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X-RAY BIBLIOGRAPHY

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1 : 1 Addition compound of molecular oxygen with ethylene bis (acetylacetoneiminato)-cobalt (II) monopyridinate, [Co(acacen). O_2.py]

($P2_1/c$) $Z = 4$, R = 19% for 587 independent reflections. The structure consists of discrete molecules in which the cobalt atom has a hexacoordinate stereochemistry; axial pyridine (Co–N, 2.15(4) Å) and one oxygen of the O_2 molecule (Co–O, 1.95(5) Å). The quadridentate ligands occupy the four equatorial positions.

M. Calligaris, G. Nardin, L. Randaccio and G. Tauzher, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 419.

$Na_2[UO_2(\text{oxydiacetate})_2] . 2H_2O$

($P2_1/c$) $Z = 4$, R = 9.5% for 1301 independent reflections. The anionic ligands act as tridentate ligands and eight-coordination to uranium is realized by an irregular hexagon of six oxygen atoms bonded in the base plane which is normal to the linear uranyl group. The sodium ions are surrounded by carboxylic oxygens in an approximate octahedron.

G. Bombieri, R. Graziani and E. Forsellini, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 551.

The silver perchlorate adduct of iron (III) trisacetylacetonate, $Fe(AA)_3.AgClO_4$

($P2_1/c$) $Z = 4$, R = 13% for 1100 reflections. The oxygens of the acetylacetonate groups are arranged octahedrally around the iron atom. Fe–O (average), 1.99 Å. Silver is bonded to an acetylacetonate oxygen and to one of the perchlorate oxygens and exhibits trigonal pyramidal coordination, as found in other silver perchlorate aromatic complexes.

L.R. Nassimbeni and M.M. Thackeray, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 539.

Erbium acetate tetrahydrate

($P\bar{1}$) $Z = 2$, R = 10.6% for 2207 reflections. Two of the independent carbonyl groups in the centrosymmetric dimer are bidentate cyclic while the third is tridentate bridge-cyclic. The coordination number of erbium is nine, and the polyhedron can be described as a three-vertex trigonal prism.

L.A. Aslanov, I.K. Abdul'Minev, M.A. Porai-Koshits and V.I. Inanov, *Dokl. Chem.*, 205 (1972) 568.

Cadmium hexacyanocobaltate (III) (A) and cadmium hexacyanocobaltate (III) hydrate (B)

R = 10.5% and 7.3% for (A) and (B) respectively. The dehydration does not affect the interatomic distances. Co–C, 1.91; C–N, 1.13, and Cd–N, 2.25 Å.

G. Ron, A. Ludi and P. Engel, *Chimia*, 27 (1973) 77.

Cis- (A) and *trans*- (B) $PtCl_2[C(NPhCH_2)_2]PEt_3$

($Pnam$) $Z = 4$, $R = 4.8\%$ for 1808 reflections (A). The coordination plane of the platinum atom coincides with the crystallographic mirror plane to which the five-membered ring containing the carbene atom is perpendicular. The geometries of the carbene ligands are very similar in the two compounds.

D.J. Cardin, B. Cetinkaya, E. Cetinkaya, M.F. Lappert, L.J. Muir and K.W. Muir, *J. Organometal. Chem.*, 44 (1972) C59.

Sodium pentaphenylchromate, $Na_2Cr(C_6H_5)_5.3C_4H_{10}O.C_4H_8O$

($P2_1/n$) $Z = 4$. A distorted trigonal bipyramidal configuration is found for the arrangement of the five phenyl ligands about the five-coordinate Cr^{III} ion. Steric hindrance and the Jahn–Teller effect explain the deviations from ideal D_{3h} symmetry. The Cr–C distances are shorter than the usual true σ-bond lengths.

E. Müller, J. Krause and K. Schmiedeknecht, *J. Organometal. Chem.*, 44 (1972) 127.

$(LiC_5H_5CrCl_3.2C_4H_8O_2).C_4H_8O_2$

($P\bar{1}$) $Z = 1$, $R = 9.3\%$ for 1614 observed reflections. The point symmetry of the molecule is C_i. The metal atoms contain Cl and dioxane bridges: Cr–Cl–3–Li–dioxane–Li′–Cl–3′–Cr′. The chromium (III) is coordinated by an antiprism with trigonal and pentagonal (π-cyclopentadienyl ring) faces. The Cr–C(Cp) distances differ significantly (mean 2.26 Å).

B. Müller and J. Krausse, *J. Organometal. Chem.*, 44 (1972) 141.

N-[2-(Chloromercuri) ethyl] diethylamine, $(CH_3)_2-NCH_2CH_2-HgCl$

($P2_1/n$) $Z = 4$, $R = 5.6\%$ for 586 independent reflections. There is no Hg–N intramolecular coordination, in accord with NMR observations. N–Hg (intermolecular), 2.77 (2) Å, which is less than the van der Waals contact distance.

K. Toman and G.G. Hess, *J. Organometal. Chem.*, 49 (1973) 133.

Two metal carbonyl derivatives of PH_3 : $(CO)_3Cr(PH_3)_3$ (A) and $(CO)_5Cr(PH_3)$ (B)

($P2_1/m$) $Z = 2$, $R = 5.6\%$ (A). The idealized octahedral molecule (A) shows approximate C_{3v} symmetry. Cr–P(mean), 2.346 (3) Å. ($Pnma$) $Z = 4$ (B). The PH_3 group is disordered over three mutually *cis* positions of the coordination octahedron. Structural parameters are very similar to those of (A).

G. Huttner and S. Schelle, *J. Organometal. Chem.*, 47 (1973) 383.

[Tris(trimethylsilyl)silylpentacarbonylmanganese $(Me_3Si)_3SiMn(CO)_5$
($P\bar{1}$) $Z = 2$, $R = 5.9\%$ for 818 independent reflections. The coordination geometry around manganese is approximately octahedral and that about the silicon atom bonded to the manganese atom is tetrahedral. Si–Mn is 2.564(6) Å and shows no multiple character.
B.K. Nicholson, J. Simpson and W.T. Robinson, *J. Organometal. Chem.*, 47 (1973) 403.

The reaction product of cyclopentadiene with 2-ferrocenyl-2-propyl cation
($P2_1/c$) $Z = 4$, $R = 11\%$, 924 independent reflections. The iron atom is a perpendicular distance of 1.64(1) Å from both planes and the two cyclopentadienyl rings are in the eclipsed conformation with mean carbon-to-carbon bond lengths of 1.422(4) and 1.420(4) Å.
T.S. Cameron, J.F. Maguire, T.D. Turbett and W.E. Watts, *J. Organometal. Chem.*, 49 (1973) C79.

Bis{μ-[(trifluoromethyl)seleno] manganese tetracarbonyl}
($P2_1/c$) $Z = 2$, $R = 11.5\%$ for 833 unique reflections. The molecule has a crystallographic centre of symmetry and is based on a planar $[MnSe]_2$ unit with four almost equal Mn–Se bonds. Mn–Mn, 3.74; Se–Se, 3.31 Å; Se–Mn–Se, 83.1°; Mn–Se–Mn, 96.9°; Mn–C(mean) 1.83; C–O(mean) 1.16; Se–C, 1.97; C–F(mean), 1.31 Å.
C.J. Marsden and G.M. Sheldrick, *J. Organometal. Chem.*, 40 (1972) 175.

