ORGANOIRON CHEMISTRY
Annual Survey for the Year 1984*

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CONTENTS

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* 1983 Survey: J. Organometal. Chem. 278 (1984) 205.

LIST OF ABBREVIATIONS USED

acac	acetylacetonate, $(MeCO)_2CH^-$
An	any arene ring, such as benzene, C_6H_6
Ar	an aryl ring, such as naphthyl, $C_{10}H_7$-
bpy	2,2'-bipyridyl
COT	1,3,5,7-cyclooctatetraene
Cp	cyclopentadienyl, C_5H_5
Cp*	pentamethylcyclopentadienyl, C_5Me_5
D	a 2-electron donor ligand such as a phosphine
DME	1,2-dimethoxyethane, sometimes called "glyme"
DMPE	1,2-bis(dimethylphosphino)ethane
DPPE	1,2-bis(diphenylphosphino)ethane
DPPM	bis(diphenylphosphino)methane
Et	ethyl, C_2H_5-
Fp	cyclopentadienyldicarbonyliron, $CpFe(CO)_2$-
Fp'	cyclopentadienyl(carbonyl)(triphenylphosphine)iron
Fp*	(pentamethylcyclopentadienyl)dicarbonyliron
Ft	the tricarbonyliron group, $Fe(CO)_3$
HMP	hexamethylphosphorictriamide
LAH	lithium aluminum hydride
M	any transition metal other than iron
Me	methyl, CH_3-
MTHF	2-methyltetrahydrofuran
NMP	N-methyl-2-pyrrolidone
Ph	phenyl, C_6H_5-
Por	any porphyrin ligand coordinated as a dianion
PPN^+	$Ph_3P{=}N{=}PPh_3^+$
R	any unicovalent organic group such as methyl
TCNE	tetracyanoethene
Tf	trifluoromethanesulfonyl group, F_3CSO_2-
THF	tetrahydrofuran
X	any halogen

1. INTRODUCTION

In this Annual Review, I attempt to cover the organoiron chemistry reported in journals published during calendar year 1984. Organoiron compounds are those compounds which contain at least one C-Fe bond; however Fe-CN compounds are not included in this review, and properties and reactions of the simple iron carbonyls are not described exhaustively. Ferrocenes are treated in Annual Surveys by B. W. Rockett and G. Marr.

The material is organized more-or-less by the Gmelin system, first by increasing number of conjoined iron atoms, then by increasing hapticity of principal organic ligand. The latter is determined by the principle of last position. Thus, (η^3-allyl)(η^5-cyclopentadienyl)(η^2-ethene)iron would be treated with cyclopentadienyliron compounds rather than with allyl- or alkene-iron species. For conciseness, many reactions of dimers such as dicyclopentadienyldiirontetracarbonyl [Fp_2, $Cp_2Fe_2(CO)_4$], in which they undergo fission into their monoiron parts, are treated alongside their monomeric derivatives such as FpR, however, and FeM_n clusters are treated as a group with other metal clusters of the same nuclearity.

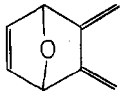
In structural drawings, lines represent electron-pair bonds unless otherwise stated. In cases where the electron pair is considered to originate from one atom, an arrowhead is used in the traditional way to show direction of electron donation. Otherwise, formal charges are shown explicitly. This reviewer finds adherence to these conventions to be possible in describing all but the largest clusters, and believes that their use provides clarity sometimes lost in casually-drawn structures. To minimize clutter in structural drawings (particularly in cluster structures), I am also introducing the use of the symbol Ft for the commonly-occurring tricarbonyliron group.

2. REVIEWS AND BOOKS

Along with many reviews and books, not detailed here, dealing with general organometallic chemistry, there were three reviews which specifically discussed aspects of organoiron chemistry. These described the use of allyltricarbonyliron lactone complexes in natural product synthesis[1]; the reactivity of dienetricarbonyliron complexes and ylides derived from them and their uses in synthesis[2]; and, in an extensive two-part review, the cyclopentadienyliron arene cations[3,4]. Part B12 of the current Gmelin organoiron series, dealing with $CpFe(CO)_2X$, $CpFe(CO)_2D^+ X^-$ and $CpFe(CO)_2R$ compound types, was published[5].

3. REACTIONS OF "NAKED" IRON ATOMS AND IONS

Codeposition of iron atoms with butadiene at -190° produced evidence of $Fe(C_4H_6)$, $Fe(C_4H_6)_2$, and $[Fe(C_4H_6)]_n$[6]. With benzene or toluene, FeAn and $FeAn_2$ were indicated by IR[7]. Mössbauer studies of the latter indicated two species, thought to be a 20-electron $Fe(\eta^6-C_6H_6)_2$ and an 18-electron $Fe(\eta^6-C_6H_6)(\eta^4-C_6H_6)$[8]. Iron atoms induced [2 + 2]-cyclodimerization involving the endocyclic double bonds of the triene 1[9].



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Reactions of butanes with Fe^+ formed by several different gas-phase methods have been found to give consistent results, suggesting that all of these methods produce ground state Fe^+. Collision-induced decomposition (CID) mass spectrometry has been used to characterize products of reaction of Fe^+ and $FeCO^+$ with alkanes[10]. In reactions with n-propylamine, as with other organics, Fe^+ is less selective than other transition metal ions. This has been interpreted in terms of Fe^+ requiring a lower promotion energy to reach the valence state conducive to the insertion reactions which dominate this chemistry[11]. A Fourier transform mass spectrometric study of ferracycloalkane ions (from reaction of Fe^+ with cycloalkanones) revealed rearrangement to alkene complexes by hydrogen shifts[12].

Ion beam studies have led to a value of 59(5) kcal for the bond energy of $Fe-H^+$ and a proton affinity of 190(5) kcal for the Fe atom. Oxidative addition of H_2 to Fe is exothermic by at least 22 kcal/mol. Unlike Fe^+, FeH^+ does not undergo facile oxidative additions with organics[13]. Likewise, $FeCH_3^+$ is unreactive with alkanes[14]; it does, however, react with cyclopropane, to give $FeEt^+$ and C_2H_4, and with cyclobutane[15]. Reaction of $FeMe^+$ with cyclopentene yields $CpFe^+$ from dehydrogenation and loss of methane; this leads to an estimate that the energy of the $Cp-Fe^+$ bond must be at least 66 kcal/mol[15]. $FeCO^+$ reacted with alkenes to form $FeC_nH_{2n}^+$ species; with butene or larger alkenes, these react by insertion of Fe^+ into an allylic C-C bond,followed by hydrogen migration to form an allyliron hydride species[16].

FeO^+ has been found to be more reactive toward alkanes than Fe^+ by virtue of the exothermic formation of water[17]. Sparking halomethanes at an iron surface yielded the iron carbenes $FeCH_2^+$, $FeCHCl^+$, $FeCCl_2^+$, and $FeCBr_2^+$ and the carbynes $FeCX^+$ [18]. CID Mass spectrometric studies of Fe_2^+ and $FeCo^+$ have indicated bond energies of 69(5) and 66(7) kcal/mol, respectively[19]. Both dimetal cations are unreactive toward alkanes in the gas phase, unlike Fe^+ or Co^+. But the trimetallic $FeCo_2^+$ reacts with propane and higher hydrocarbons by dehydrogenation[20].

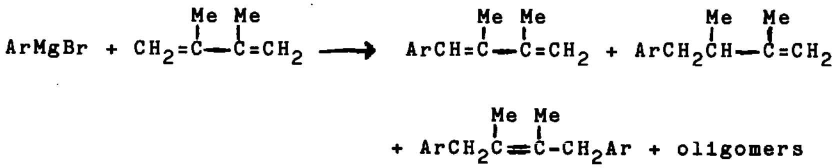
4. COMPOUNDS WITH 1-CARBON LIGANDS

a. Alkyl- and aryliron compounds. R_nFe

Several papers on dimesityliron have appeared during 1984. Reaction of $Ar_2Mg(THF)_2$ (Ar = mesityl) with $FeBr_2$ was reported to give $ArFeBr(THF)_{0.5}$[21]. Analogous results were obtained with organozinc and -aluminum compounds[22]. Reaction of dimesityliron with acetonitrile reportedly gave ArFe(N=CMeAr)(NCMe)[23]. Reaction with ethyne also proceeded with insertion, forming polyenes $Ar(CH=CH)_nAr$[24]. Reaction with two equivalents of PhC≡CPh in THF gave $(THF)_2Fe(PhC=CPhAr)_2$; with four equivalents in ether, the product was reported to be $Fe(PhC=CPhCPh=CPhAr)_2$[25].

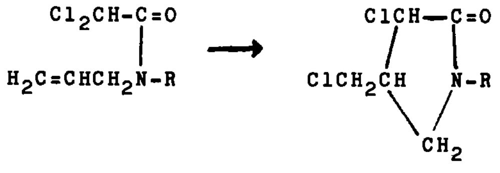
A quantitative CIDNP study of the reaction of isobutyl iodide with ethylmagnesium bromide catalyzed by $Fe(acac)_3$ has indicated that coupling of alkyl radicals with reduced iron species to form R-Fe intermediates is not an important step at usual concentrations[26].

A number of studies of organic reactions catalyzed by "inorganic" iron species, which may involve intermediates with Fe-C bonds, have been reported. $Fe(acac)_3$ catalyzed addition of various ArMgBr to dienes to produce arylated dienes and alkenes (Eq. 1)[27] and, in admixture with Ph_3P and Et_3Al, addition of methyl acrylate to 2-cyclopropylbutadiene (Eq. 2)[28]. A similar mixture catalyzed cycloaddition of norbornadiene to norbornadiene dimers to give trimers[29]. $FeCl_2$ catalyzed the cyclization shown in Eq. 3[30]. Formation of 1,5-hexadiene by (tetraphenylporphi-



$$ArMgBr + CH_2{=}\overset{Me}{C}{-}\overset{Me}{C}{=}CH_2 \longrightarrow ArCH{=}\overset{Me}{C}{-}\overset{Me}{C}{=}CH_2 + ArCH_2\overset{Me}{C}H{-}\overset{Me}{C}{=}CH_2 + ArCH_2\overset{Me}{C}{=}\overset{Me}{C}{-}CH_2Ar + \text{oligomers} \quad (1)$$

$$CH_2{=}CHCO_2Me + CH_2{=}CHCR{=}CH_2 \longrightarrow CH_3CH{=}CRCH_2CH{=}CHCO_2Me \quad (2)$$

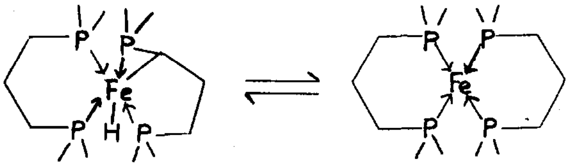


(3)

nato)iron(II)-catalyzed reduction of allyl bromide was accompanied by some allylation of the porphyrin ring[31].

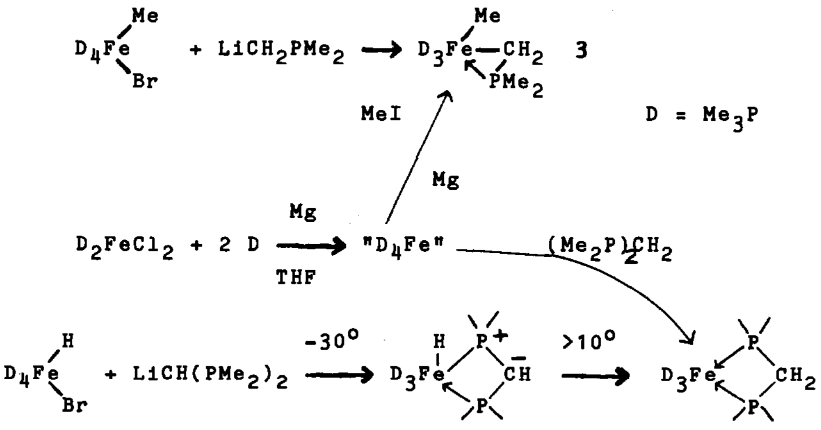
An X-ray structure of the low-spin pentacoordinate (Por)FePh, 2, (Por = tetraphenylporphyrin) showed the iron only slightly displaced from the plane of the four nitrogens, and a Fe-C bond length of 1.955(3) A[32]. Two-electron oxidation of 2 occurred with migration of the phenyl ligand to nitrogen; reduction reversed the process[33]. Binding of pyridine to 2 and its oxidized and reduced forms was studied electrochemically[34]. Sulfur dioxide inserted into the Fe-C bond of 2 to form the sulfinate (Por)FeOS(O)Ph, which has been characterized by X-ray crystallography[35]. Nitrous oxide coordinated to the vacant site of the iron atom of 2[36]. Electrochemistry of the nitrosyl porphyrin has been reported[37].

Fe(acac)$_3$ reacted with AlMe$_3$ and DMPE to form (DMPE)$_2$Fe(acac)$^+$ AlMe$_4^-$; a methyliron intermediate transferred the methyl group to AlMe$_3$ to generate the anion. Methylation of the cation gave Me$_2$Fe(DMPE)$_2$[38]. Magnesium reduction of FeCl$_2$ in the presence of Me$_2$P(CH$_2$)$_3$PMe$_2$ gave a C-H insertion product in equilibrium with a tetracoordinate 16-electron product[39]:



(4)

Reaction of D$_4$Fe(Me)Br with LiCH$_2$PMe$_2$ occurred with displacement of the bromide and a trimethylphosphine (D) to give a cyclic ferraphosphirane, 3. Reaction of LiCH(PMe$_2$)$_2$ with the hydride D$_4$FeHBr at -30° occurred with retention of the fe-H bond in the initial ylide-like product, which rearranged above 10° [40]. (Eq. 6)



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(6)

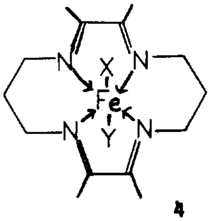
The chemistry of some related bis(diphosphine)iron hydrides is also discussed in this section. Although the substances discussed lack C-Fe bonds and are therefore not organometallics in the strict sense, their chemistry bears on that just discussed and should be of interest to organometallic chemists.

Reaction of $(DMPE)_2FeCl_2$ with $NaBH_4$ or $(DMPE)_2FeH_2$ with BH_3 gave $(DMPE)_2Fe(H)BH_4$ with an open Fe--H--BH_3 bond[41]. *Trans*-$(DPPE)_2Fe(H)N_2$ has been studied by ^{15}N and ^{31}P NMR[42]. Protonation of $(DPPE)_2FeH_2$ with the novel carbon acid Tf_2CH_2 produced molecular hydrogen and $(DPPE)_2FeH^+$ [43]. The crystal structure of the crowded dihydride, $(Ph_2MeP)_4FeH_2$ has been determined[44].

b. Iron Monocarbonyls, e.g. D_4FeCO

Trans-$HFe(CO)[P(OEt)_3]_4^+$ reacted with *p*-toluenediazonium fluoroborate to give $(ArN{=}NH)Fe(CO)D_4^{+2}$. The aryldiimine ligand was readily displaced by CO, ArNC, or ArCN[45]. Rates and equilibria of thermal ligand exchange reactions in **4** (X, Y = MeCN, H_2O, CO) have been studied[46], as has flash photolysis of **4** (X = CO, Y = MeCN, H_2O)[47]. A series of complexes $DFe(CO)(EtOCS_2)_2$ formed on carbonylation of iron(II) ethylxanthate in the presence of bases D = pyridine, pyrrolidine, triethylphosphine, etc., but most of them readily disproportionated[48].

A number of studies of iron(II) porphyrins have included addition reactions of carbon ligands such as CO, CS, and CNR. Thus, acyl isonitriles gave mono- and bis-adducts with iron(II) tetraphenylporphyrin; one isonitrile was readily displaced from the bis-adduct by bases such as pyridine, giving **5**[49].



