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X-RAY BIBLIOGRAPHY

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Dinitratobis (methyl-α-picolylamine)-copper (II)

$(P2_1/c)R$ = 7.9% for 986 reflections. The structure contains tetragonally distorted octahedrally coordinated copper ions. Cu–N is 2 03 Å Hydrogen positions are assigned and steric crowding discussed

N A Bailey and S J Bowler, *J Chem Soc A*, (1971) 1763

Potassium-μ-nitrido-bis (aquotetrachlororuthenate(IV), $K_3[Ru_2NCl_8(H_2O)_2]$

$(C2/m)R$ = 8 8% for 697 reflections The structure contains the nitrido-bridged ion $[Ru_2NCl_8(H_2O)_2]^{3-}$. Ru–N is 1 720 Å Water molecules are *trans* to nitrogen Ru–O is 2.18 Å The four chlorines about each ruthenium are bent away from the nitrogen and towards the water molecule

M. Ciechanowicz and A C. Skapski, *J Chem Soc A*, (1971) 1792

Ammonium-μ-nitrido-bis(aquo-tetrachlororuthenate(IV))

$(B2/m)R$ = 7 1% for 1054 reflections. The coordination about the two metal atoms is octahedral, the latter being joined by a centrosymmetric bridging nitrogen atom Ru=N = 1 725 Å, Ru–Cl = 2 370 Å and 2 391 Å Ru–O = 2 20 Å

R J P. Gee and H M Powell, *J Chem Soc. A*, (1971) 1795

Dicyanotris-(5-methyl-5*H*-dibenzophosphole)nickel(II) (A) and dicyanotris-(5-ethyl-5*H*-dibenzophosphole)nickel(II) (B)

All compounds contain 5-coordinate nickel

Compound A has two forms One form (Me4) has tetragonal-pyramidal nickel bonds with one dibenzophosphole bond at the apex Ni–P (axial) = 2 32 Å, Ni–P (basal) = 2 18 Å Another form (Me3) with trigonalbipyramidal bonds has one Ni–P (equatorial) = 2.29 Å and two equatorial = 2 22 and 2 23 Å.

A-Me4 $(P1)R$ = 4 4% for 3399 reflections, A-Me3 $(P2_1/b)R$ = 2.6% for 2853 independent reflections

Compound B is trigonalbipyramidal, similar to Me3

Et3, $(P2_1/b)R$ = 6 5% for 2484 independent reflections

H.M Powell, D J. Watkin and J.B Wilford, *J. Chem Soc A*, (1971) 1803

Di-μ-oxo-bis[oxo(L-cysteinato ethyl ester-*N*,*S*)-molybdenum(V)]
($P2_1$)R = 5.8% for 1053 reflections. Each molybdenum atom is in a similar trigonal-bipyramidal environment. Mo–S = 2.385(15) Å and Mo–N = 2.22(2) Å. Mo–Mo is 2.562(3) Å.
M. G. B. Drew and A. Kay, *J. Chem. Soc. A*, (1971) 1846

Di-μ-sulphido-bis[oxo-(L-cysteinato methyl ester-*N*,*S*) molybdenum(V)], $Mo_2S_2O_2(SO_2NC_4H_8)_2$
($P2_1$)R = 8.6% for 961 significant independent reflections. Both molybdenum atoms are in identical trigonal bipyramidal environments. Each is bonded to a sulphur (2.38(1) Å) and nitrogen (2.23(3) Å) of L-cysteine and to a terminal oxygen atom. Two sulphurs bridge the two molybdenum atoms. Mo–Mo = 2.804(4) Å.
M. G. B. Drew and A. Kay, *J. Chem. Soc. A*, (1971) 1851

Di-isothiocyanato-[*N*,*N*-bis(2-diethylaminoethyl)-2-methylthioethylamine-*N*,*N*,*N*] cobalt (II), $Co(NCS)_2(N_3S)$,1/2 BuOH where $N_3S = (Et_2NCH_2CH_2)_2NCH_2CH_2{\cdot}S{\cdot}Me$
($P2_1/c$)R = 6.9%. The metal atom is in a trigonal bipyramidal environment of five nitrogen atoms. Co–N(NCS) = 1.978(11) Å and Co–N (3 organic nitrogen) = 2.158(10), 2.149(10) and 2.244(9) Å. The sulphur of the ligand is not bonded to cobalt.
P. Dapporto and M. Di Vaira, *J. Chem. Soc. A*, (1971) 1891

Di-iodo[ethylenebis(oxyethylene)] bis(diphenylphosphine)nickel(II)
($P2_1/c$)R = 9.1% for 1654 independent reflections. This low spin nickel complex is intermediate between square planar and tetrahedral. The coordination to nickel is from two iodine and two phosphorus atoms while oxygen contacts of 3.20(7) and 3.16(6) Å to nickel stabilize the coordination.
P. Dapporto and L. Sacconi, *J. Chem. Soc. A*, (1971) 1914

trans-Bis[(chloroacetato)-(α-picoline)] copper(II)
($P\bar{1}$). The copper is square planar with Cu–N = 1.989(6) Å and Cu–O = 1.975(5) Å from picoline and acetate group respectively. There are two Cu–O contacts of 2.707(7) Å, from the chloroacetate groups, out of the plane.
G. Davey and F. S. Stephens, *J. Chem. Soc. A*, (1971) 1917.

