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X-RAY BIBLIOGRAPHY

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Two crystalline forms of dinitratobis(α-picoline)copper(II)
Both forms are ($P2_1/c$) with $Z = 4$, $R = 12\%$ for 1134 independent reflections and $R = 9\%$ for 1341 independent reflections for the two forms respectively. The two forms have similar monomeric structures with detailed differences in molecular geometry which may be due to packing.
A.F. Cameron, D.W. Taylor and R.H. Nuttall, *J. Chem. Soc. Dalton*, (1972) 59.

Di-μ-chloro-tris(triphenylphosphine)dicopper(I), $Cu_2Cl_2(PPh_3)_3$
($P2_1/c$) $Z = 4$, $R = 5.5\%$ for 2046 independent reflections. Discrete molecules contain two copper atoms held together by chlorine bridges. One copper is four-coordinate and is approximately tetrahedral and the other is three-coordinate and is approximately trigonal. Cu–P = 2.240 and 2.183, Cu–Cl = 2.436 and 2.272 Å.
V.G. Albano, P.L. Bellon, G. Ciani and M. Manassero, *J. Chem. Soc. Dalton*, (1972) 171.

$[(\pi\text{-}C_5H_5)Co(CO)]_2(GeCl_2)_2Fe(CO)_4$
($P2_12_12_1$) $Z = 8$, $R = 7.0\%$ for 3032 reflections. The compound contains a –Co–Ge–Fe–Ge–Co– planar ring. Fe–Ge = 2.438(4), Ge–Co = 2.341(9), Co–Co = 2.439(5) Å.
M. Elder and W.L. Hutcheon, *J. Chem. Soc. Dalton*, (1972) 175.

Chloro{methyl-di-[(6-methyl-2-pyridyl)methyl] amine} palladium(II) chloride
($Cmc2_1$) $Z = 4$, $R = 4.2\%$ for 1115 independent reflections. The palladium is in a distorted square planar environment with chlorine 0.93 Å out of the plane formed by Pd and three nitrogens. Pd–Cl = 2.331(3), Pd–N(tertiary) = 2.018(8), Pd–N(pyridine) = 2.068(6) Å.
M.G. Drew, M.J. Riedl and J. Rodgers, *J. Chem. Soc. Dalton*, (1972) 234.

Benzoylacetonato-[*N*, *N'*-ethylenebis(salicylideneiminato)]cobalt(III)–1.5 water
($P2_1/c$) $Z = 4$, $R = 6.8\%$ for 2928 independent reflections. The cobalt is octahedral with bidentate β-diketonate ligand and strained non-planar quadridentate salicylaldiminate. Co–O = 1.89(4), Co–N(mean) = 1.889(5) Å.
N.A. Bailey, B.M. Higson and E.D. McKenzie, *J. Chem. Soc. Dalton*, (1972) 503.

The 2:1 adduct of bis(pentafluoro-phenyl)mercury(II) and bis(diphenyl-arsino)methane
($C2/c$) $Z = 4$, $R = 9\%$ for 1218 independent reflections. Each mercury atom has one arsenic atom lying within the sum of the van der Waals radii from it. There is a T-shaped distribution of two C_6F_5 groups and the arsenic atom about mercury. Hg—As = 3.40(2), C—Hg—C = 1.73(4) Å.
A.J. Carty and B.M. Gatehouse, *J. Chem. Soc. Dalton*, (1972) 511.

Chloro-(2-methoxycyclo-octa-1, 5-dienyl)pyridine platinum
($P2_1/c$) $Z = 4$, $R = 8.6\%$ for 1445 independent reflections. Platinum is square planar, bonded to the cyclo-octadienyl ligand by both a π- and a σ-bond; pyridine is *trans* to the latter. Pt—N = 2.02(2) Å.
G. Bombieri, E. Forsellini and R. Graziani, *J. Chem. Soc. Dalton*, (1972) 525.

[*N*, *N'*-Ethylenebis(salicylideneiminato)](acetylacetonato)cobalt(III)—0.7water
($P2_12_12_1$) $Z = 4$, $R = 9.1\%$ for 1028 reflections. The cobalt atom is in a distorted octahedron; two *cis* positions are accupied by acac and the other four by the quadridentate ligand in a non-planar conformation.
M. Calligaris, G. Manzine, G. Nardin and L. Randaccio, *J. Chem. Soc. Dalton*, (1972) 543.

Bis(dithioacetato)dioxo(triphenylphosphine oxide)uranium(VI), $UO_2(MeCS_2)(Ph_3PO)$
($P2_1/c$) $Z = 4$, $R = 7.7\%$ for 3924 reflections. The uranium is seven-coordinate and pentagonal bipyramidal. The linear uranyl group is perpendicular to the equatorial plane in which four sulphur atoms of two dithioacetate groups and the oxygen atom of the ligand occupy the corners of an irregular pentagon.
G. Bombieri, V. Croatto, E. Forsellini, B. Zarli and R. Graziani, *J. Chem. Soc. Dalton*, (1972) 560.

Rhenium oxide tetrachloride
($P2_1/c$) $Z = 8$, $R = 6.9\%$ for 954 reflections. Two crystallographically different $ReOCl_4$ units in the crystal are square pyramidal with very similar dimensions. Re—O(mean) = 1.63, Re—Cl(mean) = 2.26 Å.
A.J. Edwards, *J. Chem. Soc. Dalton*, (1972) 582.

[Bis(diphenylarsino)methane] dibromodicarbonylmolybdenum(II)
($P2_1/c$) $Z = 4$, $R = 8.5\%$ for 2018 reflections. The molybdenum is seven-coordinate in a distorted capped octahedral fashion, with a carbonyl group in the cap position. Mo—C = 1.92 Å. One arsine is bi- and the other unidentate.
M.G.B. Drew, *J. Chem. Soc. Dalton*, (1972) 626.

Bis(pyridine-2-thiolato)bis(triphenyl-phosphine)ruthenium(II)
($Pna2_1$) $Z = 4$, $R = 3.8\%$ for 2633 independent reflections. The monomeric complex contains highly distorted octahedral ruthenium. The triphenylphosphine groups are *cis*

and the pyridine-2-thiolato ligands both coördinate through nitrogen and sulphur so that the sulphurs are *trans*. Ru–P = 2.319 and 2.332, Ru–N = 2.115 and 2.132, Ru–S = 2.434 and 2.437 Å; angles N–Ru–S ≃ 67°.

S.R. Fletcher and A.C. Skapski, *J. Chem. Soc. Dalton*, (1972) 635.

Pentacarbonyl[methyl(phenylthio)carbene]-chromium

($P2_1/c$) $Z = 4$, $R = 4.35\%$ for 2327 unique reflections. The structure is the first reported of a carbene complex with sulphur as hetero-substituent and is an octahedral molecule similar to other carbene complexes of chromium.

R.J. Hoare and O.S. Mills, *J. Chem. Soc. Dalton*, (1972) 653.

trans-Bis[methyldiphenylphosphine]bis(σ-pentafluorophenyl)nickel(II)

($Pbca$) $Z = 4$, $R = 3.8\%$ for 1806 independent reflections. The nickel atom is square-planar coordinated to two phosphine and two pentafluorophenyls. Ni–σ-C_6H_5 = 1.939(3), Ni–PPh_2Me = 2.206(1) Å.

M.R. Churchill and M.V. Veidis, *J. Chem. Soc. Dalton*, (1972) 670.

Tricarbonyl[(trimethylsilyl)-π-cyclopentadienyl]rhenium

($P2_1/c$) $Z = 8$, $R = 9.5\%$ for 2460 reflections. Re is octahedral. Angles OC–Re–CO range from 88 to 93(1.5)°, Re–CO from 1.81 to 1.91(4) Å, angles Re–C–O from 172 to 177(3)°. Re–(trimethylsilyl)cp ring centre = 1.95(4) Å.

W. Harrison and J. Trotter, *J. Chem. Soc. Dalton*, (1972) 678.

Dichlorotris(diethylphenylphosphine)oxomolybdenum(IV), *cis-mer*[$MoOCl_2(PEt_2Ph)_3$]

($P2_1/c$) $Z = 4$, $R = 8.1\%$ for 2283 reflections. The monomeric molecules contain octahedral molybdenum (distorted). Mo–O = 1.801(9), Mo–Cl(*trans* to O) = 2.424(4), Mo–Cl(*trans* to P) = 2.482(4), Mo–P(*trans* to Cl) = 2.522(4) and Mo–P(*trans* to P) = 2.580(5) and 2.553(5) Å.

L. Manojlović-Muir and K.W. Muir, *J. Chem. Soc. Dalton*, (1972) 686.

Bis(dimethyldithiocarbamato)nitrosylcobalt, $CoNO(S_2CNMe_2)_2$

($P2_1/c$) $Z = 4$, $R = 3.5\%$ for 1209 independent reflections. Cobalt shows tetragonal pyramidal coordination with NO apical. The Co atom is 0.52 Å above the basal plane of four sulphur atoms. The nitrosyl group is disordered with the oxygen atom lying alternatively above one or other Co–S bond of the same dithiocarbamate ligand. Co–N = 1.746(7), Co–S(mean) = 2.263(4) Å. Synthesis and NMR are discussed.

J.H. Enemark and R.D. Feltham, *J. Chem. Soc. Dalton*, (1972) 718.

Germanium tetrachloride–trimethylamine, $GeCl_4.NMe_3$

($P2_1/c$) $Z = 4$, $R = 13.4\%$ for 583 independent reflections. The discrete $GeCl_4.NMe_3$ molecules contain trigonal bipyramidal germanium with NMe_3 axial.

M.S. Bilton and M. Webster, *J. Chem. Soc. Dalton*, (1972) 722.

Dipotassium aquopentafluoroferrate(III)

(*C2/c*) $Z = 4$, $R = 7.4\%$ for 615 reflections. The structure consists of separate K^+ and $[FeF_5(H_2O)]^{2-}$ ions. Fe—O = 2.07, Fe—F(mean) = 1.92 Å.

A.J. Edwards, *J. Chem. Soc. Dalton*, (1972) 816.

cis-Dichlorobis(1-benzyl-Δ^3-phospholen)nickel(II)

(*P*2_1/*c*) $Z = 4$, $R = 9.5\%$ for 3112 independent reflections. The nickel atom is tetrahedrally distorted from square planar coordination. Ni—P(mean) = 2.154(2), Ni—Cl(mean) = 2.212(2) Å. The phospholen rings are puckered; <C—P—C(mean) = 94.0(4)°.

A.T. McPhail, R.C. Komson, J.F. Engel and L.D. Quin, *J. Chem. Soc. Dalton*, (1972) 874.

Bis(tetraethylammonium)oxopentachloroprotactinate(V)

(*Cc*) $Z = 4$, $R = 6.7\%$ for 558 independent reflections. The doubly bonding oxygen (Pa=O = 1.74 Å) and five chlorine atoms form a distorted octahedron around the protactinium atom. Pa—Cl = 2.42 to 2.72 Å.