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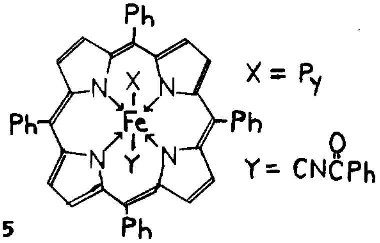

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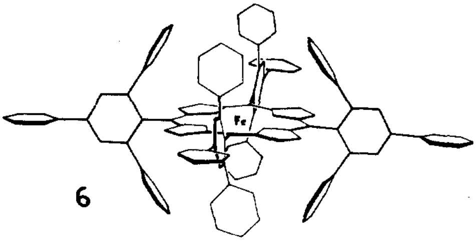
Electrochemical oxidation of tetraphenylporphinatoiron(II) thiocarbonyl, (Por)FeCS, and its nitrogen-base adducts occurred in two reversible steps, with oxidation first of iron then of the porphyrin ligand[50]. A prochiral (Por)Fe has been obtained by chemical modification of the propionic acid side chains of natural porphyrin; diastereomeric adducts with $PhCH(CH_3)NC$ and other bases were obtained[51].

Porphyrin analogs with bulky groups have been synthesized in order to assess the effects of the groups on O_2 and CO binding.

Recent examples have included 6[52] and 7[53].



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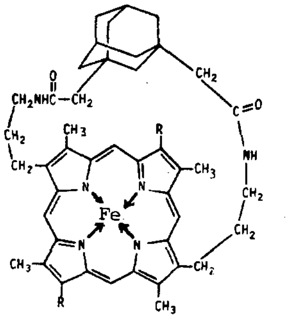

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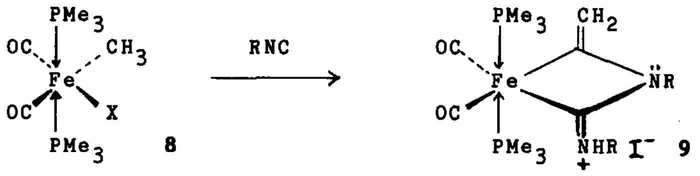
c. Iron Dicarbonyls, e.g. $D_3Fe(CO)_2$

Iron(II) diethyldithiocarbamate underwent carbonylation to give only *cis*-$(OC)_2Fe(S_2CNEt_2)_2$[48]. The 16-electron compound $(C_6H_4S_2)Fe(PMe_3)_3$ lost a phosphine ligand on reaction with CO, to give $(C_6H_4S_2)Fe(CO)_2(PMe_3)_2$ with excess CO or dimeric $[(C_6H_4S_2)Fe(PMe_3)_2(CO)]_2$ (structure not proposed) with one equivalent[54].

The primary photochemical step in photolysis of $(bpy)_2Fe(CNMe)_2^{2+}$ was found to be dissociation of an isonitrile ligand[55]. Photolysis of $Fe(CO)_2(NO)_2$ in liquid krypton containing 2% nitrogen yielded products with one and both carbonyl ligands replaced by N_2[56].

Further mechanistic studies of ^{13}CO insertion in $(OC)_2Fe(PMe_3)_2(Me)X$ compounds, 8, have been reported[57]. With X = CN the acetyl group of the product contained no label, but with X = I or SCN, ionization allowed exchange of all three carbonyl groups. 8 (X = I) reacted with cyclohexyl isocyanide to give 9[58].



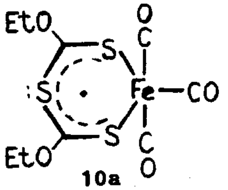
(7)

$(\eta^2\text{-}CH_2O)Fe(CO)_2D_2$ was formed by photolysis of $D_2Fe(CO)_3$ (D = triethyl- or triisopropylphosphite) followed by addition of formaldehyde; the formaldehyde ligand was displaced by ethylene, Ph_3P, CO, H_2, or MeI. With HI, $D_2Fe(CO)_2(CH_2I)(I)$ resulted[59]. Mass spectral studies of $(Ph_2AsCH_2CH_2AsPh_2)Fe(CO)_2(COMe)D^+$ BF_4^- salts by the fast atom bombardment technique have been reported[60].

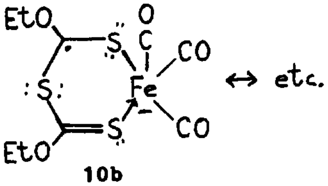
d. Iron tricarbonyls, e.g. $D_2Fe(CO)_3$

The radical-anion $Fe(CO)_3^{\dot{-}}$ could be produced in a flowing afterglow apparatus by reaction of thermal electrons with $Fe(CO)_5$. In contrast to $Fe(CO)_4^{\dot{-}}$, it readily adds H_2, CO, N_2, and alkenes[61]. The series of hydride anions $(OC)_3FeH_n^-$ (n = 1,2,3) has been characterized as resulting from reaction of $Fe(CO)_4^{\cdot -}$ with hydrogen atoms and dihydrogen[62].

Reaction of EtOC(=S)SC(=S)OEt with $Fe(CO)_5$ gave an ESR signal which was attributed to the species **10a**[63]. However, the chelate ring is unlikely to bridge two equatorial positions of a trigonal bipyramid as shown, and moreover the odd-electron species requires a charge if the stoichiometry is as shown. A structure such as **10b** may be suggested pending further characterization.



10a

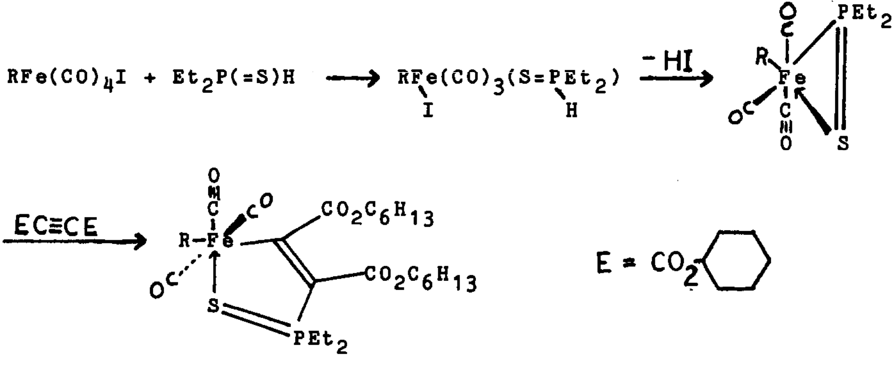


10b

mer-$(ArNC)_3Fe(DPPE)I^+I^-$ resulted from reaction of $(ArNC)_4FeI_2$ with DPPE. A monodentate perchlorate analog was formed on reaction with silver perchlorate; ClO_4^- was readily displaced by phosphines and the like[64]. Reaction of $(Ph_2P)_3CH$ (tripod) with iron carbonyls gave *inter alia* bidentate (tripod)$Fe(CO)_3$, whose free arm could coordinate to additional metals[65,66]. Similar bis-bidentate $[Fe(CO)_3]_2$ complexes were prepared using the tetraphosphorus ligand $[R_2PCH_2P(R)(CH_2)_5-]_2$ (R = isopropyl)[67].

Photolysis of (1,4-diazabutadiene)$Fe(CO)_3$ complexes in solution led to substitution of CO after breaking an Fe-N bond, but in matrices at 10 K a π-coordinated species formed[68,69]. The photochemical reactions of $HFe(CO)_3(SiEt_3)(PPh_3)$ (from photolysis of $Ph_3PFe(CO)_4$ in triethylsilane glass at 100 K) resulted both from loss of CO and from competitive reductive elimination of triethylsilane[70] .

Reaction of $Ph_3PFe(CO)_3^{2-}$ with $TfO(CH_2)_4OTf$ gave the ferracyclopentane, which was characterized by X-ray crystallography[71]. A pair of novel ferracycles, both characterized by X-ray crystallography, is shown in Eq. 8, where R = $n-C_3F_7$[72].



(8)

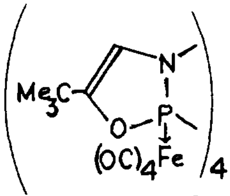
e. Iron tetracarbonyls, e.g. $DFe(CO)_4$ and $R_2Fe(CO)_4$

The non-Berry pseudorotation of triplet C_{2v} $Fe(CO)_4$, observed upon laser irradiation in matrices, has been explained in terms of a topological model and the Jahn-Teller theorem[73]. The chemistry of the radical-anion $Fe(CO)_4^{\dot{-}}$ in the gas phase has been explored, including its reactions with O_2, which gave $Fe(CO)_nO_m^-$ (n = 2,3, m = 1,2)[74]; with carbon tetrachloride, which gave $ClFe(CO)_4^-$ [75]; and with $Fe(CO)_5$, to form principally $Fe_2(CO)_8^{\dot{-}}$ [76].

$HFe(CO)_4^-$, **11**, supported in an ion exchange resin can be used to reduce acid chlorides to aldehydes under mild conditions[77]. **11** was the least reactive hydridometal ion in a comparative study, failing to react with n- or tert-butyl bromide[78]. **11** did displace a methyl group from methyltrialkylammonium ions in NMP at 200° [79]. The fact that **11** forms contact ion pairs with alkali metal cations entirely at an oxygen has been related to its low reactivity as a hydride donor[80].

SCF $X\alpha$ scattered-wave calculations on ground and excited state $H_2Fe(CO)_4$ have given results in agreement with photochemical results, including the photodissociation of H_2[81]. Reduction of coordinated N_2 by $H_2Fe(CO)_4$ has been studied[82]. Addition of $Cl_3SiFe(H)(CO)_4$ to dienes has been found to occur by a radical chain mechanism[83].

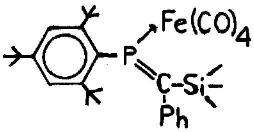
Measurement of core-electron binding energies of $DFe(CO)_4$ (D = pyridine, Me_3P, and various alkenes) has led to a better understanding of σ and π bonding in these compounds[84]. X-ray structures of several novel trivalent phosphorus-$Fe(CO)_4$ complexes have appeared. In **12**-**14**, the phosphorus ligand occupies the expected



12,Ref. 85

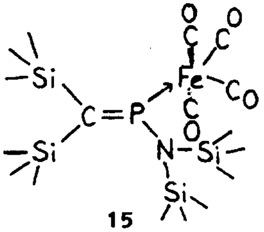
$(OC)_5MoPPh_2OPPh_2Fe(CO)_4$

13,Ref. 86



14,Ref. 87

axial site of the iron trigonal bipyramid, but a number of examples of equatorial phosphorus ligands have also appeared this year. In the cation $(Et_2N)_2P{=}Fe(CO)_4^+$, with its Fe=P bond length of 2.10(5) Å, equatorial positioning of the phosphenium ligand is a predictable consequence of its powerful π-acceptor character[88]. In **15**, the unsaturated phosphorus is also equatorial, in contrast to the seemingly similar **14**. The Fe-P distance in **15** was found to be 2.208(2) Å, and the P=C bond was twisted 30° from planarity[89]. The equatorial coordination of the unsaturated phospaalkene in **15** and of the similar diphosphene ligand in **16**[90] may be ascribed to either steric or electronic (π-acceptance) factors, but the origin of substantial π-acceptor character in the diazadiphosphetidine ring of **17**[91] is less evident.



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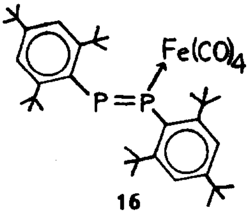

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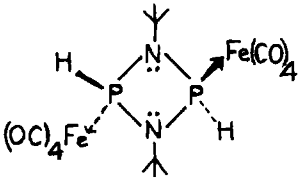


17

Reaction of the phospharsene 2,4,6-$\underline{t}$-$Bu_3C_6H_2As{=}PCH(SiMe_3)_2$ with diiron enneacarbonyl gave two $Fe(CO)_4$ complexes, one with P coordinated to Fe and one with As coordinated[90]. The $Fe(CO)_4$ complex of the distibene, $(Me_3Si)_2CHSb{=}SbCH(SiMe_3)_2$, in contrast to these cases and to **16**, was found to be a π-type complex[92].

SCF calculations have indicated that the reactivities of nucleophiles toward carbonyl attack on $Fe(CO)_5$, forming $NuC({=}O)Fe(CO)_4^-$, should $Me^- > OH^- > H^-$, although the iron-carbon bond strengths in the products should decrease in the order formyl > acetyl > hydroxycarbonyl[93]. $(H)(MeCO)Fe(CO)_4$ was calculated to be 12.6 kcal more stable than $(Me)(HCO)Fe\ (CO)_4$[94]. These calculations were consistent with the relative ease of CO insertion into Fe-H and Fe-Me bonds.

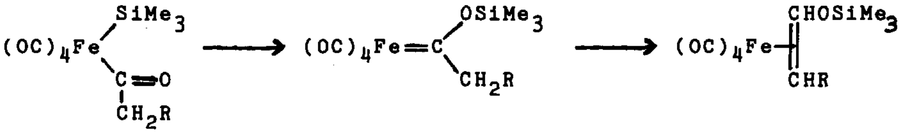
Experimentally, hydrated OH^- has been found to react with $Fe(CO)_5$ in the gas phase at or near the collision rate to produce $(OC)_4FeCOOH^-$ [95]. Electroreduction of $Fe(CO)_5$ in the presence of Bu_3SnH produced the formyliron anion[96]. ESR and extended Huckel MO studies of acyl- and alkyliron tetracarbonyl radicals have been carried out[97]. The acyliron radicals were found to have a trigonal bipyramidal geometry with the acyl ligand in an axial position. The alkyliron radicals were labile with respect to alkyl migration and exchange of equatorial CO ligands with phosphites.

$Me_3SiFe(CO)_4^-$ salts underwent alkylation with primary alkylating agents and acylation, but the products were of limited stability. The acyl(trimethylsilyl)tetracarbonyls underwent an interesting rearrangement to carbene complexes[98]. (see Eq. 9, next section). $MeOC({=}O)CH_2Fe(CO)_4^-$ resulted from reaction of MeO^- with $Fe_2(\mu\text{-}CH_2)(CO)_8$, as verified by an X-ray structure of the PPN^+ salt[99]. A structure has also been published for the zwitterion $Ph_3P^+CH_2Fe(CO)_4^-$; the Fe-C-P bond angle is widened to 119° [100]. Both of these alkyltetracarbonylferrate[-I] derivatives had axial alkyl groups in an iron trigonal bipyramid.

Reaction of α,α'-dibromo-$\underline{p}$-xylene with tetracarbonylferrate[-II] was reported to give the dialkylferrate, $(BrCH_2C_6H_4CH_2)_2$-$Fe(CO)_4$[101]. An intramolecular analog, a (tetracarbonyl) benzoferra-3-cyclopentene, was similarly reported from the o-xylene[102]. Similar reactions were reported for $Fe(CO)_4{}^{2-}$ generated from iron pentacarbonyl under phase transfer conditions; benzyl bromides could be converted to dibenzylketones using this reagent[103].

f. Carbene Complexes, e.g. $R_2C{=}Fe(CO)_4$

Rearrangement of the silyl group to the acyl oxygen in (trimethylsilyl)(acyl)tetracarbonyliron compounds generated carbene complexes, which underwent further rearrangements to alkene complexes (Eq. 9)[98]. Two types of carbene complexes, both characterized by X-ray crystallography, result from reaction of $Fe(CO)_5$ with $LiNR_2$ (R = isopropyl) and then with triethyloxonium fluoroborate (Eq. 10)[104]. **18** reacted with PPh_3 by displacement of a CO;



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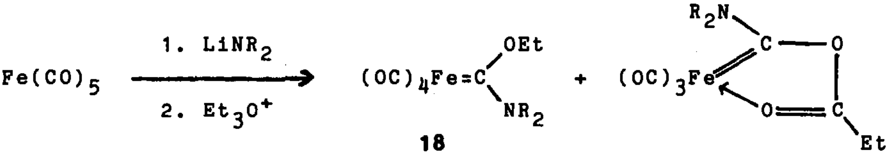
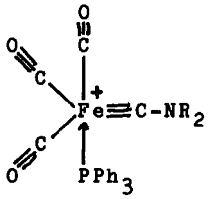


(10)

BCl_3 abstracted OEt^- from the resulting complex to give a carbyne complex, **19**, with a very short Fe≡C bond (1.73 Å)[105]. A ferracyclic bis-carbene, **20**, resulted from reaction of hydrazine with the isonitrile ligands of $(bpy)_2\overset{+2}{Fe}(CNMe)_2$[106].

