Coordination Chemistry Reviews, 13 (1974) 407–423

X-RAY BIBLIOGRAPHY

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(Received January 3rd, 1974)

Tetramethylammonium *C, C'* diphenylundecahydrodicarba-nido-dodecaborate (1-), $(Me_4N^+)(PhCHB_{10}H_{10}CPh^-)$

($P2_12_12_1$) $Z = 4$, $R = 9.3\%$ for 1544 reflections. The anion is an opened icosahedron and has C_s symmetry disregarding the orientation of the phenyl rings. One carbon is in the $B_{10}C$ fragment while the second carbon bridges two boron atoms in the open face of this fragment, the second carbon is also bonded to hydrogen and to a phenyl group.

E.I. Yolpin and W.N. Lipscomb, *Inorg. Chem.*, 12 (1973) 2257.

Triphenylmethylphosphenium 1, 1, 1-tricarbonyl-4, 6-dicarba-1-mangana-closo-nonaborate(1-), $[(C_6H_5)_3PCH_3]^+[(B_6C_2H_8)Mn(CO)_3]^-$

($P\bar{1}$) $Z = 2$, $R = 4.2\%$ for 2862 independent reflections. Manganese is bonded to two carbon atoms (at 2.04 Å) and three boron atoms (two at 2.35 and one at 2.23 Å) of the carborane cage. The anion has almost exact mirror symmetry and the manganese–carborane cage can be described as a tricapped trigonal prism with one boron and two carbon atoms as the capping atoms.

F.J. Hollander, D.H. Templeton and A. Zalkin, *Inorg. Chem.*, 12 (1973) 2262.

Dicarbonylcyclopentaboranyl iron tricarbonyl, $B_3C_2H_7Fe(CO)_3$

($P2_1/m$) $Z = 2$, $R = 2.4\%$ for 1012 observed reflections. The dicarbacyclopentaboranyl (2^-) ring is planar and the molecule possesses a mirror plane. The angles subtended by the carbonyl carbon atoms and the iron are greater than the expected 90° and the Fe–C distance in the symmetry plane is slightly elongated.

J.P. Brennan, R.N. Grimes, R. Schaeffer and L.A. Sneddon, *Inorg. Chem.*, 12 (1973) 2266.

Tris (*N, N*-diethyldithiocarbamato) iron (III) at 297°K and 79°K, $Fe(S_2CNEt_2)_3$

($P2_1/c$) $Z = 4$, $R = 5.4\%$ for 1752 reflections (297°K); ($C2/n$) $Z = 4$, $R = 6.8\%$ for 2521 reflections (79°K). At room temperature the iron atoms are located on a pseudo-two fold axis which becomes a true two-fold axis in the low-temperature structure. There is

a contraction of the Fe–S distances of 0.05 Å and a small but significant decrease in distortion from the octahedral symmetry of the FeS_6 group.
J.G. Leipoldt and P. Coppens, *Inorg. Chem.*, 12 (1973) 2269.

Bis (pentalenyl) iron, $(C_8H_6)_2Fe$
$(P2_1 2_1 2_1)$ $Z = 4$, $R = 6.65\%$ for 1475 independent reflections. The analysis shows the molecule to be [4, 5, 6, 7, 8, 4′, 5′, 6′, 7′, 8′,-decahapto-1, 1′-dihydro-1, 1′-bipentalenyl] iron. The ferrocene moiety is distorted such that iron–carbon distances are in the range 1.985 (5) Å to 2.077 (5) in ring A and 1.974 (4) to 2.088 (5) Å in ring B. Angle CpA=Fe–CpB (Cp = centroid) is 161.7° and the interplanar angle is 23.6°.
M.R. Churchill and K-K G. Lin, *Inorg. Chem.*, 12 (1973) 2275.

[Fluoroborotris (2-aldoximo-6-pyridyl) phosphine] zinc (II) tetrafluoroborate, $[\{FB(ONCHC_5H_3N)_3P\}Zn^+][BF_4{}^-]$
$(P2_1/c)$ $Z = 4$, $R = 82\%$ for 2728 independent reflections. The zinc (II) atom is in approximate trigonal–prismatic coordination to six nitrogen atoms with Zn–N (aldoximo) = 2.071 (22) Å. The six nitrogen atoms define a slightly tapered trigonal prism in which N (aldoximo)· · ·N (aldoximo) = 2.697 (12) to 2.766 (11) Å; N (pyridyl)· · ·N (pyridyl) = 2.992 (11) to 2.976 (10) Å and N (aldoximo)· · ·N (pyridyl) = 2.557 (10) to 2.617 (13) Å.
M.R. Churchill and A.H. Reis, Jr., *Inorg. Chem.*, 12 (1973) 2280.

Sodium nitrilotriacetatocopper (II) monohydrate, $NaCuN(CH_2OO)_3.H_2O$
$(P2_1 2_1 2_1)$ $Z = 4$, $R = 3.2\%$ for 947 independent reflections. Each copper atom in the structure is effectively chelated by an enveloping nitrilotriacetate (NTA) group by means of four connecting bonds, three Cu–O (1.950 (4) to 2.354 (4) Å and one Cu–N. Two long contacts between neighbouring NTA groups and the copper make up a distorted octahedron around copper. The sodium atoms are bonded to four adjacent NTA groups and a water molecule with Na–O distances from 2.282 (5) to 2.372 (5) Å.
S.H. Whitlow, *Inorg. Chem.*, 12 (1973) 2286.

Bis (triphenylphosphine) (dibenzoylhydrazido) platinum, $Pt[P(C_6H_5)_3]_2[C_6H_5CONNCOC_6H_5].C_2H_5OH$.
$(P\overline{1})$ $Z = 2$, $R = 3.7\%$ for 3906 reflections. The coordination around platinum is distorted square planar. The dibenzoylhydrazide acting as a bidentate ligand, coordinates through an oxygen atom and a nitrogen atom, resulting in a five membered, Pt–N (2)–N (1)–C (1)–O (1) ring. Pt–O (1), 2.016 (5); Pt–N (2), 2.047 (6); Pt–P (*trans* to O (1) and N (2)) 2.258 (2) and 2.268 (2) Å respectively. There is a hydrogen bond between the ethanol molecule and the exocyclic oxygen atom of the dibenzoylhydrazide ligand.
S.D. Ittel and J.A. Ibers, *Inorg. Chem.*, 12 (1973) 2290.

