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X-RAY BIBLIOGRAPHY

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trans-Bis(triphenyltin)tetracarbonylosmium, *trans*-$(Ph_3Sn)_2Os(CO)_4$
($C2/c$) $Z = 8$, $R = 6.3\%$ for 3444 independent reflections. The molecules lie on special positions in the cell (b and d) which are centres of symmetry instead of being in a general position. The main structural features of interest are the linear Sn—Os—Sn cluster with the two independent Sn—Os bond distances 2.712(1) and 2.711(1) Å. Average C—C, 1.399 Å.
J.P. Collman, D.W. Murphy, E.B. Fleischer and D. Swift, *Inorg. Chem.*, 13 (1974) 1.

Bis(1,5-diphenyl-1,3,5-pentanetrionato)tetrapyridinedinickel(II)—tetrapyridine, $Ni_2(DBA)_2(py)_4 \cdot 4py$
($P2_1/n$) $Z = 2$, $R = 7.5\%$ for 2567 independent reflections. The molecular complex, possessing crystallographic site symmetry $C - \bar{1}$, consists of two nickel atoms coordinated to two 1,5-diphenyl-1,3,5-pentanetrionate ligands and to four pyridine nitrogens located above and below the metal—oxygen plane. Ni—Ni = 3.166(3), Ni—N(ave.), 2.155(8), Ni—O(bridging)(ave.), 2.044(6) Å and Ni—O(terminal) (ave.), 2.014(6) Å. The intra-ring bond angles are O—Ni—O, 78.47(24)° and Ni—O—Ni, 101.51(24)°.
R.L. Lintvedt, L.L. Borer, D.P. Murtha, J.M. Kuszaj and M.D. Glick, *Inorg. Chem.*, 13 (1974) 18.

1,2-Bis(ferrocenyl)ethane, $(C_5H_5)Fe(C_5H_4)CH_2CH_2(C_5H_4)Fe(C_5H_5)$
($Pbca$) $Z = 4$, $R = 4.5\%$ based on 1202 observed reflections. The molecular structure of the compound has two ferrocene moieties symmetrically equivalent by virtue of an inversion centre at the midpoint of the carbon—carbon bond in the ethane fragment. The compound adopts a transoid configuration in the solid state. The cyclopentadienyl rings in the respective ferrocene moieties are parallel and separated by 3.34 Å. Fe—C distances are equivalent and equal to 2.030 Å, inner ring C—C distances average 1.413 Å. The cyclopentadienyl rings are rotated 8.5° from the fully eclipsed configuration.
J.R. Doyle, N.C. Baenziger and R.L. Davis, *Inorg. Chem.*, 13 (1974) 101.

Platinum bronzes, $CaPt_2O_4$ (A) and $Cd_{0.3}Pt_3O_4$ (B)

(D_{4h}^9—$P4_2/mmc$) $Z = 4$, $R = 16.0\%$ (A) and (O_h^3—$Pm3n$) $Z = 2$, $R = 6.8\%$ (B). The crystal structures of (A) and (B) have been determined from X-ray and neutron diffraction powder data. $CaPt_2O_4$ has a structure that is the two-dimensional analogue of the general platinum bronze structure. The unique features of the structure are the non-intersecting Pt chains in the x and y directions. Selected bond distances are Pt_2—Pt_2, 2.79(5); Pt_1—Pt_3, 2.99(5); Pt—O, 2.09(3); Pt—Ca_1, 3.17(1); Pt—Ca_2, 3.21(1); Ca—O, 2.481(3); O—O, 2.68(8), 2.7994(3) and 3.09(8) Å. Angles are O—Pt—O, 95(1)°, 171(2)°, 84(2)°; Pt_1—O—Pt_2, 110(3)°; O—Pt_1—Pt_2, 94(1)°; O—Pt_1—Pt_3, 86(1)°; Ca—O—Ca, 173(3)°. Interatomic distances in $Cd_{0.3}Pt_3O_4$ are: Pt—O, 1.9948(3); Pt—Cd, 3.1541(2); O—Cd, 2.4432(2); Pt—Pt, 2.8211(2); O—O, 2.8211(2) Å.

D. Cahen, J.A. Ibers and M.H. Mueller, *Inorg. Chem.*, 13 (1974) 110.

Tris(p,p'-diamino-2,3-diphenylbutane)—sodium chloride, —nitrate, and —cyanide, $Na(DPB)_3Cl$ (A), $Na(DPB)_3NO_3$ (B) and $Na(DPB)_3CN$ (C).

($R\bar{3}c$) $Z = 6$, $R = 8.2\%$ for 1216 reflections (A). The nitrogens of the amines are hexacoordinated to the sodium ions. Na—N, 2.608(3) Å. (B) and (C) were refined using the general positions from (A). $R = 11.3\%$ (B), 11.7% (C).

L.A. Duvall and D.P. Miller, *Inorg. Chem.*, 13 (1974) 120.

N-(3-Hydroxypropyl)salicylaldimine—iron chloride, Fe(SALPA)Cl (A) and bis-(*N*-(2-phenylethyl)salicylaldimine)—iron chloride, $Fe(SANE)_2Cl$ (B)

($P2_1/c$) $Z = 2$, $R = 9.9\%$ for 850 reflections (A). The structure contains dimeric units, $[Fe(SALPA)Cl]_2 \cdot C_6H_5CH_3$. The coordination of the iron is intermediate between a square-pyramidal and a trigonal-bipyramidal arrangement with the tridentate ligand and the bridging alkoxide oxygen in the basal plane, and Cl in the apical position. Fe—O distances are 1.983 and 1.934 Å, with angles of 75.9° and 104.1° at iron and oxygen, respectively. The chlorine—iron—basal ligand angles vary from 97.9 to 113.8°. Fe—Fe, 3.089(6); Fe—Cl, 2.226(6); Fe—N, 2.06(1) Å.

($P2_1/c$) $Z = 4$, $R = 7.4\%$ for 1955 reflections (B). The coordination of the iron(III) atom is intermediate between a square pyramid and a trigonal bipyramid. Considering it as a square pyramid, the chlorine occupies the apical position, with the iron atom displaced 0.40 Å toward the apex, and the *trans* chelate occupying the basal plane. Fe—Cl, 2.251(5); Fe—N, 2.142(11) and 2.122(11); Fe—O, 1.863(11) and 1.882(10) Å. The chlorine—iron—basal ligand angles vary from 97.3 to 106.7°.

J.A. Bertrand, J.L. Breece and P.G. Eller, *Inorg. Chem.*, 13 (1974) 125.

Pentacarbonyl(tetraphosphorus trisulphide)molybdenum, $Mo(CO)_5P_4S_3$

($P2_1/c$) $Z = 8$, $R = 8.2\%$ based on 1476 reflections. the structure shows an intact P_4S_3 cage molecule attached to a $Mo(CO)_5$ moiety through the apical phosphorus atom. Mo—P, 2.477(6); Mo—C (ave.), 2.06(2); and C—O (ave.), 1.14(4) Å.

A.W. Cordes, R.D. Joyner, R.D. Shores and E.D. Dill, *Inorg. Chem.*, 13 (1974) 132.

Bismuth(III) *O,O'*-diisopropylphosphoradithioate, $Bi[(i\text{-}C_3H_7O)_2PS_2]_3$
($P2_1/c$) $Z = 4$, $R = 7.6\%$ for 2734 independent reflections. The bismuth ion is coordinated to three ligands in a pseudo-trigonally distorted octahedral environment with C_3 symmetry. The three Bi—S bonds adjacent to the lone pair in the $:BiS_6$ group are longer (averaging 2.875 Å) than the three (averaging 2.702 Å) most remote from the lone pair. Interligand angles between the longer bonds are greater (99.9°) than those between the shorter bonds (91.2°), whereas the S—Bi—S angles average only 72.0° as dictated by the ligand bite.
S.L. Lawton, C.J. Fuhrmeister, R.G. Haas, C.S. Jarman, Jr. and F.G. Lohmeyer, *Inorg. Chem.*, 13 (1974) 135.