19



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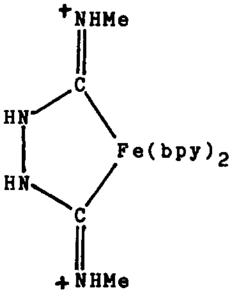
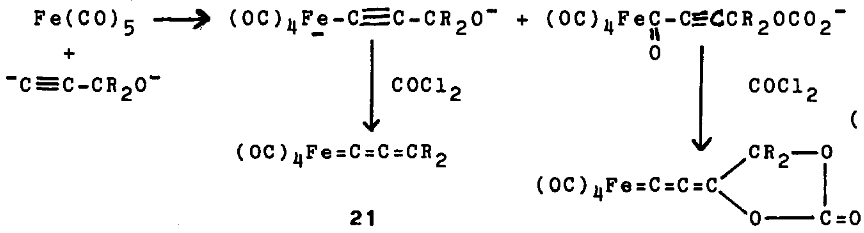


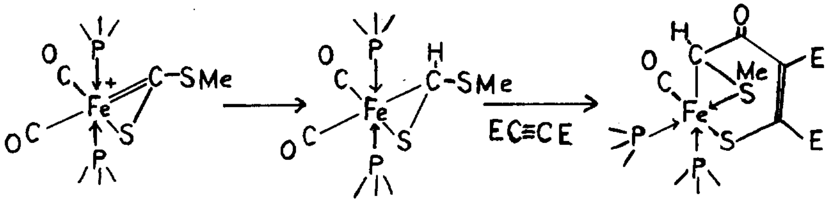
Two allenylidene complexes (Eq. 11) resulted from reaction of $Fe(CO)_5/Me_3NO$ with $^-C\equiv C\text{-}CR_2O^-$ (R = t-butyl), followed by treatment with phosgene to deoxygenate[107].



(11)

Reaction of $(OC)_4Fe=C(R)OEt$ with various alkynes under CO pressure occurred by initial [2 + 2] cycloaddition to form a ferracyclobutene; a sequence of migration and insertion steps led to $Fe(CO)_3$ complexes of 6-ethoxy-α-pyrones as the final products. (See Eq. 19)[108].

The chemistry of carbene complexes derived from $(Me_3P)_2Fe(CO)_2(\eta^2-CS_2)$, 22, has been further explored. The carbene complex from methylation of the free sulfur of 22 underwent hydride reduction at carbon to give a complex of methyl dithioformate; this in turn reacted with dimethyl acetylenedicarboxylate (DMAD) by a complex rearrangement (Eq. 12)[109]. The metal-dithiocarbene intermediate resulting from reaction of 22 with DMAD was stabilized by reaction as a Lewis base with $HgCl_2$; the X-ray structure of the adduct revealed the $HgCl_2$ unit coordinated to the iron, trans to the dithiocarbene[110]. Reaction of 22 (PPh_3 instead of PMe_3) with $Ph_2PC\equiv CCMe_3$ in methanol gave the carbene complex 23, several further reactions of which were also reported[111].



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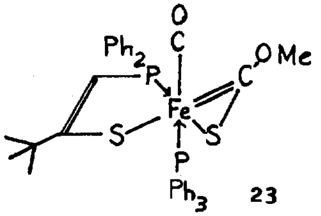


23

Reaction of tetraarylporphinatoiron(III) compounds, (Por)FeCl, with $RCCl_3$ and sodium dithionite resulted in formation of (Por)Fe=CClR; the chloride could be replaced by alkoxide or thioalkoxide groups[112]. The carbido complexes (Por)Fe=C=Fe(Por) have been studied by resonance Raman spectroscopy[113], and electrochemically, the latter indicating binding of axial pyridine ligands[114].

g. Some Selected Reactions and Properties of $Fe(CO)_5$

A pulsed laser pyrolysis technique has indicated a first Fe-CO bond dissociation energy of 41(2) kcal/mol for $Fe(CO)_5$[115]. IR laser pyrolysis has been applied in preparation of substitution products incorporating ^{13}CO, PF_3, NO, and butadiene from $Fe(CO)_5$ under mild conditions[116].

γ-irradiation of $Fe(CO)_5$ in a low-temperature krypton matrix produced $KrFe(CO)_5^+$ [117]. Photolysis of $Fe(CO)_5$ in CCl_4 resulted in formation of $FeCl_2$, C_2Cl_4 and "tar"; a reaction sequence involving $Fe(CO)_4$, $(OC)_4Fe(Cl)CCl_3$, and the carbene complex $(OC)_4Fe{=}CCl_2$ has been proposed[118].

Reaction of $Fe(CO)_5$ with 7-arylnorbornadienes gave dimeric cyclopentanones whose stereochemistry was determined by X-ray crystallography[119]. $Fe(CO)_5$ served as a precatalyst in the hydrogenation of PhCH=NPh at 150° [120]. Both $Fe(CO)_5$ and $Fe(CO)_4(CS)$ formed Lewis acid-base adducts with $HgCl_2$ in pentane. In ethanol, $(OC)_4Fe(HgCl)_2$ and $(OC)_4Fe(HgCl)[C({=}S)OEt]$, respectively, resulted. In water the thiocarbonyl gave $[HgFe(CO)_3(CS)]_x$[121].

MO calculations on $Fe(CO)_5^{\dot{-}}$ favor a square pyramidal structure in which a bent apical Fe-C-O ligand eclipses one of the basal ones[122].

5. η^2-ALKENE AND η^3-ALLYL COMPLEXES

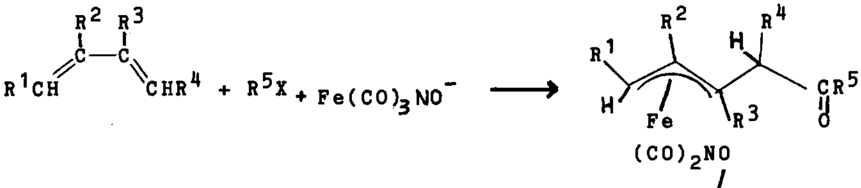
Calculations using the PRDDO method have shown the strength of binding of C_2X_4 to $Fe(CO)_4$ to decrease with X in the order Cl>CN>>F,H; the localized iron-alkene bonding was consistent with a ferracyclopropane representation[123]. Core-electron binding energies of several alkene-$Fe(CO)_4$ complexes have been measured, and the results were generally consistent with the theoretical picture[84].

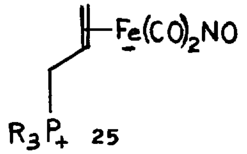
Ethylene complexes could be obtained in good yield by reaction of $DFe(CO)_3^{2-}$ (D = CO, PPh_3) with $TfOCH_2CH_2OTf$; the X-ray structure of $(C_2H_4)Fe(CO)_3PPh_3$ was reported[71]. Reaction of $Na_2Fe(CO)_4$ with β-bromostyrene was said to result in formation of $(PhCH{=}CHBr)Fe(CO)_4$[124], but the nature of the reduction product(s) which must accompany this product was not determined.

$(C_2H_4)Fe(CO)_4$ was found to be an effective precatalyst for hydrogenation of ethylene in the gas phase under UV irradiation. A turnover rate of 900 sec^{-1} was determined; the effective catalyst, thought to be $(C_2H_4)Fe(CO)_3$, owes its efficacy to a slow rate of recombination with CO[125]. Tricarbonylbis(cyclooctene)iron has been found to be a versatile source of $Fe(CO)_3$ units in solution; thermal reaction with 1-pentene led to rapid equilibration with 2-pentenes, contradicting previous assignment of the active catalyst in these isomerizations as an $(alkene)_2Fe_2(CO)_6$ species. With 2-butyne, coupling products resulted, and with dienes and styrenes, stable η^4-$Fe(CO)_3$ complexes[126].

A crystal structure of $[(\eta\text{-}C_3H_5)Fe(CO)_3]_2$, from reaction of allyl chloride with $Fe_2(CO)_9$, has been reported[127]. $(\eta\text{-}C_3H_5)$-$Fe(CO)_3Br$ in compressed disks showed electrical semiconduction when doped with I_2[128].

Reaction of $(\eta\text{-}C_3H_5)Fe(CO)_2NO$ (**24**) with trivalent phosphorus compounds gave different results depending on the nature of the nucleophile. Ph_3P and trialkyl phosphites reacted by displacement of the double bond, giving η^1-allyliron products, but trialkylphosphines attacked the allyl group, resulting in formation of zwitterionic adducts **25**[129]. **24** reacted with (S)-$Ph_2PNHCHMePh$ by CO displacement, giving separable diastereomers, whose absolute configurations were determined by X-ray crystallography[130]. Reactions of **24** and several alkyl derivatives with allylic, propargylic, and acyl halides resulted in formation of C-C bonds; further synthetic use of these complexes was suggested by reactions such as those shown in Eq. 13[131].

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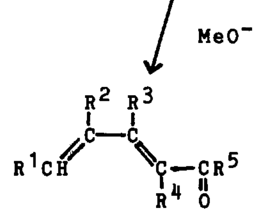

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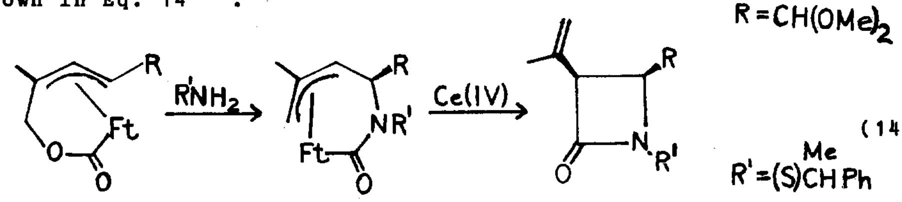
Alkenyloxiranes were converted to lactone complexes upon reaction with $Fe_2(CO)_9$[132]. This reaction has found application in a formal synthesis of (+)thienamycin, the key steps of which are shown in Eq. 14[133].



(14)

A novel bis(η^3-pentadienyl)bis(trimethylphosphine)iron (X-ray structure reported) was obtained by reaction of potassium pentadienide with $(Me_3P)_2FeCl_2$[134].

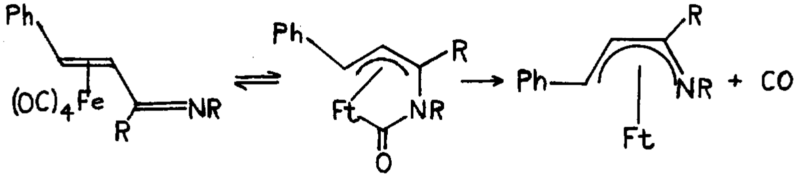
6. COMPOUNDS WITH η^4-LIGANDS

a. Complexes of Heterodienes

Reaction of naturally-occurring optically active enones with $Fe(CO)_5$ under irradiation or with $Fe_2(CO)_9$ gave (enone)$Fe(CO)_3$ complexes, which transferred the $Fe(CO)_3$ group to prochiral dienes with significant asymmetric induction[135].

Electrochemical oxidation of the complexes (PhCH=CHCOMe)-$Fe(CO)_2D$ (D = CO, PPh_3, PEt_3, and $P(OEt)_3$) in DMF has been studied[136]. These compounds have been found to be fluxional in solution, with D alternatively occupying axial or basal positions in the square pyramidal structures[137]. A kinetic study of their reactions with cyclohexadiene indicated dissociation of the ketone group as the rate-controlling step, with the tricarbonyl complex the most reactive[138].

The equilibration between (iminoalkene)$Fe(CO)_4$ complexes and their cyclic lactam tautomers (Eq. 15) has been studied[139], as has their conversion to (η^4-azadiene)tricarbonyliron complexes[140].



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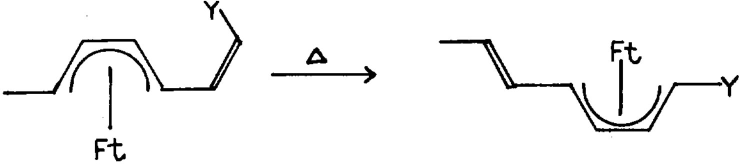
b. Complexes of Acyclic Dienes

Addition reactions of carbanions to (diene)tricarbonyliron complexes at -78° occurred primarily at an internal position (kinetic control); the resulting (homoallyl)$Fe(CO)_3^-$ complexes rearranged at 0° to more stable allyl complexes[141].

A number of studies have dealt with physical measurements on butadieneiron complexes. ^{13}C and ^{17}O T_1 studies and $^{13}C\{^1H\}$ NOE measurements of the tricarbonyl have been reported[142]. Phase transitions of $(C_4H_6)Fe(CO)_3$ physisorbed on BN have been studied by optical spectroscopy[143]. Single crystals of $(C_4H_6)_2FePMe_3$ have served as host for EPR study of the manganese compound[144,145]. Polymers with pendant (diene)tricarbonyliron groups were found to display semiconduction after doping with iodine or NO^+ SbF_6^- [128].

Tricarbonylbis(cyclooctene)iron has been suggested for use in synthesis of tricarbonyliron complexes of dienes and styrenes[126].

Silylated dienes, $Me_3SiCH=CH-CH=CR_2$, gave tricarbonyliron complexes in 40-80% yields, using $Fe(CO)_5$ or $Fe_2(CO)_9$[146]. (2,4-Hexadienal)tricarbonyliron reacted with $Ph_3P=CHY$ (Y = CO_2R, CN, COPh) to form *cis*- and *trans*- triene complexes. Upon heating to 120°, the *cis* isomers isomerize to *trans*, and the iron group shifts along the chain (Eq. 16). TCNE reacts with the products in methanol solution by dipolar [2 + 2] cycloaddition to the free double bond[147].



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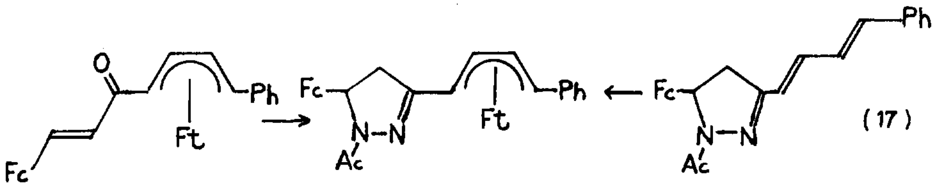
Asymmetric induction in formation of pyrazolines (Eq. 17) has been studied; in the process, crystal structures of two diastereomeric pyrazolines bearing (phenylbutadiene)tricarbonyliron and ferrocenyl substituents have been determined[148]. Functional group manipulations on resolved ($HCOCH=CH-CH=CHCO_2Me)Fe(CO)_3$ have been used to prepare free dienes (e.g. MeCH(OMe)CH=CH-CH=CHCH(OMe)Me in high stereochemical purity[149,150].

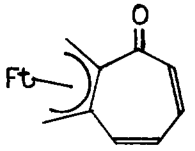


(17)

c. Complexes of Exocyclic Dienes

Conversion of 1,2-bis(bromomethyl)benzene to (*o*-xylylene)tricarbonyliron has been reported on by two groups. One used iron pentacarbonyl and calcium hydroxide in a phase-transfer system[103], and the other used conventionally-prepared disodium tetracarbonylferrate; in the latter case, a ferracyclopentene intermediate was reported[102].

Synthesis of the "tropoquinodimethane" complex **26** has been reported[151]. The effect of the *exo*- and *endo*-iron tricarbonyl groups in coordinated 5,6-dimethylidenebicyclo[2.2.2]octenes and 5,6-dimethylidenebicyclo[2.2.1]heptenes on the stereochemical outcomes of several chemical reactions has been studied. Perhaps the most interesting result is that the *endo*-$Fe(CO)_3$ group in **27** (Y = CH_2, C_2H_4) prevents base-catalyzed exchange of the *endo* C(3)-H; this phenomenon has been used to revise some previous stereochemical assignments[152]. Temperature-dependent CD spectra of some derivatives of **27** (Y = CH_2) have been reported[153]. Formation of **27** (Y = O) has been reported[9].