Dichlorotris-(1,2-dimethylimidazole)copper(II)
(Pc_a2_1)R = 3.2% for 3018 independent reflections. In this monomeric complex the copper is trigonal bipyramidal. Two chlorine and one nitrogen atom are equatorial. Cu–N = 2.145 Å and Cu–N (axial) = 2.005 Å.
F. Huq and A. C. Skapski, *J. Chem. Soc. A*, (1971) 1927

2,2-Bipyridylamine copper(II) perchlorate

$(C2/c)R$ = 9 4% for 2078 independent reflections. The pyridine nitrogen atoms coordinate the copper in a pseudotetrahedral arrangement. N—Cu—N dihedral angle is 55 6°

J E. Johnson, T A Beineke and R A. Jacobson, *J. Chem Soc A*, (1971) 1371

1,4-Diphenyl-3-phenylamino-1,2,4-triazolium-tetrakis(isothiocyanato)-cobaltate(II)

$(C2/c)R$ = 3 5% for 1402 non-zero reflections The compound contains discrete $[C_{20}H_{17}N_4]^+$ and $[Co(NCS)_4]^{2-}$ ions

S. Cerrini, M. Colapietro, R Spagna and L Zambonelli, *J. Chem Soc A*, (1971) 1375

Decacarbonyl-μ-[1,2-bis(dimethylarsino) tetrafluoro-cyclobutene-*As,As*]-*triangulo*-triruthenium, $[(Me_2AsC{=}C(AsMe_2)CF_2\ CF_2)Ru_3(CO)_{10}]$

$(P2_12_12_1)R$ = 7 6% for 1828 observed reflections The compound is a derivative of $Ru_3(CO)_{12}$ in which one equatorial carbonyl on each ruthenium is replaced by an arsenic atom of the diarsine ligand. Ru—Ru = 2 831, 2 831 and 2 858 Å

P S Roberts and J. Trotter, *J Chem Soc A*, (1971) 1479

Chloro[dodeca(dimethylamino)-cyclohexaphosphazene-*N,N,N,N*]-copper(II) dichlorocuprate(I), $[N_6P_6(NMe_2)_{12}Cu^{II}Cl]^+Cu^ICl_2^-$

$(P2/n)R$ = 8 3% for 1103 observed reflections The cation has the copper(II) atom bonded to four nitrogen atoms of the phosphazene ring and to one chlorine atom in a distorted square pyramid arrangement P—N = 1.62 and 1 55 Å

W.C Marsh and J Trotter, *J Chem Soc A*, (1971) 1482

[1,2-Bis(dimethylarsino) hexafluoropropane] tetracarbonyl molybdenum, $Me_2AsCF(CF_3)CF_2\ Mo(CO)_4$

$(C2/c)R$ = 7 3% for 1510 observed reflections. The five-membered chelate ring is nonplanar, the As—C—C—As dihedral angle is 47° and the ligand has a staggered conformation with equatorial CF_3 groups.

P.J. Roberts and J Trotter, *J. Chem. Soc. A*, (1971) 1501.

[1,2-Bis(diphenylphosphino)hexafluorocylcopentene] dinitrosyliron, $Ph_2PC.(PPh_2)\cdot(CF_2)_2\cdot CF_2—Fe(NO)_2$

$(P2_1/n)R$ = 4 4% for 2492 observed reflections The iron atom is distorted tetrahedral. Fe—N = 1 661(7), 1 645(7) Å and N—O = 1 184(10) and 1.177(10) Å.

W Harrison and J Trotter, *J. Chem Soc. A*, (1971) 1542

Chloropentacarbonylmanganese, $ClMn(CO)_5$

$(Pnma)R$ = 6.1% for 653 non-zero reflections The molecules have C_s symmetry and deviations from C_{4v} are small.

P.T. Greene and R F Bryan, *J Chem. Soc A*, (1971) 1559.

trans-Diaquobis(pyridine-2-carboxamide)copper(II) chloride, $[Cu(H_2O)_2(C_5H_4N{\cdot}CO{\cdot}NH_2)_2]Cl_2$

($P2_1/c$) The ligand molecules are coordinated through the ring nitrogen and amide oxygen with the octahedron completed by two water molecules at a distance of 2 54 Å. The IR spectrum is discussed

D H Brown, D R MacSween, M Mercer and D,W A Sharp, *J Chem Soc A*, (1971) 1574.

(1-Phenylbutane-1,3-dionato)(ethylene glycol)-sodium

($P\bar{1}$)R = 5 9% for 1145 reflections The structure consists of a polymeric chain, parallel to the *a*-axis held at alternate centres of symmetry by Na$\langle^{O}_{O}\rangle$Na bridges

D Bright, G H.W. Milburn and M R Truter, *J Chem Soc A*, (1971) 1582

μ-[1,2-Bis(dimethylarsino)tetrafluorocyclobutene-*As,As*]di-μ-carbonyl-bis(dicarbonyl cobalt)(Co—Co), $Me_2AsC{=}C(AsMe_2)CF_2CF_2\ Co_2(CO_6)$

(*Pbca*)R = 5 8% for 1688 observed reflections The structure is similar to $Co_2(CO)_8$. The cobalt atoms are octahedrally coordinated with the bent metal bond occupying the sixth position. Co—Co is 2 483 Å

W Harrison and J Trotter, *J Chem Soc A*, (1971) 1607

[Bis(diphenylphosphino)methane] tetracarbonylmolybdenum

($P2_1/c$)R = 11 2% for 4438 independent reflections The molybdenum atom is octahedrally coordinated M—C = 1 93 Å *trans* to phosphorus M—C = 2 04 Å *trans* to carbon

K K Cheung, T F Lai and K S. Mok, *J Chem Soc A*, (1971) 1644.

Isothiocyanato[*N,N*-bis(2-diphenylphosphinoethyl)-2-methoxyethylamine-*N,O,P,P*]-nickel(II) hexafluorophosphate, $[Ni(bdma)NCS]PF_6$

($P2_1/c$)R = 8.6% for 2593 reflections The nickel atom is distorted square pyramidal with an apical Ni—O distance of 2 480(9) Å The donor atom set is NOP_2

A Bianchi and C A Ghilardi, *J Chem Soc A*, (1971) 1096.

$(Me_3Sn)_2N_2C$

($P6_422$)R = 8 0% for 342 unique observed reflections The structure consists of an infinite helical network of planar trimethyltin groups, linked with linear NCN units The nitrogen atoms complete a trigonal bipyramid about the tin. Sn—N = 2 47(1) Å, Sn—C = 2 14(2) Å

R.A. Forder, G M Sheldrick, *J Chem Soc A*, (1971) 1107

Hydridobis [1,2-bis(dimethylphosphino)ethane] naphthylruthenium(II) (A) and the osmium analogue (B)

The compounds are isostructural ($P2_1/c$)R = 8 0% (A) and 7 2% (B) The metal atom is approximately octahedrally coordinated with hydridic hydrogen *cis* to σ-bonded naphthyl group. Ru—H = 1.7 Å, Ru—C = 2 16 Å, Os—C = 2.13 Å Long M—P bonds are *trans* to hydrogen

U.A Gregory, S D Ibekwe, B T. Kilbourn and D R Russell, *J Chem. Soc A*, (1971) 1118.

