

X-RAY BIBLIOGRAPHY

M. KEETON

217, Bath Rd., Kingston, Ontario (Canada)

(Received November 14th, 1972)

Trinitratobis(bipyridyl)lanthanum(III), $\text{La}(\text{NO}_3)_3(\text{C}_{10}\text{H}_8\text{N}_2)$

(*Pbcn*) $Z = 4$, $R = 6.0\%$ for 1155 reflections. The ten-coordinate lanthanum atom is chelated by two bipyridyl ligands and three bidentate nitrate groups. $\text{La}-\text{N} = 2.66(1)$, $\text{La}-\text{O}(\text{average}) = 2.59(1)$ Å

A.R. Al-Karaghoulh and J.S. Wood, *Inorg. Chem.*, 11 (1972) 2293.

Crystalline $[\{\text{FB}(\text{ONCHC}_5\text{H}_3\text{N})_3\text{P}\}\text{Fe}^{\text{II}+}][\text{BF}_4^-] \cdot \text{CH}_2\text{Cl}_2$

(*C2/c*) $Z = 8$, $R = 7.50\%$ for 2181 independent reflections. The central iron(II) cation is coordinated to six nitrogen atoms with $\text{Fe}-\text{N}(\text{aldimino}) = 1.921(7)-1.943(8)$; $\text{Fe}-\text{N}(\text{pyridyl}) = 1.972(7)-1.984(8)$ Å. The overall geometry about iron is intermediate between trigonal prismatic and octahedral.

M.R. Churchill and A.H. Reis, Jr., *Inorg. Chem.*, 11 (1972) 2299.

Bis(triphenylphosphine)bis(π -allyl) ruthenium, $(\text{C}_3\text{H}_5)_2\text{Ru}[\text{P}(\text{C}_6\text{H}_5)_3]_2$

(*P1*) $Z = 2$, $R = 4.7\%$ for 2349 reflections. $\text{Ru}-\text{C}(\text{centre allyl C}) = 2.128(16)$, $\text{Ru}-\text{C}(\text{end allyl C's}) = 2.239(16)$ Å. The bond angles between the central allyl carbons and the Ru and P atoms are all close to tetrahedral.

A.E. Smith, *Inorg. Chem.*, 11 (1972) 2306

Triclinic (A) and tetragonal (B) forms of bis(*N-tert.*-butylpyrrole-2-carbaldimino)copper(II)

(*P1*) $Z = 2$, $R = 6.9\%$ for 1089 reflections (A) and (*P4₁2₁2₁*) $Z = 4$, $R = 8.3\%$ for 504 reflections (B). The two molecular structures are similar with distorted tetrahedral copper. $\text{M}-\text{N}(\text{pyrrole}) = 2.054(8)$ (A) and $2.044(14)$ Å (B). $\text{M}-\text{N}(\text{azomethine}) = 1.939(8)$ (A) and $1.922(15)$ Å (B)

C.H. Wei, *Inorg. Chem.*, 11 (1972) 2315.

Ammonium pentafluoroantimonate(III), $(\text{NH}_4)_2\text{SbF}_5$

(*Cmcm*) $Z = 4$, $R = 2.4\%$ for 505 observed reflections. Isolated SbF_5^{2-} ions approximate a square pyramid. Apical $\text{Sb}-\text{F} = 1.916(4)$, average basal distance $\text{Sb}-\text{F} = 2.075(2)$ Å.

R.R. Ryan and D.T. Cromer, *Inorg. Chem.*, 11 (1972) 2322.

{[2-{[2(Diethylamino)ethyl]amino}ethyl]diphenylphosphine oxide}-
di-isothiocyanatocobalt(II)

($P2_1c$) $Z = 4$, $R = 9.5\%$ for 1258 reflections. The structure consists of distorted trigonal bipyramids with two nitrogen and one oxygen atom in the equatorial plane and apical nitrogens.

C.A. Ghilardi and A.B. Orlandini, *J. Chem. Soc. Dalton*, (1972) 1698.

