Coordination Chemistry Reviews, 13 (1974) 83–99

X-RAY BIBLIOGRAPHY

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$MAl(NH_2)_4$ (M = Na, K, Cs)

All the structures are built up of $[Al(NH_2)_4]^-$ tetrahedra and M^+ ions. The tetraamidogallate $NaGa(NH_2)_4$ is isotopic with $NaAl(NH_2)_4$. Refinement of hydrogen positions was carried out.

P. Molinie, R. Brec, J. Rouxel and P. Herpin, *Acta Crystallogr., Sect. B,* 29 (1973) 925.

Tellurium(IV,VI) oxyhydroxide, $H_2Te_2O_6$

(Pbn2) Z = 4, R = 3.6% for 2480 independent reflections. The structure contains $Te^{VI}O_6$ octahedra and four-coordinated Te^{IV} units with Te^{VI}–O, 1.864–1.952 Å and Te^{IV}–O, 1.861–2.107 Å. The octahedra are linked through corners to form chains.

O. Lindqvist and J. Moret, *Acta Crystallogr., Sect. B,* 29 (1973) 956.

$CuTe_2O_5$

($P2_1/c$), $Z = 4$, $R = 4.8\%$ for 3556 independent reflections. Copper and tellurium coordination polyhedra share oxygen atoms. Each oxygen atom interacts with three metal atoms, two of the interactions being strong and one weak. The Cu–O polyhedron may be described as a distorted octahedron with four strong bonds (Cu–O, 1.950–1.969 Å) and two weaker bonds (Cu–O, 2.305–2.780 Å). Both independent tellurium atoms have three strong pyramidal bonds to oxygen (Te–O, 1.859–2.019 Å) and one of them has a fourth interaction with an oxygen atom at 2.402 Å while the other has two weaker oxygen interactions at 2.607 and 2.733 Å.

K. Hanke, V. Kupčik and O. Lindqvist, *Acta Crystallogr., Sect. B,* 29 (1973) 963.

Aquo(ethylenediaminetriacetatoacetic acid)rhodium(III), $Rh(H_2O)HEDTA$

($P2_1/c$) $Z = 4$, $R = 2.5\%$ for 2183 reflections. The complex is a distorted octahedral molecule with H_2O bonded to Rh in an equatorial site and a pentadentate HEDTA bearing

a proton on its uncoordinated glycinate arm. Rh–N, 2.082 and 1.988 Å; Rh–O, 2.096, 2.030, 2.001 and 2.027 Å.
G.H.Y. Lin, J.D. Leggett and R.M. Wing, *Acta Crystallogr., Sect. B,* 29 (1973) 1023

Potassium boromalate ($KBC_8H_8O_{10}.H_2O$)
($P2_1$) $Z = 2, R = 5.6\%$ for 1638 independent reflections. The potassium and the boron atoms are coordinated to oxygen atoms of the carboxyl and hydroxyl groups. The boron atom is tetrahedrally coordinated and the potassium atom exhibits five-coordination.
R.A. Mariezcurrena and S.E. Rasmussen, *Acta Crystallogr., Sect. B,* 29 (1973) 1035.

o-Aminothiophenol-bis(tricarbonyliron), $(C_6H_4SNH)Fe_2(CO)_6$
($P2_1/c$) $Z = 4, R = 3.5\%$ for 1058 independent reflections. The molecule contains two iron tricarbonyl groups bridged through the sulphur and the nitrogen atoms of the organic ligand, with an iron–iron bond of 2.411(1) Å. The mean Fe–S is 2.283(2) and Fe–N is 2.002(4) Å.
G. Le Borgne and D. Grandjean, *Acta Crystallogr., Sect. B,* 29 (1973) 1040.

Thallium(I) cryptate, $C_{18}H_{36}N_2O_6.TlHCOO.H_2O$
($C2/c$) $Z = 4, R = 5.2\%$ for 2027 reflections. The thallium–nitrogen bonds present a slightly covalent character; the observed distances are equal to 2.946(6) Å. This may explain the higher stability of the thallium cryptate compared with the potassium cryptate. The inert lone pair of the thallium ion has probably been located.
D. Moras and R. Weiss, *Acta Crystallogr., Sect. B,* 29 (1973) 1059.

$La_{10}S_{15-x}O_x$
The structure contains a void cavity with $x = 0.5$ ($R = 13\%$). It is shown that sulphur replaces oxygen in statistical disorder, the cavity remaining unoccupied.
P. Besançon, D. Corré and P. Laruelle, *Acta Crystallogr., Sect. B,* 29 (1973) 1064.

$Ca(IO_3)_2.6H_2O$
($Fdd2$) $Z = 8, R = 3.2\%$ for 1544 intensities. The calcium ions are coordinated to the six water oxygens and to two iodate oxygens in a distorted Archimedean antiprism with edges 2.85–3.15 Å. The trigonal pyramidal iodate ions (I–O, 1.81(2) Å, ∠O–I–O, 98.2(7)°) are held in the lattice by a network of hydrogen bonds and arranged to give a highly distorted octahedral environment about the iodine.
B. Morosin, J.G. Bergman and G.R. Crane, *Acta Crystallogr., Sect. B,* 29 (1973) 1067.

Uranyl chloride, UO_2Cl_2, by powder neutron diffraction

The present analysis has revealed errors of 0.56 and 0.17 Å in earlier proposed oxygen positions and of 0.16 Å in the chlorine position. The interatomic distances and angles from the neutron diffraction analysis are normal.

J.C. Taylor and P.W. Wilson, *Acta Crystallogr., Sect. B*, 29 (1973) 1073.

$K_xRb_{1-x}HSO_4$

($P2_1/c$) $Z = 8$, $R = 12\%$ for 712 reflections. As a structure type, it exists for $0.30 < x < 0.55$. For $x = 0.55$ all the Rb^+ present is ordered into one of the two sites available to the alkali metals.