Diperchloratobis[2-(2-aminoethyl)pyridine]copper(II), $Cu(AEP)_2(ClO_4)_2$
($P\bar{1}$), $Z = 1$, $R = 3\%$ for 2275 independent reflections. The molecule is a six-coordinate monomer with tetragonally distorted geometry at the copper atom; four nitrogen atoms from two AEP ligands form the equatorial plane and oxygen atoms from two perchlorate groups occupy the axial sites. The perchlorate groups are weakly coordinated with Cu—O, 2.883(2) Å, compared with Cu—N distances of 2.005(2) and 2.024(2) Å for the AEP coordination.
D.L. Lewis and D.J. Hodgson, *Inorg. Chem.*, 13 (1974) 143.

α-Di-μ-hydroxy-bis[2-(2-dimethylaminoethyl)pyridine] dicopper(II) perchlorate, α-$[Cu(DMAEP)\text{-}OH]_2(ClO_4)_2$
($P\bar{1}$) $Z = 1$, $R = 2.7\%$ for 2134 independent reflections. The complex consists of pairs of copper atoms linked by two hydroxo bridges and two intramolecular bidentate perchlorate groups. The geometry at each copper atom is distorted octahedral, the equatorial plane consisting of two hydroxo oxygen atoms and two nitrogen atoms from a DMAEP ligand, (Cu—O, 1.947(2) and 1.936(2) Å; Cu—N, 2.017(2) and 2.056 Å), while the axial sites are occupied by perchlorate oxygen atoms (Cu—O, 2.716(3) and 2.782(2) Å).
D.L. Lewis, W.E. Hatfield and D.J. Hodgson, *Inorg. Chem.*, 13 (1974) 147.

Thallous tetracarbonylcobaltate, $TlCo(CO)_4$
($P2_13$) $Z = 4$, $R = 8.3\%$ for 135 observed reflections. The structure consists of discrete Tl^+ and $Co(CO)_4^-$ ions arranged in an NaCl-like structure with the closest approach of Tl and Co atoms being 3.55(1) Å, which is about 0.35 Å longer than the sum of their covalent radii.
D.P. Schussler, W.R. Robinson and W.F. Edgell, *Inorg. Chem.*, 13 (1974) 153.

Tris(tropolonato)scandium(III), $Sc(C_7H_5O_2)_3$ or $Sc(trop)_3$
($R\bar{3}c$) $Z = 6$, $R = 3.3\%$ for 783 significant reflections. The coordination environment is intermediate between trigonal-antiprismatic and trigonal-prismatic. The molecule has crystallographically imposed D_3 symmetry. Sc—O, 2.102 Å. The tropolonato ligand behaves as a rigid, inflexible chelating agent with a ligand "bite" of 2.523 Å causing little change in its overall structure.
T.J. Anderson, M.A. Newman and G.A. Melson, *Inorg. Chem.*, 13 (1974) 158.

Cubic phase of xenon hexafluoride, XeF_6, at 193°K

$(Fm3c)$ $Z = 144$, $R = 7.0\%$ for 487 unique reflections. The structure is not molecular but based on the association of XeF_5^+ and F^- ions into tetrameric and hexameric rings. The structure is disordered and the 1008 atoms in the cell are distributed over 1600 positions. The tetramer unit, consisting of four XeF_5^+ ions and four F^- ions, is an eight-membered ring with four long and four short bridging contacts. In XeF_5^+, distances are Xe—F(apical), 1.83(4), Xe—F(basal), 1.84(3); Xe—F(bridging)(short), 2.23(3); Xe—F(bridging)(long), 2.60(3) Å. The hexamer is an twelve-membered ring consisting of six XeF_5^+ ions and six F^- ions with 18 equal bridging contacts. In XeF_5^+, Xe—F(apical), 1.75(3); Xe—F(basal), 1.88(2); Xe—F(bridging), 2.56(2) Å.

R.D. Burbank and G.R. Jones, *J. Amer. Chem. Soc.*, 96 (1974) 43.

μ-Bis(cyanotrihydroborato)-tetrakis(triphenylphosphine)dicopper(I), $[((C_6H_5)_3P)_2Cu(NCBH_3)]_2$

$(P2_1/n)$, $Z = 4$, $R = 6.0\%$ for 2921 independent reflections. The molecular configuration of $[((C_6H_5)_3P)_2Cu(NCBH_3)]_2$ consists of dimeric units, and each copper atom is four-coordinate. The H_3BCN^- ligands bridge the two copper atoms forming a ten-membered non-planar ring, with a Cu···Cu distance of 5.637(2) Å. Each quasi-tetrahedral copper atom is bonded to two triphenylphosphine ligands and to a hydrogen and a nitrogen atom from the different H_3BCN^- groups. The Cu—N distances are 1.96(1) and 2.01(1); Cu—H(ave.), 1.86(10) Å. The Cu—P distances are 2.262(3), 2.242(3), 2.272(3) and 2.280(3) Å with $<$ P—Cu—P, 124.8(1) and 122.1(1)°.

K.M. Melmed, Ting-i Li, J.J. Mayerle and S.J. Lippard, *J. Amer. Chem. Soc.*, 96 (1974) 69.

(Norbornadiene)bis(dimethylphenylphosphine)(trichlorostannato)iridium(I), $(C_7H_8)(PMe_2Ph)_2Ir(SnCl_3)$

$(P2_1/c)$, $Z = 4$, $R = 3.93\%$ for 4704 independent reflections. The overall coordination geometry of the iridium atom is almost midway between trigonal-bipyramidal (with (*P*1) and olefin axial) and square-pyramidal (with Sn axial) Bond distances around Ir are, Ir—Sn, 2.5867(6); Ir—P(1), 2.306(2); Ir—P(2), 2.309(2); Ir—olefin, 2.068(6) and 2.123(6) Å. Selected angles are Sn—Ir—P(1), 95.79°, Sn—Ir—olefin, 103.38° and 113.91(17)°. P(1)—Ir—olefin, 93.86(18)° and 157.40(16)°.

M.R. Churchill and Kuo-Kuang G. Lin, *J. Amer. Chem. Soc.*, 96 (1974) 76.

Bis(piperidine)-$\alpha,\beta,\gamma,\delta$-tetraphenylporphinatocobalt(II), Pip_2CoTPP

$(P1)$, $Z = 1$, $R = 4.3\%$ for 4360 independent reflections. The octahedral CoN_6 coordination group in the centrosymmetric (C_i—1) molecule approximates to full tetragonal symmetry. Equatorial Co—N bond lengths are 1.987 Å, while the axial Co—N bonds are 2.436(2) Å. The porphinato core displays only trivial departures from D_{4h} symmetry.

W.R. Scheidt, *J. Amer. Chem. Soc.*, 96 (1974) 84.

(1-Methylimidazole)-$\alpha,\beta,\gamma,\delta$-tetraphenylporphinatocobalt(II), MImCoTPP
($P2_1/c$), $Z = 4$, $R = 7.3\%$ for 4065 independent reflections. The square-pyramidal CoN_5 coordination group has equatorial bond lengths of 1.977(3) Å; the axial Co—N bond is 2.157(3) Å. The cobalt is displaced 0.14 Å from the mean skeletal plane.
W.R. Scheidt, *J. Amer. Chem. Soc.*, 96 (1974) 90.

1-η-Hydrazido(2-)chlorobis[1,2-bis(diphenylphosphino)ethane]tungsten(IV) cation, $[W(N_2H_2)Cl(diphos)_2]BPh_4$
($P2_1/c$), $Z = 4$, $R = 6.4\%$ for 5599 independent reflections. The structure consists of octahedrally coordinated W, the diphos ligands occupy the equatorial plane, with Cl and N_2H_2 in the axial positions. The essential linearity of < W—N(1)—N(2), 171.(1)°, establishes the ligand as the monohapto hydrazido (2-) form (b). N(1)—N(2), 1.37(2); W—N(1), 1.73(1); W—Cl, 2.421(4); W—P, 2.51 ± 0.03 Å.
G.A. Heath, R. Mason and K.M. Thomas, *J. Amer. Chem. Soc.*, 96 (1974) 259.

