Coordination Chemistry Reviews, 13 (1974) 279–290

X-RAY BIBLIOGRAPHY

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Tetracesium-μ-oxo-decachlorodiosmate(IV), $Cs_4[Os_2OCl_{10}]$
(P_{cab}) $Z=4$. The complex anion forms corner-sharing octahedrons tetragonally deformed. Os–O, 1.778 Å, Os–Cl, 2.370 and 2.433 Å. The Cs atoms are coordinated differently. Cs–Cl range from 3.56 to 3.87 Å.
K.F. Tebbe and H.G. von Schnering, *Z. Anorg. Allg. Chem.*, 396 (1973) 66.

Hexaphenyl distannane, $(C_6H_5)_6Sn_2$
($P2_1/c$) $Z=4$, 2939 reflections. The unit cell contains two independent molecules of S_6 symmetry. The compound is isomorphous with $(C_6H_5)_6Pb_2$ but not with $(C_6H_5)_6Ge_2$.
H. Preut, H.J. Haupt and F. Huber, *Z. Anorg. Allg. Chem.*, 396 (1973) 81.

Decacarbamide nickel iodide, $NiI_2 . 10$ (urea)
($P2_1/c$) $Z=2$. $R=16\%$ for 980 independent reflections. The structure consists of octahedral $[\text{Ni urea}]^{2+}$ complexes, I^- ions and outersphere carbamide molecules. Ni–O 2.10(2) Å. The orientation of the urea molecules is such that the complex retains a three-fold pseudo-axis.
Kh. Saleimanov, M.A. Porai-Koshits, A.S. Antsyshkina and K. Sulaimankulov, *Russ. J. Inorg. Chem.*, 16 (1971) 1799.

μ-Oxo-bis [pentamminechromium(III)] chloride monohydrate, $[(NH_3)_5\text{–Cr–O–Cr}(NH_3)_5]Cl_4 . H_2O$
($Pbca$) $Z=4$, $R=14\%$ for 811 independent reflections. A bridging oxygen atom of the basic rhodochromic complex ion is situated on the inversion centre. The complex has a centre of symmetry and a linear Cr–O–Cr system. Cr–N, 2.09–2.14, Cr–O, 1.80 Å. The complex ion has approximate D_{4h} symmetry.
A. Urushiyama, *Bull. Chem. Soc. Jap.*, 45 (1972) 2406.

Cesium tetrakis(succinimidato)copper(II)dihydrate, $Cs_2(Cu(succim)_4)\ 2H_2O$
($C1$) $Z=2$, $R=11\%$. The coordination around the copper is square planar. The two independent cesium ions have a quite different environment.
T. Tsukihara, Y. Katsube, K. Fujimori and T. Ito, *Bull. Chem. Soc. Jap.*, 45 (1972) 2959.

$Co_6Te_5O_{16}$
M. Trömel and Th. Scheller, *Naturwissenschaften*, 60 (1973) 103.

Heptacesium oxide, $[Cs_{11}O_3]Cs_{10}$
($P6m2$) $R = 12\%$ for 249 reflections.
A. Simon, *Naturwissenschaften*, 58 (1971) 622.

Indium telluride chloride
($P2_1/c$) $Z = 8$, $R = 9.7\%$ for 932 reflections.
G. Roos, G. Lenber and H. Hahn, *Naturwissenschaften*, 59 (1972) 363.

Calcium vanadate, $Ca(OH)_2V_2O_5$
($Pnna$)
A. Kutoglu, *Naturwissenschaften*, 59 (1972) 36.

Bis(imidazole)octaethylporphinato iron(III) perchlorate
($P2_1/m$) $Z = 2$, $R = 12.9\%$. The iron atom is strictly coplanar with the four porphinato nitrogen atoms and two imidazole nitrogen atoms forming an approximately regular octahedron. The complex cations contact with the perchlorate anions at their non-coordinated imidazole nitrogen atoms.
A. Takenaka and Y. Sasada, *Chem. Lett. (Tokyo)*, 1 (1972) 1235.

Dichloro-tetrakis(diethyldithiocarbamato)-trimercury, $Hg_3Cl_2(S_2CNEt_2)_4$
($P1$) $Z = 2$, $R = 6.9\%$ for 3550 independent reflections. Mercury atoms are linked together by bridging carbamate groups and chlorine atoms to form an infinite chain.
H. Iwasaki, *Chem. Lett. (Tokyo)*, 1 (1972) 1105.

Chloroglycylglycinato copper(II) monohydrate
($P2_1/c$) $Z = 8$. The structure is dimeric and the peptide is bonded to two copper atoms; to one through the terminal nitrogen and the peptide oxygen and to the other through the terminal oxygen. The copper atom is five coordinated and has a distorted square pyramidal coordination polyhedron.
M. Shiro, *Chem. Lett. (Tokyo)*, 1 (1972) 123.

