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X-RAY BIBLIOGRAPHY

H. CLEARY STYNES
York University, Downsview, Ontario M3J 1P3 (Canada)
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The structures of the following compounds * appeared in:

Inorg. Chem., Vol. 13, No. 3, March 1974

(i) Pentacyanocobalt(III)-μ-isocyano-pentaamminecobalt(III) monohydrate, $(NH_3)_5CoCNCo(CN)_5.H_2O$
(ii) Trichloronitrosylbis(methyldiphenylphosphine)ruthenium(II), $RuCl_3(NO)(PMePh_2)_2$
(iii) Hydrated and dehydrated manganese(II) - exchange Zeolite A

Inorg. Chem., Vol. 13, No. 4, April 1974

(i) [Bis(triphenylphosphine)iminium]tetracarbonylcyanoiron(0), $[((C_6H_5)_3P)_2N][Fe(CO)_4CN]$
(ii) $Xe_2F_{11}^+AuF_6^-$
(iii) $[Xe_2F_3^+][AsF_6^-]$ and $[XeF_5^+][AsF_6^-]$
(iv) Dinitrosylbis(triphenylphosphine)ruthenium—hemibenzene, $Ru(NO)_2(P(C_6H_5)_3)_2.\frac{1}{2}C_6H_6$
(v) Chlorotri(3-butenyl)phosphinerhodium(I), $RhClP(CH_2CH_2CH{=}CH_2)_3$
(vi) *trans*-[(Methyl)(methyl-*N*,*N*-dimethylaminocarbene)bis(dimethylphenylphosphine)platinum(II)] hexafluorophosphate, *trans*-$[CH_3\{CH_3CN(CH_3)_2\}Pt\{P(CH_3)_2C_6H_5\}_2]PF_6$

* As a consequence of the recent proliferation of automatic diffractometers and the consequent increase in the number of published papers containing X-ray structural analyses of inorganic compounds, *Coordination Chemistry Reviews* must change its policy in regard to this service. A considerable number of X-ray structural papers are published in four important, and generally available, Journals, namely *Acta Crystallographica (Section B)*, *The Journal of the American Chemical Society*, *Inorganic Chemistry*, and *The Journal of the Chemical Society, Dalton Transactions*. Beginning with this issue, *Coordination Chemistry Reviews* will list the names of the complexes whose X-ray structural analyses are published in these Journals, but will not include an abstract. Abstracts will continue to be published of structures appearing in other Journals. The Editor regrets the necessity for contracting this service and hopes it will not detract appreciably from the value of the X-ray Bibliography. In particular he apologizes to those Readers who may not have ready access to all of the above-mentioned Journals.

(vii) Copper(I) acetate, $Cu(O_2CCH_3)$
(viii) Chlorobis(triphenylphosphine)gold(I) hemibenzenate, $[(C_6H_5)_3P]_2$-$AuCl.\frac{1}{2}C_6H_6$
(ix) Tetrakis(pentafluorophenyl)tin(IV) and tetrakis(pentafluorophenyl)-germanium(IV), $(C_6F_5)_4Sn$ and $(C_6F_5)_4Ge$
(x) Bis(6-mercapto-9-benzylpurine)palladium(II)—dimethylacetamide
(xi) Bis(cyanotrihydroborato)-1,1,4,7,7-pentamethyldiethylenetriamine-copper(II), $(Me_5dien)Cu(NCBH_3)_2$
(xii) Iron(III) salicylaldimine complexes, $[Fe_2(SALPA)_2(SALPAH)_2]$.toluene

