

27 Inorganic mechanisms

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1 Introduction

Understanding the factors which control the mechanisms of inorganic and organometallic redox and substitution reactions in solution is important environmentally, biologically, industrially as well as intrinsically. The literature on these topics for 1996, subject to the exclusions noted in the 1995 report, is organised as before.

Espenson¹ and Atwood² have updated their texts on kinetics and mechanisms. Texts³⁻⁶ and a volume of *Chem. Rev.*⁷ contain surveys of mechanistic aspects of the biological roles of metals. The increasing contribution made by computational methods to the study of mechanisms has been reviewed.^{8,9} A program which corrects for concentration gradients in stopped-flow kinetic observations on second-order processes with rate constants up to $10^8 \text{ mol l}^{-1} \text{ s}^{-1}$ has been tested¹⁰ experimentally. The design of a stopped-flow instrument for the simultaneous monitoring of absorbance and fluorescence up to 200 MPa has been described.¹¹ Short-lived intermediates, such as five-co-ordinate base-off methylcobalamin, have been characterised by time-resolved X-ray absorption spectroscopy.¹²

2 Redox reactions

Marcus¹³ has reviewed electron-transfer reactions in chemistry in a volume on protein electron transfer.⁶ Current challenges in electron-transfer mechanisms have also been addressed.¹⁴

Long range electron transfer

Biological electron-transfer has been reviewed.¹⁵⁻²³ McLendon and co-workers²⁴ have shown that Marcus theory can be applied to *in vivo* reactions of cytochrome (cyt) c. Intramolecular electron transfer between the Cys3-Cys23 radical ion and Cu^{II} in wild-type and single-site mutated azurins²⁵ occurs *via* a through-bond mechanism. The pH dependence of these processes has also been reported.²⁶ The effect of the intervening medium on electron transfer in Ru-modified azurin, cyt c and myoglobin has been studied.²⁷ Photoinduced electron-transfer rates for a series of diimine-Ru^{II} complexes with Fe^{III}(cyt c) with $\Delta G^\circ < -1.12 \text{ V}$ decrease with increasing exergonicity²⁸ and fit a bimolecular model for electron transfer over an equilibrium distribution of reactant

separations each with a different formation probability. The driving-force dependence of the reduction of $\text{Fe}^{\text{III}}(\text{cyt c})$, modified at His-33 by a series of ruthenium moieties, reveals²⁹ that the most exergonic reductions have rates which are much higher than expected for processes leading directly to ground-state products. It is suggested²⁹ that an electronically-excited ferrohaem is the initial product. Rates of oxidation of $\text{Fe}^{\text{II}}(\text{cyt c})$ by $[\text{Fe}^{\text{III}}(\text{CN})_5(\text{Rpy})]^{\text{2-}}$ depend on the effect of R on the stability of the precursor complex to electron transfer.³⁰ The rates of electron transfer between metHb^{III} and cyt b₅^{II} deviate from linear Arrhenius dependence³¹ because of specific interactions between the two proteins. Other long-range electron-transfer processes within protein–protein complexes have been described.^{32–35} The very slow rates of electron self-exchange between wild-type *Pyrococcus furiosus* 4-Fe ferredoxin, compared with two Asp-14-Cys and Asp-14-Ser mutants, are ascribed³⁶ to Asp ligation to the 4-Fe cluster. Positively charged residues near to the electron-transfer site account³⁷ for the rate of electron self-exchange for the blue copper protein, umecyanin, being among the lowest known for this class of protein. Electron self-exchange has also been studied³⁸ for *Clostridium pasteurianum* rubredoxins. Luminescence quenching of $[\text{Ru}^*(\text{bipy})_3]^{\text{2+}}$ by a copper site in sub-unit II of *Thermus thermophilus* cytochrome ba₃ is observed³⁹ at low protein concentrations. At higher concentrations, quenching is saturated as a result of ground-state complexation. Reduction of *Pseudomonas aeruginosa* cyt cd₁ nitrite reductase by $[\text{Fe}(\text{edta})]^{\text{2-}}$ leads first to reduction of haem c followed by a very slow first-order intramolecular reduction of haem d₁, one of the slowest natural electron transfers recorded in biological systems, ascribed⁴⁰ to unfavourable orientation effects and/or large separations. Slow intramolecular electron transfer is also seen⁴¹ for the reaction between a bacterial dihaem protein and $[\text{Co}(\text{bipy})_3]^{\text{3+}/\text{2+}}$. Increased ΔG^\ddagger for electron transfer in sperm-whale myoglobin modified at the distal histidine residue (His-64) is a result of hydrogen-bonding effects in the distal pocket.⁴² Rates of intramolecular electron transfer in cyt b₅ covalently modified with Ru^{II}–polypyridine moieties at Cys-73 or Cys-65, with *Ru^{II} and Fe^{III} separated by a well defined 12 covalent-bond link, show⁴³ either zero or slightly negative ΔV^\ddagger . Cobalt(II) to Ru^{III} intramolecular electron-transfer rates have been measured⁴⁴ for a cobaltocytochrome c modified at His-33 by Ru(NH₃)₅. Reversible outer-sphere electron-transfer rates for $[\text{Ru}(\text{NH}_3)_5\text{L}]^{\text{3+}/\text{2+}}$ (L = substituted py) and cyt c vary⁴⁵ with the ability of L to penetrate the haem groove on cytochrome c. Sykes and co-workers^{46–49} have studied the kinetics of the reduction of the R2 protein of *E. coli* ribonucleotide reductase with $[\text{Co}(\text{sep})]^{\text{2+}}$ and $[\text{Co}([9]\text{aneN}_3)_2]^{\text{2+}}$ ⁴⁶ and with hydrazine,⁴⁷ as well as of the reaction of the oxidised form of rubredoxin from *Clostridium pasteurianum* with $[\text{Co}(\text{sep})]^{\text{2+}}$ and its reduced form with $[\text{Co}(\text{terpy})_2]^{\text{3+}}$ and $[\text{Ru}(\text{NH}_3)_6]^{\text{3+}}$.⁴⁸ Electrostatic effects in the reactions of [2Fe-2S] ferredoxins and a series of cobalt complexes have been reported.⁵⁰ Circular dichroism has been used⁵¹ to investigate the specific sites of reaction of plastocyanin with optically-active Co^{II} complexes. The pH dependence of the oxidation of ferric microperoxidase-8 by photo-generated $[\text{Ru}(\text{bipy})_3]^{\text{3+}}$ suggests⁵² that the ferryl cation-radical porphyrin is formed by intramolecular electron transfer within a ferric cation-radical porphyrin. Reaction with cysteine has also been studied.⁵³ Electron transfer in *de novo*-designed metalloproteins has been described.⁵⁴

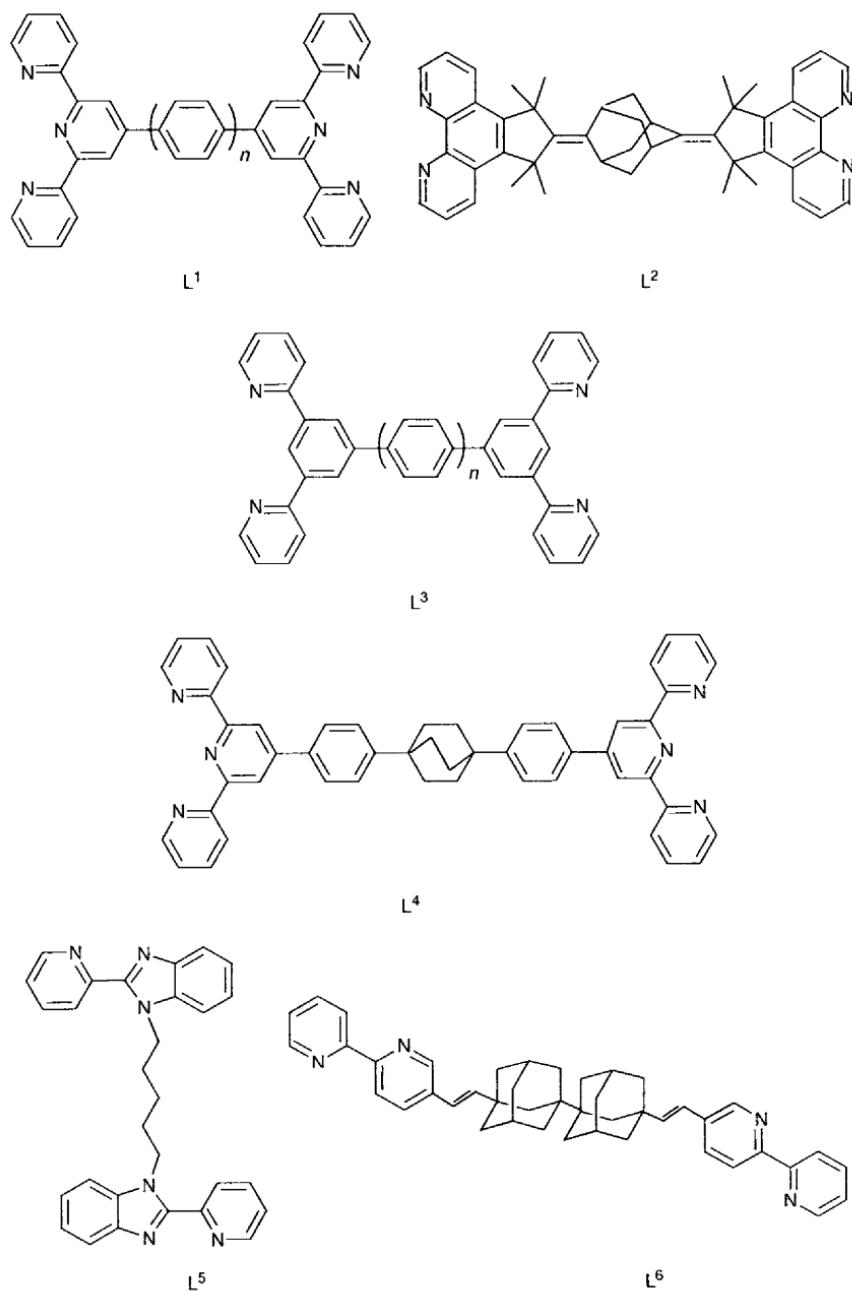
A new theoretical method⁵⁵ for describing tunnelling processes in long-range multiple-pathway electron transfer in proteins and an approach designed to permit analysis of such processes in terms of protein secondary and tertiary structure⁵⁶ have

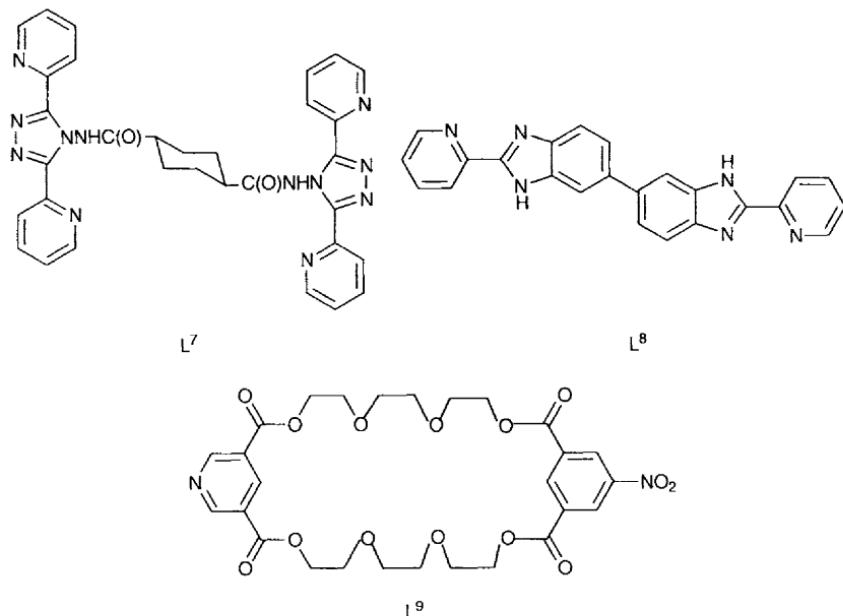
been developed. Marcus and co-workers⁵⁷ report procedures designed to reduce the computational complexity associated with an extended Hückel theory analysis of electron tunnelling in proteins by selecting for the calculation only those amino acids that are important to electron transfer. The validity of a multiple-scattering approach to electron coupling has been debated.^{58,59} Aspects of DNA-mediated electron transfer^{15,60–65} and oxidative DNA and mRNA cleavage^{66–75} have been studied mechanistically. In a series of mixed-valence diferrocenylpolyenes, $[\text{Fc}(\text{CH}=\text{CH})_n\text{Fc}]^+$ ($n = 1–6$), metal–metal coupling falls off exponentially⁷⁶ with separation, with an exponent of 0.087 \AA^{-1} , one of the smallest attenuations reported. Photoinduced electron- and energy-transfer have been studied in $[(\text{terpy})\text{Ru}^{\text{II}}(\mu-\text{L}^1)\text{Rh}^{\text{III}}(\text{terpy})]^{5+}$,⁷⁷ $[(\text{bipy})_2\text{Ru}(\mu-\text{L}^2)\text{Os}(\text{bipy})_2]^{5+}$,⁷⁸ in $[(\text{terpy})\text{Ru}^{\text{II}}(\mu-\text{L})\text{Os}^{\text{II}}(\text{terpy})]^{2+}$,⁷⁹ linked by rigid spacers, $\text{L} = \text{L}^3$ (with metal–metal separations of 11, 15.5 and 20 Å for $n = 0, 1$ and 2, respectively) or L^4 ,⁸⁰ in $[(\text{bipy})_2\text{Ru}^{\text{II}}(\mu-\text{L}^5)\text{Co}^{\text{III}}(\text{bipy})_2]^{5+}$,⁸¹ in $[(\text{bipy})_2\text{M}^{\text{II}}(\mu-\text{L}^6)\text{M}'^{\text{II}}(\text{bipy})_2]^{4+}$ ($\text{M}, \text{M}' = \text{Ru or Os}$),⁸² and $[(\text{bipy})_2\text{Ru}^{\text{II}}(\mu-\text{L}^7)\text{Ru}^{\text{III}}(\text{bipy})_2]^{5+}$,⁸³ and $[(\text{bipy})_2\text{Ru}^{\text{II}}(\mu-\text{L}^8)\text{Ru}^{\text{III}}(\text{decibipy})_2]^{5+}$.⁸⁴ Related systems⁸⁵ involving other covalent assemblies including diads and triads containing metalloporphyrin units have been studied further, particularly as photosynthetic models.^{86–94} Rates of long-range electron transfer through an aryl ether dendrimer framework have also been reported.⁹⁵

Intramolecular and intervalence electron transfer

Further investigations have been undertaken of transients formed in the electronic excitation of various *fac*-[$\text{Re}^{\text{I}}(\text{CO})_3\text{L}(\alpha\text{-diimine})]$ ⁺ **1**, *fac*-[$\text{Re}^{\text{I}}\text{X}(\text{CO})_3(\alpha\text{-diimine})$] and related Mn and Ru complexes.^{96–109} Meyer and co-workers¹⁰³ have shown that (for **1**, $\text{L} = \text{afa}^{2-}$) the rates for $\text{afa}^{3-} \rightarrow \text{Re}^{\text{II}}$ and (for **1**, $\text{L} = \text{oqd}$) $\text{oqd}^{2-} \rightarrow \text{Re}^{\text{II}}$ electron-transfer (k_{ET}) are remarkably slow, with k_{ET}^{-1} being 5.6 and 15 μs, respectively. These long lifetimes are thought to arise from weak electronic coupling through the aryloxy bridges. Solvent effects have been reported¹⁰⁵ on the intramolecular folding that precedes quenching, by a nitrobenzene moiety incorporated into a polyether macrocycle, of Re-to-pyridine charge transfer in **1**, $\text{L} = \text{L}^9$. Formation and decay of alkyl radicals formed by Re–R homolysis have been studied by FT-EPR for [$\text{ReR}(\text{CO})_3(\text{dmbipy})$] and [$\text{RuR}(\text{CO})_2(\text{dmbipy})$] ($\text{R} = \text{PhCH}_2$, 2-Pr or Et).⁹⁸ Metal–metal bond fission has also been studied for [$\text{ReR}(\text{CO})_3(\alpha\text{-diimine})$] [$\text{R} = \text{Ph}_3\text{Sn}$ or $\text{Mn}(\text{CO})_5$].¹⁰⁸ Other related photochemical and photophysical studies are noted for α -diimine complexes of Ru,^{110–122} Rh,^{114,123} Ir,¹¹⁴ Os¹¹⁷ and Cu.^{124,125}

Intramolecular electron-transfer rates in valence tautomeric complexes, [$\text{Co}(3,5\text{-dtbsq})_2(\text{L})$] ($\text{L} = \text{bipy}$, phen, dmbipy or dpbipy),¹²⁶ have been reported. Related processes in analogous Re^I and Mn^I complexes,¹²⁷ in homologous [$\text{Co}(\text{L})(\text{NH}_3)_5$]²⁺ ($\text{L} = \text{trans-stilbenecarboxylate}$) complexes¹²⁸ and in homopolymetallic complexes, such as $[(\text{bipy})(\text{terpy})\text{Ru}(\text{CN})\text{Ru}(\text{NH}_3)_5]^{3+}$,¹²⁹ $[(\text{bipy})_2\text{Ru}(\text{bbbp})\text{Ru}(\text{bipy})_2]^{5+}$,¹³⁰ $[(\text{bipy})_2\text{Ru}(\mu\text{-bpt})\{\text{Ru}(\mu\text{-2,3-dpyp})\text{Ru}(\text{bipy})_2\}_2]^{7+}$ [containing both electron-rich (bpt⁻) and electron-poor (2,3-dpyp) bridging ligands],¹³¹ $[(\text{NC})\text{Ru}(\text{bipy})_2(\text{CN})\text{Ru}(\text{bipy})_2(\text{NC})\text{Ru}(\text{bipy})_2(\text{CN})\text{Ru}(\text{NH}_3)_5]^{5+}$,¹³² and CN^{-} ,^{129,133} 1-(4-cyanophenyl)imidazole-,¹³⁴ and other ligand¹³⁵-bridged Ru dimers and heteropolymetallic complexes, such as $[(\text{NC})_5\text{Fe}^{\text{III}}(\text{pyCN})\text{Ru}^{\text{II}}(\text{NH}_3)_5]$,¹³⁶ $[(\text{NC})_5\text{Fe}(\text{CN})\text{Os}(\text{NH}_3)_5]^{-}$,¹³⁷ $[(\text{NC})_5\text{Fe}(\text{CN})\{\text{Pt}(\text{NH}_3)_4\}(\text{NC})\text{Fe}(\text{CN})_5]^{4-}$,¹³⁸ $[(\text{L}(\text{CN})\text{Fe}(\text{CN})_5)]^{3-}$ ($\text{L} = \text{cobalamin}$),¹³⁹ and $[(\text{bipy})_2\text{ClOs}(4,4'\text{-bipy})\text{Ru}(\text{NH}_3)_5]^{3+/4+}$,^{140,141} have been described.

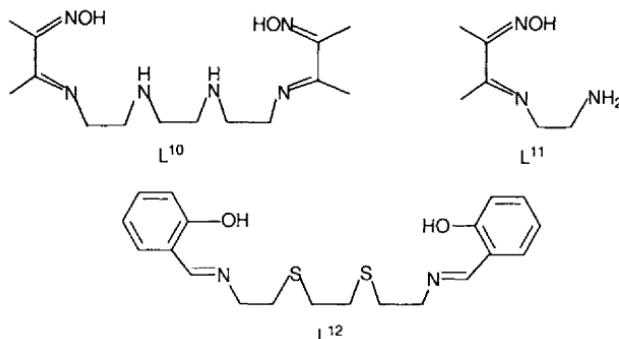




Outer-sphere electron-transfer and self-exchange reactions

Swaddle has reviewed¹⁴² the effectiveness with which theory and experiment coincide in the estimation and measurement of volumes of activation, ΔV^\ddagger , for outer-sphere electron transfer and, for aqueous systems, the use made of the departure of ΔV^\ddagger from predicted values in assessing the role of counter ions, contributions from inner-sphere processes and other phenomena. Takagi and Swaddle¹⁴³ have examined solvent effects on ΔV^\ddagger for self-exchange for the couple $[\text{Ru}\{\text{CF}_3\text{C}(\text{O})\text{CHC}(\text{O})\text{CF}_3\}_3]^{+}/0$. The ΔV^\ddagger value for the self-exchange reaction of cytochrome c is in reasonable agreement¹⁴⁴ with expectations from other observations. Values of ΔV for heterogeneous electron transfer for three couples are about half those for homogeneous bimolecular processes involving the same couples,¹⁴⁵ as expected from Marcus theory. Volume changes associated with the $[\text{ML}_6]^{3+/2+}$ couples ($\text{M} = \text{Fe}, \text{Cr}, \text{Ru}$ or Co ; $\text{L} = \text{H}_2\text{O}$ or NH_3) have also been reported.¹⁴⁶ Relevant theoretical studies^{147,148} are noted.