(A) Bis(picolinato)bis(thiourea)Cu(II) and (B) bis(picolinato)bis(allylthiourea)Cu(II)
(A) $Cu(II)(C_5H_4NCO_2)_2[SC(NH_2)_2]_2$. ($P2_1/n$) $Z = 2$, $R = 6.6\%$. (B) $Cu(II)(C_5H_4\text{-}NCO_2)_2[SC(NH_2)NHCH_2CH{=}CH_2]_2$. ($P2_1/c$) $Z = 2$, $R = 6.0\%$. In both compounds copper is on a centre of symmetry surrounded by two picolinic ions which are chelating through the pyridine nitrogen and a carbonyl oxygen. (Cu–N, 1.953, 1.984; Cu–O, 1.985, 1.974 Å). The coordination polyhedron is completed to an elongated tetragonal bipyramid by two sulphur atoms from two thiourea (or allyl thiourea) molecules. Cu–S is much longer than those usually found in metal thiourea complexes (2.943, 2.927 Å).
M.B. Ferrari, L.C. Capacchi, G.G. Fara, A. Montenero and M. Nardelli, *Sov. Phys. Crystallogr.*, 17 (1972) 16.

Cadmium hexamethylenedithiocarbamate
($P2_1/c$) $Z=4$, $R=13.6\%$ for 1510 reflections. The structure consists of dimeric molecules in which two ligand molecules form four-membered rings, while others serve as bridges between the two cadmium atoms. Each cadmium in the dimer forms five Cd–S bonds, two bonds in the ring (2.631 and 2.539 Å) two bonds with one bridge ligand group (2.539 and 2.873 Å) and one with the other ligand group (2.617 Å). The coordination polyhedron around the metal atom is between tetragonal pyramidal and trigonal bipyramidal.
V.M. Agre and E.A. Shugam, *Sov. Phys. Crystallogr.*, 17 (1972) 2.

(A) $NaLu(WO_4)_2$ and (B) $Na_5Lu(WO_4)_4$
($I4_1/a$) $Z=4$. (A) has pure scheelite structure with the Na and Lu atoms statistically distributed into the 4(*b*)(*ca*) positions. (B) is also a scheelite structure with W atoms located inside distorted tetrahedra while Lu atoms are inside 8-coordinate polyhedra.
R.F. Klevtsova, L.A. Glinskaya, L.P. Kozeeva and P.V. Klevtsov, *Sov. Phys. Crystallogr.*, 17 (1972) 672.

Sodium indium molybdate, $NaIn(MoO_4)_2$
($P1$) $Z=4$, $R=10.3\%$. The distorted Mo tetrahedra (Mo–O, 1.53–1.89 Å) and In octahedra (In-O, 2.06–2.27 Å) are joined through common oxygen apices to form a three dimensional framework, the cavities of which are occupied by Na atoms.
R.F. Klevtsova and P.V. Klevtsov, *Sov. Phys. Crystallogr.*, 17 (1972) 843.

$CeCr_2B_6$
(*Immm*) $Z=2$. The structure can be regarded as a packing of trigonal prisms or trigonal prisms, pyramids and empty tetrahedra. The B atoms form a framework of corrugated 14-member rings.
Yu.B. Kuz'ma and S.I. Svarichevshaya, *Sov. Phys. Crystallogr.*, 17 (1972) 830.

Cs_2HgI_4
($P2_1/m$) $Z=2$, $R=14.3\%$. The HgI_4 group deviates substantially from a regular tetrahedron. Attempts to establish a correlation between the Hg–I bond lengths and the mean Cs···I distance were unsuccessful.
V.I. Pakhomov and P.M. Fedorov, *Sov. Phys. Crystallogr.*, 17 (1972) 833.

$Co(NH_4)_2(SO_4)_2 . 6H_2O$
($P2_1/c$) $Z=2$. The isolated cobalt tetrahedra are formed by six water molecules; the S tetrahedra and the ammonium group being located in the outer sphere. Co–O, 2.08–2.12; O–O, 2.87–3.05 Å.
V.G. Rau and E.N. Kurkutova, *Sov. Phys. Crystallogr.*, 17 (1972) 997.

$K_2Cu(SO_4)_2 . 2H_2O$
($P2_1/c$) $Z=4$, $R=9.2\%$. Coordination of copper is through five oxygen atoms that form a pyramid with tetragonal base.

E. Philippot, J.-C. Tedenae and M. Maurin, *Rev. Chim. Miner.*, 9 (1972) 805.

$KH(SeO_3)_2$ and $NaH_3(SeO_3)_2$

(K). ($Pbcn$) $Z=4$, $R=8.2\%$ for 580 independent reflections. (Na). ($P2_1/n$) $Z=2$, $R=8.5\%$ for 472 independent reflections. The differences between the crystal structures of these compounds are reflected sharply by the contrast in physical properties.