Tetrahydridotetrakis (methyldiphenylphosphine) molybdenum (IV), $H_4Mo[CH_3P(C_6H_5)_2]_4$

($P2_1/c$) $Z = 4$, $R = 7.1\%$ for 3667 reflections. The coordination polyhedron around the eight coordinate molybdenum atom is a somewhat distorted variant of the D_{2d} dodecahedron with hydrogen atoms forming an elongated tetrahedron (A sites) and phosphorus atoms a flattened tetrahedron (B sites). All four hydride hydrogen atoms were located giving an average Mo–H of 1.70 (3) Å.

L.J. Guggenberger, *Inorg. Chem.*, 12 (1973) 2295.

2, 2, 4, 4, 6, 6-Hexaphenylcyclotriarsazene, $[(C_6H_5)_2AsN]_3$

($P\bar{1}$) $Z = 2$, $R = 6.1\%$ for 3818 independent reflections. The As_3N_3 ring of the molecule is slightly puckered. The six As–N bond distances appear to be equivalent with an average value of 1.758 (4) Å.

L.K. Krannick, U. Thewalt, W.J. Cook. S.R. Jain and H.H. Sisler, *Inorg. Chem.*, 12 (1973) 2304.

$Bi_3GaSb_2O_{11}$, a new cubic $KSbO_3$

($Pn3$) $Z = 4$, $R = 5.1\%$. The structure may be regarded as three interpenetrating networks, two of which are identical and have the composition Bi_3O_2. The other network is found in all the cubic $KSbO_3$-related structures and is based on metal–oxygen octahedra which share edges and corners.

A.W. Sleight and R.J. Bouchard, *Inorg. Chem.*, 12 (1973) 2314.

Bis (2-thiobenzaldimino)-2, 6-diacetylpyridinezinc (II)

($P2_1$) $Z = 2$, $R = 5.68\%$ for 1769 independent reflections. The ligand has a helical conformation arising from intramolecular steric interactions. The zinc (II) ion is coordinated to three nitrogen atoms and two sulphur atoms forming a five-coordinate complex with an approximate C_2 axis passing through zinc and the pyridine–nitrogen atom; Zn–S, 2.335 (3); Zn–N, 2.17 (1) Å.

V.L. Goedkin and G.G. Christoph, *Inorg. Chem.*, 12 (1973) 2316.

Dimethylammonium copper (II) formate, $NH_2(CH_3)_2[Cu(OOCH)_3]$

($I2/c$) $Z = 4$, $R = 1.7\%$ for 1009 unique reflections. The copper ion is situated at a centre of symmetry surrounded by six formate ions. Cu–O (eq) 1.969 and 1.971; Cu–O (ax), 2.492 Å. The formate ions act as bridges between copper atoms forming an infinite anti–anti chain through the crystal. C–O (formate), 1.240 and 1.253 Å; ∠O–C–O is 124.39°; C–N (dimethylammonium), 1.467 Å.

E. Sletten and L.H. Jensen, *Acta Crystallogr. B*, 29 (1973) 1752.

Six new polytypes of cadmium iodide

$16H_4$, $18H_4$, $28H_2$, $28H_3$, $36H_1$ and $40H_1$ have been determined and all are ($P3ml$). All six polytypes were found to have grown in syntactic coalescence with other polytypes showing a transformation of structure during growth.

Gyaneshwar and G.K. Chadha and G.C. Trigunayat, *Acta Crystallogr. B*, 29 (1973) 1791.

N, N-Tetramethylenebis-(2-pyridinaldimine) thioureacopper (II) perchlorate, $Cu(C_{16}H_{18}N_4)$-$[SC(NH_2)_2](ClO_4)_2$, $Cu(pib)tu(ClO_4)_2$ (A) and bis-(2, 2′ bipyridylthioureacopper (II) perchlorate, $Cu(C_{10}H_8N_2)_2[SC(NH_2)_2](ClO_4)_2$, $Cu(bipy)_2tu(ClO_4)_2$ (B)

($P2_1/c$) $Z = 4$, $R = 8.7\%$ for 4422 independent reflections (A). ($P2_1 2_1 2_1$) $Z = 4$, $R = 7.2\%$ for 1938 independent reflections (B). In both compounds the coordination around the metal is trigonal bipyramidal and involves four nitrogen atoms from the organic ligand and a sulphur atom from thiourea. In both complexes one of the two perchlorate groups is disordered.

M.B. Ferrari, A.B. Corradi, G.G. Fava, G.G. Palmieri, M. Nardelli and C. Pelizzi, *Acta Crystallogr. B*, 29 (1973) 1808.

Bis-(*N, N*-diethyldithiocarbamate) tin (II), $Sn[S_2CN(C_2H_5)_2]_2$

($P2_1/a$) $Z = 4$, $R = 7.6\%$ for 2285 independent reflections. The structure consists of monomers with each tin atom coordinated to four sulphur atoms by two bidentate ligands. The four sulphur atoms and the tin (II) lone pair form a distorted pyramidal valence polyhedron (square pyramid or trigonal bipyramid) which resembles those of yellow lead (II) oxide and several lead (II) dithioalkyl derivatives. Each bidentate ligand forms a four membered SnS_2C ring with a short (2.574 (2), 2.592 (3) Å) and a long (2.765 (3), 2.819 (3) Å) Sn–S distance.

J. Potenza and D. Mastropaolo, *Acta Crystallogr. B*, 29 (1973) 1830.

Sodium hydrogen oxydiacetate and potassium hydrogen oxydiacetate $MHO(CH_2COO)_2$ where M = Na or K

Both are ($P2_1/c$) $Z = 4$ and $R = 7.4\%$ for 864 independent reflections (Na) and $R = 3.6\%$ for 1749 independent reflections (K). The two compounds are isostructural and contain infinite chains of oxydiacetate ions linked by a short hydrogen bond. The chains are cross-linked by the alkali metal ions. H–O, 1.01 (5) and 1.47 (5); O–O, 2.462 (3) Å in the sodium compound and H–O, 1.05 (3) and 1.44 (3); O–O, 2.480 (2) Å in the potassium compound.

