Coordination Chemistry Reviews, 11 (1973) 71–89

X-RAY BIBLIOGRAPHY

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Di-μ-hydroxo-bis[2-(2-ethylaminoethyl)pyridyne] dicopper(II)perchlorate, $[\mathrm{Cu(C_4H_{14}N_2)\text{-}OH}]_2(\mathrm{ClO_4})_2$

($P2_1$) $Z = 2$, $R = 4.2\%$ for 1710 independent reflections. The complex consists of pairs of copper atoms linked by two hydroxo bridges. The coordination geometry of each copper is roughly tetragonal-pyramidal, the basal plane consisting of the two bridging oxygen atoms and the two nitrogen atoms of the ligand. The axial site is occupied by a perchlorate-anion oxygen atom. Cu–O = 1.916(bridge), Cu–Cu = 2.917 Å.

D.L. Lewis, W.E. Hatfield and D.J. Hodgson, *Inorg. Chem.*, 11 (1972) 2216.

Tris(octamethylmethylenediphosphonic diamide)copper(II) perchlorate, $\mathrm{Cu\{[(CH_3)_2N]_2\text{-}P(O)CH_2P(O)[N(CH_3)_2]\}_3(ClO_4)_2}$

($Pna2_1$) $Z = 4$, $R = 4.6\%$ for 6305 reflections. The $\mathrm{CuO_6}$ group is a slightly distorted octahedron with each chelate ring in a boat conformation. The bond in one of the chelate rings is opposite that of the other two. Cu–O = 2.085(average), P–O = 1.483(5), P–C(P) = 1.798(13), ∠O–Cu–O = 89.9(12)°, ∠Cu–O–P = 132.1(14)°, ∠O–P–C = 110.5(6)°, ∠P–C–P = 115.3(15)°.

P.T. Miller, P.G. Lenhert and M.D. Joesten, *Inorg. Chem.*, 11 (1972) 2221.

catena-Bis[μ-(*O,O'*-di-isopropyldithiophosphato)digold(I)](*Au–Au*), $\{[\mathrm{Au(i\text{-}C_3H_7O)_2\text{-}PS_2}]_2\}_n$

($P\bar{1}$), $Z=4$, $R=6.2\%$ for 2194 significant independent reflections. The structure consists of two crystallographically independent dimers, each of which contains one gold–gold bond bridged through sulphur by two bidentate $\mathrm{S_2P(OC_3H_7)_2}$ ligands. Au–Au = 2.914(5)–3.109(6). Each gold is in a distorted square-planar environment of two sulphur and two other gold atoms. Au–S(average) = 2.28 Å.

S.L. Lawton, W.J. Rohrbaugh and G.T. Kokotailo, *Inorg. Chem.*, 11 (1972) 2227.

Americium sulphate octahydrate, $Am_2(SO_4)_3 \cdot 8H_2O$

($C2/c$) $Z=4$, $R=3.5\%$ for 3035 measured intensities. Each americium atom is coordinated by four sulphate oxygen atoms (2.382–2.509 Å) and by four water molecules at 2.406–2.553 Å. The coordination is intermediate between an antiprism and a dodecahedron.

J.H. Burns and R.D. Baybarz, *Inorg. Chem.*, 11 (1972) 2233.

Carbonylchlorobis(trimethylphosphine)(1,2,3-triphenylpropenylium-1,3-diyl)iridium-(1+) tetrafluoroborate(1−)-dichloromethane, $[Ir(C_3(C_6H_5)_3)Cl(CO)(P(CH_3)_3)_2][BF_4] \cdot CH_2Cl_2$

($P2_12_12_1$) $Z=4$, $R=6.0\%$ for 3248 intensities above background. In reacting with $IrCl(CO)[P(CH_3)_3]_2$ the cyclopropenium ring is cleaved to give the monopositive bidentate propenylium-1,3-diyl group. This group, and the *trans* phosphines, chlorine, and carbonyl are arranged around the six-coordinate iridium in a distorted octahedral array. Ir–P = 2.360, 2.369; Ir–Cl = 2.472; Ir–CO = 1.94; Ir–C(propenylium) = 2.099, 1.990 Å; C–C(propylenium) = 1.36, 1.41 Å.

R.M. Tuggle and D.L. Weaver, *Inorg. Chem.*, 11 (1972) 2237.

(Cyclo-octa-1,5-diene)bis(dimethylphenylphosphino)methyliridium(I), $(C_8H_{12})[(C_6H_5)(CH_3)_2P]_2Ir(CH_3)$

($Pbca$) $Z=8$, $R=6.8\%$ for 1724 independent non-zero reflections. The iridium atom is trigonal-bipyramidal with two equatorial dimethylphenylphosphine ligands. Ir–P = 2.316, 2.329 and axial methyl Ir–C = 2.202(22) Å. The bidentate cyclo-octa-1,5-diene spans the remaining axial and equatorial positions.

M.R. Churchill and S.A. Bezman, *Inorg. Chem.*, 11 (1972) 2243.

Tetramethylammonium nonabromodiantimonate(III) dibromine, $[(CH_3)_4N]_3Sb_2Br_9 \cdot Br_2$

($P6_3/mmc$) $Z=2$, $R=13.7\%$ for 289 independent reflections. The structure contains $Sb_2Br_9{}^{2-}$ anions (in which $Sb^{III}Br_6$ octahedra share one face) bridged by Br_2 molecules. The cations as well as the Br_2 molecules are disordered.

C.R. Hubbard and R.A. Jacobson, *Inorg. Chem.*, 11 (1972) 2247.

Calcium terephthalate trihydrate

($P2_1/c$) $Z=4$, $R=13.5\%$ for 1311 observed reflections. The calcium atom is eight-fold coordinated to four oxygens of three terephthalic acid ions and four water molecules. Each carbonyl twists by about 5° from the plane of the benzene ring.

T. Matsuzaki and Y. Iitaka, *Acta Crystallogr., Sect. B*, 28 (1972) 1977.

$Cs_2UO_2F_4 \cdot H_2O$

($P2_1/c$) $Z=4$, $R=14.8\%$ for 1300 reflections. The structure consists of Cs^+ ions, water molecules and $U_2O_4F_8{}^{4-}$ anions. The latter is a dimer formed by two $UO_2F_5{}^{3-}$ pentagonal bipyramids sharing a common edge of the pentagonal base. U–F(bridging) = 2.4, U–F (terminal) = 2.2 Å. U–U (intra-dimer) = 4.04 Å; (inter-dimer) = 6.19 Å.

N.Q. Dao, *Acta Crystallogr., Sect. B*, 28 (1972) 2011.

