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Tris(triphenylphosphine)nitrosyl iridium, $Ir(NO)(PPh_3)_3$

($P3$) $R = 4.75\%$ for 1929 independent reflections. The absolute configuration of the crystal sample is R, S, S. The molecule has C_3 symmetry. Ir–N is 1.67(2), Ir–P is 2.31(1) and N–O is 1.24(3) Å.

V.G. Albano, P. Bellon and M. Sansoni, *J. Chem. Soc. A*, (1971) 2420.

Bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato)bis(*N,N*-dimethylethylenediamine)-copper(II)

($P\bar{1}$) $R = 0.052$ for 2777 independent reflections. The Cu–NMe_2 distance is 2.064(3) Å, Cu–NH_2 is 1.989(3) Å. Both en-type ligands are in the *gauche* conformation. Cu–O is 2.793(2). The copper is in an approximately octahedral environment with oxygens axial.

M.A. Bush and D.E. Fenton, *J. Chem. Soc. A*, (1971) 2446

Bis[dinitratobis(pyridine)copper(II)]-pyridine, $[Cu(NO_3)_2(py)_2]_2py$

($P2_1/c$) $R = 10.4\%$ for 2386 reflections. The molecule is centrosymmetric, with the copper surrounded by seven other atoms in a distorted arrangement. The long Cu–O distances are adjacent to each other and the position of pyridine in the unit cell suggests that it is important for stability of the structure.

A.F. Cameron, K.P. Forrest, D.W. Taylor and R.H. Nuttall, *J. Chem. Soc. A*, (1971) 2492

Dimethyltin bisfluorosulphate, $Me_2Sn(SO_3F)_2$

($P2_1/c$) $R = 3.6\%$ for 643 observed reflections. The tin is octahedrally coordinated to two methyl groups, Sn–C is 2.08(1) and four oxygens Sn–O are 2.24(1) Å. Fluorosulphate groups ($\sim C_{3v}$) bridge between linear dimethyl tin units.

F.H. Allen, J.A. Lerbscher and J. Trotter, *J. Chem. Soc. A*, (1971) 2507

Trichloro(π-*trans*-but-2-en-1,4-diammonium)platinum(II)chloride, $[PtCl_3(C_4H_{14}N_2)]Cl\frac{1}{2}H_2O$

($C2/c$) $R = 3.7\%$ for 2447 independent reflections. In the cation, platinum is four-coordinate with three chlorines and the double bond of the diammonium cationic ligand.

R. Spagna and L. Zambonelli, *J. Chem. Soc. A*, (1971) 2544

Dichloro(diphenyl)tin
($P\bar{1}$) R = 4.0% for 3800 reflections. Mean bond distances are Sn–Cl 2.346(2), Sn–C 2.114(3), C–C 1.387(13) Å. Angle Cl–Sn–Cl = 100°, Cl–Sn–C = 107° and C–Sn–C = 125.5°. The Mossbauer spectrum is discussed.
P.T. Greene and R.F. Bryan, *J. Chem. Soc. A*, (1971) 2549

Blue form of the 1,2 addition complex of copper(II) dichloroacetate with α-picoline
($P2_1/n$) R = 9.4% for 261 reflections. The copper atom is surrounded by four atoms in a distorted square planar array. Cu–O = 1.999(7) and 2.017(7), Cu–N = 1.990(10) and 2.032(10) Å. The α-picolines are mutually *trans*, with the α-methyls of the rings *cis* with respect to the copper.
G. Pavey and F.S. Stephens, *J. Chem. Soc. A*, (1971) 2577

Cyclopentadienyl(trimethyl)platinum(IV), (π-C_5H_5)$PtMe_3$
($Pna2_1$) R = 7.5% for 767 independent reflections. The platinum atom lies above a point approximately in the centre of the five-membered ring. Pt–C(Me) = 2.11, Pt–C(π-C_5H_5) = 2.32 Å.
G.W. Adamsen, J.C.J. Bart and J.J. Daly, *J. Chem. Soc. A*, (1971) 2616

1:1 Complexes of 1,3,5-trinitrobenzene and bis(*N*-*t*-butylsalicylideneiminato)-cobalt(II), nickel(II) and copper(II)
The complexes are essentially isomorphous and coordination around metal is a flattened MO_2N_2 tetrahedron with angles between M, N(1), O(1) and M, N(2) and O(2) planes of 81°34′, 78°51′ and 61°36′ for Co, Ni and Cu structures.
E.E. Castellano, O.J.R. Hodder, C.K. Prout and P.J. Sadler, *J. Chem. Soc. A*, (1971) 2620

Dipotassium pentafluoromanganate(III) hydrate
($P2_1/m$) R = 9.3% for 663 reflections. The manganese atoms are linked through *trans* bridging fluorine atoms to give endlessly linked chains parallel to the *b* axis, with distorted octahedral coordination of manganese atoms. M–F = 1.82, 1.84 and 2.07 Å.
A.J. Edwards, *J. Chem. Soc. A*, (1971) 2653

Ferrocenylmethyl(dimethyl)ammonium tetrachloro-zincate monohydrate
$[C_5H_4FeC_5H_4 \; CH_2 \; NHMe_2^+]_2 ZnCl_4^{2-} \; H_2O$
($P2_1/c$) R = 6.8% for 2012 observed reflections. Mean bond distances are Fe–C = 2.040, C–C(ring) = 1.43 Å. The structure confirms the formulation of the compound as the tetrachlorozincate. The cp rings are nearly eclipsed with iron sandwiched between.
C.S. Gibbons and J. Trotter, *J. Chem. Soc. A*, (1971) 2659

Chlorobis(π-cyclopentadienyl)(triphenylsilyl)zirconium(IV)
($P2_1/c$) R = 5.8% for 2488 intensities. The coordination of silicon is approximately

tetrahedral as is that of the zirconium if the cp ligands are regarded as unidentate, Zr–Cl is 2.430(3), Zr–Si is 2.183(2) Å.
K.W. Muir, *J. Chem. Soc. A*, (1971) 2663.

Di-iodotris(2-diphenylphosphino-ethyl)amine cobalt(II), $CoLI_2$ ($L = N(CH_2 \cdot CH_2 \cdot PPh_2)_3$)
($P2_1/c$) $R = 7.9\%$ for 2103 independent reflections. The metal atom is in a distorted square pyramidal coordination with one phosphorus atom at the apex, the other two with nitrogen and iodine atoms form the base. Factors responsible for stability of this complex are discussed.
C. Mealli, P.L. Orioli and L. Sacconi, *J. Chem. Soc. A*, (1971) 2691.

N,N'-Ethylenebis(salicylideneiminato)ethylcobalt(III)
($P2_1/c$) $Z = 4$, $R = 8.5\%$ for 1762 independent reflections. The cobalt atoms are in a distorted octahedron, the axial positions are occupied by oxygen of the other Co(salen) unit and by the σ-bonded ethyl group. Co–C = 1.990(7), Co–O equatorial salen = 1.901(5), 1.935(4) and Co–N = 1.886(6) Å. The structure is made up of centrosymmetric dimeric units.
M. Calligaris, D. Minichelli, G. Nardin and L. Randaccio, *J. Chem. Soc. A*, (1971) 2720.

Trisodium phosphinoformate hexahydrate, $Na_3PO_3CO_2 \cdot 6H_2O$ (two independent determinations)
($P2_1/n$) $Z = 4$, $R = 10.68\%$ for 2238 reflections, ($P2_1/n$) $Z = 4$, $R = 12.7\%$ for 1550 reflections. Sodium atoms are surrounded octahedrally by oxygen atoms from water and phosphinoformato ions. The octahedra share one edge. P–C = 1.869 Å mean. In general the two sets of determined atomic coordinates are in good agreement.
R.R. Nagri, P.J. Wheatley and E. Foresti-Serantoni, *J. Chem. Soc. A*, (1971) 2751.

Tris(tetraethylammonium) tris(1,2-dicyanoethylene-1,2-dithiolato)indate(III)
($P2_1/c$) $Z = 4$, $R = 7.4\%$ for 1379 observed reflections. The indium atom is coordinated to a distorted octahedron of surrounding sulphur atoms. In–S = 2.585(6)–2.626(8) Å.
F.W.B. Einstein and R.D.G. Jones, *J. Chem. Soc. A*, (1971) 2762.

Osmium pentafluoride
($P2_1/c$) $Z = 8$, $R = 10.6\%$ for 642 independent reflections. The compound is isostructural with ruthenium pentafluoride. The structural unit is a tetramer with osmium atoms at the corners of a rhombus. The fluorine atoms are hexagonal close-packed and bridge between osmium atoms.
S.J. Mitchell and J.H. Holloway, *J. Chem. Soc. A*, (1971) 2789.

The blue isomer of oxodichlorotris(dimethylphenylphosphine)molybdenum(IV), *cis-mer*-$[MoCl_2(PMe_2Ph)_3]$

(*Pbca*) $Z = 8$, $R = 5\ 7\%$ for 2261 intensities The structure is made up of monomers and molybdenum is in a distorted octahedral coordination sphere Mo–O = 1 676(7), Mo–Cl = 2 551(3) (*trans* to O), 2 464(3) (*trans* to P), Mo–P = 2 54(3), 2 558(3) (*trans* to P) and 2 500(3) Å (*trans* to Cl)
L Manojlovic-Muir, *J Chem Soc A*, (1971) 2796

Potassium dioxotetracyanorhenate, $K_3[ReO_2(CN)_4]$
($P\bar{1}$) $Z = 1$, $R = 14\ 5\%$ for 670 independent reflections The $[ReO_2(CN)_4]^{3-}$ ion contains octahedral rhenium Re–C = 2 115 and 2 132 Å, C–N = 1 148 and 1 164 Å; ReO = 1 77 Å
R H Fenn, A J Graham and N P Johnson, *J Chem Soc A*, (1971) 2880

[1,2-Bis(dimethylarsino)trifluoroethane] tetracarbonyl molybdenum, $(Me_2AsCHF\ CF_2AsMe_2)Mo(CO)_4$
(*Pbca*) $Z = 4$, $R = 5\ 2\%$ for 1757 reflections The molybdenum is in a slightly distorted octahedral coordination Mo–As = 2 563(2), 2 589(2) Å, As–Mo–As = 81 9(1)° The five-membered ring is non-planar As–C–C–As dihedral angle is 22° C–C is 1 30(2), C–F = 1 72(3), 1 73(2) Å (axial) and 1 38(2), 1 33 Å (equatorial) Angle C–C–F is 85 and 82°
I W Nowell and J Trotter, *J Chem Soc A*, (1971) 2922

Bis(pyridine-2,3-dicarboxylato)silver(II) dihydrate
($P2_1/n$) $Z = 2$, $R = 6\ 7\%$ for 864 reflections The structure consists of $Ag(C_7H_4NO_4)_2$ molecules Ag–N = 2 12(1) Å, Ag–O = 2 13(1) Å The ketonic oxygen atoms interact weakly (2 98 Å) with silver atoms in adjacent molecules which gives the silver atoms a tetragonally distorted octahedral coordination environment
M G B Drew, R W Matthews and R A Walton, *J Chem Soc A*, (1971) 2959

cis-[Carbonatobis(trimethylenediamine)cobalt(III)] perchlorate
($P2_1/c$) $Z = 4$, $R = 9\ 2\%$ for 1347 observed reflections The cobalt–tn rings are not related by molecular symmetry and have different, slightly distorted chain conformations The distortions and conformational isomers are discussed in terms of strain energy
R J Gene and M R Snow, *J Chem Soc A* (1971) 2981

Di-μ-carbonyl-nonacarbonyl-(π-cyclopentadienyl-rhodio)tri-iron (3*Rh*–*Fe*)(3*Fe*–*Fe*), $(\pi\text{-}C_5H_5)RhFe_3(CO)_{11}$
($P2_1/n$) $Z = 4$, $R = 6\ 98\%$ for 1227 independent non-zero reflections The molecule consists of three $Fe(CO)_3$ groups and a $(\pi\text{-}C_5H_5)Rh$ moiety, connected by metal–metal bonds and two bridging [Rh–(CO)–Fe] carbonyl groups
M R Churchill and M V Veidis, *J Chem Soc A*, (1971) 2995

Dipotassium *catena*-μ-fluoro-difluoro-μ-sulphato-manganate(III), $K_2MnF_3SO_4$
($Pnab$) $Z = 4$, $R = 6 4\%$ for 390 reflections Manganese atoms in distorted octahedral coordination, are linked through *trans* bridging fluorine atoms and sulphate groups to form an infinite chain along a Mn–F(terminal) = 1 82, Mn–F(bridge) = 2 04 and Mn–O = 2.01 Å
A.J Edwards, *J Chem. Soc. A*, (1971) 3074.