Dichlorobis(4-ethylpyridine)-copper(II)

($P2_1/c$)R = 10 5% for 1130 observed reflections Cu—Cl = 2 28, Cu—N = 2.00 Å The ethyl groups are packed very loosely and quite differently from vinyl groups in the 4-vinylpyridine complex

M Laing and G Carr, *J Chem Soc A*, (1971) 1141

Disodium orthophosphite pentahydrate

($Pmn2_1$)R = 7.5% for 700 reflections Water molecules link adjacent tetrahedral phosphite ions P—O = 1 48 and 1 52 Å

R H Colton and D E Henn, *J Chem Soc A*, (1971) 1207

Dichloro(*O*-methyl-*N*-allylthiocarbamato)palladium(II), $Pd[C_3H_5\text{-}NH\,C(OMe)S]\,Cl_2$

($P2_12_12_1$)R = 9 8% for 984 unique observed reflections The palladium atom is approximately square planar with two chlorine atoms and the bidentate ligand. (S and double bond). Pd—S = 2 296(6), Pd—Cl = 2 340(10) Å

P Porta, *J Chem Soc A*, (1971) 1217

Primary nickel dithizonate, $Ni(Hdz)_2$

($P\bar{1}$)R = 12% for 2580 reflections The nickel atom is square planar, each dithizone ligand (N and S) making up a 5-membered chelate ring Ni—S = 2.19, Ni—N = 1 87 Å

M Laing, P Sommerville and P A Alsop, *J Chem Soc A*, (1971) 1247

Bis(triphenylphosphine)cadmium(II) chloride

($Pna2_1$)R = 9 8% for 2280 reflections The cadmium atom is tetrahedral as indicated previously by IR studies

A F Cameron, K P. Forrest and G Ferguson, *J. Chem Soc A*, (1971) 1286.

Tetrabutylammonium tetraiodotriargentate, $Bu_4N[Ag_3I_4]$

($P2_1/c$)R = 8 3%. A chain of AgI_4 tetrahedra sharing one edge is formed and linked to other chains by sharing the four free corners.

C J. Gilmore, P A. Tucker and P. Woodward, *J. Chem Soc A*, (1971) 1337.

Hydrotris(1-pyrazolyl)borato-benzenediazodicarbonyl molybdenum, $HB(pz)_3Mo(CO)_2\text{-}NNC_6H_5$

($P\bar{1}$)R = 3 8% for 2375 observed reflections The molybdenum is octahedrally coordinated. Mo—N(pyrazolyl) = 2 22 Å, Mo—N(diazo) = 1 83 Å, B—N = 1 55 Å The boron atom is in a slightly distorted tetrahedral environment.

G. Avitabile, P. Ganis and M Nemiroff, *Acta Crystallogr., Sect B*, 27 (1971) 725

Tetraphenylphosphonium trichloro(*cis*-but-2-en-1,4-diol)platinum(II), $(C_6H_5)_4P^+[PtCl_3(C_4H_8O_2)]^-$

($P2_1/c$) R = 8 5% for 1899 reflections The anion is a π complex, platinum is square planar

M. Colapietro and L. Zambonelli, *Acta Crystallogr , Sect. B,* 27 (1971) 734.

Potassium tetrametaphosphimate tetrahydrate, $K_4(PO_2NH)_4\ 4H_2O$ (A) and caesium tetrametaphosphimate hexahydrate $Cs_4(PO_2NH)_4 \cdot 6H_2O$ (B)

A, ($P2_1/c$)R = 4.0% for 1517 reflections B, ($P4_2/nmc$)R = 5 5% for 489 reflections. The interesting feature of the structures is the conformational isomerism of the anionic P–N ring derived from early crystal vibrational spectra

B. Berking and D. Mootz, *Acta Crystallogr., Sect. B,* 27 (1971) 740.

$Ca_2SiO_4\ CaCl_2$

($P2_1/c$)R = 11 0% The structure is almost a cubic closed packed arrangement of Cl^- and SiO_4^{4-} ions and is best represented by the formula $Ca_3(SiO_4)Cl_2$

R Czaya and G Bassert, *Acta Crystallogr , Sect B,* 27 (1971) 747

Tetramethylammonium cyclopentadienyl-phenyloxycarbene-dicarbonyl manganese, $[C_5H_5Mn(CO)_2(COC_6H_5)]^-[N(CH_3)_4]^+$

(*Ib*2*a*) R = 9.8% for 5292 intensities The carbene ligand is bonded to the manganese atom (1 96 Å) by a short single bond The sp^2 carbene carbon is bound to oxygen at 1.28 Å.

E. Hadicke and W. Hoppe. *Acta Crystallogr , Sect. B,* 27 (1971) 760

Potassium magnesium tetraperoxoniobate, $KMgNb(O_2)_4 \cdot 7H_2O$

($P2_1/c$) R = 3.5% for 4006 independent reflections The $Nb(O_2)_4^-$ ion has almost D_{2d} symmetry with a mean O–O distance of 1 50 Å

G Mathern and R Weiss, *Acta Crystallogr , Sect B,* 27 (1971) 1598.

Ammonium oxotrioxalatoniobate, $(NH_3)_4NbO(C_2O_4)_3H_2O$

($P\bar{1}$) R = 4.6% for 1122 independent reflections. The niobium is pentagonal bipyramidal with inter-ligand steric interaction causing a displacement of the niobium out of the equatorial plane

G. Mathern and R Weiss, *Acta Crystallogr , Sect B,* 27 (1971) 1610.

The 4-nitropyridine *N*-oxide adduct of *trans*-dichlorodiaquo copper(II), $CuCl_2(H_2O)_2 \cdot 2(C_5H_4N_2O_3)$

($P2_1/c$) R = 4.3% for 1270 unique reflections. *trans*-Square planar $CuCl_2(H_2O)_2$ groups have the amine oxide oxygen atom of the 4-nitropyridine *N*-oxide molecule loosely bonded to the copper ion forming a distorted tetragonal bipyramid around the copper. Cu–O = 2.635(2) Å.

