SYNTHESIS AND STRUCTURAL STUDIES OF TRANSITION METAL ALKYNE AND DIAZENE COMPOUNDS

by

Kenneth George Tyers

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M.Sc., University of Western Ontario, 1980

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Name: Kenneth G. Tyers

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Examining Committee:

Chairperson: Dr. T.N. Bell

D. Sutton, Co-Senior Supervisor

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

K.N. Slessor

L.K. Peterson
Internal Examiner

Dr. Peter Legzdins, Professor
Department of Chemistry, University of British Columbia
External Examiner

Date Approved: 1985.11.26

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Author:

(signature)

Kenneth G Tyers

(name)

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ABSTRACT

A series of new heterotrimetallic alkyne clusters of general formula $CpNiCoM(CO)_6(RC_2R')$ (M = Fe, Ru, Os: R = R' = Ph, Et; R = Ph, R' = Me) has been synthesized and the compounds characterized by their infrared, NMR and mass spectral data. In addition, four of the clusters had their solid state structures examined by X-ray diffraction. In all cases examined, the alkyne is oriented parallel to one edge of the trimetallic framework but the particular location varies. In the iron complex $CpNiCoFe(CO)_6(C_2Et_2)$, it is parallel to the Ni-Co edge and all carbonyls are terminal. In the ruthenium and osmium complexes $CpNiCoRu(CO)_6(C_2Ph_2)$ and $CpNiCoOs(CO)_6(C_2Ph_2)$, the orientation is parallel to the Ni-Ru or Ni-Os edge and one of the CO groups adopts a semi-bridging position across the Co-Ru or Co-Os bond. A similar arrangement is observed in an iron complex when one of the carbonyl groups bound to cobalt is replaced by triphenylphosphine to give $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$. The alkyne ligand was shown to be fluxional in solution and to involve rotation on only one face of the heterotrimetallic core.

In Chapter 2, the X-ray structure of $CoMn(CO)_6(MeC_2Ph)_2$, a heterobimetallic 'ferrole' compound, is reported. The structure consists of a substituted cobaltacyclopentadienyl ring which is bound to the $Mn(CO)_3$ fragment by a metal-metal bond and by 4 electron π-bonding. Other significant structural differences are described.

Chapter 3 reports a study of the η^2-coordination of some two-electron donors in mononuclear cyclopentadienyl rhenium compounds. The X-ray structures of $CpRe(CO)_2(C_2Ph_2)$ and $CpRe(CO)_2(N_2Ph_2)$ reveal an overall 'four legged piano stool' geometry with η^2 coordination of both the C_2Ph_2 and

N_2Ph_2 ligand. The C_2Ph_2 group is bound symmetrically to the rhenium but the N_2Ph_2 ligand is not. In solution, $CpRe(CO)_2(C_2Ph_2)$ retains its geometry, but for $CpRe(CO)_2(N_2Ph_2)$ IR and NMR data revealed the presence of isomers which are in dynamic equilibrium and involve both η^2- and η^1-bound N_2Ph_2 with the η^1 form being favoured in solution.

In Chapter 4, the X-ray structures of cis-$Cp^*Re(CO)_2I_2$ and trans-$Cp^*Re(CO)_2Br_2$ were determined. The rhenium is seven coordinate and the overall geometry is that of a typical Cp^*ML_4 complex. The structure of cis-$Cp^*Re(CO)_2I_2$ is slightly disordered limiting the precision of the observed bond lengths and angles.

QUOTATION

William James used to preach the
"will to believe."
For my part, I should wish
to preach the "will to doubt."...
What is not wanted is not the will to believe
but the wish to find out, which is
the exact opposite.

BERTRAND RUSSELL
Skeptical Essays, 1928

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LIST OF ABBREVIATIONS

br = broad

m = medium

q = quartet

s = strong

sh = sharp

t = triplet

Cp = η^5-C_5H_5

Cp* = η^5-C_5Me_5

Et = C_2H_5

Me = CH_3

IR = infrared

NMR = nuclear magnetic resonance

GOF = goodness of fit

NOE = nuclear overhauser enhancement

THF = tetrahydrofuran

LIST OF NUMBERED COMPOUNDS

$CpNiCoFe(CO)_6(C_2Ph_2)$ (1)

$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

$CpNiCoRu(CO)_6(C_2Ph_2)$ (2)

$CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

$CpNiCoFe(CO)_6(C_2Et_2)$ (4)

$CpNiCoRu(CO)_6(C_2Et_2)$ (5)

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$CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15)

cis-$Cp^*Re(CO)_2I_2$ (16)

trans-$Cp^*Re(CO)_2Br_2$ (17)

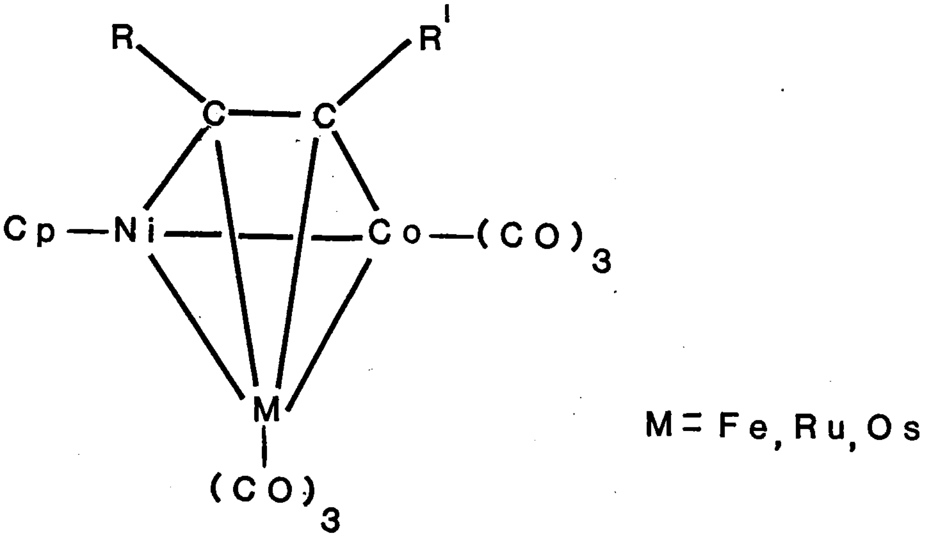
$Os_3(CO)_{10}Cl_2$ (18)

CHAPTER 1

HETEROTRIMETALLIC ALKYNE CLUSTERS

1.1 Introduction

In this chapter the synthesis, structural characterization and dynamic behaviour in solutions of a series of heterotrimetallic (MM'M") alkyne clusters[a] having the general composition shown in I-1 are detailed, where R and R' are substituents on the alkyne. The structure illustrated is representative but modifications of it are displayed by many of the examples as will be discussed.



I-1

Increasing interest in cluster compounds and their derivatives has led to many recent reviews on various aspects of their bonding[1], ligand

---

[a] The term heterotrimetallic is ambiguous since it may be used to describe clusters with three different metals, such as MM'M", as well as those with only two different metals, designated here as M_2M'.

fluxionality[2] and preparation[3,4]. Only 15 years ago the number of well defined metal clusters was small, but today many hundreds of clusters are known and a large fraction of these have been structurally characterized by X-ray crystallographic studies. One of the main reasons for the interest in this field is based on the premise that metal clusters may be viewed as models for the chemisorption of small molecules on metal surfaces[5,6], possibly during catalysis. The nature of the bonding of molecules on metal surfaces in not well understood at the present time. Analogies that might be drawn with homogeneous cluster species would be extremely beneficial since the details of structure and dynamic behaviour are more easily established for cluster chemistry than for surface chemistry. Recent assessments of the metal cluster to metal surface analogy have suggested a range of validity which improves as the cluster nuclearity increases[7-9]. A separate and distinct view is that cluster compounds may serve as incipient metal particles that may provide active aggregates of uniform size and composition when spread over a suitable support and the sheath of ligands stripped off. This is referred to as supported homogeneous catalysis[10] and is starting to receive an increasing amount of attention[11].

Cluster compounds may serve as catalyst precursors in their own right rather than simply mimicking metal surfaces. Metal cluster chemistry provides a connection between the traditionally separate areas of homogeneous and heterogeneous catalysis and may offer advantages over both areas. To this end, a great deal of effort is being directed towards the use of clusters as catalysts for such varied reactions as hydroformylation[12,13], isomerization of alkenes and alkynes[13,14], hydrogen reduction of carbon monoxide[15], oxidation of CO to CO_2[16] and many more.

In this context it is of interest to establish how simple organic molecules interact with a multimetal system and this is partly the thrust of the present study which specifically probes the bonding of alkynes. An increasingly popular and important subset of cluster chemistry comprises those clusters that contain a mixture of different transition metals in their framework and this is a second aspect of this study in that it specifically utilizes triangular clusters comprising three different metals. In particular, because of the varying reactivities of the different metals present in mixed-metal clusters, multimetal homogeneous catalysts may show reactivity patterns significantly altered from those of homometallic clusters. Of course, the bonding of various organic molecules to these heterometallic clusters may be different than to the analogous homometallic clusters also, but a very useful consequence of using heterometallic clusters is their low symmetry. This allows the dynamics (H, CO or other ligand migrations) of the cluster or cluster derivatives to be probed by IR, ^{1}H or ^{13}C NMR, in greater detail than is often possible for homometallic clusters. This is a third aspect that we shall address in this study.

In order to set the research in context a brief survey of clusters and cluster alkynes in particular is provided in the next few sections.

1.1.1 Clusters

Metal clusters are molecules or molecular ions that contain three or more metals bonded to each other in such a way that they form a triangular or polyhedral array. We shall only be concerned with transition metal-based clusters and will merely reference the reader to the subgroup of post-transition metal cluster naked ions[18] like Bi_5^{3-} and Pb_9^{4-}, and to the cubane-like M_4S_4 and M_4X_4 clusters used as models for ferredoxins[19,20]. The framework of the transition metal cluster is surrounded and stabilized by various ligands, commonly carbonyls, hydrides, phosphines and halides but may also include alkenes, alkynes, isonitriles, sulphur, arenes, nitrosyls and so on.

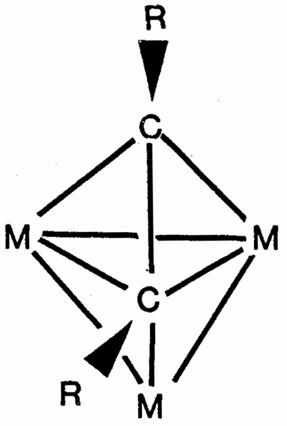
Clusters were originally synthesized by serendipitous methods and seldom by rational synthetic procedures. Gradually, as more was learned about clusters, designed synthetic strategies began to appear, but fortune still plays its role in the development of clusters and their chemistry. The particular methods used so far to synthesize homo- and heterometallic clusters have been well documented[3,4,21,22] and will not be reiterated here.

1.2 Cluster Alkynes

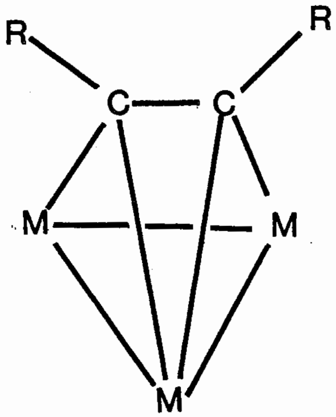
At the time work on this thesis began, a significant literature existed on the binding of alkynes to trimetallic and larger clusters. However, only one report of a heterotrimetallic (MM'M") alkyne cluster existed, which was the synthesis of $CpNiCoFe(CO)_6(C_2Ph_2)$[23], although it was incorrectly formulated at that time. Most of the workers previous[24] to our study have clearly focussed on the synthesis and characterization of M_2M' heterotrimetallic alkyne clusters although during this investigation reports were published of MM'M" heterotrimetallic alkyne clusters closely related to those discussed[25]. Specifically these two clusters are $Cp_2NiMoFe(CO)_5(PhC_2CO_2\underline{i}\text{-}Pr)$ and $CpNiCoFe(CO)_6(PhC_2CO_2\underline{i}\text{-}Pr)$.

1.2.1 Bonding and Structure

Alkynes interact with one metal atom in the classic σ-π bonding scheme described by Dewar[26] and Chatt and Duncanson[27]. This is briefly described in the introduction to Chapter 4. The bonding of an alkyne to a single metal atom may arise in the initial stages in the reaction of an alkyne with a metal cluster but typically the isolated products have the alkyne interacting with more than one metal atom. With trimetallic alkyne clusters the two basic structures that are adopted are shown below as I-2 and I-3.



I-2

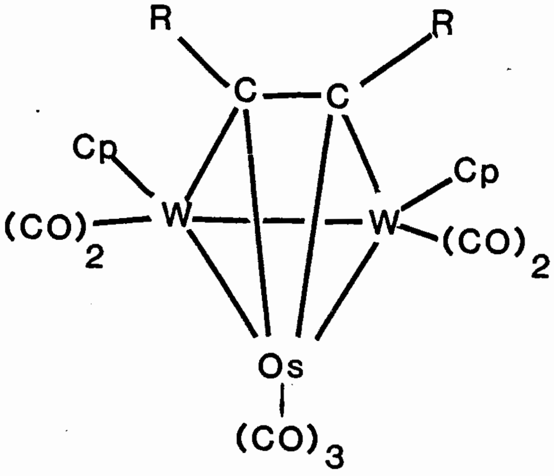


I-3

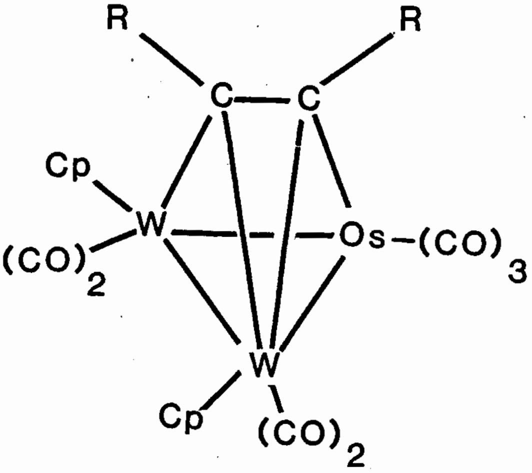
In structure I-2 the alkyne symmetrically bridges two metal atoms and the carbon of the alkyne which is above the M_3 triangle is within bonding distance of all three metal atoms. This combines features of a σ-bonded and a side-on or 'π' bonded ligand and has been designated ($\sigma + 2\pi$) in reference to classical bonding schemes[24]. In current nomenclature it is often (and properly) designated μ_3-(η^2-$\perp$), where the $\perp$ symbol refers to the perpendicular orientation of the alkyne C_2 vector with respect to the symmetrically bridged metal-metal bond. The alternative form I-3 has the alkyne C_2 vector above the metal plane and nearly parallel to the M_2 vector but displaced towards the third metal atom to such an extent that both alkyne carbons are within bonding distance. While structure I-3 can be fully described in classical bonding terms and the alkyne as a ($2\sigma + \pi$)

donor, it is properly designated as μ_3-(η^2-||).

The μ_3-(η^2-||) alkyne interaction is more widely encountered than the μ_3-(η^2-$\perp$) mode among homo- and heterometallic alkyne clusters, with 21 X-ray structures of the former listed in a recent review[24], compared with only 3 structures for the latter geometrical type. Specifically only $Fe_3(CO)_9(C_2Ph_2)$[28], $Cp_2W_2Fe(CO)_6(C_2(C_6H_4Me)_2)$[29] and $Fe_2Ru(CO)_9(C_2Ph_2)$[30] have been characterized as having a μ_3-(η^2-$\perp$) alkyne ligand. The μ_3-(η^2-||) alkyne types include $Cp_3Rh_3(CO)(C_2Ph_2)$[31] and $Cp_2W_2Os(CO)_7[C_2(C_6H_4Me)_2]$[32]. The latter alkyne cluster is of interest since it has two isomers in the asymmetric unit which differ principally by rotation of the alkyne ligand above the trimetallic face. These are shown in I-4 and I-5. Molecule I-4 is essentially the symmetric isomer where the alkyne ligand bridges two tungsten atoms and has a η^2-linkage to the osmium atom. I-5 has the alkyne ligand spanning the W-Os bond while having a η^2-linkage to the second W atom. These two isomers, observed in the same crystal, describe the two possible rotamers of alkyne bonding to a M_2M' system.

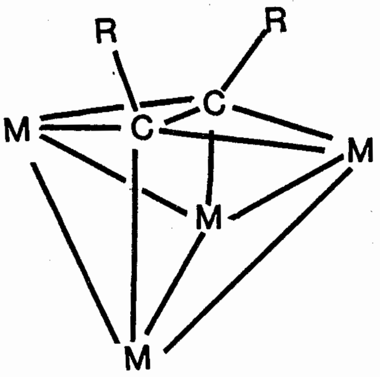


I-4

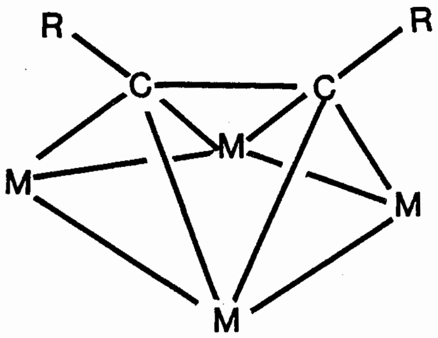


I-5

There are two types of alkyne-M_4 structures known, the butterfly and the square-planar structure. These are represented by I-6 and I-7. The arrangement of metal atoms and alkyne carbons in the butterfly structure essentially completes an irregular octahedral geometry while in the square planar form the alkyne lies above the almost planar array of metal atoms. Heterotetrametallic alkyne clusters are fewer in number than the homometallic analogues but $FeRu_3(CO)_{12}(C_2Ph_2)$ and $Cp_2Ni_2Fe_2(CO)_6(C_2Ph_2)$ serve as examples of butterfly and square planar arrangements respectively[33,34].



I-6



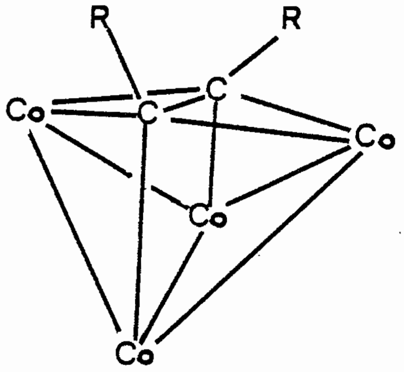
I-7

1.2.2 Synthesis of Cluster Alkynes

In reviewing this subject the synthetic strategies for the assembly of heterometallic alkyne clusters will be emphasized although the reactions are often general and could equally be used for the homometallic analogues. Indeed, many of the synthetic routes described were adopted or modified from methods used in the homometallic cluster alkyne field.

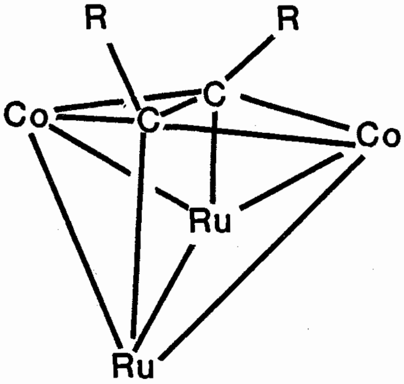
Substitution of CO

This is one of the more common methods used for the synthesis of both homo- or heterometallic alkyne clusters and certainly is the most obvious. The appropriate cluster precursor is first assembled and then the alkyne is added to the cluster with consequent loss of the correct number of CO groups. The heterotrimetallic (MMM') cluster $Co_2Ru(CO)_9(C_2R_2)$, can be synthesized in this fashion from $Co_2Ru(CO)_{11}$ and the desired alkyne ligand[35]. Rearrangement of the metal skeleton or breaking of metal-metal bonds is a possible consequence of this reaction since the alkyne fragment prefers to interact with more than one metal atom of a cluster. A well known example[36] is the reaction of $Co_4(CO)_{12}$ with an alkyne yielding $Co_4(CO)_{10}(C_2R_2)$ (I-8). Two CO ligands have been lost, a cobalt-cobalt bond has been severed and the alkyne inserted. A C_{2v} arrangement of the metal atoms or a butterfly structure is the end product.



I-8

An interesting heterometallic example is the reaction of $Co_2Ru_2(CO)_{13}$ with alkynes[37]. As in $Co_4(CO)_{12}$, the tetrahedral cluster loses two CO ligands and a butterfly cluster is obtained but exclusive formation of only one isomer (I-9) out of three possibilities is observed; specifically, that in which the Co-Co bond is cleaved and not the Ru-Co or the Ru-Ru bond.



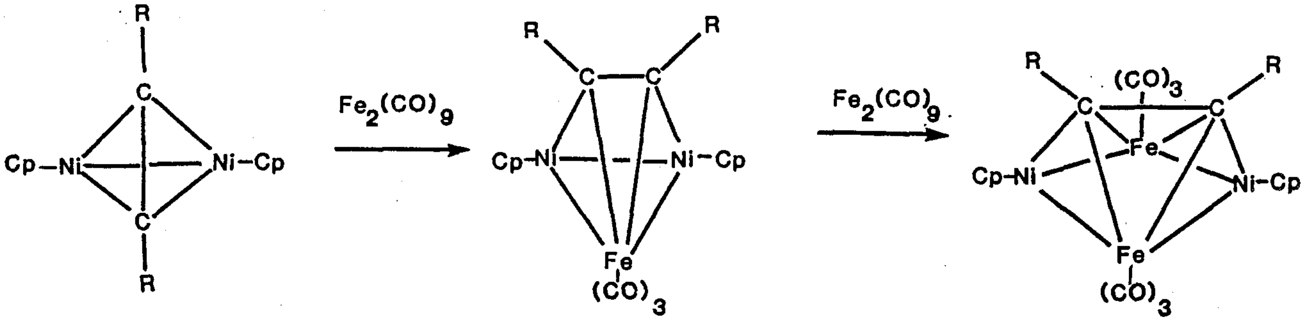
I-9

It should be noted here that, while this reaction may appear to be general, there are tetrahedral clusters which will not add alkynes in a similar fashion. $Cp_2Ni_2Fe_2(CO)_7$ has not been found to add an alkyne with loss of a carbonyl to form $Cp_2Ni_2Fe_2(CO)_6(C_2R_2)$, even though this known compound has been synthesized by a different method and structurally characterized[38].

Metal Fragment Condensation

This synthetic method is growing more popular as it often allows systematic addition of an unsaturated mononuclear fragment to an existing cluster. The unsaturated fragment (such as $Cr(CO)_5$) is commonly formed *in situ* or can be obtained by the loss of a labile ligand from a modestly stable organometallic fragment such as $(PPh_3)_2Pt(C_2H_4)$. This allows cluster growth to often appear more rational and not rely on empirical approaches with little predictability. However, generalizations are still difficult to make and approaches only experimental.

The two most common fragments that are employed in this reaction are CpNi and $Fe(CO)_3$. CpNi is usually obtained in situ[38,39] from $Cp_2Ni_2(CO)_2$ or Cp_2Ni, and $Fe_2(CO)_9$ is the most common precursor for $Fe(CO)_3$ fragments although $Fe(CO)_5$ and $Fe_3(CO)_{12}$ have also been employed[2,24]. A good example of this reaction is illustrated in Scheme I-1. $Cp_2Ni_2(C_2R_2)$ (A), a

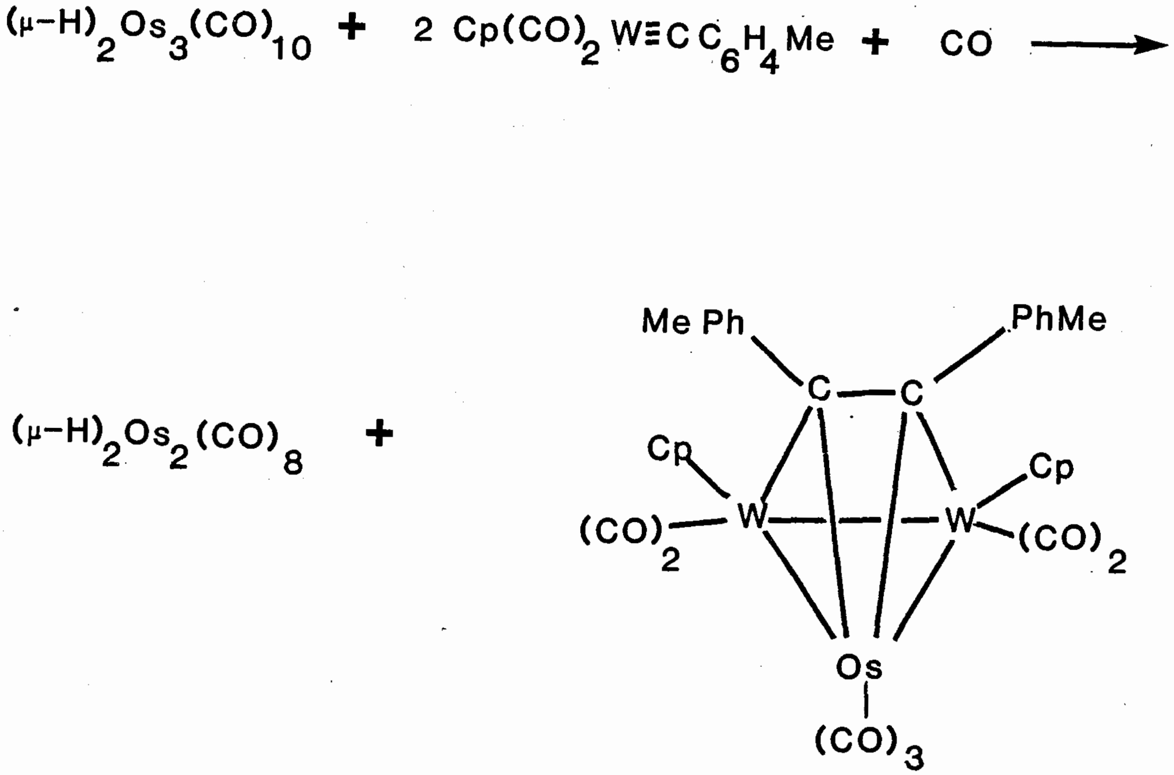


SCHEME I–1

dinuclear alkyne compound which is formally saturated (i.e., all metals have an 18 electron count) adds an $Fe(CO)_3$ group to form (B), a heterotrimetallic (MMM') cluster that can be isolated or add a further $Fe(CO)_3$ unit to form a heterotetrametallic $(M_2M'_2)$ alkyne cluster (C)[38].

Carbyne Coupling

Alkyne ligands have often been observed to cleave their carbon-carbon triple bond upon reaction with some transition metal complexes to form μ_3-carbyne species[41]. However Stone[42] and Churchill[32] have recently utilized the reverse reaction, carbyne coupling, to form a number of cluster alkyne compounds. Both workers used mononuclear carbynes ($M\equiv C-R$) as precursors in this reaction. An example is given in Scheme I-2. The mononuclear carbyne compound couples with itself and adds an $Os(CO)_2$ fragment from the



SCHEME I-2

$(\mu\text{-}H)_2Os_3(CO)_{10}$ cluster in addition to scavenging a CC to form the MMM' alkyne cluster, $Cp_2W_2Os(CO)_7(C_2(C_6H_4Me)_2)$.

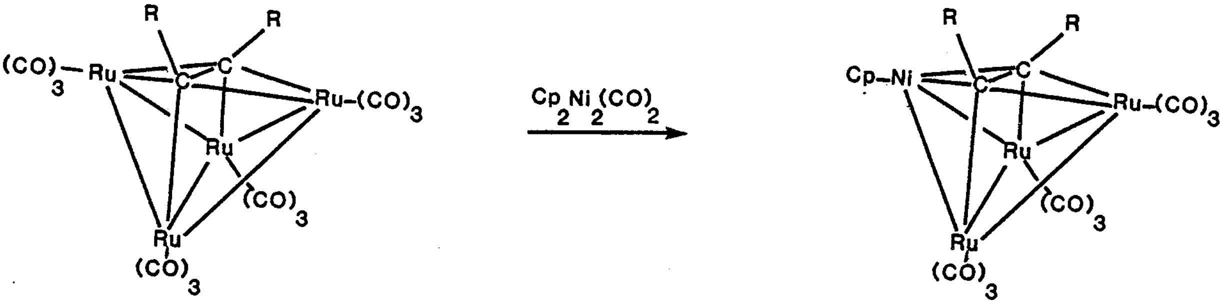
Reactions of Carbonyl Anions

The synthesis of mixed-metal clusters via the addition of a carbonylmetallate to a closed metal carbonyl cluster has been pioneered by Geoffroy[43] and shown to be a versatile method. The compounds are usually isolated as the hydride clusters after acidification rather than as the anionic cluster. Only a few homo- or heterometallic alkyne clusters have been made in this fashion. $H_2Ru_3(CO)_9(C_2Ph_2)$ can be synthesized from

$Ru_3(CO)_{12}$ and C_2Ph_2 in basic methanol followed by acidification[44].

Metal Substitution or Metal Exchange Reactions

The specific exchange of one metal unit for another in a metal cluster was first discovered by Vahrenkamp[45]. Since that time the method has been extended to the synthesis of heterometallic alkyne clusters. The example shown in Scheme I-3 has a CpNi fragment substituting for a $Ru(CO)_3$ moiety. Only exchange with a wing ruthenium is observed and no other isomers were reported[46]. This method has been growing in popularity as it allows the synthesis of mixed-metal alkyne clusters which could not be made in any other fashion. Certainly dinuclear metal-metal bonded complexes[47] and dinuclear alkyne compounds[38] equilibrate easily with formation of the mixed metal complexes. However, this reaction has not yet reached the stage in cluster alkyne chemistry where products are totally predictable but it clearly demonstrates potential.



SCHEME I–3 .

Metal Extrusion

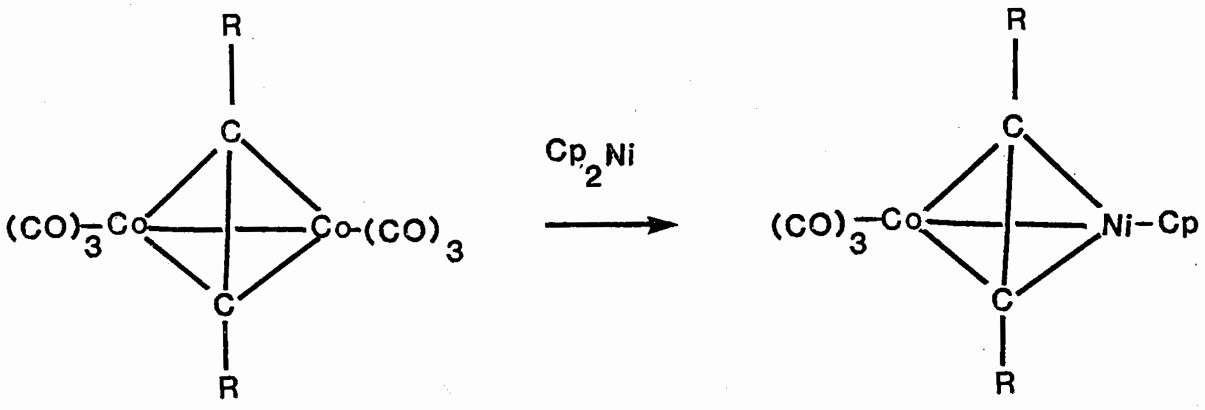
Only a few examples exist of the extrusion of a metal from a cluster alkyne species to give a cluster of lower nuclearity. These include the proton induced fragmentation[48] of $[Co_3Ru(CO)_{12}(C_2Ph_2)]^-$ to give $Co_2Ru(CO)_{10}(C_2Ph_2)$ and the phosphine induced cleavage[40] of $Cp_2Ni_2Fe_2(CO)_6(C_2Ph_2)$ to yield $[CpNi(PR_3)_2]^+[CpNiFe_2(CO)_6(C_2Ph_2)]^-$. This method is unlikely to become a general preparative reaction and will not be treated further.

1.3 Synthesis and Characterization of the Heterotrimetallic Alkyne Clusters, $CpNiCoM(CO)_6(RC_2R')$ (M = Fe,Ru,Os)

The subject of the present investigation is now described. The study entails developing a method for the synthesis of a family of heterotrimetallic alkyne clusters in which all three metal atoms are different (MM'M") and one of them is varied among the congeners Fe, Ru and Os. The solid state structures have been obtained by IR and X-ray methods of as closely representative members as possible comprising each metal of the iron group in turn. Because of inevitable difficulties in obtaining X-ray quality crystals, minor changes in the alkyne and some other modifications have been necessitated. The observed structures are not identical down the series, and the significant changes which occur will be discussed. The solid state structures are complemented by solution studies by IR and particularly dynamic ^1H NMR methods. The clusters are found to be stereochemically non-rigid in solution and the nature of this non-rigidity involving reorganization of the alkyne on the trimetallic face will be described.

1.3.1 Synthesis

It was decided to pursue the method of metal fragment condensation in order to synthesize the desired heterotrimetallic alkyne clusters. $CpNiCo(CO)_3(RC_2R')$ was chosen as the starting heterobimetallic alkyne compound because a variety of alkynes could be bound; C_2Ph_2, C_2Et_2 and MeC_2Ph were chosen. Also the compounds could be easily synthesized[38] in fair yield from $Co_2(CO)_6(RC_2R')$ and Cp_2Ni (or $[CpNi(CO)]_2$) by a metal exchange reaction as shown below. In addition, the compounds were air stable



both in the solid state and solution and could be separated easily from other materials by column chromatography.

The reaction of excess $Fe_2(CO)_9$ with $CpNiCo(CO)_3(RC_2R')$ in hexanes produces the desired NiCoFe alkyne clusters, $CpNiCoFe(CO)_6(C_2Ph_2)$ (1), $CpNiCoFe(CO)_6(C_2Et_2)$ (4) and $CpNiCoFe(CO)_6(MeC_2Ph)$ (7) in reasonable yield (25-30%). These clusters can be separated from starting materials and side-products by repeated column chromatography. $Fe_2(CO)_9$ fragments to give $Fe(CO)_4$ and $Fe(CO)_5$ groups, which can recombine with $Fe_2(CO)_9$ to form $Fe_3(CO)_{12}$ which was always present and isolated in these reactions. The formation of $Fe_3(CO)_{12}$ is enhanced by an increase in the reaction temperature or by changing solvents from hexanes to diethyl ether or THF. Also, when THF or diethyl ether was used a substantial amount of the black $Fe_3(CO)_9(C_2Ph_2)$ was isolated[28]. Replacement of $Fe_2(CO)_9$ by $Fe(CO)_5$ led to an increased number of side-products, especially $Cp_2Ni_2Fe_2(CO)_6(C_2Ph_2)$[34], at the expense of the desired NiCoFe cluster. The use of $Fe_3(CO)_{12}$ rather than $Fe(CO)_5$ or $Fe_2(CO)_9$ led to very poor yields of 1 that were not improved upon changing solvent or temperature.

The formation of $CpNiCoRu(CO)_6(C_2Ph_2)$ (2), $CpNiCoRu(CO)_6(C_2Et_2)$ (5) and $CpNiCoRu(CO)_6(MeC_2Ph)$ (8) was effective when $Ru(CO)_5$ but not $Ru_3(CO)_{12}$ was employed. The yields were low (10-15%), but the unreacted $Ru(CO)_5$ could be recovered from the reaction as $Ru_3(CO)_{12}$ and reconverted to $Ru(CO)_5$ and reused. The use of $Ru_3(CO)_{12}$ led only to trace amounts of 2, 5 or 8 being formed.

The synthesis of the osmium analogues, $CpNiCoOs(CO)_6(C_2Ph_2)$ (3), $CpNiCoOs(CO)_6(C_2Et_2)$ (6) and $CpNiCoOs(CO)_6(MeC_2Ph)$ (9) was similar to that of the ruthenium clusters. Only $Os(CO)_5$ and not $Os_3(CO)_{12}$ was successful at room temperature. When the reaction temperature was increased a small amount of NiCoOs cluster was obtained using $Os_3(CO)_{12}$, but this offered no advantage to using $Os(CO)_5$ at lower temperatures.

1.3.2 Characterization

The heterotrimetallic alkyne clusters have been characterized by solution and solid state (KBr) IR (Table 1), X-ray, ^1H NMR (Table 2), mass spectral analysis (Table 2) and by elemental analysis where possible (see Experimental). In addition, for each cluster the calculated isotopic distribution of the parent ion in the mass spectrum was compared to the experimental distribution.

The numbering scheme used for the clusters is shown below.

$CpNiCoFe(CO)_6(C_2Ph_2)$ - 1
$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ - 1a
$CpNiCoRu(CO)_6(C_2Ph_2)$ - 2
$CpNiCoOs(CO)_6(C_2Ph_2)$ - 3
$CpNiCoFe(CO)_6(C_2Et_2)$ - 4
$CpNiCoRu(CO)_6(C_2Et_2)$ - 5
$CpNiCoOs(CO)_6(C_2Et_2)$ - 6
$CpNiCoFe(CO)_6(MeC_2Ph)$ - 7
$CpNiCoRu(CO)_6(MeC_2Ph)$ - 8
$CpNiCoOs(CO)_6(MeC_2Ph)$ - 9

1.3.2a Infrared Spectroscopy

The solution (hexanes) IR spectra (listed in Table 1) did not vary greatly between compounds of the same trimetallic framework, i.e., NiCoFe, with different alkynes. The IR spectra of 1, 4 and 7, (all with NiCoFe frameworks) are shown in Figure 1 for comparison. There are certainly some small shifts in wavenumber between the different species and variations in intensity, but all have the highest wavenumber absorption at about 2068 cm^{-1} and the most intense band around 2022 cm^{-1}. The NiCoRu and NiCoOs clusters differ from the NiCoFe analogues but are fairly similar among themselves. Again, the highest wavenumber absorption is around 2068 cm^{-1} and the largest peak is now found at 2040 cm^{-1}. In addition to sharp terminal ν(CO) absorptions, all the NiCoRu and NiCoOs alkyne clusters show a weak, broadened carbonyl peak in the 1885-1895 cm^{-1} region. This is assignable to a μ_2-CO carbonyl species. While broadened in solution, an IR of the cluster as a KBr pellet revealed the band more clearly, and an example is shown in Figure 2. While 2 has a very intense and sharp bridging CO band, both the intensity and broadness of this absorption vary from cluster to cluster. The assignment of the lowest wavenumber band to a doubly bridging CO is in agreement with the X-ray structures of 2 and 3 which both contain a semibridging CO.

1a, which has only five carbonyl ligands, displays a different IR from the hexacarbonyl clusters. The highest wavenumber absorption occurs at 2039 cm^{-1} (rather than 2068 cm^{-1} for the other clusters) and a bridging CO species is noted which was not observed for other NiCoFe clusters. The infrared spectrum as a KBr pellet the IR showed the μ_2-CO species to be at a lower wavenumber than for the hexacarbonyl clusters; 1827 versus 1858-1876 cm^{-1}. The X-ray structure of 1a confirmed the existence of a semibridging CO group.

Table 1. Infrared Data for Compounds 1-9

Cluster		ν(CO) Hexanes (cm^{-1})	ν(CO) KBr (cm^{-1})
CpNiCoFe(CO)$_6$(C$_2$Ph$_2$)	(1)	2069s, 2024s, 2020s, 2006m, 1970m, 1965m	2067s, 2016s,br, 1971s
CpNiCoFe(CO)$_5$(PPh$_3$)(C$_2$Ph$_2$)	(1a)	2039s, 1974s, 1969s, 1838vbr,w	2040s, 1965s,br, 1827m
CpNiCoRu(CO)$_6$(C$_2$Ph$_2$)	(2)	2072s, 2043vs, 2010s, 2002s, 1885w,br	2072s, 2051s, 2046s, 2015s, 2008s, 1985s, 1865s
CpNiCoOs(CO)$_6$(C$_2$Ph$_2$)	(3)	2072s, 2041s, 2015m, 2002s, 1989s, 1973m, 1884w,br	2069s, 2044s, 2000vs, 1983s, 1858s
CpNiCoFe(CO)$_6$(C$_2$Et$_2$)	(4)	2065s, 2021vs, 2013s, 2004s, 1972m, 1968m	2064s, 2013vs,br, 1959m
CpNiCoRu(CO)$_6$(C$_2$Et$_2$)	(5)	2068s, 2035vs, 2005s, 1980w, 1894w,br	2065s, 2027s, 1997s,br, 1874m,br

Table 1 (Cont'd)

Cluster		ν(CO) Hexanes (cm^{-1})	ν(CO) KBr (cm^{-1})
$CpNiCoOs(CO)_6(C_2Et_2)$	(6)	2068s, 2036vs, 2008s, 2002s, 1981m, 1970m, 1874vw,br	2065s, 2031s, 1998s, 1967m,sh, 1875vw,br
$CpNiCoFe(CO)_6(MeC_2Ph)$	(7)	2067s, 2021vs, 2006s, 1980m, 1972m, 1967m	2053s, 2050s, 2029m,sh, 1996s,br
$CpNiCoRu(CO)_6(MeC_2Ph)$	(8)	2071s, 2039vs, 2008s, 1998s, 1887w,sl.br	2069s, 2033s, 1996s,br, 1876m,sl.br
$CpNiCoOs(CO)_6(MeC_2Ph)$	(9)	2069s, 2040vs, 2011m, 2002s, 1985m, 1972m, 1886w,br	2066s, 2033s, 1993s,br, 1871m,br

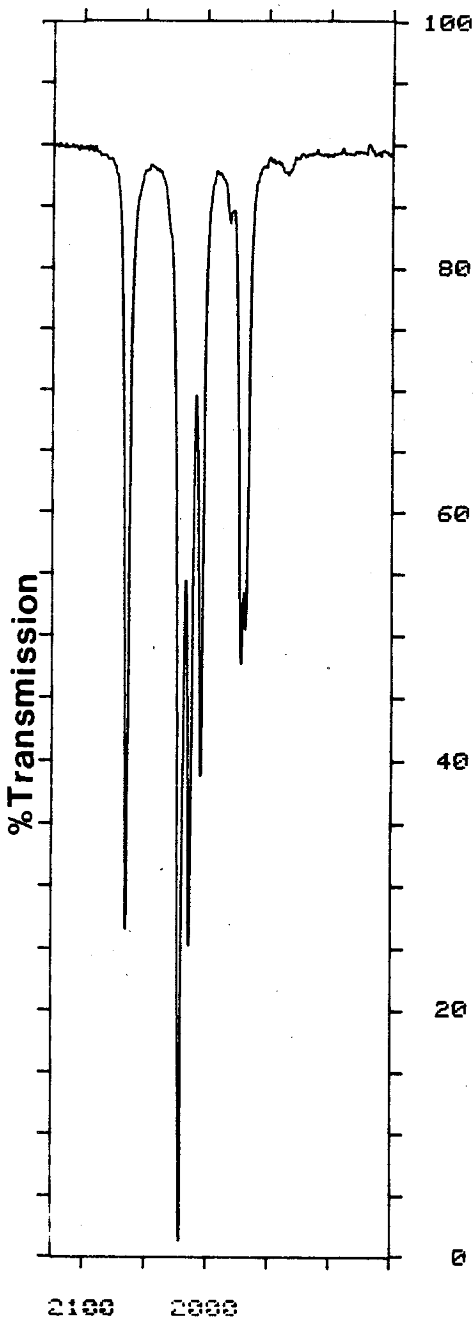
FIGURE 1

Infrared Spectra (hexanes, ν(CO)) of

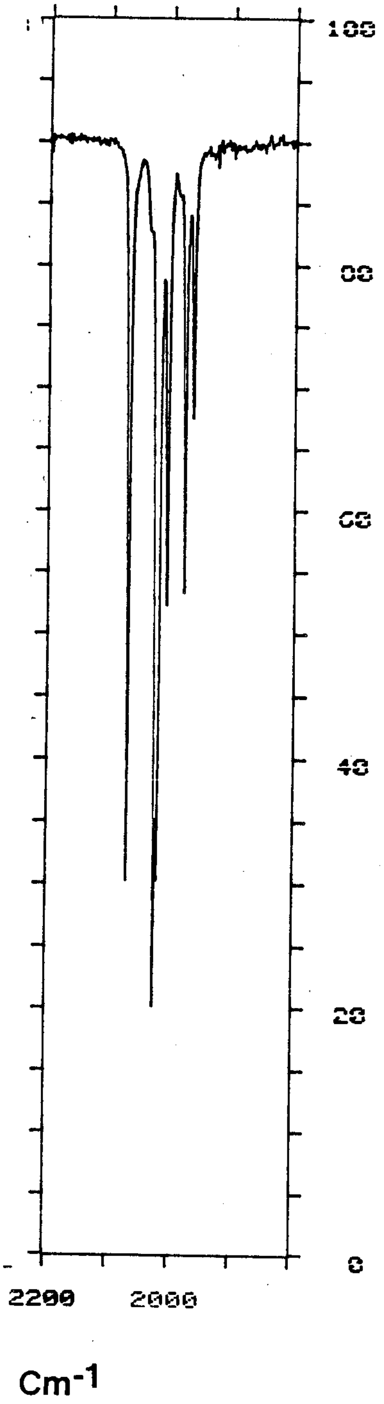
$CpNiCoFe(CO)_6(C_2Ph_2)$ (<u>1</u>),

$CpNiCoFe(CO)_6(C_2Et_2)$ (<u>4</u>),

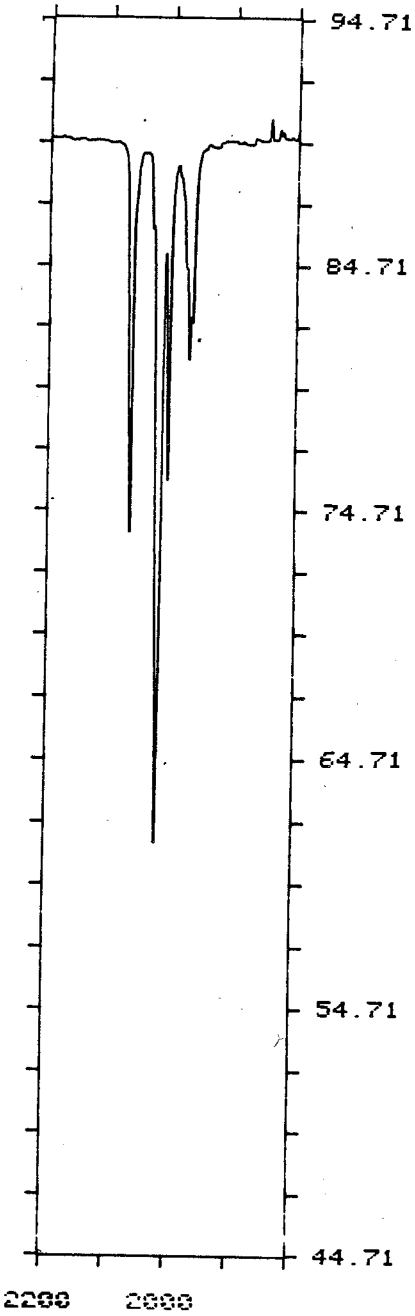
$CpNiCoFe(CO)_6(MeC_2Ph)$ (<u>7</u>)


%Transmission
100
80
60
40
20
0
2100
2000

1


100
80
60
40
20
0
2200
2000
Cm-1

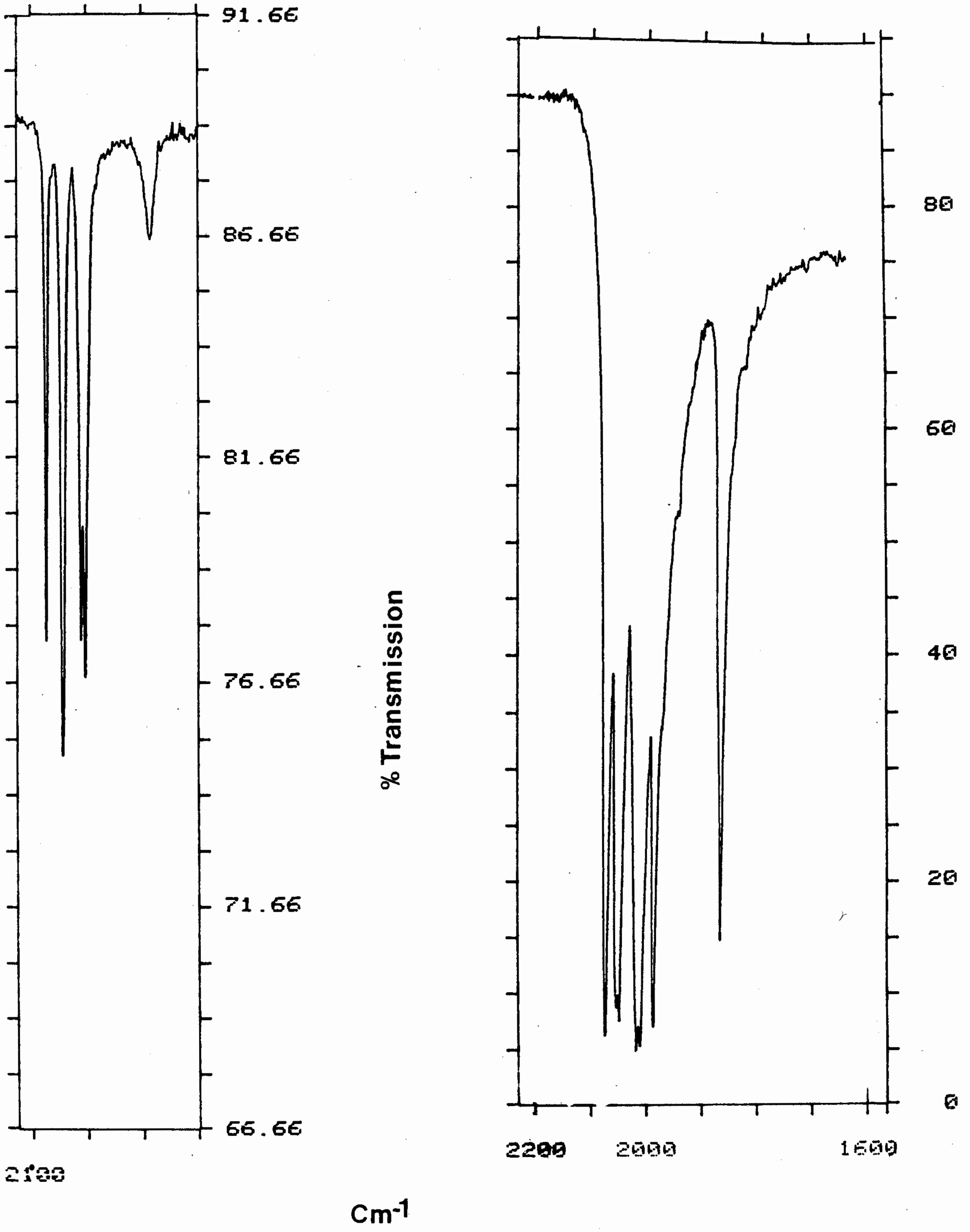
4


94.71
84.71
74.71
64.71
54.71
44.71
2200
2000

7

FIGURE 2

Infrared Spectra of $CpNiCoRu(CO)_6(C_2Ph_2)$
in hexanes and KBr



Hexanes

KBr

(2)

1.3.2b Mass Spectra

As shown in Table 2 the mass spectrum of each hexacarbonyl heterotrimetallic cluster revealed the parent ion and the sequential loss of six carbonyl groups. 1a which contains a PPh_3 group also yielded a parent ion albeit in small intensity. Often clusters with phosphine ligands do not show parent ions due to a lack of stability. Other peaks, notably those belonging to CpNi (123 amu), CpNiCoFe (238 amu), CpNiCoRu (284 amu) and CpNiCoOs (374 amu) and the appropriate alkyne ligand; C_2Et_2 (82 amu), MeC_2Ph (116 amu) or C_2Ph_2 (178 amu) were observed. In addition, 1a revealed the loss of its PPh_3 (262 amu) ligand.

The isotopic distribution of the parent ion can serve as a fingerprint for a given composition, particularly for the metal atoms, as some have a number of isotopes. All of the heterotrimetallic cluster alkynes 1-9, except 1a, had their parent ion distributions calculated and compared with the values obtained experimentally to confirm the presence of the three different metals. An example of the observed and calculated mass distribution found for the parent ion of 5 is shown in Figure 3 and clearly there is close agreement. All of the compounds 1-9 showed similar good agreement. Since 1a had a weak parent ion, the calculated mass distribution was performed with the $P-CO^+$ ion and this agreed satisfactorily with the observed values.

1.3.2c NMR Spectra

The ^1H NMR data for 1-9 are listed in Table 2. Compounds 1-3 all show a single sharp peak around δ 5.10 assignable to the Cp protons and a multiplet at δ 7.1-7.3 due to the protons of the diphenylacetylene ligand.

The diethylacetylene substituted compounds, 4-6, have an interesting ^1H

Table 2. ^{1}H NMR and Mass Spectral Data for 1-9

Cluster		^{1}H NMR (δ) ($CDCl_3$)	Mass Spectrum[a], m/e (Relative Intensity)	Colour
$CpNiCoFe(CO)_6(C_2Ph_2)$	(1)	5.05s(5H)-Cp, 7.1-7.3m(10H)-Ph	584(10), 556(30), 528(25), 500(46), 472(85), 444(100), 416(65)	light brown
$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$	(1a)	5.27s(5H)-Cp, 7.1-7.1m-Ph(C_2Ph_2,PPh_3)	846(0.5), 818(5), 790(7), 762(4), 734(10), 706(11), 444(30), 262(100)	dark brown
$CpNiCoRu(CO)_6(C_2Ph_2)$	(2)	5.13s(5H)-Cp, 7.1-7.3m(10H)-Ph	630(3), 602(12), 574(8), 546(4), 518(30), 490(15), 462(80)	dark brown
$CpNiCoOs(CO)_6(C_2Ph_2)$	(3)	5.07s(5H)-Cp, 7.1-7.4m(10H)-Cp	720(5), 692(60), 664(85), 636(12), 608(100), 580(18), 552(75)	dark brown
$CpNiCoFe(CO)_6(C_2Et_2)$	(4)	1.41t, 1.63t(6H)-CH_3, 2.47m, 2.60m, 2.82m, 3.04m(4H)-CH_2, 5.20s(5H)-Cp	488(5), 460(20), 432(20), 404(15), 376(18), 348(100), 320(42)	green

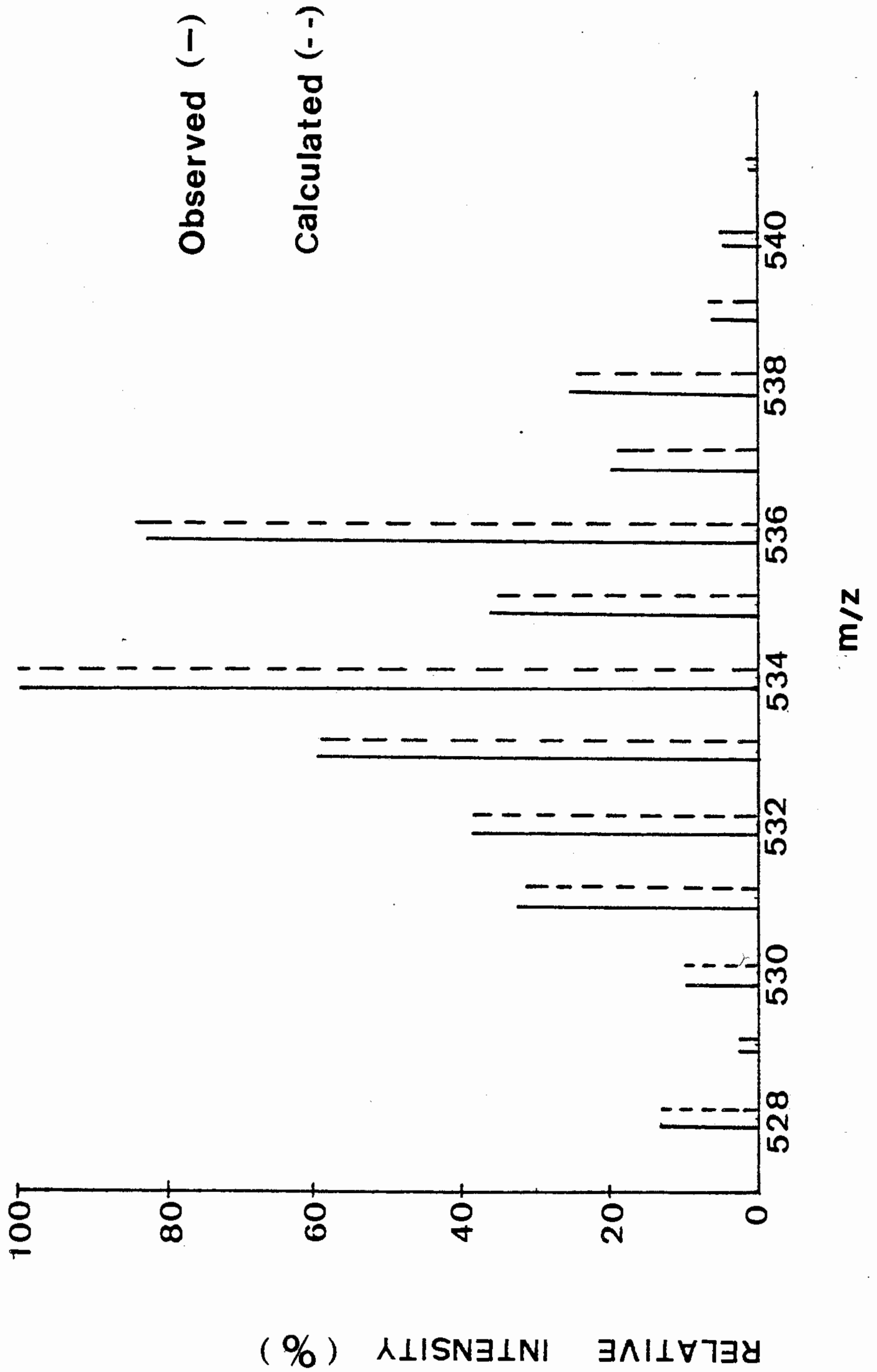
Table 2 (Cont'd)

Cluster		^{1}H NMR (δ) (CDCl$_3$)	Mass Spectrum^a, m/e (Relative Intensity)	Colour
CpNiCoRu(CO)$_6$(C$_2$Et$_2$)	(5)	1.24t, 1.37t(6H)-CH$_3$, 2.20m, 2.50m, 2.78m(4H)-CH$_2$, 5.25s(5H)-Cp	534(19), 506(22), 478(18), 450(40), 422(100), 394(100), 366(75)	dark brown
CpNiCoOs(CO)$_6$(C$_2$Et$_2$)	(6)	1.33t, 1.47t(6H)-CH$_3$, 2.62m, 2.72m, 2.89m(4H)-CH$_2$, 5.32s(5H)-Cp	624(14), 596(22), 568(26), 540(52), 512(46), 484(100), 456(65)	dark brown
CpNiCoFe(CO)$_6$(MeC$_2$Ph)	(7)	2.62s, 2.68s(6H)-CH$_3$, 4.94s, 5.22s(10H)-Cp, 7.1-7.3m(10H)-Ph	522(3), 494(19), 466(20), 438(18), 410(29), 382(93), 354(100)	green
CpNiCoRu(CO)$_6$(MeC$_2$Ph)	(8)	2.31s, 2.64s(6H)-CH$_3$, 4.90s, 5.22s(10H)-Cp, 7.1-7.4m(10H)-Ph	568(3), 540(10), 512(9), 484(9), 456(30), 428(29), 400(100)	brown
CpNiCoOs(CO)$_6$(MeC$_2$Ph)	(9)	2.53s, 2.79s(6H)-CH$_3$, 4.97s, 5.27s(10H)-Cp, 7.1-7.4m(10H)-Ph	658(2), 630(20), 602(20), 574(14), 546(100), 518(16), 488(85)	dark brown

a Mass position of the most intense peak in the isotopic envelope; Fe^{56}, Co^{59}, Ni^{58}, Ru^{102}, Os^{192}.

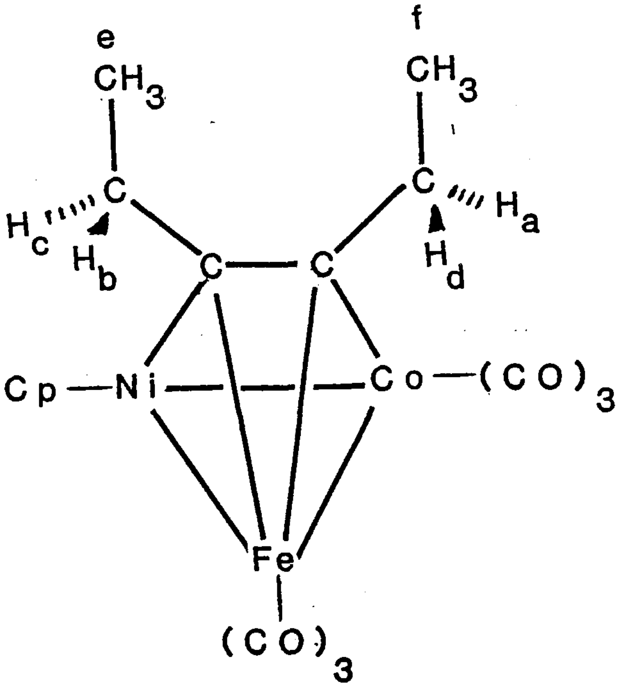
FIGURE 3

Comparison of the Observed and Calculated Isotopic Distribution of the Parent Ion of $CpNiCoRu(CO)_6(C_2Et_2)$


Observed (—)
Calculated (- -)
RELATIVE INTENSITY (%)
100
80
60
40
20
0
528
530
532
534
536
538
540
m/z

NMR. One Cp resonance is observed for each compound but the methylene and methyl protons are more complex. Figures 4, 5 and 6 show the CH_2 and CH_3 region of 4, 5 and 6 respectively. In the spectrum of both 5 and 6 it is evident that two overlapping resonances contribute to the ~δ 3.0 signal. The spectrum of 6 is deceptively similar to that of 5 but see later in the section. The methyl groups of 4 are triplets as expected because of coupling to the neighbouring CH_2 group. However, the protons on the methylene are clearly not the expected 1:3:3:1 pattern obtained from coupling with only the nearest CH_3 group.

From the representation of 4, $CpNiCoFe(CO)_6(C_2Et_2)$, below as I-10, it is clear that the cluster is chiral. The CH_2 protons and



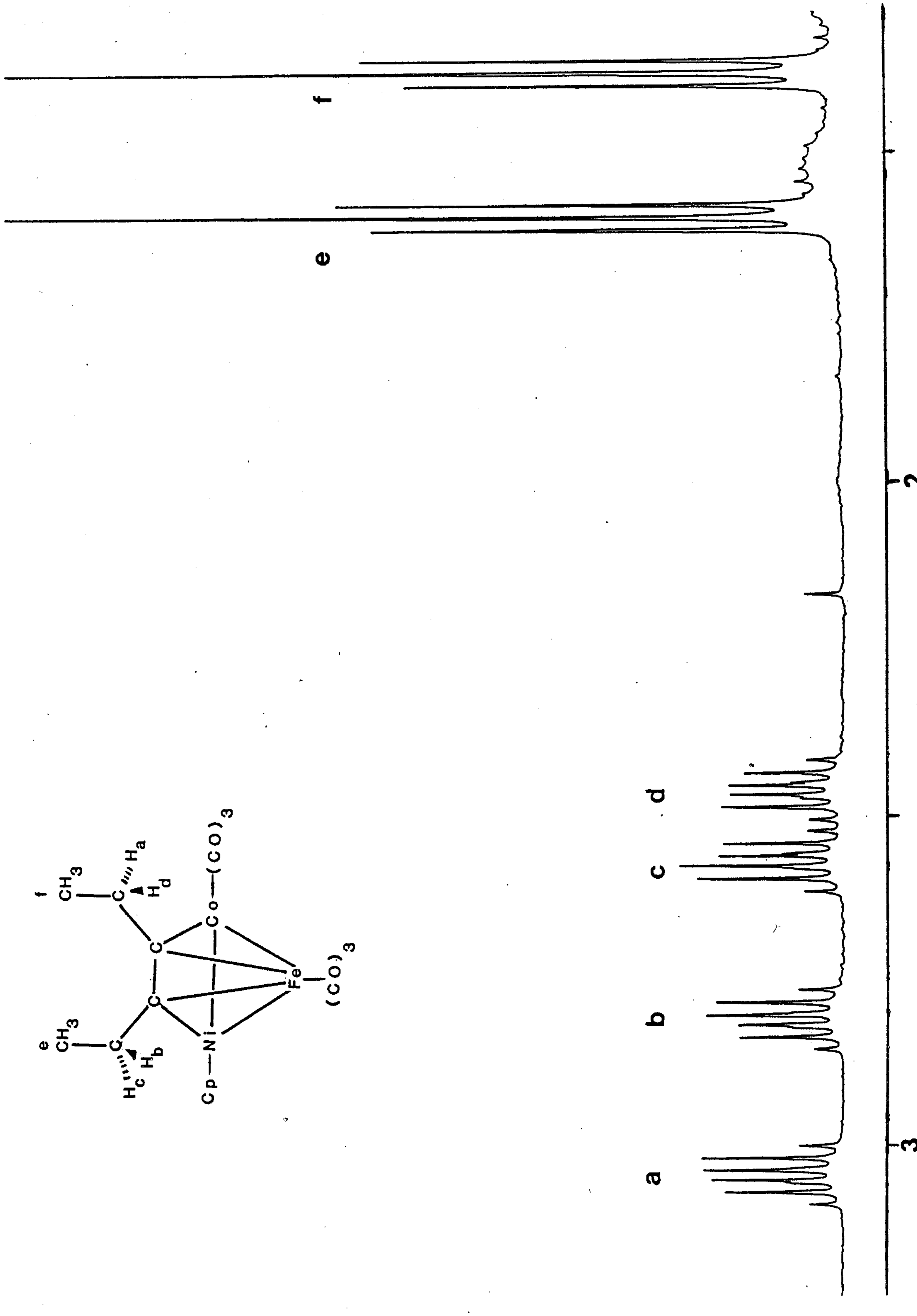
I–10

methyl groups have been labelled such that H_b, H_c and H_e are adjacent to the Ni atom and H_a, H_d and H_f to the Co atom. Since the methylene protons are diastereotopic they should have distinct chemical shifts and the coupling experienced by each methylene is now more complex. For example, H_d is split into a doublet by coupling with H_a and each line of the doublet is split into four lines of intensity 1:3:3:1 by the neighbouring CH_3 group. Ideally, a doublet of quartets should be observed for each methylene proton but some overlap has occurred, and the smaller peaks are sometimes obscured. Both 5 and 6, which are also chiral, have two methylene protons with nearly identical chemical shifts and show only 3 methylene multiplets with an intensity ratio of 2:1:1.

A decoupling experiment was performed to establish which methylene protons were α to a particular methyl group. The methylene proton multiplets in 4, labelled H_{a-d} in Figure 4, were sequentially irradiated, and decoupling of either of the methyl groups (H_e or H_f) was monitored. This revealed that the methylene protons H_a and H_d were α to H_f and H_c and H_b were α to H_e. Our labelling of the methylene protons in I-10 is consistent with this information, however we have not yet established which CH_2 group is 'above' or adjacent to the CpNi fragment or which group is near the $Co(CO)_3$ fragment. To attempt this a Nuclear Overhauser Enhancement (NOE) experiment was carried out. In this technique, one spectrum is accumulated while simultaneously saturating irradiation is applied at the transition of interest. From this spectrum is subtracted a second spectrum for which the irradiation frequency is moved to a region free from any transitions. The resultant difference spectrum shows a large negative peak corresponding to the first irradiation frequency and positive peaks for any resonances that are enhanced by the NOE. The magnitude of the indirect coupling decreases with the sixth power of the distance, so only nuclei in close proximity to the irradiated one are enhanced. Irradiation of the Cp resonance of 4 at room temperature or at 202K, unfortunately, did not result

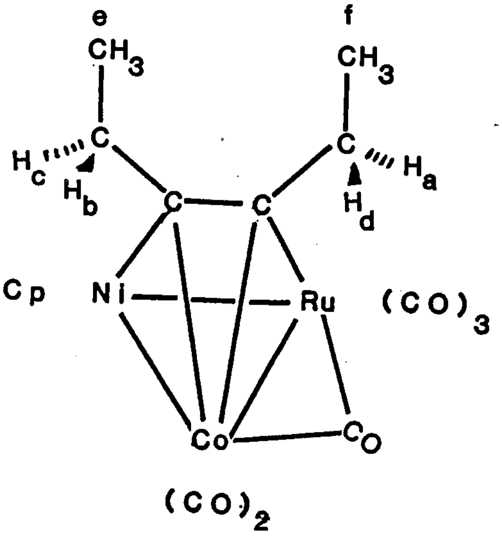
FIGURE 4

^1H NMR of the CH_2 and CH_3 region of $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

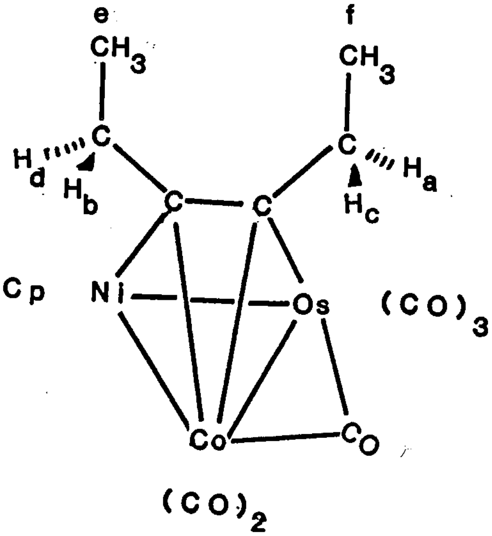

e
CH$_3$
H$_c$
H$_b$
C
Cp
Ni
Fe
(CO)$_3$
Co
(CO)$_3$
f
CH$_3$
H$_a$
H$_d$
a
b
c
d
e
f
3
2

in any observable enhancement of any of the CH_2 or CH_3 protons. The distance from the protons of the Cp ring to the CH_2 or CH_2 protons may be too large for this effect to be detected.

The methylene protons of 5 and 6 were also subjected to a decoupling experiment and are labelled in the NMR as shown in Figure 5. For 5, irradiation of the $H_{a/b}$ multiplet caused both the H_e and H_f triplets to be decoupled, irradiation of H_c resulted in the decoupling of H_e and lastly irradiation of H_d decoupled the triplet H_f. This established that the methylene proton H_c and one of the two protons under the $H_{a/b}$ envelope are ∝ to methyl group H_e, and H_d and the second proton under the $H_{a/b}$ envelope is next to H_f as illustrated in I-11.



I-11



I-12

The decoupling experiment involving the CH_2 hydrogens of 6 revealed a difference in the assignment of these peaks. The overall spectrum was very similar to 5 and the same labelling of the peaks is used in the NMR (Fig. 6). While irradiation of the $H_{a/b}$ multiplet caused collapse of both H_e and

FIGURE 5

^{1}H NMR of the CH_2 and CH_3 region of $CpNiCoRu(CO)_6(C_2Et_2)$ (5)

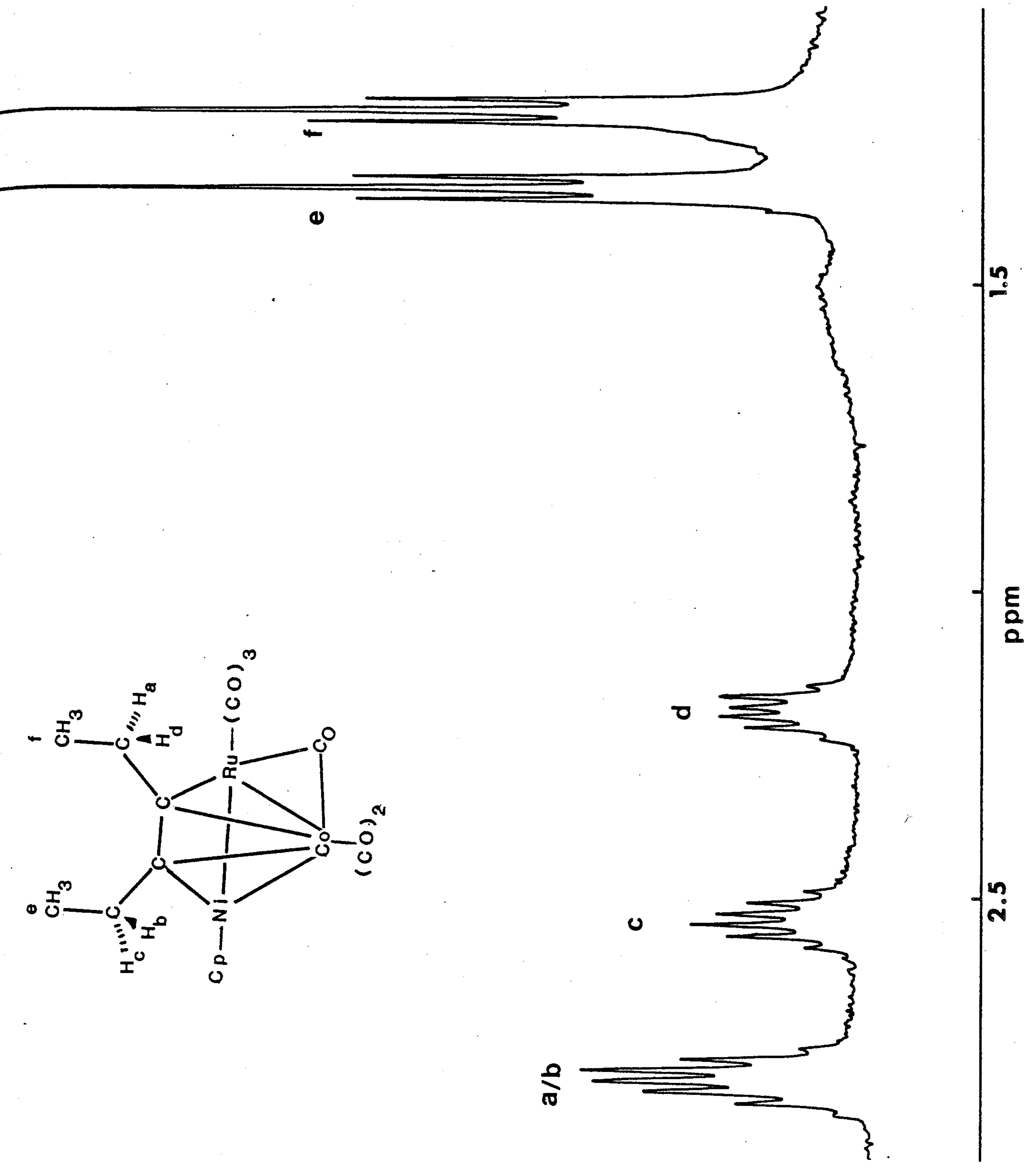

a/b
c
d
e
f
2.5
1.5
ppm
CH3
CH3
Hc
Hb
Ha
Hd
C
Ni
Ru
Co
Cp
(CO)3
(CO)2
CO

FIGURE 6

^{1}H NMR of the CH_2 and CH_3 region

$CpNiCoOs(CO)_6(C_2Et_2)$ (<u>6</u>)

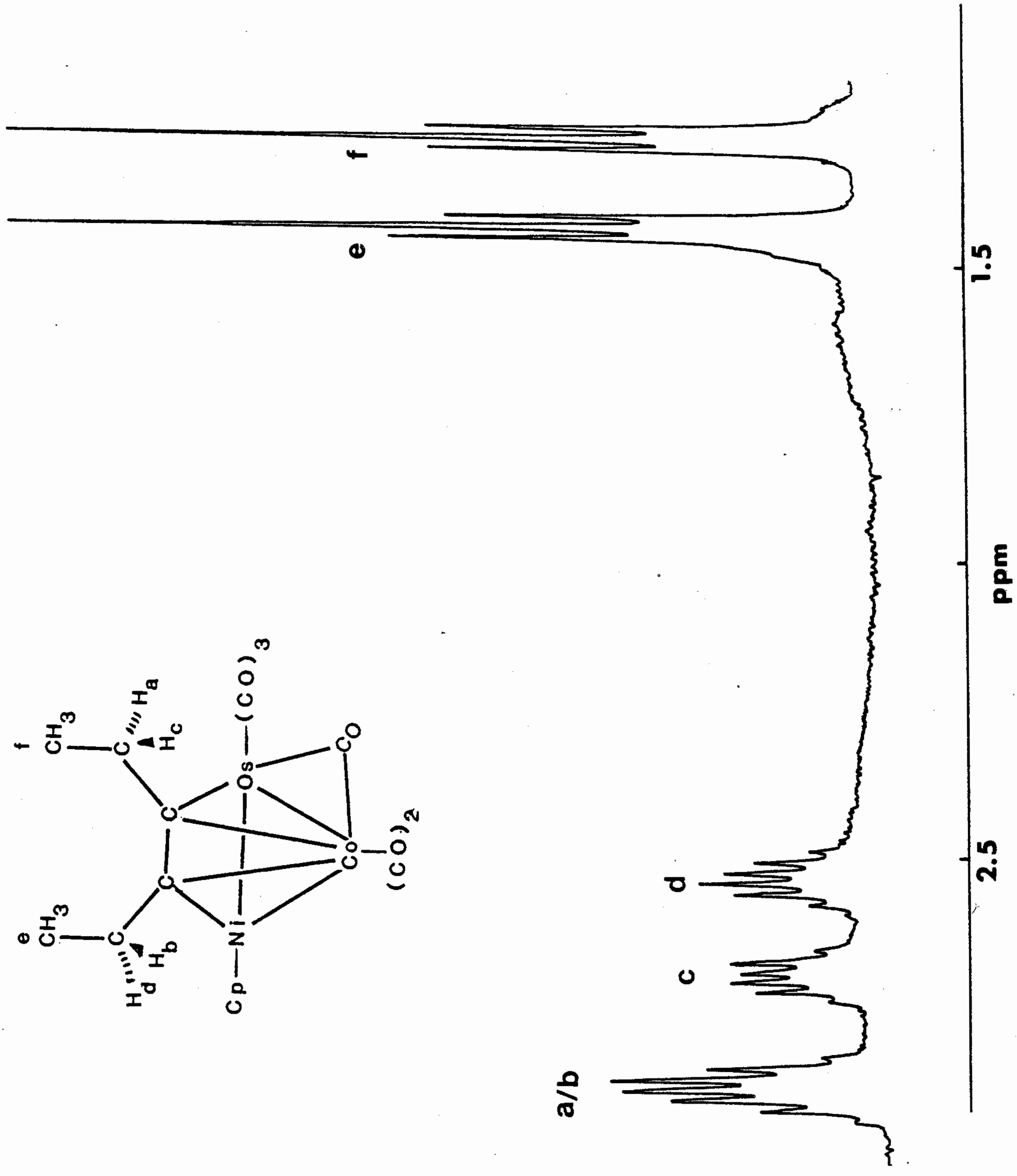
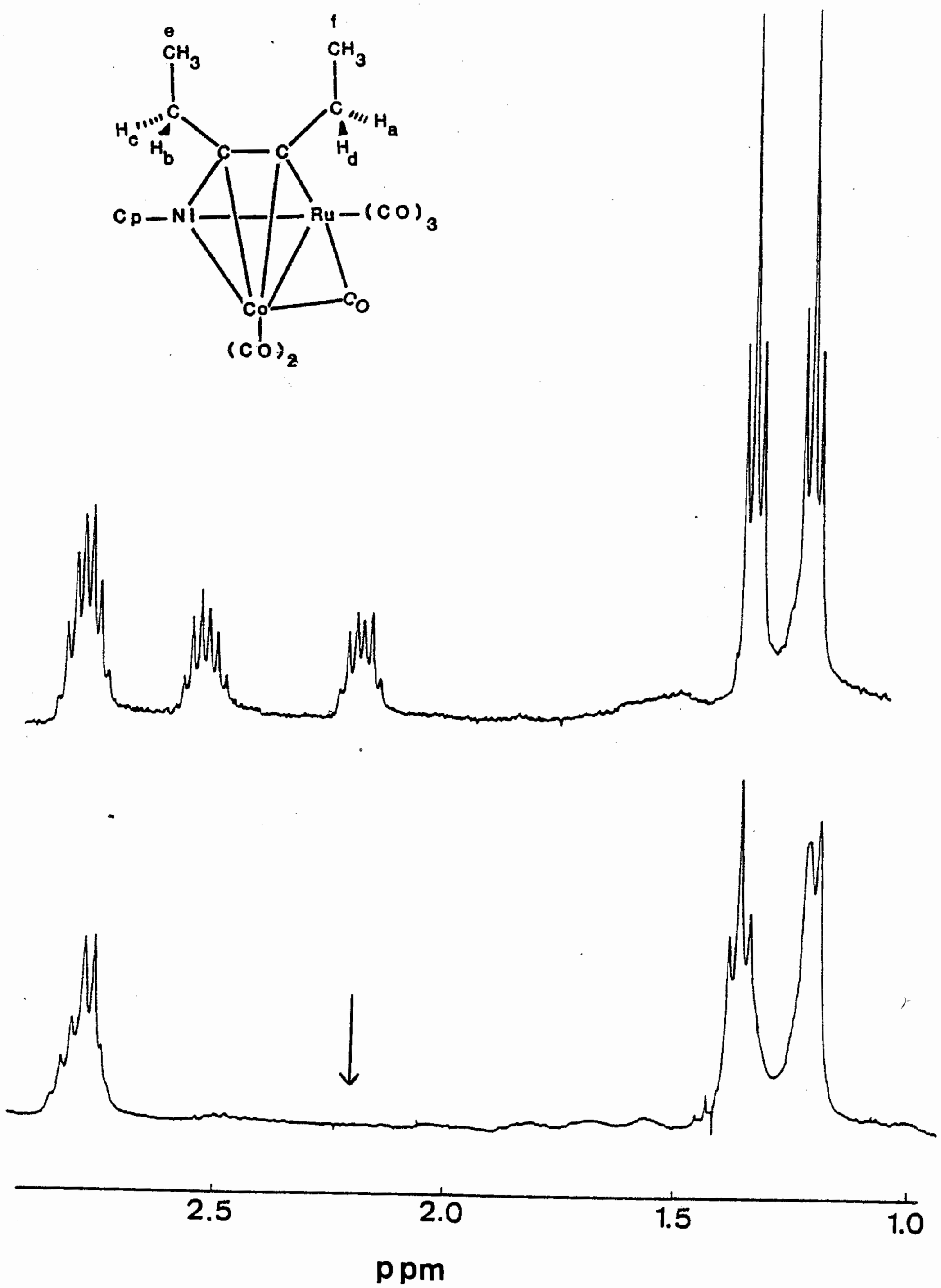

e
CH3
f
CH3
Ha
Hc
Hb
Hd
C
Os
(CO)3
CO
Co
(CO)2
Ni
Cp
a/b
c
d
e
f
2.5
1.5
ppm

FIGURE 7

Irradiation of a CH_2 Resonance
in the ^1H NMR Spectrum of
$CpNiCoOs(CO)_6(C_2Et_2)$ (<u>5</u>)

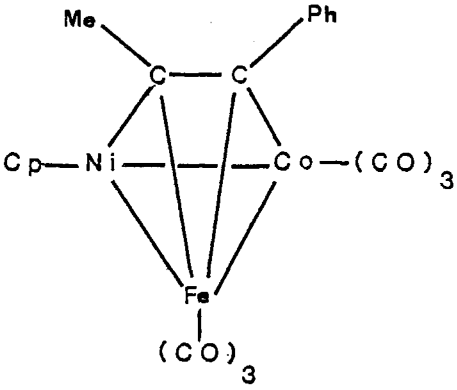

e
CH_3
f
CH_3
H_c
H_b
C
H_a
H_d
C
C
Cp—Ni
Ru—(CO)$_3$
Co
CO
(CO)$_2$
2.5
2.0
1.5
1.0
ppm

H_f, irradiation of H_c collapsed H_f and irradiation of H_d collapsed H_e, which is the opposite to that encountered for 5. The decoupling experiment is consistent with the labelling scheme for 5 in I-12.

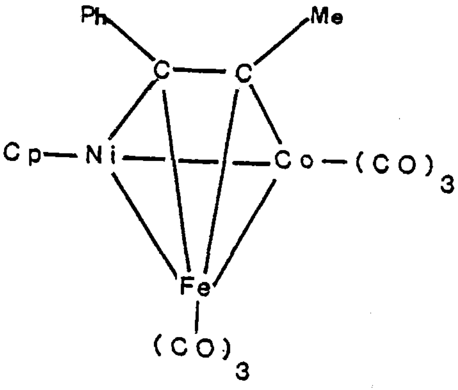
The decoupling experiments of 5 and 6 revealed further details. When methylene proton H_c of 5 was irradiated, in addition to decoupling the methyl resonance H_e, the multiplet due to proton H_d vanished. This is shown in Figure 7. The irradiation of H_d caused H_c to disappear but irradiation of the $H_{a/b}$ envelope did not affect H_c or H_d. This is equivalent to a spin saturation labelling experiment . Saturation of the H_c resonance will cause saturation and hence disappearance of the H_d resonance only if H_c and H_d are exchanging and in addition, the exchange rate must be comparable to the relaxation rate at the two sites. In compound 6, irradiation of H_c caused H_d to vanish and vice-versa and, as in 5, irradiation of the $H_{a/b}$ multiplet did not affect H_c or H_d.

Saturation of the H_a a methylene resonance in 4 did not result in transfer of the saturation to a different methylene hydrogen indicating that interchange of these hydrogens must be slower than in 5 or 6. In order to obtain a value for the activation energy of this interchange process the rate of saturation transfer between the methyl resonances in 4 at different temperatures was determined. This is the basis of the saturation transfer experiment detailed by Forsen and Hoffman[49] from which exchange kinetics are obtained. In their experiment one resonance is irradiated and the intensity of a second related resonance is determined as a function of time at varying temperatures. We pursued a slightly different approach from that which was suggested by Dahlquist[50] but one which yields the same information. A 180° pulse is applied to one resonance of interest in, for example, 4 causing the signal to be inverted. Spectra are recorded at varying delay times during which a fraction of the saturation from the first peak is transfered to the second. Both peaks are also decaying in proportion to their lifetimes so the total intensity of the two peaks decreases with longer delay times. Measurement at five different temperatures (291, 300, 307, 314, 321K) gave an activation energy $\Delta G^{\ddagger} = 74.5 \pm 1.3$ kJ mol^{-1}. An example of the calculation is provided in the Experimental Section. Unfortunately, for 5 and 6, there was enough overlap in the baseline of the methyl and methylene resonances to make it difficult to obtain accurate integrations because of some saturation transfer from one methyl group to the other even with very short delay times.

The ^{1}H NMR of compounds 7-9 were relatively simple consisting of a multiplet for the aromatic protons, two Cp peaks and two singlets assignable to the methyl protons of the alkyne ligand. The ^{1}H NMR of 7 is shown in Figure 8. The two sets of Cp, Me and Ph resonances occur because the alkyne ligand can be oriented on the heterotrimetallic framework in two different fashions, as shown below for 7 and labelled as I-13a and I-13b. The methyl group of the alkyne ligand can be adjacent to either the Ni or the Co atom



I–13a

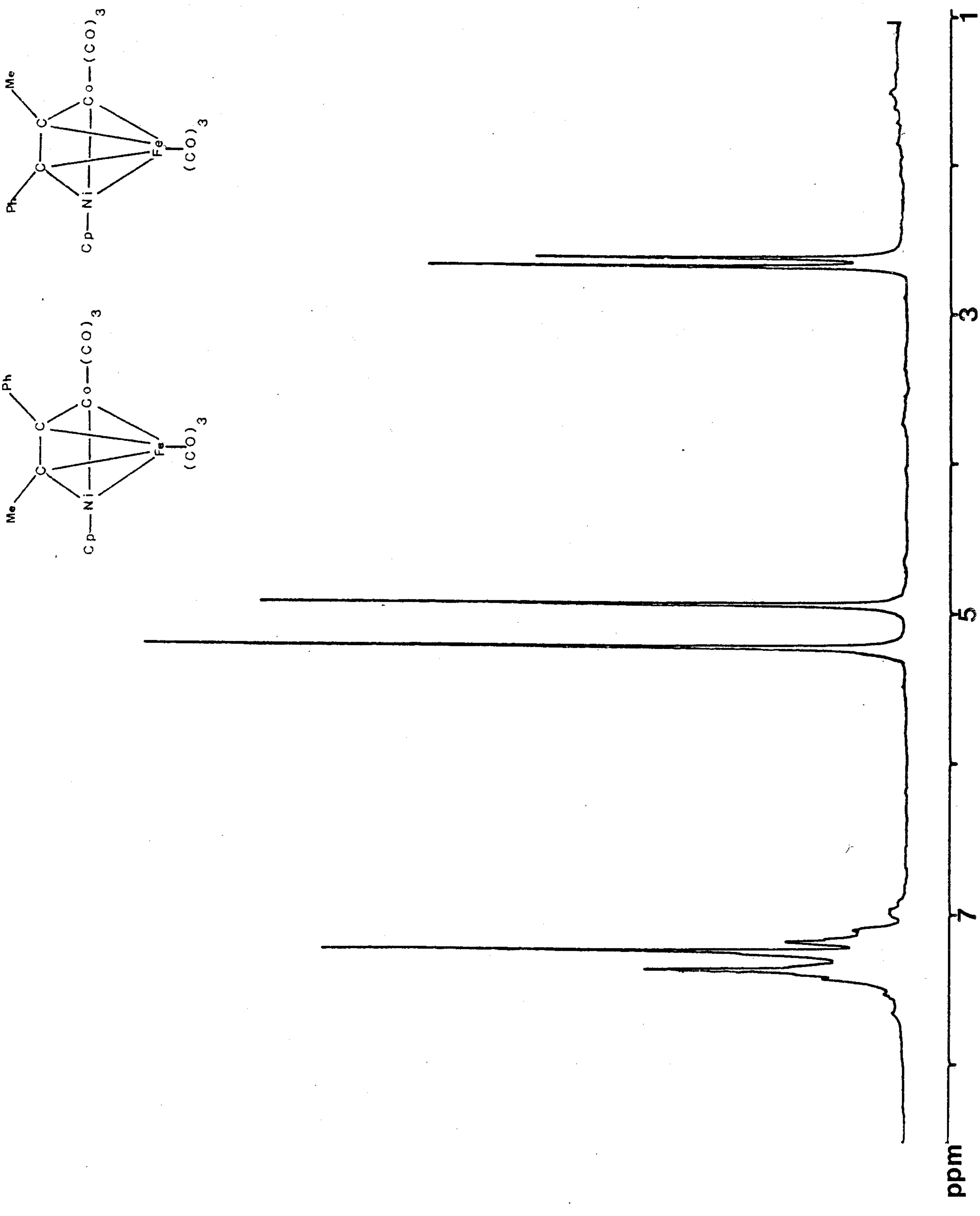


I–13b

and similarly for the phenyl ligand. Both 8 and 9 will also have two stereoisomers and the ^{1}H NMR substantiates this.

FIGURE 8

^{1}H NMR of CpNiCoFe(CO)$_6$(MeC$_2$Ph) (7)


Cp—Ni
Ph
Me
C
Co—(CO)$_3$
Fe
(CO)$_3$
Cp—Ni
Me
Ph
C
Co—(CO)$_3$
Fe
(CO)$_3$
1
3
5
7
ppm

The ratio of the intensities of the Cp peaks (and the methyls) are very solvent dependent. This ratio and the chemical shifts are listed in Table 3 for three different solvents. From the varying intensity ratios it is easily established that the Cp and Me peak of lower δ belong to the same isomer and the high δ resonances belong to the second isomer, i.e., either I-13a or I-13b. An identical rationale can be applied to 8 and 9. To attempt to establish which set of resonances are attributable to a particular isomer a NOE experiment was carried out. The Cp resonances of 7, 8 and 9 were sequentially irradiated and the phenyl and methyl peaks of the spectrum were monitored to observe any enhancement of these hydrogens. An enhancement of the methyl hydrogens might be expected if the Me group is adjacent to the Ni atom putting the group in proximity of the Cp ring, i.e., I-13a. Unfortunately, no detectable enhancement could be observed (at 294 or 202K) for 7, 8 or 9 and consequently assignment of the isomers could not be proven.

In addition to a difference in the relative intensities of the two Cp and two Me resonances of 7, 8 and 9 there is a sizeable chemical shift when the solvent is changed. As the polarity of the solvent decreases, acetone-d_6 to $CDCl_3$ to toluene-d_8, (Table 3) the Cp and Me resonances of 7, 8 and 9 shift quite significantly to higher field. The shifts are not equal, however. For example, while the Cp protons of 9 are separated by 0.35 ppm in acetone-d_6, in toluene-d_8 the difference is only 0.09 ppm. Clearly, one of the stereoisomers of 7, 8 and 9 is being affected by the change in polarity to a greater extent than the other. This problem will be examined further in the Discussion Section.

From the proportions of the two isomers at room and lower temperatures the ground state energy difference, ΔE, between the two stereoisomers of 7-

Table 3. Chemical Shifts and Intensity Ratios for the Cp and Me Resonances of 7, 8 and 9

Cluster	Solvent		
	$CDCl_3$ Chemical Shift (Ratio)	$Me_2CO\text{-}d_6$ Chemical Shift (Ratio)	$C_6D_5\text{-}CD_3$ Chemical Shift (Ratio)
$CpNiCoFe(CO)_6(MeC_2Ph)$ (7)			
Cp	5.22, 4.94 (1.24:1)	5.44, 5.11 (2.19:1)	4.78, 4.61 (1.47:1)
Me	2.68, 2.62 (1.28:1)	2.76, 2.70 (2.20:1)	2.48, 2.41 (0.78:1)
$CpNiCoRu(CO)_6(MeC_2Ph)$ (8)			
Cp	5.22, 4.90 (0.22:1)	5.45, 5.10 (0.42:1)	4.88, 4.66 (0.33:1)
Me	2.64, 2.31 (0.19:1)	2.74, 2.43 (0.43:1)	2.39, 2.24 (0.28:1)
$CpNiCoOs(CO)_6(MeC_2Ph)$ (9)			
Cp	5.27, 4.97 (0.43:1)	5.54, 5.19 (0.84:1)	4.93, 4.73 (0.63:1)
Me	2.79, 2.53 (0.36:1)	2.93, 2.67 (0.76:1)	2.56, 2.47 (0.71:1)

Table 4. Ground State Energy Difference, ΔE, Between the Isomers of 7, 8 and 9

Cluster	$CDCl_3$	ΔE kJ (mol^{-1}) Me_2CO-d_6	Toluene-d_8
7, Cp	0.527	1.92	0.946
Me	0.607	1.95	0.611
average	0.567	1.93	0.778
8, Cp	3.72	2.13	2.72
Me	4.08	2.07	3.12
average	3.90	2.10	2.92
9, Cp	2.07	0.427	1.13
Me	2.51	0.640	0.840
average	2.28	0.536	0.987

9 can be calculated. These ΔE values for the clusters are listed in Table 4 for both the Cp and Me resonances and a sample calculation is included in the Experimental Section.

The activation energy $\Delta G^{\ddagger}$, was obtained for 7, 8 and 9 from variable temperature coalescence experiments on both the methyl and cyclopentadienyl resonances by using the Eyring equation, the coalescence temperature (T_c), the rate constant (k_c) and the separation of the two peaks at 243K, $\Delta\nu_{243}$. An example calculation is included in the Experimental Section. The collapse of the methyl peaks of 7 are shown in Figure 9. The coalescence temperature for peaks of unequal intensity was approximated by the method of Fraser[51]. The rate constant K_a, represents the rate constant for conversion of the isomer that resonates at higher δ into the isomer which has the lower δ resonances and k_b represents the rate constant for the reverse reaction. All these calculated values are summarized in Table 5.

Compound 7, (NiCoFe core), has the largest activation energy by approximately 4 kJ mol^{-1} over 8 and 9 which are both very similar. In the C_2Et_2 clusters, 4-6, the NiCoFe cluster also had the highest $\delta G^{\ddagger}$ value since the methylene hydrogens did not exchange on the timescale of the saturation experiments but those of 8 and 9 did. However, this observation could not be quantified.

FIGURE 9

Coalescence of the Methyl Resonances

in the ^1H NMR (Toluene-d_8) of

$CpNiCoFe(CO)_6(MeC_2Ph)$ (7)

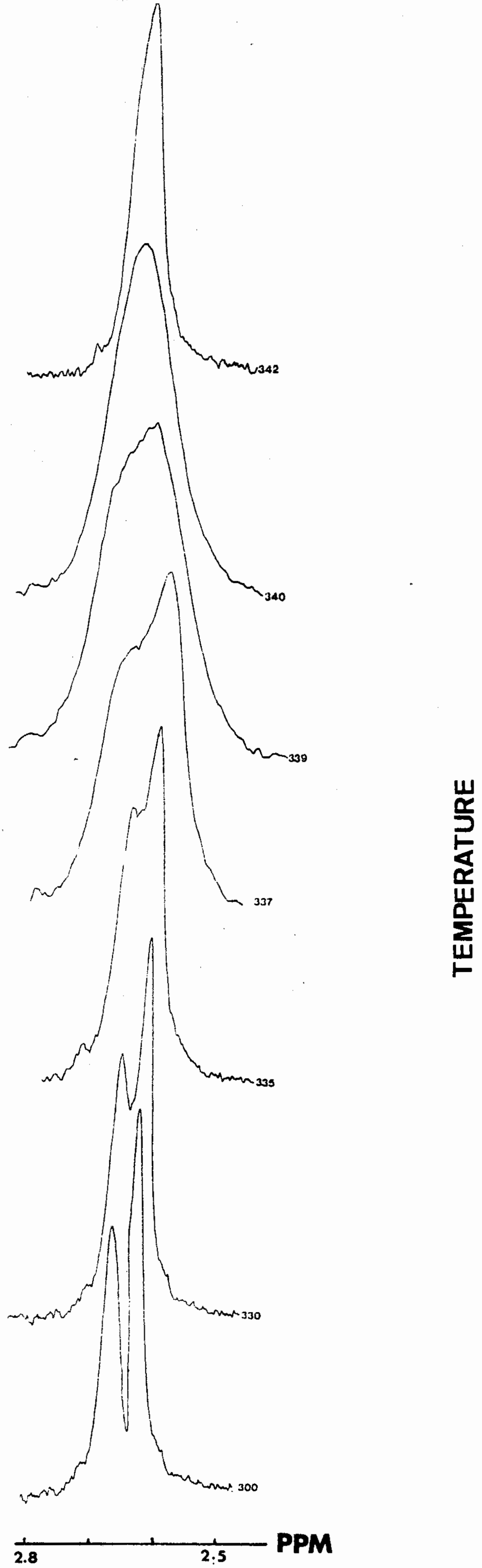

342
340
339
337
335
330
300
TEMPERATURE
2.8
2.5
PPM

Table 5. Coalescence Data for 7, 8 and 9

Cluster	T_c (K)[a]	Δ_{243}K	$\Delta G^{\ddagger}$ (kJ mol^{-1})[b]	K_{AB} (s^{-1})[c]	K_{BA} (s^{-1})[c]
7, Me	339	11.18	74.3	30.8	18.9
8, Me	321	13.97	69.6	47.8	14.3
Cp	330	20.57	70.5	70.4	21.0
average			70.1		
9, Me	315	7.15	70.0	19.1	12.7
Cp	327	18.58	70.1	47.8	34.7
average			70.1		

[a] All experiments in toluene-d_8; ± 1 K.
[b] ± 1.3 kJ mol^{-1}
[c] ± 1.1 s^{-1}

1.3.2d X-ray Structures

The X-ray structures were determined for four of the heterotrimetallic alkyne clusters, specifically 1a, 2, 3 and 4. The X-ray data collection parameters and refinement are in the Experimental Section along with the tables of bond lengths and angles, atom coordinates and temperature factors but a compilation of the more important bond lengths and angles are in Tables 6 and 7 for easy comparison. The labelled ORTEP and SNOOPI diagrams for all four cluster alkynes are shown in Figures 10, 11, 12 and 13 respectively.

All four crystal structures consist of discrete molecules with no unusually short inter- or intramolecular contacts. All the compounds have a bonded triangle of metal atoms (NiCoFe, NiCoRu or NiCoOs) which are capped by a diethyl or diphenylacetylene ligand bonded as a μ_3-(η^2-$|$) group. A cyclopentadienyl ligand is η^5 coordinated to the Ni atom in each structure. 1a, 2 and 3 have a semi-bridging carbonyl between the Co atom and the Fe, Ru or Os atom while 4 has only terminal carbonyl groups. In addition, 1a has a triphenylphosphine ligand bonded to the cobalt.

Following is a comparison (with some discussion) of the four clusters with respect to the metal triangle, alkyne and carbonyl coordination. A general overview of the structures with regard to other known structures and to the IR and NMR data is given in the Discussion Section.

Metal Triangles

As shown in Table 6, the Ni-M and Co-M bond length increases in the order 1a < 2 < 3, or Fe < Ru < Os, as expected although the difference between the Ru-M and Os-M lengths is small (0.019(2)Å). The Ni-Co distances

FIGURE 10

ORTEP Diagram of

$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (<u>1a</u>)

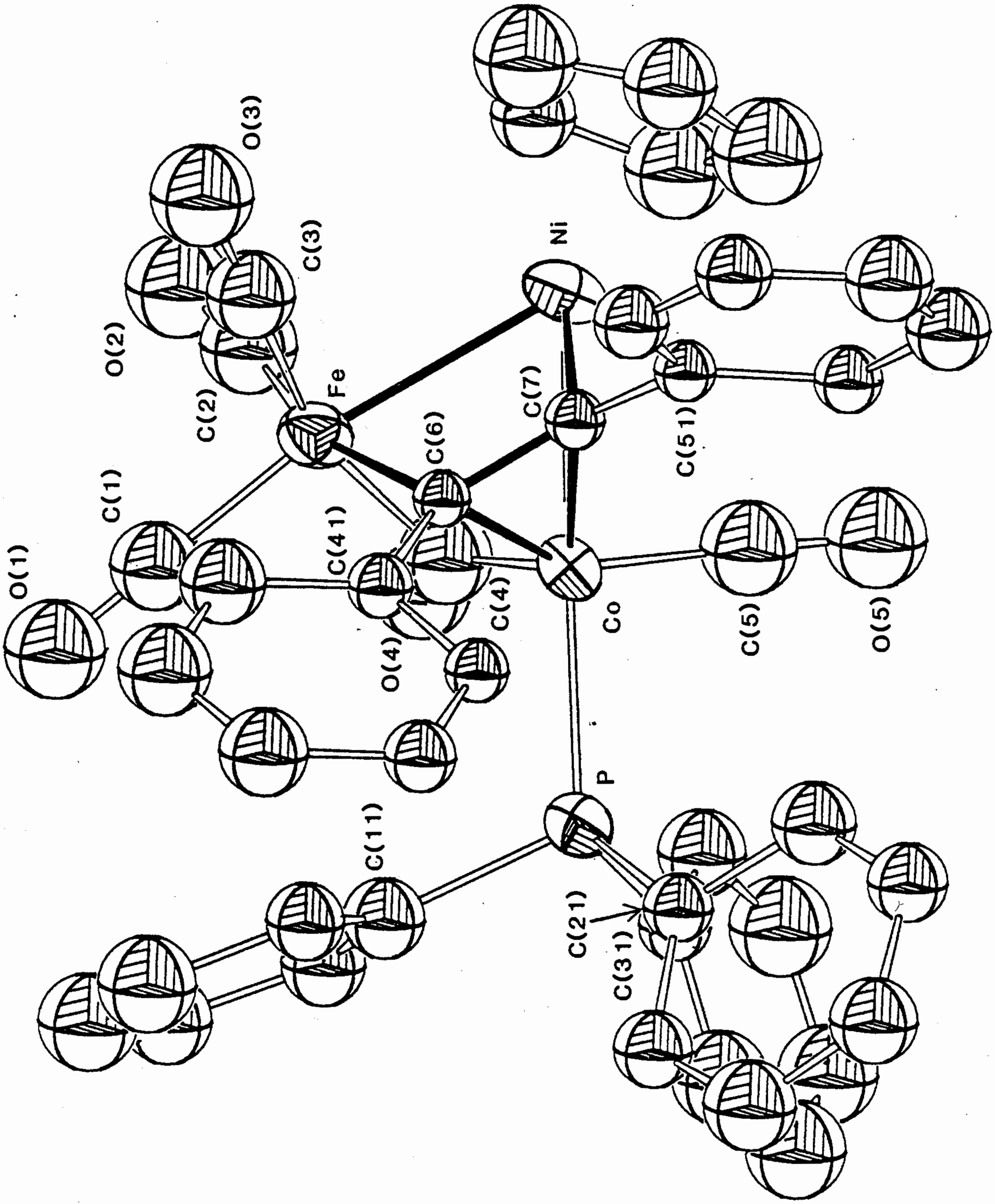

O(1)
C(1)
C(2)
O(2)
C(3)
O(3)
Fe
C(41)
C(6)
C(7)
Ni
O(4)
C(4)
Co
C(51)
C(5)
O(5)
P
C(11)
C(21)
C(31)

FIGURE 11

ORTEP Diagram of

$CpNiCoRu(CO)_6(C_2Ph_2)$ (2)

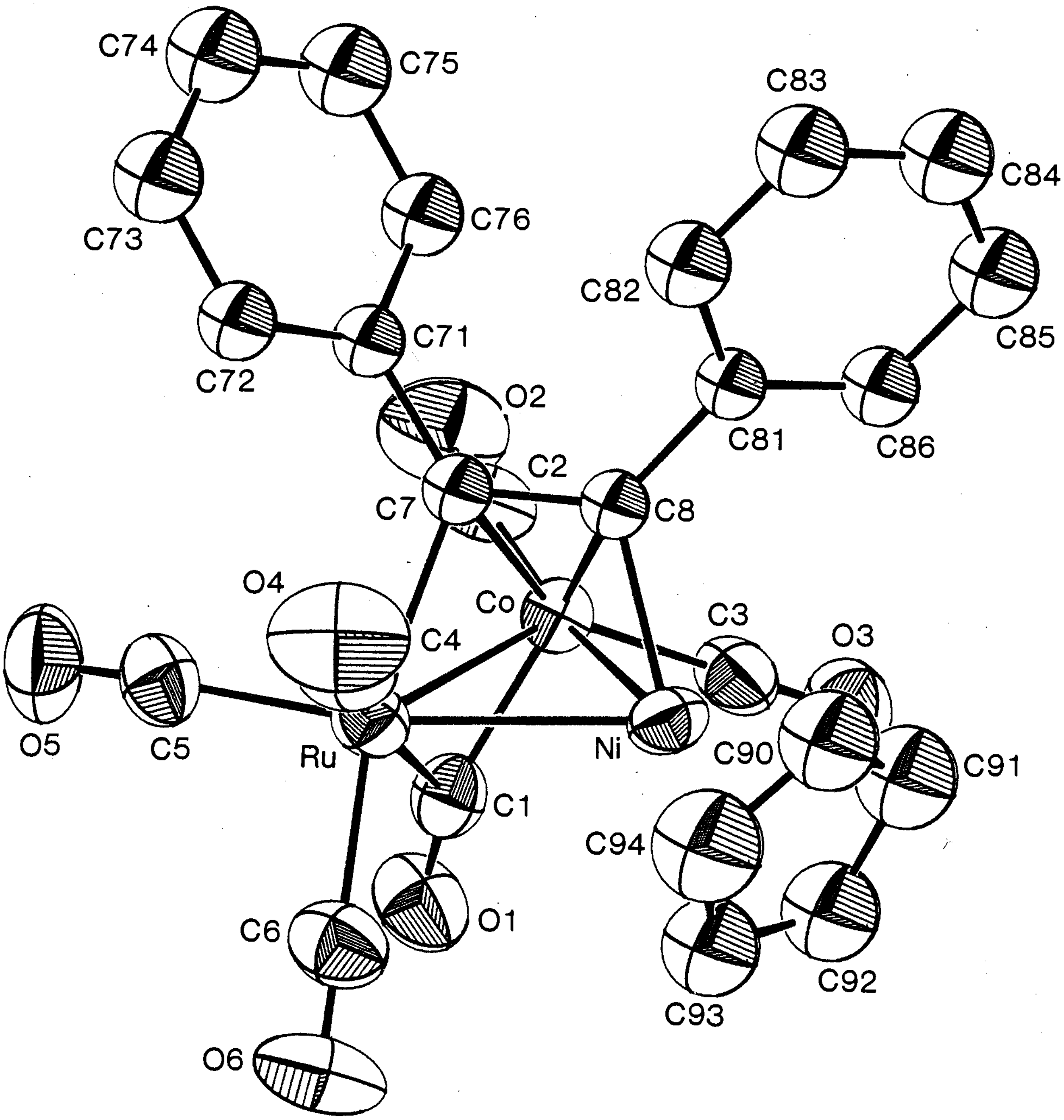

C74
C75
C83
C84
C73
C76
C82
C85
C72
C71
O2
C81
C86
C2
C7
C8
O4
Co
C3
O3
C4
O5
C5
Ru
Ni
C90
C91
C1
C94
O1
C6
C92
C93
O6

FIGURE 12

SNOOPI Diagram of

$CpNiCoOs(CO)_6(C_2Ph_2)$ (<u>3</u>)

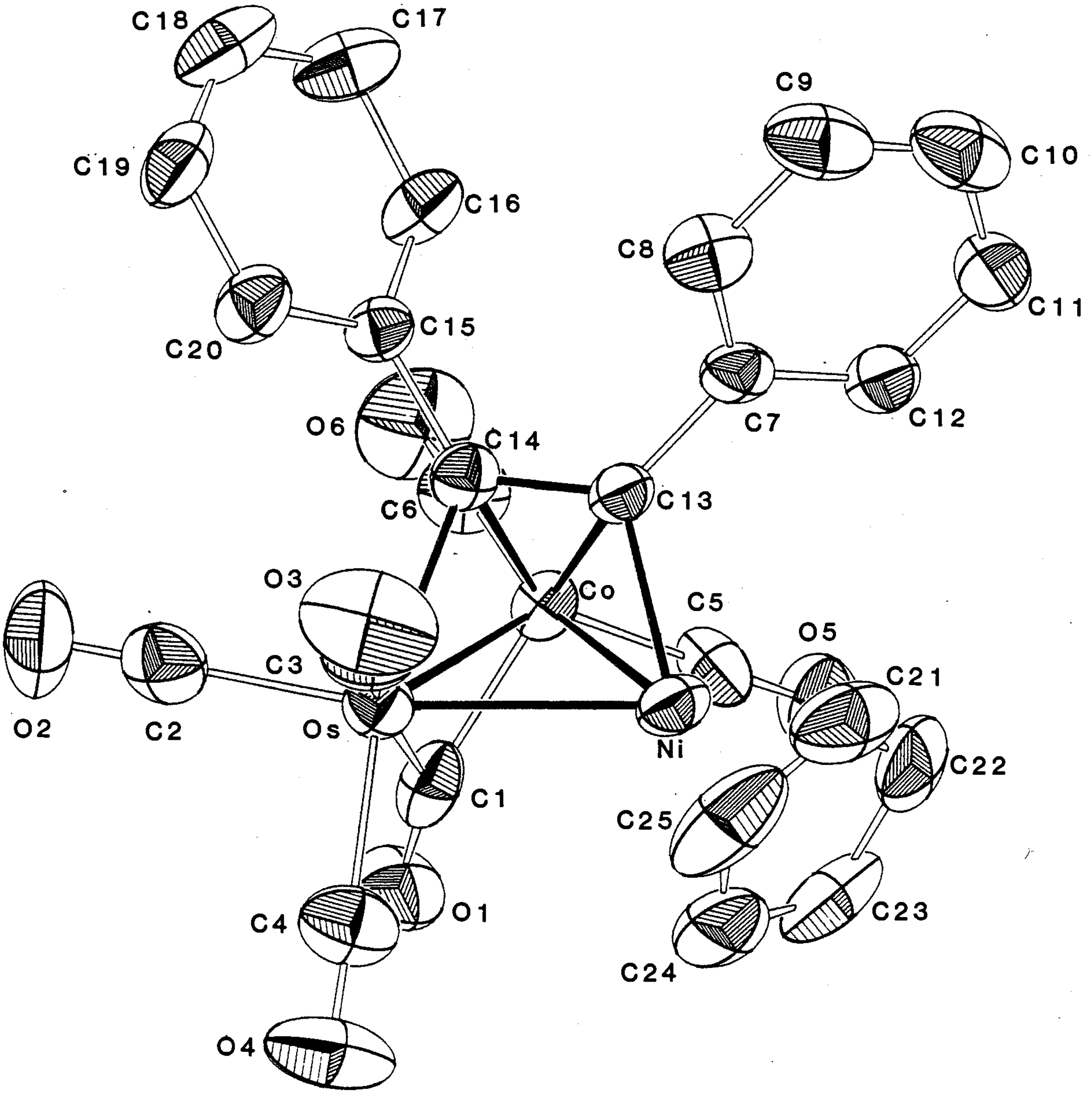
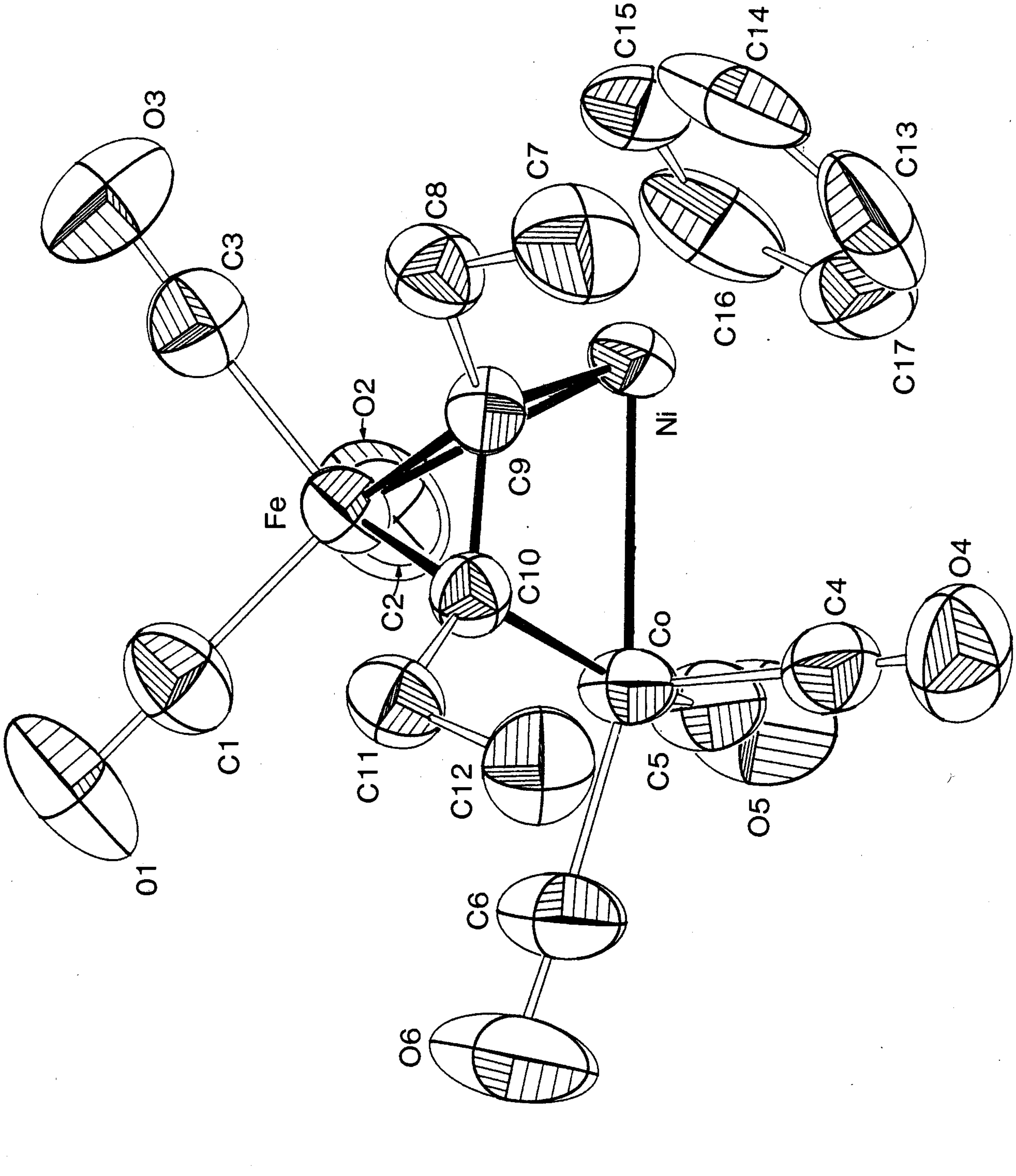

C18
C17
C9
C10
C19
C16
C8
C11
C20
C15
C7
C12
O6
C14
C13
C6
O3
Co
C5
O5
C21
C3
O2
C2
Os
Ni
C22
C1
C25
C4
O1
C23
C24
O4

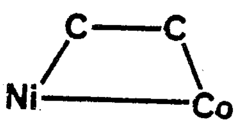
FIGURE 13

SNOOPI Diagram of

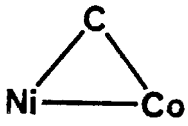
$CpNiCoFe(CO)_6(C_2Et_2)$ (4)


O3
C3
O1
C1
Fe
O2
C2
C8
C7
C9
C10
C11
C12
Ni
Co
C5
O5
C4
O4
C6
O6
C15
C14
C13
C16
C17

are very similar in 1a, 2 and 3 but they are all substantially shorter than in 4. While this may be the result of a different bridging alkyne ligand, C_2Et_2 in 4 versus C_2Ph_2 in 1a, 2 and 3, it is more likely a consequence of the different alkyne coordination with respect to the Ni-Co bond. In 1a-3 the alkyne is coordinated with respect to the Ni-Co bond as in I-14a but for 4 the orientation of the alkyne is represented by I-14b. Churchill[32] has previously noted a difference in metal-metal bond lengths due to this dissimilarity in alkyne coordination, although our difference of 0.033-0.042(5)Å is considerably smaller than recorded (0.12-0.14(3)Å) by Churchill for $Cp_2W_2Os(CO)_7[C_2(C_6H_4Me)_2]$. This difference in alkyne coordination with respect to a particular metal-metal bond also explains the large difference in distances for the Ni-Fe bond lengths of 1a and 4 (2.486(4) versus 2.402(1)Å).



I-14a



I-14b

In 1a, a triphenylphosphine ligand has been substituted for one of the carbonyl groups on the Co atom. The Co-P bond is essentially colinear (177(1)°) with the Co-Ni bond. In all the other structures it is noted that there is a carbonyl on cobalt in a similar orientation and the other terminal CO groups are in similarly related positions in all structures. The presence of the PPh_3 ligand has not disturbed the orientation of the remaining carbonyls, nor does there appear to be any effect on the Ni-Co bond length relative to 2 and 3.

Table 6. Comparison of Bond Lengths and Angles for 1a, 2, 3 and 4

Cluster		Bond Lengths (Å)				Bond Angles (deg)		
		Ni-Co	Ni-M	Co-M	C≡C	Ni-Co-M	Ni-M-Co	M-Ni-Co
$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$	(1a)	2.390(4)	2.486(4)	2.467(4)	1.34(2)	61.6(1)	57.70(9)	60.7(1)
$CpNiCoRu(CO)_6(C_2Ph_2)$	(2)	2.399(1)	2.560(1)	2.565(1)	1.377(9)	61.98(4)	55.82(4)	62.21(4)
$CpNiCoOs(CO)_6(C_2Ph_2)$	(3)	2.394(1)	2.579(1)	1.570(1)	1.377(10)	62.49(4)	55.41(3)	62.10(4)
$CpNiCoFe(CO)_6(C_2Et_2)$	(4)	2.432(1)	2.402(1)	2.494(1)	1.371(7)	58.36(3)	59.52(3)	62.11(3)

The cyclopentadienyl ring is displaced on the same side of the NiCoM plane in all four structures. The dihedral angle between the cyclopentadienyl plane and the trimetallic plane is 71.8(5), 69.6(3), 70.3(3) and 68.8(4)° in 1a, 2, 3 and 4 respectively. This and the similar Ni-Cp(center) distance of 1.75(1), 1.747(1), 1.755(1) and 1.750(4)Å reveal a negligible effect of the different alkyne bonding modes or of different metals in the cluster on the Ni-Cp distance or on its disposition relative to the metal triangle.

