STRUCTURES OF ORGANO-TRANSITION METAL COMPLEXES

ANNUAL SURVEY COVERING THE YEAR 1976*

Michael I. Bruce

Department of Physical and Inorganic Chemistry, University of Adelaide, Adelaide, South Australia, 5001.

INTRODUCTION

This survey consists of two sections comprising a collection of briefly annotated diagrams ordered according to ligand structural type, and a molecular formula index of structure determinations published during the year under review. During 1976, some 450 structures of organo-transition metal complexes were determined by diffraction methods, considerably more than appeared during the previous year.

In addition to conventional organometallics, i.e. those compounds containing at least one metal-to-carbon bond (apart from cyano complexes), I have also summarised data pertaining to other complexes of interest to many organometallic chemists. These include hydride and borohydride derivatives, nitrosyls, dinitrogen, aryldiazo and related complexes, and binary metal-tertiary phosphine complexes.

This is the last compilation in this series, and it is of interest to note that about 2475 complexes have featured in the eleven articles which survey the years 1968 - 1976 [1]. This represents nearly 83% of just over 3000 structure determinations of organo-transition metal complexes which have been published since the study of $Fe_2(CO)_9$ reported in 1939 by Ewens and Powell [2]. Readers will appreciate the difficulty of preparing

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*Annual survey 1975: M.I. Bruce, *J.Organometallic Chem.*, 126 (1977) 1-149.

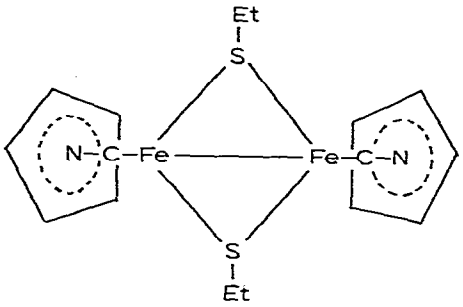
an article of this type to be competitive in publication time with other related listings, such as those appearing in The Chemical Society's reports on *Organometallic Chemistry* and *Molecular Structures by Diffraction Methods*, when the last relevant issues of many journals appear in most southern hemisphere libraries during the second half of April!

REVIEWS AND OTHER PAPERS OF GENERAL STRUCTURAL INTEREST

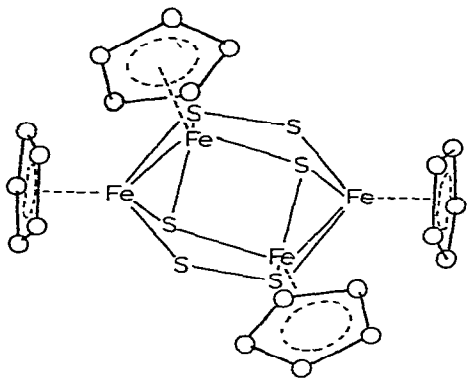
Further volumes in the Chemical Society's Specialist Periodical Report *Molecular Structure by Diffraction Methods* cover the period April 1973 - March 1975 (for X-ray studies), and to September 1975 (neutron diffraction) or August 1975 (electron diffraction) [3]. The latest volume in *Molecular Structures and Dimensions* now extends coverage to mid-1975 [4].

Following the discussion of twist angle calculations mentioned last year, another paper presents a more systematic set of definitions, and concludes that twist angles should only be used when it is appropriate to do so, carefully considering which definition is the most useful, and defining it precisely in context [5].

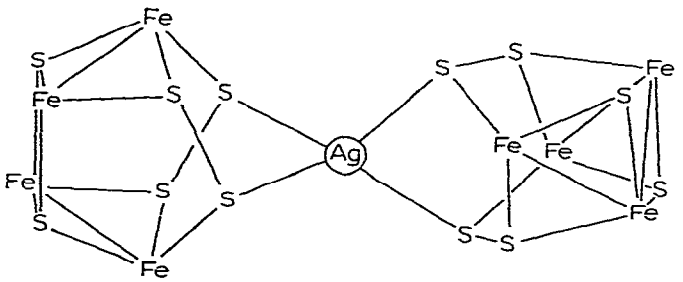
A volume in the *Progress in Inorganic Chemistry* series contains several review papers presented at a symposium held during the 167th National Meeting of the American Chemical Society at Los Angeles in April 1974, to honour Professors F.A. Cotton and L.F. Dahl, some of which are of relevance to this survey. Cotton presents some new observations in the old field of metal carbonyl studies, with particular relevance to his studies of iron carbonyls and their reaction products, and the occurrence of unsymmetrical bridging, and semi-bridging, carbonyl groups [6]. In a survey of seven and eight-coordinate molybdenum compounds, Lippard includes a discussion of the synthesis and structure of molybdenum isocyanide complexes [7]. The versatility of sulphur as a ligand in organometallic cluster complexes is the subject of an article by Vergamini and Kubas [8], who describe the structures of $Fe_2S_2(SEt)_2(C_5H_5)_2$, $Fe_2(CN)_2(SEt)_2(C_5H_5)_2$ (I), $Fe_4S_6(C_5H_5)_4$ (II) and the $\{[Fe_4S_6(C_5H_5)_4]_2Ag\}^{3+}$ cation (III). Only the first of these complexes has been reported previously.



(I) $[Fe(CN)(SEt)(C_5H_5)]_2$



(II) $Fe_4S_6(C_5H_5)_4$



(III) $\left\{[Fe_4S_6(C_5H_5)_4]_2Ag\right\}^{3+}$
C_5H_5 groups omitted

The structures of, and bonding in, 4*f* and 5*f* organometallic compounds (organolanthanides and -actinides) has been reviewed [9]. In this excellent summary, it is shown that the bond lengths in isostructural complexes show changes which correlate with changes in atomic radii. Ground

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state geometries often reflect a delicate balance between energy released by increasing coordination number, and energy lost by intramolecular repulsion, as shown by the bonding modes found in $UR(C_5H_5)_3$ (R = C_3H_5, C_4H_7 or C_5H_5, for example).

Phase transitions in organometallic molecules have been related to staggered-eclipsed transformations in the case of $MnRe(CO)_{10}$ [10], order-disorder transitions in $Ni(C_5H_5)_2$ [11], and to structural changes in ferrocenes [12].

A paper describing the structure of $Ru_3H(CO)_{10}(C{=}NMe_2)$ usefully summarises data pertaining to the location of hydrogen atoms in cluster complexes. Generalisations relating to structural data of various types of hydrido-bridged metal–metal bonds arising from these comparisons include (a) metal–metal bond distances increased by single, unsupported μ_2-hydride or μ_3-hydride ligands; (b) complexes containing one, two or three μ_2-hydrido ligands bridging one metal–metal bond can be looked upon as containing protonated single, double or triple metal–metal bonds, respectively; (c) where other bridging ligands are present, the effects of a μ_2-hydride ligand cannot easily be determined unless some knowledge of the effect of the bridging ligand is also available [13].

ELECTRON DIFFRACTION RESULTS

Organo-transition metal complexes studied by electron diffraction methods included:

$Co(C_5H_5)_2$ The average structure which has eclipsed rings (D_{5h}), is compared with those of other first-row metallocenes. The larger C–C vibrational amplitude than was found for $Fe(C_5H_5)_2$ and $Ni(C_5H_5)_2$, and predicted from a molecular force field, confirms the presence of a dynamic Jahn–Teller effect. The C–H bonds are bent towards the metal by 2.1° from the ring plane. Distances: Co–C, 2.119(3); C–C, 1.429(2); C–H, 1.111(8)Å [14].

$Ni(NO)(C_5H_5)$ The half-sandwich structure was confirmed, with the hydrogen

atoms lying in the plane of the C_5 ring. The Ni-N-O group is linear. Distances: Ni-C, 2.128(5); Ni-N, 1.58(1)Å [15].

$Co(GeH_3)(CO)_4$ The Co-Ge bond [2.416(4)Å] is shorter than the sum of covalent radii, but considerably longer than that in $Co(GeCl_3)(CO)_3$. The difference between Co-C(ax) and Co-C(eq) is very small [16].

$Re(MH_3)(CO)_5$ (M = C, Si or Ge) All three complexes contain almost identical $Re(CO)_5$ groups, with equatorial CO groups bent towards the MH_3 group by between 4-7°. The Re-M bonds are 2.308(17) (M = C), 2.562(12) (Si) and 2.628(6)Å (Ge), respectively [17].

NEUTRON DIFFRACTION RESULTS

An earlier neutron diffraction study of $HW_2(CO)_9(NO)$ revealed CO-NO disorder. The related complex $HW_2(CO)_8(NO)[P(OMe)_3]$ was studied by X-ray and neutron diffraction and revealed a slightly asymmetric W-H-W bond. Comparisons with data for other complexes leads to the suggestion that in these systems it is the W-W overlap distance which remains constant, rather than the W-H bond length [18].

Electronic Structures from Combined X-ray-Neutron Diffraction Studies The neutron diffraction study of $Cr(CO)_6$ reported in 1975 [19] has been complemented by an X-ray study carried out at 74K [20]. All Cr-C-O bonds were considered to be equivalent. The final atomic charges are: Cr, 0.15; C, 0.09; O, -0.12, which are small values which contrast with quantum chemical calculations, but agree with the electroneutrality principle. The electron density around chromium is not spherically symmetrical, with about 25% of the *d* electrons in e_g symmetry orbitals, and 75% in t_{2g} orbitals. The electron density maps allow direct "visualisation" of the classical σ-bonding, π-backbonding scheme, with incomplete occupation of the CO π^* orbitals, and a net transfer of 0.3 e per CO group.

Related to the above study are careful determinations of the structure of $[Ni(C_5H_5)]_2C_2H_2$ at 78K and 298K [21]. The acetylene is *cis*-bent,

with a C-C distance of 1.341(6)Å. Electron density maps are in agreement with a linear Ni-Ni bond, with a double maximum along the axis, and with the metal-acetylene interaction concentrated in the π^* region of the acetylene. In addition, the C-C bond appears bent with sp^2 σ hybrid orbitals pointing at an angle with the bond axis.

TRENDS IN 1976

The large numbers of complexes whose structures are determined by X-ray diffraction methods as a routine method of characterisation, confirm the power of this method of elucidating stereochemistries of molecular arrays. The rapid advances in areas such as metal cluster chemistry, the reactions and interconversions of metallo-borane and -carborane derivatives, and the stabilization of new and unusual intermediates, have been largely due to the increasing application of molecular structure determination by diffraction methods.

During the year, a variety of ligands and complexes of note were reported, and some of these which more than usually interested this reviewer were: the increasing number of transition metal complexes in which an alkali metal interacts with ligands and transition metals, including the fascinating phenylnickel-dinitrogen derivative $[Ph(NaOEt_2)_2(Ph_2Ni)_2N_2NaLi_6(OEt)_4OEt_2]_2$; phosphoro- and arseno-benzene (PhE=EPh, E=P or As) complexes; the first Co_3Si cluster to be confirmed; the series of unusual derivatives obtained from $(\eta\text{-}C_5H_5)Me(CO)_2(EPhCl_2)$ (E=P or As) and $Fe_2(CO)_9$; and the ever-increasing numbers of polynuclear carbonyl and carbido-carbonyl complexes of the Group VIII metals. Indeed, it is true to say that few, if any, of the studies of cluster complex chemistry, would have been resolved without the help of the crystallographer.

STRUCTURAL DIAGRAMS

Diagrams used in this survey have been drawn using the published

representation of the molecule as a basis.* Organic ligands have been arranged in order of increasing number of carbon atoms attached to the metal atom, and denoted by the η symbol. Where two or more different groups are present, the complex will be found under the highest η number. Thus, diagrams for $Mn(CO)_2(Ph_2C{=}C{=}O)(C_5H_5)$ and $ZrCl_2(\eta^3\text{-}C_{13}H_9)(\eta^5\text{-}C_{13}H_9)$ are in the η^5 section. Within each section, further ordering has usually been on the basis of Periodic Group. Appropriate notes in each section draw attention to any unusual features noted in the structure, to the origin of the complex if not self-evident, and to any structural comparisons, if made. Reference numbers in square brackets [ ] refer to the list at the end of this article. The headings differ slightly from those used in the 1975 survey:

η^1-Ligands

(a) Simple carbonyls

(b) Carbonyl hydrides and halides

(c) Carbonyls containing N-donor ligands

(d) Carbonyls containing P- or As-donor ligands

(e) Carbonyls containing S-donor ligands

(f) Carbonyls containing anionic ligands

(g) Thiocarbonyls

(h) Isocyanide complexes

(i) Carbenes and carbyne complexes

(j) Alkyls, aryls and acyls

(k) Complexes containing chelating η^1-ligands

η^2-Ligands

(a) $(2\eta^1)$-Ligands (Metallocycles)

(b) Olefin complexes

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*I am grateful for the tireless efforts of the Elsevier draughtsman, who has struggled over the last few years to convert my often ambiguous drawings into the finished diagrams.

(c) Alkyne complexes

(d) Olefin (or alkyne) halides

(e) Complexes containing other three-membered rings

η^3-Ligands

(a) $(\eta^1+\eta^2)$-Ligands

(b) η^3-Allyls

η^4-Ligands

(a) $(\eta^1+\eta^3)$-Ligands

(b) $2\eta^2$-Ligands

(c) η^4-Diene complexes

(d) η^4-Trimethylenemethane

(e) η^4-Cyclobutadiene

η^5-Ligands

(a) Cyclopentadienyls

(b) Cyclopentadienyls containing halide or anionic ligands

(c) Cyclopentadienyl containing other hydrocarbon ligands

(d) Cyclopentadienyl containing CO, CNR, PR_3 or NO ligands

(e) Cyclopentadienyl-dinitrogen complexes

(f) Substituted ferrocenes

(g) Other η^5-ligands

η^6-Ligands

(a) Arenes

(b) Other η^6-ligands

η^7-Ligands

η^8-Ligands

η-Heteroatom Ligands

Alkali-Metal Derivatives

Silver Complexes

Complexes Containing Metal–Metal Bonds

(a) Complexes containing transition–metal–Main Group metal bonds

(b) Binuclear transition metal complexes

(c) Binuclear complexes containing bridging hydrocarbon ligands

(d) Binuclear complexes containing other bridging ligands

Polyhedral cluster complexes

(a) Polyhedral metal carbonyl clusters

(b) Polyhedral clusters containing η–hydrocarbon groups

(c) Polyhedral clusters containing Main Group elements.

(d) Polyhedral metalloborane complexes

(e) Polyhedral metallocarborane complexes

Hydride and Borohydride Complexes

Nitrosyls

Dinitrogen, Aryldiazo and Aryldiimine Complexes

Binary Transition Metal–Tertiary Phosphine Complexes

ABBREVIATIONS

acac	acetylacetonate
apo	acetophenoneoxime
biim	2,2'-biimidazole dianion
cod	cycloocta-1,5-diene
Cy	cyclohexyl
diars	1,2-bis(dimethylarsino)benzene
diop	2,3-*O*-isopropylidene-2,3-dihydroxy-1,4-bis(diphenylphosphine)-butane

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dmg	dimethylglyoximate
dmpe	1,2-bis(dimethylphosphino)ethane
dmt	1,3-dimethyltriazenido
dppe	1,2-bis(diphenylphosphino)ethane
dppm	bis(diphenylphosphino)methane
dtt	1,3-di(*p*-tolyl)triazenido
en	1,2-diaminoethane
fn	fumarcnitrile
imH	imidazole
Meim	1-methylimidazole
mes	mesityl
nas_3	$N(CH_2CH_2AsPh_2)_3$
nbd	norbornadiene (bicyclo[2.2.1]heptadiene)
np_2o	$N[CH_2CH_2P(O)Ph_2](CH_2CH_2PPh_2)_2$
np_3	$N(CH_2CH_2PPh_2)_3$
$oepH_2$	octaethylporphyrin
phen	1,10-phenenthroline
pic	picoline
pp_3	$P(CH_2CH_2PPh_2)_3$
py	pyridine [4-Xpy ≡ 4-X-substituted pyridine]
pz	pyrazolyl
SacSac	dithioacetylacetonate
salen	ethane-1,2-salicylideniminato
salophen	N,N'-*o*-phenylenebis(salicylaldiminato)
tcnq	tetracyanoquinodimethan
thf	tetrahydrofuran
tmed	N,N,N',N'-tetramethylethylenediamine [1,2-bis(dimethylamino)ethane]
tol	tolyl
tpp	*meso*-tetraphenylporphin
trop	tropolonato

TABULATED STRUCTURAL DATA (TABLES 1 AND 2) (pages 417 and 425)

Some structural data of general interest has been collected in Tables 1 (metal-metal bond lengths) and 2 (parameters of coordinated NO), and supplements similar compilations in previous articles [1].

SUMMARY TABLES 3 AND 4 (pages 426 and 474)

These Tables list most complexes whose structures have been reported during 1976, together with a few appearing in late 1975. The arrangement (in columns) is as follows:

1. *Reference number*, referring to the structural diagram in the preceding section.
2. *Molecular formula*, arranged in order of increasing C and H content. Other elements follow in alphabetical order of symbol. Ionic complexes are listed under the *relevant* ion, e.g. $Na_2Fe(CO)_4.1.5C_4H_8O_2$ appears as $C_4FeO_4^{2-}.2Na^+.1.5C_4H_8O_2$, and molecules of solvation, if present, are listed last.
3. *Structural formula*, listed as far as is practicable, with metal atoms first, followed by attached ligand in order of increasing degree of electron donation. Thus, for some commonly found groups, the order is: M,H,M' (Main Group or Transition Metal)
   X (monodentate anionic ligand), R (σ-alkyl, aryl, etc.)
   η^1-ligands, ER_3 (E = N, P, As, Sb), SR_2, acac (and anionic bidentates), NO
   η^2-ligands (olefin, alkyne)
   η^3-ligands (allyl, enyl)
   η^4-ligands (diene, cyclo-diene)
   η^5-ligands (dienyl, cyclo-dienyl)
   η^6-ligands (triene, arene)
   η^7-ligands (cyclo-trienyl)
   η^8-ligands (cyclo-tetraene)

Of necessity, this order cannot be followed in all cases, particularly with cluster complexes.

4-8 Crystal data, comprising *crystal class*, *space group*, Z and *unit cell dimensions* (in Å and degrees).

9-11 Number of *intensity data* (observed reflections) used in structural refinement, and lowest R value reported (as %). Many reports quote both conventional (R) and weighted (R_w) values: both are listed where given.

12 *Miscellaneous notes*, often relating to low temperature determinations, etc. Here the absolute temperature at which data was collected, or cell constants determined, is listed. Other abbreviations used:

CD Cell data only given

ED Electron diffraction study

ND Neutron diffraction study

SD Structural diagram only, which may be accompanied by some bond parameters.

Other comments are given in appropriate footnotes.

13 *Reference number*, relating to the list of references at the end of the Survey.

APPENDIX (page 480)

Several structures which were noted after completion of the main part of this article are listed in an Appendix at the end of the section on tertiary phosphine complexes.

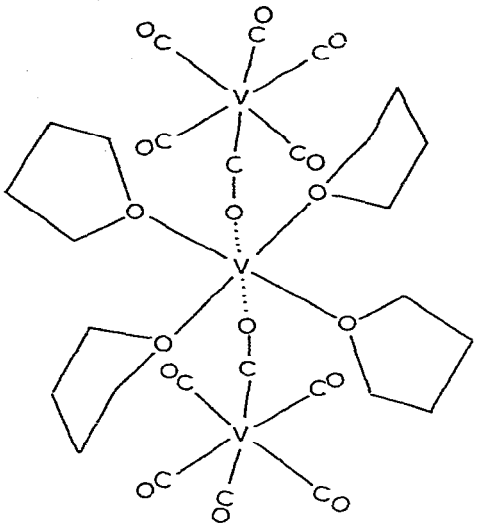
The following complexes are more correctly located in the sections indicated: (*135*) and (*323*) in Carbenes; (*321*) in Chelating η^1-ligands; (*334*) and (*335*) in Dienes; (*392*) in Three-membered rings; and although complexes (*393*), (*394*), (*395*) and (*406*) contain metal-metal bonds, they are strictly complexes containing metal sequences rather than closed clusters.

STRUCTURES ORDERED BY TRANSITION METAL

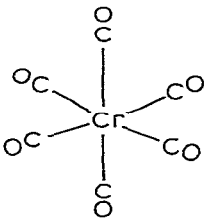
η^1-LIGANDS

(a) *Simple carbonyls*

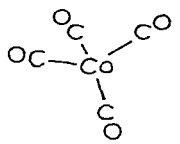
(*1*) From $V(CO)_6$ + thf; axial V–C–O from $V(CO)_6$ coordinates to planar $V(thf)_4$ to give first M–O–C–M bridge: V–O(CO) 2.079, V–O(thf) 2.170Å [38]. (*2*) Direct visualisation of classical σ,π bonding scheme from charge density distribution at -196° [20]. (*3*) Distorted tetrahedral Fe, with 3 C–Fe–C *ca.* 105°, one of 129.7°; Na coordinated by 2 dioxan, 4 O from 4 different $Fe(CO)_4$ groups to give polymeric network; Na(2) interacts with O from 2 dioxan, and at long range with C–Fe–C, resembling allyl, to give $Na_2[Fe(CO)_4]_2$ "cluster"; distortion of



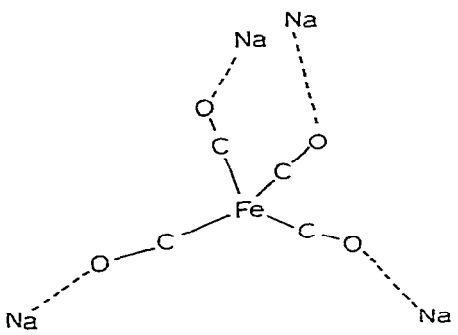
(1) $[V(thf)_4][V(CO)_6]_2$



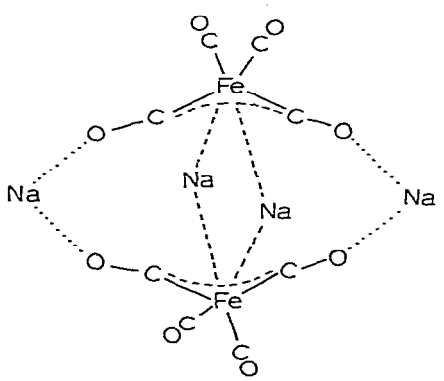
(2) $Cr(CO)_6$



(4) $[Co(CO)_4]^-$



(3) $[Fe(CO)_4]^{2-}$
Na salt.
dioxan solvate

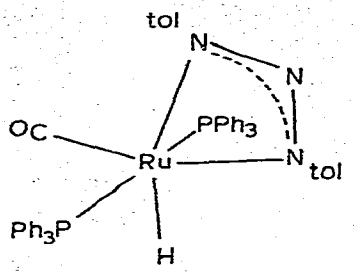


The loose cluster $(Na^+)_2[Fe(CO)_4]_2^{2-}$
in $Na_2Fe(CO)_4$

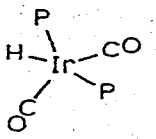
$Fe(CO)_4$ results from ion-pairing, since with isolated Na^+ in $[Na(cryptate)]_2[Fe(CO)_4]$, the carbonyl anion has regular tetrahedral geometry [32]. (4) From $Co(SiMe_3)(CO)_4$ + $PMe_2(SiMe_3)$; tetrahedral anion, Co-CO 1.73Å [30].

(b) *Carbonyl hydrides and halides*

(5) Bidentate dtt, planar strained RuN_3 ring, with small difference in Ru-N 2.149, 2.179(3)Å, from *trans* H; Ru-H 1.8Å [389,390].



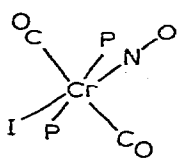
(5) $RuH(dtt)(CO)(PPh_3)_2$



P≡PPh_3

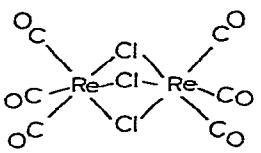
(6) $IrH(CO)_2(PPh_3)_2$

(*6*) Distorted TBP with axial H,P; equatorial ligands bend toward H; H located, gives Ir-H 1.64(5)Å [345].

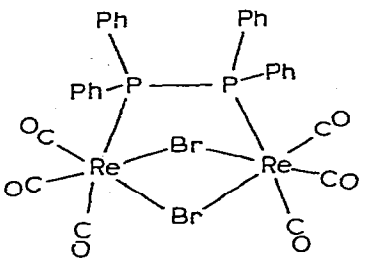


P≡$PMePh_2$

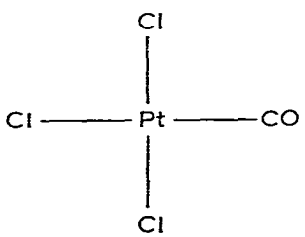
(7) $CrI(CO)_2(PMePh_2)_2(NO)$



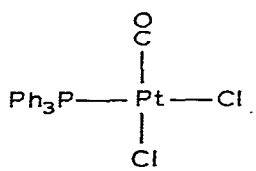
(8) $[Re_2Cl_3(CO)_6]^-$



(9) $[ReBr(CO)_3]_2P_2Ph_4$



(10) $[PtCl_3(CO)]^-$

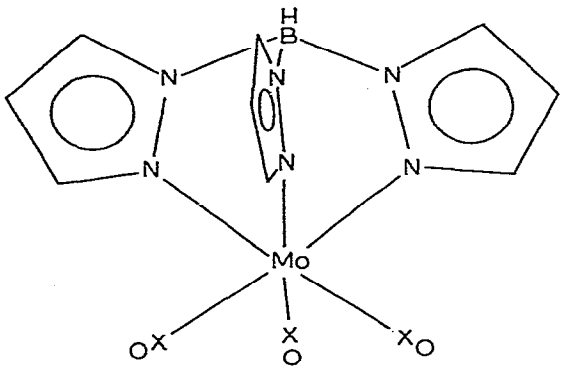


(11) *cis*-$PtCl_2(CO)(PPh_3)$

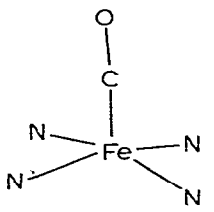
(*7*) From $[Cr(CO)_2(NO)(C_6Me_6)]^+ + I^-$, followed by addition of $PMePh_2$; CO group bent towards I [307]. (*8*) From $ReCl(CO)_5$ + $oepH_2$; first structure of anionic Re carbonyl halide [37]. (*9*) From P_2Ph_4 + $[ReBr(CO)_3(thf)]_2$; retains P-P bond, with P_2 ligand bridging 2 coordination polyhedra sharing common edge [311]. (*10*) Pt-CO 1.82(1); Pt-Cl 2.292 (av.)(*cis* CO), 2.289Å (*trans* CO); paper compares Pt-Cl bonds in a variety of complexes [22]. (*11*) Accurate determination, distances

reflect electronic *cis* influence of CO ligand: Pt-Cl 2.343(2) (*trans* to P), 2.276(1) (*trans* to CO); Pt-P 2.282(2); Pt-C 1.858(7)Å [210].

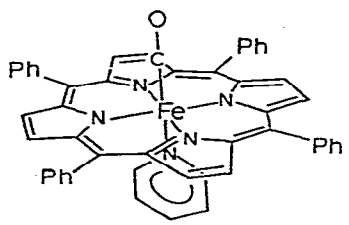
(c) *Carbonyls containing N-donor ligands*



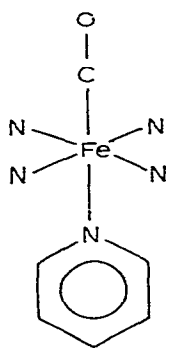
(12) $Mo(CO)_2(NO)[HB(pz)_3]$



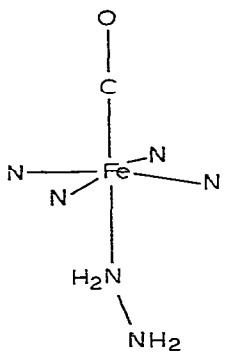
(13) $Fe(CO)(C_{22}H_{22}N_4)$



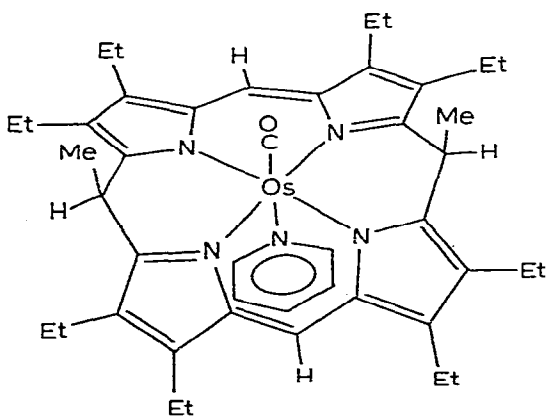
(16) Fe(CO)(py)(tpp)



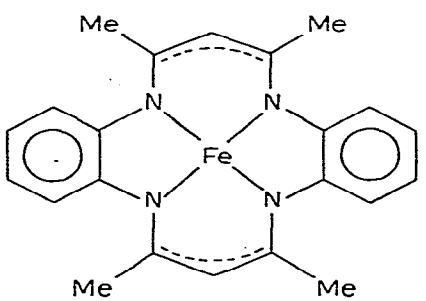
(15)
L = py



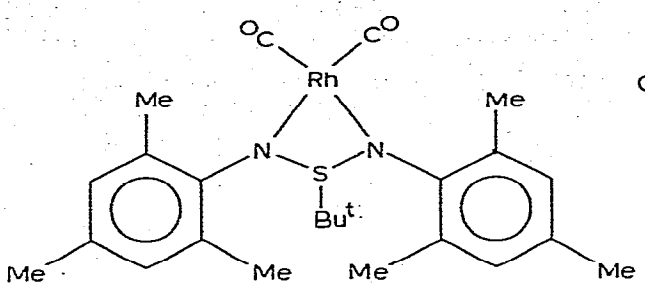
(14)
L = N_2H_4
$Fe(CO)L(C_{22}H_{22}N_4)$



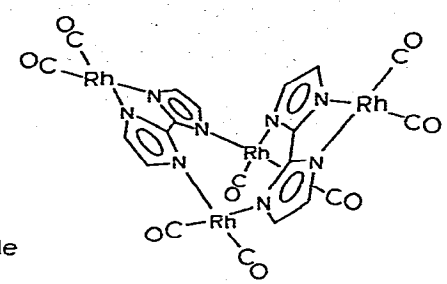
(17) $Os(CO)(py)(Me_2oep)$



Planar $Fe(C_{22}H_{22}N_4)$ group as found in (13), (14), (15)



(18) $Rh(CO)_2[Bu^tS(Nmes)_2]$

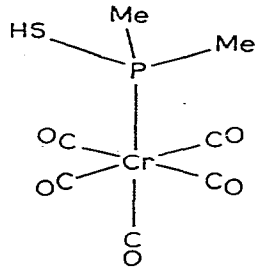


(19) $[Rh(CO)_2]_4(Biim)_2$

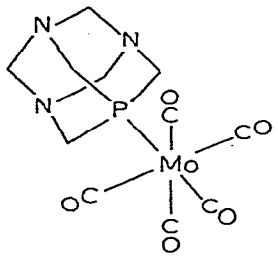
(*12*) CO-NO disorder; pz rings show twisted skewing relative to Mo-B axis [79]. (*13*)(*14*)(*15*) Comparison of macrocyclic Fe carbonyl complex with and without axial ligands; displacement of Fe from N_4 plane reflects combined effects of (i) strong Fe-CO bond, (ii) steric interactions within the macrocycle, with benzenoid rings tipped out of plane towards CO side; actual displacements: 0.29 (*13*), 0.11 (*14*), -0.05Å (*15*) [260]. (*16*) Linear Fe-CO contrasted with bent CO found for other carbonylated haemoproteins (see Appendix) [384]. (*17*) From reductive methylation of Os(CO)(py)(oep) + Na anthracenide + MeI; porphodimethene ligand folded 38.1° along line joining saturated C atoms; Os 0.18Å above N_4 plane, towards CO [367]. (*18*) Square planar Rh, N-Rh-N 70°, N-S-N 92.1°; long N-S bond in ligand [272]. (*19*) Biim bridges 3 Rh, while 2 Rh bridge 2 Biim; ligand rotated towards end Rh to give N-C$\cdots$C angles of 131, 116°; Rh-Rh distance short, indicates metal-metal interaction [see also (*158*)] [218].

(d) *Carbonyls containing P- or As-donor ligands*

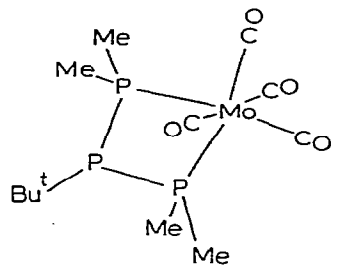
(*20*) By isomerisation of S-bonded $Cr(CO)_5(SPHMe_2)$ above 25°; Cr-P 2.344Å [40]. (*21*) Gives detailed geometry of phosphatriazaadamantane cage, and confirms P-bonded ligand [80]. (*22*) Non-planar MoP_3 ring, with PPP 85.0(1)°; other parameters unexceptional [108]. (*23*) Puckered 7-membered chelate ring, As-Mo-As 89.6(2)° [193]. (*24*) Pronounced steric hindrance gives distorted octahedral geometry; long W-P at 2.686(4)Å, P-W-CO 175.6° [160]. (*25*)(*26*) Slightly distorted



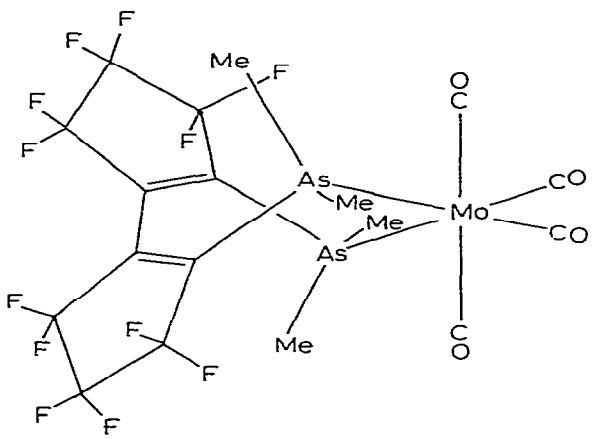
(20) $Cr(CO)_5[PMe_2(SH)]$



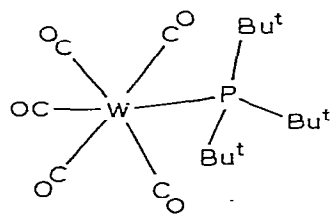
(21) $Mo(CO)_5[P(CH_2)_6N_3]$



(22) $Mo(CO)_4[Bu^tP(PMe_2)_2]$

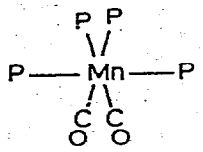


(23) $Mo(CO)_4(bif_{12}fars)$

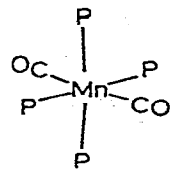


(24) $W(CO)_5(PBu_3^t)$

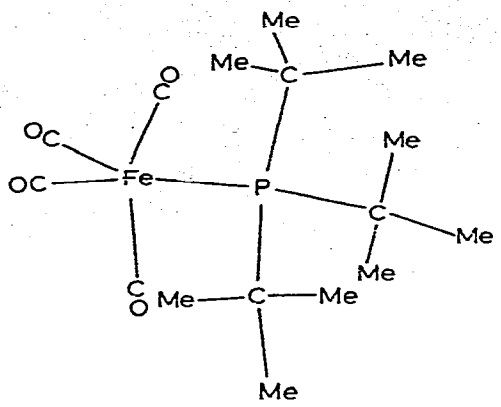
octahedra, Mn-C do not differ significantly from *fac*- and *mer*-$Mn(CO)_3(PR_3)_2Br$; Mn-CO 1.780(13) (*25*), 1.839Å(17) (*26*) [332]. (*27*) TBP with axial P; Fe-P 2.364(1), Fe-C(ax), 1.768(4), Fe-C(eq) 1.786(8)Å [177]. (*28*) Monomeric paramagnetic CoL_5 derivative; distorted SP [318]. (*29*) Nearly regular TBP, with Co-N 2.06(2)Å [See (*34*), and isoelectronic NO complex (*447*)] [362]. (*30*) SP, with apical $P(OMe)_3$ (CO expected to occupy this position); comparisons with (*464*) [355]. (*31*) Ligand tridentate, Rh approximately square planar [310]. (*32*) Ligand bidentate, fourth coordination position occupied by H_2O, which H-bonds to O in chelate chain; contains 14-membered ring [310]. (*33*) Only small distortions from tetrahedral geometry [cf. (*24*)]; Ni-P 2.291(5)Å is normal [160].



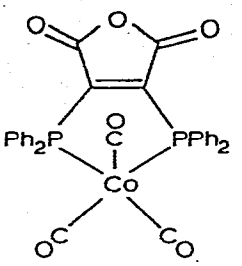
(25) *cis*



(26) *trans*



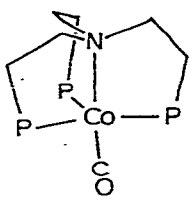
(27) $Fe(CO)_4(Bu_3^tP)$



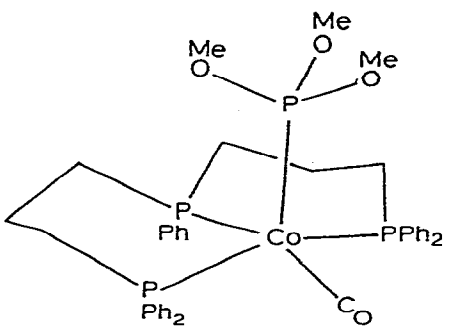
(28) $Co(CO)_3[C_4O_3(PPh_2)_2]$

$\left\{Mn(CO)_2[P(OMe)_2Ph]_4\right\}^+$

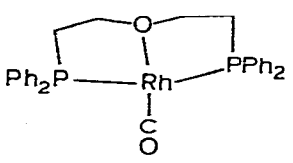
$P \equiv P(OMe)_2Ph$



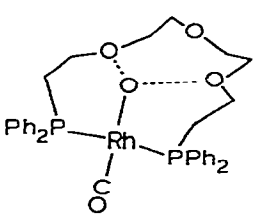
(29) $[Co(CO)(np_3)]^+$



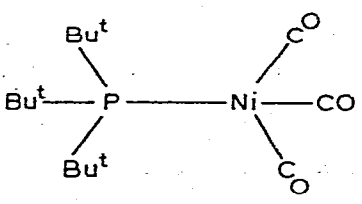
(30) $\left\{Co[PhP(CH_2CH_2CH_2PPh_2)_2][P(OMe)_3](CO)\right\}^+$



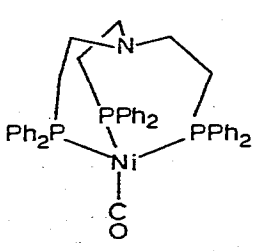
(31) $\left\{Rh(CO)[O(CH_2CH_2PPh_2)_2]\right\}^+$



(32) $\left\{Rh(CO)(OH_2)[O(CH_2CH_2OCH_2CH_2PPh_2)_2]\right\}^+$



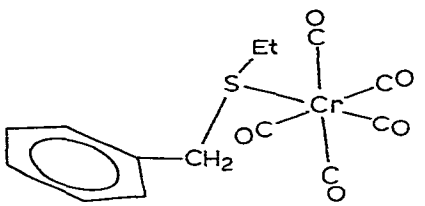
(33) $Ni(CO)_3(PBu_3^t)$



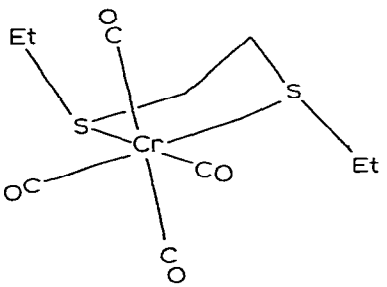
(34) $Ni(CO)(np_3)$

(*34*) Distorted tetrahedral, N does not coordinate, with Ni-N 3.25(1)Å [see also (*29*) and isoelectronic NO complex (*456*)] [362].

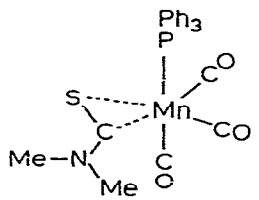
(e) *Carbonyls containing S-donor ligands*



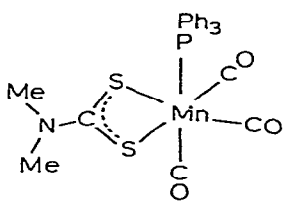
(35) $Cr(CO)_5[S(CH_2Ph)Et]$



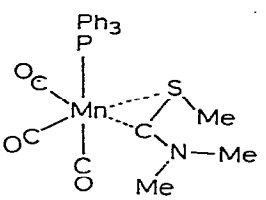
(36) $Cr(CO)_4[EtS(CH_2)_2SEt]$



(37) $Mn(CO)_3(PPh_3)(SCNMe_2)$



(38) $Mn(CO)_3(PPh_3)(S_2CNMe_2)$



(39) $[Mn(CO)_3(PPh_3)(MeSCNMe_2)]^+$

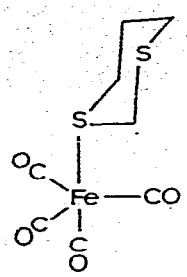
(*35*) From $Cr(CO)_5[CPh(OEt)]$ + $EtSCH_2Ph$; Cr-S 2.459(2)Å [133].

(*36*) Cr-CO (*cis* to S) 1.887(3), Cr-CO (*trans* to S) 1.831(3)Å [73].

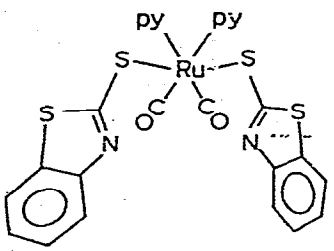
(*37*) Nearly planar thiocarboxamide group; see (*39*) for effects of S-methylation on π-interactions of C with other atoms [266].

(*38*) Reference [267]. (*39*) S-methylated (*37*); complex π interaction of C with 3 adjacent atoms; methylation reduces C-S, increases Mn-C π interactions; carbenoid $C(SMe)(NMe_2)$ ligand [266].

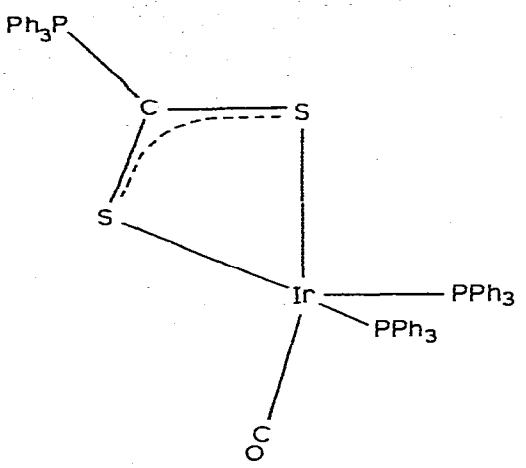
(*40*) From $Fe_2(CO)_9$ + 1,3-dithiacyclohexane; TBP with apical S; trends



(40) $Fe(CO)_4(C_4H_8S_2)$



(41) $Ru(mbt)_2(py)_2(CO)_2$



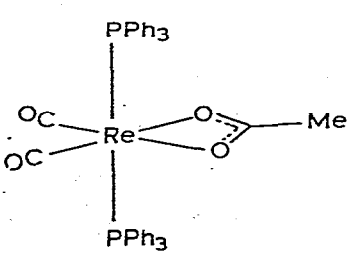
(42) $[Ir(S_2CPPh_3)(CO)(PPh_3)_2]^+$

in Fe-CO distances with N- and S-bonded ligands discussed [48]. (*41*) From $Ru_3(CO)_{12}$ + mercaptobenzothiazole, then py; ligand coordinated via exocyclic S only [288]. (*42*) Originally thought to be η-CS_2 complex, now show to contain Ph_3P^+-CS_2^- ligand; TBP has one axial Ir-S (2.379), one equatorial Ir-S (2.312Å); leads to possible reformulation of $[RuCl_2(CS_2)(PPh_3)_3]^+$ and related complexes [395].

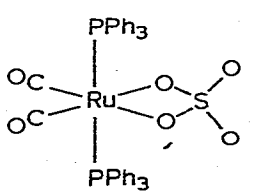
(f) *Carbonyls containing anionic ligands*

(*43*) Isomorphous with Mn compound previously determined [353].

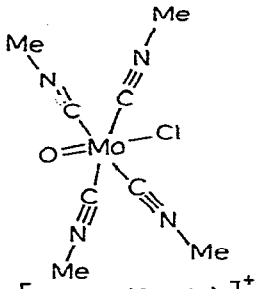
(*44*) Obtained during attempted isolation of $Ru(CO)_2(SO_2)(PPh_3)_2$ [344].



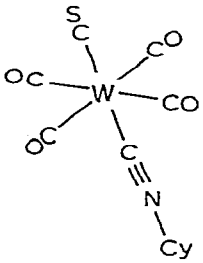
(43) $Re(OAc)(CO)_2(PPh_3)_2$



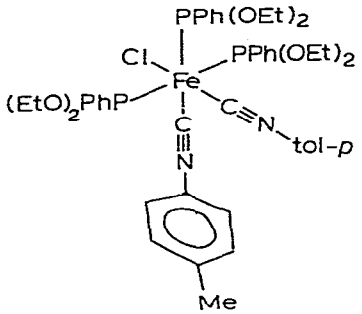
(44) $Ru(SO_4)(CO)_2(PPh_3)_2$



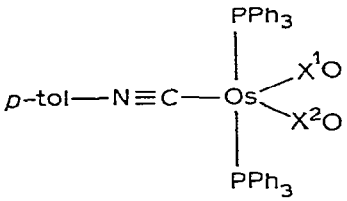
(45) $[MoOCl(CNMe)_4]^+$



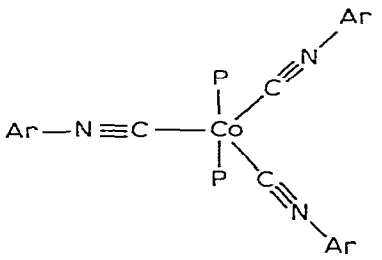
(46) $W(CO)_4(CS)(CNCy)$



(47) $\{FeCl(CNtol\text{-}p)_2[PPh(OEt)_2]_3\}^+$

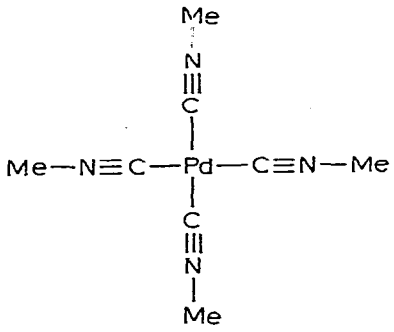


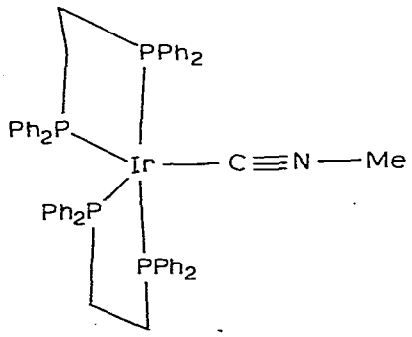
(48) $[Os(CO)(NO)(PPh_3)_2(CN\,tol\text{-}p)]^+$



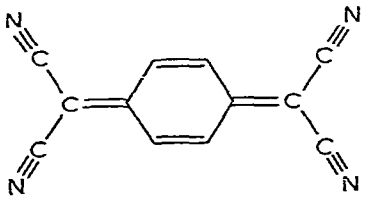
P ≡ $PPh(OEt)_2$ Ar = 4-$NO_2C_6H_4$

(49) $\{Co(CNC_6H_4NO_2)_3[PPh(OEt)_2]_2\}^+$

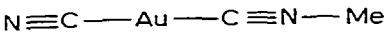




(50) $[Ir(CNMe)(dppe)_2]^+$



(51) $[Pd(CNMe)_4](tcnq)_4$



(51a) Au(CN)(CNMe)

(g) *Thiocarbonyls*

See *46*, *310*.

(h) *Isocyanide complexes*

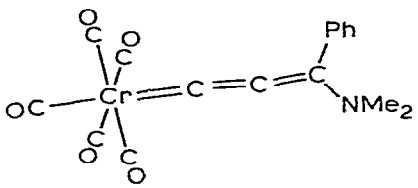
(*45*) No unusual structural features; Cl *trans* to O; paper compares other MoO complexes [51]. (*46*) W-C(CS) 1.944, W-C(CO) 2.0645, W-C(CNCy) 2.158Å; correlation of C-S bonds with ν(CS) [95]. (*47*) Long Fe-P, multiple Fe-C bonds show isocyanide to be a better π-acceptor than phosphonite; latter have *mer* configuration, in contrast to ^{1}H NMR data [371]. (*48*) TBP, apical PPh_3 groups: CO,NO not distinguished, but X^1 probably N, on basis of distances: Os-X^1 1.67(2), Os-X^2 1.84(3)Å [368]. (*49*) Slightly distorted TBP, double bond character in Co-C [1.80(av.)Å]; Co-C-N(Ar) bent *ca.* 8°; distortions around P correlate with Co-P π interaction; see also (*47*) [359]. (*50*) TBP, with linear axial MeNC; comparison with corresponding CO, NO systems; stereochemical nonrigidity discussed [394].
(*51*) Square planar Pd, no stacking interactions between cations and anions; Pd-C 1.984Å(av.) [57]. (*51a*) Weak Au...Au interactions, with each Au bonded to six other Au in regular hexagon to give central Au_7 sheet, Au...Au 3.52 - 3.72Å; C-Au-C 176(2)°, Au-C(CN) 2.01(5), Au-C(CNMe) 1.98(5)Å [23].

See also: *80*, *81*, *214*, *219*, *312*, *313*, *391*.

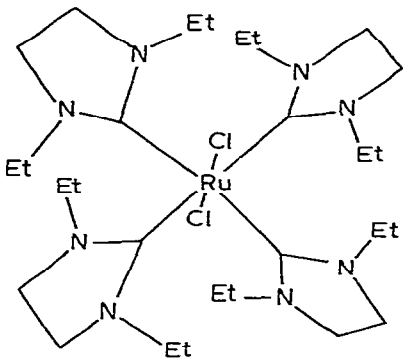
(i) *Carbene and carbyne complexes*

(*52*) Allenylidene complex from $Cr(CO)_5[C(OEt)CHCPh(NMe_2)]$ + BF_3; heterocumulene almost linear; distances Cr-C 2.015(15), C-C 1.236 and 1.372(21) show contribution from mesomeric structure

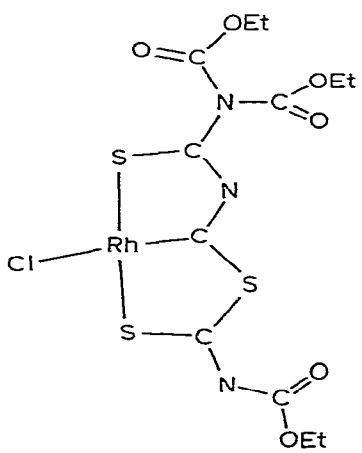
$(OC)_5\bar{C}r-C\equiv C-C^{Ph}_{+\;\;NMe_2}$ [163]. (*53*) From $LiCHS(CH_2)_3S$ + $Cr(CO)_6$, followed by alkylation, via CO insertion into carbene complex [97]. (*54*) Neutral tetra-carbene complex, ligands essentially planar in propellor-like arrangement, with planes inclined *ca.* 43° to equatorial plane [309]. (*55*) Tridentate fused-ring ligand from



(52) $Cr(CO)_5[:C:C:CPh(NMe_2)]$

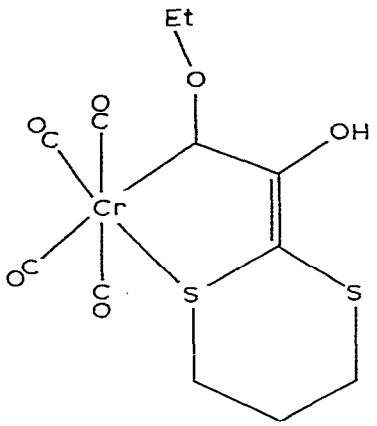


(54) $RuCl_2[\overline{CNEt(CH_2)_2NEt}]_4$

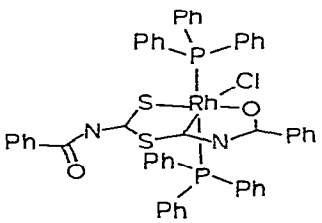


(55) $RhCl(PPh_3)_2(EtOCONCS)_3$

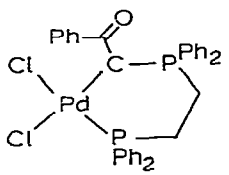
PPh_3 ligands above and below Rh omitted



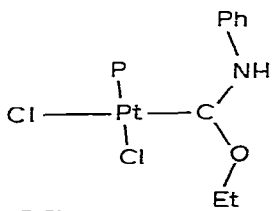
(53) $Cr\{C(OEt)[C(OH)\overline{CS(CH_2)_3S}]\}(CO)_4$



(56) $RhCl(PhCONCS)_2(PPh_3)_2$



(57) $PdCl_2[PhCOCHPPh_2(CH_2)_2PPh_2]$



P ≡ PEt_3

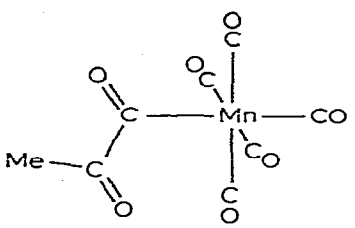
(58) *cis*-$PtCl_2[C(NHPh)(OEt)](PEt_3)$

3 EtOCONCS on Rh, essentially planar; Rh-C(carbene) 1.93Å, with large *trans* influence on Rh-Cl (2.46A); see also (*56*) [376]. (*56*) 2 PhCONCS condense on Rh via 3-fragment oxidative-addition to give nearly planar tridentate bicyclic ligand bonded via S, C and O; bonding C of ligand is tertiary carbene, *trans* influence gives long Rh-Cl 2.455(2)Å; Rh-S is short, indicating multiple bonding [391,392]. (*57*) Square planar Pd, ylid ligand confirmed [330]. (*58*) From $PtCl_2(CNPh)(PEt_3)$ + EtOH by 1,2-addition; short Pt-C 1.962(18)Å; carbene has lower *trans* influence than σ-vinyl on Pt-Cl [159].

