189
-5	66	44	8
-3	199	203	188
-2	86	68	7
-1	259	247	190
0	161	154	189
2	167	167	189
3	184	184	9
4	90	79	9
5	191	198	9
6	96	92	9
8	85	91	188
9	125	142	189
11	114	116	189
12	106	109	10
** K= 9	L= 4 **		
-3	201	203	9
-1	217	218	10
0	114	94	190
1	117	129	9
2	88	93	190
9	129	145	10
** K= 10	L= 4 **		
-8	89	88	190
-6	99	113	190
-4	110	118	10
-2	206	187	10
-1	72	59	9
2	89	74	189
3	102	94	10
4	124	122	10
5	117	127	8
6	119	111	10
7	114	113	9
8	100	99	190
9	113	94	8
** K= 11	L= 4 **		
-4	68	67	11
-2	133	97	10
** K= 0	L= 5 **		
-16	98	87	10
-14	132	111	7
-8	139	137	183
-6	159	164	188
-4	276	265	7
-2	1112	1148	5
0	472	460	8
2	260	258	186
4	223	227	186
6	259	261	7
8	286	298	7
10	137	137	7
12	145	140	9
14	139	138	10
** K= 1	L= 5 **		
-16	81	64	10
-15	154	151	10
-14	108	106	190
-13	184	193	8
-12	160	168	189
-11	113	111	8
-10	191	184	8
-8	394	405	8
-6	115	118	5
-5	101	108	8
-4	186	180	189
-3	123	114	7
-2	250	249	183
-1	80	105	2
3	110	102	7
4	268	275	8
5	75	75	9
6	358	346	8
9	248	251	187
10	203	205	188
11	200	207	188
13	65	66	188
14	134	137	9
** K= 2	L= 5 **		
-12	98	98	189
-11	76	94	8
-9	205	218	9
-7	167	162	9
-6	298	306	187
-5	89	89	6
-4	151	149	189
-2	153	166	8
-1	243	236	3
2	490	465	186
3	115	104	192
4	233	239	189
5	159	154	190
8	75	79	187
9	141	146	9
10	188	181	188
11	118	113	9
13	86	86	189

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
15	214	203	189
** K= 3 L= 5 **			
-15	91	92	9
-13	99	96	9
-12	94	99	189
-9	164	169	188
-7	168	159	187
-6	325	331	187
-4	236	244	187
-2	188	185	4
0	76	75	185
2	414	423	188
3	104	120	10
4	285	292	188
5	173	173	9
8	115	111	187
9	186	193	188
10	217	220	188
11	177	174	187
12	78	70	188
13	71	51	8
15	154	161	10
** K= 4 L= 5 **			
-15	158	152	190
-14	106	115	189
-13	173	179	189
-12	171	181	189
-11	107	106	189
-10	143	142	8
-8	329	332	8
-5	113	113	188
-4	268	271	187
-3	78	95	187
-2	218	215	185
3	129	133	186
4	173	182	8
5	50	46	193
6	215	231	8
8	106	119	187
9	189	196	8
10	230	227	189
11	199	195	9
13	81	79	8
14	94	92	10
** K= 5 L= 5 **			
-10	69	55	7
-9	58	44	190
-8	51	42	191
-7	67	65	189
-6	177	191	187
-5	68	60	189
-4	185	187	8
-2	705	707	7
0	393	410	8
2	163	169	189
4	130	120	191
6	223	215	8
7	66	49	5
8	206	218	9
10	102	83	8
12	107	105	9
14	105	106	10
** K= 6 L= 5 **			
-14	76	81	190
-13	126	112	9
-12	106	115	190
-10	160	173	9
-8	322	335	9
-6	114	105	8
-5	75	70	8
-4	142	143	187
-3	83	71	8
0	95	91	6
2	113	114	8
4	261	258	10
6	338	351	8
9	168	155	188
10	130	117	189
11	90	119	188
14	116	112	11
** K= 7 L= 5 **			
-11	94	99	9
-9	194	205	9
-7	158	157	8
-6	152	165	188
-3	64	51	10
-2	197	198	8
0	99	96	8
2	247	249	187
3	97	95	189
4	175	167	188
5	145	144	189
9	92	80	11
10	73	83	190
11	73	72	9
13	88	90	189
** K= 8 L= 5 **			
-6	204	205	189
-4	166	174	188
2	298	308	189
3	95	92	9
4	191	193	190
5	108	108	10
9	150	157	189
10	163	160	189
11	125	133	189
** K= 9 L= 5 **			
-8	146	150	10
-5	94	77	188
-4	200	204	189
-2	124	122	190
0	75	80	190
2	84	97	188
6	88	78	9
8	99	101	191
9	94	104	11
10	163	171	190
** K= 10 L= 5 **			
-7	81	81	189
-6	132	118	190
-5	104	82	188
-4	82	77	10
-2	320	324	10
0	190	189	10
2	141	134	190
4	116	112	191
6	115	97	10
8	116	106	11

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
** K= 11	L= 5 **		
2	98	84	10
4	184	183	10
5	96	54	9
** K= 0	L= 6 **		
-16	90	87	190
-14	146	148	188
-12	200	210	189
-10	71	98	188
-8	65	54	13
-4	205	220	187
-2	146	153	6
0	236	236	9
2	194	193	187
4	478	483	187
6	126	125	5
8	455	457	8
10	293	295	8
** K= 1	L= 6 **		
-16	81	67	12
-15	85	84	10
-14	104	112	9
-13	117	124	10
-12	102	110	189
-10	159	153	188
-8	97	83	8
-5	195	202	8
-4	305	300	187
-3	434	430	7
-2	187	187	186
-1	447	419	6
0	167	152	7
1	175	187	6
2	150	163	6
4	73	61	11
5	93	96	7
6	149	160	8
7	142	135	9
9	130	134	8
10	211	218	189
11	88	85	7
12	84	77	190
14	219	219	9
** K= 2	L= 6 **		
-16	90	70	11
-15	68	73	190
-14	85	75	10
-13	102	109	188
-11	84	76	9
-9	322	332	8
-8	205	208	9
-7	295	309	9
-6	82	97	7
-5	75	80	5
-4	83	88	191
-3	173	176	188
0	262	249	7
1	285	286	6
2	159	149	186
3	355	349	8
4	313	321	188
5	246	252	8
7	71	76	7
8	143	138	8
10	77	70	188
12	121	129	189
** K= 3	L= 6 **		
-15	104	83	11
-14	116	99	10
-13	101	108	9
-11	81	73	187
-10	64	49	9
-9	276	271	188
-8	228	235	8
-7	250	261	188
-6	90	91	8
-4	70	73	193
-3	289	285	7
-2	92	94	5
-1	259	244	6
0	208	212	8
1	82	85	192
2	100	108	189
3	283	296	188
4	265	279	189
5	130	125	191
8	77	94	9
10	130	124	187
12	183	189	188
14	83	72	188
** K= 4	L= 6 **		
-15	97	88	189
-14	130	136	9
-13	106	121	190
-11	76	60	188
-10	80	88	189
-8	137	137	7
-5	199	199	188
-4	163	170	189
-3	360	379	188
-2	157	154	186
-1	384	356	187
0	141	153	6
1	144	154	188
2	105	107	7
5	60	67	189
6	100	86	10
7	151	137	188
9	121	130	189
10	244	251	189
11	77	82	188
12	109	109	189
14	155	149	10
** K= 5	L= 6 **		
-12	164	176	189
-10	74	62	190
-8	83	79	9
-7	93	86	186
-4	188	189	187
-3	90	88	186
-2	120	119	7
0	230	232	8
1	92	93	186
2	160	158	188
3	82	82	189
4	322	337	188
8	355	342	9
10	218	217	9
** K= 6	L= 6 **		
-13	106	100	9

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
-12	126	127	189
-10	148	147	189
-5	105	104	8
-4	240	248	187
-3	260	272	7
-2	109	106	189
-1	195	199	9
0	111	108	9
2	147	142	7
4	81	64	12
6	167	169	8
7	108	118	8
9	85	81	9
10	145	150	190
** K=	7	L= 6	**
-11	78	74	8
-9	248	261	9
-8	125	128	9
-7	273	284	9
-4	133	133	187
-3	112	105	188
0	168	176	7
1	165	189	9
2	84	104	187
3	270	286	9
4	230	236	188
5	181	175	10
7	55	60	10
8	122	127	9
11	65	67	8
** K=	8	L= 6	**
-9	141	147	189
-8	142	149	10
-7	128	134	189
-5	53	44	11
-4	98	107	187
-3	238	241	9
-1	156	191	9
0	153	159	8
2	70	80	188
3	139	137	189
4	177	192	189
10	92	92	189
** K=	9	L= 6	**
-9	82	53	9
-8	106	105	10
-5	136	137	188
-4	87	92	190
-3	223	217	190
-1	170	191	189
0	72	84	7
7	88	86	188
9	100	95	188
10	173	179	190
** K=	10	L= 6	**
-7	83	62	190
-3	93	80	189
-1	93	62	189
0	124	141	10
2	104	96	189
3	74	83	191
4	193	195	191
5	74	71	192
7	82	85	189
8	189	185	11
** K=	11	L= 6	**
-4	109	114	191
-3	95	90	10
2	71	67	10
** K=	0	L= 7	**
-16	143	127	191
-14	153	149	188
-12	132	132	188
-10	62	47	188
-8	172	172	7
-6	59	53	3
-4	536	539	187
-2	650	656	187
2	185	185	4
4	198	197	188
6	204	203	189
12	131	133	189
14	113	105	190
** K=	1	L= 7	**
-15	80	80	191
-14	187	184	10
-10	357	359	188
-9	74	75	189
-8	258	263	188
-6	71	79	7
-4	72	68	6
-3	171	176	6
-1	124	116	9
0	71	75	5
3	110	118	187
4	282	287	187
6	140	132	189
7	147	145	12
8	111	107	6
9	241	236	10
11	154	152	9
12	114	123	188
** K=	2	L= 7	**
-14	91	69	10
-11	81	84	187
-8	222	224	8
-6	278	290	8
-4	74	59	183
-3	186	197	188
-2	107	113	188
-1	131	120	188
0	265	247	7
1	217	217	7
2	327	323	7
3	413	403	7
5	259	269	7
8	187	200	8
9	160	143	189
10	121	104	9
12	110	81	189
13	128	135	11
15	177	171	10
** K=	3	L= 7	**
-14	84	81	11
-12	77	66	9
-8	242	243	8

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
-6	291	300	8
-5	68	70	7
-3	200	205	8
-1	138	136	8
0	323	316	7
1	147	143	188
2	331	351	7
3	336	349	188
5	192	190	189
7	92	85	10
8	180	194	9
9	188	182	10
10	113	106	10
13	133	104	190
** K= 4 L= 7 **			
-15	98	80	12
-14	191	201	9
-13	85	73	10
-10	228	241	189
-9	68	77	8
-8	142	150	188
-7	90	97	7
-6	123	116	10
-4	130	123	8
-3	144	151	188
-1	169	165	186
0	85	100	9
3	93	91	8
4	183	187	187
6	91	94	186
7	165	174	189
8	100	116	8
9	220	215	191
11	180	173	189
12	96	93	189
** K= 5 L= 7 **			
-14	91	91	191
-12	84	95	189
-8	138	134	9
-4	395	409	188
-2	488	491	188
2	79	95	9
4	164	166	188
5	84	79	187
6	176	178	189
12	109	104	190
** K= 6 L= 7 **			
-10	308	320	189
-8	243	243	189
-3	81	68	11
-2	96	99	188
-1	98	95	9
2	82	93	188
3	88	91	189
4	253	266	188
6	130	144	189
7	128	141	9
9	167	169	9
12	88	98	190
** K= 7 L= 7 **			
-8	115	117	9
-6	149	154	9
-4	80	87	188
-3	169	166	188
-2	186	165	188
0	106	95	9
1	158	168	8
2	147	152	9
3	291	302	8
5	146	167	10
8	116	123	9
9	71	72	191
** K= 8 L= 7 **			
-8	142	147	10
-6	222	224	9
-3	118	130	9
-1	138	125	8
0	192	208	9
1	85	83	187
2	223	232	9
3	199	205	189
5	88	92	190
7	96	102	10
8	126	134	10
9	151	156	11
** K= 9 L= 7 **			
-6	134	137	9
-4	116	118	10
-3	88	82	191
-1	86	69	190
0	100	120	9
4	67	56	189
7	108	98	190
8	99	96	10
9	157	157	190
** K= 10 L= 7 **			
-4	189	205	190
-2	234	239	190
2	100	89	10
3	77	71	189
6	94	96	190
** K= 11 L= 7 **			
-3	92	49	8
-1	78	58	8
2	63	65	191
3	76	74	190
** K= 0 L= 8 **			
-12	102	109	9
-10	58	57	6
-8	90	79	5
-6	63	65	11
-4	208	208	187
-2	535	537	187
2	444	427	7
4	190	207	6
6	365	382	188
8	367	375	188
10	121	125	190
12	100	91	190
14	86	84	189
** K= 1 L= 8 **			
-15	108	121	191
-13	120	105	191
-12	117	107	10

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	$/F_o/$	$/F_c/$	PHI
-10	141	136	188
-8	228	234	187
-7	96	94	188
-6	120	112	9
-5	223	231	188
-4	312	321	7
-3	285	279	187
-1	182	173	188
0	153	142	188
1	125	128	186
2	219	205	186
3	139	143	186
4	237	235	188
5	94	100	188
6	147	142	190
8	151	155	7
10	169	169	10
12	97	84	190
14	173	182	190

** K= 2 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-15	73	75	12
-12	62	44	11
-11	181	188	189
-9	316	322	188
-7	192	189	188
-6	100	117	8
-2	175	171	188
-1	94	91	187
1	156	150	188
2	386	380	7
3	95	104	187
4	205	220	8
5	81	75	185
6	78	76	187
7	105	100	189
9	124	135	189
10	148	153	9

** K= 3 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-15	104	94	192
-11	136	130	10
-9	228	239	8
-7	129	140	8
-6	137	141	7
-5	72	72	187
-3	107	110	188
-2	154	144	188
1	108	117	6
2	380	399	7
4	220	228	8
7	75	85	8
9	110	105	10
10	198	200	9
12	91	90	10
13	94	67	192

** K= 4 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-13	116	118	10
-12	84	90	9
-10	115	115	189
-8	195	197	188
-7	99	96	8
-6	103	96	9
-5	228	242	8
-4	251	259	8
-3	230	229	9
-1	153	163	8
0	136	134	185
1	130	132	7
2	85	70	188
3	126	124	7
4	135	147	187
5	87	82	9
6	75	80	188
8	164	175	9
10	185	192	10
14	130	121	191

** K= 5 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-12	87	97	9
-8	68	58	6
-6	70	66	11
-5	75	76	8
-4	163	160	188
-2	417	425	188
0	111	120	187
1	89	86	6
2	299	318	8
4	146	145	8
6	279	286	189
8	266	284	189
10	112	96	191
12	73	74	189

** K= 6 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-12	105	107	9
-10	93	92	189
-8	134	150	190
-6	89	82	10
-5	132	129	189
-4	205	207	9
-3	156	175	188
-1	118	97	188
0	106	125	190
2	177	178	187
4	228	240	188
6	168	169	190
10	90	101	11
12	100	99	190

** K= 7 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-11	150	150	190
-9	232	249	189
-7	149	164	189
-6	78	72	9
-2	165	165	189
-1	121	115	188
1	136	145	188
2	209	210	9
3	98	94	188
4	97	109	10
5	57	50	189
6	83	84	189
7	60	69	190

** K= 8 L= 8 **

H	$/F_o/$	$/F_c/$	PHI
-9	104	115	9
-6	112	108	8
-5	104	100	189
-4	78	69	7
-3	130	130	189
-2	106	83	190
2	200	214	10
4	113	125	10
10	148	145	10

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
** K= 9	L= 8 **		
-8	132	125	190
-5	119	132	10
-4	158	164	10
-3	154	141	9
8	135	137	11
** K= 10	L= 8 **		
-5	93	82	10
-4	76	82	189
-2	217	217	190
2	212	209	10
4	90	101	10
6	152	148	190
** K= 11	L= 8 **		
0	84	80	191
** K= 0	L= 9 **		
-14	103	97	12
-12	124	108	11
-8	104	79	189
-6	210	199	7
-4	438	448	7
-2	118	131	9
2	204	206	8
4	327	336	7
6	98	94	188
8	289	294	188
12	125	117	9
** K= 1	L= 9 **		
-14	88	83	190
-12	134	143	10
-10	205	204	9
-9	81	79	10
-5	182	186	188
-4	176	186	6
-3	267	264	188
-1	127	132	189
0	99	114	188
4	111	124	7
5	133	129	188
7	244	241	188
8	66	58	6
9	190	197	189
10	133	138	10
11	97	92	190
14	179	173	191
** K= 2	L= 9 **		
-10	72	77	190
-9	175	174	189
-8	250	260	188
-7	78	79	189
-6	115	118	187
-5	132	119	10
-4	74	77	9
-3	239	241	7
0	246	249	188
1	301	294	189
3	364	363	188
4	154	144	7
5	151	174	186
6	108	109	188
8	186	184	188
11	83	49	193
12	115	103	9
13	85	92	191
** K= 3	L= 9 **		
-14	79	76	191
-11	73	78	9
-10	89	88	190
-9	141	150	9
-8	247	261	188
-7	62	61	9
-6	152	157	187
-5	180	183	187
-3	246	246	188
-2	112	120	189
0	312	319	187
1	220	221	10
3	299	310	8
4	108	110	8
5	71	77	8
6	85	98	189
7	122	118	188
8	186	184	188
9	87	92	188
12	77	94	11
** K= 4	L= 9 **		
-14	106	97	191
-12	90	103	9
-10	127	136	9
-9	106	90	189
-6	91	85	188
-5	207	210	7
-3	244	254	8
-1	146	142	8
0	186	187	187
4	77	66	9
5	98	101	9
7	213	216	8
9	184	183	10
10	102	114	11
11	96	101	10
** K= 5	L= 9 **		
-12	93	98	10
-8	103	76	189
-6	115	124	9
-4	305	309	8
-2	95	83	10
2	160	158	9
3	111	92	8
4	236	254	8
5	81	80	8
6	79	84	187
7	70	69	8
8	232	226	189
12	108	96	10
** K= 6	L= 9 **		
-12	156	147	11
-10	185	194	10
-5	93	109	188
-4	150	161	8
-3	169	183	188
-1	101	89	188
3	83	40	10
5	58	64	191
7	153	154	189

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
9	96	94	191
10	109	108	11
11	85	46	189
** K= 7 L= 9 **			
-9	138	137	189
-8	117	135	190
-7	89	86	188
-5	119	111	8
-4	88	88	11
-3	140	146	8
0	123	114	190
1	262	264	189
3	272	281	189
4	107	96	10
5	119	111	190
6	61	67	189
8	108	119	190
** K= 8 L= 9 **			
-9	123	100	10
-8	147	161	190
-6	106	91	190
-5	145	136	188
-3	174	191	189
-2	111	100	189
0	159	170	190
1	147	147	8
3	146	167	9
6	61	71	190
7	137	121	190
8	106	98	190
9	107	90	191
** K= 9 L= 9 **			
-8	77	74	191
-5	117	117	10
-3	161	150	10
-1	92	83	8
0	83	102	191
7	127	111	11
** K= 10 L= 9 **			
-4	151	156	10
3	103	89	10
4	122	134	11
** K= 0 L= 10 **			
-8	96	83	190
-6	88	85	8
-4	582	572	6
-2	409	401	8
0	104	114	187
2	236	225	187
4	184	179	8
6	307	324	8
8	103	111	9
10	78	69	11
12	136	145	10
** K= 1 L= 10 **			
-14	137	116	190
-10	258	245	10
-8	94	94	8
-7	119	117	8
-6	181	187	189
-5	103	101	9
-4	177	178	186
-3	78	67	8
0	87	98	7
1	112	104	10
2	146	147	8
3	114	105	10
4	231	233	8
6	83	66	8
7	140	140	188
8	152	139	188
9	111	116	189
12	122	117	10
** K= 2 L= 10 **			
-11	120	136	10
-9	116	106	11
-8	103	97	189
-6	133	129	189
-4	84	79	8
-3	171	155	7
-2	63	79	9
0	259	260	187
1	98	97	187
2	287	285	188
3	148	147	187
7	159	161	9
8	141	148	189
9	132	125	9
10	108	87	190
13	161	147	191
** K= 3 L= 10 **			
-11	82	85	191
-9	85	80	189
-8	113	100	190
-6	158	169	188
-3	79	84	187
0	270	271	188
2	290	311	188
3	113	120	9
7	189	182	188
8	179	176	189
9	127	134	190
10	96	114	191
13	120	126	11
** K= 4 L= 10 **			
-13	91	58	192
-10	194	197	10
-7	117	120	189
-6	212	218	188
-5	84	95	189
-4	192	203	186
-1	78	73	189
1	136	162	187
3	100	106	189
4	144	145	8
7	108	101	10
8	150	159	190
9	121	126	9
12	81	81	10
** K= 5 L= 10 **			
-8	95	95	188
-4	372	377	8
-2	340	331	8
0	97	90	189

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_o/	/F_c/	PHI
2	163	159	190
4	139	142	9
6	222	230	9
12	122	122	10
** K=	6	L= 10	**
-10	193	196	11
-8	91	87	9
-6	127	118	190
-2	84	94	8
0	125	129	8
1	76	87	8
2	157	170	9
3	112	109	7
4	214	236	9
7	105	104	188
8	69	65	189
** K=	7	L= 10	**
-9	86	99	11
-7	65	54	9
-4	96	96	10
-3	124	131	8
-2	106	113	9
0	141	122	189
2	165	158	190
3	86	69	190
7	83	111	10
8	89	72	190
** K=	8	L= 10	**
-6	102	93	192
0	185	185	189
1	104	91	8
2	194	194	190
3	105	109	9
7	110	120	190
8	145	136	190
** K=	9	L= 10	**
-6	141	143	191
-5	68	58	190
-4	102	112	190
** K=	10	L= 10	**
-2	156	157	10
2	102	125	190
** K=	0	L= 11	**
-14	84	93	190
-12	97	92	191
-6	136	139	189
-2	126	104	10
0	131	144	187
2	413	405	187
4	97	90	186
6	350	345	8
8	247	246	9
** K=	1	L= 11	**
-12	109	118	190
-8	92	78	7
-7	98	92	10
-6	166	158	188
-5	219	220	9
-4	218	225	187
-3	230	228	8
-1	102	107	7
0	168	163	7
5	148	136	9
7	137	114	10
8	140	146	189
9	112	100	8
10	109	92	190
11	61	77	9
12	123	132	10
13	90	75	10
** K=	2	L= 11	**
-11	155	145	11
-10	109	108	10
-9	149	146	12
-8	120	122	9
-5	109	123	189
-2	204	188	8
-1	175	169	8
1	270	266	8
2	227	208	188
3	174	169	9
4	91	63	187
5	78	78	8
6	153	148	8
9	85	77	8
10	105	108	190
** K=	3	L= 11	**
-11	130	132	191
-10	124	122	10
-9	117	114	192
-8	120	125	10
-5	163	157	9
-3	103	108	9
-2	170	179	8
-1	97	92	188
1	218	212	188
2	177	186	188
3	136	120	189
6	126	122	9
10	142	141	190
** K=	4	L= 11	**
-12	94	88	189
-7	91	91	191
-6	96	100	189
-5	201	210	189
-4	154	153	188
-3	222	228	188
-1	147	120	187
0	151	157	8
3	89	75	189
5	122	135	188
7	107	128	189
8	132	144	189
9	90	88	190
10	100	97	190
11	77	70	190
** K=	5	L= 11	**
-6	119	112	189
-2	99	112	9
0	108	95	188
2	279	296	189
4	67	45	191

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{11}(C_4Bu^t)$.