Alkyne Coordination

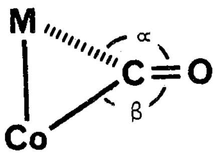
The interaction of the alkyne ligand with the three metal atoms occurs through a deformation of the alkyne fragment to an olefinic-like coordinating group forming two σ-bonds and one π-bond to the metal atoms. The formal 'triple-bond' of the alkyne has been found to range between 1.34(2)-1.377(10)Å in all 4 clusters, and is similar to the distance found for a double bond in an olefin. Similar lengthening has been noted in all (homo- and heterometallic) the μ_3-(η^2-||) bonded clusters, and examination of our data and the lengths provided by Sappa[24] does not provide any rationalization of the degree of lengthening with respect to the different metals present, even when the comparison is restricted to diphenylacetylene complexes. For example, $Os_3(CO)_{10}(C_2Ph_2)$ was found to have a C≡C distance of 1.439(10)Å, that is the longest 'triple bond' distance found, but one of the shortest is also a triosmium cluster, $Os_3(CO)_7(C_2Ph_2)_2$, having a C≡C distance of 1.33Å. It appears that factors more severe than just changing one metal atom as we have in 1a-3, are required to substantially change the length of the olefinic-type bond.

The alkyne has been found always to be symmetrically π-bonded to the appropriate metal atom although the Fe–alkyne distances in 4 are slightly shorter than the Co–alkyne lengths found in 2 and 3. A comparison with 1a is inconclusive because of the large standard errors present in the bond lengths.

Although the alkyne is π–bonded to the Fe atom in 4 and to the cobalt in 1a, 2 and 3, the similarity of the alkyne bonding to the metal triangle is most revealing when the dihedral angle between the trimetal plane and the plane formed by the NiRuC(6)C(7) atoms in 2 and equivalent planes in 1a, 3 and 4 is considered. This dihedral angle is 57.8(3), 57.6(1), 57.3(1) and 57.1(1)° in 1a, 2, 3 and 4, respectively. Dahl[31] has recorded similar dihedral angles of 56.5(2) and 56.0(2)° for $Cp_3Rh_3(CO)(C_2Ph_2)$ and $Cp_3Rh_3(CO)(C_2(C_6F_5)_2)$, compounds with only second row transition metals. The latter has a powerful electron withdrawing fluoro–alkyne ligand. This nearly constant dihedral or 'inclination' angle displayed for varying structures is probably a consequence of the alkyne ligand finding the optimum geometry for π–donation to, and π*–acceptance from, the corresponding metal atom (see Discussion).

Carbonyl Coordination

One of the more notable differences in these four structures is the presence (or absence) of a semi–bridging carbonyl ligand. Only compound 4 does not have this type of ligand. The relevant bond lengths and angles of the bridging species are summarized in Table 7 along with a recently reported and closely related structure, $Co_2Ru(CO)_9(C_2Ph_2)$[48] which also has a semi–bridging carbonyl. The two angles in the table, α and β, are defined as below as in I–15 and are consistent with those described by Cotton and



I–15

Wilkinson[52].

The M-C bond length increases for Fe < Ru < Os within our series of clusters <u>1a</u>-<u>3</u> but the Co_2Ru cluster[48] has a substantially longer Ru-C distance. This longer M-C length would suggest that in this compound the carbonyl ligand is not as distorted from linearity as is the bridging carbonyl in <u>1a</u>, <u>2</u> or <u>3</u>. The listed α angles substantiate this observation; α is smaller for <u>2</u> and <u>3</u> than for the Co_2Ru cluster (but comparison with <u>1a</u> is of limited value because of the larger standard errors).

The Co-C bond lengths also increase as Fe < Ru < Os and both <u>2</u> and the Co_2Ru cluster have similar values suggesting the difference noted for the Ru-C bond length is due to the replacement of the $Co(CO)_3$ group by the isoelectronic and isolobal CpNi unit. However, differences in M-C or any bond length associated with the semi-bridging carbonyl ligand are likely to involve very small energy changes comparable to other factors such as packing forces.

Table 7. Comparison of Bond Lengths and Angles for the Semi-bridging Carbonyl

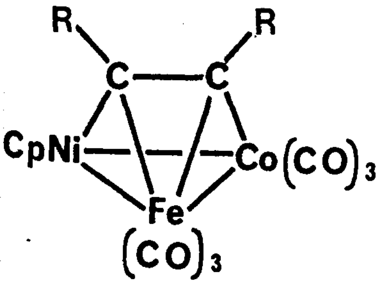
Cluster		Bond Lengths (Å)				Bond Angles (deg)	
		Co-M	Co-C	M-C	C-O	α	β
$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$	(1a)	2.467(4)	1.74(2)	2.07(1)	1.19(3)	148(1)	131(1)
$CpNiCoRu(CO)_6(C_2Ph_2)$	(2)	2.565(1)	1.828(8)	2.251(8)	1.158(9)	148.2(7)	134.5(6)
$CpNiCoOs(CO)_6(C_2Ph_2)$	(3)	2.579(1)	1.853(8)	2.273(8)	1.148(10)	147.3(7)	136.2(7)
$Co_2Ru(CO)_9(C_2Ph_2)$[48]		2.5865(5) 2.6875(5)	1.820(3)	2.284(3)	1.1498(5)	149.9(1)	132.8(1)

1.4 Discussion

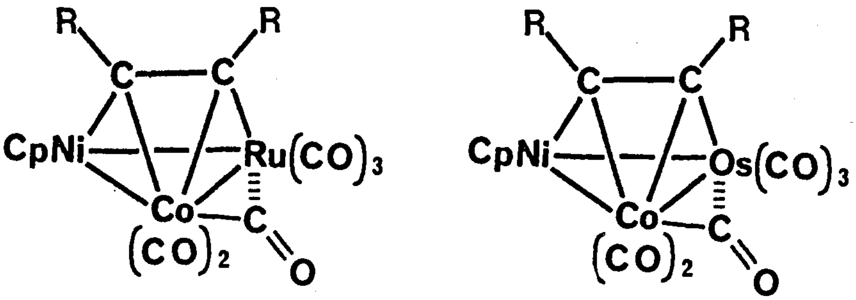
The synthesis of heterotrimetallic (MM'M") alkyne clusters of the formula $CpNiCoM(CO)_6(RC_2R')$ (M = Fe, Ru, Os: R = R' = Ph, Et; R = Ph, R' = Me), 1-9 has been achieved. We are not aware of any reports on similar series where one metal of the trimetallic framework has been systematically varied down a given triad, and this has been accomplished for three different alkyne ligands. The X-ray structures of four compounds 1a, 2, 3 and 4 have been completed and these solid state structures will be discussed next.

1.4.1 Solid State Structures

All of the clusters which have a Ru or Os atom in the trimetallic framework reveal the presence of a doubly bridging CO in the solid state IR, which is confirmed by the X-ray structures of 2 and 3. Where this semi-bridging CO is present, the alkyne ligand is found to π-bond with the Co atom and σ-bond with the Ni and Ru or Os atom. This structure is represented by I-17. Compounds where the Ru or Os has been replaced by an Fe atom all have their structures as shown by I-16 where the alkyne ligand



I-16



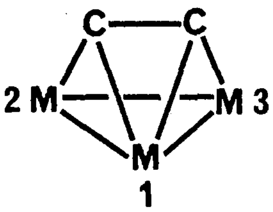
I-17

is π-bonded to the Fe atom and σ-bonded to the Ni and Co atoms. This is the arrangement found in the X-ray structure of 4. Only absorptions due to terminal carbonyl ligands are observed in both solid state and solution IR. The phosphine-substituted complex 1a, is an exception since only Ni, Co and Fe atoms are present but a bridging CO is found and this compound will be discussed later.

Since all the NiCoRu and NiCoOs clusters have the alkyne oriented as in I-17 regardless of the substituents on the alkyne (Ph, Me, Et), this suggests that minor changes in these substituents does not drastically alter the cluster-alkyne interaction. However, changing the Ru or Os for an Fe atom appears to affect the ground state structure and the alkyne has been rotated 60° on the trimetallic face.

Very recently McGlinchey et. al.,[53] have postulated that there should be a preference for certain orientations of an alkyne ligand in heterotrimetallic (M_2M' and $MM'M''$) clusters. However, experimental evidence for this was largely based upon observed structures that are generally of the M_2M' type. There has not been a systematic variation of metal atoms within the trimetallic framework until this report.

Some of the relevant arguments and conclusions put forward by McGlinchey et. al. will be briefly reviewed here but the reader is referred to the original paper[53] for more detailed calculations. The following discussion will revolve around the heterotrimetallic core which is labelled as shown below, as I-18 with the orientation of the alkyne as indicated.



I-18

The alkyne ligand was shown to behave as an overall donor of electron density towards the trimetallic framework through its π orbital. Thus the ability of the M_3 unit to accept electron density is crucial, and the positions labelled 2 and 3 were found to accept electron density more than the fragment in position 1. It is argued that the more electron-accepting fragments will prefer positions 2 and 3 since this will provide the most favourable interactions with the alkyne and thus form the stronger bonds. When the complex possesses different metal fragments, the acceptor orbitals are localized mainly on the more electronegative atoms(s). The preferred rotamer will then be the one with the more electronegative atom(s) occupying postions 2 and/or 3. In the solid state the ligands can be rearranged such that each metal obtains an 18 electron count. It should be stressed here that the authors did not consider varying atoms of the same triad in their trimetallic framework, but were generally concerned with only the 3d metals and the impact of different ligand (Cp, CO), and hence slightly different electronegativities, on the alkyne orientation. It is likely, however, that similar MO arguments will prevail with the 4d and 5d metals as with the 3d metals. This line of reasoning can be extended to help to explain our observed solid state structures.

Consideration of the Pauling electronegativities[54], 2.20 for Ru and Os and 1.81 for Fe, 1.83 for Co and 1.91 for Ni, allows predictions to be made, based upon these arguments. When the trimetallic framework consists of NiCoRu or NiCoOs atoms the Ru or Os will prefer position 2 since these atoms are the most electronegative. The Ni atom has a Cp ligand bonded to it and is formally Ni(I), making it substantially more electronegative than the Co atom and it should occupy position 3 in the triangle. This would mean that the Co atom must then be in position 1. If all the CO groups are terminal, this results in electron counts of 17(Os), 18(Ni) and 19(Co). To maintain an 18 electron count at each metal atom, a semi-bridging carbonyl ligand between the Co and Ru/Os is formed. This is the orientation of the metal atoms relative to the alkyne ligand that is indeed found in the X-ray structures of 2 and 3 and postulated for the other NiCoRu and NiCoOs clusters.

Where the trimetallic framework consists of NiCoFe atoms, the Ni atom should occupy either position 2 or 3. The Co atom has a slightly greater electronegativity than that of Fe and would be expected to occupy position 2 or 3 leaving position 1 for the Fe atom. However, it should be noted that the difference in electronegativities for Co and Fe is very small and other factors such as different M–C(alkyne) bond strengths may influence the choice of position of the metal atom on the triangle.

The structure of 1a (Figure 10) appears to be an exception as there is a semi-bridging CO present even though this is a NiCoFe cluster. However, there are two possible reasons for this anomaly. The first is that the PPh_3 ligand renders the cobalt atom less electronegative than the Fe atom (and of course, the Ni atom) and will then prefer position 1. This is the reason forwarded by McGlinchey et. al[53]. However, it is also possible that the steric bulk of the PPh_3 ligand may be incompatible with having the phenyl group of the alkyne ligand in close proximity. This steric interference is relieved when the alkyne rotates by 60° and becomes σ-bonded to the iron atom. A terminal CO becomes semi-bridging to achieve an 18 electron count at each metal atom. It is not possible to state at this point which of these two factors is responsible for the observed orientation or indeed whether both factors are partly responsible.

Earlier, in the X-ray characterization section, various aspects of each structure were examined and comparisons between them were made. The focus will now be on the similarities and differences between all the relevant structurally characterized heterotrimetallic clusters. Bond lengths of these clusters are given in Table 8.

Direct comparison between these clusters is often difficult because of the different metals involved; however, trends can often be established. The compounds $FeCo_2(CO)_9(C_2Et_2)$[55], $CpNiCoFe(CO)_6(C_2Et_2)$ and $Cp_2Ni_2Fe(CO)_3(C_2Ph_2)$[46] provide a series where there has been stepwise replacement of a $Co(CO)_3$ by CpNi. All have the alkyne oriented as in I-16, shown earlier, with the Fe atom in position 1. As expected the bond length is found to decrease in the order; Co-Co > Co-Ni > Ni-Ni, (2.576(1), 2.432(1) and 2.404(4)Å), since the Co(0) is being replaced by a Ni(I) atom. The decrease is not orderly, however. The largest difference occurs upon replacement of one Co and a much smaller difference is noted when the second Co is replaced by a Ni atom.

Little difference was found for the C≡C lengths of the alkyne ligand for different structures. These values are not listed in Table 8 as they only varied between 1.34(2)Å and 1.40(3)Å with the majority centered around 1.37Å.

1.4.2 Solution Structures

The ^1H NMR of 1-3 exhibit only one Cp resonance and a multiplet due to the phenyl protons of the diphenylacetylene ligand. However information can be obtained regarding the dynamics of these heterotrimetallic alkyne systems by studying the diethyl- and phenylmethylacetylene substituted derivatives. In particular, irradiation of the resonance due to a particular methylene hydrogen revealed that exchange was occurring with a corresponding hydrogen

Table 8. Comparison of Bond Lengths in Related Heterotrimetallic Alkyne Clusters

Cluster	Bond Lengths			Reference
	Ni-Co	Ni-M	Co-M	
$CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$*	2.390(4)	2.486(4)	2.467(4)	a, 56
$CpNiCoRu(CO)_6(C_2Ph_2)$*	2.399(1)	2.560(1)	2.565(1)	a
$CpNiCoOs(CO)_6(C_2Ph_2)$*	2.394(1)	2.579(1)	2.570(1)	a
$RuCo_2(CO)_9(C_2Ph_2)$*	Co-Co 2.4535(5)		2.5685(5) 2.6875(5)	48
$CpNiCoFe(CO)_6(C_2Et_2)$	2.422(1)	2.402(1)	2.494(1)	a
$CpNiCoFe(CO)_6(PhC_2\text{-}i\text{-}CO_2Pr)$	2.422	2.423	2.487	25, 57
$FeCo_2(CO)_9(C_2Et_2)$	Co-Co 2.576(1)		2.479(1) 2.489(1)	55
$Cp_2Ni_2Fe(CO)_3(C_2Ph_2)$	Ni-Ni 2.404(4)	2.388(4) 2.381(4)		58
$CpNi_2Ru(CO)_3(C_2Ph_2)$	Ni-Ni 2.418(4)	2.493(3) 2.496(3)		58
$[CpNiFe_2(CO)_6(C_2Ph_2)]^-$	Fe-Fe 2.506(1)	2.453(1) 2.474(1)	Fe-Fe 2.506(1)	40

[a] This work.

* Has bridging carbonyl.

of the second methylene group but none between CH protons on the same carbon atom. This was clearly seen at ambient temperatures for 5 and 6 but not for 4 as the exchange rate here was too slow at this temperature. This clearly rules out racemization of the clusters since this process equilibrates hydrogens on the same carbon atoms only, and demonstrates that no transfer of the alkyne from the front to the back face of the cluster can be occurring. For the phenylmethylacetylene substituted clusters 7-9 two methyl resonances were observed at room temperature in unequal intensities which coalesced upon warming the sample. This demonstrates exchange of groups on the alkyne ligand but does not reveal any information regarding front to back transfer of the alkyne ligand on the trimetallic face.

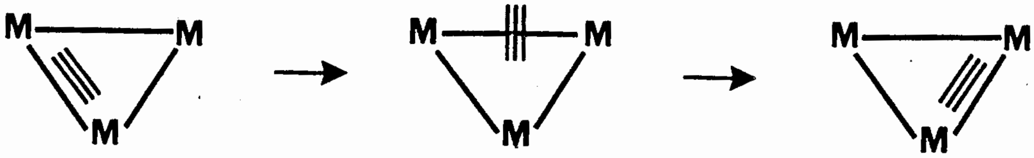
The observed fluxionality of the alkyne ligand is likely a consequence of this ligand rotating on one face of the heterotrimetallic core. It would account for the exchange of hydrogens on separate methylene groups for 4-6 and for the collapse of the methyl peaks in the NMR of 7-9. Rotation of an alkyne ligand about a trimetallic face has been noted before (see Table 9) but there are still not many examples known, or activation energies that have been determined.

In a study of $H_2Ru_3(CO)_9$(cyclooctyne) and the osmium analoge, Deeming[61] obtained activation energies of 59.8 and 70.7 kJ mol^{-1}, respectively, and suggested that exchange of the alkyne ligand resulted from a pivotal arrangement (shown in Scheme I-4) with respect to the triangular metal face through the intermediacy of the closo configuration. Extending these 60° rotations around all three vertices of the triangle, the end result will be a rotation of the alkyne ligand, including exchange of diasterotopic methylene protons. Each metal is successively σ-or π-bonded to the alkyne ligand. The alternative approach, Scheme I-5, relates the fluxionality of

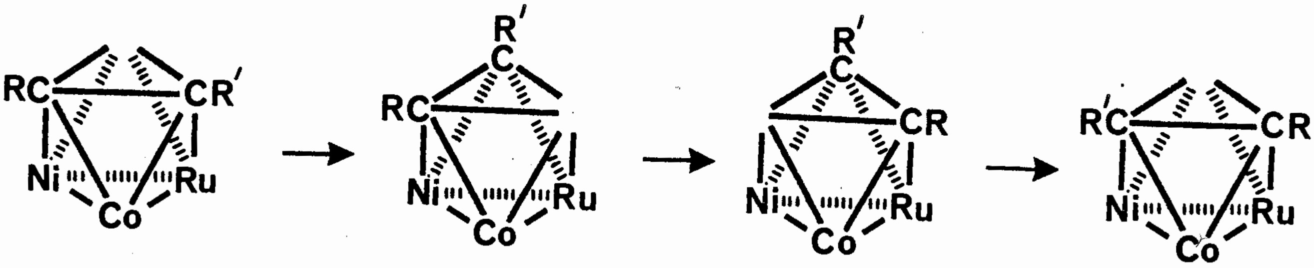
Table 9. Fluxional Trimetallic Alkyne Clusters

Cluster	ΔG‡ (kJ mol^{-1})	Reference
$Os_3(CO)_7(PMe_2)_2(C_6H_4)$	58.0	59
$Os_3(CO)_7(AsMe_2)_2(C_6H_4)$	51.3	59
$HOs_3(CO)_9(PMe_2)(C_6H_4)$	56.8	59
$HOs_3(CO)_9(AsMe_2)(C_6H_4)$	58.0	59
$Cp_3Rh_3(CO)(C_2Ph_2)$	-	60
$Cp_3Rh_3(CO)(C_2(C_6F_5)_2)$	-	60
$H_2Os_3(CO)_9(\mu_3$-indyne)	60(2)	61
$Os_3(CO)_7(AsMe_2)_2(C_6H_3$-iPr)	-	62
$Cp_2Ni_2Fe(CO)_3(PhC_2$-CO_2-iPr)	63(2)	25,57
$Cp_2Ni_2Fe(CO)_3(PPh_3)(PhC_2$-$CO_2$-iPr)	73(2)	25,57
$CpNiCoFe(CO)_6(C_2Et_2)$ (<u>4</u>)	74(1)	a
$CpNiCoFe(CO)_6(PhC_2Me)$ (<u>7</u>)	74(1)	a
$CpNiCoRu(CO)_6(PhC_2Me)$ (<u>8</u>)	70(1)	a
$CpNiCoOs(CO)_6(PhC_2Me)$ (<u>9</u>)	70(1)	a

[a] This work.



SCHEME I-4



SCHEME I-5

nido octahedra possessing one vacant site[57]. This view holds that the fluxionality is effectively the result of a migration of the vacant site over the octahedral surface. As shown in the sequence in Scheme I-5, a migration of the vacant site around one face of the octahedron by successive interchanges with a neighbouring vertex in the same sense equally results in the observed exchange of the alkyne substituents R and R'.

Both heating (to 30° above coalescence) and cooling (to 243K) compounds 7-9 did not result in any additional observable Cp or Me peaks which might have been attributable to intermediates of this rotational scheme. Similarly, cooling (to 220K) solutions of 4-6 did not result in extra Cp, CH_2 or CH_3 resonances. This implies that the intermediates have a low concentration or short lifetime. It is possible that this mechanism is not operable; if so, it is not clear what the nature of the actual mechanism could be. It is probably not dissociative, as no evidence of free alkyne was observed in any of the NMR solutions of the clusters nor was any exchange of alkynes noted between $CpNiCoFe(CO)_6(C_2Ph_2)$ and MeC_2Ph. In addition, because the chirality of the methylene protons of 4-6 is maintained, the alkyne must rotate only on one face of the core as migration to the opposite face would result in exchange of protons on the same methylene group. As shown in the Characterization section, the NMR's of 4-6 do not show this exchange of protons. Thus any postulated mechanism must not involve free alkyne or interconvert enantiomers. The carbonyl ligands are also fluxional at room temperature. The $^{13}C\{^1H\}$ NMR's of 1a and 5 showed only a single resonance attributable to the carbons of the CO. These were found at 209.3 and 193.2 ppm in these two compounds, respectively.

The ground state energy differences, ΔE, between the orientational isomers of 7-9 are the first to be calculated on this type of system.

Compound 7 (NiCoFe) shows the smallest ΔE, 0.57 kJ mol^{-1} and 8 (NiCoRu) the highest at 3.90 kJ mol^{-1}. All three compounds show similar solvent dependence in the preference for a particular conformation, but unfortunately the NOE experiment did not allow us to prove which set of signals belong to an individual orientation.

1.5 Conclusion

We have synthesized a series of heterotrimetallic alkyne clusters of general formula $CpNiCoM(CO)_6(RC_2R')$ (M = Fe, Ru, Os: R = R' = Ph, Et; R=Ph, R'= Me). This represents the largest complete series of heterotrimetallic clusters (MM'M") to be made. The alkyne ligand is shown by ^1H NMR to be fluxional in solution and in addition, rotation is occurring only on one face of the heterotrimetallic core. The solid state IR revealed that clusters where M = Fe had only terminal carbonyls but when M = Ru or Os a bridging carbonyl was present. This was confirmed by the X-ray crystal structures of four clusters, 1a, 2, 3 and 4. The bridging CO arises as the result of a different preferred orientation of the alkyne ligand with respect to the trimetallic core in the solid state. This has been interpreted in the light of a recent theoretical paper as a preference for the atom with the greatest electronegativity to be σ-bonded to the alkyne.

1.6 EXPERIMENTAL

$CpNiCo(CO)_3(C_2RR')$(R = R' = Ph, Et; R = Ph, R' = Me) and $Fe_2(CO)_9$ were prepared according to literature procedures[38,63]. $Ru(CO)_5$ was prepared by heating $Ru_3(CO)_{12}$ under 1500 psi of carbon monoxide. $Os(CO)_5$ was kindly supplied by Dr. R.K. Pomeroy of this department. Diphenylacetylene (Aldrich), 3-hexyne (Columbia Organic), phenylmethylacetylene (Aldrich) and PPh_3 (Aldrich) were used as received.

All solvents were dried and distilled over Na or Na/benzophenone or CaH_2 under nitrogen. All reactions were carried out in standard Schlenkware, connected to a switchable inert atmosphere/vacuum supply and were conducted under nitrogen.

Photochemical reactions were performed in a Pyrex vessel (~240 mL) equipped with a water cooled quartz finger. A 200 watt ultraviolet Hanovia high-pressure mercury source was placed inside the quartz finger. Nitrogen was passed through the reaction vessel prior to the addition of solvent and starting materials, and slow passage of nitrogen was maintained during the reaction.

Infrared spectra were recorded on a Perkin Elmer 983 spectrophotometer. ^1H spectra were recorded on a Brüker WM-100 or a Brüker WM-400 instrument at 100 and 400 MHz respectively. All Nuclear Overhauser Enhancement and spin saturation transfer experiments were performed on the Brüker 400. ^{13}C NMR were recorded on the Brüker WM-400 at 100 MHz. Mass spectral analyses were run by G. Owen on a Hewlett Packard 5985 system operating at 70eV. Microanalyses were performed by Mr. M.K. Yang of the microanalytical laboratory at Simon Fraser University.

The IR, ^1H NMR and mass spectral data are not documented in the experimental section but the reader is referred to the appropriate tables in the text.

1.6.1 Synthetic

Preparation of $CpNiCoFe(CO)_6(RC_2R')$ (1,4,7)

A large excess, (10 fold),of $Fe_2(CO)_9$ and the corresponding heterobimetallic alkyne compound, $CpNiCo(CO)_3(RC_2R')$ (R = R' = Ph, Et; R = Ph, R' = Me) (typically in the range of 0.1-0.2 g) were stirred overnight at room temperature in approximately 30 mL hexane. The solvent was removed *in vacuo*, 6-8 mL of fresh hexane was added, and the solution chromatographed on a 1" x 18" Florisil column. $Fe_3(CO)_{12}$ and the starting heterobimetallic alkyne were eluted with hexanes. Using a 5:1 hexanes/methylene chloride eluant, a dark green or light brown band was recovered and identified as the appropriate heterotrimetallic alkyne, $CpNiCoFe(CO)_6(RC_2R')$. Yields were variable but typically ranged from 30-60%.

The reaction of $Fe_2(CO)_9$ with $CpNiCo(CO)_3(C_2Ph_2)$ was used as a model. Warming the reaction mixture to 40-45°C and stirring overnight caused additional side-products to be formed. Some side products were made in a very small quantity to be eluted, and decomposed on the column. Two products formed in reasonable yields were $Cp_2Fe_2(CO)_4$ (IR, MS) and $Fe_3(CO)_9(C_2Ph_2)$ (IR, MS). The use of diethyl ether or THF rather than hexanes resulted in poorer yields of the desired heterotrimetallic complexes and with diethyl ether the formation of $Fe_3(CO)_9(C_2Ph_2)$ was especially favourable and often isolated in 30% yield. Additionally the amount of $Fe_3(CO)_{12}$ also increased with THF or diethyl ether as solvent.

Anal. Calcd. for $CpNiCoFe(CO)_6(C_2Ph_2)$ (1): C, 51.34; H, 2.58. Found: C, 52.06; H, 3.62. Calcd. for $CpNiCoFe(CO)_6(C_2Et_2)$ (4): C, 41.77; H, 3.09.

Found: C, 41.87; H, 3.12.

Preparation of $CpNiCoRu(CO)_6(RC_2R')$ (2, 5, 8)

In a typical reaction, $CpNiCo(CO)_3(RC_2R')$ (R = R' = Ph, Et; R = Ph, R' = Me) and excess $Ru(CO)_5$ were added to 25 mL hexanes and stirred at room temperature until the bands in the infrared spectrum due to $Ru(CO)_5$ (ν(CO) 2039, 2004 cm^{-1}) disappeared. This was usually about 18 hours. The reaction mixture could be warmed (40-45°C) safely to decrease the time required for the reaction to go to completion, but more $Ru_3(CO)_{12}$ was formed as a side product. After the reaction was complete, the solvent and any excess $Ru(CO)_5$ were removed by vacuum and the residue dissolved in the minimum amount of hexanes. Column chromatography with hexanes as eluant yielded $Ru_3(CO)_{12}$ as a yellow band followed by the starting heterobimetallic, $CpNiCo(CO)_3(RC_2R')$. The desired product, $CpNiCoRu(CO)_6(RC_2R')$ was then eluted as a brown band.

$^{13}C\{^1H\}$ (5), ($CDCl_3$, 293K); 193.2 (CO), 90.5 (Cp), 38.4, 36.9 (CH_2), 17.1, 16.7 (CH_3).

Anal. Calcd. for $CpNiCoRu(CO)_6(C_2Ph_2)$ (2): C, 47.66; H, 2.40. Found: C, 47.82; H, 2.51. Calcd. for $CpNiCoRu(CO)_6(C_2Et_2)$ (5): C, 38.24; H, 2.83. Found: C, 38.18; H, 2.79.

Preparation of $CpNiCoOs(CO)_6(RC_2R')$ (3, 6, 9)

$CpNiCo(CO)_3(RC_2R')$ (R = R' = Ph, Et; R = Ph, R' = Me) and excess $Os(CO)_5$ were added to 25 mL hexanes and heated to 40-45°C for 10-12 hours. The solvent volume was reduced to 5 mL and the solution was poured onto a Florisil column and chromatographed. Variable, but usually small, amounts of $Os_3(CO)_{12}$ were first eluted with hexanes followed by a green band identified as the starting heterobimetallic alkyne. Using a 5:1 hexane/chloroform mixture as eluant a dark brown band was next isolated and identified as $CpNiCoOs(CO)_6(RC_2R')$.

Anal. Calcd. for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3): C, 41.75; H, 2.10. Found: C, 41.79; H, 2.20. Calcd. for $CpNiCoOs(CO)_6(C_2Et_2)$ (6): C, 32.77; H, 2.43. Found: C, 32.87; H, 2.49.

Preparation of $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

Compound 1 (0.12 g, 0.21 mmol) and PPh_3 (0.07 g, 0.27 mmol) were added to 20 mL hexanes and stirred for 12 hours. The solvent volume was reduced to 4-5 mL and eluted on a Florisil column. Elution with methylene chloride/hexanes yielded a black band identified as 1a. No band due to 1 was visible although some decomposition had occurred.

Anal. Calcd for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$: C, 61.6, H, 3.7, Found: C, 62.2, H, 4.4. $^{13}C\{^1H\}$ ($CDCl_3$, 293K); 209.3 (CO), 156.2, 150.9 (acetylenic C), 133.8-125.4 (phenyl C), 92.8 (Cp).

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $M_3(CO)_{12}$ (M = Fe, Ru, Os)

$Fe_3(CO)_{12}$ (0.21 g, 0.42 mmol) and $CpNiCo(CO)_3(C_2Ph_2)$ (0.19 g, 0.41 mmol) was added to 30 mL of hexanes and refluxed for 18 hours. The solvent volume was reduced by vacuum and the trap was noted to contain some $Fe(CO)_5$. The residual hexanes was placed on a Florisil column and elution with hexanes yielded a small amount of $Fe(CO)_5$, a red-brown band identified as $Co_2(CO)_6(C_2Ph_2)$ and the starting heterobimetallic as a green band. Elution with 4:1 hexanes/methylene chloride gave $CpNiCoFe(CO)_6(C_2Ph_2)$ as a brown band, small and variable amounts of $Cp_2Ni_2Fe_2(CO)_6(C_2Ph_2)$ as a blue band (ν(CO) = 2010, 1973 cm^{-1}), $Cp_2Ni_2Fe(CO)_3(C_2Ph_2)$ as a green-brown band (ν(CO) = 2030, 1979 and 1963 cm^{-1}) and $Fe_3(CO)_9(C_2Ph_2)$ as a brown-black band. With only methylene chloride as the eluant some $Cp_2Fe_2(CO)_4$ was recovered.

$Ru_3(CO)_{12}$ (0.143 g, 0.22 mmol) and $CpNiCo(CO)_3(C_2Ph_2)$ (0.10 g, 0.225 mmol) were added to 20 mL hexanes and heated at 60°C for 16 hours. The solvent was removed by vacuum and the residue dissolved in fresh hexanes and chromatographed on Florisil. Only small amounts of 5 could be isolated.

$Os_3(CO)_{12}$ (0.20 g, 0.22 mmol) and $CpNiCo(CO)_3(C_2Ph_2)$ (0.2 g, 0.45 mmol) were added to 20 mL of hexanes and heated to 60°C for 16 hours. The solvent volume was reduced by vacuum and the residue chromatographed on a Florisil column. Only very small traces of 3 could be found.

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $Cr(CO)_5(THF)$

$CpNiCo(CO)_3(C_2Ph_2)$ (0.30 g, 0.68 mmol) was added to a THF solution of $Cr(CO)_5(THF)$ (0.5 g, 2.2 mmol) which was freshly prepared from the photolysis of $Cr(CO)_6$ in THF. The reaction was monitored after 1 hour and since no new carbonyl bands appeared, it was stirred overnight at ambient temperature. An infrared spectrum revealed carbonyl bands due to $CpNiCo(CO)_3(C_2Ph_2)$ (ν(CO) = 2060, 2014, 2008 cm^{-1}) and $Cr(CO)_6$. The solvent was removed by vacuum and the residue dissolved in 5 mL of hexanes/methylene chloride and chromatographed on Florisil. The infrared spectrum of the fractions revealed only $Cr(CO)_6$ and $CpNiCo(CO)_3(C_2Ph_2)$ being eluted. No new material could be isolated.

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $CpRe(CO)_2(THF)$

$CpRe(CO)_2(THF)$ (60 mg, 0.016 mmol) and $CpNiCo(CO)_3(C_2Ph_2)$ (70 mg, 0.16 mmol) were added to 25 mL hexanes and stirred at room temperature for 2 hours. An infrared spectrum of the solution revealed little remaining $CpRe(CO)_2(THF)$ ($\nu(CO)$ = 1925, 1857 cm^{-1}) but strong bands due to $CpRe(CO)_3$ ($\nu(CO)$ = 2031, 1940 cm^{-1}). The solvent was removed under vacuum and the residue dissolved in hexanes/methylene chloride. Chromatography on a Florisil column with hexanes as eluant yielded $CpRe(CO)_3$, then $CpNiCo(CO)_3(C_2Ph_2)$. With a stronger hexanes/methylene chloride eluant (3:1) a light yellow band was next recovered and identified as $CpRe(CO)_2(C_2Ph_2)$. This compound and its properties are discussed in Chapter 4.

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $Fe(CO)_4(PPh_3)$

$CpNiCo(CO)_3(C_2Ph_2)$ (51 mg, 0.113 mmol) and $Fe(CO)_4(PPh_3)$ (50 mg, 0.109 mmol) were added to 15 mL CH_3CN and stirred for 1 hour. As no reaction was apparent from monitoring the infrared spectrum, Me_3NO (20 mg) was added to the solution. After stirring for a further 1 hour, no new carbonyl bands in the IR were detected and the reaction was discontinued.

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $CpCo(CO)_2$

$CpNiCo(CO)_3(C_2Ph_2)$ (0.1 g, 0.225 mmol) and excess $CpCo(CO)_2$ (60 mg, 0.333 mmol) were added to 15 mL of hexanes and refluxed for 12 hours. The solution was cooled and the reaction mixture chromatographed on a Florisil column. Only $CpCo(CO)_2$ ($\nu(CO)$ = 2026, 1966 cm^{-1}) and $CpNiCo(CO)_3(C_2Ph_2)$ were identified. Some decomposition was indicated since material left at the top of the column could not be eluted with acetone or diethyl ether.

Reaction of $CpNiCoFe(CO)_6(C_2Ph_2)$ with MeC_2Ph

$CpNiCoFe(CO)_6(C_2Ph_2)$ (50 mg, 0.086 mmol) and excess methylphenylacetylene were added to 15 mL of hexanes and stirred for two hours. There was no apparent reaction as judged from an infrared spectrum and the reaction was discontinued.

Examples of the Calculation of $\Delta G^{\ddagger}$ and ΔE

a) Calculation of $\Delta G^{\ddagger}$ from Coalescence Temperature Experiments for 7

The $\Delta\nu_{\infty}$ value was the separation of the two Cp or two Me peaks in Hz at 243K. From this the rate constant could be evaluated.

$$\Delta\nu_{243}(\underline{7}) = 11.2 \pm 0.5 \text{ Hz}$$

$$K_c = \pi/\sqrt{2}\ \Delta\nu_{243} = 24.9 \pm 1.1 \text{ sec}^{-1}$$

By using the Eyring equation, the $\delta G^{\ddagger}$ value can then be calculated.

$$K_c = KT/h[e-(\Delta G^{\ddagger}/RT_c)]$$

$$K = \text{Boltzmann constant} = 1.3807 \times 10^{-16} \text{ ergs K}^{-1}$$

$$h = \text{Planck's constant} = 6.63 \times 10^{-27} \text{ ergs sec}$$

$$R = \text{gas constant} = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$$

$$T_c(\underline{7})(\text{methyl}) = 339 \pm 1\text{K}$$

then $\Delta G^{\ddagger} = 74.5 \pm 1.3 \text{ kJ mol}^{-1}$.

Additionaly, from the fractional populations of the two sites ρ_A and ρ_B, the rate constants k_{AB} and k_{BA} could be calculated.

$$\rho_A(\underline{7})(\text{methyl}) = 0.62$$

$$\rho_B(\underline{7})(\text{methyl}) = 0.38$$

where A represents the high field resonance. Then,

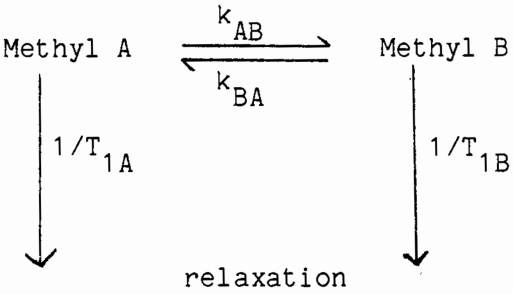
$$k_{AB} = 2(\pi/\sqrt{2})\Delta\nu_\infty(\rho_B) = 30.8\ \text{sec}^{-1}$$

and

$$k_{BA} = 2(\pi/\sqrt{2})\Delta\nu_\infty(\rho_A) = 18.9\ \text{sec}^{-1}$$

b) Calculation of $\Delta G^\ddagger$ by Spin Saturation Transfer Experiments for 4

A brief outline will be given. A selective 180° pulse is applied to a methyl resonance followed by a variable delay time and then a normal 90° observation pulse applied. The 180° pulse inverts the methyl resonance and the second methyl resonance will then decrease in intensity since it is undergoing exchange with the first, and now inverted, methyl peak. The difference spectrum (spectrum 1 - spectrum 2) is plotted and this shows the effect of only the selective pulse. Varying the delay time between the 180° pulse and the observation pulse allows a rate constant to be established for one temperature. An example of varying delay times is shown in Figure 14. The results can be easily seen with a simple kinetic scheme. The chemical



rates, k_{AB} and k_{BA} are equal and the two relaxation rates, $1/_{\rightarrow 1}{}_A$ and $1/T_{1B}$ are assumed to be equal or nearly so. If A and B represent the intensities of the two peaks, approximated by measuring the peak height of the center line of the triplet, then

$$(A + B) = A_0\exp-(1/T_{1A})t$$

and

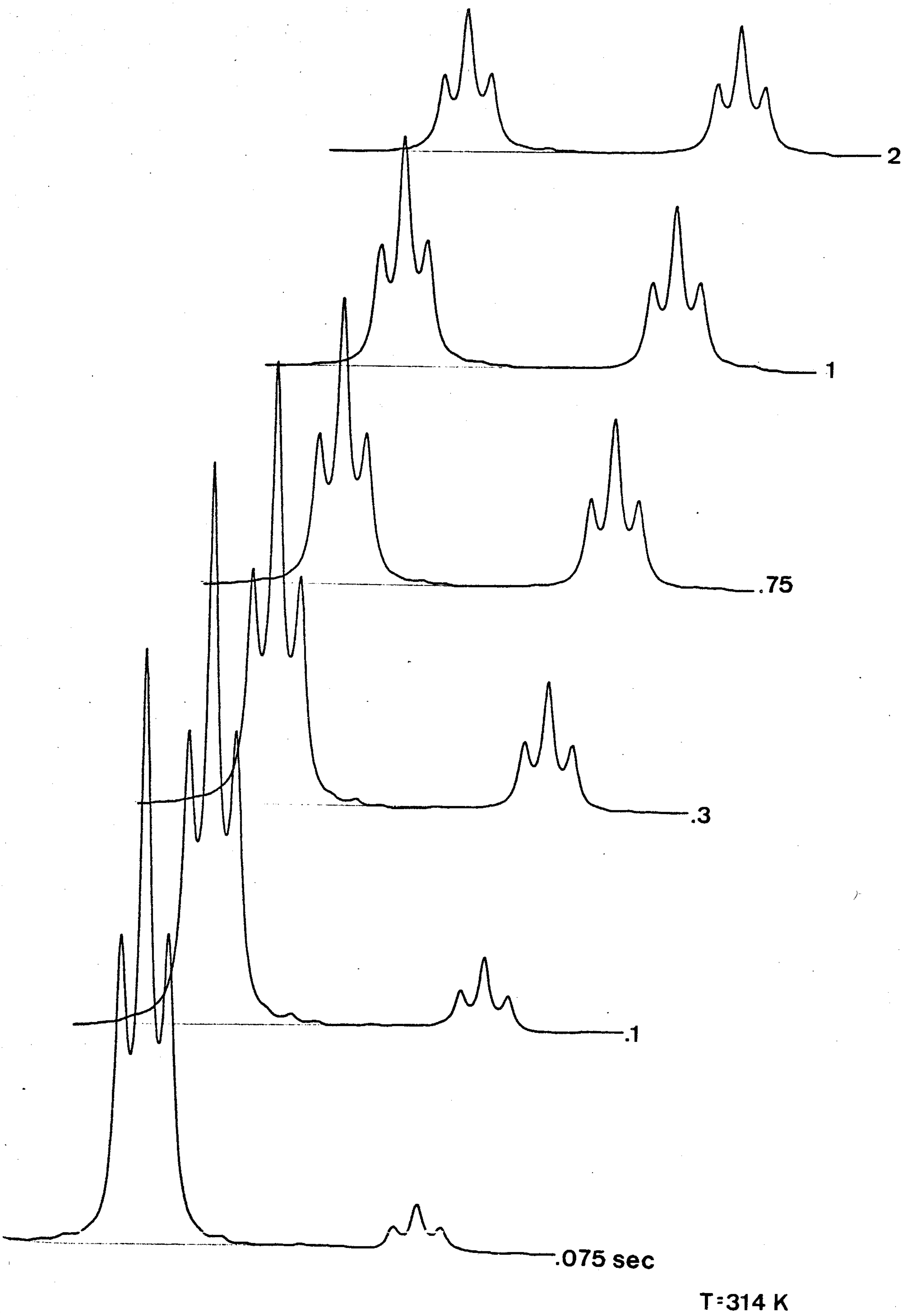
$$(A - B) = A_0\exp-(1/T_{1A} + k_{AB} + k_{BA})t$$

FIGURE 14

180° Pulse on the CH_3 Resonance

of $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

with Varying Delay Times



T=314 K

where A_0 is the intensity of the line A at time zero and t is the delay time. The sum (A + B) represents the total magnetization induced by the selective pulse and the difference (A - B) represents the equilibration of the excess magnetization between the two sites. The exchange rate constant between methyl group A and methyl group B is obtained by taking the difference between the sum and the difference totals. A plot of the k values obtained at different temperatures versus 1/T (Figure 15) gave a straight line of slope $-\Delta G^{\ddagger}/1.987$ from which the value of 74.5 ± 1.3 kJ mol^{-1} was obtained.

c) Calculation of ΔE for 7

From the population ratio of both isomers (^{1}H NMR) and by using the Boltzmann distribution equation, the ΔE value could be obtained.

$$\Delta E = -kT \ln(N_-/N_+) \times N$$

k = Boltzmann constant = 1.38×10^{-28} kJ K^{-1}

T = Temperature (K)

N = Avogadro's number = 6.023×10^{23}

N_- = integral of less dominant isomer

N_+ = integral of dominant isomer

For 7 at T = 295K,

$$\Delta E = -1.38 \times 10^{-28}\ \text{kJ K}^{-1}\ T(295\ \text{K})\ln(1.0/1.24) \times 6.023 \times 10^{23}\ \text{mol}^{-1}$$

$$= 0.53\ \text{kJ mol}^{-1}$$

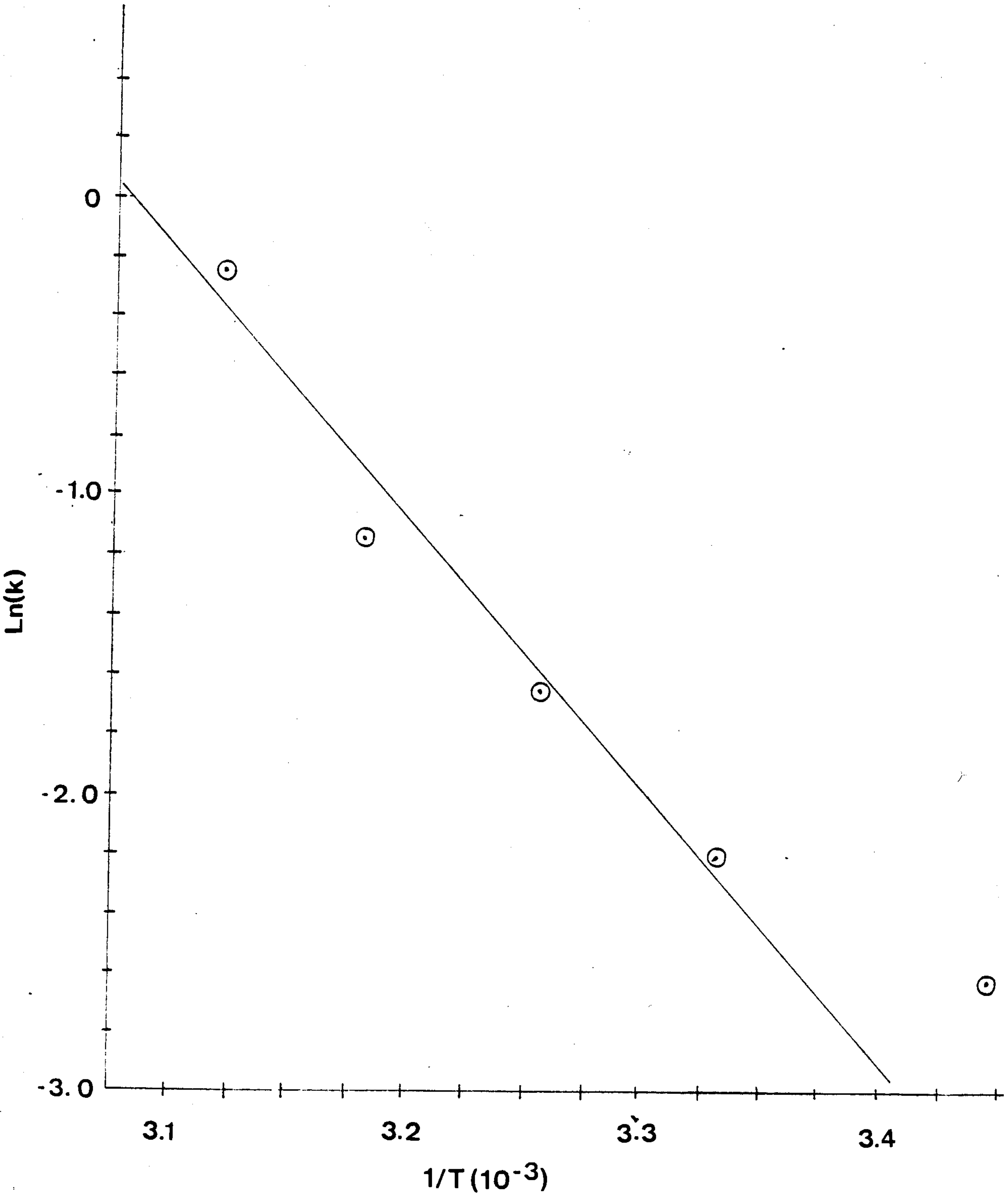
2.6.2 X-ray Structures

An overview of the X-ray data collection is given in Chapter 5. Many of the parameters and conditions used for the collection of data are listed in Table 10 but a brief introduction for each crystal structure is given

FIGURE 15

Graph of ln K versus 1/T

for Compound <u>4</u>


Ln(k)
0
-1.0
-2.0
-3.0
3.1
3.2
3.3
3.4
1/T (10^{-3})

under the appropriate heading.

1) X-ray Structure of $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

A black crystal, 0.09 x 0.11 x 0.15 mm, obtained from a hexanes/chloroform solution was chosen for the diffraction studies. Preliminary Weissenberg and precession photos (Cu radiation) indicated the crystal to be monoclinic, and systematic absences 0k0: k = 2n+1, h0l: l = 2n+1, defined the space group as $P2_1/c$. Data were then collected on a Picker FACS-I diffractometer (Mo radiation) with a scan rate of 1° min^{-1} for $3° \leqq 2\theta \leqq 35°$ and a symmetrical scan width of $(1.2 + 0.692\tan\theta)°$. There were very few reflections beyond 35° so the data collection was stopped at that point. Two standards were measured every 70 reflections and showed only random fluctuations.

Absorption corrections were applied via an analytical correction (transmission factors, 0.75-0.86). 1109 reflections out of a total of 2316 had $I \geqq 2.3\sigma(I)$ and were regarded as observed. The positions of the metal atoms and the phosphorus atom were located using MULTAN and the non-hydrogen atoms from a series of difference Fourier synthesis interspersed with least squares refinement. The hydrogen atom positions were calculated geometrically with a C-H bond length of 0.98Å and were given isotropic thermal parameters equal to the carbon atom to which they were attached and included as fixed contributions in further refinement. The metal atoms and the phosphorus atom were refined anisotropically. The largest peak in a final difference map $0.43(20)eÅ^{-3}$ was located near C(61) of the aromatic ring. Computer programs are from reference 64. Bond distances and angles are in Table 11, atom coordinates are given in Table 15, calculated hydrogen atom coordinates in Table 19, anisotropic thermal parameters in Table 23 and calculated meanplanes and dihedral angles in Table 27. An ORTEP diagram with the atom labelling scheme is shown in Figure 10 (Section 1.3.2d) and is grouped with all the heterotrimetallic alkyne structures.

2) X-ray Structure of CpNiCoRu$(CO)_6(C_2Ph_2)$ (2)

A dark brown crystal 0.11 x 0.19 x 0.24 mm, obtained from a hexane solution was chosen for the diffraction studies. Preliminary Weissenberg and precession photos (Cu radiation) indicated the crystal to be monoclinic and systematic absences 0k0: k = 2n+1, h0l: l = 2n+1, define the space group as $P2_1/c$. Data were then collected on a Picker FACS-I diffractometer (Mo radiation) with a scan range of 2° min^{-1} for $3° \leqq 2\theta \leqq 45°$ and a symmetrical scan width of $(1.4 + 0.692\tan\theta)°$. Two standards were measured for intensity control after every 70 reflections but showed only random fluctuations.

Absorption corrections were applied via an analytical correction (transmission factors, 0.64-0.79). 1816 reflections out of a total of 3100 had $I \geqq 2.3\sigma(I)$ and were regarded as observed. The positions of the metal atoms were located using MULTAN and the non-hydrogen atoms were located from a series of difference Fourier synthesis and interspersed with least squares refinement. The hydrogen atom positions were calculated geometrically with a C-H bond length of 0.98Å and were given isotropic thermal parameters equal to the carbon atom to which they were attached and were included as fixed contributions in further refinement. The metal atoms, carbonyl ligands and the cyclopentadienyl ligand were refined anisotropically. The largest peak in a final difference map, $0.53(22)eÅ^{-3}$ was located between the Ru atom and the nearest phenyl ring. Computer programs are from reference 64. Bond distances and angles are in Table 12, atomic coordinates are given in Table 16, calculated hydrogen atom coordinates in Table 20, anisotropic thermal parameters in Table 24 and calculated meanplanes and dihedral angles in Table 28. An ORTEP diagram with the atom labelling scheme is shown in Figure 11 (Section 1.3.2d) and is grouped with the other relevant structures.

3) X-ray Structure of CpNiCoOs$(CO)_6(C_2Ph_2)$ (3)

A triangular, platy dark brown crystal, 0.49 x 0.51 x 0.60 x 0.06 mm obtained from a hexane/methylene chloride solution at -20° was chosen for the diffraction studies. Preliminary oscillation and a zero-level Weissenberg photo (Cu radiation) revealed the crystal to be of good quality. Data were collected on an Enraf Nonius CAD-4 diffractometer (Mo radiation) with a variable scan speed of 0.8-5.0° min^{-1} for reflections with $3.0° \leqq 2\theta \leqq 45°$. The base scan width was 0.7° for 3-40°, and 0.9° for 40°-45° plus dispersion correction. Two standards were measured after every 150 reflections but showed only random fluctuations.

Absorption corrections were applied via an analytical correction (transmission factors, 0.23-0.66). 2503 reflections out of a total of 3063 had $I \geqq 2.3\sigma(I)$ and were regarded as observed. The positions of the metal atoms were located using MULTAN, and non-hydrogen atoms from a series of difference Fourier synthesis interspersed with least squares refinement. The hydrogen atom positions were calculated geometrically with a C-H bond length of 0.98Å and assigned isotropic thermal parameters equal to the carbon atom to which they were bonded and included as fixed contributions in further refinement. All the non-hydrogen atoms were refined anisotropically. The largest peak in a final difference map, 1.1(2)$eÅ^{-3}$ was 1.2Å from the Os atom and 0.8Å from C(3) and of no chemical significance. Computer programs are from reference 65. Bond distances and angles are in Table 13, atomic coordinates are in Table 17, calculated hydrogen atom coordinates in Table 21, anisotropic thermal parameters in Table 25 and calculated meanplanes and dihedral angles in Table 29. A labelled SNOOPI diagram is shown in Figure 12 (Section 1.3.2d) and is grouped with the other heterotrimetallic structures.

4) X-ray Structure of $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

A dark green needle, 0.07 x 0.12 x 0.36 mm recrystallized from hexane/chloroform at 0°C was used for the diffraction studies. Oscillation photos (Cu radiation) revealed the crystal to be of good quality. Data were collected on an Enraf Nonius CAD-4 diffractometer (Mo radiation) with a variable scan speed of 0.3-5.0° min^{-1} for $3° \leq 2\theta \leq 40°$ and a symmetrical base scan width of 0.7° plus dispersion correction above and below the weighted $K\alpha_1$ and $K\alpha_2$. Two standard reflections were monitored every 150 reflections and showed no indication of intensity loss.

Absorption corrections were applied via an empirical correction (ψ scans; transmission factors, 0.72-0.84). 1387 reflections from a total of 1748 had $I \geq 2.3\sigma(I)$ and were regarded as observed. The positions of the metal atoms were located using MULTAN and the non-hydrogen atoms from a series of difference Fourier synthesis interspersed with least squares refinement. The hydrogen atom positons were calculated geometrically with a C-H bond length of 0.98Å and given isotropic thermal parameters equal to the carbon atom to which they were attached and were included as fixed contributions in further refinement. All non-hydrogen atoms were refined anisotropically. The largest peak in a final difference map, $0.6(2)eÅ^{-3}$ was located between the Ni atom and the Cp ring. Computer programs are from reference 65. Bond distances and angles are in Table 14, atomic coordinates are given in Table 18, calculated hydrogen atom coordinates in Table 22, anisotropic thermal parameters in Table 26 and calculated meanplanes and dihedral angles in Table 30. A SNOOPI diagram with the atom labelling scheme is shown in Figure 13 (Section 1.3.2d) and is grouped with the other relevant structures.

Table 10. Summary of Crystal Data for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a), $CpNiCoRu(CO)_6(C_2Ph_2)$ (2), $CpNiCoOs(CO)_6(C_2Ph_2)$ (3) and $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

compound	1a	2	3	4
mol. wt.	819.15	630.1	719.3	488.8
space group	$P2_1/c$	$P2_1/c$	$P2_1/c$	$P2_1/n$
a, Å	11.698(2)	9.964(2)	9.950(2)	9.559(3)
b, Å	14.768(3)	9.5112(6)	9.505(2)	12.573(5)
c, Å	22.616(4)	24.835(2)	24.813(4)	16.175(4)
β, deg	111.82(1)	90.113(9)	90.10(3)	104.42(3)
Z	4	4	4	4
cell vol, $Å^3$	3627	2353	2347	1883
ρ calcd, gcm^{-3}	1.48	1.778	2.03	1.72
ρ obsd, gcm^{-3}	1.51	1.80	2.11	1.74
μ (MoK_α) cm^{-1}	14.7	21.7	69.4	26.4
total reflections, measured	2316	3100	3063	1748
no. reflections, $I \geq 2.3\sigma(I)$	1109	1816	2503	1387
data limits, 2θ, deg	3, 35	3.5, 45	3, 45	3, 40
final R	0.057	0.036	0.033	0.029
final R_w	0.070	0.042	0.039	0.033
GOF	1.32	1.37	1.52	1.23

Table 11. Bond Distances (Å) and Angles (deg) for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

Ni-Co	2.390(4)	Co-C(6)	2.05(2)
Ni-Fe	2.486(4)	Co-C(7)	2.04(2)
Fe-Co	2.467(4)	C(6)-C(7)	1.34(2)
Co-P	2.230(6)	C(6)-C(41)	1.50(2)
Fe-C(1)	1.70(2)	C(7)-C(51)	1.47(2)
Fe-C(2)	1.79(2)	C(1)-O(1)	1.20(3)
Fe-C(3)	1.69(2)	C(2)-O(2)	1.13(3)
Fe-C(4)	2.07(2)	C(3)-O(3)	1.21(3)
Co-C(4)	1.74(2)	C(4)-O(4)	1.19(3)
Co-C(5)	1.69(2)	C(5)-O(5)	1.15(3)
Fe-C(6)	2.03(2)	Ni-Cp (center)	1.75(1)
Ni-C(7)	1.93(2)	P-C(11)	1.86(2)
		P-C(21)	1.80(2)
		P-C(31)	1.79(2)

Ni-Co-Fe	61.55(12)	Co-C(5)-O(5)	175(2)
Co-Fe-Ni	57.71(11)	Ni-C(7)-Co	74.1(6)
Co-Ni-Fe	60.74(11)	Fe-C(6)-Co	74.3(6)
Fe-Co-P	119.4(2)	Ni-C(7)-C(6)	107(1)
Ni-Co-P	177.2(2)	Ni-C(7)-C(51)	117(1)
Fe-C(1)-O(1)	170(2)	Fe-C(6)-C(7)	107(1)
Fe-C(2)-O(2)	173(2)	Fe-C(6)-C(41)	125(1)
Fe-C(3)-O(3)	179(2)	C(6)-C(7)-C(51)	131(1)
Fe-C(4)-O(4)	131(2)	C(7)-C(6)-C(41)	126(1)
Co-C(4)-O(4)	148(2)		

Table 12. Bond Distances (Å) and Angles (deg) for CpNiCoRu(CO)$_6$(C$_2$Ph$_2$) (2)

Ni-Ru	2.560(1)	Ru-C(7)	2.126(7)
Ni-Co	2.399(1)	Ni-Cp(cntr)	1.747(1)
Co-Ru	2.565(1)	Ni-C(8)	1.903(7)
Co-C(1)	1.828(8)	C(7)-C(71)	1.480(10)
Co-C(2)	1.791(9)	C(7)-C(8)	1.377(9)
Co-C(3)	1.758(8)	C(8)-C(81)	1.489(9)
Co-C(7)	2.085(7)	C(1)-O(1)	1.158(9)
Co-C(8)	2.071(7)	C(2)-O(2)	1.135(10)
Ru-C(1)	2.251(8)	C(3)-O(3)	1.129(10)
Ru-C(4)	1.913(9)	C(4)-O(4)	1.118(10)
Ru-C(5)	1.912(9)	C(5)-O(5)	1.135(10)
Ru-C(6)	1.943(8)	C(6)-O(6)	1.130(10)

Ni-Co-Ru	61.98(4)	Ru-C(5)-O(5)	179.1(8)
Ni-Ru-Co	55.82(4)	Ru-C(6)-O(6)	179.5(8)
Ru-Ni-Co	62.21(4)	Ru-C(7)-Co	75.0(2)
Co-C(1)-O(1)	148.2(7)	Ru-C(7)-C(8)	103.4(5)
Co-C(1)-Ru	77.2(3)	Ni-C(8)-Co	74.1(2)
Co-C(2)-O(2)	177.7(8)	Ni-C(8)-C(7)	110.6(5)
Co-C(3)-O(3)	175.7(8)	Co-Ru-C(1)	44.0(2)
Ru-C(1)-O(1)	134.5(6)	Ru-Co-C(1)	58.8(3)
Ru-C(4)-O(4)	178.1(8)		

Table 13. Bond Distances (Å) and Angles (deg) for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

Ni-Os	2.579(1)	Co-C(14)	2.067(8)
Ni-Co	2.394(1)	Ni-Cp(cntr)	1.755(1)
Co-Os	2.570(1)	Ni-C(13)	1.900(7)
Os-C(1)	2.273(9)	C(7)-C(13)	1.485(10)
Os-C(2)	1.919(9)	C(13)-C(14)	1.377(10)
Os-C(3)	1.908(9)	C(14)-C(15)	1.477(10)
Os-C(4)	1.949(8)	C(1)-O(1)	1.148(10)
Os-C(14)	2.147(7)	C(2)-O(2)	1.126(11)
Co-C(1)	1.853(9)	C(3)-O(3)	1.141(11)
Co-C(5)	1.774(8)	C(4)-O(4)	1.122(10)
Co-C(6)	1.794(9)	C(5)-O(5)	1.122(10)
Co-C(13)	2.065(7)	C(6)-O(6)	1.128(10)

Ni-Co-Os	62.49(4)	Co-C(6)-O(6)	178.7(8)
Ni-Os-Co	55.41(3)	Os-C(1)-Co	76.3(3)
Os-Ni-Co	62.10(4)	Ni-C(13)-Co	74.2(2)
Os-C(1)-O(1)	136.2(8)	Ni-C(13)-C(14)	110.2(5)
Os-C(2)-O(2)	179.4(8)	Os-C(14)-Co	75.1(2)
Os-C(3)-O(3)	177.8(8)	Os-C(14)-C(13)	104.1(5)
Os-C(4)-O(4)	178.3(8)	Co-Os-C(1)	44.5(2)
Co-C(1)-O(1)	147.3(7)	Os-Co-C(1)	59.2(3)
Co-C(5)-O(5)	177.1(8)		

Table 14. Bond Distances (Å) and Angles (deg) for $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

Ni-Co	2.432(1)	Ni-C(9)	1.898(5)
Ni-Fe	2.402(1)	C(1)-O(1)	1.144(7)
Co-Fe	2.494(1)	C(2)-O(2)	1.140(7)
Fe-C(1)	1.767(6)	C(3)-O(3)	1.144(7)
Fe-C(2)	1.786(6)	C(4)-O(4)	1.137(7)
Fe-C(3)	1.773(6)	C(5)-O(5)	1.133(7)
Fe-C(9)	2.036(5)	C(6)-O(6)	1.134(7)
Fe-C(10)	2.037(5)	C(7)-C(8)	1.492(9)
Co-C(4)	1.781(6)	C(8)-C(9)	1.510(7)
Co-C(5)	1.811(6)	C(9)-C(10)	1.371(7)
Co-C(6)	1.791(6)	C(10)-C(11)	1.522(7)
Co-C(10)	1.953(5)	C(11)-C(12)	1.507(8)
Ni-Cp(cntr)	1.750(4)		

Ni-Co-Fe	58.36(3)	Co-C(5)-O(5)	178.2(5)
Ni-Fe-Co	59.52(3)	Co-C(6)-O(6)	177.0(6)
Fe-Ni-Co	62.11(3)	Ni-C(9)-C(10)	108.0(4)
Fe-C(1)-O(1)	178.2(6)	Ni-C(9)-Fe	75.2(2)
Fe-C(2)-O(2)	178.2(6)	Co-C(10)-C(9)	103.9(4)
Fe-C(3)-O(3)	178.4(5)	Co-C(10)-Fe	77.3(2)
Co-C(4)-O(4)	178.3(5)		

Table 15. Atom Coordinates (× 10^4) for CpNiCoFe(CO)$_5$(PPh$_3$)(C$_2$Ph$_2$) (1a)

Atom	X	Y	Z	B_{iso}
Co	6468.4(26)	2282.0(20)	4855.4(13)	31(2)*
Fe	7936.3(28)	1045.6(20)	5340.0(14)	33(2)*
Ni	6941.6(26)	1930.5(19)	5952.7(12)	37(2)*
P	6075(5)	2673(4)	3846(3)	36(4)*
C(1)	7443(22)	37(16)	5515(11)	58(6)
C(2)	8533(20)	535(15)	4947(10)	49(6)
C(3)	9199(19)	1057(15)	6019(10)	48(6)
C(4)	6134(19)	1136(15)	4704(10)	54(6)
C(5)	5137(21)	2693(16)	4875(11)	56(6)
O(1)	7263(14)	-729(11)	5629(7)	67(4)
O(2)	8808(14)	166(10)	4434(7)	61(4)
O(3)	10120(14)	1060(11)	6498(7)	65(4)
O(4)	5410(13)	567(11)	4428(7)	59(4)
O(5)	4263(13)	3037(10)	4888(7)	63(4)
C(6)	8342(15)	2380(12)	5312(8)	20(4)
C(7)	7782(15)	2827(12)	5642(8)	22(4)
C(11)	6900(18)	1972(14)	3445(9)	41(5)
C(12)	6270(18)	1219(15)	3100(10)	45(6)
C(13)	7038(20)	678(16)	2865(10)	50(6)
C(14)	8168(21)	827(16)	2977(11)	60(7)
C(15)	8776(20)	1562(16)	3288(10)	59(6)
C(16)	8097(18)	2156(14)	3564(9)	38(5)
C(21)	4509(17)	2596(13)	3296(9)	31(5)

Table 15 (Cont'd)

Atom	X	Y	Z	
C(22)	4083(20)	2991(16)	2705(10)	56(6)
C(23)	2858(21)	2926(16)	2300(11)	61(6)
C(24)	2020(21)	2458(17)	2461(11)	61(6)
C(25)	2393(21)	2019(17)	3024(11)	63(7)
C(26)	3645(19)	2082(15)	3439(10)	47(6)
C(31)	6440(17)	3809(13)	3712(8)	29(5)
C(32)	6763(18)	4148(13)	3220(9)	37(5)
C(33)	6914(20)	5074(15)	3141(11)	52(6)
C(34)	6772(20)	5698(15)	3556(10)	46(6)
C(35)	6406(19)	5401(14)	4034(9)	39(5)
C(36)	6243(19)	4484(15)	4128(10)	43(6)
C(41)	9328(16)	2751(13)	5102(8)	26(5)
C(42)	9276(16)	3615(13)	4883(8)	25(5)
C(43)	10171(17)	3958(13)	4667(9)	32(5)
C(44)	11138(19)	3408(15)	4690(10)	49(6)
C(45)	11230(20)	2555(15)	4906(10)	53(6)
C(46)	10349(19)	2225(15)	5135(10)	45(6)
C(51)	8062(15)	3697(12)	5978(8)	23(4)
C(52)	7108(17)	4293(13)	5952(9)	28(5)
C(53)	7473(19)	5137(14)	6303(10)	42(6)
C(54)	8621(20)	5327(15)	6636(10)	46(6)
C(55)	9543(18)	4773(13)	6665(9)	35(5)
C(56)	9270(17)	3966(13)	6328(9)	33(5)

Table 15 (Cont'd)

Atom	X	Y	Z	
C(61)	7542(21)	1502(18)	6891(11)	67(7)
C(62)	6437(19)	979(14)	6487(10)	44(5)
C(62)	5481(22)	1538(17)	6266(11)	65(7)
C(64)	5826(22)	2376(17)	6482(12)	71(7)
C(65)	7081(19)	2324(16)	6857(10)	52(6)

* Anisotropic temperature factors listed in Table 23.

Table 16. Atom Coordinates (× 10^4) and Equivalent Isotropic Temperature Factors (Å^2 × 10) for CpNiCoRu$(CO)_6(C_2Ph_2)$ (2)

	X	Y	Z	B_{iso}
Ni	3775.9(9)	2816.8(11)	913.4(4)	35.2(5)*
Co	1963.9(10)	4470.4(11)	969.6(4)	31.8(5)*
Ru	1484.3(7)	2061.6(7)	546.4(2)	36.7(3)*
C(1)	1639(8)	4291(8)	248(3)	40(4)*
O(1)	1579(6)	4819(6)	9829(2)	54(4)*
C(2)	609(9)	5620(9)	1144(3)	48(5)*
O(2)	-220(7)	6384(7)	1256(3)	78(5)*
C(3)	3272(9)	5708(9)	943(3)	44(5)*
O(3)	4053(7)	6567(7)	923(2)	66(4)*
C(4)	1738(9)	197(9)	818(3)	51(5)*
O(4)	1917(8)	-878(6)	985(3)	85(6)*
C(5)	-386(9)	1895(9)	386(3)	47(5)*
O(5)	-1496(6)	1806(7)	285(3)	68(4)*
C(6)	2040(10)	1644(9)	-186(3)	52(6)*
O(6)	2366(8)	1412(8)	-612(2)	78(5)*
C(7)	1304(7)	2669(7)	1367(3)	31(1)
C(8)	2606(7)	2967(8)	1516(3)	30(1)
C(71)	132(7)	2538(7)	1730(3)	31(2)
C(72)	-667(8)	1357(8)	1694(3)	40(2)
C(73)	-1716(9)	1125(10)	2062(3)	51(2)
C(74)	-1943(9)	2113(10)	2462(3)	55(2)

Table 16 (Cont'd)

	X	Y	Z	B_{iso}
C(75)	-1156(9)	3281(9)	2504(3)	52(2)
C(76)	-115(8)	3514(8)	2136(3)	41(2)
C(81)	3194(7)	3043(8)	2066(3)	31(1)
C(82)	4167(8)	4011(8)	2196(3)	37(2)
C(83)	4754(9)	4043(9)	2714(3)	48(2)
C(84)	4308(9)	3117(9)	3091(3)	52(2)
C(85)	3363(9)	2152(10)	2981(3)	52(2)
C(86)	2772(8)	2077(9)	2462(3)	44(2)
C(91)	4951(9)	7196(12)	4712(3)	68(8)*
C(92)	4971(10)	6205(11)	4310(5)	86(9)*
C(93)	4461(9)	6806(12)	3846(4)	74(8)*
C(94)	4128(9)	8154(12)	3954(4)	71(8)*
C(95)	4434(9)	8392(10)	4508(4)	64(6)

* Anisotropic thermal parameters listed in Table 24.

Table 17. Atom Coordinates (× 10^4) for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

	X	Y	Z
Ni	1209.3(9)	2177.7(11)	917.1(4)
Co	3032.2(9)	538.3(11)	965.8(4)
Os	3510.3(3)	2956.7(3)	544.3(2)
C(1)	3379(7)	710(9)	235(3)
O(1)	3346(6)	163(6)	-176(2)
C(2)	5392(9)	3119(9)	388(3)
O(2)	6494(6)	3225(7)	295(3)
C(3)	3276(9)	4382(10)	802(3)
O(3)	3104(8)	5937(7)	967(3)
C(4)	2932(9)	3370(9)	-190(3)
O(4)	2624(8)	3586(8)	-617(2)
C(5)	1711(8)	-712(9)	937(3)
O(5)	915(7)	-1548(7)	920(3)
C(6)	4374(9)	-624(9)	1154(3)
O(6)	5210(7)	-1372(8)	1265(3)
C(7)	1805(7)	1934(8)	2071(3)
C(8)	2204(8)	2873(8)	2472(3)
C(9)	1641(10)	2831(10)	2987(3)
C(10)	675(9)	1849(10)	3098(3)
C(11)	246(8)	926(10)	2701(3)
C(12)	806(7)	954(9)	2201(3)
C(13)	2379(7)	2007(7)	1520(3)
C(14)	3679(7)	2314(8)	1371(3)

Table 17 (Cont'd)

	X	Y	Z
C(15)	4843(7)	2473(8)	1737(3)
C(16)	5118(8)	1518(9)	2141(3)
C(17)	6163(9)	1727(10)	2511(4)
C(18)	6949(9)	2890(10)	2468(4)
C(19)	6721(8)	3854(10)	2067(3)
C(20)	5683(8)	3636(9)	1707(3)
C(21)	-550(9)	3179(12)	1160(4)
C(22)	-906(8)	1830(12)	1046(4)
C(23)	-613(9)	1620(11)	489(4)
C(24)	-89(9)	2847(13)	303(4)
C(25)	-51(10)	3801(12)	713(5)

Table 18. Atom Coordinates (× 10^4) for $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

	X	Y	Z
Ni	1867.4(8)	2196.5(6)	937.7(5)
Co	3918.4(9)	3390.7(6)	1366.1(5)
Fe	3296.0(9)	2527.4(7)	-64.1(6)
C(1)	4719(7)	2952(6)	-505(4)
O(1)	5660(6)	3210(5)	-777(4)
C(2)	2036(7)	3553(5)	-495(4)
O(2)	1231(5)	4199(4)	-788(3)
C(3)	2593(7)	1559(5)	-849(4)
O(3)	2130(6)	918(4)	-1342(3)
C(4)	4010(7)	3027(5)	2444(4)
O(4)	4060(5)	2773(4)	3123(3)
C(5)	2692(7)	4508(5)	1199(4)
O(5)	1950(5)	5223(4)	1111(4)
C(6)	5556(7)	4116(5)	1432(5)
O(6)	6615(6)	4537(4)	1461(4)
C(7)	3759(8)	-336(5)	1648(5)
C(8)	3509(7)	202(5)	800(4)
C(9)	3512(6)	1400(4)	857(4)
C(10)	4687(6)	2060(4)	1045(3)
C(11)	6270(6)	1754(5)	1219(4)
C(12)	6989(7)	1638(6)	2154(4)
C(13)	605(8)	1833(8)	1778(5)
C(14)	291(8)	1128(6)	1122(6)

Table 18 (Cont'd)

	X	Y	Z
C(15)	-252(7)	1694(7)	388(5)
C(16)	-312(7)	2729(6)	575(5)
C(17)	219(8)	2813(6)	1443(6)

Table 19. Calculated Hydrogen Atom Coordinates for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

Atom	X	Y	Z
H(12)	5325	1006	3034
H(13)	6703	107	2535
H(14)	8831	391	2864
H(15)	9785	1764	3354
H(16)	8530	2764	3857
H(22)	4701	3389	2539
H(23)	2499	3235	1824
H(24)	1042	2425	2155
H(25)	1726	1574	3168
H(26)	3926	1760	3908
H(32)	6881	3684	2872
H(33)	7232	5319	2771
H(34)	6856	6419	3490
H(35)	6330	5879	4393
H(36)	5849	4285	4487
H(42)	8339	4065	4880
H(43)	10084	4668	4499
H(44)	11823	3692	4486
H(45)	12062	2173	4975
H(46)	10446	1566	5323
H(52)	6118	4152	5675
H(53)	6786	5618	6293

Table 19 (Cont'd)

Atom	X	Y	Z
H(54)	8885	5952	6900
H(55)	9468	4923	6948
H(56)	10	3507	6330
H(61)	8447	1203	7121
H(62)	6304	226	6374
H(63)	4506	1366	5921
H(64)	5257	2992	6402
H(65)	7634	2909	7100

Table 20. Calculated Hydrogen Atom Coordinates (× 10^4) for $CpNiCoRu(CO)_6(C_2Ph_2)$ (2)

	X	Y	Z
H(72)	-481	604	1374
H(73)	-2324	173	2052
H(74)	-2772	2043	2743
H(75)	-1315	4035	2839
H(76)	524	4422	2172
H(82)	4476	4760	1893
H(83)	5540	4809	2844
H(84)	4652	3142	3522
H(85)	3150	1295	3276
H(86)	1983	1291	2375
H(91)	5267	6983	5124
H(92)	5358	5092	4344
H(93)	4292	6230	3463
H(94)	3741	8815	3630
H(95)	4310	9364	4721

Table 21. Calculated Hydrogen Atom Coordinates (× 10^4) for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

	X	Y	Z
H(8)	2898	3576	2391
H(9)	1932	3498	3265
H(10)	283	1799	3460
H(11)	-466	245	2782
H(12)	506	277	1928
H(16)	4565	668	2170
H(17)	6327	1039	2798
H(18)	7686	3038	2724
H(19)	7288	4694	2037
H(20)	5533	4329	1421
H(21)	-637	3631	1513
H(22)	-1290	1143	1296
H(23)	-762	756	281
H(24)	213	3019	-66
H(25)	280	4770	687

Table 22. Calculated Hydrogen Atom Coordinates (× 10^4) for $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

	X	Y	Z
H(13)	944	1689	2244
H(14)	447	550	1152
H(15)	-653	1509	-250
H(16)	-654	3200	248
H(17)	289	3454	1783
H(71)	4647	9787	2065
H(72)	3752	8968	1538
H(73)	2876	9831	1935
H(81)	4244	10010	526
H(82)	2641	9943	421
H(111)	6820	12339	1001
H(112)	6340	11109	922
H(121)	6422	11222	2394
H(122)	8049	11335	2314
H(123)	7060	12339	2381

Table 23. Anisotropic Thermal Parameters ($Å^2 \times 10$) for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	41.9(21)	41.0(20)	35.4(19)	5.8(17)	19.9(16)	-1.1(17)
Fe	50.6(22)	36.6(21)	37.4(19)	-3.2(20)	21.3(17)	-0.2(2)
Ni	58.3(21)	49.4(20)	32.9(18)	-6.9(18)	24.2(16)	-4.2(17)
P	56(5)	42(4)	38(4)	10(4)	26(4)	5(4)

Table 24. Anisotropic Thermal Parameters (Å^2× 10) for $CpNiCoRu(CO)_6(C_2Ph_2)$ (2)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	38.3(6)	56.1(7)	39.3(6)	8.0(6)	6.8(4)	5.8(6)
Co	39.3(6)	38.4(6)	43.0(6)	0.8(5)	0.1(5)	1.8(5)
Ru	51.3(4)	44.7(4)	43.6(4)	0.0(4)	1.3(3)	-6.7(4)
C(1)	47(5)	45(5)	59(5)	-2.0(4)	-2.0(4)	-5.0(4)
O(1)	89(5)	61(4)	57(4)	-10(4)	-14(4)	11(4)
C(2)	78(6)	56(6)	46(5)	9(5)	-2(4)	3(4)
O(2)	105(6)	94(5)	97(5)	51(5)	6(4)	-10(4)
C(3)	76(6)	58(6)	34(5)	-17(5)	-3(4)	2(4)
O(3)	92(5)	84(5)	74(5)	-43(4)	-8(4)	8(4)
C(4)	78(7)	49(6)	66(6)	-2(5)	13(5)	-16(5)
O(4)	159(7)	51(4)	106(5)	27(5)	22(5)	14(4)
C(5)	67(6)	49(6)	63(5)	-3(5)	-9(5)	-6(5)
O(5)	53(4)	93(5)	114(5)	-1(4)	-18(4)	-20(4)
C(6)	83(7)	57(6)	56(5)	-6(5)	0(4)	-1(5)
O(6)	137(7)	107(6)	51(4)	24(5)	14(4)	-12(4)
C(91)	55(6)	151(10)	50(5)	-8(7)	20(5)	21(6)
C(92)	65(7)	71(7)	139(12)	-27(6)	48(7)	10(8)
C(93)	59(6)	139(10)	82(7)	-48(7)	13(5)	-41(7)
C(94)	37(5)	139(10)	95(7)	-2(6)	5(5)	51(7)
C(95)	50(6)	92(8)	102(7)	-14(5)	41(5)	-45(6)

Table 25. Anisotropic Thermal Parameters (Å^2 × 10) for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	32.6(5)	49.6(7)	36.8(5)	7.0(5)	-8.6(4)	-4.9(5)
Co	30.5(5)	30.4(6)	41.6(6)	0.4(5)	-2.8(4)	-1.4(5)
Os	41.9(2)	38.3(2)	39.9(2)	-0.07(2)	-3.7(1)	6.7(2)
C(1)	28(4)	48(5)	69(6)	1(4)	-3(4)	4(5)
O(1)	79(4)	67(4)	43(3)	-6(4)	10(3)	-20(3)
C(2)	67(6)	45(5)	52(5)	-5(5)	-2(4)	12(4)
O(2)	46(4)	90(6)	109(5)	-13(4)	14(4)	24(4)
C(3)	66(6)	56(6)	50(5)	11(5)	-13(4)	3(5)
O(3)	153(7)	54(5)	83(5)	20(5)	-7(4)	-12(4)
C(4)	73(6)	53(6)	52(5)	19(5)	-4(4)	3(5)
O(4)	124(6)	98(6)	44(4)	30(5)	-19(4)	9(4)
C(5)	63(6)	47(5)	38(5)	-6(5)	12(4)	2(4)
O(5)	86(5)	78(5)	75(4)	-51(4)	3(4)	1(4)
C(6)	65(6)	47(6)	55(5)	7(5)	-4(4)	-1(4)
O(6)	79(5)	85(5)	114(6)	48(5)	-12(4)	9(5)
C(7)	34(4)	51(5)	34(4)	14(4)	-6(3)	3(4)
C(8)	57(5)	40(5)	50(5)	8(4)	-4(4)	-12(4)
C(9)	87(7)	65(7)	41(5)	21(6)	-3(4)	-16(5)
C(10)	71(6)	81(8)	43(5)	31(6)	9(4)	1(5)
C(11)	46(5)	68(6)	47(5)	4(5)	4(4)	10(4)
C(12)	40(5)	52(5)	40(5)	2(4)	-5(4)	-1(4)

Table 25 (Cont'd)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(13)	32(4)	35(5)	34(4)	-2(4)	-7(3)	-3(4)
C(14)	33(4)	36(4)	39(4)	7(4)	-2(3)	1(3)
C(15)	38(4)	32(4)	36(4)	-3(4)	-4(3)	-4(3)
C(16)	42(5)	48(5)	60(5)	-6(4)	-16(4)	-4(4)
C(17)	64(6)	59(6)	63(6)	15(5)	-28(5)	-5(5)
C(18)	50(5)	70(7)	74(6)	8(5)	-28(5)	-25(5)
C(19)	34(4)	68(6)	66(6)	-10(5)	-3(4)	-21(5)
C(20)	45(5)	43(5)	56(5)	-10(4)	-7(4)	-2(4)
C(21)	54(6)	119(10)	66(6)	36(6)	-16(5)	-27(6)
C(22)	26(5)	125(10)	93(7)	1(5)	-10(5)	45(7)
C(23)	44(5)	83(8)	94(7)	8(5)	-32(5)	-30(6)
C(24)	45(5)	133(11)	67(6)	21(6)	-18(5)	8(7)
C(25)	65(7)	66(7)	150(10)	10(6)	-53(7)	8(7)

Table 26. Anisotropic Thermal Parameters (Å^2 × 10) for $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	33.2(4)	48.1(5)	50.4(5)	-7.3(4)	12.9(4)	-4.3(4)
Co	37.3(5)	41.0(5)	59.9(6)	-5.4(4)	9.3(4)	-5.7(4)
Fe	39.3(5)	48.8(5)	44.7(5)	-1.1(4)	11.0(4)	7.2(4)
C(1)	62(5)	75(5)	65(5)	2(4)	18(4)	21(4)
O(1)	85(4)	150(5)	124(5)	-1(4)	61(4)	49(4)
C(2)	53(4)	69(5)	63(5)	3(4)	19(4)	13(4)
O(2)	72(3)	90(4)	102(4)	29(3)	22(3)	37(3)
C(3)	60(4)	65(5)	47(4)	7(4)	5(4)	11(4)
O(3)	111(4)	82(4)	71(4)	0(3)	-8(3)	-17(3)
C(4)	47(4)	62(4)	65(5)	-5(4)	13(4)	-14(4)
O(4)	85(4)	132(5)	56(3)	5(3)	12(3)	-1(3)
C(5)	54(4)	56(4)	83(5)	-8(4)	12(4)	-11(4)
O(5)	73(3)	63(3)	160(6)	21(3)	29(4)	3(3)
C(6)	56(5)	53(5)	109(6)	-12(4)	22(4)	-8(4)
O(6)	79(4)	96(4)	189(6)	-47(3)	42(4)	-12(4)
C(7)	109(7)	53(5)	96(6)	-2(4)	34(5)	21(4)
C(8)	58(4)	48(4)	63(5)	-2(3)	21(4)	4(4)
C(9)	47(4)	35(3)	38(4)	4(3)	13(3)	2(3)
C(10)	39(4)	43(4)	38(4)	1(3)	9(3)	0(3)
C(11)	40(4)	57(4)	61(5)	2(3)	14(3)	1(3)
C(12)	46(4)	100(6)	61(5)	11(4)	-2(4)	0(4)

Table 26 (Cont'd)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(13)	51(5)	193(9)	69(6)	-8(6)	28(4)	24(6)
C(14)	63(5)	59(5)	187(9)	-16(4)	66(6)	8(6)
C(15)	44(4)	150(8)	100(6)	-49(5)	28(4)	-56(6)
C(16)	33(4)	107(7)	128(8)	3(4)	21(4)	35(6)
C(17)	55(5)	108(7)	144(8)	-31(5)	55(5)	-69(6)

Table 27. Calculated Meanplanes[a] and Dihedral Angles for $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$ (1a)

Plane No.	Atoms Defining Plane[b]	A	B	C	D (Å)	χ^2
1	Ni, Co, Fe	-0.7095	-0.6544	-0.2163	-7.3430	0.00
2[c]	Cp ring: C61(0.2), C62(-1.1), C63(1.6), C64(-1.4), C65(0.7)	0.5630	0.1894	-0.8045	-9.5132	0.01
3[d]	Ni(-2.4), Fe(2.3), C6(-4.3), C7(4.4)	-0.5472	0.1110	-0.8296	-8.4123	1.35
4	C6(-39), C7(41), C41(16), C51(-18)	-0.3994	0.4157	-0.8171	-9.7276	12.8

[a] Plane coordination is Ax + By + Cz - D = 0, with x, y, z in orthorhombic coordinates.

[b] Deviations of the atoms (Å × 10^3) from the meanplane are given in parentheses.

[c] Ni deviation 1.752 Å.

[d] Co deviation 1.766 Å.

Table 27 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
1	2	71.8
1	3	57.8
1	4	77.0
2	3	67.6
2	4	59.3
3	4	19.5

Table 28. Calculated Meanplanes[a] and Dihedral Angles for $CpNiCoRu(CO)_6(C_2Ph_2)$ (2)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1	Ru, Ni, Co	0.2452	0.3614	-0.8996	-0.1513	0.00
2[c]	Ru(-15), Ni(18), C7(30), C8(-32)	-0.1777	0.9472	-0.2670	1.2479	
3	Ph ring: C71(-.7), C72(-2), C73(-2), C74(10), C75(-12), C76(8)	-0.6270	0.4939	-0.6024	-1.4687	4.41
4	Ph ring: C81(-4), C82(-4), C83(11), C84(-11), C85(2), C86(5)	0.7067	-0.6441	-0.29286	-1.1222	4.32
5	Cp ring: C91(-3), C92(1), C93(2), C94(-3), C95(4)	0.9201	-0.2964	-0.2559	3.8286	0.42
6	Ru, Co, C1	0.9788	-0.1321	-0.1565	0.9731	0.00
7	C7(-33), C8(33), C81(-14), C71(14)	-0.1596	0.9870	-0.0185	-2.268	

[a] Plane equation is Ax + By + Cz − D = 0, with x, y, z in orthorhombic coordinates.

[b] Deviations of the atoms (Å × 10^3) from the meanplane are given in parentheses.

[c] Co deviation 1.790 Å.

[d] Ni deviation 1.747 Å.

Table 28 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
1	2	57.4
1	3	55.5
1	4	78.3
1	5	69.6
1	6	70.6
1	7	70.5
2	3	42.3
2	4	131.1
2	5	112.1
2	6	104.9
3	4	125.8
3	5	124.7
3	6	125.8
4	5	23.6
4	6	34.7
5	6	11.5

Table 29. Calculated Meanplanes[a] and Dihedral Angles for $CpNiCoOs(CO)_6(C_2Ph_2)$ (3)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1	Ni, Co, Os	-0.2478	-0.3582	-0.9002	-3.0869	0.00
2[c]	Os(-15), Ni(17), C13(-31), C14(29)	0.1804	-0.9445	-0.2746	-2.3806	
3	Ph ring: C7(4), C8(-3), C9(8), C10(-7), C11(2), C12(-3)	-0.6741	0.6710	-0.3089	-1.6728	3.21
4	Ph ring: C15(-9), C16(7), C17(-2), C18(-3), C19(2), C20(4)	0.6212	-0.4989	-0.6043	-0.7805	2.67
5[d]	Cp ring: C21(2), C22(-3), C23(3), C24(-1), C25(-0.6)	-0.9184	0.3113	-0.2442	0.7425	0.28
6	Os, Co, C1	-0.9766	0.1226	-0.1765	-3.3026	0.00
7	C7(15), C13(-28), C14(31), C15(-14)	-0.1738	0.9848	0.0088	-1.5291	22.0

[a] Plane equation is $Ax + By + Cz - D = 0$, with x, y, z in orthorhombic coordinates.

[b] Deviation of the atoms (Å × 10^3) from the meanplane are given in parentheses.

[c] Co deviation 1.783 Å.

[d] Ni deviation 1.755 Å.

Table 29 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
1	2	57.3
1	3	78.2
1	4	55.3
1	5	70.3
1	6	69.1
1	7	71.5
2	3	132.1
2	4	41.5
2	5	112.9
2	6	104.1
3	4	104.7
3	5	25.4
3	6	37.3
4	5	125.3
4	6	124.1
5	6	12.0

Table 30. Calculated Meanplanes[a] and Dihedral Angles for $CpNiCoFe(CO)_6(C_2Et_2)$ (4)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1	Ni, Co, Fe	-0.5122	0.7746	-0.3710	0.8783	0.00
2[c]	Cp ring: C13(8), C14(-8), C15(6), C16(-0.7), C17(-4)	0.9842	0.1234	-0.1267	-0.2096	2.84
3[d]	Ni(17), Co(-16), C9(-30), C10(29)	0.0844	0.3013	-0.9498	-0.4617	
4	Fe, C9, C10	0.6173	-0.5950	-0.5147	0.1172	0.00
5	C8(-3), C9(6) C10(-6), C11(3)	0.2090	0.0670	-0.9756	-0.5709	2.99

[a] Plane equation is $Ax + By + Cz - D = 0$, with x,y,z in orthorhombic coordinates.

[b] Deviations of the atoms (Å×10^3) from the meanplane are given in parentheses.

[c] Ni deviation 1.750 Å.

[d] Fe deviation 1.786 Å.

Table 30 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
1	2	68.8
1	3	57.2
1	4	125.9
1	5	72.2
2	3	76.1
2	4	53.2
2	5	70.3
3	4	68.8
3	5	15.3
4	5	53.8

CHAPTER 2

X-RAY CRYSTAL STRUCTURE OF $CoMn(CO)_6(MeC_2Ph)_2$

2.1 Introduction

The formation of $CoMn(CO)_6(RC_2R')$ (R=Ph,R'=Me,Ph) by the thermal reaction of $CoMn(CO)_9$ with an equimolar amount of the appropriate alkyne was recently reported[66]. This is a facile reaction requiring conditions similar to that of the formation[67] of $Co_2(CO)_6(RC_2R')$ from $Co_2(CO)_8$ and C_2Ph_2 but less severe than the reaction[68] of $Mn_2(CO)_{10}$ with C_2Ph_2. Subsequently, when two moles of alkyne are reacted with one mole of $CoMn(CO)_9$, compounds of the formula $CoMn(CO)_6(RC_2R')_2$ are isolated as the major product. On the basis of the spectroscopic data[69] a 'ferrole' type structure was proposed.

Ferroles or tricarbonylferracyclopentadieneirontricarbonyl complexes are among the more common products of reactions of iron carbonyls with alkynes. Over eighty compounds have been reported and there are fourteen X-ray diffraction investigations on this structural type[70]. Other metals, notably rhodium[71], iridium[72,73], osmium[74,75] and cobalt[76] also form ferrole type structures but these compounds are far less common than those with iron. Structures where there are two different metals attached to the butadiene fragment are noticeably scarce, only $(CO)_3Fe(CH_3C_2CH_3)_2Ni(C_4(CH_3)_4)$ and $CpW(CO)_2(CF_3C_2CF_3)_2Co(CO)_2$ being structurally characterized[77,78]. Very recently, $FeRu(CO)_6(PhC_2Ph)_2$ was synthesized but an X-ray structure was not reported[30]. Because of our interest in mixed metal alkyne chemistry and the lack of heterobimetallic ferrole type structures, the X-ray structure determination of $CoMn(CO)_6(MeC_2Ph)_2$ (<u>10</u>) was undertaken.

2.2 X-ray Data Collection and Refinement

A large hexagonal red-brown crystal obtained from $CHCl_3$-hexanes, having dimensions 0.35-0.40 mm in widths and 0.21 mm thick was used for the data collection. This crystal was supplied by P.J. Manning. Preliminary oscillation photos (Cu radiation) indicated the crystal to be of good quality. The crystal was transferred to an Enraf-Nonius diffractometer (Mo radiation) and the cell dimensions and space group were determined. Data were collected with a scan rate of 1.0-6.0° min^{-1} for reflections with $3° \leqq 2\theta \leqq 45°$ and a symmetrical scan width of ±0.45° plus dispersion correction from the peak center. Four standard reflections were measured every 1 hour of X-ray exposure time, two to monitor intensity and two to monitor crystal movement. Neither pair showed any significant deviations. Absorption corrections were applied via an empirical correction (transmission factors 0.848-0.998). 2373 reflections out of a total of 2939 had $I \geqq 3.0\sigma(I)$ and were regarded as observed.

The positions of the metal atoms were located from a Patterson map and the non-hydrogen atoms were located from difference Fourier synthesis interspersed with least squares refinement. All the hydrogen atoms were placed in geometrically calculated positions with $\underline{d}$(C-H) equal to 0.98Å. The phenyl hydrogens were given isotropic thermal parameters of $0.06Å^2$ and the methyl hydrogens, $0.07Å^2$. All non-hydrogen atoms were refined anisotropically. The largest peak in a final difference map was 0.21(12) $eÅ^{-3}$ and was located near the Mn atom. Computer programs are from reference 65. A summary of the crystal data is in Table 31, bond distances and angles in Table 32, atom coordinates in Table 33, anisotropic thermal parameters in Table 34, calculated hydrogen atom coordinates in Table 35 and calculated

Table 31. Summary of Crystal Data for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

formula	$CoMnC_{24}H_{16}O_6$
mol. wt.	514.3
space group	$P2_1/c$
a, Å	16.308(3)
b, Å	8.875(2)
c, Å	15.574(4)
β, deg	93.44(2)
Z	4
cell vol, $Å^3$	2250
ρ calcd, gcm^{-3}	1.516
ρ obsd, gcm^{-3}	1.53
μ (MoK_α) cm^{-1}	13.1
total reflections, measured	2939
no. reflections, $I \geq 3.0\sigma(I)$	2373
2θ limits, deg	3, 45
final R	3.34%
final R_w	4.28%
GOF	1.56

Table 32. Bond Distances (Å) and Angles (deg) for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

Co-Mn	2.5488(8)	C(7)-C(21)	1.504(5)
Co-C(1)	1.823(4)	C(7)-C(8)	1.401(5)
Co-C(2)	1.797(4)	C(8)-C(81)	1.513(5)
Co-C(3)	1.829(4)	C(8)-C(9)	1.426(5)
Co-C(7)	1.977(3)	C(9)-C(10)	1.423(5)
Co-C(10)	1.977(3)	C(9)-C(91)	1.506(5)
Mn-C(4)	1.785(4)	C(10)-C(11)	1.486(5)
Mn-C(5)	1.798(4)	C(1)-O(1)	1.127(5)
Mn-C(6)	1.798(4)	C(2)-O(2)	1.132(5)
Mn-C(7)	2.100(3)	C(3)-O(3)	1.124(5)
Mn-C(8)	2.185(3)	C(4)-O(4)	1.152(5)
Mn-C(9)	2.170(3)	C(5)-O(5)	1.149(5)
Mn-C(10)	2.098(3)	C(6)-O(6)	1.145(5)
Co-C(1)-O(1)	179.5(4)	Mn-C(7)-C(8)	74.2(2)
Co-C(2)-O(2)	176.6(4)	Mn-C(7)-C(21)	134.2(2)
Co-C(3)-O(3)	176.0(4)	Mn-C(10)-C(9)	73.3(2)
Mn-C(4)-O(4)	178.6(4)	Mn-C(10)-C(11)	135.6(2)
Mn-C(5)-O(5)	177.7(4)	C(8)-C(7)-C(21)	120.6(3)
Mn-C(6)-O(6)	179.2(4)	C(81)-C(8)-C(7)	123.7(3)
Co-C(7)-C(8)	113.7(2)	C(81)-C(8)-C(9)	121.6(3)
Co-C(7)-C(21)	122.2(2)	C(91)-C(9)-C(8)	121.5(3)
Co-C(10)-C(9)	113.5(2)	C(91)-C(9)-C(10)	124.8(3)
Co-C(10)-C(11)	121.9(2)	C(9)-C(10)-C(11)	120.9(3)

Table 33. Atomic Coordinates (× 10^4) for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

Atom	X	Y	Z
Co	1851.5(3)	5311.1(5)	4384.2(3)
Mn	2933.1(3)	7311.8(6)	4765.0(3)
C(1)	863(2)	6219(4)	4145(2)
O(1)	254(2)	6786(4)	3993(2)
C(2)	1610(2)	3573(4)	4904(3)
O(2)	1496(2)	2480(3)	5252(2)
C(3)	1878(2)	4736(5)	3259(3)
O(3)	1846(2)	4371(5)	2566(2)
C(4)	3329(3)	8803(4)	5438(2)
O(4)	3583(3)	9746(4)	5887(2)
C(5)	3779(2)	7555(4)	4090(2)
O(5)	4335(2)	7731(3)	3682(2)
C(6)	2294(2)	8579(4)	4122(2)
O(6)	1895(2)	9393(4)	3708(2)
C(7)	1992(2)	6432(4)	5480(2)
C(8)	2727(2)	6101(4)	5954(2)
C(81)	2913(2)	6595(4)	6875(2)
C(9)	3318(2)	5315(4)	5485(2)
C(91)	4157(2)	4944(4)	5882(2)
C(10)	3041(2)	4970(4)	4625(2)
C(11)	3518(2)	3946(4)	4089(2)
C(12)	3707(2)	4281(4)	3251(2)

Table 33 (Cont'd)

Atom	X	Y	Z
C(13)	4155(2)	3294(5)	2775(2)
C(14)	4422(3)	1948(5)	3133(3)
C(15)	4237(3)	1578(4)	3953(3)
C(16)	3782(3)	2571(4)	4430(2)
C(21)	1279(2)	7111(4)	5910(2)
C(22)	883(2)	6257(4)	6504(2)
C(23)	209(2)	6827(5)	6900(2)
C(24)	-79(2)	8232(5)	6699(3)
C(25)	302(3)	9093(5)	6119(3)
C(26)	984(2)	8544(4)	5271(2)

Table 34. Anisotropic Thermal Parameters (Å^2 × 10) for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	33.3(3)	41.0(3)	36.9(3)	-0.2(2)	1.0(2)	-5.0(2)
Mn	38.3(3)	32.4(3)	31.1(3)	-0.2(2)	4.9(2)	1.1(2)
C(1)	45(2)	54(3)	48(2)	6(2)	-3(2)	-15(2)
O(1)	55(2)	88(2)	88(2)	26(2)	-17(2)	-22(2)
C(2)	47(2)	54(3)	60(3)	-5(2)	6(2)	-12(2)
O(2)	99(3)	60(2)	115(3)	-16(2)	21(2)	20(2)
C(3)	36(2)	82(3)	54(2)	3(2)	0(2)	-16(2)
O(3)	70(2)	185(4)	49(2)	5(2)	-5(2)	-43(2)
C(4)	85(3)	45(3)	41(2)	-9(2)	6(2)	7(2)
O(4)	169(4)	60(2)	65(2)	-41(2)	-7(2)	-13(2)
C(5)	50(2)	28(2)	51(2)	1(2)	5(2)	-1(2)
O(5)	59(2)	55(2)	86(2)	-6(1)	38(2)	1(2)
C(6)	54(2)	53(3)	42(2)	2(2)	17(2)	6(2)
O(6)	80(2)	83(2)	68(2)	36(2)	10(2)	34(2)
C(7)	39(2)	33(2)	34(2)	-1(2)	7(2)	2(2)
C(8)	43(2)	34(2)	29(2)	2(2)	5(2)	6(2)
C(81)	51(2)	62(3)	31(3)	3(2)	2(2)	-5(2)
C(9)	38(2)	35(2)	33(2)	-2(2)	4(2)	6(2)
C(91)	40(2)	64(3)	41(2)	9(2)	-3(2)	3(2)
C(10)	34(2)	31(2)	37(2)	-3(1)	8(1)	1(2)
C(11)	34(2)	33(2)	40(2)	1(2)	2(2)	-4(2)

Table 34 (Cont'd)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(12)	38(2)	40(2)	38(2)	4(2)	0(2)	-3(2)
C(13)	47(2)	62(3)	37(2)	1(2)	8(2)	-13(2)
C(14)	59(3)	51(3)	65(3)	13(2)	10(2)	-20(2)
C(15)	76(3)	29(2)	78(3)	13(2)	7(2)	-1(2)
C(16)	62(3)	35(2)	53(2)	1(2)	11(2)	1(2)
C(21)	37(2)	38(2)	35(2)	3(2)	5(2)	-4(2)
C(22)	47(2)	43(2)	51(2)	0(2)	14(2)	3(2)
C(23)	50(2)	61(3)	56(3)	-2(2)	24(2)	3(2)
C(24)	44(2)	73(3)	51(2)	9(2)	15(2)	-12(2)
C(25)	66(3)	54(3)	56(3)	21(2)	9(2)	-5(2)
C(26)	58(2)	48(2)	45(2)	10(2)	18(2)	3(2)

Table 35. Calculated Hydrogen Atom Coordinates (× 10^4) for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

Atom	X	Y	Z
H(12)	3518	5236	2989
H(13)	4281	3553	2185
H(14)	4745	1261	2795
H(15)	4420	611	4193
H(16)	3648	2289	5009
H(22)	1085	5245	6656
H(23)	- 60	6209	7325
H(24)	-563	8624	6980
H(25)	97	10104	5983
H(26)	1251	9181	5308
H(811)	3404	7212	6850
H(812)	3005	5723	7256
H(813)	2452	7175	7088
H(911)	4366	5924	6097
H(912)	4513	4519	5462
H(913)	4127	4221	6357

Table 36. Calculated Meanplanes[a] and Dihedral Angles for $CoMn(CO)_6(MeC_2Ph)_2$ (10)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1[c]	C7(4.7), C8(-8.7) C9(8.8), C10(-4.7)	0.4001	0.8662	-0.3225	3.487	10.5
2	C11(6.3), C12(-3.0), C13(-3.2), C14(-5.8), C15(-0.2), C16(-7.3)	0.8284	0.4356	0.3023	8.202	6.58
3	C21(-1.7), C22(-2.1), C23(6.3), C24(-4.9), C25(-0.8), C26(3.6)	0.5606	0.3791	0.7017	10.02	3.28
4	Mn(0.2), Co(0.0), C1(3.3), O1(-1.5)	-0.2611	-0.0668	0.9768	5.561	0.7
5	Co(-0.1), Mn(0.0), C2(11.3), O2(-5.6)	-0.6810	0.4788	0.5935	4.253	7.4
6	Co(-0.1), Mn(0.0), C3(19.3), O3(-12.3)	0.6496	-0.7171	0.2135	0.415	19.1
7	Co(0.0), Mn(0.1), C4(-9.9), O4(6.2)	0.4933	-0.6669	0.5282	1.956	5.3
8	Co(0.0), Mn(0.1), C5(-9.1), O5(3.6)	0.4963	-0.6684	0.5236	1.926	5.8
9	Co(0.0), Mn(0.0), C6(5.2), O6(-2.2)	-0.5354	0.2528	0.8362	5.286	1.8

[a] Plane equation is Ax + By + Cz = D with x, y, z in orthorhombic coordinates.

[b] Deviations of the atoms (Å × 10^3) from the meanplane are given in parentheses.

[c] Mn deviation 1.654(2) Å.

meanplanes and dihedral angles in Table 36. A labelled SNOOPI diagram is shown in Figure 16 and a sideview of the molecule in Figure 17.

2.3 Description and Discussion of the Structure

The crystal structure consists of the packing of discrete molecules with no unusual inter- or intramolecular contacts. The atom labelling scheme is presented in Figure 16 and a sideview of the molecule, without the substituents on the cobaltacyclopentadiene ring, is illustrated in Figure 17. Hydrogen atoms have been omitted in all diagrams.

The gross features of the structure consist of two methylphenylacetylene ligands linked in a head to head fashion with σ-bonding from the end carbons C(7) and C(10), to the $Co(CO)_3$ group forming a cobaltacyclopentadienyl ring. This ring is inclined towards the $Mn(CO)_3$ moiety, as shown clearly in Figure 17 and allows the Mn to complete its coordination by π-bonding with the delocalized electron density of the butadiene residue of the cobaltacyclopentadiene ring.

There are few structures with accurate Co-Mn bond lengths in the literature and the distance in <u>10</u> of 2.5488(8)Å, while shorter than the covalent radii[79] of 2.65Å, is similar to that found[80] in $(MeCp)MnCo_2(Me_5C_5)_2(CO)_4$ of 2.561Å. The Co-Mn distance in <u>10</u> is slighty longer than the closely related ferrole[81] $(CO)_3Fe(PhC_2Ph)_2Fe(CO)_3$, 2.505(1)Å.

The manganese has three terminal carbonyl groups with similar Mn-C bond lengths of 1.785(4) to 1.798(4)Å. The cobalt atom also has three terminal carbonyls but Co-C(2) is significantly (4.6σ) shorter than the Co-C(1) or Co-C(3) distances; 1.797(4) versus 1.823(4) and 1.829(4)Å. As shown in Figure 17 this carbonyl ligand is almost perpendicular to the

FIGURE 16

SNOOPI Diagram of

$CoMn(CO)_6(MeC_2Ph)_2$ (10)

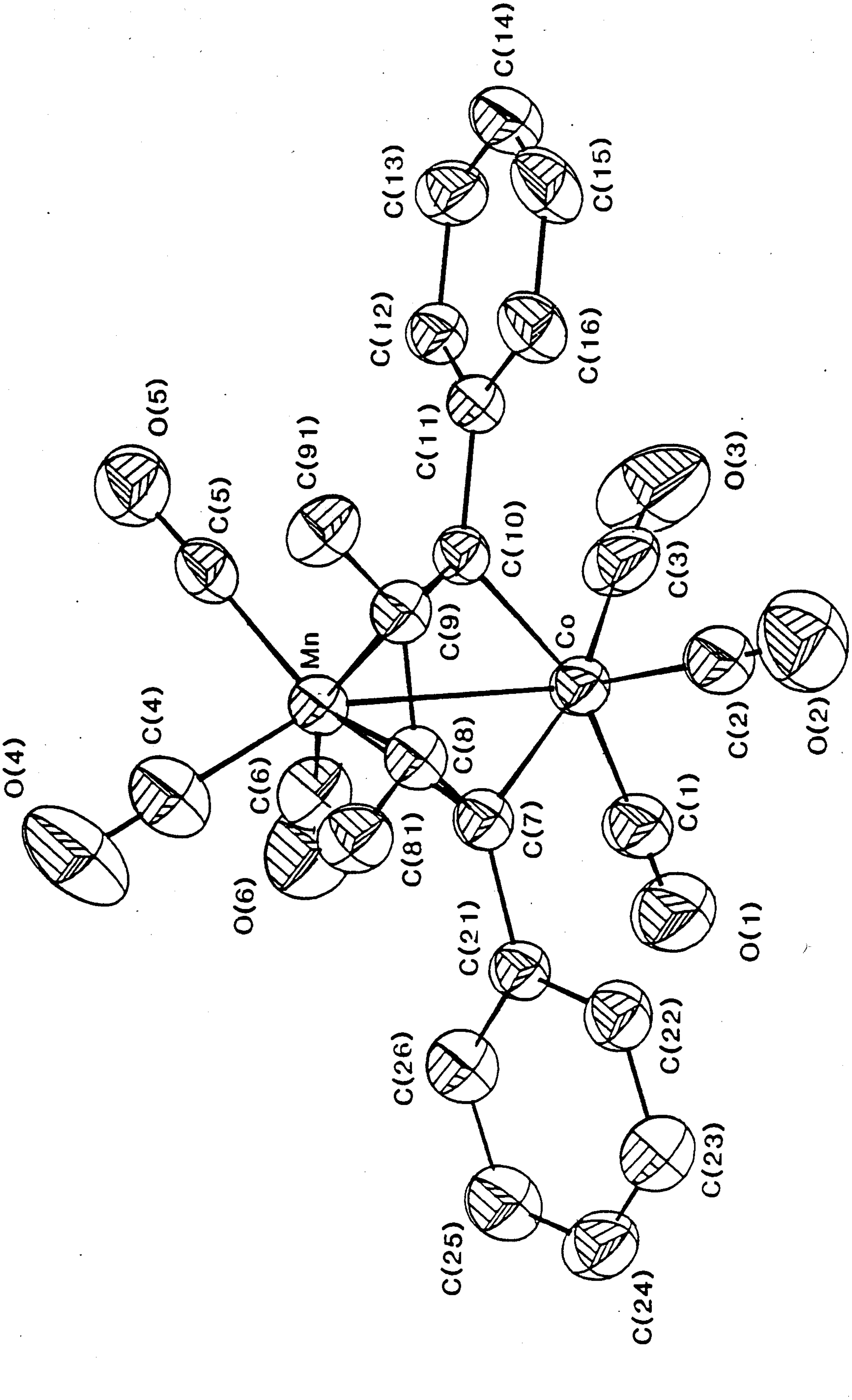
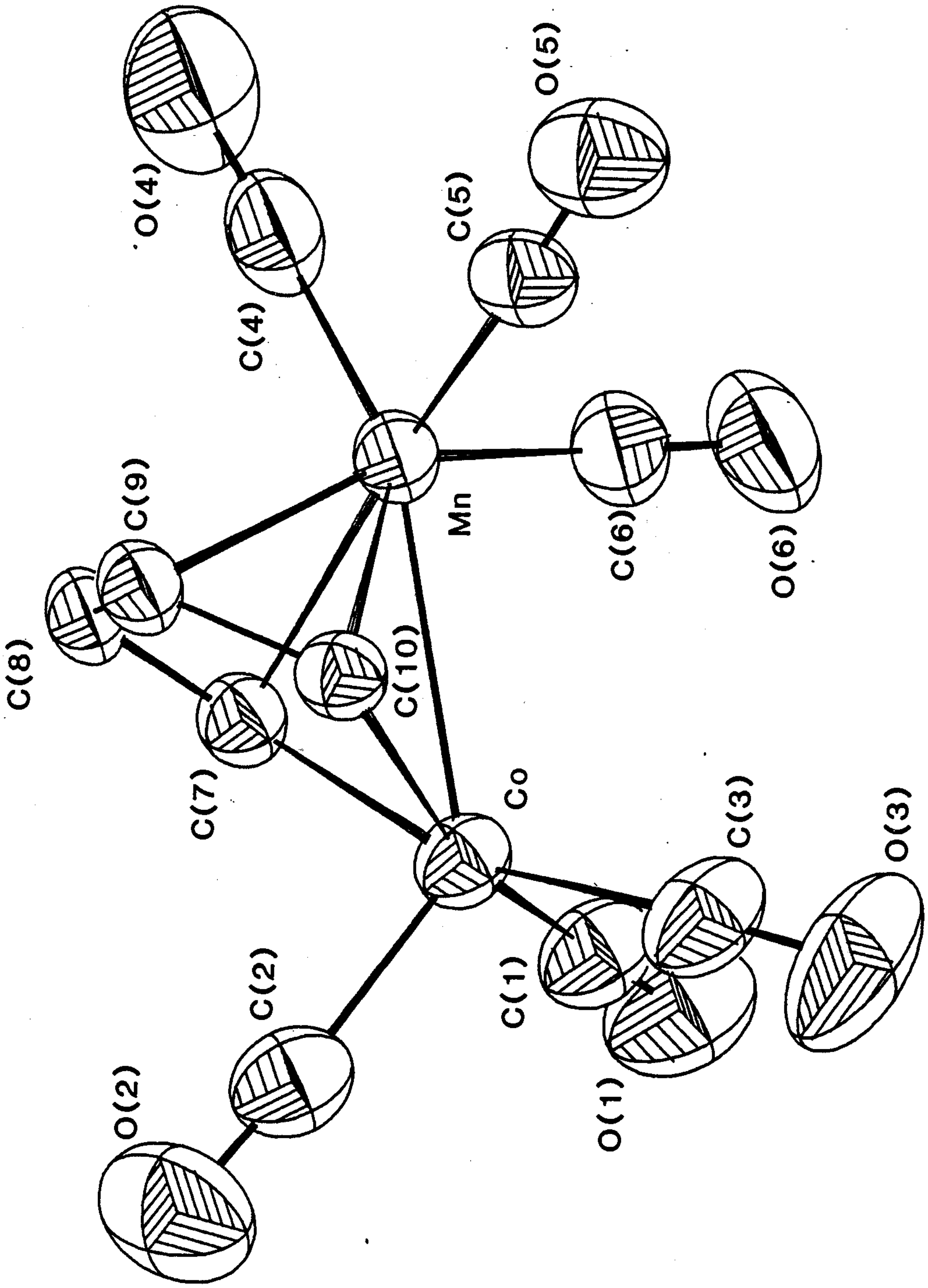

O(4)
C(4)
O(5)
C(5)
Mn
C(6)
O(6)
C(91)
C(9)
C(8)
C(81)
C(7)
C(10)
C(11)
C(12)
C(13)
C(14)
C(15)
C(16)
Co
C(3)
O(3)
C(2)
O(2)
C(1)
O(1)
C(21)
C(22)
C(23)
C(24)
C(25)
C(26)

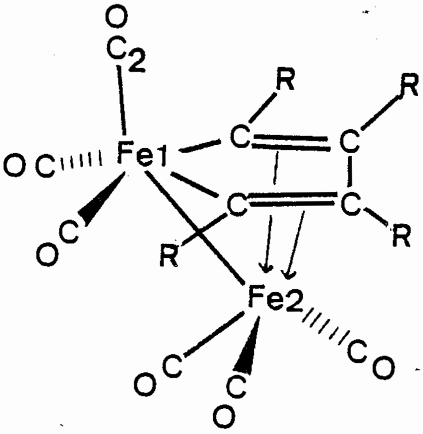
FIGURE 17

Side-view of

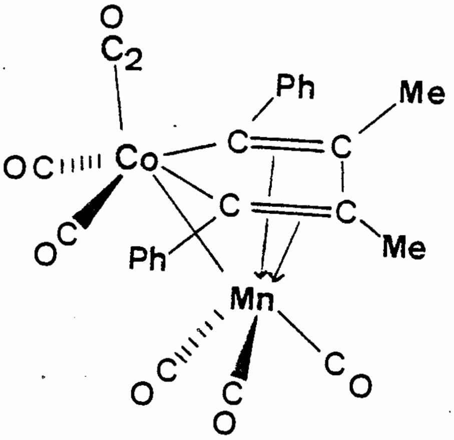
$CoMn(CO)_6(MeC_2Ph)_2$ (<u>10</u>)


O(4)
C(4)
C(5)
O(5)
Mn
C(6)
O(6)
C(8)
C(9)
C(10)
C(7)
Co
C(3)
O(3)
C(1)
O(1)
C(2)
O(2)

cobaltacyclopentadiene ring and similar shortening effects have been noted for the identically positioned carbonyl in the isoelectronic ferrole series[82,83]. However when there are two iron atoms present in the structure, as shown in II-1 a dative Fe(2)→Fe(1) bond must be invoked to allow each metal to achieve an 18 electron count. Fe(1) can relieve this excess charge by increased back donation to the carbonyl groups of which C(2) is in a unique position to accept more electron density than the other



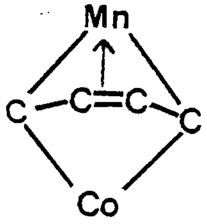
II-1



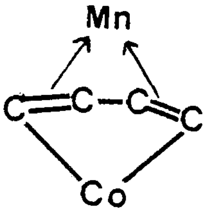
II-2

two. Alternatively, it was suggested[84] that the shortening of this Fe(1)-C(2) bond is due to an increased electron donation from this unique carbonyl to Fe(1), thus helping to balance the electron deficiency of the metal. This latter view is supported by the replacement of C(2) by PPh_3, a stronger σ-donor[83]. In the structure of 10 shown as II-2 a semi-bridging carbonyl is not observed and a dative bond does not have to be invoked since both metals have an 18 electron count. Yet the Co-C(2) bond is still shorter than the other two cobalt-carbon distances. Cobalt is slightly more electronegative than Mn (1.88 versus 1.55)[54] which lends support to increased σ-donation from C(2) to the Co atom as the reason for the observed small shortening effect.

The cobaltacyclopentadienyl ring donates four electrons to the Mn(CO)$_3$ group and two canonical forms of the structure can be written as II-3a and II-3b. Both satisfy the requirement of donating two electrons to the cobalt (2 σ-bonds) and four electrons to the Mn (2 σ and 1 π-bond or 2 π-bonds).



II-3a

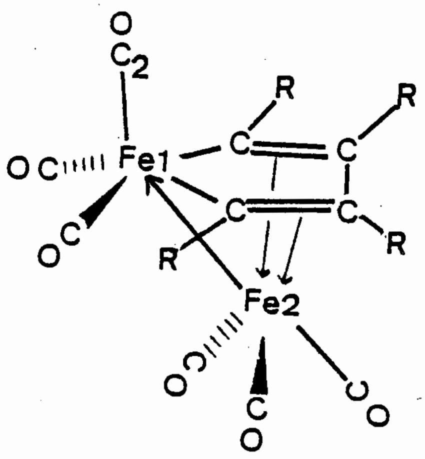


II-3b

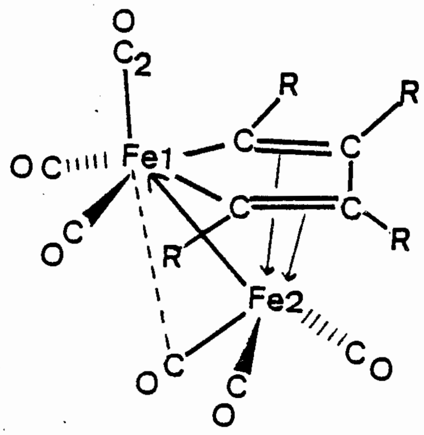
Which, if any, of these two extreme canonical forms represents the molecular structure will be determined by the relevant C-C bond lengths of the butadiene fragment. However, the observed distances in 10, C(7)-C(8), 1.401(5); C(8)-C(9), 1.426(5); C(9)-C(10), 1.423(5)Å; reveal that neither canonical form alone is correct. The true molecular structure has almost equal C-C bond distances. This 'bond levelling' effect is likely a result of back donation from the Mn into the lowest π^* orbital of the diene[84]. This orbital has nodal planes between the doubly bonded C(7)-C(8) and C(9)-C(10) atoms, thus back donation from the Mn would be expected to reduce the C-C bond order making all the distances similar. Structurally characterized ferrole structures[83] have also shown nearly identical butadiene bond lengths.

The manganese-carbon bond lengths to the cobaltacyclopentadienyl ring are not equivalent with Mn-C(7) and Mn-C(10) being shorter than Mn-C(8) and Mn-C(9), 2.099(4) versus 2.177(4)Å. Differing bond lengths between the butadiene fragment and the π-bound metal have also been noted in ferrole structures[84] and in the Fe-Ni ferrole type compound[77], although the differences in M-C bond lengths were smaller than 10 . However the pattern is always similar with the shortest M-C bond to the outer two carbons, C(7) and C(10) and the longest bond to the inner two carbons, C(8) and C(9).

The ferrole structures possess in the solid state either a 'sawhorse' geometry (carbonyls eclipsed) or a 'non-sawhorse' geometry (carbonyls staggered)[84]. These two geometries are shown as II-4a and II-4b with the former having the eclipsing carbonyls and the latter with staggered carbonyls. These structures are interconverted simply by a 60° rotation of the $Fe(CO)_3$ group which is π-bonded to the butadiene fragment. This



II-4a



II-4b

rotation positions one carbonyl in a semi-bridging position. In the structure of 10, the carbonyls do not adopt either the sawhorse or non-sawhorse geometry but are positioned intermediate between the two extremes. The relevant dihedral angles between the carbonyls on opposite metals are 20 and 26°, while angles of 0° or 60° would be expected for II-4a or II-4b, respectively. This 'semi-sawhorse' geometry is a result of two factors.

While adoption of a non-sawhorse geometry is favoured by most ferroles[84] this places one of the carbonyls on Fe(2) in a semi-bridging position approximately 2.4-2.5Å from Fe(1). The formation of a semi-bridging CO ligand in 10 is not necessary since both the Co and Mn already have 18 electrons. However the sawhorse geometry was calculated by Hoffmann[84] to be approximately 0.2eV higher in energy than the non-sawhorse configuration making the sawhorse geometry for 10 also unfavourable. To minimize both these higher energy interactions, a configuration of the carbonyl ligands approximately halfway (26°), between the eclipsed (0°) and staggered (60°) geometries was found to be adopted.

