Coordination Chemistry Reviews
Elsevier Publishing Company, Amsterdam – Printed in The Netherlands

X-RAY BIBLIOGRAPHY

M. KEETON

Department of Chemistry, McMaster University, Hamilton, Ontario (Canada)

(Received January 7th, 1971)

$Co_5(CO)_{15}C_3H$

($P\bar{1}$) triclinic. 1392 reflections were collected by film techniques and measured photometrically. The final R was 0.067. The compound is a product of the reaction of $ClCCo_3(CO)_9$ with mesitylene. The $CCo_3(CO)_9$ bridge structure is retained and is linked to a $Co_2(CO)_6$ unit through an acetylene bridge. This bridge and the two cobalt atoms form a tetrahedral group. The five Co-Co bonds in the molecule lie between 2.447 (4) to 2.485 (4) Å.

R.J. Dellaca, B.R. Penfold, B.H. Robinson, W.T. Robinson and J.L. Spencer, *Inorg. Chem.*, 9 (1970) 2197.

$Co_6(CO)_{18}C_4$

($P\bar{1}$) triclinic $R = 0.047$ for 1088 reflections. The compound is another product of the reaction of $ClCCo_3(CO)_9$ with mesitylene. The structure consists of discrete molecules formed by the insertion of a carbon-carbon triple bond between two $\cdot CCo_3(CO)_9$ units. This mean Co-Co bond length is 2.47(1) Å and the distances of C-C and C≡C in the C-C≡C-C chain are 1.37(1) and 1.24(2) Å, respectively.

R.J. Dellaca, B.R. Penfold, B.H. Robinson, W.T. Robinson and J.L. Spencer, *Inorg. Chem.*, 9 (1970) 2204.

$[(n\text{-}C_3H_7)_4N]\,[Co(S_2C_2(CN)_2)_2\,(o\text{-phen})]$, a dithiolene complex adduct

($Pbca$) $R = 0.068$ for 1490 reflections. The cobalt atom is in a trischelated octahedral environment. The average Co-N distance is 2.01(1) Å; Co-S, 2.23(2) Å.

G.P. Khare and R. Eisenberg, *Inorg. Chem.*, 9 (1970) 2211.

$[(n\text{-}C_4H_9)_4N]\,[Co(S_2C_6H_3(CH_3))_2\,(o\text{-phen})]$, a dithiolene complex adduct

($P2_1/c$) $R = 0.091$ for 1425 statistically reliable reflections. The overall structure is similar to the one above ($[(n\text{-}C_3H_7)_4N]\,[Co(S_2C_2(CN)_2)_2(o\text{-phen})]$). The Co-S distances vary according to whether they are *trans* to sulphur or nitrogen.

G.P. Khare and R. Eisenberg, *Inorg. Chem.*, 9 (1970) 2211.

$Mn_3[Co(CN)_6]_2 \cdot xH_2O$, a Prussian blue analog

($O_h\text{-}Fm3m$) $R = 10.5\%$ for 104 unique reflections. Co-C-N-Mn bridges connect octahedral CoC_6 and MnN_4 groups in a three dimensional array.

A. Ludi, H. Güdel and M. Ruegg, *Inorg. Chem.*, 9 (1970) 2224.

Sodium hexamolybdochromate(III) octahydrate, $Na_3(CrMo_6O_{24}H_6)\cdot 8H_2O$

($P\bar{1}$)R = 3.3% for 3976 reflections. The chromium atom is in an octahedral environment and is connected via six oxygen bridges to the six molybdenums, each set in a distorted octahedron of oxygens. The whole structure consists of columns of disc like $CrMo_6O_{24}H_6{}^{3-}$ groups stacked along the *c* axis held together by electrostatic bonding between oxygen and sodium ions and also by hydrogen bonding.

A. Parloff, *Inorg. Chem.*, 9 (1970) 2228.

Acenaphthylenediiron pentacarbonyl, $(C_{12}H_8Fe_2(CO)_5)$

($P2_1/n$)R = 6.71% for 1791 independent non-zero reflections. The structure consists of discreet $C_{12}H_8Fe_2(CO)_5$ units in which the carbon atoms of the five membered ring of modified acenaphthylene ligand form a π-cyclo pentadienyl system bonding to an $Fe(CO)_2$ group, while three other carbons form a π-allyl linkage to an $Fe(CO)_3$ group. The Fe-Fe distance is 2.769 Å.

M.R. Churchill and J. Wormald, *Inorg. Chem.*, 9 (1970) 2239.

π-Allyl(thiourea)nickel(II) chloride

($Pbca$)R = 0.096 for 1113 reflections. The nickel atom in $\pi\text{-}C_3H_5Ni\,(SC(NH_2)_2)_2{}^+$ units is in square planar coordination with the π-allyl and thiourea ligands. The S-Ni-S angle is 110.9 ± 1°. The characteristic allyl dihedral angle is 118 ± 3°.