D. Brown, C.T. Reynolds and P.T. Moseley, *J. Chem. Soc. Dalton*, (1972) 857.

Ammonium oxodiperoxoamminevanadate(V), $NH_4[VO(O_2)_2(NH_3)]$

(*Pnma*) $Z = 4$, $R = 3.1\%$ for 566 observed reflections. Vanadium is five-coordinate. Four peroxo oxygens and an ammonia nitrogen form the distorted base and vanadyl oxygen occupies the apical pyramidal position. V—O(peroxo) = 1.883(3) and 1.882(3); V=O = 1.606(3); V—NH_3 = 2.110; O—O = 1.472(4) Å.

R.E. Drew and R.W.B. Einstein, *Inorg. Chem.*, 11 (1972) 1079.

Chlorodinitrosylbis(triphenylphosphine)ruthenium hexafluorophosphate—benzene, $[RuCl(NO)_2(P(C_6H_5)_3)_2][PF_6].C_6H_6$

(*P*2_1/*c*) $Z = 4$, $R = 6.5\%$ for 1522 reflections. The ruthenium dinitrosyl cation is a distorted square pyramid with an apical N—O group. Ru—N—O = 138(2)°; Ru—N = 1.74(2) Å.

C.G. Pierpont and R. Eisenberg, *Inorg. Chem.*, 11 (1972) 1088.

Hydridonitrosyltris(triphenylphosphine)ruthenium, $RuH(NO)[(PC_6H_5)_3]_3$

(*P*2_1/*n*) $Z = 4$, $R = 6.2\%$ for 2773 reflections. The ruthenium atom is in a slightly distorted trigonal bipyramidal environment with axial nitrosyls and hydride. Ru—N = 1.79(1) Å, Ru—N—O— = 176(1)°. The three Ru—P bonds average 2.339(4) Å. The Ru atom is displaced 0.55 Å out of the trigonal plane of phosphorus atoms, towards the axial nitrosyl.

C.G. Pierpont and R. Eisenberg, *Inorg. Chem.*, 11 (1972) 1094.

Bis(*N-tert.*-butylpyrrole-2-carboxaldimino)cobalt(II)

(*Pbcn*) $Z = 4$, $R = 6.3\%$ for 656 independent reflections. Cobalt is coordinated tetrahedrally by two mutually perpendicular planar ligands. Co–N(pyrrole) = 1.981(7), Co–N(azomethine) = 2.066 Å.

C.H. Wei, *Inorg. Chem.*, 11 (1972) 1100.

Hydridopentaamminerhodium(III) perchlorate, $[RhH(NH_3)_5][ClO_4]_2$

($P2_12_12_1$) $Z = 4$, $R = 4.0\%$ for 1298 reflections. The discrete octahedral hydridopentaaminerhodium(III) cations are stretched in zig-zag chains with linear Rh–H- - -N–Rh. Rh–Rh = 6.103(1) Å, Rh–NH_3(*cis* to H) = 2.079(11) Å. Rh–NH_3(*trans* to H) = 2.244(13) Å. Rh–H = 1.82(17) Å.

B.A. Coyle and J.A. Ibers, *Inorg. Chem.*, 11 (1972) 1105.

Tris(1, 10-phenanthroline)nickel(II) pentacarbonylmanganate(–I), $[Ni(1,10\text{-phen})_3]\cdot[Mn(CO)_5]_2$

($P\bar{1}$) $Z = 2$, $R = 6.4\%$ for 2386 reflections. N–Ni–N(average) = 79.4(5)°. $Ni(phen)_3{}^{2+}$ has approximate three-fold symmetry. $Mn(CO)_5{}^-$ is trigonal bipyramidal with a small distortion in the axial plane. Mn–C = 1.77(1) to 1.82(1) Å.

B.A. Frenz and J.A. Ibers, *Inorg. Chem.*, 11 (1972) 1109.

trans-Diamminebis(*N*-methylimidazole)platinum(II) chloride dihydrate, *trans*-$[Pt(NH_3)_2(N_2C_4H_6)_2Cl_2.2H_2O]$

($P2_1/c$) $Z = 2$, $R = 4.9\%$ for 456 reflections. Imidazole rings are planar. Pt–N(imidazole) = 2.01, Pt–NH_3 = 2.07 Å.

J.W. Carmichael, N. Chan, A.W. Cordes, C.K. Fair and D.A. Johnson, *Inorg. Chem.*, 11 (1972) 1117.

Xenon(II) fluoride fluorosulphate, $FXeOSO_2F$

(*Pbca*) $Z = 8$, $R = 4.5\%$ for 838 reflections. In discrete $FXeOSO_2F$ molecules, xenon is coordinated approximately linearily by a fluorosulphate oxygen and a fluorine atom. Angle F–Xe–O = 177.4(3)°; Xe–F = 1.940(8), Xe–O = 2.155(8) Å.

N. Bartlett, M. Wechsberg, G.R. Jones and R.D. Burbank, *Inorg. Chem.*, 11 (1972) 1124.

Bis(cyclooctatetraenyl)uranium(IV), $U(C_8H_8)_2$ (A) and bis(cyclooctatetraenyl)thorium(IV), $Th(C_8H_8)_2$ (B)

Both compounds have similar crystal structures ($P2_1/c$) $Z = 2$, $R = 2.09\%$ for 543 reflections (A) and $R = 2.07\%$ for 730 reflections (B). The molecules have D_{8h} symmetry with the metal symmetrically π-bonded to two planar aromatic cyclooctatetraene dianion rings. U–C(mean) = 2.647(4), Th–C = 2.701(4) Å.

A. Avdeef, K.N. Raymond, K.O. Hodgson and A. Zalkin, *Inorg. Chem.*, 11 (1972) 1083.

Dichloro(tetramethylthiourea)selenium(II), $SeCl_2$ tmtu (A) and dibromo(tetramethylthiourea) selenium(II), $SeBr_2$ tmtu (B)

(A) is T-shaped in solution and solid state. (B) is also T-shaped with bromines in the

trans position of the T. Se—Br_1 = 2.569(3), Se—Br_2 = 2.620(3); S—Se = 2.212(4) Å.
K.J. Wynne, P.S. Pearson, M.G. Newton and J. Golen, *Inorg. Chem.*, 11 (1972) 1192.

[*N*, *N'*-Di(2-aminoethyl)malondiamidato]nickel(II)trihydrate, $Ni(C_7H_{14}N_4O_2).3H_2O$
($P2_1/c$) $Z = 4$, $R = 4.2\%$ for 3324 reflections, the four nitrogens form a distorted square with Ni. Ni—amide = 1.869(2); Ni—amine = 1921(2) Å.
R.M. Lewis, G.H. Nancollas and P. Coppens, *Inorg. Chem.*, 11 (1972) 1371.

$(+)_{546}$-*trans*-Dinitro(1, 10-diamino-4, 7-diazadecane)cobalt(III) bromide, $[Co\{NH_2(CH_2)_3NH(CH_2)_2NH(CH_2)_3NH_2\}(NO_2)_2]Br$
($P2_1$) $R = 3.8\%$. The inner coordination sphere around the cobalt atom is approximately octahedral with the tetradentate linear tetramine occupying the equatorial plane and two nitro ligands in the apices. The 5-membered chelate ring is *gauche*, absolute configuration δ. The two 6-membered chelate rings assume the chair conformation.
N.C. Payne, *Inorg. Chem.*, 11 (1972) 1376.

Bis(methinyltricobalt enneacarbonyl), $[CCo_3(CO)_9]_2$
(C_{2h}^6–$C2/c$) $Z = 4$, $R = 5.4\%$ for 1650 reflections. The crystal consists of discrete dimeric molecular units, each with a crysrallographic dyad axis. C—C = 1.37(1), Co—Co(mean) = 2.456(2) Å.
M.D. Brice and B.R. Penfold, *Inorg. Chem.*, 11 (1972) 1381.

Dicarbonylbis(triphenylphosphine)tris(toluene-3, 4-dithiolato)diiridium(III), $Ir_2(tdt)_3(CO)_2[P(C_6H_5)_3]_2$
($B\bar{1}$) 4 binuclear units/unit cell, $R = 6.1\%$ for 3218 reflections. Two Ir atoms are held together by bridging sulphur atoms of two tdt ligands which are chelated to only one of the Ir atoms. Ir—S(bridging) = 2.465(11), Ir—S(chelate ring) = 2.383(10); Ir—Ir = 3.668(3) Å.
G.P. Khare and R. Eisenberg, *Inorg. Chem.*, 11 (1972) 1385.

μ-2, 3-[2, 3-Diazabicyclo[2.2.1]heptane]diyl-bis(tricarbonyliron), $C_5H_8N_2Fe_2(CO)_6$
($P2_1/c$) $Z = 8$, $R = 5.6\%$ for 2513 reflections. Small but significant deviations from (C_s–m) symmetry. The two crystallographically discrete $C_5H_8N_2Fe_2(CO)_6$ molecules have nearly identical configurations, with two $Fe(CO)_3$ groups linked by the two ligand nitrogen atoms through Fe—M—Fe bridges. Fe—Fe = 2.490(2); N—N = 1.404(9); Fe—N = 1.906(7)–1.937(7) Å.
R.G. Little and R.J. Doedens, *Inorg. Chem.*, 11 (1972) 1392.

$[Mn(CO)_4(C_2P(C_6H_5)_3)Br]$, a phosphonium ylide complex
($P2_1/c$) (C_{2h}^5) $R = 6.33\%$ for 1214 reflections. The structure consists of discrete molecular units of octahedrally coordinated manganese with the $C_2P(C_6H_5)_3$ ligand *cis* to Br. C—C = 1.216(14) Å.

S.Z. Goldberg, E.N. Duesler and K.N. Raymond, *Inorg. Chem.*, 11 (1972) 1397.

2, 2'-Bipyridyltetrafluorosilicon(IV), $SiF_4C_{10}H_8N_2$ (A), 2, 2'-bipyridyltetrafluorogermanium(IV), $GeF_4C_{10}H_8N_2$ (B) and 2, 2'-bipyridyltetrafluorotin(IV), $SnF_4C_{10}H_8N_2$ (C)
($P2_1/c$) (No. 14) $Z = 4, R = 5.6\%$ (A); ($P2_1/m$) (No. 11) $Z = 2, R = 10.8\%$ (B); and ($P2_1/c$) (No. 14) $Z = 4, R = 3.2\%$ (C). The stable forms of the Ge and Sn complexes are possibly disordered; the crystal structures remain unsolved. All three compounds are monomeric.
A.D. Adley, P.H. Bird, A.R. Fraser and M. Onyszchuk, *Inorg. Chem.*, 11 (1972) 1402.