CHAPTER 3

SYNTHETIC AND STRUCTURAL STUDIES ON $CpRe(CO)_2(C_2R_2)$ and $CpRe(CO)_2(N_2R_2)$

3.1 Introduction

This chapter describes the structural investigation of two related types of mononuclear rhenium complexes; the alkyne complex $CpRe(CO)_2(C_2Ph_2)$, and the diazene complex $CpRe(CO)_2(N_2Ph_2)$. The $CpRe(CO)_2$ fragment has a varied and interesting chemistry[85]. It can bind to a wide variety of ligands such as PR_3, CO, N_2R^+, NO^+, olefins, etc. However it is not as common to find this fragment bound to another metal atom and certainly not to 2 or 3 metal atoms in a cluster arrangement. Because of our interest in heterotrimetallic (MM'M") alkyne clusters of the Ni, Co and Fe triads, we wished to extend this chemistry to include the Re group analogs in a similar arrangement such as $Cp_2NiCoRe(CO)_4(RC_2R')$. In such an attempt, using $CpRe(CO)_2(THF)$ and $CpNiCo(CO)_3(C_2Ph_2)$, a trimetallic cluster was not obtained but the mononuclear alkyne complex, $CpRe(CO)_2(C_2Ph_2)$ was isolated in low yield. This compound had been synthesized in 1976 by Nesmeyanov[86] and some properties very briefly reported at that time. We know of no further studies on it. Consequently we have synthesized the compound directly and have carried out a complete characterization including an X-ray structure determination. The analogous complex $CpRe(CO)_2(MeC_2Ph)$ has also been synthesized.

The synthesis and study of transition metal carbonyl complexes containing coordinated diazenes is becoming an area of active investigation. The two nitrogen lone pairs and the N=N π bond provide for a rich variety of coordination modes in contrast to the alkyne ligand. In connection with the structure of the alkyne complex $CpRe(CO)_2(C_2Ph_2)$ and continuing interest in the structures and properties of the related diazenes $CpRe(CO)_2(N_2R_2)$[87] in this laboratory, an investigation of some of the latter was undertaken.

To provide a brief background for the subsequent discussion of these two types of compounds, this introduction continues with an outline of some of the features of alkyne and diazene complexes.

3.1.1 Mononuclear Alkyne Complexes

Many aspects of the synthesis of transition metal alkyne compounds have been dealt with in Chapter 1 and only a brief review will be given here.

3.1.1a Synthesis

The synthesis of mononuclear transition metal complexes involves addition of the appropriate alkyne to a solution of the transition metal compound. Substitution of one of the ligands on the metal by the alkyne yielding the mononuclear alkyne complex is very common and the synthesis of $Cp_2Ti(CO)(C_2Ph_2)$ from $Cp_2Ti(CO)_2$ and C_2Ph_2 is illustrative[88].

It is possible for two alkyne ligands to bond to one metal atom. $Pt(C_2Ph_2)_2$ can be easily synthesized from $Pt(COD)_2$ and two moles of alkyne[89]. Alternatively, one alkyne ligand has been known to displace two 2 e^- electron donors on a transition metal[90] and a representative reaction is shown below.

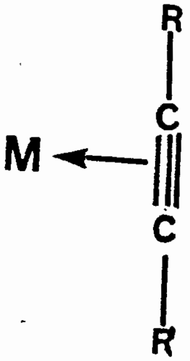
$$W(CO)_3(S_2CNR_2)_2 \quad + \quad EtC{\equiv}CEt \quad --- \quad W(CO)(EtC{\equiv}CEt)(S_2CNR_2)_2 \quad + \quad 2\ CO$$

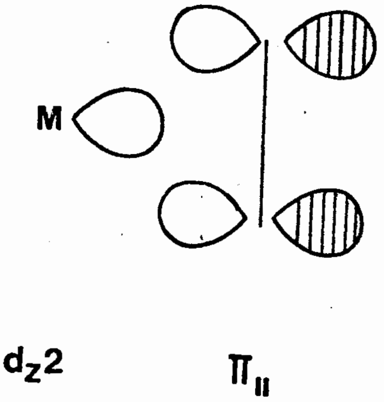
The reduction of Re(V) to Re(III) has also been found to occur upon addition of an alkyne ligand[90] and one reaction is shown below. The triphenylphosphine oxide does not appear in the absence of the alkyne.

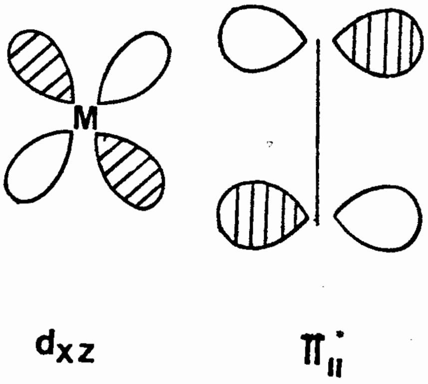
$$ReO_2I(PPh_3) + C_2Me_2 \longrightarrow Re(O)I(C_2Me_2)_2 + OPPh_3 + PPh_3$$

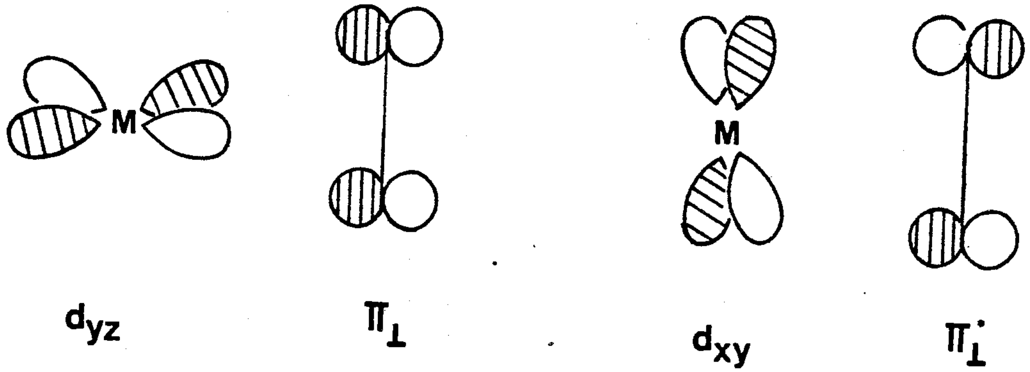
3.1.1b Bonding

A schematic of the transition metal alkyne interaction is shown below and superfically resembles the olefin-transition metal interaction forwarded by Dewar[26] and Chatt and Duncanson[27]. However additional interactions with the alkyne ligand are possible and are discussed further. The alkyne has a pair of π and π^* orbitals (the $\pi||$ and $\pi||^*$ below) which are in the MCC plane and a pair of π and π^* molecular orbitals ($\pi_\perp$ and $\pi^*_\perp$ below)









perpendicular to the MCC plane. Both the $\pi\|$ and $\pi\perp$ have the ability to donate two electrons to the metal atom making the alkyne a potential four electron donor. The majority of mononuclear compounds have the alkyne as a two electron donor but four electron behaviour is certainly known, for example, $CpMo(CO)(CH_3(RC{\equiv}CR)$[91]. Back donation from the metal d_{xz} to the $\pi^*\|$ orbital is similar to that described by Dewar, Chatt and Duncanson for ethylene complexes. An additional interaction which is not present in the ethylene compounds is the possibility of back donation from the d_{xy} orbital of the metal to the $\pi^*\perp$ orbital of the alkyne.

3.1.2 Mononuclear Diazene Complexes

While we are interested in diazenes from a coordination point of view, specifically to the $CpRe(CO)_2$ unit, it is worthwhile to note that much of the current interest in diazenes results from their postulated intermediacy in biological nitrogen fixation. Schrauzer[92] has invoked the parent diazene (N_2H_2) as a crucial intermediate in a variety of nitrogen fixation reactions and diazene has also been considered as as intermediate in the nonenzymatic conversion of coordinated dinitrogen[93]. Interest was also sparked into the coordination chemistry of diazenes when it was discovered that the introduction of metal ions into azo dyes resulted in modification of some of their properties[94]. Very recently, azobenzenes have been found to react with chromium carbenes to yield mixtures of 1,2- and 1,3-diazetidinones[95].

In this chapter we shall not be concerned with the parent diazene, HN=NH, but only with the disubstituted derivatives and in particular the aromatic acyclic derivatives, ArN=NAr.

3.1.2a Preparation of Transition Metal Diazene Complexes

i) Insertion of Diazonium Salts into Metal Hydride Bonds

This particular synthetic route was first discovered by Parshall[96] when he isolated $[Pt(NHNAr)Cl(PEt_3)_2]^+$ from $Pt(H)Cl(PEt_3)_2$ and ArN_2^+. Many other workers have utilized this method for other metals[97,98]. Two major problems occur with this synthetic route: 1) treatment of some metal hydrides with diazonium salts decomposes the diazonium salt to dinitrogen and 2) facile orthometallation of the aryl group is common. These drawbacks make this method less popular than it might have otherwise become.

ii) Reactions with Hydrazines

The reaction of substituted hydrazines with halide complexes of the transition metals can yield diazene complexes. An example[99] is the formation of a platinum azobenzene complex from the platinum (II) dihalide and diphenylhydrazine.

$$PtX_2(PPh_3)_2 + PhNHNHPh \xrightarrow{Na_2CO_3} Pt(PhN{=}NPh)(PPh_3)_2 + 2\ HX$$

iii) Reaction with Diazenes

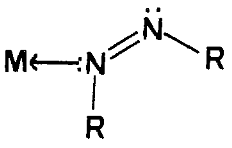
The instability of the parent diazene, HNNH, often precludes the direct reaction of this species with transition metal complexes. However, this route is the most common one for the synthesis of transition metal disubstituted diazene compounds. An example[100] is the reaction of Ni(cyclooctadiene)$_2$ with azobenzene or azotoluene in the presence of PPh$_3$ to yield Ni(N$_2$Ar$_2$)(PPh$_3$)$_2$. Generally the transition metal complex must have a labile ligand for the azo ligand to be able to displace it.

iv) Reaction of Coordinated Dinitrogen

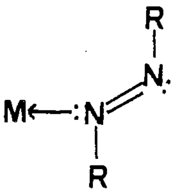
Sellmann[101] has found that phenyllithium will attack the dinitrogen ligand in CpMn(CO)$_2$(N$_2$) and when followed by protonation yields the diazene compound CpMn(CO)$_2$(PhN=NH). This compound was postulated to have a σ-bonded diazene with the bound nitrogen containing the phenyl group. However, Leigh[102] suggests the product may be a hydrazido (2-) species and not a diazene. Confirmation of the structure awaits further information.

3.1.2b Structure and Bonding

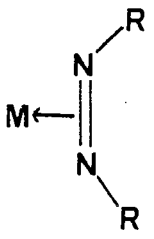
Upon coordination to a metal atom, the diazene or azo ligand can adopt one of three potential configurations shown below as III-1, III-2 and III-3.



III-1



III-2



III-3

The trans structure III-2 has not been authenticated to date although Vrieze[103] has prepared several platinum complexes of formula $(RNNR)PtCl_2L$ ($L = PPh_3$, $AsEt_3$, C_2H_4) and suggested from the $\nu(N{=}N)$ vibration in the infrared that the azo ligand is in a trans configuration. The cis form III-1 has been observed[104,105] in $[ReCl_2(HN_2C_6H_4F)(PEt_3)_2]^-$ and $[RuCl(CO)_2(HN_2Ph)(PPh_3)_2]^+$. The sideways bonded structure has been documented by X-ray crystallography[106,107] for $L_2Ni(PhNNPh)$ ($L = Me_3CNC$, $P(p\text{-}MeC_6H_4)_3$ and $Cp_2Ti(PhNNPh)$[108]. In the two nickel complexes the phenyl groups on the azobenzene ligand are oriented in a trans arrangement but adopt a cis geometry in the titanium compound.

3.2 Synthesis and Characterization

3.2.1 Synthesis

$CpRe(CO)_2(C_2Ph_2)$ (11) was initially observed to be formed in low yield as yellow crystals in the reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $CpRe(CO)_2(THF)$. Other than this, only unidentified decomposition products and $CpRe(CO)_3$ were observed to be formed in this reaction. Nesmeyanov[86] reported the synthesis of 11 in 20% yield by stirring a solution of $CpRe(CO)_2(THF)$ (formed *in situ* in THF by irradiation of $CpRe(CO)_3$ at 10°C for 30 minutes) and C_2Ph_2 for a further 15 minutes at 20°C with no further irradiation. In our hands, this procedure was ineffective at 20°C and furthermore, it gave 11 in only *ca.* 5% yield at 45°C, along with extensive decomposition. The yield improved to *ca.* 15% when the reaction of excess C_2Ph_2 with a concentrated THF solution of $CpRe(CO)_2(THF)$ was conducted in hexanes at 20°C. Finally, 11 was obtained in 30% yield by reacting a large excess of C_2Ph_2 with crystalline $CpRe(CO)_2(THF)$ entirely in hexanes at 20°C. The analogous complex $CpRe(CO)_2(MeC_2Ph)$ (12) was similarly synthesized. The *in situ* irradiation of excess C_2Ph_2 and $CpRe(CO)_3$ in hexanes produced 11 in rather low yield.

$CpRe(CO)(N_2Ph_2)$ (13), $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (14) and $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15) were synthesized by addition of the appropriate azobenzene ligand with $CpRe(CO)_2(THF)$ in hexanes. Yields were seldom greater than 25%. As with 11, very little product could be isolated when THF was used as the solvent although acetone could be substituted for hexanes with no apparent loss in % yield.

3.2.2 Properties

$CpRe(CO)_2(C_2Ph_2)$ (11) is a pale yellow crystalline solid soluble in hexane and polar organic solvents. Its solutions show remarkable thermal stability. It was recovered unchanged after 8 hours in refluxing iso-octane (99°C). The solid and its hexane solution are also stable in air for periods exceeding one week and 12 hours, respectively. 12 was obtained as a pale yellow low melting solid which appeared to be less stable and exhibited decomposition within a few hours when exposed to air.

Compounds 13, 14 and 15 were isolated as orange or light brown crystals. The solids were reasonably stable in air but in THF solution 13 decomposes within 30-45 minutes at room temperature, and 15 was found to slowly decompose in $CHCl_3$ or acetone solutions.

In attempts to substitute either the C_2Ph_2 or N_2Ph_2 ligand or a carbonyl group in these compounds we have found them to be remarkably inert. With 11, no reaction occurred with triphenylphosphine in either hexanes, octane or acetonitrile at or somewhat above room temperature, and in the case of acetonitrile, no substitution by the solvent occurred. Similarly, no reaction of 11 in hexanes was observed with CO at either 1 atm or 1200 psi pressure, and no decomposition of 11 occurred. This is in contrast to the behaviour of $Cp_2Ti(CO)(C_2Ph_2)$[88] in which the diphenylacetylene group is readily substituted by CO to give $Cp_2Ti(CO)_2$ and the decomposition of $Cp_2Ti(CO)(C_2Ph_2)$ is rapid above *ca.* 30°C. $CpRe(CO)_2(N_2Ph_2)$ (13) also did not substitute the azobenzene ligand for a CO at 1 atm.

The reaction of I_2 with either 11 or 13 formed *trans*-$CpRe(CO)_2I_2$ when the reaction was conducted in CH_2Cl_2. However when performed in hexanes, *cis*-$CpRe(CO)_2I_2$[109,110] was formed immediately rather than the corresponding trans compound. A control reaction revealed that cis-$CpRe(CO)_2I_2$ in CH_2Cl_2 is converted to trans-$CpRe(CO)_2I_2$ in less than 10 minutes.

11 and excess N_2Ph_2 were stirred for 2 hours in hexanes but no exchange of alkyne for the azo ligand was noted as monitored by IR. A similar lack of reaction was also observed for the reverse reaction, that is $CpRe(CO)_2(N_2Ph_2)$ with excess C_2Ph_2.

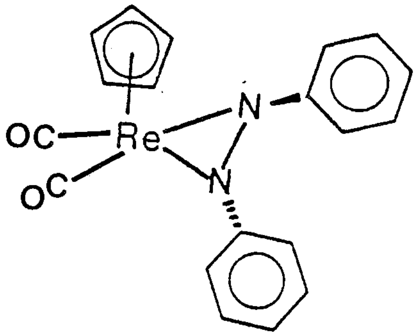
The spectroscopic properties of these compounds will be discussed after the solid state structures of 11 and 13 have been described.

3.3 X-ray Structures of $CpRe(CO)_2(C_2Ph_2)$ (11) and $CpRe(CO)_2(N_2Ph_2)$ (13)

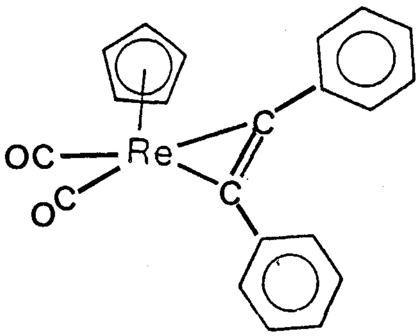
The analysis of the X-ray data and a listing of all the bond lengths and angles, atom coordinates, etc., are in the Experimental section.

Both structures are composed of discrete mononuclear molecules with no unusually short inter- or intramolecular contacts. $CpRe(CO)_2(C_2Ph_2)$ (11) crystallizes in the space group $P2_1/n$ and the asymmetric unit contains two independent molecules, referred to as molecules 1 and 2. Molecule 1 is illustrated in Figure 18, which also provides the numbering scheme. The differences between these crystallographically distinct molecules are only minor and are discussed below. When quoting bond lengths and angles in the discussion only molecule 1 will be mentioned unless there is a significant difference between molecules 1 and 2. Compound 13 crystallized in a non centrosymmetric space group $P2_1$, and a SNOOPI diagram of the molecule with atom labelling is shown in Figure 19.

In both structures the overall geometry of the rhenium coordination sphere conforms to that of a four legged 'piano stool' (shown below for 11 and 13) with the Cp ring as the seat and the four carbons in 11 [C(11), C(12), C(13), C(14)], or the two nitrogen atoms and C(1) and C(2) in 13, as



13



11

FIGURE 18

SNOOPI Diagram of

$CpRe(CO)_2(C_2Ph_2)$ (11)

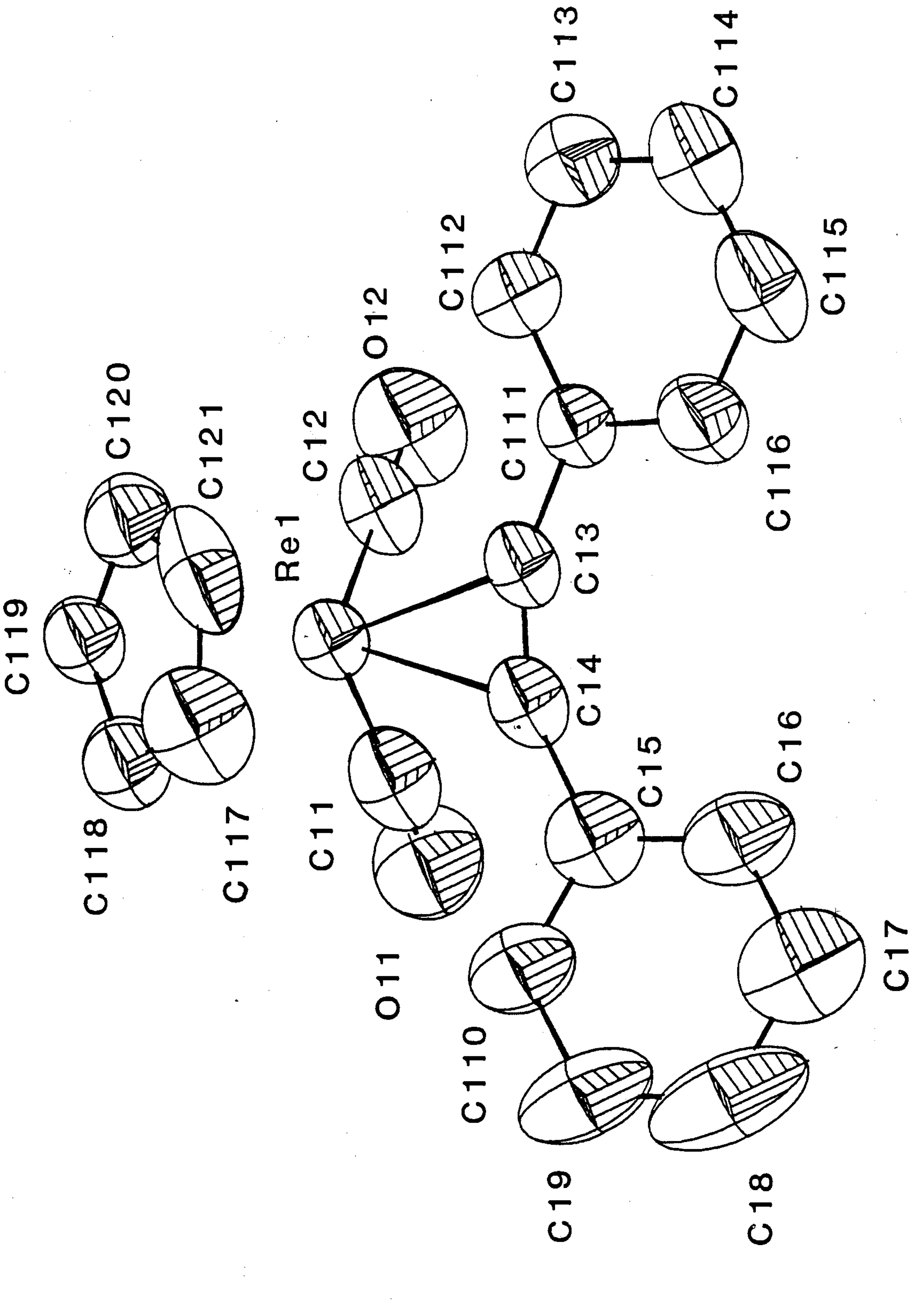
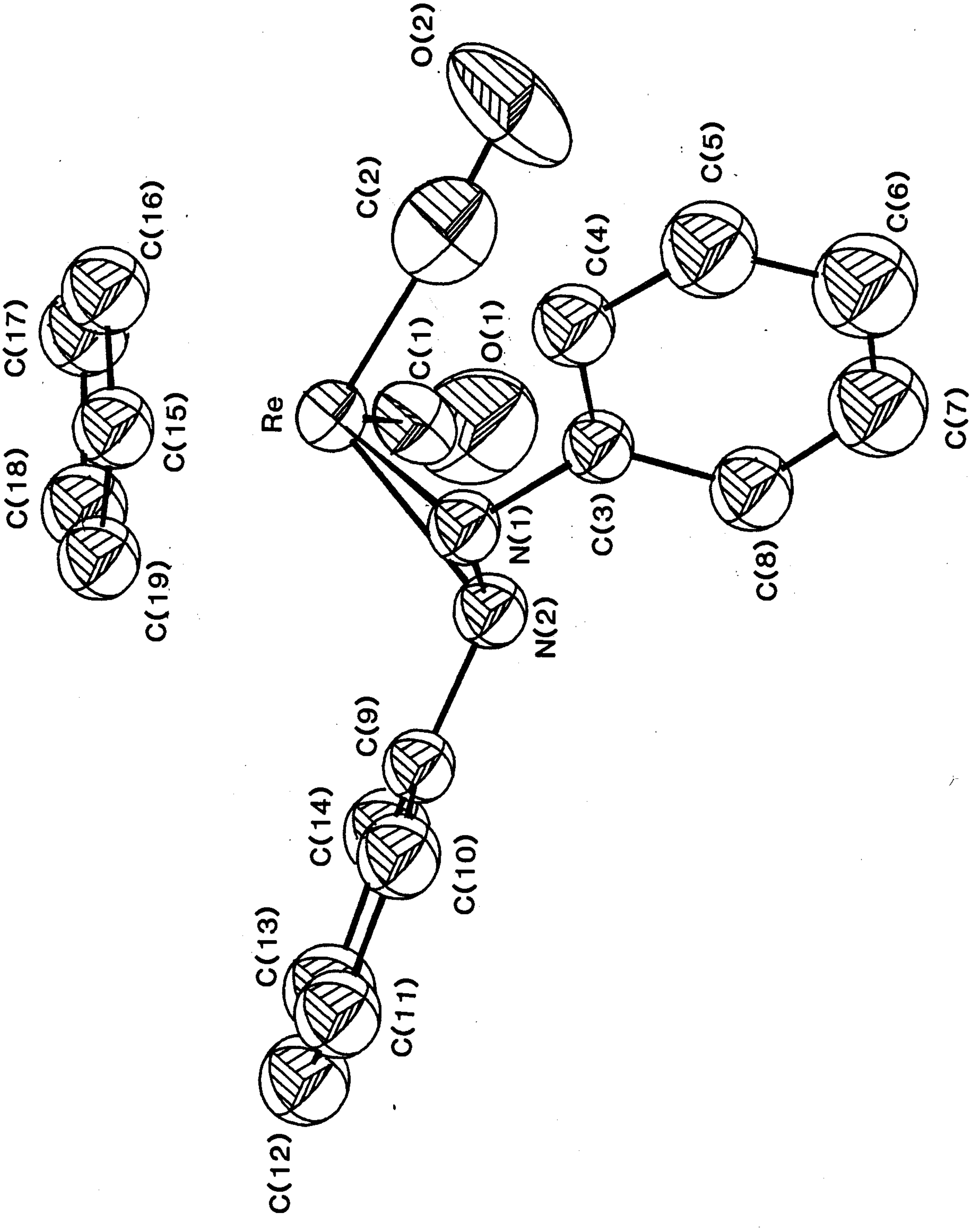

Re1
C11
C12
O11
O12
C13
C14
C15
C16
C17
C18
C19
C110
C111
C112
C113
C114
C115
C116
C117
C118
C119
C120
C121

FIGURE 19

SNOOPI Diagram of

$CpRe(CO)_2(N_2Ph_2)$ (<u>13</u>)


O(2)
C(2)
C(16)
C(17)
C(15)
C(18)
C(19)
Re
C(1)
O(1)
C(4)
C(5)
C(6)
C(7)
C(3)
N(1)
C(8)
N(2)
C(9)
C(14)
C(10)
C(13)
C(11)
C(12)

the base. However these four basal atoms in 13 do not form a well defined plane ($\chi^2 = 154$) unlike that of the four basal atoms in 11 ($\chi^2 = 1.8$) Also the dihedral angle between the Cp ring and the four basal atoms is larger in 13 at 9(1)° than in 11 at 2.3(8)° showing the greater tilt of this ring.

Both N_2Ph_2 and C_2Ph_2 are bound side-on to the rhenium atom. The C_2Ph_2 ligand is symmetrically bound with Re-C(13), 2.188(10)Å, Re-C(14), 2.182(9)Å and C(13)-Re-C(14), 33.5°. In addition, the coordinated diphenylacetylene is no longer linear but displays a cis geometry with the phenyl rings bent at angles of 149.9(9)-152.6(10)° from the C≡C bond axis. This cis bending of the C_2Ph_2 ligand has been observed in all previous mononuclear structures but it is noteworthy that the deformation angles observed here for 11 are among the largest that have been found; the reported range being 135-154°. A summary of the observed angles and C≡C bond lengths for diphenylacetylene compounds is provided in Table 37. As shown in this table all the C≡C bond lengths are elongated with respect to free diphenylacetylene but those of 11 are among the shortest observed.

The geometries of the inner coordination spheres of 11 and 13 are shown in Figure 20. Unlike the C_2Ph_2 ligand in 11, the N_2Ph_2 ligand in 13 is not symmetrically bound to the rhenium atom. One Re-N bond length is 2.048(12)Å and the second 2.136(11)Å. The Re-N-N angles are also not equal and the N-Re-N angle (the 'bite' angle) is 39.5(4)°, significantly larger than observed in 11 which was 33.5(4)°. These small bite angles preclude any expectation of approximate four fold symmetry. The N(1)-N(2) distance of 1.415(17)Å appears to be one of the longest found for a metal bound azobenzene, but as shown in Table 38, which lists the relevant N=N distances for the structurally determined azobenzene compounds, the errors in the bond lengths of the N=N bonds are too large for accurate comparisons.

TABLE 37. The C≡C Bond Lengths (Å), cis-Deformation Angles (deg) and ν(C≡C) Values (cm^{-1}) in Some Mononuclear η²- Diphenylacetylene Complexes.

Complex	d (C≡C)	Ph-C≡C angle	ν(C≡C)	Ref.
PhC≡CPh	1.198(3)	178.1(2)	2223	111,112
$CpRe(CO)_2(C_2Ph_2)$	1.261(14) 1.232(13)	152.6(10), 151.1(10) 151.6(9), 149.9(9)	1848	a
$[Co(C_2Ph_2)(PMe_3)_3]^+$	1.265(7)	143.6(5), 137.5(5)	-	113
$[Co(MeCN)(C_2Ph_2)PMe_3)_3]^+$	1.267(7)	147.0(5), 148.8(5)	1780	113
$Cp_2Mo(C_2Ph_2)$	1.269(7)	-	-	114
$CpNb(CO)(C_4Ph_4)(C_2Ph_2)$	1.26(4)	142(3)	1780	115
$Mo(CNBu)_2(SBu)_2(C_2Ph_2)$	1.28(2)	139(1)	-	116
$Pt(C_2Ph_2)_2$	1.291(5)	152.4(5), 154.1(5)	1881	117
$Ni(C_2Ph_2)(CNBu\text{-}t)_2$[b]	1.276(12) 1.291(16)	148.2(10), 147.5(11) 147.2(14), 151.3(14)	1810	118
$Cp_2Ti(CO)(C_2Ph_2)$	1.285(10)	138.8(7), 145.8(7)	1780	88
$[CpMo(CO)(PPh_3)(C_2Ph_2)]^+$	1.29(2)	135(1), 141(1)	1645	119
$CpW(O)Ph(C_2Ph_2)$	1.29(3)	143.5(4)	1748	120
$Cp_2Nb(O_2CBu\text{-}t)(C_2Ph_2)$	1.29(1)	141.2(7), 146.4(8)	1800	121
$W(CO)(C_2Ph_2)_3$	1.30	140	1702	122
$Mo(S_2CNEt_2)_2(CO)(C_2Ph_2)$	1.313(4)	140.3(3)	-	116
$Pt(C_2Ph_2)(PPh_3)_2$	1.32(9)	140	1740	123
$[pyH][TaCl_4(py)(C_2Ph_2)]$	1.325(12)	138.2(8), 141.2(8)	-	124
$Mo(Porph)(C_2Ph_2)$	1.324(5)	136.4(4), 145(5)	-	114
$Mo(TTP)(C_2Ph_2)C_6H_5CH_3$	1.324(5)	136.4(4), 145.4(4)	-	114

Table 37 (cont'd)

Complex	d (C≡C)	Ph-C≡C angle	ν(C≡C)	Ref.
$Fe(C_2Ph_2)[P(OMe_3]_3$	1.332(10)	-	-	125
$Cp^*TaCl_2(C_2Ph_2)$	1.35(2)	138.5(5), 140.3(5)	-	126
$CpNb(C_2Ph_2)_2(CO)$	1.35(2)	138(4)	-	127

[a] This work. Values are for independent molecules 1 and 2, respectively.

[b] Two independent molecules occur in the asymmetric unit.

Table 38. Structurally Characterised Azobenzene and Transition Metal Azobenzene Compounds.

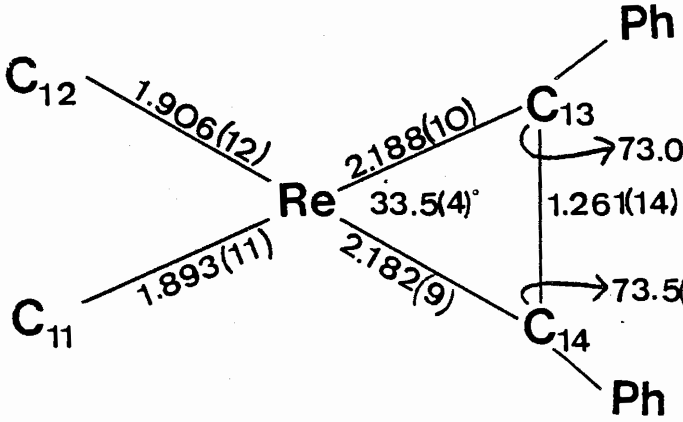
Compound	C-N	N=N	<C-N=N	M-N	<N-M-N	Ref.
trans-azobenzene	1.434(3)	1.243(3)	113.3(3)	-	-	128
cis-azobenzene	1.449(4)	1.253(4)	121.9(3)	-	-	129
trans-p,p'-azotoluene	1.433(4)	1.244(4)	113.8(3)	-	-	130
$Cp_2Ti(N_2Ph_2)$	1.397(9) 1.393(9)	1.339(8)	122.5(3) 123.7(3)	1.965(6) 1.971(5)	39.8(5)	108
$(CpTiCl)_2(\mu\text{-}NPh)(\mu\text{-}N_2Ph_2)$	1.438(6)	1.422(6)	116.1(4)	1.920(4)	-	108
$[P(C_6H_4CH_3)_3]_2Ni(N_2Ph_2)$	1.427(8) 1.423(8)	1.371(6)	111.6(5) 112.2(5)	1.932(5) 1.927(5)	41.6(2)	111
$(t\text{-}BuNC)_2Ni(N_2Ph_2)$	1.421(5) 1.424(5)	1.385(5)	111.6(3) 111.9(3)	1.897(4) 1.899(5)	42.8(2)	106
$CpRe(CO)(N_2Ph_2)$	1.406(20) 1.424(18)	1.415(17)	113.7(10) 114.4(10)	2.048(12) 2.136(11)	39.5(4)	a

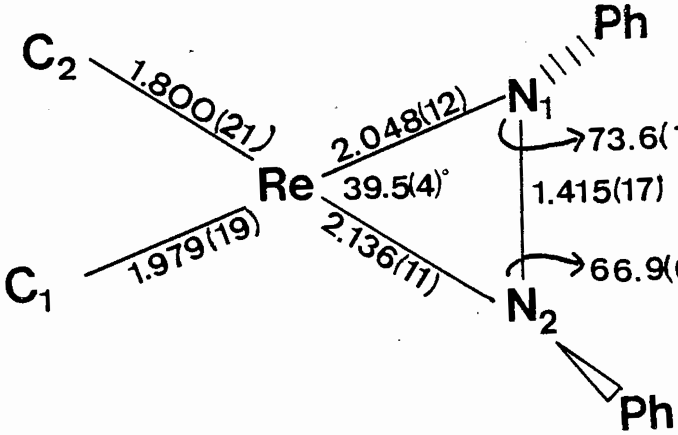
---

[a] This work.

FIGURE 20

View of the Inner Coordination Sphere of $CpRe(CO)_2(C_2Ph_2)$ (<u>11</u>) and $CpRe(CO)_2(N_2Ph_2)$ (<u>13</u>)


Ph
C12
1.906(12)
2.188(10)
C13
73.0(6)°
Re
33.5(4)°
1.261(14)
C11
1.893(11)
2.182(9)
73.5(6)°
C14
Ph


Ph
C2
1.800(21)
2.048(12)
N1
73.6(7)°
Re
39.5(4)°
1.415(17)
C1
1.979(19)
2.136(11)
66.9(6)°
N2
Ph

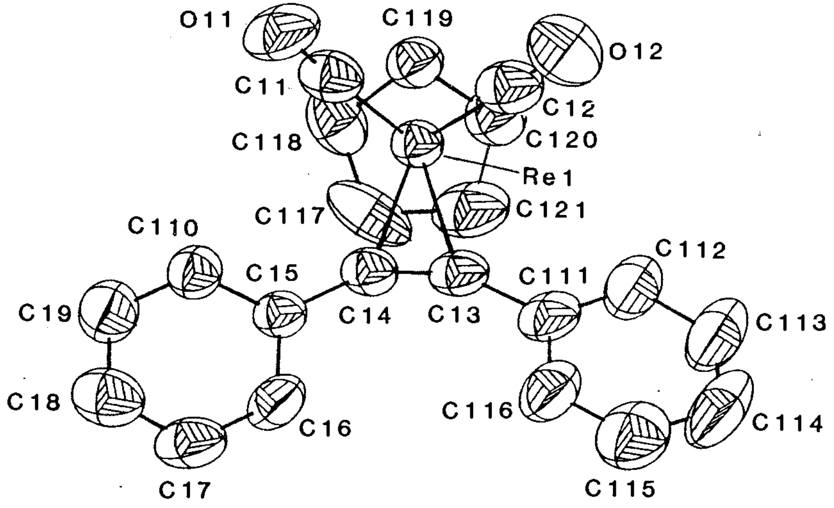
The Re-C(carbonyl) bond lengths for both molecules of 11 are very similar, with distances ranging from 1.893(11) to 1.916(11)Å. However in 13 these lengths, as shown in Figure 20, are not equal with the Re-C(carbonyl) bonds being 1.80(2)Å and 1.98(2)Å.

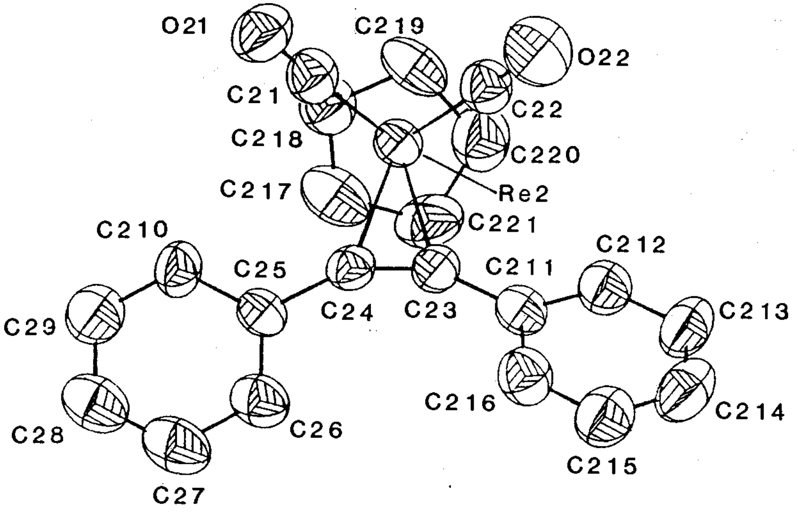
The most obvious dissimilarity between the independent molecules 1 and 2, which are compared in Figure 21 projected onto the plane of the Cp ring, is in the relative orientation of the two phenyl rings in each case. This is most evident by comparing the dihedral angles between the planes of these phenyl rings. In molecule 1 this angle is 16.2(9)°, and in molecule 2 it is 28.2(9)°. In the determination of the X-ray structure of $Ni(C_2Ph_2)(CNBu\text{-}t)_2$, which also crystallizes with two independent molecules in the asymmetric unit, Dickson and Ibers[106] observed an analogous difference in these dihedral angles to be the main dissimilarity in the molecules. They noted that two contributing features may be calculated: i) δ - the dihedral angle between an individual phenyl ring plane and its adjacent C≡C-C(phenyl) skeletal plane, ii) - a 'tilt' angle which is the angle between the two distinguishable adjacent C≡-C(phenyl) skeletal planes in the diphenylacetylene ligand. These values are listed in Table 37.

In 13 the phenyl groups are bent back away from the rhenium atom and the extent of this deviation is shown by the dihedral angle of 44(1)° between the N(1)N(2)C(3) and N(1)N(2)C(9) planes. Ibers[106] has suggested that this angle is an indication of the extent of metal-azo interaction and the larger the angle, the greater the interaction. In $[P(C_6H_4CH_3)_3]_2Ni(N_2Ph_2)$[107] and $[(CH_3)_3CNC]_2Ni(N_2Ph_2)$[106] dihedral angles of 23.5(4) and 26.8(4)° were noted which suggests that a smaller metal-azo interaction is present in these compounds than in 13. This is substantiated by the longer N=N bond length found in 13 than in the two nickel compounds.

FIGURE 21

SNOOPI Diagram of the Two Molecules in the Asymmetric Unit of $CpRe(CO)_2(C_2Ph_2)$ (11)


O11
C119
O12
C11
C12
C118
C120
Re1
C117
C121
C110
C112
C15
C111
C19
C14
C13
C113
C18
C116
C16
C114
C17
C115


O21
C219
O22
C21
C22
C218
C220
C217
Re2
C210
C221
C212
C25
C211
C29
C24
C23
C213
C216
C28
C26
C214
C215
C27

The ortho hydrogens in 13 may be causing a small amount of steric repulsion. The angles N(1)C(3)C(4) = 117.6(13)° and N(1)C(3)C(8) = 123.6(14)° for the N(1)-C(3) bond and N(2)C(9)C(10) = 122.0(11)° and N(2)C(9)C(14) = 116.8(13)° for the N(2)-C(9) bond should be equal for each individual C-N bond. But possibly because of a non-bonded interaction of the ortho hydrogens with the opposite nitrogen atom, a small amount of steric repulsion is introduced resulting in an observed slight twist (5-6°) of the individual phenyl groups.

3.4 Solution Structures

3.4.1 $CpRe(CO)_2(C_2Ph_2)$ (11) and $CpRe(CO)_2(MeC_2Ph)$ (12)

The solution structure of the alkyne compounds 11 and 12 appear to be comparable to that observed for 11 in the solid state. The IR spectra (hexanes) of 11 and 12 display the expected 2 $\nu(CO)$ absorptions with those of the MeC_2Ph derivative at slightly lower wavenumber (1976, 1899 cm^{-1}) than observed for 11 (1980, 1904 cm^{-1}). The ^1H NMR spectra of both compounds show a single peak due to the Cp protons and a multiplet assignable to the phenyl protons. In addition 12 has a single methyl resonance at δ2.75. The ^{13}C and $^{13}C\{^1H\}$ spectra allow all the carbon resonances of 11 and 12 to be assigned (see Experimental Section) and at room temperature both exhibit single carbonyl resonances. In 11 this could result from a rigid structure with equivalent carbonyl groups like that observed in the crystal structure. This cannot be true for 12 which contains the unsymmetric alkyne MeC_2Ph, and suggests in each case the probable rotation of the alkyne ligand about the Re-alkyne axis. In 12 the ^{13}C spectra at -60°C exhibit two equal intensity carbonyl carbon resonances at δ 206.5 and δ 206.0, but the resonances for all other carbon atoms remain essentially unperturbed. This is consistent with a single frozen-out structure and this must have the PhC_2Me ligand oriented parallel, not perpendicular, to the plane of the Cp ring as is observed for the PhC_2Ph ligand in the X-ray structure of 11.

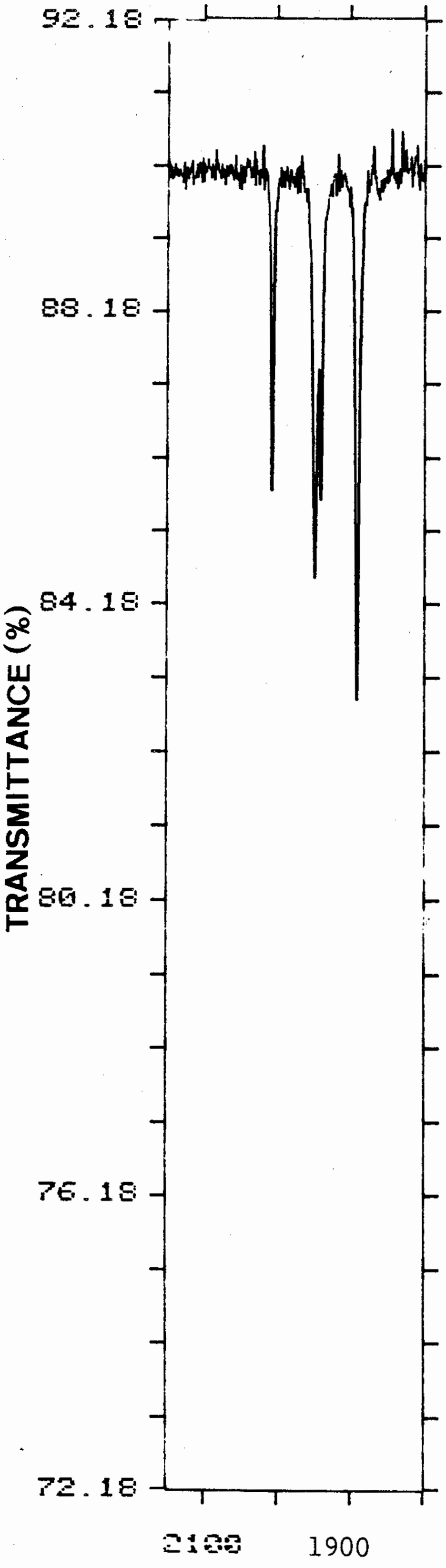
An NOE experiment was performed by irradiation of the Cp resonance and observing the increase in the resonances of the phenyl protons. The para protons were not observed to be enhanced but the meta protons were partially enhanced and the ortho protons of the phenyl ring were fully enhanced.

3.4.2 $CpRe(CO)_2(N_2R_2)$ [R = Ph (13), C_6H_4Me-p (14), C_6H_4F-p (15)]

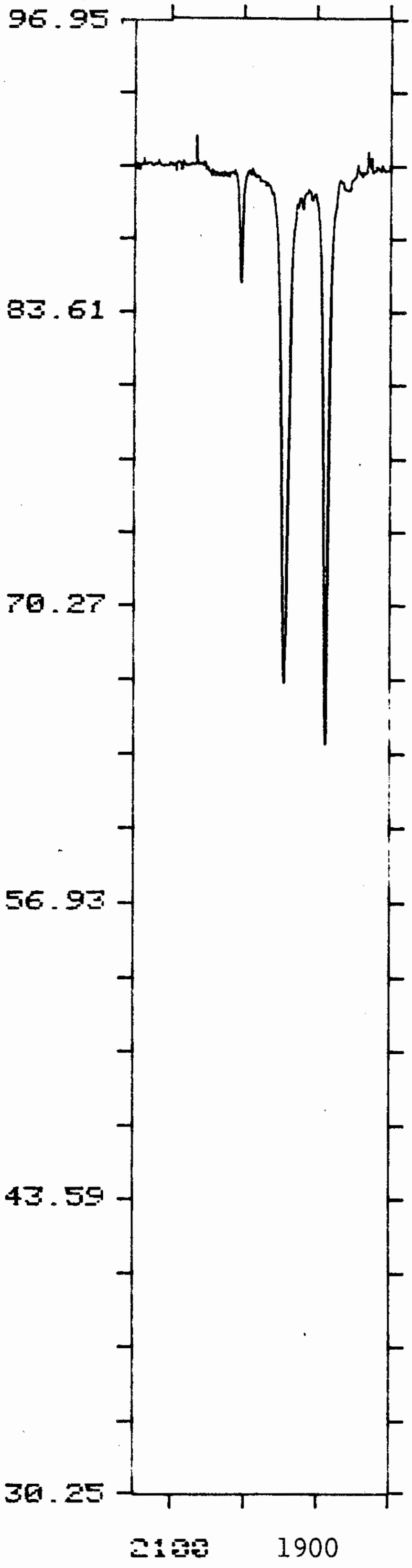
The IR spectra of 13, 14 and 15 show more carbonyl bands than are to be expected if only the structure determined for 13 is present in solution. All three spectra (hexanes) are shown in Figure 22 for comparison. Compound 13 has four sharp bands at 2006, 1947, 1939 and 1890 cm^{-1} as has 15 at 2008, 1950, 1941 and 1893 cm^{-1}. However, for 14 there are only three discernible absorptions at 2004, 1945 and 1887 cm^{-1} with the latter two very strong in comparison with the higher wavenumber band. Most probably the 1945 cm^{-1} peak consists of two nearly coincident bands. A decrease in intensity of the $\nu(CO)$ bands in different solvents, allows assignment of the spectrum as two pairs of bands, each pair attributable to an individual isomer. As an example, for 13, in CH_2Cl_2 the bands at 2006 and 1939 cm^{-1} are reduced to approximately one-third intensity clearly showing these bands belong to one isomer. Removal of the CH_2Cl_2 and redissolving in hexanes restores the original spectrum. Table 39 lists the $\nu(CO)$ bands of each isomer of 13, 14 and 15 and those of $CpRe(CO)_2(C_2Ph_2)$ and cis-$CpRe(CO)_2I_2$ for comparison. Isomer A has arbitrarily been assigned the higher wavenumber $\nu(CO)$ bands and isomer B the lower $\nu(CO)$ absorptions.

FIGURE 22

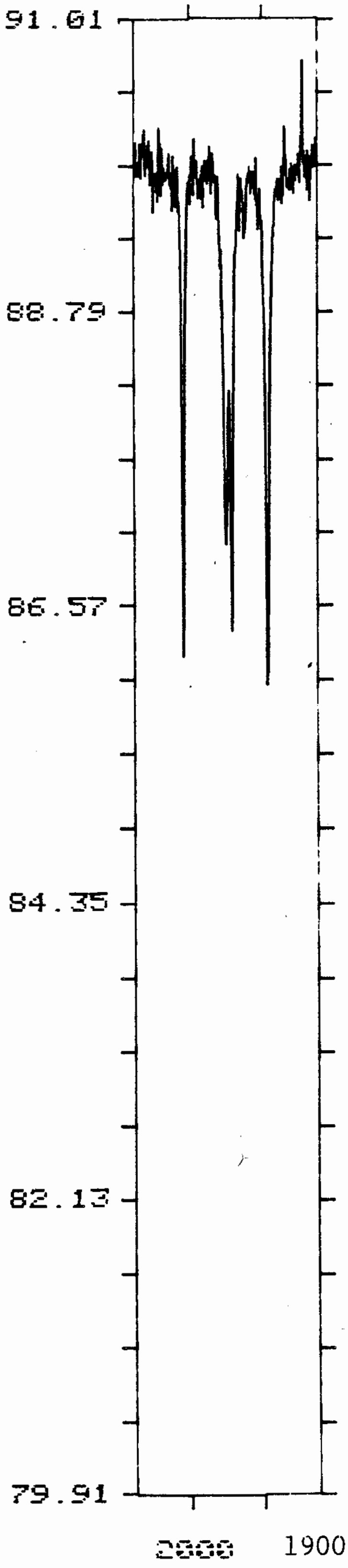
IR Spectra (hexanes) of 13, 14 and 15


92.18
88.18
84.18
80.18
76.18
72.18
TRANSMITTANCE (%)
2100
1900

13


96.95
83.61
70.27
56.93
43.59
30.25
2100
1900

14


91.01
88.79
86.57
84.35
82.13
79.91
2000
1900

15

WAVENUMBER

Table 39. ν(CO) Bands for Mononuclear Rhenium Compounds

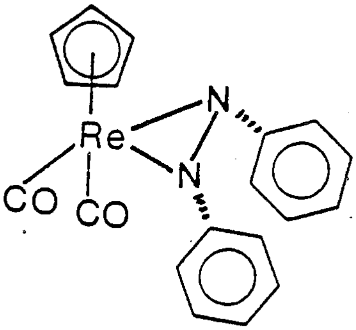
Compound	ν(CO) cm^{-1} (hexanes)
$CpRe(CO)_2(N_2Ph_2)$ (Isomer A) (13)	2006, 1939
$CpRe(CO)_2(N_2Ph_2)$ (Isomer B) (13)	1947, 1898
$CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (Isomer A) (14)	2004, (ca. 1945)
$CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (Isomer B) (14)	1945, 1887
$CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (Isomer A) (15)	2008, 1941
$CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (Isomer B) (15)	1950, 1893
$CpRe(CO)_2(C_2Ph_2)$ (11)	1980, 1904
cis-$CpRe(CO)_2I_2$	2040, 1977

The infrared spectrum of 13, 14 and 15 changes as the solvent polarity ($CHCl_3$, acetone) is increased. The lower wavenumber absorptions i.e., isomer B, become dominant and the bands arising from isomer A are reduced to approximately one-quarter their original intensity in hexanes. In addition the $\nu(CO)$ absorptions are much broader in the more polar solvents and as a consequence the 'inner two' bands, e.g., 1947 and 1939 cm^{-1} for 13, merge into one absorption.

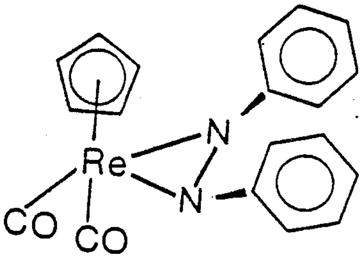
From the infrared data in Table 39 it is possible to deduce some information regarding the probable geometry of these two isomers. The $\nu(CO)$ bands of isomer A are more closely related to a Re(III) compound, for example, cis-$CpRe(CO)_2I_2$, than are the bands of isomer B. In addition, N_2Ph_2 appears to be a better π-acceptor than is C_2Ph_2[131] so an increase in wavenumber for the $\nu(CO)$ bands (by comparison with those for $CpRe(CO)_2C_2Ph_2$)) would be expected for the isomer in which the azobenzene is bound in the same fashion (side-on) as C_2Ph_2 is in $CpRe(CO)_2(C_2Ph_2)$. Since side-on configuration of the azo ligand was confirmed in the X-ray crystal structure, solution isomer A is proposed to have the N_2Ph_2 ligand bound side-on also; this is therefore present as the minor isomer in solution. Though no information is available from the IR spectrum regarding the stereochemistry of the two phenyl groups, they are for the moment regarded to be the same as in the crystal , i.e., trans (structure A-1).

The KBr spectrum of powdered $CpRe(CO)_2(N_2Ph_2)$ shows three $\nu(CO)$ bands at 2001, 1928 and 1900 cm^{-1} rather than the two anticipated from the structure. Using only a small number of crystals from the same sample that furnished the crystal used for X-ray analysis, a KBr spectrum was obtained identical to that obtained from the powder. It proved impossible to grow crystals from just a polar solvent ($CHCl_3$, CH_2Cl_2, acetone), without the addition of a non-polar solvent such as hexanes or pentanes. It was hoped that use of only a polar solvent would favour the second isomer for crystal growth. Similarily, X-ray quality crystals could not be grown for 14, which appears to exist in solution as mainly one isomer. Thus, it was not possible to obtain a satisfactory correlation between the solid state IR and X-ray structure to use in further support of the solution IR assignments of isomers A and B.

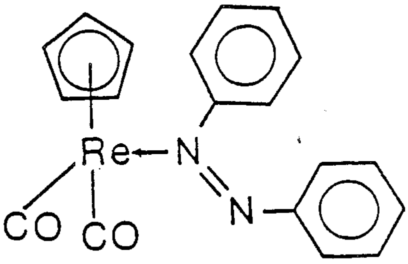
There are a number of possibilities for the geometry of isomer B. These are shown below. A-2 and A-3 have the azo ligand also bound in an η^2 fashion, but with the aromatic rings oriented in a cis geometry. The rings could be either cis to the carbonyl ligands as in A-2 or cis to the Cp



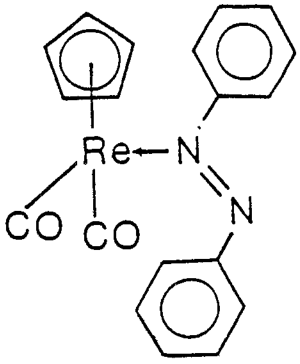
A-2



A-3



B-1



B-2

ligand as in A-3. Alternatively, the azo ligand could be bound to the rhenium in an η^1 fashion, donating the two electrons of a nitrogen lone pair. Again the aromatic rings could be in two different possible orientations, cis (B-1) or trans (B-2). Because A-2 and A-3 resemble a Re(III) species similar to isomer A-1, (13), these would be expected to have ν(CO) values similar to A-1*. But B-1 and B-2 would be expected to have lower ν(CO) values than isomer A-1 since they are more effectively Re(I) compounds with the N_2Ph_2 ligand bound as a Lewis base. Thus on the basis of the observed IR values isomer B is assigned the σ-bonded structure which is represented as B-1 or B-2. In an effort to obtain more information relating to the solution structures of isomers A and B, attention was turned to NMR spectroscopy.

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* The results of variable temperature NMR spectroscopy (see below) indicate that structures A-2 and A-3 contribute to the fluxionality of isomer A in solution.

3.4.2a CpRe(CO)$_2$(N$_2$Ph$_2$) (13)

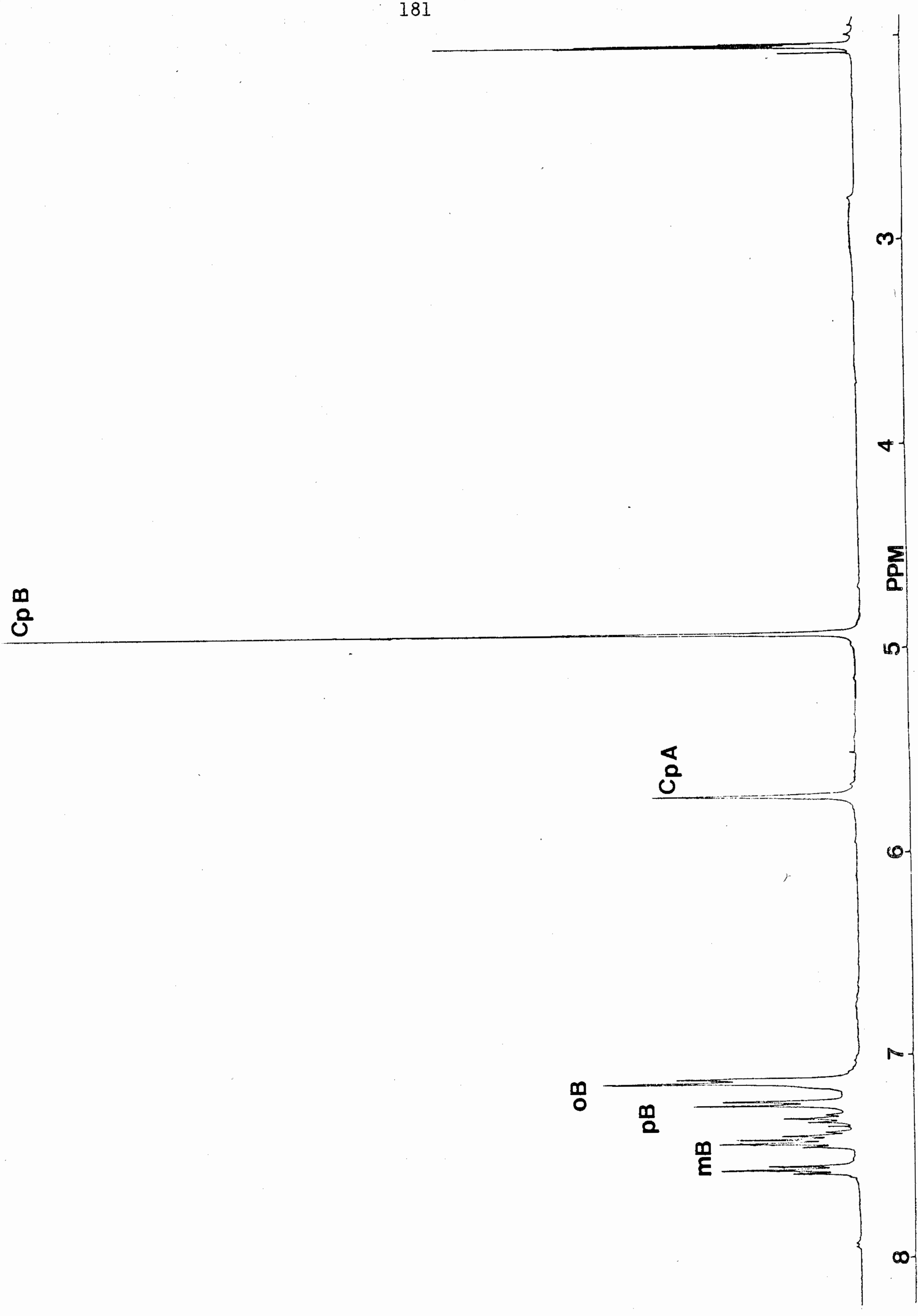
The proton spectra were obtained for solutions in acetone-d$_6$ or CDCl$_3$ which, from the IR data, will favour the presence of isomer B. Since at the present stage there is no evidence on which to distinguish between possibilities B-1 or B-2 as the correct structure for the major isomer B, any reference to the σ-bonded isomer will be made as "isomer B". As shown in Figure 23, in addition to resonances of the major isomer B, the Cp resonance due to isomer A-1 is also visible and is slightly broader than the Cp peak for the major isomer. The phenyl region of 13 only shows the ortho, meta and para protons due to the major isomer B, and none for the minor isomer A-1.

The ^{13}C and ^{13}C{^{1}H} room temperature spectra of 13 essentially confirm the proton spectra. Two Cp peaks due to the major and minor isomers, and resonances due to two distinct phenyl rings of the major isomer are observed. The Cp resonance of the minor isomer is slightly broader than the Cp peak of the major isomer, similar to the ^{1}H NMR data. Only one peak was observed in the carbonyl carbon region at room and lower temperatures.

We have suggested from the IR data that the minor isomer in solution has the side-on structure that was characterized by the X-ray structure determination. The identity of the major isomer is unknown but was tentatively assigned as either B-1 or B-2 because of the decrease in ν(CO) values that a coordinated Lewis base would be expected to cause as compared to a π-bound acid ligand. From the ^{1}H and ^{13}C data both A-2 and A-3 can again be eliminated as possible candidates for the structure of the major isomer, since resonances for two distinct phenyl rings are observed in the ^{1}H and ^{13}C NMR, and A-2 and A-3 have their phenyl rings in equivalent

FIGURE 23

^{1}H NMR (acetone-d_6) of $CpRe(CO)_2(N_2Ph_2)$ (13)


Cp B
Cp A
oB
pB
mB
PPM
3
4
5
6
7
8

environments and would only show one set of phenyl resonances in the NMR. A 50:50 mixture of A-2 and A-3 is also not possible as this would produce two equal intensity Cp peaks in the NMR and a total of six ν(CO) bands (4 for the major isomers and 2 for the minor isomer) in the IR, neither of which is observed. Both of the alternative structures B-1 and B-2, have the phenyl groups of the azobenzene ligand in non-identical environments and either structure would result in the observance of distinct phenyl proton or carbon resonances. Rotation about the Re-N bond in B-1 or B-2 would result in only one carbonyl carbon resonance being observed in the ^{13}C NMR.

To help establish or confirm the geometry of the minor isomer A in solution as A-1 an NOE experiment was performed. The Cp resonance of the minor isomer (δ 5.72) was irradiated in an effort to selectively enhance the resonances of the phenyl protons of the minor isomer which may be obscured in the ^1H NMR by the phenyl protons of the major isomer. If the geometry of the minor isomer is as observed in the X-ray structure, enhancement of the protons on the phenyl ring which is closest to the Cp ring would be expected. The phenyl rings of the alkyne ligand in $CpRe(CO)_2(C_2Ph_2)$ (<u>11</u>) are further away from the Cp ligand than the phenyl ring in $CpRe(CO)_2(N_2Ph_2)$ (<u>13</u>) and, as mentioned previously (sec. 3.4.1), enhancement of the ortho and meta protons is observed for <u>11</u>. Upon irradiation of the Cp resonance of the minor isomer of <u>13</u> an ^1H NMR spectrum identical to that shown in Figure 24 was observed. Saturation transfer from the minor Cp peak to the major Cp resonance has occurred and then subsequently, an NOE transfer from the protons of the Cp ring of the major isomer to the protons of both phenyl rings. The transfer between the Cp peaks of the major and minor isomers firmly establishes that reversible interconversion is occurring. Irradiation of the Cp resonance of the major isomer also resulted in transfer

FIGURE 24

NOE Experiment on $CpRe(CO)_2(N_2Ph_2)$

a) Expansion of the Phenyl Region

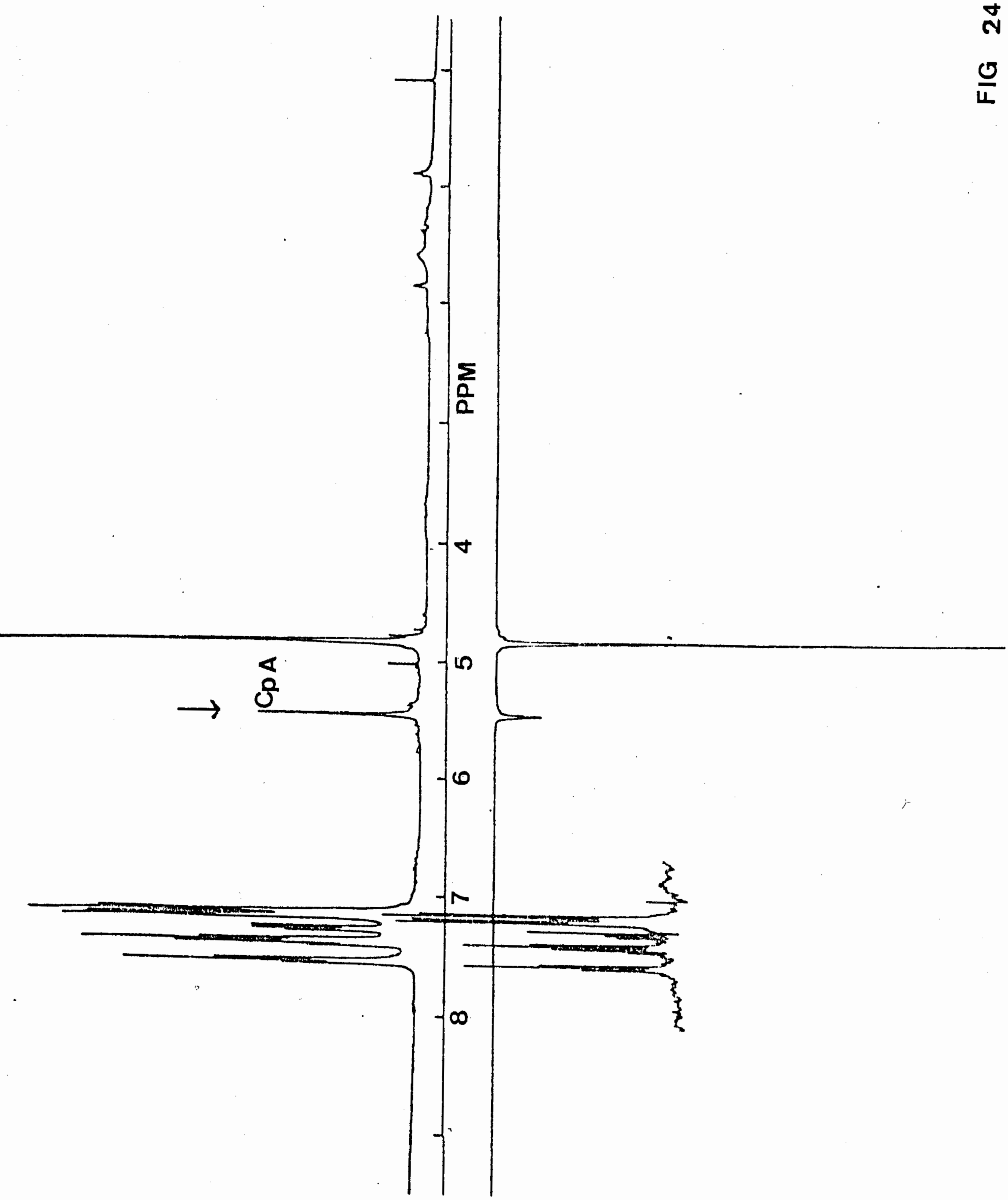


FIG 24

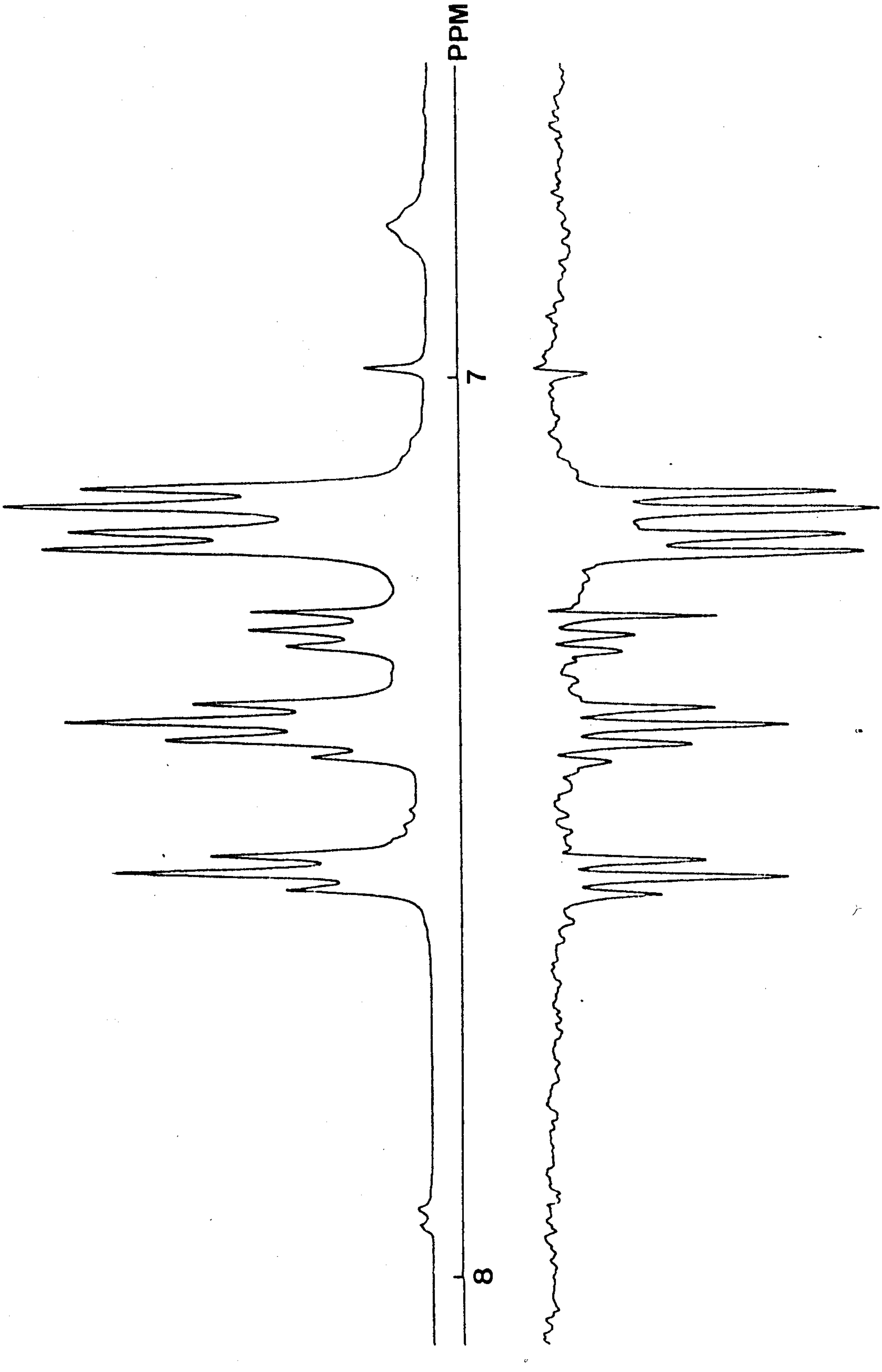
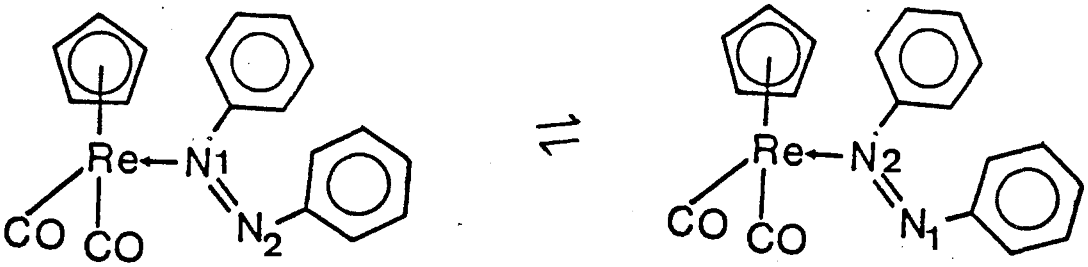


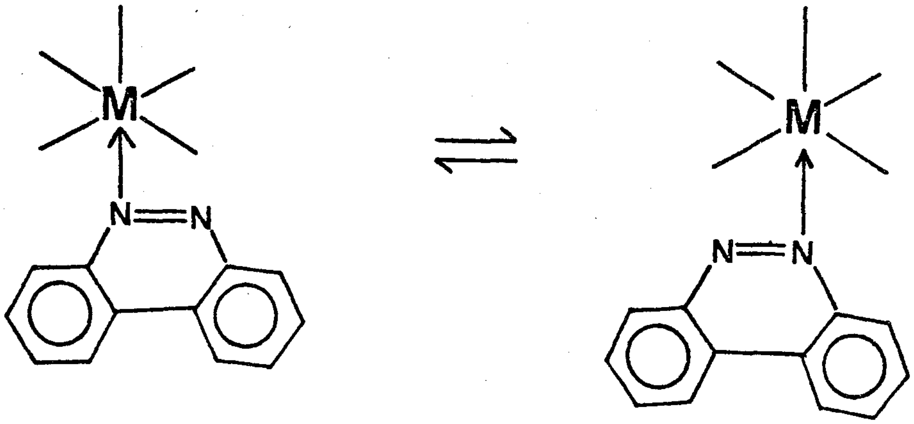
FIG 24 a

to the Cp peak of the minor isomer and enhancement of the phenyl protons of the major isomer. This transfer was observed even with using shorter NOE buildup times (5, 3, 1, 0.3, 0.1 sec) than used originally (10 sec). Shorter buildup times result in less NOE transfer per pulse and poorer quality spectra, hence buildup times less than 0.1 sec were not pursued.

Since the phenyl protons of both phenyl groups of the major isomer are enhanced by NOE transfer, both of these groups must approach the Cp ring. This can occur for the major isomer as shown below. The phenyl ring on N_1 initially is close to the Cp ring and coordination site exchange with the N_2 of the azobenzene ligand would put the second phenyl group in closer proximity to the Cp group. However this exchange is not fast enough to average the chemical shifts of the protons of the phenyl ligands.



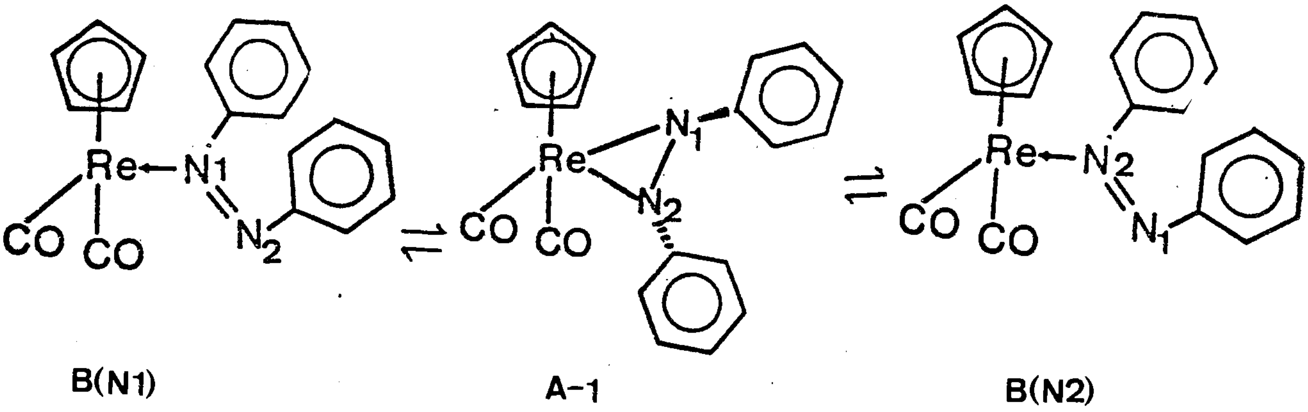
There is precedent for coordination site exchange between the nitrogens of an azo group in an η^1 bonded complex. Nixon[132] and Herberhold[133] have recorded this type of exchange in $M(CO)_5L$ (M = Cr, Mo, W; L = benzo[c]cinnoline) and $ArCr(CO)_2$(2,3-diazabicyclo-[2.2.1]hept-2-ene) (Ar = substituted benzene rings) respectively. The exchange for the $M(CO)_5$(benzo[c]cinnoline) complex is shown below. Exchange between the two



nitrogens on the benzo[c]cinnoline was found to occur and is an example of a η^1-η^1 shift. However the benzo[c]cinnoline and the 2,3-diazabicyclo-[2.2.1]hept-2-ene are fixed into a cis geometry and cannot form products where the groups on the nitrogen are in a trans position. Acyclic cis azo ligands also undergo similar coordination site exchange. $M(CO)_5L$ (M = Cr, Mo, W; L = cis-dimethyldiazene (c-DMD)) were shown recently by Ackerman[134,135] to be fluxional and undergo coordination site exchange, but in addition Ackermann notes that for the complexes where L = trans-dimethyldiazene (t-DMD) there is no observable exchange. Unfortunately, a direct analogy between Ackermann's and our work is not possible since Ackermann did not make use of any aromatic azobenzene ligands.

For the η^1-η^1 coordination site shift in $ArCr(CO)_2(azo)$[133], $\Delta G^\ddagger$ values of 71.1-84.5 kJ mol^{-1} were recorded depending upon the substituent on the benzene ring. Similarly $\Delta G^\ddagger$ values of 62.8, 48.1 and 62.8 (± 0.4) kJ mol^{-1} were recorded[134] for $M(CO)_5$(c-DIPD) with M = Cr, Mo and W respectively. Coalescence experiments (Brüker WM-100) on the Cp peaks of the major and minor isomer of 13 established a $\Delta G^\ddagger$ value of 67.4 ± 0.4 kJ mol^{-1}. This is the value for the η^1-η^2 shift between isomer A-1 and isomer B and cannot be directly compared to the values obtained by Ackermann[134] for $M(CO)_5$(c-DIPD) since the latter involve an η^1-η^1 shift. There have not been any reports in the literature of an η^1-η^2 shift of an azo ligand for comparison. Only very recently[136,137] was an η^1-η^2 shift established for any ligand, specifically a phosphaalkene ligand ($R-P=CR_2$). This ligand exhibits a coordination shift between the phosphorus lone pair and the phosphorus-carbon double bond in the complex $Pt(PPh_3)_2(P(mesityl)=CPh_2)$. However, $\Delta G^{\ddagger}$ values were not given for this process.

We have established that a reversible interconversion is occurring between isomer A-1 and isomer B, and that exchange of the phenyl group positions in isomer B is occurring. This process is probably represented by Scheme III-1. The major isomer B is interconverting with the minor isomer A-1 by a σ-π or an η^1-η^2 shift. The three membered ring of A-1 can open to



SCHEME III-1

reform B or to form B', these two compounds differing only in which particular nitrogen is bonded to the rhenium atom.

In the room temperature $^{13}C\{^{1}H\}$ NMR spectrum of 13 (Figure 25), the Cp resonance of the minor isomer, Cp A, is much broader than the Cp resonance of the major isomer, Cp B. The phenyl carbon resonances of the minor isomer are very broad and are seen as large humps centered about δ 129, 123 and 120. The broadness encountered for these carbon resonances is indicative of a second fluxional process occurring and that the minor isomer is undergoing exchange with further species. In an attempt to obtain more information on this process a number of lower temperature $^{13}C\{^1H\}$ spectra were recorded.

Figure 26 shows the aromatic region in the $^{13}C\{^1H\}$ NMR of 13 at 221 K. Two of the broad resonances in Figure 25 have sharpened into two lines and the third broad resonance at ~119.5 ppm has disappeared. However subsequent cooling to 200 K (Figure 27) and to 189 K (Figure 28) results in further splitting of the lines until the limiting spectrum is obtained. A total of 11 and possibly 12 lines (one may be under the 122 ppm peak of the major isomer) are observed. While it is difficult to follow the emergence of new peaks precisely, a labelling scheme such as that shown in Figure 28 is a possibility. If the ordering of aromatic carbons is similar to that in the major isomer, then the resonances labelled A and B would be due to the ortho protons of the minor isomer(s) and the labels C-E would be assigned to the meta and para carbons. Regardless of the actual assignment of the aromatic protons of the minor species, there are a minimum of 11 lines, and possibly 12, present. This cannot be attributed to only one minor isomer in solution, since the maximum number of phenyl carbon resonances attributable to one isomer is six (not including the ipso carbons), but must be due to two or more minor species. It is not clear whether further splitting would occur below 189 K, or whether some of the broadening of the resonances is due to the viscosity increase of the solvent. However, very little broadening was observed in the resonances of the major isomer which would tend to refute the viscosity argument.

While only the aromatic region was shown in Figure 25 to 28, the other regions were examined. However little change could be discerned. Figure 29 shows the carbonyl carbon, ipso carbon and Cp carbon region of the limiting spectrum (189 K). Only one sharp carbon resonance in the CO

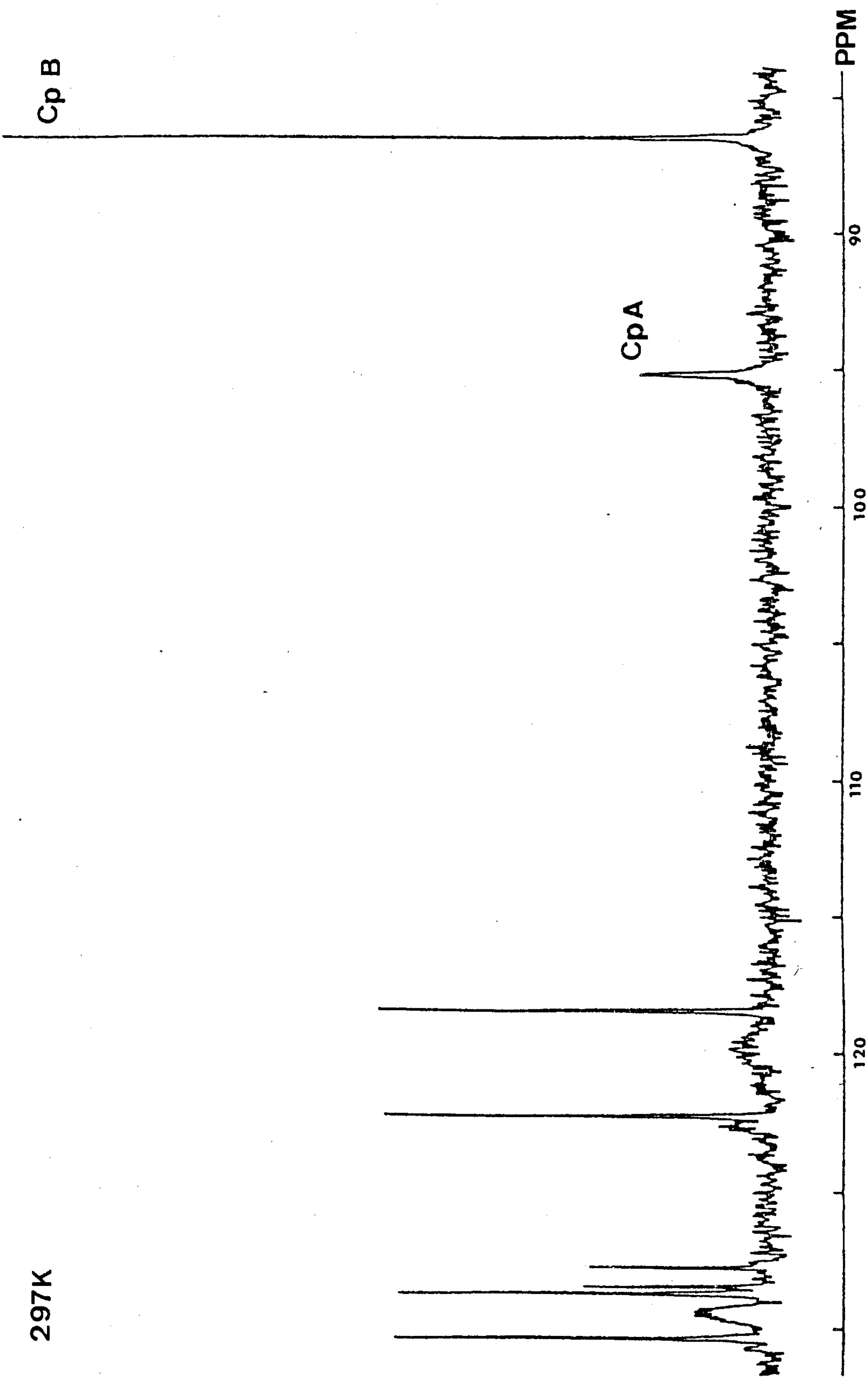
FIGURE

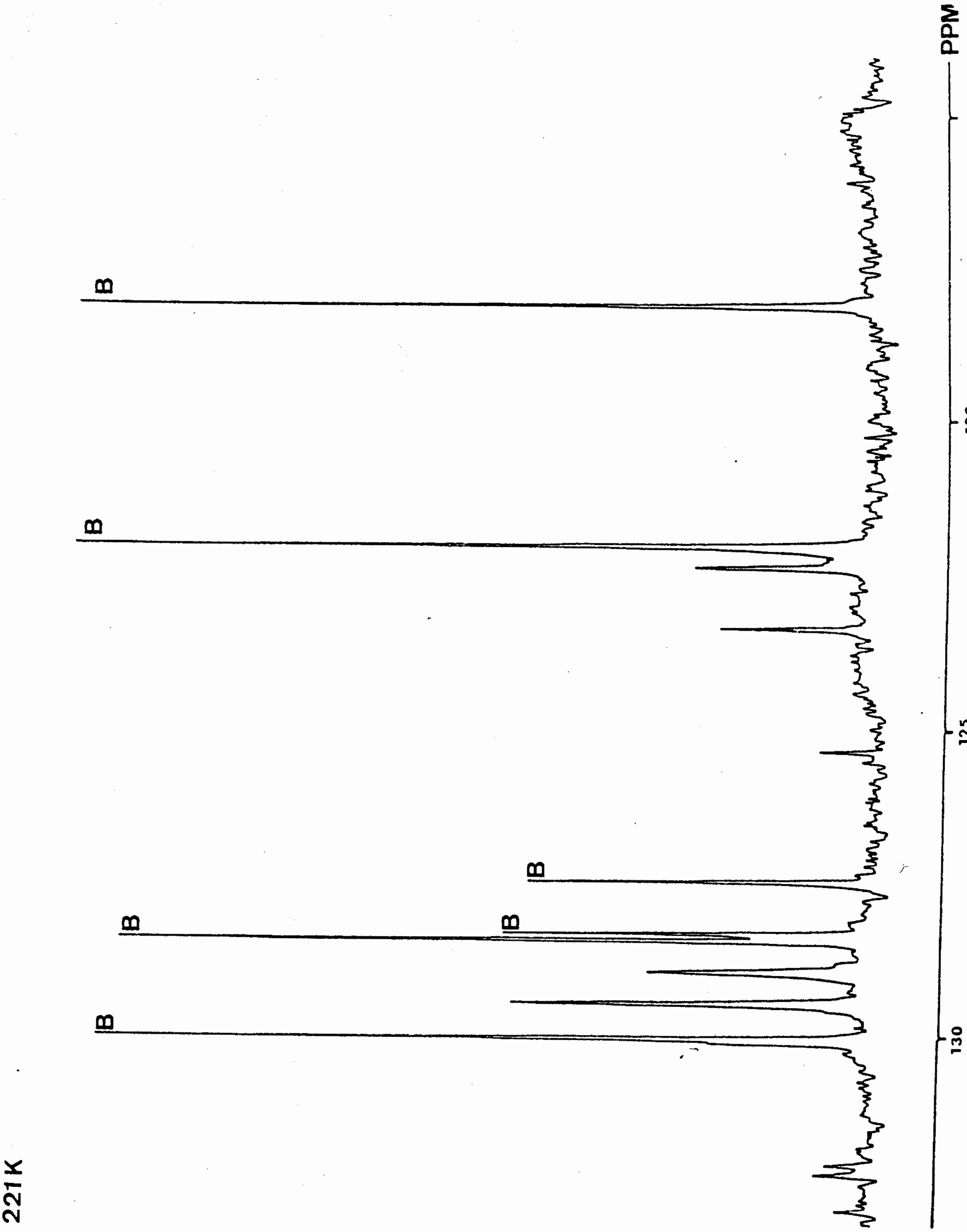
25. $^{13}C\{^1H\}$ NMR (acetone-d_6) of $CpRe(CO)_2(N_2Ph_2)$ at 297 K

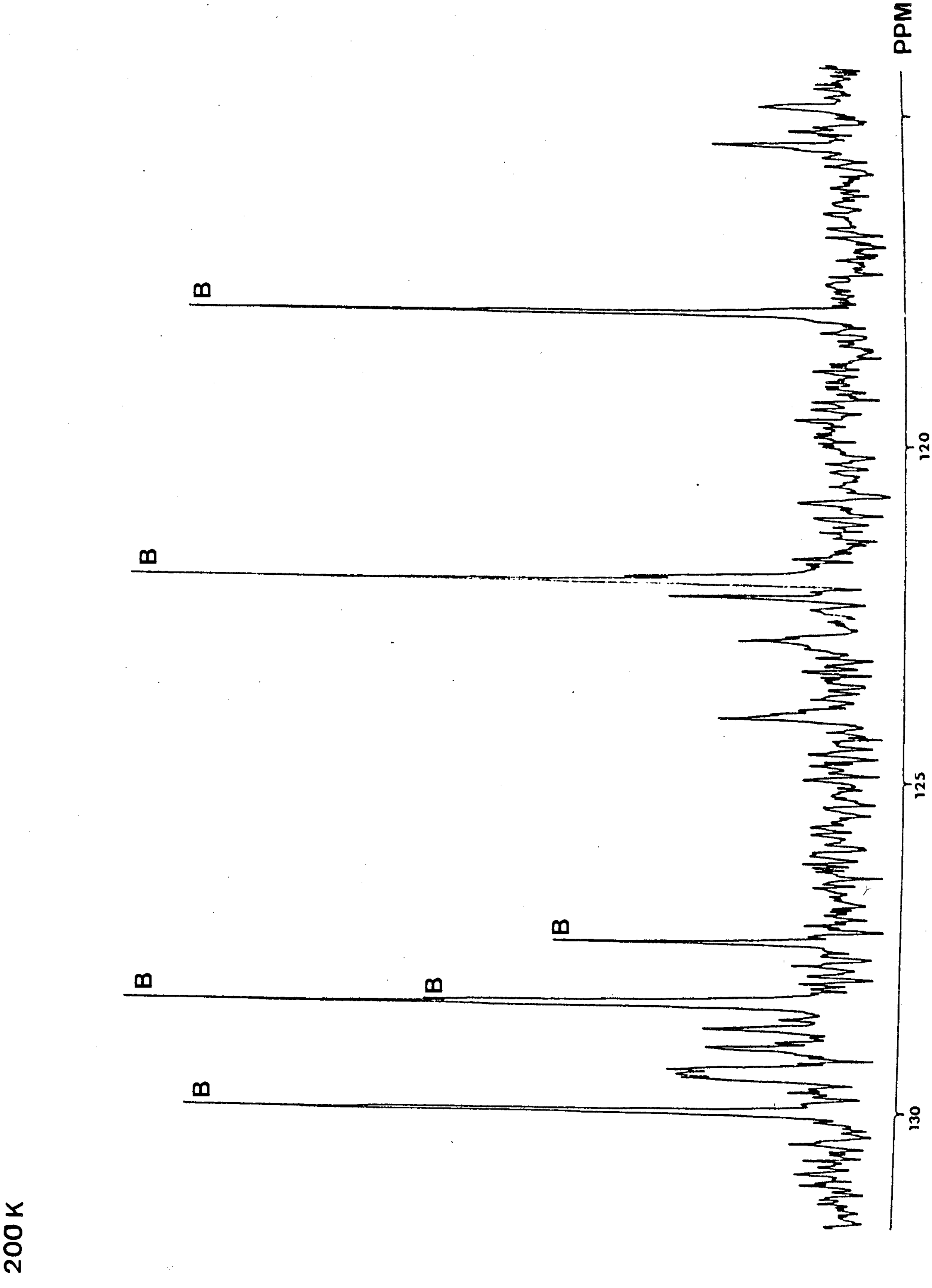
26. $^{13}C\{^1H\}$ NMR (acetone-d_6) of $CpRe(CO)_2(N_2Ph_2)$ at 221 K

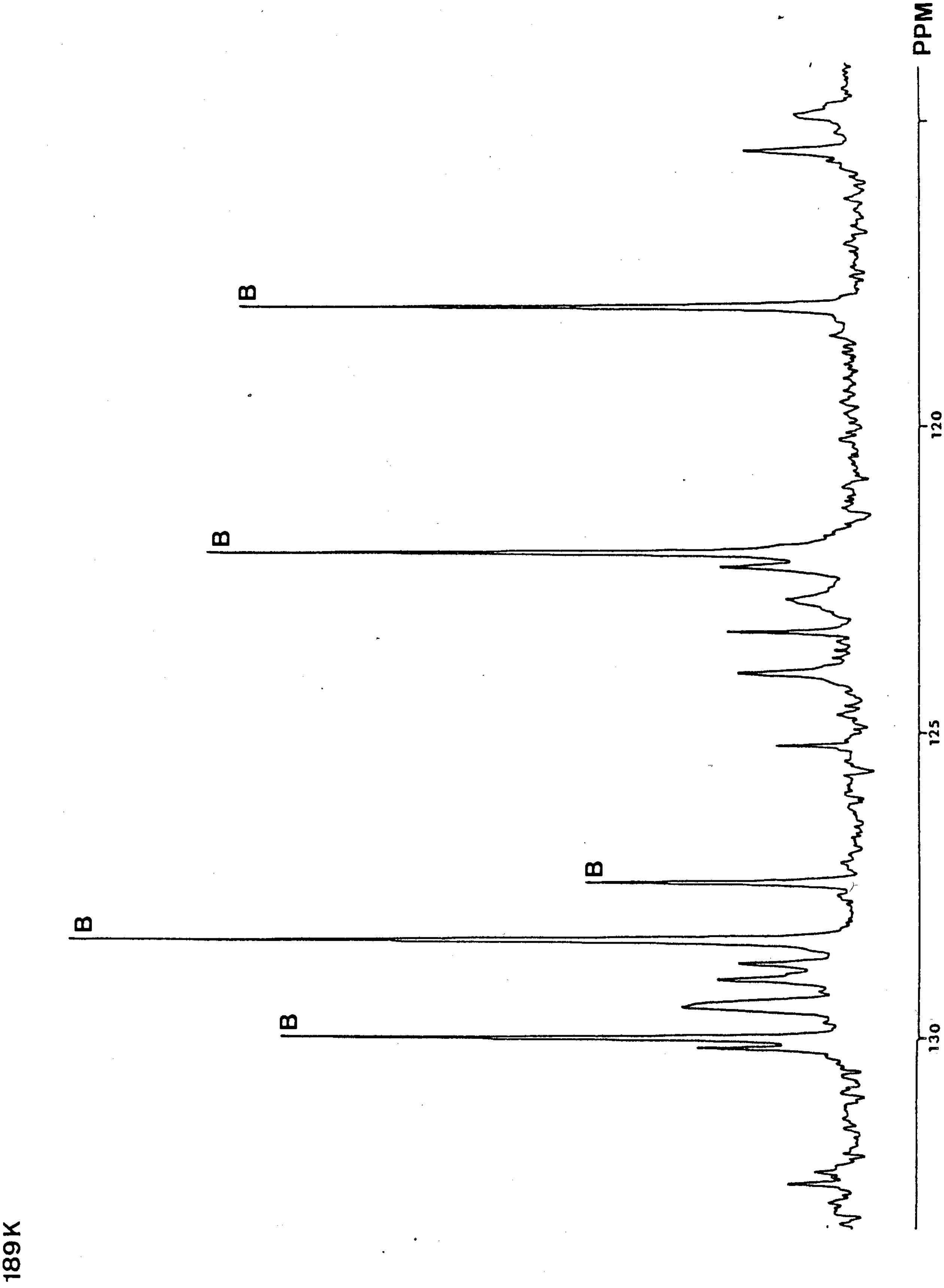
27. $^{13}C\{^1H)$ NMR (acetone-d_6) of $CpRe(CO)_2(N_2Ph_2)$ at 200 K

28. $^{13}C\{^1H\}$ NMR (acetone-d_6) of $CpRe(CO)_2(N_2Ph_2)$ at 189 K


Cp B
Cp A
297K
PPM
90
100
110
120


221 K
B
B
B
B
B
B
130
125
120
PPM


200 K
B
B
B
B
B
B
PPM
120
125
130

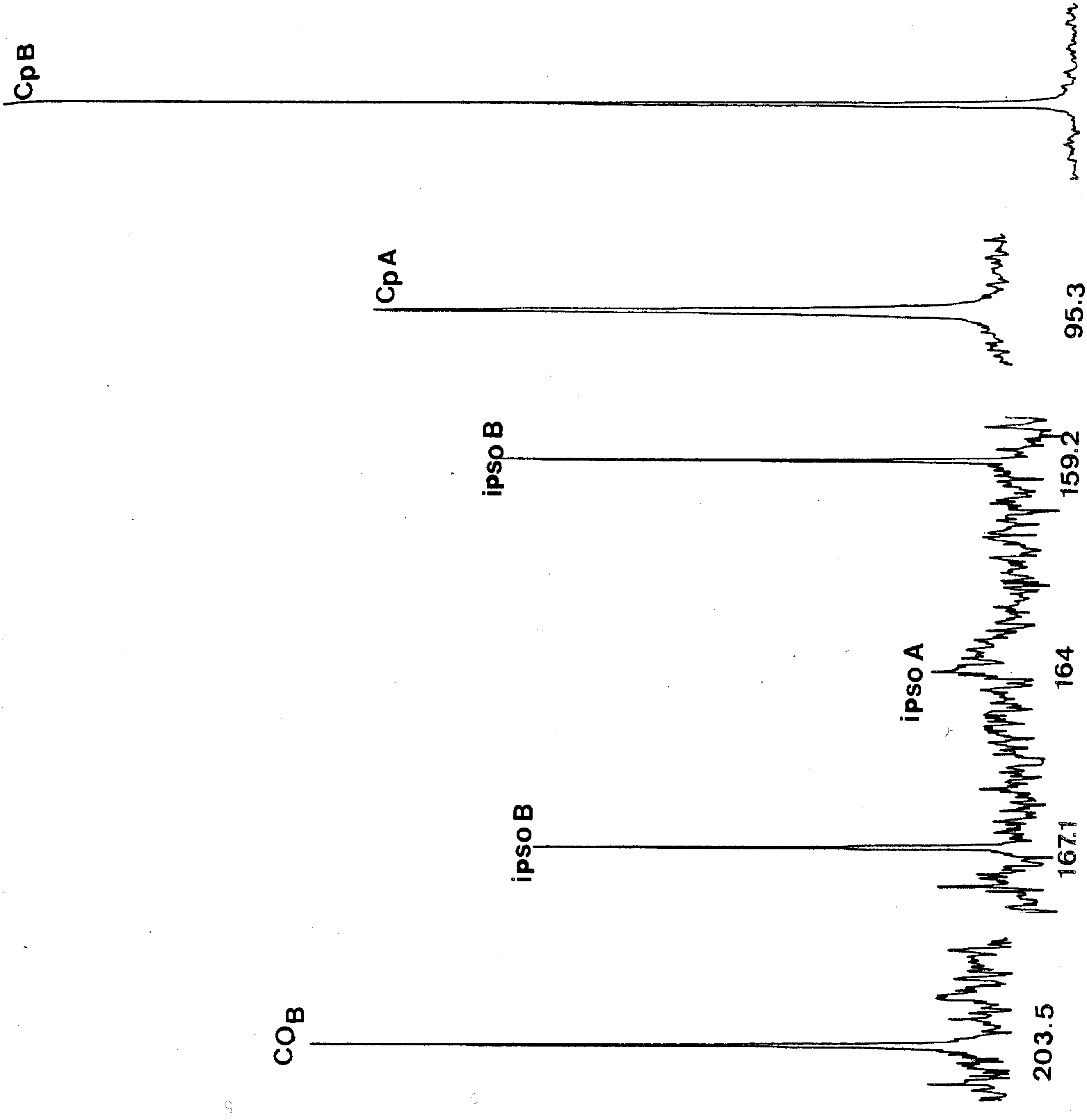

189K
PPM
B
B
B
B
B
120
125
130

region was observed which was due to the major isomer, substantiating that the major isomer is the σ-bonded azobenzene complex with free rotation about the Re-N bond. Resonances due to the carbonyl carbons of the minor isomers were not observed. The ipso carbon region shows two sharp peaks due to the major isomer (167.2 and 159.2 ppm) and a small broad resonance at approximately 163 ppm. This broad resonance could certainly be due to the ipso carbons of the minor isomers which have not been resolved. They would appear to be more similar in chemical shift than the ipso carbons of the major isomer. A π or η^2-bonded arrangement of the azobenzene ligand to the rhenium could account for this observation. It would not be expected that two ipso carbons of the azobenzene ligand, that is bound to the rhenium atom through both nitrogens so that the ipso carbons differ only in the spatial orientation of the phenyl ligand, would be as magnetically different as the two ipso carbons of an azobenzene ligand in which one nitrogen donates its lone pair to the Re and the second is unattached. The Cp resonance of the minor isomer(s) appears at 95.3 ppm. This peak is substantially broader at half height than the Cp resonance due to the major isomer (81.4 ppm), but separation into the two or more peaks which would be required for 2 or more different minor species, was not observed. However, the broadness of this resonance is certainly suggestive of an exchange process occurring within the η^2 bound form which does not involve the η^1-η^2 shift.

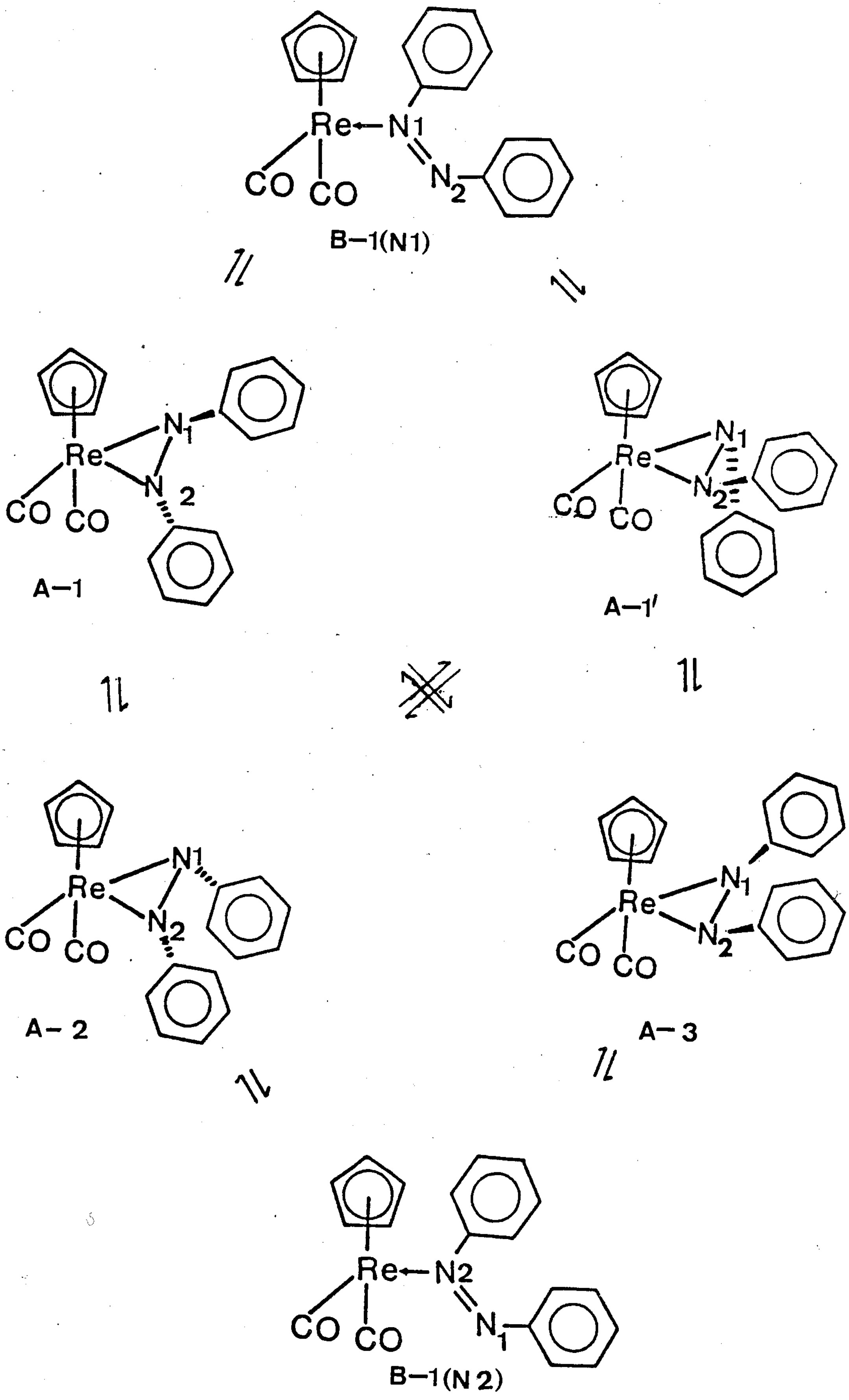
The interconversions between the η^1-η^2 species and within the η^2 bound isomers may be summarized in Scheme III-2. The major isomer in solution is isomer B (only the cis form B-1, is drawn) which can undergo a σ-π or η^1-η^2 shift to form A-1 or A-1'. These two enantiomers differ only in which phenyl ligand on N_1 or N_2 is 'close to' the Cp ring or the carbonyl ligands, and are indistinguishable to us in the IR or NMR spectrum. The nitrogen

FIGURE 29

$^{13}C\{^{1}H\}$ NMR (acetone-d_6) of the CO, ipso and Cp region of $CpRe(CO)_2(N_2Ph_2)$ (<u>13</u>) at 189 K


CO$_B$
203.5
ipso B
167.1
ipso A
164
ipso B
159.2
Cp A
95.3
Cp B

SCHEME III-2



labelled N_1 on isomer A-1 may then undergo a nitrogen inversion to yield A-2, where both phenyl ligands are cis and 'towards' the carbonyl ligand, or the nitrogen labelled N_2 may invert to form A-3 which has the phenyl groups cis and 'towards' the Cp ring. Similar arguments hold for A-1'. A π-σ or η^2-η^1 shift of the azobenzene ligand in A-2 or A-3 could reform B-1 with N_2 acting as the Lewis base.

This scheme accounts for the reversible η^1-η^2 shift between B-1 and A-1 (or A-1') and also the large number of aromatic resonances observed in the $^{13}C\{^1H\}$ spectrum of 13 at 189 K (Figure 27) for the minor isomers. The compounds A-1, A-2 and A-3 have 12 different phenyl carbon resonances (not including ipso carbons) and we observe at least 10 or 11 of them. However differing populations of the A-1, A-2 or A-3 isomers may account for the absence of, and varying intensities of the resonances.

Nitrogen inversion e.g., A-1 to A-2, is expected to have a lower barrier than an η^1-η^2 shift (B-1 to A-1) of the azobenzene ligand which involves substantial rehybridization of the orbitals. Thus it is not surprising that the low energy interconversions in this system appear to involve inversion at the nitrogen atoms. However, it is noted that diaziridines[138,139], which have a CNN ring versus our ReNN ring, can have a high barrier to inversion (111-115 kJ mol^{-1}) depending upon the substituents on the nitrogens and the carbon atom. In scheme III-2, only interconversion between stereoisomers by a consecutive nitrogen inversion is assumed and two synchronous inversions of the nitrogens i.e., A-1 to A-1' or A-2 to A-3, is not considered. This assumption is supported by calculations by Mannschreck[138] which indicate that the monoplanar transition state for inversion at one nitrogen atom is much lower in energy than the bi-planar transition state for two synchronous inversions. The latter transition state will suffer from the high strain energy caused by the two planar sp^2 hybridized nitrogen atoms. While our system is very different from the diaziridine system used for the calculations, since we have introduced a transition metal atom, there is no evidence to support a synchronous inversion in this case and it is presumed not to be occurring.

Ackermann[134,135] had only observed coordination site exchange (an η^1-η^1 shift) between nitrogens of a cis diazene ligand and not with the corresponding trans diazenes, and did not observe an η^1-η^2 shift with either a cis or trans azo ligand. However, an η^2 transition state was postulated in the η^1-η^1 shift of the cis azo ligand. Since our η^1-η^2 shift had been noted with trans-azobenzene as the starting material, in order to test whether there would be a further, different isomer formed that would display different site exchange behaviour in our system, the reaction of cis-azobenzene and $CpRe(CO)_2(THF)$ was carried out. After chromatography, a brown compound with an identical IR and ^1H NMR spectrum to 13 was isolated. No other compounds were isolated. This supports the interconversions outlined in Scheme III-2. Both cis and trans azobenzene will yield the same mixture of products upon reacting with $CpRe(CO)_2(THF)$ because both the cis and trans azo ligands go through common intermediates when coordinated in the η^2 form.

3.4.2b $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (14) and $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15)

The η^2-bonded azo structure has been shown by Albini and Kisch[140] to be a better π-acceptor than the η^1-form. Any substituent on the phenyl ring of the azo ligand that increases the π acidity should increase the proportion of the η^2 isomer. Conversely, an electron donating substituent on the phenyl rings should disfavour the η^2 form and favour the η^1 structure in which the azo ligand functions as a Lewis base. To substantiate this conjecture the p,p'-methylazobenzene and p,p'-fluoroazobenzene were synthesized and reacted with $CpRe(CO)_2(THF)$ to form $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (14) and $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15), respectively. The IR's of 14 and 15 have been mentioned briefly previously (Section 3.4.2) and are shown in Figure 22. The use of a methyl group in the para position, compound 14, causes the higher wavenumber $\nu(CO)$ bands to almost disappear. With fluorine in the para position, 15, the proportion of the higher wavenumber $\nu(CO)$ bands increases, although not to the same extent as the decrease noted for 14. These changes in the intensities of the $\nu(CO)$ bands for 14 and 15 are consistent with the higher wavenumber $\nu(CO)$ absorptions being due to the η^2 or π-bound isomer and the lower wavenumber $\nu(CO)$ bands to the η^1 or σ-bound azo ligand, and so further support the assignments already adopted.

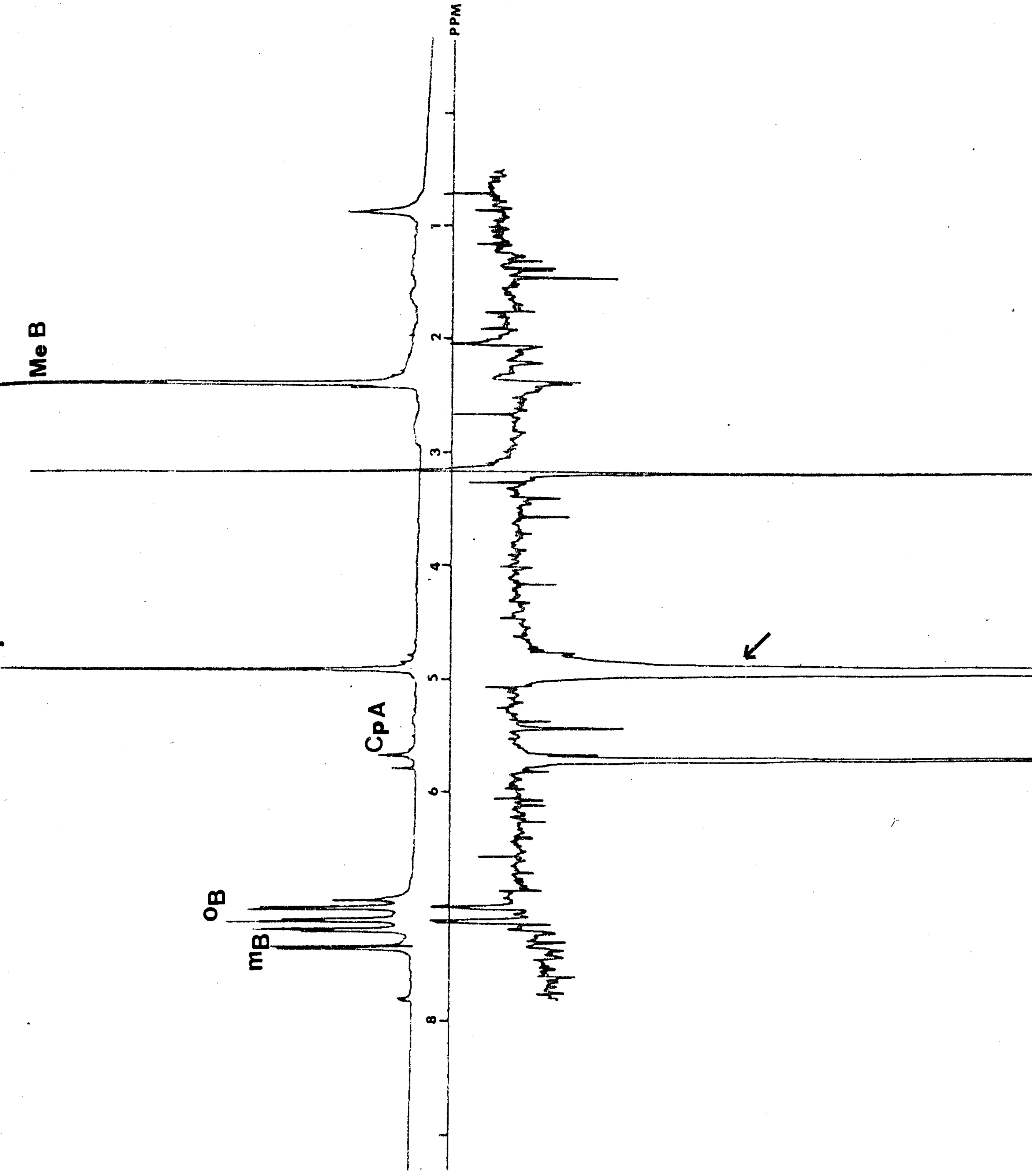
The ^1H and $^{13}C\{^1H\}$ NMR of $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2$ (14) showed resonances due to two non-identical aromatic phenyl rings and two methyl groups. The Cp resonance due to the minor isomer A-1 in $CDCl_3$ was broad and only slightly above the baseline. For $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15) the Cp resonance due to the minor isomer was broad but of reasonable intensity; however, other peaks in the aromatic region were not observed. Unfortunately, 15 was not stable enough in solution for long term NMR studies to be carried out. However, a relatively quick coalescence experiment was performed on 15, with a consequent large error in the temperature (see Experimental) and yielded a $\Delta G^\ddagger$ of 63.6 ± 1.2 kJ mol^{-1}. This is slightly smaller than the $\Delta G^\ddagger$ value of 67.4 ± 0.4 kJ mol^{-1} obtained for $CpRe(CO)_2(N_2Ph_2)$. This suggests that the η^1-η^2 shift has a lower energy barrier when the η^2 form has electron-withdrawing groups on the phenyl ligands which help to favour the η^2 structure (isomer A).

An NOE experiment was performed on $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (14). The Cp resonance of the major isomer (CpB) was irradiated (250 K) and the results of this experiment are shown in Figure 29. The ortho protons of both phenyl rings of the azotoluene ligand were found to be enhanced but not the meta protons. No enhancement of the para methyl hydrogens was found. Saturation transfer from the Cp resonance of the major isomer CpB, to the Cp resonance of the minor isomer CpA was observed, similar to the results found for $CpRe(CO)_2(N_2Ph_2)$ (13), establishing the interconversion process between isomer A and isomer B.

FIGURE 30

NOE Experiment on

$CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (<u>14</u>)


PPM
1
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Me B
Cp A
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mB

Summary

The synthesis of $CpRe(CO)_2L$ complexes, where L is a two electron donor such as C_2Ph_2, MeC_2Ph, N_2Ph_2, $N_2(C_6H_4Me\text{-}p)_2$ and $N_2(C_6H_4F\text{-}p)_2$ was accomplished. The X-ray crystal structures of $CpRe(CO)_2(C_2Ph_2)$ (11) and $CpRe(CO)_2(N_2Ph_2)$ (13) reveal both the diphenylacetylene and the azobenzene ligand to be bound to the rhenium in an η^2 fashion and the overall geometry of the complex approximates that of a 'four legged piano stool'. Fluxionality of the alkyne ligands was restricted to rotation about the Re-alkyne vector and for $CpRe(CO)(MeC_2Ph)$ (12), this rotation could be stopped at -60°C.

For the azobenzene complex $CpRe(CO)_2(N_2Ph_2)$ (13), a different type of fluxionality was present. The azobenzene ligand exhibited an η^1-η^2 shift with the η^1 (or σ) form being predominant in solution and the η^2 (or π) structure present as the minor isomer. The ^1H, $^{13}C\{^1H\}$ NMR and IR data support the η^1 isomer being the major one in solution and saturation experiments show a reversible equilibrium occurring between the major and minor isomer. At room temperature the $^{13}C\{^1H\}$ NMR spectrum had only very broad peaks in the phenyl carbon region attributable to the minor isomer. Cooling the solution to 189 K resulted in at least 11 distinct resonances being noted. This temperature dependence and larger number of minor isomers is possible because the nitrogens in the η^2 isomer can undergo nitrogen inversion. This is a low energy process causing the phenyl carbons to coalesce at room temperature but at lower temperatures the inversion is slow enough to observe at least three different minor isomers.

Coalescence of the Cp resonances of the major and minor isomers of $CpRe(CO)_2(N_2Ph_2)$ (13) gave a value of 67.4 ± 0.4 kJ mol^{-1} for the η^1-η^2 shift of the azobenzene ligand. This is the first observed η^1-η^2 shift of

an azobenzene ligand and only the second example of this type of shift for any ligand[136].

Ackermann[134] has observed coordination site exchange or an η^1-η^1 shift for the azo ligand in $M(CO)_5$(c-DIPD) (M = Cr, Mo, W) and $W(CO)_5$(c-DMD). Although an η^2 or π transition state was proposed as the intermediate in the η^1-η^1 shift, no experimental evidence was forwarded. Coordination site exchange was not found for trans azo ligands, specifically $M(CO)_5$(t-DIPD) (M = Cr, Mo, W). However, we observe an η^1-η^2 shift of the azobenzene ligand in $CpRe(CO)_2(N_2Ph_2)$ and in the η^2 isomer, as confirmed by X-ray crystallography, the azobenzene ligand has a trans geometry with the phenyl rings in nonequivalent environments.

$CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15) has a fluorine in the para position and the ν(CO) bands in the solution IR which are attributable to the η^2 isomer increased in intensity. When a methyl group was substituted in the para position, as in $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$, the η^1 isomer was found to be heavily favoured. These observations are consistent with an η^1-η^2 shift, since the use of an electron withdrawing group should favour the η^2 form relative to the η^1 isomer because in the η^2 geometry the azo ligand is a better π acid than in the η^1 form[140]. Conversely, an electron donating group should favour the η^1 isomer because in this geometry the azo ligand is acting as a Lewis base donating the lone pair of electrons of a nitrogen to the rhenium.

3.6 EXPERIMENTAL SECTION

3.6.1 Synthetic

^1H and ^{13}C NMR spectra were recorded on a Brüker WM-400 spectrometer at 400 MHz and 100 MHz, respectively. Infrared spectra were measured on a Perkin-Elmer 983 spectrophotometer, and Raman spectra using a Cary Model 81 with a Spectra-Physics Model 125 HeNe laser. Solvents were dried and distilled under nitrogen and all operations were conducted under nitrogen. $CpRe(CO)_2(THF)$ was synthesized and isolated as previously described[138]. Azobenzene was purchased from Eastman Kodak, azotoluene and p,p'-di fluoroazobenzene were made by the reduction of nitrotoluene and p-fluoronitrotoluene respectively by the method of Alper[139]. Cis-azobenzene was made by photolysis of the trans isomer and the two compounds separated by column chromatography.

Reaction of $CpNiCo(CO)_3(C_2Ph_2)$ with $CpRe(CO)_2(THF)$

$CpRe(CO)_2(THF)$ (60 mg, 0.158 mmol) and $CpNiCo(CO)_3(C_2Ph_2)$ (70 mg, 0.157 mmol) were added to hexane (25 mL) and stirred at room temperature for 2 hours. An infrared spectrum of the solution revealed litte remaining $CpRe(CO)_2(THF)$ (ν(CO) 1925, 1857 cm^{-1}) but strong bands due to $CpRe(CO)_3$ (ν(CO) 2031, 1940 cm^{-1}). The solvent was removed under vacuum and the residue dissolved in hexane-CH_2Cl_2. Chromatography on a Florisil column with hexanes as eluant yielded $CpRe(CO)_3$, then $CpNiCo(CO)_3(C_2Ph_2)$ (ν(CO) 2060, 2008 cm^{-1}) as a green solution. With hexanes:CH_2Cl_2 (3:1) a light yellow band was next recovered, and identified as $CpRe(CO)_2(C_2Ph_2)$, with physical properties identical to those listed below.