Outer-sphere processes have been studied, involving $[\text{Ru}^{\text{II}}(\text{NH}_3)_5\text{L}]^{2+}$ ($\text{L} = 3-$ or $4-\text{NH}_2\text{py}$), with $[\text{Co}^{\text{III}}(\text{edta})]^-$,¹⁴⁹ $[\text{Fe}(\text{phen})_3]^{2+}$ with periodate (autocatalysis disappears in excess of IO_3^-),¹⁵⁰ $[\text{Nb}(\text{CN})_8]^{5-}$ and alkaline $\text{S}_2\text{O}_8^{2-}$, BrO_3^- and IO_4^- (which reacts by two parallel pathways involving monomeric IO_4^- and the hydrated dimer $\text{I}_2\text{O}_{10}\text{H}_2^{4-}$),¹⁵¹ $[\text{Fe}(\text{CN})_6]^{4-}$ and $[\text{Co}^{\text{III}}(\text{H}_x\text{P}_y\text{O}_z)(\text{NH}_3)_5]^{(m-3)-}$, ($\text{H}_x\text{P}_y\text{O}_z^{m-}$ represents 12 different oxophosphorus anions),¹⁵² $[\text{VO}(\text{H}_2\text{O})_5]^{2+}$ and $[\text{Mn}^{\text{III}}(\text{cdta})(\text{H}_2\text{O})]^-$,¹⁵³ $\text{Ti}(\text{aq})^{3+}$ and $[\text{Co}(\text{CN})_5\text{X}]^{3-}$ ($\text{X} = \text{Cl}, \text{Br}$ or I),¹⁵⁴ and $[\text{Co}(\text{NH}_3)_5(\text{Him})]^{3+}$ and $[\text{Fe}(\text{CN})_6]^{4-}$.¹⁵⁵ Both outer-sphere and inner-sphere processes are involved in reactions of $[\text{Co}^{\text{II}}(\text{sep})]^{2+}$ with $[\text{Co}^{\text{III}}(\text{Hdmg})_2(\text{H}_2\text{O})_2]^{+}$ and $[\text{Co}^{\text{III}}(\text{dmgBF}_2)_2(\text{H}_2\text{O})_2]^{+}$.¹⁵⁶ In reactions of $\text{S}_2\text{O}_3^{2-}$ ¹⁵⁷ and N_3^- ¹⁵⁸ with $[\text{Ni}^{\text{IV}}(\text{L}^{10})]^{2+}$ or $[\text{Ni}^{\text{IV}}(\text{L}^{11})_2]^{2+}$,¹⁵⁷ Ni^{IV} to Ni^{III} reductions are outer-sphere and Ni^{III} to Ni^{II} inner-sphere. The anions $[\text{Fe}(\text{CN})_6]^{4-}$ and $[\text{Fe}^{\text{II}}(\text{CN})_5(\text{H}_2\text{O})]^{3-}$ react with *trans*



$[Co^{III}(pyca)(NO_2)(en)_2]^+$ by outer-sphere and inner-sphere processes, respectively.¹⁵⁹ Kinetics of a series of outer- and inner-sphere reductions of $[Co(L^{12})]^{3+}$ have been reported.¹⁶⁰

Electron-transfer self-exchange rates have been measured (using NMR spectroscopy) for three ruthenium ammine complexes¹⁶¹ (solvent effects are associated with solvent-solute hydrogen bonding), for the PMe₃ complex of cytochrome c¹⁶² (rates, extrapolated to infinite ionic strength, are in the order: myoglobin-PMe₃ complex < cytochrome b₅ < cytochrome c-PMe₃ < cytochrome c), $[Fe(Me_3NCH_2C_5H_4)Cp]^{2+/+}$ (encapsulated in cyclodextrin¹⁶³ or sulfonated calixarene¹⁶⁴). Self-exchange rates have also been estimated for $[Co(Hdmg)_2(H_2O)_2]^{+/-}$ and $[Co(dmgBF_2)_2(H_2O)_2]^{+/-}$,¹⁶⁵ $[ReX_2(dppee)_2]^{+/-}$ ($X = Cl$ or Br)¹⁶⁵ {in CH₂Cl₂, from studies of oxidation by $[Co^{III}(nox)_3(BBu)_2]^+$ (see also ref. 166)}, $[OsO_2([14]ane(NMe)_4)]^{2+/+}$ ¹⁶⁷ and for Fe^{II}Fe^{III}-cyclidene complexes.¹⁶⁸ Iron(II)-iron(III) self-exchange has been studied in a room-temperature melt comprising bipy-Fe complexes functionalised with oligo(ethylene glycol) groups.¹⁶⁹

Chelate-ring conformational effects on precursor assembly are revealed¹⁷⁰ from studies of stereoselectivities in the oxidation of $[Co^{II}(pn)_3]^{2+}$ by $\Delta-[Co^{III}(ox)_2(Gly)]^{2-}$ and in the association of the latter with (lel)₃- and (ob)₃- $[Co(pn)_3]^{3+}$. The ΔH^\ddagger and ΔS^\ddagger differences between $\Delta-\Delta$ and $\Delta-\Lambda$ pairs involved in the stereoselective oxidation of $[Co(en)_3]^{3+}$ and a series of chiral anionic Co^{III} complexes suggest¹⁷¹ that electron transfer proceeds by two different mechanisms depending on hydrogen bonding between the reactants. Oxidation (inner-sphere) by $\Delta-[Co(ox)_3]^{3-}$ of $[Co^{II}\{5(R,S)-Metrien\}(H_2O)_2]^{2+}$ produces¹⁷² a small enantiomeric excess of a Δ -product $[Co^{III}(ox)\{5(R,S)-Metrien\}]^+$ when the polyamine is in a *cis*- β (SS,RR) configuration and of the Λ -product from a *cis*- β (SR,RS) configuration. Enantioselective quenching of *rac*- $[Tb^{III}(pydca)_3]^{3-}$ by ferricytochrome c¹⁷³ and by Δ - and Λ - $[Ru^{II}(phen)_3]^{2+}$ ¹⁷⁴ have been described. In the latter study, ΔV^\ddagger values for identical diastereomeric processes in water and methanol are of opposite sign, believed to be associated with differences in solvation of the encounter complexes.

Inner-sphere electron and atom transfer

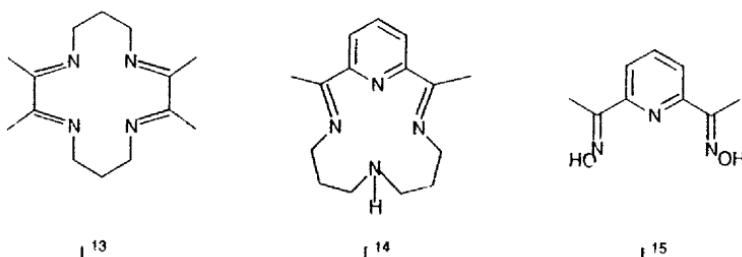
Several inner-sphere reactions associated with outer-sphere processes have already been discussed.^{156-160,172} One-electron oxidation of $[Fe(H_2O)_6]^{2+}$ by

[Cr(O₂H)(H₂O)₅]²⁺ {giving [CrO(H₂O)₅]²⁺ and [Fe(OH)(H₂O)₅]²⁺} is similar¹⁷⁵ in rate to its oxidation by H₂O₂. Acid-catalysed oxygen-atom transfer (two-electron oxidation) of I⁻ by [Cr(O₂H)(H₂O)₅]²⁺ and two *trans*-[Co(O₂H)L(H₂O)]²⁺ complexes: MO₂H²⁺ + 3I⁻ + 3H⁺ ⇌ I₃⁻ + M³⁺ + 2H₂O, are more rapid than similar processes involving H₂O₂. This arises from pK_a differences between co-ordinated M(H₂O₂)ⁿ⁺ and H₃O⁺. The anion [Co(CN)₅(O₂H)]³⁻ reacts with L-methionine to give [Co(CN)₅(L)]²⁻ (L = L-methionine-S-oxide).¹⁷⁶ Other oxygen-atom-transfer processes have been studied mechanistically, including transfers from V to S {[V^VO(O₂)L]ⁿ, L = (quin)₂, n = 3 - ; (H₂O)₂(pyca)₂, n = 0; (H₂O)₄, n = 1 + and [Co(en)₂(SCH₂CH₂NH₂)]²⁺},¹⁷⁷ from Ti to Ti {[Ti^{IV}(tpp)O] and [Ti^{III}(oep)Cl]} via two parallel pathways, one associative and one involving Cl⁻ dissociation,¹⁷⁸ from S to Mo and N to Mo {Me₂SO and py-N-O to [Mo^{VI}O(S₂PR₂-S,S')L]},¹⁷⁹ Mo to P {[Mo^{VI}O₂(S₂PR₂-S,S')L] and PPh₃},¹⁷⁹ from Mo and W to N,¹⁸⁰ and N and S to P (different mechanisms for transfer of O from R₂SO to PR₃ and from NH₂OAc to PR₃),¹⁸¹ from Re to S {[Re^{VII}(Me)O(O₂)₂] to thiophene, via nucleophilic attack of thiophene S on a co-ordinated peroxy group},¹⁸² from Re to P {[Re^{VII}(Me)O₃] and H₂P(O)OH},¹⁸³ and from Cl, N, S and V to Re {[ClO₄⁻, py-N-O, Me₂SO and VO(aq)²⁺ and [Re^V(Me)O₂]}.¹⁸³ Photoreduction of [Re^{VII}(Me)O₃] by [Fe(CN)₆]⁴⁻ is believed¹⁸⁴ to proceed via [Me(O)₃Re(NC)Fe(CN)₅]⁴⁻. An unusual N to O oxygen-atom-transfer results in O₃ formation from reaction of [Fe(oep)(NO₂)] with O₂.¹⁸⁵ Nitrogen monoxide gives NO₂ with [Fe(oep)(NO₂)].¹⁸⁶ The relationship between oxygen-transfer and ⁹⁵Mo NMR chemical shifts for Mo^{VI}O₂ complexes has been investigated.¹⁸⁷ Nitrogen-atom-transfer between [MoN(OR)₃] and [Mo(NRR')₃] has been established using labelling techniques.¹⁸⁸ Stereospecific NH transfer has been reported for the reaction of [Ta(Me)Cp₂] with aziridines.¹⁸⁹ Molybdenum to P sulfur-transfer kinetics have also been described.¹⁹⁰

Rates for the halide-transfer self-exchange processes [M(X)Cp₂]⁺/[MCp₂], (M = Ru or Os; X = Cl or Br) in MeCN span¹⁹¹ a range of 10⁶, with I > Br > Cl; Ru > Os. Oxidation of indium(I) [from indium amalgam and Ag(CF₃SO₃) in acetonitrile, followed by dilution with water] by [Co^{III}X(NH₃)₅]²⁺ (X = Cl, Br or I) occurs stepwise.^{192,193} The first slow step giving In^{II} is inner-sphere. Halide-atom-transfer between the organometallic free radical [Re(CO)₅] and [CuX(L¹³)]⁺ and [MX(L¹⁴)]⁺ (M = Cu or Ni; X = Cl, Br or I) are all diffusion-controlled processes.¹⁹⁴

Miscellaneous redox reactions

The cation [Mn₂O₂(phen)₄]³⁺ forms an outer-sphere adduct with NO₂⁻ which is reduced by one-electron steps to Mn^{II}.¹⁹⁵ However, the related adduct, [Mn₂O₂(phen)₃(H₂O)₂]³⁺, is unreactive. Reductive cleavage by catechol of {[Fe(dmgBPh₂)₂]O} in the presence of ligands, L, proceeds via monoligated intermediates.¹⁹⁶ Metal catalysis of the reduction of [Ni^{IV}(L¹⁵)₂] by NH₂OH¹⁹⁷ or thiols¹⁹⁸ involves Fe^{II}¹⁹⁷ (via acid-dependent and -independent pathways) or Cu^I¹⁹⁸ reduction of Ni^{IV} to Ni^{III}. Nitrogen monoxide reduction of [Cu(Me₂phen)₂]²⁺ in methanol involves¹⁹⁹ inner-sphere adduct formation, reaction with solvent to give an N-co-ordinated nitrite complex and dissociation to [Cu(Me₂phen)₂]⁺, MeONO and H⁺. Reductive nitrosylation of ferrihaemoproteins^{200,201} and related studies of NO formation from Cu^I-NO₂ complexes^{202,203} are reported. Reduction of Cu^{II} by excess thiourea obeys²⁰⁴ the rate law: $-d[\text{Cu}^{\text{II}}]/dt = k'[\text{Cu}^{\text{II}}]^2[\text{tu}]^7$, involving



a rate-determining bimolecular decomposition of two complexed Cu^{II} species. Copper(II)-catalysed reduction of [IrCl₆]²⁻ by tu involves rate-determining oxidation of [Cu(tu)₅]²⁺ by [IrCl₆]²⁻. Ammine deprotonation is believed to be rate-determining²⁰⁵ in the disproportionation of the N⁷-co-ordinated guanine nucleoside complexes, *trans*-[Ru^{III}(L)(py)(NH₃)₄]³⁺.

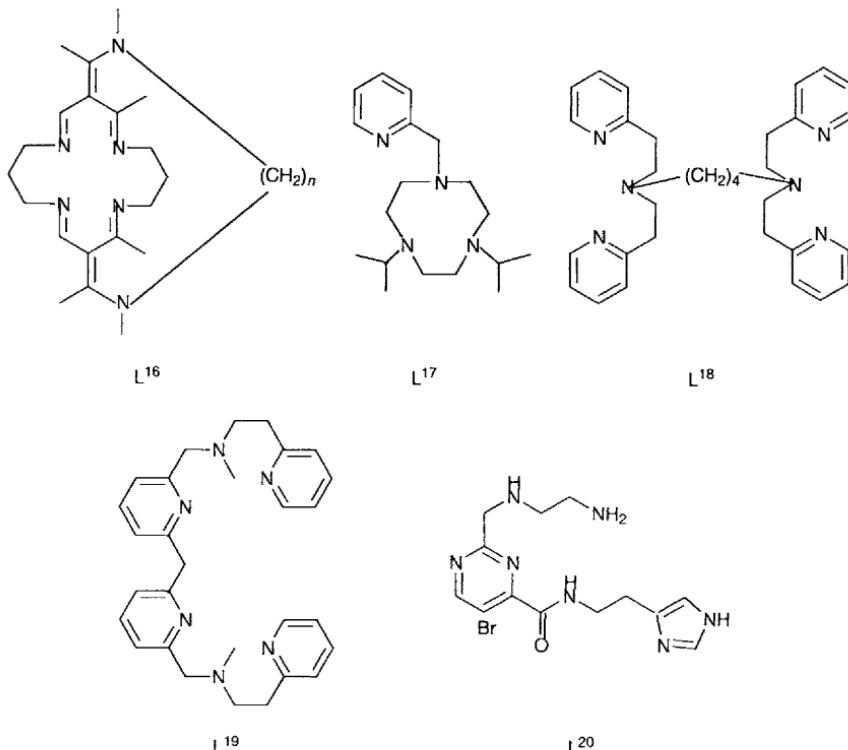
Pulse radiolysis studies of the formate reduction of Hehba-buffered HCrO₄⁻, involving the reduction of Cr^{VI} to Cr^{III} via unstable Cr^V and Cr^{IV}, show²⁰⁶ that Cr^V is not generated directly from Cr^{VI}, arising instead²⁰⁷ from oxidation of Cr^{IV} by Cr^{VI}, via hydride shifts from HCO₂⁻ to Cr=O. Other reductions of Cr^V^{208,209} and Cr^{VI}²¹⁰⁻²¹² have been described. Reduction of *trans*-[Pt(CN)₄X₂]²⁻, (X = Cl or Br) by thiols is first order in complex and [RSH]_{tot} via parallel processes involving halide-bridged electron transfer, for which RS⁻ is > 10⁵ times more reactive than RSH. The basicity of RS⁻ is the predominant determinant of reactivity.²¹³

Electronic and steric effects of phosphine ligands have been quantified for the redox stability of *trans*-[Ru(NO₂)(PR₃)(PR'₃)(terpy)]⁺.²¹⁴ Mechanisms have been described for the oxidation of SCN⁻ by Ce^{IV}²¹⁵ or alkaline diperiodatocuprate(III),²¹⁶ phosphite by diperiodatonickelate(IV),²¹⁷ iodide by diperiodatoargentate(III),²¹⁸ of the corner-shared double-cube [Mo₆MS₈(H₂O)₁₈]⁸⁺ (M = Pb or Bi) by [Fe(H₂O)₆]³⁺,^{219,220} Os^{VIII} catalysis of Sb^{III} oxidation by [Fe(CN)₆]³⁻,²²¹ chromate reduction by [Fe(H₂O)₆]²⁺,²²² Tl⁺,^{223,224} and hypophosphite²²⁵ oxidation by permanganate and V^V,^{226,227} oxidation of fluoro complexes of U^{IV} by XeF₂,²²⁸ HSO₃⁻ oxidation by [Fe(bipy)₃]³⁺,²²⁹ oxidation of [Ru(NH₃)₅(L)]²⁺ (L = butyl sulfoxide) by *cis*-[Ru(NH₃)₄{py-4-C(O)NH₂}₂]³⁺,²³⁰ of S₂O₃²⁻,²³¹ and other substrates^{232,233} by [Fe^{VI}O₄]²⁻, formation of [Rh^I(bipy)₂]⁺ from [Rh^{III}(ox)(bipy)₂]⁺,²³⁴ and oxidation of Cr^{III} by N-bromosuccinimide.^{235,236} The cation [Pt([14]aneN₄)]²⁺ reacts with OH⁻ to give intermediates believed to contain Pt^{III},²³⁷ whereas [Pd([14]aneN₄)]²⁺ is reported to suffer ligand attack.²³⁸ Reaction of [Co^{III}(NH₃)₅(X-pyO)]³⁺ with Me₂C'OH involves²³⁹ reduction of the co-ordinated pyridine-N-oxide followed by intramolecular electron transfer to Co^{III}.