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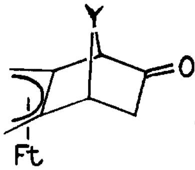

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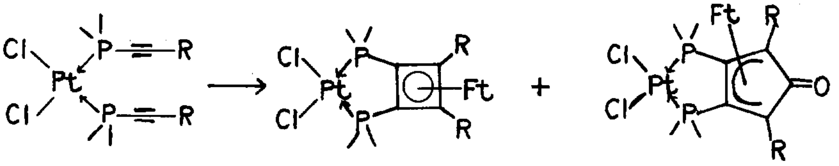
d. Complexes of Cyclic Dienes

MO calculations have compared the relative propensity of the isolobal fragments $Fe(CO)_3$ and CoCp to bind to cyclobutadiene and cyclopentadienone. The iron fragment was found to bind more strongly to the cyclopentadienone, consistent with the greater role of back-donation in the iron complexes[154]. Reaction of $(C_4Ph_4)Fe(CO)(PPh_3)NO^+$ with PPN^+X^- gave a series of products, $(C_4Ph_4)FeX(NO)(PPh_3)$. With chelating anions, both CO and PPh_3 were displaced, giving $(C_4Ph_4)FeX(NO)$; an X-ray structure for the product with X = Et_2NCS_2 was reported[155]. Reaction of *cis*-$Cl_2Pt(Ph_2PC{\equiv}CCMe_3)_2$ with $Fe_2(CO)_9$ gave the cyclobutadiene complex (Eq. 18), but other groups in place of the *t*-butyl group gave free and complexed cyclopentadienones instead[156].



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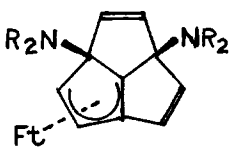
A series of complexes **28** have been prepared by complexation of the free ligands, and their reduction reactions have been reported[157]. The phosphole complex **29** underwent a number of reactions (oxidation, sulfurization, quaternization, and coordination with $W(CO)_5$) at the phosphorus atom without disruption of the diene-iron bonding. Ring expansion to **30** was accomplished by treatment with benzoyl chloride, triethylamine, then water[158].



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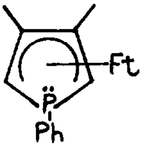

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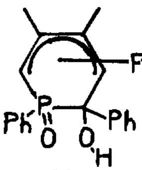
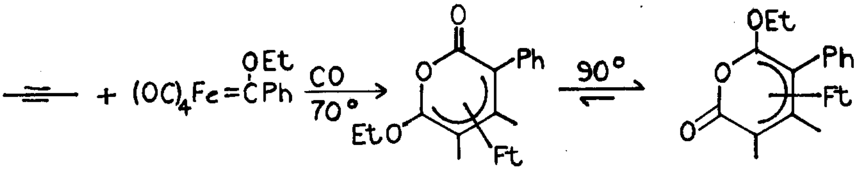


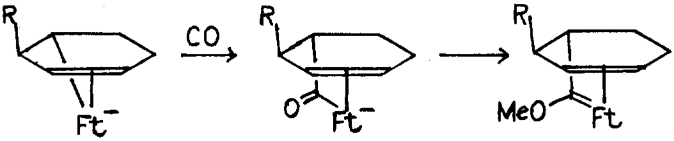
30

Irradiation of $Fe(CO)_5$ dissolved in neat N-alkoxycarbonyl-1,2-dihydropyridine gave good yields of $Fe(CO)_3$ complexes[159]. Complexes of α-pyrones resulted from reaction of carbene complexes $(OC)_4Fe{=}C(R)OEt$ with alkynes at 70° and 4 atm. CO; an example appears as Eq. 19[108].



(19)

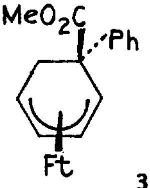
Treatment of the homoallyl anion **31**, from attack of $LiCMe_2CN$ on (cyclohexadiene)tricarbonyliron at -78°, with CO resulted in formation of an acylferrate, which could be methylated to form a bicyclic carbene complex (Eq. 20)[160].



31

(20)

Several 2-(trimethylsilyl)cyclohexa-1,3-dienes underwent complexation with $Fe(CO)_5$ in refluxing dibutyl ether without rearrangement[161]. X-ray structures of the cyclohexadiene complexes 32[162] and 33[163] have been reported.



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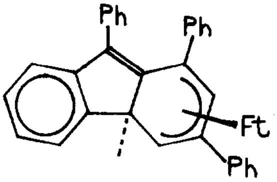
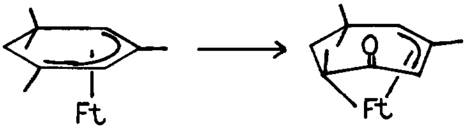


33

Acetylation of (cyclohexadiene)$Fe(CO)_2PPh_3$ under mild Friedel-Crafts conditions gave the 5-*endo*-acetyl product in 96% yield[164]. Treatment of various (cyclohexadiene)$Fe(CO)_3$ complexes with $AlCl_3$ and CO gave ring-expanded products, as shown in Eq. 21[165]. In the carbonium ion rearrangement of Eq. 22, clean retention of D implicated an iron-mediated hydride migration[166].



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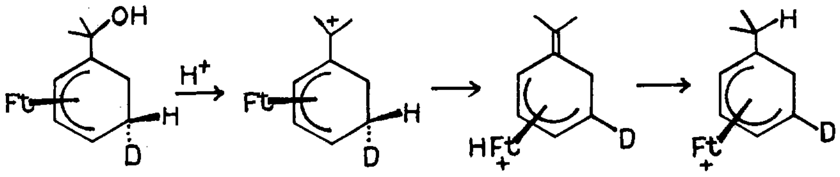

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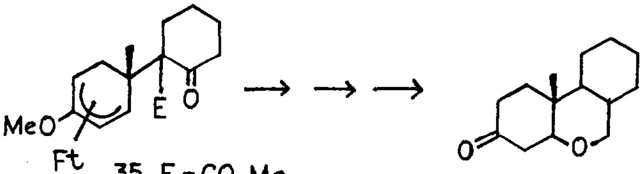
(22)

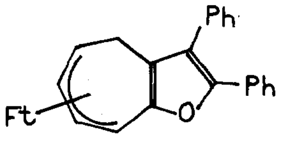
Resolved 34 (H instead of D) served as starting material in a highly stereocontrolled synthesis of (-)-gabaculine, in which the key step was specific abstraction of hydride from the exo-5-position of 34, followed by attack of a nucleophile at the same position[167]. A further use of a cyclohexadiene complex in synthesis was the conversion of 35 to an oxaperhydrophenanthrene[168]:



35, E = CO_2Me

(23)

(Cycloheptatriene)$Fe(CO)_3$ underwent normal cyclopropanation of the free double bond with ethyl diazoacetate/copper, but with $PhCOC(=N_2)Ph$ the furan 36 (X-ray) was obtained[169]. Cycloaddition of $(C_7H_8)Fe(CO)_3$ with methylphenylketene gave 37, whose endo phenyl group was consistent with a concerted [2s + 2a] cycloaddition mechanism. The X-ray structure of 37 showed a long [1.590(3) Å] bond, which was ruptured in acid or on thermal rearrangement to form a σ,π-allylic isomer[170]. The $Fe(CO)_3$ complex of 7-styrylcycloheptatriene underwent reaction with the electrophiles H^+ and TCNE at the uncoordinated C(6), analogous to the reactions of the parent $(C_7H_8)Fe(CO)_3$[171].



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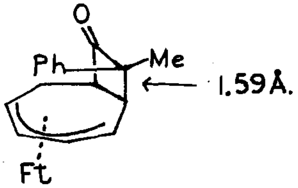

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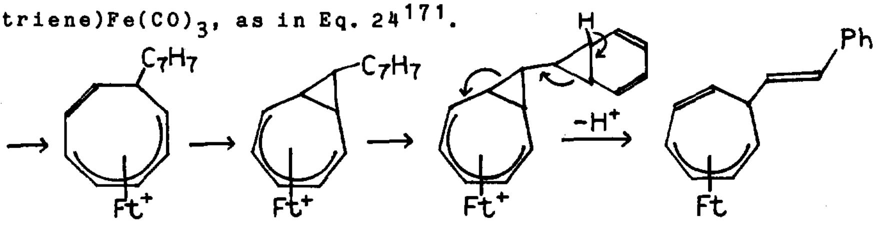
Reaction of dimethyl cyclooctatetraene-1,8-dicarboxylate with iron carbonyls gave, in addition to the known (3-6)η^4-$Fe(CO)_3$ complex, a (1-4)η^4;(5-8)η^4-bis-$Fe(CO)_3$ complex[172]. 2,3-Bis(carbomethoxy)-9,10-dichlorobicyclo[6.2.0]deca-2,5-diene was said to form a $Fe(CO)_3$ complex of the two isolated double bonds without rearrangement[173].

Electrophilic attack of tropylium ion on $COTFe(CO)_3$ proceeded with contraction of both rings to yield (7-styrylcycloheptatriene)$Fe(CO)_3$, as in Eq. 24[171].



(24)

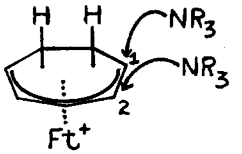
Trans, trans, trans- and *trans, trans, cis*-cyclododecatrienetricarbonyliron have been reported formed from photochemical reaction of the all-*trans* triene with $Fe(CO)_5$[174].

7. η^5-DIENYL COMPLEXES

a. Compounds with Open Pentadienyl Ligands

Interconversion of $Fe(CO)_3$-complexed cyclohexadienes and cyclohexadienyl cations by hydride removal from the former and nucleophilic attack on the latter continues to be of synthetic interest, and some examples have already been described in section 6d. INDO calculations and photoelectron spectra on the cations have indicated a stabilizing interaction between electron-withdrawing groups on the 1- and 3-positions of the cyclohexadienyl cation and the LUMO, and results on silyl-substituted cations were interpreted in terms of this interaction[61]. Results of extended Hückel calculations and crystal structures of two unsymmetrically substituted cations have led to emphasis on unequal metal bonding to the two ends of the dienyl system to explain regioselectivity in nucleophilic attack[175].

A kinetic comparison has revealed that (cyclohexadienyl)-$Fe(CO)_3$ cations are 16-74 times more reactive to nucleophilic attack by amines and phosphines than the cycloheptadienyl analogs. Steric retardation of 1-*exo* attack in the latter was proposed (see 38); this phenomenon also explains the sometime occurrence of 2-*exo* attack on the cycloheptadienyl cations, but not the cyclohexadienyl[176]. Attack of some simple anions on these cations gives 5-*exo* adducts by kinetic control, followed in some cases by rearrangements to the *endo* isomer (with NO_2^-), linkage isomerization (with SCN^-) or metal attack (with I^-)[177]. Reactions of nucleophiles with the (cycloheptadienyl)$Fe(CO)_2PY_3$ cations (Y = Ph and OPh) proceeded in many cases similarly: attack at C(1) occurred with thiophenoxides, organocuprates, and enolates. However, harder nucleophiles (cyanide, alkyllithium and Grignard reagents) preferentially attacked C(2)[178].



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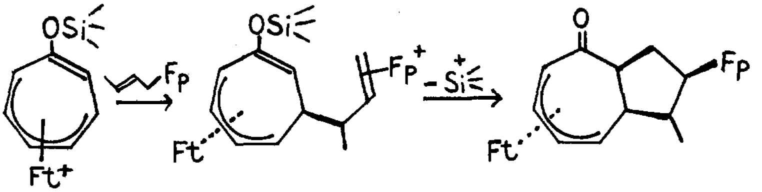
Barriers to rotation of the $Fe(CO)_3$ group in substituted cyclohexadienyl complexes have been measured and interpreted primarily in terms of steric effects[179]. Reorientation of C_6H_7-

$Fe(CO)_3$ cations in crystals of the fluoroborate salt has been studied by ^{57}Fe Mossbauer and ^{13}C-NMR spectroscopy[180]. Circular dichroism measurements have proven useful in assigning absolute configurations to resolved dienyl cations and diene complexes[181].

Attack of a β-ketoester enolate on the 2-methoxycyclohexadienyl cation was the key step in a formal alkaloid synthesis[182]. Reaction of the organoiron nucleophiles, $FpCH_2CH=CH_2$ and several derivatives, with (cycloheptatrienyl)$Fe(CO)_3^+$ derivatives provided a one-step synthesis of the azulene skeleton, as shown in Eq. 25[183].



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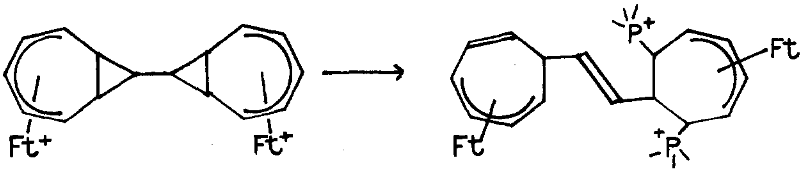
The isonitrile-substituted complexes (1,3-cyclohexadiene)- and (1,3-cycloheptadiene)$Fe(CO)_2(CNR)$, like their tricarbonyl analogs, formed dienyl cations by hydride abstraction using trityl fluoroborate. The cations were fluxional, with the isonitrile ligand favoring a basal position at low temperature. Protonation of (COT)$Fe(CO)_2(CNR)$ reversibly yielded the bicyclo[5.1.0]octadienyl cation[184]. The 8,8'-bis(bicyclo[5.1.0]octadienyl) dication, 39, simply added one mole of Bu_3P to each unit, but with Ph_3P opening of the cyclopropane rings occurred (Eq. 26)[185].



(26)

^{57}Fe Mossbauer studies of homoleptic bis(pentadienyl)iron compounds have suggested greater electron density at iron than in ferrocene[186].

b. Cyclopentadienyldicarbonyliron Hydride and Related Compounds

Free $CpFe(CO)_2^{\cdot}$ (Fp•) has been detected in cyclohexane solution using fast time-resolved IR spectroscopy, after photolysis of Fp_2. Its lifetime was less than 25μsec[187].

Baird has reviewed the literature describing FpH and concluded that it is not "unduly thermally labile." It underwent rapid CO displacement by phosphines, possibly through a radical chain mechanism involving 17-electron Fp•. Additions to double bonds were also described[188]. Fenske-Hall MO calculations on various metal hydrides supported the generalization that FpH could serve as a source of either H^- or H^+ under appropriate circumstances[189].

Reaction of $(\eta\text{-}C_6H_6)Fe(PMe_3)_2$ with CpH or Cp^*H at room temperature gave $CpFe(PMe_3)_2H$ or $Cp^*Fe(PMe_3)_2H$ directly in good yield. These reacted with CH_2Cl_2 at room temperature to give the chlorides, $Fp(^*)Cl$[190].

FpH and analogs have been most commonly prepared by reaction of a cation such as $FpCO^+$ with a hydride reagent, a reaction extensively studied during 1984. Reaction of $FpCO^+$ with NaH, $NaBH_4$, or $LiEt_3BH$ at -80° gave FpCHO, which decarbonylated to FpH upon raising the temperature[191]. Likewise, $LiAlH_4$ reduction of $CpFe(DPPE)CO^+$ at -78° proceeded via the formyl to the hydride CpFeH(CO)(DPPE), having a monodentate DPPE. In refluxing THF, the products included the Fe-Me product, and $(\eta^4\text{-}C_5H_6)Fe(CO)(DPPE)$[192]. Reaction of $Cp^*Fe(DPPE)CO^+$ with $LiAlH_4$ at -80°, in contrast, was found to proceed by single electron transfer, then H-atom abstraction, to give the analogous $Cp^*FeH(CO)(DPPE)$, but no formyl intermediate appeared to intervene[193]. In $LiAlH_4$ reductions of various $CpFeD_2CO^+$ cations, the outcome depended on the nature of the donor ligands, D. PPh_3 gave similar results as DPPE (described above), but the Fe-Me product predominated with DPPM and DMPE. $FpPPh_3^+$ gave ring reduction and the hydride, $CpFe(CO)(PPh_3)H$; $FpPMe_3^+$ gave the Fe-Me product via the formyl[194].

Oxidation of Fp^*_2 with ferricenium ion in various solvents gave the cations $Fp^*(solvent)^+$, from which the solvent was readily displaced by CO, phosphines, and the like. Reaction of the THF-solvated cation with $NaBH_4$ gave the hydride[195].