J. Albertsson, I. Orenthe, and H. Herbertsson, *Acta Crystallogr. B*, 29 (1973) 1855.

Gallium thiophosphate, $GaPS_4$

($P2_1/c$) $Z = 4$, $R = 6.6\%$ for 706 reflections. Gallium and phosphorus are each surrounded by four sulphur atoms at the corners of distorted tetrahedra. The structure consists of

puckered hexagonally packed sulphur layers with every other one occupied by gallium and phosphorus cations.
P. Buck and C.D. Carpentier, *Acta Crystallogr. B*, 29 (1973) 1864.

Bis (pyridine-2 carboxylato) copper (II) hydrate (copper picolinate)
($P\bar{1}$) $Z = 1$, $R = 3.2\%$ for 2827 observed reflections. The molecule is symmetric around the copper atom which is octahedrally coordinated by two nitrogen atoms, two carboxylic oxygen atoms and two other carboxylic oxygen atoms belonging to two molecules at $\pm a$ along [100].
R. Faure, H. Loiseleur and G.T. David, *Acta Crystallogr. B*, 29 (1973) 1890.

Barium hexahydroxocuprate (II) and strontium hexahydroxocuprate (II), $Ba_2[Cu(OH)_6]$ and $Sr_2[Cu(OH)_6]$
($P2_1/c$) $Z = 2$, $R = 2.8\%$ for 955 independent reflections (Ba). The structure of the barium compound contains isolated highly distorted $[Cu(OH)_6]^{4-}$ octahedra which appear to be held together primarily by barium ions. The structure may also be regarded as representing a strongly distorted version of the cubic K_2PtCl_6 structure. Cu–O, 1.965 (eq) Å and 2.805 (ax) Å. The configuration of the barium–oxygen polyhedron can best be described as a distorted di-capped trigonal prism with Ba–O distances of 2.642 to 2.954 Å. The strontium compound is isomorphous.
E. Dubler, P. Korber and H.R. Oswald, *Acta Crystallogr. B*, 29 (1973) 1929.

μ-Oxo-bis[*N, N'*-ethylenebis (salicylaldiminato) iron (III)], $[Fe(salen)]_2O$ (salen = *N, N'*-ethylenebis (salicyaldiminato) anion)
($P\bar{1}$) $Z = 2$, $R = 5.2\%$ for 1958 independent reflections. The molecular conformation is very similar to that observed for the same molecule in the two solvated crystal structures. $[Fe(salen)]_2O.2py$ and $[Fe(salen)]_2O.CH_2Cl_2$. The Fe–O–Fe bridge is symmetrical within experimental error with Fe–O, 1.78 Å and the angle Fe–O–Fe, 145°.
J.E. Davies and B.M. Gatehouse, *Acta Crystallogr. B*, 29 (1973) 1934.

Anhydrous uranium tetrachloride, UCl_4
($I4_1/amd$) $R = 12\%$. The uranium atom is surrounded by eight chlorine atoms, four being 2.41 Å from uranium and four 3.09 Å from uranium and the coordination polyhedron being a dodecahedron of $\bar{4}\,2m$ symmetry.
J.C. Taylor and P.W. Wison, *Acta Crystallogr. B*, 29 (1973) 1942.

Lithium maleate dihydrate, $Li_2C_2H_2O_4.2H_2O$
(*Cc* or *C2/c*) $Z = 8$, $R = 4.1\%$ for 1535 reflections. The maleate ion is non-planar and unsymmetrical, the principal bond distances (Å) are C(1)–C(2), 1.495 (3); C(2)–C(3),

1.330 (3); C(3)–C(4), 1.505 (3); C(1)–O(1), 1.277 (2); C(1)–O(2), 2.52 (2); C(4)–O(3), 1.273 (2); C(4)–O(4), 1.252 (2). Each lithium ion has an approximately tetrahedral coordination of oxygen atoms.

W.G. Town and R.W.H. Small, *Acta Crystallogr. B*, 29 (1973) 1950.

$CuZrF_6.4H_2O$

$(P2_1/c)$ $Z = 2$, $R = 4.2\%$ for 818 independent reflections. The structure is built up from infinite$\cdots[ZrF_6]^{2-}\cdots[Cu(H_2O)_4]^{2+}\cdots[ZrF_6]^{2-}$ chains linked by hydrogen bonds.

J. Fischer and R. Weiss, *Acta Crystallogr. B*, 29 (1973) 1955.

$K_2Cu(ZrF_6)_2.6H_2O$

$(P2_1/c)$ $Z = 2$, $R = 5.1\%$ for 1740 independent reflections. The structure is built up of $[Zr_2F_{12}]^{4-}$ complex ions resulting from the association of two pentagonal bipyramids sharing an edge and from $[Cu(H_2O)_6]^{2+}$ octahedral complex ions and K^+ cations linked by hydrogen bonds.

J. Fischer and R. Weiss, *Acta Crystallogr. B*, 29 (1973) 1958.

$Cu_3(ZrF_7).16H_2O$

$(P2_1/c)$ $Z = 2$, $R = 5.1\%$ for 1681 independent reflections. The structure is built up on $[Zr_2F_{14}]^{6-}$ anions resulting from the association of two ZrF_7 antiprisms sharing an edge and from $[Cu(H_2O)_6]^{2+}$ octahedra and $[Cu_2(H_2O)_{10}]^{4+}$ cations which arise from the association of $[Cu(H_2O)_6]$ octahedra sharing an edge.

J. Fisher and R. Weiss, *Acta Crystallogr. B*, 29 (1973) 1963.

$Cu_2ZrF_8.12H_2O$.

$(C2/c)$ $Z = 4$, $R = 4.7\%$ for 1229 independent reflections. The structure is built up of $[ZrF_8]^{4-}$ antiprisms and $[Cu(H_2O)_6]^{2+}$ octahedrons linked by hydrogen bonds.

J. Fischer, R. Elchinger and R. Weiss, *Acta Crystallogr. B*, 29 (1973) 1967.

The tetraphenylarsonium salt of the monoanion of adenine, $[C_{24}H_{20}As]^+[C_5N_5H_4]^-.3H_2O$

$(P2_1/n)$ $Z = 4$, $R = 10.3\%$ for 2418 non-zero reflections. The adenine ion is disordered in the crystal and accurate values for its bond lengths and angles have not been obtained.

T.J. Kistenmacher, *Acta Crystallogr. B*, 29 (1973) 1975.

Platinum silicide and platinum germacide, PtSi and PtGe

The compounds are isostructural with MnP $(Pbnm)$ $Z = 4$. $R = 1.8\%$ for 123 intensities (PtSi) and $R = 6.1\%$ for 131 reflections (PtGe) linear thermal expansivities are discussed.