$RbHSO_4$

($P2_1/c$) $Z = 8$, $R = 10.6\%$ for 800 reflections. The structure is made up of discrete SO_4^{2-} tetrahedra and Rb^+ ions which are coordinated by a shell of oxygen atoms. One Rb is nine-coordinate with bonds 2.96–3.27 Å, and the other is ten-coordinate with bonds 2.91–3.15 Å.

W.G. Mumme, *Acta Crystallogr., Sect. B*, 29 (1973) 1076.

Bis(*N*-benzyldiphenylphosphinic amide)dichlorocobalt(II), $[(C_6H_5)_2PONHCH_2C_6H_5]_2CoCl_2$

(Cc) or ($C2/c$) $Z = 4$, $R = 11.9\%$ for 1669 reflections. The molecule contains a cobalt atom which is bonded in almost regular tetrahedral directions to two chlorine atoms and two oxygen atoms, one each from the *N*-benzyldiphenylphosphinic amide ligand.

R.M. Roy and J.W. Jeffery, *Acta Crystallogr., Sect. B*, 29 (1973) 1083.

Lead thiocyanate complex with tetraoxa-1,7,10,16-diaza-4,13-cyclooctadecane, $Pb(SCN)_2 \cdot C_{12}H_{26}N_2O_4$

($C2/c$) $Z = 4$, $R = 4.7\%$ for 2180 reflections. The lead ion lies in the plane of the four oxygen atoms, the two nitrogen atoms being on both sides. The coordination sphere of lead is completed by the sulphur atoms of the thiocyanate anions, one above and one below the ring.

B. Metz and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 1088.

A dimethyl sulphoxide complex with lanthanum nitrate, $La(NO_3)_3 . 4(CH_3)_2SO$

($C2/c$) $Z = 4$, $R = 9.4\%$ for 1257 reflections. The La^{3+} ion is coordinated by ten oxygen atoms with La–O distances 2.47–2.71 Å. Six oxygens are from the three nitrate groups, which are bidentate, and four are from the four DMSO groups.

K.K. Bhandary and H. Manohar, *Acta Crystallogr., Sect. B*, 29 (1973) 1093.

Thiocyanatopenta-ammineiridium(III) diperchlorate, $[Ir(SCN)(NH_3)_5](ClO_4)_2$
($P2_1/c$) $Z = 4$, $R = 5.8\%$ for 2128 reflections. The structure consists of octahedrally coordinated monomeric Ir units $[Ir(SCN)(NH_3)_5]^{2+}$ and tetrahedral $(ClO_4)^-$ ions. The SCN group is not quite linear, with ∠ S—C—N, 174.20° and ∠ Ir—S—C, 109.0°.
H.D. Flack and E. Parthé, *Acta Crystallogr., Sect. B,* 29 (1973) 1099.

Cobalt(pyrazine-2,3-dicarboxylate) · dihydrate, Co-2,3-PYD · $2H_2O$, where PYD is $C_6H_2N_2O_4$
($Pcca$) $Z = 8$, $R = 3.4\%$ for 1542 reflections. The structure consists of chains of molecules parallel to the [010] direction; the molecules are linked by hydrogen bonds.
P. Richard, D. Tran Qui and E.F. Bertaut, *Acta Crystallogr., Sect. B,* 29 (1973) 1111.

Magnesium hypophosphite hexahydrate, $Mg(PO_2H_2)_2 \cdot 6H_2O$
($I4_1/acd$) $Z = 8$, $R = 5.8\%$ for 465 independent reflections. The $[Mg(OH_2)_6]^{2+}$ cations have 222 symmetry and the $PO_2H_2^-$ anion has symmetry 2. P—O, 1.507(3) Å; ∠ O—P—O is 116.2(3)°. Mg—O, 2.044(3) and 2.066(5) Å.
J.L. Galigné and Y. Dumas, *Acta Crystallogr., Sect. B,* 29 (1973) 1115.

α-Oxo-γ-phenyl-1,1′-trimethylene-2′-methylferrocene ($T = 228$°C)
($Aba2$) $Z = 8$, $R = 6.1\%$ for 658 reflections. The dihedral angle between the best planes through the two nearly planar cyclopentadienyl rings is about 10°. The rings are nearly in the totally eclipsed conformation. The relative conformation of the molecule is *RpSc* or *SpRc* and is in agreement with that proposed from NMR data.
C. LeComte, Y. Dusausoy, J. Protas and C. Moise, *Acta Crystallogr., Sect. B,* 29 (1973) 1127.

Bis(6-aminohexanato)copper(II) dihydrate
($P2_1/n$) $Z = 4$, $R = 6.6\%$ for 993 independent reflections. Four aminohexanoate molecules are coordinated to a central copper atom, two of them via a carboxylate oxygen atom and the other two via a nitrogen atom. Cu—O, 1.967(5); Cu—N, 1.994(6) Å. These centrosymmetric ligand atoms form an almost square plane, and they are *trans* with respect to each other. The second oxygen atoms of the two carboxylate groups are bound, one above and one below the square plane, completing a distorted octahedron. Cu—O, 2.768(6) Å. Each ligand molecule binds to two copper atoms.
B. Sjöberg, R. Österberg and R. Söderquist, *Acta Crystallogr., Sect. B,* 29 (1973) 1136.

Tris(2,4-pentanedionato)rhodium(III)
($P2_1/c$) $Z = 4$, $R = 3.5\%$ for 2881 reflections. The compound is monomeric with central

rhodium octahedrally coordinated to six oxygen atoms.

J.C. Morrow and E.B. Parker, Jr., *Acta Crystallogr., Sect. B*, 29 (1973) 1145.