μ(α, ω-Octadi-π-enyl)bisbromotri-isopropylphosphinenickel(II) (A) and μ(α, ω-Octadi-π-enyl) bisbromo(diphenylphosphinoethane)nickel(II) (B)

($P\bar{1}$) $Z = 1$, $R = 8.9\%$ for 1921 reflections (A) and ($A2/a$) $Z=4$, $R = 9.3\%$ for 1248 reflections (B). Both complexes are centrosymmetric dimers. In (A) the nickel is square-planar coordinated. Ni–Br = 2.313; Ni–P = 2.209; Ni–C = 1.99 and 2.12 Å. In (B) nickel is five-coordinate square-pyramidal, Ni–P (mean) = 2.19, Ni–C(mean) = 2.09, Ni–Br = 2.66 Å.

T.S. Cameron and C.K. Prout, *Acta Crystallogr., Sect. B*, 28 (1972) 2021.

Trihydrazinium hexafluorochromate, $(N_2H_5)_3CrF_6$

($P2_12_12_1$) $Z=4$, $R=9.9\%$ for 875 observable intensities. The structure consists of $CrF_6{}^{3-}$ octahedra and $N_2H_5{}^+$ cations connected by NH··F hydrogen bonds to form a three-dimensional network. N··F(shortest) = 2.655, Cr–F(mean) = 1.905; N–N = 1.390–1.466 Å.

B. Kojić-Prodić, S. Šćavičar, R. Liminga and M. Šljukić, *Acta Crystallogr., Sect. B*, 28 (1972) 2028.

Bis(hexamethylbenzene)ruthenium(0)

($P2_1/c$) $Z=4$, $R=4.3\%$ for 2969 independent reflections. The two hexamethylbenzene rings are non-equivalent; one is planar and bonded to the metal by six carbon–metal linkages, the other is bent along the 1, 4 axis by 42.8°. Only four adjacent carbons bond to the metal forming a butadiene-like fragment.

G. Huttner and S. Lange, *Acta Crystallogr., Sect. B*, 28 (1972) 2049.

Ennea-ammonium dichloride tetrathiosulphatoargentate, $[NH_4]_9Cl_2[Ag(S_2O_3)_4]$

($I\bar{4}2d$) $Z=4$, $R=8.4\%$ for 774 independent reflections. The structure consists of $[Ag(S_2O_3)_4]^{7-}$, ammonium and chloride ions. The thiosulphato group is monodentate, bound to silver via sulphur. The $S_2O_3{}^{2-}$ groups are approximately tetrahedrally arranged around silver; Ag–S = 2.581. The central sulphur of each $S_2O_3{}^{2-}$ is approximately tetrahedral. S—O = 1.46, 1.47, 1.48; S—S = 2.009 Å.

F. Bigoli, A. Tiripicchio and M. Tiripicchio-Camellini, *Acta Crystallogr., Sect. B*, 28 (1972) 2079.

Bis(glycinato)bis(imidazole)nickel(II)

($P2_1/c$) Z=4, R=4.9% for 2618 independent reflections. The complex is octahedral and the configuration around the metal ion is *cis*-O(carbonyl), *cis*-N(amino), *cis*-N-(imidazole). Ni—O(carbonyl) = 2.09, Ni—N(amino) = 2.10, Ni—N(imidazole) = 2.07 Å.

H.C. Freeman and J.M. Guss, *Acta Crystallogr., Sect. B*, 28 (1972) 2090.

$3HgBr_2 \cdot CH_3CN$

($C2/c$) Z=4, R=6.8% for 735 intensities. The structure is held together by Hg···Br and Hg···N contacts shorter than van der Waals distances. A short Br···Br contact is also present. The mercury atoms are surrounded either by six bromides or six bromides and one additional nitrogen.

H. Leligny, M. Frey and J.C. Monier, *Acta Crystallogr., Sect. B*, 28 (1972) 2104.

$CsBe_2F_5$

($P4_132$) Z=4. The structure has a complex succession of linked ten-membered rings of BeF_4 tetrahedra.

Y. Le Fur and S. Aléonard, *Acta Crystallogr., Sect. B*, 28 (1972) 2115.

Trimethyltin isocyanate hydroxide $(CH_3)_3SnNCO \cdot (CH_3)_3SnOH$

($P2_1/n$) Z=4, R=13.6% for 1088 independent reflections. Chains of $(CH_3)_3Sn$ groups bridged by alternating nitrogen atoms from NCO and OH oxygen atoms are interconnected by NCO···HO hydrogen bonds to form a layer structure. Sn—N—Sn distance 2.43(5), 2.75(5); Sn—O—Sn = 2.15(5), 2.14(5) Å.

J.B. Hall and D. Britton, *Acta Crystallogr., Sect. B*, 28 (1972) 2133.

Bis-(4-aminoimidazole-5-carboxamidoxime)copper(II) perchlorate

($P2_1/c$) Z=2, R=5.2% for 1458 reflections. This degradation product of adenine *N*-oxide is formed by expulsion of C(2) of adenine and a rotation around the original C(5)—C(6) bond. The copper atom is in square-planar coordination. Cu—N(1) = 1.968, Cu—N(7) = 1.970 Å.

C.D. Stout, M. Sundaralingam and G. Hung-Yin Lin, *Acta Crystallogr., Sect. B*, 28 (1972) 2136.

Nickel(II) octaethylporphyrin, $C_{36}H_{44}N_4Ni$
($I4_1/a$) $Z=4$, $R=7.7\%$ for 1205 independent reflections. The nickel atom is square-planar coordinated. Ni–N(pyrrole) = 1.929(3) Å. This is the shortest metal–nitrogen bond distance found in a metalloporphyrin. The individual pyrrole rings within the macrocycle are markedly non-coplanar.
E.F. Meyer, Jr., *Acta Crystallogr., Sect. B,* 28 (1972) 2162.

Potassium binoxalate, KHC_2O_4
($P2_1/c$) $Z=4$, $R=2.7\%$ for 1266 reflections. Potassium is seven-fold coordinated to oxygens and the structure is composed of parallel layers of anions and cations.
H. Einspahr, R.E. Marsh and J. Donohue, *Acta Crystallogr., Sect. B,* 28 (1972) 2194.

Potassium hydrogen DL-methylsuccinate
($P2_1/c$) $Z=4$, $R=4.0\%$ for 1296 independent intensities. The succinic acid skeleton of the acid methylsuccinate is bent. The short intermolecular hydrogen bond with O(H)···O distance of 2.543(3) Å is acentric.
Y. Schouwstra, *Acta Crystallogr., Sect. B,* 28 (1972) 2217.

$MoO_3 \cdot 2H_2O$
($P2_1/N$) $Z=16$, $R=4.8\%$, 3078 independent reflections. Five oxygens and one coordinated water form a highly distorted octahedron around molybdenum. Mo–O(bridge) = 1.766, 2.156(alternating); Mo–O(terminal) = 1.694; Mo–OH_2 = 2.288 Å.
B. Krebs, *Acta Crystallogr., Sect. B,* 28 (1972) 2222.