$[(\pi\text{-}C_5H_5)Fe(CO)_2(C{\equiv}CPh)CuCl]_2$
($P2_1/a$) $Z = 4$, $R = 9.5\%$ for 1838 reflections. In the dimeric complex there are two asymmetric units related by a centre of inversion. The Cu_2Cl_2 ring is planar with angles at Cu and Cl, 94.6(2)° and 85.4(2)° respectively. Cu–Cl are all 2.267(5) Å and Cu–Cu is 3.075(4) Å. Each copper is symmetrically bonded to the C_2 unit of the phenylethynyl group, the CuC_2 moieties being coplanar with the Cu_2Cl_2 ring.
M.I. Bruce, R. Clark, J. Howard and P. Woodward, *J. Organometal. Chem.*, 42 (1972) C107.

Two π-diene-π-cyclopentadienylrhodium(I) complexes
($P2_1/n$) $Z = 4$, $R = 5.7\%$ for 1724 reflections ($C_9H_9Cl_2Rh$) (A) and ($P2_1/n$) $Z = 4$, $R = 5.5\%$ for 1752 reflections ($C_{11}H_{15}Rh$) (B). The π-cyclopentadienyl ring and the butadiene group are both planar in the two compounds, intersecting at 12.3° and 10.6° respectively. The metal atom is respectively 1.88 and 1.64 Å (A) and 1.87 and 1.67 Å (B) from these two least-squares planes.
M.A.B. Drew, S.M. Nelson and M. Sloan, *J. Organometal. Chem.*, 39 (1972) C9.

The urea adduct of calcium bromide, $CaBr_2 \cdot 4CO(NH_2)_2$
($P2_1/c$) $Z = 2$. A preliminary investigation only, no structural details given.
Z. Purski and P. Kuśnierek, *Rocz. Chem.*, 46 (1972) 1441.

Rubidium trihydrogen selenite, $RbH_3(SeO_3)_2$

($P2_12_12_1$) $Z = 4$, $R = 3.0\%$ for 1172 independent reflections. The structure consists of two types of chain at right angles, one built up of H_2SeO_3 molecules linked by 2.594 (8) Å hydrogen bonds and the other $HSeO_3^-$ ions linked by 2.571 (12) Å hydrogen bonds. The rubidium atom is surrounded by eight oxygen atoms forming a distorted cube. Rb–O, 2.94–3.19 Å.

R. Tellgren, D. Ahmad and R. Liminga, *J. Solid State Chem.*, 6 (1973) 250.

$TeCl_4.PCl_5$ ($=PCl_4^+TeCl_5^-$)

($Ima2$) $Z = 8$, $R = 5.9\%$. Besides isolated PCl_4^+ cations (P–Cl(mean), 1.925 Å) with nearly T_d symmetry, the structure contains chain-like polymeric $(TeCl_5^-)_n$ anions with distorted octahedral coordination of the Te atoms. In the series $TeCl_6^{2-}$–PCl_4, $TeCl_5^-$, $TeCl_4$, $TeCl_3^+$–$AlCl_4^-$, $TeCl_3^+$–AsF_6^-, an increasing steric activity of the inert electron pair is observed and is discussed in connection with vibrational spectroscopic results.

B. Krebs, B. Buss and W. Berger, *Z. Anorg. Allg. Chem.*, 397 (1973) 1.

Pb_4SeBr_6, $Pb_5S_2I_6$ and $Pb_7S_2Br_{10}$

The compounds have common structural features with the pure halides of lead. In Pb_4SeBr_6 all Pb atoms have trigonal prismatic coordination by Br(Se), additional neighbours above the prism faces completing the coordination to 7, 8 or 9. In $Pb_5S_2I_6$ some of the Pb atoms are surrounded by 6I + 1S or 5I + 3S in the same extended trigonal prismatic arrangement, others are in the centre of PbI_6 octahedra. $Pb_7S_2Br_{10}$ is isostructural with Th_7S_{12}.

B. Krebs, *Z. Anorg. Allg. Chem.*, 396 (1973) 137.

InTeCl

($P2_1/c$) $Z = 8$, $R = 8.4\%$ for 932 observed reflections. The compound represents a new ternary defect tetrahedral type. Strongly distorted $InTe_3Cl$ tetrahedra form layer complexes situated parallel to the (100) plane by sharing corners and edges which are occupied by Te atoms. The Te atoms are surrounded by three In atoms in an approximately trigonal pyramidal arrangement.

G. Ross, G. Eulenberger and H. Hahn, *Z. Anorg. Allg. Chem.*, 396 (1972) 284.

Two mononuclear peroxotitanium(IV) dipicolinates, $[TiO_2(C_7H_3O_4N)(H_2O)_2].2H_2O$ (A) and $K_2\ [TiO_2(C_7H_3O_4N)F_2].2H_2O$ (B)

($P\bar{1}$) $Z = 2$, $R = 2.7\%$ for 3488 reflections (A); ($P2_1/c$) $Z = 4$, $R = 5.1\%$ for 2324 reflections (B). The titanium atoms are coordinated approximately pentagonal-bipyramidally, with the peroxo group and the chelate ligand occupying the equatorial sites and with H_2O or F^- forming the apices. The O–O bond length in the peroxo group is the same in all structures but there is a slight variation in the Ti–peroxide distances apparently connected with the colours of the compounds. The more basic the apical ligands, ($H_2O \rightarrow F^- \rightarrow \mu$-oxygen) the higher the frequency of the absorption band, the longer the Ti–peroxide distances and the shorter the apical bond lengths.

D. Schwarzenbach, *Helv. Chim. Acta*, 55 (1972) 2990.

Tetracarbonyl (*trans*-α, β-dibenzoylethylene) iron

($P\bar{1}$) $Z = 2$, $R = 10.2\%$ for 1800 unique reflections. The metal atom has a trigonal bipyramidal coordination with the ethylenic bond in the equatorial position. The planarity of the ethylenic ligand is destroyed and the double bond is inclined to the equatorial plane of the bipyramid. C=C(coordinated), 1.47; Fe–C(olefin) 2.10 Å,

V.G. Andrianov, Yu.T. Struchkov, M.I. Rybinskaya, L.V. Rybin and N.T. Gubenko, *J. Struct. Chem (USSR)*, 13 (1972) 86.

Lithium nitrilotriacetatocopper(II) trihydrate, $LiCu[N(CH_2CO_2)_3]^{3-}.3H_2O$

($P2_12_12_1$) $Z = 4$, $R = 12.0\%$. The sructure consists of chains of $\{Cu(N(CH_3 \cdot CO_2)_3)^{3-}]\}$ anions parallel to the C axis, lithium cations and water molecules. The coordination polyhedron of copper is a distorted tetragonal pyramid and the configuration is analogous to the coordination polyhedra in $Cu_2EDTA.4H_2O$. Cu–N, 1.85; Cu–O, 2.06, 1.93, 2.20, 2.03 Å. The long Cu–O bond is directed towards the vertex of the pyramid.

V.V. Fomenko, L.I. Kopaneva, I.N. Polynova, A.M. Porai-Koshits, N.D. Mitrofanova and L.I. Martywenko, *J. Struct. Chem. (USSR)*, 13 (1972) 166.

$Ni(en)_2I_2.H_2O$

($P2_12_12_1$) $Z = 4$, $R = 11.4\%$ for 1260 independent reflections. In the octahedral molecule I and water are *cis* to each other. Ni–N, 2.07–2.11 Å; Ni–H_2O is 2.13 Å; Ni–I, 2.91 Å. The metallocycles have *gauche* configuration and a *KK'* conformation. The parameters of the two ethylenediamine ligands are similar to those found in other Ni complexes.

L.Kh. Minacheva, A.S. Antsyshkina and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 13 (1972) 71.

