Coordination Chemistry Reviews, 15 (1975) 93—105

X-RAY BIBLIOGRAPHY

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The structures of the following compounds appeared in: Acta Crystallogr., Sect. B, Vol. 30, Jan. 15, 1974

(i) Sodium dihydrogen phosphate, NaH_2PO_4
(ii) Potassium triselenylimide, $K_3(NSeO_2)_3$
(iii) Dichloro-2-(2′-pyridyl)-3-(*N*-2-picolylimino)-4-oxo-1,2,3,4-tetrahydroquinazolinecopper(II), $C_{19}H_{15}Cl_2CuN_5O$
(iv) Magnesium dichromate hexamethylenetetramine hexahydrate, $MgCr_2O_7.2[(CH_2)_6N_4].6H_2O$
(v) Nickel Orthoborate, $Ni_3(BO_3)_2$
(vi) Octa-molybdates, $(NH_4)_6Mo_8O_{27}.4H_2O$
(vii) 2,2′2″-Trimethoxytriethylamine—sodium iodide, $NaI.N(CH_2CH_2OCH_3)_3$
(viii) Triethanolamine—sodium iodide, $[N(CH_2CH_2OH)_3]NaI$
(ix) Triethanolamine—strontium nitrate, $\{[N(CH_2CH_2OH)_3]_2Sr\}(NO_3)_2$
(x) Triethanolamine—barium acetate, $[N(CH_2CH_2OH)_3]_2Ba(CH_3CO_2)_2$
(xi) μ-Carbonato-bis(tetraphenylantimony), $[(C_6H_5)_4Sb]_2CO_3$
(xii) $CoMo_2S_4$ and $FeMo_2S_4$
(xiii) Decaammine-μ-peroxodicobalt tetrathiocyanate, $[(NH_3)_5CoO_2Co(NH_3)_5](SCN)_4$
(xiv) $ZnAs_2$
(xv) *O*-Formylphenyltellurenyl bromide, C_7H_5OTeBr
(xvi) Mercury(I) trifluoroacetate, $Hg_2(CF_3CO_2)_2$
(xvii) Silver dicyanonitromethanide, $AgC(CN)_2NO_2$
(xviii) Deuterium triuranate, $D_2U_3O_{10}$
(xix) Uranyl chloride monohydrate, $UO_2Cl_2.H_2O$
(xx) $UOCl_2$
(xxi) Barium 2-*O*-sulfonato-*L*-ascorborate dihydrate, $Ba(C_6H_6O_9S).2H_2O$
(xxii) DL-Histidine, $C_6H_9N_3O_2$
(xxiii) 5-Methoxytryptamine, $C_{11}H_{14}N_2O$
(xxiv) $(\phi_4As)CuCl_3$
(xxv) CaGaN
(xxvi) Silver$(+)_{589}$-*cis*-dinitrobis-(D-alaninato)cobaltate(III), $(+)_{589}$-$Ag[Co(NO_2)_2$-$(D\text{-}ala)_2]$
(xxvii) Potassium arsenic hexafluorine; a redetermination, $KAsF_6$

Acta Crystallogr., Sect. B, Vol. 30, Feb. 15, 1974

(i) Diamminenickel(II) tetracyanonickelate(II) di-biphenyl, $Ni(NH_3)_2Ni(CN)_4.2C_{12}H_{10}$.
(ii) Dioxobis-(1,3-diphenylpropanedionato)molybdenum(VI), $(C_{15}H_{11}O_2)_2MoO_2$
(iii) Cadmium chloride monohydrate, $CdCl_2.H_2O$
(iv) $BaCd_{4.43}$
(v) Cobalt(imidazole)$_2$(acetate)$_2$
(vi) $K_4P_2O_7.3H_2O$
(vii) Leifite, $Na_6[Si_{16}Al_2(BeOH)_2O_{39}]. 1.5H_2O$
(viii) Zinc *O*-ethoxybenzoate monohydrate, $ZnC_{18}H_{18}O_6.H_2O$
(ix) Oxydiacetatodioxouranium(VI), UO_2(oxydiacetato)
(x) Ti_8S_3
(xi) Orthorhombic antimony trioxide, Sb_2O_3
(xii) Tri-μ-hydroxo-tri(pyridine-2-carboxylato)triberyllium monohydrate, $[Be_3(OH)_3(C_6H_4NCOO)_3], H_2O$.
(xiii) Neodymium metaphosphate, (NdP_3O_9) and ultraphosphate, (NdP_5O_{14}).
(xiv) Di-μ-thiocyanato(*S,N*)-diisothiocyanatobis[2-(2′-pyridyl)-3-(*N*-2-picolyimino)-4-oxo-1,2,3,4-tetrahydroquinazoline]dicobalt(II), $[Co(C_{19}H_{15}N_5O)(SCN)_2]_2$
(xv) Tris(cyclopentadienyl)titanium, $(C_5H_5)_3Ti$
(xvi) Stercorite, $NaNH_4HPO_4.4H_2O$
(xvii) Bis(dioxane)potassium bis(trimethylsilyl)amide, $K(C_4H_8O_2)_2N[Si(CH_3)_3]_2$
(xviii) Trimethyltin methoxide, $(CH_3)_3SnOCH_3$
(xix) Tetrasodium imidodiphosphate decahydrate, $Na_4(PO_3NHPO_3).10H_2O$
(xx) Tin(II)monofluorophosphate, $SnPO_3F$
(xxi) Bis-(2-hydroxy-5-methylacetophenato)nickel(II)
(xxii) Ba_2SiS_4
(xxiii) NbS_2