Tris(ethylthioxanthato)cobalt(III), $Co(C_2H_5SCS_2)_3$
($P\bar{1}$) $Z=4$, $R=4.6\%$ for 5257 reflections. In the two independent molecules of the asymmetric unit, cobalt is surrounded by six sulphur atoms from the three ligand ions with D_3 symmetry. Co–S(mean) = 2.398(1) Å.
A.C. Villa, A.G. Manfredotti, C. Guastini and M. Nardelli, *Acta Crystallogr., Sect. B,* 28 (1972) 2231.

Cadmium ethylxanthate, $Cd(S_2COC_2H_5)_2$
(Pa) $Z=2$, $R=2.2\%$ for 1903 independent reflections. Each cadmium is tetrahedrally coordinated to four sulphur atoms belonging to different xanthic groups. Each xanthic group bridges two cadmium atoms forming a two-dimensional network. Cd–S = 2.544(2)–2.590(3) Å.
Y. Iimura, T. Ito and H. Hagihara, *Acta Crystallogr., Sect. B,* 28 (1972) 2271.

Potassium *O,O*-dibenzylphosphorodithioate, $KS_2P(O\text{-}CH_2\text{-}C_6H_5)_2$

($P\bar{1}$) $Z=2$, $R=4.3\%$ for 2098 observed reflections. The K^+ ion is coordinated to four sulphurs (3.319 Å average) and to three oxygens (2.971 Å average); P—O = 1.619(3), P—S = 1.957 Å.

J.P. Hazel and R.L. Collin, *Acta Crystallogr., Sect. B,* 28 (1972) 2279.

Bis-(L-tyrosinato)copper(II), $CuC_{18}H_{20}N_2O_6$

($P2_12_12_1$) $Z=4$, $R=4.2\%$ for 2012 reflections. The copper is distorted square-pyramidal with *trans* ligands. The conformations of the two crystallographically independent tyrosyl residues differ.

D. van der Helm and C.E. Tatsch, *Acta Crystallogr., Sect. B,* 28 (1972) 2307.

Nickel(II) ethylmethylglyoxime

($P2_1/c$) $Z=2$, $R=4.8\%$ for 802 observed reflections. Bond distances in the five-membered chelate ring indicate a significant delocalisation of the two conjugated carbon—nitrogen double bonds. Ni—N = 1.861, 1.862 Å.

R.H. Bowers, C.V. Banks and R.A. Jacobson, *Acta Crystallogr., Sect. B,* 28 (1972) 2318.

Methyltin sesquisulphide

($C2/c$) $Z=4$, $R=3.48\%$. The molecule has an adamantane-like structure, Sn and C lying at the corners of tetrahedra and S at the corners of an octahedron.

D. Kobelt, E.F. Paulus and H. Scherer, *Acta Crystallogr., Sect. B,* 28 (1972) 2323.

β-$NH_4Fe(CrO_4)_2$

($Pnma$) $Z=4$, $R=11.3\%$ for 420 reflections. The structure is formed by a three-dimensional frame of FeO_6 octahedra linked by CrO_4 tetrahedra.

A. Hardy and F. Gaboriaud, *Acta Crystallogr., Sect. B,* 28 (1972) 2329.

$BaNaP_3O_9$

($P2_12_12_1$) $Z=4$, $R=7.2\%$ for 1679 independent reflections. The P_3O_9 rings are in the chain form.

C. Martin and A. Mitschler, *Acta Crystallogr., Sect. B,* 28 (1972) 2348.

$CuLi(PO_3)_3$

($P2_12_12_1$) $Z=4$, $R=8.0\%$ for 552 reflections. PO_3 infinite chains twist around 2_1 axes along the *a* direction. LiO_6 and CuO_6 octahedra are connected to form a three-dimensional network.

M. Laugt, I. Tordjman, J.C. Guitel and M. Roudaut, *Acta Crystallogr., Sect. B,* 28 (1972) 2352.

Diphenyl ditelluride, $C_{12}H_{10}Te_2$

($P2_12_12_1$) $Z=4$, $R=7.3\%$ for 1596 independent reflections. The molecule has a non-planar configuration and the planes of the benzene rings are inclined at 20.1°. Te—Te = 2.712(2), Te—C(average) = 2.115(16) Å.

G. Llabres, O. Dideberg and L. Dupont, *Acta Crystallogr., Sect. B,* 28 (1972) 2438.

$Fe^{II}(NCCH_3)_6[Fe^{III}Cl_4]_2$

($P3$) $Z=1$, $R=4.7\%$ for 1650 reflections. The structure is composed of rows of $(FeCl_4)^-$ tetrahedra and $Fe(NCCH_3)_6{}^{2+}$ octahedra.

B.A. Stork-Blaisse, G.C. Verschoor and C. Romers, *Acta Crystallogr., Sect. B,* 28 (1972) 2445.

$Mn_2(PO_4)Cl$

($Pna2_1$) $Z=4$, $R=3.0\%$ for 1097 observed reflections. The structural units can be described as two distorted edge-sharing octahedra of anions about the two non-equivalent manganese atoms which are further linked through edge sharing by phosphate tetrahedra.

J.R. Rea and E. Kostiner, *Acta Crystallogr., Sect. B,* 28 (1972) 2505.

$Mn_2(PO_4)F$

($C2/c$) $Z=8$, $R=2.3\%$ for 1614 independent data. The compound is isostructural with triplite except there is no disorder in the fluorine position.

J.R. Rea and E. Kostiner, *Acta Crystallogr., Sect. B,* 28 (1972) 2525.

$H_3Fe^{III}(CN)_6$ (A) and $H_3Co^{III}(CN)_6$ (B)

Both crystalline in ($P31m$) or ($P\bar{3}1m$) with $Z=1$, $R=3.9\%$ and 7.4% respectively. The structures are built up from layers of $M(CN)_6{}^{3-}$ octahedra with perfect $\bar{3}m$ symmetry. The asymmetrical hydrogen bond in the iron compound is a major factor responsible for the observed piezoelectric effect.

R. Haser, C.E. deBroin and M. Pierrot, *Acta Crystallogr., Sect. B,* 28 (1972) 2530.

$H_{3.5}[Fe^{II}_{0.5}Fe^{III}_{0.5}(CN)_6]H_2O$
($P\bar{3}1m$) $Z=2$. $Fe(CN)_6$ octahedra are in layers interconnected with hydrogen bonds, N–H···N. The channel cavities are occupied by the localised water molecules statistically distributed on twelve equivalent positions surrounding the $Fe(CN)_6$ octahedra of every alternate layer.
R. Haser and M. Pierrot, *Acta Crystallogr., Sect. B*, 28 (1972) 2538.

