Coordination Chemistry Reviews, 62 (1985) 1—35
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

1. ZINC AND CADMIUM

D. DAKTERNIEKS

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Introduction

ZINC

CADMIUM

0010-8545/85/$12.25

INTRODUCTION

This review of the inorganic and coordination chemistry of zinc and cadmium covers the material which appeared in volumes 98 and 99 of Chemical Abstracts. The format established by Constable [1] has been largely retained. Classification of donor ligands is often difficult for compounds containing several different donor atoms and the reader may need to refer to more than one section.

1.1 ZINC(II)

1.1.1 Halide and pseudohalide complexes

Electron diffraction, X-ray scattering and Raman spectra of aqueous solutions of $ZnBr_2$ show the existence of octahedral hexaaquo and tetrahedral $Zn(H_2O)_{4-n}Br_n$ (n = 2,3,4) [2]. The inner sphere complexes have Zn^{2+}-$Br^- = 2.4$Å, Zn^{2+}-$H_2O = 2.2$Å. Stability constants for formation of Zn(II) bromide, iodide and thiocyanate complexes in methanol are reported [3]. Far IR studies of solid pyridinetrihalozincate anions show C_{3v} symmetry [4]. Organobimetallic compounds $(SCN)_2Zn(NCSHgR)_2$ and their reactions with Lewis bases have been reported [5,6]. The structures of various solid zinc fluoride compounds have been described [7,8]. Participation of *3d* electrons in zinc halide bonding has been investigated [9]. The reaction of metallic zinc with nonaqueous solutions of ammonium thiocyanate [10] and extraction of Zn^{2+} from hydrochloric acid solutions [11] have been reported.

1.1.2 Complexes with oxygen donor ligands

1.1.2.1 Inorganic molecules and ions

X-ray diffraction studies of aqueous $Zn(NO_3)_2$ show octahedral coordination of Zn^{2+} (Zn^{2+}-$OH_2 = 2.17$Å) and no evidence for coordination by NO_3^- [12]. X-ray diffraction studies of aqueous $ZnSO_4$ show no concentration effect in the range 0.6-3.1M [13] and again show octahedral $[Zn(OH_2)_6]^{2+}$ [14]. An *ab initio* MO study of $Zn(OH_2)_6^{2+}$ yields Zn-O distances which agree with experimental findings [15]. The use of a balanced-like-charges approach gives calculated formation constants for $[ZnHCO_3]^+$ which reproduce experimental values [16]. Chlorosulphate acts as a unidentate ligand in compounds $ZnL_4(SO_3Cl)_2$ (L = py,CH_3CN) [17].

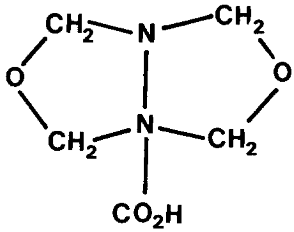
1.1.2.2 Simple organic ligands

High resolution electron energy loss spectroscopy (HREELS) was used to study the interaction of CO with the $(10\bar{1}0)$ surface of ZnO [18]. The increase of the CO stretching frequency upon adsorption of CO to ZnO is mostly due to a strengthening of the CO bond and not solely to a mechanical coupling between oscillations. Reaction of $ZnCl_2$ with $Al(O^iPr)_3$ and potassium in isopropanol gives $Zn[Al(O^iPr)_4)]_2$ which contains two $Al(O^iPr)_4$ moieties coordinated to zinc by bridging O^iPr ligands [19]. A crystal structural analysis of ZnL_2Cl_2 (L = 1,4-dioxane) shows Zn in a distorted trigonal bipyramidal environment with bonding to two chlorine atoms and an oxygen atom of a monodentate L in the equatorial plane and two bidentate bridging L molecules forming an infinite chain [20]. Various reactions of triethanolamine with zinc salts have been described [21,22]. X-ray structures of $[M(OS_3N_2)_6][AsF_6]_2$ (M = Zn,Cd) show octahedral O_6 environments about the central M atom [23]. Octahedral O_6 coordination is also implied in complexes of Zn^{2+} with embelin [24]. The compound $Zn(SO_2)_2(AsF_6)_2$ has been isolated from liquid SO_2 [25] and acts as a Lewis acid with a variety of phosphine oxides, sulphides and selenides [26-28].

1.1.2.3 Carboxylic acids and related ligands

An X-ray structural and neutron-diffraction study of zinc maleate tetrahydrate $Zn(C_4O_4H_3)_2(H_2O)_4$ shows octahedral coordination by two axial monodentate maleates and four equatorial water molecules [29]. The zinc atom in zinc citrate $Zn(C_6H_5O_7)_2$ is octahedrally coordinated by two equivalent citrates via a central hydroxyl, central carboxyl and one terminal carboxyl oxygen atom from each citrate [30]. Electron impact mass spectra have been recorded for zinc acetate [31]. Glycine coordinates through the carboxylate group in a number of its complexes with $ZnCl_2$ [32]. The crystal structure of bis(N-benzoylglycinato)triaquazinc(II)dihydrate shows coordination by an oxygen atom of the carboxyl group of each anion and by three water molecules giving a slightly distorted trigonal bipyramidal environment about zinc [33]. The compound ZnL_2 where LH is (1) has been prepared and characterised [34].

The crystal structure of $[Zn(edta)]^{2+}$ shows the metal to possess an octahedral O_4N_2 environment [35] as does the metal ion in ZnL_2 (LH = *o*-aminobenzoic acid) [36]. The interactions of a variety of substituted dicarboxylic acids with Zn^{2+} have been described [37-43]. Coordination occurs through the oxygen atom in a variety of monohydroxamine complexes of zinc [44]. Thermodynamic data for zinc complexes derived from hydroxamic acids [45], substituted carboxylic acids [46], salicylic acid [47], acetic and levulinic acids [48], substituted anthranilic acid [49] and substituted picolinic acid

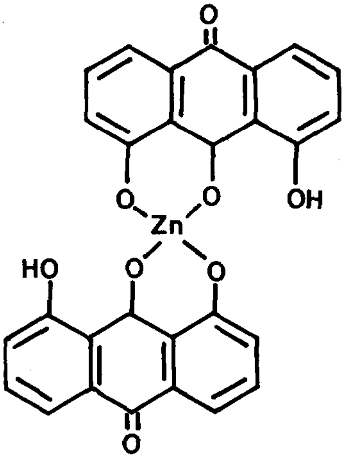


(1)

[50] have been reported. Thermodynamic parameters for the interaction between Zn^{2+} and L-3,4-dihydroxyphenylalanine (L-dopa) [51] and 2-amino-2(hydroxymethyl)-1,3-propandiol [52] are also reported. A structure-reactivity relationship which predicts formation constants based largely on the structure of the organic ligand has been presented [53].