Acta Crystallogr., Sect. B, Vol. 30, March 15, 1974

(i) Sodium diborate $Na_2O.2B_2O_3$
(ii) β-$Ca(NO_3)_2.2H_2O$
(iii) Cadmium picolinate, $Cd(C_5H_4NCOO)_2$
(iv) Twinned monoclinic potassium manganicyanide, $K_3[Mn(CN)_6]$
(v) β-Alum $CsTi(SO_4)_2.12H_2O$
(vi) (*Pentahapto*-cyclopentadienyl)hydridomolybdenum-μ-dimethylaluminum-μ-[methylaluminum-di-(μ-*pentahapto (monohapto)-cyclo*pentadienyl)dimethylaluminum]-(*pentahapto*-cyclopentadienyl)-hydridomolybdenum, $[(C_5H_5)(C_5H_4)MoH]_2Al_3(CH_3)_5$
(vii) Hofmann-type clathrate, $Mn(NH_3)_2Ni(CN)_4.2C_6H_6$
(viii) $LiKPO_3F.H_2O$
(ix) Cobalt(imidazole)$_6$ (acetate)$_2$ monohydrate
(x) Bis(trimethylphosphite)tetraphenylboronrhodium(I), $Rh[P(OCH_3)_3]_2B(C_6H_5)_4$

(xi) α-Sodium triborate, α-$Na_2O.3B_2O_3$
(xii) *Cis*-Dichloro-(1,10-diaza-4,7,13,16-tetraoxa-21,24-dithiabicyclo-[8,8,8]hexacosane)-palladium(II), $Pd(C_{18}H_{36}N_2O_4S_2)Cl_2$
(xiii) $La_4Ge_3S_{12}$
(xiv) $K_5(UO_2)_2F_9$
(xv) (1-Oxa-7,10-dithia-4,13-diazacyclopentadecane)nickel(II) nitrate, a nickel macrocycle, $[Ni(C_{10}H_{22}N_2OS_2).(H_2O)](NO_3)_2$
(xvi) Tantalum oxynitride, TaON
(xvii) $Ba_5W_3Li_2O_{15}$
(xviii) $Sm\ Au_6$
(xix) Trisacetylacetonatocobalt(III), $Co(C_5O_2H_7)_3$
(xx) Lithium hydrazinium sulphate, $Li(N_2H_5)SO_4$
(xxi) Bis(benzene)chromium iodide, $Cr(C_6H_6)_2I$
(xxii) Bistetramethylammonium dioxotetrabromouranate(VI), $[(CH_3)_4N]_2UO_2Br_4$

Acta Crystallogr., Sect. B, Vol. 30, April 15, 1974
(i) $Bi_2Cu_3S_4Cl$
(ii) Mo_6Ga_{31}
(iii) Monoclinic $NaCaHSiO_4$
(i) Bis-(3,5-dimethyl-1,2-dithiolium)tetrachloroferrate(II), $[(SacSac)_2FeCl_4]$
(v) Bis-(3,5-diphenyl-1,2-dithiolium)tetrachloroferrate(II), $(SbzSbz)_2[FeCl_4]$
(vi) Bis-(3,5-diphenyl-1,2-dithiolium)tetrachloromercurate(II), $(SbzSbz)_2[HgCl_4]$
(vii) Bis-(3,5-diphenyl-1,2-dithiolium)tetrachloroferrate(III) Chloride, $(SbzSbz)_2[FeCl_4]Cl$
(viii) Tetrameric arsenic selenide, As_4Se_4
(ix) Gypsum, $CaSO_4.2H_2O$
(x) α-N_2
(xi) Pentacoordinate phosphorus compounds. II.2-Fluoro-2,2′-spirobis(1,3,2-benzodioxaphosphole), $(C_6H_4O_2)_2PF$
(xii) Pentacoordinate phosphorus compounds.III.2-Methyl-2,2′-spirobis-(1,3,2-benzodioxaphosphole), $(C_6H_4O_2)_2\ PCH_3$
(xiii) $K_3Sb_5O_{14}$ and $K_2Sb_4O_{11}$
(xiv) Neodymium ytterbium sulphide, $NdYbS_3$
(xv) Bellingerite, $3Cu(IO_3)_2.2H_2O$
(xvi) Calcium L-glutamate trihydrate, $CaC_5H_7NO_4.3H_2O$
(xvii) $CsCuCl_3$ at 423°K
(xviii) Silver perchlorate adduct of iron(III) trisacetylacetonate monohydrate $Fe(acac)_3.AgClO_4.H_2O$
(xix) Ca_5Sb_3
(xx) β-$Cs_3Sb_2Cl_9$ and $Cs_3Bi_2Cl_9$
(xxi) $CuK_2(PO_3)_4$ and $CoK_2(PO_3)_4$

(xxii) 1,3-Diphenyl-1,3-propanedione(1,6-dichloro-1,5-cyclooctadiene)rhodium(I), $C_{23}H_{21}O_2Cl_2Rh$

(xxiii) Tris(thiosemicarbazide)nickel(II) dichloride monohydrate, $Ni(CN_3H_5S)_3Cl_2.H_2O$

(xxiv) Bisanilinebis(dimethylglyoximato)cobalt(III) chloride, $[Co(Hdmg)_2(aniline)_2]Cl$

(xxv) Silver dicyanonitromethanide, $AgC(CN)_2NO$

(xxvi) μ_3-Hydroxo(oxo)-tri-μ-(2-propylamino-2-methyl-3-butanoneoximato)-triaquo-tricopper(II)-perchlorate.4H_2O, $[Cu_3(C_8H_{17}N_2O)_3(H_2O)_3OH_{\frac{1}{2}}](ClO_4)_{\frac{3}{2}}(H_2O)_4$

Inorg. Chem., Vol. 13, No. 6, June 1974

(i) Dehydrated cobalt(II)—exchanged zeolite A and its carbon monoxide adduct, $Co_4Na_4Si_{12}Al_{12}O_{48}$ and $Co_4Na_4Si_{12}Al_{12}O_{48}.4CO$.