$H^{II}_{0.6}[Fe^{II}_{3.6}Fe^{III}_{0.4}(CN)_6]\cdot 1.6H_2O$ and $[H_9O_4\cdot H\cdot H_9O_4]^{3+}$
($R\bar{3}m$) $Z=15$, $R=6.5\%$. The structure is made up of sheets of $Fe(CN)_6$ octahedra, normal to the trigonal axis. Rhombohedral symmetry occurs with the substitution of one $Fe(CN)_6$ octahedron in six by a group of eight water molecules. This water aggregate has an octahedral configuration very close in size to the $Fe(CN)_6$ to which it is linked by hydrogen bonds. It is best described as the hydronium ion $[H_9O_4\cdot H\cdot H_9O_4]^{3+}$.
R. Haser and M. Pierrot, *Acta Crystallogr., Sect. B*, 28 (1972) 2542.

$H_{3.33}[Fe^{II}_{0.33}Fe^{III}_{0.67}(CN)_6]\cdot 2.66H_2O$
($P2_1/a$) $Z=6$, $R=13.3\%$. Eight-membered chair-shaped rings of hydrogen-bonded $Fe(CN)_6$ octahedra lie along *c*. These chairs share edges, forming zig-zag chains, and delineate channels along *c* which are occupied by two different kinds of groups of water molecules best described as hydronium ions $[H_5O_2]^+$. Their geometry is probably that of $[H_3O]^+\cdot H_2O$ rather than $[H_2O\text{–}H\cdot H_2O]^+$.
R. Haser, C. Penel and M. Pierrot, *Acta Crystallogr., Sect. B*, 28 (1972) 2548.

Bis(hydrogeno-pyridine-2,6-dicarboxylato)nickel(II) trihydrate, $Ni(HDP)_2\cdot 3H_2O$
($P2_1/c$) $Z=4$, $R=8.7\%$ for 1987 independent reflections. The nickel atom is surrounded by an unsymmetrical arrangement of two nitrogen and four oxygen atoms which belong to a different carboxylic or carboxylato group. Ni–N = 1.967(14), 1.982(15); Ni–O = 2.104(8)–2.207(6) Å.
P. Quaglieri, H. Loiseleur and G. Thomas, *Acta Crystallogr., Sect. B*, 28 (1972) 2583.

Bis(*N-tert.*-butylpyrrole-2-carbaldimino)nickel(II), $(C_9H_{13}N_2)_2Ni(II)$
($Pbcn$) $Z=4$, $R=10.2\%$ for 1160 independent reflections or 4.9% for 649 reflections $F_o^2 \geqslant 3\sigma(F_o^2)$. The configuration about the nickel atom is pseudo-tetrahedral with a dihedral angle of 88.7(3)°. The two independent Ni–N bond distances 2.032(4) and 1.935(5) Å are distinctly different.
C.H. Wei and J.R. Einstein, *Acta Crystallogr., Sect. B*, 28 (1972) 2591.

$K_5[Au_5(CN)_{10}I_2] \cdot 2H_2O$

R=7.2% for 2883 reflections. The structure consists of layers of linear $[Au^{I}(CN)_2]^-$ and planar $[Au^{III}(CN)_2I_2]^-$ ions alternating with layers of K^+ ions and water molecules. The black colour of the complex arises from a mixed-valence interaction between the two anionic groups.

C. Bertinotti and A. Bertinotti, *Acta Crystallogr., Sect. B,* 28 (1972) 2635.

trans-Dichlorotetrakis(thioacetamide)Ni^{II}

($P\bar{1}$) Z=2, R=3.4% for 3005 intensities. The two independent Ni–Cl distances are 2.427(1) and 2.430(1) Å. The four independent Ni–S distances are 2.460(1), 2.466(1), 2.458(1) and 2.459(1) Å. The thioacetamide molecules are planar. Only normal van der Waals distances are found between molecules.

R.L. Girling, J.E. O'Connor and E.L. Amma, *Acta Crystallogr., Sect. B,* 28 (1972) 2640.

$AlCl_3 \cdot o\text{-}CH_3 \cdot C_6H_4COCl$ (A), $AlCl_3 \cdot m\text{-}CH_3 \cdot C_6H_4COCl$ (B) and $AlCl_3 \cdot p\text{-}CH_3 \cdot C_6H_4\cdot COCl$ (C)

($P2_1/c$) Z=4, R_1 = 4.5% for 1226 independent reflections (A); ($P2_1/m$) Z=2, R_1 = 4.3% for 1230 independent reflections (B); ($P\bar{1}$) Z=2, R_1 = 3.9% for 2671 independent reflections (C). The adducts are composed of $AlCl_3$ groups coordinated with the oxygen of the *o*-, *m*- and *p*-toluoyl chloride groups.

B. Chevrier, J.M. Le Carpentier and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 2659.

$SbCl_5 \cdot C_6H_5COCl$ (A) and $SbCl_5 \cdot m\text{-}CH_3COCl$ (B)

Both ($P2_1/m$) Z=2; R=4.5% for 2046 (A) and R = 3.5% for 2135 (B) independent reflections. The adducts are composed of $SbCl_5$ groups coordinated with the oxygen atoms of the benzoyl and *m*-toluoyl chloride groups. The atoms coordinated to antimony form distorted octahedra.

B. Chevrier, J.M. Le Carpentier and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 2667.

2-Methylphenyloxocarbonium hexachloroantimonate, $[o\text{-}CH_3 \cdot C_6H_4CO]^+ [SbCl_6]^-$

($Pbca$) Z=8, R=4.4% for 1797 independent reflections. The salt consists of discrete hexachloroantimonate ions and *o*-methyl phenyloxocarbonium cations.

B. Chevrier, J.M. Le Carpentier and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 2673.

Potassium hydrogen bis(homophthalate), $K(C_9H_7O_4)_2 \cdot (H)$

($C2/c$) Z=4, R=9.9% for 594 independent reflections. There is four-fold coordination

around the potassium ion with K–O = 2.76–2.80 Å.
M.P. Gupta and D.S. Dubey, *Acta Crystallogr., Sect. B*, 28 (1972) 2677.

$(+)_D$-Tris(ethylenediamine)cobalt(III) nitrate
($P2_12_12_1$) Z=4, R=8.4% for 1943 reflections. The coordination about cobalt is essentially octahedral with average Co–N = 1.964(8) Å.
P. Witiak, J.C. Clardy and D.S. Martin, Jr., *Acta Crystallogr., Sect. B*, 28 (1972) 2694.

$(+)_{589}$-Tris[(–)-*trans*-1,2-diaminocyclohexane]cobalt(III) chloride monohydrate ("*ob*" isomer)
($C2$) Z=2, R=3.4% for 1251 observed reflections. The cobalt atom has a distorted octahedral coordination of six nitrogen atoms of the ligand molecules. The distortion of the octahedron is slightly more marked than that observed in the "*lel*" isomer. The central C–C bond in the chelate ring is inclined at 72° with respect to the three-fold axis, the conformation being "*ob–ob–ob*".
A. Kobayashi, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 2709.

