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IRON (1984)

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CONTENTS

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1. INTRODUCTION

This review covers the developments in the chemistry of compounds of iron reported in 1984. For operational reasons it has been necessary to consider only papers cited in Chemical Abstracts for that year. Therefore a little work from 1983 is included and some papers from late 1984 will be discussed in the survey for 1985.

The order of topics is that of decreasing oxidation state, but there are separate sections for biological and organometallic compounds.

2. GENERAL

X-ray photoelectron (XPE) spectra have been obtained [1] for many iron complexes and reveal a clear correlation between spin states and the satellite intensity in the Fe 2p spectra.

3. IRON (IV)

Moessbauer spectra show [2] the presence of high spin Fe(IV) in the perovskite type oxides $Ca_{1-x}Sr_xFeO_3$ ($x \leq 1$) or $Sr_{1-x}La_xFeO_3$ ($x < 0.6$), but at low Sr concentrations Fe^{4+} disproportionates to Fe^{5+} and high spin Fe^{3+}. Another perovskite material, $Ca_xLa_{1-x}FeO_y$ also contains Fe^{4+}; this was studied [3] by electron microscopy. The enthalpy of solution of $SrFeO_{2.97}$ in 6.32M HCl at 298K is -214 kJ mol^{-1} [4].

Among Fe(IV) coordination compounds, an alternating chain exchange mechanism was used [5] to account for the magnetochemistry of [FeO(hemiporphyrazinate)], whose doublet ground state was established by Moessbauer spectra. An Fe(IV) porphyrin complex with O^{2-} is invoked [6] to account for some electron transfer reactions of the Fe(III) - PPIXDME complex studied by cyclic voltammetry, ring-disc voltammetry and electronic spectra. Another example has been found [7] in which H_2O_2 oxidises an Fe(II) porphyrin to an $[FeO]^{2+}$ species. In related chemistry $[Fe(MeCN)_4(ClO_4)_2]$ disproportionates [8] H_2O_2 to form an $[FeO]^{2+}$ complex and $^1O_2$; this system oxygenates such varied organic molecules as ethanol or PPh_3. It is also suggested [9] that Fe(IV) is formed when Fe^{2+} reduces a Ti(IV) peroxo complex in sulphuric acid.

4. IRON (III)

4.1. Cyanide Complexes

There are a number of structural studies on $[Fe(CN)_6]^{3-}$ salts. $Li_3[Fe(CN)_6]$. 2(hexamethylenetetramine). $5H_2O$ has been shown [10] by X-rays to have the expected octahedral anion but Moessbauer spectra show there are two slightly different Fe environments. In $(Me_4N)_2Na[Fe(CN)_6].H_2O$ an $[Na(H_2O)]^+$ moiety is coordinated [11] to three N atoms, unsymmetrically occupying an N_6 cavity. $Zn_3[Fe(CN)_6]_2$ has been prepared [12] anhydrous and hydrated, with structural identification [13] of the usual anion for the 14 H_2O compound. In $(Me_4N)Mn[Fe(CN)_6].8H_2O$ the anion is linked [14] by CN bridges to $[Mn(H_2O)_4]^{2+}$ units.

In other work on $[Fe(CN)_6]^{3-}$ salts, Raman spectra were used [15] to study the phase transitions at ~ 130K for the K salt and $RbPb[Fe(CN)_6].3H_2O$ has been prepared. [16] Thermodynamic data have been obtained [17] for the formation of $\{Tl[Fe(CN)_6]\}^{2-}$. Linkage isomerism occurs [18] in $MFe(CN)_6$,(M=Al,Ga,In,Tl). Prussian blue materials in which either Fe^{2+} or Fe^{3+} are replaced by other metals have lowspin Fe when it is bound to C, but high spin when it is bound to N. [19]

Electrochemical work on $[Fe(CN)_6]^{4-}$ - $[Fe(CN)_6]^{3-}$ systems have highlighted the use of this system in photoelectrochemical cells. Such cells are improved [20] by roughening the electrodes and addition of CdSe. Spectroscopic studies in this field include reflectance spectra on Ni electrodes [21] and interfacial FTIR spectra [22].

There have been many studies of redox reactions of cyanide complexes of iron. A study [23] of reactions with reduced or oxidised plastocyanin led to a warning that tunnelling rates do not necessarily reflect the distance involved and that rearrangement energies must be considered. A kinetic investigation [24] of the reduction of $[Fe(CN)_6]^{3-}$ on Nb-doped $SrTiO_3$ electrode surfaces formed part of a study of semiconductor electrode - electrolyte interfaces. Other species oxidised by $[Fe(CN)_6]^{3-}$ are pyridinium salts (to pyrroles [25] or other more complex heterocycles [26]), quaternised 2-phenylquinolines [27] (to quinones with loss of Ph.), cresols, [28] nitrotoluenes, [29] phenanthrene, [30] glyceraldehyde phosphate, [31] 10-methylacridinium, [32] resorcinol [33] (first order in reactants and OH^-), and acrylic and lactic acids [34] (zero order in $[Fe(CN)_6]^{3-}$, catalysed by Ru (III)).

Compounds of the type $KFe[M(CN)_8]$ (M = Mo, W) have been prepared. [35] A cyclic voltammetry study of $[Fe(CN)_5L]^{n-}$ (L = various ligands, n = 2 or 3) showed [36] the effect on $E^\ominus$ was greatest when L was a good π-acceptor.

4.2. *Complexes of Anionic Nitrogen Ligands*

Amidino complexes $[Fe\{RNC(R)^1NR\}_3]$ (R = H,Me,Ph,*p*-tolyl) have been prepared [37] and are thought to be associated with Fe-N-C-N-Fe bridges like the isoelectronic carboxylates.

Magnetism studies and EPR and Moessbauer spectra showed [38] that the phthalocyanine (P) complex [Fe(P)Cl] exists in a mixture, not an equilibrium, of $^4A_2$ and $^6A_1$ states. Another Fe(III) - Pc complex was one of a number used [39] to modify a Pt electrode for the reduction of O_2 in fuel cells. Porphyrin complexes are treated in section 6.

4.3. *Amine Complexes*

The solvent extraction of Fe^{3+} by long chain amines such as dinonylamine has been studied.[40] Formation constants for glucosamine complexes have been determined.[41] The Moessbauer spectrum of an inosine -5'- monophosphate complex revealed [42] the effects of differing ribose configuration on the three iron environments.

The guanine (L) complex $[FeL_2Cl_3]$ is believed [43] to be polymeric with bridging guanines. Physical measurements suggested [44] hydrazine bridges made $[Fe(N_2H_4)_2(NCS)_2]$ an octahedral polymer. Complexes of dihydrobis (imidazolyl) borate have been prepared. [45]

Hydrolysis of the 1,4,7- triazacyclononane (L) complex $[FeLCl_3]$ at pH9 gave [46] $[Fe_2L_2(H_2O)_2O_2]^{2+}$ as I, PF_6 or ClO_4 salts, but Br^- gave the $[Fe_8L_6O_2(OH)_{12}]^{8+}$ cation in which 4[FeL] moieties are each linked to one of two other [FeL] units by an OH bridge and to one of two more [FeL] units by 2OH bridges; the latter fragments are linked by an O bridge to Fe which does not carry an amine ligand.

4.4. Pyridine Complexes

IR and Moessbauer spectra of $FeCl_3$ in pyridine suggest [47] the formation of a polymer based on $Fe(py)_3Cl_3$ units. The X-ray crystal structure of $[FeL_3Cl_3]$(L = 4-cyanopyridine) shows [48] this has a *mer*-configuration. 2-Hydroxypyridine forms a 1:1 complex with Fe^{3+} with a higher formation constant than its 3- or 4- isomers, which suggests [49] the O atom also coordinates. Pyridine -4-aldehyde (L) forms [50] $[FeL_2Cl_3].4H_2O$ and $[FeL_2(H_2O)_4]^{3+}$ complexes. Formation constants for pyridoxine-$FeCl_3$ complexes have been determined. [51]

The addition of Co^{2+} to $[Fe(2\text{-picolylamine})_3]Cl_2.EtOH$ affects [52] the temperature dependance of the highspin-lowspin transition, in line with the authors' model of "elastic" interactions between highspin and lowspin ions.

4.5. Diimine Complexes

There has been much structural work on bipy and phen complexes. Those studied are $[Fe(bipy)_3]^{2+}[Fe_2OCl_6]^{2-}$, *cis*-$[\{Fe(phen)_2Cl\}_2O]^{2+}$. $2Cl.4.5H_2O$ [53], $[Fe(phen)Cl_3L]$ (L = H_2O, MeOH, 1,2,4-triazole) (fac-isomers)[54],$[Fe(phen)Cl_4]^-$[54], $[FeL_2Cl_2]^+$ (L = bipy, phen) as $[FeCl_4]^-$ or $[FeLCl_4]^-$ salts [55], with a neutron diffraction study of a lattice bipy showing [56] a small distortion in this molecule, and a polarized neutron diffraction study of the $[FeCl_4]^-$ salt revealing [57] the spin distribution. In $[\{Fe(phen)_2(H_2O)\}_2O]^{4+}.4NO_3.5H_2O$ a typical structure was found [58] with a bridging FeOFe angle of 155.1°; ν_s(FeOFe) and ν_{as}(FeOFe) were located with ^{18}O and are excited in the Raman spectrum by the O^{2-}-Fe(III) CT band. These results were used to claim that the complex is a good model for haemerythrin and ribonucleotide reductase.

The complex $[Fe(phen)Cl_3(dmf)]$ was found [59] as an impurity in the preparation of $[Fe(phen)_3]^{2+}[Fe_2Cl_6O]^{2-}$. Formation constant determinations suggested [60] that bipy forms $[Fe(OH)_2(bipy)_n]^+$(n = 1,2) and $[Fe(OH)(bipy)_2]^{2+}$ at pH 3.5 - 5.0.

There have been many kinetic studies of $[Fe(bipy)_3]^{3+}$ and $[Fe(phen)_3]^{3+}$ reactions. The dissociation and racemization of these in SDS solutions showed [61] micelle formation effects. Their reduction by OH^- was reinvestigated [62] with a discussion of the relative probabilities of OH adduct formation and deprotonation of the ligand. The oxidation of bromomalonic acid by $[Fe(phen)_3]^{3+}$ has been studied, [63] as has the oscillating reaction [64] of this system with BrO_3^- and the cation's oxidation of 4-methoxytoluene by an outer sphere process. [65] There has also been a kinetic study [66] of the Fe^{3+} oxidation of NH_2OH to N_2 in the presence of phen and an unspecified Fe(III) - phen complex was the best catalyst for the decomposition [67] of $\overline{O\,(CH_2)_3\,C}(Me)OOH$. See also ref.147.

4.6. Schiff Base Complexes

This section includes various $>C = NR$ ligands, including semicarbazones, oximes and hydrazones. Time and space restrictions prevent detailed discussion of much work or giving structural diagrams.

Details are given [68] of the preparation of $[FeL(MeCN)_2]^{2+}$ where L is a macrocycle produced from PhCOCOMe and $NH_2C_3H_6NH_2$. In $[Fe(tim)(SCH_2Ph)_2]^+$ there are two cations in each unit cell, one with the axial phenyls tilted away from the macrocycle plane, the other with them tilted towards it. [69] Another crystal structure shows [70] square pyramidal coordination in a salicylidene semicarbazide complex.

There has been some interesting magnetochemistry. An Fe(III) bis complex of the salicylaldehyde - N - (3 - aminopropyl) - aziridine Schiff base is a spin-crossover compound [71] whose magnetism is affected by grinding and by solvation by CH_2Cl_2; an unusual result is that the rate of spin change is faster than the Moessbauer time scale if the material is more crystalline. [72] The spin state of complexes of related 14 to 16 membered macrocycles seems to depend [73] on axial ligands as well as ligand ring size, with spin states of 1/2, 3/2 and 5/2 all being found. Bis(imidazole) iron (III) complexes of the tetradentate ligand formed from hydroxyacetophenone, ethylenediamine and 2,4-pentanedione have ESR and visible spectra for a highspin - lowspin equilibrium, being thermochromic. [74] The magnetism of $[Fe_2L_2O]$ (L = tetradentate isothiosemicarbazide) has been studied, [75] and the mixed metal complex $[(PhOFe)L(CuCl_2)]$ (L is a sal_2pn derivative) is also antiferromagnetic. [76]

In solution chemistry some binuclear complexes with a sal_2en type ligand have been studied by differential pulse polarography [77], and a kinetic study has been made [78] on the electrolytic reduction of $[Fe(sal_2en)Cl]$. The hydrolysis of an Fe(III) sal_2en carboxy derivative at various pH shows the sixth coordination site is occupied by H_2O or OH^-; an $H_3O_2^-$ bridged complex is also suspected. [79] Many other, mostly preparative, studies require no detailed comment. [80 - 99]

4.7. *Oxides*

This section omits many reports where only small proportions of Fe are present or where the work is primarily technological in character.

The breakdown of passive films on Fe by Cl^- has been described [100] as involving the formation of a polymeric hydrated oxide.

There have been many studies on Fe_2O_3. For α-Fe_2O_3 these include the preparation of needles, [101] the DTA of its formation from γ-FeOOH, [102] the anisotropy of its electrical properties, [103] the effect of particle size on its Moessbauer spectrum, [104] and on the catalysis of the oxidation of 1-butene to butadiene, [105] the kinetic study of its solution in acids using Me_2COH radicals and [Fe(edta H_2)][106]. For γ-Fe_2O_3 there have been studies of preparation by sputtering, [107] neutron diffraction, [108] temperature dependence of the Moessbauer spectrum, [109] and the transition to α-Fe_2O_3. [110] It was not specified which form of Fe_2O_3 was formed [111] in the pyrolysis of $[Fe(urea)_6]^{3+}.3NO_3^-$. Together with MgO or Al_2O_3, Fe_2O_3 cyclises [112] 1,3-pentadiene to cyclic hydrocarbons. Fe_2O_3 absorbs H_2S strongly, but if O_2 is present S is formed at low and SO_2 at high temperatures. [113] The hydrogenation of CO over Fe_2O_3 - SiO_2 is most successful when $FeCl_3$ is used to prepare the catalyst. [114] In Ni-doped Fe_2O_3 the O vacancies play a key role in the oxidation of CO. [115] The Moessbauer spectrum shows [116] at least two components with different spin are present in the high pressure phase of Fe_2O_3. Unspecified Fe oxides enhance the Raney Ni - Zn catalysed hydrogenation of 2-ethylanthraquinone. [117]

Defect-free crystals of Fe_3O_4 of at least 5g weight can be made [118] by a method called "skull-melting". Gas-phase reactions of FeO^+ with alkanes have been observed [119] using collision induced dissociation mass spectrometry.

There have been many reports of work on mixed oxides which contain Fe^{3+}. These include the determination [120] of the free energy of formation of $LiFeO_2$, the preparation of the Na_2O-structured Li_5FeO_4 [121] and the X-ray crystal structures [122] of the ordered and disordered forms of $LiFe_5O_8$. A thermodynamic study [123] of the Fe-Na-O system gave ΔG° for

Na_2FeO_2,$Na_2Fe_2O_4$ and Na_4FeO_3. In work with alkaline earth oxides , the magnetoplumbite structure (based on Fe_3O_4) was found [124] for $Ca_{2.95}Fe_{14.85}O_{25}$, and a solubility study of $BaFe_{12}O_{19}$ in BaO-B_2O_3 suggests [125] this is a good medium for growing single crystals. Mixed oxides with transition metal 2+ ions have been studied, including the formation [126] of Fe_2NiO_4 from the copyrolysis of their Tutton salts, the growth [127] of long crystals of Fe_2MO_4(M = Co,Ni). These and other ferrites (Fe_2MO_4) have been made [128] by the pyrolysis of $Fe_2M(C_2O_4)_3(N_2H_6)$,(M = Mn,Co,Ni,Zn). The magnetic anisotropy of $Co_{0.16}Fe_{2.84}O_4$ has been reported. [129] Fast electron exchange was found [130] in $Fe_2Cu_xGe_{1-x}O_4$(0.6<x<0.8).

The Moessbauer spectrum shows [131] Fe^{3+} occupies both octahedral and tetrahedral sites in $FeAlO_3$, but most M^{3+} - Fe^{3+} mixed oxides involve lanthanide ions. A hydrothermal synthesis of $FeLaO_3$ crystals is reported.[132] CO oxidation by $FeLa_{0.8}Sr_{0.2}O_3$ rapidly increases [133] above 240 °C. X-ray and IR studies of $LaFe_xNi_{1-x}O_3$ are recorded. [134] There has been a kinetic study [135] of garnet formation in sintering Fe_2O_3 with Y_2O_3, and IR,DTA, and X-ray diffraction studies of this system showed [136] the formation of $YFeO_3$ and $Y_3Fe_5O_{12}$. Ordering temperatures have been found [137] for Y_2FeSbO_7, Bi_2FeSbO_7 and $BiYFeSbO_7$. Moessbauer spectra showed [138] Sc^{3+} occurs in tetrahedral and dodecahedral sites in $Eu_{3-x}Sc_{2+x}Fe_3O_{12}$. Antiferromagnetic resonance has been studied [139] in $FeTmO_3$. There is an unusually low absorption [140] at 1300 nm in $Gd_{0.2}Y_{2.8}Fe_5O_{12}$.

Some mixed oxides formally contain M^{4+}. The Fe_2O_3 - GeO_2 system produces [141] Fe_2GeO_5 and $Fe_8Ge_3O_{18}$, the latter being a new structure type, but if Fe is present, $Fe_4Ge_2O_9$, paramagnetic above 100K, is formed. [142] The $BaM_2Fe_4O_{11}$(M = Sn,Ti) ferrites have Fe^{3+} in octahedral and tetrahedral sites, but those in the latter can move between two sites. [143] There have been X-ray powder and electron microscopy studies

[144] of Fe_2TiO_5.

Bridging oxo ligands occur in $[\{FeCl_3\}_2O]^{2-}$, studied as alkylammonium [145] or $[Mg(dmf)_6]^{2+}$ [146] salts, and in the *fac*-configured $[\{Fe(H_2O)_3(phen)_2\}_2O]^{4+}$ cation.[147]

4.8. Hydroxides

A thorough review has been published [148] on the hydrolysis of Fe(III) salts. The conditions are described [149] for preparing the $Fe(OH)_3$ catalyst for reducing NO to NH_3. A new form of $Fe(OH)_3$ has been prepared [150] by the oxidation of $FeSO_4$ aqueous solutions by $[\{Co(en)(dien)\}_2O_2]^{4+}$; it has two Fe sites of about equal abundance, is paramagnetic above 190K, superparamagnetic from 97.5 to 190K and ordered below 97.5K. Magnetization measurements near 1K were used to find [151] the proportion of hydroxo oligomers in solutions of Fe^{3+} salts in aqueous ${}^{i}PrOH$.

Most work in this section concerns FeOOH. To make the α-form free from Fe_3O_4 one must use aqueous $FeSO_4$ at pH between 5 or 12 at below room temperature. [152] Other work has been published [153] on this reaction. An IR study [154] concentrated on surface behaviour and the transition to Fe_2O_3. Conditions have been described [153, 155] for forming β-FeOOH by hydrolysis. The Neel temperature of β-FeOOH depends on the preparative conditions, with interstitial H_2O having an effect. [156] A discussion [157] of β-FeOOH Cl_n particles, synthetic or natural, dealt with the possible role of this material in intercalating aminoacids for prebiotic polypeptide formation. The dehydration *in vacuo* of β-FeOOH was monitored [158] by magnetization studies. The γ-form of FeOOH was formed [159] in the air oxidation of aqueous $FeCl_2$ at pH 2-5. The light induced dissociation of γ -FeOOH is assisted when citrate is present. [160] Neutron diffraction and Moessbauer experiments showed [161] ferromagnetic coupling between layers of Fe^{3+} and antiferromagnetic coupling between layers. The deposition of hydrated FeOOH on gold electrodes has been studied. [162]

4.9. Alkoxides and Complexes with OR^- Ligands

This section includes phenoxides, catecholates and complexes of polydentate ligands in which an OR^- entity plays a significant role.

The formation of 1:1 Fe(III) complexes of nitro-, dinitro- and methylphenoxides in dmso was studied [163] by EPR, which showed reduction to Fe(II) was easy. Two crystal structures of Fe(III) complexes containing bridging OEt ligands have been reported; [164] one has a binucleating ligand, the other is $[Fe_2(acac)_4(OEt)_2]$. Comparison of the magnetochemistry of the two compounds and of similar OMe bridged ones showed J values did not follow the usual rules for antiferromagnetism and the authors wondered whether ferromagnetism was also involved. One of many bimetallic isopropoxides studied [165] is $FeAl_3(O^iPr)_{12}$.

Among studies of catechol type complexes there have been a kinetic study [166] of complex formation, a study [167] of substituent effects on formation constants with reference to modelling siderophores and determination [168] of the formation constants of 4-nitrocatechol complexes. Formation constants have also been determined [169] in the Fe^{3+} - pyrogallol - edta system.

Fe(III) is photoreduced in solutions containing gallocyanine, which may use both catechol and azole-carboxylate bidentate sites; the system might be a model for bleomycin complexes. [170]

There has been a normal vibrational analysis [171] of a tris complex with N-hydroxyurea. Alkoxo bridges have been found [172] in $[Fe_2L_2(NO_3)_2]^{2+}$ [HL = bis(2-benzimidazolylmethyl)-(2-hydroxyethyl)amine]; this antiferromagnetic compound shows pentagonal bipyramidal Fe coordination. An Fe(III) complex of catechol-3,5-disulphonate catalyses the conversion of benzenes to phenols. [173] A complex with an azocresol ligand is reported. [174]

4.10. Carboxylates

This section includes monocarboxylates, dicarboxylates, aminoacid complexes, edta complexes and those of other ligands containing O_2CR^- units.

Studies of allegedly simple $Fe(O_2CR)_3$ compounds include the thermal decomposition [175] of iron (III) benzoate and $[Fe_2(fumarate)_3.5H_2O]$, $Fe(O_2CR)_3$ species are claimed [176] as participating in the solvent extraction by $C_mH_{2m-1}(CH_2)_nCO_2H$ in benzene (m = 5,6; n = 0-3). The extraction of Fe^{3+} from H_2O by octanoic acid or α, α-dialkylcarboxylates is assisted by the presence of Al or Cr, which give heteropolynuclear complexes. [177] The reaction of $[(CpCrS)(SCMe_3)Fe(CO)_3]$ with carboxylic acids gives [178] $[Cr_2FeS(O_2CPh)_6]$ or $[(CpCr)_3(FeO_2CCMe_3)S_4]$; the structure of the latter comprises a cubane system with monodentate pivalate. [179] The products of the reaction of $FeCl_3$ with CF_3CO_2H are given [180] as $[Fe_2Cl_2(O_2CCF_3)_4]$,$[Fe_2Cl(O_2CCF_3)_5]$ and $Fe(O_2CCF_3)_3$, which are all believed to have polymeric structures with bidentate carboxylates and octahedral Fe.

The pyrazolylborate complexes $[Fe_2(O_2CMe)_2(HBpz_3)]$ and $[Fe_2(OH)(O_2CMe)_2(HBpz_3)_2]^+$ have been thoroughly investigated. They both have three bridging ligands, are antiferromagnetic and are claimed to be analogues of haemerythrin. [181, 182]

The magnetochemistry of the familiar $[Fe_3L_3O(RCO_2)_6]^+$ structure has been investigated [183] for aqua complexes of unsaturated acids and the (L = py, R = CF_3) complex has been prepared [184], but $Fe_3O(O_2CCF_3)_7.H_2O$ has two different Fe sites. [184] The electrochemistry of compounds erroneously described [185] as $[Fe_nCr_{(3-n)}(O_2CMe)_6(OH)_2]^+$ is reported. The Fe(II, III,III) mixed valence complex $[Fe_3(H_2O)_3O(O_2CPh)_6]$ gives Fe_2O_3 on thermal decomposition [186], possibly through the intermediates $[Fe_3(OH)_2O(O_2CPh)_4]^+$ and "$Fe(O_2CPh)_3$". Moessbauer spectra are recorded [187] for $[Fe_3O(O_2CR)_6]$, (R = CF_3, CCl_3), at different temperatures. Two $[Fe_3O]$ triangles are fitted together in $[Fe_4O_2(H_2O)_6(O_2CCF_3)_8].2H_2O$, which has one carboxylate bridge between each pair of Fe atoms, leaving half the O_2CCF_3 ligands monodentate; the compound is prepared [188] by evaporation in air of solutions of $[Fe_3(H_2O)_3O(O_2CCF_3)_6]$. $3.5H_2O$. The complex "$Fe_8L_2(L')_2O(O_2CMe)(OH)_{19}$", (HL = lactobionic acid, L' = dextran glucopyranose) has been used [189] for parenteral iron therapy.

Research on oxalate complexes includes the use of $K_3[Fe(C_2O_4)_3]$ as catalyst for the photochemical dissociation of arenediazonium salts, [190] the thermal decomposition of this salt and $Fe_2(C_2O_4)_3.5H_2O$ on various supports [191] and of the ammonium salts of $[Fe(C_2O_4)_3]^{3-}$ and the tricitrato complex. [192] The kinetics of the pyrolysis of the mixed metal oxalate complexes $[Fe_2Mg(C_2O_4)_8]^{8-}$, $[Fe_2Mg(C_2OH)_4]^{4-}$ and $[Fe_2Mg(C_2O_4)_2(OH)_6]^{2-}$ is also reported. [193] A 1:1 complex with maleic acid monoanion is reported. [193a]

The glycine complex $[FeCl_3(glycine)_3]$ has been prepared, [194] and the X-ray crystal structure of $[Fe_2Cr(H_2O)_3O\ (glycine)_6]^{7+}$ perchlorate is reported. [195] The Fe^{3+} - H_2O_2 - histidine system catalytically oxidises indigo carmine. [196] Formation constants were measured [197] for Fe^{3+} complexes with 3-imidazolylpropenoic acid.

