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X-RAY BIBLIOGRAPHY

M. KEETON

1260 Princess St., Apt. 89, Kingston, Ontario (Canada)

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Nitrilotriacetatodiaquopraseodymium(III) monohydrate

(*Pbca*) $Z = 8$, $R = 8.0\%$ for 1126 reflections. The metal is nonacoordinated with six carboxylate oxygens, two waters and one nitrogen atom in an arrangement described as a distorted tricapped trigonal prism, or a distorted capped square antiprism. Each nitrilotriacetate is heptadentate with one carboxylate oxygen coordinating to two adjacent metal atoms and two which coordinate to metal atoms in adjacent molecules. Pr–O 2.47(2), Pr–OH_2 2.52(2), Pr–N 2.68(2) Å.

L.L. Martin and R.A. Jacobsen, *Inorg. Chem.*, 11 (1972) 2785.

Nitrilotriacetatodiaquodysprosium(III) dihydrate

(*Pca*2_1) $Z = 2$, $R = 7.0\%$ for 3032 reflections. Both independent dysprosiums are octacoordinate with five acetate oxygens, two water molecules and one nitrogen at the corners of a distorted dodecahedron with triangular faces. Each nitrilotriacetato ligand is hexadentate with one acetate oxygen uncoordinated and two oxygens coordinated to metal atoms in adjacent molecules giving rise to a polymeric structure. Dy–OH_2 2.36(1), Dy–O 2.35(1), Dy–N 2.58(1) Å.

L.L. Martin and R.A. Jacobsen, *Inorg. Chem.*, 11 (1972) 2789.

trans-$P(OCH_2)_3PFe(CO)_3P(OCH_2)_3P$

(*Pnma*) $Z = 4$, $R = 8.9\%$ for 1313 reflections. The bond symmetry around the ion is nearly idealized trigonal bipyramidal. The $P(CH_2)_3$ bound ligand is eclipsed with respect to the trigonally arranged carbonyls, reducing steric hindrance.

D.A. Allison, J. Clardy and J.G. Verkade, *Inorg. Chem.*, 11 (1972) 2804.

Bis(6-*t*-butyl-1,3,5-trimethylcyclohexadienyl)iron(II)

($P2_1/c$) $Z = 2$, $R = 5.6\%$ for 1598 observed reflections. The π-pentadienyl angles and distances are normal. The final orientation of the π-pentadienyl system minimizes the steric interaction between the methyl groups while maximizing the number of bonds in the π system which are eclipsed.

M. Mathew and G.J. Palenik, *Inorg. Chem.*, 11 (1972) 2809.

Nitrosylcarbonylbis(triphenylphosphine)iridium, $Ir(NO)(CO)(P(C_6H_5)_3)_2$

($P2_1/c$) $Z = 4$, $R = 3.0\%$ for 2946 reflections. The coordination within the discrete molecules is distorted tetragonal. P–Ir–P 109.9(1)°, N–Ir–C 128.8(2°) and the dihedral angle between P–Ir–P and N–Ir–C planes is 84.2(2)°. The nitrosyl is coordinated to Ir almost linearly, Ir–N 1.787(8) Å, Ir–N–O 174.1(7)°. The complex is formulated as a NO^+ complex of Ir^{-I}.

C.P. Brock and J.A. Ibers, *Inorg. Chem.*, 11 (1972) 2812.

Diaquotri(nicotinic acid)holmium(III) hexa(isothiocyanate)chromate(III) dihydrate (A) and diaquotris(isonicotinato)lanthanum(III) (B)

Both are ($P2_1/c$) $Z = 4$, $R = 3.8\%$ for 2554 reflections (B) and 4.8% for 2989 reflections (A). Both compounds are made up of polymer chains of lanthanide(III) ions alternately linked by four and two bridging carboxylate groups. The eight-coordination around the metals is completed by two waters. In the acid complex, two more waters and one hexaisothiocyanatochromate ion are also present.

J. Kay, J.W. Moore and M.D. Glick, *Inorg. Chem.*, 11 (1972) 2818.

Carbonylbis(triphenylphosphine)(1,4-*p*-fluorophenyltetrazene)iridium tetrafluoroborate

($P\bar{1}$) $Z = 2$, $R = 4.9\%$ for 3252 reflections. In the complex cation there is a five-membered iridium nitrogen ring and the cation may be alternatively described as the 1-carbonyl-1,1-bis(triphenylphosphino)iridio-2,5-di-*p*-fluorophenyltetrazolium ion. The uncoordinated nitrogens are 1.270(16) Å apart and are essentially singly bonded to coordinated nitrogen atoms, 1.400(16) and 1.350(16) Å respectively.