Hydrazinium oxopentafluoroniobate monohydrate, $N_2H_5[NbOF_5].H_2O$

(*Pnam*) $Z = 4$, $R = 11.5\%$ for 700 independent reflections. The $[NbOF_5]^{2-}$ anion is octahedral with Nb–O 1.75(2) Å. Nb–F (*trans*) 2.21(2); Nb–F (*cis*) 1.93(2) and 1.96(2). The niobium atom is displaced by 0.27 Å from the equatorial plane of the sphere of the ligands in the direction of the oxygen atom. O–Nb–F (*cis*) is 101 (1)°.

Vu.E. Gorbonova, V.I. Pakhomov, V.G. Kuznetsov and E.S. Kovaleva, *J. Struct. Chem. (USSR)*, 13 (1972) 165.

Neodymium nitrate–dimethyl sulphoxide complex

($C2/c$) $Z = 4$, $R = 9.2\%$ for 1114 independent reflections. The metal atom lies on a two-fold symmetry axis and the coordination polyhedra has ten vertices, four of which are occupied by dimethyl sulphoxide oxygen atoms from three bidentate nitrogen groups. The coordination polyhedron can be described in terms of a dodecahedron with two doubled β-type vertices. Nd=O_{DMSO}, 2.37; Nd–O_{NO_3}, 2.64 Å.

L.A. Aslanov, L.I. Soleva, M.A. Porai-Koshits and S.S. Gookhberg, *J. Struct. Chem. (USSR)*, 13 (1972) 610.

π-Cyclopentadienyl-π-diphenylacetylene monocarbonyltriphenylstannyliron, $Ph_3Sn{-}Fe(CO).(\pi\text{-}C_2Ph_2)Cp$.

($P2_1/c$) $Z = 4$. The iron is coordinated with the π-cyclopentadienyl ligand (Fe—C, 2.15 Å). The valence angle at the carbon atoms of the coordinated triple bond is 153°. Sn—C(mean), 2.21 Å.

V.E. Shklober, V.V. Skripkin, A.E. Gusev and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 698.

(N-Benzyl-D-prolinato) (N-benzyl-L-prolinato) copper (II), $Cu[(CH_2)_3CH(NH.CH_2C_6H_5)COO]_2$

($P2_1/b$) $Z = 2$, $R = 16.8\%$ for 800 independent reflections. In the centrosymmetric molecule, the coordination of the copper atom is square planar with two oxygen etoms (Cu—O, 1.95 Å) and two nitrogen atoms (Cu—N, 1.92 Å) of the two chelate amino acids.

G.G. Aleksandrov, Yu.Y. Struchkov, A.A. Kurganov, S.V. Rogozhin and V.A. Dovakov, *J. Struct. Chem. (USSR)*, 13 (1972) 624.

$K_5(UO_2)_2F_9$

($B2/b$) $Z = 4$, $R = 15\%$. The structure consists of $[(UO_2)_2F_9]^{5-}$ anions and K^+ cations. The dimeric anion consists of two pentagonal bipyramids, with a bridging fluorine, as common vertex. The equatorial planes of the bipyramid form an angle of about 130° with each other and are somewhat distorted. The UO^{2+} groups in $K_5(UO_2)_2F_9$ have an almost linear and symmetrical structure and their orientation tion to the equatorial plane of the bipyramid in which the five F atoms are arranged is approximately normal.

Yu.N. Mikhailov, A.A. Udovenko, V.G. Kuznetsov and R.L. Davidovich, *J. Struct. Chem. (USSR)*, 13 (1972) 694.

$Cs_4[(UO_2)_2F_8].2H_2O$

($P2_1/c$) $Z = 2$, $R = 16\%$. The main structural units are dimeric $[(UO_2)_2F_8]^{4-}$ anions, Cs^+ cations and water molecules. The dimeric anion consists of two pentagonal bipyramids with one common edge, F—F. The almost linear and symmetric $UO_2{}^{2+}$ groups are oriented approximately normally to the equatorial plane of the bipyramids in which the five F atoms are arranged.

Yu.N. Mikhailovich, A.A. Udovenko, V.G. Kuznetsov and R.N. Shchelokov, *J. Struct. Chem. (USSR)*, 13 (1972) 695.

Neodymium ethylenediaminetetraacetate bihydrate, $(CN_3H_6)[Nd(C_2H_4N_2(CH_2COO)_4)]$

($Pn2_1a$) $Z = 4$. The structure consists of hexadentate complex anions $Nd(OH_2)_3[C_2H_4N(CH_2COO)_4]^-$ and $[CN_3H_5]^+$ cations. The neighbouring region of the neodymium atom consists of two nitrogen atoms and four oxygens, and three waters forming an asymmetric nine-apex structure. Nd—O, 2.53 and Nd—N, 2.83 Å.

Ya.M. Nesterova, S.G. Zbryskaya, T.N. Polynova and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 13 (1972) 691.

Magnesium zinc ethylenediaminetetraacetate hexahydrate, $MgZn(C_2H_4N_2(CH_2COO)_4).6H_2O$

($Pna2_1$) $Z = 4$. The coordination polyhedron of zinc is a distorted octahedron formed by four oxygen atoms and two nitrogen atoms with average distances Zn–O, 2.10(4); Zn–N, 2.24(4) Å. The magnesium atom is enclosed in an octahedron of four water molecules and two carbonyl oxygen atoms from the carbonyl groups of adjacent complexes; Mg–O (mean), 2.08(5) Å.

A.I. Pozhidalv, N.N. Neronova, T.N. Polynova, M.A. Porai-Koshits and V.A. Logvinenko, *J. Struct. Chem. (USSR)*, 13 (1972) 690.

Zinc hexamethylenedithiocarbamate

($P2_1/c$) $Z = 4$, $R = 12.6\%$ for 1516 reflections. In the centrosymmetric dimers two ligands serve as bridges between the two molecules making up the dimeric units. Two other ligands form four-membered metallocyclic rings. With weak additional bonds with the sulphur bridge atom being taken into account, the coordination number of the metal atom is five. The coordination polyhedron is intermediate between a tetragonal pyramid and a trigonal bipyramid.

V.M. Agre and E.A. Shugam, *J. Struct. Chem. (USSR)*, 13 (1972) 614.

Bis(2, 2, 6, 6-tetramethyl-1-ono-4-piperidylsalicylaldimine)zinc(II), $(C_{16}H_{22}N_2O_2)_2Zn$

(*Pbca*) $Z = 8$. 1170 unique reflections. Structural details are forthcoming.

S.D. Mamedov, M.K. Goseinova and A.D. Khalilov, *J. Struct. Chem. (USSR)* 13 (1972) 704.

Dysprosium thiocyanate heptahydrate, $Dy(NCS)_3.7H_2O$

($P2_1$) $Z = 2$, $R = 16.0\%$ for more than 1300 independent reflections. The coordination polyhedron of the dysprosium ion is formed by five water molecules and three nitrogen atoms of thiocyanate groups, arranged at vertices of a three-peak trigonal prism. A water molecule also bridges consecutive polyhedrons forming a chain.

P.I. Lazaren, M.A. Porai-Koshits, L.A. Aslanov and V.I. Ivanov, *J. Struct. Chem. (USSR)*, 13 (1972) 692.

Bis(chloromethyl) dichlorostannane, $(ClCH_2)_2SnCl_2$

(*Pmmn*) $Z = 2$, $R = 17.7\%$ for 129 independent reflections. The coordination of the tin atom differs significantly from the ideal tetrahedral one. ∠ Cl–Sn–Cl is 97°; ∠ C–Sn–C, 135°. Sn–Cl, 2.37; Sn–C, 2.18; Cl–C, 1.79 Å. The molecular conformation and the Sn–C–Cl angle of 108° show the absence of intramolecular Sn · · · Cl coordination.

N.G. Bokii, Yu.T. Struchkov and A.K. Prokof'ev, *J. Struct. Chem. (USSR)*, 13 (1972) 619.