Bis[nitratobis(pentamethylenetetrazole)silver(I)], $[Ag(C_{12}H_{20}N_8NO_3]_2$
($P\bar{1}$) $Z = 1, R = 5.0\%$ for 1673 reflections. The coordination polyhedron around the silver atom is a distorted tetrahedron consisting of bonded nitrate and monodentate and bridging tetrazoles. The monodentate tetrazole is coordinated to the silver atom via N(4) and the bridging tetrazoles are linked to the silver atoms via N(3) and N(4). Ag–N(4) (monodentate tetrazole) = 2.238(10); Ag–N(4), Ag–N(3) (bridging tetrazoles) = 2.216(10), 2.541(10), respectively; Ag–O (nitrate) = 2.422(10) Å.
R.L. Bodner and A.I. Popov, *Inorg. Chem.*, 11 (1972) 1410.

Ammonium pentachloronitrosylruthenate(II), $(NH_4)_2[Ru(NO)Cl_5]$
($P2_12_12_1$) $Z = 4, R = 2.2\%$ for 1770 independent reflections. The anion has slightly distorted octahedral geometry with Ru–Cl *trans* to R–O shorter than *cis* Ru–Cl bonds. Ru–Cl(*trans*) = 2.357(1), Ru–Cl(*cis*) = 2.373(1)–2.379(2) Å. Angle Ru–N–O = 176.7(5)°; RuN = 1.738(2), N–O = 1.31(3) Å.
J.T. Veal and D.J. Hodgson, *Inorg. Chem.*, 11 (1972) 1420.

4-Cyanopyridinemercury(I) perchlorate, $[Hg_2(C_6H_4N)_2](ClO_4)_2$
($P2_1/c$) $Z = 2$ (dimers), $R = 10.4\%$ for 1084 independent reflections. The structure consists of almost linear $[Hg_2(C_6H_4N)_2]^+$ cations and ClO_4^- anions. Hg–Hg = 2.498(2) Å.
D.L. Kepert, D. Taylor and A.H. White, *Inorg. Chem.*, 11 (1972) 1639.

Bis(methylammonium) tetrabromoferrate(III) bromide, $(H_3CNH_3)_2[FeBr_4]Br$
($Pna2_1$) $Z = 8, R = 11.5\%$ for 1135 significant reflections. The tetrahedral $[FeBr_4]^-$ occurs as two independent forms, Fe–Br(average) 2.32 ± 0.1 Å. $[H_3CNH_3]^+$ ions are hydrogen bonded through nitrogen–hydrogen atoms to both ligand and non-ligand bromide atoms.
G.D. Sproul and G.D. Stucky, *Inorg. Chem.*, 11 (1972) 1647.

$Hg[Mn(CO)_5]_2$
($P\bar{1}$) $Z = 1, R = 5.5\%$ for 1295 independent reflections. The coordination of $Mn(CO)_5$ groups about the mercury is linear and each manganese is octahedral. The four equatorial carbonyls in each half of the molecule are bent toward the mercury. Hg–Mn–C

(equatorial)(average) = 84.3(2)°; Hg—Mn = 2.610(2), Mn—C(axial) = 1.810(14); Mn—C(equatorial) = 1.848(8) Å. C—O(mean) = 1.154(10) Å. Complete mass spectral data are presented.

M.L. Katcher and G.L. Simon, *Inorg. Chem.*, 11 (1972) 1651.

Dibromobis(2-methylpyridine)copper(II), $Cu(NC_6H_7)_2Br_2$

($P\bar{1}$) $Z = 4, R = 4.4\%$ for 1481 independent reflections. The bridging Cu—Br separation in the weakly bound dimers is 3.872(5) Å. Copper is tetragonal pyramidal with *trans* bromines and nitrogens in the basal plane and the bridging bromine axial. Cu—Cu = 4.926(6) Å. The compound has an antiferromagnetic transition. *T*(Neel) is 4.5°K.

P. Singh, D.Y. Jeter, W.E. Hatfield and D.J. Hodgson, *Inorg. Chem.*, 11 (1972) 1657.

Dinitrogenpentaamineosmium(II) chloride, $[Os(NH_3)_5N_2]Cl_2$

(*Pnma*) $Z = 4, R = 3.9\%$ for 706 independent reflections. Osmium is at the centre of a distorted octahedron consisting of six nitrogen atoms, five from ammonia and one from a dinitrogen ligand. Os—N = 2.12—2.15, Os—N_2 = 1.84 Å; N—N = 1.12 Å. <Os—N—N is 179°. IR data are discussed.

J.E. Fergusson, J.L. Love and W.T. Robinson, *Inorg. Chem.*, 11 (1972) 1662.

Neutral bis(ethylene-1, 2-dithiolene)palladium and platinum complexes, $MS_4C_4H_4$ where M = Pd or Pt

The isostructural complexes crystallize in ($P2_1/c$) $Z = 4, R = 5.9\%$ for 1349 reflections (Pd) and $R = 8.1\%$ for 1116 reflections (Pt). The metal is square-planar coordinated to sulphur with two $MS_4C_4H_4$ units in eclipsed relationship joined by Pd—Pd = 2.790(2) or Pt—Pt = 2.748(2) Å bonds to form dimers which have distorted arrangements of S atoms.

K.W. Browall, T. Bursch, L.V. Interrante and J.S. Kasper, *Inorg. Chem.*, 11 (1972) 1800.

Cis and *trans* isomers of bis(trichlorogermanyl)tetracarbonylruthenium, *trans*-$Ru(CO_4)(GeCl_3)_2$ and *cis*-$Ru(CO_4)(GeCl_3)_2$

($P2_1/A$) $Z = 2, R = 1.9\%$ for 667 independent reflections (*trans*) and ($P2_1$) $Z = 4$, $R = 4.1\%$ for 1087 reflections (*cis*). Both structures have discrete octahedral monomeric molecules. The chemically different Ru—C distances in the *cis* isomer are not significantly different from each other — 1.98 (*trans* to $GeCl_3$) and 2.00 Å (*trans* to CO) — or from that in the *trans* isomer, 1.98 Å. Ru—Ge = 2.48 Å (both compounds).

R. Ball and M.J. Bennett, *Inorg. Chem.*, 11 (1972) 1806.

[Fluoroborotris(2-aldimino-6-pyridyl)phosphine]nickel(II)—tetrafluoroborate, $[FB(ONCHC_5H_3N)P\,Ni^+][BF_4^-]$

($P2_1/c$) $Z = 4, R = 7.32\%$ for 1781 independent reflections. The cation is only slightly distorted from C_{3v} symmetry. Ni—N(aldimino) = 2.030(21), Ni—N(pyridyl) = 2.043 (20) Å. The coordination sphere of the the nickel atom defines a slightly tapered trigonal prism.

M.R. Churchill and A.H. Reis, Jr., *Inorg. Chem.*, 11 (1972) 1811.

$H_6Cu_6(PPh_3)_6.HCONMe_2$
(*Pbca*) $Z = 8, R = 9.2\%$ for 3180 independent non-zero reflections. Discrete molecular units and DMF are separated by van der Waals distances. The $H_6Cu_6(PPh_3)_6$ molecules contain a slightly distorted octahedral cluster of copper atoms. Two structurally *trans* faces are enlarged with Cu–Cu = 2.632(6)–2.674(5). The remaining Cu–Cu range from 2.494(6)–2.595(5). Each copper is bonded to PPh_3 with Cu–P = 2.217(7)–2.262(7) Å.
M.R. Churchill, S.A. Bezman, J.A. Osborn and J. Wormald, *Inorg. Chem.*, 11 (1972) 1818.

Dibromo(2-(2-aminoethyl)pyridine)copper(II), $CuBr_2(CrN_2H_{10})$
(*Pbca*) $Z = 8, R = 3.9\%$ for 1371 independent reflections. Infinite Cu–Br–Cu chains are doubly bridged by Cu–Br–Cu–Br– linkages. The Cu–Cu separation is 4.006(3) Å.
V.C. Copeland, P. Singh, W.E. Hatfield and D.J. Hodgson, *Inorg. Chem.*, 11 (1972) 1826.

Hexakis(2-imidazolidinone)cadmium(II) perchlorate
($P\bar{1}$) $Z = 1, R = 4.6\%$ for 2364 reflections. The ethyleneurea ligands are coordinated through the carbonyl oxygen and form a distorted octahedron about the cadmium with Cd–O = 2.348(5), 2.282(4) and 2.239(4) Å. O–Cd–O = 86.1–93.9°.
J.N. Brown, A.G. Pierce, Jr. and L.M. Trefonas, *Inorg. Chem.*, 11 (1972) 1830.

Bis(2-imidazolidinone)mercury(II) chloride, $HgCl_2.2(C_3H_6N_2O)$
($P\bar{1}$) $Z = 1, R = 2.7\%$ for 736 significant reflections. Infinite chains of $HgCl_2$ are tied together by ethyleneurea ligands. The mercury has slightly distorted octahedral coordination and each ethyleneurea is asymmetrically clustered owing to bonding to mercury, one through O, and the other through N.
R.J. Majeste and L.M. Trefonas, *Inorg. Chem.*, 11 (1972) 1834.

μ-Bis[2(1*H*)-tetrahydropyrimidinone]-octakis[2(1*H*)-tetrahydropyrimidinone]-dicobalt(II) perchlorate, $(CoC_{20}H_{40}O_{13}Cl_2)_2$
($P\bar{1}$) $Z = 1$ (dimer), $R = 5.3\%$ for 1911 reflections. Cobalt is octahedrally coordinated to oxygen atoms. Two ligands are shared per cobalt.
M.E. Brown, J.N. Brown and L.M. Trefonas, *Inorg. Chem.*, 11 (1972) 1836.

$\{UO_2(H_2O)[CO(NH_2)_2]_4\}(NO_3)_2$ (a neutron diffraction study)
($P2_1/c$) $Z = 4, R_w = 4.7\%$ for 3702 reflections. The uranyl ion is nearly linear with a U–O distance of 1.77(2) Å and is surrounded by a planar pentagon of five oxygen atoms, four from ureas, 2.34–2.38(1) Å and one from water, 2.46(2) Å.
N.K. Dalley, M.H. Mueller and S.H. Simonsen, *Inorg. Chem.*, 11 (1972) 1840.

10-Phenoxarsine chloride, $C_{12}H_8OAsCl$
($P2_1/c$) $Z = 4, R = 4.7\%$ for 434 reflections. As–Cl = 2.255(5), As–C(average) =

1.93(2) Å. The two C_6H_4OAs halves are planar within 0.04 Å.

J.E. Stuckey, A.W. Cordes, L.B. Handy, R.W. Perry and C.K. Fair, *Inorg. Chem.*, 11 (1972) 1846.

$C_{12}H_8OAsClOSbCl_5$

($P2_1/c$) $Z = 4, R = 3.5\%$ for 813 reflections. The arsenic atom is approximately tetrahedral, As–Cl = 2.100(6), As–O = 1.73(1) and As–C = 1.83(2) Å. The slightly distorted $SbCl_5$ has Sb–Cl = 2.34(1) Å.

R.J. Holliday, R.W. Broach, L.B. Hanely, A.W. Cordes and L. Thomas. *Inorg. Chem.*, 11 (1972) 1849.

10-Phenoxarsine sulphide, $(C_{12}H_8OAs)_2S$

($P\bar{1}$) $Z = 2, R = 3.3\%$ for 1412 reflections. The molecule has nearly planar $C_{12}H_8OAs$. As–S = 2.267(3), 2.282(3) Å. <As–S–As is 99.87(6)°.