(A) Nickel(II) and (B) zinc(II) bis(trithioperoxybenzoates)
(A), ($P2_1/c$) $Z = 2$, $R = 7 1\%$ for 1097 reflections, (B) ($C2/c$) $Z = 4$, $R = 8 7\%$ for 1347 reflections The two structures are very similar except nickel is planar in (A) and zinc is tetrahedral in (B) The shortening of Ni–S bonds but not of Zn–S bonds relative to other sulphur-containing complexes is discussed
M Bonamico, G Dessy, V Fares and L Scaramuzza, *J Chem Soc A*, (1971) 3191

Dichlorobis(N,N'-diethylthiourea)zinc(II)
($P2_1/c$) $Z = 8$, $R = 8 2\%$ for 4035 independent reflections The structure consists of two independent molecules of different shapes which form polymeric chains and dimers
M Bonamico, G Dessy, V Fares and L Scaramuzza, *J Chem Soc A*, (1971) 3195.

Dicarbonyl-π-cyclopentadienyliodo[tri(n-butyl)phosphine] molybdenum(II)
($P4_2/n$) $Z = 8$, $R = 11 1\%$ for 938 independent reflections The molecule has a sandwich-type structure with the cyclopentadienyl ring on one side of the molybdenum The other ligands are roughly square planar and lie on the other side The carbonyls are mutually *cis*
R H Fenn and J H Cross, *J Chem Soc A*, (1971) 3312

(A) Bis(methoxyacetato)bis(pyridine)copper(II) tetrahydrate and (B) bis(phenoxyacetato)aquobis(pyridine)copper(II)
(A) ($P\bar{1}$) $Z = 1$, $R = 10\%$ for 1002 independent reflections, (B) ($P2_12_12_1$) $Z = 4$, $R = 8 9\%$ for 1412 independent reflections. (A) The copper is octahedrally coordinated with elongation in the direction of the Cu–OMe bonds (2 36 Å) (B) The copper atom is in a five-coordinate square pyramid with the aquo ligand at the apex and *trans* pyridine and unidentate phenoxyacetate at the base
C.K Prout, M J. Barrow and F J.C Rossotti, *J Chem Soc A*, (1971) 3326

Bis(methoxyacetato)tetrabis(imidazole)copper(II)
($P2_1/a$) $R = 9 2\%$ The copper atom is in a distorted octahedron with apical oxygens of two methoxyacetates at 2 825 Å Cu–N = 1 913 and 2 045 Å (one pair of each). Hydrogen bonding and location of protons are discussed
C K Prost, G B Allison and F J C Rossotti, *J. Chem. Soc. A*, (1971) 3331

(A) Copper(II) meso-tartrate trihydrate and (B) copper(II) *d*-tartrate trihydrate

(A) ($Pbca$) $Z = 8$, $R = 3.5\%$ for 1190 independent reflections. Meso-tartrate ligands from two chelate rings link distorted octahedral crystallographically independent, copper atoms into chains. (B) ($P2_1/a$) $Z = 4$, $R = 10\%$ for 1195 independent reflections. Copper is surrounded by six oxygen atoms, four from two chelate tartrate rings, one from water and one from a carbonyl group of a related dimeric unit.

C.K. Prout, J.R. Carruthers and F.J.C. Rossotti, *J. Chem. Soc. A*, (1971) 3336

(A) Copper(II) hydrogen maleate tetrahydrate and (B) copper(II) maleate hydrate

(A) ($I2/m$) $Z = 2$, $R = 3.9\%$ for 753 independent reflections. The copper atoms are coordinated only by water molecules as in $[Cu(H_2O)_4]_n^{2n+}$. These chain cations are hydrogen bonded to planar hydrogen maleate ions. $Cu-OH_2$ (bridging) = 1.959 and 2.682 Å, $Cu-OH_2$ (non-bridging) = 1.933 Å. (B) ($P2_1$) $Z = 2$, $R = 9.6\%$ for 628 independent reflections. The copper is in a square pyramid of coordinating water molecules, $Cu-OH_2$ = 2.25 Å, chelating maleate ligand and two oxygen atoms from two other maleate groups. Cu–O (average-basal) = 1.99 Å

C.K. Prout, J.R. Carruthers and F.J.C. Rossotti, *J. Chem. Soc. A*, (1971) 3342

Copper(II) phthalate monohydrate

($B2/b$) $R = 4.9\%$. Two types of copper have distorted octahedral coordination to two water molecules and four phthalate groups. Each phthalate group is bonded to four copper atoms through three of its oxygen atoms. Cu–O = 1.962 (mean of two water and two phthalate oxygens) and 2.462 Å (two phthalate oxygens). Water molecules bridge copper as do phthalate groups.

C.K. Prout, J.R. Carruthers and F.J.C. Rossotti, *J. Chem. Soc. A* (1971) 3350

Di-μ-[1,2-bis(dimethylarsino)tetrafluorocyclobutene]-octacarbonyltetrahedrotetracobalt, $C_{24}H_{24}As_4Co_4F_8O_8$

($C2/c$) $Z = 4$, $R = 3.4\%$ for 1057 observed reflections. The cobalt atoms form a tetrahedral cluster and each arsine ligand forms a bridge between two cobalt atoms. Each cobalt is bonded to three other cobalts and to three other atoms, thus forming a distorted octahedral coordination sphere. The two arsenic atoms and cyclobutene ring are coplanar. Co–Co = 2.446, 2.510, 2.343, Co–As = 2.307(2) and 2.242

F.W. Einstein and R.D.G. Jones, *J. Chem. Soc. A*, (1971) 3359

Di[(benzenesulphinato)-μ-hydroxo-phenylstannio]-tetracarbonyl di-π-cyclopentadienyl-di-iron

Previously formulated as $(\pi\text{-}C_5H_5)Fe(CO)_2Sn(Ph)(OSOPh)(OH)$, this compound is shown to be a centrosymmetric dimer with two five-coordinate tin atoms linked by a dihydroxo bridge. Sn–O = 2.054(2), 2.231(2) Å. O–Sn–O = 70.8(1)°, Sn–O–Sn = 109.2(1)°, Sn–Fe = 2.499(1) Å. ($P2_1/c$) $Z = 2$, $R = 2.4\%$ for 2592 independent reflections.

R. Restivo and R.F. Bryan, *J. Chem. Soc. A*, (1971) 3364.

Dinitratobis(pyridine)zinc(II), $(C_5H_5N)_2Zn(NO_3)_2$

($P2_1/n$) $Z = 4$, $R = 10\%$ for 1044 independent reflections The molecule is quasi-tetrahedral and contains an asymmetric unidentate nitrato group

A F Cameron, D W Taylor and R H Nuttall, *J Chem Soc A*, (1971) 3402.

Di-μ-carbonyl-heptacarbonyltris[(dimethyl)phenyl-phosphine]-*triangulo*-tri-iron, $Fe_3(CO)_9(PMe_2Ph)_3$

($Pbca$) $Z = 8$, $R = 11.4\%$ for 1394 independent reflections The molecule contains the $Fe_3(CO)_{12}$ skeleton Three phosphine molecules, one attached to each iron, are arranged such that the three phosphorus atoms are coplanar with the triangle of iron atoms

G Raper and W S McDonald, *J Chem Soc A*, (1971) 3430

$H_2FeRu_3(CO)_{13}$

($P2_1/a$) $R = 9\ 5\%$ for 1934 unique reflections Each molecule comprises a tetrahedron of three Ru atoms and one Fe atom, with three terminal carbonyl groups on each Fe There are two asymmetric carbonyl bridges between Fe and Ru, and Fe–C are from 1.68 to 1 86 Å, Ru–C 2 18 to 2 45(6) Å, Ru–Ru 2 78 to 2 92, Fe–Ru 2.63 to 2 70(1) Å, Ru–C (terminal) = 1 82(8), Fe–C (terminal) = 1.72(5) Å Mean C–O = 1 18(8) Å

C J Gilmore and P Woodward, *J Chem Soc A*, (1971) 3453

trans-Bis(methyldiphenylphosphine)-(σ-pentachlorophenyl)-(σ-pentafluorophenyl)-nickel(II)

($C2/c$) $Z = 4$, $R = 4\ 91\%$ for 1604 independent non-zero reflections The nickel atom is in a slightly distorted square planar environment The $Ni–C_6Cl_5$ bond length, 1 905(10) Å, is shorter than $Ni–C_6F_6$, 1.978(10) Å

M R Churchill and M V Veidis, *J Chem Soc A* (1971) 3463

Compound formed from tetracyanoethylene and tricarbonyl(cycloheptatriene)iron

($P\bar{1}$) $Z = 2$, $R = 8\ 1\%$ for 885 independent reflections The $Fe(CO)_3$ group is bonded to the seven-membered ring via a novel four-electron-donor system, comprising an allyl and σ-type bonds The iron is approximately octahedral and the tetracyanoethylene links to the cycloheptatriene ring at C_1 and C_6 to form a new five-membered "envelope" ring system *exo* to the $Fe(CO)_3$

J Weaver and P Woodward, *J Chem Soc A*, (1971) 3521

Mercuriobis(pentacarbonyl-manganese), $Hg[Mn(CO)_5]_2$

($P\bar{1}$) $Z = 1$, $R = 9\ 28\%$ for 1570 independent reflections The molecule has approximate $D4h$ symmetry with carbonyl groups in eclipsed configuration The manganese is almost regular-octahedral Hg–Mn = 2 806 Å

W Clegg and P J Wheatley, *J Chem Soc A*, (1971) 3572

Nonacarbonyl(cyclododecatrienetriyl)hydrate-*triangulo*-triruthenium, $HRu_3(CO)_9(C_{12}H_{15})$

$(P2_1/a)$ $Z = 4$, $R = 8\ 0\%$ for 1419 independent reflections The Ru atoms each carry three carbonyl groups and are bonded to one another at the corners of an isosceles triangle. Two equal sides are unbridged The apical atom is equidistant from and π-allyl bonded to cyclododecatrienyl ligand The outer two carbon atoms of the π allyl unit are each σ-bonded to a basal ruthenium atom The cyclododecatrienyl ligand is a five-electron dimer

A Cox and P Woodward, *J Chem Soc A*, (1971) 3599

Di-μ-chloro-bis[trichloro(thionylchloride)zirconium(IV)]

$(P2_1/n)$ $Z = 2$, $R = 7\ 9\%$ for 1273 independent reflections The molecule is a centrosymmetric dimer with each zirconium bonded to bridging chlorine atoms (2 580(10) Å), to three terminal chlorines (2 340(20) Å) and to an oxygen atom (2 273(13) Å) of the thienyl group The zirconium is distorted-octahedral

R K Collins and M G B. Drew, *J Chem Soc A*, (1971) 3610

Hexa-amminecobalt(III) pentachlorocadmate(II)

$(Fd3c)$ $Z = 32$, $R = 2\ 0\%$ for 835 reflections Co–N(unique) = 1 960(6) Å, N–Co–N = 89 51(12)° In the trigonal bipyramidal $CdCl_5{}^{3-}$, Cd–Cl(ax) = 2 526(1) and Cd–Cl(eq) = 2.56(2) Å

E F. Epstein and I Bernal, *J Chem Soc A*, (1971) 3628.