R.J. Williams, D.T. Cramer and W.H. Watson, *Acta Crystallogr., Sect. B,* 27 (1971) 1619.

Diethylammonium tetracyanopalladate, $[(C_2H_5)_2NH_2]_2Pd(CN)_4$

($P\bar{1}$) (Solved in $C\bar{1}$) R = 5 5% for 1169 reflections. The $Pd(CN)_4^{2-}$ is square planar and the four CN ligands are bound to adjacent cations through N—H H type hydrogen bonds forming an infinite chain.

S Jerome-Leriette, *Acta Crystallogr*, *Sect B*, 27 (1971) 1624.

In_6Se_7

($P2_1$) R = 9% for 1430 independent reflections. The structure consists of two separate sections of almost cubic close packed arrays of Se atoms with In atoms in octahedral coordination.

J H C Hogg, *Acta Crystallogr*, *Sect B*, 27 (1971) 1630.

Bis(*H*-pyrrole-2-aldimine)copper(II), $(C_5H_5N_2)Cu(II)$

($P2_1/c$) R = 6 1% for 730 reflections The copper atom is coordinated to four nitrogen atoms in square planar array Cu—N = 1.97(1) and 1.95(1) Å

R Tewari and R.C Srivastava, *Acta Crystallogr*, *Sect B*, 27 (1971) 1644

4-Ethylpyridinium tetrabromoferrate(III)

($P2_1/c$) R = 6 1% for 762 observed reflections In the $FeBr_4^-$ tetrahedra the average Fe—Br distance is 2 326 Å The structure is stabilised by weak N—H—Br bonding.

M L Hackert and R A Jacobson, *Acta Crystallogr*, *Sect B*, 27 (1971) 1658

Ethylenebisguanidenenickel(II)chloride monohydrate, $C_6H_{16}N_{10}NiCl_2 \cdot H_2O$

($P2_1/c$) R = 3 1% for 2228 unique reflections The ligand is essentially planar around the nickel Average Ni—N is 1 866 Å. By extensive hydrogen bonding between water and chloride, the molecules are arranged into infinite sheets.

D L Ward, C N Caughlan and G D. Smith, *Acta Crystallogr*, *Sect B*, 27 (1971) 1541.

Ammonium diperoxodioxalatoniobate, $(NH_4)_3Nb(O_2)_2(C_2O_4)_2 \cdot H_2O$

($P2_1/c$) R = 3 5% for 1978 independent reflections. The coordination of the niobium is dodecahedral with peroxide groups in *cis* position. O—O mean is 1.48 Å in the peroxide groups

G. Mathern and R Weiss, *Acta Crystallogr*, *Sect. B*, 27 (1971) 1572.

Potassium triperoxo-(*o*-phenanthroline)niobate, $KNb(O_2)_3(C_{12}H_8N_2) \cdot 3H_2O$ (A) and $KNb(O_2)_3(C_{12}H_8N_2)\ 3H_2O\ H_2O_2$ (B)

A, ($P2_1/c$) R = 4 9% for 1640 independent reflections. B, ($P\bar{1}$) R = 4.7% for 2348 independent reflections. The $Nb(O_2)_3(C_{12}H_8N_2)^-$ anion has nearly C_s symmetry and the niobium is in dodecahedral coordination

G. Mathern and R. Weiss, *Acta Crystallogr*, *Sect. B*, 27 (1971) 1582.

Bis(*N,N*-diethyldithiocarbamato)tellurium(II), $[Te(S_2CNEt_2)_2]$

($P2_1/c$) R = 8.1% for 2012 observed reflections. Four sulphurs bond to tellurium at 2.519(3) Å average for two and 2 830(3) and 2 893(4) Å. Another sulphur is 3 579(5) Å from the tellurium.

C. Fabiani, R Spagna, A Vaciago and L Zambonelli, *Acta Crystallogr Sect B*, 27 (1971) 1499.

Bis(2-aminoethylsalicylideneiminato)chromium(III) iodide

($P\bar{1}$) R = 11 4% for 1481 observed reflections. The chromium is octahedrally coordinated by two tridentate ligands

A.P Gardner, B M Gatehouse and J C B White, *Acta Crystallogr , Sect B*, 27 (1971) 1505.

$Cs_3Cu_2Cl_7\ 2H_2O$

($P\bar{1}$) R = 12 3% for 692 independent reflections. Chlorine positions were verified by NMR measurements and hydrogen positions were also found by NMR

Von W. Vogt and H Haas, *Acta Crystallogr., Sect. B*, 27 (1971) 1528.

Mg_3TeO_6

($R\bar{3}$) R = 4 8% Tellurium is regular octahedral Te—O = 1.91 Å Magnesium is distorted octahedral, Mg—O = 2 02–2 2 Å.

H Schulz and G Bayer, *Acta Crystallogr Sect B*, 27 (1971) 815

K_5ThF_9

($C_{mc}2_1$) R = 1 8% for 1198 reflections. The coordination number of thorium is eight in an irregular dodecahedron with triangular faces Th—F average is 2 33 Å

R R. Ryan and R A Penneman, *Acta Crystallogr , Sect B*, 27 (1971) 829

$[(NH_3\ CH_2\ CH_2)_2NH_2]Cl(CuCl_4)$ at 20°C and 120°C

(*Pnma*) Thermochromism is observed both above and below room temperature The heavy atom positions are essentially unchanged at the two temperature extremes.

B Zaslow, *Acta Crystallogr , Sect B*, 27 (1971) 849

(The following entries were received November 23rd, 1971)

Chlorotris(acetylacetonato)zirconium(IV), $Zr(acac)_3Cl$

($Pna2_1$) R = 10 4% for 1499 independent reflections. The molecule is monomeric and six acetylacetonate oxygens are bonded to Zr^{IV} at the corners of a distorted pentagonal bipyramid Chlorine occupies one axial position and an acac group spans the other axial position and one equatorial position.