A study of the hydroformylation of alkenes in the presence of Fp_2 has revealed FpH to be present during reaction, and it was proposed that the product-forming step was reaction of FpH with FpC(=O)R to give aldehyde and Fp_2[196]. The analogous hydride was indicated as an intermediate in thermolysis of $CpFe(CO)PPh_3C(=O)OH$[197]. Fp^*H reduced Fp^*CO^+ to Fp^*CH_2OH and thence to Fp^*Me. Under CO pressure, the $Fp^*(THF)^+$ oxidation product recycled to Fp^*CO^+ [198].

The long-known reduction of Fp_2 has received careful study. The initially formed $Fp_2^{\cdot-}$ radical anion cleaved to $Fp^{\cdot}$ and Fp^- at a rate of 1060 sec^{-1} at 0°; the E_a was 15.7 kcal/mol[199]. The anion Fp_*^- reacted with $(C_5Me_5)Ru(CO)_2CH_2Cl$ by a redox reaction which formed Fp^*_2[200].

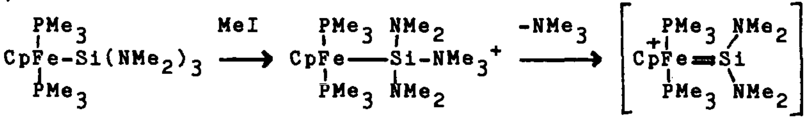
Several papers on direct conversion of Fp_2 dimers into $FpCO^+$ and related cations have appeared. In the parent system, bromine with excess aluminum chloride[201] and ferric sulfate under a CO stream[202] have been recommended for synthesis of the tricarbonyl cation. Ferricenium ion in THF has been used to oxidize $\mathrm{Fp_2}$ or $\mathrm{Fp^*{}_2}$ to the solvated cations $\mathrm{Fp(^*)(THF)^+}$, from which the solvent molecule was readily displaced by normal donor ligands[195, 203, 204].

Photolysis of $\mathrm{FpCO^+}$ in MeCN in the presence of donor ligands gave $\mathrm{CpFe(NCMe)_2D^+}$ or, in the cases of $\mathrm{Me_3P}$ and DPPE, the bis-(phosphine)(NCMe)$^+$ cations. $\mathrm{CpFe(PR_3)_3{}^+}$ cations could be obtained using excess phosphine in the cases of $\mathrm{Me_3P}$, 1-phenyl-3,4-dimethylphosphole, or $\mathrm{MeC(CH_2PPh_2)_3}$[205]. Thermolysis or photolysis of $\mathrm{FpCO^+}$ in the presence of isonitriles led likewise to replacement of two or three CO's by CNR[206]. Reaction of CpFe(DPPE)CN with $\mathrm{Me_3O^+}$ gave $\mathrm{CpFe(DPPE)CNMe^+}$ [207]. The X-ray structure of (-)-CpFe-(CO)(Norphos)$^+$ $\mathrm{PF_6{}^-}$ has been determined [Norphos = 2R,3R-bis(diphenylphosphino)norbornane][208].

A number of reports on conversion of η-cyclopentadienyl rings to η^4-cyclopentadiene complexes by attachment of nucleophiles to a ring carbon have appeared. Thus, in contrast to the results obtained with the more reactive hydride reagents described above, $\mathrm{FpCO^+}$ reacted with $\mathrm{NaBH_3CN}$ by exo attack to form $\mathrm{(C_5H_6)Fe(CO)_3}$[191]. $\mathrm{LiP(SiMe_3)_2}$ reacted similarly, but the resulting 5-*exo*-phosphino-η^4-cyclopentadiene complex rearranged at room temperature to $\mathrm{FpP(SiMe_3)_2}$[209]. Formation of $\mathrm{(C_5H_6)Fe(DPPE)CO}$ by $\mathrm{LiAlH_4}$ reduction of the corresponding cation was previously mentioned[192]; $\mathrm{FpPPh_3{}^+}$ reacted in part in the same way[194].

$\mathrm{(Ph_5C_5)Fe(CO)_2{}^-}$ has been prepared directly from $\mathrm{LiC_5Ph_5}$ and $\mathrm{Fe(CO)_5}$; methylation and bromination occurred normally[210]. FpHgX and $\mathrm{Fp_2Hg}$ have been studied by ^{57}Fe Mossbauer and ^{199}Hg NMR methods[211], and $\mathrm{Fp_2Zn}$ by photoelectron spectroscopy[212].

The structures of $\mathrm{FpSiCl_3}$ and $\mathrm{FpSiFPh_2}$ have been determined; the Fe-Si bond lengths were 2.216(1) Å and 2.278(1) Å, respectively[213]. Reaction of $\mathrm{FpSiBr_3}$ with dimethylamine gave $\mathrm{FpSi(NMe_2)_3}$. Replacement of the CO's by $\mathrm{Me_3P}$ then reaction with MeI to form $\mathrm{CpFe(PMe_3)_2I}$ may have proceeded through a silylene complex (Eq. 27)[214]:

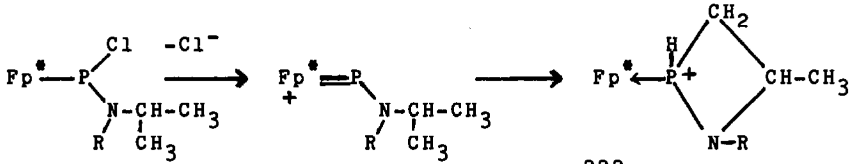


$$\underset{\mathrm{PMe_3}}{\overset{\mathrm{PMe_3}}{\mathrm{CpFe}}}\mathrm{-Si(NMe_2)_3} \xrightarrow{\mathrm{MeI}} \underset{\mathrm{PMe_3}}{\overset{\mathrm{PMe_3}}{\mathrm{CpFe}}}\text{—}\underset{\mathrm{NMe_2}}{\overset{\mathrm{NMe_2}}{\mathrm{Si}}}\mathrm{-NMe_3{}^+} \xrightarrow{\mathrm{-NMe_3}} \left[\underset{\mathrm{PMe_3}}{\overset{\mathrm{PMe_3}}{\overset{+}{\mathrm{CpFe}}}}{=}\underset{\mathrm{NMe_2}}{\overset{\mathrm{NMe_2}}{\mathrm{Si}}}\right] \qquad (27)$$

Photolysis of $\mathrm{FpSiH_3}$ in the presence of isonitrile or phosphine donors resulted in stepwise CO substitution. $\mathrm{CpFe(CNR)(CO)SiH_3}$ reacted with $\mathrm{Co_2(CO)_8}$, by replacement of a bridging CO with a bridging $\mathrm{CpFe(CNR)(CO)\underset{/\ \backslash}{Si}H}$ group[215]. Fp-substituted cyclosiloxanes resulted from reaction of $\mathrm{[-Si(Me)(H)O-]_n}$ with $\mathrm{Fp_2}$[216,217].

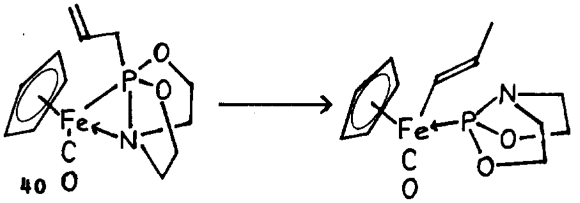
Electrochemical reduction of $\mathrm{Fp_n[Co(CO)_4]_{3-n}SnCl}$ resulted in cleavage of the Sn-Co bonds[218]. Reduction of $\mathrm{FpER_3}$ (E = Si, Ge, Sn) gave radical anions, which cleaved to Fp^- and E_2R_6. This allowed the formation of Fp-substituted distannanes such as $(Fp_2SnPh)_2$[219]. $FpPbPh_3$ decomposes thermally or photochemically with phenyl migration, forming FpPh[220].

Fp^{*-} reacted with R_2NPCl_2 (R = isopropyl) to form $FpP(Cl)NR_2$; reaction of the latter with $AlCl_3$ or Ph_3C^+ proceeded via a phosphinidene complex to a phosphazetidine complex, as in Eq. 28[221]:



(28)

R_2PS^- reacted with FpBr to form $FpP(=S)R_2$[222]. Tris(2,2,2-trifluoroethyl)phosphite displaced the CO's from FpI analogously to other phosphites, giving $CpFe[P(OR)_3]_2I$[223]. The complex phosphoranide **40** rearranged at temperatures above 0° with allyl group migration to iron (eq. 29)[224].



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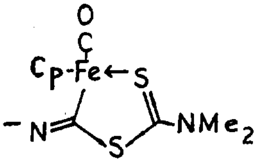
Reaction of $Fp(THF)^+$ with ER_2 (E = S, Se, Te, R = various alkyl) gave $FpER_2^+$. Photochemical reaction with additional ER_2 formed $CpFe(CO)(ER_2)_2^+$. Nucleophiles such as I^- displaced an ER_2 from either type of species[225]. Dimerization of the paramagnetic $CpFe(CO)[P(OR)_3]SPh^+$ and related species by formation of S-S bonds has been studied by visible spectroscopy[226]. Reaction of $NaSC(=S)NMe_2$ with $Fp^*(THF)^+$ gave $Fp^*SC(=S)NMe_2$[195, 203]; the analogous Fp compound was most conveniently obtained using $FpCO^+$. Several reactions of $FpSC(=S)NMe_2$ have been studied, including cyclization to **41** with KCN and electrophilic attack at the uncoordinated S[227]. Sulfur heterocycles **42** resulted when FpSMe was treated with electrophilic acetylenes (R = CF_3, CO_2Me)[227a].



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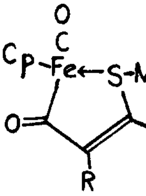


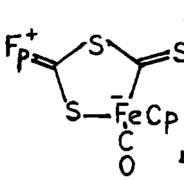
42

c. Fp-Acyl, -Alkyl, and -Carbene complexes

Reaction of Fp_2Mg at -90° with CO_2 gave $(FpCO_2)Mg$, which was alkylated with MeOTf to form the ester FpC(=O)OMe. Further methylation and hydride reduction gave $FpCH_2OMe$[228]. Similarly, $FpCS_2^-$ reacted with electrophiles E-X (Me_3Si-Cl, Me_3Sn-Cl, Fp-X, Me-X) to form FpC(=S)SE, in which the thione sulfur shows significant nucleophilicity[229]. Reaction of FpC(=S)SFp, **43**, with $(THF)M(CO)_5$ (M = Cr, Mo, W) gave $FpC[=S\rightarrow M(CO)_5]SFp$; the crystal structure of the W compound was reported[230]. Reaction of **43** with $FpCS^+$ gave the novel heterocycle **44** (X-ray structure), from which Br^- displaced the thione-bound Fp group[231].

Reaction of carboranecarbonyl chlorides $B_{10}H_{10}HCC$-C(=O)Cl with Fp^- gave the expected acyl-Fp compounds, which underwent normal decarbonylation to the carboranyl-Fp compounds. These reacted with bromine with migration of the carboranyl group to the cyclopentadienyl ring[232]. Reaction of $FpCO^+$ with diamines led to dicarbamoyl complexes such as $FpC(=O)NHCH_2CH_2NH(C=O)Fp$ (X-ray structure)[233].

Attack of the ylide $Me_3P=CH_2$ on $CpFe(CNR)_2CO^+$ led to formation of the iron-acyl ylide, $CpFe(CNR)_2C(=O)CH=PMe_3$[206]. Several studies of analogous iron-acyl enolates have been reported. Stereoselection in condensations of the diethylaluminum enolate $CpFe(CO)(PPh_3)C(=CH_2)OAlEt_2$ with aldehydes[234] and with imines[235] has been found to be very high. Oxidative decomplexation of the β-hydroxyacyls gave β-hydroxycarboxylic acids[234], but the β-aminoacyls gave β-lactams[235, 236]. Reactions of the lithium enolate with electrophiles (imines[236], RX, RC(=O)Cl[237]) have also received attention. Most reactions were unexceptional, but reaction with ethyl chloroformate led to formation of $FpPPh_3^+$, ethyl acetate, and a small amount of **45**[237].



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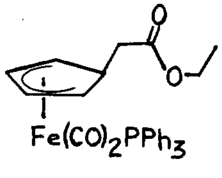
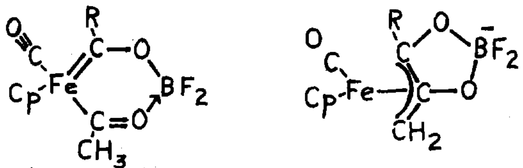


45

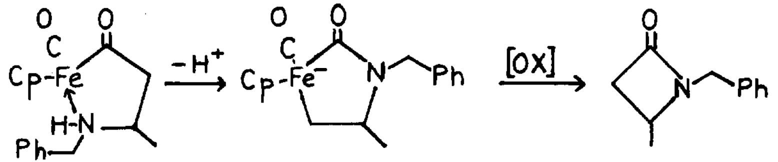
The deprotonation of the coordinated diacyl (Eq. 30) has been extended to additional metals[238].



(30)

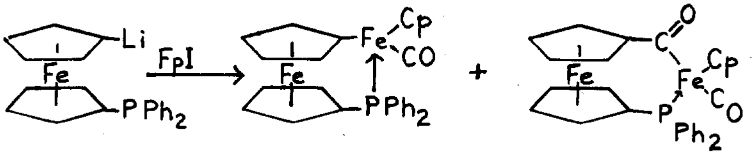
Contrary to expectation, one-electron oxidation of **46** did not generate a β-lactam, but deprotonation with NaH, followed by oxidation of the rearranged heterocycle did (Eq. 31)[239]. Oxidizing agents (Ag^+, ferricenium$^+$) catalyzed conversion of vinyliron compounds to acyliron compounds under CO, allowing use of low

46



(31)

temperatures[240]. Also at low temperatures, the oxidatively-induced metal-to-carbonyl migration of the alkyl group was able to compete with β-elimination when $FpCH_2CH_2R$ was treated with Ph_3C^+ salts in the presence of CO. The migration occurred via a free radical chain process[241]. An extensive study of "migratory insertion" reactions of optically active $CpFe(CO)(PR_3)Et$ has explored the effects of solvents and of acid catalysts[242]. Photoinduced decarbonylation of FpC(=O)Me in PVC films was found to be thermally reversible; CpFe(CO)Me was identified as an intermediate in photolysis of FpMe[243]. Reaction of the ferrocenyllithium derivative with FpI gave both the aryl-Fp product (X-ray structure) and the acyl (Eq. 32), whereas $CpFe(CO)(PPh_3)I$

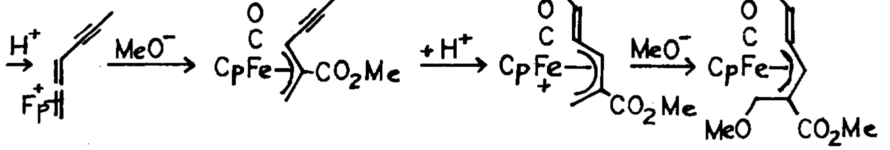


(32)

gave only the aryl[244].

Octafluorocyclooctatetraene reacted with Fp^- to give first FpC_8F_7, then $1,5\text{-}Fp_2C_8F_6$[245]. ^{57}Fe Mossbauer and ^{13}C NMR measurements on a number of aryl-Fp derivatives have been carried out[246]. PhFp and $PhCH_2Fp$ underwent metallation with BuLi on the cyclopentadienyl ring. The pK_a of the ring protons was estimated as 29-30[247]. Crystal structures of $[CpFe(CO)D\text{-}C_5H_4]Mn(CO)_3$, D = CO, PPh_3) were reported[248].