1.1.2.4 Diketone and other ligands

The electrosynthesis of $Zn(acac)_2$ in methanol has been reported [54]. Addition of cyanogen to dipivaloylmethane to give 2,2,6,6-tetramethyl-4-(aminocyanomethylene)-3,5-heptanedione is catalysed by $Zn(acac)_2$ [55]. Solvent extraction studies of zinc with 2-thenoyltrifluoroacetone continue and it has been found that addition of bidentate amines at low concentration ($<10^{-5}$M) causes a remarkable improvement in the distribution ratio while higher concentrations of amine cause a lowering of the distribution ratio [56]. Formation constants in various mixed aqueous solvents of the zinc(II) complex with dibenzoylmethane have been measured [57]. Adducts of $Zn(acac)_2$ with a variety of nitrogenous bases have been prepared and characterised [58]. Various mixed-ligand complexes of zinc involving diketones have been investigated [59-61]. The ligand 1,8-dihydroxyanthraquinone (HL) gives only a mononuclear compound ZnL_2 **(2)** [62]. The compounds ZnL_2 (LH = 2-acetyl- and 2-benzoyl-1,3-indandione) are tetrahedral with the ligands coordinated through one acyl oxygen and one oxygen of the indandione moiety [63]. Coordination complexes $MLCl_2$ (M = Zn,Hg and LH = 3-amino-2-benzoylbenzofuran) have tetrahedral O_2Cl_2 environments [64]. Zinc complexes of pyridoxine have been reported to contain the ligand as bidentate oxygen donors [65]. Stability constants for zinc(II) complexes with 8-formyl-7-hydroxy-4-methyl-2H-1-benzopyran-2-one [66] and 4-hydroxy-3-aldehydrobiphenyl [67] have been determined. A homobinuclear zinc complex Zn_2LCl_4 (L = N,N'-bis(antipyrylmethyl)piperazine) contains the ligand in a bridging oxygen role [68]. The mixed-ligand compound of zinc with



(2)

o-hydroxypropiophenone and quinoline has been reported [69]

The crystal structures of ZnX_2L_2 (X = Cl,Br and L = triphenylphosphineoxide) show the zinc to be in a O_2X_2 environment [70]. Organophosphoric acids as complexons for a variety of metal ions, including zinc and cadmium, have been studied [71]. Thermodynamic parameters for formation of simple zinc(II) complexes with adenosine-5-triphosphate (H_4ATP) show that the equilibrium

$$Zn^{2+} + ATP^{4-} \rightleftharpoons [Zn(ATP)]^{2-}$$

is enthalpically disfavoured but entropically stabilised [72].

1.1.3 Complexes with sulphur donor ligands

1.1.3.1 Simple organic ligands

Thiophenol compounds continue to be of interest and the preparations of $[M(SPh)_4]^{2-}$ (M = Zn,Cd) have been described [73]. Zinc(II) complexes with some 1,3-dithio ligands have been prepared and characterised by ESCA [74]. Only two studies involving phosphine sulphides as ligands have appeared [75,76].

1.1.3.2 Thioacids, thioamides and related compounds

The synthesis and characterisation of some alkyl substituted monothio-β-diketonate complexes have been described [77,78]. Complexes of monothiocarbamates and their reactions with pyridine have been reported [79].

The zinc(II) complexes of 4-aminophenazone dithiocarbamate [80] and *p*-ethoxyphenyl dithiocarbamate [81] have been described. A crystal and molecular structure study of zinc(II) bis(diallyldithiocarbamate) shows a dimer in which each zinc is coordinated by five sulphur atoms at distances ranging between 2.339Å and 2.846Å [82]. Zinc(II) and cadmium(II) complexes of piperazinebis(dithiocarbamate) contain tetradentate bridging ligands and appear to be polymeric [83]. Bimetallic complexes $MZnL_4$ (M = Co,Cu and LH = HS_2CNEt_2) and their reactions with pyridine have been described [84].

Complexes MX_2L (M = Zn,Cd; X = Cl,Br,NCS,OAc,NO_3; L = N,N,N'N'-tetramethylthiuram disulphide) have been reported [85]. The extraction voltametry of some zinc(II) and cadmium(II) complexes with alkylxanthogenates and nitrogen-containing ligands has been investigated [86].

The crystal structures of the anions $[Zn(S_2P\{OC_6H_4Me\}_2)_3]^-$ and $[Zn(S_2PPh_2)_3]^-$ are almost identical [87]. Both anions are distorted tetrahedral about zinc with one bidentate ligand having somewhat larger Zn-S distances than those of each of the two unidentate ligands. The polarographic behaviour of zinc(II) with diethyldithiophosphoric acid and nitrogen-bases such as pyridine was studied and in all cases the electroreduction Zn(II) $\longrightarrow$ Zn(0) was observed [88]. Electron impact mass spectra were recorded for some zinc(II) complexes of dialkyldithiophosphoric acid [89]. Thermodynamic data for zinc(II) complexes with thiodiacetic and dithioacetic acids have been reported [90]. The zinc(II) and cadmium(II) complexes of 2-aminocyclopentene-1-dithiocarboxylic acid contain the ligands as a bidentate through the CS_2 group [91].

1.1.3.3 Sulphur heterocycles

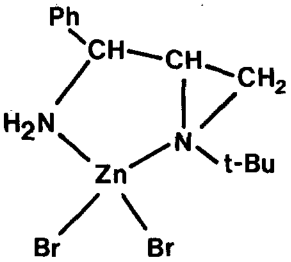
Imidazoline-2-thione and its methyl analog give complexes ZnL_2X_2 and $ZnLX_2$ (X = halide) in which coordination is through the sulphur atom of the heterocycle [92]. The ligands N-benzothiazoyl-N'-alkylthioureas act as bidentate donors towards zinc(II) and cadmium(II) with coordination occuring through nitrogen from thiazole and sulphur from thiourea [93]. The thermal stability of these complexes is Zn<Cd. Complexes ZnL_2X_2 (L = benzothiazolesulphonamidmorpholide; X = NO_3,OAc,Cl) with L being bidentate through sulphur of the thiazole ring and nitrogen of the sulphonamide have been described [94]. Formation constants for $[ZnL]^+$ and ZnL_2 (HL = N-(2,3-dimethyl-1-phenyl-5-oxo-3-pyrazoline-4-yl)-N'-benzoylthiourea) were measured in 70% EtOH-H_2O [95] and the metal complexes are thought to be octahedral with coordination through benzoyl carbonyl oxygen and thiocarbonyl sulphur. The stability constant for interaction of Zn^{2+} with 2-hydroxy-1-naphthalidenesulphamethoxazole has been measured [96]. Complexes

ZnL_2 and CdL_2 (HL = N-α-dibenzoylthioacetamide) contain bidentate ligands coordinated through thio and carbonyl groups [97].

1.1.4 *Complexes with nitrogen donor ligands*

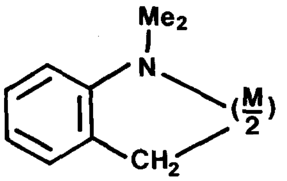
1.1.4.1 *Amines*

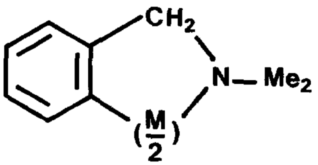
The complex $Zn(OAc)_2.2NH_3$ has been obtained by passing ammonia through a dmf solution of $Zn(OAc)_2.2H_2O$ [98]. Stability constants for the interaction of Zn^{2+} with N-(2-hydroxy-5-phenylbenzylidene)aniline have been measured [99]. A crystal structure determination of $ZnLBr_2$ (L = α-(1-tert-butyl-2-aziridinyl)benzylamine shows tetrahedral N_2Br_2 coordination (3) about zinc [100].



(3)

The crystal structure of ZnL_2 (L = N-(2-pyrrolylmethylene)-tert-butylamine) contains a tetrahedral zinc atom coordinated to four nitrogen atoms of two symmetry related bidentate ligands [101]. Some chelate-supported alkyl and aryl complexes (4,5) have been described [102].