The ever popular complexes of edta continue to appeal. Some readers may be comforted that a repeat [198] of the 1975 determination of the crystal structure of $Na[Fe(H_2O)edta]$ gives the same result. The K,Ag and Tl salts are similar. [199] Studies on the adsorption of edta and its Fe complexes on Fe_3O_4 clarified [200] the dissolution of the latter, which is faster when Fe^{2+} is added; the larger formation constant for the Fe(III) complex over its Fe(II) sister assists electron transfer in dissolution. Another report [201] also discusses this reaction. Two papers [202] describe the use of edta and similar complexes of Fe(III) as mediators in microbial fuel cells because of their fast electrode reactions and reduction by E. coli. Formation constants of hexamethylenediaminetetraacetate complexes are reported. [203] Reports on salicylate-type complexes include two preparations, [204,205] and a kinetic study which showed [206] that edta displaces salicylates from Fe(III) complexes by an associative mechanism.

Citrate complexes of Fe(III) can be solvent extracted with long chain alkylamines in $CHCl_3$. [207] A kinetic study of complexation of Fe^{3+} by citric, diglycolic and tartaric acids showed [208] that 1:1 complex formation involved $[Fe(OH)]^{2+}$. The formation constant of the 2:1 complex with tartrate has been measured; [209] coordination is believed to occur through a carboxyloxygen and two alkoxide oxygens. The $[Fe(tartrate)_2(L)]$ complexes, (L = saccharose, sucrose] have been studied [210] spectrophotometrically. Tartrate also forms [211] a mixed metal complex with Fe^{3+} and Cd^{2+}.

Formation constant determinations [212] of complexes with picolinate or other pyridinecarboxylate ligands indicate some are dinuclear. The dipicolinate (X) complexes $[FeX_2]^{n-}$, (n = 1,2) have been studied; [213] kinetic measurements show ascorbate dianion reduces the Fe(III) complex 10^5 times faster than the monoanion.

The formation of red and blue complexes of Fe^{3+} with chromoxane cyanine R, probably a carboxylate ligand, is discussed [214] for use in histological staining.

4.11. Diketonates

Complexes with 2,4-pentanedionate predominate. There has been a kinetic study [215] of the formation of its 1:1 complex with Fe^{3+}. For $[Fe(acac)_3]$ there have been measurements of ΔH of sublimation and fusion, [216] and cyclic voltammetry. [217] This molecule catalyses the cyclodimerization of norbornadiene [218] and the photochemical oxidation of tetralin by O_2. [219] $[Fe(acac)_3]$ reacts with $AlEt_3$ to give $[Et_2Al(acac)]$ and $[Al(acac)_3]$, but the Fe products were not identified. [220] $[Fe(acac)_3]$ with $AlCl_3$ or $SnCl_2$ catalyses the hydrogenation of 1-hexene and 1,3-pentadiene by forming an Fe alkene complex. [221,222]

In work with other diketonates, $[Fe(dbzm)_3]$has been found [223] to catalyse the coupling of PhMgBr to PhCH:CHBr to give styrene. Formation constants have been measured [224] for complexes with isonitrosoacetylacetonate. A kinetic study [225] of hemiacetal formation in the reaction of [Fe(hfacac)$_3$] with MeOH showed two molecules of the latter are involved in the rate-determining step, one being used to assist proton transfer. The vacuum sublimation of Fe(III) thenoyltrifluorothiodiketonate is described. [226] Fe(III) trifluoro (nicotinoyl) acetonate is another spin-crossover compound; 27% of it is high spin at room temperature. [227] [Fe(OH)]$^{2+}$ catalyses the ketoenol tautomerisation of 2,4-pentanedione or ethylacetoacetate better than Fe^{3+}, which is less effective than some M^{2+} ions. [228]

Tetracycline may coordinate as a diketonate; there have been kinetic [229] and thermodynamic [230] studies of its complexation with Fe^{3+}. Arnebin-I might also coordinate to Fe^{3+} as a diketonate. [231]

4.12. Complexes of Oxyanions

This miscellaneous section includes both inorganic and organic ligands. Moessbauer spectra were used [232] to study ordering processes in the Fe(III) layered silicates nontronite and celadonite. Other work on silicates did not include material of special interest to this journal's readers.

It was another busy year for phosphates. New compounds were prepared. [233] The crystal structures of synthetic tavorite, LiFe(PO$_4$)(OH,F) [234] and of $Na_3Fe_2(PO_4)_2(OH)_2F$ [235] show FeO$_6$ octahedra and PO$_4$ tetrahedra, but IR spectra were taken [236] to show FeO$_4$ and PO$_3^-$ being present in other phosphates. Among polyphosphates there have been more preparations, [233] including KFe(H$_2$P$_2$O$_7$)$_2$ [237], Fe$_2$P$_6$O$_{18}$ and its nonahydrate, [238] and K$_2$Fe$_2$P$_8$O$_{24}$. [239]

The conditions for precipitation of NH$_4$Fe$_3$(OH)$_6$(SO$_4$)$_2$ are described. [240] The Al and Fe structures in KM$_3$(OH)$_6$(SO$_4$)$_2$ have been compared. [241] X-ray determination of the electron distribution in monoclinic Fe$_2$(SO$_4$)$_3$ found [242] the electron density bisecting the Fe-O bonds. The pyrolysis of Fe(OH)SO$_4$ and Fe$_2$O(SO$_4$)$_2$ has been studied. [243] Fe$_2$(SO$_4$)$_3$.7H$_2$O can catalyse acid esterification. [244] Among other metals, Fe dissolves electrolyticaly in dmso

SO_2 to give [245] $Fe_2(dmso)_{10}(S_2O_7)_3$ (SO_2. Polymeric octahedral structures with bridging anions are suggested [246] for such selenites as $Fe_7(OH)_3(SeO_3)_9.18H_2O$. The reaction of $Fe_2(MoO_4)_3$ with $FeCl_3$ at 350° in vacuum gives $FeMoO_4$ Cl, a layer structure with corner - shared MoO_4 tetrahedra and FeO_4 Cl square pyramids and showing Fe-Fe interactions across layers. [247]

Many organic anionic ligands are of biochemical origin. Formation constants have been determined [248] for arabinose and galactose complexes. Fe(III) complexes of sorbitol, glucuronic acid and glucosamine have been prepared, [249] the last being given a dinuclear formulation. Various complexes of 2' - or 3' - adenosine monophosphates have been prepared, and phosphate oxygen is the probable strongest coordination site. [250] Other complexes reported include those of hydroxyanthraquinones, [251] hydroxamates [252] (of which a special case is one derived from 1-hydroxy-2-indolinone [253]), and carboxylate hydrazides. [254]

4.13. Water Complexes

By studying various Fe(III) chelates whose structures did (or did not) contain coordinated H_2O it was found [255] that the presence of this was necessary to achieve the Fenton reaction of forming OH in reaction with O_2^- and H_2O_2. Fe^{3+} catalyses the hydroxylation of benzene to phenol by H_2O_2. [256] There has been a kinetic study [257] of the oxidation of aqueous SO_2 by Fe^{3+} salts.

4.14. Complexes of Neutral Oxygen Donors

New preparations include $[FeL_4Cl_2]^+$, (L = 2-aminopyridine-N-oxide), [258] and $[Fe\{Ph_2P(O)CH_2P(O)Ph_2\}_3]^{3+}$ [259]. The dmso ligands are O-bonded in $[FeCl(dmso)_5]^{2+}[Fe_2OCl_6]^{2-}$ at 103K but are disordered at higher temperatures. [260]

Vibrational spectra were used [261] to study the conformation change in MeP(E) $(OC_6H_4OC_2H_4)_2O$ when it is coordinated to $FeCl_3$. $[Fe(dmf)_3Cl_2]^+$ $[FeCl_4]^-$ converts ketophenols to benzofurans. [262a] Cryptand (222) complexes are reported. [262b]

4.15. Sulphides

Material concerning cluster complexes and other biochemical model systems can be found in section 6.

There has been a kinetic study [263] of the effect of SO_4^{2-} on the reduction of Fe^{3+} by S^{2-}. The complex magnetic behaviour of $KFeS_2$ was analysed [264] with the Néel temperature assigned as the sharp maximum at 12.5K rather than the broad maximum at 250K; there is exchange between nearest and next-nearest neighbours in chains and between chains. $FePS_3$ has a Néel temperature of 120K; the resultant moment is perpendicular to the layer planes, with ferromagnetic nearest neighbour interactions. [265]

4.16. Thiolates and other SR^- Complexes

Biochemical model compounds are described in section 6. The Ph_4P salts of $[Fe(SR)_4]^-$, $[R = 2,4,6-(^iPr)_3\ C_6H_2]$ have been prepared and show a large FeSC angle of 112°; they are stable to self reduction to Fe(II). [266] Complexes of the S-N donors 1-amidino-2-thioureas have been prepared. [267]

4.17. Dithiocarbamates

A new example [268] of this class is $[Fe(S_2NHC_6H_4\text{-}4\text{-}OEt)_3]$. An extensive study of the structures of many tris (dithiocarbamato) complexes shows that Fe-S distances average 2.30 Å for lowspin and 2.45 Å for highspin compounds; the effect is an apparent increase in the vibrational amplitude of S atoms. [269] For the tris-morpholinedithiocarbamate as CH_2Cl_2 solvate highspin was found [270] from 77 to 320K; in solvates of this compound the solvent molecules collect round the morpholine moiety, induce its deformation and bring about a change to lowspin. Notwithstanding this result the unsolvated compound is described [271] as exhibiting a triplet - quintet equilibrium. Other workers [272] concluded that the only factor governing the spin state was steric hindrance which affects the SCS angle, with greater interference giving lowspin compounds.

The structure of $[FeBr(S_2CN^{i}Pr_2)_2]$ is normal. [273] Heat capacity measurements on $[FeI(S_2CNMe_2)_2]$ at 0.4 - 300K showed [274] 40% monomer and 60% dimer at low temperature and zero field splitting about 100 times greater in magnitude than antiferromagnetic exchange.

4.18. Complexes of Other Sulphur Ligands

New preparations of this type are $[Fe(S_2CC_6H_4OH)_3]$ (all three isomers [275]), $[Fe(H_2O)_2X_2]Cl$, [HX = 3-(4-pyridyl)triazoline-5-thione], [276] $FeCl_3$ complexes of substituted dithiobiurets (some of which may be dimers), [277] and $[Fe_2(py)_2(P_2S_8)_3]$. [278] Formation constants have been determined [279] for Fe(III) complexes of various $(RO)_2PS_2^-$ or $Me(RO)PS_2^-$.

4.19. Fluorides and Fluoro-Complexes

FeF_3 and $FeF_3.\frac{1}{3}H_2O$, described as hexagonal tungsten bronzes, have been shown [279] by Moessbauer spectroscopy to show magnetic ordering temperatures of 97± 2 and 128.7 ± 0.5K respectively; the spectrum of amorphous FeF_3 has also been described. [280] Thin films of FeF_3 have been obtained by vacuum sublimation and characterised spectroscopically. [281]

A Moessbauer study [282] of $KFeF_4$ above T_N showed spin fluctuations in this two-dimensional antiferromagnet. The same technique showed phase transitions in $RbFeF_4$ at 381 and 417K, the first abrupt and the second gradual, as the FeF_6 octahedra tilt with respect to the c-axis. [283] Angle-resolved photoemission from K_2FeF_5 and SCF $X\alpha$ calculations for $[FeF_6]^{3-}$ are reported. [284] Neutron diffraction, magnetization and Moessbauer studies for $LiMnFeF_6$ show [285] that the β-phase is ferrimagnetic below 113K with some Fe^{3+} not coupled to others. Revision of the space group for NH_4MnFeF_6 gave better R values and a more regular structure. [286] Moessbauer spectra are also reported [287] for Ba_2NiFeF_9, Ba_2CrFeF_9 and $NaBaFe_2F_9$.

The X-ray crystal structure of $Fe_3F_8.2H_2O$ shows [288] two Fe^{3+}, coordinated by six F^-, for each Fe^{2+}, coordinated by four F^- and two

H_2O. For this compound and its Mn^{2+} analogue there is a Néel temperature near 157K when only the Fe^{3+} spins order; this is anthropromorphically described [289] as "idle spin" due to "magnetic frustration".

4.20. Chlorides, Bromides, Iodides and Halide Complexes

The enthalpy of formation of $FeCl_3$ is reported [290] as - 396.02kJmol^{-1}. Similar data for four hydrates have also been determined. [291]

The biggest topic in this section is the intercalation of $FeCl_3$ in graphite. There is a new review on this. [292] Moessbauer and powder X-ray methods indicate [293] that intercalation involves the formation of $[FeCl_4]^-$, but other workers found [294, 295] Fe^{2+} sites, with Cl atoms surrounding a vacant site. Neutron diffraction studies [296, 297] showed two-dimensional ordering below 30K for the first and second stages, with the former developing three-dimensional ordering below 3.8K. $FeCl_3$ was one of the halides used [298] for graphite intercalation for Li batteries. An ammonia complex was also investigated [299] for energy storage. The $FeCl_3^-$ graphite intercalate when reduced catalyses the oxidation of SO_2 in H_2O, with probably both $FeCl_2$ and FeO(OH) present. [300] Thermal analysis studies [301] gave ΔH and ΔS for its decomposition to $C_{12}FeCl_3$ or $C + FeCl_3$. Simultaneous intercalation of $FeCl_3$ and $CoCl_2$ gives alternate layers of each; the material has magnetic phase transitions at 8.8 and 8.0K. [302]

FeOCl is an effective host for intercalation compounds. With RNH_2, (R up to C_{18}) the basal separations increase from 7.92 Å to 57.74 Å as about 0.55 mol of guest enter. [303] Moessbauer spectra of the 2-Mepy intercalate show [304] some Fe^{2+} sites form as well as an "intermediate valence" site. A phase study of NH_3 and RNH_2 intercalates found [305] that HCl was lost above 120°C. Another X-ray and Moessbauer investigation supported [306] a charge transfer model for bonding. Reactions of the aminopyridine (L) intercalate $FeOClL_{0.25}$ with methanol and ethylene glycol gave [307] FeO(OMe) and $Fe_2O_2(C_2H_4O_2)$.

New work on $[FeCl_4]^-$ and other halo complexes includes the synthesis and UV and EPR spectra [308] of the $[Mo\ (Et_2dtc)_4]^+$ salt of $[FeCl_4]^-$, and the synthesis [309] of the $[Ti(cp)_2Cl(MeCN)]^+$ salt. The crystal structure of the bis - 8-hydroxyquinolinium chloride salt is reported. [310] $FeCl_3$ reacts with $MgCl_2$ in THF to give $[MgCl(THF)_5]^+[FeCl_4]^-$ which converts to $[Mg(THF)_4(\mu\text{-}Cl)_2FeCl_2]$, whose structure is also reported. [311] The range of compounds $Li(Fe,Al)Cl_4$ have been prepared and their conductance and magnetism studied. [312] In the near IR polarized single crystal spectrum of $Cs_2FeCl_5.H_2O$ at 10K overtone and combination water bands were seen, [313] but the K analogue has a more complex spectrum. The self-ionisation of $FeCl_3$ in organic solvents has been studied by EPR; [314] in acetic acid dimerisation and trimerisation were suspected from conductance and molecular weight data. [315] $[FeCl_4]^-$ is also formed by the reaction of Fe_2O_3 with PCl_5 in $POCl_3$ or with S_2Cl_2 and chlorine; the former reaction also produces $2FeCl_3.3POCl_3$, but reaction with $POCl_3$ alone gives $FeCl_3$ and $Fe(PO_2Cl_2)_3$. [316] The resonance Raman spectrum of $[Et_4N][FeI_4]$ is reported. [317] Kinetic studies [318,319] of the reaction of $[FeX_4]^-$ with Y^-, (X,Y = Cl,Br,NCS) show ion pairs may be formed in an associative mechanism but the dielectric constant of the solvent is important. Solvent extraction of $[FeCl_4]^-$ can occur by the quaternary ammonium salt "Hyamine 1622", [320] or $Me(octyl)_3NH^+$, which selectively transports Fe^{3+} through a liquid membrane. [321]

New reactions of $FeCl_3$ include the formation of a complex with the cyanamide derivative ${}^iPr_2NC{\vdots}N$ and the complex's reaction with chloroalkanes. [322] $FeCl_3$ catalyses the bromination of anisole, [323] the copolymerization of styrene and methylmethacrylate, [324] initiates the polymerization of S-carboxyalkylcysteine (though other chlorides are better), [325] is used with $PdCl_2$ in the hydrochlorination of ethene, [326] in the benzylation of alkyltoluenes, [327] the conversion of azoxybenzenes into 4-chloroazobenzenes,

[328] and the oxidative coupling of isoeugenol, [329] and it reacts with derivatives of benzilic acid. [330]

4.21. Other Iron (III) Compounds

The iron (III) complex of bleomycin A_2 is highspin at pH 4, but when the solution pH is raised to 7 a lowspin complex forms as the α-amino nitrogen of β-aminoaniline coordinates; lowspin complexes can be formed at pH 4 by adding NCS^-, N_3^- or $S_2O_3^{2-}$ or DNA, which can displace these ligands, possibly with interaction between DNA and bleomycin. [331]

5. IRON (II)

5.1. Cyanide Complexes

A series of papers [332-4] investigates the overtone and combination water IR spectrum in $K_4[Fe(CN)_6].3H_2O$. Electronic spectra show [335] charge transfer bands between $[Fe(CN)_6]^{4-}$ and pyridinium type cations.

A number of mixed metal complexes are described. The photodecomposition of n-CdS in $[Fe(CN)_6]^{4-/3-}$ cells containing K^+ and Cs^+ leads to the formation [336] of $(K,Cs)_2$ $[CdFe(CN)_6]$, while a thermodynamic study of the interaction of $[Fe(CN)_6]^{4-}$ with a Tl amalgam electrode showed [337] the formation of $[TlFe(CN)_6]^{3-}$. Trivalent lanthanide ions appear from IR spectra to bond to N atoms in forming $K[MFe(CN)_6].4H_2O$. [338,339] The thermal decomposition of $UFe(CN)_6$ has been studied. [340]

Reactivity studies of $[Fe(CN)_6]^{4-}$ include the pyrolysis of R_3S^+ and Ph_2I^+ salts, [341] its use as a commercial NH_3 synthesis catalyst, [342] its reaction with HCHO under light to give aminoacids, [343] its use with $[Cr(C_2O_4)_3]^{3-}$ to generate H_2 from irradiated 10^{-2}M aqueous solutions ($FeSO_4$ can also be used), [344] and unspecified products from a reaction with styrene oxide. [345]

In the area of mixed ligand complexes, electronic spectra were used [346] to study solvent effects in $[Fe(CN)_5py]^{3-}$, $[Fe(CN)_4\ bipy]^{2-}$ and $[Fe(CN)_2(bipy)_2]$. There has been a careful discussion [347] of solvent and pressure effects on the substitution kinetics of $[Fe(CN)_5L]^{3-}$, (L = 4-CNpy, 2-Mepyrazine). A bright red material is formed when Ni^{2+} or a nickel electrode reacts with $[Fe(CN)_4\ bipy]^{2-}$ and this is believed to have a nitrogen from one CN^- bound to Fe [348] The reaction of pyridine with $[Fe(CN)_5(Hen)]^{2-}$ is unusually fast in MeCN. [349] $[Fe(CN)_6]^{4-}$ reacts with $[Co(en)_2(NH_3)(H_2O)]^{3+}$ to give $[(NC)_5Fe(\mu\text{-}CN)Co(NH_3)(en)_2]$ which shows an intervalence transition. [349] An ingenious kinetic study of the intramolecular electron transfer in $[(NC)_5FeLCo(dien)]^{2-}$, (L = pyrazine-2,6-dicarboxylate) shows that the outer sphere reaction between $[Fe(CN)_5(LH)]^{4-}$ and $[Co(dien)L]^+$ is 5.7 times faster at 25 °C, suggesting that a stacked ion-pair may give a suitable structure. [350]

5.2. Phthalocyanines and other Complexes of Anionic Nitrogen Ligands

New preparations include phthalocyanines with 4 ^{t}Bu, 8 Me or MeO or 16 Cl substituents and their bispyridine derivatives. [351] Raman spectra are recorded [352] for Fe(II) tetrasulphonated phthalocyanine, as has the EPR spectrum of [FePc] in a polymer matrix. [353] The question of the electronic groundstate of [FePc] has been re-examined; electron density maps and Moessbauer spectra indicated [354] a ^3E_g A ground state, while magnetochemistry and Moessbauer studies led to a description [355] as mostly ^3A.

[FePc] has been prepared as a stable electrode film on Au, [356] and C electrodes in fuel cells are improved by incorporating Fe tetrachlorophthalocyanine. [357] Magnetochemistry and dielectric measurements were included in a study of biphthalocyanines, which appear to undergo a structural change near 340K. [358]

Fe(II) polyphthalocyanine catalyses the decomposition of H_2O_2 by two mechanisms. [359] Partial oxidation is located on Fe in $[Fe(Pc)(pz)I_{0.26}]$, which contains I^-, and $[Fe(Pc)(bipy)I_{1.06}]$, which contains both I^- and I_3^-. [360] Reaction of [FePc] with KCN gives [361] [Fe(PcH)CN] and [Fe(Pc)CN] ; the conductance of both has been measured. [362]

The preparation of $FeX_2.2H_2O$, ($HX = H_2N.NH.C(S)OEt$) is reported. [363]

5.3. *Ammonia Complexes*

Molecular orbital calculations, [364] of which few particulars are given, purport to show that Fe^{2+} (and other metal ions) do not use d orbitals in forming complexes with F^-, H_2CO and H_2O, but they do with NH_3!

5.4. *Amine Complexes*

The trinuclear complex of 4-ethyltriazole (L), $[Fe_3L_6(H_2O)_6]^{6+}$ exhibits a gradual highspin - lowspin transition for the central Fe^{2+} ion; its Fe-N bonds contract 0.14Å on cooling, but no such effect is seen for the outer Fe^{2+} and there is no magnetic exchange. [365] In related work $[Fe(1\text{-propyltetrazole})_6]^{2+}$ also shows a spin equilibrium, [366] the first such to be convertible by light, with the $^5T_{2g}$ state stable below 50K. New preparations include other azole complexes, [367] and $[FeCl_2(PPh_3)(\underline{N}\text{-sulphinylaniline})]$. [368] A kinetic study of the reaction of histamine with $[Fe(CN)_5(H_2O)]^{3-}$ and the spectroelectrochemistry of the product indicated [369] that histamine coordinates through the azole nitrogen.

5.5. *Pyridine Complexes*

Two studies involve highspin-lowspin transitions. For the tris complex of 2-(pyridin-2-yl) benzothiazole this transition shows thermochromism. [370] The effect of particle size on the equilibrium is studied when $[Fe(NCS)_2(2\text{-pyCH:NHPh})]$ is supported on to C or MgO, with a greater fraction of lowspin as loading increases. [371]

In new preparative work, complexes have been prepared with 2-picolyl-p-nitrophenylketone, [372] bromazepam, [373] 8-hydroxyquinolate-5-(p-tolyl)sulphonamide, [374] bis(antipyryl)piperazine, [375] and pyridoxine. [204]

5.6. *Diimine Complexes*

Resonance Raman spectra of fully deuterated $[Fe(bipy)_3]^{2+}$ allowed the C-H modes to be identified. [376] The same technique, together with surfaceenhanced spectra, studied the absorption of $[Fe(phen)_3]^{2+}$ on Ag and Fe. [377] $[Fe(bipy)_3]^{2+}$ on graphite was used [378] to release H_2 and O_2 from H_2O.

The kinetics of racemisation of $[Fe(phen)_3]^{2+}$ continues to be studied. In the presence of $C_nH_{2n+1}SO_3^-$ stronger hydrophobic interactions coming when n is increased accelerated this reaction and the dissociation of the complex, but the latter reaction is retarded at low sulphonate concentration. [379] These reactions have also been studied [380] for $[Fe(4\text{-}MeC_6H_4N{:}C(Ph)\text{-}2\text{-}py]_3^{2+}$ as well as $[Fe(phen)_3]^{2+}$.

Nucleophilic attack on the tris-complexes is still attracting interest. The kinetics of the reaction of OH^- with $[Fe(4,7\text{-}diphenylphen)_3]^{2+}$ are reported. [381] Another study, using the nucleophiles OH^- and CN^- reached the unsurprising conclusion that this was a very complex reaction witH many possible intermediates. [382] The aquation of $[Fe(phen)_3]^{2+}$ and the Fe^{2+} complex of the hexadentate ligand $(2\text{-}py\text{-}CH{:}NC_2H_4NHCH_2)_2$ in microemulsions shows that in some cases the reacting complex is at the interface. [383] $[Fe(bipy)_3]^{2+}$ is said [384] to exist as a covalent hydrate during its role in the oscillating reaction of BrO_3^- with malonic acid. An equilibrium constant for the association of $[Fe(phen)_3]^{2+}$ with ClO_4^- is reported. [385]

Much work concerns spin transitions in $[Fe(diimine)_2(NCS)_2]$ compounds. DSC experiments [386] showed a first order transition for the bipy complex, but two steps were found, suggesting the spin transition and structure modification do not quite coincide. ^{13}C spin lattice relaxation time studies [387] indicated that there is no spin diffusion in the solid phen complex as the transition occurs. The $4,7\text{-}Me_2phen$ complex exhibits a 45K hysteresis, the exact values depending on the crystallinity of the sample, [388] but there is a much smaller hysteresis for the bis complex of 1,10-phenanthroline-2-carbaldehydephenylhydrazone. [389] For the latter complex there is a first order transition, but not for $[FeL_2(NCS)_2]$, (L =

2,2'-bi-5-methyl-2-thiazoline according to DSC results. [390] Resonance Raman spectra show special enhancement of some C-C vibrations in tris complexes of bipy substituted at the 4 and 4' positions by C_{12} or C_{18} ether chains. [391] New preparations include bis complexes with a new phenyl substituted terpyridyl, [392] a pyridine-phenanthroline tridentate, [393] and a pyridine-thiazole-pyridine tridentate ligand. [394] Studies in solution concern the formation of ternary complexes with polypyridine ligands, [395] a ferrozine (sulphonated bipy) complex, [396] and a ternary bipy-benzohydroxamate complex. [397]

5.7. Schiff Base Complexes

In addition to Schiff base complexes, this section includes semicarbazones, thiosemicarbazones, oximes, hydrazones and other complexes in which a $>C=N-$ unit is significant.