A. Sirigu, *Inorg. Chem.*, 9 (1970) 2245.

trans-Chloro-2-(phenylazo)phenylbis(triethylphosphine)palladium(II)

($P2_1/c$)R = 9.0% for 2862 reflections. Two phosphorus atoms (*trans*), chlorine and a bonded carbon atom of the 2-(phenylazo)phenyl group coordinated in approximate square planar geometry to the palladium atom. The Pd-C distance is 1.994(15) Å.

D.L. Weaver, *Inorg. Chem.*, 9 (1970) 2250.

Ca_2VO_4Cl and Ca_2AsO_4Cl, synthetic spodiosites

(D_{2h}-$Pbcm$)R = 0.024 for 1147 reflections. The structures consist of distorted $XO_4{}^{3-}$, tetrahedra held together by calcium ions. Interatomic distances are normal and distortions cannot be explained by crystal packing forces alone. The distortions however are considerably smaller than in $CrO_4{}^{3-}$ which is explained by the electronic ordering of the lone electron in the d orbital.

E. Banks, M. Greenblatt and B. Post, *Inorg. Chem.*, 9 (1970) 2259.

Xenon difluoride-iodine pentafluoride, $XeF_2\cdot IF_5$

($I4/m$)R = 0.034 for 403 non-zero reflections. The orientation of the linear XeF_2 units in which each fluorine atom is directed towards the iodine atom of a neighbouring IF_5 molecule suggests that electrostatic attraction is the main lattice binding force. The F(Xe)-I distances are 3.142 ± 0.007 Å.

G. R. Jones, R.D. Burbank and N. Bartlett, *Inorg. Chem.*, 9 (1970) 2264.

Bis(*O,O'*-diisopropylphosphorothionyl) disulfide, $[(i\text{-}C_3H_7O)_2PS_2]_2$

($P\bar{1}$)R = 0.059 for 1073 significant reflections. The structure consists of planar

S=P-S-S-P=S units. The phosphorus atoms are in a tetrahedral environment with OC_3H_7 groups making up the 3rd and 4th positions. Some relevant distances are S-S, 2.109 ± 0.004 Å; P-S, 2.072 ± 0.002 Å; P=S, 1.908 ± 0.003 Å; P-O, 1.56 ± 0.005 Å.

S.L. Lawton, *Inorg. Chem.*, 9 (1970) 2269.

Dimethyl-1-6-dicarba-closo-decaborane (10)

($P\bar{1}ca$)R = 0.084 for 1615 reflections collected at –20° to –35°C. The structure shows only slight distortions of the bicapped square antiprism cage from the full D_{4d} symmetry.

T.F. Koetzle and W.N. Lipscomb, *Inorg. Chem.*, 9 (1970) 2279.

1,2′; 1′,2-Di-μ-carbonyl-bis [1.2] -dicarba-closo-dodecaborane (12)

($P2_1/a$)R = 7.1% for 1093. The icosahedral molecules are located on crystallographic centres of symmetry at the centres of the planar, six carbon atom rings. The average bonds distances were found to be C-B, 1.730(7) Å; B-B, 1.778(8) Å; C-C, 1.645(4) Å (polyhedral) and C-C, 1.517(4) Å (exopolyhedral). B-H average is 1.09(5) Å.

R.W. Rudolph, J.L. Pflug, C.M. Bock and M. Hodgson, *Inorg. Chem.*, 9 (1970) 2274.

$K_2[Ti_2O_5(C_7H_3O_4N)_2 \cdot 5H_2O$, a chelated dinuclear peroxytitanium(IV)

($C2/c$)R = 11.3% for 1439 observed reflections. The titanium atoms are in an approximate pentagonal bipyramid sharing an apical bridging oxygen atom. The equatorial sites are occupied by the peroxy groups and axial sites by dipicolinic acid and water. The O-O distance in the TiO_2 ring is 1.45 ± 0.007 and 1.905 ± 0.007 Å.

D. Schwarzenbach, *Inorg. Chem.*, 9 (1970) 2391.

Tris(ethylenediamine)cobalt pentacyanonitrosylchromate dihydrate, $[Co(C_2H_8N_2)_3]$-$[Cr(CN)_5(NO)] \cdot 2H_2O$

($P2_1/c$)R = 0.053 for 1106 observed significant reflections. The structure consists of $Co(C_2H_8N_2)_3{}^{3+}$ cations and $Cr(CN)_5(NO)^{3-}$ anions linked by hydrogen bonds. Cr-N is 1.71(1) Å; N-O is 1.21 Å and the angle Cr-N-O is 176°(1). Cr-C is 2.033(7) Å (equatorial) and 2.075(14) Å (axial).