$CuNa_2(PO_3)_4$
($C2/c$) Z=4, R=4.2% for 1679 reflections. PO_3 infinite chains cross the cell along the *a* direction. Their period is of four PO_4 tetrahedra.
M. Laügt, I. Torpjman, J.C. Guitel and G. Bassi, *Acta Crystallogr., Sect. B*, 28 (1972) 2721.

Copper 2-pyridylacetate dihydrate, $Cu(C_5H_4N\text{-}CH_3\text{-}CO_2)_2 2H_2O$.
(Pc) Z=4, R=5.5% for 1682 independent reflections. Each copper atom is surrounded by an octahedral arrangement of two pyridine nitrogen atoms and two carboxyl oxygen atoms belonging to the same chelate molecule and of two oxygen (carboxyl) atoms at the vertices, belonging respectively to the upper and lower molecules.
R. Faure and H. Loiseleur, *Acta Crystallogr., Sect. B*, 28 (1972) 2733.

$Mn_2Cl_4 \cdot 3C_2H_5OH$
($P\bar{1}$) Z=2, R=8.9% for 1334 significant reflections. The manganese atoms are octahedrally coordinated. An important deformation of the ethanol molecules has been found in this compound.
P. l'Haridon, M. Le Bihan and Y. Laurent, *Acta Crystallogr., Sect. B*, 28 (1972) 2743.

Two crystalline forms of trichloro(pent-4-enylammonium)-platinum(II), $[PtCl_3(C_5H_{12}N)]$
Both forms ($P2_1/c$) Z=4, R=6.0 for 1245 and R=6.4% for 799 independent reflections, for the orange and yellow forms respectively. In both forms the $[PtCl_3(C_5H_{12}N)]$ zwitterion is a π-complex and platinum is square-coordinated by three chlorine atoms and the double bond of the pent-4-enylammonium ion. Dimensions and geometry are similar in the two compounds. The only significant difference is in the conformation of part of the olefinic chain.
R. Spagna and L. Zambonelli, *Acta Crystallogr., Sect. B,* 28 (1972) 2760.

Azepine iron tricarbonyl, $(C_6H_7N)Fe(CO)_3$
(*Pbca*) Z=2, R=6.3% for ~ 5000 reflections. The $Fe(CO)_3$ group is bonded to a planar *s-cis*-butadiene fragment of the azepine ring. At the end atoms of the complexed butadiene fragment the azepine ring is bent so that its conformation is dihedral.
A. Gieren and W. Hoppe, *Acta Crystallogr., Sect. B,* 28 (1972) 2766.

α-Rubidium iodate, $RbIO_3$
($R3m$) Z=1, R=2.3% for 923 observed reflections. The structure is a distortion of the perovskite structure in which the environment of the iodine atom is unsymmetrical with three near oxygen atoms (I–O = 1.807 Å; ∠O–I–O = 99.63°) and three at 2.753 Å. Rb–O = 3.103–3.77 Å.
N.W. Alcock, *Acta Crystallogr., Sect. B,* 28 (1972) 2783.

$K_2Ge(IO_3)_6$
($R\bar{3}$) Z=1, R=4.0% for 531 independent reflections. Germanium is octahedrally coordinated to six oxygens. Each iodine has three oxygen neighbours at 1.79–1.86 Å and three more at 2.50–2.98 forming a much distorted octahedron. There are two potassium atoms in a tube of 18 oxygens.
F. Schellhaas, H. Hartl and R. Frydrych, *Acta Crystallogr., Sect. B,* 28 (1972) 2834.

The following entries were received on December 28, 1972.

Di-triethanolamine-Ni^{II} dinitrate, $Ni[N(CH_2CH_2OH)_3]_2(NO_3)_2$
($P2_1/c$) Z=2, R=6.8%. Coordination of the triethanolamine to nickel is via nitrogen and two hydroxyl oxygen atoms and the complex is octahedral. Ni–N = 2.115, Ni–O = 2.052 Å.
K. Nielsen, R.G. Hazell and S.E. Rasmussen, *Acta Chem. Scand.,* 26 (1972) 889.

Monoclinic trisodium tris-(pyridine-2,6-dicarboxylato)ytterbate(III) 13-hydrate, $Na_3[Yb(C_7H_3NO_4)_3] \cdot 13H_2O$

($P2_1/c$) $Z=4$, $R=9.3\%$ for 2300 independent reflections. The Yb ion is surrounded by six carboxylato oxygen atoms and three nitrogen atoms, which form a tricapped trigonal prism with Yb–O and Yb–N 2.34–2.43, and 2.50–2.53 Å respectively.

J. Albertsson, *Acta Chem. Scand.*, 26 (1972) 985.

Hexagonal trisodium tris(pyridine-2,6-dicarboxylate)ytterbate(III) mono(sodium perchlorate) decahydrate, $Na_3[Yb(C_7H_3NO_4)_3] \cdot NaClO_4 \cdot 10H_2O$

($P\bar{6}2c$) $Z=2$, $R=7.6\%$ for 583 independent reflections. The Yb is surrounded by six carboxylato oxygens and three nitrogen atoms, which form a distorted tricapped trigonal prism. Yb–O = 2.38, Yb–N = 2.43 Å. Four of the ten water molecules and the perchlorate oxygen atoms were not located and assumed to occupy disordered positions.

J. Albertsson, *Acta Chem. Scand.*, 26 (1972) 1005.

Triclinic trisodium tris(pyridine-2,6-dicarboxylato)neodymate(III) 15-hydrate, $Na_3[Nd(C_7H_3NO_4)_3] \cdot 15H_2O$

($P\bar{1}$) $Z=2$, $R=9.7\%$ for 2366 reflections. The six carboxylate oxygens and three nitrogens form a tricapped trigonal prism around the neodymium ion. Nd–O, Nd–N are 2.37–2.61 and 2.57–2.59 Å respectively. Five waters were not located.

J. Albertsson, *Acta Chem. Scand.*, 26 (1972) 1023.

Dirubidium 1,1,2,2-tetranitroethanediide

($Pccn$) $Z=4$, $R=7.7\%$ for about 1900 reflections. The anions are centred on two-fold rotation axes. The angle between the essentially planar $C(NO_2)_2$ groups is 68°.

B. Klewe, *Acta Chem. Scand.*, 26 (1972) 1049.

The tetragonal form of YbOOH

($P\bar{4}2_1m$) $Z=4$, $R=9.5\%$ for 163 independent reflections. The ytterbium atom is seven-coordinated with oxygen atoms. The structure has weak hydrogen bonding.