Reactions of oxygen-containing oxidants and reductants

Reaction of the superoxo complexes [Cr^{III}(¹⁶O₂)(H₂O)₅]²⁺ and [Cr^{III}(¹⁸O₂)(H₂O)₅]²⁺ at pH 1 produces²⁴⁰ no detectable ¹⁶O/¹⁸O, ruling out the process: 2[Cr(O₂)(H₂O)₅]²⁺ ⇌ 2[CrO(H₂O)₅]²⁺ + O₂. Homolytic processes are favoured for the initial stages of decomposition in acid, whereas, at higher pH, disproportionation to [Cr^{III}(O₂H)(H₂O)₅]²⁺, [Cr(H₂O)₆]³⁺ and O₂ occurs, via initial formation of O₂^{•-}. The cation [Cr(O₂)([14]aneN₄)(H₂O)]²⁺ reacts with [Fe(H₂O)₆]²⁺ in a three-stage

process: $\text{Cr}(\text{O}_2)^{2+} + \text{Fe}(\text{aq})^{2+} + \text{H}^+ \rightleftharpoons \text{Cr}(\text{O}_2\text{H})^{2+} + \text{Fe}(\text{aq})^{3+}$, followed by the formation and decay of a Cr^{V} species, suggested²⁴¹ to be $[\text{CrO}([\text{14}]\text{aneN}_4)]^{3+}$. Oxygen-binding kinetics²⁴² for five-co-ordinate Co^{II} complexes of lacunar-cyclidene ligands, L^{16} are uncomplicated by solvent dissociation (since ligand or solvent binding in the sixth position is prevented). Variation of oxygen-binding rate with bridge length (optimum at C_6) is associated with entropic effects arising from constraints on O_2 entrance into the cavity. Dioxygen dissociation was solvent independent but sensitive to the axial base. Reaction with O_2 of a series of alkoxo-bridged diiron complexes differing in accessibility to the diiron(II) centre, display kinetics which are first order in the two reacting species when access is unimpeded, whereas impeded access leads to a reduced reaction order in $[\text{O}_2]$.²⁴³ In the latter case, a two-step mechanism was proposed. Photoexcited $[\text{Pt}^{\text{II}}(\text{dpdt})(4,4'\text{-Bu}_2\text{bipy})]$ is deactivated with ${}^3\text{O}_2$ yielding ${}^1\text{O}_2$ which then dehydrogenates the thiolate ligand.²⁴⁴ Dioxygen also results²⁴⁵ from the reaction of $[\text{Mn}^{\text{III}}_2(2\text{-OHsalpn})_2]$ with excess $\text{Bu}^{\text{i}}\text{OOH}$. Reaction of dioxygen with $[\text{Cu}(\text{L}^{17})]^{+}$ at -78°C yields a *trans*-1,2-diperoxocuppper(II) complex, $[(\text{L}^{17}\text{Cu})_2\text{O}_2]^{2+}$. This decays in MeCN with first-order kinetics by converting a pyCH₂ ligand moiety to pyC(O). Isotope effects and activation parameters point²⁴⁶ to an isomerisation to $\mu\text{-}\eta^2\text{:}\eta^2$ -bonding of the peroxy group prior to four-electron oxidation of the ligand. Kinetics of the interconversion²⁴⁷ of *trans*-1,2-diperoxo and (the more stable) $\mu\text{-}\eta^2\text{:}\eta^2$ -peroxy-bonding modes in $[\text{Cu}_2(\text{O}_2)\text{(L}^{18})]^{2+}$ have been studied in detail. Interconversion does not occur directly, but involves either partial or complete O_2 dissociation. Using related ligands, Karlin and co-workers²⁴⁸ conclude that oxygenation of the pyCH₂-moiety in L^{19} does not proceed *via* oxygen activation. Studies of the reaction with O_2 of a dicopper(I) complex with the oxidised form of L^{19} yields a *gem*-diolate, which suggests that both oxygen atoms are used in ligand oxidation *via* oxodicopper(II) rather than *via* peroxodicopper(II) complexes. Interconversions between $\text{M}_2(\mu\text{-}\eta^2\text{:}\eta^2\text{-O}_2)$ and $\text{M}_2(\mu\text{-O})_2$ moieties are thought²⁴⁹ to be generally relevant to both metal-complexed catalysed reductive cleavage of O_2 and water oxidation to O_2 . Reaction of O_2 with $[\text{VO}(\text{ma})_2(\text{H}_2\text{O})]$ involves initial attack²⁵⁰ of O_2 on V^{IV} leading ultimately to *cis*- $[\text{V}^{\text{V}}(\text{O})_2(\text{ma})_2]^-$. A correlation is found ($\rho = -1.1$) between the rate constant for O–O cleavage in $[\text{Fe}_2(\mu_{1,2}\text{-O}_2)(\text{N-Et-hptb})(\text{O}_2\text{CC}_6\text{H}_4\text{X})]^{2+}$ and the Hammett σ value for X, showing the effect of electron-donating substituents.²⁵¹ Peroxodiphosphate formation by Cu^{II} -catalysed oxidation of the phosphinate ion by oxygen has a maximum rate at pH 3.5.²⁵² Kinetics of the reaction between H_2O_2 and Au^{III} in aqueous HCl display²⁵³ an inverse dependence on $[\text{H}^+]$ and $[\text{Cl}^-]$. Thiocyanate inhibition of vanadium bromoperoxidase-catalysed bromide oxidation by H_2O_2 is the result of competitive SCN^- oxidation.²⁵⁴ Kinetics and mechanisms have been studied for the oxidations by H_2O_2 of $[\text{Fe}(\text{CN})_6]^{4-}$,^{255,256} $[\text{Cr}^{\text{III}}(\text{NCS})_2(\text{tn})_2]^{+}$,²⁵⁷ $[\text{W}^{\text{V}}(\text{CN})_8]^{3-}$,^{258,259} $[\text{Ru}(\text{Me}_2\text{phen})_2(\text{H}_2\text{O})_2]^{2+}$,²⁶⁰ copper(II)-Schiff base complexes²⁶¹ of Br^- catalysed by $[\text{VO}(\text{O}_2)\text{(Hheida)}]^-$ and $[\text{VO}(\text{O}_2)\text{(bpg)}]^{2+}$ ²⁶² and of transient formation²⁶³ (see also ref. 264) from H_2O_2 and Mn-reconstituted horse-radish peroxidase. The question of OH[·] formation from H_2O_2 and edta, heda and tcmo complexes of Fe^{II} has been reviewed.²⁶⁵ The mechanisms of a series of H_2O_2 oxidations catalysed by $[\text{Re}(\text{Me})\text{O}_3]$ have been described.^{182,183,266} The complex $[\text{Re}(\text{Me})\text{O}_3]$ catalyses²⁶⁷ the heterolytic decomposition of cumyl hydroperoxide. The radical OH[·] is formed when photoexcited UO_2^{2+} ,²⁶⁸ or $[\text{Fe}(\text{ox})_3]^{3-}$,²⁶⁹ is quenched by H_2O_2 . *tert*-Butyl hydroperoxide reacts with $[\text{Fe}^{\text{III}}(\text{L}^{20})]^{2+}$ to give²⁷⁰



$[Fe^{III}(L^{20})(OOBu^t)]^+$ which suffers O–O homolysis to generate $[Fe^{III}(L^{20})O^\bullet]^+$. Similar $Fe-\eta^1$ -hydroperoxide, $Fe^V=O$ and related species are implicated in a range of organic oxygens.^{253,271-274} Oxidation of $[Fe(CN)_6]^{4-}$ by RO_2^\bullet is an outer-sphere process²⁷⁵ in contrast to the inner-sphere processes involving RO_2^\bullet and $[Fe(H_2O)_6]^{2+}$. The cation $[Fe^{III}(O_2R)(H_2O)_5]^{2+}$ then decomposes by H^+ - and Fe^{2+} -catalysed processes. Catalysis of superoxide dismutation, $2 O_2^\bullet^- + 2 H^+ \rightleftharpoons O_2 + H_2O_2$, has been reported for Mn^{II} -macrocyclic,^{276,277} dimanganese²⁷⁸ and Cu^{II} ²⁷⁹ complexes. The absence of pH dependence for copper-zinc superoxide dismutase suggests²⁸⁰ that Zn and the histidyl imidazolate residue that bridges Cu and Zn facilitate peroxide dissociation.

Water-soluble Fe^{III} -porphyrin complexes catalyse isomerisation of peroxy nitrite to nitrate at the expense of processes leading to nitrite, probably²⁸¹ via $Fe^{IV}=O$ intermediates formed by reversible homolysis of co-ordinated $ONOO^-$.

Oxidation of Co^{II} to Co^{III} by HSO_5^- in the presence of $[MoO_4]^{2-}$ occurs²⁸² via one-electron oxidation of cobalt, after molybdate loss from a heteropolyanion, $[H_6Co^{II}Mo_6O_{24}]^{4-}$, finally yielding $[H_4Co^{III}_2Mo_{10}O_{38}]^{6-}$. The HSO_5^- oxidation of Mn^{III} -porphyrin complexes proceeds via oxygen-atom transfer.²⁸³ Kinetic studies of the oxidation of $[(NC)_5Fe^{II}(im)Ru^{III}(NH_3)_5]^-$ and $[(NC)_5Fe^{II}(im)Ru^{II}(NH_3)_5]^{2-}$ by $S_2O_8^{2-}$ reveal²⁸⁴ the role of Ru^{II} in assisting the oxidation of the Fe^{II} centre. The difference in reactivity with $S_2O_8^{2-}$ (after correction for differences in reduction

potentials and charge) between $[\text{Fe}^{\text{II}}(\text{CN})_5\text{L}]^{3-}$ and the corresponding $[\text{Ru}^{\text{II}}(\text{NH}_3)_5\text{L}]^{2+}$ are ascribed²⁸⁵ to hydrogen bonding between the peroxydisulfate and the amines in the latter case and non-adiabaticity in the former. Oxidation by $\text{S}_2\text{O}_8^{2-}$ of Cu^{II} macrocyclic complexes²⁸⁶ and decomposition of H_2O_2 and H_2O and O_2 catalysed by a dinuclear Fe^{III} complex²⁸⁷ and Mn^{II} complexes²⁸⁸⁻²⁹¹ have also been described. The redox chemistry of sulfur is discussed in the next section.

Non-metal redox reactions

Kinetic studies of the acid-catalysed reduction of nitrite by $\text{Me}_3\text{N}\cdot\text{BH}_3$ (giving H_2 and N_2O) are consistent²⁹² with rate-determining attack of H^- on H_2NO_2^+ (or NO^+) to give HNO as a reactive intermediate. Iodination of $\text{R}_3\text{N}\cdot\text{BH}_3$ has also been studied.²⁹³ Carbon dioxide is reduced to CO and formate with $[\text{Fe}^0(\text{tpp})]$ (in the presence of Lewis²⁹⁴ or weak Brønsted²⁹⁵ acids) and other metal catalysts.^{296,297} The radical H^\bullet gives either H_2 and ${}^{\bullet}\text{N}_2\text{H}_3$ with N_2H_4 in aqueous acid²⁹⁸ (*via* hydrogen-atom abstraction) or ${}^{\bullet}\text{NH}_2$ and NH_3 from N_2H_5^+ (*via* addition-fragmentation). The constancy of the rate of decomposition of HOONO in solutions of varying viscosity argues against a free-radical process for this unimolecular reaction.²⁹⁹ Peroxynitrite oxidations of As^{III} and S^{IV} proceed *via* O-bridged precursor complexes for which reactions *via* H_2AsO_3^- or HSO_3^- with HOONO dominate.³⁰⁰ The reactivity series with peroxynitrite, $\text{Sn}^{\text{II}} > \text{Sb}^{\text{III}} > \text{As}^{\text{III}} > \text{S}^{\text{IV}} \gg \text{P}^{\text{I}} > \text{P}^{\text{III}}$, reflects the relative accessibilities of the electron-rich sites of the reductants. No evidence has been found³⁰¹ for the direct nitrosation of H_2O_2 by ${}^{\bullet}\text{NO}$, a process which only takes place in the presence of O_2 . The rate law supports the intermediacy of ONOONO (or isomeric forms), the precursor to ${}^{\bullet}\text{NO}_2$ and N_2O_3 . The reaction of ${}^{\bullet}\text{N}_3$ and ${}^{\bullet}\text{NO}$ in the presence of H_2O_2 has also been studied.³⁰² The kinetics of the oxidation of NH_2OH by aqueous bromine,³⁰³ of the reaction between OBr^- and NH_3 or $\text{C}(\text{O})(\text{NH}_2)_2$,³⁰⁴ and reactions of *N*-haloamines,^{305,306} -amino acids³⁰⁷ and -amides³⁰⁸ have been reported. The biological role of NO^{309} and the atmospheric role of nitrous acid³¹⁰ have both been reviewed. The 10^5 -fold acceleration by freezing³¹¹ of the reaction of nitrous acid with oxygen has been investigated in detail.³¹² The radical ${}^{\bullet}\text{NO}$ is thought³¹³ to be an intermediate in the UV irradiation of alkaline ${}^{15}\text{NO}_2^-$ in the presence of the aci-form of nitromethane, $\text{CH}_2={}^{14}\text{NO}_2^-$, since the adduct, $\text{CH}({}^{14}\text{NO}_2)({}^{15}\text{NO})^{2+-}$, is formed.

Three studies³¹⁴⁻³¹⁶ have been reported of the mechanism of ozone decomposition in water. Photodissociation of aqueous O_3^- and its recombination have been studied³¹⁷ on the picosecond time-scale. The kinetics of the $\text{HCO}_2\text{H}-\text{H}_2\text{O}_2$ ³¹⁸ and $\text{ox}^{2-}-\text{H}_2\text{O}_2$ ³¹⁹ reactions and the rates of the uncatalysed and ${}^1\text{O}_2$ -forming ketone-catalysed decomposition of peroxymonosulfuric acid³²⁰ have been reported. Formations of SO_4^{2-} and $\text{S}_2\text{O}_6^{2-}$ from the photodecomposition of HSO_3^- arises³²¹ from self-reaction of $\text{SO}_3^{\bullet-}$. In oxygen-saturated solution, a short-chain process occurs leading to SO_4^{2-} and $\text{S}_2\text{O}_8^{2-}$, the latter arising from recombination of $\text{SO}_5^{\bullet-}$. Two other groups^{322,323} have studied these processes. The rate constant of the reaction of $\text{SO}_5^{\bullet-}$ and HO_2^{\bullet} (possibly forming a tetroxide transient) has led to the suggestion³²⁴ that this process is a chain-terminating step in the chain-oxidation of S^{IV} to S^{VI} in cloud water.^{325,326} Kinetic data for the oxidation of S^{IV} by H_2O_2 in aqueous base have been interpreted in terms of two processes,³²⁷ $\text{SO}_3^{2-} + \text{H}_2\text{O}_2$ and $\text{SO}_3^{2-} + \text{HO}_2^-$. Additional industrially- and environmentally-relevant studies have been reported, including the $\text{Fe}^{\text{III}}-\text{Fe}^{\text{II}}$ -complex catalysed H_2S oxidation by air,^{328,329} bisulfite photooxidation

tion,³³⁰ metal-catalysed oxidation of S^{IV} by oxygen^{331,332} and the involvement of HONH(SO₃)⁻ and HON(SO₃)₂²⁻ in such processes.³³²⁻³³⁵

Photochemical decomposition of ClO₂ in water involves Cl⁺ loss from thermally-equilibrated ClOO.³³⁶ The reaction of ClO₂⁻ (generated *in situ* from ClO₂ and I⁻) with I⁻ is both substrate-inhibited and auto-catalytic, involving subsequent ClO₂⁻-I₂ and HOI and HIO₂ disproportionation reactions.³³⁷ The reaction of BrO₃⁻ and I₂, of stoichiometry 2 BrO₃⁻ + I₂ ⇌ 2 IO₃⁻ and Br₂, involves an acid-dependent induction time associated with the sudden depletion of I₂ and formation of IBr as a transient.³³⁸ A 17-step mechanism is proposed. Bromine hydrolysis has been re-investigated,³³⁹ with the forward rate constant, k_f , for Br₂(aq) + H₂O ⇌ HOBr + Br⁻ + H⁺ being 97 s⁻¹ (25.0 °C, $\mu = 0.50 \text{ M}$) and for the reverse, k_b , $1.6 \pm 10^{10} \text{ M}^{-2} \text{ s}^{-1}$. Oxidations by ClO₂⁻³⁴⁰ and BrO₃⁻^{341,342} of sulfur-containing organic substrates have been studied in detail by Simoyi and co-workers. Rates of oxide-radical and hydrated-electron reactions with IO₃⁻ have been reported.³⁴³

Oscillating reactions and chemical chaos

A special issue of *Faraday Trans.*³⁴⁴ on chemical instabilities was devoted to the work of Peter Gray. New means of developing chemical patterns from coupling diffusion and complex kinetics have been reviewed.³⁴⁵ Studies relevant to the chemistry of oxyhalogen-sulfur systems³⁴⁶ were discussed in the previous section.³⁴⁰⁻³⁴³

Further reports have appeared on various aspects of the Belousov-Zhabotinsky (B-Z) reaction.³⁴⁷⁻³⁸⁸ Studies have shown that oxygen shortens the duration of B-Z oscillations.³⁴⁷ The effect of light³⁵⁶⁻³⁶² has been reported, including light-induced quenching of oscillations³⁵⁸ and spiral waves.³⁶¹ Temperature,³⁶⁵ stirring,^{366,367} dc electric^{368,369} and electric current³⁷⁰ effects, perturbations by Ag⁺,³⁵⁰ the dependence of system dynamics on initial reagent concentrations,^{353,354} the development of different modes of oscillatory behaviour from mixed substrates compared with those seen from single substrates,³⁵⁵ the identity of malonic-acid derived³⁶³ and other³⁶⁴ intermediates, and stochastic resonance^{371,372} have all been examined. Magnetic resonance images have been obtained³⁷⁵ for the Ce^{III}- and Ru^{II}-catalysed B-Z reactions but not for the Fe^{II}-catalysed reaction. Belousov-Zhabotinsky reactions have been carried out, gel-^{376,377} or membrane-immobilised,³⁷⁸⁻³⁸¹ cation-exchange resin bead-loaded³⁸² and in water-in-octane reverse microemulsions.³⁸³ In the microemulsion, the rate is *ca.* 10-fold larger than in homogeneous aqueous solution. Rate depends on [NaBrO₃]² in the microemulsion and on [NaBrO₃] in the homogeneous aqueous solution. The origins of the induction period for the bromate-ferroin clock reaction^{389,390} and Leisegang-precipitation phenomena from Co^{II} and NH₄OH in gelatin have been studied.³⁹¹ Chinake and Simoyi³⁹² have drawn attention to the environmental relevance of non-linear kinetic behaviour in sulfur-compound oxidation. Photoinduction and photoinhibition of oscillations are observed³⁹³ in the BrO₃⁻-HSO₃⁻-[Fe(CN)₆]⁴⁻ (BSF) system in a continuous-flow stirred tank reactor (CSTR). A mechanistic model for the BSF system is the subject of debate.^{394,395} Chemical instability in the BrO₃⁻-HSO₃⁻³⁹⁶ and the H₂O₂-HSO₃⁻³⁹⁷ flow systems has been studied. Swinney and co-workers³⁹⁸ have studied the IO₃⁻-HSO₃⁻-[Fe(CN)₆]⁴⁻ system in a thin-gel reactor. Chaotic temporal pH changes occur³⁹⁹ as a result of coupling of the CO₂ hydration equilibrium and slow CO₂ removal with the H₂O₂-HSO₃⁻-[Fe(CN)₆]⁴⁻ oscillatory system in a CSTR. pH

Oscillations are also seen in H_2O_2 ⁻⁴⁰⁰ or BrO_3^- ⁻⁴⁰¹ HSO_3^- -solid marbleflow systems. Intermediacy of HOS_2O_3^- and its reaction with H_2O_2 and $\text{S}_2\text{O}_3^{2-}$ are proposed⁴⁰² for the Cu^{II}-catalysed thiosulfate oxidation by H_2O_2 which displays oscillatory behaviour in a flow system. Copper(II)-catalysed thiourea- H_2O_2 and $\text{S}_2\text{O}_8^{2-}$ - SCN^- oscillators have also been reported.^{403,404} Oscillations without kinetic bistability have been observed⁴⁰⁵ in a $[\text{MnO}_4^-]$ -cyclic diketone system in a CSTR. Other permanganate-based oscillators have been reported.⁴⁰⁶ Travelling waves in the IO_3^- - HSO_3^- system,⁴⁰⁷ models of the Briggs-Rauscher (malonic acid- IO_3^- - H_2O_2)^{408,409} and Bray-Liebhafsky (I_2 - IO_3^- - H_2O_2)⁴¹⁰ systems have been reported. Other related studies have appeared.⁴¹¹⁻⁴²⁶

3 Substitution

Jordan⁴²⁷ has proposed a new model which analyses the effect of leaving groups, Y, on ΔV^* for dissociative substitution in ML_5Y . Apparently contradictory views of the origins of non-linear temperature behaviour of product ratios in processes showing selectivity can be reconciled⁴²⁸ by viewing the phenomenon as arising from the non-linear change with temperature of the ratio of the concentrations of two intermediates.