(ii) Tetraphenylphosphonium trichlorocuprate(II), $(C_6H_5)_4PCuCl_3$

(iii) Potassium tetrabromoplatinate(II), K_2PtBr_4

(iv) *trans*-Chloro(methylimido)tetrakis(methylamine)rhenium(V) perchlorate, $[Re(CH_3NH_2)_4(CH_3N)Cl](ClO_4)_2$

(v) Platinum bronzes.IV. $Na_xPt_3O_4$, $Cd_xPt_3O_4$ and $CaPt_2O_4$

(vi) Structure of metallocarboranes I. 2,6-Di-η-cyclopentadienyloctahydro-1,10-dicarba-2,6-dicobalta-*closo*-decarborane at −150°, a bimetallocarborane with a metal—metal bond, 2,6-$(\eta\text{-}C_5H_5)_2$-2,6-Co_2-1,10-$C_2B_6H_8$.

(vii) Structures of metallocarboranes II. Cesium 3-η-cyclopentadienyloctahydro-4-carba-3-cobalta-*closo*-nonaborate(1—), $Cs^+[(C_5H_5)Co(CB_7H_8)]^-$.

(viii) Structures of metallocarboranes III. 2,3-Di-η-cyclopentadienyl-1,7-dicarba-2,3-dicobaltadodecaborane(10), $(\eta\text{-}C_5H_5)_2Co_2C_2B_8H_{10}$

(ix) *cis*-Bis(*pentahapto*cyclopentadienyl)tricarbonyl(triphenylphosphito)diiron, $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3P(OPh)_3$

(x) (*Pentahapto*cyclopentadienyl) (7,8,9,10,11,12-*hexahapto*dodecahydro-7,9-dicarba-*nido*-dodecaborato)cobalt(III), $(\pi\text{-}C_5H_5)Co(B_{10}C_2H_{12})$

(xi) Bis(triphenylphosphine)octakis(phenylethynyl)tetracopperdiiridium(4Cu—Cu) (8Cu—Ir), $Cu_4Ir_2(PPh_3)_2(C{\equiv}CPh)_8$.

(xii) Molecules with an M_4X_4 core II. Tetrameric triphenylphosphinecopper(I) bromide, $[PPh_3CuBr]_4.2CHCl_3$.

J. Amer. Chem. Soc., Vol. 96, No. 9, May 1, 1974.

(i) Five-membered cyclic acyl phosphate, $(PO_4C_6H_9O_2)$

(ii) α,γ-Dimethyl-α,γ-dihydrooctaethylporphinatonickel(II), $Ni(OEPMe_2)$

(iii) Potassium bis(dithiosquarato)nickelate(II), $K_2Ni(S_2C_4O_2)_2.2H_2O$

(iv) $B_5H_9[P(CH_3)_3]_2$

J. Amer. Chem. Soc., Vol. 96 No. 10, May 15, 1974

(i) Two-coordinate palladium(0) complexes, $Pd[PPh(t\text{-}Bu)_2]_2$ and $Pd[P(t\text{-}Bu)_3]_2$

J. Amer. Chem. Soc., Vol. 96 No. 11, May 29, 1974

(i) Pyridinetetracarbonyliron and pyrazinetetracarbonyliron, $Fe(CO)_4C_5H_5N$ and $Fe(CO)_4C_4H_4N_2$
(ii) $[Cu_2([14], 4, 11\text{-diene-}N_4)_2CN](C10_4)_3$ where [14] 4, 11-diene-N_4 is 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene, and $[Cu_2(tren)_2(CN)_2](PPh_4)_2$ where tren = 2,2′,2″-triaminotriethylamine
(iii) Tris(pyrrolidyldithiocarbamato)iron(IV) perchlorate, $[Fe(pyrr(dtc))_3]$-(ClO_4)
(iv) Condensation product of acetylacetone with pyruvilidinatotetraamminecobalt(III)ion.
(v) Bacteriochlorophyll b

J. Amer. Chem. Soc., Vol. 96, No. 12, June 12, 1974

(i) Octamethyldimolybdate(II) ion, $Li_4[Mo_2(CH_3)_8] \cdot 4C_4H_8O$
(ii) A tetranuclear basic quinoline adduct of copper(II) trifluoroacetate, $[Cu_2OH(O_2CCF_3)_3(quinoline)_2]_2$
(iii) Chlorotris(*N*,*N*-dimethyldithiocarbamato)titanium(IV), $Ti[S_2CN(CH_3)_2]_3Cl$
(iv) η′-Pentachlorocyclopentadienylpentacarbonylmanganese(I), $(\eta' C_5Cl_5)(CO)_5Mn$
(v) A cubane-like cobalt—nitrosyl complex, $Co_4(NO)_4(\mu_3\text{-}NC(CH_3)_3)_4$
(vi) Two cubane-like iron—Nitrosyl complexes, $Fe_4(NO)_4(\mu_3\text{-}S)_4$ and $Fe_4(NO)_4(\mu_3\text{-}S)_2(\mu_3\text{-}NC(CH_3)_3)_2$
(vii) $(BzPh_3P)_2\,Fe(DED)_3$, where DED^{2-} = 1,1-dicarboethoxyethylene-2,2-dithiolate anion.
(viii) 2-$[IrBr_2(CO)(PMe_3)_2]B_5H_8$

Tridecafluorohexaantimony(III) pentakis[hexafluoroantimonate(V)]
$(P\bar{1}$, C_i^1 No. 2), $Z = 1$, $R = 7.6\%$ from 1703 independent reflections. The new antimony fluoride of empirical composition $Sb_{11}F_{43}$ consists of SbF_6^- anions and a polymeric chain cation $[Sb_6F_{13}]_n^{5+}$. In the polymeric chain separate $SbF_2{}^+$ and $Sb_2F_5{}^+$ units can be distinguished if the shorter Sb^{III}—F bonds only are considered. The coordination around Sb in $Sb_2F_5{}^+$ is pyramidal, F—Sb—F(mean) = 82°, Sb—F = 2.06Å. In $SbF_2{}^+$, Sb—F = 1.90Å, and 1.98Å, F—Sb—F = 89° and 91°.
A.J. Edwards and D.R. Slim, *J. Chem. Soc. Chem. Commum.*, (1974) 178.