Inorg. Chem., Vol. 13, No. 5, May 1974
(i) β-Di-μ-hydroxo-bis[2-(2-dimethylaminoethyl)pyridine]dicopper(II) perchlorate, $[Cu(C_9N_2H_{14})OH]_2(ClO_4)_2$
(ii) Chloro(difluoromethyl) (*O*-chlorodifluoroacetato)carbonylbis(triphenyl-phosphine)iridium(III) benzene, $IrCl(CHF_2)(OCOCF_2Cl)(CO)(PPh_3)_2.C_6$-$H_6$
(iii) $TaH(CO)_2[(CH_3)_2PCH_2CH_2P(CH_3)_2]_2$
(iv) (*E*)-5-Methylpyridine-2-carboxaldehyde-2′-pyridylhydrazonetetracarbonyl-molybdenum(0), $Mo(CO)_4$-(*E*)-5-Me(paphy)
(v) Tri(cyclopentadienylmanganese) tetranitrosyl, $(\eta^5\text{-}C_5H_5)_3Mn_3(\mu_3\text{-}NO)(\mu_2\text{-}NO)_3$
(vi) *catena*-μ-Chloro-dichloro-μ-[*N*,*N*′-bis[2-(2-pyridyl)ethyl]-2,3-pyrazinedi-carboxamidato-N,N^N,N': $N',N^{N'}$, N^4]-dicopper, Cu_2LCl_3
(vii) Bis(diphenylarsino)methanechromium dicarbonyl, $[\{(\pi\text{-}C_6H_5)(C_6H_5)$-$AsCH_2As(C_6H_5)_2\}Cr(CO)_2]$.
(viii) Dibenzosemibullvaleneiron tetracarbonyl
(ix) Caesium tetrachlorozincate, Cs_2ZnCl_4
(x) Pentakis(2-imidazolidinone)copper(II) perchlorate, $Cu(C_3H_6N_2O)_5(ClO_4)_2$
(xi) Tetrameric triphenylphosphinecopper(I) chloride, $[PPh_3CuCl]_4$, a "cubane-like" molecule
(xii) Iodine pentafluoride at −80°C
(xiii) μ-Dimethylgermyl-μ-carbonyl-dicyclopentadienyldicarbonyldiiron, $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_2(\mu\text{-}CO)(\mu\text{-}GeMe_2)$
(xiv) Bis(η^5-cyclopentadienyl)hexacarbonylditungsten and its molybdenum analogue, $(\eta^5\text{-}C_5H_5)W_2(CO)_6$ and $(\eta^5\text{-}C_5H_5)Mo_2(CO)_6$
(xv) Bis(tetraphenyldithiomidodiphosphinato)manganese(II)
(xvi) Bis[bis(triphenylphosphine)silver(I)]bis(1,2-dicyano-1,2-ethylenedi-thiolato)nickelate(II), $[Ag(P(C_6H_5)_3)_2]_2Ni(S_2C_2(CN)_2)_2$ and bis[bis-(triphenylphosphine)silver(I)]bis(1,2-dicyano-1,2-ethylenedithiolato)-nickelate(II), $[Ag(P(C_6H_5)_3)_2]_2Ni(S_2C{=}C(CN)_2)_2$

J. Amer. Chem. Soc., Vol. 96, No. 6, March 20, 1974
(i) Diastereoisomeric forms of β_1-glycinatotriethylenetetraminecoablt(III) iodide, Δ-$(-)_{589}$-β_1-(*RR*)- and Δ-$(-)_{589}$-β_1-(*RS*)-[Co(trien)gly]I_2

J. Amer. Chem. Soc., Vol. 96, No. 7, April 3, 1974

(i) The triclinic form of 1,2,3,4,5,6,7,8-octaethylporphinatonickel(II), NiOEP

(ii) *trans*-Bromo(*trans*-styryl)bis(triphenylphosphine)platinum(II), $PtBr(HC{=}CHC_6H_5)(P(C_6H_5)_3)_2$

(iii) Di-μ-thiocyanatobis[hydrogen bis(diphenylphosphinato)]dipalladium(II), $Pd_2(SCN)_2[Ph_2PO)_2H]_2$

J. Amer. Chem. Soc., Vol. 96, No. 8, April 17, 1974

(i) Bis(monothioacetylacetonato)nickel(II), $Ni(TAA)_2$

(ii) Bis{μ-[*N*,*N*'-dimethyl-*N*,*N*'-bis(β-mercaptoethyl)ethylenediamine]}diiron(II) and bis{μ-[*N*,*N*'-dimethyl-*N*,*N*'-bis(β-mercaptoethyl)-1,3-propanediamine]}diiron(II)

(iii) Platinum carbonyl cluster dianions, $[Pt_3(CO)_3(\mu_2\text{-}CO)_3]_n^{2-}$ (n = 2, 3, 4, 5)

(iv) Hexanuclear nickel carbonyl dianion, $[Ni_3(CO)_3(\mu_2\text{-}CO)_3]_2^{2-}$

(v) Monoazide bridged dimer of Ni(*N*-tetramethylcyclam)$^{2+}$, $[Ni_2(C_{14}H_{32}N_4)_2(N_3)_3]^+ I^-$

J. Chem. Soc. Dalton, No. 1, 1974

(i) The 1 : 1 and 2 : 1 complexes of bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato)copper(II) with pyrazine, $Cu(hfac)_2(pyz)$ and $Cu(hfac)(pyz)_2$

(ii) Di-μ-carbonyl-carbonyl(tricarbonylcobalt)-π-indeyliron, $(\pi\text{-}C_9H_7)FeCo(CO)_6$

