Coordination Chemistry Reviews, 11 (1973) 263–271

X-RAY BIBLIOGRAPHY

M. KEETON
1260 Princess St., Apt. 89, Kingston, Ontario (Canada)
(Received March 26th, 1973)

Tetra-ammonium uranyl tricarbonate

($C2/c$) $Z = 4$, $R = 7.6\%$ for 1833 reflections. The uranium atom is eight-coordinated in a distorted hexagonal environment. The linear uranyl group is perpendicular to the equatorial plane in which three carbonate groups are chelated. The NH_4^+ groups link the anions through hydrogen bonding.

R. Grazianio, G. Bombieri and E. Forsellini, *J. Chem. Soc. Dalton*, (1972) 2059.

PdCl(cmp)(cmpH) (cmp = 3,4′-bis(ethoxycarbonyl)-5-chloro-3′,4′,5′-trimethyldipyrromethane)

(Cc) $Z = 4$, $R = 5.7\%$ for 2257 independent reflections. The palladium is essentially square planar, coordinated to three nitrogens and one chlorine atom. The uni- and bidentate dipyrromethane ligands deviate from planar configurations. The uncoordinated nitrogen atom lies above the palladium square plane but is non-bonding.

F.C. March, J.E. Fergusson and W.T. Robinson, *J. Chem. Soc. Dalton*, (1972) 2069.

Tris(hexamethyldisilylamido)iron (III)

($P\overline{3}_1c$) $Z = 2$, $R = 0.94$ for 1051 independent reflections. The molecule has D_3 symmetry with an angle of 49.2° between the FeN_3 and NSi_2 planes. The Fe–N bond is 1.917(4) Å, somewhat short.

M.B. Hursthouse and P.F. Rodesiler, *J. Chem. Soc. Dalton*, (1972) 2100.

catena-μ-Acetato-{di-(3-aminopropyl)amine} copper(II) perchlorate

($Pna2_1$) or ($Pnam$) $Z = 4$, $R = 10.0\%$ for 1350 reflections. Copper is square-planar coordinated with three amino nitrogens and one acetate oxygen. Cu–N 1.92–2.05 Å; Cu–O 2.02 Å. The second oxygen of the acetate bonds weakly in a tetragonal site of a second copper atom, Cu–O 2.29, and completes a square pyramidal coordination. The same oxygen participates in a still weaker interaction with the initial copper in a direction displaced ca. 40° from the tetragonal axis so that a distorted octahedral coordination geometry is present at a tertiary level. Cu–O 2.74 Å.

B.W. Shelton, T.N. Waters and N.F. Curtis, *J. Chem. Soc. Dalton*, (1972) 2133.

Acetatobis(phenylazophenyl-2*C*,*N*′)rhodium(II)

(*Cc*) $Z = 4$, $R = 5.0\%$ for 2318 reflections. The structure determination confirms that azobenzene chelates by means of σ-bonds at an *ortho* carbon and the lone pair of the further nitrogen, forming a five-membered chelate ring.

R.J. Hoare and O.S. Mills, *J. Chem. Soc. Dalton*, (1972) 2138.

Di-μ-chloro-dicarbonylrhodium(I)bis(phenylazophenyl-2*C*,*N*′)rhodium(III)

($P\bar{3}ci$) $Z = 12$, $R = 6.3\%$ for 1797 unique reflections. The structure contains an octahedral bis(phenylazophenyl) rhodium moiety with two five-membered Rh–C–C–N–N chelate rings attached to a square-planar dicarbonylrhodium group via two chlorine bridging atoms Rh···Rh 3.551(2), Rh–Cl (*trans* to Rh–C(CO)) 2.374(5) and (*trans* to Rh–C(Ph$_2$N$_2$)) 2.532(5) Å. The molecule is folded about the chlorine bridge by 27°.

R.J. Hoare and O.S. Mills, *J. Chem. Soc. Dalton*, (1972) 2141.

Penta-amminenitroruthenium(II) chloride hydrate

($Ccm2_1$) $Z = 4$, $R = 2.5\%$ for 495 observed reflections. The metal ion is octahedrally coordinated by six nitrogen atoms with Rh–NO$_2$ 1.906(5), Rh–NH$_3$ (equatorial) 2.131(5) and 2.123(5) and Rh–NH$_3$ (axial) 2.199(6) Å.