Electrophilic cycloaddition of $FpCH_2CH{=}CHOMe$ with $MeO_2CH{=}C(CN)CO_2Me$ was the initial step in syntheses of some naturally occurring cyclopentanoids[249]. Both *cis*- and *trans*- NCCH=CHCN cycloadded to $Fp(\eta^1\text{-}C_5H_5)$ at comparable rates and without *cis-trans* interconversion between starting material and product, implying a concerted Diels-Alder reaction[250]. Analogous cycloadditions of activated alkenes have been exploited synthetically[251]. Acid-induced addition of methanol to $FpCH_2C{\equiv}C\text{-}C{\equiv}C\text{-}Me$ occurred as in Eq. 33[252]:



(33)

Results of extended Hückel calculations on FpMe served as models for calculations on carbon-uranium bonding[253]. EH calculations and NMR data have been applied to conformational analysis of $CpFe(PPh_3)(CO)R$, based on a pseudo-octahedral model. That the phenyl groups of the phosphine play a dominant role in determining most stable conformations was suggested[254].

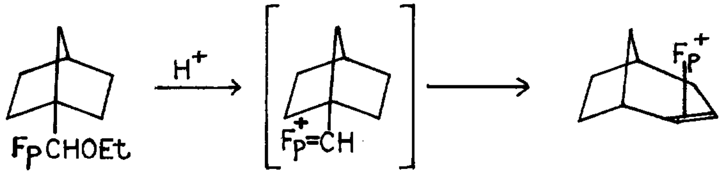
A modified flash chromatography procedure for purifying air-sensitive organometallics has been devised and applied to FpCH(Me)SPh[255]. A study of thermal and photochemical decomposition of $Fp(CH_2)_nFp$ (n = 3-5) indicated alkenes to be the chief products, presumably formed via transient diferracycles[256].

Reaction of $CpFe(PMe_3)_2Cl$ or its Cp^* analog with Grignard reagents gave the expected alkyl derivatives. A bridged example, $(Me_3P)_2Fe(CH_2CH_2-\eta^5-C_5H_4)$, was obtained directly from reaction of $(\eta-C_6H_6)Fe(PMe_3)_2$ with spiro[2.4]hepta-4,6-diene. $Cp^*Fe(PMe_3)_2Et$ in solution existed in equilibrium with $Cp^*Fe(PMe_3)(C_2H_4)H$ and PMe_3 [190].

FpMe reacted with $Cp^*_2ZrH_2$ and PMe_3 to form $CpFe(PMe_3)(CO)H$ and $Cp^*_2Zr(H)(OCH=CH_2)$. The iron hydride reacted further to form $CpFe(PMe_3)_2CH_2OZr(H)Cp^*_2$[257].

FpC_6F_5 showed no halogen exchange when treated with BCl_3[258], but $FpCX_3$ (X = F, Cl, Br) were quite reactive; carbene complexes $Fp=CX_2^+$ were characterized spectroscopically. $FpCCl_3$ reacted readily with NH_3, RNH_2, and H_2O to give, respectively, FpCN, $FpCNR^+$, and $FpCO^+$[259].

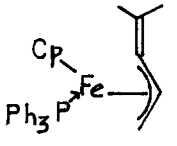
The ethylidene complex $Fp=CHMe^+$ transferred the ethylidene group to alkenes, giving methylcyclopropane derivatives. Bond rotation occurred in ethylidene addition to *p*-$MeOC_6H_4CH=CHD$, which indicated an unusually well-stabilized carbonium ion intermediate[260]. An attempt to generate (1-norbornyl)$CH=Fp^+$ resulted in β-alkyl migration, yielding a bicyclo[3.2.1]octene complex (Eq. 34)[261].



(34)

Complexes of unsaturated carbenes have received considerable attention during 1984. Conformational barriers and conformational equilibria have been studied in various $CpFe(diphosphine)=C=CHR^+$ species with R = Me, t-Bu, Ph[262]. Nucleophiles (PhS^- and $Cu(CN)R_2^-$) attacked the α-carbon of $Fp'=C=CMe_2^+$ [Fp' = $CpFe(CO)(PPh_3)$], yielding vinyl complexes $Fp'-C(Nu)=CMe_2$. The 2-butadienyl-Fp' product (Nu = $CH=CH_2$) rearranged readily to the η^3-butatrienyl complex **46**[263]. The 3-methylbut-2-en-1-ylidene complex

$Fp{=}CHCH{=}CMe_2^+$ has been characterized by low-temperature NMR, and found to transfer the alkenylidene unit to isobutene, cyclooctene, and styrene[264]. Similar alkenylidene complexes $Fp(')=CHCH{=}CRCH_2R^+$ resulted from δ-protonation of 1-dienyl complexes $Fp(')CH{=}CHCR{=}CHR$, based on NMR and reactions with nucleophiles[265]. The delocalized carbene complex 47 added CN^- to an α-carbon as expected[266].



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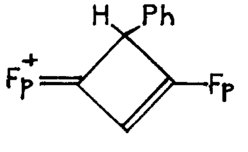

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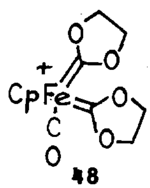

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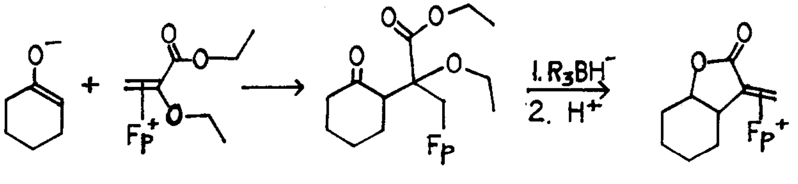
An extensive study of reactions of the heterocycles oxirane, thiirane, and aziridine with $FpCO^+$ and $FpCS^+$, to yield cyclic heteroatom-stabilized carbene complexes has been published. Prolonged reaction of oxirane with $FpCO^+$ actually generated a bis-(dioxycarbene), 48[267]. A cyclic oxycarbene complex was quaternized by consecutive deprotonation and alkylation steps[268]. $CpFe(PMe_3)_2\text{-}C(OMe){=}CH_2$ showed the substantial basicity of its β-carbon by removing protons from $HW(CO)_3Cp$ and $MeC({=}O)Cl$, forming the 1-methoxyethylidene complex. It also added CS_2 to form $CpFe^+(PMe_3)_2{=}C(OMe)CH_2CS_2^-$ [269].

High stereoselectivity in addition of hydride to $Fp'{=}C(OMe)Et^+$ and of the ethyl group to $Fp'{=}CHOMe^+$ has been found[270]. β-Deprotonation of methoxycarbene complexes $Fp'{=}C(OMe)CH_2R^+$ gave the (Z)-methoxyvinyl products (X-ray structure reported), which could be alkylated stereoselectively[271].

d. Cyclopentadienyliron Derivatives of η^2 to η^5 Ligands

The alkene complex $(CH_2{=}C(OEt)CO_2Et)Fp^+$ has been prepared from ethyl bromopyruvate, and used as a synthetic equivalent of $CH_2{=}C(+)CO_2Et$, to convert enolates to α-methylenelactones (Eq. 35)[272]. Likewise, alkylation of Fp^+ complexes of $ROCH{=}CHOR$ with cuprates, Grignard reagents, and enolates resulted in formation of $RCH{=}CHR'$[273]. Additions of similar nucleophiles to $(alkyne)Fp'^+$ complexes have resulted in formation of a variety of alkenyl-Fp' products[274]. The presence of detergent micelles affected the relative reactivity and products of reaction of $(alkene)Fp^+$ with



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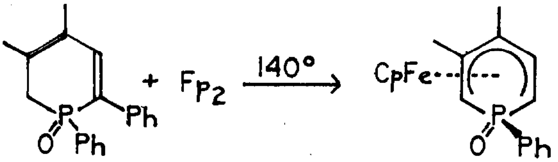
water[275].

Some (η^3-allyl)FeCp(CO) derivatives have already been described (Eqs. 30 and 33, and compound 46). Some CpFe(η^5-carbollyl) compounds have been described. Protonation of the ethynyl compound, CpFe(η^5-B_9H_9CHC-C$\equiv$CH) on the β-carbon gave a stabilized vinyl cation[276]. Bis(dicarbollyl)iron(III) salts could be obtained by electrolysis of (3)-1,2- and (3)-1,7-dicarbadodecahydroundecaborate salts at an iron anode[277].

2-Phenylphosphaferrocenes resulted from reaction of 1-phenylphospholes with Fp_2 at 150° under CO pressure[278]. A similar reaction of a phosphorin oxide gave a η^5-phosphorin complex (Eq. 36). The crystal structure showed an Fe-P distance of 2.784(3) A. Acylation occurred on the Cp ring rather than on the heterocycle[279].



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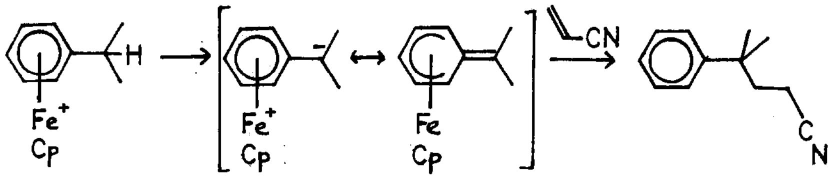
8. COMPOUNDS WITH η^6-ARENE LIGANDS

The use of (η-benzene)Fe$(PMe_3)_2$ as a source of Fe$(PMe_3)_2$ groups in reactions with cyclopentadienes has already been described[190]. In an analogous way, (η-p-xylene)FeCp$^+$ has been used as a source of CpFe$^+$ groups in reactions with 6-aminopentafulvenes to give ferrocene derivatives[280]. An extensive study of photoremoval of the arene ligand from AnFeCp$^+$, including the effects of arene substituents, solvent, and temperature, has appeared[281]. CpFeAn$^+$ catalyzed photoisomerization of hexamethyl Dewar benzene to hexamethylbenzene, via an exciplex intermediate[282].

A number of CpFeAn$^+$ complexes containing Cl, F, and OMe substituents on the arene ring have been prepared by the standard method, reaction of the arenes with ferrocene and $AlCl_3$. The chloro substituent was readily displaced by amines[283]. Ethyl group rotation in (C_6Et_6)FeCp$^+$ was found to be slow enough at 140 K to allow detection of several geometrical isomers[284]. The X-ray structure of the zwitterion $Ph_3\bar{B}$-(η-C_6H_5)FeCp$^+$ has been determined[285].

CpFe$^+$ complexes of xanthene and thioxanthene have been prepared, and oxidized with $KMnO_4$ to xanthone, thioxanthone, and thioxanthone dioxide complexes. Xanthone and fluorenone complexes have been found to add nucleophiles to the carbonyl group from the exo face; in the case of xanthone, nucleophilic attack on the coordinated ring with displacement of a phenoxide leaving group also occurred[286]. (η-C_6H_5R)FeCp$^+$ (R = $CHMe_2$, NH_2, and NHMe), as well as $CpFe^+$ complexes of carbazole and fluorene, were readily deprotonated; the resulting species served as nucleophiles in Michael addition reactions (Eq. 37)[287]. Reaction of $(PhCl)FeCp^+$



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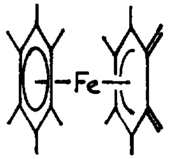
with NaOH or NaSH gave phenoxide or thiophenoxide complexes, which underwent homologation with CH_2N_2 to give (tropone)- or (thiotropone)-$FeCp^+$ [288].

Electrochemical reduction of several (η^6-cyclophane)$FeCp^+$ has been reported. The reduced species decomposed with dissociation of the cyclophane and formation of ferrocene and Fe(0)[289]. The reduced 19-e species (η^6-fluorene)FeCp has been generated as a metastable species at -50°. Reaction with O_2 generated (fluorenyl)FeCp via electron transfer then proton abstraction[290]. The 19-e species (η-C_6Me_6)$Fe(C_5H_4CO_2H)$ has also been generated; it lost the acidic proton fairly readily[291].

Iron atom chemistry has been used to prepare several examples of (η-arene)ferraborane and -ferracarborane clusters through condensation with B_5H_9 or $B_{10}H_{14}$ in the presence or arenes and 2-butyne. The arene ligands derived variously from added arene or from simultaneous cyclotrimerization of 2-butyne. The compounds reported, several characterized by X-ray crystallography, included several different arenes and such fragments as $B_8H_{10}O$, B_9H_9, B_9H_{13}, $B_{10}H_{10}$, $(CMe)_2B_4H_4$, $(CMe)_4B_3H_3$, and $(CMe)_4B_5H_5$ as η^5-ligands[292,293].

Two studies have focussed upon the 20-electron bis(arene)iron(0) species. Mössbauer and magnetic studies of $(C_6Me_6)_2Fe$, along with semiempirical MO calculations, have confirmed its triplet nature[294]. It reacted with organic halides RX by electron transfer, then coupling of R· with An_2Fe^+. Reaction with O_2 eventuated in formation of an o-xylylene complex, 49[295].



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Hydride added to An_2Fe^{2+} to generate derivatives of (η-C_6H_6)$Fe(\eta^5$-$C_6H_7)^+$, which reacted with nucleophiles on the η^5-coordinated ring to give (arene)Fe(cyclohexadiene) derivatives. However, the permethylated series gave bis(cyclohexadienyl)iron complexes instead[296].

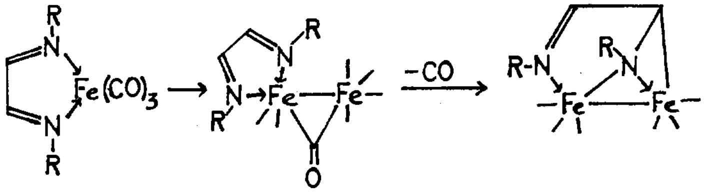
9. BIMETALLIC COMPOUNDS

a. Diiron compounds. Derivatives of $Fe_2(CO)_9$

In the gas phase, $Fe(CO)_4^{\dot{-}}$ reacts very rapidly with $Fe(CO)_5$ to form $Fe_2(CO)_8^{\dot{-}}$. $Fe_2(CO)_{5-7}^-$, thought to have Fe=Fe double bonds, have also been identified[76].

Although $Fe_2(CO)_9$ in the crystal has three bridging carbonyl groups, replacement of CO's by other two-electron ligands commonly produces products with only one bridging group. SCF-MO calculations have been applied to the interpretation of the structure of $(\mu\text{-DPPM})Fe_2(CO)_7$, having one symmetrically bridging CO and a short Fe-Fe bond[297]. A number of diphosphorus ligands R_2PYPR_2 (for example, R = Ph, Me, OMe, OEt; Y = CH_2, NMe) have given similar products, $(\mu\text{-}R_2PYPR_2)Fe_2(CO)_7$ and also $(\mu\text{-}R_2PYPR_2)_2Fe_2(CO)_5$, upon photoreaction with $Fe_2(CO)_9$[298,299].

Labelling studies using ^{13}CO have indicated that reaction of $(RN{=}CH{-}CH{=}NR)Fe(CO)_3$ with $Fe_2(CO)_9$ goes through a $(RN{=}CH{-}CH{=}NR)Fe_2(CO)_7$ intermediate (Eq. 38)[300].



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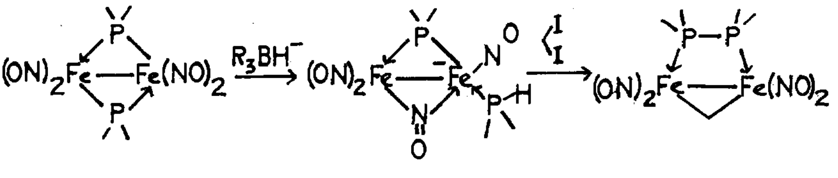
Reactions of $Fe_2(\mu\text{-}CH_2)(CO)_8$ with nucleophiles appeared to involve initial attack at a CO ligand, but the products in most cases eluded complete characterization[99]. Among the products of attack of $LiOCR_2C{\equiv}CLi$ on $Me_3NFe(CO)_4$ was $(OC)_4Fe(\mu\text{-}C{=}C{=}CR_2)\text{-}Fe(CO)_4$ (R = t-Bu); the same product also resulted in high yield from reaction of $R_2C{=}C{=}C{=}Fe(CO)_4$ with $Fe_2(CO)_9$[301]. A bridging CH_2 ligand was introduced, with rearrangement, by reaction of CH_2I_2 with $Fe_2(NO)_4(PPh_2)_2$ (Eq. 39)[302]:



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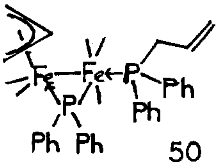
Reaction of $Fe_2(CO)_8^{2-}$ with $(\text{-}N{=}PCl_2)_{3,4}$ resulted in formation of diferraphosphirane rings, with one phosphorus of the cyclophosphazene bridging a $(OC)_4Fe\text{-}Fe(CO)_4$ unit[303]. Reactions of the arsenido-bridged $(OC)_4Fe(\mu\text{-}AsMe_2)Fe(CO)_2(NO)$ with Group V donor ligands occurred with attack at the nitrosyl-bearing Fe and eventuated in heterolysis of the Fe-Fe bond[304]. The phosphido-bridged product **50** (X-ray) was obtained by reaction of allyl iodide with deprotonated $(OC)_3Fe(\mu\text{-}CO)(\mu\text{-}PPh_2)Fe(CO)_2(PPh_2H)^-$ [305].