L.V. Gorbatyi, V.I. Ponomarev and D.M. Kheiker, *Sov. Phys. Crystallogr.*, 16 (1972) 781.

$PbBi_4Te$, $PbBi_2Te_4$, $SnBi_4Te_7$, $SnBi_2Te_4$, $SnSbTe_4$ and $GeBi_4Te_7$

All the substances have a layer structure and apart from $GeBi_4Te_7$ are isostructural. The rhombohedral cell (D^5_{3d}) contains 21 atoms. $GeBi_4Te_7$ has a trigonal cell (D_3d^3) with 12 atomic layers.

T.B. Zhukova and A.I. Zaslavskii, *Sov. Phys. Crystallogr.*, 16 (1972) 796.

$K_2Mo_2O_7$

($P1$) $Z=2$, $R=11.7\%$. Chains of Mo tetrahedra and Mo octahedra run parallel to the *b* axis. Connected chains of K14-hedra of two types form layers which are responsible for the (101) cleavage planes.

S.A. Magarill and R.F. Klevtsova, *Sov. Phys. Crystallogr.*, 16 (1972) 645.

1-(*p*-Methylphenyl) ethyldioxy-bis-(dimethylglyoximato) pyridine cobalt(III)

($P2_1/c$) $R = 9.0\%$ for 2726 observed reflections. In this cobaloxime molecule, the axial sites are occupied by a pyridine molecule and by the group p-$CH_3C_6H_4CH$-$(CH_3)O_2$—. The O—O distance is 1.451 and Co—O 1.897 Å. Co—N (ring) are 1.867, 1.897, 1.890 and 1.884, and Co—N (pyridine), 2.013 Å. The cobalt atom is slightly out of the plane of the ring.

A. Chiaroni and C. Pascard-Billy, *Bull. Soc. Chim. Fr.*, (1973) 781.

Bis(5-phenyltetrazolato-bis(triphenylphosphine)palladium

The phenyltetrazole ligand is essentially planar, the angle between the five-membered ring and phenyl group being only 3.9°. The ligand is virtually orthogonal to the coordination plane defined by the palladium phosphorus and coordinated nitrogen atoms.

C. Spencer and R. Mason, *Z. Naturforsch. B*, 27 (1972) 745.

$Ba_3(CrO_4)$

$R3m$. There are isolated tetrahedra of O^{2-} surrounding Cr^{5+} and octahedra surrounding Ba^I. Ba^{II} has a different complicated coordination.

H.J. Mattausch and H.K. Muller-Buschbaum, *Z. Naturforsch. B*, 27 (1972) 739.

Salts of the nickel(II) complex, C-rac-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane $[Ni(tet^b)]^{2+}$, in the α^-, β^- and γ^- configurations

α, ($Pbca$) $R=7.1\%$ for 1536 reflections. β, ($Pna2$) $R=3.9\%$ for 1698 reflections. γ, ($P2_1/a$) $R=7.5\%$ for 1184 reflections. All are coordinatively planar and thus strained with respect to the preferred conformation attained by coordination of

the ligand to adjacent octahedral sites.
N.F. Curtis, D.A. Swann and T.N. Waters, *J. Chem. Soc., A*, (1973) 1963.

μ-Oxo-bis[bis-(2-methyl-8-hydroxyquinolinate)iron(III)]-chloroform
($P\bar{1}$) $Z=2$, $R=12.6\%$ for 2289 independent reflections. Fe—O—Fe is 151.6° and with this geometry a pair of 2-methyl-8-hydroxyquinolinato ligands adopt a parallel arrangement. The mean magnetic susceptibilities are discussed in terms of a spin—spin coupling model involving two spin-free iron(III) ions.
F.E. Mabbs, V.N. McLachlan, D. McFadden and A.T. McPhail, *J. Chem. Soc. Dalton*, (1973) 2016.

Bis[hydroxo-(triphenyl)arsonium(V)]dodeca-μ-chloro-hexachloro-octahedrohexaniobate(2-)
($P2_1/n$) $Z=2$, $R=13\%$ for 2326 reflections. The asymmetric unit is a $[Ph_3AsOH]^+$ cation and one-half of a centrosymmetric $[(Nb_6Cl_{12})Cl_6]^{2-}$ anionic cluster unit. Bond lengths within the cation agree well with previously determined values for similar species. The point symmetry of the cluster does not differ significantly from *Oh*, the octahedron of niobium atoms (Nb—Nb, 3.01_6 Å) being surrounded by the usual two sets of axial (6) and edge (12) chlorine atoms.
R.A. Field, D.L. Kepert, B.W. Robinson and A.H. White, *J. Chem. Soc. Dalton*, (1973) 1858.