(A) Dichlorotetrakis(acetaldoxime)nickel(II) and (B) tetrakis(acetamide)bisaquonickel (II) dichloride

(A) $(P2_1/c)$ $Z = 2$, $R = 3\ 5\%$ for 1904 reflections Each nickel atom is coordinated to four acetaldoximes and two *trans* chlorines (B) $(P2_1/c)$ $Z = 2$, $R = 3\ 3\%$ for 2018 reflections Each nickel atom is bonded to four acetamides and two *trans* waters The chlorines lie outside the coordination sphere but are involved in strong intermolecular hydrogen bonding

M E Stone, B E Robertson and E Stanley, *J Chem Soc A*, (1971) 3632

Trimethylamine complexes of (A) titanium tribromide and (B) chromium trichloride

$(Pnma)$ $Z = 4$, $R = 10\ 4$ for 565 reflections (A) and $(Pnma)$ $Z = 4$, $R = 6\ 4$ for 492 reflections (B) Both compounds are monomeric and trigonal-bipyramidal five-coordinate. There are deviations from idealised trigonal symmetry of equatorial halogens, related to electron configuration

P.T Greene, B.J Russ and J S Wood, *J Chem Soc A*, (1971) 3636

Tri-μ-(dimethylgermanio)-tris-(tricarbonylruthenium), $[Ru(CO)_3(GeMe_2)]_3$

$(P6_3/m)$ $Z = 2$, $R = 7\ 2\%$ for 137 independent reflections The three germanium atoms are coplanar with the three ruthenium atoms, which form an equilateral triangle of side 2 930(10) Å Two carbonyls on each Ru atom are *trans* and almost perpendicular to the Ru_3Ge_3 plane. The third lies equatorially along the mirror line of the Ru_3 triangle The molecule has D_{3h} symmetry and is the first example of a six-membered heterocyclic ring system

J Howarth and P Woodward, *J Chem Soc A*, (1971) 3648

Tetraphenylgermane, $(C_6H_5)_4Ge$

($P\bar{4}2_1c$) $Z = 2$, $R = 3$ 9% and 5 0% for two sets of (437 and 417 reflections) data Ge–C = 1 954, C–C = 1 380 Å, C–Ge–C = 109 5° Molecular symmetry = $\bar{4}$

P C Chieh, *J Chem Soc A*, (1971) 3243

Tetraphenyl silane, $(C_6H_5)_4Si$

($P\bar{4}2_1c$) $Z = 2$, $R = 4$ 3% for 413 independent reflections Molecular symmetry $\bar{4}$ Si–C = 1 872, C–C = 1 383, C–H = 1 00 Å

C. Glidewell and G M Sheldrick, *J Chem Soc A*, (1971) 3127.

Piperidinium hexathiotetra-arsenate $[C_5H_{12}N]_2^+[As_4S_6]^{2-}$

($Imm2$) $Z = 2$, $R = 12$ 9% for 660 independent reflections As–As = 2 579(12), As–S (terminal) = 2 183(10), As–S (bridge) = 2 302(11) and 2 231(8)Å The anion has a basket-like structure with four arsenics and two sulphurs lying on crystallographic mirror planes

E J Porter and G M Sheldrick, *J Chem Soc A*, (1971) 3130

Bis-(*O*-methylphenylthiocarbamato)(triphenylphosphine)-palladium(II), $[Pd(PhN{-}C{-}(OMe)S)_2(PPh_3)]$

($P\bar{1}$) $Z = 2$, $R = 8$ 3% for 3812 independent non-zero reflections The palladium atom is in square planar coordination to two thiocarbamic ligands one bidentate N and S, the other unidentate, S The fourth bond is from sulphur Pd–S = 2 33 Pd–N = 2 09 and Pd–P = 2 23 Å

C Furlani, T Tarantelli, L Gastaldi and P Porta, *J Chem Soc A*, (1971) 3778

An unusual five-coordinated aminecarboxylic acid nickel(II) chelate, 1, 5-diazacyclooctane-*N*, *N'*-diacetatoaquonickel(II) dihydrate, $NiO_7N_2C_{10}H_{22}$

($P2_1c$) $Z = 4$, $R = 8$ 3% for 1622 unique non-zero reflections The tetradentate aminecarboxylate ligand is coordinated on a plane with a water molecule occupying an apical position to form an approximate square pyramid The sixth coordination position is blocked by a methylene hydrogen, thus forcing the unusual five-coordination

D O Nielson, M L Larson, R D Willett and J I Legg, *J Amer Chem Soc*, 93 (1971) 5079

Tetraallyldimolybdenum

$(P\bar{4}2_1c)$ $Z = 8$, $R = 6.7\%$ for 1209 independent non-zero reflections. Mo–Mo = 2.183(2). Two allyl groups bridge the metals symmetrically with their planes parallel to the Mo–Mo line. The other two allyl groups are each bonded to Mo such that the six Mo–C distances are almost equal, 2.28–2.35 Å.

F. A. Cotton and J. R. Pipal, *J. Amer. Chem. Soc.*, 93 (1971) 5441.

Octadecacarbonylhexaruthenium dihydride, $H_2Ru_6(CO)_{18}$

$(P2_1/c)$ $Z = 4$, $R = 5.72\%$ for 2780 independent non-zero reflections. The six ruthenium atoms define an octahedron, each Ru atom is associated with three terminal carbonyl ligands. Triply bridging hydride ligands are postulated in mutually *trans* sites.

M. R. Churchill and J. Wormald, *J. Amer. Chem. Soc.*, 93 (1971) 5670.

(A) Dichlorobis(2,3,5,6-tetrahaptonorbornadienedicarbonylcobalt)-tin(IV), $Cl_2Sn[Co(CO)_2C_7H_8]_2$ and (B) diphenylbis(2,3,5,6-tetrahaptonorbornadienedicarbonylcobalt)tin(IV), $(C_6H_5)_2Sn[Co(CO)_2C_7H_8]_2$

$(P2_1/c)$ $Z = 4$, $R = 4.8\%$ for 2977 reflections (A) and $(P2_1/c)$ $Z = 4$, $R = 7.8\%$ for 1965 reflections (B). The coordination at the tin is a distorted tetrahedron. The cobalt atoms have distorted trigonal bipyramidal geometries in which the norbornadiene double bonds chelate one apical and one equatorial site and carbonyls occupy the remaining two equatorial positions. Co-unsaturated diene carbon atom 2.076–2.192 Å.

F. P. Boer and J. J. Flynn, *J. Amer. Chem. Soc.*, 93 (1971) 6495.

$W_2(CO)_8H_2[Si(C_2H_5)_2]_2$

$(P2_1/n)$ $Z = 2$, $R = 3.8\%$ for 945 significant independent reflections. The structure consists of a planar $(CO)_2WSi_2W(CO)_2$ fragment with tungsten metal–metal bonding and two silicon bridges. W–Si = 2.586(5) and 2.703(4) Å. Hydrogen insertion between silicon and tungsten atoms is postulated to account for the long distance. The tungsten atoms are in pentagonal bipyramidal environments with axial bonds to two carbonyls and equatorial to two carbonyls, a silicon and a tungsten and bonding to the two-electron, three-centre W–H–Si bond.

M. J. Bennett and K. A. Simpson, *J. Amer. Chem. Soc.*, 93 (1971) 7156.

Potassium triiodidomercurate(II) monohydrate, $KHgI_3H_2O$

$(Pna2_1)$ $Z = 4$, $R = 8.1\%$ for 554 observed reflections. HgI_4 tetrahedra are linked through common corners to form infinite chains parallel to *a*. Hg–I = 2.70, 2.73, 2.83, 2.90 Å.

L. Nyovisit and G. Johansson, *Acta Chem. Scand.*, 25 (1971) 1615.

Diazidodiamminezinc(II), $Zn(N_3)_2(NH_3)_2$

$(Pnma)$ $Z = 8$, $R = 8.5\%$ for 544 independent reflections. The zinc is tetrahedral. Zn–N = 1.97–2.03. The azide groups are almost linear and appear to be asymmetric. N–N =

1 14–1.20 Å. The infrared spectrum of the compound is reported
I Agrell and N Vannerberg, *Acta Chem Scand*, 25 (1971) 1630

$Hg(OH)BrO_3$
($Pbcm$) $Z = 4$, $R = 9$ 1% The structure consists of planar zigzag $[Hg(OH)]_n^{n+}$ chains and pyramidal BrO_3^- ions Hg–O = 2 07 Å, angle O–Hg–O = 175°
G Bjornlund, *Acta Chem Scand*, 25 (1971) 1645

Mercury (I) hexafluorosilicate dihydrate
($P2_1/c$) $Z = 2$, $R = 8$ 5% for 341 independent reflections The structure contains $H_2O–Hg–Hg–H_2O$ groups. Hg–Hg is 2.495(3), Hg–O is 2 20(3) Å No deviation from a normal SiF_6 anion was found Si–F (mean) is 1 70(2) Å
E Dorm, *Acta Chem Scand*, 25 (1971) 1655.

Diimidazole silver(I) nitrate, $Ag(C_3H_4N_2)_2NO_3$
($P2_12_12_1$) $Z = 4$, $R = 3$ 9% for 1021 intensities The coordination of silver is almost linear (172 00(32)°) Ag–N = 2 120 and 2 132(8) Å
C Antti and B K S Lundberg, *Acta Chem Scand*, 25 (1971) 1758

Hexaimidazole cobalt(II) carbonate pentahydrate, $Co(C_3H_4N_2)_6CO_3(H_2O)_5$
($P6_3$) $Z = 2$, $R = 8$ 8% for 822 independent reflections. The coordination geometry of cobalt is octahedral Co–N = 2 16 Å and 2 18 Å References to other imidazole complexes are given
R Strandberg and B K S Lundberg, *Acta Chem Scand*, 25 (1971) 1767.