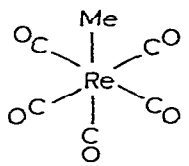
See also: *135, 140, 227a, 323, 327.*

(j) *Alkyls, aryls and acyls*

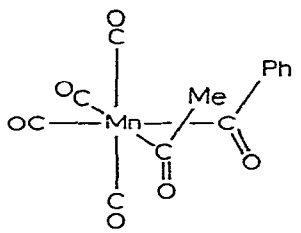
(*59*) Decarbonylates 21-times more slowly than acetyl complex; *s-trans*-CO groups in organic ligand [43]. (*60*) Electron diffraction study: Re-C 2.308(17)Å [17]. (*61*) Angles: *cis*-OC-Mn-CO 96.5°, *cis*-acyl-Mn-acyl 81.2° [118]. (*62*) Enol tautomer of metallo



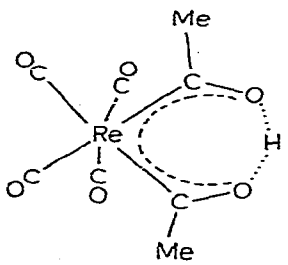
(59) $Mn(COCOMe)(CO)_5$



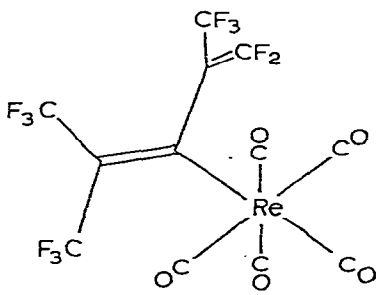
(60) $ReMe(CO)_5$



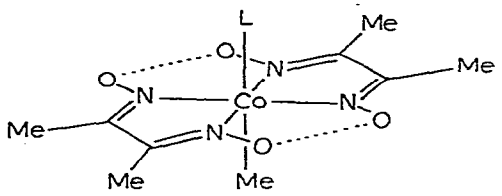
(61) $[cis\text{-}Mn(COMe)(COPh)(CO)_4]^-$



(62) $\overline{Re(CMeO\cdots H\cdots OCMe)(CO)_4}$



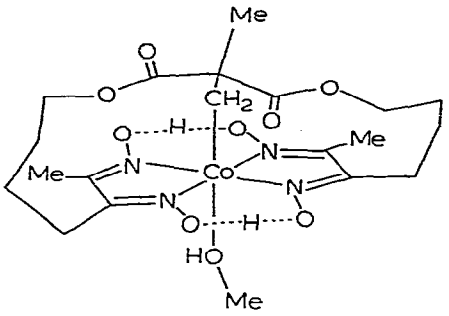
(63) $Re\{C[:C(CF_3)_2]C(CF_3):CF_2\}(CO)_5$



(64) L = Meim

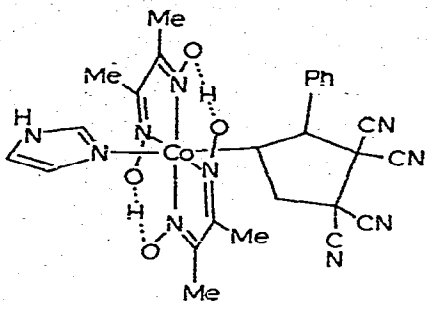
(65) L = py

$COMe(L)(dmg)_2$

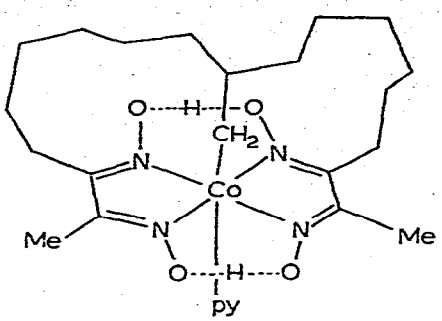


(66) Cobaloxime

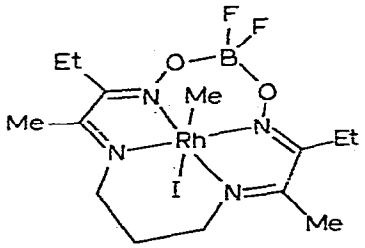
acetylacetone; Re-C 2.16Å(av.) [46]. (*63*) From $NaRe(CO)_5$ + $(CF_3)_2C{=}C{=}C(CF_3)_2$ [112]. (*64*)(*65*) Co-C 2.009(7), 1.998(5)Å, respectively; discussion of *cis* influence of ligands on Co-C bond length in 12 related complexes [123]. (*66*) Bridged cobaloxime, with chiral ester groups pointing in opposite directions [cf. (*68*)] [236]. (*67*) From addition tcne to allylcobaloxime; mechanistic consequences of observed Ph *trans* to Co discussed [292]. (*68*) Chiral, with comparatively rigid bridge; structure contrasted with bridged but not intramolecularly alkylated cobaloxime; see also (*66*) [325]. (*69*) Confirms structure; Rh-Me 2.090(4)Å; B atom above macrocyclic plane; H bond from Me to F not confirmed [147]. (*70*) Unusual Rh-Me 2.031(6)Å in SP complex, with 4N forming basal plane; explained using d_{z2} for Rh-C, dsp^2 hybrids for Rh-N bonds [342]. (*71*) No π interaction between Ir and Ph; Ir-Br 2.625(3)Å shows *trans* influence of H, not located but probably in vacant coordination site [217]. (*72*) First acyl-Ni complex, Ni-C 1.84, long C-Me 1.57Å [56]. (*73*) From CO + Ni-Me complex; 5-coordinate Ni in distorted TBP [366]. (*74*) From $NiCl_2$ + nas_3 + $NaBPh_4$ in BuOH; transfer of Ph from B to Ni; no unusual structural features, except angle at ring C bonded to Ni [113.5(15)°], attributed to some multiple Ni-C bond character [377]. (*75*) Insertion of $CH(CO_2Et)$ into Ni-N distorts porphin core so that pyrrole containing N-C inclined toward Ni; Ni-C 1.905(4)Å [374,375]. (*76*) C-bonded $AcCH_2CO_2Et$, from keto-ester and Na_2PdCl_4, then



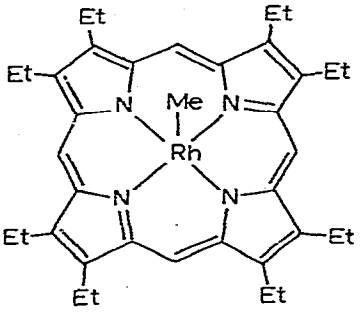
(67) CoR(imH)(dmg)$_2$
R = 3,3,4,4-(CN)$_4$-2-Ph-cyclopentyl



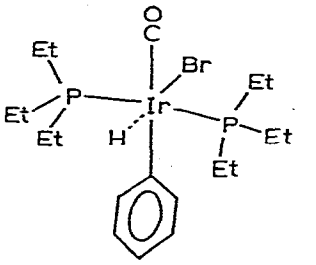
(68) Cobaloxime



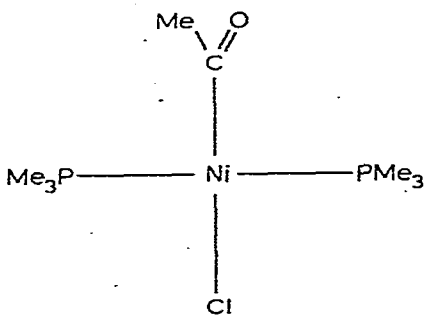
(69) RhMeI[C$_2$(do)(doBF$_2$)]



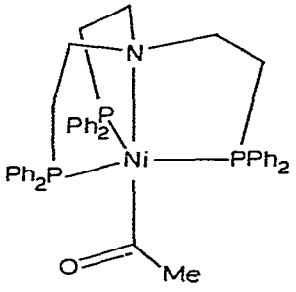
(70) RhMe(oep)



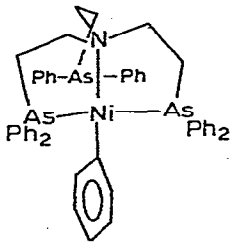
(71) IrHBrPh(CO)(PEt$_3$)$_2$



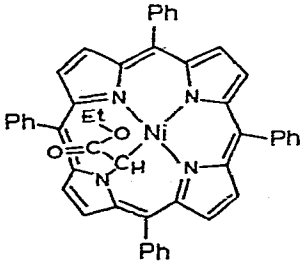
(72) *trans*-NiCl(COMe)(PMe$_3$)$_2$



(73) [Ni(COMe)(np$_3$)]$^+$

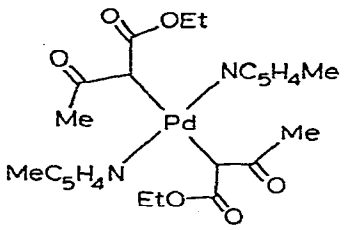


(74) [NiPh(nas$_3$)]$^+$

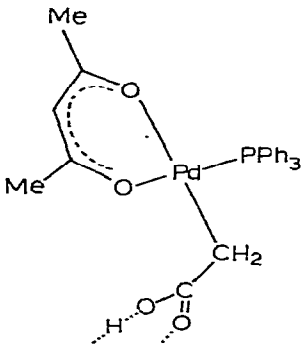


(75) Ni[tppHC(CO$_2$Et)]

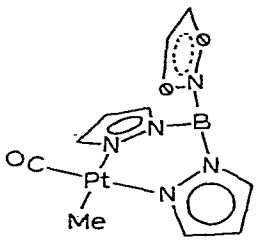
2-Mepy [273]. (*77*) Substituted acetic acid, dimeric via H-bonds to second molecule; from Pd[CH$_2$C(O)O](PPh$_3$)$_2$ + acacH [283]. (*78*) Unusually



(76) $Pd(CHAcCO_2Et)_2(2\text{-}Mepy)_2$

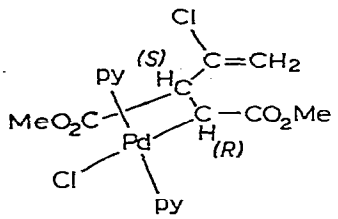


(77) $Pd(CH_2CO_2H)(acac)(PPh_3)$

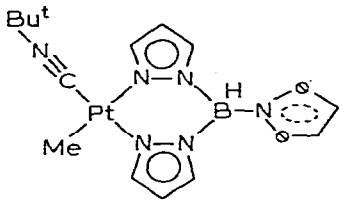


(79) $PtMe(CO)[HB(pz)_3]$

⊙ disordered C, N

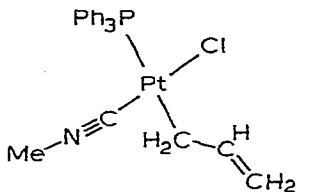


(78) $PdCl[CH(CO_2Me)CH(CO_2Me)CCl{:}CH_2](py)_2$

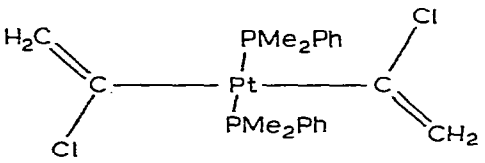


(80) $PtMe(CNBu^t)[HB(pz)_3]$

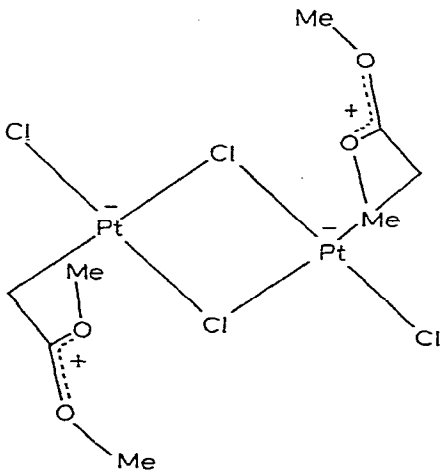
⊙ disorderded C, N



(81) $PtCl(CH_2CH{:}CH_2)(CNMe)(PPh_3)$



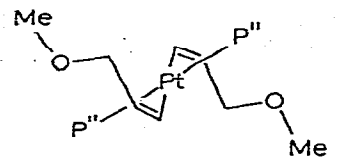
(83) *trans*-$Pt(CCl{=}CH_2)_2(PMe_2Ph)_2$



(82) $\{PtCl_2[CH_2C(OMe)_2]\}_2$

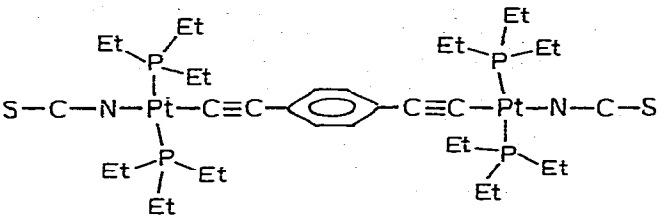
stable, from corresponding η^3-butenyl halide + py; 2 chiral centres have opposite configuration, with 2 H being mutually *trans* [199].

(*79*) Flattened PtN_4B boat; C,N of uncoordinated pz disordered; fluxional in solution [81]. (*80*) Similar to CO complex, with bidentate $HB(pz)_3$

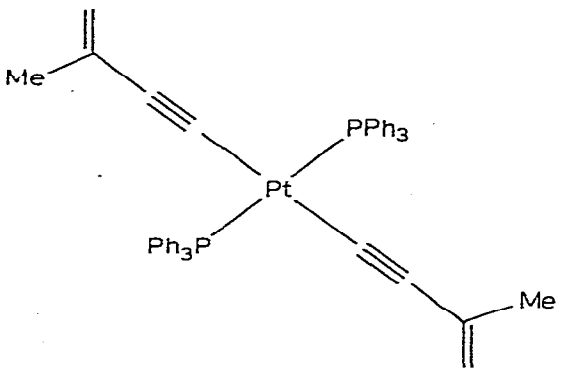


P"≡PMe_2Ph

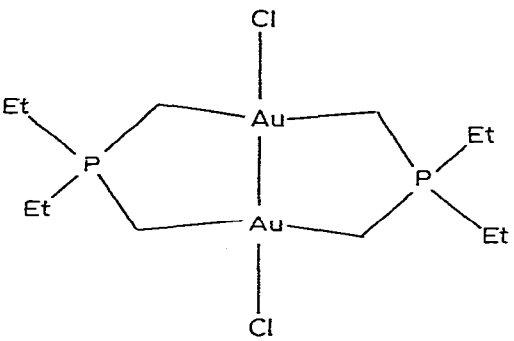
(84) $Pt(CH{:}CHCH_2OMe)_2(PMe_2Ph)_2$



(86) $[Pt(NCS)(PEt_3)_2]_2$-*p*-$C_6H_4(C{\equiv}C)_2$



(85) $Pt(C_2CMe{:}CH_2)_2(PPh_3)_2$

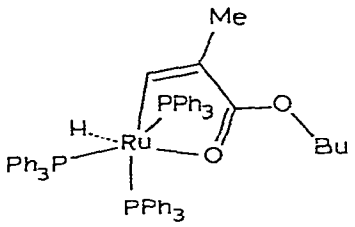


(87) $[AuCl(CH_2)_2PEt_2]_2$

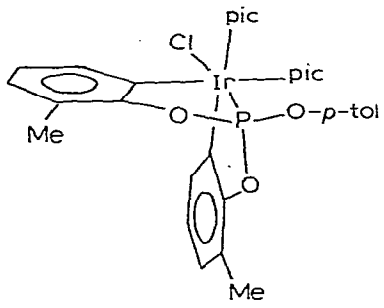
ligand, non-coordinated pz on equatorial B position; PtN_4B ring in boat form; Pt-Me 2.12, Pt-CN 1.857, two Pt-N 2.082 (*trans* to Me), 2.006Å (*trans* to Bu^tNC) [158]. (*81*) σ-Allyl has large thermal motion; long Pt-P *trans* to allyl, 2.359(4)Å; allyl parameters: Pt-C 2.14(2), C-C 1.32(5), C=C 1.24(6)Å [261]. (*82*) From $[PtCl_2(C_2H_4)]_2$ + $CH_2{=}C(OMe)_2$, complex formulated as σ-bonded zwitterion [54]. (*83*) C=C 1.316(7), C-Cl 1.809(6) [cf. $CH_2{=}CHCl$, 1.728(7)Å]; explains lability of (*83*), which on heating gives C_2H_2 + $PtCl(CCl{=}CH_2)(PPh_3)_2$ [229]. (*84*) Hydrazine reduction of $Pt(C_2CH_2OMe)_2(PMe_2Ph)_2$ by *cis* addition gives *Z* stereochemistry shown [274]. (*85*) From *cis*-$PtCl_2(PPh_3)_2$ + 2-methylbut-3-yn-2-ol in presence of aqueous NH_3; Pt-C 2.024(6)Å [370]. (*86*) Square planar Pt, with Pt-C 1.921(12), 0.09Å shorter than Pt-C single bond [338]. (*87*) From gold(I) complex by addition of Cl_2; contains short Au-Au bond [2.597(5)Å, compared to distances of 2.68-2.98Å in cluster compounds, 2.884Å in metallic gold [110].

See also: *192*, *193*, *194*, *195*, *196*, *197*, *204*, *205*, *206*, *207*, *222*, *232*, *233*, *235*, *237*, *238*, *239*, *264*, *302*, *303*, *304*, *309*.

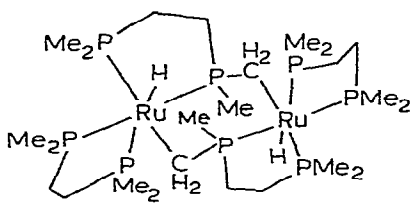
(k) *Complexes containing chelating* η^1*-ligands*



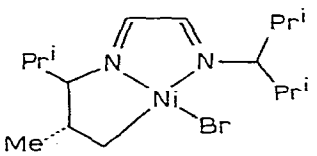
(88) $RuH[CH{:}CMeC(O)OBu](PPh_3)_3$



(90) $IrCl[(C_6H_3MeO)_2P(OC_6H_4Me)](\gamma\text{-pic})_2$

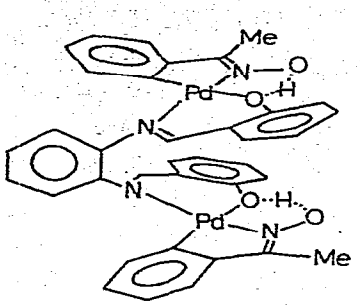


(89) $[RuH[CH_2PMe(CH_2)_2PMe_2](dmpe)]_2$

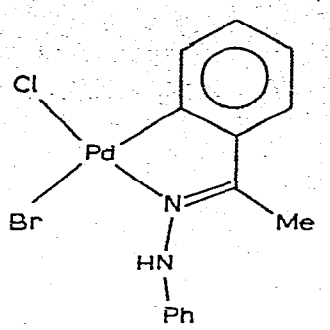


(91) $NiBr(C_{16}H_{31}N_2)$

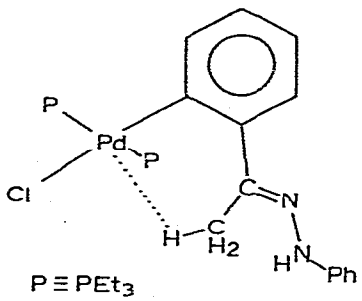
(*88*) From $RuH_2(PPh_3)_4$ + butyl methacrylate via vinylic C-H bond cleavage; Ru has distorted octahedral geometry, with *mer*-RuP_3; Ru-C(sp^2) 2.061(10)Å; Ru-H not refined [397]. (*89*) Supposed $Ru(dmpe)_2$; Ru-C 2.203(6), Ru-H 1.47(7)Å; 6-membered ring with '*para*' Ru atoms [276]. (*90*) Ready metallation in reaction $Ir_2Cl_2[P(O\text{-}o\text{-}tol)_3]_3$ + γ-picoline; short Ir-P, Ir-C, long Ir-Cl, may reflect relative *trans* influences of γ-picoline and $P(OR)_3$ [327]. (*91*) Contains unusual metallated ligand, formed from non-metallated $NiBr_2$ complex + tolyl Grignard; metallation occurs at an i-propyl methyl group [179]. (*92*) Salophen bridge twists sal out of C_6H_4 plane, lengthens Pd-O bonds; no metal-metal bond, H-bonding with OH groups [337]. (*93*) Pd-C 1.981(6)Å; 80% Br – 20% Cl in position *trans* to Pd-C; Pd also interacts weakly with H from NBu_4^+, in axial



(92) $[Pd(apo)]_2$(salophen)

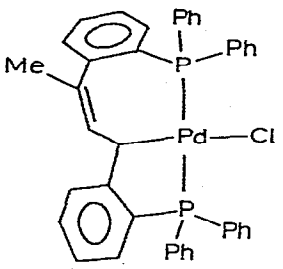


(93) $[PdClBr(C_6H_4CMe{:}NNHPh)]^-$

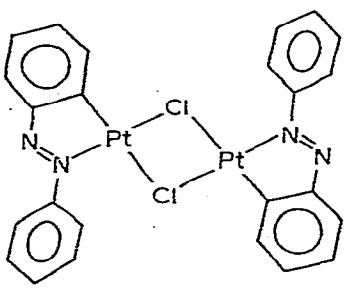


P ≡ PEt_3

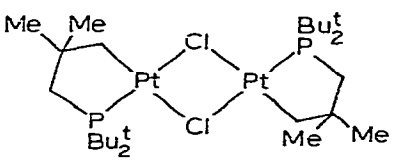
(94) $PdCl(C_6H_4CMe{:}NNHPh)(PEt_3)_2$



(95) $PdCl(Ph_2PC_6H_4CHCH{:}CMeC_6H_4PPh_2)$



(96) $[PtCl(C_6H_4N{:}NPh)]_2$



(97) $[Pt(CH_2CMe_2CH_2PBu^t_2)Cl]_2$

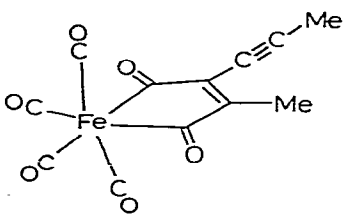
position of SP, with Pd-H 2.86Å [134]. (*94*) Non-chelated aryl by addition of PEt_3 to chelate complex; Pd-C 2.008(5)Å; Pd coordination non-planar, and ligand distorted, by Pd...Me interaction [2.92(8)Å] [134]. (*95*) Chelate η^1-allyl; severe steric strain causes Pd, Cl, P, P to deviate significantly from planarity; contains fused 5:7 chelate rings [352]. (*96*) Prototypical cyclometallated complex, with square planar Pt, planar chelate ring, free Ph twisted 39° out of plane;

Pt-C 1.94(2), PtCl 2.33 (*trans* N), 2.46Å (*trans* C) [263]. (*97*) From $PtCl_2(PhCN)_2$ + $PBu^t_2(CH_2CMe_3)$ [298].

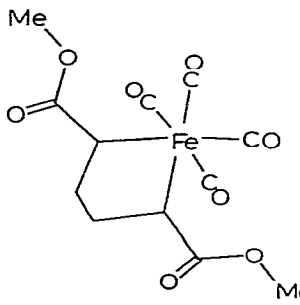
See also: *215*, *240*, *321*.

η^2-LIGANDS

(a) *($2\eta^1$)-Ligands (Metallocycles)*

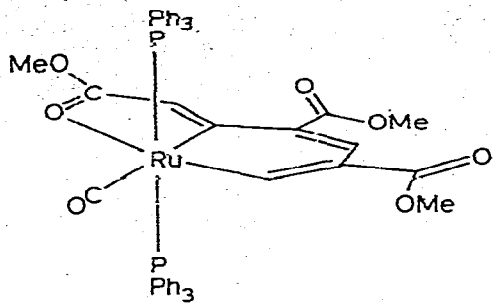


(98) $Fe(CO)_4(C_8H_6O_2)$

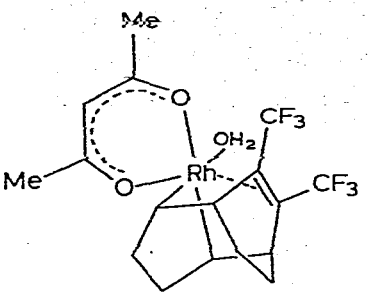


(99) $Fe(CO)_4[C_4H_6(CO_2Me)_2]$

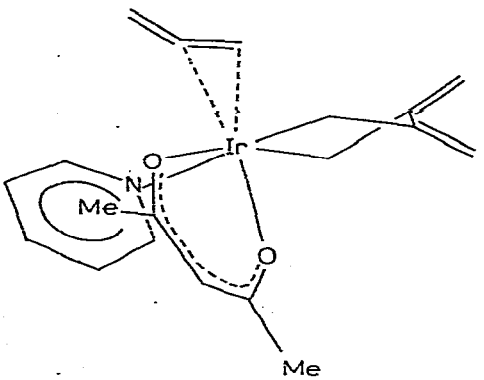
(*98*) Metallocycle derived from $Fe(CO)_5$ + MeC_2C_2Me; axial CO slightly tilted toward ferrole ring [88]. (*99*) From $Fe(CO)_4(\eta\text{-}CH_2{=}CHCO_2Me)$ + $CH_2{=}CHCO_2Me$/UV; long Fe-C from electron-withdrawing CO_2Me groups, which are mutually *trans*; apical CO groups bend towards organic fragment [98]. (*100*) From $RuH_2(CO)(PPh_3)_3$ + HC_2CO_2Me, with trimerisation of alkyne to give methylenemetallocyclohexadiene and coordinated ester group [383]. (*101*) From $[RhCl(cod)]_2$ + $C_2(CF_3)_2$, then acac; Rh(cod)(acac) + $C_2(CF_3)_2$ gives acac-$C_2(CF_3)_2$ adduct [188]. (*102*) From $Ir(C_8H_{14})_2(acac)$ + allene, then pyridine; for allene, C=C 1.32 (free), 1.42 (coordinated); C_6H_8Ir = 3,4-dimethyleneiridocyclopentane [214]. (*103*)(*104*) From $Pt(cod)_2$ + butadiene, probably via octadienediyl complex similar to (*154*); vinyl groups are on opposite sides of metallocyclic ring [82]. (*105*) Some disorder of 2 central C in ring, puckering relieves crowding of H [232]. (*106*) Platinalactone from CO + hydroxyacetylene complex; chelate ring has envelope conformation, with nearly planar Pt-CO-O-C; tetrahedrally distorted Pt [360]. (*107*) From $Pt(C_2H_4)(PPh_3)_2$ +



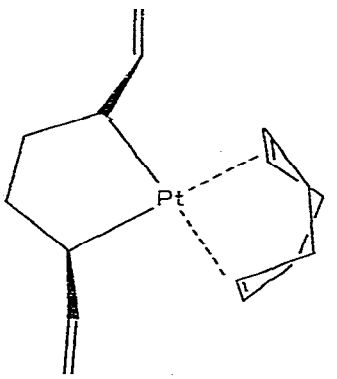
(100) $Ru(CO)(PPh_3)_2(HC_2CO_2Me)_3$



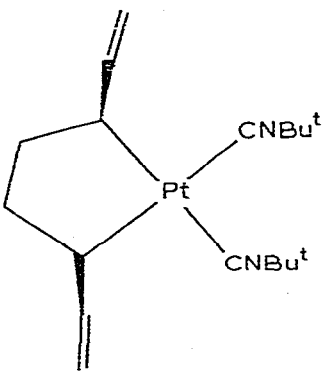
(101) $Rh(OH_2)(acac)(C_8H_{12}C_4F_6)$



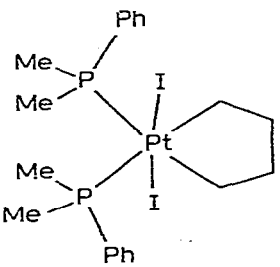
(102) $Ir(C_3H_4)(C_6H_8)(acac)(py)$



(103) $Pt[CH(CH{:}CH_2)CH_2CH_2CH(CH{:}CH_2)](C_8H_{12})$



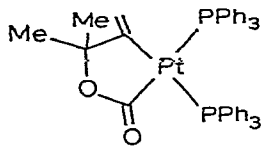
(104) $Pt[CH(CH{:}CH_2)CH_2CH_2CH(CH{:}CH_2)](CNBu^t)_2$



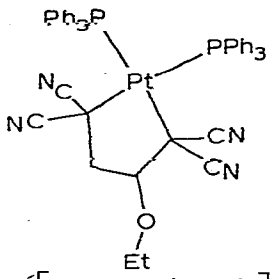
(105) $Pt(C_4H_8)I_2(PMe_2Ph)_2$

1,1,2,2-tetracyano-3-ethoxycyclobutane, Pt inserts into $(NC)_2C-C(CN)_2$ bond [369].

See also: *162, 200, 201, 202, 234, 241, 242, 243, 325, 326, 333, 337, 339, 343.*

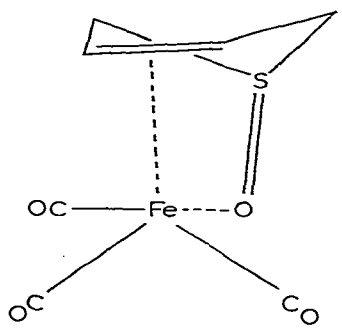


(106) $Pt[C(O)OCMe_2C{:}CH_2](PPh_3)_2$

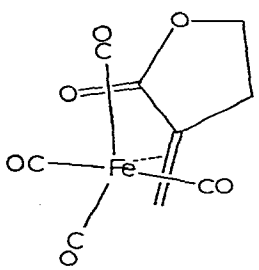


(107) $Pt[C_4H_3(OEt)(CN)_4](PPh_3)_2$

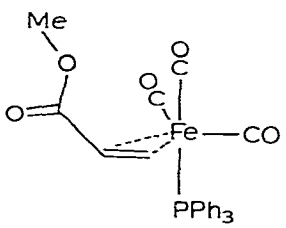
(b) *Olefin complexes*



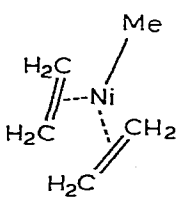
(108) $Fe(CO)_3(C_8H_6SO)$



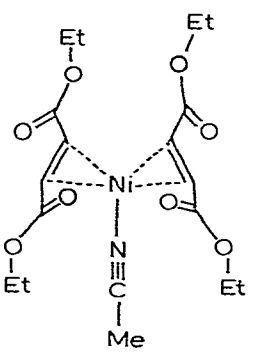
(109) $Fe(CO)_4(C_7H_8O_2)$



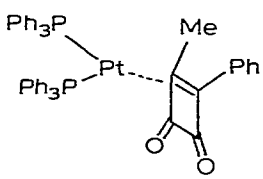
(110) $Fe(CO)_3(PPh_3)(CH_2{:}CHCO_2Me)$



(111) $[NiMe(C_2H_4)_2]^-$



(112) *rac*-$Ni[C_2H_2(CO_2Et)_2]_2(NCMe)$



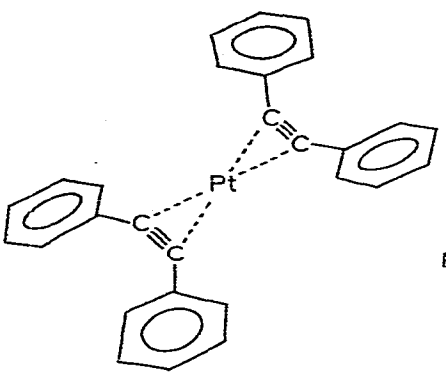
(113) $Pt(C_4MePhO_2)(PPh_3)_2$

(*108*) Chelated thiophene-S-oxide via C=C and S-O [39]. (*109*) Distorted TBP, equatorial olefin; Fe bonded on *opposite* side of organic ligand to vinyl group [see also (*155*)] [66]. (*110*) C=C tilted by 11.2° from

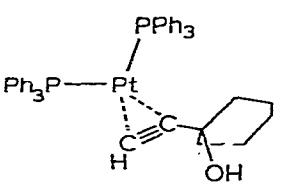
$Fe(CO)_2$ plane, with CO_2Me bent away from PPh_3 [279]. (*111*) Trigonal planar Ni, with weak Ni–olefin bond; Ni–Me 1.91, C=C 1.37(av.)Å; $Li(tmed)_2$ cation contain tetrahedral Li [33]. (*112*) Slightly distorted planar trigonal Ni; 4 asymmetric C have same absolute configuration, unit cell contains *SSSS* and *RRRR* molecules [203]. (*113*) Coordinated methylphenylcyclobutenedione, steric crowding gives distorted non-planar geometry with Pt–C 2.00(2) (Ph), 2.12(2) (Me), Pt–P 2.271, 2.309(4)Å [372].

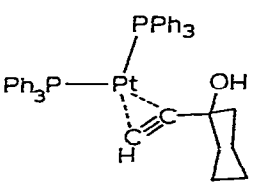
See also: *213*, *225*, *262*, *335*.

(c) *Alkyne complexes*



(114) $Pt(C_2Ph_2)_2$





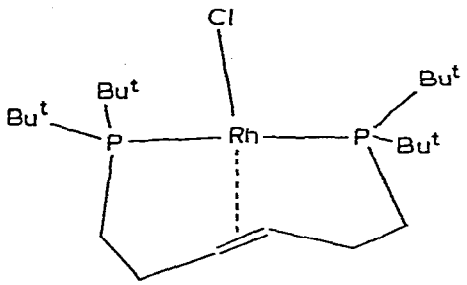
(115) $Pt(HC_2C_6H_{10}OH)(PPh_3)_2$

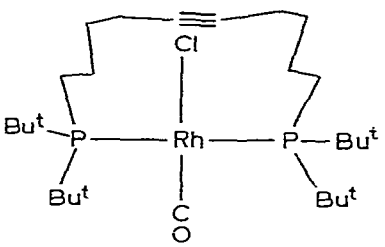
(*114*) Tetrahedral Pt, with dihedral between 2 PtCC planes 82°; Ph rings bent back 153°, C≡C 1.280(6)Å; reacts with PMe_3 to give (*351*) [306]. (*115*) Alkyne *cis*-bent, symmetrically bonded to Pt; 2 molecules in asymmetric unit with equatorial or axial C_2H groups, CCCy 139.4(12), 146.8(14)°, respectively; C≡C 1.312Å(av.) [365].

See also: *208*, *209*, *217*.

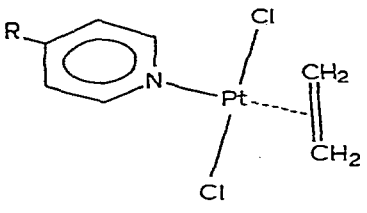
(d) *Olefin and alkyne halides*

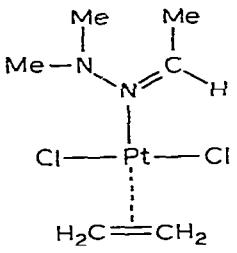
(*116*) C=C coordinated, transoid geometry about olefin, *trans* bridging P [257]. (*117*) C≡C not coordinated, with *trans* bridging P; both

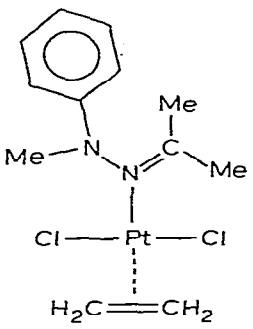




(116) $RhCl[Bu^t_2P(CH_2)_2CH{=}CH(CH_2)_2PBu^t_2]$ (117) *trans*-$RhCl(CO)[Bu^t_2P(CH_2)_4C{\equiv}C(CH_2)_4PBu^t_2]$







$PtCl_2(C_2H_4)(NC_5H_4R)$

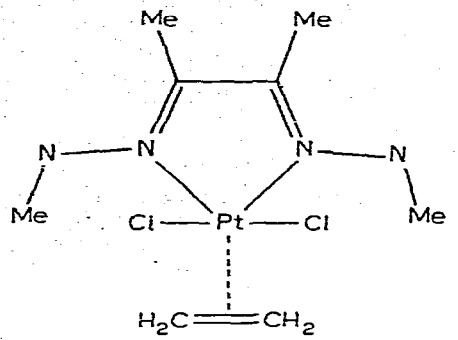
(118) R = CN

(119) R = Me

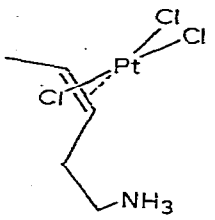
(120) $PtCl_2(C_2H_4)(MeCH{:}NNMe_2)$

(122) $PtCl_2(C_2H_4)(Me_2C{:}NNMePh)$

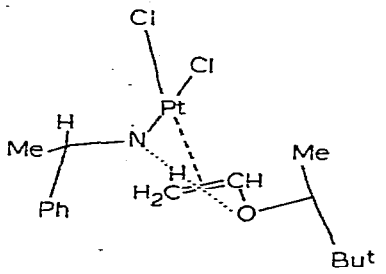
(*116*) and (*117*) are further examples of large ring complexes [257]. (*118*)(*119*) Complexes show extremes of ν(PtN) in series of 11 related complexes; corresponding bond lengths similar in both complexes; C=C 1.323, 1.354; Pt-N 2.051, 2.062Å, respectively [47]. (*120*)(*122*) Bonding of hydrazones determined; no comment in paper concerning differing C=C bonds of 1.71(9), 1.33(5), respectively [36]. (*121*) 5-coordinate olefin complex, axial Cl in TBP geometry; some N⋯N⋯C delocalisation in hydrazone ligand; olefin C=C 1.46(2)Å [55]. (*123*) Zwitterionic; this molecule has C(3)*S*:C(4)*R* configuration, crystal is racemic with C(3)*R*:C(4)*S* form; probably contains N——H···Cl bonded network [34]. (*124*)(*126*)(*127*) Correlation of J(PtC) with electron donating power of aryl substituent, and with C=C dimensions: NO_2 1.374(18), NMe_2 1.419(25), H 1.454(17)Å; Pt-C=C bond asymmetric with $Pt-CH_2$ < Pt-CHAr [119]. (*125*) Absolute configuration; arene rings overlap; C=C 1.360(11), unsymmetrically bonded to Pt, with Pt-C



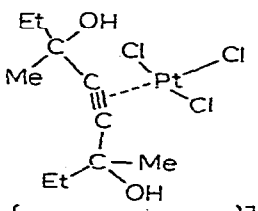
(121) $PtCl_2(C_2H_4)(MeHNN{:}CMeCMe{:}NNHMe)$



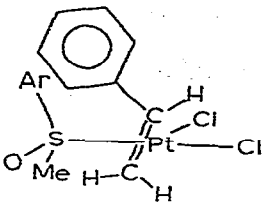
(123) $PtCl_3(C_5H_9NH_3)$



(128) *cis*-$PtCl_2[(R)\text{-}NH_2CHMePh]$-$[H_2C{:}CHOCHMeBu^t]$

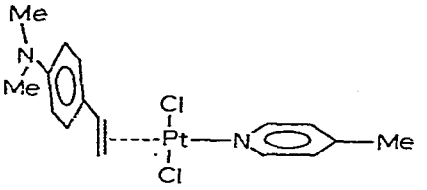


(129) $\left[PtCl_3\{C_2[CMeEt(OH)]_2\}\right]^-$

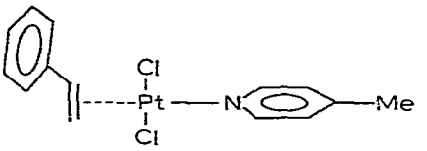


Ar = *p*-tol

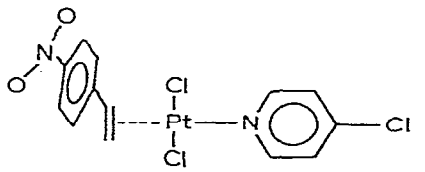
(125) $PtCl_2[Me(O)SC_6H_4Me][CH_2{:}CHPh]$



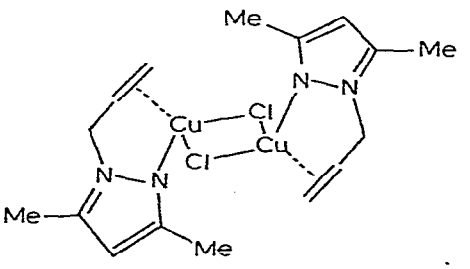
(126) $PtCl_2(CH_2{:}CHC_6H_4NMe)(NC_5H_4Me)$



(124) $PtCl_2(CH_2{:}CHPh)(NC_5H_4Me)$



(127) $PtCl_2(CH_2{:}CHC_6H_4NO_2)(NC_5H_4Cl)$

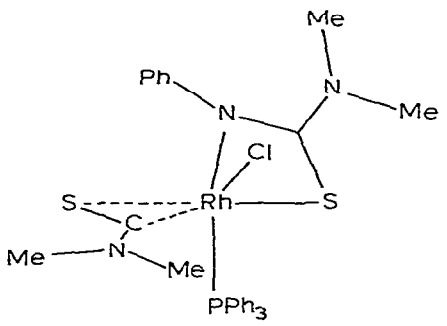


(130) $[CuCl(C_8H_{12}N_2)]_2$

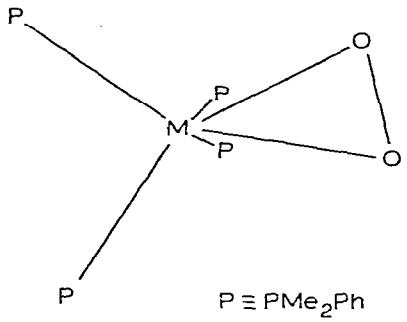
2.219, 2.188(9), the longer to CPh [168]. (*128*) Determines absolute configuration of whole molecule (*RRS*); amine NH bonds to ether O, proton located from difference map; C=C [1.33(4)Å], midpoint displaced

0.38Å from coordination plane [176]. (*129*) Alkyne C≡C 1.23Å, substituents bent back by 18, 23° [74]. (*130*) Dimeric via Cl bridges, quasi-tetrahedral Cu; C=C shows little increase (1.34Å), but Δν(C=C) is 103 cm^{-1} [50].

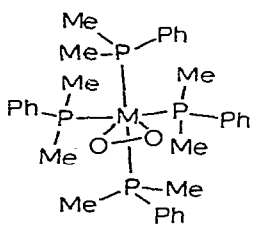
(e) *Complexes containing other three-membered rings*



(131) $RhCl[SC(NPh)NMe_2][SCNMe_2](PPh_3)$

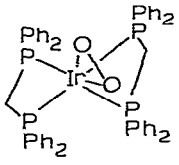


Inner coordination of metal in $[M(O_2)(PMe_2Ph)_4]^+$

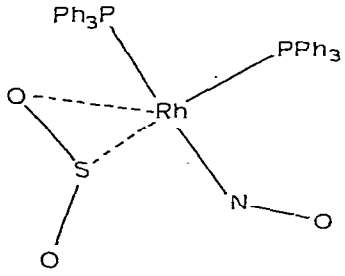


$[M(O_2)(PMe_2Ph)_4]^+$

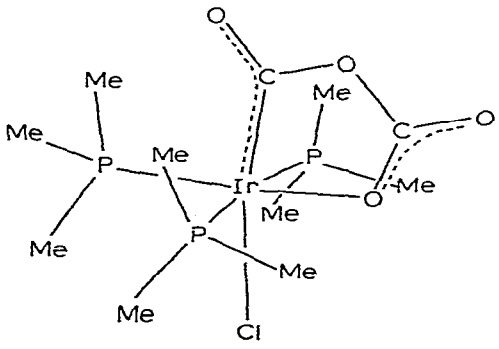
(132) M = Rh; (133) M = Ir



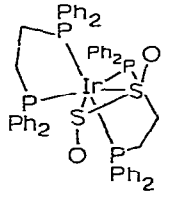
(134) $[Ir(O_2)(dppm)_2]^+$



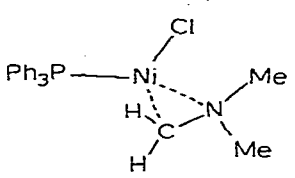
(136) $Rh(SO_2)(NO)(PPh_3)_2$



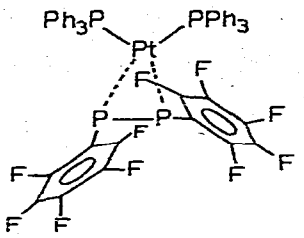
(135) $IrCl(C_2O_4)(PMe_3)_3$



(137) $[Ir(S_2O_2)(dppe)_2]^+$



(138) $NiCl(CH_2{:}NMe_2)(PPh_3)$



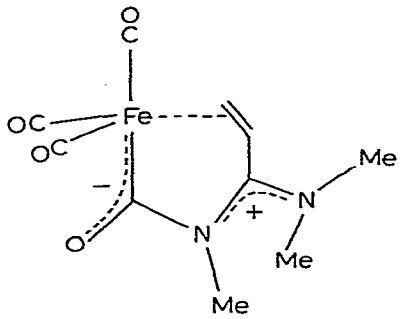
(139) $Pt[P_2(C_6F_5)_2](PPh_3)_2$

(*131*) Nearly planar thioureido group with C-S 1.66(2), Rh-C 1.955(17), Rh-S 2.419(5)Å [314]. (*132*) O-O 1.43, Rh-O 2.03Å [324]. (*133*) O-O 1.485(17), Ir-O 2.037, 2.050(10)Å [323]. (*134*) In PF_6^- and ClO_4^- salts, O-O identical within precision of analysis (1.45, 1.49Å, respectively). Chiral cation, TBP geometry with O_2, occupying one equatorial site. This and related work indicates O-O bond length is independent of metal and/or ligands, and does not correlate with reversibility of uptake of O_2 by these complexes; side-on O_2 is in peroxo form, with O-O between 1.45-1.50Å. Some differences in geometries result from different ligands and follow expected trends [386]. (*135*) From $Ir(C_8H_{14})(PMe_3)_3 + CO_2$; Ir-C bond has carbene character [83]. (*136*) Novel η^2-SO_2 coordination, with S-O 1.48, S=O 1.41, Rh-S 2.33, Rh-O 2.36Å [336]. (*137*) From η^2-S_2 complex + $NaIO_4$; S-S 2.041, S-O 1.43Å [383]. (*138*) From $Ni(PPh_3)_4$ and $[CH_2{=}NMe_2]Cl$; π-complexed iminium cation, more strongly bonded than olefin: C-N 1.392, Ni-C 1.884, Ni-N 1.920Å [243]. (*139*) From $Pt(PPh_3)_3 + P_4(C_6F_5)_4$, contains stabilised phosphorobenzene ligand; P-P 2.156(7), Pt-P 2.319, 2.364(5)Å; C_6F_5 groups on opposite sides of "square plane" and almost perpendicular to it, reducing interactions with PPh_3; two PtP_2 planes have dihedral of 20.4°, larger than found for similar olefin complexes [373].

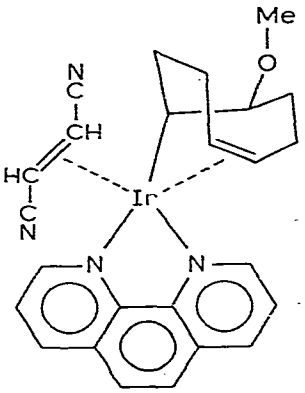
See also: *37*, *39*, *207*, *392*.

η^3-LIGANDS

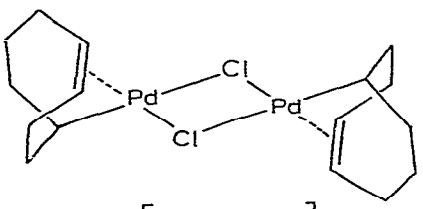
(a) *($\eta^1 + \eta^2$)-Ligands*



(140) $Fe(CO)_3[CONMeC(NMe_2)CH{:}CH_2]$



(141) $Ir(fn)(C_8H_{12}OMe)(phen)$



(142) $[PdCl(C_8H_{11})]_2$

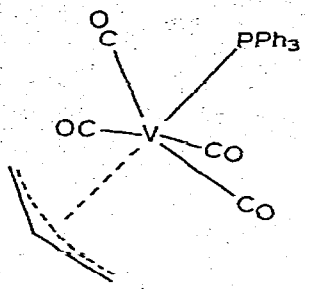
(*140*) Zwitterionic complex by alkylation of η^2-CH_2=CHCONMe$_2$ complex to give $CH_2=CH-C(OEt)=\overset{+}{N}HMe$; reaction with $HNMe_2$ gives olefin carbamylate [71].

(*141*) From $[Ir(cod)(phen)]^+$ + fn + MeOH; distorted TBP with axial σ-C, N; in fn, the CN group is bent back from Ir, C=C 1.49(4)Å [282].

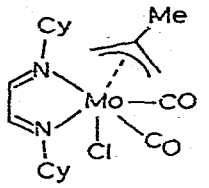
(*142*) From 1,5-cod, with skewed conformation in complex; Pd-Cl 2.527 (*trans* to σ-C), 2.366 (*trans* to C=C) [173].

(b) η^3-*Allyls*

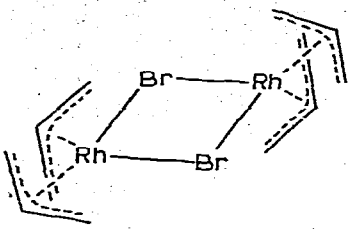
(*143*) V octahedral if allyl considered monodentate, with P *trans* to allyl; allyl plane inclined 13.6° to $V(CO)_4$ plane [278]. (*144*) N-bonded cyclohexylethylenediimine ligand, C:N 1.283(7), C-C 1.448(7)Å [233]. (*145*) Equal Rh-Br in central Rh_2Br_2 group contrasts with chloro complex studied earlier [107]. (*146*) Square-planar Ni, *exo*-pinenyl group; absolute configuration determined as (*S*)-PR_3, (+)-1*R*,5*R*-pinenyl [245]. (*147*) Ni disordered over 3 sites, giving 2 possible formal structures (a) and (b); stereochemistry of Ni-C_8 interaction unresolved [167].



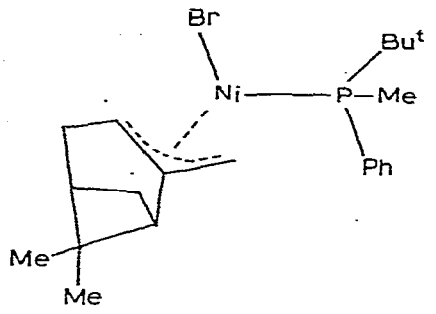
(143) $V(CO)_4(PPh_3)(C_3H_5)$



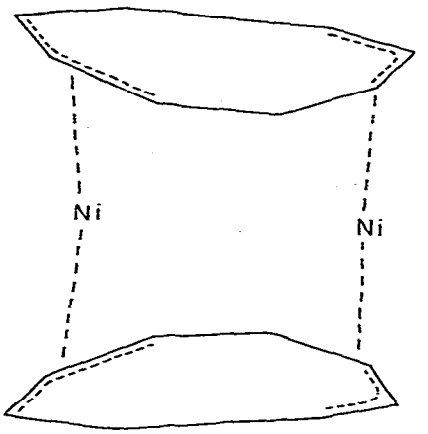
(144) $MoCl(CO)_2(CyN{:}CHCH{:}NCy)(C_4H_7)$



(145) $[RhBr(C_3H_5)_2]_2$

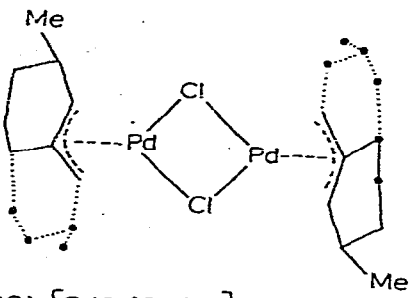


(146) $NiBr(C_{10}H_{14})(PMeBu^tPh)$



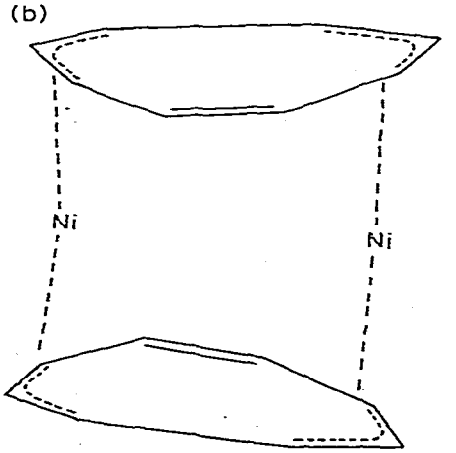
(a)

(147) $Ni_2(C_8H_8)_2$

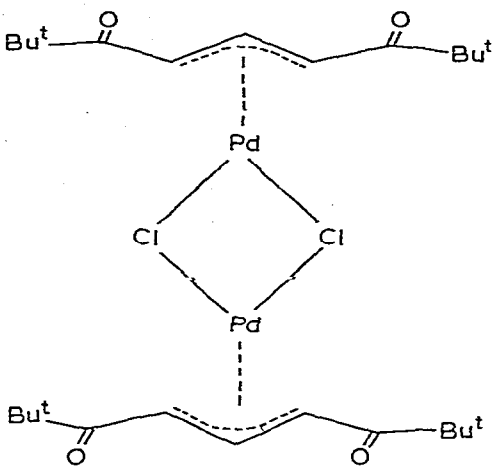


(148) $[PdCl(C_8H_{13})]_2$
skowing two orientations of ligand

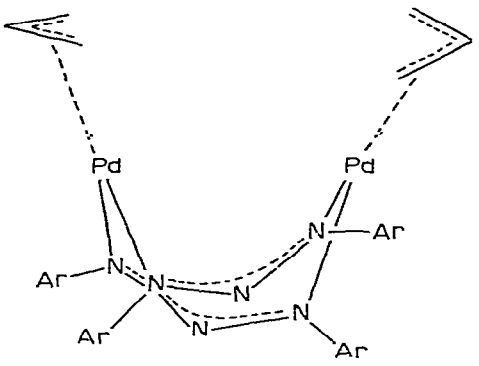
(b)



(*148*) From $PdCl_2(PhCN)_2$ + 1,3-dimethylenecyclohexane; two orientations of hydrocarbon found in crystal, shown superimposed on diagram [175]. (*149*) From $PdCl_2$ + 2,6-di-*t*-butylpyrylium cation,

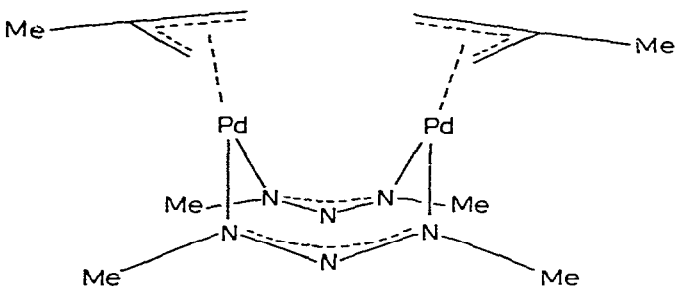


(149) $\left\{PdCl\left[C_3H_3(COBu^t)_2\right]\right\}_2$



Ar = p-MeC_6H_4

(151) $\left[Pd(C_3H_5)(N_3Ar_2)\right]_2$

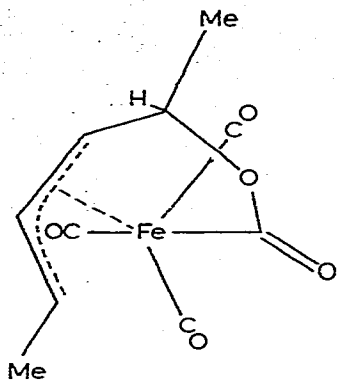


(150) $\left[Pd(C_4H_7)(N_3Me_2)\right]_2$

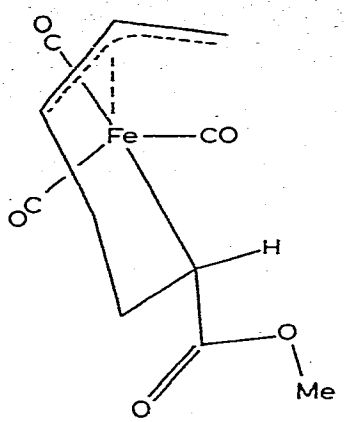
ring opened to give η-allyl-Pd complex [297]. (*150*) Triazenide ligand not completely delocalised, as shown by asymmetry in N-N, allyl C-C and Pd-C bonds; Pd....Pd separation 2.97Å [109]. (*151*) Approximate square planar Pd bridged by triazenido groups, with π conjugation over whole N_3 ligand; allyls stereochemically equivalent [331].

η^4-LIGANDS

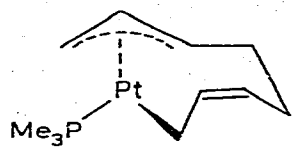
(a) *(η^1 + η^3)-Ligands*



(152) $Fe(CO)_3[C(O)OC_4H_4Me_2]$



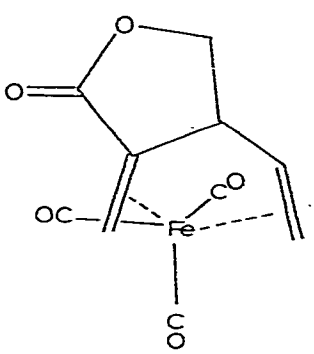
(153) $Fe(CO)_3[CH(CO_2Me)CH_2C_3H_4]$



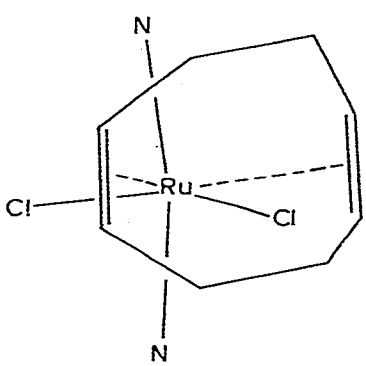
(154) $Pt(C_8H_{12})(PMe_3)$

(*152*) Ferrelactone from $Fe_2(CO)_9$ + *cis*, *trans*-hexa-2,4-diene monoepoxide; Fe-C (carboxylate) 1.9848(24)Å, shorter than Fe-C(sp^3) found earlier [69]. (*153*) 1,4,5,6-η^4-bonded CH_2:$CHCO_2Me$-butadiene adduct formed by irradiation of components + $Fe(CO)_5$ [100]. (*154*) From $Pt(C_2H_4)_2(PMe_3)$ + butadiene; intermediate in formation of metallocycles (*103*) and (*104*) [82].

(b) *($2\eta^2$)-Ligands*

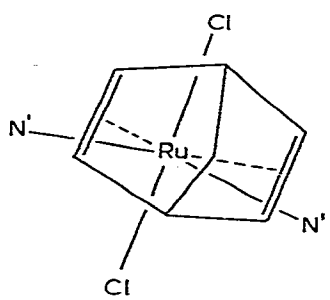


(155) $Fe(CO)_3(C_7H_8O_2)$



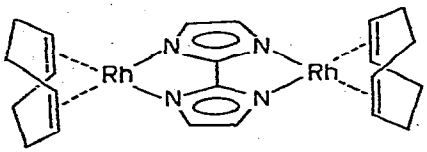
N = $NH_2C_6H_{13}$

(156) $RuCl_2(cod)(NH_2C_6H_{13})$

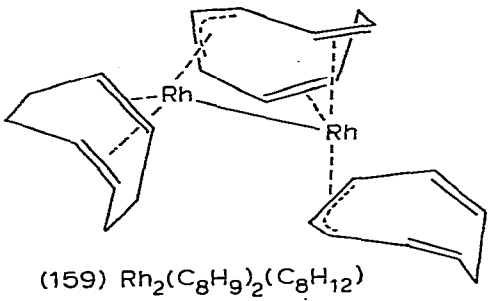


N' = piperidine

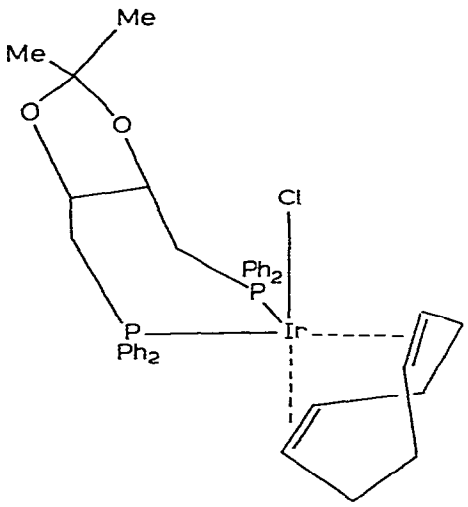
(157) $RuCl_2(nbd)(pip)$



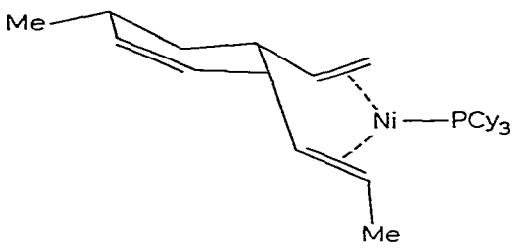
(158) $[Rh(C_8H_{12})]_2$(biim)



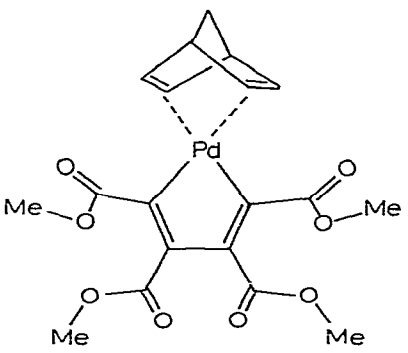
(159) $Rh_2(C_8H_9)_2(C_8H_{12})$



(160) $IrCl(C_8H_{12})$[(+)-diop]



(161) $Ni(C_{12}H_{18})(PCy_3)$

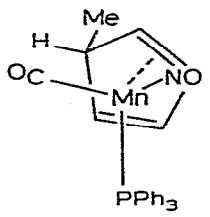


(162) $PdC_4(CO_2Me)_4$(nbd)

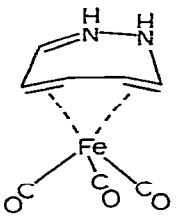
(*155*) First η^4-1,4-diene with alkenes not in a ring system; intermediate between SP and TBP coordination for Fe; this and (*109*) from $Fe_2(CO)_9$ + 2,3-bis(hydroxymethyl)methylenecyclopropane [66]. (*156*)(*157*) Two complexes have respectively *cis* or *trans* Cl_2; *trans* influence of olefin on Ru–Cl or Ru–N measured [190]. (*158*) Quadridentate planar bridge between 2 Rh [254]. (*159*) From $[RhCl(cod)]_2$ + $Na_2C_8H_8$; contains Rh–Rh bond, and C_8H_9 ligands are η^3, and $\eta^3 + \eta^4$ [271]. (*160*) Distorted TBP, with diop chelating apical and equatorial

positions; relation to asymmetric induction in reductions catalysed by Rh-diop systems [351]. (*161*) From Ni-PCy_3 complex + 2,4,6-octadiene + butadiene; divinylcyclohexene derivative, Ni not exactly trigonal [316]. (*162*) Olefinic C=C in nbd [1.352(4)Å] suggests transfer of electron density to metallocycle; related to involvement of $[PdC_4(CO_2Me)_4]_n$ in cyclotrimerisation of $2C_2(CO_2Me)_2$ + nbd to give $C_6H_2(CO_2Me)_4$ [213].