W.K. Grindstaff, A.W. Cordes, C.K. Fair, R.W. Perry and L.B. Handy, *Inorg. Chem.*, 11 (1972) 1852.

$(C_6H_5)CCo_3(CO)_6\pi\text{-}C_6H_3(CH_3)_3$

($Pnma$) $Z = 4, R = 5.7\%$ for 969 reflections. The plane of the mesitylene ring is almost normal to the line joining the nearest Co atom to the centroid of the tetrahedral Co_3C group. Co–Co = 2.441(2) to 2.477(3); Co–C(mesitylene)(average) = 2.15(3) Å.

R.J. Dellaca and B.R. Penfold, *Inorg. Chem.*, 11 (1972) 1855.

cis-β-Benzoylvinylmercury(II) chloride

($P2_1/b$) $Z = 4, R = 13.6\%$ for 576 reflections. The molecule has a *cis* configuration and a cisoidal conformation relative to the C(2)–C(3) bond. Hg–O = 2.88, Hg–Cl =2.37, Hg–C = 2.23 Å.

L.G. Kuzmina, N.G. Bokii, M.I. Rybinskaya, Yu.T. Struchkov and T.V. Popova, *J. Struct. Chem. (USSR)*, 12 (1971) 943.

Triphenyl-7-cyclohepta-1, 3, 5-trienyltin

($P\bar{1}$) $Z = 2, R = 6.1\%$ for 2380 reflections. The molecule consists of an approximately tetrahedral triphenyltin ligand σ-bonded to a monocyclic seven-membered ring with alternating C–C distances indicative of double bond character.

J.E. Weidenborner, R.B. Larrabee and A.L. Bednowitz, *J. Amer. Chem. Soc.*, 94 (1972) 4140.

11, 13-Dimethyl-1, 4, 7, 10-tetraazacyclotrideca-10, 12-dienato-nickel(II) perchlorate, $[NiAT]ClO_4$

($Pbcn$) $Z = 4, R = 7.2\%$ for 659 independent reflections. The nickel is square planar with a slight tetrahedral distortion. Ni–N(trigonal) = 1.83(1) and Ni–N(tetrahedral) =

1.88(1) Å.
M.F. Richardson and R.E. Sievers, *J. Amer. Chem. Soc.*, 94 (1972) 4134.

12-Oxa[4.4.3]propella-2, 4, 7, 9-tetraene bis(iron tricarbonyl), $(C_{12}H_{12}O)Fe_2(CO)_6$
($P\bar{1}$) $Z = 2, R = 3.5\%$ for 3288 reflections. Each iron tricarbonyl is coordinated to a cyclic diene system. One system is perturbed by interaction with a pair of electrons on the oxygen atom of the propellane.
G.I. Birnham, *J. Amer. Chem. Soc.*, 94 (1972) 2455.

Tetrakis(1-isopropyltetrazol-5-ato)aurate(III) anion in the salt $[As(C_6H_5)_4][Au(CN_4R)_4]$-($R = i\text{-}C_3H_7$)
($P2_1$) $Z = 2, R = 6.0\%$ for 1426 independent reflections. The four tetrazole rings are coordinated in square planar fashion around the Au^{III} with tetrazole rings oriented such that four rings lie in two mutually perpendicular planes with each pair of *trans* rings coplanar. Au—C = 1.95(4)—2.00(4) Å.
W.P. Fehlhammer and L.F. Dahl, *J. Amer. Chem. Soc.*, 94 (1972) 3370.

Di-μ-azido-bis(diazidopalladate(II)) anion, $[Pd_2(N_3)_6]^{2-}$
($P\bar{1}$) $Z = 1, R_1 = 5.8\%$ for 2183 independent reflections. The anion contains a symmetrically planar Pd_2N_2-bridged system with square-like environment of two terminal and two bridging azide nitrogen atoms about each palladium(II). Pd—Pd = 3.142 Å.
W.P. Fehlhammer and L.F. Dahl, *J. Amer. Chem. Soc.*, 94 (1972) 3377.

$Fe_4(h^5\text{-}C_5H_5)_4(CO)_4$
($Pbca$) $Z = 8, R = 3.5\%$ for 2266 independent reflections. The molecule contains the cubane-like M_4X_4 structure, in which X is a symmetrically coordinated, triply bridging carbonyl group. The four iron atoms are positioned at the vertices of a bonded tetrahedron of average length 2.520 Å which is inter-locked with a non-bonded tetrahedron of carbonyl carbon atoms of average length 3.046 Å such that the two interpenetrating tetrahedra form a distorted cube with 12 Fe—C edges of average length 1.986 Å.
M.R. Neuman, T. Troan and L.F. Dahl, *J. Amer. Chem. Soc.*, 94 (1972) 3383.

Disulphurbis(bis(diphenylphosphino)ethane)iridium(1) chloride—acetonitrile, $[Ir(S_2)((C_6H_5)_2PCH_2CH_2P(C_6H_5)_2)_2]Cl.CH_3CN$
($P\bar{4}2_1c$) $Z = 4, R = 5.0\%$ for 3041 independent reflections. The cation has approximately trigonal-bipyramidal coordination and is nearly isostructural with the previously reported oxygen carriers $[M(O_2)((C_6H_5)_2PCH_2P(C_6H_5)_2)_2]^+$ (M = Ir, Rh). The disulphur is equatorial. Ir—S(average) = 2.41(2), Ir—P = 2.33—2.37 Å.
W.D. Bonds, Jr. and J.A. Ibers. *J. Amer. Chem. Soc.*, 94 (1972) 3413.

μ-Oxo-bis[α, β, γ, δ-tetraphenylporphinato iron(III)]

(*C2ca*) $Z = 4$, $R = 7.2\%$ for 3501 independent reflections. The binuclear $N_4FeOFeN_4$ coordination group of the oligomer approximates D_{4d} symmetry. Fe—O = 1.7631(1), Fe—N(average) = 2.087(3) Å. Each iron is square pyramidal.

A.B. Hoffman, D.M. Collins, V.W. Day, E.B. Fleischer, T.S. Srivastava and J.L. Hoard, *J. Amer. Chem. Soc.*, 94 (1972) 3620.

$(C_5H_5)Rh(C_2F_4)(C_2H_4)$

(*P*2_1/*c*) $Z = 4$, $R = 3.1\%$ for 1465 observed reflections. The molecule has idealised C_s symmetry with rhodium coordinated to a cp ring, an ethylene ligand and a tetrafluoroethylene ligand. Rh—cp = 1.899; Rh—C(ethylene) = 2.167, C—C(ethylene) = 1.358 (9); Rh—C(tetrafluoroethylene) = 2.024; C—C(tetrafluoroethylene) = 1.405 Å.

L.J. Guggenberger and R. Cramer, *J. Amer. Chem. Soc.*, 94 (1972) 3779.

trans-Diiodobis(*t*-butylisonitrile)-palladium(II)

(*Pbca*) $Z = 8$, $R = 9.3\%$ for 839 reflections. The molecule has a *trans* square planar geometry in which isonitrile groups are linear.

N.A. Bailey, N.W. Walker and J.A.W. Williams, *J. Organometal. Chem.*, 37 (1972) C49.

Monoclinic trisodium tris(pyridine-2, 6-dicarboxylato)ytterbate(III)-13(hydrate), $Na_3[Yb(C_7H_3NO_4)_3].13H_2O$

(*P*2_1/*c*) $Z = 4$, $R = 10.2\%$ for 2300 independent reflections. The metal is surrounded by six carboxylate oxygen atoms and three nitrogens which form a tricapped trigonal prism. Yb—O = 2.34—2.43; Yb—N = 2.50—2.53 Å.

J. Albertssen, *Acta Chem. Scand.*, 26 (1972) 983.

Triclinic trisodium tris(pyridine-2, 6-dicarboxylato)neodymate(III)-15(hydrate), Na_3-$[Nd(C_7H_3NO_4)_3].15H_2O$

($P\bar{1}$) $Z = 2$, $R = 12.6\%$ for 2366 reflections. The Nd is surrounded by six carboxylate oxygen atoms and three nitrogens forming a tri-capped trigonal prism. Nd—O = 2.37—2.61, Nd—N = 2.55—2.59 Å.

J. Albertsson, *Acta Chem. Scand.*, 26 (1972) 1023.

Dirubidium-1, 1, 2, 2-tetranitro-ethanediide

(*PccN*) $Z = 4$, $R = 7.7\%$ for 926 reflections. The anion $RC(NO_2)^{2-}$ has a planar $C(NO_2)^{2-}$ group with $R{=}C(NO_2)^{2-}$ twisted 68° from a planar conformation. The cation is bonded to eight oxygen atoms of six different anions in an irregular arrangement.

B. Kleive, *Acta Chem. Scand.*, 26 (1972) 1049.

Basic mercury perchlorate, $Hg_2OOHClO_4$

(*P*2_1/*c*) $Z = 4$, $R = 11.7\%$ for 2350 reflections. Each mercury atom forms two short Hg—O bonds, 2.0 Å and <O—Hg—O = 180°. Other Hg—O contacts are longer than

2.7 Å. The characteristic OHg_3 groups can be distinguished.
G. Johansson and E. Hansen, *Acta Chem. Scand.*, 26 (1972) 796.

catena-μ-Ethylenediaminecadmium(II) tetracyanoniccolate(II) dibenzene clathrate,. $Cd(en)Ni(CN)_4.2C_6H_6$
($P4/m$) $Z = 1, R = 8.9\%$ for 619 reflections. Each en molecule bridges between two cadmium atoms in the adjacent layers to make a three-dimensional host lattice.
T. Miyoshi, T. Iwamoto and Y. Sasaki, *Inorg. Chim. Acta*, 6 (1972) 59.

Bis(cyclopentamethylenedithiocarbamato)Fe^{II} dicarbonyl, $(Cpdtc)_2Fe(CO)_2$
($Pbca$) $Z = 8, R = 9.3\%$ for 983 independent reflections. The molecule contains six-fold coordinated Fe^{II} in which the carbonyl groups are *cis* to each other and the limited ligand bite distorts the octahedron. Two dtc ligands consist of nearly planar fragments making an angle of 88.8° to each other. The piperidine rings are in chain form.
J.S. Ricci, Jr., C.A. Eggers and J. Bernal, *Inorg. Chim. Acta*, 6 (1972) 97.

Trimethylphenylammonium nonachlorodirhodate(III), $[(CH_3)_3C_6H_5N]_3Rh_2Cl_9$
($Pmc2_1$) $Z = 12, R = 9.5\%$ for 4113 independent reflections. The $Rh_2Cl_9{}^{3-}$ ion has virtual D_{3h} symmetry and shows symmetry-preserving distortions from the ideal bioctahedron structure indicative of Rh–Rh repulsion. Rh–Rh = 3.121(5) Å, <Rh–Cl–Rh(average) = 81.3(3)°.
F.A. Cotton and D.A. Ucko, *Inorg. Chim. Acta*, 6 (1972) 161.

