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X-RAY BIBLIOGRAPHY

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Bis[*N*-(2-hydroxyethyl)ethylenediamine]copper(II) perchlorate, $[Cu(C_4H_{12}N_2O)]$-$ClO_4)_2$

($P2_1/n$) Z=4, R=7.9% for 3872 independent reflections. The compound consists of discrete anions and cations and perchlorate anions. Each of the *N*-(2-hydroxyethyl)ethylenediamine (*hn*) ligands is tridentate, coordinating in a facial configuration. The geometric arrangement of ligand dimers about the central copper atom is a distorted octahedron with the chemically equivalent amino groups *cis* to each other at Cu–N distances of 2.012(4), 2.018(4), 2.036(3) and 2.041(3) Å while the alcohol groups are *trans* to each other at Cu–O distances of 2.419(3) and 2.518(3) Å.

R.V. Chastain, Jr. and T.L. Dominick, *Inorg. Chem.*, 12 (1973) 2621.

trans--Dinitro-2,2'-(1,3-diaminopropane)bis(2-methyl-3-butanone)dioximatocobalt-(III), $[Co(PnAOH)(NO_2)_2]^0$

($P2_1/c$) Z=4, R=3.5% for 2963 reflections. The structure consists of discrete neutral molecules with near octahedral coordination about the cobalt atom. The nitro groups are nitrogen bound and are in the *trans* configuration. The nitro nitrogens are different distances from the cobalt atom, 1.984(3) and 1.937(3) Å with the cobalt slightly out of the plane of the amine and oxime nitrogens toward the nitro nitrogen with the short Co–N distance.

R. Kent Murmann and E.O. Schlemper, *Inorg. Chem.*, 12 (1973) 2625.

Rhodium pentafluoride

($P2_1/a$) Z=8, R=2.9% for 1207 non-zero reflections. The structural unit is a fluorine bridged tetramer similar to those reported for $(RuF_5)_4$ and $(OsF_5)_4$. Each rhodium atom is coordinated by six fluorine atoms in an approximately octahedral arrangement. Each of a *cis* pair of fluorine atoms in the RhF_6 group is shared with another rhodium atom. Rh–F–Rh is 135±1° and Rh–F 2.01(1) Å.

B.K. Morrell, A. Zalkin, A. Tressaud and N. Bartlett, *Inorg. Chem.*, 12 (1973) 2640.

5,12-Dichloro-1,7-dimethyl-1,7-dicarba-closo-dodecaborane(12)
($Pmn\ 2_1$) $Z=2$, $R=3.2\%$ for 460 reflections.
H.V. Hart and W.N. Lipscomb, *Inorg. Chem.*, 12 (1973) 2644.

Chloroaquobis(trimethylarsine)tetrakis(trifluoromethyl)rhodiacyclopentadiene, $RhCl(H_2O)(As(CH_3)_3)_2C_4(CF_3)_4$
($P2_12_12_12_1$) $Z=4$, $R_W=6.3$ and 6.0% for 1689 reflections. The coordination about rhodium is in the form of a distorted octahedron with the chlorine atom, the oxygen atom of the water molecule and the 1 and 4 carbon atoms of the $C_4(CF_3)_4$ moiety approximately defining a plane and the arsenic atoms disposed above and below it. Rh–Cl, 2.448(5); Rh–O, 2.241(12); Rh–C, 2.047(16) and 1.998(16) Å.
J.T. Mague, *Inorg. Chem.*, 12 (1973) 2649.

$[Gd(NO_3)_3(C_{14}H_{14}N_4)]$
(*Pbca*) $Z=8$, $R=3.3\%$ for 1478 observed reflections. Gadolinium is ten coordinated with the polyhedron approximating a distorted pentagonal bipyramid where four equatorial positions are occupied by the nitrogen atoms of the tetradentate amino ligand, the remaining three positions being occupied by the three bidentate nitrate groups.
G.D. Smith, C.N. Coughlan, M.-UL-Hague and F.A. Hart, *Inorg. Chem.*, 12 (1973) 2654.

Monothiodibenzoylmethanato-1-norbornenyl-2-methylallylpalladium(II)
($P2_1/C$) $Z=4$, $R=6.4\%$ for 1411 independent reflections. There is essentially square planar geometry about the palladium atom with the coordinated olefin *trans* to sulphur. The long palladium–olefin bond length of 2.13(1) Å as well as the palladium–oxygen bond of 2.10(1) Å is attributed to the *trans* influence of the sulphur and σ-bonded carbon atoms respectively. Pd–C is 2.05(2) Å.
J.A. Sadownick and S.J. Lippard, *Inorg. Chem.*, 12 (1973) 2659.

1-Oxa-4-telluracyclohexane-4, 4-diiodide, $C_4H_8OTeI_2$
($P2_1/C$) $Z=4$, $R=3.1\%$ for 1661 reflections. The bonding about tellurium is octahedral. There are two Te–C bands at 2.15(1) and 2.17(1) Å making C–Te–C 94.1(4)°. Approximately perpendicular to the C–Te–C plane, tellurium forms axial bonds with iodine(1) at 2.886(1) Å and iodine(2) at 2.938(1) Å and I–Te–I, 177.08(4)°. The octahedron about tellurium is completed through formation of weak intermolecular bands with an iodine(1) atom in a second molecule (3.814 Å) and an iodine(2) atom in a third molecule at 3.692(1) Å.
H. Hope, C. Knobler and J.D. McCullough, *Inorg. Chem.*, 12 (1973) 2665.