The trigonal bipyramidal complex of the bis-Schiff base of salicylaldehyde with N'-methyldipropylenetriamine has the two Schiff base nitrogens in axial positions. [398] The Fe(II) complex of the pentadentate ligand from diacetylpyridine and two methyl histidinates reacts irreversibly with O_2 in py, probably by ligand oxidation. [399] The tris complex of dimethylpyrimidinetetrone monoxime shows coordination through the oxime N and a neighbouring O. [400] The $[Fe(dmg)_2]$-py(or 4-CN py) system is used [401] to illustrate a spectrophotometric method for studying two successive equilibria. The product of the reaction of $FeCl_2.4H_2O$ with cyclohexanedioxime and $BF_3.2HO_2CMe$ is an oxime complex in which two ligands are linked by two borates in a manner already known for other metals. [402] The most interesting of many semicarbazone complexes is one with one anionic and one bidentate NO_3^-. [403] A Schiff base from 1,3-diaminobenzene may form a binuclear Fe(II) complex. [404] Other formation constant [405] and preparative work [90,96,406-416] requires no further comment.

5.8. Complexes of Phosphorus Ligands

$[Fe(acac)_3]$ reacts with dmpe and $AlMe_3$ to give $[Fe(dmpe)(acac)]^+$ $[AlMe_4]^-$; the BPh_4 salt can be converted to $[Fe(dmpe)Me_2]$ with LiMe. [417] $[Fe(PMe)_4MeBr]$ reacts with $LiCH_2PMe_2$ to give $[Fe(PMe_3)_3XPMe_2CH_2]$, (X = H, Me), but $LiCH(PMe_2)_2$ gives $[Fe(PMe_3)_3H(Me_2PCHPMe_2)]$ which reductively eliminates to $[Fe(PMe_3)_3(dmpm)]$[418].

5.9. Oxides

The rotational spectrum of the FeO molecule has been observed and assigned. [419] Also for FeO(g), the laser excitation and rotationally resolved laser induced fluorescence spectra have been measured, apparently with unusual results.[420]

Studies of solid oxides include the magnetism [421] of Fe_xO, (x = 0.929 - 0.950), the determination [422] of ΔG of formation of Fe_2SnO_4, studies of the $FeO\text{-}V_2O_3$ - other oxide systems, [423,424] and the discovery [425] that the rutile structured semiconductor $FeVNbO_6$ becomes a "spin glass" below 20K.

5.10. Hydroxide and Hydroxo-Complexes

The decomposition of $Fe(OH)_2$ to Fe_3O_4, H_2O and H_2 has been studied [426] by Moessbauer spectra. The hydrolysis of Fe^{2+} in aqueous NaCl gave formation constants [427] for $[Fe(OH)]^+$ and $[Fe_2(OH)_2]^{2+}$.

5.11. Complexes of OR^- Ligands

Polarography studies on $[FeX_3]^-$ or $[Fe(phen)X_2]$, (X = 2-nitrosophenols) show the ligands are reduced before Fe.[428] A complex of lapachol (HX), a quinone, $[Fe(H_2O)_2X_2]$, has been prepared. [429]

5.12. Carboxylates, Aminoacids and Related Complexes

Crystals of $[FeX(O_2CR)]$, (X = Cl, Br, I; R = Ph, $PhCH_2$, PhCH:CH) have been prepared [430] by the new but simple reaction of Fe with RCO_2H and organohalides. Formation constants have been determined [431] for complexes of Fe(II) with 5-hydroxy-8-quinolinecarboxylic acid.

Two groups have studied the pyrolysis of FeC_2O_4 and its dihydrate in differing conditions and have identified the various products. [432, 433] Equilibrium constants for the system FeC_2O_4 - N_2H_4 - H_2O/EtOH have been determined. [434] New aminoacid compounds prepared are [Fe(L - β - phenylalaninate)$_2$] [435] and [Fe(glutamate)(lysinate)]. [436] An Fe(II) complex with benzylpenicillin is believed to show coordination through a carboxylate O and a thiazolidine N. [437] The oxidation [438] of aminoacids by Fenton's reagent is first order in Fe^{2+}.

An interesting dinitrogen complex $[Fe(edta)N_2]^{2-}$ has been prepared, together with its cdta analogue; [439] pyrolysis studies are also reported. [440] The reactions of $[Fe(edta)]^{2-}$ with SO_3^{2-} and NO have been studied with special reference to the N and S compounds which are formed. [441, 442] The Fe(II) complex of $C_3H_7NHCOCH_2N(CH_2CO_2H)C_2H_4N(CH_2CO_2H)_2$ has been synthesized; it cleaves DNA if O_2 is present, especially in the presence of reducing agents like Na ascorbate, possibly by an OH radical attacking the deoxyribose ring. [443]

5.13. Diketonates

$[Fe(N_2H_4)_2(tfacac)_2]$ has been prepared. [444] $[Fe(hfac)_2]$ forms 1:1 complexes with bidentate or tetradentate N ligands and with [M(acen)] and [M(salen)], (M = Co, Ni, Cu). [445]

Formation constants have been determined [224] for the isonitrosoacetylacetone (HX) complexes $[FeX_2]_2$ and $[FeX_3]^-$. Fe(II) complexes are included among many prepared by arnebin-I [231] and 1-hydroxy-9-fluorenone. [446]

5.14. Compounds of Oxyanions

Studies on $FeSO_4$ predominate. When the heptahydrate is heated with M_2CO_3 the Li system is unique in not reacting until all the water is lost, [447] but for $Be_2(OH)_2CO_3$ there is no mutual reaction. [448] $FeSO_4$ is implicated in many organic reactions: the dimerisation of amines by H_2O_2, [449] the ring cleavage of oxaziridines, [450] the oxidation of alkenes to vicinal diacetates by $S_2O_8^{2-}$/$MeCO_2H$, [451] the dealkylation at the

6-position of 2,6-di-t-butyl-4-methylphenol, [452] and the promotion of graphite oxidation. [453]

The chlorosulphates $[Fe(SO_3Cl)_2]$, $[FeL_4(SO_3Cl)_2]$ and $[FeL_2'(SO_3Cl)_2]$, (L = monodentate ligand, L' = bidentate ligand), have been prepared, [454] as have $[FeL_4(MeSO_3)_2]$, (L = py, ½ bipy), [455] and $[Fe(O_2SeC_6H_4R)_2]$, (R = H,Me,Cl,Br). [456]

$[Fe_9(PO_4)O_8]$ has been prepared and shown to have a structure comprising FeO blocks of NaCl structure and $FePO_6$ blocks which are alternating rows of FeO_4 and PO_4 tetrahedra; the loss of electron to give the mixed valence formulation occurs in the FeO region. [457] Thermodynamic data were obtained [458] for the formation of this from $Fe_3(PO_4)O_3$ and its reduction to $Fe_3(PO_4)_2$.

$[Fe(H_2O)_2$(uracil [illegible] is believed [459] to show coordination through N(3) and the keto O at position 2. Complexes with 2'-AMP and 3'-AMP are believed to show phosphate coordination, perhaps with adenine coordination also in the 2'-isomer. [250]

Fe(II) complexes with substituted hydroxamic acids have been prepared, [460] and formation kinetics studied for the 1:1 acetohydroxamic acid complex. [461]

Complexes of $[ONC(CN)C(O)NH_2]^-$ have been prepared in which coordination is probably through the amide O and nitroso N. [462]

5.15. Water Complexes

Moessbauer spectra of various $[Fe(H_2O)_6]^{2+}$ materials are all, unsurprisingly, explained by the same crystal field parameters; [463] the phase-change in the SiF_6^{2-} salt is of the order-disorder type. [464]

There is an S_N1 rate-determining step in the solid state reaction of $FeSO_4.7H_2O$ with KNCS to give $[Fe(H_2O)_4(NCS)_2]$. [465] The mechanism of the photooxidation of aqueous solutions of Fe^{2+} salts has been studied, [466] as has the air oxidation of these solutions in the presence of Cu^{2+} ions. [467]

5.16. Complexes of Neutral Oxygen Ligands

A wide range of complexes of dmf and its thio analogue have been prepared. [468] Heat capacity data on $[Fe_xCo_{(1-x)}(C_5H_5NO)_6](ClO_4)_2$ showed no coupling between Fe and Co; crystal field anisotropies appeared much greater than exchange effects. [469] The 2:1 complex of py-4CHO with $FeCl_2$ is thought, implausibly, to show O coordination. [50] Other new complexes are $[Fe_2Cl_4X]^{4-}$, (X = tetrasuccinylurea), [470] and $[Fe\{PhS(O)C_2H_4S(O)Ph\}_3]^{2+}$, also O bonded. [471]

The use of trioctylphosphine oxide and other organophosphorus compounds in the solvent extraction of Fe^{2+} from acetate eutectics into $C_{12}H_{26}$ is described. [472]

5.17. Sulphides and Sulphido-Complexes

Studies on the material commonly bearing the formula FeS include its electrochemical reduction in propylenecarbonate, [473] its use as an electrode material in button-sized batteries with Li, [474] and its preparation from FeS_2 reacting with SO_2, N_2 and S. [475] For both FeS and FeS_2 there are reports on the K-absorption edge structure [476] and solubility in LiCl-KCl eutectics. [477] Various spectra of FeS_2 have been recorded, including the reflectance spectrum, [478] the PE, electronic and Moessbauer (with MO calculations), [479] and a more complete Raman spectrum, in which mixing of S-Fe-S stretches and lattice bands showed one ought not to think of this as a molecular crystal, which we probably would not have done anyway. [480] A photoelectrochemical study of FeS_2 showed that SO_4^{2-} was formed. [481] Ferrimagnetic Fe_7S_8 is formed [482] from hydrated iron (III) sulphate, H_2 and H_2S.

Among mixed metal sulphides there have been studies [483] on the pressure effect on T_c for $FeCr_2S_4$, magnetization studies [484] on $FeSb_2S_4$ and on [485] $Fe_xZn_{(1-x)}Cr_2S_4$. $X\alpha$ calculations on $[FeS_4]^{n-}$ (n = 4-6) were used to interpret bond lengths and magnetochemical and Moessbauer data. [486] The new cluster complex $[Fe_8S_6I_8]^{3-}$ is formed by the reaction of Fe, I_2, Et_4NI and $[Fe_6S_6I_6]^{2-}$ in CH_2Cl_2; its structure comprises an Fe_8 cube with an I on each Fe and an S atom over each cube face. [487] $[Fe(WS_4)_2]^{3-}$ has been prepared, with the same structure as its Mo analogue, two bidentate WS_4 ligands coordinating tetrahedral Fe; physical data suggest the extra negative charge over the 2-anion is held on a S atom, not a metal.[488] An X-ray structure for $(Ph_4P)_2$ $[FeCl_2(MoOS_3)]$. MeCN shows bridging by two S atoms. [489] Work on Fe-S compounds of avowed biochemical relevance is described in section 6.

5.18. Complexes of Other Sulphur Ligands

The complexes $[X_2Fe(S\ ^tBu)_2Mo(CNR)_4]$, (X = Cl, Br; R = C_6H_{11}, ^tBu) have been prepared; their electronic spectra vary with temperature according to whether syn or anti conformers are present. [490] Ethyl N-phenyldithiocarbamate coordinates Fe^{2+} by its thione S. [491] A cis configuration is adopted by $[Fe(CO)_2(dithioacetylacetonate)_2]$. [492] All three potential donor atoms coordinate Fe^{2+} in its bis complex with 1, 5-dithiacyclooctan-3-ol. [493] Complexes of tetradentate thio-enol-hydrazine ligands have been prepared. [494] The reaction of $MoCl_5$, $FeCl_2.4H_2O$, BzSH and Et_2NCS_2H gives [495] $[Mo(Et_2dtc)_4]^+$ $[FeCl_3(BzSSBz)]^-$.

5.19. Fluorides and Fluoro-Complexes

The far IR spectrum of FeF_2 was used to study its antiferromagnetism. [496] Its Raman scattering has also been recorded. [497] Coherent anti-Stokes Raman spectra in $Fe_x\ M_{(1-x)}\ F_2$, (M = Mn, Zn) revealed electronic bands in the $^5T_{2g}$ state. [498] Ordering transitions in $K_2\ Fe_x\ Co_{(1-x)}\ F_4$ were studied by elastic neutron scattering. [499] Jahn-Teller effects in $[FeF_6]^{4-}$ were found [500] in EPR and IR spectra by doping in $KZnF_3$. Powder neutron diffraction in $FeZrF_6$ is recorded. [501]

5.20. Other Halides and Halo-Complexes

$FeCl_2$, with Fe and Fe_3O_4, is found by Moessbauer, EPR and SEM studies of graphite. [502] Mass spectra gave [503] thermochemical, thermodynamic and volatility data for FeI_2, Fe_2I_4 and Fe_2I_2. Laser properties in the visible region have been found [504] when FeI_2 is photodissociated at 193 nm above 720K. PE spectra of FeI_2 led to calculation of the energetics of the Fe(II) I^- system, including gaseous and ionic species. [505]

Among mixed metal halides there have been neutron scattering [506] and magnetization [507] studies on the 3-D Ising system $Fe_xMg_{(1-x)}Cl_2$. The magnetic phase diagram of $Fe_x Mn_{(1-x)}Cl_2.2H_2O$ shows remarkable complications, with two antiferromagnets, ferromagnetism, intra- and interchain interactions and spin-glass regions. [508] Moessbauer spectra are recorded for $Fe_x M_{(1-x)}Cl_2.yH_2O$, (M = Mn,Co,Ni; y = 4,6) [509] and for $CsFe_xM_{(1-x)}Cl_3.2H_2O$. [510] Below 0.7 times T_N $RbFeCl_3.2H_2O$ is a pure one-dimensional Ising antiferromagnet; its spin-cluster resonance intensity was studied. [511]

New preparations of halo complexes include a phenothiazine salt [512] of $[FeCl_3]^-$, and the group $[Fe(MeCN)_6][FeI_4]$, $[Fe(MeCN)_6](I_3)_2$ and $[Fe(thf)_6][Fe(thf)I_3]_2$. [317] Phase changes in $(MeNH_3)_2[FeCl_4]$ were studied by Brillouin scattering. [513] The salts $(RC_6H_4NH_3)_2[FeCl_4]$, (R = H, Cl, OMe) are weakly ferromagnetic below their Neel temperatures of 80-90K as canting of the antiferromagnetic spins occurs. [514] For the salt with R = Br and for $[3\text{-}ClC_6H_4NH_3][FeBr_2Cl_2]$ the transitions to ferromagnetism occur at 94 and 98K respectively and short range two-dimensional antiferromagnetism sets in at 140 and 190K. [515] In other work on $[RNH_3]_2[FeCl_4]$ salts heat capacity measurements were used to investigate phase transitions. [516]

The free energy of formation of $[Fe(H_2O)_4Cl_2]$ has been calculated [517] as -1280 kJ mol^{-1}. The enthalpy of solution in methanol has also been measured. [518] X-ray diffraction on concentrated aqueous solutions of $FeBr_2$ revealed the presence of $[FeBr(H_2O)_5]^+$. Solvates of Fe(II) halides and perchlorate with dmf and its thio analogue were assessed for charge distributions. [520]

The reaction of $FeCl_3$ *in thf with* $[Re(cp)_2H]$ *gave* $[Fe_4Cl_8(thf)_6]$, with 4 doubly and 2 triply bridging Cl. [521] With $FeCl_2$ or $FeBr_2$ this reagent gives $[Fe(cp)_2H.2FeCl_2]$ or $[Fe(cp)_2H.FeBr_2]$, whose structures are not known. [522]

5.21. Hydrides and Hydrido Complexes

Moessbauer spectra showed the existence up to *70K* of Fe*H in an* H_2 matrix. [523] At 12K in an H_2 or rare gas matrix FeH_2 can be formed. [524] An ion beam study of the reaction of Fe^+ with H_2 or D_2 led to a study of $[FeH]^+$, including its dissociation energy and Lewis acid reactions with organooxygen compounds. [525]

Mg and Fe react together with H_2 to give Mg_2FeH_6, in which D substitution helped physical studies to show the presence of $[FeH_6]^{4-}$ in a K_2PtCl_6 structure. [526]

5.22. Other Iron (II) Compound

Doping Fe^{2+} into InP allowed the study of photoluminescence in the $^5T_{2g}$ → ^5E transition. [527]

5.23. Mixed Valence Compounds

Pyrazine (pz) has been associated with two reports which describe Fe(II) and *mixed valence compounds. The compounds* $[Fe(acac)_2L]_n$, *(L = pz,* 4,4'-bipy or derivatives) react with I_2 to give partial oxidation; the more conjugated the bridging ligand is the higher the solid state conductance. [528] In $[(NC)_5FepzFe(CN)_5]^{5,6-}$ there are localized Fe(II) or Fe(III) valencies; in $[(NC)_5FepzRu(NH_3)_5]^{0,1-}$ Fe is in oxidation state II in both complexes. [529]

6. BIOLOGICAL IRON COMPOUNDS

This section includes iron proteins and other well-defined entities which contain iron and occur in living systems. It also includes surveys of work on model compounds. Work on natural systems which does not bear on the role of iron is excluded.

6.1. Haem Proteins

The many spectroscopic studies on deoxy haems will be discussed in order of increasing energy. ^{1}H NMR spectra of carp and adult human Hb (and its oxy and carbonyl derivatives) showed differences in the tertiary and quaternary structures. [530] Such spectra have also been used to probe the electronic structure of Fe and haem-protein interactions. [531] A clear and thorough review of EPR work on Hb and Mb includes derivatives in all oxidation states, treatment of spin states and compounds with other metals; there are very clear diagrams and a useful analysis of the bonding question. [532]

The use of isotopic substitution of Fe and N in Mb led to the assignment of the 243 cm^{-1} band in its resonance Raman (rR) spectrum as a "pyrrole tilting" mode in histidine and the 220 cm^{-1} band as an Fe-N stretch mixed with histidine and/or porphyrin vibrations; [533] this is supported by another group who emphasise the strong coupling of the latter band to a Soret band, [534] but others find the Fe-N stretch ranges over 40 cm^{-1} in response to protein effects, though six-coordination renders the structure more rigid. [535] The pH dependence of the rR spectrum of Hb can be accounted for by haem-protein interactions during protonation [536]; a similar study was made for Mb and cyano-Mb. [537] Yet another report showed clear differences between the O_2 carrying and peroxidase proteins both in protein interactions affecting side chain conformations and in the axial histidines. [538] There are similarities in the rR spectra of a highspin haem protein first reported in 1983 when isolated from <u>Chromatium vinosum</u> with that of Hb, but it is thought that the new material may use vinyl groups for the haem-protein link. [539]

The Moessbauer spectrum of Mb shows less conformational variety than Hb, which shows less still than carbonyl Hb; this is related to cooperativity effects. [540]

In other physical studies, magnetic susceptibility studies down to 1.7K on Mb and photodissociated carbonyl Mb used carbonyl Mb for diamagnetic correction and found both compounds under study had S = 2 and g = 2, with the only difference being in the zero field splitting. [541]

Many physical studies have investigated the R and T states of haem proteins. Kinetic and thermodynamic data for the noncooperative reactions of dimeric Mb from Busycon canaliculatum suggest that a protein may be in an R state as regards one ligand, but this might be a T state for another. [542] A mathematical treatment of the T - R transition claims to produce results in agreement with earlier experiments. [543] The effect of sucrose, glycerol or ethylene glycol on the T - R transition correlates with slower rates in more viscous media, using CO rebinding experiments from laser photolysed carbonyl-Hb. [544] Studying the oxygenation in aqueous alcohols with changes in pH and KCl concentration showed that the energy difference between the T and R states depended on the polarity of the solvent as well as protein-solvent interactions. [545]

A most interesting crystallography result showed an Hb tetramer with the α units oxygenated but not the β ones; the authors claimed that in living systems Hb might gain or lose O_2 from only the β haems. [546] However the work was criticised on the grounds that the material studied may have been an equal mixture of oxygenated and deoxygenated Hb. [547] In another lengthy theoretical paper minimal energy structures for R and T states were calculated. [548a] Other workers emphasise the effect of quaternary structural changes. [548b] In sea cucumbers deoxy-Hb is tetrameric but it becomes dimeric on oxygenation. [549]

The rR spectra of some oxy Hb vibrations shows complex pH dependant excitation profiles which show the haem distorting from D_{4h} symmetry. [550]

Redetermination of the magnetic susceptibilities of aqueous solutions of oxy-Hb and carbonyl-Hb found that both were diamagnetic; values greater than zero which are sometimes reported might arise from the presence of some deoxy-Hb. [551]

A kinetic study of O_2 and CO binding by the dimeric Hb from the mollusc

Scapharca inaequivalis shows that this has cooperativity and leads to the assertion that one does not need to have different types of chain to induce cooperativity, as had previously been supposed. [552] Quantum yields for the photodissociation of oxy-Hb suggest there are two distinct phases in the O_2 binding process, [553] while another photochemical study of this reaction using either α or β haems in which Fe was replaced by Co showed that O_2 was more easily lost from β haems. [554] Aniline reacts with haems during oxygenation by π - π interaction with a pyrrole ring. [555]

The ΔE_q values obtained from Moessbauer spectra of haem complexes of polyvinylimidazoles and their CO and O_2 adducts were used to assess steric effects. [556] Removal of methyl or vinyl groups from haems unsurprisingly affects CO and O_2 binding through size and conformation changes. [557]

The most striking result from much work on carbonyl, alias carboxy, haems comes from rR spectra of carbonyl-Hb and cyanomet-Hb from Chrinomus thummi thummi, which indicate that earlier crystallographic results on these must be mistaken and the Fe-C bonds are less than 2Å long and the FeCO angle is 8° greater than supposed; the new values are more in line with those from human Hb. [558] There has been a flash photolysis study [559] of CO binding to chains of Hb - Zurich at 20-300K and a theoretical kinetic study of the rebinding of small ligands like CO to haem proteins after flash photolysis. [560] Variations in the reactivity of various myoglobins to CO is attributed to differences in the $Fe-N_4$ plane distance as well as in base dissociation. [561] Time resolved X-ray absorption spectra were used to follow the recombination of carbonyl-Mb after laser photolysis to test the technique of using synchotron sources for such experiments. [562] A kinetic study of the oxidation of carbonyl-Hb by $[Fe(CN)_6]^{3-}$ including the effects of D_2O and inositol hexaphosphate showed the reaction was a 3 stage process but that deoxy-Hb was not an intermediate. [563] $[Mn_2Fe_2]$ hybrid haemoglobins react with CO only at Fe; placing Mn in either the α or the β haems and pH variation allowed development of a two state model for cooperativity. [564]

Other small molecules interact with haems. EPR was used to follow the reaction of NO with oxy-Hb and establish that 3 NO molecules bind by 200 min but the remaining β haem is not bound until much later. [565] Exciting the Soret bands of nitrosyl haems (and horseradish peroxidase) using ^{15}N and ^{18}O allowed location of the Fe-NO stretching (600 cm^{-1}), and Fe-NO bending (574 cm^{-1}) vibrations in the rR spectra, but a band at 554 cm^{-1} previously thought to be a stretch is now thought to be a bend, which casts doubt on the assignment of a 567 cm^{-1} band in oxyhaems as an Fe-O_2 stretch; the clear differences between nitrosyl haem and carbonyl haem spectra are attributed to stronger binding by NO, probably using the π* orbital. [566] Applying pressure of up to 1400 atm on aqueous solutions of nitrosyl -Hb and other derivatives produced changes in the ^{1}H NMR spectra which were attributed to quaternary effects rather than R - T state transitions. [567] Applying γ-radiation to solutions of oxy-Mb saturated with N_2O yields deoxy, met and ferryl proteins. [568] The equilibrium constants for the binding of $MeCO_2PbEt_3$ to Hb were redetermined and show that 2 molecules coordinate, not 3 as previously reported. [569] The oxidising agent $[FeO_4]^{2-}$ binds Hb and other enzymes at phosphate sites, but then oxidises them powerfully; this reaction is prevented by adding 2,3-diphosphoglycerate, in line with this thinking. [570] There has also been a kinetic study of the oxidation of Hb by diazonium salts. [571] EPR and ^{13}C NMR spectra were used to follow the oxidation of nitroxide by Hb with H_2O_2. [572] O_2 bound to Hb, rather than free Hb, promotes the autocatalysis of the NO_2^- - Hb reaction. [573]

Some miscellaneous results. Reconstitution of Mb using Fe(II) chlorins allowed the formation of O_2 complexes, but not with good reversibility. [574] A chlorin ring system was found as a tetracarboxylate as the haem d, residue in nitrate reductase. [575]

Turning to met-Hb and related species, the photochemical formation from oxy-Hb has been further studied. [576] Reagents for effecting this oxidation include $[Fe(bipy)_3]^{3+}$, for which the effect of such added ligands as citrate was studied; [577] ferricytochrome b_5 also performs this oxidation, which in this case is inhibited by CO, which leads the authors to believe the oxidation occurs after loss of O_2. [578] An X-ray study to 1.9Å resolution of met-Mb after saturation with 7 atm of Xe shows 4 sites for these in cavities and suggests molecules entering these cavities may induce conformational change. [579] The heat capacity and dielectric constant of met-Mb crystals below 30K have been determined. [580] There has been a magnetization study [581] of met-Hb and met-Mb from 2 to 0.1K.