H	$/F_o/$	$/F_c/$	PHI
6	260	259	9
8	200	200	9
** K= 6 L= 11 **			
-6	111	118	191
-5	140	146	9
-4	148	153	190
-3	143	128	9
0	114	121	8
5	88	93	10
8	89	98	190
** K= 7 L= 11 **			
-9	130	133	11
-2	115	112	9
-1	173	156	9
1	194	222	9
2	130	146	189
3	124	144	9
6	107	116	10
** K= 8 L= 11 **			
-8	98	91	10
-5	127	131	10
-3	139	131	9
-2	117	121	10
2	102	107	190
6	98	82	10
** K= 9 L= 11 **			
-5	133	142	190
-3	124	124	189
0	88	87	10
** K= 0 L= 12 **			
-6	214	206	190
-4	376	372	189
-2	111	111	189
2	118	141	187
4	267	257	188
10	100	119	189
12	154	142	191
** K= 1 L= 12 **			
-12	171	165	190
-10	158	158	190
-6	95	85	10
1	122	123	188
2	143	135	188
4	164	158	188
5	97	110	9
7	177	171	9
9	108	97	10
10	86	83	189
** K= 2 L= 12 **			
-8	138	146	9
-5	152	150	190
-4	106	88	188
-3	125	116	189
-2	85	90	9
0	232	233	9
1	231	237	8
2	98	76	8
3	171	168	7
6	122	126	9
7	133	136	189
8	151	141	10
11	109	103	11
** K= 3 L= 12 **			
-10	63	65	10
-8	150	153	10
-5	123	117	11
-3	105	108	9
-2	152	147	8
-1	94	76	189
0	270	270	8
1	248	229	188
3	143	137	188
5	104	88	8
6	119	141	9
7	158	153	10
8	150	156	10
** K= 4 L= 12 **			
-10	112	114	190
-8	88	74	11
-6	110	105	11
1	116	105	9
5	103	101	189
7	145	161	190
9	119	122	189
** K= 5 L= 12 **			
-6	156	171	190
-4	270	271	190
-2	97	89	189
0	101	78	9
2	92	90	189
4	186	178	190
** K= 6 L= 12 **			
-10	148	150	191
2	130	144	189
4	161	152	190
7	108	121	10
** K= 7 L= 12 **			
-8	75	76	11
-5	110	121	191
-4	79	100	190
0	135	131	9
1	170	178	9
3	95	104	9
** K= 8 L= 12 **			
-2	112	97	11
0	185	181	10
1	140	140	190
5	70	74	10
** K= 0 L= 13 **			
-12	90	91	9
-4	201	206	189
0	231	233	9
2	182	175	9
4	225	214	188
6	319	315	188
8	97	93	190

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
** K= 1 L= 13 **			
-10	139	127	189
-7	107	119	190
-6	210	215	9
-5	128	130	190
-3	105	99	190
-2	132	135	188
-1	66	83	189
0	108	124	188
1	144	127	187
2	101	100	187
3	116	102	189
4	111	73	190
8	121	120	10
** K= 2 L= 13 **			
-11	161	166	189
-9	97	93	189
-4	121	86	190
-1	87	89	191
0	138	156	9
2	154	166	9
5	118	92	190
7	120	127	190
8	108	93	10
** K= 3 L= 13 **			
-11	122	121	10
-10	104	78	190
-1	83	76	7
0	155	162	9
2	153	171	8
7	110	113	9
8	141	121	10
10	111	109	10
** K= 4 L= 13 **			
-10	121	128	190
-7	112	123	10
-6	159	155	11
-5	123	125	11
-3	115	115	9
-2	103	97	189
-1	106	93	9
0	78	64	191
1	105	111	9
3	134	102	9
6	82	90	8
8	134	130	11
** K= 5 L= 13 **			
-4	122	147	191
0	209	198	9
2	173	167	9
4	158	158	189
6	221	238	190
** K= 6 L= 13 **			
-6	142	158	11
-5	102	83	190
-2	96	92	190
0	88	93	190
4	83	94	189
** K= 7 L= 13 **			
-2	79	79	187
-1	125	106	190
0	100	105	10
2	111	113	10
5	81	95	189
6	81	56	191
** K= 8 L= 13 **			
2	106	99	10
** K= 0 L= 14 **			
-6	204	213	9
-4	122	121	10
0	100	106	10
2	212	218	10
6	183	167	189
10	135	122	11
** K= 1 L= 14 **			
-11	81	75	10
-5	106	107	190
-4	88	83	10
2	97	69	10
3	72	67	189
5	168	146	189
7	128	130	189
9	66	73	190
** K= 2 L= 14 **			
-10	104	103	190
-8	98	91	190
-7	102	92	11
-5	135	133	10
-2	145	143	190
-1	112	116	194
1	165	173	190
6	126	150	190
** K= 3 L= 14 **			
-10	122	113	190
-8	131	105	190
-7	118	97	190
-5	146	132	190
-2	154	162	190
-1	128	132	10
0	101	86	190
1	159	159	10
6	158	166	189
** K= 4 L= 14 **			
-5	110	99	11
-2	79	76	190
5	146	135	9
7	130	129	10
** K= 5 L= 14 **			
-6	147	158	10
2	209	198	9
6	137	150	189
** K= 6 L= 14 **			
-4	120	99	10
5	100	106	189

Table H.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(CNBu^t)$.

H	/F_O/	/F_C/	PHI
** K= 7	L= 14 **		
-5	120	100	10
-1	129	139	190
1	138	138	191
** K= 0	L= 15 **		
-6	207	194	8
-4	169	170	11
0	121	116	191
2	83	78	8
4	210	208	10
6	116	110	9
** K= 1	L= 15 **		
-9	105	84	9
-8	109	109	190
-7	125	82	8
-6	144	137	188
-2	97	84	10
-1	89	86	12
1	106	97	11
2	116	89	10
** K= 2	L= 15 **		
-5	119	99	9
-2	99	73	189
0	147	156	190
5	148	137	9
7	90	96	11
8	106	97	190
** K= 3	L= 15 **		
-2	90	76	191
0	170	169	190
5	88	105	191
6	106	107	189
7	104	105	190
8	119	125	190
** K= 4	L= 15 **		
-8	98	106	190
-6	147	127	189
1	82	98	191
** K= 5	L= 15 **		
-6	158	149	9
-4	186	156	10
0	126	131	189
4	174	168	10
** K= 6	L= 15 **		
-2	76	85	10
2	117	96	11
** K= 0	L= 16 **		
-2	86	79	187
0	194	196	190
2	99	85	192
4	155	145	10
6	152	154	10
** K= 1	L= 16 **		
-7	109	104	9
-6	141	145	189
-5	118	100	9
-2	100	95	10
3	78	71	12
** K= 2	L= 16 **		
-4	79	84	10
-3	104	91	9
-1	127	131	10
** K= 3	L= 16 **		
-7	92	88	10
-1	107	101	190
1	95	68	189
** K= 4	L= 16 **		
-6	98	105	189
-2	106	112	9
3	79	79	190
** K= 5	L= 16 **		
0	163	172	190
2	80	93	190
** K= 0	L= 17 **		
-6	155	157	190
2	141	142	190
** K= 1	L= 17 **		
-1	88	87	190
2	106	81	191
** K= 2	L= 17 **		
-2	126	114	10
-1	113	108	9
** K= 3	L= 17 **		
-2	140	134	10
-1	115	105	190
1	94	67	189
** K= 4	L= 17 **		
-1	103	91	10
** K= 0	L= 18 **		
-2	112	118	10
0	149	140	9
** K= 1	L= 18 **		
-1	77	75	189

Table I.1. U_{ij} or U_{iso} Values (*10⁴) for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Os(1)	485(6)	494(6)	533(6)	-87(5)	251(5)	-83(5)
Os(2)	461(5)	400(5)	391(5)	-17(5)	177(4)	-37(5)
Os(3)	476(6)	456(6)	517(6)	-57(5)	184(5)	-110(5)
Os(4)	545(6)	471(6)	380(5)	12(5)	192(4)	-53(5)
P(1)	512(38)	448(39)	665(41)	-78(33)	311(34)	-36(32)
O(1)	668(167)	1203(277)	3014(574)	904(361)	264(219)	-218(171)
O(2)	781(155)	836(174)	2640(393)	-869(224)	918(202)	-322(138)
O(3)	795(178)	3496(703)	877(164)	-678(252)	475(139)	-916(297)
O(10)	668(167)	1203(277)	3014(574)	904(361)	264(219)	-218(171)
O(20)	781(155)	836(174)	2640(393)	-869(224)	918(202)	-322(138)
O(30)	795(178)	3496(703)	877(164)	-678(252)	475(139)	-916(297)
O(11)	1441(214)	754(156)	1360(198)	-491(146)	867(174)	-217(144)
O(12)	714(133)	990(166)	942(153)	247(140)	115(113)	133(129)
O(13)	962(146)	755(137)	785(128)	-208(112)	506(116)	-35(119)
O(14)	1059(166)	1262(195)	529(116)	193(129)	263(113)	278(151)
O(21)	1080(162)	417(110)	1069(159)	155(109)	520(132)	84(113)
O(22)	1022(151)	413(107)	895(138)	204(101)	419(117)	86(106)
O(23)	1105(166)	1171(185)	433(104)	-178(110)	269(107)	-252(140)
O(31)	933(152)	566(131)	1183(173)	173(125)	419(132)	30(121)
O(32)	702(128)	764(144)	917(143)	-6(124)	201(109)	1(120)
O(33)	1734(244)	1234(209)	433(119)	-171(127)	145(136)	-415(185)
O(34)	893(157)	766(153)	2019(261)	68(158)	956(178)	-226(127)
O(41)	1299(200)	691(146)	1106(177)	304(135)	450(153)	249(144)
O(42)	743(126)	414(103)	927(138)	-57(103)	245(106)	70(98)
O(43)	829(143)	1394(206)	535(116)	-124(130)	68(105)	-394(146)
O(44)	1404(202)	1125(197)	1039(162)	-21(143)	869(162)	-143(160)
C(1)	1632(189)					
C(2)	1192(133)					
C(3)	1505(176)					
C(4)	566(65)					
C(5)	774(83)					
C(11)	628(72)					
C(12)	715(79)					
C(13)	658(75)					
C(14)	641(71)					
C(21)	616(71)					
C(22)	558(64)					
C(23)	713(80)					
C(31)	621(70)					
C(32)	688(77)					
C(33)	654(75)					
C(34)	781(85)					
C(41)	595(69)					
C(42)	473(56)					
C(43)	578(67)					
C(44)	613(68)					
Cl(1)	1776(57)					
Cl(2)	1776(57)					
Cl(3)	1776(57)					
Cl(4)	1773(57)					
C(98)	2250(280)					

Table I.2. Hydrogen Atom Coordinates for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

Atom	*x/a*	*y/b*	*z/c*	U(iso)
H(1)	0.818(3)	0.779(3)	0.528(3)	0.09(3)
H(2)	0.802(3)	0.724(3)	0.448(3)	0.09(3)
H(3)	0.724(2)	0.999(2)	0.434(2)	0.09(3)
H(4)	0.743(2)	0.940(2)	0.513(2)	0.09(3)
H(5)	0.708(2)	0.911(3)	0.318(2)	0.09(3)
H(6)	0.770(2)	0.817(3)	0.323(2)	0.09(3)
H(7)	0.790(3)	0.786(3)	0.528(3)	0.09(3)
H(8)	0.827(3)	0.731(3)	0.468(3)	0.09(3)
H(9)	0.753(2)	0.954(2)	0.511(2)	0.09(3)
H(10)	0.709(2)	0.994(2)	0.423(2)	0.09(3)
H(11)	0.741(2)	0.900(3)	0.313(2)	0.09(3)
H(12)	0.749(2)	0.790(3)	0.331(2)	0.09(3)
H(13)	0.891	0.960	0.423	0.09(3)
H(14)	0.925	0.910	0.507	0.09(3)
H(15)	0.928	0.854	0.433	0.09(3)

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
** K= 0 L= 0 **			
1	388	710	11
2	1349	1439	183
3	1025	1100	192
4	2136	2204	11
5	4693	4975	7
6	2396	2429	188
7	1346	1452	189
8	2539	2714	189
9	1343	1505	9
10	529	590	192
11	1703	1825	188
12	1367	1341	189
13	826	888	190
15	730	712	192
** K= 1 L= 0 **			
1	5760	6081	7
2	1743	1827	4
3	676	599	2
4	4896	5303	185
5	3784	4146	8
6	2490	2771	8
7	1696	1731	6
8	1020	1101	187
9	702	778	190
10	1916	2006	10
13	1274	1252	189
** K= 2 L= 0 **			
0	6651	6575	185
1	2961	2867	5
2	1622	1718	6
3	2691	2756	7
4	942	985	189
5	4344	4337	185
6	1600	1644	8
8	1501	1652	8
9	2157	2207	189
14	618	721	191
** K= 3 L= 0 **			
1	1539	1341	187
2	1929	1784	9
3	567	538	180
4	4030	4194	8
6	2220	2109	7
7	882	1063	6
8	1023	1114	8
9	1635	1756	8
10	800	831	188
11	780	625	11
12	952	914	190
14	742	606	189
** K= 4 L= 0 **			
0	2565	2403	183
1	4948	4808	186
4	1193	1176	191
5	415	369	181
6	876	895	188
7	2669	2798	7
8	939	967	186
9	1453	1606	8
11	1156	1181	10
12	1112	1198	8
14	943	716	11
** K= 5 L= 0 **			
1	3506	3372	184
2	3642	3600	186
3	3017	2999	9
4	927	985	185
6	2813	2947	188
8	1162	1266	8
9	688	713	189
11	1240	1339	189
12	869	957	10
** K= 6 L= 0 **			
1	1659	1594	7
2	5552	5552	187
3	2383	2348	187
4	1891	1845	7
5	1707	1744	7
7	3075	3116	188
8	1320	1305	10
9	1020	865	10
10	1475	1548	9
13	784	823	11
** K= 7 L= 0 **			
1	2387	2299	8
2	2194	2229	6
3	5765	5894	187
4	2144	2208	188
5	1364	1546	188
6	1659	1691	9
7	1389	1449	189
8	2890	2930	188
11	1328	1245	10
12	757	740	191
** K= 8 L= 0 **			
1	1073	1095	187
2	4241	4298	8
3	1931	1965	8
4	1036	1177	187
5	1332	1319	187
7	2352	2326	9
8	1036	982	189
10	1452	1458	190
13	797	719	191
** K= 9 L= 0 **			
1	1071	1045	189
2	2015	1972	188
3	2834	2845	7
5	689	680	6
6	1631	1768	189
8	1230	1239	7
11	899	783	191
** K= 10 L= 0 **			
1	2190	2275	9
7	1445	1409	190
9	1219	1148	189
11	550	548	189
** K= 11 L= 0 **			
1	727	854	188
2	1488	1554	7

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
4	2171	2102	9
6	947	969	10
7	827	1008	8
8	779	807	10
9	902	746	9
11	583	487	13
** K= 12 L= 0 **			
0	1832	1828	9
1	1026	970	189
2	902	742	191
3	1071	1083	190
5	940	966	10
6	1035	1068	190
9	950	1155	10
** K= 13 L= 0 **			
1	1856	1858	10
4	1392	1443	189
5	1543	1554	10
6	943	1009	9
9	602	352	191
** K= 14 L= 0 **			
0	2794	2850	190
3	656	572	10
4	803	879	191
5	1171	1282	191
6	802	697	11
** K= 15 L= 0 **			
1	1435	1453	190
4	958	793	11
5	1104	1016	192
** K= 16 L= 0 **			
0	1111	1076	11
** K= 1 L= 1 **			
-11	570	677	188
-10	759	830	188
-8	741	969	188
-7	1742	1857	188
-6	1433	1627	189
-5	2315	2363	182
-4	1453	1529	2
-3	496	448	189
-2	1616	1531	188
-1	350	50	205
0	2793	2621	7
1	3722	3633	5
2	882	745	7
3	548	539	182
4	1992	2071	6
5	2361	2456	10
6	1803	1807	7
7	634	505	186
8	697	677	186
9	479	676	8
12	1032	936	190
13	714	626	189
** K= 2 L= 1 **			
-13	569	526	11
-12	861	892	10
-11	666	649	12
-8	1374	1521	5
-6	1822	1941	187
-5	4219	4303	185
-4	2682	2752	183
-3	724	741	4
-2	1622	1620	188
-1	6616	6431	186
0	4638	4239	187
1	1698	1520	4
2	1146	1042	14
3	1259	1254	188
4	4122	4163	186
5	1235	1286	2
6	3849	3922	7
7	2325	2526	8
9	615	623	185
10	1506	1574	9
11	1862	1994	9
12	684	722	9
** K= 3 L= 1 **			
-11	532	548	9
-10	1001	808	7
-7	2122	2216	7
-6	2291	2391	7
-5	709	815	357
-4	3590	3585	185
-3	1055	986	4
-2	3003	2872	9
-1	804	826	10
0	3083	2951	189
1	4475	4336	186
2	35[illegible]	242	34
3	325[illegible]	3307	6
4	1453	1519	186
5	4476	4753	187
6	2348	2436	186
7	2034	2139	7
8	1521	1617	8
9	1347	1447	188
10	1665	1641	189
12	1472	1438	10
13	701	542	10
14	415	396	191
15	448	88	193
** K= 4 L= 1 **			
-13	468	437	9
-10	880	857	7
-8	825	819	188
-7	923	1051	186
-6	1201	1199	6
-5	1206	1253	9
-4	1382	1385	187
-3	3912	3858	187
-2	2119	1981	185
-1	3146	3037	6
0	454	396	28
1	4143	3980	186
2	5034	4821	186
4	3931	4064	7
5	687	670	9
6	2308	2357	188
7	1145	1182	188
8	2517	2424	8
9	2378	2571	8
11	993	1113	189
13	979	955	10

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
** K= 5	L= 1 **		
-10	796	726	10
-9	1750	1787	8
-8	1314	1288	10
-7	422	447	8
-6	469	530	9
-5	1916	1960	8
-4	2395	2415	9
-2	2739	2725	187
-1	1872	1704	186
0	1929	1862	6
1	1180	1055	5
2	4050	3930	186
3	4094	4056	189
4	1456	1485	188
5	891	812	10
7	2373	2505	188
8	1863	1875	189
9	1014	979	7
10	1502	1522	8
11	821	528	8
14	810	832	12
** K= 6	L= 1 **		
-13	549	579	189
-11	660	731	189
-5	501	534	6
-4	2107	2096	7
-3	2646	2616	8
-2	1868	1887	6
0	461	482	11
1	2425	2373	7
2	2014	2041	7
4	1743	1844	187
6	456	295	9
8	1444	1399	189
9	1731	1704	189
13	868	629	191
** K= 7	L= 1 **		
-6	625	570	187
-3	804	857	4
-1	746	721	182
0	477	409	0
2	640	733	1
** K= 8	L= 1 **		
-10	795	692	190
-9	673	689	188
-7	707	628	10
-4	835	940	7
-3	2371	2382	7
-2	1412	1372	9
1	1448	1478	8
2	2286	2246	6
4	1219	1219	189
5	780	732	188
8	1412	1368	189
9	1111	968	191
** K= 9	L= 1 **		
-9	1105	1152	189
-8	1377	1497	189
-7	503	470	192
-5	1338	1380	188
-4	1925	1923	189
-3	607	666	188
-2	1913	1882	8
-1	1149	1231	8
0	904	1055	186
1	901	875	186
2	2384	2377	8
3	3700	3698	8
4	1314	1341	7
5	680	791	188
7	1985	1916	10
8	971	960	11
10	1244	1091	190
** K= 10	L= 1 **		
-8	475	481	190
-7	878	922	189
-5	624	557	11
-4	853	890	189
-3	2450	2406	189
-2	1255	1264	188
-1	1425	1416	8
0	968	873	7
1	2116	2106	188
2	2420	2478	189
4	1875	1943	9
6	1393	1461	190
7	524	498	190
8	1341	1318	10
9	1504	1566	9
** K= 11	L= 1 **		
-7	719	792	189
-6	1034	979	190
-4	1039	1028	9
-3	598	463	184
-2	1740	1798	189
-1	415	419	192
0	1598	1541	10
1	1811	1888	8
2	964	787	189
3	1570	1539	189
5	1873	1910	9
6	739	819	9
7	1414	1425	189
8	758	907	190
10	905	877	10
** K= 12	L= 1 **		
-6	1121	958	189
-5	956	1028	190
-2	654	615	192
-1	1846	1856	190
0	887	976	193
2	748	806	10
4	1281	1294	188
6	1602	1714	9
7	1091	1084	10
** K= 13	L= 1 **		
-8	723	723	10
-7	954	808	10
-6	816	694	9
-3	523	405	9
-2	594	475	11
0	645	692	192
1	862	931	190
4	819	914	187
5	1092	1140	190
6	737	889	190

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_o	F_c	PHI
** K= 15 L= 1 **			
-6	485	586	11
0	689	683	189
1	707	678	191
4	488	422	189
5	789	743	191
** K= 16 L= 1 **			
-1	973	1036	11
0	930	940	11
** K= 0 L= 2 **			
-16	1001	1025	11
-15	1247	1199	10
-13	741	639	8
-12	496	424	187
-11	1971	2108	9
-7	2350	2308	186
-5	5134	5253	186
-4	725	747	10
-3	4577	4424	186
-2	2737	2689	185
-1	5461	5377	186
0	314	139	204
1	3926	3761	7
2	1834	1791	185
3	1072	1105	182
4	3095	3143	186
5	4718	4910	8
6	2467	2502	11
7	709	896	7
8	632	664	191
10	3687	3864	8
13	1639	1435	190
14	668	421	9
15	638	605	10
** K= 1 L= 2 **			
-16	792	734	192
-15	959	908	11
-10	1895	2040	9
-8	1892	1970	9
-7	931	1065	187
-6	2466	2547	6
-5	2077	2110	184
-4	1109	1076	182
-3	1871	1838	7
-2	2050	2040	185
-1	467	422	359
0	7840	7761	186
1	1117	1032	7
3	685	536	1
4	2723	2710	189
5	4189	4412	187
6	2568	2806	7
8	1197	1202	8
9	2254	2332	190
11	1887	1943	9
12	1119	997	10
14	935	926	191
15	812	705	11
** K= 2 L= 2 **			
-14	781	732	9
-12	1079	1059	9
-11	1093	1304	189
-10	725	680	11
-6	1958	1926	186
-5	1647	1640	7
-4	3680	3595	187
-3	866	837	10
-2	770	769	189
-1	1310	1265	9
0	590	527	188
1	2700	2667	188
2	1177	1161	11
3	945	979	1
4	3797	3844	6
5	2637	2699	187
7	812	680	9
8	1803	1780	7
9	984	886	11
10	1744	1880	189
11	685	545	6
13	873	990	10
** K= 3 L= 2 **			
-14	683	592	10
-12	1033	1147	8
-11	952	1100	10
-9	2500	2409	8
-8	702	698	184
-7	2856	3047	7
-5	1957	1836	6
-3	1945	1896	187
-2	1331	1302	3
-1	3349	3133	187
0	1064	868	8
1	4769	4633	186
2	1129	1024	189
3	2723	2699	186
4	856	931	189
5	965	909	5
6	2004	2080	189
7	640	809	8
8	1254	1050	188
9	1886	1869	8
12	723	747	9
14	951	999	11
** K= 4 L= 2 **			
-15	681	487	191
-14	771	769	190
-12	1315	1393	189
-10	935	946	189
-9	1174	1248	7
-6	2899	2918	7
-4	3662	3585	8
-3	430	468	353
-2	2329	2318	10
-1	1085	875	11
0	1218	1121	5
1	858	866	7
2	2666	2597	186
3	2004	1959	7
4	2149	2211	188
6	2446	2483	189
7	751	685	191
8	800	746	183
9	1735	1692	189
11	1372	1360	190
12	947	765	9
** K= 5 L= 2 **			
-14	910	1044	190
-9	1382	1486	188