Phenoxatellurin-10, 10-diiodide, $C_{12}H_8OTeI_2$
($P2_1/n$) $Z=4$, $R=4.3\%$ for 2276 reflections. The phenoxatellurin moiety consists of planar halves with a fold angle of 163.9(4)° along the line of the shared tellu-

rium and oxygen atoms. Te—C 2.09(2), 2.11(2) Å; C—Te—C is 91.5(6)°. Approximately perpendicular to the C—Te—C plane, tellurium forms axial bonds Te—I of 2.945(2) and 2.941(2) Å in length. Tellurium also forms weak bonds with iodines from neighbouring molecules Te—I 3.739 and 3.788 Å.

J.D. McCullough, *Inorg. Chem.*, 12 (1973) 2669.

$[Me_4N^+][Me_2B_{10}C_2H_{11}^-]$

($P2_1/c$) $Z=4$, $R=6.22\%$ for 2981 independent reflections. The $Me_2B_{10}C_2H_{11}^-$ anion has (non-crystallographic) *Cs* symmetry with ten borons and one carbon defining an icosahedral fragment with an apex B_4C face. The second carbon atom of the dicarbanid dodecaborate framework bridges two boron atoms in the apex B_4C face and is bound to an exo methyl group and to an endo hydrogen.

M.R. Churchill and B.G. DeBoer, *Inorg. Chem.*, 12 (1973) 2674.

(A) Mercaptoacetatobis(ethylenediamine)cobalt(III)chloride monohydrate. $[Co(en)_2(SCH_2COO)]Cl.H_2O$; (B) mercapto acetatobis(ethylenediamine)chromium(III) perchlorate, $[Cr(en)_2(SCH_2COO)]ClO_4$; and (C) β mercapto ethylaminobis(ethylenediamine)cobalt(III)dithiocyanate $[Co(en)_2(SCH_2.CH_2NH_2)](SCN)_2$

($P2_1/a$) $Z=4$, $R=3.3\%$ for 2234 unique reflections (A); ($P2_1/n$) $Z=4$, $R=3.7\%$ for 1502 unique reflections (B); (*Pbca*) $Z=8$, $R=3.2\%$ for 1493 unique reflections (C). All three complex cations are *pseudo*-octahedral six-coordinate species. Both mercaptoethylamine and β-mercaptoethylamine anions chelate to form five membered rings. The mercaptoacetate complexes both exhibit Δλδ and Λδλ configuration in their centric crystals. The β mercapto ethylamine complex adopts Δλλλ and Λδδδ configuration.

R.C. Elder, L.R. Florian, R.E. Lake and A.M. Yacynych, *Inorg. Chem.*, 12 (1973) 2690.

The adduct of anhydro-5-mercapto-2,3-diphenyltetrazolium hydroxide and mercury(II) chloride, $C_{13}H_{10}N_4S.HgCl_2$

(C_2) $Z=4$, $R=6.0\%$ for 1134 independent reflections. The structure consists of parallel chains of alternating mercury and sulphur atoms. The coordination polyhedron around the mercury atom is a distorted trigonal bipyramid with two apical sulphur atoms at distances of 3.28(2) Å from the mercury atom. The equatorial plane contains the mercury atom, two chlorine atoms and one sulphur atom. Hg—Cl, 2.34(1) and 2.57(1); Hg—S, 2.40(1) Å.

W.J. Kozarek and Q. Fernando, *Inorg. Chem.*, 12 (1973) 2129.

$[(C_5H_5)Fe(SC_2H_5)S]_2$, a possible model for ferredoxin

($P2_1/c$) $Z=4$ (dimers); $R=2.2\%$ for 1781 non-zero reflections. The complex contains a planar Fe—S—S—Fe bridge and the two iron atoms are also linked by two bridging mercapto ligands. The structural evidence suggests that the S_2 oxidation state is intermediate between S_2^0 and S_2^{2-}. The structure raises the possibility that the ferredoxins with two irons and two labile sulphur atoms might contain a Fe–S–S–Fe bridge.

A. Terzis and R. Rivest, *Inorg. Chem.*, 12 (1973) 2132.

Manganese pyrophosphate dihydrate, $Mn_2P_2O_7 . 2H_2O$

($P2_1/n$) Z=4, R=3.8% for 1506 reflections. Chains of octahedra with shared edge run through the structure. The two ends of the pyrophosphate anion are 20° from the completely eclipsed conformation and the P–O–P bridge angle is 127.5(2)°. The mean P–O distance for terminal P–O is 1.520(2) and P–O (bridge) is 1.613(3) Å.

S. Schneider and R.L. Collin, *Inorg. Chem.*, 12 (1973) 2136.

(A) Bis(tetraphenylarsonium) tris(1,2-dicyanoethylenedithiolato) molybdenate(IV), $[(C_6H_5)_4As]_2[Mo(mnt)_3]$ and (B) its tungsten analogue $[(C_6H_5)_4As]_2$ $[W(mnt)_3]$

($Pbcn$) Z=4, R=6.7% for 1643 reflections (A); ($Pbcan$) Z=4, R=5.5% for 1465 independent reflections (B). Despite their spectral differences these compounds are isomorphous and nearly isostructural. The coordination geometry of the MS_6 framework is close to D_3 symmetry and consists of two parallel S_3 triangles twisted 28° from the trigonal prism. The triangles are at a distance such that the trigonal compression is midway between values for the octahedron and the known trigonal prisms. Metal–sulphur bonds average 2.38 Å in both cases.

G.F. Brown and E.I. Stiefel, *Inorg. Chem.*, 12 (1973) 2140.

Bis(cyclopentamethylenethiocarbamato)bis(piperidine)zinc(II), $Zn(SOCN(C_5H_{10}))_2$ $(NC_5H_{11})_2$

($P1$) Z=2, R=7.3% for 2520 non-zero reflections. The coordination geometry about the Zn(II) ion is approximately tetrahedral with the sulphur atoms of the two thiocarbamate ligands occupying two coordination sites and nitrogen atoms of the piperidine ligands in the remaining two sites. The molecule has approximate two-fold symmetry.