Preparation of $CpRe(CO)_2(C_2Ph_2)$ (11)

Method 1

$CpRe(CO)_2(THF)$ (51 mg, 0.132 mmol) and a large excess of C_2Ph_2 (115 mg, 0.645 mmol) was added to 12 mL of hexanes and stirred vigorously for 4 hours. Chromatography on Florisil with hexane as an eluant gave yellow $CpRe(CO)_2(C_2Ph_2)$ (19 mg, 30% yield). IR (hexane): ν(CO): 1980 s, 1904 s cm^{-1}. Raman (solid): 1848 cm^{-1} (ν(C≡C)). ^1H NMR ($CDCl_3$): δ 7.85-7.32 m (Ph), 5.55 s (Cp). $^{13}C\{^1H\}$ NMR ($CDCl_3$: δ 204.5 (CO), 131.8, 131.6, 128.6, 127.6 (Ph), 88.2 (Cp), 81.7 (acetylenic carbons). Mass spectrum: m/e (based on Re^{185} and Re^{187}) 484, 486 (M^+), 456, 458 ($M-CO^+$), 428, 430 ($M-2CO^+$). Anal. calcd.: C, 51.95; H, 3.11. Found: C, 52.21; H, 3.26%.

Method 2

$CpRe(CO)_3$ (130 mg, 0.336 mmol) and C_2Ph_2 (220 mg, 1.23 mmol) were added to 30 mL hexanes in a quartz Carius tube under vacuum and irradiated with a 200 W Hanovia high pressure mercury source. Chromatography on Florisil yielded $CpRe(CO)_3$ (colorless) and $CpRe(CO)_2(C_2Ph_2)$ (yellow) (18 mg, 11% yield).

Preparation of $CpRe(CO)_2(PhC_2Me)$ (12)

This compound was synthesized as in Method 1 (above) using $CpRe(CO)_2(THF)$ (100 mg, 0.259 mmol) and PhC_2Me (0.15 mL, ~1.2 mmol). $CpRe(CO)_2(PhC_2Me)$ was isolated, after chromatography, as an oily, yellow solid and identified by spectroscopy. IR (hexane): ν(CO): 1976 s, 1899 s cm^{-1}. ^1H NMR ($CDCl_3$): δ 204.9 (CO), 131.4, 128.3, 126.8 (Ph), 87.9 (Cp), 78.2 (CMe), 69.4 (CPh), 15.2 (Me). (in the ^1H-coupled spectrum: δ 78.2 q

(^2J=9 Hz), 69.4 s, 15.2 q (^1J=130 Hz). At -60°C $^{13}C\{^1H\}$ coupled spectrum: δ 206.5 s, 206.0 s (CO), 132.6-126.2 complex (Ph), 88.3 (^1J=180 Hz, Cp), 78.9 q (^2J=9 Hz, CMe), 69.3 s (CPh), 15.9 q (^1J=130 Hz, Me) Mass spectrum: m/e 422, 424 (M^+), 394, 396 ($M-CO^+$), 366, 368 ($M-2CO^+$).

Oxidative Stability of $CpRe(CO)_2(C_2Ph_2)$ (11)

The color of solid samples of $CpRe(CO)_2(C_2Ph_2)$ (yellow) remained unchanged after standing in air for one week and they could then be dissolved to give essentially clear IR and NMR spectra. A solution of $CpRe(CO)(C_2Ph_2)$ in hexanes was left open to the air and periodically monitored by IR. No decomposition was noted in 12 hours.

Thermal Stability of $CpRe(CO)_2(C_2Ph_2)$

A solution of $CpRe(CO)_2(C_2Ph_2)$ in *iso*-octane was refluxed (99°C) for 8 hours and no decomposition or loss of CO was noted by IR. A solution of $CpRe(CO)_2(C_2Ph_2)$ in THF was refluxed (66°C) for 1 hour, after which no starting material remained and no carbonyl containing products were apparent from the IR spectrum.

Reaction of $CpRe(CO)_2(C_2Ph_2)$ with Carbon Monoxide

$CpRe(CO)_2(C_2Ph_2)$ (15 mg, 0.039 mmol) was dissolved in 10 mL hexanes in a Paar bomb and pressurized with CO (1200 psi, 82 atm). After 1 hour, the pressure was released and an IR spectrum of the yellow solution revealed only CO bands due to 11 and none for $CpRe(CO)_3$ (2031, 1940 cm^{-1}).

Reaction of $CpRe(CO)_2(C_2Ph_2)$ with PPh_3

To a solution of $CpRe(CO)_2(C_2Ph_2)$ (15 mg, 0.039 mmol) in 6 mL hexanes, octane or acetonitrile, excess PPh_3 (20 mg, 0.076 mmol) was added. As monitored by IR, no reaction was observed after 2 hours. Me_3NO (6 mg, 0.080 mmol) was then added to the acetonitrile solution but no reaction was noted after a further 2 hours. The CH_3CN solution was refluxed for 5 hours but no loss of CO ligand was observed.

Reaction of $CpRe(CO)_2(C_2Ph_2)$ with I_2

$CpRe(CO)_2(C_2Ph_2)$ (10 mg, 0.026 mmol) was dissolved in 5 mL hexanes and I_2 (10 mg, 0.039 mmol) in 5 mL CH_2Cl_2 was added slowly to the solution. After 2 hours only bands for trans-$CpRe(CO)_2I_2$ (ν(CO) 2048 m, 1987 s cm^{-1}) were observed in the IR.

Reaction of $CpRe(CO)_2(C_2Ph_2)$ with Azobenzene

$CpRe(CO)_2(C_2Ph_2)$ (75 mg, 0.195 mmol) and excess N_2Ph_2 (90 mg, 0.494 mmol) were added to 10-15 mL hexanes and vigorously stirred at room temperature. After 2 hours, monitoring of the IR revealed only 11 present and no evidence for $CpRe(CO)_2(N_2Ph_2)$ (ν(CO) 2007 m, 1948 s, 1939 m, 1890 s cm^{-1}) were noted. The solution was warmed to 55°C for three hours but still the IR showed that $CpRe(CO)_2(N_2Ph_2)$ was not formed.

Reaction of $CpRe(CO)_2(C_2Ph_2)$ with H_2

To a solution of $CpRe(CO)_2(C_2Ph_2)$ (50 mg, 0.130 mmol) in 8 mL hexanes, H_2 was bubbled through at a constant rate. No new ν(CO) bands in the infrared spectrum were observed and the experiment was discontinued after 1 hour.

Reaction of $CpRe(CO)_3$ with C_2Ph_2

$CpRe(CO)_3$ (50 mg, 0.149 mmol) and C_2Ph_2 (31 mg, 0.174 mmol) were added to 15 mL hexanes-CH_2Cl_2 (13:2) and stirred overnight at room temperature. Inspection by IR revealed only bands due to $CpRe(CO)_3$ (ν(CO) 2031, 1940 cm^{-1}) and none for 11. Me_3NO (8 mg, 0.107 mmol) was added to the solution and stirred for a further two hours. As there were no new ν(CO) bands the solution was heated to 55°C for an additional two hours at which point the reaction was discontinued as no additional ν(CO) bands were apparent.

Preparation of $CpRe(CO)_2(N_2Ph_2)$ (13)

In a typical reaction, excess azobenzene was added to a hexane solution of $CpRe(CO)_2(N_2Ph_2)$ (100 mg, 0.26 mmol) and stirred vigorously for 6 hours. Chromatography on Florisil with hexanes yielded excess azobenzene and with a 1:1 hexane-CH_2Cl_2 as eluant a brown band identified as $CpRe(CO)_2(N_2Ph_2)$ was isolated in 25% yield. IR (hexane): 2006 m, 1947 s, 1939 m, 1890 s cm^{-1} ν(CO), IR (KBr): 2001 s, sl.br., 1928 s, 1900 s cm^{-1}, ν(CO). ^1H NMR (Acetone-d_6): δ 7.59, 7.57, 7.55 (Ph, meta), 7.45, 7.44, 7.42, 7.40 (Ph, meta and para), 7.34, 7.32, 7.30 (Ph, para), 7.25, 7.23 (Ph, ortho), 7.14, 7.12 (Ph, ortho), 5.72 s (Cp, minor isomer), 4.95 s (Cp, major isomer). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 201.1 (CO), 166.8, 158.6 (Ph, ipso) 129.4, 127.8 (Ph, ortho), 127.7, 127.0 (Ph, meta), 121.3, 117.7 (Ph, para), 93.4 (Cp, minor isomer), 85.8 (Cp, major isomer). Mass spectrum: m/e (based on Re^{185} and Re^{187}) 489, 491 (M^+), 461, 463 ($M-CO^+$), 433, 435 ($M-2CO^+$). Anal. calcd.: C, 46.62, H, 3.09, N, 5.72; found: C, 46.81, H, 3.11 N, 5.80%.

Preparation of $CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ (14)

$CpRe(CO)_2(N_2(C_6H_4Me\text{-}p)_2)$ was prepared in an identical fashion to 13 in

20% yield using azotoluene rather than azobenzene. IR (hexane): 2004 w, 1945 s, 1887 s cm^{-1}. IR (KBr): 1997 w, 1932 s, 1868 s cm^{-1} $\nu(CO)$. ^1H NMR (Acetone-d_6): δ 7.30, 7.28 (Ph, meta), 7.22, 7.20 (Ph, meta), 7.14, 7.12 (Ph, ortho), 7.04, 7.02 (Ph, ortho), 5.68 s (Cp, minor isomer), 4.92 s (Cp, major isomer), 2.40, 2.38 (Me). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 202.4 (CO), 156.7, 156.1 (Ph, ipso), 141.1, 138.9 (Ph, para), 129.9, 129.7, 128.2, 122.7, 121.2, 117.8 (Ph), 85.6 (Cp), 29.7 (CH_3), 21.4 (CH_3). Mass spectrum: m/e, 516, 518 (M^+), 488, 490 ($M-CO^+$), 460, 462 ($M-2CO^+$). Anal. calcd.: C, 48.73, H, 3.70, N, 5.41; found: C, 48.84, H, 3.80, N, 5.48%.

Preparation of $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (15)

$CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ was prepared in an identical fashion to 13 but using p,p'-difluoroazobenzene rather than azobenzene. IR (hexanes): 2008 s, 1950 s, 1941 s, 1893 s cm^{-1}. ^1H NMR ($CDCl_3$): δ 6.8-7.4 (Ph), 5.83 s (Cp, minor isomer), 5.05 s (Cp, major isomer). $^{13}C\{^1H\}$ ($CDCl_3$): δ 202.2 (CO), 162.4, 160.0 (Ph, para, $^1J_{C-F}$=246 Hz), 154.07 (Ph, ipso), 131.1, 128.9 (Ph, para, $^1J_{C-F}$=223 Hz), 123.12, 123.04 (Ph, ortho, $^3J_{C-F}$=8.4 Hz), 119.53, 119.44 (Ph, ortho, $^3J_{C-F}$=8.5 Hz), 116.54, 116.32 (Ph, meta, $^2J_{C-F}$=22.6 Hz), 114.64, 114.41 (Ph, meta, $^2J_{C-F}$=22.7 Hz), 93.4 s (Cp, minor isomer), 86.8 s (Cp, major isomer). Mass spectrum: m/e, 524,526 (M^+), 496, 498 ($M-CO^+$), 468, 470 ($M-2CO^+$).

Reaction of $CpRe(CO)_2(THF)$ with cis-azobenzene

Excess cis-azobenzene was added to a hexane (8 mL) solution of $CpRe(CO)_2(THF)$ (20 mg, 0.041 mmol) and stirred vigorously overnight. Chromatography on Florisil with hexane-CH_2Cl_2 (1:2) yielded a dark brown compound with an identical IR and NMR to that found for $CpRe(CO)_2(N_2Ph_2)$

(ν(CO) (hexane) 2006 m, 1947 s, 1939 m, 1890 s cm^{-1}).

Reaction of $CpRe(CO)_2(N_2Ph_2)$ with C_2Ph_2

C_2Ph_2 (16 mg, 0.089 mmol) and $CpRe(CO)_2(N_2Ph_2)$ (20 mg, 0.041 mmol) were added to 6-7 mL of hexanes and 0.5 mL of CH_2Cl_2. The reaction was stirred at room temperature for 2 hours and the IR spectrum showed only 13 and no ν(CO) bands due to $CpRe(CO)_2(C_2Ph_2)$ (ν(CO) 1980, 1904 cm^{-1}). The hexanes-CH_2Cl_2 was removed and 6 mL of benzene was added. After stirring at ambient temperature for 1 hour, the solution was warmed to 40°C where after 1 hour no formation of $CpRe(CO)_2(C_2Ph_2)$ was detected, although some decomposition was noted.

Reaction of $CpRe(CO)_2(N_2Ph_2)$ with I_2

To a solution of 13 (920 mg, 0.041 mmol) in 5-6 mL hexanes an I_2/hexanes solution was added dropwise until the IR revealed the ν(CO) bands due to 13 had disappeared. The hexanes were removed and the residue dissolved in CH_2Cl_2. An IR spectrum revealed two carbonyl absorptions (2041 s, 1977 m cm^{-1}) corresponding to cis-$CpRe(CO)_2I_2$. When the identical experiment was conducted using benzene as solvent, only trans-$CpRe(CO)_2I_2$ was formed (ν(CO) (CH_2Cl_2): 2047 m, 1988 s cm^{-1}). A control experiment revealed that pure cis-$CpRe(CO)_2I_2$ was fully converted into trans-$CpRe(CO)_2I_2$ in benzene in 10 minutes at ambient temperature.

Reaction of $CpRe(CO)_2(N_2Ph_2)$ with Carbon Monoxide

A hexane-benzene solution of $CpRe(CO)_2(N_2Ph_2)$ (15 mg, 0.031 mmol) was purged with CO gas. As monitored by IR, no formation of $CpRe(CO)_3$ (ν(CO); 2031, 1940 cm^{-1}) was noted, nor that of any other carbonyl containing species.

Reaction of $CpRe(CO)_3$ with N_2Ph_2

$CpRe(CO)_3$ (25 mg, 0.075 mmol) and azobenzene (28 mg, 0.15 mmol) were added to 12 mL hexanes-CH_2Cl_2 (10:2) and stirred overnight. Inspection by IR revealed bands due to $CpRe(CO)_3$ only and none for $CpRe(CO)_2(N_2Ph_2)$. Addition of Me_3NO or iodosobenzene to the solution and further stirring for 2 hours did not yield any 13 as monitored by IR and the reaction was discontinued.

Photolysis of $CpRe(CO)_3$ with N_2Ph_2

Photolysis of $CpRe(CO)_3$ (20 mg, 0.060 mmol) and azobenzene (27 mg, 0.14 mmol) in 15 mL hexanes using pyrex-filtered UV radiation yielded only small amounts of 13 as monitored by IR. The reaction was discontinued after 2 hours because of extensive darkening of the solution.

Calculation of $\Delta G^\ddagger$ for $CpRe(CO)_2(N_2Ph_2)$ (toluene-d_8)

The variable temperature behaviour of the Cp resonances for isomers A and B was observed, with coalescence at 332 ± 1 K.

$$T_c = 332 \pm 1\ \mathrm{K}$$

$$\Delta\nu_\infty = 77.1 \pm 0.5\ \mathrm{Hz}$$

$$K_c = \pi/\sqrt{2}\ \Delta\nu_\infty = 171.3 \pm 1.1\ \mathrm{sec}^{-1}$$

and since,

$$\Delta G^\ddagger = -RT_c \ln(K_c h/KT_c)$$

where,

$$K = \text{Boltzmann constant} = 1.3807 \times 10^{-16}\ \mathrm{ergs\ K}^{-1}$$

$$h = \text{Plank's constant} = 6.63 \times 10^{-27} \text{ erg sec}$$

$$R = \text{gas constant} = 1.987 \text{ cal K}^{-1} \text{ mol}^{-1}$$

thus $$\Delta G^{\ddagger} = 67.4 \pm 0.4 \text{ kJ mol}^{-1}$$

Calculation of $\Delta G^{\ddagger}$ for $CpRe(CO)_2(N_2(C_6H_4F\text{-}p)_2)$ (toluene-d_8)

Coalsecence of the Cp resonances of the A and B isomers was again monitored.

$$T_c = 315 \pm 3 \text{ K}$$

$$\Delta\nu_{\infty} = 82.6 \pm 0.5 \text{ Hz}$$

$$K_c = \pi/\surd 2 \ \pi\nu_{\infty} = 183.3 \pm 1.1 \text{ sec}^{-1}$$

and thus,

$$\Delta G^{\ddagger} = 63.6 \pm 1.2 \text{ kJ mol}^{-1}$$

3.6.2 X-ray Structures

X-ray Structure of $CpRe(CO)_2(C_2Ph_2)$ (11)

A pale yellow needle-like crystal, 0.06 x 0.11 x 0.30 mm, obtained from a hexanes solution, was chosen for the diffraction studies. Preliminary oscillation photos (Copper radiation) indicated the crystal to be of good quality. Data were collected on an Enraf-Nonius diffractometer (Mo radiation) with a scan speed of 0.8-4.0° min^{-1} for $2.5° \leqq 2\theta \leqq 45°$ and a symmetrical scan width of 0.9° plus dispersion correction. Two standards were measured for intensity control and two standards were measured for orientation control every 150 reflections. Neither pair showed any significant deviation. Absorption corrections were applied via an emperical correction (ψ scans, transmission factors 0.447-0.993). 3008 reflections out of a total of 4545 had I ≧ 3.0σ(I) and were regarded as observed.

The coordinates of the Re atoms were derived from a three dimensional Patterson map and non-hydrogen atoms from successive difference Fourier syntheses following least squares refinement. The hydrogen atom positions were calculated geometrically with d(C-H) of 0.95Å and assigned isotropic thermal parameters equal to their parent carbon atoms. All non-hydrogen atoms were refined anisotropically. The largest peaks in a final difference map, 0.56(12)eÅ^{-3}, were located near the Re atoms. Computer programs are from reference 65. A summary of the crystal data is in Table 40, bond distances and angles are in Table 41, atom coordinates in Table 43, anisotropic thermal parameters in Table 45, calculated hydrogen atom coordinates in Table 47 and calculated meanplanes and dihedral angles in Table 49. A labelled SNOOPI diagram is shown in Figure 18 and a view of the two independent molecules in the unit cell is shown in Figure 20.

X-ray Structure of $CpRe(CO)_2(N_2Ph_2)$ (13)

A small orange crystal, 0.08 x 0.11 x 0.13 mm, obtained from hexanes/$CHCl_3$, was chosen for the diffraction studies. Data were collected on an Enraf Nonius diffractometer (Mo radiation) with a scan speed of 0.5-4.0° min^{-1} for 3° ≦ 2θ ≦ 45° and a symmetrical scan width of 0.6° plus dispersion correction. Two standards were measured for intensity control after every 3600 seconds of X-ray beam time and showed only random variations. Absorption corrections were applied via an emperical method (ψ scans, transmission factors 0.492-0.992). 991 reflections out of a total of 1180 had I ≧ 3.0σ(I) and were regarded as observed.

The Re atom coordinates were derived from a three dimensional

Patterson map and non-hydrogen atoms from successive difference Fourier synthesis following least squares refinement. The hydrogen atom positions were calculated geometrically with $\underline{d}$(C-H) of 0.98Å and assigned isotropic temperature factors of 0.075Å^2 and included as fixed contributions in further refinement. The Re atom, carbonyl groups and nitrogen atoms were refined anisotropically and all other non-hydrogen atoms isotropically. An extinction correction was included and refined to 0.473 x 10^{-6}. To ascertain that the correct enantiomorph had been chosen, the coordinates of all the atoms were inverted and the model refined until there was no further change. This gave values of $\underline{R}_{\underline{F}}$ = 0.0248 and $\underline{R}_{\underline{WF}}$ = 0.0279 and GOF = 1.60 and showed the original model to be correct. The coordinates were set back to their original values and a final difference map showed the highest peak to be 0.55(21)eÅ^{-3} and located near the cyclopentadienyl ring. Computer programs are from reference 65. A summary of the crystal data is in Table 40, bond distances and angles in Table 42, atom coordinates in Table 44, anisotropic thermal parameters in Table 46, calculated hydrogen atom coordinates in Table 48 and calculated meanplanes and dihedral angles in Table 50. An SNOOPI diagram showing the labelling scheme is presented in Figure 19.

Table 40. Summary of Crystal Data and Intensity Collection for $CpRe(CO)_2(C_2Ph_2$ (*11*) and $CpRe(CO)_2(N_2Ph_2)$ (*13*)

cmpd	$C_{21}H_{15}O_2Re$	$C_{19}H_{15}N_2O_2Re$
mol wt	485.55	489.52
a, Å	10.841(1)	8.315(1)
b, Å	13.961(2)	11.214(2)
c, Å	23.121(2)	9.511(2)
β, deg	91.360(8)	105.75(2)
cell vol, $Å^3$	3498	853.6
Z	8	2
ρ (calcd) g cm^{-3}	1.490	1.905
ρ (obsd) g cm^{-1}	1.53(1)	2.02(1)
space group	$P2_1n$	$P2_1$
cryst dimens, mm	0.06x0.11x0.30	0.08x0.11x0.13
t, °C	21	21
radiation	MoK_α	MoK_α
linear abs coeff, cm^{-1}	70.5	72.3
transmission factors	0.447-0.993(emperical)	0.492-0.992(emperical)
scan speed, deg/min	0.8-4.0 (ω-2θ scan)	0.5-4.0 (ω-2θ scan)
2θ limits, deg	2.5, 45.0	3.0, 45.0
data collected	4545	1180
unique data, $I \geq 3.0\sigma(I)$	3008	991
R_f, %	2.95	2.44
R_{wf}, %	3.40	2.71
goodness of fit	1.17	1.55
extinction	-	0.473×10^{-6}

Table 41. Bond Distances (Å) and Angles (deg) for CpRe(CO)$_2$(C$_2$Ph$_2$) (11).

Metal Atom Coordination

Bond Lengths

Re(1)-C(11)	1.893(11)	Re(2)-C(21)	1.916(11)
Re(1)-C(12)	1.906(12)	Re(2)-C(22)	1.902(10)
Re(1)-C(13)	2.188(10)	Re(2)-C(23)	2.190(9)
Re(1)-C(14)	2.182(9)	Re(2)-C(24)	2.194(9)
Re(1)-Cp(center)	2.039(12)	Re(2)-Cp(center)	1.968(12)

Bond Angles

C(13)-Re(1)-C(14)	33.5(4)	C(23)-Re(2)-C(24)	32.7(3)
Re(1)-C(13)-C(14)	73.0(6)	Re(2)-C(23)-C(24)	73.8(6)
Re(1)-C(14)-C(13)	73.5(6)	Re(2)-C(24)-C(23)	73.5(6)
Re(1)-C(13)-C(111)	134.4(7)	Re(2)-C(23)-C(211)	134.5(7)
Re(1)-C(14)-C(15)	135.3(7)	Re(2)-C(24)-C(25)	136.4(7)
Re(1)-C(11)-O(11)	177.2(9)	Re(2)-C(21)-O(21)	177.2(9)
Re(1)-C(12)-O(12)	177.6(10)	Re(2)-C(22)-O(22)	178.4(9)

Acetylene Coordination

Bond Lengths

C(13)-C(14)	1.261(14)	C(23)-C(24)	1.232(13)
C(13)-C(111)	1.45(1)	C(23)-C(211)	1.44(1)
C(14)-C(15)	1.43(1)	C(24)-C(25)	1.46(1)

Bond Angles

C(14)-C(13)-C(111)	152.6(10)	C(24)-C(23)-C(211)	151.6(9)
C(13)-C(14)-C(15)	151.1(10)	C(23)-C(24)-C(25)	149.9(9)

Table 42. Bond Distances (Å) and Angles (deg) for $CpRe(CO)_2(N_2Ph_2)$ (13)

Bond Lengths

Re-N(1)	2.048(12)	N(1)-N(2)	1.415(17)
Re-N(2)	2.136(11)	N(1)-C(3)	1.406(20)
Re-C(1)	1.979(18)	N(2)-C(9)	1.424(18)
Re-C(2)	1.800(21)	C(1)-O(1)	1.150(23)
Re-Cp(center)	1.988(15)	C(2)-O(2)	1.189(26)

Bond Angles

Re-N(1)-N(2)	73.6(7)	N(2)-Re-C(1)	81.3(5)
Re-N(2)-N(1)	66.9(6)	N(1)-N(2)-C(9)	113.7(10)
Re-N(1)-C(3)	119.7(9)	N(2)-N(1)-C(3)	114.4(10)
Re-N(2)-C(9)	115.0(8)	Re-C(1)-O(1)	175.6(14)
N(1)-Re-N(2)	39.5(4)	Re-C(2)-O(2)	174.6(19)
N(1)-Re-C(2)	94.7(7)	C(1)-Re-C(2)	81.2(4)

Table 43. Atom Coordinates for $CpRe(CO)_2(C_2Ph_2)$ (11)

Atom	x	y	z
Molecule 1			
Re(1)	0.07979(4)	0.16622(3)	0.09831(2)
C(11)	0.1422(9)	0.0563(7)	0.0610(5)
C(12)	0.0553(10)	0.0788(8)	0.1603(5)
O(11)	0.1774(8)	-0.0137(6)	0.0395(4)
O(12)	0.0370(8)	0.0255(6)	0.1965(4)
C(13)	0.2242(9)	0.2255(7)	0.1555(5)
C(14)	0.2693(8)	0.2183(7)	0.1059(4)
C(15)	0.3679(8)	0.2329(7)	0.0660(4)
C(16)	0.4779(10)	0.2744(8)	0.0863(5)
C(17)	0.5750(9)	0.2861(18)	0.0495(6)
C(18)	0.5658(10)	0.2562(8)	-0.0069(6)
C(19)	0.4602(10)	0.2156(9)	-0.0280(5)
C(110)	0.3638(10)	0.2053(7)	0.0092(4)
C(111)	0.2309(10)	0.2519(7)	0.2163(5)
C(112)	0.1275(12)	0.2616(9)	0.2487(5)
C(113)	0.1369(15)	0.2938(9)	0.3050(5)
C(114)	0.2509(14)	0.3126(9)	0.3315(6)
C(115)	0.3541(14)	0.3003(9)	0.2977(6)
C(116)	0.3460(12)	0.2708(8)	0.2417(5)
C(117)	0.0379(10)	0.3047(9)	0.0482(6)
C(118)	-0.0177(10)	0.2293(9)	0.0183(5)
C(119)	-0.1063(9)	0.1897(7)	0.0533(5)
C(120)	-0.1075(10)	0.2397(8)	0.1042(5)
C(121)	-0.0206(12)	0.3105(8)	0.1030(6)
Molecule 2			
Re(2)	0.27088(4)	0.87942(3)	0.66720(2)
C(21)	0.3365(9)	0.7683(7)	0.6302(4)
C(22)	0.2314(9)	0.7893(8)	0.7256(4)
O(21)	0.3708(7)	0.7013(5)	0.6075(3)

Table 43 (Cont'd)

Atom	x	y	z
O(22)	0.2087(8)	0.7331(6)	0.7601(4)
C(23)	0.4086(8)	0.9317(7)	0.7305(4)
C(24)	0.4616(8)	0.9261(7)	0.6840(4)
C(25)	0.5705(8)	0.9435(7)	0.6495(4)
C(26)	0.6709(9)	0.9905(8)	0.6736(5)
C(27)	0.7723(10)	1.0108(9)	0.6407(6)
C(28)	0.7751(10)	0.9848(9)	0.5845(5)
C(29)	0.6765(10)	0.9361(8)	0.5593(5)
C(210)	0.5733(9)	0.9166(8)	0.5917(4)
C(211)	0.4077(9)	0.9529(7)	0.7916(4)
C(212)	0.2979(9)	0.9704(8)	0.8215(4)
C(213)	0.2971(11)	0.9903(8)	0.8801(4)
C(214)	0.4072(12)	0.9927(8)	0.9099(5)
C(215)	0.5170(11)	0.9792(8)	0.8825(5)
C(216)	0.5162(10)	0.9584(8)	0.8245(5)
C(217)	0.2413(10)	1.0081(8)	0.6069(5)
C(218)	0.1720(10)	0.9366(8)	0.5859(4)
C(219)	0.0846(9)	0.9130(8)	0.6249(5)
C(220)	0.0978(11)	0.9747(10)	0.6737(5)
C(221)	0.1989(11)	1.0358(8)	0.6610(5)

Table 44. Atom Coordinates (× 10^4) for $CpRe(CO)_2(N_2Ph_2)$ (13)

Atom	X	Y	Z	B_{iso}
Re	429.8(6)	2500	2932.8(5)	2.63(6)
N(1)	1616(1)	1306(1)	1927(1)	3.2(6)
N(2)	2834(1)	2157(1)	2615(1)	2.9(6)
C(1)	1826(2)	3245(1)	4740(2)	4.2(8)
O(1)	2698(2)	3613(1)	5805(1)	8.3(8)
C(2)	71(2)	1441(2)	4237(2)	5.4(10)
O(2)	-300(2)	740(1)	5032(2)	8.3(9)
C(3)	1936(2)	143(1)	2484(2)	3.0(3)
C(4)	614(2)	-637(2)	2225(2)	4.4(3)
C(5)	900(2)	-1826(2)	2705(2)	5.6(4)
C(6)	2445(2)	-2225(2)	3466(2)	5.7(4)
C(7)	3731(2)	-1404(2)	3773(2)	5.8(4)
C(8)	3508(2)	-254(2)	3269(2)	4.0(3)
C(9)	3310(2)	2941(1)	1622(1)	3.4(3)
C(10)	3287(1)	2590(1)	215(1)	3.8(2)
C(11)	3907(2)	3397(2)	-652(2)	4.2(3)
C(12)	4559(2)	4500(2)	-102(2)	5.0(4)
C(13)	4611(2)	4830(2)	1308(2)	5.1(4)
C(14)	3974(2)	4060(2)	2177(2)	4.7(4)
C(15)	8313(1)	2736(2)	779(1)	3.5(3)
C(16)	7635(2)	2791(2)	1917(1)	4.3(3)
C(17)	8249(2)	3826(2)	2716(2)	4.7(4)
C(18)	9466(2)	4375(2)	2013(2)	4.5(3)
C(19)	9374(2)	3658(1)	822(2)	3.8(3)

Table 45. Anisotropic Thermal Parameters (× 100) for $CpRe(CO)_2(C_2Ph_2)$ (11)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Molecule 1						
Re(1)	4.91(2)	5.09(3)	6.17(3)	0.01(2)	-0.027(2)	-0.33(2)
C(11)	5.3(7)	6.1(8)	9.7(9)	-0.8(6)	-1.0(6)	-0.1(7)
C(12)	6.6(8)	6.4(8)	11.0(10)	0.7(6)	1.4(7)	-1.7(7)
O(11)	10.6(7)	6.5(6)	14.0(8)	0.5(5)	1.3(6)	-4.2(5)
O(12)	11.5(7)	9.2(7)	11.2(7)	1.2(5)	3.2(6)	3.0(5)
C(13)	5.4(6)	4.4(7)	8.4(8)	0.5(5)	-0.7(6)	-1.3(6)
C(14)	4.6(6)	5.6(7)	7.5(8)	0.2(5)	-0.5(5)	-0.7(6)
C(15)	5.1(6)	4.8(7)	6.6(7)	0.1(5)	-0.6(5)	-0.7(5)
C(16)	6.3(7)	8.9(9)	8.3(9)	0.2(7)	-0.3(6)	-3.9(7)
C(17)	5.4(7)	7.2(8)	13.4(11)	-1.3(6)	0.1(7)	-3.6(8)
C(18)	6.5(8)	7.7(9)	12.3(11)	-2.4(7)	3.1(7)	-0.2(8)
C(19)	8.3(9)	8.9(9)	7.5(8)	-1.6(7)	2.1(7)	-0.5(7)
C(110)	7.6(8)	5.4(7)	6.4(7)	-1.0(6)	-2.1(6)	0.0(7)
C(111)	8.8(8)	3.9(7)	8.7(8)	0.4(6)	-1.4(7)	-1.6(6)
C(112)	11.2(10)	8.4(9)	6.7(8)	-0.7(8)	-0.3(7)	-1.4(7)
C(113)	18.7(15)	8.3(9)	6.1(8)	-1.7(10)	2.0(9)	-1.5(7)
C(114)	27.9(21)	6.4(9)	5.9(9)	-1.8(11)	-3.4(11)	-1.5(7)
C(115)	15.9(14)	7.1(9)	10.6(11)	0.4(9)	-6.3(10)	-0.5(8)
C(116)	11.6(10)	6.9(9)	7.6(9)	0.4(7)	-2.2(8)	-1.8(7)
C(117)	5.3(7)	8.2(9)	15.9(13)	-1.3(6)	-3.8(8)	6.2(9)
C(118)	7.0(8)	10.7(10)	7.4(9)	2.1(7)	-0.1(6)	2.6(7)
C(119)	5.5(6)	6.4(8)	7.4(8)	0.1(5)	-1.5(6)	-1.2(6)
C(120)	6.9(8)	7.8(9)	8.1(8)	1.7(6)	-1.6(6)	-0.3(7)
C(121)	10.5(10)	5.3(8)	13.7(12)	3.4(7)	-6.2(9)	-2.6(8)
Molecule 2						
Re(2)	4.96(2)	5.15(3)	5.36(2)	-0.36(2)	-0.31(2)	0.41(2)
C(21)	7.2(7)	6.6(8)	5.1(7)	-1.0(6)	-0.1(5)	1.1(6)
C(22)	6.4(7)	6.7(7)	5.9(7)	0.3(6)	0.2(6)	0.2(6)

Table 45 (Cont'd)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(21)	10.3(6)	6.9(5)	7.8(6)	0.3(5)	0.8(5)	-1.2(4)
O(22)	13.0(7)	7.9(6)	9.7(6)	-0.7(5)	2.4(6)	2.7(5)
C(23)	5.5(6)	5.4(7)	5.2(6)	0.0(5)	-0.9(5)	-0.3(5)
C(24)	5.8(6)	4.2(6)	5.2(6)	-0.4(5)	-0.6(5)	-0.3(5)
C(25)	5.1(6)	4.7(6)	5.5(6)	0.9(5)	0.0(5)	1.1(5)
C(26)	6.7(7)	8.0(8)	4.2(6)	-0.2(6)	1.3(5)	0.1(6)
C(27)	8.6(9)	8.1(9)	7.5(8)	-0.6(7)	3.3(7)	-0.4(7)
C(28)	6.0(7)	7.9(9)	11.0(10)	0.1(6)	2.0(7)	1.0(7)
C(29)	4.7(7)	8.8(9)	12.0(10)	0.1(6)	0.3(7)	0.2(8)
C(210)	5.0(6)	7.2(8)	8.5(8)	0.1(6)	-0.9(6)	-0.1(7)
C(211)	5.5(6)	5.2(7)	6.4(7)	0.7(5)	0.0(5)	0.7(5)
C(212)	6.9(7)	8.0(9)	6.8(8)	1.7(6)	-0.6(6)	0.8(6)
C(213)	11.0(10)	7.2(9)	7.2(8)	0.6(7)	-2.9(7)	0.0(7)
C(214)	15.1(12)	6.2(8)	6.1(8)	-1.5(8)	-2.6(8)	-0.4(6)
C(215)	11.1(10)	7.9(9)	5.4(7)	-1.1(7)	3.0(7)	-1.0(6)
C(216)	6.4(7)	7.2(8)	6.7(7)	-0.3(6)	0.2(6)	-0.1(6)
C(217)	7.0(8)	6.9(8)	11.1(10)	-0.5(6)	-1.6(7)	2.7(7)
C(218)	8.6(8)	7.5(8)	6.1(7)	-0.4(7)	-1.7(6)	1.1(6)
C(219)	4.3(6)	7.5(8)	11.0(9)	0.1(6)	-2.0(6)	2.9(7)
C(220)	8.0(8)	12.0(11)	7.1(8)	4.2(8)	-0.7(7)	-0.1(8)
C(221)	9.8(9)	5.2(8)	11.5(10)	2.8(7)	-5.2(8)	-2.3(7)

Table 46. Anisotropic Thermal Parameters (× 100) for $CpRe(CO)_2(N_2Ph_2)$ (13)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re	3.63(2)	3.16(2)	3.16(2)	-0.55(1)	0.86(2)	-0.43(1)
N(1)	4.9(7)	3.9(8)	4.0(7)	0.5(6)	1.9(6)	-0.5(6)
N(2)	3.9(6)	3.1(10)	3.7(6)	0.2(5)	0.5(5)	-0.3(5)
C(1)	6.3(10)	4.3(10)	5.0(9)	2.3(9)	0.8(8)	0.0(8)
O(1)	10.2(10)	13.2(14)	5.9(8)	2.8(10)	-1.8(7)	-2.3(9)
C(2)	9.0(10)	6.6(13)	6.0(11)	-1.3(11)	3.5(11)	-1.8(10)
O(2)	19.0(15)	6.5(9)	9.1(9)	1.3(10)	9.2(10)	2.7(8)

Table 47. Calculated Hydrogen Atom Coordinates (× 10^2) for $CpRe(CO)_2(C_2Ph_2)$ (11)

Atom	x	y	z
H(16)	486	295	127
H(17)	652	316	64
H(18)	636	264	-32
H(19)	453	194	-68
H(110)	287	176	-6
H(112)	46	246	232
H(113)	62	304	327
H(114)	258	333	372
H(115)	436	314	315
H(116)	421	263	219
H(117)	104	346	34
H(118)	2	208	-21
H(119)	-159	135	43
H(120)	-162	227	136
H(121)	-2	357	134
H(26)	670	1010	714
H(27)	843	1044	658
H(28)	847	1000	561
H(29)	679	916	519
H(210)	502	884	574
H(212)	219	968	800
H(213)	219	1002	900
H(214)	408	1004	952
H(215)	595	984	904
H(216)	595	947	806
H(217)	312	1037	587
H(218)	182	906	548
H(219)	23	862	620
H(220)	48	975	709
H(221)	232	1087	685

Table 48. Calculated Hydrogen Atom Coordinates (× 10^4) for $CpRe(CO)_2(N_2Ph_2)$ (13)

Atom	X	Y	Z
H(4)	-507	-374	1703
H(5)	-41	-2385	2493
H(6)	2633	-3063	3781
H(7)	4837	-1649	4371
H(8)	4455	293	3462
H(10)	2854	1807	-165
H(11)	3878	3173	-1654
H(12)	4988	5044	-723
H(13)	5081	5603	1697
H(14)	3989	4291	3175
H(15)	8067	2112	28
H(16)	6857	2216	2146
H(17)	7946	4133	3573
H(18)	10163	5085	2316
H(19)	10000	3795	101

Table 49. Calculated Meanplanes[a] and Dihedral Angles for $CpRe(CO)_2(C_2Ph_2)$ (11)

Plane No.	Atoms Defining Plane[b]	A	B	C	D (Å)	χ^2
Molecule 1						
1[c]	C11(-4), C12(4), C13(-9), C14(8)	-0.7014	0.6010	-0.3831	-1.1215	1.85
2	Ph ring: C15(-3), C16(-2), C17(5), C18(-4), C19(-1), C110(4)	-0.3401	0.9025	-0.2642	1.1866	0.64
3	Ph ring: C111(9), C112(-16), C113(13), C114(-3), C115(-4), C116(1)	-0.0652	0.9507	-0.3031	1.6644	3.49
4[d]	Cp ring: C117(1), C118(3), C119(-3), C120(2), C121(0)	-0.6724	0.6280	-0.3919	1.9777	0.18
5[e]	C13(5), C14(-5), C15(2), C111(-2)	-0.3567	0.9050	-0.2318	1.1749	0.50
6	C13, C14, C15	-0.3722	0.8949	-0.2461	1.0601	0.00
7	C13, C14, C111	-0.3338	0.9141	-0.2304	1.2669	0.00
8	Re1, C13, C14	-0.3092	0.9251	-0.2203	1.3954	0.00
Molecule 2						
9[f]	C21(-6), C22(6), C23(-12), C24(12)	-0.6430	0.6359	-0.4269	-1.5150	3.82

Table 49 (Cont'd)

Plane No.	Atoms Defining Plane[b]	A	B	C	D (Å)	χ^2
10	Ph ring: C25(1), C26(4), C27(1), C28(7), C29(10), C210(6)	-0.4006	0.8760	-0.2685	5.1724	1.55
11	Ph ring: C211(-8), C212(7), C213(5), C214(-15), C215(13), C216(-2)	0.0716	0.9773	-0.1994	9.647	4.49
12[g]	Cp ring: C217(-7), C218(7), C219(-3), C220(-1), C221(5)	-0.6175	0.6555	-0.4349	1.7220	1.04
13[h]	C23(14), C24(-14), C25(5), C211(-5)	-0.3157	0.9237	-0.2169	7.0664	4.85
14	C23, C24, C25	-0.3573	0.8957	-0.2647	5.7421	0.00
15	C23, C24, C211	-0.2456	0.9472	-0.2060	7.8525	0.00
16	Re2, C23, C24	-0.2582	0.9424	-0.2127	7.6255	0.00

[a] Plane equation is Ax + By + Cz - D = 0, with x, y, z in orthorhombic coordinates.

[b] Deviations of the atoms (Å × 10^3) from the meanplane are given in parentheses. The e.s.d.'s in these values are 0.01Å.

[c] Re1 deviation 1.077Å.

[d] Other deviations (Å): Re1 -1.956, C11 -3.05, C12 -3.08, C13 -2.99, C14 -2.95, C15 -3.20, C111 -3.33.

[e] Re1 deviation 0.109Å.

[f] Re2 deviation 1.086Å.

[g] Other deviations (Å): Re2 -1.968, C21 -3.07, C22 -3.10, C23 -3.03, C24 -2.98, C25 -3.22, C211 -3.42.

[h] Re2 deviation 0.118Å.

Table 49 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
Molecule 1		
1	4	2.3
1	5	28.0
2	3	16.2
2	5	2.1
2	6	2.2
3	5	17.5
3	7	16.1
5	8	3.0
6	7	2.6
Molecule 2		
9	12	1.9
9	13	28.0
10	11	28.2
10	13	6.3
10	14	2.7
11	13	22.6
11	15	18.3
13	16	3.5
14	15	7.8

Table 50. Calculated Meanplanes[a] and Dihedral Angles for $CpRe(CO)_2(N_2Ph_2)$ (13)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1[c]	Cp ring: C15(-3), C16(16), C17(-22), C18(18), C19(-9)	0.6194	-0.5492	0.3719	2.875	2.0
2	Ph ring: C9(-7), C10(7), C11(-8), C12(-5), C13(11), C14(-2)	0.8463	-0.4099	0.0978	1.135	0.8
3	Ph ring: C3(-9), C4(18), C5(-9), C6(-17), C7(23), C8(-5)	-0.4467	0.2640	0.9440	1.562	2.3
4	N1(-108), N2(98), C1(-137), C2(178)	0.48986	-0.6174	0.4595	0.7054	153.8
5	Re, N1, N2	0.0125	-0.5587	0.7948	0.6552	0.0
6	Re, C1, C2	-0.8415	0.5338	0.3087	2.057	0.0

[a] Plane equation is Ax + By + Cz = D, with x, y, z in orthorhombic coordinates.

[b] Deviations of the atoms (Å × 10^3) from the meanplane are given in parentheses.

[c] Re deviation Å.

Table 50 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (deg)
1	2	19.9
1	3	86.7
1	4	9.0
1	5	38.8
1	6	140.1
2	3	101.3
2	4	28.7
2	5	58.5
2	6	154.5
3	4	81.7
3	5	56.2
3	6	54.0
4	5	30.5
4	6	133.7
5	6	103.9

CHAPTER 4

X-RAY STRUCTURES OF cis-$Cp^*Re(CO)_2I_2$ and trans-$Cp^*Re(CO)_2Br_2$

4.1 Introduction

In view of the current activity in cyclopentadienyl and pentamethylcyclopentadienyl carbonyl chemistry, it is surprising that several members of the series of simple rhenium dicarbonyl dihalides $CpRe(CO)_2X_2$ and $Cp^*Re(CO)_2X_2$ ($Cp = \eta^5\text{-}C_5H_5$, $Cp^* = \eta^5\text{-}C_5Me_5$, X = Cl, Br, I) were unknown and reports of the others were fragmentary. $CpRe(CO)_2Br_2$ and $CpRe(CO)_2I_2$ (cis and trans) have been synthesized[109,110,144,145,146] but $CpRe(CO)_2Cl_2$ was unknown. $Cp^*Re(CO)_2I_2$ was briefly mentioned[147] but without details of the synthesis or stereochemistry; the dibromides and dichlorides were unknown.

It was hoped preferably to determine the X-ray structures of the cis and trans isomers of a single halide for better comparison, but unfortunately no single pair could be found which gave acceptable crystals for both isomers. However the structure determinations of cis-$Cp^*Re(CO)_2I_2$ (16) and trans-$Cp^*Re(CO)_2Br_2$ (17) were carried out to confirm the geometries deduced from the infrared intensities and to provide typical metrical data for this class of compounds, none of which has been structurally investigated before.

4.2 X-ray Data Collection and Refinement

A needle-like orange-red crystal of 16 and a small red crystal of 17, both obtained from methylene chloride/hexanes, was supplied by A.H. Klahn and used for the diffraction studies. The major morphological axis was aligned with the glass fibre and preliminary oscillation photos (Cu radiation) showed the crystals to be of an acceptable quality. The crystal was transferred to an Enraf-Nonius diffractometer, and the cell dimensions and space group were determined. The crystallographic data and scan conditions are listed in Table 51. The data collection was monitored by measuring two standard reflections every 1 hour of X-ray exposure time and showed only random fluctuations. The data were then corrected for Lorentz, polarization and absorption errors (ψ scans) and only the reflections with $I \geqq 3.0\sigma(I)$ were regarded as observed.

The heavy atoms were located from a Patterson map and all other non-hydrogen atoms from a series of difference Fourier syntheses interspersed with least squares refinement. All hydrogen atoms were placed in geometrically calculated positions with $\underline{d}$(C-H) = 0.98Å and thermal parameters of $\underline{U} = 0.09Å^2$ for 16 and $\underline{U} = 0.08Å^2$ for 17, and included as fixed contributions in further refinement. The disposition of the hydrogen atoms of the methyl groups was set identical to that found experimentally by Dahl[148] for $Cp^*Co(CO)_2$. The Re atom and halide ligands were refined anisotropically and all other atoms isotropically. For the final cycles the weighting scheme was of the form $\omega = 1/(\sigma(\underline{F}_o)^2 + n(\underline{F}_o)^2)$. The value of the parameter n (= 0.0005 for 16 and 0.0004 for 17) was that for which the variation of the averaged $\sum\{w|\underline{F}_o| - |\underline{F}_c|\}^2$ as a function of $|\underline{F}_o|$ and $(\sin\theta/\lambda)$ was kept to a minimum. Final residuals for 16 were $\underline{R}_f = 0.046$, $\underline{R}_{wf} = 0.058$ and for 17, $\underline{R}_f = 0.043$, $\underline{R}_{wf} = 0.054$. A small extinction correction was necessary for 17. The largest peaks in a final difference map for 16 were 1.7(2) and 1.2(2)eÅ^{-3} located near the C(1)O(1) and C(2)O(2) ligands respectively, and both 2.62Å from the Re atom. The location of the peaks was suggestive of a small fractional disorder of I(2) with the carbonyl group C(2)O(2) and I(1) with C(1)O(1). The two peaks were refined as iodine atoms with variable occupancy and a fixed isotropic temperature factor of 0.05Å^2. After several cycles, the peaks had refined to occupancies of 2.2% and 1.4%, respectively, with Re-I bond lengths of 2.65Å. The residuals were R_f = 0.044, R_{wf} = 0.056. However, because of the small occupancy of the partial iodine atoms this model was not pursued. A final difference map for 17 revealed numerous peaks and troughs of ±0.8-0.9(2)eÅ^{-3} around the Re and Br atoms and a single large peak of 1.6(2)eÅ^{-3} located 1.5Å from the Re and 1.2Å from C(1) having no apparent chemical significance.

Computer programs are from reference 65. A summary of the crystal data is in Table 51, bond lengths and angles in Table 52 for 16 and in Table 53 for 17, atom coordinates for 16 in Table 54 and for 17 in Table 55, anisotropic thermal parameters for 16 and 17 in Table 56, calculated hydrogen atom coordinates in Table 57 for 16 and in Table 58 for 17 and calculated meanplanes and dihedral angles in Table 59 for 16 and Table 60 for 17. Labelled SNOOPI diagrams are presented in Figures 31 and 32 for 16 and 17, respectively.

4.3 Results and Discussion

Both compounds exist as discrete molecular species in the solid state with no unusually short inter- or intramolecular contacts. SNOOPI diagrams and the atom labelling schemes for 16 and 17 are presented in Figures 31 and

FIGURE 31

SNOOPI Diagram of cis-$Cp^*Re(CO)_2I_2$

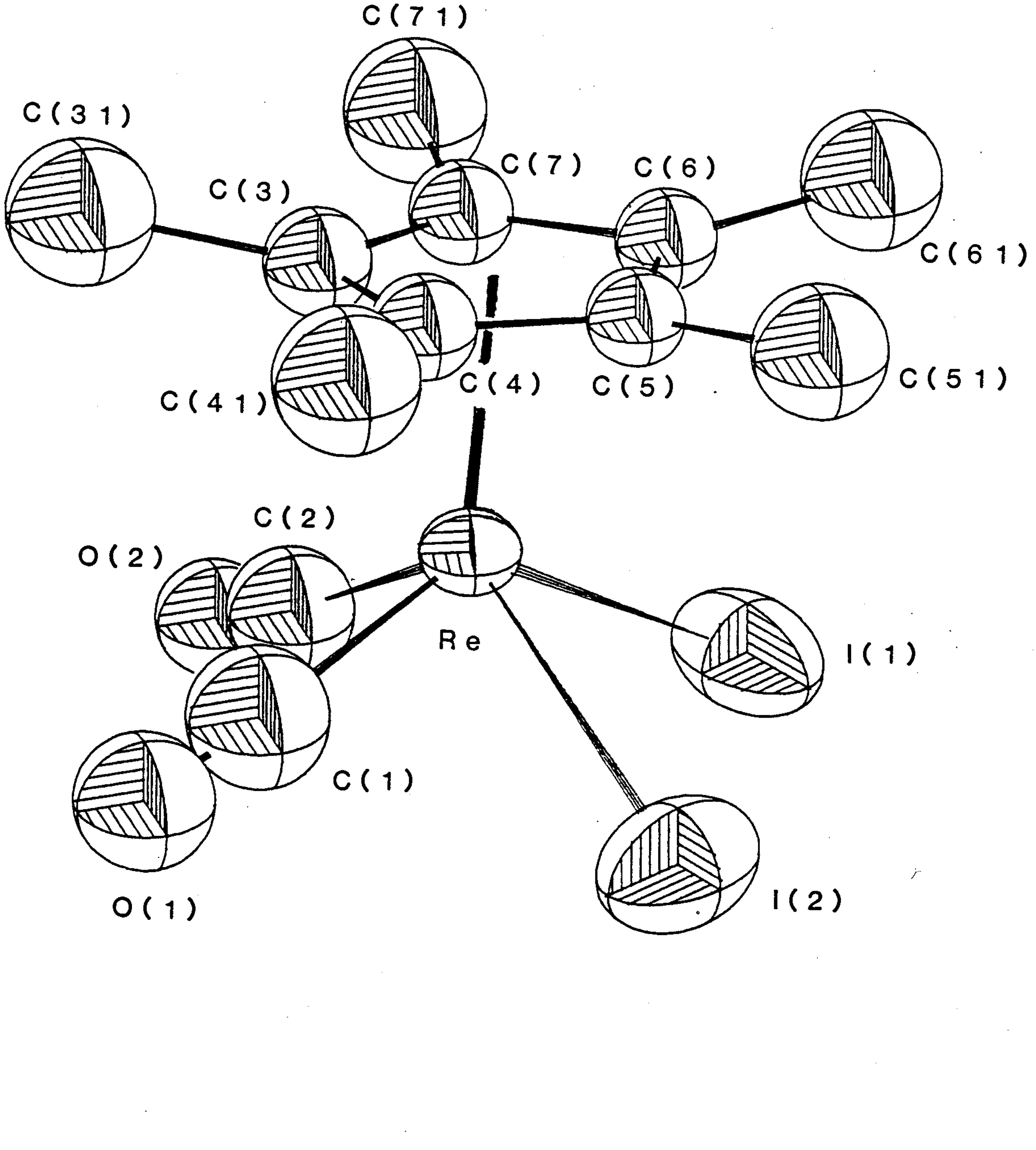

C(71)
C(31)
C(3)
C(7)
C(6)
C(61)
C(41)
C(4)
C(5)
C(51)
C(2)
O(2)
Re
I(1)
C(1)
O(1)
I(2)

FIGURE 32

SNOOPI Diagram of trans-$Cp^*Re(CO)_2Br_2$

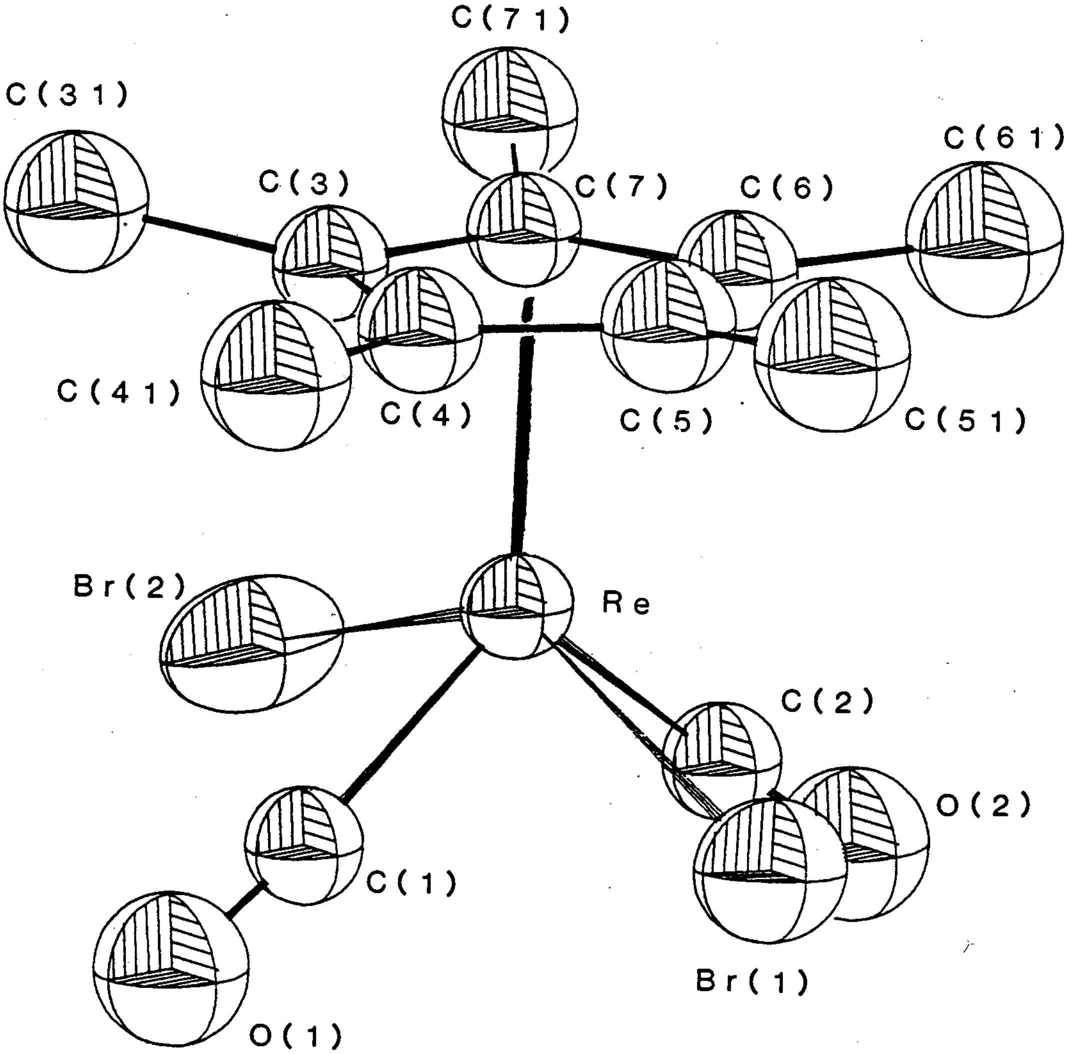

C(71)
C(31)
C(3)
C(7)
C(6)
C(61)
C(41)
C(4)
C(5)
C(51)
Br(2)
Re
C(2)
O(2)
C(1)
Br(1)
O(1)

Table 51. Summary of Crystal Data and Data Collection Conditions. Collection Conditions for 16 and 17.

compound	cis-$(C_5Me_5)Re(CO)_2I_2$	trans-$(C_5Me_5)Re(CO)_2Br_2$
formula	$C_{12}H_{15}I_2O_2Re$	$C_{12}H_{15}Br_2O_2Re$
mol. wt.	631.3	537.3
space group	$P2_1/n$	$P2_1/c$
a, Å	7.201(3)	8.512(2)
b, Å	27.254(5)	12.610(4)
c, Å	8.682(2)	13.794(6)
β, deg	110.04(4)	102.40(3)
Z	4	4
cell vol., $Å^3$	1600.2(2)	1446.1(3)
d_{calcd} g cm^{-3}	2.62	2.47
d_{meas} g cm^{-3}	2.64(1)	2.43(1)
cryst dimension, mm	0.05 × 0.08 × 0.25	0.09 × 0.12 × 0.15
radiation	Mo	Mo
μ, cm^{-1}	115.08	139.82
transmission factors	0.565-0.996	0.679-0.988
scan speeds, deg min^{-1}	1.0-4.0	0.5-4.0
scan width, deg	0.70 + dispersion	0.75 + dispersion
data limits, 2θ deg	3, 45	3, 46
measured reflections	2081	2008
observed reflections $I > 3.0\sigma(I)$	1510	1509
no. variables	84	85
R_F	0.046	0.043
R_{wF}	0.058	0.054
GOF	1.83	2.05
extinction	-	1.28×10^{-7}

Table 52. Bond Distances (Å) and Angles (deg) for cis-(C_5Me_5)Re$(CO)_2I_2$

Re-I(1)	2.773(2)	C(3)-C(4)	1.40(3)
Re-I(2)	2.761(2)	C(4)-C(5)	1.40(3)
Re-C(1)	1.92(3)	C(5)-C(6)	1.38(2)
Re-C(2)	2.03(3)	C(6)-C(7)	1.46(3)
Re-C(3)	2.20(2)	C(7)-C(3)	1.41(3)
Re-C(4)	2.26(2)	C(3)-C(31)	1.56(3)
Re-C(5)	2.37(2)	C(4)-C(41)	1.54(3)
Re-C(6)	2.41(2)	C(5)-C(51)	1.56(3)
Re-C(7)	2.26(2)	C(6)-C(61)	1.52(3)
C(1)-O(1)	1.03(3)	C(7)-C(71)	1.48(3)
C(2)-O(2)	0.85(3)	Re-Cp*(center)	1.951(9)

I(1)-Re-I(2)	83.00(7)	I(2)-Re-C(1)	75.3(8)
C(1)-Re-C(2)	78.5(10)	I(2)-Re-C(2)	122.4(6)
I(1)-Re-C(1)	129.5(7)	Re-C(1)-O(1)	177(2)
I(1)-Re-C(2)	75.4(7)	Re-C(2)-O(2)	173(2)

Table 53. Bond Distances (Å) and Angles (deg) for trans-$(C_5Me_5)Re(CO)_2Br_2$

Re-Br(1)	2.579(2)	C(3)-C(4)	1.46(2)
Re-Br(2)	2.579(2)	C(4)-C(5)	1.42(2)
Re-C(1)	1.93(2)	C(5)-C(6)	1.39(2)
Re-C(2)	1.92(2)	C(6)-C(7)	1.43(2)
Re-C(3)	2.25(2)	C(7)-C(3)	1.41(2)
Re-C(4)	2.31(2)	C(3)-C(31)	1.52(2)
Re-C(5)	2.36(2)	C(4)-C(41)	1.49(2)
Re-C(6)	2.31(2)	C(5)-C(51)	1.56(3)
Re-C(7)	2.33(2)	C(6)-C(61)	1.49(3)
C(1)-O(1)	1.10(2)	C(7)-C(71)	1.50(2)
C(2)-O(2)	1.15(2)	Re-Cp*(center)	1.945(7)
Br(1)-Re-Br(2)	138.97(7)	Br(2)-Re-C(2)	78.1(4)
Br(1)-Re-C(1)	76.9(4)	C(1)-Re-C(2)	104.3(6)
Br(1)-Re-C(2)	77.7(4)	Re-C(1)-O(1)	174(1)
Br(2)-Re-C(1)	77.6(4)	Re-C(2)-O(2)	176(1)

Table 54. Atom Coordinates (× 10^4) for Re and I Atoms and × 10^3 for Others) and Equivalent Isotropic Temperature Factors ($Å^2$ × 10) for cis-$(C_5Me_5)Re(CO)_2I_2$

Atom	X	Y	Z	B_{iso}
Re	748(1)	1315.5(3)	2286.7(8)	30.7(4)*
I(1)	3104(2)	525.7(7)	3791(2)	70.2(11)*
I(2)	2206(3)	1113.6(8)	-200(2)	79.2(14)*
C(1)	151(4)	194(1)	169(3)	74(6)
O(1)	185(3)	227.9(7)	130(2)	70(4)
C(2)	250(4)	157.9(9)	450(3)	59(5)
O(2)	311(2)	168.2(5)	546(2)	49(3)
C(3)	-205(3)	160.6(7)	242(2)	43(4)
C(31)	-236(4)	211.0(10)	314(3)	78(6)
C(4)	-240(3)	150.5(7)	76(2)	41(4)
C(41)	-307(4)	185.4(10)	-72(3)	79(7)
C(5)	-232(3)	99.7(7)	57(2)	34(3)
C(51)	-286(4)	70.2(9)	-107(3)	66(6)
C(6)	-196(3)	76.9(7)	207(2)	36(4)
C(61)	-196(4)	22.3(10)	242(3)	70(6)
C(7)	-170(3)	115.6(7)	328(2)	39(4)
C(71)	-152(4)	108.5(10)	502(3)	75(6)

Table 55. Atom Coordinates (× 10^4 for Re and Br Atoms and × 10^3 for Others) and Equivalent Isotropic Temperature Factors ($Å^2$ × 10) for trans-$(C_5Me_5)Re(CO)_2Br_2$

Atom	X	Y	Z	B_{iso}
Re	7392.4(6)	7800.5(5)	7093.1(4)	31.7(3)*
Br(1)	10184(2)	7859(2)	6649(1)	57.6(11)*
Br(2)	4403(2)	7696(2)	6207(1)	68.3(116)*
C(1)	735(2)	659(1)	624(1)	38(3)
C(2)	719(2)	899(1)	621(1)	39(3)
O(1)	734(1)	584(1)	582(1)	65(3)
O(2)	708(2)	974(1)	573(1)	65(3)
C(3)	650(2)	724(1)	842(1)	46(3)
C(4)	819(2)	695(1)	860(1)	42(3)
C(5)	906(2)	792(1)	869(1)	50(3)
C(6)	801(2)	878(1)	855(1)	39(3)
C(7)	742(2)	836(1)	839(1)	36(3)
C(31)	514(2)	647(1)	846(1)	55(4)
C(41)	884(2)	585(2)	873(1)	62(4)
C(51)	1093(2)	795(2)	902(1)	68(5)
C(61)	850(2)	991(2)	863(1)	67(4)
C(71)	494(2)	899(1)	841(1)	52(3)

Table 56. Anisotropic Thermal Parameters (Å^2 × 10^3) for

cis-Cp*Re(CO)$_2$I$_2$ (16)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re	33.0(4)	47.3(5)	36.3(4)	-3.6(4)	9.1(3)	2.3(4)
I(1)	59.2(9)	78.7(13)	129(2)	16.8(9)	14.9(9)	27.9(10)
I(2)	76.3(11)	140(2)	84.3(11)	-14.6(11)	48.2(9)	-28.2(11)

trans-Cp*Re(CO)$_2$Br$_2$ (17)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re	30.9(4)	51.2(4)	38.5(4)	-0.2(3)	8.1(2)	-0.4(3)
Br(1)	51.7(10)	93.3(14)	73.7(11)	-2.1(9)	24.8(8)	-2.6(10)
Br(2)	45.5(10)	152(2)	62.7(11)	-7.3(11)	1.7(8)	3.6(11)

Table 57. Calculated Hydrogen Atom Coordinates (× 10^3) for *cis*-$(C_5Me_5)Re(CO)_2I_2$ (16)

Atom	X	Y	Z
H(31)	-376	213	302
H(32)	-136	208	423
H(33)	-185	233	247
H(41)	-449	184	-120
H(42)	-238	215	- 16
H(43)	-224	171	-131
H(51)	-286	70	-107
H(52)	-187	44	- 67
H(53)	-231	94	-168
H(61)	-332	12	218
H(62)	-126	11	167
H(63)	-101	22	352
H(71)	-281	101	506
H(72)	- 49	83	532
H(73)	- 87	140	547

Table 58. Calculated Hydrogen Atom Cordinates (× 10^3) for trans-(C_5Me_5)Re$(CO)_2$$Br_2$ (17)

Atom	X	Y	Z
H(31)	507	639	916
H(32)	550	587	811
H(33)	426	683	800
H(41)	884	578	944
H(42)	997	579	862
H(43)	820	532	831
H(51)	1119	798	974
H(52)	1112	851	860
H(53)	1112	726	871
H(61)	867	1011	934
H(62)	753	1021	818
H(63)	937	990	829
H(71)	487	908	911
H(72)	414	852	799
H(73)	513	959	802

Table 59. Calculated Meanplanes[a] and Dihedral Angles for *cis*-$(C_5Me_5)Re(CO)_2I_2$ (16)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1	Re, I1, I2	0.6629	0.6219	0.1639	2.912	0
2	Re, C1, C2	-0.898	0.419	0.433	1.88	0
3	I1(0.0), I2(0.0), C1(-15), C2(12)	0.945	0.203	-0.083	2.13	32.1
4[c]	C3(-1.5), C4(0.1) C5(1.0), C6(-1.7), C7(1.9)	0.986	0.055	-0.194	-1.61	1.71

[a] Plane equation is Ax + By + Cz = D, with x,y,z in orthorhombic coordinates.

[b] Deviation of the atoms (Å × 10^2) from the meanplane are given in parentheses

[c] Deviations: Re, 1.951(9); C31, 0.28(4); C41, 0.19(4); C51, 0.14(3); C61, 0.16(3); C71, 0.15(4) Å.

Table 59 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (Deg)
1	2	106.2(7)
1	3	30.7(4)
1	4	41.2(6)
2	3	136.8(8)
2	4	147.4(9)
3	4	10.6(7)

Table 60. Calculated Meanplanes[a] and Dihedral Angles for trans-$(C_5Me_5)Re(CO)_2Br_2$ (17)

Plane No.	Atoms Defining Plane[b]	A	B	C	D	χ^2
1	Re, Br1, Br2	-0.0383	0.9986	-0.0278	9.310	0
2	Re, C1, C2	0.9904	0.0439	-0.0848	5.835	0
3	Br1(0.4), Br2(0.6), C1(-28), C2(-25)	-0.1247	0.0200	0.9956	8.244	380
4[c]	C3(0.6), C4(-1.3), C5(1.5), C6(-0.7), C7(0.1)	-0.1767	0.0240	0.9990	10.842	1.14

[a] Plane equation is Ax + By + Cz = D, with x, y, z in orthorhombic coordinates.

[b] Deviation of the atoms (Å × 10^2) from the meanplane are given in parentheses.

[c] Deviations: Re, 1.944(7); C31, 0.24(3); C41, 0.04(3); C51, 0.19(3); C61, 0.08(3); C71, 0.28(3) Å.

Table 60 (Cont'd)

Dihedral Angles (Between Normals to Meanplanes)

Plane No.	Plane No.	Angle (Deg)
1	2	89.9(4)
1	3	90.7(4)
1	4	90.3(5)
2	3	89.6(6)
2	4	92.6(7)
3	4	30.0(5)

32, respectively.

The overall molecular configuration consists of a rhenium atom bonded to a Cp* ligand and to two CO ligands and two halide ligands in a cis arrangement for 16 and a trans arrangement for 17. This is in agreement with the ν(CO) infrared intensity patterns observed by Klahn[149]. Neither molecule has any crystallographically imposed symmetry in the lattice; however both molecules (including the hydrogen atoms) possess an approximate mirror plane that is easily seen in Figure 33 which shows the molecules side by side projected onto the plane of the Cp* ring. In 16 this bisects the C_5Me_5 ligand at C(3) and C(31) and bisects the angles I(1)-Re-I(2) and C(1)-Re-C(2). In 17 it bisects C(5) and C(51) and both of the Re-Br bonds.

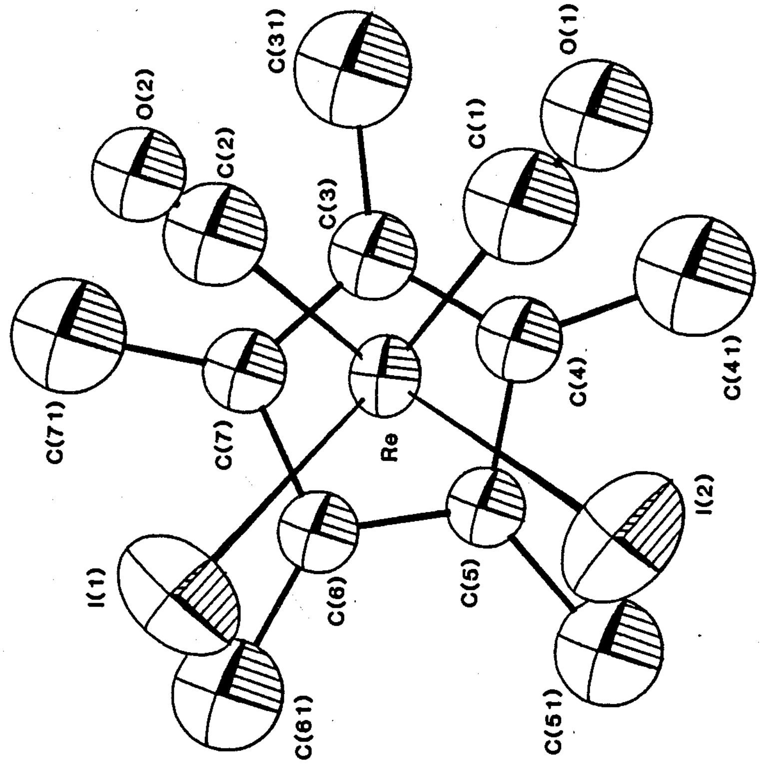
4.3.1 Rhenium Coordination

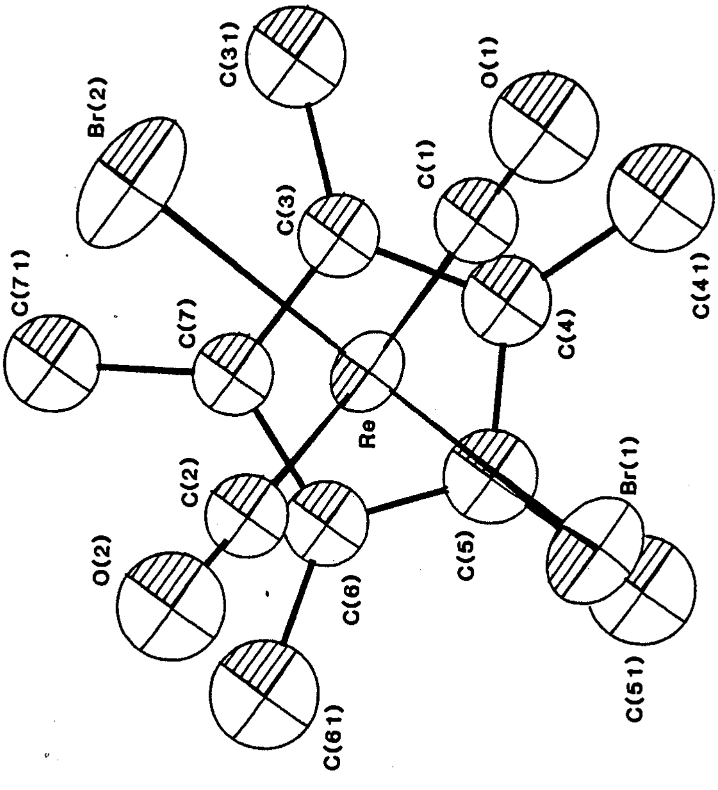
The rhenium (III) atoms are seven coordinate if the Cp* group is considered as a three coordinate monoanion and the overall geometry is that of a typical four-legged piano stool Cp^*ML_4 complex[150]. The Re-C(CO) bond lengths for both compounds are in the range 1.9-2.0Å that we have observed in $CpRe(CO)_2(\eta^2\text{-}C_2Ph_2)$ and $CpRe(CO)_2(\eta^2\text{-}N_2Ph_2)$ (Chapter 3). The Re-Br and Re-I bond lengths are unexceptional.

The Re-Cp* (centroid) distances of 1.945(7)Å and 1.951(9)Å in 16 and 17 are identical and evidently insensitive to the differing cis-and trans-arrangements of the carbonyl and halide basal ligands in the two structures. Examination of the individual Re-C bond lengths to the Cp* ligand reveals, however, differences in 16 and 17. The individual Re-C bonds in 17 vary from 2.25(2) to 2.36(2)Å in no evidently regular manner, but in 16 they vary systematically in a way that is chemically sensible even though only marginally insignificant at the level of precision. The bonds Re-C(5) and

FIGURE 33

Projection onto the Cp* plane of (16) and (17)


C(31)
O(2)
C(2)
C(3)
O(1)
C(1)
C(71)
C(7)
Re
C(4)
C(41)
I(1)
C(6)
C(5)
I(2)
C(61)
C(51)


C(31)
Br(2)
O(1)
C(1)
C(3)
C(71)
C(7)
C(41)
C(4)
Re
Br(1)
C(2)
O(2)
C(6)
C(5)
C(51)
C(61)

Re-C(6) are long at 2.37(2) and 2.41(2)Å, Re-C(4) and Re-C(7) intermediate at 2.26(2)Å and Re-C(3) the shortest at 2.20(2)Å. With reference to the projection in Figure 33, it can be seen that the long Re-C bonds are 'opposite' or pseudotrans to the two CO groups, while the shortest is 'opposite' the iodide ligands. The asymmetry therefore follows a pattern that would be expected considering the π-acceptor properties possessed by CO ligands but not by iodide. The CO ligands withdraw π-electron density and diminish the π-contribution to the Re-C(Cp*) bonds trans to them thereby resulting in lengthening. The asymmetry pattern also can be interpreted as a tilt of the Cp* ring away from the bulky iodide ligands. Hoffmann[150] has suggested that a degree of tilting of the Cp ring observable in some $CpML_4$ compounds is accountable by interactions involving δ orbitals of the metal and Cp ligand. In essence, it appears that (in projection) the eclipsing of a M-C(Cp) bond with a M-L bond results in a slightly long M-C(Cp) bond length, whereas when a M-C(Cp) bond is staggered between the M-L bonds, it is short. It is possible that such an effect also contributes to the pattern of Re-C(Cp*) bond lengths observed here, but it must be pointed out that in cis-$Cp^*Re(CO)_2I_2$ (<u>16</u>) none of the Re-C(Cp*) is exactly eclipsed. Nevertheless, it is true (Fig. 30) that the longer bonds that occur for Re-C(5) and Re-C(6) are the ones most nearly eclipsed by the Re-I bonds, while the shortest (Re-C(3)) is the most staggered one. In trans-$Cp^*Re(CO)_2Br_2$ (<u>17</u>) eclipsing does occur, but no significant statistical variation of the Re-C(Cp*) bond lengths is observable.

The C-O bond lengths is <u>17</u> are in the expected range of 1.10-1.15(2)Å for metal carbonyl groups, but abnormally short values (0.85(3) and 1.03(3)Å) were obtained for <u>16</u>. These unusually short distances are attributed to the 1-2% disordered iodine atom electron density contributions in these regions, causing the apparent shortening. The C(1)-Re-C(2) angle in 16 is 78.5(10)° compared with the estimated value of 80° from the infrared ν(CO) intensities[149]. The I(2)-Re-C(1) and I(1)-Re-C(2) angles are both equal at 75.4(7)° however the trans angles I(1)-Re-C(1) and I(2)-Re-C(2) are not equal, being 129.5(7) and 122.4(6)° respectively, although it would appear from the (pseudo)symmetry of the molecule (Fig. 33) that the two angles should be equal. The C(1)-Re-C(2) interbond angle in 17 is 104.3(6)° substantially less than that estimated 115°, from the ν(CO) intensities[149]. The Br(1)-Re-Br(2) angle is 138.9(7)° and the Br-Re-C angles are all very similar with the average of 77.6(3)°. These Br-Re-C angles are larger than the I-Re-C cis angles in 16 (75.3(7)°) and is attributed to the repulsion between the mutually cis iodine atoms contracting the I-Re-C cis angles.

4.3.2 The Pentamethylcyclopentadienyl Ligand

The C-C bond lengths in the Cp* ligands in 16 and 17 are similar and normal ranging from 1.38(2)-1.46(3)Å for the ring carbons and 1.48(3)-1.56(3)Å for the C(ring)-C(methyl) distances. The carbon atoms of the ring form well defined planes (χ^2 values = 1.71, 1.14) and there is no systematic distortion from this planarity as was observed for $(C_5Me_5)Co(CO)_2$[148]. The methyl substituents are all displaced from these planes in a direction away from the Re atom with average deviations of 0.18(2)Å in 16 and 0.17(2)Å in 17.

A survey by Dahl[148] of various pentamethylcyclopentadienyl-metal complexes revealed this type of displacement to be general, with average deviation ranging from 0.037Å to 0.205Å. In 17 there are additional features to note, which are that the methyl carbon deviations conform to the approximate molecular mirror plane symmetry, and the largest deviations are displayed by the methyl groups C(31), C(51) and C(71) that are closest to eclipsing the bromine positions in projection (Fig. 33). Thus, the related pair C(41) and C(61) show very small deviations (+0.04Å and +0.08Å) while those of the pair C(31) and C(71) are much larger (+0.24Å and +0.28Å) and that of C(51) is +0.19Å. In 16 the methyl carbon deviations also conform to the mirror symmetry, but C(41)-C(71) all have similar displacements of *ca*. 0.14-0.19Å while that of the unique methyl group C(31) is larger at 0.28Å.

APPENDIX A

X-RAY STRUCTURE OF $(\mu\text{-Cl})_2Os_3(CO)_{10}$

A.1 Introduction

A number of X-ray crystal structures have been performed on triosmium carbonyl clusters of the general formula $(\mu\text{-H})(\mu\text{-Y})Os_3(CO)_{10}$ where Y = H, Cl, Br, SEt, OMe, $CHCH{=}NEt_2$, etc.[151,152,153,154] However, only in the case of the methoxide ligand[154] has the series $(\mu\text{-H})_{0-2}(\mu\text{-Y})_{2-0}Os_3(CO)_{10}$ been structurally characterized. In order to complete a second series of compounds, specifically that of $(\mu\text{-H})_{0-2}(\mu\text{-Cl})_{2-0}Os_3(CO)_{10}$, we undertook to determine the X-ray structure of the missing member of this series; $(\mu\text{-Cl})_2Os_3(CO)_{10}$. This would provide the final link in this series so valid metrical comparisons could be made.

A.2 X-ray Data Collection and Refinement

A yellow crystal, 0.25 x 0.30 x 0.22 mm, was obtained by recrystallization from hexanes from Dr R.K. Pomeroy and used for the diffraction studies. Weissenberg and precession photographs (Cu radiation) showed the crystal to be orthorhombic and systematic absences 0kl: k = 2n+1, h0l: l = 2n+1, hk0: h = 2n+1, uniquely defined the space group as Pbca. Data were collected on a Picker FACS-I diffractometer (Mo radiation) with a scan rate of $2°\ min^{-1}$ for $3.0° \leq 2\theta \leq 45°$ and a symmetrical scan width of $(1.2 + 0.692\tan\theta)°$. Peak profile analyses were performed on all reflections. Two standards were measured after every 70 reflections and showed a small overall gradual decline (4%) in average intensity; the data were scaled accordingly.

Absorption corrections were applied via an analytical correction (transmission factors 0.039-0.070). 2838 reflections out of a total of 4619 had I ≧ 2.3σ(I) and were regarded as observed. The positions of the osmium atoms were located by MULTAN and the chlorine, carbon and oxygen atoms were located from a series of difference Fourier synthesis and interspersed with least squares refinement. The metals and chlorine atoms were refined anisotropically and the carbonyl groups isotropically. A final difference Fourier map revealed little evidence of anisotropy around the carbonyl ligands. Computer programs are from reference 64. A summary of the crystal data and collection parameters are given in Table 61, bond distances and angles in Table 62, atom coordinates in Table 63 and anisotropic thermal parameters in Table 64.

A.3 Results and Discussion

The crystal consists of discrete molecular units of $(\mu\text{-Cl})_2Os_3(CO)_{10}$, with no abnormally short intermolecular contacts. The complex crystallizes in the space group Pbca with 16 molecules per unit cell giving two independent molecules in the asymmetric unit. The two molecules did not differ significantly except in the unbridged Os-Os distance, (Table 65) 3.233(1)Å versus 3.260(1)Å and in the Os-Cl-Os angle, 82.04(8)° versus 82.65(8)°. No disorder was apparent and the other bond lengths are in good agreement with each other. Only one of the molecules is presented in the ORTEP view (Fig. 34) along with the atom labelling scheme.

The molecule has approximate mirror symmetry and is based upon a triangular arrangement of osmium atoms. Os(1) is associated with four terminal carbonyl ligands and Os(2) and Os(3) are linked to three terminal carbonyl ligands and are mutually bridged by two chloride ligands.

The three Os atoms essentially define an isosceles triangle with the

FIGURE 34

ORTEP Diagram of $(\mu\text{-Cl})_2Os_3(CO)_{10}$

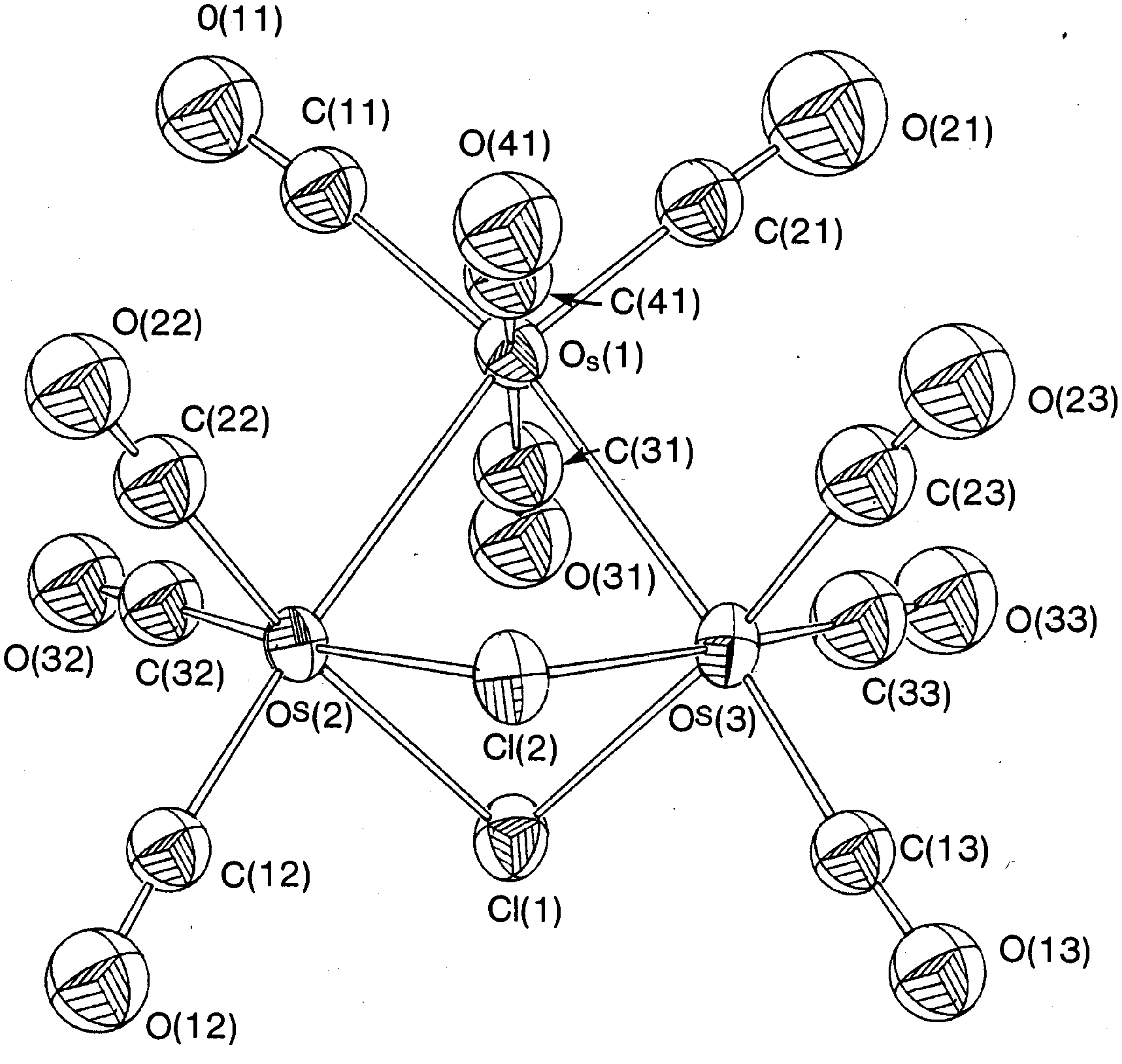

O(11)
C(11)
O(41)
O(21)
C(21)
C(41)
O(22)
Os(1)
C(22)
O(23)
C(31)
C(23)
O(31)
O(33)
O(32)
C(32)
C(33)
Os(2)
Os(3)
Cl(2)
C(13)
C(12)
Cl(1)
O(13)
O(12)

Table 61. Summary of Crystal Data and Collection Parameters for $Os_3(CO)_{10}(Cl_2)$ (18)

compound	$C_{10}Cl_2O_{10}Os_3$
mol. wt.	921.61
a, Å	25.580(8)
b, Å	22.832(6)
c, Å	12.036(3)
space group	Pbca
cell vol., $Å^3$	7029.5
Z	16
ρ (calcd) g cm^{-3}	3.483
ρ (obsd) g cm^{-3}	3.25
μ (MoK_α) cm^{-1}	217.0
transmission factors	0.039-0.070
total reflections, measured	4619
total reflections, $I \geq 2.3\sigma(I)$	2838
data limits (deg)	3, 45
final R	4.1
final R_w	4.9

Table 62. Bond Distances (Å) and Angles (deg) for $Os_3(CO)_{10}Cl_2$ (18)

Bond Lengths

Os(1)-Os(2)	2.849(1)	Os(4)-C(44)	1.95(3)
Os(1)-Os(3)	2.855(2)	Os(5)-C(15)	1.85(2)
Os(2)-Os(3)	3.233(1)	Os(5)-C(25)	1.83(2)
Os(4)-Os(5)	2.846(1)	Os(5)-C(35)	1.91(2)
Os(4)-Os(6)	2.850(2)	Os(6)-C(16)	1.83(3)
Os(5)-Os(6)	3.260(1)	Os(6)-C(26)	1.82(2)
Os(2)-Cl(1)	2.461(6)	Os(6)-C(36)	1.82(3)
Os(2)-Cl(2)	2.462(6)	C(11)-O(11)	1.09(3)
Os(3)-Cl(1)	2.462(6)	C(21)-O(21)	1.21(3)
Os(3)-Cl(2)	2.467(6)	C(31)-O(31)	1.14(3)
Os(5)-Cl(3)	2.473(6)	C(41)-O(41)	1.12(3)
Os(6)-Cl(3)	2.479(6)	C(12)-O(12)	1.15(3)
Os(5)-Cl(4)	2.457(6)	C(22)-O(22)	1.17(3)
Os(6)-Cl(4)	2.463(6)	C(32)-O(32)	1.16(3)
Os(1)-C(11)	1.92(3)	C(13)-O(13)	1.15(3)
Os(1)-C(21)	1.85(3)	C(23)-O(23)	1.17(3)
Os(1)-C(31)	1.91(3)	C(33)-O(33)	1.18(3)
Os(1)-C(41)	1.95(3)	C(14)-O(14)	1.20(3)
Os(2)-C(12)	1.92(2)	C(24)-O(24)	1.14(3)
Os(2)-C(22)	1.87(3)	C(34)-O(34)	1.17(3)
Os(2)-C(32)	1.83(2)	C(44)-O(44)	1.14(3)
Os(3)-C(13)	1.91(2)	C(15)-O(15)	1.15(3)
Os(3)-C(23)	1.88(3)	C(25)-O(25)	1.15(3)

Table 62 (Cont'd)

Os(3)-C(33)	1.84(3)	C(35)-O(35)	1.15(3)
Os(4)-C(14)	1.83(3)	C(16)-O(16)	1.18(3)
Os(4)-C(24)	1.90(2)	C(26)-O(26)	1.17(3)
Os(4)-C(34)	1.90(2)	C(36)-O(36)	1.15(3)

Bond Angles

Os(1)-Os(2)-Os(3)	55.56(3)	Os(2)-Os(3)-Cl(2)	48.9(1)
Os(1)-Os(3)-Os(2)	55.39(3)	Os(1)-Os(3)-Cl(1)	86.1(2)
Os(2)-Os(1)-Os(3)	69.06(4)	Os(1)-Os(3)-Cl(2)	86.2(2)
Os(4)-Os(5)-Os(6)	55.13(3)	Os(3)-Os(2)-Cl(1)	49.0(1)
Os(4)-Os(6)-Os(5)	55.04(3)	Os(3)-Os(2)-Cl(2)	49.1(1)
Os(5)-Os(4)-Os(6)	69.83(4)	Os(4)-Os(5)-Cl(3)	86.8(1)
Os(2)-Cl(1)-Os(3)	82.1(2)	Os(4)-Os(5)-Cl(4)	85.5(1)
Os(2)-Cl(2)-Os(3)	82.0(2)	Os(4)-Os(6)-Cl(3)	86.6(1)
Os(5)-Cl(3)-Os(6)	82.3(2)	Os(4)-Os(6)-Cl(4)	85.4(1)
Os(5)-Cl(4)-Os(6)	83.0(2)	Os(5)-Os(6)-Cl(3)	48.8(1)
Os(1)-Os(2)-Cl(1)	86.3(2)	Os(5)-Os(6)-Cl(4)	48.4(1)
Os(1)-Os(2)-Cl(2)	86.4(2)	Os(6)-Os(5)-Cl(3)	48.9(1)
Os(2)-Os(3)-Cl(1)	48.9(1)	Os(6)-Os(5)-Cl(4)	48.6(1)

Table 63. Atom Coordinates (× 10^4) for $Os_3(CO)_{10}Cl_2$

Atom	X	Y	Z	B_{iso}
Os(1)	2517.3(4)	1274.5(4)	6779.0(8)	28.7(4)*
Os(2)	1644.0(3)	782.3(4)	7913.9(8)	25.3(4)*
Os(3)	2628.3(4)	1398.4(4)	9127.9(8)	29.6(5)*
Os(4)	318.8(4)	1826.2(4)	3263.2(8)	24.3(4)*
Os(5)	-598.2(3)	1400.4(4)	2194.6(8)	26.4(4)*
Os(6)	480.0(3)	657.1(4)	2507.9(8)	25.5(4)*
Cl(1)	2284(2)	393(3)	9252(5)	34(3)*
Cl(2)	1689(2)	1642(3)	9145(5)	35(3)*
Cl(3)	-426(2)	457(3)	3136(5)	33(3)*
Cl(4)	4(2)	969(3)	830(5)	34(3)*
C(11)	2303(10)	1103(11)	5282(21)	39(5)
O(11)	2163(7)	971(9)	4461(16)	59(5)
C(21)	3138(10)	1643(11)	6421(21)	40(6)
O(21)	3563(9)	1847(10)	6214(19)	82(6)
C(31)	2858(10)	535(11)	6979(22)	42(6)
O(31)	3054(7)	91(8)	7095(15)	54(4)
C(41)	2126(10)	2009(11)	6827(21)	41(6)
O(41)	1910(7)	2436(9)	6831(16)	61(5)
C(12)	1080(9)	469(11)	8789(20)	37(5)
O(12)	768(7)	252(8)	9345(15)	56(4)
C(22)	1199(11)	1179(11)	6932(22)	45(6)
O(22)	908(8)	1443(9)	6375(16)	58(5)

Table 63 (Cont'd)

Atom	X	Y	Z	B_{iso}
C(32)	1704(10)	164(11)	6954(20)	37(5)
O(32)	1746(7)	-241(8)	6375(15)	49(4)
C(13)	2650(9)	1435(11)	10709(20)	37(5)
O(13)	2692(7)	1474(8)	11658(15)	49(4)
C(23)	2855(11)	2144(12)	8836(24)	52(7)
O(23)	2973(8)	2636(9)	8761(18)	69(5)
C(33)	3299(11)	1119(12)	8982(23)	49(6)
O(33)	3733(8)	954(9)	8883(16)	59(5)
C(14)	74(10)	2563(12)	3565(21)	43(6)
O(14)	-91(8)	3043(9)	3763(17)	70(5)
C(24)	999(9)	1913(10)	3872(19)	33(5)
O(24)	1401(7)	1976(8)	4269(14)	51(4)
C(34)	46(9)	1488(10)	4579(18)	29(5)
O(34)	-123(6)	1250(7)	5364(13)	42(4)
C(44)	552(9)	2064(11)	1791(19)	35(5)
O(44)	687(6)	2218(7)	936(14)	44(4)
C(15)	-971(9)	1696(10)	3386(19)	33(5)
O(15)	-1195(7)	1874(7)	4142(14)	46(4)
C(25)	-636(9)	2120(10)	1522(19)	33(5)
O(25)	-662(7)	2577(9)	1121(16)	57(5)
C(35)	-1189(9)	1078(10)	1449(20)	34(5)
O(35)	-1561(7)	933(8)	986(15)	54(4)
C(16)	563(10)	-108(12)	1950(21)	44(6)

Table 63 (Cont'd)

Atom	X	Y	Z	B_{iso}
O(16)	617(8)	-594(9)	1644(17)	68(5)
C(26)	1109(9)	921(11)	2013(20)	37(5)
O(26)	1511(7)	1081(8)	1662(15)	53(4)
C(36)	754(10)	513(11)	3873(20)	37(5)
O(36)	946(7)	383(8)	4704(15)	52(4)

* Anisotropic thermal parameters are found in Table 60.

Table 64. Anisotropic Thermal Parameters ($Å^2 \times 10^2$) for $[(\mu\text{-Cl})_2Os_3(CO)_{10}]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Os(1)	3.04(5)	3.66(6)	4.12(6)	-0.07(5)	0.22(5)	0.14(5)
Os(2)	2.60(5)	3.26(5)	3.66(5)	-0.09(4)	-0.08(4)	-0.23(4)
Os(3)	3.15(6)	3.72(6)	4.30(6)	-0.07(5)	-0.77(5)	-0.44(5)
Os(4)	2.73(5)	3.11(5)	3.37(5)	-0.14(4)	-0.23(4)	-0.19(4)
Os(5)	2.57(5)	3.86(6)	3.47(6)	-0.16(4)	-0.35(4)	0.38(4)
Os(6)	3.07(5)	3.05(5)	3.53(5)	0.16(4)	-0.21(5)	0.12(4)
Cl(1)	3.7(3)	4.0(4)	5.1(4)	0.3(3)	-1.0(3)	0.2(3)
Cl(2)	3.8(4)	3.7(3)	5.5(4)	0.3(3)	-0.2(3)	-1.0(3)
Cl(3)	3.5(3)	4.4(4)	4.7(4)	-0.8(3)	-0.7(3)	1.0(3)
Cl(4)	4.3(4)	5.0(4)	3.7(3)	0.0(3)	0.0(3)	0.1(3)

angle at the unique osmium, Os(2)-Os(1)-Os(3), being 69.04(3)°, while the angles defined by Os(1)-Os(2)-Os(3) and Os(1)-Os(3)-Os(2) are 55.56(3) and 55.39(3)° respectively (in the second molecule the corresponding osmium angles are 69.83(4), 55.13(3) and 55.04(3)°). This compares[152] with the 'close to' equilaterial triangle found for the three osmium atoms in $(\mu\text{-H})(\mu\text{-Cl})\text{Os}_3(\text{CO})_{10}$ and the isosceles triangle noted[151] for $(\mu\text{-H})_2\text{Os}_3(\text{CO})_{10}$. In the former the angle at the unique osmium is 60.32(2)° while in the latter the same angle is 56.93(4)°. This is a clear demonstration of the Cl ligands pushing the two bridged osmium atoms further apart with a concommitant increase in the angle at the unique osmium.

The dibridged osmium-osmium distance in <u>18</u> is 3.233(2)Å (and 3.260(2)Å in the other molecule). As shown in Table 65, as the bridging hydride is replaced by bridging chloride ligands the corresponding Os-Os vector increases significantly. This increase in the Os-Os separation reflects the decrease in Os-Os bond order from 2 in $(\mu\text{-H})_2\text{Os}_3(\text{CO})_{10}$ to 1 in $(\mu\text{-H})(\mu\text{-Cl})\text{Os}_3(\text{CO})_{10}$ to 0 in $(\mu\text{-Cl})_2\text{Os}_3(\text{CO})_{10}$. A parallel increase, although not as large, was found for the $(\mu\text{-H})_2$,$(\mu\text{-H})(\mu\text{-OMe})$ and $(\mu\text{-OMe})_2$ series[154]. However, no increase was noted for the non-bridged Os-Os distances in the methoxide series but a substantial increase is observed in this length , 2.815(1) to 2.833(1) to 2.852(2)Å, for the series $(\mu\text{-H})_{2-0}(\mu\text{-Cl})_{0-2}\text{Os}_3(\text{CO})_{10}$.

The bridging Cl atoms are bound to the triosmium core with an average bond length of 2.463(3)Å (2.468(3)Å in molecule 2) and Os-Cl-Os angle of 82.04(8)°. The Os-Cl-Os angle is larger than found[151] with (μ-H)(μ-Cl) as bridging ligands at 70.83(9)° but the Os-Cl bond lengths are similar. The acute value of this angle is symptomatic of the presence of metal-metal bonding and the larger angle observed in <u>18</u> is consistent with no metal-

Table 65. Distances (Å) and Angles (deg) Within the Complexes $(\mu\text{-H})_2Os_3(CO)_{10}$, $(\mu\text{-H})(\mu\text{-Cl})Os_3(CO)_{10}$ and $(\mu\text{-Cl})_2Os_3(CO)_{10}$

	$(\mu\text{-H})_2$ Compound[a]	$(\mu\text{-H})(\mu\text{-Cl})$ Compound[b]	$(\mu\text{-Cl})_2$ Compound[c]
Bridged Os-Os	2.682(1)	2.846(1)	3.233(1) 3.260(1)
Non-bridged Os-Os (av)	2.815(1)	2.833(1)	2.852(2) 2.848(3)
Os-Cl (av)	-	2.456(3)	2.463(3) 2.468(3)
Os-Cl-Os	-	70.83(9)	82.04(8) 82.65(8)

[a] Ref.

[b] Ref.

[c] This work; molecule 1, molecule 2.

metal bonding being present.

The carbonyl groups are in both axial and equatorial positions but because of the errors on the Os-C and C-O bond lengths, no clear distinction is possible in the present structural determination.

The present structural analysis completes the investigation of $(\mu\text{-H})_{0-2}(\mu\text{-Cl})_{2-0}Os_3(CO)_{10}$, complexes providing the final link so that valid metrical comparisons could be made.

APPENDIX B

COLLECTION AND TREATMENT OF THE X-RAY DIFFRACTION DATA

B.1 Organization

The X-ray data of the compounds comprising this investigation were collected on two different diffractometers, a Picker FACS-I and an Enraf Nonius CAD-4. The data collected on the Picker diffractometer were solved and refined on a PDP 8/e computer and the data collected on the Enraf-Nonius diffractometer were solved and refined on a VAX 11-750 computer, both utilizing the NRC crystal structure package appropriate for each computer[64,65]. Since the diffractometers used, and thus the collection of data (and to a lesser extent the refinement of the structures) are not identical for every structure investigated, a short account specific to each determined structure is included in the relevant section of the thesis. Many of the specific data collection parameters are tabulated in the appropriate section.

B.2 Preliminary Investigation

Single crystals were mounted with epoxy cement either on the end of a thin glass fibre or in an appropriately sized Lindemann glass capillary with the major morphological axis, where possible, parallel to the fibre. Copper radiation was used to obtain oscillation photographs on all compounds and many (see individual sections) also had zero and upper level Weissenberg and oscillation photographs taken so as to obtain the approximate cell parameters and correct space group.

Crystals mounted on the Picker FACS-I were optically centered and strong reflections with χ close to 0°, and separated in ϕ by approximately 90°, were indexed using the photographic information. Twelve of the strongest reflections having $2\theta > 20°$ (and their Freidel equivalents) well spread over a large area of reciprocal space were used for the least-squares refinement of the cell and orientation parameters.

Crystals mounted on the Enraf-Nonius CAD-4 were optically centered and 25 reflections were found by the automatic SEARCH routine. Weak reflections were manually discarded and a wide area of reciprocal space was sampled. The reflections were centered using an automatic centering program, SETANG, and the cell and orientation parameters were then calculated.

The density of the crystals used for the X-ray work was determined by flotation of the crystal in the appropriate solvent mixture.

B.3 Data Collection

Data on the Picker FACS-I were collected using graphite monochromated Mo-K_α radiation (λ = 0.7093Å) at a takeoff angle of 4°, and a scintillation detector with pulse height analysis. A symmetrical θ-2θ scanning mode was employed, the scan being from below $K_{\alpha 1}$ in 2θ to above $K_{\alpha 1}$ with a margin dependent on the mosaic spread of the particular crystal and an allowance for dispersion. Stationary-crystal stationary-counter background counts equal to 10 seconds were recorded at both limits of the scan. The appropriate 2θ limit for data collection, the scan width and measurement of standards are noted individually for each compound.

Data on the Enraf-Nonius CAD-4 were collected using graphite monochromated Mo-Kα radiation at a takeoff angle of 4° (for $CpRe(CO)_2(C_2Ph_2)$ (11) a takeoff angle of 3.5° was used). An ω-2θ scanning mode was employed scanning symmetrically above and below the weighted mean for MoKα with allowance made for dispersion corrections. The background was calculated by extending the scan by 25% on each side. A prescan was first conducted by scanning through the reflection at high speed (5°-10°/min) and if the reflection was strong ($\geq$ 25 or 30 $\sigma(I)$) no further measurements were made. If the reflection was weak ($\leq 1.5\sigma(I)$) it was rejected and when the reflections were intermediate they were remeasured at a slower scan speed according to the equation:

$$\mathrm{NPI} = \mathrm{NPIPRE} \times [\sigma(\mathrm{pre})/\sigma]^2$$

where NPI = final scan speed parameter

NPIPRE = prescan speed parameter; scan speed = 16.48/NPIPRE (°/min)

σ(pre) = prescan acceptance parameter

σ = the $\sigma(I)/I$ required for final scan

A minimum speed NPI(max) was determined by the maximum time set for a scan and if the calculated NPI > NPI(max), the final scan was done using NPI(max).

B.4 Structural Determinations

The data were corrected for Lorentz, polarization and absorption errors[155,156] (using either analytical corrections or emperical ψ scans). The heavy atoms were located by directed methods or by solving a three-dimensional Patterson map. Non-hydrogen atoms were located from a series of difference Fourier synthesis interspersed with least-squares refinement. Isotropic thermal parameters were used initially for several cycles and any anisotropic thermal parameters that were introduced had the form

$\exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\}$. All hydrogen atom positions were calculated geometrically with C-H bond lengths and isotropic thermal parameters as noted for each individual structure. The weighting scheme used in the least squares cycles was of the form $w = 1/(\sigma \underline{F}_o^2 + n\underline{F}_o^2)$ and n was determined by minimizing the average $\sum w(|\underline{F}_o| - |\underline{F}_c|)^2$ as a function of $(\sin \theta)/\lambda$, $|\underline{F}_o|$ and Miller indices. The final discrepancy indices, the residual $R = (\sum||\underline{F}_o| - |\underline{F}_c||/\sum|\underline{F}_o|)$, the weighted residual, $R_w = [\sum w(|\underline{F}_o| - |\underline{F}_c|)^2/\sum w|\underline{F}_o|^2]^{1/2}$ and the goodness of fit, $GOF = [\sum w(|\underline{F}_o| - |\underline{F}_c|)^2/(m-n)]^{1/2}$ are quoted for each crystal structure. Atomic scattering factors are taken from reference 157 and included corrections for anomalous dispersion.