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
-8	1361	1337	8
-7	1783	1915	188
-6	486	377	16
-4	569	567	6
-3	1004	1016	4
-2	2066	2137	187
-1	2496	2464	9
0	463	451	1
1	2174	2084	8
2	2494	2481	187
4	2211	2280	8
5	778	824	7
6	878	799	6
7	2507	2562	189
8	1351	1362	9
11	1052	1116	189
12	893	858	191
13	597	665	10

** K= 6 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-13	1418	1398	190
-10	798	792	9
-9	729	908	189
-7	1282	1399	7
-6	640	676	188
-5	2255	2252	7
-3	3631	3526	7
-2	816	808	192
0	1193	1158	8
1	1524	1513	7
2	1904	1863	7
3	4617	4592	187
6	1882	1929	8
7	1267	1350	190
8	3028	3037	188
11	1230	1184	10
12	1327	1308	190

** K= 7 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-14	715	755	11
-13	862	814	191
-12	1135	1050	190
-11	628	487	192
-10	1166	1121	188
-8	2395	2387	189
-6	1615	1693	189
-5	565	558	185
-3	668	574	9
-2	3154	3137	8
-1	1106	1143	189
0	1267	1219	9
1	1009	1028	4
2	5449	5411	7
3	2270	2236	7
4	1256	1186	188
6	924	766	7
7	3075	3175	9
8	1433	1370	189
9	1138	1258	191
10	1238	1359	189
11	814	538	10
13	1246	1306	191

** K= 8 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-13	1223	1119	11
-10	869	965	189
-8	507	488	186
-7	973	906	189
-6	378	397	4
-5	2426	2401	188
-3	3069	3071	187
-2	1189	1163	7
0	1270	1258	189
1	1311	1347	187
2	1093	1174	189
3	4161	4193	7
6	1414	1364	189
7	1010	1056	12
8	2547	2604	9
9	780	226	10
11	965	858	190
12	1256	1164	11

** K= 9 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	1284	1149	189
-8	778	710	10
-7	857	874	191
-6	973	933	9
-4	487	484	185
-2	1261	1240	189
-1	2185	2192	8
1	660	673	11
2	1910	1876	188
4	924	952	12
7	2204	2186	189
8	679	626	10

** K= 10 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-10	998	889	10
-7	503	489	188
-6	1168	1164	190
-5	768	771	6
-4	2050	2084	188
-2	2538	2534	187
1	593	600	188
2	1388	1385	5
3	1230	1214	188
4	1420	1441	10
6	1304	1334	10
7	536	602	9
9	998	856	9
11	891	753	11

** K= 11 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-9	1416	1434	10
-7	1203	1144	10
-5	579	476	13
-4	549	572	7
-3	708	606	190
-1	1723	1868	189
0	407	441	11
1	1685	1748	188
2	932	854	188
3	1369	1416	189
4	713	604	191
6	1069	1127	190
9	1039	1009	10

** K= 12 L= 2 **

H	$/F_o/$	$/F_c/$	PHI
-11	622	681	11
-6	640	674	10
-5	692	813	189
-4	1355	1421	9
-2	911	846	8
-1	653	691	190
1	1377	1456	9
2	468	528	187

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
4	1609	1658	188
5	794	902	10
8	746	835	190
10	880	716	11
** K= 13	L= 2 **		
-10	793	738	11
-6	1021	860	11
-5	564	361	188
-3	596	513	11
0	1878	1871	190
4	976	1035	189
5	1443	1307	190
6	1153	964	11
9	883	850	192
** K= 14	L= 2 **		
-5	1076	1133	11
-3	916	924	10
-1	1394	1396	9
1	1137	1171	189
4	772	716	11
5	1365	1451	191
6	1063	1024	191
** K= 15	L= 2 **		
-2	418	381	11
0	1585	1593	10
4	812	839	11
5	993	991	12
** K= 16	L= 2 **		
-4	562	565	11
-3	502	398	192
** K= 1	L= 3 **		
-15	684	629	11
-14	666	442	11
-11	1110	1199	10
-10	1638	1691	8
-7	446	376	6
-6	750	741	7
-5	750	727	359
-4	840	827	191
-3	1162	1202	189
-2	1380	1301	188
-1	3085	2922	185
0	1759	1720	188
1	1347	1233	188
2	1330	1227	186
3	2017	2036	185
4	668	692	194
5	464	64	181
6	1151	1288	7
7	724	742	9
10	1093	1152	9
11	1202	1239	9
12	706	521	8
** K= 2	L= 3 **		
-16	1166	1039	191
-14	580	550	9
-12	920	1041	189
-11	1048	969	187
-10	1534	1557	7
-9	2365	2377	8
-8	1760	1730	6
-7	411	462	6
-6	1074	1182	9
-5	3545	3553	6
-4	2792	2683	6
-3	1691	1606	6
-2	544	526	169
-1	669	662	7
0	941	879	355
1	1456	1405	186
2	764	750	355
3	1138	1041	184
4	2884	2955	186
5	3516	3559	187
6	2119	2232	186
7	494	391	3
9	1681	1604	189
10	1117	1138	190
12	1206	1126	10
** K= 3	L= 3 **		
-15	1135	1066	191
-13	760	904	8
-11	1435	1409	189
-10	2237	2216	188
-8	1191	1263	8
-6	1265	1286	188
-5	605	661	185
-4	828	840	10
-3	1560	1519	6
-2	503	449	13
-1	1635	1584	6
0	436	227	44
2	715	697	1
3	1806	1734	6
4	2672	2662	6
6	2031	2095	187
7	534	591	191
8	1002	1167	9
9	899	919	7
10	1268	1370	189
11	1509	1576	190
13	764	780	10
** K= 4	L= 3 **		
-14	1171	1231	190
-13	578	589	190
-12	833	778	11
-11	1423	1435	7
-10	739	795	187
-9	1470	1369	188
-7	2296	2259	7
-6	1888	1890	6
-5	501	486	184
-3	1082	1061	5
-2	665	704	7
-1	299	255	175
0	679	600	5
2	1431	1486	187
3	1546	1576	188
4	334	272	1
5	916	1004	8
7	2325	2386	187
8	1064	984	190
9	1227	1237	8
10	1021	1046	10
12	1101	1104	189
14	1060	904	12
** K= 5	L= 3 **		

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_o	F_c	PHI
-13	1468	1504	189
-12	1361	1438	189
-9	1235	1189	189
-8	1807	1845	188
-7	664	558	192
-6	612	593	11
-5	928	980	8
-4	806	726	7
-3	769	802	8
-2	2083	2037	6
-1	2076	1983	7
0	2802	2752	5
1	2419	2391	8
2	1431	1392	10
3	1228	1194	3
4	1356	1408	183
5	835	900	5
6	760	855	9
7	575	446	184
8	1760	1761	189
9	1700	1756	188
12	751	709	189
13	1072	1149	191

** K= 6 L= 3 **

H	F_o	F_c	PHI
-14	520	665	11
-11	740	739	189
-8	846	752	190
-7	1399	1435	190
-6	1681	1676	189
-5	858	841	190
-4	975	1051	186
-3	599	539	191
-2	814	818	189
-1	753	822	186
0	392	346	185
1	428	348	15
2	1128	1106	9
3	1528	1504	7
4	864	865	7
5	636	615	8
6	1244	1236	9
7	1573	1607	8
8	829	891	11
12	545	540	7

** K= 7 L= 3 **

H	F_o	F_c	PHI
-6	383	331	360
-4	602	535	187
0	687	637	188
1	393	316	190
3	414	395	181
5	771	648	186

** K= 8 L= 3 **

H	F_o	F_c	PHI
-8	948	869	188
-7	1461	1454	188
-6	812	827	188
-5	578	540	188
-4	713	725	190
-3	1331	1378	187
-2	415	440	188
2	720	667	10
3	1403	1347	8
4	1103	1027	8
6	693	475	9
7	940	1068	11

** K= 9 L= 3 **

H	F_o	F_c	PHI
-13	1129	1096	11
-12	990	1071	10
-9	739	772	11
-8	1338	1318	10
-6	743	719	188
-5	584	618	187
-3	1237	1203	186
-2	1905	1970	187
-1	1632	1657	187
0	1258	1210	187
1	1573	1565	188
2	1764	1735	189
6	783	726	191
8	1175	1178	10
9	1237	1118	10

** K= 10 L= 3 **

H	F_o	F_c	PHI
-12	754	767	11
-11	661	741	10
-8	531	538	10
-7	1269	1305	11
-6	590	649	11
-3	742	705	10
2	321	377	8
4	481	449	5
2	811	848	189
3	1100	1075	189
7	1648	1606	189
8	825	787	190
10	813	636	10
11	508	456	191

** K= 11 L= 3 **

H	F_o	F_c	PHI
-11	851	680	11
-10	905	839	10
-8	754	745	189
-6	608	727	9
-5	474	493	8
-4	503	274	192
-3	580	452	189
-1	459	458	187
4	546	551	190
6	1124	1147	10

** K= 12 L= 3 **

H	F_o	F_c	PHI
-9	856	877	10
-8	836	762	8
-6	590	450	10
-5	912	922	10
-4	805	820	13
-3	609	546	10
-2	567	318	10
4	1207	1202	188
5	1132	1240	190
6	847	760	189
9	599	537	190

** K= 13 L= 3 **

H	F_o	F_c	PHI
-3	949	880	8
-2	704	705	13
-1	835	875	8
0	443	420	10
2	758	654	9
3	759	772	11

** K= 15 L= 3 **

H	F_o	F_c	PHI
0	733	751	10

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
** K= 16	L= 3 **		
0	482	195	7
2	592	351	9
** K= 0	L= 4 **		
-16	1247	1098	191
-15	1009	1088	12
-12	1486	1541	189
-11	825	763	5
-10	4285	4402	9
-9	814	629	13
-8	1040	1036	8
-7	2673	2569	187
-6	5112	5097	7
-5	4409	4359	6
-4	2644	2651	2
-3	1499	1480	185
-2	4032	3935	186
-1	5107	4911	5
0	3092	2919	186
1	2276	2264	182
2	4116	4018	187
3	2226	2219	186
4	923	948	190
5	3372	3477	187
6	1411	1410	4
7	1563	1676	188
9	1302	1220	191
11	1417	1393	9
12	451	525	11
14	502	465	191
** K= 1	L= 4 **		
-16	965	944	192
-15	1165	1192	189
-14	937	929	9
-11	3366	3384	189
-9	1210	1362	9
-8	1249	1298	9
-7	543	500	190
-6	3102	3122	188
-5	4150	4154	7
-4	2715	2592	5
-3	3832	3682	7
-2	753	656	200
-1	454	393	12
0	2401	2248	8
1	2002	1946	2
2	2825	2714	6
3	1613	1601	190
4	1587	1685	7
5	2056	2063	187
6	345	411	187
7	485	454	186
8	741	548	188
10	2173	2211	189
13	621	563	10
** K= 2	L= 4 **		
-16	472	543	12
-15	1058	1081	191
-12	1382	1390	8
-10	2206	2192	189
-9	865	958	9
-8	318	298	2
-7	3681	3763	7
-6	2317	2266	186
-5	336	420	197
-4	1049	1022	4
-3	1221	1278	3
-2	3218	3070	6
-1	5013	4844	185
0	1350	1343	6
1	2841	2771	185
2	777	743	11
4	1174	1177	186
5	1028	1022	7
6	1886	1950	188
7	948	905	8
8	783	798	189
9	875	937	12
14	785	744	12
** K= 3	L= 4 **		
-14	1223	1172	191
-12	1382	1382	190
-11	555	684	10
-10	1689	1660	188
-9	666	729	184
-8	539	444	186
-6	2850	2827	8
-5	1140	1084	185
-4	4205	4233	5
-3	1910	1807	181
-2	3610	3414	7
-1	1058	1071	13
1	2338	2321	7
2	1394	1304	187
3	2555	2503	6
4	1671	1667	188
5	602	530	9
6	1533	1640	187
7	1082	1156	189
8	852	873	186
9	1345	1424	190
11	1411	1266	189
** K= 4	L= 4 **		
-14	848	785	189
-11	1080	989	189
-10	690	630	188
-9	2825	2819	188
-8	555	552	9
-7	2272	2356	188
-6	972	997	185
-5	2642	2681	186
-4	586	451	195
-3	891	856	11
-2	2661	2640	184
-1	3969	3826	4
0	2012	1839	184
1	3575	3538	7
2	1334	1126	6
3	1676	1648	7
4	2074	2201	7
5	823	818	5
6	2277	2295	8
7	629	700	189
8	893	1079	10
11	501	419	189
12	956	694	191
** K= 5	L= 4 **		
-13	1308	1244	190
-12	1340	1293	9
-11	543	516	7
-10	1176	1175	9

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_O	F_C	PHI
-9	1689	1802	184
-8	800	894	189
-7	1535	1575	8
-5	1053	1054	5
-4	4350	4271	187
-3	578	464	16
-2	892	847	184
0	1250	1191	185
1	2315	2447	186
2	1961	1923	7
3	1790	1723	187
4	1422	1356	7
6	1126	1129	8
9	908	934	9
11	1104	931	11
12	649	620	191

** K= 6 L= 4 **

H	F_O	F_C	PHI
-14	1355	1350	10
-13	1058	868	189
-12	1446	1446	189
-10	448	513	10
-9	1200	1222	9
-8	3763	3818	188
-7	1253	1237	187
-6	706	603	189
-5	1795	1813	8
-4	783	799	185
-3	3234	3266	187
-2	1207	1216	7
-1	292	206	186
0	3478	3283	7
1	679	695	193
2	1058	1108	8
3	892	877	9
5	1359	1316	8
6	520	451	189
7	1770	1861	8
8	906	895	190
11	644	311	9
13	1018	926	192

** K= 7 L= 4 **

H	F_O	F_C	PHI
-14	898	867	10
-13	1948	2047	11
-10	537	547	188
-9	2686	2730	8
-8	1398	1378	8
-7	2445	2408	188
-6	1813	1736	187
-5	1982	1968	188
-4	2264	2255	8
-3	2743	2725	188
-2	2461	2389	188
-1	2751	2782	188
0	629	652	191
1	966	1005	6
2	2459	2446	187
3	1134	1192	7
4	1210	1226	187
5	1170	1267	9
7	807	832	12
8	1910	1875	9
12	1297	1151	12

** K= 8 L= 4 **

H	F_O	F_C	PHI
-14	872	839	192
-13	1018	846	11
-12	1321	1308	10
-9	857	816	189
-8	3124	3172	9
-7	1469	1330	7
-6	689	692	9
-5	1553	1470	188
-3	2383	2434	8
-2	578	552	189
0	2741	2697	189
2	988	938	189
3	666	671	189
4	749	735	186
5	1516	1499	189
7	1650	1719	189
8	685	695	9

** K= 9 L= 4 **

H	F_O	F_C	PHI
-13	849	811	192
-12	930	878	11
-10	734	820	10
-9	1342	1248	188
-7	1227	1260	9
-5	1122	1116	8
-4	2869	2866	187
-3	1005	1051	5
-1	773	862	8
1	1517	1588	187
2	1325	1291	6
3	1101	1107	187
4	1519	1511	8
9	777	681	10

** K= 10 L= 4 **

H	F_O	F_C	PHI
-11	708	684	9
-9	1389	1299	11
-8	441	342	190
-7	1158	1210	10
-6	595	439	4
-5	1133	1178	10
-4	659	739	10
-3	554	621	189
-1	1455	1451	189
0	906	865	9
1	1369	1323	189
2	619	462	191
3	1215	1283	189
4	871	932	191
6	979	945	190
8	845	875	189

** K= 11 L= 4 **

H	F_O	F_C	PHI
-12	856	825	192
-10	833	733	189
-6	1414	1447	9
-4	1679	1682	8
-2	1969	2032	9
-1	546	558	10
1	1416	1420	8
3	1037	1079	9
4	1138	1144	190
9	1095	1065	190

** K= 12 L= 4 **

H	F_O	F_C	PHI
-10	850	883	9
-9	693	491	192
-7	1369	1403	190
-6	675	649	11
-2	601	568	191
-1	1432	1419	10

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_3)_2CMe]$.

H	$/F_o/$	$/F_c/$	PHI
3	804	759	9
4	734	526	12
6	884	800	10
8	737	688	10
** K= 13 L= 4 **			
-6	1216	1230	190
-5	1344	1329	9
-4	548	477	10
-3	938	876	10
-2	803	830	190
0	987	907	10
3	844	792	190
4	698	672	9
5	594	603	190
** K= 14 L= 4 **			
-7	708	619	10
-6	1349	1336	190
-5	1044	1073	191
-3	404	371	9
-2	753	789	10
-1	766	903	190
0	530	643	10
2	992	1019	11
3	612	434	11
5	847	838	11
** K= 15 L= 4 **			
-6	813	692	11
-5	896	975	190
-4	621	604	189
-3	1134	1044	190
0	722	706	190
2	489	623	191
** K= 16 L= 4 **			
-1	581	608	10
** K= 1 L= 5 **			
-16	662	694	190
-12	987	1047	190
-11	975	793	189
-10	826	832	7
-9	1326	1383	8
-7	532	593	187
-6	714	678	12
-5	2569	2570	9
-4	3771	3694	5
-1	1540	1440	8
0	2066	1983	8
1	1730	1580	179
2	1342	1323	187
3	1062	1096	187
4	460	392	187
6	1161	1197	189
7	1157	1216	189
8	773	835	188
9	636	633	189
10	588	469	190
** K= 2 L= 5 **			
-15	754	770	191
-13	801	708	8
-11	2456	2501	188
-10	2817	2902	188
-9	959	917	182
-8	1484	1466	6
-7	1799	1771	186
-6	3599	3549	188
-5	1529	1413	187
-4	1335	1282	16
-3	3001	2910	7
-2	562	528	186
-1	997	959	187
0	4093	4040	4
1	5495	5414	6
2	2658	2536	8
4	418	437	9
5	1677	1648	7
6	987	921	10
10	840	557	190
11	808	742	190
** K= 3 L= 5 **			
-16	999	885	10
-14	930	872	190
-12	1611	1680	10
-11	1473	1508	7
-10	1929	1882	188
-9	2859	2895	187
-7	2660	2734	8
-5	4344	4281	188
-4	2559	2630	188
-3	1366	1354	9
-2	1572	1569	11
-1	1728	1646	188
0	3689	3596	187
1	436	321	349
2	2380	2372	8
3	1793	1832	5
4	660	651	188
5	655	717	184
6	1105	1170	7
7	954	1017	10
** K= 4 L= 5 **			
-15	822	834	11
-13	1701	1601	189
-12	798	790	189
-11	1288	1276	9
-10	1144	1174	9
-9	1931	1985	188
-8	3286	3346	188
-6	3026	3049	8
-5	1015	1046	7
-4	3000	3026	187
-3	2698	2634	187
-2	2143	2109	8
-1	3394	3298	8
0	435	403	8
1	1861	1824	188
3	2607	2566	7
4	1406	1408	8
6	1008	1014	186
** K= 5 L= 5 **			
-15	872	753	10
-14	1406	1420	10
-13	726	600	9
-11	647	617	187
-10	1651	1538	8
-9	1086	1042	11
-8	1402	1452	188
-7	2968	2915	189

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
-6	2018	1986	187
-5	1267	1226	6
-4	517	578	185
-3	3395	3277	188
-2	4277	4224	187
0	1557	1657	7
2	1209	1174	189
4	1254	1262	11
5	1974	2012	8
6	929	938	10
7	915	814	6
8	1011	997	8
** K= 6 L= 5 **			
-13	1057	1028	9
-9	1377	1338	10
-8	1971	1967	8
-7	789	749	7
-6	991	988	186
-4	1225	1254	6
-3	1067	1034	4
-2	1979	1987	186
-1	2520	2492	187
0	1818	1869	186
2	492	384	195
3	1900	1937	187
4	935	917	193
5	640	739	188
** K= 7 L= 5 **			
-8	428	271	9
-5	323	79	162
-4	383	405	4
-3	351	260	0
-1	804	784	3
5	530	430	183
** K= 8 L= 5 **			
-9	1024	1025	9
-8	1546	1610	10
-7	577	743	6
-6	816	719	187
-4	1365	1378	6
-2	923	968	191
-1	1687	1637	189
0	721	815	186
2	1157	1153	188
3	1276	1303	189
4	976	953	188
** K= 9 L= 5 **			
-14	1111	970	191
-13	659	680	191
-10	812	638	192
-9	1015	1023	190
-8	946	917	9
-7	2563	2579	9
-6	1104	1102	10
-5	696	647	188
-3	2924	2870	8
-2	2694	2766	8
0	1036	1082	189
2	1069	1081	8
4	1528	1458	189
5	1134	1139	190
7	788	837	187
8	773	669	191
9	828	785	190
** K= 10 L= 5 **			
-13	1090	1087	191
-12	878	754	191
-11	753	677	10
-9	1080	1000	191
-8	1975	1986	190
-6	1714	1725	9
-5	691	778	7
-4	1491	1565	190
-3	1328	1392	187
-2	1681	1732	7
-1	2313	2284	9
0	352	342	8
1	907	1028	188
3	1357	1395	10
4	1172	1194	9
** K= 11 L= 5 **			
-12	1023	1011	191
-11	668	697	190
-10	786	796	9
-9	1110	1094	10
-8	564	507	189
-7	1616	1595	189
-5	1873	1845	9
-4	1054	1100	10
-3	1062	1072	189
-2	1072	1173	189
-1	556	471	12
0	1551	1555	10
2	1253	1213	189
3	803	816	189
** K= 12 L= 5 **			
-11	1060	1072	191
-10	1000	1010	190
-8	643	634	7
-7	933	810	189
-6	1426	1454	190
-5	518	525	191
-4	927	903	11
-3	1033	1100	10
0	611	658	10
1	1583	1611	10
2	1274	1255	8
** K= 13 L= 5 **			
-9	486	451	190
-6	460	362	195
-5	1042	1113	190
-4	902	878	191
0	930	915	189
3	686	613	10
** K= 15 L= 5 **			
-5	590	681	192
-1	730	705	188
** K= 16 L= 5 **			
-3	521	598	191
0	723	720	190
** K= 0 L= 6 **			
-15	1108	1022	189

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
-14	663	627	9
-13	1064	1042	188
-11	3291	3364	190
-9	675	792	11
-8	804	861	182
-7	2246	2270	188
-6	4115	3987	188
-5	4866	4852	8
-4	6109	5928	4
-3	2576	2476	8
-2	4504	4413	186
-1	1547	1615	5
0	7182	7033	7
1	3242	3012	8
2	722	722	11
3	3388	3316	189
4	2327	2350	8
5	1162	1187	10
6	892	865	5
7	2031	2041	188
8	1604	1751	190
10	1097	1073	188
12	1325	1223	190

** K= 1 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-16	972	883	11
-15	614	718	191
-12	1159	1082	8
-10	3214	3228	189
-9	1058	982	6
-7	1779	1870	8
-6	5195	5143	187
-5	4377	4335	186
-4	2027	1923	4
-3	2347	2312	5
-2	1258	1242	9
-1	6206	6071	187
0	689	780	177
1	1228	1205	11
2	4539	4509	7
4	2325	2360	187
5	1885	1879	8
6	866	1058	10
7	2344	2409	9
8	1284	1214	189
13	711	657	191

