

X-RAY BIBLIOGRAPHY

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Bis(cyclopentadienyl)methylnitrosylmolybdenum

($P2_1/c$), $Z=4$, $R=6.8\%$ for 2475 independent reflections. The structures of the two independent, approximate mirror image molecules are very similar to that of tricyclopentadienylmethylnitrosylmolybdenum. The Mo-N-O systems are linear, $178(1)$ and $178(2)^\circ$, with short Mo-N distances $1.75(1)$ and $1.76(1)$ Å. N-O distances are elongated: $1.23(1)$ and $1.21(1)$. The relationship of the structure to the FMR spectrum of $(C_5H_5)_3MoNO$ is discussed.

F.A. Cotton and G.A. Rusholme, *J. Amer. Chem. Soc.*, 94 (1972) 402.

Bis(dimethylgloximate)diimidazoleiron(II)-dimethanol, $Im_2Fe(DMG)_2 \cdot 2CH_3OH$

($P2_1/c$), $Z=2$, $R=5.2\%$ for 1061 unique reflections. The iron is in a tetragonally distorted octahedral environment. O-O of adjacent DMG's is $2.588(8)$ Å. There is hydrogen bonding between each pair of oxygen atoms and the two O-H distances are $1.0(1)$ and $1.5(1)$ Å. N-O = $1.408(7)$ and $1.365(7)$ Å; Fe-N(eq) = $1.893(6)$, $1.918(6)$ Å; Fe-N(ax) = $1.985(5)$ Å.

K. Bowman, A.P. Gaughan and Z. Dori, *J. Amer. Chem. Soc.*, 94 (1972) 727.

cis-Dihydridotetrakis(diethyl phenylphosphonite)iron(II), $H_2Fe[(C_6H_5)P(OC_2H_5)_2]_4$

($P\bar{1}$) $Z=2$, $R=4.9\%$ for 3659 reflections. The iron-phosphorus geometry is intermediate between octahedral and tetrahedral and each molecule has *cis* hydrogens. Fe-H = $1.51(4)$; Fe-P(*cis* to H) = $2.128(2)$ and (*trans* to H) = $2.150(2)$ Å.

L.J. Guggenberger, D.D. Titus, M.T. Flood, R.E. Marsh, A.A. Orio and H.B. Gray, *J. Amer. Chem. Soc.*, 94 (1972) 1135.

Hydrated lanthanide nicotines, $La_2(C_5H_4NCO_2)_6(H_2O)_4$ (A) and $Sm_2(C_5H_4NCO_2)_6(H_2O)_4$ (B)

($P2_1/c$) $Z=2$; $R=2.1\%$ for 2388 reflections (A) and ($P2_1/c$) $Z=2$; $R=2.5\%$ for 2398 reflections (B). The complexes are dimeric and the eight-coordinate metal ions are bridged by four carbonyl groups, the remaining two carbonyl groups being chelated to the metals. The nicotinate nitrogens are all hydrogen bonded to water molecules of adjacent

dimers. The magnetic moments are discussed.

J.W. Moore, M.D. Glick and W.A. Baker, Jr., *J. Amer. Chem. Soc.*, 94 (1972) 1858.

A nitrogen molybdenum chelate, $(\pi\text{-C}_5\text{H}_5(\text{CO})_2\text{MoN.N}(\text{CH}_3)_3\text{C}(\text{CO}_2\text{C}_2\text{H}_5)\text{COH})\text{PF}_6$
($P\bar{1}$) $Z=2$, $R=13.8\%$ for 2090 independent reflections. In the cation a planar five-atom ring is joined to molybdenum by a carbene bond $\text{Mo}=\text{C}$, 2.03 Å. The structure can only be written as a non-classical ion analogous to that derived from a sydnone.

C.K. Prout, T.S. Cameron and A.R. Gent, *Acta Crystallogr., Sect. B*, 28 (1972) 32.

The hexahydrate of bis-salicylidene-triethylenetetramine nickel(II)

($P4/ncc$) $Z=8$, $R=10.1\%$ for 1425 observed reflections. The nickel atom is coordinated octahedrally to the hexadentate ligand. The water molecules form columns by sharing faces between decahedral units.

P.D. Cradwick, M.E. Cradwick, G.G. Dodson, D. Hall and T.N. Waters, *Acta Crystallogr., Sect. B*, 28 (1972) 45.

Calcium barbitol trihydrate, $\text{Ca}(\text{C}_8\text{N}_2\text{O}_3\text{H}_{11})_2 \cdot 3\text{H}_2\text{O}$

($P\bar{1}$) $Z=4$, $R=4.6\%$ for 8237 reflections. The two calciums are seven-fold coordinated by six oxygens and one nitrogen at the corners of irregular pentagonal bipyramids. Deprotonation of the four crystallographically independent pyrimidine rings causes changes in bond lengths and internal bond angles.

B. Berking, *Acta Crystallogr., Sect. B*, 28 (1972) 98.

Hydrogen triuranate, $\text{H}_2\text{U}_3\text{O}_{10}$

($P\bar{1}$) $Z=1$, $R=6.0\%$ for 756 independent reflections. The configuration of oxygen atoms about one uranium is octahedral whilst at the other it is pentagonal-bipyramidal.

S. Siegel, A. Viste, H.R. Hoekstra and B. Tani, *Acta Crystallogr. Sect. B*, 28 (1972) 117.

Dimeric dibromobis(pyridine *N*-oxide) copper(II), $[(\text{pyridine } N\text{-oxide})_2\text{CuBr}]_2$

($P\bar{1}$) $Z=2$, $R=4.6\%$ for 4415 observed reflections. In the two dimeric molecules each copper atom is coordinated by two bromine atoms and by three oxygen atoms, of which two are bridging. The geometry of this complex differs markedly from other complexes of the aromatic *N*-oxide copper(II) halide series.

A.D. Mighell, C.W. Reimann and A. Santoro, *Acta Crystallogr., Sect. B*, 28 (1972) 126.

Calcium fumarate trihydrate, $\text{CaC}_4\text{H}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$

($Pna2_1$) $Z=4$, $R=9.5\%$ for 424 non-zero reflections. Chains of fumarate ions lie parallel to the *b* axis. The structure is held together by ionic $\text{Ca}\cdots\text{O}$ linkages with eight-fold coordination round Ca (2.45 Å average).

M.P. Gupta, S.M. Prasad, R.G. Sahu and B.N. Sahu, *Acta Crystallogr., Sect. B*, 28 (1972) 135.

An antimony chloride-phenanthrene molecular complex, $2\text{SbCl}_3(\text{phen})$

($P\bar{1}$) $Z=2$, $R=3.8\%$ for 5097 independent reflections. The coordination around antimony is a distorted trigonal bipyramid. Two independent Sb atoms are on opposite sides of the phenanthrene plane (3.27 and 2.94 Å).

A. Demaldé, A. Mangia, M. Nardelli, G. Pelizzi and M.E. Vidoni Tani, *Acta Crystallogr., Sect. B*, 28 (1972) 147.

 $\text{ZnCl}_2 \cdot \text{C}_6\text{H}_{14}\text{O}_3\text{N}$ (where $\text{C}_6\text{H}_{14}\text{O}_3\text{N}$ is triethanolamine)

($P\bar{1}$) $Z=2$, $R=5.2\%$ for 438 reflections. The complex contains a *trans*-annular donor-acceptor bond between zinc and nitrogen.

H. Föllner, *Acta Crystallogr., Sect. B*, 28 (1972) 157.

The dimethyldicyano compounds of silicon, germanium, tin and lead

($Pnma$) $Z=4$, Si and Ge compounds; ($Fmm2$) $Z=4$, Sn and Pb compounds. In the Si and Ge compounds, approximately tetrahedral molecules form linear chains through weak N-M interactions. In the Sn and Pb compounds the interactions are stronger and planar sheets are formed. Each tetrahedral molecule is octahedrally distorted.