$[ReCl_2(NH_3)(NNHC_6H_5)(P(CH_3)_2C_6H_5)_2]Br$(ethanol)
($P2_1/n$), $R = 5.4\%$ for 2197 independent reflections. The octahedral geometry around the Re^{III} consists of axially coordinated $(P(CH_3)_2C_6H_5)$ with Cl, NH_3 and $(N_2HC_6H_5)$ in the equatorial plane. Both the linearity of Re—N(1)—N(2) and the difference electron density confirm the site of protonation as N(2), its stereochemistry being essentially planar. Bond distances are Re—N(1), 1.750(12); N(1)—N(2), 1.28(2); N(2)—H$\cdots$$B_R$, 3.09(1); mean Re—P, 2.445(5); Re—NH_3, 2.20(1); mean Re—Cl, 2.405(6) Å. Bond angles are Re—N(1)—N(2), 172(1)°; N(1)—N(2)—C(1), 119(1)°; N(1)—N(2)—H, 118(5)°; C(1)—N(2)—H, 121.5°.
R. Mason, K.M. Thomas, J.A. Zubieta, P.G. Douglas, A.R. Galbraith and B.L. Shaw, *J. Amer. Chem. Soc.*, 96 (1974) 260.

Phenyl(6,7,13,14=tetramethyl-1,2,4,5,8,9,11,12-octaazacyclotetradeca-5,7,12,14-tetraene)iron(III), $[Fe(C_{22}H_{22}N_4)(C_6H_5)]$. An organoiron(III) macrocycle.
($P\bar{1}$), $Z = 2$, $R = 6.6\%$ for 4206 independent reflections. The molecular structure consists of five-coordinate iron(III) σ-bonded to the carbon of the phenyl ring and the four nitrogen atoms of a dianionic macrocyclic ligand. The ligands form a square pyramid around the iron, with the phenyl group in the axial position. Fe—N(equatorial), 1.90—1.91, Fe—C(axial), 1.933(3) Å. The iron atom is displaced 0.23 Å from the least-squares plane of the four nitrogen atoms.
V.L. Goedken, Shie-Ming Peng and Young-ae Park, *J. Amer. Chem. Soc.*, 96 (1974) 284.

trans-Tetraaminecarbonyl-2-(4,5-dimethylimidazolium)ruthenium(II) cation, $[(C_5H_8N_2)Ru(NH_3)_4(CO)](PF_6)_2$
($C2/m$), $Z = 4$, $R = 3.2\%$ for 3565 reflections. The structure consists of dis-

crete $[(C_5H_8N_2)Ru(NH_3)_4(CO)]^{2+}$ and PF_6^- ions. The attachment of the imidazolium moiety to the Ru atom through the central carbon atom is clearly established (Ru—C, 2.128(4) Å). The Ru has octahedral geometry with the four amine ligands in the equatorial plane and CO *trans* to $(C_5H_8N_2)$ in the axial positions. Ru—NH_3, 2.126(1); Ru—CO, 1.849; C—O, 1.49 Å; < Ru—C—O, 176°; < $(C_5H_8N_2)$—Ru—NH_3, 89°.

R.J. Sundberg, R.F. Bryan, I.F. Taylor, Jr. and H. Taube, *J. Amer. Chem. Soc.*, 96 (1974) 381.

cis-anti-Bis(pentahaptocyclopentadienyl)dicarbonylbis(μ-methyl isocyanide)-diiron(*Fe*—*Fe*), $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_2(\mu\text{-}CNCH_3)_2$

($P\bar{1}$), $Z = 2$, $R = 3.9\%$ for 2050 independent reflections. This tautomer has the *cis* conformation and an anti relationship of the two bent $CNCH_3$ groups in the bridging positions. The two three-membered rings involving the bridging isocyanides, joined along the Fe—Fe bond, are far from planar. Each Fe atom has a tripod arrangement of the two bridging carbon atoms and the carbon atom of the terminal CO group, with < C—Fe—C, 90°. Fe—Fe, 2.538(1); Fe—C(terminal), 1.714(8); Fe—C(bridging), 1.937; Fe—centroid of Cp rings, 1.754(6) Å.

F.A. Cotton and B.A. Frenz, *Inorg. Chem.*, 13 (1974) 253.

Bis(pentahaptocyclopentadienyl)tricarbonyl(*tert*-butyl isocyanide)diiron(*Fe*—*Fe*), *cis*-$(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3[CNC(CH_3)_3]$

($P2_1/c$), $Z = 4$, $R = 3.1\%$ for 1658 reflections. The structure is that of the *cis* isomer with a terminal isocyanide group, and bridging carbonyls. Fe—Fe, 2.523(2); Fe—C(bridging), 1.93 ± 0.01, Fe—C(isocyanide), 1.81(1), Fe—CO-(terminal), 1.73(1) Å. < Fe—C—N(isocyanide), 178.5(8)°.

R.D. Adams, F.A. Cotton and J.M. Troup, *Inorg. Chem.*, 13 (1974) 257.

trans-2-(2'-Quinolyl)methylene-3-quinuclidinonedichloronickel(II), $[Ni(Qnqn)Cl_2]_2$

($P\bar{1}$), $Z = 1$, $R = 2.6\%$ for 2122 independent reflections. The complex is binuclear, containing two nickel(II) ions, each with square-pyramidal coordination, with two slightly asymmetric bridging chloride ions (Ni—Cl, 2.408(2) and 2.422(2) Å) in equatorial sites. Ni···Ni, 3.652(1) Å. The two remaining equatorial coordination sites on each nickel ion are occupied by a terminal chloride ion (Ni—Cl, 2.296(2) Å) and by a quinolyl nitrogen (Ni—N, 2.047(3) Å). The apical coordination position is occupied by a quinuclidinone nitrogen (Ni—N, 2.067(2) Å). The nickel ion is 0.40 Å above the basal plane of the square pyramid.

G.J. Long and E.O. Schlemper, *Inorg. Chem.*, 13 (1974) 279.

cis-Thiocyanatoisothiocyanatobis(3,3-dimethylbutynyldiphenylphosphine)-platinum(II), *cis*-$Pt(NCS)(SCN)(Ph_2PC{\equiv}C\text{-}t\text{-}Bu)_2$

($Pca2_1$), $Z = 4$, $R = 5.7\%$ for 1951 independent reflections. The platinum atom is coordinated in square-planar fashion by two phosphorus atoms of the acetylenic phosphines, a nitrogen atom of an isothiocyanate group, and a sulphur atom of a thiocyanate group. Pt—P(1), 2.275(6); Pt—P(2), 2.444(7); Pt—S, 2.374(8); Pt—N, 2.061(29) Å.

Y.S. Wong, S. Jacobson, P.C. Chieh and A.J. Carty, *Inorg. Chem.*, 13 (1974) 284.

Ammonium samarium sulphate tetrahydrate, $NH_4Sm(SO_4)_2 \cdot 4H_2O$

($P2_1/c$), $Z = 4$, $R = 4.6\%$ for 1145 reflections. Sm is coordinated by six oxygen atoms from SO_4^{2-} ions (Sm—O, 2.378—2.559 Å) and by three H_2O molecules (Sm—O, 2.442—2.512 Å). The nine oxygen atoms form a polyhedron — either a tricapped trigonal prism or a monocapped square antiprism.

B. Eriksson, L.O. Larsson, L. Niinisto and U. Skoglund, *Inorg. Chem.*, 13 (1974) 290.

Tris(tetramethylammonium)hexachloro(dodeca-μ-chloro-hexaniobate), $[(CH_3)_4N]_3[(Nb_6Cl_{12})Cl_6]$

($R3$), $Z = 3$, $R = 6.9\%$ for 1377 independent reflections. The structure contains a close-packed cubic arrangement of $[(Nb_6Cl_{12})Cl_6]^{3-}$ complex cluster anions which are based on $(Nb_6Cl_{12})^{3+}$ central cations. The $(CH_3)_4N^+$ cations occupy all tetrahedral and octahedral holes. Nb—Nb, 2.97; Nb—Cl(bridging), 2.43; Nb—Cl(terminal), 2.52 Å.