J.H. Enemark, M.S. Quinby, L.L. Reed, M.J. Steuck and K.K. Walthers, *Inorg. Chem.*, 9 (1970) 2397.

Hydridotetrakis(trifluorophosphine)cobalt(I), $CoH(PF_3)_4$

($C2/c$)R = 0.11 based on 664 visually estimated intensities. The PF_3 groups occupy a distorted tetrahedron around the cobalt atom. The hydrogen atom was not located but the most favoured position is the apical position of a distorted trigonal bipyramid. Co-P average is 2.052(5) Å which suggests more multiple bond character than in metal, alkyl or aryl phosphines.

B.A. Frenz and J.A. Ibers, *Inorg. Chem.*, 9 (1970) 2403.

D-β_2-(*SSS*)-(Triethylenetetramine-(*S*)-prolinato)cobalt(III) tetrachlorozincate

($P2_1$)R = 0.055 for 1694 independent reflections. The structure consists of discreet D-β_2-(*SSS*)-Co(trien)((*S*)-Pro)$^{2+}$ cations and $ZnCl_4{}^{2-}$ anions held together by electrostatic attraction and hydrogen bonding. Large angular distortions in the cation relieve steric crowding in the complex.

H.C.Freeman, L.A. Marzilli and I.E. Maxwell, *Inorg. Chem.*, 9 (1970) 2408.

Tris(ethylenediamine)nickel(II) sulfate, $Ni(NH_2CH_2CH_2NH_2)_3SO_4$

($P\overline{3}1c$)R = 3.1% for 294 observed reflections. The sulphate ions are disordered in a previously unknown way and hydrogen bonding between the amine hydrogens of the ethylene diamine and sulphate group is unusual in that in one case, all the 'axial' hydrogens are involved in bonding to an adjacent sulphate group.

Mazhar-Ul-Haque, C.N. Caughlan and K. Emerson, *Inorg. Chem.*, 9 (1970) 2421.

Diacetylferrocene

($P2_1/c$)R = 6.9% for 2570 observed reflections. The cyclopentadienyl rings are planar and parallel with an average C-C distance of 1.426 ± 0.014 Å. The Fe-C distances range from 2.030 ± 0.005 to 2.060 ± 0.005 Å. The acetyl groups are in a 1,3′ configuration.

G.J. Palenik, *Inorg. Chem.*, 9 (1970) 2424.

Butadieneiron tricarbonyl-sulphur dioxide-boron trifluoride adduct, a sulphur dioxide insertion compound

($P2_1$)R = 6.60% for 813 independent non-zero reflections. Sulphur dioxide is found to be S bonded to a terminal methylene group of the modified butadiene ligand which is linked in π allyl fashion to the $Fe(CO)_3$ unit. A most important feature of the structure is that the sulphur dioxide is O bonded to the central iron atom. The other oxygen atom of SO_2 is linked to the boron atom of the BF_3.

M.R. Churchill and J. Wormald, *Inorg. Chem.*, 9 (1970) 2430.

Isothiocyanatothiocyanato-(1-diphenylphosphino-3-dimethylaminopropane) palladium (II)

($P2_1/c$)R = 0.041 for 2716 observed reflections. The palladium atom is coordinated in square planar fashion to phosphorus and nitrogen atoms of the organic ligand and one nitrogen and one sulphur atom from each thiocyanate group. The isothiocyanate linkage is *trans* to the phosphorus atom. Relevant bond distances are Pd-P, 2.243 ± 0.002 Å; Pd-S, 2.295 ± 0.002 Å, showing a shortening and Pd-N (ligand), 2.417 ± 0.006 Å; Pd-N (NCS), 2.063 ± 0.007 Å, a lengthening.

G.R. Clark and G.J. Palenik, *Inorg. Chem.*, 9 (1970) 2754.

trans-Chloronitrosylbis(ethylenediamine)cobalt(III)perchlorate

($P2_1/c$)R = 0.062 for 1671 independent reflections. The cobalt atom in *trans*-CoCl-(NO)$(en)_2^+$ is in a distorted octahedral coordination. A long Co-Cl distance, 2.575(3) Å indicates that the NO^- ligand has a high *trans* influence, attributed to its strong σ donor and weak π acceptor capacity.

D.A. Snyder and D.L. Weaver, *Inorg. Chem.*, 9 (1970) 2760.

Azidodinitrogenbis(ethylenediamine)ruthenium(III) hexafluorophosphate

($P2_1/n$)$R = 5.6\%$ for 1375 independent significant reflections. The ruthenium atom is octahedrally coordinated to six nitrogen atoms. Ru-N(N_2) is 1.894(9) Å and the Ru-N-N angle is 179.3(9)°; Ru-N(N_3) is 2.121(8) Å with the Ru-N-N angle 116.7(7)° The average Ru-N(en) distance is 2.125(18) Å.