Aquocopper(II)-μ-(triethylenetetraaminehexaacetato)aquocopper(II) heptahydrate, $Cu_2(ttha)(H_2O)_2$
$(P2_1/c)$ $Z = 2$, $R = 6.8\%$ from 2803 unique reflections. The structure shows the title compound to be in its fully open or extended molecular form, in

apparent disagreement with earlier esr results. The molecule is centrosymmetric about the mid-point of the central ethylene group. The stereochemistry about the copper atom is distorted octahedral with a water molecule, O(7), filling the sixth coordination site. The copper atom is virtually coplanar with coordination square comprising O(1), N(1), O(5) and O(7), with Cu—O(1) = 1.961(9), Cu—O(5) = 1.949(9), Cu—N(1) = 2.065(8) and Cu—O(7) = 1.988(8). The Cu—O(6) distance, 2.512(9) Å, is much longer than both Cu—O(1), and Cu—O(5), indicating O(6) to be the protonated oxygen. The intramolecular Cu(II)—Cu(II) distance = 7.655(2) Å.

P. Leverett, *J. Chem. Soc. Chem. Commun.*, (1974) 161.

A ruthenium complex containing nonafluoro(phenylazo)phenyl-2C,N′ and diphenyl-2-(η-cyclopentadienylphenyl)phosphine groups, $\overline{\mathrm{RuC_6F_4N}}:\mathrm{NC_6F_5}$-$(\mathrm{Ph_2PC_6H_4}$-η-$\mathrm{C_5H_4})$

($P2_1/c$) $Z = 4$, $R = 7.5\%$ based on 3092 independent non-zero reflections. The coordination geometry is of the familiar (η-C_5H_5)ML_3 type, distorted somewhat by the constraints of the chelate rings. The metallated nonafluoro(phenylazo)phenyl ligand is non-planar, with the free pentafluorophenyl ring twisted by 64.2° from the Ru—C(1)—C(6)—N(1)—N(2) plane, Ru—C(1) = 2.018(10), Ru—N(2) = 2.014(8), N(1)—N(2) = 1.324(11) Å. Linkage of the cyclopentadienyl carbon atom C(17) with the phenyl carbon C(19) forms a σ, π-chelating arrangement, Ru—cp (mean) = 2.190(11) and 2.248(12), C(17)—C(19) = 1.472(12).

M.I. Bruce, R.C.F. Gardner, B.L. Goodall, F.G.A. Stone, R.J. Doedens and J.A. Moreland, *J. Chem. Soc. Chem. Commun.*, (1974) 185.

Novel polynuclear complex halogeno anions containg mercury and platinum or palladium, $[\mathrm{Et_4N}]_2\mathrm{Hg_2PtCl_8}$, A, $[\mathrm{Et_4N}]_2\mathrm{Hg_3PtCl_{10}}$, B and $[\mathrm{Et_4N}]_2\mathrm{Hg_3PdCl_{10}}$, C

A. ($P2_1/c$) $Z = 2$, $R = 7.3\%$ from 888 visually observed independent reflections. B. ($P4_2/mmc$) $Z = 2$, $R = 12\%$ based on 428 independent visually estimated reflections. C. ($P4_2/mmc$) $Z = 2$, $R = 13\%$ based on 329 visually observed independent reflections. The common feature of all three compounds is the trinuclear anionic unit $[\mathrm{Hg_2MCl_8}]^{2-}$. The anion in A is centrosymmetric, in B and C, it has *mmm* symmetry. Bond lengths in the anion are as expected. The main differences between the structures of A and B or C are found in the long intermolecular bonds. In A, terminal chlorine atoms, Hg—Cl = 3.15(2), link the anions to form polymeric zig-zag chain anions $[\mathrm{Hg_2PtCl_8}]_n^{2n-}$. In B and C no such direct links exist, the closest contact is Hg(1)—Hg(1′) = 3.51(1) B, or 3.48(1) C. These give a direct measure of the van der Waals' radius for mercury(II) as 1.75 Å.

.M. Barr, M. Goldstein, T.N.D. Hairs, M. McPartlin and A.J. Markwell, *Chem. Soc. Chem. Commun.*, (1974) 221.

$\mathrm{UOF_4}$

($I\bar{4}2d$, D_{2d}^{12}, No 122), $Z = 8$, $R = 11.0\%$ from 637 independent reflections.

The configuration around the uranium atom is a pentagonal bipyramid of six fluorine atoms and one oxygen atom. U—F = 2.34(3), U—O = 1.87(15) Å.
J.C. Taylor and P.W. Wilson, *J. Chem. Soc. Chem. Commum.*, (1974) 232.

A novel bimetallocarborane anion, $[(\eta\text{-}C_5\text{-}H_5)\text{—}Co\text{—}C_2B_8H_{10}\text{—}Co\text{—}C_2B_8H_{10}]^-$
$(P2_1/c)$ $Z = 4$, $R = 7.5\%$ from 2895 reflections. The structure of $[Et_4N]$-$[(C_5H_5)Co_2(C_2B_8H_{10})_2]$ contains a bimetallic anion $Co_2C_2B_8H_{10}$ in which one cobalt atom is shared with a terminal $C_2B_8H_{10}$ group and the other cobalt atom is terminally bonded to a cyclopentadienyl ring.
G. Evrard, J.A. Ricci, Jun., I. Bernal, W.J. Evans, D.F. Dustin and M.F. Hawthorne, *J. Chem. Soc. Chem. Commum.*, (1974) 234.