F.W.B. Einstein and D. Sutton, *Inorg. Chem.*, 11 (1972) 2827.

Tris(cyclopentadienyl)indium(III), $In(C_5H_5)_3$, at −100°C

($P2_12_12_1$) $Z = 4$, $R = 3.6\%$ for 1587 observed reflections. The structure consists of infinite polymeric chains with each chain unit comprising an indium atom linked to two terminal and two bridging cyclopentadienyl groups which give rise to a slightly distorted $In-C_4$ tetrahedron. In–C 2.24(1) Å (terminal); 2.27(1) and 2.47(1) Å for bridging contacts.

F.W.B. Einstein, M.M. Gilbert and D.G. Tuck, *Inorg. Chem.*, 11 (1972) 2832.

Di-μ-oxo-bis(diacetylacetonatotitanium(IV))bisdioxane, $(TiO(C_5H_7O_2)_2)_2.2C_4H_8O_2$ (A) and di-μ-oxo-bis(diacetylacetonatotitanium(IV)), $(TiO(C_5H_7O_2)_2)_2$ (B)

($P\bar{1}$) $Z = 1$, $R = 9.7\%$ for 963 reflections (A) and ($P2_1/c$) $Z = 2$, $R = 5.0\%$ for 1549 reflections (B). The structures consist of a cyclic dimer with the titanium atom linked through oxygen atoms. The di-μ-oxo-dititanium ring is planar and slightly distorted from a square. The acetylacetonate rings are very nearly planar. The octahedral coordination around titanium is somewhat distorted. The solvent in (A) is loosely bound and is located in approximately spherical holes between dimer units.

G.D. Smith, C.N. Caughlin and J.A. Campbell, *Inorg. Chem.*, 11 (1972) 2989.

Chloro(bis{2-[(2-pyridylmethyl)amino]ethyl}disulphide)nickel(II) perchlorate, $Ni(C_{16}H_{22}N_4S_2)Cl(ClO_4)$

(*Pbca*) $Z = 8$, $R = 5.8\%$ for 4473 unique reflections. The metal is coordinated octahedrally by four nitrogen atoms, a chloride ion *cis* to a sulphur atom of the disulphide group. The two pyridine rings are *cis*. Ni–S 2.472(5) Å.

P.E. Riley and K. Seff, *Inorg. Chem.*, 11 (1972) 2993.

Bis(dithiocumato)platinum(II) dimer, $Pt_2(p\text{-dtc})_4$

Triclinic space group, $Z = 2$, $R_1 = 9.9\%$ for 3459 reflections. The dithiolate dimer shows a Pt–Pt distance of 2.870(2) Å in a distorted square antiprism of sulphur atoms. There are two bridging and two terminal dithiolate ligands accounting for the observed multiplicity of ligand bands in the vibration spectrum.

J.M. Burke and J.P. Fackler, Jr., *Inorg. Chem.*, 11 (1972) 3000.

Uranium borohydride (neutron diffraction)

($P4_32_12$) $Z = 4$. All hydrogens are located within 0.04 Å. The coordination geometry is that of a cupped hexagonal antiprism with distortions. Four of the six BH_4^- ions surrounding the uranium are attached to it by two hydrogen atoms and bridge to other uraniums through the other two hydrogens. Two more BH_4 groups in *cis* configuration are bonded to uranium by three hydrogens, resulting in a coordination number of 14.

E.R. Bernstein, W.C. Hamilton, T.A. Keiderling, S.J. LaPlace, S.J. Lippard and J.J. Mayerle, *Inorg. Chem.*, 11 (1972) 3009.

Bis(9-phenyl-9-phosphabicyclo[3.3.1]nonane)nickel chloride, $(C_8H_{14}PC_6H_5)_2NiCl_2$

($P\bar{1}$) $Z = 1$, $R = 3.9\%$ for 2900 reflections. The nickel atom occupies a site of inversion symmetry requiring the Cl and P atoms to be in a *trans*-square-planar configuration. The phosphorus is tetrahedrally coordinated with two of the bonds symmetrically bridging the cyclooctane molecule. P–C 1.834(3) and 1.831(3) Å.