Six-co-ordination

Rates of hexameric and tetrameric Cr^{III}-aqua-hydroxo complex formation from $[\text{Cr}_3(\mu\text{-OH})_4(\text{OH})(\text{H}_2\text{O})_9]^{4+}$ and $[\text{Cr}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$ and of the cleavage of the trimer and tetramer have been investigated by Drljaca and Spiccia.^{429,430} Interconversion and cleavage of singly- and doubly-bridged ions, $[(\text{H}_2\text{O})_5\text{Rh}(\mu\text{-OH})\text{Cr}(\text{H}_2\text{O})_5]^{5+}$ and $[(\text{H}_2\text{O})_4\text{Rh}(\mu\text{-OH})_2\text{Cr}(\text{H}_2\text{O})_4]^{4+}$ are Cr^{III}-centred substitution processes.⁴³¹ Hydrolysis of $[(\text{tmpa})\text{Cr}(\mu\text{-O})(\mu\text{-L})\text{Cr}(\text{tmpa})]^2+$ (L = sulfate or molybdate) displays⁴³² a fast water-induced fission of the ligand bridge (facilitated by *cis*-labilising $\mu\text{-O}$) followed by L loss from either $[\text{CrL}(\text{tmpa})(\mu\text{-O})\text{Cr}(\text{tmpa})(\text{H}_2\text{O})]^2+$ or its conjugate base. Anation of $[\text{Cr}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$ by $[\text{Mo}(\text{CN})_8]^{4-}$ is an associative-interchange process.⁴³³ Complexation kinetics have been reported for $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ and L-histidine,^{434,435} for $[\text{Rh}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$ and DL-methionine⁴³⁶ or pyridine-2-aldoxime,⁴³⁷ for $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ and 8-hydroxyquinoline,⁴³⁸ for $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ with folic acid⁴³⁹ or $[\text{Co}(\text{His})(\text{NH}_3)_5]^{2+}$,⁴⁴⁰ for $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$ and amino acids or H₃nta⁴⁴¹ or 7-substituted 8-hydroxyquinoline-5-sulfonates.^{442,443} Formation (from molybdate and the corresponding aquo-complex) and hydrolysis of $[\text{Co}(\text{OMoO}_3)(\text{NH}_3)_5]^+$ involve Mo^{VII}-O not Co^{III}-O cleavage.⁴⁴⁴ The ΔV^* measurements (with other data) for SCN⁻ substitution into $[\text{Cr}(\text{Hedta})(\text{H}_2\text{O})]$ and $[\text{Cr}(\text{edta})]^-$ support⁴⁴⁵ the suggestion that the lability of Cr^{III}-edta complexes is associated with activation by transient chelation of the pendant arm of the pentadentate edta. Water-exchange in the ion pair, *p*-[$[\text{Co}(\text{NH}_3)(\text{H}_2\text{O})(\text{tren})]^{3+}\cdot\text{Cl}^-$] occurs as readily as anation by Cl⁻, suggesting⁴⁴⁶ that entry of Cl⁻ and H₂O from positions adjacent to the group *trans* to tren NH₂ (the *p* site) affects re-entry of the leaving water. Anation of $(\alpha,\beta)[\text{Co}(\text{OH})(\text{tetren})]^{2+}$ by S₂O₃²⁻ gives both S- and O-bonded $[\text{Co}(\text{S}_2\text{O}_3)(\text{tetren})]^+$ via an internal conjugate-base process⁴⁴⁷ involving $[\text{Co}(\text{Htetren})(\text{H}_2\text{O})]^{2+}$. Substitution of H₂O in $[\text{Os}^{\text{III}}(\text{NH}_3)_5(\text{H}_2\text{O})]^{3+}$ by $[\text{Fe}^{\text{II}}(\text{CN})_6]^{4-}$, giving $[\text{Os}^{\text{IV}}(\text{NH}_2)(\text{NH}_3)_4$

$\{Fe^{II}(CN)_6H\}$], involves⁴⁴⁸ oxidation of Os^{III} to the more labile Os^{IV} by residual oxygen. Binuclear-complex formation between *trans*-[$Co^{III}(\text{salen})(H_2O)_2$]⁺ and $[Fe(CN)_6]^{3-}$ proceeds by an I_d mechanism.⁴⁴⁹ Formation of the analogous intermediate from $[Fe(CN)_6]^{4-}$ precedes Fe^{II} -to- Co^{III} electron transfer. Nucleophilic substitution of water has been studied for $[Cr(\text{bipy})(H_2O)_4]^{3+}$ and azide ion,⁴⁵⁰ for $[Re(CN)_4(\text{NO})(H_2O)]^{2-}$ and SCN^- , N_3^- and tu,⁴⁵¹ for *trans*- $[Ru(NH_3)_4L(H_2O)]^{2+}$ ($L = EPh_3$; $E = P$, As or Sb) and imidazole (by an I_d mechanism),⁴⁵² for *cis*- $[Ru(NH_3)_4(\text{Him})(H_2O)]^{2+}$ or *cis*- $[Ru(\text{bipy})_2(\text{Him})(H_2O)]^{2+}$ and imidazole,⁴⁵³ for $[Ru^{III}(\text{edta})(H_2O)]^-$ and cysteine,⁴⁵⁴ for $[Ru^{III}(\text{tpps})(H_2O)_2]^{3-}$ and SCN^- ,⁴⁵⁵ (by a D mechanism), for $[Ru^{II}(\text{tpps})(CO)(H_2O)]^{4-}$ and CN^- ,⁴⁵⁶ for $[Cr_3O(OAc)_6(H_2O)_3]^+$ and urea,⁴⁵⁷ and at the tetrahedral Ni of $[Mo_3NiS_4(H_2O)_{10}]^{4+}$ by tppts³⁻, Br^- , I^- and SCN^- (by an I_d mechanism).⁴⁵⁸ Substitution of H_2O by SCN^- at Mo is a much slower process. Transformation of the edge-linked double-cube, $\{[Mo_3PdS_4(H_2O)_9]_2\}^{9+}$, to $[Mo_3(PdL)S_4(H_2O)_9]^{4+}$, by $L = Cl^-$, Br^- or SCN^- , is rapid⁴⁵⁹ and is followed by substitution of non-identical waters on each Mo. For $L = SCN^-$, a slow isomerisation of Pd-NCS to Pd-SCN is also observed. The replacement of water by HCO_3^- in a series of polyvanadate ions, such as $[V^{IV}_2O_3(OH)_3(H_2O)_5]^-$ has also been reported.⁴⁶⁰

Stereoselective ternary complexation of Cu^{II} with (*S*)-amino acid amides and (*R*)- or (*S*)-histidine and (*R*)- or (*S*)-tyrosine has been described.⁴⁶¹ The dynamics of ternary-complex formation between $[Zn(\text{nta})(H_2O)]^-$ and bipy and $Me_2\text{bipy}$,⁴⁶² between $[Ni^{III}(\text{ada})(H_2O)]^-$, with bipy and phen,⁴⁶³ between $[Mo^{IV}O_2(\text{CN})_4]^{4-}$ and bipy,⁴⁶⁴ and between $[Fe^{III}(\text{nta})(H_2O)_2]$ and bipy⁴⁶⁵ have also been reported.

From studies of the acid-catalysed aquation of $[Co(O_2H)(CN)_5]^{3-}$, the relative affinity of Co^{III} for a series of oxygen donors is found¹⁷⁶ to be $HO_2^- > H_2O > H_2O_2$. Spontaneous, acid- and base-catalysed aquation of the neutral complexes *trans*(*O*)- $[Co(\text{taud})X]$ ($X = Cl^-$, Br^- or NO_2^-) has been reported.⁴⁶⁶ Square-pyramidal and trigonal-bipyramidal intermediates produced in acid- and base-hydrolysis of Co^{III} complexes of the type *trans*- $[CoCl_2(L-L)_2]^+$ have been studied by molecular-orbital and molecular-mechanics methods.⁴⁶⁷ Acid-catalysed hydrolysis of $[Co(CO_3)(\text{Him})_4]^+$ involves a rapid protonation, rate-determining carbonate-chelate ring-opening and subsequent rapid loss of HCO_3^- .⁴⁶⁸ The Hg^{2+} -assisted removal of Cl^- from *trans*- $[CrCl_2(NH_2R)_4]^+$ ($R = Et$, Pr or Bu) to give *trans*- $[Cr(NH_2R)_4(H_2O)_2]^{3+}$, proceeds⁴⁶⁹ in two well separated interchange processes. The kinetics of dissociation of Tiron (= H_2L) complexes of Fe^{III} point to⁴⁷⁰ parallel processes involving aqua- and hydroxo- $Fe^{III}-L^{2-}$ and $-LH^-$ complexes. Urea dissociation from $[Cr_3O(OAc)_6(\text{urea})_3]^+$ is strongly labilised⁴⁷¹ compared with $[Cr(\text{urea})_6]^{3+}$. Methanol may be replaced in $[Co^{III}(L)(MeO)(MeOH)]$ ($L = \text{PPIX dimethyl ester}$) by a series of substituted pyridines in a dissociative process,⁴⁷¹ with the electron-donating MeO^- favouring entry of the least basic pyridine. A plot of $\ln k_{obs}$ vs. pK_a exhibits a minimum related to changes in pyridine π - and σ -bonding in the transition state. Photolysis of $[Fe^{II}(\text{PPIX})(CO)]$ in dmso yields the same five-co-ordinate transient seen upon photolysis of $[Fe^{II}(\text{PPIX})(\text{dmso})_2]$, namely $[Fe^{II}(\text{PPIX})(\text{dmso})]$. This decays by dmso co-ordination followed by substitution of bound dmso by CO.⁴⁷² Structural changes, occurring on the nanosecond time-scale, which result from the photolysis of the myoglobin-CO complex reveal that geminate CO rebinding is more important in the crystalline solid⁴⁷³ than in solution. Time-

resolved UV circular dichroism,⁴⁷⁴ molecular dynamics⁴⁷⁵ and other⁴⁷⁶ simulations and IR spectroscopy⁴⁷⁷ have been applied to similar systems. The kinetics of azide binding to ferricytochrome c, both wild-type and a series of site-specific mutated variants, reveal⁴⁷⁸ a two-step reversible process with saturation.

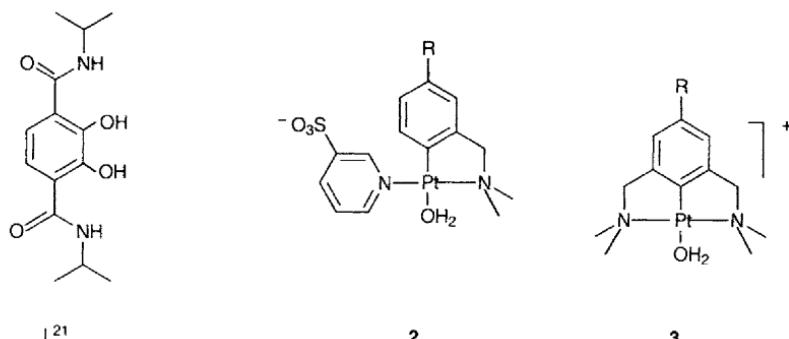
Racemisation kinetics have been studied for *fac*- Λ -(+)(546)- and *fac*- Δ -(−)(546)-[Cr(Val)₃] in dmso, in which Λ -(+)(546)- and Δ -(−)(546)-[Cr(Val)₂(N-Val)(dmso)] are also formed.⁴⁷⁹ Isomerisation and racemisation (and other conformational and configurational changes) for [Cr{(RO)₂bipydo}₃]³⁺ in aqueous solution are all intramolecular processes.⁴⁸⁰ Related studies on [M(S₂C₂R¹R²)₃], (M = Mo or W)⁴⁸¹ and [Ga(L²¹)₃]³⁺⁴⁸² have also been reported.

Base hydrolysis

Both the steric course and rate of the base hydrolysis for *trans*-[CoX(NH₃)₄(NH₂-Me)]²⁺ (X = Cl, Br or NO₃), indicative⁴⁸³ of a dissociative conjugate-base mechanism, differ markedly from those of [CoX(NH₃)₅]²⁺. Variation with temperature of ΔV^* for the base hydrolysis of *trans*-[CoCl₂([14]aneN₄)]⁺ is ascribed⁴⁸⁴ to a change from rate-determining Cl[−] release to rate-determining conjugate-base formation. Base-hydrolysis of *trans*(O)-[Co(taud)X] (X = Cl[−], Br[−] or NO₂[−])⁴⁶⁶ of *cis*-[CoX(en)₂L]²⁺ (L = NH₃ or amine; X = Cl or Br)⁴⁸⁵ of α,β -anti-[CrCl(picdien)]²⁺⁴⁸⁶ of *cis*-[Co(salicylato)(en)₂(NH₂R)]²⁺ (R = H, Me or Et)⁴⁸⁷ and of *cis*-[CoCl(β -Ala)(en)₂]²⁺⁴⁸⁸ have been reported. Gillard and co-workers⁴⁸⁹ argue that the higher rate of base hydrolysis for [Pt([¹H₈]bipy)₂]²⁺ compared with [Pt([²H₈]bipy)₂]²⁺ supports OH[−] attack at ligand not metal. Calculation suggests⁴⁶⁷ that the square-pyramidal and trigonal-bipyramidal intermediates produced in base-hydrolysis of Co^{III} complexes (neglecting solvation effects) are much lower in energy than corresponding intermediates formed in acid hydrolysis.

Four-co-ordination

Rate constants, *k*, for complex formation between [Pd(H₂O)₄]²⁺ and a series of thioethers follow trends similar to those for other Pd complexes,⁴⁹⁰ viz. $\log k = \gamma + \alpha\Sigma\sigma^* + \beta\theta$, the latter representing intrinsic, electronic and steric terms respectively. A trigonal-bipyramidal transition state, formed⁴⁹¹ when acetic or propionic acid and [Pd(H₂O)₄]²⁺ react to give [Pd(O₂CR)(H₂O)₃]⁺, is stabilised by hydrogen bonding between the entering RCO₂H and the departing H₂O. The claim that the lability of the aqua ligand in [Pt^{II}{C₆H₃R(CH₂NMe₂)₂}(pySO₃-3)(H₂O)] **2** is associated with the Pt-C σ bond of a cyclometallated ligand has been criticised⁴⁹² and defended.⁴⁹³ Large negative ΔV^* and ΔS^* for ligand substitution in the related compound, [Pt{C₆H₂R(CH₂NMe₂)₂-2,6}](H₂O)]⁺ **3**, show⁴⁹⁴ H₂O substitution to be associative. Direct isomerism between *trans*- and *cis*-[Pt(CH₃)Cl(dmso)₂] occurs solely by a water-catalysed process.⁴⁹⁵ All configurational isomers of [Pt(edda-*N,N'*)] aquate to give tridentate edda-*N,N'* with a pendant glycinate residue. The related [Pt(edda-*N,N'*)] is inert under the same conditions.⁴⁹⁶ The anion [Pt(nta)Cl]^{2−} has a pendant glycinate residue, which is believed⁴⁹⁷ to be weakly axially associated. The kinetics of complex formation between M²⁺ (M = Ni, Co or Cu) and the β-diketone, Hamac, reveal⁴⁹⁸ two processes, involving reactions of enolate and the enol tautomer only. Dissociation of [M(amac)]⁺ has also been studied. Replacement of ClO₄[−] by



nitriles in $[Ir(ClO_4)(CO)(PPh_3)_2]$ is an associative process.⁴⁹⁹ Initial rapid complex formation between SCN^- and $trans-[Au^{III}(CN)_2X_2]^{2-}$ ($X = Cl$ or Br) gives $[Au(CN)_2X(SCN)]^{2-}$ followed by slower intermolecular outer-sphere reduction by SCN^- to $[Au^I(CN)_2]^-$.⁵⁰⁰ Substitution of Cl^- by substituted benzenethiolates, RS^- , in $[Cl_2FeS_2VS_2FeCl_2]^{3-}$ to give $[(RS)ClFeS_2VS_2FeCl(SR)]^{3-}$ proceeds by non-catalysed dissociative and acid-catalysed associative processes.⁵⁰¹ Studies of the mechanisms of reactions of Fe-S clusters have been reviewed.⁵⁰²

Mechanistic studies of Pt^{II} ⁵⁰³⁻⁵⁰⁹ Pd^{II} ⁵¹⁰ and other metal⁵¹¹ co-ordination complexes, relevant to their anti-tumour activity,⁵¹²⁻⁵¹⁴ have been reviewed. *trans* Analogue⁵⁰⁷ of the active species from the prototypical anti-tumour complex, cisplatin,⁵⁰³ *trans*- $[PtCl(NH_3)_2(H_2O)]^+$ ⁵¹⁵ or *trans*- $[Pt(NH_3)_2(H_2O)_2]^{2+}$ ⁵¹⁶ react with inosine or 1-methylinosine by displacement of H_2O rather than OH^- , with the N^7 -co-ordination site being preferred over the N^1 site.

The cation *trans*- $[Pt(NH_3)_2(H_2O)_2]^{2+}$ is 7–8-fold more reactive than *trans*- $[Pt(OH)(NH_3)_2(H_2O)]^+$. Reaction of inosine, 5'-INP or 5'-GMP with $[PdCl_2(R_2NCH_2CH_2NR_2)]$ ($R = Me$ or Et)⁵¹⁷ also reveals preference for N^7 -bonding in acid and N^1 -bonding in base. Both bis-chelated $[Pt(Me_2NCH_2CH_2PPh_2-N,P)_2]^{2+}$ and its ring-opened form, $[PtCl(Me_2NCH_2CH_2PPh_2-N,P)(Me_2NCH_2CH_2PPh_2-P)]^+$, which exist in a $[Cl^-]$ -dependent equilibrium, react with 5'-GMP to give diastereomeric forms of *cis*- $[Pt(Me_2NCH_2CH_2PPh_2-N,P)(Me_2NHCH_2CH_2PPh_2-P)(5'-GMP-N^7)]^{3+}$.⁵¹⁸ Interestingly, 5'-GMP may readily be displaced by Cl^- , but not by the sulfur-donor *N*-acetyl-L-methionine. Nucleophilic displacements by I^- at Pt^{II} in $[Pt(OAc)_2(R_2NCH_2CH_2NR_2)]$ ($R = 4$ -fluorophenyl) occur predominantly *via* the aqua and diaqua intermediates.⁵¹⁹ Further studies have appeared on the reactions of single-⁵²⁰⁻⁵²² and double-stranded^{521,523} oligonucleotides with *cis*- $[Pt(NH_3)_2(H_2O)_2]^{2+}$ ^{520,523} and $-[Pt(NH_3)_3(H_2O)]^{2+}$ ^{520,522,523} or *cis*- $[PtCl_2(NH_3)_2]$ and *cis*- $[PtCl(NH_3)_2(H_2O)]^+$ ⁵²¹ as analogues of DNA platinating at guanine residues in intrastrand cross-links. Chottard and co-workers^{520,523} show that rates of platinating increase slightly with oligonucleotide chain-length for both *cis*- $[Pt(NH_3)_2(H_2O)_2]^{2+}$ and $-[Pt(NH_3)_3(H_2O)]^{2+}$, with rate constants for reaction at each guanine in a single-strand nucleotide reported.⁵²⁰ Chelation processes involving *cis*- $[Pt(NH_3)_2(H_2O)_2]^{2+}$ were also described. The double-stranded oligonucleotide d(TTGGCCAA)₂ reacts to give mono adducts with both *cis*- $[Pt(NH_2)_2(H_2O)_2]^{2+}$ and $[Pt(NH_3)_3(H_2O)]^{2+}$ more rapidly than the single-stranded d(CTGGCTCA),⁵²³ asso-

ciated with increased rate of reaction at the 5'-guanine. The cation *cis*-[Pt-(NH₃)₂(H₂O)₂]²⁺ reacts with both more rapidly than does [Pt(NH₃)₃(H₂O)]²⁺. The latter not only gives the two singly-platinated oligonucleotides with d(CTGG) but also doubly-platinated species, both N⁷-bound at adjacent guanines.⁵²² Rate studies highlight the importance of ionic strength effects. Chelation of the monoadduct from *cis*-[Pt(NH₃)₂(H₂O)₂]²⁺ is slower for the double-stranded compared with the single-stranded oligonucleotide. Sadler and co-workers⁵²¹ report related studies on cisplatin, with *cis*-[PtCl(NH₃)₂(H₂O)]⁺ forming two monofunctional adducts with both single- and double-stranded oligonucleotides, with one of the two guanines in the two substrates reacting faster than the other. Ring closure to form the complex in which both guanines in the double-stranded substrate are bonded occurs *ca.* 10-fold faster for one singly-bonded adduct compared with the other, an observation confirmed by Chottard and co-workers.⁵²³ Dinuclear intrastrand adduct formation follows initial rapid complexation at either 5'G or 3'G guanines in a single-strand oligonucleotide on reaction with [{*trans*-PtCl(NH₃)₂} {μ-NH₂(CH₂)_nNH₂}]²⁺ (*n* = 2–6) with ring closure for *n* = 4–6 being faster than for *n* = 2–3.⁵²⁴ Thioethers, such as L-methionine, are much more reactive than thiols in reaction⁵²⁵ with [Pt(cbdca-O,O')(NH₃)₂] (carboplatin), giving stable ring-opened species, *cis*-[Pt(cbdca-O)(L-HMet-S)(NH₃)₂]. The latter ring-closes to [Pt(L-Met-N,S)(NH₃)₂] very slowly. Related reactions of [PtCl(en)(MeCO-Met-S)]⁺ with nucleotides have also been studied.⁵²⁶ Palladium-complex promoted peptide cleavage is discussed below. Relevant structural studies are noted.^{527–533}

Activation parameters for olefin replacement in [Pd(2-pyCH=NMe)(η²-olefin)], in CHCl₃, are consistent with an associative mechanism.⁵³⁴ Formation of μ-amido complexes from [{Pd(Ph)(PPh₃)(μ-OH)}₂] and Bu^sNH₂ shows⁵³⁵ second-order dependence on [Bu^sNH₂]. Two theoretical approaches to modelling the substitution process, [Pd(CH₃)(NH=CHCO₂)(PH₃)] + CO ⇌ [Pd(CH₃)(NH=CHCO₂)(CO)] + PH₃, have been compared.⁵³⁶ The solvent effects on the kinetics of cyclopalladation in [Pd(Bn₂Medptn)(solv)]²⁺, (rate for solv = py ≫ dmso > dmf ≫ MeCN), are inconsistent⁵³⁷ with a mechanism involving a three-co-ordinate intermediate. A five-co-ordinate intermediate is proposed⁵³⁸ for the intramolecular exchange associated with the fluxionality of [PtX₂(triphos)] (X = CN or SCN).