F.W. Koknat and R.E. McCarley, *Inorg. Chem.*, 3 (1974) 295.

Iodoruthenocene(IV) triiodide, $[Ru(cp)_2I]I_3$

($Pnma$), $Z = 4$, $R = 8.9\%$ for 1093 independent reflections. The cp rings in $Ru(cp)_2I^+$ are eclipsed, each ring being tilted back 16° to accommodate the I^- ligand. Ru—C(ave.), 2.20 ± 0.01; Ru—I, 2.732(3) Å.

Y.S. Sohn, A.W. Schlueter, D.N. Hendrickson and H.B. Gray, *Inorg. Chem.*, 13 (1974) 301.

Bis[copper(II) D-penicillamine disulphide] nonahydrate, $[(H_2O)Cu(O_2CCH(NH_2)C(CH_3)_2SSC(CH_3)_2CH(NH_2)CO_2)_2Cu(H_2O)] \cdot 7H_2O$

(C_2), $Z = 4$, $R = 7.7\%$ for 1229 unique reflections. The structure consists of dimeric [copper(II) D-penicillamine disulphide] units with non-equivalent Cu atoms bridged by disulphide. The Cu coordination is tetragonal. Two nitrogen and two oxygen atoms from the terminal amino acid are coordinated to Cu in a *trans* square-planar fashion. Cu is displaced 0.06—0.11 Å out of the plane toward a H_2O molecule occupying the fifth coordination site. Disulphide units are located in the interior of the dimer. Apical O—Cu—S angles are 169.6(7) and 166.5(7)°; distances are Cu—S, 3.057(10) and 3.138(9); Cu—O(equatorial), 1.96 ± 0.02; Cu—N, 2.05(2) and 1.97(2) Å.

J.A. Thich, D. Mastropaolo, J. Potenza and H.J. Schugar, *J. Amer. Chem. Soc.*, 96 (1974) 726.

Naphthalene—tetrakis(silver perchlorate) tetrahydrate, $C_{10}H_8(AgClO_4)_4 \cdot 4H_2O$ ($P\bar{1}$), $Z = 1$, $R = 6.1\%$ from 1796 reflections. The structure is comprised of parallel series of sheets in the order of aromatic—$AgClO_4$—water—water—$AgClO_4$—aromatic—. Each naphthalene is associated with four different hydrated silver ions with equal Ag—C bonds, 2.61(1) Å, to each of the α,β positions.

E.A. Hall Griffith and E.L. Amma, *J. Amer. Chem. Soc.*, 96 (1974) 743.

Dicyclopentadienylhexacarbonyldichromium, $(\eta^5\text{-}C_5H_5)_2Cr_2(CO)_6$ ($P2_1/c$), $Z = 2$, $R = 2.7\%$ using 1269 reflections. The molecule has a crystallographic centre of symmetry and has virtual symmetry of C_{2h}. Each half or the molecule has a pseudo-square-pyramidal configuration with $(\eta^5\text{-}C_5H_5)$ occupying the apical position and the three CO ligands and the Cr—Cr bond completing the square base. Cr—Cr, 3.281(1); Cr—CO(ave.), 1.86; Cr—Cp, 2.20 Å.

R.D. Adams, D.E. Collins and F.A. Cotton, *J. Amer. Chem. Soc.*, 96 (1974) 749.

(Diethyldi-1-pyrazolylborato)(trihapto-2-phenylallyl)(dicarbonyl)molybdenum, $[Et_2B(pz)_2][\eta^3\text{-}CH_2C(Ph)CH_2](CO)_2Mo$ ($P2_12_12_1(D_2^4)$), $Z = 4$, $R = 4.4\%$ from 2397 independent reflections. This structure shows unequivocally an interaction between an aliphatic C—H bond and a metal atom. The α-carbon—Mo distance is 3.07 Å and H· · ·Mo lies between 2.15 and 2.27(8) Å.

F.A. Cotton, T. LaCour and A.G. Stanislowski, *J. Amer. Chem. Soc.*, 96 (1974) 754.

Ferrocenediphenylcyclopropenium tetrafluoroborate, $C_{25}H_{19}FeBF_4$ ($P2_1/c$), $Z = 4$, $R = 5.7\%$ for 2003 reflections. The cp rings are eclipsed and are offset from parallelism by 2.7°. The three rings attached to the cyclopropenium ring are twisted propeller-like. The structure consists of discrete $C_{25}H_{19}Fe$ cations — a stable carbonium ion.

R.L. Sime and R.J. Sime, *J. Amer. Chem. Soc.*, 96 (1974) 892.

Perchloratotetraphenylporphinatozinc(II), $ZnTPPClO_4$ ($P2_1/c$), $z = 4$, $R = 7.3\%$ for 2244 reflections. The ClO_4 ion is covalently coordinated to Zn with Zn—O, 2.079(8) Å and < Zn—O—Cl, 130.2(5)°. The porphyrin nitrogens form the base of a square pyramid with the Zn 0.347(9) Å above this plane toward the ClO_4^-. Zn—N(mean), 2.076. A radical porphyrin species is involved.

L.D. Spaulding, P.G. Eller, J.A. Bertrand and R.H. Felton, *J. Amer. Chem. Soc.*, 96 (1974) 982.

Benzene-1,2-dithiolene-di(π-cyclopentadienyl)tungsten(VI), $(C_5H_5)_2WS_2C_6H_4$ ($Pnma$), $Z = 4$, $R = 6.0\%$ for 2082 independent reflections. The coordination around W is a distorted tetrahedron (nearly C_{2v}), defined by the cp ring

centroids and the sulphur atoms. The $S_2C_6H_4$ plane is inclined to the WS_2 plane at an angle of 8°. W—S, 2.421(2); W—cp, 2.004 Å.
T. Debaerdemaeker and A. Kutoglu, *Acta Crystallogr.*, *Sect. B*, 29 (1973) 2664.

μ-Oxo-bis[bis-(*N*-*p*-chlorophenylsalicylaldiminato)iron(III)], $C_{52}Cl_4Fe_2N_{36}N_4O_5$
($Pna2_1$), $Z = 4$, $R = 9.2\%$ for 2146 independent reflections. Both iron atoms are five-coordinate with a stereochemistry intermediate between trigonal-bipyramidal (the two nitrogen atoms apical) and square-pyramidal (the two oxygen bridge atoms apical). The nitrogen atoms are *trans* to each other. Fe—N, 2.11(2); Fe—O, 1.92(1) Å. The Fe—O—Fe bridge is symmetrical with Fe—O(bridge), 1.76 Å, and < Fe—O—Fe, 175°.
J.E. Davies and G.M. Gatehouse, *Acta Crystallogr.*, *Sect. B*, 29 (1973) 2651.

Di-μ-(*N*,*N*-diethylnicotinamide-*O*,*N*)tetraisothiocyanatozinc, $Zn_2(C_{10}H_{14}N_2O)_2(NCS)_4$ or $Zn_2(DENA)_2(NCS)_4$
($P\bar{1}$), $Z = 1$, $R = 4.5\%$ for 2767 observed reflections. The structure consists of centrosymmetric dimers with tetrahedral coordination around Zn. The two tetrahedra of the dimer are held together by pyridine N and amine O bridged of *N*,*N*-diethylnicotinamide molecules. Zn—NCS, 1.921(5) and 1.929(4); Zn—N(pyridine), 2.006(5); Zn—O, 2.014(4) Å.
F. Bigoli, A. Braibanti, M.A. Pellinghelli and A. Tiripicchio, *Acta Crystallogr.*, *Sec. B*, 29 (1973) 2708.