B.R. Davies and J.A. Ibers, *Inorg. Chem.*, 9 (1970) 2768.

Bis(ethyl-thioxanthato)-μ-bis(ethyl-thioxanthato)-μ'-bis(ethylthio)-diiron(III), $[Fe(SC_2H_5)(S_2CSC_2H_5)_2]_2$

($P2_1/c$)$R = 0.048$ for 1350 independent statistically significant reflections. The structure consists of centrosymmetric dimers in which two ethyl mercaptide and two ethylthioxanthate groups bridge the two ions and two more ethylthioxanthate ligands complete the distorted octahedral coordinations around the iron atoms. The Fe-Fe distance is 2.618(2) Å.

D. Coucouvanis, S.J. Lippard and J.A. Zubieta, *Inorg. Chem.*, 9 (1970) 2775.

$Cu(NO_3)_2 \cdot 2.5\ H_2O$

($I2/a$)$R = 0.037$ for 2338 reflections. The copper atom is surrounded by four oxygen atoms from two water molecules and two NO_3^- units in an approximate square planar fashion. Cu-O, ~1.99 Å. Two more oxygen atoms from the same NO_3^- group coordinate at a distance of ~2.66 Å making up a pseudo octahedron. The molecules are connected via the oxygen atoms of the NO_3^- group, forming a chain.

B. Morosin, *Acta Crystallogr. Ser. B*, 26 (1970) 1203.

Dichloro-π-methallyl-bis-(triphenylarsine)rhodium(III)

($P\bar{1}$)$R = 0.065$ for 2694 independent reflections. The analysis confirms that the triphenyl arsine ligands are *trans* to the allyl group. The dihedral angle is 126.6°. The rhodium atom is in a very slightly distorted octahedron.

T.G. Hewitt, J.J. De Boer and K. Anzenhofer, *Acta Crystallogr. Ser. B*, 26 (1970) 1244.

Dichloro-DL-methioninepalladium(II)

($P2_1/n$)$R = 12.0\%$ for ~1500 observed reflections. Two chlorine atoms, sulphur and nitrogen, form a square plane around the palladium atom. The methionine molecule does not have the zwitter ion structure as in the free state. The molecules are associated as dimers via hydrogen bonding between adjacent carbonyl groups.

R.C. Warren, J.F. McConnell and N.C. Stephenson, *Acta Crystallogr. Ser. B.*, 26 (1970) 1402.

$(-)_{546}$ *cis*-β-Dinitro-(L-3,8-dimethyltriethylenetetramine)cobalt(III)perchlorate, $(-)_{546}$ *cis*-β-$[Co(NO_2)_2$(L-3,8-dimetrien)$]\ ClO_4$

($P2_12_12_1$)$R = 0.093$ for 883 observed reflections. The quadridentate ligand is coordinated to cobalt through its four nitrogen atoms. The strain in the *cis*-β-coordination is relieved by an adjustment in the conformation of the five numbered ring. The absolute configuration of the complex ion is that of a skew chelate pair, Δ.

M. Ito, F. Marumo and Y. Saito, *Acta Crystallogr. Ser. B*, 26 (1970) 1408.

Copper(II)chloride-bis(N,N-dimethylacetamido) thioether

($P\bar{1}$)$R = 0.016$ for 833 observed reflections. The copper atom is in a five coordinate square pyramidal environment. An oxygen atom occupies the apical position with the thioacetyl groups perpendicular to each other.

J. Coetzer, *Acta Crystallogr. Ser. B*, 26 (1970) 1414.

Zirconium sulphate, $Zr(SO_4)_2 \cdot H_2O$

($P\bar{1}$)$R = 12.6\%$ for 708 reflections. A layer compound with seven coordinate zirconium, using six oxygen atoms from bridging sulphates, and a water oxygen.

I.J. Bear and W.G. Mumme, *Acta Crystallogr. Ser. B*, 26 (1970) 1125. See also pp. 1131 and 1140.

Bis(thiourea)copper(I) chloride

($P2_1/a$)$R = 0.102$ for 1474 reflections. The structure consists of trigonal planar Cu^I triangles sharing vertices with adjacent triangles to form a chain spiralling along the c direction. The triangles are made up of a copper atom with sulphur atoms from different thioureas at the vertices. The Cu-Cu distances alternate between 4.311(4) and 2.981(4) Å, respectively.

W.A. Spofford and E.L. Amma, *Acta Crystallogr. Ser. B*, 26 (1970) 1474.

$(-)_{589}$ Tris(+*trans*-1,2-diaminocyclohexane)cobalt(III) chloride pentahydrate,
$(-)_{589}$ $[Co(+chxn)_3]Cl_3 \cdot 5H_2O$

($P61$)$R = 0.11$ for 1123 reflections. The complex ion is of approximate D_3 symmetry with the central cobalt atom bonded almost octahedrally to six nitrogen atoms. The complex is of the lel form, its absolute configuration is $\Lambda\delta\delta\delta$ (IUPAC convention).