J. Konnert, D. Britton and Y.M. Chow, *Acta Crystallogr., Sect. B*, 28 (1972) 180.

 $\text{Ti}(\text{urea})_6(\text{ClO}_4)_3$

($R\bar{3}c$) $Z=6$, $R=12.2\%$ for 642 independent reflections. The geometry of the $\text{Ti}(\text{urea})_6^{3+}$ ion is formally octahedral with superimposed distortion which involves a twist about the three-fold axis.

B.N. Figgis, L.G.B. Wadley and J. Graham, *Acta Crystallogr., Sect. B*, 28 (1972) 187.

Copper tetrametaphosphate, $\text{Cu}_2\text{P}_4\text{O}_{12}$

($C2/c$) $Z=4$, $R=5.8\%$ for 389 reflections.

M. Läügi, J.C. Guitel, I. Tordjman and G. Bassi, *Acta Crystallogr., Sect. B*, 28 (1972) 201.

Strontium dipicolinate tetrahydrate, $\text{Sr} \cdot \text{C}_7\text{H}_3\text{NO}_4 \cdot 4\text{H}_2\text{O}$

($C2/c$) $Z=4$, $R=4.4\%$ for 1135 independent reflections. The dipicolinate ion is tridentate and the strontium is coordinated to eight oxygen atoms and one nitrogen atom. Two oxygens are from carbonyl groups of the same dipicolinate ion; the four others are from water molecules.

K.J. Palmer, R.Y. Wong and J.C. Lewis, *Acta Crystallogr., Sect. B*, 28 (1972) 223.

Copper(II) violurate tetrahydrate, $(\text{C}_4\text{O}_4\text{N}_3\text{H}_2)_2\text{Cu} \cdot 4\text{H}_2\text{O}$

($P\bar{1}$) $Z=1$. The copper coordination is distorted octahedral with two waters ($\text{Cu}-\text{O} = 2.06\text{Å}$) and two oxime nitrogens (1.97Å) in a square plane and two ketonic oxygens

(2.21 Å).

M. Hamelin, *Acta Crystallogr., Sect. B*, 28 (1972) 228.

A symmetrical 12-oxa [4.4.3] propella-2, 4, 7, 9-tetraene bis-irontricarbonyl, $C_{18}H_{12}O_7Fe_2$ ($C2/c$) $Z=8$, $R=4.5\%$ for 1323 observed reflections. Each iron atom is complexed by a butadiene group of four planar carbon atoms of the ligand with C—C 1.413(6), 1.401(6) and 1.422(6) Å respectively.

K. Bjamer Birnbaum, *Acta Crystallogr., Sect. B*, 28 (1972) 161.

Ferric ammonium sulphate trihydrate, $FeNH_4(SO_4)_2 \cdot 3H_2O$

($P2_1/c$) $Z=4$, $R=3.8\%$ for 1504 reflections. The iron is octahedrally coordinated to four sulphate oxygen atoms and two water oxygens. A third water is held in the structure by hydrogen bonds.

K.J. Palmer, R.Y. Wong and K.S. Lee, *Acta Crystallogr., Sect. B*, 28 (1972) 236.

Anhydrous copper butyrate, $[CH_3(CH_2)_2COO]_2Cu$

($P\bar{1}$) $Z=2$, $R=9.2\%$ for 832 independent reflections. Two of the hydrocarbon chains in each dimer are in the usual zig-zag conformation, but the other two are deformed by rotations about single bonds. Cu—Cu=2.565 Å.

M.J. Bird and T.R. Lomer, *Acta Crystallogr., Sect. B*, 28 (1972) 242.

Trimethyltin nitrate monohydrate $(CH_3)_3SnNO_3 \cdot H_2O$

($C2/c$) $Z=4$, $R=3.9\%$ for 463 observed reflections. The tin atom is in a distorted trigonal-bipyramidal environment consisting of a trimethyltin fragment with a unidentate nitrate group and a water occupying the apical positions in a disordered manner.

Sn—O=2.22(3) to nitrate and 2.47(2) Å to water. Sn—C(mean) is 2.11(2) Å.

R.E. Drew and F.W.B. Einstein, *Acta Crystallogr., Sect. B*, 28 (1972) 345.

K_2PtCl_4 and K_2PdCl_4

($P4/mmm$) $Z=1$, $R=1.8\%$ for 2434 observations (K_2PtCl_4) and ($P4/mmm$) $Z=1$, $R=4.1\%$ for 191 independent reflections (K_2PdCl_4). Pt—Cl = 2.316 Å; Pd—Cl = 2.318 Å.

R.H.B. Mais, P.G. Owston and A.M. Wood, *Acta Crystallogr., Sect. B*, 28 (1972) 393.

Triclinic hexakis(thiourea)lead(II) perchlorate, $Pb(tu)_6(ClO_4)_2$

($P\bar{1}$) $Z=1$, $R=14.2\%$ for 1752 reflections. The lead atom is surrounded by a slightly distorted octahedron of sulphur atoms. Pb—S = 3.06 ± 0.01 Å. S—Pb—S angles are $79-100^\circ$.

I. Goldberg and F.H. Herbstein, *Acta Crystallogr., Sect. B*, 28 (1972) 400.

Tetrakis(thiourea)lead(II) picrate, $\text{Pb}(\text{tu})_4(\text{picrate})_2$

(*Cc* or *C2/c*) $Z=4$, $R=12.4\%$ for 1842 reflections. The crystal contains polymeric chains of edge-sharing distorted $\text{Pb}(\text{tu})_6$ octahedra and picrate ions. $\text{Pb}-\text{S}(\text{shared}) = 2.87$ and 3.23 ; $\text{Pb}-\text{S}(\text{unshared}) = 2.96 \text{ \AA}$.

F.H. Herbststein and M. Kaftory, *Acta Crystallogr., Sect. B*, 28 (1972) 405.

 $3/4 \text{ Pb}(\text{HCOO})_2 \cdot 4$ thiourea

(*I422*) $Z=8$, $R=10.2\%$ for 470 reflections. The lead is eight-coordinate and lead ions lie along the *c* axis surrounded by twisted tetragonal prisms of sulphur atoms. Every fourth lead ion position is vacant.

I. Goldberg and F.H. Herbststein, *Acta Crystallogr., Sect. B*, 28 (1972) 410.

Di- μ -thio-*n*-butyl(bis- π -cyclopentadienylmolybdenum)iron dichloride, a model compound of the nitrogenase system

(*P2₁/b*) $Z=4$, $R=7.0\%$ for 1835 independent reflections. Fe...Mo in the bent sulphur bridge is 3.660 \AA and hence there is no metal-metal bond. The FeS_2 and MoS_2 planes are inclined at 148° . $\text{Mo}-\text{S}(\text{mean}) = 2.464$; $\text{Fe}-\text{S}(\text{mean}) = 2.386 \text{ \AA}$. The iron is approximately tetrahedral.

T.S. Cameron and C.K. Prout, *Acta Crystallogr., Sect. B*, 28 (1972) 453.

(-)₅₈₉-*cis*- α -Dinitro-(1,3,8-dimethyltriethylenetetramine)-cobalt(III) perchlorate, *cis*- α -[Co(NO₂)₂(1,3,8-dimetricien)]ClO₄

(*P2₁2₁2₁*) $Z=4$, $R=5.6\%$ for 2268 observed reflections. Six nitrogen atoms of the dimetricien ligand and nitro groups are bonded nearly octahedrally to the central cobalt atom. $\text{Co}-\text{N}(\text{dimetricien}) = 1.958(7) \text{ \AA}$ and $\text{NCoN} = 86.1(3)^\circ$. Absolute complex configuration is Λ and the chelate rings are δ , λ and δ . The two methyl groups are equatorial with respect to the chelate rings.

