Coordination Chemistry Reviews, 17 (1975) 317—342

X-RAY BIBLIOGRAPHY

H. CLEARY STYNES

York University, Downsview, Ontario, M3J 1P3 (Canada)

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The structures of the following compounds appeared in :
Inorg. Chem., Vol. 13, No. 11, November 1974

(i) Chloro[bis[2-[(-pyridylmethyl)imino]phenyl]disulfide]nickel(II) perchlorate, [Ni(py—CH=N—ph—S—S—ph—S—S—ph—N=CH—py)Cl]-(ClO_4)
(ii) Tris(bicarbonato)tetraaquoholmium(III) dihydrate, $Ho(H_2O)_4$-$(HCO_3)_3,2H_2O$
(iii) Dicarbonylpentamethylcyclopentadienylchromium dimer, a complex with a Cr≡Cr bond, $[(\pi\text{-}(CH_3)_5C_5)Cr(CO)_2]_2$
(iv) Crystallographic data of some substituted-pyridinium antimony(III) bromide salts

Inorg. Chem., Vol. 13, No. 12, [illegible]ember 1974

(i) A polymeric copper(I) [illegible]phatic disulfide complex; {bis[2-(2-pyridyl)-ethyl] disulfide}copper(I) perchlorate, $Cu(C_5H_4NCH_2CH_2SSCH_2CH_2$-$C_5H_4N)ClO_4$
(ii) A 2:1 octahydrate complex of 1,4,7,10-tetraoxacyclododecane with sodium hydroxide, $NaOH.2C_8H_{16}O.8H_2O$
(iii) The dimers of tris(thiourea)copper(I) tetrafluoroborate, $Cu(tu)_3BF_4$ and tris(*s*-dimethylthiourea)copper(I) tetrafluoroborate, $Cu(s\text{-}dmtu)_3$-BF_4
(iv) Structures of metallocarboranes IV. The *nido* metallocarborane complex; 8-η-cyclopentadienyl-6,7-dicarba-8-cobalta-*nido*-nonaborane(11), 8-η-C_5H_5-8-Co-6,7-$C_2B_7H_{11}$, at −160°
(v) Iodo[1,1,1-tris(diphenylphosphinomethyl)ethane] nickel(I), $Ni(p_3)I$
(vi) A non-stoichiometric hydrido complex of nickel with the tetradentate ligand tris(2-diphenylphosphinoethyl)amine, $[NiH_{0.5}(np_3)](BF_4)$ and $[Co(np_3)]BF_4$
(vii) *N*-Methylphenethylammonium trichlorocuprate(II), $[(C_6H_5)CH_2CH_2$-$NH(CH_3)H^+](CuCl_3^-)$
(viii) 4,4′-Dinitro-*trans*-stilbenebis(triphenylphosphine)platinum, $Pt[(C_6H_4NO_2)CHCH(C_6H_4NO_2)][P(C_6H_5)_3]_2$

(ix) μ-*trans*-1,1,2,3,4,4-Hexacyanobutenediido-bis[(carbonyl)bis(triphenylphosphine)rhodium], $[Rh(CO)[P(C_6H_5)_3]_2]_2(HCBD)$ where HCBD = *trans*-1,1,2,3,4,4-hexacyanobutenediide
(x) Ferrous phosphate, $Fe_3(PO_4)_2$
(xi) (1,1'-Trimethylene-π-dicyclopentadienyl)hafnium dichloride, $(CH_2)_3(\pi\text{-}C_5H_4)_2HfCl_2$
(xii) Dinitro(1,4,7,10-tetraazacyclodecane)cobalt(III) chloride, $[Co(cyclen)(NO_2)_2]Cl$
(xiii) Transition metal dimers III. Copper(II) di-μ-cyanato, di-μ-thiocyanato and di-μ-selenocyanato complexes, $[Cu_2(tren)_2(OCN)_2](BPh_4)_2$, $[Cu_2(tren)_2(CNS)_2](SCN)_2](BPh_4)_2$
(xiv) Sulfate coordination; chlorosulfatonitrosylbis(triphenylphosphine)ruthenium(II), $RuCl(SO_4)(NO)(PPh_3)_2$
(xv) Mercury(II) hydroxide nitrate, $Hg(OH)NO_3$

J. Amer. Chem. Soc., Vol. 96, No. 19, September 18, 1974
(i) Cyclohexyllithium, a hexameric organolithium, $[C_6H_{11}Li]_6(C_6H_6)_2$
(ii) Tris(dimethylaminato)tris(*N*,*N*-dimethylcarbamato)tungsten(VI), $W[N(CH_3)_2]_3[O_2CN(CH_3)_2]_3$

J. Amer. Chem. Soc., Vol. 96, No. 20, October 2, 1974
(i) $(\Pi\text{-}C_5H_5)Co(\Pi\text{-}C_5H_4.B_9C_2H_{11})$
(ii) Tricarbonylmanganese tridecahydrooctaborate, $(CO)_3MnB_8H_{13}$
(iii) 4-Chlorobenzoic acid
(iv) *cis*-Azobenzene dioxide, $C_{12}H_{10}N_2O_2$ and *trans*-2,2'-dicarboxyazobenzene dioxide, $C_{14}H_{10}N_2O_6$
(v) $[(FeL)_2NO]PF_6$.acetone, where L = *N*,*N*'-dimethyl-*N*,*N*'-bis(β-mercaptoethyl)ethylenediamine
(vi) The bis(triphenylphosphine)platinum complex of $\Delta^{1,4}$-bicyclo[2.2.0]-hexene, $Pt(C_6H_8)(PPh_3)_2$ and $Pt(C_6H_9OEt)(PPh_3)_2$

J. Amer. Chem. Soc., Vol. 96, No. 21, October 16, 1974
(i) Potassium calcium hexanitrocuprate(II), $K_2CaCu(NO_2)_6$
(ii) Neutron diffraction studies of the metal—hydrogen—metal bond I. The symmetric, bent, three-center, two-electron molybdenum—hydrogen—molybdenum bond in μ-hydrido-μ-dimethylphosphido-bis(η^5-cyclopentadienyldicarbonylmolybdenum), $Mo_2(\eta^5\text{-}C_5H_5)_2(CO)_4(\mu_2\text{-}H)(\mu_2\text{-}P(CH_3)_2)$
(iii) X-ray and neutron diffraction studies on μ-hydrido-nonacarbonyl-(nitrosyl)ditungsten, $[HW_2(CO)_9(NO)]$
(iv) The dimer of diphenylantimony trichloride, $[(C_6H_5)_2SbCl_3]_2$

J. Amer. Chem. Soc., Vol. 96, No. 22, October 30, 1974
(i) The metallooxacyclobutane complex, $Pt[C_2(CN)_4O][As(C_6H_5)_3]_2$
(ii) Bis(pyridine)osmate(VI) ester of adenosine

J. Amer. Chem. Soc., Vol. 96, No. 23, November 14, 1974

(i) *nido* 11-Atom metallocarboranes and their Lewis base adducts
(ii) X-ray and neutron diffraction studies of cubic tetracyanoethylene, TCNE
(iii) The aluminotitanium hydrides, $[(C_5H_5)Ti]_2(H)(H_2AlEt_2)(C_{10}H_8)$ and $[(C_5H_4)TiHAlEt_2]_2(C_{10}H_8)$

J. Amer. Chem. Soc., Vol. 96, No. 24, November 27, 1974

(i) Bis[(hydridotris(1-pyrazolyl))copper(I)], $Cu[HBpz_3]$
(ii) Diaqua(2,6-diacetylpyridinebis(semicarbazone))nickel(II) nitrate, DAPSC.$Ni(NO_3)_2.3H_2O$ and diaqua(2,6-diacetylpyridinebis(semicarbazone))copper(II) nitrate, DAPSC.$Cu(NO_3)_2.3H_2O$, where DAPSC = 2,6-diacetylpyridinebis(semicarbazone)
(iii) μ-Di(η^5:η'-cyclopentadienyl)dithorium(IV), $[(C_5H_5)_2ThC_5H_4]_2$
(iv) Hexameric trimethylsilyllithium, $(LiSiMe_3)_6$
(v) The linear $[(Ph_3P)_2N]^+$ cation in $[(Ph_3P)_2N]^+[V(CO)_6]^-$

J. Amer. Chem. Soc., Vol. 96, No. 25, December 11, 1974

(i) Iron(II) complexes of a new completely conjugated macrocyclic ligand from 2,6-diacetylpyridine and hydrazine: structure of the bis-acetonitrile complex $[Fe(C_{18}H_{18}N_6)(CH_3CN)_2](ClO_4)_2$
(ii) Carbon monoxide and methyl groups as symbiotic *trans* ligands in an iron(II) complex; structure of $[Fe(C_{10}H_{19}N_8)(CO)(CH_3)]$, an iron-macrocyclic complex

J. Amer. Chem. Soc., Vol. 96, No. 26, December 25, 1974

(i) A new cyanomanganate(III) complex, hexapotassium μ-oxo-bis-[pentacyanomanganate(III)] cyanide, $K_7[Mn_2O(CN)_{10}]CN$

J. Chem. Soc. Dalton, No. 12, 1974

(i) Structural studies of eight-coordinate metal complexes I. Tetrakis-(phenyldithioacetato)vanadium(IV) and tetrakis(dithiobenzoato)-vanadium(IV)
(ii) Crystal structures of thiocyanate polyamine copper(II) complexes VI. Di-(3-aminopropyl)aminebis(isothiocyanato)copper(II), Cu(dpt)-$(NCS)_2$
(iii) π-Cyclopentadienyl-η-(tetraphenylborato)ruthenium(II), (π-C_5H_5)-$Ru(BPh_4)$
(iv) Metal—carbonyl and metal—nitrosyl complexes XV. Structure of (3-formyl-*N*-ethoxycarbonylazepine)tricarbonyliron(0)
(v) Structural theory for non-stoichiometry I. Lanthanoid oxides MO_x with $1.7 < x < 2.0$, M = Pr
(vi) Studies on sulphur—nitrogen compounds I. Structure of bis(diphenylmethylene)trisulphurtetranitride, $[(C_6H_5)_2CNSN]_2S$

J. Chem. Soc. Dalton, No. 13, 1974

(i) 1,3-Bis(2-iminobenzylideneimino)propanenickel(II), [Ni(ab-tn)]
(ii) The adduct phosphorus pentafluoride–pyridine, $(C_5H_5N)PF_5$
(iii) Complexes with sulphur and selenium donor ligands IV. Structure of [1-(diphenylarsino)-2-(diphenylphosphino)ethane](O-methyl phosphorodithioato)nickel(II)-½ benzene, $[S_2PO(OMe)]—(Ph_2P\cdot C_2H_4\cdot AsPh_2)Ni, 0.5C_6H_6$

J. Chem. Soc. Dalton, No. 14, 1974

(i) Bis-(2,2′-bipyridyl)copper(II) bis[dichlorocuprate(I)], $[Cu(bipy)_2][CuCl_2]_2$
(ii) Alkylideneamido-derivatives of metals and metalloids VI. Structure of *trans*-$Rh[N{:}C(CF_3)_2][C(NMeCH_2)_2](PPh_3)_2$