D.L. Greene, B.J. McCormick and C.G. Pierpont, *Inorg. Chem.*, 12 (1973) 2148.

"Ruthenium Black" – di-μ-amido-bis[tetra ammineruthenium(III)] chloride tetrahydrate, $[(NH_3)_4Ru(NH_2)_2Ru(NH_3)_4]Cl_4 . 4H_2O$

($P2_1/n$) Z=2, R=3.6% for 2114 non-zero reflections. The centrosymmetric cation features distorted octahedral ruthenium centres, two amido bridging groups and a relatively short Ru–Ru distance, 2.625(1) Å. The Ru–NH_2–Ru bridging bond angle is 81.0(1)° suggesting Ru–Ru bonding interaction.

M.T. Flood, Z.F. Ziolo, J.E. Earley and H.B. Gray, *Inorg. Chem.*, 12 (1973) 2153.

Bis[2-(2-aminoethyl)pyridine]copper(II) iodide, $[Cu(C_7N_2H_{10})_2]I_2$

($P2_1/c$) Z=2, R=2.9% for 2239 independent reflections. The complex consists of four-coordinated cations which are well separated from discrete iodine anions. The copper atom is in square planar coordination, in contrast to the bromo analogue

which is five coordinate.
V.C. Copeland and D.J. Hodgson, *Inorg. Chem.*, 12 (1973) 2157.

Bis(1-ethyl-4-carbomethoxypyridinium) bis(maleonitriledithiolato)nickelate(II)
($P2_1/c$) $Z=2$, $R_1=4.1\%$ for 993 reflections. The compound contains planar centrosymmetrical $[Ni(mnt)_2]^{-2}$ dianions sandwiched between planar (excepting the alkyl groups) 1-ethyl-4-carbomethoxypyridinium cations (KOS^+). Within this sandwich ion associate, the dihedral angle between the $Ni(mnt)_2$ and KOS planes is 10.8°.
I.G. Dance, P.J. Solstad and J.C. Calabrese, *Inorg. Chem.*, 12 (1973) 2161.

Bis(triphenylphosphine)tetramethylene platinum(II), $Pt[P(C_6H_5)_3]_2C_4H_8$
($P\bar{1}$) $Z=2$, $R=7.4\%$ for 3504 independent reflections. The phosphine ligands and the terminal carbon atoms of the tetramethylene ring comprise a distorted square planar configuration about the platinum atom in which the coordinated carbon atoms are 2.12(2) and 2.05(2) Å from the metal. The angle between the tetramethylene ring and the plane defined by platinum, phosphorus(1) and phosphorus(2) is 175(1)°.
C.G. Biefield, H.A. Eick and R.H. Grubbs, *Inorg. Chem.*, 12 (1973) 2166.

μ-Peroxo-bis[ethylenediamine)(diethylenetriamine)cobalt(III)] perchlorate, $[(en)(dien)Co(O_2)Co(en)dien)](ClO_4)_4$
($P2_1/n$) $Z=2$, $R=5.3\%$ for 2130 independent reflections. The oxygen bridge is a peroxide group with O–O, 1.488(6) Å. The Co–O–O–Co linkage is planar, the halves of the cation being related by a centre of symmetry centred on the O–O bond. The coordination about the cobalt atom is slightly distorted octahedral Co–O, 1.896(4)° Co–O–O is 110.0(3)°. The *trans* effect of the peroxide atom is apparent in the significant lengthening of the *trans* Co–N bond relative to the *cis* Co–N bonds.
J.R. Fritch, G.G. Christoph and W.P. Schaefer, *Inorg. Chem.*, 12 (1973) 2170.

Triborane-carbonyl, $B_3H_7 . CO$
($P\bar{1}$) $Z=2$, $R=3.88\%$ for 670 observed reflections. The structure is described by the styx notation 1104.
J.D. Glore, J.W. Rathke and R. Schaeffer, *Inorg. Chem.*, 12 (1973) 2175.

Tetrakis(*O*-ethyl dithiocarbonato)dimolybdenum bis(tetrahydrofuran)
($P\bar{1}$) $Z=1$, $R=3.3\%$ for 4559 reflections. Two tetrahydrofuran molecules occupy the free axial positions of the molecule. The structure of the complex is that of a transition metal carboxylate of general formula $M_2L_4 . 2L'$ and the centrosymmetric molecule has almost ideal D_{4h} symmetry at the level of the Mo_2S_8 skeleton.
L. Ricard, P. Karagiannidis and R. Weiss, *Inorg. Chem.*, 12 (1973) 2179.

Bis{μ-sulphido-thiocarboxamido(di-n-propyldithiocarbamato)molybdenum],
$[MoS(SCN(C_3H_7)_2(S_2CN(C_3H_7)_2\}_2$

($C2/c$) $Z=4$, $R=5.0\%$ for 2049 reflections. The analysis reveals one dithiocarbamato per metal atom, the second one having been cleaved as a C–S bond to yield a thiocarboxamidomolybdenum moiety. The presence of a short Mo–C bond (2.069 Å) indicates the character of the Mo–C interaction. The Mo–S–Mo angle of 106.5(1) and 72.3(1)° respectively indicate a Mo–Mo interaction.

L. Ricard, J. Estienne and R. Weiss, *Inorg. Chem.*, 12 (1973) 2182.

Chloro(5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradeca-4,11-diene)-iron(II) iodide, $[Fe(C_{16}H_{32}N_4)Cl]I$

(C_2) $Z=2$, $R=7.9\%$ for 1192 independent reflections. The structure consists of five-coordinate iron(II) bound to the four nitrogen atoms of a folded form of the macrocyclic ligand and a chloride ion occupying the axial site. Fe–N, 2.134(4) and 2.166(8) Å; Fe–Cl, 2.306(4) Å. The unusual coordination geometry about the iron(II) arises because the steric constraints of the cyclic ligand do not permit the iron to fit into the plane of the four nitrogen atoms and the ligand is not flexible enough to permit folding to accommodate a sixth ligand, *cis* to the chloride.