E.J. Graeber, R.J. Baughman and B. Morosin, *Acta Crystallogr. B*, 29 (1973) 1991.

β-Barium orthotitanate, β-Ba_2TiO_4

($P2_1/n$) $Z = 4$, $R_w = 5.1\%$ for 1693 reflections. The asymmetric unit contains one almost regular TiO_4 tetrahedron (mean Ti–O = 1.808 Å) and two crystallographically distinct barium atoms. A revised bond strength (S) – bond length (R) relationship of the form $S = 0.666\,(R/1.953)^{-5.25}$ is proposed for Ti–O bonds.

K.K. Wu and I.D. Brown, *Acta Crystallogr. B*, 29 (1973) 2009.

Tricarbonylbis (dimethylphenylphosphine) iridium (I) perchlorate, $[Ir(CO)_3(PMe_2Ph)_2]ClO_4$

($P2_1/c$) $Z = 4$, $R = 10.4\%$ for 1020 independent reflections. The coordination of iridium is trigonal bipyramidal with the phosphine ligands in the axial positions. The orientation of the phenyl rings gives the cations approximate C_2 symmetry.

G. Raper and W.S. McDonald, *Acta Crystallogr. B*, 29 (1973) 2013.

Tetra-arsenic tetraselenide, As_4Se_4

($P2_1/n$) $Z = 4$, $R = 7.4\%$ for 684 unique reflections. The structure is isostructural with α As_4S_4 and consists of discrete As_4Se_4 molecules which possess almost exact D_{2d} symmetry. As–As, 2.564; As–Se, 2.388 Å.

E.J. Smail and G.M. Sheldrick, *Acta Crystallogr. B*, 29 (1973) 2014.

Chromium iodide, CrI_2

($Cmc2_1$) $Z = 4$, $R = 11\%$. The structure shows layers of deformed octahedra due to the Jahn–Teller effect.

F. Besrest and S. Jaulmes, *Acta Crystallogr. B*, 29 (1973) 1560.

YSF (β)

($P6_3/mmc$) $Z = 4$, $R = 5.1\%$. The structure consists of compact layers of YF_2 and YS_2 parallel to (001).

N. Rysanek and O. Loye, *Acta Crystallogr. B*, 29 (1973) 1567.

$Co_3U_2O_8$

($Pnnm$) $Z = 2$, $R = 6.3\%$. The cations are octahedrally surrounded by oxygen ions. The octahedra of nearest neighbour cobalt atoms form chains along the c axis.

M. Bacmann, *Acta Crystallogr. B*, 29 (1973) 1570.

As_2Se_3 (A) and As_4Se_4(B)

As_4Se_4 is isomorphic with regular As_4S_4. ($P2_1/c$) and $Z = 4$, As–Se is 2.38 Å. As–As is 2.44 (B). $P2_1/c$, As–Se is 2.40 Å for (A).

A.L. Renninger and B.L. Averbach, *Acta Crystallogr. B*, 29 (1973) 1583.

Tetraindium triselenide, In_4Se_3

(*Pnnm*) $Z = 4$, $R = 6.0\%$ for 754 reflections. Endless interlocking chains run parallel to carbon and consists of five membered indium–selenium rings, the chains being cross-linked by strongly bound indium–indium–indium units to form a continuous sheet. The structure contains the homonuclear triatomic cation $(In_3)^{5+}$.

J.H.C. Hogg, H.H. Sutherland and D.J. Williams, *Acta Crystallogr. B*, 29 (1973) 1590.

Tripotassiumhydrogenpyrophosphate trihydrate, $K_3HP_2O_7.3H_2O$.

($P2_1/c$) $Z = 4$, $R = 5.9\%$ for 1532 reflections. The hydrogen pyrophosphate anion $HP_2O_7{}^{3-}$ has a bent P–O–P bond with a bond angle of 132.8° and the mean P–O (bridge) distance of 1.62 Å. P–O (terminal) is 1.54; P–OH (terminal) is 1.574 Å. A very short hydrogen bond of 2.489 Å involving the acid hydrogen atom is found in the structure.

Y. Dumas, J.L. Galigne and J. Falgueirettes, *Acta Crystallogr. B*, 29 (1973) 1623.

Mercury (I) orthoarsenate, $(Hg_2)_3(AsO_4)_2$

($P2_1/c$) $Z = 2$, $R = 8.5\%$ for 1137 independent reflections. The structure is built up of $Hg_2{}^{2+}$ and $AsO_4{}^{3-}$ ions such that three of the four arsenate oxygen atoms are bonded to three different Hg–Hg pairs, thus forming puckered layers. An effectively tetrahedral coordination about the mercury atoms is established by one mercury atom from the same $Hg_2{}^{2+}$ pair, one oxygen (2.16 to 2.23 Å) and two other oxygens (2.42 to 2.71 Å).

B. Kamenar and B. Kaitner, *Acta Crystallogr. B*, 29 (1973) 1666.

myo-Inositol-magnesium chloride-hydrate (1:1:4)

($P2_1/c$) $Z = 4$, $R = 7.3\%$ for 2433 independent reflections. The magnesium is coordinated to two cis-vicinal hydroxyl groups of inositol and four water molecules in an octahedral geometry. As a consequence of the cation binding the cyclohexane ring geometry in the vicinity of the coordination site is distorted.

G. Blank, *Acta Crystallogr. B*, 29 (1973) 1677.

$2Zn(OH)_2.Zn(NO_3)_2$

($P2_1/c$) $Z = 4$, $R = 12.1\%$ for 419 independent reflections. The structure is related to the $M(OH)_2$ type, based on hexagonal close packed X atoms [$X = O(OH^-), O(NO_3{}^-)$] forming layers perpendicular to *a*. The zinc atoms occupy octahedral holes and are surrounded by $4OH^-$ and $2NO_3^-$.

M. Louer, D. Grandjean and D. Weigel, *Acta Crystallogr. B*, 29 (1973) 1703.

$Zn(OH)_2.Zn(NO_3)_2.2H_2O$ (A) and $Ni(OH)_2.Ni(NO_3)_2.2H_2O$ (B)

($P2_1/c$) $Z = 4$ for both. Both structures are related to the $M(OH)_2$ type and consist of

stacks of three hexagonal close packed layers of atoms X ($X = OH^-$, H_2O, NO_3^-); one half of the octahedral holes are occupied by metal ions.
M. Louer, D. Grandjean and D. Weigler, *Acta Crystallogr. B*, 29 (1973) 1707.