(iii) {[2-(Diphenylphosphino)ethyl]diphenylphosphine oxide}iodonitrosylcobalt(0), $Co(NO)_2\{PPh_2.(CH_2)_2.(CH_2)_2.PPh_2O\}I$

J. Chem. Soc. Dalton, No. 2, 1974

(i) Aquo-{*o*-[(2-pyridylmethylene)amino]benzamide}-[2-(2-pyridyl)-1,2,3,4-tetrahydroquinazolin-4-one]nickel(II) dinitrate tetrahydrate, $Ni(C_{26}H_{22}N_6O_2)(NO_3)_2(H_2O)_5$

(ii) *trans*-Dichlorobis-(*N*-methylsalicylaldiminato)molybdenum(IV), $Mo(sal\text{-}NMe)_2Cl_2$

(iii) *trans*-Chlorobis(dimethylphenylphosphine) (trimethylsilylmethyl)-platinum(II), $[PtCl(CH_2SiMe_3)(PMe_2Ph)_2]$

(iv) Trichlorobis-(*N*-methylthioacetamido)niobium(V), $NbCl_3[NMeC(=S)Me]_2$

(v) Bromotriscarbonyl(phenylisocyanide)manganese(I), $MnBr(CO)_3(CNPh)_2$

(vi) Hexakisantipyrineyttrium triiodide

J. Chem. Soc. Dalton, No. 3, 1974

(i) [2-(Diphenylphosphinoyl)ethyl]diethylammonium dichlorocuprate(I), $\{Ph_2P(O).[CH_2]_2.N(H)Et_2\}^+CuCl_2^-$

(ii) Cadmium(II) cyanoacetate

(iii) Aquomethylbis(dimethylglyoximato)cobalt(III), $MeCo(dmg)_2(H_2O)$

J. Chem. Soc. Dalton, No. 4, 1974

(i) Nonamethylcyclotetraphosphonitrilium pentacarbonyliodochromate(0) $[N_4P_9Me_9][Cr(CO)_5I]$

(ii) 1,5-Dihydrodecamethylcyclopentaphosphonitrilium tetrachlorocuprate-(II) monohydrate, $[(NPMe_2)_5H_2][CuCl_4].H_2O$

(iii) Tetraphenylcyclotetraphosphine monosulphide, $C_{24}H_{20}P_4S$

(iv) Acetatohydridotris(triphenylphosphine)ruthenium(II), $RuH(CO_2Me)(PPh_3)_3$

(v) α,α,γ-Trimethyl-γ-ferrocenyl-1,2-trimethyleneferrocene

(vi) μ-[Di-(2-methoxyethyl)ethercadmio]-bis(pentacarbonylmanganese)-(2*Cd—Mn*), (diglyme)Cd$[Mn(CO)_5]_2$

(vi) Bis(tetramethylamonium)octa-μ_3-carbonyl-hexacarbonyl-*octahedro*-dinickeltetracobaltate(2—), $(NMe_4)_2[Co_4Ni_2(CO)_{14}]$

(vii) Tetraethylammonium-μ-chloro-*abefh*-pentachloro-μ-nitrosyl-nitrosyldi-platinate(II), $(Et_4N)_2[Pt_2(NO)_2Cl_6]$

Cobalt arsenate, $Co_{6.95}As_{3.62}O_{16}$

(*Pnma*) $Z = 1$, $R = 7.5\%$ from 1175 independent reflections. The structure is based on a hexagonally close-packed arrangement of oxygen layers with As in tetrahedrally and Co in octahedrally coordinated interstices. As—O-(mean), 1.676 Å; Co—O(mean), 2.139, 2.174 Å.

N. Krishnamachari and C. Calvo, *Can. J. Chem.*, 52 (1974) 46.

Bis[hexakis(dimethylamino)cyclotriphosphonitrilium] tetrachlorocobaltate-(II), $[N_3P_3(NMe_2)_6H]_2CoCl_4$

($P2_12_12_1$) $Z = 4$, $R = 8.8\%$ for 2178 observed reflections. Both rings are slightly non-planar, with three distinct pairs of NP bonds: commencing at the protonated nitrogen atom, 1.68, 1.56 and 1.58 Å. The $CoCl_4^{2-}$ ion is tetrahedral and is hydrogen bonded to both rings, N—H· · · Cl 3.32, 3.36 Å

A.L. MacDonald and J. Trotter, *Can.J. Chem.*, 52 (1974) 734.

Alchemists' gold, $Hg_{2.86}AsF_6$

($I4_1/amd$) $Z = 4$, $R = 7.9\%$ using 109 independent reflections. The structur may be described as consisting of octahedral AsF_6^- ions arranged on a lattice which contains linear, non-intersecting channels in two mutually perpendicular directions. Within these channels are infinite chains of mercury atoms, each with a formal charge of +0.35, and with Hg—Hg 2.64 (1) Å.