V.L. Goedken, J.M. Case and G.G. Christoph, *Inorg. Chem.*, 12 (1973) 2894.

Bis(2.6-diacetylpyridine dioximato)nickel(IV), $Ni(C_9N_3O_2H_9)$

($I4_1/a$) $Z=4$, $R=2.8\%$ for 702 unique reflections. The molecular symmetry is $4(S_4)$ with the nickel atom coordinated to two 2,6-diacetylpyridine dioximate ligands via the six nitrogen atoms of these ligands. A comparison of these nickel–nitrogen distances with those found in nickel(IV) complexes suggests a shortening of 0.17 Å in the Ni(IV)–N bond.

G. Sproul and G.D. Stucky, *Inorg. Chem.*, 12 (1973) 2898.

Two isomeric tri(tertiary phosphine) complexes of manganese(I) and chromium(0), $Br(CO)_3Mn$ (bis(2-diphenylphosphinoethyl)phenylphosphine)$Cr(CO)_5$ α and β isomers

($Pbcn$) $Z=8$, $R=9.9\%$ for 3789, (α); (Cc) $Z=4$, $R=8.7\%$ for 3265 intensities (β). Coordination of the triphos ligand to the manganese atom occurs through two adjacent phosphorus atoms and results in the formation of a five membered chelate ring. The phenyl ring attached to the central phosphorus atom can take a position in *cis* or *trans* to the bromine atom relative to the five membered chelate ring and results in the two isomers being formed.

P.H. Bird, N.J. Coville, I.S. Butler and M.L. Schneider, *Inorg. Chem.*, 12 (1973) 2902.

μ-Hydroxy-μ-di(p-tolyl)phosphido-hexacarbonyldiiron, $Fe_2(CO)_2[P(p\text{-}C_6H_4CH_3)_2]$-OH

($C2/c$) $Z=8$, $R=4.6\%$ for 1219 independent reflections. The compound was previously characterised as $Fe_2(CO)_6[P(p\text{-}C_6H_4CH_3)_2]H$ but the analysis reveals a

bridging hydroxide, not hydrogen. Two $Fe(CO)_3$ units are linked by bridging bis(tolyl)phosphido and hydroxide groups and by an iron–iron bond.
P.M. Treichel, W.K. Dean and J.C. Calabrese, *Inorg. Chem.*, 12 (1973) 2908.

Two crystal structures of 6-isothiocyanodecaborane, $6\text{-}B_{10}H_{13}NCS$
Both are ($P2_1/c$) with Z=4. Low temperature form, R=6.3% for 1181 reflections room temperature from, R=8.7% for 716 reflections. The molecule is an isothiocyanate, the nitrogen atom is bonded to the (B6) atom of the decaborane framework.
D.S. Kendall and W.N. Lipscomb, *Inorg. Chem.*, 12 (1973) 2915.

Ammonia-isothiocyanatoborane, $NH_3 . BH_2 . NCS$
($Pnma$) Z=4, R=4.3% for 324 intensities. The distance between the in-plane hydrogen of ammonia and the nearest sulphur is 2.62 Å. A normal bond distance of 1.58(2) Å between four-coordinated nitrogen and four-coordinated boron shows some correlation between distance and relative strengths of donor and acceptor.
D.S. Kendall and W.N. Lipscomb, *Inorg. Chem.*, 12 (1973) 2920.

((Triphenylphosphinemethylide)diphenylphosphine oxide)pentacarbonyltungsten(0), $W(CO)_5(O{=}P)(C_6H_5)_2CHP(C_6H_5)_3$
($P2_1/c$) Z=4, R=5.05% for 1833 independent reflections. The ligand is bound to the tungsten through oxygen; W–O, 2.199(7) Å. There is a pronounced *trans* interaction; the axial W–C bond length of 1.949(10) Å is significantly shorter than the average of the four equatorial lengths, 2.025(6) Å. P–C bond lengths are equal, 1.690(10) Å and the multiple bond order is approximately equally distributed between the two P–C bonds.
S.Z. Goldberg and K.N. Raymond, *Inorg. Chem.*, 12 (1973) 2923.

μ-Hydroxy-bis[pentamminechromium(III)] chloride monohydrate, $[(NH_3)_5Cr{-}OH{-}Cr(NH_3)_5]Cl_5 . H_2O$ (the acid rhodo chloride)
($P4_2/mnm$) Z=4, R=7.7% for 228 reflections. The cation is dimeric, the bridging oxygen atom lying on a site of C_{2v} symmetry with a Cr–O–Cr bridging angle of 165.6(9)° and Cr–O 1.94(1) Å. The coordination around each Cr(III) atom is roughly octahedral. The magnetic susceptibilities in the temperature range 6.8-300°K lead to a value of -31.5 cm^{-1} for the singlet-triplet splitting parameter 2J.
J.T. Veal, D.Y. Jeter, J.C. Hempel, R.D. Eckberg, W.E. Hatfield and D.J. Hodgson, *Inorg. Chem.*, 12 (1973) 2928.

Dinitrato[2-(2-dimethylaminoethyl)pyridine]copper(II), $Cu(DMAEP)(NO_3)_2$
($P2_1/c$) Z=4, R=3.7% for 1576 independent reflections. The copper(II) ions are unsymmetrically coordinated to two bidentate nitrate groups and to the nitrogen atoms of the DMAEP ligand. The geometry at the copper(II) ion is distorted octahedral with elongated axial positions occupied by oxygen atoms from both nitrate groups with Cu–O of 2.468 and 2.474(4) Å; Cu–N, 1.966(3) and 2.014(4);

Cu–O(eq) 1.990(3) and 2.019(4) Å.