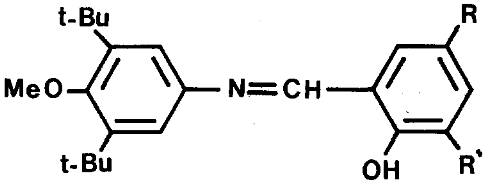
M = Zn, Cd

(4) (5)

The zinc atom in $[ZnL_2(lactate)][lactate]$ (L = N-isopropyl-2-methyl-1,2-propanediamine) has a distorted trigonal bipyramidal N_4O environment [103]. The synthesis and characterisation of a series of tris-chelate compounds $[Zn(LL)_3](T)_2$ (LL = (R.S)-1-amino-2-propanol; T = triflate) has been described [104]. Proton NMR has been used to study dmf solvent exchange on the five coordinate compound $[Zn(Me_6\text{-tren})(dmf)]^{2+}$ (Me_6-tren = 2,2'.2''-tris(N.N-dimethylamino)triethylamine) [105]. Dissociation of arylamine (*p*-anisidine. *p*-toluidene, aniline, *p*-chloroaniline) complexes with $ZnBr_2$ and ZnI_2 in acetonitrile was studied by following $-NH_2$ proton shifts [106] which are linear functions of the fraction of coordinated amine. Formation constants for the interaction of Zn^{2+} with 2-hydroxy-1-naphthalidene-*o*-fluoroaniline have been measured [107]. Formation constants of quaternary Zn(II) complexes comprising pyridoximine as a first. glycine as a second and ethylenediamine as a third ligand were determined potentiometrically [108]. The ligand in $(ZnClL)_2O$ (L = N.N'-bis(*o*-hydroxybenzyl)ethylenediamine is coordinated through one nitrogen atom and one hydroxy oxygen atom resulting in formation of two six-membered chelate rings [109]. The adduct $ZnCl_2(LH_2)$ (LH_2 = N.N'-(dipicolyl)-1.8-naphthylenediamine) is tetrahedral whereas ZnL is square planar with a N_4 environment about the zinc atom [110].

1.1.4.2 Schiff bases, hydrazones and oximes

The Schiff base derived from salicylaldehyde and 2-hydrazinobenzoxazole reacts with Zn^{2+} as a monobasic tridentate ligand with phenolic oxygen and both azomethinyl nitrogen atoms as donors [111]. The ligand formed from condensation of 5.5'-methylenebis(salicylaldehyde) and diaminopropane gives a polymeric Schiff base complex with Zn^{2+} [112]. Schiff bases derived from substituted salicylaldehydes and *o*-hydroxybenzylamine give complexes ZnL where the ligands. L. behave as tridentate *ONO* donors [113]. Schiff base complexes of salicylaldehyde and *o*-mercaptobenzaldehyde with bulky amines give pseudo-tetrahedral zinc compounds [114]. The ligands in ZnL_2 (HL = **(6)**) act as *ON* donors giving tetrahedral coordination about the zinc atom [115]. Mixed-ligand complexes of N-6-methylbenzothiazol-2-yl-salicylaldimine and 2-methylbenzimidazole with Zn^{2+} are octahedral with coordination of the ligands through phenolic oxygen and exocyclic nitrogen atoms [116]. The Schiff base **(7)** derived from salicoylhydrazine and biacetylmonoxime gives octahedral compounds with Zn^{2+} and Cd^{2+} [117]. Pyridoxal-5'-phosphate-α-amino acid zinc(II) complexes and their salicylaldehyde analogues show photochromic behaviour in borate buffer at pH 9.5 [118]. Zinc(II) complexes of non-ionised and of zwitterionic forms of Schiff bases have been described [119.120].

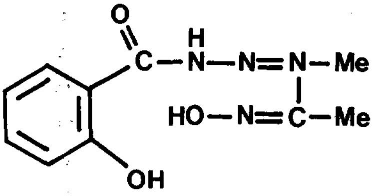


$R = H$ $R' = H, NO_2$

$R = Cl$ $R' = H$

$R = Br$ $R' = NO_2$

(6)



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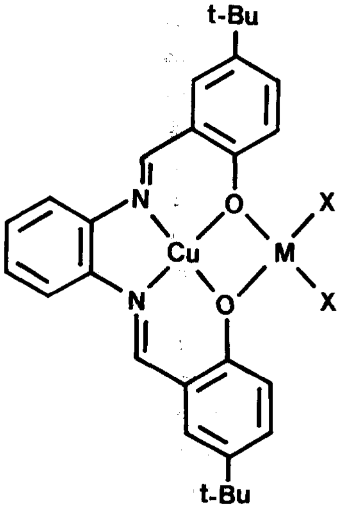


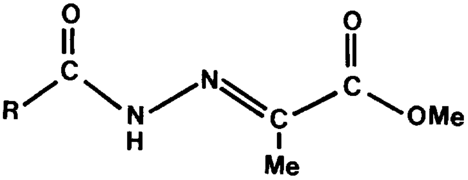
M = Zn, Cd

X = Cl, Br, I

(8)

Stability constants for the interaction of Zn^{2+} with Schiff bases of 3-formyl-4-hydroxybiphenyl have been determined [121]. Binuclear metal complexes $CuLMX_2$ (8) (M = Zn,Cd; X = Cl,Br,I) have been described [122]. The condensation product of ethanolamine with benzil biacetyl gives tetradentate ligand complexes with Zn^{2+} and Cd^{2+} [123]. In $ZnLCl_2$ (L = 2-pyridylaldehyde-2-pyridylcarboxylic acid hydrazide) the chlorines and the 2-pyridyl and imino nitrogens are coordinated to zinc [124]. A number of other ZnL_2 Schiff base complexes are also tetrahedral [125-129]. Schiff base complexes $ZnLCl_2$, $CdLCl_2$ (L = benzidine acetylacetone) are dimeric with L acting as a bidentate bridging ligand with coordination through the nitrogen atoms [130].

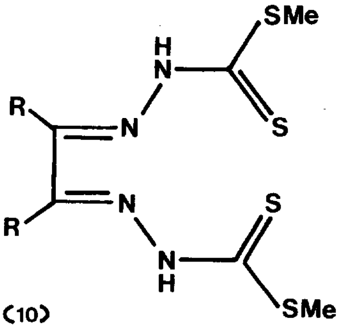
Methyl pyruvate aroylhydrazones (9) undergo deprotonation with $Zn(OAc)_2$ to form neutral hexacoordinated 1:2 complexes ZnL_2 with the ligands acting as tridentates through carbonyl oxygen atoms and the azomethine nitrogen [131].



R = Ph, o-C_4H_4OH

(9)

The X-ray structure of bis(2,6-diacetylpyridinebis(2-thenoylhydrazonato)(2-))dizinc(II) contains a pyridine ring unusually bridging two zinc atoms (Zn-Zn = 3.55Å) which have highly distorted octahedral environments [132]. The complexes ZnL_2, CdL_2 (LH_2 = (10)) are probably penta-coordinated dimers (11) [133].



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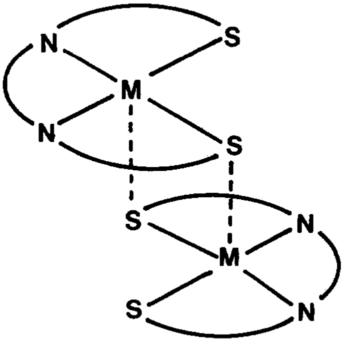
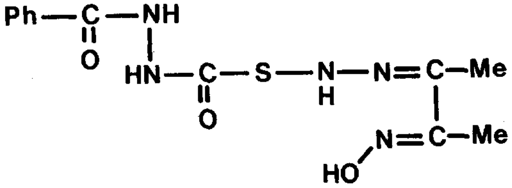


(11)

The preparation and characterisation of a variety of substituted hydrazone complexes with Zn^{2+} have been described [134-138] as have a number of oxime derivatives [139-141].