APPENDIX C

LISTING OF THE OBSERVED AND CALCULATED STRUCTURE FACTORS

OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR $CpNiCoFe(CO)_5(PPh_3)(C_2Ph_2)$

```
    COLUMNS ARE 10FO,10FC,  10SIG                                               COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG
      -9,    0,  L       18   773   806    32    13   307   308    58         -3,    1,  L        7   475   457    24         -7,    2,  L
  2   692   650    34          0,    0,  L        -8,    1,  L       10  1109  1052    20     8  1162  1099    19     1   521   465    36
  4  1116  1188    28     2  3111  3139     8     2  1408  1298    26    12   511   529    27     9   381   365    33     2   886   964    25
  6   565   538    37     6  2103  1991    14     4  1515  1419    24    13   800   742    25    10   874   895    23     4   810   846    26
 12   988   997    28     8   828   820    18     5   321   283    53    14   469   426    31    11   514   507    28     5   746   677    26
      -8,    0,  L       10   700   686    21     6   544   528    34    16   411   509    40    14   350   339    45     7   574   569    30
 12   461   437    37    14   640   653    28     7   431   449    42    18   292   331    53          3,    1,  L        8   382   260    35
      -7,    0,  L       16   700   674    31    10   507   389    35         -2,    1,  L        1  1291  1257    15    12   462   457    35
  2  1171  1145    24          1,    0,  L       11   352   362    46     1   343   392    18     2   447   444    24         -6,    2,  L
  4  1421  1441    22     0  1054  1215    10    12   914   929    28     2  1658  1539    11     3   553   609    22     2  1352  1411    20
 16   512   423    38     2  1637  1667    10    14   722   792    33     4   975   893    13     4   217   176    35     3  1009  1022    22
      -6,    0,  L        4  2499  2589    13         -7,    1,  L        5  2442  2108    13     5   941  1005    19     4   669   651    23
  2   879   864    23     6   686   680    18     2   599   552    32     6   954   912    16     6   857   896    20     5   760   722    22
  6   535   539    26     8   619   609    21     3   374   369    39    10   968  1001    19     7   958   981    20     8   602   561    25
  8   693   716    26    10  1157  1019    21     5   376   423    41    12  1023  1005    22     8  1425  1426    20     9   418   390    35
 12  1501  1567    22    16   552   556    39     6   442   389    37    16   391   408    44    10   856   790    24    10   662   690    28
 14   451   488    38          2,    0,  L        8   311   257    49         -1,    1,  L             4,    1,  L       11   740   762    25
 16   325   279    55     0  2344  2616    11    10   472   439    38     1   337   333    17     0   651   696    19    12  1149  1122    23
      -5,    0,  L        2  1040  1039    14    12   786   767    30     2  2786  2678     9     1   668   682    19    13   569   590    32
  2   843   806    20     4   723   734    17    14   527   560    32     3  1132  1076    11     2  1128  1110    17    14   296   407    53
  4   532   491    23     6  1568  1636    17         -6,    1,  L        4   788   717    13     4   295   282    34    17   470   380    37
 10  1496  1545    20     8  1458  1500    19     2  1039   969    22     5   561   595    15     7   536   551    26         -5,    2,  L
 12  1804  1818    21    10   658   664    25     4  1079  1070    21     6  1017   899    15     9   647   633    28     1   365   374    33
 16   409   386    43    12   895   906    25     6   531   521    28     7   504   458    19          5,    1,  L        2   213   231    41
 18   888   916    31    14   526   464    38    12   430   479    36     8   900   963    18     0   965  1007    18     7   652   689    23
      -4,    0,  L             3,    0,  L       18   443   438    44    10  1969  1874    18     4   425   466    30     9  1071  1101    20
  2   813   865    17     0  1734  1916    14         -5,    1,  L       12   508   489    26     5   938  1060    23    10   811   825    23
  4   681   654    21     2  1408  1428    16     1   407   438    28    16   411   421    38     6  1044  1063    24    12   674   733    28
  6   583   637    21     4  2944  2953    17     2  1184  1215    18          0,    1,  L        7   737   761    27    13   856   855    26
  8   677   667    20     6   595   611    24     4   608   630    23     2   320   304    17     8  1174  1135    25         -4,    2,  L
 10   386   329    31     8   734   798    23     6   433   386    27     3  1137  1094    12          6,    1,  L        1   369   364    26
 12   440   411    28    10   809   768    24     8   965   975    21     4  1078  1012    13     0   575   597    23     2  1067  1124    17
      -3,    0,  L             4,    0,  L        9   269   294    41     5   809   752    14     2   424   514    32     3   667   654    19
  2  1192  1172    14     0   772   867    17    10   575   549    25     6   767   801    17     3   279   341    42     4   358   367    28
  4   780   821    16     2   343   393    29    11   630   637    26     7   823   799    17     4   395   405    36     5  1058  1019    17
  6  1231  1199    16     4   329   351    26    12   951   864    23     8  1344  1270    18     5   405   430    37     7   333   373    30
  8   484   559    24     6  1641  1682    20    13   536   526    31     9   492   518    26     6   681   750    27     9   866   979    21
 10  1356  1376    19     8  1891  1928    21         -4,    1,  L       10   678   654    22     7   454   415    35    11  1280  1295    20
 12  2202  2102    20    10   983   994    26     2  1213  1139    16    11   563   547    25     8   683   633    32    13   857   848    25
 16   323   292    45          5,    0,  L        4   373   332    29          1,    1,  L             7,    1,  L       18   385   368    46
      -2,    0,  L        2  1316  1444    19     6   369   292    27     0  3533  3800     9     0   450   488    31         -3,    2,  L
  2   653   628    14     4   573   604    25     8   901   915    19     2  1173  1160    12     3   285   211    46     1   315   368    27
  4   627   677    14     6   666   678    26     9   822   781    21     3   821   878    13          8,    1,  L        2  1098  1138    15
 10  1722  1735    18     8   575   575    31    10  2229  2219    19     4  1083  1024    14     0   405   451    41     3   270   228    29
 12   871   845    23          6,    0,  L       12  2283  2267    20     8   353   312    27         -9,    2,  L        5   427   466    23
 14   570   594    29     0   541   595    24    18   634   595    33    12   413   377    32     5   352   320    47     6   379   372    23
 16   477   491    37     6  1004   956    25         -3,    1,  L       14   676   648    33     7   440   459    41     7  1395  1385    16
      -1,    0,  L        8   667   645    33     1   375   385    25    16   605   587    38    12   838   846    32     9  1069  1019    18
  2   991  1015     9          7,    0,  L        2   206   169    33          2,    1,  L            -8,    2,  L       10   689   705    20
  4   904   874    12     0   584   509    27     4   830   738    16     0  1310  1498    11     1   391   337    47    12  1102  1095    21
  6   482   463    21     2   374   426    38     5   811   733    18     1  2000  2050    12     5   540   536    34    13   326   327    40
  8   317   290    27     6   719   697    31     6   629   615    20     3  1740  1713    14    10   387   396    42         -2,    2,  L
 12   662   623    23         -9,    1,  L        7  1998  1918    17     4  1329  1270    16    12   734   779    32     2  1551  1340    12
 14   254   248    46     4   701   737    32     9   735   757    20     6   586   515    20    14   531   540    37     4   881   745    15
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    COLUMNS ARE 10FO,10FC,  10SIG                                              COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
      -2,    2,   L        6  1070  1098    18      6   748   752    27          -2,    3,   L       10   289   256    43      5  1031  1014    20
  5   340   367    23      8   748   795    21      8   809   802    26     11   491   505    25     12   410   496    46      6   449   465    28
  7   673   693    18      9   452   432    28     10   833   823    28     14   398   380    36           4,    3,   L        8   313   294    36
  8   270   288    34     10   760   756    23     12   969   927    26          -1,    3,   L        0  1142  1250    18     11   482   439    29
  9   614   654    21           3,    2,   L       14   764   763    29      1  1189  1136    13      1   263   326    38          -4,    4,   L
 10  1381  1338    19      0   644   681    19          -6,    3,   L        2   614   570    15      2   751   741    22      1  1274  1248    18
 11   763   806    23      1   550   545    21      1   777   802    27      3  1280  1218    13      3   236   199    39      2  1258  1196    18
 12   852   857    22      2   949   968    17      3   986   940    23      4   892   884    16      4   367   436    29      4   700   655    20
 13   855   886    24      4   712   773    21      7   396   431    32      5   647   614    19      6   391   302    31      5   943   861    19
 14   275   247    44      5   533   588    23     11   483   430    32      6   307   371    35      7   762   759    25      7   519   513    25
 18   550   607    37      6   431   433    28     12   483   449    35      7   741   711    19      9   379   389    38      8   616   655    23
      -1,    2,   L        7   502   491    26     13   622   616    32      8   910   910    19           5,    3,   L        9   423   426    31
  1  1485  1559    11      8   804   815    25          -5,    3,   L       10  1121  1062    20      0   772   753    22     10   494   425    30
  2   646   645    12      9   526   511    30      2  1412  1466    18     11   602   612    24      3   553   579    25     16   447   545    41
  3   549   604    14     10   555   575    33      3   774   749    21     12  1187  1140    22      5   489   559    31     18   271   233    56
  4   565   557    16     11   396   317    37      4   815   802    20     14   834   825    26      7   455   421    37          -3,    4,   L
  5  1803  1836    14           4,    2,   L        5  1050  1078    20     16   498   484    34           6,    3,   L        2   924   910    19
  6  1322  1183    15      0   749   697    19      6   615   631    27     17   575   566    35      1   357   388    34      3   770   760    18
  7  1326  1314    16      1   680   664    20      8   541   542    25           0,    3,   L        3   295   281    45      4  1203  1097    17
  8   584   546    20      2   481   485    22      9   589   602    25      1  1180  1191    12      8   542   599    36      5  1292  1238    17
  9   382   418    27      3   576   642    21     10   795   823    23      2   629   656    15      9   427   380    43      6   568   552    22
 10   725   728    22      5  1025  1058    20     11  1108  1094    23      3  2374  2098    13           7,    3,   L        7   727   737    20
 15   621   586    31      6  1015   967    21     12   625   574    30      6  1132  1025    17      4   390   310    43      8   517   490    24
 17   688   711    31      7   937   849    23     16   494   496    37      8  1217  1203    18          -8,    4,   L        9   789   738    22
       0,    2,   L        8  1140  1197    23          -4,    3,   L       10  1115  1175    21      2   407   332    42     10   978   959    22
  0  2534  2592     9     11   363   296    44      2  1210  1254    17     11   866   866    22      3   371   324    41     11   434   458    30
  1   436   421    14           5,    2,   L        3   740   700    20     12   515   525    27      5   325   268    46     14   829   840    26
  2   984   903    12      1   263   346    38      4  1556  1470    17     14   465   439    34      6   514   590    34     17   584   569    36
  3  1020  1018    13      5   498   479    26      7   980   990    19     15   644   662    32     10   352   253    46          -2,    4,   L
  4  1184  1081    14      7   773   749    26      8   425   482    27     17   607   561    34     12   560   570    36      1  1573  1570    15
  5   454   493    21      9   680   694    33      9  1142  1140    20           1,    3,   L       14   535   495    37      2   718   713    18
  6  1150  1069    16           6,    2,   L       10   590   544    25      0   593   564    15          -7,    4,   L        3  1207  1156    16
  8  2000  1948    17      3   324   429    45     11   299   197    37      2   906   848    23      9   398   352    40      4   363   354    30
  9   737   764    20      5   541   563    32     13   839   807    25      3   302   307    26     11   799   890    28      5   386   350    29
 10  1332  1384    20           7,    2,   L       14   332   271    49      4  1136  1085    22     12   530   549    37      6   472   541    23
 11   664   701    23      0   316   253    36          -3,    3,   L        6   311   385    30     13   512   514    35      7  1375  1260    18
 12   871   830    23      6   693   602    31      1  1631  1545    16      7   413   411    26          -6,    4,   L        8   365   382    29
 13   621   651    28          -9,    3,   L        4   888   908    17      9   561   609    24      1   809   781    24      9   476   471    25
 17   396   371    45      2   474   411    41      6   694   613    20           2,    3,   L        2   884   887    25     10   283   244    38
       1,    2,   L        3   426   349    42      9   208   297    45      0  1355  1499    14      4  1020  1024    22     11   915   868    22
  0  1407  1649    11      4   521   574    38     11  1657  1642    20      1  1127  1160    16      5   686   719    24     12   577   643    29
  1  2087  2058    11          -8,    3,   L       13  1065  1021    22      2   948   924    18      6   509   482    29     13   420   484    41
  2   514   497    15      4   492   490    35     14   677   665    28      3   398   388    24      8   639   649    27     14   813   796    28
  7   779   758    19      5   582   628    35     16   662   653    30      4  1109  1088    19      9   684   744    28     16   574   591    32
  8  1529  1492    18      7   448   452    43     18   445   501    42      5   432   418    25     10   980  1008    25     17   485   523    43
  9   423   407    26     10   541   553    34          -2,    3,   L        6   735   732    20     12  1061  1131    26          -1,    4,   L
 10  1170  1072    21     12   922   847    29      1  1244  1118    14      7   935   858    21     13   464   452    39      3   413   464    24
 13   488   493    33     14   778   819    30      2  1975  1880    14      8  1696  1724    20     14   439   467    42      4  1374  1184    16
 15   751   703    30          -7,    3,   L        3   668   662    16     10  1150  1182    22     15   542   600    36      5  1178  1087    18
       2,    2,   L        1   310   193    47      4   412   399    23     15   472   377    39          -5,    4,   L        7   464   434    26
  1   354   401    21      2   641   690    32      5  1141  1082    16           3,    3,   L        1   982   936    20      8   256   239    33
  2   348   358    24      3   860   793    26      6   464   482    23      1   925   966    17      2   624   642    24      9   578   583    26
  4  1170  1155    16      4   700   627    28      7  1105  1119    17      5   485   474    24      3  1864  1748    19     10   620   595    24
  5   495   446    21      5  1024   948    24      9   825   928    20      8   672   716    24      4   725   786    23     11  1023  1091    22
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO   FC   SIG
     -1,   4,  L
12   543   561   28
13   372   380   38
14   431   508   39
      0,   4,  L
 0  1226  1173   14
 1   865   853   16
 3   721   735   18
 4   554   583   21
 5   945   860   18
 7  1337  1300   18
 8   614   669   22
 9   963   982   21
10   504   544   26
12   802   821   25
16   492   486   41
      1,   4,  L
 0  1197  1235   16
 1  1188  1210   15
 2   750   837   18
 3   533   539   22
 4   867   857   18
 5   644   639   21
 6   700   739   21
 7   425   396   27
 8   862   813   22
10  1111  1039   22
11   970   947   24
12   458   504   37
13   469   376   34
      2,   4,  L
 1   956  1010   17
 3   674   631   20
 4   274   282   35
 7   832   742   23
 9  1229  1218   22
11   550   604   32
      3,   4,  L
 0   568   613   22
 1   611   647   21
 2   533   486   21
 5   721   766   22
 6   712   637   23
 7   872   918   24
 8   708   713   26
 9   497   499   34
11   373   406   40
      4,   4,  L
 0   750   760   21
 1   760   795   20
 2   476   530   28
 5   410   373   33
 6   346   242   40
11   483   493   39

 L   KFO   FC   SIG
      5,   4,  L
 0   392   453   31
 4   254   318   45
 8   477   508   39
      6,   4,  L
 1   488   540   30
      7,   4,  L
 3   726   760   31
 5   673   727   35
 6   356   260   54
      8,   4,  L
 1   391   458   45
 2   499   510   40
     -9,   5,  L
 7   487   419   39
     -8,   5,  L
 5   375   439   46
 7   323   294   53
 9   554   621   41
10   291   272   62
11   778   790   31
12   413   386   50
13   558   549   42
     -7,   5,  L
 4   519   492   31
 5   563   579   33
 6   622   538   29
12   484   438   38
13   565   627   37
15   851   855   30
     -6,   5,  L
 1   880   827   24
 3   905   912   23
 4   388   359   35
 5   432   451   31
 7   381   421   45
 9   674   683   28
11   973   956   26
13   786   794   31
14   383   424   44
     -5,   5,  L
 1  1026  1060   21
 2   765   769   22
 4   527   501   27
 5   462   454   28
 8   345   402   38
 9   498   466   29
10   575   561   28
12   340   246   40
13   493   461   34
15   587   577   33
     -4,   5,  L
 1   756   801   22
 2   280   261   35

 L   KFO   FC   SIG
 3  1145  1179   19
 4   611   626   26
 5   865   819   20
 7   268   354   42
 9   555   544   26
10   479   454   30
12   614   603   27
13   507   547   34
17   480   485   45
     -3,   5,  L
 1  1582  1589   17
 2   809   838   19
 3   654   664   20
 4   318   281   29
 5   360   361   29
 6   492   471   23
 8   247   258   43
 9   270   259   39
10   429   341   30
11   484   467   30
12   518   445   28
13   447   489   35
17   466   438   40
     -2,   5,  L
 2   381   364   26
 3  2025  2006   16
 4   328   390   29
 5   915   842   18
 8   570   519   24
 9  1180  1125   20
10   311   345   39
11   820   817   24
17   545   587   37
     -1,   5,  L
 2  1184  1228   17
 3   266   306   37
 4  1007   970   19
 5   762   784   20
 6   635   661   21
 7  1298  1241   19
 8   498   478   26
 9  1785  1767   20
10   517   529   28
11   365   365   38
14   368   365   43
15   428   423   40
      0,   5,  L
 2   938   955   18
 3   320   327   33
 4   630   625   22
 5   837   861   20
 9   557   504   25
11   846   815   26
13   567   553   31
```

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COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO   FC   SIG
      1,   5,  L
 0   264   293   36
 7  1388  1436   19
 9  1437  1485   21
12   522   479   34
      2,   5,  L
 0   684   723   19
 1   516   721   25
 4   243   264   41
 6   454   490   27
 8   525   563   28
10   546   523   35
11   790   855   26
12   476   482   38
13   620   663   34
      3,   5,  L
 0   651   716   22
 1  1246  1358   19
 3   868   791   22
 7   378   389   35
 9   890   827   27
10   259   243   56
13   325   424   57
      4,   5,  L
 6   255   303   53
 7   482   415   35
      5,   5,  L
 0   518   496   27
 1   996  1018   23
 6   380   488   42
 7   309   364   51
      6,   5,  L
 0   279   275   50
 1   405   472   42
 5   514   509   40
 6   368   424   55
      7,   5,  L
 1   397   345   41
     -8,   6,  L
 2   377   305   46
10   532   469   37
     -7,   6,  L
 1   736   715   29
 2   456   460   39
 4   379   313   41
 8   512   506   37
 9   581   615   34
10   440   411   41
11   755   767   30
13   535   542   38
14   455   416   41
     -6,   6,  L
 1   784   806   27
 6   491   465   32

 L   KFO   FC   SIG
10   275   316   55
     -5,   6,  L
 1   923   879   24
 3   889   853   24
 5   548   552   31
 6   312   307   41
 7   411   434   34
 9   523   467   29
10   437   415   37
12   765   759   28
14   921   979   28
16   638   641   35
     -4,   6,  L
 1   475   451   27
 4   595   592   24
 9   530   519   27
11   283   345   47
14   429   444   38
     -3,   6,  L
 1   339   396   33
 3  1237  1264   19
 4   312   354   39
 5  1525  1528   20
 6   820   818   22
 7   414   414   34
 8   625   652   26
 9   807   750   23
10   423   467   34
     -2,   6,  L
 1   757   781   22
 2   956  1003   19
 3   934   933   19
 4  1715  1706   19
 6   409   425   28
 7   785   804   23
10   438   460   34
12   403   462   37
13   368   390   41
15   294   221   51
     -1,   6,  L
 3   456   403   28
 4   216    72   41
 5   667   682   22
 6   841   842   22
 7   407   405   30
 8   434   435   29
 9   762   783   26
10   332   271   43
11   425   467   35
      0,   6,  L
 0  2105  1931   17
 2  1076  1071   18
 4   803   878   21
 5   532   506   24

 L   KFO   FC   SIG
 6   871   847   21
 7  1595  1527   20
 8   581   541   25
 9  1173  1099   23
      1,   6,  L
 0   460   438   26
 1   887   932   20
 2   410   413   28
 8   649   707   26
 9   620   521   26
11   741   754   30
12   428   412   46
13   686   648   36
      2,   6,  L
 1   733   793   21
 2   562   526   25
 3   374   407   33
 5   540   574   28
 7   776   801   26
 9   863   855   27
10   815   856   28
11   499   461   42
12   713   701   33
      3,   6,  L
 1   288   201   34
 4   492   490   30
 7   320   330   43
      4,   6,  L
 0   425   402   32
 1   941   981   22
 2   280   243   44
 8   631   732   37
10   486   425   43
      5,   6,  L
 0   810   896   25
 1   457   510   33
 2   485   545   33
      6,   6,  L
 1   547   517   34
 4   732   776   34
 6   504   526   42
      7,   6,  L
 0   701   619   33
 3   580   589   36
     -8,   7,  L
 4   442   385   42
 5   324   229   53
 8   571   491   36
     -7,   7,  L
 5   472   467   36
     -6,   7,  L
 1   515   478   35
 2   652   575   30
 5   356   362   43
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        COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
      -6,   7,  L        10   284   197   50     12   618   609   36
 12   500   535   36     12   618   637   35     13   680   742   33
 13   277   271   55            1,   7,  L            -3,   8,  L
 14   660   654   39      0  1103  1134   20      4   376   340   37
      -5,   7,  L         3   356   409   35      5   502   487   33
  4   493   508   31      4   309   357   41      6  1074  1065   25
  9   503   532   35      5   348   358   36      7   844   885   27
 10   281   133   52      6   980   994   25      8   661   600   31
 14   571   614   37     12  1042  1040   30      9   572   571   34
      -4,   7,  L               2,   7,  L        10   429   453   45
  1   307   355   38      1   671   758   25           -2,   8,  L
  2   346   389   38      4   331   282   35      2   867   888   24
  3   910   847   22      7   472   402   32      3   598   540   28
  5   321   284   36            3,   7,  L         4  1282  1215   22
  6   336   371   43      1   623   715   26      5   951   940   23
 10   476   457   34      2   623   717   28     11   624   608   36
 12   570   573   38      4   459   441   35     12   391   398   50
 14   408   421   46      8   646   647   32     13   766   721   51
      -3,   7,  L        10   893   786   31           -1,   8,  L
  1   424   405   29            4,   7,  L         1   478   565   32
  2   964   882   22      0   666   704   28      2   620   615   25
  3   369   337   31      2   560   599   32      3   902   949   24
  4  1504  1493   20      5   445   543   40      4   307   313   43
  5   635   624   26      7   542   497   40      5   884   839   25
  6   884   911   23            5,   7,  L         6   737   753   27
  8   531   486   30      1   409   403   40      7   874   861   26
  9   432   523   33      2   433   335   40      8   824   822   27
 11   710   732   29      4   387   447   49      9   502   452   36
 12   681   653   31      6   536   550   39     12   507   543   37
 13   754   713   30            6,   7,  L             0,   8,  L
 14   318   325   54      0  1048  1105   26      0   481   537   33
      -2,   7,  L         1   577   639   36      2   426   437   32
  1   413   417   27      2   850   792   31      3   456   398   34
  3   576   416   26            7,   7,  L         4   585   647   30
  5   642   586   26      1   331   296   51      6  1039  1056   25
  6  1314  1274   22           -7,   8,  L              1,   8,  L
  7   581   619   27      2   484   483   39      0   536   536   29
  8  1363  1325   22      8   565   607   40      1   669   648   28
  9   971   956   26     10   566   450   34      3   630   690   28
 10   468   520   34           -6,   8,  L         8   580   558   35
 11   321   301   49      1   573   600   34            2,   8,  L
      -1,   7,  L         3   631   662   32      0   437   399   33
  2  1463  1480   19      5   648   615   28      1   446   554   32
  3   880   862   21      8   388   359   42      2   422   431   35
  4   872   905   23           -5,   8,  L         3   426   369   36
  6   892   892   24      1   395   390   41     10   949   816   29
  7   588   615   28      4   508   519   33            3,   8,  L
  9   585   615   29      6   521   506   35      0   486   480   37
       0,   7,  L             -4,   8,  L         1   586   579   30
  1   897   860   21      2   372   432   38      4   419   451   39
  3   309   302   35      4   765   771   27      5   756   816   31
  5   527   530   29      7   826   724   28      6   431   359   38
  6   553   642   31      9   881   854   28      7   485   441   45
  8   983   986   23     11   709   687   32      9   483   470   42
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        COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
       3,   8,  L         2   603   580   29      3   509   541   43
 10   475   524   48      3   804   822   30      4   594   523   37
       4,   8,  L         4   798   768   47            2,  10,  L
  2   609   679   31      5   644   707   33      0   703   707   33
  4   578   582   37      6   375   283   44      2   615   608   37
  5   514   506   43     12   415   401   53      3   624   645   35
  7   525   508   40            1,   9,  L         5   589   639   80
       5,   8,  L         0   300   351   53      6   352   376   57
  0   739   857   32      6   501   558   40      8   519   489   45
  1   439   426   45            2,   9,  L              3,  10,  L
  2   501   479   37      1   640   689   31      4   616   645   39
  3   646   632   37      3   407   407   44      6   388   330   53
  5   394   360   51      4   801   825   30            4,  10,  L
  6   330   299   67      9   530   353   42      0   511   461   48
       6,   8,  L               3,   9,  L         2   449   577   45
  1   391   417   49      0   612   707   34            5,  10,  L
  4   729   809   35      1   353   287   48      1   413   438   55
       7,   8,  L         2   516   566   39           -4,  11,  L
  0   697   664   38            4,   9,  L         3   379   393   48
      -6,   9,  L         1   381   355   51      4   392   366   51
  4   441   487   40      3   645   616   33      5   459   514   41
  7   525   532   36      4   503   604   45           -3,  11,  L
 10   558   540   34      5   577   661   39      5   673   704   33
      -5,   9,  L         6   435   385   44      7   382   494   51
  8   611   575   35      7   354   400   52      8   524   473   40
      -4,   9,  L               6,   9,  L             -2,  11,  L
  4   581   608   33      1   376   321   44      5   468   511   43
  6   661   675   31           -5,  10,  L         7   687   610   33
  7   427   416   41      1   383   386   43           -1,  11,  L
  9   748   716   31      4   595   553   35      2   469   575   43
 10   535   527   38      5   621   555   36            0,  11,  L
      -3,   9,  L         7   442   405   42      4   523   497   39
  4   496   477   33      8   310   254   54      5   867   832   32
  5   750   726   30           -4,  10,  L              1,  11,  L
  7  1030   964   26      5   478   420   39      0   617   499   35
  8   900   943   28      6   665   624   33      2   357   431   51
  9   537   540   34      8   659   597   34      3   779   780   59
 11   810   819   31           -3,  10,  L         5  1008   869   47
 13   823   870   32      7   771   712   32      7   548   452   43
      -2,   9,  L              -2,  10,  L              2,  11,  L
  1   579   574   35      5   762   763   31      4   599   661   39
  3   691   597   27      7   383   391   45            3,  11,  L
  5  1064  1076   25           -1,  10,  L         1   475   439   45
  6   552   590   34      2   584   514   31      3   861   846   59
  7   864   870   29      3   421   422   44           -2,  12,  L
  8   522   567   34      4   933   944   27      5   427   411   45
 12   531   510   38      5   826   851   31           -1,  12,  L
      -1,   9,  L         7   576   546   38      5   729   741   37
  3   323   322   43     10   479   479   43            0,  12,  L
  4   478   485   38            0,  10,  L         3   583   553   35
  5   545   520   32      0   824   793   28      5   533   570   41
  7   545   471   36            1,  10,  L              1,  12,  L
       0,   9,  L         1   436   418   40      1   465   405   43
  1   753   756   28      2   727   707   31            2,  12,  L

                                                               PAGE 9

        COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
       2,  12,  L         1   538   497   41
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OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR $CpNiCoRu(CO)_6(C_2Ph_2)$

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                                                                                                                                              PAGE 2
 COLUMNS ARE 10FO,10FC,  10SIG                                                     COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG
      H,  0,  0                H,  7,  0            2  1487  1447     6          H,  7,  1            4  1790  1854     7     -2   558   578     9
  1   691   704     3      1   486   488    12      3  1533  1533     7      1   645   638    11      5   517   529     9     -1   230   229    12
  2   631   623     3      2   505   486    12      4   132   143    20      3   388   398    14      6   559   560    10      0  1060  1030     8
  3  2282  2315     6      3   371   383    14      6   773   769    10      4   185   211    25      8   645   640    12      1   500   533     9
  4  1253  1359     7      4   398   414    14      8   215   206    20      5   241   240    18          H,  2,  2            2   381   368    10
  5  1473  1528     7          H,  8,  0           10   310   272    19      6   503   469    15    -10   373   381    17      3   175   200    19
  6  1064  1133     8      0   742   715    11          H,  4,  1            7   504   502    14     -9   351   360    20      4   585   613    10
  7   349   361    14      2   422   421    13     -8   432   418    15          H,  8,  1           -8   776   801    11      5   407   398    13
  9   486   467    13      3   454   421    13     -7   365   379    15     -6   446   391    15     -7   329   351    14      6   294   289    16
      H,  1,  0            4   156   192    25     -6   473   493    11     -4   231   250    20     -5   888   895     8          H,  6,  2
  1  2577  2504     4      5   219   183    22     -5   717   687    10     -2   804   792    11     -4   324   336     9     -1   463   459    10
  2   122   150    12      6   172   151    25     -4  1363  1340     8      1   393   377    13     -3   246   226    11      1   501   490    11
  4   452   473     8          H,  9,  0           -3   491   489     9      2   478   466    14     -2   297   287    10      6   185   189    26
  5  1313  1315     8      1   195   210    23     -2  1985  1921     7      3   347   302    15      0   328   388     7      7   271   208    17
  8   423   407    13      3   171   169    28     -1   720   717     7      5   218   217    23      1   461   452     6          H,  7,  2
  9   503   484    14          H, 10,  0            0   412   430     8          H,  9,  1            2   925   880     6     -5   273   272    18
      H,  2,  0            0   248   218    21      1  1466  1471     7     -3   592   596    13      3  1276  1266     6     -4   294   275    17
  0  2890  2787     5          H,  1,  1            2  1084  1097     7     -1   431   394    15      4  1122  1125     7     -3   401   402    13
  1   428   403     6     -8   404   418    14      3  1221  1183     8      0   307   303    17      5   823   839     8     -2   660   656    11
  2   454   454     7     -6   316   317    13      4  1039  1041     8      1   181   171    24      6   344   344    12      0   383   361    12
  3   543   497     7     -5   815   841     8      5   254   250    15      2   396   390    14      7   786   816    10      1   440   420    12
  4  1281  1274     7     -4   566   535     7      7   436   437    13      3   353   324    17      9   876   867    12      3   440   441    13
  7   678   657    11     -3   436   437     7          H,  5,  1                H, 10,  1                H,  3,  2                H,  8,  2
  8   839   858    11     -2   417   424     6     -7   694   676    12     -2   292   303    20    -10   225   236    24     -6   304   302    22
  9   328   293    17     -1   765   744     4     -5   336   325    13      2   207   209    25     -9   415   427    15     -4   376   343    15
 10   307   348    18      0   679   646     3     -4   356   351    12          H,  0,  2           -8   416   422    14     -3   361   380    15
      H,  3,  0            1   929   913     4     -3   945   917     9    -10   469   478    15     -7   480   513    13     -1   773   786    11
  1   958   928     6      3   276   281     9     -2   369   383    10     -9   263   260    18     -6   207   209    18      0   359   372    15
  2   389   367     8      4   309   293     9     -1  1000   966     8     -8   460   458    14     -5   260   283    14      1   220   211    19
  3   956   935     7      5   453   439     9      0   480   506     9     -6  1331  1350     8     -4   555   575     9      2   389   394    14
  4   632   591     8      6   344   357    12      1   862   846     8     -5   932   921     8     -2   375   358     8      5   276   290    19
  5   853   846     8      7   456   435    12      2   643   637     9     -4  2102  2044     7      0  1259  1258     6          H,  9,  2
  6   299   308    14      9   262   249    20      3   975   952     8     -3  2029  1954     6      3  1174  1133     7     -2   215   271    22
  7   424   401    13     10   336   315    18      4   144    82    22     -2   141   157    10      4  1046  1048     8      2   196   160    23
  8   416   388    14          H,  2,  1            6   789   759    11     -1  3325  3270     3      5   509   506    10      4   325   298    18
  9   361   381    14    -10   347   347    17      7   370   365    14      1  2326  2194     3      8   733   741    11      5   205   199    23
      H,  4,  0           -9   250   254    20          H,  6,  1            2  1547  1472     5     10   286   287    19          H,  1,  3
  0   630   645     7     -6   736   745     9     -8   282   236    20      3  2057  2028     6          H,  4,  2           -9   261   265    19
  2   655   630     8     -5   299   319    12     -6   742   760    12      4   223   179    13     -6   463   502    12     -8   299   308    17
  4   852   828     9     -2   240   233     9     -3   339   351    13      5  2036  1965     7     -5   180   177    20     -6   532   552    10
  5   239   202    14     -1   740   704     5     -2   682   691    10      7   232   239    17     -4   686   716     9     -3   725   698     7
  6   371   371    13      0   385   357     6     -1   403   393    11      9   507   516    13     -2   390   356     9     -1   305   490     5
  8   319   294    17      1   508   499     6      0   263   264    13          H,  1,  2           -1  1245  1224     7      0   323   517     4
  9   219   170    24      3   271   267    10      2   777   774    09     -9   558   591    13      0   655   665     7      1   853   833     4
      H,  5,  0            5   432   430     9      5   576   554    12     -7   270   274    16      1   660   670     7      3   135    97    17
  1  1158  1126     8      6   441   436    11      6   568   540    13     -5  1472  1499     7      2   165   124    15      5   267   255    12
  2   377   376    10          H,  3,  1            7   390   399    15     -4   446   452     8      3   695   754     8      6   681   702     9
  3   417   387    10     -7   397   402    13      8   328   311    17     -3   734   776     6      4   371   387    10      9   214   229    26
  4   697   701    10     -6   567   563    11          H,  7,  1           -2  1730  1749     5      5   388   402    11          H,  2,  3
  5   416   440    12     -5   341   333    11     -7   553   514    14     -1   956   978     4      9   375   381    17    -10   229   235    23
  6   321   307    15     -3  2032  1990     7     -5   325   308    15      0  2783  2965     4          H,  5,  2           -7   475   493    12
      H,  6,  0           -1  1302  1255     6     -3   493   496    12      1  1116  1147     4     -5   957   966    10     -6   259   251    15
  2   203   186    17      0  1416  1397     6     -2   378   386    13      2  1646  1675     5     -4   273   270    14     -5   705   709     9
  7   243   265    19      1  1417  1346     6     -1   240   238    17      3   639   635     6     -3   440   441    10     -2  1207  1172     6
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COLUMNS ARE 10FO,10FC,  10SIG

   H    KFO     FC   SIG        H    KFO     FC   SIG        H    KFO     FC   SIG
      H,  2,  3                 3    120     92    25       -2    967    893     5
  -1    625    606     6        5    328    348    14       -1   2845   2740     4
   0    421    429     6        6    821    811    11        0    687    634     4
   1   1157   1173     5           H,  6,  3                 1   1985   1873     4
   2   1777   1763     6       -7    588    565    13        2   1149   1094     5
   4    408    418     9       -6    401    374    14        3   1811   1746     6
   5    777    782     8       -5    233    221    19        4   1301   1227     7
   6    319    311    14       -3    418    422    12        5    563    563     9
   7    609    634    11       -2    138    106    22        7    393    389    13
  10    282    261    21       -1    241    257    16        8    303    272    17
      H,  3,  3                 0    240    246    15           H,  1,  4
  -9    229    240    23        1   1084   1075     9      -10    415    408    16
  -8    363    381    16        2    195    209    18       -8    327    301    15
  -7    809    784    10        3    546    530    11       -6   1120   1157     9
  -6    145    153    30        4    468    471    11       -4   1456   1437     7
  -5    274    288    13        5    905    887    11       -3   1155   1147     6
  -4    844    838     8        7    560    560    13       -2    462    389     6
  -3    419    430     8        8    191     85    26       -1    222    191     7
  -2    697    712     7           H,  7,  3                 0   2068   2091     4
  -1    858    853     6       -7    207    176    23        1    776    702     5
   0   1343   1351     6       -6    414    418    14        2    223    203     9
   1    418    386     8       -4    418    425    13        3   1452   1409     6
   2   1477   1470     6       -3    245    207    18        4   1569   1538     7
   4    266    239    11       -2    436    426    13        5    524    504    10
   5    352    382    12        0    550    551    11        7    479    464    12
   6    662    670    10        1    327    295    14        8    379    376    14
  10    223    234    24        2    747    732    11        9    543    530    13
      H,  4,  3                 3    308    246    16           H,  2,  4
  -8    391    396    16        6    660    645    12       -9    554    570    13
  -7    374    319    15           H,  8,  3                -7    514    495    12
  -6    946    944    10       -6    291    258    18       -5   1085   1119     8
  -5    617    629    10       -3    637    631    12       -4    403    402     9
  -4    141    148    21       -2    303    260    16       -3    316    312     9
  -3   1538   1551     8        1    559    521    12       -2    940    900     6
  -2    828    845     8        3    418    393    14       -1   1277   1302     6
  -1    725    751     7        5    324    353    18        0    445    445     6
   0    900    923     7           H,  9,  3                 1    421    424     7
   1   1877   1858     7       -5    194    155    25        2    530    543     7
   2    261    283    12       -4    448    463    15        3   1780   1860     6
   3   1384   1426     8       -3    209    154    25        4    716    730     8
   6    378    399    13       -2    600    598    13        5    877    894     8
   7    333    344    15       -1    318    318    16        6    341    560    10
   9    264    273    20        1    361    375    16        7    248    251    17
      H,  5,  3                 2    574    557    13        8    819    836    11
  -8    312    327    17           H, 10,  3                 9    332    344    17
  -7    516    534    14        1    279    255    19           H,  3,  4
  -5    458    461    12           H,  0,  4               -10    380    396    18
  -4    680    664    10      -10    526    501    14       -8    335    378    17
  -3    537    555    10       -9    287    267    18       -4    905    895     8
  -2    851    878     8       -8    331    343    16       -3    620    606     8
  -1    549    571     9       -7    764    731    10       -2    473    508     8
   0    876    864     8       -6    758    754     9       -1    225    220    12
   1    155    131    18       -5   2574   2516     7        0   1247   1293     6
   2   1411   1431     8       -4    905    873     7        1    271    278     9
```

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COLUMNS ARE 10FO,10FC,  10SIG

   H    KFO     FC   SIG        H    KFO     FC   SIG        H    KFO     FC   SIG
      H,  3,  4                -4    393    426    14        3    135    112    19
   2    438    453     8       -2    526    493    13        4    379    376    10
   3    627    638     8       -1    433    427    13        5    584    600    10
   4   1311   1336     8        1    374    316    14        6    290    286    15
   7    525    556    12        4    238    260    19        9    408    422    16
   9    502    496    14           H,  9,  4                    H,  4,  5
      H,  4,  4                 0    368    373    15       -9    508    496    14
  -9    198    143    26        1    358    344    17       -7    785    830    11
  -6    163    149    24        3    209    222    24       -6    447    472    13
  -5    753    762    10        4    241    236    21       -4    772    807     9
  -4    172    172    17           H, 10,  4                -3   1568   1585     8
  -1    898    925     7        0    231    244    22       -2    427    462     9
   0    211    193    12           H,  1,  5                -1   1091   1098     7
   1    468    486     9      -10    220    232    24        0   1241   1255     7
   3    247    244    13       -7    442    428    14        1    283    260    10
   4    569    583     9       -6    289    307    14        2   1129   1147     7
   5    233    231    16       -5    405    399    10        3    225    262    15
      H,  5,  4                -3    238    219    10        6    322    340    16
  -9    227    251    23       -2    218    212    10           H,  5,  5
  -8    164    182    27       -1    173    155    10       -8    643    619    13
  -6    503    516    13        0   1027    978     5       -6    783    791    11
  -5    375    358    13        1    136    124    12       -4    690    716    10
  -4    734    708    10        2    732    719     6       -3    397    415    11
  -3    374    388    11        3    398    397     8       -2   1000   1015     8
  -2    639    638     9        5    774    733     8       -1    339    330    11
  -1    547    551     9        6    260    278    15        0    256    246    12
   0    838    831     8        7    440    422    13        1   1302   1316     8
   1    312    330    12        8    390    360    15        2    611    620     9
   2    375    354    11       10    233    214    25        4    188    183    19
   4    196    214    18           H,  2,  5                 5    847    842    10
   5    514    472    11       -8    393    384    14        6    291    285    17
   6    243    222    20       -7    254    259    16        7    248    225    19
   7    239    229    20       -5    338    350    11        9    387    394    17
      H,  6,  4                -3    310    328     9           H,  6,  5
  -7    171    205    25       -2    538    531     7       -7    589    604    13
  -5    183    175    22       -1    175    169    13       -3    692    686    10
   0    442    449    11        0    112    100    17       -1    187    181    18
   1    664    666    10        1    268    298     9        0   1035   1039     9
   2    301    273    14        2    425    441     8        1   1072   1086     9
   3    194    197    21        3    951    991     7        2    517    535    11
   4    223    220    18        4    874    908     8        3    566    576    11
   6    232    237    23        6    856    859    10        4    755    749    11
      H,  7,  4                 7    357    398    14        6    778    766    12
  -6    232    190    19        9    223    203    22        7    276    295    20
  -5    265    306    18       10    298    278    20           H,  7,  5
  -4    293    317    17           H,  3,  5                -6    293    281    18
  -3    585    551    12       -8    467    505    13       -4    322    301    16
  -1    489    482    12       -6    795    809    10       -2    428    421    12
   0    511    510    12       -4    751    786     8       -1    233    231    19
   1    211    194    19       -3    425    456     9        0    529    503    12
   2    268    260    17       -2   1071   1114     7        1    554    529    11
   3    273    291    16        0    513    512     7        2    637    600    11
      H,  8,  4                 1    772    787     7        5    623    591    12
  -5    416    412    15        2    229    244    11        6    360    376    16
```

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COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO    FC   SIG
      H,  7,  5
  7   290   243   17
      H,  8,  5
 -3   575   580   12
 -1   335   328   15
  0   455   455   13
  1   367   376   15
  2   403   438   14
  6   292   246   20
      H,  9,  5
 -4   296   258   19
 -3   283   251   18
 -2   518   515   14
  1   456   417   14
  3   290   309   21
      H, 10,  5
  0   188   174   26
      H,  0,  6
 -9   515   508   13
 -8   378   365   15
 -7   261   310   18
 -6  1076  1061    9
 -5  1640  1602    8
 -4  1051  1012    7
 -3  1205  1146    6
 -2  1403  1394    6
 -1   138   136   14
  0  3238  3063    5
  1  1490  1300    5
  2   572   526    7
  3   261   251   10
  4   893   893    7
  5   455   452    9
  6   532   531   10
  7   322   295   15
  8   535   526   13
 10   355   315   17
      H,  1,  6
-10   562   559   15
 -8   258   240   18
 -7   447   445   13
 -6  1165  1173    9
 -5   931   936    8
 -4   484   507    8
 -3   461   493    8
 -2   343   334    8
 -1  2247  2188    5
  0  1392  1389    5
  1   674   651    6
  2   349   324    8
  3  1151  1166    7
  4   205   191   13
  5   398   570    9
  6   465   414   11

  H   KFO    FC   SIG
  7   814   790   11
  9   433   408   15
      H,  2,  6
 -9   319   315   17
 -8   254   309   20
 -5   347   556    9
 -4   661   657    8
 -3   527   551    8
 -2  1100  1094    6
 -1  1146  1208    6
  0   394   419    7
  1   262   264    9
  2  1221  1205    6
  3   196   200   12
  4  1655  1715    7
  5   501   502   10
  6   313   336   15
  7   519   519   12
  8   709   703   12
  9   308   319   19
      H,  3,  6
 -9   348   329   17
 -8   193   219   24
 -6   333   353   13
 -5   665   682   10
 -4   237   254   12
 -3   253   265   12
 -2   977   980    7
 -1   699   724    7
  0   166   225   13
  1   242   238   10
  2   189   162   13
  3  1406  1489    7
  4   294   323   12
  5   527   543   11
  6   528   549   12
  7   448   465   13
  8   233   261   22
  9   379   394   16
      H,  4,  6
 -9   240   249   23
 -7   325   334   17
 -6   301   293   17
 -5   388   412   13
 -4   305   300   13
 -2   769   770    8
 -1   208   198   12
  0   282   294   11
  1   214   220   13
  4   691   708    9
      H,  5,  6
 -6   230   253   20
 -5   462   448   12
 -4   543   594   11

  H   KFO    FC   SIG
 -1   506   528   10
  0   326   328   11
  1   678   679    9
  4   493   499   11
  6   452   458   13
      H,  6,  6
  0   302   296   13
  1   209   197   18
  2   240   252   17
  7   233   273   20
      H,  7,  6
 -7   301   280   18
 -5   289   294   17
 -4   376   365   14
 -2   475   488   13
 -1   349   338   15
  2   302   296   15
  3   196   250   22
      H,  8,  6
 -6   368   362   17
 -5   426   439   16
 -3   329   281   16
 -2   312   338   16
  0   447   450   14
  1   329   339   16
      H,  9,  6
 -4   281   296   20
  0   354   316   17
  3   453   410   14
      H,  1,  7
 -8   273   279   18
 -4   562   528    8
 -3   698   671    7
 -1   178   186   11
  0   309   333    8
  1  1152  1089    6
  3   673   629    7
  4   575   583    8
  5   497   521   10
  6   474   491   11
  7   438   445   12
  9   266   265   21
 10   225   239   24
      H,  2,  7
 -8   292   310   18
 -5   313   301   13
 -4   648   635    8
 -3   546   577    8
 -2   164   153   14
 -1   755   765    7
  0   925   917    6
  1   330   301    9
  2   804   806    7
  3   965   963    7
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COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO    FC   SIG
      H,  2,  7
  4   543   560    9
  5   246   254   15
  6   745   758   10
  9   440   417   15
      H,  3,  7
 -9   330   339   17
 -7   390   407   15
 -6   429   439   13
 -5   308   319   14
 -4   268   279   12
 -3  1213  1219    7
 -2   138   111   17
 -1   786   798    7
  0   199   194   12
  1   503   540    8
  2   861   873    8
  3   283   268   11
  5   571   601   10
  9   229   222   21
      H,  4,  7
 -9   233   232   22
 -8   743   739   12
 -7   425   408   14
 -5   525   506   11
 -4  1526  1552    9
 -3   210   208   15
 -2  1467  1479    8
 -1   842   868    8
  0   269   300   12
  1   352   361   10
  2   527   531    9
  4   407   406   11
  5   238   239   19
  6   432   432   13
  9   312   323   19
      H,  5,  7
 -7   530   559   13
 -5   510   553   13
 -3  1012  1014    9
 -2   524   527   10
  0   283   287   12
  1  1202  1245    8
  3   324   321   13
  4   562   560   11
  5   720   701   11
  6   225   217   20
  7   362   400   15
  8   190   190   31
      H,  6,  7
 -8   394   396   16
 -4   743   734   11
 -3   473   469   12
 -2   430   411   11

  H   KFO    FC   SIG
 -1   343   364   14
  0  1058  1035    9
  2   985  1032    9
  3   583   540   12
  4   265   270   18
  5   761   757   11
  6   811   821   12
      H,  7,  7
 -7   328   310   17
 -5   204   226   22
 -3   608   614   12
 -1   265   275   16
  1  1038  1052   10
  2   304   309   16
  4   324   332   16
  5   594   567   13
  6   257   283   22
  7   332   384   18
      H,  8,  7
 -4   478   460   14
 -3   170   144   25
 -2   567   554   12
  0   339   354   15
  2   341   356   15
  4   211   220   26
  6   322   307   18
      H,  9,  7
 -3   601   596   14
 -1   238   203   19
  0   218   251   22
  1   289   288   17
  2   209   178   22
      H,  0,  8
-10   555   550   15
 -9   499   463   14
 -7   430   416   13
 -6  1693  1671    9
 -5   211   230   17
 -4  1139  1171    8
 -2   824   838    7
 -1  1834  1768    6
  0   835   852    6
  1  1702  1593    6
  2   503   508    7
  3   793   820    7
  6   679   637   10
  7   470   413   12
  8   389   388   15
  9   186   191   24
      H,  1,  8
-10   221   213   26
 -9   295   315   19
 -8   238   228   20
 -7   593   602   12

  H   KFO    FC   SIG
 -5  1625  1586    8
 -4   196   217   16
 -3   374   345    9
 -2   891   876    7
 -1  2090  2055    6
  1   254   266   10
  2   383   410    8
  3  1235  1217    7
  4   803   777    8
  6   539   523   12
  7   432   433   13
  8   371   336   15
 10   337   360   17
      H,  2,  8
-10   422   423   16
 -6   701   726   10
 -3   216   177   12
 -2  1548  1561    7
  0  1101  1089    6
  1  1177  1218    7
  2  1323  1304    7
  3  1061  1047    7
  4  1210  1209    8
  6   230   268   17
  7   767   740   11
  9   350   348   17
      H,  3,  8
 -9   215   247   24
 -7   402   419   14
 -6   208   237   22
 -5   347   362   12
 -3   627   640    8
 -1  1541  1580    7
  0   383   352    8
  1   166   155   13
  2   823   865    8
  3  1296  1314    8
  4   862   882    9
  5   692   677   10
  6   359   375   14
  8   486   477   13
      H,  4,  8
 -6   558   602   12
 -4   359   343   12
 -2   282   273   13
 -1   393   376   10
  0   234   226   13
  2   446   467    9
  5   260   231   16
      H,  5,  8
 -8   229   211   23
 -7   408   434   15
 -5   434   481   13
 -4   254   270   15
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      COLUMNS ARE 10FO,10FC,   10SIG                                                      COLUMNS ARE 10FO,10FC,   10SIG

   H    KFO     FC   SIG       H    KFO     FC   SIG       H    KFO     FC   SIG         H    KFO     FC   SIG       H    KFO     FC   SIG       H    KFO     FC   SIG
       H, 5,  8               -7    629    608    12       7    302    330    18                H, 2, 10                2    333    368    16      -7    479    474    13
  -3    387    411    12      -6    202    222    20            H, 7,  9                  -5    204    212    17             H, 8, 10               -6    374    367    14
  -2    357    370    12      -4    753    771     9      -5    325    307    18          -3   1269   1244     8      -5    249    234    20      -5    416    432    13
  -1    202    178    15      -3    453    454    10      -4    410    434    14          -2   1120   1110     7      -4    258    247    21      -4    529    525    11
   0    495    509    10      -2    887    874     8      -3    481    474    13          -1   1790   1763     7      -1    242    237    21      -3    619    634    10
   2    317    337    12      -1    332    357    10       0    702    697    11           0   1311   1337     7             H, 9, 10                1    319    335    12
   5    443    481    13       0    894    921     7       1    462    424    13           1   1266   1236     7      -2    329    343    19       2    169    168    20
   7    271    263    19       1    259    270    11       2    502    508    12           2    673    677     8      -1    268    234    19       3    502    521    11
       H, 6,  8                2    506    523     9       3    402    375    13           3   2173   2144     8       2    266    255    20       5    664    662    11
   2    218    245    18       3    477    492     9       4    603    596    12           6    379    347    13             H, 1, 11                6    223    145    21
   3    210    188    19       4    385    383    12       6    507    516    14           7    495    498    13      -6    291    287    16       8    346    342    18
   6    200    181    23       8    312    356    19            H, 8,  9                   8    393    388    16      -5    136    135    27             H, 5, 11
       H, 7,  8                     H, 4,  9              -5    262    236    20                H, 3, 10               -4    524    521    10      -8    408    410    16
  -6    303    281    17      -9    228    205    24      -4    357    319    16          -6    374    374    14      -3    445    437     9      -7    218    200    21
  -5    310    292    17      -8    524    517    14      -3    394    406    15          -5    456    473    12      -2    210    197    13      -4    777    805    11
  -4    221    216    21      -6    408    458    14      -2    216    207    23          -3    633    647     9      -1    473    488     8      -2    277    310    14
  -3    372    422    13      -5    631    632    11       1    460    471    13          -2   1437   1460     8       0    962    963     7      -1    464    475    11
  -2    393    401    14      -4    205    234    17       5    408    400    16          -1    469    491     9       1    523    520     8       0    917    944     9
   0    204    213    19      -3   1068   1095     9            H, 9,  9                   1    318    319    10       2    861    849     8       2    378    370    12
   1    311    303    15      -2    502    523     9      -2    227    233    20           2   1095   1106     8       3    598    590     9       3    326    334    14
       H, 8,  8                0    253    247    12            H, 0, 10                   4    961    981     9       4    192    143    17       4    587    609    11
  -4    316    336    18       1    690    696     9      -8    454    459    13           6    273    245    17       5    546    546    10       5    464    488    13
  -3    243    272    21       2    168    172    19      -7   1095   1059    11           7    387    348    14       6    241    251    17       6    370    368    15
  -1    287    303    16       4    316    352    15      -6    395    416    13           8    356    373    16       7    345    330    17             H, 6, 11
       H, 9,  8                5    495    516    12      -5   1678   1640     9                H, 4, 10                      H, 2, 11               -7    181    168    26
  -2    199    132    24       6    516    532    13      -4    456    445     9          -7    330    356    16      -5    535    534    11      -5    707    697    12
  -1    385    406    17       9    510    529    15      -3    633    578     8          -6    272    300    17      -4    301    293    12      -4    273    259    19
   0    192    191    25            H, 5,  9              -2    306    290     9          -5    320    339    14      -3    258    273    13      -3    612    635    11
   2    388    387    15      -8    242    179    19      -1   2170   2098     6          -3    271    258    14      -2    676    692     8      -2    578    583    12
   3    315    326    18      -7    552    520    14       0    662    613     7          -1    764    815     9      -1    641    660     8      -1    608    603    11
       H, 1,  9               -6    323    344    16       1    405    362     8           1    140    101    20       0    420    436     9       0    476    487    13
  -5    488    471    10      -5    264    242    18       3   1271   1284     7           3    584    592    10       1    772    793     8       1    958    972    10
  -3    239    255    12      -4    715    712    10       4    611    588     9           7    459    461    14       2    308    314    11       3    232    237    20
   0   1501   1451     6      -3    815    835    10       5    653    654    10                H, 5, 10                3    417    414    10       4    705    721    12
   1   1248   1200     6      -2    241    224    15       7    740    731    11          -6    554    560    13       4    629    617    10       5    350    359    16
   2    346    353     9      -1    126    142    24       8    241    193    20          -4    432    452    12       6    319    336    15       6    398    425    15
   3    251    280    11       0    921    950     9       9    289    306    20          -2    225    219    17             H, 3, 11                      H, 7, 11
   4    552    537     9       1    481    487    10            H, 1, 10                  -1    379    387    12      -8    506    545    14      -4    492    484    13
   5    361    348    13       2    629    652    10      -9    447    406    15           0    333    321    12      -7    213    218    20      -2    181    164    22
   6    501    489    12       3    259    266    15      -8    324    283    18           1    435    455    12      -5    407    419    12      -1    443    459    13
   8    227    225    23       4    728    721    10      -7    319    277    15           4    354    338    13      -4    901    945     9       0    873    882    11
   9    243    255    24       6    514    547    13      -6   1059   1037    10           5    268    261    18      -3    245    229    15       2    500    484    13
       H, 2,  9                8    230    242    23      -5    469    487    11           6    413    431    14      -2    293    319    14       3    335    332    13
  -4    783    782     9            H, 6,  9              -3    471    442     9           8    143    168    31      -1    247    237    13       4    275    270    18
  -2    465    477     9      -5    236    256    20      -2   1365   1360     7                H, 6, 10                0    531    552     9       5    461    450    14
  -1   1254   1263     7      -4    697    691    11       0    299    310     9          -4    164    191    29       1    475    480     9       6    322    335    18
   0    626    639     7      -2    423    434    12       1    614    597     7           0    350    346    15       2    359    354    11             H, 8, 11
   1    846    806     7      -1    618    630    11       2   1419   1394     7           2    471    474    12       3    392    383    11      -3    394    401    16
   2    627    662     8       0    716    736    10       4    544    549    10           5    211    168    23       4    441    476    11      -1    206    199    20
   4    834    824     9       1   1024   1034    10       6    737    734    10                H, 7, 10                5    463    449    12       1    360    320    16
   5    949    976     9       2    717    745    10       8    645    649    12          -6    170    154    26       6    488    494    13       4    240    245    22
       H, 3,  9                3    326    327    15            H, 2, 10                  -4    278    297    20       9    399    396    17       5    379    349    17
  -9    302    291    18       4    294    315    17      -7    331    310    17          -3    372    391    14             H, 4, 11                      H, 0, 12
  -8    249    239    19       5    974    971    11      -6    732    728    10          -1    294    293    16      -8    266    256    21      -9    472    452    14
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COLUMNS ARE 10FO,10FC,  10SIG

  H    KFO     FC    SIG
       H,  0, 12
 -8    364    351     16
 -7    626    619     12
 -6    684    679     11
 -5    939    940     10
 -2   1386   1368      8
 -1    655    645      8
  1    559    588      8
  2    410    406      9
  3    792    788      8
  6   1271   1265     10
  7    666    649     12
  8    535    521     13
       H,  1, 12
 -7    364    370     15
 -6    843    819     11
 -4    300    290     13
 -3    649    618      9
 -2   1172   1156      8
 -1    546    555      8
  0    426    411      9
  1    746    744      8
  2    583    589      9
  3    619    639      9
  4    377    381     12
  5    581    581     11
  6    328    340     14
  7    722    706     12
       H,  2, 12
 -7    660    665     12
 -5    470    455     12
 -4    562    549     10
 -3   1045   1081      8
 -2    856    864      8
 -1   1491   1494      8
  0    368    362     10
  1    368    361     10
  2   1430   1417      8
  3    721    704      9
  4    586    566     10
  6    695    711     12
  8    413    402     16
       H,  3, 12
 -8    175    168     26
 -6    570    565     12
 -5    327    309     15
 -4    347    332     12
 -3    582    591     10
 -2   1524   1547      8
 -1    353    371     11
  0    499    494      9
  1    799    821      9
  2    376    388     11
  3   1203   1244      9
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  H    KFO     FC    SIG
  4    405    389     13
  7    449    453     13
       H,  4, 12
 -7    216    213     21
 -6    344    382     16
 -5    518    493     12
 -3    362    362     13
 -2    418    393     12
 -1    327    337     12
  0    268    275     13
  1    140    102     22
  2    434    455     13
  3    350    333     13
  6    288    309     18
       H,  5, 12
 -6    189    252     24
 -5    334    348     16
 -3    221    220     19
 -1    353    382     13
  0    317    322     13
  2    272    257     15
  5    420    455     13
  6    397    409     14
       H,  6, 12
 -6    220    233     24
 -1    280    302     16
  0    191    181     22
  1    379    394     13
  3    188    200     21
  4    269    286     18
  5    239    248     20
  7    174    191     26
       H,  7, 12
 -4    203    204     22
  5    226    258     23
       H,  9, 12
  0    222    217     22
  1    288    268     18
       H,  1, 13
 -7    327    328     16
 -6    289    264     16
 -4    547    540     10
 -2    204    199     15
 -1    544    534      9
  1    702    720      8
  2    359    350     10
  4    246    271     16
  5    286    255     15
  6    361    369     16
       H,  2, 13
 -9    198    169     25
 -6    353    373     15
 -5    426    417     13
 -4    442    450     11
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  H    KFO     FC    SIG
 -3    852    860      9
 -2    277    302     13
 -1    422    427     10
  0    720    725      8
  1    383    389     11
  2    502    488     10
  3    560    576     10
  4    397    419     12
  5    395    387     13
  6    369    349     15
       H,  3, 13
 -8    409    375     15
 -5    383    381     14
 -4    366    372     12
 -3    514    502     10
 -2    184    202     20
 -1    413    444     11
  0    397    402     11
  1    389    397     11
  2    199    208     17
  3    769    796     10
  5    593    606     11
  8    285    268     20
       H,  4, 13
 -8    384    373     17
 -7    409    394     15
 -4    553    562     11
 -1    295    292     14
  0    320    317     13
  2    269    293     17
  4    982   1028     10
  5    299    311     16
  7    312    262     17
  8    388    435     17
       H,  5, 13
 -5    561    568     13
 -4    524    523     13
 -3    290    291     17
 -2    292    293     15
 -1    636    647     11
  0    369    371     12
  1    538    565     12
  2    151    119     23
  3    499    491     12
  4    311    333     17
  5    516    529     13
       H,  6, 13
 -6    169    188     28
 -5    503    511     14
 -4    443    436     13
 -3    559    560     12
 -2    253    247     18
  0   1017   1030     10
  1    353    322     14
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COLUMNS ARE 10FO,10FC,  10SIG

  H    KFO     FC    SIG
       H,  6, 13
  2    249    209     18
  3    310    322     17
  4    553    558     12
  5    325    350     17
       H,  7, 13
 -5    484    474     15
 -4    520    493     14
 -2    225    266     20
 -1    681    685     12
  0    220    223     20
  1    635    664     12
  2    352    339     15
  3    195    165     22
  4    271    287     18
  5    595    581     14
       H,  8, 13
 -4    275    280     19
  0    391    371     15
  1    199    206     24
  4    449    445     15
       H,  0, 14
 -6   1009    995     10
 -3    164    106     19
 -2   1445   1409      8
 -1    346    324     11
  0    650    636      9
  1    561    517      9
  2   1458   1454      8
  3    265    296     13
  5    775    734     11
  6    970    943     11
  7    406    383     14
  8    455    450     15
  9    371    328     18
       H,  1, 14
 -7    703    662     12
 -5    455    432     12
 -4    223    231     18
 -3    870    880      9
 -1    982    955      8
  1    371    384     11
  2    311    319     13
  3   1201   1195      9
  4    264    264     14
  5    430    420     13
  6    592    576     12
  7    586    591     13
       H,  2, 14
 -8    273    278     19
 -7    524    541     13
 -6    502    490     12
 -5    430    429     13
 -4    565    560     11
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  H    KFO     FC    SIG
 -3    205    195     17
 -2   1906   1897      8
 -1    298    293     12
  1    704    707      9
  2   1011   1021      9
  3    520    539     11
  4    537    523     11
  5    397    400     13
  6    247    249     19
  7    295    320     17
       H,  3, 14
 -8    289    325     20
 -7    458    433     14
 -6    272    260     18
 -4    257    274     17
 -3   1073   1069      9
 -1   1112   1125      9
  0    311    290     13
  1    595    601     10
  2    667    641     10
  3    710    691     10
  4    237    228     18
  5    142    178     29
       H,  4, 14
 -8    206    208     24
 -6    315    328     16
 -2    668    663     10
  2    517    536     11
  4    306    330     15
  5    268    313     19
  6    406    423     15
       H,  5, 14
 -5    372    359     13
 -4    225    231     21
  0    397    411     13
  1    442    440     12
  2    303    313     16
  4    142    191     29
  5    364    377     17
  7    300    330     19
       H,  6, 14
 -5    311    303     18
 -4    145    167     31
 -2    222    206     20
  1    437    441     14
       H,  7, 14
 -3    171    175     25
  4    277    268     19
       H,  8, 14
  3    240    230     21
       H,  1, 15
 -5    598    587     11
 -3    474    463     11
 -2    477    478     10
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  H    KFO     FC    SIG
 -1    323    325     12
  0    640    678      9
  1    408    390     11
  2    502    500     11
  3    339    362     12
  4    320    321     13
  5    284    260     15
       H,  2, 15
 -6    448    434     13
 -4    567    564     11
 -3    162    186     22
 -2    371    356     12
 -1    869    870      9
  0    739    774      9
  1    290    280     13
  2    512    509     10
  5    333    321     15
       H,  3, 15
 -7    281    253     18
 -5    482    488     13
 -3    258    304     17
 -2    223    227     19
 -1    977    994      9
  1    414    420     12
  2    376    359     13
  3    285    318     15
  4    582    596     12
  5    401    422     15
  6    239    290     20
  8    483    471     15
       H,  4, 15
 -3    270    283     16
 -2    371    381     13
  0    664    680     10
  1    244    241     16
  3    574    576     12
  4    552    559     12
  6    431    425     15
  7    402    395     16
       H,  5, 15
 -7    254    202     21
 -5    534    584     13
 -4    237    239     20
 -3    269    256     17
 -1    586    599     12
  0    301    313     15
  1    466    448     13
  2    268    287     19
  4    353    335     15
  5    268    256     19
       H,  6, 15
 -6    367    395     17
 -4    792    786     12
 -1    612    607     12
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COLUMNS ARE 10FO,10FC, 10SIG

H	KFO	FC	SIG
	H, 6, 15		
0	703	733	11
1	298	339	16
2	462	499	14
3	322	335	16
5	309	309	18
	H, 7, 15		
-5	636	618	14
-3	260	234	20
-2	321	295	17
-1	474	457	13
0	442	437	14
1	621	613	12
4	401	386	15
	H, 8, 15		
-2	267	214	19
0	471	465	15
3	312	326	18
	H, 0, 16		
-7	423	425	15
-5	313	288	16
-4	397	393	12
-3	294	267	14
-2	676	676	10
-1	187	176	19
0	697	702	9
1	1180	1163	9
2	969	950	9
3	451	453	12
4	330	324	14
5	1071	1067	11
7	725	716	12
8	270	253	22
	H, 1, 16		
-8	208	164	23
-7	556	546	13
-6	423	406	14
-3	506	504	11
-2	786	787	10
-1	429	420	11
0	352	328	10
1	323	322	12
2	835	821	10
3	394	396	12
5	190	198	22
6	720	694	12
8	259	303	22
	H, 2, 16		
-8	506	514	15
-7	234	216	19
-6	604	571	12
-5	292	286	16
-3	976	956	10
-2	869	872	10
-1	614	620	10
0	443	448	11
1	736	730	10
3	894	879	10
5	215	203	20
7	338	325	17
	H, 3, 16		
-7	553	554	14
-4	500	491	12
-3	418	423	13
-2	875	868	10
-1	503	487	11
0	290	318	16
2	717	743	10
4	356	347	15
6	340	356	16
	H, 4, 16		
-6	150	171	31
-3	324	304	14
-2	221	202	19
1	361	344	14
2	281	295	18
5	222	169	21
7	350	368	18
	H, 5, 16		
-6	241	239	20
-4	328	320	16
-1	280	303	18
1	578	575	12
4	285	236	18
6	437	501	16
	H, 6, 16		
0	262	316	19
	H, 7, 16		
0	254	207	18
3	384	384	16
4	154	141	29
	H, 8, 16		
2	273	255	19
	H, 1, 17		
-6	340	353	16
-4	274	281	15
-3	282	263	16
-1	350	350	12
0	640	637	10
1	411	407	12
2	403	394	12
5	215	183	21
	H, 2, 17		
-8	200	180	26
-5	618	604	13
-4	477	504	13
-2	378	407	12
-1	844	636	10
1	565	581	11
2	299	290	15
	H, 3, 17		
-2	487	491	14
-1	297	320	14
0	699	696	10
1	296	286	15
2	311	320	17
4	773	790	11
7	341	347	17
	H, 4, 17		
-7	167	160	27
-5	314	309	16
-3	246	271	18
-1	738	728	11
0	439	435	14
2	205	162	20
3	786	814	11
5	596	605	13
6	344	361	17
	H, 5, 17		
-6	226	282	23
-4	357	361	17
-2	390	394	14
0	700	691	11
4	642	661	13
	H, 6, 17		
-5	561	566	15
-4	297	278	19
-2	409	423	15
-1	763	753	12
1	524	542	13
3	244	239	19
	H, 7, 17		
-3	310	313	18
-2	518	503	14
0	648	637	13
	H, 0, 18		
-7	463	449	16
-6	284	297	17
-4	420	392	13
-3	439	460	13
-2	194	186	18
-1	384	377	13
1	1499	1479	9
3	492	485	12
4	482	461	13
5	359	336	15
6	502	515	14
7	386	353	16
	H, 1, 18		
-6	366	352	15
-4	469	484	14
-2	769	752	10

COLUMNS ARE 10FO,10FC, 10SIG

H	KFO	FC	SIG
	H, 1, 18		
0	699	702	10
1	353	357	13
2	934	943	10
5	613	610	12
6	346	365	16
7	289	271	18
	H, 2, 18		
-7	726	688	13
-6	252	272	20
-4	311	293	16
-3	827	828	11
-2	310	306	16
-1	548	549	11
1	503	497	12
2	584	587	11
3	259	242	17
6	261	253	20
	H, 3, 18		
-7	243	247	20
-6	291	280	18
-5	334	304	16
-4	474	467	13
-3	329	315	15
-2	825	835	11
1	418	425	13
2	622	609	12
3	381	366	14
	H, 4, 18		
-5	155	150	26
-3	314	288	16
1	482	495	13
4	155	160	26
6	208	219	25
	H, 5, 18		
-5	268	292	20
-4	174	156	25
-3	281	261	16
0	315	267	16
2	310	301	15
	H, 7, 18		
3	175	111	28
	H, 1, 19		
-5	312	320	18
-4	409	405	14
-1	458	463	12
0	147	68	25
1	224	270	19
	H, 2, 19		
-5	305	327	16
-3	330	317	15
-2	338	311	14
0	284	325	17
2	151	184	29
4	155	184	29
	H, 3, 19		
-6	254	262	22
-4	188	196	22
-2	366	399	16
-1	332	354	15
0	335	307	15
1	317	313	15
3	635	644	12
4	316	308	17
	H, 4, 19		
-2	311	302	16
-1	666	701	12
2	683	687	12
3	306	306	16
4	688	716	13
5	421	435	15
	H, 5, 19		
-4	371	388	16
-1	495	500	13
0	229	239	21
3	569	561	13
4	185	196	26
	H, 6, 19		
-3	384	367	17
-2	477	468	15
0	469	483	14
2	174	171	24
4	303	318	20
	H, 7, 19		
-1	501	498	15
	H, 0, 20		
-7	299	264	19
-6	222	239	24
-5	308	315	18
-4	402	399	15
-3	436	422	14
0	976	958	10
1	397	391	13
2	992	1032	10
3	399	385	14
4	493	502	13
6	607	554	13
	H, 1, 20		
-7	369	369	17
-6	223	190	23
-5	215	198	24
-3	419	405	13
-1	315	290	15
1	819	810	11
2	279	279	17
3	250	195	18
5	528	517	13
	H, 2, 20		
-4	681	680	12
-3	275	293	17
-2	469	503	13
0	402	418	14
2	602	586	13
4	261	278	18
6	267	248	20
	H, 3, 20		
-6	353	306	17
-3	609	593	13
1	586	582	12
5	266	301	20
	H, 4, 20		
-5	233	177	23
0	350	319	15
2	324	322	17
	H, 5, 20		
-4	343	368	17
-1	282	266	17
0	233	299	19
1	264	299	21
	H, 1, 21		
-5	290	301	19
-3	279	283	18
-2	204	227	23
0	200	234	23
2	257	214	19
	H, 2, 21		
-6	424	419	15
-4	259	296	19
-3	300	329	17
6	210	164	24
	H, 3, 21		
-5	267	240	19
-1	542	537	12
2	320	347	16
3	422	454	15
5	178	239	30
	H, 4, 21		
-2	606	608	13
0	271	290	17
1	366	367	15
3	382	342	16
4	518	531	14
	H, 5, 21		
-1	566	549	14
2	342	349	17
3	378	403	16
	H, 6, 21		
0	294	281	19
	H, 0, 22		
-4	640	617	13
-2	545	528	12
-1	320	340	17

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        COLUMNS ARE 10FO,10FC,  10SIG

   H    KFO     FC    SIG       H    KFO     FC    SIG        H    KFO     FC    SIG
        H,   0, 22                   H,   5, 22                -4    663    660    14
   0    464    470     14      -2    154    177     29         -2    264    284    20
   1    693    685     12       1    370    361     16         -1    269    284    19
   2    559    554     13            H,   1, 23                 0    624    612    13
   5    627    606     13       1    287    259     17          2    364    366    16
        H,   1, 22              3    168    172     26               H,   2, 24
  -5    268    292     22            H,   2, 23                -3    515    540    14
  -3    753    759     12      -1    210    241     21         -1    239    214    19
  -2    274    254     18            H,   3, 23                 1    325    308    17
   0    316    318     16      -2    380    392     16          3    258    256    22
   1    540    509     13      -1    295    312     18               H,   3, 24
   2    283    258     18       0    262    269     21          0    260    264    19
   4    504    502     13       1    241    202     22               H,   4, 24
   5    224    244     22       2    418    401     16          1    351    351    17
   6    317    279     18       4    317    308     18               H,   1, 25
        H,   2, 22                   H,   4, 23                 0    216    187    22
  -4    483    468     14      -3    374    377     16               H,   3, 25
  -3    321    323     16      -2    297    300     18         -2    482    471    15
  -2    446    445     13      -1    465    463     16          0    268    253    18
   0    192    182     24       0    382    358     16          1    283    257    18
   1    223    225     19       1    250    234     19               H,   0, 26
   4    335    324     17       3    548    541     15          0    803    814    13
   5    329    314     21            H,   0, 24                 1    408    396    16
        H,   3, 22             -4    421    419     15          2    356    355    17
  -3    423    408     15      -3    713    694     13               H,   1, 26
   0    177    174     26      -2    278    316     20         -1    408    391    16
   1    253    220     18      -1    600    620     13          1    383    378    17
   3    150    102     29       0    184    136     22          2    247    249    21
   5    376    364     17       1    780    740     12               H,   2, 26
        H,   4, 22              3    149    164     29         -1    187    176    27
  -4    379    376     17       4    426    397     15          0    255    263    21
  -1    193    199     24            H,   1, 24                 1    314    283    18
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OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR $CpNiCoOs(CO)_6(C_2Ph_2)$

```
COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
     -10,   0,  L
  2   334   319    29
  4     9    67  691*
  6   264   259    22
  8   403   411    15
     -10,   1,  L
  1   503   487    15
  2   474   469    19
  3    98    61   60*
  4   468   470    13
  5   275   276    20
  6   325   310    17
  7   337   345    17
  8   535   559    12
  9   116    48   46*
     -10,   2,  L
  1   135    84   37*
  2   373   393    15
  3   474   503    12
  4    42   135  126*
  5   491   505    12
  6   267   266    21
  7     9   138  609*
     -10,   3,  L
  1   626   608    14
  2   449   436    21
  3   431   408    17
  4   444   418    14
  5     9    41  643*
      -9,   0,  L
  2   966   992    11
  4   562   572    11
  6   221   208    21
  8   122   148   38*
 10   602   603    10
 12   604   627    10
 14   649   654    10
      -9,   1,  L
  1   419   396    16
  2   115    41   53*
  3   424   401    13
  4   735   745     8
  5   186   203    25
  6   760   778     8
  7   270   308    17
  8   570   586    10
  9   337   337    15
 10   378   408    14
 11   141    82   35*
 12   117    41   42*
 13     9    31  576*
 14   161   179    32
      -9,   2,  L
  1   269   203    18

  L   KFO    FC   SIG
  2  1337  1309     7
  3    65    11   70*
  4   674   695     9
  5   378   394    13
  6   175   182    26
  7   720   732     8
  8   410   430    13
  9   263   269    19
 10   427   437    13
 11   108    80   47*
 12   163   180    32
 13   262   261    21
      -9,   3,  L
  1   323   344    23
  2   124   112   56*
  3   416   359    15
  4   667   662    10
  5   685   702     9
  6   624   628    10
  7   284   278    19
  8   275   270    19
  9   156   275   36*
 10     9    41  590*
 11   709   705    10
 12     9    75  637*
      -9,   4,  L
  1    97   136   72*
  2   526   550    15
  3   292   283    24
  4   315   268    21
  5   210   222    29
  6     9    67  669*
  7   721   721    10
  8   171    72    34
  9   882   861     9
      -9,   5,  L
  1   304   323    24
  2     9    39  783*
  3   560   571    15
  4     9    48  835*
  5   719   719    12
  6   185    88    34
      -8,   0,  L
  2   192   212    32
  4   797   806     8
  6  1067  1083     6
  8   771   771     7
 10   213   235    20
 12   742   780     8
 14   811   826     8
 16   591   601    10
      -8,   1,  L
  1   171   136    33
  2  1240  1213     7

  L   KFO    FC   SIG
  3   369   360    12
  4   644   674     8
  5   567   566     9
  6   151   189    28
  7   212   190    19
  8   710   729     8
  9   171   196    24
 10  1051  1121     7
 11   194   188    22
 12   414   425    12
 13    80    23   56*
 14   112    22   40*
 15   160    24    30
 16   414   408    13
 17   118    91   42*
      -8,   2,  L
  1   478   458    13
  2   235   225    21
  3   451   476    11
  4  1313  1320     6
  5     8    15  545*
  6  1146  1183     6
  7   263   287    17
  8   355   387    13
  9   447   448    11
 10   411   403    11
 11   190   166    23
 12   572   582    10
 13   126   201   39*
 14   323   322    16
 15    96    29   52*
 16     9   134  576*
      -8,   3,  L
  1   192   230    35
  2  1131  1104     9
  3   144    68   35*
  4   502   482    11
  5   323   328    15
  6   354   341    13
  7   560   556     9
  8   735   771     8
  9   516   535    10
 10   657   638     9
 11   157    57    30
 12    95    88   52*
 13   558   543    11
 14     9   123  604*
 15   749   776     9
      -8,   4,  L
  1   364   418    18
  2   126    95   47*
  3   214   223    28
  4   405   394    15
  5   318   319    17
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -8,   4,  L
  6   359   375    15
  7     8    40  591*
  8   326   325    16
  9   397   386    14
 10     8    81  600*
 11   739   749     9
 12   312   279    18
 13   671   653    10
 14   313   292    19
      -8,   5,  L
  1   239   232    27
  2   200   136    31
  3   295   284    23
  4     8   148  801*
  5   516   491    13
  6   198   124    30
  7   622   596    11
  8    79    44   72*
  9   487   449    13
 10   161   184   36*
 11   133    29   43*
      -8,   6,  L
  1   716   732    12
  2   150    98   42*
  3   404   424    17
  4   145   183   50*
  5     9   108  811*
  6     9    46  769*
  7   340   333    21
  8   159   152   41*
      -7,   0,  L
  2   693   670     9
  4   330   333    12
  6     7   174  528*
  8  1058  1056     6
 10  1220  1244     6
 12   850   838     7
 14   521   550    10
 16  1074  1093     7
 18   738   728     9
 20   161   132    32
      -7,   1,  L
  1   697   639     9
  2   657   664     7
  3   121    89   27*
  4  1108  1149     5
  5   524   539     8
  6  1264  1290     5
  7   621   640     7
  8   509   532     8
  9   251   240    15
 10   289   266    14
 11   289   270    14

  L   KFO    FC   SIG
 12  1203  1218     6
 13    72   128   59*
 14   983  1005     7
 15     8    14  525*
 16   220   221    21
 17     8    43  556*
 18   348   333    15
 19   142   130   35*
 20   516   515    12
      -7,   2,  L
  1    86   223   66*
  2  1240  1173     6
  3  1037  1041     6
  4   228   224    16
  5   729   738     7
  6   936   941     6
  7     7    39  513*
  8  1259  1289     6
  9   335   341    13
 10   907   918     6
 11   396   397    11
 12   107    57   36*
 13   140   113    29
 14   464   474    11
 15   207   238    22
 16   624   634     9
 17     9    69  590*
 18   350   336    15
 19     9    24  595*
      -7,   3,  L
  1   404   475    16
  2   497   480    13
  3   150    57    30
  4  1012   986     7
  5    49   144   85*
  6   776   760     7
  7   438   428    10
  8    79    23   50*
  9   517   519     9
 10   593   568     9
 11   424   413    11
 12   748   733     8
 13   153   197    29
 14   380   380    13
 15   339   327    15
 16     9    54  594*
 17   636   643    10
 18   152   143   35*
      -7,   4,  L
  1   725   783    11
  2   231   218    26
  3   929   855     9
  4   295   298    18
  5   548   541    10

  L   KFO    FC   SIG
  6    98    61   46*
  7   406   424    12
  8   344   363    14
  9   111    87   40*
 10   647   638     9
 11   302   290    16
 12   143   105   31*
 13   713   691     9
 14   227   192    22
 15   716   683     9
 16   510   473    12
 17   192   200    29
      -7,   5,  L
  1   741   763    10
  2   129    32   41*
  3     8    64  762*
  4   187   230    33
  5   465   479    14
  6   169   111    31
  7   841   830     9
  8   273   293    20
  9   562   536    11
 10    79    91   63*
 11   388   369    14
 12   230   272    24
 13   172   139    30
 14   392   383    15
 15     9   147  661*
      -7,   6,  L
  1   662   677    11
  2   245   289    25
  3   899  1017    10
  4     8    96  807*
  5   750   708    11
  6   367   372    18
  7     8   140  722*
  8   197    96    32
  9   428   422    15
 10     9   113  704*
 11   462   450    14
 12   169   206    35
      -7,   7,  L
  1   827   842    10
  2   210   193    31
  3   140   105   44*
  4   248   274    27
  5   457   440    16
  6    23    68  313*
  7   739   709    12
  8     9    85  781*
      -6,   0,  L
  2   889   859     6
  4   594   572     6
  6   652   661     6
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
     -6,   0,  L           1  1204  1356     8     14     8   120  598*
  8   330   332    10      2   215   157    22     15   207   234    26
 10   570   565     7      3  1275  1202     6     16   549   541    11
 12  1830  1879     5      4   404   404    10     17   114   172   48*
 14  1248  1262     6      5   569   551     8          -6,   6,  L
 16     8    14  517*      6  1014  1030     6      1   707   746    10
 18   853   821     7      7   186   137    19      2   273   319    21
 20  1009   978     7      8   741   765     7      3   330   323    18
 22   359   369    16      9   596   601     8      4   374   360    18
     -6,   1,  L          10   435   435    10      5  1352  1287     8
  1   587   596    11     11   912   922     7      6     8    35  757*
  2   593   589     6     12   217   198    19      7  1408  1359     8
  3   895   936     5     13   466   453    10      8   306   291    19
  4   565   574     6     14   486   463    10      9   384   378    15
  5   367   386     9     15   363   383    13     10   291   272    19
  6  1154  1162     5     16   655   650     9     11   407   406    15
  7   557   564     7     17     8   176  563*     12     8   134  668*
  8  1119  1155     5     18   260   260    19     13   448   416    13
  9   665   670     6     19   457   452    12     14   310   317    18
 10   977  1009     6     20   116   224   47*     15   286   270    20
 11   462   438     8          -6,   4,  L               -6,   7,  L
 12   180   150    20      1     7    72  709*      1   931   929     9
 13   250   243    16      2   224   236    24      2    41   108  139*
 14  1055  1062     6      3   689   672     9      3   971  1086     9
 15     8   121  532*      4   118   142   38*      4   240   225    25
 16  1171  1186     6      5   893   898     7      5   616   610    12
 17     8     9  562*      6   272   247    16      6   291   253    23
 18   674   649     8      7  1056  1020     6      7   385   383    18
 19    66    26   72*      8     7    36  559*      8   258   246    26
 20     9     7  563*      9   901   891     7      9   856   824    10
 21   164   180    32     10   219   211    20     10   133   180   50*
     -6,   2,  L          11   151   182    28     11   723   682    11
  1   518   578    12     12   512   510    10     12   159    55   39*
  2  1063   981     6     13   310   336    15          -6,   8,  L
  3   529   529     7     14   545   564    10      1   260   216    25
  4  1111  1092     5     15   856   847     8      2   306   287    21
  5  1282  1280     5     16    88    40   55*      3   131   149   48*
  6   457   471     8     17   717   709     9      4   243   224    26
  7  1167  1197     5     18   258   289    21      5   460   516    16
  8   527   519     8     19   197   240    29      6     9   153  790*
  9   314   326    12          -6,   5,  L           7   577   571    15
 10   912   903     6      1  1294  1428     8          -5,   0,  L
 11   369   361    11      2   297   296    19      2  2678  2575     4
 12  1201  1225     6      3  1440  1407     8      4  1386  1417     4
 13   565   566     9      4   303   254    18      6   288   295     9
 14   485   496    10      5   601   580    11      8   218   212    12
 15   248   232    17      6   489   465    12     10   499   504     7
 16   110   139   38*      7   358   349    15     12   479   465     8
 17   237   217    18      8    68    15   69*     14  1398  1396     5
 18   508   515    11      9  1003   968     7     16  1522  1522     6
 19   118   222   43*     10   534   536    10     18   422   426    11
 20   597   567    10     11   851   843     8     20   423   411    12
 21     9   139  594*     12   426   445    12     22  1037  1016     7
     -6,   3,  L          13   341   335    15          -5,   1,  L
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
     -5,   1,  L           6   751   745     6     15   699   674     9
  1   385   370     7      7   983   986     6     16     8    21  596*
  2  1184  1201     4      8  1128  1106     5     17   134   148   37*
  3   540   524     6      9   127    78    25     18   170   151    31
  4   357   342     8     10   351   358    10     19    76   169   72*
  5  1024  1043     4     11   761   765     6          -5,   6,  L
  6   912   903     5     12   488   494     8      1  1162  1207     8
  7   694   688     5     13  1049  1046     6      2     7   105  702*
  8   674   687     6     14   189   172    21      3  1204  1261     8
  9   350   348     9     15   816   824     7      4   186   125    31
 10   793   801     5     16   195   210    22      5   103    70   55*
 11   677   673     6     17   364   349    13      6   352   339    16
 12  1001  1025     5     18   329   327    14      7  1287  1233     7
 13   454   452     9     19   232   256    21      8   127    74   41*
 14   430   413     9     20   546   535    11      9  1571  1530     7
 15   113   128   32*     21   426   459    14     10   293   298    19
 16   577   579     8     22   465   467    13     11   801   749     8
 17     8   126  559*          -5,   4,  L          12   355   350    15
 18  1083  1090     7      1   884   945     8     13   221   231    24
 19     8   125  578*      2   568   564    10     14   135   233   38*
 20   918   910     7      3   528   500    10     15   480   461    13
 21     8    29  615*      4   564   540     9     16     9     5  627*
 22   380   354    14      5     7    69  567*     17   466   468    13
 23     9    96  590*      6   127   136   28*          -5,   7,  L
     -5,   2,  L           7   245   250    15      1   155   140   34*
  1   905   902     8      8   123   123   30*      2   142   104   36*
  2  1395  1294     5      9  1107  1127     6      3   417   430    14
  3  1152  1105     5     10   148    42    24      4   176   150    29
  4  1618  1593     4     11  1147  1127     6      5  1146  1125     9
  5     6    12  425*     12   130    63   29*      6   197    63    31
  6  1111  1122     5     13   469   449    10      7   995   931     9
  7   598   603     6     14   493   488    10      8   194   162    31
  8   207   213    14     15   308   316    15      9     8    62  752*
  9  1421  1423     5     16   393   356    12     10   245   246    25
 10   215   223    14     17   985  1005     7     11   705   663    10
 11   465   463     8     18   319   306    16     12   359   356    18
 12   570   571     7     19   819   783     8     13  1008   940     9
 13   351   349    11     20    70    38   75*     14    86     6   68*
 14   937   928     6     21   159   179   35*          -5,   8,  L
 15   479   465     9          -5,   5,  L           1   536   575    13
 16   574   565     8      1     7   122  654*      2   498   515    13
 17   358   358    13      2   360   387    14      3   536   544    12
 18   171   207    25      3   969   947     8      4   344   347    18
 19   322   315    14      4   541   501    11      5   133   152   46*
 20     8   126  579*      5  1616  1554     7      6   344   328    20
 21   142   192   36*      6   128   147   37*      7   406   409    18
 22   642   620    10      7  1227  1189     7      8   216   191    33
 23     9   211  699*      8   488   463    10      9   718   706    12
     -5,   3,  L           9   188   164    23     10     9    64  815*
  1   184   226    24     10   214   197    21          -5,   9,  L
  2   981   931     7     11   839   808     7      1   142   147   45*
  3   987   935     6     12   569   574     9          -4,   0,  L
  4     6    31  491*     13  1031  1014     7      2   490   490     6
  5  1247  1244     5     14   446   460    12      4  1698  1693     4
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   COLUMNS ARE 10FO,10FC,  10SIG                                                    COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG       L   KFO    FC   SIG       L   KFO    FC   SIG       L   KFO    FC   SIG       L   KFO    FC   SIG       L   KFO    FC   SIG
      -4,  0,  L         18   386   372   11             -4,  5,  L                -4,  7,  L         13     6    72  590*    15   283   269   13
  6  1702  1704    4     19   401   395   11        1   491   498   10       12   175   174   32       14  1862  1889    5     16   434   428    9
  8   989   989    4     20   473   478   11        2   597   640    9       13   400   397   15       15     7   385  939*    17   437   445   10
 10   150   165   16     21   390   404   13        3     7    78  686*      14   440   440   15       16   904   947    6     18   135   195   31*
 12    47    72   56*    22   181   241   28        4   228   222   19       15   720   704   10       17     7   324  743*    19  1096  1084    6
 14   346   338    9     23   182   274   31        5   715   688    8       16   180   139   33       18   129   221   31*    20     8    77  542*
 16   917   902    6     24   288   306   19        6   527   533    9             -4,  8,  L         19     8   128  561*    21   784   757    8
 18  1005  1003    6           -4,  3,  L           7  1330  1279    6        1   393   361   15       20   253   282   16     22     8    81  623*
 20   718   729    8      1   518   539    9        8   110   102   35*       2     8     9  635*    21     8   213  617*    23     9    22  573*
 22     8    39  543*     2  1758  1649    6        9  1332  1333    6        3   295   343   19       22   328   394   15     24   136   192   40*
 24   695   711    9      3   326   298   10       10   362   378   12        4   405   440   14       23     8   186  769*          -3,  4,  L
      -4,  1,  L          4  1923  1862    5       11   839   803    7        5   340   333   17       24   533   550   11      1  1183  1256    6
  1   534   494    5      5   117   136   23       12    80   107   53*       6   447   457   15       25     9   119  801*     2  1089  1101    7
  2  2881  2848    3      6   544   551    6       13   525   524    9        7   122   298   56*            -3,  2,  L          3  2088  1999    5
  3    38   122   53*     7   772   737    5       14   209   204   21        8   396   404   18        1   280   251   12      4   469   438    8
  4  2361  2376    3      8   999   988    5       15   826   809    8        9     9    42  757*      2  2237  2110    4      5  1099  1094    5
  5   249   264    9      9   910   928    5       16   360   370   14       10   241   229   27        3   460   452    6      6     6    29  475*
  6   560   587    5     10  1343  1310    5       17  1126  1132    7       11   544   530   14        4  2307  2272    4      7   428   439    8
  7   863   874    4     11   640   668    7       18   152   124   34*      12     9    99  744*      5  1565  1573    4      8   250   255   12
  8   938   930    4     12   842   836    6       19   460   441   13       13   792   740   11        6    61    94   33*     9   368   368    9
  9   781   783    5     13   235   227   15       20   148   124   36*            -4,  9,  L           7  1454  1456    4     10   911   915    6
 10  1115  1119    4     14   145    84   24       21   159    93   34        1   129    26   44*      8  1656  1639    4     11   343   334   10
 11   115    75   23     15  1011  1012    6             -4,  6,  L           2   690   676   11        9   126   150   20     12   569   558    7
 12  1072  1089    5     16   189   219   22        1   439   419   12        3   213   210   30       10  2837  2880    4     13   413   425    9
 13   342   346   10     17  1242  1218    6        2   200   184   23        4   557   568   13       11   547   564    6     14    62    21   57*
 14   778   780    6     18     8    44  559*      3  1049  1147    8        5     9    58  717*     12  1256  1267    5     15  1130  1144    6
 15   498   500    8     19   547   548    9        4   168   148   32        6   281   293   23       13   937   954    5     16   207   212   20
 16   214   263   17     20    52   145   89*      5  1268  1257    7        7   167   111   37*     14   400   408    8     17  1195  1212    6
 17     7   107  502*    21   198   195   24        6    68   117   72*            -3,  0,  L          15   561   582    7     18   272   283   17
 18   310   339   13     22   486   470   12        7   412   400   13        2  3122  3086    3       16  1086  1078    6     19   359   348   13
 19     8    18  554*    23   466   490   13        8   239   216   20        4  2242  2269    3       17   159   270   26     20   144   141   33*
 20   560   548    9           -4,  4,  L           9   645   619    9        6   484   489    5       18   684   686    7     21   543   570   11
 21     8    24  741*     1  1567  1681    6       10     8    35  621*      8  1595  1616    4       19     8    43  535*    22   169   138   32
 22   932   938    7      2   475   467   11       11  1287  1257    7       10  2117  2164    4       20   309   308   14     23   941   918    8
 23     9    81  814*     3   909   901    7       12   346   344   15       12  1397  1454    4       21     8   156  519*          -3,  5,  L
 24   485   474   12      4   746   713    7       13  1112  1051    7       14   430   440    8       22   353   346   13      1  1732  1808    6
      -4,  2,  L          5   562   554    8       14   199   115   24       16   670   695    6       23   264   292   20      2   251   254   17
  1   548   524    9      6  1001   986    6       15   202   177   25       18  1011  1021    6       24   206   295   27      3   729   728    8
  2  1392  1297    5      7   497   494    8       16   240   219   22       20   903   898    7       25   239   302   24      4   138   212   29
  3   953   960    5      8   431   419    9       17   164   147   33       22   354   345   13             -3,  3,  L           5    90    58   41*
  4  1106  1078    4      9    34    60   95*     18   179    55   30       24   154   142   31        1  2286  2351    5      6   165   133   22
  5  1353  1340    4     10   143   101   23       19   596   594   11             -3,  1,  L           2   982   930    6      7   692   675    7
  6  2348  2373    4     11   720   696    7             -4,  7,  L           1   659   622    5        3   561   571    6      8    76    34   44*
  7   643   651    5     12   154    38   23        1   525   527   11        2  1053  1041    3        4  1261  1230    4      9   912   912    6
  8  1886  1894    4     13  1558  1585    5        2   480   485   12        3   358   350    5        5   267   257   10     10   272   255   14
  9  1066  1085    5     14   434   437   10        3     8   174  699*      4  2328  2302    3        6  2134  2131    4     11  1005   988    6
 10   327   346    9     15   852   831    7        4     8   121  697*      5   452   442    5        7   619   613    6     12   132   110   28
 11  1050  1068    5     16   248   243   18        5   307   321   19        6  2115  2155    3        8  1726  1721    4     13   882   861    7
 12   537   533    7     17    75    60   56*      6    90    36   62*      7   790   789    4        9   155   162   17     14     8    80  541*
 13   799   808    6     18   340   312   14        7   898   867    9        8  1817  1859    4       10    35     3   73*    15    89    34   46*
 14   869   845    6     19  1020  1040    7        8    82    22   66*      9   544   546    5       11   962   989    5     16     8   139  551*
 15   117    69   29*    20   214   278   26        9  1090  1037    8       10     6    15  391*     12  1506  1508    5     17   394   401   12
 16   547   534    8     21   929   923    8       10   157   133   34       11   844   840    5       13  1122  1140    5     18   280   293   17
 17   219   261   18     22     9    49  646*     11   456   432   13       12  1094  1155    5       14  1115  1111    5     19  1053  1052    7
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC    SIG      L   KFO    FC    SIG      L   KFO    FC    SIG
     -3,  5,  L              11    76    74    79*     25     9   226   955*
 20     9    17   623*     12   312   286     21     26   495   568     13
 21   760   727      9     13    98   192    66*         -2,  2,  L
 22    94   138    59*     14   306   317     22      1   467   456      7
     -3,  6,  L                  -3,  9,  L           2   203   173     11
  1     7    72   629*      1   553   548     12      3  2055  1963      3
  2   212   213     21      2   157   177    39*      4  1485  1474      3
  3   956  1007      8      3     9    60   693*      5   704   702      4
  4   332   292     15      4   564   578     12      6  1864  1872      3
  5  1287  1236      7      5     9   327   773*      7  1293  1298      4
  6     7    18   652*      6   773   818     11      8  1467  1466      4
  7  1205  1165      7      7   122   166    53*      9  1172  1201      4
  8   359   347     13      8   538   550     14     10  1072  1077      4
  9   646   620      9      9     9    35   792*     11   640   640      5
 10   273   284     17     10    71   118   102*     12  2059  2079      4
 11   381   343     12         -2,  0,  L          13   498   520      7
 12   116   117    38*      2  2551  2516      2     14  1616  1652      4
 13   790   770      8      4  2312  2292      3     15   831   858      6
 14     8    73   554*      6   315   330      5     16   145   188     23
 15   880   865      8      8   582   579      4     17   671   675      7
 16   236   242     21     10   592   567      5     18   649   685      8
 17   602   601     10     12  1250  1288      4     19   407   468     12
 18     9    85   635*     14  2176  2206      4     20   929   919      7
 19     9   124   615*     16  1295  1322      5     21     8   115   621*
 20     9    14   609*     18    65   150    51*     22   518   510      9
     -3,  7,  L              20  1415  1423      6     23     8    65   605*
  1   949   939      8     22  1034  1014      7     24   355   331     15
  2   561   585     10     24   306   311     16     25    77   244    73*
  3   678   722     10     26   269   264     20         -2,  3,  L
  4   546   555     11         -2,  1,  L           1  1994  1964      5
  5   156    99     34      1   134   149     14      2   599   583      6
  6   177    71     29      2  2403  2322      3      3  2367  2288      4
  7   415   388     14      3   503   504      4      4   435   412      6
  8     8    76   700*      4   305   276      5      5   896   903      4
  9   901   848      8      5  1055  1037      3      6   258   265      9
 10   180   104     27      6   401   414      5      7   640   613      5
 11   799   777      9      7    84    77    19*      8  1482  1495      4
 12   174    77     29      8  1395  1435      3      9   706   711      5
 13   477   444     12      9   448   448      5     10  1594  1614      4
 14   225   185     24     10  2154  2194      4     11   903   907      5
 15     9     2   643*     11  1082  1075      4     12   552   579      7
 16   585   566     11     12   770   847      5     13   783   772      5
 17   347   329     17     13   529   577      7     14   815   815      6
     -3,  8,  L              14   544   604      7     15   810   805      6
  1   324   320     17     15     6   355   872*     16  1090  1090      6
  2   478   493     12     16  1374  1436      5     17   379   399     11
  3   626   657     10     17     7   359  1598*     18  1004   991      6
  4     8    75   641*     18  1490  1564      6     19    65   158    63*
  5   400   416     14     19     7   188  1516*     20     8    49   529*
  6   217   202     26     20   572   609      9     21   729   713      8
  7   308   254     20     21     8    71   749*     22     8   129   601*
  8   334   318     18     22   113   243    39*     23   740   727      9
  9   293   279     21     23     8   206   769*     24     9   186   649*
 10   406   399     16     24   523   587     12     25   170   175     32
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC    SIG      L   KFO    FC    SIG      L   KFO    FC    SIG
     -2,  4,  L               6   351   341     13      3   790   832     10
  1  1706  1839      5      7  1706  1654      6      4   132    81    43*
  2   447   425      9      8   268   254     17      5   308   275     19
  3   110    97    28*      9  1420  1356      6      6   484   512     14
  4   250   258     12     10   615   613      9      7   200   124     32
  5  1608  1575      5     11   329   306     14      8   771   794     11
  6   374   399      8     12   373   349     12      9   216   161     32
  7  1329  1300      5     13   204   219     22     10   569   532     14
  8   556   564      6     14     8    15   549*     11     9    65   778*
  9   540   550      6     15   789   810      8         -2, 10,  L
 10   143   139     20     16     8   128   583*      1   317   299     20
 11   222   235     14     17   477   469     11      2   222   219     28
 12   714   730      6     18   125   149    40*         -1,  0,  L
 13   245   258     14     19   563   575     11      2  2282  2202      2
 14   836   830      6     20     9    95   632*      4  2693  2637      2
 15   467   472      9     21   315   292     18      6   353   326      5
 16   401   403     10         -2,  7,  L           8   686   646      4
 17   671   677      7      1   385   392     13     10   722   714      4
 18     8    52   526*      2     7   115   661*     12   243   252      9
 19  1164  1142      6      3  1235  1319      7     14   228   230     11
 20   404   408     13      4   300   331     18     16  1872  1898      4
 21   458   439     11      5  1165  1201      8     18  1927  1947      5
 22   311   272     17      6   585   545     11     20   440   450     10
 23   276   295     20      7   595   584     11     22   979   957      7
 24   187   184     30      8   313   279     17     24  1246  1210      7
     -2,  5,  L               9   622   619     10     26   799   787      9
  1  1158  1201      6     10   190   176     26         -1,  1,  L
  2   317   316     13     11   893   869      8      1  1125  1047      3
  3  2345  2316      6     12     8    15   622*      2  2340  2278      2
  4   411   394     10     13   845   793      8      3   732   694      3
  5  1423  1412      6     14     8    32   601*      4   186   200      7
  6   132    93     27     15   417   405     14      5   449   455      4
  7   396   380     10     16   169   184     32      6   856   875      3
  8   243   252     14     17     9    96   634*      7  1471  1461      3
  9   845   828      6     18   398   386     15      8   738   751      4
 10   181   171     19         -2,  8,  L           9  1390  1375      3
 11   924   927      6      1   848   834      9     10  1488  1513      4
 12   337   346     11      2   760   772      9     11   495   502      6
 13   771   758      7      3   121   105    42*     12  1547  1557      4
 14   359   339     12      4   526   557     12     13   765   845      6
 15   724   725      8      5   588   640     11     14   771   873      6
 16     8    68   511*      6   228   225     24     15   552   613      8
 17     8    77   505*      7   709   712     11     16     7   221   572*
 18   376   350     12      8     8   103   722*     17     7   232   730*
 19   146   171    32*      9   517   498     12     18   796   842      7
 20     8   141   576*     10   197   202     29     19     7   179  1450*
 21   755   747      9     11   164   160     35     20  1396  1389      6
 22   112    39    48*     12    40   194   150*     21     8    24  1632*
     -2,  6,  L              13   131   126    46*     22   903   911      7
  1  1385  1401      6     14   475   443     14     23     8   163   978*
  2     7    31   591*     15   101   139    65*     24     8   100   562*
  3   372   396     13         -2,  9,  L          25     9    64   810*
  4   452   445     11      1   582   574     11     26   457   531     14
  5   857   863      8      2   406   386     15         -1,  2,  L
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     COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
      -1,  2,  L         1  2223  2361    5      6   410   385   11
 1  1202  1136    4      2   664   646    7      7    14   102  302*
 2   557   545    5      3  2887  2793    5      8   305   273   14
 3  1827  1770    3      4   744   720    6      9  1589  1569    6
 4   192   221    9      5   681   686    6     10     7    12  609*
 5   601   600    4      6   568   573    6     11  1717  1661    6
 6  1136  1163    3      7   564   577    6     12   497   498   10
 7   268   261    7      8   441   431    7     13   815   789    8
 8  1913  1952    3      9  1413  1412    5     14   599   605    9
 9  1250  1249    4     10   244   252   12     15    24   224  197*
10  1570  1568    4     11  1078  1092    5     16     8    35  546*
11  1286  1324    4     12   158   186   19     17   770   758    8
12   534   538    6     13   690   681    6     18   178   144   28
13   763   785    5     14   347   347   10     19   479   477   12
14  1271  1260    5     15   363   360   10     20   245   211   22
15    94   205   34*    16   595   620    8     21   390   374   15
16  1375  1366    5     17   349   346   12           -1,  7,  L
17   739   787    7     18   664   660    8      1   841   830    8
18   881   907    6     19   549   572    9      2   724   775    9
19   447   499   11     20     8   140  546*     3   101   134   44*
20     8    94  538*    21   802   811    8      4   391   406   13
21   227   221   18     22     8   160  595*     5  1065  1072    8
22   463   464   10     23   439   473   13      6     7    88  766*
23     8   154  620*    24   481   476   13      7  1715  1670    7
24   688   665    9           -1,  5,  L         8   534   504   11
25     9     9  791*     1  1063  1098    6      9   864   850    8
26   564   544   11      2   571   584    8     10   236   205   21
      -1,  3,  L         3   237   240   16     11   116   186   43*
 1  1663  1605    5      4   287   286   13     12   224   236   23
 2   824   818    5      5  2190  2136    5     13   984   980    8
 3   117   103   19      6   699   705    7     14    44   109  113*
 4   247   263    9      7  2101  2097    5     15  1077  1044    8
 5  1564  1547    4      8   135   121   25     16     8    14  621*
 6   376   384    6      9  1016  1008    6     17   345   334   17
 7  1280  1321    4     10   432   428    9     18   123   170   46*
 8   118   135   19     11   160   179   21     19   143    68   39*
 9   699   696    5     12     7    99  517*          -1,  8,  L
10   861   856    5     13  1009  1014    6      1   781   773    9
11   462   471    7     14   525   534    9      2   272   281   20
12  1325  1343    4     15  1082  1063    6      3   917   933    8
13   756   790    6     16   645   631    8      4   600   645   11
14   920   930    5     17   626   607    8      5   584   600   11
15   943   963    5     18   130   107   32*     6   707   734   10
16     7    44  470*    19   154   169   29      7   268   271   23
17   730   736    7     20   390   357   13      8   229   191   25
18   707   688    7     21    91   142   55*     9   779   756   10
19   497   509    9     22   467   450   12     10     8   106  697*
20   966   956    6     23   418   409   14     11   730   700   10
21     8   106  529*          -1,  6,  L        12     8    38  709*
22   476   461   11      1   764   798    7     13   494   456   13
23   447   469   12      2   455   486   11     14   244   240   25
24     9   204  633*     3  1768  1832    6     15   125    79   47*
25   575   577   11      4   605   580    9     16   454   436   15
      -1,  4,  L         5  1513  1495    6           -1,  9,  L
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     COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
      -1,  9,  L        22   814   788    7     21   349   342   13
 1   261   222   21     23     8   141  570*    22   469   453   10
 2   544   538   12     24  1093  1076    7     23   266   285   18
 3   488   487   13     25     9   120  672*    24   570   544   10
 4   642   656   11     26   454   456   12     25   475   469   12
 5   630   655   11            0,  2,  L               0,  4,  L
 6   217   209   27      0  3556  3809    4      0   940   947    6
 7   463   489   15      1   125   146   19      1   755   760    6
 8   289   313   23      2  1107  1133    3      2   846   910    6
 9     9    68  803*     3   998   987    3      3  1611  1706    5
10   591   571   14      4   642   616    4      4   129    61   20
11   191     4   35      5   729   738    4      5  2175  2208    4
12   610   606   13      6   818   840    4      6   409   425    7
      -1, 10,  L         7  1405  1384    3      7   897   911    5
 1   162   171   37*     8  1875  1887    3      8   518   523    6
 2   108   150   57*     9   707   697    4      9   304   295    9
 3   358   342   18     10  2141  2112    4     10   362   351    8
 4   282   252   22     11   755   747    5     11   673   664    6
       0,  0,  L        12   864   854    5     12    27    78  104*
 2  2998  2993    1     13  1266  1272    4     13  1003  1021    5
 4   377   356    3     14     6    34  439*    14   214   230   16
 6  3729  3655    2     15  1210  1185    5     15  1250  1254    5
 8  1931  1920    3     16   867   873    6     16     7    75  505*
10   412   435    5     17     7    44  441*    17   728   720    7
12   384   385    6     18   764   722    6     18   143    90   26
14   603   588    5     19   410   425   10     19   214   220   19
16   196   203   13     20   936   912    6     20   555   530    9
18   822   844    6     21   383   378   11     21   660   646    9
20  1460  1516    6     22   366   343   11     22   334   339   15
22   686   698    7     23   104    93   38*    23   784   756    8
24   301   300   15     24     8    48  534*    24     9    33  584*
26  1191  1150    7     25   164   199   30            0,  5,  L
       0,  1,  L        26   541   528   11      1  1408  1389    6
 1   339   301    4            0,  3,  L         2  1130  1099    6
 2  3752  3715    2      1  2182  2244    4      3  1332  1349    6
 3   743   717    2      2  1587  1683    4      4   893   914    6
 4  3301  3239    2      3  1772  1783    4      5   126   126   25
 5   993   928    3      4  1951  1960    4      6   303   286   11
 6  2379  2346    3      5   392   398    6      7   860   906    6
 7   571   608    4      6   784   807    4      8   487   489    8
 8   551   552    4      7   848   823    4      9  1783  1771    5
 9  1839  1791    3      8   669   636    5     10   349   343   11
10   504   527    5      9  1690  1672    4     11  1610  1593    5
11  1218  1189    4     10   256   257   10     12   354   342   11
12  1126  1123    4     11  1114  1139    4     13   589   574    8
13     6    37  386*    12   958   930    5     14   374   374   11
14   844   885    5     13   592   574    6     15   580   564    8
15   772   774    5     14   789   788    6     16     8    90  528*
16  1133  1125    5     15   279   295   12     17  1242  1235    6
17   876   872    6     16   681   702    7     18   385   372   12
18   826   840    6     17  1182  1194    5     19   757   736    8
19   107    83   31*    18     7    82  498*    20   315   311   15
20   234   248   16     19   780   737    7     21   323   304   15
21     8   130  463*    20   105   102   36*    22   204   217   25
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC   SIG
      0,    5,   L
23     9    91  577*
      0,    6,   L
 0   133    66   27
 1   114    47   33*
 2   258   260   16
 3   411   416   11
 4   495   486   10
 5  1697  1802    6
 6   228   224   18
 7  1574  1609    6
 8   308   295   13
 9   853   833    7
10   488   513   10
11   858   848    7
12   212   264   21
13  1687  1643    6
14    66   212   71*
15  1337  1293    6
16   432   436   12
17     8   114  562*
18   193   187   25
19   607   574   10
20     9    23  586*
21   634   601   10
      0,    7,   L
 1   469   453   11
 2   653   640    9
 3   933   909    8
 4   913   859    8
 5   505   499   12
 6   343   325   15
 7   400   429   13
 8   172   153   27
 9  1300  1301    7
10   175   154   28
11  1373  1369    7
12   281   281   17
13   396   399   13
14   180   287   29
15   597   605   10
16   371   343   15
17  1019  1000    8
18   197   203   29
19   561   537   11
      0,    8,   L
 0  1210  1201    7
 1   188   163   27
 2   745   720    9
 3   389   378   14
 4   123    58   43*
 5   896   886    9
 6   720   706   10
 7   681   663   11
```

```
 L   KFO    FC   SIG
 8   448   461   14
 9   263   245   19
10   262   289   20
11   340   342   16
12   236   231   23
13   696   687   10
14   175   161   33
15   781   770   10
16     9   117  682*
      0,    9,   L
 1   580   558   11
 2   482   476   13
 3   249   222   22
 4   836   834    9
 5   192   221   30
 6   731   684   10
 7   428   413   15
 8   358   316   18
 9   314   288   21
10     9    79  727*
11   246   266   26
12   487   524   15
      0,   10,   L
 0   603   589   12
 1   167   103   36
 2   496   443   14
 3   133    35   45*
 4   341   331   19
 5   259   261   25
      1,    0,   L
 0  1651  1434    2
 2  4696  4449    2
 4  4058  3884    2
 6   728   702    3
 8  2083  2073    3
10  2997  2982    3
12  1625  1642    4
14   293   302    9
16    60    30   44*
18   134   138   22
20   361   346   10
22   853   863    7
24   910   928    7
26   158    42   31
      1,    1,   L
 0  3013  3424    3
 1   487   466    3
 2  1323  1295    2
 3   174   137    7
 4   335   311    4
 5   422   410    4
 6  3259  3178    3
 7   197   190    9
 8  3133  3102    3
```

```
 L   KFO    FC   SIG
 9   123   141   15
10   683   699    4
11   781   766    4
12   542   553    5
13   807   814    5
14  1469  1496    4
15   405   447    8
16  1140  1147    5
17   379   385   10
18   708   710    7
19   645   649    7
20   554   580    8
21   278   288   14
22   138    70   27
23     8    51  533*
24   664   641    8
25     9    37  668*
26   767   781    9
      1,    2,   L
 0   170   202   19
 1   392   365    7
 2   967   958    4
 3   621   593    4
 4  2211  2203    3
 5   602   610    4
 6  1586  1572    3
 7  1385  1375    3
 8   100    99   17
 9  1771  1773    4
10  2388  2360    4
11   833   845    5
12  2254  2243    4
13   566   587    6
14   846   833    5
15  1338  1325    5
16   425   445    9
17  1296  1302    5
18   794   793    6
19   170   209   22
20   534   540    8
21     8    85  492*
22   587   577    8
23   423   457   12
24   454   448   11
25   299   330   17
26   147   164   35*
      1,    3,   L
 0  1439  1503    5
 1  1872  1988    5
 2   254   267   10
 3  1751  1734    4
 4   447   437    6
 5   661   648    4
 6  1300  1306    4
```

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COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC   SIG
      1,    3,   L
 7   749   745    4
 8  2193  2203    4
 9   774   776    5
10   890   884    5
11   912   916    5
12   346   344    8
13  1035  1051    5
14  1442  1410    5
15  1300  1329    5
16   916   902    6
17   223   242   16
18   154   187   23
19   673   696    8
20     8   133  533*
21   992   968    7
22     8    15  506*
23   612   605    9
24     9    78  564*
25     9    10  569*
      1,    4,   L
 0   330   332   10
 1  1230  1259    6
 2  1471  1562    5
 3   835   854    5
 4  1248  1264    5
 5  1782  1760    4
 6   426   428    7
 7  1770  1777    4
 8   433   424    7
 9   556   584    6
10  1025  1016    5
11   229   215   13
12   661   663    6
13    19    66  151*
14   262   275   13
15   703   685    7
16     7    13  480*
17  1303  1324    6
18     8   110  542*
19  1049  1049    7
20    77   121   56*
21   144   131   29
22   351   342   15
23   752   730    9
24   168   247   34
      1,    5,   L
 0  1261  1231    6
 1  1751  1756    6
 2   246   248   16
 3  1377  1468    6
 4   546   566    8
 5   902   953    6
 6   593   623    7
```

```
 L   KFO    FC   SIG
 7    58    61   51*
 8   244   244   13
 9   562   586    7
10   349   350   10
11  1186  1190    6
12   348   336   11
13  1301  1304    6
14    71   145   52*
15   964   941    6
16   309   313   13
17     8    96  503*
18     8    94  511*
19   890   903    8
20   356   352   15
21  1067  1035    7
22   162   206   33
23   575   551   11
      1,    6,   L
 0   199   196   20
 1    58    88   64*
 2   336   319   13
 3   321   320   14
 4    43    73   92*
 5   656   691    8
 6    58    23   64*
 7  1031  1046    7
 8     7   116  566*
 9  1236  1239    6
10   326   333   13
11   861   849    7
12   457   463   10
13    90   168   48*
14   277   260   16
15  1114  1090    7
16     8    11  584*
17  1359  1311    7
18   181   206   28
19   573   554   11
20   155   152   34*
21   116   201   46*
      1,    7,   L
 0   766   772    8
 1   751   727    8
 2   181   115   25
 3   591   585   10
 4   698   682    9
 5   678   685   10
 6   643   672   10
 7   419   427   12
 8   315   313   15
 9   144   114   32*
10   208   154   22
11   941   939    8
12   199   178   24
```

```
 L   KFO    FC   SIG
13  1217  1191    7
14   118   190   43*
15   732   739    9
16   171   131   29
17   176    71   29
18   338   328   17
19   790   769    9
      1,    8,   L
 0   443   434   13
 1   137   112   38*
 2  1249  1244    7
 3   163    98   31
 4   857   827    9
 5   575   578   11
 6   168    52   32
 7   332   358   17
 8   403   456   15
 9   416   447   14
10   547   556   11
11   381   363   15
12   345   361   16
13    40    93  136*
14   223   203   26
15   294   261   20
16   110   104   52*
      1,    9,   L
 0   600   599   11
 1   662   652   10
 2   182   180   30
 3   570   566   12
 4   383   368   16
 5   205   203   29
 6   651   652   11
 7   165   187   37*
 8   785   797   11
 9   105    84   61*
10   442   455   15
11    70    42   86*
12     9    69  664*
      1,   10,   L
 0   204   181   30
 1     9    16  669*
 2   628   620   12
 3   162    51   38*
 4   629   605   12
      2,    0,   L
 0  1129   980    3
 2   626   588    3
 4  2076  1933    3
 6  2615  2513    3
 8  1569  1562    3
10   252   262    8
12  1995  1959    4
14  2278  2276    4
```

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COLUMNS ARE 10FO,10FC,  10SIG                                              COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG        L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG
       2,    0,   L      18    70    92   51*    18     8   117  530*              2,    7,   L             3,    0,   L      12  1310  1342     5
 16  1368  1370     5    19   726   708     7    19   811   811     7        0   803   798     8     0  3887  3445     3    13  1346  1348     5
 18    20    92  156*    20   580   615     8    20   116    98   37*        1   439   432    12     2  2731  2517     3    14    72    55   40*
 20   134    97    25    21   152   226    27    21   991   998     7        2   969   973     8     4    64    54   25*    15   502   512     8
 22  1018  1003     7    22   846   832     7    22    84   136   60*        3   809   829     8     6  2075  1954     3    16  1488  1493     5
 24   772   746     8    23     8    60  551*    23   428   427    13        4     7   100  693*     8  1137  1132     4    17     7    48  523*
 26   582   586    10    24   379   381    13    24     9   167  597*        5   914   970     8    10  1466  1403     4    18  1422  1412     6
       2,    1,   L      25   130   145   38*           2,    5,   L         6   563   589    11    12   197   217    12    19   519   537     9
  0  1186  1245     4           2,    3,   L       0   449   450    10        7   728   753     9    14   202   210    14    20   619   636     9
  1   466   447     4     0   299   288    11     1   420   425    10        8   671   678     9    16   951   939     5    21   631   642     9
  2  2886  2827     3     1    47    66   59*     2   650   676     8        9   255   255    19    18  1103  1097     6    22   325   323    14
  3   180   202     8     2   236   232    11     3  1375  1493     6       10   328   337    15    20   802   785     7    23     8   153  609*
  4   205   216     7     3  1276  1269     4     4   699   726     7       11   253   282    19    22     8    15  503*    24   760   769     9
  5   559   553     4     4   873   865     4     5  1864  1897     5       12     8    29  573*    24  1014   992     7    25     9   191  607*
  6   502   493     4     5  1952  1921     4     6   205   170    16       13   601   614    10           3,    1,   L             3,    3,   L  
  7   381   365     5     6   955   942     4     7  1257  1274     5       14     8   119  569*     0   282   282     8     0  1086  1182     6
  8  1631  1623     3     7   555   571     5     8   437   436     9       15   757   755     9     1   712   710     4     1  2524  2655     5
  9   191   227    11     8   539   536     6     9    65    70   49*       16   163    55    31     2  1680  1667     3     2   596   616     6
 10  2332  2320     4     9   695   672     5    10   288   286    12       17   879   850     8     3   694   641     4     3   642   628     5
 11     6   113  422*    10  2093  2068     4    11   540   539     8       18   169   243    34     4  2257  2180     3     4   122   121    19
 12  1908  1891     4    11   573   572     6    12   144    84    23              2,    8,   L         5   365   361     6     5   831   827     5
 13   425   420     7    12  2052  2046     4    13   841   846     7        0   566   576    11     6  1210  1196     4     6   655   673     5
 14   119   150    22    13   745   746     6    14     7    42  517*        1  1298  1240     7     7   947   952     4     7  1944  1944     4
 15   714   709     6    14   174   168    17    15   784   786     7        2   441   444    13     8   481   459     5     8   750   740     5
 16  1124  1106     5    15   766   777     6    16   217   181    19        3   610   598    11     9   561   566     5     9  1155  1161     5
 17     7   293  606*    16  1058  1058     6    17   826   798     7        4   895   922     9    10     6   121  394*    10   418   396     7
 18  1329  1347     5    17   830   845     7    18     8   121  525*        5     8    72  674*    11   639   612     5    11   139   129    20
 19     7   123  582*    18  1238  1216     6    19   182   155    24        6   706   720    11    12  1485  1441     4    12  1069  1062     5
 20   704   695     7    19   434   456    10    20   100   179   47*        7   733   768    10    13     6   117  438*    13   587   597     7
 21   271   350    17    20   470   462    10    21   407   393    13        8   209   219    25    14  1642  1635     5    14  1577  1575     5
 22   541   570     9    21   296   302    15    22   230   250    23        9   509   534    12    15   597   634     7    15   686   675     7
 23   166   220    27    22     8   115  555*           2,    6,   L        10   239   228    22    16   874   849     6    16   712   690     7
 24   295   291    16    23   766   760     8     0   123   140   35*       11   341   346    17    17   499   489     8    17   411   397    10
 25     9   101  578*    24     9    92  610*     1  1519  1528     6       12   427   426    14    18   143   137    25    18   376   365    11
 26   291   297    18    25   812   792     9     2    85    75   50*       13   154   190   36*    19    36   278  116*    19   496   510    10
       2,    2,   L             2,    4,   L       3   662   685     9       14   370   360    16    20   840   854     7    20   911   862     7
  0   124   130    26     0   755   738     7     4     7    76  631*       15   288   275    21    21     8   271  719*    21     8    98  529*
  1   377   391     7     1  2836  2864     5     5   114   130   35*              2,    9,   L        22  1270  1293     6    22   776   758     8
  2   684   671     4     2   492   500     8     6   153   102    25        0   501   496    13    23     8   174  770*    23     9   106  568*
  3   832   791     4     3  1512  1531     5     7   776   795     8        1     8    24  664*    24   471   468    11    24   131   151   39*
  4   244   213     7     4   312   322     9     8    72    87   53*        2   686   679    10    25     9    52  594*           3,    4,   L  
  5   627   618     4     5   338   345     9     9  1069  1057     7        3   831   822    10           3,    2,   L       0   455   463    10
  6  1983  2013     3     6  1164  1147     5    10     7    83  572*        4   193   209    32     0   297   361    11     1   970  1047     7
  7   414   411     6     7  1977  1936     4    11  1250  1213     6        5   810   786    10     1   462   468     7     2   186   188    18
  8  2103  2106     4     8   549   534     6    12   321   341    14        6   124    45   50*     2   229   223     9     3  2396  2436     5
  9   971   964     4     9  1324  1340     5    13   629   607     9        7   293   273    22     3   510   510     5     4    43    90   69*
 10  1239  1217     4    10   108   163   27*    14   369   379    13        8   475   459    15     4   444   439     5     5  2462  2434     5
 11  1174  1172     4    11   508   507     7    15     8    55  517*        9    23   182  280*     5   832   864     4     6   117    58    24
 12  1295  1277     4    12   582   576     7    16   185   207    24       10   709   733    12     6    68   126   28*     7   107   128   26*
 13   545   575     7    13     7   101  481*    17   791   780     8       11    99   144   64*     7   819   803     4     8   235   247    13
 14  2580  2559     4    14   954   931     6    18     8    55  593*              2,   10,   L         8   582   610     5     9  1354  1347     5
 15   359   405    10    15   290   300    13    19   952   939     8        0     9   136  674*     9   224   205    10    10   588   569     7
 16  1449  1454     5    16   463   454     9    20   247   259    22        1   426   407    16    10  1916  1905     4    11  1268  1269     5
 17   690   692     7    17   147   141    25    21   622   601    11        2    29   220  217*    11   626   644     6    12   521   519     8
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COLUMNS ARE 10FO,10FC,  10SIG                                           COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
       3,    4,  L       17    96   128   48*     4  1807  1687    4             4,    2,  L       18     8    30  507*     4   494   530   13
 13   614   614    7     18     9    46  590*     6   941   881    4     16     7   144  521*    19   158   167   28      5   533   566   12
 14    21    67  163*    19   562   580   11      8  1751  1710    4     17   860   845    7     20   342   334   15      6   682   698   10
 15   113   131   32*    20     9   101  601*    10  1391  1345    4     18   656   651    8     21   175   144   29      7   174   141   29
 16   516   518    9            3,    7,  L      12   784   772    5     19     8    86  539*    22   586   542   10      8   433   441   13
 17   362   351   11      0   300   313   17     14   307   311   10     20  1154  1161    7            4,    5,  L        9   787   786    9
 18   531   537    9      1   918   929    8     16   289   298   12     21   332   387   16      0   780   786    8     10   262   268   20
 19   192   172   21      2   686   676   10     18    36    94   98*    22   837   852    8      1  1039  1088    8     11   986   946    8
 20   418   403   11      3   351   362   16     20   948   938    7     23   368   430   16      2   320   343   15     12   350   328   16
 21   520   535   10      4   908   905    9     22  1025  1038    7     24   153   134   33*     3  1363  1391    6     13   933   916    8
 22    78    61   64*     5   182   243   30     24   597   597   10            4,    3,  L        4   797   796    7     14   282   260   20
 23   767   757    9      6   344   320   15            4,    1,  L        0   320   308   13      5  1068  1115    6     15   136    56   39*
       3,    5,  L        7  1060  1110    8      0  1042  1020    4      1   775   778    7      6   711   698    7     16   180   210   32
  0   439   432   10      8   421   429   13      1   301   303    7      2   601   614    7      7    72    30   50*           4,    8,  L
  1  1571  1614    6      9   986   997    8      2   500   538    5      3  1522  1498    5      8   215   206   18      0   485   490   13
  2   509   537   10     10   614   583   10      3    88    57   21*     4  1371  1348    5      9  1313  1314    6      1   364   354   16
  3   718   748    7     11   429   413   12      4  2165  2174    4      5  1138  1173    5     10   420   430   11      2   737   741   10
  4   472   497    9     12   300   282   16      5     5    83  377*     6   272   288   10     11  1515  1492    6      3     8    30  678*
  5   780   812    7     13     8    67  559*     6  1490  1467    4      7   164   184   16     12   152   151   28      4   655   687   11
  6    64    83   55*    14    97    38   50*     7   542   506    5      8   450   456    7     13  1049  1010    7      5   464   488   15
  7  1817  1798    5     15   532   505   11      8  1053  1038    4      9  1316  1310    5     14   245   223   17      6    98    18   62*
  8   409   418   10     16   179    81   30      9   135   137   17     10   426   409    8     15    23   177  179*     7   783   820   10
  9  1589  1593    5     17   736   703   10     10   332   314    8     11  1594  1608    5     16   307   334   15      8   543   565   13
 10   193   161   18            3,    8,  L      11   822   802    5     12   254   241   13     17   636   618    9      9   564   556   13
 11   393   393   10      0   809   827    9     12   547   529    6     13   794   808    6     18   202   229   25     10   591   587   12
 12   200   211   18      1   368   342   15     13   772   748    6     14   626   618    7     19   859   834    8     11    70    30   84*
 13   343   336   12      2   589   584   11     14   855   851    6     15   127    34   28     20   423   439   14     12   320   283   20
 14     8    24  504*     3  1068  1058    8     15   117   149   28*    16   910   891    7     21   466   467   13     13   467   477   15
 15   660   643    8      4   119   164   48*    16   857   891    6     17   262   277   16            4,    6,  L             4,    9,  L
 16     8   109  510*     5   922   968    9     17   337   388   13     18   793   777    7      0    45    68   96*     0   334   351   19
 17   770   738    8      6   593   616   12     18   900   927    7     19   554   575    9      1   163    71   28      1   194   252   33
 18   329   328   14      7   318   333   19     19   571   604    9     20   107   210   41*     2     7    43  639*     2     9    47  711*
 19   500   498   11      8   613   639   11     20   187   235   23     21   388   401   13      3   284   320   18      3   698   698   11
 20    60    41   78*     9   485   508   13     21   165   197   26     22   354   366   15      4     7   107  638*     4   481   473   15
 21   217   197   23     10   346   345   17     22   381   360   13     23   280   297   19      5   380   389   13      5   419   411   17
 22   106   276   53*    11   666   668   11     23     9    80  789*           4,    4,  L        6    56    15   83*     6   719   697   11
       3,    6,  L       12     9   125  659*    24  1116  1110    8      0  1091  1130    7      7  1390  1383    7      7   156    49   43*
  0   110   161   40*    13   398   399   16            4,    2,  L        1  1501  1607    7      8     7    39  621*           5,    0,  L
  1   788   784    8     14   118   112   50*     0  1826  1950    6      2   984  1028    7      9  1161  1133    7      0  1767  1593    5
  2   310   296   16            3,    9,  L       1   131   127   19      3   453   461    9     10   365   362   13      2  1603  1500    4
  3  1107  1163    8      0   296   297   20      2  1183  1176    4      4   391   376    9     11   307   316   15      4  3689  3493    4
  4     7    68  663*     1   843   832    9      3   225   222   10      5  1537  1518    5     12   128   161   36*     6  2615  2531    4
  5  1281  1341    7      2   553   559   13      4   192   204   11      6   234   213   14     13   800   801    8      8   184   141   15
  6    81    25   51*     3   275   273   23      5   391   406    7      7  2372  2403    5     14   219   243   23     10  2027  1957    4
  7   808   804    7      4   644   620   12      6   616   594    5      8   494   500    8     15  1322  1300    7     12  1674  1629    5
  8   194   183   22      5   373   377   18      7  1146  1151    5      9   705   707    7     16   127   153   41*    14   772   767    6
  9   170   272   25      6   424   401   16      8   384   388    7     10   328   335   11     17   725   700    9     16   149   128   24
 10   138   133   30*     7   810   847   11      9  1102  1119    5     11   574   567    7     18   271   289   21     18   168   175   23
 11  1131  1129    7      8   127    47   53*    10   589   599    6     12   436   431    9     19     9    81  615*    20     8    92  503*
 12   131   172   34*     9   425   423   17     11   296   283   10     13  1035  1028    6            4,    7,  L       22   341   331   15
 13  1157  1132    7     10   269   282   25     12  1180  1161    5     14    85   168   45*     0   618   613   10            5,    1,  L
 14     8    55  561*           4,    0,  L      13   747   737    6     15   739   718    7      1   581   549   11      0  2167  2189    4
 15   643   627    9      0  2439  2225    4     14   938   956    6     16     8    66  500*     2   217   240   25      1   990   993    5
 16   197   248   24      2  3402  3200    3     15   958   966    6     17   436   415   11      3   861   874    9      2  2230  2255    4
```