A.E. Smith, *Inorg. Chem.*, 11 (1972) 3017.

Bromotricarbonylbis(methyl isocyanide) manganese, $Mn(CO)_3(CNCH_3)Br$

($P\bar{1}$) $Z = 2$, $R = 4.0\%$ for 831 independent reflections. The molecule has a nearly octahedral configuration with *cis* methyl isocyanide ligands. Mn–CO 1.854(12), 1.815(11) and 1.789(11) Å with the latter distance *trans* to bromine. $Mn-CNCH_3$ 1.992(11) and 1.950(12) and ∠ C–N–C does not deviate from linearity by more than 3°.

A.C. Sarapu and R.F. Fenske, *Inorg. Chem.*, 11 (1972) 3021.

1-Thia-4-telluracylohexane 4,4-dibromide, $C_4H_8STeBr_2$

($P2_1/c$) $Z = 4$, $R = 5.4\%$ for 1468 reflections. The molecule approximates mirror symmetry with the six-membered ring in the chair form. The bonding about tellurium is octahedral. Te–C 2.14(1) and 2.16(1); C–Te–C 99.4(6)°; Te–Br 2.657(2) and 2.689(2) Å; Br–Te–Br 176.63(6)°; Te–S 3.588(4) and Te–Br 3.591. The latter two contacts are between adjacent (different) molecules.

C. Knobler and J.D. McCullough, *Inorg. Chem.*, 11 (1972) 3026.

$[K((CH_3OCH_2CH_2)_2O)]\ [Ce(C_8H_8)_2]$

($Pnma$) $Z = 4$, $R = 5.9\%$ for 2789 independent reflections. The structure consists of discrete $[Ce(C_8H_8)_2]^-$ anions combined in a contact ion pair with the $[K((CH_3OCH_2CH_2)_2O)]^+$ solvated anion. The cyclooctatetraene rings are planar with equal aromatic C–C bonds (1.39(3) Å). The symmetry of the cation is D_{8d}.

K.O. Hodgson and K.N. Raymond, *Inorg. Chem.*, 11 (1972) 3030.

Nickel and cobalt dichlorobis(diarsine) monochlorides

Both are ($P2_1c$) $Z = 2$; $R = 3.3\%$ for 1424 reflections (Co) and $R = 3.8\%$ for 1500 reflections (Ni). The compounds are approximately isostructural. The monocation $M(diars)_2Cl_2$ (M = Ni or Co) is monomeric and centrosymmetric. The four arsenic atoms form a square planar around the metal and two chlorines complete a slightly distorted octahedron. Ni–As 2.345(2), Co–As 2.334(2), Ni–Cl 2.425(3), Co–Cl 2.256(3) Å.

P.K. Bernstein, G.A. Rodley, R. Marsh and H.B. Gray, *Inorg. Chem.*, 11 (1972) 3040.

Bromoazido-1,1,7,7-tetraethyldiethylenetriaminecopper(II), $Cu(Br)N_3(HN((CH_2)_2N(C_2H_5)_2)_2)$

($P\bar{1}$) $Z = 2$, $R = 5.0\%$ for 2494 non-zero reflections. The closest Cu–Cu approach is 6.395(1) Å. The metal is coordinated in a distorted trigonal bipyramidal with bromine equatorial and the azido group apical and approximately linear (N–N(average) 1.144(12) Å).

R.F. Ziolo, M. Allen, D.D. Titus, H.B. Gray and Z. Dori, *Inorg. Chem.*, 11 (1972) 3044.

3-Methylpyridine-tris(2,2,6,6-tetramethyl-3,5-heptanedionato)lutetium(III), $Lu(C_{11}H_{19}O_2)_3C_6H_7N$

($P2_1/c$) $Z = 4$, $R = 7.9\%$ for 6385 independent reflections. The lutetium ion is seven-coordinate, with the six oxygen atoms of the three diketo groups and the nitrogen atom of the 3-methylpyridine molecule occupying the vertices of a capped trigonal prism.

S.J. Schuchart Wasson, D.E. Sands and W.F. Wagner, *Inorg. Chem.*, 12 (1973) 187.