Zinc complexes of benzalacetone and dibenzalacetone-semicarbazone have been described [142]. Complexes $ZnLX_2$ and ZnL_2X_2 (L = benzil semicarbazone; X = Cl.NCS.ClO_4) are apparently also tetrahedral [143]. The zinc complex ZnL_2 (LH = 2-hydroxy-1.4-naphthaquinone thiosemicarbazone) contains the ligands coordinated through oxygen and imino nitrogen atoms [144]. Zinc and cadmium complexes containing tridentate benzoin semicarbazone and thiosemicarbazone have been described [145]. Complexes $ZnLX_2$ (LH_2 = 4-benzamido-1-diacetylmonoxime-3-thiosemicarbazone **(12)**; X = Cl.Br) contain tridentate N_2S coordinated ligand and coordinated halides [146].

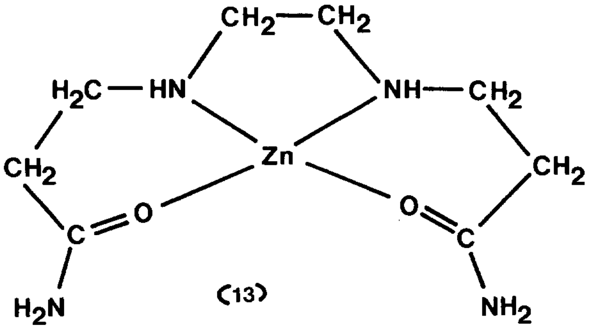
(12)



Stability constants for zinc complexes with some substituted diphenylcarbazones have been reported [147].

1.1.4.3 *Amides, hydrazides and related compounds*

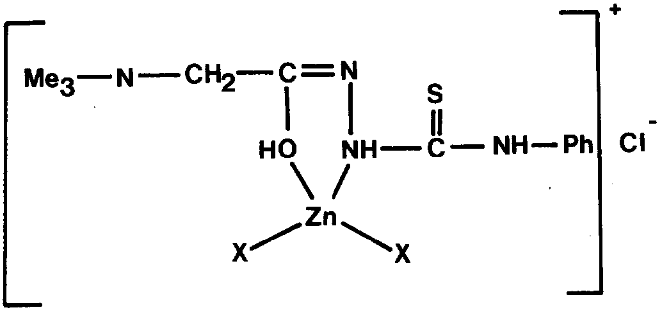
The preparation and thermochemistry of N.N-dimethylformamide adducts $ZnX_2(dmf)_{1,2}$ (X = Cl.Br.I) has been described [148]. The thermal stability of the zinc adducts is less than that of the corresponding cadmium adducts. Some zinc lactam complexes have been reported [149]. Zinc mixed-ligand complexes with some substituted acetamides and amines have distorted octahedral configurations [150]. The stability constant for formation of **(13)** has been measured potentiometrically [151]. Zinc formylhydrazide complexes ZnL_n (n = 1.2.3) contain bidentate ligands coordinated through carbonyl oxygen and terminal nitrogen [152]. The compound $Zn(HL)Cl_2$ (HL = 2-furanthiocarboxyhydrazide) is octahedral whilst $ZnL_2(H_2O)_2$ appears to be square planar [153]. The compound $Zn(tmc)Cl_2$ (tmc = 1.1.5.5-tetramethylcarbohydrazide) is monomeric and tetrahedral whilst $Zn(tmc)_2Cl_2$ is octahedral in the solid state but dissociates in solution [154]. Zinc and cadmium complexes of 3-nitrobenzoic acid hydrazide (L). $ML_4(NO_3)_2$ have been reported [155]. Salicylidene aroyl hydrazide ligands show dibasic tridentate behaviour towards zinc and cadmium [156]. The ligand *p*-hydroxyphenylthiohydrazide behaves as a sulphur-nitrogen donor towards zinc and cadmium [157].



(13)

Complexes [Zn(ATPTS)X_2]Cl (X = Cl,Br, $\frac{1}{2}SO_4$,OAc,NO_3) are thought to have structure (14) [158]. Coordination polymers of zinc and cadmium with 1-*iso*nicotinoyl-4-allyl-3-thiosemicarbazide have been described [159] and contain a tetradentate ligand. Zinc and cadmium complexes with 1-*iso*valeryl-4-phenyl-3-thiosemicarbazide are monomeric with ligand donation through oxygen and nitrogen atoms [160]. The ligand 1-acetylpyridium chloride-4-phenyl-3-thiosemicarbazide acts as a nitrogen donor towards zinc and cadmium [161]. Polymeric octahedral ZnL.$2H_2O$ (LH_2 = 5,5'-(*p*-phenylenebisazo)diquinolin-8-ol) has been described [162]. The crystal structure of zinc(II) dithizonate shows a N_2S_2 environment about zinc [163]. A variety of tetrahedral zinc and cadmium complexes of vanillin azine complexes have been reported [164]. Stability constants for zinc and cadmium complexes with phenylhydrazoacetoacetanilide ligands have been determined [165].

(14)

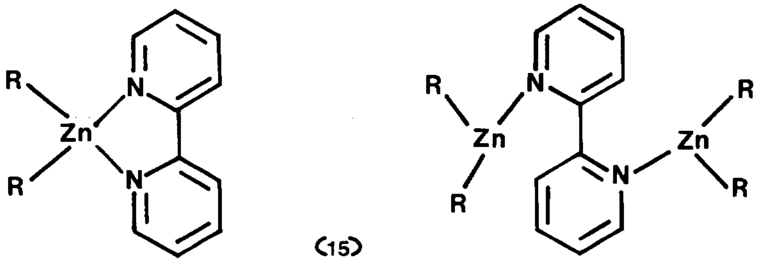


1.1.4.4 Amino acids

A polarographic study of L-4-hydroxyproline (LHP) complexes of zinc shows the existence of $[Zn(LHP)]^+$, $Zn(LHP)_2$ and $[Zn(LHP)_3]^-$ [166]. In $Zn(LHP)_2$ the ligand coordinates through the nitrogen atom of the pyrroline ring and an oxygen atom of the carboxylate group [167]. Stability constants for 1:2 complexes of zinc and cadmium with L-lysine have been determined [168]. Mixed-ligand complexes of zinc with anions of orotic acid and amino acids have been studied [169].

1.1.4.5 Heterocycles

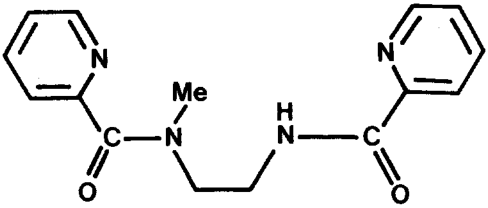
Solvent extraction measurements have been used to determine formation constants for mixed-ligand complexes of zinc with chloride and some substituted pyridines [170]. Aminopyridine adducts with zinc and cadmium formates are octahedral [171]. The 1:2 adduct between zinc ascorbate and pyridine has been studied [172]. Some substituted pyridine adducts of zinc halides have been described [173]. Crystal structures for $Zn(NO_3)_2(py)_2$, $Zn(NO_3)_2(2\text{-}Mepy)_2$ and $Zn(NO_3)_2(quin)_2$ show *cis*-coordination of the heterocyclic bases with various degrees of involvement of the nitrate ligands [174]. Complexes of $Zn(NCS)_2$ with some benzoylpyridines have been described [175]. The crystal structure of bis(1,4-dihydro-1-pyridyl)bispyridinezinc(II) shows a distorted tetrahedral N_4 environment about zinc [176]. Two bipyridine adducts of bis(3-nortricyclyl)zinc have been described (15) [177].



(15)

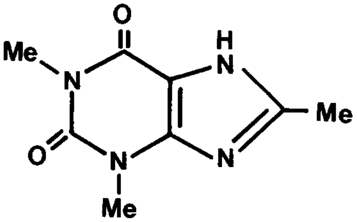
In $[Zn_2L(bipy)_2](PF_6)_2$ (LH_2 = naphthazarin) the ligand acts as a bridge between two metal centres, each zinc is in a N_2O_2 tetrahedral environment [178]. Synthetic evidence for a monodentate intermediate in the formation of chelated bipyridine complexes of zinc has been presented [179].