Trichloro-(π-*trans*-pent-2-enylammonium)platinum(II), $PtCl_3(C_5H_{12}N)$

($P2_1/a$) $Z = 4$, $R = 6.0\%$ for 1560 independent reflections. The platinum atom is four coordinate square-planar from three chlorine atoms and the olefinic double bond of the pentenylammonium cation. The midpoint of the double bond is 0.42 Å out of the square plane and the double bond makes an angle of 76.8° with it.

R. Spagna, G. Ughetto and L. Zambonelli, *Acta Crystallogr., Sect. B*, 29 (1973) 1151.

Refinement of potassium isocitrate lactone

($P2_12_12_1$) $Z = 4$, $R = 4.6\%$ for 322 intensities. All hydrogen atoms are located. The carbonyl group adjacent to the ring oxygen atom is the one that is predominantly ionized.

H.M. Berman, H.L. Carrell and J.P. Glusker, *Acta Crystallogr., Sect. B*, 29 (1973) 1163.

Redetermination of potassium sulphate and potassium chromate

K_2SO_4: ($Pnam$) $Z = 4$, $R = 5.1\%$ for 864 reflections. K_2CrO_4 is isostructural. $R = 3.3\%$. The shortest contacts between potassium and oxygen are in the range 2.68–2.79 Å and amongst the shortest contacts there is one potassium atom approximately collinear with each of the bond axes within the anion. The three independent Cr–O lengths are 1.636(3), 1.651(2) and 1.643(3) Å.

J.A. McGinnety, *Acta Crystallogr., Sect. B*, 28 (1972) 2845.

Sodium rubidium dichromate(A) and sodium caesium dichromate(B)

($P2_1/c$) $Z = 8$, $R = 4.6\%$ for 1406 reflections(A); (B) is isotypic. The structure contains blocks of composition $(RbCr_2O_7)_4$ which are similar to those found in $Rb_2Cr_2O_7$ structures.

N.Ch. Panagiotopoulos and I.D. Brown, *Acta Crystallogr., Sect. B*, 28 (1972) 2880.

Cobalt diphosphate, $Co_2P_2O_7$

($B2_1/c$) $Z = 8$, $R = 5.6\%$ for 1123 reflections. The anion showed significant deviations from C_2 symmetry caused by six strong Co–O bonds on one side and only five on the other.

N. Krishnamachari and C. Calvo, *Acta Crystallogr., Sect. B*, 28 (1972) 2883.

Tetraphenylgermanium

($P\bar{4}2_1c$) $Z = 2$, $R = 2.6\%$ for 546 reflections. The germanium atoms lie on $\bar{4}$ axes and there is only one independent phenyl ring in the asymmetric unit. The plane of the phenyl ring is rotated 54.1° (measured clockwise) from the C–Ge–C plane.

A. Karipides and D.A. Haller, *Acta Crystallogr., Sect. B*, 28 (1972) 2889.

LaF_3, CeF_3, PrF_3 and NdF_3

The difference in the screening constants for various fluorine positions was used to determine the symmetry and basic structures of these compounds.

M.L. Afanasiev, S.B. Habuda and A.G. Lundin, *Acta Crystallogr., Sect. B*, 28 (1972) 2903.

Silver diethyl phosphate, $AgPO_2(OC_2H_5)_2$

($Pccn$) $Z = 2$, $R = 4.3\%$ for 853 reflections. The silver ion is coordinated to four oxygen atoms in a distorted tetrahedral arrangement with a mean Ag–O of 2.26 Å. The ester conformation with respect to the phosphate group is *syn*-clinal, *anti*-clinal.

J.P. Hazel and R.L. Collin, *Acta Crystallogr., Sect. B*, 28 (1972) 2951.

Tin(II) iodide, SnI_2

($C2/m$) $Z = 6$, $R = 4.9\%$ for 234 independent reflections. The compound is shown to possess a unique AX_2 layer structure in which the metal atoms occur in two distinct sites. Two-thirds of the tin atoms occupy sites similar to those in $SnCl_2$ ($PbCl_2$ type). The remaining tin atoms are in $PdCl_2$-type chains.

R.A. Howie, W. Moser and I.C. Trevena, *Acta Crystallogr., Sect. B*, 28 (1972) 2965.

The anisotropic thermal expansion of β-uranyl dioxide

The positional parameters for all the atoms in β-$UO_2(OH)_2$ at six temperatures from 21 to 260°C were obtained. The results confirm that the strongly anisotropic thermal expansion of this material is caused by the rotation of oxygen octahedra in the structure.

J.C. Taylor and M.J. Bannister, *Acta Crystallogr., Sect. B*, 28 (1972) 2995.

Hydrogen di(bis-π-cyclopentadienyl)-L-cysteinatomolybdenum(IV) chloride (A); hydrogen di(bis-π-cyclopentadienyl)-L-cysteinatomolybdenum(IV) hexafluorophosphate (B); bis-π-cyclopentadienylglycinatomolybdenum(IV) chloride monohydrate (C); and bis-π-cyclopentadienylsarcosinatomolybdenum(IV) chloride methanolalate

($P2_12_12_1$) $Z = 2$, $R = 3.8\%$ for 1785 reflections (A); (P_1) $Z = 1$, $R = 11.9\%$ for 2205 reflections (B); ($P2_1/b$) $Z = 4$, $R = 10.9\%$ for 1819 reflections (C); ($P2_12_12_1$) $Z = 4$, $R = 7.2\%$ for 1140 reflections (D). In the bis-π-cyclopentadienyl molybdenum groups, the rings are staggered in the sarcosine and cysteine chloride complexes but eclipsed in the glycine and cysteine hexafluorophosphate. The mean length of the normals to the rings from molybdenum is 1.98 Å and the mean angle between them is 133°. The cysteine chelates through nitrogen and sulphur; Mo–N, 2.256; Mo–S, 2.446 Å in the chloride. The carboxylate groups are hydrogen bonded to form a typical carboxylate dimer. The cysteine has different conformations in the PF_6^- and Cl^- complexes. In the glycine and sarcosine complexes, the molybdenum is chelated through oxygen and nitrogen. Mo–N, 2.24 (mean) and Mo–O, 2.10 Å (mean).