The greatest weight of papers on met-haems concerns intricacies in NMR spectra (^1H in all cases). Changes in pH for solutions of met-Hb, cyano-Hb and deoxy-Hb change the hydrogen bonding of the histidines and induce conformational changes; Hb seems to have a more flexible pocket than Mb. [582] Similar work involved tryptophan residues also and included carbonyl-Mb. [583] A further assignment of histidine protons in cyano-Mb and some derivatives with Et, Br or O_2CMe ring substituents is presented. [584] The met-Mb of Indian elephants has distal glutamine, not histidine, and for this it is suggested from NMR that either amine N or carbonyl O can be used for hydrogen bonding to water; [585] a later paper from this group gave more assignments. [586] Other pH variation work on cyano-Mb and deoxy-Mb suggests that at pH 5-6 there is a structural modification of the haem pocket related to π-interactions between a histidine and the haem ring. [587] Also for cyano-Mb, NOE and relaxation rate experiments centred on the haem pocket isoleucine indicate that the magnetic anisotropy of the Fe is rhombic. [588] For azido-Hb the NMR spectra showed a spin equilibrium between $S = 1/2$ and $S = 5/2$ states. [589]

EPR is also applied to haem derivatives, as in the use of spin traps to follow the photoreduction of chlorohaemin; H and C_2H_4OH radicals form in EtOH, but no radicals are formed if py is absent, suggesting py stabilises Fe(II) here. [590]

There are also many studies of electronic spectra of met-haems. Temperature

variation of these confirms fluoro-Mb as highspin, cyano-Mb as lowspin, and a mixture of each for the N_3^-, OH^- and imidazole derivatives. [591]
Electronic spin-echo envelope spectroscopy confirms earlier experiments in showing that H_2O is directly bonded to Fe in met-Mb but not in met-Hb. [592]
Comparison of the crystal and solution spectra of oxy-Hb, cyano-Hb or azido-Hb using photoacoustic difference spectra showed no differences, but there were for fluoro-Hb and aquomet-Hb, for which the strong hydrogen bonding proclivities of the axial ligands were thought responsible for minor structural changes on crystallisation. [593] Electronic, CD and MCD spectra of mixed valence haemoglobins with specific association of oxidation states with the α or β haems showed the Soret MCD was sensitive to the spin state of Fe(III) and the CD were not the sum of the component units and must therefore reflect the interaction of haem units. [594]
Electron transfer to met-Hb by cytochrome b_5 was followed kinetically by MCD spectra, with identification of a hydroxy-Hb complex. [595] Another kinetic study followed the aquomet-Hb reaction with Tb complexes. [596]
The electroreduction of the ferrihaem dimer by cyclic voltammetry involves the formation of Fe(II) monomers. [597] A polymer containing haemin acts as a CN^- ion exchanger. [598] The α and β haems of Hb were separated and fitted to Fe(III) sulphopc; the α globin forms a monomer and the β a tetramer with a new CD spectrum. [599]

6.2. Peroxidases

The variation of the ^1H NMR spectrum of horseradish peroxidase (HRP) with pH using selective deuteration to assist assignments showed that at high pH the deprotonated distal histidine coordinates Fe with its plane perpendicular to the proximal histidine. [600] The rR spectrum of HRP supports this, showing highspin 5-coordinate Fe at neutral pH and lowspin 6-coordination at higher pH; in these alkaline forms bands were found which innocents might confuse with the "marker" bands of Fe(IV). [601] Nevertheless a short communication on X-ray absorption edge and EXAFS for HRP and related compounds reports HRP-I as being an Fe(IV) - porphyrin π complex. [602] CD, MCD and EPR spectra of the haem protein indoleamine -2,3-dioxygenase show similarities to HRP and it is concluded histidine coordinates the fifth coordination site and the sixth position is hindered; when the substrate L-tryptophan binds the EPR and MCD spectra are those for lowspin Fe(III), so that some conformational change has occurred. [603]

Hartree-Fock-Slater molecular orbitals were used to show how the bond strength of CO varies when it is coordinated to haem proteins with explanations of IR data in terms of redox potentials of proteins, with peroxidases being able to form better hydrogen bonds to CO than other proteins. [604] Kinetic data for the rebinding of CO to cytochrome c peroxidase and HRP after flash photolysis show similar rates which are different for that with Mb. [605] Another kinetic study, of the reaction of CN^- with Fe(III) chloroperoxidase at various pH and in the presence of NO_3^-, claims to show that this anion bonds to the protonated enzyme and CN^- to the unprotonated form. [606]

There has also been a rate study for the decomposition of O_2^{2-} by Fe(II) in L- α-phosphatidylcholine. [607]

Investigation of a model complex led to the conclusion that HRP works by an outer sphere mechanism but cytochrome P450 operates by substrate formation. [608]

6.3. Cytochromes

Sorting out this big topic is made difficult by the non-systematic nomenclature. Many papers concerned with toxicology or other aspects of cytochromes (cyt) in which serious Fe chemistry does not appear are not cited. Two general papers which require recording concern the use of ^1H NMR of haems to reveal the electronic structure of Fe and the haem protein interactions in, inter alia cyt c and cyt c oxidase, [531] and the discovery of a cyt reduction type mechanism in the simultaneous electron transfer to Fe and proton transfer to alkoxide in the reduction of a haemin-bispilocarpate complex. [609] Many papers discuss the properties and reactivity of cyt c in its many manifestations. There has been a more extensive assignment of the haem and histidine ^{1}H resonances for ferricyt c. [610] NOE techniques were used to probe the methionine-haem interactions in ferrocyt c, finding 4 conformers with the aid of computer graphics and concluding the solid and solution structures were similar. [611] The ^{1}H spectra of the oxidised and reduced forms of cyt c-550 from *Paracoccus denitrificans* have also been assigned, with successive changes with increasing pH attributed to loss of coordinated histidine, of propionate, the displacement of methionine by lysine and the loss of lysine. [612] In an NMR study of a dihaem cyt c-552 from *Pseudomonas perfectomarinus* in the oxidised, reduced and intermediate forms no coordination by methionine was found. [613] The combined use of CD and ^{1}H (NOE) NMR spectra for cyt c-553 from *Desulfovibrio vulgaris* and *D. desulfuricans* showed that coordinated sulphur showed S chirality in the reduced form and R chirality when oxidised. [614] The observation of non-Curie law behaviour for the ^{1}H NMR spectrum of oxidised cyt c-554 from *Alcaligenes faecalis* could not be explained; this cytochrome has the fastest self-exchange rate yet found. [615] For cyt c-555, which has an unusually low redox potential, there are large ring shifts for the methyls in the oxidised form; many other lines in the ^{1}H spectrum are assigned. [616]

The EPR spectrum of a cyt in ubiquinolcytochrome c reductase (complex III) showed three signals, showing a 1:1:1 mixture of cyt b-566, cyt b-562 and cyt c_1, the two former having two histidine ligands and the latter one histidine and one methionine. [617]

The observation of both Stokes and Anti-Stokes lines in the rR spectrum of cyt c is the first time for a molecular system, so the paper includes an extensive theoretical treatment. [618] The excitation profiles for the Soret b and rR spectrum of reduced cyt c are also discussed. [619] Such a spectrum for cyt c-554 gave the allegedly unusual result that the oxidised form appears 5-coordinate. [620]

It is suggested that the CT spectrum of oxidised cyt c is a good measure of its ease of reduction, and that the axial ligands are important because each process involves electron transfer to a d_{xy} orbital. [621] Electronic spectra and differential pulse and cyclic voltammetries for cyt c-551 at various pH showed the loss of methionine. [622]

The reactions of cyt c have attracted many studies, including a short review. [623] The reaction of O_2 with cyt c and cyt c oxidase is faster for the former in low ionic strength buffers but faster for the latter with high ionic strength buffers. [624] Electron transfer reactions with $[Fe(CN)_5X]^{n-}$, (X = aminoacid) are second order processes with two binding sites. [625] Reduction by $[Fe(CN)_6]^{4-}$, followed by ^1H NMR, is faster in cyt c from Candida krusli than that from a horse. [626] The reduced and oxidised forms give comparably strong complexes with $[Co(CN)_6]^{3-}$, according to ^{59}Co NMR. [627] There have also been kinetic studies of reactions with Ru and Os bipy and phen complexes, [628] and reductions of ferricyt c by $[Ru(NH_3)_5L]^{2+}$, (L = N donor). [629]

The use of RuO_2 or IrO_2 or other electrodes in CV studies of cyt c showed the significance of surface charge on the electrodes. [630,631] Adding Cu or Fe compounds to cyt c enhances the rate of reduction by thioglycolic acid, probably through the formation of a negatively charged metal-thioglycolate complex; adding phen to this brew assists catalysis by Cu, destroys that by Fe and induces pH dependence. [632] Oxidation of cyt c by oxidised azurin or plastocyanin is not a simple process. [633] The reduction of cyt c by ascorbate proceeds by electron tunnelling over ~ 16Å below 26°C but at higher temperatures there is complex formation before reduction occurs. [634] The reduction of cyt c in the presence of 6-hydroxydopamine and O_2 is an oscillating system; this was then used to induce amusing oscillations in reactions of catalase, superoxide dismutase, mannitol and desferrioxamine. [635] Cyt c does not form a strong complex with cyt c_1, so the earlier suggestion that such a complex was formed in the reaction between them is discredited. [636] There have also been kinetic studies of the reactions of cyt c with cyt oxidase [637] and bacterial reaction centres. [638]. Now for cyt c_3. This has been identified, along with some 4Fe-4S ferredoxins, in electron transport proteins in Desulfovibrio desulfuricans. [639] A most useful X-ray structural analysis (R = 0.176) of cyt c_3 from Desulfovibrio vulgaris found the 4 haems to be closely packed with adjacent haem planes perpendicular; histidine acts as axial ligands and all the Fe-N (hist) distances are slightly different in the range 1.88-2.12Å. The haems are exposed to solvent, are non-equivalent and up to 0.37Å distorted from planarity. [640] Fuller assignment of the ^{1}H NMR spectrum of cyt c_3 has been obtained by 2D methods; the paper includes an analysis of the method. [641] Studying the 'H spectrum at various pH and redox potential shows electron exchange between the haems is faster than 10^5 s^{-1}; the midpoint redox potentials of some haems are pH dependent and it was concluded that the protein has to operate at different redox potentials. [642] EPR was used to follow the dithionite reduction of orthorhombic lowspin Fe(III) in oxidised cyt c_3 to lowspin Fe(II) and its restoration on oxygenation; it was concluded that the oxidised form took on a tetrahedral distortion after many redox cycles. [643] In another EPR study 2 haems showed high and 2 showed low redox potentials; reaction with colloidal sulphur was believed to involve an exposed low potential haem. [644] Another kinetic study of the cyt c_3 reduction was followed by electronic and CD spectra and stopped flow methods; there was special interest in the fast third step which may involve unusual interactions of some kind. [645] Cyt c_3 is one of many systems whose flavin reduction gave correlation of rate with redox potential and the redox centre's accessibility to solvents. [646] Other workers have also compared the redox properties of various cyt c_3 species with the exposure of the haems. [647] An experiment in which the rR spectra of cyt c_4 single crystals were measured is suggested as a prototype for assessing whether an X-ray crystal structure is being carried out on the reduced or oxidised form of a protein. [648] A cyt c_6 from a red alga is thought to contain a c type haem bound to two cysteine residues. [649] The magnetic properties of cyt c' show this has a mixed spin state. [650] Cyt c' species react with EtNC with a wide range of equilibrium constants which do not correlate with CO reactivity and are attributed to steric effects, showing the distal site to be more accessible than had hitherto been supposed. [651]

The next cytochrome system to be reviewed is cytochrome oxidase and its variants. A new cyt c peroxidase has been isolated from Pseudomonas stutzeri and has two interacting haems with distinct redox potentials; the fully oxidised form contains two lowspin Fe(III), one coordinated by 2 histidines and one by a histidine and a methionine, both being lowspin at low temperatures but one showing a highspin - lowspin transition. The half-reduced state has highspin Fe(III) and lowspin Fe(II); the fully reduced form has one highspin Fe(II) and one lowspin. [652] An extensive EPR study showed that the signal at g = 12 coming from an alternative resting state corresponded to a spin greater than 1, that the signal at g = 5 from the pulsed state had an excited intiger spin 7cm^{-1} above the EPR - silent ground state and that the Cu_A site was the same in the resting, pulsed and anaerobically oxidised states. [653]

A pulsed EPR study revealed similar spin-lattice relaxation times for haem a and Cu_A, indicating either they had similar environments or that they were near and relaxed one another; the relaxation of nitrosylferrocyt a_3 in mixed valence oxidase showed enhancement by a nearby paramagnetic centre which is probably haem a. [654]

Raman spectral bands at 210-220 cm^{-1} for Fe-N(hist) were found in the highspin a_3 haem of aa_3 cyt oxidases, but the bands are lost when the pH is raised or CN^- is complexed. [655] The similarity of the MCD spectrum of cyt a in cyt c oxidase with that of a haem a bisimidazole complex shows this structure probably occurs in the enzyme. [656] The X-ray absorption of the "pulsed" (oxygenated) oxidase shows that the S bridge between Fe and Cu is lost, probably being replaced by H_2O or O_2^{2-}; the haem in this form is like ferric peroxidases and if CN^- is present there is a resemblance to cyanohaems. [657]

There have been many reactivity studies in cyt oxidase systems. Four stages were found in the reaction of O_2 with the half reduced form, with first O_2 binding to Fe; this then gains an electron in a distinct process. [658] Another kinetic study of this reaction showed that O-O bond breaking is associated with change in the cyt a_3 - Cu cluster. [659] When CO complexes cyta$_3$ the Fe-C-O unit is linear but not at 90° to the haem and there is strain on the proximal histidine. [660] For the complex with N_3^- and NO a $\Delta m_s = 2$ transition occurs through the interaction of electrons on cyt a(NO) and Cu_B; at 77K the photolysed complex shows cyt a_3-N_3-Cu structure but in the triplet it is thought NO bridges and N_3 is on Cu. [661] Half-reduced cyt c peroxidase easily forms complexes with F^-, CN^- and N_3^- at the low-potential haem, but only F^- induces highspin; the resting enzyme does not form these complexes. [662] Another report confirms the existence of weak catalase activity. [663] Adding H_2O_2 to the pulsed oxidase gave an electronic spectrum which was interpreted as showing a peroxide derivative, [664] but another believes [665] the product is the oxygenated oxidase, which may be a peroxide anyway. Cyt c peroxidase catalyses the oxidation by H_2O_2 of $[Co(terpy)_2]^{2+}$, $[Fe(CN)_6]^{4-}$, $[Ru(RNH_2)_5py]^{2+}$, probably by substrate binding to an H_2O_2 - haem complex and with electron transfer within the enzyme as the rate determining step. [666] The use of $^{51}Cr^{2+}$ isotope showed subunit II was the electron transfer site for that reagent. [667]

Cytochrome P-450 is another intensely active field. Its activity is well-reviewed, [668-671] including emphasis on camphor-5-exohydroxylase [670] and on the different spin and oxidation states and their effects on the redox processes. [671] The theory of the electronic structure of Fe(III) haem proteins has been reworked to add to the $^4T_2$ ground state some ^6A, or other excited state, with the inevitable better fit to experimental data; cyt

P-450 is said specially to benefit from this treatment. [672] The sensitivity of CD spectra in the Soret region proved useful in replacing the sixth ligand in cyt P-450 to model the interaction of haem proteins with the environment. [673] MCD also provides extra detail and confirms the lowspin character of the oxidised form and the highspin nature of the reduced form, which starts to become lowspin at low temperature. [674] Cyt P-450 complexes with esters, ethers and ketones show spectral effects which have hitherto been attributed to solvent action. [675] The two different processes with different rates which were found for cyt P-450 reduction are attributed to different spin states. [676] Other cyt P-450 reactions studied include the conversion of CCl_4 to $COCl_2$, [677] of vinylcyclooctane to epoxide, [678] and the demethylation of $PhNMe_2$. [679]

Related reactions to those of cyt P-450 which have been studied include those of a protein containing 7 or 8 haems, one having highspin Fe(II) with a very high ΔE_q, which converts NH_2OH to NO_2^-; [680] a rat liver phenylalanine hydroxylase which shows two signals for nonhaem highspin Fe(III); [681] a mechanistic study of the action of protocatechuate 3,4-dioxygenase. [682]

There remain some miscellaneous papers on other cytochromes, and these reports cannot be put in any logical order. Electron and proton transfer through cyt bc complexes is reviewed. [683] rR spectra are used to characterise a, b and c cytochromes with the conclusion that haem-haem interactions may vary within the b class and perhaps also the a class. [684] The nitrate reductase haem has been found to be like cyt b_5. [685] Six different types of haem occur in hydroxylamine oxidoreductase; 4 haems are c_{553} type, 2 are c_{559} and one is a P-460 haem. [686] The CD spectrum of NADH-cyt b_5 reductase is reported. [687] EPR showed the two cyt b-563 in the cyt bf complex from spinach are both lowspin with slightly different g-values. [688] A discussion of the role of $[Fe(CN)_6]^{3-}$ in restoring the activity of inhibited cyt bc_1 complex led to the view that this reagent can bind to haem centres. [689] The polarised single crystal electronic spectrum of cyt cd, shows these two haems are perpendicular in reduced and oxidised forms. [690] The formation of a 1:1 complex between cyt f and plastocyanin was established by gel electrophoresis and electronic spectra. [691]

6.4. Porphyrin Complexes

This section comprises the large volume of work on Fe complexes of natural or synthetic porphyrins designed to illuminate biochemical topics involving various haem type proteins.

We begin with Fe(II) complexes of simple porphyrins. Xα calculations on these predict a $^3A_{2g}$ ground state with some ^3E_g excited state mixing if the Fe-N bond is shorter than 2.0Å but a $^5B_{2g}$ state at longer Fe-N distances. [692] Moessbauer spectra were recorded for various highspin 5-coordinate [Fe PPIX] complexes with axial ligands including improbably phenol and catechol. [693] The role of axial ligands (eg NO, py) was one of the variables assessed in a study of factors affecting the redox properties of Fe porphyrins. [694] A combined electrochemical and spectroscopic study of [Fe(TPP)(CS)] and its adducts with N donors showed it can undergo two electron transfer steps, first to Fe(III) and then to a radical with electron loss on the porphyrin. [695] Fitting TPP with phosphorylcholine groups lets its Fe complexes occupy phospholipid bilayers. [696] $[Fe(OEP)(2\text{-}Meim)_2]$ has been prepared and shows a 1/2 - 5/2 spin equilibrium; it is suggested that the orientation of the imidazoles determines the spin state with steric hindrance in the solid state keeping these highspin but this does not apply in solutions. [697] The rR spectra of other 2-Meim porphyrin complexes have been assigned to show which bands are sensitive to ring substituents. [698] O_2 forms complexes with [Fe(TPP)] and [Fe(OEP)] in Ar matrices at 15K which are isomers with monodentate O_2 (ν 1190cm^{-1}) or bidentate O_2 (ν1105cm^{-1}); these are S = 1 or S = 0 spin states respectively. [699] Comparison of CO and O_2 binding to [Fe(TPP)] and its 2,4,6-triphenylphenyl analogue in the presence of imidazoles showed that CO binding was not much affected by steric effects or by H bonding of phen to imidazole; more polar solvents enhance O_2 binding but hinder CO binding, perhaps because a polar solvent will stabilise the charge separation involved in O_2 complexation. [700]

Other reactions of Fe(II) porphyrins include the synthesis of the $[Na(crown\ ether)(thf)_2]^+$ salt of $[Fe(TTP)]^-$ which has a trinegative porphyrin; this group also prepared the diamagnetic $[Na(thf)_3]^+$ salt of $[Fe(TPP)]^{2-}$, in which the porphyrin has 2N atoms out of the ring plane. [701] [Fe(TPP)] catalyses the reduction of allyl bromide to give a porphyrin complex where the α and σ positions have $CH_2.C:CH$ or $CH:C:CH_2$ groups. [702]

Among Fe(III) porphyrins it has been shown that [Fe(TPP)Ph] is lowspin with the Fe only 0.17Å out of the ring plane. [703] Other X-ray structures reported are those of $[Fe(TPP)(SC_6HF_4)]$[704] and $[Fe_2(TPP)_2SO_4]$ in which SO_4^{2-} bridges by one O atom bonding to each Fe. [705] Various porphyrin (por) complexes [Fe(por)Cl] have been synthesised using haems with selective deuteration of Me. [706] The extensively studied $[FeL(ClO_4)]$ complex, L = tetrakis(trimethoxyphenyl)porphyrin, has a 3/2 spin ground state but the bis-thf complex has some 5/2 state mixed in. [707] The Fe(III) - PPIX complex gives 1:1 complexes with α and β cyclodextrins and their methyl and acetyl derivatives. [708] $[Fe(por)(3\text{-}Clpy)_2]^+$ complexes also show interesting magnetochemistry, with basic porphyrins like OEP giving highspin, less basic ones like tetra-3-NO_2PP giving lowspin and intermediate ligands such as PPIXDME giving intermediate results. [709] $E_{\frac{1}{2}}$ values for reduction of $[Fe(TPP)(imidH)_2]^+$ complexes depend on the H bonding power of the imidazole; [710] others studied similar systems in an unsatisfactory attempt to correlate EPR data with various cytochrome systems. [711]

We have already met a sulphate-bridged dimer, and many others were studied this year. The ^{13}C NMR spectra of $[Fe_2(por)_2O]$, (por = OEP, TPP) give similar antiferromagnetism to the earlier ^1H results. [712] Using carbethoxytetramethyl-porphyrins allowed ^1H spectra to reveal conformational isomers as well as antiferromagnetism. [713] Tetrasulphophenylporphine complexes can be monomers or dimers in aqueous solutions depending on the pH. [714] $[Fe_2(TTP)_2O]$ reacts with HN_3 to give $[Fe(TTP)N_3]$; if this is exposed to UV light $[Fe_2(TTP)_2N]$ is formed, but if py is present this photolysis gives $[Fe(TTP)(py)_2]$ and $[Fe_2(TTP)_2O]$. [715] CV experiments showed 2 reductions and 4 oxidations for $[Fe_2(TPP)_2C]$, with the formation [716] also of such py complexes as $[Fe_2(TPP)_2C(py)_2]^{2+}$.