F. Bottomley, *J. Chem. Soc. Dalton*, (1972) 2148.

Tricarbonyl-π-[1,1,1-tricarbonyl-2,3-dimethoxy-5-(diphenylmethyl)ferracyclopentadiene] iron(*Fe–Fe*)

($P2_1/c$) $Z = 4$, $R = 12.5\%$ for 1547 reflections. Both iron atoms have distorted octahedral coordination. The Fe–C–O systems are slightly bent. The three C–C bond lengths in the five-membered ring are all 1.40 ± 0.01 Å. Fe–Fe is 2.54 Å.

J.A.D. Jeffreys and C.M. Willis, *J. Chem. Soc. Dalton*, (1972) 2169.

Chlorobis[2-(diphenoxyphosphino-oxy)phenyl] (triphenyl phosphite)iridium(III)

($P\bar{1}$) $Z = 2$, $R = 5.2\%$ for 5780 observed reflections. The structure consists of three independent monomeric units. The iridium atom is in a distorted octahedral environment with three phosphorus atoms, two *ortho* carbon atoms of the triphenyl phosphite groups and one chloride ion as ligands. Ir–C 2.09 and 2.12; Ir–P (*trans* to C) 2.268 and 2.291, Ir–P (*trans* to Cl) 2.194 and Ir–Cl 2.416 Å.

J.M. Guss and R. Mason, *J. Chem. Soc. Dalton*, (1972) 2193.

Cis- (A) and *trans*- (B) methylamido-tris(dimethylaluminium) and cyclo-di-μ-dimethylamido-bis(dimethylaluminium) (C)

(A) ($R3$) $Z = 2$, $R = 5.5\%$ for 996 reflections. This compound has six-membered (AlN)$_3$ rings in chair conformation and all methyl substituents on nitrogen atoms are equatorial. Al–N 1.940(5), Al–C 1.973(5), N–C 1.504(10) Å.

(B) ($C2/c$) $Z = 4$, $R = 12.1\%$ for 621 reflections. The molecule has the same dimensions as the *cis* isomer, but the configuration at one of the nitrogen atoms is reversed and the $(AlN)_3$ rings are in the skew boat form.

(C) ($P2_1/a$) $Z = 2$, $R = 6.9\%$ for 525 reflections. The molecules have four-membered $(AlN)_2$ rings and approximate D_{2h} symmetry. Al–N 1.958(5), Al–C 1.950(8), N–C 1.509(10) Å.

G.M. McLaughlin, G.A. Sim and J.D. Smith, *J. Chem. Soc. Dalton*, (1972) 2197.

$Cu(hfac)_2(ted)$ (hfac = $(CF_3.CO)_2CH$ and ted = $N(CH_2CH_2)_3N$)

($Pnnm$) $Z = 2$, $R = 6.0\%$ for 832 reflections. The copper and two β-diketonates lie in a plane of symmetry. The ligands are related by a two-fold axis giving square planar coordination about copper with Cu–O 1.958 and 1.954 Å. The fifth and sixth axial positions are occupied by nitrogen atoms at 2.566 Å.

R.C.E. Belford, D.E. Fenton and M.R. Truter, *J. Chem. Soc. Dalton*, (1972) 2208.

Salicylate-(1,10-phenanthroline)thallium(I)

($Pbcn$) $Z = 4$, $R = 10.1\%$ for 1599 reflections. The five atoms of the thallium coordination sphere form the base of a shallow, irregular pentagonal pyramid which has the thallium ion at its apex. The phenanthroline ligand coordinates to the Tl^+ ion through both nitrogen atoms with Tl···N 2.65 and 2.71 Å. The salicylate ion forms an asymmetrical bidentate coordination with the Tl^+ through the two oxygen atoms of the carboxylate group. Tl···O 2.65 and 2.98 Å. The first oxygen interacts with the other thallium of the dimer (3.0 Å) and the second atom of the carboxylate group is hydrogen-bonded to the phenolic group (2.52 Å).