F. Murumo, Y. Utsumi and Y. Saito, *Acta Crystallogr. Ser. B*, 26 (1970) 1492.

$(+)_{589}$-*cis*-Dinitrobis[$(-)_{589}$-1,2-propylenediamine] cobalt(III) chloride

($P2_12_12_1$)$R = 0.12$ for 800 independent reflections. In the complex octahedral cations the two *cis* nitro groups are planar and the propylenediamine chelate rings have the λ conformation. The methyl groups occupy *trans* positions. The known absolute configuration of $(-)_{589}$-1,2-propylenediamine was used to determine that of the complex cation.

G.A. Barclay, E. Goldschmied and N.C. Stephenson, *Acta Crystallogr. Ser. B*, 26 (1970) 1559.

Ethylenebis(biguanidine)nickel(II) dichloride monohydrate, $Ni(C_6H_{16}N_{10})Cl_2 \cdot H_2O$

($P2_1/c$)$R = 0.048$ for 2879 reflections. The tetradentate organic ligand coordinates in square planar fashion to the nickel atom. Ni-N is 1.865 Å (average). All available hydrogen atoms are involved in hydrogen bonding to chloride ions or water molecules.

B.L. Holian and R.E. Marsh, *Acta Crystallogr. Ser. B*, 26 (1970) 1049.

Bis(L-serinato)zinc, $ZnC_6H_{12}O_6N_2$

($P2_1/c$)R = 0.036 for 950 reflections. The coordination sphere of the zinc atom is intermediate between square pyramidal and trigonal bipyramidal. One serine molecule only has the unusual anti-gauche conformation.

D. Van der Helm, A.F. Nicholas and C.G. Fisher, *Acta Crystallogr. Ser. B*, 26 (1970) 1172.

π-Cyclopentadienyl(triethylphosphine)copper(I), π-$C_5H_5CuP(C_2H_5)_3$

($P2_1m$)R = 0.145 for 615 independent reflections. The C_5H_5 ring is π-bonded to copper with a Cu-C average length of 2.24 Å and C-C of 1.38 Å. The Cu-P length is 2.14 Å.

L.T.J. Delbaere, D.W. McBride and R.B. Ferguson, *Acta Crystallogr. Ser. B*, 26 (1970) 515.

Hexapyrazolenickel(II) nitrate, $Ni(C_3H_4N_2)_6(NO_3)_2$

($P\overline{3}$)R = 0.053 for 1115 reflections. The nickel atom lies at the centre of a nearly regular octahedron and is coordinated to non planar pyrazole molecules.

C.W. Reimann, A. Santoro and A.D. Mighell, *Acta Crystallogr. Ser. B*, 26 (1970) 521.

Cyclotetra [μ-oxo-chloro-π-cyclopentadienyltitanium(IV)]

($Cmc2_1$)R = 0.042 for 1347 independent reflections. The molecule consists of a tetrameric ring with the titanium tetrahedrally coordinated. The eight membered -Ti-O-Ti ring is not planar and the chlorine atoms and cyclopentadienyl rings are alternatively situated above and below the ring.

A.C. Skopski and P.G.A. Troughton, *Acta Crystallogr. Ser. B*, 26 (1970) 716.

Bis-(2-methylpentane-2,4-dioxydimethyltitanium), $[(CH_3)_2TiO_2C_6H_{12}]_2$

($P2_1/n$)R = 0.158 for 1107 reflections. The structure consists of two similar units of 2-methylpentane-2,4-dimethyltitanium joined by an oxygen atom at the 4 position. Each titanium is in trigonal bipyramid coordination to three oxygens and two methyl groups. Ti-C range from 2.11 to 2.19 Å.

A. Yoshino, Y. Shuto and Y. Iitoha, *Acta Crystallogr. Ser. B*, 26 (1970) 744.

Bis(hydrazinecarbonylato-*N'*, *O*)-nickel dihydrate

($C2/c$)R = 0.089. Ni-O distance is 2.038 Ni-N is 2.092 Å. The coordination of the nickel is octahedral and the whole structure is held together by hydrogen bonds.

A. Braibanti, A.M. Manotti Lanfredi, A. Tiripicchio and F. Bigoli, *Acta Crystallogr. Ser. B*, 26 (1970) 806.

$Cu(NO_3)_2 \cdot (C_4N_2H_4)$, a 1:1 cupric nitrate-pyrazine complex

($Pmna$)R = 0.04 for 425 unique observed reflections. The structure is a coordination polymer consisting of -Cu-pyrazine chains.