Titrating OH^- with [Fe(PPIX)Cl] or [Fe(PPIXDME)Cl] in the presence of imidazoles at 77K gave all sorts of azole derivatives including protonation and deprotonation, but at 298K all these revert to [Fe(por)OH]. [717] The reaction of alkalis with iron (III) [Fe(PPIX)] appears to involve the interaction of the alkali metal with carboxylate before giving $[Fe_2(por)_2O]^{4-}$ species. [718]

Iron (III) porphyrin complexes also form an active area. Methoxide reacts with [Fe(TPP)Cl] to give successively [Fe(TPP)(OMe)] and $[Fe(TPP)(OMe)_2]$, the latter being the first lowspin Fe(III) porphyrin complex with two O axial ligands. [719] In a spectroelectrochemical and ^1H NMR study of [Fe(por)Ph], (por = OEP, TPP) and pyridine complexes thereof it was found that redox changes gave delocalisation of charge between cation and porphyrin; [720] however, when slow scan rates were used, the first oxidation step is followed by an irreversible reaction to an intermediate which can lose one electron to form an Fe(III) - $\underline{N}$Ph complex which in turn can be reduced to an Fe(II) - $\underline{N}$Ph one which can then be irreversibly reduced back to the starting material. [721] Determining the equilibrium constant for forming a complex between NCO^- and Fe(III) haem octapeptide in 50% aqueous glycol showed this was a weaker complex than that with met Hb or Met Mb. [722] In the latest of a series of papers MCD spectra were used to conclude that a highspin 5-coordinate Fe(II) complex was an intermediate in the catalysis of the electroreduction of O_2 by an Fe(III) TCPP complex (TCCP = 4-carboxyphenyl analogue of TPP). [723] An Fe(III) deuteroporphyrin IX complex acts as a peroxidase model in catalysing the chlorination of monochlorodimedone by $NaClO_2$ with the formation of hypochlorite. [724] This complex also oxidises dithionite in a diffusion controlled process which is greatly retarded when human serum albumin is added and which is also affected by different anions and the pH. [725] The solvent dependence of the electronic spectra of [Fe(PPIXDME)NO] and its complexes with N ligands is reported. [726] [Fe(por)NO], (por = PPIXDME, TPP) reacts with NO to give N_2O and $[Fe(por)(NO)(NO_2)]$. [727]

An SCF calculation for [Fe(porphine)O] shows the Fe = O configuration is less stable by 66kJ mol^{-1} than inserting the O between Fe and N and suggests such may be the situation in catalase. [728]

Various studies describe reactions which might model cytochromes. These include a study of redox catalysis at an octane-water interface by a coproporphyrin complex, [729] the demetallation of Fe(III) TPP (or its 4-OMe derivative) complexes by reactions with $B(OH)_3$ under high pressure and transmetallation reactions with Cu_2O, [730] and the partial dechlorination of CCl_4, $CHCl_3$ and CH_3CCl_3 but not some other chlorohydrocarbons by Fe(II) porphyrins. [731] The self-exchange electron transfer rate for $[Fe(TPP)(imid)_2]^{2+/3+}$ is slightly higher than for short cytochromes. [732] The reduction of Fe(II) deuterioporphyrin by Me_2CO radicals produced by the irradiation of alkaline aqueous isopropanol is followed by slow reoxidation, probably by water, but this process is much faster if $S_2O_4{}^{2-}$ is the reductant. [733]

The tetra(4-methoxyphenyl)porphyrin complex [Fe(por)SH] is prepared from the chloride analogue by reaction with S and $LiBHEt_3$; its spectral and magnetic data are similar to cyt P-450 and chloroperoxidase. [734] The X-ray structure gives the expected result for [Fe(OEP)SPh]. [735] The carbanion porphyrin complexes $[Fe(TPP)(CF_3CHCl)SR]$ have uv-visible and EPR spectra like halothane - cyt P-450 products. [736] Moessbauer spectra of [Fe(PPIX)] complexes with S ligands show unusually strong Fe-S bonds. [737] [Fe(TPP)] reduces many cyt P-450 substrates, including $R_3N \rightarrow O$ and arene oxides. [738] Of Fe porphyrins only [Fe(TpivPP)(O_2CMe)] catalyses alkene oxidation by hypochlorite. [739] The [Fe(TPP)] complex with thioglycolate ethyl ester forms a 1:1 adduct with O_2 which is a superoxide complex and which has spectral similarities with cyt P-450 intermediates. [740] [Fe(TPP)Cl] cleaves selectively some C-C bonds in 1,3-diols as a model of the enzymic degredation of lignins. [741]

Cyt-c models include compounds of haemin with haemoctapeptide, wherein a histidine from the peptide takes the 5th position and modifies the effects of added 6th ligands such as picoline or azide. [742] Crystal field calculations on histidine peptide Fe(III) complexes are claimed to show these model cyt c. [743] [Fe(4-MeO-TPP)Cl] is converted by ^{t}BuOOH to an isoporphyrin with a t-butylperoxy group entering at the meso-position; this may be an alternative process to that usually considered for reactions between metalloporphyrins and organoperoxides. [744]

There has been more useful progress on picket fence and other porphyrins with elaborate structures. The field has been given a readable review. [745] The EPR of some "basket handle" porphyrins [Fe(por)Cl] shows Fe(III) in a mixed 3/2 - 5/2 spinstate. [746] New cyclophane-capped porphyrins include Fe(III) complexes where the spin state is affected by the length of the bridging chains or the size of the axial ligand, which also controls whether 5- or 6-coordination is achieved. [747] Using $2\text{-}C_6H_4NHCOCMe_2(CH_2)_nCO_2R$, (n = 1,2,3,10; R = H,Me,CH_2Ph), "pickets" gave Fe(III) complexes with Cl or Br as 5th ligands. [748] Another capped porphyrin links C_6H_6 to [Fe(TPP)] by 4 [$O(CH_2)_4OC(O)$] units, with less steric hindrance for CO binding than for analogues with 2 or 3 CH_2 groups. [749] Oxidation of an Fe(II) - tetra(2,5-^{t}Bu$_2$-4-OH-phenyl) porphyrin complex with peracids or O_2 and base gives a stable complex which oxidises phenols, while its Fe(III) complex with 1-Meimid is reduced to Fe(II); comparisons were drawn with peroxidase or cyt c peroxidase. [750] Another picket fence porphyrin complex with axial 1-lauryl-

-2-methylimidazole, when dissolved in a phospholipid bilayer, binds O_2 reversibly at pH 7 at 37°C; electric dipole measurements indicate the porphyrin and bilayer lie parallel. [751] The X-ray structure of a ^{t}Bu picket fence porphyrin-O_2-SC_6F_5 complex shows O_2 is monodentate inside the porphyrin cup; the O-O distance is apparently surprisingly short (1.14Å) due to disorder and decomposition by X-rays and the Moessbauer spectrum resembles that of oxygenated cyt P-450. [752] Four isomers were found for another [Fe(por)OH] picket fence complex, which is converted to an O-bridged dimer by 8hr refluxing in $CHCl_3$. [753] The enthalpy and entropy for O_2 and CO binding to the Fe(II) tetrapivalamidophenylporphyrin in phosphatidylcholine lipids are similar to those for Hb. [754] In an extension of earlier work it was found that O_2 and NO have similar trends in binding constants with various Fe(II) capped porphyrin azole complexes and it was concluded that the diatomic ligands adopted the bent monodentate configuration but the enforced linearity of CO coordination would generate different steric constraints for the different regions of the ligand. [755] The ingenious model for cyt c oxidase, $[Fe(tetranicotylporphyrin)Cu]^{2+}$, has been prepared and shows weak ferromagnetism between two spins of ½, but this coupling is lost in some solvents, though not in MeOH/$CHCl_3$ with or without 1-Meimid; a diamagnetic product or products are formed in dmf. [756]

6.5. Iron-Sulphur Compounds

This section concerns proteins containing iron and sulphur and synthetic compounds specifically designed to mimic them. Compounds containing molybdenum and other metals are deferred to the next section.

General papers include a wide ranging review on S ligands in metalloproteins and enzymes [757] and two reviews on core extrusion analysis, [758,759] one of which highlights the use of detergents and chromatography. [758] EPR is used to determine spin-lattice relaxation times; adding the Dy-edta complex as paramagnetic relaxer allows the determination of the distance of a paramagnetic centre from the protein surface. [760]

Too many papers refer to ferredoxins without specifying which of the various ferredoxin structures is present. Examples of these are an rR study of Se replacement of S, [761] the discovery of an electron transport protein in nitrite reduction, [762] electron transfer with gold electrodes using "rotating disc hydrodynamic voltammetry", [763] and the discovery of [Fe-S] proteins in spinach glutamate synthase [764] and mitochondrial NADH dehydrogenase. [765]

A preliminary X-ray study on a rubredoxin from Desulfovibrio desulfuricans shows this is the smallest so far, with only 45-48 residues, but including the unusual histidine. [766] Rubredoxin and a [4Fe-4S] ferredoxin have been found in Clostridium formicoacetum. [767] The role of surface charge is important in CV studies of rubredoxin and other proteins using RuO_2 or IrO_2 electrodes. [630]

Turning to [2Fe-2S] proteins, a new ferredoxin (Fd) has been isolated from Trichomas vaginalis. [768] Benzene dioxygenase from Pseudomonas putida consists of a flavoprotein, a [2Fe-2S] Fd and two other [2Fe-2S] units whose EPR resembles Rieske proteins. [769] An admirable group of papers [770-4] uses modern NMR methods to obtain structural information. ^{13}C enrichment of Fd from the bacterium Anabaena variabilis allowed full assignments with the aid of 2-D and decoupling techniques; relaxation times were shorter in the more paramagnetic reduced form. [770] Similar work on Fd from another bacterium or from spinach gave improved assignments of the cys protons and studies of the hyperfine shifts. [771] The ^1H and ^{13}C spectra for the histidine units gave conformational information in reduced and oxidised forms. [772] $[Cr(NH_3)_6]^{3+}$ and Fd-NADP reductase bind in a similar area containing at least 3 glutamate residues. The primary sequence of aminoacids was obtained using 360 or 470 MHz spectra, 2-D methods, $[Cr(NH_3)_6]^{3+}$ binding and chemical modification with carboxypeptidase A and trinitrobenzenesulphonic acid. [774] Another ^1H NMR study assigned all the protons near Fe and distinguished CH_2 units near Fe(II) from those near

Fe(III) by the different temperature dependence of their chemical shifts. [775] The rR spectrum of the Fd from *Clostridium pasteurianum* which is known as the "red paramagnetic protein" (RPP) shows this has the usual structure but a slight weakening of all the Fe-S bonds and a change in the conformation occur. [776] The kinetics of reductions by $[Cr(15\text{-}aneN_4)]^{2+}$ of [2Fe-2S] and [4Fe-4S] proteins show both are inner sphere processes and that binding Cr^{3+} to the reduced protein does not affect oxidation by $[Co(NH_3)_6]^{3+}$; addition of $[Cr(en)_3]^{3+}$ does not inhibit reduction by the Cr(II) complex but actually accelerates it for the [2Fe-2S] protein. [777] The Rieske [2Fe-2S] protein from *Thermus thermophilus* was isolated and the iron atom which is reduced was shown to have at least one non-cys ligand, not identified at this stage, there being just 2 cys residues between the 2Fe. [778] The behaviour of ferredoxins containing 3Fe atoms is curiouser and curiouser. There was a report of the crystallisation and preliminary structural work on a 3Fe Fd from *Desulfovibrio gigas*, but the authors were unable at the time to determine whether they had encountered a planar or 3/4 cubic configuration. [779] ESR spectra on [3Fe-3S] and [3Fe-4S] Fd proteins suggest these are more similar than the X-ray work claimed but this inconsistency was thought possibly to arise from modifications when crystals became frozen aqueous solutions. [780] Unusually fast relaxation occurs in the 3Fe proteins from *D. gigas* or *Azotobacter vinelandii*, confirming earlier ideas that these have low lying excited states arising from spin coupling. [281] An EPR study of the conversion of the [3Fe-4S] cluster of Fd II from *D. gigas* to [4Fe-4S] shows this could occur in *in vivo* conditions. [782] The [3Fe-3S] and [3Fe-4S] cores can be distinguished by rR spectra. [783] Fd proteins from *Pseudomonas ovalis* and *Mycobacterium smegmatis* are shown by rR spectra to contain both 3Fe and 4Fe clusters. [784] *A. vinelandii* FdI is normally isolated as a combination of [4Fe-4S] and [3Fe-3S] cores, but it can be reconstituted as an 8Fe core, so it was suggested that one Fe is lost in the isolation procedure. [785] The use of $^{35}S^{2-}$ to study the exchange of S^{2-} in aconitase showed

3S exchanged easily, so it was supposed that the [3Fe-4S] cluster was accessible to solvent; Fe is also taken up easily, suggesting formation of a [4Fe-4S] cluster to convert the inactive enzyme to an active one. [786] The oxidation of Fd I from *A. vinelandii* by $[Fe(CN)_6]^{3-}$ produced an intermediate corresponding to a 3-electron oxidation of the [4Fe-4S] unit; this was believed to be a cysteinyldisulphide radical formed by the loss of S and Cys from the [4Fe-4S] cluster. [787] In another study of this reaction, using ^1H NMR spectra, excess of $[Fe(CN)_6]^{3-}$ converted the [4Fe-4S] core to a [3Fe-4S] and then a [3Fe-3S] unit. [788]

In work on [4Fe-4S] proteins it was shown that this structure is found in FdI from *D. africanus*, [789] and in a hydrogenase from *D. vulgaris*. [790] EXAFS work on a hydrogenase from *Methanobacterium thermautotrophicum* shows this is similar to $[Fe_4S_4(SPh)_4]^{2-}$, but there was uncertainty on the S/Fe ratio. [791] Substituting Se for the core S in oxidised Fd from *Clostridium pasteurianum* gave little change in the Moessbauer spectrum, but reduction gave a much more complex spectrum and a triple EPR spectrum, attributed to the usual spin state of ½ being supplemented by 3/2 and 7/2 states. [792] The [4Fe-4S] proteins exchange D for H more readily than HP or [2Fe-2S] proteins, especially in the oxidised state; electron spin echo decay measurements show this occurs close to the metals. [793]

A [4Fe-4S] cluster in a 7Fe Fd from *P. ovalis* was found to be both oxidisable and reversible; it is asserted that this is the first such Fd to show this in its native form. [794] A kinetic study of the reaction of the oxidised forms of plastocyanin and azurin with HP proteins showed this was not a simple process. [633]

A new class of Fd containing 5Fe atoms has been found in a lichen *Lobaria pulmonaria*. [795]

Next, model compounds, beginning with mononuclear ones. Unstable $[Fe(SR)_4]^-$ compounds (R = Ph, C_6H_4Me) have been prepared [796] from $[Fe\{o\text{-}(SCH_2)_2C_6H_4\}_2]^-$. $[Fe(H_2O)_4Cl_2]$ reacts with

o-$C_6H_4S_2^{2-}$ to give planar $[Fe(S_2C_6H_4)_2]^{2-}$, but if PMe_3 is present square pyramidal $[Fe(S_2C_6H_4)(PMe_3)_3]$ is formed, which in turn reacts with CO to form $[Fe(S_2C_6H_4)(PMe_3)_2(CO)_2]$ and $[Fe(S_2C_6H_4)(PMe_3)_2(CO)]_2$. [797] Some Fe(III) complexes of short peptides containing two cys units in the sequence found in rubredoxins had similar electronic, CD and MCD spectra to the protein. [798] Fe(II) complexes of similar type showed similar likenesses to the reduced form but only the complex of $PhCH_2O$ cysproleucysOMe had reversible redox behaviour; it could transfer electrons from NADP oxidoreductase to ferricytochrome c. [799]

Some dinuclear Fe-S complexes have been studied. A variety of routes may produce $[Fe_2S_2(S_5)_2]^{2-}$, the simplest method being the reaction of $FeCl_2$, S and Na; using Se for S in this synthesis gave the Se analogue whose X-ray structure shows the expected 2 Se bridges and 2 bidentate Se_5^{2-} ligands, resulting in tetrahedral Fe. [800] $[Fe_2S_2Cl_4]^{2-}$ was used to prepare compounds with dicysteinepeptides; an Fe(II) complex with the peptide can be blended with S to give the [2Fe-2S] model system. [801]

Another series of derivatives from $[Fe_2S_2Cl_4]^{2-}$ is prepared with 4 molecules of pyrrole or 2 of 2,2'-biphenyldiol. [802] Ferrous ammonium sulphate reacts with $(NH_4)_2WS_4$ and Et_4NI in the presence of saccharoses in water to give $(Et_4N)_2[Fe(H_2O)_2(WS_4)_2]$, and $[FeCl_2(WS_4)]^{2-}$ reacts [803] with NaSH to give $[Fe_2S_2(WS_4)_2]^{2-}$.

A thorough investigation of linear $[Fe_3S_4(SR)_4]^{3-}$, (R = Et, Ph) includes magnetization studies and Moessbauer spectra in dmf to show that in both solid and solution phases there is strongly antiferromagnetic Fe(III), though the coupling between the end Fe atoms is described as being between -100 and +100 cm^{-1}! These results contrast with the lowspin character of ferredoxins; the authors comment that the linear structure has been found in aconitase. [804]

Improved preparations of $[Fe_4S_4(SR)_4]^{3-}$, (R = alkyl), from $FeCl_2$, NaSR and NaSH in dmf allow these to be formed without previously isolating the dianions. The Et and ^{t}Bu compounds have compressed tetragonal structures which are about 2.5% bigger in volume than the dianions. [805] Reacting $[Fe_4S_4Cl_4]^{2-}$ with $2EPh^-$, (E = O,S), gives $[Fe_4S_4Cl_2(EPh)_2]^{2-}$, which are said to be the first Fe_4S_4 cores which do not have the compressed D_{2d} structure; further reaction of the SPh complex with $2OR^-$, (R = Ph, 4-MePh) gives replacement of the remaining chlorine. [806] As models for the [4Fe-4S] clusters in nitrogenase various $[Fe_4S_4L_4]^{2-}$ species were studied with the expected result that for L = SPh, OPh or Cl there was charge delocalisation, but not for L = Et_2NCS_2; it was suggested that the unusual spin state of the iron-sulphur cube in nitrogenase was due to unusual coordination. [807] $[Fe_4S_4(S^{t}Bu)_4]^{2-}$ exchanges its thiolate ligands with N - Phenylacetylcysteinyl-glycylcysteinamide. [808] Reacting $[Fe_4(cp)_4S_6]$ with $[MoOCl_3(thf)_2]$ gives the unusual cation $[Fe_4(cp)_4S_5]^+$ with a modified Fe_4S_4 cube in which one S^{2-} is replaced by an S_2^{2-} which is η^2 to one Fe and η^1 to two others. [809] Two new hexanuclear clusters are described. $[Fe_6S_6I_6]^{2-}$, consisting of 2 Fe_3S_3 "chairs" linked by Fe-S bonds, is prepared [810] by the reaction of specific proportions of Fe,I_2,S and I^-. $[Fe_6S_6Cl_6]^{3-}$, a hexagonal prismatic structure, is produced from $FeCl_2$, SPh^-, S and Cl^- in MeCN. [811] The new clusters $[Fe_6S_9(SR)_2]^{4-}$, (R = Et, CH_2Ph) and $[Fe_6S_9(SMe)_2Na_2]^{6-}$ are described; the latter has a condensation of $4[Fe_3NaS_4(SR)_6]^{5-}$ units. [812]

6.6. Iron-Molybdenum-Sulphur Compounds

This section describes nitrogenases, compounds which are designed to mimic this protein and other Fe-Mo species whose connection with nitrogenase is more tenuous.

The unlikely reagent bipy gives a [2Fe-2S] compound on reaction with oxidised Fe protein from Azotobacter vinelandii nitrogenase. [813] Reactions of Fe_3MoS_4 clusters include that of $[Fe_7Mo_2S_8(SEt)_{12}]^{3-}$ with tetrachlorocatechol (H_2cat) to give $[Fe_6Mo_2(SEt)_6(cat)_2]^{4-}$ which in MeCN forms $[Fe_3MoS_4(SR)_3(cat)(MeCN)]^{2-}$; the MeCN can be replaced by CN^-, N_3^-, N_2H_4,NH_3,NH_2NHPh or pip and other SR^- derivatives were prepared. [814] The crystal structure of $(Et_4N)_3$ $[FeMo_2S_8O_2]$.EtCN confirms that O is bound only to Mo. [815] Another structure report describes [816] $[Fe\ \ MoS_3(SC_2H_4S)\ \ _2]^{3-}$ as "S-shaped". New preparations [817] include $[Fe_2MoS_4(PhSH)_2(OMe)_4]^-$ and $[Fe_8MoS_9(PhCH_2SH)(OMe)_6]$, which both reduce C_2H_2 and N_2, $[Fe_2MoS_4Cl_4]^{2-}$, $[Fe_2MoS_4Cl_4(SR)]^{2-}$, (R = Ph,CH_2Ph), and $[S_2MoS_2FeS_2MoOS_2]^{3-}$. The formation of an FeMo complex was indicated by CD studies of the interaction of $[Fe_4S_4(SR)_4]^{2-}$ with Mo(IV) peptide complexes, (R = ^{i}Pr, Ac-cys-OH). [818] $Fe(O_2CMe)_2$ is used to prepare $[(AcO)_2FeS_2MS_2]^{2-}$, (M = Mo,W), from which $[(PhS)_2FeS_2MS_2]^{2-}$ can be prepared, and $[Fe(MoO_2S_2)_2]^{2-}$, which can also be prepared from $Fe(S_2CNR_2)_2$ and which is thought to have Fe-O-Mo-O-Fe bridging. [819]

6.7. Haemerythrin

There have been many valuable developments on this interesting protein. Two readable reviews have been published. [820, 821] The structure of metaquohaemerythrin from Thermiste dyscritum shows one 6- and one 5-coordinate (sort of trigonal bipyramidal) Fe, but unlike the metazido- form there is no small ligand on the sixth site of the 5-coordinate Fe so the designation "metaquo" is a misnomer. [822] There has been further work on the metazido structure. [823]

A thorough ^{1}H NMR study of methaemerythrin and semimethaemerythrin concentrated on the bridging histidine resonances, which are not shifted as much as normal for highspin Fe(III) because of the antiferromagnetism. In the semimet-form the his bonded to Fe(II) could be distinguished from those bonded to Fe(III); sulphatosemimet- is more antiferromagnetic than the azidosemimet-form [824] The rR spectra of oxyhaemerythrin and the azidomet-form gave identification of many important vibrations; D_2O substitution indicated

[825] that O_2 binds as HO_2^-.

Oxidation of deoxyhaemerythrin by NO_2^- is unusual in producing the semimet-form, probably by an inner-sphere process, whereas outer-sphere oxidants like $[Fe(CN)_6]^{3-}$ give methaemerythrin; the NO_2^- oxidation proceeds via an EPR - silent nitrosyl intermediate. [826] In another kinetic study this deoxy- to semimet- oxidation was achieved [827] by various $[Fe(CN)_4XY]^{n-}$ species (X = CN^-, Y = CN^-, NH_3, PPh_3, 4-NH_2py; XY = bipy) and of semimet- to met- by these reagents and $[Co(terpy)_3]^{3+}$. There have been further studies on the related protein rusticyanin, including the kinetics of Fe^{2+} reduction and EPR of reaction with the Dy(III) - edta complex. [828]

6.8. Iron Transport Proteins

The subject of siderophores has been reviewed again, with emphasis on ligand synthesis, complex formation energetics and complex configuration. [829] Fe^{3+} is stereospecifically complexed by the Δ-isomer of rhodotorulic acid. [830] Various $RN(OH)C(O)(CH_2)_nC(O)N(OH)R$, (R = Ph, Pr; n = 3-10) ligands form non-antiferromagnetic Fe_2X_3 complexes like rhodotorulic acid and these can be converted to $[Fe_2X_2(OR)_2]$, (R = Me,Et) in alcohols. [831, 832] Mono-, di- and trihydroxamate complexes of Fe can be distinguished by their response to CN^-; the first forms $[Fe(CN)_5(NO)]^{3-}$, the second loses its blue colour and the third does not react. [833] The reactions of ferrioxamine B with edta and hydroxamates were studied as models for the release of Fe from siderophores. [834] Retrohydroxamate, a polypeptide with 3 *N*-methylhydroxamate units, has some biological activity as a synthetic siderophore despite being a weaker ligand. [835]

Many new siderophores have been characterized. The bacterial siderophore pyoverdine Pa was found by mass and NMR spectra to be an octapeptide bound to 2,3-diamino-6,7-dihydroxyquinodine to give two hydroxamate and one catechol functional groups. [836] *Vibrio cholerae* has a siderophore, vibriobactin, which contains three 2,3-dihydroxybenzoate and two threonine residues. [837]

Rhizobactin comes from the N_2-fixing bacterium Rhizobium melitoti, but has neither catechol nor hydroxamate ligands. [838] A siderophore of ferrichrome type appears in the bacterium Aspergillus ochraceous. [839] That from the microbe Proteus mirabilis contains a unit of isovaleric acid, $Me_2CHCH(OH)CO_2H$. [840] A strain of smut fungus has a ferrichrome siderophore of glycyl units in a heptapeptide instead of the usual hexapeptide. [841]

The rR spectrum shows 2,3-dihydroxybenzoate and EXAFS shows thioglycolate bond to Fe(III) in ovotransferrin. [842] Pyrophosphate can transport Fe liberated from transferrin into rat liver mitochondria and might fulfil this role in vivo. [843] ESR spectra of an Fe^{3+}-hist-citrate model system for transferrin confirm Fe is bound to 2 his, 3 citrate and possibly 1 HCO_3^- but the natural system probably has coordination by ascorbate. [844]

6.9. Ferritin

Two reviews [845,846] have appeared, one of them being a good easy general survey suitable for novices. [845] The structure of horse spleen ferritin has been described afresh. [847] The X-band EPR spectra of ferritin and haemosiderin show two features from $Fe(OH)_3$ cores which are similar; [848] electron microscopy and Moessbauer evidence reinforce the conclusion that these are similar materials but that haemosiderin particles are smaller. [849] The molecular weight of pigeon ferritin is given, with incredible precision, as 354,813, including 5810 Fe atoms per mol; chicken ferritin is smaller. [850] The UV difference spectra of the Fe^{2+} - apoferritin reaction, together with the effects of Zn^{2+} and Tb^{3+}, shows there is more than one site both for Fe^{2+} binding and for Fe^{3+} binding after the reorganization that follows oxidation. [851] The release of Fe from ferritin is promoted by O_2^- and suppressed by adding superoxide dismutase. [852] The commercial preparation Niferex, a neutralised solution of an Fe(III) carbohydrate complex, has a Moessbauer spectrum and X-ray diffraction like ferrihydrite and is therefore like ferritin. [853]

7. ORGANOMETALLIC COMPOUNDS

Organising material in this vast topic is very difficult. Papers are described under the heading for the dominant ligand in the chemistry which is described, but such a classification is occasionally both arbitrary and inconsistent. Readers are advised to consult more than one section when looking for a particular topic.