Phenylmercury(II) acetate
($P2_1/c$) $Z = 4, R = 11.1\%$ for 551 independent reflections. The mercury atom is covalently bound to the phenyl ring on one side and to the acetate–oxygen atom on the other. Hg–C = 1.92(6), Hg–O = 2.11(4) Å; <C–Hg–O = 170(2)°.
B. Kamenar and M. Penavic, *Inorg. Chim. Acta*, 6 (1972) 191.

Tris(dithioacetylacetonato)rhodium(III)
($C2/c$) $Z = 8, R = 11\%$ for 3117 reflections. Each rhodium atom is surrounded by six sulphur atoms at 2.314(3)–2.333(3) Å arranged in a distorted octahedron. The ligand dimensions are normal.
R. Beckett and B.F. Hoskins, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 683.

trans-Dinitrobis(triethylphosphine)-platinum(II)
($Pca2_1$) $Z = 4, R = 9.9\%$. The complex is *trans* square planar with the two NO_2^- groups N-coordinated and lying in a plane perpendicular to the equatorial plane.
R. Graziani, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 701.

2, 2-Dicyanovinylferrocene, $C_5H_5FeC_5H_4CH{=}C(CN)_2$
($P2_1/c$) $Z = 4, R = 7.3\%$ for 884 observed reflections. The almost parallel rings are

separated by 3.28 Å and deviate 7° from the eclipsed conformation. The substituent vinyl group is turned 13° away from the ring plane.

A.P. Krukonis, J. Silverman and N.F. Yannoni, *Acta Crystallogr., Sect. B*, 28 (1972) 987.

Bis(amidoxalato-*O, O*)-cobalt(II) tetrahydrate

($P2_1/c$) $Z = 2, R = 3.7\%$ for 833 independent reflections. In this *trans* octahedral complex the amidoxalato anion forms chelate pentatomic rings with amidic oxygen and carboxylate oxygen as donor atoms. The remaining octahedral positions are occupied by waters.

M.A. Pellinghelli, A. Tiripicchio and M. Tiripicchio-Camellini, *Acta Crystallogr., Sect. B*, 28 (1972) 998.

Lead diethyldithiophosphate, $Pb[S_2P(OC_2H_5)_2]_2$

($P\overline{1}$) $Z = 2, R = 8.6\%$ for about 2000 independent reflections. The two ligands are coordinated to lead with the four sulphur atoms in a distorted tetragonal-pyramidal environment. Pb–S = 2.754, 2.790, 2.990 and 3.022(6) Å.

T. Ito, *Acta Crystallogr., Sect. B*, 28 (1972) 1034.

N-(1-Methyl-3-oxo-butyliden)-*N'*-(1-methyl-2-isonitroso-3-oxobutyliden)-ethylenediamine copper(II), $[Cu^{II}(C_{12}H_{17}N_3O_3)]$

($P\overline{1}$) $Z = 2, R = 5.1\%$ for 2538 independent reflections. Copper is coordinated by two oxygens (1.887 and 1.926 Å) and two nitrogen atoms (1.937 and 1.937 Å) in a square planar array. The nitric oxide does not behave as a free ligand.

M.B. Cingi, A.C. Villa, A.G. Manfredotti, C. Guastini and M. Nardelli, *Acta Crystallogr., Sect. B*, 28 (1972) 1075.

Trichlorotris (3-sulphanilamido-6-methoxypyridazine)bismuth(III)

($R3$) $Z = 3, R = 5.9\%$ for 953 independent reflections. The bismuth atom is on a three-fold axis surrounded by three chlorine atoms, Bi–Cl = 2.529(8); three nitrogen atoms, Bi–N = 2.90(1); and three oxygen atoms, Bi–O = 3.09(2) Å of three sulphamido groups from three different organic molecules.

M.B. Ferrari, L.C. Capacchi, L. Cavalca and G.F. Gasparri, *Acta Crystallogr., Sect. B*, 28 (1972) 1169.

3-(2-Diethylammoniumethoxy)-1, 2-benzidothiazole tetrachlorocobaltate, $(C_{13}H_{19}N_2OS)_2{}^{+}\cdot[CoCl_4]^{2-}$

($P\overline{1}$) $Z = 2, R = 8.7\%$. The organic cation shows no coordinative interaction with the metal, which forms a slightly distorted tetrahedral tetrachloro cobaltate anion.

A.C. Bonamartini, M. Nardelli and C. Palmieri, *Acta Crystallogr., Sect. B*, 28 (1972) 1207.

Cs_2MnBr_4

($Pnma$) $Z = 4, R = 10.8\%$ for 380 reflections. The structure is isomorphous with that of Cs_2ZnBr_4.

J. Goodyear, G.A. Stagmann and D.J. Kennedy, *Acta Crystallogr., Sect. B,* 28 (1972) 1231.

1:1 Compound formed between PCl_5 and $TeCl_4$, Cl_9PTe
(*I2mb*) $Z = 8, R = 12.2\%$ for 1408 independent reflections. The structure consists of discrete, approximately tetrahedral, $[PCl_4]^+$ cations and polymeric chain anions in which each tellurium atom is surrounded by a distorted octahedron of chlorine atoms.
P.H. Collins and M. Webster, *Acta Crystallogr., Sect. B,* 28 (1972) 1260.

Bis(thioglycinato)nickel(II), $Ni(C_2H_4NOS)_2$
($P2_1/c$) $Z = 4, R = 13.9\%$ for 1524 independent reflections. In the two chemically similar but crystographically non-equivalent molecules the nickel atom is square planar.
J.R. Ruble and K. Seff, *Acta Crystallogr., Sect. B,* 28 (1972) 1272.

Oxodiperoxo-hexamethylphosphoramido-aquo-molybdate(I), $MoO(O_2)PO[N(CH_3)_2]_3H_2O$ (A) and oxodiperoxo-hexamethylphosphoramido-pyridino-molybdate(VI), $MoO(O_2)PO$-$[N(CH_3)_2]_3H_2O$ (B)
($P2_1/c$) $Z = 4, R = 3.2\%$ for 2431 independent reflections (A) and (*Pbca*) $Z = 8, R = 4.8\%$ for 1330 independent reflections (B). In each compound the molybdenum atoms are surrounded by seven atoms forming a pentagonal bipyramid. The equatorial plane contains the two peroxo groups and the phosphoramide oxygen. The apical positions are occupied by a double bonded oxygen and a water in (A) and a pyridine molecule in (B).
J.M. LeCarpentier, R. Schliepp and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 1278.

The pyridinium salts of μ-oxo-tetraperoxo-dioxo-diaquo-dimolybdate(VI), $[pyH^+]_2$-$O[MoO(O_2)_2H_2O]_2^{2-}$ (A) and μ-hydroperoxo-tetraperoxo-dioxo-dimolybdate(VI), $[pyH^+]_2]MoO(O_2)_2OOH]_2^{2-}$ (B)
(*Cc*) or (*C2/c*) $Z = 4, R = 3.5\%$ for 1799 reflections (A) and ($P\bar{1}$) $Z = 1, R = 3.2\%$ for 2381 independent reflections (B). In each compound molybdenum is surrounded by seven oxygen atoms in a pentagonal bipyramidal arrangement. In (A) the anion consists of two pentagonal bipyramids joined by one corner and in (B) of two bipyramids sharing one edge.
J.M. LeCarpentier, A. Mitschler and R. Weiss, *Acta Crystallogr., Sect. B,* 28 (1972) 1288.

Tetrakis(dithioacetato)vanadium(IV), $V(CH_3CS_2)_4$
(*P2/c*) $Z = 4, R = 12\%$ for 850 independent reflections. The structure confirms the existence of VS_8 chromophores with V–S distances of two types $V–S_A$, 2.50 and $V–S_B$, 2.46 Å. Two non-equivalent stereoisomeric complexes of the D_{2d} and C_2 symmetry classes lie on two-fold axes in the structure.
L. Fanfani, A. Nunzi, P.F. Zanazzi and A.R. Zanzari, *Acta Crystallogr., Sect. B,* 28 (1972) 1298.

Bis(L-histidinato)zinc(II) dihydrate, $Zn(C_6H_8N_3O_2)_2.2H_2O$, refinement
R = 7.1% and inclusion of hydrogen atoms improved the model.
T.J. Kistenmacher, *Acta Crystallogr., Sect. B*, 28 (1972) 1302.

β-$Na_2Cr_2O_7$
($P\bar{1}$) $Z = 4, R$ = 9% for about 1700 reflections. The two dichromate ions are normal. Cr–O(bridging) = 1.78(1), Cr–O(terminal) = 1.61(1) Å; <O–Cr–O = 109(1)° and <Cr–O–Cr = 131(1)°.
N.Ch. Panagiotopoulos and I.D. Brown, *Acta Crystallogr., Sect. B*, 28 (1972) 1352.

The structure of the methyloxocarbonium ion in the salts $SbCl_6^-CH_3CO^+$, $AlCl_4^-CH_3CO^+$ and $GaCl_4^-CH_3CO^+$
The crystal structures provide proof of an ionic structure in the solid state. In each salt, the methyl carbonium ion is linear and no association of the oxocarbonium ion with the anions other than the ionic crystal packing is observed, in accordance with the IR spectrum.
J.M. LeCarpentier and R. Weiss, *Acta Crystallogr., Sect. B*, 28 (1972) 1421.

$GaCl_4^-CH_3CH_2CO^+$ (A) and $SbCl_6(CH_3)_2CHCO^+$ (B)
($P2_1/c$) $Z = 4, R$ = 5.2% for 802 observed reflections (A) and ($P2_12_12_1$) $Z = 4, R$ = 3.9% for 1298 observed reflections (B). Values for C–C and C–O bond lengths are very similar to those obtained in the methyl oxocarbonium ion. In each compound there is no association of the oxocarbonium ion with its counterion.
J.M. LeCarpentier and R. Weiss, *Acta Crystallogr., Sect. B*, 28 (1972) 1430.

$AlCl_3.CH_3CH_2COCl$
($P2_1/6$) $Z = 4, R$ = 4.3% for 731 observed reflections. The adduct consists of an $AlCl_3$ group coordinated with the oxygen of the propionyl chloride.
J.M. LeCarpentier and R. Weiss, *Acta Crystallogr., Sect. B*, 28 (1972) 1437.

$2SbCl_5.COClCH_2CH_2COCl$
($P2_1/c$) $Z = 2, R$ = 5.8% for 1434 observed reflections. The adduct is composed of two $SbCl_5$ groups coordinated with the two oxygen atoms of the dicarboxylic acid chloride. The nature of the donor–accepter complex is discussed.
J.M. LeCarpentier and R. Weiss, *Acta Crystallogr., Sect. B*, 28 (1972) 1442.