Isothiocyanato-{ N,N -bis[2-(diethylamino)ethyl]-2-(diphenylarsino)ethylamine-
 N,N,N }-nickel(II) tetraphenylborate

($P\bar{1}$) $R = 8.0\%$, $Z = 2$, for 4097 reflections. The nickel is square planar coordinated.

Ni—N bond distances are compared with those in a related five-coordinate complex.

M. Di Vaira and A.B. Orlandini, *J. Chem. Soc. Dalton*, (1972) 1704.

cis-Di- μ -carbonyl-bis[carbonyl-(π -cyclohexa-1,3-diene)cobalt]

($Cmcm$) $R = 9.1\%$, $Z = 4$ for 690 unique reflections. The dimer has an almost planar $\text{Co}(\text{CO})_2\text{Co}$ bridging system. $\text{Co}\cdots\text{Co} = 2.559 \text{ \AA}$. The cyclohexadiene ligands bond to the cobalt atoms via butadiene fragments. $\text{Co}-\text{C} = 2.04$ and 2.13 \AA .

F.S. Stephens, *J. Chem. Soc. Dalton*, (1972) 1752.

Di- μ -carbonyl-tetracarbonyl-(π -norbornadiene)dnicobalt

($P\bar{1}$) $Z = 2$, $R = 4.9\%$ for 2996 unique reflections. The molecule has approximate m symmetry, $\text{Co}\cdots\text{Co} = 2.531(1) \text{ \AA}$. The norbornadiene ligand bonds to one cobalt atom via the $\text{C}=\text{C}$, which is $1.373(3) \text{ \AA}$. The $\text{Co}(\text{CO})_2\text{Co}$ bridge is non-planar; 135° is the angle between the $\text{Co}(\text{CO})\text{Co}$ planes and $\text{Co}-\text{C}(\text{norbornadiene}-\text{cobalt}) = 1.871(3)$ and $\text{Co}-\text{C} = 1.987(3) \text{ \AA}$.

F.S. Stephens, *J. Chem. Soc. Dalton*, (1972) 1754.

Trichloro-[N,N -bis(6-methyl-2-pyridylmethyl)methylamine- N,N,N] titanium(III)

($Pna2_1$) $Z = 4$, $R = 7.7\%$ for 772 independent reflections. The titanium is in a distorted octahedral environment with three Ti—Cl bonds of $2.292(7)$, $2.364(6)$ and $2.418(7) \text{ \AA}$.

Ti—N(*tert*) = 2.212 and Ti—N(pyridine) = $2.269(14)$ and $2.243(16) \text{ \AA}$.

R.K. Collins, M.G.B. Drew and J. Rodgers, *J. Chem. Soc. Dalton*, (1972) 899.

5, 7, 7, 12, 12, 14-hexamethyl-1, 4, 8, 11-tetra-azacyclotetradeca-4, 8, 10, 14-tetraenenickel(II)
perchlorate

($Pbcn$) $Z = 4$, $R = 12\%$ for 1229 independent reflections. The nickel atom lies in the plane of the four nitrogens which deviate $\pm 0.18 \text{ \AA}$ from the plane. Bond angles and lengths are discussed in terms of the amount of strain present in the molecule.

I.E. Maxwell and M.F. Bailey, *J. Chem. Soc. Dalton*, (1972) 935.

rac-5, 7, 7, 12, 14, 14-Hexamethyl-1, 4, 8, 11-tetra-azacyclo-tetradeca-4, 11-diene nickel(II) perchlorate

(*Pbca*) $Z = 8$, $R = 12\%$ for 2048 reflections. The nickel and four nitrogen atoms deviate slightly from square planar coordination and deviations of other angles and bond lengths from those expected are evidence of intramolecular strain.

M.F. Bailey and I.E. Maxwell, *J. Chem. Soc. Dalton*, (1972) 938.

Cadmium diacetate dihydrate

($P2_1 2_1 2_1$) $Z = 4$, $R = 2.8\%$ for 1292 observed reflections. The coordination geometry is that of a distorted square-base trigonal cap with cadmium coordination to seven oxygens. The acetates are bridging with one oxygen of one acetate group forming a continuous cadmium-oxygen spiral. $\text{Cd}-\text{O} = 2.294-2.597 \text{ \AA}$.