J. Chem. Soc. Dalton, No. 15, 1974

(i) Tetraphenylarsonium aquotetrachlorohydroxotellurate(IV)
(ii) Trichlorobis-(*N*,*N*′-di-isopropylacetamidinato)tantalum(V), $TaCl_3[C_3H_7NC(Me)NC_3H_7]_2$
(iii) Carbene complexes VII. Structure of trichloro(dimethylaminomethylene)bis(triethylphosphine)rhodium(III), $[RhCl_3(PEt_3)_2CHNMe_2]$
(iv) Reinvestigation of $[Ru(NH_3)_5(NO)]Cl_3$, H_2O and *trans*-$[Ru(OH)(NH_3)_4(NO)]Cl_2$
(v) Steric effects in the reversible oxygenation of cobalt-Schiff-base complexes III. Structure of the *meso*-form of *N*,*N*′-cyclohexylenebis(salicylideneiminato)cobalt(II), [Co(*meso*-salschx)]
(vi) Dimethyl-[3,3′-(trimethylenedinitrilo)bis(butan-2-one oximato)]-cobalt(III), $C_{13}H_{25}CoN_4O_2$
(vii) (*N*,*N*′-Dimethylbenzylamine-2*C*,*N*)(*N*-phenylsalicylaldiminato)-palladium(II), (dmba)Pd(sal:NPh)

J. Chem. Soc. Dalton, No. 16, 1974

(i) A dinuclear dinitrogen complex: tetrachloro{chlorotetrakis[dimethyl(phenyl)phosphine]rhenium(1)}-μ-dinitrogenmethoxymolybdenum(V)-methanol-hydrochloric acid, $[(PMe_2Ph)_4ClReN_2MoCl_4(OMe)]$, CH_3OH, HCl
(ii) Tricarbonyliodotris(dimethylphenylphosphino)tungsten(II) tetraphenylborate, $[W(CO)_3(PMe_2Ph)_3I]^+BPh_4^-$
(iii) Molecular structures of non-geminally substituted phosphazenes VI. Structure of 2,*cis*-4,*trans*-6,*trans*-8-tetrachloro-2,4,6,8-tetraphenylcyclotetraphosphazatetraene, β-*trans*-$N_4P_4(Cl)_4Ph_4$
(iv) Di-μ-chloro-bis{chloro[2-(hydroxymethyl)pent-4-enyl]rhodium(III)}-methanol, $[RhCl_2(C_6H_{11}O)]_2$,MeOH
(v) Structural studies in main-group chemistry VI. Structure of 2,2′-bipyridyldichlorodiphenyltin, $Ph_2SnCl_2(bipy)$
(vi) Fluoride crystal structures XXII. Chloryl μ-fluorobis[pentafluoroantimonate(V)], $[ClO_2]^+[Sb_2F_{11}]^-$

(vii) *cis*-Bis-(*N*-isopropyldithiocarbamato)nickel(II)
(viii) *mer*-Tris-(*N*-benzylsalicylaldiminato)manganese(III)
(ix) A linear polymeric 1:1 adduct of copper(I) chloride and 2,3-diazabicyclo[2,2,1]hept-2-ene, CuCl, $C_5H_8N_2$
(x) {7,8,15,17,18,20-Hexahydrodibenzo[*e*,*m*]pyrazino[2,3-*b*][1,4,8,11]-tetraazacyclotetradecinato(2-)}nickel(II) and {7,8,15,16,17,18-hexahydrodibenzo[*e*,*m*][1,4,8,11]tetraazacyclotetradecinato(2-)}nickel(II)
(xi) Bis(2,2′:6′,2″-terpyridyl)cobalt(II) bromide trihydrate, $[Co(terpy)_2]Br_2, 3H_2O$
(xii) Nonacarbonyl-μ-(1,2,3,4-tetraphenylbutadiene-1,4-diyl)-*triangulo*-triosmium, $(Ph_4C_4)Os_3(CO)_9$
(xiii) *trans*-Di-iodobis(tricyclohexylphosphine)platinum(II), an overcrowded molecule, *trans*-$[PtI_2\{(C_6H_{11})_3P\}_2]$

J. Chem. Soc. Dalton, No. 17, 1974

(i) Phosphorus—fluorine chemistry XXXIV. Structure of the product of the reaction of phosphorus pentafluoride with 2-methyl-8-trimethsiloxyquinoline, $PF_4(oxMe)$
(ii) Crystal structure and spectroscopic properties of mercury(II) halide complexes II. The dimethyl sulphoxide—mercury(II) chloride (2/3) adduct, $2Me_2SO, 3HgCl_2$
(iii) Metal complexes of sulphur ligands VIII. Structure of *cis*-bis(diethylphosphinodithioato)bis(dimethylphenylphosphine)ruthenium(II), $[Ru(S_2PEt_2)_2(PMe_2Ph)_2]$
(iv) Bis(diethylenetriamine)zinc(II) dibromide monohydrate, $[Zn(dien)_2]Br_2, H_2O$
(v) Tetracarbonyl-(7,7-dimethoxynorborn-2-ene)chromium(0)
(vi) Pyramidal five-coordination with flattened tetrahedral basal bonds. Structures of dibromotris-(5-ethyl-5H-dibenzophosphole)palladium(II)-chlorobenzene, dibromotris-(5-ethyl-5H-dibenzophosphole)platinum-(II)-bromobenzene and dibromo-(5-methyl-5H-dibenzophosphole)-platinum(II)-bromobenzene
(vii) The high-spin five-coordinate complex aquo-*N*,*N*′-ethylenebis(3-methoxysalicylideneiminato)cobalt(II), [Co(3-MeOsalen)(H_2O)]
(viii) Thallium(I) fluoride, TlF
(ix) Polymeric cadmium(II) malonate monohydrate

J. Chem. Soc. Dalton, No. 18, 1974

(i) Dioxo(pyridine)bis(tropolonato)uranium(VI)
(ii) Tris(1,8-naphthyridine)(perchlorato)mercury(II)perchlorate, $[Hg(N_2C_8H_6)_3(ClO_4)][ClO_4]$ and tetrakis (1,8-naphthyridine)cadmium(II) bis(perchlorate), $[Cd(N_2C_8H_6)_4][ClO_4]_2$
(iii) Two trinuclear Schiff-base copper(II) complexes, $[\{Cu(es)\}_2Cu(H_2O)_2][ClO_4]_2, H_2O$, [es = *N*,*N*′-ethylenebis(salicylaldimine)] and $[\{Cu(eha)\}_2 Cu(H_2O)][ClO_4]$, [eha = *N*,*N*′-ethylenebis(*o*-hydroxyacetophenimine)]

(iv) Dichlorobis-(*N*,*N*-dicyclohexylacetamidinato)methyltantalum(V), $MeTaCl_2[C_6H_{11}\cdot N\cdot C(Me)\cdot N\cdot C_6H_{11}]_2$

(v) Studies on sulphur—nitrogen compounds II. Structure of triphenylphosphinetrisulphurtetranitride, $(C_6H_5)_3PN_4S_3$

(vi) Heteropolytungstate complexes of the lanthanoid elements III. Structure of sodium decatungstocerate(IV)—water(1/30) $Na_6CeW_{10}O_{36}H_2$, $30H_2O$

J. Chem. Soc. Dalton, No. 19, 1974

(i) 1-(Dicarbonyl-π-cyclopentadienylferrio)-2-(phenyl)-ethyne, $[(\pi\text{-}C_5H_5)Fe(CO)_2(C{:}CPh)]$

(ii) Di-μ-chlorobis-{[1-(dicarbonyl-π-cyclopentadienylferrio)-2-phenylethyne]copper(I)}, $[(\pi\text{-}C_5H_5)Fe(CO)_2Cl]\,[Cu(C{:}CPh)]$

(iii) [Iminobis(ethyleneoxy)]diphenylsilane, a five-coordinate silicon compound, $Ph_2Si(OCH_2\cdot CH_2)_2NH$

(iv) A palladium(II) carbene complex; 4-[(diethylamino)(*t*-butylamino)-methylene]-4-(*t*-butyl isocyanide)-2,2,5,5-tetrakis(trifluoromethyl)-1,3,4-dioxapalladolan, $\{[Pd.C(CF_3)_2O\cdot C(CF_3)_2O](CNBu^t)\text{-}[C(NEt_2)NHBu^t]\}$

(v) *cis*-2,4,6-Trichloro- and *cis*-2,4,6-tribromo-2,4,6-trifluorocyclotri(phosphazene)

(vi) Bis(*N*-cyclohexylthiopicolinamidato)copper(II)

(vii) Comparison of stereochemistry in racemic and optically resolved forms of a five-coordinate compound. Structures of dibromotris-(2-phenylisophosphindoline)-palladium(II)-acetone(orange), and dibromotris-(2-phenylisophosphindoline)palladium(II), red

(viii) Stereochemistry of the MX_4Y system, (X = unidentate, Y = bidentate ligand): tetrachloro-[1,2-bis(dimethylarsino)-3,3,4,4-tetrafluorocyclobut-1-ene]rhenium(IV), $[Re\{C_4F_4(AsMe_2)_2\}Cl_4]$

J. Chem. Soc. Dalton, No. 20, 1974

(i) Sodium tetratungstate, $Na_2W_4O_{13}$

(ii) Stereochemistry of model compounds for pyridoxal-catalysed reactions. Structures of the hydrated complexes bis(pyridoxylidene-DL-valinato)nickel(II) and bis(pyridoxylidene-L-valinato)zinc(II)

(iii) Surface and intercalate chemistry of layered silicates III. X-ray investigation of tetrahydropyran and 1,4-dioxan intercalates of montmorillonite

J. Chem. Soc. Dalton, No. 21, 1974

(i) Structural studies in main-group chemistry VII. Structure of *N*-benzoyl-*N*-phenyl-*O*-(triphenylstannyl)hydroxylamine, $Ph_3Sn(ONPh\cdot COPh)$

(ii) Metal—metal bonding in coordination complexes XII. Structure of tetracarbonyl(triphenylstibine)iron, $Ph_3SbFe(CO)_4$

(iii) Di-μ-chloro-dichlorobis(methyl pyrrolidine-1-carbodithioate)dimercury(II), $HgCl_2[MeS\cdot CS\cdot N(CH_2)_4]$

(iv) The dihydroperchlorate of 1,4,8,11-tetra-azacyclotetradecane(cyclam), $[C_{10}H_{26}N_4][ClO_4]_2$