Azulenetriruthenium heptacarbonyl, $(C_{10}H_8)Ru_3(CO)_7$

($P2_1/c$) $Z = 8$, $R = 8.9\%$ for 2285 independent non-zero reflections. Within each molecule the three ruthenium atoms define an isosceles triangle with Ru(1)–Ru(2) ≡ Ru(1)–Ru(3) ≡ 2.937(4)–2.949(4) and Ru(2)–Ru(3) 2.740(4)–2.741(4) Å. Each ruthenium is bonded to two terminal carbonyl ligands with the seventh carbonyl group symmetrically bridging Ru(2) and Ru(3). Ru(1) interacts with all atoms of the five-membered ring of the azulene. Ru(2) and Ru(3) lie below the seven-membered ring and are linked in a delocalized manner to the remaining five carbon atoms of the azulene system.

M.R. Churchill and J. Wormald, *Inorg. Chem.*, 12 (1973) 191.

Nitrosylbis[1,2-bis(diphenylphosphino)ethane] ruthenium tetraphenylborate–acetone, $[Ru(NO)(diphos)_2][B(C_6H_5)_4] \cdot (CH_3)_2CO$

($P2_1n$) $Z = 4, R = 7.4\%$ for 3195 reflections. The complex has approximate trigonal bipyramidal geometry with bidentate diphos ligands bridging axial and equatorial positions and nitrosyl on the remaining equatorial position, coordinating almost linearly. Ru–N 1.74 Å; Ru–N–O 1.74(1)°. Average Ru–P 2.39(1) Å.

C.G. Pierpont and R. Eisenberg, *Inorg. Chem.*, 12 (1973) 199.

Di-μ-acetato-tetrakis{μ_3-methane-2,4-pentanedienatocobalt(II,III)}, $Co_4(CH_3O)_4(CH_3CO_2)_2(C_5H_7O_2)_4$

($P\overline{1}$) $Z = 2, R = 6.8\%$. The structure consists of a distorted cube with cobalt ions and methoxide oxygens at alternate corners. The octahedral coordination of each cobalt is completed by a chelated 2,4-pentanedionate group and by the oxygen of one of the acetate group which bridge the top and bottom faces of the cube. Co–O distances for one pair are consistently shorter by 0.16 to 0.20 Å than the other pair, suggesting that the former are the Co^{III} ions.

J.A. Bertrand and T.C. Hightower, *Inorg. Chem.*, 12 (1973) 206.

Hexamminecobalt hexachloroantimonate(III), $Co(NH_3)_6SbCl_6$

($Pbca$) $Z = 4, R = 6.1\%$ for 416 independent reflections. $SbCl_6^{3-}$ and $Co(NH_3)_6^{3+}$ ions both have exact $\overline{1}$ crystallography symmetry (and approximate octahedral symmetry). Sb–Cl 2.643(6), 2.652; mean Co–N 1.982(10) Å.

D.R. Schroeder and R.A. Jacobson, *Inorg. Chem.*, 12 (1973) 210.

Bromobis(triphenylphosphine)copper(I) hemibenzenate, $[(C_6H_5)_3P]_2CuBr.\frac{1}{2}C_6H_6$

($P\overline{1}$) $Z = 2, R = 6.9\%$ for 3484 reflections. The coordination about the copper(I) atom is essentially trigonal. Cu–Br 2.346(2), Cu–P 2.282(3) and 2.263(2) Å. P–Cu–P is 126.0(1)°.

P.H. Davis, R.L. Belford and I.C. Paul, *Inorg. Chem.*, 12 (1973) 213.

Tris(tetraisopropylmethylenediphosphinate)copper(II) perchlorate, $Cu\{[(CH_3)_2CHO]_2P(O)CH_2P(O)[OCH(CH_3)_2]_2\}_3(ClO_4)_2$

($C2/c$), $Z = 8, R = 6.3\%$ for 11.541 reflections. The CuO_6 group is a slightly distorted octahedron. Cu–O 2.06–2.111, P–O 1.478 Å. Jahn–Teller effect is discussed.

P.T. Miller, P.G. Lenhert and M.D. Joesten, *Inorg. Chem.*, 12 (1973) 219.

Iodo(sulphur dioxide)methylbis(triphenylphosphine)platinum, $Pt(CH_3)(PPh_3)_2I{-}SO_2$

($P\overline{1}$) $Z = 2, R = 2.3\%$ for 3143 independent reflections. The $Pt(CH_3)(PPh_3)_2I$ part of the molecule has triphenylphosphine groups *trans* to one another. The iodine is weakly bonded to sulphur (3.391(3) Å). Comparisons of infrared spectra and molecular geometry show the complex to be similar to adducts of sulphur dioxide with metal complexes and with amines.