W. Harrison and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 956.

Caesium tetradecahydrononaborate (1-), $\text{CsB}_9\text{H}_{14}$

($P\bar{1}$) $Z = 2$, $R = 7.0\%$ for 2817 independent reflections. The anion contains three BH_2 groups and two $\text{B}-\text{H}-\text{B}$ groups in the open face of the icosahedral fragment of the B_9 cluster (2o13 topology).

N.N. Greenwood, J.A. McGinnety and J.D. Owen, *J. Chem. Soc. Dalton*, (1972) 986.

Bis[bis(diethyl ether)- μ -(dodecahydro-*nido*-decaborato)-cadmium], $\{(\text{Et}_2\text{O})_2\text{Cd}(\text{B}_{10}\text{H}_{12})\}_2$
($P\bar{1}$) $Z = 1$ (dimer), $R = 9.8\%$ for 3423 independent reflections. Each cadmium is bonded to two $\text{B}_{10}\text{H}_{12}$ icosahedral fragments via three-centre bonds, and the coordination is completed by two ether ligands.

N.N. Greenwood, J.A. McGinnety and J.D. Owen, *J. Chem. Soc. Dalton*, (1972) 989.

Tetrakis(tricarbonyl- μ_3 -methane-thiolato-rhenium)

($P3_1 2_1$) $Z = 3$, $R = 5.4\%$ for 812 observed reflections. In a structure with an almost regular tetrahedron of rhenium atoms each sulphur is equidistant from the nearest three rheniums and the $\text{C}-\text{S}$ bonds normal to the triangular planes of the tetrahedron. $\text{Re}-\text{S} = 2.48-2.52(1)$, $\text{Re} \cdots \text{Re} = 3.853-3.957 \text{ \AA}$.

W. Harrison, W.C. Marsh and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 1009.

Tetrakis(*N,N*-diethyldithiocarbamato) titanium(IV)

($P\bar{1}$) $Z = 4$, $R = 9.7\%$ for 3637 independent reflections. The titanium atom is eight-coordinated by sulphur atoms from four chelating dithiocarbamato ligands. The geometry is close to dodecahedral with chelation along the *m* edges giving the D_{2d} isomer.

M. Colapietro, A. Vaciago, D.C. Bradley, M.B. Hursthouse and I.F. Rendall, *J. Chem. Soc. Dalton*, (1972) 1052.

Octacarbonyl- μ -(1,2,3,4-tetraphenyl-but-2-ene-1,1,4,4-tetrayl)-*triangulo*-triosmium,
 $(\text{Ph}_4\text{C}_4)\text{Os}_3(\text{CO})_8$

$(P2_1/n) Z = 4, R = 12\%$ for 2178 reflections. The molecule consists of a cluster of three osmium atoms at the corners of an equilateral triangle, and the organic ligand. The possibility of a C—H—Os three-centre bond or of an Os—H bond is discussed.

G. Ferraris and G. Gervasio, *J. Chem. Soc. Dalton*, (1972) 1057.

μ -Oxo-bis[oxo-bis(*N,N*-diethyldithiocarbamato)rhenium(V)]

$(P2_1/n) Z = 4, R = 3.7\%$ for 3523 independent reflections. The molecule has a linear O—Re—O—Re—O system. Each rhenium atom is bonded to two bidentate *N,N*-diethyldithiocarbamato ligands to give a slightly distorted octahedral coordination. Re—S (mean) = 2.438, Re—O(bridging) = 1.910, Re—O(terminal) = 1.722 Å.

S.R. Fletcher and A.C. Skapski, *J. Chem. Soc. Dalton*, (1972) 1073.

Nitridobis(*N,N*-diethyldithiocarbamato)rhenium(V)

$(P2_1/c) Z = 4, R = 3.4\%$ for 1606 independent reflections. In the monomeric complex, rhenium is square pyramidal with apical nitrogen and four basal sulphurs from the two bidentate ligands. Rhenium is almost 0.73 Å above the pyramid base and N—Re—S are ca. 108°. Re≡N = 1.656 and Re—S = 2.381–2.391 Å.