D.L. Lewis and D.J. Hodgson, *Inorg. Chem.*, 12 (1973) 2935.

Quinolinium hexabromoantimonate(V) tribromide, $(C_9H_7NH)_2Sb^{IV}Br_9$

($P2_1/n$) $Z=2$, $R=9.0\%$ for 2111 reflections. The slightly distorted octahedral $SbBr_6^-$ ion has C_i symmetry with bond lengths 2.556(3) to 2.581(3) Å. The tribromide ion is also centrosymmetric with bond length of 2.566(3) Å.

S.L. Lawton, E.R. McAfee, J.E. Benson and R.A. Jacobson, *Inorg. Chem.*, 12 (1973) 2939.

μ-(*o*-Tetrafluorophenylene)-diiron octacarbonyl, $C_6F_4Fe_2(CO)_8$

($P2_1/n$) $Z=4$, $R=2.9\%$ for 1942 significant reflections. The analysis reveals that the compound is the *o*-phenylene adduct which was originally proposed. Fe–Fe is 2.797 Å.

M.J. Bennett, W.A.G. Graham, R.P. Stewart and R.M. Tuggle, *Inorg. Chem.*, 12 (1973) 2944.

Dinitrosylbis(μ-diphenylphosphido)-bis(tertiary phosphine)diruthenium, $[Ru(\mu\text{-}PPh_2)(NO)L]_2$ (L - $PMePh_2$, PPh_3)

($P2_1/c$) $Z=2$, $R=5.4\%$ for 1659 reflections. The coordination about each ruthenium atom (excluding the Ru–Ru bond) is a distorted tetrahedron. The nitrosyl is terminally coordinated in a linear manner with an Ru–N distance of 1.697(12) Å and an Ru–N–O bond angle of 174.1(10)°. Ru–Ru is 2.629 Å, Ru–P is 2.315(4) for terminal phosphine and 2.304(4) Å for bridging phosphine.

J. Reed, A.J. Schultz, C.G. Pierpont and R. Eisenberg, *Inorg. Chem.*, 12 (1973) 2949.

Tris(dibenzylideneacetone)palladium(0), $Pd(C_{17}H_{14}O)_3$

($P\bar{1}$) $Z=2$, $R=8.4\%$ for 2449 non-zero reflections. Each pentadiene ligand is coordinated to the metal atom through one olefin group. The coordination about the palladium atom is essentially trigonal with the molecule having approximate C_3 symmetry.

M.C. Mazza and C.G. Pierpont, *Inorg. Chem.*, 12 (1973) 2955.

The benzene solvate of tris(triphenylphosphine)(sulfur dioxide)platinum

($P6_3$) $Z=2$, $R=6.7\%$ for 375 independent reflections. The complex is four-coordinate, essentially trigonal pyramidal with the sulphur dioxide in the apical position. P–Pt–P=118.4(4), P–Pt–S=97.2(2)°. Pt–P=2.80(9); Pt–S, 2.399(13) Å. The benzene solvate was found to have a fractional occupancy of approximately 0.5.

J.P. Linsky and C.G. Pierpont, *Inorg. Chem.*, 12 (1973) 2959.

A new hexamolybdate-cyclophosphazene complex, $[HN_3P_3(NMe_2)_6^+]_2[Mo_2O_{19}^{2-}]$

($P2_1/c$) $Z=2$, $R=2.9\%$ for 3113 unique reflections. The structure is characterized by the presence of puckered, ring-protonated cyclotriphosphazene structures in which long P–N skeletal bonds (1.663, 1.675(5) Å) and short skeletal bonds (1.560–1.599(5) Å) are found in the same ring. The N–P–N angles are 109.6 to 114.5(3)° and P–N–P and 126 to 129(3)°. The $Mo_6O_{19}^{2-}$ ion consists of a slightly distorted cage of six molybdenum atoms located octahedrally around a central oxygen atom with twelve oxygen atoms disposed in Mo–O–Mo units and one terminal oxygen attached to each molybdenum.

H.R. Allcock and E.T. Shawl, *Inorg. Chem.*, 12 (1973) 2963.

trans-Dibromobis[di(*tert*-butyl)fluorophosphine]nickel(II)

($P2_1/n$) $Z=2$, $R=8.8\%$ for 1243 reflections. Ni–Br 2.288(1); Ni–P 2.232(3); P–C, 1.86(1) and 1.88(1); P–F 1.579(7) Å; C–P–F, 97.3(5) and 97.9(5)°. The complex is *trans* square planar and possesses a centre of symmetry.

W.S. Sheldrick and O. Stelzer, *J. Chem. Soc. Dalton*, (1973) 926.

The adduct formed between bis(*cis*-1,2 diphenylethylene-1,2-dithiolato)palladium and cyclohexa-1,3 diene

($P\bar{1}$) $Z=2$, $R=4.7\%$ for 2829 observed intensities. The analysis shows that a 1,8 addition reactions has occurred between donor and acceptor resulting in the formation of two new carbon–sulphur bonds.

G.R. Clark, J.M. Waters and K.R. Whittle, *J. Chem. Soc. Dalton*, (1973) 821.

(Diphenylmethyleneamino)dimesitylborane

($Pbca$) $Z=8$, $R=7.8\%$ for 608 reflections. The $>B\overset{\leftarrow}{-}N{=}C<$ system in the molecule has an allene-type geometry with B–N 1.38(2); C–N 1.29(2) Å and B–N–C 173(2)°.

G.J. Bullen, *J. Chem. Soc. Dalton*, (1973) 858.

Dichlorobis-(*N,N'*-diethylthiourea)cobalt(II) (triclinic form)

($P\bar{1}$) $Z=2$, $R=10.1\%$ for 1303 independent reflections. The structure consists of polymeric chains of molecules connected to each other by hydrogen bonds between chlorine and NH groups. The cobalt is tetrahedrally coordinated. Mean Co–S 2.323; Co–Cl, 2.254 Å.