Acta Crystallogr., Sect. B, Vol. 30, August 1974

(i) Complex of palladium(II) nitrate with the macrocyclic ligand 1-oxa-7,10-dithia-4,13-diazacyclopentadecane, $[Pd(C_{10}H_{22}N_2OS_2)](NO_3)$
(ii) Tetrachlorobis-[*o*-phenylenebis(dimethylarsino)]molybdenum(V) triiodide, $[MoCl_4(diars)_2]I_3$
(iii) γ-Ni_2SiO_4
(iv) $Hg_3O_2Cl_2$, a compound with three-coordinate mercury(II)
(v) Hydrogen bond studies LXXXVI. An asymmetric non-centred $H_5O_2^+$ ion: neutron diffraction study of picrylsulphonic acid tetrahydrate, $[H_5O_2]^+[C_6H_2(NO_2)_3SO_3]^-.2H_2O$
(vi) The bis-(5,5′-diethylbarbiturato)bispicoline complex of zinc(II)
(vii) Crystallographic studies of metal—nucleotide base complexes III. Dichlorobis-(9-methyl-6-oxypurine)diaquocopper(II) trihydrate $[(C_6H_6N_4O)_2CuCl_2\cdot 2H_2O].3H_2O$
(viii) Ammonium pentafluorouranate(N), β-H_4NUF_5
(ix) Thallium tetrametaphosphate, $Tl_4P_4O_{12}$
(x) Diaquo-μ-triethylenetetraaminehexaacetatodichromium(III) hexahydrate, $[Cr_2TTHA.2H_2O].6H_2O$
(xi) Ba_2Bi
(xii) Benzimidazole, $C_7N_2H_6$
(xiii) Ca_5Bi_3
(xiv) α-Mg_3Sb_2
(xv) The fluoroberyllate $KHoBeF_6$
(xvi) $(+)_{589}$-Tris-[(−)-*trans*-1,2-diaminocyclohexane]rhodium(III) nitrate trihydrate, $(+)_{589}$-$[Rh(-chxn)_3](NO_3)_3.3H_2O$
(xvii) Calcium hydrogen citrate trihydrate, $CaC_6H_6O_7.3H_2O$
(xviii) Tetrachloro-μ-dichloro-tetrakis(tri-*n*-butylphosphine)dirhodium(III), $Rh_2(PBu_3)_4Cl_6$
(xix) $BaWO_4$-II (A high-pressure form)

Acta Crystallogr., Sect. B, Vol. 30, September 1974

(i) Fe_6Ge_5 and Fe_6Ga_5
(ii) α-Stannous tungstate, α-$SnWO_4$, and its relationship to ferroelectric $SbTaO_4$ type compounds
(iii) The hexamolybdotellurate ion and telluric acid, $(NH_4)_6[TeMo_6O_{24}]\cdot Te(OH)_6.7H_2O$
(iv) Structural researches on organometallic π-complexes: structures of (π-cyanocyclopentadienyl)tetraphenylcyclobutadienecobalt, $(\pi\text{-}C_5H_4CN)Co[C_4(C_6H_5)_4]$, ($\pi$-iodocyclopentadienyl)tetraphenylcyclobutadienecobalt $(\pi\text{-}C_5H_4I)Co[C_4(C_6H_5)_4]$ and (π-1,2-diiodocyclopentadienyl)tetraphenylcyclobutadienecobalt, $(\pi\text{-}C_5H_3I_2)Co[C_4(C_6H_5)_4]$

(v) $Cu_2Li_2P_6O_{18}$
(vi) Er_3Ni_2
(vii) *facial*-Tris-(*R*-propylenediamine)cobalt(III) hexacyanocobaltate(III), dihydrate, $(+)_{589}$-$[Co(R\text{-pn})_3][Co(CN)_6].2H_2O$
(viii) 1,8-Bis-(2′-pyridyl)-3,6-diazaoctanecopper(II) perchlorate
(ix) *B*,*B*-Bis-(*p*-fluorophenyl)boroxazolidine, $(p\text{-}FC_6H_4)_2BO(CH_2)_2NH_2$
(x) Structural researches on metal complexes of polydentate ligands containing carbonyl and α-diimine moieties III. Bis{dichloro-[2,6-diacetylpyridinebis(picolinoylhydrazonato)]dicopper(II)}dihydrate, $Cu_2(DIP)Cl_2.H_2O$
(xi) Bis(ethylenethiourea)zinc(II) thiosulphate, $Zn[SC(NHCH_2)_2]_2S_2O_3$
(xii) Bis[diethyl bis-(1-pyrazolyl)borato]nickel(II), $[(CH_3CH_2)_2B(C_3N_2H_3)_2]_2Ni(II)$
(xiii) Tetraethylammonium bis-(2,3-quinoxalinedithiolato)nickelate(II) dihydrate, $[(C_2H_5)_4N]_2[C_6H_4N_2C_2S_2]_2Ni(II).2H_2O$
(xiv) The series $M^IM^{III}(XO_4)_2.nH_2O$, $KFe(CrO_4)_2.H_2O$
(xv) Kurnakovite, $Mg[B_3O_3(OH)_5](H_2O)_4.H_2O$
(xvi) $CdCl_2.2NaCl.3H_2O$, a mixed salt
(xvii) A new synthetic silicate, $K_4[Si_8O_{18}]$
(xviii) $K_4[Si_8O_{18}]$, a desymmetrized OD structure
(xix) Salt hydrates X. Heptasodium fluoride bisphosphate 19-hydrate and heptasodium fluoride bisarsenate 19-hydrate and the computer simulation of the isomorphous vanadate salt
(xx) Hydrazinium azide, $N_2H_5N_3$
(xxi) Manganese acetate tetrahydrate, $Mn(CH_3COO)_2.4H_2O$
(xxii) Dinitratobis(antipyrine)cobalt(II), $Co(C_{11}ON_2H_{12})_2(NO_3)_2$
(xxiii) Dinitratobis(antipyrine)copper(II), $Cu(C_{11}ON_2H_{12})_2(NO_3)_2$
(xxiv) Bis-π-cyclopentadienylmolybdenum-(μ-bismethanethiolato)-bis-π-allylrhodium hexafluorophosphate, $C_{18}H_{26}MoRhS_2PF_6$
(xxv) μ-Dichloro-bis-[(π-benzene-π-allyl)molybdenum(II)], $C_{18}H_{22}Cl_2Mo_2$
(xxvi) Thiazolidine-2-thionepentacarbonyltungsten, $(CO)_5WS{=}C{-}NH{-}CH_2{-}CH_2{-}S$
(xxvii) $(+)_{510}$-Oxalatobis-(*R*,*S*-2,4-diaminopentane)cobalt(III) perchlorate monohydrate, $(+)_{510}$-$[Co(ox)(R,S\text{-ptn})_2](ClO_4).H_2O$
(xxviii) $Ca(NO_3)_2.4CH_3OH$
(xxix) Bis(chlorocyclopentadienylzirconium)oxide, $[(\pi\text{-}C_5H_5)_2ZrCl]_2O$

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(i) La_2SnS_5
(ii) Diaquo-2,2′-bipyridylnickel sulphate, $Ni(C_{10}H_8N_2)(H_2O)SO_4$
(iii) Structures of bent bis-π-cyclopentadienyl—metal complexes:
(a) bis-π-cyclopentadienyldibromorhenium(V) tetrafluoroborate
(b) bis-π-cyclopentadienyldichloromolybdenum(IV)
(c) bis-π-cyclopentadienylhydroxomethylaminomolybdenum(IV) hexafluorophosphate

(d) bis-π-cyclopentadienylethylchloromolybdenum(IV)
(e) bis-π-cyclopentadienyldichloroniobium(IV)
(f) bis-π-cyclopentadienyldichloromolybdenum(V) tetrafluoroborate
(g) μ-oxo-bis[bis-π-cyclopentadienylchloroniobium(V)] tetrafluoroborate
(h) bis-π-cyclopentadienyldichlorozirconium

(iv) Bis[bis-(π-cyclopentadienyl)niobium(V)-bis-μ-methanethiolato]-nickel(0) tetrafluoroborate dihydrate, $C_{24}H_{32}Nb_2NiS_4(BF_4)_2.2H_2O$ and bis[bis-(π-cyclopentadienyl)molybdenum(IV)-bis-μ-methanethiolato] nickel(II) tetrafluoroborate, $C_{24}H_{32}Mo_2NiS_4(BF_4)_2$

(v) (*pentahapto*-Cyclopentadienyl)hydridomolybdenum-μ-dimethylaluminum-μ-{methylaluminumdi-[μ-*pentahapto*(*monohapto*)-cyclopentadienyl]dimethylaluminum}(*pentahapto*-cyclopentadienyl)hydridomolybdenum, $[MoH(C_5H_5)(C_5H_4)]_2Al_3(CH_3)_5$ and dimolybdenumbis-μ-{methylaluminumdi-[μ-*pentahapto*(*monohapto*)cyclopentadienyl]-dimethylaluminum}, $[Mo(C_5H_4)_2Al_2(CH_3)_3]_2$

(vi) Cyclotetra-μ-lithio-tetra[hydrido{bis-(π-cyclopentadienyl)}molybdenum] and its tungsten analogue, $[(\pi\text{-}C_5H_5)_2M(H)Li]_4$ M = Mo, W

(vii) β-1-Phenylsilatrane, $C_6H_5Si(OCH_2(CH_2)_3)N$

(viii) Dibromobis-[5-(2-hydroxyethyl)-4-methylthiazole]cobalt(II)

(ix) 1,3,3,5-Tetramethyl-6-(1',2'-naphtho)bicyclo[3,2,1]octenechromium(0) tricarbonyl, $C_{23}H_{24}O_3Cr$

(x) Sodium chloride dihydrate, $NaCl.2H_2O$

(xi) Triamines of cobalt(III) III. *mer*-Triazidodiethylenetriaminecobalt(III), $Co(dien)(N_3)_3$

(xii) Structural researches on metal complexes of polydentate ligands containing carbonyl and α-diimine moieties IV. Pentacoordination in dichloro-2-(2'-pyridyl)-3-(*N*-2-picolylimino)-4-oxo-1,2,3,4-tetrahydroquinazolinemanganese(II), $(C_{19}H_{15}Cl_2MnN_5O)$

(xiii) Zeolites of the phillipsite famiiy, phillipsite and harmotome

(xiv) High (γ)-Li_2BeSiO_4, a tetrahedral structure

(xv) Crystallographic studies of metal—nucleotide base complexes IV. Tetraaquo-(9-methyladenine)copper(II) sulphate monohydrate, $[(C_6H_7N_5)Cu(H_2O)_4]SO_4.H_2O$

(xvi) Tricesium-cyclo-tri-μ-oxo-tris(tetrafluoroantimonate), $Cs_3(Sb_3F_{12}O_3)$

(xvii) {5-(2-Hydroxyethyl)-4-methylthiazolium} tribromo-{5-(2-hydroxyethyl)-4-methyl-3-thiazolo}cuprate(II), $[C_6H_{10}NOS][CuBr_3(C_6H_9NOS)]$

(xviii) Magnesium divanadate, $Mg_2V_2O_7$

(xix) Hydrogen bond studies LXXXVII. A neutron diffraction study of ammonium trihydrogen selenite, $NH_4H_3(SeO_3)_2$