D.L. Hughes and M.R. Truter, *J. Chem. Soc. Dalton*, (1972) 2214.

Bis(*N*-methyl-*N*-phenyldithiocarbamato)-nickel(II) (A) and -copper(II) (B)

($P2_1/a$) $Z = 2$ (A and B), $R = 11.6\%$ for 1106 reflections (A) and $R = 12.5\%$ for 1202 reflections (B). The metal atom in both structures is planar coordinated by four sulphurs. Ni–S 2.208 and 2.198; Cu–S 2.329 and 2.274 Å. Both compounds are monomeric.

J.M. Marlin, P.W.G. Newman, B.W. Robinson and A.H. White, *J. Chem. Soc. Dalton*, (1972) 2233.

Bis(*N*,*N*-di-isopropyldithiocarbamato)nickel(II)

($P2_1/a$) $Z = 4$, $R = 11.0\%$ for 1909 reflections. The structure consists of two independent sets of planar molecules, the nickel atoms each being planar coordinated by four sulphur atoms at equal distances. Ni–S(mean) 2.181(5) Å.

P.W.G. Newman and A.H. White, *J. Chem. Soc. Dalton*, (1972) 2239.

Bis[dicarbonyl(triethylphosphine)-nitrosylferrio] mercury

($P2_1/n$) $Z = 2$, $R = 6.0\%$ for 1800 unique reflections. The two sets of radial carbonyl groups adopt a staggered configuration as does each set of ethyl groups with respect to its set of carbonyl groups. Hg—Fe 2.534(2) Å. The crystal is disordered such that nitrosyl groups are indistinguishable from carbonyl groups.

F.S. Stephens, *J. Chem. Soc. Dalton*, (1972) 2257.

(Pentafluorophenyl) (triphenylphosphine)gold(I)

($P2_1/c$) $Z = 4$, $R = 5.5\%$ for 1834 reflections. The gold atom is linearly coordinated; C—Au—P 178°; Au—P 2.27, Au—C 2.07(2) Å. The phenyl and pentafluorophenyl rings are planar.

R.W. Baker and P.J. Pauling, *J. Chem. Soc. Dalton*, (1972) 2264.

Dicarbonyl(thiocarbonyl)bis(triphenyl phosphine)iridium(I) hexafluorophosphate—acetone

($P2_1/m$) $Z = 4$, $R = 9.83\%$ for 7113 visually estimated intensities. The iridium atoms are at the centre of a trigonal bipyramid with PPh_3 at the apices and CO and CS groups coplanar with iridium. Ir—C 1.867, C—S 1.511 Å; Ir—C—S 178.2°.

J.S. Field and P.J. Wheatley, *J. Chem. Soc. Dalton*, (1972) 2269.

μ-[1,3,6-η-1,4,6-η-1,3,6-tris(trifluoromethyl)hexa-1,3,5-trien-1,6-diyl]-bis-(dicarbonylcobalt) (*Co—Co*)

($Pna2_1$) $Z = 4$, $R = 9.7\%$ for 757 independent observed reflections. The complex contains a six-carbon-atom ligand bridging two $[Co(CO)_2]$ moieties. This bridging ligand is composed of two allylic functions joined by a C—C single bond and a 60° angle between the two allylic planes. The allyl groups are each bonded to cobalt atoms, Co—Co 2.459(6) Å. Regarding allyls as tridentate, cobalts are seven-coordinate.

R.S. Dickson, P.J. Fraser and B.M. Gatehouse, *J. Chem. Soc. Dalton*, (1972) 2278.

Tricarbonyl(1,3-η-hexen-5-one)iron hexafluorophosphate

($Pbca$) $Z = 8$, $R = 7.1\%$ for 2365 independent reflections. The tricarbonyliron group is bound to the allyl fragment and the oxygen atom of the $CH_2 \overset{\cdots}{-} CH \overset{\cdots}{-} CH \cdot CH_2 \cdot COCH_3$ ligand. Fe—C 2.141(10), 2.063(9), 2.144(8); Fe—O 1.999(5); Fe—CO(*trans* to oxygen) 1.787(7); Fe—CO(*trans* to allyl) 1.840(9) and 1.828(8) Å.