** K= 2 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-14	1255	1087	189
-12	719	679	190
-11	1505	1574	10
-10	1301	1352	188
-9	1796	1789	186
-8	373	324	188
-7	547	359	15
-6	2651	2662	9
-5	3638	3594	187
-4	684	752	182
-3	475	405	195
-2	4119	4043	7
-1	1204	1202	6
0	2421	2455	188
1	584	484	179
3	3866	3818	7
4	1464	1457	189
5	607	551	189
6	958	882	188
7	688	758	6
8	634	655	11
9	1173	1190	189
11	586	693	189

** K= 3 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-15	1084	1092	10
-11	531	599	187
-10	900	900	8
-9	2529	2499	188
-7	3207	3238	187
-6	401	437	2
-5	1188	1230	188
-4	1974	1921	189
-2	2767	2697	187
-1	2771	2657	7
0	1488	1443	186
1	2186	2156	5
2	428	362	186
3	1540	1482	7
4	3078	3076	7
6	2091	2133	7
7	568	596	187
8	1313	1427	9

** K= 4 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-14	947	1040	9
-12	1548	1679	10
-10	1724	1780	9
-7	2251	2283	6
-6	1374	1366	185
-5	759	693	9
-4	3704	3586	188
-3	393	350	5
-2	2024	2058	186
-1	756	737	195
0	1447	1445	190
1	3462	3451	187
2	818	792	8
3	1695	1655	189
4	440	366	12
5	1497	1429	188
6	842	809	6
7	824	811	11
9	1177	1129	9
11	1291	1233	10

** K= 5 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-14	882	810	10
-13	511	488	192
-12	857	793	188
-11	753	775	10
-10	657	462	10
-9	1537	1569	10
-8	2602	2589	188
-7	1224	1179	5
-6	1053	1042	8
-5	2875	2800	7
-3	3681	3714	186
-2	1830	1845	7
-1	735	689	7
0	2631	2624	8
1	3020	2916	188
2	1017	1099	188
3	429	393	11
4	630	600	7
6	2418	2517	189
10	716	657	188

** K= 6 L= 6 **

H	$/F_O/$	$/F_C/$	PHI
-13	1628	1552	10
-12	931	904	189

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
-9	2847	2815	8
-8	1349	1364	8
-7	2433	2397	189
-4	4196	4157	7
-3	2625	2641	188
-2	3204	3212	188
-1	1986	1999	187
0	1271	1295	7
1	2181	2152	8
2	3862	3829	187
3	1112	1144	188
4	1215	1135	186
5	2522	2467	9
7	784	784	190
9	471	396	9
10	1254	1265	10
** K= 7 L= 6 **			
-14	1106	981	191
-12	816	814	10
-11	882	662	189
-8	4255	4372	8
-7	1635	1628	7
-5	1079	1098	189
-4	1821	1754	9
-3	5018	4973	8
-2	643	638	187
-1	1291	1351	188
0	2842	2869	189
1	2639	2636	8
2	543	681	9
3	2211	2191	188
4	2987	3027	188
5	1808	1811	189
6	1073	1094	11
7	1116	1003	190
9	1128	1179	190
** K= 8 L= 6 **			
-13	1212	1279	191
-12	580	689	10
-9	2322	2275	189
-8	949	917	188
-7	2423	2383	8
-6	580	524	7
-4	3042	3104	188
-3	2396	2431	8
-2	2839	2856	8
-1	1937	1892	8
0	622	601	190
1	2027	2020	187
2	3520	3493	8
3	1123	1148	9
4	918	993	9
5	1765	1741	190
7	743	741	9
10	1181	1174	191
** K= 9 L= 6 **			
-12	510	619	188
-11	765	780	8
-9	691	537	12
-8	1788	1750	190
-6	791	700	7
-5	1216	1218	10
-3	2272	2246	188
-2	887	826	11
-1	751	610	4
0	1579	1653	9
1	2000	1953	189
2	745	767	190
5	680	780	9
6	1334	1327	190
9	765	744	10
** K= 10 L= 6 **			
-12	1252	1205	191
-10	841	722	190
-8	735	599	187
-7	1273	1314	188
-6	567	696	10
-5	370	392	190
-4	2000	1978	8
-2	881	942	8
-1	616	581	10
0	672	667	11
1	2054	2051	8
3	685	778	10
4	820	776	189
5	713	578	11
7	609	588	188
9	1017	890	190
** K= 11 L= 6 **			
-9	853	829	191
-7	1725	1817	190
-5	456	424	190
-4	943	816	192
-3	657	567	189
-2	888	928	192
-1	947	973	12
0	764	725	187
3	1204	1153	8
4	1267	1280	10
6	865	917	10
8	951	1036	11
** K= 12 L= 6 **			
-11	955	857	190
-9	574	613	11
-8	514	399	9
-6	1562	1570	189
-5	975	1063	10
-2	1665	1712	190
0	888	860	10
3	1650	1715	189
4	743	683	10
** K= 13 L= 6 **			
-10	1016	1119	191
-7	829	834	11
-6	1440	1498	190
-5	1202	1201	191
-4	604	478	8
-3	642	634	11
-2	562	548	10
-1	1890	1887	191
1	731	636	11
2	1342	1408	10
4	819	758	192
5	726	723	10
** K= 14 L= 6 **			
-7	674	716	11
-6	1160	1170	11
-5	1420	1430	190
-4	998	1009	190

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
-3	811	773	189
-2	1014	1028	10
0	1874	1909	190
1	902	862	191
3	990	1096	11
4	838	696	191
** K= 15	L= 6	**	
-6	1317	1302	10
-5	1096	1048	10
-2	532	383	189
-1	1622	1590	10
2	917	989	191
** K= 1	L= 7	**	
-12	645	445	186
-11	1046	1016	188
-10	874	849	191
-9	738	808	186
-7	1561	1506	188
-6	2252	2222	188
-5	1368	1490	185
-4	703	692	9
-3	661	806	5
-2	1809	1815	185
-1	426	417	193
0	2616	2720	5
1	3008	2990	7
2	1053	1073	9
4	623	618	8
5	1833	1820	8
6	1514	1556	8
8	549	482	191
** K= 2	L= 7	**	
-14	668	563	9
-13	715	707	10
-12	904	1026	11
-11	1183	1141	8
-9	633	504	188
-8	648	728	8
-7	1288	1367	6
-6	1101	1135	189
-5	3855	3837	187
-4	2263	2092	188
-3	940	866	5
-1	3896	3763	187
0	4012	3965	188
2	2496	2457	7
4	2200	2194	187
5	871	852	186
6	2033	2030	9
7	2109	2151	10
11	1298	1115	10
** K= 3	L= 7	**	
-11	1190	1154	8
-10	922	866	11
-8	1129	1175	185
-7	2011	1989	6
-6	3213	3207	7
-4	2328	2323	189
-3	995	1032	190
-2	2424	2485	8
-1	2457	2336	8
0	2190	2165	188
1	4418	4382	187
2	917	922	186
3	2908	2940	7
4	537	348	10
5	3010	3002	187
6	2136	2281	188
7	487	550	10
8	1522	1446	9
10	808	901	190
12	862	697	11
** K= 4	L= 7	**	
-10	1275	1271	8
-9	884	895	9
-8	1024	1011	188
-7	1749	1705	189
-5	2069	2063	8
-3	3805	3745	187
-2	2722	2776	188
-1	1440	1433	9
0	2984	3017	7
1	1633	1715	185
2	3156	3146	189
3	578	469	189
4	2749	2821	8
5	2220	2198	7
6	1296	1194	187
7	1289	1253	190
8	860	816	9
9	1581	1455	10
** K= 5	L= 7	**	
-10	632	660	8
-9	1661	1694	10
-8	2112	2162	8
-7	453	436	10
-6	596	575	187
-5	1329	1348	7
-4	2790	2802	8
-3	1492	1424	6
-2	2569	2498	187
-1	2406	2380	188
1	878	787	12
2	1237	1269	188
3	3664	3670	188
4	2049	2029	188
5	575	482	7
6	759	638	9
7	1060	968	189
8	1571	1509	188
10	804	806	11
** K= 6	L= 7	**	
-12	619	619	187
-10	937	881	188
-8	684	637	9
-7	854	895	9
-6	375	411	6
-4	1340	1335	7
-3	2394	2447	8
-2	2045	1982	8
-1	436	306	6
1	1350	1284	6
2	1589	1601	9
4	1253	1155	188
5	978	1033	189
8	803	799	189
9	866	914	190
10	1044	845	189
** K= 7	L= 7	**	

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_O	F_C	PHI
-6	448	325	185
-5	484	466	187
-1	486	381	183
1	280	294	6
** K= 8 L= 7 **			
-13	617	685	190
-9	769	766	189
-7	1307	1278	7
-4	363	369	7
-3	1825	1855	8
-2	1875	1827	8
-1	551	458	5
0	788	785	186
1	825	912	7
2	1076	1029	10
4	1171	1147	189
5	888	819	190
9	997	911	191
** K= 9 L= 7 **			
-9	1086	1138	190
-8	1721	1752	190
-7	642	633	189
-5	579	572	187
-4	2161	2155	188
-3	1249	1256	187
-2	1592	1520	8
-1	2214	2274	8
1	1005	978	188
2	1354	1413	7
3	2215	2201	10
4	1377	1487	10
7	834	837	10
8	911	953	10
10	736	759	191
** K= 10 L= 7 **			
-10	752	620	9
-9	483	467	13
-8	816	694	189
-7	1468	1518	188
-5	1148	1069	9
-3	2281	2359	189
-2	1935	1944	188
-1	553	577	9
0	1377	1375	10
1	1118	1208	187
2	1892	1961	190
4	1472	1463	10
5	1061	1107	9
6	717	792	190
7	718	666	189
9	1133	1166	11
** K= 11 L= 7 **			
-8	607	549	9
-7	830	779	189
-6	1305	1302	190
-4	1089	1160	11
-3	662	752	7
-2	1161	1209	190
-1	1086	1067	191
0	1023	1126	8
1	1830	1892	10
3	1281	1229	189
4	654	537	191
5	1313	1254	10
6	973	930	9
8	954	944	190
** K= 12 L= 7 **			
-6	529	535	193
-5	1413	1393	190
-4	574	555	193
-3	389	302	12
-1	1247	1230	191
0	1631	1732	189
2	1043	975	9
4	737	750	189
6	1057	919	10
7	1045	961	11
** K= 13 L= 7 **			
-7	822	828	11
-6	577	603	12
0	732	814	188
1	994	1000	190
2	459	445	191
5	759	686	192
6	573	617	190
** K= 14 L= 7 **			
-2	464	449	8
** K= 15 L= 7 **			
-6	784	729	10
** K= 0 L= 8 **			
-16	1209	1244	11
-14	1307	1269	8
-11	1010	1022	10
-10	1162	1141	188
-9	1117	1143	7
-8	2108	2129	188
-7	650	714	3
-6	4214	4269	186
-5	3133	3087	186
-3	1980	2130	184
-1	6552	6489	187
0	444	437	11
1	1335	1346	9
2	2360	2254	8
3	679	653	192
4	1938	1976	189
5	2904	2890	8
6	2336	2433	8
7	2024	2056	9
8	1351	1359	190
10	1189	1272	10
11	991	926	11
** K= 1 L= 8 **			
-13	1231	1353	10
-12	954	875	189
-11	1190	1195	11
-10	453	472	4
-9	634	627	12
-8	1276	1283	8
-7	711	666	3
-6	1988	2007	9
-5	2822	2867	187
-3	797	763	188
-2	2601	2570	6

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
-1	873	943	189
0	4386	4388	188
1	842	782	187
3	2089	2082	9
4	2985	2980	188
5	1702	1735	189
7	1655	1624	9
8	1137	1092	10
9	1454	1410	190
11	660	319	11
12	1267	1288	10
** K=	2	L=	8 **
-14	801	427	188
-13	802	759	8
-11	721	644	190
-10	989	971	9
-9	1431	1462	189
-8	1288	1369	7
-7	1408	1360	189
-6	1455	1380	6
-4	1840	1784	188
-2	1859	1871	186
-1	3734	3741	7
0	820	840	193
1	487	484	190
2	1157	1106	189
3	1169	1149	8
4	2628	2623	7
5	1461	1435	187
7	836	796	190
8	1502	1560	10
11	473	367	191
** K=	3	L=	8 **
-14	829	727	10
-13	440	517	188
-12	1565	1711	9
-10	1676	1719	9
-9	1083	1111	7
-8	561	518	8
-7	1686	1614	9
-6	681	785	188
-5	2283	2338	8
-4	1717	1740	188
-2	1729	1672	189
-1	1059	1082	188
1	2446	2489	188
2	493	402	188
3	3434	3480	187
4	561	446	10
5	653	549	190
9	979	1056	11
11	709	724	11
** K=	4	L=	8 **
-15	1166	1096	191
-13	751	605	189
-12	827	737	188
-10	850	776	190
-9	1812	1870	8
-8	1049	1017	187
-7	2056	2008	8
-6	697	731	9
-5	2094	2109	7
-4	1949	1902	8
-3	515	424	186
-2	2607	2578	8
-1	539	450	183
0	2520	2522	7
1	1315	1336	188
4	1280	1301	186
6	2461	2530	188
8	730	728	191
11	518	474	191
** K=	5	L=	8 **
-14	1178	976	190
-13	491	532	11
-12	921	884	189
-11	655	555	9
-10	1126	1066	189
-7	1615	1649	188
-6	849	878	7
-5	1509	1539	186
-4	2702	2730	7
-3	1454	1440	187
-1	550	499	6
0	1147	1173	9
1	2358	2372	8
2	2149	2137	188
5	2050	2115	9
7	1380	1349	188
10	733	796	11
11	1021	810	191
** K=	6	L=	8 **
-15	523	505	13
-14	947	726	191
-10	993	863	8
-9	1051	1018	190
-8	1960	1962	8
-7	587	520	184
-6	630	753	6
-4	1025	965	9
-3	3547	3610	7
-2	1640	1657	186
0	1701	1690	187
1	2764	2763	8
2	657	669	9
3	1801	1881	189
4	1718	1738	188
5	803	699	190
6	1990	1960	9
7	728	799	191
8	996	1041	190
9	918	988	191
10	713	662	9
11	774	723	11
** K=	7	L=	8 **
-13	967	975	191
-11	1602	1605	189
-9	1325	1308	189
-8	1182	1152	190
-7	508	484	10
-6	1483	1475	188
-5	524	495	5
-4	2699	2638	187
-3	2759	2753	7
-2	2233	2148	8
-1	835	881	8
1	612	657	186
2	4296	4275	8
3	1515	1594	8
5	1705	1682	190
7	1589	1603	10
9	938	735	192

Table I.3. Obs and Calcd Structure Factors for $Os_3(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_O	F_C	PHI
10	1585	1413	191
** K= 8	L= 8 **		
-11	508	418	9
-10	766	917	189
-8	1446	1461	190
-6	518	471	192
-5	580	515	187
-4	1324	1327	188
-3	2648	2651	189
-2	958	1012	8
0	771	739	10
1	2403	2498	188
2	641	619	188
3	1399	1407	10
4	1357	1370	9
5	543	605	11
6	1519	1417	191
8	942	893	12
9	1091	1072	11
** K= 9	L= 8 **		
-12	485	458	191
-11	814	834	9
-7	890	948	190
-6	858	976	9
-5	584	643	188
-4	1515	1515	8
-3	1407	1402	188
-2	513	454	186
-1	490	507	8
0	706	577	10
1	1320	1283	8
2	1765	1701	189
5	1067	1052	11
7	947	1018	190
** K= 10	L= 8 **		
-13	467	340	13
-10	862	810	10
-9	683	572	191
-7	1270	1303	191
-6	614	622	187
-5	494	552	190
-4	811	664	191
-2	1437	1453	192
0	815	793	190
1	823	796	10
4	573	578	11
6	1229	1278	10
8	541	562	10
** K= 11	L= 8 **		
-12	1100	1086	10
-10	798	799	9
-8	580	624	10
-7	845	860	10
-6	857	864	189
-5	913	913	10
-2	1263	1223	189
-1	836	860	191
1	1064	1178	189
3	1538	1641	190
** K= 12	L= 8 **		
-11	665	526	10
-9	620	498	10
-7	905	979	11
-4	720	776	9
-2	858	866	11
-1	1134	1132	191
2	881	806	9
4	1093	1088	190
5	692	464	10
** K= 13	L= 8 **		
-6	756	963	11
-5	1039	899	190
-2	909	872	10
0	1499	1521	190
3	1120	1108	10
4	875	883	191
5	906	617	191
** K= 14	L= 8 **		
-8	610	545	10
-6	999	968	11
-5	744	837	10
-1	1653	1671	10
2	786	708	192
** K= 15	L= 8 **		
-5	724	685	11
0	1056	1101	11
** K= 1	L= 9 **		
-15	887	697	10
-14	882	783	9
-11	978	973	8
-10	1192	1158	10
-9	963	843	8
-8	538	475	1
-5	562	541	186
-4	1185	1230	186
-3	1039	1022	187
-2	1515	1479	186
-1	1799	1789	187
0	1146	1161	191
1	528	589	190
3	759	776	186
4	992	1032	188
6	764	684	11
7	734	813	10
** K= 2	L= 9 **		
-16	1098	1037	191
-15	830	863	189
-12	403	375	189
-10	598	555	10
-9	2084	2145	7
-8	1803	1737	7
-7	880	855	9
-6	1303	1369	7
-5	1369	1313	10
-4	1468	1458	7
-3	823	781	8
-2	1451	1415	6
-1	540	530	8
0	1334	1366	186
1	1881	1910	187
2	956	1002	185
4	1102	1171	188
5	2477	2465	188
6	1608	1613	189

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_O	F_C	PHI
8	724	634	7
9	700	531	192
** K= 3 L= 9 **			
-15	1001	975	191
-14	1059	1016	189
-12	691	650	9
-11	903	935	187
-10	1604	1562	189
-9	612	695	187
-8	532	568	9
-7	576	665	5
-6	543	454	185
-4	514	518	8
-3	427	234	12
-2	871	881	4
-1	1660	1649	8
0	1668	1683	7
2	872	880	189
3	741	807	6
4	1883	1829	8
5	580	639	7
6	1432	1430	189
7	1194	1284	189
9	861	766	10
11	1097	1047	191
** K= 4 L= 9 **			
-14	1215	1124	188
-13	875	912	190
-11	1046	1084	10
-10	545	457	4
-9	1043	1005	186
-7	799	802	11
-6	920	953	8
-5	558	543	7
-3	753	735	4
-1	406	388	188
0	800	771	7
1	783	746	8
2	1042	1049	187
3	2282	2261	188
4	564	531	188
5	1016	1002	9
7	1227	1197	190
8	1236	1297	189
9	461	420	9
10	1105	906	11
** K= 5 L= 9 **			
-13	1100	1020	190
-12	1422	1531	190
-11	502	461	193
-8	1079	1074	189
-7	592	615	189
-5	689	659	10
-4	1334	1388	6
-3	1388	1403	8
-2	1205	1229	9
-1	1000	903	6
1	1869	1836	8
2	1755	1733	9
4	612	739	191
6	704	710	8
8	1162	1150	189
9	1310	1201	190
10	548	504	189
** K= 6 L= 9 **			
-14	772	524	10
-11	725	728	190
-10	686	613	188
-8	748	716	187
-7	1182	1128	189
-6	1320	1393	188
-5	1466	1440	187
-4	480	485	193
-3	620	609	187
1	508	380	6
2	788	831	9
3	1440	1478	8
7	944	827	11
8	906	869	9
** K= 7 L= 9 **			
-4	611	532	187
0	423	459	190
** K= 8 L= 9 **			
-8	879	750	188
-7	849	909	190
-6	566	701	189
-4	932	930	187
-3	611	750	188
-1	547	490	8
2	551	700	8
3	917	939	10
4	906	902	10
** K= 9 L= 9 **			
-13	858	874	11
-12	944	914	12
-11	504	496	9
-9	587	501	10
-8	915	960	8
-4	381	409	188
-3	1197	1164	188
-2	1371	1352	188
-1	667	818	189
1	1168	1164	189
2	1127	1196	191
4	646	618	10
6	512	556	190
** K= 10 L= 9 **			
-12	441	428	11
-11	895	778	11
-7	781	830	9
-6	683	733	9
-3	333	438	8
0	688	462	7
3	1093	1047	190
4	596	566	191
5	707	590	10
7	883	847	191
8	870	909	191
** K= 11 L= 9 **			
-10	701	701	11
-1	527	389	192
0	648	673	190
4	893	840	189
6	962	917	10
7	789	558	11

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	/F_O/	/F_C/	PHI
** K= 12	L= 9 **		
-9	709	781	10
-8	675	621	11
0	493	507	190
1	746	595	191
5	1002	1095	190
** K= 13	L= 9 **		
-3	443	339	11
-2	792	868	9
-1	720	604	11
** K= 0	L= 10 **		
-16	1184	1181	190
-15	915	875	9
-14	461	465	15
-13	1214	1032	10
-12	2045	2025	190
-11	605	587	12
-10	2321	2316	8
-9	2650	2642	9
-8	1187	1174	7
-7	1882	1937	188
-6	2451	2507	9
-5	1777	1786	5
-4	2994	2931	7
-3	2207	2198	187
-2	454	446	194
-1	410	507	183
0	1623	1680	187
2	2218	2213	189
4	2331	2275	189
5	816	750	192
9	1023	1155	190
** K= 1	L= 10 **		
-16	1300	1348	191
-15	773	696	191
-14	715	599	189
-13	1235	1102	9
-11	2593	2549	190
-8	3661	3729	7
-7	1177	1198	188
-6	579	560	189
-5	570	572	7
-4	1291	1211	9
-3	3515	3545	7
-2	1155	1160	188
-1	1872	1835	8
0	415	373	191
1	1267	1245	9
4	842	813	8
5	1467	1478	189
7	947	961	190
8	614	664	10
10	1033	851	191
** K= 2	L= 10 **		
-16	646	555	10
-15	905	791	190
-13	838	891	189
-12	1873	1973	10
-10	582	589	184
-9	704	620	191
-8	398	279	7
-7	2699	2762	8
-6	948	907	190
-5	366	258	21
-4	1516	1522	190
-3	1282	1309	10
-1	615	599	187
1	1362	1391	189
2	666	707	11
3	1484	1462	188
4	890	840	9
5	506	429	185
9	1040	965	10
** K= 3	L= 10 **		
-15	767	805	190
-13	1311	1235	188
-12	952	911	190
-11	481	446	10
-10	1203	1149	189
-9	1066	1073	7
-8	1530	1552	189
-7	1535	1571	8
-6	587	615	9
-5	786	825	11
-4	1636	1662	8
-3	328	284	186
-2	2762	2697	8
0	2374	2405	7
4	791	829	190
5	552	542	8
6	1176	1221	190
8	1027	1065	190
** K= 4	L= 10 **		
-14	854	886	191
-13	595	494	11
-12	1010	1079	188
-11	372	384	7
-10	1551	1690	189
-9	1358	1397	188
-7	1701	1736	188
-5	2163	2183	190
-4	641	610	12
-3	876	909	186
-2	769	776	7
-1	806	859	6
1	2320	2319	8
3	1800	1795	9
5	1433	1407	8
** K= 5	L= 10 **		
-15	1342	1195	11
-13	670	648	189
-11	577	501	8
-10	1618	1664	9
-9	1945	1990	188
-7	690	651	190
-6	1204	1207	9
-4	2009	2015	188
-2	1116	1206	189
-1	1121	1121	9
0	1642	1659	188
1	424	474	6
6	1396	1280	9
** K= 6	L= 10 **		
-14	1586	1544	11
-13	641	679	191
-12	684	607	190
-11	1430	1454	190