M. Ito, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 457.

(-)₅₈₉-*trans*-Dinitro-(1,3,8-dimethyltriethylenetetramine)-cobalt(III) perchlorate, *trans*-[Co(NO₂)₂(1,3,8-dimetricien)]ClO₄

(*P2₁2₁2₁*) $Z=4$, $R=8.8\%$ for 888 observed reflections. $\text{Co}-\text{N}(\text{dimetricien}) = 1.98 \text{ \AA}$; $\text{Co}-\text{N}(\text{NO}_2) = 1.96 \text{ \AA}$. Conformations of the chelate rings are δ , λ , δ .

M. Ito, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 463.

s-facial-Bis(diethylenetriamine)cobalt(III) bromide, *s*-facial-[Co(dien)₂]Br₃

(*C2/c*) $Z=4$, $R=7.4\%$ for 1021 observed reflections. Each dien molecule acts as a tridentate ligand with terminal nitrogen atoms *cis*. The conformations of the two independent chelate rings are enantiomeric with each other.

M. Kobayashi, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 470.

Lead(II) diethyldithiocarbamate

(*Cc*) $Z=4$, $R=7.9\%$ for 1491 independent reflections. Two carbamate ions are coordinated to the central metal atom through sulphur atoms, forming a monomeric complex $[\text{Pb}(\text{S}_2\text{CNet})_2]$. One ligand is closer to the lead atom than the other by 0.05 Å. The coordination sphere is distorted pyramidal.

H. Iwasaki and H. Hagihara, *Acta Crystallogr., Sect. B*, 28 (1972) 507.

The four-layer orthorhombic polytype of potassium hexacyanocobaltate(III), $\text{K}_3\text{Co}(\text{CN})_6$ (*Pnc2*) $Z=8$, $R=9.0\%$ for 854 intensities.

E.C. Reynhardt and J.C.A. Boeyens, *Acta Crystallogr., Sect. B*, 28 (1972) 524.

 $\text{Cr}(\text{NH}_3)_6\text{MnF}_6$ (A) and $\text{Cr}(\text{NH}_3)_6\text{FeF}_6$ (B)

(*Pa3*) $Z=4$, $R=3.0\%$ for 294 independent reflections (A) and (*Pa3*) $Z=4$, $R=3.4\%$ for 648 independent reflections (B). A dynamic Jahn–Teller effect for the MnF_6^{3-} ion is discussed.

K. Wieghardt and J. Weiss, *Acta Crystallogr., Sect. B*, 28 (1972) 529.

Bis(dimethyl-*o*-thiophenylarsine)palladium(II)

(*Cc* or *C2/c*) $Z=4$, $R=10.5\%$ for 1722 independent intensities. The crystal contains pyridine of crystallization; $\text{PdAs}_2\text{S}_2\text{C}_{16}\text{H}_{20}\cdot\text{C}_5\text{H}_5\text{N}$. Thiol functions as a bidentate ligand to the *trans* square palladium(II).

J.P. Beale and N.C. Stevenson, *Acta Crystallogr., Sect. B*, 28 (1972) 557.

Dichlorobis(antipyrine)zinc, $\text{Zn}(\text{C}_{11}\text{H}_{12}\text{ON}_2)_2\text{Cl}_2$

(*P2₁/c*) $Z=4$, $R=8.3\%$ for 3239 independent reflections. Two oxygen atoms from two antipyrine molecules and two chlorine ions make up a tetrahedral coordination sphere around the zinc atom. The pyrazolone and benzene rings are planar and there is no indication of conjugation between the rings. The cobalt compound is isostructural with the zinc compound.

M.B. Cingi, C. Guastini, A. Musatti and M. Nardelli, *Acta Crystallogr., Sect. B*, 28 (1972) 667.

Strontium di-iodate(V) monohydrate, $\text{Sr}(\text{IO}_3)_2\cdot\text{H}_2\text{O}$

(*C2/c*) $Z=4$, $R=3.9\%$ for 1281 reflections. The structure consists of pyramidal IO_3^- anions ($\text{I}-\text{O}=1.786, 1.806$ and 1.825 Å), Sr^{2+} cations and water molecules.

A.M. Manotti Lanfredi, M.A. Pellinghelli, A. Tiripicchio and M. Tiripicchio Camellini, *Acta Crystallogr., Sect. B*, 28 (1972) 679.

 $\text{NaNH}_4\text{CrO}_4\cdot 2\text{H}_2\text{O}$ (A) and $\text{Mg}(\text{NH}_4)_2(\text{PO}_3\text{OH})_2\cdot 4\text{H}_2\text{O}$ (B)

(A) (*P2₁2₁2₁*) $Z=4$, $R=13.2\%$ for 642 observed reflections. The Na ions are coordinated octahedrally by four water molecules and two chromate oxygen atoms. The salt should

be formulated $[\text{Na}(\text{OH})_2\text{OCrO}_3][\text{NH}_4]$.

(B) (*Pbca*) $Z=8$, $R=7.3\%$ for 307 reflections. In the Mg octahedron $\text{Mg}(\text{OH}_2)_4(\text{OPO}_2\text{OH})_2$ two tetrahedral PO_3OH groups are *trans*. The ammonium ions in both structures are five-coordinated.

A.A. Khan and W.H. Baur, *Acta Crystallogr., Sect. B*, 28 (1972) 683.

$\text{CsTi}^{\text{III}}(\text{SO}_4)_2 \cdot 2\text{H}_2\text{O}$

(*C2/c*) $Z=4$, $R=6.5\%$ for 515 observed reflections. The structure consists of infinite chains of $[\text{Ti}^{\text{III}}(\text{SO}_4)_2(\text{H}_2\text{O})_2]^-$ parallel to $[101]$ and linked by Cs^+ cations. $\text{Ti}^{\text{III}}-\text{O} = 2.24 \text{ \AA}$.

J.-M. Manoli, P. Herpin and A. Dereigne, *Acta Crystallogr., Sect. B*, 28 (1972) 806.

Zinc pyridyl-2-acetate, $\text{Zn}(\text{C}_5\text{H}_4\text{N}-\text{CH}_2-\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$

(*P2₁/c*) $Z=2$, $R=8.8\%$ for 777 observed reflections. The zinc atom is octahedrally coordinated by two nitrogens, two carboxylic oxygens and two water oxygens. The chelate ring is non-planar.

R. Faure and H. Loiseleur, *Acta Crystallogr., Sect. B*, 28 (1972) 811.

Nickel picolinate tetrahydrate, $\text{Ni}(\text{C}_5\text{H}_4\text{NCOO})_2 \cdot 4\text{H}_2\text{O}$

(*P2₁/c*) $Z=2$, $R=6.0\%$ for 1060 reflections. The nickel atom is octahedrally surrounded by two nitrogens, two chelating oxygens and two water oxygens.

H. Loiseleur, *Acta Crystallogr., Sect. B*, 28 (1972) 816.

Aquobis(en)copper(II) di-[catena-di- μ -cyanocuprate(I)], $\text{Cu}_3(\text{en})_2(\text{CN})_4\text{H}_2\text{O}$

(*Cc*) $Z=4$, $R=3.75\%$ for 3456 measured reflections. The coordination about the Cu^{II} atom is square-pyramidal with two bidentate (en) molecules at the basal positions and a water molecule at the apex.

R.J. Williams, A.C. Larson and D.T. Cromer, *Acta Crystallogr., Sect. B*, 28 (1972) 858.