Hexammineruthenium(II) iodide, $[Ru(NH_3)_6]I_2$ (A) and hexammineruthenium(III) tetrafluoroborate, $[Ru(NH_3)_6][BF_4]_3$ (B)
($Fm3m$) $Z = 4$, $R = 2$ 1% for 139 unique significant reflections (A), ($Fm3m$) $Z = 4$, $R = 4$ 7% for 385 unique significant reflections (B) $Ru^{II}–NH_3$ = 2.144(4), $Ru^{III}–NH_3$ = 2 104(4) This difference together with the difference between the $Co^{II}–NH_3$ and $Co^{III}–NH_3$ bonds implies that the electrostatic effect is significantly smaller than the effect of spin change.
H C Stynes and J A Ibers, *Inorg Chem*, 10 (1971) 2304.

cis-Dichlorobis(4, 4′-dichlorodiphenyl sulphide)platinum(II), $Pt[S(p\text{-}ClC_6H_4)_2]_2Cl_2$
($P2_1/c$) $Z = 4$, $R = 4$ 2% for 4732 intensities The structure consists of discrete molecules with platinum in a tetrahedrally distorted square planar coordination. The Pt–S–Cl bond angle is 109.1 ± 0 6° Some average bond distances are Pt–S = 2 292(6) and 2 278(7); S–C = (1 8 Å), Cl–C = (1 74 Å)
W.A. Spofford, E.L. Amma and C V Senoff, *Inorg Chem.*, 10 (1971) 2309

Stannic ethylenediaminetetraacetate monohydrate, [$C_{10}H_{14}N_2O_9Sn$]

($P2_1/c$) $Z = 4$, $R = 4.1\%$ for 2943 significant reflections. Tin(IV) is coordinated by two nitrogens and four carboxylate oxygens of the ligand, and by one water to form a seven-coordinate complex. Sn–O (average) is 2.083 Å, Sn–N (average) is 2.313 Å, $Sn{-}OH_2$ = 2.124 Å.

F.P. Remoortere, J.J. Flynn and F.P. Boer, *Inorg. Chem.*, 10 (1971) 2313.

Tetraselenium bis(hydrogen disulfate), $Se_4(HS_2O_7)_2$

($P2_1/c$) $Z = 2$, $R = 6.6\%$ for 552 observed reflections. Se–Se = 2.283(4), S–O(H) = 1.56(2) Å. S–O = 1.59(2) and 1.67(2) Å. Bond angle S–O–S = 123(1)°. The Se^{4+} cation is strictly square planar.

I.D. Brown, D.B. Crump and R.J. Gillespie, *Inorg. Chem.*, 10 (1971) 2319.

Cesium 3,3′-*commo*-bis[nonahydro-1,2-dimethyl-1,2-dicarba-3-chroma-*closo*-dodecaborate] hydrate

($P4_12_12$) $Z = 4$, $R = 4.7\%$ for 1957 independent reflections. The metallocarborane anion consists of two icosahedra sharing a chromium atom as a common vertex. Each icosahedron is made up of chromium, nine borons each with a terminal hydrogen, and two carbons each with a methyl group. Average distances Cr–C, 2.27, Cr–B, 2.26, C–C(Me), 1.52, C–C(cage), 1.64, C–B, 1.72, and B–B, 1.77 Å.

D. St. Clair, A. Zalkin and D.H. Templeton, *Inorg. Chem.*, 10 (1971) 2587.

Tris(1,2-dimethylhydrazino)diphosphine, $P_2(NCH_3)_6$

($P2_1/c$) $Z = 2$, $R = 8.7\%$ for 261 observed reflections. The molecule consists of three 1,2-dimethylhydrazino groups linking two phosphorus atoms.

W. Van Dorne, G.W. Hunt, R.W. Perry and A.W. Cordes, *Inorg. Chem.*, 10 (1971) 2591.

Dibromotetrakis(5-methylpyrazole)manganese(II), $Mn(C_4H_6N_2)_4Br_2$

($P\bar{1}$) $Z = 1$, $R = 4.5\%$ for 2590 significant reflections. The manganese coordination sphere is approximately D_{4h} with four nitrogens of the 5-methylpyrazole ligands and two bromine atoms.

J. Reedijk, B.A. Stork-Blaisse and G.C. Verschoor, *Inorg. Chem.*, 10 (1971) 2594.

π-Triphenylcyclopropenylchlorodipyridinenickel(0)-pyridine, $[\pi\text{-}C_3(C_6H_5)_3NiCl(C_5H_5N)_2]\cdot C_5H_5N$

($P2_1/c$) $Z = 4$, $R = 8.1\%$ for 2361 significant reflections. The triphenylcyclopropenyl group is assumed to occupy one coordination position so that the nickel is described as distorted tetrahedral. $Ni{-}C_3(C_6H_5)_3$ = 1.896(8) and 1.968(8) Å, i.e. π-bonding.

R.M. Toggle and D.L. Weaver, *Inorg. Chem.*, 10 (1971) 2599.

Hexamminechromium(III) pentabromocuprate(II) $[Cr(NH_3)_6]$ $[CuBr_5]$ (A) and hexamminechromium(III) tribromodichlorocuprate(II) $[Cr(NH_3)_6]$ $[CuBr_3Cl_2]$ (B)

(A) ($Fd3C$) $Z = 32$, $R = 3$ 1% for 189 independent significant reflections. Angle N–Cr–N = 89 6(2)°, Cr–N = 2 059(6) Å The anion is a regular trigonal bipyramid Axial bond length 2 4500(22) and equatorial 2 5191(17) Å. (B) The crystal is found to have random disorder of bromine and chlorine atoms between axial and equatorial positions

S A Goldfield and K N Raymond, *Inorg Chem*, 10 (1971) 2604.

Bis(phenoxyacetato) triaquocopper(II)

($P\bar{1}$) $Z = 4$, $R = 8$ 5% for 2185 reflections The structure is made up of monomeric pentacoordinate molecules linked by hydrogen bonds into sheets perpendicular to C The copper coordination is distorted square pyramidal to two monodentate phenoxyacetates and three waters Cu–O = 1954(16)–2 280(17) Å

C V Goebel and R J Doedens, *Inorg Chem*, 10 (1971) 2607

Diazido-μ-1, 2-bis(diphenylphosphino)ethane-bis(1, 2-bis(diphenylphosphino)ethane)-dicopper(I), $Cu_2(N_3)_2$ $[(CH_2P(C_6H_5)_2)_2]_3$

($Pbca$) $Z = 4$, $R = 6$ 5% for 1136 non-zero reflections The molecule is centrosymmetric about a point located between the methylene carbons of a bridging diphos ligand. The distorted tetrahedron around copper is completed by a bidentate diphos and an azide N–N (azide) is 1 196 and 1 076 Å

A P. Gaughan, R F Ziolo and Z Dori, *Inorg Chem*, 10 (1971) 2776

Octasulfur bis(hexafluoroarsenate), $S_8(AsF_6)_2$

($P2_1/c$) $Z = 8$, $R = 6$ 7% for 1975 reflections The configuration of the S_8^{2+} cation is that of a folded ring with approximate Cs symmetry having an *endo–exo* conformation S–S (average) = 2 04 Å Average As–F in the approximately octahedral AsF_6^- is 1 67 Å

C G Davies R J Gillespie, J J Park and J Passmore, *Inorg Chem*, 10 (1971) 2781

Tetraammine platinum(II)-μ-oxo-bis[oxotetracyanorhenium(V)], $[ORe(CN)_4ORe(CN)_4O]^{4-}$

($C2/m$) $Z = 4$, $R = 3$ 2% for 1729 significant independent reflections The two independent $Pt(NH_3)_4^{2+}$ ions are planar Pt–N (average) is 2 051(9)Å. The anion is binuclear with linear OReORe and cyanide ligands completing an octahedron around each rhenium Re–C (av) is 2 120(7) Å, Re–O (bridge) is 1 915 Å and (terminal) 1.698(7) Å

R Shandles, E O Schlemper and R K Murmann, *Inorg Chem*, 10 (1971) 2785.

Triindenyluranium chloride, $U(C_9H_7)_3Cl$
($P2_12_12_1$) $Z = 4$, $R = 4.7\%$ for 2685 reflections. Uranium is almost symmetrically bonded to the carbon atoms of the five-membered ring of each of thee planar indenyl rings at 2.67–2.89 Å and also to one chloride at 2.953 Å in a tetrahedral array.
J.H. Burns and P.G. Laubereau, *Inorg. Chem.*, 10 (1971) 2789

Cesium heptafluorodiantimonate(III), $CsSb_2F_7$
($C2/c$) $Z = 4$, $R = 2.0\%$ for 1019 observed reflections. The geometry of $Sb_2F_7^-$ is that of two trigonal bipyramids (including equatorial lone pairs) joined by an axial corner. Sb–F–Sb is 125.3°. The overall structure agrees well with electron pair repulsion theory predictions.
R.R. Ryan, S.H. Mastin and A.C. Larson, *Inorg. Chem.*, 10 (1971) 2793

Tellurium tetrachloride
($C2/c$) $Z = 16$, $R = 6.1\%$ for 2948 independent reflections. The structure consists of isolated Te_4Cl_{16} molecules having a cubane-like structure of approximate T_d symmetry. Te and Cl occupy alternate corners. Te–Cl (average) is 2.311 Å. The coordination of Te is a distorted octahedron. Te–Cl (bridge av.) is 2.929 Å. The structure data suggest concentration of non-bonding Te electrons towards the centre of the cubane skeleton.
B. Buss and B. Krebs, *Inorg. Chem.*, 10 (1971) 2795

trans-Bis(triphenylphosphite)tetracarbonylchromium(0)
($P2_1/c$) $Z = 2$, $R = 5.6\%$ for 2066 significant reflections. The coordination geometry of the chromium is octahedral with four carbonyl groups, Cr–C = 1.88(1) and two *trans* phosphorus atoms, Cr–P = 2.252(1).
H.S. Preston, J.M. Stewart, A.J. Plastus and S.O. Grim, *Inorg. Chem.*, 11 (1972) 161.

Bis(cyclopentadienyl)-2,2′-bi-π-allyl-bis(nickel), $C_5H_5 \cdot Ni \cdot C_3H_4C_3H_4 \cdot Ni \cdot C_5H_5$
($P2_1/n$) $Z = 2$, $R = 3.47\%$ for 1387 reflections. The cyclopentadienyl ring consists of an allyl group, C–C = 1.398 and 1.394 Å and a bond of 1.401 separated by two distances of 1.438 and 1.423 Å. Observed partial localisation of cyclopentadienyl ring bonds is discussed in terms of extended Huckel M.O. calculations.
A.E. Smith, *Inorg. Chem.*, 11 (1972) 165

A dimeric π-cyclooctatetraene dianion cerium(III) complex, $[Ce(C_8H_8)Cl \cdot 2OC_4H_8]_2$
($P2_1/c$) $Z = 2$ (dimers), $P = 2.95\%$ for 2626 independent reflections. The two cerium atoms in the dimeric unit are bridged asymmetrically by two chloride ions, Ce–Cl = 2.855(2) and 2.935(2) Å. The cyclooctatetraene ring is planar (C–C = 394 Å) and is symmetrically bonded to cerium (Ce–C (average) is 2.710(2), Ce–O (av.) is 2.583(2) Å). The two THF oxygens and two chlorine atoms form a plane parallel to and below the COT ring.
K.O. Hodgson and K.N. Raymond, *Inorg. Chem.*, 11 (1972) 171

Trichloro(1,10-phenanthroline)thallium(III), $TlCl_3(phen)$

$(P\bar{1})$ $Z = 2$, $R = 6.1\%$ for 4250 reflections. The thallium atom is in a distorted octahedral environment in which the nitrogen atoms of the ligand occupy two positions, the rest being occupied by chlorines.