Dicarbonyl-π-cyclopentadienyl[tetrakis(pyrazol-1-yl)borato] molybdenum
($C2/c$) $Z=8$, $R=9.2\%$ for 1926 independent reflections. The tetrakis-(1-pyrazolyl)-borate group acts as a bidentate ligand while the remainder of the molybdenum coordination sphere consists of two σ-bonded CO groups and the π-bonded C_5H_5-group.
E.H. Holt and S.L. Holt, *J. Chem. Soc. Dalton*, (1973) 1893.

Bis(*N*-hexyl-7-methylsalyldiminato) copper(II)
($P2_1/c$) $Z=2$, $R=10.2\%$ for 1390 independent reflections. The configuration around the copper is square planar. The dihedral angle between the mean planes of the benzene and chelate rings is 2.5°. The coordination plane is tilted 38° from the benzene, 37° from the salicylaldimine and 15° from the *n*-hexyl planes.
P.C. Jain and V.K. Syal, *J. Chem. Soc. Dalton*, (1973) 1908.

Heptacarbonyl-μ_3-diphenylacetylene-μ(1,2,3,4-tetraphenylbutadiene 1,4-diyl)-triangulo-triosmium
($P\bar{1}$) $Z=2$, $R=7.8\%$ for 4053 non zero reflections. The molecule is built up by a cluster of three osmium atoms at the corners of a nearly equilateral triangle and by two organic ligands; the osmium atoms are bonded to two or three carbonyl groups while the chelation between Ph_4C_4 and the cluster is attained via σ-bonds and the donation of π-electrons of the osmacyclo-pentadienyl ring, Ph_2C_2 appears to be a four-electron donor on the whole cluster.
G. Ferraris and G. Gervasio, *J. Chem. Soc. Dalton*, (1973) 1933.

Ammonium uranyl trioxalate

($C2/c$) $Z=4$, $R=7.2\%$ for 1287 reflections. The crystal contains NH_4^+ and $UO_2\cdot(C_2O_4)_3^{4-}$ ions. The latter have two bidentate oxalate groups forming five-membered rings with U–O, 2.43 Å and one such group forming a four-membered ring with U–O, 2.57 Å. The geometry around the uranium ion is approximately hexagonal bipyramidal.

R.W. Alcock, *J. Chem. Soc. Dalton*, (1973) 1610.

Ammonium uranyl dioxalate

($P1$) $Z=2$, $R=13.0\%$ for 1351 reflections. The crystal contains NH_4^+ and $[(UO_2)(C_2O_4)_2]_n^{2n-}$ ions. In the latter one oxalate group is bidentate and one bidentate to one uranium and unidentate to another. U–O(uranyl) 1.77; U–O (oxalate) 2.37 Å.

R.W. Alcock, *J. Chem. Soc. Dalton*, (1973) 1614.

Ammonium diuranyl oxalate

($P2_1/n$) $Z=2$, $R=11.3\%$ for 886 reflections. The crystal contains NH_4^+ and $[(UO_2)_2(C_2O_4)_3]_n^{2n-}$ ions. In the latter one oxalate group is ter- and one quadridentate giving double chains parallel to C. Mean U–O, 1.77 (uranyl) and 2.38 Å (oxalate).

N.W. Alcock, *J. Chem. Soc. Dalton*, (1973) 1616.

Hexakis(triphenylphosphine oxide) dimercury(I) bis perchlorate

($P1$) $Z=1$ (dimer), $R=12\%$ for 1696 independent reflections. The best formulation for the dimeric unit is $[(Ph_3PO)_3Hg{-}Hg(Ph_3PO)_3]\,[ClO_4]_2$. Hg–Hg is 2.522_2 Å. The centrosymmetric cationic species is discrete and the mercury environment is approximately tetrahedral Hg–O, 2.29 to 2.43 Å. There is one inverse relationship between the Hg–O, distance and the Hg–Hg–O angle.

D.L. Kepert, D. Taylor and A.H. White, *J. Chem. Soc. Dalton*, (1973) 1658.

The β form of tetra-arsenic trisulphide, As_4S_3

($Pnma$) $Z=4$, $R=9\%$ for 438 reflections. The structure is built of As_4S_3 molecules similar to those observed for α-dimorphite.

H.J. Whitfield, *J. Chem. Soc. Dalton*, (1973) 1737.

Tetra-arsenic tetraselenide

($P2_1/n$) $Z=4$, $R=11\%$ for 1008 reflections. The structure is isostructural with α-realgar As_4S_4. As–As, 2.565; As–Se, 2.39 Å.

T.J. Bastow and H.J. Whitfield, *J. Chem. Soc. Dalton*, (1973) 1739.