S.R. Fletcher and A.C. Skapski, *J. Chem. Soc. Dalton*, (1972) 1079.

[1*RS*, 3*SR*, 8*RS*, 10*SR*]-3,5,7,7,10,12,14,14-Octamethyl-1,4,8,11-tetra-azacyclotetradeca-4,11-diene]nickel(II) perchlorate

$(P2_1/c) Z = 4, R = 8.0\%$ for 2211 independent reflections. In the complex cation the two secondary amine and two imino-donor groups are in *trans* planar coordination.

The two asymmetric carbon centres and the two asymmetric nitrogen centres occur in racemic pairs with the racemic configuration given above.

D.A. Swann, T.N. Waters and N.F. Curtis, *J. Chem. Soc. Dalton*, (1972) 1115.

Bis(1-oxopyridine-2-thiolato)phenyl bismuth

$(P2_1/a) Z = 4, R = 3.9\%$ for 931 reflections. The coordination around bismuth is distorted octahedral with a lone pair presumably occupying one coordination position. Sulphur and oxygen atoms are *cis*.

J.D. Curry and R.J. Jandacek, *J. Chem. Soc. Dalton*, (1972) 1120.

cis-Dichloro{diethyl(phenyl)phosphine}(ethyl isocyanide)platinum(II),
cis-[PtCl₂(CNEt)(PEt₂Ph)]

$(P2_12_12_1) Z = 4, R = 6.2\%$ for 1062 independent reflections. Within the monomeric molecule coordination around platinum is square planar.

Pt—C = 1.83(4), Pt—P = 2.244(8), Pt—Cl(*trans* to C) = 2.310(10) and Pt—Cl(*trans* to P) = 2.390(8) Å.

B. Jovanović and L. Manojlović-Muir, *J. Chem. Soc. Dalton*, (1972) 1176.

cis-Dichlorobis(phenyl isocyanide) platinum(II), *cis*-[PtCl₂(CNPh)₂]

(*P2*₁/*c*) *Z* = 4, *R* = 4.6% for 1644 independent reflections. There is a Pt··Pt contact of 3.48 Å and coordination around each platinum atom is square planar. Pt—C(mean) = 1.896(16), Pt—Cl(mean) = 2.308(5) Å.

B. Jovanović, L. Manojlović-Muir and K.W. Muir, *J. Chem. Soc. Dalton*, (1972) 1178.

Tetramethylammonium triacetatodisphenylplumbate(IV)

(*Pcan*) *Z* = 4, *R* = 3.7% for 886 reflections. The lead atom is eight-coordinate with approximately hexagonal bipyramidal geometry. The Pb—C(axial) bonds are 2.16(3) Å Pb—O(equat) = 2.55 Å (mean).

N.W. Alcock, *J. Chem. Soc. Dalton*, (1972) 1189.

7,8- μ -Dimethylalumina-1,2-dicarba-*nido*-undecaborane(13), B₉C₂H₁₂AlMe₂, at -100°C

(*P1*) *Z* = 1, *R* = 13.5% for 875 non-zero reflections. All hydrogens were located. The B₉C₂H₁₂ moiety is present as an icosahedral fragment with the AlMe₂ group lying outside the cage and interacting with only two boron atoms.

M.R. Churchill and A.H. Reis, Jr., *J. Chem. Soc. Dalton*, (1972) 1314.

3-Ethyl-3-alumina-1,2-dicarba-*closo*-dodecaborane(12), 1,2-B₉C₂H₁₁AlEt

(*Pnma*) *Z* = 4, *R* = 8.49% for 499 independent reflections. The crystal consists of discrete, well separated molecular units of 1,2-B₉C₂H₁₁AlEt in which the aluminium atom is σ -bonded to an ethyl group and linked symmetrically to the open B₃C₂ face of a dicarba-*nido*-undecaborane ligand.

M.R. Churchill and A.H. Reis, Jr., *J. Chem. Soc. Dalton*, (1972) 1317.