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_o	F_c	PHI
-10	1313	1298	8
-9	608	562	10
-8	1811	1824	188
-7	1175	1198	188
-6	1784	1851	188
-5	2221	2229	8
-4	1205	1146	187
-2	696	695	188
-1	449	489	8
0	1547	1487	8
1	685	627	187
2	1755	1760	8
4	881	890	9
7	908	830	10
** K= 7 L= 10 **			
-15	656	751	191
-14	1015	955	12
-13	1732	1706	10
-12	560	557	11
-10	1370	1369	190
-9	2085	2088	9
-6	2393	2481	189
-5	1406	1307	189
-4	476	452	10
-3	1414	1388	189
-2	890	718	186
-1	2418	2473	189
1	1014	976	189
5	780	728	10
6	683	636	190
7	1184	1121	10
9	673	739	11
** K= 8 L= 10 **			
-14	1113	1051	192
-13	810	856	10
-12	682	697	10
-11	1552	1598	9
-10	869	940	188
-8	1465	1482	10
-7	1204	1157	9
-6	1609	1649	9
-5	1776	1819	188
-4	907	869	7
-2	654	637	10
0	1448	1439	188
2	1332	1361	190
4	932	761	190
6	460	501	191
7	859	878	190
** K= 9 L= 10 **			
-13	452	516	190
-10	1078	1167	11
-9	1137	1120	190
-7	483	489	189
-6	846	939	10
-4	1050	945	190
-2	574	637	193
-1	982	1025	11
0	458	444	191
1	432	476	8
6	724	773	11
7	567	514	189
** K= 10 L= 10 **			
-13	564	485	191
-10	1044	992	9
-9	703	598	12
-7	847	960	9
-6	521	437	189
-5	1447	1577	9
-3	654	600	9
-2	748	680	189
-1	796	807	188
1	915	935	189
3	1241	1318	190
** K= 11 L= 10 **			
-12	747	634	190
-10	701	609	190
-7	728	760	12
-4	845	732	10
-2	1569	1646	10
0	653	758	10
4	629	639	191
6	634	545	191
** K= 12 L= 10 **			
-7	1223	1105	191
-6	658	617	9
-5	389	344	188
-3	856	869	190
-1	707	598	8
2	466	398	188
3	897	927	10
** K= 13 L= 10 **			
-8	1046	1013	11
-4	435	410	12
-3	839	769	11
-1	552	600	10
** K= 14 L= 10 **			
-6	823	784	191
-4	853	773	190
-3	564	580	11
** K= 1 L= 11 **			
-16	862	721	191
-12	672	754	190
-11	914	828	190
-9	1297	1273	10
-8	916	880	8
-5	1978	1955	8
-4	2152	2250	8
-2	323	348	184
-1	753	772	4
0	1000	1070	7
2	452	611	189
3	750	638	190
** K= 2 L= 11 **			
-15	975	1001	190
-14	795	814	189
-12	679	612	5
-11	1566	1572	190
-10	2446	2514	190
-9	1078	1061	188
-8	837	916	10
-6	2295	2398	188
-5	1449	1434	190
-4	1468	1439	7

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
-3	2426	2404	9
-2	762	768	8
-1	571	551	185
0	935	926	7
1	2385	2349	8
2	1812	1791	8
3	635	578	8
** K= 3	L= 11 **		
-16	1016	1040	11
-14	959	1041	189
-13	701	678	190
-12	1145	1114	11
-11	1819	1704	9
-9	2623	2645	188
-7	1848	1826	9
-6	637	634	11
-5	2083	2076	189
-4	2788	2795	188
-3	485	519	2
-2	1411	1429	9
0	1737	1765	188
1	1153	1108	186
2	1145	1142	7
3	858	746	10
** K= 4	L= 11 **		
-15	871	981	10
-13	1246	1197	190
-12	1501	1534	189
-11	770	799	7
-10	1416	1446	10
-9	588	546	188
-8	2474	2492	189
-7	1355	1344	188
-6	1907	1933	8
-5	1654	1724	9
-4	1069	1055	188
-3	1804	1848	189
-1	2265	2322	8
0	1100	1150	8
1	607	736	188
3	506	558	10
4	803	755	8
** K= 5	L= 11 **		
-14	1371	1402	10
-13	890	928	10
-11	944	997	190
-9	1345	1388	9
-8	617	583	185
-7	2553	2641	189
-6	2268	2307	188
-4	928	804	5
-3	1761	1772	187
-2	2040	2110	189
-1	936	991	188
0	380	392	17
1	944	994	7
2	615	405	185
4	637	578	9
5	1137	1017	9
6	743	788	8
7	703	547	11
** K= 6	L= 11 **		
-13	683	725	12
-12	888	990	10
-9	666	766	10
-8	1444	1493	9
-7	1063	1062	7
-6	848	816	185
-1	1270	1366	189
0	1228	1258	190
2	492	470	188
4	569	661	190
5	433	372	193
** K= 8	L= 11 **		
-13	625	602	11
-12	637	733	11
-9	614	568	9
-8	1379	1381	9
-7	579	613	11
-6	627	429	190
-5	593	533	188
-2	598	666	189
-1	1236	1263	189
0	722	623	187
3	800	697	190
** K= 9	L= 11 **		
-13	792	812	191
-11	881	816	10
-9	1061	959	190
-7	1897	1929	9
-6	1711	1782	8
-3	1141	1081	10
-2	1845	1847	9
-1	407	435	9
0	589	643	189
2	618	274	7
4	742	750	191
5	589	596	190
6	594	459	189
** K= 10	L= 11 **		
-12	1034	1025	191
-10	850	775	12
-8	1510	1577	190
-7	822	869	189
-6	1328	1309	8
-5	930	997	10
-4	530	635	190
-3	1180	1066	189
-1	1419	1476	10
** K= 11	L= 11 **		
-11	1057	942	191
-9	1085	1172	11
-7	973	995	191
-6	612	595	191
-5	1065	1069	10
-4	1120	1150	10
-2	1051	1052	190
0	662	726	10
3	704	603	191
** K= 12	L= 11 **		
-10	1114	1121	191
-6	808	914	191
-3	880	905	11
-2	713	593	9
0	426	403	8
1	835	717	10
2	919	864	10

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
** K= 13	L= 11 **		
-4	771	790	191
** K= 0	L= 12 **		
-16	1064	1186	191
-15	514	385	193
-13	613	589	8
-12	648	654	188
-11	3096	3103	190
-8	2842	2909	7
-7	2767	2822	187
-6	2565	2573	187
-5	1303	1315	8
-4	3538	3539	7
-3	3817	3851	7
-2	2918	2860	187
-1	834	806	6
0	1224	1261	11
1	3198	3274	9
2	707	677	6
3	1845	1798	188
4	411	437	7
6	1090	828	11
7	1180	1094	191
8	588	630	189
9	574	492	189
** K= 1	L= 12 **		
-16	798	739	11
-15	707	737	189
-12	1952	1943	10
-11	686	617	191
-10	1899	1988	188
-9	1454	1431	189
-8	563	465	6
-7	2276	2289	9
-6	3110	3101	188
-5	2477	2545	187
-4	2195	2204	188
-3	2499	2550	9
-2	833	830	10
-1	1914	1932	189
0	1128	1070	187
2	3215	3200	9
6	871	900	10
7	1031	920	11
** K= 2	L= 12 **		
-16	844	738	11
-13	953	879	189
-11	1463	1581	10
-10	926	841	189
-9	345	179	188
-8	2408	2439	188
-7	2041	2057	7
-6	1232	1184	10
-4	526	466	196
-3	1854	1919	187
-2	3230	3267	8
1	1399	1546	189
3	1485	1490	8
6	1004	904	191
** K= 3	L= 12 **		
-15	870	899	10
-14	671	229	192
-13	683	744	10
-12	1190	1107	190
-11	643	388	10
-9	778	624	189
-8	673	740	189
-7	2244	2316	189
-6	602	646	6
-5	1719	1807	188
-4	362	352	7
-3	1827	1900	188
-2	663	529	187
-1	638	600	10
1	1346	1436	8
2	707	669	189
3	1347	1315	10
5	1051	1009	10
8	624	572	10
** K= 4	L= 12 **		
-15	987	973	11
-12	1060	896	10
-11	608	474	8
-10	1708	1764	10
-9	1022	1013	188
-8	909	927	8
-6	749	797	7
-4	2217	2227	188
-2	1722	1818	188
-1	525	566	6
0	1990	2045	189
1	915	946	187
3	369	343	190
5	704	817	190
6	584	648	11
** K= 5	L= 12 **		
-14	1046	1089	10
-13	726	798	190
-11	630	593	191
-10	1473	1405	10
-9	966	915	11
-8	1120	1172	189
-5	3119	3177	8
-4	518	443	190
-3	785	747	189
-2	819	814	188
-1	454	567	12
0	1609	1612	9
1	1408	1409	189
3	1006	988	188
4	961	921	10
6	724	762	188
** K= 6	L= 12 **		
-15	733	706	191
-14	976	790	10
-13	1296	1284	10
-10	1038	1017	190
-9	2480	2472	10
-8	700	739	8
-7	620	560	186
-6	2308	2348	189
-5	364	315	187
-4	2347	2317	9
-3	490	515	193
-2	1498	1487	188
-1	2733	2782	189
0	1127	1179	9
1	596	679	10
3	971	977	190

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
4	748	760	189
5	1482	1448	9
** K= 7 L= 12 **			
-14	1396	1279	191
-13	895	813	10
-12	604	526	10
-11	1088	1060	9
-10	1267	1311	189
-8	2460	2508	10
-7	1846	1846	9
-6	1119	1136	9
-5	2422	2353	189
-4	1363	1342	9
-3	2145	2186	9
-2	1415	1533	9
-1	1122	1147	189
0	2438	2468	189
1	844	859	9
4	1852	1882	191
5	840	803	190
** K= 8 L= 12 **			
-14	943	774	192
-13	851	1059	191
-10	648	663	10
-9	2255	2251	190
-8	595	585	188
-7	444	451	8
-6	1855	1863	10
-4	1684	1750	190
-3	493	526	13
-2	1404	1361	8
-1	2424	2475	10
0	702	807	189
3	958	985	10
4	767	823	10
5	766	890	191
** K= 9 L= 12 **			
-13	883	891	190
-11	660	495	188
-10	1106	1096	9
-9	450	295	14
-8	1043	1038	190
-6	592	501	186
-5	2229	2247	8
-4	589	572	189
-3	589	572	190
-2	527	650	190
0	1209	1246	9
1	923	930	189
3	840	773	190
4	709	694	11
** K= 10 L= 12 **			
-12	1021	832	190
-10	660	734	192
-8	672	662	191
-4	1139	1122	10
-2	1028	1048	9
-1	445	409	188
0	928	914	11
4	557	419	193
** K= 11 L= 12 **			
-7	1381	1384	190
-5	831	847	189
-3	1006	1081	189
-1	671	615	8
1	742	618	9
3	929	915	11
** K= 12 L= 12 **			
-8	924	952	12
-7	654	575	192
-6	758	648	192
-2	1388	1374	191
** K= 13 L= 12 **			
-7	924	944	11
-6	915	937	191
-5	647	674	191
-4	587	699	190
-3	967	998	12
-1	925	764	191
** K= 1 L= 13 **			
-11	598	679	192
-10	1078	1125	189
-9	736	677	187
-7	837	745	188
-6	1782	1839	188
-5	929	966	189
-4	798	768	7
-3	973	991	8
-1	883	820	187
0	463	497	10
1	1531	1487	10
2	913	978	10
5	598	547	12
6	794	777	10
** K= 2 L= 13 **			
-16	544	472	12
-12	1172	1129	9
-11	1197	1122	9
-9	1210	1282	188
-7	1007	1043	10
-6	535	516	185
-5	2618	2631	189
-4	2541	2609	188
-2	663	856	8
-1	1389	1466	188
0	2721	2697	189
1	1022	1100	187
2	1438	1463	8
3	1005	883	10
5	568	533	192
6	759	643	11
7	1167	1225	10
** K= 3 L= 13 **			
-12	382	381	185
-11	1162	1147	9
-10	1420	1414	9
-8	1024	1032	191
-7	592	325	10
-6	2400	2449	9
-5	1104	1098	11
-4	1515	1540	189
-3	1970	2026	189
-2	641	651	12
-1	2298	2267	8

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
1	2687	2725	188
2	1227	1273	189
3	749	793	11
4	847	867	10
5	819	721	191
6	1376	1276	191
** K= 4	L= 13 **		
-14	663	644	9
-13	453	390	10
-10	626	676	11
-9	1287	1200	10
-7	1883	1893	190
-6	760	638	190
-5	1346	1390	10
-4	1332	1348	7
-3	2084	2088	188
-2	2339	2386	189
0	1829	1901	9
1	793	718	6
2	1901	1869	189
3	759	789	191
4	1042	1037	9
5	1409	1436	10
7	671	705	190
** K= 5	L= 13 **		
-13	532	461	12
-12	655	725	8
-9	1319	1273	9
-8	1786	1832	10
-7	990	1020	7
-6	908	915	188
-4	1645	1714	8
-3	1216	1262	10
-2	924	1002	190
-1	2040	2013	190
0	764	668	191
1	849	951	8
3	1643	1622	190
4	1614	1599	189
** K= 6	L= 13 **		
-10	492	443	192
-9	467	528	188
-7	1098	1000	10
-6	478	496	10
-3	1536	1490	9
-2	1415	1346	10
2	759	931	9
5	910	751	191
** K= 8	L= 13 **		
-13	602	534	191
-9	643	543	192
-7	846	891	9
-6	867	882	9
-3	833	901	9
-2	1271	1260	10
2	540	526	10
** K= 9	L= 13 **		
-9	790	786	190
-8	1567	1482	190
-7	1045	971	188
-6	653	609	9
-4	1164	1191	189
-3	948	1036	190
-2	652	666	9
-1	1546	1592	11
0	508	422	10
1	533	445	191
3	1189	1293	10
4	1028	1041	11
** K= 10	L= 13 **		
-9	802	810	11
-7	1152	1178	191
-6	685	737	190
-5	861	943	10
-4	633	549	9
-3	1160	1161	190
-2	1613	1599	191
0	947	992	11
2	1049	1066	190
3	878	839	190
4	761	702	11
** K= 11	L= 13 **		
-10	576	571	192
-8	702	604	12
-6	1174	1168	191
-4	972	969	10
-3	981	891	11
-1	1112	1211	190
1	1043	1134	11
2	798	716	10
3	600	626	190
** K= 12	L= 13 **		
-5	1064	1043	192
-4	830	798	191
-1	658	588	190
0	1016	1135	190
** K= 0	L= 14 **		
-14	1137	1126	10
-13	577	486	190
-12	1019	1007	10
-8	752	843	186
-7	593	596	11
-6	2832	2847	189
-5	1598	1707	187
-4	1393	1499	188
-3	1361	1339	9
-2	476	495	186
-1	2324	2363	190
0	582	569	191
1	558	635	8
2	2479	2510	10
3	630	597	189
6	1659	1664	10
7	1110	1232	11
** K= 1	L= 14 **		
-14	841	711	9
-11	1473	1486	9
-9	1070	1063	9
-8	861	942	187
-7	1642	1714	9
-6	961	949	9
-5	964	906	190
-4	641	664	192
-3	1526	1456	188

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_2)_3CMe]$.

H	$/F_o/$	$/F_c/$	PHI
-2	2434	2427	8
-1	709	752	189
0	1272	1225	190
1	1807	1858	191
3	1338	1368	10
4	842	777	190
5	888	841	190
6	906	865	191
7	1042	1090	11
** K= 2 L= 14 **			
-14	560	604	190
-13	948	860	10
-12	667	522	192
-11	559	588	9
-9	356	314	188
-7	1367	1369	189
-6	1227	1264	10
-5	438	469	189
-4	370	322	8
-3	1642	1686	189
-2	538	338	187
-1	1317	1319	10
2	1551	1555	190
3	1029	1012	9
4	675	607	12
5	447	454	9
** K= 3 L= 14 **			
-15	813	566	10
-12	877	838	10
-10	1729	1736	9
-8	1201	1331	9
-7	602	459	186
-5	561	650	8
-4	794	714	188
-3	559	491	7
-2	2056	2075	189
0	882	850	191
2	715	655	191
3	1191	1108	190
6	590	479	11
** K= 4 L= 14 **			
-15	769	857	191
-14	536	234	12
-13	913	882	190
-10	670	397	10
-9	776	780	9
-7	1223	1230	10
-5	1926	2022	10
-3	822	814	8
0	923	889	11
1	936	996	190
3	752	812	190
6	1003	803	190
** K= 5 L= 14 **			
-10	1173	1178	190
-9	741	828	11
-6	773	686	191
-4	1571	1607	9
-1	806	909	191
0	1455	1532	10
1	877	869	9
3	654	437	190
5	1297	1379	10
** K= 6 L= 14 **			
-14	728	687	191
-12	704	676	190
-8	1188	1261	10
-6	604	697	11
-5	1285	1245	191
-4	1426	1359	9
-3	1395	1399	9
-2	577	672	9
-1	730	735	191
0	1430	1401	190
1	1448	1409	10
4	1768	1730	190
** K= 7 L= 14 **			
-11	967	1013	190
-9	1685	1690	190
-8	367	373	185
-6	605	511	12
-4	1377	1425	190
-3	1081	1073	10
-2	1291	1377	8
-1	1861	1902	10
0	979	976	189
2	1096	1090	11
3	1395	1403	10
4	724	584	11
5	1277	1189	191
** K= 8 L= 14 **			
-13	467	346	189
-12	728	637	10
-8	1171	1251	190
-6	735	720	189
-5	1071	1111	8
-4	1445	1447	189
-3	1247	1287	189
-2	429	526	190
-1	477	524	11
0	1129	1126	9
1	1418	1356	190
4	1353	1292	12
** K= 9 L= 14 **			
-9	826	712	11
-4	933	1003	10
-3	621	677	190
-1	637	729	189
0	1053	973	11
** K= 10 L= 14 **			
-7	1086	1057	190
-5	1166	1206	189
-4	454	235	9
1	808	695	10
** K= 11 L= 14 **			
-8	978	946	11
-2	1058	1062	191
** K= 12 L= 14 **			
-7	1014	897	11
-3	800	873	11
-2	521	441	10
-1	514	552	190

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
** K= 1	L= 15 **		
-15	524	449	11
-11	714	448	9
-10	603	664	9
-4	661	606	190
-1	883	847	190
0	816	901	191
4	442	454	190
** K= 2	L= 15 **		
-15	554	627	192
-13	689	631	8
-9	870	879	10
-8	976	955	9
-7	654	652	12
-6	857	907	10
-5	745	769	10
-2	820	792	8
0	564	588	188
1	1404	1452	189
2	705	638	190
5	1056	1023	190
6	1080	1002	191
** K= 3	L= 15 **		
-15	633	580	190
-14	824	821	191
-12	466	386	9
-10	785	659	190
-4	680	584	9
-3	456	470	185
-1	1103	1065	8
0	1291	1314	10
1	348	377	6
2	950	1075	189
4	1065	945	10
5	649	688	11
** K= 4	L= 15 **		
-13	881	798	189
-11	524	482	12
-7	546	516	5
-5	420	418	6
-4	718	726	8
-2	503	541	190
-1	865	868	188
1	862	890	10
3	1245	1251	190
4	923	872	189
6	720	639	11
** K= 5	L= 15 **		
-12	951	971	190
-11	784	778	189
-3	1182	1230	9
-2	978	932	11
1	535	526	11
2	1136	1237	10
3	460	381	9
4	696	567	190
5	722	620	191
** K= 6	L= 15 **		
-10	614	569	190
-9	494	439	189
-8	421	361	191
-7	590	485	189
-6	514	544	191
-5	626	642	191
-4	737	709	188
3	698	668	12
4	482	596	10
** K= 8	L= 15 **		
-7	670	680	190
-3	443	395	191
3	711	650	11
** K= 9	L= 15 **		
-3	711	698	190
-2	908	948	191
1	449	451	190
2	731	767	191
** K= 10	L= 15 **		
-4	641	517	10
-3	472	388	11
-1	641	601	190
** K= 0	L= 16 **		
-14	1263	1254	10
-13	729	790	12
-12	943	920	190
-9	2873	2988	9
-5	559	480	10
-4	1371	1397	8
-3	1068	1124	188
-2	683	566	6
-1	1182	1142	189
0	576	459	188
1	823	794	191
2	544	501	188
4	1044	995	191
5	589	547	190
** K= 1	L= 16 **		
-15	810	607	192
-14	1192	1027	191
-13	1236	1151	11
-11	589	512	190
-10	1204	1218	189
-8	1549	1639	10
-6	779	639	9
-5	769	832	189
-4	1113	1161	11
-3	652	517	10
-2	454	470	8
-1	761	757	10
2	795	752	190
3	643	594	11
** K= 2	L= 16 **		
-14	736	756	190
-12	952	991	10
-10	638	636	10
-9	1503	1432	189
-8	902	811	8
-4	1046	1079	188
-3	799	778	9
-2	814	871	188
-1	673	576	10
3	676	706	189

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{13}[P(OCH_3)_3CMe]$.

H	$/F_O/$	$/F_C/$	PHI
4	508	437	11
** K= 3 L= 16 **			
-13	1174	1190	191
-11	375	430	192
-8	690	624	190
-7	916	931	10
-5	1305	1365	11
-3	794	785	10
0	910	978	10
2	740	565	10
3	584	573	190
** K= 4 L= 16 **			
-10	1398	1384	190
-8	1118	1123	190
-5	642	741	191
-3	576	558	190
-2	960	901	10
0	829	912	9
1	685	621	9
3	733	601	10
** K= 5 L= 16 **			
-12	861	808	190
-11	833	766	10
-7	915	891	190
-6	878	928	11
-5	709	699	190
0	866	814	189
1	616	634	9
** K= 6 L= 16 **			
-11	1505	1452	190
-10	584	659	11
-7	1156	1212	190
-5	561	559	10
-3	608	526	10
-1	791	815	10
** K= 7 L= 16 **			
-12	1380	1359	10
-11	552	581	190
-10	606	571	191
-7	578	528	9
-6	1781	1779	189
-4	793	828	190
-1	964	802	189
1	853	843	190
** K= 8 L= 16 **			
-11	1295	1291	11
-7	978	1097	11
-3	506	495	191
-2	652	565	10
-1	660	512	191
** K= 9 L= 16 **			
-10	468	422	12
-7	661	751	190
-6	930	881	9
** K= 10 L= 16 **			
-8	752	742	12
-3	446	420	13
-2	580	648	191
** K= 1 L= 17 **			
-11	647	610	190
-9	703	722	12
-8	877	833	9
-5	719	811	8
-4	1054	1023	10
-3	512	423	12
** K= 2 L= 17 **			
-14	993	915	191
-12	456	432	12
-11	577	555	190
-10	1598	1620	191
-9	975	1002	190
-7	474	472	12
-6	604	649	189
-5	988	975	190
-4	574	521	5
-3	1210	1192	10
-2	865	824	10
1	790	764	10
2	695	602	10
** K= 3 L= 17 **			
-13	1007	972	190
-11	1220	1228	11
-9	1338	1327	191
-8	1134	1068	190
-7	600	611	10
-6	845	744	12
-5	682	664	189
-4	1470	1490	190
-2	660	609	9
** K= 4 L= 17 **			
-13	696	625	191
-12	1164	1226	191
-10	1206	1154	10
-8	1213	1179	191
-7	1036	1025	191
-5	1192	1281	10
-3	735	743	190
-1	807	746	10
0	697	722	9
** K= 5 L= 17 **			
-13	1092	940	11
-11	853	883	191
-9	776	727	10
-7	1169	1154	190
-6	1389	1376	191
-2	968	959	189
-1	674	553	192
** K= 6 L= 17 **			
-12	731	702	12
-8	725	784	10
-7	624	601	11
-5	625	696	189
-1	431	510	191
0	733	705	190
** K= 8 L= 17 **			

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_O	F_C	PHI
-8	715	560	11
-7	649	506	11
** K= 9	L= 17 **		
-9	420	453	191
-7	882	885	11
-6	1092	1049	11
-2	858	791	11
** K= 0	L= 18 **		
-13	1013	942	12
-12	526	442	191
-11	1390	1401	190
-10	1515	1468	190
-8	1980	1956	10
-7	634	599	192
-6	867	742	190
-5	609	652	192
-4	2084	2117	10
-3	1477	1473	10
1	1718	1666	11
** K= 1	L= 18 **		
-12	1330	1317	10
-10	688	485	190
-9	2156	2221	190
-8	445	518	10
-7	806	886	10
-5	1170	1266	190
-4	1512	1590	190
-3	1361	1377	10
0	1038	1050	190
2	835	983	11
** K= 2	L= 18 **		
-13	1071	1157	191
-11	473	445	8
-10	641	436	12
-8	1297	1356	191
-7	651	648	11
-6	691	467	8
-5	1051	1016	10
-4	813	912	191
-2	597	597	11
-1	620	623	8
0	518	442	11
1	810	867	191
** K= 3	L= 18 **		
-12	836	730	190
-11	628	333	7
-10	730	677	191
-9	406	417	10
-8	870	934	191
-7	658	602	191
-6	511	441	190
-5	632	459	190
-4	614	460	9
-3	869	907	191
0	548	609	11
** K= 4	L= 18 **		
-11	824	753	11
-10	725	597	11
-7	556	612	191
-6	653	659	12
-5	784	739	190
-3	733	771	190
0	974	885	190
2	560	554	191
** K= 5	L= 18 **		
-11	776	835	191
-10	1253	1206	10
-7	805	865	191
-5	1334	1265	10
-2	1011	1089	190
-1	556	581	11
** K= 6	L= 18 **		
-11	773	768	191
-10	614	614	191
-9	935	885	11
-8	667	748	11
-7	609	499	9
-6	1916	1909	190
-4	453	388	12
-3	532	561	12
-2	724	636	191
-1	1450	1386	190
** K= 7	L= 18 **		
-10	770	882	191
-7	1860	1846	11
-6	689	719	10
-5	993	993	191
-2	1143	1298	11
-1	574	613	192
** K= 8	L= 18 **		
-8	645	665	191
-6	1629	1627	10
-3	517	494	191
** K= 1	L= 19 **		
-10	817	833	190
-6	634	731	190
-5	569	669	191
-3	766	723	10
1	737	540	13
** K= 2	L= 19 **		
-12	616	571	11
-11	666	808	11
-9	848	911	191
-7	657	669	9
-5	1165	1136	190
-4	1531	1628	190
-2	757	673	9
0	962	978	191
1	714	684	191
** K= 3	L= 19 **		
-10	1324	1204	10
-8	938	949	190
-6	964	1066	11
-5	1415	1329	10
-4	415	460	191
-3	1361	1407	190
-1	1136	1051	10
0	485	448	10

Table I.3. Obs and Calcd Structure Factors for $Os_4(CO)_{15}[P(OCH_2)_3CMe]$.