Tetra-arsenic pentasulphide

($P2_1/m$) $Z=2$, $R=11\%$ for 613 reflections. The structure consists of discrete As_4S_5 molecules of approximate C_{2v} symmetry; As–As, 2.55 Å and means As–S, 2.24 Å.

H.J. Whitfield, *J. Chem. Soc. Dalton*, (1973) 1740.

Trichlorotris(diethyl phenylphosphine)rhodium(III)

($P2_1/c$) $Z=4$, $R=6.7\%$ for 3037 independent reflections. The complex is monomeric and the metal atom has a somewhat distorted octahedral coordination with two pairs of like ligands mutually *trans*. Rh—P are 2.325 to 2.415 and Rh—Cl, 2.362 to 2.429 Å.

A.G. Skapski and F.A. Stephens, *J. Chem. Soc. Dalton* (1973) 1789.

Sulphatobis(thiourea)tin(II)

($P2_12_12_1$) $Z=4$, $R=8.3\%$ for 1023 reflections. The tin atoms are in distorted pyramidal sites surrounded by two sulphate oxygen atoms at 2.41 and 2.24 Å and by two thiourea sulphur atoms at 2.60 and 2.86 Å. The thiourea groups are planar but not equivalent and are linked to the sulphate ions by hydrogen bonding.

J.D. Donaldson, D.G. Nicholson, D.C. Puxley and R.A. Howie, *J. Chem. Soc. Dalton*, (1973) 1810.

The 2:1 adduct between 1,3,5-trinitrobenzene and bis(*N*-methylsalicylaldiminato)-copper(II)

($P2_1/c$) $Z=2$, $R=7.2\%$ for 1972 reflections. The complex shows the familiar stepping feature and the copper atom completes a distorted but centrosymmetrical octahedral stereochemistry by interaction with an oxygen atom of two symmetry related nitro groups at 3.08 Å. The two species are arranged alternately so that each donor interacts with two acceptors and vice versa.

A.W. King, D.A. Swann and T.N. Waters, *J. Chem. Soc. Dalton*, (1973) 1819.

trans-Tetrachlorobis(triethylphosphine) tin(IV)

($P2_1/n$) $Z=2$, $R=9.9\%$ for 1206 reflections. The centrosymmetric molecules have bond angles at tin within 1.3° of 90°; Sn—P is 2.615_5; Sn—Cl are 2.445_5 and 2.455_5 Å. The Mössbauer spectrum is discussed.

G.G. Mather, G.M. McLaughlin and A. Pidcock, *J. Chem. Soc. Dalton*, (1973) 1823.

2',2'-Bipyridyldichloro(trimethyl)tantalum(V)

($P2_1/n$) $Z=2$, $R=8\%$ for 762 independent reflections. In each individual molecule the tantalum atom has a distorted capped trigonal prismatic environment, with one chlorine atom in the unique capping position (2.540_{10} Å). The nitrogen atoms (2.291_{21} Å), one chlorine, and one methyl group form the capped quadrilateral face with two methyl groups (2.24_4 and 2.16_6 Å) occupying the remaining edge.

M.G.B. Drew and J.D. Wilkins, *J. Chem. Soc. Dalton*, (1973) 1830.

(A) Hexacarbonyl-*trans*-6a,12a-dihydro-octalene dichromium(0), (B) tricarbonyl 1,4-dihydrophenanthrene chromium(0) and (C) tricarbonylphenanthrene chromium(0)

(A), ($P1$) $Z=1$, $R=10.3\%$ for 693 reflections. (B), ($P2_1/n$) $Z=4$, $R=8.4\%$ for 1456 reflections. (C), ($P2_1/c$) $Z=4$, $R=5.0\%$ for 1898 reflections. (A) has C_i symmetry with two $Cr(CO)_3$ moieties bound to the triene fragments of the dihydro octalene ring. In (B) the $Cr(CO_3)$ is symmetrically bound to a benzene fragment of 1,4-dihydrophenanthrene. The results for (C) are compared with those obtained for an orthorhombic modification.

J.M. Guss and R. Mason, *J. Chem. Soc. Dalton*, (1973) 1834.

cis-Fluoro-(1,1,1,3,3,3-hexafluoro-isopropyl)bis(triphenyl-phosphine) platinum, $PtF[CH(CF_3)_2](PPh_3)_2$

($P2_1/a$) $Z=4$, $R=6.3\%$ for 1937 reflections. The platinum atom is in a square planar environment with two adjacent sites occupied by triphenylphosphine groups, the other two by fluorine and a hexafluoro-isopropyl group. Pt–F is 2.218_7 and Pt–$CH(CF_3)_2$ is 2.310_7 Å.