Tin(II) sulphate, SnSO_4

(*Pnma*) $Z=4$, $R=4.4\%$ for 418 independent reflections. The tin atoms have pyramidal three-coordination with $\text{Sn}-\text{O} = 2.27, 2.27$ and 2.25 \AA ; $\text{O}-\text{Sn}-\text{O}$ angles are $79.0, 77.1$, and 77.1° .

J.D. Donaldson and D.C. Puxley, *Acta Crystallogr., Sect. B*, 28 (1972) 864.

μ -Allyl- μ -iodo-bis(triphenylphosphine palladium) benzene solvate, $\text{C}_3\text{H}_5\text{Pd}_2(\text{PPh}_3)_2\text{I}$

(*C2/c*) $Z=4$, $R=13.0\%$ for 1628 observed reflections. The two $(\text{PPh}_3)\text{Pd}$ moieties are bound together through a $\text{Pd}-\text{Pd}$ bond and palladium atoms are bridged by an iodine atom to form a triangular Pd_2I group. The allyl group lies parallel to the $\text{Pd}-\text{Pd}$ bond and is σ -bonded through terminal carbon atoms.

Y. Kobayashi, Y. Iitaka and H. Yamazaki, *Acta Crystallogr., Sect. B*, 28 (1972) 899.

Mono-(*N,N*-diethylnicotinamide) cadmium dithiocyanate

($P2_1/c$) $Z=4$, $R=3.6\%$ for 2594 significant independent reflections. Octahedral metal complexes are held together by thiocyanato and nicotinamide bridges. The Cd polyhedron is made up of 2 N atoms from NCS, 2 S atoms from NCS, a pyridine N atom and amide O atom.

F. Bigoli, A. Braibanti, M.A. Pellinghelli and A. Tiripicchio, *Acta Crystallogr., Sect. B*, 28 (1972) 962.

Chromium(III) tris(*O*-ethylxanthate)

($R\bar{3}$) $Z=2$, $R=6.9\%$ for 902 independent reflections. The ligands coordinate to chromium through sulphur atoms forming a distorted octahedron. Cr-S = 2.393(3) Å; S-C = 1.691(7) Å; C-O = 1.297(11) Å.

S. Merlino and F. Sartori, *Acta Crystallogr., Sect. B*, 28 (1972) 972.

Racemic- μ -amido- μ -superoxo-bis[bis(ethylenediamine) cobalt(III)] tetranitrate hydrate, DL-[(en)₂Co- μ -(NH₂, O₂)-Co(en)₂](NO₃)₄·H₂O

($P2_1/n$) $Z=4$, $R=7.5\%$ for 2312 reflections. The two cobalt atoms are octahedrally coordinated. The central five-membered ring of the cation is slightly puckered. O-O is 1.35 Å.

U. Thewalt and R.E. Marsh, *Inorg. Chem.*, 11 (1972) 351.

Tetratellurium tetrachloroaluminate, Te₄²⁺(AlCl₄⁻)₂ (A) and heptachlorodialuminate, Te₄²⁺(Al₂Cl₇⁻)₂ (B)

(A) (*Pbca*) $Z=4$, $R=7.3\%$ for 2616 independent reflections. The structure consists of planar and almost square Te₄²⁺ units. Te-Te = 2.6738(17) and 2.6633(16) Å, and approximately tetrahedral AlCl₄⁻ anions.

(B) ($P2_1/c$) $Z=2$, $R=9.0\%$ for 4896 independent reflections. This also has nearly square Te₄²⁺ units plus (Cl₃AlClAlCl₃)⁻ anions in the staggered conformation. Al-Cl-Al (bridge) angle is 110.8(3)°.

T.W. Couch, D.A. Lokken and J.D. Corbett, *Inorg. Chem.*, 11 (1972) 357.

O, O'-Diisopropylphosphorodithioate lead(II), Pb[(i-C₃H₇O)₂PS₂]₂

($P2_1/n$) $Z=4$, $R=5.6\%$ for 2034 reflections. Nearly planar Pb(S₂P)₂ groups stack one above the other in a staggered arrangement. Each lead is surrounded by six sulphurs, two at 2.766(8) Å, two at 3.01(3) Å and two at 3.20(4) Å. There are seven lone pairs around the lead and the overall stereochemistry is irregular pentagonal-bipyramidal in accordance with VSEPR rules.

S.L. Lawton and G.T. Kokotailo, *Inorg. Chem.*, 11 (1972) 363.

2, 2'-*commo*-Bis(nonahydrodicarba-2-cobalt-*clos*o-decaborate), $(C_2H_5)_4NCo(B_7C_2H_9)_2$ ($P4/n$) $Z=4$, $R=9.7$ for 926 reflections. The anion is a sandwich compound of cobalt with two carborane fragments in the shape of two distorted bicapped square antiprisms having one vertex occupied by the common cobalt.

D.St. Clair, A. Zalkin and D.H. Templeton, *Inorg. Chem.*, 11 (1972) 377.

Ruthenium (A) and osmium (B) nitrosyl carbonyl clusters with double nitrosyl bridges, $M_3(CO)_{10}(NO)_2$ (only (A) was solved)

($P2_1/n$) $Z=4$, $R=2.8\%$ for 1237 reflections (A). The structure consists of monomeric units each containing three ruthenium atoms at the vertices of an isosceles triangle. Two sides have $Ru-Ru = 2.866(5)$ Å and the third side has $Ru-Ru = 3.15$ Å, the lengthening caused by the two bridging NO groups ($N-O = 1.22(2)$ Å).

J.R. Norton, J.P. Collman, G. Dolcetti and W.T. Robinson, *Inorg. Chem.*, 11 (1972) 382.

Acenaphthenesilver perchlorate (A) and acenaphthylenesilver perchlorate (B)

(A) (Pmn) $Z=8$, $R=7.4\%$ for 1236 reflections. The structure consists of one-dimensional infinite chains of $AgClO_4$ with the aromatic groups lying on either side of the chains.

(B) ($P2_12_1$) $Z=4$, $R=20\%$ for 547 independent reflections. The structure consists of one-dimensional chains of alternating aromatic and perchlorate groups. The five-membered ring does not bond to the silver atom.

P.F. Rodesiler and E.L. Amma, *Inorg. Chem.*, 11 (1972) 388.

Ethylidyneheptacarbonyl- μ -[1, 2-bis(dimethylarsino)tetrafluorocyclobutene]-*triangulo*-tricobalt, $Co_3As_2F_4O_7C_{17}H_{15}$

($P2_1/n$) $Z=4$, $R=2.6\%$ for 1673 observed reflections. The cobalt atoms have a triangular arrangement with $Co-Co = 2.440(1)$ to $2.479(2)$ Å. A carbon atom in the ethylidyne group is symmetrically placed with respect to this triangle. $C-Co$ (average) = $1.903(7)$ Å. The *ffars* ligand bridges $Co-Co$ with $Co-As = 2.337(1)$ and $2.358(1)$ Å.

F.W.B. Einstein and R.D.G. Jones, *Inorg. Chem.*, 11 (1972) 395.

Li_4RhH_4 (A) and Li_4RhH_5 (B)

(I_4/m) $Z=2$, $R=6.25\%$ (A) and ($Cmcm$) $Z=4$, $R=14.5\%$ (B). Both compounds possess weak temperature-independent paramagnetism over 51–297°K range and both appear to be related to LiH suggesting ionic bonding. $Rh-Li = 2.55$ and 2.79 Å; $Li-Li = 2.46$ – 2.75 Å; $Rh-H = 1.81$ – 1.96 Å.

L.B. Lundberg, D.T. Cromer and C.B. Magee, *Inorg. Chem.*, 11 (1972) 400.