Tris-acetylacetonatoruthenium(III), $(C_5H_7O_2)_3Ru$ or $(tris\text{-}acac)_3Ru$
($P2_1/c$), $Z = 5$, $R = 9.6\%$ for 1317 independent reflections. The ruthenium atom is surrounded octahedrally by six oxygen atoms. Ru—O(ave.), 2.00 Å.
G.K. Chao, R.L. Sime and R.J. Sime, *Acta Crystallogr. Sect. B*, 29 (1973) 2845.

Copper fluorosilicate hexahydrate, $CuSiF_6 \cdot 6H_2O$
($R\bar{3}$), $Z = 4$, $R = 4\%$ for 592 independent reflections. The structure is made up of discrete $Cu(H_2O)_6^{2+}$ and SiF_6^{2-} octahedra, stacked alternately along the direction of the three-fold axis. SiF_6^{2-} octahedra are regular with Si—F, 1.676 ± 0.003; $Cu(H_2O)_6^{2+}$ are tetragonally distorted in half of the columns, with Cu—O(regular), 2.074(7); Cu—O(irregular), 1.970(7) Å.
S. Ray, A. Zalkin and D.H. Templeton, *Acta Crystallogr.*, *Sect. B*, 29 (1973) 2748.

Ethylene-1,2-dithiolato-di(π-cyclopentadienyl)titanium(VI), $(C_5H_5)_2TiS_2(CH)_2$
($Pca6$), $Z = 8$, $R = 5.6\%$ for 2018 independent reflections. The plane TiS_2 is folded out of the $S_2C_2H_2$ plane at an angle of 46.1°. Ti is tetrahedrally coordinated by the two S atoms and cp centroids. Ti—S, 2.403(1); 2.430(1); Ti—cp, 2.375 Å.
V.A. Kutoglu, *Acta Crystallogr.*, *Sect. B*, 29 (1973) 2891.

Rubidium hydrogen oxydiacetate, $RbHO(CH_2COO)_2$

($I\bar{4}2d$), $Z = 8$, $R = 2.8\%$ for 1468 reflections. The structure has infinite chains of oxydiacetate ions linked by short hydrogen bonds. The chains are cross-linked by Rb ions. H···O, 1.23; O—O, 2.45 Å.

J. Albertson, I. Grenthe and H. Herbertsson, *Acta Crystallogr., Sect. B*, 29 (1973) 2839.

Fluorosilicate hexahydrates of cobalt, nickel and zinc, $NiSiF_6 \cdot 6H_2O$ (A), $CoSiF_6 \cdot 6H_2O$ (B) and $ZnSiF_6 \cdot 6H_2O$ (C)

($R\bar{3}$), A, B and C are isomorphous. $Z = 3$, $R = 2.2\%$ (A), 3.3% (B) and 1.8% (C). The structures are essentially the same with $M(H_2O)_6^{2+}$ (M = Co, Ni or Zn) and SiF_6^{2-} octahedra. There is disorder of F. Packing is approximately CsCl-type. The hydrogen atoms take part in hydrogen bonds with O—F distances of 2.68—2.88 Å. Co—O, 2.081; Ni—O, 2.047; Zn—O, 2.078; Si—F-(ave.), 1.676 Å.

S. Ray, A. Zalkin and D.H. Templeton, *Acta Crystallogr., Sect. B*, 29 (1973) 2741.

Tetranitratobis(triphenylphosphine oxide)thorium(IV), $Th(NO_3)_4[(C_6H_5)_3PO]_2$

($P2_1/n$), $Z = 4$, $R = 10.1\%$ for 3933 independent reflections. The Th atom has a coordination number of ten, involving eight oxygen atoms from four-bidentate NO_3^-, and two oxygen atoms from $(C_6H_5)_3P$—O. Th—O($(C_6H_5)_3$-P—O), 2.37(2) and 2.33(2); Th—O(nitrate)(mean), 2.55(2) Å. NO_3^- groups are planar. P—O, 1.45(2); P—C, 1.80(3) Å.

K.M.A. Malik and J.W. Jeffrey, *Acta Crystallogr., Sect. B*, 29 (1973) 2687.

Hydrazinium fluoroberyllate, $(N_2H_5)BeF_4$

($P2_1/c$), $Z = 4$, $R = 4.3\%$ for 770 reflections. The structure consists of BeF_4^{2-} tetrahedra (Be—F(mean), 1.547 Å) and $N_2H_6^{2+}$ ions linked by hydrogen bonds.

M.R. Anderson, S. Vilminot and I.D. Brown, *Acta Crystallogr., Sect. B*, 29 (1973) 2961.

β-Ethylenediaminetetraacetic acid, β-EDTA

($C2/c$), $Z = 4$, $R = 5.6\%$ for 1020 reflections. Two structure determinations are compared.

M.F. Ladd and D.C. Povey, *Acta Crystallogr., Sect. B*, 29 (1973) 2973.

$VOSO_4 \cdot 3H_2O$

($P2_1/c$), $Z = 4$, $R = 10.7\%$ for 686 reflections. The structure consists of molecular units built with two $[SO_4]$ tetrahedra and two $[VO_6]$ octahedra sharing corners; the blocks are linked by hydrogen bonds.

F. Theobold and J. Galy, *Acta Crystallogr., Sect. B*, 29 (1973) 2735.

Bis(5,5'-diethylbarbiturato)bispyridine copper(II), $CuC_{26}H_{32}N_6O_6$ or Cu^{II} (5,5'-diethylbarbiturate)$_2$(pyridine)$_2$

($P2_1/c$), $Z = 2$, $R = 3.6\%$ for 1236 independent reflections. Cu is bonded to the deprotonated nitrogen atoms of the barbital anions, (Cu—N, 1.983(5)) and the pyridine nitrogens (Cu—N, 2.032(5) Å). There is weak coordination (off the Z axis) by oxygen from barbital (Cu—O, 2.722(5)). The molecules are joined by relatively short (2.86 Å) N—H···O hydrogen bonds.

M.R. Caira, G.V. Fazakerley, P.W. Linder and L.R. Nassimbeni, *Acta Crystallogr. Sect. B*, 29 (1973) 2899.

Hexakis[tris(p-tolyl)phosphine]-*octahedro*-hexagold bis(tetraphenylborate), $[C_{126}H_{126}Au_6P_6][C_{24}H_{20}B]_2$

($P\bar{1}$), $Z = 1$, $R = 5.2\%$ from 3501 independent reflections. The hexanuclear dication is a distorted centrosymmetric octahedron. Two mutually opposite faces have edges ranging from 2.932(2) to 2.990(2), the remaining edges ranging from 3.043(2) to 3.091(2) Å. Mean Au—Au, 3.019 Å.

P. Bellon, M. Manassero and M. Sansoni, *J. Chem. Soc. Dalton*, (1973) 2423.

Dithiocyanatobis(triphenylphosphine)mercury(II), $Hg(SCN)_2(PPh_3)_2$

($P2_1/c$), $Z = 4$, $R = 4.8\%$ for 2769 observed reflections. The compound is monomeric with the Hg in a distorted tetrahedral environment. Bond lengths are: Hg—S, 2.565 and 2.577; Hg—P, 2.487 and 2.484 Å.

R.C. Makhija, A.L. Beauchamp and R. Rivest, *J. Chem. Soc. Dalton*, (1973) 2447.

Dicarbonyl{hydrotris(pyrazol-1-yl)borato-$N(2)$,$N(2)'$,$N(2)''$}-π-(2-methylallyl)-molybdenum, $C_{15}H_{17}BMoN_6O_2$

($P2_1/a$), $Z = 4$, $R = 3.3\%$ for 3325 independent reflections. The octahedral coordination sphere of Mo consists of one nitrogen from each of the three pyrazolyl rings of the pyrazolylborate ligand, two C atoms from carbonyls, and a π-2-methylallyl group. Mo—C(2-methylallyl), 2.337(5), 2.258(4) and 2.364(4) Å. M—CO, 1.958(4); Mo—N, 2.312(3), 2.207(3) and 2.283(4) Å.