Five-, seven- and higher-co-ordination

The mechanism by which Cr^V complexes, such as five-co-ordinate [CrO(ehba)₂][−], cleave DNA⁷⁴ has been explored.⁵³⁹ Reaction with pyrophosphate gives distorted trigonal-bipyramidal [CrO(ehba)(H₂P₂O₇)][−] and square-pyramidal [CrO(H₂P₂O₇)₂][−], in contrast to isomeric forms of six-co-ordinate [CrO(ehba)₂(H₂PO₄)]^{2−} with phosphate. Large negative ΔV[‡] and ΔS[‡] for the substitution of X by P(OMe)₃ in trigonal-bipyramidal [PdX(pp₃)]⁺ are consistent⁵⁴⁰ with an associative mechanism. Very rapid initial addition of CO to [Ni^I([14]aneN₄)]⁺ to give [Ni^I(CO)([14]-aneN₄)]⁺ is followed⁵⁴¹ by a slower, first-order process, believed to involve a ligand isomerisation. The unusual structure of [Cu^{II}(tren)(dzf)]²⁺, with one Cu–N bond to dzf being much longer than the other, has prompted the suggestion⁵⁴² that the compound models the associative complex for ligand substitution in five-co-ordinate [Cu(tren)L]²⁺. Origins of the difference in photodissociation of NO from [M^{II}(tpp)(NO)] (M = Fe or Co) have been explored.⁵⁴³

The exchange between free F^- or HF and $[UO_2F_n(H_2O)_{5-n}]^{2-n}$, faster for $n = 4$ and 5 compared with other values of n , is dominated by two pathways, one involving exchange between two uranyl complexes, probably fluoride-bridged, the other involving exchange between free F^- and a uranyl complex.⁵⁴⁴ The mechanism involves ligand-promoted rate-determining water dissociation. Substitution of arsenazo III by edta or dtpa on Eu^{III} involves a fast step and then a slow step, the latter believed⁵⁴⁵ to involve acid-catalysed dechelation of arsenazo III. Other relevant dynamic⁵⁴⁶ and structural⁵⁴⁷ studies of lanthanide complexes have been reported and reviewed.⁵⁴⁸ The hydrolytic equilibrium between $[Zr_8Cl_{12}(OH)_{20}(H_2O)_{24}]$ and $[Zr_4Cl_6(OH)_8(H_2O)_{16}]^{6+}$ has also been studied.⁵⁴⁹

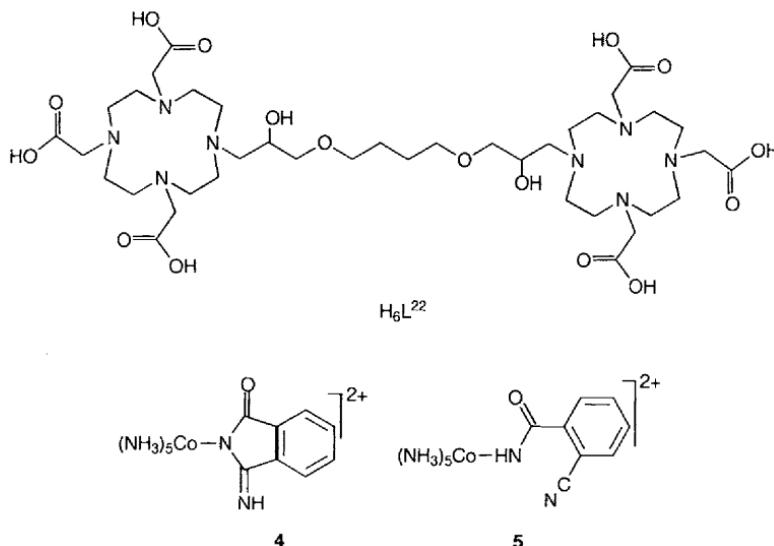
Ligand exchange

The use of variable-pressure NMR spectroscopy to study solvent exchange on transitional-metal ions has been reviewed.⁵⁵⁰ Water exchange on $[Ir(H_2O)_6]^{3+}$ takes place via an I_a mechanism and on $[Ir(OH)(H_2O)_5]^{2+}$ via an I process, with the observed⁵⁵¹ overall rate constant taking the form, $k = k_1 + k_2/[H^+]$. Exchange is extremely slow, with a residence time corresponding to k_1^{-1} of ca. 300 years. Separate pathways for water exchange at sites *cis* and *trans* to the bridging OH are reported⁵⁵² from ¹⁸O and ¹⁷O studies of $[(H_2O)_4Rh(\mu-OH)_2Rh(H_2O)_4]^{4+}$. The bridging OH is inert to exchange. Theoretical approaches have been used to model transition states and intermediates for water exchange in first-row transition-metal hexaaqua complexes,⁵⁵³ in $[Pd(H_2O)_4]^{2+}$, $[Pt(H_2O)_4]^{2+}$ and *trans*- $[PtCl_2(H_2O)_2]^{2+}$,⁵⁵⁴ in $Na(aq)^+$,⁵⁵⁵ in $[M(H_2O)(bipy)Cp']^{2+}$ ($M = Co$, Rh or Ir)⁵⁵⁶ and the second co-ordination sphere of $[Cr(H_2O)_6]^{3+}$.⁵⁵⁷ An experimental value for the exchange rate in the latter leads to a lifetime of 128 ps for one H₂O compared with 144 ps from a simulation. Experimental and theoretical approaches have been compared for water exchange on $[Ln(H_2O)_n]^{3+}$ ($Ln = Nd$, Sm or Yb).⁵⁵⁸ An I_d mechanism via an eight-co-ordinate square-antiprism transition-state is preferred for $Ln = Nd$, $n = 9$. For $Ln = Yb$, $n = 8$, an I_a process via a tricapped trigonal-prism transition-state is proposed. The need for rapid water exchange in effective Magnetic Resonance Imaging contrast-agents motivates a continuing focus on exchange processes involving lanthanide, particularly Gd^{III}, complexes.⁵⁵⁹⁻⁵⁶² Water exchange on the Gd^{III} complex with $[L^{22}]^{6-}$ is shown to have a ΔV^* of $+0.5 \pm 0.2 \text{ cm}^3 \text{ mol}^{-1}$, pointing to an interchange process.⁵⁶⁰ The ΔV^* values for water exchange, $+3.1 \pm 0.2$ to $+7.7 \pm 0.5 \text{ cm}^3 \text{ mol}^{-1}$, in related dendrimer complexes, are consistent with I_d processes.⁵⁵⁹ Water exchange in $[Mo_3NiS_4(H_2O)_{10}]^{4+}$ was too slow⁵⁴⁸ for direct determination by NMR spectroscopy. Solvent-exchange kinetics have been measured for *trans*- $[Os(\eta^2-H_2)(en)_2(solv)]^{2+}$ ($solv = H_2O$ or MeCN)⁵⁶³ for $[Pt(CH_3)(Me_4en)(dmsO)]^{+}$ ⁵⁶⁴ and *cis*- and *trans*- $[Pt(CH_3)Cl(dmsO)_2]^{4+}$.⁴⁹⁵ The kinetics of ¹⁷O loss from ¹⁷O-labelled $[CrO_4]^{2-}$,⁵⁶⁵ and of related processes involving catalysis by tellurate,⁵⁶⁶ have been described. Oxygen- and vanadium-exchange for $[V_3O_{10}]^{5-}$ and $[V_4O_{13}]^{6-}$ is thought to proceed by cyclic intermediates.⁵⁶⁷ Other exchange processes have been studied, involving Bu^tOH-exchange with Sn(OBu^t)₄,⁵⁶⁸ *n*-propylamine with $[Co(NH_2Pr^n)_6]^{2+}$,⁵⁶⁹ Cl⁻ with $[PdCl_4]^{2-}$,⁵⁷⁰ X⁻ with allyltin halides,⁵⁷¹ nitrile exchange with Ir-amidine complexes,⁵⁷² CF₃CO₂⁻ in $[Tl(tpp)(O_2CCF_3)]$,⁵⁷³ and intramolecular arene exchange in (phosphinoalkyl)arene-Rh^I complexes.⁵⁷⁴

Reactions of co-ordinated ligands and linkage isomerism

Redox-induced ONO to NO₂ linkage-isomerism in *cis*-[Ru(NO)(ONO)(bipy)₂]²⁺ results in oxygen transfer between NO and ONO whereas thermal-induced isomerism does not.⁵⁷⁵ Decomposition of *trans*-[Ru^{III}(NO₂)(PR₃)(terpy)]²⁺ is believed to proceed *via* nitro–nitrito linkage isomerism.⁵⁷⁶ X-Ray studies⁵⁷⁷ on a metastable form of [Ru(NO₂)₄(NO)(OH)]²⁻ suggest that NO is bound *via* oxygen. Interchange between linkage isomers of [Re(CO)L(¹⁵N¹⁴N)Cp*] [L = CO, PMe₃ or P(OMe)₃] is non-dissociative and intramolecular *via* a $\eta^2\text{-}{}^{15}\text{N}^{14}\text{N}$ intermediate.⁵⁷⁸ Nitrogen monoxide release from the decomposition of [Cu{Et₂N(N₂O₂)₂}][9]ane(NⁱPr)₃]⁺ in aqueous acetic acid appears not to involve prior dissociation of the Et₂N(N₂O₂)⁻ ligand.⁵⁷⁹ Thermolysis of [Ta(CH₃)(NN¹⁵NPh)Cp₂] in the presence of a ¹³CH₃⁻¹⁴N₃Ph analogue leads⁵⁸⁰ exclusively to [Ta(CH₃)¹⁵NPh)Cp₂]. Loss of N₂ is accelerated by electron-withdrawing substituents on the phenyl ring. The complex [{Mo(NRR')₃}₂(μ -N₂)] decomposes to [MoN(NRR')₃] unimolecularly.⁵⁸¹ The barrier to interconversion between [{Cu(NH₃)₃}₂(μ - η^2 : η^2 -O₂)]²⁺ and [{Cu(NH₃)₃}₂(μ -O₂)]²⁺ is estimated⁵⁸² from *ab initio* methods to be low. The cations [Os(qnt)(CO)(η^2 -H₂)(PPh₃)₂]⁺ and [OsH(CO)(PPh₃)₂(Hqnt)]⁺ are in tautomeric equilibrium at low temperatures.⁵⁸³ Metallotropic processes are seen in *fac*-[ReX(CO)₃(bmmpp)] (X = Cl, Br or I)⁵⁸⁴ in related complexes of 2,4,6-tris(pyrazolyl)pyrimidine,⁵⁸⁵ 2,4,6-tris(pyrazol-1-yl)-1,3,5-triazines,⁵⁸⁶ and in terpy-Pd^{II}, Pt^{II}⁵⁸⁷ and -Ru^{II}⁵⁸⁸ complexes. The view that such processes are associative has been challenged⁵⁸⁹ and defended.⁵⁹⁰ Potentially tridentate 7-substituted 8-hydroxyquinoline-5-sulfonate ligands first complex rapidly with [M(H₂O)₆]³⁺ (M = Ga⁵⁹¹ or Al^{442,443}) *via* the N and O of the 8-hydroxyquinoline. A slower stage follows in which a phenolic group attached *via* position 7 co-ordinates. The cation [Mo₃{Pd(NCS)}S₄(H₂O)₉]⁴⁺ undergoes a slow isomerisation, from Pd-NCS to Pd-SCN.⁴⁵⁹

Reactions of Co^{III} complexes continue to be a rich source of novel reactions of co-ordinated ligands. The Hg²⁺-catalysed aquation of *cis*-[CoCl(β-Ala)(en)₂]²⁺ has also been studied.⁴⁸⁸ (Related studies⁴⁶⁹ on Cr^{III} complexes have already been mentioned). Sulfinate-linkage isomers, *trans*- and *cis*-[{¹⁵N,O(S)}-Co{OS(O)CH(CH₃)CO₂-O,O}(tren)]⁺ are among the products from the photolysis or *trans*-[{¹⁵N,S}-Co{S(O)₂CH(CH₃)CO₂-O,S}(tren)]⁺.^{592,593} Kinetics of the equilibria between Co^{III}-S and -C bonded forms of the ligands dathid⁵⁹⁴ and aeaps⁵⁹⁵ point to the intermediacy of carbanions. The carbanion from aeaps reacts with water 170 times faster than its capture by Co^{III}; that from dathid 270 times. Hydrogen-deuterium exchange on C² of 1-methylimidazole is enhanced 10²–10³-fold by co-ordination to Pt^{II}.⁵⁹⁶ The hydrolyses of a series of mono- and di-nitrile complexes of [Co^{III}-(NH₃)₅]³⁺ have been investigated mechanistically.⁵⁹⁷ For example, amido-N-co-ordinated 2-cyanobenzamide cyclises in base to give [Co^{III}(NH₃)₅L]²⁺ **4** (L = 1-oxo-3-iminoisoindolino-*endo*-N) which is hydrolysed in acid following protonation of the exo-imine. In acid, **5** (L = NHC(O)C₆H₄CN-2) is protonated on the amido-O followed by linkage-isomerisation to the nitrile-bound form. Hydrolysis of MeCN by NbCl₅,⁵⁹⁸ hydration of RCN by [RuCl(H)(CO)(PPh₃)₃],⁵⁹⁹ the effect of intramolecular hydrogen bonding on the rates of η^1 -N-bound imine formation from amines and [IrH₂(pyCHO-*N*,*O*)(PPh₃)₂]⁺,⁶⁰⁰ ring closure of a Cu^{II}-β-aminoketone intermediate {from Me₂CO and [Cu(en)₂]²⁺} to give [Cu^{II}(*trans*-[14]dieneN₄)]²⁺,⁶⁰¹ enamine



addition to NO in *trans*-[RuCl(NO)(py)₄]²⁺,^{602,603} template Schiff-base formation from amino acids and ninhydrin in the presence of Cd²⁺⁶⁰⁴ or Cu²⁺⁶⁰⁵ have all been studied.

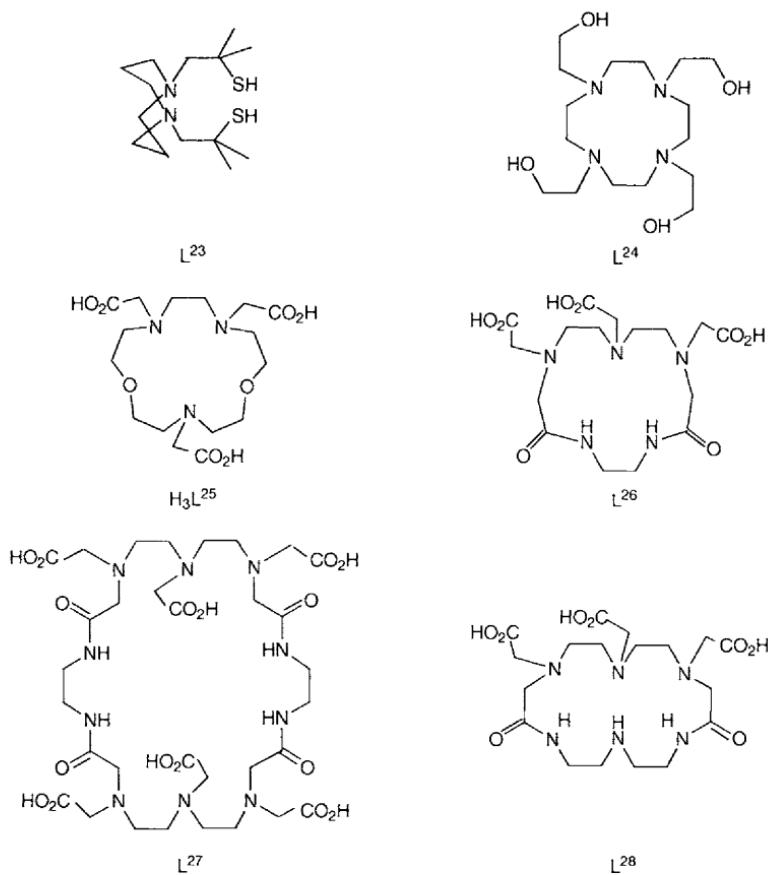
Two-centre metal-ion catalysis of acyl- and phosphoryl-transfer processes^{606,607} and the role of lanthanides in phosphate hydrolysis⁶⁰⁸ have been reviewed. Phosphate ester hydrolysis is accelerated by complexes of nickel(II), cadmium(II), lead(II),^{609,610} zinc(II),^{610,611-614} magnesium(II),⁶¹³ copper(II),^{610,613,615-618} lanthanide(III),^{613,619-623} and other metals^{611,624,625} and by dinuclear complexes of zinc(II),^{626,627} lanthanide(III),⁶²⁸ and cobalt(III),^{629,630} peptide hydrolysis by mono-,⁶³¹⁻⁶³⁴ bi-,⁶³⁵ and poly-nuclear⁶³⁶ palladium(II) complexes, and ester hydrolysis by mono-^{637,638} and di-nuclear⁶³⁹ complexes of zinc(II) and by uranium(VI),⁶⁴⁰ palladium(II),⁶⁴¹ and platinum(II),⁶⁴¹ complexes. The rate of hydrolysis of [Co^{III}L(en)₂]ⁿ⁻, decreases along the series, L = PO₄³⁻ > P₂O₇⁴⁻ > P₃O₁₀⁵⁻.⁶⁴² DNA- and RNA-hydrolysis by metal complexes has been reviewed⁶⁴³ and mechanistic aspects studied, including cleavage by lanthanide complexes,^{628,644-648} by Cu^{II}^{649,650} in combination with H₂O₂ (which on its own, as cetyltrimethylammonium hydroperoxide, promotes phosphate hydrolysis⁶⁵¹), by other copper(II),^{652,653} cobalt(III),⁶⁵⁴ and other metal⁶⁵⁵ complexes. Related processes involving nucleosides and oligonucleotides have also been described.^{613,614,623,656-661}

Second-order rate constants for the quaternisation of 1-(2-pyridyl)alkyl-Pt^{II} and -Pd^{II} complexes by organic halides are higher than the analogous 1-(4-pyridyl) alkyl compounds.⁶⁶² Steric effects on the addition of imidazoles to [Fe(CO)₃(1,5- η -dienyl)]⁺ are less significant than for related reactions of pyridines.⁶⁶³ Incorporation of the isotope label from reaction of ¹⁸O₂-¹⁶O₂ mixtures with [Ni^{II}(L²³)] into the mono- and the bis-sulfenato complexes shows that the initially formed persulfoxide adduct gives the mono- and bis-adducts by two distinct pathways, one intermolecular, the other intramolecular.⁶⁶⁴ The complex *cis*-[Pt(S₃O)(PPh₃)₂] is a catalytically active intermediate resulting from the reaction of *cis*-[Pt(SH)₂(PPh₃)₂] with SO₂,

mimicking a step in the Claus process for hydrodesulfurisation,
 $2\text{H}_2\text{S} + \text{SO}_2 \rightleftharpoons 3/8\text{S}_8 + \text{H}_2\text{O}$.⁶⁶⁵