Stability constants for ternary mixed-ligand complexes of zinc with bipy, phen and N-(thiobenzoyl)-N-phenylhydroxylamine have been determined [180]. Stability constants for zinc complexes with Tiron and phen have also been determined [181]. Various adducts of $Zn(CN)_2$ with bipy and phen have been synthesised and characterised by IR [182]. The crystal structure of $Zn(terpy)Cl_2$ shows discrete monomeric molecules in which the zinc atoms have an almost trigonal bipyramidal N_3Cl_2 environment [183]. The ligand N-methyl-N.N'-bis(2'-pyridinecarboxamide)-1.2-ethane (16) forms non-deprotonated 1:1 complexes with zinc in which the ligand acts as a bis-bidentate forming five or six-coordinate polymeric complexes involving coordination through pyridyl nitrogen and amide oxygen atoms [184].


(16)

Some heterobinuclear complexes involving zinc and the tetradentate ligand N.N'-bis(antipyrylmethyl)piperazine have been described [185]. The compound $ZnL(NCS)_2$ (L = bis(methylaminoantipyryl)ethane) has also been characterised [186]. Substituted pyrimidine adducts with $ZnCl_2$ have been reported [187].

Imidazole and substituted imidazoles continue to be popular ligands with zinc and cadmium. The crystal structure of the imidazole complex $[Zn(imidH)_6]Cl_2.4H_2O$ shows a zinc atom octahedrally coordinated by six planar imidazole rings with Zn-N distances in the range 2.183-2.196Å [188]. Thermodynamic parameters for the interaction of Zn^{2+} with 1-methylimidazole have been measured [I89]. The zinc complex ZnL_2 (LH = 4-(3'.3'.3'-trifluoro-2'-oxopropylidene)-2.2.5.5-tetramethylimidazolidine-1-oxyl) where the ligand coordinates through the oxygen atom of the hydroxy group and the N atom of the imine group has been described [190]. Zinc complexes with substituted imidazole ligands and substituted phenols as anions have been reported [191]. The crystal structure of bis(4.4'.5.5'-tetramethyl-2.2'-biimidazole)zinc(II)$_{0.90}$copper(II)$_{0.10}$dinitrate consists of discrete $[Zn(Me_4bim)_2ONO]^+$ cations giving an approximately square pyramidal N_4O environment about zinc [192]. The crystal structure of the imidazole-bridged bimetallic center in the copper-zinc superoxide dimutase has been described [193] as has the structure of the zinc complex with cyclo(L-methionyl-L-histidyl) [194]. Stability constants for the interaction of Zn^{2+} with 2,4-dimethyl and 2,7-dimethylbenzimidazole have been determined [195]. Zinc and cadmium complexes with benzimidazole and 2-methylbenzimidazole have been reported [196]. Formation constants for zinc and cadmium chelates of ((1H-benzimidazol-2-ylmethylene)amino)phenol have been measured [197] and the zinc complex with 5-nitrobenzimidazole reported [198]. The 2-methyl-5-nitrobenzimidazole and metronidazole adducts of $ZnCl_2$ are thought to be tetrahedral [199] as are zinc and cadmium complexes of 2-aminobenzothiazole [200]. Some complexes ZnL_2X_2 and ZnL_4X_2 (L = 6-methyl-2-aminobenzothiazole, X = I,NCS,OAc) have been described [201]. The synthesis and structures of some 1,2,4-triazole and substituted triazole complexes with zinc have been described [202-204]. The crystal structure of $[Zn_3L_6(H_2O)_6](CF_3SO_3)_6$ (L = 4-ethyl-1,2,4-triazole) shows linear ligand bridged trinuclear cations with the central zinc atom octahedrally coordinated by six nitrogen atoms and the terminal zincs by three nitrogen atoms and three oxygen atoms of water molecules [205]. The ligand 1,4,7-trizacyclononane-N,N',N''-tris(2-ethanesulphonate) provides a N_3O_3 donor set in its zinc complex [206]. Complexes of (17) appear to involve the ligand via deprotonated nitrogen [207].



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Zinc halide complexes with 4,6-dimethylpyrimidine-2(1H)-one have been described [208]. The complexes $Zn(LH)_2Cl_2$ and ZnL_2 (LH = 5-aminotetrazole) are polymeric with bridging ligands [209]. Stability constants for a number of aminoalkyltetrazole complexes have been determined [210]. ESR studies on zinc pyrazine complexes doped with Mn(II) show these compounds to be polymeric with pz bridges [211]. An infra-red spectroscopic study of the pyrazole adduct of $ZnSO_4$ has been reported [212].

1.1.5 Complexes with phosphorus, arsenic and carbon donor ligands

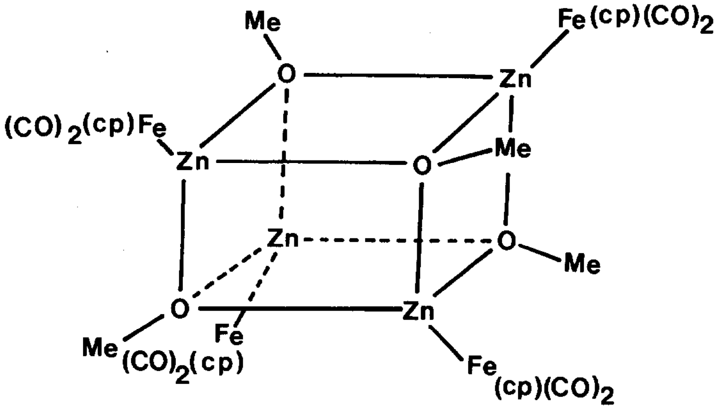
The far infra-red, Raman and phosphorus-31 NMR spectra of 1:1 and 1:2

adducts of zinc and cadmium halides with tricyclohexylphosphine have been studied [213]. Zinc and cadmium complexes of 2-tertiary arsinobenzoic acids contain the ligands as oxygen-arsenic donors [214].

Gas-phase electron diffraction studies on dimethyl-, diethyl- and dipropylzinc show C-Zn-C angles of about 114° [215]. *Ab initio* molecular orbital calcultions on dimethylzinc indicate that the *3d* electrons of zinc are non-bonding and that the Zn-C bonds are pure σ-bonds [215]. The synthesis and structure of zincacyclopentane, zincacyclohexane and zincacycloheptane have been described [216] and appear to exist exclusively as dimers in thf solution. A study of dinorbornylzinc complexes has been reported [217]. The crystal structure of a trimetallic neopentylidyne complex shows zinc in a tetrahedral C_2Cl_2 environment [218].

1.1.6 *Complexes with transition metal complexes as donor ligands*

The crystal structure of the $[Zn(Fe(cp)(CO)_2)_3]^-$ anion is the first for a tetrametal trigonal-planar zinc complex and shows Zn-Fe distances of 2.541Å [219]. The synthesis of a series of zinc(transition-metal carbonyl) alkoxides has been reported and the crystal structure of $[ZnFe(cp)(CO)_2OMe]_4$ (18) described [220].