Copper (II) aspirinate, $Cu(C_9H_7O_4)_2$
($P2_1/c$) $Z = 4$, $R = 7.8\%$ for 1635 independent reflections. The structure is made up of binuclear units $Cu_2(C_9H_7O_4)_4$, linked via Cu–O (acetyl) bonds to form polymeric chains parallel to the *a* axis. The metal atoms in each binuclear unit are bridged by four syn–syn carboxylate groups. Cu–Cu, 2.617; Cu–O (acetyl), 2.241 (8) and Cu–O (carboxylate), 1.963 (4) Å.
L.M. Muir, *Acta Crystallogr. B*, 29 (1973) 2033.

Tetracalcium diphosphate monoxide, $Cu_4(PO_4)_2O$.
($P2_1$) $Z = 4$, $R = 3.7\%$ for 3288 reflections. The dimensions of the unit cells of $Ca_4(PO_4)_2O$ and $Ca_5(PO_4)_3OH$ (hydroxyapatite) are simply related. One calcium ion is weakly coordinated to a face of a PO_4 group and the two crystallographically discrete oxide ions are surrounded by a tetrahedron of calcium ions with Ca–O 2.136 (4) to 2.277 (3) Å.
B. Dickens, W.E. Brown, G.J. Kruger and J.M. Stewart, *Acta Crystallogr. B*, 29 (1973) 2046.

$La_2(MoO_4)_3$
($C2/c$) $Z = 12$, $R = 5.5\%$ for 2031 reflections. Lanthanum is in eightfold and molybdenum in slightly distorted tetrahedral oxygen coordination. One third of the oxygen atoms are coordinated by one molybdenum and two lanthanum atoms, the other two-thirds are coordinated by one molybdenum and one lanthanum atom.
W. Jeitschko, *Acta Crystallogr. B*, 29 (1973) 2074.

A potassium salt of a 1:1 mixture of dihydrogen *trans*-aconitate and monohydrogen *trans*-aconitate, $K_3(H_2C_6O_6H_3)(HC_6O_6H_3).2H_2O$
($P\bar{1}$) $Z = 2$, $R = 4.7\%$ for 4075 non-zero weight data. The two anionic species are hydrogen bonded to each other through a short (2.50 Å) hydrogen bond and ions of the mono-ionic species are further hydrogen bonded to each other through a 2.53 Å hydrogen bond.
H.L. Carrell, *Acta Crystallogr. B*, 29 (1973) 2082.

Yttrium fluoroselenite, YSeF
($Pnam$) $Z = 4$, $R = 6.0\%$ The selenium atoms are at the vertex of a slightly deformed octahedron and have four-fold coordination while the three-coordinated fluorine atoms

are at the centre of an yttrium isoceles triangle. The yttrium atoms have seven-fold coordination. The main feature is that there are plane groups of Y–F along two diagonals of the projected selenium hexagons.

N. H-Dung, *Acta Crystallogr. B*, 29 (1973) 2095.

$KICl_2$ (A) and $KICl_2.H_2O$ (B)

($P2_1/C$) $Z = 8$, $R = 5.7\%$ for 1249 independent reflections (A). ($P2_1/m$) $Z = 2$, $R = 6.0\%$ for 542 reflections (B). In the anhydrous compound the two independent dichloroiodide ions are nearly linear and symmetric with average I–Cl lengths of 2.55 Å. In the hydrate the dichloroiodide ion is linear and symmetric by virtue of lying across a centre of symmetry and has an I–Cl bond length of 2.55%.

S. Soled and G.B. Carpenter, *Acta Crystallogr. B*, 29 (1973) 2104.

Dimeric (A) and monomeric (B) forms of *N, N*-diethyldithiocarbamate, $Hg_2(S_2CNEt_2)_4$ and $Hg(S_2CNEt_2)_2$

Both are ($P2_1/c$) with $Z = 2$. $R = 6.7\%$ for 1849 independent reflections (A); $R = 4.5\%$ for 1048 reflections (B). In (A) the α form there are isolated dimeric units and the molecular structure is similar to those of *N,N*-diethyldithiocarbamates of zinc and cadmium. The metal ion is five-coordinate with Hg–S, 2.418, 2.520, 2.663, 2.698 and 3.137 Å. In (B) (β form), the crystals are composed of essentially monomeric bis-(*N,N*-diethyldithiocarbamato) mercury (II). Each mercury atom occupies a centre of symmetry with the two ligand molecules coordinated in a plane with the sulphur atoms. The two independent Hg–S bonds are 2.398 and 2.965 Å.

H. Iwasaki, *Acta Crystallogr. B*, 29 (1973) 2115.

Rubidium tetrachromate, $Rb_2Cr_4O_{13}$.

($P2_1/c$) $Z = 4$, $R = 3.8\%$ for 2089 independent reflections. The structure contains $Cr_4O_{13}{}^{2-}$ ions composed of four CrO_4 tetrahedra joined by shared corners. Cr–O (bridge) 1.691 to 1.846 Å with mean 1.77 Å. Cr–O (non-bridge) 1.576 to 1.621, mean 1.61 Å. The two independent rubidium atoms are each irregularly coordinated to eleven oxygen atoms with Rb–O, 2.88 to 3.47 Å.

P. Lofgren, *Acta Crystallogr. B*, 29 (1973) 2141.

Bis (sarcosinato) nickel (II) dihydrate, $Ni(CH_3.NH.CH_2COO)_2.2H_2O$

(Triclinic) $Z = 1$, $R = 10.3\%$ for 875 reflections. The chelation around nickel is octahedral. One oxygen from the carbonyl group and the nitrogen from sarcosinato residue are bonded to nickel in a plane, while the oxygen atoms of the two water molecules occupy the other two sites. Ni–N (1), 1.989 (9); Ni–O (1), 1.880 (8); Ni–OH_2, 2.236 (8) Å.

S. Guha, *Acta Crystallogr. B*, 29 (1973) 2167.

Sodium niobate, low temperature phase, N.

$(R3c)$. The NbO_6 octahedra remain nearly regular, but are tilted about the triad axis and displacements of niobium atoms from the centres of octahedra are parallel to this axis. The temperature dependence of the lattice parameter of both phases, N and P, below room temperature is reported.