H	F_o	F_c	PHI
** K= 4 L= 19 **			
-11	693	593	192
-9	1107	999	11
-7	1160	1145	191
-6	1070	1004	191
-4	1017	980	11
-2	1519	1458	190
-1	722	495	191
0	984	857	11
** K= 5 L= 19 **			
-9	503	430	10
-8	1229	1206	11
-4	562	524	10
-3	717	809	11
-1	1038	1045	191
** K= 6 L= 19 **			
-7	571	630	10
** K= 0 L= 20 **			
-9	1039	1145	190
-6	728	749	191
-5	1057	1172	191
-4	1081	1118	190
-3	1056	1045	11
** K= 1 L= 20 **			
-8	748	716	191
-7	862	838	12
-6	500	477	8
-4	1151	1211	191
-2	747	637	12
** K= 2 L= 20 **			
-9	582	608	10
-8	724	650	191
-7	584	486	190
-4	520	510	10
-3	1134	1103	191
** K= 3 L= 20 **			
-8	759	672	11
-7	555	476	190
-2	647	596	191
** K= 4 L= 20 **			
-8	495	548	10
-5	966	922	11
-3	724	575	9
** K= 5 L= 20 **			
-6	1006	1051	190
** K= 2 L= 21 **			
-7	502	406	9
-6	690	618	10

Table J.1. U_{ij} or U_{iso} Values (*100) for $Os_4(CO)_{15}(PF_3)$.

	U11(U)	U22	U33	U12	U13	U23
Os(1)	3.61(7)	3.16(6)	3.25(6)	0.23(6)	-0.57(6)	-0.13(5)
Os(2)	3.56(7)	3.19(6)	2.84(6)	0.43(6)	0.42(6)	0.04(5)
Os(3)	3.55(7)	3.36(6)	2.43(5)	-0.02(5)	-0.02(6)	0.31(5)
Os(4)	3.24(7)	3.05(6)	3.74(6)	0.10(5)	0.96(6)	-0.22(5)
F(1)	12.4 (22)	7.5 (15)	21.6 (30)	5.5 (15)	-7.0 (22)	-1.6 (17)
F(2)	5.8 (17)	22.3 (32)	18.4 (31)	1.1 (20)	-1.5 (19)	6.5 (24)
F(3)	17.2 (30)	22.7 (31)	17.2 (29)	10.1 (25)	-15.2 (26)	-10.5 (25)
P	6.0 (7)	5.5 (5)	7.4 (7)	-0.2 (5)	-2.2 (6)	-1.0 (5)
O(11)	12.7 (23)	2.6 (10)	4.6 (13)	-2.0 (12)	-2.0 (14)	-0.7 (10)
O(12)	12.3 (24)	8.6 (18)	4.5 (15)	2.0 (17)	-0.8 (16)	1.2 (13)
O(13)	7.3 (17)	6.1 (14)	5.2 (13)	-1.9 (13)	-2.3 (13)	-2.6 (11)
O(21)	4.8 (14)	6.8 (14)	3.3 (12)	-0.8 (11)	0.9 (10)	1.0 (10)
O(22)	6.6 (19)	9.6 (19)	8.9 (19)	0.8 (16)	-0.4 (16)	3.3 (15)
O(23)	9.9 (19)	4.3 (12)	4.4 (13)	0.2 (14)	-0.9 (13)	0.7 (10)
O(24)	5.4 (16)	8.7 (16)	5.1 (14)	2.8 (14)	1.1 (12)	0.0 (12)
O(31)	9.1 (19)	6.0 (13)	5.7 (14)	-4.2 (14)	-1.1 (14)	0.3 (11)
O(32)	10.4 (21)	10.7 (18)	2.6 (12)	1.3 (16)	0.2 (14)	-0.2 (12)
O(33)	7.1 (16)	5.0 (12)	8.1 (17)	3.7 (12)	-0.7 (14)	1.5 (12)
O(34)	7.4 (17)	5.9 (13)	7.5 (15)	0.1 (13)	-2.1 (14)	0.7 (13)
O(41)	11.2 (24)	11.4 (20)	3.6 (13)	2.2 (18)	1.2 (14)	0.6 (13)
O(42)	5.2 (16)	6.5 (15)	10.8 (20)	-3.7 (13)	4.3 (14)	-3.5 (13)
O(43)	5.6 (18)	9.9 (19)	11.8 (23)	2.5 (15)	3.8 (16)	4.5 (16)
O(44)	6.4 (16)	5.0 (13)	6.6 (15)	-3.7 (12)	2.6 (13)	0.1 (11)
C(11)	4.0(7)					
C(12)	6.1(10)					
C(13)	5.7(9)					
C(21)	4.0(7)					
C(22)	4.6(8)					
C(23)	5.2(9)					
C(24)	6.1(9)					
C(31)	4.9(8)					
C(32)	3.8(7)					
C(33)	6.7(10)					
C(34)	5.8(9)					
C(41)	4.6(8)					
C(42)	3.8(7)					
C(43)	4.4(8)					
C(44)	4.0(8)					

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	0, 0, L		
2	2179	2036	336
4	8831	8738	294
6	3357	3400	462
8	5930	5945	459
10	2051	2082	806
14	1350	1520	1509
18	1421	1193	1818
20	3465	3344	1049
	0, 2, L		
0	1915	1694	377
1	10572	10459	217
2	2195	2142	424
4	604	521	1125
5	5789	5599	369
6	7555	7379	383
7	6590	6402	425
8	917	839	746
9	1089	1168	1249
10	1911	1922	850
11	1607	1603	1031
12	2890	2974	791
14	4052	3988	738
15	1478	1449	1478
16	1262	1235	1688
17	1940	1985	1400
18	3122	3120	1092
19	3791	3857	1093
	0, 4, L		
0	5618	5492	322
1	1657	1685	647
2	4950	4862	352
3	1940	1905	614
4	2878	2832	474
5	5103	5116	415
6	1726	1735	761
7	4802	4644	468
9	1345	1324	1053
11	2268	2338	879
12	1298	1214	1406
13	7576	7570	627
15	2753	2818	976
16	1101	1096	1179
17	2980	3085	1042
19	2683	2641	1237
	0, 6, L		
0	1083	1083	1009
1	5777	5592	392
3	2395	2369	568
4	2047	1998	621
6	9385	9346	443
7	2030	2070	742
8	2675	2716	683
9	3824	3885	612
10	3887	3969	647
11	1165	1058	1412
12	1794	1768	1174
13	3534	3642	828
14	5013	4959	736
17	2078	2164	1331
18	3423	3465	1035
	0, 8, L		
0	11319	11331	421
1	3185	3187	550
2	6012	5830	456
3	2782	2782	594
4	1677	1568	788
5	1378	1313	959
6	1130	1311	1252
7	3310	3269	607
8	5322	5430	582
9	2267	2329	818
10	922	703	1613
11	5522	5599	657
12	1002	1036	1407
13	4190	4191	760
14	1753	1687	1312
15	1393	1564	1628
18	2258	2349	1392
	0, 10, L		
0	6077	5978	513
1	6621	6665	512
4	4592	4686	567
5	1551	1670	1004
6	1350	1430	1126
7	3449	3504	689
8	2371	2402	880
9	4146	4053	684
10	2371	2432	924
11	2222	2141	988
12	1184	1221	1771
13	1980	1973	1210
14	1239	1080	1344
16	2702	2641	1146
17	1007	900	1553
	0, 12, L		
0	5570	5605	584
1	1552	1629	1111
2	2509	2566	779
3	1107	657	1317
5	5422	5464	614
8	4191	4103	735
9	3013	2932	850
11	3033	2963	882
12	1471	1393	1419
13	1066	831	1340
15	3786	3987	968
16	2649	2796	1268
	0, 14, L		
0	2240	2178	938
1	1175	1211	1496
2	4174	4253	725
3	3523	3727	774
4	2760	2690	904
6	2418	2399	948
7	2210	2259	1048
8	1270	1341	1666
10	1385	1435	1611
11	1230	1107	1697
13	1611	1555	1561
14	2612	2586	1150
	0, 16, L		
0	1766	1924	1261
1	2225	2443	1108
2	3995	4067	788
3	3673	3667	801
5	2659	2839	978
8	1179	1148	1527
9	1816	1902	1391
11	1474	1284	1591
	0, 18, L		
1	2112	1882	1150
2	4096	4265	881
9	2747	2633	1091
10	1490	1623	1767
11	1645	1677	1632
	0, 20, L		
3	1485	1460	1764
4	1960	1810	1336
	1, 0, L		
2	8741	8772	263
4	4598	4608	388
6	13359	13318	378
8	2486	2588	662
10	1224	1336	1237
12	3700	3729	712
14	1812	1718	1142
18	2951	2923	1087
	1, 1, L		
1	3041	2750	267
2	4885	4789	276
4	2668	2552	476
6	6323	6205	398
7	3813	3883	505
8	3706	3730	543
11	764	980	1308
12	4672	4630	635
20	1982	2208	1744
	1, 2, L		
1	9485	9497	230
2	1018	853	806
3	774	919	1101

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	1, 2, L		
4	2011	2002	565
5	8241	8107	354
6	1742	1702	728
7	1067	1088	1167
8	3259	3214	557
9	1041	952	880
10	2247	2274	762
11	1575	1750	1166
12	4657	4645	645
18	1788	1585	1521
19	1474	1637	2077
20	1488	1327	1460
21	1210	1322	2246
	1, 3, L		
1	5951	5759	286
2	791	877	1142
3	894	893	1077
4	1510	1409	713
6	1876	1798	700
7	5729	5648	447
8	1724	1781	836
10	873	754	936
11	2700	2762	749
12	1300	1276	1468
13	2417	2424	934
15	1880	1864	1198
17	1385	1333	1745
	1, 4, L		
1	3800	3549	374
2	1515	1366	668
4	1820	1752	654
5	3227	3167	497
6	2532	2526	571
7	4911	4813	473
8	2663	2769	629
9	2238	2244	736
10	1178	1330	1301
11	6062	6193	583
12	2859	2911	815
13	3823	3879	759
14	1261	1355	1640
16	1108	1098	1716
18	1758	1887	1692
19	3012	2861	1104
	1, 5, L		
1	3290	3182	448
2	1571	1563	728
4	2578	2520	542
6	3940	3911	499
7	5502	5573	492
10	1233	1224	1246
11	1852	1782	1010
12	2539	2554	894
13	1613	1543	1290
14	1386	1272	1108
15	1012	1045	1736
18	1295	1100	1299
20	1232	1180	2137
	1, 6, L		
1	3212	3115	486
2	1146	1129	995
3	3797	3733	462
4	2337	2307	595
5	960	974	1233
6	1939	2076	737
7	4331	4319	539
8	4407	4422	559
11	928	871	1588
12	6450	6602	638
18	2414	2144	1189
20	1427	1313	2017
	1, 7, L		
1	1381	1449	872
2	3315	3199	515
3	1132	1143	1073
4	1944	1949	693
5	1698	1646	823
6	3888	3995	537
8	2467	2464	746
9	2373	2243	728
12	4239	4222	714
14	928	923	1665
	1, 8, L		
1	4285	4291	493
2	4080	4055	510
3	988	1083	1237
4	4770	4761	509
5	5895	5921	505
6	3730	3744	593
7	1895	1906	875
8	2760	2755	722
9	1614	1561	1015
10	1979	2069	1024
11	1895	1919	1098
12	1292	1246	1542
13	1179	951	1687
14	1345	1308	1563
16	995	1203	1411
19	1541	1439	1503
	1, 9, L		
1	2738	2658	619
2	1506	1531	887
3	944	929	1282
5	1444	1520	1047
6	3006	3067	685
7	3702	3730	655
8	2341	2350	831
9	2452	2480	832
10	1642	1739	1195
11	1290	1042	1381
12	1879	1762	1132
15	1686	1786	1478
17	1312	1374	1326
	1, 10, L		
1	3034	2990	611
2	1229	1133	1089
3	3409	3345	599
5	3396	3289	646
6	3979	3951	637
7	2759	2702	756
9	1433	1331	1208
10	4476	4496	717
11	1493	1477	1330
12	1381	1282	1533
14	2814	2648	960
16	1760	1751	1533
17	1733	1597	1545
	1, 11, L		
1	2823	2880	714
2	937	874	1162
4	3044	3025	671
7	3351	3328	735
8	954	1182	1487
10	2521	2428	929
11	1724	1422	1247
12	1018	988	1871
13	1543	1410	1414
14	1450	1289	1557
	1, 12, L		
1	3155	3062	684
2	2869	2907	755
3	3334	3520	707
5	2053	2042	949
6	1984	1786	964
8	1756	1692	1149
9	3275	3349	814
10	1793	2067	1313
11	2484	2625	1030
12	1358	1651	1790
13	1637	1610	1540
17	1284	1460	2007
	1, 13, L		
2	1906	1902	1011
3	1245	1199	842
4	2528	2461	826
5	2374	2247	874
6	2064	1969	1008
7	1322	1315	1473
9	1797	1864	1309
10	1333	1368	1595
11	1456	1098	1336

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$.
Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	1, 13, L		
12	1752	1734	1362
14	1342	769	1711
	1, 14, L		
1	1440	1367	1327
3	1195	1311	1542
4	3645	3663	759
7	1512	1549	1338
9	1448	1514	1546
10	3467	3399	878
11	1439	1766	1706
14	1479	1747	1847
	1, 15, L		
1	2139	1998	1065
3	1716	1615	1196
5	1319	1359	1252
6	1672	1658	1231
9	2480	2378	1039
12	1569	1200	1419
	1, 16, L		
3	3952	3905	788
4	2610	2535	971
5	1621	1580	1328
6	1222	628	1585
7	1149	1239	1582
8	1981	1998	1274
11	1024	914	1980
	1, 17, L		
1	1550	1408	1413
4	1884	1894	1260
8	1618	1502	1520
9	1310	1340	1541
10	1415	1392	1805
	1, 18, L		
3	2954	2959	970
4	2357	2578	1240
7	1845	1707	1388
8	1732	1528	1452
	1, 19, L		
4	1879	2009	1548
8	1293	580	1886
	1, 20, L		
4	1201	1129	1890
	2, 0, L		
0	7795	7001	360
2	951	1129	1359
4	3451	3373	512
6	13222	13273	412
10	4709	4756	599
14	4352	4239	768
18	2676	2461	1175
20	1669	1629	1942
	2, 1, L		
0	8660	8659	291
1	2073	2091	557
2	1958	1976	593
4	4383	4250	411
5	5162	5184	420
6	4223	4167	478
8	2390	2337	671
11	1742	1621	1041
12	2204	2389	1016
14	3691	3837	849
15	1295	1451	1782
18	2416	2451	1245
19	1481	1410	2103
20	1475	1317	2157
	2, 2, L		
0	6906	6890	291
1	3337	3173	382
3	2847	2759	460
4	697	990	1244
5	2342	2389	586
6	1971	1825	654
7	5419	5381	467
8	2944	2960	595
9	1717	1726	900
11	5568	5695	596
13	5302	5377	661
16	1333	1598	1811
17	1682	1579	1487
	2, 3, L		
0	3541	3480	383
1	5452	5289	329
2	2756	2704	458
3	1474	1450	757
4	2064	1956	588
5	4514	4572	440
6	1044	1058	1124
8	1505	1503	913
9	2010	1968	763
12	1236	1114	1411
13	3708	3757	798
14	1053	1030	1164
15	1648	1900	1371
17	1196	1209	1879
18	1309	1203	1590
19	2738	2734	1268
	2, 4, L		
0	3440	3243	409
1	5581	5462	354
2	1686	1634	637
3	2351	2244	547
4	2101	2145	622
6	3981	3982	490
7	7271	7240	454
8	1794	1819	803
9	2743	2774	691
10	2055	2164	824
13	1136	943	1625
14	1114	1150	1673
15	1381	1225	1376
17	2595	2692	1152
19	1630	1773	1903
	2, 5, L		
0	6371	6231	363
1	5612	5485	381
2	1915	1762	631
3	2976	2953	495
4	1600	1611	753
5	3135	3011	500
6	1539	1418	839
8	1547	1725	973
9	2546	2497	709
13	3880	3970	770
14	1295	1307	1628
17	1912	1858	1363
18	1490	806	1747
19	1847	1791	1644
	2, 6, L		
0	11964	12053	372
1	1351	1443	875
2	3651	3528	464
3	2270	2309	584
4	2127	2131	640
5	2762	2734	587
6	2820	2810	612
7	1134	1013	1118
8	4258	4290	573
9	2175	2168	795
10	1067	1159	1460
11	1353	1332	1313
15	1879	1730	1202
16	2095	2261	1282
	2, 7, L		
0	5242	5173	442
1	1609	1606	781
2	1626	1621	786
3	2195	2249	654
4	1331	1354	940
5	1033	1074	1212
6	3883	3982	556
8	2571	2701	754
11	2021	2069	1055
12	1762	1766	1179
14	2697	2853	1031
15	1794	1903	1430
17	1454	1167	1750
18	2418	2381	1335
	2, 8, L		
1	2990	3008	587
2	5042	5072	494

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	2, 8, L		
3	2458	2459	639
4	1586	1616	865
5	2808	2689	648
6	3327	3349	618
7	4853	4848	574
8	1809	1739	909
10	2080	2062	952
11	1220	1053	1241
13	1374	1527	1546
14	2190	2240	1141
17	2157	2418	1508
19	2079	1784	1477
	2, 9, L		
0	4026	3915	552
1	1341	1351	1026
2	2708	2700	646
3	2031	1885	728
4	2817	2813	652
5	2074	2052	787
6	1863	1921	904
9	1195	1280	1561
10	990	1027	1082
11	1733	1728	1188
12	2050	1989	1114
13	1754	2011	1361
14	2253	2317	1179
16	1787	1664	1417
18	1467	1611	2048
	2, 10, L		
0	4024	3991	586
1	2763	2650	693
2	3893	3835	578
3	3449	3412	620
4	2858	2865	682
5	4050	4040	625
6	1570	1744	1121
7	1771	1894	1044
8	1074	992	1264
9	1743	1559	1135
11	1982	1966	1128
13	2501	2479	1095
15	1333	1298	1794
	2, 11, L		
0	2649	2642	726
1	3351	3295	660
2	2160	2181	841
4	2421	2316	793
5	2844	2939	765
6	1443	1435	1236
8	1671	1637	1136
10	1837	1847	1183
13	1910	1947	1325
15	1553	1446	1682
16	1796	1853	1559
	2, 12, L		
1	1917	1795	938
2	2623	2690	804
3	1108	917	1395
5	2485	2385	814
6	4030	4035	704
9	3739	3754	801
10	2239	2213	1032
11	2586	2509	1002
13	1449	1488	1653
14	2022	2146	1352
15	2473	2394	1187
	2, 13, L		
0	3367	3513	753
1	3195	3280	785
2	2511	2454	870
3	2706	2767	844
4	1216	1129	1173
5	1183	1165	1608
8	1202	1076	1496
11	1592	1401	1202
13	1043	1125	1444
15	1377	1361	1150
	2, 14, L		
0	2184	2078	954
2	2958	2808	830
4	1943	1899	1054
8	4163	4218	791
11	1514	1729	1629
13	1542	1672	1688
	2, 15, L		
0	2335	2346	995
2	1175	1275	1657
3	2640	2691	919
4	779	770	1476
7	1170	971	1679
11	1617	1642	1478
	2, 16, L		
1	2360	2243	970
2	1934	1835	1135
4	1298	1419	1668
7	1514	1421	1465
8	1781	1654	1333
9	2920	2878	1015
11	1799	1837	1522
	2, 17, L		
2	2828	2816	986
3	1347	1122	1398
12	1073	1101	1559
	2, 18, L		
0	1418	1403	1672
3	1793	1719	1351
5	2173	2118	1169
8	2249	2053	1277
9	2089	1903	1319
	2, 19, L		
2	1526	1600	1744
4	947	1165	1699
7	980	629	1515
	2, 20, L		
2	2971	2935	1108
4	1672	1380	1561
	3, 0, L		
4	3748	3650	548
8	6776	6873	521
12	10833	10903	613
14	2237	2130	1095
18	2171	2165	1384
20	2578	2537	1377
	3, 1, L		
1	861	805	1186
2	1058	1011	1263
4	2536	2538	639
5	3279	3239	566
6	1989	2047	774
8	1891	1909	849
9	2548	2656	790
10	4259	4450	643
11	1304	1444	1452
14	1756	1774	1319
17	1075	1129	1491
	3, 2, L		
1	1649	1584	788
2	4290	4245	416
4	676	616	1169
5	5766	5620	432
6	6044	6079	458
7	6483	6543	481
8	1151	995	1161
11	4926	5004	654
13	3488	3729	844
15	1785	1965	1361
19	2621	2817	1385
	3, 3, L		
1	1582	1577	745
3	1719	1782	755
4	2732	2601	533
5	3497	3567	513
8	2656	2646	678
9	4288	4346	589
10	1204	1062	747
11	1306	1376	1357
12	1184	1635	1727
13	1193	1224	1673
15	1348	1299	1595
17	1293	1233	1182
	3, 4, L		