M. Bonamico, G. Dessy, V. Fares and L. Scaramuzza, *J. Chem. Soc. Dalton*, (1973) 876.

Di-μ-acetato-bis[(methylallyl-3-norbornyl)palladium(II)]

($Pbcn$) $Z=8$, $R=6.2\%$ for 2215 observed reflections. The coordination geometry about each palladium ion is approximately square pyramidal with the vinyl end of the lateral chain of the hydrocarbon ligand being shifted from the 'normal' position in related palladium and platinum complexes. The Pd–O distances show evidence of *trans*-influence by the coordinated carbon atom of the norbornyl moiety.

M. Zocchi, G. Tieghi and A. Albinati, *J. Chem. Soc. Dalton*, (1973) 883.

Trichloro(triphenylphosphine)gold(III)

($P\bar{1}$) Z=2, R=5.0% for 2076 reflections. The structure consists of discrete molecules with near square-planar coordinated gold atoms. Au–P, 2.335(4), Au–Cl 2.273(4), 2.282(4) and 2.347(4) Å.

G. Bandoli, D.A. Clemente, G. Marangolini and L. Cattalini, *J. Chem. Soc. Dalton*, (1973) 886.

Bis[(3-chloropyridine)mercury(I)] diperchlorate

($C2/m$) Z=4 (dimers), R=1.4% for 625 independent reflections. Hg–Hg is 2.487 Å and is approximately coordinated by the ligand. Hg–N, 2.21 Å; Hg–Hg–N, 167.4°.

D.L. Kepert, D. Taylor and A.H. White, *J. Chem. Soc. Dalton*, (1973) 893.

Di-μ-tropolonato-bis[aquo(tropolonato)nickel(II)]

($P2_1/n$) Z=4, R=8.2% for 1191 observed reflections. The molecule exists as a centrosymmetric dimer and contains two types of tropolone ligand; one coordinated to one nickel atom only, the other coordinated to both the first nickel atom and to a second nickel atom via a bridging oxygen atom. This, together with a coordinated water molecule gives the nickel atoms six coordination.

R.J. Irving, M.L. Post and D.C. Povey, *J. Chem. Soc. Dalton*, (1973) 697.

Sodium di-μ-sulphido-bis[(L-cysteinato)oxomolybdate(V)]dihydrate

($P2_1$) Z=2, R=15.1% for 1808 reflections. The structure is similar to that of the corresponding compound with bridging oxygen atoms. Mo–Mo is 2.82 Å. A tetrahydrate also exists. The molybdenum is in distorted octahedral coordination and around each molybdenum atom the three sulphur and one nitrogen atoms are coplanar.

D.H. Brown and J.A.D. Jeffreys, *J. Chem. Soc. Dalton*, (1973) 732.

Tricarbonyl-π-[1,1,1-tricarbonyl-2-methyl-3-diphenylmethylene-6-methoxy-ferra-2-oxacyclohexenyl]iron (Fe–Fe)

($P2_1/c$) Z=4, R=13.8% for 1527 reflections. Both iron atoms have distorted octahedral coordination. The C–C bond lengths in the π-allyl system are 1.38 and 1.44 Å and the mean C–Fe distance to this system is 2.08 Å. Fe–Fe is 2.62 Å. The structure possesses a dative bond from oxygen to iron of 2.07 Å.

J.A.D. Jeffreys, C.M. Willis, I.C. Robertson, G. Ferguson and J.A. Sime, *J. Chem. Soc. Dalton*, (1973) 749.

Dihydrogen ethylenediamminetetra-acetatostannate(II)

($Pbca$) Z=8, R=5.6% for 2130 observed reflections. The tin atom is in the centre of the rectangular face of a distorted triangular prism. Sn–N, 2.388 and 2.442; Sn–O, 2.196, 2.343, 2.635 and 3.057 Å.

K.G. Shields, R.C. Seccombe and C.H.L. Kennard, *J. Chem. Soc. Dalton*, (1973) 741.

Bis(π-2-methylallyl)bis(trimethylphosphite)ruthenium

($P2_1/a$) $Z=4$, $R=5.1\%$ for 577 independent reflections. The ruthenium atom is octahedrally coordinated and has the trimethyl phosphite ligands mutually *cis*; the methylallyl groups each occupy two adjacent sites. The bonding of each methylallyl group to the ruthenium is asymmetric. Mean Ru–C (*trans* to P) 2.38-(2); mean Ru–C (*trans* to C) 2.18(2) Å.

R.A. Marsh, J. Howard and P. Woodward, *J. Chem. Soc. Dalton*, (1973) 779.

4-Chloropyridinium hexachlorostannate(IV)

($P2_1/c$) $Z=2$, $R=3.9\%$ for 1600 independent reflections. The cation is regular and normal. The $SnCl_6^{2-}$ ion is tetragonally distorted with three crystallographically inequivalent Sn–Cl bonds of 2.414(4), 2.419(2) and 2.463(3) Å, the source of the distortion being Cl···H–N hydrogen bonding between the long Sn–Cl bonds of $SnCl_6^{2-}$ and the cation.

R.C. Gearhart, T.B. Brill, W.A. Walsh and R.H. Wood, *J. Chem. Soc. Dalton*, (1973) 359.

Bis[dimethyl(phenyl)phosphine]bis-(5-methyltetrazolato)palladium(II)

($P2_12_12_1$) $Z=4$, $R=4.2\%$ for 1461 reflections. A distorted *cis*-square planar configuration is found around the palladium atom. Both tetrazole rings are nitrogen-(1) bonded, planar and almost regular pentagons with mean bond length of 1.33 Å. There are no unusually short intermolecular distances.