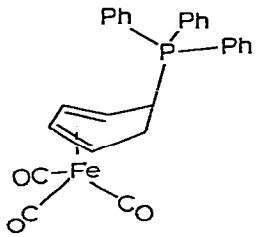
(c) η^4-*Diene complexes*



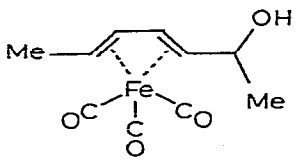
(163) $Mn(CO)(NO)(PPh_3)(exo\text{-}MeC_5H_5)$



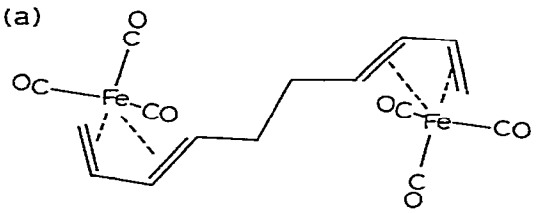
(164) $[Fe(CO)_3(C_5H_7N_2)]^+$

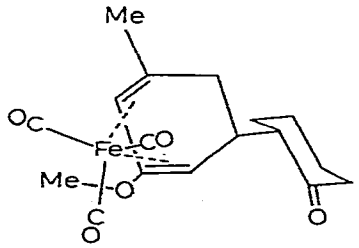


(165) $[Fe(CO)_3(C_6H_7PPh_3)]^+$

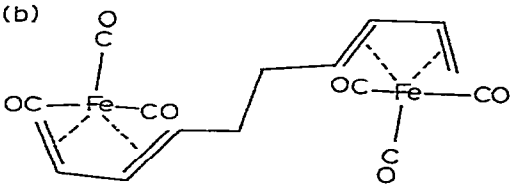


(166) $Fe(CO)_3(C_7H_{11}OH)$



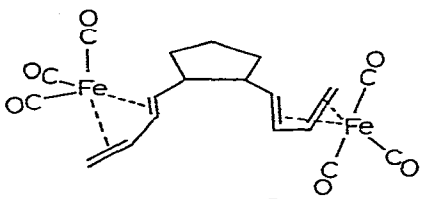


(168) $Fe(CO)_3(C_{14}H_{20}O_2)$

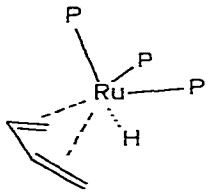


(167) $[Fe(CO)_3]_2C_{10}H_{14}$

(a) *meso* (b) racemic



(169) $[Fe(CO)_3]_2C_{13}H_{18}$



P ≡ PMe_2Ph

(170) $[RuH(C_4H_6)(PMe_2Ph)_3]^+$

(*163*) Tetrahedral Mn, with linear NO [280]. (*164*) η^4-1(1H), 2(2H)-diazepinium complex; CF_3CO_2 H-bonded to both N-H of heterocyclic cation; dihedral in C_7 ring η^4-C_4/CCNNC 35.25° [44,45]. (*165*) From attack of PPh_3 on cationic dienyl complex, with *exo* phosphine; other parameters similar to other diene-$Fe(CO)_3$ complexes [301]. (*166*) Hydrolysis product of *syn*, *syn*-1,5-dimethylpentadienyl-$Fe(CO)_3$ cation gives racemic mixture of *RR* and *SS*, confirms original proposals [72]. (*167*) *meso* and *rac* forms from $Fe(CO)_5$ + penta-1,4-dien-3-ol; no other structural details [165]. (*168*) From dienyl-$Fe(CO)_3$ cation + enamine; contains usual distorted cyclohexadiene ring [187]. (*169*) By ring closure of acyclic bis-[pentadienyl-$Fe(CO)_3$] dication with Zn [212]. (*170*) H not located, but probably in vacant octahedral site; *fac* isomer sterically favoured, but labile in solution [308].

See also: *223*, *263*, *334*, *349*.

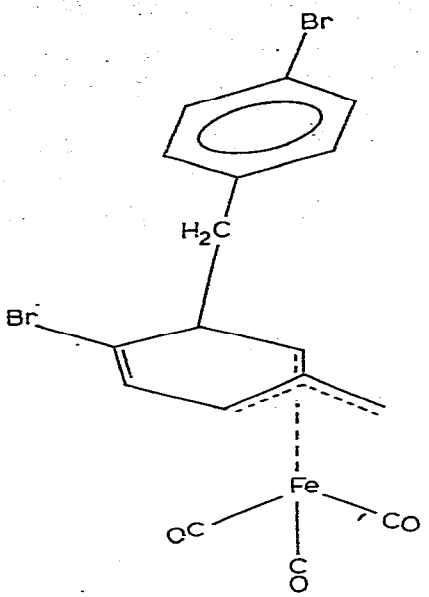
(d) η^4-*Trimethylenemethane complex*

(*171*) Comparison with other η^4-$C(CH_2)_3$ complexes [181].

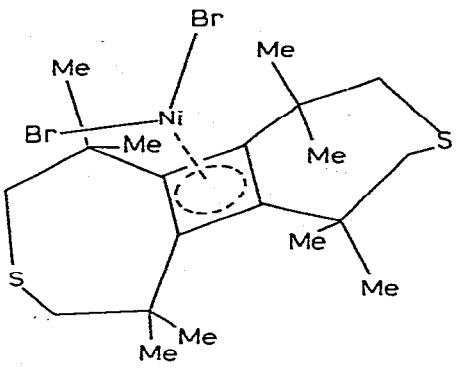
(e) η^4-*Cyclobutadiene complex*

(*172*) From $NiBr_2$ + the cyclobutadiene; unlike the C_4Me_4 complex studied earlier, (*172*) is a 16e complex [234].

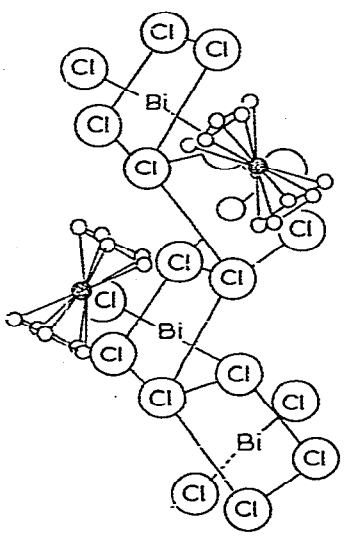
See also: *212*.



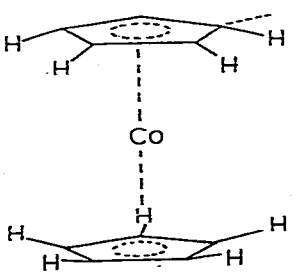
(171) $Fe(CO)_3(C_{14}H_{12}Br_2)$



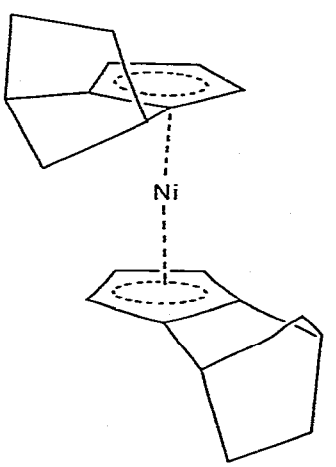
(172) $NiBr_2(C_{20}H_{32}S_2)$



(173) $[Fe(C_5H_5)_2]BiCl_4$



(174) $Co(C_5H_5)_2$



(175) $Ni(C_{10}H_{11})_2$

(a) *Cyclopentadienyls*

(*173*) From ferrocene + $BiCl_3/C_6H_6$; octahedral Bi in infinite chain,

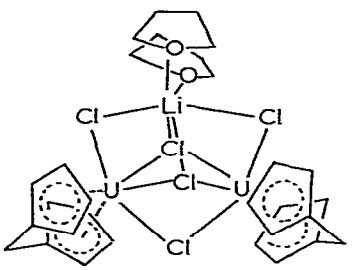
with cations occupying spaces between $(BiCl_4)_n$ chains; C_5 rings eclipsed, Fe–C (ring) 1.70 (cf. ferrocene, 1.66Å) [68].

(*174*) Eclipsed rings, with mean Co–C 2.119(3), C–C 1.429(2)Å; dynamic Jahn–Teller effect confirmed; H bent towards Co by 2.1(0.8)° [14].

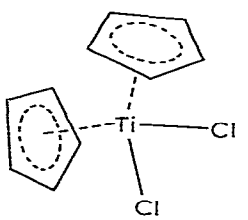
(*175*) η^5-isodicyclopentadienyl, paramagnetic; eclipsed C_5 rings, CH_2 bridges directed toward Ni; Ni-centroid 1.824(3)Å; paper also describes NMR measurements of unpaired spin density in series of related complexes [228].

(b) *Cyclopentadienyls containing halides or anionic ligands*

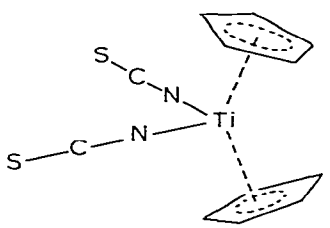
(*176*) U bonded to 2 rings, 1 terminal Cl and 3 bridge Cl; Li approximately octahedral, coordinated to bridging Cl to give discrete neutral trinuclear units; it is not clear how far long Li–Cl



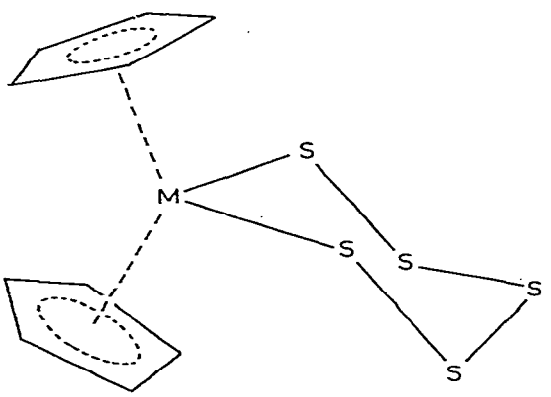
(176) $Li(thf)_2U_2Cl_5[(C_5H_4)_2CH_2]_2$



(177) $TiCl_2(C_5H_5)_2$

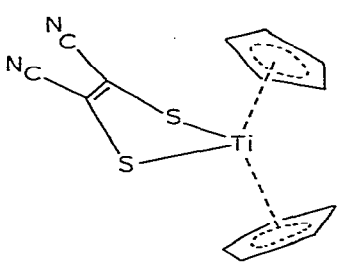


(178) $Ti(NCS)_2(C_5H_5)_2$

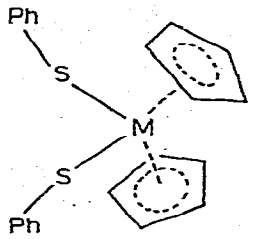


$M(S_5)(C_5H_5)_2$

(179) M = Ti; (180) M = V

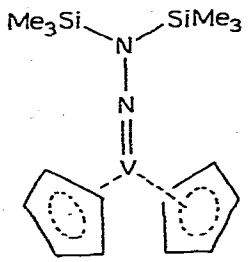


(181) $Ti[S_2C_2(CN)_2](C_5H_5)_2$

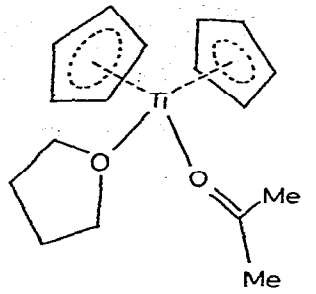


$M(SPh)_2(C_5H_5)_2$

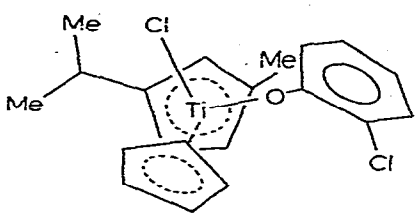
(182) M = Ti; (183) M = V



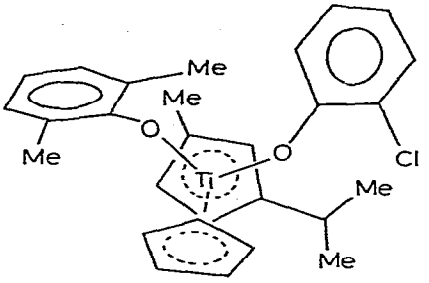
(184) $V[N_2(SiMe_3)_2](C_5H_5)_2$



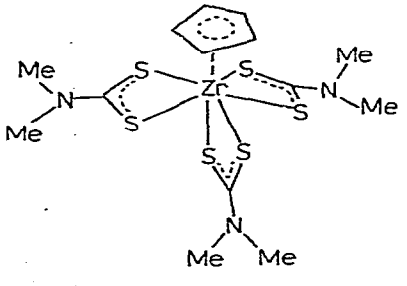
(186) $[Ti(thf)(Me_2CO)(C_5H_5)_2]^+$



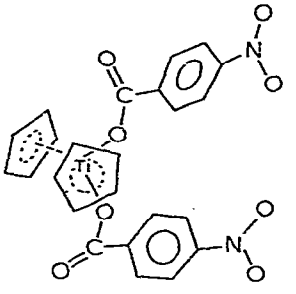
(185) $TiCl(OC_6H_4Cl)(C_5H_5)(C_5H_3MePr^i)$



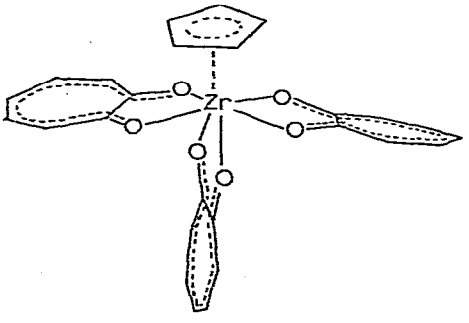
(187) $Ti(OC_6H_4Cl)(OC_6H_3Me_2)(C_5H_5)(C_5H_3MePr^i)$



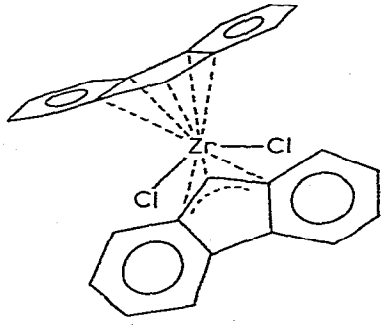
(189) $Zr(S_2CNMe_2)_3(C_5H_5)$



(188) $Ti(OCOC_6H_4NO_2)_2(C_5H_5)_2$



(190) $Zr(trop)_3(C_5H_5)$

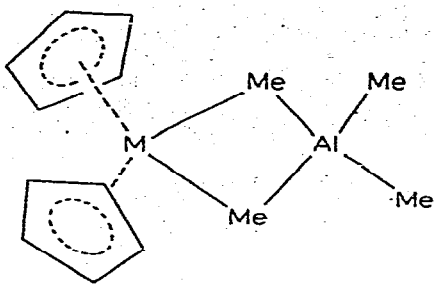


(191) $ZrCl_2(C_{13}H_9)_2$

interactions (2.74Å) hold structure together [249]. (*177*) Distorted tetrahedral Ti, staggered C_5 rings; ring normals 130.97°, Ti-Cl 2.364(3)Å [67]. (*178*) Eclipsed Cp rings, angle between ring normals 133.7° [94]. (*179*)(*180*) S_5 in chair conformation; increase in S-V distances (over S-Ti) arises because (i) unpaired electron occupies MO which is antibonding with respect to the V-S bonds, (ii) increased intramolecular repulsive forces from shorter M-C_5H_5 distances; see also (*182*), (*183*) [70]. (*181*) Staggered Cp rings (Ø 21°), angle between ring normals 130.7° [94]. (*182*)(*183*) Isomorphous complexes, distorted tetrahedral, with V-S 2.448(3), Ti-S 2.395(8)Å (but covalent radii: V 1.22, Ti 1.32Å) for same reasons as discussed above for (*179*) and (*180*); discussion of bonding in these complexes suggests Ballhausen-Dahl model incorrect [250]. (*184*) Coordinated isodiazene from $V(C_5H_5)_2 + N_2(SiMe_3)_2$; V=N 1.666(6), N-N 1.369(9); C_5 rings tend to η^3 with 0.2Å difference in V-C bonds, also bent by 6.2° away from metal [178]. (*185*)(*187*) Confirms stereospecific substitution of OR by Cl, and establishes absolute configuration of diastereoisomers: (*185*) m.p. 134°, (*187*) m.p. 171° [227]. (*186*) Paramagnetic complex from $TiCl(C_5H_5)_2 + ZnB_{10}H_{12}$ [189]. (*188*) Reference [264]. (*189*) Pentagonal bipyramid, axial C_5H_5; crowding leads to short S...S and C...S contacts [146]. (*190*) Distorted pentagonal bipyramid, with axial C_5H_5, 5 equatorial Zr-O nearly coplanar [289]. (*191*) One η^3, one η^5 fluorenyl groups, staggered, to accommodate bulky ligands, which have 10 C....Cl distances less than sum of van der Waals radii; ring normals opened out by *ca.* 17° from tetrahedral angle, and increased distance from Zr by *ca.* 0.1Å [287].

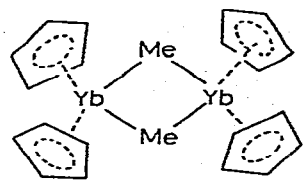
(c) *Cyclopentadienyls containing other hydrocarbon ligands*

(*192*)(*193*) From $MCl_2(C_5H_5)_2 + LiAlMe_4$; Al tetrahedral, considered as $AlMe_4$ derivatives; (*192*) Fluxional, bridge and terminal alkyl groups exchange [145]. (*194*) From (*193*) and py; symmetrical $YbMe_2Yb$ double bridge, with angle at C *ca.* 86.6° [253]. (*195*) Reference [185].

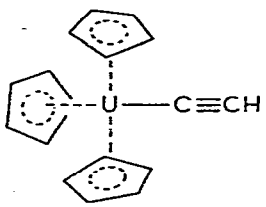


$M(C_5H_5)_2Me_2AlMe_2$

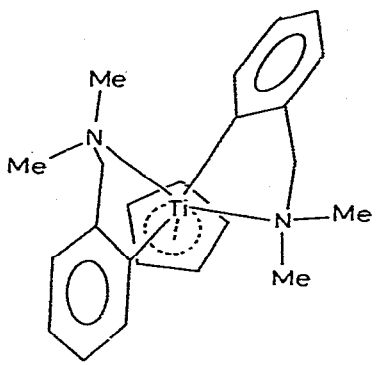
(192) M = Y; (193) M = Yb



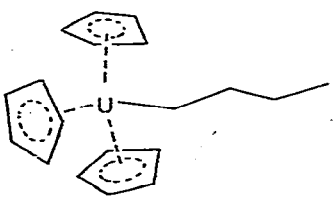
(194) $[YbMe(C_5H_5)_2]_2$



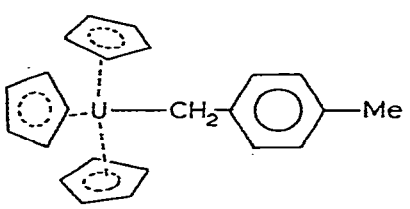
(195) $U(C_2H)(C_5H_5)_3$



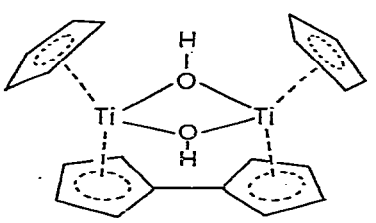
(198) $Ti(C_6H_4CH_2NMe_2)_2(C_5H_5)$



(196) $UBu(C_5H_5)_3$



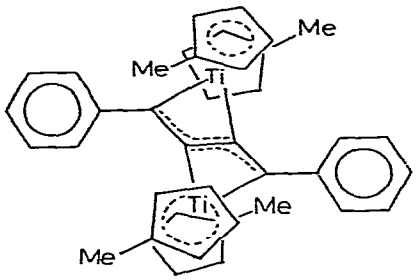
(197) $U(CH_2C_6H_4Me)(C_5H_5)_3$



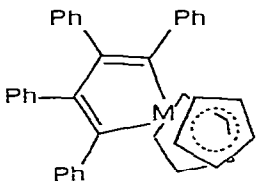
(199) $[Ti(OH)(C_5H_5)]_2C_{10}H_8$

(*196*)(*197*) Both have unusual geometries, with U-C-C 128°; adjacent carbons in (*196*) have C-C-C angles of 123.3(C_β) and 116.3(C_γ), respectively, while C-C bonds are shorter than normal; this effect ascribed to ionicity of the alkyl group [215]. (*198*) Paramagnetic; chelate 5-ring folded along Ti-CH_2 127.9°, with dihedral between

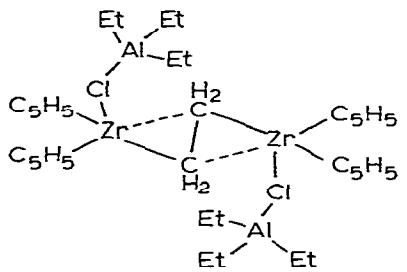
2 bidentate planes 112.4°; approximate *trans* configuration [262]. (*199*) From $[TiH(C_5H_4)(C_5H_5)]_2 + H_2O$; confirms fulvalene structure for "titanocene", with μ-H replacing μ-OH found here; solvated thf H-bonds to one OH [226]. (*200*) From $TiCl(C_5H_4Me)_2 + NaC_2Ph$, with oxidative coupling of PhC_2 residues to give 1,3-η^2;2,4-η^2-diphenylbutadiene, also obtained from



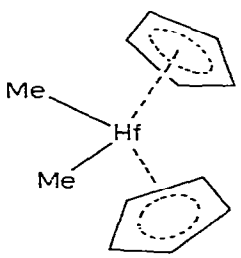
(200) $[Ti(MeC_5H_4)_2]_2C_4Ph_2$



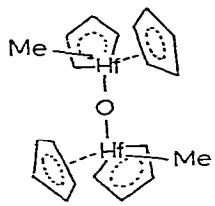
$(C_5H_5)_2MC_4Ph_4$.
(201) M = Ti; (202) M = Hf



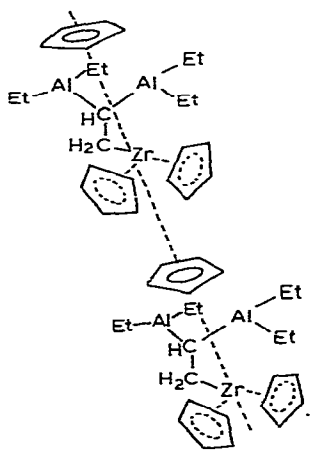
(203) $[Zr(ClAlEt_3)(C_5H_5)_2]_2C_2H_4$



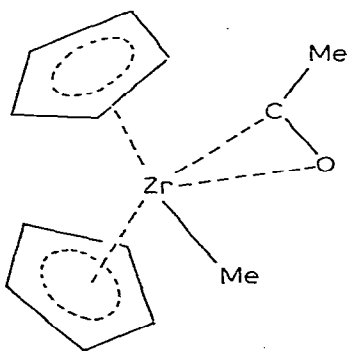
(205) $HfMe_2(C_5H_5)_2$



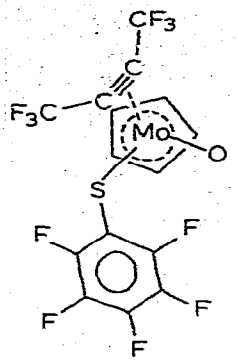
(206) $[HfMe(C_5H_5)_2]_2O$



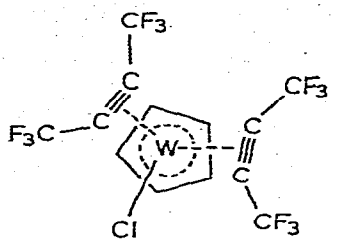
(204) 2 molecules of
$[(C_5H_5)_2ZrCH_2CH(AlEt_2)_2]^+(C_5H_5)^-$



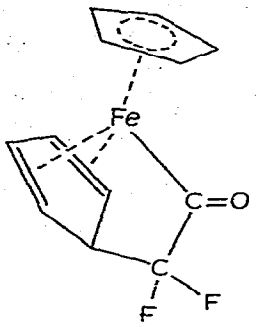
(207) $ZrMe(COMe)(C_5H_5)_2$



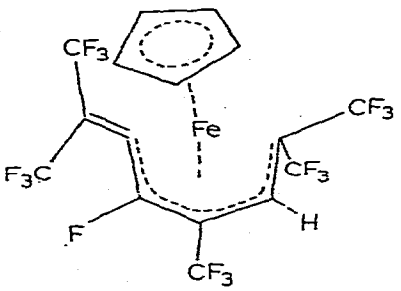
(208) $MoO(SC_6F_5)[C_2(CF_3)_2](C_5H_5)$



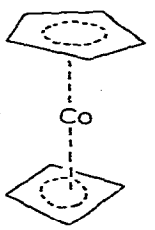
(209) $WCl[C_2(CF_3)_2]_2(C_5H_5)$



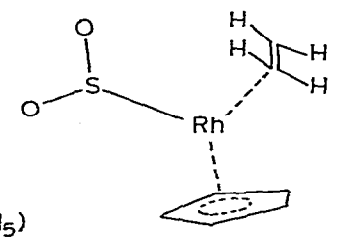
(210) $Fe(COCF_2C_5H_5)(C_5H_5)$



(211) $Fe[C(CF_3)_2CHC(CF_3)CFC{:}C(CF_3)](C_5H_5)$



(212) $Co(C_4H_4)(C_5H_5)$

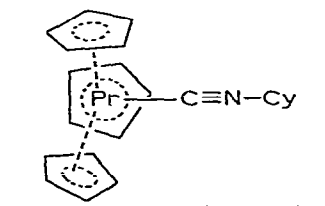


(213) $Rh(C_2H_4)(C_5H_5)(SO_2)$

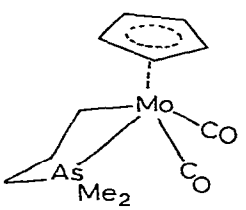
"$Ti(C_5H_5)_2$" + PhC_2C_2Ph; Ti-C bonds consistent with σ-bonded 4-membered metallocycles [354]. (*201*)(*202*) Metallocycles with localised C-C, C=C bonds; distances: M-C 2.172, 2.141 (Ti), 2.22, 2.18 (Hf); centroid-M-centroid both 134.5°; staggered C_5 rings [343]. (*203*)(*204*) Zr-C-C(Al) always *ca.* 76° in these, and also in $(C_5H_5)_2Zr(Cl)CH_2CH(AlEt_2)_2$; note isolated $C_5H_5^-$ symmetrically arranged between 2 Zr centres in (*204*) [235]. (*205*)(*206*) Hf-C bond distance sensitive to subtle effects; Hf-Me 2.318, 2.382 in (*205*), 2.295Å in (*206*); angle of centroid normals 132.1, 128.5°, respectively; nearly linear Hf-O-Hf 173.9(3)° [102]. (*207*) Side-on acyl as 3e donor, Zr-C(Me) 2.197(6), Zr-O 2.290(4), C-O 1.211(8)Å, long Zr-Me [2.336(7)Å] [121]. (*208*) Obtained with (*217*) from $Mo(SC_6F_5)(CO)_3(C_5H_5)$ + $C_2(CF_3)_2$; Mo-O 1.68Å, suggests Mo≡O system; alkyne C≡C 1.267(12)Å [153]. (*209*) 16e complex, C≡C 1.27(2)Å, CCC 139(2)°; paper also

describes further reactions with other alkynes [116]. (*210*) From $Fe(CO)_2(\eta^1\text{-}C_5H_5)(\eta^5\text{-}C_5H_5)$ + C_2F_4 via unusual cleavage of latter [92]. (*211*) Obtained in same reaction as (*236*) in small yield; fluorinated pentadienylidene ligand acts as 5e donor [112]. (*212*) At -35°; Co-C 1.964(av.)(C_4), 2.036(av.)(C_5); C-C 1.44(av.)(C_4), 1.39Å(av.)(C_5); C_4 ligand almost square [64]. (*213*) Planar S-bonded SO_2, with Rh-S 2.096(2)Å; general MO model developed for M-SO_2 complexes [41].

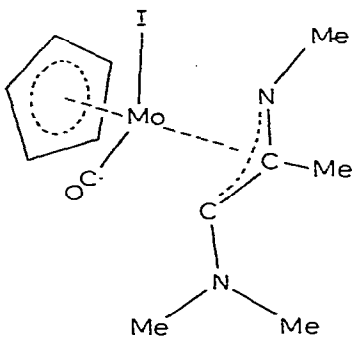
(d) *Cyclopentadienyls containing CO, CNR, PR_3 or NO ligands*



(214) $Pr(CNCy)(C_5H_5)_3$

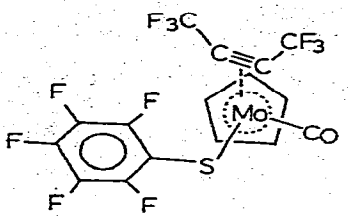


(215) $Mo[CH_2(CH_2)_2AsMe_2](CO)_2(C_5H_5)$

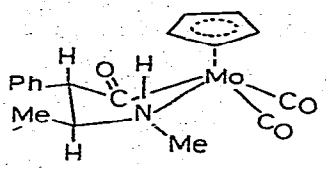


(216) $MoI(CO)[C(CMe{:}NMe)NMe_2](C_5H_5)$

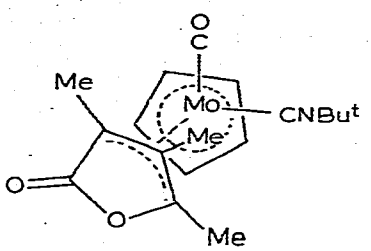
(*214*) Trigonal pyramid, with Cp centroids forming base; isocyanide slightly bent, with Pr-C-N 174.1(1.1)°, Pr-C(CN) 2.65Å [252]. (*215*) Reductive cyclisation of halopropylarsine-Mo halide complex with NaHg; Mo-As 2.533(2)Å is shorter than previous reports [104]. (*216*) Contains novel η^3-imino-dimethylamino-carbene ligand [122]. (*217*) Slow oxidation of CO in this 16e system gives (*208*); alkyne C≡C 1.298(6)Å, significantly different from (*208*) [153]. (*218*) Asymmetric synthesis; absolute configuration at Mo is (1*R*, 2*S*, 3*S*) [198]. (*219*) Formed via vinyl ketone complex by oligomerisation of C_2Me_2 with $MoMe(CO)_3(C_5H_5)$, followed by addition of Bu^tNC; lactone



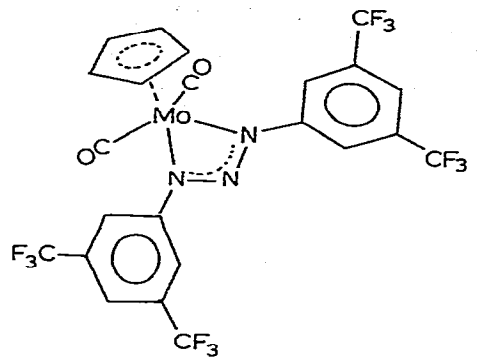
(217) $Mo(CO)[C_2(CF_3)_2](SC_6F_5)(C_5H_5)$



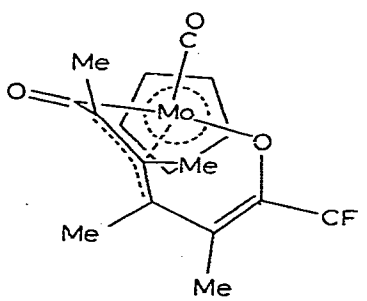
(218) $Mo(CoCHPhCHMeNHMe)(CO)_2(C_5H_5)$



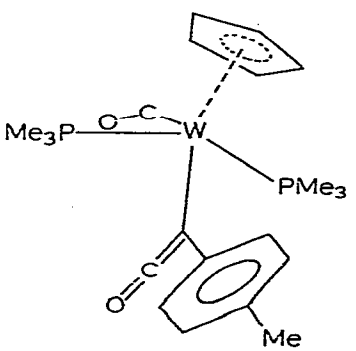
(219) $Mo(CO)(CNBu^t)[OC(O)C_2Me_2CMe](C_5H_5)$



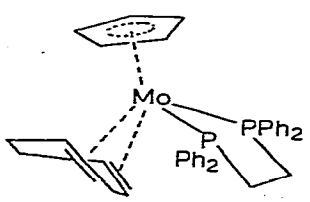
(220) $Mo(CO)_2(N_3[C_6H_3(CF_3)_2]_2)(C_5H_5)$



(221) $Mo[C(O)C_4Me_4C(CF_3)O](CO)(C_5H_5)$

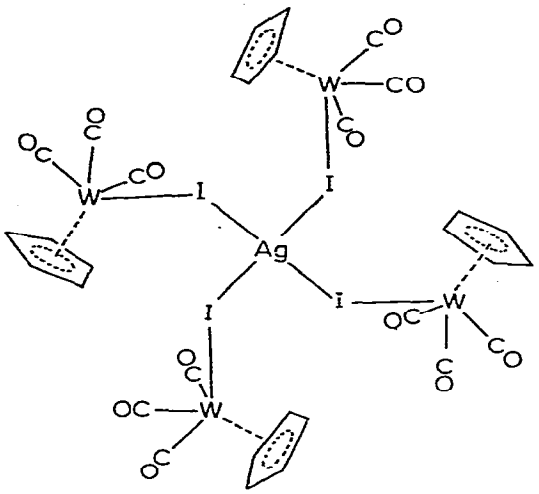


(222) $W[C(p\text{-tol}):C:O](CO)[PMe_3]_2(C_5H_5)$



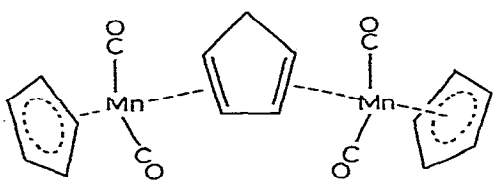
(223) $[Mo(dppe)(C_6H_8)(C_3H_5)]^+$

ring formed stepwise [186]. (*220*) Some σ,π bonding of triazenide to Mo suggested by Mo-N distances of 2.12Å, and N-N-N angle 101° [259]. (*221*) From $Mo(CF_3)(CO)_3(C_5H_5)$ + C_2Me_2 via an oxidative reaction after insertion of second C_2Me_2 into vinyl ketone; contains 8-membered

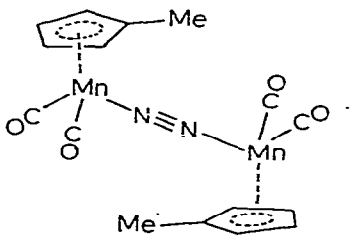


(224) $\left\{[W(CO)_3(C_5H_5)I]_4Ag\right\}^+$

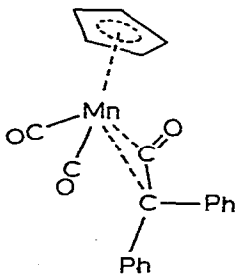
metallocycle, with ligand bonded to Mo via 2σ, η^3 linkage, distorted to σ–π localised form [186]. (*222*) From tolylcarbyne complex + PMe_3; W–C 2.27(2), short W–P 2.471(6)Å [244]. (*223*) From bis(acetonitrile) cation and 1,3-cyclohexadiene [341]. (*224*) Silver coordinated to 4 I



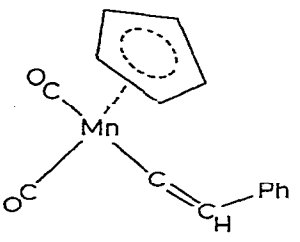
(225) $[Mn(CO)_2(C_5H_5)]_2C_5H_6$



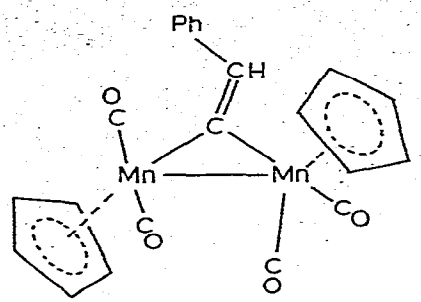
(226) $[Mn(CO)_2(C_5H_5)]_2N_2$



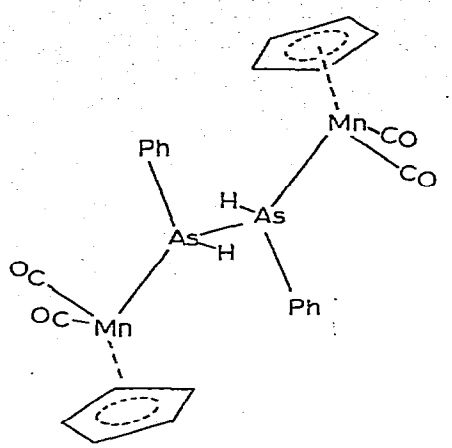
(227) $Mn(CO)_2(Ph_2C{:}C{:}O)(C_5H_5)$



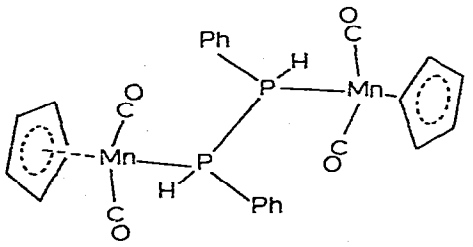
(227a) $Mn(CO)_2(C{:}CHPh)(C_5H_5)$



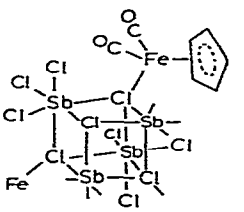
(228) $Mn_2(CO)_4(C{:}CHPh)(C_5H_5)_2$



(230) *meso*-$[Mn(CO)_2(C_5H_5)]_2As_2H_2Ph_2$

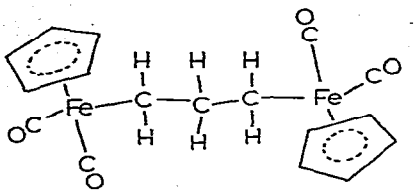


(229) $[Mn(CO)_2(C_5H_5)]_2P_2H_2Ph_2$

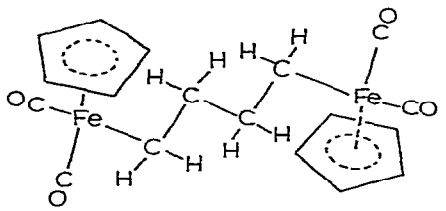


(231) $[Fe(CO)_2(C_5H_5)Cl]_4[SbCl_3]_4$

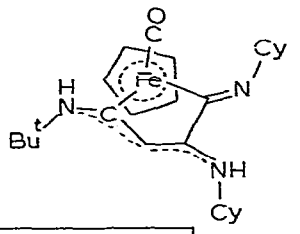
in compressed tetrahedron, with 4 I-Ag-I 111.71(3), 2 angles at 105.0(6)° [319]. (*225*) C=C bond lengthened to 1.41Å; Mn-CO (1.77) is shorter than in present $Mn(CO)_3(C_5H_5)$ (1.80Å); Mn groups are *trans* as originally suggested [211]. (*226*) Almost linear Mn-N-N-Mn [176.5(4)°] [53]. (*227*) Contains η^2-ketene, with C-C-O 145° (out-of-plane deformation); Mn-C(Ph_2) 2.17, Mn-C(O) 1.96, C-C 1.35Å [241]. (*227a*) From $Mn(CO)_2(thf)(C_5H_5)$ + HC_2Ph; phenylvinylidene ligand, although Mn-C [1.68(2)Å] suggests metalallene formulation [156]. (*228*) Contains phenylvinylidene carbene bridging Mn-Mn bond [156]. (*229*) Similar to (230), structure of *meso* complex; short Mn-P 2.216(3)Å [291]. (*230*) From $Mn(CO)_2(thf)(C_5H_5)$ + $PhAsH_2$; *meso* complex determined, and epimerisation reactions described [290]. (*231*) From $[Fe(CO)_2(C_5H_5)]^-$ + $SbCl_3$; 12 edges of Sb_4Cl_4 core 3.175-3.375(5)Å; comparison of bonding with $[Fe(CO)_2(C_5H_5)Cl]_4[SbCl_3]_2$ described earlier shows steric crowding in both, with steric deactivation of



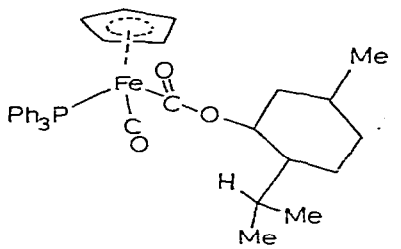
(232) n = 3



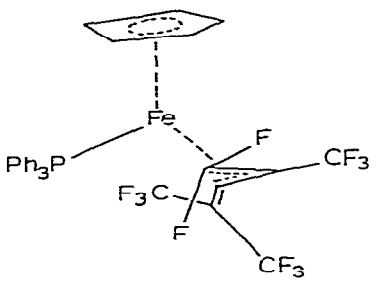
(233) n = 4
$[Fe(CO)_2(C_5H_5)](CH_2)_n$



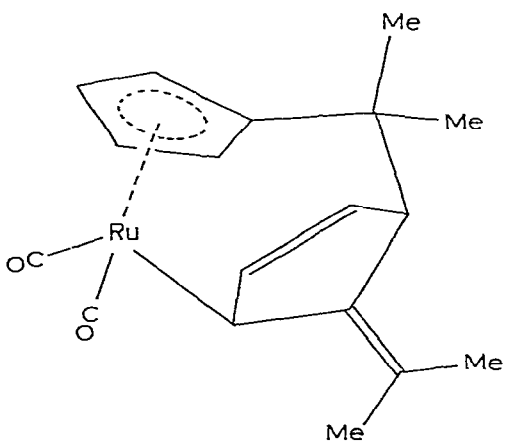
(234) $Fe[C(:NCy)C(NHCy)CHC(NHBu^t)](CO)(C_5H_5)$



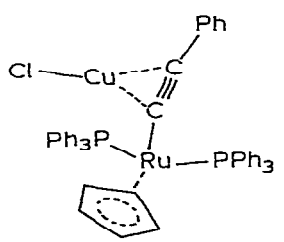
(235) $Fe[C(O)OC_6H_9MePr^i](CO)(PPh_3)(C_5H_5)$



(236) $Fe[CF_2C(CF_3)C{:}C(CF_3)_2](PPh_3)(C_5H_5)$



(237) $Ru(CO)_2(C_{16}H_{20})$

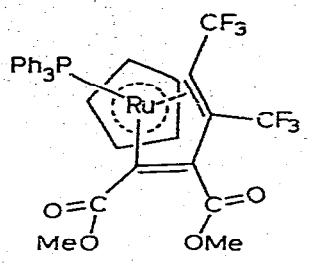


(238) $Ru[C_2Ph(CuCl)](PPh_3)_2(C_5H_5)$

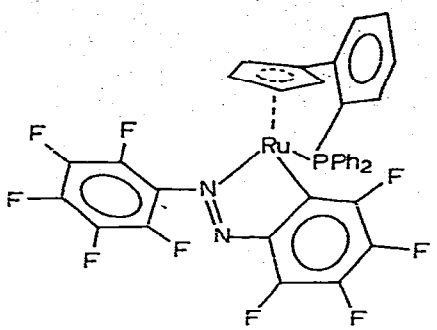
lone pair, and 6s orbital available for charge donation from bridge Cl; diagram shows full coordination only for rear Sb and Cl [303].

(232)(233) Two Fe linked by polymethylene chains [184].

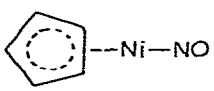
(234) From $FeMe(CO)(CyNC)_2(C_5H_5)$ + Bu^tNC; electron delocalisation in



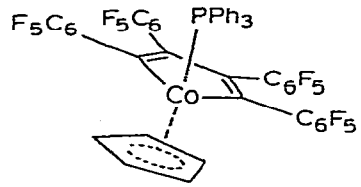
(239) $Ru[C_2(CO_2Me)_2C_2H(CF_3)_2](PPh_3)(C_5H_5)$



(240) $Ru(C_6F_4N_2C_6F_5)(Ph_2PC_6H_4C_5H_4)$



(244) $Ni(NO)(C_5H_5)$

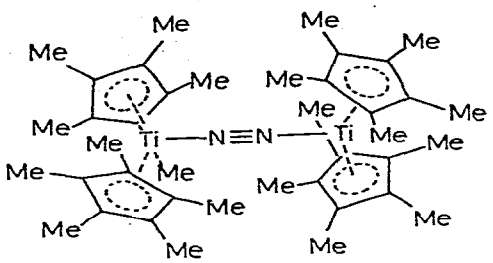


$MC_4(C_6F_5)_4(PPh_3)(C_5H_5)$

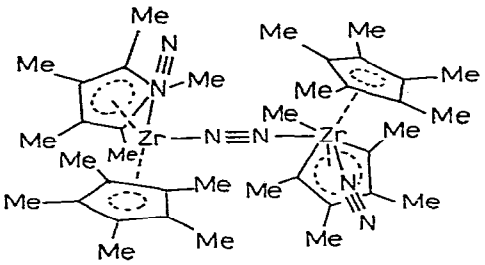
(242) M = Co; (243) M = Rh

NH-C-CH-C-NH portion of chelate ligand [296]. (*235*) Absolute configuration determined as (*S*), with Fe as chiral centre [335]. (*236*) From butadienyl complex [analogue of (*63*)] + UV, PPh_3; ligand bonded via η_3-allyl group [112,312]. (*237*) From dimerisation of dimethylfulvene (originally proposed to chelate via Ru-CMe_2 bond) [200]. (*238*) From $RuCl(PPh_3)_2(C_5H_5)$ + CuC_2Ph; acetylide non-linear, with Ru-C-C 173.0(8), C-C-Ph 164.8(11)°, and C≡C 1.242(13)Å [381]. (*239*) Confirms unusual mode of addition of $C_2(CF_3)_2$ to $C_2(CO_2Me)_2$ complex [326]. (*240*) Steric crowding gives tilted C_5 ring, plane inclined 13.9° from RuL_3 plane, free C_6F_5 twisted out of chelate ring plane by 64.6°; short F...N and F...H contacts, with long N-N 1.310(7), short Ru-C 2.013(6), and different N-C 1.365(8), 1.431(8)Å, latter to C_6F_5 [333]. (*241*) Entry deleted. (*242*)(*243*) Metallocycles, both compounds isomorphous; C_4 similar to butadiene, with C-C bonds 1.300, 1.487 (Co); 1.343, 1.457Å (Rh) [387]. (*244*) Reference [15].

e) *Cyclopentadienyl-dinitrogen complexes*



(245) $[Ti(C_5Me_5)_2]_2N_2$



(246) $[Zr(N_2)(C_5Me_5)_2]_2N_2$

(*245*)(*246*) Ti complex contains linear Ti-N-N-Ti bridge, Zr complex also has linear end-on bonded N_2; Me groups bent away from metal; N-N bonds: bridging 1.155, 1.165 (Ti), 1.182(5) (Zr); terminal 1.116(8)Å (Zr); qualitative MO schemes given for both molecules [356, 357,358].

See also: *226*.

(f) *Substituted ferrocenes*

(*247*) Bu^t groups staggered, bent out of C_5 planes by 7° ($C_5H_3Bu^t_2$) and 9° ($C_5H_4Bu^t$); rings midway between eclipsed and staggered (Ø 19°) [256].

(*248*) From cleavage of ferrocene by $AlCl_3$; C_5 rings distorted from planarity, with Fe-C(C_5H_5) 2.008(3), 2.050(2)Å [222,223].

(*249*) Si-bridged C_5 rings tilted 19.2°, with Fe-C 2.01-2.11Å, C-Si-C 99.1°; exocyclic C-Si bond angled at 40° to ring plane [248].

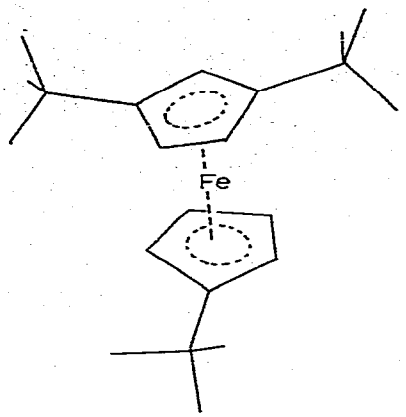
(*250*) β-form, m.p. 184°; rings almost eclipsed (Ø 3°) [268].

(*251*) From 1,1'-$(ClHg)_2Fc$ + $AlMe_3$, rearrangement with H transfer from one C_5 ring to the other, with one Cp C bridging 2Al, other attached to one Al; no Fe-Al bond [294].

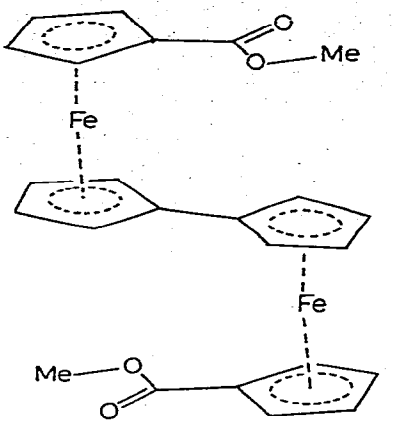
Complexes *308*, *310*, *311*, *315*, *320*, *324*, *348*, *390*, *397*, *398*, *400*, *420*, *421*, *422*, *423*, *430*, *431* and *432* also contain η^5-cyclopentadienyl ligands.

(g) *Other* η^5*-ligands*

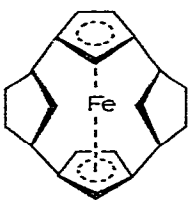
(*252*) From Fe vapour and cycloheptatriene; open C_5 faces are skew to each other [139]. (*253*) From cycloheptatriene + $[RuCl_2(C_6H_6)]_n$ + Pr^iMgBr; similar to (*252*) [140].



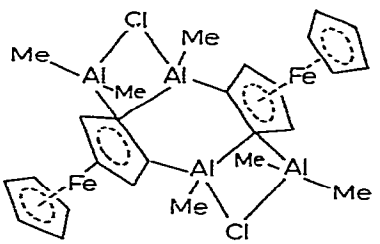
(247) $Fe(C_5H_4Bu^t)(C_5H_3Bu^t_2)$



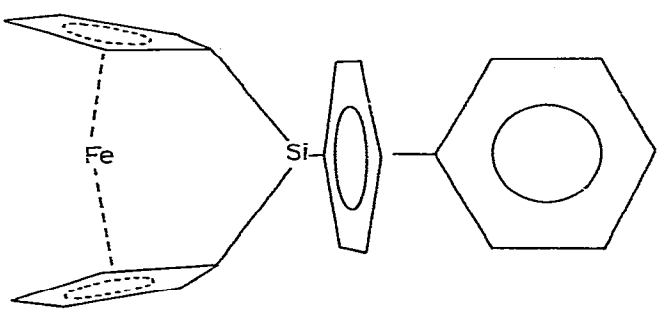
(250) $\left[Fe(C_5H_4CO_2Me)(C_5H_4)\right]_2$



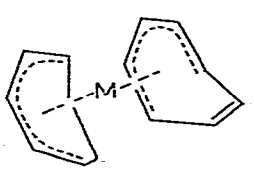
(248) *syn*-bis(cyclopentylene)-(1,1')(3,3') ferrocene



(251) $\left[(C_5H_5)Fe(C_5H_3)Al_2Me_3Cl\right]_2$



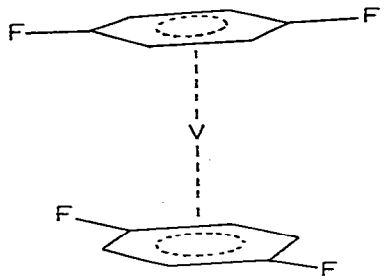
(249) $Fe(C_5H_4SiPh_2C_5H_4)$



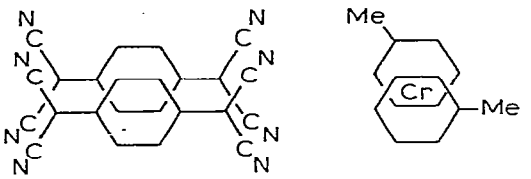
(252) M = Fe; (253) M = Ru

η^6-LIGANDS

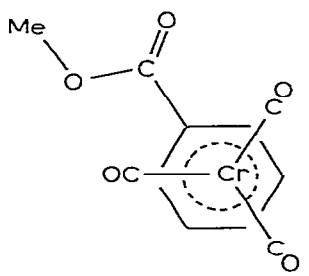
(a) *Arene complexes*



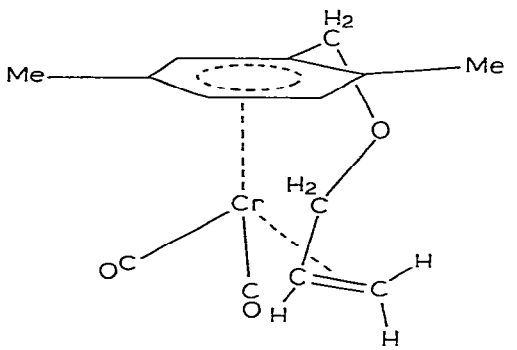
(254) $V(C_6H_4F_2)_2$



(255) $[Cr(PhMe)_2](tcnq)$

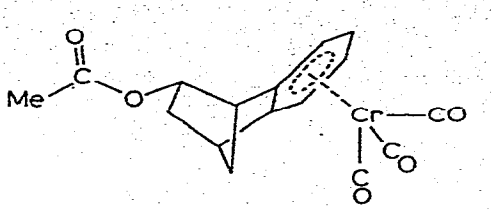


(257) $Cr(CO)_3(PhCO_2Me)$

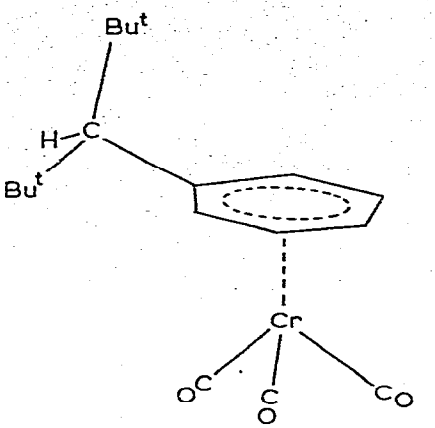


(258) $Cr(CO)_2(Me_2C_6H_3CH_2OCH_2CH{:}CH_2)$

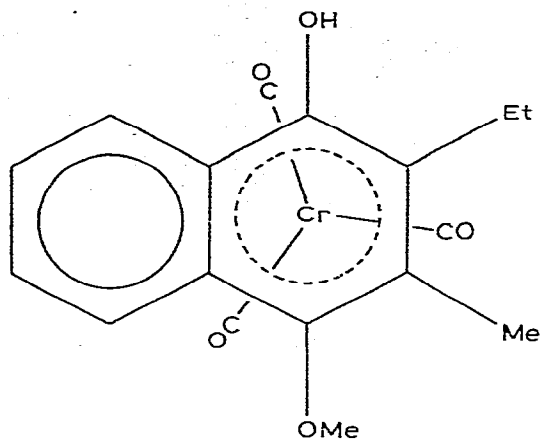
(*254*) Rings parallel, with small boat deformation, with C(F) displaced away from V; dihedral between 2F on opposite rings 53°; C-C bonds differ: HC-HF 1.386, HC-CH 1.415(4)Å, angles at C 123.5, 118.1(3)°, respectively [90]. (*255*) 1:1 adduct of $[Cr(PhMe)_2]^+$ and tcnq radical anion, consists of infinite columns of anions and cations; anion columns are conducting [135]. (*256*) Anions and cations stack separately along *c* axis; charge delocalised, with anion interplanar distance 3.16(1)Å [136]. (*257*) Comparison made with $Cr(CO)_2(CS)(PhCO_2Me)$: latter has shorter Cr-C(benzene) bonds: 2.209(2) *vs* 2.226(3)Å [78]. (*258*) Crystal racemic; contains η^8-benzyl allyl ether ligand via η^6-arene + η^2-olefin [138]. (*259*) Structure determination relates to controversy



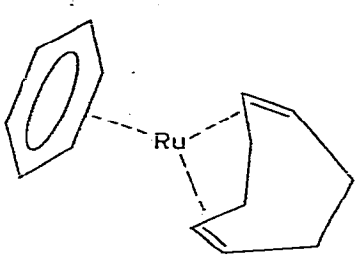
(259) $Cr(CO)_3(C_{13}H_{14}O_2)$



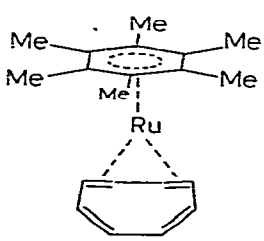
(260) $Cr(CO)_3(C_6H_5CHBu^t_2)$



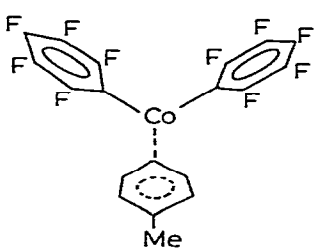
(261) $Cr(CO)_3(C_{14}H_{16}O_2)$



(262) $Ru(C_6H_6)(C_8H_{12})$



(263) $Ru(C_6Me_6)(C_8H_8)$



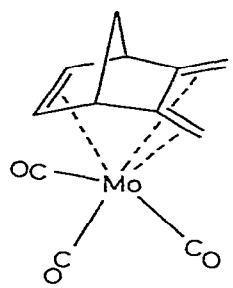
(264) $Co(C_6F_5)_2(PhMe)$

over detailed mechanism of solvolysis of benzonorbornenyl sulphonates; neither *exo* substituent causes distortion of norbornene system, C atoms of C_6 ring are staggered with respect to the $Cr(CO)_3$ group [164]. (*260*) Molecular strain results in arene and $(CO)_3$ planes being inclined at 8.0°; $Cr(CO)_3$ twisted 44.4° from eclipsing alkyl group; 2 conformers detected in solution [201]. (*261*) η^6-Tetrasubstituted

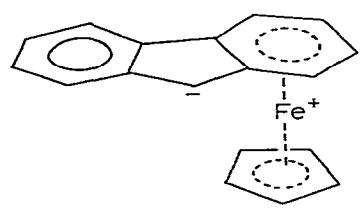
naphthalene, from $Cr(CO)_5[CPh(OMe)]$ + MeC_2Et; alkyne incorporated into 2,3 positions of naphthalene [183]. (*262*) From cod + $[RuCl_2(C_6H_6)]_n$ + Pr^iMgBr; C_6 ligand slightly distorted, with 2 Ru-C 2.197(av.), 4 Ru-C 2.255(av.), giving tub configuration [143]. (*263*) Highly fluxional; C_8 ring similar to $Ru(CO)_3(C_8H_8)$, C_6Me_6 planar [230]. (*264*) From Co vapour + C_6F_5Br, and toluene; paramagnetic; Co-C 1.931(5)Å, C-Co-C 88.3° [207].