M.R. Snow and J.A. Ibers, *Inorg. Chem.*, 12 (1973) 224.

Chlorocyano(thiocyanato)-*N*-carbonylbis(triphenylphosphine)iridium(III), $IrCl(CN)(NCS)(CO)(PPh_3)_2$

$(P\bar{1})$ $Z = 1$, $R = 3.2\%$ for 1258 reflections. Ir–P 2.418, Ir–Cl 2.37(1), Ir–CO 1.84(4), Ir–CN 1.99(1) and Ir–CNS 1.62 Å. The geometry of the complex is essentially octahedral and the complex refined has CN *trans* to NCS and CO *trans* to Cl.

J.A. Ibers, D.S. Hamilton and W.H. Baddley, *Inorg. Chem.*, 12 (1973) 229.

Tetrahydroboratobis(cyclopentadienyl)titanium(III), $(h^5\text{-}C_5H_5)Ti(BH_4)$

$(Fm2m)$ $Z = 4$, $R_1 = 3.0\%$ for 249 independent reflections. The coordination geometry about titanium is quasi-tetrahedral with an average (ring centroid)–Ti–(ring centroid) angle of 136.7° and H–Ti–H of 60± 5°. Ti–H 1.75(8), Ti–B 2.37(1) and Ti–C 2.35(4) Å. The carbon atoms of the cyclopentadienyl groups are disordered.

K.M. Melmed, D. Coucouvanis and S.J. Lippard, *Inorg. Chem.*, 12 (1973) 232.

Bis(tetra-*n*-butylammonium)-μ-*S′*,*S′*-[(tetrakis(ethane-1,2-dithiolate)diiron(III)], $[(n\text{-}C_4H_9)_4N]_2[Fe(edt)_2]_2$

$(P2_1/c)$ $Z = 4$, $R = 5.6\%$ for 1609 reflections. Bridging ethanedithiolate sulphur atoms gives rise to a planar and nearly square Fe_2S_2 ring. Fe–Fe 3.410 Å. Each iron is coordinated in a trigonal bipyramidal geometry (distorted) and four Fe–S distances are 2.442(10) mean, and a bridging Fe–S is 2.503(3) Å.

M.R. Snow and J.A. Ibers, *Inorg. Chem.*, 12 (1973) 249.

1-(diphenylphosphino)-2-(dimethylarsino)tetrafluorocyclobut-1-ene-μ-(1-(diphenylphosphino)-2-(dimethylarsino)tetrafluorocyclobut-1-ene)-tetracarbonyldiiron, $[Ph_2P\overline{C=C(Me_2As)C}F_2CF_2]_2Fe_2(CO)_4$

$(C2/c)$ $Z = 8$, $R = 3.3\%$ for 3458 reflections. One iron has distorted octahedral geometry, coordinated to two bidentate $(Ph_2P\overline{C=C(Me_2As)C}F_2CF_2 \equiv f_4asp$, ligands and a terminal carbonyl with the second iron in the ninth position. The latter is in an approximately trigonal bipyramidal environment with two terminal carbonyls and a C-C double bond of one $f_4$asp in the equatorial plane and carbonyl and the other iron at the apices. One $f_4$asp bridges the irons.

F.W.B. Einstein and R.D.G. Jones, *Inorg. Chem.*, 12 (1973) 255.

(Cycloocta-1,5-diene)[1,2-bis(diphenylphosphino)ethane] methyliridium(I), $(C_8H_{12})(diphos)Ir(CH_3)$

$(Pnma)$ $Z = 4$, $R = 5.6\%$ for 1698 reflections. The central iridium has distorted trigonal-bipyramidal coordination with the chelating diphos ligand occupying two equatorial sites and the methyl group axial and the cycloocta-1,5-diene ligand takes up the other two positions. Ir–P 2.308(3); Ir–CH_3 2.133(16); Ir–olefin(equatorial) 2.011(9), (axial) 2.106(9); P–Ir–P 84.9(2)°.

M.R. Churchill and S.A. Bezman, *Inorg. Chem.*, 12 (1973) 260.