Copper complex of *N*- β -dimethylaminoethylsalicylaldimine, $Cu[OC_6H_4CHNCH_2CH_2N(CH_3)_2]_2$

($P2_1/n$) $Z=4$, $R=4.8\%$ for 3081 observed reflections. The copper coordination is nearer

to trigonal-bipyramidal rather than square-pyramidal; the β -nitrogen of one ligand is uncoordinated. Cu—O = 1.930(3) and 1.943(3) Å; Cu—N (azomethine) = 1.944(3) and 1.948(3); Cu—N(dimethylamino) = 2.553(4) Å. The latter distance is in the equatorial plane of the trigonal bipyramid.

P.C. Chieh and G.J. Palenik, *Inorg. Chem.*, 11 (1972) 816.

Tris(*tert*-butylthioxanthato)iron(III)

($P\bar{1}$) $Z=2$, $R=6.0\%$ for 2330 independent reflections. The iron atom is surrounded by a distorted octahedron of sulphur atoms from three chelating thioxanthate ligands. Fe—S (mean) = 2.297(7) Å. The geometries of coordinated 1:1 dithiolate ligands are discussed.

D.F. Lewis, S.J. Lippard and J.A. Zubieta, *Inorg. Chem.*, 11 (1972) 823.

Di-iodocarbonylferrocene-1, 1'-bis(dimethylarsine)nickel(II), $\text{NiI}_2(\text{CO})(\text{fdma})$

($P2_1 2_1 2_1$) $Z=4$, $R=3.8\%$ for 3437 reflections. The coordination of the nickel atom is nearly regular trigonal-bipyramidal with (fdma) occupying both an axial and an equatorial position. Ni—As_{ax} = 2.310(3); Ni—As_{eq} = 2.331(3) Å. The carbonyl occupies the other axial position; Ni—C = 1.82(2) Å.

C.G. Pierpont and R. Eisenberg, *Inorg. Chem.*, 11 (1972) 828.

Trimercury chloroaluminate, $\text{Hg}_3(\text{AlCl}_4)_2$

($P2_1/c$) $Z=4$, $R=8.2\%$ for 5580 reflections. The crystal contains discrete molecules having a nearly linear —Cl—Hg—Hg—Hg—Cl— central skeleton in which the chlorine atoms are shared with two nearly tetrahedral terminal AlCl_4 groups. Hg—Hg(mean) = 2.56 Å; Hg—Cl(mean) = 2.54 Å.

R.D. Ellison, H.A. Levy and K.W. Fung, *Inorg. Chem.*, 11 (1972) 833.

Diphenyldiarsenic trisulphide, $\text{As}_2\text{S}_3(\text{C}_6\text{H}_5)_2$

($C2/c$) $Z=4$, $R=6.1\%$ for 532 reflections. The molecule consists of a non-planar arsenic sulphur ring with the sequence —As—S—As—S—S—. One phenyl ring is bonded to each arsenic atom.

A.W. Cordes, P.D. Gwinup and M.C. Malmstrom, *Inorg. Chem.*, 11 (1972) 836.

Tetranuclear ruthenium carbonyl hydride, $\alpha\text{-H}_2\text{Ru}_4(\text{CO})_{13}$

($P2_1/c$) $Z=8$, $R=5.9\%$. The ruthenium atom is tetrahedrally coordinated. Eleven carbonyls are terminal and two form asymmetric Ru—C—Ru bridges; Ru—C = 1.94(5) and Ru—C = 2.40(6) Å. Ru—Ru = 2.93(1) (two) and 2.78(2) (mean of four). Indirect evidence implies that hydrogen atoms are situated in bridging configurations on the two long Ru—Ru edges.

D.B.W. Yawney and R.J. Doedens, *Inorg. Chem.*, 11 (1972) 838.

Beryllium borohydride, $\text{Be}(\text{BH}_4)_2$

($I4_1cd$) $Z=16$, $R=4.0\%$ for 276 reflections. The structure consists of a helical polymer of BH_4Be and BH_4 units.

D.S. Marynick and W.N. Lipscomb, *Inorg. Chem.*, 11 (1972) 820.

Tris(ethylenediamine)cobalt(III) hexachlorocadmate(II) dichloride dihydrate,

$[\text{Co}(\text{N}_2\text{C}_2\text{H}_8)_3]_2[\text{CdCl}_6]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$

($P2_1/c$) $Z=2$, $R=4.9\%$ for 1941 independent reflections. The CdCl_6^{4-} anion is a distorted octahedron. $\text{Cd}-\text{Cl}=2.588(7)$, $2.617(2)$ and $2.765(2)$ Å. The $\text{Co}(\text{en})_3^{3+}$ cation has the low-energy conformation $\Lambda(\delta\delta\delta)$.

J.T. Veal and D.J. Hodgson, *Inorg. Chem.*, 11 (1972) 597.

Di-2-(5-perfluoromethyltetrazolato)- μ -1, 2-bis(diphenylphosphino) ethane-bis(1, 2-bis(diphenylphosphino)ethane) dicopper(I), $\text{Cu}_2(\text{CF}_3\text{CN}_4)_2[(\text{CH}_2\text{P}(\text{C}_6\text{H}_5)_2)_2]_3$

($C2/c$) $Z=4$, $R=6.8\%$ for 1436 non-zero reflections. The centrosymmetric complex has a bridging diphos ligand. The copper is tetrahedrally coordinated to a bidentate diphos ligand and a nitrogen atom in the 2(3) position of the 5-perfluoromethyltetrazolate ion. $\text{Cu}-\text{P}=2.277(5)$ – $2.338(5)$ Å; $\text{N}-\text{N}=1.119(17)$ – $1.482(20)$ Å; $\text{C}-\text{N}=1.372(24)$ and $1.413(22)$ Å.

A.P. Gaughan, K.S. Bowman and Z. Dori, *Inorg. Chem.*, 11 (1972) 601.

 $[\text{BrF}_4^+][\text{Sb}_2\text{F}_{11}^-]$

($P2_1/a$) $Z=4$, $R=14\%$ for 773 reflections. The structure has infinite chains of BrF_4^+ and $\text{Sb}_2\text{F}_{11}^-$ ions coupled by weak fluorine bridges. This is the first direct evidence for the BrF_4^+ cation.

M.D. Lind and K.O. Christie, *Inorg. Chem.*, 11 (1972) 608.

Copper(I) *O, O'*-diisopropylphosphorodithioate, $\text{Cu}_4[(i\text{-C}_3\text{H}_7\text{O})_2\text{PS}_2]_4$

($P2_12_12_1$) $Z=4$, $R=7.2\%$ for 4667 independent reflections. The crystal consists of tetrameric molecules, each containing a central tetrahedron of copper atoms. Four $(\text{C}_3\text{H}_7\text{O})_2\text{PS}_2$ ligands chelate to the tetrahedron through sulphur atoms. One sulphur in each ligand bridges two copper atoms ($2.256(9)$ and $2.272(16)$ Å), the other bonds to a third copper at $2.272(9)$ Å. $\text{P}-\text{O}(\text{average})=1.56(2)$ Å.

S.L. Lawton, W.J. Rohrbach and G.T. Kokotailo, *Inorg. Chem.*, 11 (1972) 612.

Triethylammonium phenylthioarsenate, $(\text{C}_2\text{H}_5)_3\text{NH}^+(\text{C}_6\text{H}_5)\text{AsSO}(\text{OH})^-$

($P\bar{1}$) $Z=2$, $R=5.1\%$ for 362 observed reflections. The anion has an arsenic atom tetrahedrally bonded to a phenyl group, two oxygens and a sulphur atom at $1.90(2)$, $1.65(2)$, $1.75(3)$ and $2.08(2)$ Å respectively.

L.G. McRae, R.W. Perry, C.K. Fair, A. Hunt and A.W. Cordes, *Inorg. Chem.*, 11 (1972) 618.