R B. VanDreele, J.J Stezowski and R C. Fay, *J Amer. Chem. Soc*, 93 (1971) 2887

Dicarbonyl-3-[π-(2-cyclohexadienyl)]-σ-propenoyliron

($P2_1/c$) R = 5.2% for 1836 reflections An asymmetrically substituted π-cyclohexadienyl ligand is bonded to iron by a propenoyl side chain resulting in considerable intermolecular strain.

P.J VanVuuren, R J Fletterich, J Meïnwald and R E Hughes, *J Amer. Chem Soc*, 93 (1971) 4394.

Cobalt(III) and cobalt(II) complexes of the cage ligand, 1,8-bis(fluoroboro)-2,7,9,14,15, 20-hexaoxa-3,6,10,13,16,19-hexaaza-4,5,11,12,17,18-hexamethylbicyclo-[6 6 6]-eicosa-3,5,10, 12,16,18-hexaene, $CoLBF_4$ and CoL where L = $C_{12}H_{18}N_6O_5B_2F_2^{2-}$

$CoLBF_4$, ($Fdd2$) R = 3.7% for 1582 reflections Coordination is slightly distorted D_3, between octahedral and trigonal prismatic.

CoL, ($R\bar{3}c$) R = 4 3% for 2107 reflections Coordination is D_3 trigonal prismatic Bond distances and angles within the ligand are the same in both compounds.

G A Zakrzewski, C A Ghilardi and E C Lingafelter, *J Amer Chem Soc*, 93 (1971) 4411

(Cyano(dicyanomethyl)ketenimınato)carbonyl-(tetracyanoethylene)bis(triphenylphosphine) ındium, $Ir(C_6N_4H)(CO)[(CN)_2C{=}C(CN)_2][P(C_6H_5)_3]_2.1/2\,C_6H_6$

($P2_1/c$) R = 4 1% for 3853 reflections The stereochemistry is trigonal bipyramidal with two phosphorus atoms and π-TCNE in the equatorial plane Carbonyl and ketenimınato groups occupy axial positions. Ir—P is 2 392(7), Ir—C is 2 166(15) and Ir—N is 2 204(8) Å.

J S. Ricci and J A. Ibers, *J Amer. Chem Soc*, 93 (1971) 2391

The iron—antimony cluster complex $\{[Fe(h^5\text{-}C_5H_5)(CO)_2]_3SbCl\}_2[FeCl_4]\cdot CH_2Cl_2$

($I2/c$) R = 6 4% for 2040 independent reflections In the cation the one chlorine ligand and three $[Fe(h^5\text{-}C_5H_5)(CO_2)_2]$ ligands are in a distorted tetrahedral arrangement around a central antimony Fe—Sb = 2 54 Å, Sb—Cl = 2.401(4) Å

Trinh-Toan and L.F Dahl, *J Amer Chem Soc.*, 93 (1971) 2654

Tetra(cyclopentadienyl)titanium, $(C_5H_5)_4Ti$

($P6_122$) R = 3.6% for 1205 reflections The molecule contains pentahapto and two monohapto-cyclopentadienyl rings The metal has the 16 electron configuration

J.L Calderon, F A. Cotton, B G. DeBoer and J Takats, *J Amer. Chem Soc.*, 93 (1971) 3592

Bis(dithiotropolonato)nickel(II), $Ni(S_2C_7H_5)_2$

($P2_1/c$) R = 2.4% for 976 independent reflections The coordination geometry is planar with the complex having a centre of symmetry Ni—S, 2.147(3) Å

G P. Khare, A J Schultz and R. Eisenberg, *J. Amer. Chem Soc*, 93 (1971) 3597

$[(C_2H_5)_4N]_2[W_2Ni_3(CO)_{16}]$, $[PPN]_2[Mo_2Ni_3(CO)_{16}]$, $[PPN]_2[W_2Ni_3(CO)_{16}]$ ($C2/c$), ($P\bar{1}$), ($P\bar{1}$), respectively. The study reveals a trigonal bipyramidal arrangement of bonding transition metal atoms The configuration of the $[M_2Ni_3(CO)_{16}]^{2-}$ anion can be described as a planar $Ni_3(CO)_6$ residue of $D3h$—$\bar{6}2m$ geometry symmetrically coordinated to two identical $M(CO)_5$ groups by Ni—M interactions

J R Ruff, R.P White and L F. Dahl, *J. Amer Chem Soc*, 93 (1971) 2159

Triammonium-bis(oxalato)dioxovanadate(V) dihydrate

($P2_12_12_1$) R = 4 1% for 2566 reflections The vanadium is in octahedral coordination with two V—O bonds of 2 185 and 2 235 Å to carboxylate oxygens *trans* to oxo-oxygens. The other two carboxylate oxygens are at 1 972 and 1.988 Å from vanadium

W R Scheidt, Chun-che Tsai and J L Hoard, *J Amer Chem Soc*, 93 (1971) 3867.

Ammonium(dihydrogen ethylenediaminetetraacetato)dioxovanadate(V) trihydrate

($P2_1/c$) R = 5 2% for 3975 reflections The VO_2 is *cis* and V—N (2 35 and 2 36 Å) bonds are *trans* to oxo oxygen atoms Two carboxylate oxygens complete the octahedron.

W R Scheidt, D M Collins and J L Hoard, *J Amer Chem Soc*, 93 (1971) 3873

Trisodium(ethylenediaminetetraacetato)dioxovanadate(V) tetrahydrate

($P2_1/c$) R = 4 8% for 6266 reflections The vanadium is octahedral V—O (carboxylate) = 2 0 Å and *trans* to oxo ligands V—N is 2 36 Å

W R Scheidt, R Countryman and J L Hoard, *J Amer Chem Soc*, 93 (1971) 3878

Bis(cyclohexylbenzene)silver(I) perchlorate, $(C_6H_{11}C_6H_5)_2AgClO_4$

(*Pmcn*) R = 6 9% for 981 non-zero reflections. The structure is composed of alternate layers of $AgClO_4$ and hydrocarbon. The silver is tri-coordinate with one bond from each of the two aromatic rings and the third from a perchlorate oxygen.

E A Hall Griffith and E L Amma, *J. Amer Chem Soc*, 93 (1971) 3167.