Comparison of $MgSO_4.5H_2O$ with $CuSO_4.5H_2O$ and $MgCrO_4.5H_2O.MgSO_4.5H_2O$
($P\bar{1}$) $Z = 2, R$ = 5.7% for 1366 observed reflections. The Mg coordination octahedra and sulphate tetrahedra form chains of composition $Mg(OH_2)_4SO_4$. The salt should be formulated as $[Mg(OH_2)_4.SO_4].H_2O$. $CuSO_4.5H_2O$ is isostructural with $MgSO_4.5H_2O$.
W.H. Baur and J. Rolin, *Acta Crystallogr., Sect. B*, 28 (1972) 1448.

Glycine silver(I) nitrate, $NH_2CH_2COOH.AgNO_3$ (in the non-ferroelectric phase)
$(P2_1/a)$ $Z = 4, R = 11\%$. The centrosymmetrically related silver atoms are separated by 2.88 Å and bridge centrosymmetric pairs of carboxyl groups forming dimers of glycines. The glycines exist in zwitterion form.
J.K. Mohana Rao and M.A. Viswamitra, *Acta Crystallogr., Sect. B,* 28 (1972) 1484.

Tris(*O, O'*-dimethyldithiophosphato)cobalt(III), $Co[(CH_3O)_2PS_2]_3$
$(C2/c)$ $Z = 4, R = 6.9\%$ for 1884 reflections. The molecule has a two-fold axis through the Co atom and one P atom, and the six dimer S atoms form a distorted octahedron. The S–P, P–O, O–C bonds average 1.983, 1.574 and 1.440 Å respectively. Co–S (average) is 2.322 Å.
J.F. McConnell and A. Schwartz, *Acta Crystallogr., Sect. B,* 28 (1972) 1546.

Tris-sarcosine calcium chloride, $CaCl_2.3(CH_3.NH_2^+CH_2COO^-)$
$(Pnma)$ $Z = 4, R = 5.9\%$ for 1116 reflections. The Ca atom is located in a mirror plane and is coordinated by six oxygen atoms in an octahedral array.
T. Ashida, S. Bando and M. Kakudo, *Acta Crystallogr., Sect. B,* 28 (1972) 1560.

A sodium triborate modification, β-$Na_2O.3B_2O_3$
$(P2_1/c)$ $Z = 6, R = 4.3\%$ for 2275 independent reflections. The borate polyanion in this phase is made up of pentaborate, triborate and BO_4 tetrahedra. B–O(average) = 1.474 (tetrahedral) and 1.368 Å (triangular).
J. Krogh-Moe, *Acta Crystallogr., Sect. B,* 28 (1972) 1571.

Carbonatodiamminecopper(II), $Cu(NH_3)_2CO_3$
$(P2_1/c)$ $Z = 4, R = 3.2\%$ for 932 significant reflections. The copper atom is tetragonal pyramidal with two ammine nitrogens and two oxygens from one carbonate in the basal plane. The copper atom of each formula unit is bonded to the adjacent unit through the terminal carbonate oxygen atom. CuO = 2.303(2), Cu–N = 1.971(2) and 1.984(2), Cu–O(bidentate) = 1.989(2) Å; <N–Cu–N = 97.80(12)°, O–Cu–O = 66.38(8)°.
M.H. Meyer, P. Singh, W.E. Hatfield and D.J. Hodgson, *Acta Crystallogr., Sect. B,* 28 (1972) 1607.

$Al_4N_4(C_6H_5)_8$
$(I4_1/a)$ $Z = 4, R = 11.3\%$ for 732 independent reflections. The compound has a cubical Al_4N_4 skeleton with Al and N atoms at alternate vertices of a cube of edge 1.91 Å and one phenyl group attached to each atom of the cube.
T.R.R. McDonald and W.S. McDonald, *Acta Crystallogr., Sect. B,* 28 (1972) 1619.

Nickel ethylenedithiodiacetate dihydrate (NEDDD), $(OOCCH_2SCH_2CH_2SCH_2COO)$-$Ni.2H_2O$
$(P4_12_12_1)$ $Z = 4, R = 8.5\%$ for 656 independent reflections. Two *cis* sulphurs, two

trans carboxylic oxygens and two *cis* water molecules are coordinated to nickel in a slightly distorted octahedral arrangement. Ni–S = 2.44, Ni–O = 2.01, Ni–OH_2= 2.04 Å.
J. Podlahová, J. Loub and C. Novak, *Acta Crystallogr., Sect. B,* 28 (1972) 1623.

Cu_2CdSiS_4
($Pnm2_1$) $Z = 2, R = 3.3\%$ for 2440 reflections. Half the tetrahedral voids of the hexagonal close sulphur packing are regularly occupied by two Cu, one Cd and one Si atom, while the coordination of the electropositive elements by sulphur is also tetrahedral. The structure is a normal tetrahedral one.
G. Chapus and A. Niggli, *Acta Crystallogr., Sect. B,* 28 (1972) 1626.

Hydrogen tetracyanoaurate(III) dihydrate, $HAu(CN)_4.2H_2O$
($P2_1/c$) $Z = 2, R = 4.3\%$ for 646 observed reflections. The structure consists of square planar gold tetracyanide ions linked by $H_5O_2^+$ ions in which the O–O distance is 2.47 Å. The $H_5O_2^+$ ion is inferred to be in the *trans* conformation from the positions of the nitrogen atoms, which are hydrogen bonded at 2.71 and 2.75 Å.
R.A. Penneman and R.R. Ryan, *Acta Crystallogr., Sect. B,* 28 (1972) 1629.

Bis(cyclopentadienyl)beryllium (–120°C), $Be(C_5H_5)_2$
($P2_1/n$) $Z = 2, R = 10.5\%$ for 272 independent reflections. The two cp rings are related by a centre to form a slip sandwich. The normal distance between the rings is 3.33 Å. Be is π-bonded to one ring at 1.94(4) Å and appears to be σ-bonded to the other ring at 1.85(5) Å. This Be–C bond is normal to the plane of the ring.
C. Wong, T. Lee, K. Chao and S. Lee, *Acta Crystallogr., Sect. B,* 28 (1972) 1662.

Zinc isopropylxanthate, $Zn(S_2CO{-}i{-}C_3H_7)_2$
($P\bar{1}$) $Z = 4, R = 6.8\%$ for 1550 independent reflections. The four molecules in the unit cell are linked together by –Zn–S–C–S–Zn– bridges to form a tetramer with a 16-membered ring. The zinc atom is tetrahedral and Zn–S bond lengths are 2.307–2.408 Å.
T. Ito, *Acta Crystallogr., Sect. B,* 28 (1972) 1697.

sym-trans-Di-μ-phenoxyhexaphenoxydiphenolatodititanium(IV), $[Ti(OC_6H_5)_4HOC_6H_5]_2$
($P2_1/n$) $Z = 2, R = 8.1\%$ for 1076 observed reflections. The dimer has two titanium atoms bridged by two phenoxy groups. The three phenoxy Ti–O bonds are 1.789(9), 1.842(11) and 1.884(10) Å. The coordination around titanium is a distorted octahedron.
G.W. Svetich and A.A. Voge, *Acta Crystallogr., Sect. B,* 28 (1972) 1760.

Octammine-μ-amido-μ-hydroxodicobalt disulphate dihydrate
($P\bar{1}$) $Z = 1, R = 2.7\%$ for 1229 reflections (pure compound) and ($P\bar{1}$) $Z = 1, R = 3.6\%$

for 1106 reflections (mixed compound). In the mixed compound the OH bridges were partially substituted for by chlorine. In both compounds the cation is disordered about a centre of symmetry.

W.P. Schaeffer and R.A. Lighty, *Acta Crystallogr., Sect. B*, 28 (1972) 1777.

Triuranium pentaselenide, U_3Se_5

(*Pnma*) $Z = 4, R = 11.4\%$ for 615 reflections. Two environmentally different uraniums and two types of selenium have distances which range from 2.80–3.18 Å and vary with coordination number.

P.T. Moseley, D. Brown and B. Whittaker, *Acta Crystallogr., Sect. B*, 28 (1972) 1816.

Potassium$(-)_{546}$-trimethylenediaminetetraacetatocobaltate(III) dihydrate,
$(-)_{546}K\{Co[(OOCCH_2)_2N(CH_2)_3N(CH_2COO)_2]\}2H_2O$

($B22_12$) $Z = 4, R = 9.8\%$ for 888 observed reflections. The ligand is hexadentate. Cobalt is bonded octahedrally to two nitrogens and an oxygen atom from each of the four carboxylate groups of the ligand. The central six-membered trimethylenediamine cobalt ring takes a twist-boat form. The absolute conformation of the complex anion can be designated as skew pairs, ΛΔΛ.

R. Nagao, F. Marumo and Y. Saito, *Acta Crystallogr., Sect. B*, 28 (1972) 1852.

π-Cyclopentadienyl-iridium-π-duroquinone

($P2_1/b$) $R = 14.9\%$ for 1200 independent reflections. The structure is similar to the Rh analogue. Ir—cp = 2.16–2.17 Å. As a result of coordination by the metal atom, the duroquinone ligand acquires a dish conformation.

G.G. Aleksandrov and Yu.T. Struchkov, *J. Struct. Chem. (USSR)*, 12 (1971) 953.

Trihydrate of the guanidine salt of iron(III) ethylenediaminetetraacetate,
$(CN_3H_6)FeY.3H_2O$, where $Y = [C_2H_4N_2(CH_2COO)_4]^{4-}$

($P2_1/b$) $Z = 4$. The structure consists of seven-coordinate $[Fe(OH)_2Y]^-$ (in which Y appears to be hexadentate), $[CN_3H_6]^+$ cations and water of crystallization. Fe—O = 2.0, Fe—N = 2.38 Å.

Ya.M. Nesterova, T.N. Polynova, L.I. Martyneko and N.I. Pechurova, *J. Struct. Chem. (USSR)*, 12 (1971) 1028.

Tris-ethylenediamine zinc(II) thiosulphate (A) and tris-ethylenediamine cadmium(II) selenosulphate (B)

($P3_1c$) $Z = 2, R = 13.5\%$ (A) and ($P3_1c$) $Z = 2, R = 17.7\%$ for (B).

N.V. Podberezskaya and S.V. Borisov, *J. Struct. Chem. (USSR)*, 12 (1971) 1034.

$ZnSO_3.2\frac{1}{2}H_2O$

($P\bar{1}$) $Z = 4, R = 9.3\%$ for 598 reflections. The structure consists of pyramidal sulphite ions (S—O(mean) = 1.54 Å), ZnO_4 tetrahedra (Zn—O = 1.99 Å) and ZnO_6 octahedra

(Zn—O = 2.10 Å). The tetrahedral coordination around half of the zinc atoms is provided by four oxygen atoms of two SO_3^{2-} groups and four water oxygens.
B. Nyberg, *Acta Chem. Scand.*, 26 (1972) 857.

A bridged *cis*-bis(π-allyl) nickel system; the complex of nickel(0) with tricyclohexylphosphine and a trimerization product of allene
($P\overline{1}$) $Z = 2$, $R = 8.9\%$ for 4533 reflections. The coordination of the metal is trigonal planar with respect to the centres of gravity of the allyl groups. Each of the two π-allyl groups occupies a coordination position of the nickel, as does the distorted tricyclohexylphosphine tetrahedron.
B.L. Barnett, C. Krüger and Y. Tsay, *Angew. Chem.*, 11 (1972) 137.