W. J. Baxter and G. Gafner, *Inorg. Chem.*, 11 (1972) 176

Dichlorobis(triphenylphosphine oxide)copper(II)

$(Fdd2)$ $Z = 8$, $R = 4.4\%$ for 1386 independent non-zero reflections. The coordination geometry of the copper is that of an elongated tetrahedron. Cu–Cl = 2.170(2) and Cu–O = 1.958(4) Å. Cu–O–P is 150.9(3)°

J. A. Bertrand and A. R. Kalyanaraman, *Inorg. Chim. Acta*, 5 (1971) 341

Dichloro-(acetonethiosemicarbazone)zinc(II)

$(P2_1/n)$ $Z = 4$, $R = 4.5\%$ for 1322 reflections. The zinc atom is in a distorted tetrahedral environment, Zn–Cl = 2.203(2), 2.252(2) Å, Zn–S = 2.303(2), Zn–N = 2.116(4) Å. The difference in Zn–Cl distances results from hydrogen bonding involving only one of the chlorine atoms.

M. Mathew and G. J. Palenik, *Inorg. Chim. Acta*, 5 (1971) 349.

α-Chloro(ethylenediamine)(dipropylenetriamine)cobalt(III) iodide monohydrate, α-$[Co(en)(dpt)Cl]I_2 \cdot H_2O$

$(Pna2_1)$ $Z = 4$, $R = 6.6\%$ for 468 reflections. The cobalt is in octahedral coordination with one chlorine and five nitrogen atoms. The three nitrogen atoms of dpt are in one plane plus one en nitrogen. The other en nitrogen is *trans* to chlorine. Strain energy minimisation calculations are discussed.

D. A. House, P. R. Ireland, I. E. Maxwell and W. T. Robinson, *Inorg. Chim. Acta*, 5 (1971) 397

Bis-(μ-acetato)hexacarbonyldiosmium, $(CH_3CO_2)_2Os_2(CO)_6$

$(P2_1/c)$ $Z = 8$, $R = 9.2\%$ for 3919 independent reflections. The structure is made up of dinuclear units. Two independent molecules are identical, with the coordination around each osmium being roughly octahedral. Os–Os is 2.73(2) Å.

J. G. Bullitt and F. A. Cotton, *Inorg. Chim. Acta*, 5 (1971) 406

A π-arene complex of uranium(III)–aluminium chloride, $UAl_3Cl_{12} \cdot C_6H_6$

$(P\bar{1})$ $Z = 2$, $R = 10.9\%$ for 4268 independent reflections. The structure is made up of a uranium atom coordinating three $AlCl_3$ tetrahedra through U–Cl–Al bonds, and a benzene ring. Uranium is linked to five chlorines in a plane while the sixth chlorine and the benzene molecule constitute two apical groups.

M. Cesari, U. Pedretti, A. Zazzetta, G. Lugli and W. Marconi, *Inorg. Chim. Acta*, 5 (1971) 439

Bis(thiocarbohydrazide-*N*, *S*)cadmium dichloride, $Cd[S{=}C(NH{-}NH_2)_2]_2Cl_2$
($P2_1/c$) $Z = 2$, $R = 9.17\%$ for 934 independent reflections. The structure consists of *trans* octahedral complexes in which the ligand forms five-membered chelate rings with N and S as donor atoms. Cd–N = 2.34(3), Cd–S = 2.59(1) and Cd–Cl = 2.73(1) Å.
F. Bigoli, A. Braibanti, A. M. M. Lanfredi, A. Tiripicchio and M. T. Camellini, *Inorg. Chim. Acta*, 5 (1971) 392.

$Co_3(CO)_9Se$ (A), $FeCo_2(CO)_9Se$ (B) and $FeCo_2(CO)_9Te$ (C)
($C\bar{1}$) $Z = 4$, $R = 4.3\%$ (A), ($C\bar{1}$) $Z = 4$, $R = 3.0\%$ (B) and ($C\bar{1}$) $Z = 4$, $R = 2.3\%$ (C). The interrelationship of crystal packing is discussed in terms of X-ray and ESR measurements. The study provides quantitative information of distribution of valence electrons in an organometallic cluster system. The molecular structures may be described as tetrahedrally shaped M_3X cluster systems formed by the symmetrical coordination of an apical chalcogen atom X to a basal $M_3(CO)_9$ fragment containing three $M(CO)_3$ groups situated at the corners of an equilateral triangle and linked by metal–metal bonds.
C. E. Strouse and L. F. Dahl, *J. Amer. Chem. Soc.*, 93 (1971) 6032.

7,7′-*commo*-Bis[dodecahydro-7-nickela-*nido*-undecaborate] dianion,
$[(CH_3)_4N]_2Ni(B_{10}H_{12})_2$
($P2_1/c$) $Z = 2$, $R = 9.6\%$ for 1056 independent reflections. The nickel atom bonds to eight boron atoms and fuses two atom polyhedral fragments giving two chemically different Ni–B bonds of 2.23 and 2.15 Å. All hydrogens were located and extended Huckel calculations are discussed.
L. J. Guggenberger, *J. Amer. Chem. Soc.*, 94 (1972) 114.

Dimethylsulphoxide(pentammine)ruthenium(II) hexafluorophosphate,
$[Ru(NH_3)_5{-}DMSO]^{2+}\ 2(PF_6^-)$
($P2_1/c$) $Z = 4$, $R = 5.4\%$ for 1985 significant reflections. The DMSO ligand coordinates through the sulphur atom. S–O = 1.527(7), Ru–S = 2.188(3) Å. Coordination about the Ru atom is roughly octahedral.
F. C. March and G. Ferguson, *Can. J. Chem.*, 49 (1971) 3590.

Dichlorobis(4-methylpyridine)zinc(II), $Zn(4\text{-}CH_3Py)_2Cl_2$
($P2_1/c$) $Z = 4$, $R = 4.3\%$ for 1348 observed reflections. The zinc atom is tetrahedrally coordinated. A large Cl–Zn–Cl angle 121.8(1)° is attributed to repulsions between non-bonding electrons on adjacent chlorine atoms. Zn–N = 2.046(5) and 2.042(5), Zn–Cl = 2.211(2) and 2.04(2) Å.
H. Lynton and M. C. Sears, *Can. J. Chem.*, 49 (1971) 3418.

$(PPh_3)_2PtO_2 \cdot 2CHCl_3$
($C2/c$) $Z = 4$, $R = 6.6\%$ for 2740 independent reflections. The molecule has a two-

fold axis of symmetry with the O–O normal to the axis O–O = 1.505(16), Pt–O = 2.006(7) Å.

P.T. Cheng, C D. Cook, S C Nyburg and K Y Wan, *Can J Chem*, 49 (1971) 3772

catena-Di-μ-fluorodifluorodiaquohafnium(IV)

Dimorphic forms were characterised – only one structure was successfully ascertained The coordination number of the metal is eight with antiprismatic or distorted dodecahedral stereochemistry One water is not coordinated but is held between the chain-like complexes by hydrogen bonds

D. Hall, C E F Rickard and T N Waters, *J Inorg Nucl Chem*, 33 (1971) 2395

Dimethyl *N*-phenylbenzimidate dimer, $[(CH_3)_2AlOC(C_6H_5)N(C_6H_5)]_2$

$(P2_1/c)$ $Z = 2$, $R = 10\ 7\%$ for 1096 non-zero reflections. The dimeric molecule is composed of a centro-symmetrical eight-membered ring The aluminum atoms are bridged by OCN groups The coordination geometry of the aluminum is a distorted tetrahedron Al–O = 1 805(6), Al–N = 1 947(7), Al–C = 1 940(11) and 1 940(11) Å The N and C atom environments are trigonal The two adjacent benzene rings are *cis* with respect to the N–C bond

Y. Kai, N Yasuoka, N. Kasai and M Kahudo, *J Organometal Chem*, 32 (1971) 165

1,6-Dichloro-2,3-trimethylene-4,5-bis(pyridine)platinum(IV), $[PtCl_2(C_5H_5N)C_3H_6]$

$(P2_1/c)$ $Z = 4$, $R = 9\ 4\%$ for 1119 independent reflections The platinum atom is roughly octahedral with the outer two carbon atoms occupying *cis* positions and being σ-bonded to platinum The other two positions in this plane are occupied by pyridine nitrogen atoms, with chlorine *trans*, making up the octahedron NMR and IR data are correlated with the structure.

R.D. Gillard, M. Keeton, R. Mason, M.F. Pilbrow and D.R. Russel, *J. Organometal. Chem.*, 33 (1971) 247

Two pyridinium propylide complexes of platinum(II) and platinum(IV), $C_{13}H_{16}Cl_2N_2Pt$ (A) and $C_{13}H_{16}Cl_4N_2Pt \cdot CHCl_3$ (B)

$(P2_1/c)$ $Z = 8$, $R = 9\%$ for 1560 independent reflections (A) and $(P2_1/c)$ $Z = 4$, $R = 8\ 9\%$ for 1913 independent reflections (B) Apart from a conformational difference, the geometries of the two crystallographically non-equivalent platinum(II) ylide complexes are identical Evidence for the formation of the carbon-bonded ligand as a pyridinium propylide complex rather than a carbene complex is unequivocal

M Keeton, R Mason and D R Russell, *J Organometal Chem*, 33 (1971) 259

Bis(1,3-cyclohexadiene)monocarbonyliron, $(C_6H_8)_2FeCO$

$(P2_12_12_1)$ $Z = 4$, $R = 3\ 3\%$ for 1697 non-zero reflections. The rings are arranged around the central atom with the saturated part pointing to the carbonyl group. The central iron atom is five-coordinated with one carbonyl group and two of the butadiene groups of the 1,3-cyclohexadiene moiety as ligands

C. Kruger and Y. Tsay, *J Organometal Chem.*, 33 (1971) 59

(Tri(cyclohexyphosphine)nickel)dinitrogen

($P2_1/n$) $Z = 4$, $R = 11\%$ for 5977 reflections The molecule contains a linear Ni—N—N—Ni system in which the nitrogen molecule is enclosed in a cage formed by four of the cyclohexyl rings N—N is 1 12 Å The nickel atoms have planar trigonal-bipyramidal geometry with somewhat distorted bonding angles to the phosphorus atoms

P W Jolly, K. Jonas, C. Kruger and Y -H Tsay, *J Organometal Chem*, 33 (1971) 109

μ-Chloro-chloro-di-π-allyl(cyclohexanone oxime)dipalladium

($P\bar{1}$) $Z = 2$, $R = 8.6\%$ for 3060 independent reflections The complex has a binuclear structure with one palladium atom coordinating to a π-allyl group and two chlorines in a square-planar fashion, the other coordinates to one chlorine, a π-allyl group and the nitrogen atom of the oxime

Y Kitano, T Kajimoto, M Kashiwagi and Y Kinoshita *J Organometal Chem.*, 33 (1971) 123

Trimethyl(quinuclidine)aluminum, $(CH_3)_3Al \cdot NC_7H_{13}$

($P2_1/n$) $Z = 2$, $R = 11$ 3% for 675 independent reflections. The aluminium atom is approximately tetrahedrally coordinated Al—N = 2 06 Å. The methyl groups are in staggered configuration with respect to the methylene groups of the quinuclidine molecule.