(xx) Dinitratobis(antipyrine)copper(II), another form

(xxi) Dirubidium-μ-oxo-bis(pentafluoroantimonate), $Rb_2(Sb_2F_{10}O)$

(xxii) Sodium copper pyrophosphate hexadecahydrate, $Na_6Cu(P_2O_7)_2.16H_2O$

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(i) Hydrogen bond studies XCII. A neutron diffraction study of $KHCO_3$ and $KDCO_3$, disorder in $(HCO_3)_2^{2-}$ and $(DCO_3)_2^{2-}$ dimers
(ii) Boron phosphides, $B_{12}(P_{1.36}B_{0.64})$
(iii) $Ag_8S_2SiO_4$
(iv) PdPS and PdPSe
(v) Potassium and rubidium amidoberyllates, $KBe(NH_2)_3$ and $RbBe(NH_2)_3$
(vi) The monoclinic, CrB-related, crystal structure of Tb_3Ni_2, Dy_3Ni_2 and Ho_3Ni_2
(vii) $Ca_3WO_5Cl_2$ and the configuration of the WO_5^{4-} ion
(viii) Bis-(5-ethyl-5-isoamylbarbiturato)bis(imidazole) complex of nickel(II), $NiC_{28}H_{42}N_8O_6$
(ix) A neutron-diffraction study of holmium ethylsulfate enneahydrate, $Ho(C_2H_5SO_3)_3.9H_2O$
(x) The OD structure of Na_2SnS_3
(xi) $(Pb_{1-x}, Bi_x)Bi_2Cu_2Cu_{2-x}S_5I_2$ ($x \simeq 0.88$)
(xii) V_3O_7
(xiii) Structural studies on the actinide carboxylates II. Structure of bis(iminodiacetato)dioxouranium(VI)
(xiv) Uranium tetrabromide, UBr_4
(xv) K_6MgO_4
(xvi) Hydromagnesite, $4MgCO_3.Mg(OH)_2.4H_2O$
(xvii) 1,2:3,4:5,6-Tris-(*o,o'*-biphenylylene)borazine, $C_{36}H_{24}B_3N_3$
(xviii) $Na_3Li(BeF_4)_2$
(xix) Rb_2CrF_5
(xx) The orthorhombic crystal structure of Ru_2Si_3, Ru_2Ge_3, Os_2Si_3 and Os_2Ge_3
(xxi) $Na_2KP_3O_9$
(xxii) Calcium binding to carbohydrates: structure of calcium ascorbate dihydrate, $Ca(C_6H_7O_6)_2.2H_2O$
(xxiii) Calcium L-ascorbate dihydrate
(xxiv) Bis-π-cyclopentadienyltungsten(IV)(bis-μ-benzenethiolato)metal(0) tetracarbonyls, $(\pi\text{-}C_5H_5)_2W(\mu\text{-}SC_6H_5)_2M(CO)_4$, M = Cr, Mo or W
(xxv) Silver sulphimide trihydrate, $Ag_3(NSO_2)_3.3H_2O$
(xxvi) 1,4,7,10,13,16-Hexaoxacyclooctadecane and its complexes with alkali thiocyanates, NaNCS, KNCS, RbNCS, CsNCS and $Ca(NCS)_2$
(xxvii) Hydrated sodium thiocyanate complex of 1,4,7,10,13,16-hexaoxacyclooctadecane, $C_{12}H_{24}O_6.NaNCS.H_2O$
(xxviii) Potassium thiocyanate complex of 1,4,7,10,13,16-hexaoxacyclooctadecane, $C_{12}H_{24}O_6.KNCS$
(xxix) Rubidium thiocyanate complex of 1,4,7,10,13,16-hexaoxacyclooctadecane, $C_{12}H_{24}O_6.RbNCS$
(xxx) Caesium thiocyanate complex of 1,4,7,10,13,16-hexaoxacyclooctadecane, $C_{12}H_{24}O_6.Cs.NCS$

(xxxi) Calcium thiocyanate complex of 1,4,7,10,13,16-hexaoxacyclooctadecane, $C_{12}H_{24}O_6.Ca(NCS)_2$
(xxxii) Tris(methyldiphenylsilylmethyl)amine, $N(CH_2SiPh_2Me)_3$
(xxxiii) Dimolybdenum tetraacetate, $Mo_2(O_2CCH_3)_4$, a redetermination
(xxxiv) Tris(ethylene-1,2-dioxo)tungsten(VI), $W(OC_2H_4O)_3$
(xxxv) Potassium thioethanoate, KCH_3COS
(xxxvi) μ-Dinitrogen-bis{(π-mesitylene)[1,2-bis(dimethylphosphino)ethane]-molybdenum}, $C_{30}H_{56}Mo_2N_2P_4$
(xxxvii) $Au_{11}Mn_4$
(xxxviii) SrFCl and BaFCl
(xxxix) Tris(ethylthioxanthato)cobalt(III), $Co(C_2H_5SCS_2)_3$, an emendation

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(i) Cyclo-tri-μ-nitrido-dichlorophosphorusbis(oxofluorosulphur), $NPCl_2(NSOF)_2$ at −160 °C
(ii) *trans*-$Fe(CO)_3[P(OCH_3)_3]_2$
(iii) UCl_3, a neutron diffraction study
(iv) Crystallographic studies of selenium(IV) I. Structure of VSe_2O_6
(v) Crystallographic studies of selenium(IV) II. Structure of $ZnSe_2O_5$
(vi) Germanium-hemiporphyrazine, $C_{34}H_{32}N_8O_4Ge$
(vii) Dinitratobis(antipyrine)zinc(II), $Zn(C_{11}ON_2H_{12})_2(NO_3)_2$
(viii) 2,2,6,6-Tetrachloro-4,4,8,8-tetraphenylcyclotetraphosphazatetraene, $N_4P_4Cl_4(C_6H_5)_4$
(ix) Barium dititanate, $BaTi_2O_5$
(x) Bis-(3,5-dimethylpyrazolyl)borane dimer, $C_{20}H_{30}B_2N_8$
(xi) Cobalt vanadate, $Co_2V_2O_7$ and nickel vanadate, $Ni_2V_2O_7$
(xii) Copper—gallium, $\gamma_1 Cu_9Ga_4$
(xiii) Anhydrous copper(II) octanoate, $Cu[CH_3(CH_2)_6COO]_2$
(xiv) Anhydrous copper(II) decanoate, $Cu[CH_3(CH_2)_8COO]_2$
(xv) Copper(II) bis-(*N*,*N*-dimethyldithiocarbamate), $[(CH_3)_2NCS_2]_2Cu$
(xvi) Lanthanum tetraboride, LaB_4

J. Chem. Soc. Chem. Commun., No. 17, 1974
(i) An unusual heteronuclear complex, $RhAg_2(C{\equiv}CC_6F_5)_5(PPh_3)_3$
(ii) Coordinatively unsaturated molybdenum and tungsten cyclopentadienyl complexes; $[WCl(CF_3C_2CF_3)_2(\eta^5\text{-}C_5H_5)]$ and $[Mo(C_4F_6)_2\text{-}(C_5H_5)_2]$

J. Chem. Soc. Chem. Commun., No. 18, 1974
(i) A novel series of planar pentadentate macrocyclic ligands. Structure of 10,11,12,13-tetrahydrodibenzo[*b*,*k*]pyrido[*g*,*f*][1,4,7,10,13]pentaazacyclopentadecin-N^5,N^{10},N^{13},N^{18},N^{19b}-di(perchlorato)manganese
(ii) Tetramethylammonium *cis*-acetylbenzoyltetracarbonylmanganate(I), $(\eta\text{-}C_5H_5)_2W\{CH_2(3,5\text{-}Me_2C_6H_3)\}_2$
(iii) The dimer of bis(tetramethyldiphosphinoethane)ruthenium, $Ru(dmpe)_2C_{10}H_8$

J. Chem. Soc. Chem. Commun., No. 19, 1974

(i) Pentalene complexes from cyclo-octatrienes: crystal structure of η-(1,5-bistrimethylsilyl)pentalene-1,1,2,2,3,3,3,3-octacarbonyl-*triangulo*-triruthenium, $\{Ru_3(CO)_8[C_8H_4(SiMe_3)_2]\}$

(ii) Synthesis of a boron analogue of coordinated cyclobutadiene; structures of *closo*- and *nido*-$L_2PtC_2B_6H_6R_2$, L = PEt_3 or PMe_3, R = H or Me

(iii) 1,4-Addition of hexafluorobut-2-yne to coordinated cyclo-octa-1,5-diene: structures of the adducts [Rh(acetylacetonato)$(C_8H_{12})(C_4F_6)$-$(1\frac{1}{2}\ H_2O)$] and [Ir(acetylacetonato)$(C_8H_{12})(C_4F_6)_2$]

(iv) A sulphur analogue of coronene: crystal structure of benzo[1,2-c; 3,4-c′;5,6-c″]tris[1,2]dithiole-1,4,7-trithione, C_9S_9

J. Chem. Soc. Chem. Commun., No. 20, 1974

(i) Novel cation sited in a dehydrated divalent copper-exchanged zeolite, $Cu_{28}(AlO_2)_{56}(SiO_2)_{136}$

(ii) Structural and chemical aspects of phosphino-ethers as chelating ligands in rhodium(I) cationic complexes. Crystal structures of two phosphino-ether rhodium carbonyl complexes

(iii) Inorgano-Grignard reagents. Reactions and crystal structure of bis-μ-[bis-(η-cyclopentadienyl)hydridomolybdenum]-bis-{di-μ-bromo-[cyclohexylmagnesium(diethyl ether)magnesium]}; a cyclic compound containing covalent $MoMg_2$ systems, [$(\eta$-$C_5H_5)_2MoHMg(C_6H_{11})$-$(\mu$-$Br_2)Mg(Et_2O)$]

(iv) A trinuclear cobalt(III) carboxylate, $[Co_3(quin)_2(PhCO_2)_6]$, quin = quinoline

J. Chem. Soc. Chem. Commun., No. 21, 1974

(i) Product of cobaloxime and DDT, an unexpected vinyl—cobalt(III) complex

(ii) A novel zwitterionic organometallic complex, $[ReCu(C_2C_6F_5)_2(CO)_3$-$(PPh_3)_2]$

(iii) A novel homoporphyrin system, structure of the *endo* nickel complex

(iv) $[(Me_3Si)_2CH]_2SnCr(CO)_5$

(v) The pentacyanocobaltate(II) anion in diethyldiisopropylammonium pentacyanocobaltate, $[NEt_2Pr^i_2]_3[Co(CN)_5]$

(vi) Carbon monoxide complexes of carbonyl-(6,7,13,14-tetramethyl-1,2,4,5,8,9,11,12-octaazacyclotetradeca-2,5,7,12,14-pentaenato)-cobalt(I), $[Co(C_{10}H_{17}N_8)(CO)]$

J. Chem. Soc. Chem. Commun., No. 22, 1974

(i) μ-Carbonyl-bis-μ-hexafluoroisopropylidenamido-hexacarbonyldimanganese, $[Mn_2\{(CF_3)_2C{=}N\}_2(CO)_7]$

(ii) Unusual phosphinoacetylene—iron carbonyl complex: $[Fe_3(CO)_7\{Ph_2PC(CO_2Me)C(CF_3)C_2(CF_3)\}(PPh_2)]\,2C_6H_6$

J. Chem. Soc. Chem. Commun., No. 23, 1974

(i) $[Cr_2(CO)_6(AsMe)_9]$ and $[Mo_2(CO)_6(AsPr^n)_8]$: the stabilization of an As_9 ring and an As_8 chain by coordination