See also: *350*.

(b) *Other* η^6*-ligands*



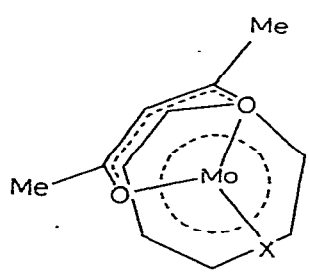
(265) $Mo(CO)_3(C_9H_{10})$



(266) $Fe(C_5H_5)(C_{13}H_9)$

(*265*) Ligand requirements make exocyclic C=C bonds to Mo asymmetric; CO groups staggered with respect to C=C; PMR of this and related less strained bicyclic trienes [93]. (*266*) η^6-Fluorenyl, best represented with Fe^+, negative C_9 ligand; unusual structure arises from preferred coordination to end ring [195].

η^7-LIGANDS

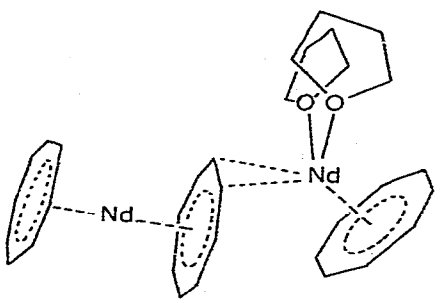


(267) $[Mo(OH_2)(acac)(C_7H_7)]^+$ (X = H_2O)
(268) $[Mo(NCS)(acac)(C_7H_7)]$ (X = NCS)

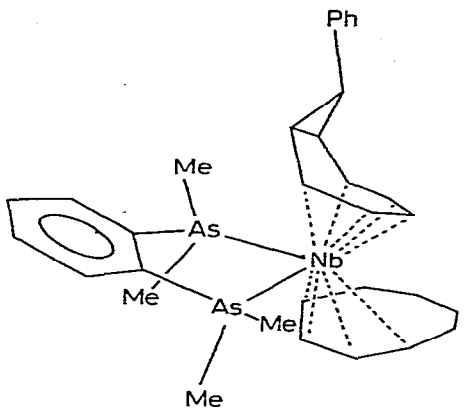
(*267*)(*268*) Paramagnetic; from corresponding tricarbonyl cation + acetylacetone (*267*) followed by SCN^- (*268*) [103].

η^8-LIGANDS

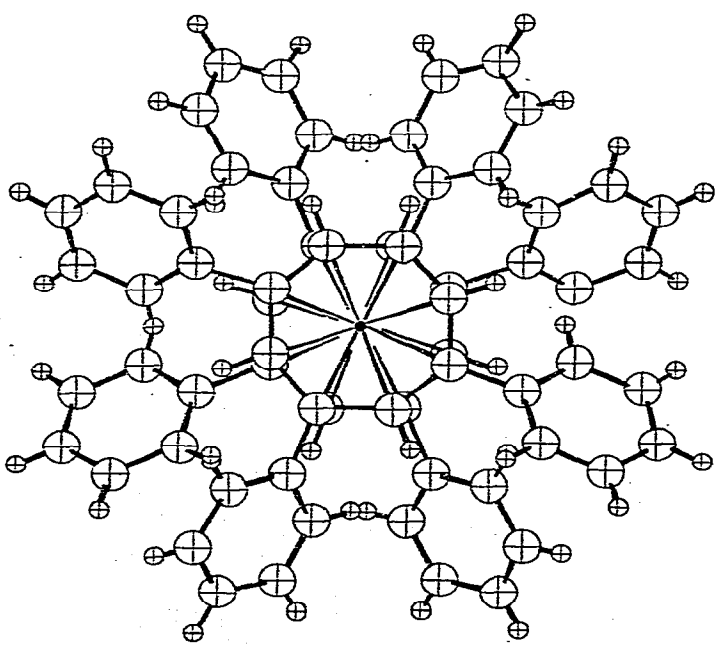
(*269*) Anion-cation pair; asymmetric $Nd(C_8H_8)_2$, with 0.1Å(av.) difference between Nd-C in 2 rings, planes inclined 8.25°; one C_8 ring asymmetrically bonded (η^2) to other Nd [174]. (*270*) Rings



(269) $[Nd(C_8H_8)(thf)_2][Nd(C_8H_8)_2]$



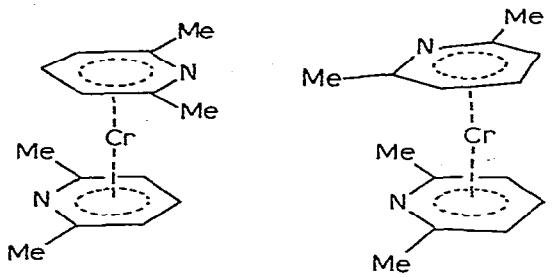
(271) $Nb(C_8H_8)(C_8H_8Ph)(diars)$



(270) $U(C_8H_4Ph_4)_2$

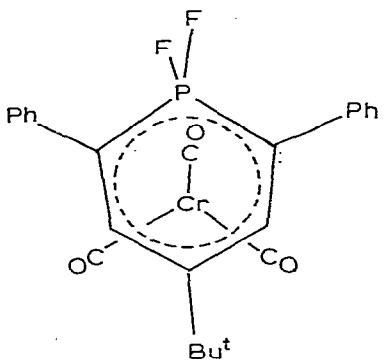
staggered, C_8 not quite eclipsed; Ph tilted 42° from plane; angles at C in C_8 ring: CPh 130, CH 140° [399]. (*271*) C_8H_8 is η^4; C_8H_8Ph is η^5-bicyclo[5.1.0]octadienyl with *endo* phenyl [322].

η-HETEROATOM LIGANDS

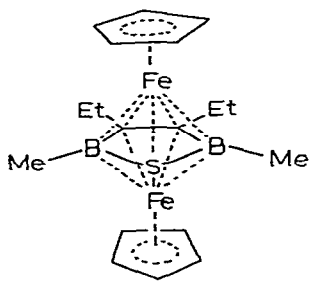


(272) $Cr(C_5H_3Me_2N)_2$

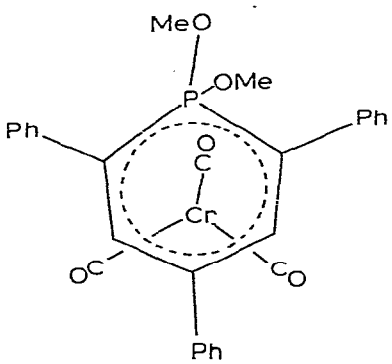
(A) Two conformations (B)



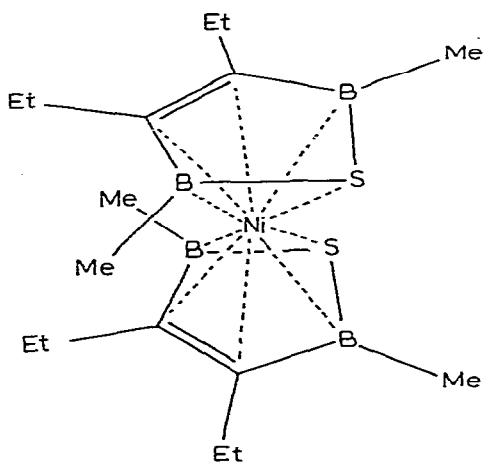
(273) $Cr(CO)_3[C_5H_2Bu^tPh_2PF_2]$



(275) $[Fe(C_5H_5)]_2(C_2Et_2B_2Me_2S)$



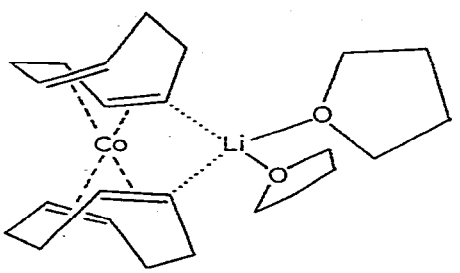
(274) $Cr(CO)_3[C_5H_2Ph_3P(OMe)_2]$



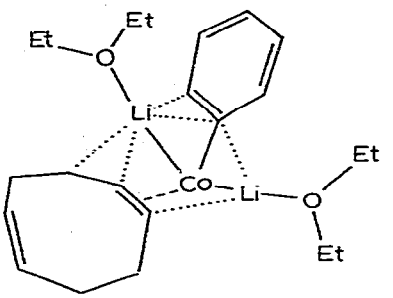
(276) $Ni(C_2Et_2B_2Me_2S)_2$

(*272*) 2 forms studied; (A) completely eclipsed, parallel rings, (B) has staggered Me groups, and boat conformation; intermolecular contacts more important in (B); H bent away from Cr [141,142]. (*273*)(*274*) η-Phosphorin ring planar, with Cr-P 2.681, 2.770Å, respectively; the relative extents of ylide and arene forms could not be decided [265]. (*275*) From the thiadiborolene and $[Fe(CO)_2(C_5H_5)]_2$, triple-decker sandwich complex; rotational disorder in Cp group [202]. (*276*) From the thiadiborolene and $Ni(CO)_4$; ligand is η^5, with electron donating groups (C=C and S) occupying approximately tetrahedral coordination positions [321].

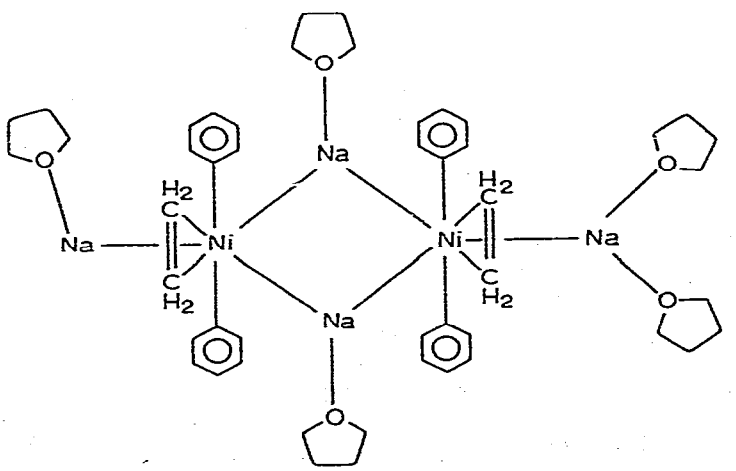
ALKALI-METAL DERIVATIVES



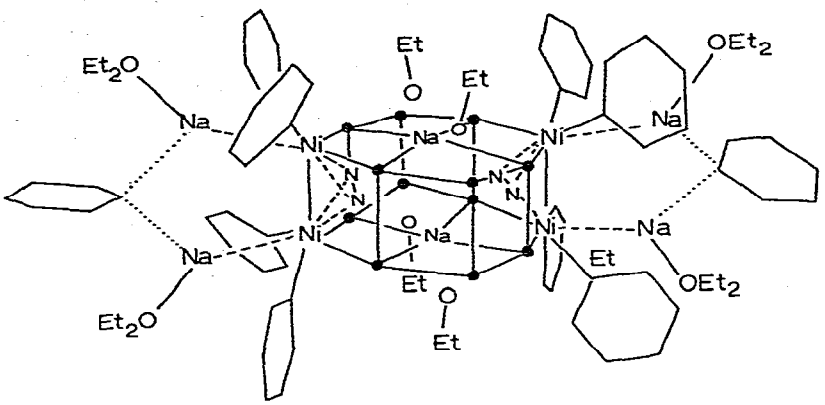
(277) $Li(thf)_2Co(C_8H_{12})_2$



(278) $[Li(OEt_2)]_2[CoPh(C_8H_{12})_2]$
one C_8H_{12} omitted



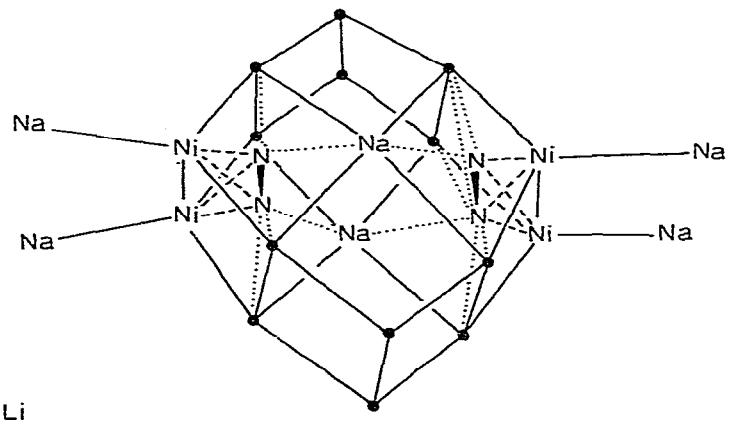
(279) $[NiPh_2(C_2H_4)]_2Na_4(thf)_5$



• Li

(280) $\left\{Ph\left[Na.OEt_2\right]_2\left[Ph_2Ni\right]_2N_2NLi_6(OEt)_4.OEt_2\right\}_2$

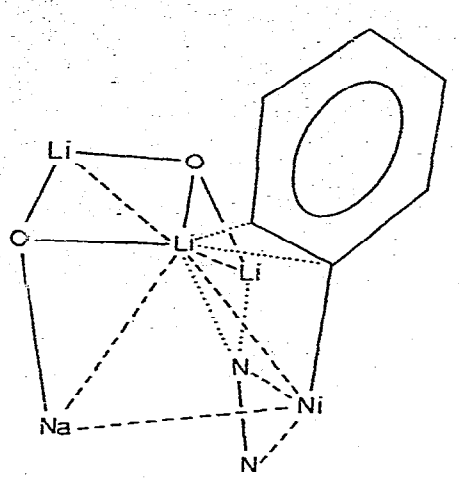
several organic groups omitted



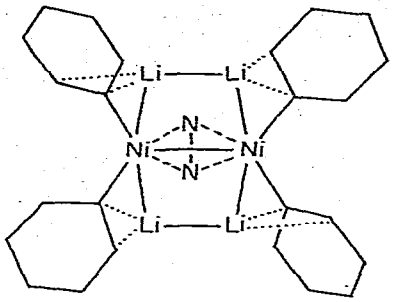
• Li

(280) Geomety of $Li_{12}Na_6Ni_4(N_2)_2$ unit

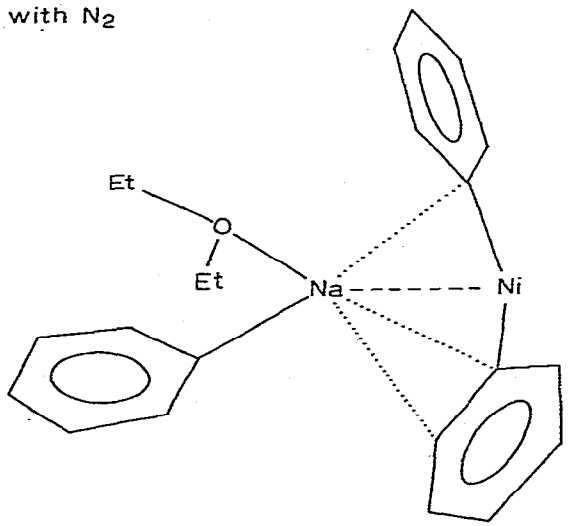
(*277*) Co has TBP geometry; cod has highly distorted boat form; no Li-Co bond, but strong ion-pair interaction with equatorial C=C [275]. (*278*) Diagram omits one conventionally-bonded cod; multicentre bonds join metals to Ph and cod ligands; 3-centre Li-Co-C(11) has Co-C(11) 1.888, Li-Co 2.39, 2.44Å [295]. (*279*) Ph and C_2H_4 are trigonal about Ni [i.e. Ni(0)], with 2 bridging Na; Na is within ion-pair bonding distance of Ph groups; one Na bonds to C_2H_4 of neighbouring molecule to give polymeric chain; one CH of olefin also forms weak ion-pair



(280) Interaction of Li atoms with N_2



(280) Interactions between $N_2N_2Ph_4$ and Li atoms

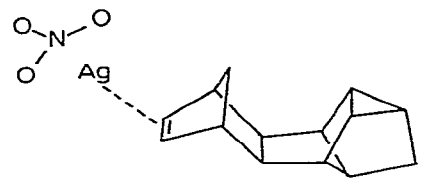


(280) Coordination of outer Na atoms

with Na; Na_2Ni_2 ring formed from 2 3-centre electron-deficient bonds, with Na-Ni 2.795, 3.037; Ni-C(Ph) 1.98-2.03Å [379]. (*280*) Contains 2 $(NiPh_2)_2N_2$ moieties linked to 2 Na, 2 $Li_6(OEt)_4(OEt_2)$ cages, with outer side of cage containing 2 $Ph(NaOEt_2)_2$ bonded to $(NiPh_2)_2N_2$ by Na-C, Na-Ni bonds; diagrams show interactions of N_2 with Ni, Li, and of Ni with outer Na; EtO groups arise from ether clearage; N_2Ni_2 interaction probably similar to alkynes bridging Co_2 or Ni_2, with negative charge on N_2 stabilised by interaction with Na^+ [401].

See also: *3, 111.*

SILVER COMPLEX



(281) $Ag(C_{14}H_{16})NO_3$

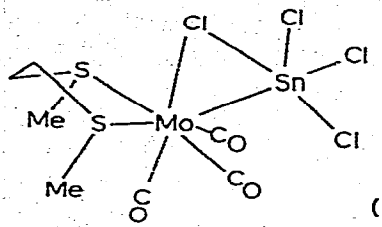
(*281*) Norbornadiene dimer from $Ni(CO)_4$-catalysed photochemical dimerisation; symmetrical C=C-Ag bond, extensive Ag-O (nitrate) interaction within crystal such that each Ag interacts with 3 NO_3 groups, and each NO_3 with 3 different Ag [137].

COMPLEXES CONTAINING METAL-METAL BONDS

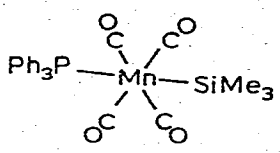
Complexes *87*, *159* and *228* also contain metal-metal bonds.

(a) *Complexes containing transition metal-main group metal bonds*

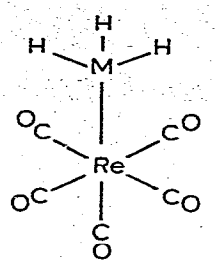
(*282*) Mo has capped octahedral geometry, with Cl bridging Sn, Mo to give irregular TBP geometry about Sn [42]. (*283*) PPh_3 *trans* to Mn-Si [281]. (*284*) New cell dimensions given; shows "striking similarities" but not isomorphous with (*287*) [49]. (*285*)(*288*) Electron diffraction studies; equatorial CO bent away from MH_3 by 4-7° in these and (*60*) [17]. (*286*) Reference [49]. (*287*) Re-Si significantly longer than in (*286*), minimises steric repulsions [49]. (*289*)(*290*) Distorted tetrahedral Sn, octahedral Mn, with CO groups bent towards Sn; Mn-Sn-Mn 126° [75]. (*291*) Similar to hydride; Sn distorted tetrahedral, crowding around $Mn(CO)_5$ fragments gives 2 Mn-Sn bonds; electronic and steric effects suggested to account for data [238]. (*292*) Short Fe-Si [2.224(9)Å]; equatorial CO and Cl staggered, CO bent towards Si, with C-Fe-Si *ca.* 86°; comparisons with



(282) $Mo(SnCl_3)Cl(CO)_3[MeS(CH_2)_2SMe]$

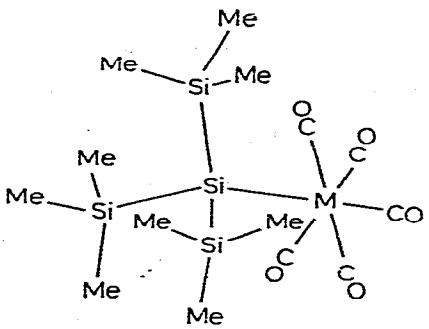


(283) $Mn(SiMe_3)(CO)_4(PPh_3)$



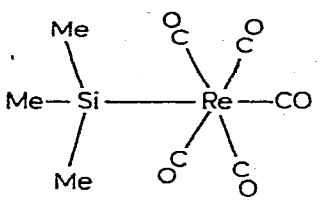
$Re(MH_3)(CO_5)$

(285) M = Si; (288) M = Ge

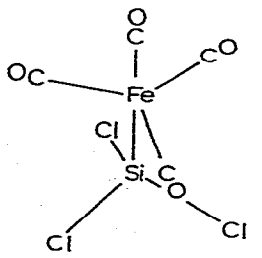


$M[Si(SiMe_3)_3](CO)_5$

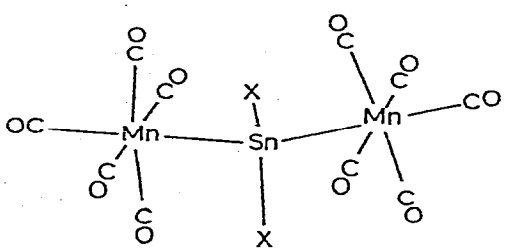
(284) M = Mn; (287) M = Re



(286) $Re(SiMe_3)(CO)_5$

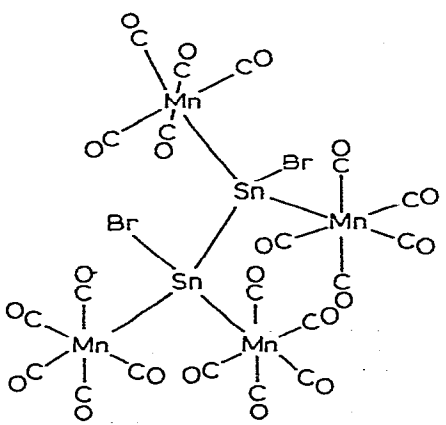


(292) $[Fe(CO)_4(SiCl_3)]^-$



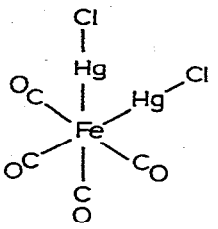
$[Mn(CO)_5]_2SnX_2$

(289) X = Cl; (290) X = Br

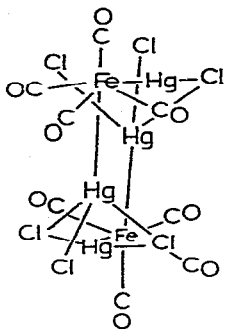


(291) $[Mn(CO)_5]_4Sn_2Br_2$

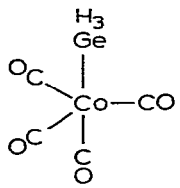
isoelectronic $Co(GeCl_3)(CO)_4$ [29]. (*293*) In one molecule (of 4), one of CO sites is occupied by larger group, possibly representing partial occupancy by HgCl, from disorder or decomposition; Hg-Fe-Hg 80.9° [27]. (*294*) Complex more correctly regarded as dimer, as



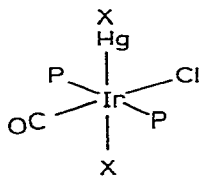
(293) *cis*-$Fe(HgCl)_2(CO)_4$



(294) $[Fe(CO)_4(HgCl)(HgCl_2)]^-$



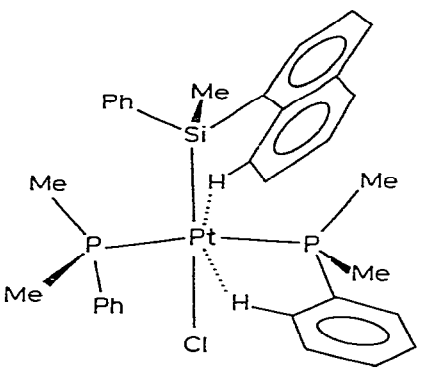
(295) $Co(GeH_3)(CO)_4$



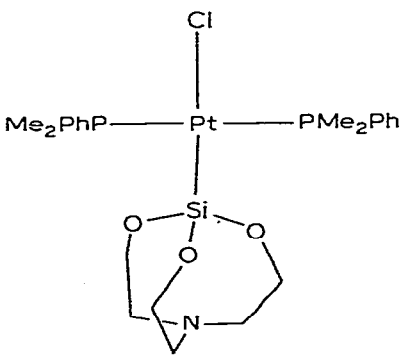
$P \equiv PPh_3$

$IrClX(HgX)(CO)(PPh_3)_2$

(296) X = Br; (297) X = Cl



(298) (+)-*trans*-$PtCl[SiMePh(C_{10}H_7)](PMe_2Ph)_2$

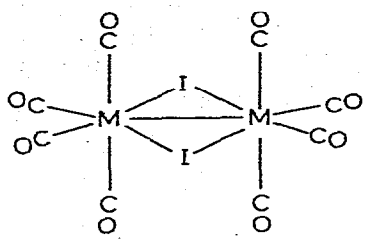


(299) *trans*-$PtCl[Si(OCH_2CH_2)_3N](PMe_2Ph)_2$

monomer interacts with its mirror-image in crystal; Hg-Fe-Hg system non-linear [80.3(1)°] [28]. (*295*) Electron diffraction study; equatorial CO bent toward GeH_3, with C-Co-Ge 83.8(3)° [16]. (*296*)(*297*) Oxidative adducts of $IrCl(CO)(PPh_3)_2$ + HgX_2; IrHgCl 172.2°; in (*297*) equatorial Cl site is 15% Br [340]. (*298*) Coordination of silatrane gives complex with no Si-N bond (2.89Å); Si becomes

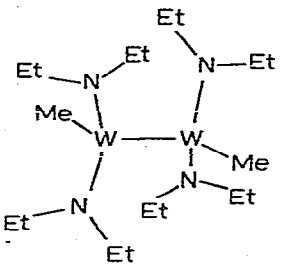
sufficiently anionic for Si–N interaction to become repulsive, with N lone pair inside cage, and trigonal planar N [255]. (*299*) Absolute configuration (*S*), corresponds to (*R*)(+)-silane from which it is formed; Si group has high *trans* influence, Pt–Cl 2.462(2)Å; two short Pt...H (2.72, 2.78Å) give distorted octahedral geometry [328].

(b) *Binuclear transition metal complexes*

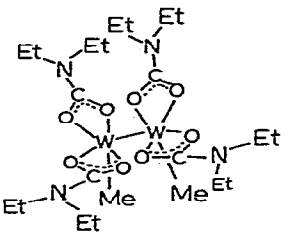


$[MI(CO)_4]_2$

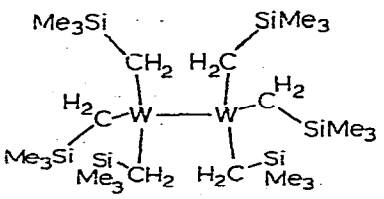
(300) M = Mo; (301) M = W



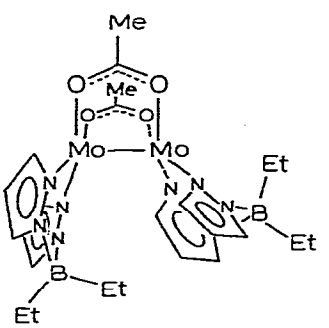
(302) $W_2Me_2(NEt_2)_4$



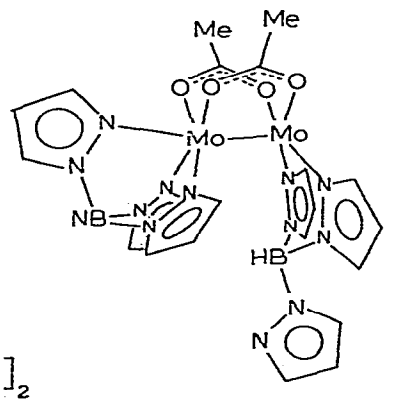
(303) $W_2Me_2(O_2CNEt_2)_4$



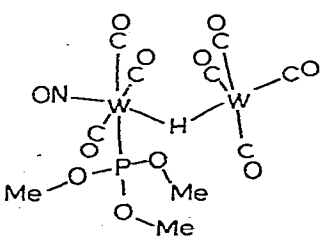
(304) $W_2(CH_2SiMe_3)_6$



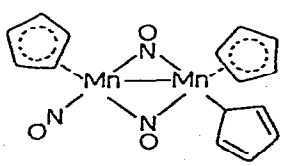
(305) $Mo_2(O_2CMe)_2[Et_2B(pz)_2]_2$



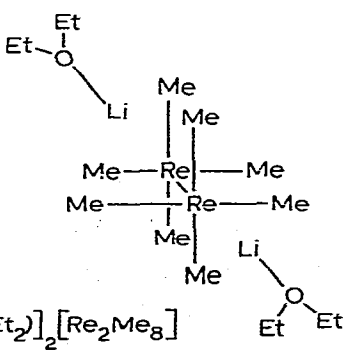
(306) $Mo_2(O_2CMe)_2[HB(pz)_3]_2$



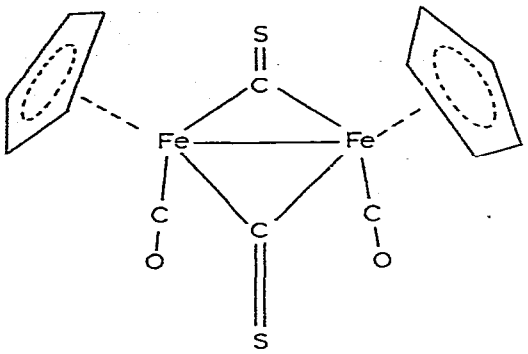
(307) $HW_2(CO)_8(NO)[P(OMe)_3]$



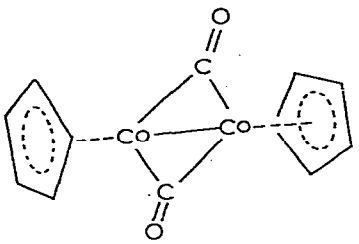
(308) $Mn_2(NO)_3(C_5H_5)_3$



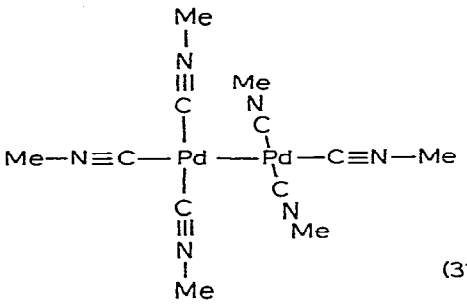
(309) $[Li(OEt_2)]_2[Re_2Me_8]$



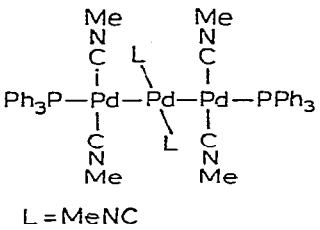
(310) $[Fe(CO)(CS)(C_5H_5)]_2$



(311) $\{[Co(CO)(C_5H_5)]_2\}^-$

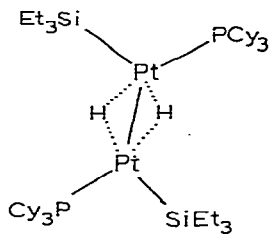


(312) $[Pd_2(CNMe)_6]^{2+}$



L = MeNC

(313) $[Pd_3(CNMe)_6(PPh_3)_2]^{2+}$

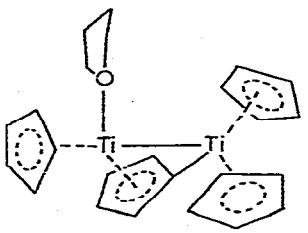


(314) $[PtH(SiEt_3)(PCy_3)]_2$

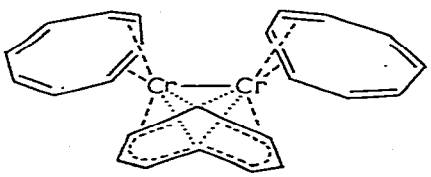
(*300*)(*301*) Structures consist of two edge-sharing octahedra [60,61]. (*302*) *Anti* rotamer in solid, 3:2 *anti-gauche* in solution [205]. (*303*) From $W_2Me_2(NEt_2)_4 + CO_2$ [258]. (*304*) Shorter W-W bond than (*302*) [277]. (*305*) Reference [251]. (*306*) Weakly bonded axial ligand on one Mo; related to formation of axial donor bonds to multiple metal-metal bonded species; cf. also (*305*) [251]. (*307*) Combined X-ray: neutron diffraction study; $P(OMe)_3$ derivative did not have disorder problems found with nonacarbonyl; asymmetric, bent W-H-W, with W-H 1.859, 1.894(6)Å, W-H-W 129.4(3)° [18]. (*308*) Reference [157]. (*309*) Eclipsed Me groups, Re-Re quadruple bond [58]. (*310*) Similar to *cis*-$[Fe(CO)_2(C_5H_5)]_2$, with bridging CS; Fe-Fe significantly shorter than carbonyl [2.482(1) *vs.* 2.531(2)Å] [131]. (*311*) Radical anion, with coplanar $Co(CO)_2Co$ perpendicular to C_5H_5; Co-Co bond order *ca.* 1.5 [91]. (*312*) Pd-C(ax) 2.049(6) > Pd-C(eq)

1.963(5)Å; *cis*-C-Pd-C 95.0(6)°; bonding and fluxional behaviour discussed [106]. (*313*) Diagram foreshortens 2 MeNC; equatorial ligands displaced to centre of molecule; short Pd-Pd bonds [378]. (*314*) From $Pt(C_2H_4)_2(PCy_3)$ + $HSiEt_3$, catalyst for addition of HMR_3(M = Si,Ge) to olefins and alkynes [380].

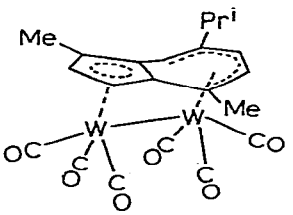
(c) *Binuclear complexes containing bridging hydrocarbon ligands*



(315) $Ti_2(C_5H_4)(C_5H_5)_3(thf)$

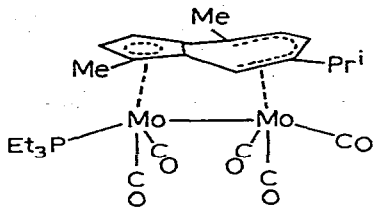


(316) $Cr_2(C_8H_8)_3$

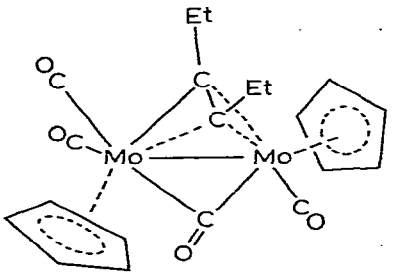


(319) $W_2(CO)_6(C_{15}H_{18})$

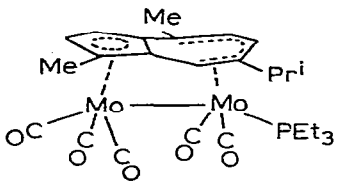
(*315*) From $TiCl_2(C_5H_5)_2$ + $KC_{10}H_8$ at −80°; both types of C_5 ring planar, no H atom (hydride) found; short Ti-C 2.19(2); open structure reflected in high reactivity [270]. (*316*) C_8 rings contain butadiene, pentadienyl fragments; both Cr bond to 2 C of bridging C_8 ligands; short quadruple Cr-Cr bond [269]. (*317*)(*318*) Two isomers differ in position of attachment of PEt_3; studied in connection with CO scrambling studies [293]. (*319*) Structure very similar to Mo analogue, with identical metal-metal distance [242].
(*320*) Semi-bridging CO, with Mo-CO 1.936, 2.826(6)Å; short Mo-Mo bond; fluxional in solution [225]. (*321*) Terminal CO attacked in reaction $MnH(CO)_5$ + CH_2N_2/−85° to give acyl function, and four-membered ring [77]. (*322*) Staggered CO groups; Mo≡C 1.835(25)Å [162]. (*323*) From $Re_2(CO)_9[CPh(OMe)]$ + $AlBr_3$, contains μ-carbyne ligand, with Re-C 2.144(41)Å; *no* Re-Re bond [152]. (*324*) From $Mn(CO)_2(C{=}CHPh)(C_5H_5)$ + $Fe_2(CO)_9$; viewed as heteroatom analogue of trimethylene-methane, although two alternatives are (b) and (c); short Fe-Mn 2.760(4)Å, bent Mn-C-C 124.8(1.6)° and one Mn-C-O 169.4(1.6)° (because of short



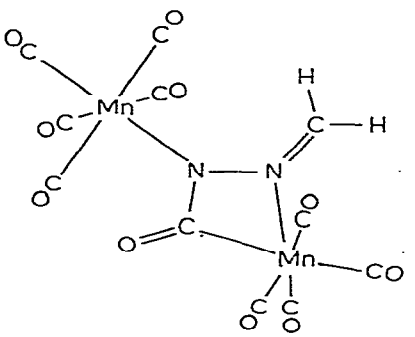
(317) $Mo_2(CO)_5(PEt_3)(C_{15}H_{18})$
isomer A



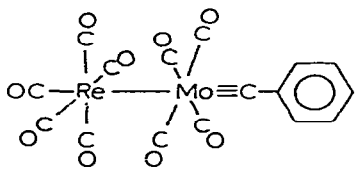
(320) $Mo_2(CO)_4(C_2Et)_2(C_5H_5)_2$



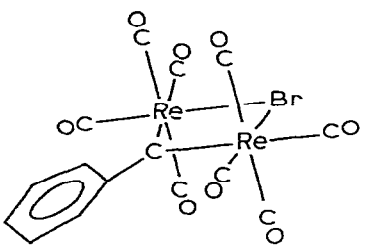
(318) $Mo_2(CO)_5(PEt_3)(C_{15}H_{18})$
isomer B



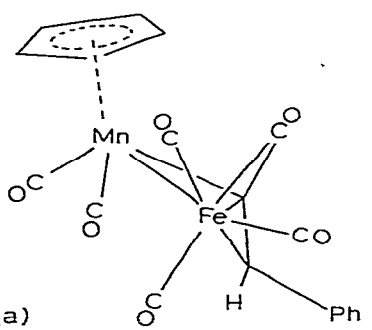
(321) $Mn_2(CO)_{10}(CH_2N_2)$



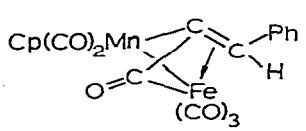
(322) $(CO)_5ReMo(CO)_4(CPh)$



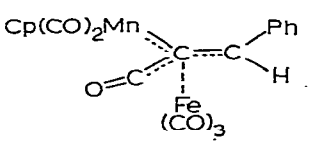
(323) $Re_2(CO)_8(CPh)Br$



(324) $[(C_5H_5)(CO)_2MnC(CHPh]Fe(CO)_3$

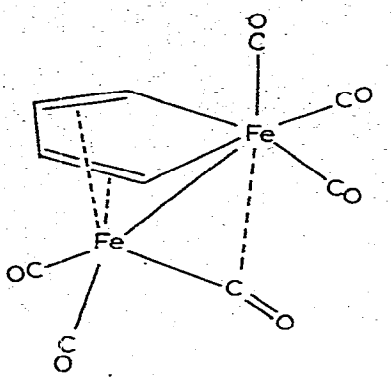


(b)

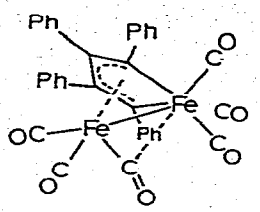


(c)

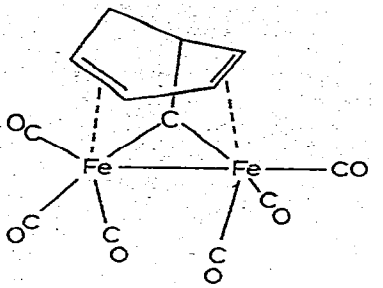
intermolecular Fe...C contact) [209]. (325) From $Fe_3(CO)_{12}$ + thiophene, almost planar FeC_4 ring, all bonds have multiple bond order; one CO



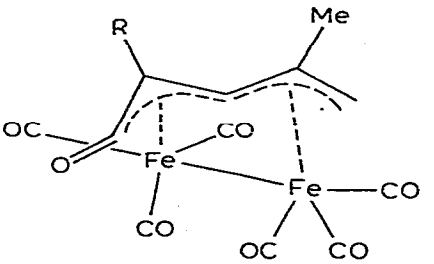
(325) $Fe_2(CO)_6(C_4H_4)$



(326) $Fe_2(CO)_6(C_4Ph_4)$

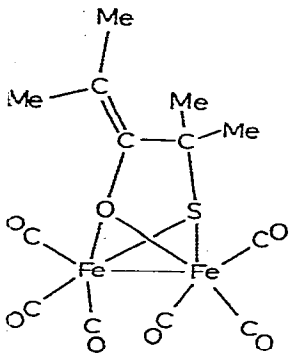


(327) $Fe_2(CO)_6(C_7H_7)$

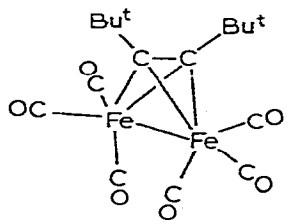


(329) $Fe_2(CO)_6(C_7H_{10}CO)$ R = Et

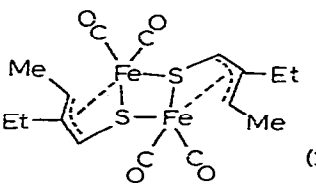
(330) $Fe_2(CO)_6(C_9H_{14}CO)$ R = Bu^t



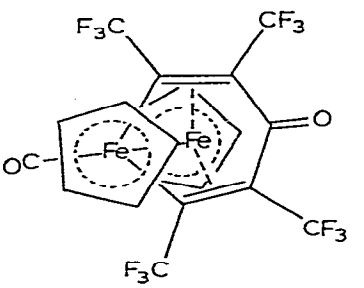
(328) $Fe_2(CO)_6[Me_2C{:}C(O)CMe_2(S)]$



(331) $Fe_2(CO)_6(C_2Bu^t_2)$

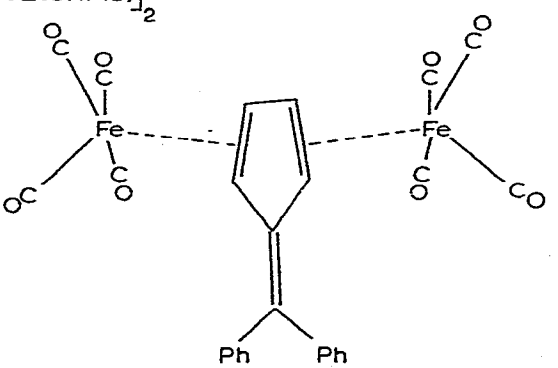


(332) $[Fe(CO)_2(SCHCEtCHMe)]_2$

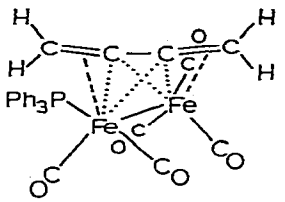


(333) $Fe_2(CO)(C_8F_{12}CO)(C_5H_5)_2$

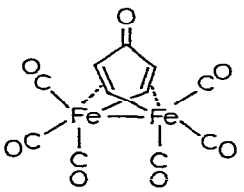
(334) $[Fe(CO)_3]_2C_{16}H_{16}$



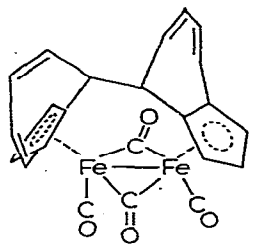
(335) $Fe_2(CO)_8(C_5H_4CPh_2)$



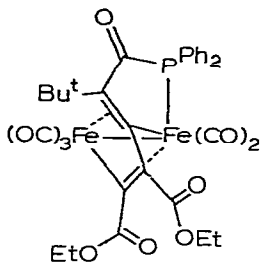
(336) $Fe_2(CO)_5(PPh_3)(C_4C_4)$



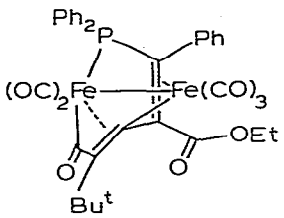
(337) $Fe_2(CO)_6(C_4Ph_4CO)$
Ph groups omitted



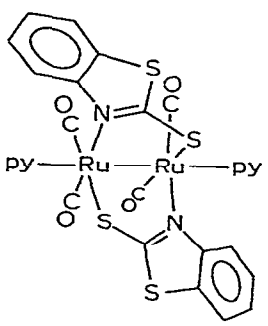
(338) $Fe_2(CO)_4(C_{10}H_{16})$



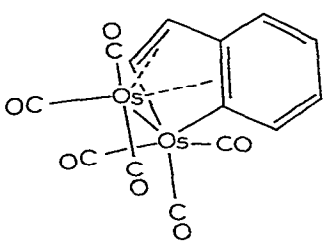
(340) $Fe_2(CO)[PPh_2C(O)CBu^tCC(CO_2Et)C(CO_2Et)]$



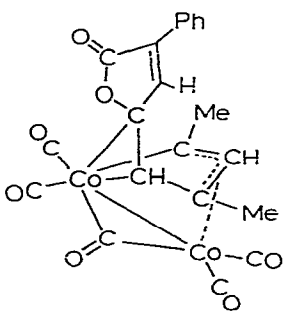
(341) $Fe_2(CO)_5[PPh_2CPhC(CO_2Et)CCBu^tCO]$



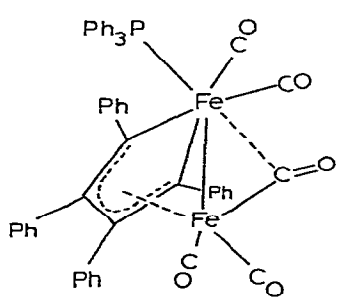
(342) $[Ru(mbt)(py)(CO)_2]_2$



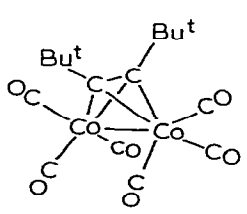
(343) $Os_2(CO)_6(C_8H_6)$



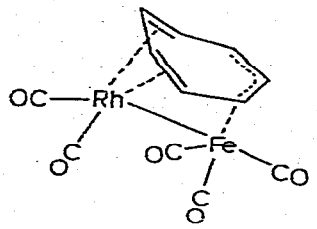
(345) $Co_2(CO)_5[CH{:}CPhC(O){-}OC{:}CHCMeCHCMe]$



(339) $Fe_2(CO)_5(PPh_3)(C_4Ph_4)$

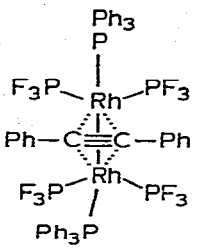


(344) $Co_2(CO)_6(C_2Bu^t_2)$

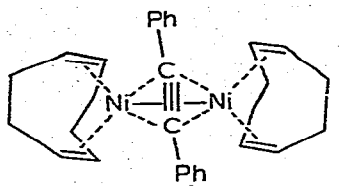


(346)

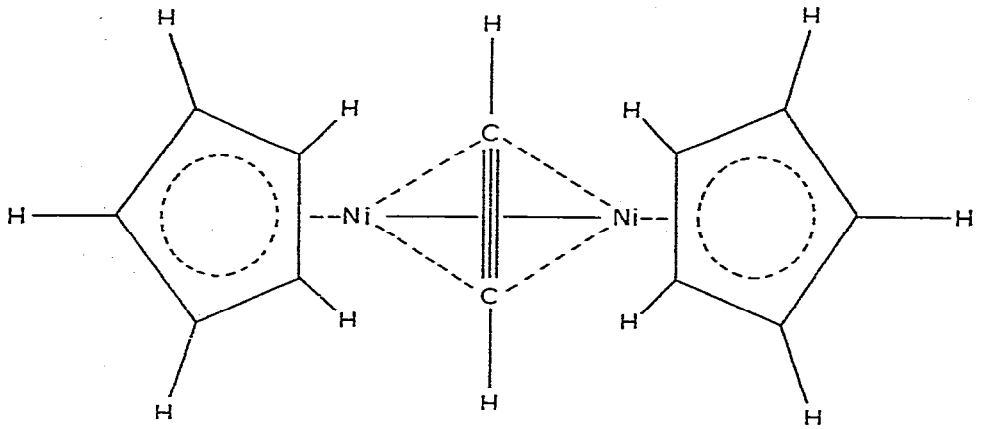
$(C_7H_7)[Fe(CO)_3][Rh(CO)_2]$



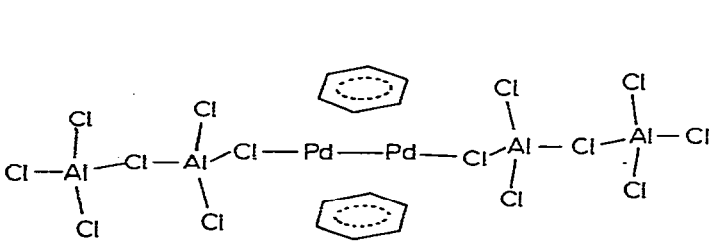
(347) $[Rh(PF_3)_2(PPh_3)]_2C_2Ph_2$

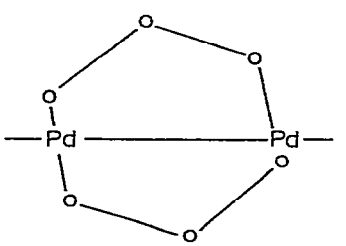


(349) $[Ni(C_8H_{12})]_2C_2Ph_2$



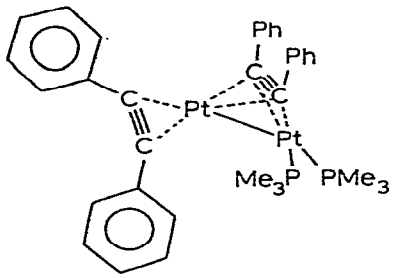
(348) $[Ni(C_5H_5)]_2C_2H_2$





(350) $[PdAl_2Cl_7(C_6H_6)]_2$

is semi-bridging [65]. (*326*) Butadiene fragment delocalised; Fe-Fe bond bridged by semi-bridging CO; see also (*339*) [329]. (*327*) Bridging carbene complex, with unequal Fe-C(carbene) 1.977, 2.064(3)Å; this results from carbene occupying apical position on one, equatorial on other Fe; also gives twisted $Fe(CO)_3$ groups; 1,3-diene is bonded to 2 Fe atoms [117]. (*328*) Stable iron enolate from $Fe_2(CO)_9$ +



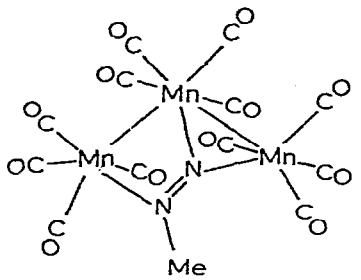
(351) $Pt_2(C_2Ph_2)_2(PMe_3)_2$

tetramethylthietanone [120]. (*329*)(*330*) From $Fe_2(CO)_9$ + CH_2:CMeC≡CR (R = Et or Bu^t, respectively); alkenyne links with CO to give 5 carbon, 6e donor to $Fe_2(CO)_6$ unit, with CCRCO group intermediate between allyl and C=C-C=O; the common C atom belongs to 2 nearly perpendicular 3 carbon, 3e donors, and bridges the 2 Fe atoms [132].
(*331*)(*344*) Differ in orientation of $M(CO)_3$ groups; shorter Fe-Fe gives steric interaction of CO groups, which rotate by 60°; (*331*) is Hubel's $Fe_2(CO)_7(C_2Bu^t_2)$ [169]. (*332*) From $Fe(CO)_5$ + a thiacyclobutene; contains nearly square Fe_2S_2 group; no structural details [170].
(*333*) Ferracyclohexadienone from $[Fe(CO)_2(C_5H_5)]_2$ + $C_2(CF_3)_2$ [92].
(*334*) Product from olefin metathesis reaction using $Fe(CO)_3(C_8H_8)$ and WCl_6-EtOH-$EtAlCl_2$; hydrocarbon has 5 fused rings [246].
(*335*) Transoid $Fe(CO)_4$ groups, with C=C occupying equatorial site in TBP; mean Fe-CO(ax) 1.816, Fe-CO(eq) 1.783Å; *no* metal-metal bond [286].
(*336*) Butatriene bridging Fe_2, with C-C distances of 1.358, 1.367, 1.370(18)Å, considered to bond as η^3-allyl to each Fe [300].
(*337*) Confirms structure of Hubel's $Fe_2(CO)_7(C_2Ph_2)_2$, here obtained from $Fe_2(CO)_9$ + C_2Ph_2 in thf; Ph groups on each C=C omitted in diagram [334]. (*338*) Derived from azulene + $Fe_3(CO)_{12}$ via dimerisation of hydrocarbon; comparisons with $Fe_4(CO)_{10}(C_{20}H_{16})$ studied earlier, particularly in relation to bending of C_7 rings [364]. (*339*) Shows PPh_3 bonded to ferrole-Fe; butadiene fragment delocalised [see (*326*)]; studied in connection with ^{13}C NMR studies of solution dynamics [388].
(*340*)(*341*) Obtained from $Fe_2(CO)_6(PPh_2)(C{\equiv}CBu^t)$ + alkyne; new ligand

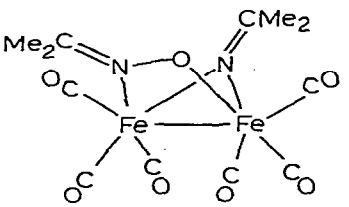
in (*340*) formed from $C{\equiv}CBu^t$, PPh_2, CO and incoming alkyne to give α-ketophosphine, which cleanly rearranges to analogue of (*341*) on heating; (*341*) formed from PhC_2CO_2Et, showing that α-C of acetylide bonds to C of incoming alkyne which bears most electronegative group [320]. (*342*) From $Ru_3(CO)_{12}$ + mercaptobenzothiazole, then py; contains distorted $SCNRu_2$ ring [288]. (*343*) Osmaindenyl system from $OsMe_2(CO)_4$ + C_8H_8; bond distances suggest delocalisation in 5-membered ring, localised bonds in 6-membered ring, but distortions also present [128]. (*344*) See (*331*). (*345*) From 2 alkynes (as a σ,π-butenyl unit) inserting into butenolide-$Co_2(CO)_6$ complex [240]. (*346*) From $LiFe(CO)_3(C_7H_7)$ + $[Rh(CO)_2Cl]_2$; bridging C_7 ligand (fluxional) gives long Fe-Rh bond [89]. (*347*) Similar to $Co_2(CO)_6(C_2R_2)$, with PPh_3 on same side as alkyne; C-C 1.369(7)Å; paper contains discussion of fluxional behaviour [385]. (*348*) Short Ni-Ni, with *cis* bent alkyne; electron density maps support *straight* Ni-Ni bond, with double maximum along axis, and predominance of bonding between Ni and slightly antibonding C_2H_2 b_1 orbital (i.e. π*); acetylene affected, with mid-point of C-C not coincident with geometric mid-point, and apparently bent (as with strained 3-membered rings) away from Ni, allowing a_1, b_1 orbitals to point to Ni-Ni region [21]. (*349*) From $Ni(cod)_2$ + C_2Ph_2, long Ni-Ni bond [2.617(2)Å], as expected from electron deficiency [cf. $[Ni(C_5H_5)_2]_2C_2Ph_2$, 2.329(4)Å]; alkyne has C-C 1.386(11)Å, C-C-Ph 140.6(4)° [315]. (*350*) Low temperature study freezes C_6H_6 in one of 2 disordered conformations found in room temperature study; diagram also shows perpendicular view of C_6H_6 relative to Pd-Pd bond [96]. (*351*) From (*114*) and PMe_3; C_2Ph_2 shows different coordination modes, with terminal alkyne having C-C 1.26(5)Å, Ph-C≡C 153°; bridge alkyne has C-C 1.36(5)Å [306].