7.1. Carbonyls

We begin with [Fe(CO)$_5$]. Ab initio calculations on its photodissociation lead to the conclusion that excitation to a singlet state occurs before crossing to $^3E'$ and dissociating. [854] The laser induced fluorescence of [Fe(CO)$_5$] is described. [855]

Numerous examples have been described of the use of [Fe(CO)$_5$] in the isomerization of alkenes. Substrates reported in 1984 include 1- and trans-2-pentene, [856,857] 1-hexene, [858] CH_2:$CH(CH_2)_nN(SiMe_3)_2$, [859] and this general reaction has undergone kinetic study. [860] Laser irradiation of [Fe(CO)$_5$] produces a thermally active catalyst for the gas phase hydrogenation of C_2H_4. [861] PhN:CHPh has also been hydrogenated with [Fe(CO)$_5$] catalysis, [862] and the role of [HFe(CO)$_4$] in this reaction has been described. [863] [Fe(CO)$_5$] with CO,H_2O, base and a crown ether catalyses the regiospecific hydrogenation of anthracene, quinoline and other N-heterocycles. [864] Two reports describe its use in the carbonylation of benzyl halides to phenylacetic acid derivatives. [865,866] It can, in dmf, induce the replacement of one Cl in $Cl(CH_2)_4CCl_2$ by H. [867]

Decomposition studies of [Fe(CO)$_5$] include experiments on the process carried out after adsorption on zeolites, [868] or on polythene, where [Fe(CO)$_4$] and [Fe(CO)$_3$] were spectroscopically detected. [869] Photooxidation in CCl_4 gave $FeCl_2$ and organics formed from a dichlorocarbene resulting from the oxidative addition of CCl_4 to [Fe(CO)$_4$]. [870] γ-irradiation of [Fe(CO)$_5$] in a Kr matrix gave, [871] according to EPR, $[Fe(CO)_5Kr]^+$. The carbonyl reacts with 2-EtimidH (L) to give $[FeL_6]^{2+}[FeH(CO)_5]^-$, whose anion is fluxional. [872]

Turning to $[Fe(CO)_4]$ species, $[Fe(CO)_4]^-$ has been shown to abstract halogen from CCl_4, CF_3Br and CF_3I. [873] $[Fe(CO)_4]^{2-}$ is a catalyst in the formation of hydrocarbons from CO and H_2 at 350-450 °C; the role of K^+ is discussed. [874] Most work in this section concerns $[Fe(CO)_4L]$ complexes. Their XPE spectra show changes in d orbital energies in line with expectations on whether L is π-bonding. [875] The ligand RP:PR, [R = 2,4,6-$(^tBu)_3C_6H_2$], is monodentate in $[Fe(CO)_4(RPPR)]$. [876] $HC(PPh_2)_3$ is more versatile, forming monodentate complexes $[Fe(CO)_4L]$, bidentate complexes $[Fe(CO)_3L]$ and polymetallic complexes $[(CO)_4FeLFe(CO)_3]$ and $[(CO)_4FeLMo(CO)_4]$. [877] There seems to be a craze for forming phosphine complexes with molybdenum moieties tacked on. Other examples for 1984 include $[Fe(CO)_4\{P(NMe_2)_2Mo(cp)(CO)_2\}]$ with an FeMoP ring, [878] $[Fe(CO)_4\text{-}\{P(NPhPF_2)Mo(CO)_3\}]$, [879] and $[Fe(CO)_4\{PPh_2OPPh_2Mo(CO)_5\}]$. [880] There is an unusual equatorial phosphine in $[Fe(CO)_4\text{-}\{(Me_3Si)_2C:PN(SiMe_3)\}]$, whose C=P bond of 1.657Å is described as short and this ligand has a 30° twist. [881] A tetrameric phosphazene with oxazole rings on alternate P-N bonds forms a complex with $4[Fe(CO)_4]$ groups. [882] The phosphorus ylide complexes $[Fe(CO)_4CH(R)P^+Ar_3]$,(Ar = Ph, p-tolyl) are formed when the parent phosphine is added to a mixture of $[Fe(CO_4(SiMe_2CH_2)_2]$ and an aldehyde; alkylidene intermediates are invoked for the reaction and the Ph_3 derivative reacts with CF_3SO_3H to give $(PhCH_2PPh_3)^+(CF_3SO_3)^-$. [883] New arsine complexes of $[Fe(CO)_4]$ include those with $AsMe(NMe_2)_2$, $AsMeX_2$ or $AsMe_2X$, (X = Cl, OEt, SEt), [884] and antimony provides $[Fe(CO)_4\text{-}\{(Me_3Si)_2CHSbSbCH(SiMe_3)_2\}]$ and $[\{Fe(CO)_4\}_2SbCH(SiMe_3)_2]$ which have $FeSb_2$ and Fe_2Sb triangles respectively. [885] The reaction of $(Me_3E)_3Sb$, (E = Si,Ge) with $[Fe_2(CO)_9]$ gives $[Fe(CO)_4\ Sb(EMe_3)_2]$ but if $(Me_3Sn)_3Sb$ is used the product is $[Fe(CO)_4(SnMe_3)_2]$. [886]

Bismuth is not so varied and is content with $[Fe(CO)_4(BiR_3)]$, (R = alkyl). [887]

$[Fe(CO)_4]$ bonds to group IV ligands in forming $[Fe(CO)_4(CO_2)]^-$ when $[Fe(CO)_5]$ is adsorbed on MgO. [888] Kinetic studies and the observation of CIDNP showed H atom transfer occurs in the reaction between $[Fe(CO)_4(SiCl_3)H]$ and dienes. [889] $[Fe_2(CO)_9]$ reacts with $[\{(MeC_5H_4)(CO)_2Mn\}_2Ge]$ to give $[\{Fe(CO)_4\}_4Ge]$ and $[\{Fe(CO)_4\}_3{-}Ge\{Mn(CO)_2(MeC_5H_4)\}]$. [890] Sn or Ge porphyrins form $[Fe(CO)_4\{M(por)\}]$; the X-ray structure shows the Sn-OEP complex has square pyramidal Sn with Fe axial and trigonal bipyramidal Fe. [891] One of the most exotic $[Fe(CO)_4]$ complexes is formed when $[Fe(CO)_5]$ reacts with $[Cr_2(cp)_2(O^tBu)_2]$ to produce $[Cr(cp)(\mu\text{-}O^tBu)_2Cr(cp)Fe(CO)_4]$ which has an equilateral Cr_2Fe triangle with no bridging ligands between Cr and Fe. [892]

There is also a good selection of $[Fe(CO)_3]$ species. Me_2S_2 reacts with $[Fe_3(CO)_{12}]$ to give $[\{Fe(CO)_3\}_2SMe]_2$ and $[Fe_2(CO)_6(SMe)_2]$. [893] Dithiocarbamate, dithiophosphate and xanthate (X) complexes which have been prepared are $[Fe(CO)_3IX]$ and $[Fe(CO)_2X_2]$. [894] Photochemistry in organic glasses at 100K converts $[Fe(CO)_4PPh_3]$ into $[Fe(CO)_3PPh_3]$ or $[Fe(CO)_3(PPh_3)L]$, (L = glass medium); in $SiEt_3H$ the product is $[Fe(CO)_3H(PPh_3)(SiEt_3)]$ whose <u>fac</u> - isomer can be converted to the <u>mer</u> - form by further irradiation. [895] Bipy and phen (L) complexes $[Fe(CO)_3L]$ can be prepared by carbonylation of $[Fe_2(CO)_7L]$ or by reacting the diimine with $[Fe(CO)_3(PhCH{:}CHCMe{:}CH_2)]$. [896] The silicon ligand $Me_2SiCH{:}CMeCMe{:}CH$ also forms an $[Fe(CO)_3]$ complex, [897] but this might be a diene complex rather than with bonding through Si. Gaseous $[Fe(CO)_4]$ radicals react with H in H_2 to give $[Fe(CO)_3H_n]^-$ (n = 0-3). [898]

There are a few complexes with fewer than 3 carbonyl groups per iron atom. These include $[Fe(CO)_2(dtc)_2]$ and $[Fe(CO)(xanthate)_2L]$, for which the PPh_3 complex was more stable than those with N donors. [899] There has been a thorough study of the complexes $[Fe(CO)_2(PPh_3)_2(RNCS)]$, (R = Me, Ph) prepared by the reaction of RNCS with the tris-phosphine complex; RNCS acts as an η^2-ligand with back-bonding to both the CN-π^* and S-d orbitals, and further reactions are described substituting one PPh_3 by $P(OPh)_3$ and the RNCS by $4F-C_6H_4N_2^+$, while various electrophiles E convert RNCS to RENCS. [900] Reacting $[Fe(CO)_2(PPh_3)_2(CS_2)]$ with Ph_2PCC^tBu gave $[Fe(CO)(PPh_3)(SCOMe)(SC^tBu{:}CHPPh_2)]$ which could in turn be converted to a wide range of other phosphinoalkenethiolate complexes. [901]

There is the usual abundance of dinuclear complexes. Reacting $[Fe_3(CO)_{12}]$ with 2-nitropropane in PhMe gives $[Fe_2(CO)_6(\mu\text{-}OCCH_2Ph)(\mu\text{-}NCMe_2)]$. [902] EHMO calculations on $[Fe_2(CO)_6S_2]$ and related compounds were compared to their PE spectra. [903] The reaction of $[Fe(CO)_5]$ with $[Cr(CO)_5(SCSC_2H_4S)$ gives $[Fe_2(CO)_6(\mu\text{-}SC_2H_4S)]$, $[Fe_2(CO)_6(\mu\text{-}SCSC_2H_4S)]$, $[Fe_3(CO)_9)(\mu_3\text{-}S)_2]$ and $[Fe_3(CO)_9(\mu_3\text{-}S)(\mu_3\text{-}CSC_2H_4S)]$; in the last of these a C atom bridges 2Fe and an S links across to the other one. [904] A dithiolate complex of $[Fe_2(CO)_6]$ is said to be produced in the reaction of $[Fe_3(CO)_{12}]$ with sulphur. [905] This carbonyl reacts with $(Me_3SiN)_2S$ to form $[Fe_2(CO)_6(SNSiMe_3)]$, from which $[Fe_2(CO)_6(SNH)]$ and $[Fe_2(CO)_6(SNMe)]$ were prepared. [906] The dithioformate esters HCS_2R, (R = Me, Et, allyl) form $[Fe_2(CO)_6L]$ with $[Fe_2(CO)_9]$ with both S and CS bridging; a similar product is formed with RCSOEt, but HCSOEt gives $[Fe_2(CO)_6L_2]$ in which only S bridges. [907] Among the products of the reaction of $[Fe_3(CO)_{12}]$ with benzalazine [?] are $[Fe_2(CO)_6]$ and $[Fe_3(CO)_8]$ complexes of $C_6H_4CH_2NN{:}CHPh$. [908] The structure of $[Fe_2(CO)_6(PR)_4]$ complexes are reported. [909] The reaction of 1,2,3-triphenyl-1,2,3 - triphosphaindane with $Fe_2(CO)_9$ gives many products including complexes with $[Fe_{2\ or\ 3}(CO)_{8,9,10}]$ units and an $[Fe_2(CO)_6]$ complex with 1,2-bisphenylphospidobenzene. [910] $(PNCl_2)_n$ react with $[Fe_2(CO)_8]^{2-}$ to give $[Fe_2(CO)_8(P_nN_nCl_{2n-2})]$, (n = 3,4), with P bridging 2Fe or an $[Fe_3(CO)_{10}]$ complex with bridging by P and CO. [911] The reaction of $[Fe(CO)_5]$ or $[Fe(CO)_4(CS)]$ with $HgCl_2$ in various solvents gives the binuclear complexes $[Fe(CO)_5\ HgCl_2]$, $[Fe(CO)_4(CS)HgCl_2]$ and $[Fe(CO)_4\{C(S)OEt\}(HgCl)]$ as well as trinuclear $[Fe(CO)_4(HgCl)_2]$ and polymeric $[Fe(CO)_3(CS)Hg]$. [912] The versatile triphosphine $HC(PPh_2)_3$, (L), mentioned earlier [877] also forms $[Fe(CO)_5RhL]^+[Rh(CO)_2Cl_2]^-$ in which 2P bond the Fe, one bonds Rh and there is an Fe-Rh bond. [913]

$[Fe_2(CO)_9]$ has been used to prepare tetrahydrobenzotriazenes by reaction with adiponitriles and RCN. [914]

Trinuclear complexes make up a very big section. $[Fe_3(CO)_{12}]$ catalyses the reaction of perfluoroalkyl halides with allylsilanes to give polyfluoroalkenes. [915] The conditions for the conversion of $[Fe(CO)_4H]^-$ into $[Fe_3(CO)_{11}H]^-$ have been reinvestigated. [916] This anion adsorbed on NaY zeolite is a good catalyst for the water gas shift reaction, comparable to $[Fe(CO)_5]$ in the homogeneous mode. [917] Reactions of $[Fe_3(CO)_9(OCMe)]^-$ have been reported with mechanistic discussion about C-O cleavage; HBF_4 gives $[Fe_3(CO)_9H(OCMe)]$, $[Fe_3(CO)_{10}H]$ and $[Fe_3(CO)_9(HOCMe)]$, but $MeSO_2F$ gives $[Fe_3(CO)_9(CMe)(OMe)]$ and $[Fe_3(CO)_9(CMe)(OCMe)]$. [918]

There has been some interesting work on trinuclear complexes with sulphur ligands. Na_2S and $Na[HFe(CO)_4]$ give $[Fe_3(CO)_9(S)H_2]$, which easily loses two protons to give $[Fe_3(CO)_9S]^{2-}$, isolated as the Et_4N^+ salt. [919] $[Fe_3(CO)_{12}]$ reacts with $(4\text{-}MeOC_6H_4PS_2)_2$ to give $[Fe_3(CO)_9S(PC_6H_4OMe\text{-}4)]$, with μ_3-S and PR groups; [920] this and related compounds can also be prepared by the reaction of $[Fe(CO)_4]^{2-}$ with $RPSCl_2$, (R = Me, Ph, $4\text{-}MeOC_6H_4$). [921] 3-Chloroperbenzoic acid oxidises $[Fe_3(CO)_9S_2]$ to $[Fe_3(CO)_9S(SO)]$. [922]

There are a number of phosphorus ligands which form Fe_3 trinuclear cluster complexes. $[Fe_3(CO)_8(PPh_2)_2H_2]$ is prepared from

$[Fe_4(CO)_{13}]^{2-}$, CF_3CO_2H and Ph_2PH; it has an isosceles Fe_3 triangular structure and reaction with CO converts it to $[Fe_3(CO)_9$-$(\mu_3\text{-}PPh)_2]$.[923] Reactions of $RHPCH_2PRR'$, (R = alkyl; R' = R,H) with $[Fe_2(CO)_9]$ gave $[Fe_3(CO)_9]$ cluster compounds with or without P-C cleavage. [924]

The isocyanide F_3CNC can be stabilised by acting as a bridge in the complex $[Fe_3(CO)_{11}(CNCF_3)]$; [925] the ${}^tBuNC$ analogue rearranges to $[Fe_3(CO)_9(CN{}^tBu)]$, which can be reduced by $KHB{}^iPr_3$ to $[Fe_3(CO)_9HC{:}NC{}^tBu]$ which is in turn converted by H_3PO_4 into $[Fe_3(CO)_9H(HC{:}NC{}^tBu)]$. [926] The enthalpy change for the reaction of $[Fe_3(CO)_{10}H(CMe)]$ with H_2 to form CO and $[Fe_3(CO)_9H_3(CMe)]$ was used to show that the FeHFe bond energy is greater than that for Fe(CO)Fe. [927] $[Fe_3(CO)_9H(BH_4)]$ has been prepared from $[Fe(CO)_4(COMe)]^-$ and $[BH_3.thf]$; its structure was solved using the H positions of the isoelectronic $[Fe_3(CO)_9H_3(CH)]$ and it was seen to have BH_4^- capping an isosceles Fe_3 triangle, but the BH_4 hydrogens can exchange with the hydride one at 60 °C. [928] $[Fe_3(CO)_{10}H(BH_2)]$, having a BH_2 cap, is prepared from $[Fe(CO)_5]$, $[Fe(CO)_4(COMe)]^-$ and $[BH_3.thf]$ and deprotonates [929] to $[Fe_3(CO)_{10}(BH_2)]^-$.

There are also a number of trinuclear heteropolymetallic clusters. The reaction of $[Fe_3(CO)_{12}]$ with ${}^tBuSH$ and $[MnL(CO)_3]$, (L = cp, Mecp), gives $[Fe_2(CO)_6(\mu\text{-}CO)_2(\mu_3\text{-}S)(MnL)]$. [930] The reactions of $[Fe_2Ru(CO)_{12}]$ and $[FeRu_2(CO)_{12}]$ with PPh_3 or $P(OMe)_3$ are compared; if one carbonyl is replaced the structure expands but for double reaction some CO bridges form, leading to the conclusion that the di-iron carbonyl has the $[Fe_3(CO)_{12}]$ structure but the diruthenium one does not have that of $[Ru_3(CO)_{12}]$. [931] Isonitrile derivatives of $[FeCo_2(CO)_9S]$ have been prepared and their redox behaviour compared with many $[Fe_nCo_{(3-n)}E]$ derivatives, (E = S,Se,PR,CR). [932] Other examples of this type are $[FeCo_2(CO)_9PR]$, (R = Me, ${}^tBu$,Ph,PMe_2Ph), the last of these giving chiral or meso complexes when a CO on one or both Co are replaced respectively; this paper also describes the preparation of $[\mathrm{FeCoM(CO)_9H(PR)}]$, (M = Fe,Ru), which is also chiral and has a hydride bridge between Fe and M. [933] Also in this area there are descriptions of reactions of $[\mathrm{FeCo(CO)_6\,S\,M(cp)(CO)_2}]$, (M = Cr,Mo,W) with $\mathrm{M'AsMe_2}$, $[\mathrm{M = M(cp)(CO)_3,\ Fe(cp)(CO)_2}]$ leading to the preparation of $[\mathrm{Fe(CO)_3S\{M(cp)(CO)_2\}_2}]$, (M = Mo,W); [934] On to larger clusters. Low temperature NMR (^{1}H, ^{13}C) of $[\mathrm{Fe_4(CO)_{13}H}]^-$ shows the butterfly and tetrahedral structures are in equilibrium in $\mathrm{CD_2Cl_2}$. [935] This area is a favourite with molecular orbitallers. One group sought to account for the difference between the structure of $[\mathrm{Fe_4(CO)_{13}H}]^-$ and that of $[\mathrm{Fe_4(CO)_{12}H(CH)}]$; [936] another established the metal-domination of frontier orbitals in $[\mathrm{Fe_4(CO)_{13}C}]$, $[\mathrm{Fe_4(CO)_{12}C}]^{2-}$, $[\mathrm{Fe_4(CO)_{12}(H)C}]^-$ and $[\mathrm{Fe_4(CO)_{12}(CCO_2Me)}]^-$; [937] a third was interested in the unreactivity of the exposed C atoms in $[\mathrm{Fe_4(CO)_{12}C}]^{2-}$ and $[\mathrm{Fe_5(CO)_{15}C}]$. [938] The structure of $[\mathrm{Fe_4(CO)_{12}S_4}]^{2-}$, formed from $[\mathrm{Fe_2(CO)_6S_2}]$ and $\mathrm{LiEt_3BH}$ in THF, consists of two $[\mathrm{Fe_2(CO)_6S_2}]^-$ ions linked by a S-S bond 2.164A long. [939]

The tetrahedral cluster $[\mathrm{Fe_3M(CO)_{14}}]^{2-}$, (M = Cr,Mo,W), prepared by the reaction of $[\mathrm{Fe_3(CO)_{11}}]^{2-}$ with $[\mathrm{M(CO)_3(MeCN)_3}]$ can also be described as a hexacapped rectangular antiprism of carbonyls. [940] The adventitious preparation of $[\mathrm{FeRu_3(CO)_{13}H_2}]$ allowed a more precise determination of the structure of the second form of this compound. [941] Use of ^{2}D NMR led to finding the broad hydride resonance in $[\mathrm{FeCo_3(CO)_{12}H}]$. [942] Reacting $[\mathrm{Ni(CO)_4}]$ with $[\mathrm{Fe_3(CO)_{11}}]^{2-}$ gives $[\mathrm{Fe_3Ni(CO)_{12}}]^{2-}$, which can be protonated to $[\mathrm{Fe_3Ni(CO)_{12}H}]^-$, which can also be prepared from $[\mathrm{Fe_3(CO)_{11}}]^{2-}$ and $\mathrm{NiCl_2}$ and whose X-ray structure shows an $\mathrm{Fe_3Ni}$ tetrahedron with 8 terminal and 4 edge-bridging CO and a hydrogen over an $\mathrm{Fe_2Ni}$ face. [943] $[\mathrm{Fe_2(CO)_9}]$ and $[\mathrm{Mn(cp)(CO)_2PBr_3}]$ give the linear cluster $[\mathrm{\{Mn(cp)(CO)_2P\}_2Fe_2(CO)_6}]$, with each P bridging 2Fe as well as forming a double bond to Mn. [944] The most complicated piece of chemistry I have read this year describes reactions of CO with $[MM^1M^2M^3(\mu_3\text{-}E)(\mu_2\text{-}AsMe_2)]$, ($M\text{-}M^3$ = Fe,Ru,Co,Mo,W, all with various cp and CO ligands), to give tetranuclear complexes with loss of a Co-As fragment; among the species which are produced are $[FeCo_2Mo(cp)(CO)_{10}S(AsMe_2]$ and $[FeCo_2Mo(cp)(CO)_8S(AsMe_2)]$ and they all revert to the starting materials in a vacuum. [945] $[FeCoPdPt(CO)_6(NO)(dppm)_2]$ is prepared from $[Fe(CO)_3(NO)]^-$ and $[CoPdPt(CO)_3I(dppm)_2]$. [946] $[FeCo_3(CO)_{12}]^-$ and MLCl, (M = Cu, Ag; L = PPh_3, PhPCH:CMe:CH, MeCN), react to give $[FeCo_3(CO)_{12}ML]$. [947] The seemingly innocuous combination of $[Fe(CO)_5]$, $[Mo(CO)_6]$ and Na/Hg in diglyme gives $[Fe_4Mo_2(CO)_{18}C]^{2-}$ which is said to react with $FeCl_3$ to give $[Fe_4Mo_2(CO)_{19}C]$. [948] Many Fe-Rh clusters are reported, e.g. $[Fe_4Rh(CO)_{14}C]$ and $[Fe_4Rh_2(CO)_{16}C]$. [949] Moessbauer spectra of iron cluster compounds and mixed metal clusters allow correlations of Moessbauer parameters with whether the Fe atom has all its carbonyls terminal, some bridging or with H bridges. [950]

7.2. Nitrosyls

Preparations are reported for $[Fe(NO)(PR_3)_4]^+$, (R = OMe, OEt), and $[Fe(NO)(PR_3)_2(CNR')_2]$, (R' = aryl); all of these have trigonal bipyramidal geometry with equatorial linear NO. [951] The nitrosyls $[Fe(NO)_2(PPh_3)_2]$, $[Fe(NO)_2(CO)(PPh_3)]$ and $[Fe(NO)(CO)(PPh_3)L]$, (L = $4\text{-}Ac\text{-}C_6H_4N_2$), are prepared [952] by the action of NO_2^- on $[Fe(NO)(CO)_2(PPh_3)_2]^+$ and $[Fe(CO)_2(PPh_3)_2L]^+$. In an elaborate series of reactions $[Fe_2(NO)_4(PPh_2)_2]$ is reduced by Na, $LiAlH_4$ or $Na[AlH_2(OCH_2OMe)_2]$ to $[Fe_2(NO)_4(PPh_2)_2]^{2-}$ with bridging PPh_2^- ligands. This is reduced by $LiBHEt_3$ to $[Fe_2(NO)_4(PPh_2)(PPh_2H)]^-$, with bridging by NO and PPh_2^-. Reaction of this with BuLi gives the dianion again, but with mixed bridges; the other isomer forms on storing at -70 °C, but reaction with CH_2I_2

gives an isomerising mixture of $[Fe_2(NO)_4(CH_2)(Ph_2PPPh_2)]$ and $[Fe_2(NO)_4(CH_2PPh_2)]$. [953] $[Fe(cp)(NO)]_2$ is reduced by Na to the dinuclear monoanion; CH_3I converts this to $[Fe(cp)(NO)Me]_2$, which is in rapid equilibrium with the monomer, and thermally unstable $[Fe(cp)(NO)(Me)_2]$, which reacts with PMe_3 to form $[Fe(cp)(PMe_3)(NOMe)Me]$ wherein bonding of NOMe is through N. [954]

7.3. Alkyls and Aryls

This section comprises compounds whose behaviour centres on the formation, reactivity or theory of a sigma bond between Fe and C, but this classification has been interpreted generously. We begin with alkyls and acyls. Ab initio SCF calculations on CO insertion into Fe-C or Fe-H bonds in $[Fe(CO)_4(CH_3)H]$ indicate that the hydrido-acetyl complex is more stable than the methyl-formyl one. [955] The $[FeMe]^+$ ion cleaves C_3H_8 to form $[FeEt]^+$ in equilibrium with $[Fe(C_2H_4)H]^+$, but a stable C_2H_4 complex is formed from C_4H_8. [956] Mass spectra have been used to study the gas phase reactions of Fe^+ with cyclic ketones to give metallacycles and their subsequent decomposition. [957] Ion-molecule reactions of $Fe(CO)_n^+$ with hydrocarbons and ketones gave a range of products, including $[FeC_2H_4]^+$, $[Fe(CO)H_2]^+$, $[Fe(CO)C_2H_6]^+$, $[FeC_3H_8]^+$ and four varieties of $[FeC_4H_8]^+$. [958] A mass spectrometric study of various $[FeC_nH_{2n}]^+$ found that for n>4 the hydrocarbon broke down; mechanisms are discussed. [959] Sparking halomethanes on an Fe surface gave $[FeCXY]^+$, $[FeCX]^+$ and $[FeX_2]^+$, (X,Y = H,Cl,Br). [960]

Turning to conventional preparative chemistry, $[FeBr(mesityl)(thf)_{0.5}]$ has been prepared from $FeBr_2$ and $[Mg(mesityl)_2(thf)_2]$. [961] ESR spectra of ^{13}C-labelled $[Fe(CO)_4(RCO)]$, (R = long chain alkyl) shows these have axial acyl groups with an unpaired electron in the equatorial plane; this result is said to explain the radicals' failure to dimerise. [962]

$[Fe(CO)_4(CH_2CO_2Me)]^-$ is formed from $[Fe_2(CO)_8(CH_2)]$ by reaction with NaOMe in dmso; other reactions of the dinuclear precursor are reported.