Iodoheptakis(*tert.*-butylisocyanide)molybdenum(II) iodide, $\text{Mo}(\text{CNR})_6\text{I}_2$ ($\text{R} = t\text{-Bu}$) (*Acam*) $Z=8$, $R=6.8\%$ for 1408 independent reflections. The coordination geometry of the molybdenum is that of a monocapped trigonal prism, with carbon atoms at the vertices and an iodine above one of the square faces. $\text{Mo}-\text{C} = 2.06(2)$ *trans* to iodine and 2.12 \AA *cis*.

D.F. Lewis and S.J. Lippard, *Inorg. Chem.*, 11 (1972) 621.

Diphenylethylphosphine oxide adduct of tetrakis- $(\mu_3\text{-hydroxotricarbonyl hydridotungsten})$, $[\text{W}(\text{OH})(\text{CO})_3\text{H}]_4 \cdot 4\text{OPPh}_2\text{Et}$

(C_2/c) $Z=4$, $R=4.9\%$ for 1246 independent reflections. The tungsten atoms occupy the corners of a regular tetrahedron and are held together by four $\mu_3\text{-hydroxo}$ groups so that the W_4O_4 forms a distorted cube. $\text{W}-\text{W}(\text{mean}) = 3.48 \text{ \AA}$. Each tungsten is in a coordination polyhedron, which by apex sharing is a combination of one triangular and one square pyramid.

V.G. Albano, G. Ciani, M. Manassero and M. Sansoni, *J. Organometal. Chem.*, 34 (1972) 353.

Bis(π -ethylene)(tricyclohexylphosphine)nickel

($P2_1/c$) $Z=4$, $R=6.4\%$ for 1776 observed reflections. The nickel atom shows distorted trigonal symmetry with planar arrangements of the ligands. Both ethylenes have $\text{C}-\text{C} = 1.41 \text{ \AA}$ and distance from nickel $1.987\text{--}2.042 \text{ \AA}$. $\text{Ni}-\text{P}$ is 2.196 \AA . All cyclohexyl rings are in chair form and bonded equatorially to phosphorus.

C. Kruger and Y. Tsay, *J. Organometal. Chem.*, 35 (1972) 387.

Dirhenium decacarbonyl

The molecule consists of two $\text{Re}(\text{CO})_5$ fragments. $\text{Re}-\text{Re} = 3.04 \text{ \AA}$. The Re atom is in a slightly distorted octahedral configuration with angle $\text{C}(\text{axial})-\text{Re}-\text{C}(\text{equatorial}) = 88^\circ$. $\text{Re}-\text{C} = 2.01 \text{ \AA}$; $\text{C}-\text{O} = 1.16 \text{ \AA}$.

N.I. Gapotchenko, N.V. Alekseev, N.E. Kolobova, K.N. Anisimov, I.A. Ronova and A.A. Johansson, *J. Organometal. Chem.*, 35 (1972) 319.

Dicarbonylbis(diphenylethylphosphine)platinum(0), $\text{Pt}(\text{CO})_2[\text{P}(\text{C}_6\text{H}_5)_2(\text{C}_2\text{H}_5)]_2$

($P3_12_1$ or $P3_22_1$) $Z=3$, $R=4.2\%$ for 2051 reflections. The molecule is roughly tetrahedral. $\text{Pt}-\text{C} = 1.92(2) \text{ \AA}$; $\text{Pt}-\text{P} = 2.360(4) \text{ \AA}$. Angle $\text{C}-\text{Pt}-\text{C} = 117(1)^\circ$ and $\text{P}-\text{Pt}-\text{P} = 97.7(2)^\circ$.

V.G. Albano, P.L. Bellon and M. Manassero, *J. Organometal. Chem.*, 35 (1972) 423.

Bis(*tert.*-butyl isocyanide)(diphenylacetylene)nickel(0), $\text{Ni}(\text{C}_6\text{H}_5\text{C}\equiv\text{CC}_6\text{H}_5)[(\text{CH}_3)_3\text{C}-\text{N}\equiv\text{C}]_2$

($P2_1/a$) $Z=8$, $R=9.1\%$ for 2493 reflections. The nickel atom has trigonal coordination if diphenylacetylene is regarded as monodentate; the inner coordination sphere is planar.

C—C = 1.285(20) Å.

R.S. Dickson and J.A. Ibers, *J. Organometal. Chem.*, 36 (1972) 191.

Tetracyclopentadienylhafnium, $(C_5H_5)_4Hf$ or $(h^5-C_5H_5)_2Hf(h^1-C_5H_5)_2$

($P\bar{4}2_1m$) $Z=2$, $R=12\%$ for 567 independent reflections. The coordination of hafnium is distorted tetrahedral and the structure is similar to $(C_5H_5)_4Ti$.

V.I. Kuliskov, E.M. Brainina, N.G. Bokiý and Yu.T. Struchkov, *J. Organometal. Chem.*, 36 (1972) 333.

Dicarbonyl-(2, 2'-bipyridine)-pyridine- π -allylmolybdenum tetrafluoroborate, $[(CO)_2(C_{10}H_8N_2)(C_5H_5N)(\pi-C_3H_5)Mo]^+(BF_4^-)$

($P2_1/c$) $Z=4$, $R=8.15\%$ for 2488 independent reflections. The molybdenum is octahedral with *cis* carbonyls and bipyridine in one plane, and π -allyl and pyridine axial.

Mo—C(allyl) = 2.292(9) Å; C—C—C angle is $111.4 \pm 0.8^\circ$; C—C = 1.369(13) Å; Mo—C, C—O are 1.986(8) and 1.187(11) Å respectively.

R.H. Fenn and A.J. Graham, *J. Organometal. Chem.*, 37 (1972) 137.

Trihydridobis(1, 2-bisdiphenylphosphinoethane) rhenium(III), $ReH_3(DPPE)_2$

($P2/c$) $Z=2$, $R=7.5\%$ for 656 non-zero reflections. The hydrido ligands are assigned to occupy the three equatorial positions of a slightly distorted pentagonal bipyramid.

V.G. Albano and P.L. Bellon, *J. Organometal. Chem.*, 37 (1972) 151.

$(\pi-C_5H_5)Fe(CO)(f_6fos)SnMe_3$, where f_6fos is $(PPh_2)\overline{C=C(PPh_2)-(CF_2)CF_2}$

(A_1) $Z=4$, $R=8.0\%$ for 1703 observed reflections. The hexacoordinate iron atom is bonded to a carbonyl group, a cyclopentadienyl group, a trimethyltin group and a phosphorus atom of the f_6fos . Fe—Sn = 2.562 Å.

F.W.B. Einstein and R. Restivo, *Inorg. Chim. Acta*, 5 (1971) 501.

catena- μ -Bis-(1, 2-diphenylphosphiny)ethane-dichlorocopper(II)

(Cs/c) $Z=4$, $R=4.7\%$ for 2161 observed reflections. Infinite chains of $CuCl_2$ are linked by the phosphine oxide, which functions as a bridging ligand. Cu—O = 1.953(3),

Cu—Cl = 2.195(1) Å.

M. Mathew and G.J. Palenik, *Inorg. Chim. Acta*, 5 (1971) 573.

Calcium di-iodate(V) hexahydrate, $Ca(IO_3)_2 \cdot 6H_2O$

($Fdd2$) $Z=8$, $R=11.8\%$ for 626 independent reflections. The coordination polyhedron around calcium is a square antiprism. Ca—O = 2.43–2.75 Å. The environment of iodine is approximately octahedral.