J. Howard and P. Woodward, *J. Chem. Soc. Dalton*, (1973) 1840.

Tetramethylammonium hexachlorodigallate(II), $(Me_4N)Ga_2Cl_6$

($Pa3$) $Z=4$, $R=7.6\%$ for 384 observed reflections. The analysis confirms the existence of a $Ga_2Cl_6^{2-}$ ion, of symmetry D_{3d}, with a Ga–Ga bond of 2.39 Å.

K.L. Brown and D. Hall, *J. Chem. Soc. Dalton*, (1973) 1843.

(A) *μ*-Oxalato-di[bisethylenediamine nickel(II)] dinitrate, (B) *μ*-Oxalato-bis[di-(2-aminoethyl)amine copper(II)]diperchlorate and (C) *μ*-Oxalato-bis[di-(3-aminopropyl)amine zinc(II)]diperchlorate

(A) ($P2_1/n$) $Z=2$, $R=10.5\%$ for 1936 reflections. (B) ($Pbc2_1$) $Z=4$, $R=10.1\%$ for 1948 reflections. (C) ($Pna2_1$) $Z=4$, $R=9.0\%$ for 1688 reflections. In each complex the cation is dimeric with the planar oxalate group acting as a bridge by forming five-membered chelate rings with two metal centres. The coordination geometries are; octahedral in (A), square pyramidal in (B) and trigonal bipyramidal in (C).

N.F. Curtis, I.R.N. McCormick and T.N. Waters, *J. Chem. Soc. Dalton*, (1973) 1537.

Fluoro-[6,6′,6″-phosphinidynetris-(pyridine-2-carbaldehyde oximato)(3-)]borato-(1–) cobalt(1+)tetrafluoroborate-acetonitrile

($Aba2$) $Z=8$, $R=10.5\%$ for 1126 independent non-zero reflections. The cation has approximate C_{3v} symmetry, with the central Co^{II} atom coordinated to six nitrogen atoms, mean Co–N(aldoximo) 2.063_{18} and Co–N(pyridyl) 2.118_{28} Å. The coordination sphere of the $cobalt^{II}$ atom defines a slightly tapered trigonal prism, with the N(aldoximo)···N(pyridil) bite 2.512_{30} to 2.598_{29} Å.

M.R. Churchill and A.H. Reis, *J. Chem. Soc. Dalton*, (1973) 1570.

Bis(aminomethanesulphonato) copper(II)

($P2_1/n$) $Z=2$, $R=5.3\%$ for 555 independent reflections. The crystals are formed from *trans*-bis(aminomethanesulphonato)copper(II)chelates at centres of symmetry, linked into a three dimensional array by hydrogen bonds and the long copper–sulphonate–oxygen contacts which give copper a distorted octahedral

coordination sphere (Cu–O, 1.995 and 2.506; Cu–N, 1.999 Å).
T.S. Cameron, C.K. Prout, F.J.C. Rossotti and D. Steele, *J. Chem. Soc. Dalton*, (1973) 1590.

Bis(adeninium)*trans*-bis(adenine)tetra-aquocobalt(II) bis(sulphate)hexahydrate
($P2_1/n$) $Z=2$, $R=6.0\%$ for 2275 reflections. The structure contains the centrosymmetric $[Co(H_2O)_4(adenine)_2]^{2+}$ ion, adeninium and sulphate ions, and molecules of water of solvation, held together by an intricate network of hydrogen bonds. In the complex cation the cobalt atom has octahedral coordination, and the coordinated adenine is unidentate Co–N(9) is 2.164 Å. Co–H_2O are 2.073 and 2.114 Å.
P. de Meester and A.C. Skapski, *J. Chem. Soc. Dalton*, (1973) 1596.

Di-μ-carbonyl-*cis*-μ-(1-5-η:1'-5'-η-dicyclopentadienyldimethyl-silane)bis(carbonyliron)-(Fe–Fe)
($P2_1/n$) $Z=4$, $R=7.2\%$ for 1309 independent reflections. The silicon atom is tetrahedrally σ-bonded to two methyl groups and to two cyclopentadienyl rings; the latter in turn are each π-bonded to one iron atom. The two iron atoms are bonded to one another (2.51 Å) and are also linked by two symmetrical carbonyl bridges and each iron also carries one terminal carbonyl making the overall coordination approximately octahedral.
J. Weaver and P. Woodward, *J. Chem. Soc. Dalton*, (1973) 1439.

Dichloro[bis(diphenylphosphino)-ethylamine] palladium(II)
($Pbca$) $Z=8$, $R=10.9\%$ for 2735 reflections. Pd–P, 2.22; Pd–Cl, 2.37; P–N, 1.72 Å; P–Pd–P is 71.4° and P–N–P is 97.7°. The palladium, phosphorus and chlorine atoms very nearly lie in a plane.
J.A.A. Mokuolu, D.S. Payne and J.C. Speakman, *J. Chem. Soc. Dalton*, (1973) 1443.