"Non-oxidized" hexamethylbenzene niobium chloride cluster, $[(Me_6C_6)_3Nb_3Cl_6]Cl$
$(P6/m, C^1_{6h}$, No 175) $Z = 1$, $R = 12\%$ for 413 independent reflections. The structure shows $[(Me_6C_6)_3Nb_3Cl_6]Cl$ to be trinuclear, rather than hexanuclear. The crystal is disordered, with the metal cluster lying on a site of crystallographic C_{6h} symmetry. The molecular symmetry approximates to D_{3h}. Nb—Nb = 3.334(6), Nb—Cl (bridging) = 2.466(8) and 2.499(9) Å.
M.R. Churchill and S.W.-Y. Chang, *J. Chem. Soc. Chem. Commun.*, (1974) 248.

Stereochemistry of electrophilic addition to tricarbonyldieneiron complexes; acylation of tricarbonyl-(*trans, trans*-hexa-2,4-diene)iron
$(P2_1/c)$ $Z = 4$, $R = 8.0\%$ from 1538 independent reflections. The structure of a substituted tricarbonylallyliron cationic intermediate unambiguously demonstrates that tricarbonyl (*trans, trans*-hexa-2,4-diene)iron undergoes stereospecific *endo* attack under Friedel—Crafts conditions.
E.O. Greaves, G.R. Knox, P.L. Pauson, S. Toma, G.A. Sim and D.I. Woodhouse, *J. Chem. Soc. Chem. Commun.*, (1974) 257.

Bis(tetracarbonylphenyldiazomanganese), $[PhN{=}NMn(CO)_4]_2$
$(P\bar{1}, C^1_i$, No. 2) $Z = 1$, $R = 3.43\%$ for 1974 reflections. The molecule lies on a crystallographic centre of symmetry, two $Mn(CO)_4$ units are linked together by two bridging phenyldiazo ligands, with Mn—N(1) = Mn′—N(1′) = 2.031(2), and Mn—N(1′) = Mn′—N(1) = 2.021(2) Å. Mn · · · Mn′ = 3.234(1) non bonding. Mn_2N_2 is strictly planar. Distances and angles in the CO ligands are normal.
E.W. Abel, C.A. Burton, M.R. Churchill and Kuo-Kuang G. Lin, *J. Chem. Soc. Chem. Commun.*, (1974) 268.

Carbido-carbonyl clusters of rhodium, $Rh_8(CO)_{19}C$, A, and $[H_3O][Rh_{15}(CO)_{28}$-$C_2]$, B
A. $(P\bar{1})$ $Z = 2$, B. $(Pbca)$ $Z = 4$. A contains a metal atom cluster derived from the prismatic unit present in the dianion to which two extra atoms have

been attached, one in a capping position on one rectangular face and the other in a bridging position on one base edge. Rh—Rh(mean) = 2.81 Å. The carbido C is situated in the centre of the prism, Rh—C(mean) = 2.127 Å. B possesses C_{2v} idealized symmetry and the Rh_{15} cluster can be described as a centred tetracapped pentagonal prism, Rh—Rh = 2.738—3.332 Å. The central rhodium atom, which has a highly metallic character, is linked to 12 other Rh atoms (mean distance = 2.90 Å) and to both carbido carbon atoms, Rh—C(mean) = 2.06 Å.

V.G. Albano, P. Chini, S. Martinengo, M. Sansoni and D. Strumolo, *J. Chem. Soc. Chem. Commun.*, (1974) 299.

Tetraethylammonium monothiothenoyltrifluoroacetonatopentacarbonyltungstate(0) $[Et_4N][W(CO)_5(C_8H_4OF_3S_2)]$

($C222_1$) $Z = 8$, $R = 4.4\%$ based on 2559 unique reflections. This is the first metal complex in which a β-diketone type of ligand has been observed in its *trans*-isomeric form.

M. McPartlin, G.B. Robertson, G.H. Barnett and M.K. Cooper, *J. Chem. Soc. Chem. Commun., (1974) 305.*

The μ-dichlororhenium(III) complex of 1,2-bis(diphenylphosphino)ethane, $Re_2Cl_6(dppe)_2 \cdot 2MeCN$

($P\bar{1}$) $Z = 1$ (dimeric unit), $R = 6\%$ from 3250 non-zero reflections. The structure is a centrosymmetric chlorine bridged dimer with the rhenium atoms in a distorted octahedral environment. Within the dimer there is no significant Re—Re interaction, Re—Re = 3.809 Å. Re—Cl(terminal) = 2.307—2.322, Re—P = 2.370—371 Å.

J.A. Jaecher, W.R. Robinson and R.A. Walton, *J. Chem. Soc. Chem. Commun.*, (1974) 306.

cis-Dichloro[(*R*)-α-methylbenzylamine][(*S*)-1,2,2-trimethylpropyl (*R*)-vinyl ether]platinum(II)

($P2_12_12_1$) $Z = 4$, $R = 7.1\%$ based on 1764 independent reflections. The structural analysis of the title compound shows that the absolute configuration of the chiral centre formed by complexation of the vinyl group to Pt^{II} is *R* and gives indications on the conformation of the amine and olefin ligands. The complex shows almost undistorted square planar arrangement of the ligands around the Pt atom with two chlorines in a *cis* position.

F. Sartori, L. Leoni, R. Lazzaroni and P. Salvadori, *J. Chem. Soc. Chem. Commun.*, (1974) 322.

Hydrido-tris(triphenylphosphine)ruthenium(II) ion, $[RuH(PPh_3)_2(\eta\text{-Ph—PPh}_2)]$

($P2_1/c$) $Z = 4$, $R = 5.4\%$ based on 4658 reflections. The structure contains separate $[RuH(PPh_3)_3]^+$ and BF_4^- ions. The complex cation has one of the PPh_3 groups bonded to the metal atom via a η-phenyl group, while the other two PPh_3 ligands bond normally via phosphorus atoms. A hydride hydrogen atom completes the coordination about Ru. Ru—C (mean) = 2.28, Ru-η-phenyl ring centroid = 1.78 Å.