A. Braibanti, A.M. Manotti Lanfredi, M.A. Pellinghelli and A. Tiripicchio, *Inorg. Chim. Acta*, 5 (1971) 590.

(A) π (Racemic)- and (B) χ -chloro(ethylenediamine)(diethylenetriamine)cobalt(III) tetrachlorozincate

($P2_1/n$) $Z=4$, $R=6.1\%$, for 2098 reflections (A) and (Cc) $Z=4$, $R=5.7\%$ for 1353 reflections (B). In both isomers cobalt is in an approximate octahedron of five nitrogens and one chlorine atom. In (A), the three dien nitrogens are in *facial* configuration and chlorine is in a *cis* position relative to the coordinated $-NH_2$ group of the dien. In (B), three nitrogens of dien are in a plane with one en nitrogen, and the second en nitrogen is *trans* to chlorine.

A.R. Gainsford, D.H. House and W.T. Robinson, *Inorg. Chim. Acta*, 5 (1971) 595.

1, 1'-Dimethylferricinium tri-iodide

($P\bar{1}$) $Z=2$, $R=3.5\%$ for 1278 independent reflections. $I-I = 2.85(2)$, $2.97(2)$ and $2.86(3)$ and $3.00(3)$ Å for the two asymmetric anions. The cp rings are almost exactly eclipsed with the methyl groups adjacent. $Fe-ring = 1.695(1)$, $Fe-C(ring) = 2.073(5)$, $C-C = 1.401(5)$, $C-CH_3 = 1.49(1)$ Å.

J.W. Bats, J.J. de Boer and D. Bright, *Inorg. Chim. Acta*, 5 (1971) 605.

8-Hydroxyquinolinatocarbonyltriphenylphosphine rhodium(I), $[RhOxq(Ph_3P)CO]$ (A) and 8-hydroxyquinolinatodicarbonylrhodium(I), $[RhOxq(CO)_2]$ (B)

(A) ($P1$) $Z=2$, $R=13.8\%$ for 1000 independent reflections. The interaction of the dicarbonyl complex with PPh_3 leads to substitution of the carbonyl group in the *trans* position with respect to the nitrogen of the 8-hydroxyquinoline.

(B) ($C2cm$) $Z=4$, $R=24\%$ for 128 independent reflections. $Rh-Rh$ is ~ 3.1 Å.

L.G. Kuzmina, Y.S. Varshavskii, N.G. Bokii, Y.T. Struchkov and T.G. Cherkasova, *J. Struct. Chem. (USSR)*, 12 (1971) 593.

Tris(acetylacetonato)holmium(III) tetrahydrate

($P2_1/n$) $Z=4$, $R=13.7\%$ for 2300 non-zero independent reflections. The holmium atom is surrounded at the apices of a dodecahedron by six (acac) oxygen atoms and two water oxygens.

L.A. Aslanov, E.F. Korytnyi and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 12 (1971) 600.

$(\pi-C_5H_5)_2Ti(C_6H_4CO_2)$

($Pbca$) $Z=8$, $R=13\%$ for 800 independent reflections. The carbon dioxide is fixed in the *ortho* position, with respect to the $C-Ti$ bond, of one of the phenyl rings of the starting complex in the form of a carboxylate group formed by an intramolecular $O-Ti$ bond with closing of the five-membered metallo cycle and displacement of the second phenyl group.

G.G. Aleksandrov and Y.T. Struchkov, *J. Struct. Chem. (USSR)*, 12 (1971) 605.

Mo₂Cl₂·H₂O

(*Pmn*2₁) *Z*=2, *R*=10.8% for 238 reflections. Infinite chains of MoO₃Cl₂H₂O octahedra are linked by common oxygen vertices in a manner similar to the WOCl₄ structure.

L.O. Atovmyan and A.G. Aliev, *J. Struct. Chem. (USSR)*, 12 (1971) 668.

Bis(ethylenediamine)copper(II) thio (A) and thioselenosulphates (B), Cu(en)₂S₂O₃ and Cu(en)₂SSeO₃

(*P*2₁/*c*) *R*=4. The compounds are isostructural. The nitrogen atoms of the two en groups form almost a square plane with Cu—N 2.06 in (A) and 2.07 in (B). The copper is square bipyramidal. Cu—S = 2.71 and 3.37, Cu—Se = 2.89 and 3.28 Å. The en ring has the *gauche* and a *KK'* confirmation.

N.V. Podberezskaya, S.V. Borisov and V.V. Bakakin, *J. Struct. Chem. (USSR)*, 12 (1971) 770.

Chlorobis(ethylenediamine)nickel perchlorate, Ni(en)₂ClClO₄

(*P*2₁/*n*) *Z*=4, *R*=12% for 1800 independent reflections. The octahedral nickel atoms are chlorine bridged and en ligands are *cis*. Ni—N = 2.05–2.13 Å. The longest bond is *trans* to the shortest. N—C (average) is 1.49 Å.

L.Kh. Minacheva, A.S. Antsyshkina and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 12 (1971) 775.

Dioxo-bis-(8-hydroxyquinolate)molybdenum(VI), MoO₂(C₉H₆NO)

(*C*2/*c*) *Z*=4, *R*=11.3% for 760 independent reflections. The molybdenum atom has distorted octahedral coordination and the two oxygen atoms are *cis*. O₁—Mo—O₁' is 104.1°. Mo—O = 1.71 ± 0.02 Å.

L.O. Atovmyan and Y.A. Sokolova, *J. Struct. Chem. (USSR)*, 12 (1971) 780.

Guanidinium tetraacetatocerate monohydrate, (CN₃H₆)[Ce(CH₃COO)₄]·H₂O

(*P*2₁/*b*) *Z*=4, *R*=17.7%. The structure consists of guanidinium ions and [Ac₃H₂O—Ce—Ac₂—Ce—Ac₃H₂O]²⁻ units. The coordination polyhedron of Ce contains nine acetate oxygens and a water oxygen. Three acetates are bidentate-chelating while two are tridentate-chelating-bridging. Ce—O = 2.45–2.83 Å, Ce—H₂O = 2.52 Å.

G.G. Sadikov, G.A. Kukina and M.A. Porai-Koshits, *J. Struct. Chem. (USSR)*, 12 (1971) 787.

Two polymorphic modifications of a π-complex-π-butadiene-π-cyclopentadienyl chloromethylgermyl iron compound

(*Pn*2₁/*a*) *Z*=4, *R*=12.8% (I) and (*P*1̄) *Z*=4, *R*=12.9% (II). The cp and butadiene ligands attach to iron and form a wedge-shaped sandwich. The iron atom is distorted octahedral and Ge is tetrahedral owing to steric hindrance between the ligands and a shortening of Fe—Ge from 2.28 to 2.29 (refs. given) to 2.56 Å.

V.G. Andrianov, V.P. Martynov and Y.T. Struchkov, *J. Struct. Chem. (USSR)*, 12 (1971) 793.

3, 6-Diphenylpyridazino-di-iron-triphenylphosphine pentacarbonyl, $[(C_6H_5)_2C_4H_2N_2] \cdot [Fe_2P(C_6H_5)_3(CO)_5]$

($P2_1/a$) $Z=4$, $R=12\%$ for 1180 independent reflections. The pyridazine ring acts as a diazo bridge between the two iron atoms which are metal-metal bonded. $Fe-Fe = 2.53$, $N-N = 1.43$, $Fe-N = 1.92$ Å. The iron atoms are octahedral, distorted by formation of the Fe_2N_2 tetrahedral cluster system.

L.G. Kuz'mina, N.G. Bokil, Yu.T. Struchkov, A.V. Arutyun'yan, L.V. Rybin and M.I. Rybinskaya, *J. Struct. Chem. (USSR)*, 12 (1971) 801.

Caesium octachloroaquodirhenate(III), $Cs_2[Re_2Cl_8H_2O]$

($P2_1/b$) $Z=4$, $R=16.6\%$ for 1800 independent reflections. The structure is made up of two types of dimeric complex anions, $[Re_2Cl_8]^{2-}$ and $[Re_2Cl_8 \cdot 2H_2O]^{2-}$ and Cs^+ cations.