I.D. Brown, B.D. Cutforth, C.G. Davies, R.J. Gillespie, P.R. Ireland and J.E. Vekris, *Can. J. Chem.*, 52 (1974) 791.

$H_3Os_3(CO)_9CCH_3$

(*Pnma*). X-ray powder photography. Osmium is approximately octahedral. Os—H(bridging), 1.82 Å (ave.); <Os—H—Os, 103°; Os—C, 2.08 Å; Os—Os, 2.84 Å.

J.P. Yesinowski and D. Bailey, *J. Organometal. Chem.*, 65 (1974) C27.

$K[Al_2(CH_3)_6N_3]$

($P\bar{1}$) $Z = 4$, $R = 9.5\%$ for 1631 observed reflections. In the asymmetric unit there are two geometrically different $Al_2(CH_3)_6N_3^-$ ions. One has approximate C_{2v} point symmetry and exhibits an eclipsed configuration of the methyl groups viewed down the Al—Al vector. The other belongs to the C_s point groups and shows a staggered methyl group conformation. Al—N, 2.00(1), 2.03(1); Al—C, 1.94—2.03 Å; LAl—N—Al, 128°.

J.L. Atwood and W.R. Newberry, III, *J. Organometal. Chem.*, 65 (1974) 145.

$Li_3Cr(CH_3)_6.3C_4H_8O_2$

($R3c$) $Z = 6$, $R = 12.3\%$ for 278 independent reflections. The six methyl groups form an octahedron around Cr, Cr—CH_3 2.30 Å. The octahedron is weakly distorted by interaction of three Li atoms at three edges, Li· · ·CH_3 2.17 Å. Each Li atom is further coordinated (tetrahedrally) by two oxygen atoms of two different dioxanes; Li· · ·O 2.1 Å. The dioxane molecules connect the Cr complexes.

J. Krausse and G. Marx, *J. Organometal. Chem.*, 65 (1974) 215.

Bis(triphenylphosphine)allenepalladium, $[(C_6H_5)_3P]_2Pd(C_3H_4)$

($P\bar{1}$), $Z = 2$, $R = 5.1\%$ for 4096 non-zero reflections. One of the olefinic bonds of allene is coordinated to the palladium atom: Pd—C(1), 2.118(9); Pd—C(2), 2.067(8) Å. The coordinated allene is no longer linear, the C(1)—C(2)—C(3) angle is 148.3(8)°; C(1)—C(2), 1.401(11); C(2)—C(3), 1.304(12) Å. The Pd, P(1), P(2), C(1) and C(2) atoms lie almost in the same plane.

K. Okamoto, Y. Kai, N. Yasuoka and N. Kasai, *J. Organometal. Chem.*, 65 (1974) 427.

cis-Dichlorocarbonyl(triphenylphosphine)platinum(II), *cis*-$PtCl_2(CO)(PPh_3)$

($P\bar{1}$) $Z = 2$, $R = 6.7\%$ for 4150 independent reflections. The molecules have the expected *cis* square-planar configuration. Pt—CO, 1.849(14); Pt—Cl, 2.277(3); Pt—Cl(*trans* P), 2.342(3); Pt—P, 2.279(3) Å. The geometry around the platinum atom is slightly distorted, with C—Pt—P and Cl—Pt—C angles of 95.2(4) and 172.4(3)°.

L. Manojlovic-Muir, K.W. Muir and R. Walker, *J. Organometal. Chem.*, 66 (1974) C21.

$K[Al_2(CH_3)_6F].C_6H_6$

(*Pnma*) $Z = 4$, $R = 3.7\%$ for 973 independent reflections. In the fluorine-bridged anion, the aluminium—fluorine—aluminium bond angle is fixed at 180° by crystallographic symmetry. Al—F, 1.782(2) Å. The K ion coordination sphere contains six methyl groups at distances from 3.23 to 3.47 Å; the benzene functions as a molecule of crystallization with K—C-(benzene) shortest bond 3.947(7) Å.

J.L. Atwood and W.R. Newberry, III, *J. Organometal. Chem.*, 66 (1974) 15.

Triphenylgermanium *p-tert.*-butylphenyl mercaptide, $Ph_3GeSC_6H_4$-*p*-Bu^t
($P2_1/c$) $Z = 4$, $R = 6.9\%$ for 2711 independent reflections. The compound is isostructural with its tin analogue. The coordination of the germanium is approximately tetrahedral; Ge—S, 2.229(2); Ge—C(mean), 1.938 Å.
M.E. Cradwick, R.D. Taylor and J.L. Wardell, *J. Organometal. Chem.*, 66 (1974) 43.