C.K. Prout, G.B. Allison, L.T.J. Delbaere and E. Gore, *Acta Crystallogr., Sect. B,* 28 (1972) 3043.

$CaKAsO_4 . 8H_2O$

($Cm2m$) $Z = 2, R = 4.3\%$ for 1023 reflections. Ca coordinates to eight water oxygen atoms with Ca · · · O, 2.460(5)–2.490(3) Å. K coordinates to eight water oxygen atoms with K · · · O, 2.756(3)–2.960(7) Å. The coordination polyhedra of Ca share one face of four water molecules and two edges with neighbouring coordination polyhedra of K.

B. Dickens and W.E. Brown, *Acta Crystallogr., Sect. B,* 28 (1972) 3056.

Potassium diborate, $K_2O.2B_2O_3$

($P\bar{1}$) $Z = 4, R = 3.2\%$ for 2063 intensities. The structure contains a single three-dimensional borate anion framework, composed of mutually linked planar BO_3 triangles, diborate and triborate groups in which two of the three borate atoms are tetrahedrally coordinated. B–O is 1.477 Å and 1.369 Å for boron in four-fold and three-fold coordination respectively.

J. Krogh-Moe, *Acta Crystallogr., Sect. B,* 28 (1972) 3089.

$ErCd_3$ and its isomorphous compounds

($Cmcm$) $Z = 4, R = 10.5\%$ for 217 independent reflections. The packing is closely related to the arrangement in $GdCd_3$, with the hexagonal Ni_3Sn structure type.

M.L. Fornasini and F. Merlo, *Acta Crystallogr., Sect. B,* 28 (1972) 3094.

$Bi_2In_4S_9$, bismuth indium sulphide

($P2_1/m$) $Z = 2, R = 5.1\%$ for 2052 independent reflections. The structure consists of a sulphur framework forming closest packing only in a prismatic domain parallel to the *b* axis. Three indium atoms are octahedrally surrounded by sulphur atoms; the coordination of the fourth is a distorted tetrahedron.

G. Chapius, Ch. Gnehm and V. Krämer, *Acta Crystallogr., Sect. B,* 28 (1972) 3128.

β-$SnWO_4$

($P2_13$) $Z = 4, R = 3.4\%$ for 248 observed reflections. The tungsten atom is located in a fairly regular oxygen tetrahedron. Sn^{2+} is coordinated to six oxygen atoms forming three short (2.21 Å) and three long (2.81 Å) bonds with a configuration characteristic of a "lone pair" cation.

W. Jeitschko and A.W. Sleight, *Acta Crystallogr., Sect. B,* 28 (1972) 3174.

Uranyl oxalate trihydrate, $UO_2C_2O_4 . 3H_2O$

($P2_1/c$) $Z = 4, R = 7.9\%$ for 513 reflections. Each uranium atom exists as a linear $(O_1-U-O_1)^{2+}$ ion with five secondary oxygen atoms coordinated to it in a perpendicular plane. $U-O_1$, 1.63(4) and $U-O_2$, 2.49(4) Å. The oxalate groups are planar and tetradentate, each bridging two uranyl ions. Only one water molecule is coordinated to the uranium atom.

N.C. Jayadevan and D.M. Chackraburtty, *Acta Crystallogr., Sect. B,* 28 (1972) 3178.

Tricarbonyl(*N*-cinnamylideneaniline)iron, $C_6H_5CH{=}CH{-}CH{=}N{-}C_6H_5 . Fe(CO)_3$

($P2_1/c$) $Z = 4, R = 5.4\%$ and ($pbca$) $Z = 8, R = 3.6\%$. The two crystal forms contain experimentally indistinguishable molecular units of tricarbonyl(*N*-cinnamylideneaniline)-iron. Although the nitrogen lone pair does not play an important part in the bonding with the metal, the heterobutadienic fragment –CH=CH–CH=N– is not conserved by coordination with the $Fe(CO)_3$ group.

A. De Cian and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 3264.

Tricarbonyliron(cinnamaldehyde)iron, $C_6H_5CH{=}CH{-}CHO . Fe(CO)_3$

($P2_1/c$) $Z = 4, R = 4.8\%$ for 1119 independent reflections. The compound must be considered as a π complex. The two oxygen lone pairs do not play a part in the bonding with the metal and the structure is similar to that found in tricarbonyl(*N*-cinnamylideneaniline)iron.

A. De Cian and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 3273.

Dipyridine adduct of tris(2,2,6,6-tetramethylheptane-3,5-dionato)europium(III), $Eu(dpm)_3(py)_2$

($P\bar{1}$) $Z = 2, R = 3.9\%$ for 6840 observed reflections. The molecule contains a near two-fold axis and the coordination sphere is best described as a square antiprism. Eu^{III} lies 0.3–0.4 Å out of the molecular planes of four of the five ligands. Most hydrogen atoms were located.

R.E. Cramer and K. Seff, *Acta Crystallogr., Sect. B,* 28 (1972) 3281.

Thallium oxyfluoride, TlOF
($I\bar{4}3m$) $Z = 32$, $R = 4.8\%$ for 165 independent reflections. The structure is related to that of fluoride but with the Tl atoms in a distorted eight-fold coordination.
M. Vlasse, J. Grannec and J. Portier, *Acta Crystallogr., Sect. B*, 28 (1972) 3426.