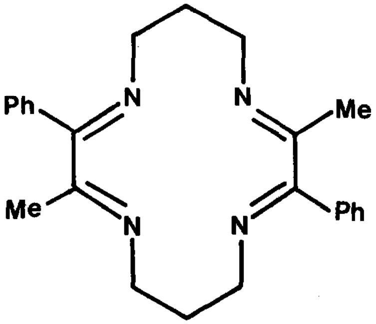
(18)

In the presence of Lewis bases $[ZnFe(CO)_4]_n$ complexes undergo mild stepwise reduction to give the kinetically stable $[Zn\{Fe(CO)_4\}_2]^{2-}$ anion [221]. Zinc and cadmium halides react with $[Fe_2(cp)_2(CO_3)(CNMe)]$ to give adducts, dimer scission, or both [222]. Reactions of allylic and propargylic amines and ethers with $Zn\{Co(CO_4)\}_2$ have been studied [223]. The crystal structure of $\{Zn(cp)\}_2Co(Cp)P(C_6H_5)_3$ has been determined [224] as has been the structure for

$(CH_3)_2N(CH_2)_3ZnW(cp)(CO)_3$ [225]. The structure of the metal-cluster compound $[Zn_4Ni_2(cp)_6]$ has also been described [226]. The dinuclear compound $ZnPt(th)Cl_3$ (th = thiamine) contains a two coordinate zinc atom bonded to platinum [227].

1.1.7 *Macrocyclic complexes*

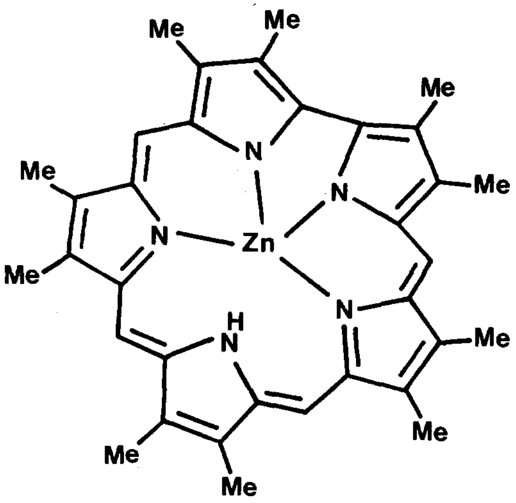
Formation constants for zinc complexes of tetramethylcyclam [228] and N,N',N''-tris(2-hydroxyethyl)-1,4,7-triazacyclononane [229] have been measured. The synthesis of a mono-N-functionalised tetraazamacrocycle zinc complex has been described [230]. Axial ligand derivatives of the macrocyclic complex **(19)** of zinc with one axial ligand (Cl,Br,I,py or Me_2benzimidazole) or two axial ligands (both I) are described [231].



(19)

The imine carbon-13 chemical shifts of these complexes are affected most by coordination number and to a lesser extent by the axial ligand. Equilibrium data for the interaction of Zn^{2+} with N,N',N''-tris(2-(N-hydroxycarbamoyl)ethyl)-1,3,5-benzenetricarboxamide have been determined [232]. Reversible oxidation and re-reduction of entire thin films of zinc phthalocyanines has been observed [233]. An electrochemical study of $Zn(Pc\text{-}CN_8)$ ($Pc\text{-}CN_8$ = octacyanophthalocyanine) has established the existence of a stable tetraanion $[Zn(Pc\text{-}CN_8)]^{4-}$ in non-aqueous solution [234]. The physical and chemical properties of a zinc complex of a sapphyrin **(20)** show it to be aromatic similar to porphyrins and corroles [235]. Stability data and crystal structure of zinc and cadmium complexes of a series of O_2N_3 donor macrocycles show that systematic minor perturbations of ligand structure can lead to a dramatic dislocation in geometry at one point along a series of metal complexes [236].

Zinc complexes of some cyclic urethanes **(21)** appear to be coordinated only through oxygen atoms [237].



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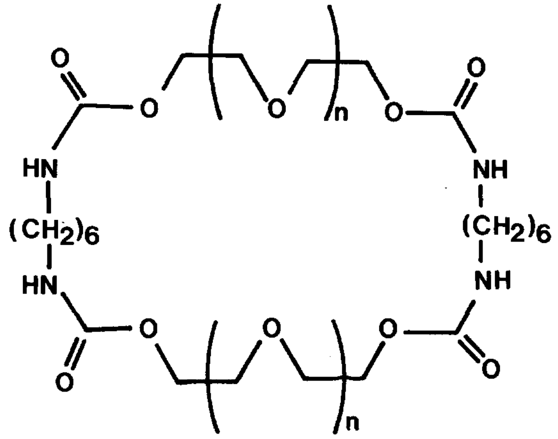
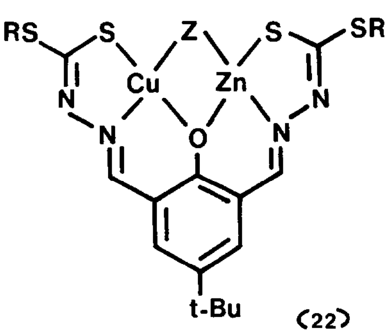


n=1,2

(21)

The synthesis and characterisation of some soluble bimetallones (22) have been described [238]. Equilibrium constants for the formation of some mononuclear and dinuclear macrocyclic complexes of zinc with (23) have been determined [239] as have stability constants for some polyaza-crown ether complexes [240]. The ligand (24) forms pentadentate complexes with zinc and cadmium [241].



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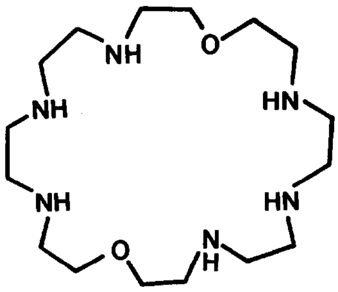

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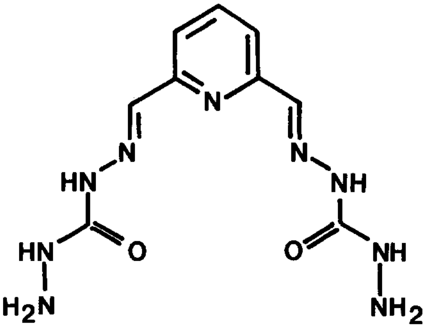
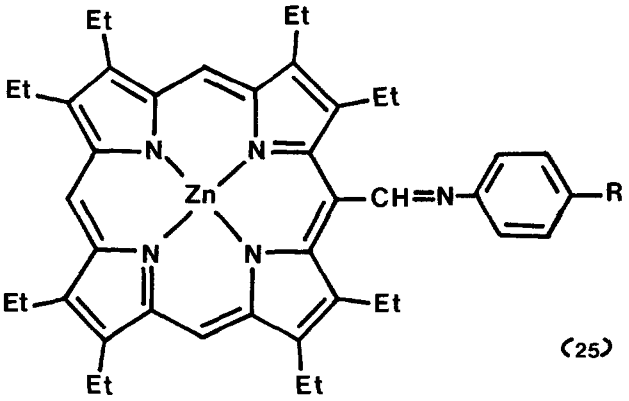


(24)

Carbon-13 and proton NMR spectra of some porphyrin complexes where the quality of the spectra improves with the addition of excess pyrrrolidine have been described [242]. Photophysical properties of some water soluble zinc porphyrins have been studied [243]. Proton NMR was used to study some capped porphyrin derivatives [244]. The IR and mass spectra of zinc porphyrin Schiff base complexes **(25)** have been reported [245]. A full pH study of incorporation of Zn^{2+} into uroporphyrin has been made [246]. Equilibrium studies of complexation of zinc tetraphenylporphyrin by adsorbed poly(4-vinylpyridine) have been reported [247]. Coordination kinetics of tetra(5,6-dimethylbenzo)porphine with $Zn(OAc)_2$ in pyridine have been studied spectrophotometrically [248]. Some imidazolato metalloporphyrins have been synthesised and studied as cytochrome oxidase models [249]. The synthesis and characterisation of zinc complexes $Zn(hp)(H_2O)$, $Zn(hp)(HCl)$, $Zn(hp)(HCl)_2$ and $Zn(hp)(HBr)$ (hpH_2 = **(26)**) have been reported [250].