Di-μ-chloro-tetrakis-(4-methylpenta-1,3-diene) dirhodium(I)
($I2/a$) $Z=4$, $R=4.3\%$ for 1473 independent reflections. In the dimeric molecule the environment of each metal atom is square planar, the cocrdination plane being defined by two bridging chlorine atoms and the mid-point of the less substituted C=C double bonds of two 1,3-diolefins. Rh···Rh is 3.090_3 Å, Rh–Cl are 2.410_3 and 2.402_3 Å. The coordinated C=C bonds are not symmetric with respect to the coordination plane.
M.G.B. Drew, S.M. Nelson and M. Sloan, *J. Chem. Soc. Dalton*, (1973) 1484.

Bis[(trimethylsilyl)methyl]bis-(2,2'-bipyridyl) chromium(III)iodide
($P2_1/c$) $Z=4$, $R=5.7\%$ for 3758 reflections. The *cis*-octahedral organometallic cation has an approximate two-fold symmetry axis. Cr^{III}–C(sp^3) is 2.107_{12} Å, Cr–N (*trans* to carbon), 2.156_7 and Cr–N(*trans* to nitrogen) is 2.103_7 Å.
J.J. Daly, F. Sanz, R.P.A. Sneedon and H.H. Zeiss, *J. Chem. Soc. Dalton*, (1973) 1497.

1,2-bis-[(2-aminobenzylidene)-amino] propanato(2−) copper(II)

($P2_12_12_1$) Z=4, R=7.6% for 1068 reflections. There is an umbrella shaped molecule with a disordered 1,2-propane bridge, which is indicative of electronic rather than steric demands. The copper atom is four coordinate and mean Cu–N is 1.91 Å.

D. Hall, T.N. Waters and P.E. Wright, *J. Chem. Soc. Dalton*, (1973) 1508.

(A) Bis(pyrrolidonecarbodithioato) nickel(II) and (B) copper(II)

(A) ($P\bar{1}$) Z=1, R=10% for 857 reflections. (B) ($P\bar{1}$) Z=1, R=10% for 953 independent reflections. The structure of (A) consists of centrosymmetric molecules, the metal atom being planar coordinated by four sulphur atoms at 2.217_2, and 2.201_2 Å (2.342_2 and 2.345_2 in (B)). There is no evidence in the copper derivative for dimeric formation by intermolecular metal sulphur interaction.

P.W.G. Newman, C.L. Raston and A.H. White, *J. Chem. Soc. Dalton*, (1973) 1332.

Tetrachloro-di-μ_3-oxo-tetra-μ-propoxo-tetraoxodipropoxotetramolybdenum (2Mo–Mo)

($P\bar{1}$) Z =1, R=6.3% for 1366 independent reflections. The two molybdenum atoms in the asymmetric unit have different environments although both are formally seven coordinate, being bonded to each other (2.669_2 Å), to one chlorine (2.395_5, 2.367_6) and five oxygens. The metal–metal bond has little effect on the geometries of the coordination polyhedra of the molybdenum atoms which are best described as distorted octahedra. These four octahedra in the tetramer are bridged through edges.

J.A. Beaver and M.G.B. Drew, *J. Chem. Soc. Dalton*, (1973) 1376.

Two five-coordinate cobalt(II) complexes with an N_4P donor set

(A) Co(nnp)$(NCS)_2$ when nnp is *N* [(diphenyl-phosphino)ethyl-*N'*-diethylenediamine ($P\bar{1}$) Z=2, R=7.4% for 1437 reflections. (B) Co(nnpMe)$(NCS)_2$ where nnpMe is the methyl derivative of the nnp ligand. In both complexes the cobalt atom is five coordinated by the two nitrogen and the phosphorus atoms of the ligand and the two nitrogen atoms of the thiocyanate groups. The two coordination polyhedra can be described as distorted trigonal bypyramids that of (A) tending towards an elongated square pyramid, that of (B) towards the tetrahedron.

A.B. Orlandini, C. Calabresi, C.A. Ghilardi, P.L. Orioli and L. Sacconi, *J. Chem. Soc. Dalton*, (1973) 1383.

Acetylacetonato-C-*meso*-(5,5,7,12,12,-14-hexamethyl-1,4,8,11-tetra-azacyclotetradecane) nickel(II) perchlorate

(*Cc*) Z=4, R=6.3% for 1580 independent reflections. The nickel(II) ion is in approximately octahedral coordination with the macrocycle folded at N(4) and N(11) to occupy four adjacent coordination sites, a symmetrical, planar acetylacetonatochelate occupying the two remaining sites. In the structure, the config-

uration of the amine is 1RS, 4RS, 7RS, 8RS, 11RS, 14RS.
N.F. Curtis, D.A. Swann and T.N. Waters, *J. Chem. Soc. Dalton*, (1973) 1408.