[963] In the presence of various ligands L, $[Fe(CO)_3(PMe_3)_2Me]^+$ and $[Fe(CO)_2(PMe_3)(Me)X]$, (X = I,CN) are converted to $[Fe(CO)_2(PMe_3)_2(OAc)L]$, $[Fe(CO)(PMe_3)_2(OAc)LX]$ and $[Fe(CO)_2(PMe_3)(Me)L]^+$. [964] $[Fe(CO)_4(C_3F_7)I]$ reacts with $SPHEt_2$ to give $[Fe(CO)_3(C_3F_7)(SPHEt_2)]$; reaction of this with NH_3 gives a cyclic FePS compound which inserts into alkynes to give a C_2PSFe ring which reacts with more alkyne to give a bicyclic structure. [965] At -30 °C, $[Fe(CO)_2(PMe_3)_2(Me)X]$, (X = I,CN,Me,$CNBPh_3$) react with CO to give unstable $[Fe(CO)(PMe_3)_2(Me)(COMe)X]$ which revert to the starting material even under argon. [966] The Fe-C bond in $[Fe(CO)_2(PMe_3)_2MeI]$ is subject to insertion with $C_6H_{11}NC$; subsequent oxidative addition and rearrangement yield $[Fe(CO)_2(PMe_3)_2\{C(:CH_2)N(C_6H_{11})C(:N^+HC_6H_{11})\}]^+$. [967] The conversion of acetyl to methyl was observed in the FAB mass spectra of $[Fe(CO)_2(diars)(COMe)L]^+$, (L = monodentate ligand). [968] The photodecarbonylation in a PVC matrix at 12-200K of $[Fe(cp)(CO)_2(COMe)]$ is thermally reversible; the photolysis of $[Fe(cp)(CO)_2Me]$ gives reversible loss of one CO. [969] The latter compound reacts with $[Mo(cp)(CO)_3]^+$ to give the acetyl-bridging $[Fe(cp)(CO)_2(COMe)Mo(cp)(CO)_3]^+$. [970] Carbonyl insertion into $[Fe(cp)(CO)_2CH_2CH_2R]$ is retarded by adding Ph_3C, which then leads to elimination. [971] The acetoxy compound $[Fe(cp)(CO)_2(CO_2Me)]$, which can be converted to $[Fe(cp)(CO)_2(CH_2OMe)]$, is prepared from $[Fe(cp)(CO)_2]_2Mg$ by the reaction of CO_2 to give $[Fe(cp)(CO)_2(CO_2)]_2Mg$ and the reaction of that with CF_3SO_3Me. [972] $[Fe(cp)(CO)_2C(S)SR]$, [R = $Fe(cp)(CO)_2$,$Re(CO)_5$] react with $[M(CO)_5(thf)]$, (M = Cr,Mo,W) or $[Mn(cp)(CO)_2(thf)]$ with the thf being replaced by the uncoordinated S; the R component can be lost to give a CS_2 moiety bidentate to Cr,Mo,W or Mn. [973] Another mixed metal complex, $[Fe(cp)(CO)(\mu\text{-}PPh_2)(\mu\text{-}COMe)Mn(CO)_4]$, is prepared from $[Fe(cp)(CO)_2(PPh_2)]$ and $[Mn(CO)_5Me]$, but $[Mn(CO)_5H]$ gives the analogue with bridging hydride, not formyl. [974] Condensation of imines with

$[Fe(cp)(CO)(PPh_3)(CO_2AlEt_2)]$ gives $[Fe(cp)(CO)(PPh_3)\ COCH_2CH(Ph)NHPh$ stereoselectively; iodine in CH_2Cl_2 converts these to β-lactams. [975] *o* -$C_6H_4(CH_2Br)_2$ converts $[Fe(CO)_4]^{2-}$ to $[Fe(CO)_3(CH_2C_6H_4CH_2Br)_2]$; if PPh_3 is present $[Fe(CO)_2(PPh_3)(COCH_2C_6H_4CH_2Br)(CH_2C_6H_4CH_2Br)]$ is formed. [976] In another reaction involving dibromoxylene $[Fe_2(CO)_6(PHPh_2)_2]$ is converted to $[Fe_2(CO)_6\{PC_6H_4(CH_2PPh)_2\}]$; LiBu converts this to a complex in which one Fe is bonded to a xylyl carbon next to an alkylated P. [977] The porphyrin complexes [FeLR(NO)], (L = OEP,TPP; R = Me, aryl) have been prepared. [978]

Aryls are outnumbered by alkyls, but there is some interesting work. Interactions between the 2-hydrogen and Fe in [Fe(cp)(Ar)(CO)L] complexes, (L = CO,PPh_3) have been detected by IR, ^{1}Hand ^{13}C NMR and Moessbauer spectra. [979] The mass spectra of $[Fe_2(CO)_6(C_6H_4CH_2NPh)]$ and similar complexes are reported. [980] A similar complex to this, but with ^{i}Pr instead of Ph on N is prepared from $[Fe_2(CO)_9]$ and $[(CO)_5W{:}C(Ph)(NH^iPr)]$ using $[(CO)_5M{:}C(COR)(NHC_6H_{11})]$, (M = Cr,W; R = Me,Ph) gives a complex with $2Fe(CO)_3$ moieties bridged by (RCO) and $(CNHC_6H_{11})$ groups. [981] $[Me_2Si\{(C_5H_4)Fe(CO)_2\}_2]$ reacts with $Ph_2P(CH_2)_nPPh_2(L)$ to give either $[Me_2Si\{(C_5H_4)Fe(CO)\}L]$ or $[Me_2Si\{(C_5H_4)_2Fe_2(CO)_3\}_2L]$; if n is small the dimer (CV study) is preferred but if n is large or more reagent is used the tetramer is favoured. [982]

Four papers describe metallocyclic compounds. A ferracyclopentane is prepared by reacting $[Fe(CO)_3L]^{2-}$, (L = CO, PPh_3) with $F_3CSO_3(CH_2)_4O_3SCF_3$; using the dimethylene reagent gave $[Fe(CO)_3L(C_2H_4)]$. [983] Aziridine, oxirane and thiirane react with $[Fe(cp)(CO)_2L]^+$, (L = CO,CS,PPh_3) to give five-membered cyclic carbene complexes. [984] Fe atoms react with B_5H_9, toluene and 2-butyne to give $[Fe(C_6Me_6)(2,3\text{-}Me_2C_2B_4H_4)]$ and three similar products; the structures of all were described. [985] The reaction of [Fe$_2$(CO)$_9$] with ROC(S)SMe, (R = adamantyl) gives [Fe$_2$(CO)$_6$ ROC(S)SMe], which contains an FeSCS ring with the other Fe bonded to C, the thione S and Fe. [986] The next section is about alkylidene and related compounds, taken in order of progressively fewer carbonyl groups and increasing numbers of Fe atoms. [Fe(cp)(CO)$_2$(CH$_2$CH:CHOMe)] is prepared from [Fe(cp)(CO)$_2$(CHOMe)]$^+$ *via* the intermediate [Fe(cp)(CO)$_2$(CROMe)]$^+$, (R = alkyl, aryl). [987] Rearrangement of N(C$_2$H$_4$O)$_2$PCH$_2$CH:CH$_2$ in an [Fe(cp)CO] complex gave a methylvinyl complex with a P-bonded residue. [988] Alkenyl complexes such as [Fe(cp)(CO){P(OPh)$_3$}(CMe:CMePh)] can be converted into acylalkenyl ones by reaction with CO catalysed by Ag$^+$, Ce^{4+} or ferricinium ions. [989] [Fe(cp)(CO)(PPh$_3$)(C:CMe$_2$)]$^+$ reacts with Li$_2$Cu(CN)R$_2$, (R = Ph, CH:CH$_2$) to give [Fe(cp)(CO)(PPh$_3$)(CR:CMe$_2$)] and with PhS$^-$ to give a complex with R = SPh; the vinyl derivative rearranges to an η^3 - CH$_2$CHC:CMe$_2$ complex. [990] Complicated bridged complexes are formed when alkynes react with [Fe$_2$(CO)$_7$(CPh:CHPh)]. [991] The synthesis of [Fe(TPP)C:CR$_2$], (R = 4-ClC$_6$H$_4$) has been achieved starting from [Fe$_3$(CO)$_{12}$] [992].

Ferracarbene complexes, containing (Fe:C) moieties, will be discussed in a similar order to the alkylidene complexes. [Fe(CO)$_4$(CROEt)], R = Bu, Ph react with alkynes and CO to give pyrone complexes of [Fe(CO)$_3$] which rearrange to isomers through a ferracyclobutene intermediate which was isolated. [993] The action of LiN^{i}Pr$_2$ followed by Et$_3$O$^+$ on [Fe(CO)$_5$] produced [Fe(CO)$_4${C(OEt)N^{i}Pr$_2$}] and [Fe(CO)$_3${C(N^{i}Pr$_2$)OC(O)Et}], in which the ketonic O atom is also coordinated. [994] The complex [(cp)(CO)$_2$Fe(CH.CH:CMe$_2$)]$^+$ can be used to form cyclopropanes. [995] The preparations and reactions of [Fe(cp)(CO)L Fe(CH.CH:CRCH$_2$R')]$^+$, [L = CO, P(OMe)$_3$; R = H, R' = Me; R = Me, R' = H] are reported. [996] The alkenyl complex [Fe(cp)(CO)(PMe$_3$){C(OMe):CH$_2$}] has been converted to various carbene complexes $[Fe(cp)(CO)(PMe_3)\{:C(OMe)R\}]$, (R = Me, CH_2CS_2, $CH_2C(S)SMe$). [997] The reaction of $[Fe_2(CO)_6(\mu\text{-}SMe)(\mu\text{-}SC(OR):)]$ with $P(OMe)_3$ shows a CO on the carbene-bonded Fe is the most labile; [998] there are X-ray and ^{13}C NMR studies on the mono-$P(OMe)_3$ complexes for the R = mesityl and adamantylmethyl. [999] The cyclopropylidene bridging complex $[Fe_2(cp)_2(CO)_5(C_3H_4)]$ is prepared by the reaction of H_2CN_2 with a bridging vinylidene complex. [1000]

$[Fe(cp)(CO)(MeCN)\ C(SMe)_2\ ]^+$ reacts with $[Co(CO)_4]^-$ to form $[Fe(cp)(CO)(\mu\text{-}CO)\ \mu\text{-}C(SMe)_2\ Co(CO)_2]$ which has an S-Co bond. [1001]

The simplest carbene complex, with CH_2, is given when $[Fe(cp)(CO)_2]^-$ reacts with $Me_3CCO_2CH_2Cl$ to form $[Fe_2(cp)_2(\mu\text{-}CO)(\mu\text{-}CH_2)]$ as the cis-isomer, but C_5Me_5 gives the trans-isomer. [1002] A ^{31}P NMR study of the rotation barriers in [Fe(cp)(diphosphine)(vinylidene)] complexes showed steric effects were important from both non-cp ligands. [1003] The porphyrin carbene complexes [Fe(por)CRR'], (por = TPP, TTP; R = Me, ^{i}Pr; R' = MeO, EtO, $PhCH_2S$ can be made from [Fe(por)Cl] and Cl_3CR, which gives a (CClR) chlorocarbene complex, which is then treated with R'H. [1004]

The ketenylidene complex $[Fe_3(CO)_9(CCO)]^{2-}$ reacts with $[M(CO)_3(NCMe)_3]$, (M = Cr,W) to give carbide complexes $[Fe_3MC(CO)_{13}]^{2-}$ which are similar to $[Fe_4C(CO)_{12}]^{2-}$, but $[Co_2(CO)_8]$ gives $[Fe_2Co(CO)_9(CCO)]^-$ in which the CCO is closer to perpendicular than in the Fe_3 analogue. [1005] Successive reaction of the carbene complex $[Fe(CO)_4\{C(OEt)N^iPr_2\}]$ with PPh_3 and BCl_3 gives the carbyne complex $[Fe(CO)_3(PPh_3)\{CN^iPr_2\}]$ in which the carbyne is equatorial, the phosphine axial and with the shortest Fe-carbyne bond of 1.734 Å. [1006]

$[Fe_4(CO)_{12}H(CH)]$ in CF_3SO_3H gives CH_4 only after a long induction period, but this reaction is accelerated by adding [1007a] such "reducing agents" as $TiCl_3$, $MoBr_2$, Fe or $[Fe_2(CO)_8]^{2-}$. The use of deuterium substitution in the reaction of alkenes with $[Fe_2(cp)_2(CO)_3(CD)]^+$ allowed the mechanism to be established. [1007b]

7.4. Alkene Complexes

This section describes complexes where the major point of interest lies in the behaviour of a C=C bond linked to an Fe atom. Allyl complexes are included and the ordering of topics is in decreasing numbers of carbonyl ligands and increasing nuclearity.

PRDDO molecular orbital calculations on $[Fe(CO)_4(C_2R_4)]$, (R = H,F,Cl,CN) show the alkene should bond in the equatorial position for each one. [1008] NMR spectra of the vinyl protons in $[Fe(CO)_4]$ complexes of chiral alkenes were used to analyse their stereochemistry. [1009] Bromostyrene complexes were formed by reaction with $Na_2[Fe(CO)_4]$. [1010] $[Fe(CO)_4(SiMe_3)R]$, produced by the alkylation of $[Fe(CO)_4(SiMe_3)]^-$, rearranges to give a siloxycarbene complex and $[Fe(CO)_4(RCH:CHOSiMe_3)]$. [1011] The hexaalkene hericene forms $[Fe(CO)_4]$ complexes with up to 3 of these units on each alkene molecule. [1012] The tautomerism of $[Fe(CO)_4$ Ph.CH:CHC(Me):NR] and its π-allyl-σ-carbamoyl $[Fe(CO)_3]$ analogue has been studied. [1013]

The complex $[Fe(CO)_3L_2]$, (L = cis-cyclooctene) is recommended as a source of $[Fe(CO)_3]$. [1014] Reactions of exo-and endo- complexes of $[Fe(CO)_3]$ with 5,6-dimethylidenebicyclo[2.2.2.]oct-2-ene leading to hydroxy and keto ligands are described and lead to the revision of earlier configurational assignments. [1015] Various allyl-alkyl complexes of $[Fe(CO)_3]$ have been used in lactam synthesis. [1016] The reaction of $[FeH(CO)_4]^-$ with the diazabutadiene (L) complexes $[M(CO)_3BrL]$, (M = Mn, Re) gave $[Fe(CO)_3]$ complexes with Fe-M and Fe-allyl bonds. [1017]

Use of the chiral phosphines $Ph_2PNRCHMePh$,(L), gave chiral complexes $[Fe(CO)_2(NO)(allyl)L]$ for allyl or 2-methylallyl, [1018] but alkyl phosphines PR_3 gave reaction at the allyl ligand to form $[Fe(CO)_2(NO)(\eta^2\text{-}CH_2:CH.CH_2PR_3]$. [1019] A bicyclo [3.2.1] octene complex of $[Fe(cp)(CO)_2]^+$ was formed by a new ring expansion reaction of a norbornyl complex. [1020] A number of complexes of the type $[Fe(cp)(CO)_2(ROCH{:}CHOR)]^+$ are reported. [1021] The reaction of $[Fe(CO)_2(PMe_3)_2MeI]$ with ^{t}BuNC gives $[Fe(CO)_2(PMe_3)_2(MeC{:}N^tBu)]^+$, in which the C=N bond coordinates like an alkene. [1022]

An improved preparation of allyltricarbonyliron lactone complexes involves the reaction of $[Fe_2(CO)_9]$ with alkenyl epoxides. [1023] The methylidyne-bridged complex $[Fe_2(cp)_2(CO)_3CH]^+$ reacts with alkenes to give alkylidynes and δ,π-bridging vinyl complexes. [1024a] Benzylideneacetone, PhCH:CH.CMe:O, acts as an allyl ligand in various CO and PR_3 complexes. [1024b]

$[FeCl_2(PMe_3)_2]$ and potassium pentadienide give $[Fe(PMe_3)_2(pentadienyl)_2]$. [1025]

7.5. Alkyne Complexes

Alkynes react with $[Fe(CO)_2(PMe_3)_2(MeSCHS)]$ to give as final product the complex $[Fe(CO)_2(PMe_3)_2(MeSCHCOCR{:}CRS)]$, (R = e.g. MeO_2C); an alkyne complex might be an intermediate. [1026] Alkyne (L) complexes $[Fe(cp)(CO)\{P(OPh)_3\}L]^+$ react with nucleophiles like $CH_2{:}CHCu(CN)Li_2$ to give alkenyl or alkynyl complexes. [1027] With most alkynes RC:CR, (R = Me,Ph,CF_3, p-MeC_6H_4), $[Fe_2W(cp)(CO)_9(p\text{-}MeC_6H_4)]$ gives $[FeW(cp)(CO)_5\{(p\text{-}MeC_6H_4)CCRCR\}]$, but $RC{:}CSiMe_3$, (R = ^{t}Bu, Me_3Si) gives $[Fe_2W(cp)(CO)_7(p\text{-}MeC_6H_4)(RCCSiMe_3]$. [1028] The alkynyl complex $[Fe_3(CO)_9(CCMe)]$, in which the organo ligand is σ-bonded to one Fe and π-bonded to the others, can be prepared either by the reaction of methanol with electrochemically generated $[Fe_3(CO)_{10}(CMe)]^{2-}$ or by the reaction of $[Fe_3(CO)_9(CMe)(COEt)]$ with $[Mn(CO)_5]^-$. [1029] The three-way reaction of $[Fe_3(CO)_{12}]$, $[Ru_3(CO)_{12}]$ and Ph:CPh gives $[Fe_3(CO)_9(PhC{:}CPh)]$ and ruthenacyclopentane complexes containing 1 or 2 Fe atoms; reaction of $Ru_3(CO)_{12}$ with $[Fe_3(CO)_9(RCCR)]$, (R = Et,Ph) gives replacement of an Fe by Ru but CNDO calculations rationalise the non-formation of a diruthenium complex by maintaining that it is necessary to maintain alkyne to Fe_2 back donation. [1030] Benzonitrile is found to show both σ and π coordination in both $[Fe_3(CO)_9(NCPh)]$ and $[Fe_4(CO)_{12}(NCPh)]$, which are prepared when this ligand and hydrogen react with $[Fe_3(CO)_{12}]$. [1031] A complex formulated as $[Fe_3(CO)_9(MeC{:}CO)]^{2-}$ is prepared by the electrochemical reduction of $[Fe_3(CO)_{10}(CMe)]^-$. [1032]

7.6. Diene and Polyene Complexes

This section considers complexes of ligands containing more than one C=C bond. Monomeric complexes are treated first and the order is of increasing complexity of the diene after any general papers. Cyclopentadienyl complexes make up section 7.7. Allyls have been included, possibly unwisely, in section 7.4.

A number of general papers concern various $[Fe(CO)_3(diene)]$ complexes. A short review discusses their use in natural product synthesis. [1033] They have been used for the regio- and stereospecific synthesis of olefinic triphenylphosphonium salts through reactions with PPh_3. [1034] There has been a CD study of complexes $[Fe(CO)_3]$ with chiral dienes. [1035] The natural chiral diene aminoacid gabaculine has been synthesised through these intermediates. [1036] Iron carbonyls react with chiral dienones to give chiral complexes which preserve their chirality when reacting with prochiral dienes to give $[Fe(CO)_3(diene)]$. [1037] Cyclopentanones are formed by reacting nucleophiles like $LiCMe_2CN$ with these complexes. [1038]

The X-ray crystal structure of the butadiene complex $[Fe(PMe_3)(diene)_2]$ is reported. [1039] Butadiene also forms $[Fe(PPhMe_2)_3(diene)]$, by reaction with $[Fe(PPhMe_2)_3(C_2H_4)_2]$, which is in turn prepared from $[Fe(acac)_3]$, $Et_2Al(OEt)$ and the phosphine. [1040] Various butadienes containing an $SiMe_3$ group form $[Fe(CO)_3(diene)]$ complexes on reaction with iron carbonyls. [1041] $[Fe_2(CO)_9]$ and <u>cis</u>-$[PtCl_2(Ph_2P{:}C^tBu)_2]$ react to give an $[Fe(CO)_3]$ complex of cyclobutadiene, but other Pt complexes produced cyclopentadienone complexes. [1042] Various reactions of C_4Ph_4

complexes are reported: $[Fe(CO)(NO)(PPh_3)(diene)]^+$ and $(Ph_3P)_2N^+.Cl^-$ give $[Fe(NO)(PPh_3)Cl(diene)]$; $[Fe(CO)_2(NO)(diene)]^+$ and $(Ph_3P)_2N^+.X^-$ give $[Fe(NO)X(diene)]^2$, (X = Cl, NCO, NCS, NO_2, N_3); the dicarbonyl cation gives [Fe(NO)(X)(diene)] monomer with X = NO_3, Et_2dtc or acac, the last being prepared with Tlacac. [1043]

Reactions of $[Fe(CO)_3]$ complexes of ^{i}PrN:CH.CH:N^{i}Pr and other diazadienes with dimethylacetylenedicarboxylate are reported with crystallographic confirmation of the extensive ligand rearrangement. [1044]

The Moessbauer spectrum of bis(pentadienyl) iron (II) complexes show more electron density on the Fe nucleus than for ferrocene. [1045] The reaction of nucleophiles like $LiCHPh_2$ with such diene complexes as $[Fe(CO)_3(isoprene)]$ gives allyls at high temperatures and homoallyls at low temperatures; a mechanistic discussion is given. [1046] 1,1-Dimethylsilole, $Me_2Si.CH:CH_2.CH:CH_2$, reacts with $Fe_2(CO)_9$ to give $[Fe(CO)_3(diene)]$; one CO ligand can be replaced by PPh_3. [1047] *Ab initio* molecular orbital calculations on $[Fe(CO)_3(diene)]$ complexes show cyclopentadienone coordinates more strongly than cyclobutadiene. [1048]

The stereochemistry of reactions of substituted cyclohexadienes when coordinated to $[Fe(CO)_3]$ is discussed; [1049] there is similar work on these and cycloheptatrienyl complexes. [1050] The cyclohexadiene complex reacts with $LiCMe_2CN$ to give an allyl complex but if CO is present an acyl-alkene complex is given. [1051] Rotational barriers in cyclohexadiene complexes have been assessed by ^{13}C NMR, with methyl being considered more bulky than methoxy. [1052] $[Fe(CO)_2(PR_3)(\eta^4\text{-}PhCH:CHCOR')]$ complexes have the phosphine in axial or equatorial positions depending on the steric properties of it and the enone; [1053] there has been a kinetic study of the exchange of cyclohexadiene with the enone in these complexes. [1054] New $[Fe(CO)_3(diene)]$ complexes using 3-methoxy-6,6-disubstituted cyclohexa-1, 3-dienes is reported. [1055] The acetylation of the cyclohexadiene complex [Fe(CO)$_2$(PPh$_3$)(C$_6$H$_8$)] gave a 5-<u>endo</u>-acetyl complex. [1056] Ph$_3$C$^+$ converts [Fe(CO)$_3$L], (L = 5,5-dimethyl-2-trimethylsilyl-1, 3-cyclohexadiene), to η^5-cations with SiMe$_3$ at the centre of delocalisation; this and similar reactions were rationalised with PE spectra and MO calculations. [1057] [Fe(CO)$_3$(diene)] complexes have been prepared with N-alkoxycarbonyl-1,2-dihydropyridine. [1058] EHMO calculations on [Fe(CO)$_3$(cyclohexadienyl)] complexes showed how these could be formed from the diene complex and how nucleophilic addition gave regioselectivity. [1059] There has been a ^{13}C NMR and Moessbauer study on solid state motions in this cyclohexadienyl complex. [1060]

In cycloheptadienyl complexes [Fe(CO)$_3$(diene)]$^+$ nucleophilic addition is more inhibited by steric effects than in cyclohexadienyl ones. [1061] The action of "soft" nucleophiles like NaCH(CO$_2$CH$_3$)$_2$ on [Fe(CO)$_2$(PR$_3$)(cycloheptatrienyl)]$^+$, (R = Ph, OPh) gave cyctoheptadiene complexes but "hard" nucleophiles such as NaCN produced allyl complexes [1062]. Wittig reactions on a [Fe(CO)$_3$(diene)] complex gave heptatriene complexes which act as diene ligands and may then undergo isomerization [1063]. Addition of the carbonylcarbenes N$_2$CHCO$_2$Et or N$_2$CPhCOPh to [Fe(CO)$_3$(C$_7$H$_8$)] gave reactions on the C$_7$H$_8$ ligand including formation of a cycloheptafuran complex [1064]. [Fe(CO)$_3$(cot)] and C$_7$H$_7$$^+$ react together to form [Fe(CO)$_3$(η^4-styrylcycloheptatriene)] but the use of methylcyclooctatetraene gives a product with a methyl group on the C$_7$ ring, implying extensive rearrangement; the new complexes react with HBF$_4$ and tcne [1065].

Dimethylcyclooctatetraene-1,8-dicarboxylate forms complexes with 1 or 2 [Fe(CO)$_3$] units; one of the latter has an Fe-Fe bond [1066]. The reaction of [Fe(CO)$_4$]$^{2-}$ and α,α'-dihaloxylenes gives 1,2-bismethylenebenzene complexes which easily rearrange to π-xylylene complexes [1067]. Organic halides react with [Fe(C$_6$Me$_6$)$_2$] to give [Fe(C$_6$H$_6$)(diene)] complexes which react with oxygen to give o-xylylene complexes coordinated by the exocyclic double bonds [1068]. Bis η^5 complexes of bicyclooctadiene with $2[Fe(CO)_2R]$ moieties, [R = CO, $P(OPh)_3$], give ring expansion with PPh_3 to 7-membered triene - diene complexes [1069]. Another triene - diene complex is given when Fe atoms react with bicyclononatriene; when this reacts with $P(OMe)_3$, $[Fe\{P(OMe)_3\}_3(diene)]$ is formed [1070].

Turning next to bigger ring systems, octalene can be converted to bicyclodecapentaene with $[Fe(CO)_5]$ by the intermediate formation of allyl complexes [1071]. Heating many $[Fe(CO)_3(C_7H_7.CH{:}CHR)]$ complexes gave duron complexes but $[Fe(CO)_3(C_7H_7.CH{:}CHPh)]$ gave migration of the $[Fe(CO)_3]$ unit from the arene position to a diene configuration using one <u>endo-</u> and one <u>exo-</u>cyclic double bond [1072]. An $[Fe(CO)_3]$ complex of a bicyclododecadiene has been prepared [1073]. Cyclododecatriene and $[Fe(CO)_5]$ react to form a complex which reacts with Ph_3C^+ to give a trienyl salt [1074]. The reaction of diaminodihydracepentalenes with $[Fe_2(CO)_9]$ gives diene complexes which can be reduced electrochemically or with Na to anionic monoene complexes [1075].

IR spectra have been used to study the tautomerism of the newly prepared $[Fe(CO)_4(\eta^2\text{-}PhCH{:}CH.CR{:}NR')]$, with a σ-N donor, with an $[Fe(CO)_3]$ configuration involving η^3-carbarmoylallyl or η^4-azadiene coordination [1076].