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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
       5,  1,  L           7   732   740     6     14   366   386    14
  3   212   202    11      8   814   809     6     15  1069  1070     7
  4   136   146    18      9    13    47  234*     16     8   106  577*
  5   178   161    14     10   894   945     6     17   359   347    15
  6  1328  1318     4     11   755   777     7     18   307   282    17
  7     6    46  430*     12   609   605     7     19   280   289    20
  8  2485  2415     4     13   913   908     6            5,  6,  L
  9   518   500     7     14   114   140   34*      0   154   157   34*
 10  1248  1222     5     15   927   967     7      1   559   561    12
 11    32    14   90*     16   349   336    13      2    76    75   70*
 12   419   416     9     17   307   324    14      3     7    62  705*
 13   377   353    10     18   665   659     9      4   175    94    28
 14   519   514     8     19   219   216    21      5   165   101    28
 15   825   819     7     20   510   519    11      6   138   100   33*
 16   355   356    11     21   522   547    11      7   281   278    17
 17   271   259    15     22   295   277    18      8   100    55   47*
 18   513   529     9            5,  4,  L          9   805   782     8
 19   389   387    12      0   171   192    28     10     8    25  562*
 20   594   605     9      1  1326  1385     7     11  1169  1201     7
 21   430   461    12      2   353   361    13     12   143    33   34*
 22   457   470    12      3  1000   998     7     13   702   699     9
 23   191   281    29      4  1084  1086     6     14   459   465    13
       5,  2,  L           5   221   219    16     15   334   302    16
  0   102    79   31*      6   639   676     8     16   147   230   39*
  1   179   169    16      7  1058  1056     6     17  1016   984     8
  2  1415  1364     5      8     7    39  490*            5,  7,  L
  3   586   546     6      9  1353  1330     6      0   486   478    13
  4  1890  1858     4     10   376   381    11      1   527   526    12
  5   463   465     7     11   959   971     6      2   482   497    13
  6  1231  1243     5     12   735   731     7      3   542   566    13
  7   115    77    23     13   181   149    22      4   330   340    19
  8   332   347     9     14   129   103   31*      5   366   401    16
  9   473   461     7     15   345   371    13      6   425   436    14
 10   349   353    10     16     8    83  504*      7   488   515    13
 11   889   907     6     17   790   777     8      8   583   590    11
 12   934   939     6     18   202   201    23      9   434   433    14
 13   542   543     8     19   629   624     9     10   362   337    16
 14   984   976     6     20    89   122   57*     11    71    66   77*
 15   100   108   38*     21   344   369    16     12     9   144  682*
 16   646   678     8            5,  5,  L         13   875   860     9
 17   926   956     7      0   486   502    11     14   163   176   37*
 18   120   185   35*      1   565   608    11            5,  8,  L
 19   604   621     9      2  1026  1104     8      0   213   210    27
 20   396   398    13      3  1062  1095     8      1   432   448    15
 21   147   181   33*      4   451   450    11      2   218   184    27
 22   484   484    12      5   743   779     8      3   258   262    24
       5,  3,  L           6   485   489    10      4   763   787    11
  0  1295  1403     7      7  1021  1007     7      5   163    20   40*
  1   619   605     8      8   573   569     9      6   727   779    12
  2   703   694     7      9   312   314    14      7   229   231    28
  3   861   882     6     10   276   282    16      8   169    68    37
  4   105    96   27*     11   310   321    15      9   528   535    14
  5   751   758     6     12   330   336    14     10   398   381    17
  6   916   928     6     13  1133  1130     7            5,  9,  L
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
       5,  9,  L          14   837   833     7      2   121   195   43*
  0   532   515    14     15   604   620     9      3   288   252    18
  1   179    30    32     16  1067  1055     7      4   607   632    10
       6,  0,  L          17     8    41  542*      5  1322  1325     7
  0  1998  1818     5     18   683   685     9      6   310   307    15
  2  1668  1553     5     19   413   407    13      7   666   647     9
  4   508   483     7     20     9    35  579*      8   223   222    20
  6  1855  1821     5     21   734   736     9      9   815   856     8
  8  2576  2490     5            6,  3,  L         10   662   673     9
 10   934   936     6      0   575   619    10     11   131   143   34*
 12   762   752     7      1   909   924     7     12   289   292    16
 14  1525  1536     6      2   681   675     7     13   143    74    31
 16   705   712     8      3   152   144    22     14   175   116    26
 18   100   145   41*      4   583   549     8     15   468   463    12
 20    92   118   50*      5  1271  1273     6     16   240   233    22
 22   139   124   36*      6   504   532     9     17   706   692    10
       6,  1,  L           7  1024  1021     6            6,  6,  L
  0   370   351     8      8   209   202    17      0     8    60  710*
  1   207   178    13      9   679   684     7      1  1196  1254     9
  2   939   958     5     10   702   696     7      2     8   226  838*
  3   647   619     6     11   178   156    22      3   947   980     9
  4  1880  1908     5     12   952   990     7      4   181   109    28
  5   506   493     7     13   232   221    18      5   133   152   39*
  6  1585  1573     5     14   571   578     9      6   134    32   36*
  7   215   187    14     15   417   429    11      7   161   154    30
  8   270   255    12     16    83    77   51*      8   165   191    30
  9    63    94   50*     17   438   441    12      9   158   214    31
 10  1661  1635     5     18   431   422    12     10     8    10  588*
 11   365   354    11     19   525   527    11     11   429   406    13
 12  1588  1558     5     20   578   587    11     12   229   235    23
 13   246   236    16            6,  4,  L         13   584   574    11
 14   681   650     7      0   511   568    13     14   119    44   45*
 15   302   289    14      1   963  1050     9     15   638   665    11
 16   356   356    12      2   569   588    10            6,  7,  L
 17   541   521     9      3  1670  1696     7      0   182    96    31
 18   648   631     9      4   114    81   36*      1   160   114   36*
 19   349   350    14      5   949   979     7      2   340   366    19
 20   624   632    10      6   518   532     9      3   492   528    15
 21     9   139  593*      7   101    83   39*      4   433   419    16
       6,  2,  L           8   856   869     7      5   549   591    13
  0   710   689     7      9   772   787     8      6   254   291    24
  1  1161  1129     5     10   499   502    10      7   240   225    25
  2    53    70   54*     11   952   944     7      8   388   395    16
  3   726   707     6     12   392   384    12      9   344   326    18
  4   138   133    20     13   571   565     9     10   373   345    17
  5   239   235    13     14   449   451    11     11   388   386    16
  6   728   747     6     15   306   338    15     12   302   327    21
  7     7    88  459*     16   394   379    13            6,  8,  L
  8  1328  1324     5     17    97   121   51*      0   408   398    17
  9   140   187    24     18   169   178    30      1   691   675    12
 10  1142  1150     6     19   300   278    18      2   398   421    18
 11   399   404    10            6,  5,  L          3   589   591    13
 12   203   181    19      0   299   325    20      4     9    94  767*
 13   734   717     7      1   471   509    14      5   239   271    30
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
     -10,   0,  L
  2   334   319    29
  4     9    67   691*
  6   264   259    22
  8   403   411    15
     -10,   1,  L
  1   503   487    15
  2   474   469    19
  3    98    61    60*
  4   468   470    13
  5   275   276    20
  6   325   310    17
  7   337   345    17
  8   535   559    12
  9   116    48    46*
     -10,   2,  L
  1   135    84    37*
  2   373   393    15
  3   474   503    12
  4    42   135   126*
  5   491   505    12
  6   267   266    21
  7     9   138   609*
     -10,   3,  L
  1   626   608    14
  2   449   436    21
  3   431   408    17
  4   444   418    14
  5     9    41   643*
      -9,   0,  L
  2   966   992    11
  4   562   572    11
  6   221   208    21
  8   122   148    38*
 10   602   603    10
 12   604   627    10
 14   649   654    10
      -9,   1,  L
  1   419   396    16
  2   115    41    53*
  3   424   401    13
  4   735   745     8
  5   186   203    25
  6   760   778     8
  7   270   308    17
  8   570   586    10
  9   337   337    15
 10   378   408    14
 11   141    82    35*
 12   117    41    42*
 13     9    31   576*
 14   161   179    32
      -9,   2,  L
  1   269   203    18
```

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  L   KFO    FC   SIG
  2  1337  1309     7
  3    65    11    70*
  4   674   695     9
  5   378   394    13
  6   175   182    26
  7   720   732     8
  8   410   430    13
  9   263   269    19
 10   427   437    13
 11   108    80    47*
 12   163   180    32
 13   262   261    21
      -9,   3,  L
  1   323   344    23
  2   124   112    56*
  3   416   359    15
  4   667   662    10
  5   685   702     9
  6   624   628    10
  7   284   278    19
  8   275   270    19
  9   156   275    36*
 10     9    41   590*
 11   709   705    10
 12     9    75   637*
      -9,   4,  L
  1    97   136    72*
  2   526   550    15
  3   292   283    24
  4   315   268    21
  5   210   222    29
  6     9    67   669*
  7   721   721    10
  8   171    72    34
  9   882   861     9
      -9,   5,  L
  1   304   323    24
  2     9    39   783*
  3   560   571    15
  4     9    48   835*
  5   719   719    12
  6   185    88    34
      -8,   0,  L
  2   192   212    32
  4   797   806     8
  6  1067  1083     6
  8   771   771     7
 10   213   235    20
 12   742   780     8
 14   811   826     8
 16   591   601    10
      -8,   1,  L
  1   171   136    33
  2  1240  1213     7
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  L   KFO    FC   SIG
  3   369   360    12
  4   644   674     8
  5   567   566     9
  6   151   189    28
  7   212   190    19
  8   710   729     8
  9   171   196    24
 10  1051  1121     7
 11   194   188    22
 12   414   425    12
 13    80    23    56*
 14   112    22    40*
 15   160    24    30
 16   414   408    13
 17   118    91    42*
      -8,   2,  L
  1   478   458    13
  2   235   225    21
  3   451   476    11
  4  1313  1320     6
  5     8    15   545*
  6  1146  1183     6
  7   263   287    17
  8   355   387    13
  9   447   448    11
 10   411   403    11
 11   190   166    23
 12   572   582    10
 13   126   201    39*
 14   323   322    16
 15    96    29    52*
 16     9   134   576*
      -8,   3,  L
  1   192   230    35
  2  1131  1104     9
  3   144    68    35*
  4   502   482    11
  5   323   328    15
  6   354   341    13
  7   560   556     9
  8   735   771     8
  9   516   535    10
 10   657   638     9
 11   157    57    30
 12    95    88    52*
 13   558   543    11
 14     9   123   604*
 15   749   776     9
      -8,   4,  L
  1   364   418    18
  2   126    95    47*
  3   214   223    28
  4   405   394    15
  5   318   319    17
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -8,   4,  L
  6   359   375    15
  7     8    40   591*
  8   326   325    16
  9   397   386    14
 10     8    81   600*
 11   739   749     9
 12   312   279    18
 13   671   653    10
 14   313   292    19
      -8,   5,  L
  1   239   232    27
  2   200   136    31
  3   295   284    23
  4     8   148   801*
  5   516   491    13
  6   198   124    30
  7   622   596    11
  8    79    44    72*
  9   487   449    13
 10   161   184    36*
 11   133    29    43*
      -8,   6,  L
  1   716   732    12
  2   150    98    42*
  3   404   424    17
  4   145   183    50*
  5     9   108   811*
  6     9    46   769*
  7   340   333    21
  8   159   152    41*
      -7,   0,  L
  2   693   670     9
  4   330   333    12
  6     7   174   528*
  8  1058  1056     6
 10  1220  1244     6
 12   850   838     7
 14   521   550    10
 16  1074  1093     7
 18   738   728     9
 20   161   132    32
      -7,   1,  L
  1   697   639     9
  2   657   664     7
  3   121    89    27*
  4  1108  1149     5
  5   524   539     8
  6  1264  1290     5
  7   621   640     7
  8   509   532     8
  9   251   240    15
 10   289   266    14
 11   289   270    14
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  L   KFO    FC   SIG
 12  1203  1218     6
 13    72   128    59*
 14   983  1005     7
 15     8    14   525*
 16   220   221    21
 17     8    43   556*
 18   348   333    15
 19   142   130    35*
 20   516   515    12
      -7,   2,  L
  1    86   223    66*
  2  1240  1173     6
  3  1037  1041     6
  4   228   224    16
  5   729   738     7
  6   936   941     6
  7     7    39   513*
  8  1259  1289     6
  9   335   341    13
 10   907   918     6
 11   396   397    11
 12   107    57    36*
 13   140   113    29
 14   464   474    11
 15   207   238    22
 16   624   634     9
 17     9    69   590*
 18   350   336    15
 19     9    24   595*
      -7,   3,  L
  1   404   475    16
  2   497   480    13
  3   150    57    30
  4  1012   986     7
  5    49   144    85*
  6   776   760     7
  7   438   428    10
  8    79    23    50*
  9   517   519     9
 10   593   568     9
 11   424   413    11
 12   748   733     8
 13   153   197    29
 14   380   380    13
 15   339   327    15
 16     9    54   594*
 17   636   643    10
 18   152   143    35*
      -7,   4,  L
  1   725   783    11
  2   231   218    26
  3   929   855     9
  4   295   298    18
  5   548   541    10
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  L   KFO    FC   SIG
  6    98    61    46*
  7   406   424    12
  8   344   363    14
  9   111    87    40*
 10   647   638     9
 11   302   290    16
 12   143   105    31*
 13   713   691     9
 14   227   192    22
 15   716   683     9
 16   510   473    12
 17   192   200    29
      -7,   5,  L
  1   741   763    10
  2   129    32    41*
  3     8    64   762*
  4   187   230    33
  5   465   479    14
  6   169   111    31
  7   841   830     9
  8   273   293    20
  9   562   536    11
 10    79    91    63*
 11   388   369    14
 12   230   272    24
 13   172   139    30
 14   392   383    15
 15     9   147   661*
      -7,   6,  L
  1   662   677    11
  2   245   289    25
  3   899  1017    10
  4     8    96   807*
  5   750   708    11
  6   367   372    18
  7     8   140   722*
  8   197    96    32
  9   428   422    15
 10     9   113   704*
 11   462   450    14
 12   169   206    35
      -7,   7,  L
  1   827   842    10
  2   210   193    31
  3   140   105    44*
  4   248   274    27
  5   457   440    16
  6    23    68   313*
  7   739   709    12
  8     9    85   781*
      -6,   0,  L
  2   889   859     6
  4   594   572     6
  6   652   661     6
```

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   COLUMNS ARE 10FO,10FC,  10SIG

L   KFO    FC   SIG       L   KFO    FC   SIG       L   KFO    FC   SIG
    10,   1,  L                10,   2,  L          7    75    62    70*
3   128   113    40*      0   640   676    10            10,   3,  L
4   840   815     8       1   468   478    12       0    28    71   215*
5   343   318    16       2   683   675     9       1   415   367    15
6   996   984     8       3   502   504    12       2   466   451    13
7   258   258    21       4    98    76    52*      3    80   105    67*
8   433   439    13       5   298   265    18       4   677   698    10
9    56    19    95*      6   450   440    13       5    25    77   211*
```

OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR

$CpNiCoFe(CO)_6(C_2Et_2)$

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  COLUMNS ARE 10FO,10FC,  10SIG                                                 COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG
       -9,     1,  L         4    185    183   12       8     99    114   19            -6,     1,  L              -6,     5,  L         9    227    212    9
  1     10     45  227*      5      9     21  227*      9    367    357    7       2    564    570    5       1    236    242    8      11      8    109  254*
  2     85     26   21*      6    206    207   11      10     12      4  173*      3    171    170    9       2    335    344    7      13      9     41  253*
  3    215    211   11       7    334    319    8      11    485    463    7       4   1070   1055    4       3     68     93   24*           -5,     1,  L
  4     10     90  238*      8    263    253   10            -7,     4,  L         5     55     16   27*      4    285    286    7       1   1561   1601    4
  5     78     83   26*           -8,     5,  L         1     21     78   92*      6    114    113   14       5    218    224    9       2     91     39   14
  6     10     10  221*      1    364    375    8       2    109    114   17       7      8     42  194*      6    112    123   16       3   1503   1519    4
       -9,     2,  L         2    216    207   11       3    236    234    9       8    801    823    5       7    219    222    9       4    159    158    8
  2    252    255   10       3      9     54  216*      4    339    336    7       9    196    201   10       8    100    100   18       5     51     34   26*
  3     82     88   26*      4    101     33   19       5    293    294    8      10    477    481    6       9    196    202   11       6    168    172    9
  4     10     21  200*      5    252    255   10       6    246    254    9      11     92     61   21*     10    283    275    8       7    250    256    7
  5    253    262   10       6    494    476    7       7    376    373    7      12     89     68   22*     11      9     34  205*      8     79     16   18*
       -8,     0,  L         7    137    128   16       8    149    150   13      13    211    198   11      12    252    246   10       9    473    465    6
  2     72     30   25*           -7,     0,  L         9    205    197   11            -6,     2,  L              -6,     6,  L        10    347    354    7
  4    304    308    8       1    485    488    6      10    103     55   18       1    469    477    5       1    204    204   10      11    481    489    6
  6    125    130   16       3    357    369    7      11    199    205   12       2   1014   1035    5       2    215    211    9      12    144    140   14
  8    346    341    8       5    253    253    8            -7,     5,  L         3    163    165   10       3    202    202   10      13    504    502    7
 10    228    225   11       7    698    705    5       1    570    587    6       4    302    314    6       4    287    280    8      14    212    194   11
       -8,     1,  L         9   1015   1000    6       2    324    321    8       5     56     66   26*      5    359    361    7            -5,     2,  L
  1    250    247    9      11    249    235   10       3     40     23   45*      6    106    134   16       6    416    418    7       1    193    192    8
  2    312    321    8            -7,     1,  L         4    350    350    7       7    181    178   10       7     68     64   25*      2    197    202    7
  3      9     49  215*      1    555    551    6       5    111    117   18       8    238    241    8       8    230    236   10       3    735    751    4
  4    301    307    8       2    372    368    7       6      9      7  210*      9      8      9  204*      9    248    236    9       4    764    757    4
  5    119    121   17       3    704    692    5       7    344    346    8      10    604    595    6      10     69     56   27*      5   1092   1104    4
  6     98     26   18       4    145    146   12       8    218    218   10      11    325    323    8      11     10     42  219*      6    284    280    6
  7     83     17   23*      5     99     43   17       9    279    269    9      12    367    365    8            -6,     7,  L         7    101    105   14
  8    404    405    7       6    209    205    9      10    177    177   13      13      9     34  226*      1    171    180   12       8    455    460    5
  9    115    142   19       7    333    340    7            -7,     6,  L              -6,     3,  L         2    144    155   13       9      8    103  223*
 10    585    566    7       8      9     51  217*      1    216    227   10       1    183    186    9       3    256    260    9      10    232    227    9
       -8,     2,  L         9    162    159   13       2    194    201   11       2    368    370    6       4    108     27   17      11      8      8  204*
  1     94     69   20      10     63     62   32*      3     83     61   22*      3    274    272    7       5    420    412    7      12    335    324    8
  2    277    283    9      11    732    709    6       4     92    118   23*      4    269    283    7       6    145    134   14      13      9     84  233*
  3    445    452    7      12    176    156   13       5    343    347    8       5     81     78   18*      7    451    443    7      14     62     55   33*
  4     31     54   65*           -7,     2,  L         6    438    455    7       6     82     49   19*      8      9     42  214*           -5,     3,  L
  5     13     20  154*      1    149    148   13       7    238    242   10       7     90     55   17       9    118    109   17       1   1006   1033    4
  6    140    132   14       2      8     70  214*      8    185    175   12       8    263    252    8            -6,     8,  L         2    600    592    4
  7    333    329    8       3    124    149   15       9     10     44  222*      9    392    395    7       1    376    373    7       3    819    831    4
  8    119    110   17       4    601    590    6            -7,     7,  L        10    392    384    7       2    275    272    9       4     94     93   14
  9     90     72   23*      5    139    152   13       1    386    401    7      11     96     89   20       3    196    188   11       5    303    314    6
 10    378    368    8       6    144    127   13       2      9     79  216*     12    147    157   15       4    236    216   10       6    555    565    5
       -8,     3,  L         7    474    481    6       3     56     61   34*     13    298    292    9       5    387    385    7       7    198    189    8
  1    468    472    7       8    281    270    8       4    373    384    8            -6,     4,  L         6    102     83   19       8     36     57   43*
  2      9     42  228*      9    641    629    6       5     33     83   62*      1    521    524    5       7      9    140  230*      9    311    305    7
  3    131    133   15      10     71     38   26*      6    109    115   19       2    490    508    6       8    163    152   13      10    589    605    6
  4      9     42  211*     11    215    209   11       7    304    301    9       3      8     66  211*           -6,     9,  L        11    441    446    7
  5    258    255    9      12     67     44   30*           -6,     0,  L         4    212    216    8       1    244    243   10      12    156    156   13
  6    332    328    8            -7,     3,  L         2   1706   1743    4       5    253    254    8       2     66     47   32*     13    440    436    7
  7     91     57   20*      1    766    800    5       4    361    379    6       6    310    321    7       3    335    333    8      14    353    337    8
  8    566    556    7       2    699    722    5       6    558    568    5       7    130    126   14       4     47     28   43*           -5,     4,  L
  9    278    271    9       3    218    220    9       8     92     42   17       8    251    255    8            -5,     0,  L         1    205    194    8
       -8,     4,  L         4    341    347    7      10    638    649    6       9      9     74  217*      1    790    743    4       2      7      1  193*
  1    247    249   10       5      8    135  224*     12    418    414    7      10    181    180   12       3   1245   1246    4       3    185    196    8
  2    426    427    7       6    374    388    7            -6,     1,  L        11    295    288    9       5   1605   1554    4       4    563    574    5
  3    453    462    7       7     90     58   20*      1      7     41  201*     12    431    433    7       7    250    247    7       5    683    702    5
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COLUMNS ARE 10FO,10FC, 10SIG

L	KFO	FC	SIG
	-5,	4,	L
6	157	158	10
7	490	490	5
8	505	502	6
9	241	244	8
10	363	365	7
11	362	371	7
12	316	314	8
13	9	16	213*
	-5,	5,	L
1	149	160	10
2	185	193	9
3	438	453	5
4	19	31	75*
5	882	891	5
6	143	152	12
7	197	200	9
8	8	9	213*
9	182	165	11
10	342	348	7
11	415	398	7
12	137	138	15
13	508	477	7
	-5,	6,	L
1	451	462	6
2	449	446	6
3	152	158	11
4	87	63	19*
5	99	76	18
6	367	370	6
7	484	484	6
8	270	262	8
9	9	11	200*
10	86	53	22*
11	269	243	9
12	139	150	16
	-5,	7,	L
1	274	271	7
2	76	72	21*
3	224	232	9
4	451	477	6
5	474	474	6
6	9	22	214*
7	287	302	8
8	206	194	10
9	45	46	44*
10	88	49	22*
11	285	276	9
	-5,	8,	L
1	243	245	9
2	419	426	7
3	252	261	9
4	125	124	16
5	139	139	14

L	KFO	FC	SIG
6	349	352	7
7	9	68	224*
8	137	139	14
9	9	21	212*
	-5,	9,	L
1	250	241	9
2	31	48	55*
3	217	218	10
4	332	340	8
5	53	19	36*
6	76	48	27*
7	200	184	11
	-5,	10,	L
1	105	92	21
2	413	403	8
3	225	229	10
4	77	92	27*
	-4,	0,	L
2	820	842	4
4	578	575	4
6	81	104	15
8	83	111	16
10	174	175	10
12	487	486	6
14	731	702	6
	-4,	1,	L
1	543	553	4
2	736	770	4
3	6	17	180*
4	980	969	4
5	438	444	4
6	502	500	4
7	112	124	12
8	371	372	5
9	149	147	10
10	263	276	8
11	238	250	8
12	70	65	27*
13	9	24	204*
14	177	184	13
15	10	93	230*
	-4,	2,	L
1	482	503	4
2	761	770	4
3	64	24	18*
4	957	954	4
5	570	599	4
6	160	165	8
7	7	120	197*
8	7	52	194*
9	718	729	5
10	230	245	8
11	278	278	8
12	284	288	8

L	KFO	FC	SIG
13	179	176	12
14	531	508	7
15	128	129	18
	-4,	3,	L
1	368	388	5
2	93	121	12
3	820	838	4
4	1038	1056	4
5	568	579	4
6	1115	1130	4
7	615	600	5
8	318	322	6
9	384	380	6
10	158	168	11
11	369	364	7
12	97	49	18
13	83	90	25*
14	275	266	9
	-4,	4,	L
1	706	709	4
2	779	796	4
3	334	338	5
4	1126	1116	4
5	384	393	5
6	30	44	45*
7	491	493	5
8	357	346	6
9	527	526	5
10	282	293	8
11	465	459	6
12	568	568	6
13	85	83	24*
14	297	296	9
	-4,	5,	L
1	204	206	7
2	391	404	5
3	493	498	5
4	598	601	5
5	64	82	23*
6	1117	1110	4
7	489	494	5
8	340	353	7
9	314	320	7
10	214	219	10
11	211	213	10
12	9	38	235*
13	150	169	15
	-4,	6,	L
1	615	609	5
2	176	180	9
3	234	239	7
4	995	1021	5
5	465	465	5
6	227	223	8

COLUMNS ARE 10FO,10FC, 10SIG

L	KFO	FC	SIG
	-4,	6,	L
7	100	108	16
8	356	350	7
9	262	265	8
10	219	221	10
11	304	313	8
12	496	502	7
13	158	154	15
	-4,	7,	L
1	486	496	5
2	144	139	11
3	358	360	6
4	362	353	6
5	101	102	17
6	718	711	5
7	58	155	35*
8	290	292	8
9	211	208	10
10	103	92	19
11	162	158	13
12	141	135	16
	-4,	8,	L
1	279	279	7
2	8	116	228*
3	388	394	7
4	209	209	9
5	8	31	202*
6	144	156	14
7	48	8	38*
8	132	127	15
9	130	122	16
10	296	279	9
	-4,	9,	L
1	452	440	6
2	519	508	6
3	187	195	11
4	288	291	8
5	167	138	12
6	9	42	225*
7	123	122	16
8	61	101	32*
9	172	172	13
	-4,	10,	L
1	209	218	11
2	75	123	28*
3	150	155	14
4	267	274	9
5	145	153	15
6	195	198	12
7	10	40	249*
	-3,	0,	L
1	1863	1803	3
3	80	78	14
5	836	814	3

L	KFO	FC	SIG
7	1428	1464	4
9	548	561	5
11	316	312	7
13	80	111	27*
15	507	482	7
	-3,	1,	L
1	810	801	3
2	718	696	3
3	447	421	4
4	139	127	8
5	383	387	4
6	321	317	5
7	164	161	8
8	482	504	5
9	450	462	5
10	19	119	83*
11	161	160	11
12	166	183	12
13	691	662	6
14	86	53	25*
15	417	379	8
	-3,	2,	L
1	968	955	3
2	627	635	3
3	682	674	3
4	911	917	3
5	908	926	4
6	346	351	5
7	1004	1037	4
8	437	439	5
9	475	479	5
10	156	169	11
11	177	171	10
12	87	75	22*
13	87	100	23*
14	9	99	236*
15	412	378	8
	-3,	3,	L
1	331	322	4
2	963	948	3
3	960	951	3
4	477	482	4
5	328	334	5
6	807	843	4
7	225	235	7
8	497	492	5
9	359	349	6
10	477	494	6
11	8	10	215*
12	8	75	225*
13	376	362	7
14	38	42	55*
15	291	269	10
	-3,	4,	L

L	KFO	FC	SIG
1	300	295	5
2	718	708	4
3	250	254	6
4	963	974	4
5	1316	1362	4
6	405	411	5
7	913	940	4
8	697	703	5
9	124	129	13
10	127	129	13
11	59	71	30*
12	173	178	12
13	372	353	8
14	106	45	19
	-3,	5,	L
1	66	28	17*
2	669	671	4
3	1143	1165	4
4	201	209	7
5	129	138	11
6	792	787	5
7	593	606	5
8	69	78	22*
9	28	70	59*
10	433	423	6
11	361	364	7
12	138	131	15
13	159	166	14
14	51	39	41*
	-3,	6,	L
1	479	480	5
2	847	857	4
3	383	396	5
4	370	358	6
5	935	953	4
6	160	163	10
7	537	548	5
8	291	283	7
9	81	86	22*
10	146	145	13
11	349	358	8
12	93	84	22*
13	256	244	10
	-3,	7,	L
1	128	125	12
2	7	28	186*
3	583	594	5
4	96	109	16
5	8	10	207*
6	332	333	7
7	287	292	7
8	335	336	7
9	73	123	28*
10	201	204	11

COLUMNS ARE 10FO,10FC, 10SIG

COLUMNS ARE 10FO,10FC, 10SIG

L	KFO	FC	SIG	L	KFO	FC	SIG	L	KFO	FC	SIG	L	KFO	FC	SIG	L	KFO	FC	SIG	L	KFO	FC	SIG
	-3,	7, L		5	195	193	6	11	46	60	39*		-2,	8, L		13	69	80	28*	5	643	640	4
11	300	299	9	6	1208	1221	3	12	22	112	94*	8	97	30	18	14	202	191	12	6	154	169	9
12	409	416	8	7	84	111	15	13	122	122	18	9	143	156	14	15	77	24	27*	7	98	115	15
	-3,	8, L		8	1488	1553	4	14	9	69	248*	10	9	1	223*		-1,	2, L		8	414	423	6
1	336	340	6	9	127	134	12		-2,	5, L		11	375	370	8	1	337	326	3	9	241	236	8
2	91	103	18	10	198	198	9	1	176	181	7		-2,	9, L		2	491	476	3	10	173	172	11
3	541	549	6	11	20	59	87*	2	74	47	15	1	353	347	7	3	229	232	5	11	470	467	7
4	184	176	10	12	158	148	12	3	134	142	9	2	440	445	6	4	79	21	14	12	327	316	8
5	342	344	7	13	246	242	9	4	1130	1179	4	3	8	22	203*	5	291	294	5	13	226	227	11
6	391	392	6	14	268	253	10	5	209	212	7	4	82	101	22*	6	72	84	16*	14	110	124	21
7	214	214	10	15	72	44	29*	6	620	617	5	5	570	554	6	7	999	1030	4		-1,	6, L	
8	195	196	11		-2,	2, L		7	7	72	194*	6	237	232	9	8	531	543	4	1	61	26	18*
9	199	190	11	1	451	452	3	8	168	173	10	7	9	72	215*	9	792	799	5	2	389	377	5
10	396	394	7	2	54	68	19*	9	8	32	201*	8	324	324	8	10	138	137	13	3	991	974	4
11	334	326	8	3	1224	1209	3	10	525	521	6	9	373	357	8	11	41	9	44*	4	449	459	5
	-3,	9, L		4	223	228	6	11	8	73	230*	10	275	263	9	12	464	469	7	5	309	319	6
1	42	61	42*	5	157	168	8	12	196	187	11		-2,	10, L		13	9	33	241*	6	494	487	5
2	50	42	35*	6	524	529	4	13	9	16	227*	1	207	207	11	14	109	83	20	7	197	206	9
3	300	306	8	7	685	679	4	14	94	29	22*	2	492	494	6	15	207	189	12	8	8	103	232*
4	81	140	25*	8	612	631	4		-2,	6, L		3	450	441	7		-1,	3, L		9	550	556	6
5	82	48	20*	9	299	298	6	1	571	572	4	4	143	139	14	1	511	498	3	10	304	316	8
6	108	103	18	10	824	838	5	2	448	433	5	5	9	16	193*	2	293	294	5	11	268	262	9
7	287	292	9	11	346	335	7	3	448	457	5	6	175	181	13	3	323	332	4	12	89	9	22*
8	302	299	8	12	74	60	25*	4	7	31	184*	7	339	326	8	4	157	154	7	13	54	56	42*
9	191	187	12	13	31	27	62*	5	252	263	7	8	72	98	30*	5	138	147	9		-1,	7, L	
10	111	122	19	14	190	188	13	6	486	503	5		-2,	11, L		6	261	265	6	1	208	219	7
	-3,	10, L		15	159	162	15	7	538	542	5	1	113	27	17	7	391	383	5	2	768	788	4
1	702	686	6		-2,	3, L		8	64	123	28*	2	276	276	9	8	311	318	6	3	178	176	9
2	241	240	9	1	956	961	3	9	71	88	24*	3	100	100	21	9	503	542	5	4	391	403	6
3	280	275	9	2	267	270	5	10	8	14	209*	4	69	15	28*	10	477	483	6	5	520	537	5
4	53	49	36*	3	1356	1350	3	11	246	244	9	5	221	222	11	11	632	610	6	6	524	536	5
5	9	70	202*	4	588	597	4	12	147	151	15	6	313	314	9	12	318	336	8	7	389	393	6
6	32	35	61*	5	6	59	190*	13	214	215	11		-1,	0, L		13	232	224	10	8	497	510	6
7	283	272	9	6	1301	1322	4		-2,	7, L		1	350	339	2	14	219	215	11	9	96	81	20
8	58	27	34*	7	217	230	7	1	267	273	7	3	1218	1232	2		-1,	4, L		10	170	175	12
	-3,	11, L		8	684	729	4	2	357	352	5	5	519	533	4	1	621	600	3	11	259	256	10
1	9	18	219*	9	71	39	20*	3	236	240	7	7	1739	1773	4	2	753	721	3	12	267	277	10
2	56	121	39*	10	158	162	11	4	730	730	5	9	831	841	4	3	202	208	6		-1,	8, L	
3	343	342	8	11	45	45	37*	5	830	835	5	11	235	231	8	4	143	153	8	1	8	15	197*
4	23	71	89*	12	113	100	17	6	497	509	5	13	81	68	25*	5	303	318	5	2	271	280	7
5	88	60	24*	13	233	231	10	7	8	44	216*	15	278	263	10	6	483	493	5	3	298	295	7
	-2,	0, L		14	174	180	14	8	128	145	14		-1,	1, L		7	500	530	5	4	857	863	5
2	773	764	3	15	42	63	54*	9	567	573	6	1	2798	2727	2	8	534	543	5	5	290	296	8
4	255	246	5		-2,	4, L		10	412	415	7	2	279	274	4	9	686	719	5	6	301	304	7
6	683	687	4	1	1231	1248	3	11	109	66	20	3	100	95	10	10	281	279	8	7	150	148	13
8	848	858	4	2	544	546	4	12	217	212	11	4	71	77	16*	11	272	265	8	8	537	537	6
10	780	797	5	3	365	371	4		-2,	8, L		5	1484	1526	3	12	395	395	7	9	305	310	8
12	330	322	7	4	417	409	4	1	140	126	11	6	892	867	4	13	40	59	54*	10	219	205	11
14	182	176	13	5	764	768	4	2	166	159	10	7	79	104	15	14	218	206	11	11	259	271	10
	-2,	1, L		6	379	388	5	3	707	710	5	8	251	261	6		-1,	5, L			-1,	9, L	
1	150	138	7	7	24	68	54*	4	171	174	10	9	1030	1049	4	1	60	70	18*	1	354	345	7
2	592	574	3	8	431	442	5	5	59	56	28*	10	633	627	5	2	372	364	5	2	719	729	5
3	210	207	6	9	232	236	8	6	138	124	13	11	549	537	5	3	604	594	4	3	470	470	6
4	688	703	3	10	223	229	9	7	670	643	6	12	52	33	35*	4	360	368	5	4	407	407	6

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COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG
      -1,   9,  L            4    501    524     4     12      9    114  254*
  5    217    211    10      5   1472   1507     3     13    104    '65    20
  6    469    474     6      6     15     38   76*            0,   6,  L
  7    170    169    12      7    116    106    11      0     81     51    14
  8    408    406     7      8    493    487     5      1    170    158     8
  9    123    121    17      9    880    892     5      2    621    657     4
 10    221    230    11     10    696    712     5      3    818    819     4
      -1,  10,  L           11     80     82   21*      4    755    763     4
  1    246    251     9     12    438    437     7      5     65     72   20*
  2    160    185    13     13    343    346     8      6    282    291     7
  3     80     81   23*     14     67    103   33*      7    491    501     5
  4    507    506     6            0,   3,  L            8    125    137    14
  5    291    283     8      1    175    163     6      9    287    296     8
  6    363    356     8      2    663    654     3     10    541    555     6
  7    325    315     8      3    114    114    10     11    123    127    17
  8    196    196    12      4    615    631     4     12    427    418     7
      -1,  11,  L            5    374    359     4     13     10    152  263*
  1    491    475     7      6    342    355     5            0,   7,  L
  2    343    351     8      7    643    660     4      1    384    391     5
  3    243    250    10      8    595    642     5      2    395    397     5
  4    120    124    17      9    394    404     6      3    478    477     5
  5     87     20   24*     10    291    303     8      4    249    247     7
  6    166    161    14     11    622    624     6      5    507    522     5
      -1,  12,  L           12     91     24   22*      6    209    224     9
  1    290    291    10     13    100    113    20      7     69    135   26*
       0,   0,  L           14      9    105  257*      8    317    314     8
  2   1672   1625     2            0,   4,  L            9    224    246    10
  4   2220   2211     3      0    943    900     3     10    403    413     7
  6    334    319     5      1    374    368     4     11     70     39   32*
  8    872    900     4      2    877    871     3     12     10     13  237*
 10    544    571     5      3    617    602     4            0,   8,  L
 12    405    394     7      4    750    741     4      0    181    175     9
 14    275    272     9      5    181    210     7      1    469    471     5
       0,   1,  L            6    152    158     9      2    514    522     5
  1    181    176     4      7    145    169    10      3    676    676     5
  2   2198   2254     2      8     90    130    17      4    546    537     5
  3    167    171     6      9    406    412     6      5    311    312     7
  4     68     15   17*     10    791    811     5      6    269    269     8
  5    239    228     6     11    172    187    12      7    424    445     7
  6   1315   1325     4     12    530    561     7      8    103    113    19
  7    514    522     4     13    292    274     9      9     55     63   37*
  8    595    602     5     14    114     35    20     10    308    312     9
  9     98     52    16            0,   5,  L           11     24     60   87*
 10    143    155    12      1    289    272     5            0,   9,  L
 11    380    370     6      2    366    372     5      1    589    595     6
 12     85     81   22*      3      6     82  211*      2     13     46  133*
 13     90     16   22*      4    613    617     4      3    508    509     6
 14     74     69   30*      5    265    270     6      4      8     21  218*
 15    158    146    15      6    133    145    11      5    575    565     6
       0,   2,  L            7    545    559     5      6     84    103   23*
  0   2900   2813     2      8    542    558     5      7     73     64   26*
  1    272    265     4      9    131    146    14      8     10     74  194*
  2   1156   1148     2     10    479    495     6      9     90    134   24*
  3    559    585     3     11    482    484     7     10     10     94  245*
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COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO     FC   SIG      L    KFO     FC   SIG      L    KFO     FC   SIG
       0,  10,  L            9      7     27  217*      1    726    697     4
  0    433    431     7     10    486    496     6      2     93    119    15
  1    350    354     7     11    253    254     9      3    459    486     5
  2    262    260     9     12    115    133    18      4    487    490     5
  3    505    512     6     13    187    188    13      5    897    919     5
  4    110    134    18     14    113    119    21      6    409    409     6
  5    107    111    19            1,   3,  L            7     81     23   20*
  6    442    420     7      0   1492   1427     3      8     87    104   21*
  7    350    355     8      1   1299   1276     3      9    371    371     7
  8    267    264    10      2    293    296     5     10     16    121  139*
       0,  11,  L            3   1421   1457     3     11    180    184    13
  1    166    157    13      4    858    907     4     12    123    114    18
  2    100    111    20      5    456    442     4            1,   7,  L
  3     84    122   24*      6     50     27   26*      0     86     84    16
  4    304    310     9      7    223    224     7      1    806    815     4
  5    255    256    10      8    490    491     5      2    695    694     5
  6    205    191    12      9    516    541     6      3    660    667     5
       0,  12,  L           10      8     48  235*      4     97     18    15
  0    230    222    11     11    570    565     6      5    121    129    14
  1     47     10   40*     12    336    343     8      6    100    130    18
       1,   0,  L           13    322    314     9      7     13     17  137*
  1   1804   1682     2     14     10     28  213*      8    114    140    17
  3    839    770     3            1,   4,  L            9    500    499     7
  5    540    527     4      0    253    248     5     10    234    233    11
  7     71     41   21*      1    649    625     3     11    360    351     8
  9    147    149    12      2    281    291     5            1,   8,  L
 11    361    371     7      3   1317   1337     3      0    655    644     5
 13    362    351     8      4    214    215     7      1      8     25  198*
       1,   1,  L            5    633    666     4      2    282    279     7
  0    538    516     2      6    832    877     4      3    432    423     6
  1   1956   1819     2      7    338    336     6      4    301    305     7
  2      4     34  249*      8    245    241     8      5    255    243     8
  3    894    874     3      9    426    451     6      6     51     44   34*
  4    618    606     4     10    566    553     6      7      9     38  214*
  5    188    185     7     11    123    108    17      8    134    125    16
  6    174    174     8     12    215    230    11      9     91    109   26*
  7    784    780     4     13    184    184    13     10    152    156    15
  8    359    366     6            1,   5,  L                   1,   9,  L
  9    315    305     6      0    352    364     4      0    143    138    13
 10    209    202     9      1    838    837     4      1    346    343     7
 11    518    505     6      2    267    264     5      2    578    559     6
 12    193    217    12      3   1230   1243     4      3    258    247     8
 13    620    604     6      4     94     94    13      4      8      2  217*
 14     87     81   26*      5      7     20  188*      5    277    271     8
       1,   2,  L            6     35     54   41*      6     96    103    21
  0    199    209     5      7    166    175    10      7    252    260    10
  1    787    734     3      8    214    221     9      8    110    114    19
  2   1182   1159     3      9    794    790     5      9    176    185    13
  3   1075   1019     3     10    193    191    11            1,  10,  L
  4    577    555     4     11    536    543     7      0    471    475     6
  5    298    294     5     12    114     94    19      1    339    343     8
  6    830    859     4     13     91    102   26*      2      9      4  212*
  7     72     65   19*            1,   6,  L            3    103     92    19
  8      7     79  219*      0    538    526     4      4    292    304     9
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   COLUMNS ARE 10FO,10FC,  10SIG                                                    COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO     FC    SIG     L    KFO     FC    SIG     L    KFO     FC    SIG     L    KFO     FC    SIG     L    KFO     FC    SIG     L    KFO     FC    SIG
     1, 10,  L              2    925    881      4         2,  7,  L                  3,  1,  L             11     67     80    33*     4    171    167     13
 5    297    283      9     3    385    366      5     0     79     74    18*     0    345    336      4    12     10     21   229*     5    189    194     12
 6    267    263      9     4   1080   1055      4     1    395    372      6     1    212    203      6         3,  5,  L              6    405    407      8
 7    215    207     11     5   1108   1086      4     2    340    338      6     2     53     55    21*     0    625    587      4     7      9     62   245*
     1, 11,  L              6    629    608      5     3    236    231      8     3    666    636      4     1    757    749      4         3, 10,  L
 0    107     20     19     7    433    435      5     4    716    737      5     4    163    144      8     2    279    266      7     0     88    116    24*
 1    104     75     19     8      8     41   214*     5    127    141     14     5    184    195      8     3     99    139     17     1    345    336      8
 2    203    221     12     9    403    421      7     6    291    294      8     6    297    281      6     4    513    480      5     2    232    221     10
 3      9     95   216*    10     43    131    47*     7    321    322      8     7    697    697      5     5     14     21   122*     3    126    137     18
 4    149    139     14    11     59     40    34*     8    105     90     20     8     97    118     19     6      8     30   235*     4    236    242     11
 5    407    408      8    12    107     96     21     9    464    487      7     9    347    337      7     7    271    277      8     5     41    164    58*
     1, 12,  L             13      9     18   232*    10    170    172     15    10    151    139     14     8    117    119     18         3, 11,  L
 0    103     14     20         2,  4,  L                  2,  8,  L             11    542    543      7     9    192    205     12     0    285    286      9
     2,  0,  L              0    683    678      4     0    337    333      6    12      9     23   257*    10    138    141     17     1    255    260     10
 0    634    637      3     1    324    328      5     1      8     40   209*         3,  2,  L             11      9      9   226*     2     74    104    30*
 2    316    312      4     2   1566   1541      4     2    400    394      6     0    834    798      3         3,  6,  L                  4,  0,  L
 4    168    166      8     3    699    697      4     3    264    275      8     1    321    305      5     0    212    220      8     0    916    900      4
 6     72     62    18*     4    238    234      7     4    238    246      9     2    460    456      4     1    534    530      5     2    992    962      4
 8      7     37   204*     5    227    219      7     5    231    234      9     3    777    758      4     2    730    747      5     4    881    878      4
10    452    457      6     6    249    248      7     6    119    133     17     4    412    393      5     3   1149   1150      5     6    590    589      5
12    843    841      6     7    502    511      5     7    292    299      9     5   1321   1328      4     4    451    455      6     8    766    753      5
     2,  1,  L              8    323    320      7     8    216    208     11     6    159    154     10     5    488    514      6    10    163    150     13
 0    584    554      3     9    176    179     12     9    260    257     10     7    799    791      5     6    300    297      8         4,  1,  L
 1    243    226      5    10    461    468      7         2,  9,  L              8    336    328      7     7    140    170     16     0    662    633      4
 2    166    171      7    11     97    147    22*     0    575    559      6     9    102     50     20     8    392    403      8     1     24     28    48*
 3      5     32   229*    12    321    311      9     1     28     32    64*    10    128    127     16     9    259    248     10     2     28     57    46*
 4    980    953      4    13    125    101     18     2    334    349      7    11     92     99    22*    10     81     69    27*     3    333    338      5
 5     38     43    33*         2,  5,  L              3      8     29   221*    12     76     68    28*         3,  7,  L              4    959    937      4
 6   1164   1136      4     0    411    394      4     4    152    155     13         3,  3,  L              0    726    704      5     5    135    123     12
 7    214    215      8     1    892    902      4     5     84     97    25*     0    501    496      4     1    477    472      5     6   1283   1271      5
 8      7     47   215*     2    868    902      4     6     55     30    37*     1    857    828      4     2    512    517      6     7    214    204      9
 9    146    131     12     3    266    269      7     7    160    172     14     2    843    828      4     3     40     37    44*     8     30    148    72*
10    380    379      7     4   1364   1406      4     8    215    211     12     3   1129   1062      4     4    377    393      7     9     89     55    22*
11     22     40    89*     5    508    529      5         2, 10,  L              4    669    662      4     5      8     39   231*    10      9     57   227*
12     76     88    28*     6    162    174     11     0    240    238     10     5      7     15   215*     6    516    522      7    11     93     82    24*
13     25      5    89*     7    513    521      6     1     52     29    37*     6    431    440      6     7    223    207     11         4,  2,  L
     2,  2,  L              8    172    160     12     2    110    144     18     7    635    615      5     8    227    226     11     0    276    281      6
 0    224    219      6     9      8     41   223*     3      9     38   228*     8     85     71    21*     9     93    150    27*     1    185    202      7
 1    319    312      5    10    214    205     11     4    181    177     12     9     94     83    21*    10    482    460      8     2    803    780      4
 2    934    906      3    11     62    112    37*     5    175    172     13    10    117    138     19         3,  8,  L              3     34     25    40*
 3    637    618      4    12    118    135     19     6    320    321      9    11    363    374      8     0    141    149     13     4    604    587      5
 4    803    780      4         2,  6,  L                  2, 11,  L             12    238    252     11     1    457    460      6     5    187    190      9
 5    234    237      7     0    494    489      5     0    488    467      7         3,  4,  L              2    258    254      8     6    503    495      6
 6    271    282      7     1     21     63    61*     1      9     55   231*     0    679    649      4     3    334    342      7     7    232    221      9
 7    694    683      5     2   1062   1069      4     2     46     52    46*     1    473    473      5     4    661    674      6     8    683    688      6
 8    166    147     10     3    181    179      9     3     56    131    40*     2    463    455      5     5    166    191     14     9    174    164     13
 9    285    280      7     4    120    115     13     4    236    231     10     3   1381   1350      4     6    107    144     21    10     77     14    26*
10    273    265      8     5    246    261      8         3,  0,  L              4     57     75    27*     7     88    135    27*    11      9    121   255*
11    272    288      9     6      8     14   214*     1    408    400      4     5    836    844      5     8    592    584      7         4,  3,  L
12    674    679      6     7     92     57    20*     3    430    417      4     6    239    231      8         3,  9,  L              0    296    274      5
13      9      4   247*     8    494    495      6     5   1650   1685      4     7    333    318      7     0    296    282      8     1    484    484      5
     2,  3,  L              9    322    314      8     7    845    846      5     8    108    135     18     1    169    169     12     2    420    394      5
 0    904    838      3    10    281    282      9     9    142    143     14     9    180    179     12     2    435    443      7     3     85     88     17
 1   2352   2217      3    11    226    240     12    11      9     40   244*    10     60     92    33*     3      9      8   243*     4    725    702      5
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COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC  SIG        L   KFO    FC  SIG        L   KFO    FC  SIG
      4,    3,  L          0    63   100   32*       3     8    58  233*
 5     8    63  222*       1   769   784    6        4   350   353    7
 6   872   845    5        2   247   274   10        5   122   137   16
 7   107   138   20        3    61    79   36*       6   182   185   12
 8   400   406    7        4     9    63  258*       7   299   315    9
 9    75   141   30*       5   657   653    7        8   315   309    9
10     9    30  250*       6    80    35   28*       9   326   323    9
11    88    61   24*       7     9    42  255*            5,    4,  L
      4,    4,  L               4,    9,  L          0    88    85   18
 0     7     9  200*       0   386   378    7        1   288   291    7
 1   151   150   10        1     9    60  233*       2   100   115   17
 2   331   307    6        2    66    71   32*       3    56    47   30*
 3   117   110   14        3   526   523    7        4   264   267    8
 4    54    16   30*       4   112    80   20        5   285   284    8
 5    77    66   23*       5    73    33   31*       6   233   224   10
 6   194   184   11             4,   10,  L          7   606   585    6
 7   246   246   10        0   354   342    8        8   161   176   16
 8    84    95   25*       1   348   332    8        9   210   213   13
 9     9    27  250*       2    94    71   23*            5,    5,  L
10   164   165   15        3    10    66  243*       0   123   128   14
      4,    5,  L               5,    0,  L          1   565   562    5
 0   308   310    6        1   775   799    4        2   362   357    7
 1    66    36   24*       3   476   482    5        3   495   482    6
 2   975   938    5        5   928   939    5        4     8    85  245*
 3   406   403    6        7    93    77   22*       5   298   292    9
 4   433   420    6        9   150   131   15        6   223   231   11
 5   178   181   12             5,    1,  L          7    57    55   40*
 6   295   305    9        0   155   153    9        8   132   143   19
 7    80   145   29*       1    85    96   16             5,    6,  L
 8   542   535    7        2   310   314    6        0   253   241    8
 9   149   137   16        3   328   318    6        1   511   521    6
10   120   135   20        4   225   230    8        2   411   406    7
      4,    6,  L          5   303   293    7        3    94     8   21*
 0   276   276    7        6    88    87   22*       4   298   290    9
 1   510   497    6        7   452   447    7        5   116   156   20
 2   489   463    6        8   202   201   11        6   137   156   18
 3     8    17  235*       9   166   170   14        7   506   496    7
 4   303   293    8       10     9    12  249*       8   230   242   12
 5   506   493    6             5,    2,  L               5,    7,  L
 6   279   277    9        0   328   324    6        0   711   690    5
 7   124   143   20        1   298   318    6        1   447   422    7
 8   103   111   24*       2   321   321    6        2   323   310    8
 9   289   310   10        3   194   197    9        3    85   166   29*
      4,    7,  L          4   143   144   13        4   452   448    7
 0   420   401    7        5   683   673    5        5   119    73   20
 1   168   156   12        6   341   335    7        6   259   271   11
 2   586   569    6        7   358   356    8        7    97   106   26*
 3   829   827    6        8   113   118   19             5,    8,  L
 4   292   283    9        9   152   136   15        0    17    60  132*
 5    93    10   21*      10   239   239   11        1     9    43  268*
 6   244   243   11             5,    3,  L          2   449   439    8
 7   526   517    7        0   414   409    5        3     9    31  273*
 8   392   391    8        1   128   112   12        4    81    67   30*
      4,    8,  L          2   163   166   10        5   153   170   17
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COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC  SIG        L   KFO    FC  SIG        L   KFO    FC  SIG
      5,    9,  L               6,    5,  L          2   590   594    6
 0   526   500    7        0   369   373    7        3     9    18  244*
 1   286   275   10        1     8    89  240*       4     9    35  244*
 2   150   141   17        2   304   300    8        5    50    15   44*
 3   196   180   13        3   242   245   10        6   364   362    9
      5,   10,  L          4    78    30   26*            7,    4,  L
 0   199   209   13        5   291   303    9        0   387   395    7
      6,    0,  L          6   372   384    8        1   444   454    7
 0   648   635    5        7    74    39   30*       2   194   201   12
 2     8     9  201*            6,    6,  L          3   314   325    9
 4   268   272    8        0   650   627    6        4   419   423    8
 6   225   212   10        1   415   403    7        5   128   133   19
 8   232   235   11        2   462   446    7             7,    5,  L
      6,    1,  L          3   111   103   20        0   149   149   15
 0   689   682    5        4   115   135   21        1   645   619    6
 1   326   324    6        5    77   107   31*       2   371   370    8
 2    79    79   22*       6    10    51  269*       3     9     5  249*
 3     8     7  227*            6,    7,  L          4   102   116   23*
 4   176   190   12        0     9    34  233*            7,    6,  L
 5   348   340    8        1   105   113   22        0   318   311    9
 6   111   115   19        2   194   202   13        1   280   284   10
 7     9    19  233*       3   171   167   14        2    47   113   52*
 8   326   323    9        4    84    82   27*       3   280   277   10
      6,    2,  L               6,    8,  L               7,    7,  L
 0   557   547    5        0   398   403    8        0   233   241   11
 1   284   283    7        1   345   331    9        1   445   447    8
 2   353   363    7        2   242   225   11             8,    0,  L
 3   487   484    6        3   143   120   17        0   336   341    8
 4   221   227   10             7,    0,  L          2   501   508    7
 5   111    79   18        1   212   218   10        4    68    78   35*
 6   191   191   12        3   263   274    9             8,    1,  L
 7   460   458    7        5   142   130   16        0    87    76   25*
 8   284   299   10             7,    1,  L          1    75   118   31*
      6,    3,  L          0   160   157   12        2   468   483    7
 0   131   108   13        1   395   405    7        3   210   212   12
 1   544   520    6        2   338   353    8             8,    2,  L
 2   222   216    9        3   221   203   10        0   446   440    7
 3   217   219   10        4   108   104   20        1   384   401    8
 4    98    60   19        5     9    69  234*       2   349   349    8
 5   574   590    7        6   245   248   11        3     9    59  243*
 6   395   388    8             7,    2,  L               8,    3,  L
 7     9    91  275*       0   624   643    6        0   252   254   10
 8   285   297   10        1   252   252    9        1     9    39  238*
      6,    4,  L          2   158   168   14        2   468   458    7
 0   858   851    5        3   316   322    8        3   361   363    8
 1   170   162   12        4   580   570    7             8,    4,  L
 2   504   519    6        5     9    18  237*       0   528   527    7
 3   396   405    7        6    24    35   95*       1   349   341    8
 4   205   211   11             7,    3,  L          2   168   146   15
 5     9    49  248*       0   106   157   20             8,    5,  L
 6    74    32   29*       1   632   616    6        0   243   228   11
 7   383   398    9
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OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR $CoMn(CO)_6(MeC_2Ph)_2$

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   COLUMNS ARE 10FO,10FC,  10SIG                                              COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG        L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG
     -17,    0,  L           -8,    0,  L         4  1720  1732     2              4,    0,  L           10,    0,  L         1   267   269     5
  2     7    60  140*     2   760   746     2     6    56    54     7        0   688   686     2     0   985   982     3     2   151   145     7
  4     7    60  138*     4   858   847     2     8   649   655     3        2  1978  1947     2     2    49    17   10*     3   192   193     6
     -16,    0,  L        6   492   466     3    10   905   934     3        4   404   377     2     4   290   296     3     4   169   181     6
  2   218   219     5     8   492   485     3    12   564   561     3        6   506   492     2     6   500   518     3     5    24    77   39*
  4   193   188     6    10    91    99     9    14   412   401     4        8   642   629     3     8   390   395     4     6   268   259     5
  6   145   150     7    12   397   410     4    16   103    94     9       10   701   701     3    10   123   118     8     7   112    82     8
     -15,    0,  L       14   152   143     7          -1,    0,  L         12   524   511     3    12    77    64    12          -15,    1,  L
  2    50   107   18*          -7,    0,  L         2  1125  1100     1       14   315   312     4          11,    0,  L         1   236   224     5
  4     6    29  121*     2   524   516     2     4   986   975     2       16   167   174     7     0   922   908     3     2    78    91    11
  6     6    57  135*     4     4    21   98*     6   714   740     2              5,    0,  L         2   344   366     3     3   235   230     5
  8   155   147     7     6   512   500     3     8   601   602     3        0  1254  1231     2     4   361   358     3     4   102   112     9
     -14,    0,  L        8   363   356     3    10   431   414     3        2   645   612     2     6   213   201     4     5     6    51  132*
  2   132   135     7    10   485   487     3    12   158   171     6        4   709   725     2     8    73    15     9     6    60    79   14*
  4   151   155     6    12    75    78    11    14    81    22     9        6   904   933     2    10    76    31    11     7    85    25    10
  6   245   257     5    14   255   263     5    16     8    29  109*        8   385   389     3    12   205   213     6     8    96   113    10
  8   171   174     6          -6,    0,  L           0,    0,  L         10    52    16   13*          12,    0,  L         9    50   100   20*
 10   100   111    10     2   104   105     4     2  2402  2577     1       12   292   303     4     0   226   218     4          -14,    1,  L
     -13,    0,  L        4   431   416     2     4   427   427     2       14   312   306     4     2   130   126     5     1   142   142     6
  2     6    48  131*     6   244   243     3     6   925   912     2              6,    0,  L         4     6    12  122*     2    43    12   17*
  4   214   235     5     8   250   250     3     8   800   761     3        0  1088  1073     2     6   123   122     7     3   155   154     6
  6   568   556     3    10   441   454     3    10    41    59   15*        2  1561  1508     2     8    85    85    10     4   138   143     6
  8   505   506     4    12   394   388     4    12   140   137     6        4   746   724     2    10   307   304     5     5    57    66   14*
 10   403   392     4    14    78    82    12    14   184   194     5        6   199   215     3          13,    0,  L         6   151   153     6
     -12,    0,  L       16   399   382     5    16     7    49  147*        8   980   982     3     0   389   396     4     7   236   236     5
  2   703   710     3          -5,    0,  L           1,    0,  L         10  1137  1145     3     2     6    46  121*     8   348   355     4
  4   460   450     3     2   693   675     2     2  1165  1147     1       12   537   519     4     4   260   267     4     9   172   171     6
  6   334   336     4     4  1030  1012     2     4   523   534     2       14   232   242     6     6   309   331     4    10   351   353     5
  8    64   108   14*     6   907   899     2     6   540   584     2              7,    0,  L         8    33    71   27*          -13,    1,  L
 10     6    36  134*     8   579   583     3     8   342   328     3        0   815   778     2    10   106   116    10     1   372   385     4
 12   315   320     5    10   589   601     3    10    80    72     8        2  1073  1050     2          14,    0,  L         2   138   149     6
     -11,    0,  L       12    83    87    10    12   112   109     7        4  1019  1058     2     0    41    28   18*     3   224   231     4
  2    74    59     8    14   116   105     8    14   242   247     5        6   856   856     3     2   145   138     6     4    70    63    11
  4   605   624     3    16   378   361     5    16   314   316     5        8   517   529     3     4   295   306     4     5    88    65     9
  6   585   570     3          -4,    0,  L           2,    0,  L         10   199   199     5     6   354   349     4     6    41    78   19*
  8   902   885     3     2  1060  1019     2     0  1615  1547     1       12   265   270     5     8   305   296     5     7   424   412     4
 10   548   512     4     4    57    64     7     2  2110  2062     1       14   173   175     6          15,    0,  L         8   332   325     4
 12     6    33  126*     6   426   414     2     4   928   906     2              8,    0,  L         0   291   300     4     9   324   320     4
     -10,    0,  L        8   245   243     3     6    28    22   13*        0  1368  1358     2     2   378   388     4    10    38    49   22*
  2   890   909     3    10   509   514     3     8     4    55  115*        2   445   426     3     4   198   205     5    11   109    98     9
  4   953   963     3    12   894   899     3    10    34    46   17*        4    17    19   28*     6   268   287     5          -12,    1,  L
  6   665   668     3    14   729   719     4    12   332   327     4        6   513   534     3          16,    0,  L         1   151   168     5
  8    32    41   20*    16   208   203     6    14   232   230     5        8   719   741     3     0   201   202     5     2    44    56   15*
 10   458   448     4          -3,    0,  L      16   120   104     9       10   870   860     3     2    92   146    12     3   355   342     3
 12    93   124    10     2   226   219     2           3,    0,  L         12   209   218     5     4   270   265     5     4   232   236     4
 14    65   123   16*     4   842   819     2     0   433   414     1       14    25    12   36*          17,    0,  L         5   550   555     3
      -9,    0,  L        6   912   949     2     2    48    53     7              9,    0,  L         0   260   262     5     6   268   270     4
  2    84    77     6     8   699   712     3     4   966   957     2        0   411   407     3     2   421   441     5     7   120   105     7
  4    70    79     7    10   758   744     3     6   124   118     4        2   976   994     3          -17,    1,  L         8   277   279     4
  6   449   454     3    12     6    18  145*     8   366   336     3        4  1034  1058     3     1     7    28  133*     9   248   256     5
  8   215   219     4    14   271   273     5    10   385   378     3        6   615   610     3     2    81    88    11    10   385   388     4
 10   364   361     4    16   219   202     6    12    92    91     9        8   247   248     4     3    68    77   14*    11   261   266     5
 12     6    31  130*          -2,    0,  L      14   548   560     4       10   159   166     6     4   100   113    10    12   293   281     5
 14    47     5   19*     2  1622  1613     1    16   571   560     4       12    74    33    10          -16,    1,  L          -11,    1,  L
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     COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG         L   KFO    FC   SIG         L   KFO    FC   SIG
     -11,  1,  L             10    77    91    10         16    64    53    14*
  1   393   393     3        11   158   169     6              -4,  1,  L
  2   418   431     3        12   127   133     7          1    89    95     4
  3   165   156     5        13     6     9   126*         2   755   734     2
  4   524   525     3        14   272   271     5          3   207   199     2
  5   274   270     4        15    73    78    13*         4  1546  1529     2
  6   173   164     5              -7,  1,  L              5   533   541     2
  7   390   381     4         1   378   382     2          6   551   569     2
  8    34    23    21*        2   278   282     3          7   369   371     3
  9   348   342     4         3   506   502     2          8   186   213     4
 10   380   378     4         4   673   655     2          9    51    59    11*
 11     6    40   144*        5   650   649     2         10   447   457     3
 12    75    24    11         6   461   466     3         11   589   584     3
 13    97    96    10         7     5    14   111*       12     6    51   121*
     -10,  1,  L              8   682   700     3         13   378   382     4
  1   748   762     3         9   395   383     3         14    77    46     9
  2    33    63    17*       10   411   412     3         15   146   143     7
  3   524   530     3        11   363   354     4         16     7    90   148*
  4   160   159     4        12   153   163     6              -3,  1,  L
  5   455   458     3        13    70    45    10          1   620   605     2
  6   159   154     5        14     6    51   137*         2   811   790     2
  7    70    48     8        15     7    13   135*         3   449   449     2
  8   200   194     4              -6,  1,  L              4   595   598     2
  9   437   445     4         1    75    80     5          5   351   352     2
 10   237   237     5         2   463   454     2          6   308   303     2
 11   405   417     4         3    85    76     5          7   817   833     2
 12   260   265     5         4   337   338     2          8   242   232     3
 13    88    86    10         5    93   119     5          9   651   665     3
 14   184   182     6         6   329   327     3         10   348   354     3
      -9,  1,  L              7   395   391     3         11     5    12   116*
  1   356   361     3         8   242   253     3         12   410   401     4
  2   868   877     3         9    41    68    14*        13   292   292     4
  3    62    41     8        10    14    78    48*        14   469   463     4
  4   612   607     3        11   187   178     5         15   235   237     5
  5   712   698     3        12    44    88    18*        16   223   231     6
  6   800   801     3        13    67    37    12*             -2,  1,  L
  7   556   548     3        14    95   113     9          1  1272  1260     1
  8   250   257     4        15   280   274     5          2   842   829     1
  9   125   126     6              -5,  1,  L              3   830   808     2
 10   154   147     6         1  1015   995     2          4   867   867     2
 11   348   336     4         2   623   608     2          5   923   922     2
 12    99   114     9         3   864   862     2          6  1328  1338     2
 13   249   235     5         4    17    26    20*         7   437   427     3
 14   131   120     7         5   371   371     2          8   958   969     3
      -8,  1,  L              6   566   563     2          9   650   659     3
  1   761   756     2         7   579   581     3         10   288   296     4
  2   175   153     3         8   292   298     3         11   486   484     3
  3   432   430     3         9   773   768     3         12   169   162     5
  4   164   159     4        10   409   422     3         13   251   242     5
  5    56    43     9        11   339   336     4         14    58    56    15*
  6   143   147     5        12   341   339     4         15    84    75    10
  7   397   411     3        13   224   233     5         16    96    89    10
  8   156   153     5        14   255   262     5              -1,  1,  L
  9   183   189     5        15   212   207     6          1   271   273     1
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     COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG         L   KFO    FC   SIG         L   KFO    FC   SIG
      -1,  1,  L              2   155   155     2          3   213   196     3
  2  1041  1015     1         3   128   131     3          4    25    38    14*
  3   340   330     2         4   771   752     2          5   319   331     3
  4   131   125     3         5   539   551     2          6   841   832     2
  5  1192  1210     2         6   130   110     4          7   759   771     3
  6   121   119     4         7   451   457     3          8   752   756     3
  7   651   653     2         8    67    44     7          9   199   218     4
  8   419   406     3         9   248   251     3         10   567   564     3
  9   113   121     6        10   241   244     4         11    86    92    10
 10    44    64    14*       11   225   221     4         12   358   345     4
 11   355   369     4        12   343   329     4         13   121   118     7
 12   146   146     6        13   154   149     6         14   180   171     6
 13   209   196     5        14   341   346     4         15   204   193     6
 14   224   218     5        15   195   186     6               6,  1,  L
 15   213   198     5        16   239   225     5          0   576   545     2
 16   255   248     5               3,  1,  L              1  1246  1218     2
       0,  1,  L              0   893   864     1          2   419   414     2
  1  1680  1713     1         1   583   559     2          3   821   787     2
  2   583   574     1         2    58    67     6          4   609   619     2
  3   413   401     2         3   414   402     2          5   308   321     3
  4  1118  1094     2         4  1326  1316     2          6   444   464     3
  5   359   349     2         5   116   122     4          7   495   505     3
  6   194   170     3         6   442   459     2          8   112   117     6
  7   813   813     2         7   221   241     3          9   748   752     3
  8   547   546     3         8    13    27    37*        10     5    47   137*
  9   705   703     3         9   255   273     3         11   416   422     4
 10   648   636     3        10   461   451     3         12    82    44    10
 11   118   122     6        11   266   263     4         13     6    19   137*
 12   262   261     4        12   154   152     6         14   125   137     8
 13   130   108     6        13   112   100     8         15     7    45   147*
 14   261   248     5        14   143   154     7               7,  1,  L
 15    87    62    10        15   156   148     7          0   281   299     3
 16   284   288     5        16    32    64    33*         1   472   456     2
       1,  1,  L                     4,  1,  L              2   226   217     3
  0  1764  1748     1         0  1642  1593     2          3   760   764     2
  1   795   768     1         1   411   408     2          4   389   367     3
  2  1222  1173     1         2   144   146     3          5   562   586     3
  3   613   585     2         3   764   741     2          6   235   237     3
  4   416   439     2         4   164   159     3          7    87    99     7
  5  1034  1047     2         5   600   592     2          8   651   663     3
  6   185   171     3         6   230   242     3          9   189   197     5
  7   190   176     3         7   484   479     3         10   553   555     4
  8   213   231     3         8    51    88    11*        11   201   198     5
  9   253   264     3         9   146   152     5         12   344   342     4
 10   142   153     5        10   105    94     7         13   117   111     8
 11   433   433     3        11   504   496     3         14   163   154     7
 12     6     5   129*       12     6    53   144*              8,  1,  L
 13    32    28    23*       13   226   225     5          0   263   272     3
 14     6    28   134*       14   361   346     4          1   833   809     2
 15     6    28   139*       15   186   186     6          2   500   506     3
 16    52    74    19*              5,  1,  L              3   514   507     3
       2,  1,  L              0   743   714     2          4   321   335     3
  0   295   289     2         1   365   353     2          5   184   170     4
  1   823   786     1         2   296   293     2          6   303   305     3
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG        L   KFO    FC  SIG        L   KFO    FC  SIG
       8,  1, L             0   228   222    4        2     7    18  140*
  7   654   674    3        1   307   317    4            -17,  2, L
  8   327   342    4        2   192   200    5        1    93    99   12
  9   720   725    3        3   351   367    4        2   211   207    6
 10   234   248    5        4     6    41  127*       3   133   109    8
 11   200   212    5        5   234   236    4            -16,  2, L
 12    53    28   16*       6   105   107    8        1    97   110   10
 13   202   198    6        7    70    61   11        2   161   159    7
 14    60    75   15*       8   191   192    5        3    92    63   10
       9,  1, L             9   150   146    7        4   116   118    9
  0   529   556    3       10   236   242    5        5     7    34  146*
  1   502   516    3       11   274   279    5        6    77    49   12
  2   413   402    3            13,  1, L                 -15,  2, L
  3   154   169    4        0   206   203    5        1     6    11  143*
  4   103   118    6        1   189   186    5        2   491   480    4
  5   648   627    3        2   307   294    4        3   237   226    5
  6    81    79    8        3    60    96   14*       4   399   400    4
  7   114   103    6        4   407   417    4        5     6    34  138*
  8   278   287    4        5   341   346    4        6   293   290    5
  9   160   167    6        6   251   239    5        7   181   195    6
 10   303   293    4        7   311   305    4        8   138   149    8
 11   218   223    5        8    97    90    9            -14,  2, L
 12   159   170    7        9   119   117    8        1   139   140    6
 13   115   122    9       10   119   125    9        2    72   105   12*
      10,  1, L                 14,  1, L             3   292   303    4
  0    57    71   10*       0   185   176    5        4    32    58   27*
  1   131   128    5        1   229   237    5        5    39    41   20*
  2   174   176    4        2    58    83   14*       6    67    52   13*
  3   256   256    3        3     9    33   79*       7   162   151    6
  4   447   457    3        4     6    55  145*       8   158   170    7
  5   425   420    3        5    56    10   14*       9   160   159    7
  6   260   262    4        6   126   109    7       10   186   172    6
  7   461   478    3        7   127   120    7            -13,  2, L
  8   147   150    6        8    83    86   11        1    37    31   22*
  9    81    74    8        9   250   246    5        2   517   513    4
 10   198   184    5            15,  1, L             3   199   195    5
 11   190   196    6        0   122   110    7        4   320   319    4
 12   203   205    6        1   121   126    7        5   150   142    6
 13    91    77   11        2   179   175    5        6   131   138    7
      11,  1, L             3   174   175    6        7    45    66   19*
  0   750   751    3        4   272   270    5        8    63     1   12*
  1   557   558    3        5   311   315    5        9     6    67  146*
  2   521   505    3        6   206   211    6       10    71    32   12*
  3   245   258    4        7   236   249    6       11    79    76   11
  4    48   118   16*           16,  1, L                 -12,  2, L
  5     6    18  126*       0    96   114   10        1   263   258    4
  6    85    77    9        1   117   119    8        2   270   278    4
  7   251   243    4        2   171   162    6        3   154   153    5
  8   119   115    7        3     7    16  135*       4    84    71    9
  9   266   268    5        4   157   147    6        5    63    29   11*
 10   126   107    7        5   128   128    8        6   335   335    4
 11    17    55   48*           17,  1, L             7   131   135    7
 12    59    26   15*       0     7    31  133*       8     6    44  130*
      12,  1, L             1   248   259    5        9   317   319    4
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG        L   KFO    FC  SIG        L   KFO    FC  SIG
     -12,  2, L             7   278   289    4       14    42    41   18*
 10   228   240    5        8   675   696    3       15   167   176    6
 11    57    52   15*       9   209   209    5             -4,  2, L
 12   193   184    6       10   627   624    3        1   195   186    2
     -11,  2, L            11   241   250    5        2   743   724    2
  1   218   219    4       12   161   155    6        3   585   566    2
  2    59    16    9       13   113   120    8        4  1091  1088    2
  3   278   274    4       14   122   127    8        5    78    81    6
  4   143   147    5             -7,  2, L             6   466   512    3
  5   398   411    3        1   322   312    3        7   455   453    3
  6    71   114   11        2   861   904    2        8   601   598    3
  7    30    39   25*       3   332   321    3        9   542   556    3
  8    55    16   13*       4   715   737    2       10    74    68    9
  9   174   175    5        5    36    70   15*      11   128   134    6
 10    85   118   10        6   738   745    3       12    70    75   11
 11    41   112   24*       7   293   292    3       13   126   145    7
 12   296   301    5        8     5     5  119*      14   131   122    7
 13   141   127    7        9   129   127    6       15    77    76   12
     -10,  2, L            10   311   311    4       16    58    63   16*
  1   207   208    4       11    31    19   23*            -3,  2, L
  2   289   292    3       12   423   421    4        1    84    86    4
  3   176   174    4       13    71   134   14*       2   580   561    2
  4   157   157    5       14   320   316    5        3   219   213    2
  5   325   322    3       15    99   113   10        4   214   209    3
  6   271   269    4             -6,  2, L             5   264   277    3
  7   134   128    6        1   190   185    3        6   242   241    3
  8   252   249    4        2   175   180    3        7     4     3  104*
  9    72    58   11        3   496   476    2        8    98   105    6
 10   399   388    4        4   192   212    3        9   508   524    3
 11    83    57   10        5     4    20  110*      10    52    17   11*
 12   305   308    4        6   989  1009    3       11   303   311    4
 13    61    39   15*       7    43    57   13*      12   347   349    4
      -9,  2, L             8   912   906    3       13   199   193    5
  1   307   312    3        9   217   218    4       14   330   329    5
  2   676   686    3       10   589   610    3       15    90    44    9
  3   344   332    3       11   296   303    4       16   311   304    5
  4   772   758    3       12   185   203    5             -2,  2, L
  5    65    33    8       13     6    71  142*       1  1077  1061    2
  6   255   253    4       14    99    88    9        2  1131  1115    2
  7    99   118    7       15   186   188    6        3   404   393    2
  8    52    43   12*            -5,  2, L             4   730   705    2
  9   136   140    6        1   422   411    2        5   465   476    2
 10   345   349    4        2  1500  1508    2        6    60    55    7
 11     6    27  126*       3     4     8   94*       7   730   724    2
 12   501   497    4        4   555   567    2        8   466   470    3
 13   102   104    9        5   230   208    3        9   541   535    3
 14   433   419    4        6     4    46  106*      10   350   349    4
      -8,  2, L             7    57    33    8       11    59    25   10*
  1   301   291    3        8    53    16    9*      12   117   114    7
  2   112   103    5        9   179   180    4       13   269   279    5
  3   327   330    3       10    68    78    9       14   169   158    6
  4   305   293    3       11    82    69    9       15    75    60   13*
  5   386   378    3       12   113   122    7       16   381   369    5
  6   675   677    3       13    16   101   52*            -1,  2, L
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   COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
      -1,  2, L            0   848   831     2      2    42    95    8*
  1   467   455     2      1   433   424     2      3   484   470     2
  2   246   256     2      2  2179  2147     2      4   180   185     3
  3   567   556     2      3   658   642     2      5   415   402     3
  4   211   204     2      4  2053  2042     2      6   157   171     4
  5   179   175     3      5   120   108     4      7   158   146     4
  6   117   116     4      6  1203  1211     2      8   227   235     4
  7   413   414     3      7    80    67     6      9   322   334     3
  8   217   211     4      8   558   564     3     10   182   188     5
  9   495   505     3      9    23    15   24*     11   289   286     4
 10   374   387     3     10   280   294     4     12    44    43   18*
 11   126   106     6     11    52    83   13*     13    38    29   19*
 12   410   411     4     12   101   104     8     14    43     9   20*
 13   121   128     7     13   101   112     9     15     7    28  144*
 14   522   515     4     14   123   116     8            6,  2, L
 15    56    58   17*     15     7    30  135*      0   875   865     2
 16   312   298     5     16   191   191     7      1   278   272     3
       0,  2, L                  3,  2, L          2   489   481     2
  0   275   282     2      0  1129  1123     2      3   232   225     3
  1   597   578     2      1   346   326     2      4    34    45   13*
  2   230   220     2      2  1187  1179     2      5    36    35   13*
  3   793   780     2      3   536   507     2      6   163   155     4
  4   606   592     2      4   379   374     2      7   189   190     4
  5   416   429     2      5   101   112     5      8   232   235     4
  6  1044  1073     2      6   287   276     3      9   232   225     4
  7   122   127     4      7   304   301     3     10   119   116     7
  8   682   697     3      8   640   675     3     11   124   132     7
  9    55    83   11*      9   360   358     3     12    73    46    11
 10   591   573     3     10   578   600     3     13   129   132     7
 11    57    23   11*     11   128   122     6     14    67    94   14*
 12    88    76     9     12   314   311     4            7,  2, L
 13    89    87     9     13   102    95     9      0  1326  1323     2
 14   209   200     5     14   187   180     6      1   145   144     4
 15    10    20   89*     15    36    54   26*      2     4    30  100*
 16   341   327     5            4,  2, L          3    50    74   10*
       1,  2, L            0   346   352     2      4   166   161     4
  0   365   348     2      1   119   116     4      5   172   172     4
  1    35    46   10*      2   588   576     2      6    26    16   21*
  2   220   218     2      3   284   283     2      7   428   441     3
  3   256   247     2      4  1218  1208     2      8   114   109     6
  4     4    22  104*      5   586   582     2      9   399   399     4
  5    90    89     5      6   990  1003     2     10   338   335     4
  6   142   144     4      7   295   314     3     11     6    24  142*
  7    56    29     8      8   402   395     3     12   347   316     4
  8    91   111     6      9   149   148     5     13   101    93     9
  9    67    21     8     10   110   122     7     14   120    98     8
 10   815   823     3     11     6    49  133*            8,  2, L
 11    98    92     8     12     6    55  133*      0   683   683     3
 12   696   691     4     13    33    16   23*      1   266   258     3
 13    86    80     9     14    92   112    10      2   524   517     3
 14   359   348     4     15   138   132     7      3   309   326     3
 15    64    52   13*            5,  2, L          4    35     5   13*
 16    97    79    10      0  1125  1140     2      5    62    71     9
       2,  2, L            1   548   535     2      6   576   581     3
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   COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
       8,  2, L            2   678   688     3      4   362   350     5
  7    70    68     9      3    31    44   24*      5    11    74   96*
  8   378   390     4      4   477   469     4          -15,  3, L
  9   215   216     5      5    55    42   14*      1   192   175     6
 10    44    60   18*      6   248   236     5      2    85    89    11
 11   121   132     8      7   164   166     6      3   313   304     5
 12   257   256     5      8    40    16   20*      4    65    52   14*
 13    64    46   15*      9    24    66   39*      5   449   435     4
 14   311   325     5     10   152   145     7      6     7    41  143*
       9,  2, L           11     7   104  165*      7    86   108    12
  0     5    28  107*           13,  2, L              -14,  3, L
  1   536   540     3      0   542   539     3      1   315   314     4
  2    67    61     8      1    69    51    11      2   396   405     4
  3    66    68     9      2   141   156     6      3     6    13  140*
  4     5    22  122*      3     6    14  124*      4   368   354     4
  5   207   208     4      4   178   177     6      5   176   182     6
  6   203   199     4      5    85    99    10      6   269   260     5
  7    90    77     8      6   373   384     4      7   177   172     6
  8   368   360     4      7    55     5   14*      8   176   167     6
  9   192   194     5      8   368   373     5      9   175   172     7
 10   452   446     4      9     7   102  170*          -13,  3, L
 11    41    57   20*           14,  2, L          1   172   175     6
 12   286   285     5      0   577   574     4      2   328   323     4
 13    81    52    12      1    87     4     9      3   104   105     8
      10,  2, L            2   567   553     4      4    87    51     9
  0    94    85     7      3     6    36  133*      5     6    22  129*
  1   130   129     5      4   415   403     4      6   119   123     7
  2   524   540     3      5   162   165     6      7    33    70   24*
  3   458   469     3      6   129   130     8      8   150   149     6
  4   387   399     3      7    90    34    10      9    67    65   13*
  5    68    70    10      8     7    57  137*     10    50    85   17*
  6   364   361     4           15,  2, L              -12,  3, L
  7   164   157     5      0   300   315     5      1    72    83    11
  8   162   155     6      1     6    22  136*      2   363   353     4
  9   153   146     6      2    16    62   51*      3    58    51   13*
 10    76    66    10      3     6    10  136*      4   117   116     7
 11   101   102     9      4   183   189     6      5   223   216     5
 12   246   255     5      5   109   128     9      6    62    59   13*
      11,  2, L            6   169   160     6      7   309   312     4
  0   417   429     3      7    52    52   18*      8    63    16   12*
  1   309   294     3           16,  2, L          9     6    51  125*
  2   210   207     4      0   297   292     5     10   107   104     9
  3    31     9   19*      1    68    55   13*     11   151   168     7
  4   221   217     4      2   193   181     6          -11,  3, L
  5   142   135     6      3   105   101     9      1    66    59    10
  6   396   380     4      4   152   151     7      2   235   237     4
  7   111    93     7      5    44   114   25*      3    43    30   15*
  8   399   401     4           17,  2, L          4   149   150     6
  9    87   121    11      0    86    42    10      5    75    94    10
 10   404   397     4      1   147   143     8      6    82    78     9
 11    84   107    12          -16,  3, L          7   406   408     4
      12,  2, L            1   228   242     6      8    16    17   49*
  0   503   500     3      2   331   327     5      9   149   141     6
  1   168   169     5      3     7    58  155*     10   102   106     9
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   COLUMNS ARE 10FO,10FC,  10SIG                                              COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG
     -11,  3,  L          8   644   656     3    15   180   178     6          0,  3,  L          7   580   572     3    11   451   468     4
 11    48    28   17*     9   213   206     5         -3,  3,  L          5   223   225     3     8   182   195     4    12     6    14  136*
 12   194   179     6    10   566   554     4     1   413   423     2     6   174   184     4     9   459   475     3    13     6    60  144*
     -10,  3,  L         11   301   300     4     2   591   568     2     7   435   436     3    10     5    10  134*    14   129   131     8
  1   299   300     3    12   265   273     5     3   480   449     2     8   482   481     3    11    71    79    12          7,  3,  L
  2    94    99     7    13   222   228     5     4   116   121     4     9   717   741     3    12    59    70   14*     0   159   162     4
  3   294   288     3    14    47    64   19*     5   849   835     2    10   633   653     3    13    59    21   13*     1   159   152     4
  4   383   387     3         -6,  3,  L          6   207   177     3    11   216   222     5    14    18     9   47*     2   312   320     3
  5    40    65   18*     1    65    48     7     7   427   436     3    12   581   587     4    15   106   122    10     3   101    91     6
  6    52    39   14*     2  1038  1054     2     8    76    72     7    13     6    43  141*          4,  3,  L          4   170   185     4
  7   134   136     6     3   103   109     5     9    98   102     7    14   429   425     4     0   566   569     2     5   141   127     5
  8   135   126     6     4   243   255     3    10    75    79     9    15   186   182     6     1   457   471     2     6   103    94     6
  9   233   233     5     5   306   316     3    11   370   372     4          1,  3,  L          2   950   950     2     7   379   387     4
 10   151   154     6     6   127   118     5    12   222   209     5     0   131   124     3     3   989   965     2     8    73    60    10
 11   195   184     5     7   543   553     3    13   198   197     5     1   360   367     2     4   231   235     3     9   172   173     5
 12   219   207     5     8    90    92     8    14   165   151     6     2   276   270     2     5   753   769     3    10   232   230     5
 13    54    79   18*     9   461   467     3    15   201   187     6     3   760   749     2     6   126   121     5    11   305   308     5
      -9,  3,  L         10   282   281     4         -2,  3,  L          4   882   879     2     7   258   250     3    12    69    68   12*
  1   232   218     4    11   132   133     7     1   340   353     2     5   411   398     3     8   244   247     4    13   180   190     6
  2   166   162     4    12   385   390     4     2   494   505     2     6   586   592     3     9     5    48  138*    14   177   164     7
  3   319   307     3    13    96    95    10     3   392   386     2     7     5    39  121*    10   249   254     4          8,  3,  L
  4   334   331     3    14    53    95   17*     4   106   103     4     8   494   483     3    11   300   297     4     0   419   434     3
  5   369   372     3    15     7    17  147*     5   382   358     2     9   614   623     3    12   194   188     6     1   512   514     3
  6   439   472     3         -5,  3,  L          6   771   797     2    10   198   190     5    13   282   280     5     2   192   192     4
  7   333   342     4     1   597   616     2     7   109   116     5    11   495   501     4    14     7     6  126*     3    70    73     9
  8   427   428     4     2   107   100     5     8   378   394     3    12   165   175     6    15   105   101     9     4   198   195     4
  9   144   152     6     3   123   149     4     9   180   191     4    13    73    50    10          5,  3,  L          5   244   245     4
 10   230   230     5     4   292   305     3    10   273   276     4    14   169   165     6     0   253   251     3     6   289   296     4
 11   160   151     6     5   171   182     4    11   418   403     4    15   120   129     9     1   576   587     2     7   485   487     3
 12   286   292     5     6   494   515     3    12   454   457     4          2,  3,  L          2   849   847     2     8   319   309     4
 13   283   289     5     7   205   220     4    13    65    91   12*     0  1119  1122     2     3   326   304     3     9   185   173     5
      -8,  3,  L          8   542   552     3    14   347   346     4     1    26    25   14*     4   876   871     3    10   266   262     5
  1   543   543     3     9   346   352     4    15    57    85   16*     2   793   787     2     5   421   412     3    11     6    48  133*
  2   470   493     3    10   530   535     3         -1,  3,  L          3    58    58     7     6   713   723     3    12    91   132    12
  3   126   112     5    11    90    40     9     1   943   931     2     4   337   322     3     7    40    45   14*    13    99   103    10
  4   381   381     3    12   287   305     5     2   714   709     2     5   900   911     2     8   425   432     3          9,  3,  L
  5   346   328     3    13    53    31   15*     3   288   287     2     6   471   464     3     9   202   199     5     0    26    16   20*
  6   161   157     5    14     6    47  127*     4   169   172     3     7   779   782     3    10    53    43   14*     1    69    13     8
  7   352   344     3    15    91    76    10     5    83    85     5     8   369   386     3    11   315   301     4     2   112   120     6
  8   188   189     5         -4,  3,  L          6   558   586     3     9   380   385     3    12   186   188     6     3   297   303     3
  9   442   458     4     1   175   176     3     7   294   306     3    10   352   360     4    13   148   134     7     4   452   465     3
 10   421   432     4     2   742   752     2     8   573   593     3    11    83    77    10    14    79    72    12     5     5     8  131*
 11   180   179     6     3   857   850     2     9   164   166     5    12   391   398     4          6,  3,  L          6   222   229     4
 12   226   228     5     4   719   710     2    10   370   364     3    13   261   266     5     0   690   691     2     7     6    17  133*
 13    99   100     9     5   640   635     2    11   301   296     4    14   239   242     5     1   230   238     3     8   280   278     4
 14   156   156     7     6   294   290     3    12    64    55   13*    15   161   152     7     2   699   688     2     9    95    97     8
      -7,  3,  L          7   304   323     3    13   299   308     5          3,  3,  L          3   385   405     3    10    87    81     9
  1   487   492     3     8   161   147     4    14   320   321     5     0   127   125     3     4   297   279     3    11   259   261     5
  2    43    48   12*     9     5    50  124*    15   303   280     5     1   798   819     2     5   675   665     3    12   356   356     5
  3   444   443     3    10   188   184     5          0,  3,  L          2  1244  1229     2     6   251   246     3         10,  3,  L
  4   678   691     3    11   227   220     5     1   366   357     2     3   175   184     3     7   139   137     5     0    84    86     7
  5   681   687     3    12    36    40   22*     2   691   650     2     4  1457  1438     2     8    57    60   12*     1     5    46  118*
  6   478   478     3    13     6    11  136*     3   132   122     4     5   264   250     3     9   372   366     4     2   209   208     4
  7   164   156     5    14     6    45  128*     4   256   247     3     6   606   605     3    10    35    18   23*     3   173   157     5
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     COLUMNS ARE 10FO,10FC,  10SIG

   L   KFO    FC    SIG        L   KFO    FC    SIG        L   KFO    FC    SIG
       10,    3,  L            7   159   162      7        3   212   211      5
   4   193   181      5           15,    3,  L             4   317   311      4
   5   489   482      3        0   217   206      5        5   117   125      7
   6   436   443      4        1    87    79     10        6   475   482      4
   7   238   243      5        2   377   374      4        7    35     9    22*
   8   382   391      4        3   213   211      5        8   365   356      4
   9    85    75     10        4   178   165      6        9    81    73     10
  10   293   295      5        5   177   179      6       10   252   253      5
  11   216   223      6        6     7    33   149*       11   132   128      8
  12   186   182      7           16,    3,  L               -10,    4,  L
       11,    3,  L            0   268   260      5        1   166   177      5
   0   270   264      4        1   256   264      5        2   793   800      3
   1   213   220      4        2     7    28   136*        3    59     4    12*
   2   287   299      4        3    84    53     11        4   681   693      3
   3    74    16      9          -15,    4,  L             5   336   332      4
   4   492   515      4        1   188   187      6        6   308   319      4
   5   173   187      6        2     7    39   133*        7   268   273      4
   6   348   337      4        3    30    50    32*        8     6    29   134*
   7    85    51      8        4     7    78   141*        9   124   125      7
   8   122   122      8        5   111   134      9       10   136   140      7
   9   314   319      4        6     7    34   156*       11   224   234      5
  10     7    14   140*          -14,    4,  L            12    12    40    73*
  11   197   195      6        1   201   195      5           -9,    4,  L
       12,    3,  L            2    47    28    19*        1   281   273      4
   0   218   214      4        3   191   182      6        2    43    33    15*
   1   486   494      3        4   148   142      7        3    28    16    22*
   2    82    67     10        5   305   300      5        4   225   225      4
   3   224   230      5        6   181   179      6        5   142   141      6
   4   191   174      5        7    77    59     12        6   548   551      3
   5   117   117      7        8   150   152      7        7   279   278      4
   6   272   268      5          -13,    4,  L             8   559   584      4
   7   162   152      6        1   190   192      5        9   308   301      4
   8   257   250      5        2    87    94     10       10   203   204      5
   9   127   125      8        3    78    54     11       11   133   123      7
  10   167   161      7        4   176   179      6       12    35     2    24*
       13,    3,  L            5    91    83     10       13     7    27   150*
   0   495   478      4        6   182   179      6           -8,    4,  L
   1   237   246      5        7   242   255      5        1   282   278      3
   2   382   373      4        8   271   266      5        2   543   558      3
   3     6   113   148*        9    86    95     12        3   241   250      4
   4   171   162      6          -12,    4,  L             4   352   348      3
   5     6    72   147*        1   119   123      7        5   195   202      4
   6   195   193      6        2   539   550      4        6   170   171      5
   7   224   215      5        3   216   220      5        7   175   186      5
   8    97    98     10        4   372   364      4        8    76    77     10
   9   253   251      6        5    63    30    11*        9   127   130      7
       14,    3,  L            6   231   228      5       10    40    40    18*
   0   227   223      5        7    80    22     10       11   200   194      5
   1   256   263      5        8   133   132      7       12   202   202      5
   2     6    35   135*        9     7    10   131*       13   312   314      5
   3   344   356      4       10     7    73   142*           -7,    4,  L
   4    78    85     12          -11,    4,  L             1   122   121      5
   5   177   171      6        1   365   386      4        2   176   172      4
   6    49    96    22*        2   207   205      5        3    31    35    17*
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     COLUMNS ARE 10FO,10FC,  10SIG

   L   KFO    FC    SIG        L   KFO    FC    SIG        L   KFO    FC    SIG
       -7,    4,  L           12   533   531      4        2  1060  1050      2
   4    78    83      8       13    90    98     10        3   279   284      3
   5   380   398      3       14   402   379      4        4   572   568      3
   6    76    55      8           -3,    4,  L             5   385   371      3
   7   400   401      3        1   171   173      3        6   102   108      6
   8     6    47   131*        2   585   574      2        7    68    71      8
   9   348   353      4        3   497   502      3        8   195   190      4
  10    64    16    11*        4   897   905      3        9   396   388      3
  11   140   129      6        5   227   243      3       10   252   250      4
  12    34    47    23*        6   752   793      3       11   273   263      4
  13    35    22    26*        7    77    89      8       12   156   168      6
  14    44    17    20*        8   450   468      3       13   369   367      4
       -6,    4,  L            9    94    86      7       14    76    37     11
   1   123   124      4       10   251   250      4       15    67    48    15*
   2   305   307      3       11    59    77    14*            1,    4,  L
   3   734   739      3       12   208   215      5        0   589   555      2
   4    92    76      6       13    21    27    38*        1   107    96      5
   5   418   427      3       14    91    70     10        2   328   330      3
   6   165   164      4       15    72    61    13*        3   108   113      5
   7    27    16    22*           -2,    4,  L             4   753   774      3
   8     5    68   129*        1   389   404      3        5   472   489      3
   9   341   337      4        2   744   746      2        6   679   674      3
  10   223   224      5        3    46    15     9*        7   680   698      3
  11   140   135      6        4   489   486      3        8   492   487      3
  12   422   415      4        5     5    40   103*        9   172   184      5
  13    73    95    13*        6     5     6    99*       10   114   114      7
  14   348   358      5        7   256   254      3       11   188   181      5
       -5,    4,  L            8   336   346      4       12     7     9   100*
   1   198   199      3        9   102    99      7       13   179   163      6
   2   358   349      3       10   603   614      3       14   111   115      9
   3   337   340      3       11   410   413      4       15   273   272      5
   4   481   471      3       12   425   429      4            2,    4,  L
   5   571   580      3       13   180   180      6        0   849   847      2
   6   384   395      3       14   214   219      6        1   427   425      2
   7   397   405      3       15   193   187      6        2   436   445      3
   8   562   589      3           -1,    4,  L             3   556   536      3
   9   193   197      5        1   140   143      4        4   390   384      3
  10   355   361      4        2   791   779      2        5   148   144      4
  11     6    40   140*        3   431   430      3        6   254   260      4
  12     6    51   130*        4   409   432      3        7   153   163      5
  13     6    73   148*        5   124   128      4        8   197   191      4
  14   101   125     10        6   729   751      3        9   612   634      3
       -4,    4,  L            7   202   204      4       10   230   223      5
   1   652   639      2        8   692   706      3       11   144   156      7
   2   119   130      4        9   369   387      4       12   252   249      5
   3   494   527      3       10   239   236      4       13   131   119      8
   4   393   383      3       11   154   151      6       14   141   144      7
   5   159   145      4       12    72    75     11            3,    4,  L
   6   217   223      4       13   188   189      6        0    66    60      7
   7   288   299      3       14    67    96    15*        1    55    64      8
   8   142   134      5       15   177   168      7        2     4    19   102*
   9     5    35   123*            0,    4,  L             3   917   881      3
  10   607   601      3        0   903   923      2        4    56    68    10*
  11    38    25    20*        1     4    56   115*        5   884   884      3
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    COLUMNS ARE 10FO,10FC,  10SIG                                                       COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO     FC    SIG      L    KFO     FC    SIG      L    KFO     FC    SIG      L    KFO     FC    SIG      L    KFO     FC    SIG      L    KFO     FC    SIG
       -1,     5,  L         11     41     57    18*      4    654    657      3            10,     5,  L          1    260    259      5      1      5     53   136*
  5    637    652      3     12     84     47     10      5      5     73   145*      5     60     62    13*      2     93     78      9      2     29     67    25*
  6    171    171      4     13     86    101     12      6    611    608      3      6    146    138      7      3    331    329      5      3    124    125      6
  7    373    398      3     14    108    102     10      7    151    152      6      7    147    157      7      4     50     53    17*      4     71     81     10
  8    183    196      5            3,     5,  L          8    305    291      4      8     73     52     12      5    241    242      5      5      6     27   120*
  9    189    192      5      0    244    251      3      9    395    410      4      9     59     46    14*      6    239    243      5      6     66     58    11*
 10    255    252      4      1    318    293      3     10     61     65    15*     10     45     25    21*      7    176    177      6      7    219    219      5
 11     46     79    18*      2     94     93      6     11    365    353      5           11,     5,  L          8     73     32    13*      8    287    286      4
 12    298    296      5      3    271    271      3     12     83    104     12      0     88     95      9          -10,     6,  L          9    220    220      5
 13      7     17   142*      4    225    208      4            7,     5,  L          1     44     17    17*      1     76     65     10     10    274    282      5
 14     97     54     10      5    152    146      5      0    528    540      3      2     50     19    16*      2    209    218      5     11    162    161      7
        0,     5,  L          6     72     91     10      1    337    355      4      3    332    331      4      3     45     69    19*     12    158    144      7
  1    644    649      3      7    165    167      5      2    237    234      4      4    167    165      6      4    171    165      6           -5,     6,  L
  2    728    732      3      8      6     13   142*      3    485    477      3      5    284    282      5      5    180    173      6      1    233    239      4
  3    573    554      3      9    233    239      5      4     20      2    33*      6     45     67    21*      6    143    144      7      2    149    160      5
  4    547    570      3     10     88     81     10      5    273    276      4      7    154    155      7      7    269    264      5      3    204    200      4
  5    199    188      4     11    186    183      6      6    296    299      4      8     61     58    15*      8    168    173      6      4    262    257      4
  6    549    562      3     12    356    352      5      7     22    114    40*      9     40     11    21*      9    249    256      5      5    489    520      3
  7    178    181      5     13    333    351      5      8    405    400      4           12,     5,  L               -9,     6,  L          6    237    230      4
  8    420    420      4            4,     5,  L          9     83     90     11      0    183    183      6      1    289    276      4      7     78     84     10
  9    195    195      5      0    319    300      3     10    335    350      5      1    165    173      6      2    145    151      6      8    194    197      5
 10    236    244      5      1    144    136      4     11     64     15    15*      2    120    133      7      3    646    653      4      9    262    252      4
 11    250    238      5      2     73     78      8     12    285    291      6      3     44     43    19*      4     61     22    12*     10    120    118      7
 12      6      6   134*      3     70     68      8            8,     5,  L          4     60     41    14*      5    343    350      4     11    287    280      5
 13    163    154      6      4    577    567      3      0    404    404      3      5     74     68    13*      6     51     70    18*     12     62     25    14*
 14      7      1   147*      5      5     60   140*      1    326    328      4      6      7     22   151*      7     57     32    14*           -4,     6,  L
        1,     5,  L          6    227    215      4      2    782    774      3      7     77     82     13      8     99    101     10      1     99     96      6
  0    644    685      3      7     15     64    52*      3     46     42    17*           13,     5,  L          9     88     78     10      2     92     78      7
  1    293    294      3      8     89     89      9      4    614    618      3      0     79     85     12     10     59    117    17*      3    245    238      4
  2    598    612      3      9    262    263      5      5    166    162      5      1      6     19   129*           -8,     6,  L          4      5     47   129*
  3      5     69   116*     10     23     39    35*      6    365    353      4      2     91     86     10      1      6     66   144*      5    360    375      4
  4     66     90      9     11    344    338      4      7    196    193      5      3     61     67    14*      2    172    170      5      6    385    389      4
  5    116    125      6     12    120    130      9      8    167    195      6      4    151    153      7      3    144    144      6      7    423    438      4
  6    302    304      4     13    227    238      6      9    291    292      5      5      7     39   143*      4     34     32    22*      8      6     18   134*
  7    201    212      5            5,     5,  L         10     43     25    18*      6      7     17   133*      5    177    173      5      9    213    215      5
  8    113    114      7      0    119    120      5     11    196    172      6           14,     5,  L          6    295    293      4     10      6     12   133*
  9    223    224      5      1     97    108      7            9,     5,  L          0     94    106     10      7     36     67    25*     11     66     12    12*
 10    147    154      6      2    351    360      3      0    500    492      3      1    197    200      6      8    406    428      4     12    187    184      6
 11    141    141      7      3    228    228      4      1    382    388      4      2      7     59   148*      9     63     86    14*           -3,     6,  L
 12     75    121    13*      4     38     45    17*      2    215    211      5      3    101    140     10     10    136    140      7      1    353    378      3
 13     82     81     11      5    294    295      4      3    215    209      5      4    247    236      5     11     57     63    17*      2    251    265      4
 14    262    256      6      6    353    361      4      4    147    152      6          -13,     6,  L               -7,     6,  L          3    387    386      3
        2,     5,  L          7    332    333      4      5    280    266      4      1      7     14   129*      1    474    477      4      4      5     29   115*
  0    333    322      3      8    443    435      4      6    215    218      5      2    239    229      5      2    517    513      3      5    360    358      3
  1    273    277      3      9     67     53    13*      7    305    317      5      3    186    175      6      3     80     96     10      6     62     50    11*
  2    208    216      4     10    206    200      5      8    113    106      8      4    176    173      7      4    424    424      4      7    273    274      4
  3    132    127      5     11     94     88     10      9     83     63     10          -12,     6,  L          5     84     79     10      8     94    107      9
  4    548    539      3     12    317    289      5     10    139    142      8      1    126    125      8      6    155    159      6      9    200    192      5
  5    400    394      3     13    242    241      6           10,     5,  L          2    157    157      7      7     50     17    14*     10    262    274      5
  6    399    381      3            6,     5,  L          0    211    202      5      3      7      7   133*      8     87    118     10     11    397    380      4
  7     76     33      9      0    172    160      4      1    171    160      5      4     93     32      9      9      6     35   146*     12    160    171      7
  8     15     44    49*      1    397    400      3      2    649    649      3      5    190    187      6     10    226    234      5           -2,     6,  L
  9    216    214      5      2    242    249      4      3    127    131      7      6     43     15    20*     11     98    117     11      1     61     30      9
 10     56      7    13*      3     85     99      8      4    348    358      4          -11,     6,  L               -6,     6,  L          2    267    258      4
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      COLUMNS ARE 10FO,10FC,  10SIG                                                        COLUMNS ARE 10FO,10FC,  10SIG

   L   KFO    FC  SIG         L   KFO    FC  SIG         L   KFO    FC  SIG              L   KFO    FC  SIG         L   KFO    FC  SIG         L   KFO    FC  SIG 
       -2,    6,  L                  2,    6,  L        11   407   400    5                   10,    6,  L           6    41    63   20*         8     6    39  124*
   3   508   511    3          0   239   232    4              6,    6,  L               4   160   154    6          7   144   151    8          9    29    18   27*
   4   182   183    4          1   321   328    3          0    68    46   11               5   303   289    5          8    72    34   12         10   127   110    8 
   5   692   722    3          2   401   397    3          1   150   151    6               6   152   143    7             -7,    7,  L          11    78    37   12 
   6    54     7   12*         3   459   450    3          2   112   125    8               7   193   199    6          1   193   197    5             -2,    7,  L   
   7   452   446    4          4   477   480    3          3   400   387    4               8   151   156    8          2     6    41  134*         1    85    66    9 
   8    66    62   11          5   144   138    6          4   112   107    8                   11,    6,  L           3   199   215    5          2    76    97   10 
   9    65    63   12*         6   361   351    4          5   457   465    4               0    37    42   23*         4   132   128    7          3    74    91   10 
  10   168   174    6          7   146   136    6          6   152   149    6               1    47    10   17*         5    61    16   13*         4    50    61   16*
  11    34    41   25*         8   276   274    4          7   233   229    5               2     6     5  142*         6   501   500    4          5    61    52   12*
  12     7    53  141*         9    95    85    9          8     8    16  108*              3   181   174    6          7   143   143    7          6     6    65  129*
       -1,    6,  L           10    73    78   13*         9    55    11   15*              4     6    19  133*         8   450   449    4          7    81     9    8 
   1   631   642    3         11    59    85   16*        10     7    62  154*              5   202   199    6          9    39     4   21*         8    95   119   10 
   2   261   252    4         12   175   190    7         11    41    66   25*              6   136   147    8             -6,    7,  L           9   158   166    6 
   3   650   660    3              3,    6,  L               7,    6,  L               7    83    72   13          1   138   139    7         10   213   216    5 
   4    50    14   11*         0   318   310    3          0    47    95   19*                  12,    6,  L           2   565   551    4         11    54    24   16*
   5   159   167    5          1   294   300    4          1   504   505    4               0   316   317    5          3   149   152    6             -1,    7,  L   
   6   141   137    5          2   475   459    3          2   187   197    5               1   163   151    7          4   344   339    4          1   140   134    6 
   7   125   122    6          3     5    48  134*         3   265   260    4               2   227   210    5          5    86    72    9          2   301   304    4 
   8     6    40  122*         4   254   256    4          4    47    49   17*              3   134   136    8          6    28    16   30*         3    90    90    8 
   9    93    93    9          5   301   301    4          5   103   106    8               4   156   140    7          7   191   191    6          4    15    33   48*
  10   101    92    8          6   135   131    7          6     6    29  136*              5    73    76   14*         8   225   233    5          5   136   132    6 
  11   212   213    5          7   233   224    5          7   393   410    4                   13,    6,  L           9   208   201    5          6   308   308    4 
  12   211   229    6          8   222   224    5          8   119   120    8               0   158   159    7         10   124   127    8          7   224   238    5 
  13   117   145   10          9    41    61   20*         9   351   354    5               1    39    65   26*            -5,    7,  L           8   339   367    4 
        0,    6,  L           10   282   289    5         10    83    85   12               2    83    17   10          1   376   373    4          9    89   105   10 
   0     5    27  106*        11   238   221    5              8,    6,  L               3   133   133    8          2     6    45  142*        10   125   114    7 
   1   137   137    5         12    76   126   16*         0   196   197    5                  -11,    7,  L           3   462   475    4         11   130   126    8 
   2   254   244    4              4,    6,  L           1   269   275    4               1   115   108    8          4   514   527    4              0,    7,  L   
   3   446   452    3          0   105   114    6          2    38    12   19*              2     7    11  133*         5   192   184    5          1     6    84  155*
   4   162   157    5          1    92    86    8          3   394   391    4               3     7     9  141*         6   443   457    4          2    71    87   11 
   5   476   480    3          2   278   275    4          4    89   102    9               4    73    97   14*         7     6    18  132*         3   181   185    5 
   6   269   255    4          3   125   114    6          5   470   476    4               5    78     7   11          8   243   247    5          4   328   332    4 
   7   297   296    4          4   316   335    4          6    64    69   14*                 -10,    7,  L           9     7    19  138*         5    43    70   19*
   8   390   383    4          5    79    69   10          7   381   373    4               1   107    94    9         10    98    85    9          6    70    71   12*
   9    29    25   26*         6   369   372    4          8    46    81   21*              2     7    16  136*            -4,    7,  L           7    51    19   16*
  10    45    54   19*         7   274   270    5          9    62    13   13*              3    76    73   10          1    40    65   19*         8   197   196    6 
  11    72    27   12          8   287   293    4         10     7    36  154*              4   101    88    9          2   485   474    4          9    53    70   19*
  12    63     6   14*         9   207   202    5              9,    6,  L               5    94   113   10          3   118   117    7         10   244   233    6 
  13    56    21   15*        10     6    35  141*         0   278   283    4               6    95    90   10          4   136   121    6         11   116   114    9 
        1,    6,  L           11   112   109    9          1   112    97    8                   -9,    7,  L           5   115   137    7              1,    7,  L   
   0   242   243    4         12    85    60   12          2   133   128    6               1    50    18   16*         6    79    75   11          0   111   106    7 
   1   611   615    3              5,    6,  L           3    10    15   76*              2   105   120    8          7    58    62   14*         1    46    96   18*
   2    32    18   20*         0   424   423    3          4    33    62   26*              3     6     5  132*         8    66    82   13*         2    89   115    9 
   3   495   491    3          1   292   286    4          5   183   174    6               4    29    33   29*         9   112   116    8          3     6    75  154*
   4    49    29   14*         2   483   485    3          6    34    10   28*              5    66    56   13*        10   168   171    6          4   288   281    4 
   5    84    91    9          3   446   439    4          7   242   234    5               6   157   160    7             -3,    7,  L           5     6    10  142*
   6    41    14   19*         4   222   226    5          8   129   122    8               7     7    29  138*         1   367   372    4          6   327   322    4 
   7   325   335    4          5   222   216    5          9   215   216    6                   -8,    7,  L           2    64    52   12*         7     6    63  146*
   8   289   286    4          6   134   125    7             10,    6,  L               1    73    96   12          3   280   268    4          8   313   306    5 
   9   294   311    5          7   168   164    6          0    80    93   10               2   225   222    5          4    70   102   12*         9   127   146    8 
  10   172   177    6          8   198   204    6          1    84   103   11               3   235   239    5          5   377   375    4         10   230   237    6 
  11    66    32   13*         9   410   399    4          2    16    41   52*              4   239   234    5          6   217   222    5         11   161   164    7 
  12   131   133    8         10    79    43   12          3   368   358    4               5   169   174    6          7   185   191    5              2,    7,  L   
```

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    COLUMNS ARE 10FO,10FC,  10SIG                                                    COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG          L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
      2,  7,  L           4   167   167     6      5   210   193     6              -1,  8,  L           0    90    89     9           -3,  9,  L
 0   401   399     4      5   113    86     8           -7,  8,  L               1    57    19    14*     1   304   303     5      1   247   238     5
 1   212   207     5      6   167   177     7      1   182   199     6           2   121   106     7      2    47    58    21*     2   281   276     5
 2   474   459     4      7   143   125     7      2   186   182     6           3    81   100    11      3    93    80     9      3   224   210     5
 3   270   264     4      8     7    25   139*     3    51     7    16*          4    70   117    12*     4   160   151     6      4   316   310     5
 4   209   223     5      9   118   125     9      4   226   216     5           5    60     6    13*     5    98   121    10           -2,  9,  L
 5   156   156     6           7,  7,  L           5   388   389     4           6     6    48   130*     6   207   208     6      1   140   137     7
 6    33    45    24*     0   292   294     4      6    47    68    22*          7   131   121     7      7    68   107    15*     2     7    32   145*
 7   162   158     6      1    82    78    10           -6,  8,  L               8    88    92    10      8   142   122     8      3     7    24   149*
 8   267   267     5      2   333   323     4      1   450   454     4           9   268   271     5           5,  8,  L           4   247   243     5
 9    85    87    12      3   161   162     6      2    69    77    14*               0,  8,  L           0   274   266     5      5    11    49    77*
10   293   290     5      4    67    30    12*     3   339   325     4           0   415   424     4      1   269   267     5           -1,  9,  L
11   109   123    11      5    99   109    10      4   194   193     6           1    73   124    13*     2   104   115     9      1    99    80    10
      3,  7,  L           6    85    60    10      5   165   163     7           2   477   481     4      3   396   388     4      2   298   295     5
 0     6    34   145*     7    59    31    14*     6   150   139     7           3   299   314     5      4    63    90    16*     3   186   188     6
 1   258   253     4      8     7    28   145*     7    88    62    11           4   155   156     7      5   287   280     5      4   173   172     6
 2   384   378     4           8,  7,  L                -5,  8,  L               5     6    32   140*     6    48    92    21*     5   136   140     8
 3    88    74     9      0   178   167     5      1    29    15    30*          6    73    59    12      7   134   119     8            0,  9,  L
 4   475   479     4      1   181   179     6      2    23    51    36*          7   250   246     5           6,  8,  L           1   253   263     5
 5    40    29    20*     2     6    28   142*     3   202   185     5           8     7    15   142*     0    81    26    10      2   187   189     7
 6   458   451     4      3    33     8    24*     4    84    75    11           9   324   319     5      1   375   363     4      3   166   174     7
 7     6    39   142*     4   130   131     7      5   266   272     5                1,  8,  L           2    36    61    25*     4   165   162     7
 8   225   229     5      5    42    24    22*     6   192   201     6           0    94    98    10      3   176   188     6      5    56    11    16*
 9    96   101    10      6    79    67    12      7   428   425     5           1     6    31   137*     4   105   122    10            1,  9,  L
10    32    14    31*     7    51    54    18*     8   147   137     7           2    93    84     9      5    56    78    16*     0   404   397     4
      4,  7,  L                9,  7,  L                -4,  8,  L               3    51   121    20*     6    26    45    35*     1     7    51   136*
 0   419   411     4      0    73    90    12      1   528   548     4           4   164   170     6           7,  8,  L           2   210   205     6
 1   173   157     5      1     6     7   129*     2    88    99    11           5   393   376     4      0     6    45   141*     3    63     7    14*
 2   566   555     4      2    92    81     9      3   455   467     4           6   189   194     6      1   268   268     5      4    52    91    20*
 3   156   140     6      3    45    81    21*     4    23     8    34*          7   418   418     5      2   109   104     9      5   159   163     7
 4   228   240     5      4   159   155     7      5     6    26   144*          8    45    68    23*     3    63    72    16*           2,  9,  L
 5    62    65    14*     5   203   199     6      6    77    92    12           9   318   308     5      4     7    68   145*     0     7    27   148*
 6   252   242     5      6   193   190     6      7    83    81    11                2,  8,  L           5    31    30    28*     1   183   196     6
 7   294   295     5          10,  7,  L           8     7     8   145*          0   313   306     4           8,  8,  L           2   146   117     7
 8   325   320     5      0   100    99    10           -3,  8,  L               1   142   146     7      0    63    73    14*     3   269   276     5
 9   267   265     5      1    45     8    19*     1     6    30   131*          2   266   274     5      1   145   137     7      4   165   167     7
10   177   176     7      2   111    91     8      2   204   204     5           3   205   203     5      2    66    54    15*     5   249   235     6
      5,  7,  L           3   169   172     7      3   237   238     5           4   289   290     5      3   103   112    10            3,  9,  L
 0   419   438     4      4    90    70    10      4    75    10    11           5   101    89     9      4    44    81    23*     0   121   129     9
 1   298   295     4      5   102   121    10      5   272   278     5           6    81   104    13           9,  8,  L           1    69    94    16*
 2   441   443     4          11,  7,  L           6     6    61   129*          7   242   220     5      0    98    98    10      2    81    72    12
 3    50    75    18*     0    58    49    16*     7     7    16   125*          8    90    68    11      1     7    41   132*     3   134   146     8
 4   459   449     4      1     7    18   137*     8   190   199     6                3,  8,  L           2    60    54    16*     4   121   127    10
 5     6    34   141*     2   278   281     5           -2,  8,  L               0   151   151     6          -5,  9,  L                4,  9,  L
 6   172   173     6      3    60    60    17*     1   153   153     6           1   213   206     5      1   258   252     5      0    39    53    24*
 7    60    53    15*          -9,  8,  L           2   123   125     7           2   154   149     6      2    93    96    11      1   160   133     7
 8    94    49    10      1     7    64   144*     3   177   182     6           3   307   298     5      3   144   150     8      2    75    48    12
 9    31    13    30*     2   111   105     8      4    59    16    13*          4    79    87    12          -4,  9,  L           3   219   200     6
10    70     8    13*     3     7    35   139*     5    81    61    10           5   357   349     5      1     7    34   134*           5,  9,  L
      6,  7,  L               -8,  8,  L           6    69    61    12*          6   183   188     7      2     7    30   137*     0    93    71    10
 0   532   516     4      1   109   114     9      7    80    56    10           7   426   407     4      3    50    32    17*     1   256   271     6
 1    77    23    10      2    87    89    11      8    63   138    16*          8    86   100    12      4    96   117    11      2   157   170     8
 2   182   184     6      3   300   282     5      9    99   106    10                4,  8,  L
 3    87    92    11      4    98   112    11           -1,  8,  L
```

OBSERVED AND CALCULATED
STRUCTURE FACTOR LISTINGS FOR
$CpRe(CO)_2(C_2Ph_2)$

```
COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC   SIG
    -11,   0,  L
 5   820   821   22
    -11,   1,  L
 1   840   864   23
 2   413   354   38
 3   309   243   46
 4   536   490   31
 5   635   680   28
 6   373   356   43
    -11,   2,  L
 4   483   532   35
 6   535   526   31
 7   333   111   43
 8   730   693   24
    -11,   3,  L
 1   479   420   34
 2   456   482   37
 4   708   732   25
 5   345   361   48
    -11,   4,  L
 1   334   226   48
 2   653   634   27
 3   344   264   48
 5   482   476   35
    -10,   0,  L
 2  1458  1506   16
10   925   908   19
12   581   580   29
    -10,   1,  L
 1   534   526   29
 2   916   907   20
 3   387   473   39
 5   532   531   28
 6  1088  1056   18
 7   764   745   22
 9   321   111   44
10   643   617   26
11   329   358   47
13   299    67   48
    -10,   2,  L
 1   898   942   20
 3   726   737   23
 4   540   513   27
11   928   911   20
    -10,   3,  L
 3   777   819   22
 5   517   506   29
 6   498   419   30
 7   902   882   20
 8   305   358   50
 9   370   379   41
11   319   363   48
12   371   354   41
    -10,   4,  L
```

```
 L   KFO    FC   SIG
 1   473   505   33
 2   678   664   24
 4   515   466   30
 5   564   544   28
10   573   564   28
    -10,   5,  L
 1   590   582   28
 2   523   534   31
 6   839   840   22
 9   429   377   36
10   437   430   37
    -10,   6,  L
 2   447   410   35
 3   617   566   27
 4   548   552   31
 5   419   370   38
 8   470   473   35
    -10,   7,  L
 3   386   383   42
 4   328   372   50
 5   346   370   44
     -9,   0,  L
 1   530   500   26
 3  1383  1453   15
 5   950   960   18
 7   924   922   17
 9   284   292   46
13   555   533   28
15   527   531   30
     -9,   1,  L
 2   286   209   44
 5   290   352   47
 7   608   634   24
 8   801   781   19
 9   833   824   19
11   663   656   23
12   632   628   25
13   664   660   24
     -9,   2,  L
 2   588   590   24
 3   328   413   42
 4  1551  1530   14
 7   427   420   31
 8   696   705   21
12   413   406   35
     -9,   3,  L
 1   771   754   20
 2   536   535   26
 3   471   454   30
 4   285   248   46
 5   286   328   47
 7   575   576   25
 8  1052  1044   17
12  1062  1056   18
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```
 L   KFO    FC   SIG
13   411   380   37
     -9,   4,  L
 3  1239  1275   16
 4   537   528   27
 5   609   610   24
 6   535   544   27
 7   438   422   32
 9   423   408   33
10   495   491   30
11   298   337   47
13   316   228   45
14   399   356   38
     -9,   5,  L
 1   289   139   45
 2   908   921   19
 3   295   226   47
 4   310   376   44
 5   295   331   48
 6   758   744   21
 7   561   565   27
 9   564   532   27
11   673   667   24
13   409   436   39
     -9,   6,  L
 1   587   619   27
 2   528   529   29
 3   559   568   28
 4   751   779   22
 7   739   737   22
 8   321   328   45
10   491   482   32
11   504   443   31
     -9,   7,  L
 1   909   884   20
 3   439   416   35
 5   849   847   21
 7   426   432   36
 8   553   493   28
10   345   126   44
     -9,   8,  L
 1   328   317   46
 4   322   336   47
 5   430   455   36
 6   490   462   32
 9   563   589   30
     -9,   9,  L
 2   887   885   22
     -8,   0,  L
 2   591   623   21
 4   911   904   16
 6  1483  1463   13
 8  1108  1092   14
10  1283  1279   14
12   408   386   32
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COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC   SIG
     -8,   0,  L
14   302   255   46
18   609   559   28
     -8,   1,  L
 1  1340  1374   14
 2   861   857   16
 4   738   736   18
 5   653   657   20
 6   443   430   28
 9   312   320   41
10   605   609   23
12   389   368   34
13   724   687   21
14   907   884   19
17   438   410   35
18   645   557   26
     -8,   2,  L
 2   633   647   20
 5  1477  1469   12
 6   353   342   35
 7   399   409   31
 9  1608  1578   13
11   429   447   31
13   507   483   28
17   415   387   37
     -8,   3,  L
 1  1534  1523   13
 2   759   785   19
 3   274   141   41
 5   913   926   17
 6   522   514   24
 8   647   606   21
 9   384   327   33
11   455   428   29
12   302   346   46
13   954   951   18
14   491   502   31
15   497   496   30
17   636   626   26
     -8,   4,  L
 1   353   324   36
 3   594   613   23
 4   916   924   17
 5   433   467   30
 6  1093  1075   15
 7   286   238   44
 8   681   670   21
 9   656   669   22
10  1031  1034   17
11   310   191   43
13   396   351   35
15   434   402   34
     -8,   5,  L
 1   295   311   46
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 L   KFO    FC   SIG
 2   944   983   18
 3   734   690   20
 4  1022  1041   16
 6   299   264   44
 7  1108  1110   16
 9   369   347   37
10   491   500   29
11   475   488   30
14   833   816   20
     -8,   6,  L
 2   781   740   20
 3   744   738   21
 5  1008  1023   17
 6   678   713   23
 8   563   536   26
 9   611   600   24
10   333   279   40
11   534   535   28
12   582   634   28
15   388   425   41
     -8,   7,  L
 1  1203  1248   16
 4   761   794   22
 5   611   655   25
 6   734   755   22
 8  1072  1078   17
 9   284   153   45
10   394   438   38
12   561   548   28
13   450   430   34
     -8,   8,  L
 2   675   679   24
 3   698   693   23
 4   870   888   19
 5   410   405   35
 9   399   401   36
10   506   485   30
12   602   576   28
     -8,   9,  L
 3   666   691   25
 6   340   311   43
 7  1096  1105   18
     -8,  10,  L
 3   994   976   20
     -7,   0,  L
 1   727   737   16
 3  1009  1033   13
 7  1150  1146   13
 9   349   382   32
11  1877  1906   12
15  1140  1165   16
17   313   195   44
     -7,   1,  L
 1   385   414   27
```

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 L   KFO    FC   SIG
 2  1249  1282   12
 3  1747  1728   11
 4   562   558   19
 5   746   755   16
 6  1264  1261   12
 7  1662  1664   11
 8   773   752   16
 9   408   414   28
10   336   323   34
11   278   333   45
14   433   410   30
15   689   680   22
16   501   487   29
18   297   288   47
19   743   699   23
20   547   526   31
     -7,   2,  L
 1   961   954   14
 2  1438  1431   12
 5   660   665   18
 6   541   523   20
 8   514   506   22
 9   279   230   41
10  1465  1494   13
11   446   456   28
12  1069  1060   15
14   982   982   17
16   764   722   20
     -7,   3,  L
 1  1262  1279   13
 2  1701  1713   11
 3   514   500   21
 4   573   583   20
 5   370   338   29
 6  1546  1583   12
 7   813   815   16
 8   473   458   24
 9   601   578   20
10   764   783   19
11   378   423   34
13   566   550   24
16   509   534   29
18   429   412   35
     -7,   4,  L
 1   690   694   18
 2   862   855   16
 3   897   906   15
 4  1231  1216   13
 5   550   540   22
 7   366   382   32
 8   884   879   16
 9   636   602   20
10   281   281   44
11  1354  1360   14
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    COLUMNS ARE 10FO,10FC,  10SIG                                        COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG
     -7,  4,  L          1   396   425   36    12   267   133   41        -6,  6,  L          2  1160  1168   17     9   741   751   14
 12   415   425   32     3   297   230   44    13  1235  1210   13     2   448   421   25     3   366   293   39    10  1353  1344   11
 14   325   386   41     5   298   170   41    14   415   392   31     3  1061  1076   14     6   700   741   24    13   650   675   19
 15   832   850   19     6   292   306   45    15   678   650   20     4   865   873   16        -6, 12,  L         14  1611  1628   12
 18   494   469   31     8   570   588   27    17  1297  1303   15     5  1210  1219   13     6   740   749   24    15   617   631   22
     -7,  5,  L          9   302   273   48    21   537   508   31     6  1260  1257   13        -5,  0,  L         16   276   195   43
  1   462   474   26    10   703   730   23        -6,  3,  L          7   400   355   29     1  1303  1287    9    18   746   726   20
  2   387   441   31    12   678   663   24     1   365   374   26     8   317   331   36     3  1095  1070   10    19   538   557   28
  3  1488  1496   12        -7, 10,  L          2  1654  1649   10     9   710   729   19     5  3138  3135    8        -5,  4,  L     
  5   652   691   20     1   496   481   31     4   396   398   24    10   987   980   16     7  1451  1408    9     1   990   998   12
  7  1237  1235   14     4  1055  1063   19     5  1461  1454   11    11   810   815   18     9  2569  2623    9     2  1283  1291   10
  8   870   841   17     5   535   546   30     6   936   938   13    14   408   424   33    11   702   697   15     3   596   606   16
  9   479   471   27     6   527   539   30     7   996   982   13    15   297   321   47    13   326   324   32     5  2202  2174    9
 10   401   420   32     8   786   750   22     8   847   858   14    16   315   285   43    15   602   637   22     6   891   907   12
 12   834   847   18        -7, 11,  L          9  1459  1459   11    17   494   513   31    17   607   592   23     7   571   551   17
 14   460   452   31     1   682   673   26    10   319   323   33        -6,  7,  L         19  1013  1000   17     8   873   856   13
 15   488   470   30     4   337   214   44    11   996  1032   14     2  1751  1754   12    21   602   588   27     9  1184  1234   12
     -7,  6,  L             -7, 12,  L         12   587   575   22     4   383   414   31        -5,  1,  L         10   417   364   24
  1   913   956   17     1   535   562   34    13   955   931   16     5   278   257   40     1  2573  2570    8    11   889   897   15
  2   536   510   24        -6,  0,  L         14   828   841   18     6  1051  1054   15     2  1791  1800    8    12   844   855   16
  4  1120  1109   15     4  2992  3013    9    15   442   451   30     7   710   679   19     5  1006  1009   11    16   452   519   30
  5  1076  1074   16     6   313   317   27    19   321    30   42     9   716   738   20     6   662   648   14    17   704   705   22
  6   547   571   25     8   851   832   14    21   503   512   33    11   408   442   32     7   560   539   15    18   345   361   40
  7   624   595   22    12  1147  1147   13        -6,  4,  L         13   377   362   36     8   556   576   16    19   539   534   28
  9   518   526   26    14   298   359   41     1   732   729   16    14   838   798   19     9  1111  1130   11    21   414   391   38
 10   772   766   20    16  1574  1516   13     2   585   605   18    16   433   370   33    10   994   995   12        -5,  5,  L     
 11   582   566   24    18   791   749   20     3  1132  1157   12    18   704   675   25    11   840   816   14     1  2215  2275   10
 12   492   528   29    20   650   601   25     4  1976  2023   10        -6,  8,  L         13  1251  1272   12     2   900   905   13
 13   299    82   43        -6,  1,  L          6   659   663   17     1   316   386   39    14  1395  1383   12     4  1296  1251   11
 16   583   529   27     1   893   905   13     7  1207  1242   13     3   700   729   20    15  1176  1139   14     5   758   765   14
 17   423   431   38     2   490   501   19     8   572   568   19     4   483   529   27    16   498   518   27     6   799   803   14
     -7,  7,  L          3   595   601   16     9   774   788   17     6   925   899   16    17   734   746   20     7   415   402   23
  1   903   901   18     4  1006  1003   12    10   541   544   22     7   679   710   20    18   670   602   22     8  1833  1810   10
  2   763   803   20     5   316   291   27    11   608   604   21     9   393   402   33    19   296   306   47     9   611   621   19
  3   285   315   45     7  1440  1412   11    12   861   861   17    10   963   974   17        -5,  2,  L         11   763   729   16
  4   497   476   27     8  2229  2198   10    13   707   731   20    11   553   583   25     2  1304  1298    9    12   580   600   22
  5   279   168   45     9  1199  1188   12    14   307   344   41    14   481   462   30     3   936   964   11    13  1151  1113   14
  6  1110  1124   16    11   618   621   19    16   915   917   18        -6,  9,  L          4  1678  1695    9    15   433   420   30
  8   369   374   36    12  1391  1358   12    18   485   489   31     1  1632  1616   13     5   520   524   17    17   613   645   24
  9  1090  1075   16    13   888   892   16    19   327   300   45     2   397   393   34     6  2623  2607    8    20   666   602   24
 13  1112  1070   17    15   310   214   39        -6,  5,  L          3   676   718   22     8  1057  1054   11        -5,  6,  L     
     -7,  8,  L         20   377   360   40     1  1315  1329   12     5   918   950   17     9   719   712   14     1   374   381   27
  1   561   546   25    21   501   450   32     2   423   400   25     7   329   364   40    10  1823  1805   10     2   247    71   38
  2   483   508   29        -6,  2,  L          3  1407  1390   12     8   456   473   29    11   279   321   36     3  1135  1144   12
  3   770   747   20     1  1634  1600   10     4   838   814   15    12   299   243   45    13   522   549   22     4  1263  1231   12
  4   703   710   21     2   226   118   36     5  1053  1037   13    15   775   752   23    15   291   329   43     5   984   993   13
  5  1143  1158   16     3  2273  2260    9     6   370   370   28        -6, 10,  L         18  1137  1133   16     6  1119  1144   12
  6   316   335   43     4   641   619   16     7   779   793   16     5   576   578   25    22   835   807   22     9  1100  1089   13
  8   728   737   21     5  1595  1618   10     8  1419  1395   12     7   738   714   21        -5,  3,  L         10   991   998   15
  9   589   622   25     6   630   633   16    12  1161  1117   14     9   665   670   24     1  1482  1512    9    11   530   534   23
 12   301   255   46     7   895   918   13    13   533   529   26    10   516   544   30     2  1522  1523    9    12   692   689   20
 13   309   318   47     9   262   270   37    15   768   800   20    11   855   843   21     3  1067  1063   10    13   703   703   20
 14   404   393   38    10   567   567   20    17   303   260   43        -6, 11,  L          4  1516  1500    9    15   414   396   32
     -7,  9,  L         11   532   528   21    19   540   550   30     1   515   546   31     7  1611  1601    9    16   792   763   20
```