(d) *Binuclear complexes containing other bridging ligands*

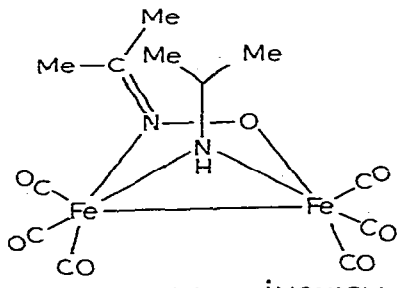
(*352*) From $MnH(CO)_5$ + CH_2N_2; bent Mn-Mn-Mn sequence with asymmetric bridging N, Mn-N 1.964, 1.879(8)Å [114]. (*353*) Unsymmetrically



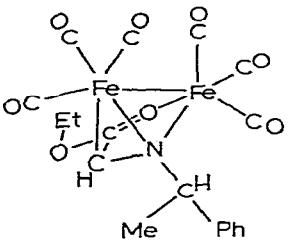
(352) $[Mn(CO)_4]_3N_2Me$



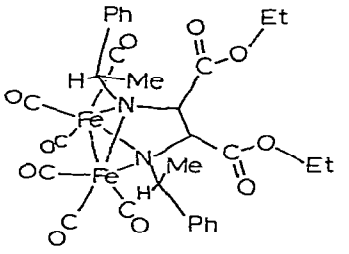
(353) $Fe_2(CO)_6(Me_2C{:}N)(Me_2C{:}NO)$



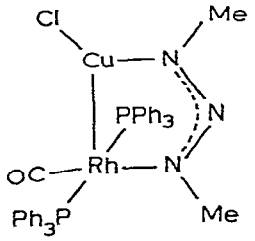
(354) $Fe_2(CO)_6(NHPr^i)(ON{:}CMe_2)$



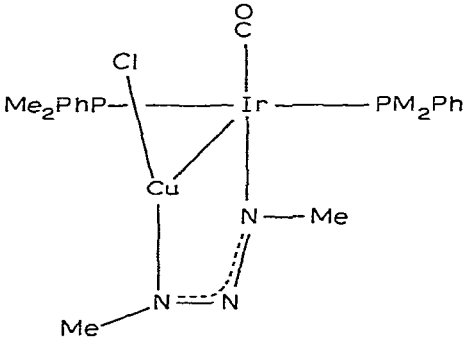
(355) $Fe_2(CO)_6$-
$(PhCHMeNCHCO_2Et)$



(356) $Fe_2(CO)_6(PhCHMeNCHCO_2Et)_2$

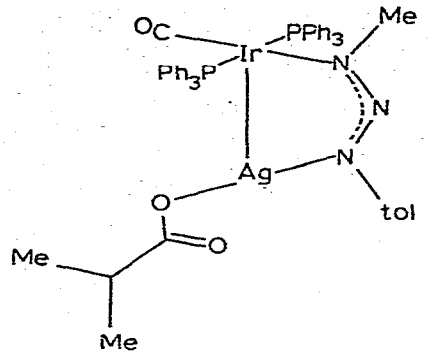


(357) $RhCuCl(N_3Me_2)(CO)(PPh_3)_2$



(358) $IrCu(CO)Cl(N_3Me_2)(PMe_2Ph)_2$

bridged $Fe_2(CO)_6$ complex from 2-bromo-2-nitrosopropane; new bridging ligand not found in nitrosoarene complexes [99]. (*354*) From $Fe_3(CO)_{12}$ + 2-nitropropane; larger Fe-N-Fe (77.6°) [101]. (*355*)(*356*) Dimerisation of Et N-α-methylbenzyliminoacetate with $Fe_2(CO)_9$ gives intermediate (*355*) and final product (*356*); latter is diaminosuccinic ester derivative, with N=CH forming unsymmetrical



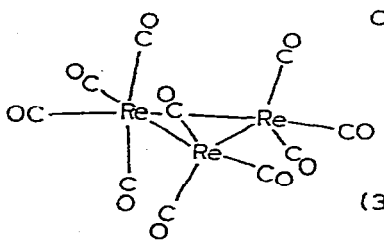
(359) $IrAg(CO)(O_2CCHMe_2)(MeN_3\ tol\text{-}p)(PPh_3)_2$

σ,π bridging group [197]. (*357*)(*358*) Contain metal-to-Cu donor bonds; tetragonal pyramidal Rh or Ir, with bridging triazenide; $MCuN_3$ approximately coplanar [348,349,216]. (*359*) Ir→Ag donor bond as in (*357*) or (*358*); isostructural with these, with bridging triazenide, monodentate carboxylate [383].

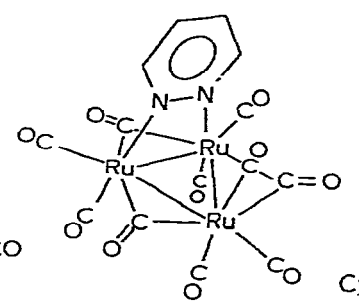
POLYHEDRAL CLUSTER COMPLEXES

(a) *Polyhedral metal carbonyl clusters*

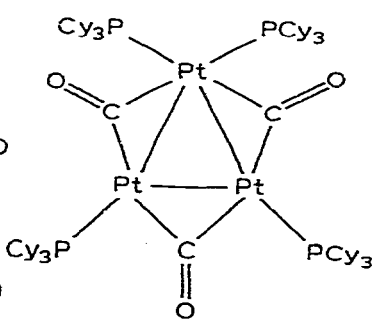
Three-atom clusters:



(361) $[H_3Re_3(CO)_{10}]^{2-}$



(362) $Ru_3(CO)_{10}(C_4H_4N_2)$



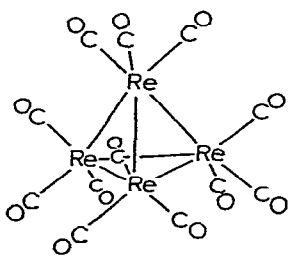
(363) $Pt_3(CO)_3(PCy_3)_4$

(*360*) Entry deleted. (*361*) Re-Re bonds are non-bridged, H-bridged, and 2H-bridged, tautomeric; 46 e cluster, disordered [see also (*399*)] [62]. (*362*) From pyridazine + $Ru_3(CO)_{12}$; Ru_3 triangle bridged by CO on each edge; asymmetric bridges probably due to packing forces;

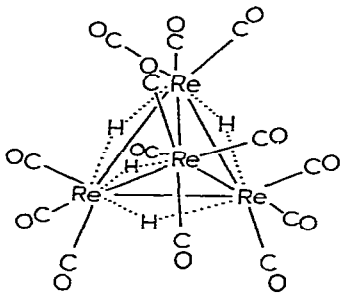
related to possible intermediate in CO scrambling process [127].

(*363*) Pt–P lengthened, attributed to steric interaction of PCy_3 groups [400].

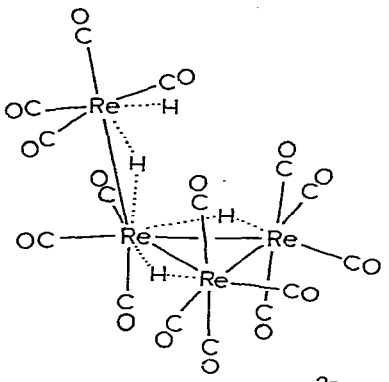
Four-atom clusters:



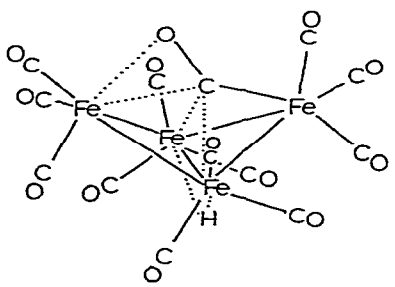
(364) $H_4Re_4(CO)_{12}$
H atoms face-bonded



(365) $[H_4Re_4(CO)_{13}]^{2-}$

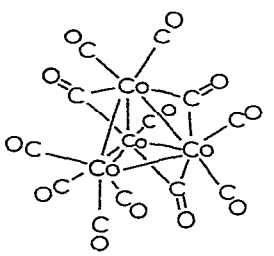


(366) $[Re_4H_4(CO)_{15}]^{2-}$

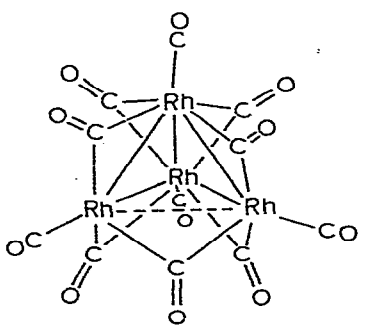


(367) $[HFe_4(CO)_{13}]^-$

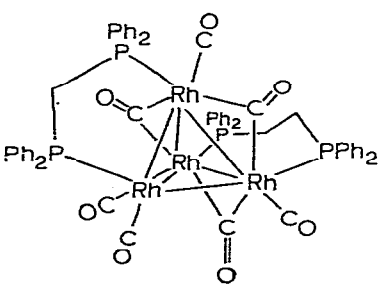
(368) No diagram available



(369) $Co_4(CO)_{12}$



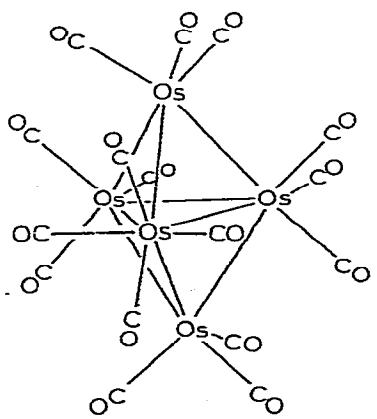
(370) $[Rh_4(CO)_{11}]^{2-}$



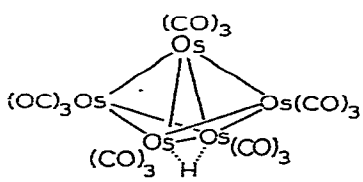
(371) $Rh_4(CO)_8(dppm)_2$

(*364*) CO groups eclipsed with Re-Re edges [contrasts with $Ir_4(CO)_{12}$ and $[Re_4H_6(CO)_{12}]^{2-}$], suggests face-bonded H, confirmed by difference Fourier; multiple Re-Re bonds [87]. (*365*) All CO groups terminal; H atom positions derived from steric arguments, and from computed maps of non-bonding interactions [115]. (*366*) 3 forms studied, differ in packing and conformations of cation; open-cluster anion is flexible, and detailed geometries differ; 64 valence e require breaking of 2 tetrahedral edges in most electron-rich M_4 cluster [150]. (*367*) Fe_4 forms open tetrahedron ('butterfly'); 12 terminal CO, 13th CO acts as 4 e ligand, interacts with all 4 Fe; H assumed to bridge Fe-Fe hinge [113]. (*368*) Preliminary report shows H bridge four edges, shown by longer Ru-Ru bonds (2.93 *vs*. 2.76Å) [87]. (*369*) Further refinement of Wei's work, attempted, but close-lying pairs of CO groups not able to be refined as separate half-CO groups [111]. (*370*) Tetrahedron distorts to give one short, one long Rh-Rh bond, latter by electrostatic repulsion between 2 Rh carrying negative charge; this also results in asymmetry of bridge CO on these atoms, and in shorter Rh-CO (terminal) (by 0.06Å) [84]. (*371*) P ligand spans 2 Rh, 3 edge-bridging CO [396].

Five-atom clusters:



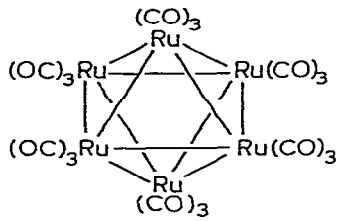
(372) $Os_5(CO)_{16}$



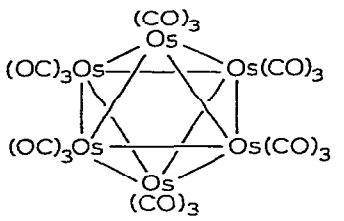
(373) $[HOs_5(CO)_{15}]^-$

(*372*) TBP (D_{3h}) Os_5 slightly distorted toward capped butterfly (C_{2v}); bonds to $Os(CO)_4$ longer than others; arrangement of CO is preferred interstitial packing of Os_5 within 16 CO [180]. (*373*) Similar to 16 CO polyhedron found for $Os_5(CO)_{16}$, with one CO missing, assumed to be site of H; Os_5 forms TBP [149].

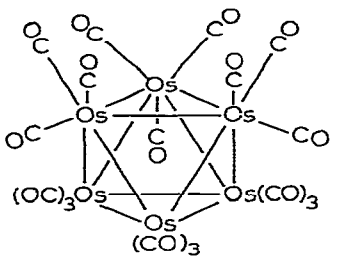
Six-atom clusters:



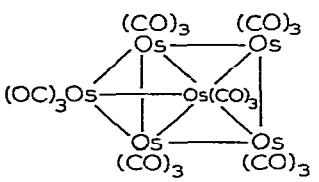
(374) $[HRu_6(CO)_{18}]^-$



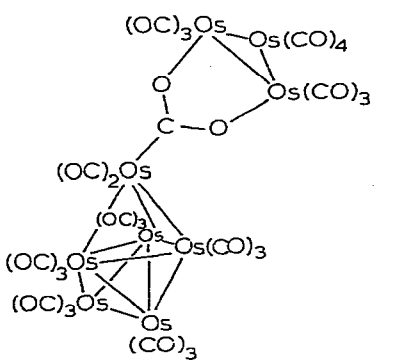
(375) $[Os_6(CO)_{18}]^{2-}$



(376) $[HOs_6(CO)_{18}]^-$



(377) $H_2Os_6(CO)_{18}$

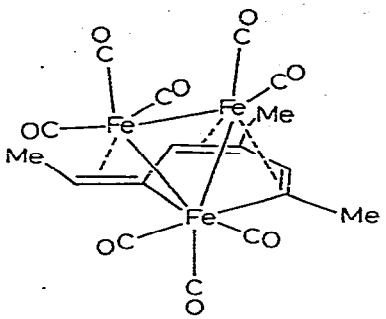


(378) $[HOs_3(CO)_{10}(O_2C)Os_6(CO)_{17}]^-$

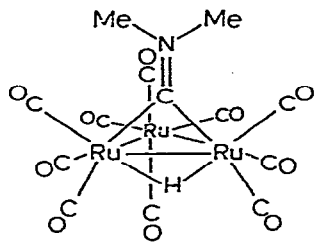
(*374*) H at centre of octahedral Ru_6 (τ-6.4), in 2 crystal modifications; all CO terminal [192]. (*375*) 2 equivalent parallel faces exactly equilateral (2.876Å), but cluster twisted from octahedral symmetry so that remaining bonds alternate long (2.886) and short (2.814Å) [191]. (*376*) One triangular face has significantly longer edges [2.973Å(av.)], CO ligands pushed back from this face, suggesting location for face-bonding H [191]. (*377*) Monocapped SP, with 1 H associated with edge, 1 H face-bonded, judged from Os-Os distances [191]. (*378*) From $Os_6(CO)_{18}$ + $[HOs_3(CO)_{11}]^-$; delocalisation in O_2COs_2 group, with C-Os bonded as carbene (Os-C 1.96Å); organic

ligand *formally* CO_2 [302].

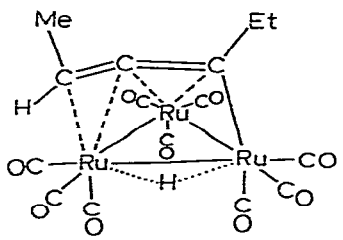
(b) *Polyhedral clusters containing* η-*hydrocarbon groups*



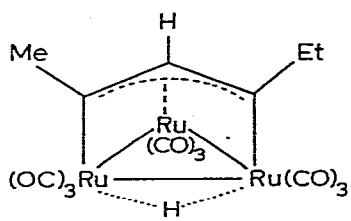
(379) $Fe_3(CO)_8(C_9H_{12})$



(380) $Ru_3H(CO)_9(C{:}NMe_2)$

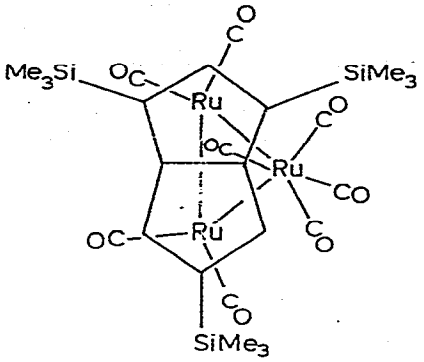


(381) $Ru_3H(CO)_9(C_6H_9)$ isomer A

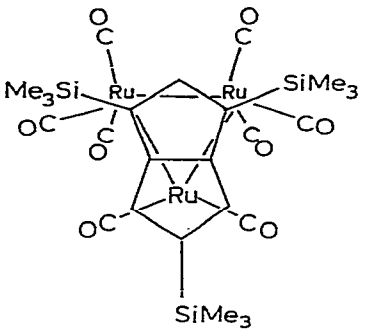


(381) isomer B

(*379*) From $Fe_3(CO)_{12}$ + HC_2Me, contains nearly planar 6-membered heterocycle; one CO bridges 2 Fe in very unsymmetrical fashion [182]. (*380*) H and C bridged Ru-Ru shorter than other two; dipolar $C{=}NMe_2$ ligand acts as 3 e donor; extended discussion of bridging H and M-M distances [13]. (*381*) Isomer A contains bent allenyl ligand which is η^2 to 2 Ru, η^1 to the third; thermal rearrangement via H shift to isomer B, containing σ,π-allyl group [155]. (*382*)(*383*) General isomerism for $Ru_3(CO)_8$ complexes of pentalenes involving edge and face bridging by hydrocarbon; equilibrium in solution, edge-bridged is fluxional; conversion (*382*)→(*383*) requires CO shift as well [284]. (*384*)(*385*) Two isomers from $Os_3H_2(CO)_{10}$ + butadiene; in (*384*), Os-Os bond *trans* to equatorial C=C somewhat longer than others; for (*385*),

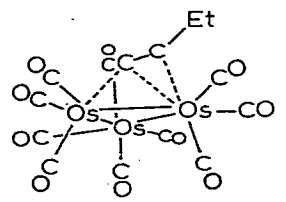


edge-bonded
(382)

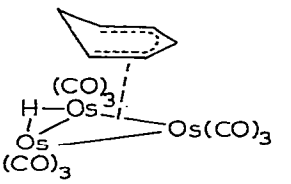


face-bonded
(383)

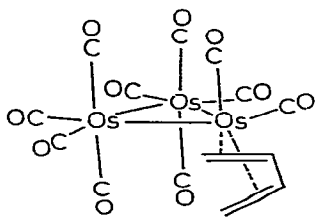
$Ru_3(CO)_3[C_8H_3(SiMe_3)_3]$



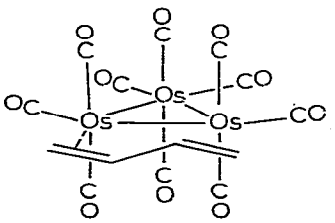
(386) $Os_3H(CO)_{10}(C_4H_7)$



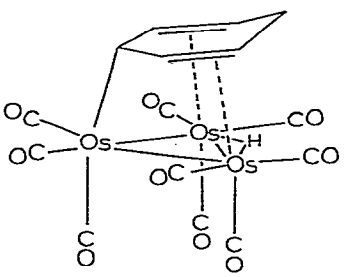
(387) $Os_3H(CO)_9(C_6H_7)$



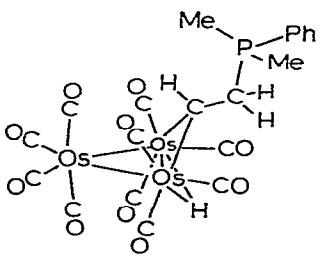
(384) $Os_3(CO)_{10}$(*s-cis*-C_4H_6)



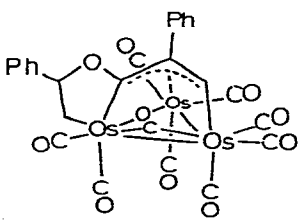
(385) $Os_3(CO)_{10}$(*s-trans*-C_4H_6)



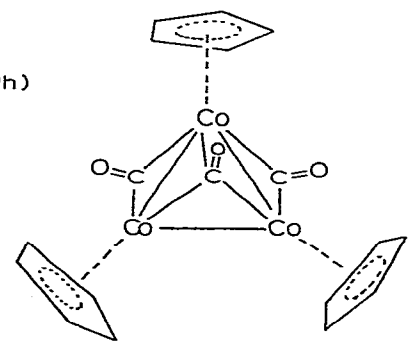
alternative formulation of (387)



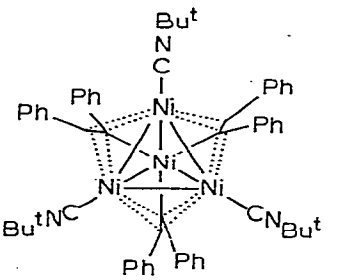
(388) $HOs_3(CO)_{10}(\bar{C}HCH_2\overset{+}{P}Me_2Ph)$



(389) $Os_3(CO)_9(HC_2PhCOCPhCH)$



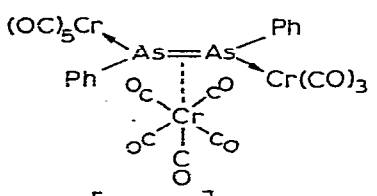
(390) $[Co(CO)(C_5H_5)]_3$



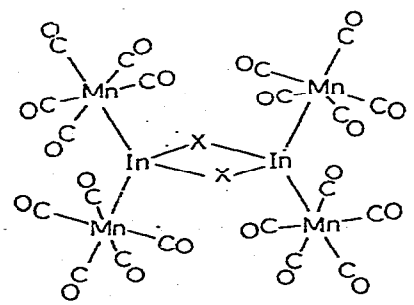
(391) $Ni_4(CNBu^t)_4(C_2Ph_2)_3$
rear $CNBu^t$ ligand omitted

Os–Os bridged by diene is substantially longer [129]. (*386*) From $Os_3H_2(CO)_{10}$ + HC_2Et; Et group disordered, acetylenic C bridges 2 Os, also bridged by H (not located) [130]. (*387*) Planar dienyl complex from $Os_3H_2(CO)_{10}$ + 1,3-C_6H_8; dimensions also consistent with Os–C σ bond, with 2 C=C coordinated to other Os (see two forms illustrated) [154]. (*388*) Formed by attack of PMe_2Ph at bridging vinyl to give dipolar ligand symmetrically bridging Os–Os; Os–H 1.88Å(av.), Os–H–Os 97(3)°, Os–H–Os plane inclined 110.25° to Os_3 plane [221]. (*389*) From HC_2Ph + $Os_3(CO)_{12}$; 2 alkynes linked through CO to give bicyclic 5:5 rings, with σ,π-allyl system previously found in Ru_3 systems [285]. (*390*) Isosceles Co_3 cluster, with 2 semi-bridging (edge) CO, one face-bonded CO; in solution is exceptionally deformable (fictile), with IR dependent on solvent [196]. (*391*) Slight trigonal compression of Ni_4 tetrahedron (*cf.* 0.34Å); each C_2Ph_2 coordinated by formal Ni–C σ bond to apical Ni, and to 2 basal Ni by 3-centre μ bonds; hydrogenation catalyst for alkynes to olefins [398].

(c) *Polyhedral clusters containing Main Group elements*

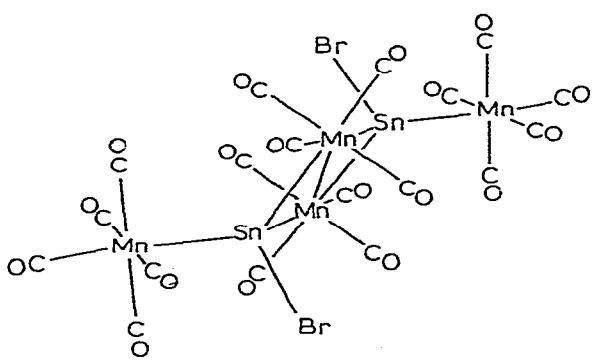


(392) $[Cr(CO)_5]_3As_2Ph_2$

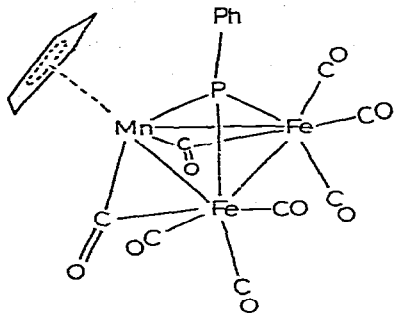


$\left\{[Mn(CO)_5]_2InX\right\}_2$

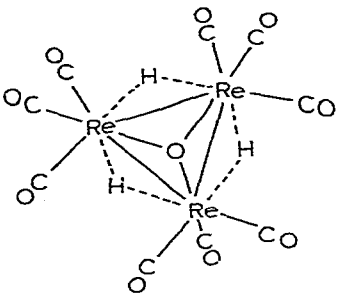
(393) X = Cl; (394) X = Br; (395) X = I



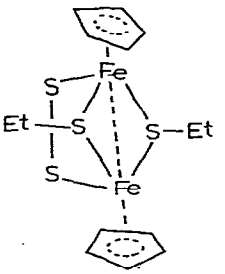
(396) $Mn_2(CO)_8[SnBrMn(CO)_5]_2$



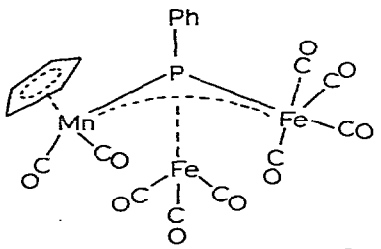
(397) $(C_5H_5)MnFe_2(CO)_8(PPh)$



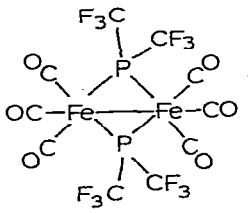
(399) $[H_3Re_3O(CO)_9]^{2-}$



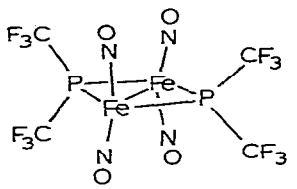
(400) $[Fe_2(S_2)(SEt)_2(C_5H_5)_2]^+$



(398) $[(C_5H_5)(CO)_2MnP(Ph)Fe(CO)_4]Fe(CO)_3$

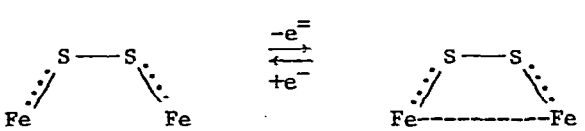


(401) $Fe_2(CO)_6[P(CF_3)_2]_2$

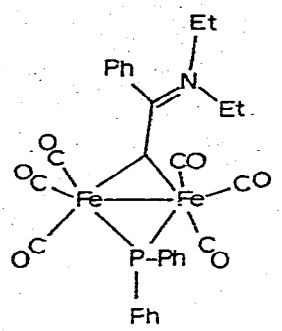


(402) $Fe_2(NO)_4[P(CF_3)_2]_2$

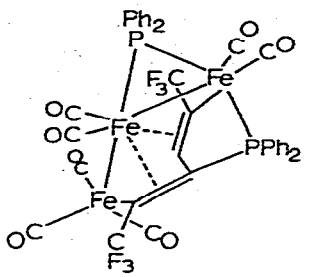
(*392*) From $Na_2Cr_2(CO)_{10}$ + $PhAsCl_2$, or $Cr(CO)_5AsPhLi_2$ + $Cr(CO)_5AsPhCl_2$; contains stabilised arsenobenzene (PhAs=AsPh) ligand, with As–As 2.371Å [299]. (*393*)(*394*)(*395*) From $Mn_2(CO)_{10}$ + InX; In has distorted tetrahedral geometry, Mn–In–Mn angle (*ca.* 125°) not affected by X; planar In_2X_2 ring, perpendicular to Mn_4 plane [237]. (*396*) From $Mn_2(CO)_{10}$ + $SnCl_2$ in xylene at 150° [206]. (*397*) From $Mn(CO)_2(PPhCl_2)(C_5H_5)$ + $Fe_2(CO)_9$; one CO asymmetrically bridging Mn–Fe, with Mn–C < Fe–C [208]. (*398*) Another product from above reaction; contains 3-centre 4e Mn–P–Fe system bonded to $Fe(CO)_3$; trigonal planar P in stabilised phosphinidene complex [220]. (*399*) Symmetrical H bridges, contrasts with (*361*) [62]. (*400*) From 1e oxidation of neutral complex; change in bond lengths for neutral dimer and cation respectively: Fe–Fe 3.307, 3.059; S–S 2.023, 1.987Å; qualitative MO scheme suggests:



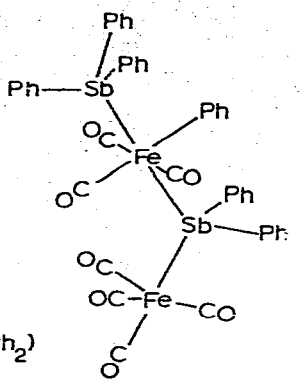
[144].



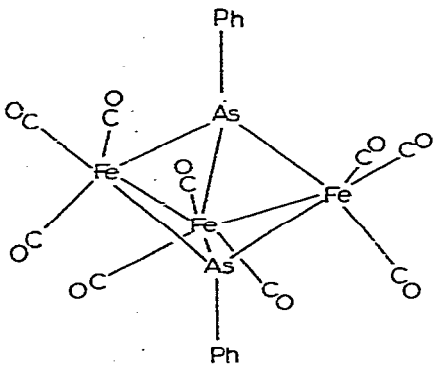
(403) $Fe_2(CO)_6[CHCPh{:}NEt_2](PPh_2)$



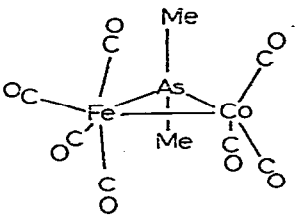
(404) $Fe_3(CO)_7[Ph_2PC_4(CF_3)_2](PPh_2)$



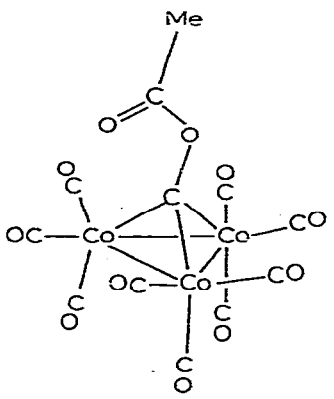
(406) $Fe_2(SbPh_2)Ph(CO)_7(SbPh_3)$



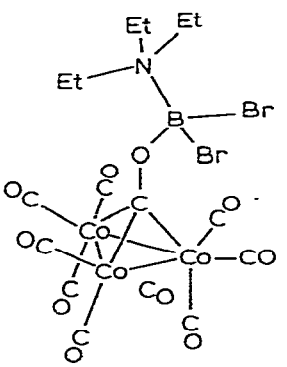
(405) $Fe_3(AsPh)_2(CO)_9$



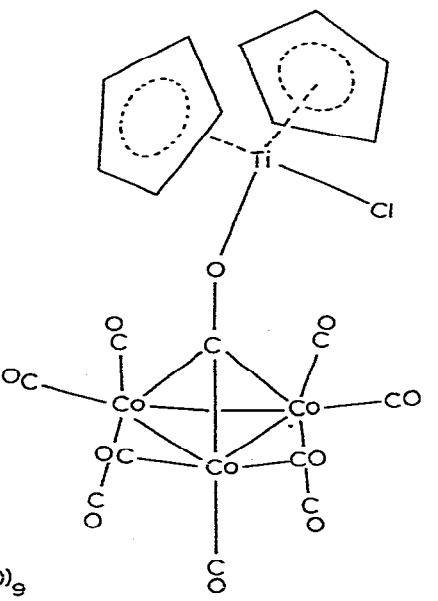
(407) $FeCo(AsMe_2)(CO)_7$



(408) $Co_3(COCOMe)(CO)_9$

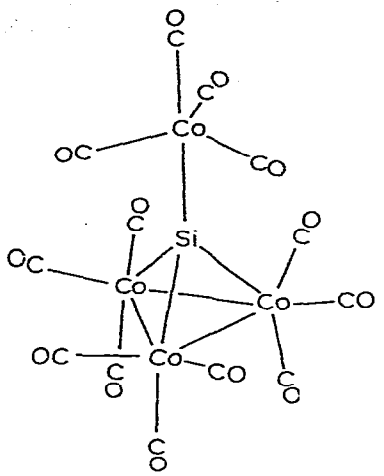


(409) $Co_3(COBBr_2NEt_3)(CO)_9$

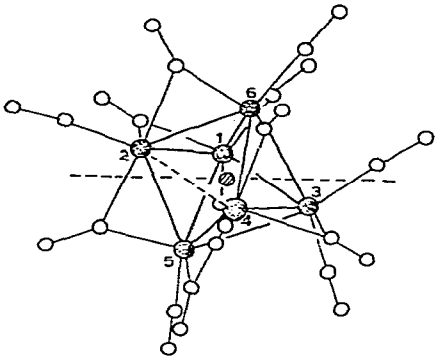


(410) $Co_3[COTiCl(C_5H_5)_2](CO)_9$

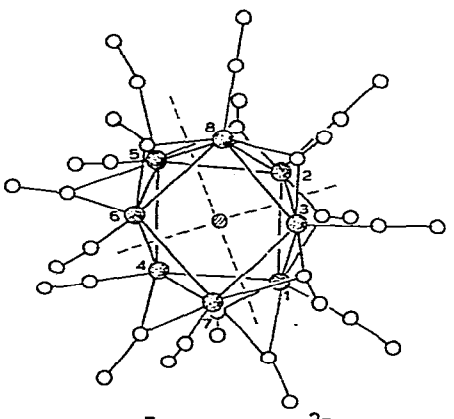
(*401*) Long Fe-Fe 2.821Å, folded Fe_2P_2 unit 118.9°, related to strong electron withdrawing power of CF_3; comparison with (*402*) [76].



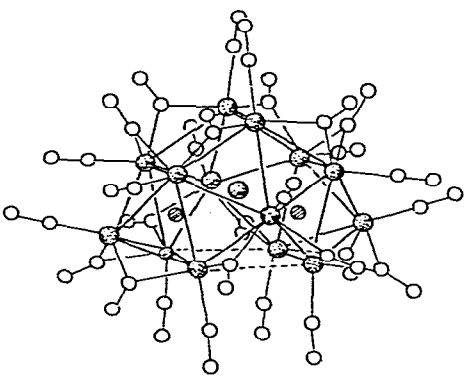
(411) $Co_3Si[Co(CO)_4](CO)_9$



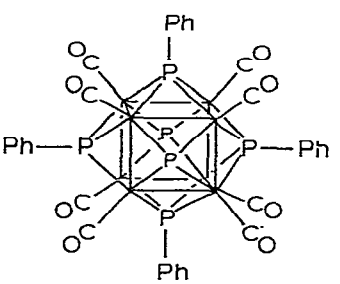
(412) $[Co_6C(CO)_{14}]^{2-}$



(413) $[Co_8C(CO)_{18}]^{2-}$



(414) $[Rh_{15}C_2(CO)_{28}]^{-}$



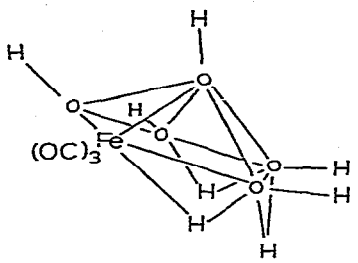
(415) $Ni_8(CO)_8(PPh)_6$
2 Ph groups omitted

(*402*) Fe_2P_2 ring planar, with more acute Fe-P-Fe angle (77°) than for (*401*) (80°); electronic effects not yet resolved [31]. (*403*) From

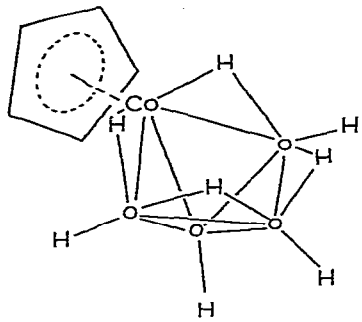
$Fe_2(CO)_6(PPh_2)(C{\equiv}CPh)$ + NEt_3; contains 1,3-dipolar ligand as bridging one centre, 3e ligand [313]. (*404*) From octacarbonyl complex, contains ferracyclobutene (FeC_4) ring, and dimerised phosphinoalkyne as *trans*-butadiene σ- and π-bonded to 3 Fe atoms [339]. (*405*) From $Fe_2(CO)_9$ + $Mn(CO)_2(AsPhCl_2)(C_5H_5)$ + other Fe-Mn-As clusters [239]. (*406*) From $Fe_3(CO)_{12}$ + $SbPh_3$; contains Sb-Fe-Sb-Fe chain, with Ph migration from Sb to Fe; one Fe distorted octahedral, the other TBP [361]. (*407*) Bond distances in $FeMn(AsMe_2)(CO)_8$, $[FeCo(CO)_8]^-$ and this complex determined by arrangement of CO groups rather than other factors [63]. (*408*) From $LiCo_3(CO)_{10}$ + MeCOBr [85]. (*409*) Similar to chloro complex described earlier [166]. (*410*) From $Co_2(CO)_8$ + $TiCl_2(C_5H_5)_2$; Ti geometry almost the same as in $TiCl_2(C_5H_5)_2$ [219]. (*411*) From $NaCo(CO)_4$ + SiI_4; first Co_3Si cluster structurally characterised [126]. (*412*) Paramagnetic, distorted octahedron, with 12 Co–Co in 3 sets: one 2.96(1) (stretched), 6 2.53 (CO-bridged), 5 2.75(av.); Co–C 1.84–1.94Å [161]. (*413*) Tetragonal antiprism elongated along one of 2-fold symmetry axes; 9 terminal CO, 9 edge-bridging CO, some of latter markedly asymmetric; different skeleton from isoelectronic $Rh_8C(CO)_{19}$ [161]. (*414*) Metal atom framework is centred tetracapped pentagonal prism with bases and two side faces capped; 2 Rh...Rh too long to be bonding (3.33Å); central Rh coordinates to 12 external Rh, and to 2 C, i.e. true metal bonding; structure rationalised by condensation and isomerisation of parent $[Rh_6C(CO)_{15}]^{2-}$ cluster; 14 terminal CO, 14 bridge CO (2 bridge connections per Rh) [317]. (*415*) Completely bonding Ni_8 cube, with face-bridging P; first electron-precise metal cube, analogue of cubane; P_6 forms non-bonding octahedron, interpenetrated by Ni_8 [363].

(d) *Polyhedral metalloborane complexes*

(*416*) Shorter Fe-B than in $[Fe(CO)_4B_7H_{12}]^-$; H bridges Fe-B with apparent Fe-H 1.52Å [24]. (*417*) Borane formally η^3; analogue of B_5H_9 with basal BH replaced by $Co(C_5H_5)$ [35].



(416) $[Fe(CO)_3B_5H_8]^-$

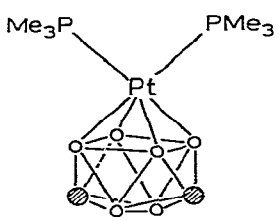


(417) 2-$[(C_5H_5)Co]B_4H_8$

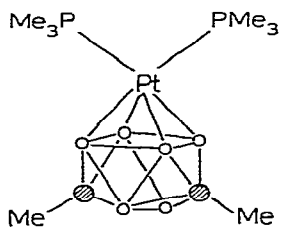
(e) *Polyhedral metallocarborane complexes*

Nine-vertex polyhedra:

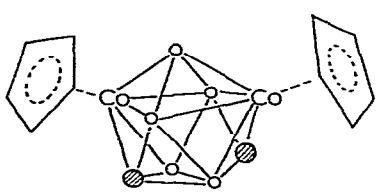
(*418*)(*419*) From Pt(stilbene)$(PMe_3)_2$ + *closo*-1,6-$R_2C_2B_6H_6$ (R = H or Me), structure of β-form of (*419*); latter has shorter Pt-B, longer Pt-P



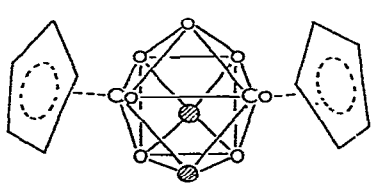
(418) 1,1-$(Me_3P)_2$-1,6,8-$PtC_2B_6H_8$



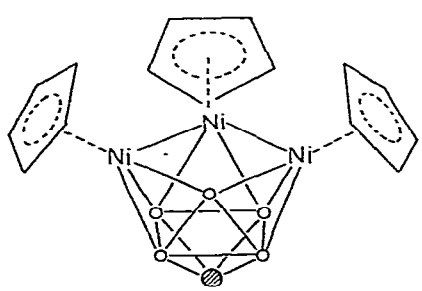
(419) 1,1-$(Me_3P)_2$-6,8-Me_2-1,1,6,8-$PtC_2B_6H_6$



(420) 1,8-$[(C_5H_5)Co]_2$-5,6-$C_2B_5H_7$



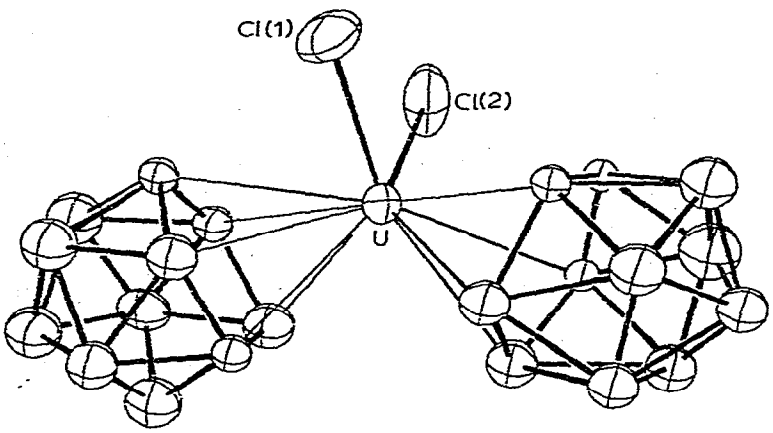
(421) 1,7-$[(C_5H_5)Co]_2$-5,6-$C_2B_5H_7$



(422) 7,8,9$[(C_5H_5)Ni]_3$-1-CB_5H_6

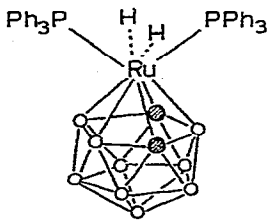
bonds [59]. (*420*)(*421*) Both 9-vertex tricapped trigonal prisms; thermal interconversion may result from opposing tendencies of Co-Co bond formation, and H...H repulsion of adjacent C_5H_5 rings [105]. (*422*) One of several isomers from *nido*-CB_5H_9 + $Ni(C_5H_5)_2$ + NaHg (compound III); electron-rich cluster, 2 e accommodated by elongation of an Ni-B bond to give 4-membered open face, distorted monocapped square antiprism [171,172]. (*423*) Entry deleted.

12-vertex polyhedra:

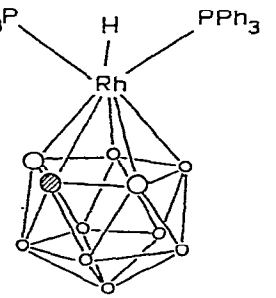


(*424*) $[UCl_2(C_2B_9H_{11})_2]^{2-}$

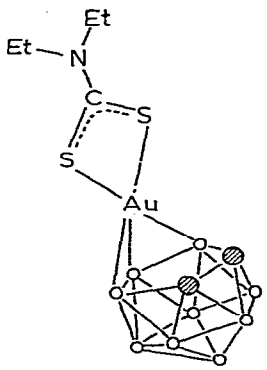
(*424*) First actinide metallocarborane complex; U has distorted tetrahedral geometry, η^5-carborane; centroid normals at 137°, Cl-U-Cl 90.3(5)°, U-B(C) 2.73Å(av.) [26]. (*425*) By oxidative addition of $7,9\text{-}C_2B_9H_{12}^-$ to $RuHCl(PPh_3)_3$; reversibly eliminates H_2 on heating in vacuum [347]. (*426*) Rh-H 1.54(9)Å, asymmetry in Rh-P bonds [2.357(3), 2.301(1)Å] probably results from strong packing interaction [346]. (*427*) 18 e slipped structure, Au associated with 3 B of pentagonal face [25]. (*428*) 20 e structure, also slipped (as expected); in both, Au position similar, with slip the result of lengthening B-C bonds, bending C_2B_3 plane [25].



(425) 2,1,7-$[(Ph_3P)_2RuH_2]C_2B_9H_{11}$



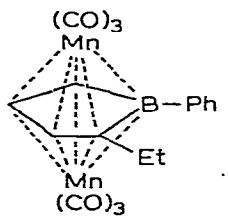
(426) $RhH(PPh_3)_2(C_2B_9H_{11})$



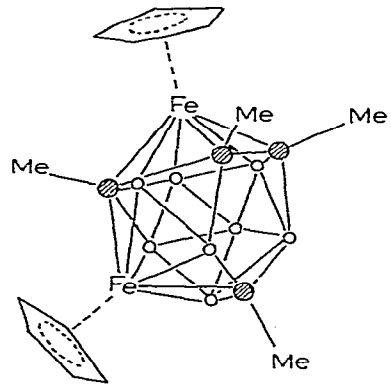
(427) 3-$[(Et_2NCS_2)Au]$-1,2-$C_2B_9H_{11}$

(428) No diagram available

Other complexes:

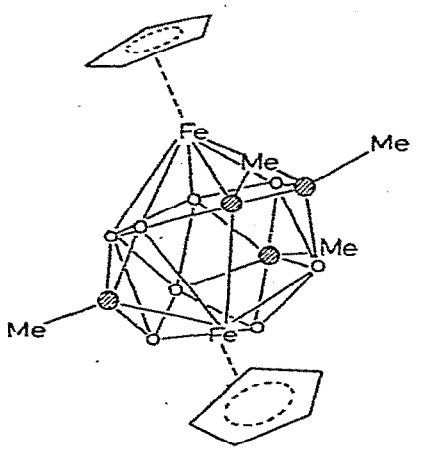


(429) $[Mn(CO)_3]_2C_4H_3EtBPh$

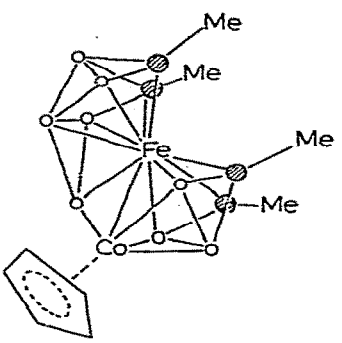


$[(C_5H_5)Fe]_2C_4Me_4B_8H_8$
(430) isomer (I) (brown)

(*429*) Contains central borole ligand, structure related to (*275*) [194]. (*430*)(*431*) Two of 4 isomers from $Me_4C_4B_8H_8 + NaC_{10}H_8/NaC_5H_5 + FeCl_2$; 14-vertex polyhedra, neither has predicted *closo* structure, with 5- and 4-membered open faces; (*430*) has 2 6-coordinate vertices (both Fe), open face has 3 C; (*431*) has 3 6-coordinate vertices (2 Fe, 1 B), with open face having 2 C [204]. (*432*) From $Co(CO)_2(C_5H_5)$ + $Fe(Me_2C_2B_4H_4)_2$; 2 pentagonal bipyramids fused at common Fe, with additional BH capping triangular faces on both polyhedra simultaneously; cluster is 2 e deficient [124,125].



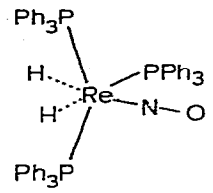
$[(C_5H_5)Fe]_2C_4Me_4B_8B_8$
(431) isomer (II) (green)



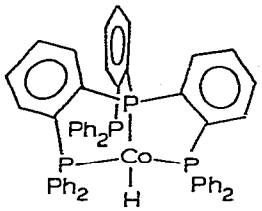
(432) $Me_4C_4B_8H_8FeCo(C_5H_5)$

HYDRIDE AND BOROHYDRIDE COMPLEXES

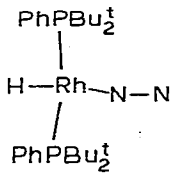
(*433*) Distorted octahedral Re, H partially located, with Re-H 1.57Å, H-Re-H 160°; axial P bent towards H, with P-Re-P 143° [408,409]. (*434*) Reference [407]. (*435*) Linear H-Rh-N-N, PR_3 ligands bent towards H; paper summarises structural data for M-N_2 complexes; some disorder, Rh-N 1.970(4), N-N 1.074(7)Å; latter distance is less than N_2, but anisotropic thermal motion correction gives length of 1.18Å [402]. (*436*) Isostructural with $Os(CO)_3(PPh_3)_2$; 2 types of molecule, disordered NO_3 in both, arrangement differs for different site symmetries; H not located, but occupies sites left statistically unoccupied by NO_3 [403]. (*437*) H not located, assumed to be in



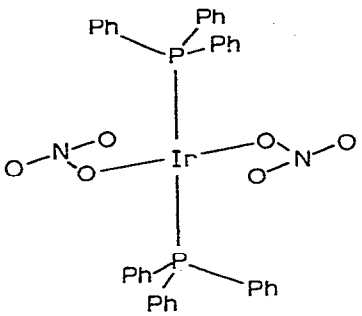
(433) $ReH_2(NO)(PPh_3)_3$



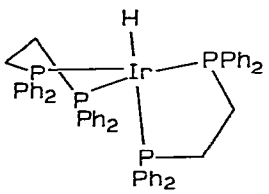
(434) $CoH[P(C_6H_4PPh_2)_3]$



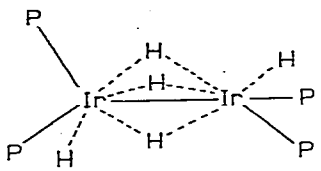
(435) $RhH(N_2)(PPhBu^t_2)_2$



(436) $IrH(NO_3)_2(PPh_3)_2$

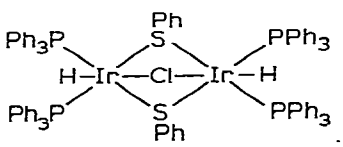


(437) $IrH(dppe)_2$

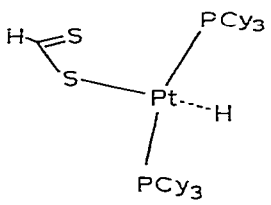


$P \equiv PPh_3$

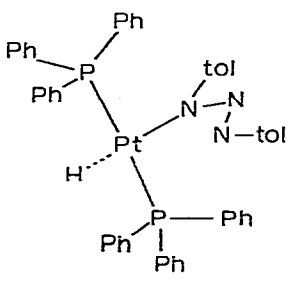
(438) $[Ir_2H_5(PPh_3)_4]^+$



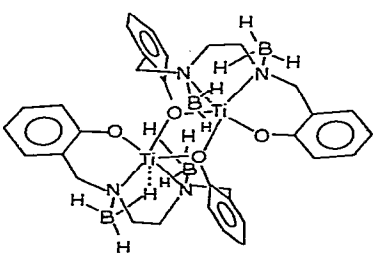
(439) $[Ir_2H_2Cl(SPh)_2(PPh_3)_4]^+$



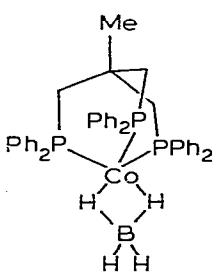
(440) *trans*-$PtH(S_2CH)(PCy_3)_2$



(441) $PtH[N_3(tol\text{-}p)_2](PPh_3)_2$



(443) $[Ti(BH_3)_2(salen)]_2$



(444) $Co(H_2BH_2)(P_3)$

$P_3 \equiv C_5H_{11}(PPh_2)_3$

axial position of TBP [406]. (*438*) Multiple Ir-Ir bond, bridged by H [410]. (*439*) 2 Ir bridged by Cl, 2 SPh; terminal H not located, but in free coordination site *trans* to Cl [411]. (*440*) H not located;

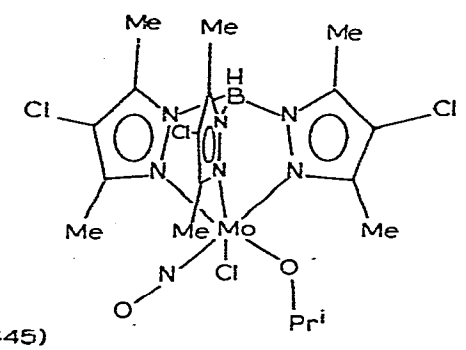
monodentate HCS_2 from $PtH_2(PCy_3)_2 + CS_2$ [404]. (*441*) H not located, *trans* to monodentate triazenide group [405]. (*442*) See (*314*).

See also: *5*, *6*, *79*, *88*, *89*, *170*, *425*, *426*.

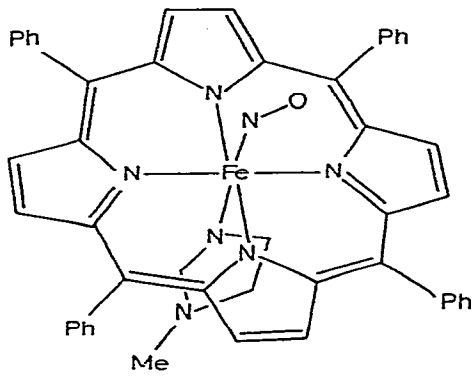
(*443*) BH_3 coordinates to salen N, with Ti-H interaction [1.85(15)Å] forming electron deficient N-B-H-Ti rings, with 7-coordinate Ti(IV); amine-borane is donor [412]. (*444*) Very distorted SP, with P at apex; Co is at centre of BP_3 tetrahedron, constrained P_3 ligand allows much closer approach of BH_4 to Co than found in $CoH(H_2BH_2)(PCy_3)_2$ [414].

NITROSYLS

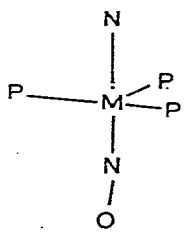
(*445*) From halogenation of $Mo(CO)_2(NO)[HB(Me_2pz)_3]$ in Pr^iOH; significant multiple bond character for Mo-O from $P_\pi(O) \rightarrow d_\pi(Mo)$ bonding; formal coordinative unsaturation results in NMR deshielding of C,H [418]. (*446*) NO disordered between 2 orientations to minimise intermolecular packing contacts; long Fe-N(im) bond [424]. (*447*)(*451*)(*456*) Comparison between complexes shows Fe TBP, and Co and Ni tetrahedral, with N not coordinated; structures rationalised



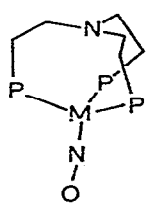
(445)
$MoCl(OPr^i)(NO)[HB(3,5\text{-}Me_2\text{-}4\text{-}ClC_3N_2)_3]$



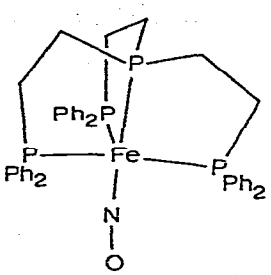
(446) Fe(NO)(Meim)(tpp)



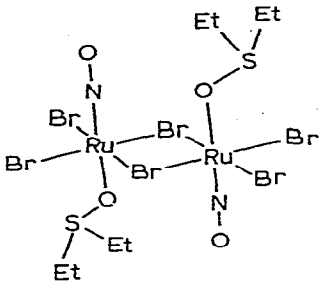
central coordination polyhedron
(447) $[Fe(NO)(up_3)]^+$



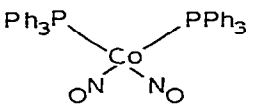
(451) M = Co^+; (456) M = Ni^+



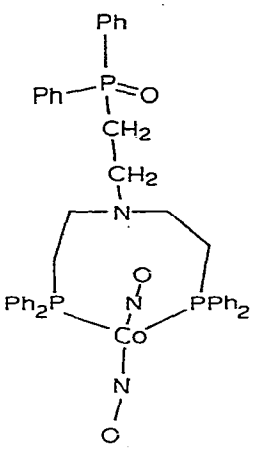
(448) $[Fe(NO)(PP_3)]^+$
$P{\equiv}PPh_2(CH_2)_2$



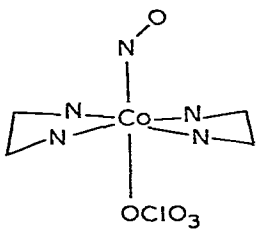
(449) $[RuBr_3(NO)(Et_2SO)]_2$



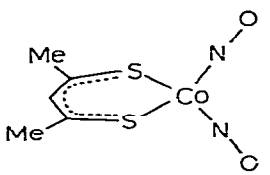
(450) $[Co(NO)_2(PPh_3)_2]^+$



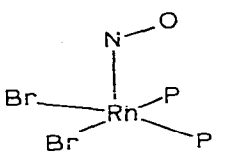
(452) $[Co(NO)_2(np_2o)]^+$



(453) $[Co(NO)(OClO_3)(en)_2]^+$



(454) $Co(NO)_2(sacsac)$



$P \equiv P(OPh)_3$
(455) $RhBr_2[P(OPh)_3]_2(NO)$

by simple MO approach [421]. (*448*) TBP, linear NO; Fe 0.28Å above plane [423]. (*449*) From oxidation of $RuBr_3(NO)(SEt_2)_2$ [417]. (*450*) Enables comparison with Rh,Ir complexes to be made; cobalt coordination is flattened tetrahedral, nearly SP, i.e. more NO^+ character, hence lack of reactivity to CO [420]. (*452*) Distorted tetrahedral Co, ligand bidentate via 2 P; linear NO group [422]. (*453*) Disorder in NO gives two O positions [415]. (*454*) Tetrahedral Co, bent NO; correlation of O-M-O and N-M-N angles for 13 $\{M(NO)_2\}^{10}$ complexes, interpretation in terms of 'attracto' and 'repulso' conformers [416]. (*455*) SP, with apical non-linear NO; long Rh-N 2.04(4)Å [cf. Ir-N 1.94(2)Å in $IrCl_2(NO)(PPh_3)_2$, but note high R value] [419]. (*456*) Entry deleted.

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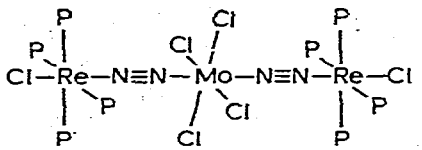
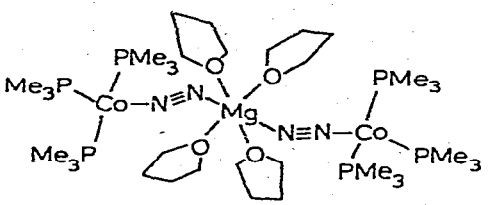
Complexes *46*, *136*, *244*, *307*, *308*, *402* and *433* also contain NO groups.