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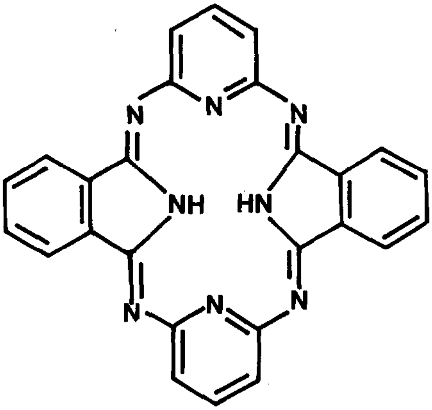


(26)

The decrease in rate constants of Zn^{2+} incorporation into fully and partially benzo-15-crown-5, *meso*-substituted porphyrins in the presence of cations has been explained in terms of cation complexation ability of the crown ether cavities [251]. Complexation of zinc tetraphenylporphyrin by 2,6-lutidine and dmso has been studied by UV-visible spectroscopy [252]. Some metalloprotoporphyrin films prepared by electro-oxidation have been investigated [253]. Polyvalent porphyrins of zinc have been studied [254]. Spectroscopic studies on the reaction of superoxide ion with zinc tetraphenylporphyrin have been reported [255]. The photochemical activation of zinc-carbon and zinc-sulphur bonds in zinc porphyrins has been studied [256].

1.2 *Cadmium(II)*

1.2.1 *Halide and pseudohalide complexes*

The preparation of some phosphorus-quaternary compounds have been described [257] and IR bands proposed for $[CdCl_4]^{2-}$, $[CdBr_4]^{2-}$ and $[Cd_2Cl_6]^{2-}$ species. An infra-red study of some thiosemicarbazide adducts of $CdCl_2$ shows the existence of a $[Cd_3Cl_8]^{2-}$ species [258]. The vibrational spectra of some solid pyridinetrihalocadmiate complexes have been described [4]. Equilibrium constants for cadmium bromide complexes have been measured [259]. Solvent extraction of Cd^{2+} from thiocyanate solutions has been studied [260].

1.2.2 *Complexes with oxygen donor ligands*

1.2.2.1 *Inorganic molecules and ions*

The crystal structure of $Cd(OH)(NO_3)$ shows a hexa-coordination about cadmium by three hydroxyl and three nitrate groups [261]. A competing ligand method has been used to study formation of monomeric Cd^{2+} hydroxy complexes [262]. Stability constants for $[Cd(OH)_3]^-$ were obtained by determining the solubility of $Cd(OH)_2$ in LiOH. NaOH and KOH solutions [263]. The crystal structure of $Cd_4Na(VO_4)_3$ has been redetermined [264]. Chlorosulphate acts as a unidentate oxygen donor in compounds $CdL_4(SO_3Cl)_2$ (L = py,CH_3CN) [17].

1.2.2.2 *Simple organic ligands*

The crystal structure of the 1,2-ethanediol complex $[CdCl_2(C_2H_4(OH)_2]_3.C_2H_4(OH)_2$ shows a chlorine-bridged endless chain with cadmium in a O_2Cl_4 environment [265]. The bimetallic isopropoxide $Cd[Al(O^iPr)_3]_4$ has been described [19]. A polarographic study of complexes between mono-, di- and tri-ethanolamines has been reported [21] as has a study of mixed-ligand complexes of Cd^{2+} with some carboxylic acids and ethylenediamine [266]. Formation constants for Cd^{2+} with some monocarboxylic acids have been reported [267]. The structure of dichloro(L-proline)cadmium(II) hydrate is a one dimensional polymer bridged by chlorine atoms and a carboxyl group [268]. In the structure of $NH_4[Cd(HCOO)_3]$ each cadmium atom is in a distorted octahedral environment with all the formates being bidentate and bridging to other cadmium atoms [269]. Antimicrobial activity of mixed-ligand complexes with HO-8-quin and various salicylic acids have been studied [270]. A polarographic study of mixed-ligand complexes with mercaptobenzoate and oxalate shows the presence of tetraionic species [271]. Trimethylamine adducts of some cadmium monocarboxylates have been described [272]. The crystal structure $CdL_2.7H_2O$ (L = *p*-hydroxybenzoate) shows a dimeric complex with cadmium atoms in a $O_5O'_2$ environment [273]. Stability constants for the interaction of Cd^{2+} with py-2,6-$(CO_2H)_2$ have been measured [41]. Formation constants for cadmium complexes with hydroxamic acids [45], salicylic acids [47] and an anthranilic acid [49] are reported. The electrosynthesis of $Cd(acac)_2$ has been reported [54, 274]. The crystal structure of $Cd(acac)_2(phen)$ shows a distorted octahedral O_4N_2 environment about cadmium [274]. The anionic ligand $[(cp)Co\{P(O)(OMe)_2\}_3]^-$ acts as a phosphineoxide donor towards Cd^{2+} [275]. The complex CdI_2L_2 (L = isobutyric acid amide) is tetrahedral with the ligand acting as a simple oxygen donor [276].

1.2.3 Complexes with sulphur donor ligands

The structure of the $[Cd_4(SPh)_{10}]^{2-}$ anion consists of an adamantane-like $Cd_4(\mu\text{-}S)_6$ cage composed of a distorted Cd_4 tetrahedron and a highly and irregularly distorted S_6 octahedron [277]. The crystal structure of bis(carbethoxymethanethiolato)cadmium(II) consists of infinite linear chains along which cadmium atoms are in alternating tetrahedral S_4 and dodecahedral S_4O_4 environments [278]. Cadmium halide adducts with imidazoline-2-thione and its 1-methyl analogue contain the ligand as simple sulphur donors [92]. Potentiometric and polarographic studies of cadmium complexes with 2-mercaptopropionic acid [279] and dithiopropionic acid [280] have been described. A variety of cadmium dithiocarbamate complexes have been described [81, 84, 281] as have cadmium complexes with phosphinesulphides [75, 76]. The structure of the complex $Cd(tmtu)_2(NO_3)_2$ consists of an approximately tetrahedral S_2O_2 environment about the cadmium atom [282] whereas in the structure of $CdL_4(NO_3)_2$ (L = N,N'-dimethylthiourea) the cadmium possesses a very distorted tetrahedral S_4 environment [283]. Thermodynamic parameters for mixed complexes with tu and nitrite or thiosulphate ions have been reported [284]. Some disubstituted thiourea complexes of cadmium have been described [285]. Polarographic studies of mixed-ligand thiolactate and glutamate complexes have been reported [286, 287] as has been a polarogaphic study on mixed-ligand complexes of thioacetamide, thiocyanate and nicotinamide [288].

1.2.4 Complexes with nitrogen donor ligands

1.2.4.1 Amines

Both the compounds $Cd(NH_3)_4I_2.I_2$ and $Cd(NH_3)_4(I.I_2)_2$ contain four coplanar nitrogen atoms and two axial iodide ions with form a *quasi*-octahedral environment about cadmium [289]. The cadmium atoms in CdI_2(*p*-toluidine)$_2$ are in a distorted tetrahedral N_2I_2 environment [290]. The compound $[Cd(NH_3)_2Ni(CN)_4]$ has an infinite layer structure with each cadmium cooordinated by four cyano nitrogen and two amine nitrogen atoms [291]. Stability constants of protonated, non-, and partially chelated complexes with en have been measured [292]. Mixed-ligand complexes of Cd^{2+} with en and diaminopropane have been investigated [293]. The crystal structure of the hydrated adduct of CdI_2 with hmta shows two differently coordinated cadmium atoms [294], one is in an octahedral $N_2O_2I_2$ environment and the other has a distorted tetrahedral NI_3 environment. The structure of the hydrated adduct of $Cd(NO_3)_2$ with hmta comprises chains in which cadmium atoms have an $O_2O'_2N_2$ environment and are linked by bridging hmta groups [295].