Bis(triphenylphosphine)tetrakis(dimethylglyoximato)dirhodium, $Rh_2(C_4H_7N_2O_2)_4 \cdot [(C_6H_5)_3P]_2 \cdot H_2O \cdot C_3H_7OH$

($P2_1/c$) R = 6 8% for 3307 reflections The dinuclear molecule consists of two equivalent halves linked by Rh—Rh of 2.936 Å. The P—Rh—Rh—P chain is almost linear The two $Rh(C_4H_7N_2O_2)_2$ groups have mean planes perpendicular to the axis and also have an eclipsed rotational relationship

K G. Caulton and F.A. Cotton, *J. Amer Chem. Soc*, 93 (1971) 1914.

Rubidium dichromate ($C2/c$)

($C2/c$) R = 8 3% for 445 independent reflections The compound is isostructural with ammonium dichromate. Cr—O (terminal) is 1.609—1.627(14) Å, Cr—O (bridging) is 1 774 (10) Å

P. Löfgren and K. Waltersson, *Acta. Chem. Scand.*, 25 (1971) 35.

Rubidium dichromate ($P2_1/n$)

($P2_1/n$) R = 8.5% The structure contains $Cr_2O_7^{2-}$ units in which two CrO_4 tetrahedra share one corner Cr—O (terminal) is 1 580—1 617(18) Å, (bridging) = 1 795 and 1.796 (14) Å

P. Lofgren, *Acta Chem Scand*, 25 (1971) 44

Nb_5P_3

(*Pnma*) R = 11 4% for 17 01 reflections The phosphorus atoms are situated in triangular prisms of niobium atoms with one to three niobium atoms outside the quadrilateral faces.

E. Hassler, *Acta. Chem Scand*, 25 (1971) 129

Silver(I)dipropylmonothiocarbamate hexamer, $(C_3H_7)_2NCOSAg$

($P2_1/a$) R = 10 8% for 1091 independent reflections The silver atoms form an almost regular octahedron with each having three-fold non-planar coordination The metal—metal distances are slightly larger than in metallic silver.

P Jennische and R Hesse, *Acta Chem Scand*, 25 (1971) 423

Tri-aquo-imino-diacetato-neodymium(III) chloride, $Nd[OCOCH_2NHCH_2OCO)Cl\ 3H_2O$

($P2_12_12_1$) R = 8 7% The neodymium is coordinated in a distorted tricapped trigonal prism by eight oxygen atoms and one nitrogen Nd—O is 2 39 or 2 79 Å The iminodiacetate groups bridge between neodymiums.

A Oskarsson, *Acta Chem. Scand*, 25 (1971) 1207

Magnesium orthovanadate, $Mg_3(VO_4)_2$

(*Cmca*)R = 4.7% for 1697 reflections. Nearly cubic close packed oxygen atoms layers are almost parallel to $[01\bar{1}]$ direction with magnesium ions in two types of octahedral sites and the vanadium in a tetrahedral site. Mg—O = 2 092 and 2 098 Å V—O = 1 70 Å

N Krishnamachari and C Calvo, *Can. J Chem*, 49 (1971) 1623.

$[Rh(f_6fos)_2]^+[cis\text{-}(CO)_2RhCl_2]^-$ where ($f_6$fos) = 1,2-bis(diphenylphosphino)hexafluorocyclopentene

($I4_1/a$) R = 8 8% for 2934 reflections Each ion is nearly square planar and has a crystallographic two-fold axis passing through the rhodium atom. Rh—P = 2.282(6), 2 300(6) Å.

F.W B. Einstein and C R S M Hampton, *Can J Chem*, 49 (1971) 1901.

α-$Zn_3(VO_4)_2$

(*Cmca*) R = 8.9% for 726 unique reflections The Zn ions occupy two types of octahedral sites while the V ions lie in sites showing tetrahedral coordination to oxygen. V—O = 1.71 Å and 1 78 Å. Zn—O = 2.13 and 2.10 Å.

R. Gopal and C. Calvo, *Can. J. Chem*, 49 (1971) 3056

Tris(*N,N*-di-*n*-butyldiselenocarbamato)nickel(IV) bromide, $Ni(Se_2CN\text{-}(C_4H_9)_2)_3Br$ (twinned crystals)

$P\bar{3}1c$ R = 10 0% for 307 unique reflections. The quadrivalent nickel atom is octahedrally coordinated by six selenium atoms Ni–Se = 2 391(5) Å Se–Se = 3 00(1) Å (intraligand) and 3 51(1), 3 52(1) and 3 55(1) Å (interligand)

P T Beurskens and J A Cras, *J Crystallogr Mol Struct*, 1 (1971) 63.

cis-Dicarbonyltetraphosphinechromium(0), $[(CO)_2(PH_3)_4Cr]$

($C2/c$) R = 7 8% for 1030 independent reflections The chromium atoms lie in rotation diads, the molecules having $2(C_2)$ symmetry Cr–P (*trans* to CO) = 2 338(4), Cr–P (*trans* to PH_3) = 2.282(4) Å

G Huttner and O Schelle, *J Crystallogr Mol Struct*, 1 (1971) 69.

2,2'-bipyridine, pyridine tricarbonyl molybdenum(III), $[(C_{10}H_8N_2)(C_5H_5N)(CO)_3Mo]$

(*Pnam*) R = 6 3% for 1394 independent reflections The molecule is octahedral with bipyridine and pyridine forming one triangle and the three carbonyl group forming the opposite triangle

A Griffiths, *J Crystallogr Mol Struct*, 1 (1971) 75

Tris(2,2,6,6-tetramethyl-3,5-heptanedionato)aquodysprosium(III). $Py(thd)_3H_2O$

($P\bar{1}$) R = 5.8% for 3015 intensities The oxygen polyhedron around dyprosium has seven-coordinate geometry, the seventh ligand being water

C S. Erasmus and J C A Boeyens, *J Crystallogr Mol Struct*, 1 (1971) 83

Diiodo-*N,N,N',N'*-tetramethylthiuramdisulphide mercury(II), $HgI_2(S_2CN(CH_3)_2)_2$

($P2_1/c$) R = 4 4% for 1294 reflections The mercury is in a distorted tetrahedral coordination with two iodines and two sulphurs Hg–I = 2 654(2), 2 661(2), Hg–S = 2 651(7), 2 882(7) Å.