A.D.U. Hardy and G.A. Sim, *J. Chem. Soc. Dalton*, (1972) 2305.

Niobium pentafluoride—antimony pentafluoride

($P\bar{1}$) $Z = 2$, $R = 6.5\%$ for 906 reflections. The structure consists of a zigzag endless chain arrangement with alternating antimony and niobium atoms linked by *cis* bridging fluorine atoms. Both metal atoms are octahedrally coordinated by fluorine atoms (distorted).

A.J. Edwards, *J. Chem. Soc. Dalton*, (1972) 2325.

Bis(*N,N,N'N'*-tetraethylethylenediammonium)hexa-μ-chloro-μ_4-oxo-tetra[chlorocuprate(II)]
($C2/c$) $Z = 4, R = 6.0\%$ for 1478 reflections. The $[Cu_4OCl_{10}]^{4-}$ ion is tetrahedral with two bridging chlorines and the oxygen atom lying on the crystallographic two-fold axis. The copper atoms are trigonal bipyramidal, surrounded by four chlorines, 2.40 (bridge) and 2.24 (terminal) and one oxygen, Cu–O 1.92 Å.
R. Belford, D.E. Fenton and M.R. Truter, *J. Chem. Soc. Dalton*, (1972) 2345.

[1,3-Bis(dimethylarsino)-2-chloro-1,1,3,3-tetrafluoropropane] tetracarbonylchromium, $(Me_2AsCF_2 \cdot CHCl \cdot CF_2AsMe_2)Cr(CO)_4$
($P2_1/c$) $Z = 4, R = 6.5\%$ for 1475 reflections. The six-membered ring has a chair conformation, with chlorine occupying the least sterically hindered position. Chromium is in a distorted octahedron made up of four carbonyls and two arsenic atoms, Cr–As 2.423(3), 2.439(3) Å.
I.W. Nowell and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 2378.

Disordered structures of $Me_2XCR^1R^2 \cdot CF_2XMe_2 \cdot M(CO)_4$ where M = Mo or Cr; X = As or P; R^1 = F or H; R^2 = H, CF_3 or Cl
Four compounds with R^2 = H are isostructural (($Pbca$) $Z = 8$). Two compounds with R^2 = Cl and CF_3 are ($Pbca$) and ($C2/c$). R factors are 5.0% to 8.0% for 1200–1800 reflections. The metal atoms are coordinated in a distorted octahedral arrangement to two arsenic or phosphorus atoms, and to four carbonyl groups. X–M–X 82–85°; Mo–As 2.58, Mo–P 2.48 and Cr–As 2.43 Å. Each crystal has two molecular configurations distributed (1:1 to 6:1) at each lattice site.
I.W. Nowell, S. Rettig and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 2381.

Dicarbonyl-π-cyclopentadienyl-cobalt complex with mercury(II) chloride, $(\pi\text{-}C_5H_5)(CO)_2Cl \cdot HgCl_2$
($P2_1/n$) $Z = 4, R = 9.3\%$ for 1002 non-equivalent reflections. The complex is a 1:1 Lewis acid–base adduct bonded through the metal atom, the resulting Ag–Co bond length 2.578(4) being that expected for a single covalent bond. Mercury is trigonal planar with two additional Ag–Cl contacts making it effectively trigonal bipyramidal.
I.W. Nowell and D.R. Russell, *J. Chem. Soc. Dalton*, (1972) 2393.

1:3 Complex between dicarbonyl-π-cyclopentadienylcobalt and mercury(II) chloride
($P1$) $Z = 2, R = 9.5\%$ for 1354 reflections. The complex is best formulated as a salt $[(\pi\text{-}C_5H_5)Co(CO)_2(HgCl)]^+Cl^-$ with two additional molecules of mercury(II) chloride. Hg–Co 2.504(9) Å.
I.W. Nowell and D.R. Russell, *J. Chem. Soc. Dalton*, (1972) 2396.