Pentacarbonyltriphenylphosphinechromium(O), $(C_6H_5)_3PCr(CO)_5$ (A) and pentacarbonyl (triphenyl phosphite)chromium(O), $(C_6H_5O)PCr(CO)_5$ (B)

Both are ($P\bar{1}$) $Z = 2$. $R = 4.3\%$ for 3450 reflections (A) and $R = 4.8\%$ for 3573 independent reflections (B). Both structures exhibit slightly distorted octahedral geometry about the chromium with phosphorus and five carbons occupying the coordination positions. Cr–P 2.422(1), Cr–C(*trans*) 1.861(4) Å in (B).

H.J. Plastas, J.M. Stewart and S.O. Grim, *Inorg. Chem.*, 12 (1973) 265.

Dimethyldiisothiocyanato(terpyridyl)tin(IV), $(CH_3)_2Sn(NCS)_2(C_{15}H_{11}N_3)$

($P\bar{1}$) $Z = 2$, $R = 2.9\%$ for 4395 reflections. Tin is bonded to three terpyridyl nitrogens so as to form a pentagonal plane. The methyl groups are *trans* at axial positions with C–Sn–C 173.7(2)°; Sn–C 2.105, Sn–NCS 2.283, Sn–N(terpyridyl) 2.515 Å.

D.V. Naik and W.R. Scheidt, *Inorg. Chem.*, 12 (1973) 272.

4-Methylpyridinium nonabromoantimonate(V), $(C_6H_7NA)_2Sb^VBr_9$

($C2/m$) $Z = 2$, $R = 5.0\%$ for 1531 independent reflections. The structure comprises 4-methylpyridinium, $Sb^VBr_6^-$, and Br_3^- ions. The $SbBr_6^-$ has C_{2h} symmetry and Sb–Br (average) is 2.563(4) Å. The tribromide is centrosymmetric with bond length of 2.561 (4) Å. These two ions form almost linear chains with Br · · · Br 3.444(4) Å.

S.L. Lawton, D.M. Hoh, R.C. Johnson and A.S. Knisely, *Inorg. Chem.*, 12 (1973) 277.

Structures of $Cs_2LiM(CN)_6$, where M = Mn, Fe, Co

The compounds crystallize in ($Fm3m$) $Z = 4$, and $R = 2.05$, 1.69 and 1.60% respectively. The M–C bond lengths decrease significantly proceeding from Mn to Co whereas the C–N lengths do not change.

B.I. Swanson and R.R. Ryan, *Inorg. Chem.*, 12 (1973) 283.

Angeli's Salt, sodium trioxodinitrate(II) monohydrate, $Na_2N_2O_3 \cdot H_2O$

($Pbcm$) $Z = 4$, $R = 3.2\%$ for 352 reflections. The anion is planar. N=N 1.264, N–O 1.347, N–$O_{(1)}$ 1.310, N–$O_{(2)}$ 1.322, where the subscripts denote two oxygens bonding to one nitrogen. The shortest Na–O distance is 2.35 Å.

H. Hope and M.R. Sequeira, *Inorg. Chem.*, 12 (1973) 286.

5,6-μ-Diphenylphosphino-decaborane(14), $B_{10}H_{13}P(C_6H_5)_2$

($P2_12_12_1$) $Z = 4$, $R = 10.2\%$ for 833 independent reflections. The phosphorus atom occupies a bridging position between B_5 and B_6 of a distorted B_{10} icosohedral fragment and confirms the substitution of a non-metallic atom in a bridge hydrogen position in the decaborane(14) molecule.

L.B. Friedman and S.L. Perry, *Inorg. Chem.*, 12 (1973) 288.

Niobocene, $[(C_5H_5)(C_5H_4)NbH]_2$

($P2_12_12_1$) $Z = 12$ (dimers), $R = 6.3\%$ for 2620 reflections. The structure has terminal hydrides and C_5H_5 ligands (pentahapto) and bridging C_5H_4 ligands (pentahapto and monohapto). The angle between the cyclopentadienyl rings in each monomer unit is 39.6(4)°. Both types of cyclopentadienyl ring are planar with Nb–C(C_5H_5) 2.398 and Nb–C(C_5H_4) 2.297(3)–2.439(8) Å; Nb–Nb 3.105(6) Å.

L.J. Guggenberger, *Inorg. Chem.*, 12 (1973) 294.