A.N. Christensen and R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1171.

Tetra-aquo tris-oxalato-discandium(III) dihydrate, $Sc_2(C_2O_4)_3 \cdot 6H_2O$

($P\bar{1}$) $Z=2$, $R=7.6\%$ for 843 independent reflections. The scandium is eight-coordinated and is surrounded by six carboxylic and two water oxygen atoms in an approximately dodecahedral arrangement. Each oxalate is coordinated to two scandium ions forming

two chelates. Sc—O = 2.18—2.26 Å.
E. Hansson, *Acta Chem. Scand.*, 26 (1972) 1337.

Cis (A) and *trans* (B) isomers of nickel(II) dithiosemicarbazide dinitrate, $Ni(CH_5N_3S)_2 \cdot (NO_3)_2$

($P2_1/a$) $Z=4$, $R=7.3\%$ for 818 significant reflections (A) and ($P\overline{1}$) $Z=1$, $R=5.6\%$ for 1061 significant reflections (B). The bond lengths found in the two structures agree with those of related compounds, with Ni—S in the *trans* complex longer than in the *cis*.
R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1365.

Two modifications of chromium(III) tris(diethyldithiophosphate), $Cr[S_2P(OC_2H_5)_2]_3$

($C2/c$) $Z=4$, $R=8.1\%$ for 1368 reflections. The chromium atoms are surrounded by six sulphur atoms in a slightly flattened octahedron, the angles at the chromium atom in the Cr—S—P—S ring being only 82.3°. Cr—S = 2.42—2.46 Å. S—P(average) = 1.99 Å. The other complex is isomorphous with the corresponding vanadyl complex.
H. Vincents, F. Schousboe-Jensen and R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1375.

$CuTeO_3$

($Pmcn$) $Z=8$, $R=8.4\%$ for 546 independent reflections. Copper and tellurium are linked to form a three-dimensional network. There are two chemically different tellurium atoms in the structure, both of which are strongly bonded to three oxygen atoms at Te—O = 1.86—1.96 Å. One tellurium has a fourth oxygen at 2.32 Å. Copper is surrounded by four oxygens at 1.94—1.98 Å and a fifth at 2.38 Å.
O. Lindqvist, *Acta Chem. Scand.*, 26 (1972) 1423.

Orthorhombic trishydroxyacetates of lanthanum(III) and gadolinium(III)

The compounds are isostructural with space group ($Pn2_1a$); $R=4.3\%$ and 3.5% respectively. A comparison of the corresponding interatomic bond distances indicates that the average metal—oxygen distances decrease approximately as the decrease in the ionic radius of the central ion. The latter also affects oxygen—oxygen contacts in the coordination polyhedron and the conformation of the ligands.
I. Grenthe, *Acta Chem. Scand.*, 26 (1972) 1479.

Di-μ-bromo-μ-1,2-cyclohexylenetetrabromoditellurium, $Te_2Br_6C_6H_{10}$

($Pnma$) $Z=4$, $R=6.1\%$ for 574 significant reflections. The tellurium atoms are surrounded by four bromine atoms in a plane and by one carbon atom at the apex of the pyramid. The two tellurium atoms are linked by two bromine atoms and the cyclohexane group. Te–Br(mean bridging) = 2.880(6), Te–Br(terminal) = 2.542(6) Å.

A.C. Hazell, *Acta Chem. Scand.*, 26 (1972) 1510.

Pentathiazyl tetrachloroaluminate, $S_5N_5AlCl_4$

(*Pnma*) Z=8, R=8.7% for 1523 reflections. The structure consists of $S_5N_5^+$ and $AlCl_4^-$ ions. The $S_5N_5^+$ cation is heart-shaped and almost planar. S–N = 1.465–1.590 Å; Al–Cl(mean) = 2.117 Å.

A.C. Hazell and R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1987.

Mercury(II) chromate hemihydrate, $HgCrO_4 \cdot \frac{1}{4}H_2O$

(*C*2/*c*) Z=8, R=10.1% for 1679 independent reflections. Each mercury is bonded to two oxygen atoms forming chromate tetrahedra, bond lengths 2.05(2) and 2.06(2) Å. ∠O–Hg–O is 176.2(9)°. The chromate tetrahedra are slightly distorted.

K. Aurivillius, *Acta Chem. Scand.*, 26 (1972) 2113.

Cadmium(II) hexacyanochromate(III), $Cd_3[Cr(CN)_6]_2 \cdot xH_2O$

(*F*432), (*F*$\bar{4}$3*m*) or (*Fm*3*m*) Z=1.3, R=3.5% for 615 reflections. The structure is closely related to the structures of other Prussian Blue analogue compounds. One third of all $Cr(CN)_6$ groups are statistically absent so that each cadmium atom has a mixed coordination. All cyanide groups are bridged between chromium and cadmium atoms with carbon atoms bonded to chromium. Cr–C = 2.05(2), C–N = 1.14(3), Cd–N = 2.27(2) and Cd–O = 2.39(4) Å.

H.U. Güdel, *Acta Chem. Scand.*, 26 (1972) 2169.

Cobalt(II) (–)malate trihydrate, $Co(OOC\text{-}CH_2\text{-}CHOH\text{-}COO) \cdot 3H_2O$

(*P*2_1) Z=2, R=2.8% for 1101 independent reflections. The cobalt atom has six oxygens surrounding it in an octahedral array, two belonging to water molecules and the rest to malate groups. Co–O = 2.07–2.09 Å.

L. Kryge and S.E. Rasmussen, *Acta Chem. Scand.*, 26 (1972) 2349.

A tetracarbonyl(carbene)iron complex, $C_9H_8N_2O_4Fe$

(*C*2/*c*) Z = 8, R = 4.4%. The carbene ligand substitutes an axial carbonyl group of the parent compound $Fe(CO)_5$. Some of the bond angles within the coordination polyhedron deviate largely from ideal trigonal-bipyramidal values.

G. Huttner and W. Gartzke, *Chem. Ber.*, 105 (1972) 2714.

Tricarbonyl (hexaethylborazine)chromium(0)

($P2_1/c$) $Z=4$, $R=7.6\%$. The borazine ring assumes a not very pronounced chair conformation. It is bonded centrically to the $Cr(CO)_3$ moiety. The nitrogen atoms are staggered with respect to the CO groups and are thus *trans* to the carbonyl groups.

G. Huttner and B. Krieg, *Chem. Ber.*, 105 (1972) 3437.

Bis(1-methoxyborinato)cobalt (A) and bis(1-methylborinato) cobalt (B)

($P2_1/c$) $Z=2$, $R_1 = 4.4\%$ (A) and ($P2_1/c$) $Z=2$, $R=3.8\%$ (B). Both molecules are centrosymmetric. The newly planar rings are parallel to each other and form sandwich-type complexes with the metal atom. Within the limits of error, the distances are identical for the two ring systems.