Octachlorobis(adeninium)tricopper(II) tetrahydrate
($P2_1/c$) $Z = 2, R = 5.1\%$ for 1187 independent reflections. The complex is trinuclear

with two adenines bridging three coppers, Cu···Cu 3.479 Å. The central copper is six-coordinate with two nitrogens (2.027 Å) and four chlorines (bridging) forming two strong (2.313 Å) and two weak (2.766 Å) bonds. The two terminal coppers are five-coordinate square pyramidal, Cu–N 2.028; Cu–Cl (terminal) 2.272, 2.291; Cu–Cl (bridge) 2.324, 2.743 Å. A sixth pseudo-octahedral position is occupied by a chlorine from an adjacent complex (3.274 Å).

P. de Meester and A.C. Skapski, *J. Chem. Soc. Dalton*, (1972) 2400.

$Ru_4(CO)_{10}(C_{12}H_{16})$

($P2_1/n$) $Z = 4$, $R = 8.6\%$ for 1102 independent reflections. An open C_{12} ring is attached to a "butterfly" Ru_4 cluster by means of a unique allyl group which is σ-bonded to the cluster "hinge" at both ends and π-bonded to the cluster "tips" on both sides. There is a further attachment from the ring to one of the butterfly tips by a second (normal) π-allyl group.

R. Belford, H.P. Tayler and P. Woodward, *J. Chem. Soc. Dalton*, (1972) 2425.

[*N,N'*-Ethylenebis(2-amino-5-chlorobenzylidene-iminato)] cobalt(II)

(*Pnam*) $Z = 4$, $R = 7.0\%$ for 707 data. There is no coordination by the metal normal to the plane of the complex. The CoN_4 unit is a very squat pyramid, with Co lying 0.14 Å above the N_4 plane.

R. Karlsson, L.M. Engelhardt and M. Green, *J. Chem. Soc. Dalton*, (1972) 2463.

[Bis(biphenylylidene)butatriene] hexacarbonyldi-iron

($P2_1/n$) $Z = 4$, $R = 6.9\%$ for 1351 reflections. The butatriene chain is bent into a zigzag in which the bond angles at the central chain atoms are 128° and the iron atoms may be regarded as bonded to the chain via σ-bonds to the central carbons and via π-bonds to the terminal double bonds. Fe–Fe 2.596(4) Å.

D. Bright and O.S. Mills, *J. Chem. Soc. Dalton*, (1972) 2465.

Bis(ω-nitroacetophenonato)copper(II) (A) and addition complexes with α-picoline (B) and γ-picoline (C)

($P\overline{1}$) $Z = 1$, $R = 7.2\%$ for 1248 independent reflections (A), ($P\overline{1}$) $Z = 2$, $R = 9.6\%$ for 2066 independent reflections (B) and ($C2/c$) $Z = 4$, $R = 7.2\%$ for 1983 independent reflections (C). In (A) and (C) the copper atom is in a pseudo-octahedral environment, in (B) tetragonal. The N bases are coordinated on the basal plane of the pyramid in (B) and in the equatorial plane in (C).

M. Bonamico, G. Dessy, V. Fares and L. Scaramuzza, *J. Chem. Soc. Dalton*, (1972) 2477.

Aquo-2-benzothiazolin-2-ylideneaminomethylpyridine(picolinato)copper (II) perchlorate

($P\overline{1}$) $Z = 2$, $R = 5.9\%$ for 3515 reflections. The copper is five-coordinated by an oxygen and a nitrogen atom from the picolinic ligand, Cu–O 1.957(4), Cu–N 2.021(5), and from two nitrogen atoms (pyridine and imine of 2-benzothiazolin-2-ylidene-amino-methylpyridine). Cu—N 1.998(4), 1.981(5); Cu—OH_2(apical) 2.256(5) Å.

A. Mangia, M. Nardelli, C. Pelizzi and G. Pelizzi, *J. Chem. Soc. Dalton*, (1972) 2483.

Di-μ-(dimethylsilylene)-bis[tricarbonyl(trimethylsilyl)ruthenium(III)]

($P2_1/c$) $Z = 2, R = 6.0\%$ for 658 independent reflections. The molecule, which is similar to its tin analogue, contains a Ru—Ru bond of length 2.96 Å. This result is discussed in relation to the metal—metal bond in tris-μ-chloro-diruthenium complexes.