Dibromotricarbonyl[1,2-bis(diphenyl-phosphino)ethane]molybdenum(II)-1-acetone

(*P2*₁/*c*) *Z* = 4, *R* = 6.4% for 1833 independent reflections. The molybdenum atom is seven-coordinate with a slightly distorted capped octahedral environment. A carbonyl group occupies the cap position. Mo—C = 1.895(19) Å. The capped face of the octahedron contains the other two carbonyl groups, Mo—C = 1.958(20) and 1.935(22) Å, and a phosphorus, Mo—P = 2.500(4) Å. The uncapped face has bond lengths Mo—Br = 2.658(2) and 2.665(3), Mo—P = 2.618(5) (*trans* to CO).

M.G.B. Drew, *J. Chem. Soc. Dalton*, (1972) 1329.

Amminobis(2,2'-bipyridyl)copper(II) tetrafluoroborate

(*P2*₁/*c*) *Z* = 4, *R* = 8.7% for 2894 independent reflections. The environment about the copper atom is approximately trigonal bipyramidal. The trigonal plane is made up of a nitrogen atom from each bipyridyl ligand, Cu—N = 2.09 Å, \angle N—Cu—N = 108°, and an ammonia molecule, Cu—NH₃ = 2.05 Å. Two bipyridyl nitrogens occupy the axial positions, Cu—N = 1.97 Å.

F.S. Stephens, *J. Chem. Soc. Dalton*, (1972) 1350.

Nitratotetrakis(2-methylimidazole)cobalt(II) nitrate-0.5 ethanol

($P\bar{1}$) $Z = 2$, $R = 10.8\%$ for 1434 independent reflections. The structure contains $[\text{Co}(\text{2-meim})_4\text{NO}_3]^+$ ions and free nitrates. In the cation, one bidentate nitrate group and four 2-meim form a very distorted octahedral coordination about the cobalt atom. $\text{Co}-\text{O} = 2.115$ and 2.269 Å, $\text{Co}-\text{N} = 1.960$ – 2.253 Å. There is some disordered ethanol in the lattice.

F. Alkhtar, F. Huq and A.C. Skapski, *J. Chem. Soc. Dalton*, (1972) 1353.

Iodobis(*N,N*-diethyldithiocarbamato)iron(III)

($P2_1/n$) $Z = 4$, $R = 11\%$ for 567 independent reflections. The coordination about iron is an approximate square pyramid consistent with the observed spin state of $S = \frac{3}{2}$. The iodine is at the apex and iron lies 0.6 Å above the basal plane of the dtc sulphurs. $\text{Fe}-\text{S} = 2.28(2)$, $\text{C}-\text{S} = 1.73(4)$, $\text{C}-\text{N} = 1.37(8)$, $\text{Fe}-\text{I} = 2.59(1)$ Å.

P.C. Healy, A.H. White and B.F. Hoskins, *J. Chem. Soc. Dalton*, (1972) 1369.

A metal ring compound di- μ -dimethylstannylene-bis(tetracarbonyliron),

$[(\text{Me}_2\text{Sn})\text{Fe}(\text{CO})_4]_2$

(*Pbam*) $Z = 4$, $R = 12\%$ for 544 independent reflections. The tin and iron atoms alternate around a planar four-membered ring with distorted octahedral iron and slightly disordered tetrahedral tin. $\text{Fe}-\text{Sn} = 2.647(8)$, $\text{Fe}-\text{C} = 1.68(8)$, $\text{C}-\text{O} = 1.27(8)$ Å. $\angle\text{Sn}-\text{Fe}-\text{Sn} = 77^\circ$. The metal atoms and "equatorial" carbonyls are coplanar while axial carbonyls bend in slightly. $\text{Sn}-\text{C}(\text{mean}) = 2.22(7)$ Å.

C.J. Gilmore and P. Woodward, *J. Chem. Soc. Dalton*, (1972) 1387.

[*N,N'*-Ethylenebis(salicylideneiminato)]pyridine(vinyl)cobalt(III)

(*Pnma*) $Z = 4$, $R = 7.0\%$ for 917 reflections. The stereochemistry of the cobalt is distorted octahedral with equatorial salen and axial vinyl and pyridine. The complex has an umbrella-shaped conformation with apparent *m* symmetry.