P T Beurskens, J A Cras, J H Noordik and A M Spruijt, *J Crystallogr Mol Struct.*, 1 (1971) 93.

Orthorhombic and monoclinic isomers of catena-di-μ-chloro-semicarbazidecopper(II), $[Cu(Cl)_2(NH_2\cdot NH\cdot CO\cdot NH_2)]$

($Pna2_1$) R = 8 6%, 572 reflections (A), and ($P2_1/c$) R = 8.2%, 832 reflections (B). In both complexes the copper is octahedral. In B, the two semicarbazide molecules have oxygen and nitrogen *trans* In A they are *cis* to each other.

A Chiesivilla, A G. Manfrepotti, M. Nardelli and G. Pelizzi, *J Crystallogr Mol. Struct*, 1 (1971) 245

Bis(*N*,*N*-diethyldithiocarbamato)palladium(II), $Pd[S_2CN(C_2H_5)_2]_2$

($P4_2/n$) R = 3 8% for 600 reflections. The crystals are isomorphous with the Pt^{II} compound. Palladium is in planar coordination to four sulphur atoms. Pd—S = 2 317(3) and 2 315(3) Å

P T Buerkens, J A Cras, Th.W Hummelink and J.H. Noordik, *J Crystallogr Mol Struct* 1 (1971) 253.

Bis(2,2,6,6-tetramethylheptane-5-thiolo-3-onato)nickel(II)

($P2_1/c$) R = 10 5% for 2109 reflections The nickel shows a slightly tetrahedrally distorted planar coordination to two *cis* oxygens and two sulphur atoms.

J Coetzer and J C A. Boeyens, *J Crystallogr Mol Struct*, 1 (1971) 277

Tetrakis(*N*',*N*'-diallylthiourea)nickel(II) iodide, $[Ni(C_7H_{12}N_2S)_4I_2]$

($P4/n$) R = 8.8% for 931 independent reflections The nickel is in a flattened pyramid involving four sulphur atoms from four diallylthiourea molecules Ni—S is 2.221 Å

A Chiesi, A Mangia, M Nardelli and G. Pelizzi, *J Crystallogr Mol Struct*, 1 (1971) 285

Di-μ-chloro(dicyandiamide)cadmium(II)

($P2_1/n$) R = 3 7% for 1711 independent reflections The cadmium is surrounded by four chlorines and two nitrogen atoms in a distorted octahedron Cd—Cl = 2 572, 2 607, 2 630, 2 647 Å Cd—N = 2 358 and 2 430 (guanidinic)

A C. Villa, L Coghi, A Mangia, M Nardelli and G Pellizi, *J Crystallogr Mol Struct*, 1 (1971) 291

Tris(1,1,1,2,2,3,3-heptafluoro-7,7-dimethyl-4,6-octanedionato)aquolutetium(III), $Lu(fod)_3 \cdot H_2O$

($P\bar{1}$) R = 12 8% Two crystallographically independent formula units are similar and are dimerised through water molecules The coordination of lutetium is seven-fold in a mono-capped trigonal prism

J C A Boeyens and J P.R deVilliers, *J Crystallogr Mol Struct*, 1 (1971) 297

Tricobalt(II)-dihydroxysulphate-dihydrate, $Co_3(OH)_3(SO_4)_2 \cdot 2H_2O$

(*Pbcm*) Infinite chains of Co—O octahedra share one edge and are linked by alternating SO_4 tetrahedral and additional Co—O octahedra. There is no similarity to the well known layer structures of most hydroxide salts of divalent metals

E Dubler and H R Oswald, *Helv Chim Acta*, 54 (1971) 1621

The antibiotic monensin — the free acid and metal complex

The crystal structure of the crystalline monohydrate was determined and the relative differences in hydrogen bonding pattern in the silver complex are given (Monensin reverses the translocation of alkali metals across mitrochondrial membranes)

W K Lutz, F K Winkler and J D Dunitz, *Helv Chim Acta*, 54 (1971) 1103

Dichromium tetraacetate dihydrate (A) and dirhodium tetraacetate (B)

($C2/c$) R = 2.7% for 1573 reflections (A) and ($C2/c$) R = 3.4% for 1313 reflections (B) The coordination around the metal in both compounds is octahedral with multiple metal–metal bonding Rh–Rh is 2 3855(5) and Cr–Cr is 2 362(1) in the carboxylate-bridged dimers

F A Cotton, B G Deboer, M D LaPrade, J R. Pipal and D A Ucko, *Acta Crystallogr., Sect B*, 27 (1971) 1664

Na_2MnCl_4

($Pbam$) R = 10.4% for 182 CuKα and 107 MoKα reflections. Mn ions are surrounded octahedrally by chloride ions with octahedra sharing opposite edges to form infinite chains These are held together by Na^+ ions, each of which is surrounded by six chlorine ions at the corners of a trigonal prism

J Goodyear, S A.D. Alli and G A Steigmann, *Acta Crystallogr., Sect B*, 27 (1971) 1672

Bis(L-prolinato)palladium(II)

($B22_12$) R = 8 5% for 694 reflections. The palladium is square planar and the chelate ring is bent at the two coordinating atoms. Bond lengths are normal. Pd–O = 2 02 Å, Pd–N = 2 03 Å

F. Marumo and Y Saito, *Acta Crystallogr., Sect B*, 27 (1971) 1062

$SnHPO_4$

($P2_1/c$) R = 10 5% The structure contains pairs of hydrogen bonded HPO_4^{2-} ions in sheets with the two atoms lying midway between

A F Berndt and R Lamberg, *Acta Crystallogr., Sect B*, 27 (1971) 1092

Bis(*o,o'*-diethyl-dithiophosphato)-1,10-phenanthrolinenickel(II)

($C2/c$) R = 5.9% for 2605 reflections. The unit cell contains the *d* and *l* isomers, each of which are in *cis* configuration

D.C Craig, E T Pallister and N C Stephenson, *Acta Crystallogr., Sect B*, 27 (1971) 1163.