C D. Whitt, L M Parker and J L. Atwood, *J Organometal Chem*, 32 (1971) 291

$Rb_4[Ni_2(CN)_6]$

($P\bar{1}$) $Z = 4$, $R = 6$ 9% for 1624 reflections The structure consists of dimeric $[Ni_2(CN)_6]^{4-}$ surrounded by 16 Rb cations The monomeric $Ni(CN)_3$ units of the complex are held together by strong metal—metal bonding. Ni—Ni = 2.32 and 2.29 Å

O Jarchow, *Z Anorg Allg Chem*, 383 (1971) 40

$[Co_3(CN)_2[(OH)_4](NH_3)_8][Co_2(NO_2)_6[(OH)_2]NO_2]$ H_2O

($P2_1/n$) $Z = 4$ The crystals contain trinuclear cations in which three Co^{III} atoms are bound to two pairs of oxygen atoms At the central Co atom there are two CN^- ligands in the *cis* position The anion is a binuclear Co^{III} complex The two Co atoms are bound to two OH^- and to one NO_2^- group

K. Weighardt, J Weiss and H Siebert, *Z Anorg Allg Chem.*, 383 (1971) 151.

Pyruvylidene(glycinato)aquocopper(II) dihydrate, $[\mathrm{Cupyv = gly(H_2O)}]\ 2H_2O$
($P2_1/c$) $Z = 4$, $R = 12\%$ for 1658 independent reflections. The copper atom is in an axially distorted octahedron The C=N distance is longer than normal. C=N = 1 310, C–N = 1 434 and Cu–N = 1 917 Å
A Torii, H. Tamura-Kogayashi, K Ogawa and T Watanabe, *Z Kristallogr., Kristallgeometrie, Krystallphys, Kristallchem*, 133 (1971) 179

Bis(π-cyclopentadienyl)molybdenum(IV) benzene-1,2-dithiolate, $(C_6H_5)_2MoS_2C_6H_4$
(*Pnma*) $Z = 4$, $R = 6\ 0\%$ Mo–S = 2 440 Å, S–C (benzene) = 1 78 Å, C–C = 1 35–1 43 (benzene) and 1 37–1 42 (Cp), Mo–C = 2.29–2 36
A Kutoglu, *Z Kristallogr Kristallgeometrie, Kristallphys, Kristallchem*, 132 (1971) 437

Bis-pinenyl nickel(0), $Ni(C_{10}H_{17})_2$
($P2_1/c$) $Z = 2$ The bonding is of a π-allyl nature. No further details are given
C Kruger, *Z Kristallogr, Kristallgeometrie, Kristallphys, Kristallchem*, 132 (1971) 436.

Zinc chloride triethanolamine, $ZnCl\ C_6H_{14}ON$
($P2_1/c$) $Z = 4$
H Follner, *Z Kristallogr, Kristallgeometrie, Kristallphys, Kristallchem*, 132 (1971) 428

trans-Bis(dinitrogen)bis[1,2-bis(diphenylphosphino)ethane] molybdenum(0), $Mo(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$
($P\bar{1}$) $Z = 1$, $R = 14\ 8\%$ for 3253 reflections Molybdenum occupies the centre of symmetry and four phosphorus atoms and two nitrogen atoms (*trans*) form a distorted octahedron There is no significant lengthening of the N–N bond on coordination The Mo–N–N fragment is slightly bent
T Uchida, Y Uchida, M Hidai and T Kodama, *Bull Chem Soc Jap*, 44 (1971) 2883

$(+)_{589}$-Dicyanobis(ethylenediamine)cobalt(III) chloride monohydrate, $(+)_{589}$-$[Co(CN)_2en_2]Cl\ H_2O$
($P2_1$) $Z = 2$, $R = 11\ 4\%$ for 860 independent reflections The absolute configuration is $\Lambda(\lambda\lambda)$ The complex ion has an approximate two-fold axis, both en chelate rings are of the *ob* configuration
K Matsumoto, S Ooi and H Kuroya, *Bull Chem Soc Jap*, 44 (1971) 2721

2 3 complex of zinc phthalocyanine and *n*-hexylamine
($P2_1/c$) $R = 15\%$ for 3000 reflections The phthalocyanine molecule is not planar and the central square pyramidal zinc is displaced 0 48 Å from the plane towards the amine nitrogen
T Kobayashi, T Ashida, N Uyeda, E Suito and M Kakudo, *Bull Chem Soc Jap*, 44 (1971) 2095

Bis(1,3-diaminopropane)copper(II) thiocyanate
(*C*2/*c*) $Z = 4$, $R = 6.1\%$. The copper atom is coordinated to four nitrogen atoms from two 1,3-diaminopropane molecules. Cu–N = 2.029(5), 2.005(5), and two thiocyanate sulphur atoms Cu–S = 3.154(2) Å, forming an elongated square pyramid. The chelate ring has a chair conformation.
G. D. Andreetti, L. Cavalca and P. Scarabotto, *Gazz. Chem. Ital.*, 101 (1971) 483

Di-*catena*-di-μ-chloromonochloromono(1,3-diaminopropane) cadmium(II)
($Pna2_1$) $Z = 4$. The cadmium atom coordinates octahedrally to four chlorine atoms and two nitrogens. Cd–Cl = 2.494(2), 2.754(2) and 2.742(2) Å. Cd–N = 2.292(9) and 2.289(9) Å. The structure consists of double chains of octahedra, sharing two edges. The chelate ring has a chain conformation.
G. D. Andreetti, L. Cavalca, M. A. Pellinghelli and P. Scarabotto, *Gazz. Chim. Ital.*, 101 (1971) 488

Perchlorate-bis(2,2′-bipyridine)-copper(II) perchlorate, $[Cu(ClO_4)(bipy)_2]ClO_4$
($P\bar{1}$) $Z = 2$, $R = 13.4\%$. The copper atom is coordinated in a tetragonally distorted octahedron. The four (bipy) nitrogen atoms are arranged in a flattened tetrahedron. The polar positions are occupied by perchlorate oxygen atoms. Cu–N = 1.99 Å (average), Cu–O = 2.45 and 2.73 Å.
H. Nahai, *Bull. Chem. Soc. Jap.*, 44 (1971) 2412

cis-Dichloro(meso-2,3-diaminobutane)palladium(II)
(*Pbca*) $Z = 8$, $R = 3.8\%$ for 1628 independent reflections. The palladium atom is square-planar coordinated. Two *cis* chlorines are Pd–Cl = 2.316 and 2.318 Å. The five-membered chelate ring is puckered. The molecule is dissymmetric and two enantiomeric molecules exist in the crystal. Pd–Pd = 3.34 Å.
T. Ito, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 27 (1971) 1695.

$Cu_3(NH_3)_3(CN)_4$, a mixed-valence complex
(*Cmca*) $Z = 8$, $R = 5.5\%$ for 2588 reflections. Each Cu^{II} atom is coordinated to three NH_3 molecules distributed over four positions and to the nitrogen atoms of two cyanide groups in a distorted octahedron. The Cu^{I} atoms have a distorted trigonal planar coordination with the carbon atom of the ordered cyanide group and the ends of two disordered cyanide groups. The Cu^{II} and four Cu^{I} atoms form the apices of a nearly planar pentagon with cyanide groups bridging the copper atoms.
R. J. Williams, D. T. Cromer and A. C. Larson, *Acta Crystallogr., Sect. B*, 27 (1971) 1701

Racemic μ-amido-μ-sulfato-bis[bis(ethylenediamine)cobalt(III)] tribromide, DL-$[(en)_2Co\ \mu(NH_2\ SO_4)\ CO(en)_2]Br_3$
($P2_1/c$) $Z = 4$, $R = 6.6\%$ for 2944 independent reflections. The endocyclic S–O bonds (1.493 Å (average)) appear to be longer than the exo S–O (1.442 Å and S–O in free

sulphate The cation has C_2 symmetry The configurations of the two cation antipodes are ${}^{\delta}_{\delta}\Lambda\lambda\Lambda^{\delta}_{\delta}$ and ${}^{\lambda}_{\lambda}\Delta\delta\Delta^{\lambda}_{\lambda}$

U. Thewalt, *Acta Crystallogr., Sect B,* 27 (1971) 1745

3-(2-Diethylammoniumethoxy)-1, 2-benzisothiazole tetrachlorocuprate, $(C_{13}H_{19}N_2OS)_2^{+}[CuCl_4]^{2-}$

($C2/c$) $Z = 4$, $R = 8\ 5\%$ for 3532 independent reflections The organic ion shows no direct coordination to copper which forms a tetrachlorocuprate ion with a flattened tetrahedral structure Cu–Cl = 2 259(3) and 2 220(2)

A.C Bonamartini, M Nardelli, C Palmieri and C Pelizzi, *Acta Crystallogr, Sect B,* 27 (1971) 1775.

Potassium tricyanomethide, $KC(CN)_3$

($P\bar{1}$) $Z = 2$, $R = 11\%$ for 457 reflections The tricyanomethide group has approximate D_{3h} symmetry C–C (average) = 1 39(2) Å, C–N (average) = 1 17(1) Å The potassium is surrounded by seven N atoms at 2 86–2 98 Å

J R Witt and D Britton, *Acta Crystallogr, Sect B,* 27 (1971) 1835

$SnHAsO_4$

($P2_1/c$) $Z = 4$ The compound is isostructural with $SnHPO_4$

A F Berndt, *Acta Crystallogr, Sect. B,* 27 (1971) 1837

Dinitro(2, 9-dimethyl-1, 10-phenanthroline)-palladium(II)

($P2_1np$) $Z = 4$, $R = 7\%$ for 1155 reflections In the Pd–N coordination square the Pd atom lies in the general plane of the organic molecule However, the two nitrate nitrogen atoms are displaced above this plane Pd–NO_2 = 1 98 and 2 02, Pd–N(phen) = 2 09 Å N–N (phen) = 2 74, N–N(NO_2) = 2 54 Å

J Fridrichsons, A Mel Mathieson and L F Power, *J Cryst Mol Struct,* 1 (1971) 333

Bis(N,N-di-n-butyldithiocarbamato)gold(III)bis(1, 2-dicyanoethane-1, 2-dithiolato)-aurate(III), $Au[S_2CN(C_4H_9)_2]_2{}^{-}Au[S_2C_2(CN)_2]_2{}^{+}$

($P2_1/c$) $Z = 2$, $R = 4\%$ for 1874 non-zero independent reflections In both anion and cation the gold atom is in square-planar coordination with four sulphur atoms Au–S = 2 333(4), 2 337(4) in the cation and 2 312(4), 2 306(5) Å in the anion

J H Noordik and D T Beurskens, *J Cryst Mol Struct,* 1 (1971) 339

The complex hydrate $Cr_2O_7Mg\ 2[(CH_2)_6N_4]\ 6H_2O$

($P\bar{1}$) $Z = 2$, 11 3% for 2800 reflections

F Dahan and G Champetier, *C R Acad Sci, Ser C,* (1971) 805 *cis*(Diphosphine)(diiodo)irondicarbonyl, $FeI_2(CO)_2(PH_3)$

($P2_1/a$) $Z = 4$, $R = 9.2\%$ for 426 independent reflections. The iron atom is octahedral with two iodines *cis* and two carbonyls *cis* in the equatorial plane. The two phosphine phosphorus atoms are *trans* apical. Fe–I = 2.64(1), 2.63(1); Fe–C = 1.85(7), Fe–P = 2.24(2), 2.27(2) Å. Angles are I–Fe–I, 93.0(3)°, C–Fe–C, 99(4)°, P–Fe–P, 173.2(7)°.