Tetra-*t*-butylferrocene, $Fe(C_5H_3(t\text{-}C_4H_9)_2)_2$

(*Pbnb*) $Z = 8$, $R = 12\%$, for 800 independent reflections. The iron atoms in the two independent molecules are located at about the same distance from the cyclopentadienyl carbon atoms. The rings themselves are not quite parallel, being inclined at 7°, and this is attributed to steric repulsion between the bulky *t*-butyl groups in different rings.

Z.L.Kaluski, A.I. Gusev, A.E. Kalinin and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 888.

Properties of the crystalline structure of bismuth oxides

These oxides have basically either a hexacoordinate (prism and octahedron) or an octacoordinate (cube) structure with respect to oxygen. Taking into account that the oxygens occupy their positions statistically, the average coordination number of bismuth is less than eight. The oxides studied are BiO, $Bi_2O_{2,7,8}$, $Bi_2O_{2,3-2,4}$, $\beta.Bi_2O_{2,5}$ and $\delta.Bi_2O_3-x$.

A.A. Zav'yalova and R.M. Imamov, *J. Struct. Chem. (USSR)*, 13 (1972) 811.

Bis (triphenylgermyl) ether

$(P\bar{1})$ or $(P1)$ $Z = 2$, $R = 16.9\%$ for 1370 unique reflections. The molecule consists of two triphenylgermyl fragments joined by an oxygen bridge. ∠ Ge–O–Ge, 137°.

L.G. Kuz'mina and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 884.

$K_3(UO_2)_2F_7.2H_2O$

$(P2_1/m)$ $Z = 2$, $R = 14\%$ for 2500 unique reflections. The crystal is composed of infinite chains of $[(UO_2)_2F_7]^{3-}$ between which the potassium cations and water molecules are situated. The fluorouranylate chains are formed from slightly distorted UO_2F_5 pentagonal bipyramid linked to each other on the one side by a common edge and on the other side by a corner. The uranyl groups are almost linear and are directed approximately along the normal to the equatorial plane of the bipyramid in which five fluorine atoms are situated. U–F, 2.22–2.38 Å.

Yu.N. Mikhailov, A.A. Udovenko, V.G. Kuznetsov and R.L. Davidovich, *J. Struct. Chem. (USSR)*, 13 (1972) 879.

Bis(π-cyclopentadienylvanadium-bis-α-furancarboxylate), $[(\pi\text{-}C_5H_5)V(C_4H_3OCOO)_2\}_2$

$(B2/b)$ $Z = 4$, $R = 14.5\%$ for 1000 reflections. The complex is dimeric through four bridging carboxyl groups and there is no V–V bond. V • • • V, 3.625 Å. The vanadium atom has square pyramidal coordination with $\pi\text{-}C_5H_5$ in the apical position and four oxygens from bridging carboxylate ligands in the base positions.

N.I. Kirillova, A.I. Gusev, A.A. Pasynskii and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 820.

Potassium trisulphatozirconate, $K_4[Zr_2(SO_4)_6.4H_2O]$

$(P2_1/a)$ $Z = 4$, $R = 14.5\%$ for 855 independent reflections. The crystal structure is made up of $[(SO_4)_2(H_2O)_2Zr(SO_4)_2Zr(H_2O)_2(SO_4)_2]^{4-}$ anions and potassium cations. The zirconium coordination is a bicapped trigonal prism with Zr–O 2.04–2.31 Å. Each zirconium is surrounded by two bidenate and sulphate groups, one bridging sulphate and two water molecules.

M.A. Porai-Koshits, V.I. Sokol and V.N. Vorotnikova, *J. Struct. Chem. (USSR)*, 13 (1972) 815.

Dimethyl sulphoxide complex of erbium nitrate, $Er(NO_3)_3.3DMSO$

($P2_1/b$) $Z = 4$, $R = 9.2\%$ for 1240 independent reflections. The erbium atom is nine-coordinate; the coordination polyhedron is a tricapped trigonal prism. Oxygen atoms from these dimethyl sulphoxides are coordinated to one of the triangular bases of the trigonal prism. The other triangular base contains three oxygen atoms from three nitrate groups. Average Br–O(DMSO) is 2.27 Å; Er–O(NO_3) is 2.48 Å.

L.A. Aslanov, L.I. Soleva and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 13 (1972) 1021.

Ethylenediaminetetraacetates of divalent metals of formula $M_2A.nH_2O$ and $MgMA.nH_2O$, where A is $[C_2H_4N_2(CH_2COO)_4]^{4-}$ and M = Mg, Cu, Ba, Sr, Mn, Co, Ni, Cu, Zn and Cd

Parameters of unit cells only are presented. All three hexahydrates (Zn, Co, Mg, Zn) are isostructural and two of the four monohydrates (Mg and MgMg) are isostructural. Complete structural analysis of the above compound is at present being undertaken.

A.I. Pozkidaev, N.N. Noronova, T.N. Polynova, M.A. Porai-Koshits and V.A. Logvinenko, *J. Struct. Chem. (USSR)*, 13 (1972) 323.

Dimethylaminoaluminium, $[H_2NAlMe_2]$

($P2_1/b$) $Z = 12$, $R = 15\%$. The molecule is similar to a cyclohexane six-membered ring constructed from alternating AlH_2 and NMe_2 groups. Al–N(average), 1.93(3) Å. Coordination of the aluminium is a distorted tetrahedron. N–C(average), 1.54(4) Å. $\angle$ Al–N–Al, 115(2)°; $\angle$ N–Al–N, 108(2)°; $\angle$ C–N–C, 110(3)°.

K.N. Semenenko, L.B. Lubkovskii and A.L. Dorosinskii, *J. Struct. Chem. (USSR)*, 13 (1972) 696.

Bonito Katsuo ferrocytochrome *c* at 2.3 Å resolution

($P2_12_12_1$) $Z = 1$ (two molecules). The most prominent feature in the electron density map is the planar haem group with two dense peaks of sulphur atoms of Cys 14 and 17 located close to the two corners of the haem plane. The 5th and 6th ligands of the haem iron also stand out clearly in the map. The α-helices are seen at each terminus of the polypeptide chain. The helix at the amino terminus is about 2.5 turns and is somewhat deformed.

T. Ashida, N. Tanaka, T. Yamane, T. Trukihara and M. Kahudo, *J. Biochem. (Tokyo)*, 73 (1973) 463.

A model carrier complex formed between sodium bromide and 2, 3, 11, 12-dicyclohexyl-1, 4, 7, 10, 13, 16-hexaoxocyclo-octadeca-2, 11-diene ("dicyclohexyl-18-crown-6")

($P\bar{1}$) $Z = 1$, $R = 4.7\%$ for 1510 independent reflections. The six oxygens of the ligand form a planar ring around the sodium ion. The coordination sphere of sodium is completed by two water molecules, one above and one below the plane of this ring.

D.E. Fenton, M. Mercer and M.R. Truter, *Biochem. Biophys. Res. Commun.*, 48 (1972) 10

$(C_5H_5)_2TiCl_2$

($P\bar{1}$) $Z = 4$, $R = 13.5\%$ for 1650 independent reflections. Ti–Cl, 2.36; Ti–C, 2.43; C–C, 1.44 Å; Cl–Ti–Cl, 95.2°. The angle between the ring planes belonging to the same molecule is equal to 51° and there is a rotation of 35° of the rings relative to one another.

V.V. Tkachev and L.O. Atovmyan, *J. Struct. Chem. (USSR)*, 13 (1972) 263.