P.A. Kozmin, G.N. Novitskaya, V.G. Kuznetsov and A.S. Kotal'nikova, *J. Struct. Chem. (USSR)*, 12 (1971) 861.

Ammonium *cis*-dioxalatovanadate(V)

($P2_12_12_1$) $Z=4$, $R=11.8\%$ for 986 independent non-zero reflections. The vanadium atom in the $[VO_2(C_2O_4)_2]^{3-}$ anion has six oxygen atoms in the octahedral environment. $V-O = 1.64 \pm 0.02$ Å (*cis* vanadyl group) The V-O oxo bonds in the *trans* positions are elongated.

L.O. Atovmyan and Y.A. Sokolova, *J. Struct. Chem. (USSR)*, 12 (1971) 863.

Bis(*O*-ethyl-*N*-phenylthiocarbamatopalladium(II)

($P2_1/n$) $Z=4$, $R=8.9\%$ for 1315 independent non-zero reflections. The palladium is square-planar, chelated by two chelates whose donor atoms are nitrogen and sulphur. $Pd-S = 2.36$; $Pd-N = 2.07$ Å.

L. Gastaldi and P. Porta, *Gazz. Chim. Ital.*, 9 (1971) 641.

Bis(*N*,isonicotinato)tetraaquazinc(II), $[Zn(C_5H_4NCO_2)_2(OH_2)_4]$ (A) and bis(*N*,isonicotinato)tetraaquocadmium(II), $[Cd(C_5H_4NCO_2)_2(OH_2)_4]$ (B)

($P\bar{1}$) $Z=1$, $R=5.1\%$ for 1330 independent reflections (A) and ($P\bar{1}$) $Z=1$, $R=2.1\%$ for 1980 reflections. In both complexes the metal is octahedral and is coordinated by two isonicotinate nitrogen atoms and four waters. $Zn-N = 2.144(5)$ and $Cd-N = 2.310(3)$ Å.

M.B. Cingi, A.G. Manfredotti, C. Guastini, A. Musatti and M. Nardelli, *Gazz. Chim. Ital.*, 11 (1971) 815.

(Pyridine-2, 6-dicarboxylato)diaquocopper(II), $[Cu(C_7H_3NO_4)(OH_2)_2]$

($P2_1/c$) $Z=4$, $R=3.1\%$ for 1627 reflections. The organic ligand is tridentate and coordinates through nitrogen and two carboxylic oxygen atoms. The coordination of copper is square-bipyramidal and there are two long Cu-O bonds (2.423 and 2.396 Å respectively).

A.C. Villa, C. Guastini and M. Nardelli, *Gazz. Chim. Ital.*, 11 (1971) 825.

Barium salt of the chromium(III) chelate with the thiosemicarbazone of pyruvic acid ($C2/c$). The structure contains discrete $Ba[Cr(L)_2]_2 \cdot 5H_2O$ units. In one ligand only are both carboxylate oxygens bonded to the barium ion, which is surrounded by the oxygens of two thiosemicarbazone molecules and five waters, forming an irregular nonagon. G.F. Volodina, G.A. Kiosse, N.V. Gêrbôteu and A.V. Ablov, *Dokl. Chem.*, 4-6 (1971) 853.

$[Te(Et_2(NCS_2))_3 C_6H_5]$ (A) and $[Te(Et_2NCS_2)_4]$ (B)
($P2_1/c$) $Z=4$, $R=6.2\%$ for 4183 reflections (A) and ($Pna2_1$) $Z=4$, $R=8.8\%$ for 2645 reflections (B). In (A) the Te atom is seven-coordinate and in (B), eight-coordinate. The geometries do not agree with those predicted by VSEPR theory.

S. Esperås, S. Husebye and S.E. Svaeren, *Acta Chem. Scand.*, 25 (1971) 3539 (preliminary communication).

Tris(hydroxyacetato)europium(III), $Eu(HOCH_2COO)_3$
($P2_1$) $Z=2$, $R=8.7\%$ for 1560 reflections. The metal atom is nine-coordinated in a tri-capped prism type. All ligands chelate with hydroxyl oxygens in the equatorial positions. One carboxylate oxygen occupies a corner position of the prism, and carboxylate oxygens which are bonded to neighbour europium atoms occupy the remaining three positions.

I. Grenthe, *Acta Chem. Scand.*, 25 (1971) 3347.

trans-Dithioureabis(tetramethylthiourea)tellurium(II) chloride and bromide, $[Te(SC(NH_2)_2)_2 \cdot \{SC(N(CH_3)_2)_2\}_2]^{2+} Br_2$ and Cl_2
($P2_1/c$) $Z=2$, $R=8.0\%$ for 1512 reflections (chloride) and ($P2_1/c$) $Z=2$, $R=5.1\%$ for 1452 reflections (bromide). $Te-S_1$ (thiourea) = 2.710(3); $Te-S_2$ (tetramethylthiourea) = 2.688(3) Å; angle $S_1-Te-S_2 = 91.37(10)^\circ$ for the Cl salt. $Te-S_1 = 2.706(3)$, $Te-S_2 = 2.679(3)$ Å; angle $S_1-Te-S_2 = 91.45(9)^\circ$ for the Br salt.

O.P. Anderson, *Acta Chem. Scand.*, 25 (1971) 3593.

Tris(hydroxyacetato)erbium(III) dihydrate, $Er(HOCH_2COO)_3 \cdot 2H_2O$
($P2_1/c$) There are two non-equivalent metal atoms located on two-fold axes and each coordinated by eight oxygen atoms forming distorted dodecahedra. One metal is coordinated by four hydroxyacetate ligands and the other by two hydroxyacetates and four waters.

I. Grenthe, *Acta Chem. Scand.*, 25 (1971) 3721.

trans-Diselenocyanatobis(trimethylenethiourea)tellurium(II), $Te(SC[NH(CH_2)_3NH])_2 \cdot (SeCN)_2$ (A) and *trans*-diselenocyanatobis(tetramethylthiourea)tellurium(II), $Te(SC[N(CH_3)_2NH])_2 \cdot (SeCN)_2$ (B)

($P2_1/c$) $Z=2$, $R=7.3\%$ for 924 independent reflections (A) and ($P2_1/c$) $Z=2$, $R=7.6\%$ for 1119 independent reflections (B). In both complexes tellurium is bonded to two selenium atoms and two sulphur atoms in a *trans* square-planar arrangement. Te—Se = 2.834(2), 2.815(3); Te—S = 2.670(5), 2.728(4) Å. Angle Se—Te—S = 89.73(11)°, 91.61(10) where in each case the two data refer to (A) and (B) respectively.

K.A.O. Foss and I. Roti, *Acta Chem. Scand.*, 25 (1972) 3809.

Di- μ -sulphato- μ -hydroxo-bis[triammine cobalt(III)]sulphate 8.hydrate

($P2/a$) $Z=2$, $R=3.8\%$ for 2515 independent reflections. Within the cation two bidentate SO_4 ligands form bridges between two cobalt atoms. S—O(endocyclic) = 1.50 and (exocyclic) = 1.45 Å.

K. Wieghardt and G. Maas, *Z. Anorg. Allg. Chem.*, 385 (1971) 289.

Tetrachloro(trichloro-acetonitrile)pentachloroethylnitrido-tungsten(VI)

The tungsten atom is in a distorted octahedral environment, bonded to four chlorines and two apical nitrogens. The WCl_4 unit is not planar.

M.G.B. Drew, K.C. Moss and N. Rolfe, *Inorg. Nucl. Chem. Lett.*, 7 (1971) 1219.