Di-μ-hydroxo-tetraglycinatodichromium(III), $[Cr(gly)_2OH]_2$

($P2_1/n$) $Z = 2$ (dimer), $R = 4.0\%$ for 775 independent reflections. Well separated dimeric pairs of octahedrally coordinated chromium atoms are linked by two hydroxo bridges. The Cr–O–Cr–O unit is strictly planar with Cr–O 2.974(2), O–O 2.575(6) Å and Cr–O–Cr bridge angle 98.2(2)°. Each glycine coordinates through nitrogen (2.064(6) Å average) and oxygen (1.973(4) Å average). Magneta data are discussed.

J.T. Veal, W.E. Hatfield, D.Y. Jeter, J.C. Hempel and D.J. Hodgson, *Inorg. Chem.*, 12 (1973) 342.

$CsCuBr_3$

($C222_1$) $Z = 8$, $R = 6.9\%$ for 842 unique reflections. The structure contains facial-bridged $[Cu_2Br_9]^{5-}$ dimers which share corners with six other dimers in a three-dimensional network. Exchange interactions are discussed and the complex $CsCuBr_4$ is also refined.

Ting-I Li and G.D. Stucky, *Inorg. Chem.*, 12 (1973) 441.

Chlorobis(acetone thiosemicarbazene)nickel(II) chloride monohydrate (A) and nitratobis (acetone thiosemicarbazene)nickel(II) nitrate monohydrate (B)

Both are ($Pca2_1$) $Z = 4$, $R = 3.6\%$ for 1426 reflections (A) and $R = 6.1\%$ for 1016 reflections (B). In (A) the cation is trigonal bipyramidal with one chlorine and two sulphur atoms equatorial. Ni–Cl 2.305, Ni–S 2.290 and 2.131 Å. The nitrate appears to be octahedral although there are only slight changes in the ligand geometry from the chloro case.

M. Mathew, G.J. Palenik and G.R. Clark, *Inorg. Chem.*, 12 (1973) 446.

Chugaev's Red Salt, $[(C_4H_8N_4CH_3)Pt(CNCH_3)_2][B(C_6H_5)_4]$

($C2/c$) $Z = 8$, $R = 5.3\%$ for 1563 reflections. The Pt atom is coordinated by two methyl isocyanide ligands and by two C atoms of the $(CH_3)_3N_4H_2C_2^-$ chelating ligand formed by the addition of methyl hydrazine to two methyl isocyanide ligands. The entire cation is almost planar. Pt–C(chelate) 1.95(2), 2.063(3); Pt–$CNCH_3$(average) 1.96(2) Å.

W.M. Butler, J.H. Enemark, J. Parks and A.L. Balch, *Inorg. Chem.*, 12 (1973) 451.

Bis(1,3,5,7-tetramethylcyclooctatetraenyl)uranium(IV), $U(C_8H_4(CH_3)_4)_2$
($P2_1/c$) $Z = 8$, $R = 3.68\%$ for 4341 independent reflections. The uranium atom is symmetrically bonded to planar, methyl-substituted cyclooctatetraenyl dianion rings. U–C lengths are equal and average 2.658(6) Å. The two crystallographically independent molecules differ in the relation of the substituent methyl groups.
K.O. Hodgson and K.N. Raymond, *Inorg. Chem.*, 12 (1973) 458.

$LiCo(NH_3)_6Np_2O_8(OH_2) \cdot 2H_2O$
($C2/c$) $Z = 4$, $R = 5.7\%$ for 2715 independent reflections. Two independent neptuniums are octahedrally coordinated to oxygen atoms with four short and two long bonds. The octahedra are linked by two of their oxygen atoms. Lithium ions share four oxygens with three octahedra and are also linked to two H_2O molecules while the $Co(NH_3)_6^{3+}$ octahedra lie between chains of Np octahedra and cross-link them.
J.H. Burns, W.H. Baldwin and J.R. Stokely, *Inorg. Chem.*, 12 (1973) 466.

Acrylonitrile bis(tri-*o*-tolyl phosphite)nickel, $(C_2H_3CN)Ni[(o\text{-}CH_3C_6H_4O)_3P]_2$ (A) and (ethylene)bis(tri-*o*-tolyl phosphite)nickel, $(C_2H_4)Ni[(o\text{-}CH_3C_6H_4O)_3P]_2$ (B)
($P2_1/c$) $Z = 4$, $R = 9.7\%$ for 2182 reflections (A) and (Pc) $Z = 2$, $R = 8.8\%$ for 2059 reflections (B). Both structures have a nickel atom trigonally coordinated to two phosphite ligands and an olefin which is nearly in the PNiP plane. (A) Ni–P 2.095, Ni–C 2.016(10) and 1.911(12) Å and angle between Ni–olefin and Ni–P planes 3.9(1)°. (B) The corresponding values are 2.095 for Ni–P; Ni–C 2.02 Å. The dihedral angle is 6.6 (11)°.
L.J. Guggenberger, *Inorg. Chem.*, 12 (1973) 499.