Bis(μ-diphenylphosphido-μ'-carbonyl-π-methylcyclopentadienylcarbonyliron) rhodium hexafluorophosphate(2*Rh—Fe*), $[Rh\{Fe(\pi\text{-}CH_3C_5H_4)(CO)_2(PPh_2)\}_2]PF_6$
(*Pbca*) $Z = 8$, $R = 6.2\%$ for 2541 independent reflections. The structure consists of a trinuclear non-closed rhodium—iron complex with μ-diphenylphosphido and carbonyl groups bridging Rh and Fe. Rh—Fe, 2.659(2), 2.674(1) Å. Each Fe is additionally bonded to a methylcyclopentadienyl group and a terminal CO. The stability of the closed structure (2*Rh—Fe*, 1*Fe—Fe*) is discussed.
R. Mason and J.A. Zubieta, *J. Organometal. Chem.*, 66 (1974) 279.

Tricyclic tetracarbonyliron carbonyltris(triphenylphosphite)diplatinum, $FePt_2(CO)_5[P(OPh)_3]_3$
($P\bar{1}$) $Z = 2$, $R = 6.2\%$ for 2353 reflections. The molecule contains a triangular $FePt_2$ cluster to which the ligands are bound as follows: four CO groups about Fe in distorted octahedral geometry, the fifth CO and the phosphite ligands about the platinums giving distorted square-planar geometries. Pt—Pt, 2.633(1); Fe—Pt, 2.550(5), 2.583(6); Pt—phosphite(mean), 2.22 Å.
V.G. Albano and G. Ciani, *J. Organometal. Chem.*, 66 (1974) 311.

$Pd(acac)_2(C_4F_6)_2$
($P2_1/n$) $Z = 4$, $R = 5.8\%$ for 4302 significant reflections. The structure consists of $Pd[O{=}C(Me)CH(COMe)C(CF_3){=}C(CF_3)]_2$ in which the hexafluoro-2-butyne links the γ-CH of the β-diketonate ligands to palladium. The Pd atom is essentially four-coordinate with two Pd—C bonds, averaging 1.993 ± 0.01 Å, and two Pd—O bonds, averaging 2.114 ± 0.01 Å, in a *cis*- square-planar arrangement.
A.C. Jarvis, R.D.W. Kemmitt, B.Y. Kimura, D.R. Russell and P.A. Tucker, *J. Organometal. Chem.*, 66 (1974) C53.

Potassium diglyme 1,3,5,7-tetramethylcyclooctatetraene dianion, $[K((CH_3OCH_2CH_2)_2O)]_2[C_8H_4(CH_3)_4]$
($P\bar{1}$), $Z = 1$, $R = 5.5\%$ for 855 reflections. This 10π electron system is aromatic with eightfold molecular symmetry, C—C(ave.) 1.407(6) Å. The structure consists of an ion trimer which lies on an inversion centre. The K ions are related by inversion and lie on either side of the cyclooctatetraene ring, K—C, 3.003(8) Å. The opposite side of each K is coordinated

by the three ether oxygens, K—O, 2.835(14) Å. All atoms in the carbocyclic ring lie in the plane.
S.Z. Goldberg, K.N. Raymond, C.A. Harmon and D.H. Templeton, *J. Amer. Chem. Soc.*, 96 (1974) 1348.

Tetraphenylarsonium tetranitratocuprate(II), $((C_6H_5)_4As)_2Cu(NO_3)_4.1CH_2Cl_2$
($P2_1/n$) $Z = 2$, $R = 6.6\%$ for 2086 independent reflections. The geometry of $Cu(NO_3)_4^{2-}$ approximates C_{2h} symmetry. The stereochemistry of the first coordination sphere may be described as either four-coordinate square coplanar, with four unidentate nitrato groups, Cu—O 1.95 Å, or eight-coordinate (not dodecahedral) with four grossly unsymmetrical bidentate nitrates, Cu· · · · · · · · · O 2.73 Å.
T.J. King and A. Morris, *Inorg. Nucl. Chem. Lett.*, 10 (1974) 237.

trans-$MoCl_3(C_5H_5N)_3$
($P2_1/c$) $Z = 4$, $R = 9.1\%$ from 1594 independent film data. Three chlorine atoms and three nitrogen atoms of the pyridine molecules form an octahedron about Mo in the 1, 2, 6 or *trans* configuration. Mo—Cl, 2.437(5), 2.424(5) and 2.423(5) Å, and Mo—N(pyridine), 2.189(13), 2.163(15) and 2.223(15) Å.
J.V. Brencic, *Z. Anorg. Allg. Chem.*, 403 (1974) 218.