$Pr_2Sr_3(BO_3)_4$

($P2_1cn$) $Z = 4$ for 490 unique reflections. The structure contains the boron–oxygen anions BO_3^{3-}, having the form of approximately equilateral triangles with the boron atom in the centre and Pr^{3+} and Sr^{2+} cations. The latter link the boron–oxygen anions in a three-dimensional network: B–O 1.33, O–O 2.45 Å.

K.K. Palkina, V.G. Kuznetsov and L.G. Moruga, *J. Struct. Chem. (USSR)*, 13 (1972) 317.

$(NH_4)_4Mo_8O_{26}.4H_2O$

($P\bar{1}$) $Z = 1$, 2280 unique reflections, $R = 9.6\%$. Each molybdenum atom has a distorted octahedral environment. In three of the octahedra two oxygens are terminal and in the fourth, there is only one terminal oxygen. This octahedra is joined to six others by its edges.

L.O. Atovmyan and O.N. Krasochka, *J. Struct. Chem. (USSR)*, 13 (1972) 319.

Zirconium, hafnium and tetravalent cerium sulphates

$MO_3-SO_3-H_2O$ where M is Zr, Hf or Ce. Isomorphism was found among these sulphates.

D.L. Rogachev, A.S. Antsyshkina and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 13 (1972) 239.

α-2,2′-Dipyridyl lanthanum nitrate, $La(NO_3)_3.(C_{10}H_8N_2)_2$

($Pbcn$) $Z = 8$. The coordination number of lanthanum is ten, with four dipyridyl nitrogens and six nitrato oxygen atoms. The polyhedron can be roughly described as a distorted tetragonal antiprism capped on both bases. La–La, 7.72 Å. The two crystallographically different nitrate groups are both bidentate.

V.B. Krovchenko, *J. Struct. Chem. (USSR)*, 13 (1972) 324.

Lithium galloborate, $Li_6[Ga_2(BO_3)_4]$

($P\bar{1}$) $Z = 1$ for 247 non-zero reflections. The structure consists of alternating sheets formed from infinite chains of $[Ga_2(BO_3)_4]^{6-}$ and strips of lithium tetrahedra. The galloborate-radical chains consist of rings formed from two gallium tetrahedra and two boron triangles which are linked to each other in the same direction by two other boron triangles.

G.K. Abdullaev and Kh.S. Mamedov, *J. Struct. Chem. (USSR)*, 13 (1972) 881

Tetracyclopentadienylhafnium

($P\bar{4}2_1m$) $Z = 2$, $R = 14.7\%$ for 567 unique reflections. The hafnium atom is attached to two cyclopentadienyl rings in a sandwich-type structure, Hf–C, 2.50; C–C, 1.41 Å and forms localized σ-bonds of 2.34 Å with the other two rings. The coordination of hafnium can be regarded as strongly distorted tetrahedral.

V.I. Kulishov, N.G. Bokii and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 1029.

1,4-Diphenylbutadienetricarbonyliron, $[C_4H_4(Ph)_2Fe(CO)_3]$

($P\bar{1}$) $Z = 4$, $R = 15\%$ for 1587 reflections. The iron atom is coordinated in a tetrahedral pyramid by the butadiene and three carbon atoms. The average Fe–C distances to terminal and central butadiene carbons are 2.14 and 2.12 Å; Fe–CO, 1.80; C–O, 1.14 Å. The diphenylbutadienyl ligand is not flat; the angles between benzene rings and butadiene system are 26° and 17°.

L.G. Kuz'mina, Yu.T. Struchkov and A.I. Nekhaev, *J. Struct. Chem. (USSR)*, 13 (1972) 1003.

The normal (piperidinium salt) and acid (diethylamine adduct) salt of tetrakis(benzoylacetonato) europate, $HEu(BA)_4.nHAEt_2$ (A) and HPip$[Eu(BA)_4]$ (B)

($B2b$) $Z = 4$, $R = 15.0\%$ (A) and ($P2_1/c$) $Z = 4$, $R = 20.0\%$ (B). Crystals of both compounds contain monomeric tetrakis(benzoylacetonato) europate complexes. The coordination polyhedra are the same in the two complexes: *SSS* tetragonal antiprisms.

A.L. Il'inskii, M.A. Porai-Koshits, L.A. Aslanov and P.I. Lazarev, *J. Struct. Chem. (USSR)*, 13 (1972) 254.

Potassium bis(nitriloacetato) nickelato(II)

($P\bar{1}$) $Z = 1$, $R = 17\%$. The structure is built up from six-coordinate complex anions $[NiX_2]^{4-}$, K^+ cations and water molecules. The coordination polyhedron of nickel is a distorted tetragonal bipyramid with two nitrogens and four oxygens of the two nitriloacetate ions. $Ni-O_{eq}$, 2.12; $Ni-O_{ax}$, 2.06 and $Ni-N_{eq}$, 2.12(5) Å.

V.V. Fomenko, T.N. Polynova, M.A. Porai-Koshits and N.D. Mitrofanova, *J. Struct. Chem. (USSR)*, 13 (1972) 321.

Lithium triuranate, $Li_2U_3O_{10}$

The structure was partly determined from powder data. ($P2_1/c$) $Z = 4$. In contrast to most other polyuranate structures, it is not a layer-type structure. The basis is a three-dimensional uranium–oxygen network in which U–O–U–O–U groups can be distinguished.

L.M. Kovba, *J. Struct. Chem. (USSR)*, 13 (1972) 428.

Rubidium dioxotetrafluoromolybdate, $Rb_2[MoO_2F_4]$

($Amam$) $Z = 4$, $R = 13.5\%$ for 265 independent reflections. The complex $[MoO_2F_4]$ ion is tetragonal bipyramidal with fluorine atoms on the bipyramidal axis. The equatorial plane contains two oxygen and two fluorine atoms which are statistically distributed in the coordination positions. Mo–F, 2.00 and 2.02 Å.

V.S. Sergienko, M.A. Porai-Koshits and T.S. Khodashova, *J. Struct. Chem. (USSR)*, 13 (1972) 431.

Praesodymium nicotinate dihydrate

$(P2_1/a)$ $Z = 4$, $R = 9.2\%$ for 1700 independent reflections. The coordination polyhedron of praesodymium is a tetragonal antiprism capped on one of the tetragonal faces. The atoms in the coordination polyhedron are two water oxygens and seven carboxyl oxygens. One carboxyl is bidentate chelating, one is bidentate bridging and the third is tridentate, bridging and chelating. The complex is dimeric.

L.A. Aslanov, I.K. Abdul'minev and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 13 (1972) 437.

Di(π-cyclopentadienyl) niobium carbonyl hydride, $(\pi\text{-}C_5H_5)_2Nb(CO)H$

$(P\bar{1})$ or $(P1)$ $Z = 2$, $R = 16.9\%$ for 1200 non-zero reflections. The structure is made up of discrete molecules. The Nb atom is coordinated with two π-cyclopentadienyl ligands and the carbonyl group. The position of the hydride was located in the bisector plane of the wedge-shaped sandwich; Nb—H, 1.5 Å.

N.I. Kirillova, A.I. Gusev and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 441.

Aluminium borohydride

X-ray patterns of $Al(BH_4)_3$ were obtained at low temperature between $-60°$ and $-150°C$. These patterns attest to the existence of two crystalline modifications. Crystallochemical analysis and the IR spectrum are discussed.

K.N. Semenenko, O.V. Kravchenko and E.B. Lobkovskii, *J. Struct. Chem. (USSR)*, 13 (1972) 509.

Praesodymium thiocyanate heptahydrate, $Pr(NCS)_3.7H_2O$

$(Pna2_1)$ $Z = 4$. The metal coordination number is nine and the polyhedron is a distorted tetragonal antiprism capped by a water oxygen atom which is located above the midpoint of one of the tetragonal faces. Six positions are occupied by water molecules and three by thiocyanate nitrogens.