$BaTbO_3$
($R\bar{3}c$). The magnetic structure is G-type antiferromagnetic (collinear) and the magnetic moment of Tb^{4+} is calculated to be 6.66(11) B.M.
E. Banks, S.J. La Placa, W. Kunnmann, L.M. Corliss and J.M. Hastings, *Acta Crystallogr., Sect. B*, 28 (1972) 3429.

Potassium salt of μ_4-oxo-hexa-μ-chloro-tetra[chlorocuprate(II)], $K_4Cu_4OCl_{10}$
($C2/c$) $Z = 2$, $R = 6.8\%$ for 544 unique reflections. The tetrahedron formed by the Cu^{2+} ions and the oxygen in the centre is somewhat irregular (Cu–O–Cu, 108.9(1)–110.3(1)°), as is the octahedron formed by the bridging chlorines. The potassium ions are in the open spaces between the anions; K–Cl, 3.1 Å; cf. solid KCl, 3.138 Å.
J.J. deBoer, D. Bright and J.N. Helle, *Acta Crystallogr., Sect. B*, 28 (1972) 3436.

Potassium$(-)_{589}$-tris(1.2-benzenediolato)arsenate(V) sesquihydrate, $(-)_{589}$-K[As(cat)$_3$].1.5H_2O
($P2_12_12_1$) $Z = 8$, $R = 5.5\%$ for 1428 reflections. The complex anion has approximate D_3 symmetry. Each ligand molecule is coordinated to the central arsenic atom by their oxygen atoms; As–O(average), 1.84 Å. The configuration of the anion is Δ, in accordance with the circular dichroism spectra.
A. Kobayashi, T. Ito, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 3446.

Magnesium chlorophosphate, $Mg_2(PO_4)Cl$
($Pna2_1$) $Z = 4$, $R = 2.5\%$ for 784 independent reflections. The compound is isostructural with the previously reported $Mn_2(PO_4)Cl$.
J.R. Rea and E. Kostiner, *Acta Crystallogr., Sect. B*, 28 (1972) 3461.

γ-Uranyl dihydroxide, $UO_2(OH)_2$
($P2_1/c$) $Z = 2$, $R = 6.1\%$ for 317 independent reflections. The configuration about uranium is a distorted octahedron composed of two O(1) uranyl and four O(2) secondary oxygen atoms. Each O(2) atom is shared between two octahedra while each O(1) is associated with one octahedron but hydrogen bonds to an O(2) atom.
S. Siegel, H.R. Hoekstra and E. Gebert, *Acta Crystallogr., Sect. B*, 28 (1972) 3469.

Potassium pentachloronitrosylruthenate(II), $K_2[Ru(NO)Cl_5]$

($P2_12_12_1$) $Z = 4, R = 4.0\%$ for 1347 independent reflections. The $[Ru(NO)Cl_5]^{2-}$ anion is a slightly distorted octahedron with Ru–Cl equal to 2.359(2) for Cl *trans* to the nitrosyl group, shorter than the average *cis* Ru–Cl, 2.372(8) Å. Ru–N, 1.747(6); N–O, 1.112(7) Å.

J.T. Veal and D.J. Hodgson, *Acta Crystallogr., Sect. B,* 28 (1972) 3525.

Sborgite, $NaB_5O_6(OH)_4.3H_2O$

($C2/c$) or (Cc) $Z = 8, R = 6.2\%$ for 1618 independent reflections. The structure contains the pentaborate ion $[B_5O_6(OH)_4]^-$ characterized by the double ring built up from one BO_4 tetrahedron and three BO_3 triangles. One sodium is octahedrally coordinated by four water molecules and two hydroxyl ions, the other is tetrahedrally coordinated by two water molecules and two hydroxyl ions.

S. Merlino and F. Sartori, *Acta Crystallogr., Sect. B,* 28 (1972) 3559.

$Na_2Mg_2Si_6O_5$

($Cmca$) or ($C2cb$) $Z = 8, R = 8.2\%$ for 358 reflections. The silicate anions were corrugated ribbons, two tetrahedra wide and are made up from rings of four tetrahedra fused together.

M.E. Cradwick and H.F.W. Taylor, *Acta Crystallogr., Sect. B,* 28 (1972) 3583.

$HK_8Rh_3Br_{18}.10H_2O$

($Pbam$) $Z = 2, R = 9.0\%$ for 454 independent reflections. Two monomeric octahedra exist which are unrelated by crystal symmetry. Rh–Br, 2.50 Å.

J. Coetzer, W. Robb and P.V.Z. Bekker, *Acta Crystallogr., Sect. B,* 28 (1972) 3587.

$(+)_{546}$-Tris-(R,R-2,4-diaminopentane)cobalt(III) chloride monohydrate,
$(+)_{546}$-$[Co(R,R\text{-ptn})_3]Cl_3.H_2O$

($P2_12_12_1$) $Z = 4, R = 7.6\%$ for 701 reflections. The cation has approximate D_3 symmetry. The six-membered chelate ring has a twisted boat form. The two methyl groups are in equatorial positions with respect to the average plane of the chelate ring. The average NCoN angle is 87.9 ± 1.3°. The absolute configuration of the complex cation is Δ.

A. Kobayashi, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B,* 28 (1972) 3591.

Copper(II) bis(N,N'-di-n-propyldithiocarbamate)

($P2_1/a$) $Z = 4, R = 7.0\%$ for 2534 reflections. The copper atom is located inside a tetragonal pyramid, because of a centrosymmetric dimerization of the complex, at 0.38 Å above the base plane of four sulphur atoms, coplanar within ± 0.006 Å and at 2.74 Å below the fifth sulphur atom belonging to the second molecule of the dimer. The copper atom has a short Cu$\cdots$H intermolecular contact of 2.93 Å corresponding to the sixth position of a distorted octahedral coordination.