Ytterbium chloride, $YbCl_2$
(*Pbca*) $R = 2.5\%$ from 452 independent reflections. The geometrical details of the coordination polyhedra around the sevenfold coordinated Yb^{2+} ion and the three- and fourfold coordinated Cl^- ions are discussed.
H. Barnighansen, H. Patow and H.P. Beck, *Z. Anorg. Allg. Chem.*, 403 (1974) 45.

Chalkogenomolybdates and -tungstates(I)
Crystallographic data of compounds of the type A_2MeX_4, A_2MeOX_3 and $A_2MeO_2X_2$ (A = K, NH_4^+, Rb, Cs; Me = Mo, W; X = S, Se) are discussed and general trends are illustrated. The molybdates and tungstates A_2MeX_4 and A_2MeOX_3, crystallize in space group (*Pnma*), and the compounds $(NH_4)_2MeO_2X_2$ in (*C2/c*).
V.A. Muller and W. Sievert, *Z. Anorg. Allg. Chem.*, 403 (1974) 251.

Chalkogenomolybdates and -tungstates(II)
The chalkogenomolybdates and -tungstates $(NH_4)_2MoS_3Se$, $(NH_4)_2MoS_2Se_2$, $(NH_4)_2MoSSe_3$, $(NH_4)_2WSSe_3$, Cs_2MoS_2Se, $Cs_2MoS_2Se_2$, Cs_2MoSSe_3, Cs_2WSSe_3, Cs_2MoOS_2Se, Cs_2WOS_2Se, $Cs_2MoOSSe_2$ and Cs_2WOSSe_2 were investigated by X-ray powder diffractometry. All compounds crystallize in space group (*Pnma*—D_{2h}^{16}) and are isomorphous with β-K_2SO_4. Systematic relations between lattice constants and occupation of atomic positions in the unit cell are discussed.
V.A. Muller and W. Sievert, *Z. Anorg. Allg. Chem.*, 403 (1974) 267.

$K_3La(NH_2)_6$
($C2/m$) $Z = 2$, $R = 3.6\%$ for 815 reflections. The amide ions are arranged in a strongly distorted cubic close-packing. All cations occupy edging anion octahedra. La—N, 2.61; K—N, 2.90—3.10 Å.
V.C. Hadenfeldt, B. Geiger and H. Jacobs, *Z. Anorg. Allg. Chem.*, 403 (1974) 319.

Diethylthioselenophosphinatothallium(I), $[Tl(Et_2PSeS)]$
($C2/m$—C_{2h}^3) $Z = 4$, $R = 3.8\%$ for 997 reflections. The structure consists of dimeric units, $[Tl(Et_2PSeS)]_2$, linked in two-dimensional polymeric layers. Each Tl is coordinated to two S and two Se atoms in the dimer and to two more distant Se atoms of neighbouring dimers. Tl—S, 3.237(5); Tl—Se, 3.424(4) Å; Tl—Se(intermolecular), 3.594(3) Å.
S. Esperas and S. Husebye, *Acta Chem. Scand.*, 27 (1973) 3355.

$Na_4H_2Mo_5P_2O_{23}(H_2O)_{10}$
($P2_1/n$) $Z = 4$, $R = 5.3\%$ based on 3897 independent reflections. The structure consists of $H_2Mo_5P_2O_{23}^{4-}$ groups linked together by direct sodium bridges (O—Na—O) in the y and z directions forming infinite layers parallel to the yz plane. The layers are held together by O—Na—H_2O—Na—O linkages. Each sodium ion is surrounded by six oxygen atoms (water oxygens and group oxygens), which form an octahedron.
B. Hedman, *Acta Chem. Scand.*, 27 (1973) 3335.

cis-Dichloro-bis(1,2-ethanediol)manganese(II), $[MnCl_2(C_2H_6O_2)_2]$
($P2_1/c$) $Z = 4$, $R = 5.3\%$ from 1969 independent reflections. The Mn^{2+} ion is octahedrally surrounded by the four glycol (1,2-ethanediol) oxygens and the two chlorine atoms. The neutral molecules $[MnCl_2(C_2H_6O_2)_2]$ are held together through hydrogen bonds of the type O—H · · · Cl. Mn—O, 2.184—2.247; Mn—Cl, 2.463, 2.464 Å.
B. Antti, *Acta Chem. Scand.*, 27 (1973) 3513.