C.N.W. Darlington and H.D. Megaw, *Acta Crystallogr. B*, 29 (1973) 2171.

5 $MnCl_2.14\ C_2H_5OH$

$(P\bar{1})$ $Z = 1$, $R = 5.4\%$ for 2273 reflections. The manganese atoms are octahedrally coordinated. The three dimensional arrangement of atoms forms chains along *c*.

P. L'Haridon and M.T. LeBihan, *Acta Crystallogr. B*, 29 (1973) 2195.

Lead metaniobate, $PbNb_2O_6$

$(Bb2_1m)$ $Z = 40$, $R = 11.1\%$ for 328 reflections. The NbO_6 octahedron framework is very similar to those of previously determined tungsten bronze structures. The lead atoms fill all the pentagonal and half the tetragonal sites.

P.H. Labbe, M. Frey and G. Allais, *Acta Crystallogr. B*, 29 (1973) 2204.

μ-Oxy-bis (triphenylazidoantimony), $[(C_6H_5)_3SbN_3]_2O$

$(C2/c)$ $Z = 4$, $R = 4.4\%$ for 1982 reflections. The antimony atom in the asymmetric unit is in a slightly distorted trigonal bipyramidal environment by the bridging oxygen at one vertex and the azide group at the other. Sb–O (bridge), 1.985 (3); Sb–N (1), 2.236 (8); Sb–C, 2.155 (8) Å mean.

G. Ferguson and D.R. Ridley, *Acta Crystallogr. B*, 29 (1973) 2221.

Hexalead pentaborate, $6PbO.5B_2O_3$

$(P\bar{1})$ $Z = 1$, $R = 6.5\%$ for 2874 independent reflections. The structure contains isolated $B_{10}O_{21}{}^{12-}$ polyions which are built up from two diborate groups linked by two BO_3 triangles. The lead atoms are covalently bonded to oxygen (Pb–O 2.23 Å upwards) as well as being asymmetrically coordinated

J.K.-Moe and P.S. W-Hanson, *Acta Crystallogr. B*, 29 (1973) 2243.

Tetrapyridine-bis (diethylphosphorothioato) magnesium (II)

$(P2_1/c)$ $Z = 2$, $R = 4.9\%$ for 2101 reflections. Discrete molecules of the centrosymmetric complex show approximate octahedral coordination through pyridine nitrogen atoms and phosphorothioate oxygen atoms. Sulphur atoms are not involved in coordination. P–S, 1.951; P–O, 1.495 Å are consistent with an almost doubly bonded, nearly neutral sulphur atom and a singly bonded, negatively charged oxygen atom.

C.H. Schwalbe, R. Goody and W. Saenger, *Acta Crystallogr. B*, 29 (1973) 2265.

β-LiIO$_3$

($P4_2/n$) $Z = 8$, $R = 5.9\%$ for 836 (F_o>O) independent reflections. The structure consists of discrete IO$_3$ groups in the form of disorted trigonal pyramids and distorted LiO$_4$ tetrahedra. Each tetrahedron shares two corners with two other tetrahedra. I–O, 1.79; Li–O,*1.98 Å.

H. Schulz, *Acta Crystallogr. B*, 29 (1973) 2285.

Lead phthalocyanine

($P2_1/b$) $Z = 4$, $R = 5.7\%$ for 349 independent reflections. The best plane through the isoindole ring of a PbPc molecules makes an angle of 76° with the fourfold axis of the molecule and the lead atom deviates by 0.4 Å from the plane.

K. Ukei, *Acta Crystallogr. B*, 29 (1973) 2291.

Li$_2$ZrF$_6$

($P\bar{3}1m$) $Z = 1$, $R = 6.05\%$ for 368 independent reflections. Each ZrF$_6^{2-}$ ion is coordinated by twelve Li$^+$ ions. Zr–Li is 3.6971 (5) Å. Each Zr^{4+} is coordinated by six F$^-$ ions at the corners of a regular octahedron.

G. Brunton, *Acta Crystallogr. B*, 29 (1973) 2294.

Tetraphenylphosphonium trichloro-(2-5-dimethylhex-3-yne-2, 5-diol) platinate (II).

($P2_1/c$) $Z = 2$, $R = 5.4\%$ for 2752 independent reflections. The anion [PtCl$_3$(C$_8$H$_{14}$O$_2$)]$^-$ is a square planar complex. The 2,5-dimethylhex-3-yne-2,5-diol behaves as a monodentate ligand and is coordinated to the metal through its triple bond by a π-type bond. Platinum and the three chlorines lie almost exactly in a plane.

R. Spagna and L. Zambonelli, *Acta Crystallogr. B*, 29 (1973) 2302.

Dicarbonylnitrosyltriphenylarsine cobalt (0)

($P\bar{1}$) $Z = 2$, $R = 8.4\%$ for 1499 reflections. The structure consists of discrete molecular units. The coordination around cobalt is approximately tetrahedral and nitrogen and carbon atoms in the nitrosyl and carbonyl groups are disordered.

G. Gilli, M. Sacerdoti and G. Reichenbach, *Acta Crystallogr. B*, 29 (1973) 2306.

Acetato (dimethyl) indium (III)

($Pnma$) $Z = 4$, $R = 4.0\%$ for 527 reflections. The six coordinate indium lies on a mirror plane and is bonded to two approximately *trans*-methyl groups (which also lie in the mirror plane) and four oxygen atoms. A bidentate acetate group lies across the mirror plane with In–O, 2.371 (5) Å while two weaker bridging contacts from different adjacent acetate groups with In–O, 2.600 (5) Å serve to link the structure as a linear polymer.

F.W.B. Einstein, M.M. Gilbert and D.G. Tuck, *J. Chem. Soc. Dalton*, (1973) 248.

Bis (ethylenediamine) copper (II) thiocyanate perchlorate

(*Pnam*) $Z = 4$, $R = 8.3\%$ for 1090 observed reflections. The copper ion is in a tetragonally distorted octahedral environment with four nitrogen atoms from ethylenediamine in an approximately square planar configuration (Cu—N, 2.02 Å) and the thiocynate nitrogen atoms in the tetragonal positions (Cu—N, 2.73 Å).