J.C. McConway, A.C. Skapski, L. Phillips, R.J. Young and G. Wilkinson, *J. Chem. Soc. Chem. Commun.*, (1974) 327.

Octaethylporphinium(monocation)triiodide

($Cmca$) $Z = 8$, $R = 10.9\%$ from 1002 non-zero reflections. The x-ray analysis shows that in the porphyrin monocation one pyrrole ring, probably protonated, is tilted 14° from the plane through the other three pyrrole rings by the repulsions between the inner three hydrogen atoms.

N. Hirayama, A. Takenaka, Y. Sasada, E.-I. Watanabe, H. Ogoshi and Z-I. Yoshida, *J. Chem. Soc. Chem. Commun.*, (1974) 330.

A novel ligand-bridged olefinic tertiary arsine complex, $C_{11}H_{15}AsAg_2(NO_3)_2$

($P2_12_12_1$) $Z = 4$, $R = 7.8\%$ from 774 independent visually estimated reflections. The structure shows that in $C_{11}H_{15}AsAg_2(NO_3)_2$, the normally chelating *o*-allylphenyldimethylarsine ligand is bonded via its arsine and olefin groups to two different silver atoms. The ligand links two silver atoms, the olefin being π-bonded to Ag(1) and the arsenic atom coordinated to Ag(2). These two silver atoms are also bridged by a doubly-bidentate nitrate group with four similar Ag—O bond lengths. A second bridging nitrate completes the chain-like structure forming two unsymmetrical bidentate linkages, Ag—O (long) = 2.85, 2.81 Å Ag—O (short) = 2.43, 2.35 Å.

M.K. Cooper, R.S. Nyholm, P.W. Carreck and M. McPartlin, *J. Chem. Soc. Chem. Commun.*, (1974) 343.

Rhodium-diarylcarbene complex, $[Rh(CPh_2)Cl(C_5H_5N)]_2CO.2CH_2Cl_2$

($C2(C_2^3)$) $Z = 2$, $R = 7\%$ based on 1831 reflections. The molecule contains two-fold symmetry such that the CO group lies on a crystallographic two-fold axis. Rh—Rh = 2.51 Å, indicative of a Rh—Rh bond. The CPh_2 group occupies bridging positions.

T. Yamamoto, A.R. Garber, J.R. Wilkinson, C.B. Boss, W.E. Streib and L.J. Todd, *J. Chem. Soc. Chem. Commun.*, (1974) 354.

Reaction of trifluoroacetonitrile with π-allylic-iridium complexes; structure of an iridium(I) six-membered ring chelate complex, $C_{27}H_{22}F_6IrN_2OP$

($P2_1/c$) $Z = 4$, $R = 4.5\%$ from 3277 reflections. $Ir(\pi\text{-}2\text{-}Me{-}C_3H_4)(CO)(PPh_3)_2]$ reacts with CF_3CN to afford an Ir(I) six-membered chelate complex. The central Ir exhibits square-planar coordination with Ir—P = 2.271(3), Ir—C(1) = 1.831(13), Ir—N(1) = 2.028(10) and Ir—N(2) = 2.023(9) Å.

M. Green, S.H. Taylor, J.J. Daly and F. Sanz, *J. Chem. Soc. Chem. Commun.*, (1974) 361.

D-Penicillaminatolead(II), $Pb[SCMe_2CH(NH_2)—CO_2]$

($P2_1$) $Z = 2$, $R = 4.7\%$ from 2129 non-equivalent reflections. The amino acid acts as a tridentate chelate ligand, bonded to the lead atom, and forms weaker bonds with the lead atoms of two neighbouring complexes in the crystal.

H.C. Freeman, G.N. Stevens and I.F. Taylor Jun., *J. Chem. Soc. Chem. Commun.*, (1974) 366.

Two coordinate phosphine—palladium(O) complexes, tricyclohexyl- and di(*t*-buty)phenylphosphine derivatives, Pd[P(cyclohexyl)$_3$]$_2$, A and Pd(PBu$_2^t$Ph)$_2$, B

A. (*C*2/*c*) $Z = 4$, $R = 6.6\%$ based on 2181 reflections. B. (*Fdd*2) $Z = 8$, $R = 10\%$ based on 1264 reflections. The most remarkable occurence is that the molecules, both having molecular and crystallographic C_2 symmetry, display quite different geometry of the P—Pd—P group. For A the P—Pd—P group is bent (∡ = 158.4(3)°) whereas in B it is almost linear (∡ = 175.7(3)°). Pd—P = 2.26, A; 2.28 Å, B.

A. Immirizi and A. Musco, *J. Chem. Soc. Chem. Commun.*, (1974) 400.

Bis[dimethylbis(1-pyrazolyl)gallato]nickel(II), [Me$_2$Ga(N$_2$C$_3$H$_3$)$_2$]$_2$Ni

(*P*2$_1$/*c*) $Z = 2$, $R = 4.9\%$ based on 1352 observed reflections. The structure shows that the two-six-membered Ga(N—N)$_2$Ni rings are in the boat conformation with a planar arrangement of four nitrogen atoms about the nickel atom which lies on a crystallographic centre of symmetry, giving the whole molecule a pseudo-chair conformation. Ga—N(mean) = 1.977(4), Ga—C = 1.944(8) Å.