J. L. Birck, Y. LeCars, N. Baffier, J. Legendre and M. Huber, *C. R. Acad. Sci., Ser. C*, (1971) 880.

Dinitro(hydroxo)nitroso(diammine)ruthenium, $RuNO(OH)(N)_2)_2(NH_3)_2$

($P2_1/c$) $Z = 8$, $R = 8.3\%$ for 2247 independent reflections. The metal is octahedrally coordinated, NO and OH groups are *trans* to each other while the two NH_3 and two NO_2 are *cis* to each other. The Ru–N–O group is typical of metal nitroso compounds. Ru–N = 1.76(1), N–O = 1.12(2); bond angle Ru–N–O = 176.6(1.3)°

T. S. Khodashova, V. S. Sergienko and M. A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 12 (1971) 439

Neodymium trisacetylacetonate dihydrate

($P\bar{1}$) $Z = 2$, $R = 10.7\%$. The coordination polyhedron is described as a distorted tetragonal antiprism, a distorted two-centred trigonal prism or a distorted dodecahedron. The resulting isomer designations are *sss* for the antiprism and *mmm* for the dodecahedron.

L. A. Aslanov, M. A. Porai-Koshits and M. O. Dekaprilevich, *J. Struct. Chem. (USSR)* 12 (1971) 431

Neodymium pyrosilicate, $Nd_2Si_2O_7$

($P2_12_12_1$) $Z = 4$, $R = 7.7\%$ for 1820 reflections. The coordination number of both neodymium atoms is 8. Nd–O = 2.370–2.772 Å, Si–O = 1.59–1.667 Å, angle Si–O–Si = 132°36′. The bridging oxygen does not coordinate in the cation. Si–O (mean, bridge) = 1.613 Å

Y. I. Smolin and Y. F. Shepelev, *J. Struct. Chem. (USSR)* 12 (1971) 425

$Ti_3Co_5B_2$

($P4/mbm$) $Z = 20$, $R = 15.3\%$ ($hk0$). The Ti and B atoms form a lattice of quadrangles and rhombs, the Co atoms form a lattice of pentagon-, square and triangles. The B atoms lie at the centre of trigonal prisms of Co atoms connected in pairs by the edges

Y. B. Kuz'ma and Y. P. Yarmolyuk, *J. Struct. Chem. (USSR)*, 12 (1971) 422

Ytterbium pyrosilicate, $Yb_2Si_2O_7$

(C_2/m) $Z = 2$, $R = 5.4\%$. The ytterbium octahedron is greatly distorted and has inherent symmetry 2. Yb–O = 2.218(4), 2.209(4), 2.284(4). O–O = 2.756 and 2.815 Å. The Si–O–Si bond in the pyro group is linear. Each polyhedron is linked to three others by common edges

Y. L. Smolin, Y. F. Shepelen and I. K. Butikova, *J. Struct. Chem. (USSR)*, 12 (1971) 248

$(Sn^{IV}Cl_3OH \cdot H_2O)_2 \cdot 3C_4H_8O_2$

($P\bar{1}$) $Z = 2$, R = 17 4% for 832 non-zero reflections Octahedral complexes of tin(IV) are linked by hydroxyl bridges into dimers The dioxane molecules are not coordinated to tin but are hydrogen-bonded to the oxygens which are coordinated to tin.

N G Bokii and Y T Struchkov, *J Struct Chem (USSR),* 12 (1971) 253

Uranyl tetranitrates

The compounds of general type $M_2[UO_2(NO_3)_4]$ where $M = NH_4$, Rb, Cs are described. The structures consist of M^+ and $[UO_2(NO_3)_4]^{2-}$ ions In the centrosymmetric anion two nitrate groups are joined to a uranium with two oxygens and two other groups joined with one oxygen forming in the equatorial plane six-fold coordination of oxygen atoms around a uranium

I I Kapshukov, Y F Volkov, E P Moskvichev, I A Lebedev and G N Yakovlev, *J Struct Chem (USSR),* 12 (1971) 77

Piperazine thiomolybdate, $[MoS_4](C_4N_2H_{12})$

($P2_1/b$) $Z = 4$, R = 11 2% The structure is made up of $(MoS_4)^{2-}$ tetrahedra and piperazine ions $[C_4H_2H_{12}]^{2+}$ Mo–S = 2 18 Å, S–S = 3 56 Å. The piperazine ion has the 'chair' form

P A Koz'min and Z V Popova, *J Struct Chem (USSR),* 12 (1971) 81

Lead 8-mercaptoquinolate, $Pb(SNC_9H_6)_2$

($P2_12_12_1$) $Z = 2$, R = 13 2% for 823 reflections The lead atom is in a distorted tetragonal pyramid with lead at the apex Pb–N = 2 485 Å, Pb–S = 2 74 Å A long Pb–S contact (3 47 Å) allows the molecules to be arranged parallel to each other forming columns along *c*

V M Agre and E A Shugam, *J Struct Chem (USSR),* 12 (1971) 84

p-*N*,*N*-Dimethylaminophenyldiazonium chlorozincate, $2[(CH_3)_2NC_6H_4N_2Cl]\ ZnCl_2$

($B2/b$) $Z = 4$, R = 14 6% for 660 non-zero reflections The structure consists of separate ions with the usual tetrahedral structure for $ZnCl_4{}^{2-}$

Y M Nesterova and M A Porai-Koshits, *J Struct Chem (USSR),* 12 (1971) 89

π-Indenyl-rhodium-π-duroquinone

($P2_1/c$) R = 9 0% for ~ 1000 reflections The Rh atom is coordinated to the five-membered ring of the planar indenyl ligand (Rh–C = 2 18–2 25 Å) and to the duroquinone ligand (Rh–C = 2 15 and Rh–C(CO) = 2 43 Å) As a result the duroquinone ligand is distorted from planarity with a dihedral angle of 25° between "bottom" and "prows"

G.G Aleksandrov and Y T Struchkov, *J Struct. Chem (USSR),* 12 (1971) 99

Racemic tris[di-μ-hydroxo-bis(ethylenediamine)cobalt(III)]cobalt(III) trisdithionate octahydrate, $[Co_4C_{12}H_{54}N_{12}O_6]\,[S_2O_6]_3\ \ 8H_2O$

($P2_1/n$) The analysis confirms Werner's structural formulae for the cobalt, 'hexel' complexes The cation antipodes exhibit the configurations Δ(ΔΛΛ) and Λ(ΛΔΔ) The en rings lie such that the cations have approximate C_2 symmetry.

U Thewalt, *Chem Ber*, 104 (1971) 2657

(2-Diethylaminoethanolato)copper(II) bromide

($P2_1/n$) $Z = 4$, R = 5 8% for 1032 observed reflections The complex is binuclear with an oxygen bridge between adjacent coppers Cu–O = 1 904 and 1 924 Å. Angle Cu–O–Cu is 104.5°. The copper coordination is distorted square planar. The μ_{eff} and solid state electronic and absorption spectra in various solvents are recorded

A Pajunen and M L Eatonen, *Suom Kemistilehti B*, 44 (1971) 200.

μ_3-Trimethylsilylimido-μ_3-carbonyltris(tricarbonyl iron)

($Pca2_1$) $Z = 4$, R = 4 3% for 1639 reflections The structure has pseudo-C_3 symmetry of the molecular skeleton including a triply coordinated carbonyl group on the iron cluster as well as a trimethylsilylimino group The coordination of the equidistant iron atoms is distorted octahedral in which three caroonyl groups are twisted out of the Fe atomic plane Mean Fe–C = 1 798 and C–O = 1 141 Å

B.L Barnett and C Kruger, *Angew Chem*, 10 (1971) 910

Bis(π-cyclopentadienyl)tetrasulfidotungsten(IV)

($P2_1/n$) $Z = 4$, R = 4 9% for 989 reflections The structure is compared with compounds of the same type (Mo and Cr) NMR results are rationalised The coordination sphere of the tungsten is roughly tetrahedral with two cp rings taking up two positions and the bidentate tetrasulphide the other two

B R Davis, I Bernal and H Kopf, *Angew Chem*, 10 (1971) 921

π-Allyl-dihydrobis-(3, 5-dimethyl-1-pyrazolyl)boratodicarbonyl-molybdenum, $H_2B[(CH_3)_2PZ]_2Mo(CO)_2C_3H_5$

($P\bar{1}$) R = 5 4% for 2224 observed reflections. The coordination about the molybdenum atom is distorted-octahedral and that about boron, nearly tetrahedral Mo–N (average) = 2 21 Å, B–N (av) = 1 54 Å Terminal carbon atoms of the allyl group are further from molybdenum than the central carbon atom There is a possibility of an Mo–H bond

C A Kosky, P Gans and G Avitabile, *Acta Crystallogr*, *Sect B*, 27 (1971) 1859

[1, 2-Bis-(diphenylphosphino)ethane] -tetracarbonyl chromium, $Cr(CO)_4$ (diphos)

($Pbca$) $Z = 8$, R = 6 6% for 2707 independent reflections Each chromium is surrounded by a distorted octahedral array of two *cis* phosphorus atoms and four carbons Cr–P = 2 360(2) Å, angle P–G–P = 83 41(8)°, Cr–C (*trans* to P) = 1 831(7), Cr–C = 1 884(7), C–O = 1 141(12)–1 165(12) Å

M J Bennett, F A. Cotton and M.D. LaPrade, *Acta Crystallogr.*, *Sect B*, 27 (1971) 1899

Bis(triphenylphosphine)(ethylene)nickel, a refinement

The coordinate changes are such as to give an ethylene-bond twist of 5 0° out of the P—Ni—P plane compared with the original estimate of 12° (see W Dreissig and H. Dietrich, *Acta Crystallogr , Sect. B,* 24 (1968) 108 for the original structure determination)

P.-T. Chena, C D Cook, C.H Koo, S C Nyburg and M Tshiomi, *Acta Crystallogr., Sect. B,* 27 (1971) 1904.