M. Calligaris, G. Nardin and L. Randaccio, *J. Chem. Soc. Dalton*, (1972) 1433.

Bromobis(π -cyclopentadienyl) (tribromostannyl) molybdenum(IV)

(*Pnma*) or (*Pna2_1*) $Z = 4$, $R = 5.6\%$ for 601 independent reflections. The tin atom forms four bonds directed towards the corners of a distorted tetrahedron with $\text{Sn}-\text{Mo} = 2.691$ and mean $\text{Sn}-\text{Br} = 2.505$ Å. The tin completes a distorted trigonal bipyramid coordination sphere with a long (3.411 Å) bromine contact. $\angle\text{Sn}-\text{Mo}-\text{Br} = 80.2^\circ$, $\text{Mo}-\text{Br} = 2.605$ Å.

T.S. Cameron and C.K. Prout, *J. Chem. Soc. Dalton*, (1972) 1447.

Bis(*N*-methyldithiocarbamato)nickel(II)

($P2_1/a$) $Z = 2$, $R = 12\%$ for 671 independent reflections. The nickel atom occupies the special position with $\bar{1}$ symmetry and is square planar coordinated by four sulphur atoms. $\text{Ni}-\text{S} = 2.203(9)$, $2.196(9)$ Å; $\angle\text{S}-\text{Ni}-\text{S} = 79.2^\circ$.

P.W.G. Newnham and A.H. White, *J. Chem. Soc. Dalton*, (1972) 1460.

Tri-iodoheptakis(tri-*p*-fluorophenyl-phosphine)undecagold

(*R*3) $Z = 1$, $R = 3.4\%$ for 1959 independent reflections. The metal-atom cluster derives from a centred icosahedron in which one triangular face has been substituted by a single gold atom Au—Au = 2.68–3.187, Au—P = 2.21(1)–2.29(1), Au—I = 2.600(5) Å.

P. Bellon, M. Manassero and M. Sansoni, *J. Chem. Soc. Dalton*, (1972) 1481.

Chloro{bis-[2-diethylamino)ethyl]-2-(diphenylphosphino)ethylamine}cobalt(II) perchlorate

(*Cc*) $Z = 4$, $R = 9.7\%$ for 1115 independent reflections. The cobalt atom is five-coordinate with an N₃PCl donor set arranged in a very distorted trigonal bipyramid or capped tetrahedron. Co—N = 2.30 (tripod ligand), Co—P = 2.42, Co—N = 2.17(3), 2.15(2) and Co—Cl = 2.28(1) Å.

P. Dapporto and G. Fallani, *J. Chem. Soc. Dalton*, (1972) 1498

Dichloro-bis- μ -ethanethiolate-bis(triphenylphosphine)diplatinum(II)

(*Pbca*) $Z = 4$, $R = 5.0\%$ for 2115 independent reflections. The platinum atom is in a strained square planar environment. There is severe steric repulsion between the aryl groups in the phosphorus and the sulphur atoms. Pt—S(*trans* to P) = 2.37(1), Pt—S(*trans* to Cl) = 2.27 Å. Pt—P = 2.26 Å.

M.C. Hall, J.A.J. Jarvis, B.T. Kilbourn and P.G. Owston, *J. Chem. Soc. Dalton*, (1972) 1544.

The aziridinylgallane trimer

(*Pnma*) $Z = 4$, $R = 7.7\%$ for 252 observed reflections. The six-membered (Ga—N)₃ ring is in the chair conformation, torsion angles 59–61°. Ga—N = 1.97, N—C = 1.55, C—Cl = 1.55 Å.

W. Harrison, A. Storr and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 1554.

Two crystalline forms of isothiocyanato-[1,7-bis(2-pyridyl)-2,6-diazaheptane]·copper(II) thiocyanate

(*B*2₁/*c*) $Z = 2$, $R = 10.3\%$ for 2648 independent reflections (α), and

(*P*1) $Z = 8$, $R = 11.6\%$ for 2358 independent reflections (β). The structures are conformational isomers each involving a complex cation containing five-coordinate copper(II) in a distorted trigonal bipyramidal geometry and a thiocyanate in the lattice. The isomerism arises from the different configurations at the aliphatic nitrogen atoms of the tetramine ligands in the two compounds.