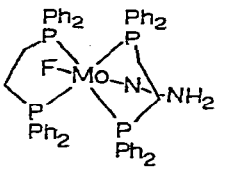
DINITROGEN, ARYLDIAZO AND ARYLDIIMINE COMPLEXES

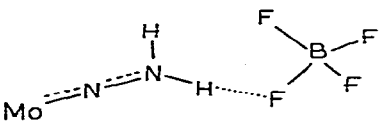


(457) $MoCl_4[(\mu\text{-}N_2)ReCl(PMe_2Ph)_4]_2$

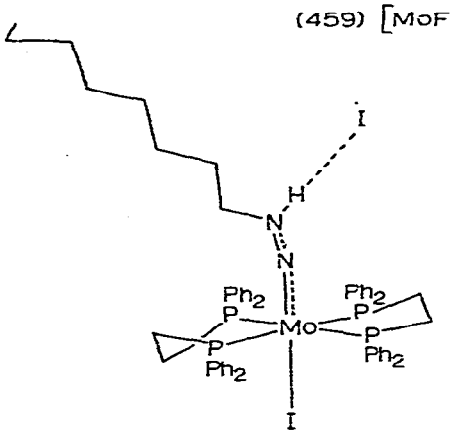


(458) $Mg(thf)_4[Co(PMe_3)_3N_2]_2$

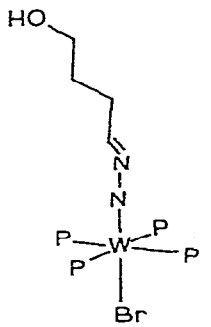




(459) $[MoF(N_2H_2)(dppe)_2]^+$



(460) $[MoI(N_2HC_8H_{17})(dppe)_2]I$

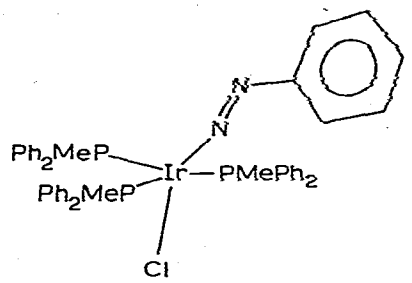


(461) $\{WBr[N_2CH(CH_2)_3OH](dppe)_2\}^+$

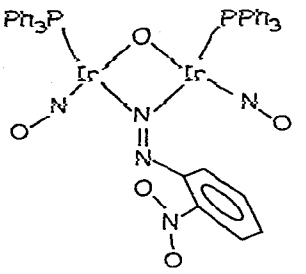
(*457*) Linear ReN_2MoN_2Re chain [426]. (*458*) Long N–N 1.18Å, with bent Mg–N–N(Co) 158°, modified N–N bond [425].

See also: *226*, *245*, *246*, *435*.

(*459*) Linear Mo–N–N precludes protonation of N^1 i.e. η^1-hydrazido ligand present; N–N 1.333(24)Å, bond order *ca.* 1.6; H-bond N–H...F–BF_3 as shown [429]. (*460*) Protonation of $N_2C_8H_{17}$ gives N-alkylhydrazido complex, with Mo=N=N multiple bond system indicated by bond distances [431]. (*461*) From $W(N_2)_2(dppe)_2$ + thf, with



(462) $[IrCl(N_2Ph)(PMePh_2)_3]^+$

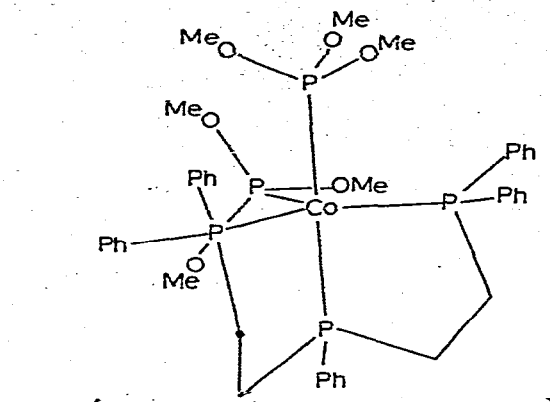


(463) $[Ir_2O(N_2C_6H_4NO_2)(NO)_2(PPh_3)_2]^+$

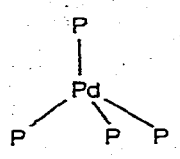
ring-opening of furan to give ω-diazo-butanol, originally thought to be tetrahydropyridazo ligand [430]. (*462*) Complex prepared with ArN_2 having geometry intermediate between singly and doubly bent, using other ligands of appropriate bulk; highly distorted. 5-coordinate Ir: Ir-N 1.835(8), N-N 1.241(11), N-C 1.421(11)Å; Ir-N-N 155.2(7)°, N-N-C 118.8(8)° [428]. (*463*) Aryldiazo group bridges non-bonding Ir...Ir atoms; N=N-Ar 140(4)°; no details of NO group parameters [427].

BINARY TRANSITION METAL-TERTIARY PHOSPHINE COMPLEXES

(*464*) Comparison with (*30*); TBP, with phosphite in axial and equatorial positions; changes in geometry of 2 complexes rationalised in terms of ligand bite; correlation of J(PP) with structure in non-fluxional 5-coordinate Co(I) complexes [355]. (*465*) Tetrahedral Pd, with equal Pd-P 2.443(5)Å [435]. (*466*)(*467*) Distances: *a* 2.70, 2.77; *b* 2.83, 2.83; *c* 2.6, 2.5; M-P 2.285, 2.252Å; P-M-P 176.6, 177° respectively; substituents eclipsed, and *o*-H interaction may be attractive (cf. strong deshielding by Pt) [433]. (*468*) Only 2 P in each ligand coordinated to Pt in axially-distorted tetrahedron; P-Pt-P 97.0(3)°, with Pt-P 2.287(8)Å, similar to other determinations [436]. (*469*) Disordered centrosymmetric, P-Au-P 180°, Au-P 2.316Å [432].

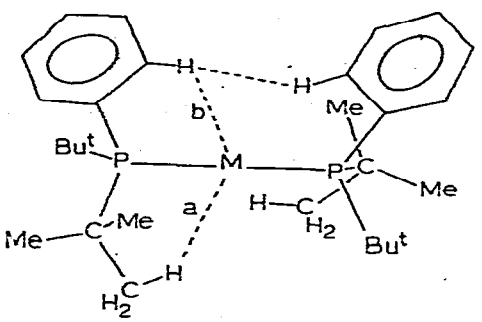


(464) $\left\{Co\left[PhP(CH_2CH_2PPh_2)_2\right]\left[P(OMe)_3\right]_2\right\}^+$



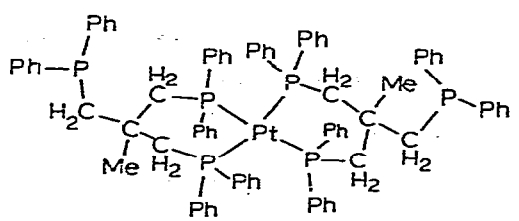
P ≡ PPh_3

(465) $Pd(PPh_3)_4$



$M(PBu^t_2Ph)_2$

(466) M = Pd; (467) M = Pt



(468) $Pt\left[(Ph_2PCH_2)_3CMe\right]_2$

P—Au—P

P ≡ $PMePh_2$

(469) $\left[Au(PMePh_2)_2\right]^+$

TABLE 1 *Metal-metal bond distances reported in 1976.*

Bond	Length(Å)	Complex		Structure	Reference
(a) *Non-cluster complexes*					
Ti-Ti	3.336(4)	$(C_5H_5)_2Ti(C_5H_4)Ti(thf)(C_5H_5)$		(*315*)	270
Cr-Cr	2.214(1)	$Cr_2(C_8H_8)_3$		(*316*)	269
Cr-As	2.53	$[Cr(CO)_5]_3As_2Ph_2$	As → Cr	(*392*)	299
	2.64		As_2 → Cr		
Mo-Mo	2.129(1)	$Mo_2(OAc)_2[Et_2B(pz)_2]_2$		(*305*)	251
	2.147(3)	$Mo_2(OAc)_2[HB(pz)_3]_2$		(*306*)	251
	2.977(1)	$[Mo(CO)_2(C_5H_5)]_2C_2Et_2$		(*320*)	225
	3.16	$[MoI(CO)_4]_2$		(*300*)	60
	3.270(1)	$Mo_2(CO)_5(PEt_3)(gazl)$	(isomer I)	(*317*)	293
	3.274(2)		(isomer II)	(*318*)	293
Mo-Re	3.111(2)	$MoRe(CO)_9(CPh)$		(*322*)	162
Mo-Sn	2.688(2)	$Mo(SnCl_3)Cl(CO)_3[MeS(CH_2)_2SMe]$		(*282*)	42
W-W	2.255	$W_2(CH_2SiMe_3)_6$		(*304*)	277
	2.275(4)	$W_2Me_2(CO_2NEt_2)_4$		(*303*)	258
	2.288	$W_2Me_2(NEt_2)_4$		(*302*)	205
	3.155	$[WI(CO)_4]_2$		(*301*)	61

	3.264(1)	$W_2(CO)_6$(gazl)		(*319*)	242
	3.393(4)	$W_2H(CO)_8(NO)[P(OMe)_3]$	H-bridged	(*307*)	18
Mn-Mn	2.734(2)	$[Mn(CO)_2(C_5H_5)]_2C{:}CHPh$		(*228*)	156
	2.807(4)	$[Mn(CO)_4]_3N_2Me$	Bridged	(*352*)	114
	2.826(3)	$[Mn(CO)_4]_3N_2Me$			
	3.086	$[Mn(CO)_4]_2[SnBrMn(CO)_5]_2$		(*396*)	206
Mn-Fe	2.742,2.752(4)	$[(C_5H_5)(CO)_2MnFe_2(CO)_6]PPh$		(*397*)	208
	2.760(4)	$[(C_5H_5)Mn(CO)_2C(CO)CHPh]Fe(CO)_3$		(*324*)	209
	2.94	$[(C_5H_5)(CO)_2MnFe(CO)_4PPh]Fe(CO)_3$		(*398*)	220
Mn-In	2.664(1) (av.)	$\{[Mn(CO)_5]_2InBr\}_2$		(*394*)	237
	2.665(1) (av.)	$\{[Mn(CO)_5]_2InCl\}_2$		(*393*)	237
	2.672(1) (av.)	$\{[Mn(CO)_5]_2InI\}_2$		(*395*)	237
Mn-Si	2.453(4)	$Mn(SiMe_3)(CO)_4(PPh_3)$		(*283*)	281
Mn-Sn	2.630	$[Mn(CO)_4]_2[SnBrMn(CO)_5]_2$		(*396*)	206
	2.635	$[Mn(CO)_5]_2SnCl_2$		(*289*)	75
	2.642	$[Mn(CO)_5]_2SnBr_2$		(*290*)	75
	2.707,2.747(3)	$[Mn(CO)_5]_4Sn_2Br_2$		(*291*)	238
Re-Re	2.178(1)	$[Re_2Me_8]^{2-}$		(*309*)	58
Re-Si	2.562(12)	$Re(SiH_3)(CO)_5$		(*285*)	17
	2.600(1)	$Re(SiMe_3)(CO)_5$		(*286*)	49
	2.665(9)	$Re[Si(SiMe_3)_3](CO)_5$		(*287*)	49
Re-Ge	2.628(6)	$Re(GeH_3)(CO)_5$		(*288*)	17

Fe-Fe	2.316(1)	$Fe_2(CO)_6(C_2Bu^t_2)$	(*331*)	169
	2.354(5)	$Fe_2(CO)_6(PhCHMeNCHCO_2Et)_2$	(*356*)	197
	2.415	$Fe_2(CO)_6[Me_2C{:}C(O)CSMe_2]$	(*328*)	120
	2.470(2)	$Fe_2(CO)_6(ONCMe_2)(NHCHMe_2)$	(*354*)	101
	2.482(1)	$[Fe(CO)(CS)(C_5H_5)]_2$	(*310*)	131
	2.484(2)	$Fe_2(CO)_6(N{:}CMe_2)(ONCMe_2)$	(*353*)	99
	2.495(4)	$Fe_2(CO)_6(C_4Ph_4)$	(*326*)	329
	2.500(2)	$[Fe(CO)_2(C_{10}H_8)]_2$	(*338*)	364
	2.515(2)	$Fe_2(CO)_5(PPh_3)(C_4Ph_4)$	(*339*)	388
	2.536(1)	$Fe_2(CO)_6(C_4Ph_4CO)$	(*337*)	334
	2.548(1)	$Fe_2(CO)_6(PPh_2)(CHCPhNEt_2)$	(*403*)	313
	2.551(1)	$Fe_2(CO)_6(PhCHMeN{:}CHCO_2Et)$	(*355*)	197
	2.5876(14)	$Fe_2(CO)[C_4(CF_3)_4CO](C_5H_5)_2$	(*333*)	92
	2.627(3)	$Fe_2(CO)_5(PPh_3)(C_4H_4)$	(*336*)	300
	2.637	$Fe_2(CO)_6(C_7H_7)$	(*327*)	117
	2.686(4)	$[(C_5H_5)(CO)_2MnFe_2(CO)_6]PPh$	(*397*)	208
	2.74	$[(C_5H_5)(CO)_2MnFe(CO)_4PPh]Fe(CO)_3$	(*398*)	220
	2.745	$Fe_2(CO)_6(CH_2CMeCCRCO)$	(*329,330*)	132
	2.747(1)	$Fe_2(NO)_4[P(CF_3)_2]_2$	(*402*)	31
	2.821	$Fe_2(CO)_6[P(CF_3)_2]_2$	(*401*)	76
	3.059(1)	$[(C_5H_5)_2Fe_2(S_2)(SEt)_2]^+$	(*400*)	144
Fe-Co	2.480(1)	$[(C_5H_5)Co]FeMe_4C_4B_8H_8$	(*432*)	124,125
	2.70	$FeCo(AsMe_2)(CO)_7$	(*407*)	63
Fe-Rh	2.764(1)	$(C_7H_7)FeRh(CO)_5$	(*346*)	89
Fe-Hg	2.498	*cis*-$Fe(HgCl)_2(CO)_4$	(*293*)	27
	2.516(4)	$[Fe(HgCl)(HgCl_2)(CO)_4]^-$ Fe-HgCl	(*294*)	28
	2.560(3)	$[Fe(HgCl)(HgCl_2)(CO)_4]^-$ $Fe-HgCl_2$	(*294*)	28

Fe-Si	2.224(9)	$[Fe(SiCl_3)(CO)_4]^-$	(*293*)	29
Fe-As	2.33	$FeCo(AsMe_2)(CO)_7$	(*407*)	63
Fe-Sb	2.515,2.525 2.606	$Fe(CO)_4(SbPh_2)FePh(CO)_7(SbPh_3)$	(*406*)	361
Ru-Ru	2.759(4)	$Ru_2(CO)_4(py)_2(mbt)_2$	(*342*)	288
Os-Os	2.754(2)	$Os_2(CO)_6(C_8H_6)$	(*343*)	128
Co-Co	2.36	$[Co(CO)(C_5H_5)]_2^{\cdot-}$	(*311*)	91
	2.39	$Co_2(CO)_5[CH{:}CPhC(O)OC{:}CHCMeCHCMe]$	(*345*)	240
	2.444(2)	$[(C_5H_5)Co]_2C_2B_5H_7$	(*421*)	105
	2.463(1)	$Co_2(CO)_6(C_2Bu^t_2)$	(*344*)	169
Co-Li	2.39,2.44	$(cod)_2CoLi(LiPh)(OEt_2)$	(*270*)	295
Co-Si	2.288	$Co_3Si[Co(CO)_4](CO)_9$ *extra*-cluster	(*411*)	126
Co-Ge	2.416(4)	$Co(GeH_3)(CO)_4$	(*295*)	16
Co-As	2.24	$FeCo(AsMe_2)(CO)_7$	(*407*)	63
Rh-Rh	2.740(1)	$[Rh(PF_3)_2PPh_3]_2C_2Ph_2$	(*347*)	385
	2.975	$Rh_4(CO)_8(biim)_2$	(*19*)	218
	3.063(3)	$Rh_2(C_8H_9)_2(cod)$	(*159*)	271
Rh-Cu	2.73	$RhCuCl(N_3Me_2)(CO)(PPh_3)_2$	(*357*)	348,349
Ir-Ir	2.518	$[Ir_2H_5(PMe_2Ph)_4]^+$	(*438*)	410
Ir-Cu	2.686(3)	$IrCu(CO)Cl(N_3Me_2)(PMe_2Ph)_2$	(*358*)	216
Ir-Ag	2.874(2)	$IrAg(CO)(O_2CCHMe_2)(MeN_3tol)(PPh_3)_2$	(*359*)	383
Ir-Hg	2.570(1)	$IrCl_2(HgCl)(CO)(PPh_3)_2$	(*296*)	340
	2.578(2)	$IrClBr(HgBr)(CO)(PPh_3)_2$	(*297*)	340

Ni-Ni	2.345(3)	$[Ni(C_5H_5)]_2C_2H_2$	(*348*)	21
	2.4040(5)	$[Ni(C_5H_5)]_3CB_5H_6$	(*422*)	171,172
	2.617(2)	$[Ni(cod)]_2C_2Ph_2$	(*349*)	315
	2.749(7) (av.)	$NiPh_2(N_2)$-NaLi complex	(*280*)	401
Ni-Li	2.63(4) (av.)	$NiPh_2(N_2)$-NaLi complex	(*280*)	401
Ni-Na	2.795	$[NiPh_2(C_2H_4)]_2Na_4(thf)_5$	(*279*)	379
	2.963(20) (av.)	$NiPh_2(N_2)$-NaLi complex	(*280*)	401
	3.037	$[NiPh_2(C_2H_4)]_2Na_4(thf)_5$	(*279*)	379
	3.105(18) (av.)	$NiPh_2(N_2)$-NaLi complex	(*280*)	401
Pd-Pd	2.5310(9)	$[Pd_2(CNMe)_6]^{2+}$	(*312*)	106
	2.57	$[PdAl_2Cl_7(C_6H_6)]_2$	(*350*)	96
	2.5921(5)	$[Pd_3(CNMe)_6(PPh_3)_2]^{2+}$	(*313*)	378
Pt-Pt	2.692(3)	$[PtH(SiMe_3)(PCy_3)]_2$	(*314*)	380
	2.890(2)	$Pt_2(C_2Ph_2)_2(PMe_3)_2$	(*351*)	306
Pt-Si	2.292	$PtCl[Si(OCH_2CH_2)_3N](PMe_2Ph)_2$	(*299*)	328
	2.317(2)	$PtCl[SiMePh(nap)](PMe_2Ph)_2$	(*298*)	255
	2.33(1)	$[PtH(SiMe_3)(PCy_3)]_2$	(*314*)	380
Au-Au	2.597(5)	$[AuCl(CH_2)_2PEt_2]_2$	(*87*)	110

(b) *Cluster complexes*

Re-Re	2.797(4)	$[Re_3H_3(CO)_{10}]^{2-}$	(*361*)	62
	2.913 (av.)	$Re_4H_4(CO)_{12}$	(*364*)	87
	2.963,2.973(1)	$[Re_3OH_3(CO)_9]^{2-}$	(*399*)	62
	3.026(2)	$[Re_4H_4(CO)_{15}]^{2-}$	(*366*)	150

	3.031(5)	$[Re_3H_3(CO)_{10}]^{2-}$	H-bridged	(*361*)	62
	3.09 (av.)	$[Re_4H_4(CO)_{13}]^{2-}$		(*366*)	115
	3.181,3.184 3.287(2)	$[Re_4H_4(CO)_{15}]^{2-}$	H-bridged	(*366*)	150
Fe-Fe	2.531(10)	$Fe_3(CO)_7(PPh_2)[Ph_2PC_4(CF_3)_2]$		(*404*)	339
	2.542,2.590(3)	$Fe_3(CO)_8(C_9H_{12})$	Bridged	(*379*)	182
	2.627 (av.)	$[Fe_4H(CO)_{13}]^{2-}$		(*367*)	113
	2.662(7)	$Fe_3(CO)_7(PPh_2)[Ph_2PC_4(CF_3)_2]$	PPh_2-bridged	(*404*)	339
	2.708(3)	$Fe_3(CO)_8(C_9H_{12})$		(*379*)	182
	2.738(6),2.792(4)	$Fe_3(AsPh)_2(CO)_9$		(*405*)	239
Fe-As	2.317,2.344(4)	$Fe_3(AsPh)_2(CO)_9$		(*405*)	239
Ru-Ru	2.741,2.766(1)	$Ru_3H(CO)_9(C_6H_9)$		(*381*)	155
	2.743(4)	$Ru_3(CO)_{10}(C_4H_4N_2)$	Bridged	(*362*)	127
	2.76	$Ru_4H_4(CO)_{11}[P(OMe)_3]$			
	2.792(1)	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]$ (II)		(*383*)	284
	2.7997(5)	$Ru_3H(CO)_{10}(C{:}NMe)$	Bridged	(*380*)	13
	2.810,2.827(1)	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]$ (I)		(*382*)	284
	2.828 (av.)	$Ru_3H(CO)_{10}(C{:}NMe)$		(*380*)	13
	2.850,2.861(1)	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]$ (II)	Under C_5 ring	(*383*)	284
	2.858(4)	$Ru_3(CO)_{10}(C_4H_4N_2)$		(*362*)	127
	2.87(1)	$[Ru_6H(CO)_{18}]^-$		(*374*)	192
	2.93 (av.)	$Ru_4H_4(CO)_{11}[P(OMe)_3]$	Bridged	(*368*)	87
	2.933(1)	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]$ (I)	Bridged	(*382*)	284
	2.994(1)	$Ru_3H(CO)_9(C_6H_9)$	Bridged	(*381*)	155

Os-Os	2.710-2.867	$[Os_5H(CO)_{16}]^-$		(373)	149
	2.738, 2.749, 2.764(3)	$Os_5(CO)_{16}$		(372)	180
	2.771-2.900	$[Os_6(CO)_{17}CO_2Os_3H(CO)_{10}]^-$	(Os_6)	(378)	302
	2.794(2)	$Os_3(CO)_9[(HC_2Ph)_2CO]$		(389)	285
	2.8002(6)	$Os_3H(CO)_{10}(CHCH_2PMe_2Ph)$	Bridged	(388)	221
	2.805-2.965	$Os_6H_2(CO)_{18}$		(377)	191
	2.814	$[Os_6(CO)_{18}]^{2-}$		(375)	191
	2.834, 2.858(1)	$Os_3H(CO)_{10}(C_4H_7)$		(386)	130
	2.856(3)	$Os_3(CO)_{10}(C_4H_6)$ (II)		(385)	129
	2.857(2)	$Os_3(CO)_9[(HC_2Ph)_2CO]$	Asym CO-bridged	(389)	285
	2.860, 2.863	$Os_3H(CO)_9(C_6H_7)$		(387)	154
	2.861, 2.863	$Os_3(CO)_{10}(C_4H_6)$ (I)		(384)	129
	2.863 (av.)	$[Os_6H(CO)_{18}]^-$		(376)	191
	2.864-2.895	$[Os_6(CO)_{17}CO_2Os_3H(CO)_{10}]^-$	(Os_3)	(378)	302
	2.866, 2.889(3)	$Os_5(CO)_{16}$	$Os-Os(CO)_4$	(372)	180
	2.8688, 2.8729	$Os_3H(CO)_{10}(CHCH_2PMe_2Ph)$		(388)	221
	2.876, 2.886	$[Os_6(CO)_{18}]^{2-}$		(375)	191
	2.880(2)	$Os_3(CO)_9[(HC_2Ph)_2CO]$	allyl-bridged	(389)	285
	2.884(3)	$Os_3(CO)_{10}(C_4H_6)$ (I)	*trans* to C=C	(384)	129
	2.923(1)	$Os_3H(CO)_{10}(C_4H_7)$		(386)	130
	2.932	$Os_3(CO)_{10}(C_4H_6)$ (III)	C_4H_6-bridged	(385)	129
	2.973	$[Os_6H(CO)_{18}]^-$	H-bridged face	(376)	191
	3.054	$Os_3H(CO)_9(C_6H_7)$	H-bridged	(387)	154
Co-Co	2.440, 2.458(4)	$[Co(CO)(C_5H_5)]_3$	CO-bridged	(390)	196
	2.457-2.527	$Co_4(CO)_{12}$		(369)	111

	2.477 (av.)	$Co_3C[OTiCl(C_5H_5)_2](CO)_9$		*(410)*	219
	2.479 (av.)	$Co_3C(OCOMe)(CO)_9$		*(408)*	85
	2.484 (av.)	$Co_3C(OBBr_2NEt_3)(CO)_9$		*(409)*	166
	2.52 (av.)	$[Co_8C(CO)_{18}]^{2-}$		*(413)*	161
	2.521(4)	$[Co(CO)(C_5H_5)]_3$		*(390)*	196
	2.53	$[Co_6C(CO)_{14}]^-$	CO-bridged	*(412)*	161
	2.604 (av.)	$Co_3Si[Co(CO)_4](CO)_9$		*(411)*	126
	2.75 (av.)	$[Co_6C(CO)_{14}]^-$		*(412)*	161
	2.961(1)		"stretched"		
Co-C	1.84-1.94	$[Co_6C(CO)_{14}]^-$		*(412)*	161
	1.889 (av.)	$Co_3C(OCOMe)(CO)_9$		*(408)*	85
	1.93 (av.)	$Co_3C(OBBr_2NEt_3)(CO)_9$		*(409)*	166
	1.95-2.20	$[Co_8C(CO)_{18}]^{2-}$		*(413)*	161
	1.96 (av.)	$Co_3C[OTiCl(C_5H_5)_2](CO)_9$		*(410)*	219
Co-Si	2.218 (av.)	$Co_3Si[Co(CO)_4](CO)_9$		*(411)*	126
Rh-Rh	2.66,2.75,2.99	$[Rh_4(CO)_{11}]^{2-}$		*(370)*	84
	2.671-2.740(1)	$Rh_4(CO)_8(dppm)_2$		*(371)*	396
	2.87 (av.)	$[Rh_{15}C_2(CO)_{28}]^-$		*(414)*	317
	2.90 (av.)	$[Rh_{15}C_2(CO)_{28}]^-$	From central Rh	*(414)*	317
Rh-C	2.04 (av.)	$[Rh_{15}C_2(CO)_{28}]^-$		*(414)*	317
	2.374	$Ni_4(CNBu^t)_4(C_2Ph_2)_3$	apex-base	*(391)*	398
Ni-Ni	2.648 (av.)	$Ni_8(CO)_8(PPh)_6$		*(415)*	363
	2.686	$Ni_4(CNBu^t)_4(C_2Ph_2)_3$	base-base	*(391)*	398
	2.749(7)	$NiPh_2(N_2)$-NaLi complex		*(280)*	401
Ni-P	2.183 (av.)	$Ni_8(CO)_8(PPh)_6$		*(415)*	363

TABLE 2 *M-N-O Bond Parameters in Nitrosyl Complexes*

Complex	Structure	M-N(Å)	N-O(Å)	M-N-O(°)	Reference
$Cr(NO)I(CO)_2(PMePh_2)_2$	(*7*)	1.705(14)	1.117(20)	*ca.* 180	307
$Mo(NO)Cl(OPr^i)[HB(C_3Me_2ClN_2)_3]$	(*445*)	1.764(8)	1.176(11)	179.4(8)	418
$HW_2(NO)(CO)_8[P(OMe)_3]$	(*307*)	1.818(4)	1.172(4)	178.4(3)	18
$Mn(NO)(CO)(PPh_3)(exo\text{-}MeC_5H_5)$	(*163*)	1.674(5)	1.200(7)	178.4(5)	280
$Re(NO)H_2(PPh_3)_3$	(*433*)	1.77(2)	1.25(3)	175(2)	409
$[Fe(NO)(np_3)]^+$	(*447*)	1.60(7)	1.19(9)	164(7)	421
$[Fe(NO)(pp_3)]$	(*448*)	1.67(1)	1.16(1)	177.4(7)	423
Fe(NO)(Meim)(tpp)	(*446*)	1.743(5)	1.121(8)	142.1(6)	424
$[Ru(NO)Br_3(Et_2SO)]_2$	(*449*)	1.71(1)	1.16(1)	178(1)	417
$[Co(NO)(OClO_3)(en)_2]^+$	(*453*)	1.806(6)	1.044(11)	135(1),141(2)	415
$[Co(NO)(np_3)]^+$	(*451*)	1.83(6)	1.14(10)	165(7)	421
$[Co(NO)_2(PPh_3)_2]^+$	(*450*)	1.645(6)	1.174(6)	171.0(5)	420
$[Co(NO)_2(np_2o)]^+$	(*452*)	1.61,1.62(1)	1.17,1.19(1)	175.3,175.8(10)	422
$Co(NO)_2$(sacsac)	(*454*)	1.650(6)	1.120(5)	168.9(5)	416[a]
$Rh(NO)(SO_2)(PPh_3)_2$	(*136*)	1.81	1.20	140	336
$Rh(NO)Br_2[P(OPh)_3]_2$	(*455*)	2.04(4)	1.26(9)	109(5)	419
$[Ni(NO)(np_3)]^+$	(*456*)	1.59(2)	1.14(3)	167.7(2.1)	421

[a] This paper contains a summary of bond parameters for four-coordinate tetrahedral $M(NO)_2$ complexes.

TABLE 3. ORGANOMETALLICS

No.	FORMULA	STRUCTURE	CRYSTAL CLASS	SPACE GROUP	Z
	C_1				
10	$CCl_3OPt^-.C_{16}H_{36}N^+$	$NBu_4[PtCl_3(CO)]$	M	$P2_1/n$	4
	C_3				
51a	$C_3H_3AuN_2$	Au(CN)(CNMe)	O	Pbcm	4
416	$C_3H_8B_5FeO_3^-.C_{15}H_{36}N^+$	$NBu_4[Fe(CO)_3(B_5H_8)]$	M	$P2_1/b$	4
	C_4				
295	$C_4H_3CoGeO_4$	$Co(GeH_3)(CO)_4$			
428	$C_4H_{22}AuB_{18}^-.C_{10}H_{20}AuN_2S_4^+$	$[Au(S_2CNEt_2)_2][Au(C_2B_9H_{12})_2]$	M	$P2_1/c$	2
424	$C_4H_{22}B_{18}Cl_2U^{2-}.2C_{16}H_{32}LiO_4^+$	$[Li(thf)_4]_2[UCl_2(C_2B_9H_{11})_2]$	M	Cc	4
293	$C_4Cl_2FeHg_2O_4$	*cis*-$Fe(HgCl)_2(CO)_4$[c]	M	C2/c	32
294	$C_4Cl_3FeHg_2O_4^-.C_{24}H_{20}As^+$	$AsPh_4[Fe(HgCl)(HgCl_2)(CO)_4]$	Tri	$P\bar{1}$	2
292	$C_4Cl_3FeO_4Si^-.C_8H_{20}N^+$	$NEt_4[Fe(SiCl_3)(CO)_4]$	M	$P2_1$	2
4	$C_4CoO_4^-.C_8H_{24}PSi_2^+$	$[PMe_2(SiMe_3)_2][Co(CO)_4]$	M	$P2_1/n$	n.g.
402	$C_4F_{12}Fe_2N_4O_4P_2$	$\{Fe(NO)_2[P(CF_3)_2]\}_2$	Tri	$P\bar{1}$	1
3	$C_4FeO_4^{2-}.2Na^+.1\frac{1}{2}C_4H_8O_2$	$Na_2Fe(CO)_4.1\frac{1}{2}C_4H_8O_2$	Tet	$P4_2/m$	4

[c] Formulated as "$[Fe(CO)_4(HgCl)_2]_3[Fe(CO)_3(HgCl)Cl]$".

	C_5				
288	$C_5H_3GeO_5Re$	$Re(GeH_3)(CO)_5$			
285	$C_5H_3O_5PeSi$	$Re(SiH_3)(CO)_5$			
244	C_5H_5NiNO	$Ni(NO)(C_5H_5)$			

a	b	c	α	β	γ	DATA	R	R'	NOTES	REFERENCE
14.949(8)	8.892(7)	18.232(10)		105.5(3)		2358	4.2	4.4		22
10.989(6)	6.899(6)	6.359(5)				325	6.8			23
11.108(6)	15.462(12)	15.443(9)			95.87(5)	1557	8.0	9.2		24
									ED	16
8.3786(9)	20.6068(22)	10.0656(11)		105.346(8)		1486	2.2			25
26.410(7)	11.248(5)	20.163(6)		102.35(1)		2020	5.8	6.1		26
36.81(1)	11.181(2)	20.369(5)		95.28(3)		2647	7.1	7.2		27
11.848(3)	13.524(4)	11.624(3)	123.87(2)	94.01(2)	90.91(2)	2917	5.9	7.1		28
8.390(1)	10.482(1)	11.528(1)		77.32(2)		846	8.7			29
14.713(5)	9.877(3)	15.537(4)		115.36(2)		1644	8.7	12.6		30
7.294(1)	7.492(1)	8.147(2)	81.18(2)	73.49(2)	84.77(2)	2156	5.19	4.75		31
10.690(5)		12.283(6)				1175	6.1	7.8		32
									ED	17
									ED	17
									ED	15

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111	$C_5H_{11}Ni^-.C_{12}H_{32}LiN_4^+$	[Li(tmed)][NiMe$(C_2H_4)_2$]	M	$P2_1/n$	4
123	$C_5H_{12}Cl_3NPt$	$PtCl_3(C_5H_{12}N)^a$	M	$P2_1/c$	4
417	$C_5H_{13}B_4Co$	$Co(B_4H_8)(C_5H_5)^b$	O	Pnma	4

^{a} $C_5H_{12}N$ = *cis*-pent-3-enylammonium. ^{b} 2-[(C_5H_5)Co]-*nido*-pentaborane(9).

C_6

60	$C_6H_3O_5Re$	$ReMe(CO)_5$			
120	$C_6H_{14}Cl_2N_2Pt$	*trans*-$PtCl_2(C_2H_4)$(CHMe:$NNMe_2$)	M	$P2_1$	2
8	$C_6Cl_3O_6Re_2^-.C_{36}H_{47}N^+.H_2O$	[oepH$_3$][$Re_2Cl_3(CO)_6$].H_2O	M	$P2_1/a$	4
2	C_5CrO_6	$Cr(CO)_6$	O	Pnma	4
1	$2C_6O_5V^-.C_{16}H_{32}O_4V^{2+}$	[V(thf)$_4$][$V(CO)_6$]	Tri	$P\bar{1}$	1

^{a} 1482 low order refl. ($2\theta<65°$), 2645 high order refl. ($2\theta<65°$), collected at 74K; combined X-ray–neutron diffr. study.

C_7

108	$C_7H_6FeO_4S$	$Fe(CO)_3(C_4H_6SO)^a$	M	$P2_1/c$	4
20	$C_7H_7CrO_5PS$	$Cr(CO)_5[PMe_2(SH)]$	M	$P2_1/c$	4
213	$C_7H_9O_2RhS$	$Rh(SO_2)(C_2H_4)(C_5H_5)$	O	Pcmn	4
282	$C_7H_{10}Cl_4MoO_3S_2Sn.CH_2Cl_2$	$Mo(SnCl_3)Cl(CO)_3$[MeS$(CH_2)_2$SMe].CH_2Cl_2	Tri	$P\bar{1}$	2
427	$C_7H_{21}AuB_9NS_2$	3-(Et_2NCS_2)-3,1,2-$AuC_2B_9H_{11}$	M	$P2_1/c$	4

^{a} C_4H_6SO = 2,5-dihydrothiophene-1-oxide.

C_8

59	$C_8H_3MnO_7$	Mn(COCOMe)$(CO)_5$	M	$P2_1/n$	4
164	$C_8H_7FeN_2O_3^+.C_2F_3O_2^-$	[Fe$(CO)_3(C_5H_7N_2)$]OCOCF$_3^a$	Tri	$P\bar{1}$	2

10.022(2)	15.121(3)	14.501(1)		96.82(1)		1701	6.9			33
8.521(5)	7.569(8)	15.073(16)		102.45(7)		2554	4.4	4.4		34
12.014(21)	9.635(17)	7.588(14)				738	5.3			35
									ED	17
6.652(7)	11.961(8)	7.143(7)		98.8		866	6.5			36
18.140(3)	19.847(3)	13.625(2)		111.64(2)		2197	9.1	10.3		37
11.505(4)	10.916(3)	6.203(2)				a		2.9	74K	19
9.467(2)	10.312(2)	11.128(3)	83.29(2)	109.56(3)	117.26(2)	3296	6.3			38
7.997	10.086	13.534		124.64						39
6.882(4)	13.290(6)	13.224(4)		91.60(3)		1579	8.5			40
7.230(2)	8.420(2)	14.124(3)				696	2.9			41
9.835(3)	11.068(3)	9.006(2)	92.04(2)	119.28(2)	96.36(2)	2192	8.6			42
7.3710(6)	11.0368(9)	21.1564(17)		93.22(4)		1962	4.0			25
6.320(3)	6.340(3)	29.971(13)			119.48(3)	775	2.7	3.6		43
10.812(7)	9.403(6)	6.690(4)	94.72(7)	78.16(5)	103.59(5)	3385	3.7	4.3		44,45

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62	$C_8H_4O_8Re_2$	*cis*-Re[(MeCO)$_2$H](CO)$_4$	M	P2$_1$/n	4
118	$C_8H_8Cl_2N_2Pt$	PtCl$_2$(C$_2$H$_4$)(4-CNpy)	M	Cc	4
40	$C_8H_8FeO_4S_2$	Fe(CO)$_4$(C$_4$H$_8$S$_2$)[b]	O	Pna2$_1$a	4
286	$C_8H_9O_5ReSi$	Re(SiMe$_3$)(CO)$_5$	M	P2$_1$/c	4
119	$C_8H_{11}Cl_2NPt$	PtCl$_2$(C$_2$H$_4$)(4-Mepy)	M	P2$_1$/n	4
130	$C_8H_{12}ClCuN_2$	CuCl(C$_8$H$_{12}$N$_2$)[c]	Tri	P$\bar{1}$	2
45	$C_8H_{12}ClMoN_3O^+.I_3^-$	[MoOCl(CNMe)$_4$]I$_3$	M	I2/m	2
–	$nC_8H_{12}Cl_3CuHg$	[CuCl(HgCl$_2$)(C$_8$H$_{12}$)]$_n$	O	Cmca *or* C2ca	8
226	$C_8H_{14}Mn_2N_2O_4$	[Mn(CO)$_2$(C$_5$H$_4$Me)]$_2$N$_2$	M	P2$_1$/n	2
82	$C_8H_{16}Cl_4O_4Pt_2$	{PtCl$_2$[CH$_2$C(OMe)$_2$]}$_2$	M	P2$_1$/c	2
121	$C_8H_{18}Cl_2N_4Pt$	PtCl$_2$(C$_2$H$_4$)(C$_6$H$_{14}$N$_4$)[e]	O	Cmcm	4
72	$C_8H_{21}ClNiOP_2$	*trans*-NiCl(COMe)(PMe$_3$)$_2$	O	P2$_1$2$_1$2$_1$	4
51	$C_8H_{24}N_4Pd^{2+}.4C_{12}H_4N_4.2C_2H_3N$	[Pd(CNMe)$_4$](tcnq)$_4$.2MeCN	Tri	P$\bar{1}$	1
309	$C_8H_{24}Re_2^{2-}.2C_4H_{10}LiO^+$	[Li(OEt$_2$)]$_2$[Re$_2$Me$_8$]	Tri	P$\bar{1}$	1
418	$C_8H_{26}B_6P_2Pt$	1,1-(Me$_3$P)$_2$-1,6,8-PtC$_2$B$_6$H$_8$	M	A2	2
300	$C_8I_2Mo_2O_8$	[MoI(CO)$_4$]$_2$	Tri	P$\bar{1}$	2
301	$C_8I_2O_8W_2$	[WI(CO)$_4$]$_2$			

[a] $C_5H_7N_2$ = η^4-1(1H),2(2H)-diazepinium. [b] $C_4H_8S_2$ = 1,3-dithiacyclohexane.

[c] $C_8H_{12}N_2$ = 1-allyl-3,5-dimethylpyrazole. [d] Cell data only. [e] $C_6H_{14}N_4$ = MeNH.N:CMeCMe:NNHMe.

[f] Diagram, bond distances only.

C_9

399	$C_9H_3O_{10}Re_3^{3-}.2C_8H_{20}N^+$	(NEt$_4$)$_2$[Re$_3$H$_3$O(CO)$_9$]	O	Pnma	4
407	$C_9H_6AsCoFeO_7$	FeCo(AsMe$_2$)(CO)$_7$	M	P2$_1$/m	2
212	C_9H_9Co	Co(C$_4$H$_4$)(C$_5$H$_5$)	O	Pnma	4

6.264(1)	17.510(4)	10.036(6)		96.18(2)		1285	5.1	6.2	46
4.839(3)	23.432(3)	10.342(3)		97.26(1)		1030	7.1		47
13.488(3)	9.058(2)	9.771(2)				857	5.6	7.5	48
6.972(4)	13.418(6)	13.400(5)		91.85(5)		1297	8.0	9.8	49
4.988(1)	21.670(1)	10.107(1)		97.47(1)		1651	5.9		47
8.442(4)	8.873(6)	8.112(3)	103.44(3)	116.42(3)	63.37(3)	1196	5.6		50
6.068(4)	10.670(9)	15.087(10)		97.96(3)		855	4.2	7.8	51
7.193	17.032	19.643						*d*	52
8.004(2)	10.344(3)	10.135(3)		97.69(4)		1455	5.8		53
8.460(3)	8.300(3)	11.700(4)		97.71(4)		1050	7.3		54
7.912(7)	15.821(9)	10.718(8)				712	3.5		55
18.62(1)	12.02(1)	6.306(6)				1113	5		56
7.730(2)	14.978(7)	14.389(4)	65.08(2)	81.29(2)	73.46(2)	2266	4.59	4.72	57
8.343(4)	10.436(3)	7.551(2)	106.91(2)	96.59(3)	69.47(3)	1370	4.8	6.4	58
5.716(2)	9.785(4)	15.329(8)		94.24(3)		698	7.9		59
9.44(2)	10.59(2)	9.52(2)	91.8(2)	123.0(1)	94.5(1)	1867	6.0		60
								f	61

15.05(2)	12.32(1)	18.35(2)				1147	3.8		62
9.090(9)	10.804(10)	7.593(6)		108.0(1)		602	5.1		63
10.462(2)	9.044(2)	7.672(3)				681	2.5	1.9	64

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C_{10}

361	$C_{10}H_3O_{10}Re_3^{2-}.2C_8H_{20}N^+$	$(NEt_4)_2[Re_3H_3(CO)_{10}]$		O	Cmcm	4
325	$C_{10}H_4Fe_2O_6$	$Fe_2(CO)_6(C_4H_4)$		M	$P2_1/c$	4
155	$C_{10}H_8FeO_5$	$Fe(CO)_3(C_7H_8O_2)$[a]		Tri	$P\bar{1}$	2
177	$C_{10}H_{10}Cl_2Ti$	$TiCl_2(C_5H_5)_2$		Tri	$P\bar{1}$	4
174	$C_{10}H_{10}Co$	$Co(C_5H_5)_2$				
173	$C_{10}H_{10}Fe^+.BiCl_4^-$	$[Fe(C_5H_5)_2]BiCl_4$		M	$P2_1/c$	4
152	$C_{10}H_{10}FeO_5$	$Fe(CO)_3[C(O)OC_4H_4Me_2]$		O	Pbca	8
179	$C_{10}H_{10}S_5Ti$	$Ti(S_5)(C_5H_5)_2$		M	$P2_1/c$	8
				O	$P2_12_12_1$	4
180	$C_{10}H_{10}S_5V.\frac{1}{2}H_2O$	$V(S_5)(C_5H_5)_2.\frac{1}{2}H_2O$		O	$P2_12_12_1$	4
140	$C_{10}H_{12}FeN_2O_4$	$Fe(CO)_3[CONMeC(NMe_2)CH{:}CH_2]$		M	B2/b	16
166	$C_{10}H_{12}FeO_4$	$Fe(CO)_3(C_7H_{12}O)$[b]		M	$P2_1/c$	8
36	$C_{10}H_{14}CrO_4S_2$	$Cr(CO)_4[EtS(CH_2)_2SEt]$		M	$P2_1/a$	4
129	$C_{10}H_{13}Cl_3O_2Pt^-.K^+$	$K[PtCl_3\{C_2[C(OH)MeEt]_2\}]$		M	$P2_1/c$	4
419	$C_{10}H_{30}B_6P_2Pt$	1,1-$(Me_3P)_2$-6,8-Me_2-1,6,8-$PtC_2B_6H_6$	α	M	A2/a	4
			β	M	$P2_1/c$	4
289	$C_{10}Br_2Mn_2O_{10}Sn$	$[Mn(CO)_5]_2SnBr_2$		M	$P2_1/c$	4
290	$C_{10}Cl_2Mn_2O_{10}Sn$	$[Mn(CO)_5]_2SnCl_2$		M	$P2_1/n$	4
401	$C_{10}F_{12}Fe_2O_5P_2$	$Fe_2(CO)_6[P(CF_3)_2]_2$		O	Pnma	4

[a] $C_7H_8O_2$ = η^4-3-methylene-*endo*-4-vinyldihydrofuran-2(3H)-one.

[b] $C_7H_{12}O$ = *trans*, *trans*-3,5-heptadiene-2-ol.

C_{11}

321	$C_{11}H_2Mn_2N_2O_{10}$	$Mn_2(CO)_{10}(CH_2N_2)$		M	$P2_1/c$	4
257	$C_{11}H_8CrO_5$	$Cr(CO)_3(PhCO_2Me)$		Tri	$C\bar{1}$	4
109	$C_{11}H_8FeO_5$	$Fe(CO)_4(C_7H_8O_2)$[a]		M	$P2_1/a$	4

9.96(1)	22.78(2)	15.16(2)				384	6.4			62
11.61(1)	6.40(1)	16.35(1)		107.6(2)		2056	5.8			65
7.745(5)	6.787(3)	9.873(4)	96.46(3)	86.79(4)	94.05(4)	1435	2.9	3.1		66
7.882(5)	19.478(10)	12.156(9)	90.46(2)	102.58(2)	143.49(2)	3742	3.2	2.3		67
									ED	14
10.988	17.449	7.569		98.46		1374	3	3.7		68
12.0732(16)	15.2272(22)	12.2010(15)				1434	3.21	3.06		69
22.843(2)	7.958(1)	14.465(1)		90.074(1)		1712	5.3	5.9		70
20.40	7.93	8.03								
13.491(1)	12.748(1)	7.715(1)				750	2.4	3.0		70
45.401(8)	15.246(3)	7.4071(5)			102.08(1)	1163	6.5			71
16.885(8)	17.831(3)	8.275(2)		111.91(1)		2791	4.2	3.3	241K	72
13.657(6)	14.168(5)	8.560(3)		123.48(2)		2453	4.0			73
10.721(10)	9.592(7)	16.440(8)		103.19(6)		2291	8.4	9.5		74
12.002(5)	10.415(7)	16.559(7)		102.47(3)		1077	7.0			59
11.892(4)	9.289(4)	18.724(8)		106.17(3)		2487	4.4	5.0		
15.275(6)	7.650(3)	16.855(6)		110.70(3)		1470	4.2			75
14.144(5)	12.223(4)	10.303(4)		100.09(2)		2107	3.7			75
20.261(2)	12.591(2)	7.422(1)				2167	6.88	5.09		76
10.526(3)	10.620(1)	14.295(7)		93.62(5)		2932	4.6			77
10.501(3)	11.488(11)	10.475(8)	107.82(6)	101.26(6)	101.33(7)	2008	3.4	3.5		78
12.419(4)	6.527(1)	15.321(4)		91.21(2)		1679	4.1	4.4		66

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12	$C_{11}H_{10}MoN_7O_3$	$Mo(CO)_2(NO)[HB(pz)_3]$	Trig	$P\bar{3}$	2
307	$C_{11}H_{10}NO_{12}W_2$	$W_2H(CO)_8(NO)[P(OMe)_3]$	Tri	$P\bar{1}$	2
21	$C_{11}H_{12}MoN_3O_5P$	$Mo(CO)_5[P(CH_2)_6N_3]$[b]	O	Pbca	8
79	$C_{11}H_{13}BN_6OPt$	$PtMe(CO)[HB(pz)_3]$	M	$P2_1/c$	4
154	$C_{11}H_{21}PPt$	$Pt(C_8H_{12})(PMe_3)$[c]	O	Pnam	4
135	$C_{11}H_{27}ClIrO_4P_3$	$IrCl(C_2O_4)(PMe_3)_3$	Tri	$P\bar{1}$	
370	$C_{11}O_{11}Rh_4^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2][Rh_4(CO)_{11}]$	Tri	$P\bar{1}$	2

[a] $C_7H_8O_2$ = η^2-3-methylene-*exo*-4-vinyldihydrofuran-2-(3H)-one.

[b] $P(CH_2)_6N_3$ = phosphatriazaadamantane. [c] C_8H_{12} = $(\eta^3\text{-}C_3H_4)(CH_2)_2CH{:}CHCH_2CH_2$.

C_{12}

408	$C_{12}H_3Co_3O_{11}$	$Co_3C(OCOMe)(CO)_9$	Tri	$P\bar{1}$	2
360	$C_{12}H_3Mn_3O_{12}$	$Mn_3H_3(CO)_{12}$	Tri	$P\bar{1}$	2
364	$C_{12}H_4O_{12}Re_4$	$Re_4H_4(CO)_{12}$	M	$P2_1/c$	4
98	$C_{12}H_6FeC_6$	$Fe(CO)_4(C_8H_6O_2)$[a]	M	$P2_1/c$	4
346	$C_{12}H_7FeO_5Rh$	$[Fe(CO)_3][Rh(CO)_2](C_7H_7)$	M	C2/c	8
254	$C_{12}H_8F_4V$	$V(C_6H_4F_2\text{-}p)$	M	I2/c	4
311	$C_{12}H_{10}Co_2O_2^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2]\{[Co(CO)(C_5H_5)]_2\}$	Tri	$P\bar{1}$	2
210	$C_{12}H_{10}F_2FeO$	$Fe(COCF_2C_5H_5)(C_5H_5)$	M	Cc	4
265	$C_{12}H_{10}MoO_3$	$Mo(CO)_3(C_9H_{10})$[b]	M	$P2_1/c$	4
178	$C_{12}H_{10}N_2S_2Ti$	$Ti(NCS)_2(C_5H_5)_2$	O	Imma	4
46	$C_{12}H_{11}NO_4SW$	*trans*-$W(CO)_4(CS)(CNCy)$	M	$P2_1/n$	n.g.
350	$C_{12}H_{12}Al_4Cl_{14}Pd_2$	$[PdAl_2Cl_7(C_6H_6)]_2$	Tri	$P\bar{1}$	1
53	$C_{12}H_{12}CrO_6S_2$	$Cr[C(OEt)[\overline{C(OH)CS(CH_2)_3S}]](CO)_4$	M	C2/c	8
99	$C_{12}H_{12}FeO_8$	$Fe(CO)_4[C_4H_6(CO_2Me)_2]$[c]	M	$P2_1/c$	4
353	$C_{12}H_{12}Fe_2N_2O_7$	$Fe_2(CO)_6(N{:}CMe_2)(ON{:}CMe_2)$	Tri	$P\bar{1}$	2

11.435(5)		8.054(4)				1572	4.7			79
10.012(7)	11.261(8)	9.062(6)	90.84(5)	90.52(6)	76.35(4)	2481	5.0	5.5		18
10.012(5)	11.249(5)	9.046(5)	90.96(4)	90.53(4)	76.24(2)	2385	8.9	10.7	ND	
14.357(2)	26.509(2)	8.1528(6)				1194	6.9			80
14.416(1)	7.951(1)	12.990(1)		104.35(1)		2124	4.1	3.9		81
15.979(15)	9.017(24)	8.992(7)				1291	6.4			82
16.142(6)	16.626(7)	9.335(14)	96.8(3)	98.3(3)	115.6(2)	3567	4.9			83
22.394(3)	14.351(2)	11.937(2)	93.05(2)	96.25(2)	88.38(2)	3884	6.0	6.9		84
13.997(10)	8.579(5)	8.117(7)	112.20(5)	102.16(5)	76.44(4)	1921	5.5			85
8.215(10)	14.864(19)	8.990(14)	105.22(10)	118.50(11)	96.95(9)	1537	5.8	7.0		86
14.449(3)	10.103(2)	13.860(3)		110.66(1)		1539	7.2			87
6.848(2)	32.396(8)	7.361(2)		128.91(2)		997	4.2	5.9		88
12.756(6)	15.044(9)	13.446(5)		99.62(1)		1672	2.7	2.6		89
13.850(6)	7.104(3)	11.192(4)		109.11(2)		1378	4.7	4.4		90
11.279(3)	17.593(5)	10.428(1)	101.59(3)	93.52(6)	91.69(2)	2871	7.3			91
12.204(3)	8.386(4)	11.262(3)		120.11(2)			2.8	3.8		92
7.4451(3)	13.5783(7)	11.2691(4)		92.593(3)		1754	3.1	5.1		93
13.58(1)	8.14(1)	11.81(1)				635	5.8			94
11.645(3)	21.205(6)	6.146(3)		92.10(5)		1862	6.5			95
9.42(3)	9.24(3)	9.56(3)	77.9(5)	96.2(5)	108.1(3)	1699	7.9		173K[c]	96
20.19(2)	7.22(1)	22.10(2)		106.9(5)		754	7.0			97
7.5605(9)	25.939(3)	7.4210(7)		82.60(1)		5074	3.71	4.31		98
12.061(10)	9.119(7)	8.599(7)	66.81(2)	99.94(3)	107.08(2)	1363	3.6	5.2		99

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348	$C_{12}H_{12}Ni_2$	$[Ni(C_5H_5)]_2(C_2H_2)$	O	Fm2m	4
153	$C_{12}H_{14}FeO_5$	$Fe(CO)_3[CH(CO_2Me)(CH_2)_2\overline{CMeCHCH_2}]$	O	Pbca	8
354	$C_{12}H_{14}Fe_2N_2O_7$	$Fe_2(CO)_6(NHCHMe_2)(ON{:}CMe_2)$	Tri	$P\bar{1}$	2
205	$C_{12}H_{16}Hf$	$HfMe_2(C_5H_5)_2$	M	$P2_1/c$	4
267	$C_{12}H_{16}MoO_3^+.BF_4^-$	$[Mo(acac)(OH_2)(C_7H_7)]BF_4$	M	$P2_1/n$	4
215	$C_{12}H_{17}AsMoO_2$	$Mo[(CH_2)_3AsMe_2](CO)_2(C_5H_5)$	O	Pcab	8
420	$C_{12}H_{17}B_5Co_2$	1,7,5,6-$[(C_5H_5)Co]_2C_2B_5H_7$	M	C2/c	8
421	$C_{12}H_{17}B_5Co_2$	1,8,5,6-$[(C_5H_5)Co]_2C_2B_5H_7$	M	C2/c	8
122	$C_{12}H_{18}Cl_2N_2Pt$	*trans*-$PtCl_2(C_2H_4)(CMe_2{:}NNMePh)$	M	$P2_1/c$	4
312	$C_{12}H_{18}N_6Pd_2^{2+}.2F_6P^-.\frac{1}{2}C_3H_6O$	$[Pd_2(CNMe)_6](PF_6)_2.\frac{1}{2}Me_2CO$	Tri	$P\bar{1}$	2
145	$C_{12}H_{20}Br_2Rh_2$	$[RhBr(C_3H_5)_2]_2$	M	$P2_1/c$	2
22	$C_{12}H_{21}MoO_4P_3$	$Mo(CO)_4[(Me_2P)_2PBu^t]$	M	$P2_1$	2
150	$C_{12}H_{26}N_6Pd_2$	$[Pd(C_4H_7)(N_3Me_2)]_2$	M	C2/c	8
87	$C_{12}H_{28}AuCl_2P_2$	$[AuCl[(CH_2)_2PEt_2]]_2$	Tri	$P\bar{1}$	1
369	$C_{12}Co_4O_{12}$	$Co_4(CO)_{12}$	M	C2/c	4
63	$C_{12}F_{11}O_5Re$	$Re[C[C(CF_3){:}CF_2]{:}C(CF_3)_2](CO)_5$	Tri	$P\bar{1}$	2

[a] $C_8H_6O_2$ = 2-methyl-3-prop-1-ynylmaleoyl. [b] C_9H_{10} = 5,6-dimethylenebicyclo[2.2.1]hept-2-ene.

[c] Cell dimensions at room temperature. [d] Ferracyclopentane derivative. [e] Redetermination.