E.M. Holt, S.L. Holt and K.J. Watson, *J. Chem. Soc. Dalton*, (1973) 2444.

catena-μ-Acetato-[N,N'-ethylenebis(salicylaldiminato)] manganese(III), Mn(salen)(AcO)

($P2/c$), $Z = 2$, $R = 7.3\%$ for 1211 observed reflections. The structure consists of linear polymeric chains of Mn(salen) moieties bridged by single acetate groups in an *anti—anti* configuration. Mn—N, 1.989(5); Mn—O, 1.888(4); Mn—O(acetate), 2.201(5) Å.

J.E. Davies, B.M. Gatehouse and K.S. Murray, *J. Chem. Soc. Dalton*, (1973) 2523.

Di-μ-chloro-bis[di-η-cyclopentadienylscandium(III)], $[Sc(C_5H_5)_2Cl]_2$

($P2_1/c$), $Z = 6$, $R = 7.2\%$ for 1680 observed reflections. The molecules exist as chlorine-bridged dimers, Sc—Cl(mean), 2.575 Å. The cp groups are bonded in a penta-*hapto*-fashion, Sc—C(mean), 2.46 Å.

J.L. Atwood and K.D. Smith, *J. Chem. Soc. Dalton*, (1973) 2487.

Bis(2-hydroxy-*N*-methyl-1-naphthylmethyleneiminato)copper(II), $C_{24}H_{20}$-CuN_2O_2

($P2_1/c$), $Z = 2$, $R = 9.7\%$ for 1024 reflections. The copper is strictly square-planar, with Cu 0.19 Å out of the plane. Cu—O, 1.87(1); Cu—N, 1.98(1) Å.

D.W. Martin and T.N. Waters, *J. Chem. Soc. Dalton*, (1973) 2440.

A new metasilicate, $Na_2BaSi_2O_6$

($P2_1$), $Z = 2$, $R = 6.0\%$ for 839 observed independent reflections. The structure contains infinite SiO_3 chains parallel to *b* linked through five- and six-coordinate Na and eight-coordinate Ba atoms.

R.P. Gunawardane, M.E. Cradwick and L.S. Dent Glasser, *J. Chem. Soc. Dalton*, (1973) 2397.

Trifluoro-oxosulphur(VI) hexafluoroarsenate(V), $OSF_3^+AsF_6^-$

(*Pnma*), $Z = 4$, $R = 4.6\%$ for 331 reflections. The structure contains discrete OSF_3^+ and AsF_6^- ions. OSF_3^+ exists as a distorted tetrahedron: S—O, 1.35(1); S—F, 1.45 ± 0.01 Å.

C. Lau, H. Lynton, J. Passmore and Pik-Yuen Siew, *J. Chem. Soc. Dalton*, (1973) 2535.

Chlorodifluoro-oxosulphur(VI) hexafluoroarsenate(V), $OSClF_2^+AsF_6^-$

($P2_1/n$), $Z = 4$, $R = 4.5\%$ for 304 observed reflections. The structure contains discrete $OSClF_2^+$ and AsF_6^- ions. S—Cl, 1.86(1) Å. Cl···F(AsF_5^-), 2.67(2) Å.

R.F. Dunphy, C. Lau, H. Lynton and J. Passmore, *J. Chem. Soc. Dalton*, (1973) 2533.

μ-Oxalato-bis[(di-*n*-propyl sulphoxide)nitratodiphenyltin(IV)], $(C_{19}H_{24}NO_6$-$SSn)_2$

($P\bar{1}$), $Z = 1$, $R = 4.5\%$ for 3083 independent reflections. The coordination around Sn^{IV} is pentagonal-bipyramidal, the NO_3^-, $C_2O_4^{2-}$ anions and SO_2 being equatorial, with the two phenyl rings at the apices. The oxalate ion acts as a centrosymmetric bridge between two coordination polyhedra. Sn—O-(oxalato), 2.282(5), 2.248(5); Sn—O(SO_2), 2.178(6); Sn—O(NO_3^-), 2.388(6) and 2.408(6); Sn—C, 2.115(7) Å.

A. Mangia, C. Pelizzi and G. Pelizzi, *J. Chem. Soc. Dalton*, (1973) 2557.

Dicarbonylchlorobis[*o*-phenylenebis(dimethylarsino)]molybdenum(II) triiodide—bischloroform, $[Mo(diars)_2(CO)_2Cl]^+I_3^-$, $2CHCl_3$

(*Pnma*); $Z = 4$, $R = 8.0\%$ from 1048 independent reflections. The Mo has a 1,4,2-capped trigonal-prismatic environment with a chlorine atom in the unique capping position (2.575(11) Å), four arsenic atoms in the capped quadrilateral face (2.614(5), 2.617(5) Å), and two carbonyl groups making up the remaining edge (1.92(5), 1.87(5) Å). I—I, 2.903(2); < I—I—I, 176.9(2)°.

M.G.B. Drew and J.D. Wilkins, *J. Chem. Soc. Dalton*, (1973) 2664.

[Octakisdimethylaminocyclotetraphosphazene] tetracarbonyltungsten, $N_4P_4(NMe_2)_8 \cdot W(CO)_4$
($P2_1/n$), $Z = 4$, $R = 7.1\%$ for 2372 observed reflections. The phosphonitrile is coordinated to W through a ring nitrogen atom and an oxocyclic dimethylamido-nitrogen atom. The coordination around W is distorted octahedral with *cis* nitrogens, < N—W—N, 65.4°. W—N, 2.33(2), 2.37(1); W—C, 1.88(3)—2.11(4) Å.
H.P. Calhoun, N.L. Paddock and J. Trotter, *J. Chem. Soc. Dalton*, (1973) 2708.

Bis(dithioformato)bis(triphenylphosphine)ruthenium(II), $Ru(HCS_2)_2(PPh_3)_2$
($P2_1/c$), $Z = 4$, $R = 5.9\%$ for 4012 independent reflections. The molecule has distorted octahedral coordination, with mutually *cis* thioformate groups. Ru—P, 2.357(2) and 2.335(3); Ru—S, 2.386(3), 2.445(3), 2.386(3) and 2.448(3) Å.
R.O. Harris, L.S. Sadavoy, S.C. Nyburg and F.H. Pickard, *J. Chem. Soc. Dalton*, (1973) 2646.

Aquobis(*N,N*-dimethylglycinato)copper(II) dihydrate, $C_8H_{22}CuN_2O_7$
($P2_12_12_1$), $Z = 4$, $R = 12\%$ for 1111 independent reflections. The copper atom is five-coordinate, a square pyramid with H_2O as the apical ligand, Cu—N(mean), 2.05; Cu—O, 1.97; Cu—OH_2, 2.28 Å. The basal ligands are in the *trans* configuration.
T.S. Cameron, K. Prout, F.J.C. Rossotti and D. Steele, *J. Chem. Soc. Dalton*, (1973) 2626.

Di-μ-hydroxo-*trans*-diaquo-bis[triamminecobalt(III)] tetranitrate dihydrate, $[Co_2(OH)_2(H_2O)_2(NH_3)_6](NO_3)_4 \cdot 2H_2O$
($P2_1/n$), $Z = 2$, $R = 3.2\%$ for 2248 independent reflections. The H_2O molecules coordinated to the Co atoms are *trans*. Each Co atom has octahedral geometry, with bridging OH groups and two NH_3 ligands in the equatorial planes. Co—O(OH)(bridging), 1.905; Co—N(equatorial), 1.953; Co—N(axial), 1.922 and 1.939; Co—O(OH_2), 1.938(3); Co—Co, 2.880 Å.
W.H. Baur and K. Wieghardt, *J. Chem. Soc. Dalton*, (1973) 2669.