1.2.4.2 Schiff bases, hydrazones and related donors

The cadmium analogues of many of the zinc Schiff base complexes reported in the section on zinc have been reported and in particular references [116, 117, 119, 120, 122, 123, 126-128, 130, 133, 136] describe complexes of both metals. The cadmium complex with the Schiff base derived from benzoin and diaminopropane is thought to be tetrahedral [296]. Complexes $CdLX_2$ (L = N,N'-bis(acetoacetanilide); X = Cl,Br,I,NO_3,SCN,ClO_4) have been described [297]. Cadmium complexes with the condensation product of biacetylmonoxime and N,N-dimethylglycine hydrazide hydrochloride are thought to be coordinated through a N_2O_2 ligand donor set [298]. The cadmium complexes of some salicylaldoximates have been investigated [299]. Many hydrazone and thiocarbazone complexes have already been described in the section on zinc [137, 138, 142-146]. Cadmium complexes with acetone and furfural selenosemicarbazones have been described with the ligands being either monodentate through selenium or bidentate through selenium and the hydrazine nitrogen [300, 301]. Stability constants for cadmium complexes with *p*-nitrobenzaldehyde thiosemicarbazone have been determined [302]. Some complexes with substituted salicylhydrazines appear to be oxygen-bonded [303]. The structure of dichloroiodo(methylisothiocarbonohydrazidium)cadmium(II) consists of polymeric chains of edge-shared octahedra with the cadmium atom in a NCl_4I environment [304]. The crystal structure of potassium cadmium azide

$K_2Cd(N_3)_4$ shows cadmium octahedrally surrounded by six azide groups [305]. A somewhat similar structure is found in $Tl_8Cd_3(N_3)_{14}$ [306].

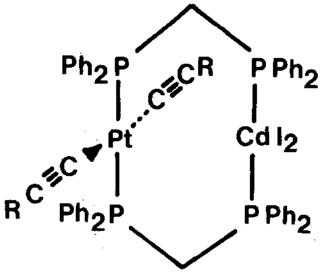
1.2.4.3 *Heterocycles*

Many cadmium complexes with heterocycles closely resemble the corresponding zinc complexes [171, 173, 175, 180, 182, 186, 187, 196-198, 200-203, 207-210]. Solubilities of $Cd(OAc)_2$ in pyridine-hexane and in dmf-benzene solutions were determined as a function of solvent composition and a number of mixed-solvate complexes identified [307]. The coordination and extraction properties of N-(pyrid-2-yl)thiobenzamide have been studied [308]. Mixed-ligand anionic complexes of $CdCl_2$ with a variety of substituted pyridines have been characterised [309] as have mixed-ligand complexes with oxime and pyridines [310]. The ligand bis(2-(picolinoylamino)ethyl)sulphide acts as a tetradentate without involvement of sulphur [311]. The complexing ability of bipy towards Cd^{2+} has been studied polarographically in aqueous-ethanol mixtures [312].

The compound $CdCl_2L_2$ (L = 5-amino-1,2,3,4-thiatrazole) has been prepared and characterised [313]. The structure of $CdL_2(NCS)_2$ (L = triazole) shows cadmium octahedrally coordinated by two triazole ligands and four bridging NCS groups giving a N_4S_2 environment [314]. Some 1:1 adducts between $CdCl_2$ and $CdBr_2$ with 2-(2'-pyridyl)imidazoline have been described [315]. The crystal structure of $Cd(NCS)_2L_2(H_2O)_2$ (L = dimethyltriazolopyrimidine) shows cadmium in a distorted octahedral $N_2N_2'O_2$ environment [316]. Thermal studies of some cadmium xanthine complexes have been reported [317] and the physical properties of a $CdCl_2$ adduct with adenine described [318]. The complex CdL_2 (LH = 2-thiobarbituric acid) contains the ligand coordinated through nitrogen and not sulphur [319].

1.2.5 *Complexes with phosphorus and arsenic donor ligands*

The structure of $CdI_2P(C_2H_5)_3$ is dimeric with iodide bridges giving cadmium a tetrahedral PI_3 environment whereas the structure of $CdCl_2P(c\text{-}C_6H_{11})_3$ is tetrameric with two cadmium atoms each having PCl_4 and PCl_3 environments [320]. Vibrational and phosphorus-31 spectra of 1:1 adducts of cadmium halides with tricyclohexylphosphine are described [213]. The heterobimetallic complex (27) has been synthesised and characterised by phosphorus-31 NMR spectroscopy [321]. Various cadmium halide complexes with arylarsolanes have been reported [322].



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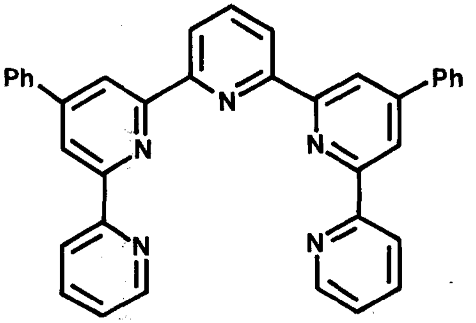
1.2.6 *Macrocyclic complexes*

Some macrocyclic complexes containing cadmium have already been referred to in the corresponding section on zinc [228, 236, 241, 248, 255]. The preparation and electrochemistry of the cadmium complex with **(28)** have been described [323].



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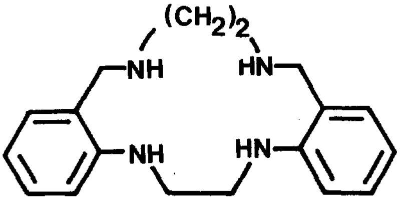
The crystal structure of the 1:1 compound of CdI_2 with **(29)** shows cadmium in a distorted square pyramidal N_3I_2 environment [324]. Incorporation of Mn(II) into $tsppH_2$ **(30)** is catalytically assisted by Cd^{2+} at concentrations as low as $10^{-7}mol.dm^{-3}$ [325].

1.2.7 *Cadmium-113 NMR spectroscopy*

The ^{113}Cd NMR shifts for a number of CdX_2L (L = 2,6-diacetylpyridine(bis(imine)); X = Cl,NCS,I,OAc) complexes have been measured and show a dependence on the coordination number of the cadmium [326].



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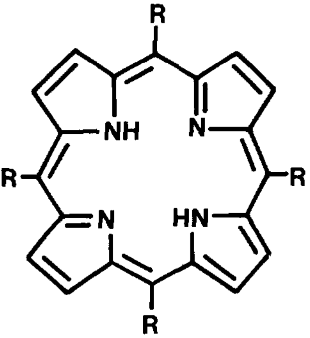

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The solid state ^{113}Cd NMR spectrum of $[CdL_4](NO_3)_2$ (L = N,N'-dimethylthiourea) has been measured [283] as has been the spectrum for $[CdL_2(NO_3)_2]$ (L = 1,1,3,3-tetramethyl-2-thiourea) [282]. The single crystal cadmium-113 NMR spectrum of $Cd(NO_3)_2.4H_2O$ shows that shielding tensors do not exhibit axial symmetry [327]. Carbon-13 and ^{113}Cd NMR spectra have been re-recorded for a number of cadmium amino acid complexes and imply formation of five-membered chelate rings involving cadmium [328].

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