D.F. Rendle, A. Storr and J. Trotter, *J. Chem. Soc. Chem. Commun.*, (1974) 406.

cis-Dichloropentane-2,4-dionato-*trans*-bis(triphenylphosphine)rhenium(III), ReCl$_2$(C$_5$H$_7$O$_2$)(P(C$_6$H$_5$)$_3$)$_2$

(*P*2$_1$/*c*) $Z = 4$, $R = 6.8\%$ based on 2686 observed reflections. The ligands which surround the rhenium atom are at the apices of a distorted octahedron, with the (PPh$_3$) groups *trans*. Re—Cl(avg) = 2.37, Re—O(avg.) = 2.03. Re—P = 2.485(4), 2.469(5) Å.

I.D. Brown, C.J.L. Lock and C. Wan, *Can. J. Chem.*, 52 (1974) 1704.

Sodium-coordinated heptamolybdate anions, Na$_6$Mo$_7$O$_{24}$(H$_2$O)$_{14}$

(*P*2$_1$*ab*) $Z = 4$, $R = 5.6\%$ using 2372 independent reflections. The structure consists of Mo$_7$O$_{24}$$^{6-}$ anions embedded in a sodium—water oxygen double-chain arrangement along the *y*-axis, with the sodium ions directly coordinated to the anions. Seven MoO$_6$ octahedra are joined together by common edges. Each sodium ion is octahedrally coordinated to six water and group oxygen atoms. These octahedra form chains by sharing edges and corners. Mo—Mo = 3.19—3.45, Mo—O = 1.67—1.76, 1.88—2.01, 2.11—2.33 Å.

K. Sjöbom and B. Hedman, *Acta. Chem. Scand.*, 27 (1973) 3673.

A non-stoichiometric boride, $IrB_{\sim 1.35}$
(*C*2/*m*), *R* = 8.2% based on 632 reflections. The structure is described in terms of puckered boron layers and puckered metal double layers, which also contain boron atoms in trigonal prismatic interstices. The structure does not contain a three dimensional boron network.
T. Lundström and L.-E. Tergenius, *Acta. Chem. Scand.*, 27 (1973) 3705.

Monomeric dimethyl(cyclopentadienyl)aluminum, $(CH_3)_2Al(C_5H_5)$. A gas phase electron diffraction investigation of monomeric Me_2AlCp
The structure contains *polyhapto* (asymmetrically π-bonded) rings to Al, perpendicular distance from Al to ring = 2.10(2) Å, perpendicular distance from Al to the symmetry axis of the ring = 0.99(10), C—C = 1.422(2), Al—C(Me) = 1.952(3) Å.
D.A. Drew and A. Haaland, *Acta. Chem. Scand.*, 27 (1973) 3735.

Calcium sulphosilicate, $Ca_5(SiO_4)_2SO_4$
(*Pcmn*) *Z* = 4, *R* = 4.2% from 570 independent reflections. The structure is isostructural with the mineral silicocarnotite.
P.D. Brotherton, J.M. Epstein, M.W. Pryce and A.H. White, *Aust. J. Chem.*, 27 (1974) 657.

Hexakis(hydroxylamine-N)nickel(II) sulphate, $[Ni(NH_2OH)_6]SO_4$
($P\bar{1}$) *Z* = 2, *R* = 9.5% from 1166 independent reflections. In the two crystallographically independent centrosymmetric cations, the nickel atoms are octahedrally coordinated by the ligand nitrogen atoms, Ni—N (mean) = 2.12 Å. N—O (mean) = 1.44 Å. The Ni—N—O angles range from 110 to 117°. The $SO_4^{=}$ geometry is normal and undistorted.
L.M. Engelhardt, P.W.G. Newman, C.L. Raston and A.H. White, *Aust. J. Chem.*, 27 (1974) 503.

Comparison of the molecular structures of $(\pi\text{-}C_5H_5)Fe(CO)_2P(CF_3)_2$, A and $(\pi\text{-}C_5H_5)Fe(CO)_2P(O)(CF_3)_2$, B. A. ($P2_1/c$, C^5_{2h}, No. 14) *Z* = 4, *R* = 4.8% from 1882 reflections. B. ($P2_1/c$, C^5_{2h}, No. 14) *Z* = 4, *R* = 4.5% from 1777 reflections. On oxidation of A at phosphorus the Fe—P bond length decreases from 2.265(3) to 2.191(3) Å, while the mean Fe—CO bond length increases from 1.768(7) to 1.780(7) Å.
M.J. Barrow, G.A. Sim, R.C. Dobbie and P.R. Mason, *J. Organometal. Chem.*, 69 (1974) C4.

Tris(triphenylphosphine)rhodium benzoate, $(Ph_3P)_3RhOOCPh.O.5C_6H_6$
(*P*1) *Z* = 2, *R* = 6.8% from 2587 reflections. The complex is monomeric. The rhodium coordination is almost square planar, Rh being coordinated to three PPh_3 ligands and a benzoate ligand by monodentate bonding. Rh—O(1) = 2.10 Å, Rh · · · O(2) = 3.230 Å, non-bonding but not excluding the possibility of a weak additional coordination.
I.S. Kolomnikov, A.O. Gusev, T.S. Belopotopova, M. Kh. Grigoryan, T.V.

Hysyak, Yu. T. Struchkov and M.E. Vol'pin, *J. Organometal. Chem.*, 69 (1974) C10.

Methyldichlorobis(*N*-methyl-*N*-nitrosohydroxylaminato)tantalum(V)

($P2_1/n$) $Z = 4$, $R = 8.7\%$ based on 1336 reflections. The coordination sphere of the tantalum atom is a slightly distorted pentagonal bipyramid with two chlorine atoms in axial positions [2.359(8), 2.357(10) Å], four oxygens [2.090(17), 2.081(21), 2.108(18), 2.066(26) Å] and one methyl group [2.248(28) Å] in a pentagonal girdle.

J.D. Wilkins and M.G.B. Drew, *J. Organometal. Chem.*, 69 (1974) 111.