P.I. Lazarev, L.A. Aslanov, M.A. Porai-Koshits and V.I. Ivanov, *J. Struct. Chem. (USSR)*, 13 (1972) 511.

π-Cyclopentadienylazotoluenenickel, $(\pi\text{-}C_5H_5)Ni(CH_3C_6H_3N_2C_6H_4.CH_3)$

$(P\bar{1})$ $Z = 2$, $R = 15\%$ for 897 independent reflections. The azotoluene ligand coordinates the metal atom by an Ni—Co σ-bond and Ni—N bond resulting from the unshared pair of the more distant nitrogen atom. Ni—C(σ), 1.92 Å.

V.A. Semion, I.V. Barinov, Yu.A. Ustynyuk and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 13 (1972) 512.

Two forms of *trans* square planar complex of tellurium dimethane-thiosulphonate with ethylenethiourea, $Te(etu)_2(S_2OCH_3)_2$

$(P\bar{1})$ $Z = 1$, $R = 9.1\%$ for 912 independent reflections (A) and $(P2_1/c)$ $Z = 2$, $R = 6.8\%$ for 800 independent reflections (B). The centrosymmetric TeS_4 coordination groups are very alike in the two dimorphs. Te—S(etu), 2.66(6) in (A) and 2.678(5) in (B). Te—S (thiosulphonate), 2.694(6) in (A) and 2.685(4) Å in (B). There are some small differences from one dimorph to another in the rotational positions of the ligand groups.

O. Foss, N. Lyssandtrae, K. Maartmann-Moe and M. Tysseland, *Acta Chem. Scand.*, 27 (1973) 218.

$Tl_2[Cu(SO_3)_2]$

($P1$) $Z = 1$, $R = 3.12\%$ for 554 independent reflections. The structure consists of trigonal pyramids of SO_3^{2-} and somewhat distorted CuO_6 octahedra. The six oxygen atoms around Cu belong to different SO_3^{2-} groups. Average distances are S–O, 1.54; O–O, 2.43 Å. ∠ O–S–O, 104.5°.

I. Hjerten and B. Nyberg, *Acta Chem. Scand.*, 27 (1973) 345.

Neutron diffraction refinement of telluric acid, $Te(OH)_6$(mon)

$R = 2.4\%$ for 591 observed reflections. The Te–O and O–H bonds have been determined with a precision of 0.001 and 0.003 Å respectively. The O–H bonds range from 0.977 to 0.990 Å with a mean value of 0.985 Å.

O. Lundquist and M.S. Lehmann, *Acta Chem. Scand.*, 27 (1973) 85.

Nb_8P_5

($Pbam$) $Z = 4$, $R = 11.9\%$ for 399 observed reflections. The structure can be described as an array of interconnected Nb_6P triangular prisms with additional niobium atoms inserted between the prisms.

S. Anugual, C. Pontchow and S. Rundquist, *Acta Chem. Scand.*, 27 (1973) 27.

ℓ-Tris(1,10-phenanthroline)iron(II) bis(antimony(III)*d*-tartrate)octahydrate, $Fe(C_{12}N_2H_8)_3(C_4O_6H_2Sb)_2$

($P3_22_1$) $Z = 3$, $R = 4.8\%$ for 1600 reflections. The structure consists of a ferrous tris (1,10-phenanthroline) cation and a bis(antimonous *d*-tartrate) anion. The iron atom is octahedrally coordinated to the nitrogen atoms of three phenanthroline groups at an average distance of 1.97(1) Å. The antimony tartrate complex is a dimer in which each of the two antimony atoms is coordinated to four oxygen atoms of the tartrate.

A. Zalkin, D.H. Templeton and T. Ueki, *Inorg. Chem.*, 12 (1973) 1641.

μ-(Sulphur dioxide)-bis(π-cyclopentadienyldicarbonyliron), $[\pi\text{-}C_5H_5Fe(CO)_2]_2SO_2$

($P2_1/c$) $Z = 4$, $R = 3.01\%$ for 3472 independent reflections. All atoms including hydrogens were located. The SO_2 moiety is inserted symmetrically between two iron atoms with Fe(1)–S, 2.2790(6) and Fe(2)–S, 2.2814(7) Å; S–O, 1.4797(15) and 1.4764(14) Å.

M.R. Churchill, B.G. DeBoer and K.L. Kalra, *Inorg. Chem.*, 12 (1973) 1646.

cis-μ-Carbonyl-μ-(sulphur dioxide)-bis(π-cyclopentadienylcarbonyliron), *cis*-$(\pi\text{-}C_5H_5)_2Fe_2(CO)_3(SO_2)$

($Pbca$) $Z = 16$, $R = 5.18\%$ for 3975 independent reflections. The two independent molecules have essentially identical conformations, each consisting of two $(\pi\text{-}C_5H_5)Fe(CO)$ units linked by a direct iron–iron bond which is bridged symmetrically by a carbonyl and a sulphur dioxide ligand.

M.R. Churchill and K.L. Kalra, *Inorg. Chem.*, 12 (1973) 1650.

trans-Bromobis(methyldiphenylphosphine)(σ-pentafluorophenyl) nickel (II),
trans-$(PPh_2Me)_2Ni(O{-}C_6F_5)Br$

($C2/c$) $Z = 8, R = 6.16\%$ for 3459 independent reflections. The central nickel (II) atom is in a slightly distorted square planar coordination. Ni–PPh_2Me, 2.215(1) and 2.216(1); Ni–Br, 2.325(1); Ni–σ-C_6F_5, 1.880(4) Å.

M.R. Churchill, K.L. Kalra and M.V. Veidis, *Inorg. Chem.*, 12 (1973) 1656.

Tetramethylcyclobutadienetrifluoromethylbis(dimethylphenylphosphine) platinum (II) hexafluoroantimonate, $[(C_4(CH_3)_4)CF_3Pt(P(CH_3)_2C_6H_5)_2]SbF_6$

($P2_1/c$) $Z = 4, R = 4.9\%$ for 2866 reflections. Assuming tetramethylcyclobutadiene to be a monodentate ligand, the angles P–Pt–P, 95.2(1)° and P–Pt–C, 98.7(5) and 93.0(5)° would indicate a distorted tetrahedral geometry about the platinum atom. The cyclobutadiene ring is square planar with mean C–C of 1.47(2) Å. Sb–F, 1.83 Å.

D.B. Crump and N.C. Payne, *Inorg. Chem.*, 12 (1973) 1663.

Trigonal bipyramidal (A) and square pyramidal five-coordinate cobalt (II) complexes. Chlorobis [1,2-bis(diphenylphosphino) ethane] cobalt(II) trichlorostannate (II), red and green isomers, $[Co(dpe)_2Cl]SnCl_3$

($P2_1/c$) $Z = 4, R = 6.5\%$ for 8407 independent reflections (red complex); ($P\bar{1}$) $Z = 2, R = 10.1\%$ for 5868 independent reflections (green complex). The structures both consist of discrete five-coordinate cations and pyramidal $SnCl_3^-$ anions. The red complex is a tetragonal pyramid with an apical Cl and four P atoms in the basal plane. The green complex is trigonal bipyramidal with two P atoms at the axial positions and two P atoms and one Cl atom axially. Co–Cl, 2.398(2); Co–P, 2.276 (red form) and Co–Cl, 2.251(5); Co–P, 2.258 Å (green form). The $SnCl_3^-$ anion is pyramidal in both complexes.

J.K. Stalich, P.W.R. Corfield and D.W. Meek, *Inorg. Chem.*, 12 (1973) 1668.