G. Peyronel, A. Pignedoli and L. Antolini, *Acta Crystallogr., Sect. B*, 28 (1972) 3596.

αUO_3 by neutron and electron diffraction

12 ± 1% of the uranium sites are vacant in a disordered manner and neighbouring U–O distances along the uranium–oxygen chains are shortened to 1.64(4) Å.

C. Greaves and B.E.F. Fender, *Acta Crystallogr., Sect. B*, 28 (1972) 3609.

Hanksite, $KNa_{22}[Cl(CO_3)_2(SO_4)_9]$

($P6_3/m$) $Z = 2$, $R = 3.4\%$. The structure exhibits an ordered distribution of both SO_4 tetrahedra and CO_3 groups. The relationship to the K_2SO_4 I structure type is discussed.

K. Kato and H. Saalfeld, *Acta Crystallogr., Sect. B*, 28 (1972) 3614.

Chlorobis(*N*-*n*-propylsalicylaldiminato)iron(III), $C_{20}H_{24}N_2O_2ClFe$

($C2/c$) $Z = 4$, $R = 11.6\%$ for 571 independent reflections. The structure consists of discrete monomeric units with symmetry 2. The iron atom is five-coordinate, intermediate between trigonal bipyramidal and square pyramidal. The bond distances Fe–Cl, Fe–O and Fe–N are 2.23, 1.89 and 2.09 Å.

J.E. Davies and B.M. Gatehouse, *Acta Crystallogr., Sect. B*, 28 (1972) 3641.

Cl_2TeS_7 and Br_2TeS_7

The compounds are isomorphous; ($Pmnb$) $Z = 4$, $R = 3.1\%$ (chloride) and $R = 5.3\%$ (bromide). The structure of the molecules is derived from S_8 by substituting one sulphur atom by the X_2Te group.

J. Weiss and M. Pupp, *Acta Crystallogr., Sect. B*, 28 (1972) 3653.

Strontium hydroxide phosphate, $Sr_5(PO_4)_3OH$

($P6_3/m$) $Z = 2$, $R = 5.3\%$. The phosphate tetrahedra are less distorted than in the isostructural hydroxyapatite $Ca_5(PO_4)_3OH$. Both substances are OH-deficient with charge balance maintained by Sr or Ca vacancies.

K. Sudarsanan and R.A. Young, *Acta Crystallogr., Sect. B*, 28 (1972) 3668.

Bis(cyclopentadienyl)beryllium in the crystal at −120° and in the gas phase

The structure contains two π-bonded rings, one of which has slipped (or one pentahapto and one poly-hapto ring). The BeC(5′) bond is perpendicular to the plane at the ring and appears to be longer (1.81(5) Å) than the single Be–C(Me) distance in $C_5H_5BeCH_3$ (1.706(7) Å).

D.A. Drew and A. Haarland, *Acta Crystallogr., Sect. B,* 28 (1972) 3671.

Cu_3PSe_4

($Pmn2_1$) $Z = 2, R = 10\%$ for 311 independent reflections.

Cu_3SbS_4

($I\bar{4}2m$) $Z = 2, R = 9.0\%$ for 166 reflections.

Both compounds crystallize with the famatinite structure, a zinc-blende related normal tetrahedral structure type.

J. Garin and E. Parthé, *Acta Crystallogr., Sect. B,* 28 (1972) 3672.

Dirubidium dimercaptomaleonitrile monohydrate, $Rb_2DMMN.H_2O$

($Pmc2_1$) $Z = 2$. The coordination of Rb inside the layers is 8, and that inside Rb–water sheets parallel to the x–y plane is 7, thus yielding the formula $[Rb^{[7]}(H_2O)_n]_n[Rb^{[8]}(DMMN)]_n$.

M. Drager and G. Gatton, *Z. Anorg. Allg. Chem.,* 391 (1972) 203.

Lithium amide

($I\bar{4}$) $Z = 8$. The nitrogen has a distorted cubic close-packed arrangement. The cations occupy tetrahedral interstices.

H. Jacobs and R. Juza, *Z. Anorg. Allg. Chem.,* 391 (1972) 271.

$NaBiO_2$

($C2/c$) $Z = 4, R = 10.9\%$ for 555 reflections. Angular $[BiO_2]$ groups are twisted by 180°, forming chains along the c axis. Lattice energy is discussed.

B. Schwedes and R. Hoppe, *Z. Anorg. Allg. Chem.,* 391 (1972) 313.

$H_4W(CN)_8.4HCl.12H_2O$

($C2/c$) $Z = 4, R = 11.2\%$ for 2372 reflections. The $W(CN)_8^{4-}$ ion has only C_2 symmetry whilst the coordination polyhedron is an approximate square antiprism. W–C, 2.176; C–N, 1.151; W–N, 3.323 Å. Angle W–C–N is 175.3°.

L.D.C. Bok, J.G. Leipoldt and S.S. Basson, *Z. Anorg. Allg. Chem.,* 392 (1972) 303.

μ-Butadiene-dimanganese-octacarbonyl, $Mn_2(CO)_8C_4H_6$(I)
($P2_1/n$) $Z = 4$, $R = 5.9\%$. The two manganese atoms are linked by a *trans*-butadiene bridge. The carbon—carbon distances in the butadiene ligand are all equally long; they are shortened in comparison with other butadiene complexes.
H.E. Sasse and M.L. Ziegler, *Z. Anorg. Allg. Chem.*, 392 (1972) 167.

Refinements of KN_3, RbN_3, CsN_3 and TlN_3
These isostructural compounds crystallize in a tetragonal variety of the CsCl type ($I4/mcm$) with $Z = 4$. The metal ions are surrounded by eight closest N atoms in a distorted quadratic antiprismatic arrangement at distances which correspond to the sum of ionic radii. The azide ions are strictly linear; N–N, 1.16–1.18 Å.
U. Muller, *Z. Anorg. Allg. Chem.*, 392 (1972) 159.