N.A. Bailey and E.D. McKenzie, *J. Chem. Soc. Dalton*, (1972) 1566.

Dinitratotris(pyridine)-cobalt(II), copper(II) and nickel(II)

($C2/c$) $Z = 4$ in each case. $R = 7\%$ for 1104 reflections (Co), $R = 8\%$ for 1138 reflections (Cu), and $R = 11\%$ for 1020 reflections (Ni). The geometries are similar in each case with three pyridines and two nitrate groups coordinated to the metal. Large distortions attributable to Jahn–Teller effects are discussed.

A.F. Cameron, D.W. Taylor and R.H. Nuttall, *J. Chem. Soc. Dalton*, (1972) 1603.

Dinitratotrispyridinecadmium(II)

($C2/c$) $Z = 4$, $R = 8.4\%$ for 1491 independent reflections. The cadmium is seven-fold coordinated to three pyridine nitrogens, $Cd-N = 2.301, 2.347$, and two bidentate nitrates, $Cd-O = 2.444, 2.491$.

A.F. Cameron, D.W. Taylor and R.H. Nuttall, *J. Chem. Soc. Dalton*, (1972) 1608.

A diastereoisomeric pair of $\alpha\beta$ -chloro(tetraethylenepentamine)-cobalt(III) cations

($P2_12_12_1$) $Z = 4$, $R = 13.5\%$ for 1601 reflections ($\alpha\beta R$) and ($P2_12_12_1$) $Z = 4$, $R = 12.9\%$ for 1529 reflections ($\alpha\beta S$). In both cations the ligand chain is bound by nitrogen coordination successively to the cobalt octahedral sites. The cations differ only by an inversion at the secondary nitrogen atom.

M.R. Snow, *J. Chem. Soc. Dalton*, (1972) 1627.

Tetra-acetyl diborate

($P2_1/c$) $Z = 4$, $R = 5.0\%$ for 1337 independent reflections. Two distorted tetrahedra sharing a vertex are present in the molecule. Two acetoxy groups have only a B–O bond and the remaining two are each linked to both boron atoms. $B-O = 1.384-1.561$ Å

A. Dal Negro, L. Ungaretti and A. Perotti, *J. Chem. Soc. Dalton*, (1972) 1639.

Aquo-bis(2,2'-bipyridyl)palladium dinitrate

($P2_1/c$) $Z = 4$, $R = 6.0\%$ for 2589 reflections. The steric strain imposed by the interaction of interligand *ortho* hydrogens is reduced by a distortion from a square planar configuration around the metal.

P.C. Chieh, *J. Chem. Soc. Dalton*, (1972) 1643.

Bis[(acetato)aquo(pyridine)]nickel(II)

($Pbca$) $Z = 4$, $R = 8.0\%$ for 865 independent reflections. In the centrosymmetric monomeric complex nickel is octahedrally coordinated by pyridine and water and by two unidentate acetate groups. $Ni-N = 2.100$, $Ni-H_2O = 2.102$, $Ni-OAc = 2.050$ Å.

J. Drew, M.B. Hursthouse and P. Thornton, *J. Chem. Soc. Dalton*, (1972) 1658.

**Aquobis(ethylenediamine)(tetrafluoroborato)nickel(II) tetrafluoroborate,
[Ni(en)₂(H₂O)(BF₄)](BF₄)**

(*P*2₁2₁2₁) *Z* = 4, *R* = 8.2% for 1267 independent non-zero reflections. The en ligands are *cis* about the metal and in *gauche* configuration. A water and a BF₄⁻ fluorine occupy the remaining positions of the octahedral coordination sphere. Ni—F = 2.12 Å. Electronic spectra are discussed.

A.A.G. Tomlinson, M. Bonamico, G. Dessy, V. Fares and L. Scaramuzza, *J. Chem. Soc. Dalton*, (1972) 1671.