N-(Trimethylstannyl)-*N*-nitromethylamine, $Me_3SnN(Me)NO_2$

($P2_1/c$) $Z = 8$, $R = 5.7\%$ from 600 unique observed reflections. Planar trimethyltin groups are linked by planar *N*-nitromethylamine groups to form infinite —Sn(Me)$_3$—N(Me)—N(O)—O—Sn(Me$_3$)— chains in the axis direction. There are two sets of crystallographically non-equivalent chains, but the bond lengths and angles agree within experimental error. The coordination of Sn is approximately trigonal bipyramidal, with axial nitrogen and oxygen. Mean bond lengths are Sn—C = 2.16, Sn—N = 2.33, N—C = 1.45, N—N = 1.29, N—O = 1.24 and O—Sn = 2.39 Å.

A.M. Domingos and G.M. Sheldrick, *J. Organometal. Chem.*, 69 (1974) 207.

Tetracyclopentadienyluranium(IV), $(C_5H_5)_4U$

($I\bar{4}2m$) $Z = 2$, $R = 2.1\%$ from 700 reflections. A structural model based on a random mixture of a pair of enantiomorphic molecules in the crystal accounts for the space group symmetry, $I\bar{4}2m$, while individual molecules have point symmetry, S_4. In each molecule planar C_5 rings are in a regular tetrahedral array around a uranium atom and are *pentahapto*-bonded to it. C—C = 1.386(5), U—C(mean) = 2.807(1) Å.

J.H. Burns, *J. Organometal. Chem.*, 69 (1974) 225.

Tetracarbonyliodo[*O*-phenylenebis(dimethylarsino)]tungsten(II) triodide, $[W(CO)_4(diars)I][I_3]$.

($P2_1/m$) $Z = 2$, $R = 7.0\%$ for 1544 independent reflections. The cations have crystallographic *m* symmetry with the metal and iodine atoms on the mirror plane. The metal atom has a capped trigonal prismatic environment with the iodine atom in the unique capping position, W—I = 2.842(3) Å, two arsenic atoms, W—As = 2.636(2) Å, and two CO groups, W—CO = 1.988(5) Å in the remaining edge. The I_3^- anion is asymmetrical with dimensions 2.895(3), 2.957(4) Å, 178.6(1)°

M.G.B. Drew and J.D. Wilkins, *J. Organometal. Chem.*, 69 (1974) 271.

Two iron carbonyl complexes derived from barbaralone and the isobullvalene ring skeleton, characterization of a possible "Homobutadiene—Fe(CO)$_3$" complex, $C_9H_8OFe(CO)_3$, A and $C_{11}H_{10}OFe_2(CO)_6$, B

A. ($Pna2_1$) $Z = 4$, $R = 6.1\%$ based on 727 non-zero reflections. B. ($P2_1/c$) $Z = 4$, $R = 5.2\%$ based on 1632 non-zero reflections. A contains a "homobutadiene unit", alternative modes of bonding are discussed.
A.H.J. Wang, I.C. Paul and R. Aumann, *J. Organometal. Chem.* 69 (1974) 301.

Dicesium octafluoro-di-μ-oxodiarsenate, $Cs_2(As_2F_8O_2)$, A; $K_2(As_2F_8O_2)$, B and $Rb_2(As_2F_8O_2)$, C
A. ($P2_1/c$) $Z = 2$, $R = 8.7\%$ based on 875 reflections. B. ($P2_1/n$) $Z = 2$, $R = 9.0\%$ for 1208 reflections. C. ($P2_1/n$) $Z = 2$, $R = 6.9\%$ for 566 reflections. The three structures are isomorphic. The anion $(As_2F_8O_2)^{2-}$ is centrosymmetric-dimeric with the molecular point group D_{2h}(approx.). Characteristic of the ion is the very short As $\cdots$ As′ distance = 2.665(3) Å (mean value), and the large angle As—O—As′ = 95.1(8)°.
W. Haase, *Chem. Ber.* 107 (1974) 1009.

cis-Dichloro(4,7,13,16-tetraoxa-1,10-dithiacyclooctadecane)palladium(II) $PdCl_2.C_{12}H_{24}O_4S_2$
($P2_12_12_1$) $Z = 4$, $R = 3.6\%$ for 1356 significant reflections. A *cis*-square planar configuration is found around the palladium atom. Pd—Cl = 2.314(2), Pd—S = 2.303(2) Å. The discrete molecules are held together by hydrogen bonds.
B. Metz, D. Moras and R. Weiss, *J. Inorg. Nucl. Chem.*, 36 (1974) 785.

Uranyl chloride-bis-triethylammonium chloride, $UO_2Cl_2.2[(C_2H_5)_3NHCl]$
($I4_1/a$) $Z = 8$, $R = 15\%$ based on 1043 reflections. The uranium atom has octahedral coordination in the $UO_2Cl_4{}^{2-}$ anion, with four chlorine atoms at the corners of a rectangular plane, while the uranyl group $UO_2{}^{2+}$ is linear and almost perpendicular with regard to the rectangular plane, the symmetry approximates D_{4h}. The $(C_2H_5)_3NH$ chains are similar to those found in $(C_2H_5)_3NCl$. The cohesion in the molecule is assured by weak hydrogen bonds N $\cdots$ H $\cdots$ Cl of length 3.6(1) Å. Two of the four Cl atoms are linked to the nitrogen.
H. Brusset, N.-Quy-Dao and F. Haffner, *J. Inorg. Nucl. Chem.*, 36 (1974) 791.

Nonstoichiometric Uranium Sesquinitride, α-U_2N_3 in the range $UN_{1.40}$ to $UN_{1.76}$ H. Tagawa, and N. Masaki, *J. Inorg. Nucl. Chem.*, 36 (1974) 1099.