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   COLUMNS ARE 10FO,10FC,  10SIG                                                     COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG     L   KFO    FC   SIG
      -5,  6,  L          8   457   480    32    16   491   525    26         -4,  5,  L         11   514   491    25    13  2405  2444     9
 18   434   405    34     9   481   456    31    17   310   188    40    12   408   416    27    13   325   339    39    15   622   627    18
 19   335   353    46    11   487   528    33    23   687   629    25    13  1193  1196    13    15   478   503    30    17  1773  1753    12
      -5,  7,  L             -5, 12,  L             -4,  3,  L         14   338   344    36    16   606   616    24    21   563   550    27
  2  1055  1065    13     2   316    77    44     1  1013  1037    10    15   330   313    37    17   615   602    26         -3,  1,  L
  3  1569  1583    11     3   853   851    21     2  1568  1572     8    16  1061  1062    15    18   794   793    22     2   701   689    10
  4   466   479    24     4   347   302    42     3  3581  3597     7    18   450   408    30         -4,  9,  L          3   275   297    21
  5   382   383    28         -5, 13,  L          4   889   868    10    20   676   654    24     1  1208  1223    13     4  1432  1395     7
  7  1622  1627    11     2   319   365    51     5   579   585    14    21   435   467    37     2   656   680    19     5  2236  2181     7
  9   610   626    21     4   435   443    37     6   655   649    13         -4,  6,  L          5   555   584    22     6   192   251    31
 10   644   665    21     5   319   199    50     7  2055  2064     8     1   513   515    18     6   493   527    25     7  1704  1688     7
 11   615   590    22     6   334   332    47     8   861   853    12     2  1097  1101    11     8   443   423    27     8  2079  2129     7
 14   648   668    22         -4,  0,  L          9   439   436    19     3   777   786    13     9  1395  1400    14     9  2019  2015     8
 15   314   242    40     2  1344  1348     8    10   485   502    19     4  1421  1426    10    13  1233  1207    15    10   852   847    11
 17   452   418    33     4   266   291    22    12   460   484    22     5  1324  1327    11    14   292   338    48    11   683   683    13
 18   361   406    43     6  1597  1589     8    13   604   598    18     6   476   489    21    17   329   243    46    12  1063  1046    11
 19   353   417    46     8  1678  1673     8    14   834   839    16     7   870   862    13         -4, 10,  L         13   995   999    12
      -5,  8,  L         10  1837  1839     9    15  1141  1092    14     8  1247  1228    12     1  1435  1425    14    14   651   620    17
  2  1077  1097    14    12  2053  2056     9    16   387   379    32     9   710   725    16     2   385   372    33    15   279   246    37
  3  1165  1173    14    14   994   981    13    17   636   631    22    10   611   625    19     3   598   596    23    17   418   387    29
  5   706   740    18    16   866   852    16    19   954   979    18    11  1215  1223    13     4   564   568    24    20   623   593    23
  6   714   748    18    20   639   633    24    20   463   465    32    12   454   470    25     5  1541  1523    13    21   814   794    21
  7   297   283    39         -4,  1,  L         21   309   278    47    13   458   470    27     8   382   379    34    22   443   451    35
  8   601   572    21     1  1282  1289     8    23   402   349    41    14   903   912    17     9   674   675    21         -3,  2,  L
 10   281   310    43     2  2209  2219     7         -4,  4,  L         15   603   590    22    13   526   516    29     1   591   579    12
 11  1011  1032    16     3  1621  1620     8     1  1344  1337     9    17   807   807    20         -4, 11,  L          2   410   394    15
 12   809   829    18     4   576   596    12     2   925   935    11    18   690   703    23     1   411   416    35     3  1101  1077     8
 13   477   500    29     5   876   864    10     3   535   555    15    19   412   354    36     6   417   424    33     4  3455  3457     6
 15   691   695    22     6  2089  2079     8     4   636   632    13    20   322   240    44     8   760   769    21     5  1100  1109     8
 16   428   482    36     7  1421  1395     9     5  1705  1722     9         -4,  7,  L          9   508   528    29     6   612   572    11
 17   607   590    26     8  1123  1105     9     6  1206  1189    10     1   568   592    18    10   806   797    20     7  1253  1227     8
 18   323    74    44    10   332   344    24     7   725   722    13     2   441   448    22    12   803   797    22     8   335   349    21
      -5,  9,  L         12   324   328    28     8   798   799    13     3  1311  1296    11    13   518   550    32     9   689   712    12
  1   644   632    22    13   248   220    37     9   566   593    17     4   227   147    38    14   768   756    23    12  1789  1777     9
  2   705   749    20    14   438   467    25    10  1651  1653    10     5   425   431    23         -4, 12,  L         13   499   518    20
  3   304   337    41    15   940   971    15    11  1024  1020    13     6   633   631    17     2   525   530    29    14  1089  1125    13
  4  1315  1341    14    16  1105  1097    14    12  1664  1675    11     7  1282  1287    12     4  1248  1230    16    16  1723  1746    12
  6   853   869    17    18   284   325    46    13   391   398    28     8  1282  1292    12     5   327   245    43    18  1001   993    16
  7   369   406    34    19   832   818    19    14   424   461    27    10  1453  1485    12     6   678   714    24    20   513   473    27
  8  1199  1193    15    20   960   955    18    15   625   621    21    12   690   696    19     8   631   646    25    23   320   218    46
 10   316   335    40    21   407   401    37    16   749   737    19    14   782   808    19         -4, 13,  L              -3,  3,  L
 13   491   423    28         -4,  2,  L         21   359   360    43    15   662   641    21     1   594   583    28     1   940   912     9
 16   544   517    29     1   743   727    10    22   329   333    47    16   309   207    41     6   342   302    43     2   496   497    13
      -5, 10,  L          2   932   945     9         -4,  5,  L         19   545   576    30     7   364   325    42     3  1584  1597     7
  2   983   952    17     3   793   801    10     1   856   866    12         -4,  8,  L          9   691   689    25     4  2281  2271     7
  3   455   454    30     4  1655  1634     8     2  2497  2475     8     1  1415  1473    12         -4, 14,  L          6   653   615    11
  4   771   766    19     5   523   499    14     3   518   533    17     2   461   463    24     1   716   713    26     7  1073  1078     9
 10   329   357    42     7  1793  1815     8     4  1889  1886     9     4  1495  1537    11         -3,  0,  L          8  2770  2769     8
 12  1006   998    18     8   615   601    14     5   608   591    15     5  1004  1017    13     1  2986  2965     6     9  1256  1251     9
 15   355   348    45    11  2610  2601     9     6  1678  1715     9     6  1015  1030    14     3  2291  2260     6    10   484   477    17
      -5, 11,  L         12   388   392    25     7   580   592    16     7   670   682    18     5  1882  1876     7    12  1729  1734    10
  3   964   997    18    13   468   456    22     8   816   819    13     8   364   373    30     7   627   627    11    13   761   789    15
  4   448   485    33    14   493   504    23     9  2089  2095    10     9   342   375    34     9   388   399    19    15   801   831    16
  7  1123  1139    17    15  1163  1144    13    10   668   647    17    10   589   569    21    11   546   566    16    16   281    86    38
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -3,   3,  L
 18   768   759    19
 19   314   332    43
 20   552   535    26
 22   727   715    23
      -3,   4,  L
  1  2073  2060     8
  2   531   514    13
  3  2145  2123     7
  4  1259  1247     8
  5  2189  2182     8
  6  1603  1584     8
  7   404   412    19
  8   563   572    15
 10  1385  1427    10
 11  1042  1098    12
 13  1418  1388    11
 14   694   699    18
 15   436   474    27
 16   578   587    23
 17  1420  1444    14
 18   304    21    41
 20   355   304    38
      -3,   5,  L
  1   324   303    22
  2  2019  2023     8
  3   610   612    13
  4   476   472    16
  5  1810  1791     8
  6   767   772    12
  7   782   766    12
  8   479   479    18
  9  2228  2254     9
 10   811   799    14
 11   893   924    13
 12   440   442    24
 13   913   896    14
 14  1136  1153    14
 16   331   349    38
 17   515   476    27
 18   610   603    23
 21   764   749    22
      -3,   6,  L
  1   924   955    12
  2   401   392    20
  3  1334  1323     9
  4  1198  1204    10
  6  1458  1440     9
  7  1700  1689     9
  8   419   380    21
  9   771   764    14
 10   898   914    14
 11  1121  1119    13
 12  1265  1284    13
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  L   KFO    FC   SIG
 13   753   776    17
 16   610   580    22
 17   511   519    27
 18   631   650    24
 21   302   235    50
 22   338   384    51
      -3,   7,  L
  1  2052  2074     9
  3  1677  1710    10
  4   729   740    14
  5  1009  1024    11
  6   301   253    28
  7   529   521    19
  8  1478  1528    11
 10   592   566    19
 11  1169  1149    13
 12   774   774    17
 13   334   357    36
 15  1362  1376    14
 19   741   729    23
 20   371   335    42
      -3,   8,  L
  1   274   375    35
  2   336   348    28
  4   777   765    14
  5  1235  1261    11
  6  1152  1142    12
  7  1097  1123    13
  8   484   494    23
  9   673   691    18
 10   939   971    15
 11  1184  1193    13
 13   297   140    39
 14   413   410    31
 16   317   322    42
 19   596   610    28
 20   464   514    37
      -3,   9,  L
  1   535   531    22
  2  2268  2284    10
  3   562   563    20
  6   931   952    15
  7   779   795    16
  9   416   460    29
 10   580   580    22
 12   493   505    27
 13   360   388    37
 14  1182  1177    16
 15   333   315    42
 16   854   869    20
 18   546   550    31
      -3,  10,  L
  3   634   639    20
  5   269   225    43
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  L   KFO    FC   SIG
  6  1413  1415    13
  7   631   631    21
  8   382   341    32
  9   298   254    41
 10  1311  1322    14
 14   357   350    40
      -3,  11,  L
  1  1368  1374    14
  2   866   855    18
  3  1016  1025    16
  4   279   150    45
  5   604   634    23
  6   413   402    31
 11   592   584    25
 14   497   502    33
 15   902   911    21
      -3,  12,  L
  3   519   512    27
  5   618   622    24
  7  1111  1131    17
  9   714   689    22
 11   877   904    21
      -3,  13,  L
  1   706   675    24
  2   986  1001    18
  3   524   563    30
  5   564   539    27
  6   463   398    32
 10   345   353    45
      -3,  14,  L
  1   329   331    49
  6   885   875    22
      -2,   0,  L
  2  4818  4841     5
  4  2025  2004     6
  6  3380  3324     6
  8   672   656    10
 10  2060  2062     8
 14   647   652    16
 18  2033  1986    12
 20   481   535    30
 22   882   863    19
 24   340   353    47
      -2,   1,  L
  1  3007  2962     5
  2  2961  2920     5
  3  1833  1807     6
  4  1464  1418     6
  5   297   298    19
  9  1557  1544     8
 10  2439  2462     8
 11  1425  1399     9
 13  1224  1223    11
 14  2021  2009    10
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -2,   1,  L
 15  1102  1110    13
 18   624   603    22
 19   326   349    41
 22   412   360    35
      -2,   2,  L
  1  2481  2411     5
  2  1264  1254     7
  3  1262  1190     7
  4   375   394    15
  5  3882  3899     6
  6  1002   971     8
  7  1708  1694     7
  8   761   747    10
  9  2124  2160     7
 10   411   395    18
 11   769   771    12
 12   606   621    15
 15   645   657    18
 16   403   434    29
 17  1025  1028    15
 19  1378  1356    14
 21   358   417    40
 23   821   788    22
      -2,   3,  L
  1  2333  2322     6
  2  1994  1965     6
  3   193   130    29
  4  1868  1855     7
  5   896   882     9
  7   993   994     9
  8  1522  1525     8
  9  1351  1348     9
 10  1008   988    10
 11  1104  1118    10
 13  1749  1751    10
 14   938   954    14
 15  1112  1119    13
 16   628   649    20
 17   690   690    20
 20   651   673    23
 22   300   300    50
 23   463   436    35
      -2,   4,  L
  1   434   419    15
  2  1238  1228     8
  3  1346  1332     7
  4  1574  1567     7
  5  1716  1659     7
  6  3588  3611     7
  7   287   293    24
  9  1839  1811     8
 10  1439  1451     9
 11   945   943    12
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  L   KFO    FC   SIG
 12   692   717    15
 13   932   952    13
 14   484   492    23
 15   978   973    14
 18  1046  1026    16
 20   301   343    46
 21   306   275    47
 22   754   726    23
      -2,   5,  L
  1   312   312    20
  2  1522  1485     8
  3  2157  2140     7
  4   819   816    10
  5   723   748    12
  6   499   475    15
  7  2116  2129     8
  9  1410  1437    10
 10  1617  1644    10
 11   241   265    38
 12   294   294    32
 14  1798  1788    11
 15   764   798    18
 17   612   612    23
 18   548   534    26
 19   726   708    21
 21   613   644    27
      -2,   6,  L
  1  1636  1614     9
  2  1643  1608     8
  3   508   470    15
  5  1898  1885     8
  6  1416  1389     9
  7  1522  1510     9
  8  1289  1263    10
  9   386   372    24
 10   446   474    22
 11  1452  1443    11
 12  1405  1425    12
 13   804   811    16
 16   817   838    18
 17   666   666    22
 19   486   482    31
 22   391   366    43
      -2,   7,  L
  1  1785  1758     9
  2   695   685    13
  4  2469  2497     8
  5   323   328    26
  6  1339  1353    10
  8  2104  2140     9
  9   883   892    14
 10   327   315    30
 11  1020  1015    13
 12   343   317    32
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  L   KFO    FC   SIG
 13   755   784    18
 15   717   698    20
 16   885   886    18
 18   308   257    43
 20   851   855    21
      -2,   8,  L
  3  1159  1194    11
  4   871   872    13
  5   504   529    19
  6   686   671    15
  7   270   115    34
  8   753   760    15
  9   921   938    14
 10   664   684    18
 11   555   568    21
 12  1164  1186    14
 13   633   692    22
 15   519   532    26
 16   681   692    22
 17   328   300    42
 18   334   332    43
      -2,   9,  L
  1   493   492    21
  2   566   552    19
  3  1905  1925    10
  4   276   395    38
  5   943   939    14
  7  1788  1764    11
  8   425   394    27
  9   524   515    22
 10   421   470    30
 11   628   637    21
 15   350   318    39
 16   329   211    43
 17   768   802    22
      -2,  10,  L
  1   327   276    33
  2   398   430    28
  3   898   930    15
  7   550   539    22
  8   383   436    32
  9   517   524    25
 11  1124  1104    15
 12   593   609    24
 13   943   945    18
 15   599   609    26
 17   493   506    33
 18   331    44    46
      -2,  11,  L
  2   665   690    21
  3   794   803    18
  4  1308  1324    14
  5   379   405    34
  6   297   262    41
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
     -2, 11,  L           1  2259  2214    5     23   636   620   28
  7   688   658   21      3  1022   971    6          -1,  5,  L
  8  1209  1202   15      4   712   659    8      2   927   932    9
 12   296   243   47      5  2439  2417    6      3  3196  3161    7
 16   535   537   32      6  2681  2667    6      4  1005  1008    9
     -2, 12,  L           7   538   533   12      5   296   284   22
  2   639   623   23      8  2542  2535    7      6   544   541   14
  3   293   121   42     10  2174  2184    8      7  1848  1843    8
  4   473   454   28     11   870   905   12      8  1662  1665    9
  8   684   685   23     12  2084  2138    9      9   406   423   21
 12  1256  1249   17     13   243   345   38     10  1757  1751    9
     -2, 13,  L          14   575   589   18     11   549   542   18
  3   896   878   19     15   587   595   19     12   651   629   16
  4   757   787   22     16   610   598   20     13   549   535   20
  5   412   421   35     17   352   379   34     14   957   955   14
  7   707   711   23     20   897   911   18     15  1181  1204   14
  8   596   613   27     21   330   266   45     17   688   697   21
  9   599   594   28     24   953   924   21     19   887   897   19
     -2, 14,  L               -1,  3,  L         22   696   698   25
  2   363   373   42      1  1236  1219    6     23   335   304   49
  3   518   552   32      2  3960  3935    5          -1,  6,  L
  6   497   473   32      3  2097  2038    6      1  2211  2204    8
     -1,  0,  L           4  3296  3255    6      2   195   208   32
  1   808   806    5      5  1102  1089    8      3   656   657   12
  5   479   478   11      6  2707  2713    6      4  1357  1335    9
  7  2871  2885    6      7  1212  1199    8      5  1795  1822    8
  9   322   305   19      8   764   755   10      6  1467  1464    9
 11  2725  2799    8      9  1393  1415    8      7  1032  1021   10
 13   918   962   12     10   451   459   17      8   763   756   13
 15  1025  1036   13     11   757   765   13      9   537   533   18
 19   594   582   23     12  1057  1054   11     10   896   907   13
 21   865   900   19     13   772   797   14     11  1137  1160   12
 23   644   638   26     14   381   424   27     12  1628  1625   11
     -1,  1,  L          15   917   938   15     13   457   486   25
  1  1324  1254    4     16  1559  1606   12     14   453   458   26
  2  3107  3077    4     17   298   381   42     15   658   659   21
  3  4290  4287    4     20   979   983   18     16   659   649   22
  4  2052  1985    5     21   426   508   36     17   762   736   20
  5  1119  1107    7          -1,  4,  L         18   835   830   19
  6   955   943    8      1   282   310   20     21   348   355   45
  7  2047  1990    6      2   979   980    8     22   424   397   39
  8  1179  1158    8      4  2059  2091    7          -1,  7,  L
  9   280   286   22      5   620   612   11      1  1149  1155   10
 10   479   507   15      7  2995  2960    7      2   933   932   11
 12   465   450   18      8  1716  1711    8      3   235   251   30
 13   508   539   18     10   911   939   12      4   552   564   15
 14   843   867   14     11  2586  2627    9      5  1592  1621    9
 15  1546  1589   11     12   539   572   19      6   720   748   14
 16  1370  1351   12     13   688   699   16      8   550   561   18
 17   807   827   17     14  1095  1122   13      9  2687  2741    9
 19  1078  1068   16     15   688   688   18     12   388   414   29
 20   868   840   19     17   450   493   29     13  1173  1207   14
 21   530   554   29     18   687   721   22     16   801   789   19
     -1,  2,  L          19   500   532   29     20   568   560   28
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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
     -1,  7,  L          14   331   307   46      5  2159  2090    6
 21   571   581   29     16   341    28   44      6  1904  1894    6
     -1,  8,  L               -1, 12,  L          7   212   273   30
  1  2159  2203    9      1  1604  1636   13      8  1479  1434    8
  2  1038  1029   11      5  1272  1253   15      9  1365  1392    8
  3   409   398   21      6   393   386   35     10   803   826   12
  4  1317  1319   10     13   632   608   27     11   924   905   11
  5  2245  2305    9          -1, 13,  L         13  2616  2596    9
  6   452   461   21      1   435   466   34     16   291   448   44
  7   466   453   21      4   437   400   32     17  1696  1693   12
  8   950   972   14      5   325   348   45     20   462   512   31
  9   249   227   40      8   582   547   27           0,  3,  L
 10   401   413   27      9   699   699   24      1  1638  1585    6
 11   470   456   25     10   763   734   23      2  2441  2423    5
 12   293   287   39          -1, 14,  L          3   548   507   11
 13  1022  1024   15      1   296   108   49      4   514   488   12
 14   686   719   21      2   660   652   24      5  2974  2952    6
 15   306   313   42      4   759   752   22      7   973  1000    9
 17   823   834   20      5   295    50   47      8   924   918   10
 18   574   608   27      6   540   552   30      9  2457  2471    8
 19   584   604   27      7   477   512   35     10  1214  1250   10
     -1,  9,  L                0,  0,  L         11  1220  1210   10
  3   532   529   18      2   799   788    5     13   404   470   25
  4   348   386   28      4  4352  4443    4     14  1207  1215   12
  5   260   240   37      8   975   951    9     15   306   420   39
  6  1253  1286   12     10  1289  1259    8     16   886   878   16
  7   424   410   26     12  2654  2688    8     17   837   849   18
  8  1251  1246   12     14  2239  2210    9     19   317   386   44
  9   585   589   21     16  1608  1570   11     21  1039  1037   18
 10  1467  1477   12     18  1290  1279   14           0,  4,  L
 12   796   809   18           0,  1,  L          0   630   644   12
 13   288   169   43      2  1166  1158    4      1  1184  1151    7
 14   769   793   20      3  2002  1987    4      2   533   542   12
     -1, 10,  L           4  4051  4018    5      3  1679  1628    7
  1   842   864   16      5  1572  1562    6      4  2074  1999    7
  2  1166  1198   13      6   846   844    9      6  1444  1448    8
  4  1468  1476   12      7  1358  1375    8      7  2078  2059    8
  5   716   697   18      8  3226  3247    7      8  1001   994   10
  6  1100  1066   14      9  2372  2423    8      9   406   436   19
  8   416   432   30     10   931  1011   12     11   776   774   14
 12   377   320   34     12   960   954   12     12  1736  1722   10
 14   846   850   20     13  1192  1205   11     13   750   785   16
 17   308   362   51     15   495   498   22     14   947   929   14
 18   847   803   22     17   627   658   21     15   289   370   42
     -1, 11,  L          18   920   873   17     16   972   986   15
  1   577   570   22     20   464   515   32     17   581   653   26
  2   293   308   42     21   705   742   25     18   984   973   17
  3   272   204   43     22   981   955   19     19   445   511   34
  5   741   744   19           0,  2,  L         23   440   471   39
  8   452   495   31      0  1577  1555    5           0,  5,  L
  9  1289  1263   15      1  4432  4655    4      1  2756  2746    7
 10   482   487   30      2  1101  1061    6      3  2172  2134    7
 11   302   317   45      3  2619  2583    5      4  2815  2772    7
 13   937   938   19      4   905   881    8      6  1085  1063    9
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    COLUMNS ARE 10FO,10FC,  10SIG                                                 COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
       0,    5,   L        8   442   421    24         0,   14,   L           1,    2,   L       10   499   508    19      8  1060  1048    14
  7   327   337    23      9   894   894    15      0   388   401    44     15   695   718    18     11  1636  1632    11      9   750   780    18
  8  2209  2215     8     10  1059  1076    14      3   640   653    26     16   335   376    35     13   972   953    14     10   368   408    32
  9   558   548    16     11   694   734    19      5   345   346    46     18  1635  1610    13     15  1089  1076    15     11  1126  1135    14
 10   696   706    14     12   805   812    17      7   923   915    21     20   653   735    27     16  1089  1125    16     12  1003   997    16
 11  1608  1644    10     13   701   694    20         0,   15,   L       22   734   717    24     18   385   399    37     13  1117  1131    15
 12  1018  1020    13     17   353   365    40      1   522   531    32     24   358   410    50     20   703   693    24     16   491   511    30
 15  1377  1361    13     18   533   564    29         1,    0,   L           1,    3,   L           1,    6,   L       17   535   570    29
 16   297   226    44     19   550   562    30      1  3665  3770     4      0  2390  2337     6      0  1993  1974     8     19   317   300    50
 18   681   680    22     20   564   559    30      3  2593  2537     5      1   233   170    29      1   553   531    17     20   341    53    45
 19   397   512    38         0,    9,   L        5  4544  4561     5      2  2599  2518     7      2   719   715    14         1,    9,   L  
 20   612   636    26      1  1555  1511    10      7  1337  1326     8      3  1341  1314     8      3   853   844    13      0  1185  1193    12
 22   744   685    24      3  1657  1632    10      9  1567  1558     8      4   592   576    13      4   945   928    11      2   819   833    17
       0,    6,   L        5   596   586    19     11   362   362    22      5  1011  1006     9      5  1287  1254    10      3   905   917    16
  0  1263  1282     9      8   906   891    15     13   570   557    17      6  1399  1411     8      6  1854  1869     9      4  2397  2408    11
  1  1419  1417     8     10   526   551    25     15  1663  1725    11      7   309   296    22      7   311   329    29      5   556   522    22
  2  1184  1156     9     11  1223  1242    14     17   941   964    15      8   557   553    14      8   645   634    16      6   440   414    27
  4  1910  1871     8     15  1309  1331    15     19  1841  1843    13      9  1748  1727     9      9  1443  1427    11      8  1603  1615    12
  5  1335  1323     9     19   440   486    39     21   299   215    47     10  2918  2942     8     10   633   606    17      9   705   696    20
  6  1485  1487     9         0,   10,   L       23   817   792    22     11   821   849    13     11   747   782    16     11   320   365    42
  7  1282  1264    10      1   401   387    27         1,    1,   L       14  1984  2014    11     12  1218  1211    13     15   383   280    36
 10  1745  1723    10      3   894   886    15      0   444   422     9     15   931   974    16     13  1003  1016    15     16   932   905    19
 11  1102  1116    13      5  1306  1277    13      1  3211  3259     4     17   585   596    23     14   699   702    20         1,   10,   L  
 13  1169  1164    13      6   796   786    17      2  1577  1551     5     24   386   470    48     15   727   759    20      0  1518  1570    12
 14   816   805    17      7  1706  1740    13      3   238   253    22         1,    4,   L       18   848   846    21      2   361   371    37
 16   753   763    20      9   376   398    35      4   728   719     9      0  1143  1100     8     19   497   547    33      3   437   440    30
 17   828   793    20     10   431   411    31      6   927   923     9      1  3389  3404     7     21   386   467    46      5   467   477    28
 19   509   493    30     11   902   913    18      7  1048  1036     8      2  1928  1894     8         1,    7,   L        8  1220  1222    15
 20   686   640    24     17   314    92    46      8   703   728    11      3  1303  1265     9      0   721   722    13      9   581   573    25
       0,    7,   L            0,   11,   L        9   894   912    10      4   324   346    22      1   349   368    26     12  1308  1319    15
  1   247   268    32      1   599   589    22     10  1902  1956     8      5  2514  2523     7      2   642   655    17     13   361   346    41
  2  2843  2850     8      2  1602  1536    12     11  2035  2052     9      6  1422  1414     8      3  2942  2972     9     16   660   642    26
  3   812   801    12      3   591   565    23     12   297   416    32      7  2270  2258     8      5  1376  1359    10     17   321   135    48
  4   303   292    26      4   295   265    42     13  1676  1680    10      8   885   895    11      6   370   336    25         1,   11,   L  
  5  1492  1490     9      6   313   304    42     14  1456  1481    11      9   577   554    16      7  1635  1662    10      0   466   494    30
  6   779   781    13     10   706   692    22     15  1344  1341    12     10   796   796    13      8   275   305    36      1   537   533    28
  7   979   984    12     12   520   560    30     16   520   648    26     11   779   793    14      9   826   850    15      2   324   356    48
  9  1447  1469    11     14   829   842    22     23   523   628    36     12  1538  1504    11     10  1206  1203    12      3  1500  1538    15
 10  1151  1139    12     15   602   552    28         1,    2,   L       13   929   949    14     11   271   142    38      4   808   788    21
 12  1068  1095    14     16   681   649    27      0  1747  1737     5     15   635   682    21     12   290   280    39      5   918   882    19
 13   461   406    26         0,   12,   L        1   184   195    30     16   725   700    19     13   309   354    41      7  1301  1262    15
 14  1088  1083    15      2   713   712    21      2  4653  4715     5     17   641   663    23     14   944   925    17      8   571   576    26
 16   881   920    18      4   341   327    39      3  1038  1023     8     19  1059  1086    18     15   339   503    43      9   518   507    28
 17   324   347    42      6  1627  1559    14      4  1099  1073     8     23   475   527    38     17  1058  1063    17     15   319   357    51
 18   463   458    31      7   310   196    44      5  1267  1270     8         1,    5,   L       19   416   428    37     16   359   344    47
 21   598   642    29      8   601   611    26      6  3820  3802     6      0  1267  1273     8     21   802   801    24         1,   12,   L  
       0,    8,   L       10  1027   995    18      7   224   221    29      1  2571  2563     8         1,    8,   L        0   453   411    30
  0   675   681    16         0,   13,   L        8   361   356    19      2  1058  1070    10      0   916   883    12      1   318   401    49
  1   758   751    13      1   568   624    27      9  1779  1771     8      3   460   456    17      1   673   695    17      3   489   500    33
  2   988  1035    12      2   987   954    18     10  1068  1085    10      4  2115  2104     8      2   666   673    18      7   638   688    26
  3  1021  1019    11      3   699   678    23     11   464   463    18      6   595   591    14      3   462   475    23      8   300   292    47
  5   516   516    19      8   358   396    45     12   267   318    32      7  1113  1110    11      5   419   441    24      9   692   712    24
  6  1846  1838    10      9   450   368    35     13  1057  1054    12      8  1270  1291    10      6   848   882    15     11   887   898    21
  7  1333  1348    11     11   494   495    34     14  1344  1361    12      9  1552  1536    10      7  1302  1320    12     13   856   839    23
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COLUMNS ARE 10FO,10FC,  10SIG                                                       COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG       L   KFO    FC  SIG       L   KFO    FC  SIG       L   KFO    FC  SIG       L   KFO    FC  SIG       L   KFO    FC  SIG
      1, 13, L             9  1058  1082   10       1   448   442   18           2,  7, L             9   741   718   23       3   698   677   13
  0   402   431   37      10   422   427   21       2  2738  2731    9      18   503   471   32      10  1112  1127   18       4  2011  2013    8
  1   432   413   39      11  2668  2720    9       3   350   351   24      19   458   513   36      12   350   346   44       5   608   593   15
  2   535   465   33      13   887   883   14       4  2386  2371    9      21   341   267   49      14   403   451   43       6   486   482   17
  3   738   684   25      14   716   728   17       5  1601  1620   10           2,  8, L                 2, 12, L         7   942   985   12
  4  1044  1035   19      15   454   447   26       6  1666  1620   10       0  2268  2282   10       0  1222  1207   16       8  1434  1448   10
  5   399   368   40      19   714   715   23       7   343   313   26       1  1983  1994   11       2  1151  1154   19       9   387   402   24
  7   662   657   26      21   764   767   23       8   388   390   24       2  1734  1764   12       4  1073  1080   20      10  1284  1262   11
  8   744   720   24      23   531   583   34       9  2244  2235   10       3   516   536   24       6   800   818   24      12  2286  2301   10
      1, 14, L                 2,  3, L            11   902   884   14       4  1257  1221   13      10   311   255   52      13   537   544   23
  0   828   839   22       0   556   539   12      12  1173  1193   13       5  1146  1108   14      14   906   886   23      14  1714  1675   12
  5   592   567   31       1   363   358   19      13   623   646   21       6  1209  1258   13           2, 13, L            16   906   918   17
  8   769   745   25       2   723   707   12      15   285   372   47       7   581   603   22       0   384   409   40      18   849   874   20
      2,  0, L             3  2344  2304    8      16  1595  1551   13       8   359   303   32       2   353   319   46      19   355   406   42
  0  1358  1330    6       4  1246  1236    9      17   446   472   34      11   858   880   18       5   690   660   27           3,  3, L
  2   321   328   20       5   941   933   11      20   712   706   25      12   826   815   19       6   642   632   29       0   750   736   11
  4  1286  1254    8       6   491   487   16      21   625   654   28      13   338   421   43       9   704   708   27       1   364   355   19
  6  2567  2534    7       7  1581  1550    9           2,  6, L            14  1051  1050   17      10   634   557   29       2   318   294   23
  8  2815  2821    8       8   809   845   12       0   994  1002   11      15   445   501   34      11   410   367   45       3   481   492   17
 10  2387  2379    8       9   593   577   16       1  1857  1855    9      16   355   350   42           2, 14, L             4  2926  2940    8
 12  2462  2450    9      10  1180  1205   11       2  1668  1683   10      17   667   649   26       1  1054  1026   21       5  1065  1073   11
 14   321   342   31      11   776   778   14       3  1488  1461   11      18   863   891   22       5   630   637   33       6  1688  1663    9
 20  1136  1158   17      12   347   348   28       4  1368  1389   11           2,  9, L                 3,  0, L             7   255   145   33
 22   310    53   45      13  1140  1141   13       5  1524  1554   11       1   389   383   29       1  3720  3734    6       8  2782  2776    9
 24  1008  1007   21      14   708   723   18       6   270   258   35       2   458   481   28       3   909   905   11       9  1341  1329   11
      2,  1, L            15  1491  1567   13       7  1628  1648   10       4   562   534   24       5  1247  1240    9      10   926   936   14
  0  2141  2067    5      16   848   854   18       8  1253  1285   12       5  1678  1644   13       9  2108  2081    9      11  1152  1182   13
  1   761   733    9      17   953   963   17       9   378   386   27       7   394   386   33      11   914   891   13      12   734   744   17
  2  3000  2958    6      19   768   776   21      10   760   732   16       8   414   436   32      13  2657  2710   10      15   741   750   19
  3  3046  3023    6      20   376   439   42      11   843   834   16       9  1950  1956   12      17  1112  1159   16      16   400   472   35
  4  2097  2070    7      21   321   265   49      12   695   681   19      10   407   389   35           3,  1, L            17   565   565   27
  6  1084  1104    9      22   503   527   34      13  1256  1208   14      11   582   606   26       1  1493  1453    8      18  1171  1147   17
  7   890   909   10           2,  4, L            14  1161  1115   15      13   868   913   20       2   772   763   11      20   401   438   40
  8   440   415   17       0   765   776   10      17   583   588   26      14   314   319   47       3  1557  1547    8      22   844   843   24
  9   383   396   20       1   350   355   20      18   700   671   24           2, 10, L             4  1522  1543    9      23   349   389   49
 10   220   192   36       4  1384  1340    9      19   646   667   27       0   713   703   19       5  3271  3216    8           3,  4, L
 11   950   983   12       5  1390  1374    9      20   319   403   53       1  2334  2353   12       6  1266  1265   10       0  1597  1624    8
 12  1385  1391   11       6  1698  1695    9           2,  7, L             2   653   677   24       7   719   709   13       1  2474  2450    8
 14   250   272   41       7  1094  1105   11       0   987   975   12       4   637   664   24       8  1729  1732    9       2   720   750   13
 15  1281  1318   13       8  2737  2704    9       1   400   427   24       5  1762  1782   14       9  2307  2299    9       3   815   801   12
 16  1738  1740   12       9   600   598   16       2   311   339   32       6   340   420   43      10   905   956   13       4   948   947   12
 17   722   763   21      10   474   486   21       3  2737  2723   10       7   387   370   37      11   637   644   17       5  1014  1031   12
 19   604   594   26      11  1070  1105   13       4   784   755   16      10   296   321   48      14   314   329   37       6  1555  1580   10
 20   839   815   20      12  1724  1742   11       5   250   270   39      13  1015   994   19      16   501   477   26       7  1077  1062   12
 21   421   473   39      13   518   534   23       6  1699  1718   11      14   520   502   31      17   860   852   19       8   567   581   19
      2,  2, L            14   316   359   37       7   780   764   16      15   436   472   38      18   519   490   28       9  1408  1411   11
  0  1021  1015    8      15  1029   999   15       8  1458  1473   12      17   806   828   25      20   349   418   46      10  1527  1542   11
  1   496   481   14      16   312   105   40      10  1989  2039   11           2, 11, L            21   991   960   20      11   418   416   26
  2  2888  2831    7      17   561   567   26      11   546   578   24       0   579   567   24      22   652   666   29      12   391   370   30
  3   348   284   22      18   468   483   32      12   457   475   28       2   322    45   46      23   324   320   53      13  1975  1975   12
  4  1027  1041   10      20   787   765   22      13   420   398   32       4   757   792   25           3,  2, L            14   285   482   46
  5  1053  1035   10      21   444   496   38      14   756   768   20       5   681   667   25       0  4198  4176    6      16   767   775   21
  7  3440  3457    7           2,  5, L            15   839   834   20       6  1129  1085   18       1   412   414   17      17  1062  1036   17
  8   664   656   13       0  1946  1916    8      17   476   491   33       8   651   640   25       2   831   820   11      19   385   420   41
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COLUMNS ARE 10FO,10FC,  10SIG                                                  COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG      L   KFO    FC   SIG
        3,   4,  L         3  1390  1378    13      9   335    35    49            4,   3,  L        19   320    39    47            4,   9,  L
 20   529   576    31      4   993   987    16     10   507   507    38      2   840   813    12     20   320   288    51      0   389   393    31
        3,   5,  L         5   696   689    20            3,  14,  L         3   433   415    19     21   616   618    31      1  1070  1077    15
  0   640   635    14      6  1207  1189    14      2   617   635    32      4  1481  1460    10            4,   6,  L         3  1645  1675    13
  1  1410  1405    10      7  1763  1770    13      3   336   197    53      5   438   435    22      0   674   677    15      4   451   428    31
  2  1679  1716    10      8   509   511    27      6   793   754    28      6   735   729    15      1  1595  1624    11      5  1085  1094    17
  3   831   809    14     10   932   951    18            4,   0,  L         7  2059  2040    10      2  1173  1197    12      7   958   948    19
  4  1146  1151    12     11   650   669    24      0  1050  1031     9      8   678   686    17      3   359   371    29      9   502   446    32
  5  2547  2560    10     13   512   517    30      2  4378  4387     7      9  1162  1173    13      4   698   661    17     10   562   531    29
  7   904   925    14     15   675   694    26      4   983   976    11     10  1202  1242    13      5   526   551    23     13   551   540    30
  9  1829  1799    11     16   480   492    33      6  3973  3990     9     11  2137  2098    11      6   603   574    20     15   358   287    47
 10  1650  1654    12     17   316   171    47     10   513   505    21     12   806   798    18      7  1464  1445    13     17   921   917    23
 12   811   834    17     19   812   835    25     14  1563  1575    13     13   869   867    17      8  1581  1609    13            4,  10,  L
 14   673   676    22            3,   9,  L        16   445   474    32     15   842   833    19      9   668   673    21      0   562   535    25
 16   858   861    20      0   516   517    22     18  1586  1581    15     16   537   587    29     10   350   328    37      1   667   686    23
 17   995  1007    19      2  1845  1858    12     20   895   869    21     18   302   276    47     11   763   764    20      2   300   286    45
 19   358   400    46      3   497   509    27     22   551   486    32     22   485   437    37     12  1220  1222    16      7   717   650    24
 21   744   730    26      4   280   258    46            4,   1,  L               4,   4,  L        13   908   877    19      8   511   492    33
        3,   6,  L         5   643   627    23      0  1135  1140     9      0  1048  1056    10     15   506   484    31      9  1086  1068    19
  0  1709  1671     9      8   678   679    24      1  1270  1280     9      1   631   647    14     16   609   618    28     11   722   699    26
  1   651   638    15      9   351   413    44      2   526   553    15      2  2720  2728     9     18   495   482    33     12   527   501    34
  2  1583  1615    10     10   847   876    21      3   677   691    13      3  1106  1125    12     19   613   595    29     13   948   947    22
  3  1820  1770    11     11   489   421    30      5   739   726    13      5  1448  1443    11            4,   7,  L               4,  11,  L
  5   357   392    30     12  1265  1254    16      6  2078  2074     9      6  2245  2264    10      0  1715  1715    10      0  1145  1135    16
  6  1159  1167    13     14  1098  1085    18      7  1127  1133    12      7   515   525    22      1   541   565    20      1   567   506    27
  7  1712  1689    11     16   811   808    23      9  1550  1528    11      8  1088  1070    13      2   694   698    18      3   547   556    31
  8  1038  1002    14            3,  10,  L        10  2477  2454    10      9  1450  1450    12      4  2377  2390    11      4  1481  1468    16
  9   991  1015    15      2  1194  1191    16     11  1487  1526    12     11  1015  1018    15      6   292   310    42      5   617   582    29
 11  1016   990    16      3   862   867    20     13   610   576    21     12   472   461    28      7   539   497    24      8   753   723    26
 12   971   925    16      4   968   939    19     14  1397  1353    13     13   839   827    18      8  1061  1057    16     10   349    53    45
 13   710   711    21      6  1801  1839    14     15   682   639    21     14  1239  1215    15      9   601   616    25     12   319   275    53
 14   981   995    18      7   709   708    24     20   346   324    47     15   614   593    25     11   810   797    20            4,  12,  L
 17   630   624    26      8   987   992    19     22   515   455    35     16   616   639    26     12   324   403    46      0   900   897    20
 18   659   665    26     10   771   803    23     23   514   526    37     18   928   939    20     13   367   428    42      1   321   294    46
 19   546   532    31            3,  11,  L               4,   2,  L        20   731   696    25     14   394   353    39      8  1025  1018    23
 21   315   206    52      1   627   619    25      0   769   765    11     21   343   393    53     15   677   665    25     12   963   944    24
        3,   7,  L         2   829   838    21      1  2953  2968     8            4,   5,  L        16  1142  1153    18            4,  13,  L
  0   464   439    20      3   979   969    20      2  1023  1055    11      0   747   743    14     18   519   520    34      0   659   595    26
  1  1833  1826    10     11   917   911    22      3  2948  2932     8      1   959   949    12     20   629   593    30      3   740   733    27
  2   724   696    16     12   355   296    47      5  2904  2902     9      2   939   914    12            4,   8,  L         4   812   776    26
  3  1696  1657    11     14   471   454    38      6   491   468    20      3  1888  1925    10      0  1569  1557    11      5   635   592    32
  4  1499  1493    12     15   893   857    23      7  2186  2160    10      4   587   580    19      2   392   438    30      8   385   378    51
  6   869   861    16            3,  12,  L         8   246   348    40      5  1954  1935    11      3   330   382    37            4,  14,  L
  7   292   355    41      1   392   405    38      9   534   539    21      6   925   935    15      5   581   541    24      2   361   397    52
  8  1025   974    15      2   398   369    39     10   388   398    27      7  1245  1252    13      6   391   410    34            5,   0,  L
 10   657   618    21      3  1202  1188    19     11   285   200    38      8   303   301    38      7   325   292    41      3  1891  1901     9
 11  1866  1888    12      5   642   621    29     12   620   653    21      9   819   816    17      8  1151  1177    16      5  1980  1965    10
 12   304   245    44      7  1343  1306    18     13   780   768    18     10  1811  1755    12      9   937   917    19      7  3222  3293    10
 15  1429  1442    16     11   582   573    32     15  1507  1505    14     11   336   326    38     10   372   338    39      9  1352  1329    12
 18   513   558    32            3,  13,  L        17   555   582    28     12   422   452    32     11   448   478    35     11  1766  1809    12
        3,   8,  L         1   552   576    31     19  1453  1401    16     13   621   631    25     12  1041  1061    19     13   671   656    22
  0   988   999    13      2   691   708    27            4,   3,  L        14  1204  1218    16     13   576   593    29     17   544   535    29
  1  1099  1116    14      3   370   436    49      0   717   718    12     15   718   717    23     14   423   405    38     19   715   726    26
  2   407   372    27      4   366   343    49      1  1849  1847     8     17  1043  1054    19     15   538   540    31     21   913   904    23
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COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO    FC   SIG      L    KFO    FC   SIG      L    KFO    FC   SIG
      5,  1,  L             3   1432  1471    11     13    547   520    31
  0   1539  1547     9      4   1177  1197    12     16    572   586    32
  2   1444  1432    10      5    746   744    16           5,  8,  L
  3   3001  3022     9      7   2133  2134    11      0    789   809    18
  4   2316  2275     9      8    912   899    16      1   1796  1774    12
  5    626   611    17      9    970   951    16      2    801   816    19
  7    324   335    31     10    682   718    22      3    440   402    30
  9    488   464    23     11    870   871    18      4    965   970    17
 10    580   545    21     12    498   487    28      5    656   668    23
 11    978   954    16     13    645   656    24      7    446   468    33
 12   1195  1248    15     14    820   815    21      8    490   506    31
 13   1144  1163    16     18    589   584    30      9    588   572    27
 15   1019  1025    18     19    623   617    30     10    647   633    26
 16   1098  1072    18     21    691   655    29     13   1128  1141    19
 17    759   745    23           5,  5,  L            14    684   683    27
 18    356   436    46      3   1445  1481    12     15    660   707    29
 19    524   530    33      4    921   895    15     17    484   467    38
 20    351   288    48      5    497   507    24           5,  9,  L
      5,  2,  L             6   1778  1748    12      0    288   328    44
  0    446   449    19      7    547   541    25      2    538   591    28
  1    861   864    12      8    875   892    18      3    532   519    28
  2    708   674    14      9    459   466    30      4    613   612    26
  3    691   720    15     10   1365  1363    15      5    544   517    29
  4   2084  2046    10     11    708   708    22      6   1396  1361    16
  5   1000   995    13     13    597   592    27      8    638   612    27
  6   1433  1487    12     14    326   378    50      9    357   300    45
  7    883   899    15     15   1051  1012    18     10   1296  1268    18
  8   2784  2854    10     17    706   675    26     11    403   388    43
 10    607   620    21     18    558   491    32     12    340   368    48
 11    795   801    19     19    392   461    46     16    364    58    46
 12   1326  1398    15           5,  6,  L                 5, 10,  L
 15    489   497    32      0   1227  1262    12      0    995  1006    18
 17    556   578    30      1   1795  1765    11      1    788   809    21
 20   1132  1112    20      2   1485  1484    12      2   1255  1269    17
      5,  3,  L             5    925   926    16      4    664   595    25
  0   2521  2498     9      6   1008   981    16      5    463   510    36
  1    262   262    31      7    690   658    21      6    564   554    31
  2   2208  2200     9      8   1013  1010    16     10    584   619    32
  3   1030  1065    12     11    709   671    24     11    458   501    40
  4   1449  1436    11     12    718   733    24     12    403   360    44
  5   1505  1469    11     13    436   458    37     14   1021   978    22
  6   1518  1496    11     14    837   833    22           5, 11,  L
  7    278   252    38     15    640   635    28      4    343   250    49
  9   1043  1040    15     17    366   412    49      5   1091  1067    20
 11   1013  1012    16     18    404   373    43      6    503   527    37
 12   1321  1299    15           5,  7,  L             7    547   463    33
 15    430   400    36      0    957   981    15      9   1102  1067    21
 16   1670  1627    14      2    543   573    23     10    491   422    39
 17    822   817    22      4    660   664    21     11    455   431    43
 20    604   618    32      5   1539  1489    13           5, 12,  L
      5,  4,  L             7    347   350    40      1   1263  1283    18
  0   1015   986    12      9   1441  1438    16      5    661   687    30
  1    783   781    14     11    518   531    32           5, 13,  L
  2   1089  1089    12     12    877   886    21      4    374   343    50
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COLUMNS ARE 10FO,10FC,  10SIG

  L    KFO    FC   SIG      L    KFO    FC   SIG      L    KFO    FC   SIG
      5, 13,  L            17    858   823    23      2   1065  1072    16
  5    491   473    40     18    331   326    53      3    276   310    45
  6    535   489    39     19    505   471    37      5    927   942    19
      6,  0,  L                  6,  4,  L             6    324   320    43
  0   2973  3034     9      0   1782  1806    11      8    607   623    28
  2    757   767    15      1    387   410    27      9    719   718    24
  4    757   778    16      2   1185  1192    13     10    815   805    23
  8    914   908    17      3   1460  1506    12     12   1050  1042    20
 10   1720  1700    13      5    342   358    35     14    542   592    34
 12   1178  1176    16      7   1096  1076    15     16    583   586    34
 14   1447  1447    16      8   1176  1192    15     17    413   459    46
 16    743   745    24      9    747   778    21           6,  8,  L
 18    524   527    34     10    798   784    20      2   1083  1112    17
      6,  1,  L            11    296   309    48      3    976   976    18
  0    962   974    12     12    990   961    19      4    472   476    31
  1   1020  1020    12     13    692   666    25      6    992   976    19
  2    952   977    13     14   1170  1172    18      7    792   780    22
  3    792   793    15     17    514   503    34      8    953   977    20
  4   2000  2032    11     19    338   456    56      9    400   455    42
  5   1919  1946    11           6,  5,  L            12    350   373    49
  6   1485  1530    12      0    750   744    16     13    525   524    34
  8   1192  1165    14      1    444   468    26     14    566   516    32
  9   1018   969    16      2    807   795    16     16    356   407    53
 10    689   684    21      3    795   810    17           6,  9,  L
 13    365   329    40      4   1454  1463    13      0    417   438    34
 17    801   782    24      6    809   823    19      1    575   560    25
 18   1105  1106    20      7    618   635    24      3    666   664    24
 19    388   357    45      8   1161  1132    16      4    503   515    31
      6,  2,  L             9    326   278    41      6    340   341    44
  0    716   722    15     10    473   468    33      7    749   695    23
  1   2406  2455    10     11   1263  1252    17     10    473   383    37
  2    746   745    15     12    339   259    45     11   1300  1308    19
  6    491   504    23     14    525   515    32           6, 10,  L
  9   1799  1820    13     15    780   794    25      1    375   411    41
 10    448   471    31     18    693   635    28      3   1167  1203    18
 13   1764  1725    14           6,  6,  L             5    313   286    52
 15    355   420    46      0    928   934    15      6    533   486    33
 17    688   686    26      1    994  1009    15      7   1286  1274    19
 19    359   150    47      2    777   807    18     11    421   367    44
      6,  3,  L             3    789   840    19           6, 11,  L
  0    915   892    13      4    898   876    17      2    687   738    26
  1   1868  1878    10      5    329   298    42      8    529   466    35
  2    296   310    34      6   1193  1188    16     10    696   662    31
  3    459   461    23      7   1095  1067    17           6, 12,  L
  4   1248  1266    13      9    689   673    25      2    836   828    24
  5   2526  2561    11     10    837   857    22      4    568   566    34
  6    673   667    20     12    476   515    36      6    930   913    25
  7    400   411    31     13    943   916    21           7,  0,  L
  8    356   338    36     14    426   406    41      1   2028  2065    11
  9   1352  1367    14     15    586   574    30      3   1883  1926    12
 10    738   719    21     16    624   621    30      5   1154  1176    14
 12    599   620    27           6,  7,  L             7   1146  1162    15
 15    447   422    36      0    539   513    23     11    592   566    27
 16    645   669    28      1    638   667    21     13    554   525    30
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COLUMNS ARE 10FO,10FC,  10SIG                                                   COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG      L   KFO    FC  SIG
       7,    0,  L        8   850   822   19           7,    9,  L             8,    3,  L             8,    8,  L        7   331   346   45
 15  1569  1553   17     12   830   818   22      0  1139  1168   17      4   691   703   23      0   690   646   23      8   755   724   24
 19  1030   996   22     13   383   393   44      1   311   144   43      6   523   548   30      1   782   800   22      9   343   366   52
       7,    1,  L       15   884   893   23      4  1326  1377   17      9   511   514   31      2   958   956   19     11   633   623   29
  1   720   738   17     18   364   319   51      5   458   551   38     10   565   539   30      4   345    67   44     13   356   313   51
  3   307   295   35          7,    5,  L         8   435   482   42     11   752   763   24      5   502   485   33     14   507   498   39
  5   616   610   21      0  1346  1379   14      9   379   419   48     12   530   527   33      7   410   420   42          9,    4,  L
  6   941   954   17      1   539   538   24     12   523   513   38     13   813   798   24      8   340   393   51      0   762   782   21
  7  1373  1402   14      3   340   371   37           7,   10,  L       15   699   697   29     10   527   518   37      1  1046  1067   18
  9   600   607   25      4  1266  1274   15      0   591   581   27     16   452   406   42     11   551   531   35      2   316   339   47
 10  1202  1168   16      5   304   320   46      5   366   380   47           8,    4,  L             8,    9,  L        3   439   438   34
 11  1357  1380   16      6   312   305   47      8  1138  1164   22      1   917   921   18      0   398   439   38      5   329   192   43
 12   564   510   28      7  1141  1164   17           7,   11,  L        2   801   807   19      1   392   325   39      6   811   813   22
 13   356   413   46      9   507   542   32      0   541   551   32      4  1212  1185   16      5  1115  1098   20      7   349   287   44
 14   490   489   34     10   344   226   44      1   637   606   28      5   557   590   28      7   667   646   29      9  1069  1072   20
 15   442   415   37     11  1211  1202   18      3   790   821   26      6   675   646   24      9   875   864   25     10   588   569   31
 18   364   277   48     12   622   594   28      4   478   442   38      7   747   710   23           8,   10,  L       13   750   734   27
 19   377   293   47     14   324   274   52      5   681   694   30      8  1383  1394   16      1  1031  1025   21          9,    5,  L
       7,    2,  L       16   676   679   29           8,    0,  L       11   578   588   31      2   367   353   46      0   637   631   24
  0   447   488   25          7,    6,  L         4  1453  1482   14     12   583   558   31      5   385   387   48      1  1185  1171   17
  2  2730  2748   11      0   580   589   23      6   358   338   39     13   509   499   36           9,    0,  L        5  1116  1120   19
  5   524   521   24      1   410   417   32      8  1824  1824   14     14   344   394   54      1   790   815   20      6   579   551   29
  6  1668  1676   13      2   840   875   19     12   837   784   22     16   414   358   45      9  1295  1310   18      8   427   365   39
  8   322   350   41      4   297   331   48     16   588   578   33           8,    5,  L       11   346   389   49      9   498   491   36
 13   326   190   48      5   852   847   20           8,    1,  L        0  1072  1103   16     13  1225  1249   20     10   667   635   28
 14  1166  1196   18      6   519   501   30      0  1532  1513   12      1   350   346   40     15   361   337   52     12   649   641   31
 16   779   721   24      8   779   755   23      1   525   519   24      2   838   856   20           9,    1,  L             9,    6,  L
 18   930   882   23      9   954   901   20      2   739   747   19      3   312   115   42      0   735   685   20      0   322   375   46
       7,    3,  L       11   471   479   37      3   850   878   18      4   701   676   23      1   868   875   19      2   748   795   24
  0   536   553   21     12   467   422   38      4   849   883   19      5   990   986   19      2   495   403   28      3   910   876   21
  1   576   579   21     13   622   552   29      5   408   411   33      6   328   267   45      4   863   860   20      4   469   478   35
  3   456   473   27     14   642   626   30      8   341   419   45      7   474   438   34      5  1398  1404   16      7   776   787   25
  4   386   403   31          7,    7,  L         9   292   224   45      9   724   738   25      6   722   763   24      8   612   603   31
  5   714   721   20      1   864   869   19     11   715   711   25     11   340   492   53      9   627   604   28      9   423   433   43
  6  1449  1485   14      3  1078  1115   18     12  1247  1214   18     12   879   871   24     13   344   300   51     10   503   486   37
  7   297   283   45      5  1069  1043   18     13   731   693   25           8,    6,  L             9,    2,  L             9,    7,  L
  8   475   484   29      6   768   764   23     15   558   530   34      0   657   628   23      0  1127  1113   16      0   459   446   34
  9   566   536   27      7   346   366   45     16  1017   966   22      1   789   796   21      1   400   420   35      4   539   524   32
 10  1530  1513   15     10   975   951   21     17   554   517   35      2   571   536   27      2   381   428   38      6   439   469   40
 11   735   724   24     12   428   400   42           8,    2,  L        3   891   896   19      5   294    76   48      7   737   704   27
 12   330   405   49     13   804   771   26      2   369   340   34      4   793   783   22      6   412   383   37          9,    8,  L
 13   352   257   44     14   366   293   50      3  1354  1355   14      7   731   751   25      7   338   324   45      0   314   385   50
 14   730   737   25          7,    8,  L         4   524   506   27      8   584   582   29      8   832   855   22      1   338   329   45
 15   409   508   43      0   391   335   34      5   718   740   21      9   760   750   25      9   359   351   47      2   507   472   33
 17   582   540   33      1   426   486   34      7  1684  1653   14     10   541   524   34     10  1055  1058   20      3   844   895   23
       7,    4,  L        2   591   619   26      8   467   434   33     13   548   540   35     12   641   642   30      4   426   478   42
  0   648   657   20      3   348   372   42      9   919   934   20     14   752   720   28     14   857   862   25      6   568   520   32
  1  1595  1595   12      6   609   640   28     11   707   708   25           8,    7,  L             9,    3,  L        7   714   735   28
  2   890   891   16      7   645   589   26     13   405   351   40      2   597   593   26      0   737   747   21          9,    9,  L
  3  1420  1443   13      8   713   670   25     14   436   480   41      3   712   717   24      1   602   627   26      0   351   259   44
  4   469   450   28      9  1006  1021   21     17   368   465   52      4   318   364   50      2  1038  1019   17      2   458   446   37
  5   773   759   19     10   326   305   53           8,    3,  L        6  1222  1210   18      4  1194  1210   17         10,    0,  L
  6   541   562   26     12   613   593   31      0   672   689   20     10   986   985   22      5   759   745   22      2  1786  1757   15
  7  1005  1024   18     13   773   800   27      3  1309  1348   15     13   393   444   52      6   821   830   22      6   815   801   23
```

```
   COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO    FC    SIG        L    KFO    FC    SIG         L    KFO    FC    SIG
      10,  0,  L              7    961   990     22         4    742   719     27
10    458   412     40        9    360   409     51              11,   0,  L
12    534   538     36       10    349   330     52         3    964   978     22
      10,  1,  L             11    613   645     34         5    689   727     27
 5    460   511     36             10,   4,  L              7    821   799     25
 6    936   938     22        2    966   991     19              11,   1,  L
 7    756   767     25        4    347   422     48         0    582   569     30
 8    519   472     34        5    592   557     31         3    576   621     32
10    885   889     24        8    369   366     48              11,   2,  L
11    739   735     28        9    566   532     33         0    322   351     51
      10,  2,  L                   10,   5,  L              1    331   323     48
 1    988  1016     19        1    720   712     24         4   1086  1046     20
 3   1055  1120     19        3    466   469     35         6    350   192     49
 5    475   462     34        5    551   546     33              11,   3,  L
 6    346   286     47        6    668   701     29         0    886   934     22
 7    430   455     40             10,   6,  L              1    364   343     45
 8    503   437     35        1    374   415     45         2    421   460     42
11    519   527     37        2    533   503     32         5    498   464     36
      10,  3,  L              3    630   629     29              11,   4,  L
 0    783   766     22        6    347   347     52         0    357   361     46
 1    308   298     49        7    389   402     48         3    713   710     28
 3    578   601     29             10,   7,  L              4    358   426     51
 4    522   510     32        0    922   896     21
```

OBSERVED AND CALCULATED
STRUCTURE FACTOR LISTINGS FOR
$CpRe(CO)_2(N_2Ph_2)$

```
COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO     FC  SIG    L    KFO     FC  SIG    L    KFO     FC  SIG
      -8,     0,  L      3    216    225   13    3    389    379    8
 1    359    358   10    4    364    365   10    4    250    250   11
 3    466    457    9    5    443    427    9    5    555    540    7
 5    311    323   11    7    414    418   10    7    416    417    9
 6    313    307   11         -7,     4,  L          -6,     4,  L  
      -8,     1,  L      1    560    548    8    1    636    647    7
 1    149    144   18    3    470    466    8    2    239    238   11
 2    482    479    8    4    309    320   11    3    427    430    8
 4    434    440    9    5    225    200   14    4    548    550    8
 5    253    286   13    6    440    444   10    6    529    520    8
 6    206    218   15         -7,     5,  L      8    363    342   11
      -8,     2,  L      2    437    439    9         -6,     5,  L  
 1    422    412    9    3    240    251   13    2    554    538    8
 3    460    462    9    4    278    288   12    3    381    374    9
 4    225    206   13    5    361    369   11    4    180    181   16
 5    311    312   11         -7,     6,  L      5    499    497    9
 6    382    363   10    1    408    397   10    7    418    434   10
      -8,     3,  L      3    389    371   10         -6,     6,  L  
 2    432    433    9    4    295    292   12    1    545    545    8
 3    132    124   22         -7,     7,  L      3    323    321   11
 4    393    394   10    2    397    387   10    4    457    442    9
 5    291    282   12    3    242    201   14    6    445    449   10
 6    192    192   17    4    290    277   13    7    144    126   22
      -8,     4,  L          -6,     0,  L          -6,     7,  L  
 1    340    342   11    1    660    665    6    2    428    423    9
 3    403    407   10    2    393    387    7    3    337    322   11
 4    207    191   15    3    527    522    7    5    394    391   11
 5    285    271   12    4    540    549    7         -6,     8,  L  
      -8,     5,  L      5    155    154   16    1    465    456   10
 2    410    408   10    6    488    502    8    2    268    259   13
 4    319    319   12    7    188    193   16    3    319    307   12
      -7,     0,  L      8    385    358   10    4    402    377   11
 1    564    563    8    9    263    282   14         -5,     0,  L  
 3    456    479    8         -6,     1,  L      1    619    625    6
 4    338    330    9    1    238    237   10    2    619    603    5
 5    248    254   12    2    559    577    7    3    497    498    6
 6    491    492    8    3    397    403    8    4    897    883    6
 8    380    395   10    4    326    316    9    6    726    734    7
      -7,     1,  L      5    644    652    7    7    151    122   17
 2    515    524    8    7    478    461    8    8    323    320   11
 3    272    293   11    8    299    294   12    9    360    351   11
 4    382    379    9    9    278    280   13         -5,     1,  L  
 5    402    420    9         -6,     2,  L      1    492    480    6
 7    502    501    9    1    642    648    7    2    507    525    6
      -7,     2,  L      2    253    258   10    3    834    828    6
 1    523    510    8    3    438    454    8    4    285    256    9
 2    137    144   19    4    525    523    7    5    825    810    6
 3    471    460    8    5    148    132   17    7    542    546    8
 4    336    347   10    6    557    556    8    8    322    311   11
 5    249    237   12    7    173    165   17         -5,     2,  L  
 6    460    458    9    8    360    364   11    1    720    748    6
 8    383    394   11         -6,     3,  L      2    553    540    6
      -7,     3,  L      1    149    147   16    3    467    454    6
 2    558    561    8    2    558    561    7    4    753    738    6
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO     FC  SIG    L    KFO     FC  SIG    L    KFO     FC  SIG
      -5,     2,  L      5    411    411   11    6    315    314   10
 6    578    592    7         -4,     0,  L      7    358    335   10
 7    255    251   12    1    633    629    5    8    430    440   10
 8    259    264   12    2    805    807    5         -4,     6,  L  
 9    373    377   10    3    381    368    6    1    561    547    7
      -5,     3,  L      4   1079   1087    5    2    590    593    7
 1    267    303    9    5    163    156   12    3    148    161   16
 2    620    616    6    6    661    642    6    4    664    673    7
 3    484    512    7    7    516    528    7    5    140    154   19
 4    197    191   12    8    373    373   10    6    420    436    9
 5    662    661    7    9    418    423    9    7    307    319   12
 7    380    377    9         -4,     1,  L      8    140    182   23
 8    346    341   11    1    655    682    5         -4,     7,  L  
 9    221    191   15    2    523    504    5    1    367    377    9
      -5,     4,  L      3   1066   1041    5    2    281    330   11
 1    663    677    6    5    827    800    6    3    645    631    7
 2    491    486    7    6    374    373    8    5    546    558    8
 3    301    296    9    7    573    553    7    6    262    269   13
 4    666    658    7    8    491    496    8    7    316    318   13
 6    545    561    8   10    374    376   12         -4,     8,  L  
 7    228    215   13         -4,     2,  L      1    339    358   10
 8    282    278   13    1    771    768    5    2    529    529    8
 9    358    362   12    2    831    814    5    4    499    523    9
      -5,     5,  L      3    215    263    9    5    148    144   21
 1    214    235   12    4    840    848    5    6    427    414   10
 2    520    502    7    5    151    147   14    7    338    337   12
 3    596    581    7    6    596    603    7         -4,     9,  L  
 5    654    641    7    7    407    422    8    1    395    384   10
 7    431    433    9    8    267    268   12    2    202    213   17
 8    294    306   13    9    411    406   10    3    421    450   10
      -5,     6,  L          -4,     3,  L      5    395    399   11
 1    565    577    8    1    501    508    6    6    155    214   25
 2    491    499    8    2    571    572    6         -4,    10,  L  
 3    330    319   10    3    913    912    5    1    293    297   14
 4    546    566    8    4    160    205   12    2    382    381   12
 6    489    476    9    5    721    720    6    4    420    423   11
 7    194    219   18    6    392    399    8         -3,     0,  L  
 8    214    228   17    7    355    345    9    1    609    578    4
      -5,     7,  L      8    461    470    9    2   1133   1123    4
 1    313    322   11         -4,     4,  L      4    741    757    5
 2    459    453    9    1    723    726    6    5    467    455    6
 3    437    442    9    2    806    806    6    6    505    500    7
 5    527    528    9    3    170    174   12    7    605    602    7
 7    364    341   11    4    758    783    6    9    524    523    8
      -5,     8,  L      5    201    181   11         -3,     1,  L  
 1    428    433   10    6    485    487    8    1    802    811    4
 2    443    448    9    7    410    425    9    2    317    332    6
 3    219    240   15    8    220    234   15    3   1128   1129    4
 4    467    484   10    9    456    473   11    4    358    353    6
 6    440    451   11         -4,     5,  L      5    701    708    5
      -5,     9,  L      1    481    490    7    6    666    671    6
 1    266    252   13    2    366    366    8    7    284    276   10
 2    323    324   12    3    824    822    6    8    685    681    8
 3    370    371   11    5    636    647    7   10    355    351   12
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COLUMNS ARE 10FO,10FC,  10SIG
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```
 L   KFO    FC   SIG
    -3,   2,  L
 1   722   779    4
 2  1091  1132    4
 4   834   851    5
 5   307   351    8
 6   544   525    7
 7   699   717    7
 8   218   204   14
 9   507   520    9
    -3,   3,  L
 1  1156  1122    4
 2   262   335    7
 3  1014   998    5
 4   299   282    7
 5   685   672    6
 6   579   587    7
 7   204   203   14
 8   615   617    8
    -3,   4,  L
 1   452   459    6
 2   906   906    5
 4   958   959    6
 5   408   416    8
 6   393   379    8
 7   585   600    8
 9   491   493   10
    -3,   5,  L
 1   810   815    6
 2   186   192   11
 3   932   950    6
 4   281   261    9
 5   621   609    7
 6   447   435    8
 7   293   258   11
 8   498   513    9
    -3,   6,  L
 1   393   401    8
 2   712   704    6
 3   133   105   17
 4   794   798    7
 5   291   296   11
 6   339   347   10
 7   458   467    9
    -3,   7,  L
 1   694   682    7
 2   252   270   11
 3   626   653    7
 4   153   162   18
 5   482   496    9
 6   401   396   10
 7   193   185   17
 8   418   390   11
    -3,   8,  L
 1   315   309   11
```

```
 L   KFO    FC   SIG
 2   496   508    8
 4   506   503    9
 5   261   293   13
 6   329   316   12
 7   436   434   11
    -3,   9,  L
 1   383   404   11
 3   487   474    9
 5   442   440   10
 6   333   336   13
    -3,  10,  L
 1   244   231   14
 2   480   478   10
 4   439   427   10
    -3,  11,  L
 1   368   365   12
 3   454   449   11
    -2,   0,  L
 1   248   254    6
 2  1013  1016    4
 4   811   804    5
 5   651   630    5
 6   379   373    7
 7   675   682    7
 9   455   442    9
10   266   272   14
    -2,   1,  L
 1  1464  1529    3
 2   324   340    6
 3   811   832    4
 4   686   688    5
 5   495   524    6
 6   821   816    6
 7   186   175   14
 8   571   573    8
 9   228   225   14
10   328   306   12
    -2,   2,  L
 1   235   279    7
 2  1378  1399    4
 3   410   431    5
 4   840   850    5
 5   792   795    5
 6   529   530    7
 7   728   738    7
 9   473   478   10
10   226   226   17
    -2,   3,  L
 1  1100  1082    4
 3   997   975    5
 4   483   467    6
 5   546   540    6
 6   655   645    7
 8   521   526    9
```

```
 L   KFO    FC   SIG
    -2,   4,  L
 1   213   203    9
 2  1173  1142    5
 4   746   753    5
 5   572   584    7
 6   333   327    9
 7   579   571    8
 9   425   411   11
    -2,   5,  L
 1   925   926    5
 3  1056  1036    5
 4   498   504    7
 5   531   531    7
 6   564   565    8
 8   490   499   10
    -2,   6,  L
 2   925   929    6
 3   190   168   12
 4   760   754    6
 5   595   598    7
 6   284   300   12
 7   495   498    9
    -2,   7,  L
 1   706   715    7
 3   704   689    7
 4   386   381    9
 5   438   440    9
 6   522   506    9
 8   420   420   11
    -2,   8,  L
 2   645   644    8
 3   168   146   17
 4   366   378   10
 5   485   481    9
 6   248   254   15
 7   477   468   11
    -2,   9,  L
 1   634   650    8
 3   489   488    9
 4   263   262   13
 5   312   305   13
 6   456   452   10
    -2,  10,  L
 2   501   510   10
 4   412   404   11
 5   321   319   13
    -2,  11,  L
 1   461   477   11
 3   433   429   11
 4   196   216   20
    -1,   0,  L
 1   124   114    8
 2  1588  1585    3
 4   960   958    4
```

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COLUMNS ARE 10FO,10FC,  10SIG
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```
 L   KFO    FC   SIG
    -1,   0,  L
 5  1010   984    5
 6   277   259    8
 7   639   666    7
 8   240   238   13
 9   325   331   11
10   333   348   13
    -1,   1,  L
 1  1413  1384    3
 3   571   591    4
 4   705   732    5
 5   393   393    6
 6   794   786    6
 8   533   533    8
 9   329   315   12
10   267   268   15
    -1,   2,  L
 2  1341  1370    4
 3   752   757    4
 4   627   618    5
 5   942   941    5
 6   226   236   11
 7   757   768    7
 8   149   148   22
 9   341   317   12
    -1,   3,  L
 1  1181  1171    4
 2   305   298    7
 3  1049  1038    5
 4   626   621    6
 5   451   468    7
 6   801   788    6
 8   445   429   10
 9   237   238   15
    -1,   4,  L
 2  1279  1267    5
 3   589   561    6
 4   805   791    6
 5   656   686    6
 6   262   253   11
 7   549   555    8
 8   144   139   22
 9   323   317   13
    -1,   5,  L
 1   816   844    5
 2   289   306    8
 3  1036  1014    6
 4   590   614    7
 5   355   340    9
 6   651   655    8
 8   393   400   11
 9   239   223   17
    -1,   6,  L
 2   861   851    6
```

```
 L   KFO    FC   SIG
 3   423   418    7
 4   521   525    7
 5   673   665    7
 7   511   502    9
    -1,   7,  L
 1   825   837    6
 3   712   688    7
 4   523   512    8
 5   249   227   13
 6   609   614    8
 8   441   424   11
    -1,   8,  L
 2   640   668    8
 3   391   392   10
 4   363   384   10
 5   536   517    9
 7   482   477   11
    -1,   9,  L
 1   658   688    9
 2   193   199   17
 3   483   488    9
 4   368   370   11
 5   198   222   19
 6   420   426   11
    -1,  10,  L
 2   561   572    9
 3   176   224   21
 4   311   326   13
 5   400   417   12
    -1,  11,  L
 1   489   478   10
 3   407   394   12
 4   315   296   14
     0,   0,  L
 1   256   246    4
 2  1443  1403    3
 3   746   742    4
 4   763   750    5
 5   778   791    6
 7   576   595    8
 8   361   340   11
 9   308   309   13
     0,   1,  L
 1  1773  1687    3
 2   225   331    7
 3   958   958    4
 4   844   824    5
 5   297   283    8
 6   789   812    6
 7   277   271   11
 8   464   462    9
 9   368   374   12
     0,   2,  L
 0  1694  1822    4
```

```
 L   KFO    FC   SIG
 1   309   323    6
 2  1011   995    4
 3   982   957    5
 4   439   427    6
 5   897   873    6
 7   664   652    8
 8   364   354   11
 9   348   357   12
     0,   3,  L
 1  1183  1174    4
 2   558   551    5
 3   669   654    5
 4   907   894    5
 5   245   240    9
 6   762   753    6
 7   240   248   12
 8   468   468    9
 9   331   319   12
     0,   4,  L
 0  1166  1182    5
 2  1068  1073    5
 3   845   842    6
 4   464   449    7
 5   877   877    6
 7   615   591    8
 8   243   242   15
 9   245   277   17
     0,   5,  L
 1  1047  1065    5
 2   470   482    7
 3   574   590    7
 4   774   772    6
 5   189   191   14
 6   646   630    7
 7   161   160   18
 8   416   434   10
     0,   6,  L
 0   964   975    6
 2   800   795    6
 3   578   582    7
 4   446   446    9
 5   622   623    8
 7   459   455   10
 8   251   263   17
     0,   7,  L
 1   944   930    7
 2   385   370    9
 3   554   546    8
 4   672   689    8
 6   485   490   10
 8   366   348   12
     0,   8,  L
 0   759   776    7
 1   154   150   18
```

```
COLUMNS ARE 10FO,10FC,  10SIG                                          COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG       L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG
     0,  8,  L          6   271   273   11         1,  9,  L                2,  4,  L          2   393   387   12         3,  6,  L
 2   638   645    8     7   497   518    9     0   143   189   22       2   338   335    7         3,  0,  L          0   619   619    6
 3   541   549    9     8   449   441   10     1   641   633    8       3   908   919    6     0   935   923    4     1   719   720    6
 4   213   224   16         1,  3,  L          2   448   436   10       5   679   715    7     1  1166  1158    4     2   254   278   11
 5   525   534    9     0   332   312    6     3   303   279   13       6   379   365    9     3   969   998    5     3   634   632    8
 7   431   409   11     1   949   921    4     4   549   533   10       7   415   429   10     4   175   170   13     5   464   472    9
     0,  9,  L          2   814   828    5     6   403   383   13       8   370   382   13     5   485   500    8     6   361   353   11
 1   651   634    8     3   441   443    7         1, 10,  L                2,  5,  L          6   469   475   10         3,  7,  L
 2   268   269   13     4   968   977    6     0   557   554    9       0   365   364    7     7   231   229   17     0   589   586    7
 3   380   387   11     6   658   673    7     1   227   250   17       1   596   584    6     8   458   443   12     1   265   272   11
 4   508   484   10     7   370   364   10     2   347   364   12       2   836   842    6         3,  1,  L          2   744   737    7
 5   203   155   18     8   321   319   11     3   344   368   14       3   177   211   14     0   755   732    4     4   590   562    9
 6   441   435   12     9   393   391   11     5   379   379   13       4   747   754    7     1   562   570    5     5   281   257   13
     0, 10,  L              1,  4,  L              1, 11,  L           5   370   376   10     2   964   954    5     6   278   288   15
 0   574   594    9     0   912   921    5     1   392   404   12       6   567   541    9     4   710   718    6         3,  8,  L
 2   352   373   11     1   775   755    5     2   272   261   15       7   404   418   12     5   399   388    9     0   387   389   10
 3   395   404   11     2   626   645    6         2,  0,  L                2,  6,  L          6   308   314   12     1   552   569    8
 4   182   200   20     3   914   883    6     0  1011   990    3       0   813   792    6     7   435   428   11     3   581   581    9
 5   399   400   12     5   842   823    7     1   575   575    4       1   638   643    7     8   151    99   25     5   369   352   12
     0, 11,  L          6   257   242   12     2   343   344    6       2   339   325    9         3,  2,  L              3,  9,  L
 1   402   389   11     7   454   462    9     3   990   989    5       3   692   665    7     0   846   842    4     0   423   420   10
 2   286   277   14     8   391   400   10     5   867   876    7       5   472   477   10     1   732   719    5     1   198   184   17
 3   297   270   14         1,  5,  L          6   430   410   10       6   291   332   15     2   277   262    7     2   576   549    9
 4   372   381   14     0   156   153   12     7   317   342   14       7   261   306   18     3   946   940    5     4   455   428   11
     0, 12,  L          1   809   831    5     8   440   430   12           2,  7,  L          4   166   168   13         3, 10,  L
 0   377   386   13     2   757   747    6         2,  1,  L           0   354   366    9     5   505   521    8     0   368   361   11
     1,  0,  L          3   439   437    8     1   322   335    6       1   540   552    7     6   432   427    9     1   436   426   11
 0  1604  1587    2     4   898   866    7     2  1217  1197    4       2   672   684    7     7   248   254   15     3   437   448   11
 1  1407  1385    3     6   619   616    9     4   829   837    6       3   195   200   16     8   367   389   13         3, 11,  L
 2   814   828    4     7   362   374   11     5   307   306   10       4   576   588    9         3,  3,  L          0   272   289   15
 3  1030  1028    5     8   342   347   11     6   497   494    9       5   149   137   23     0   862   840    5     1   193   214   21
 4   387   399    8         1,  6,  L          7   406   401   12       6   427   409   12     1   546   541    5         4,  0,  L
 5   874   872    6     0   994   983    6     8   182   194   23       7   329   324   15     2   938   932    5     0   504   504    5
 6   166   165   18     1   446   455    7         2,  2,  L                2,  8,  L          4   641   654    6     1  1038  1040    5
 7   443   443   10     2   593   587    7     0   483   468    5       0   510   502    8     5   375   379    9     3   735   751    6
 8   414   425   11     3   623   627    7     1  1036  1123    4       1   498   486    9     6   341   350   10     4   490   492    8
 9   265   272   16     5   705   682    9     2   459   460    5       2   280   275   12     7   397   404   10     5   365   366   10
     1,  1,  L          6   167   180   23     3  1090  1070    5       3   622   624    9         3,  4,  L          6   435   445   10
 0   517   527    3     7   387   363   11     5   731   751    7       5   456   458   11     0   642   638    5         4,  1,  L
 1  1293  1236    3     8   360   351   12     6   365   369   11       6   234   227   19     1   747   735    5     0   869   857    5
 2   777   777    4         1,  7,  L          7   363   340   12           2,  9,  L          2   439   424    6     1   260   257    8
 3   388   410    6     0   191   173   13     8   396   405   13       0   386   372   10     3   735   755    6     2   837   842    5
 4  1130  1125    6     1   716   722    7         2,  3,  L           1   432   410   10     4   219   211   12     3   257   268   10
 6   675   682    7     2   570   567    8     0   597   613    5       2   624   613    9     5   466   475    8     4   558   584    7
 7   435   450   10     3   333   333   11     1   566   585    5       4   492   481   11     6   498   500    9     5   432   442    9
 8   309   319   13     4   655   654    9     2  1099  1044    5       5   158   139   26     7   166   201   19     6   199   228   17
 9   454   445   11     6   503   490   11     3   312   293    7           2, 10,  L              3,  5,  L          7   434   462   11
     1,  2,  L          7   246   237   17     4   930   958    6       0   500   501   10     0   717   718    6         4,  2,  L
 0  1339  1316    3         1,  8,  L          5   282   282    9       1   364   383   12     1   408   415    7     0   456   442    6
 1   577   605    4     0   672   685    7     6   525   548    8       2   274   272   14     2   743   722    6     1  1139  1120    5
 2   661   688    5     1   325   311   11     7   432   396   11       3   457   456   11     4   535   534    7     2   170   152   12
 3  1123  1112    4     2   452   474    9         2,  4,  L               2, 11,  L          5   329   317   11     3   758   780    6
 4   150   125   14     3   627   621    8     0   750   755    5       0   205   204   19     6   299   296   11     4   385   389    8
 5   899   904    6     5   499   498   11     1   788   789    5       1   319   315   14     7   429   440   10     5   371   380   10
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC    SIG
      4,   2,  L
 6   441   454     9
      4,   3,  L
 0  1103  1083     5
 1   398   381     7
 2   795   787     6
 4   522   517     7
 5   359   349    10
 6   191   175    15
 7   361   368    11
      4,   4,  L
 0   477   475     6
 1   781   789     6
 2   155   174    14
 3   556   568     7
 4   328   324    10
 5   331   330    10
 6   406   394    10
      4,   5,  L
 0   705   695     6
 1   249   259    10
 2   648   633     7
 3   157   164    16
 4   418   407     9
 5   423   429     9
 6   200   201    16
      4,   6,  L
 0   372   367     8
 1   710   715     7
 3   556   544     8
 4   303   298    12
 5   350   348    11
 6   407   388    11
      4,   7,  L
 0   683   681     7
 2   564   561     8
 3   230   212    14
 4   402   404    10
 5   352   350    12
      4,   8,  L
 0   255   258    13
 1   484   503     9
 3   413   419    10
 4   241   267    16
      4,   9,  L
 0   430   435    10
 1   139   106    21
 2   445   443    11
      4,  10,  L
 0   230   214    16
 1   399   416    11
      5,   0,  L
 0   265   262     8
 1   741   755     6
```

```
 L   KFO    FC    SIG
 2   272   254     9
 3   547   550     7
 4   508   496     8
 5   237   230    14
 6   443   450    10
      5,   1,  L
 0   713   717     6
 2   674   686     6
 3   413   424     8
 4   311   317    11
 5   463   463     9
      5,   2,  L
 0   118   130    16
 1   747   753     6
 2   346   354     8
 3   523   541     7
 4   546   543     8
 5   237   228    13
 6   420   425    10
      5,   3,  L
 0   695   713     6
 2   661   671     7
 3   339   337     9
 4   384   389     9
 5   469   458     9
      5,   4,  L
 1   718   714     7
 2   194   184    14
 3   422   427     9
 4   409   398     9
 5   205   199    16
      5,   5,  L
 0   632   641     7
 2   511   495     8
 3   325   326    11
 4   348   328    10
 5   454   434    10
      5,   6,  L
 1   613   600     8
 2   213   187    14
 3   397   391    10
 4   389   370    10
      5,   7,  L
 0   493   514     9
 2   435   445    10
 3   264   235    13
 4   297   273    13
      5,   8,  L
 0   175   170    18
 1   453   454    10
 3   332   320    12
      5,   9,  L
 0   461   452    10
 2   385   378    12
```

```
 L   KFO    FC    SIG
      6,   0,  L
 1   578   572     7
 2   452   436     8
 3   347   332    10
 4   391   396    10
      6,   1,  L
 0   578   578     7
 1   163   151    15
 2   450   447     8
 3   424   428     9
 4   190   191    16
 5   397   402    11
      6,   2,  L
 1   471   484     8
 2   377   379     9
 3   253   271    13
 4   397   392    10
      6,   3,  L
 0   667   673     7
 2   402   435     9
 3   426   419     9
 4   238   233    13
 5   384   373    11
      6,   4,  L
 1   474   472     8
 2   354   317    10
 3   331   334    11
 4   413   413    10
      6,   5,  L
 0   509   493     8
 1   223   229    14
 2   385   373    10
 3   377   366    10
 4   177   175    19
      6,   6,  L
 1   412   414    10
 2   275   270    13
 3   315   276    12
      6,   7,  L
 0   478   464     9
 2   376   350    11
      6,   8,  L
 1   370   364    11
      7,   0,  L
 0   163   199    18
 1   461   453     9
 2   354   342     9
 3   170   160    18
      7,   1,  L
 0   532   533     8
 1   236   255    13
 2   315   318    11
 3   329   335    11
      7,   2,  L
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L   KFO    FC    SIG
      7,   2,  L
 1   428   403     9
 2   289   294    12
      7,   3,  L
 0   535   526     8
 1   267   292    12
 2   321   324    11
 3   349   355    11
      7,   4,  L
 0   176   175    17
 1   406   398     9
```

```
 L   KFO    FC    SIG
 2   332   326    11
      7,   5,  L
 0   455   455     9
 1   188   207    18
 2   231   245    15
      7,   6,  L
 1   288   303    13
      7,   7,  L
 0   342   347    12
      8,   0,  L
 0   287   292    12
```

```
 L   KFO    FC    SIG
 1   296   287    12
      8,   1,  L
 0   375   374    10
 1   335   353    11
      8,   2,  L
 0   203   208    15
 1   251   265    14
      8,   3,  L
 0   331   316    11
 1   283   294    13
```

OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR

cis-$Cp^*Re(CO)_2I_2$

```
COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO    FC   SIG
      -7, K,  1
  0   525   479    18
  1   554   533    18
  3   237   226    33
  4   539   534    18
  5   953   980    14
  7   494   522    20
  8   509   493    19
  9   688   680    16
      -7, K,  2
  2   422   403    21
  3   729   739    15
  7   830   839    14
  8   333   251    25
  9   258   276    34
 10   237   240    35
 11   458   500    21
 12   318   249    27
      -7, K,  3
  0   529   503    18
  1   710   692    16
  3   596   565    16
  5   725   764    15
  7   525   519    18
  8   232    69    30
  9   648   668    16
 10   484   481    20
 11   382   367    25
 12   444   388    21
      -7, K,  4
  1   761   767    16
  2   923   944    15
  3   576   545    18
  4   228   211    35
  5   561   591    17
  6   545   547    18
  7   525   526    20
 10   551   535    20
 11   580   622    21
      -7, K,  5
  0   885   926    18
  1   294   305    37
  4   783   769    18
  5   298   256    34
  8   690   596    18
  9   563   483    22
      -7, K,  6
  1   228   284    35
  3   247   280    31
  4   256   239    33
  6   354   349    27
  7   339   372    30
      -6, K,  1
  1   695   658    14
```

```
  K   KFO    FC   SIG
  2  1451  1489    11
  5   312   253    24
  6  1215  1230    11
  7   463   462    18
  8   265   294    29
  9   262   277    30
 10  1057  1071    13
 14   787   786    14
 16   258   257    33
      -6, K,  2
  0  1033  1072    12
  1   381   309    20
  3   661   636    14
  4   606   646    14
  6   312   309    24
  7   201   178    33
  8   587   585    15
 10   905   869    13
 11   250   170    32
 12  1014  1005    13
 14   576   541    17
 15   377   350    25
 16   666   709    17
 18   386   375    27
      -6, K,  3
  1   247   307    31
  2   925   919    12
  5   208   276    34
  6   402   411    18
  8   583   572    15
 10   761   783    15
 12   835   832    15
 14   961   944    15
 16   340   398    29
 18   429   450    25
      -6, K,  4
  0  1762  1797    13
  1   261    87    33
  2   208    58    33
  3   327   321    22
  4  1418  1419    11
  5   396   354    22
  8  1127  1071    14
 10   313   274    30
 12  1008   984    16
 16   641   631    19
 17   348   269    27
      -6, K,  5
  1   428   408    19
  2  1137  1242    12
  3   701   632    14
  5   672   625    16
  6  1190  1167    13
  7   526   512    20
```

```
  K   KFO    FC   SIG
  9   804   768    16
 10   929   874    16
 13   388   310    25
 14   548   549    22
      -6, K,  6
  1   385   400    20
  2   241   249    30
  3   390   377    21
  4   439   455    20
  5   466   448    19
  6   422   434    22
  7   488   475    20
  8   604   570    17
 10   360   408    26
 12   354   406    28
 14   535   537    23
      -6, K,  7
  2   378   457    22
  6   600   644    18
  7   275   316    30
 10   466   527    24
      -6, K,  8
  0   777   749    16
  1   336   329    26
      -5, K,  1
  0  1777  1804    10
  1  1026  1040    10
  2   340   294    19
  3  1459  1488     9
  4  1255  1253    10
  5   411   423    17
  7   524   478    14
  8   867   877    11
  9   917   944    12
 10   456   432    16
 11   439   400    18
 12   853   890    13
 13  1151  1167    12
 15   760   726    14
 16   469   488    20
 17   461   490    19
 21   340   383    29
 22   372   365    27
      -5, K,  2
  1  1199  1180    10
  2   650   627    12
  3   930   907    10
  4   403   372    15
  5  1163  1175    10
  6   301   341    21
  7   897   922    11
  8   743   752    12
  9   679   725    13
 10   216   226    32
```

```
COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO    FC   SIG
      -5, K,  2
 11  1069  1091    12
 12   927   967    13
 13   541   554    17
 14   622   653    17
 15   944   929    14
 16   282   279    30
 17   351   300    25
 19   498   567    22
 20   418   395    25
      -5, K,  3
  0   660   661    13
  1   506   510    15
  2   270   290    22
  5  1118  1174    11
  7   296   303    24
  9   957   964    13
 10   793   806    14
 12   500   452    19
 13   899   880    15
 14   446   451    21
 16   329   324    28
 17   999   969    15
 19   258    75    33
 21   763   760    18
      -5, K,  4
  1   194   234    31
  2  1089  1130    10
  3  1224  1243    11
  5   349   370    23
  6   729   692    15
  7  1299  1326    12
  9   645   625    17
 10   490   480    21
 11   836   796    15
 14   316   276    30
 15   892   815    16
 19   880   894    17
      -5, K,  5
  0   250   205    24
  1  1169  1193    11
  3   973  1009    12
  4   362   295    20
  5  1366  1361    11
  7  1324  1350    12
  8   267   213    31
  9   975   960    13
 11   785   825    15
 13   751   746    16
 15   241   196    35
 17   785   750    17
 20   262    86    36
      -5, K,  6
  1   704   722    13
```

```
  K   KFO    FC   SIG
  2   807   806    13
  3   821   867    13
  4   408   385    19
  5   651   667    15
  6   384   369    21
  7   817   773    13
  9   507   547    19
 10   427   314    21
 11   770   776    16
 14   237   269    38
 15   744   764    17
 16   458   425    24
      -5, K,  7
  0   222   231    32
  1   397   402    19
  5   470   511    18
  9   719   691    16
 13   774   821    18
      -5, K,  8
  1   686   633    16
  3   588   577    18
  4   240   134    32
  5   500   491    19
  7   602   586    18
  8   257   233    33
  9   232   258    38
      -4, K,  1
  1  2678  2659     8
  2   496   433    12
  3  1294  1369     8
  4   692   728    10
  5  1880  1897     8
  6   958   963     9
  7  1048  1041     9
  9   619   630    12
 10   373   426    18
 11  1425  1445    10
 13   685   727    14
 15   944  1042    13
 16   793   843    14
 17   500   449    18
 18   461   474    20
 19   449   463    21
 20   430   442    22
 22   375   379    24
 23   479   497    23
      -4, K,  2
  0  1080  1101     9
  1  1416  1405     8
  2   449   521    13
  3  1411  1422     8
  4  2129  2163     8
  5  1423  1440     8
  6   294   258    19
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  K   KFO    FC   SIG
  7  1122  1184    10
  8  1877  1899     9
  9  1207  1256    10
 10   764   788    12
 11   655   654    13
 12   216   340    35
 13   922   965    12
 16   234   304    37
 17   905   904    14
 18   462   501    22
 20   589   595    19
 21   486   619    28
 25   383   394    28
      -4, K,  3
  1   304   357    17
  2  1290  1292     9
  3   971   907    10
  4   512   487    14
  5   304   258    22
  6  1911  1848    10
  7  1464  1397    11
  8   208    88    32
 10   932   937    12
 11   792   750    14
 13   561   555    17
 14   404   361    20
 15   905   850    14
 16   512   471    20
 17   399   386    23
 18   536   490    20
 19  1090  1098    14
 22   334   407    31
 23   573   581    20
      -4, K,  4
  1   576   590    11
  4   298   272    19
  5  1314  1328    10
  6   479   434    16
  7   596   559    14
  8   376   418    19
  9   898   868    12
 10   858   935    13
 11   249   184    29
 12   601   597    15
 13   470   461    19
 14   922   919    14
 15   338   342    28
 16   600   620    19
 17   908   890    16
 18   632   683    20
 19   236    55    34
 20   429   452    26
 21   647   675    19
 22   509   508    23
```

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      COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO    FC   SIG      K   KFO    FC   SIG      K   KFO    FC   SIG
     -4, K,  5             10   450   466    22     12  1259  1146    11
  1   926   983    10     12   440   495    22     13   638   684    15
  2  1104  1161    10          -4, K,  9            14   741   685    14
  3  1129  1143    10      1   261   224    34     15  1001   966    13
  5  1102  1100    10      2   771   826    16     16   812   905    14
  6   581   611    14      5   241   185    35     18   703   703    16
  7   906   894    11          -3, K,  1            20   747   767    16
  8   511   467    15      0   594   519     9     22   481   491    21
  9  1104  1096    12      2  1028  1086     7     24   411   400    25
 10   828   829    13      3   949  1019     7     26   486   502    24
 11   365   397    23      4  2078  2065     7          -3, K,  4
 12   768   759    14      5   186   151    22      1  1190  1170     8
 13   403   383    22      6  1077  1092     8      2   816   775     9
 14  1011  1000    13      7   804   830     9      3   188    73    23
 15   277   353    32      8  2376  2391     8      4   195   203    25
 16   369   404    24     11   558   587    12      5   565   558    12
 17   233    95    35     12   824   827    11      6   573   484    12
 18   579   643    20     13   304   279    20      8  1044  1069    10
 19   421   423    23     14  1039  1080    11      9   348   337    18
 20   431   417    24     15   668   630    14     10  1006   910    11
 22   286   359    37     16   701   720    14     11   642   696    14
     -4, K,  6             18  1426  1401    12     12  1295  1291    11
  0  1957  2114    10     20  1065  1035    14     13   470   401    18
  1   432   442    17     22   420   504    25     14  1077  1052    12
  2   247   185    26     24   435   429    24     15   448   494    20
  3   411   409    18     26   441   446    24     16   896   860    13
  4  1524  1552    10          -3, K,  2            17   282   320    32
  5   307   340    23      1   637   692     9     18   598   646    17
  8  1149  1117    12      2  2699  2789     7     20   768   739    16
  9   322   372    25      4  1042  1007     9     22   374   326    27
 12  1117  1063    13      5   239   250    24     23   297   317    36
 13   312   331    27      6  3864  3875     8     24   743   701    18
 16   700   697    17      7   195    31    28          -3, K,  5
 17   242   281    39      9   765   810    11      0  2055  2111     8
 18   224   115    35     10  2237  2212     9      4  1382  1405     9
 20   323   363    30     12   322   363    21      5   284   302    22
     -4, K,  7             14   784   788    13      7   649   626    12
  2  1079  1038    12     16   770   757    14      8  1147  1119    10
  5   365   352    20     18   956   965    13      9   622   655    14
  6   813   757    13     20   491   502    20     10   880   885    12
  8   287   230    26     22   809   822    15     11   367   399    21
  9   572   549    17     24   255   287    34     12  1356  1271    11
 10   688   711    16          -3, K,  3            13   650   587    15
 11   403   390    22      0  1830  1950     7     14   462   473    19
 12   461   418    20      1  1018  1106     8     15   257   122    29
 14   494   540    21      2   217   242    20     16   739   820    15
 15   370   361    27      3  2209  2301     8     17   564   544    17
 16   259   364    38      4  2254  2140     8     21   534   534    21
     -4, K,  8              6   535   524    12     22   606   549    20
  0   539   560    15      7   626   630    11          -3, K,  6
  1   221   219    33      8  1920  1845     9      1   491   543    15
  4   505   535    18      9   587   548    13      2  2000  2068     9
  6   347   346    24     10   414   409    17      3   849   765    11
  8   549   543    18     11   561   583    15      4   214   231    32
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      COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO    FC   SIG      K   KFO    FC   SIG      K   KFO    FC   SIG
     -3, K,  6              9   442   453    13      2   357   350    13
  5   538   563    14     10   713   714    11      3  1482  1491     8
  6  1306  1317    11     11  1888  1839     8      4  1201  1212     8
  7   937   927    12     15  1572  1529    10      5  1253  1165     8
  8   224   179    30     18   604   598    18      7   513   536    12
  9   569   510    15     19  1071  1198    16      8  1064  1022     9
 10  1184  1138    12     22   313   575    42      9  1646  1630     9
 11   573   614    16     23   953  1017    15     10  1183  1242    10
 12   270   246    29     27   522   671    25     11   493   435    14
 14   802   822    15          -2, K,  2            13  1650  1582    10
 15   432   462    22      0  1436  1555     6     14   742   783    13
 16   226    49    31      1  1848  1900     6     15   464   450    17
 18   342   357    27      2   453   471    10     16   218   238    35
 19   472   513    23      3  2280  2097     6     17   843   809    13
     -3, K,  7              4  3020  2923     6     21   781   764    16
  0   521   580    16      5  1020   871     8     22   332   363    29
  1   897   917    12      6   226   113    21     23   270   306    36
  3   815   820    12      8  2035  2027     8     25   782   783    18
  4   499   499    16      9  1821  1721     8          -2, K,  5
  5   992   943    12     10   929   930    10      1   476   451    12
  6   355   332    22     11   250   222    22      2   299   378    17
  7   931   908    13     13  2242  2256     9      3  1084  1034     9
  8   559   538    17     14   229   260    32      4   444   429    13
  9   618   629    16     15   904   878    11      5   178   151    28
 10   447   427    19     17  1043  1030    12      6   547   548    13
 11   579   557    17     18   265   267    29      7  1464  1428     9
 12   417   483    22     20   404   394    23     11  1478  1429    10
 13   340   325    23     21   808   764    15     14   473   454    18
 14   309   369    29     23   319   325    27     15  1253  1208    12
 17   419   397    24     25   882   896    16     16   334   291    25
 18   370   408    28          -2, K,  3            19  1023  1027    14
     -3, K,  8              1  3234  3402     6     23   824   819    17
  1   215   312    33      2   536   496     9     24   233   130    38
  3   517   552    17      3  1588  1643     7          -2, K,  6
  4   370   330    22      5  2567  2529     7      0   605   643    12
  6   277   282    28      6   717   718     9      1  1081  1106    10
  7   589   588    16      7  1295  1201     8      2   241   215    23
  8   386   438    21      8   167   159    27      3   752   735    11
 10   340   335    24      9  1048  1068     9      5  1584  1557    10
 11   430   458    21     10   221   238    24      6   470   476    16
 12   390   411    23     11  2193  2147     9      7  1116  1090    10
 14   245   266    35     12   282   358    25      8   229    40    29
     -3, K,  9             13  1621  1580    10      9  1295  1279    11
  0   489   442    19     15  1413  1423    11     10   303   320    25
  1   318   314    26     16   224   182    33     11   786   785    13
  4   525   498    19     17   760   757    13     13   946   969    13
  5   459   445    20     19   404   417    22     14   475   429    19
     -2, K,  1             21   360   319    23     17   898   920    15
  2  1160  1142     6     23   738   733    17     18   331   333    28
  3  1201  1065     6     25   248   273    37     21   756   743    17
  4  1281  1364     6     27   544   618    23          -2, K,  7
  5   531   549    10          -2, K,  4             1   803   820    12
  6  2350  2244     7      0   673   708     9      2   556   549    15
  7  1722  1623     7      1  1291  1306     8      3  1159  1142    11
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COLUMNS ARE 10FO,10FC, 10SIG

K	KFO	FC	SIG
	-2,	K, 7	
5	995	964	12
6	490	512	18
7	1022	1033	12
9	976	967	13
10	414	397	21
11	683	717	15
13	361	370	24
14	458	364	19
15	603	625	17
18	260	291	37
19	571	607	22
	-2,	K, 8	
0	703	659	14
1	444	486	19
4	571	542	16
5	546	611	17
7	239	282	34
8	401	442	23
9	609	570	16
12	325	387	30
13	619	586	18
	-2,	K, 9	
1	344	300	25
3	424	427	21
7	513	489	19
	-1,	K, 1	
0	2713	2735	4
1	1744	1574	4
2	741	780	6
3	1039	909	6
4	877	806	7
5	1141	923	7
6	416	403	13
7	1514	1612	7
9	1686	1625	8
10	1716	1675	8
11	1131	1187	9
12	1677	1683	9
13	1833	1866	9
14	400	399	17
16	1216	1185	11
17	1315	1285	11
21	911	934	14
22	780	801	16
23	276	322	34
25	778	841	19
26	484	529	23
	-1,	K, 2	
1	2703	2758	5
2	346	267	12
3	566	562	8
5	1502	1431	6
7	1325	1232	7

K	KFO	FC	SIG
8	950	996	8
9	701	704	9
10	723	658	10
11	2249	2199	8
12	1442	1410	9
13	490	552	14
14	1225	1225	10
15	1349	1352	10
16	890	894	12
17	451	438	18
18	540	517	17
19	366	388	22
20	1029	1003	13
21	295	395	30
23	711	705	17
24	710	734	18
25	240	237	37
26	313	294	32
27	597	631	20
28	403	358	25
	-1,	K, 3	
0	1155	1104	7
1	1673	1775	6
2	603	640	9
3	3426	3477	6
4	2253	2270	7
5	495	448	10
6	611	582	9
7	1144	1174	7
8	2207	2278	7
9	1314	1379	8
11	765	738	10
12	1157	1194	9
13	1381	1366	9
14	390	413	17
15	1094	1065	11
16	736	694	13
17	389	416	19
18	855	801	13
20	645	643	16
21	310	313	29
22	529	544	19
24	402	408	24
25	584	537	19
26	295	358	33
	-1,	K, 4	
1	711	715	9
2	2161	2219	7
3	291	244	16
4	685	714	9
6	3084	3051	8
7	580	592	11
8	198	257	25
9	381	383	15

K	KFO	FC	SIG
10	1594	1627	9
11	742	777	11
12	347	329	17
14	713	725	13
15	697	646	14
16	649	629	14
18	803	781	14
19	574	608	17
20	477	499	20
22	600	630	18
23	589	570	19
	-1,	K, 5	
0	439	416	12
1	236	224	20
2	436	493	13
3	575	585	12
4	1247	1255	9
5	624	591	11
6	627	567	11
7	357	303	15
8	1328	1324	9
9	661	701	12
10	221	210	27
11	247	290	24
12	692	699	13
13	434	407	18
14	826	824	13
15	487	441	18
16	743	698	14
17	659	639	16
18	892	929	14
19	228	194	37
20	691	687	17
21	643	633	18
22	452	469	22
24	337	320	26
	-1,	K, 6	
2	804	732	11
3	666	655	12
4	383	387	17
5	533	531	15
6	1010	1039	11
7	647	659	13
8	524	509	15
9	664	643	13
10	857	871	12
12	771	759	13
14	873	823	13
15	233	202	30
16	843	798	15
18	742	730	16
19	393	362	23
20	510	539	20
22	454	466	23

COLUMNS ARE 10FO,10FC, 10SIG

K	KFO	FC	SIG
	-1,	K, 7	
0	1653	1653	11
1	417	457	20
3	358	349	21
4	1461	1441	11
5	403	415	19
7	518	477	16
8	1071	1097	12
10	350	309	23
11	222	201	34
12	1006	968	14
13	214	113	35
14	352	363	24
16	793	774	15
	-1,	K, 8	
1	221	117	31
2	1222	1226	12
4	211	80	33
6	886	922	13
10	782	790	15
12	250	278	35
13	257	223	34
14	622	635	19
	-1,	K, 9	
0	570	643	17
4	490	557	20
	0,	K, 0	
4	4484	4683	4
6	562	616	9
8	1661	1730	7
10	1211	1183	8
12	2694	2776	8
14	187	214	28
16	1615	1600	10
18	209	311	29
20	225	318	31
22	736	729	14
24	394	369	23
26	683	703	17
28	504	429	21
	0,	K, 1	
1	500	554	6
2	4119	4133	4
3	1650	1768	4
4	430	415	9
5	1454	1506	6
6	995	950	7
7	796	854	8
8	1194	1204	7
9	2491	2430	7
10	2201	2148	7
11	556	644	11
12	1630	1558	8
13	459	459	15

K	KFO	FC	SIG
14	1840	1844	9
16	638	597	13
18	764	742	12
19	449	479	17
20	758	754	14
21	255	223	31
22	341	391	24
24	911	906	15
26	562	520	18
28	525	488	22
	0,	K, 2	
0	744	698	7
2	306	350	12
3	1468	1402	6
4	526	395	9
5	1818	1822	6
6	1117	1249	7
7	1002	1075	8
8	1396	1262	7
9	313	321	15
10	1517	1437	8
11	330	326	16
12	1396	1384	8
13	490	533	13
14	1493	1539	9
15	906	970	11
16	991	1006	11
17	742	707	13
18	1401	1391	11
20	925	894	13
21	544	598	18
22	988	928	14
24	619	623	18
26	660	576	17
28	242	275	38
	0,	K, 3	
1	754	809	8
2	2294	2263	6
3	797	741	8
4	1052	1090	7
5	676	680	9
6	2705	2726	7
7	934	870	8
8	365	334	13
9	172	161	25
10	1745	1777	8
13	896	845	10
14	851	845	10
15	723	749	12
16	987	1008	11
17	404	350	19
18	1130	1102	12
19	1040	999	13
20	813	741	14

K	KFO	FC	SIG
22	876	903	14
23	294	329	31
27	244	212	37
	0,	K, 4	
0	1822	1841	7
1	962	1018	8
2	481	486	11
3	960	1016	8
4	2773	2777	7
5	720	774	10
6	418	430	13
7	668	671	11
8	2534	2554	8
9	711	696	12
10	544	543	12
11	324	364	19
12	893	824	11
13	849	877	11
14	301	245	21
15	325	347	22
16	550	521	15
17	767	802	14
18	711	724	15
20	777	761	15
21	328	366	26
26	299	346	32
	0,	K, 5	
1	1447	1484	9
2	712	705	10
3	729	696	10
4	469	451	13
5	1093	1137	9
6	1204	1227	9
7	577	567	12
8	418	445	15
9	388	379	17
10	641	641	13
11	868	837	12
12	636	581	14
13	559	510	15
15	642	669	15
16	930	929	14
17	452	363	20
18	476	479	20
19	274	283	28
20	501	537	19
22	354	306	25
23	241	344	39
	0,	K, 6	
0	717	687	11
1	649	626	12
3	721	766	12
4	636	636	12
5	371	342	18

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    COLUMNS ARE 10FO,10FC,  10SIG

   K    KFO     FC   SIG       K    KFO     FC   SIG       K    KFO     FC   SIG
         0, K,   6            16    924    933    12       7    459    409    12
   6    492    499    15      18    250    306    28       8   1438   1421     8
   8    435    493    17      19   1308   1343    11       9   1641   1588     8
   9    668    683    14      23    713    765    17      10    912    897     9
  10    789    732    13      24    350    602    35      12    174     65    28
  11    219    145    31      27    455    455    24      13   1536   1549    10
  12    506    437    18             1, K,   1            15    237    277    27
  13    863    817    14       0    555    524     7      17   1613   1532    11
  14    697    694    16       1   2388   2371     5      18    242    279    33
  16    531    510    19       2    311    264    12      20    338    303    25
  17    550    527    18       3   1193   1210     6      21   1014   1012    14
  18    456    471    21       4    176    114    22      25    645    643    17
  20    311    344    30       5   3381   3470     6             1, K,   4
  21    449    438    23       7   3669   3709     6       1   2067   2046     8
         0, K,   7             9   1776   1737     7       3   1620   1685     8
   1    614    627    15      10    883    871     9       4    315    294    16
   2    955    980    13      11   2465   2517     8       5   1734   1622     8
   3    599    548    15      12    853    840    10       6   1033   1056     9
   5    354    355    20      13    735    727    11       7   1414   1412     9
   6    654    682    15      14    256    296    24       8    666    647    11
   7    684    711    15      16    596    585    14       9    898    873    11
  10    641    604    15      17   1550   1527    10      11   1644   1644    10
  11    814    819    14      21   1155   1164    13      12    697    668    13
  12    343    288    23      22    611    568    16      13    605    636    14
  14    532    516    19      23    297    260    28      14    370    330    19
  15    694    672    17      25    260    356    36      15   1290   1306    11
  16    239    108    32      26    249    258    37      16    230     64    29
         0, K,   8                   1, K,   2            17    370    387    22
   0    549    564    18       1    268    171    15      19    662    637    15
   1    669    689    16       2   1404   1442     6      23    536    529    19
   3    607    640    17       3   2651   2555     6      24    251    236    35
   4    444    405    19       4    789    860     8             1, K,   5
   5    737    727    15       6    243    266    17       0   1096   1104    10
   7    603    516    17       7   2039   2005     7       1   1613   1595     9
   8    379    341    23       8    418    403    12       3   1733   1712     9
   9    633    701    18       9   1411   1389     8       4    618    601    13
  11    401    372    24      11    876    845     9       5    949    965    11
  12    340    341    27      12    304    228    18       7    622    665    13
         1, K,   0            15   1385   1395    10       8    456    406    16
   1   1684   1635     4      16    670    685    14       9   1152   1139    11
   2   3055   3033     4      17    307    283    22      11    473    459    16
   3   2584   2562     5      19   1755   1753    11      12    395    409    19
   4   1182   1322     6      20    656    617    16      13   1242   1222    12
   5   1594   1556     6      21    265    273    31      15    584    570    17
   6    866    901     8      23    764    799    15      17    611    611    16
   7   2398   2376     6      25    233    121    31      21    466    437    20
   8    440    453    13      27    482    469    21             1, K,   6
   9   1859   1876     7             1, K,   3             1    636    683    14
  10   1264   1372     8       0    677    731     8       2    512    584    15
  11   1449   1445     8       1   1199   1161     7       3    596    587    13
  12    673    645    11       2    664    680     9       5    241    262    27
  13    444    461    14       4   1139   1157     8       6    613    601    14
  14   1873   1813     9       5   1921   1883     7       7    804    804    13
  15   1308   1289    10       6    400    355    13      10    391    399    21
```

```
    COLUMNS ARE 10FO,10FC,  10SIG

   K    KFO     FC   SIG       K    KFO     FC   SIG       K    KFO     FC   SIG
         1, K,   6            10   1210   1237     9      23    368    333    27
  11   1120   1093    12      11    309    277    17      24    734    778    18
  12    227    134    33      12    315    327    17      25    252    156    38
  15    849    821    15      13    455    410    14             2, K,   4
  19    551    560    20      14   1252   1220    10       0   1603   1600     9
         1, K,   7            15    682    674    13       1    690    718    11
   1    421    459    19      16    411    362    17       3   1137   1139    10
   2    225    165    32      18    902    907    13       4    974    971    10
   3    314    371    25      19   1129   1210    13       6    505    454    14
   5    670    634    14      20    398    354    22       7    361    332    17
   6    260    307    30      22    333    355    27       8    788    792    12
   7    217    218    35      23    386    406    23       9    560    545    14
   8    377    364    22      24    423    439    23      10   1057   1031    11
   9    829    851    14             2, K,   2            12   1279   1300    11
  13    801    787    15       0   3493   3539     7      13    821    827    13
  14    350    265    24       1    486    440    11      14    623    602    16
         1, K,   8             2    414    458    12      15    593    534    16
   1    443    473    21       3    484    452    11      16    899    876    14
   2    357    352    25       4   2309   2321     7      17    300    352    28
   3    597    579    16       5   1695   1675     8      18    492    497    20
   5    472    430    20       6    365    407    14      19    237    176    35
   6    476    442    19       7    749    736     9      20    340    371    25
   7    568    612    19       8   1299   1315     8      22    723    711    17
         2, K,   0             9    804    748    10             2, K,   5
   0   3128   3003     5      10    618    670    12       1    733    759    13
   1   1414   1341     6      12   1680   1671     9       2   1518   1534    10
   2    890    990     7      13    419    428    16       5    446    486    18
   3    188    212    21      15    568    565    14       6   1520   1474    10
   5   2534   2541     6      16   1136   1095    11      10   1397   1363    11
   6   1097   1180     7      17   1054   1016    12      11    413    418    20
   7   1884   1866     7      21    765    720    15      12    217    168    34
   8   1477   1334     8      22    487    543    20      13    282    290    29
   9   1380   1325     8      24    224    127    34      14    927    958    14
  10    317    329    17      26    510    553    22      17    242    225    33
  11   1323   1233     9             2, K,   3            18    474    545    22
  12    294    273    19       1    279    208    17      20    497    490    21
  13    906    887    11       2   1519   1523     8             2, K,   6
  14   1174   1176    10       3    490    494    11       0    799    872    13
  16    554    569    15       4    373    378    14       4   1108   1124    12
  17   1413   1389    11       5    177     75    26       7    228    212    35
  18   1343   1373    12       6    464    363    12       8   1237   1165    12
  20    840    821    14       7    599    591    12      12    746    780    15
  21   1058   1073    13       8    887    896    10      14    327    305    26
  24    266    306    35       9    227    267    26      16    482    486    20
         2, K,   1            10   1028   1032    11             2, K,   7
   1    598    585     9      11    762    759    12       2    555    621    18
   2   2369   2272     6      12   1267   1301    11       4    316    290    25
   3   2624   2667     6      13    253    266    31       6    771    793    15
   4    249    197    18      14   1358   1352    11      10    601    604    17
   5   1601   1690     7      15    973   1001    12             3, K,   0
   6   1976   2012     7      16    361    369    23       1    509    544    10
   7   1946   1993     7      18    352    359    24       2   1184   1066     8
   8    158    118    26      19    706    651    15       3    693    737     9
   9   2665   2649     8      20    585    634    18       4   1050   1055     8
```

```
COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO     FC   SIG        K   KFO     FC   SIG        K   KFO     FC   SIG
      3, K,  0               12   806    865    12        8   939    908    13
  5   271    316    18       13   636    668    14        9   639    646    16
  6  1586   1579     8       14  1480   1434    11       11   263    290    31
  7   411    458    14       15   362    337    22       12   467    533    20
  8   739    692    10       18   590    600    16       13   544    571    19
  9  1567   1597     9       20   243    344    35       17   774    744    17
 10  1134   1111    10       21   226    251    36            3, K,  6
 11   850    844    11       23   329    331    30        1   660    663    17
 12   977    985    11       24   625    636    19        2   427    507    22
 14   776    770    12            3, K,  3               3   743    767    15
 15   477    523    17        0  1554   1610     9        4   233    168    34
 16  1515   1468    11        1   722    722    11        5   727    639    15
 17   261    262    33        2   294    297    20        6   663    681    16
 18   985    961    13        3   513    532    13        7   594    623    18
 19   264    175    31        4   515    527    13        9   497    471    19
 20   882    870    14        5  1022    994    10       10   382    414    24
 21   394    404    25        6   199    225    30       11   438    504    23
 22   682    632    17        7   754    776    12            4, K,  0
 24   536    543    20        8   309    279    21        0   416    369    14
      3, K,  1                9   590    624    14        1  1319   1308     9
  0  3294   3290     7       10  1180   1150    11        3  1620   1582     9
  1   182    154    23       11   625    596    15        5   442    417    14
  3   692    669    10       12  1125   1068    12        6   242    298    24
  4  2392   2350     8       13   235    256    32        7   369    385    17
  5   534    513    11       14   686    675    15        8   204    170    29
  7  1070   1139     9       16   802    758    14        9  1506   1437    10
  8  1533   1562     9       17   396    419    22       10   391    329    17
  9   705    722    11       18   397    425    22       11   240    289    32
 10   705    727    11       21   505    506    20       13  1748   1669    11
 11   787    758    12       22   652    676    19       14   478    472    19
 12  1439   1475    10            3, K,  4              15   463    441    19
 13  1154   1136    11        2   887    912    12       16   472    500    19
 14   607    629    15        3   738    700    12       17   703    717    15
 15   389    351    21        4   231    167    28       18   363    316    23
 16  1098   1111    12        6   586    572    15       20   347    353    24
 18   357    325    23        7   872    857    12       21   596    595    18
 19   235    264    32        8   675    637    14       23   359    318    27
 20   546    546    18        9   354    412    21            4, K,  1
 22   466    509    21       10   785    805    13        1  1420   1396     9
 23   411    384    25       11   403    385    20        2  1315   1260     9
 24   347    312    26       12   569    547    16        3   651    685    12
 25   481    461    24       14   669    673    15        4   511    562    14
      3, K,  2               15   587    552    17        5   427    417    15
  1  1114   1148     9       16   381    374    23        7   964    997    11
  2  2822   2831     8       18   367    375    27        9   326    338    22
  3   550    573    12       19   842    805    16       11  1729   1752    11
  4   670    667    11       20   515    513    22       13   316    317    25
  5  1062    990     9            3, K,  5              14   764    748    15
  6   954    973    10        0   939    982    13       15  1249   1231    13
  7   739    737    11        1   385    425    20       16   335    383    26
  8   416    436    15        4  1007   1002    13       18   361    298    23
  9   516    491    14        5   828    873    14       19   707    733    17
 10  1184   1211    10        6   210    184    33       23   841    805    17
 11   938    930    12        7   598    561    16            4, K,  2
```