β-Tetrakis(thiourea)mercury(II) chloride, $HgCl_2(tu)_4$
($P2_1/a$), $Z = 4$, $R = 9.6\%$ for 1203 independent reflections. The cell contains discrete $[Hg(CS(NH_2)_2)_4]^{2+}$ and Cl^- units, the mercury atom being in a highly distorted tetrahedron of sulphur atoms (Hg—S, 2.51—2.62 Å; < S—Hg—S, 112—117°)
P.D. Brotherton and A.H. White, *J. Chem. Soc. Dalton*, (1973) 2696.

Tetra-μ-acetato-bisaquocopper(II), $[Cu_2(CH_3CO_2)_4H_2O]$
($C2/c$), $Z = 4$, $R = 4.7\%$ for 1280 independent reflections. The structure is dimeric, Cu—O(mean)(acetate), 1.969; Cu—O(H_2O), 2.156, and Cu···Cu, 2.616 Å. The geometry of the acetate bridges is planar.
P. de Meester, S.R. Fletcher and A.C. Skapski, *J. Chem. Soc. Dalton*, (1973) 2575.

A new Hg^{II} complex, dichloromercury—$\frac{2}{3}$ thiourea, $[HgCl_2 . \frac{2}{3}\ tu]$
($P2_1/c$), $Z = 6$, $R = 10\%$ for 408 visually estimated reflections. The complex is effectively $[\{HgCl(tu)\}Cl]_n$, $n/2\ HgCl_2$, and consists of Hg atoms coordinated by thiourea (Hg—S, 2.40 Å) and chlorine (Hg—Cl, 2.39 Å; < S—Hg—Cl, 155°); a further chlorine bridges these units in an infinite chain (Hg—Cl, 2.84 and 2.94 Å) parallel to *c*. Discrete $HgCl_2$ molecules, centres on special positions, occupy interstices between the chains.
P.D. Brotherton and A.H. White, *J. Chem. Soc. Dalton*, (1973) 2698.

Dicarbonyl(π-cyclopentadienyl) [di-*tert*-butylmethyleneamino] molybdenum(II), $(\pi\text{-}C_5H_5)Mo(CO)_2(N{:}CBu^t_2)$
($P2_1/n$), $Z = 4$, $R = 3.3\%$ for 1534 observed reflections. The coordination about Mo is a distorted octahedron with the cp occupying three sites. The other three sites are occupied by two mutually *cis* CO groups and the $(N{:}CBu^t_2)$ ligand Mo—N, 1.892(5); Mo—CO, 1.957 and 1.966 Å; < Mo—N—C, 171.8°.
H.M.M. Shearer and J.D. Sowerby, *J. Chem. Soc. Dalton*, (1973) 2629.

Hexaaquo tris-malonato di-neodymium(III) dihydrate, $Nd_2(C_3H_2O_4)\cdot 8H_2O$
($Pbcn$), $Z = 4$, $R = 9.2\%$ for 1876 independent reflections. Each Nd is coordinated by six carboxylate and three water oxygens which form a monocapped square antiprism. Nd—O, 2.41—2.72 Å. The structure contains bridged malonate ions and other malonate ions which form six-membered chelate rings. The H_2O molecules are hydrogen bonded with O—O, 2.63—2.85 Å.
E. Hansson, *Acta Chem. Scand.*, 27 (1973) 2441.

Zn^{II} (+)-aspartate trihydrate
($P2_12_12_1$), $Z = 4$, $R = 2.5\%$ using 1279 reflections. the absolute configuration has been determined. L-(+)-Aspartic acid has the *S* configuration. Zn—N, 2.077; Zn—O, 2.106 and 2.173 Å.
L. Kryger and S.E. Rasmussen, *Acta Chem. Scand.*, 27 (1973) 2674.

$Hf(OH)_2SO_4$
($Pnma$), $Z = 4$, $R = 5.5\%$ for 405 independent reflections. The structures contains infinite chains of $(Hf(OH)_2]^{2n+}_n$ which are joined by SO_4^{2-}, each of which connects three chains. Hf—Hf, 3.562 Å. Hafnium exhibits eight-fold oxygen coordination, the O atoms forming a square antiprism. Hf—O(ave.), 2.17 Å.
M. Hansson, *Acta Chem. Scand.*, 27 (1973) 2455.

$Zr(OH)_2SO_4H_2O$
($C2/c$), $Z = 4$, $R = 6.0\%$ using 986 independent reflections. The structure consists of almost planar, infinite $[Zr(OH)_2]^{2n+}_n$ chains, joined in one direction by SO_4^{2-} groups. Zr exhibits sevenfold oxygen coordination in the form of a distorted pentagonal bipyramid. Zr—O(ave.), 2.13(8) Å)
M. Hansson, *Acta Chem. Scand.*, 27 (1973) 2614.

$[Pb_4(OH)_4]_3(CO_3)(ClO_4)_{10} \cdot 6H_2O$
($P6_3/m$), $Z = 2$, $R = 6.0\%$ for 1095 reflections. The crystal contains discrete $[Pb_4(OH)_4^{4+}]$ units. The four lead atoms occupy the corners of a slightly distorted tetrahedron and the $(OH)^-$ groups are located outside the faces of this tetrahedron. Pb—Pb, 3.76; Pb—O, 2.29—2.49 Å.
Sam-Hyo Hong and A. Olin, *Acta Chem. Scand.*, 27 (1973) 2309.

Pentaaquo(trismalonato)dieuropium(III) trihydrate, $Eu_2(C_3H_2O_4)_3 \cdot 8H_2O$
(*Pnma*), $Z = 4$, $R = 9.1\%$ for 692 visually observed independent reflections. There are two non-equivalent Eu^{III} ions, and three non-equivalent malonate ions in the structure. They are linked to Eu—malonate layers which are in turn held together by hydrogen bonds via water molecules between the layers. One Eu is eight-coordinate, with oxygens forming a distorted square antiprism, the other is nine, with oxygens forming a distorted tricapped trigonal prism. Eu—O(ave.), 2.42 and 2.51 Å respectively.
E. Hansson, *Acta Chem. Scand.*, 27 (1973) 2827.

Positions of the water molecules in hexaaquo(trisoxalato)dineodymium(III) 4½ hydrate at —50° C, $Nd_3(C_2O_4)_3 \cdot 10\frac{1}{2}\, H_2O$
($P2_1/c$), $Z = 4$, $R = 12.0\%$ for 756 visually observed reflections. Several new water positions with occupancies in the range 0.3—0.5 have been found at —50° C. They are located in cavities in the structure, and are within hydrogen bonding distance (2.7—3.2 Å) from each other and from the oxygens surrouding the cavity.
E. Hansson, *Acta Chem. Scand.*, 27 (1973) 2852.

Hexaaquo(trismalonato)dineodymium(III), $Nd_2(C_3H_2O_4)_3 \cdot 6H_2O$
($I2/a$), $Z = 4$, $R = 9.4\%$ for 1520 reflections. The structure is a three-dimensional Nd—malonate network. Nd^{III} is coordinated by six carboxylate and three water oxygens forming a distorted monocapped square antiprism. Nd—O, 2.35—2.61 Å. The water molecules form hydrogen bonds with O—O, 2.74—2.85 Å.
E. Hansson, *Acta Chem. Scand.*, 27 (1973) 2813.

Monoaquo hydroxo malonato scandium(III) monohydrate, $Sc(OH)C_3H_2O_4 \cdot 2H_2O$
($P2_1/n$), $Z = 4$, $R = 8.1\%$ for 779 independent reflections. The structure has infinite scandium—hydroxo—malonate chains running in the x direction and linked to each other by hydrogen bonds. Each Sc^{III} is octahedrally surrounded by two hydroxy oxygens, three carboxylate oxygens and one water oxygen. Sc—O, 2.05—2.12 Å.
E. Hansson, *Acta Chem. Scand.*, 27 (1973) 2841.