(ii) Benzyltriphenylmethylthiomercury, $PhCH_2HgSCPh_3$

(iii) A novel quadridentate complex, racemic (*ΛSR* + *ΛRS*)-[(1,8-diamino-2,5-dimethyl-5-hydroxy-3,6-diaza-oct-2-ene)(1,2-diaminoethane)-cobalt(III)]hexachlorothallate(III), $2H_2O$

(iv) (1-5-η-Fluorenyl)(1-3-η-fluorenyl)dichlorozirconium(IV), $(fl)_2ZrCl_2$

(v) A novel bidentate polypyrazolylborate complex: carbonyl[hydrido-tris(pyrazol-1-yl)borato]methylplatinum, $C_{11}H_{13}BN_6OPt$

(vi) Bis(η-cyclopentadienyl)bis-(*N*-cyanato)titanium(IV), $[Ti(\eta\text{-}C_5H_5)_2(NCO)_2]$

(vii) [*cis*-1-Bistrifluoromethylphosphino-2-(diphenylphosphino)ethane]-dichloropalladium(II), *cis*-$[PdCl_2\{Ph_2PCH_2CH_2P(CF_3)_2\}]$

(viii) A trinuclear cobalt(III) carboxylate, $[Co_3(quin)_2(PhCO_2)_6]$, quin = quinoline

Trichlorotantalum(V)-bis-(2-methoxy)ethanol, $TaCl_3[C_3H_7{-}N{-}C(Me){-}N{-}C_3H_7]_2$

($P2_1/a$) R = 10% for 878 independent reflections. The structure is a seven coordinate monomer with pentagonal bipyramidal geometry, the two bidentate ligands and one chlorine atom occupying the equatorial plane. Ta—Cl = 2.355(16), 2.367(17), 2.374(13), Ta—O = 1.80(3), 1.91(3), 2.23(3) Å.

M.G.B. Drew and J.D. Wilkins, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 549.

Dinitrosyl di(phenyl-dimethoxyphosphine)manganese(I) chloride, $[Mn(NO)_2(P(OMe)_2Ph)_2Cl]$; three independent determinations. Two forms of crystals were obtained

A. ($P\bar{1}$) Z = 4, R = 6.5% for 3711 observed reflections. B. ($C2/c$) Z = 8, R = 5.8% for 2265 observed reflections. In each case, the four atoms of the $[MnP_2Cl]$ moiety are coplanar within 0.05 Å. The oxygen atoms of the NO groups lie within 0.05 Å of the plane defined by the $[MnN_2Cl]$ system and the NO groups are bent in towards each other. Mn—N—O = 165 ± 1°, Mn—N = 1.62—1.64, N—O, 1.19—1.20 Å.

M. Laing, R. Reimann and E. Singleton, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 557.

The cage molecule tetrathiophosphorus hexa-*N*-methylimide, $P_4S_4(NCH_3)_6$ ($P\bar{4}2_1c$) Z = 2, R = 10.9% for 520 unique reflections. The molecular structure consists of a central cage formed by a tetrahedron of phosphorus atoms linked by six NCH_3 groups. P—S = 1.93(2), P—N(ave) = 1.66(3) Å, N—P—N(ave) = 103(2)°.

G.W. Hunt and A.W. Cordes, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 637.

The absolute configuration of $(+)_{589}$-tris(1,4-diaminobutane)cobalt(III) ion, $(+)_{589}$-$[Co(tmd)_3]^{3+}$

($P321$) $Z = 3$, $R = 4.7\%$ for 2133 independent reflections. The absolute configuration is Δ. Co—N(ave) = 2.000 Å, N—Co—N(ave) = 89.8°
S. Sato, Y. Saito, J. Fujita and H. Ogino, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 669.

Iodo-1,10-phenanthroline-thiourea copper(I), [Cu(tu)(*o*-phen)I]
($P2_1/c$) $Z = 4$, $R = 14.8\%$ for 486 film intensity data. The coordination about copper(I) is essentially tetrahedral. Cu—I = 2.65(1), Cu—S = 2.28(2), Cu—N = 2.03(5), 2.08(7) Å.
A.K. Jain and A.J. Smith, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 707.

o-Nitrosophenol complexes which are analogues of ferroverdin $K[Ni(qo)_3]$, $(CH_3)_2CO$, (qoH = 4-vinylphenyl ester of 4-hydroxy-3-nitrosobenzoic acid)
($P2_1/c$) $Z = 4$, $R = 11\%$ for 1575 independent reflections. The complex anion has three nitrosophenol ligands bonded to the nickel(II) atom in a distorted octahedral arrangement with *cis* conformation, that is all the NO groups adjacent. Ni—N(mean) = 2.009, Ni—O = 2.062, K—O = 2.72—2.88 Å.
P.W. Carreck, J. Charalambous, M.J. Kensett, M. McPartlin and R. Sims, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 749.

X-ray powder diffraction study of poly(carbon monofluoride), $CF_{1.12}$
V.K. Mahajan, R.B. Badachhape and J.L. Margrave, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 1103.

Bis(silver trifluoroacetate) benzene, $\{[AgC_2F_3O_2]_2.C_6H_6\}$
($A2/m$) $Z = 9$, $R = 8.7\%$ for 2824 observed reflections. The structure may be described as two parallel chains of silver atoms with alternating short and long distances, of ~ 2.9 and 6.8 Å respectively. Ag—Ag (between chains) = 3.640(3) Å.
G.W. Hunt, T.C. Lee and E.L. Amma, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 909.

Red(A) and blue(B) modifications of dihydrate (*N*-benzyl-L-valinato)-(*N*-benzyl-D-valinato)copper(II)
A. ($P2_1/c$) $Z = 2$, $R = 11.4\%$ for 1200 reflections. B. ($P1$) $Z = 1$, $R = 16.0\%$ for 1000 reflections. The coordination geometry of the amino acids about Cu^{II} is square-planar, with the *trans* arrangement of substituents at the nitrogen and α-carbon atoms $<N—Cu—O \cong 85°$, Cu—N = 1.997, 2.03, Cu—O = 1.933, 1.94 Å.
G.G. Aleksandrov, Yu.T. Struchkov, A.A. Kurganov, S.V. Rogozhin and V.A. Davankov, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 959.

A complex of nona-coordinate U^{IV}, $(Ph_4As)_2[U(\text{pyridine-2,6-dicarboxylato})_3].3H_2O$
($P2_1/c$, C_{2h}^5) $Z = 4$, $R = 11.5\%$ based on 8541 reflections. The uranium

atom is coordinated by three dipicolinato ions acting as terdentates. The coordination polyhedron has approximate D_{3h} symmetry and can be described as a distorted tricapped trigonal prism. U—O = 2.37—2.42, U—N = 2.51—2.53 Å.

L. Baracco, G. Bombieri, S. Degetto, E. Forsellini, R. Graziani and G. Marangoni, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 1045.

$M_2Mn_4(CO)_{18}$ (M = In, Ga) and $M_2Mn_4(CO)_{18}.2D$ (M = In, D = pyridine, acetone; M = Ga, D = pyridine)

All $M_2Mn_4(CO)_{18}$ compounds are isomorphous, space group $I4_1/a$. They contain a planar bridged ring of 2M and 2Mn atoms, in which two $Mn(CO)_4$ groups form the Mn—Mn bond, each being connected with $2[\mu\text{-}MMn(CO)_5]$ units; the $Mn(CO)_5$ ligands at M have *trans* positions with respect to the planar metal ring. The new clusters coordinate donor molecules such as pyridine and acetone at M(C.N. = 3) to form complexes $M_2Mn_4(CO)_{18}.2D$, M(C.N. = 4)

H.-J. Haupt and F. Neumann, *J. Organometal. Chem.*, 74 (1974) 185.

The molecular adduct of dimethyltin(IV) chloride with *N,N'*-ethylenebis-(salicylideneiminato)nickel(II), $[Me_2SnCl_2.Ni(salen)]$

($P2_1/c$) $Z = 4$, $R = 7.5\%$ using 2807 independent reflections. *N,N'*-ethylenebis(salicylideneiminato)nickel(II) behaves as a neutral bidentate ligand through its oxygen atoms forming a binuclear complex. The tin atom has distorted octahedral geometry with *trans*-methyl groups. Sn—C = 2.12(2), 2.13(2) Å. The nickel environment is similar to that found in Ni(salen).

M. Calligaris, L. Randaccio, R. Barbieri and L. Pellerito, *J. Organometal. Chem.*, 76 (1974) C56.

Syn- and *anti*-substituted (1,3-butadiene)iron tricarbonyl compounds. A. [2-{(*m*-nitrophenyl)amino}-*trans,trans*-3,5-heptadiene]iron tricarbonyl, $FeC_{16}H_{16}N_2O_5$. B. [2-{(phenyl)amino}-*cis,trans*-3,5-heptadiene]iron tricarbonyl, $FeC_{16}H_{17}NO_3$

A. ($P\bar{1}$) $Z = 2$, $R = 2.6\%$ for 2472 independent reflections. B. ($P2_1/c$) $Z = 4$, $R = 2.4\%$ for 1998 independent reflections. The structures of A and B, having substituents in *syn* and *anti* positions provide a reliable model for the parent compound π-butadiene-$Fe(CO)_3$ (a ligand at R.T.). *Anti*-H atoms deviate by 30° from the diene plane away from the metal, and *syn*-H atoms deviate by 20° toward the metal.

A. Immirzi, *J. Organometal. Chem.*, 76 (1974) 65.

A tricarbonyliron derivative of barbaralone, (2,3,4,8-η^4-bicyclo[3.2.2]nona-3,6-diene-2,8-yl-9-one)tricarbonyliron

($P\bar{1}$) $Z = 2$, $R = 2.1\%$ for 1283 reflections. The coordination about iron is distorted octahedral if η^3-allyl is considered bidentate. The 3 CO ligands are mutually *cis*. The allyl group is *trans* to two CO groups, the Fe—C

σ bond is *trans* to the third. Fe—CO(mean) = 1.795(5), Fe—C(allyl) = 2.069(2), 2.137(3), 2.195(3), Fe—C(σ) = 2.110(2) Å.
F.A. Cotton and J.M. Troup, *J. Organometal. Chem.*, 76 (1974) 81.

Bis(tricarbonyliron) derivative of 3,3'-bis(bicyclo[4.2.0]octa-2,4-diene)
($P\bar{1}$) $Z = 1$, $R = 3.0\%$ for 1788 reflections. The structure consists of two bicyclo[4.2.0]octa-2,4-dienetricarbonyliron moieties related by an inversion centre. The molecule shows some distortion of the usual hexa-1,3-diene-$Fe(CO)_3$ bond distances and angles, probably due to the C—C bond between the two halves of the molecule at the second carbon in the 1,3-diene group.
F.A. Cotton and J.M. Troup, *J. Organometal. Chem.*, 77 (1974) 83.