M. Cannas, G. Carta and G. Marongiu, *J. Chem. Soc. Dalton*, (1973) 251.

Bis (*N,N*-diethyldithiocarbamate) mercury (II)

($P2_1/a$) $Z = 2$, $R = 12\%$ for 810 reflections. The centrosymmetric mercury atoms have three pairs of sulphur contacts in a highly distorted array, one pair close [Hg—S, 2.397 (6) Å] and the others arising out of a sharing of the second sulphur between adjacent mercury atoms [Hg—S, 2.990 (7), Hg—S′, 3.307 (7) Å]. The CS_2 geometry is distorted; C—S, 1.69 (2) and 1.76 (2) Å; S—C—S, 120 (1)°.

P.C. Healy and A.H. White, *J. Chem. Soc. Dalton*, (1973) 284.

trans-Bis (acetylacetonato) di-iodoplatinum (IV), Pt $(acac)_2I_2$

($P\overline{1}$) $Z = 1$, $R = 4.0\%$ for 654 independent reflections. Each platinum (IV) is octahedrally coordinated by two oxygen-chelated acetylacetonate ligands and by two iodine atoms in a *trans*-configuration which deviates only slightly from D_{2h} symmetry.

P.M. Cook, L.F. Dahl, D. Hopgood and R.A. Jenkins, *J. Chem. Soc. Dalton*, (1973) 294.

Di-μ_3-arsino-tris(tricarbonylium) (3Fe—Fe), $As_2[Fe(CO)_3]_3$

(*Bmmb*) $Z = 4$, $R = 10.7\%$ for 630 independent reflections. The molecule is characterized by an equilateral triangle of iron atoms, mean Fe—Fe is 2.62 (1) Å. The two arsenic atoms lie above and below the iron triangle and are related by a mirror plane containing the iron atoms. Both arsenic atoms are bonded equally to the iron triangle, mean As—Fe, 2.35 Å. Other mean bonds lengths. Fe—C, 1.80; C—O, 1.12 Å.

L.J.J. Delbaere, L.J. Kruczynski and D.W. McBride, *J. Chem. Soc. Dalton*, (1973) 307.

Chlorobis (thiourea) mercury (II) chloride

(*Pmmn*) $Z = 2$, $R = 8.0\%$ for 359 reflections. The mercury atom is coordinated in an unusual approximately trigonal planar conformation by the two equivalent thiourea sulphur atoms and a chlorine atom. Hg—S, 2.42 (1); Hg—Cl, 2.57 (1); S—Hg—Cl 110.8° (2). The other 'ionic' chlorine atom lies normal to this plane, between and equidistant from a pair of mercury atoms at 3.22 Å. The thiourea molecules, mercury and coordinated chlorine atoms are coplanar.

P.D. Brotherton, P.C. Healy, C.L. Raston and A.H. White, *J. Chem. Soc. Dalton*, (1973) 334.

trans-Chlorohydridobis (triethylphosphine) palladium

($P2_1/c$) $Z = 4$, $R = 7.2\%$ for 1807 observed reflections. The two phosphorus atoms which are mutually *trans*, and the chlorine atom are situated at three corners of a distorted square, centred on the palladium, with the fourth site believed to be occupied by hydrogen. Pd–Cl is 2.427 Å.

M.L. Schneider and H.M.M. Shearer, *J. Chem. Soc. Dalton*, (1973) 354.

The 1:1 adduct between diphenyl sulphoxide and mercury (II) chloride

($P\bar{1}$) $Z = 2$, $R = 5.0\%$ for 2967 reflections. The coordination around mercury involves the diphenyl sulphoxide molecule via the oxygen atom at 2.58 Å, two close chlorine atoms at 2.291 and 2.289 Å and two chlorine atoms of neighbouring molecules at 3.230 and 3.284 Å. A sixth position is occupied by a phenyl ring at 3.51 Å.

P. Biscarini, L. Fusina, G.P. Nivellini, A. Mangia and G. Pelizzi, *J. Chem. Soc. Dalton*, (1973) 159

Dimethyl dinitrato tin (IV)

($P2_1/c$) $Z = 4$, $R = 8.9\%$ for 1258 reflections. Within each molecule the two nitrate groups are almost coplanar and are bonded to the tin atom in an unsymmetrical bidentate manner. The two C–Sn bonds (2.11 and 2.12 Å) make an angle of 144° and their plane is very nearly perpendicular to that of the nitrate groups.

J. Hilton, E.K. Nunn and S.C. Wallwork, *J. Chem. Soc. Dalton*, (1973) 173.

Copper (I) complex of 2,5-dithiahexane-1,6-dicarboxylic acid

($P2/n$) $Z = 2$, $R = 8.1\%$ for 1339 reflections. The copper atoms, tetrahedrally surrounded by the sulphur atoms of two complexing molecules, are on special positions on diad axes.

H. van der Meer, *J. Chem. Soc. Dalton*, (1973) 1.

μ-[1, 2-Bis (phenylthio) ethane] -bis[chlorogold (I)]

($P\bar{1}$) $Z = 2$, $R = 7.4\%$ for 1040 independent reflections. The asymmetric unit contains two independent C_7H_7SClAu half dimer units. Thus both dimers are centrosymmetric with planar S–C–C–S linkages. Each gold atom is bonded to a sulphur (2.258 (11) and 2.260 (12) Å) and to a chlorine (2.329 (8) and 2.293 (10) Å) with almost linear Cl–Au–S groups (177.8 (4) and 173.5 (4)°).

M.G.B. Drew and M.J. Riedl, *J. Chem. Soc. Dalton*, (1973) 52.

Di-μ-chloro-bis[dichlorocobaltate (II)]-bischloroform

($P2_1/c$) $Z = 2$, $R = 9.3\%$ for 2695 observed reflections. The cation is similar to that in a related copper complex, cobalt being bonded to four nitrogen atoms of the phosphazene ring and to one chlorine atom in a configuration between square pyramid and trigonal

bipyramid. The arsenic cobalt is present as the hitherto unrecognised $Co_2Cl_6^{2-}$ ion, in which two chlorine bridges complete tetrahedral coordination about the cobalt atoms.
W. Harrison and J. Trotter, *J. Chem. Soc. Dalton*, (1973) 61.

Ethylzinc iodide
(*Pnma*) $Z = 4, R = 11.0\%$ for 335 reflections. In the coordination polymer each zinc atom is 2.64 Å from an iodine in the same mirror plane and 2.91 Å from iodines in mirror planes half-a-cell translation above and below, Zn–C, 1.95 and I–Zn–C is 144.4°.
P.T. Moseley and H.M.M. Shearer, *J. Chem. Soc. Dalton*, (1973) 64.