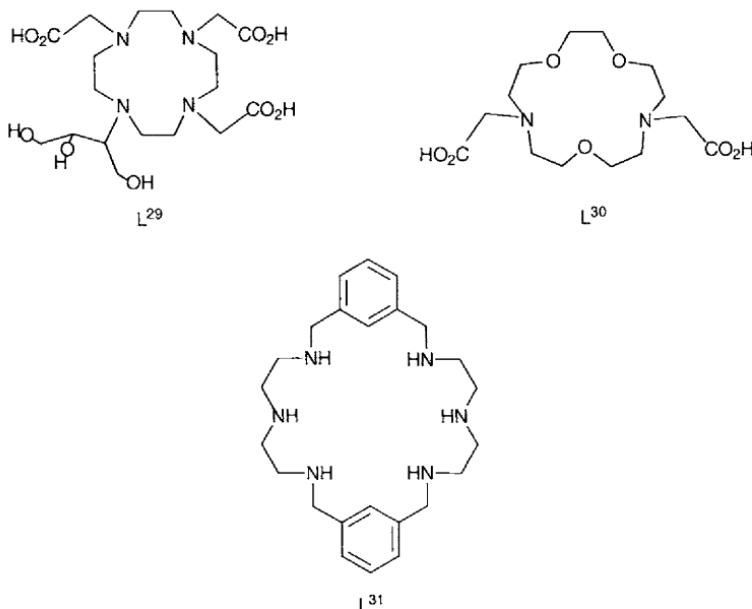
Metal-ion complexation with macrocycles

Intermolecular exchange of L^{24} in the square anti-prismatic $[\text{Na}(\text{L}^{24})]^{+}$ in MeCN and propylene carbonate occurs more slowly⁶⁶⁶ than enantiomerisation of the complex, whereas for $[\text{K}(\text{L}^{24})]^{+}$ enantiomerisation proceeds mainly via intermolecular exchange. Related studies on $[\text{M}(\text{tmec12})]^{+}$ ($\text{M} = \text{Li}, \text{Na}$ or K) in methanol⁶⁶⁷ and for $[\text{Lu}(\text{dota})(\text{H}_2\text{O})]^{-}$ ⁶⁶⁸ have also been described. The role of pendant groups is probed in studies which demonstrate⁶⁶⁹ that incorporation of M^{2+} ($\text{M} = \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}, \text{Cd}, \text{Hg}$ or Pb) into the tetraaza ring of [14]aneN₄ can be facilitated by just two 2-hydroxyethyl groups attached to adjacent nitrogens. The cation $[\text{Ni}(\text{dmf})_6]^{2+}$ and $\text{Me}_2[14]\text{aneN}_4$ react⁶⁷⁰ initially to form a partially co-ordinated complex which rapidly converts to first one and then a second intermediate in which all four N atoms are bound. This then rearranges further to give *trans*- $[\text{Ni}(\text{Me}_2[14]\text{aneN}_4)(\text{dmf})_2]^{2+}$. Rapid initial formation in aqueous solution of a complex between M^{2+} ($\text{M} = \text{Co}, \text{Ni}, \text{Cu}$ or Zn) and H_3L^{25} involves⁶⁷¹ partial ligand co-ordination followed by rate-determining product formation, in processes in which $[\text{HL}^{25}]^{2-}$ is kinetically the most important ligand species. In related studies, Gröss and Elias show⁶⁷² that Ni^{2+} rapidly equilibrates with $\text{H}_2\text{tcta}^{-}$ to form the intermediate $[\text{Ni}(\text{Htcta})]$. This forms $[\text{Ni}(\text{tcta})]^{-}$ in a slow first-order rearrangement. Complexation of Ni^{2+} by polycarboxylate ions has also been studied⁶⁷³ using pressure- and temperature-jump relaxation methods. Lanthanide(III) complexes of cy(dtpa-en) (L^{26}) and cy(dtpa-en-dtpa-en) (L^{27}), exist, respectively, in two and four isomeric forms. In the latter case, two types of exchange processes are seen,⁶⁷⁴ one very rapid which does not involve decomplexation and the other, a slower process, for which decomplexation is necessary. The ligand $\text{H}_3\text{dtpa-dien}$ (L^{28}) rapidly forms an equilibrium mixture of two species on reaction with Eu^{3+} , with one more slowly transforming into the other, $[\text{Eu}(\text{Hdtpa-dien})(\text{H}_2\text{O})]^{+}$, in parallel proton-dependent and -independent processes.⁶⁷⁵ More complex processes involving incorporation of Cu^{2+} into a series of bis-macrocycles⁶⁷⁶ and processes involving the second co-ordination sphere of metal complexes^{677,678} have also been investigated. The rate of ligand exchange between $[\text{Gd}(\text{do3a-b})]^{+}$ ($\text{do3a-b} = \text{L}^{29}$) and Eu^{3+} is directly dependent on $[\text{H}^{+}]$ and proceeds⁶⁷⁹ via rate-determining rearrangement and dissociation of the monoprotonated complex. pH and $[\text{dtpa}]$ dependence of the kinetics of displacement of ox^{2-} by dtpa from $[\text{Cr}(\text{ox})_3]^{3-}$ points⁶⁸⁰ to two parallel pathways. Related ligand-exchange reactions of $[\text{Cr}(\text{en})_3]^{3+}$ with cddta⁶⁸¹ and $[\text{Cu}^{\text{II}}(\text{Gly-Gly})_2]$ with $[\text{X}] \text{aneN}_4$ ($X = 14-16$)⁶⁸² have been reported. Dissociation of L^{3-} , $\text{H}_3\text{L} = \text{H}_3\text{notmp}$, from its complex with Mg^{II} is dominated⁶⁸³ by a proton-assisted pre-equilibrium followed by rate-determining dissociation of HL^{2-} . Dissociation of M^{II} ($\text{M} = \text{Ni}, \text{Zn}$ or Cd) from complexes with $[\text{L}^{30}]^{2-}$ and related ligands,⁶⁸⁴ of Cu^{II} from the mono- and di-nuclear complexes with L^{31} ,⁶⁸⁵ and of Cu^{II} and Ni^{II} from complexes with hexacyclen⁶⁸⁶ have all been described. The rate constant for the loss of ligand, L , $\{\text{L} = \text{R}_4[14]\text{aneN}_4, \text{R} = \text{H}, \text{Me}, \text{Et}$ or Pr^{n} $\}$ from $[\text{NiL}]^{2+}$ is 50–120-fold smaller than for $[\text{Ni}(\text{HL})]^{3+}$ with both rate constants relatively insensitive to the nature of R .⁶⁸⁷ The displacement of L by cyanide ion is also described. Complexation kinetics of the cryptand 221 with Ca^{2+} ,⁶⁸⁸ of ligand interchange for the complex of 18-crown-6 with Ba^{2+} ,⁶⁸⁹ and with $\text{La}^{3+}, \text{Ca}^{2+}$,

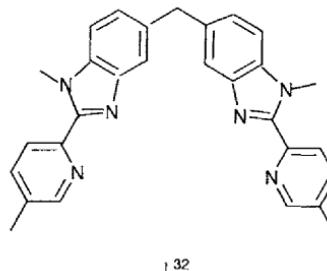


Pb²⁺ and Ba²⁺⁶⁹⁰ have been reported, as have studies on the rapid exchange⁶⁹¹ involving a series of 1:1 complexes of K⁺ and bis(benzo-crown ether) ligands linked by HN(CH₂)_nNH bridges ($n = 2-6, 8$ or 10). The rate of aquation of [Ni(bptan)]²⁺ follows⁶⁹² the rate law rate = $k_1[H^+][Cl^-][NiL]/(1 + k_2[Cl^-])$ in the presence of chloride, whereas for the highly inert species, [Ni(noda)(H₂O)], the rate, given by $k_1[H^+][NiL]/(1 + k_2[H^+])$, is independent of [Cl⁻]. The slow rate and associated activation energy (199 kJ mol⁻¹) for irreversible topomerisation from [Th^{IV}(heha)]²⁻ of C₂ symmetry to a topomer of S₆ symmetry involves⁶⁹³ complete reorganisation of the chelate, a process not seen in the related U^{IV} complex.

In a fascinating report, a 35-nucleotide RNA has been characterised⁶⁹⁴ which catalyses the incorporation of Cu^{II} into mesoporphyrin IX. Cobalt(II) removal by CN⁻ from dinuclear cobalt(II) substituted derivatives of *Carcinus maenas* haemocyanin involves⁶⁹⁵ a rapid pre-equilibrium cyanide addition to the protein, followed by slow cyanide-assisted loss of the metal ions in two steps. Complexation kinetics of [XO₄]ⁿ⁻ (X = Mo^{VII}, W^{VII}, V^V or As^V) with purple acid phosphatase in the Fe^{II}Fe^{III} form have also been reported.⁶⁹⁶ Iron release from a bacterial ferritin was measured by the



reduction of iron with $S_2O_4^{2-}$ and the complexation of the resulting Fe^{2+} with bipy. 91% of the iron, believed⁶⁹⁷ to be bulk iron, reacted more swiftly than the remaining 9%. The latter is thought to be attached to the interior of the protein shell. Related studies have been reported.^{698,699} The kinetics of metal-ion incorporation into azaporphyrins have been reviewed.⁷⁰⁰ The porphyrin, H₂tpp, in MeCN reacts rapidly with Cu^{II} to give a 'sitting-atop' complex, $[Cu(H_2tpp)]^{2+}$, with the activation process requiring⁷⁰¹ porphyrin-ring deformation. Addition of a base results in deprotonation of porphyrin nitrogens and Cu^{II} incorporation into the N₄ ring. Kinetics and activation parameters of Hg^{II} complexation with *N-p*-nitrobenzyl-5,10,15,20-tetrakis(4-sulfonatophenyl)porphyrin suggests the reaction of Hg(OH)₂ with a protonated ligand.⁷⁰² The kinetics of metal-ion incorporation into tpp²⁻, a C⁴-capped porphyrin, a cofacial bis-porphyrin and a spheroidal bis-porphyrin in the mixed solvent system toluene-acetic acid-methanol have been described.⁷⁰³ Reactions of $[M(H_2O)_6]^{2+}$ ($M = Mn, Fe, Co, Ni$ or Cu) with tetrakis(4-sulfonatophenyl)mesoporphyrin are reported⁷⁰⁴ to be factors of 10^7 - 10^{10} slower than the corresponding rate of water exchange. The rate laws⁷⁰⁵ for the solvolysis of $[M(N\text{-Metpps})]$ ($M = Zn, Cu, Co$ or Ni) in aqueous acetic acid have a term which is first order in complex and $[H^+]$, whereas only for $M = Cu$ or Zn is there an additional term revealing a dependence on both $[H^+]$ and $[OAc^-]$. Catenane formation from two molecular rings^{706,707} proceeds by two sequential ligand exchanges between the two rings (following Pd-N cleavage) which occur at the same time as the rings twist around each other, rather than by the conventional process which involves initial dissociation of one ring, threading of the open-chain species through the second ring followed by reconnection. Because of the need to break or stretch several Co-N bonds simultaneously, racemisation of the dinuclear Co^{II} complex from L³² occurs with a rate constant some 10^6 -fold



smaller⁷⁰⁸ than for the analogous mononuclear complex $[Co(phen)_3]^{2+}$. Qualitatively, the rates of self-assembly of pentanuclear double helicates from Cu^1 and three oligobipyridine ligands are dependent⁷⁰⁹ on the steric bulk of the substituent in the 4,4'-positions of the bipy units. Related studies of metal-complexed catenanes,^{710,711} calix[4]arenes⁷¹² and knots⁷¹³ have been studied and material related to self-assembly reviewed.⁷¹⁴⁻⁷¹⁸

Main-group reactions

Phenyl boronic acid complexes ethane-1,2-diol *via* $BPh(OH)_3^-$, with OH^- addition to $BPh(OH)_2$ being much slower than diffusion controlled.⁷¹⁹ Values of ΔV^* and other parameters for the carbonic anhydrase II-catalysed hydration of CO_2 and dehydration of HCO_3^- suggest⁷²⁰ that nucleophilic attack of Zn^{2+} -bound OH^- on CO_2 is associative and that substitution of H_2O by HCO_3^- on Zn^{2+} is dissociative in character. Carbon dioxide release and proton transfer are concerted during the dehydration process. Other studies on CO_2 have also been reported.⁷²¹⁻⁷²³ Kinetics at 725 K and 335 bar for the decomposition of aqueous urea to CO_2 and NH_3 ⁷²⁴ and of its alkaline hydrolysis in 0.5–3.0 M NaOH *via* OCN^- ⁷²⁵ have been reported. Hydrolysis of $HC(O)Cl$ (from vinyl chloride and O_3) to HCO_2H and HCl competes with decay to CO and HCl only at high pH.⁷²⁶ Computational methods⁷²⁷ suggest that CCl_2 reacts with H_2O to give $CHCl_2OH$. This eliminates HCl to give $HC(O)Cl$, reported⁷²⁶ to have $t_{\frac{1}{2}} < 20 \mu s$. Rapid intramolecular fluoride exchange between silicons occurs synchronously with ring inversion in fluorinated di-⁷²⁸ and tri-silacyclohexane⁷²⁹ anions. Dihydrogen loss from $SiH(OEt)_3$ is promoted by tartaric acid *via* an initial transesterification.⁷³⁰ Addition of water and alcohols to $Ph_2Si=CH_2$,⁷³¹ of phenols to $Me_2Si=SiMe_2$,⁷³² and the alcoholysis of silanols⁷³³ have been studied.

Kinetics and isotope studies of the decomposition of aqueous NH_4NO_2 point to⁷³⁴ pre-equilibrium formation of N_2O_3 from HNO_2 followed by rate-determining attack of N_2O_3 on NH_3 . Detailed analysis of the decomposition of NH_2NO_2 (an ‘honorary’ organic molecule⁷³⁵!) shows that in dilute acid the reaction is base catalysed. An acid-catalysed process appears in more concentrated acid.⁷³⁵ Decomposition of $RNHNO_2$ to N_2O and ROH is an acid-catalysed S_N2 process involving the aci-nitro tautomer reacting with H_2O at low acidities and HSO_4^- at high.⁷³⁶ Nitrogen exchange between NO and aqueous HNO_3 (> 1.5 M) involves N_2O_3 .^{737,738} Hydrolysis of nitrosoureas, $RNHCO(N(R)NO$, in base involve rate-determining decomposition of the conjugate base.⁷³⁹ Related nitrosations⁷⁴⁰⁻⁷⁴² have also been studied. The kinetics of nitrous acid-catalysed oxidation of SCN^- in nitric acid are controlled by rate-determining N_2O_4 formation.⁷⁴³ The red species formed in acidic HNO_2-SCN^-

solution is confirmed to be ONSCN . Nitric oxide reacts with amines or thiols in the presence of O_2 according⁷⁴⁴ to the stoichiometry: $4\text{NO} + \text{O}_2 + \text{EH} \rightleftharpoons 2\text{ENO} + 2\text{NO}_2^- + 2\text{H}^+$ ($\text{E} = \text{RS}$ or $\text{RR}'\text{N}$). Formation of ONOONO is rate-determining and gives NO_2 and N_2O_3 . The rate of NO autoxidation is shown to be unchanged by the presence of the amine or thiol and it was concluded that S-nitrosothiols cannot serve as carriers of NO *in vivo* (see ref. 745). Nitrogen monoxide formation from S-nitrosothiols (and the role of Cu^{2+} in this process)^{746–748} and from thionitrate esters⁷⁴⁹ have been reported. Exchange of the tripeptide glutathione, GSH, with complexed ligand in $[\text{Bi}(\text{GS})_3]$ is slow at low pH and more rapid at physiological pH, observations which may be relevant to the pharmacology of bismuth drugs.⁷⁵⁰

The rates of the forward and reverse reactions $\text{OH}^- + \text{OH}^- \rightleftharpoons \text{O}^- + \text{H}_2\text{O}$ have been measured and lead to values of $\text{pK}(\text{OH})$ of 12.0 ± 0.2 and $\text{pK}(\text{OD})$ of 12.6 ± 0.2 .⁷⁵¹ Sulfur reacts with RS^- in parallel pathways⁷⁵² involving oxidation, giving RSSR and S_3^{2-} , and nucleophilic attack, giving RS_x^- , with a key equilibrium $2\text{RS}_4^- \rightleftharpoons \text{RSSR}$ and S_3^{2-} being studied. Selenium-77 NMR spectroscopic studies suggest the following stoichiometry when selenous acid is reduced by thiosulfate: $\text{H}_2\text{SeO}_3 + 4\text{S}_2\text{O}_3^{2-} + 4\text{H}^+ \rightleftharpoons \text{Se}(\text{S}_2\text{O}_3)_2^{2-} + \text{S}_4\text{O}_6^{2-} + 3\text{H}_2\text{O}$, with S-S bond formation occurring by direct attack at a Se-linked thiosulfate.⁷⁵³ The kinetics of the photochemical decomposition of XeO_4 have been reported.^{754,755}

Solvent and other medium effects

Relevant studies have been described in previous sections.^{105,169,485,487,537} Photoinduced ring closure for $[\text{Fe}(\text{CN})_5(\text{tn})]^{3-}$ shows a sharp decrease in quantum yield with increasing viscosity of water-glycerol mixtures.⁷⁵⁶ The role of ether phosphine ligands ('intramolecular solvents') on the reactivity of 'pseudo-underco-ordinated' complexes of ruthenium has been reviewed.⁷⁵⁷ Aquation kinetics in mixed solvents have been reported for *cis*- and *trans*- $[\text{CoCl}(\text{NO}_2)(\text{en})_2]^+$,⁷⁵⁸ *trans*- $[\text{CoCl}_2(\text{N-Meen})]_2^+$,⁷⁵⁹ *trans*- $[\text{CoCl}_2(\text{NH}_3)_4]^+$,⁷⁶⁰ and $[\text{Co}(\text{NO}_3)_3(\text{NH}_3)_5]^{2+}$.⁷⁶¹ Solvent effects on electron transfer in $[\text{NH}_3]_4\text{Co}^{\text{III}}(\mu\text{-pzCO}_2)\text{Fe}^{\text{II}}(\text{CN})_5]^{-}$,⁷⁶² the consequences of binding of *cis*- $[\text{Co}(\text{en})_2(\text{H}_2\text{O})]^{3+}$ to a sulfonated polymer on its reactivity with $[\text{Fe}(\text{edta})]^{2-}$,⁷⁶³ and superoxide disproportionation⁷⁶⁴ have all been described. The difference in reactivity of e_{solv}^- with NH_4^+ in water and butanol is attributed to the different solvation structure around the cation.⁷⁶⁵ Micellar effects on the replacement of H_2O in Pd^{II} complexes,^{766,767} on complexation of Cu^{II} ,⁷⁶⁸ on anation of $(\alpha,\beta)\text{S}-[\text{Co}(\text{OH})(\text{tetren})]^{2+}$ by SO_3^{2-} ,⁷⁶⁹ on aquation of $[\text{Fe}(\text{Ph}_2\text{phen})_3]^{2+}$,⁷⁷⁰ and $[\text{Fe}(\text{Me}_4\text{phen})_3]^{2+}$,⁷⁷¹ and on the reaction of $[\text{IrCl}_6]^{2-}$ with $\text{S}_2\text{O}_3^{2-}$,⁷⁷² have been described. Micelle formation from Co^{III} ,⁷⁷³ Ni^{II} (and its role in ester cleavage^{774,775}), Cu^{II} ,⁷⁷⁶ and organometallic⁷⁷⁷ complexes have been investigated. Micellar effects have been studied on electron transfer between Co^{III} and Fe^{II} in *cis*- $[\text{Co}(\text{NH}_3)_4(\text{H}_2\text{O})_2]^{3+}$ and $[\text{Fe}(\text{CN})_6]^{4-}$,⁷⁷⁸ and in the ion pair $[\text{Co}(\text{NH}_3)_5(\text{L})]^{3+} \cdot [\text{Fe}(\text{CN})_6]^{4-}$ ($\text{L} = N\text{-cyanopiperidine}$),^{779,780} on the oxidation of $[\text{Co}(\text{bipy})_3]^{2+}$ by $[\text{Fe}(\text{bipy})_3]^{3+}$,⁷⁸¹ of $[\text{Fe}(\text{phen})_3]^{3+}$ by $[\text{CrO}_4]^{2-}$,⁷⁸² and $[\text{Ru}(\text{NH}_3)_5(\text{pz})]^{2+}$ by $[\text{Co}(\text{ox})_3]^{3-}$,⁷⁸³ The reaction of $[\text{Fe}(\text{CN})_4(\text{bipy})]^{2-}$ with $\text{S}_2\text{O}_8^{2-}$ in a water-in-oil microemulsion has also been reported.⁷⁷² Second-order rate constants for the reaction with CO of $[\text{M}(\text{CO})_5\text{L}]$ in supercritical L ($\text{L} = \text{Kr, Xe or CO}_2$) follow the order $\text{Kr} > \text{Xe} \cong \text{CO}_2$.⁷⁸⁴ The effect of solvent density on the dissociative substitution of CO in $[\text{W}(\text{CO}_2)(\text{CO})_5]$ was examined. Iodine recombination in supercritical argon has been probed on the femtosecond

time-scale.⁷⁸⁵ Metal-complex-catalysed hydrogenation of CO₂ to formic acid in supercritical CO₂⁷⁸⁶ and reaction of [Ir(CO)₂Cp*] with H₂ and C₂H₆ in supercritical CO₂ and supercritical C₂H₆⁷⁸⁷ have been studied. High-pressure photoacoustic calorimetry has been used⁷⁸⁸ to follow the formation of [Cr(CO)₂L(η -C₆H₆)] from L (L = H₂ or N₂) and [Cr(CO)₃(η -C₆H₆)]. Complexation kinetics between Ni^{II} and 5-octyloxymethyl-8-quinolinol at the hexane–water interface⁷⁸⁹ and related studies⁷⁹⁰ have been reported. Mention has already been made of the acceleration of chemical processes by freezing.³¹² Heterogeneous processes involving HNO₂, H₂SO₄, HCl^{791,792} and ClO₂^{793,794} at the surface of ice and sea-salt aerosols⁷⁹⁵ relevant to the trace chemistry of the atmosphere have been studied mechanistically. The effects of microwaves,⁷⁹⁶ ultrasound⁷⁹⁷ and magnetic fields^{798,799} have also been described.