Ammonium heptafluorocerate(IV) monohydrate, $(NH_4)_3CeF_7 \cdot H_2O$

($P2_1/n$) $Z = 4$, $R = 1$ 8% for 1029 reflections Cerium is eight-coordinated in dimeric $Ce_2F_{14}{}^{6-}$ units. The fluorine coordination around cerium is described as a dodecahedron with triangular faces All hydrogen atoms are located

R R Ryan and R A Penneman, *Acta Crystallogr , Sect B,* 27 (1971) 1939

$Rb_5Zr_4F_{21}$

($P2_1$) $Z = 2$, $R = 5.08\%$ for 1376 reflections The F^- coordination polyhedra are different for each of the four crystallographically independent Zr ions

G. Brunton, *Acta Crystallogr , Sect. B,* 27 (1971) 1944.

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

($I2/a$) $Z = 4$, $R = 10$ 2% for 1341 independent reflections. The sulphur atoms are arranged about the tin in a distorted octahedron with two bidentate and two monodentate ligands The latter occupy *cis* positions Sn—S(average) = 2 546(7) for tridentate ligands Sn—S (monodentate) = 2 504(7) Å

C S Harreld and E O Schlemper, *Acta Crystallogr , Sect B,* 27 (1971) 1964

$Cu_3Mo_2O_9$

($Pna2_1$) $Z = 4$, $R = 6$ 0% for 679 significant reflections The structure contains MoO_4 tetrahedra and CuO_6 octahedra distorted in two ways and CuO_5 tetragonal pyramids

L Kihlborg, R. Norrestam and B Olivecrona, *Acta Crystallogr , Sect B,* 27 (1971) 2066

Hexakis(imidazole)cadmium(II) nitrate, $[Cd(C_3H_4N_2)_6](NO_3)_2$ (A) and hexakis(imidazole)cadmium(II) hydroxide nitrate tetrahydrate, $[Cd(C_3H_4N_2)_6](OH)(NO_3) \cdot 4H_2O$ (B)

($R\bar{3}$) $Z = 3$, $R = 3.6\%$ for 1343 reflections (A) and ($P6_3/m$) $Z = 2$, $R = 5$ 4% for 996 reflections (B). Both structures consist of discrete $Cd(C_3H_4N_2)_6{}^{2+}$ and $NO_3{}^-$ ions but in the OH complex the packing is very different Cd—N(average) is 2 363. The imidazole rings are hydrogen bonded through pyrrole-type nitrogen atoms, to nitrate groups There is considerable difference in orientation of the imidazole groups in the two complexes

A D Mighell and A. Santoro, *Acta Crystallogr , Sect B,* 27 (1971) 2089

$5Hg(CN)_2 \cdot 4C_4H_8O$

$R = 6.8\%$ for 439 reflections The mercury atoms are in octahedral coordination Two positions are occupied by CN carbons and four others are occupied by either four THF oxygens or by four nitrogen atoms of neighbouring Hg(CN)· molecules

M. Frey and M Ledesert, *Acta Crystallogr., Sect. B*, 27 (1971) 2119.

Pseudo-trisodium orthoarsenate dodecahydrate and the isomorphous phosphate and vanadate salts

The crystal structures show that the compounds should be formulated as $[Na_{12-13}(OH)_{0-1}(OH_2)_{48}](XO_4)_4$

E. Tillmans and W H. Baur, *Acta Crystallogr., Sect. B*, 27 (1971) 2124

$(-)_{589}$-Tris-(+*trans*-1,2-diaminecyclopentane)cobalt(III) chloride tetrahydrate, $(-)_{589}$-$[Co(+cptn)_3]Cl_3 \cdot 4H_2O$

($P6_122$) $Z = 6$, $R = 12\%$ for 786 observed reflections The complex is mononuclear with pseudo-P_3 symmetry. Three ligands are bonded to cobalt through nitrogen atoms, the five-membered chelate rings are not planar They are similar in shape and size to those of $[Co(en)_3]^{3+}$ The conformation of the chelate ring is *lel* The cyclopentane rings have half-chair conformation.

M Ito, F. Marumo and Y Saito, *Acta, Crystallogr , Sect B*, 27 (1971) 2187.

Nitratobis-(2,2′-dipyridyl)cobalt(III) hydroxide nitrate tetrahydrate, $[Co(C_{10}H_8N_2)_2(NO_3)](NO_3)(OH)\ 4H_2O$

($C2/c$) $Z = 4$, $R = 6\ 2\%$ for 2995 reflections The cobalt atom is octahedrally coordinated by two molecules of bipy and by a bidentate nitrate group Water nitrate and hydroxide are involved in extensive hydrogen bonding

C W Reimann, M Zocchi, A.D. Mighell and A. Santoro, *Acta Crystallogr., Sect B*, 27 (1971) 2211

Calcium hydrazinecarboxylate monohydrate, $Ca(NH_2{-}NH{-}COO)_2\,H_2O$

($P\bar{1}$) $Z = 2$, $R = 10\ 3\%$ for 1113 independent reflections The coordination around calcium is a distorted pentagonal bipyramid with a centred face Ca–O (average) 2 44 Å Two crystallographically independent anions form pentatomic rings with Ca

A. Braibant, A M Manotti Lanfredi, M A Pellinghelli and A Tiripicchio, *Acta Crystallogr., Sect B*, 27 (1971) 2261

A mercury carbene complex, $((C_6H_5)_2C_3N_2H_2)_2Hg\ \ 2ClO_4$

($P\bar{1}$) $Z = 1$, $R = 5\ 2\%$.

P. Loger and G. Ruban, *Acta Crystallogr , Sect B*, 27 (1971) 2276 (in German).

Ammonium heptafluorothorate, ammonium *catena*-di-μ-fluoro-pentafluorothorate(IV), $(NH_4)_3ThF_7$

($Pnma$) $Z = 4$, $R = 4.2\%$ for 1053 significant reflections. Thorium is nine-coordinated by fluorine atoms in a tricapped trigonal prism arrangement. Th–F = 2.25–2.59Å

R.A. Penneman, R.R. Ryan and I.K. Kressin, *Acta Crystallogr., Sect. B* 27 (1971) 2279

Bismuth trichloride

($P2_1a$) $R = 4.43\%$ for 935 reflections. A bismuth atom is associated with three chlorine atoms in the shape of a distorted trigonal pyramid (2.468, 2.513 and 2.518 Å) and with five others at bridging distances 3.216–3.450 Å

S.C. Nyburg, G.A. Ozin and J.T. Szymanski, *Acta Crystallogr., Sect. B*, 27 (1971) 2298

[Co(QP)Cl] BPh_4 (QP = tris(*o*-diphenylphosphinophenyl)phosphine)

($P2_1nb$) $Z = 4$, $R = 11\%$ for some 4700 reflections. The cobalt in the cation has a distorted trigonal-bipyramidal coordination. The angle between equatorial and axial Co–P bonds is 86.5 ± 0.6°. Co–P(axial) = 2.057 Å, Co–P(equatorial) = 2.261, 2.280 and 2.318. There are many Van der Waals contacts between phenyl groups of the QP ligand and between these and the axial chlorine atom

T.L. Blundell and H.M. Powell, *Acta Crystallogr., Sect. B*, 27 (1971) 2304

Bis-(1-methyl-3-*O*-chlorophenyltriazene 1-oxide) cobalt(II)

($P2_12_12_1$) $Z = 4$, $R = 7.7\%$ for 853 observed reflections. Two ligands are *cis* to each other and are tridentate through O, N and Cl. The chlorine atoms attached to the benzene rings coordinate weakly to the cobalt in completing a distorted octahedron. Co–Cl = 2.98 Å

G.L. Dwivedi and R.C. Srivastava, *Acta Crystallogr., Sect. B*, 27 (1971) 2316

Iron(II) chloride tetrahydrate, $FeCl_2 \cdot 4H_2O$

($P2_1/c$) $R = 3.2\%$ for 765 reflections. Fe–Cl = 2.53, Fe–O = 2.12 and 2.08 Å

J. Meunier-Piret and M. Van Meerssche, *Acta Crystallogr., Sect. B*, 27 (1971) 2329

Tris-(2,2,6,6-tetramethylheptane-2,5-dionato)erbium(III), $Er(thd)_3$

($Pmn2_1$) $Z = 2$, $R = 4.1\%$ for 1844 reflections. The erbium ion is coordinated by six oxygens in a trigonal prism. The relationship between this structure and known hydrated and lanthanide diketonates is discussed. The structure is the first example of a monomeric anhydrous lanthanide chelate

J.P.R. de Villiers and J.C.A. Boeyens, *Acta Crystallogr., Sect. B*, 27 (1971) 2335

cis-Tetrachlorodiformonitrile titanium(IV), $TiCl_4(NCH)_2$

($Pnma$) $Z = 4$, $R = 5.7\%$ for 778 reflections. The titanium atom is surrounded by four chlorine and two *cis* nitrogen atoms, in an octahedron. Ti–Cl = 2.226(2), Ti–N = 2.198(7), N–C = 1.11(1) Å

G. Constant, J.C. Daran and Y. Jeannin, *Acta Crystallogr., Sect. B*, 27 (1971) 2388

Bis-(L-phenylalaninato)copper(II)

($P2_1$) $Z = 2$, $R = 4.1\%$ for 1475 reflections. The structure closely resembles that of bis-(l-alaninato) Cu$^{\text{II}}$. The copper is in a tetragonally distorted octahedron of bis-(L-alaninato) Cu$^{\text{II}}$. The conformations of both phenylalanine molecules are similar, the alanato rings being pointed away from the metal coordination.

D. Van Der Helm, M. B. Lawson and E. L. Enwall, *Acta Crystallogr., Sect. B*, 27 (1971) 2411.

CuBr · CN$_3$CN

($P2_1nb$) $Z = 4$, $R = 9.6\%$. Infinite chains of –Cu–Br– extend along the *a* axis. Each copper is surrounded by three bromine atoms and one nitrogen at the vertices of a tetrahedron. The CH$_3$CN molecule is linear.

M. Massaux, M. J. Bernard and M. LeBihan, *Acta Crystallogr., Sect. B*, 27 (1971) 2419.

Sr(HCOO)$_2$ · 2H$_2$O

($P2_12_12_1$) $Z = 4$, $R = 2.1\%$ for 673 reflections.

J. L. Galigne, *Acta Crystallogr., Sect. B*, 27 (1971) 2429 (in French).

Cobalt(II) monoglycerate

($P2_1/c$) $Z = 4$, $R = 12\%$ for 564 reflections. The oxygen atoms form a distorted trigonal bipyramid around the cobalt ion. A magnetic moment of 3.94 BM is recorded. Infrared data are also discussed.

P. G. Slade, E. W. Radoslovich and M. Ravpach, *Acta Crystallogr., Sect. B*, 27 (1971) 2432.

Anhydrous calcium hydrazinecarbonylate, Ca(N'H$_2$–NH–COO)$_2$

($P2_1/c$) $Z = 4$, $R = 7.5\%$ for 1058 independent reflections. Calcium is in a pentagonal bipyramid. The complexes are joined by oxygen bridges. Cu–Cu = 3.7 Å. Two crystallographically independent ligand molecules are practically equal.

A. Braibanti, A. M. Manotti Lanfredi, M. A. Pellinghelli and A. Tiripicchio, *Acta Crystallogr., Sect. B*, 27 (1971) 2448.

Bis(hydrazinecarboxylato-*N'*, *O*)-zinc, Zn(N'H$_2$–NH–COO)$_2$

(*Pc*) $Z = 2$, $R = 7.9\%$ for 665 reflections. The zinc is square-pyramidal with basal corners occupied by two oxygens and two nitrogen atoms, the apical position belonging to an adjacent complex. Zn–O (average) = 2.04(2) Å. As usual for this ligand, one part of the molecule lies in the same plane while a nitrogen is displaced from the plane.

F. Bigoli, A. Braibant, A. Tiripicchio and M. Tiripicchio Camellini, *Acta Crystallogr., Sect. B*, 27 (1971) 2453.

Lithium triethylene diamine iodide, LiI · 3en

($P\bar{3}1c$) $Z = 2$. The lithium and iodine ions occupy the centres of distorted octahedra of

nitrogen atoms The en group is *cis*
H. Gillier-Pandraup and S. Jamet-Delcroix, *Acta Crystallogr., Sect. B*, 27 (1971) 2476

A mercuric bromide-1,4-dioxane solvate, $HgBr_2 \cdot 2C_4H_8O_2$
($I4/m$) $Z = 2$, $R = 7.9\%$ Hg—Br is 2.43 Å The dioxane ring has a chair conformation Coordination about mercury is octahedral. Hg–O (dioxane) 2.83 Å
M. Frey and J. Monier, *Acta Crystallogr., Sect. B*, 27 (1971) 2487