Tetraaquothiodiacetatoneodymium(III) chloride, $Nd(C_4H_4O_4S)(H_2O)_4^+Cl$
(*Pnnm*), $Z = 4$, $R = 12.7\%$ for 416 reflections. The Nd^{III} ion is surrounded by

four carboxylate oxygen atoms, four water molecules, and one sulphur atom, which form a distorted tricapped trigonal prism. Nd—O, 2.31—2.53; Nd—S, 3.15 Å.

T. Malmborg and A. Oskarsson, *Acta Chem. Scand.*, 27 (1973) 2923.

An unusual trinuclear ferracyclobutene, $Fe_3(CO)_8[Ph_2PC_4(CF_3)_2](PPh_2)$

($P\bar{1}$), $Z = 2$, $R = 7.9\%$ for 4059 observed reflections. The structure contains a *trans*-butadiene unit with two of its carbon atoms coordinated to Fe(3) forming a nearly planar ferracyclobutene ring. Fe—C, 2.021(13) and 1.959(12); Fe(3)—Fe(2), 2.514(3) Å. Fe(2) is also π-bonded to the *trans*-butadiene.

M. Mathew, G.J. Palenik, A.J. Carty and H.N. Paik, *J. Chem. Soc. Chem. Commun.*, (1974) 25.

$Ru(NPEt_2Ph)Cl_3(PEt_2Ph)_2$; a complex with a coordinated tertiary phosphineiminato(1—) ion.

($P2_12_12_1$), $Z = 4$, $R = 3.5\%$ for 3591 independent reflections. The Ru atom has a distorted octahedral coordination. Ru—P(mean), 2.426; Ru—Cl(*trans* to Cl)(mean), 2.388; Ru—Cl(*trans* to N), 2.398 Å. The phosphineiminato ligand is essentially linear, < Ru—N—P, 175.0(4)°; P—N, 1.571; Ru—N, 1.855 Å.

F.L. Phillips and A.C. Skapski, *J. Chem. Soc. Chem. Commun.*, (1974) 49.

Aziridinylmethyltetracarbonylmanganese

($P2_1/n$), $Z = 4$, $R = 7.8\%$ for 1314 independent reflections. There is essentially symmetric π-bonding fo the aminomethylene ligand. Mn—C, 2.09; Mn—N, 1.98; Mn—CO(ave.), 1.80 Å.

E.W. Abel, R.J. Rowley, R. Mason and K.M. Thomas, *J. Chem. Soc. Chem. Commun.*, (1974) 72.

Dicyclopentadienylmagnesium and dicyclopentadienylchromium, $(C_5H_5)Mg$ and $(C_5H_5)_2Cr$

The structures have been determined by gas-phase electron diffraction; the C—H bonds in $(C_5H_5)_2Cr$ are bent 2.9 Å (1.1°) out of the plane of the C_5 ring toward the metal atom.

A. Haaland, J. Lusztyk, D.P. Novak, J. Brunvoll and K.B. Starowieyski, *J. Chem. Soc. Chem. Commun.*, (1974) 54.

π-Cyclopentadienyl-1,2-bis(diphenylphosphino)ethaneironmagnesium bromide tris(tetrahydrofuran) — a transition metal Grignard reagent — $(\pi\text{-}C_5H_5)$(diphos)-Fe—MgBr, 2(THF)

($P2_1/c$), $Z = 4$, $R = 8\%$ for 1505 independent reflections. The $(\pi\text{-}C_5H_5)$-(diphos)Fe fragment is directly linked to the MgBr, 2(THF) fragment. Fe—Mg 2.593(7) Å. Mg has a distorted tetrahedral environment. < Fe—Mg—Br, 129.2°; < Fe—Mg—O, 119.0(5)° and 115.8(6)°; < O—Mg—O, 91.7(9)°. Mg—Br, 2.522(7); Mg—O, 2.075(15) and 2.098(15) Å.

H. Felkin, P.J. Knowles, B. Meunier, A. Mitschlier, L. Ricard and R. Weiss, *J. Chem. Soc. Chem Commun.*, (1974) 44.

Tricarbonyl(1-keto-π-2,3,4-triphenylcyclobutenyl)cobalt, $(C_6H_5)_3C_3COCo(CO)_3$

($P2_1/a$), $Z = 8$, $R = 9.9\%$ from 2016 independent reflections. Each cobalt atom is bonded to three terminal carbonyls and to a keto-π-cyclobutenyl ligand. Co—CO(ave.), 1.83 Å. The cyclobutenyl rings are symmetrically coordinated to Co via allyl carbon atoms (Co—C, 2.121 and 2.135 Å), the other ring carbons are exo with respect to Co (Co—C, 2.440 (2.370) Å).

J. Potenza, R. Johnson, D. Mastropaolo and A. Efraty, *J. Organometal. Chem.*, 64 (1974) C13.

Cyclopentadienyliron nitrosyl dimer, $[(h^5\text{-}C_5H_5)Fe(\mu\text{-}NO)]_2$

($P2_1/c$), $Z = 2$, $R = 6.3\%$ for 574 independent reflections. The two iron atoms are bonded to each other, symmetrically bridged by two crystallographically equivalent NO groups, and also pentahapto-bonded to a cyclopentadienyl ring. $Fe(\mu\text{-}NO)_2Fe$ is virtually coplanar. Fe—C(ave.), 2.09(1); Fe—N(ave.), 1.768(9); N—O, 1.254(12); Fe—Fe, 2.325(4) Å; < Fe—N—O, 138.8(8)°.

H.L. Calderon, S. Fontana, E. Frauendorfer, V.W. Day and S.D.A. Iske, *J. Organometal. Chem.*, 64 (1974) C16.

$[Ph_4As]^+[Mn_3CO)_{14}]^-$

($P\overline{1}$), $Z = 2$, $R = 9.2\%$ for 1414 non-zero reflections. The $[Mn_3(CO)_{14}]^-$ anion has essentially D_{4h} symmetry with the three manganese atoms collinear. The equatorial CO groups on the terminal manganese are staggered with respect to those on the central manganese and are bent away from the axial CO groups. Mn—Mn(ave.), 2.895; M—C(axial), 1.758; M—C(equatorial), 1.827 Å.

R. Bau, S.W. Kirtley, T.N. Sorrell and S. Winarko, *J. Amer. Chem. Soc.*, 96 (1974) 988.

Bis(indenyl)magnesium, $(C_9H_7)_2Mg$

($P2_12_12_1$), $Z = 8$, $R = 6.6\%$ for 1112 observed reflections. The structure exhibits magnesium atoms in two different environments: one is bonded to the five-membered portion of an indenyl ring in a pentahapto fashion and to two others through essentially only one carbon atom; the second magnesium atom is coordinated to one ring in a pentahapto fashion and to two others through two carbon atoms. There are both bridging and terminal indenyl groups. Mg—C(bridging), 2.26(1); Mg—C(pentahapto), 2.43 Å.

J.L. Atwood and K.D. Smith, *J. Amer. Chem. Soc.*, 96 (1974) 994.

$BaMnS_2$

($Pnma$), $Z = 4$. MnS_4 tetrahedra linked by corners form waved layers, connected by barium ions.

V.D. Schmitz and W. Brouger, *Z. Anorg. Allg. Chem.*, 402 (1973) 225.

X-ray studies of the interaction of CO_2 with human deoxyhaemoglobin
This study confirms that CO_2 reacts with the N-terminal amino groups of the β chains to form carbamate ions.
A. Arnone, *Nature (London)*, 247 (1974) 143.

Dinitrato(triphenylphosphine)mercury(II), $Hg(NO_3)_2 \cdot P(C_6H_5)_3$
($P2_1/c$), $Z = 4$, $R = 6.1\%$ from 2470 independent observed reflections. The Hg has a distorted tetrahedral coordination with Hg—O, 2.190(8), 2.428(8) and 2.590(9); Hg—P, 2.359(3) Å. There are two distinct types of NO_3 groups. One is an unshared, unidentate ligand, the other acts as a bridging group joining two Hg atoms. Chains of $Hg—NO_3—Hg$ are formed.
S.H. Whitlow, *Can. J. Chem.*, 52 (1974) 198.