4 Organometallics

σ -Bonded organotransition-metal compounds

The possible evolutionary significance of the axial base to the relative importance of Co–C heterolysis and homolysis in coenzyme B₁₂ (adocobalamin, AdoCbl) has been investigated by Finke and co-workers.^{800,801} Variation with the basicity of series of axial bases of the rate of Co–C bond thermolysis in adocobinamide (AdoCbi⁺) in anaerobic ethylene glycol, and the relative importance of homolysis and heterolysis, reveals that the rate of base-on homolysis is invariant for 4-pyX (X = H or NH₂) whereas the rate constant for base-on heterolysis increases 17-fold from X = H to NH₂. The selection of 5,6-dimethylbenzimidazole as axial base in AdoCbl may be a consequence of its limitation of Co–C heterolysis relative to the more biologically relevant homolysis. The expected inverse dependency on solvent viscosity⁸⁰² of the thermal homolysis rate constants for α - and β -(cyanomethyl)Cbi⁺ point to the importance of diffusional effects. The ratio of in-cage recombination to diffusional separation, k_c/k_d , was estimated with $k_c^{\alpha}/k_c^{\beta} = 2.6 \pm 0.6$ revealing a surprising kinetic preference of the α diasteromer. Corrin ring side chains determine equilibrium composition of α - and β -RCbi⁺⁸⁰³ in the anaerobic reaction of α - or β -alkylcobinamides, RCbi⁺, with R via dialkylcobalt(IV)corrinoids. The ΔS^{\ddagger} values of Co–C homolysis in neopentyl-cobalamin analogue are influenced⁸⁰⁴ by the thermal motions of groups attached to the haptocorrin rings. He and Dowd⁸⁰⁵ suggest that free radicals are not involved in coenzyme B₁₂-dependent carbon-skeletal rearrangements. A kinetic model has been developed^{806–808} to describe the reaction of olefins with [Co(tap)] in the presence of radicals and which involve organocobalt(III) intermediates. Axial ligands (L = py or PEt₃) accelerate the rate of Fe–C homolysis⁸⁰⁹ in [Fe(Me)(oep)L]. Methylation of [Ni^{II}(ctpp)] by MeI leads to C²¹ methylation of the macrocycle via organonickel(II) complexes.⁸¹⁰ A reinvestigation⁸¹¹ of CH₄ formation from MeSCH₂CH₂SO₃[–] and [NiL]_n (H₂L = 1,4,7,10,13-pentaazacyclohexadecane-14,16-dione) has shown the original report to be incorrect. EXAFS studies reveal⁸¹² rapid exchange of R and acac[–] between [NiR(alkene)(acac)] and [Ni(acac)₂] and AlEt₂(OEt) in toluene. The properties of transient σ -bonded organocupper(II) and -(III) complexes have been reviewed.⁸¹³

Negative ΔV^{\ddagger} and ΔS^{\ddagger} and a large deuterium isotope effect suggest⁸¹⁴ that the reaction [PtMe₄(bipy)] + RCO₂H \rightleftharpoons [PtMe₃(O₂CR)(bipy)] + CH₄ is an S_{E2} pro-

cess, with $\log k$ and pK_a for the acid being linearly related. Rates of inversion at the α -C in $[\text{Pt}\{\text{CHX}(\text{SiMe}_3)\}\text{X}(\text{chiraphos})]$ increase in the order $\text{Cl} < \text{Br} < \text{I}$.⁸¹⁵ The thermolysis of *cis*- $[\text{PtR}_2]$ ($\text{R} = \eta^2,\eta^1\text{-pent-4-en-1-yl}$) in aromatic solvents proceeds by reversible β -H elimination.⁸¹⁶ The RH elimination from $[\text{ZrR}\{\text{Bu}'_3\text{Si}(\text{NH})\}_3]$,⁸¹⁷ silane elimination from $[\text{TaCl}\{\text{CH}_2\text{SiMe}_3\}_3\{\text{Si}(\text{SiMe}_3)_3\}]$,⁸¹⁸ silanone generation from $[\text{Pt}(\text{Me})(\text{OTf})(\text{dmpe})]$ and $\text{SiPr}_2(\text{H})\text{OH}$ ⁸¹⁹ and mechanistically interesting intermediates in reactions of $[\text{Ta}(\text{CH}_3)(\text{CH}_2)\text{Cp}_2]$,⁸²⁰ $[\text{Ti}(\text{CH}_3)_2\text{Cp}_2]$,⁸²¹ $[\text{Pd}(\text{SnR}_2)(\text{dppe})]$,⁸²² $[\text{Cr}^{\text{VI}}(\text{CH}_2\text{CMe}_3)_2(\text{=NR}_2)]$,⁸²³ $[\text{W}(\text{=CHPh})(\text{CO})_2\text{Cp}]^-$,⁸²⁴ $[\text{W}(\text{=CHR})(\text{CO})_5]$,⁸²⁵ $[\text{M}=\text{CR}(\text{OR})(\text{CO})_5]$ ($\text{M} = \text{Cr}$ or W),⁸²⁶ $[\text{IrH}(\text{=CH}(\text{OR}))(\text{PMe}_3)\text{Cp}^*]^+$ (evidence for α -H migration),⁸²⁷ $[\text{Os}(\text{NH}_3)_5(\text{L})]^{2+}$ ($\text{L} = \eta^2\text{-vinyl ether}$),⁸²⁸ and $[\text{Fe}(\text{SiMe}_2\text{SiMe}_3)(\text{CO})_2\text{Cp}^*]$ ⁸²⁹ have also been described. The complex *cis*- $[\text{Pt}(\text{CH}_2\text{GeMe}_3)_2\text{L}_2]$ is more thermally labile than the Si analogue, giving *cis*- $[\text{Pt}(\text{Me})(\text{CH}_2\text{GeMe}_2\text{CH}_2\text{GeMe}_3)\text{L}_2]$ ⁸³⁰ by β -alkyl migration. The acid cleavage of $[\text{PtR}(\mu\text{-H})(\mu\text{-dppm})_2\text{MR}']$ ($\text{M} = \text{Pt}$ or Pd),⁸³¹ of $[\text{Mo}(\text{CCBu}')\text{H}_3(\text{dppe})_2]$,⁸³² of $[\text{Mo}(\text{CCBu}')\text{H}_2(\text{depe})_2]$,⁸³³ and $[\text{Mo}(\eta^2\text{-MeCCMe})(\text{dppe})_2]$ ⁸³⁴ have been described and reviewed.⁸³⁵ Theoretical studies of Ti-C⁸³⁶ and Hg-C⁸³⁷ cleavage have been reported.

Hydrolysis of aqueous $[\text{Re}(\text{Me})\text{O}_3]$ follows the rate law, $-\frac{d}{dt}[\text{ReMeO}_3] = k[\text{OH}^-][\text{ReMeO}_3]$ to give CH_4 and $[\text{ReO}_4]^-$.^{838,839} At high concentrations polymerisation occurs. In the presence of H_2O_2 , MeOH is the organic product,⁸³⁹ with the kinetics suggesting either OH^- attack on $[\text{Re}(\text{Me})\text{O}_2(\eta^2\text{-O}_2)]$ or HO_2^- on $[\text{Re}(\text{Me})\text{O}_3]$. Decomposition via $[\text{Re}(\text{Me})\text{O}(\eta^2\text{-O}_2)_2]$ represents a minor pathway. Formation and decay of alkyl radicals from Re-R homolysis in $[\text{ReR}(\text{CO})_3(\text{x-diimine})]$ has already been mentioned.^{98-100,108} Interestingly, MLCT excitation of $[\text{MnR}(\text{CO})_3(\text{R'-dab})]$ leads to CO loss for $\text{R} = \text{Me}$ and Mn-R homolysis for $\text{R} = \text{PhCH}_2$.¹⁰⁰

Carbonyl-insertion and alkyl-migration reactions

Barriers to migratory insertions in carbonyl alkyl, carbonyl acyl, ethylene alkyl and ethylene acyl complexes of the type $[\text{Pd}(\text{R})\text{L}(\text{phen})]^+$ increase in the order R to $\text{CO} < \text{RC(O)} < \text{C}_2\text{H}_4 < \text{R}$ to C_2H_4 . Associated mechanistic studies⁸⁴⁰ suggest that the catalytic resting state in the industrially important copolymerisation of ethylene and CO (also studied theoretically⁸⁴¹⁻⁸⁴⁴) is a carbonyl acyl complex. This is in equilibrium with an ethylene acyl species which undergoes β -acyl migratory insertion to give a metal-bound alkyl which reforms the resting state by rapid reaction with CO. The cation $[\text{Pd}\{(\text{CH}_2\text{CH}_2\text{C}(\text{O})\text{Me})\}(N,O\text{-pyCO}_2\text{Me}-2)(\text{PPh}_3)]^+$, from ethylene insertion into a Pd-acyl bond, has been characterised by X-ray crystallography.⁸⁴⁵ Cavell⁸⁴⁶ has reviewed recent developments in migratory-insertion reactions of M-C bonds and has reported ligand effects on CO insertion processes for $[\text{PdR}(\text{N-O})\text{L}]$.⁸⁴⁷ Reaction of $[\text{Pd}(\text{Me})(\text{OR})(\text{N-N})]$ with CO may lead either to insertion into Pd-C at -25°C or into Pd-O at -60°C .⁸⁴⁸ Rates of CO insertion in a series of $[\text{Pd}(\text{Me})\text{L}(\text{th-metMe})]^n^+$ ($n = 0$ or 1) decrease in the order, $\text{L} = \text{CF}_3\text{SO}_3^- > \text{Br}^- > \text{MeCN} > 2,6\text{-dmpy} > \text{Cl}^- > \text{py}$ ⁸⁴⁹ (see also refs. 850-852). Steric effects of α -diimine ligands, N-N, control $t_{1/2}$ for CO insertion in $[\text{Pd}(\text{Me})\text{Cl}(\text{N-N})]$ complexes.⁸⁵³ The cation $[\text{Pd}(\text{Me})(\text{N-N-N})]^+$ has been studied similarly.⁸⁵⁴ Rates of carbonylation of $[\text{Pt}(\text{OC}_6\text{H}_4\text{X}-4)(\text{triphos})]^+$ to $[\text{Pt}\{\text{C}(\text{O})\text{OC}_6\text{H}_4\text{X}-4\}(\text{triphos})]^+$ follow the order, $\text{X} = \text{F} > \text{Me} > \text{OMe}$,⁸⁵⁵ and proceed via migratory insertion rather

than by nucleophilic attack of RO^- on a co-ordinated CO. Electron-withdrawing groups accelerate the rearrangement of $[\text{Pd}(\text{CH}_3)(\text{CH}_2=\text{CHC}_6\text{H}_4\text{X}-4)(\text{phen})]^+$ to $[\text{Pd}\{\eta^3-\text{CH}(\text{CH}_2\text{CH}_2)(\text{C}_6\text{H}_4\text{X}-4)\}(\text{phen})]^+$ via β - CH_3 migratory insertion.⁸⁵⁶ The exchange, $[\text{Pd}\{\eta^3-\text{CH}(\text{CH}_2\text{CH}_3)(\text{C}_6\text{H}_5)\}(\text{phen})]^+ + \text{styrene} \rightleftharpoons [\text{Pd}\{\eta^3-\text{CH}(\text{CH}_3)(\text{C}_6\text{H}_5)\}(\text{phen})]^+ + (\text{E})\text{-}\beta\text{-methyl styrene}$, was also studied. The importance of agostic interactions in related processes has been noted.^{857,858} Aryl and vinyl migration are proposed for vinyl aryl ketone formation in $[\text{Ru}(\text{R}')(\text{CO})_2(\text{CH}=\text{CHR})\text{L}(\text{L}')]$ complexes.⁸⁵⁹ The complex $[\text{FeR}(\text{CO})_2\text{Cp}]$ reacts with PR'_3 in a pre-equilibrium followed by rate-determining solvent-independent alkyl migration to give $[\text{Fe}\{\text{C}(\text{O}R)\}(\text{CO})(\text{PR}'_3)\text{Cp}]$.⁸⁶⁰ Reaction with CO leads to decomposition of the Rh-Me compound $[\text{RhIr}(\text{CH}_3)(\text{CO})_3(\text{dppm})_2]$, whereas SO_2 gives the IrC(O)Me compound $[\text{RhIr}\{\text{C}(\text{O})\text{CH}_3\}(\text{CO})_2(\mu\text{-SO}_2)(\text{dppm})_2]$.⁸⁶¹ The Rh-I-catalysed process for methanol carbonylation has been reviewed.⁸⁶² Ethene inserts into Ni-C of $[\text{Ni}(\text{R})(\text{N-O})\text{L}]$ via a $[\text{NiR}(\text{C}_2\text{H}_4)(\text{N-O})\text{L}]$ intermediate.⁸⁶³ Insertions of alkynes into Ln-N ^{864,865} bonds or Pt-B ⁸⁶⁶ bonds, allylidene into Rh-O ,⁸⁶⁷ $\text{CH}_2=\text{CHR}$ into Rh-H ,⁸⁶⁸ and pentadienes into Pd-C⁸⁶⁹ bonds have been described. Phenylacetylene inserts into the Pt-Si bond of *cis*- $[\text{Pt}(\text{CH}_3)(\text{SiPh}_3)(\text{PMMe}_2\text{Ph})_2]$ (via rate-determining displacement of PMMe_2Ph)⁸⁷⁰ whereas the *trans* isomer is inert. Interconversion of *trans*- $[\text{RhCl}(\text{FcC}\equiv\text{CSiMe}_3)(\text{PPr}'_3)_2]$ and *trans*- $[\text{RhCl}\{=\text{C}(\text{Fc})\text{SiMe}_3\}(\text{PPr}'_3)_2]$ involves⁸⁷¹ sigmatropic 1,2-migration of SiMe_3 . Reaction of a fluoride-co-ordinated tungsten *o*-metallocycle with hexafluorobut-2-yne gives first a kinetic η^2 -vinyl product with F *trans* to the inserted alkyne and *cis* to both COs.^{872,873} An isomerisation to the thermodynamically-favoured product proceeds via a limiting dissociative mechanism. The process is promoted by CO or $\text{P}(\text{OMe})_3$ by a η^1 -vinyl adduct-forming pre-equilibrium. Carbon dioxide inserts into Ru-H of $[\text{RuH}_2(\text{dmpe})_2]$ to give *cis*- and *trans*- $[\text{RuH}(\text{O}_2\text{CH})(\text{dmpe})_2]$ and the bis(formate) complex,⁸⁷⁴ into the O-O bond of $[\text{RhCl}(\eta^2\text{-O}_2)(\text{PEtPh}_2)_3]$,⁸⁷⁵ and reversibly into W-O of $[\text{W}(\text{OH})(\text{CO})_5]^-$ to give $[\text{W}(\text{O}_2\text{COH})(\text{CO})_5]^-$.⁸⁷⁶ Phenyl-to-oxo migration in $[\text{Re}(\text{Ph})(\text{OTf})(\text{O})\text{Tp}]$ in dmso gives $[\text{ReO}(\text{OPh})(\text{dmso})\text{Tp}]^+$.^{877,878} The transformation $[\text{Mo}\{\eta^2\text{-C}(\text{O})\text{CH}_2\text{SiMe}_3\}(\text{CO})(\text{CNR}')(\text{PMMe}_3)\text{Tp}']$ to $[\text{Mo}\{\eta^2\text{-C}(\text{NR}')\text{CH}_2\text{SiMe}_3\}(\text{CO})_2(\text{PMMe}_3)\text{Tp}']$ involves rate-determining deinsertion of CO to give a seven-co-ordinate Mo-alkyl intermediate.⁸⁷⁹ β -Hydrogen elimination of $\text{PhN}=\text{CH}(\text{Ph})$ from *trans*- $[\text{Ir}\{\text{N}(\text{CH}_2\text{Ph})\text{Ph}\}(\text{CO})(\text{PPh}_3)_2]$ has also been studied.⁸⁸⁰

Ligand-displacement reactions of metal carbonyl and other low-valent compounds

Geminate recombination of $[\text{M}(\text{CO})_5]$ ($\text{M} = \text{Cr, Mo or W}$) and CO {formed on $[\text{M}(\text{CO})_6]$ UV photodissociation} occurs within 300 fs, that is, after only one or two collisions with the solvent cage in alkane solution.⁸⁸¹ Similar studies⁸⁸² on $[\text{M}(\text{CO})_2\text{Cp}^*]$ ($\text{M} = \text{Ir or Rh}$) show that transients decay without CO loss. Photodissociation pathways in $[\text{Mn}_2(\text{CO})_{10}]$ have been studied theoretically.⁸⁸³ Room-temperature UV photochemistry of $[\{\text{Fe}(\text{CO})_2\text{Cp}\}_2]$ in alkane gives $[\text{CpFe}(\mu\text{-CO})_3\text{FeCp}]$ within 10 ps.⁸⁸⁴ New intermediates are also seen. The solvent-cage effect on radical processes resulting from the photolysis of $[\{\text{Mo}(\text{CO})_3\text{Cp}\}_2]$ has been studied⁸⁸⁵ in thf-tetraglyme mixtures of varying viscosity. Thermal reaction of phosphines and phosphites with $[\text{Cp}(\text{CO})_2\text{FeCo}(\text{CO})_4]$ gives either neutral $[\text{Cp}(\text{CO})_2\text{FeCo}(\text{CO})_3\text{L}]$ via a dissociative mechanism or $[\text{Fe}(\text{CO})_2\text{LCp}][\text{Co}(\text{CO})_4]$ by a radical-chain process.⁸⁸⁶ Intramolecular CO exchange between metals in $[\{\text{Mo}(\text{CO})_3\text{Cp}\}_2]$ via

[Cp(CO)₂Mo(μ-CO)Mo(CO)₂Cp]⁸⁸⁷ formation of [Re₂(μ-H)(μ-alkenyl)(CO)₈] from [Re₂(CO)₁₀]⁸⁸⁸ displacement of acetone in [Fe(CO)₂(Me₂CO)Cp]⁺ by thioethers,⁸⁸⁹ and mechanistically-relevant structural studies on [Re(NO)(PPh₃)(η²-O=CHR)-Cp]⁺⁸⁹⁰ have also been reported.

Activation parameters and solvent dependence for the chelate-ring closure process [Mo(CO)₅(N-N)] \rightleftharpoons [Mo(CO)₄(N-N)] + CO (N-N = a series of phen ligands) support an interchange mechanism⁸⁹¹ (see also ref. 756). An associative process leading to a seven-co-ordinate intermediate is preferred for the related process, [Cr(CO)₅(dppm)] \rightleftharpoons [Cr(CO)₄(dppm)] + CO.⁸⁹² The values of ΔV^{*} for the I_d photosubstitutions, [Cr(CO)₄(phen)] + PMe₃ \rightleftharpoons [Cr(CO)₃(PMe₃)(phen)] + CO⁸⁹³ decrease on increasing radiation wavelength from 366 to 546 nm. Replacement of benzylidene acetone (bda) in [Fe(CO)₃(bda)] by bipy occurs⁸⁹⁴ by parallel dissociative and associative pathways. Reaction in heptane of the square-pyramidal [Fe₅C(CO)₁₅] with P donors with small Tolman cone angles, θ, leads initially to reversible formation of [Fe₅C(CO)₁₅L], having a butterfly structure.⁸⁹⁵ The latter loses CO slowly to give [Fe₅C(CO)₁₄L]. Rates of adduct formation are up to 10³-fold slower for [Fe₅C(CO)₁₅] compared with the Ru analogue. Donors with θ > 136° react very much more slowly. The initial step in the reaction of [Ru₃(CO)₉(μ₃-η²-Xpy)]⁻ (X = PhN, MeN or S) with phosphines (L), is an associative attack⁸⁹⁶ on Ru, with fission of the μ-X bridge bond to give [Ru₃(CO)₉L(μ-η²-Xpy)]⁻. The cation [RuL(η⁴-C₅H₄O)Cp]⁺ reacts with PR₃ to give 1,l'- or 1,2-disubstituted ruthenocenes depending on P basicity and L.⁸⁹⁷ Drago and Joerg⁸⁹⁸ have proposed a scale of σ-donor strengths for phosphines. Replacement of one PPh₃ by PMePh₂ or PMe₂Ph in [RuCl(PPh₃)₂(η⁵-C₉H₇)] occurs dissociatively.⁸⁹⁹ The negative ΔS^{*} for chloride displacement by PR₃ from [MnCl(CO)₂Cp']⁻ to give [Mn(CO)₂(PR₃)Cp'] is thought⁹⁰⁰ to involve a solvent-co-ordinated intermediate. trans Co-ordination to Cr(CO)₃ of the benzene moiety of the indenyl ligand in trans-[Cr(CO)₃(ind)Rh(CO)₂] accelerates the associative substitution of CO by bidentate alkenes, L = cod or nbd, via a [Rh(η¹-ind)(CO)₂L] intermediate.⁹⁰¹ An equilibrium between trans-[Cr(CO)₃(μ-η⁶:η³-ind)Rh(CO)₂] and trans-[Cr(CO)₃(μ-η⁴:η⁵-ind)Rh(CO)₂] is proposed. The indenyl complex is ca. 10-fold more reactive than the corresponding Cp complex. Ring-slippage and hapticity-change processes have also been examined for [M(CO)₂L₂(ind)]⁺,⁹⁰² [MX(CO)₂(η⁷-C₇H₆R)]ⁿ⁺,⁹⁰³ [Cr(CO)₃(η⁶-C₅H₃X₂N)],⁹⁰⁴ and [RuCl(NO)(H₂O)Cp]⁺.⁹⁰⁵ Kinetics for substitution of the π donor in [Cr(CO)₃(η⁶-arene)] by L to give [Cr(CO)₃L₃] display a second-order rate law⁹⁰⁶ with lability decreasing along the series naphthalene > thiophene > cycloheptatriene > 2,5-dimethylpyridine. Other arene-exchange processes on Cr⁰ and Cr⁹⁰⁷ and Rh¹⁹⁰⁸ have been described.