A. Santoro, A.P. Mighell and C.W. Reimann, *Acta Crystallogr. Ser. B*, 26 (1970) 979.

Di-μ-chloro-bis(triphenylphosphite)-(cyclo-octa-1,5-diene)dirhodium(I)
($P2_1/c$)R = 0.066 for 2500 observed reflections. The rhodium atoms are in square planar coordination, with a folded $RhCl_2Rh$ bridge. An unusually long C=C bond distance of 1.42 Å in the cyclo-octadiene ring is the result of the σ-π bonding with the rhodium atom.
J. Coetzer and G. Gafner, *Acta Crystallogr. Ser. B*, 26 (1970) 985.

Di-μ-N,N'-*m*-phenylene-tetrakis(salicylideneiminato)dicopper(II)
($C2/c$)R = 0.11 for 1592 reflections. A dimeric, centrosymmetric, structure in which two bis(salicylideneiminato)copper(II) residues are bridged with two *m*-phenylene groups. The coordination at each copper is intermediate between *cis* planar and tetrahedral.
C.A. Bear, J.M. Waters and T.N. Waters, *J. Chem. Soc. A*, (1970) 2494.

Bis-[1,2-bis(diphenylphosphino)ethane] rhodium(I) perchlorate
($P2_1/c$)R = 0.09 for 2476 reflections. A square planar rhodium atom is coordinated by the bidentate diphosphine ligand. A comparison with the structures indicates that the ligands are flexible.
M.C. Hall, B.T. Kilbourn and K.A. Taylor, *J. Chem. Soc. A*, (1970) 2539.

Chloro-π-cyclopentadienylbis-8-quinolinolatotitanium(IV), $[TiCl(\pi\text{-}C_5H_5)(C_9H_6NO)_2]$
($P2_1/c$)R = 0.098 for 2402 independent reflections. The structure consists of monomeric units of dodecahedral symmetry. The oxygen atoms are *trans* at 1.987 and 1.954 Å from the titanium atom. The nitrogen atoms are *cis* at 2.224 and 2.270 Å. The Ti-Cl distance is 2.372 Å.
J.D. Mathews, N. Singer and A.G. Swallow, *J. Chem. Soc. A*, (1970) 2545.

Carbonatodiaquobis(imidazole)cobalt(II), $[Co(CO_3)(OH_2)_2(imH)_2]$
($A2/a$)R = 0.062 for 1029 reflections. The cobalt atom is in a distorted octahedral environment with *trans* aquo, *trans* imidazole and the carbonato group surrounding it. Magnetic moments, UV, visible and IR spectra are recorded and interpreted.
E. Baraniak, H.C. Freeman, J.M. James and C.E. Nockolds, *J. Chem. Soc. A*, (1970) 2558.

Dichloro-di-μ-thiocyanato-bis(tri-n-propylphosphine)-platinum(II), α and β forms
$\alpha(P2_1/c)R(\alpha)$ = 0.054 for 1729 reflections; $\beta(P2_1/n)\,R(\beta)$ = 0.038 for 1348 independent reflections. In both isomers there is an almost planar eight membered -Pt-S-C-N-Pt-S-C-N ring. There is no satisfactory evidence for a delocalised π bond system extending over the molecular plane whose planarity is attributed to normal steric interactions.
U.A. Gregory, J.A.J. Jarvis, B.T. Kilbourn and P.G. Owston, *J. Chem. Soc. A*, (1970) 2770.

Bis(pentafluorobenzenethiolato)bis(tributylphosphine)platinum(II)

(C2/c)R = 0.11 for 1971 independent reflections. The molecule is almost square planar with the pentafluoro thiophenolato ligands in the *trans*-configuration with the platinum atom 0.28 Å out of the plane.

R.A. Fenn and G.R. Segrott, *J. Chem. Soc. A*, (1970) 2781.

Mono-α-picolinecopper(II) chloroacetate

$(P\bar{1})R$ = 0.089 for 1654 reflections. The copper atoms are bridged in pairs by four chloroacetate groups to form binuclear $[Cu(ClCH_2OCO_2)_2(MeC_5H_4N)]_2$ units. The copper atom lies in a distorted octahedron with four oxygen atoms in the plane, a nitrogen of the picoline ligand and the second copper at 2.247 Å.

G. Davey and F.S. Stephens, *J. Chem. Soc. A*, (1970) 2803.

Dimethyl tin dichloride

(Imma)R = 0.086 for 101 independent reflections. The environment of the tin atoms is distorted from the regular tetrahedron towards octahedral coordination due to association of neighbouring molecules. The Mossbauer spectrum is also reported.

A.G. Davies, H.J. Milledge, D.C. Puxley and P.J. Smith, *J. Chem. Soc. A*, (1970) 2862.