The Grignard reagent, dimeric ethylmagnesium bromide, diisopropyl ether, $[C_2H_5MgBr.O(i\text{-}C_3H_7)_2]_2$
($P2_1/n$) $Z = 2$, $R = 7.3\%$ for 1473 non-zero reflections. The magnesium is four coordinate; dimers are formed through bridging bromide atoms. Mg—Br(mean) = 2.58 Å. There is only one solvent molecule bound to each magnesium atom.
A.L. Spek, P. Voorbergen, G. Schat, C. Blomberg and F. Bickelhaupt, *J. Organometal. Chem.*, 77 (1974) 147.

$[(\pi\text{-}C_6H_{11})Ni(PPh_3)_2]^+ZnCl_3^-$, a structure containing a 1,1',2-trimethylallyl ligand
(*Pbca*) $Z = 8$, $R = 10.3\%$ for 2321 non-zero reflections. The structure consists of $[C_6H_{11}Ni(PPh_3)_2]^+$ cations and $Zn_2Cl_6^{2-}$ dimeric anions. The coordination about Ni is square planar if one considers the trimethylallyl ligand as bidentate. Ni—P = 2.220(5), 2.235(4), Ni—C = 2.04(2), 1.98(3), 2.13(3) Å.
M. Zocchi and A. Albinati, *J. Organometal. Chem.*, 77 (1974) C40.

cis-Dichloro{bis-1-*anti*,3-*syn*-(*o*-diphenylphosphino)phenyl-1-methallyl-(1-3η)}rhodium(III), $RhCl_2[o\text{-}Ph_2PC_6H_4CH{=}CH{-}C(CH_3)C_6H_4PPh_2\text{-}o]$
(*Cc*) $Z = 4$, $R = 2.3\%$ for 3811 reflections. There is essentially octahedral geometry about Rh^{III}. Important features of the coordinated π-allylphosphine ligand are discussed.
M.A. Bennett, R.N. Johnson, G.B. Robertson, I.B. Tomkins and P.O. Whimp, *J. Organometal. Chem.*, 77 (1974) C43.

Tricyclo[6.2.0.$0^{2,7}$]deca-3,5-dienetricarbonyliron, $(C_{10}H_{12})Fe(CO)_3$
($P2_12_12_1$) $Z = 4$, $R = 3.3\%$ from 1359 reflections. The molecular structure consists of an $Fe(CO)_3$ moiety bonded to the diene portion of a cyclohexadiene ring. This six-membered ring is fused to a second four-membered ring. Both four-membered rings are essentially planar.
F.A. Cotton and J.M. Troup, *J. Organometal. Chem.*, 77 (1974) 369.

Two complexes with σ and π nickel—carbon bonds: A. (π-pentenyl)(diisopropylphenylphosphine)methylnickel(II), B. (π-pentenyl)(dimenthylmethylphosphine)methylnickel(II)

A. ($P2_1/c$) $Z = 4$, $R = 6.26\%$ from 1404 observed reflections. B. ($P2_12_12_1$) $Z = 4$, $R = 3.65\%$ from 2689 observed reflections. The bonding situation of the π-allylic fragments as well as the steric conformation of the phosphine ligands is discussed. Ni—C(σ) in A = 1.99(1), in B = 1.975(4) Å. The absolute configuration of B has been determined to be *R*.

B.L. Barnett and C. Krüger, *J. Organometal. Chem.*, 77 (1974) 407.

Tetramethylethylenenickel 1,2-bis(dicyclohexylphosphino)ethane, $(CH_3)_2C_2(CH_3)_2Ni[(C_6H_{11})_2PCH_2CH_2P(C_6H_{11})_2]$

($P2_1/n - C_{2h}^5$) $Z = 4$, $R = 3.6\%$ for 4995 observed reflections. If one coordination site is assigned to the olefin, the nickel atom is three coordinate. Ni—C(avg) = 1.981(2), Ni—P = 2.156(6) Å. The tetramethylethylene moiety is distorted from planarity with the $C(CH_3)_2$ planes bent away from the Ni atom by 27.2(6)°.

D.J. Brauer and C. Krüger, *J. Organometal. Chem.*, 77 (1974) 423.

A complex of tetramethylcyclobutadiene and aluminum chloride, containing a σ-Al—C bond, $[C—CH_3]_4^+AlCl_3^-$

(*Pnma*) $Z = 4$, $R = 5.4\%$ from 1148 reflections. The aluminum chloride is coordinated via a σ-aluminum carbon bond (1.979 Å) to a non-planar tetramethylcyclobutadiene moiety. The bond length pattern indicates a charge dislocation within the four-membered ring.

C. Krüger, P.J. Roberts, Y.-H. Tsay and J.B. Koster, *J. Organometal. Chem.*, 78 (1974) 69.

The chemistry and the stereochemistry of poly(*N*-alkyliminoalanes)=(PIA). I

The structural features of poly(*N*-alkyliminoalanes) have been investigated for different amines. In general all the results agree with the formation of oligomers of —H—Al—NR— units, with three dimensional structures made up by four- and six-membered rings. A cage structure has been found for $(H—Al—N=iso—C_3H_7)_6$.

S. Cucinella, T. Salvatori, C. Busetto, G. Perego and A. Mazzei, *J. Organometal. Chem.*, 78 (1974) 185.

$(HAlN—i\text{-}Pr)_6$

($P\bar{1}$) $Z = 4$, $R = 5.0\%$ using 6491 independent reflections. The molecule consists of a hexagonal cage formed by two almost planar six-membered rings, $(AlN)_3$, joined together by six transverse Al—N bonds. Al—N = 1.898(2) in six-membered ring, = 1.956(2) in transverse bonds, Al—H = 1.49(1), N—C = 1.514(2) Å.

M. Cesari, G. Perego, G. Del Piero, S. Cucinella and E. Cernia, *J. Organometal Chem.*, 78 (1974) 203.

4-*tert*-Butyl-π-(tricarbonylchromium)benzoic acid, $CrC_{14}O_5H_{14}$
($P2_1/c$) $Z = 4$, $R = 4.8\%$ for 1717 independent reflections. The compound adopts a conformation which departs from perfect staggering by about 7°.
F. Van Meurs and H. Van Koningsveld, *J. Organometal. Chem.*, 78 (1974) 229.

The twinned and disordered crystal structure of tetrahedral tri-μ-carbonyl-enneacarbonyldicobaltdiiridium, $Co_2Ir_2(CO)_{12}$
($P2_1/c$) $Z = 4$, $R = 5.1\%$ for 570 composite reflections. The molecular structure of $Co_2Ir_2(CO)_{12}$ is very similar to that of $Co_4(CO)_{12}$ and $Rh_4(CO)_{12}$. Of the CO ligands, 9 are bonded linearly and 3 are involved in edge bridging about a tetrahedron of metal atoms. Co and Ir are partially disordered, Ir has a pronounced preference for the apical position to which only terminal ligands are coordinated.
V.G. Albano, G. Ciani and S. Martinengo, *J. Organometal. Chem.*, 78 (1974) 265.

Ring closure of cyclooctatetraenetricarbonyliron complexes; structure of a bicyclo[4.2.0]-2,4,7-octatrienetricarbonyliron compound, $C_8H_6(SiMe_3)$-$(CPh_3)Fe(CO)_3$
($P\bar{1}$) $Z = 2$, $R = 5.2\%$ for 3620 reflections. The six-membered ring is folded into 2 planar segments, with 4 carbon atoms approximately 2.085 Å from the iron atom. The bond lengths (C—C = 1.414) indicate extensive delocalization in the diene moiety. Complete structure is discussed.
M. Cooke, J.A.K. Howard, C.R. Russ, F.G.A. Stone and P. Woodward, *J. Organometal. Chem.*, 78 (1974) C43.

Trichlorogermylcobalt tetracarbonyl, $Cl_3GeCo(CO)_4$
(Cc) $Z = 8$, $R = 9.8\%$ for 1647 observed reflections. The coordination of the Co atoms is trigonal bipyramidal, and that of the Ge atoms is tetrahedral. The equatorial carbonyl groups are in a staggered conformation with respect to the chlorine atoms. The corresponding carbonyl—cobalt—germane angles are less than 90°. Ge—Co(ave.) = 2.31 Å.
G.C. v.d. Berg, A. Oskam and K. Olie, *J. Organometal. Chem.*, 80 (1974) 363.

sym-trans-Di-μ-acetobis[*o*-(*t*-butyl-*o*-tolylphosphino)benzyl]dipalladium(II), $[Pd(OAc)\{CH_2C_6H_4P$-*t*-$Bu(o$-tolyl$)\}]_2$
($P\bar{1}$) $Z = 2$, $R = 4.4\%$ based on 5436 observed reflections. The structure consists of independent dimeric molecules packed with cyclohexane molecules of crystallization. [*o*-(*t*-Butyl-*o*-tolylphosphino)benzyl]palladium(II) fragments are bridged by the two acetate groups so that each palladium has a slightly distorted planar coordination; the molecule has approx. C_2 symmetry. Pd···Pd = 3.413(1), Pd—P (mean) = 2.204(2), Pd—C (mean) = 2.033(5), Pd—O (mean) = 2.13(1) Å.
G.J. Gainsford and R. Mason, *J. Organometal. Chem.*, 80 (1974) 395.

Structural studies of $(\pi\text{-}C_5H_5)_2MX_2$ complexes and their derivatives; structure of 1,1′-trimethylenedicyclopentadienyldichlorozirconium, $(CH_2)_3(C_5H_4)_2ZrCl_2$

(*Pbca*) $Z = 8$, $R = 2.64\%$ based on 1049 independent reflections. The coordination about the zirconium atom is that of a distorted tetrahedron comprised of the chlorine atoms and the centroids of the cp rings. Cl—Zr—Cl = 96.92(7)°, centroid—Zr—centroid = 129.5°. Zr—Cl = 2.451(2), 2.431(2), Zr—C (range) = 2.477—2.516 Å.

C.H. Saldarriaga-Molina, A. Clearfield and I. Bernal, *J. Organometal. Chem.*, 80 (1974) 79.

Unexpected behaviour of alkoxycarbonyl ligands; structure of *trans*-bis(ethoxycarbonyl)bis(triphenylphosphine)platinum, $C_{42}H_{40}PtO_4P_2$

($P\bar{1}$) $Z = 1$, $R = 3.8\%$ based on 3435 independent reflections. The structure consists of discrete centrosymmetric molecules, the Pt atom lies on an inversion centre. Pt—P = 2.302(2), Pt—C = 2.059(7). The Pt—COO moieties are essentially planar, making an angle of 80.8° with the coordination plane. A *trans* configuration for a complex containing two Pt^{II}—C bonds is unexpected, arising from the presence of alkoxycarbonyl ligands.

P.L. Bellon, M. Manassero, F. Porta and M. Sansoni, *J. Organometal. Chem.*, 80 (1974) 139.

The dimer of heptafulveneiron tricarbonyl, $C_{22}H_{14}Fe_2O_6$

(*Pbca*) $Z = 8$, $R = 7.0\%$ based on 771 independent reflections. The structure and NMR data indicate the complex to be 3,3′-ethano-1,1′-bis(cyclohepta-2,4,6-trienyl)diiron hexacarbonyl, with diene fragments of the tricyclohexadecahexaene ligand bound to iron tricarbonyl moieties. Intramolecular bonding together with mechanism of formation is discussed.