Rates have been measured⁹⁰⁹ for the inversion at Re of the enantiomerically pure (SR)-[Re{NHCH(Me)Ph}(PPh₃)(NO)Cp] to the (RR) epimer. Replacement of PPh₃ in the (SR) isomer by PTol₃ gives 66:34 RR:SR products via rate-determining PPh₃ dissociation (with amide lone-pair anchimeric assistance) giving a trigonal-planar intermediate.

Redox reactions

Reviews have appeared by Poli,⁹¹⁰ Espenson,⁹¹¹ and, in a recent text, by Astruc.⁹¹² The rate of solution-phase electron transfer within the ion pair, [CoCp₂]⁺[V(CO)₆]⁻,

increases *ca.* two-fold for each additional vibrational quantum in the CO stretching mode.⁹¹³ The enhancement of the rate of substitution of CO in $[\text{Cr}(\text{CO})_3(\eta\text{-C}_6\text{H}_6)]$ by $\text{P}(\text{OEt})_3$ from the incorporation of a ferrocenium (Fc^+) group into the π -bonded arene is ascribed⁹¹⁴ to internal electron transfer from Cr^0 to Fe^{III} . Electron-transfer catalysed replacement of CO by PPh_3 in $[\text{Ru}_3(\text{CO})_{12}]$ has also been reported.⁹¹⁵ Further reports of 17-electron species derived from metal–metal bonded complexes include studies of $[\text{M}(\text{CO})_3(\eta^5\text{-C}_5\text{H}_4\text{R})]$ ($\text{M} = \text{Cr}$ or Mo),⁹¹⁶ $[\text{Ta}(\text{CO})_4(\text{dppe})]$,⁹¹⁷ $[\text{Fe}(\text{CO})_3(\eta^5\text{-C}_5\text{Ph}_5)]$ ^{918,919} and the isomeric diradicals from the Cr–Cr homolysis of $[\text{Cr}_2(\text{CO})_4\text{L}_2(\text{fv})]$.⁹²⁰ Dimerisation and halogen-atom abstraction processes of these species have been described. In addition, third-order kinetics have been observed⁹²¹ in the reaction of $[\text{Cr}(\text{CO})_3\text{Cp}^*]$ with thiols. A pre-equilibrium addition of RSH to the radical to give a 19-electron intermediate followed by attack of a second radical is proposed. Reaction of disulfides with the heterobimetallic radical, $[\text{Cp}_2\text{Ta}(\mu\text{-CH}_2)_2\text{CoCp}]$, to give the monothiolato complex, $[\text{Cp}_2\text{Ta}(\mu\text{-CH}_2)_2\text{Co}(\text{SR})\text{Cp}]$, can occur either *via* an outer-sphere electron transfer (seen also in the reaction of $[\text{CoCp}_2]$ and R_2S_2) or by an initial pre-equilibrium between the reactants.⁹²² Electron-withdrawing groups in R favour an outer-sphere mechanism. The stable radical $[\text{Fe}(\text{CO})_2\{\eta^5\text{-C}_5(\text{CH}_2\text{Ph})_5\}]^*$ has been characterised.⁹²³ The Rh–Rh bond in $[\text{Rh}^{\text{II}}_2(\text{NCMe})_{10}]^{4+}$ suffers homolytic cleavage on photolysis in MeCN to give $[\text{Rh}^{\text{II}}(\text{NCMe})_6]^{2+}$ which then disproportionates.⁹²⁴ Rhenium–rhenium bond fission in a 3,4-dirhenacyclobutene complex has also been studied.⁹²⁵ Initial studies have appeared⁹²⁶ of the reduction of the $\text{Au}^{\text{II}}\text{-}\text{Au}^{\text{II}}$ bonded complex $[\{\text{AuCl}(\text{dppn})\}_2]^{2+}$ to $[\text{Au}(\text{dppn})_2]^+$ by benzyl alcohol. The radical cation resulting from one-electron oxidation of $[\text{Mo}_2(\mu\text{-Cl})(\mu\text{-SMe})_3\text{Cp}_2]$ retains the quadruply-bridged structure, but the bridging chloride is labilised to substitution by MeCN and other ligands.⁹²⁷ An unusual isomerism, involving $\text{LRe}-\text{ReL} \rightleftharpoons \text{Re}(\mu\text{-L})_2\text{Re}$, has been observed in mixed-valence dirhenium complexes.⁹²⁸ Substitution of X in $[\text{FeX}(\text{CO})_2\text{Cp}]$ (X = Cl, Br or I) by phosphines may be catalysed⁹²⁹ by an electron-transfer chain process. A similar mechanism is proposed⁹³⁰ for the catalysis by $[\text{Fe}^{\text{I}}\text{Cp}(\eta\text{-C}_6\text{H}_6)]$ of formation of zwitterionic $[(\text{CO})_3\text{Mo}^-(\mu\text{-}\eta^5\text{:}\eta^5\text{-fv})\text{Mo}^+(\text{CO})_2(\text{PR}_3)_2]$ from $[\text{Mo}_2(\text{CO})_6(\text{fv})]$ and phosphines. Activation of alkyl C–H^{931,932} and ligand C–C coupling⁹³³ may be induced by metal-complex oxidation. A $\kappa^2\text{-}\kappa^3$ isomerisation may be induced by the oxidation of $[\text{Rh}^{\text{I}}(\text{CO})(\text{PPh}_3)(\kappa^2\text{-Tp}')]^+$ to $[\text{Rh}^{\text{II}}(\text{CO})(\text{PPh}_3)(\kappa^3\text{-Tp}')]^+$.⁹³⁴ Dimerisation of $[\text{Mn}(\text{CO})_3(\eta\text{-C}_6\text{H}_6)]^+$ to $[\{\text{Mn}(\text{CO})_3\}_2\{\mu\text{-}(\eta^4\text{-C}_6\text{H}_6\text{-}\eta^4\text{-C}_6\text{H}_6)\}]^{2-}$ (containing a bridging tetrahydronaphthalene ligand) on reduction proceeds by reaction with the parent cation of a $[\text{Mn}(\text{CO})_3(\eta^4\text{-C}_6\text{H}_6)]^-$ intermediate.⁹³⁵ Electrogenerated 17-electron radical cations, $[\text{M(ind)}_2]^+$ ($\text{M} = \text{Ru}$ or Os) react with nucleophiles (X = Cl[−] or MeCN) to give the 19-electron adducts, $[\text{MX(ind)}_2]^+$ which oxidise further to 18-electron $[\text{MX(ind)}_2]^{2+}$.^{936,937} Replacement of CO by solv (solv = MeCN or tbf) in $[\text{Fe}(\text{CO})(\eta^2\text{-dtc})\text{Cp}^*]^+$ {formed by oxidation of $[\text{Fe}(\text{CO})(\eta^2\text{-dtc})\text{Cp}^*]$ by Fc^+ } proceeds by an associative mechanism.⁹³⁸ Fifteen-electron intermediates are proposed⁹³⁹ to arise from the decarbonylation of $[\text{MoCl}(\text{CO})(\text{PMc}_3)_2\text{Cp}]^+$. Related processes involving low-valent Mn and Re carbonyl complexes^{98–101,108,109} and halogen transfer^{941,942} have been discussed earlier. The complexes $[\text{Re}(\text{CO})_3(\text{bipy})]^+$ and $[\text{Re}(\text{CO})_3(\text{bipy})]^-$ rather than $[\text{ReCl}(\text{CO})_3(\text{bipy})]^-$ are the active species in the electroreduction of CO_2 catalysed by $[\text{ReCl}(\text{CO})_3(\text{bipy})]$.⁹⁴⁰ The rate of halogen transfer between $[\text{W}(\text{CO})_3\text{Cp}]^-$ and $[\text{MoX}(\text{CO})_3\text{Cp}]$ shows⁹⁴¹ little dependence on X. The

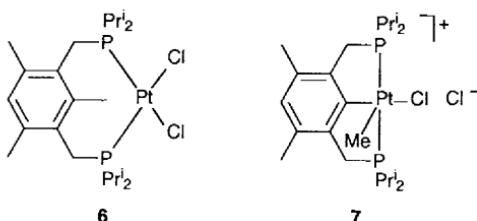
kinetics of the one-electron steps, Pd^{II} to Pd^{I} and Pd^{I} to Pd^{0} have been characterised for $[\text{Pd}(\text{mtas})_2]^{2+}$.⁹⁴²

Redox processes involving $[\text{Re}(\text{Me})\text{O}_3]$ have already been described.^{182–184,266} Mechanisms of oxidations of organic substrates catalysed by $[\text{Re}(\text{Me})\text{O}_3]$ have also been reported.^{943–950}

Oxidative addition and reductive elimination

Computational and experimental estimation of secondary α -deuterium isotope-effects suggest⁹⁵¹ that the oxidative addition of MeI to $cis\text{-}[\text{RhI}_2(\text{CO})_2]^-$ (see also ref. 862) occurs via an $S_{\text{N}}2$ mechanism with inversion of configuration at carbon. The logs of the second-order rate constants for the reaction of $[\text{Ir}(\text{CO})(\text{PR}_3)\text{Cp}^*]$ with MeI , to give $[\text{IrMe}(\text{CO})(\text{PR}_3)\text{Cp}^*]^+\text{I}^-$, correlate linearly⁹⁵² with measures of the basicity of the complexes. The calculated exothermicity of the addition of CH_4 to $\{\text{Rh}(\text{CO})\text{Cp}\}$ to give $[\text{RhH}(\text{Me})(\text{CO})\text{Cp}]$ is very sensitive to the representation of the 5s orbital.⁹⁵³ Tunnelling effects on the relative rates of reaction of the C–C and C–H bonds in C_2H_6 and a bare Pd atom have been computed.⁹⁵⁴ Initial studies⁹⁵⁵ of the early stages (ps) of the photoreaction of $[\text{Rh}(\text{CO})_2\text{Tp}']$ in hydrocarbon solution suggest that solvated monocarbonyl intermediates form prior to RH oxidative addition to give $[\text{Rh}(\text{H})\text{R}(\text{CO})\text{Tp}']$. Quantum efficiency of C–H activation is lower at 458 nm than at 366 nm.⁹⁵⁶ Theory supports⁹⁵⁷ an oxidative-addition process over a four-centre process for the σ -bond metathesis, $\text{Ir}(\text{CH}_3) + \text{RH} \rightleftharpoons \text{MR} + \text{CH}_4$. Related calculations⁹⁵⁸ for the activation of alkane C–H by Pt^{II} or Pd^{II} favour a four-centre process for the former (thereby avoiding the formation of Pt^{IV}). An oxidative addition-reductive elimination process is not favoured energetically for Pd, but cannot be excluded for Pt. From related calculations, Russian workers⁹⁵⁹ propose the existence of $[\text{PtCl}_2(\eta^2\text{-CH}_4)(\text{PH}_3)_2]$. Oxidative addition of SiH_4 to $[\text{MCl}(\text{CO})(\text{PH}_3)_2]$ ($\text{M} = \text{Rh}$ or Ir) has been studied theoretically.⁹⁶⁰ Slow elimination of $\text{Bu}'\text{Et}$ from $[\text{Ir}(\text{H})(\text{CH}_2\text{CH}_2\text{Bu}')\text{Cl}(\text{PPr}_3^i)_2]$, formed from $[\text{IrCl}(\text{H})_2(\text{PPr}_3^i)]$ and $\text{Bu}'\text{CH}=\text{CH}_2$, is thought to proceed via a $[\text{IrCl}(\text{CH}_3\text{CH}_2\text{Bu}')(\text{PPr}_3^i)_2]$ complex.⁹⁶¹ The cation $[\text{Zr}(\text{CH}_2\text{CMe}_3)\text{L}_2]^+$ gives butene by reversible β -Me elimination⁹⁶² for $\text{L} = \text{Cp}$ but irreversibly for the more sterically demanding $\text{L} = \text{Cp}'$.

Protonolysis of $[\text{Pt}(\text{R})\text{Cl}(\text{Me}_4\text{en})]$ by HCl involves⁹⁶³ initial formation of $[\text{Pt}^{\text{IV}}(\text{H})(\text{R})\text{Cl}_2(\text{Me}_4\text{en})]\text{Cl}^-$ dissociation to give $[\text{Pt}^{\text{IV}}(\text{H})(\text{R})\text{Cl}(\text{Me}_4\text{en})]^+$, C–H bond formation giving the alkane σ complex $[\text{Pt}^{\text{II}}\text{Cl}(\text{RH})(\text{Me}_4\text{en})]^+$, followed by RH loss. The importance of five-co-ordinate intermediate formation is underlined by Hill and Puddephatt⁹⁶⁴ who have synthesised $[\text{PtH}(\text{Me})_3(4,4'\text{-Bubipy})]$ and shown it to be stable to reductive elimination of MeH . The intermediate, $[\text{Pt}(\text{CH}_2\text{CH}_3)\text{Cl}(\eta^2\text{-C}_2\text{H}_4)]$, isolated from the reaction of $[\text{PtCl}_4]^{2-}$ and ethene in water,⁹⁶⁵ gives $[\text{Pt}(\text{CH}_2\text{CH}_2\text{OH})\text{Cl}_5]^{2-}$, *trans*- $[\text{Pt}(\text{CH}_2\text{CH}_3)(\text{CH}_2\text{CH}_2\text{OH})\text{Cl}_4]^{2-}$ and ethanol on reaction with $[\text{PtCl}_6]^{2-}$. Platinaoxetane formation from ethylene oxide and $[\text{PtCl}_4]^{2-}$ has also been studied.⁹⁶⁶ Methane formation from $[\text{Ru}_2(\mu\text{-CH}_2)(\mu\text{-CO})(\text{CO}_2\text{Cp}_2)]$ in the presence of SiMe_3H proceeds via initial CO dissociation, oxidative addition of SiMe_3H , to give $[\text{Ru}_2(\text{H})(\text{SiMe}_3)(\mu\text{-CH}_2)\text{CO}_2\text{Cp}_2]$, equilibration with $[\text{Ru}_2(\text{CH}_3)(\text{SiMe}_3)(\text{CO}_2\text{Cp}_2)]$, followed by oxidative addition of a second mol of SiMe_3H and then elimination of CH_4 .⁹⁶⁷ Silylene–metal and –alkylidene intermediates are implicated in related reactions.^{829,968–970} The complex $[\text{Mo}(\text{PMMe}_3)_6]$ reacts with phenols to give $[\text{MoH}(\text{OR})(\text{PMMe}_3)_4]$ as the thermodynamically



cally favoured product, rather than an orthometallated species which deuterium-labelling and magnetisation-transfer experiments suggest is kinetically accessible.⁹⁷¹ Oxidative addition of $4\text{-RC}_6\text{H}_4\text{I}$ to $[\text{Pd}^0(\text{R})(\text{PPh}_3)_2]^-$ leads to $[\text{Pd}^{\text{II}}\text{R}(\text{R}')(\text{I})(\text{PPh}_3)_2]^-$ with $t_{1/2}$ ca. 1–5 ms.⁹⁷² Reductive elimination of RR' then follows. The ΔV^\ddagger and other kinetic parameters⁹⁷³ suggest that oxidative addition of PhI to Pd is not the rate-determining step in its coupling with 2,3-dihydrofuran. Aryl coupling with Ni⁰ complexes has also been studied.⁹⁷⁴ Cyclometallation processes have also been investigated,^{975–978} with complex 6⁹⁷⁶ suffering C–C fission and MeCl loss on reaction with HCl, via a proposed Pt^{IV} intermediate 7.

Reductive C–C elimination from $[\text{Os}^{\text{IV}}(\text{C}_6\text{H}_3\text{Me}_2\text{-2,4})_4]$ induced by phosphines and phosphites is an associative process in which P-donor co-ordination and intramolecular C–C elimination are synchronous.⁹⁷⁹ Reductive elimination of aryl ethers from $[\text{Pd}(\text{OC}_6\text{H}_4\text{X-4})(\text{OBu}')(\text{dppe})]$ ⁹⁸⁰ has been described. The varied [CO] dependence of the C–H reductive-elimination process, of $[\text{Ru}_3(\mu_3\text{-CX})(\mu\text{-H})_3(\text{CO})_9]$ to $[\text{Ru}_3(\mu_3\text{-}\eta^2\text{-CHX})(\mu\text{-H})_2(\text{CO})_9]$ (X = CO₂Me) or to $[\text{Ru}_3(\mu_3\text{-}\eta^2\text{-CH}_2\text{X})(\mu\text{-H})(\text{CO})_9]$ (X = SET)⁹⁸¹ are ascribed to anchimeric assistance by X. The unusual oxidative elimination of H₂ from $[\text{U}^{\text{III}}_2(\mu\text{-OH})_2(\eta^5\text{-C}_5\text{H}_3\text{R}_2\text{-1,3})_4]$ to give $[\text{U}^{\text{IV}}_2(\mu\text{-O})_2(\eta^5\text{-C}_5\text{H}_3\text{R}_2\text{-1,3})_4]$, (R = SiMe₃) obeys first-order kinetics with $k_{\text{H}}/k_{\text{D}} = 4.1$ and is believed to be an intramolecular process.⁹⁸²

Hydrogen and hydrido complexes

The rate constant for oxidative addition of H₂ to *trans*-[IrCl(CO)L₂] is 45-fold larger for L = PPh₂(C₆H₄SO₃K-*m*) in water than for L = PPh₃ in toluene.⁹⁸³ The activation barrier for the reaction of Fe(CO)₄ and H₂ in the gas phase to give [FeH₂(CO)₄] is <4 kcal mol⁻¹.⁹⁸⁴ Reaction of $[\text{Ru}_3(\mu\text{-H})_2(\mu\text{-PBu}'_2)_2(\text{CO})_8]$ with H₂ involves rapid initial CO loss followed by oxidative addition of H₂ to a single Ru atom.⁹⁸⁵ Parahydrogen-induced polarisation has been used to show that H₂ addition to $[\text{Pt}\{\text{Ph}_2\text{PCH}_2\text{CH}(\text{Me})\text{OPPh}_2\}]$ is a concerted process,⁹⁸⁶ to characterise intermediates and minor isomers in Ir–H and Ru–H complexes,^{987,988} to establish reversibility in a Rh-catalysed hydrogenation process⁹⁸⁹ and to characterise related proton-exchange processes.⁹⁹⁰ Spin-saturation transfer also reveals⁹⁹¹ that exchange occurs between Ru-bound- and Si-bound-H in $[\text{Ru}(\text{H})(\text{SiHPh}_2)(\text{CO})\text{L}_2]$, a process which occurs intramolecularly in the absence of SiH₂Ph₂. The ΔG^\ddagger value for H₂ loss increases with R_HH for a series of isoelectronic analogous Os complexes, $[\text{OsH}(\text{X})(\eta^2\text{-H}_2)(\text{CO})(\text{PPr}'_3)_2]$.⁹⁹² In an independent study on $[\text{Osh}(\text{Cl})(\eta^2\text{-H}_2)(\text{CO})(\text{PPr}'_3)_2]$,⁹⁹³ hydrogen exchange between H and H was found to be very slow, with $k_{\text{H}}/k_{\text{D}} = 4.6$.

The cation $[\text{Os}(\text{CO})(\eta^2\text{-H}_2)(\text{PPh}_3)_2(\text{bipy})]^{2+}$ is inert to exchange with D₂ over several weeks at room temperature.⁹⁹⁴ The cation *trans*-[Os($\eta^2\text{-H}_2$)(NCMe)(dppe)₂]²⁺, with a pK_a of –2, is claimed to be the most acidic fully characterised⁹⁹⁵

stable H₂ complex. No evidence has been found that [MH₂(pp₃)]⁺ (M = Co, Rh or Ir) contains ligated η²-H₂, though a kinetic isotope effect, k_H/k_D, of 1.3 for M–H site exchange, suggests that an η²-H₂ bonded species might be an intermediate.⁹⁹⁶ Theoretical studies suggest that the difference in energy between [Os(H)Cl(CO)(η²-SiH₄)] and [OsCl(CO)(η²-H₂)(SiH₃)]⁺ is very small.⁹⁹⁷ Equilibria involving [Ir(H)₂X(S)(PPr₃)₂], H₂ and [Ir(H)₂X(η²-H₂)(PPr₃)₂] in hydrocarbons have been characterised.⁹⁹⁸ The classical polyhydride, [ReH₅(cytpp)], reacts with H⁺ to give [ReH₄(η²-H₂)(cytpp)]⁺ which is inert to CO and P(OMe)₃ at room temperature.⁹⁹⁹ Hydrogen-deuterium exchange catalysed by Ru^{II}-porphyrin¹⁰⁰⁰ and Pt–Au clusters¹⁰⁰¹ has also been studied. Related spectroscopic,^{1002–1008} structural,^{1009–1013} theoretical^{1014–1018} and reactivity^{1019–1024} studies have also been reported.

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