```
COLUMNS ARE 10FO,10FC,  10SIG

  K   KFO     FC   SIG        K   KFO     FC   SIG        K   KFO     FC   SIG
      4, K,  2                    5, K,  0                4  1296   1265    12
  0  1043   1099    11        1   845    859    12        5   632    627    17
  1  1405   1400    10        2  1073   1123    11        7   456    453    19
  2   541    541    15        5   231    147    27        8  1100   1080    13
  3  1212   1229    10        6  1398   1386    11        9   237    221    35
  5  1220   1260    11        7   352    356    21       10   215     81    35
  6   456    455    17        9   554    577    17       11   340    325    26
  7   851    869    12       10  1002   1032    13       12   626    625    18
  8   281    225    24       11  1163   1151    13            5, K,  4
  9  1266   1307    11       13   256    246    29        1   260    254    34
 11   820    775    14       14   488    492    17        2   994    967    14
 12   558    496    16       15   815    809    14        6   685    734    17
 13  1013   1022    13       16   295    321    28        7   253    247    34
 15   347    341    26       17   302    238    27        9   257    208    33
 16   239    211    35       18   297    362    29            6, K,  0
 17   780    807    15       19   334    343    26        0   878    867    13
 18   427    417    22       20   359    422    27        1   340    289    22
 20   342    301    28       21   242    262    39        4  1219   1210    11
 21   731    757    17            5, K,  1                5   716    697    14
      4, K,  3                1   449    470    17        6   311    303    25
  1   749    724    13        2   482    478    15        7   729    686    14
  2   236    211    30        3   484    492    17        8  1229   1229    12
  3  1313   1300    11        4   756    774    13        9   246    226    31
  4   261    235    28        5   348    399    23       11   393    364    20
  5   682    691    14        6   612    598    15       12   655    631    15
  6   627    644    14        7   240     36    28       14   227    310    38
  7  1346   1284    11        8  1121   1084    12       16   291    336    30
  8   317    270    25        9   781    822    15            6, K,  1
  9   937    921    13       12   365    385    25        1   324    325    24
 11   643    701    16       13   843    778    15        2   341    396    24
 12   253    217    32       14   561    563    18        3   446    434    20
 13   325    257    22       15   264    270    34        4   629    593    16
 15   606    599    17       16   313    345    29        5   428    404    22
 18   269    260    34       17   255    360    41        6  1041   1025    14
 19   786    813    17       18   800    806    17        7   443    363    21
      4, K,  4                    5, K,  2               10   687    654    16
  0   282    282    26        1   376    436    21       11   253    260    34
  1   629    635    15        2   624    626    15       14   246    235    35
  5  1141   1109    12        3   689    700    14            6, K,  2
  7   468    499    19        4   705    659    15        0   494    488    19
  9   788    770    14        5   598    551    16        1   245    306    36
 13   546    558    19        6  1357   1328    12        2   303    276    28
 15   296    255    31        7   734    728    15        3   374    366    25
 16   242    164    37        8   255    231    33        4   802    792    15
      4, K,  5                9   710    691    16        6   371    276    23
  1   455    444    19       10   869    814    14        8   735    765    16
  2   347    357    23       11   339    385    27        9   429    411    24
  3   737    788    15       13   414    391    22            6, K,  3
  5   419    402    20       14   370    359    24        1   533    555    19
  7   757    780    15       15   237    192    35        2   673    691    18
  8   255    221    31       16   669    695    18            7, K,  0
  9   368    393    24            5, K,  3                1   653    605    17
 10   236     58    34        0  1168   1101    12        3   852    850    15
 11   578    597    19        1   218    236    35        4   247    185    34


  K   KFO     FC   SIG        K   KFO     FC   SIG        K   KFO     FC   SIG
      7, K,  0                6   457    388    21        8   248    134    38
  5   656    640    17        7   786    780    16
```

OBSERVED AND CALCULATED
STRUCTURE FACTOR LISTINGS FOR
trans-Cp*Re(CO)$_2$Br$_2$

```
COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
     -9,   0,  L
  2   221   158   25
  6   498   488   13
     -9,   1,  L
  1   200    66   28
  2   398   449   15
  3   183   126   30
  4   733   721   10
  6   957   838    9
     -9,   2,  L
  2   194   142   30
  6   453   468   14
     -9,   3,  L
  1   247   232   24
  2   331   330   18
  3   292   316   20
  4   627   624   11
  5   433   458   15
     -8,   0,  L
  2  1170  1131    7
  4   936   850    8
  6   186   199   25
     -8,   1,  L
  4   548   535   10
  6   707   683    9
  8   572   563   10
 10   657   628   10
     -8,   2,  L
  1   307   301   17
  2   948   916    8
  3   318   365   16
  4   905   878    8
     -8,   3,  L
  3   186   107   26
  4   452   431   12
  5   286   302   18
  6   639   639   10
  7   191   351   28
  8   447   458   13
  9   254   305   21
     -8,   4,  L
  1   550   552   11
  2   609   602   11
  3   493   506   12
  4   642   623   10
  5   312   339   17
  6   226   247   23
     -8,   5,  L
  3   219   177   23
  4   317   293   17
  5   389   420   15
  6   429   452   14
  7   444   500   13
  8   289   326   19
```

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  L   KFO    FC   SIG
     -8,   6,  L
  1   638   636   11
  2   387   347   15
  3   580   577   11
  4   321   332   18
  5   344   352   16
     -7,   0,  L
  2   673   617    8
  4  1418  1388    6
  6   895   855    7
  8   717   686    8
 10   752   742    9
 12   446   451   13
     -7,   1,  L
  1   440   388   11
  2  2399  2170    6
  3   456   414   11
  4  1052   969    7
  8   189   212   24
 10   400   366   13
 12   541   542   12
     -7,   2,  L
  2   421   402   11
  3   321   297   14
  4  1137  1149    7
  5   431   478   11
  6  1000   930    7
  7   317   340   14
  8   678   650    9
  9   202   244   23
 10   609   636   10
     -7,   3,  L
  1   988   981    7
  2  1553  1507    6
  3   830   860    8
  4   761   780    8
  5   191   218   22
 10   222   254   22
     -7,   4,  L
  1   190   303   26
  2   229   205   20
  3   444   445   11
  4   840   840    8
  5   784   836    8
  6   703   706    9
  7   646   719    9
  8   489   522   11
  9   322   382   16
 10   407   402   13
     -7,   5,  L
  1  1273  1222    7
  2   861   854    8
  3   921   927    8
  4   355   351   14
```

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  L   KFO    FC   SIG
  5   247   293   19
  9   185   173   27
     -7,   6,  L
  1   329   348   16
  3   547   530   10
  4   376   348   14
  5   907   902    8
  6   325   316   15
  7   728   756    9
  8   193   278   26
  9   411   469   14
     -7,   7,  L
  1  1225  1140    8
  2   309   290   18
  3  1005   967    8
  5   278   302   19
     -7,   8,  L
  3   466   426   13
  5   764   728    9
  7   599   601   11
     -7,   9,  L
  1   910   842    9
  3   766   718   10
     -6,   0,  L
  2  1372  1315    5
  4  1542  1490    5
  6  1608  1494    6
 10  1345  1304    7
 12  1062  1005    8
     -6,   1,  L
  4   393   365   10
  6  1253  1193    6
  7   273   276   15
  8  1820  1744    6
  9   227   306   19
 10  1083  1052    7
     -6,   2,  L
  1   505   511    9
  2  1119  1084    6
  3   668   671    7
  4  1426  1389    6
  5   553   539    8
  6  1234  1166    6
 10  1072  1006    7
 11   340   407   14
 12   897   896    8
     -6,   3,  L
  1   257   235   15
  4   212   185   17
  5   535   530    9
  6  1200  1155    6
  7  1007   960    7
  8  1459  1469    6
  9   624   735    9
```

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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
     -6,   3,  L
 10   769   795    8
     -6,   4,  L
  1   869   812    7
  2   690   698    8
  3   781   768    7
  4   863   849    7
  5   920   900    7
  6   887   828    7
  7   354   410   13
  9   378   425   13
 10   685   699    9
 11   591   681   10
 12   557   571   11
     -6,   5,  L
  1   461   437   10
  4   176   152   23
  5   740   708    8
  6   631   662    9
  7  1180  1262    7
  8   834   912    8
  9   891   948    8
 10   403   433   13
 11   199   256   26
     -6,   6,  L
  1   887   921    8
  2   298   307   15
  3   805   828    7
  4   312   336   14
  5   825   862    7
  6   355   376   14
  7   461   475   11
  9   372   375   14
 10   246   295   21
 11   731   731   10
     -6,   7,  L
  1   370   348   13
  5   739   756    8
  6   211   182   22
  7  1117  1114    8
  8   252   264   20
  9   862   873    9
     -6,   8,  L
  1   797   819    9
  3   834   822    8
  5   692   681    9
  7   284   281   18
  9   294   239   18
     -6,   9,  L
  3   185   171   27
  5   542   539   11
  7   805   780    9
     -6,  10,  L
  1   620   626   11
```

```
  L   KFO    FC   SIG
  2   279   245   20
  3   576   557   12
  4   281   246   20
  5   465   448   13
     -5,   0,  L
  2   188   158   14
  4   670   612    6
  6  1214  1197    6
  8  1423  1397    6
 10  1040  1021    7
 12   651   638    9
     -5,   1,  L
  2   797   796    6
  3   283   297   11
  4  1670  1658    5
  5   240   235   13
  6  1135  1135    6
  7   147   106   22
 10   604   616    9
 12   844   830    8
 14   936   877    8
     -5,   2,  L
  2   321   369   10
  4   543   485    7
  5   331   339   11
  6  1369  1404    6
  7   455   480    9
  8  1181  1189    6
  9   446   463   10
 10   855   873    7
 11   242   326   18
 12   554   544   10
     -5,   3,  L
  1   223   232   14
  2   710   709    6
  3   628   602    7
  4  1554  1519    5
  5   803   792    6
  6   795   766    6
  7   260   276   14
  8   161   172   23
 10   480   476   10
 11   336   411   14
 12   625   644    9
 13   398   488   14
     -5,   4,  L
  2   230   234   14
  4   327   314   11
  5   493   512    8
  6  1002   992    6
  7   883   897    7
  8   921   911    7
  9   634   757    8
 10   512   550   10
```

```
  L   KFO    FC   SIG
 11   457   494   11
 12   344   366   15
     -5,   5,  L
  1   510   494    9
  2   502   526    8
  3   655   648    7
  4   824   832    7
  5   977   963    6
  6   491   490    9
  7   461   447    9
  8   160   177   25
  9   266   320   16
 10   294   345   16
 11   478   550   11
 12   270   319   19
 13   548   580   12
     -5,   6,  L
  1   167   166   23
  4   175   238   22
  5   647   638    8
  6   615   617    8
  7   933  1019    7
  8   401   439   11
  9   832   878    8
 11   505   515   11
     -5,   7,  L
  1   700   662    8
  3   713   699    8
  4   332   334   13
  5   683   677    8
  7   335   319   13
 11   511   509   12
     -5,   8,  L
  5   621   663    9
  7   847   860    8
  9   764   765    9
     -5,   9,  L
  1   624   671    9
  3   623   646    9
  5   500   491   11
     -5,  10,  L
  3   180   163   26
  5   389   389   14
  7   620   615   11
  8   203   198   26
     -5,  11,  L
  1   420   473   14
  2   192   215   27
  3   429   474   14
  4   214   199   24
  5   334   350   17
     -4,   0,  L
  2  3209  3174    4
  4   462   436    7
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO     FC   SIG
      -4,    0,  L
 6    651    657     6
 8    203    183    16
12    719    735     8
14    978    935     8
      -4,    1,  L
 1    117     26    18
 2   1768   1775     4
 3    432    452     7
 4   2400   2386     4
 5    331    318     9
 6   1038    999     5
 8    495    508     8
10    903    920     7
11    153    156    25
12    724    698     8
      -4,    2,  L
 1    974   1004     5
 2   2402   2418     4
 3    658    662     6
 4    770    723     5
 5    147    128    18
 6    542    592     7
 7    147    183    20
10    158    105    24
12    816    809     8
13    193    333    26
14    859    809     9
      -4,    3,  L
 1    140    149    19
 2    949    939     5
 3    983   1026     5
 4   2057   2040     5
 5   1106   1126     5
 6   1086   1104     5
 7    363    383     9
 8    247    292    14
 9    203    212    17
10    631    625     8
11    317    382    14
12    592    564     9
13    232    216    20
      -4,    4,  L
 1   1411   1397     5
 2   1679   1665     5
 3    985    995     5
 4    624    633     6
 5    221    176    13
 6    139    156    21
 7    186    178    17
10    220    234    18
11    275    335    16
12    537    547    10
13    448    498    12
```

```
 L    KFO     FC   SIG
14    549    548    11
      -4,    5,  L
 1    348    350    10
 2    663    662     6
 3   1406   1402     5
 4   1103   1094     5
 5   1055   1088     6
 6    575    591     7
 7    558    601     8
 8    309    327    12
 9    399    415    11
10    347    384    13
11    469    464    10
12    340    323    14
13    295    289    18
      -4,    6,  L
 1   1767   1764     5
 2    799    814     6
 3   1043   1028     6
 4    274    270    12
 7    180    154    19
11    341    391    14
12    251    283    20
13    573    566    11
      -4,    7,  L
 3   1024   1009     6
 4    344    346    11
 5   1172   1159     6
 6    204    240    18
 7    615    638     8
 9    494    537    10
10    182    226    24
11    555    526    10
      -4,    8,  L
 1   1640   1576     6
 2    182    213    21
 3    962    988     7
 5    157     44    24
 7    209    229    19
11    332    347    16
      -4,    9,  L
 3    657    707     8
 5    694    720     8
 7    561    568    10
 9    552    571    11
      -4,   10,  L
 1    995   1060     7
 2    254    282    18
 3    635    698     9
 6    178     35    23
 9    191    195    27
      -4,   11,  L
 2    188    194    25
 3    402    394    13
```

```
 L    KFO     FC   SIG
 4    247    225    20
 5    471    457    12
 7    427    432    13
      -4,   12,  L
 1    436    454    14
 2    361    397    15
 3    396    421    15
 4    185    218    28
      -3,    0,  L
 2   2213   2211     3
 4   1507   1457     4
 6   1565   1593     5
 8   2067   2150     5
10   1024    994     6
12    212    184    19
14    428    392    12
      -3,    1,  L
 1    874    919     4
 2   2956   2947     4
 3    425    469     6
 4   1169   1119     4
 6    555    538     6
 8    576    574     7
 9    230    219    14
10   1443   1378     6
11    233    212    17
12   1017   1006     7
14    486    478    11
      -3,    2,  L
 1    291    305     8
 2    818    876     4
 3    560    601     5
 4   1949   1957     4
 5    922    997     5
 6   1934   1928     4
 7    679    739     6
 8   1609   1620     5
 9    408    434     9
10    874    806     6
12    164    150    24
13    182    105    22
14    307    290    16
      -3,    3,  L
 1   1328   1388     4
 2   1624   1659     4
 3   1006   1088     4
 4   1048   1056     4
 5    523    546     6
 6    488    486     7
 8    351    337     9
 9    561    563     7
10   1122   1084     6
11    552    637     9
12    897    888     7
```

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COLUMNS ARE 10FO,10FC,  10SIG

 L    KFO     FC   SIG
      -3,    3,  L
13    307    347    15
14    368    376    14
      -3,    4,  L
 1    115     96    18
 2    725    760     5
 3    999   1037     5
 4   1151   1162     5
 5   1423   1488     5
 6   1310   1337     5
 7   1248   1298     5
 8   1226   1242     5
 9    683    696     7
10    507    482     9
14    188    141    25
      -3,    5,  L
 1   1863   1933     4
 2    896    950     5
 3   1275   1325     5
 4    503    502     6
 5    849    838     5
 6    468    490     7
 8    234    229    14
 9    926    908     6
10    648    691     8
11    718    768     8
12    482    483    11
13    390    420    13
      -3,    6,  L
 1    145     60    18
 2    527    508     6
 3   1222   1198     5
 4    389    388     8
 5   1359   1391     5
 6    445    475     8
 7   1277   1300     6
 8    601    631     8
 9    859    823     7
10    303    340    14
13    177     90    27
      -3,    7,  L
 1   1857   1849     5
 2    267    291    11
 3   1423   1427     5
 4    159    116    18
 5    636    611     7
 7    164    153    21
 9    703    707     8
11    821    806     8
      -3,    8,  L
 2    140    140    23
 3    818    837     6
 5   1139   1105     6
 7    940    948     7
```

```
 L    KFO     FC   SIG
 9    770    803     8
11    353    370    15
      -3,    9,  L
 1   1606   1525     6
 2    372    314    11
 3   1132   1119     6
 4    268    230    15
 5    368    398    11
 7    175    171    23
 8    186    166    22
 9    545    524    10
11    588    552    11
      -3,   10,  L
 1    163    159    24
 3    500    538    10
 4    276    263    15
 5    591    604     9
 6    283    309    16
 7    780    769     8
 8    174    221    27
 9    627    618    10
      -3,   11,  L
 1    762    807     8
 2    544    574    10
 3    589    605     9
 4    327    341    14
 5    256    300    18
 8    204    221    24
      -3,   12,  L
 4    354    348    15
 5    407    429    14
 6    417    440    14
      -3,   13,  L
 1    319    313    17
 2    469    462    13
 3    277    287    20
      -2,    0,  L
 2   1670   1589     3
 4   2477   2550     4
 6   1538   1593     4
 8    845    870     5
10   1421   1393     5
12   1073   1026     6
14    947    855     7
      -2,    1,  L
 1    163    170    10
 4    470    432     5
 5    257    288     8
 6   2685   2737     4
 7    523    582     6
 8   2649   2644     4
 9    357    359     9
10    836    815     6
12    172    128    22
```

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 L    KFO     FC   SIG
14    315    290    15
      -2,    2,  L
 1    183    156     9
 2   1050    964     3
 3    841    903     4
 4   2725   2763     4
 5    800    854     4
 6   1101   1170     4
 8    754    734     5
 9    437    427     8
10   1339   1260     5
11    493    488     9
12   1010    968     6
13    290    349    15
14    741    682     9
      -2,    3,  L
 1    563    604     4
 2   1197   1255     3
 3    162    179    11
 4    715    692     4
 5   1021   1061     4
 6   2311   2378     4
 7   1324   1384     5
 8   1845   1842     5
 9    902    893     6
10    687    635     7
11    224    191    17
14    190    187    25
      -2,    4,  L
 1    499    462     5
 2    934    931     4
 3   1399   1451     4
 4   1706   1787     4
 5   1391   1459     4
 6    781    810     5
 8    739    744     6
 9    871    862     6
10    965    891     6
11    689    753     8
12    537    549     9
13    498    526    11
14    524    478    11
      -2,    5,  L
 4    212    194    11
 5    902    930     5
 6   1312   1328     5
 7   1867   1883     5
 8   1172   1162     5
 9   1170   1151     6
10    289    281    14
11    228    223    18
      -2,    6,  L
 1    700    696     5
 2    392    435     7
```

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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -2,   6,  L
  3  1448  1461    4
  4   777   806    5
  5  1548  1519    5
  6   378   380    8
  8   359   354   10
  9   997   943    6
 10   416   447   11
 11   882   894    7
 12   208   264   23
 13   629   604   10
      -2,   7,  L
  1   147   108   17
  3   414   414    8
  4   195   196   14
  5  1160  1130    5
  6   416   426    9
  7  1390  1335    5
  8   292   260   13
  9  1018   998    6
 11   398   394   13
      -2,   8,  L
  1   790   762    6
  3  1369  1366    5
  5  1054  1034    6
  8   174    76   21
  9   712   668    8
 11   900   837    8
      -2,   9,  L
  3   323   324   11
  5   772   729    7
  7  1039  1034    7
  8   205   211   21
  9   818   814    8
 11   290   308   18
      -2,  10,  L
  1   890   846    7
  2   392   363   11
  3   860   828    7
  4   259   277   15
  5   564   577    9
  8   167    82   25
  9   502   468   11
      -2,  11,  L
  3   166   168   24
  4   164   127   25
  5   493   500   11
  6   304   325   16
  7   737   717    9
  8   380   410   14
  9   487   485   12
      -2,  12,  L
  1   427   435   12
  2   391   398   13
```

```
  L   KFO    FC   SIG
  3   445   478   12
  4   347   376   14
  5   244   274   20
  7   184    61   27
      -2,  13,  L
  3   186   110   25
  4   235   242   22
      -1,   0,  L
  2   882   839    3
  4  1353  1343    3
  6  1982  1857    4
  8  1525  1466    4
 10   956   960    6
 12   487   480    9
 14   355   293   14
      -1,   1,  L
  2  2705  2677    2
  3   162   152    8
  4   226   232    7
  5   106   135   16
  6   756   796    4
  7   130    69   15
  8   143   104   17
 12  1210  1110    6
 13   175   241   24
 14  1337  1155    7
      -1,   2,  L
  1   195   235    6
  2   661   664    3
  3   514   531    4
  4  2223  2229    3
  5   623   637    4
  6  1484  1480    4
  7   426   415    6
  8   820   769    5
  9   448   425    7
 10  1020   989    6
 11   384   347   10
 12   587   537    8
 14   211   252   24
      -1,   3,  L
  1   851   858    3
  2  2323  2332    3
  3  1063  1053    3
  4  1127  1094    3
  6   611   592    5
  7   204   175   11
  8   262   232   10
  9   145    73   19
 10   284   271   12
 11   429   409   10
 12   896   840    7
 13   495   550   11
 14   891   804    8
```

```
  L   KFO    FC   SIG
      -1,   4,  L
  1   734   767    4
  2   584   596    4
  3   690   677    4
  4  1248  1245    4
  5   895   886    4
  6  1056  1048    4
  7   752   780    5
  8   573   540    6
  9   651   644    7
 10   631   600    7
 11   489   510    9
 12   462   449   10
 13   166   134   27
      -1,   5,  L
  1  1701  1671    4
  2  1298  1366    4
  3   936   948    4
  4   598   612    5
  5   298   260    8
  6   144   136   16
  7   155   154   17
  8   340   320   10
  9   326   329   11
 10   280   244   13
 11   505   533    9
 12   416   460   12
 13   749   719    9
 14   491   484   12
      -1,   6,  L
  1   798   798    4
  2   395   384    6
  3  1123  1134    4
  4   771   830    5
  5  1029  1015    5
  6   529   548    7
  7   830   818    6
  8   241   244   13
  9   690   659    7
 10   250   260   16
 11   586   564    9
 12   190   193   24
 13   187   148   25
      -1,   7,  L
  1  1137  1099    4
  2   542   540    6
  3  1040  1010    5
  4   251   260   11
  5   308   283   10
  6   161   121   18
  9   258   254   15
 11   694   692    8
 13   776   724    9
      -1,   8,  L
```

```
COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
      -1,   8,  L
  1   397   420    8
  3  1115  1088    5
  4   305   313   10
  5  1047  1039    6
  6   172   145   19
  7   606   596    8
  9   689   640    8
 11   542   499   10
      -1,   9,  L
  1  1203  1180    5
  3   748   722    6
  9   232   230   19
 11   540   526   11
      -1,  10,  L
  1   374   378   11
  2   168   172   21
  3   622   592    8
  5   591   625    9
  7   647   637    8
  8   176   270   26
  9   491   472   11
      -1,  11,  L
  1   707   743    8
  2   241   288   17
  3   492   551   10
  5   184   195   22
  9   277   258   18
      -1,  12,  L
  2   221   193   19
  3   309   315   15
  5   430   436   12
  6   298   311   16
  7   338   353   16
      -1,  13,  L
  1   331   321   15
  2   292   342   17
  3   312   328   16
  5   180   122   26
       0,   0,  L
  2  3058  3134    2
  4  2267  2181    3
  6   838   823    4
  8   235   214   11
 10   827   788    6
 12  1141  1027    6
 14   719   641    9
       0,   1,  L
  1   238   251    3
  2  2386  2476    2
  3   388   401    4
  4  2218  2205    3
  5   393   361    5
  6  1626  1457    4
```

```
  L   KFO    FC   SIG
  7   328   295    7
  8  1516  1426    4
  9   170   192   17
 10   859   838    6
 14   477   416   11
       0,   2,  L
  0  4726  4884    2
  1  1684  1723    2
  2  2862  2831    2
  3   551   541    4
  4   349   405    5
  5   352   360    6
  6   764   740    4
  7   331   321    7
  8   213   211   12
  9   252   235   12
 10   914   857    6
 11   339   315   12
 12  1037   952    6
 13   178   251   25
 14   542   497   10
       0,   3,  L
  1   688   684    3
  2  2285  2370    3
  3  1758  1754    3
  4  2533  2472    3
  5  1240  1237    4
  6  1053  1007    4
  7   477   477    6
  8   851   773    5
  9   443   420    8
 10   744   693    6
 11   163   208   24
 14   286   275   18
       0,   4,  L
  0  2750  2795    3
  1  2368  2407    3
  2  1543  1555    3
  3   690   701    4
  4   197   139    9
  5   163   152   12
  7   315   342    9
  8   343   333    9
  9   575   562    7
 10   745   701    7
 11   529   603   10
 12   667   680    9
 13   359   420   14
 14   350   358   15
       0,   5,  L
  1  1129  1080    4
  2  1535  1550    4
  3  1943  1948    4
  4  1063  1084    4
```

```
  L   KFO    FC   SIG
  5  1487  1462    4
  6   705   669    5
  7   928   890    5
  8   634   592    6
  9   436   406    9
 10   378   341   11
 11   208   203   19
       0,   6,  L
  0  1251  1281    4
  1  2327  2378    4
  2   663   655    5
  3   773   757    5
  6   136    76   20
  7   333   319    9
  9   650   637    7
 10   366   408   12
 11   685   704    8
 12   347   373   15
 13   527   518   11
       0,   7,  L
  1   931   917    5
  2   415   424    7
  3  1971  1949    4
  4   289   304   10
  5  1355  1365    5
  7   659   649    7
  8   299   309   12
  9   552   576    9
 10   190   239   22
 11   310   301   15
       0,   8,  L
  1  1961  1883    5
  3   893   825    5
  4   137    41   20
  7   261   247   14
  9   443   439   10
 11   704   676    9
       0,   9,  L
  1   601   592    7
  2   135    58   23
  3  1207  1205    5
  4   296   274   12
  5   995   998    6
  6   229   202   16
  7   707   699    8
  9   527   492   10
       0,  10,  L
  0   624   600    7
  1  1345  1275    6
  2   368   335   10
  3   460   458    9
  6   174   104   22
  9   379   359   13
       0,  11,  L
```

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COLUMNS ARE 10FO,10FC,  10SIG                                        COLUMNS ARE 10FO,10FC,  10SIG
  L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG     L   KFO    FC  SIG
     0, 11, L           12   438   400   11     1  1287  1283    4        2, 0, L             7   301   323   10        2, 10, L
  1   305   336   14    14   299   272   17     2   156   167   15     0  1286  1150    3     8   668   675    7     0   240   213   17
  2   239   242   17        1, 3, L             3  1573  1559    4     2  1385  1343    3     9   622   627    8     1   675   618    7
  3   628   674    8     0  1124  1051    3     4   230   247   13     4  1535  1572    4    10   438   447   10     3   407   405   10
  4   397   425   11     1   897   873    3     5   781   753    6     6   218   248   11    11   512   571   10     5   254   233   17
  5   629   644    9     2  1731  1641    3     7   435   403    9     8   846   853    6    12   503   519   12     6   179   152   24
  6   392   412   12     3  1525  1574    3     8   189   179   20    10   941   942    6    13   384   472   16     7   193   112   22
  7   500   499   11     4  1887  1949    3     9  1038  1000    6    12  1062   998    7        2, 5, L             9   375   351   15
  8   175   187   26     5   709   729    5    10   240   282   19        2, 1, L             0   126   118   15        2, 11, L
  9   273   276   19     6   290   278    8    11   848   817    8     2   444   381    5     1   217   178    9     1   160    91   24
     0, 12, L            7   441   430    7    12   172   120   28     4  1670  1726    4     2   117    97   18     3   401   409   11
  0   514   560   10     8  1219  1190    5        1, 8, L             5   439   446    6     3   633   604    5     4   248   237   17
  1   520   529   10     9   935   909    6     0   322   324    9     6  2008  1997    4     4   924   919    5     5   645   656    9
  2   453   449   11    10  1361  1250    6     1   198   205   14     7   376   382    8     5  1245  1257    5     6   337   374   15
  3   360   403   13    12   572   587   10     3   953   927    5     8  1069  1059    5     6  1132  1125    5     7   539   543   11
     0, 13, L               1, 4, L             5  1370  1300    5     9   164   145   19     7  1136  1120    5        2, 12, L
  2   173   237   28     0   157   167   10     7  1222  1152    6    10   572   557    8     8   444   463    9     0   303   301   16
  3   269   287   18     1   368   373    5     9   662   649    8    12   266   239   17     9   510   531    9     1   410   419   12
  4   439   451   13     3   827   826    4        1, 9, L                2, 2, L            11   250   261   19     2   247   259   18
  5   322   325   17     4  1078  1056    4     0   264   193   12     0   412   345    5    12   197   118   24     3   342   338   15
     1, 0, L             5  1698  1686    4     1  1161  1081    5     1   499   504    4        2, 6, L                2, 13, L
  0   174   157    5     6  2026  2037    4     2   278   230   12     2  2479  2475    3     0   317   319    8     3   203   224   25
  2   449   387    4     7  1577  1559    5     3   889   831    6     3   782   785    4     1   762   770    5        3, 0, L
  4   994   989    3     8  1308  1267    5     5   485   461    9     4  1551  1537    4     2   636   656    5     0   705   673    4
  6  3114  3102    4     9   576   562    8     9   694   660    9     5   216   211   10     3  1162  1107    5     2  3290  3357    4
  8  2364  2318    4    12   195   222   23        1, 10, L            6   358   358    8     4   513   507    7     4  1999  1978    4
 10   236   216   15        1, 5, L             0   312   279   12     7   173   181   15     5   675   630    6     6   517   500    7
 12   496   415   10     0  1051  1036    4     2   146    65   23     8   801   772    6     7   324   294   11     8   550   573    8
 14   337   310   15     1  1257  1227    4     3   594   588    8     9   276   270   12     8   353   337   12    10   760   766    7
     1, 1, L             2   433   457    5     4   378   387   11    10   771   763    7     9   653   655    8    12   171   121   27
  0  3439  3301    2     3  1207  1276    4     5   928   952    7    11   335   372   13    11   659   675    9        3, 1, L
  1   512   557    3     4   935   981    4     6   379   394   12    12   849   824    8    12   196   233   25     0  3874  3698    4
  2  2104  2052    3     5  1145  1139    4     7   929   902    7    13   237   326   22        2, 7, L             1   350   360    6
  3   439   473    4     6   495   465    6     8   188   194   25        2, 3, L             0   144   151   18     2   973   974    4
  4  1925  2010    3     7   434   422    8     9   350   358   15     0   101    28   15     1   246   262   11     4   724   805    5
  5   272   273    7     8   713   723    7        1, 11, L            1   109   107   14     3   848   824    5     5   130   108   19
  6   557   619    5     9  1251  1195    6     0   429   438   10     2   332   390    6     4   475   487    7     6   317   322    9
  7   193   206   12    10   694   770    9     1   590   616    9     3   338   320    6     5  1199  1177    5    10   715   762    8
  8  1523  1524    4    11   689   772    9     2   384   394   11     4  1514  1511    4     6   345   332   11    12  1003   982    8
  9   323   309   10    12   178   352   29     3   464   457   10     5   904   915    5     7   960   940    6        3, 2, L
 10  1741  1654    5    13   319   383   17     4   231   234   18     6  1642  1642    4     9   619   612    9     0   871   867    4
 11   164   210   23        1, 6, L             5   292   286   15     7   813   821    5    11   258   227   20     1   622   661    4
 12   799   741    7     0   247   253    9     8   177   176   28     8   866   833    6        2, 8, L             2  2533  2559    4
 14   386   350   13     1   250   205    9        1, 12, L            9   533   532    8     1   814   789    6     3   763   774    5
     1, 2, L             2   293   280    8     3   328   332   14    10   478   482    9     2   186   186   16     4  1849  1883    4
  3   385   350    4     3   974   960    4     4   432   441   12    12   197   161   24     3   882   866    6     5   442   453    7
  4  1578  1549    3     4   391   382    7     5   522   557   11        2, 4, L             5   305   298   12     6   319   311    9
  5   942   955    4     5  1735  1662    4     6   477   491   12     0   614   585    5     6   177   137   20     7   191   196   16
  6  2540  2572    4     6   796   799    6        1, 13, L            1   780   743    4     9   493   472   10     8   533   542    8
  7   938   946    5     7  1628  1577    5     0   411   387   12     2  1358  1375    4        2, 9, L             9   270   273   14
  8  1962  1903    4     8   621   617    7     1   297   290   16     3  1159  1188    4     3   742   707    7    10   790   767    7
  9   442   426    8     9   563   612    8     2   477   432   11     4  1050  1104    4     5   952   993    7        3, 3, L
 10   348   316   11        1, 7, L             3   253   265   20     5   535   560    6     7   827   793    7     0  2407  2411    4
 11   158    59   24     0   259   301   10        2, 0, L             6   208   262   13     9   433   423   12     1  1286  1312    4
```

```
COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
       3,   3,  L
  2  1564  1593    4
  3   256   253    9
  4   323   337    8
  5   361   391    8
  6   548   562    7
  7   192   196   16
  8   170   180   20
  9   243   253   15
 10   560   584    9
 11   379   406   13
 12   704   730    9
       3,   4,  L
  0   574   617    6
  1  1359  1377    4
  2  1665  1672    4
  3  1470  1461    4
  4  1224  1220    5
  5   554   586    7
  6   377   386    9
  7   288   315   12
  8   371   373   11
  9   424   454   10
 10   523   516   10
 11   314   277   15
       3,   5,  L
  0  1490  1498    4
  1  1341  1305    4
  2   708   733    5
  3   268   257   10
  6   239   260   14
  7   316   334   12
  8   205   222   18
  9   385   445   12
 10   322   340   15
 11   491   510   11
       3,   6,  L
  1  1441  1353    5
  2   809   819    5
  3  1507  1495    5
  4   536   555    7
  5   787   810    6
  6   220   234   15
  7   437   457   10
  8   282   319   15
  9   470   487   11
 10   278   248   17
 11   286   273   19
       3,   7,  L
  0   550   547    7
  1  1457  1375    5
  2   364   343    9
  3   270   211   12
  4   162   132   20
```

```
  L   KFO    FC   SIG
  5   387   365   10
  7   186   208   21
  9   279   320   17
       3,   8,  L
  0   199   175   17
  1   840   794    6
  3  1308  1246    6
  4   158   135   22
  5   769   813    7
  7   508   516   10
  9   483   482   11
       3,   9,  L
  1  1071   978    6
  5   154   189   26
  9   272   278   19
       3,  10,  L
  1   552   549    9
  2   266   234   15
  3   757   775    8
  5   626   638    9
  7   473   461   12
       3,  11,  L
  0   419   418   11
  1   642   633    9
  3   250   222   18
       3,  12,  L
  0   176   152   27
  2   192   256   26
  3   408   412   13
  4   315   341   17
       3,  13,  L
  0   418   395   14
  1   334   309   16
       4,   0,  L
  0  2896  2856    4
  2   777   778    5
  4   538   539    7
  8  1123  1121    6
 10  1122  1166    7
       4,   1,  L
  0  1101  1054    4
  1   286   292    9
  2  2057  2036    4
  3   288   317   10
  4  1427  1430    5
  5   335   373   10
  6  1409  1445    5
  7   208   218   17
  8  1046  1046    6
       4,   2,  L
  0  2188  2177    4
  1   653   709    5
  2  1170  1152    5
  3   406   405    8
```

```
  L   KFO    FC   SIG
  4   481   443    7
  7   322   305   12
  8   915   922    7
  9   373   400   12
 10   988  1041    7
 11   244   284   20
       4,   3,  L
  0   752   715    5
  1   720   692    5
  2  1439  1423    5
  3  1006  1000    5
  4  1255  1316    5
  5   798   832    6
  6  1084  1132    6
  7   610   638    8
  8   753   771    7
  9   246   235   17
       4,   4,  L
  0  1377  1323    5
  1  1181  1209    5
  2   681   699    6
  3   709   734    6
  4   522   536    7
  7   564   584    8
  8   706   745    8
  9   645   708    9
 10   716   720    9
 11   326   397   17
       4,   5,  L
  0   637   625    6
  1  1121  1089    5
  2   813   794    6
  3  1205  1185    5
  4   536   534    8
  5  1044  1045    6
  6   720   733    7
  7   738   831    7
  8   511   540   10
  9   302   292   16
       4,   6,  L
  0   783   786    7
  1  1401  1363    5
  2   277   284   12
  3   686   690    7
  7   597   636    9
  8   308   340   15
  9   673   712    9
 10   332   361   17
       4,   7,  L
  0   285   257   12
  1   743   723    7
  2   163   161   21
  3  1066  1031    6
  5   986  1005    6
```

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COLUMNS ARE 10FO,10FC,  10SIG

  L   KFO    FC   SIG
       4,   7,  L
  6   203   246   21
  7   858   848    7
  8   328   309   15
  9   398   406   14
       4,   8,  L
  1  1376  1253    6
  2   157   120   24
  3   426   448   10
  5   172   155   23
  7   389   413   13
       4,   9,  L
  1   539   550    9
  2   188   209   22
  3   782   780    8
  4   219   207   20
  5   797   791    8
  6   170    61   27
  7   681   676   10
       4,  10,  L
  0   349   322   13
  1   740   730    8
  2   268   250   17
  3   312   290   15
       4,  11,  L
  1   343   320   15
  2   262   260   19
  3   501   495   11
  4   408   379   14
  5   553   537   11
       4,  12,  L
  0   434   419   13
  1   415   379   14
  2   343   315   16
       5,   0,  L
  0   256   225   11
  2   433   410    8
  4  1465  1488    6
  6  1807  1811    6
  8   679   668    9
       5,   1,  L
  0   622   602    6
  1   149    99   19
  2  1536  1466    5
  3   325   327   10
  4  1190  1232    6
  5   155   135   23
  6   340   301   13
  7   160   148   24
  8  1087  1072    7
 10   758   787    9
       5,   2,  L
  0   179   196   17
  1   155   105   18
```

```
  L   KFO    FC   SIG
  2   223   195   14
  3   344   363   10
  4  1496  1554    5
  5   704   742    7
  6  1621  1623    6
  7   429   465   11
  8   606   602    9
       5,   3,  L
  0   404   400    8
  1   667   674    6
  2  1307  1278    5
  3   794   749    6
  4   957   925    6
  5   155   126   24
  6   361   358   12
  7   347   383   13
  8   823   832    8
  9   412   455   12
 10   533   575   11
       5,   4,  L
  3   535   524    8
  4  1065  1074    6
  5  1123  1086    6
  6  1110  1173    7
  7   793   841    8
  8   387   378   13
  9   197   193   24
       5,   5,  L
  0   347   331   10
  1   948   923    6
  2   722   682    7
  3  1005  1015    6
  4   614   603    8
  6   216   232   20
  7   603   637    9
  8   540   567   10
  9   550   597   11
       5,   6,  L
  0   184   120   21
  1   152   160   23
  3   572   553    8
  4   405   418   11
  5  1069  1096    7
  6   516   546   10
  7   851   872    8
  8   231   228   21
       5,   7,  L
  1  1055   992    6
  3   871   841    7
  6   166    78   26
  7   502   521   11
  8   196   193   26
       5,   8,  L
  0   163    49   25
```

```
  L   KFO    FC   SIG
  1   165   198   24
  3   570   581    9
  5   880   858    8
  7   740   707    9
       5,   9,  L
  1   693   704    8
  3   553   532   10
       5,  10,  L
  1   212   219   22
  3   434   434   13
  4   180   167   28
       5,  11,  L
  0   269   232   19
  1   436   420   13
  2   229   219   23
       6,   0,  L
  0  1374  1278    6
  2   531   537    8
  4   205   159   20
  6   164   234   27
       6,   1,  L
  0   468   455    9
  2  1335  1343    6
  3   244   222   16
  4  1270  1227    6
  5   171   215   24
  6   552   544    9
  8   306   365   17
       6,   2,  L
  0  1286  1194    6
  1   240   230   15
  2   684   706    7
  6   307   318   15
  8   195   199   24
  9   188   137   26
       6,   3,  L
  0   262   221   14
  1   322   316   12
  2   942   940    6
  3   552   556    9
  4   994  1038    7
  5   441   472   11
  6   424   438   11
  7   274   325   18
  8   248   300   20
       6,   4,  L
  0   967   874    7
  1   726   700    7
  2   668   649    8
  4   172    72   23
  5   181   214   24
  6   354   369   13
  8   204   225   24
       6,   5,  L
```

```
COLUMNS ARE 10FO,10FC,  10SIG

L   KFO     FC   SIG      L   KFO     FC   SIG      L   KFO     FC   SIG
     6,   5,  L           4   365    341    13           7,   8,  L
0   248    226    16      6   195    116    24      1   749    712     9
1   635    624     8           7,   2,  L                8,   0,  L
2   563    562     9      0   950    923     7      0   814    782     8
3   572    611     9      1   407    428    12      2   602    557    11
4   548    591     9      2  1296   1325     7      4   322    282    18
5   521    541    10      3   376    396    13           8,   1,  L
6   303    321    16      4   707    737     9      0   302    308    17
7   428    427    12      5   252    279    19      2   488    496    12
     6,   6,  L           6   456    483    12      4   954    935     9
0   551    522    10      7   177    201    29           8,   2,  L
1   761    756     8           7,   3,  L           0   621    638    10
2   398    348    11      0  1183   1168     7      1   272    275    19
3   206    230    21      1   419    449    12      2   581    561    11
7   257    293    20      2   192    229    23      3   261    209    20
     6,   7,  L           4   198    204    24      4   218    256    26
1   586    603     9           7,   4,  L                8,   3,  L
2   277    267    16      0   735    741     9      0   259    230    19
3   703    712     9      1   785    775     8      2   418    430    13
4   304    262    15      2   849    855     8      3   415    445    14
5   521    523    11      3   640    635     9      4   765    780    10
     6,   8,  L           4   432    474    12           8,   4,  L
1   643    641     9      5   394    427    13      0   418    396    14
3   190    171    25      6   307    349    17      1   518    511    11
     6,   9,  L                7,   5,  L           2   432    430    13
1   540    515    11      0   770    736     8      3   333    291    17
3   622    577    10      1   643    650     9           8,   5,  L
     6,  10,  L                7,   6,  L           0   216    220    24
1   477    459    12      0   360    334    15      1   299    286    18
     7,   0,  L           1   890    863     8      2   218    225    24
0  1245   1165     7      2   407    381    13           8,   6,  L
2  1507   1457     6      3   727    706     9      0   197    188    29
4   782    775     8      4   195    199    25      1   572    559    11
6   563    599    10           7,   7,  L                9,   1,  L
     7,   1,  L           0   307    270    16      0   594    552    11
0  1538   1532     6      1   608    602    10
```

OBSERVED AND CALCULATED STRUCTURE FACTOR LISTINGS FOR $Os_3(CO)_{10}Cl_2$

```
COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG
    H,  0,  0            20  3441  3237    69      8  6941  6534    49
 4 15032 14342    25     22  3429  3293    79     10  1106  1105    67
 6  7695  6937    30     26  2502  2513    92     12  6700  6386    53
 8  2292  2179    38         H,  5,  0            14  1973  2041    69
10 20325 19770    43      2  9078  8630    32     16  8428  7977    63
12  4577  4162    47      4  2568  2376    36     22  1828  1867    99
14  9520  8718    50      6  7128  6625    36     24  2475  2567    95
16  2870  2751    60      8  2109  1961    44         H, 10,  0
18  4122  4091    60     10  1526  1548    53      0  7038  6990    51
20  3695  3612    70     12 11529 10658    48      2  1329  1324    60
22  2237  2488    85     16  8756  8271    57      4  5450  5388    48
24  3104  3149    86     18  4165  3954    62      6  9753  9452    47
26  1677  1648   103     20  1601  1420    90      8  3393  3032    53
    H,  1,  0            22  6344  6246    73     10  6350  6123    51
 4   606   432    41     26  4061  4399    84     14   758   563   117
 8 10899  9840    37         H,  6,  0            16  4584  4396    67
12  8634  7972    45      0   930   933    54     18  2686  2560    72
14  2045  1948    57      4  6530  6088    36     20  2532  2506    83
18  7186  6806    59      6  6982  6593    38     22  1522  1473   119
22  6994  6930    90      8  3847  3453    44     24  1506  1505   124
    H,  2,  0            10   561   253    89         H, 11,  0
 2  1685  1499    26     12  1415  1421    62      2  3891  3905    54
 4  4315  3661    27     14  2103  2025    59      6  2100  1942    61
 6  1552  1337    36     18  5963  5800    61      8  6868  6651    53
 8  4080  3678    38     20  2803  2667    75     10  2578  2440    58
10  2750  2653    43     24  4121  4050    81     16  4206  3947    69
12  1348  1322    62         H,  7,  0            18  2525  2574    84
18  5216  5045    61      2  2737  2619    42     20  1475  1368   105
20  2097  2213    83      4  6223  5795    38         H, 12,  0
22  1711  1899   106      6  5835  5516    41      0  1948  1960    61
24  2632  2561    83      8  2541  2410    47      2  3779  3871    56
    H,  3,  0            10  4700  4322    48      4  6933  6814    53
 2  5646  5033    25     14  1189  1195    78      6 11424 11481    54
 4  3242  2668    30     16  1390  1305    89      8  5687  5515    55
 6  1188  1182    44     20  2888  2863    74     10  2916  2759    60
 8  4826  4387    39     24  1586  1802   116     12  1044   947    95
10  4842  4467    42         H,  8,  0            16  5806  5674    67
14   750   780    87      0  1423  1440    57     18  3128  3226    82
16  3229  3074    60      2 13023 12813    43     20  2549  2503    86
18  5313  4978    61      4  6294  6112    43     24  1729  1547   115
20  3635  3390    70      6 12200 11768    43         H, 13,  0
22  2765  2829    82      8  7994  7715    46      2  2222  2106    64
24  2073  2163    99     12  9624  8601    52      6  4825  4719    57
26  3063  3097    88     14  2935  2744    62     10  2433  2352    65
    H,  4,  0            16 11311 10644    61     12  2811  2699    66
 2  7712  7154    28     18  2483  2528    72     14  4035  3710    67
 4  9909  9115    31     20  2441  2547    82     16  1776  1796    91
 6  8769  7995    34     22  2924  2800    79     18  3726  3674    78
 8  8202  7448    39     24  2476  2747   100     22  1075  1315   147
10 12590 11914    42     26  2612  2728    98         H, 14,  0
12  8154  7561    47         H,  9,  0             0  7486  7579    58
14  4844  4366    52      2  9975  9631    46      2  3036  3203    61
16  6101  5730    58      4  3937  3843    46      4  1684  1599    72
18   986  1062   111      6 12340 11966    45      6  5183  5051    59
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG
    H, 14,  0                H, 21,  0             1  2130  2224    35
 8  3879  3718    62      4  1462  1651   120      3   687   699    63
10  8294  7959    61      8  2415  2087   101      4  4275  4211    33
12  3516  3559    68     12  2436  2366    97      5  2613  2412    37
16  4602  4732    72     14  2624  2426   107      7  2429  2285    41
18  5629  5586    77         H, 22,  0             8  3486  3210    41
20  3627  3539    82      0  3771  3730    82      9  5925  5500    41
22   801   390   173      4  3718  3476    85     10  7492  7210    42
    H, 15,  0             6  2668  2519    93     11  4684  4304    46
 4  5630  5194    63      8  2093  2155   110     12  4506  4363    49
 6  5521  5325    61     10  1558  1432   132     13  1357  1326    67
10  5011  4916    66         H, 23,  0            14  7305  7297    51
14  1260  1220   112      2  2687  2603   100     15  4808  4389    55
18  1259  1181   126      4  1837  1833   107     19  3631  3620    67
20  3018  3160    94         H, 24,  0            20  5797  5578    65
    H, 16,  0             0  1578  1692   119     22  2884  2969    86
 0  7801  7644    62      2  4554  4295    83     24  4345  4313    82
 2  2511  2381    71      4  1605  1470   122         H,  4,  1
 4  5478  5557    65         H,  1,  1             0  4229  4384    33
 6  2755  2816    69      2  2963  3030    30      3  3111  3133    39
10  6267  6147    66      3  1054  1103    40      4  2883  2874    37
12  2067  2211    82      4  6227  5956    28      6  4694  4495    38
14  5131  5304    75      6  5627  5226    33      7  4437  4310    40
18  2796  2665    89      7  1919  1773    41      8   748   824    76
20  2212  2354   107      8  1175  1073    49      9  2400  2194    46
    H, 17,  0             9  3763  3369    41     11  1867  1788    53
 2  2096  2051    82     10  8465  7761    40     12  3303  3181    50
 6   791   754   139     11  2599  2308    46     14  1075   858    77
 8  6384  6208    69     12   904   899    72     16  4472  4231    58
12  3313  3294    77     13   764   790    90     17  1049  1012    92
18  4504  4383    86     14  3218  2996    53     20  4933  4857    65
    H, 18,  0            15  2404  2322    60     22  4983  5103    75
 0  7375  7110    65     16   703   436   113         H,  5,  1
 2  2538  2675    75     20  6549  6353    65      1  2703  2813    42
 4  2342  2258    82     22  1465  1484   109      2  3989  4070    39
 6  1913  1860    84         H,  2,  1             3   921   995    61
10  4099  3898    81      0  1490  1536    30      6  1565  1496    46
12  3554  3602    81      1  3140  3204    29      7  3339  3133    42
16  1005  1075   154      3  4573  4543    32      8  6582  6138    43
18  1091  1184   181      5  1865  1863    37      9  1733  1683    52
    H, 19,  0             7  2212  2125    41     10  1229  1167    60
 2  3169  3258    74      8 12077 11365    39     11   711   560    82
 4  4027  3904    74     10   900   806    62     12  1976  1881    54
 6  4942  4847    76     11  2405  2269    50     13  1850  1761    61
 8  2446  2419    89     12 14366 13925    46     14  2878  2763    59
12  3469  3620    86     13  1377  1394    64     17  1841  1706    71
16  3687  3696    86     14  3754  3434    54     18  1063  1005   112
    H, 20,  0            17  1517  1591    75     20  2157  2060    76
 0  3871  3899    76     18  2910  2855    66     22  2757  2942    81
 2  1400  1232   106     19  2721  2545    70     24  2188  2132   100
 6  1282  1228   124     22  3473  3704    77         H,  6,  1
10  3682  3502    88     24  4219  4110    81      0  6560  6940    38
12  1806  1904   114     27  2485  2483    98      2  2259  2194    46
14  3949  3786    88         H,  3,  1             4  8039  8055    41
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COLUMNS ARE 10FO,10FC,  10SIG                                                     COLUMNS ARE 10FO,10FC,  10SIG

 H    KFO     FC   SIG     H    KFO     FC   SIG     H    KFO     FC   SIG         H    KFO     FC   SIG     H    KFO     FC   SIG     H    KFO     FC   SIG
     H,  6,  1             3   3257   3316    55    12   4050   3929    63             H, 16,  1            11   1342   1099   143    12   3155   3064    50
 6   4989   4699    42     4   5520   5589    50    13   2254   2150    70         5   1455   1418    93    12   2108   2062   109    13   4179   3985    52
 8   6611   6200    44     6   5057   4880    50    14   3603   3616    67         6   3399   3250    73    14   4399   4181    92    14   3617   3508    55
 9   2021   1916    50     8   3135   3091    53    16   4824   4662    70         8   3626   3526    73         H, 22,  1            15   4222   3873    56
10   5989   5847    46    10   2926   2934    55    18   4534   4575    74         9   1784   1866    89     0   4602   4827    78    16   2422   2389    66
11   1429   1467    60    14   3064   2958    66    20   3457   3486    84        10    839    626   124     3   1585   1493   101    17   5089   4711    60
13   1297   1220    75    17   1102    965   112    22   2259   2178    99        12   3400   3521    76     5   1467   1521   109    18   2136   2228    74
14   9454   8894    54    18   2153   2158    78         H, 13,  1                13   1809   1675   103     7   2030   1895   103    20   2795   2685    71
15   1536   1439    77    20   1896   1978    88     1   2336   2363    63        14   3611   3450    78     9   1217   1102   155    21   2699   2683    74
18   6023   5942    62         H, 10,  1             2   3487   3534    63        16   4036   3889    81    10   3569   3308    94    22   2591   2538    87
20   2364   2287    76     0   5387   5704    52     4   2449   2405    63        18   1774   1672   112    12   1716   1956   144    24   1948   1972    95
21   1641   1695    95     2   1244   1223    69     7   1636   1471    76             H, 17,  1                 H, 23,  1                 H,  2,  2
24   3878   3791    87     4  10036  10362    52     8   4517   4304    63         1   1589   1625    92     2   1631   1549   108     0   4117   4223    30
25   1547   1606   134     5   3014   3131    58     9   3102   2891    65         2   4250   4410    67     4   1832   1585   102     2    579    547    64
26   1102    850   172     6   7368   7218    51    10   2544   2400    65         3   1540   1624    97     6   1677   1445   112     3   1207   1247    50
     H,  7,  1             7    861    883   102    11    970    980   111         4   4445   4273    65     8   1895   1643   116     4   1768   1972    46
 1    863    888    71     8   6248   5986    54    12    807    816   132         6   5231   5154    71         H, 24,  1             5   4326   4498    42
 2   2694   3041    46     9   1616   1561    65    13   1925   1775    81         8   4996   4820    74     0   1586   1616   122     6   2514   2440    42
 3   1060   1185    70    10   3419   3439    55    14   4179   4038    70         9   2003   1801    89     2   1994   1848    97     7   3746   3876    42
 4  14026  14569    43    11   2467   2317    62    17   1398   1467   107        12   1291   1360   128     3   1331   1142   120     8   3047   2911    44
 5   2788   2782    49    12   3921   3824    59    20   2373   2422    93        13   1456   1411   126         H,  0,  2             9   2572   2495    48
 6   7045   6845    44    14   6829   6435    62    21   1596   1811   117        14   1619   1374   105     0  16186  18953    29    10   4409   4120    45
 8   1418   1321    57    15   1365   1529    90    22   1916   2059   108        16   2059   1974    97     1   5060   4889    29    11   2070   2090    55
 9   2968   2911    51    18   6334   6329    68         H, 14,  1                17   1310   1311   136     2   7890   8051    29    14   1612   1445    63
11    961    903    78    20   2524   2660    81     0   2094   2223    70        18   1835   1664   109     3   3662   3629    36    18   3451   3353    63
12   2717   2856    55    21   2193   2327    92     1   1724   1630    72             H, 18,  1             4   1968   1755    43    19   1550   1532    85
13   4431   4165    56    22   1331   1564   132     2   5424   5686    58         1   1701   1613    84     5   2286   2216    42    20   1967   2071    85
14  10491   9815    56    24   1350   1431   139     4   3309   3315    64         2   5465   5793    67     7   6323   6109    40    24   2196   2385   103
15   3332   3140    64    25   2348   2353   108     6   4810   4579    64         3    880   1088   143     8  10746  11075    40         H,  3,  2
17   2818   2676    67         H, 11,  1             8   6593   6372    63         5   2821   2723    80     9   2990   2876    45     2   4300   4482    34
18   1728   1653    81     2   1734   1905    64    10   1402   1410    87         8   2740   2563    84    10  13613  13315    43     3   3519   3632    42
19   1372   1542    99     3   2232   2169    61    11   1620   1555    85         9   2463   2372    91    12   3352   3291    49     4   2298   2334    41
20   1058    986   112     4   8187   8410    54    12   3187   3156    72        12   5998   5958    78    14   5990   5721    52     6    657    615    78
21   1451   1662   105     5   1405   1474    74    13   1500   1413    99        14    966    801   165    15   2903   2728    57     7   3663   3542    43
22   2097   2282    96     6   5282   5104    57    16   2880   2989    80        16   2768   2769    95    16   1287   1157    81     8   6828   6478    43
24   3928   4005    85     8   2669   2491    63    18   3349   3229    85             H, 19,  1            17   4503   4259    61     9   3946   3863    46
26   2216   2037   102     9   1755   1746    72    22   1376   1213   136         2   1826   1923    90    18   3737   3587    63    10   2162   2056    51
     H,  8,  1            10   2023   1977    64         H, 15,  1                 4   2318   2405    83    19   3713   3517    65    11   1647   1474    57
 0   2583   2761    47    12   1235   1234    89     3    979    796   113         6   1561   1518   113    20   3752   3937    69    12   1205   1043    62
 1   3453   3530    52    13   3816   3878    64     5   1536   1507    88         9   1073    989   145    21   2782   2779    79    14   1911   1787    62
 2   3372   3374    47    14   5018   4752    65     6   2618   2416    77        10   4650   4620    84    22   2618   2617    86    16   4964   4811    60
 3   5078   5007    50    16   1939   1943    86     9   2301   2402    76        12   1875   1832   108    26   2729   2943   100    18   2585   2560    70
 4   2301   2282    50    17   2810   2705    78    10   3430   3383    69        15   1298   1227   131         H,  1,  2            19   1702   1739    80
 5   2250   2143    52    21   2167   2049    93    13   1854   1959    88             H, 20,  1             1   2793   2749    31    21   1850   1928    83
 6   3297   3145    48         H, 12,  1            16   2299   2370    94         1   1365   1390   109     2   1671   1700    34    23   1034    971   138
 7   3009   2980    50     1   3324   3402    58    17   1351   1407   128         2   3169   3131    81     3   1642   1763    42    26   3804   4028    93
 8   5034   4755    49     2   5170   5250    54    19   1609   1360   109         3   2416   2473    85     4   2141   2179    40         H,  4,  2
 9   1828   1713    56     3   2437   2530    67    20   3545   3542    92         7   2412   2390    93     5   2086   2107    46     0   5957   5931    34
10   2543   2431    54     4   1529   1449    70    21   1267   1228   143         8   1651   1558   111     6   1688   1568    47     1   1548   1529    43
16   1281   1110    89     5   2864   2971    65         H, 16,  1                10   1460   1498   132     7   6066   5863    41     2   1977   2027    40
18   2605   2543    73     6   3635   3518    59     0   2926   2741    69        11   1301    986   137     8   4795   4619    42     3   1888   2032    48
22   1886   1935   107     8   7396   7136    58     1   2391   2372    72             H, 21,  1             9   1876   1832    52     4   1403   1560    48
     H,  9,  1             9   3902   3714    62     2   4539   4445    69         4   3890   3960    78    10   3965   3880    45     5    854    862    68
 1   1896   1963    58    10   2686   2584    63     3   2384   2458    74         6   2011   2053   103    11   1067   1069    65     6   4289   4498    45
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        COLUMNS ARE 10FO,10FC,  10SIG

  H    KFO     FC    SIG      H    KFO     FC    SIG      H    KFO     FC    SIG
       H,  4,  2             25   2016   2248    117     20   3641   3646     77
  7   5323   5139     44            H,  7,  2            23   2087   2048     99
  8   3244   3135     47      1   8926   9468     45            H, 10,  2
 10   5980   6061     47      2   1401   1369     53      0   2617   2657     53
 12   1383   1279     67      3   1123   1239     65      1   2122   2170     60
 13   1747   1666     63      4   3957   4106     46      2   4783   4966     52
 16   2928   2863     67      5   2087   2172     53      3   2286   2290     63
 17   2766   2652     64      6   3353   3318     52      5   2082   2153     60
 18   1498   1378     89      8   1838   1884     56      6   2151   2246     61
 19   3528   3236     68     10   2832   2848     56      7   2780   2883     60
 20   1811   1836     85     11   2340   2104     60      8   4473   4514     58
 21   1211   1335    118     12    857    634     88     10   2693   2791     63
 22   3067   3047     80     13   2168   2079     64     11   2956   2893     64
 23   1508   1682    107     14   1702   1631     75     12   2765   2845     63
 26   2723   2920    102     15   2360   2422     69     14   1780   1714     79
       H,  5,  2             17   1180   1179    100     16   3048   3226     73
  1   1394   1474     48     19   1794   1735     87     18   2471   2498     79
  2   5728   6237     40     20   1584   1539     97     19   1710   1884    105
  3   1624   1654     53            H,  8,  2            20   2521   2663     89
  4   3895   3965     43      0   5361   5468     46     21   1024   1085    154
  6   3997   3984     46      1   2663   2852     51     23   1941   1920    105
  7   3429   3374     47      2   4750   4907     49            H, 11,  2
  8   5822   5630     47      4   1827   1833     58      2   3686   3807     57
 10   6559   6488     48      6   5217   5187     52      3   1670   1773     66
 11   2310   2399     58      7   2314   2247     59      6   3104   3115     59
 12   4958   4413     51      8   2698   2765     56      7   2113   2154     70
 13   4943   4664     53      9   4388   4208     55      8   3287   3282     62
 14   5097   4937     56     10   2994   2798     55      9   1918   2009     72
 15   1685   1491     70     12   3442   3395     60     11   1370   1430     93
 16   3798   3653     62     13   5775   5506     60     12   1662   1761     78
 17   4682   4573     63     14   3527   3378     65     16   4139   4024     71
 18   1512   1353     83     16   6353   6137     64     18   1866   2025     97
 20   4808   4847     68     18   1996   1920     81     19   1722   1775    100
 21   1587   1753    100     19   3056   3051     78     21   1471   1583    130
 22   1912   1726     93     22   2688   2614     93     23   1359   1129    130
 24   2836   2934     92     23   4451   4585     83            H, 12,  2
       H,  6,  2             24   2385   2467    100      0   2089   2078     61
  0   4092   4161     41            H,  9,  2             1   2826   2777     63
  1    831    964     66      1   4719   4976     52      4   2169   2355     59
  2   3146   3338     44      2   8063   8532     51      6   4633   4572     59
  4   2739   2839     47      4   5374   5449     50      7   1844   1802     73
  5   7149   7205     47      5   2044   1980     59      8   1625   1563     81
  6   3025   3132     50      6   6163   6214     54      9   3246   3346     70
  8   1139   1019     65      7   1121   1111     79     11   2239   2255     74
  9   3589   3465     51      8   7110   7046     55     13   3662   3464     68
 10   3638   3589     51      9   1968   2045     63     16   4097   4073     74
 11   1785   1738     61     10   3448   3344     57     18   1887   2014     98
 12   2615   2599     56     11   3219   3197     62     19   1821   1580    102
 14   2221   2188     69     12   2753   2656     62     20   1863   1816     98
 16   1753   1617     80     14   1996   1919     76     23   3540   3564     91
 18   3974   3853     67     15   4335   4203     64            H, 13,  2
 20   1441   1452     91     16   3824   3619     68      1   2858   3027     68
 21   1614   1667     96     18   2096   2058     79      2   1124   1148     80
 24   3080   3272     90     19   1696   1736    100      3    909    878     94
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        COLUMNS ARE 10FO,10FC,  10SIG

  H    KFO     FC    SIG      H    KFO     FC    SIG      H    KFO     FC    SIG
       H, 13,  2                   H, 17,  2              9   1641   1671    116
  4   1499   1512     71      1   1504   1496     94            H, 23,  2
  5   1514   1624     79      2   2863   2832     67      2   1804   1728    113
  7   2348   2466     73      4   1119   1113    108      3   2129   2277     99
  8   1099   1116    101      6   2182   2155     78            H, 24,  2
  9    818    760    143      7   2787   2960     78      1   1389   1332    120
 10   1376   1456     86      8   3457   3184     74      2   4066   3912     84
 11   2820   2709     71     14   2407   2349     98            H,  1,  3
 15   2882   2866     76     16   2878   2944     92      1   1799   1769     40
 16   1670   1732     96     17   2047   1972    108      2   1667   1615     40
 19   1999   1912    102     18   1500   1565    124      3   4495   4451     39
 20   2050   2038     98            H, 18,  2             4   4088   4120     40
       H, 14,  2              0   2415   2444     80      6   1953   2003     51
  0   6047   6186     61      1   2682   2713     79      8    771    661     92
  1   2355   2488     73      4   1612   1587     88      9   2882   2846     53
  2   2231   2202     65      5   2092   2225     85     10   2237   2191     54
  3   2437   2345     70      6   1711   1587     89     11   1399   1298     66
  4   1805   1917     75      8   2633   2554     84     12   2299   2291     56
  7   1818   1842     78      9   1603   1394    105     13   3933   3729     55
  8   6313   6083     64     11   1609   1764    110     14   4692   4512     57
 10   4515   4465     69     12   1897   1834    100     15    978    970     96
 11   4448   4316     73     18   1858   1823    115     17   3397   3162     65
 14   1609   1377     92            H, 19,  2            18   3944   3685     67
 16   2214   2261     89      2   2615   2668     83     19   3026   2971     70
 18   5636   5600     80      4   4354   4390     70     20   2130   2187     81
 19   1627   1817    119      6   3568   3536     76     21    925    823    141
 20   2549   2462     97      8   2653   2596     83     23   1127   1169    138
 21   1831   1770    123     12   1823   1732    113     24   2021   2252    109
       H, 15,  2             14   3000   3100     94            H,  2,  3
  1   3707   3538     69     16   1688   1610    119      0   8967   9356     36
  3   3879   3788     67     17   1343   1451    155      1   3384   3415     37
  4   5242   5233     61            H, 20,  2             2   3175   3280     37
  6   2626   2689     69      0   4217   4146     78      4   6382   6381     41
  7   1951   1962     76      2   1262   1432    120      5  11281  12412     46
  9   1585   1451     94      3   2380   2384     85      6   8429   9014     44
 11   3458   3438     79      5   1712   1798     96      7   1471   1551     60
 14   3388   3264     81      6    809    886    174      8   3705   3696     52
 15   1776   1738    107      7   4078   3992     81      9   5551   5644     50
 16   1872   1760     96     10   2762   2638     89     10   7312   7408     50
 19   2439   2442    103     11   1706   1743    119     12   4520   4359     54
       H, 16,  2             12   1902   1851    105     13   1393   1301     78
  0   6912   6818     66     14   2500   2437    100     14   1365   1180     72
  1   2313   2230     74     15   1900   1560    108     15   4425   4370     58
  2   2011   2084     76            H, 21,  2            16   3583   3516     64
  3   3653   3546     70      1   1545   1613    110     17   5216   5032     62
  4   3966   3936     64      4   2531   2479     83     18   1867   1868     80
  6   1626   1591     88      5   1216   1002    121     19   3681   3784     69
  7   3962   3938     71      8   1151   1129    121     20   1528   1404     89
 10   4314   4216     72     12   1285   1324    140     21   1783   1894     93
 11   3168   3192     79     13   1015    931    182     22   2608   2558     86
 12   1589   1496    115            H, 22,  2            23   1778   1770    103
 14   2887   2987     86      1   1405   1333    121     24   3122   3175     87
 17   1650   1553    110      2   1251   1111    126            H,  3,  3
 20   1863   1973    129      5   2867   2762     88      1   1700   1641     43
```

```
COLUMNS ARE 10FO,10FC,  10SIG
```

```
 H   KFO    FC   SIG
     H,  3,  3
 2  3912  4141    38
 3  1823  1813    47
 4  3436  3460    43
 5  1563  1649    57
 6  6166  6264    45
 7  1023  1209    68
 8  2375  2512    54
 9  2168  2161    57
10  1523  1517    61
12  7327  6784    53
13  3611  3397    57
14  1019  1059    84
15  4864  4765    59
16  3066  2997    66
17  4937  4860    64
18  3191  3164    70
19  2592  2561    73
22  4503  4566    75
     H,  4,  3
 0  2124  2247    42
 1  4779  4972    40
 2  5079  5302    40
 3  4030  4165    44
 4   763   772    69
 5  1721  1677    54
 6  4483  4648    46
 7  7240  7450    48
 8   998   988    73
 9  4834  4712    53
12  7404  7466    54
13  3421  3521    61
16  1129   893    98
17  2828  2759    68
20  2501  2557    79
22  3631  3570    77
23  3279  3437    86
     H,  5,  3
 1  5874  6105    42
 2  2283  2482    44
 4  3665  3696    47
 5  3561  3522    49
 7  1289  1189    63
 9  2840  2917    57
10  1118  1353    85
11  1687  1799    67
14  3142  3173    64
15  5290  5133    62
16  1257  1318    95
17  1426  1401    87
18  2186  1985    77
20  1524  1567    95
21  2157  2215    90
     H,  6,  3
```

```
 H   KFO    FC   SIG
 0  3063  3255    44
 1  4032  4239    44
 2  3346  3437    44
 3  1981  1975    55
 4   991  1027    70
 5  3411  3492    51
 6  2997  3026    51
 7  4595  4676    51
 9  1626  1571    66
10  1216  1308    80
11  6543  6607    57
12  2953  3227    60
13  1489  1631    77
14  1471  1453    84
15  5028  4966    63
16  2250  2153    70
17  2598  2567    80
18   875   900   139
20  1377  1456   119
21  4539  4482    76
22  1830  1865    99
24  2479  2673   106
25  3183  3327    93
     H,  7,  3
 1   850   931    70
 2  1663  1692    54
 3  5061  5225    52
 4  8215  8749    50
 6  7751  8330    49
 7  3013  3031    53
 8  1422  1413    65
10  3770  3904    58
11  2386  2513    67
12  6131  6201    58
13  2317  2255    67
14  4793  4584    64
15  3062  2914    68
16  4559  4603    65
18  1935  1919    83
20  2234  2326    87
21  1477  1761   133
22  5167  5413    78
     H,  8,  3
 0  8939  9410    47
 1  2886  3101    52
 2  2327  2449    50
 3  1670  1514    66
 4  1775  1906    59
 6   970   946    75
 7  2670  2805    55
 9   961   903    85
10  4035  3991    59
11  2747  2788    67
12  3702  3716    63
```

```
 H   KFO    FC   SIG
18  2801  2872    77
21   917  1104   168
23  1094  1231   159
     H,  9,  3
 1  2769  3122    54
 2  1856  2083    59
 3  3446  3507    59
 4  3061  3028    55
 5  1358  1243    65
 6  3316  3363    55
 8  1853  1716    65
10  1177  1246    85
11  2604  2586    69
13  1960  1837    77
14  2111  2087    75
15  1943  1896    85
16  2203  2254    77
17  1688  1552    89
18  1405  1416    97
19  1457  1692   106
24  1440  1487   135
     H, 10,  3
 0  2388  2393    55
 1  5456  5727    54
 2  4097  4147    55
 3  4238  4261    61
 7  4151  4382    55
 8  1322  1408    76
11  7809  7955    64
12  4537  4626    64
13  2171  2120    81
14  1849  1873    85
15  3048  3109    72
17  1445  1547    97
18  1998  1975    95
19  1009  1044   147
20  1807  2057   107
21  4773  4897    82
22  1990  1997   111
     H, 11,  3
 1  4043  4225    57
 2  1206  1210    73
 3  6052  5868    61
 4  3585  3909    59
 6  3600  3634    57
 7  1509  1648    72
 8  2104  2003    65
10   969  1044   105
11  2706  2538    72
12  3292  3447    69
13  2768  2794    78
16  1327  1349   106
18  3270  3254    85
19  1779  1780   102
```

```
COLUMNS ARE 10FO,10FC,  10SIG
```

```
 H   KFO    FC   SIG
     H, 11,  3
22  2902  3057    96
     H, 12,  3
 0  7948  8413    55
 2  2226  2262    65
 3   956   813   101
 4  3826  4003    61
 6  5070  5118    58
 7  3352  3377    64
 8  1915  1917    73
 9  3211  3283    66
10  5438  5445    62
11  3294  3303    71
12  1988  1948    78
13  2774  2756    76
15  1730  1576    95
16  3397  3397    81
17  1518  1728   111
18  2771  2793    94
20  2685  2881    92
21  2549  2732   111
22  1820  1904   126
23  2099  2276   115
     H, 13,  3
 1  4263  4270    63
 2  1509  1561    81
 4  2569  2666    67
 5  2277  2276    68
 9  3870  3865    67
12  1970  1828    80
14  3279  3312    78
15  2411  2562    84
17  1059   931   145
18  2261  2217   100
20  1271  1300   129
21  2025  2258   114
     H, 14,  3
 0  4237  4125    62
 1  4446  4565    66
 2  1424  1538    83
 3  1849  1880    79
 5  3074  3081    67
 6  1051   993    88
 7  1356  1251    84
 8  1653  1712    82
 9  3595  3702    66
10  2134  2158    73
11  2768  2773    73
13  3159  3278    79
17  1391  1159   117
18  2287  2195   104
21  1657  1647   120
     H, 15,  3
 1  2189  2244    76
```

```
 H   KFO    FC   SIG
 2  1318  1299    86
 4  3190  3189    67
 8  1962  1885    74
 9  2563  2468    72
10   832   566   114
12  2073  2198    86
14  3597  3690    78
18  3562  3405    90
     H, 16,  3
 0  2718  2619    70
 2  1271  1437    97
 4  3765  3685    65
 5  2812  2837    75
 6  5877  5936    67
 7  2216  2264    73
10  2033  2108    82
11  1132   943   120
12  1567  1555   103
13  2199  2243    93
16  3315  3379    87
17  1756  1723   115
     H, 17,  3
 1  3414  3456    74
 2  3257  3289    74
 3  1707  1757    87
 4  1923  1978    80
 6  3912  3850    71
 8  1661  1550    89
 9  2547  2544    84
12  2897  2937    83
13   966  1392   180
16  3202  3275    89
     H, 18,  3
 1  2297  2333    65
 2  3912  3931    72
 5  5284  5280    71
 8  1357  1315   108
 9  4224  4378    78
12  2397  2586    88
13  2164  2157   103
17  1755  1406   118
     H, 19,  3
 2  2187  2228    86
 3  2766  2876    82
12  1653  1810   117
13  2502  2610    93
14  1555  1484   122
16  2027  2019   106
     H, 20,  3
 2  1703  1644    99
 4  1842  1818    97
 5  3296  3221    80
 6  3163  3095    81
 7   922  1071   166
```

```
 H   KFO    FC   SIG
 8  1745  1514    96
 9  1647  1729   107
     H, 21,  3
 2  1789  1508    99
 4  2805  2924    86
 6  3223  3149    83
12  2920  2867    92
     H, 22,  3
 0  3022  2955    87
 3  2348  2309    88
 4  1016   322   132
 5  2390  2281    90
 7  2950  2877    91
10  2748  2706    95
     H, 23,  3
 2  1491  1427   114
 4  1766  1636   105
 6  2037  1908   113
     H,  0,  4
 0  1842  1917    48
 1  7960  8626    42
 2  7256  7433    40
 3 12774 13120    44
 4  2555  2661    46
 5  4945  5202    46
 6  1317  1326    64
 7 13575 14046    50
 8  8920  9751    50
 9  2545  2407    59
10  4395  4529    55
11  3426  3380    58
12  3834  3837    57
13  3358  3244    60
14  1211  1229    86
15  3268  3315    66
17  5333  5237    64
18  5684  5855    67
19  3776  3823    69
20  3045  2993    74
21  1469  1352   107
25  2077  1955   112
26  1450  1862   129
     H,  1,  4
 1  2570  2557    44
 2  1189  1125    49
 3  2335  2177    50
 4  4215  4251    44
 5  5248  5314    47
 7  2022  2028    56
 9  1832  1653    60
10  1659  1718    62
11  2134  1978    66
12  1413  1413    71
14  1709  1612    76
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC   SIG
     H,  1,  4
15  7462  7367    61
16  2570  2619    70
17  1694  1745    85
19  3877  3891    71
20  1355  1536   106
25  3638  3636    91
26  2577  2580   109
     H,  2,  4
 0  7599  7939    41
 2  2168  2208    44
 3  4009  3972    46
 4  1852  1761    50
 5  5910  6062    47
 7  1765  1936    60
 8  2137  2248    55
 9  1677  1576    63
10  2754  2871    56
11  3751  3470    60
12  1566  1599    74
13  1310  1416    85
14  2116  2077    73
16  1273  1360    90
18  1085   932   106
19  1229  1292   106
21  1575  1728   110
22   984   774   145
24  1345  1436   130
25  1956  1926   113
     H,  3,  4
 1  1041   871    57
 2  4232  4310    43
 3  4963  5007    46
 5  3893  3936    50
 6  2792  2852    56
 7  3057  2946    55
 8  4354  4494    52
 9  1204  1348    65
11  1250  1150    81
12  1156  1181    82
13  3560  3532    65
15  3646  3558    65
16  5160  5034    64
17  2165  2056    75
18  1966  1944    81
20  1179  1074   110
25  3007  3046    91
26  3011  3014    96
     H,  4,  4
 1  4629  4795    44
 2  5394  5839    43
 3  9840 10162    47
 4  2137  2173    54
 5  3948  3915    51
```

```
 H   KFO    FC   SIG
 6  1233  1227    69
 7  9531  9940    51
 8  6500  6449    52
 9  6580  6572    52
11  2718  2503    63
12  1924  1923    72
13  6414  6269    61
14  3064  2932    70
15  2230  2321    76
16  1037  1032   114
17  4212  4104    69
18  3154  3149    75
19  3854  3796    73
23  1910  1833   111
24  1158  1075   141
     H,  5,  4
 1  2833  2888    48
 2  5767  6027    45
 3  1192  1376    66
 4  6590  6915    48
 5  2098  2199    60
 6  5351  5475    52
 7  1211  1378    71
 8  6357  6365    52
 9  7884  7859    53
10  3812  3966    55
11  2505  2472    63
12  1517  1659    77
14  4806  4843    66
15  3911  4031    68
16  2055  2121    81
18  3033  3039    74
19  5985  6014    69
20  2985  3075    79
21  2886  2821    85
23  1598  1674   118
24  2372  2369   106
25  1540  1774   130
     H,  6,  4
 0  5918  6161    46
 1  4331  4527    48
 2   752   764    71
 3  5053  5180    52
 4  1432  1564    68
 5  5234  5353    55
 6  1404  1323    66
 7  2176  2188    57
 9  1197  1032    82
10  4269  4145    57
11  5697  5811    59
13  3711  3810    68
14  4623  4534    68
16  2952  3095    75
17  1344  1376   107
```

```
 H   KFO    FC   SIG
19  2022  1975    91
21  1268  1255   133
24  1857  2104   118
25  2473  2468   102
     H,  7,  4
 1  2729  2744    53
 2  1117  1126    64
 3  4396  4635    55
 4  1169  1260    71
 9  1653  1652    74
10  1012  1029    85
11  2499  2655    66
13  5775  5719    66
14  1905  1888    85
15  1254  1201   100
17  3685  3826    75
     H,  8,  4
 0  3604  3745    51
 1  7722  8176    51
 2  2786  2946    53
 3  5326  5662    55
 4  2094  2182    59
 5  3751  3853    60
 6   960  1114    86
 7   911   877    88
 9  8408  8475    56
10  3834  3951    61
11  2194  2106    70
13  8610  8600    64
14  2748  2642    75
15  1730  1736    89
18  1416  1373   117
19  3183  3345    82
20  2387  2390    89
22  1683  1739   131
23  5081  5257    88
24  2524  2670   109
     H,  9,  4
 1  6459  6683    53
 2  4241  4427    54
 3  1213  1163    75
 4  3352  3290    59
 5  1118  1254    81
 6  1575  1493    67
 7  1808  1797    62
 8  3612  3673    59
 9  7037  7256    58
10  4223  4130    60
11  1186  1333    91
12  2270  2335    68
13  1099  1255   106
14  3653  3505    71
17  1409  1341    98
18  1298  1363   128
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC   SIG
     H,  9,  4
19  3036  3043    86
20  3972  4042    81
21  4063  4121    87
24  2099  2186   108
     H, 10,  4
 0  2595  2619    59
 1  3366  3483    59
 2  6369  6504    55
 3  3568  3526    63
 5  2630  2771    64
 8  3421  3472    60
 9  3870  3907    60
10  2181  2058    69
12  3289  3264    65
13  4127  4233    69
16  1206  1264   105
18  1497  1612   124
19  1246  1486   144
20  1626  1622   108
21  2035  2037   107
23  1579  1746   135
     H, 11,  4
 1  1172   948    74
 2  2296  2414    65
 3  2873  3081    67
 4  1686  1790    75
 6  3220  3252    62
 7   832   766   102
 8  2642  2676    63
 9  1095  1115    89
12  1394  1458    87
13  1505  1362    95
15  1924  1945    91
16  2924  2999    78
18  1867  1944    98
     H, 12,  4
 0  2247  2247    66
 1  6329  6594    60
 2  2685  2842    64
 3  4242  4252    67
 5  2868  2986    63
 7  1551  1504    81
 9  5886  5891    60
10  1949  1972    74
11  3526  3452    67
12  1644  1686    91
13  5071  5138    68
15  1171  1281   117
19  1698  1835   112
     H, 13,  4
 1  3769  3881    65
 3  3466  3621    72
 6  2715  2861    66
```

```
 H   KFO    FC   SIG
 9  3261  3315    67
10  3267  3172    68
13  2324  2399    80
15  1621  1767    95
16  2501  2501    86
17  1495  1559   110
18  1182  1057   136
19  1653  1712   129
20  2676  2838    99
21  1798  1710   126
     H, 14,  4
 0  5939  6006    63
 2  5589  5761    64
 3  3154  3197    76
 5  4426  4523    65
 6  1977  1968    74
 7  2602  2593    73
 8  5061  5092    64
 9  4571  4491    66
10  3492  3569    69
11  3610  3407    70
12  2267  2376    75
13  2576  2610    83
17  2305  2033    91
18  2730  2832    93
19  3505  3353    93
21  2078  2169   121
     H, 15,  4
 1  2730  2831    73
 3  5861  5689    71
 4  2330  2357    73
 7  1383  1264    94
10  1221  1237    99
11  1946  1921    91
13  3493  3634    78
14  2702  3043    88
17  1949  2117   104
19  1180  1108   155
     H, 16,  4
 0  1432  1623    93
 1  1615  1523    91
 2  1668  1792    88
 3  3890  3811    75
 7  4584  4499    65
 8  2291  2424    78
10  1315  1501   108
11  3339  3556    80
12  1769  1769   101
15  1854  1953   106
17  2527  2513    96
18  2132  2124   104
19   903   798   184
     H, 17,  4
 1  1101  1152   122
```

```
 H   KFO    FC   SIG
 2   902  1203   147
 5  2485  2652    78
12   993  1110   132
13  1288  1372   131
14   936  1039   163
15  4563  4689    77
16  2500  2369    94
     H, 18,  4
 3  4025  4117    75
 4  2410  2480    82
 5  1894  2014    93
 6  1183  1413   125
 7  4833  4905    73
 8  2382  2506    81
 9  1223  1277   123
11  1090   678   129
14  1418  1198   117
     H, 19,  4
 1  1305  1461   109
 2  2100  1898    91
 4  3455  3546    78
 6  2526  2417    84
 8  1580  1676   106
 9  1649  1454   107
12  1251  1247   132
14  3081  3065    92
     H, 20,  4
 0  1192  1156   125
 3  1763  1864   107
 5  1591  1769   111
 6  1014   897   135
 7  3522  3704    81
     H, 21,  4
 1  1677  1961   115
 4  2931  2850    82
 5  1773  1712   102
 6  2033  2144    99
 7   929  1260   191
     H, 22,  4
 0  2525  2401    96
 3  3511  3488    88
 4  1786  1817   104
 7  3324  3368    89
     H, 23,  4
 2  1712  1507   105
 3  1898  1839   113
     H,  1,  5
 1  1745  1783    54
 3  5487  5311    49
 4  2009  2062    51
 6  1679  1546    58
 7  2035  2103    62
 9  3456  3537    57
10  1391  1492    74
```

```
COLUMNS ARE 10FO,10FC,  10SIG

 H    KFO     FC    SIG
      H,  1,  5
11    954   1110    114
13   4008   4163     63
14   2308   2387     76
15   1560   1432    100
16   2276   2249     78
17   4142   4219     72
18   1645   1786     93
20   1650   1712    101
22   1252   1264    128
23   2045   2245    113
24   1441   1734    151
      H,  2,  5
 0   3617   3508     47
 1   6673   6862     47
 2   2364   2128     47
 3   3501   3485     51
 4   1290   1311     64
 5  13198  14270     51
 6   4879   4825     52
 7   4940   5053     54
 8   2230   2295     61
 9   5875   5677     55
10   3370   3244     56
11   2926   2862     61
14   1187   1113    112
15   5401   5271     68
16   5596   5660     68
17   4278   4198     72
18   1761   1840     99
19   1554   1641    101
20   4356   4644     76
      H,  3,  5
 2   3383   3435     47
 3   1163   1143     69
 4   5023   4896     49
 5   2405   2526     55
 6   4713   4767     54
 7   5023   5347     56
 8   5509   5455     56
 9   1092    983     74
11   3541   3583     60
13   3967   4031     62
14   2627   2488     72
15   1062    804    108
16   1191   1173    125
17   5905   5738     69
18   1567   1539     97
21   2955   3175     88
23   2479   2649    102
      H,  4,  5
 0   5247   5035     48
 1   5477   5494     49
 2   5606   6292     47
```

```
 H    KFO     FC    SIG
 3   1673   1686     64
 4   4092   4178     51
 5   1385   1501     68
 6    916    988     82
 7   5546   5797     56
 8   1702   1719     67
 9   3656   3654     56
10   3295   3266     57
11   3127   3111     60
12   4839   4877     59
13   3127   3011     67
14   3927   3992     68
16   1728   1772     90
17   2492   2711     88
18    956    939    140
19   1039    998    140
20   1440   1314    109
22   1591   1698    120
23   2999   3208     94
      H,  5,  5
 1   4421   4654     50
 3   1871   1874     59
 4   3912   3856     54
 5   2453   2499     59
 6   3969   3964     56
 8   2097   2214     63
 9   3843   3852     59
10   3592   3827     59
11   1824   1858     68
12   1503   1412     75
15   5117   4876     65
16   1028   1004    114
18   1239   1283    112
20   1855   2044     97
21   1959   1854    105
22    990    923    182
      H,  6,  5
 0   2567   2644     53
 1   6158   6435     51
 3   3548   3515     59
 4   2145   2230     59
 5   5562   5928     55
 6   1789   1706     66
 7   4059   3969     58
 9   3291   3540     59
10   1129   1203     85
11   7394   7446     57
12   1947   1978     67
13   1099   1030     96
15   4423   4518     69
16   1317   1169    110
17   2018   2105     87
18   1617   1738    108
21   3469   3461     89
```

```
 H    KFO     FC    SIG
22   2683   2650     95
      H,  7,  5
 1   2106   2112     59
 2   2411   2419     56
 3   5162   5155     56
 4   5847   6045     56
 6   3945   4335     60
 7   6423   6511     57
 8   2077   2041     64
 9   2515   2519     68
10   1388   1336     76
11   5024   5212     62
12   2810   2821     66
14   5608   5641     69
15   1686   1617     88
16   1782   1718     89
17   3340   3370     77
18   1528   1535    105
19   2670   2624     92
21   4022   4238     87
22   1559   1557    133
23   3067   3157    104
24   2159   2337    118
      H,  8,  5
 0    802    804     93
 1   6640   6717     55
 4   2242   2448     61
 5   1566   1535     73
 7   1331   1321     79
 8   1597   1739     73
 9   2108   2093     66
11   4934   4934     60
13   1974   1920     76
14   1101   1031    101
16   1232   1214    114
17   1444   1521    105
19   1915   2032    104
21   1594   1420    124
      H,  9,  5
 1   1035   1244     90
 3   1267   1327     78
 4   2238   2253     65
11   1465   1698     91
12   1826   1913     79
13   2042   2099     77
14   2189   2254     80
15   1676   1614     88
16   2036   2042     80
18   1509   1461    113
19   1065   1194    152
22   1852   1619    124
      H, 10,  5
 0   1874   1666     65
 1   7646   8034     58
```

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COLUMNS ARE 10FO,10FC,  10SIG

 H    KFO     FC    SIG
      H, 10,  5
 2   1103   1047     82
 3   3697   3733     65
 5   1153   1292     85
 7   2830   2939     63
 8   1672   1842     75
 9   1221   1189     82
11   8042   8275     62
12   2584   2642     71
13   2118   2292     78
14   1847   1984     85
15   2380   2384     78
18   2602   2560     86
19   1945   1703     96
20   1043   1108    145
21   3419   3661     93
22   3283   3413     93
      H, 11,  5
 1   3250   3379     64
 2   1251   1209     87
 3   4350   4436     66
 5   1432   1434     79
 6   1386   1523     85
 7   3530   3708     63
 8   3073   2991     65
10   2979   2920     71
11   4107   4140     67
12   2452   2501     74
13   1153    843     99
14   2343   2326     76
17   1871   1992     93
18   1253   1229    127
19   2398   2549     94
20   2271   2347     97
21   2134   2034    108
22   1694   1657    131
      H, 12,  5
 0   5950   6203     59
 1   2064   2220     72
 2   6350   6574     61
 3   1867   1812     79
 4   1940   1870     78
 5   4062   4006     67
 6   1062    980     95
 7   4078   4078     65
 8   4180   4267     63
10   4076   3960     65
11   3747   3865     67
12   3880   3945     67
13   1938   1999     93
14   1792   1826     91
15   1852   2012     85
17   1665   1782     99
20   1206   1081    138
```

```
 H    KFO     FC    SIG
21   2439   2375    109
      H, 13,  5
 1   4036   4121     67
 4   2161   2373     80
 5   2132   2167     77
 6   2890   3015     70
 9   2464   2518     72
10   3037   2986     70
11   1668   1515     84
15   3272   3302     76
20   2184   2197    101
      H, 14,  5
 0   2674   2660     70
 1   3698   4019     71
 2   1238   1316     95
 3   2305   2237     81
 5   2150   2198     78
 6   1410   1606     98
 7   1220   1209     90
 9   2801   2897     72
10   2677   2774     76
11   2228   2519     86
13   2040   2303     88
14   1348   1364    108
16   1430   1401    114
20   1681   1818    110
      H, 15,  5
 1   3179   3345     74
 3   1061   1170    139
 4   1124   1146    113
 6   2079   2239     78
 7   1745   1938     87
 8    945   1260    131
 9   1254   1334    106
10   1563   1590     96
11   1664   1722     96
13   2664   2673     86
15   1550   1674    105
16   1909   2006     96
17   3056   3222     87
      H, 16,  5
 0   2394   2383     79
 2   3062   3160     78
 3   1351   1126    107
 5   5966   6013     69
 7   3203   3338     77
 8   1695   1750     95
10   1478   1539    107
12   2277   2220     90
13   2483   2450     91
15   2351   2343     87
17   2479   2447     91
      H, 17,  5
 1   2864   3051     81
```

```
 H    KFO     FC    SIG
 4   1542   1546     99
 6    885    905    150
 9   1818   1758    100
10   1738   1854    102
11   1801   1943     98
12   1785   1823     96
14   2491   2391     84
15   2179   2009     94
16   1138   1174    146
      H, 18,  5
 0   1370   1239    114
 2   1333   1364    109
 5   3274   3503     83
 6   2583   2671     82
 9   2529   2508     88
10   2592   2660     85
15   1795   1453    109
      H, 19,  5
 2   1553   1368    101
 3   2891   2923     83
 8   1953   1971     96
 9   1489   1553    118
13   3034   2915     90
      H, 20,  5
 0   2483   2356     94
 1   1692   1791    108
 5   4880   4847     79
 7   1016   1029    159
 9   1967   2031    105
10   1761   1690    108
12   1859   1820    108
      H, 21,  5
 4   3073   3037     88
 6   2207   2113     97
 7   2471   2454    100
 8   1467   1470    121
      H, 22,  5
 2   1791   1774    115
 6   1346   1108    138
      H,  0,  6
 0   2768   2839     55
 1   1642   1569     63
 3   5874   5820     53
 4   1037   1046     70
 5   1300   1400     69
 6   2248   2208     59
 7   7003   7192     55
 8   3151   3295     58
10   2056   2063     72
11   3599   3825     63
14   4091   4180     67
15   1003    927    115
16   2606   2644     81
17   2695   2985     83
```

```
   COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG
      H,  0,  6           16  1977  1883    80     10  1450  1480    78
 18  6935  6929    76     18  2753  2893    79     11  2368  2420    67
 19  3229  3403    88     19  1305   940   113     12  4160  3975    63
 21  1379  1349   137     21  1344  1327   136     13  3271  3297    67
 22  3947  3999    89     22  1021   940   164     14  2562  2496    73
 24  1489  1683   147     23  1200  1186   162     15  1168  1097   108
      H,  1,  6           24  1369  1319   141     16  2176  2337    80
  2  1320  1364    65          H,  4,  6           17  2317  2470    84
  3  2664  2672    58      0  1683  1817    64     22  1184  1066   146
  4  1388  1266    68      1   625   803   119     23  1658  1908   144
  5  2040  2142    65      2  1260  1371    65          H,  7,  6
  6  3261  3267    56      3  4893  4898    56      1   846   752    94
  7  1886  2010    65      5   783   854   107      3  2911  3044    62
  9   876   697   104      6   955   977    91      4  1991  2082    65
 10  3972  3938    62      7  5421  5506    60      6  1176  1228    84
 14  2351  2355    76      8  2322  2354    65      7   997   991    91
 15  4081  3931    68      9  2367  2486    66      8  2249  2271    68
 17  1813  1783    89     11  1671  1603    73     10  2718  2676    65
 18  1659  1501   102     13  3027  2938    65     11  1252  1281    89
 20  1688  1864   113     14  1975  2005    80     12  2741  2914    69
 24  3055  2996   103     17  1365  1403   109     13  1412  1361    91
      H,  2,  6           18  3280  3357    78     16  1288  1205   100
  0  4142  4157    53     19  2440  2395    86     20  1333  1098   124
  1  1736  1815    62     21  1808  1769   115     22  1698  1829   121
  2  4243  4248    51     22  1660  1831   125          H,  8,  6
  3  1641  1664    62     24  1948  1936   117      1  3555  3592    61
  4  3228  3302    55          H,  5,  6            2  2321  2300    62
  5  2592  2493    61      1  4425  4381    56      3  3441  3299    63
  6   871   687    83      2  4202  4230    55      4   916   948    97
  8  1422  1398    78      3  1125  1036    83      5  2271  2308    68
  9   813   829   107      4  5627  5994    55      6  1659  1660    77
 10  1854  1753    69      6  7125  7793    57      9  2867  2854    68
 11  1781  1820    68      7  1644  1742    76     10  2105  2032    73
 12  3263  3211    62      8  1785  1748    70     13  4144  4299    67
 13  1589  1619    80      9  5515  5415    61     16  3380  3390    72
 14  2207  2072    77     10  3552  3596    62     17  1104   900   118
 15   905   907   137     12  4095  4227    63     18   868  1110   163
 16  1648  1766    92     13  1219  1046    94     20  3889  4230    86
 17  1204  1280   112     16  4431  4524    70          H,  9,  6
 19  1246  1109   125     17  2144  2057    82      1  4363  4413    61
      H,  3,  6           19  2813  3062    91      2  1099  1105    86
  2  1844  1833    62     20  2344  2346    89      3  1270  1440    83
  3  4998  4971    55     21  1506  1494   115      5   888   849   109
  4  4160  4111    54     22  1702  1651   123      8  2525  2548    65
  5  2785  2618    59          H,  6,  6            9  6344  6327    61
  6  4582  4697    56      0  3439  3466    56     10  1413  1347    81
  7  3472  3349    59      1  1923  1953    63     12  4539  4514    64
  8  3440  3434    61      2  5815  6580    53     16  2661  2652    76
  9  1469  1362    74      3  2929  2961    61     17  1632  1775    97
 10  1404  1276    82      4  2411  2291    63     18  1905  1949    97
 11  1388  1378    79      5  4469  4532    61     19  2467  2608    89
 13  2141  2127    72      6  1922  2069    68     21  2310  2443    97
 14  2751  2770    76      7   960  1043   106     22  3428  3608    92
 15  4227  4125    66      8  1860  1973    72          H, 10,  6
```

```
   COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO    FC   SIG      H   KFO    FC   SIG      H   KFO    FC   SIG
      H, 10,  6           12  1996  2061    88      4  2077  1848    92
  0  5387  5488    60     14  2069  2110    88      6  2438  2472    91
  3  2504  2465    70     17  1031   825   133      9  1527  1541   120
  4  3316  3233    66     18  3182  3237    87     12  2385  2268    98
  5  1261  1338    94          H, 14,  6                H, 20,  6
  6  2979  2831    70      0  6968  7060    66      0  2528  2488    95
  9  1461  1556    91      1  1824  1892    93      2  1352  1266   126
 10  4332  4356    64      2  3065  3194    73      4  2365  2344    99
 11  1153  1034   101      3  2830  2826    79      5  1935  1826   101
 12  1455  1404   100      4  3745  3794    78      7  1803  1859   109
 13  2297  2298    75      6  1765  1805    85      8  2395  2293    91
 14  1690  1790    85      7  2670  2698    76          H, 21,  6
 19  1423  1178   118      8  1306  1493   113      3  1202  1241   137
 21  1380  1469   135      9  1088   894   118      4  2279  2205    98
      H, 11,  6           10  4396  4636    74      6  2299  2436    95
  2  1590  1797    78     11  3326  3249    77          H,  1,  7
  3  2385  2368    72     12  2745  2856    82      1  3947  4295    61
  4  2152  2216    77     14  2788  2893    80      2  1443  1681    72
  5  1380  1430    96          H, 15,  6            3  1691  1671    69
  6  2290  2237    77      3  3360  3207    76      4   819   889   100
  7  1940  1948    76      7  2087  2073    85      5   940   955    89
  8  2770  2819    69      8  2785  2884    78      6  2137  2100    65
  9  1430  1289    98     11  1719  1712    97      9  5013  4861    65
 13  1391  1294   100     12  2935  3006    79     11  1163  1360   101
 14  2015  2132    85     13  1912  1916    96     12  2410  2515    73
 15  1941  2224    85     15  1635  1699   105     13  1644  1637    84
 16  1614  1815   102          H, 16,  6           14  1176  1254   102
 18  2394  2353    89      0  2449  2436    84     16  3169  3289    78
 21  1167  1128   147      1   840   865   163     18  1080   968   133
      H, 12,  6            2  1615  1723   101     19  3005  2982    85
  0  1134  1163    95      3  1153  1337   122     21  2307  2449    99
  1  2974  3031    72      4  2663  2837    85          H,  2,  7
  3  1709  1815    85      7  1248  1414   129      0  2007  2112    67
  5  1796  1738    87      8  3189  3168    79      1  4361  4566    61
  9  1822  1837    82     11  1296  1036   117      2  1040  1015    77
 10  1641  1685    87     12  2395  2430    88      3  4991  4815    60
 11  1939  2020    80     14  1545  1695   107      5  2574  2537    64
 12  1747  1798    91          H, 17,  6            6  2741  2786    64
 13  3125  3119    75      3  1939  2234   104      7  1094  1110    93
 14  1221  1287   110      5  1223  1242   121      9  3260  3154    69
 16  2041  2114    87      7  1827  1881   105     10  3202  3340    71
 17  1466  1579   116     10  1586  1431   110     11  1221  1165   105
 20  2166  2340   101     14  1860  2047   104     12  2283  2313    76
      H, 13,  6           15  2525  2591    89     13  6285  6171    65
  1  2557  2591    73          H, 18,  6           16  4457  4551    71
  2  1021  1100   107      1  2061  1897    96     17  1504  1542   104
  3  1320  1343    99      2  1639  1782   107     18  1200  1146   128
  4  3597  3578    70      3  2112  2310   100     20  4060  4325    81
  5   977   813   124      6  1817  1965   105     21  1542  1760   124
  6  2269  2295    79      7  2720  2723    86     22  1174  1163   148
  7  1414  1326    93     11  2291  2247    91     23  2817  2758    98
  8  4061  4316    70          H, 19,  6                H,  3,  7
  9  2700  2574    73      1  1382  1182   124      1  2843  2926    65
 10  1295  1309   127      2  2008  1840    96      2  1954  2045    64
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COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC    SIG
     H,  3,  7
 3  3264  3211    61
 4  4531  4526    58
 5  1658  1684    73
 6  3836  3868    61
 7  1151  1070    68
 8  5747  5823    63
 9  4414  4132    67
12  1880  1977    79
13  1651  1694    90
14  1341  1372   102
15  2361  2289    81
18  1978  2086    92
19  2392  2305    87
21  1342  1364   134
22  1952  2049   112
     H,  4,  7
 0  7777  8052    56
 1  1735  1735    71
 2  4502  4677    57
 3  1341  1460    63
 4  3859  3988    60
 5  2172  2105    70
 6  1574  1547    75
 7  2191  2118    71
 8  2069  2142    76
 9   921   871   134
10  4623  4613    64
11  3283  3167    69
12  2538  2676    76
14  3272  3295    72
16  2386  2449    82
17  1827  1986    90
19  2011  2076    96
     H,  5,  7
 1  1918  1967    67
 2  2250  2211    63
 3  1682  1642    77
 4  2592  2651    63
 6  3562  3704    65
 7  1934  1898    76
 8  3738  3883    66
 9  3050  2887    69
10  2265  2283    75
11  1309  1291    85
13  1479  1552    91
16  2237  2095    85
17  1317  1249   115
19  2572  2545    86
20  1297  1424   125
21  1183  1209   149
22  1442  1481   125
     H,  6,  7
 0  1938  1987    65
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 H   KFO    FC    SIG
 2   681   126   114
 3  4703  4770    62
 4  2773  2818    65
 5  1298  1220    89
 6   976   989   109
 7  3547  3563    67
 8  2790  2902    73
 9  1490  1519    98
12  1689  1760    82
13  2960  2874    72
14  2577  2502    77
18  3418  3488    83
19  1765  1860   103
22  2761  2873   104
     H,  7,  7
 1  1635  1634    78
 2  2610  2697    63
 3  4292  4262    66
 4  3315  3427    65
 5  3088  3094    69
 6  2313  2376    74
 7  3237  3313    70
 8  3078  3231    72
 9  1920  1874    84
10  2290  2256    76
11  1003  1001   106
13  2668  2740    75
14  2933  3180    74
15  4957  5226    68
16  1358  1266   105
18  1989  2022    99
19  1263  1333   135
20  1523  1543   118
     H,  8,  7
 0  3189  3080    62
 3  3520  3543    70
 4  1722  1692    75
 5  1666  1623    81
 7  3520  3386    68
 9  1495  1646    85
11  2014  2132    78
13  1230  1116   107
14  1349  1356   110
16  1121  1337   129
19  1973  2065   109
21  1915  2067   111
     H,  9,  7
 3  1153  1272   110
 4   870  1027   119
 9   795   931   140
12  2241  2189    77
14  1635  1666    89
16  1860  1959    95
     H, 10,  7
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 H   KFO    FC    SIG
 0  1761  1717    76
 1  1610  1734    81
 2  1082  1003    98
 3  3092  3124    74
 4  3009  2981    69
 5  2619  2611    76
 6   897   859   126
 7  4926  4949    71
 8  3128  3242    70
11  1953  2100    79
12  1656  1611    90
14  2397  2493    78
17  1804  1922    97
18  4337  4400    79
19  2072  2128    96
     H, 11,  7
 2  1023  1094   107
 3  2169  2083    83
 4  1860  1856    83
 5  2913  2727    76
 6  3825  3746    74
 7  2984  2860    73
 8  2080  2138    80
 9  1540  1528    97
10  3443  3496    73
11   998   914   127
14  1279  1024   100
15  4088  4150    73
16  1280  1362   120
     H, 12,  7
 0  4739  4598    66
 1  1794  1762    88
 2  5308  5371    67
 3  3904  3914    75
 4  1179  1188   113
 5  2084  2054    85
 7  1802  1738    93
 8  3104  3335    72
10  3325  3374    79
12  3423  3467    79
13  2911  2888    80
14  2370  2369    82
16  1058  1187   141
     H, 13,  7
 1  2085  2144    83
 2  1785  1840    84
 3  2305  2367    83
 4  1486  1527    94
 6  2737  2856    86
 7  1084  1256   120
 8  2075  2209    92
 9  3687  3723    79
10  1858  1892    90
11   920  1021   135
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COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC    SIG
     H, 13,  7
13  1055   824   126
16  1843  1898   101
     H, 14,  7
 0  2015  2002    83
 2  1930  1919    93
 4  1663  1607    92
 5  1700  1617   101
 6  2746  2865    82
 7  1686  1723   101
10  1960  1974    91
11  1224  1245   112
12  1196  1215   114
14  1210  1251   120
16  2324  2222    88
17   989  1033   158
     H, 15,  7
 1  1244  1332   117
 2  1655  1675    99
 4   858   804   157
 6  2112  2222    93
 9  3295  3271    80
16  2495  2400    91
     H, 16,  7
 0  2388  2330    87
 1  1874  1915    93
 2  2514  2341    84
 3  2731  2988    85
 5  2344  2509    92
 6  1135  1048   119
 7  1228  1175   130
 8  2005  1855    88
 9  1379  1268   117
10  1564  1632   114
13  3224  3378    85
14  1225  1359   127
     H, 17,  7
 1  1586  1555   104
 4  1151  1374   139
 9  2051  2179    99
10  1919  1987   102
     H, 18,  7
 0  2561  2607    90
 2  2250  2237    98
 6  2633  2525    87
10  2539  2595    94
     H, 19,  7
 1  1863  1865   103
 4  1992  1953   100
 6  1713  1723   111
 8  1430  1447   118
 9  2835  2804    91
     H, 20,  7
 0  3076  2982    95
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 H   KFO    FC    SIG
 1  1254  1326   142
 2  2260  2086   100
 3  2271  2232   104
 4  2031  2045   118
     H,  0,  8
 0  5639  5904    64
 1  7657  8387    66
 2  4519  4849    62
 3  2727  2580    70
 4  3991  4331    63
 5  2472  2444    70
 7  1404  1383    90
 8  4461  4735    66
10  2539  2543    81
11  9451  9240    70
12  3860  4181    74
14  3450  3505    76
15  2803  2877    76
18  3015  3050    89
19  2437  2530    89
21  3133  3199    93
     H,  1,  8
 1  3600  3726    70
 2  1266  1541    92
 3  3945  3941    65
 5   871   798   124
 7  3723  3542    68
 8  2243  2461    75
11  3500  3491    79
12  2074  1839    84
16  1142  1298   120
17  1802  1684    94
18   767   633   163
19  1588  1688   113
21  1541  1528   136
     H,  2,  8
 0  1942  1913    74
 1   742   654   154
 2  3034  2986    63
 4  1524  1653    82
 7  2629  2428    71
 9  1621  1512    89
10  2010  2019    86
12  2786  2785    79
13  1505  1434   107
14  2148  2128    85
     H,  3,  8
 3  2485  2421    71
 4  4638  4677    63
 5  1125  1253    97
 6  4691  4625    66
 7  2813  2849    70
 9  2033  2109    83
10  2226  2144    81
```

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 H   KFO    FC    SIG
11  1567  1640   108
14  2622  2763    80
15  2745  2583    74
16  1699  1718    89
17  1927  2046    88
20  2284  2289    98
21  2450  2475   100
     H,  4,  8
 0  4124  3983    66
 1  6148  6252    64
 2  3526  3528    62
 3  1159  1125    92
 4  3465  3434    66
 5  4693  4677    66
 6  2164  2066    75
 8  3499  3636    71
 9  1835  1862    93
10  2712  2582    79
11  5832  5835    71
12  3505  3351    71
14  2262  2445    85
15  2614  2628    80
16  1811  1690    95
21  2297  2345   101
     H,  5,  8
 1  4106  4181    68
 2  1173  1150    95
 4  5108  4971    64
 5  1746  1635    84
 6  3285  3115    68
 7  2939  2991    74
 8  3196  3280    74
 9  1892  2051    91
11  3486  3501    74
13  2160  2187    83
14  3456  3312    74
15  2008  2049    85
17  3138  3155    81
18  2426  2452    89
     H,  6,  8
 0  3452  3529    65
 2  3289  3315    67
 3  1006  1124   118
 4  3168  3241    70
 5  3402  3428    70
 6  1916  1919    84
 7  3916  3915    71
 9  1188  1323   117
10  2713  2745    77
12  3489  3450    74
13  2303  2325    81
14  2592  2333    77
15  1590  1604    95
17  2028  2071    99
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COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC    SIG
     H,  6,  8
18  1242  1250   127
     H,  7,  8
 1  1267  1179    96
 6  1259  1571   114
 8   859  1060   141
 9  1038  1104   117
10  1727  1900    89
13  1494  1574   115
14  1508  1656    95
15  1778  1869    93
17  1990  2047    94
20  1451  1368   121
     H,  8,  8
 0  2457  2450    73
 1  2317  2376    80
 2  2177  2154    72
 4  2736  2741    73
 5  7324  7135    68
 6  4913  4771    69
 7  1616  1573    95
 8  1553  1422    95
 9  3112  3063    83
10  3276  3121    73
12  1010  1146   147
14  1318  1315   109
15  2102  2086    88
16  4093  4178    76
17  1955  1973   101
     H,  9,  8
 1  2301  2457    81
 3  2874  2876    75
 7  2458  2523    82
 8  1422  1513   102
 9  3072  2904    79
10  1525  1476   106
11  1422  1622    97
13  3660  3743    77
14  2590  2764    81
17  4605  4648    76
19  1505  1315   133
     H, 10,  8
 0  1570  1613    88
 1  2958  2834    76
 2  3388  3248    69
 5  4979  4955    74
 8  1846  1921    96
 9  1182  1220   134
11  1646  1692    99
12  1859  1809    92
15  1568  1739   103
     H, 11,  8
 2  1019   863   109
 3  1997  1929    93
```

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 H   KFO    FC    SIG
 4  3067  3065    75
 6  2412  2334    88
 7  1813  1910    99
 9  1723  1644    99
10  1432  1314   105
11  1399  1451   119
14  2337  2247    85
     H, 12,  8
 0  1680  1690    85
 2  1991  1865    88
 3  1071  1141   123
 4  2985  2975    85
 5  4977  5034    79
 6  3725  3819    84
 7  2349  2390    93
 8  1917  1967   100
 9  1784  1708   101
10  2101  2170    92
15  1626  1544   112
16  2409  2373    92
17  1914  1998   100
     H, 13,  8
 2  2121  2226    86
 3  1116  1173   128
 4  2168  2178    90
 6  3144  3236    87
 7  2912  2837    86
 8  2107  1978   100
10  1464  1568   125
11  1832  1831    98
13  2007  2214   100
16  2403  2260    89
     H, 14,  8
 0  1902  1956   104
 1  3906  3860    80
 2  4264  4440    80
 4  1556  1523   123
 5  3325  3388    85
 7  1778  1660   107
 8  2087  1966    98
 9  2436  2342    95
10  1386  1012   115
11  2289  2369    89
12  3899  3910    78
14   796   853   165
     H, 15,  8
 1  1107   802   124
 4  1298  1219   118
 5  1300  1173   131
 7  2529  2357    93
 9  1767  1709   111
10  2061  2010   101
13   923  1155   165
     H, 16,  8
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 H   KFO    FC    SIG
 0   972   958   145
 2  2022  2023    94
 4  1665  1573   104
 7  1720  1825   109
 8  2358  2393    91
11  1375  1487   141
12  1354  1556   127
     H, 17,  8
 3  1677  1740   112
 7  2154  2212   100
 8  1427  1349   122
     H, 18,  8
 0  2578  2539    92
 1  3058  3004    92
 5  1715  1798   114
     H, 19,  8
 4  1479  1439   128
     H,  1,  9
 1  3405  3418    78
 2  2908  2930    72
 4  1434  1456    92
 5  1034   816   119
 6  1741  1674    81
 8  3954  3870    72
 9  3458  3410    79
10  2187  2150    90
14  1664  1572    98
16  1948  1832    97
18  2414  2487    98
19  1654  1484   109
     H,  2,  9
 0  5696  5808    67
 1  3213  3196    76
 2  4878  4970    69
 3  3929  3835    71
 4  2260  2426    80
 6  2613  2669    72
 8  1214  1095   106
 9  4473  4260    78
10  5008  4893    76
12  2885  2846    89
13  5441  5383    79
14  2826  2781    82
19  2168  2012   101
     H,  3,  9
 1  4591  4607    73
 2   824  1084   150
 3  3465  3387    73
 4  1051  1169   103
 6  2691  2569    74
 7  1288  1258   100
 9  2505  2564    86
10  2527  2568    88
11  1756  1834   108
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COLUMNS ARE 10FO,10FC,  10SIG

 H   KFO    FC    SIG
     H,  3,  9
12  1504  1559   120
14  1693  1691    97
16  2177  2278    89
     H,  4,  9
 0  4470  4563    69
 1   858   868   144
 2  2716  2709    74
 5  3440  3223    73
 6  1617  1440    94
 8  3340  3199    73
 9  2467  2521    87
10  3129  3098    88
11  3269  3407    82
12  2182  1947    91
14  1501  1535   105
16  1555  1582   106
18  1562  1580   109
19  2116  2224   110
     H,  5,  9
 2  1968  2054    79
 3  2651  2517    75
 4  1557  1449    95
 6  1748  1667    90
 8  3607  3537    74
 9  1609  1615   113
11  1438  1323   110
13  2132  2206    94
17  2042  2098    93
18  1660  1787   112
19  2373  2320    99
     H,  6,  9
 0  1117  1218   111
 1  1279  1051   104
 2  3559  3546    71
 3  4924  4783    71
 4  2573  2538    78
 6  2457  2431    81
 7  4458  4377    74
 8  3927  4055    77
10  1504  1292   111
12  2085  2026    95
13  2219  2395    90
14  2046  1975    85
17  1149  1179   136
18  2875  2742    85
     H,  7,  9
 1  1346  1219   103
 2  2632  2683    77
 3  2688  2770    78
 5  2084  1928    87
 6  2791  2707    77
 7  2833  2798    83
 8  1127  1103   142
```

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 H   KFO    FC    SIG
11  1112  1019   144
12  2790  2570    84
15  3540  3538    81
16  3949  3986    82
17  1398  1283   110
     H,  8,  9
 1  1551  1516   101
 2  1644  1790    94
 3  1701  1699    92
 4  2296  2199    81
 6  1986  1854    91
 8  2336  2489    94
 9  1448  1255   114
11  2111  2251    89
     H,  9,  9
 1  1935  1924    91
 2  1893  1996    98
 6  1810  1791   101
 8  1496  1341   121
 9   928   906   164
15  1325  1372   117
16  2247  2174    94
     H, 10,  9
 2  3009  3010    80
 3  3840  3797    77
 4  2836  2808    82
 6  3266  2928    84
 7  5191  5204    83
 8  4244  4200    81
12  1454  1145   110
     H, 11,  9
 3   855   587   162
 4  2863  2937    88
 5  2408  2503    94
 7  1892  2052   121
 8  1288  1411   153
 9   902   849   181
14  2513  2426    87
15  4008  3962    79
     H, 12,  9
 0  5058  5123    81
 3  2757  3082    87
 4  2939  2931    87
 5  3043  3155    89
 7  2712  2707    92
 8   906   473   162
10  3205  3171    84
11  1090   819   134
14  2047  1966    98
     H, 13,  9
 2  2072  1923    94
 3  2208  2344   104
 8  3063  3046    88
 9  1712  1655   111
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 H   KFO    FC    SIG
11   976   955   158
13  2124  1913    97
     H, 14,  9
 0   970   904   152
 3  1666  1542   102
 4  2235  2134    98
 5  1931  1852   113
 6  2479  2411   102
 7  1741  1607   110
 8  1455  1481   125
 9  1443  1605   132
     H, 15,  9
 1  1212  1266   125
 2  1103  1230   149
 4  2047  2053   106
 6  1107   960   150
 8  2338  2336    99
 9  1895  1734   118
     H, 16,  9
 0  3664  3467    86
 1  1043   879   160
 3  2421  2449    97
 4  1592  1558   116
 5  2741  2594    97
     H, 17,  9
 3   991  1024   166
     H,  0, 10
 0  2789  2906    81
 1  1966  2082    98
 2  4396  4273    74
 9  1660  1648   111
11  4854  4717    85
12  3774  3987    89
13  1368  1363   137
15  2505  2428    90
     H,  1, 10
 1  3441  3320    90
 3  2819  2763    93
 4  2059  1902    85
 6  2246  2187    91
 7  1271  1271   123
 9  2203  2035    92
10  1783  1784   108
11  2009  1921   108
12  1532  1596   130
13  2340  2105   100
     H,  2, 10
 1   855   552   149
 2   970  1246   142
 4  1240  1086   104
 6  1884  1986    92
 7  2051  1982    93
 9  2136  2049    96
12  1870  1856   108
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COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO     FC    SIG        H   KFO     FC    SIG        H   KFO     FC    SIG
     H,  2, 10                10  2427   2413     98        9  2128   1979    102
 13  1903   1626    114       12  2224   2172     94       11  1240   1292    154
 16   880    850    151       15  1424   1283    122       13  3075   2875     97
     H,  3, 10                     H,  9, 10                    H,  3, 11
  3  1020    993    115        1  1135   1067    126        1  2743   2861     92
  4  3636   3696     77        2  1368   1560    113        3  1582   1456    101
  6  3437   3274     78        3   829    914    171        4   976    984    154
  7  2048   1996     96        4  1804   1737    100        6  1399   1268    115
 10  2070   1816    100        7  1190   1067    141        7  1963   1933    112
 11  1116    770    145        9  2618   2639     97       11  2965   2694     93
 12  1748   1722    111       12  1370   1398    133            H,  4, 11
 14  2916   2716     86       13  1616   1669    112        0  2870   2812     88
 15  1950   2036     96       14  2477   2323     90        8  2257   2138    100
     H,  4, 10                     H, 10, 10                9  1568   1645    123
  0  2525   2672     86        2  1639   1580    104       10  2691   2452    102
  1  1707   1799     99        3  2844   2794     83       11  2849   2571    104
  2  1532   1615    109        5  2080   2083    101            H,  5, 11
  5  2213   1960     88        7  2000   2052    107        2  1983   1894    102
  6  1250    803    111        8   929    863    167        3  1914   1818     97
 10  1774   1811    108       13  1141   1296    148        7   923    875    168
 11  2993   2909     93            H, 11, 10               12  1561   1685    132
 12  1692   1610    108        4  2988   2996     89            H,  6, 11
 15  1714   1723    113        6  2051   2013    108        0  3257   3352     84
     H,  5, 10                     H, 12, 10                3  1690   1789    102
  1  3451   3513     79        4  2021   1851    108        4  1212   1232    130
  4  2523   2494     84        5  2978   2832     91        7  1918   1838    108
  9  3389   3352     88        6  2635   2458     98        8  1379   1171    119
 14  2995   2851     85        9  1345   1285    144       10  3000   2827     98
     H,  6, 10                     H, 13, 10               13  1254   1340    138
  1  1794   1866     98        2  1397   1463    124            H,  7, 11
  4  2218   2208     91        6  2076   2044    114        1   917   1017    159
  6  3025   3047     80        9  1342   1365    149        2  1975   1927     98
  7  2227   2261     95            H, 14, 10                7  2546   2464     98
  9  2469   2332     97        2  2487   2521     98        8  2829   2753     95
 10  1509   1480    120        3  2354   2467    100       11  2009   1782    111
 11  1122    816    142        5  1940   1872    112            H,  8, 11
 12  2807   2590     93        7  3245   3214     97        0  2789   2670     91
 13  2724   2591     91            H, 15, 10                1  1902   1754    103
 16  1551   1454    111        1  1710   1413    112        5  1162   1105    141
     H,  7, 10                 5  2075   1882    116       10  1711   1556    124
  1  2542   2527     67            H,  1, 11                    H,  9, 11
  3  1373   1446    108        1  1830   1621    106        2  1197   1378    137
  5  1894   1837    112        2  2953   2910     88        4  1864   1695    111
 11  1310   1119    130        4  1471   1299    117        6  1863   1861    111
 15  2032   2160     96        6  2542   2447     91        8  2397   2166    102
     H,  8, 10                 8  3833   3561     87            H, 10, 11
  0  2672   2556     84        9  1372   1262    130        0  5342   5137     85
  1  1390   1263    110       11  1880   1649    110        3  2249   2087     98
  2  1687   1582     95            H,  2, 11                4  1215   1109    142
  3  1112   1109    118        0  2127   2375    100        7  2441   2368    105
  4  2718   2686     88        2  1480   1441    115        8  1479   1361    135
  5  4032   4173     82        5  1181    878    126            H, 11, 11
  6  3169   3033     85        6  3158   3159     89        4  1505   1407    133
  9  2625   2527    101        8  1228   1092    144        7  2101   1967    113
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COLUMNS ARE 10FO,10FC,  10SIG

  H   KFO     FC    SIG        H   KFO     FC    SIG        H   KFO     FC    SIG
     H, 12, 11                 8  2170   2132    110        1  1610   1450    119
  1  1676   1631    119            H,  2, 12                2   979    949    169
  5  3380   3294     99        0  1212    990    129        8  2247   2013    114
     H,  0, 12                 2   890    970    173            H,  6, 12
  0  3638   3299     90            H,  3, 12                0  1790   1701    116
  4  5562   5108     88        4  3550   3221     90        2  2396   2313    107
  5  1854   1774    130            H,  4, 12                6  1725   1595    121
  6  3254   2849     96        0  1292   1225    140        8  1153   1265    171
  8  2774   2665    106        1  1240   1024    128            H,  7, 12
 10  2394   2354    112        2  2534   2480    100        6  2032   1783    108
     H,  1, 12                 4  2909   2668     96            H,  8, 12
  1  1753   1576    126        5  1661   1602    127        2  5916   5462     84
  4  3378   3258     98        8  3088   2980     96        5  2076   1919    116
  6  2470   2366    103            H,  5, 12
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