Copper complex of *o*-hydroxyacetophenone imine, $Cu(C_8H_8NO)_2$

($P\bar{1}$) R = 3 7% for 5170 independent reflections Two independent molecules have similar bond distances but different distortions The copper is square planar Cu–N is 1 922(2) Å, Cu–O is 1 880(2) Å.

G. Marongiu and E C Lingafelter, *Acta Crystallogr., Sect B*, 27 (1971) 1195

1,2-Bis(2-methylpyridinium)ethane tetracyanonickelate trihydrate

(*Pnnm*) R = 13 3% for 1612 independent reflections. The methyl groups in position 2 of the pyridine rings are arranged *cis* to one another

L D.C Bok, S S. Basson, J.G. Leipoldt and G.F S. Wessels, *Acta Crystallogr., Sect. B*, (1971) 1233

(+)-*cis*-Dichloro[(S)-1-butene] [(S)-α-Methyl-benzylamine] platinum(II)
($P2_12_12_1$) R = 4 2% for 1108 reflections The platinum is square planar with the C=C bond perpendicular to the plane. The absolute configurations of two asymmetric centres in the molecule have been determined
C. Pedone and E Benedetti, *J Organometal. Chem*, 29 (1971) 443.

(Dithioformato)bis(triphenylphosphine)dicarbonyl rhenium, $Re(HCS_2)(CO)_2[P(C_6H_5)_3]_2$
($P\bar{1}$) R = 3.9% for 2682 reflections The metal is in a distorted octahedral coordination and the structure is monomeric. Re–S = 2.500(3), 2 532(5) Å, C–S = 1 64(2), 1 68(2) Å, Re–C = 1.91 Å.
V.G. Albano, P L. Bellon and G. Ciani, *J Organometal Chem*, 31 (1971) 75.

(1,1′-Trimethylenedicyclopentadienyl)titanium dichloride
($P2_1/n$) R = 2 9% for 1668 reflections. The titanium atom is in a distorted tetrahedron of two chlorine atoms and centroids of π-cp rings of the ligand Ti–πcp = 2 061, 2 060 Å, Ti–C = 2.407 – 2 360 Å.
B R. Davies and I. Bernal, *J. Organometal Chem*, 30 (1971) 75

Two diastereoisomeric complexes of *cis*-dichloro(1,5-hexadiene)platinum(II) with (S)α-methylbenzylamine
Both ($P2_12_12_1$) and R = 6 2% and 7.4% for 1454 and 1132 reflections. The platinum is square planar in both cases The absolute configurations of the three asymmetric centres in each molecule have been determined
C Pedone and E. Benedetti, *J. Organometal. Chem*, 31 (1971) 403.

Catena-di-μ- fluorodifluorodiaquohafnium(IV) monohydrate, $HfF_4 \cdot 3H_2O$ (two dimorphic forms)
The basic polyhedral unit is found to have stoichiometry $HfF_4(H_2O)_2$. The coordination number of the metal is eight with distorted dodecahedral symmetry
D Hall, C E.F Rickard and T N Waters, *J Inorg. Nucl. Chem*, 33 (1971) 2395

Tris(μ-acetato-μ-acetonimato)palladium(II) 0.5 benzene, $\{Pd(OAc)(ONCMe_2)\}_3 \cdot 1/2C_6H_6$
($P\bar{1}$) R = 13 0% for 1838 reflections Each pair of square planar palladiums is bridged by acetate and acetonime. Pd–Pd is 3.009(2) Å.
A. Mawby and G.E. Pringle, *J Inorg Nucl Chem.*, 33 (1971) 1989.

Bis(diaminomaleonitrile)dichloropalladium
($P2_1/a$) R = 10 6 for 732 independent reflections. The complex is square planar with monodentate DMN ligands coordinating through one amine group
M.G Miles, M.B. Hursthouse and A G. Robinson, *J Inorg Nucl. Chem*, 33 (1971) 2015

Tetraethylammonium octathiocyanato-*N*-uranate(IV), $[(C_2H_5)N]_4[U(NCS)_8]$
(*I4/mmm*) R = 6 29% for 771 independent reflections. The eight N-bonded ligands are dispersed at the vertices of a cube
R Countryman and W.A. McDonald, *J. Inorg Nucl Chem.*, 33 (1971) 2213.

Rb_2UF_6
(*Cmcm*) R = 8 9% for 99 independent reflections The structure consists of infinite chains of UF_8 polyhedra which are dodecahedral with triangular faces.
F.H. Kruse, *J Inorg Nucl Chem*, 33 (1971) 1625

Bis(amidoxalato-*O*-*O*)-zinc dihydrate, $Zn(OOC{-}CO{-}NH_2)_2 2H_2O$
($P\bar{1}$) R = 3 8% for 860 independent reflections The octahedral coordination sphere of zinc is made up of oxygen atoms, one carboxylic, one amidic, and apical water oxygens
A Braibanti, M A Pellinghelli A, Tiripicchio and M T Camelli, *Acta Crystallogr*, *Sect B*, 27 (1971) 1240

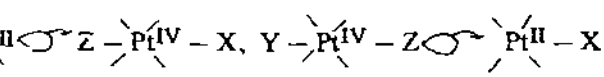
$K_2VO_2F_3$
(*Pnma*) R = 2 6% for 668 observed reflections. Octahedrally coordinated vanadium atoms are linked by *cis* bridging fluorine atoms into infinite chains The structure is an example of a non-linear dioxovanadium(V) group.
R R Ryan, S H. Mastin and M.J. Reisfield, *Acta Crystallogr.*, *Sect B*, 27 (1971) 1270

Bis(2,4-pentanedionato)cyclohexylaminecobalt(II)
($P2_1/c$) R = 5 7% for 1413 reflections The centrosymmetric dimer consists of two octahedra sharing an edge The two chelate rings coordinated to cobalt are not coplanar
J A Bertrand and A R. Kalyanaramam, *Inorg Chem. Acta*, 5 (1971) 167.

Errata

Coordination Chemistry Reviews, Vol 7, No 3 (January, 1972)

p. 248, eqn (13) should read



(13)

p 314, eqn. (17) should read

$$[Mn(Hpor)(H_2O)_2]^+ \rightleftharpoons [Mn(Hpor)(OH)_2] + 2H^+ \qquad (17)$$