A. Eisenstadt, J.M. Guss and R. Mason, *J. Organometal. Chem.*, 80 (1974) 245.

Coordination complexes of bis(2,2-dimethyl-3,5-hexanedionato)zinc with organozinc—oxygen and —nitrogen compounds. Structure of phenyl—zinc phenoxide complex, $[PhZnOPh.Zn(pac)_2]_2$

($P2_1/c$) $Z = 2$, $R = 12.9\%$ based on 2310 observed reflections. The structure contains an eight-membered Zn_4O_4 ring formed by alternating zinc and oxygen atoms. This ring is bridged twice by the oxygen atoms of the two PhZnOPh moieties. The dimeric structure contains four- and six-coordinate zinc atoms and two-, three- and four-coordinate oxygen atoms.

J. Boersma, A.L. Spek and J.G. Noltes, *J. Organometal. Chem.*, 81 (1974) 7.

The secondary carbene complex, $RuI_2[CHN(CH_3)(p\text{-}CH_3C_6H_4)](CO)(CN\text{-}p\text{-}CH_3C_6H_4)(PPh_3)$

($P\bar{1}$) $Z = 2$, $R = 5.5\%$ for 2731 observed reflections. The structure shows the iodine atoms mutually *cis*, and the carbene and isocyanide ligands *trans* to one another. The coordination octahedron is not regular, with the carbene

carbon being most displaced from its ideal position. A noticeably short C—N bond, 1.28 Å, is found. Internal comparisons of Ru—C(carbene), Ru—C(isocyanide) and Ru—C(CO) bond lengths are made.

D.F. Christian, G.R. Clark and W.R. Roper, *J. Organometal. Chem.*, 81 (1974) C7.

Rhodacyclopentane derivatives, A. (acac)Rh(C_6H_8)($py)_2$ and B. $(acac)_2$-$Rh_2(C_6H_8)(PPh_3)$

A. ($P\bar{1}$) $Z = 2$, $R = 3.3\%$ for 3373 independent reflections. B. ($P\bar{1}$) $Z = 2$, $R = 4.1\%$ for 4473 observed reflections. A has octahedral geometry with acac and C_6H_8 bonded as chelate ligands and the two pyridine ligands mutually *cis*. B displays a two-centre geometry with the Rh atoms in different environments. One Rh has a tetragonal pyramidal geometry, the other has octahedral. In this complex the unsaturated hydrocarbon part is also π-bonded to another Rh atom, and the one acac group acts as a five-electron donor.

A. Immirzi, *J. Organometal. Chem.*, 81 (1974) 217.

Tris(triphenylphosphine)gold(I)[dodecahydrido-6-thia-nido-decaborate-(1-)], $[(C_6H_5)_3P]_3AuB_9H_{12}S$

($P\bar{1}$) $Z = 2$, $R = 7.7\%$ based on 3094 observed reflections. The compound is a salt consisting of $[(C_6H_5)_3P]_3Au^+$ cations and $B_9H_{12}S^-$ anions. The cation is trigonal and nearly planar, Au—P = 2.382(5) Å, P—Au—P = 119.3(36)°. The $B_9H_{12}S^-$ thiaborane anion is an open icosahedral fragment with the S atom in the 6 position, on the periphery of a decaborane polyhedron.

L.J. Guggenberger, *J. Organometal. Chem.*, 81 (1974) 271.

μ-*N,N'-o*-Phenylenebis(salicylideneiminato)bis(2-*C,N*-acetophenoneoxime-palladium(II)), $C_{36}N_4O_4H_{30}Pd_2$, $(ApoPd)_2$ salophen

($P2_1/c$) $Z = 4$, $R = 6.9\%$ based on 3122 observed reflections. Each palladium atom in the molecule possesses approximate square planar geometry being coordinated by N and C of the Apo ligand and N and O of a Schiff base moiety. Pd···Pd = 3.696(4) Å (intramolecular). Bridging by the salophen ligand is achieved by twisting the salicylidene—Pd—Apo moieites out of the plane of the phenylene bridge.

G.D. Fallon, B.M. Gatehouse, B.E. Reichert and B.O. West, *J. Organometal. Chem.*, 81 (1974) C28.

S,S'-Diethyl dithiooxalate, $(CO—SC_2H_5)_2$ at —60°C

($P2_1/c$) $Z = 2$, $R = 5.8\%$ based on 3000 reflections. C=O = 1.209, C—S = 1.749, C—C = 1.533 Å.

G. Kiel, M. Dräger and U. Reuter, *Chem. Ber.*, 107 (1974) 1483.

(2,2-Diethoxyvinylidene)triphenylphosphorane

($P2_1/c$) $Z = 4$, $R = 7.9\%$ based on 2078 reflections. Each phenyl group is

rotated in the same sense around the P—C bond to give a "paddle wheel" arrangement. The diethoxyvinylidene group does not deviate significantly from planarity and adopts a staggered position.

H. Burzlaff, U. Voll and H.-J. Bestmann, *Chem. Ber.*, 107 (1974) 1949.

On chalcogenolates LXIX. Structure of 3,4,5-tris(methylthio)-1,2-dithiolium iodide, $[(H_3CS)_3C_3S]^+I^-$

($P2_12_12_1$-D_2^4) $Z = 4$, $R = 4.9\%$ based on 844 reflections. The existence of charge transfer relations between ring-sulfur and iodine is verified. I—S = 3.442 and 3.394 Å.

G. Kiel, U. Reuter and G. Gattow, *Chem. Ber.*, 107 (1974) 2569.

On heterocyclic systems containing arsenic I. 2-Chloro-1,3,6,2-trithiarsaoctane, $C_4H_8AsClS_3$

($C2/c$) $Z = 8$, $R = 3.7\%$ based on 5000 reflections. The eight-membered ring has a deformed boat conformation with 1,5-transannular As—S-interaction. The coordination of the As atom is ψ-trigonal-bipyramidal with axial distances As—Cl = 2.36, As—S = 2.72 and equatorial As—S = 2.25, 2.26 Å. This is a model compound for the inhibition of enzyme systems by As^{III}.

M. Dräger, *Chem. Ber.*, 107 (1974) 2601.

A covalent metal—metal bond I. A. Octacarbonyl-bis[μ-pentacarbonylmanganesegallium(III)]-dimanganese and B. octacarbonyl-bis[μ-pentacarbonylmanganeseindium(III)]-dimanganese, $Mn_2(CO)_8\{\mu\text{-}MMn(CO)_5\}_2$ (M = Ga, In)

A. ($I4_1/a$) $Z = 8$, $R = 3.2\%$ based on 2392 reflections. B. ($I4_1/a$) $Z = 8$, $R = 4.0\%$ based on 3172 reflections. Both structures are similar; they contain a planar M_2Mn_2 ring in which the Mn—Mn distance [Mn—Mn(Ga) = 3.052(1), Mn—Mn(In) = 3.227(1) Å], and the acute angles at the bridging Ga(76.86(2)°) or In(76.36(2)°) are consistent with the existence of a Mn—Mn bond. Mean values for other distances are: Ga—Mn = 2.541(1), In—Mn = 2.605(1), Mn—C = 1.842(5), 1.804(5) Å.

H. Preut and H.-J. Haupt, *Chem. Ber.*, 107 (1974) 2860.

On heterocyclic systems containing phosphorus I. *trans*-1,6-Diphenyl-1λ^5,6λ^5-diphosphacyclodecane-1,6-dione dihydrate

($P2_1/c$) $Z = 2$, $R = 3.9\%$ based on 2726 reflections. The ten-membered ring has the normal cyclodecane conformation with the lowest strain energy. The geminal phenyl and oxo groups are both extraannular. C—P(ave) = 1.804(3), P=O = 1.502(3) Å. The structure consists of two molecules connected by P=O···HOH···O=P hydrogen bonds.

M. Dräger, *Chem. Ber.*, 107 (1974) 3246.

Structures of the pyridine-*N*-oxide complexes, A. ZnI_2L_2, B. CdI_2L and C. $HgCl_2L$ (L = C_5H_5NO)

A. ($P2_1/c$) $Z = 4$, $R = 21\%$ based on 538 reflections. B. ($P2_1/a$) $Z = 4$, $R = 9\%$ based on 569 reflections. C. ($P\bar{1}$) $Z = 2$, $R = 11.8\%$ based on 782 reflections. A consists of discrete monomeric complexes with Zn atoms tetrahedrally bonded to two I atoms (Zn—I = 2.56, 2.58 Å) and two O atoms (Zn—O = 1.98, 1.99 Å). B is composed of infinite chains with units of [CdI] alternately bridged through two I atoms and two O atoms. The coordination geometry around the penta-coordinated Cd atom is a trigonal bipyramid, Cd—I = 2.724, 2.834, 2.963, Cd—O = 2.275, 2.383 Å. C contains bent molecules $HgCl_2$ (Hg—Cl = 2.316, 2.339 Å, $\beta = 163°$) held together in a two-dimensional layer type network by markedly weaker mutual interactions and by C_5H_5NO molecules (Hg—Cl = 3.185, 3.318, Hg—O = 2.59, 2.60 Å).

G. Sawitzki and H.G. von Schnering, *Chem. Ber.*, 107 (1974) 3266.

$SbCl_3.S_2C_4H_8$

($P2_12_12_1$ No. 19) $Z = 4$, $R = 6\%$ based on 2426 reflections. The structure is built up by molecules of $SbCl_3$ and $S_2C_4H_8$. Sb—Cl = 2.386—2.441 Å.

G. Kiel and R. Engler, *Chem. Ber.*, 107 (1974) 3444.

4,4-Dimethyl-2,2-diphenyl-1,3-dioxa-4-azonia-2-boranatacyclopentane

($Pna2_1$) $Z = 4$, $R = 7.1\%$ for 1100 reflections. Bond angles in the five-membered ring, which has a distorted half-chair conformation, range from 101.5(4) for OBO to 107.1(4) for NOB. The structure consists of discrete molecules separated by normal van der Waals' distances. Mean B—C = 1.632(8), B—O = 1.506(7), 1.556(8), N—O = 1.409(5), C—O = 1.378(9), C—N = 1.467—1.509(7—10) Å.

S.J. Rettig, J. Trotter and W. Kliegel, *Can. J. Chem.*, 52 (1974) 2531.

Lead metavanadate, PbV_2O_6

(*Pnma*) $Z = 4$, $R = 5.2\%$ for 816 reflections. The two unique vanadium ions lie on mirror planes and are each coordinated to five oxygen atoms, V—O = 1.61—2.06 Å. The distorted octahedra are completed by a sixth V—O interaction, V—O = 2.73, 2.57 Å. The Pb ion is coordinated to nine oxygen atoms lying in a spherical shell, Pb—O = 2.56—2.90 Å.

B.D. Jordan and C. Calvo, *Can. J. Chem.*, 52 (1974) 2701.

cis-Dithiocyanatobis(1,10-*o*-phenanthroline)mercury(II), *cis*-$Hg(SCN)_2(phen)_2$

($P\bar{1}$) $Z = 2$, $R = 6.9\%$ for 1718 reflections. The structure contains discrete molecules, in which Hg is coordinated to four nitrogen atoms from 2 phenanthroline molecules and to two sulfur atoms from SCN groups. Hg—N = 2.42(2)—2.52(2), Hg—S = 2.622(8), 2.582(8) Å. The terminal nitrogen atoms of the SCN groups are uncoordinated.