Refinement of carbonatotetramminecobalt(III) bromide
The details of molecular geometry agree closely with the earlier refinement but the conclusion is reached that the carbonate ion does not give rise to a structural *trans* effect above the 0.02 Å level.
M.R. Snow, *Aust. J. Chem.*, 25 (1972) 1307.

Bis(L-prolinamidato)-nickel(II) dihydrate
($P2_1$) $Z = 2$, $R = 5.4\%$ for 1115 independent reflections. The nickel atom exhibits square planar coordination. Two proline—amide molecules are approximately related to each other by a two-fold axis and they coordinate to nickel in *trans* form.
T. Tsukihari, Y. Katsube, K. Fujimori and Y. Ishimura, *Bull. Chem. Soc. Jap.*, 45 (1972) 1367.

$Hg(SCN)_2$
($C2/m$) $Z = 2$, $R = 3.2\%$ for 743 reflections. The centrosymmetric mercury forms collinear Hg—S bonds to two coplanar SCN groups and completes an octahedral environment by weakly interacting with nitrogen atoms on four neighbouring molecules.
A.L. Beachamp and D. Gouter, *Can. J. Chem.*, 50 (1972) 977.

Nitrato tris(diphenylmethylphosphine)copper(I)
($P2_1/n$) $Z = 4$, $R = 8.1\%$ for 5382 independent reflections. Cu—P = 2.259(2), 2.315(2), 2.270(2); Cu—O = 2.206(6) Å.
M. Mathew, G.J. Palenik and A.J. Carty, *Can. J. Chem.*, 49 (1971) 4119.

Bis(triphenylphosphine)-(ethylene)platinum, $(PPh_3)_2PtC_2H_4$
($P2_1/a$) $Z = 4$, $R = 4.3\%$ for 4174 observed reflections. The ethylene C—C distance is 1.434(13); Pt—C = 2.116(9), 2.106(8); Pt—P = 2.265(4), 2.270(4) Å.
P.T. Cheng and S.C. Nyburg, *Can. J. Chem.*, 50 (1972) 912.

Tricarbonyl(*N*-methylpyrrole)chromium(0)

($P2_1/c$) $Z = 4, R = 5.29\%$ for 1501 independent reflections. In the typical tricarbonyl (arene) metal complex, the three $Cr{-}C_{CO}$ bonds are approximately mutually orthogonal. On the side opposite to the CO groups the pyrrole ligand is bonded to the metal with the ring plane nearly parallel to the plane formed by the carbon atom of the CO groups.

G. Huttner and O.S. Mills, *Chem. Ber.*, 105 (1972) 301.

Pentacarbonyl[dimethylamino(ethoxy) carbene]chromium(0)

($P\bar{1}$) $Z = 2, R = 5.4\%$ for 2329 reflections. The metal atom is approximately octahedrally coordinated by five carbonyl groups and the carbene moiety. C(carbene)–Cr = 2.133(4), $Cr{-}C_{CO}$ = 1.847(5) (axial) and 1.89 Å (average equatorial).

G. Huttner and B. Krieg, *Chem. Ber.*, 105 (1972) 67.

Alkali methane thiolates CH_3SM (M = Li, Na, K)

All compounds are ($P4/nmm$). CH_3SLi and CH_3SNa are of the same type as CH_3OLi and CH_3ONa but CH_3SK is of the CH_3OK type. Both types represent layer structures with CH_3S groups having an antiparallel arrangement to each other along the *c* axis.

E. Weiss and V. Joergens, *Chem. Ber.*, 105 (1972) 481.

Nonacarbonyl((phenyl)(methoxy)carbene)dimanganese(0)

($P2_1/c$) $Z = 4, R = 4.3\%$ for 2237 independent reflections. One of the equatorial carbonyl groups in $Mn_2(CO)_{10}$ is replaced by the phenyl (methoxy) carbene moiety in the complex. Mn–C(carbene) = 1.950(5), $Mn{-}C_{CO}$ = 1.796(5) Å.

G. Huttner and D. Regler, *Chem. Ber.*, 105 (1972) 1230.

$(CO)_5Cr{-}As(CH_3)_2{-}Mn(CO)_5$

($P2_1/c$) $Z = 4$, As–Cr = 2.52, As–Mn = 2.54 Å. The molecule has approximately mirror symmetry when the metal carbonyl groups rotate freely.

H. Vahrenkamp, *Chem Ber.*, 105 (1972) 1486.

Reaction product of bis(*S*-aminodithionitrito)nickel(II) with ammonia formaldehyde and methanol, $Ni(N_2S_2)_2C_4H_9NO$

($P2_1/c$) $Z = 4, R = 3.6\%$. The two planar NiN_2S_2 rings intersect with a dihedral angle of 4.5°.

V. Thewalt and C.E. Bugg, *Chem. Ber.*, 105 (1972) 1614.

Carbonatozirconate(IV) carbonatothorate(IV) and carbonatocerate(IV)

S. Voliotis, J. Faucherre and J. Dervin, *C. R. Acad. Sci.*, 274 (1972) 1163.

Uranyl diformate monohydrate

B. Claudel, B. Mentzen, A. Navarro and J. Paun, *C. R. Acad. Sci.*, 274 (1972) 1572.

Copper acetate–urea addition product, $Cu_2(CH_3COO)_4.2CO(NH_2)_2.2H_2O$

($P2_1/a$) $Z = 2$, $R = 16.7\%$. The structure consists of binuclear centrosymmetric complexes of the composition given. The four acetates bridge the two coppers. Cu—Cu = 2.637(3), Cu—O = 2.00–2.01, $Cu—O_{ur} = 2.09$ Å.

A.V. Ablov, Yu.V. Yablokov, Yu.A. Simonov, L.I. Landa, T.I. Malinovskii and L.N. Milkova, *Dokl. Chem.*, 201 (1971) 945.

cis-Cyclodecene.$\frac{1}{2}AgNO_3$ (A) and 1, 1, 4, 4-tetramethyl-*cis*-cyclodec-7-ene.$\frac{1}{2}AgNO_3$ (B)

($C2/c$) $Z = 4$, $R = 6.5\%$ for 709 reflections (A) and ($C2, 2, 2_1$) $Z = 4$, $R = 6.1\%$ for 889 reflections (B). The relative arrangement of Ag^+ cations and NO_3^- anions and cycloolefin molecules are very similar in the two crystals; however, conformations of the ring skeletons are very different.

O. Ermer, H. Eser and J.D. Dunitz, *Helv. Chim. Acta*, 54 (1971) 2469.

Primary zinc dithizonate, $Zn(HDz)_2$ (HDz = 3-mercapto-1, 5-diphenylformazan)

($P2_1/a$) $Z = 4$, $R = 12.2\%$. The molecule consists of two bidentate dithizone residues coordinated tetrahedrally to zinc through sulphur and nitrogen.

A. Mawby and H.M.N.H. Irving, *J. Inorg. Nucl. Chem.*, 34 (1972) 109.

Methyltetracarbonyltriphenylphosphinemanganese(I)

($P2_1 2_1 2_1$) $Z = 8$, $R = 12.7\%$ for 786 independent reflections. The asymmetric unit contains two molecules, both of which are in slightly distorted octahedral arrangements around the manganese atom.

A. Mawby and G.E. Pringle, *J. Inorg. Nucl. Chem.*, 34 (1972) 877.

Bis(1, 3-propanediamine)-copper(II) benzoate

($P2_1/c$) $Z = 4$, $R = 11.9\%$ for 499 reflections. Two oxygen atoms of two benzoate groups are coordinated to each central copper and complete a distorted octahedral array.

R. Uggla and M. Klinga, *Suom. Kemistilehti B*, 45 (1972) 10.

Bis(*N*-methylethylenediamine)copper(II) thiocyanate

($P2_1/n$) $Z = 4$, $R = 11.9\%$ for 1191 independent reflections. The compound has a square-based pyramidal structure with copper coordinated to four nitrogens of two *N*-methylethylenediamines, Cu—N = 2.067, 2.049, 2.029, 2.070 Å, and to nitrogen and sulphur atoms of two bridging thiocyanate groups, Cu—N = 2.238, Cu—S = 3.348 Å.

A. Pajunen and R. Hämäläinen, *Suom. Kemistilehti B*, 45 (1972) 122.

$(CH_3NH_3)_4InCl_7$

($P2/c$) $Z = 4$, $R = 7.6\%$ for 834 independent reflections. The salt consists of $InCl_6^{3-}$ and Cl^- anions and $CH_3NH_3^+$ cations.

H. Schlimper and M.L. Ziegler, *Z. Naturforsch. B*, 27 (1972) 377.

Di-μ-hydroxobis(aquotriammine cobalt(III)) nitrate dihydrate
K. Wieghardt, *Z. Naturforsch. B*, 26 (1971) 987.

Adducts of diphenylmercury with bidentate ligands: diphenylbis-(2, 4, 7, 9-tetramethyl-1, 10-phenanthroline) mercury(II) (A) and diphenylbis-(2, 9-dimethyl-1, 10-phenanthroline)-mercury(II) (B)
(*Cm*) $Z = 4$ (A) and ($P\bar{1}$) $Z \doteq 4$ (B). Both crystals are disordered. Both adducts have their planar ligands in [040] with the C—Hg—C units of Ph_2Hg moieties aligned in the [010] direction. Each Ph_2Hg has one ligand adjacent to it, Hg—N 2.8–3.0 Å, and one half of the ligands do not have a mercury adjacent to their nitrogen atoms.
A.J. Carty and B.M. Gatehouse, *Acta Crystallogr., Sect. B*, 28 (1972) 1872.

Dichloro-*N, N'*-ethylenebis(salicylideneiminato)titanium(IV)-tetrahydrofuran, $C_{16}H_{14}N_2O_2Cl_2Ti.C_4H_8O$
(*C2/c*) $Z = 4$, $R = 6.5\%$ for 1392 significant reflections. The structure consists of monomeric units of $C_{16}H_{14}N_2O_2Cl_2Ti$ and C_4H_8O. The titanium atom is approximately octahedrally coordinated to two chlorine atoms in *trans* configuration, two *cis* oxygens and two *cis* nitrogens. Ti—Cl = 2.346, Ti—O = 1.835, Ti—N = 2.141 Å.
G. Gilli, D.W.J. Cruickshank, R.L. Beddoes and O.S. Mills, *Acta Crystallogr., Sect. B*, 28 (1972) 1889.

CrP_4 and MoP_4
Both have isotropic monoclinic crystal structures, (*C2/c*) $Z = 4$. For CrP_4, $R = 3.4\%$ for 1017 observed reflections. Two non-equivalent P atoms are tetrahedrally coordinated, one by three P atoms and one Cr, the other by two P and two Cr atoms. P—P = 2.21 ± 0.02 Å. Cr is coordinated in a distorted octahedron by six P atoms. Cr—P = 2.32–2.39 Å.
W. Jeitschko and P.C. Donohue, *Acta Crystallogr., Sect. B*, 28 (1972) 1893.