Basic hafnium chromate, $Hf_4(OH)_8(CrO_4)_4.H_2O$
($Pnnm$) $Z = 2$, $R = 6.1\%$ based on 818 independent reflections. The hafnium atoms are joined by double oxygen bridges to form planar infinite chains with the composition $[Hf(OH)_2]_n^{2n+}$. The chains are connected by chromate groups to form a three-dimensional structure. Each hafnium is coordinated to seven oxygen atoms in a pentagonal-bipyramidal arrangement. Hf—O, 2.01—2.25 Å.
M. Hansson and W. Mark, *Acta Chem. Scand.*, 27 (1973) 3467.

Thallium(I) dimethyldithiocarbamate, $TlS_2CN(CH_3)_2$
($P2_1/a$) $Z = 4$, $R = 10.5\%$ for 786 independent reflections. The structure is composed of dimers, $[TlS_2CN(CH_3)_2]_2$, which forms layers parallel to the ab plane. Only van der Waals forces exist between successive layers, which

are stacked in the *c* direction. The thallium atoms achieve seven coordination by using sulphur atoms from five ligands in the layer. The four closest sulphur atoms, Tl—S 2.99—3.44 Å, belong to the same dimer as the thallium atom; Tl—S(intermolecular), 3.64—3.85 Å.

P. Jennische and R. Hesse, *Acta Chem. Scand.*, 27 (1973) 3531.

Potassium oxopentacyanovanadate(IV), $K_3[VO(CN)_5]$; MDO_1 structure (A) and superposition structure (B)

($Pna2_1$) $Z = 4$, $R = 8.3\%$ based on 527 reflections (A) and ($C_{mc}2_1$) $Z = 2$ (B). The structure contains discrete $[VO(CN)_5]^{3-}$ ions, V=O 1.64, V—C(equatorial) 2.14, V—C(axial) 2.31 Å. The $[VO(CN)_5]^{3-}$ ions contain a long vanadium—ligand bond *trans* to the $V—O^{2+}$ entity.

S. Jagner and N. Vannerberg, *Acta Chem. Scand.*, 27 (1973) 3482.

Diperchlorato-tetraimidazolocopper(II), $Cu(C_3H_4N_2)_4(ClO_4)_2$

($P2_1/n$) $Z = 2$, $R = 6.0\%$ for 1612 reflections. The structure contains uncharged complexes $Cu(C_3H_4N_2)_4(ClO_4)_2$. Cu—N, 1.997—2.007; Cu—O(ClO_4), 2.625 Å.

G. Ivarsson, *Acta Chem. Scand.*, 27 (1973) 3523.

Bis{[2-(2-hydroxyethyl)imine-3-oximato-butane]copper(II) perchlorate monohydrate}, $[Cu(C_6H_{11}N_2O_2)(ClO_4)(H_2O)]$

($P2_1/c$) $Z = 2$, $R = 6.8\%$ from 1602 independent reflections. The molecular unit consists of two formula units that are related by an inversion centre. Each Cu is strongly coordinated to four donor groups, two nitrogens, Cu—N 1.932(6), and two oxygens, Cu—O 2.042(6) in a plane, and weakly coordinated to two axial groups, a water oxygen at 2.500(9) Å, and a perchlorate oxygen at 2.89(2) Å. The tridentate ligands occupy the planar positions.

J.A. Bertrand, J.H. Smith and P. Gary Eller, *J. Chem. Soc. Chem. Commun.*, (1974) 95.

Molybdenum nitrosyl complex containing bridging hydrazido group, $\{(\pi\text{-}C_5H_5)Mo(NO)I\}_2(\mu\text{-}NNMe_2)$

($P2_1/c$) $Z = 4$, $R = 6.9\%$ from 3837 reflections. The molecule contains a unique asymmetric bridging hydrazido ligand, bridging two $\{(\pi\text{-}C_5H_5)MoNO\}$ units. Mo(1) is bound to the terminal N atom and Mo(2) to both N atoms of the hydrazido ligand. Mo(1)—N(1), 1.920(13); Mo(2)—N(1), 2.054(12); Mo(2)—N(2), 2.133(12); N(1)—N(2), 1.400(17); Mo—NO, 1.765(12), 1.763(13) Å.

W.G. Kita, J.A. McCleverty, B.E. Mann, D. Seddon, G.A. Sim and D.I. Woodhouse, *J. Chem. Soc. Chem. Commun.*, (1974) 132.

Nickel(II) inosine 5'-monophosphate, $[Ni(imp)(H_2O)_5.2H_2O]$

($P2_12_12_1$) $Z = 4$, $R = 8.5\%$ from 1471 unique reflections. The structure of

the Ni—nucleotide complex, Ni(imp) $(H_2O)_5.2H_2O$ shows that the octahedral nickel ion is coordinated to the N(7) atom of the hypoxanthine unit and to five water molecules, and that hydrogen bonding occurs between two of the coordinated H_2O molecules and two of the phosphate oxygen atoms; the structure previously proposed is shown to be incorrect.
G.R. Clark and J.D. Orbell, *J. Chem. Soc. Chem. Commun.*, (1974) 139.