The greatest number of derivatives of $Fe_2(CO)_9$ is the group in which the three bridging carbonyls have been replaced by one or two ligands which contribute a total of six electrons, represented by structural types **51** and **52**. In these structures the symbol Ft represents the $Fe(CO)_3$ group. Hartree-Fock-Slater calculations on **51** (X = Y = S) have shown the LUMO to be S-S antibonding, consistent with the opening of that bond upon two-electron reduction[306]. EHMO calculations on these species have also indicated, however, that addition of H_2 or acetylene to the Fe-Fe bond may be a concerted thermal process[307]. Reaction of $(\mu\text{-}S)_2Fe_2(CO)_6$ with $LiEt_3BH$ produced the dianion $Fe_4S_4(CO)_{12}{}^{2-}$, **53**[308] (X-ray structure). A related S-linked structure, **54**, resulted, alongside the major products, *syn*- and *anti*-$Fe_2(SMe)_2(CO)_6$, from reaction of $Fe_3(CO)_{12}$ with Me_2S_2[309].



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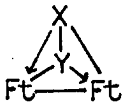

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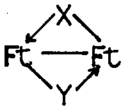

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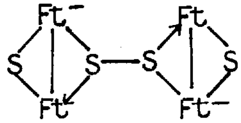
1,1-dimethylsilacyclopent-3-ene reacted with sulfur and $Fe_3(CO)_{12}$ to give *inter alia* **55**, the addition product of the alkene across the S-S bond of **51** (X = Y = S)[310]. A similar ethanedithiolate derivative, **52** (X, Y = $-SCH_2CH(SiMe_3)S-$) reacted with $LiAlH_4$ to form principally a product assigned the linked structure **56**[311]. The thermal decomposition of the same starting material at 165° has also been studied[312]. A presumably related compound of composition $(\mu\text{-}C_3H_4S_3)Fe_2(CO)_6$, structure unknown, was obtained from reaction of (ethylenetrithiocarbonate)$Cr(CO)_5$ with $Fe(CO)_5$ under irradiation[313].



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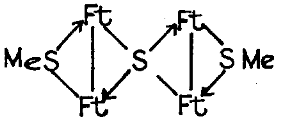

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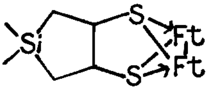

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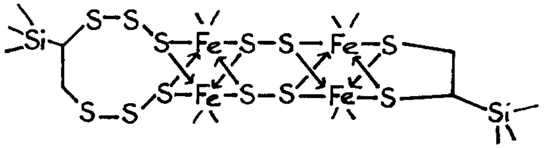

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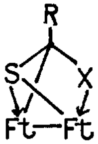
The compound (μ-HNS)$Fe_2(CO)_6$, with the unknown substance sulfur imide (H-N=S) stabilized by coordination to iron, has been prepared[314]. Compounds with thio- and dithioesters donating six electrons to the $Fe_2(CO)_6$ unit, **57**, (R = Me, H; X = SMe, SEt, OMe, etc.) formed upon direct reaction of the esters with $Fe_2(CO)_9$; however, thioformate esters gave the dimeric **58** instead[315]. The adamantyldithiocarbonate ROC(=S)SMe gave a product analogous to **57** (R = $OC_{10}H_{15}$, X = SMe); this rearranged thermally to the carbene complex **59**[316]. Eq. 40 shows another example of bifurcation of a ligand with formation of a carbene complex[317].



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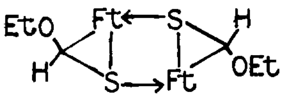

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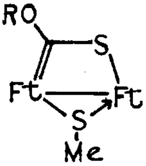

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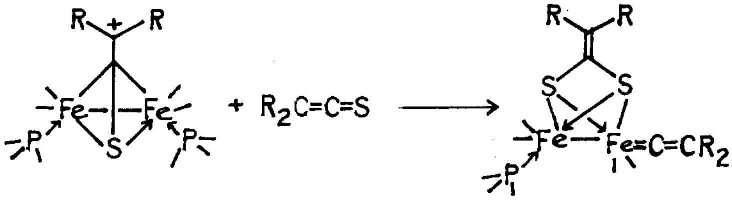

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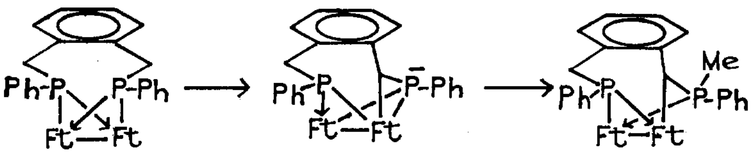
X-ray structures of two tetraphosphane-bridged compounds (**52**, X, Y = -P(R)P(R)P(R)P(R)-, R = Me and Ph) have been reported[318]. Reaction of **52** (X = Y = PhPH) with α,α'-dibromo-o-xylene in the presence of base gave **52** (X, Y = $-P(Ph)CH_2(\underline{o}-C_6H_4)CH_2P(Ph)-$). Reaction of the latter product with BuLi formed a transient carbanion which attacked the iron; methylation on phosphorus then



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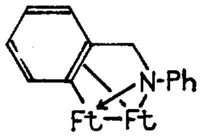
60

gave **60** (Eq. 41)[319]. Deprotonation of **52** (X = Y = PhPH) with MeLi gave a dianion, which reacted with $BrCH_2CH_2Br$ in a redox manner, forming a dimeric product having two $(OC)_3Fe(\mu\text{-PMe})_2Fe(CO)_3$ units joined by P-P bonds[320].

An X-ray structure of **61**, obtained from benzalazine and $Fe_3(CO)_{12}$, has appeared[321]. Reaction of the same iron carbonyl with 2-nitropropane in toluene formed $(OC)_3Fe(\mu\text{-}O{=}CCH_2Ph)(\mu\text{-}N{=}CMe_2)\text{-}Fe(CO)_3(Fe\text{-}Fe)$, characterized by X-ray crystallography[322]. Reaction of $Fe_3(CO)_{12}$ with $MeC{\equiv}CNEt_2$ produced a ferrole complex, **62**, through head-to-head coupling of the aminoalkyne moieties[323]. The parent ferrole complex, $C_4H_4Fe_2(CO)_6$, resulted in low yield from reaction of (thiophene)$Mn(CO)_3^+$ with $HFe(CO)_4^-$, along with



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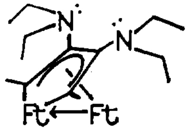

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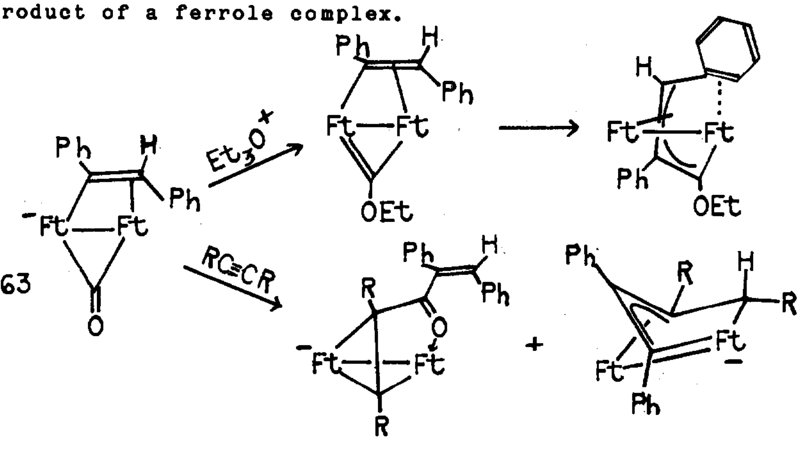
$(OC)_3Fe(\mu\text{-}CH{=}CH\text{-}CH{=}CH\text{-}S\text{-})Fe(CO)_3(Fe\text{-}Fe)$[324]. Thermal decomposition of ferrole complexes was studied as part of a search for metathesis-like reactions[325].

The conformation of (μ-butatriene)$Fe_2(CO)_6$ in solution has been investigated by study of the proton NMR spectrum in nematic phases[326]. Substituted butatriene complexes were synthesized by reaction of HOCPhRCC≡CCPhROH with $Fe_3(CO)_{12}$, and the cis-trans isomer ratios obtained depended on the group R[327].

The (μ-diphenylethenyl) complex **63** has been the subject of two interesting chemical studies. Reaction of **63** with triethyloxonium fluoroborate (Eq. 42) proceeded through alkylation of the bridging CO, forming a μ-carbyne ligand, to a ferrabutadiene complex[328]; the Fe-phenyl distances represented by the dotted line are about 2.43 Å, as compared to 2.18 and 2.30 Å in **61**. Reaction of **63** with electrophilic alkynes (RC≡CR, R = CO_2Me, CF_3) produced two types of products, both characterized by X-ray crystallography, shown in Eq. 43. Mechanisms for these conversions have been proposed[329]. The latter product is formally a hydride addition product of a ferrole complex.



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The principal product of reaction of iron carbonyls with dimethyl cyclooctatetraene-1,8-dicarboxylate was a non-fluxional skew $Fe_2(CO)_6$ complex with the ester groups located on the σ-bonded and an uncoordinated carbon[172].

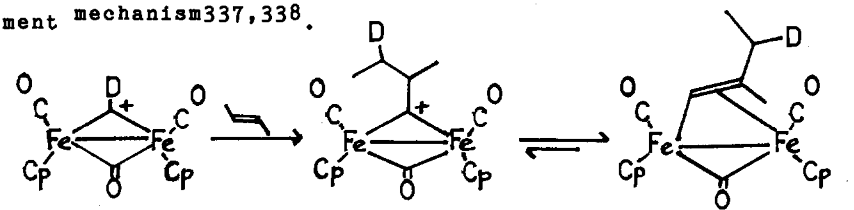
b. Derivatives of $Cp_2Fe_2(CO)_4$

Photolysis of *trans*-$CpFeCO(\mu\text{-}CO)_2FeCOCp$ derivatives in low temperature matrices resulted in CO loss and formation of a triply-bridged $CpFe(\mu\text{-}CO)_3FeCp$, presumably having a Fe=Fe double bond[330]. Photolysis of Fp_2 in solution in the presence of DPPE generated a strong reducing agent which reduced, for example, $Mn_2(CO)_{10}$. A 19-electron iron species, $CpFe(CO)_{1\ \mathrm{or}\ 2}(DPPE)$ was proposed as the reducing agent[331]. Reaction of bis(dimethylphosphino)methane with Fp_2 produced the product $CpFe(\mu\text{-}CO)_2(\mu\text{-}Me_2P\text{-}CH_2PMe_2)FeCp$[298].

A number of mixed Fp_2 derivatives having one or two CS and one or two isonitrile ligands have been prepared and studied to determine the relative bridging tendencies of the ligands and mechanism of interconversion[332]. The radical-cation $CpFeCO(\mu\text{-}SEt)_2FeCpCO^+$ has been studied crystallographically; the long Fe-Fe distance [2.957(4) A] was consistent with a one-electron bond[333].

K^+Fp^- (or $K^+Fp^{*\,-}$) reacted with chloromethyl pivalate in refluxing THF to provide $CpFeCO(\mu\text{-}CH_2)(\mu\text{-}CO)FeCpCO$ or its decamethyl derivative[334]. The analogous μ-cyclopropylidene compound by reaction of the μ-vinylidene compound with diazomethane/cuprous chloride[335]. Reaction of $CpFe(\mu\text{-}NO)_2FeCp(Fe{=}Fe)$ with diazoacetaldehyde produced $CpFeNO[\mu\text{-}CH(CHO)]FeCpNO(Fe\text{-}Fe)$[336].

Reactions of the bridging methylidyne complex, $CpFeCO(\mu\text{-}CH^+)\text{-}(\mu\text{-}CO)FeCpCO(Fe\text{-}Fe)$ with alkenes have been studied. 2-Butenes reacted by concerted hydrocarbation to give $\mu\text{-}C^+CHMeCHDMe$ products, which rearranged to μ-vinylic compounds (Eq. 44). 1-Methylcyclohexene reacted via a carbonium ion rearrangement mechanism[337,338].



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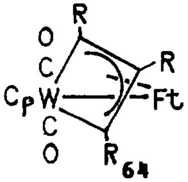
c. Heterobimetallic Compounds

Reaction of $HFe(CO)_4^-$ with $(THF)M(CO)_5$ (M = Cr, Mo, W) produced $(OC)_4Fe(\mu\text{-}H)M(CO)_5(Fe\text{-}M)$, and the tungsten compound was characterized crystallographically[339]. Products of structure **64**, (R's = CMe_3, $SiMe_3$) which may be considered $Fe(CO)_3$ derivatives of tungstenacyclobutadienes, have been prepared and characterized[340]. In the reaction of $(HBPz_3)W(CO)_2(\equiv CR)$ (Pz = 1-pyrazolyl, R = Me, *p*-tolyl) with $Fe_2(CO)_9$, the $W{\equiv}C$ triple bond was capable of acting as a four-electron donor to iron, forming **65** (X-ray)[341].

Whereas reaction of $HMn(CO)_5$ with $FpPPh_2$ gave $Cp(CO)Fe(\mu\text{-}H)\text{-}$

(μ-PPh$_2$)Mn(CO)$_4$(Fe-Mn), the analogous methyl-manganese compound reacted to form Cp(CO)Fe(μ-MeC=O)(μ-PPh$_2$)Mn(CO)$_4$, having a bridging acetyl group[342]. (1,4-diazabutadiene)Mn(CO)$_3^+$ reacted with HFe(CO)$_4^-$ to produce 1-azaallyl complexes **66**[343].



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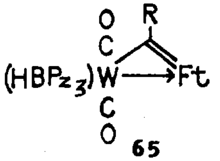

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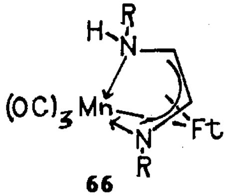
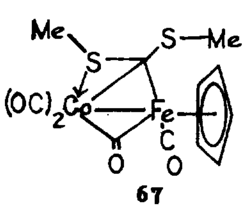


66

The crystal structure, cis-trans isomerization, and chemical reactions of the mono-ruthenium derivative of Fp$_2$ have been studied [344]. A tetraphenylruthenole-Fe(CO)$_3$ complex was among the products obtained from reaction of PhC≡CPh with Fe$_3$(CO)$_{12}$ and Ru$_3$(CO)$_{12}$[345].

Photolysis of FpCo(CO)$_4$ at 10 K in argon matrix gave evidence of proceeding through two competing pathways, one involving CO loss, and one producing FpCO$^+$ and Co(CO)$_4^-$, in the presence of excess CO[346]. Cobalt tetracarbonyl anion reacted with dithiocarbene complex, CpFe(CO)(NCMe)[=C(SMe)$_2$]$^+$, to form **67**, (X-ray), which was the subject of MO calculations[347]. Hydrogenation of **68** has been studied[348]. Reaction of Fe(CO)$_4$(PR$_2$Li) with (Me$_3$P)$_2$CoCl$_2$ gave (OC)$_3$(Me$_3$P)Fe(μ-PR$_2$)Co(CO)$_2$(PMe$_3$) (R = t-Bu), in which exchange of CO and phosphine units between iron and cobalt had occurred[349].