The remaining complexes in this section are dinuclear. The chemistry of the $[Fe_2(CO)_7]$ complex of the phosphole 2,3-Me_2-1-Ph-C_4H_2P, which shows both diene and phosphine coordination is reported; $AlCl_3$ and NH_4OH remove the iron unit, reactions at P are achieved with H_2O_2, S_8, MeI and $PhCH_2Br$, reaction with PhCOCl, Et_3N and H_2O give the six-membered ring $Ph\overline{P(O).CH{:}CMe.CMe{:}CH.C}(Ph)OH$ coordinated as a diene to Fe; $[PdCl_2(NCMe)_2]$ converts it to a bimetallic complex [1077]. Deprotonation of $[Fe_2(CO)_6(PPh_2H)]^-$ to $[Fe_2(CO)_6(PPh_2)_2]^{2-}$ followed by reaction with allyliodide gives (X-ray) $[Fe_2(CO)_5(PPh_2)(PPh_2C_3H_5)(\eta^3\text{-}C_3H_5)]$ [1078]. An $[Fe_2(CO)_6]$ complex of the diene

MeC:C(NEt$_2$).C(NEt$_2$):CMe is formed when [Fe$_3$(CO)$_{12}$] reacts with Et$_2$NC:CMe [1079]. An allene-type complex is formed by refluxing in hexane [Fe$_2$(CO)$_6$(μ-COEt)(μ-CPhCHPh)], prepared from [Fe$_2$(CO)$_4$(μ-CPhCHPh)] by reaction with Et$_3$O$^+$BF$_4$$^-$ [1079]. The alkyne HOCPhRC:C.CPhR'OH reacts with [Fe$_3$(CO)$_{12}$] to give [Fe$_2$(CO)$_6$(μ-PhRC:C.C:CPhR)] with each double bond binding different Fe's [1080].

7.7. Cyclopentadienyl and Related Compounds

This very large survey begins with ferrocene and other compounds containing two π-bonded cyclic ligands. A new preparation of ferrocene uses controlled potential electrolysis of TlCp with an iron anode [1081]. The new unstable 19 - electron complex [Fe(cp)(η^6-fluorene)], prepared from the Fe(II) cation, is a good intermediate for reactions of fluorene [1082]. The preparation and X-ray structure of the ferrocene analogue of isodicyclopentadienide are reported [1083]. New complexes [Fe(cp)(C$_5$H$_4$R)], (R = NO,NO$_2$) are prepared by the action of H$_2$O$_2$ on the product, [Cr(cp)(NO)(CN^{t}Bu){Fe(cp)(C$_5$H$_4$NO)}], from the reaction of [Fe(cp)(C$_5$H$_4$Li)] with [Cr(cp)(NO)$_2$Cl] and ^{t}BuNC; the complexes react with CH$_2$(CN)$_2$ or PEt$_3$ to give complexes with R = N:C(CN)$_2$ or N:PEt$_3$ [1084]. New tropone (L) complexes [Fe(cp)L]$^+$ are prepared by ring expansion using CH$_2$N$_2$ on the products of e.g. [Fe(cp)(C$_6$H$_5$Cl)]$^+$ with NaOH or NaSH [1085].

There have been many other reports of new arene - cyclopentadienyl complexes, including examples with di- or tetrahydroanthracene which have been prepared by the reaction of anthracene with [Fe(C$_5$Me$_5$)(CO)$_2$X], (X = Br,Cl) [1086]. Xanthene or thioxanthene react with ferrocene to give new η^6 - complexes with the loss of one cp ligand; subsequent stereospecific reactions occur on the new complexed ligand [1087].

Many new compounds contain hetero-atoms in the ligand. Using an Fe anode in the controlled potential electrolysis of carbaborane anions gave salts of [Fe(C$_2$B$_9$H$_{11}$)$_2$]$^-$ [1088]. Iron atoms react with B$_{10}$H$_{14}$ and toluene or mesitylene to form arene ferraboranes with 8, 9 or 10 boron atoms; one of the products, arising from an impurity reaction, was the first polyhedral boron compound to contain oxygen at a cluster vertex [1089]. [Fe(cp)(1-phenyl-3,4-dimethylphosphole] is prepared by the reaction of the phosphole with $[Fe_2(cp)_2(CO)_4]$ [1090].

Now for crystal structures and other physical studies. 1,1',2,2'-Tetrachloroferrocene has three crystal phases; the low temperature phase has the rings eclipsed but the others are more disordered [1091]. Ab initio MO calculations show anomalously large quadrupole splittings in polychlorinated ferrocene due to less electron donation to iron as the ring becomes more distant from the metal [1092]. The PE spectra of ferrocene and bis(pentadienyl) iron are compared [1093]. PE spectra are combined with EHT and Xα calculations for $[Fe(C_4H_4P_2]$ [1094]. Intervalence transitions are described for many polyferrocene complexes and correlated with the presence of π- and σ- bonding [1095].

Studies of Moessbauer spectra include the observation of changes in the quadrupole splitting of substituted phosphaferrocenes when a phenyl substituent is protonated by CF_3SO_3H to give a σ-bonded species [1096]. Vibrational assignments and the splitting of the two lowest Kramers doublets in $[Fe(C_5D_5)]^+$ were established by inelastic neutron scattering spectroscopy at 10K; the analysis includes non-axial ligand fields, spin-orbit coupling and the dynamic Jahn-Teller effect [1097]. For $[Fe(cp)(C_6Me_6)]^{n+}$, (n=0,1), the extra electron in the reduced compound was shown to spend 83% of its time on the iron atom [1098]. Moessbauer and NMR (^1H) spectra of $[Fe(cp)\{C_5H_4CH(Ar)\}]^+$ salts [1099]. Spectra of $[XC_5H_4FeC_5H_4.C_5H_4FeC_5H_4X]^+$ show delocalisation for X = Br or I but localisation for Cl, despite similar redox potentials [1100]. For triiodide salts of mixed valence biferrocenes spectra were taken in magnetic fields to assign the unpaired electron's orbital; the problem is complicated by spin-lattice relaxation effects [1101]. There is a linear relationship between quadrupole splitting and redox potentials in some substituted ferrocenes [1102].

Gas phase thermal decomposition gives 381 ± $12kJmol^{-1}$ for the first bond dissociation energy of ferrocene [1103]. The ionisation of $[Fe(cp)\{C_5H_4CH(OH)C_6H_4R\}]$ has been studied [1104]. A mechanistic study of the photochemical loss of arene from $[Fe(cp)(arene)]^+$ includes the effects of steric interactions on the arene which may prevent the solvent or anions participating in the transition state [1105]. The reduction of ferrocene and related compounds has been reviewed [1106].

Cyclic voltammetry has been widely applied to ferrocene derivatives; subjects this year have included $[Fe(RCOC_5H_4)_2]$ [1107] and $[Fe(cp)(C_5H_4.C_6H_4R)]$ [1108]. Ferrocenes including C_4H_4P and $Me_2C_4H_2P$ rings can lose an electron from Fe, but loss of a second electron gives irreversible oxidation of the ligand [1109]. $[Fe(cp)(cyclophane)]^+$ and $[Fe_2(cp)_2(cyclophane)]^{2+}$ are compared with corresponding arene complexes; two distinct steps are found in the reduction of the diiron complexes [1110].

In chemical reactions which are not purely organic in character, the oxidation of ferrocene to $[Fe(C_5H_4R)(C_5H_4R')]^+ I_3^-$ is described [1111]. Ferrocene or ferricenium sulphate react with Cl_3CCO_2H to give that ferricenium salt with solvated acid [1112]. Ferrocenes carrying one or two crown ether ligands linked to cp by CH_2SCH_2 units have been synthesised and found to complex alkali metals [1113]. $[Fe(cp)(arene)]^+$ salts of $B_{10}H_{10}{}^{2-}$, $B_{11}H_{14}{}^{2-}$ and $B_{12}H_{12}{}^{2-}$ have been prepared [1114]. Ferrocene reacts with $FeCl_3$ to give $2[Fe(cp)_2]^+ [Fe_2OCl_6]^{2-}$ and $[Fe(cp)_2]^+ [FeCl_4]^-$, but not the previously reported $[FeCl_3]^{2-}$ [1115]. Measurement of $J(^{57}Fe-^{13}C)$ in "$[Fe(cp)_2(CRR')]$", [R = H, Me; R' = H, Me, Ph, $Mn(CO)_3cp$, $Ru(cp)_3$, $Fe(cp)(C_5H_4)$], show these really have bulvalene type bonding [1116].

The next section concerns complexes of ferrocenes which contain other metals in addition to iron. We work from left to right in the usual representation of the periodic table to order the heterometal. Yb displaces Hg from $[Hg(C_5H_4Fecp)_2]$ [1117]. Reacting $[Fe(C_5H_4SeOH)_2]$ with $[M(C_5H_4R)_2Cl_2]$, (M = Zr, Hf; R = H, ${}^{t}Bu$) gives $[Fe(C_5H_4Se)_2M(C_5H_4R)_2]$ [1118]. A Zr-H moiety adds across CO when $[Fe(cp)(CO)H(PMe_3)]$ reacts with $[Zr(C_5Me_5)H_2]$ and PMe_3 to give $[Fe(cp)(PMe_3)_2CH_2OZrH(C_5Me_5)_2]$ [1119]. In a new vanadium complex two $[V(C_5H_4R)]$ units are linked to one ferrocene component by V-C bonds so that the three sandwich fragments are perpendicular; the result is that the cp rings distort towards η^4-geometry [1120]. Chiral $[Fe(cp)(CO)(PMe_3)(EMe_2)]$, (E = As, Sb, Bi) react with $[Mo(CO)_4(norbornadiene)]$ to give diastereomeric $[Fe(cp)(CO)(PMe_3)(EMe_2)_2Mo(CO)_4]$ [1121]. The preparation and X-ray structure of $[Fe(cp)C_5H_4CH{:}CHSi(Me)(Ph)C_5H_4Mn(CO)_3]$ are reported [1122]. Many complexes with Ru(II) and various zerovalent metals are described with $[Fe(C_4H_2Me_2P_2)_2]$, (L), in which one or both P atoms coordinate; in $[Ru_2Cl_4(PPh_3)_2L_2]$ the C_4P rings are still aromatic as the ligands bridge but the P atoms are eclipsed, which is not the case for uncomplexed L [1123]. The C_7H_7 ring in $[Fe(CO)_3(C_7H_7)Rh(CO)_2]$ is η^3 to Fe and η^4 to Rh, but if the carbonyls are replaced by dppe the bonding becomes η^3 to Rh and η^4 to Fe [1124]. $[NiCl_2(Ph_2PC_5H_4)_2Fe]$ catalyses the stereoselective silylation of crotylmagnesium bromide [1125]. Ferrocenes with a Pd-cp σ-bond and a cyclometallated Pd-π-cp linkage are described [1126]. $[Fe(cp)\{C_5H_4(AuPPh_3)\}]^+$ is described [1127]. Moessbauer and ${}^{199}Hg$ NMR spectra of $[Fe(cp)(CO)_2HgX]$, (X = halide or bidentate S ligand), show the S ligand forces more electron density on to the Fe atom; the effect is even greater in $[Hg\{Fe(cp)(CO)_2\}_2]$[1128]. Moessbauer spectra suggest an Hg-Fe bond is present in the compounds formed when Hg(II) compounds react with ferrocenophanes [1129]. Ferricinium ions oxidise $[Fe_2(C_5H_4HgC_5H_4CH_2NMe_2)_2]$ to the mono- and dications [1130].

$[Fe(C_5H_4COCH_2COMe)_2]$ forms diketonate complexes with other metals [1131].

Now we come to cyclopentadienyl compounds containing carbonyl ligands. The order is monomers before polymers and in decreasing number of carbonyls. $[Fe(cp)(CO)_3]^+$ reacts with diamines to give $[\{Fe(cp)(CO)_2CONR\}_2(CH_2)_n]$, $[Fe(cp)(CO)_2CONHCH_2CH_2NMe_2]$ and $[Fe(cp)(CO)_2(CONHCH_2CMe_2NH_3)]^+$; the latter can be deprotonated and react with the starting material to give $[Fe(cp)(CO)_2CONHCH_2CMe_2NHCOFe(cp)(CO)_2]$ [1132]. Products formed by reactions which start with $[Fe(C_5Me_5)(CO)_3]^+$ include $[Fe(C_5Me_5)(CO)_2(PR_3)]$, (R = Me,Ph), $[Fe(C_5Me_5)(CO)(dppe)]$, $[Fe(C_5Me_5)(CO)H_2dppe]$, $[Fe(C_5Me_5)(CO)_2H]$ and $[Fe(C_5Me_5)(CO)(PMe_3)Me]$ [1133].

An interesting transformation occurs when SO_2 reacts with $[Fe(cp)(CO)_2(PbPh_3)]$ to give $[Fe(cp)(CO)_2(SO_2Ph)]$[1134]. Various $[Fe(cp)(CO)_2L]$ complexes, (L = alkene, alkyne, allene) react with [(cyclotropilium $Fe(CO)_3]^+$ salts to give oxohydroazulene complexes, with the $[Fe(cp)(CO)_2]$ moiety connected to the five-membered ring [1135]. A short review on $[Fe(cp)(CO)_2H]$ has appeared [1136]. $[Fe(cp)(CO)_2(EPh_3)]$, (E = Si, Ge, Sn) can be reversibly reduced electrochemically to its monoanion, in which the unpaired electron is located in a phenyl π-system; further electrochemical reduction gives decomposition to $[Fe(cp)(CO)_2]^-$ and Ph_3EH or $SnPh_3^-$ [1137]. $[Fe(cp)(CO)_2SiMe_2SiPh_3]$ is prepared by the reaction of $ClSiMe_2SiPh_3$ with $Na[Fe(cp)(CO)_2]$[1138]. Acetic acid converts $Na[Fe(C_5Me_5)(CO)_2]$ to $[Fe(C_5Me_5)(CO)_2H]$; one of these carbonyl groups can be substituted by PMe_3 and further reaction with $Me_3P{:}CH_2$ gives $[Fe(C_5Me_5)(CO)(PMe_3)Me]$[1139]. $[Fe(cp)(CO)_2Cl]$ is formed from SO_2Cl_2 and $[Fe(cp)(CO)_2]_2$ in CH_2Cl_2, but in C_6H_6 this reaction also gives $[Fe(cp)(CO)_3]^+[FeCl_4]^-$[1140].

$Na[Fe(cp)(CO)_2]$ reacts with $ClC(O)CCHB_{10}H_{10}$ to give a carbaborane complex which loses CO to give $[Fe(CCHB_{10}H_{10})(cp)(CO)_2]$[1141]. $H_2C{:}C(OEt)CO_2Et$ acts as an alkene ligand (L) in $[Fe(cp)(CO)_2L]^+$ for reactions to prepare lactones [1142]. $[Fe(C_5Me_5)(CO)_2CH_2OH]$ is formed from $[Fe(C_5Me_5)(CO)_3]^+$ and $[Fe(C_5Me_5)(CO)_2]$ or $[Mo(dppe)_2H_4]$ [1143]. $[Fe(C_5Me_5)(CO)_2Br]$ can be converted to $[Fe(C_5Me_5(CO)_3]^+$ with CO and $AlCl_3$ or with $[Fe(cp)_2]^+$; the latter reacts with $[Fe(C_5Me_5)(CO)_2]_2$ in THF to give $[Fe(C_5Me_5)(CO)_2(THF)]^+$, from which the THF can be substituted to give many cationic or neutral complexes [1144, 1145]. Various $[Fe(C_5H_4CO_2H)(CO)_2R]$, [R = Ph, CH_2Ph, $C_5H_4Mn(CO)_3$], have been prepared; all are stronger acids than ferrocenecarboxylicacid [1146]. The complexes $[Fe(cp)(CO)_2AsR_2]$, (R = Me, ^{t}Bu) and their C_5Me_5 analogues are prepared from $[Fe(cp)(CO)_2]^-$; the products of their reactions with MeI, $P(OMe)_3$ and S are described [1147]. (Mesityl)PCl_2 converts $[Fe(C_5Me_5)(CO)_2]^-$ to $[Fe(C_5Me_5)(CO)_2]_2$, but $^iPr_2NPCl_2$ gives $[Fe(C_5Me_5)(CO)_2(R_2NPCl)]$, whose reduction by $LiEt_3BH$ gives $[Fe(C_5Me_5)(CO)_2(RNPH)]$, which in turn reacts with Ph_3C^+ or $AlCl_3$ to give $[Fe(C_5Me_5)(CO)_2\{PHNRCMeCH_2\}]^+$, which may be formed via an N-P-Fe heteroallyl intermediate [1148]. Thiophosphinite complexes $[Fe(cp)(CO)_2\{P(S)R_2\}]$, (R = alkyl, aryl) are prepared from $[Fe(cp)(CO)_2Br]$, R_2HPS and NEt_3 [1149]. Electrochemical reduction of $[Fe(cp)(CO)_2SnR_3]$ and other tin complexes gave new distannanes carrying $[Fe(cp)(CO)_2]$ groups [1150]. Norbornyl (X) complexes $[Fe(cp)(CO)_2X]$ have been prepared [1151]. $[Fe(cp)(CO)_2\ (CS_2)]^-$ replaces Cl in Me_3ECl, (E = Si,Sn) or I in $[Ru(cp)(CO)_2I]$; the Ru complex and related $[Re(CO)_5]$ and $[Fe(cp)(CO)_2]$ species are alkylated at the thione S and the reactions of those products are also described [1152].

There are not so many articles about complexes which contain an $[Fe(cp)(CO)]$ unit. $[Fe(cp)(CO)(Et)L]$, $\{L = PPh_3, P(OCH_2)_3CMe\}$, react with CO with or without $C_6H_{11}NC$ to give $[Fe(cp)LL'(COEt)]$, (L' = CO, $C_6H_{11}NC$); the reaction is stereospecific in nitroalkanes [1153]. BuLi reacts with $[\mathrm{Fe(cp)(CO)(PPh_3)(COMe)}]$ to form $[\mathrm{Fe(cp)(CO)(PPh_3)COCH_2}]^-$, which can be alkylated to give $[\mathrm{Fe(cp)(CO)(PPh_3)COR}]$ or $[\mathrm{Fe(cp)(CO)(PPh_3)COCH_2CRR'OH}]$ [1154]. $[\mathrm{Fe(cp)(dppe)(CO)}]^+$ is reduced by $\mathrm{LiAlH_4}$ to $[\mathrm{Fe(cp)(dppe)(CO)H}]$ (A) at -78 $^{\circ}$C via a formyl intermediate but refluxing in THF gives $[\mathrm{Fe(\eta^4\text{-}C_5H_6)(dppe)(CO)}]$ and $[\mathrm{Fe(cp)(dppe)Me}]$ (B); (A) decomposes to $[\mathrm{Fe(cp)(CO)H}]_2(\mu\text{-dppe})$ in toluene but to (B) in THF [1155]. A similar starting material containing dppp instead of dppe gives $[\mathrm{Fe(cp)(dppp)(CO)H}]$, with monodentate dppp, but dmpe gives $[\mathrm{Fe(cp)(dmpe)_2Me}]$ [1156]. Similar chemistry is reported for the formation of $[\mathrm{Fe(C_5Me_5)(dppe)(CO)H}]$ [1157]. A more complex new compound of this type is $[\overline{\mathrm{FeC_5H_4PPh_2\{Fe(cp)(CO)\}C(O)C}}\mathrm{_5H_4}]$ [1158]. Reacting $\mathrm{Me_3P{:}CH_2}$ with $[\mathrm{Fe(cp)(CO)_n(RNC)_{3-n}}]^+$ gives ylide complexes whose reactivity was investigated, with mechanistic discussions [1159]. Electronic spectra were used to study solution equilibria between monomeric $[\mathrm{Fe(cp)(CO)(SPh)\{P(OR)_3\}}]^+$, (R = Et,Ph) or $[\mathrm{Fe(cp)(CNC_6H_4R')_2(SPh)}]^+$, (R' = $\mathrm{OCH_3}$, $\mathrm{CF_3}$) and dimers with PhSSPh ligands; electron withdrawing groups favour dimers [1160]. The X-ray structures of $[\mathrm{Fe(cp)(CO)\{C_5H_5Mn(CO)_3\}L}]$, (L = CO,$\mathrm{PPh_3}$), confirm that iron has one σ- and one π-bonded cp ligand [1161]. Paramagnetic $[\mathrm{(C_{10}H_8)_2Fe(CO)}]^+$ salts have been prepared [1162]. Other new compounds reported are $[\mathrm{Fe(cp)(CO){:}CRPPh_3}]$, (R = H,Me)[1163], $[\mathrm{Fe(cp)(CO)L(CO{}^{\mathit{i}}Bu)}]$, (L = $\mathrm{PPh_3}$, $\mathrm{PPh_2NEt_2}$) [1164], various phosphine complexes $[\mathrm{Fe(cp)(CO)L(PhSEt)}]^+$[1165] and $[\mathrm{Fe(C_5Me_5)(PMe_3)_l(CO)_m(NCMe)_n}]$, (l+m+n=3) [1166].

Turning now to polynuclear compounds, $\mathrm{GeF_2}$ reacts with $[\mathrm{Fe(cp)(CO)_2R}]$, $\{\mathrm{R = Mn(CO)_5,\ Mo(cp)(CO)_3}\}$, to give $\mathrm{F_2Ge[Fe(cp)(CO)_2]_2}$, but reaction with R = $\mathrm{SiPh_3}$ gives polymers [1167]. $[\mathrm{Fe(cp)(CO)_2}]_2$ and its $\mathrm{C_5Me_5}$ and $\mathrm{C_5H_4CH_2Ph}$ analogues, when photolysed at 77K in organic matrices, give $[\mathrm{Fe_2(cp)_2(CO)_3}]$ which have a triple CO bridge and will be analogues of $[\mathrm{Fe_2(cp)(NO)_2}]$ with an Fe Fe bond. [1168, 1169]. The new compound

$[Fe_2(CO)_4(C_5H_4SiMe_2C_2H_4SiMe_2C_5H_4)]$ has two bridging carbonyls as well as a ferrocenophane bridge [1170]. New silicon containing complexes include $[Fe(cp)L_2(SiR_3)]$, ($L = CO, PMe_3$; $R = H, NMe_2$), some of which form polynuclear complexes on reaction with $[Co_2(CO)_8]$[1171,1172], and polymeric $[OSi(Me)\{Fe(cp)(CO)_2\}]$[1173]. Studies on $[Fe(cp)(CO)_2]_2$ and related compounds include cyclic voltammetry [1174] and Moessbauer and ir spectra [1175]. Alkenes and cycloalkanes can be prepared from such as $[Fe(cp)(CO)_2(CH_2)_nFe(cp)(CO)_2]$[1176]. When $[Fe(cp)(CO)_2]_2$ is used in the hydroformylation of propene or 1-pentene, $[Fe(cp)(CO)_2H]$ was detected [1177]. Interesting fluxional behaviour is found in the structures of $[Fe_2(cp)_2(CO)_2(CS)(CNR)]$; ($R = Me, Pr$) [1178].

The structure of $[\{Fe(cp)(CO)\}_2(SEt)_2]^+ClO_3^-$ has been determined [1179]. $FeCl_3$ only oxidises one Fe in $[\{Fe(cp)(C_5H_4)\}_2CO]$; the paper includes a discussion of Moessbauer quadrupole splittings in diferrocenyl monocations [1180].

Many complexes have been prepared which contain other ligands instead of CO on cyclopentadienyl iron units. Poly (1,1'-ferrocenylene) adducts with I_2, TCNQ or TCNE show partial oxidation with very high conductance; the TCNQ complex is the best characterised [1181]. $[Fe(cp)\{P(OMe)_3\}_3]^+$ has been prepared by controlled potential electrolysis of $[Fe(cp)(C_6H_nMe_{6-n})]^+$ with added $P(OMe)_3$ [1182]. $O\overline{P(Ph)CHC(Me)C(Me)CHC}Ph$ behaves as an η^4-ligand to an [Fe(cp)] moiety with the P atom folded away from Fe [1183]. Crown ethers containing two ferrocene units have been prepared and found to coordinate Ag^+ with possibly some electron transfer from Fe to Ag [1184]. [Fe(cp)(dppe)(CN)] has been prepared from the Cl analogue [1185]. Acetylferrocene has been used to prepare $[Fe(cp)\{C_5H_4C(Cl){:}CHCHO\}]$, whose reactions were studied [1186]. The carbene complex $[Fe(cp)\{C(Et)OMe\}CO(PPh_3)]^+$ and its stereoselective addition reactions have been described [1187]. Reaction of N_2CHCHO with $[Fe(cp)(NO)]_2$ gives addition across the

Fe$=$Fe bond to form $[\{Fe(cp)(NO)\}_2CHCHO]$[1188].

There have been very many studies on cyclopentadienyl complexes, usually ferrocenyl, containing substituents on the cp rings. These include X-ray structures of ferrocenyl pyrazoles [1189] and of 1,1',2,2'-tetrachloroferrocene [1190], ^{13}C NMR of ferrocenyl ketones [1191], ir studies on these [1192], vibronic coupling in biferrocene and bis(fulvalene) diiron cations [1193] and Moessbauer spectra of ferrocenyl dimers [1194]. However, most work on substituted ferrocene complexes can be described as organic synthesis. Work of this type has been reviewed at length [1195]. It is impossible to do justice to this field in the available time and space; interested readers should consult the bibliography [1196-1229].

Ferrocenophanes form a special class. Most of the work on these has also been synthetic but the work has been supported by X-ray structures [1230, 1231], NMR spectra [1232, 1233], Moessbauer spectra [1234, 1235], electrochemistry [1234,1236]. Much other work, though often ingenious, is essentially synthetic in character [1237-1247]. There remain one kinetic study [1248] and some INDO-SCF calculations on ferrocenophane reactivity [1249].

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