A.L. Beauchamp, B. Saperas and R. Rivest, *Can. J. Chem.*, 52 (1974) 2923.

Diazido-2,2'-bipyridinecopper(II), $C_{10}H_8N_2(N_3)_2Cu$

($P\bar{1}$) $Z = 2$, $R = 6.7\%$ based on 1396 reflections. The copper coordination is square planar with two additional longer bonds. Cu—N(bipy) = 2.016(6), 2.019(6), Cu—N(N_3) = 1.949(6), 1.966(6). The longer bonds are Cu—N = 2.680(8), 2.682(8) Å. Each azide takes part in asymmetric bridging through a single nitrogen atom.

G.W. Bushnell and M.A. Khan, *Can. J. Chem.*, 52 (1974) 3125.

[2,3-Bis(dimethylarsino)-1,1,1,4,4,4-hexafluorobut-2-ene-As,As] tricarbonyldiiodotungsten(II), $Me_2AsC(CF_3):C(CF_3)AsMe_2WI_2(CO)_3$

($P2_1/c$) $Z = 4$, $R = 6.9\%$ for 1823 observed reflections. The tungsten atom is seven-coordinate with a distorted capped octahedral environment, the capping group being a CO group W—C = 1.94(3). The capped face consists of the two remaining CO groups (W—C = 1.97(4), 2.00(4)) and one As, W—As = 2.556(3) Å. The uncapped face contains the two iodine atoms, W—I = 2.848(2), 2.856(2) and the remaining As from the bidentate ligand, W—As = 2.618(3). The *trans* effect is discussed.

A. Mercer and J. Trotter, *Can. J. Chem.*, 52 (1974) 3331.

$TlVO_3$

(*Pbcm*) $Z = 4$, $R = 5.5\%$ for 255 independent reflections. Vanadium has tetrahedral coordination; the tetrahedra are joined by having two oxygens in common to form chains $(VO_3)_n^{n-}$ parallel to the *c* axis.

M. Ganne, Y. Piffard and M. Tournoux, *Can. J. Chem.*, 52 (1974) 3539.

The nickel complex of the Schiff base bis(trifluoroacetylacetone)triethylenetetraamine, H_2L, $NiC_{16}F_6H_{22}N_4O_2(NiL)$

($P2_1/c$) $Z = 4$, $R = 5.7\%$ for 1686 independent reflections. The structure confirms that condensation occurs through the acetyl group, not through the trifluoroacetyl group. The Ni is octahedrally coordinated by the four N atoms and the two O atoms of L^{2-}. Distortions in the octahedral geometry arise from three contiguous five-membered rings, N—Ni—N(ave) = 81.1°. Average distances are Ni—O = 2.037, Ni—N(trigonal) = 2.044 and Ni—N(tetrahedral) = 2.128 Å.

M.F. Richardson, *Can. J. Chem.*, 52 (1974) 3716.

Dichloro(acetylacetonato)-2,2'-bipyridylindium(III), $Cl_2InC_{15}O_2N_2H_{15}$, $Cl_2In(acac)(bipy)$

($P2_1/c$) $Z = 4$, $R = 4.3\%$ for 3524 observed reflections. Indium is six-coordinate, surrounded by *cis* chlorine atoms, In—Cl = 2.443(1), 2.394(1), a bidentate bipyridyl group, In—N = 2.276(4), 2.299(4), and a bidentate acac group, In—O = 2.124(3) and 2.164(3) Å.

J.G. Contreras, F.W.B. Einstein and D.G. Tuck, *Can. J. Chem.*, 52 (1974) 3793.

Stereochemistry of polynuclear cadmium(II)thioglycolates: structure of cadmium(II)bisthioglycolate, $Cd(SCH_2CH_2OH)_2$

($P2_1/c$) $Z = 4$, $R = 4.1\%$ using 4265 reflections. The structure shows that Cd ions and mercaptide sulfur atoms form a two-dimensional array of six-membered and four-membered rings. All sulfur atoms bridge two cadmium ions. Of the two kinds of cadmium ions, one shows distorted tetrahedral coordination to four sulfurs, the other one distorted trigonal bipyramidal coordination to four S atoms and to an axial alcohol oxygen atom.

$Cd_{(1)}$—S = 2.526—2.584, Cd_2—$S_{(eq)}$ = 2.517—2.556, Cd_2—$S_{(ax)}$ = 2.692 Å.

H.-B. Bürgi, *Helv. Chim. Acta*, 57 (1974) 513.

Three palladium(II) complexes with the terdentate chelating amine, *cis*-3,5-diaminopiperidine (DAPI), A. $[Pd(HDAPI)_2](ClO_4)_4.2H_2O$, B. $[Pd(DAPI)_2](ClO_4)_2$, C. $[Pd(DAPI)(HDAPI)](NO_3)_3$

A. ($Pbca$) $Z = 4$, $R = 4.6\%$ for 2259 reflections, B. ($P2_1$) $Z = 2$, $R = 5.2\%$ for 3002 reflections, C. ($P2_1/c$) $Z = 2$, $R = 10\%$ for 1314 reflections. The structures of all three show that Pd has a square planar coordination. The DAPI groups are *trans* in the protonated and mixed complexes. The unprotonated complex has a basket-like structure with the DAPI groups *cis*.

H. Manohar and D. Schwarzenbach, *Helv. Chim. Acta*, 57 (1974) 519.

Sulphate complexes of cyanoguanidin and lupetidin, $H_2SO_4.2(C_7H_{15}N).C_2H_4N_4$

($C2/c$) $Z = 8$, $R = 5.3\%$ for 1209 reflections. The structure prepared from sulfuric acid, cyanoguanidin and lupetidin consists of a sulfate ion, multiply hydrogen bonded to two protonated molecules of lupetidin and a neutral molecule of cyanoguanidin.

H.P. Weber, *Helv. Chim. Acta*, 57 (1974) 623.

The NaNCS complex of nonactin

($C2/c$) $Z = 4$, $R = 17.2\%$ based on 2205 reflections. The NaNCS complex of the macrotetrolide antibiotic nonactin shows that Na^+ is coordinated by four carbonyl oxygen atoms (Na^+ — —O = 2.42 Å) and four ether oxygen atoms (Na^+ — —O = 2.77 Å).

M. Dobler and R.P. Phizackerley, *Helv. Chim. Acta*, 57 (1974) 664.

Bis(tribenzyl-titanium(IV))-μ-oxo, $[(PhCH_2)_3Ti]_2O$

($R\bar{3}$) $Z = 1$, $R = 11.2\%$ for 2234 reflections. The structure contains the linear bridge Ti—O—Ti, Ti—O = 1.80 Å, Ti—CH_2 = 2.08 Å, <O—Ti—CH_2 = 113.0°.

H. Stoeckli-Evans, *Helv. Chim. Acta*, 57 (1974) 684.

The orthorhombic modification of a mononuclear peroxotitanium(IV) dipicolinate, $[TiO_2(C_7H_3O_4N)(H_2O)_2]2H_2O$

($Pbcn$) $Z = 4$, $R = 3.4\%$ for 1321 reflections. Titanium has an approximately

pentagonal bipyramidal coordination with the peroxo group and the chelate ligand occupying equatorial sites and the waters forming the apices. Ti-O(peroxo) = 1.833, Ti—O(chelate) = 2.073, Ti—N = 2.164, O—O = 1.458 Å.

H. Manohar and D. Schwarzenbach, *Helv. Chim. Acta*, 57 (1974) 1086.

Staining and fixation of unsaturated membrane lipids by osmium tetroxide; structure of a model osmium(VI) di-ester, $OsO(O_2C_2H_4)_2$

(*Pbcn*) $Z = 4$, $R = 3.5\%$ for 676 independent reflections. The osmium has an essentially square-based pyramidal coordination, the four ester oxygen atoms constituting the basal plane (Os—O(mean) = 1.89(1) Å), and the terminal oxo ligand in the apical position, Os—O = 1.66(1) Å.

R. Collin, W.P. Griffith, F.L. Phillips and A.C. Skapski, *Biochim. Biophys. Acta*, 354 (1974) 152.

Bis(tetraphenylarsonium)uranylbis(pyridine-2,6-dicarboxylate)hexahydrate, $(Ph_4As)_2[UO_2(PDC)_2].6H_2O$, (PDC = pyridine-2,6-dicarboxylic acid)

($P\bar{1}$) $Z = 1$, $R = 7.3\%$ for 2750 reflections. The uranium atom is eight-coordinate, the linear uranyl group being equatorially surrounded by an irregular hexagon of four oxygen atoms and two nitrogen atoms from two PDC ligands. U—O(PDC) = 2.37(2), 2.42(3), U—N = 2.73(2) Å.

G. Marangoni, S. Degetto, R. Graziani, G. Bombieri and E. Forsellini, *J. Inorg. Nucl. Chem.*, 36 (1974) 1787.

$Ca_2Nb_2O_7$

($Pn2_1a$) $Z = 8$, $R = 5.6\%$ for 972 reflections. The single-crystal investigation revealed relationships with distorted orthorhombic perovskites. The results obtained are discussed.

K. Scheunemann and H.K. Müller-Buschbaum, *J. Inorg. Nucl. Chem.*, 36 (1974) 1965.

Bis(*N*-ethylenedimethylaminesalicylaldiminato)dioxouranium(VI), $C_{22}H_{30}N_4O_4U$

($P\bar{1}$) $Z = 2$, $R = 3.4\%$ for 2973 reflections. Each uranium atom is in a pentagonal-bipyramidal environment; the coordination plane is comprised of the two oxygen and three nitrogen atoms of two potentially tridentate Schiff bases with the UO_2 group perpendicular to this plane. U—O(apical) = 1.78(1)Å, U—O(eq) = 2.24(1)Å, U—N = 2.57(1), 2.59(1), 2.69(1)Å.

D.A. Clemente, G. Bandoli, F. Benetollo, M. Vidali, P.A. Vigato and U. Casellato, *J. Inorg. Chem. Nucl.*, 36 (1974) 1999.

Californium oxysulfate and oxysulfide, (CfO_2SO_4) and (CfO_2S)

The lattice parameters for the orthorhombic oxysulfate and trigonal oxysulfide are given.

R.D. Baybarz, J.A. Fahey and R.G. Haire, *J. Inorg. Nucl. Chem.*, 36 (1974) 2023.

Hexakis(di-isopropylthiourea)nickel(II) diperchlorate
($P2_1/c$) $Z = 2$, $R = 10.6\%$ for 1450 independent reflections. Coordination of the bulky ligands to nickel occurs via sulphur and some distortion from the ideal octahedral geometry is observed. Weak intermolecular hydrogen bonds are formed between the perchlorate group and thioamide nitrogen atoms.

G.A. Bentley and J.M. Waters, *J. Inorg. Nucl. Chem.*, 36 (1974) 2247.