Anhydrous potassium carbonate.
($P2_1/c$) $Z = 4, R = 12.9\%$ for 316 reflections. The structure consists of a slightly distorted hexagonal close packed array of carbonate ions in which potassium ions occupy both octahedral and trigonal bipyramidal sites.
B.M. Gatehouse and D.J. Lloyd, *J. Chem. Soc. Dalton*, (1973) 71.

cis-Diphenylbis-(2,2′-bipyridyl)-chromium (III) iodide.
($C2/c$) $Z = 4, R = 5.4\%$ for 2227 reflections. The organometallic cation, which has the *cis*-octahedral configuration, possesses a crystallographic two-fold symmetry axis. The chromium–carbon σ bond length of 2.087 (4) Å is no different from the corresponding length in the *cis*-$[(2\text{-methoxyphenyl})_2Cr(bipy)_2]^+$ (bipy = 2,2′-bipyridyl) cation.
J.J. Daly, F. Sanz, R.P.A. Sneedon and H.H. Zeiss, *J. Chem. Soc. Dalton*, (1973) 73.

μ-(2,2′:6′,2″-Terpyridylcadmium)-bis(pentacarbonylmanganese) (2Cd–Mn)
($P2_1/c$) $Z = 4, R = 9.11\%$ for 3502 reflections. The coordination of cadmium is very distorted trigonal bipyramidal and there is considerable distortion of the octahedral manganese coordination. The molecule has approximate C_2 symmetry. The equatorial positions around cadmium are occupied by Mn (1), Mn (2) and N (2), and axial by N (1) and N (3).
W. Clegg and P.J. Wheatley, *J. Chem. Soc. Dalton*, (1973) 90.

Octacarbonyl-μ-[1, 2-bis (dimethylarsino)-3, 3, 4, 4-tetrafluorocyclobutene] dimanganese-(Mn–Mn).
($Pna2_1$) $Z = 4, R = 3.5\%$ for 1439 reflections. Each manganese is bonded to an arsenic atom, four carbonyl groups and a manganese atom. Mn–Mn, 2.971 (2); mean Mn–As 2.404 (3) Å. The molecule is twisted about the Mn–Mn bond such that the coordinated groups normal to the bond vector are staggered.
L.Y.Y. Chan and F.W.B. Einstein, *J. Chem. Soc. Dalton*, (1973) 111.

Palladium monothiodibenzoylmethanate

($P2_1/a$) $Z = 4$, $R = 14.7\%$, 1459 independent reflections. The palladium atom is bonded to two sulphur atoms and two oxygen atoms in *cis* positions. The average Pd—S distance is 2.235 Å and is due to the *cis*-positioning of the sulphur atoms and the strengthening of the d_π-p_π M–S interactions. Pd—O is 2.10 Å and the bond is weakened by the strong labilizing effect of the sulphur atom *trans* to the oxygen atom.

L.M. Shkonikova, Yu. M. Yutal, E.A. Shugam and A.N. Knyazeva, *J. Struct. Chem.*, 14 (1973) 80.

π-Cyclopentadienyl-σ-methylrhenium dicarbonylbromide

($P2_1/a$) $Z = 2$, $R = 11\%$ for 1200 independent reflections. The molecular geometry is that usual for haeme-sandwich semicoordinate complexes of the transition metals. Re—CH_3 is 2.32 Å, the Br—Re—CH_3 angle is 139°, and the mean distance from the rhenium atom to the ring carbon atoms is 2.31 Å.

G.G. Aleksandrov, Yu T. Struchkov and Yu V. Markarov, *J. Struct. Chem.*, 14 (1973) 86.

1-(α-naphthyl) Germantrane, $C_{10}H_7Ge(OCH_2CH_2)_3N$

($Pna2_1$) $Z = 4$, $R = 10.3\%$ for 1023 independent reflections. The coordination polyhedron of the germanium atom is a distorted trigonal bipyramid and the length of the Ge—N bond is 2.24 Å. The germanium atom is displaced from the equatorial plane by 0.25 Å toward the carbon atom of the naphthyl group.

A.A. Kemme, Yu Yu Bleidelis, R.P. Shibaeva and L.O. Atovmyan, *J. Struct. Chem.*, 14 (1973) 90.

$K_4Ln(NCS)_7nH_2O$ where Ln is Nd or Eu and $n = 4$ and 6 respectively.

($\beta 2/P$) $Z = 4$, $R = 14.0\%$ for 2300 independent reflections. ($\beta 2/b$) $Z = 4$, $R = 11\%$ for 1780 independent reflections. The rare earth atoms are located on the two-fold axes in the centre of dodecahedra which are formed by four water molecules and four nitrogen atoms from the thiocyanate groups. Each trapezoid of the dodecahedron contains two oxygen atoms from the water molecules on the type A vertices and two nitrogen atoms from the thiocyanate groups on the β type vertices. The other three thiocyanate groups are not coordinated to rare earths.

P.I. Lasarev, V.M. Ionov, L.A. Aslanov and M.A. Porai-Koshits, *J. Struct. Chem.*, 14 (1973) 151.

$ErNO_3(NCS)_2.2[N_4(CH_2)_6].8H_2O$

$R = 16\%$ for 1600 independent reflections. The coordination polyhedron of erbium is a tricapped trigonal prism with coordination number nine. The inner coordination sphere is made up of six water molecules, a bidentate NO_3 group and a nitrogen atom from the

thiocyanate group.
P.F. Lazarev, L.A. Aslanov, and M.A. Porai-Koshits, *J. Struct. Chem.*, 14 (1973) 153.

$Ni(NH_4)_2[(UO_2)_2F_8].6H_2O$
($P2_1/b$) $Z = 2$, $R = 8.5\%$ for 2000 independent reflections. The structure is that of the island type with $[(UO_2)_2F_8]^{4-}$ anions, nickel and ammonium cations and water molecules. The dimeric anion can be regarded as two pentagonal bipyramids having a common F–F edge. The UO_2 group is linear and symmetrical if perpendicular to the equatorial plane of the bipyramid.
Yu N. Mikhailov, A.A. Udovenko, V.G. Kuznetsov, L.A. Butman and L.A. Kokh, *J: Struct. Chem.*, 14 (1973) 154.