Trichloro (*p*-tolylazo) bis(triphenylphosphine) ruthenium (II) – acetone, $RuCl_3(p\text{-}N_2C_6H_4Me)(PPh_3)_2.MeCo$

($P2_1/c$) $Z = 4, R = 4.5\%$ for 2478 independent reflections. The structure of the complex is essentially octahedral with the two triphenylphosphine ligands in the *trans* position. The *p*-tolylazo group coordinates in a linear manner with an Ru–N distance of 1.796(9) Å and ∠Ru–N–N, 171.2°; N–N, 1.144(10); Ru–P, 2.434(4); Ru–Cl, 2.388(3); ∠N–N–C, 135.9(11)°.

J.V. McArdle, A.J. Schultz, B.J. Corden and R. Eisenberg, *Inorg. Chem.*, 12 (1973) 1676.

Dinitratobis [2-(2-diethylammonioethyl) pyridine] copper (II) nitrate, $[Cu(DEAEPH)_2(NO_3)_2](NO_3)_2$

($P\bar{1}$) $Z = 1, R = 3.7\%$ for 2948 reflections. The copper (II) ions are unsymmetrically coordinated to two bidentate nitrate ions with Cu–O, 1.992(2) and 2.562(3) Å and to the pyridine nitrogen atoms of two DEAEPH ligands with Cu–N, 1.984(2) Å. The amine nitrogen atoms of the ligand are protonated, which causes the ligands to be unidentate and positively charged.

D.L. Lewis, D.J. Hodgson, *Inorg. Chem.*, 12 (1973) 1682.

Bis(triphenylphosphine)–diborane(4), $B_2H_4.2P(C_6H_5)_3$

($P\bar{1}$) $Z = 1$, $R = 7.2\%$ for 798 reflections. The central $B_2H_4P_2$ part of the molecule has an ethane-like structure in the anti conformation. The B–B and P–B distances are 1.76(2) and 1.935(9); P–C, 1.83; C–C, 1.38 Å.

W. Van Doorne, A.W. Cordes and G.W. Hunt, *Inorg. Chem.*, 12 (1973) 1682.

$[Cl_2Sb\{Fe(CO)_2(h^5\text{-}C_5H_5)\}_2][Sb_2Cl_7]$

($P2_1/n$) $Z = 8$, $R = 5.6\%$ for 5097 observed reflections. In each cation two chlorine atoms and two iron atoms form a grossly distorted tetrahedron about antimony with average Sb–Fe 2.440 and Sb–Cl 2.42 Å. Each iron can be considered to have octahedral coordination. Sb–Cl, 2.354–2.955 Å and the anion can be considered to contain $SbCl_3$, $SbCl_4^-$ and $Sb_2Cl_7^-$.

F.W.B. Einstein and R.D.G. Jones, *Inorg. Chem.*, 12 (1973) 1690.

Ditellurium pentoxide, Te_2O_5

($P2_1$) $Z = 2$, $R = 4.0\%$ for 2205 independent reflections. The molecule contains octahedrally coordinated tellurium(VI) and four-coordinated tellurium(IV) atoms $Te^{(VI)}$–O are 1.84 to 1.972 and $Te^{(IV)}$–O are 1.892–2.080 Å.

O. Lindquist and J. Moret, *Acta Crystallogr., Sect. B*, 29 (1973) 643.

Magnesium hexahydrate, $MgSO_3.6H_2O$

($R3$) $Z = 5.9\%$ for 213 unique reflections. The structure consists of a slightly distorted CsCl arrangement of octahedral $[Mg(H_2O)_6]^{2+}$ and pyramidal SO_3^{2-} ions.

H. Flack, *Acta Crystallogr. Sect. B*, 29 (1973) 656.

Magnesium picolinate dihydrate, $Mg(C_5H_4NCOO)_2.2H_2O$

($P2_1/c$) $Z = 4$, $R = 11.6\%$ for 1420 observed reflections. The molecule is dihedral with an angle of about 95° between the two pyridine rings. The Mg atom is coordinated by an unsymmetrical octahedral arrangement of the two N atoms, two chelating carboxylic oxygens and the two molecules of water.

J.-P. Deloume, H. Loiselew and G. Thomas, *Acta Crystallogr., Sect. B*, 29 (1973) 668.

Rubidium ammonium hydrogen fluorocitrate dihydrate, $RbNH_4H(C_6H_4O_7F).2H_2O$

($P\bar{1}$) $Z = 2$, $R = 7.2\%$ for 1799 independent reflections. The fluorocitrate ion forms a tridentate chelate with a rubidium ion, the fluorine atoms being shared by two rubidium coordination polyhedra.

H.L. Carrell and J.P. Glusker, *Acta Crystallogr., Sect. B*, 29 (1973) 674.

Lithium formate monohydrate, $LiHCOO.H_2O$

($Pbn2_1$) $Z = 4$, $R = 4.9\%$ for 1027 reflections. The lithium ion is tetrahedrally coordinated with three formate oxygens and one water oxygen. Li–O, 1.923–1.974 Å. The formate group is probably planar.

A. Enders-Beumer and S. Harkema, *Acta Crystallogr., Sect. B*, 29 (1973) 682.

Hexakis(2-methylimidazole) cadmium(II) tetrafluoroborate, $Cd(2MIz)_6(BF_4)_2$
($R\bar{3}m$) $Z = 1$, $R = 4.1\%$ for 876 reflections. The cadmium ion is octahedrally coordinated to the six imine nitrogen atoms of the ligands, with Cd—N 2.413 Å. The octahedron is slightly compressed in the direction of the trigonal axis.
J. Reedijk and G.C. Verschoor, *Acta Crystallogr., Sect. B,* 29 (1973) 721.

(1—3, 3*a*, 8*a*—*η*) (5, 7-Dimethyl-4*H*-cyclohepta[*c*]thiophene)tricarbonyl chromium
($P2_1/c$) $Z = 4$, $R = 5.9\%$ for 552 reflections. The tricarbonyl chromium group is fixed on the thiophene ring. The conformation of the latter is different from that in the free ligand and the tropiline ring has a "boat" conformation.
Y. Dusausoy, J. Protas and R. Guilard, *Acta Crystallogr., Sect. B,* 29 (1973) 726.

$(+)_{589}$-*cis*-β-Dinitro-(*R*-5-methyltriethylenetetramine)cobalt(III)chloride,
$(+)_{589}$-*cis*-β[$Co(NO_2)_2$(*R*-5-metrien)] Cl
($P2_12_12_1$) $Z = 4$, $R = 6.9\%$ for 1567 observed reflections. The quadridentate ligand is linked to the central cobalt atom with its four nitrogen atoms in *cis*-β coordination. A substituted methyl group lies in an equatorial position relative to the plane of the chelate ring. It is attached to the carbon atom next to the secondary nitrogen atom common to the two chelate rings both in the equatorial plane of the octahedron.
K. Tanaha, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B,* 29 (1973) 733.

$(-)_{589}$-*μ*-*facial*-Bis(diethylenetriamine)cobalt(III) hexacyanocobaltate(III) dihydrate,
$(-)_{589}$-*μ*-*facial*-[Co(dien)$_2$] [$Co(CN)_6$] .$2H_2O$
($B22_12$) $Z = 8$, $R = 3.5\%$ for 2510 reflections. There are two isomeric complex cations in the asymmetric unit. They both have a two-fold axis and the absolute configuration can be designated as skew chelate pairs ΔΔΔ. The conformations of the chelate rings formed by a dien molecule in one complex ion are δλ, while those in the other are λλ.
M. Konno, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B,* 29 (1973) 739.