Reaction of [HC(PPh$_2$)$_3$]Fe(CO)$_3$ with Rh$_2$(CO)$_4$Cl$_2$ resulted in formation of a product with a triphosphine-supported Fe→Rh dative bond, (OC)$_2$Rh[μ-CH(PPh$_2$)$_3$]Fe(CO)$_3$[66]. Phosphines displaced CO's only from Rh in reaction with (OC)$_2$Rh(μ-C$_7$H$_7$)Fe(CO)$_3$; using DPPE, the product showed a reversal of bonding mode from η^4-Rh and η^3-Fe in the starting material to η^3-Rh and η^4-Fe in the product, **69**[350].



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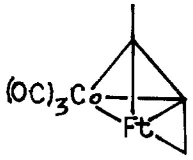

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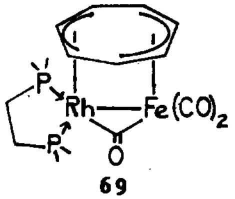

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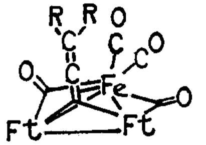
10. TRINUCLEAR CLUSTER COMPOUNDS

a. Tri-iron Clusters

Fe$_3$(CO)$_{12}$ catalyzed the reaction of polyfluoroalkyl halides, R$_F$X, with allylsilanes to produce R$_F$CH$_2$CH=CHR[351]. In the substitution product $Fe_3(CO)_{11}(CNCF_3)$, the isocyanide ligand, unstable in the free state, occupies a bridging position[352]. Condensation of $HFe(CO)_4^-$ to $HFe_3(CO)_{11}^-$ has been studied. The tri-iron anion was not formed under water gas shift conditions[353]. Ion pairing of the $Fe_3(CO)_{11}^-$ anion has been studied[354]. Alumina impregnated with $K^+Fe_3(CO)_{11}^-$ brings about some conversion of CO to hydrocarbons at 400° [355].

The fluxional allenylidene complex **70** was obtained as a minor product of reaction of iron carbonyls with $LiC{\equiv}CCR_2OLi$ (R = *t*-Bu)[301]. One-electron reduction of the anion **71**, $Fe_3(CO)_9(\mu_3\text{-}CO)(\mu_3\text{-}CMe)^-$ led to coupling of the ethylidyne fragment with a CO, giving a propynolate anion ligand in **72**[356]. The equilibrium constant for reaction of protonated **71** with H_2 at 60° to form $(\mu\text{-}H)_3Fe_3(CO)_9(\mu_3\text{-}CMe)$ was 0.66, and the enthalpy +4 kcal/mol[357].



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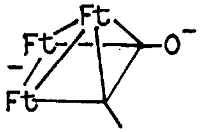

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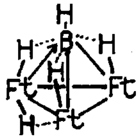
Triiron clusters containing borohydride ligands have been prepared and characterized. These included **73** and **74** and their deprotonated monoanions. Analogies to the isoelectronic hydrocarbyl clusters were drawn[358, 359].



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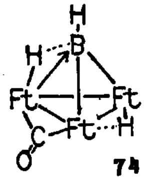

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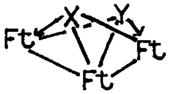
The X-ray structure of $(PhCN)Fe_3(CO)_9$ showed the benzonitrile ligand donating its non-bonding electron pair to one Fe and a π-electron pair to each of the other two Fe's in a Ft_3 triangle[360]. The X-ray structure of $(\mu_3\text{-}NPh)_2Fe_3(CO)_9$, **75** (X = Y = NPh) has also been reported[361]. Cyclotri- and cyclotetraphosphazenes have been found coordinated as four-electron ligands to $Fe_3(CO)_{10}$ units[303]. Reaction of $Fe_4(CO)_{13}^{2-}$ with diphenylphosphine in



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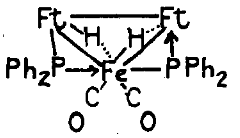

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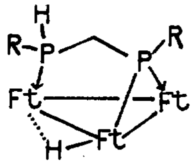

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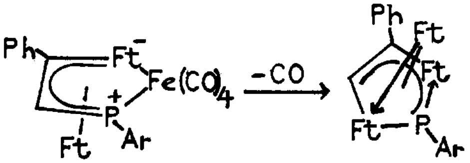
strong acid produced **76** (X-ray), which lost two molecules of benzene at room temperature to form **75** (X = Y = PPh)[362]. Excess $Fe_2(CO)_9$ reacted with $CH_2(PRH)_2$ (R = i-Pr) to give **77** (X-ray)[363].

Phenylacetylene inserted into an Fe-P bond of $(\mu_3\text{-PAr})Fe_3(CO)_{10}$ to produce **78**, which rearranged upon photochemical decarbonylation to **79** (Eq. 45)[364]. The first bismuth-containing cluster,



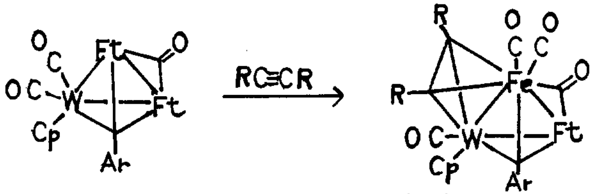
(45)

78 **79**

$(\mu_3\text{-Bi})Fe_3(CO)_9(\mu_3\text{-CO})^-$, resulted from reaction of sodium bismuthate with $Fe(CO)_5$, through clusterification upon oxidation of the intermediate $Bi[Fe(CO)_4]_4^{3-}$ [365]. The X-ray structure of **75** (X = S, Y = PAr) has been reported[366]. An analogous compound (X = S, Y = 2-carbena-1,3-dithiolane), in which a dithiocarbene functions as a four-electron ligand, was obtained as the principal product from photoreaction of $Fe(CO)_5$ with (ethylenetrithiocarbonate)-$Cr(CO)_5$[313].

b. Fe_2M Clusters

Silylacetylenes displaced two CO's from a Fe_2W cluster upon heating, as shown in Eq. 46 (Ar = p-tolyl, R = Me_3Si)[340].



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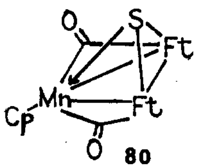
Irradiation of a mixture of $CpMn(CO)_3$ and t-BuSH, then treatment with $Fe_3(CO)_{12}$ gave **80** (X-ray)[367]. The tellurium analog (Cp* instead of Cp) was obtained from reaction of $Te[{=}Mn(CO)_2Cp^*]_2$ with $Fe_2(CO)_9$[368].

Reaction of $M_3(CO)_{12}$ mixtures (M = Fe, Ru) with diphenylacetylene resulted in formation of Fe_2Ru products, especially $Fe_2Ru(CO)_9(PhCCPh)$ and the ruthenole derivative $Fe_2Ru(CO)_8(C_4Ph_4)$, but no $FeRu_2$ products. CNDO calculations were performed to rationalize this result[345]. $Fe_2Os(CO)_{12}$ was the major product when $Fe_2(CO)_9$ was allowed to react with $H_2Os(CO)_4$[369].

One Fe vertex of the ketenylidene complex $Fe_3(CO)_9(CCO)^{2-}$ was replaced upon reaction with $Co_2(CO)_8$, giving **81** (X-ray), which showed reduced nucleophilicity consonant with its reduced charge[370]. A coordinated P≡C triple bond resulted when (DPPE)-Pt(PCR) (R = t-Bu) was allowed to react with iron carbonyls, giving 82[371]. A linear arrangement of metals in the trimetallic cluster Fp_2Au^- was indicated by ^{197}Au Mossbauer spectroscopy[372].



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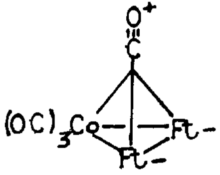

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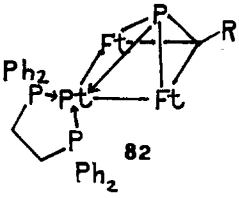

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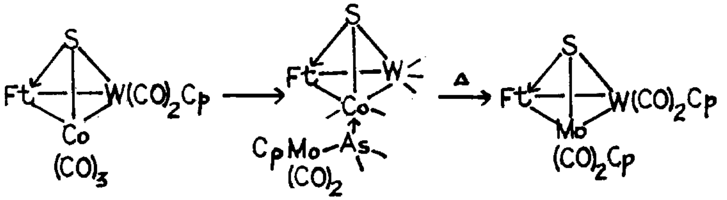
c. FeM_2 Clusters

The activity of mixed Fe-Ru $M_3(CO)_{12}$ species as hydrogenation catalysts increased with the Ru content[373]. An extensive study of substitution reactions of PPh_3 and $P(OMe)_3$ for CO's in the $FeRu_2$ and Fe_2Ru dodecacarbonyls has been made. Substitution occurred preferentially at Ru. Disubstitution in the $FeRu_2$ species led to formation of CO bridges[374]. $FeOs_2(CO)_{12}$ was the principal product when $H_2Os_2(CO)_8$ and $Fe_2(CO)_9$ reacted[369].

The Freiburg group has continued its extensive studies of tetrahedral clusters $EM^1M^2M^3$, in which E is commonly a chalcogen, alkyl- or aryl-pnicogen, or alkylidyne, and the M's represent a variety of metal moieties from Groups 6-9 of the (new style) periodic table. A sample of the synthetic chemistry is presented in Eq. 47[375]. Electrochemical measurements on a series of phosphinidene-bridged $FeCo_2$, FeCoMo, and FeCoNi clusters were performed; fragmentation often followed reduction[376]. The effects of different metals, and of varying isonitrile substituents in the $SFeCo_2$ clusters, were extensively studied[377]. The bridging hydride ligand in 83 was replaced by the Ph_3PAu- group upon treatment with KH then Ph_3PAuCl[378].



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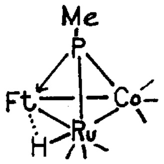
11. TETRA- AND POLYNUCLEAR CLUSTER COMPOUNDS

An X-ray structure of the octahedral cluster anion $RhFe_5C(CO)_{16}^-$ has appeared[379].

Coexistence of another isomer of $HFe_4(CO)_{13}^-$, in addition to the previously crystallographically characterized butterfly isomer, has been indicated by low-temperature NMR studies[380]. The intriguing chemistry of this species, the related $HFe_4(CO)_{12}CH$, and its mono- and di-anions have stimulated theoretical study by three different groups, using Fenske-Hall[381,382] and extended Hückel[383] methods. Protonation of $Fe_4(CO)_{12}CMe^-$ gave **84**, which appeared to add another proton on standing in TfOH; the solution slowly evolved small amounts of methane and ethane[384].



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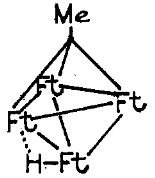

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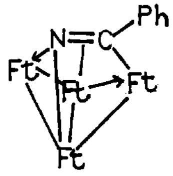


85[360]

Reaction of the adamantanoid $(CpFe)_4S_6$ with $MoOCl_3(THF)_2$ formed a desulfurated cation $(CpFe)_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)^+$, whose X-ray structure has been reported[385].

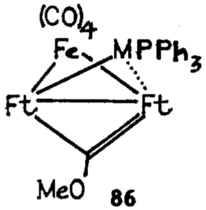
Two new tetrahedral Fe_3M clusters have been reported. Reaction of $Fe_3(CO)_{11}^{-2}$ with $Ni(CO)_4$ followed by protonation, or reaction with $NiCl_2$ in ethanol led to formation of $Fe_3Ni(CO)_8\text{-}(CO)_4(\mu_3\text{-}H)^-$ [386]. The sterically crowded series $Fe_3M(CO)_{14}^{2-}$ (M = Cr, Mo, W) was prepared by straightforward reaction of the triiron dianion with $M(CO)_3(NCMe)_3$. The chromium cluster did not show signs of exceptional crowding in its structure or reactions[387]. The complete family of clusters $Fe_3(CO)_{10}(\mu\text{-}COMe)(\mu\text{-}MPPh_3)$ (M = Cu, Ag, Au), **86**, has also been prepared by reaction of the corresponding Fe_3 anion with Ph_3PMX[388].

A number of new iron-coinage metal clusters have been reported. Planar Cu-Fe clusters, $Cu_3Fe_3(CO)_{12}^{2-}$ and $Cu_5Fe_4(CO)_{16}^{3-}$, resulted when $Fe(CO)_4^{2-}$ reacted in appropriate ratio with copper halides. The latter shows a square planar array of Cu's about a central Cu, with the edges of the square bridged by $Fe(CO)_4$ groups[389]. Reaction of the iron tetracarbonyl dianion with $CH(PPh_2AgCl)_3$ produced $CH[PPh_2Ag_2Fe(CO)_4]_3$, whose X-ray structure showed a distorted octahedron of silver atoms with three faces capped by $Fe(CO)_4$ groups and another by the tripod ligand[390].

Reaction of $Fe_3(CO)_{12}$ with $Cp_2Ni_2(\mu\text{-}HC{\equiv}CCMe{=}CH_2)$ gave the product **87**, characterized by X-ray crystallography, and an isomer assigned a square planar structure[391]. The structure of $FeOs_3H(CO)_{13}^-$ showed a tetrahedron of metal atoms with two (semi)-bridging Fe-CO-Os edges and the hydride bridging an Os-Os edge[392].

Some derivatives of $FeCo_3(CO)_{12}^-$ have been studied. Formation of ammonia and hydrazine in reaction of $HFeCo_3(CO)_{12}$ with $(PMe_2Ph)_4W(N_2)_2$ has been studied[82]. An X-ray structure of $HFeCo_3(CO)_{10}(PPh_3)_2$ showed the two phosphine ligands attached to two cobalts axial to the Co_3 plane and three Co-CO-Co bridges; the H was thought to underly the Co_3 plane[393]. The $CpMo(CO)_3Hg$ group occupied the same triply-cobalt-bridging position when attached to the $FeCo_3(CO)_{12}$ cluster[394], as did copper and silver in $(Ph_3PM)FeCo_3(CO)_{12}$[395].

$Fe(CO)_3NO^-$ displaced iodide from attachment to a Co-Pd-Pt triangular cluster, to form 88[396]. It reacted with $Ru_3(CO)_{12}$ to form $FeRu_3(CO)_{12}NO^-$ (X-ray) which was deoxygenated by CO to form the butterfly μ_4-nitrido clusters $FeRu_3N(CO)_{12}^-$. The isomers with the Fe in the wing-tip and in the hinge positions interconverted in solution[397].



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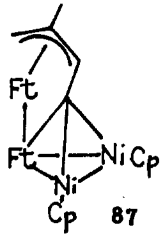

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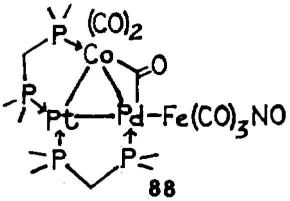

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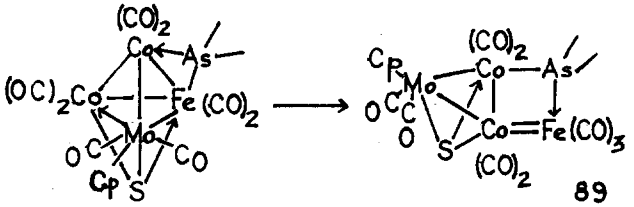
A "bowtie" $FeCr_4$ cluster, $[(Cp'Cr)_2(\mu\text{-}SR)(\mu_3\text{-}S)_2]_2Fe$ resulted when Fp_2 and $Cp'Cr(\mu\text{-}SR)_2(\mu\text{-}S)CrCp'$ ($Cp' = MeC_5H_4$, R = t-Bu) were allowed to react at 110° [398]. Reaction of many mixed-metal tetrahedral clusters with CO has been studied, and in some cases intermediates in cluster fragmentation have been intercepted. The product 89 (Eq. 48) was subjected to X-ray crystallographic study[399].



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POST-SCRIPT A computer scan of the list of references for this 1984 survey reveals again that the top three journals accounted for more than half of all work published in this field. Of about 400 citations, J. Organometal. Chem. and Organometallics share the lead, with 86 citations each. Then, in order, come J. Am. Chem. Soc. (46), Inorg. Chem. (40), and J. Chem. Soc., Chem. Comm. (30).

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