Dichloro(benzylacetophenone)bis(dimethylsulphoxide)iridium(III), $[Ir(C_{15}H_{13}O)Cl_2(Me_2SO)_2]$

$(P\bar{1})R$ = 0.073 for 2301 independent reflections. The iridium has a distorted octahedral coordination made up of two dimethylsulphoxides (Ir-S, 2.229(7) and 2.243(7) Å). Two chloride ions (Ir-Cl, 2.376(9) and 2.496(9) Å) and a benzylacetophenone ring form a five membered puckered chelate ring (Ir-O, 2.09(2) and Ir-C,2.16(2) Å).

M. McPartlin and R. Mason, *J. Chem. Soc. A*, (1970) 2206.

Dicarbonylcyclopentadienyl(tribromostannyl)iron, $[Fe(\pi\text{-}C_5H_5)(CO)_2SnBr_3]$

(Pbca)R = 0.04 for 1474 intensities. The Sn-Fe bond is 2.462(2) Å which is considerably shorter than the metal-metal separation found in the triphenyl stannyl analogue.

G.A. Melson, P.F. Stokely and R.F. Bryan, *J. Chem. Soc. A*, (1970) 2247.

Bis(pyridine-2-carboxylato)-copper(II), a 1:1 adduct with potassium thiocyanate

(Pnam)R = 0.09 for 1114 reflections. The copper atom is square planar coordinated, Cu-O, 1.942 and Cu-N, 1.961 Å. The thiocyanate ion is not coordinated to the copper atom.

F.S. Stephens, *J. Chem. Soc. A*, (1970) 2377.

Dichloro(oxo)-2,2′-bipyridylethoxyniobium(V) and tetraphenylarsonium oxopentathiocyanatoniobate(V)

Both complexes contain octahedral niobium with an oxygen atom multiply bonded to niobium. The *R* factors are 0.165 and 0.149, respectively.

B. Kamenar and C.K. Prout, *J. Chem. Soc. A*, (1970) 2379.

Bis [di(2-aminopropyl)amine] nickel(II) perchlorate (a) and bis [di(2-aminoethyl)amine]-nickel(II) chloride monohydrate (b)

The ligands coordinate in the equatorial planes of an octahedron. (a) $(Pbca)R = 0.090$ for 3236 observed reflections. (b) $(P2_1/c)R = 0.092$ for 1900 reflections.

S. Biagini and M. Cannas, *J. Chem. Soc. A*, (1970) 2398.

The *N,N'*-ethylenebis(salicylideneiminato) cobalt(II) monopyridine adduct

$(Cmc2_1)R = 0.071$ for 571 independent reflections. The cobalt atom is in pyramidal coordination with the apical position occupied by a pyridine molecule parallel to the CH_2-CH_2 bond. The fact that the compound is an oxygen carrier in solution and not in the solid state is rationalised by crystal packing.

M. Calligaris, D. Minichelli, G. Nardin and L. Randaccio, *J. Chem. Soc. A*, (1970) 2411.

$(+)_{495}$-[L-Glutamatobis(ethylenediamine)cobalt(III)] perchlorate

$(P2_12_12_1)R = 0.073$ for 1039 unique reflections. The absolute configuration of the cation is D. The cobalt is in octahedral coordination to five nitrogen atoms and an oxygen.

R.D. Gillard, N.C. Payne and G.B. Robertson, *J. Chem. Soc. A*, (1970) 2579.

A complex of mercury(II) chloride and histidine hydrochloride

The complex crystallises in the space group $P2_12_12_1$. The mercury chlorine molecules are loosely associated via a long Ag-Cl contact of 3.25 Å to form an infinite chain. The smallest other contact is 2.54 Å between mercury and oxygen of a carbonyl group. This completes a distorted trigonal bipyramid at the mercury atom. The histidine molecules are stacked parallel, lying along the *C* axis.

M.J. Adams, D.C. Hodgkin and V.A. Raeburn, *J. Chem. Soc. A*, (1970) 2632.

trans-Dichlorobis- [*o*-phenylene(bisdimethylarsine)] cobalt(*III*) perchlorate

$(C2)R = 0.08$ for 549 reflections. The cobalt atom is octahedrally coordinated. The mean Co-As bond length is significantly shorter than the covalent radius sum.

P.J. Pauling, D.W. Porter and G.B. Robertson, *J. Chem. Soc. A*, (1970) 2728.

trans-Di-μ-carbonyl-bis-(π-2,3-*cis*-dimethylbutadiene carbonylcobalt), $[Co(\pi\text{-}CH_2CMeCMeCH_2)(CO)_2]$

$(C2/c)R = 0.041$ for 1744 unique reflections. The molecule is centrosymmetric with a planar $Co(CO)_2Co$ bridge. Co-Co is 2.549 Å. The butadiene is in *cis*, planar configuration with C-C bond lengths equal, the mean is 1.417 Å.

F.S. Stephens, *J. Chem. Soc. A*, (1970) 2745.