$KBiO_2$, $RbBiO_2$ and $CsBiO_2$
$KBiO_2$ crystallizes in ($C2/c$) $Z = 4$. Angular BiO_2 groups (104.5°) are twisted by 180° forming chains along [101]. According to powder data $RbBiO_2$ and $CsBiO_2$ are isotypic with $KBiO_2$.
B. Schwedes and R. Hoppe, *Z. Anorg. Allg. Chem.*, 392 (1972) 97.

$Cs_2Mn_2(C_2O_4)_3 . 3H_2O$
($P\bar{1}$) $Z = 1$, $R = 4.9\%$.
H. Siems and J. Lohn, *Z. Anorg. Allg. Chem.*, 393 (1972) 97.

$Na_3[Yb(NH_2)_6]$
($Pbca$) $Z = 8$, $R = 5.2\%$ for 1237 reflections. The amide ions have a distorted close packed arrangement with the layer sequence ABAC in the direction [010]. Ytterbium occupies one sixth, sodium one half of the octahedral interstices.
C. Hadenfeldt and H. Jacobs, *Z. Anorg. Allg. Chem.*, 393 (1972) 111.

Thallium trifluoride, TlF_3
($Pnma$) $Z = 4$, $R = 15\%$ for 342 reflections. Each Tl has 8 nearest F-neighbours and Tl–F distances are 2.09–2.49 Å.
Ch. Hebecker, *Z. Anorg. Allg. Chem.*, 393 (1972) 223.

Thorium(IV) tetrakis(trifluoroacetylacetonate)
($C2/c$) $Z = 4$, $R = 14.8\%$ for 1974 reflections. The thorium atom is coordinated by 8

oxygen atoms in the form of a 1111 (D_4-422) antiprism. Th–O(average) is 2.39 ± 0.04 Å. The trifluoroacetylacetonate rings are approximately planar except for CH_3 and CF_3 groups, which show significant deviations.

G.F.S. Wessels, J.G. Leipoldt and L.D.C. Bok, *Z. Anorg. Allg. Chem.*, 393 (1972) 284.

Racemic μ-amido-μ-peroxo-bis[bis(ethylenediamine)cobalt(III)] trithiocyanate hydrate, DL-$[(en)_2Co.\mu(NH_3, O_2).Co.Co(en)_2](SCN)_3.H_2O$

(*Pbca*) $Z = 8$, $R = 7.6\%$ for 2172 reflections. The cobalt atoms are octahedrally coordinated. The five-membered central ring of the cations is considerably puckered. The O–O distance in the O_2 bridge is 1.46 Å. This geometry indicates that the O_2 bridge is properly described as a peroxo group.

U. Thewalt, *Z. Anorg. Allg. Chem.*, 393 (1972) 1.

Cs_2PbO_3

(*Cmc*2_1) $Z = 4$, $R = 11.8\%$ for 318 reflections. Pb^{4+} possesses a coordination number of five to O^{2-}, and in the structure there are ${}^1_\infty[PbO_{4/2}O_1]$ chains with a tetragonal-pyramidal environment for Pb^{4+}; Cs^+ has 3 + 1 + 2 O^{2-} neighbours.

P. Panek and R. Hoppe, *Z. Anorg. Allg. Chem.*, 393 (1972) 13.

trans-$[CoCl_2(en)_2][TlCl_4]$

($P2_1/m$) $Z = 2$, $R = 10.2\%$ for 372 reflections. The structure consists of isolated units of pseudo-octahedral *trans*-$[CoCl_2(en)_2]^+$ cations and slightly distorted tetrahedral $TlCl_4^-$ ions which have *Cs* symmetry but virtually equal Tl–Cl bond lengths.

K. Brodersen, J. Rath and G. Thiele, *Z. Anorg. Allg. Chem.*, 394 (1972) 13.

Dioxodifluorovanadates and dioxodichlorovanadates(V), $(C_6H_5)_4M[VO_2X_2]$ (M = P, As; X = F, Cl)

The preparation, IR spectra and crystal structures are discussed.

E. Ahlborn, E. Diemann and A. Müller, *Z. Anorg. Allg. Chem.*, 394 (1972) 1.

$2ZnCl_2.C_6H_5O_3N.H_2O$

($P2_1/c$) $Z = 4$, $R = 4.4\%$ for 579 reflections. One half of the zinc atoms are coordinated octahedrally by a chlorine, water and by the nitrogen and three oxygen atoms of the triethanolamine molecule. The other half is tetrahedrally surrounded by chlorine. The two zinc complexes are connected via a common chlorine atom.

H. Follner, *Z. Anorg. Allg. Chem.*, 387 (1972) 43.

[XeF_3^+] [$Sb_2F_{11}^-$]

($P\bar{1}$) $Z = 2, R = 3.5\%$ for 1823 independent reflections. The T-shaped cation is planar lying in the same plane as a fourth fluorine atom, which makes close contact of 2.5 Å with the xenon atom. This F atom, although part of the $Sb_2F_{11}^-$ ion, has a longer Sb–F bond of 1.90 Å.

D.E. McKee, A. Zalkin and N. Bartlett, *Inorg. Chem.*, 12 (1973) 1713.

[XeF^+] [RuF_6^-] (A) and [XeF_5^+] [RuF_6^-] (B)

(A) ($P2_1/n$) $Z = 4, R = 7.0\%$ for 1044 independent reflections. Within the discrete $XeRuF_7$ units, the xenon atom is approximately linearly coordinated to two fluorine atoms. ∠F–Xe–F is 177.1(1.2)°. One fluorine is bound to xenon alone and the other to ruthenium as well. Five other fluorines make up a distorted octahedron around ruthenium.