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	3,	4, L	
1	7897	7867	367
3	4064	4034	445
4	1870	2000	719
5	7395	7343	430
6	2084	2094	724
7	5151	5208	513
8	2192	2161	742
9	1783	1797	910
10	2163	2372	891
12	3478	3520	742
14	1902	1988	1278
15	1256	1247	1773
17	2053	1977	1256
19	1113	852	2134
	3,	5, L	
1	3866	3757	458
3	3831	3866	476
4	985	1203	1222
5	2925	2862	549
7	3085	3060	593
8	647	575	1238
9	2793	2741	700
10	1177	1204	1414
11	1768	1824	1066
12	1333	1316	1417
	3,	6, L	
1	2722	2779	548
2	7299	7298	420
3	2271	2292	611
5	2472	2531	664
6	7464	7425	479
7	682	867	1210
8	1121	1072	1294
9	2778	2793	726
10	1061	1222	1551
11	1918	1996	1020
12	1280	1085	1404
13	1614	1630	1282
18	3221	3120	1075
	3,	7, L	
1	1248	1123	915
4	3544	3564	530
5	758	885	969
7	1858	1957	884
8	2490	2597	777
9	1082	1154	1473
10	1891	1978	1012
11	1934	1855	1055
12	1138	1213	1131
13	1107	1119	1441
16	958	777	1398
18	1878	1607	1601
	3,	8, L	
1	6261	6217	475
2	1080	1111	1182
3	2407	2353	682
4	4030	4042	561
5	2388	2378	699
6	2859	2841	675
8	1744	1702	974
9	2005	1946	914
10	3264	3428	770
11	1351	1556	1497
12	3938	3948	757
16	1506	1328	1542
19	1487	1352	1246
	3,	9, L	
1	3767	3821	574
3	2276	2229	734
4	977	1018	1211
6	2490	2457	728
8	1405	1578	1293
9	1318	1233	1017
10	3024	3121	835
14	1272	1347	1836
16	1319	689	1780
	3,	10, L	
1	2569	2616	732
2	961	811	978
3	3725	3747	632
4	3613	3729	646
5	2861	2909	751
7	2474	2559	853
8	3772	3811	741
11	3565	3600	855
12	2373	2453	1111
13	2943	2902	984
14	1036	971	1516
15	1207	1282	1319
16	1455	1450	1856
18	1293	1415	1438
	3,	11, L	
2	1366	1135	1113
5	2854	2839	764
6	2383	2546	908
9	2793	2757	877
11	874	795	1382
	3,	12, L	
1	951	866	1608
3	5440	5494	629
4	5265	5225	636
6	1117	735	1567
7	4526	4304	699
9	1267	1081	1556
12	2903	3170	1012
17	1702	1513	1735
	3,	13, L	
1	1491	1367	1249
3	1133	1152	1362
5	1843	1870	1125
7	1639	1779	1355
10	1284	1246	1712
12	1530	1568	1646
	3,	14, L	
2	2903	2882	861
4	1632	1803	1306
5	3340	3286	805
6	1557	1655	1380
8	1415	1601	1615
9	2662	2545	971
10	3258	3395	946
12	1387	1397	1806
14	1965	2149	1535
	3,	15, L	
3	960	881	1280
4	1330	1170	1307
7	2397	2562	1107
11	1323	1487	1946
	3,	16, L	
1	1210	1084	1269
3	1826	1862	1215
6	1322	1329	1641
7	1926	2030	1334
9	2884	3127	1137
10	2793	2579	991
	3,	17, L	
1	1043	760	1425
8	1713	1864	1579
12	1711	1813	1709
	3,	18, L	
1	1785	1724	1349
2	1335	1098	1796
3	2854	2814	1048
10	2541	2678	1307
	3,	19, L	
6	1368	1403	1147
	3,	20, L	
4	3549	3546	1063
5	1212	1057	1530
	4,	0, L	
0	3800	3657	641
2	4333	4213	598
6	4771	4779	573
8	2046	2137	950
10	1232	1205	1358
12	3598	3679	823
14	4836	4659	778
16	3404	3394	1001
18	3352	3454	1107
	4,	1, L	
0	2977	2900	714

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	4,	1,	L
1	2327	2147	750
2	1812	1626	908
6	3408	3207	606
8	4604	4594	591
9	3139	3129	718
10	2679	2782	850
11	3730	3788	731
13	2169	2309	1147
14	1598	1515	1450
15	1408	1471	1715
16	2083	2050	1305
18	1223	1151	2172
	4,	2,	L
0	6217	6229	447
1	1113	1248	1350
2	4842	4904	470
3	3757	3748	515
4	1928	1804	785
7	6363	6492	515
8	1358	1490	1173
9	992	1050	1471
10	1876	1894	982
11	1509	1417	1040
12	1254	1220	1537
13	4777	4780	764
15	1220	1248	1179
17	4069	3987	918
	4,	3,	L
0	1153	1248	1199
1	1477	1461	884
2	4273	4325	477
3	4211	4187	483
4	2767	2799	594
5	4257	4274	516
7	1056	1026	1373
8	1352	1235	1076
9	1862	1841	915
11	3356	3520	784
13	2772	2811	958
17	1080	924	2081
	4,	4,	L
0	6304	6375	424
1	12203	12439	395
2	1825	1846	722
3	5053	5058	457
4	3092	3070	570
5	877	802	1168
6	2913	3028	639
7	4061	4164	569
9	1738	1611	956
10	1366	1206	1216
11	3921	3936	717
13	1267	1331	1648
15	1642	1683	1473
19	1954	1931	1701
20	1482	989	2013
	4,	5,	L
0	1550	1402	792
1	888	929	1324
2	2009	2135	715
3	2887	2808	577
5	3956	4011	550
6	3189	3220	613
7	2066	1979	767
8	2570	2651	729
9	1115	1135	954
10	1497	1512	1204
14	1745	1648	1294
16	1724	1672	1495
19	2003	1429	1405
	4,	6,	L
0	8435	8428	432
1	2362	2399	640
2	3291	3335	552
3	1947	1999	739
4	4233	4182	518
5	3135	3176	594
6	4449	4486	554
8	1626	1685	1019
9	1747	1753	1048
10	2340	2297	881
11	2510	2633	912
13	1324	1170	1566
15	3253	3229	935
17	1519	1215	1753
19	1536	1611	2001
	4,	7,	L
0	5972	5922	478
2	5236	5260	496
3	2073	2185	718
4	1381	1367	986
5	1885	1926	821
6	931	866	1317
8	2278	2162	833
10	1414	1357	1218
14	1877	1792	1264
16	1547	1533	1308
	4,	8,	L
0	1562	1591	908
1	1124	1116	1177
3	1090	947	1206
4	3844	3923	593
6	2056	1948	762
7	3462	3484	676
8	3091	3164	749
9	1368	1201	1277
11	1887	1976	1194
12	2270	2288	1052
13	3296	3314	864
14	3153	3226	979
16	3594	3461	980
17	1874	1829	1533
18	1458	1517	2096
	4,	9,	L
1	1931	2027	838
2	1014	771	1263
3	3546	3640	629
4	880	1078	1538
5	3436	3507	643
6	766	660	1408
7	2067	2252	913
8	1785	1938	1115
11	1180	1198	1766
16	1022	804	1599
17	1415	1291	1786
	4,	10,	L
1	1671	1676	968
2	5926	5985	561
4	1175	859	1256
5	1689	1846	1103
6	2962	2994	756
7	3085	3135	789
8	1722	1647	1144
9	1963	1946	1104
12	1636	1494	1402
13	1782	1721	1325
14	2350	2019	1072
15	2407	2413	1218
17	1903	1893	1648
18	2136	1981	1513
	4,	11,	L
0	5042	5031	609
2	2217	2372	899
3	1952	1836	930
5	1482	1479	1225
8	1665	1676	1281
9	1723	1654	1230
11	2598	2549	1004
13	1957	1919	1344
	4,	12,	L
0	4528	4611	669
1	4589	4614	668
2	2052	1960	949
3	3803	3779	725
6	1334	1292	1421
8	1874	1884	1161
9	2939	2995	958
10	1422	1435	1541
13	1985	1980	1314
	4,	13,	L
0	992	955	1725

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	4, 13, L		
4	1373	1334	1344
6	2809	2889	917
7	1217	903	1553
8	2794	2710	941
10	1624	1634	1443
14	1059	1232	1733
	4, 14, L		
0	916	1037	1665
1	2768	2792	899
2	4389	4364	748
3	1556	1461	1323
4	1393	1258	1457
5	1655	1573	1344
8	2601	2585	1045
11	1104	1303	2079
15	1569	1414	1768
	4, 15, L		
3	1444	1602	1528
5	1296	1300	1625
9	1674	1502	1411
11	1810	1968	1592
13	1893	1901	1505
	4, 16, L		
0	2314	2353	1096
4	2595	2535	991
5	2030	2033	1161
8	1374	1296	1541
9	1730	1821	1568
	4, 17, L		
3	1391	1484	1738
5	1253	1267	1586
6	1611	1689	1539
8	1443	1033	1639
10	1290	1019	1922
	4, 18, L		
0	1452	1378	1616
4	1575	1503	1581
5	1666	1600	1555
6	1256	1063	1868
9	1938	1907	1513
	4, 19, L		
0	2109	2431	1351
2	1422	1372	1708
	4, 20, L		
1	1469	1493	1885
2	2767	2874	1211
	5, 0, L		
2	7144	6828	549
4	4481	4546	605
6	4076	4183	654
8	5441	5510	630
10	4403	4470	728
12	3622	3667	859
18	4711	4614	981
	5, 1, L		
1	4172	4104	623
2	3359	3300	665
3	4039	4051	592
4	690	597	1364
6	2209	2246	851
7	3054	3042	707
8	2999	3027	728
9	826	870	1227
12	2390	2417	1050
17	1525	1284	1689
	5, 2, L		
1	8462	8584	473
3	3246	3270	632
4	4482	4526	539
5	2420	2377	706
6	2644	2652	688
9	4853	4877	622
10	2294	2389	911
11	2687	2788	935
12	2344	2380	1025
13	1491	1582	1474
14	1603	1442	1430
15	1417	1585	1791
17	1799	1754	1605
19	1766	2071	1822
	5, 3, L		
2	743	691	1349
3	1707	1675	840
4	1779	1794	869
5	1005	1120	1316
6	2594	2641	728
7	3567	3546	619
9	1664	1689	1088
10	3940	3948	693
11	2062	2127	1048
13	1262	1332	1650
14	1433	1524	1593
16	1483	1485	1710
20	1393	1218	1417
	5, 4, L		
2	3262	3348	589
3	5666	5837	493
4	1328	1239	1022
5	8603	8842	487
6	3448	3507	625
8	1402	1569	1240
9	3508	3539	698
11	2205	2176	978
13	1528	1655	1519
15	2547	2531	1059
16	1037	1201	1473
18	1693	1519	1663
19	1521	1310	1920
	5, 5, L		
1	3359	3432	576
3	1342	1341	1064
4	928	786	1385
5	3135	3208	629
6	1059	936	1270
7	1220	1367	1309
8	1231	1252	1295
9	3465	3500	728
11	1941	2017	1095
12	1192	1283	1501
13	1657	1629	1422
	5, 6, L		
1	1018	1002	1302
2	1767	1786	817
3	1854	1807	809
4	6983	7012	501
5	2637	2741	690
6	2777	2849	700
8	1314	1238	1224
9	3974	3975	695
10	3053	3029	792
11	1521	1807	1409
12	5243	5394	748
13	1222	1300	1817
14	2402	2372	1064
17	1181	1282	2089
	5, 7, L		
2	1605	1563	869
3	2421	2482	701
5	1195	1199	1198
6	3617	3568	636
7	1771	1674	978
9	1014	920	1277
10	2692	2733	879
14	1335	1316	1647
	5, 8, L		
1	1004	788	1323
2	2828	2980	672
5	3931	4017	644
7	3743	3882	683
8	2061	2171	954
9	1376	1415	1223
10	5958	5952	678
11	2733	2588	939
12	1218	1216	1812
13	1733	1843	1449
14	2464	2536	1126
15	1547	1638	1703
18	1890	2021	1737
	5, 9, L		
1	2815	2841	700
2	1441	1464	1073

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	5, 9, L		
3	1839	1772	940
4	2440	2515	794
5	1788	1869	948
6	1869	1925	949
8	2850	2773	848
12	1308	1350	1611
	5, 10, L		
1	3651	3679	663
3	1429	1477	1175
4	1807	1724	997
7	2703	2527	822
8	3547	3699	804
9	4645	4586	735
10	963	848	1241
11	1864	1991	1293
12	2699	2942	1070
13	1228	957	1790
14	1139	1029	1241
15	1918	1879	1520
17	2062	2031	1525
	5, 11, L		
3	2194	2165	907
4	1443	1463	1262
7	2542	2569	924
8	2351	2448	1024
10	986	801	1274
11	1547	1545	1500
12	1841	1685	1304
	5, 12, L		
1	2202	2145	949
2	3511	3560	761
3	4986	5073	674
5	1307	1183	1349
6	3400	3313	823
7	2574	2522	936
10	2078	2120	1225
14	1386	1117	1699
	5, 13, L		
7	2102	2047	1098
8	1435	1129	1390
9	1529	1691	1521
10	1379	1281	1033
11	1820	1660	1390
12	1610	1479	1572
	5, 14, L		
1	2980	2932	876
4	4889	4689	752
5	1837	1775	1222
7	2226	2044	1148
8	1695	1224	1309
12	1010	1141	1972
	5, 15, L		
1	1247	1313	1654
5	2205	2189	1185
8	1341	1005	1732
9	1378	1412	1815
	5, 16, L		
1	2272	2210	1160
3	2921	3006	1020
6	1646	1525	1479
7	1580	1275	1478
10	2268	2242	1245
11	1962	1934	1545
	5, 17, L		
6	1813	1810	1451
10	1352	1429	2011
	5, 18, L		
3	2242	2017	1171
4	1856	1733	1446
6	1411	1551	2005
7	1591	1319	1647
9	2144	1918	1357
	5, 19, L		
5	1334	1326	1984
	5, 20, L		
2	1924	1861	1569
	6, 0, L		
0	7706	7559	553
2	1860	1855	1149
4	1988	1916	1021
6	3741	3764	726
8	4531	4601	721
16	2321	2441	1393
	6, 1, L		
1	2265	2182	989
2	7357	7394	565
3	3676	3738	675
4	1732	1706	1056
5	2123	2326	933
7	1146	1035	1464
9	2070	1924	1023
10	1629	1896	1352
12	2375	2340	1107
16	2985	3218	1181
17	1744	1694	1785
	6, 2, L		
0	1115	1056	1508
1	4162	4230	629
2	2433	2395	801
3	2501	2501	784
5	2274	2312	796
6	2943	2821	697
7	5290	5346	601
10	1752	1868	1302
11	1364	1298	1530
13	1420	1653	1712
14	1468	1576	1670
17	2265	2183	1423
19	1375	1154	1338
	6, 3, L		
0	3721	3758	643
1	1428	1357	1134
2	2949	2926	687
3	6650	6670	517
4	4766	4783	574
5	1467	1552	1136
7	2621	2760	819
8	2486	2545	855
10	1445	1649	1441
11	3117	2987	862
13	946	843	1524
14	1316	1454	1810
15	2001	2140	1373
16	1636	1919	1747
17	2882	2852	1213
	6, 4, L		
0	4428	4588	582
1	2578	2628	693
2	2236	2299	772
3	2500	2639	748
4	1978	2047	847
5	3205	3151	650
6	1576	1601	1068
7	4346	4412	648
8	937	926	1099
11	3362	3416	845
13	3524	3448	847
	6, 5, L		
0	2381	2549	762
1	2090	2118	817
2	2180	2102	772
3	4138	4320	604
4	1147	1090	1300
5	2476	2576	783
8	2769	2844	807
9	2306	2291	934
10	1959	1934	1134
11	4780	4820	719
15	4065	4068	859
16	1904	1749	1423
17	1183	1312	1764
19	1208	1144	2183
	6, 6, L		
0	4223	4317	590
2	1962	2105	875
4	1094	1015	1249
6	4608	4721	619
9	1328	1688	1531
10	3016	3052	874
14	2766	2700	1117
15	1423	1290	1617

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	6,	6, L	
18	1511	1405	2080
	6,	7, L	
0	3273	3329	682
1	3633	3762	626
2	2432	2467	746
3	1414	1521	1123
4	4115	4141	609
5	1135	1183	1419
6	1952	1974	992
8	3607	3741	743
9	1708	1792	1156
10	2699	2709	912
12	1303	1368	1649
13	1541	1306	1432
16	3407	3337	1068
	6,	8, L	
0	3152	3159	650
1	1339	1389	1186
2	1983	1930	887
3	1116	1151	1392
4	1866	1915	945
5	1092	1095	1488
6	1000	1045	1402
7	2652	2710	860
8	4161	4273	739
11	2672	2608	961
13	2559	2726	1125
16	1753	1881	1658
	6,	9, L	
0	2390	2281	769
2	5870	6006	600
3	2389	2383	801
5	1213	1150	1401
6	1212	990	1381
7	1365	1145	1338
8	995	1059	1177
9	2486	2582	983
10	981	1031	1515
11	3410	3474	886
12	1508	1592	1577
13	2572	2727	1120
15	1262	1347	1319
16	1489	1458	1931
	6,	10, L	
0	1602	1627	1079
1	3266	3340	737
2	2275	2500	955
3	1214	918	873
4	2108	2177	972
6	1406	1619	1408
7	2947	3008	854
8	1374	1411	1528
9	2329	2467	1016
15	1464	1750	2049
17	1384	1131	2062
	6,	11, L	
0	1471	1583	1197
1	1434	1333	1298
3	3620	3648	756
5	1372	1359	1384
6	3010	3128	890
7	1613	1770	1351
8	1567	1332	1271
9	1249	905	1688
12	1179	1101	1461
14	2216	2343	1349
	6,	12, L	
0	3287	3150	784
1	1268	991	1425
3	1490	1515	1347
5	2066	2158	1110
8	2529	2547	1041
9	2128	2169	1156
15	1430	1488	2075
	6,	13, L	
0	3315	3437	852
1	1806	1626	1165
2	2677	2538	913
4	1113	961	1661
5	1685	1778	1348
7	1250	1294	1792
8	1225	1140	1640
13	2170	2341	1520
15	1487	1400	1289
	6,	14, L	
2	3462	3482	865
4	1335	1287	1349
6	1220	1061	1434
7	1351	1037	1552
8	1310	1318	1541
10	1429	1052	1768
	6,	15, L	
1	2828	2685	959
2	1664	1698	1388
3	1206	835	1754
5	2748	2737	1084
7	1421	1667	1786
	6,	16, L	
1	1315	904	1714
2	1308	1469	1886
3	1802	1911	1404
5	2272	2328	1233
8	1211	1102	1429
9	1367	1394	1997
	6,	17, L	
0	4659	4482	881
4	2199	2158	1291
	6,	18, L	
1	1375	1407	1821
2	1468	1374	1755
	6,	19, L	
0	1729	1600	1738
1	1546	1266	1837
6	1998	1755	1507
	7,	0, L	
2	3666	3692	760
4	7735	7522	656
6	4205	4275	761
10	2296	2387	1135
12	3834	3792	923
14	1630	1330	1584
	7,	1, L	
1	3887	3696	749
3	4921	4835	678
4	5637	5731	651
7	1832	1704	1135
8	2851	2923	891
10	2132	2246	1141
18	1626	1658	1928
	7,	2, L	
1	1717	1718	1154
2	1406	1316	1281
3	2260	2252	911
5	5867	5888	618
8	1979	2035	1074
9	2281	2391	1081
10	2786	2847	938
11	1671	1659	1351
12	1516	1569	1597
13	1628	1516	1510
15	1380	1534	1934
18	1587	1586	1940
19	1405	1110	2101
	7,	3, L	
1	3616	3591	687
3	2963	2956	732
4	1108	1048	1487
5	2259	2293	861
6	2086	2080	924
9	4221	4242	740
10	4272	4339	751
11	1467	1332	1488
14	1860	2022	1445
15	1725	1665	1522
16	1705	1730	1714
17	1726	1509	1696
	7,	4, L	
1	3614	3613	690
2	895	599	1100
4	852	851	1688
6	1063	1079	1580

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	7, 4, L		
8	2304	2233	916
9	4405	4553	724
10	1624	1358	1273
11	3306	3424	910
12	2532	2493	1023
13	1925	1809	1282
14	868	910	1734
15	1393	1169	1182
17	1674	1579	1819
19	1700	1839	1920
	7, 5, L		
1	1427	1480	1159
2	2434	2392	783
3	1624	1466	981
4	2571	2501	806
5	4045	4044	658
6	1297	1181	1293
8	1067	1042	1596
9	5482	5524	686
10	2336	2373	1049
11	1402	1071	1450
15	1504	1295	1705
17	2239	2056	1382
	7, 6, L		
2	3212	3357	725
3	3441	3507	687
5	1400	1539	1256
6	2602	2637	808
7	2131	2201	970
8	2603	2673	905
9	1095	966	1580
10	3104	3087	924
12	2216	2249	1160
14	865	971	1627
18	2412	2347	1406
	7, 7, L		
2	889	827	1100
3	2277	2439	883
4	2811	2866	764
5	1315	1159	1312
6	1742	1806	1085
7	2548	2482	883
10	4935	4965	759
12	2047	2117	1282
14	2972	2997	1040
	7, 8, L		
1	4103	4176	650
2	2368	2428	866
4	6054	6046	621
5	2700	2894	838
6	2172	2503	983
7	1114	1067	1193
8	1583	1441	1220
12	971	1078	1635
	7, 9, L		
2	1069	892	1581
4	3813	3820	706
5	2556	2780	934
6	1274	1309	1483
7	2907	2910	869
8	2325	2252	1023
9	2338	2303	1046
11	1184	1174	1708
15	1988	1820	1313
16	1192	1006	1346
17	1251	1179	1960
	7, 10, L		
2	1468	1551	1227
3	3488	3693	783
5	3468	3357	749
6	2567	2501	901
10	2172	2180	1122
11	1327	1402	1766
13	1185	1292	1940
14	1167	1180	1388
	7, 11, L		
1	2472	2585	944
3	2956	3140	907
8	2496	2549	1039
9	1611	1620	1460
12	3105	3094	1048
	7, 12, L		
3	1589	1471	1269
4	2313	2418	986
5	1377	996	1407
9	2430	2444	1074
11	1518	1732	1661
12	2196	2259	1348
13	1035	1010	1915
	7, 13, L		
2	1859	1817	1170
4	2403	2662	1078
6	2140	2244	1174
7	1792	1722	1324
8	1447	1391	1523
9	1347	1344	1773
11	2081	1967	1362
	7, 14, L		
2	1394	1365	1584
7	1980	1857	1262
10	2715	2896	1193
11	1163	1305	1620
	7, 15, L		
1	3171	3326	984
3	1259	1065	1622
5	1831	1834	1364
7	1209	1312	1864
10	1399	1414	1185
	7, 16, L		
1	1275	1178	1126
3	2336	2317	1224
4	1614	1743	1636
7	1756	2058	1678
8	1125	1342	2047
9	1517	1226	1863
	7, 17, L		
2	1721	1837	1647
6	2163	2436	1517
7	1638	1240	1458
	7, 18, L		
2	1757	1445	1526
3	2063	2056	1466
4	1366	950	1960
	7, 19, L		
1	1301	1212	1923
	8, 0, L		
0	6764	6768	636
2	7074	7055	647
4	4061	3964	825
6	4492	4460	804
8	824	886	1610
10	4822	4828	845
14	1452	1504	1936
16	2676	2671	1362
	8, 1, L		
1	1833	1754	1219
2	2211	2257	1116
4	1895	1899	1223
5	1590	1630	1306
6	1379	1243	1526
8	2173	2063	1095
9	2291	2482	1169
11	1848	1827	1402
15	1723	1748	1726
	8, 2, L		
0	2195	2139	1130
1	3531	3474	802
2	3147	3148	849
3	5313	5349	686
4	1466	1640	1425
5	1570	1675	1338
6	1281	1171	1504
7	1964	1914	1111
8	2721	2895	942
11	4189	4091	816
12	2131	1991	1171
13	1851	1929	1466
14	1771	1609	1441
15	1450	1285	1876
16	2913	3075	1281
	8, 3, L		