G. Huttner, B. Krieg and W. Gartzke, *Chem. Ber.*, 105 (1972) 3424.

trans-Chloro(methyl)bis(trimethylarsine)-platinum(II) hexafluorobut-2-yne

($P2_1/c$) $Z=4$, $R=7.7\%$ for 1156 reflections. The coordination of the platinum atom is trigonal-bipyramidal, with the acetylene considered a monodentate ligand, and the two trimethylarsine ligands occupying the equatorial plane. The chlorine and methyl ligands occupy apical positions. Pt—Cl = 2.47(1); Pt—C = 2.10(4) Å.

B.W. Davies, R.J. Puddephatt and N.C. Payne, *Can. J. Chem.*, 50 (1972) 2276.

1-h^1 : 1:2-h^2-*trans*-2-Bromovinyl-μ-bromo-bis(tricarbonyl iron) (*Fe-Fe*), $Fe_2Br_2C_8O_6H_2$

(Cc) $Z=4$, $R=6.07\%$ for 1970 reflections. The molecular framework consists of two distorted octahedral $Fe(CO)_3$ moieties linked together by a slightly asymmetric bromine bridge, an Fe—Fe bridge (2.525 Å) and a π-bonded bromovinyl group. The bonding of the organic ligand suggests the formation of a 1-ferra—π-allyl system. Within the irontricarbonyl fragments, strong *trans* effects are observed.

C. Krüger and Y.H. Tsay, *Isr. J. Chem.*, 10 (1972) 201.

$(-)_{589}$-Dinitrobis(ethylenediamine)cobalt(III)$(+)_{589}$-bis(malonato)ethylenediamine-cobaltate(III), $(-)_{589}$-$[Co(NO_2)_2(en)_2]$ $(+)_{589}$-$[Co(mal)_2en]$

($P1$) $Z=1$, $R=10.1\%$ for 1371 observed reflections. The Δ configuration has been assigned to the complex cation and the anion. The two Co—mal chelate rings are nearly planar. ∠O—Co—O in this ring is 96°, which is slightly larger than ∠N—Co—N in the Co^{III}—trimethylenediamine ring. All en ligands have *ob*-conformation.

K. Matsumoto and H. Kuroya, *Bull. Chem. Soc. Jap.*, 45 (1972) 1755.

Bis(hydrogen-pyridine-2,6-dicarboxylato)nickel(II) trihydrate, $Ni(C_7H_4NO_4)_2 \cdot 3H_2O$
($P2_1/c$) Z=4, 2138 intensities. The nickel is octahedrally coordinated and the structure is held together by hydrogen bonding.
A.C. Villa, C. Guastini, A. Masatti and M. Nardelli, *Gazz. Chem. Ital.*, 102 (1972) 226.

Cis (A) and *trans* (B) (π-cyclopentadienyl)-[π-bis(trimethylsilyl)diphenylcyclobutadiene]-cobalt
($Pbca$) R=9.2% for 3697 reflections (A) and ($P2_1/c$) R=7.3% for 4773 reflections (B). Both five- and four-membered rings are π-bonded to the metal and in both the molecular skeleton is of similar geometry. Thus the cobalt atom is sandwiched between the cyclobutadiene and cyclopentadiene groups which are both planar and parallel to each other (within 0.8°).
C. Kabuto, J. Hayashi, H. Sakurai and Y. Kitahara, *J. Organometal. Chem.*, 43 (1972) C23.

Bis-(N-n-septylsalicylaldiminato)nickel(II), $C_{28}H_{40}N_2O_2Ni$
($P2_1/c$) Z=2, R=10.5% for 776 reflections. The nickel atoms have *trans* planar coordination. Some distortion is present in the molecule. The conformation of the chain starting from the nitrogen atom may be represented as *trans–gauche–trans–trans–trans.*
R. Graziani and E. Forsellini, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 775.

Tris(dithioacetylacetonato)rhodium(III)
($C2/c$) Z=8, R=7.1% for 3117 reflections. Each rhodium is surrounded by six sulphur atoms at distances ranging from 2.134(3) to 2.333(3) Å, in a distorted octahedral array. The ligand dimensions are normal but chelate rings are not exactly planar as the rhodium atom is out of the plane.
R. Beckett and B.F. Hoskins, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 683.

Bis(O-ethylthioacetothioacetato)zinc(II), $[CH_3C(S)CHC(S)OEt]_2Zn$
($C2/c$) Z=4, R=11%. The complex contains a tetrahedral ZnS_4 core, and the ligands are essentially planar. Bond lengths suggest extensive delocalisation of the double bonds. Zn–S = 2.333(1) and 2.270(1) Å.
R. Beckett and B.F. Hoskins, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 679.

trans-Dinitrobis(triethylphosphine)platinum(II)
($Pca2_1$) or ($Pbcm$) Z=4, R=9.9% for 1100 independent reflections. The compound is

trans square-planar and quasi-centrosymmetric. The two NO_2^- groups are coordinated through nitrogen atoms and lie in a plane which is almost perpendicular to the equatorial plane. Pt–P = 2.41 Å.

R. Graziani, G. Bomberi and E. Forsellini, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 701.

Trichlorotripyridine titanium(III)-1-pyridine

($C222_1$) Z=4, R=7.4% for 567 independent reflections. The metal coordination sphere is octahedral and angles subtended at the metal atom are all within 2.6° of 90°. Ligand–ligand repulsions and pair repulsions dominate over *trans* effects within the molecule.

R.K. Collins and M.G.B. Drew, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 975.

[*N*,*N*′-Ethylene-bis(salicylideneiminato)UO_2(MeOH)]

($P2_1/n$) Z=4, R=5.5% for 2239 independent reflections. The coordination sphere is that of a slightly irregular pentagon and the uranyl group is linear within experimental error. U–O(equatorial) = 2.235 and 2.330; (apical) = 1.764 and 1.771.

G. Bandoli, D.A. Clemente, U. Croatto, M. Vidali and P.A. Vigato, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 961.

Tetraethylammonium tris(*o*-ethylxanthato) cadmiumate(II), $[(CH_3)_4N][Cd(S_2COC_2H_5)_3]$

($P2_1/c$) Z=4, R=7.5% for 2612 independent observable reflections. The anion is five-coordinate with bidentate *o*-ethylxanthates and one bonded to cadmium via only one of its sulphur atoms. The five sulphur atoms form a tetragonal pyramid. Cadmium is 1.079(1) Å above the basal plane.

B.F. Hoskins and B.P. Kelly, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 875.