Dicesium-μ-(ethylenediaminetetra-acetato)-di-μ-sulphido-bis [oxomolybdate(V)]-dihydrate
(*Pnam*) Z=4, R=5.9% for 2196 reflections. The hexadentate ethylenediaminetetra-acetate (EDTA) ligand coordinates to each molybdenum atom through two carboxylato oxygens and a nitrogen while two bridging sulphur atoms and a terminal oxygen complete the coordination sphere around each molybdenum. The short Mo—Mo bond of 2.799_1 Å suggests that the coordination geometry around each metal atom may be described as a distorted capped octahedron sharing a common edge determined by the two sulphur atoms.
B. Spivack and Z. Dori, *J. Chem. Soc. Dalton*, (1973) 1173.

Cyclo-bis-(μ-acetato-μ-nitrosyl)-bis[di-μ-acetato-diplatinum(II)]
($C2/c$) Z=4, R=6.6% for 1521 reflections. The molecule is tetranuclear with the four platinum atoms forming an approximate rectangle of sides 2.955 and 3.311 Å. Each of the two independent metal atoms is bonded to three oxygen atoms from bridging acetate groups and to one nitrogen atom from a bridging nitrosyl group to give a square planar coordination. The mean Pt—N of 1.91 and N—O, 1.22 and the sp^2 geometry at the bridging nitrogen are all consistent with the formulation of the nitrosyl group as $N{-}O^-$.
P. de Meester and A.C. Skapski, *J. Chem. Soc. Dalton*, (1973) 1194.

Chloro-[1,6-bis(2′-pyridyl)-2,5-diazahexane] copper(II) chloride
($P2_1/c$) Z=4, R=10.2% for 1246 independent reflections. The cation is five coordinate with approximate square pyramidal geometry. One of the pyridyl nitrogens occupies the axial position with Cu—N 2.21 Å. The other bonds are normal although there are distortions of the coordination polyhedron resulting from ligand constraints and some distortions of bond angles in the chelating ligand itself.
N.A. Bailey, E.D. McKenzie and J.U. Worthington, *J. Chem. Soc. Dalton*, (1973) 1227.

Tris-(1,10-phenanthroline)copper(II) perchlorate
($C2/c$) Z=8, R=13.3% for 2662 reflections. The monomeric complex cations exhibits a distorted octahedral configuration about copper(II) with equal distortions of the axial Cu—N bonds; mean Cu—N(eq) 2.04_1, mean Cu—N(ax) 2.33_1 Å.
O.P. Anderson, *J. Chem. Soc. Dalton*, (1973) 1237.

1,1,3,3,5,5-Hexaphenylcyclotriborataphosphoniane $[(Ph_2P)BH_2]_3$
($P\bar{1}$) Z=2, R=7.5% for 5940 independent reflections. The six membered boron—phosphorus ring has the chair formation with a close approach to symmetry *Cs*(*m*). The torsion angles in the ring lying in the range 40—55°. Mean distances are B—P, 1.948; B—H, 1.11; B→P π bonding.
G.J. Bullen and P.R. Mallinson, *J. Chem. Soc. Dalton*, (1973) 1295.

Di-μ-dimethylstannylene-bis(carbonyl-π-cyclopentadienyl cobalt)

($P\bar{1}$) $Z=2$, $R=8.5\%$ for 1520 independent reflections. Sn and Co atoms alternate around a planar four membered ring, the Sn atoms having only slightly distorted tetrahedral coordination, and Co having octahedral coordination if the cyclopentadienyl ring is assumed to occupy three sites. Co—Sn is 2.542_2; Sn—Co—Sn is 78° mean Sn—C is 2.20_1 Å.

J. Weaver and P. Woodward, *J. Chem. Soc. Dalton*, (1973) 1060.

Bis-[di-μ-(phenylmethoxo)-bis(pentane-2,4-dionato)dicopper(II)]

($P2_1/c$) $Z=8$, $R=10.1\%$ for 1691 independent reflections. The unit cell contains two centrosymmetric tetrameric molecules, each consisting of two approximately planar benzoxy-bridged dimers (acac)Cu(μ-$OCH_2Ph)_2$Cu(acac) joined together by four apical Cu-O bonds involving one benzoxy and one acetylacetonate (acac) oxygen atom of each dimer, of lengths 2.42 Å (benzoxy) and 2.71 Å (acac). The Cu_2O_2 unit in the dimer is planar, with mean bond lengths 1.88_1 to the three and 1.94_1 Å to the four coordinate oxygen atom.

J.E. Andrew and A.B. Blake, *J. Chem. Soc. Dalton*, (1973) 1102.