C_{13}

367	$C_{13}HFe_4O_{13}^-.C_{10}H_{16}N^+$	$[NMe_3(CH_2Ph)][Fe_4H(CO)_{13}]$	O	$P2_12_12_1$	4
352	$C_{13}H_3Mn_3N_2O_{12}$	$[Mn(CO)_4]_3(N_2Me)$	M		4
365	$C_{13}H_4O_{13}Re_4^{2-}.2C_8H_{20}N^+$	$(NEt_4)_2[Re_4H_4(CO)_{13}]$	M	$P2_1/c$	4
209	$C_{13}H_5ClF_{12}W$	$WCl[C_2(CF_3)_2]_2(C_5H_5)$	Tri	$P\bar{1}$	2
327	$C_{13}H_7Fe_2O_6$	$Fe_2(CO)_6(C_7H_7)$[c]	O	Pbca	8
380	$C_{13}H_7NO_{10}Ru_3$	$Ru_3H(CO)_{10}(C{:}NMe_2)$	M	$P2_1/n$	8
61	$C_{13}H_3MnO_6^-.C_4H_{12}N^+$	$NMe_4[Mn(CO)_4(COMe)(COPh)]$	M	$P2_1/c$	4

17.209(8)	6.606(3)	9.311(5)				800	2.2		298K	21
17.195(14)	6.498(14)	9.162(8)				914	1.9		77K	
7.4525(7)	15.786(1)	21.020(2)				1727	3.33	3.68		100
12.008(5)	9.827(4)	8.580(4)	120.53(4)	99.29(1)	96.80(1)	2536	9.0			101
6.965(2)	11.857(4)	15.655(6)		118.38(2)		2408	3.6	4.5		102
8.664(9)	18.997(14)	9.623(13)		112.39(8)		3662	6			103
15.903(5)	13.297(2)	12.925(2)				2013	6.0	6.6		104
15.959(2)	7.846(2)	23.049(3)		102.33(2)		848	5.4	6.4		105
24.138(6)	7.803(2)	16.127(4)		111.26(4)		1648	5.3	7.1		105
9.488(8)	11.315(8)	14.726(9)		105.2		1476	6.6			36
12.281(2)	12.544(3)	10.388(2)	111.03(1)	111.12(1)	75.95(1)	3249	4.5	5.7		106
6.774(3)	7.387(3)	15.494(6)		102.87(8)		964	5.0	6.1		107
28.498(1)	9.6882(1)	13.805(1)		93.419(2)		5147	4.8			109
8.432(11)	12.751(14)	9.243(6)		107.51(8)		1417	5.4	5.0		108
7.296(9)	8.626(9)	7.945(8)	91.19(8)	102.53(8)	97.35(8)	455	8.7			110
8.995(4)	11.704(3)	17.260(4)				936	7.8	8.9	*e*	111
8.842(3)	11.950(5)	8.817(3)	92.80(4)	115.80(4)	90.70(4)	2000	9.9			112
24.62(2)	9.20(1)	12.57(1)				1350	5.7			113
9.891(2)	14.607(3)	13.196(5)		96.23(5)		1647	4.5			114
11.830(4)	20.847(6)	16.148(5)		92.12(4)		1282	7.6			115
7.942(1)	9.302(1)	12.828(2)	93.76(1)	104.76(1)	111.57(1)	2210	5.8	7.1		116
19.273(3)	13.858(1)	10.2347(7)				2147	2.95	3.5		117
9.3171(9)	35.8137(35)	11.9616(10)		97.06(1)		3702	2.41	3.45		13
10.346(5)	15.578(7)	12.094(3)		100.99(3)		1249	6.9	7.0		118

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127	$C_{13}H_{11}Cl_3N_2O_2Pt$	$PtCl_2(CH_2{:}CHC_6H_4NO_2)(ClC_5H_4N)$	M	$P2_1/c$	4
328	$C_{13}H_{12}Fe_2O_7S$	$Fe_2(CO)_6[CMe_2{:}C(O)C(S)Me_2]$	M	$P2_1/c$	1
268	$C_{13}H_{14}MoNO_2S$	$Mo(NCS)(acac)(C_7H_7)$	M	$P2_1/a$	4
207	$C_{13}H_{16}OZr$	$ZrMe(OCMe)(C_5H_5)_2$	O	Pnma	4
216	$C_{13}H_{17}MoN_2O_2$	$Mo(CO)_2(MeNCMeCNMe_2)(C_5H_5)$[b]	Tri	$P\bar{1}$	2
64	$C_{13}H_{23}CoN_6O_4$	$CoMe(Meim)(dmg)_2$	M	$P2_1/c$	4
432	$C_{13}H_{25}B_8CoFe$	$FeCo(C_5H_5)C_4B_8H_8Me_4$	M	$P2_1$	2
411	$C_{13}Co_4O_{13}Si$	$Co_3Si[Co(CO)_4](CO)_9$	Tri	$P\bar{1}$	2

[a] C_7H_7 = 1-(μ-carbeno)-2,3-η^2:4,5-η^2-cyclohexa-2,4-diene. [b] Ligand = an iminodimethylaminocarbene.

C_{14}

362	$C_{14}H_4N_2O_{10}Ru_3$	$Ru_3(CO)_{10}(C_4H_4N_2)$	Tri	$P\bar{1}$	2
343	$C_{14}H_6O_6Os_2$	$Os_2(CO)_6(C_8H_6)$	Tri	$P\bar{1}$	2
384	$C_{14}H_6O_{10}Os_3$	$Os_3(CO)_{10}(s\text{-}cis\text{-}C_4H_6)$	M	$P2_1/n$	
385	$C_{14}H_6O_{10}Os_3$	$Os_3(CO)_{10}(s\text{-}trans\text{-}C_4H_6)$	M	C2/c	
386	$C_{14}H_8O_{10}Os_3$	$Os_3H(CO)_{10}(C_4H_7)$	M	C2/c	8
310	$C_{14}H_{10}Fe_2O_2S_2$	$cis\text{-}[Fe(CO)(CS)(C_5H_5)]_2$	M	$P2_1/c$	4
329	$C_{14}H_{10}Fe_2O_7$	$Fe_2(CO)_6[CH_2CMeCEtCO]$	M	$P2_1/n$	4
181	$C_{14}H_{10}N_2S_2Ti$	$Ti[S_2C_2(CN)_2](C_5H_5)_2$	M	$P2_1/n$	4
35	$C_{14}H_{12}CrO_5S$	$Cr(CO)_5[S(Et)CH_2Ph]$	M	$P2_1/c$	4
93	$C_{14}H_{13}BrClN_2Pd^-.C_{16}H_{36}N^+$	$NBu_4[Pd(C_6H_4CMe{:}NNHPh)ClBr]$	M	$P2_1/c$	4
368	$C_{14}H_{13}O_{14}PRu_4$	$Ru_4H_4(CO)_{11}[P(OMe)_3]$	Tri	$P\bar{1}$	2
255	$C_{14}H_{14}Cr^+.C_{12}H_4N_4^-$	$[Cr(PhMe)_2](C_{12}H_4N_4)$	M	$P2_1/n$	4
256	$C_{14}H_{14}Cr^+.2C_{12}H_4N_4^-$	$[Cr(PhMe)_2](C_{12}H_4N_4)_2$	Tri	$P\bar{1}$	1
124	$C_{14}H_{15}Cl_2NPt$	$PtCl_2(CH_2{:}CHPh)(NC_5H_4Me)$	M	$P2_1/c$	4
281	$C_{14}H_{16}Ag^+.NO_3^-$	$AgNO_3(C_{14}H_{16})$[b]	M	$P2_1/c$	4
258	$C_{14}H_{16}CrO_3$	$Cr(CO)_2(Me_2C_6H_3CH_2OCH_2CH{:}CH_2)$	M	$P2_1/c$	4

12.831(3)	5.400(5)	22.874(6)		97.42(5)		3057	4.8	5.6		119
8.492(1)	15.737(1)	15.499(1)		125.129(5)		6568	3.9	5.7	123K	120
12.762(2)	12.634(4)	8.813(3)		100.70(2)		2788	7			103
12.397(1)	7.086(1)	14.051(1)				1039	3			121
8.296(4)	7.492(4)	13.945(8)	85.95(4)	72.01(3)	63.46(3)	1803	2.5	2.7		122
9.25(1)	11.77(1)	19.80(1)		124.4(1)		1801	5.1			123
7.203(4)	14.77(2)	8.830(2)		99.7(1)		1657	6.9	8.1	292K	124,1
15.697(3)	8.883(2)	8.131(1)	110.84(2)	105.30(1)	80.46(2)	2976	3.7			126
9.272(2)	13.727(2)	8.973(1)	103.44(1)	120.53(1)	90.01(1)	2304	6.1	9.1		127
7.973(4)	10.684(4)	9.191(3)	89.16(3)	99.28(3)	91.75(3)	2152	5.3	6.4		128
8.051(2)	14.778(3)	15.356(2)		94.60		2418	3.6			129
30.638(6)	9.770(2)	13.285(3)		112.82(2)		2473	3.6			129
20.30(1)	15.54(1)	18.19(2)		137.18(3)		1415	3.2	3.2		130
14.409(5)	12.560(4)	8.177(3)		90.3(2)		2000	7.7			131
9.354(3)	12.775(5)	13.840(5)		92.71(3)		1912	4.9	6.5		132
13.35(1)	6.88(1)	15.38(1)		105.6(1)		2233	4.4			94
9.70(1)	19.11(1)	9.92(1)		122.9(1)		1128	8.2			133
10.260(2)	15.984(4)	19.567(5)		96.48(2)		3123	3.8	3.8		134
13.69	9.13	11.26	116.0	93.2	97.1				α	87
7.00(2)	15.45(3)	20.50(6)		97.0(5)		1754	12.8			135
13.380(10)	8.126(8)	7.505(6)	112.23(8)	96.40(8)	92.07(8)	1718	6.2			136
13.928(6)	8.963(5)	12.084(6)		93.06(6)		2090	4.6	7.1		119
17.554(9)	6.908(4)	11.031(5)		103.10(3)		841	3.3	4.2		137
6.626(1)	12.140(3)	15.893(4)		98.12		1452	3.2			138

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252	$C_{14}H_{16}Fe$	$Fe(C_7H_7)(C_7H_9)$		M	$P2_1/c$	4
253	$C_{14}H_{16}Ru$	$Ru(C_7H_7)(C_7H_9)$		M	$P2_1/n$	4
272	$C_{14}H_{18}CrN_2$	$Cr(C_5H_3Me_2N)_2$	A	Tri	$P\bar{1}$	1
			B	O	Pbcn	4
262	$C_{14}H_{18}Ru$	$Ru(C_6H_6)(C_8H_{12})$		M	$P2_1/n$	4
–	$C_{14}H_{20}Fe_2S_4^+.F_6P^-$	$[Fe_2(S_2)(SEt)_2(C_5H_5)_2]PF_6$		M	$P2_1/m$	
400	$C_{14}H_{20}Fe_2S_4^+.F_6Sb^-$	$[Fe_2(S_2)(SEt)_2(C_5H_5)_2]SbF_6$		M	$P2_1/m$	
192	$C_{14}H_{22}AlY$	$Y(C_5H_5)_2Me_2AlMe_2$		O	$Pna2_1$	4
193	$C_{14}H_{22}AlYb$	$Yb(C_5H_5)_2Me_2AlMe_2$		O	$Pna2_1$	4
65	$C_{14}H_{22}CoN_5O_4$	$CoMe(py)(dmg)_2$		Tri	$P\bar{1}$	2
–	$C_{14}H_{23}N_3S_6Zr.CH_2Cl_2$	$Zr(S_2CNMe_2)_3(C_5H_5).CH_2Cl_2$		Tri		2
189	$C_{14}H_{23}N_3S_6Zr.C_6H_5Cl$	$Zr(S_2CNMe_2)_3(C_5H_5).C_6H_5Cl$		M	$P2_1/c$	4
69	$C_{14}H_{25}BF_2IN_4O_2Rh$	trans-$RhIMe[C_2(do)(doBF_2)]$[d]		M	$P2_1/n$	4
284	$C_{14}H_{27}MnO_5Si_4$	$Mn[Si(SiMe_3)_3](CO)_5$		Tri	$P\bar{1}$	2
287	$C_{14}H_{27}O_5ReSi_4$	$Re[Si(SiMe_3)_3](CO)_5$		Tri	$P\bar{1}$	2

[c] Preliminary results only. [b] $C_{14}H_{16}$ = Norbornadiene dimer. [c] Crystal data only.

[d] $C_2(do)(doBF_2)$ = difluoro[3,3'-(trimethylenedinitrilo)bis-(2-pentanoneoximato)]borate.

[e] Corrected cell constants (see ref. 148).

C_{15}

373	$C_{15}HO_{15}Os_5^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2][Os_5H(CO)_{15}]$		M	C2/c	8
366	$C_{15}H_4O_{15}Re_4^{2-}.2C_8H_{20}N^+$	$(NEt_4)_2[Re_4H_4(CO)_{15}]$	I	M	$P2_1/c$	4
			II	M	$P2_1/n$	4
			III	M	$P2_1/c$	4
323	$C_{15}H_5BrO_8Re_2$	$Re_2(CO)_8(CPh)Br$		M	$P2_1/c$	4
208	$C_{15}H_5F_{11}MoOS$	$MoO(SC_6F_5)[C_2(CF_3)_2](C_5H_5)$		O	Pbca	8
387	$C_{15}H_9O_9Os_3$	$Os_3H(CO)_9(C_6H_7)$[b]		M	$P2_1$	2
381	$C_{15}H_{10}O_9Ru_3$	$Ru_3H(CO)_9(C_6H_9)$[c]		Tri	$P\bar{1}$	2
227a	$C_{15}H_{11}MnO_2$	$Mn(CO)_2(C{:}CHPh)(C_5H_5)$		O	Pccn	8

10.991(1)	8.628(1)	12.594(1)		112.11(1)		1951	4.9			139
6.442(1)	17.758(2)	9.899(1)		97.72(2)		2040	3.5			140
7.307(1)	8.242(2)	6.316(2)	112.50(3)	104.01(2)	102.46(2)	1064	8.6	10.6	238K	141,142
9.421(1)	10.496(2)	12.725(2)				909	4.5	3.5	238K	
6.486(1)	11.883(2)	14.861(2)		91.43(2)		2032	3.4			143
6.711(3)	19.57(9)	7.87(4)		91.2(4)					*c*	144
6.777(3)	19.849(7)	7.909(3)		91.56(3)		2854	3.3			
17.969(6)	7.988(4)	10.870(4)				663	5.2			145
17.866(5)	7.973(3)	10.871(3)				1124	7.8			145
14.38(1)	10.02(1)	9.41(1)	56.3(1)	127.3(1)	106.6(1)	3083	6.4			123
9.975(2)	14.258(3)	9.627(2)	97.53(2)	110.16(1)	92.70(2)				*c*	146
11.582(2)	16.756(3)	14.016(2)		90.58(1)		7384	7.9	9.0		146
9.424(4)	17.36(1)	12.40(1)		94.18(5)		2977	3.1	3.8		147
9.002(2)	9.655(2)	15.639(3)	83.66(1)	105.65(1)	114.61(1)				*e*	49
9.131(2)	9.358(2)	15.931(3)	84.78(2)	105.46(2)	111.99(2)	1149	6.0	7.5		49

21.98(1)	15.76(1)	31.01(2)		99.3(1)		6001		5.9		149
11.355(2)	21.204(4)	17.416(3)		94.15(2)		3042	5.5	7.8		150
21.831(4)	17.584(3)	11.446(2)		96.02(2)		2870	6.3	7.5		
11.60(1)	20.68(2)	17.97(2)		95.90(10)					*a*	
13.309	10.839	18.171		134.6		956	5.7			152
25.130(6)	10.933(4)	13.210(4)				2251	5.0	5.7		153
8.477(2)	12.056(4)	9.013(3)		92.88(2)		2044	4.4			154
9.569(11)	11.231(7)	9.379(5)	95.09(1)	90.96(4)	99.75(1)	2890	3.5	4.4		155
10.492(2)	33.038(6)	7.475(2)				600	9.3			156

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308	$C_{15}H_{15}Mn_2N_3O_3$	$Mn_2(NO)_3(C_5H_5)_3$	O	Pnma	4
80	$C_{15}H_{22}BN_7Pt$	$PtMe(CNBu^t)[HB(pz)_3]$	Tri	$P\bar{1}$	2
58	$C_{15}H_{25}Cl_2NOPPt$	*cis*-$PtCl_2[C(OEt)(NHPh)](PEt_3)$	O	$P2_12_12_1$	4
33	$C_{15}H_{27}NiO_3P$	$Ni(CO)_3(PBu^t_3)$	M	$P2_1/a$	4
412	$C_{15}Co_6O_{14}^-.C_4H_{12}N^+$	$NMe_4[Co_6C(CO)_{14}]$	Tri	$P\bar{1}$	2

[a] Crystal data only: see ref. 151. [b] C_6H_7 = 1,2(Os^3):3(Os^2):4,5(Os^1)-η^5-cyclohexadienyl.

[c] Isomer A; C_6H_9 = μ_3-η-(1-methyl-3-ethyl-3-allenyl).

C_{16}

217	$C_{16}H_5F_{13}MoOS$	$Mo(CO)(SC_6F_5)[C_2(CF_3)_2](C_5H_5)$	Tri	$P\bar{1}$	2
322	$C_{16}H_5MoO_9Re$	$(OC)_5ReMo(CPh)(CO)_4$	M	C2/c	4
211	$C_{16}H_5F_{16}Fe$	$Fe[C_6HF(CF_3)_5](C_5H_5)$	M	C2/c	8
52	$C_{16}H_{11}CrO_5$	$Cr(CO)_5[C{:}C{:}CPh(NMe_2)]$	M	$P2_1/c$	4
259	$C_{16}H_{14}CrO_5$	$Cr(CO)_3(C_{13}H_{14}O_2)$[a]	M	$P2_1/a$	4
167	$C_{16}H_{14}Fe_2O_6$	$[Fe(CO)_3]_2C_{10}H_{14}$[b]			
330	$C_{16}H_{14}Fe_2O_7$	$Fe_2(CO)_6[CH_2CMeCCBu^tCO]$	M	$P2_1/n$	4
409	$C_{16}H_{15}BBr_2Co_3NO_{10}$	$Co_3(CO3Br_2NEt_3)(CO)_9$	Tri	$P\bar{1}$	2
147	$C_{16}H_{16}Ni_2$	$[Ni(C_8H_8)]_2$	M	$P2_1/a$	4
125	$C_{16}H_{18}Cl_2OPtS$	*cis*-$PtCl_2[(S)$-$MeC(O)S(C_6H_4Me$-$p)]$-$[(R)$-$PhCH{:}CH_2]$	O	$P2_12_12_1$	4
344	$C_{16}H_{18}Co_2O_6$	$Co_2(CO)_6(C_2Bu^t_2)$	Tri	$P\bar{1}$	2
331	$C_{16}H_{18}Fe_2O_6$	$Fe_2(CO)_6(C_2Bu^t_2)$	M	$P2_1/n$	4
126	$C_{16}H_{20}Cl_2N_2Pt$	$PtCl_2(CH_2{:}CHC_6H_4NMe_2)(NC_5H_4Me)$	Tri	$P\bar{1}$	2
332	$C_{16}H_{20}Fe_2O_4S_2$	$[Fe(SCHCEtCHMe)(CO)_2]_2$			
422	$C_{16}H_{21}B_5Ni_3$	$[Ni(C_5H_5)]_3CB_5H_6$	O	Pnma	4
142	$C_{16}H_{22}Cl_2Pd_2$	$[PdCl(C_8H_{11})]_2$[c]	Hex	$R\bar{3}$	9
269	$C_{16}H_{16}Nd^-.C_{16}H_{24}NdO_2^+$	$[Nd(C_8H_8)(thf)_2][Nd(C_8H_8)_2]$	M	$P2_1/c$	4

18.201(3)	10.802(2)	7.877(2)				928	3.1		157
9.352(2)	11.280(2)	10.710(2)	76.08(2)	94.00(1)	65.71(1)	3417	6.5	6.3	158
15.063(5)	14.102(5)	9.306(3)				1772	6.2	6.3	159
14.708(10)	15.594(10)	8.327(9)		105.02(5)		1266	12		160
12.17(1)	13.28(1)	8.67(1)	97.78(9)	89.93(9)	98.23(9)	1474	7.6		161
8.267(4)	13.548(5)	8.337(3)	99.16(3)	97.08(4)	86.61(4)	2722	3.8	4.3	153
10.844	17.127	10.424		91.2		973	5.1		162
23.70(1)	10.046(8)	15.41(1)		98.32(5)		1500	8.0		112
7.479	20.789	10.916		102.73		1869	6.7		163
21.898(7)	10.428(7)	6.911(4)		102.2(1)		1057	6.92	7.55	164
								[b]	165
10.025(3)	11.168(4)	16.482(7)		101.94(3)		1818	3.1	4.3	132
13.277(6)	10.17(1)	9.22(2)	91.12(6)	87.61(4)	98.79(2)	1969	10.7		166
8.2906(3)	11.4185(5)	7.2340(3)		115.978(3)		1055	4.56	7.95	167
11.633(4)	15.055(5)	9.776(3)				2414	2.92	2.68	168
8.394(4)	8.491(6)	13.825(6)	88.39(5)	94.73(5)	106.86(4)	2246	3.8	6.2	169
13.824(4)	9.776(3)	13.826(5)		94.26(3)		1767	4.9	6.9	169
5.144(1)	11.273(3)	15.657(4)	94.24(1)	95.44(2)	103.28(2)	2541	8.5	10.3	119
								[c]	170
7.518(1)	14.740(2)	15.711(3)				1840	3.16	3.93	171,172
27.155(7)		6.669(6)				731	4.8		173
16.664(3)	12.778(3)	14.347(4)		108.90(2)		1371	3.30		174

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103	$C_{15}H_{24}Pt$	$Pt[CH(Vi)(CH_2)_2CH(Vi)](C_8H_{12})$	M	$P2_1/n$	4
148	$C_{16}H_{26}Cl_2Pd_2$	$[PdCl(C_8H_{13})]_2$[e]	M	$P2_1/c$	2
128	$C_{16}H_{27}Cl_2NOPt$	*cis*-$PtCl_2(PhCHMeNH_2)(CH_2{:}CHOC_6H_{13})$	O	$P2_12_12_1$	4
27	$C_{16}H_{27}FeO_4P$	$Fe(CO)_4(PBu^t_3)$	O	$P2_12_12_1$	4
184	$C_{16}H_{26}N_2Si_2V$	$V[N_2(SiMe_3)_2](C_5H_5)_2$	O	Fdd2	8
91	$C_{16}H_{31}BrN_2Ni$	$NiBr(C_{16}H_{31}N_2)$[f]	M	$P2_1/a$	4
372	$C_{16}O_{16}Os_5$	$Os_5(CO)_{16}$	Trig	$P3_121$	3

[a] $C_{13}H_{14}O_2$ = *exo*-2-acetoxybenzonorbornenyl. [b] $C_{10}H_{14}$ = deca-1,3,7,9-tetraene; diagrams only of *meso* and *rac* forms. [c] Diagram, distances and bond angles only. [d] C_8H_{11} = cycloocta-1,4-dien-8-yl.

[e] C_8H_{13} = *exo*-η^3-2-methylene-6-methylcyclohexyl. [f] $C_{16}H_{31}N_2$ = 3,8-diisopropyl-2,9-dimethyl-4,7-diaza-4,6-decadienyl-*N*,*N*'.

C_{17}

171	$C_{17}H_{12}Br_2FeO_3$	$Fe(CO)_3(C_{14}H_{12}Br_2)$[a]	M	B2/b	8
379	$C_{17}H_{12}Fe_3O_8$	$Fe_3(CO)_8(C_5H_{12})$[b]	M	$P2_1/c$	4
261	$C_{17}H_{16}CrO_5$	$Cr(CO)_3[C_{10}H_4(OH)EtMe(OMe)]$	O	Pbca	8
232	$C_{17}H_{16}Fe_2O_4$	$[Fe(CO)_2(C_5H_5)]_2(CH_2)_3$	M	C2/c	4
195	$C_{17}H_{16}U$	$U(C_2H)(C_5H_5)_3$	M	$P2_1/n$	8
221	$C_{17}H_{17}F_3MoO_3$	$Mo[COC_4Me_4C(CF_3)O](CO)(C_5H_5)$	M	$P2_1/n$	8
168	$C_{17}H_{20}FeO_5$	$Fe(CO)_3(C_{14}H_{20}O_2)$[c]	M	$P2_1/c$	4
101	$C_{17}H_{21}F_6O_3Rh.\frac{1}{2}H_2O$	$Rh(OH_2)(acac)(C_8H_{12}C_4F_6).\frac{1}{2}H_2O$	M	C2/c	8
423	$C_{17}H_{23}B_5Ni_3$	*nido*-$[(C_5H_5)Ni]_3C_3H_5Me$	O	Fbnm	4
186	$2C_{17}H_{24}O_2Ti^+.H_{24}B_{20}Zn^{2-}.C_4H_8O$	$[Ti(OPr^i)(thf)(C_5H_5)_2]_2[Zn(B_{10}H_{12})_2].$-thf	M	$P2_1/a$	4
24	$C_{17}H_{27}O_5PW$	$W(CO)_5(PBu^t_3)$	M	$P2_1/n$	4
157	$C_{17}H_{39}Cl_2N_2Ru$	$RuCl_2(C_5H_{11}N)_2(C_7H_8)$	Tri	$P\bar{1}$	2

[a] $C_{14}H_{12}Br_2$ = 4,5,6,7-η^4-1-(*p*-bromobenzyl)-2-bromo-5-methylenecyclohexa-2,5-dien-4-yl.

[b] C_8H_{12} = 1,3,6-trimethylhexa-1,3,5-triene-1,5-diyl. [c] $C_{14}H_{20}O_2$ = 3-methoxy-1-methyl-5-(2-oxocyclohexyl)-cyclohexadiene.

9.082(6)	10.554(13)	15.293(4)		92.13(7)		1487	12		82
4.9997(8)	13.422(3)	13.796(4)		99.43(2)		1045	5.6		175
25.56(3)	11.37(1)	6.84(1)				1764	6.9	8.9	176
18.842(7)	10.062(4)	9.790(2)				1689	2.8	3.1	177
16.92(1)	21.40(1)	11.053(7)				1056	4.6		178
14.654(7)	11.333(6)	11.664(7)		92.51(4)		1556	9.0		179
9.204(2)		24.818(4)				809	3.9		180
31.539(9)	12.523(3)	9.417(2)			109.65(15)		6		181
8.971(6)	9.881(6)	21.570(9)		93.8(1)		2590	3.8		182
14.08(1)	16.22(1)	13.87(1)				1338	5.5		183
21.20(2)	10.39(1)	7.88(1)		101.65(5)		1036	5.9		184
18.047(9)	8.243(6)	19.428(9)		92.21(6)		1851	7.2	8.1	185
18.389(4)	11.137(3)	16.686(5)		92.55(2)		5411	8.6		186
6.769(1)	9.487(2)	26.054(3)		100.57(2)		1875	5.7	1.9	187
8.84(1)	19.34(2)	22.61(2)		102.8(5)		1591	9.2	9.4	188
7.518(1)	14.740(2)	15.711(3)				1840	3.16	3.93	172
22.920(9)	16.608(7)	14.360(6)		97.475(15)		4449	6.4		189
14.514(12)	16.439(6)	8.957(2)		102.16(5)		1627	6.3		160
14.864(4)	9.888(3)	6.695(2)	80.24(3)	102.31(2)	104.50(9)	6835	3.5	4.2	190

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C_{18}

376	$C_{18}HO_{18}Os_6^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2][Os_6H(CO)_{18}]$		Tri	$P\bar{1}$	2
374	$C_{18}HO_{18}Ru_6^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2][Ru_6H(CO)_{18}]$	I	Tri	$P\bar{1}$	4
			II	M	$P2_1/c$	12
377	$C_{18}H_2O_{18}Os_6.\frac{1}{2}CH_2Cl_2$	$Os_6H_2(CO)_{18}.\frac{1}{2}CH_2Cl_2$		M	$P2_1/c$	8
23	$C_{18}H_{12}As_2F_6MoO_4$	$Mo(CO)_4(bif_{12}fars)$[b]		O	Pbcn	4
429	$C_{18}H_{13}BMn_2O_6$	$[Mn(CO)_3]_2(C_4H_3EtBPh)$		O	Pbca	8
266	$C_{18}H_{14}Fe$	$Fe(C_5H_5)(\eta^6\text{-}C_{13}H_9)$		M	$P2_1/c$	8
390	$C_{18}H_{15}Co_3O_3$	$[Co(CO)(C_5H_5)]_3$		Tri	$P\bar{1}$	
355	$C_{18}H_{15}Fe_2NO_8$	$Fe_2(CO)_6[PhCHMeN{:}CH(CO_2Et)]$		M	$P2_1/c$	4
233	$C_{18}H_{18}Fe_2O_4$	$[Fe(CO)_2(C_5H_5)]_2(CH_2)_4$		M	$P2_1/c$	4
218	$C_{18}H_{19}MoNO_3$	$Mo(COCHPhCHMeNHMe)(CO)_2(C_5H_5)$		Tet	$P4_12_12$	8
78	$C_{18}H_{20}Cl_2N_2O_4Pd$	$PdCl(py)_2[CH(CO_2Me)CH(CO_2Me)CCl{:}CH_2]$		Tri	$P\bar{1}$	4
237	$C_{18}H_{20}O_2Ru$	$Ru(CO)_2(C_5H_4CMe_2)_2$		Tri	$P\bar{1}$	2
219	$C_{18}H_{23}MoNO_3$	$Mo(CO)(CNBu^t)[OCOC_2Me_2CMe](C_5H_5)$		M	$P2_1/a$	2
260	$C_{18}H_{24}CrO_3$	$Cr(CO)_3(C_6H_5CHBu^t_2)$		M	$P2_1/c$	4
275	$C_{18}H_{26}B_2Fe_2S$	$[Fe(C_5H_5)]_2[S(BMe)_2(CEt)_2]$		M	$P2_1/c$	4
112	$C_{18}H_{27}NNiO_8$	*rac*-$Ni(MeCN)[C_2H_2(CO_2Et)_2]_2$		Tri	$P\bar{1}$	2
430	$C_{18}H_{30}B_8Fe_2$	$[(C_5H_5)Fe]_2Me_4C_4B_8H_8$	(I)	M	$P2_1/n$	
431	$C_{18}H_{30}B_8Fe_2$	$[(C_5H_5)Fe]_2Me_4C_4B_8H_8$	(II)	M	C2/c	
104	$C_{18}H_{30}N_2Pt$	$Pt[CH(Vi)(CH_2)_2CH(Vi)](CNBu^t)_2$		M	$P2_1/n$	4
302	$C_{18}H_{46}N_4W_2$	$W_2Me_2(NEt_2)_4$		M	C2/c	4
396	$C_{18}Br_2Mn_4O_{18}Sn_2$	$Mn_2(CO)_8[\mu\text{-}SnBrMn(CO)_5]_2$		M	$P2_1/n$	2
375	$C_{18}O_{18}Os_5^{2-}.2C_{19}H_{18}P^+$	$(PMePh_3)_2[Os_6(CO)_{18}]$		Trig	$P\bar{3}1c$	2

[a] Refinement for Ru only. [b] $bif_{12}fars$ = 2,2'-bis(1-dimethylarsinohexafluorocyclopentenyl).

9.508	16.690	18.170	97.83	97.80	90.05	2928	6.7		191
18.083	19.101	19.238	117.70	78.13	97.05	9165	10		192
33.82	52.55	9.832		92.66		1480	23	a	192
16.607	15.564	22.303		90.97		3502	5.9		191
16.26(1)	11.55(1)	13.34(1)				715	10.1		193
14.29(1)	16.70(1)	15.41(1)				1113	5.7		194
8.944(3)	14.600(4)	20.201(5)		100.08(2)		2323	4.5		195
9.128(4)	11.691(5)	7.948(4)	105.45(4)	100.70(4)	91.21(3)	2738	9.4		196
8.164(5)	10.986(6)	23.780(8)		101.78(10)		2105	4.9		197
									183
7.63(1)	10.54(1)	21.87(2)		96.38(5)		1418	5.1		184
16.63(1)		14.49(1)					5.6		198
14.794(8)	11.599(8)	13.131(8)	89.53(5)	113.04(4)	91.50(5)	5397	7.5		199
11.862(6)	10.039(5)	6.934(3)	106.05(2)	87.52(2)	97.45(2)	2675	2.73		200
9.301(3)	22.161(6)	9.353(3)		102.76(2)		3412	5.8		186
8.321(3)	14.741(5)	14.369(5)		98.95(3)		2738	3.2		201
8.729(1)	16.800(2)	13.474(2)		72.20(1)		2151	5.9		202
9.219(6)	13.257(3)	10.521(9)	92.57(9)	109.25(10)	110.0(9)	3034	4.5		203
10.676(2)	14.009(5)	13.669(5)		93.97(3)		2796	5.1		204
16.338(3)	8.210(2)	31.547(12)		103.74(2)		2468	4.4		204
9.317(4)	12.284(12)	19.217(15)		99.16(5)		1100	10		82
17.033(5)	8.258(2)	18.817(4)		103.34(2)		2030	5.7	7.1	205
8.817	12.376	15.511		63.54		2601	2.7		206
12.045		24.767				739	5.7		191

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C_{19}

264	$C_{19}H_8CoF_{10}$	$Co(C_6F_5)_2(C_6H_5Me)$	O	Pnma	4
397	$C_{19}H_{10}Fe_2MnO_8P$	$[MnFe_2(CO)_8(C_5H_5)]PPh$	M	C2/c	8
324	$C_{19}H_{11}FeMnO_6$	$Fe(CO)_3\{Mn[C(O)CHPh](CO)_2(C_5H_5)\}$	M	$P2_1/c$	4
11	$C_{19}H_{15}Cl_2OPPt$	*cis*-$PtCl_2(CO)(PPh_3)$	Tri	$P\bar{1}$	2
225	$C_{19}H_{16}Mn_2O_4$	$[Mn(CO)_2(C_5H_5)]_2(C_5H_6)$	M	$P2_1/c$	4
169	$C_{19}H_{18}Fe_2O_6$	$[Fe(CO)_3(C_4H_5CH)]_2(CH_2)_3$	O	Pbcn	4
162	$C_{19}H_{20}O_8Pd$	$Pd[C_4(CO_2Me)_4](nbd)$	Tri	$P\bar{1}$	2
102	$C_{19}H_{24}IrNO_2$	$Ir(py)(acac)(C_3H_4)(C_6H_8)$	M	$P2_1/c$	4
196	$C_{19}H_{24}U$	$UBu(C_5H_5)_3$	O	$P2_12_12_1$	4
358	$C_{19}H_{29}ClCuIrN_3OP_2$	$IrCuCl(Me_2N_3)(CO)(PMe_2Ph)_2$	Tri	$P\bar{1}$	2
71	$C_{19}H_{36}BrIrOP_2$	$IrHBrPh(CO)(PEt_3)_2$	M	$P2_1/c$	4
413	$C_{19}Co_8O_{18}^{2-}.2C_{10}H_{16}N^+$	$[NMe_3(CH_2Ph)]_2[Co_8C(CO)_{18}]$	Tri	$P\bar{1}$	2

C_{20}

19	$C_{20}H_8N_8O_8Rh_4$	$Rh_4(CO)_8(Biim)_2$	O	Pbcn	4
410	$C_{20}H_{10}ClCo_3O_{10}Ti$	$Co_3[COTiCl(C_5H_5)_2](CO)_9$	M	$P2_1/n$	4
333	$C_{20}H_{10}F_{12}Fe_2O_2$	$Fe_2(CO)[C_4(CF_3)_4CO](C_5H_5)_2$	O	$P2_12_12_1$	4
398	$C_{20}H_{10}Fe_2MnO_9P$	$[(C_5H_5)(CO)_2MnP(Ph)Fe(CO)_4]Fe(CO)_3$			
388	$C_{20}H_{15}O_{10}Os_3P$	$Os_3H(CO)_{10}(\bar{C}H\overset{+}{C}H_2PMe_2Ph)$	M	$P2_1/n$	4
472	$C_{20}H_{18}Mo_2O_2$	$Mo_2(CO)_2(C_5H_5)_2(C_8H_8)$ (orange)	M	$P2_1/n$	4
473	$C_{20}H_{18}Mo_2O_2$	$Mo_2(CO)_2(C_5H_5)_2(C_8H_8)$ (purple)	M	$P2_1/c$	12
320	$C_{20}H_{20}Mo_2O_4$	$[Mo(CO)_2(C_5H_5)]_2(C_2Et_2)$	M	$P2_1/n$	4
199	$C_{20}H_{20}O_2Ti_2.C_4H_8O$	$[Ti(OH)(C_5H_5)]_2(C_{10}H_8).thf$	O	$Pna2_1$	

11.465(9)	16.025(12)	9.503(8)				1335	6.1	5.8	207
15.79(2)	15.92(2)	17.00(3)		97.2(1)		1717	6.6		208
10.344(1)	7.9855(3)	21.796(2)		102.924(9)		1165	7.0	5.3	209
10.4822(9)	9.5929(7)	11.0065(8)	97.57(1)	117.96(1)	93.80(1)	4198	3.7	4.1	210
12.26(1)	11.44(1)	12.31(1)		102.30(8)		1277	4.4		211
12.854	13.248	11.646				1151	16		212
10.560(2)	10.583(2)	9.710(2)	99.33(1)	113.24(1)	85.53(2)	3646	2.7	3.9	213
9.248(1)	18.547(5)	10.853(1)		96.53(1)			7.1		214
8.64(1)	22.69(2)	8.66(1)				1290	4.9		215
9.024(1)	10.842(2)	13.073(2)	107.79(2)	91.87(1)	96.57(2)	3378	6.2		216
12.43(1)	12.68(1)	15.37(4)		98.18(10)		2798	3.9		217
19.02(2)	10.37(1)	12.81(1)	116.43(9)	88.90(9)	95.53(9)	3560	8.9		161
15.034(3)	8.257(1)	20.891(4)				1771	3.2	3.8	218
13.532(6)	22.939(10)	7.760(8)		90.67(4)		2187	10.3		217
9.446(3)	13.708(6)	15.748(6)					4	4.6	92
						1135	5.8		220
11.3389(18)	16.4265(25)	13.8840(20)		100.64(1)		3342	3.61	3.31	221
8.150(3)	11.978(9)	17.529(10)		92.28(4)		2095	8.4		224
8.629(2)	39.580(10)	15.759(7)		103.90		4164	7.9		224
9.262(2)	13.765(4)	15.554(3)		105.45(2)			3.6	5.7	225
10.143(6)	23.571(14)	9.009(8)				1122	11.0	8.8	226

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185	$C_{20}H_{22}Cl_2OTi$	$TiCl(OC_5H_4Cl)(C_5H_3MePr^i)(C_5H_5)$	M	$P2_1/c$	
248	$C_{20}H_{22}Fe$	$Fe[C_5H_3(C_5H_8)_2C_5H_3]$[a]	M	$C2/c$	4
175	$C_{20}H_{22}Ni$	$Ni(C_{10}H_{11})_2$[b]	M	$C2/c$	4
83	$C_{20}H_{26}Cl_2P_2Pt$	*trans*-$Pt(CCl{:}CH_2)_2(PMe_2Ph)_2$	M	$P2_1/c$	2
263	$C_{20}H_{26}Ru$	$Ru(\eta^4\text{-}C_8H_9)(C_6Me_6)$	M	$P2_1/n$	4
471	$C_{20}H_{30}F_6O_6P_3Rh_2^+.F_6P^-$	$[(C_5Me_5)Rh(PO_2F_2)_3Rh(C_5Me_5)]PF_6$	M	$P2_1$	6
105	$C_{20}H_{30}I_2P_2Pt$	$Pt(C_4H_8)I_2(PMe_2Ph)_2$	M	$C2/c$	4
144	$C_{20}H_{31}ClMoN_2O_2$	$MoCl(CO)_2(CyN{:}CHCH{:}NCy)(C_4H_7)$	O	$Pnam$	4
172	$C_{20}H_{32}Br_2NiS_2$	$NiBr_2(C_{20}H_{32}S_2)$[c]	O	$P2_12_12_1$	4
203	$C_{20}H_{33}Al_2Zr^+.C_5H_5^-$	$\{Zr[CH_2CH(AlEt_2)_2](C_5H_5)_2\}(C_5H_5)$			
66	$C_{20}H_{33}CoN_4O_9$	Cobaloxime (*SS*)	Tri	$P\bar{1}$	2
156	$C_{20}H_{42}Cl_2N_2Ru$	$RuCl_2(cod)(NH_2C_5H_{13})$	M	$C2/c$	4
394	$C_{20}Br_2In_2Mn_4O_{20}$	$\{[Mn(CO)_5]_2In(\mu\text{-}Br)\}_2$	Tri	$P\bar{1}$	1
291	$C_{20}Br_2Mn_4O_{20}Sn_2$	$[Mn(CO)_5]_4Sn_2Br_2$	M	$P2_1/c$	4
393	$C_{20}Cl_2In_2Mn_4O_{20}$	$\{[Mn(CO)_5]_2In(\mu\text{-}Cl)\}_2$	Tri	$P\bar{1}$	1
395	$C_{20}I_2In_2Mn_4O_{20}$	$\{[Mn(CO)_5]_2In(\mu\text{-}I)\}_2$	Tri	$P\bar{1}$	1

[a] *syn*-Bis(cyclopentyl-1",3"-ene)-(1,1')(3,3')-ferrocenophane.

[b] $C_{10}H_{11}$ = η^5-tricyclo[5.2.1.$0^{2,6}$]deca-2,4-dien-6-yl. [c] $C_{20}H_{32}S_2$ = 1-9:2-8-η-3,3,7,7,10,10,14,14-octamethyl-5,12-dithiatricyclo[7.5.0.$0^{2,8}$]tetradeca-1(9),2(8)-diene. [d] Diagram only.

C_{21}

405	$C_{21}H_{10}As_2Fe_3O_9$	$Fe_3(AsPh)_2(CO)_9$	M	$P2_1/c$	4
345	$C_{21}H_{14}Co_2O_7$	$Co_2(CO)_5(C_{16}H_{14}O_2)$[a]	Tri	$P\bar{1}$	2
227	$C_{21}H_{15}MnO_3$	$Mn(CO)_2(Ph_2C{:}C{:}O)(C_5H_5)$	M	$P2_1/c$	4
319	$C_{21}H_{18}O_6W_2$	$W_2(CO)_6(C_{15}H_{18})$[b]	M	$P2_1/n$	
138	$C_{21}H_{23}ClNNiP$	$NiCl(CH_2NMe_2)(PPh_3)$	M	$P2_1/c$	4
222	$C_{21}H_{30}O_2P_2W$	$W[C(p\text{-}tol){:}C{:}O](CO)(PMe_3)_2(C_5H_5)$	M	$P2_1/c$	4

14.50(2)	13.03(2)	19.38(3)		98.4(4)		1840	9.5			227
20.119(5)	5.974(1)	14.456(9)		125.47(2)		2741	5.9	4.4		222,223
18.898(4)	7.578(3)	10.967(4)		109.5(1)		1621	5.6	7.4		228
9.039(2)	6.322(1)	19.415(4)		91.91(2)		3250	6.3	7.2		229
8.846	18.062	10.818		91.5		1931	5.4			230
10.186(5)	37.68(2)	12.82(2)		98.06(1)		4210	11.2			231
15.286(6)	9.709(4)	17.036(6)		107.49(5)		1575	3.5	4.4		232
12.796(2)	8.234(1)	19.631(2)				1248	4.4			233
14.976(6)	15.154(4)	10.028(3)				2441	5.1			234
							3		*d*	235
11.090	13.772	8.971	99.75	119.49	84.83	2269	5.6			236
24.804(5)	7.760(2)	12.318(3)		99.71(2)		2802	4.7	5.5		190
10.634(4)	10.396(8)	8.823(1)	87.79(2)	95.33(1)	124.32(1)	3707	3.9			237
16.591(5)	12.455(2)	17.214(8)		108.10(3)		4396	4.8	5.3		238
10.622(3)	10.393(3)	8.722(3)	87.56(2)	95.59(2)	124.58(2)	4038	5.6			237
10.649(2)	10.457(2)	9.073(1)	87.67(1)	95.07(1)	123.79(1)	3597	4.4			237
15.62(1)	15.44(1)	14.58(1)		135.70(8)		1539	6.5			239
9.41	9.98	11.90	97.3	100.9	108.3	946	1.97			240
6.78(2)	13.97(2)	18.63(2)		99.55		686	8.3			241
11.767(2)	16.404(3)	10.879(1)		100.07(1)		2996	3.6	5.6		242
9.695(3)	14.749(3)	14.276(3)		101.42(4)		2243	4.1	4.5		243
12.725	14.775	15.224		129.6		1609	5			244

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146	$C_{21}H_{31}BrNiP$	$NiBr(C_{10}H_{14})(PMeBu^tPh)$[c]	O	$P2_12_12_1$	4

[a] $C_{16}H_{14}O_2$ = $\overline{CH{:}CPhC(O)OC{:}CHCMeCHCMe}$. [b] $C_{15}H_{18}$ = guaiazulene. [c] $C_{10}H_{14}$ = (+)-(1*R*,5*R*)-3,2,10-η^3-pinenyl.

C_{22}

334	$C_{22}H_{16}Fe_2O_6$	$Fe_2(CO)_6(C_{16}H_{16})$	O	$Pbc2_1$	4
228	$C_{22}H_{16}Mn_2O_4$	$[Mn(CO)_2(C_5H_5)]_2(C{:}CHPh)$	O	$P2_12_12_1$	4
249	$C_{22}H_{18}FeSi$	$Fe(C_5H_4SiPh_2C_5H_4)$	O	Pnma	4
176	$C_{22}H_{20}Cl_5U_2^-.C_8H_{16}LiO_2^+$	$[Li(thf)_2][U_2Cl_5[CH_2(C_5H_4)_2]_2]$	O	$P2_12_12_1$	4
182	$C_{22}H_{20}S_2Ti$	$Ti(SPh)_2(C_5H_5)_2$	M	C2/c	8
183	$C_{22}H_{20}S_2V$	$V(SPh)_2(C_5H_5)_2$	M	C2/c	8
306	$C_{22}H_{25}B_2Mo_2N_{12}O_4$	$Mo_2\{HB(pz)_3\}_2(O_2CMe)_2$	M	$P2_1$	2
206	$C_{22}H_{26}Hf_2O$	$[HfMe(C_5H_5)_2]_2O$	Trig	$P3_221$	3
214	$C_{22}H_{26}NPr$	$Pr(CNCy)(C_5H_5)_3$	M	$P2_1/c$	4
194	$C_{22}H_{26}Yb_2$	$[YbMe(C_5H_5)_2]_2$	M	$P2_1/n$	2
158	$C_{22}H_{28}N_4Rh_2$	$[Rh(cod)]_2(Biim)$	M	$P2_1/c$	4
298	$C_{22}H_{34}ClNO_3P_2PtSi$	*trans*-$PtCl[Si(OCH_2CH_2)_3N](PMe_2Ph)_2$	M	$P2_1/c$	4
247	$C_{22}H_{34}Fe$	$Fe(C_5H_4Bu^t)(C_5H_3Bu^t_2)$	O	$P2_12_12_1$	4
116	$C_{22}H_{46}ClP_2Rh$	$RhCl[Bu^t_2P(CH_2)_2CH{:}CH(CH_2)_2PBu^t_2]$	M	$P2_1/c$	4
303	$C_{22}H_{46}N_4O_8W_2$	$W_2Me_2(O_2CNEt_2)_4$	M	$P2_1/c$	4

C_{23}

220	$C_{23}H_{11}F_{12}MoN_3O_2$	$Mo(CO)_2(C_5H_5)\{[(CH_3)_2C_6H_3]_2N_3\}$	O	Pbca	8
13	$C_{23}H_{22}FeN_4O.\frac{1}{2}C_7H_8$	$Fe(CO)(C_{22}H_{22}N_4).\frac{1}{2}PhMe$[c]	Tri	$P\bar{1}$	2
81	$C_{23}H_{23}ClNPPt$	$PtCl(C_3H_5)(CNMe)(PPh_3)$	Tri	$P\bar{1}$	2
197	$C_{23}H_{24}U$	$U(CH_2C_6H_4Me\text{-}p)(C_5H_5)_3$	O	Pnma	4

10.813(4)	11.449(4)	17.341(9)				1830	442	5.64	245
6.368(1)	22.888(4)	13.185(2)				1538	5.3		246
15.336(3)	15.391(3)	7.998(1)				1200	7.1		247
14.18(2)	12.54(2)	9.28(1)				588	6.8	9.0	248
11.510(3)	15.357(4)	19.036(5)				1731	6.0		249
31.76(10)	7.97(2)	15.45(5)		104.87(17)		1048	10.7	11.7	250
31.454(5)	7.835(1)	15.545(2)		104.35(1)		1793	6.8	8.1	250
11.239(9)	13.698(6)	9.681(7)		99.34(6)		1482	6.4	6.7	251
8.011(2)		28.300(8)				1103	3.3	3.2	102
8.298(3)	21.66(1)	11.943(4)		104.98(3)		2254	6.4		252
10.656(5)	7.535(4)	13.216(5)		112.62(4)		1435	6.3		253
9.842(2)	14.590(3)	13.929(3)		90.45(2)		2531	3.2	3.9	254
6.630(4)	17.465(6)	22.297(6)		97.39(2)		2165	4.8		255
12.363(3)	10.342(2)	15.853(2)				805	9.8		256
20.783(12)	8.580(4)	14.799(9)		100.70(2)		1417	7.1		257
16.014(4)	10.433(4)	18.983(5)		107.51(2)		2671	4.0	5.4	258
8.826(1)	33.808(2)	17.006(1)				3405	10.4		259
8.6254(19)	13.0137(31)	10.9199(32)	102.32(2)	104.39(2)	78.60(2)	3488	4.9	5.3	260
13.38(1)	9.626(8)	9.188(8)	99.8(1)	81.8(1)	107.3(1)	2166	5.5		261
19.64(2)	11.81(1)	8.19(1)				1148	4.1		215

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14	$C_{23}H_{26}FeN_6O$	$Fe(CO)(N_2H_4)(C_{22}H_{24}N_4)$[a]	M	$P2_1/c$	4
198	$C_{23}H_{29}N_2Ti$	$Ti(C_6H_4CH_2NMe_2\text{-}o)_2(C_5H_5)$	M	C2/c	4

[a] $C_{22}H_{22}N_4$ = 7,16-dihydro-6,8,15,17-tetramethyldibenzo[*b*,*i*][1,4,8,11]tetraazacyclodecinato.

C_{24}

96	$C_{24}H_{18}Cl_2N_4Pt_2$	$[\overline{PtCl(C_6H_4N:NPh)}]_2$	M	C2/c	4
188	$C_{24}H_{18}N_2O_8Ti$	$Ti(OCOC_6H_4NO_2\text{-}p)_2(C_5H_5)_2$	Tri	$P\bar{1}$	2
273	$C_{24}H_{21}CrF_2O_3P$	$Cr(CO)_3(C_5H_2Bu^tPh_2PF_2)$	M	$P2_1/a$	4
37	$C_{24}H_{21}MnNO_3PS$	$Mn(CO)_3(CSNMe_2)(PPh_3)$	Tri	$P\bar{1}$	2
38	$C_{24}H_{21}MnNO_3PS_2$	$Mn(S_2CNMe_2)(CO)_3(PPh_3)$	M	C2/c	16
250	$C_{24}H_{22}Fe_2O_4$	$[Fe(C_5H_4CO_2Me)(C_5H_4)]_2$	M	$P2_1/n$	2
316	$C_{24}H_{24}Cr_2$	$Cr_2(C_8H_8)_3$	M	$P2_1$	2
39	$C_{24}H_{24}MnNO_3PS^+.BF_4$	$\{Mn(CO)_3[C(SMe)NMe_2](PPh_3)\}BF_4$	M	$P2_1$	2
315	$C_{24}H_{27}Ti_2O.C_4H_8O$	$(C_5H_5)_2\overline{Ti(\eta^1:\eta^5\text{-}C_5H_4)Ti}(C_5H_5)(thf).thf$	M	I2/a	8
159	$C_{24}H_{30}Rh_2$	$Rh_2(C_8H_9)_2(cod)$	M	$P2_1/c$	4
18	$C_{24}H_{31}N_2O_2RhS$	$Rh(CO)_2[(Nmes)_2SBu^t]$	Tri	$P\bar{1}$	2
305	$C_{24}H_{32}B_2Mo_2N_8O_4.CS_2$	$Mo_2[Et_2B(pz)_3]_2(O_2CMe)_2.CS_2$	Tri	$P\bar{1}$	2
76	$C_{24}H_{32}N_2O_6Pd$	$Pd[CH(COMe)CO_2Et]_2(2\text{-}Mepy)_2$	M	C2/c	8
84	$C_{24}H_{36}O_2P_2Pt$	*trans*-$Pt(CH:CHCH_2OMe)_2(PMe_2Ph)_2$	M	$P2_1/a$	2
277	$C_{24}H_{40}CoLiO_2$	$Co(cod)_2Li(thf)_2$	M	C2/c	
89	$C_{24}H_{64}P_8Ru_2$	$\{RuH[CH_2PMe(CH_2)_2PMe_2](dmpe)\}_2$	Tri	$P\bar{1}$	2
304	$C_{24}H_{56}Si_6W_2$	$W_2(CH_2SiMe_3)_6$	M	$P2_1$	4

C_{25}

143	$C_{25}H_{20}O_4PV$	$V(CO)_4(PPh_3)(C_3H_5)$	M	$P2_1/c$	8
110	$C_{25}H_{21}FeO_5P$	$Fe(CO)_3(PPh_3)(CH_2:CHCO_2Me)$	Tri	$P\bar{1}$	2
163	$C_{25}H_{23}MnNO_2P$	$Mn(CO)(PPh_3)(NO)(C_5H_5Me\text{-}exo)$	O	Pbca	8
283	$C_{25}H_{24}MnO_4PSi$	$Mn(SiMe_3)(CO)_4(PPh_3)$	Tri	$P\bar{1}$	2

9.422(2)	11.015(1)	21.264(4)		106.58(1)		2802	7.19	5.59	260
16.143(5)	7.889(3)	16.347(4)		100.63(6)		1382	11		262
27.07(3)	4.087(3)	20.91(2)		106.46(8)		1670	5.2		263
12.96(2)	11.53(2)	7.62(1)	84.3(5)	95.2(5)	90.5(5)	1620	14		264
15.092	12.442	13.119		115.08			5.9		265
9.605(11)	11.005(12)	11.516(12)	77.71(6)	97.83(6)	97.62(7)	3884	8.9	8.1	266
56.14(11)	8.778(9)	19.75(3)		102.05(6)		2756	8.3	8.9	267
10.65(2)	15.88(3)	5.90(2)		90(1)		652	13.4		268
10.485(3)	11.819(4)	7.571(2)		99.64(2)		2358	2.8	3.2	269
9.581(12)	16.559(9)	8.727(15)		101.81(6)		1813	9.1	9.2	266
28.567(19)	8.061(2)	19.691(7)		101.11(2)		2403	5.1	6.2	270
16.54(1)	15.08(2)	12.91(2)		77.05(8)		2105	4.97		271
8.439(1)	8.605(1)	19.110(3)	79.41(2)	83.90(2)	63.41(2)	2484	9.3		272
12.933(4)	13.088(4)	11.592(4)	101.68(3)	112.86(3)	69.69(2)	4841	6.1	9.9	251
23.811(5)	9.222(2)	24.013(5)		104.51(2)		1896	6.6		273
23.26(2)	6.16(1)	9.28(1)		107.5(2)		1652	6.89		274
14.094(1)	18.799(3)	17.500(3)		97.74(1)		1734	6.4		275
10.028(4)	13.880(6)	15.018(6)	95.87(3)	101.27(3)	112.02(3)	3870	7.2		276
12.890(1)	18.546(2)	18.347(2)		109.829(8)		3136	6.6	8.3	277
8.627(1)	16.294(3)	32.451(31)		90.09(2)		3700	6.2		278
8.006(3)	9.942(5)	14.876(7)	101.95(5)	85.29(4)	99.06(4)	4547	4.18	4.75	279
17.969(53)	26.631(78)	9.277(35)				2269	5.7	9.6	280
12.510(2)	11.503(2)	10.115(2)	117.62(2)	101.72(2)	91.90(2)	1537	7.0	9.3	281

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141	$C_{25}H_{25}IrN_4O$	$Ir(fn)(C_8H_{12}OMe)(phen)$	Tri	$P\bar{1}$	2
77	$C_{25}H_{25}O_4PPd$	$Pd(CH_2CO_2H)(acac)(PPh_3)$	Tri	$P\bar{1}$	2
382	$C_{25}H_{30}O_8Ru_3Si_3$	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]^a$	M	$P2_1/c$	4
383	$C_{25}H_{30}O_8Ru_3Si_3$	$Ru_3(CO)_8[C_8H_3(SiMe_3)_3]^b$	M	$P2_1/n$	4

[a] Edge-bonded hydrocarbon. [b] Face-bonded hydrocarbon.

C_{26}

389	$C_{26}H_{12}O_{10}Os_3$	$Os_3(CO)_9[(HC_2Ph)_2CO]^a$	O	$P2_12_12_1$	4
335	$C_{26}H_{14}Fe_2O_8$	$Fe_2(CO)_8(C_5H_4CPh_2)$	M	C2/c	4
191	$C_{26}H_{18}Cl_2Zr$	$ZrCl_2(\eta^3\text{-}C_{13}H_9)(\eta^5\text{-}C_{13}H_9)$	M	$P2_1/n$	4
41	$C_{26}H_{20}N_4O_2RuS_4$	$Ru(C_7H_5NS_2)_2(CO)_2(py)_2$	M	C2/c	
190	$C_{26}H_{20}O_6Zr.CH_2Cl_2$	$Zr(trop)_3(C_5H_5).CH_2Cl_2$	Tri	$P\bar{1}$	2
230	$C_{26}H_{22}As_2Mn_2O_4$	*meso*-$[Mn(CO)_2(C_5H_5)]_2As_2H_2Ph_2$	M	$P2_1/c$	2
229	$C_{26}H_{22}Mn_2O_4P_2$	$[Mn(CO)_2(C_5H_5)]_2P_2H_2Ph_2$	M	$P2_1/c$	2
67	$C_{26}H_{27}CoN_{10}O_4$	$Co\{\overbrace{CHCH_2[C_2(CN)_4]CHPh}\}(imH)(dmg)_2$			
317	$C_{26}H_{33}Mo_2O_5P$	$Mo_2(CO)_5(PEt_3)(C_{15}H_{18})^d$ (dark red)	M	$P2_1/c$	
318	$C_{26}H_{33}Mo_2O_5P$	$Mo_2(CO)_5(PEt_3)(C_{15}H_{18})^d$ (black)	M	$P2_1/a$	
251	$C_{26}H_{34}Al_4Cl_2Fe_2$	$[(C_5H_5)Fe(C_5H_3)Al_2ClMe_3]_2$	Tri	$P\bar{1}$	1
278	$C_{26}H_{39}CoLi_2O$	$(cod)_2CoLi(LiPh)(OEt_2)$	Tri	$P\bar{1}$	2
234	$C_{26}H_{39}FeN_3O$	$Fe(CO)[(C{:}NCy)(CNHCy)CH(CNHBu^t)]\text{-}(C_5H_5)$	M	$P2_1/c$	4
149	$C_{26}H_{42}Cl_2O_4Pd_2$	$\{PdCl[C_3H_3(COBu^t)_2]\}_2$	M	$P2_1/b$	2
94	$C_{26}H_{43}ClN_2P_2Pd$	*trans*-$\overbrace{PdCl(C_6H_4CMe{:}NNHPh)}(PEt_3)_2$	M	P2/c	8
97	$C_{26}H_{56}Cl_2P_2Pt_2$	$[PtCl(CH_2CMe_2CH_2PBu^t_2)]_2$	Tri	$P\bar{1}$	1

[a] $(HC_2Ph)_2CO$ = 4,5,6-η^3-2,5-diphenyl-3-oxahexa-1,5-diene-1,4,6-triyl. [b] Not yet refined.
[c] No crystal data given. [d] $C_{15}H_{18}$ = guaiazulene. [e] Contains disordered CH_2Cl_2 molecules (not refined).