G.B. Ansell, *J. Chem. Soc. Dalton*, (1973) 371.

Dimeric-pyridine 1-oxide mercury chloride perchlorate, $[Hg(C_6H_5NO)_2(ClO_4)_2]$

($P1$) $Z=2$, $R=10.0\%$ for 1502 independent reflections. The mercury coordination does not exhibit the usual two axially coordinated ligands; three of the four pyridine 1-oxide molecules bridge adjacent dimers. Hg–Hg, 2.523(2); Hg–O, 2.19 to 2.77 Å. Mercury thus has a coordination number of four or five.

D.L. Kepert, D. Taylor and A.H. White, *J. Chem. Soc. Dalton*, (1973) 392.

1,*cis*-3 *trans*-5, *trans*-7-Tetrakis(dimethylamino)-1,3,5,7-tetrafluorotetraphosphonitrile

($P2_1/c$) $Z=2$, $R=7.3\%$ for 1281 reflections. The molecule has a centre of symmetry and the eight membered P_4N_4 ring has a chair conformation.

D. Millington, T.J. King and D.B. Sowerby, *J. Chem. Soc. Dalton*, (1973) 396.

Active and meso-forms of *N,N'*butylenebis(salicylideneiminato)cobalt(II)

($P2_12_12_1$) $Z=4$, $R=5.0\%$ for 1226 observed reflections (active). ($P2_1/c$) $Z=4$, $R=3.3\%$ for 2301 observed reflections (meso). The molecules of both compounds are similar, the cobalt atom having a square planar stereochemistry slightly tetrahedrally distorted. The main difference is in the butylene bridge; in the active form the methyl groups are both *pseudo*-axial but in the meso form one is *pseudo*-

axial.

M. Calligaris, G. Nardin and L. Randaccio, *J. Chem. Soc. Dalton*, (1973) 419.

Dibromodiadeniniumcopper(II) dibromide

($C2/c$) Z=4, R=5.6% for 1657 independent reflections. The structure contains complex $[\mathrm{Cu(adenine\ H)_2Br_2}]^{2+}$ cations and bromide anions. In the cation the copper atom has a coordination intermediate between tetrahedral and square planar; Cu–N, 2.013; Cu–Br, 2.361. The copper is coordinated to two nitrogen(9) atoms of unidentate adenine ligands and to two bromine atoms.

P. de Meester and A.C. Skapski, *J. Chem. Soc. Dalton*, (1973) 424.

Carboxylchloro(4-fluorophenyl-di-imide-2*C*,*N'*)bis(triphenylphosphine)iridium(III) tetrafluoroborate acetone solvate

($P2_12_12_1$) Z=4, R=3.2% for 882 reflections. The complex cation contains an ortho-metallated aryl di-imide moiety with acetone of crystallisation weakly hydrogen bonded to the hydrogen atom on N(1), the coordinated nitrogen atom of the azo-function. The octahedral environment of the iridium is distorted by the five-membered ring.

F.W.B. Einstein and D. Sutton, *J. Chem. Soc. Dalton*, (1973) 434.

Bis(ethyl carbamate)dinitratodioxouranium(VI), $[\mathrm{UO_2(NO_3)_2L_2}]$ (L = ethyl carbamate)

($P2_1/a$) Z=2, R=7.6% for 1369 reflections. The linear uranyl group is perpendicular to the equatorial plane in which four oxygen atoms of two nitrate groups and two amidic oxygen atoms of the organic ligands occupy the corners of an irregular hexagon.

R. Graziani, G. Bombieri, E. Forsellini, S. Degetto and G. Marangoni, *J. Chem. Soc. Dalton*, (1973) 451.

trans-Dithiocyanato-bis-[(3,3-dimethylbutynyl)diphenylphosphine]palladium(II), $\mathrm{Pd(SCN)_2(Ph_2P\ .\ C{:}CBu^t)_2}$

($P2_1/c$) Z=2, R=6.9% for 2321 reflections. Both thiocyanate groups are S bonded. There is a centre of symmetry with Pd–P, 2.326(3); Pd–S, 2.336(3) Å; P–Pd–S, 93.7(3)°. Previous explanations for the predominance of N-bonded $\mathrm{CNS^-}$ in palladium(II) complexes are incompatible with the present work in emphasizing the role of π bonding.

G. Beran, A.J. Carty, P.C. Chieh and H.A. Patel, *J. Chem. Soc. Dalton*, (1973) 488.

2,3,6-Triphenylarsenin

($C2/c$) Z=8, R=8.5% for 1648 reflections. The arsenic ring shows no significant departures from mm2(C_{2v}) symmetry and exhibits only two different bond lengths; As–C, 1.872(14); C–C, 1.405(20) Å.

F. Sanz and J.J. Daly, *J. Chem. Soc. Dalton*, (1973) 511.

Tetra(methylgermanium) hexasulphide

($C2/c$) $Z=4$, $R=5.2\%$ for 738 independent reflections. The analysis reveals an adamantane-type structure. Ge–S, 2.218(3); Ge–C, 1.922(10).

R.H. Benno and C.J. Fritchie, *J. Chem. Soc. Dalton*, (1973) 543.

Bis(dimethylphosphonato) mercury(II)

($I2/c$) $Z=4$, $R=11.6\%$ for 804 observed reflections. The mercury atom forms strong bonds with only two phosphorus atoms, P–Hg–P is 165.9° and molecules of $[Hg\{(MeO)_2PO\}_2]$ are clearly distinguishable. Two phosphoryl oxygen atoms from neighbouring molecules interact weakly with mercury making up a very distorted tetrahedral mercury coordination geometry.

G.G. Mather and A. Pidcock, *J. Chem. Soc. Dalton*, (1973) 561.