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$.
Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	8, 3, L		
0	1956	1986	1155
2	4439	4482	687
4	3307	3316	787
6	1489	1539	1293
7	1324	1154	1443
8	3033	3086	866
9	1248	1204	1501
11	1664	1354	1457
13	1247	1186	1623
	8, 4, L		
1	1036	1070	1475
3	5158	5245	647
4	1343	1105	1281
6	2591	2580	900
7	3972	4133	754
9	1900	2011	1236
10	1263	1195	1706
11	2643	2734	1065
13	1792	1775	1455
14	1539	1750	1801
15	3804	3724	951
17	3528	3425	1138
	8, 5, L		
2	1714	1577	1084
3	1625	1584	1159
5	2327	2337	900
6	1064	1061	1317
7	1212	1204	1614
9	2381	2333	1027
11	1875	1781	1301
13	882	911	1667
15	1886	1886	1515
	8, 6, L		
0	4635	4814	671
1	2978	3023	769
2	2751	2766	821
3	3662	3700	714
4	792	754	1357
6	1525	1509	1282
8	4352	4288	753
9	1498	1438	1401
12	1441	1494	1466
13	1467	1394	1202
16	3806	3676	1009
17	1590	1455	1806
	8, 7, L		
0	3356	3448	761
1	1750	1715	1098
2	2824	2976	823
3	802	595	1230
4	2074	2228	1010
5	1387	1484	1425
6	1452	1367	1321
7	1280	1218	1529
	8, 8, L		
0	3998	4012	722
1	3530	3500	752
2	6121	6447	649
3	1201	1324	1621
4	2190	2125	968
5	2177	2303	1048
6	2011	1965	1064
7	2666	2902	986
10	2516	2648	1089
16	1455	1399	1999
	8, 9, L		
1	1491	1243	1185
2	2139	2293	1043
3	1435	1517	1433
4	1639	1747	1243
5	1408	1319	1428
7	2132	1994	1068
8	1902	1910	1212
9	1051	1169	1476
12	1208	278	1742
13	1526	1423	1711
15	1367	1212	2033
	8, 10, L		
0	3399	3564	779
1	2041	2063	1060
3	4630	4706	730
4	2558	2503	980
5	2751	2727	917
9	1192	1052	1350
11	2172	2269	1298
13	1367	1517	1978
14	1597	1575	1797
16	1411	1426	1967
	8, 11, L		
0	2227	2385	1096
2	1383	1155	1380
3	1034	779	1239
9	1474	1788	1718
11	1405	1379	1161
15	1361	1072	1592
	8, 12, L		
2	1850	1792	1131
3	1197	1397	1667
5	1479	1708	1514
6	2340	2449	1119
9	1416	1560	1752
10	1399	1554	1628
11	2186	2104	1374
14	2108	2325	1631
	8, 13, L		
0	1375	1381	1579
2	1064	1014	1259
4	1313	1377	1714
6	1703	1636	1396
7	1437	1372	1545
	8, 14, L		
0	1920	1828	1262
1	1701	1621	1399
4	1066	1342	1767
5	1843	1900	1369
8	2903	3097	1142
	8, 15, L		
7	1592	1735	1791
9	1341	1153	1759
11	1466	1581	2007
	8, 16, L		
0	1739	1738	1623
1	2854	2850	1131
2	2315	2308	1301
7	1614	1392	1286
9	2298	2129	1363
	8, 17, L		
0	1961	1896	1435
6	1944	1730	1457
	8, 18, L		
0	2927	2747	1197
2	1247	1187	1986
5	1030	1155	1804
6	1152	1031	1594
	9, 0, L		
2	3589	3590	803
4	5008	5037	784
6	4188	4070	893
8	1493	1506	1769
12	1119	1296	1604
	9, 1, L		
2	1247	1114	1663
3	2691	2608	1023
4	3078	2988	945
5	2096	2067	1252
7	2028	2022	1263
9	3538	3615	947
10	5154	5071	851
12	1651	1608	1636
14	2097	2413	1640
17	1751	1832	1923
	9, 2, L		
1	2126	2080	1166
3	2078	2162	1202
5	4230	4384	796
7	1024	1006	1209
8	2057	2085	1246
10	2521	2639	1152
12	1931	1800	1396
	9, 3, L		
1	1744	1449	1255

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	9, 3, L		
3	1497	1482	1468
4	3827	3904	804
5	3845	3984	831
7	1988	1905	1151
8	2455	2555	1105
9	4136	4181	844
10	2200	2213	1203
12	1311	1296	1887
14	1888	1750	1461
15	2077	2074	1560
16	2189	1923	1445
17	1217	1409	2038
	9, 4, L		
1	1754	2008	1339
2	1162	1270	1702
6	955	1012	1891
7	1570	1730	1458
8	1196	1083	1429
9	3184	3182	943
11	3320	3289	965
12	1325	1368	1194
13	2249	2056	1158
17	1183	942	2005
	9, 5, L		
1	4027	4018	739
2	1199	1159	1623
3	5997	6183	674
4	3959	4011	746
7	1988	2014	1111
9	1391	1359	1499
10	1145	1146	1314
12	2070	2061	1358
14	1290	1077	1877
	9, 6, L		
2	1941	1828	1048
3	2110	2182	1040
5	1938	1767	1052
6	1048	1099	1675
7	2103	1938	1022
8	1778	1784	1253
9	1190	1193	1207
10	2187	2164	1155
12	2227	2245	1234
14	1151	898	1658
	9, 7, L		
1	1364	1214	1418
2	1970	2095	1123
3	1714	1788	1228
4	5025	5227	714
6	1543	1796	1470
8	2896	3029	940
9	1697	1540	1337
11	1812	1923	1433
16	1846	1511	1640
	9, 8, L		
1	1957	1886	1113
2	1782	2059	1259
4	3613	3866	807
5	2053	2089	1143
6	2404	2476	995
8	1800	1778	1285
	9, 9, L		
1	4383	4500	761
3	2459	2454	1002
5	1099	652	1493
6	1596	1521	1395
8	1309	994	1585
9	1288	1591	1891
10	3313	3480	982
14	1494	1467	2005
	9, 10, L		
1	1002	830	1724
3	2448	2642	1054
5	2139	2443	1204
6	1837	1959	1328
7	918	962	1468
8	1217	696	1778
10	2152	2059	1227
11	1152	1206	1963
	9, 11, L		
2	2368	2450	1061
3	890	776	1324
5	2300	2281	1125
6	2893	2902	979
7	1704	1496	1359
9	2794	2940	1063
	9, 12, L		
3	878	771	1445
4	1235	1330	1844
9	2181	2305	1252
10	1531	1323	1821
11	2036	1914	1457
12	1231	1327	2046
	9, 13, L		
1	1845	1698	1291
2	860	856	1577
4	1323	1260	1266
5	2558	2395	1076
7	1118	1255	1301
8	1489	1298	1763
10	2142	2310	1440
12	2724	2646	1306
	9, 14, L		
2	1330	1146	1659
9	1534	1252	1744
10	2141	2013	1400
11	1271	1142	1359
	9, 15, L		
4	1549	1600	1782
5	1842	1665	1480
7	3222	3107	1098
	9, 16, L		
3	1903	1788	1460
4	1518	1572	1826
5	1386	1161	2018
7	1264	1177	1807
	9, 17, L		
1	1486	1375	1871
	10, 0, L		
0	6093	6037	698
8	4456	4371	983
16	2513	2254	1485
	10, 1, L		
0	2466	2384	1001
2	985	1116	1886
4	4168	4060	890
5	1978	2056	1453
8	2079	2263	1418
9	2393	2419	1290
10	1639	1633	1719
11	2852	2610	1124
12	1381	1583	1966
14	1497	1434	1909
15	2831	2851	1306
16	2602	2855	1480
	10, 2, L		
1	3455	3455	917
2	1626	1438	1470
3	1811	1827	1398
5	1317	1683	1959
6	2753	2671	1074
7	2528	2643	1175
9	2088	2197	1350
10	1434	1219	1791
11	917	912	1789
14	1714	1679	1759
15	1493	1075	1978
17	1234	1265	1843
	10, 3, L		
1	2467	2544	1143
2	5421	5555	800
3	3573	3511	918
4	1515	1642	1593
8	2232	2394	1254
11	2819	2576	1021
15	2550	2369	1279
17	1381	1107	1729
	10, 4, L		
0	2930	2773	1011
3	2870	2856	980
5	3451	3442	882

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	10, 4, L		
7	1684	1773	1411
8	1205	1365	1905
11	1733	1867	1512
13	2090	2052	1449
	10, 5, L		
0	1172	1078	1318
1	2659	2795	1048
2	2167	2057	1126
3	4620	4783	775
5	1302	1410	1741
6	1495	1480	1512
7	1722	1787	1362
9	2723	2749	1065
10	1100	1107	1644
13	2334	2274	1271
14	973	649	1870
	10, 6, L		
2	2398	2366	1054
6	3361	3331	886
9	1362	1201	1622
10	2061	2370	1358
14	2412	2375	1345
	10, 7, L		
0	1869	1621	1170
2	4929	5242	772
5	2385	2493	1069
6	1481	1612	1 .7
11	1075	1121	1485
12	1254	1276	1929
13	1336	1228	1779
15	1867	1915	1721
16	1646	1407	1930
	10, 8, L		
0	3387	3609	909
3	1429	1499	1551
5	1507	1338	1459
8	2830	2920	1007
11	1463	1756	1811
13	1630	1592	1765
	10, 9, L		
0	1713	1839	1367
3	1531	1775	1449
4	2613	2715	1010
5	1735	1532	1280
6	789	751	1570
7	2416	2374	1054
8	1915	1859	1349
14	2088	1907	1469
	10, 10, L		
1	2451	2540	1072
7	1962	1809	1294
9	2408	2370	1166
11	1324	1209	1890

L	KF_o	F_c	σ
	10, 11, L		
0	4156	4160	840
3	1285	1455	1835
4	2061	2061	1248
9	1342	1461	1738
11	1789	2181	1769
	10, 12, L		
0	2829	2926	1061
5	1504	1602	1646
8	2847	2773	1154
9	1015	1069	1957
	10, 13, L		
0	1555	1422	1052
1	2923	3014	1096
3	1658	1644	1515
4	1612	1633	1559
6	2113	2094	1369
7	1103	1144	1631
8	1662	1525	1638
10	1478	952	1605
11	1517	1308	1892
	10, 14, L		
2	2038	2040	1373
3	976	995	1790
5	981	806	1606
6	1795	1820	1540
7	1189	914	1349
	10, 15, L		
2	1371	1461	1952
5	1481	745	1612
7	1535	1591	1967
	10, 16, L		
3	1731	1683	1583
5	1977	1815	1510
	10, 17, L		
2	1589	1681	1303
	11, 0, L		
2	2813	2705	1020
4	1376	1208	1597
6	1449	1232	1813
8	2210	1975	1423
10	3801	3733	1137
14	2181	1875	1585
	11, 1, L		
1	1867	2015	1318
2	2312	2282	1144
3	2589	2829	1196
4	1569	1691	1662
6	3317	3144	1017
9	1746	1569	1623
10	2512	2332	1319
11	1661	1477	1718
12	1527	1292	1849
13	1496	1171	1876

L	KF_o	F_c	σ
16	1231	1129	2238
	11, 2, L		
1	2635	2528	1050
2	1127	1010	1953
4	3324	3500	1090
6	1669	1496	1630
9	3167	3118	1117
11	1097	1223	1913
15	1261	1026	1771
	11, 3, L		
1	3394	3336	1019
4	3203	3269	1056
5	1654	1654	1581
7	2558	2641	1193
8	2050	2213	1378
10	2302	2516	1344
12	1379	1358	2025
13	1268	1290	2071
16	2029	1714	1656
	11, 4, L		
2	1385	1255	1802
3	4005	4053	887
5	4512	4520	869
6	1441	1526	1727
9	1871	1857	1422
15	1143	1149	1788
	11, 5, L		
1	1299	873	1815
2	2153	2139	1228
3	4391	4333	842
6	1617	1558	1447
7	3645	3698	952
10	1519	1440	1722
	11, 6, L		
4	4104	4148	848
9	1567	1602	1585
11	1524	1170	1519
12	2163	2057	1392
	11, 7, L		
2	1373	1289	1252
8	3031	2982	1019
9	2168	2166	1320
10	1750	1620	1480
12	3144	3124	1143
14	1462	1490	1797
	11, 8, L		
3	1162	941	1619
5	1425	1209	1561
10	2569	2633	1188
	11, 9, L		
5	2410	2254	1138
6	1353	1567	1860
7	2709	2632	1085
9	1256	1178	1500

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	11, 9, L		
10	2571	2644	1248
	11, 10, L		
1	1703	1785	1416
7	1435	1148	1385
9	2148	2416	1437
11	1362	1223	1230
	11, 11, L		
1	1940	2203	1442
3	1451	1457	1613
4	2286	2225	1160
5	1312	1444	1896
10	1176	1333	2152
12	1662	1515	1781
	11, 12, L		
2	2082	2240	1396
3	2139	2182	1291
5	2172	2130	1307
6	2059	1993	1431
9	1235	856	1828
10	1742	1308	1593
	11, 13, L		
1	1207	1248	1449
2	1361	1277	1790
4	1569	1544	1811
5	2502	2504	1233
6	1469	1657	1978
8	1219	1147	1856
	11, 14, L		
4	2654	2773	1194
7	1739	1571	1444
	11, 15, L		
1	2402	2462	1388
3	1739	1713	1633
6	1486	1188	1854
	12, 0, L		
0	1429	1528	1668
4	2084	2204	1328
6	2679	2730	1223
8	1390	1334	1979
12	1278	1222	2265
14	3555	3279	1232
	12, 1, L		
0	2771	2837	1056
2	2862	2863	1066
3	1867	2074	1482
4	1438	1377	1689
5	2805	2805	1172
6	1512	1163	1857
9	2316	2095	1320
11	1420	771	1893
15	1756	1575	1283
	12, 2, L		
0	3529	3694	986
2	2232	2404	1318
4	1253	1129	1382
5	2470	2268	1212
7	1841	1667	1644
8	1647	1505	1030
13	1851	1771	1669
15	2147	2040	1686
	12, 3, L		
1	1635	1460	1602
2	2944	2919	1139
3	2209	2111	1443
4	2145	2018	1322
5	2681	2338	1152
7	1448	1689	2001
8	2492	2339	1270
	12, 4, L		
1	4640	4589	957
2	1401	1119	1869
3	2229	2081	1331
7	1188	910	1360
9	2555	2322	1159
	12, 5, L		
0	3326	3312	1066
1	1504	1353	1550
8	2064	2121	1410
9	2857	2982	1213
11	2575	2752	1303
13	2058	1903	1517
15	1857	1915	1948
	12, 6, L		
0	2744	2901	1195
1	1324	1123	1974
2	1833	1831	1419
5	1200	1257	1887
7	1281	953	1898
8	2144	2187	1373
13	1590	1398	1841
	12, 7, L		
1	1434	1276	1675
2	875	1040	1651
4	2053	1826	1261
6	2666	2794	1140
8	2984	2922	1121
9	1400	1614	2047
10	1571	1443	1742
	12, 8, L		
0	1188	934	1801
4	1581	1241	1491
6	1678	1937	1667
12	1053	1102	1871
	12, 9, L		
2	1561	1714	1587
3	1309	1265	1459
4	1359	1065	1762
5	883	1078	1690
11	2001	2177	1579
13	2389	2272	1379
	12, 10, L		
2	1685	1546	1522
4	1712	1303	1507
5	1532	1783	1755
7	1450	1359	1806
	12, 11, L		
0	1687	1457	1510
3	1510	1548	1718
5	1841	1976	1527
6	2447	2499	1260
	12, 12, L		
0	1359	1263	1919
1	2598	2546	1184
2	2024	1906	1469
3	1420	1728	2073
10	1456	986	1859
	12, 13, L		
0	3323	3460	1081
1	1296	1148	1974
2	2307	2447	1415
4	1220	1181	1362
7	1381	1346	2011
	12, 14, L		
0	1857	2073	1740
1	1489	1339	1765
3	1658	1594	1710
	12, 15, L		
3	1425	1484	2078
5	1583	1459	1783
	13, 0, L		
4	2841	2892	1166
6	1831	2126	1609
8	1430	1570	2033
10	1261	1299	2184
12	3651	3389	1201
	13, 1, L		
3	2976	3155	1159
4	2402	2327	1255
7	1518	1452	1888
8	2186	2065	1443
	13, 2, L		
1	2112	1977	1339
2	2026	2266	1445
3	1913	2027	1583
6	2758	2693	1178
7	1476	1687	2202
11	2155	1748	1403
13	2091	1969	1707
	13, 3, L		
3	1368	1273	1270
4	2095	1863	1357

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{13}(PF_3)$. Columns are $10F_o$, $10F_c$, 100σ. * denotes an insignifcant reflection.

L	KF_o	F_c	σ
	13, 3, L		
7	1709	1593	1788
10	3090	3183	1285
14	1569	1399	2133
	13, 4, L		
1	2315	2243	1291
3	2305	2309	1404
4	1733	1636	1766
5	1497	1447	1917
7	3929	4068	1062
	13, 5, L		
1	1693	1705	1765
3	1883	1987	1531
5	1859	1925	1529
9	2603	2417	1283
	13, 6, L		
2	2807	2784	1156
6	2799	2743	1162
7	1080	1329	2021
10	1738	1545	1584
12	1746	1187	1642
	13, 7, L		
3	1793	1893	1519
5	1365	1193	1905
6	2127	1800	1289
10	1749	2097	1905
	13, 8, L		
1	1393	1158	1778
4	1361	1524	1953
6	1640	1309	1627
7	1457	1486	1943
8	1636	1421	1690
9	1656	1748	1857
12	2475	2512	1429
	13, 9, L		
1	1752	1771	1580
2	1226	1115	1816
4	2114	2312	1512
5	1860	1761	1490
8	1735	1343	1457
	13, 10, L		
3	1536	1661	1828
7	1723	1331	1654
10	1409	1324	1574
	13, 11, L		
2	1317	942	1904
3	1841	1969	1560
7	1411	1241	2073
8	1626	1558	1131
	13, 12, L		
3	1052	1043	1806
4	2422	2721	1475
5	1862	1591	1583
7	1520	1435	1848

L	KF_o	F_c	σ
	13, 13, L		
1	1121	951	1514
7	1401	1092	2070
	13, 14, L		
2	1856	1715	1696
	14, 0, L		
2	2303	2373	1337
8	1510	1307	1922
	14, 1, L		
0	3032	3138	1054
1	2188	2133	1268
2	2225	2280	1344
6	2138	2083	1408
8	2444	2448	1486
9	2391	2180	1455
11	1514	1509	2166
	14, 2, L		
3	1334	973	1956
5	1405	1285	1748
8	1738	1414	1782
	14, 3, L		
1	2167	2034	1322
2	1889	1827	1458
4	2224	2339	1460
5	2206	2313	1484
8	2131	1811	1462
9	1384	1187	2198
11	1256	1014	2009
13	2933	2621	1422
	14, 4, L		
1	1209	1385	1963
2	1410	1055	1809
7	1477	1320	1783
	14, 5, L		
1	1207	1427	2227
5	3463	3478	1210
6	2125	1985	1529
9	1440	1317	2001
11	1482	1251	2110
	14, 6, L		
0	1375	1450	1863
8	2123	2108	1556
	14, 7, L		
0	4624	4572	1030
1	1547	1697	1958
2	1455	1469	2033
3	2068	1912	1518
4	2372	2328	1331
9	1217	1061	1708
	14, 8, L		
2	1405	993	1460
	14, 9, L		
0	2037	2157	1554
1	1454	1556	2170

L	KF_o	F_c	σ
2	2828	2689	1240
3	1414	1400	2070
5	1656	1615	1782
7	1766	1687	1690
8	1405	1422	1679
	14, 11, L		
0	1762	1840	1833
1	1856	1652	1692
2	1403	1674	2067
	15, 0, L		
2	1824	2142	1591
4	1174	1144	1409
6	2439	2623	1365
8	1522	1493	1939
12	1091	1163	1942
	15, 1, L		
2	1452	1573	1955
3	2696	2610	1212
6	1464	1491	1926
7	1568	1679	2083
8	1570	1462	1956
9	1148	1312	1643
12	1588	1616	2177
	15, 2, L		
3	1903	1799	1529
7	2232	2627	1621
	15, 3, L		
4	1760	1617	1688
7	2529	2310	1349
10	2272	2257	1674
11	1401	1248	1543
	15, 4, L		
1	1341	1337	1364
3	1464	1468	1851
7	1509	1177	2132
11	1506	1394	2253
	15, 5, L		
1	2397	2309	1339
3	2034	1813	1479
4	1519	1471	2211
9	1900	1459	1810
	15, 6, L		
4	2501	2163	1463
5	1737	1393	1890
	15, 7, L		
2	1485	1443	1569
3	1667	2084	2093
4	1453	1123	2162
5	1362	1365	2186
6	2409	2470	1502
	15, 8, L		
2	1721	1694	1971
3	1528	1678	2175
6	2087	2058	1630

Table J.2. Obs and Calcd Structure Factors for $Os_4(CO)_{15}(PF_3)$.
Columns are $10F_O$, $10F_C$, 100σ. * denotes an insignifcant reflection.

L	KF_O	F_C	σ
	15,	9,	L
1	1895	1953	1733
2	1477	1327	2042
4	1860	1754	1657
6	1702	1197	1777
	15,	10,	L
1	1267	1230	1535
	16,	0,	L
0	3217	3188	1135
2	1960	1962	1543
6	1827	1716	1655
8	2348	2487	1466
	16,	1,	L
2	1673	1614	1754
5	1450	1485	1920
9	1215	1137	1777
	16,	2,	L
0	1635	1547	1748
1	1923	2045	1624
6	1259	1205	1436
9	1843	1656	1779
	16,	3,	L
2	1978	1893	1605
4	1188	1246	1593
8	1548	1651	2159
	16,	4,	L
5	2515	2503	1344
8	1097	1131	1947
	16,	6,	L
0	1781	1892	1889
1	1567	1638	1955
2	1174	1597	2298
4	1294	1228	2227
6	1625	1439	2115
	16,	7,	L
4	1028	1142	1953
7	1184	977	1856
	16,	8,	L
0	3081	3121	1377
3	1497	1461	1761
	16,	9,	L
2	1273	1158	1952
	16,	10,	L
1	1799	1516	1817
2	1893	1652	1827
	17,	0,	L
4	2135	2000	1533
	17,	1,	L
3	1436	1770	1887
4	1768	1404	1686
6	1837	1504	1677
	17,	2,	L
7	1045	1345	2003
	17,	3,	L
1	1523	1729	2055
4	1714	1594	1859
7	1534	1619	2206
	17,	5,	L
2	1547	1524	2134
3	1712	1847	1947
	18,	0,	L
0	1484	1503	2009
2	2352	2092	1375
	18,	2,	L
3	1790	1476	1762
	18,	3,	L
0	1193	930	1819
1	1489	1392	2111
2	1887	1774	1730