9.848(6)	14.25(1)	17.93(1)	36.28(4)	128.5(1)	125.0(1)	1921	7.8			282
10.433(2)	12.746(2)	10.224(2)	106.62(1)	112.41(2)	75.13(2)	4973	3.1	4.1		283
16.264(13)	9.023(6)	22.769(16)		97.95(6)		8013	4.0		160K	284
11.490(2)	18.646(6)	15.381(17)		91.04(3)		3795	3.8			284
20.10(1)	15.30(1)	8.57(1)				2587	8.6	8.8		285
22.995(16)	10.096(6)	12.682(8)		124.86(3)		2053	3.67			286
12.347	13.771	11.922		98.2		2697	5.3			287
22.38(3)	9.32(1)	16.77(2)		129.21(8)		2400	*b*			288
9.83	12.44	13.88	103.7	94.2	128.4	3453	9.5			289
10.58(1)	11.48(1)	12.00(1)		120.85(8)		1040	4.2			290
10.95(1)	11.45(1)	11.86(1)		124.73(5)		1113	4.5			291
						2973	8.2		*c*	292
16.151(10)	12.336(8)	15.558(9)		104.27(6)		3972	11.1	15.5	*e*	293
14.379(11)	14.569(7)	13.034(6)		94.67(5)		2903	7.1	9.9		293
8.44(1)	9.07(1)	11.38(1)	78.61(6)	68.92(6)	62.44(6)	1027	12			294
11.020(5)	11.417(3)	14.532(4)	71.50(2)	79.85(3)	58.44(3)	2042	6.8			295
11.732(5)	10.380(5)	22.831(7)		112.97(3)		3276	5.05	4.00		296
12.310(2)	10.932(2)	12.357(1)			112.71(1)	2595	3.4			297
15.732(4)	18.746(5)	20.285(5)		106.29(2)		2444	3.4	3.9		134
8.411(4)	10.948(7)	9.106(4)	102.86(4)	90.64(4)	97.95(5)	1259	4.3	5.2		298

C_{27}

392	$C_{27}H_{10}As_2Cr_3O_{15}$	$[Cr(CO)_5]_3As_2Ph_2$			
336	$C_{27}H_{19}Fe_2O_5P$	$Fe_2(CO)_5(PPh_3)(C_4H_4)$	M	$P2_1/c$	4
165	$C_{27}H_{22}FeO_3P^+.BF_4^-$	$[Fe(CO)_3(C_6H_7PPh_3)]BF_4$	M	$P2_1/c$	4
117	$C_{27}H_{52}ClOP_2Rh$	*trans*-$RhCl(CO)[Bu^t_2P(CH_2)_4C{\equiv}C(CH_2)_4PBu^t_2]$	O	$Pna2_1$	4

[a] No crystal data. [b] Corrected lattice constants: see *Acta Cryst.*, 1975, B31, 40.

C_{28}

378	$C_{28}HO_{29}Os_9^-.C_{36}H_{30}NP_2^+$	$[N(PPh_3)_2][Os_3H(CO)_{10}O_2COs_6(CO)_{17}]$	M	$P2_1/c$	4
231	$C_{28}H_{20}Cl_{16}Fe_4O_8Sb_4$	$[FeCl(CO)_2(C_5H_5)]_4[SbCl_3]_4$	O	$Aba2$	4
–	$C_{28}H_{20}CoN_4^{2+}.2I^-$	$[Co(CNPh)_4]I_2$			
342	$C_{28}H_{20}N_4O_4Ru_2S_4$	$Ru_2(CO)_4(py)_2(C_7H_5NS_2)_2$	Tri	$P\bar{1}$	
114	$C_{28}H_{20}Pt$	$Pt(C_2Ph_2)_2$	M	$P2_1/n$	2
274	$C_{28}H_{23}CrO_5P$	$Cr(CO)_3[C_5H_2Ph_3P(OMe)_2]$	O	$P2_12_12_1$	4
7	$C_{28}H_{26}CrINO_3P_2$	$CrI(CO)_2(PMePh_2)_2(NO)$	M	$P2_1$	2
15	$C_{28}H_{27}FeN_5O$	$Fe(CO)(py)(C_{22}H_{22}N_4)$ [b]	O	$Pbca$	8
187	$C_{28}H_{32}ClO_2Ti$	$Ti(OC_6H_4Me_2)(OC_6H_4Cl)(C_5H_3MePr^i)(C_5H_5)$	O	$Pna2_1$	
170	$C_{28}H_{40}P_3Ru^+.F_6P^-$	$[RhH(C_4H_6)(PMe_2Ph)_3]PF_6$	M	$P2_1/n$	4
54	$C_{28}H_{56}Cl_2N_8Ru$	*trans*-$RuCl_2[\overline{CNEt(CH_2)_2NEt}]_4$	M	$P2_1/c$	4

[a] Corrected lattice constants: see ref. 305. [b] $C_{22}H_{22}N_4$ = 7,16-dihydro-6,8,15,17-tetramethyldibenzo-[*b*,*i*][1,4,8,11]tetraazacyclodecinato.

C_{29}

31	$C_{29}H_{28}O_2P_2Rh^+.F_6P^-$	$\{Rh(CO)[O(CH_2CH_2PPh_2)_2]\}PF_6$	Tri	$P\bar{1}$	2

						1157	6	*a*	299
11.277(4)	13.349(6)	17.604(7)		107.64(1)		1403	6.3	5.7	300
9.24(1)	18.85(2)	16.82(2)		118.04(5)		1644	5.6		301
21.991(2)	11.915(1)	11.890(1)				1763	9.7		257
18.75(1)	15.20(1)	28.41(2)		114.5(1)		3274	7.2		302
15.282(3)	15.215(3)	23.191(6)				1614	5.2	3.6	303
12.462	11.058	11.878	104.64	93.19	107.69			*a*	304
12.34(3)	13.53(3)	11.05(2)	96.82(8)	93.89(8)	103.11(8)	3288	9		288
13.163(5)	6.062(5)	14.354(7)		115.04(3)		3423	3.9		306
18.655	15.550	8.529					11.4		265
9.796(4)	18.019(8)	8.968(1)		116.96(3)		2233	5.5	6.8	307
30.907(13)	9.619(5)	16.615(7)				3696	4.2	3.6	260
16.42(3)	12.45(2)	12.14(2)				995	7.8		227
17.85	18.96	10.31		106.1		2429	7.0		308
15.04(1)	13.44(1)	19.43(1)		104.8(1)		974	15		309
10.907(3)	11.047(2)	13.405(3)	94.19(2)	82.30(2)	107.98(2)	2389	5.0		310

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C_{30}

9	$C_{30}H_{20}Br_2O_6F_2Re_2$	$Re_2Br_2(CO)_6(P_2Ph_4)$	Tri	$P\bar{1}$	2
236	$C_{30}H_{20}F_{11}FeP$	$Fe[C(CF_3)_2{=}C{-}C(CF_3){=}CF_2](PPh_3)(C_5H_5)$	M	$P2_1/c$	4
403	$C_{30}H_{26}Fe_2NPO_6{\cdot}C_6H_6$	$Fe_2(CO)_6(CHCPh{=}NEt_2)(PPh_2){\cdot}C_6H_6$	M	$P2_1/n$	4
356	$C_{30}H_{30}Fe_2N_2O_{10}$	$Fe_2(CO)_6[PhCHMeNCH(CO_2Et)]_2$	O	Pcca	8
131	$C_{30}H_{32}ClN_3PRhS_2{\cdot}0.8CHCl_3$	$RhCl[SC(NPh)(NMe_2)](SCNMe_2)(PPh_3){\cdot}0.8CHCl_3$	M	$P2_1/a$	4
349	$C_{30}H_{34}Ni_2$	$[Ni(cod)]_2(C_2Ph_2)$	Tet	$P4_12_12$	4
161	$C_{30}H_{51}NiP$	$Ni(C_{12}H_{18})(PCy_3)$[a]	M	$P2_1/n$	4
414	$C_{30}O_{28}Rh_{15}^{-}{\cdot}H_3O^{+}$	$H_3O[Rh_{15}C_2(CO)_{28}]$	O	Pbca	4

[a] $C_{12}H_{18}$ = 6,8-dimethyl-3,4-divinylcyclohexene.

C_{31}

28	$C_{31}H_{20}CoO_6P_2$	$Co(CO)_3[Ph_2PC{:}\overline{C(PPh_2)CO}{\cdot}OCO]$	O	Pnam	4

C_{32}

224	$C_{32}H_{20}AgI_4O_{12}W_4^{+}{\cdot}BF_4^{-}$	$\{[WI(CO)_3(C_5H_5)]_4Ag\}BF_4$	Tet	$I\bar{4}$	2
340	$C_{32}H_{29}Fe_2O_{10}P$	$Fe_2(CO)_5[Ph_2PC(O)CBu^tCC(CO_2Et)C(CO_2Et)]$	M	$P2_1$	2
276	$C_{32}H_{32}B_4NiS_2$	$Ni(\overline{SBMeCEtCEtBMe})_2$	M	$P2_1/c$	4
271	$C_{32}H_{37}As_2Nb$	$Nb(C_8H_8)(C_8H_8Ph)$(diars)	M	$P2_1/c$	4
133	$C_{32}H_{44}IrO_2P_4^{+}{\cdot}C_{24}H_{20}B^{-}$	$[Ir(O_2)(PMe_2Ph)_4]BPh_4$	Tri	$P\bar{1}$	2
132	$C_{32}H_{44}O_2P_4Rh^{+}{\cdot}C_{24}H_{20}B^{-}$	$[Rh(O_2)(PMe_2Ph)_4]BPh_4$	Tri	$P\bar{1}$	2
68	$C_{32}H_{51}CoN_6O_4$	$Co\{CH_2CH[(CH_2)_6]_2\}(py)(dmg)_2$	M	$P2_1/c$	4

11.110(9)	11.538(9)	12.913(9)	95.95(5)	102.54(5)	95.92(5)	2373	7.2		311
10.423(3)	19.375(4)	15.202(4)		116.77(3)		2006	3.9		112,312
14.694(7)	13.653(7)	17.787(7)		99.03(7)		3760	3.5		313
30.699(8)	14.731(4)	13.768(4)				1100	8.3		197
19.030(13)	19.380(10)	9.570(8)		92.5(2)		2039	6.6		314
11.595(2)		18.083(2)				748	2.4		315
20.401	13.634	10.282		n.g.			4.9		316
15.01(2)	17.34(2)	18.85(2)				2387	2.67	3.05	317
16.76(1)	7.871(6)	21.24(1)				1561	4.6		318
18.0755(15)		6.8263(5)					9.8		319
11.474(4)	16.779(5)	9.926(3)		97.35(4)		3314	4.4		320
19.594(12)	11.8593(4)	9.3736(4)		102.848(6)		3624	11		321
8.981(16)	31.930(26)	12.177(7)		106.97(8)		2201	13.2	14.1	322
17.62(2)	14.30(2)	11.46(2)	104.85(5)	105.20(5)	93.18(5)	3312	5.4		323
17.56(1)	14.24(1)	11.38(1)	104.80(5)	104.9(1)	93.05(5)	3594	8		324
9.275	23.308	12.908		114.48		2307	7.3		325

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C_{33}

239	$C_{33}H_{27}F_6O_4PRu$	$Ru[C_2(CO_2Me)_2C_2(CF_3)_2H](PPh_3)(C_5H_5)$	Tri	$P\bar{1}$	2
90	$C_{33}H_{33}ClIrN_2O_3P$	$Ir\overline{Cl[(C_6H_3MeO)_2P(Ctol\text{-}o)]}(\gamma\text{-pic})_2$	M	$P2_1/c$	4
299	$C_{33}H_{37}ClP_2PtSi$	(+)-*trans*-$PtCl[SiMePh(1\text{-}C_{10}H_7)](PMe_2Ph)_2$	M	$P2_1$	2
32	$C_{33}H_{38}O_5P_2Rh^+.F_6P^-$	$[Rh(CO)(OH_2)\{O[(CH_2)_2O(CH_2)_2PPh_2]_2\}]PF_6$	Tri	$P\bar{1}$	2

C_{34}

326	$C_{34}H_{20}Fe_2O_6$	$Fe_2(CO)_6(C_4Ph_4)$	M	$P2_1$	2
470	$C_{34}H_{30}Cl_2OP_2Pd.C_7H_8$	$Pd\overline{Cl_2[CH(COPh)PPh_2(CH_2)_2PPh_2]}.PhMe$	M	$P2_1/c$	4
151	$C_{34}H_{38}N_6Pd_2$	$\{Pd(C_3H_5)[N_3(p\text{-tol})_2]\}_2$	M	$P2_1/c$	
351	$C_{34}H_{38}P_2Pt$	$Pt_2(C_2Ph_2)_2(PMe_3)_2$	M	$P2_1/c$	4
25	$C_{34}H_{44}MnO_{10}P_4^+.F_6P^-$	$\{cis\text{-}Mn(CO)_2[PPh(OMe)_2]_4\}PF_6$	M	$P2/c$	2
26	$C_{34}H_{44}MnO_{10}P_4^+.F_6P^-$	$\{trans\text{-}Mn(CO)_2[PPh(OMe)_2]_4\}PF_6$	M	$P2_1/n$	4
204	$C_{34}H_{54}Al_2Cl_2Zr_2$	$[Zr(ClAlEt_3)(C_5H_5)]_2C_2H_4$			

[a] Diagram only.

C_{35}

240	$C_{35}H_{18}F_9N_2PRu$	$\overline{Ru(C_5F_4N{:}NC_6F_5)(C_5H_4C_6H_4PPh_2)}$	M	$P2_1/c$	4
337	$C_{35}H_{20}Fe_2O_7.\frac{1}{2}C_6H_6$	$Fe_2(CO)_6(C_4Ph_4CO).\frac{1}{2}C_6H_6$	M	$P2_1/n$	4
341	$C_{35}H_{29}Fe_2O_8P$	$Fe_2(CO)_5[Ph_2PC(Ph)C(CO_2Et)CCBu^tCO]$	Tri	$P\bar{1}$	2
235	$C_{35}H_{33}FeO_3P$	$Fe[C(O)OC_6H_3MePr^i](CO)(PPh_3)(C_5H_5)$	O	$P2_12_12_1$	4

9.353(4)	16.346(16)	10.875(6)	93.82(7)	113.07(4)	94.26(6)	4184	3.9		326
9.12(2)	17.36(2)	20.65(2)		90.5(1)		2583	7.6		327
12.797(2)	12.491(1)	10.186(2)		104.49(2)		2876	3.0	3.4	328
9.837(2)	11.416(2)	17.554(3)	94.08(2)	79.64(1)	111.98(1)	3106	5.7		310
16.519(8)	7.895(5)	11.386(5)		98.38(2)		1234	6.90	4.04	329
13.39(5)	16.88(1)	19.85(6)		123.54(1)		2652	6.5		330
8.510(2)	40.652(9)	9.762(2)		103.61(2)		3978	4.1	6.0	331
12.963(3)	16.486(8)	17.498(10)		116.04(4)		2158	6.6		306
14.51(1)	9.42(1)	18.60(2)		124.5(2)		1550	6.7		332
22.99(3)	14.38(2)	14.21(2)		107.0(2)		2310	7.6		332
								[a]	235
15.650(14)	13.338(11)	14.741(11)		97.38(3)		3092	4.6	5.8	333
13.167(5)	11.535(3)	21.787(8)		95.11(2)		4277	6.4	9.7	334
11.308(6)	14.343(12)	11.424(14)	86.68(16)	69.93(6)	104.87(11)	4408	2.9		320
11.201(3)	14.817(7)	18.976(3)				2194	6.6	7.2	335

C_{36}

136	$C_{36}H_{30}NO_3P_2Rh$	$Rh(SO_2)(NO)(PPh_3)_2$	O	Pbca	8
92	$C_{36}H_{30}N_4O_4Pd_2$	[Pd(apo)$_2$](salophen)	M	$P2_1/c$	4
86	$C_{36}H_{54}N_2P_4Pt_2S_2$	$[Pt(NCS)(PEt_3)_2C_2]_2C_6H_4$-*p*	M	$P2_1/c$	2

C_{37}

404	$C_{37}H_{20}F_6Fe_3O_7P_2$	$Fe_3(CO)_7[Ph_2PC_4(CF_3)_2](PPh_2)$	M	$P2_1/c$	4
296	$C_{37}H_{30}Br_2ClHgIrOP_2$	$Ir(HgBr)BrCl(CO)(PPh_3)_2$	M	$P2_1/n$	4
297	$C_{37}H_{30}Cl_3HgIrOP_2$	$Ir(HgCl)Cl_2(CO)(PPh_3)_2$	M	$P2_1/n$	4
223	$C_{37}H_{37}MoP_2^+.F_6P^-.O_2S$	$[Mo(dppe)(C_6H_8)(C_5H_5)]PF_6.SO_2$	M	$P2_1/c$	4
70	$C_{37}H_{47}N_4Rh$	RhMe(oep)	Tri	$P\bar{1}$	2

C_{38}

202	$C_{38}H_{30}Hf$	$Hf(C_4Ph_4)(C_5H_5)_2$	M	$P2_1/n$	4
44	$C_{38}H_{30}O_6P_2RuS$	$Ru(SO_4)(CO)_2(PPh_3)_2$	O	Pbcn	4
201	$C_{38}H_{30}Ti$	$Ti(C_4Ph_4)(C_5H_5)_2$	M	$P2_1/n$	4
6	$C_{38}H_{31}IrO_2P_2$	$IrH(CO)_2(PPh_3)_2$	O	$Pna2_1$	4
		(form I)	M	$P2_1/a$	
		(form II)	M	$P2_1/c$	
426	$C_{38}H_{42}B_9P_2Rh$	3-H-3,1,2-$(Ph_3P)_2RhC_2B_9H_{11}$	M	$P2_1/c$	4
425	$C_{38}H_{43}B_9P_2Ru$	2,1,7-$(Ph_3P)_2RuH_2C_2B_9H_{11}$	M	Cc	4

C_{39}

357	$C_{39}H_{36}ClCuN_3OP_2Rh$	$RhCuCl(CO)(PPh_3)_2$(dmt)	M	$P2_1/c$	4
241	$C_{39}H_{36}CoO_6P$	$Co[[PhC_2(CO_2Me)][CH(CO_2Me)]_2](PPh_3)$-$(C_5H_5)$	Tri	$P\bar{1}$	2
160	$C_{39}H_{44}ClIrO_2.C_2H_6O$	IrCl[(+)-diop](cod).EtOH	O	$P2_12_12_1$	

10.338(2)	18.500(4)	33.933(7)				2615	7		336
7.109(4)	25.321(13)	17.979(8)		107.8(4)		3119	6.1	6.9	337
9.136(2)	13.388(8)	19.394(18)		109.05(2)		1954	5.1		338
12.325(4)	21.075(4)	19.701(9)		133.54(1)		2921	5.3	7.4	339
16.021(7)	21.788(6)	10.568(4)		93.11(2)		3810	7.2	7.8	340
15.805(2)	21.734(3)	10.537(1)		92.13(2)		3430	5.9	7.6	340
10.879	15.205	22.357		97.50		3513	6		341
15.891(10)	10.890(4)	10.753(1)	100.96(2)	95.44(3)	59.53(3)	4186	5.2		342
13.822(8)	11.149(8)	18.694(9)		93.18(5)		1360	4.8	5.7	343
19.720(5)	10.655(2)	15.964(4)				2147	5.4		344
13.758(5)	11.059(5)	18.492(6)		93.76(3)		1790	3.9	4.1	343
17.759(3)	10.001(3)	18.389(2)				2518	1.87		345
18.036(5)	10.075(2)	19.474(5)		113.37(3)					
17.679(4)	10.205(3)	18.390(5)		91.78(2)					
12.669(6)	18.587(4)	16.041(7)		97.08(4)		2362	5.3		346
24.01(1)	13.523(2)	19.344(5)		123.32(3)		1982	9.8		347
11.93(2)	18.26(4)	17.60(3)		101.54(1)		3282	6.8		348,349
12.330	17.834	8.955	98.12	116.13	81.13	4673	10.6		350
10.248	18.223	20.212							351

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C_{40}

95	$C_{40}H_{33}ClP_2Pd$	$PdCl[o\text{-}Ph_2PC_6H_4CHCH{:}CMeC_6H_4PPh_2\text{-}o]$	N	$P2_1/n$	4
43	$C_{40}H_{33}O_4P_2Re$	$Re(OAc)(CO)_2(PPh_3)_2$	M	$P2_1/c$	4
200	$C_{43}H_{38}Ti_2$	$[Ti(C_5H_4Me)]_2(PhC_2C_2Ph)$	M	$P2_1/c$	2
30	$C_{40}H_{46}CoO_4P_3^+.BF_4^-.C_4H_8O$	$[Co(CO)[P(OMe)_3][PhP[(CH_2)_3PPh_2]_2]]\text{-}BF_4\text{-}thf$	Tri	$P\bar{1}$	2
245	$C_{40}H_{60}N_2Ti_2$	$[Ti(C_5Me_5)_2]_2N_2$	Tri	$P\bar{1}$	4
246	$C_{40}H_{50}N_6Zr_2$	$[Zr(N_2)(C_5Me_5)_2]_2N_2$	M	$P2_1/n$	4

C_{41}

49	$C_{41}H_{42}CoN_6O_{10}P_2^+.ClO_4^-$	$\{Co(CNC_6H_4NO_2)_3[PPh(OEt)_2]_2\}ClO_4$	M	$P2_1/c$	4

C_{42}

106	$C_{42}H_{38}O_2P_2Pt$	$Pt(CO_2CMe_2C{:}CH_2)(PPh_3)_2$[a]	M	$P2_1/c$	4

[a] $CO_2CMe_2C{:}CH_2$ = 1-oxo-2-oxa-3,3-dimethylpent-4-ene-1,4-diyl.

C_{43}

406	$C_{43}H_{30}Fe_2O_7Sb_2$	$Fe_2(SbPh_2)Ph(CO)_7(SbPh_3)$	M	$P2_1/c$	4
29	$C_{43}H_{42}CoNOP_3^+.C_{24}H_{20}B^-.C_3H_6O$	$[Co(CO)(np_3)]BPh_4.Me_2CO$	M	$P2_1/c$	4
34	$C_{43}H_{42}NNiOP_3$	$Ni(CO)(np_3)$	M	$P2_1$	2

C_{44}

415	$C_{44}H_{30}Ni_8O_8P_6$	$Ni_8(PPh)_6(CO)_8$	O	Pcab	4
338	$C_{44}H_{32}Fe_2O_4$	$Fe_2(CO)_4(C_{20}H_{16})_2$	M	C2/c	4
115	$C_{44}H_{42}OP_2Pt$	$Pt(HC_2C_6H_{10}OH)(PPh_3)_2$	Tri	$P\bar{1}$	4
73	$C_{44}H_{45}NNiOP_3^+.C_{24}H_{20}B^-$	$[Ni(COMe)(np_3)]BPh_4$	Tri	$P\bar{1}$	2

13.055(5)	18.151(7)	15.224(6)		112.43(2)		4436	2.9	3.7	352
17.63(2)	9.72(1)	20.95(2)		104.63(10)		2776	3.5	3.8	353
10.133(2)	14.838(4)	10.530(3)		106.97(1)		1450	5.2	3.7	354
14.310(1)	11.080(2)	11.099(3)	68.30(2)	90.78(2)	75.04(1)	3123	11.7		355
18.867(1)	8.968(2)	22.767(1)	98.22(1)	101.83(1)	93.86(1)	7137	5.8		356
14.831(1)	16.992(1)	16.260(3)		90.00		4612	3.5		357,358
13.085(5)	10.826(4)	33.019(8)		91.92(3)		2358	7.2		359
12.298(2)	11.038(3)	27.207(3)		102.66(1)		3732	6.21		360
17.95	10.76	23.98		116.6		4219	4.4		361
12.43(1)	20.29(2)	23.85(2)		103.0(1)		1076	7.3	7.8	362
20.44(1)	8.87(1)	10.34(1)		90.4(1)		1480	4.9	5.7	362
20.489(13)	16.861(11)	14.485(6)				1505	6.2	6.3	363
8.971(2)	15.267(3)	14.316(2)		93.56(1)		656	2.9	3.7	364
17.72(2)	17.44(2)	12.78(1)	97.8(1)	101.6(1)	86.9(1)	7205	5.3	5.3	365
18.168(4)	17.537(4)	10.789(3)	95.62(4)	100.88(4)	95.80	1208	9.8		366

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17	$C_{44}H_{55}N_5OOs$	$Os(CO)(py)(oepMe_2)$[a]	O	Pnma	4

[a] $oepMe_2$ = α,γ-dimethyl-α,γ-dihydrooctaethylporphin.

C_{45}

48	$C_{45}H_{37}N_2O_2OsP_2^+.ClO_4^-$	$[Os(CO)(CNtol\text{-}p)(NO)(PPh_3)_2]ClO_4$	M	$P2_1/n$	4

C_{46}

107	$C_{46}H_{38}N_4OP_2Pt$	$Pt[C_4H_3(CN)_4(OEt)](PPh_3)_2$	M	$P2_1/c$	4
85	$C_{46}H_{40}P_2Pt$	$Pt(C_2CMe{:}CH_2)_2(PPh_3)_2$	Tri	$P\bar{1}$	1
47	$C_{46}H_{53}ClFeN_2O_6P_3^+.ClO_4^-$	$\{FeCl(CNtol)_2[PPh(OEt)_2]_3\}ClO_4$	M	$P2_1/n$	4

C_{47}

113	$C_{47}H_{33}O_2P_2Pt$	$Pt(C_4MePhO_2)(PPh_3)_2$[a]	Tri	$P\bar{1}$	2

[a] C_4MePhO_2 = 1-methyl-2-phenylcyclobutenedione.

C_{48}

139	$C_{48}H_{30}F_{10}P_4Pt$	$Pt[P_2(C_6F_5)_2](PPh_3)_2$	M	$P2_1$	2
75	$C_{48}H_{34}N_4NiO_2.CHCl_3$	$NiCH(CO_2Et)(tpp).CHCl_3$	Tri	$P\bar{1}$	2
55	$C_{48}H_{45}ClN_3O_6P_2RhS_3.C_3H_6O$	$RhCl[(EtOCONCS)_3](PPh_3)_2.Me_2SO$	Tri	$P\bar{1}$	4
74	$C_{48}H_{47}As_3NNi^+.C_{24}H_{20}B^-$	$[NiPh(nas_3)]BPh_4$	Tri	$P\bar{1}$	2
313	$C_{48}H_{48}N_6P_2Pd_3^{2+}.2F_6P^-$	$[Pd_3(CNMe)_6(PPh_3)_2](PF_6)_2$	Tri	$P\bar{1}$	1
279	$C_{48}H_{56}Na_4Ni_2O_5$	$[NiPh_2(C_2H_4)]_2Na_4(thf)_5$	Tri	$P\bar{1}$	2
314	$C_{48}H_{97}P_2Pt_2Si_2$	$[PtH(SiEt_3)(PCy_3)]_2$	M	$P2_1/c$	4

16.937(1)	24.694(2)	9.778(1)				4644	3.1	3.8		367
13.257(4)	21.48(2)	14.820(6)		99.33(2)		2423	7.2	8.7		368
10.586(6)	20.422(10)	23.234(10)		115.23(10)		2636	9.2			369
11.79(1)	10.33(1)	8.32(1)	90.9(1)	106.6(1)	105.6(1)	4787	3.7			370
11.926(4)	15.189(5)	29.36(2)		90.57(3)		1273	7.4			371
20.967(10)	12.478(6)	9.358(6)	109.7(4)	107.1(4)	103.5(5)	4413	8.9			372
9.286(5)	20.95(1)	11.226(5)		90.7(1)		2477	4.3	4.7		373
13.677(6)	14.212(8)	11.958(5)	98.85(8)	99.21(8)	113.73(7)	5493	6.1	8.6		374,375
15.214(6)	27.571(9)	12.857(6)	103.12(2)	90.24(2)	100.03(2)	7375	7.2	9.1		376
18.132(3)	13.377(2)	13.162(2)	84.65(2)	73.80(2)	86.93(2)	3008	6.9	6.8		377
12.094(2)	12.127(2)	10.660(2)	102.70(2)	112.92(2)	75.49(2)	3339	6.2		85K	378
9.779(2)	12.884(3)	20.811(6)	94.29(2)	93.05(2)	104.25(2)	2536	7.42	9.09		379
20.72(2)	13.88(1)	18.97(1)		101.15(7)		3150	7.5		200K	380

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C_{49}

238	$C_{49}H_{40}ClCuP_2Ru.C_3H_6O$	$Ru(C_2PhCuCl)(PPh_3)_2(C_5H_5).Me_2CO$	M	$P2_1/c$	4
100	$C_{49}H_{42}O_7P_2Ru$	$Ru[(HC_2CO_2Me)_3](CO)(PPh_3)_2$	Tri	$P\bar{1}$	2
359	$C_{49}H_{47}AgIrN_3O_3P_2.C_4H_8O_2$	$IrAg(MeN_3tol\text{-}p)(OCOPr^i)(CO)$-$(PPh_3)_2.Pr^iCO_2H$	M	$P2_1/n$	4

C_{50}

16	$C_{50}H_{33}FeN_5O.2C_6H_6$	$Fe(CO)(py)(tpp).2C_6H_6$	M	$P2_1/c$	4
347	$C_{50}H_{40}F_{12}P_6Rh_2.C_4H_{10}O$	$[Rh(PF_3)_2(PPh_3)]_2(C_2Ph_2).Et_2O$	Tri	$P\bar{1}$	2
134a	$C_{50}H_{44}IrO_2P_4^+.ClO_4^-$	$[Ir(O_2)(dppm)_2]ClO_4$	M	$P2_1/c$	4
134b	$C_{50}H_{44}IrO_2P_4^+.F_6P^-$	$[Ir(O_2)(dppm)_2]PF_6$	M	$P2_1/c$	4

C_{51}

242	$C_{51}H_{20}CoF_{20}P$	$Co[C_4(C_6F_5)_4](PPh_3)(C_5H_5)^a$	Tri	$P\bar{1}$	2
243	$C_{51}H_{20}F_{20}PRh$	$Rh[C_4(C_6H_5)_4](PPh_3)(C_5H_5)^a$	Tri	$P\bar{1}$	2
339	$C_{51}H_{35}Fe_2O_5P$	$Fe_2(CO)_5(PPh_3)(C_2Ph_2)_2$	Tri	$P\bar{1}$	2
5	$C_{51}H_{45}N_3OP_2Ru$	$RuH(CO)(PPh_3)_2(dtt)$	Tri	$P\bar{1}$	2

[a] Also contains one ill-defined solvent molecule, possibly C_6H_{14} for the Co compound, not defined for Rh.

C_{52}

56	$C_{52}H_{40}ClN_2O_2P_2RhS_2.C_4H_{10}O$	$RhCl[(PhCONCS)_2](PPh_3)_2.Et_2O$	M	$P2_1/n$	4
137	$C_{52}H_{48}IrO_2P_4S_2^+.Cl^-$	$[Ir(S_2O_2)(dppe)_2]Cl$	O	Pcab	8

C_{54}

50	$C_{54}H_{51}IrNP_4^+.ClO_4^-$	$[Ir(CNMe)(dppe)_2]ClO_4$	Tri	$P\bar{1}$	2

12.914(1)	22.111(1)	16.534(1)		110.77(1)		4496	8.4	7.5		381
11.118(5)	19.926(7)	11.518(5)	104.09(3)	119.66(3)	87.61(3)	6359	5.4			382
12.412(1)	21.701(1)	19.027(1)		97.223(2)		5161	8.3			383
13.246(7)	19.555(26)	19.822(25)		105.49(3)		1223	9.0	9.4		384
21.186(9)	12.994(5)	12.942(5)	114.10(2)	64.36(2)	115.33(2)	6834	4.2	4.6		385
11.87(1)	11.12(1)	34.61(3)		96.1(1)		3495	6			386
11.40(1)	19.26(1)	22.06(4)		101.2(1)		3371	6			386
11.680(3)	14.008(4)	20.455(9)	114.08(3)	107.41(3)	106.72(2)	5479	7.7			387
11.715(4)	14.015(6)	20.420(6)	114.07(3)	106.97(3)	107.28(3)	5235	6.5			387
12.868(9)	11.667(8)	13.867(9)	85.98(3)	90.35(3)	83.36(3)	3453	9.2	9.2	113K	388
14.074(2)	15.264(3)	12.195(2)	109.78(1)	111.74(1)	65.03(2)	5847	3.8	6.0		389,390
21.184(3)	19.963(2)	12.226(2)		100.03(1)		6266	6.1	7.9		391,392
27.421(8)	15.321(6)	24.908(8)				4811	9.8			393
13.004(7)	17.224(11)	12.127(7)	106.57(5)	109.71(5)	79.71(4)	6313	4.60	5.31		394

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C_{56}

42	$C_{56}H_{45}IrOP_3S_2^+.BF_4^-$	$[Ir(S_2CPPh_3)(CO)(PPh_3)_2]BF_4$	M	$P2_1/c$	4

C_{58}

371	$C_{58}H_{44}O_8P_4Rh_4$	$Rh_4(CO)_8(dppm)_2$	M	C2/c	8

C_{62}

88	$C_{62}H_{59}O_2P_3Ru$	$RuH(C{\equiv}CMeCO_2Bu)(PPh_3)_3$	M	C2/c	8
391	$C_{52}H_{66}N_4Ni_4.C_6H_6$	$Ni_4(CNBu^t)_4(C_2Ph_2)_3.C_6H_6$	M	$P2_1/n$	4

C_{64}

270	$C_{64}H_{42}U$	$U(C_6H_4Ph_4)_2$	M	P2/c	4
			O	Pccn	2

[a] Monoclinic unit cell for ideal ordered structure, orthorhombic cell for disordered structure; refined in latter.

C_{75}

363	$C_{75}H_{132}O_3P_4Pt_3$	$Pt_3(CO)_3(PCy_3)_4$	Tri	$P\bar{1}$	2

C_{100}

280	$C_{100}H_{150}Li_{12}N_4Na_6Ni_4O_{14}$	$\{Ph[Na(OEt_2)]_2(NiPh_2)_2N_2NaLi_6-(OEt)_4(OEt_2)\}_2$	Tri	$P\bar{1}$	2

10.5876(9)	31.581(10)	16.2164(5)		92.521(1)		4648	11		395
22.962(6)	17.230(5)	28.946(8)		97.42(1)		4362	6.5	12.1	396
29.691(8)	21.865(5)	21.245(6)		122.26(1)		4801	6.7	11.1	397
15.794(1)	25.180(3)	16.011(2)		102.77(1)		7056	8.9		398
24.86(2)	7.587(6)	27.78(3)		116.58				α	399
24.84(3)	12.43(1)	7.587(6)				723	3.3	8.1	
20.342(10)	15.372(10)	13.876(10)	97.92(4)	86.89(5)	79.36(5)	6476	4.9		400
15.775(4)	18.689(5)	23.607(5)	66.02(2)	66.39(2)	78.87(2)	4750	7.9	9.5	401

TABLE 4. HYDRIDE AND BOROHYDRIDE COMPLEXES

No.	FORMULA	STRUCTURE	CRYSTAL CLASS	SPACE GROUP	Z
435	$C_{28}H_{47}N_2P_2Rh$	$RhH(N_2)(PBu^t_2Ph)_2$	M	C2/c	4
436	$C_{36}H_{31}IrN_2O_6P_2$	$IrH(NO_3)_2(PPh_3)_2$	Trig	$P\bar{3}c1$	6
440	$C_{37}H_{53}P_2PtS_2$	*trans*-$PtH(S_2CH)(PCy_3)_2$	Tri	$P\bar{1}$	2
441	$C_{50}H_{45}N_3P_2Pt$	*trans*-$PtH[N_3(tol\text{-}p)_2](PPh_3)_2$	Tri	$P\bar{1}$	2
437	$C_{52}H_{49}IrP_4$	$IrH(dppe)_2$	M	$P2_1/n$	4
434	$C_{54}H_{43}CoP_4$	$CoH[P(C_6H_4PPh_2)_3]$	O	Pbca	8
433	$C_{54}H_{47}NOP_3Re.\frac{1}{2}C_6H_6$	$ReH_2(NO)(PPh_3)_3.\frac{1}{2}C_6H_6$	O	$Pca2_1$	8
438	$C_{72}H_{65}Ir_2P_4^+.F_6P^-.CH_2Cl_2$	$[Ir_2H_2(\mu\text{-}H)_3(PPh_3)_4]PF_6.CH_2Cl_2$	M	$P2_1/c$	4
439	$C_{94}H_{72}ClIr_2P_4S_2^+.ClO_4^-.3C_5H_6O$	$\{[IrH(PPh_3)_2]_2Cl(SPh)_2\}ClO_4.3Me_2CO$	M	$P2_1$	2
443	$C_{32}H_{44}B_4N_4O_4Ti_2.2C_4H_8O$	$[Ti(salen)(BH_3)_2]_2.2thf$	M	$P2_1/c$	2
444	$C_{41}H_{43}BCoP_3$	$Co(H_2BH_2)[(Ph_2PCH_2)_3CMe]$	O	$Pn2_1a$	4

[a] Disorder in PF_6^-, Ph groups of PPh_3.

a	b	c	α	β	γ	DATA	R	R'	NOTES	REFERENCE
22.187(2)	8.340(1)	15.979(2)		93.108(6)		2760	3.4	5.0		402
16.33(1)		22.85(2)				1738	9.8			403
13.794(10)	14.194(12)	11.942(10)	103.87(5)	96.03(6)	78.42(4)	3271	6.7			404
14.649(2)	15.082(2)	10.929(1)	110.99(5)	102.90(5)	84.54(5)	4673	6.9			405
21.260(5)	20.809(3)	10.107(3)		91.44(2)		3452	3.65	4.15		406
17.040(4)	22.312(6)	22.581(6)				1203	6.8	4.8		407
25.069(6)	18.737(4)	20.644(5)				2938	4.8			408,409
16.505	17.260	22.213		91.546		11724	12.91[a]			410
15.013(3)	12.918(1)	22.044(4)		91.94(1)		3726	4.7	7.1		411
11.802(3)	20.039(7)	9.272(3)		90.00(5)			5			412
20.530(6)	17.120(5)	10.316(3)				1013	6.6	4.6		413,414

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TABLE 5. NITROSYL COMPLEXES

No.	FORMULA	STRUCTURE	CRYSTAL CLASS	SPACE GROUP	Z
453	$C_4H_{16}ClCoN_5O_5^+.ClO_4^-$	$[Co(NO)(OClO_3)(en)_2]ClO_4$	M	$P2_1/c$	4
454	$C_5H_7CoN_2O_2S_2$	$Co(NO)_2(SacSac)$	M	$C2/m$	4
449	$C_8H_{20}Br_6N_2O_4Ru_2S_2$	$[Ru(NO)Br_3(OSEt_2)]_2$	Tri	$P\bar{1}$	1
445	$C_{18}H_{26}BCl_4MoN_7O_2$	$Mo(NO)Cl(OPr^i)[HB(C_3Me_2ClN_2)_3]$	M	$P2_1/n$	4
455	$C_{36}H_{33}Br_2NO_7P_2Rh$	*cis*-$Rh(NO)Br_2[P(OPh)_3]_2$	M	$P2_1/c$	4
450	$C_{36}H_{30}CoN_2O_2^+.F_6P^-$	$[Co(NO)_2(PPh_3)_2]PF_6$	M	$C2/c$	4
451	$C_{42}H_{42}CoN_2OP_3^+.C_{24}H_{20}B^-$	$[Co(NO)(np_3)]BPh_4$	Tri	$P\bar{1}$	2
452	$C_{42}H_{42}CoN_3O_3P_3^+.C_{24}H_{20}B^-$	$[Co(NO)_2(np_2o)]BPh_4$	M	$P2_1/n$	4
448	$C_{42}H_{42}FeNOP_4^+.C_{24}H_{20}B^-$	$[Fe(NO)(pp_3)]BPh_4$	Tri	$P\bar{1}$	2
447	$C_{42}H_{42}FeN_2OP_3^+.C_{24}H_{20}B^-$	$[Fe(NO)(np_3)]BPh_4$	O	$Pccn$	8
456	$C_{42}H_{42}N_2NiOP_3^+.C_{24}H_{20}B^-$	$[Ni(NO)(np_3)]BPh_4$	Tri	$P\bar{1}$	2
446	$C_{48}H_{34}FeN_7O.CHCl_3$	$Fe(NO)(Meim)(tpp).CHCl_3$	O	$P2_12_12_1$	4

a	b	c	α	β	γ	DATA	R	R'	NOTES	REFERENCE
7.831(3)	15.996(6)	12.429(4)		113.26(2)		1437	3.2	4.4		415
16.565(2)	7.604(2)	8.709(1)		114.90(1)		580	4.0	4.0		416
8.042(1)	11.020(1)	7.324(1)	104.83(1)	102.31(1)	88.54(1)	1337	4.7	5.8		417
12.268(4)	16.041(4)	15.033(5)		91.44(4)		2450	4.7			418
14.78(2)	13.50(2)	20.35(2)		109.6(2)		1688	11.4			419
17.431(9)	12.352(6)	20.256(10)		125.27		3271	4.48	5.4		420
18.009(15)	16.498(11)	10.262(6)	86.27(8)	74.52(7)	80.43(8)	796	14.2	14.5		421
20.144	15.530	18.941		101.68		2647	6.7	7.2		422
19.106(3)	12.762(2)	12.452(3)	93.69(2)	107.12(2)	106.02(2)	4250	7.8			423
33.35(8)	18.54(2)	18.47(2)				1021	19.9			421
18.038(8)	16.517(7)	10.185(5)	86.78(8)	74.91(8)	80.99(8)	1840	6.8	7.2		421
17.733(13)	25.339(22)	9.752(10)				4148	5.2	7.4		424

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TABLE 6. DINITROGEN, ARYLDIAZO AND RELATED COMPLEXES

No.	FORMULA	STRUCTURE	CRYSTAL CLASS	SPACE GROUP	Z
458	$C_{34}H_{85}Co_2MgN_4O_4P_6$	$[Co(PMe_3)_3N_2]_2Mg(thf)_4$			
457	$C_{54}H_{83}Cl_6MoN_4P_8Re_2.CH_2Cl_2$	$Mo[N_2ReCl(PMe_2Ph)_4]_2Cl_4.CH_2Cl_2$	M	$P2_1/c$	2
463	$C_{42}H_{34}Ir_2N_3O_5P_2^+.F_6P^-$	$[Ir_2(N_2C_6H_4NO_2)O(NO)_2(PPh_3)_2]PF_6$	Tet	$I4_1/a$	
462	$C_{45}H_{44}ClIrN_2P_3^+.F_6P^-$	$[Ir(N_2Ph)Cl(PMe_2Ph)_3]PF_6$	M	$P2_1$	2
459	$C_{52}H_{59}FMoN_2P_4^+.BF_4^-.CH_2Cl_2$	$[Mo(N_2H_2)F(dppe)_2]BF_4.CH_2Cl_2$	O	$P2_12_12_1$	4
461	$C_{56}H_{56}BrN_2OP_4W^+.F_6P^-$	$\{W[NN:CH(CH_2)_3OH]Br(dppe)_2\}PF_6$	M	$P2_1/n$	4
460	$C_{60}H_{66}IMoN_2P_4^+.I^-.\frac{1}{2}C_5H_6$	$[Mo(EN_2C_6H_{17})I(dppe)_2]I.\frac{1}{2}C_6H_6$	M	$P2_1/n$	4

TABLE 7. BINARY TERTIARY PHOSPHINE COMPLEXES

No.	FORMULA	STRUCTURE	CRYSTAL CLASS	SPACE GROUP	Z
469	$C_{26}H_{26}AuP_2^+.F_6P^-$	$[Au(PMePh_2)_2]PF_6$	M	C2/c	4
466	$C_{28}H_{46}P_2Pd$	$Pd(PBu^t_2Ph)_2$	O	Fdd2[a]	8
467	$C_{28}H_{46}P_2Pt$	$Pt(PBu^t_2Ph)_2$	O	Fdd2[a]	8
464	$C_{40}H_{51}CoO_6P_5^+.BF_4^-$	$\{Co[P(OMe)_3]_2[(Ph_2PCH_2CH_2)_2PPh]\}BF_4$	M	$P2_1/c$	4
465	$C_{72}H_{60}P_4Pd.\frac{1}{2}C_6H_6$	$Pd(PPh_3)_4.\frac{1}{2}C_6H_6$	Cub	Pa3	8
468	$C_{32}H_{78}P_6Pt$	$Pt[(Ph_2PCH_2)_3CMe]_2$	Tri	$A\bar{1}$	4

[a] Space group corrected from ref. 434.

a	b	c	α	β	γ	DATA	R	R'	NOTES	REFERENCE
							7.6		203K	425
16.710(10)	14.164(10)	19.084(11)		114.3(5)		1893	6.8			426
35.96(6)		14.63(2)				1420	9.3			427
15.767(7)	15.583(7)	9.002(4)		91.67(2)		7134	4.6	7.2		428
18.966(7)	20.740(7)	13.362(5)				4992	8.41			429
17.43(1)	17.28(1)	19.00(1)		100.85(2)		4167	6.8			430
12.638(2)	25.901(4)	19.337(2)		109.75(1)		4750	4.5			431

a	b	c	α	β	γ	DATA	R	R'	NOTES	REFERENCE
23.23(2)	10.33(1)	15.31(2)		131.28(10)		1760	4.83	5.18		432
45.337(7)	12.628(2)	10.070(2)				1644	5.4	6.0		433
45.100(4)	12.590(1)	10.048(1)				1973	3.3	3.4		433
13.18(4)	11.49(3)	29.66(6)		101.59(2)		2256	10.0			355
22.863(5)						1340	6.6			435
15.018(5)	25.937(9)	22.722(8)	104.16(2)	115.80(2)	88.65(2)	4783	8.4			436

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APPENDIX

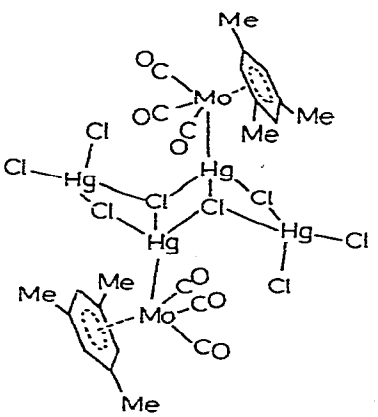
After completion of the main portion of this article, a number of structures reported during 1976 came to the author's attention. For completeness, these are listed below, together with such details as were available.

(470) $[TiCl(BH_4)(C_5H_5)]_2$: BH_4 attached to Ti via ternary H-bridge [437].

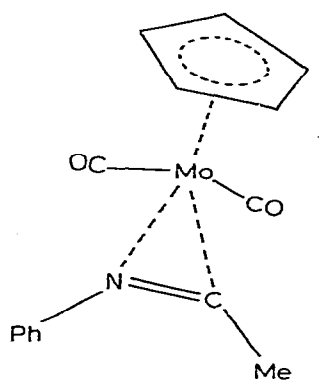
(471) $[Ti(OCOPh)_2(C_5H_5)]_2$: 2 crystallographically different molecules, each a dimer via 4 carboxylate bridges [438].

(472) $[Mo(HgCl_2)_2(CO)_3(C_6H_3Me_3)]_2$: 1:1 Lewis adduct, with central Hg_2Cl_2 unit, and chlorine bridges extending the $HgCl_2$ network; Mo→Hg dative bonding, with Mo-Hg 2.745(1)Å [439].

(473) $Mo(CO)_2(MeCNPh)(C_5H_5)$: From $[Mo(CO)_2(CNPh)(C_5H_5)]^-$ + MeI; contains π-bonded iminoacyl group [440].



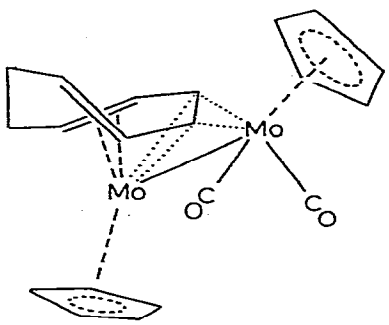
(472) $[Mo(CO)_3(C_6H_3Me_3)(HgCl_2)_2]_2$



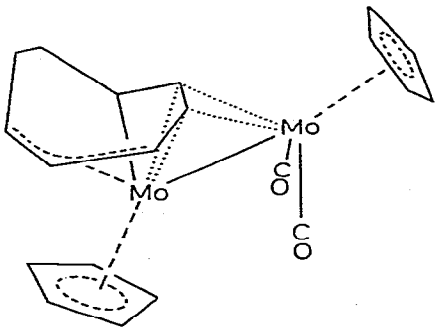
(473) $Mo(MeCNPh)(CO)_2(C_5H_5)$

(474) $[Mo(CO)_2(C_5H_5)]_2C_8H_8$ *(purple isomer)*: C_8 ligand bonded $\eta^2 + \eta^2$ together with a bridging alkyne unit; irreversibly isomerises to (475) by H shift; Mo-Mo 3.057(3)Å [441].

(475) $[Mo(CO)_2(C_5H_5)]_2C_8H_8$ *(orange isomer)*: C_8 ligand bonded $\eta^3 + \eta^1$ together with bridging C-C between 2 Mo atoms, i.e. as an η-alkyne; Mo-Mo 3.032(3)Å [441].



(474) purple



(475) orange

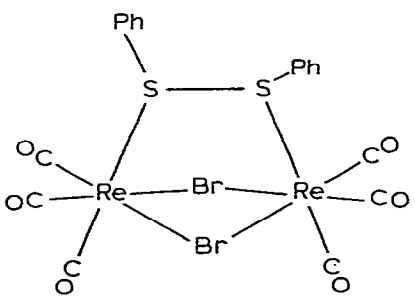
$Mo_2(CO)_2(C_5H_5)_2(C_8H_8)$

(227a) *$Mn(CO)_2(C{=}CHPh)(C_5H_5)$*: Full details of structure determination are in reference [442].

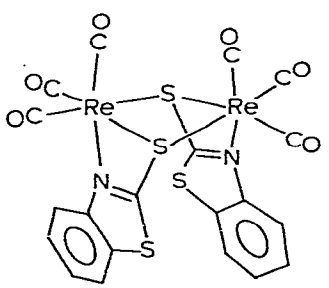
(228) *$[Mn(CO)_2(C_5H_5)]_2(C{:}CHPh)$*: Full details of structure determination are in reference [443].

(476) *$[ReBr(CO)_3]_2S_2Ph_2$*: From addition Ph_2S_2 + $[ReBr(CO)_3(thf)]_2$; contains non-planar Re_2Br_2 unit; S-S at 2.140(9)Å longer than in parent ligand [cf. (9)] [444].

(477) *$[Re(CO)_3(mbt)]_2$*: Bent Re_2S_2 moiety, with boat conformation of tricyclic $Re_2S_2C_2N_2$ unit [445].



(476) $[ReBr(CO)_3]_2S_2Ph_2$



(477) $[Re(mbt)(CO)_3]_2$

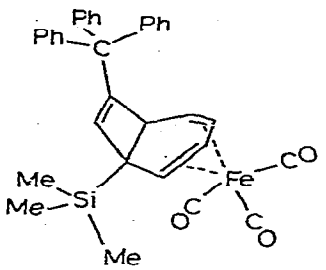
(41) *$Ru(CO)_2(py)_2(mbt)_2$*: Full details of structure determination are in reference [446].

(342) *$Ru_2(CO)_4(py)_2(mbt)_2$*: Full details of structure determination are

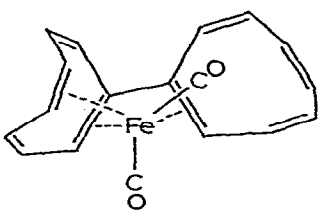
in reference [447].

(*478*) *$Fe(CO)_3[C_8H_6(SiMe_3)(CPh_3)]$*: Isomerised product from corresponding cyclooctatetraene, with $SiMe_3$ *endo* to metal [448].

(*479*) *$Fe(CO)_2(C_{16}H_{14})$*: 1,3-diene complex, with 1 C=C from each ring of bicyclooctatetraenyl ligand, + 3rd C=C coordinated to give tub conformation of one C_8 ring; latter group readily replaced by CO [449].



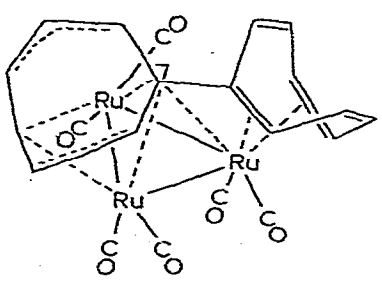
(478) $Fe(CO)_3[C_8H_6(SiMe_3)(CPh_3)]$



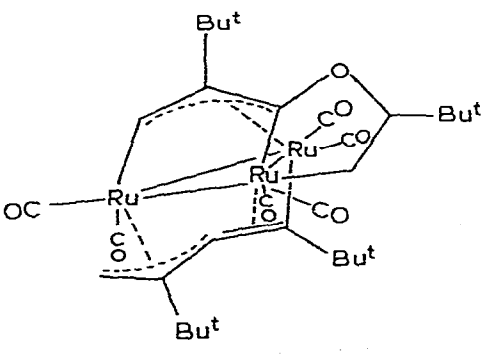
(479) $Fe(CO)_2(C_{16}H_{14})$

(*480*) *$Ru_3(CO)_6(C_{16}H_{14})$*: From $Ru_3(CO)_{12}$ + bicyclooctatetraenyl; unusual attachment of one C_8 ring, especially C(7), which is bonded to all three metal atoms [449].

(*481*) *$Ru_3(CO)_6(C_{12}H_{20})(C_{13}H_{20}O)$*: Isosceles Ru_3 cluster, with Ru-Ru 2.686 (doubly bridged), 2.820, 2.828(1)Å; each ligand acts as 6e donor, one formed from 2 HC_2Bu^t, the other from 2 HC_2Bu^t + CO [cf. (*389*)] [450].

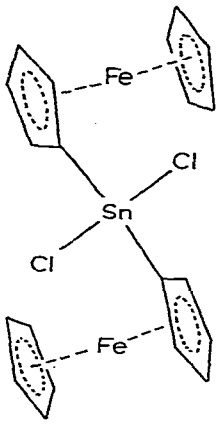


(480) $Ru_3(CO)_6(C_{16}H_{14})$



(481) $Ru_3(CO)_6(C_{12}H_{20})(C_{13}H_{20}O)$

(482) Fc_2SnCl_2: Reference [451].



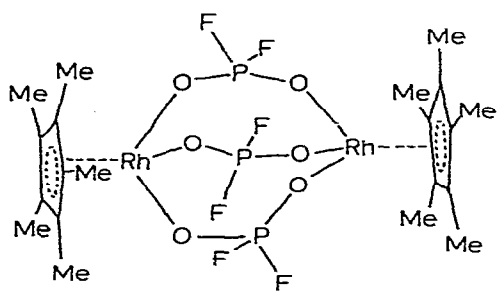
(482) Fc_2SnCl_2

(471) and (483) no diagrams available

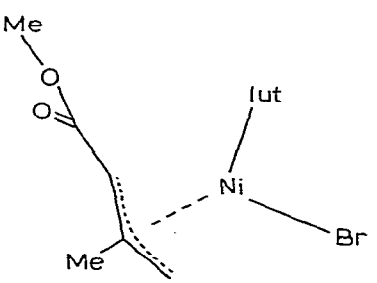
(483) $Co(B_9C_2H_{10})_2$-8,8'-OMe: Consists of 2 dicarbollide units linked by common apical Co, and also by bridging OMe; pentagonal faces bonded to Co are inclined to each other [452].

(484) $[(C_5Me_5)Rh(PO_2F_2)_3Rh(C_5Me_5)]PF_6$: Results from partial hydrolysis of PF_6^- during attempted isolation of $[(C_5Me_5)Rh(acetone)_3]^+$ complex; O, F not distinguished, but PO_2F_2 ligand O-bonded by NMR [453].

(485) $NiBr(lut)(C_3H_3MeCO_2Me)$: Square planar Ni, with no *trans* influence on geometry of η-allyl group [454].



(484) $[(C_5Me_5)Rh(PO_2F_2)_3Rh(C_5Me_5)]^+$



(485) $NiBr(lut)[C_3H_3Me(CO_2Me)]$

Carbonylated heme molecules

Four biologically important macromolecules containing heme groups have been studied structurally in recent years. A difference map of (CO)- *versus* (deoxy)erythrocruorin (a monomeric insect hemoglobin) showed that the CO ligand was inclined to the heme plane, with the Fe-C-O angle *ca.* 145(15)°, and pointing between two vinyl-substituted rings. The iron atom is nearly coplanar with the four heme nitrogens [455]. The hemoglobin from the annelid *Glycera dibranchiata*, or the common blood worm, was isolated as the CO-complex by passing carbon monoxide through the extract during isolation "whenever convenient". The heme was found to be essentially planar, with iron in the centre; the CO ligand appeared as an unresolved peak of electron density *ca.* 2.5Å from the iron atom, elongated toward the opening of heme activity. In this case, the Fe-C-O angle is *ca.* 135° [456]. A 2.8Å resolution structure of horse (CO)-hemoglobin showed the CO group apparently pushed off the heme axis by two of the side chains [457]. A comparison of (CO)-myoglobin and metmyoglobin by neutron diffraction showed relatively few changes, but the (CO)-Fe group moves into the heme plane in the former compound [458]. The displacement of CO off the axis of the heme group in these derivatives has recently been discussed by Hoffmann [459].

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