Bis[dihydrobis(1-pyrazolyl)borato] cobalt(II) $[H_2B(pz)_2]_2Co$
($Pc2_1b$) $Z = 4$, $R = 6.5\%$ for 1250 reflections. Each cobalt is tetrahedrally coordinated to two bidentate $(H_2B(pz)_2)$ ligands. Co–N, 1.967(12) and distortion lowers the overall symmetry to $C_2(2)$. Single-crystal electron paramagnetic resonance data are discussed.
L.J. Guggenberger, C.T. Prewitt, P. Meakin, S. Trofimenko and J.P. Jesson, *Inorg. Chem.*, 12 (1973) 508.

Hexaminocobalt nonafluoroantimonate(III), $Co(NH_3)_6Sb_2F_9$
($P2_1/c$) $Z = 2$, $R = 6.0\%$ for 1037 reflections. The asymmetric unit consists of one $Co(NH_3)_6^{3-}$ ion and one distorted $Sb_2F_9^{3-}$ ion which resembles two octahedra sharing a corner with each octahedra possessing a lone pair. Sb–F(bridge) 2.249(12), 2.449(4) Å. Sb–F(opposite the lone pair) 1.948(7); Sb–F(opposite bridging fluorine) 2.015(5) Å; Co–N 1.990(5) Å.
D.R. Schroeder and R.A. Jacobsen, *Inorg. Chem.*, 12 (1973) 515.

Tri-μ-methylmercapto-hexacarbonyldiiron(II) tetrakis(*cis*-1,2-di(perfluoromethyl)-ethylene-1,2-dithiolato)diiron, $[Fe_2(\mu\text{-}SCH_3)_3(CO)_6][Fe_2(S_2C_2(CF_3)_2)_4]$

($P\bar{1}$) $Z = 2, R = 5.8\%$ for 4335 reflections. The structure consists of two face-shared octahedra with methylmercapto groups occupying the bridging positions in the cation. Average Fe–S and Fe–C are 2.305(2) and 1.80(1) Å. Fe · · · Fe is 3.062(4) Å with octahedral Fe^{II} d^6. In the centrosymmetric anion each metal has square pyramidal geometry and dimerization occurs through Fe–S linkages (2.311(5) Å). Fe–S(dithiolene) 2.190(6), Fe–Fe 2.767(4) Å.

A.J. Schultz and R. Eisenberg, *Inorg. Chem.*, 12 (1973) 518.

1,6,7,8-Tetrahaptoheptafulveneiron tricarbonyl, $(C_7H_6CH_2)Fe^0(CO)_3$

($P2_1/c$) $Z = 4, R = 3.41\%$ for 1886 independent reflections. The heptafulvene and Fe $(CO)_3$ moieties interact via a trimethylene methane–metal linkage with Fe–C(central) 1.946(2) and Fe–C(peripheral) 2.120(3), 2.175(3) and 2.192(3) Å.

M.R. Churchill and B.G. DeBoer, *Inorg. Chem.*, 12 (1973) 525.

(Cycloocta-1,5-diene)[1,3-bis(diphenylphosphino)propane] methyliridium(I), $(C_8H_{12})[(C_6H_5)_2P(CH_2)_3P(C_6H_5)_2]Ir(CH_3)$

($C2/c$) $Z = 8, R = 7.6\%$ for 3573 independent reflections. The iridium atom is in a slightly distorted trigonal-bipyramidal coordination environment with the chelating 1,3-bis (diphenylphosphino)propane ligand occupying two equatorial sites; the methyl is axial. The cycloocta-1,5-diene ligand spans the remaining axial and equatorial positions. Ir–P 2.309(4), 2.337(4); Ir–CH_3 2.153; Ir–olefin(equatorial) 2.033(12); Ir–olefin(axial) 2.127(12) Å.

M.R. Churchill and S.A. Bezman, *Inorg. Chem.*, 12 (1973) 531.