M.M. Crozat and S.F. Watkins, *J. Chem. Soc. Dalton*, (1972) 2512.

Bis(dithiobenzoato)zinc(II)

($P\bar{1}$) $Z = 2, R = 7.1\%$ for 1200 observed reflections. The presence of two four-membered rings in the monomeric unit and the resulting severe distortion of the zinc coordination tetrahedron proves that conjugation on the whole ligand molecule is a determining factor in allowing such chelation to exist.

M. Bonamico, G. Dessy, V. Fares and L. Scaramuzza, *J. Chem. Soc. Dalton*, (1972) 2515.

Nonacarbonyl-μ-dimethylarsino-μ-[2-(diphenylphosphino)tetrafluoro-cyclobut-1-enyl] triiron

($P2_12_12_1$) $Z = 8, R = 5.2\%$ for 2175 reflections. The molecule is based on three $Fe(CO)_3$ groups and an Me_2As group which are linked to form a four-membered ring. The mean Fe—As is 2.387 Å. The $\mathrm{Ph_2P{\cdot}\overline{C{:}C{\cdot}CF_2{\cdot}C}F_2}$ forms a bond with each iron atom through phosphorus, Fe—P 2.48 Å; the cyclobutenyl has Fe—(C=C) 1.96 Å, and the carbon of the cyclobutene ring has Fe—C 1.94 Å, thus bridging the two iron—iron bonds. Every iron is approximately octahedral.

F.W.B. Einstein and R.N.G. Jones, *J. Chem. Soc. Dalton*, (1972) 2563.

2,2′-Dimethylarsino-octafluoro-1,1′-bicyclobut-1-enyl cobalt carbonyl complex

($C222_1$) $Z = 4, R = 4.1\%$ for 566 reflections. The molecule has a crystallographic two-fold axis bisecting the bond linking the fluorocyclobutenyl groups and the Co—Co bond (2.40(9) Å). A bridging carbonyl also lies on this axis. One cobalt is in a distorted octahedral environment coordinated by π-fluorocyclobutenyl (1.90(3) Å), terminal carbonyl, postulated hydrogen and dimethylarsine (Co—As 2.333(6) Å). The latter is linked to a second cobalt which is trigonal bipyramidal with axial (bridging) dimethylarsine and carbonyls, and equatorial carbonyls (terminal) and the arsenic atom which is attached to the fluorocyclobutenyl ring (Co—As 2.323(6) Å).

F.W.B. Einstein and R.D.G. Jones, *J. Chem. Soc. Dalton*, (1972) 2568.

cis-Bis(2-methoxyphenyl)bis(2,2′-bipyridyl)chromium(III) iodide monohydrate

($P\bar{1}$) $Z = 2, R = 7\%$ for 3168 reflections. The organometallic cation has *cis*-octahedral configuration and has an approximate two-fold axis bisecting the C—Cr—C angle.

Cr—C 2.101(12); Cr—N(*trans* to C) 2.156(10), Cr—N(*trans* to N) 2.071(10) Å.

J.J. Daly and F. Sanz, *J. Chem. Soc. Dalton*, (1972) 2584.

Tris(2,2′-bipyridyl)copper(II) perchlorate

$(P\bar{1})$ $Z = 2$, $R = 9.1\%$ for 3972 reflections. The monomeric complex ion exhibits a distorted octahedral configuration, with unequal distortion of the axial Cu—N bonds. Cu—N (equatorial) 2.031(6), Cu—N (axial) 2.226(7) and 2.450(7) Å.

O.P. Anderson, *J. Chem. Soc. Dalton*, (1972) 2597.

Di-μ-phenyl-bis-(diphenylaluminium) (triphenylaluminium dimer)

$(P\bar{1})$ $Z = 1$, $R = 9.1\%$ for 1636 reflections. Al···Al is 2.702 Å and is normal to the planes of the bridging phenyl group. Al—C(bridge) 2.182; Al—C(terminal) 1.958 Å. ∠ Al—C—Al is 76.5°.

J.F. Malone and W.S. McDonald, *J. Chem. Soc. Dalton*, (1972) 2646.