Thiocyanato and isothiocyanatopenta-aminecobalt(III) dichloride, $[Co(SCN)(NH_3)_5]Cl_2.H_2O$ (A) and $[Co(NCS)(NH_3)_5]Cl_2$ (B)
($P2_12_12_1$) $Z = 4$, $R \doteq 9.7\%$ for 1109 unique reflections (A) and (*Fm3m*) (disordered) $Z = 4$, $R_1 = 7.7\%$ for 47 unique reflections (B). In (A) the CoSCN moiety is bent, the angle at sulphur is 105(1.1)°. No structural *trans* effect of the SCN group was found. All Co—N are equivalent, 1.94(average); Co—S = 2.272(7) Å. (A) transforms slowly into (B) in the X-ray beam and gives a very different diffraction pattern. (B) is octahedral and has the expected linear NCS coordination.
M.R. Snow and R.F. Boomsma, *Acta Crystallogr., Sect. B*, 28 (1972) 1908.

Calcium nitrilotriacetate dihydrate, $Ca.HN^+(CH_2COO^-)_3.3H_2O$
($P2_1/c$) $Z = 4$, $R = 3.7\%$ for 2680 reflections. Each nitrilotriacetate is protonated at the nitrogen atom and is bonded to calcium atoms through five of the six oxygen atoms.

Ca–O = 2.316–2.560(5) Å. The sixth oxygen forms a hydrogen bond of 2.767 Å with one of the two waters coordinated to the calcium. Calcium is in a pentagonal bipyramidal coordination sphere of oxygen atoms.

S.H. Whitlow, *Acta Crystallogr., Sect. B*, 28 (1972) 1914.

Bis[bis(dicyclohexylphosphino)methane]nickel, $\{[(C_6H_{11})_2P]_2CH_2\}_2$ Ni

($P2_1/c$) $Z = 4, R = 6.9\%$ for 3982 observed reflections. Nickel is in a distorted tetrahedral coordination sphere. Ni–P bonds are bent. All cyclohexyl rings are in the chair conformation and bond equatorially to the phosphorus.

C. Krüger and Y. Tsay, *Acta Crystallogr., Sect. B*, 28 (1972) 1941.

Dicarbonylnitrosyl triphenylphosphine cobalt(0), $C_{20}H_{15}CoNO_3P$

($P\overline{1}$) $Z = 2, R = 3.2\%$ for 2006 observed reflections. The coordination around cobalt is slightly distorted tetrahedral. Angles between phosphorus and nitrosyl average 113.4° and between phosphorus and nitrosyl or carbonyl 105.0°. There is no indication of intermolecular hydrogen bonding.

D.L. Ward, C.N. Caughlan, G.E. Voecks and P.W. Jennings, *Acta Crystallogr., Sect. B*, 28 (1972) 1949.

Bis(acetylacetonato-μ-allyl-platinum), $[Pt(C_3H_5)(C_5H_7O_2)]_2$ (A) and bis[di-μ-allyl-(di-μ-choro-diplatinum)], $[Pt(C_3H_5)Cl]_4$ (B)

($P2_1/c$) $Z = 4, R = 11.3\%$ for 1948 independent reflections (A) and ($P\overline{1}$) $Z = 4$, $R = 12.0\%$ for 2975 independent reflections (B). In (A) each allyl group of the Pt–Pt bridge is σ-bonded to one platinum and π-bonded to the other. Similarly diallyl bridges occur in (B) where non-planar dichloro bridges alternate with diallyl bridges in the rings. The Pt–O lengths in (B) reflect the differing *trans* influences of the σ- and π-bonded allyl group.

G. Raper and W.S. McDonald, *J. Chem. Soc. Dalton*, (1972) 265.

μ_3-Hydroxo-tri-μ-(pyridine-2-carbaldehydeoximato)-μ_3-sulphate-tricopper(II)–16.3 water

($P\overline{3}$) $Z = 2, R = 15\%$ for 2043 unique reflections. The structure is made up of trinuclear units of copper atoms centred about a three-fold crystallographic axis so that the three copper atoms fall at the corners of an exact equatorial triangle of side 3.22 Å. There are three bridging systems.

R. Beckett and P. F. Hoskins, *J. Chem. Soc. Dalton*, (1972) 291.

α-Diiodo(dimethyl) tellurium

($P2_1/c$) $Z = 12, R = 5.4\%$ for 2191 reflections. Each tellurium has a distorted octahedral environment with two iodine atoms in a *trans* arrangement, two *cis* methyls and two contacts from iodines attached to neighbouring molecules. Te–T = 2.854(3)–2.994(3), Te–C = 2.10(3)–2.16(3) Å.

L.Y.Y. Chan and R.W.B. Einstein, *J. Chem. Soc. Dalton*, (1972) 316.

Di-μ-pentafluorobenzenthiolato-*trans*-bis[(pentafuorobenzenethiolato)(triphenylphosphine)palladium(II)]

($P2_1/c$) $Z = 2, R = 12.8\%$ for 2230 independent reflections. The ligands are approximately square planar about each palladium atom and the non-bridging pentafluorobenzenthiolato-ligands are *trans*.

R.H. Fenn and G.R. Segrott, *J. Chem. Soc. Dalton*, (1972) 330.

cis-Dichlorobis(2, 2'-bipyridyl)gallium(III) tetrachlorogallate(III), $[GaCl_2(bipy)_2]^+ \cdot [GaCl_4]^-$ (formerly thought to be $GaCl_3$(bipy)

($Pccn$) $Z = 8, R = 5.2\%$ for 1907 observed reflections. The cation has a *cis* configuration, Ga–Cl = 2.265, Ga–N = 2.095 and 2.111 Å. <N–Ga–N = 77.2 ± 0.1°.

R. Restivo and G.J. Palenik, *J. Chem. Soc. Dalton*, (1972) 341.

Tetrameric copper complex of the terdentate ligand *N*-2-pyridyl-salicylaldimine

($P4_22_12$) $Z = 2, R = 7.2\%$ for 1570 independent reflections. The four coppers in the cluster are in an almost square array. Two independent Cu–Cu are 3.077(2) and 3.171(2) Å. The ligand is coordinated to three different copper atoms, the phenolic oxygen bridges two atoms and pyridine nitrogen coordinates the third. Water occupies the fourth position of a distorted square around copper while nitrate does not coordinate.

J. Drummond and J.S. Wood, *J. Chem. Soc. Dalton*, (1972) 365.

catena-μ-Oxalato-ammine copper(II), $Cu(NH_3)(C_2O_4)$

($Pbca$) $Z = 8, R = 5.0\%$ for 587 independent reflections. In the polymeric structure copper is in a distorted square bipyramidal coordination with ammonia in the more strict coordination plane. One oxalate oxygen bridges two coppers and gives rise to strong antiferromagnetism. $\mu(302°K) = 1.46$; $\mu(94.5°K) = 0.53$ B.M.

L. Cavalca, A.C. Villa, A.G. Manfredotti, A. Mangia and A.A.G. Tomlinson, *J. Chem. Soc. Dalton*, (1972) 391.

Isomorphous tris(hexafluoroacetylacetonato)copper(II) (A) and tris(hexafuoroacetylacetonato)magnesium (B) salts of monoprotonated 1, 8-*bis*(dimethylamino)naphthalene, $[C_{14}H_{19}N_2]^+ [(CF_3.CO.CH.CO.CF_3)_3M]^-$ (M = Mg or Cu)

Both have ($P2_1/n$) $Z = 4$; $R = 10.8\%$ for 2478 reflections (A) and $R = 9.5\%$ for 1998 reflections (B). In the cation the proton lies in the plane of the naphthalene ring between the two nitrogens which are slightly displaced on opposite sides of the ring and are 2.60(1) (B) and 2.65(2) Å (A) apart. Mg is octahedrally coordinated by oxygen atoms, Mg–O = 2.06(2) Å. Copper shows some tetragonal distortion; Cu–O = 2.01(2) for the four short bonds, and also 2.20(3) Å.

M.R. Truter and B.L. Vickery, *J. Chem. Soc. Dalton*, (1972) 395.

Bisaquonitratobispyridinenickel(II), $[Ni(C_5H_5N)_2(NO_3)_2(H_2O)_2]$
($P2_1/c$) $Z = 2$, $R = 4.4\%$ for 1849 reflections. The molecule is a centrosymmetric six-coordinate monomer.
A.F. Cameron, D.W. Taylor and R.H. Nuttall, *J. Chem. Soc. Dalton*, (1972) 423.

Tetracarbonyl-[1-(dimethylarsino)-2-(diphenylphosphino) tetrafluorocyclobutene-*P*]-iron
($P2_1/c$) $Z = 4$, $R = 4.0\%$ for 1149 reflections. The iron atom is in a slightly distorted trigonal bipyramid. The ligand coordinates through phosphorus in an axial position. Fe—P = 2.224(3) (axial) and Fe—C = 1.74(2) Å (equatorial).
F.W.B. Einstein and R.D.G. Jones, *J. Chem. Soc. Dalton*, (1972) 442.

Bis(α-ethylthioacetothioacetato)zinc(II)
($C2/c$) $Z = 4$, $R = 5.6\%$. The structure consists of monomeric units each having a ZnS_4 core. The metal-bonded sulphur atoms are displaced slightly from the ideal tetrahedral positions. Zn—S = 2.333(1) and 2.270(1) Å. The ligand is planar with zinc 0.47 Å out of the plane.
R. Beckett and B.F. Hoskins, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 679.

$(+)_{589}$-Tris(1, 2-dithiooxalato)cobaltate(III) ion, in $KCa(+)_{589}[Co(thiox)_3].4H_2O$
($P2_12_12_1$) $Z = 4$, $R = 6.2\%$ for 1450 reflections. The three ligands are bidentate through sulphur atoms and the complex has the Λ configuration. Co—S = 2.230—2.259(4) Å.
K.R. Butler and M.R. Snow, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 541.

Bis(*N*, *N*-diethyldithiocarbamato)dioxotrimethylamineoxide uranium(VI), $UO_2[Et_2NCS_2]_2Me_3NO$
($P2_12_12_1$) $Z = 4$, $R = 8.3\%$ for 1551 reflections. The uranium atom is in a seven-coordinate pentagonal bipyramidal environment. The linear uranyl group is perpendicular to the equatorial plane containing four sulphurs of two carbamate groups and the oxygen of the ligand at the corners of an irregular pentagon.
E. Forsellini, G. Bombieri, R. Graziani and B. Zarli, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 461.

N, *N'*-Ethylenebis(acetylacetoneiminato)(methyl)pyridinecobalt
($P2_1/c$) $Z = 4$, $R = 13\%$ for 1601 independent reflections. The cobalt atom is in a distorted octahedral coordination polyhedron. The tetradentate ligand occupies the equatorial positions and methyl and pyridine are *trans* bonded.
M. Calligaris, G. Nardin and L. Randaccio, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 477.