Vanadium(III) tris (*O,O*-diethylphosphorodithioate), $V[PS_2(OEt)_2]_3$

$(C2/c)R = 0.077$ for 1124 observed independent reflections. The vanadium is coordinated to the three ligands in a distorted trigonal bipyramidal fashion. Polarised crystal spectra resolve the trigonal distortions and allow a D_3 symmetry assignment. Typical distances are V-S, 2.446(5) Å; S-P, 1.989(9) Å; P-O, 1.601(12) Å.

C. Furlani, A.A.G. Tomlinson, P. Porta and A. Sgamellotti, *J. Chem. Soc. A*, (1970) 2929.

Chloro-1,2-bis(diphenylphosphino)-ethanemonocarbonyl-π-cyclopentadienylmolybdenum(II), $Mo(\pi\text{-}C_6H_5)(CO)\text{-}(Ph_2PCH_2)_2Cl$

(Pbca)R = 7.47% for 2544 reflections. The molecule is a sandwich type having the cyclopentadienylring on one side of the molybdenum. The carbonyl ligand is *cis* to the chlorine atom, while the phosphorus is chelating and is not bridging.

J.H. Cross and R.H. Fenn, *J. Chem. Soc. A*, (1970) 3019.

trans-Di-μ-carbonyl-π-cyclopentadienyldi-iron (Fe-Fe), a redetermination

$(P2_1/c)R = 0.044$ for 904 reflections. The bridge is completely planar. Fe-Fe, 2.534(2); Fe-C(bridge), 1.910, 1.918(5); Fe-C terminal, 1.748(6); Fe-C(ring), 2.082-2.121(8) Å.

R.F. Bryan and P.T. Greene, *J. Chem. Soc. A*, (1970) 3064.

cis-Di-μ-carbonyl-dicarbonyldi-π-cyclopentadienyldi-iron (Fe-Fe)

$(P2_1/c)R = 0.051$ for more then 1500 reflections. The cyclopentadienyl rings are in a *cis*, almost eclipsed conformation. The molecular dimensions are very similar to those in the *trans* complex except for the nonplanarity of the Fe_2C_2 ring system where the angle between the two Fe_2CO planes is 164°.

R.F. Bryan, P.T. Greene, M.J. Newlands and D.S. Field, *J. Chem. Soc. A*, (1970) 3068.

Acetylacetonato(π-pentamethylbicyclo [2.2.0] hexa-2,5-dienylmethyl)palladium(II)

$(P2_1/n)R = 10\%$ for 1325 independent reflections. The olefin unit is bonded as a π-allyl group to the palladium; the dihedral angle is 121.5°. The palladium atoms and the acetylacetonato group are almost coplanar.

J.F. Malone and W.S. McDonald, *J. Chem. Soc. A*, (1970) 3124.

$[M_2(CO)_{10}]^{2-}$ (M = Cr, Mo) and $[Cr_2(CO)_{10}H]^-$, structural characterisation of the dinuclear metal anions

Both the $[Cr_2(CO)_{10}]^{2-}$ and $[Mo_2(CO)_{10}]^{2-}$ ions are unambiguously established as the $D4d\text{-}\overline{8}2m$ $Mn_2(CO)_{10}$ type configuration. The results allow a detailed appraisal of conformation changes on protonation of the metal-metal bond.

L.H. Handy, J.K. Ruff and L.F. Dahl, *J. Amer. Chem. Soc.*, 90 (1970) 7312.

$\{[(C_6H_5)_3P]_2N\}[Cr_2(CO)_{10}I]$

A monohalogen bridged dinuclear metal carbonyl anion. The structure has a bent -Cr-I-Cr framework which may be described in terms of localised electron pair bonds. The geometry in the cation is primarily governed by steric effects and is related to the conformation of $N_3P_3Cl_2(C_6H_5)_4$. The effect of increased $P(d_\pi)\text{-}N(p_\pi)\text{-}P(d_\pi)$ bonding, stronger P-Nσ bonding in the cation and the positive charge does not alter appreciably the bond lengths from those in the $(C_6H_5)_2P\text{-}N\text{-}P(C_2H_5)_2$ segment in the cyclotriphosphazene molecule.

L.B. Handy, J.K. Ruff and L.F. Dahl, *J. Amer. Chem. Soc.*, 92 (1970) 7327.

$Co_4(CO)_{12}Sb_4$

The structure ideally displays cubic $Td\text{-}\overline{4}3m$ geometry with an average Co-Sb distance of 2.614(2) Å and Sb-Co-Sb angle (acute) of 74.3(1)° and Co-Sb-Co (obtuse) of 103.8(1)°. It is the first known (main group element)-(metal carbonyl) cubane-type structure.

A.S. Foust and L.F. Dahl, *J. Amer. Chem. Soc.*, 92 (1970) 7337.