Tris(tropolonato)aluminium(III)

($C2/c$) Z=4, R=4% for 1510 reflections. The complex has C_2 symmetry but departure from D_3 is slight. The tropolinate ligands are bent slightly in the vicinity of the aluminum atoms with resultant angles (AlO_2-C_7H_5 dihedral) of 6.24° for the ligand on the C_2 axis and 3.73° for the other unique ligand.

E.L. Muetterties and L.J. Guggenberger, *J. Amer. Chem. Soc.*, 94 (1972) 8046.

$[Co(dpe)_2X]SnX_3$ (A) and $[Co(dpe)_2Cl]SnX$(B), where dpe is $(C_6H_5)_2PCH_2CH_2P$-$(C_6H_5)_2$ and X = Cl

($P2_1/c$) Z=4, R=6.5% for 5706 reflections (A) and ($P\bar{1}$) Z=2, R = 10.1% for 3484

reflections. Both structures have discrete [Co(dpe)$_2$Cl] cations and pyramidal $SnCl_3^-$ anions in the unit cell. (A) is a square pyramid with apical Cl whereas (B) is trigonal-bipyramidal with two P atoms in the axial positions and two P atoms and one Cl equatorially.
J.K. Stalick, P.W.R. Corfield and D.W. Meek, *J. Amer. Chem. Soc.*, 94 (1972) 6194.

Di-μ-hydrido-octacarbonyldirhenium, $H_2Re_2(CO)_8$
($P2_1/c$) Z=4, R=10% for 1163 significant reflections. The structure has D_{2h} symmetry. Re–Re is 2.90 Å. The two hydrogen atoms would appear to be best placed in the plane defined by the rhenium atoms and 4 bis-dihedral carbonyl groups.
M.J. Bennett, W.A.G. Graham, J.K. Hoyano and W.L. Hutcheon, *J. Amer. Chem. Soc.*, 94 (1972) 6232.

Monothiodibenzoylmethanato-π-*syn*-1-*tert*.-butyl-2-methallyl-palladium(II), 1,2,3-h^3-[(t-C_4H_9)CHC(CH_3)CH_2]Pd($C_6H_5COCHCSC_6H_5$)
($P\bar{1}$) Z=2, R=7.3% for 2363 independent reflections. The *tert*.-butyl substituent *syn* to the methyl group in the π-allyl ligand is *trans* to the sulphur atom of the chelating SDBM ligand (monothiodibenzoylmethane). Pd–S = 2.297(3), Pd–O = 2.047 Å; ∠S–Pd–O = 94.4(2)°.
S.J. Lippard and S.M. Morehouse, *J. Amer. Chem. Soc.*, 94 (1972) 6956.

Bis(trithioperoxycumato)zinc(II) (A) and dithiocumato(trithioperoxycumato)nickel(II) (B)
($P\bar{1}$) Z=2, R_1 = 6.1% for 2776 unique reflections (A) and ($C2/c$) Z=4, R_1 = 6.5% for 1754 unique reflections (B). Five-membered disulphide (S–S bonds ~ 2.01 Å) containing chelate rings are found with planar NiS_4 and tetrahedral ZnS_4 geometries.
J.P. Fackler, Jr., J.A. Fetchin and D.C. Fries, *J. Amer. Chem. Soc.*, 94 (1972) 7323.

α, β, γ, δ-Tetraphenylporphinatodichlorotin(IV), Cl_2SnTPP
($I4/m$) Z=2, R=5.2% for 4450 independent reflections. The octahedral coordination group of the Cl_2SnTPP molecule has Sn–Cl = 2.420(1) and Sn–N = 2.098(2).
D.M. Collins, W.R. Scheidt and J.L. Hoard, *J. Amer. Chem. Soc.*, 94 (1972) 6689.

Zinc sulphite dihydrate, $ZnSO_3 \cdot 2H_2O$
($P\bar{1}$) Z=4, R=12.6% for 2076 independent reflections. The structure consists of ZnO_4 tetrahedra and ZnO_6 octahedra linked together with pyramidal sulphite groups.
H. Quinones and S. Baggio, *J. Inorg. Nucl. Chem.*, 34 (1972) 2153.

$Ca(NO_2)_2 \cdot CO(NH_2)_2 \cdot 3H_2O$
($P2_1/c$) $Z=4$, $R=12.8\%$ for 978 reflections. The calcium atom coordinates eight oxygens, Ca–O = 2.35–2.68 Å. Urea is coordinated via oxygen to calcium.
L. Lebioda, *Rocz. Chem.*, 46 (1972) 373.

Benzene 1,2-dithiolatodi(π-cyclopentadienyl)titanium(IV), $(C_5H_5)_2TiS_2C_6H_4$
($P2_1/c$) $Z=8$. The $S_2C_6H_4$ plane of the molecule is folded out of the TiS_2 plane by an angle of 46°. The coordination around titanium defined by the centroids of the cp ring and the sulphur atoms is a distorted tetrahedron of nearly $mm2$–C_{2v} symmetry.
A. Kutoglu, *Z. Anorg. Allg. Chem.*, 390 (1972) 195.

Dicarbonyldiacetylbis(dimethylhydrazone) nickel(0)
($Pbca$) $Z=8$, $R=11.7\%$. Each Ni atom is tetrahedrally coordinated to two carbonyl groups and the bidentate ligand, which forms an almost planar five-membered ring with the central atom.
H.D. Hausen and K. Krogmann, *Z. Anorg. Allg. Chem.*, 389 (1972) 247.

Tris(ethylenediamine)cobalt(III) hexacyanoferrate(III) dihydrate, $Co(C_2H_8N_2)_3 \cdot [Fe(CN)_6] \cdot 2H_2O$
($P2_1/n$) $Z=4$. The structure contains discrete $Co(en)_3^{3+}$ and $Fe(CN)_6^{3-}$ octahedral ions. Fe–C(mean) = 1.91 and Co–C(mean) = 2.01 Å respectively. Hydrogen bonding is very weak in the compound and does not play an important structural role.
L.D.C. Box, J.G. Leipoldt and S.S. Basson, *Z. Anorg. Allg. Chem.*, 289 (1972) 307.

$SbCl_2F_3$
($P2_1/n$) $Z=12$. The structure consists of tetrahedral $SbCl_4^+$ and $(F_4SbCl)_2F^-$ ions. In the latter, octahedral Sb are linked by a fluorine bridge.
H. Preiss, *Z. Anorg. Allg. Chem.*, 389 (1972) 254.

Hexaphenyldiplumbane
($P2_1/c$) $Z=4$, 1800 reflections. Pb–Pb = 2.83(1) Å.
H. Preut, H.J. Haupt and F. Huber, *Z. Anorg. Allg. Chem.*, 388 (1972) 165.

Potassium trithiotungstate chloride, $K_3(WOS_3)Cl$
B. Krebs, B. Buss and A. Ferwanah, *Z. Anorg. Allg. Chem.*, 387 (1972) 142.