$CsMnCl_3$
($R3m$) $Z = 9$, $R = 11.6\%$ for 687 reflections. The *c* parameter accommodates nine close-packed layers of composition $CsCl_3$ and the Mn ions are octahedrally coordinated by Cl ions. The structure is closely related to that of $CsNiCl_3$.
J. Goodyear and D.J. Kennedy, *Acta Crystallogr., Sect. B,* 29 (1973) 744.

Trigonal tris(diphenyltriazine)cobalt(III)
($P\bar{3}$) $Z = 6$, $R = 7.8\%$ for 4197 reflections. The refined structure confirms those features indicative of trigonal distortion, which appear not to be unique to the diphenyltriazine complex, however, but may be typical of octahedral complexes formed from bidentate ligands having a short bite. Co—N(average), 1.92(1) Å.
W.R. Krigbaum and B. Rubin, *Acta Crystallogr., Sect. B,* 29 (1973) 749.

Dichloro-(*S*-methyl-α-crysteine)palladium(II) monohydrate, $[Pd(SmCH)Cl_2]H_2O$ when $SmCH = CH_3SCH_2CH(NH_2)COOH$

($P2_12_12_1$) $Z = 8$, $R = 4.9\%$ for 1763 independent reflections. There are two crystallographically independent molecules with the same square planar coordination involving sulphur and nitrogen from the amino acid ligand and two chlorine atoms. The coordination five-membered ring has λ conformation.

L.P. Battaglia, A. B-Corradi, C.G. Palmieri, M. Nardelli and M.E. V-Tani, *Acta Crystallogr., Sect. B*, 29 (1973) 762.

CrP_2 and $CrAs_2$

($C2/m$) $Z = 4$, $R = 3.0\%$ for 253 reflections. $CrAs_2$ was found from X-ray powder data to have the same structure as CrP_2. Two families of transition metal dipnictides can be distinguished, one having structural characteristics of semiconductors.

W. Jeitschko and P.C. Donahue, *Acta Crystallogr., Sect. B*, 29 (1973) 783.

Twinned $Cd_5(PO_4)_3Cl$, "cadmium chlorapatite"

($P6_3/m$) $Z = 2$, $R = 5.6\%$ and 5.5% for the two crystals in the twin, for 1840 reflections. The material is isostructural with fluorapatite. The side length of the Cd^{II} triangle is 4.41 Å compared with 3.97 Å in the fluorapatite.

K. Sudarsanan, R.A. Young and J.D.H. Donnay, *Acta Crystallogr., Sect. B*, 29 (1973) 808.

La_2GeS_5

($P2_1/a$) $Z = 4$, $R \approx 5.3\%$ for 745 reflections. The structure is made up of two kinds of sheet parallel to the plane $x-z$; alternately a sheet of $[GeS_4]_n$ tetrahedra and a sheet of $[LaS]_{2n}$.

A. Mazurier and J. Etienne, *Acta Crystallogr., Sect. B*, 29 (1973) 817.

Hexamminecobalt(III) hexacyanocobaltate(III)

($R\bar{3}$) $Z = 1$, $R \approx 2.8\%$ for 2205 independent reflections. Both complex ions possess the rigorous crystallographic symmetry $\bar{3}$ and the packing is much the same as in caesium chloride. The coordination around the metal atoms is regular octahedral; Co–N, 1.972(1); Co–C 1.894(1) Å; C–N, 1.157(2) Å. The direct integration of electron density around the central cobalt atom within a sphere of radius 1.22 Å shows that each metal atom has a largely neutralized resultant charge; $[Co(NH_3)_6]^{3+}$, $26.3 \pm 0.3e$ and $[Co(CN)_6]^{3-}$ $26.8 \pm 3e$.

M. Iwata and Y. Saito, *Acta Crystallogr., Sect. B*, 29 (1973) 822.

Barium diferrite, $BaO.2Fe_2O_3$

($P6_3/m$) $Z = 2$, $R = 8\%$ for 111 independent reflections. Ferric ions are distributed equally into two different sites; one surrounded tetrahedrally and the other octahedrally by oxygen ions.

S. Okamoto, S.I. Okamoto and T. Ito, *Acta Crystallogr., Sect. B*, 29 (1973) 832.

A refinement of arsenstruvite, $MgNH_4AsO_4.6H_2O$

$(Pmn2)$ $Z = 2, R = 3.0\%$ for 1110 reflections. The crystal structure, built up from Mg octahedra and ammonium groups sandwiched between As tetrahedra is discussed in the light of bond length and bond strength correlations. The hydrogen atoms were located.

G. Ferraris and M. Franchini-Angela, *Acta Crystallogr., Sect. B*, 29 (1973) 859.

Caesium oxobisoxalatobisaquoniobate(V)dihydrate, $Cs[NbO(C_2O_4)_2(H_2O)_2].2H_2O$

$(P2_1/n)$ $Z = 2, R = 4.6\%$ for 2411 independent reflections. The coordination polyhedron around niobium is a pentagonal bipyramid. The ligands in the equatorial plane are two oxygen atoms from an oxalato group at 2.169 Å. The apices of the bipyramid are occupied by the double-bonded oxygen atom and one water molecule at 1.691 and 2.328 Å respectively.

B. Kojić-Prodić, R. Liminga and S. Šcavničar, *Acta Crystallogr., Sect. B*, 29 (1973) 864.

$HgMoO_4$ and related compounds

$(C2/c)$ $Z = 4, R = 2.3\%$ for 849 reflections. The Hg atom is coordinated by two oxygen atoms, forming a linear O–Hg–O arrangement; Hg–O, 2.03 Å. Four more oxygens are further away at 2.67 Å (2X) and 2.77 Å (2X), completing an approximately octahedral grouping. The oxygen environment of the Mo is essentially octahedral with Mo–O distances between 1.72 and 2.23 Å, indicating bond orders of one to two respectively. $HgWO_4$ has the same structure.

W. Jeitschko and A.W. Sleight, *Acta Crystallogr., Sect. B*, 29 (1973) 869.

Trichloroadeniniumzinc(II), $C_5H_6N_5Cl_3Zn$

$(P2_1/c)$ $Z = 4, R = 3.5\%$ for 1831 independent reflections. The zinc atom is coordinated to the three chlorine atoms and to N(7) of the adenine moiety in a distorted tetrahedral arrangement. This indicates that N(7) is a possible coordination site for bivalent metal ion interaction with nuclei acids. The six-membered ring of the adenine moiety, which is protonated at N(1), is significantly non-planar.

M.R. Taylor, *Acta Crystallogr., Sect. B*, 29 (1973) 884.

α-$Na_2Cr_2O_7$ and the $\alpha-\beta$ phase transition

$(A\bar{1})$ $Z = 4, R = 5.4\%$ for 714 non-equivalent reflections. The dichromate geometry is normal with $\angle$Cr–O–Cr, 135°. The thermal vibrations of the dichromate ion indicate that it is undergoing large antisymmetric torsional vibrations which are probably associated with the soft mode that causes the transition.

N. Ch. Panagiotopoulos and I.D. Brown, *Acta Crystallogr., Sect. B*, 29 (1973) 890.

catena-μ-Dichlorobisimidazolecadmium(II), $[Cd(ImH)_2Cl_2]_\infty$

$(P2_1/c)$ $Z = 2, R = 3.9\%$ for 2032 independent reflections. The cadmium atoms lie in positions of symmetry $\bar{1}$ and are octahedrally coordinated. They are linked into infinite chains by double chlorine bridges. Each cadmium forms, in addition to four Cd–Cl bonds (2.706(2), 2.73(2) Å), two Cd–N (imidazole) bonds (2.248(3) Å) which are almost perpendicular to the plane of the Cd and Cl atoms.

R.J. Flock, H.C. Freeman, F. Aug and J.M. Rosalky, *Acta Crystallogr., Sect. B*, 29 (1973) 903.