Tetra-μ-*o*-bromobenzoato-bis[aquo-copper(II)]

(*P*2₁/*n*) *Z* = 2, *R* = 9.9% for 985 observed reflections. The structure consists of centrosymmetric dimers with four *o*-bromobenzoate anions forming *syn-syn* bridges between the pairs of copper atoms. Cu—Cu = 2.624(7), Cu—O(carboxylate) = 1.99(3), Cu—OH₂ = 2.17(2) Å.

W. Harrison, S. Rettig and I. Trotter, *J. Chem. Soc. Dalton*, (1972) 1852.

Tris(*N,N*-diethylidithiocarbamato)manganese(III)

(*P*2₁/*a*) *Z* = 4, *R* = 11% for 1059 independent reflections. Three ligands surround the central manganese, chelating through sulphur. The geometry is very distorted from the axial *D*₃ point symmetry. Mn—S = 2.38(1), 2.43(1) and 2.55(1); C—S = 1.65–1.77 Å; ∠C—S—Mn = 82–90°.

P.C. Healy and A.H. White, *J. Chem. Soc. Dalton*, (1972) 1883.

π-(Cyclopentadienyl)- and π-(methylcyclopentadienyl)-*trans*-iododicarbonyl-(trimethylphosphite)molybdenum ((A) and (B) respectively)

R = 7.8% for 906 reflections (A) and *R* = 5.9% for 1847 reflections (B). Both compounds crystallize in (*P*2₁2₁2₁) with *Z* = 4. The Mo—P distances in (A), viz. 2.406(9), 2.388(8), are significantly shorter than in π-cyclopentadienyl-*trans*-iododicarbonyl-(triphenylphosphine)molybdenum (C). Mo—I distances 2.836(4) and 2.850(3) in (A) and (B) and 2.858 in (C) are constant.

A.D.U. Hardy and G.A. Sim, *J. Chem. Soc. Dalton*, (1972) 1900.

**Dichlorotris[1,2-bis(diphenylphosphino)ethane]dicopper(I)-bisacetone,
Cu₂Cl₂(dppe)₃·2(C₃H₆O)**

(*P*2₁/*c*) *Z* = 2, *R* = 6.2% for 1429 independent reflections. The structure consists of dinuclear centrosymmetric Cu₂Cl₂(dppe)₃ molecules and disordered acetone molecules in a ratio 1:2. The copper atoms are tetrahedrally coordinated to a chlorine and three phosphorus atoms. Two dppe ligands are chelating, one for each copper, and the third bridges. Cu—Cl = 2.312(4), Cu—P = 2.291(5), 2.311(4), 2.284(6) Å.

V.G. Albano, P.L. Bellon and G. Ciani, *J. Chem. Soc. Dalton*, (1972) 1938.

Di- μ -chloro(dichloromercurio)bis(dimethylphenylphosphine)platinum(II)

($P2_1/c$) $Z = 4$, $R = 5.7\%$ for 2184 independent reflections. The complex contains two bridging chlorine atoms between Pt and Hg with planar coordination about Pt and distorted tetrahedral about Hg.

R.W. Baker, M.J. Braithwaite and (the late) R.S. Nyholm, *J. Chem. Soc. Dalton*, (1972) 1924.

[N,N' -Ethylenebis(salicylideneiminato)]dimethyltin(IV)

($Pca2_1$) $Z = 8$, $R = 9.8\%$ for 1636 reflections. The crystal consists of $[\text{Me}_2\text{Sn}(\text{salen})]$ molecules with the salen ligand occupying the four equatorial positions of a distorted octahedron around the tin atom; methyl groups are *trans*.

M. Calligaris, G. Nardin and L. Randaccio, *J. Chem. Soc. Dalton*, (1972) 2003.

Carbonylchloro(tetracyanoethylene)bis(triphenylarsine)iridium

($P2_1n$) $Z = 4$, $R = 7.8\%$ for 4837 independent reflections. The coordination geometry is trigonal bipyramidal with apical carbonyl and chlorine. The central C—C bond of the tetracyanoethylene is almost parallel to the equatorial plane.

J.B.R. Dunn, R. Jacobs and C.J. Fritchie, Jr., *J. Chem. Soc. Dalton*, (1972) 2007.