Di-μ-phenyl-bis(dimethylaluminium)

$(P\bar{1})$ $Z = 3$, $R = 10.6\%$ for 3126 reflections. A third of the molecules lie on centres of symmetry with the $\overline{\mathrm{Al{-}Cl{-}Al{-}C}}$ ring planar, the remainder in general positions have the central ring folded by 14° about the Al···Al line. Al···Al 2.687, Al—C (bridge) 2.144, Al—C(terminal) 1.977 Å; ∠ Al—C—Al is 77.6°.

J.F. Malone and W.S. McDonald, *J. Chem. Soc. Dalton*, (1972) 2649.

trans-Dichlorobis(4,4-dimethoxy-1-phenylphosphorinan)nickel(II)

$(P2_1/c)$ $Z = 4$, $R = 9.9\%$ for 3709 independent reflections. The nickel atom is in *trans* square planar coordination with Ni—P 2.250(2), Ni—Cl 2.171(2) Å mean distances. Phosphorinan ligands are in chair conformations with the phenyl substituent of one ring axial and that of the other ring equatorial.

A.T. McPhail and J.C.H. Steele, *J. Chem. Soc. Dalton*, (1972) 2680.

Nitrilotriacetatodiaquopraeseodymium(III) monohydrate

$(Pbca)$ $Z = 8$, $R = 8.5\%$ for 1126 reflections. The metal is nonacoordinated with six carboxylate oxygens, two waters and one nitrogen atom. The geometry is described as a distorted, tricapped, trigonal prism or a distorted, capped, square antiprism. Each nitrilotriacetate is heptadentate with one carboxylate oxygen atom which coordinates to two adjacent metal atoms and two carboxylate oxygen atoms which coordinate to metal atoms in adjacent molecules. Pr—O(carboxylate) 2.52(2), Pr—OH_2 2.68(2) Å.

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

Nitrilotriacetatodiaquodysprosium(III) dihydrate

$(Pca2_1)$ $Z = 2$, $R = 7.0\%$ for 3032 reflections. Both independent metal atoms are octacoordinate with five acetate oxygen atoms, two water molecules and one nitrogen atom at the corners of a distorted dodecahedron with triangular faces.

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

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

($P2/c$) $Z = 2, R = 5.6\%$ for 1598 observed reflections. The fixed 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 of iridium is distorted tetrahedral. The nitrosyl is coordinated to the metal almost linearly; Ir–N 1.787(8), Ir–N–O 174.1(7)°. The compound is thus formulated as an NO^+ complex of iridium(–I). Ir–P 2.323(2), Ir–C 1.873(11), C–O 1.144(11), N–O 1.180(9) Å.

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

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

($P2_1/c$) $Z = 4, R = 4.8\%$ for 2989 reflections (A) and ($P2_1/c$) $Z = 4, R = 3.8\%$ for 2554 reflections (B). Both compounds consist of polymeric chains of lanthanide (octacoordinated) axis alternately linked by four and two bridging carboxylate groups. The eight-coordination about each metal is made up by two water molecules. In the acid complex two additional waters and one hexaisothiocyanatochromate ion are also present. The acid ligand appears to be in zwitterion form with hydrogen attached to nitrogen.

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. The complex cation has a five-membered iridium–nitrogen ring that can be alternatively described as the 1-carbonyl-1,1-bis(triphenylphosphino)iridio-2,5-di-*p*-fluorophenyltetrazolium ion. The uncoordinated nitrogen atoms are 1.270(16) Å apart and are bound to the coordinated nitrogen by single bonds (1.400(16) and 1.350(16) Å).

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

Tris(cyclopentadienyl)iridium(III), $In(C_5H_5)_3$, at –100° C

($P2_12_12_1$) $Z = 4, R = 3.6\%$ for 1587 reflections. The structure consists of infinite polymeric chains with each chain unit comprising an iridium atom linked to two terminal and two bridging cyclopentadienyl groups which give rise to a slightly distorted $In–C_4$ tetrahedral environment around the indium atom. In–C 2.24(terminal), 2.37(1) and 2.47 Å (two contacts with bridging groups).

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