(B) ($Pnma$) $Z = 4, R = 4.2\%$ for 556 reflections. There are discrete XeF_5 and RuF_6 units with each XeF_5 group coordinated to four RuF_6 groups via one F atom on each RuF_6 group. The four fluorines and five XeF_5 fluorines pack in a distorted capped Archimedean antiprism arrangement. The RuF_6 group is a slightly distorted octahedron.

N. Bartlett, M. Gennis, D.D. Gibler, B.K. Morrell and A. Zalkin, *Inorg. Chem.*, 12 (1973) 1717.

[XeF_5^+]$_2$ [PdF_6^{2-}]

($Pca2_1$) $Z = 4, R = 2.56\%$ for 1464 independent reflections. The PdF_6^{2-} ion is almost octahedral and only one PdF distance is significantly different from the average of 1.893 Å. The XeF_5^+ ions approximate to C_{4v} symmetry.

K. Leary, D.H. Templeton, A. Zalkin and N. Bartlett, *Inorg. Chem.*, 12 (1973) 1726.

A pentacoordinate copper(II) hippurate dimer, tetraaquobis(μ-hippurato-*O*)-bis(hippurato-*O*) dicopper, $Cu[(C_6H_5)CONHCH_2.COO]_2.4H_2O$

($P2_1/C$) $R = 1.4\%$ for 1693 reflections. Each copper atom has distorted [4 + 1] tetragonal-pyramidal geometry and bridging is through two carboxyl oxygens with distances of 1.93 and 2.37 Å. The hippurate ligands are monodentate.

J.N. Brown and L.M. Trefonas, *Inorg. Chem.*, 12 (1973) 1730.

$(+)_{470}$-Δ-β-*cis*-Dinitro(5-methyl-1,4,8,11-tetraazaundecane)cobalt(III) bromide, $[Co\{NH_2(CH_2)_2NH(CH_2)_2CH(CH_3)NH(CH_2)_2NH_2\}(NO_2)_2]Br$

($P2_1$) $Z = 2, R = 3.4\%$ for 2570 reflections. The coordination around cobalt is slightly distorted octahedral with average Co–N, 2.000(2) Å for three in-plane atoms. The in-plane terminal group Co–N is 1.968(4) Å: Co-NO_2, 1.936(2) Å. The cation has *β-cis* geometry with substituent methyl groups adjacent to the out-of-plane chelate ring. The arrangements at the asymmetric centres are *S* for each nitrogen and *R* for the carbon atom.

P.W.R. Corfield, J.C. Dabrowiak and E.S. Gore, *Inorg. Chem.*, 12 (1973) 1734.

$[\pi\text{-}\{O \overline{\cdots} C(CH_3) \overline{\cdots} C(CH_3)\}\text{–}C_6H_4P(C_6H_5)_2Mn(CO_3)]$

($P\bar{1}$) $Z = 2$, $R = 3.8\%$ for 2953 independent reflections. The manganese coordination is distorted octahedral with three carbonyl ligands occupying mutually *cis* sites. The remaining sites are occupied by the phosphorus atom and the delocalized pseudo-π-allylic isopropenyl substituent of the 2-(1′,2′-dimethyloxopropenyl)phenyldiphenyl phosphine ligand. The π-oxopropenyl group is asymmetrically bonded to the manganese atom; Mn–C, 2.219(3) and 2.126(3) Å; Mn–O, 2.058(2) Å.

G.B. Robertson and P.O. Whimp, *Inorg. Chem.*, 12 (1973) 1740.

$\pi\text{-}C_5H_5Fe(CO)_3PF_6$

(*Pbca*) $Z = 8$, $R = 8.3\%$ for 2662 reflections. The iron coordination is octahedral with three carbonyl monodentate ligands and one $C_5H_5^-$ tridentate ligand. The positively charged iron carbonyl complex exhibits C–O distances that are significantly shorter and Fe–C distances that are longer than in the neutral species.

M.E. Gress and R.A. Jacobson, *Inorg. Chem.*, 12 (1973) 1746.

The ethylenediamine tetraacetatoaquomagnesate ion in a crystalline sodium salt, $Na_2[H_2OMgEDTA].5H_2O$

(C_2) $Z = 2$, $R = 6.5\%$ for 2791 independent reflections. The seven-coordinate pentagonal bipyramidal ion $[H_2OMgEDTA]^{2-}$ of symmetry C_2 carries two pairs of very long bonds (Mg–N, 2.378(2) and Mg–O, 2.270(3) Å) along with a short pair (Mg–O, 2.078(2) and Mg–OH_2, 2.060(3) Å).

J.J. Stezowski, R. Countryman and J.L. Hoard, *Inorg. Chem.*, 12 (1973) 1749.

Hexaaquomagnesium dihydrogenethylenediamine tetraacetate, $Mg(OH_2)_6H_2EDTA$

($P2_1/c$) $Z = 2$, $R = 4.5\%$ for 2529 reflections. The presence of the N–H bonds in H_2EDTA^{2-} leads to an averaged C–N bond length of 1.498 as compared with 1.471 Å in the $[H_2OMgEDTA]^{2-}$ chelate.

M. O'Donnell Julian, V.W. Day and J.L. Hoard, *Inorg. Chem.*, 12 (1973) 1754.

Oxoisopropoxobis(8-hydroxyquinolinato)vanadium(V)

($P\bar{1}$) $Z = 2$, $R = 4.3\%$ for 3317 reflections. The VO(O–i-Pr) group is in the *cis* configuration with ∠O–V–O, 101.9(1)°; V–O are 1.600(1) and 1.774(2) Å to the oxo and isopropoxo ligands respectively. The V–N bond *trans* to oxo is 2.317(2) and that *trans* to isopropoxo is 2.209(3) Å. Two oxygen atoms of the 8-hydroxyquinolato ligands, V–O 1.902(2) and 1.925(2) Å complete the octahedral coordination group.

W.R. Scheidt, *Inorg. Chem.*, 12 (1973) 1758.