Di[iodobis-(2,2'-bipyridylamine)-copper(II)]iodide perchlorate

($P2_1/c$) $Z=4$, $R=8.6\%$ for 3921 observed reflections. The two symmetry independent copper(II) moieties in the unit cell adopt essential identical trigonal bipyramidal geometries with two bidentate ligands and an iodide coordinated to each metal ion. The iodide occupies an equatorial position.

J.E. Johnson and R.A. Jacobson, *J. Chem. Soc. Dalton*, (1973) 543.

Tristrimethylammonium catena-tri-μ-chlorocuprate(1–) tetrachlorocuprate(2–)

(Cc) $Z=4$, $R=10.1\%$ for 1480 observed reflections. The compound is unusual in having two types of copper-containing anion, a $[CuCl_3^-]_n$ chain and a $[CuCl_4]^{2-}$ tetrahedron. The crystal lattice symmetry is almost hexagonal and the distortion from hexagonal is attributed to the Jahn–Teller effect in the $[CuCl_3^-]$ chains. The distortion of the tetrahedron approximates to C_{3v}.

R.M. Clay, P. Murray-Rust and J. Murray-Rust, *J. Chem. Soc. Dalton*, (1973) 595.

μ-Hyponitrito-bis[penta-ammine-cobalt(III)]salt, $[Co(NH_3)_5NO]_2Br_{3-x}(NO_3)_{1+x}\cdot 2H_2)$ (where x = 0.5)

($A2/m$) $Z=4$, $R=13\%$ for 1264 independent reflections. The complex is binuclear with the two metal atoms bridged by a hyponitrito-group in a *cis*-asymmetric manner via a nitrogen atom and the oxygen atom bonded to the other nitrogen atom of the hyponitrite group.

B.F. Hoskins and F.D. Whillans, *J. Chem. Soc. Dalton*, (1973) 607.

(A) [CoLI] and (B) [NiLI]I (where I = *N*,*N*-bis-(2-diphenylphosphineoethyl)-*N*-(2-diethylaminoethyl)amine)

Both are ($P2_1/c$) $Z=4$. $R=6.9\%$ for 1875 independent reflections (A) and $R=5.2\%$ for 3093 independent reflections (B). The geometries of both complexes are trigonal-bipyramidal, but the nickel complex is distorted toward an elongated square-pyramid whereas the cobalt complex is distorted toward a tetrahedron. This distortion can account for the different spin states of the two complexes.

A. Bianchi, P. Dapperto, G. Fallini, C.A. Ghilardi and L. Sacconi, *J. Chem. Soc. Dalton*, (1973) 641.

Dicesium-μ_3-chloro-hexachloro-octahedro-hexa-tungstate(II) and molybdate(II), $Cs_2[(W_6Cl_8)Br_6]$ (A), $Cs_2[(Mo_6Cl_8)Br_6]$ (B)

($P31c$) $Z=2$, $R=11\%$ for 665 observed reflections (A). ($P31c$) $Z=2$, $R=12\%$ for 598 observed reflections (B). The anion consists of an octahedral cluster of metal atoms with the chlorine atoms bridging the octahedral faces and the bromine atoms axially coordinated to the M_6 core. The mean metal–metal distances are W–W, 2.620(7) and Mo–Mo, 2.615(6) Å.

P.C. Healy, D.L. Kepert, D. Taylor and A.H. White, *J. Chem. Soc. Dalton*, (1973) 646.

The carbidopentadecacarbonylhexarhodate dianion in its bis(benzyltrimethylammonium) salt, $[NMe_3(CH_2Ph)]_2[Rh_6(CO)_{15}C]$

($C2/c$) $Z=4$, $R=3.3\%$ for 1654 reflections. The anion has precise C_2 and idealized D_{3h} symmetry. The six rhodium atoms define a trigonal prism whose edges are associated with symmetrical bridging CO groups, the remaining six carbonyl ligands being linearly bonded, one per rhodium atom. The carbide ion occupies the centre of the prism. Mean basal and inter-basal Rh–Rh are 2.776(3) and 2.817(2) Å respectively. Rh–C (carbide), 2.134(6) Å.

V.G. Albano, M. Sansoni, P. Chini and S. Martinengo, *J. Chem. Soc. Dalton*, (1973) 651.

Potassium *cis*-bis(iminodiacetato)-cobaltate(II)-2.5 water

($Pbcn$) $Z=8$, $R=5.4\%$ for 1884 reflections. Coordination around cobalt(III) is octahedral and involves two iminodiacetate ions which are terdentate through the nitrogen atom (Co–N, 1.937(4); 1.946(4) Å) and two oxygen atoms from carboxylic groups (Co–O, 1.875(4), 1.883(4); 1.896(4), 1.902(4) Å).

A. Bonamartini Corradi, C.G. Palmieri, M. Nardelli, M.A. Pellinghelli and M.E. Vidoni Tani, *J. Chem. Soc. Dalton*, (1973) 655.

Hexakis(pyridine 1-oxide)mercury(II) bisperchlorate

($R3$) $Z=1$, $R=8.0\%$ for 345 independent reflections. The structure consists of cubic close packed layers of $[Hg(C_5H_5NO)_6]^{2+}$ cations and ClO_4^- ions. The mercury has regular octahedral coordination; Hg–O, 2.35 Å. Distances and angles within the ligand and perchlorate ion are as expected.

D.L. Kepert, D. Taylor and A.H. White, *J. Chem. Soc. Dalton*, (1973) 670.

fac-Chloro-[1,3-bis(dimethylarsino)propane]tricarbonylmanganese

($Pbca$) $Z=8$, $R=11.6\%$ for 783 reflections. The conformation found is that which shows the least interaction between the axial methyl group of the arsenic atoms and the substituents of the metal atom. The coordination geometry of the manganese atom is a slightly distorted octahedral.

C.A. Bear and J. Trotter, *J. Chem. Soc. Dalton*, (1973) 673.