Carbadibora-allyl nickel, $[Ni(B_7C_2H_9Me_2)(PEt_3)_2]$
($P2_1/n$) $Z = 4$, $R = 10.4\%$ for 3449 independent non-zero reflections. The geometry of $[Ni(B_7C_2H_9Me_2)(PEt_3)_2]$ is that of a *nido*-metallocarborane isoelectronic and approximately isostructural with decaborane. The nickel is joined to a B_2C system in a 1,2,3-η-bonding mode. Ni—B(7), 2.14(1); Ni—B(2), 2.11(1); Ni—C(5), 2.08(1) Å; <P(1)—Ni—P(2), 102.7(2)°; <C-(5)—Ni—B(7), 85.4(4)°; Ni—P(1), 2,242(3); Ni—P(2), 2.200(3) Å.
M. Green, J. Howard, J.L. Spencer and F.G.A. Stone, *J. Chem. Soc. Chem. Commun.*, (1974) 153.

N-Substituted porphyrin, 21-ethoxycarbonylmethyl-2,3,7,8,12,13,17,18-octaethylporphyrin
($P\bar{1}$) $Z = 2$, $R = 7.1\%$ based on 2145 independent reflections. Three geometrically different pyrrole rings are observed and the stereochemical consequences of substitution at nitrogen are discussed.
G.M. McLaughlin, *J. Chem. Soc. Perkin II*, 2 (1974) 136.

(Ethylenediamine)zinc(II) benzohydroxamate hydrate, $ZnC_{23}H_{29}N_5O_7$
($P2_1/c$) $Z = 4$, $R = 12\%$ for 3321 reflections. The zinc ion, two benzohydroxamate ions, and an ethylenediamine molecule form a distorted octahedron. There is also a H_2O molecule and a benzohydroxamic acid molecule of crystallization in the asymmetric unit. Zn—N(en), 2.134, 2.157; Zn—OC, 2.141, 2.149; Zn—ON, 2.003, 2.021 Å.
S. Gottlicher and P. Ochsenreiter, *Chem. Ber.*, 107 (1974) 391.

Bis[tricarbonyl(diethyldithiophosphinato)rhenium(I)], $[(C_2H_5)_2PS_2Re(CO)_3]_2$
($P\bar{1}$—C_i) $Z = 1$, $R = 6.2\%$ for 922 independent reflections. The dimeric structure has bridging diethyldithiophosphinato groups. Re—Re, 3.886(02); Re—S, 2.570, 2.535 and 2.534; Re—CO, 1.857, 1.519 and 1.793 Å. Each Re atom has octahedral coordination.
G. Thiele, G. Liehr and E. Lindner, *Chem. Ber.*, 107 (1974) 442.

Tris(π-cyclopentadienyl nickel)*tert.*-butylammonium, $t\text{-}C_4H_9N(\pi\text{-}C_5H_5Ni)_3$
($C2/c$) $Z = 8$, $R = 13.8\%$ for 1620 reflections. The molecule is approximately of trigonal-pyramidal structure with an apical nitrogen atom on a triangle of nickel atoms. Ni—Ni, 2.33—2.39; Ni—N, 1.93—1.98; Ni—cp, 1.74—1.81 Å. The three cp rings are inclined to the nickel triangle plane at 68—69°.
N. Kamijyo and T. Watanabe, *Bull. Chem. Soc. Jap.*, 47 (1974) 373.

Triple-decker sandwich complex tris(η-cyclopentadienyl)dinickel tetrafluoroborate, $[Ni_2(C_5H_5)_3]BF_4$

($P2_12_12_1$) $Z = 4$, $R = 6.3\%$ from 1216 independent reflections The structure of the complex cation $[Ni_2(cp)_3]^+$ is essentially a triple-decker sandwich structure. There is some disorder regarding the twisting of the ring plane. Ni—C(terminal) (ave.), 2.09, 2.08; Ni—C(bridging) (ave.), 2.13, 2.16 Å.

E. Dubler, M. Textor, H. Oswald and A. Salzer, *Angew. Chem.*, 13 (1974) 135.

Ethyl chlorophyllide $a.2H_2O$

($P3_1$) or ($P3_2$) $Z = 3$, $R = 12.7\%$ for 781 independent reflections. The aggregation observed in this structure serves as the basis for a model of chlorophyll aggregation in vitro and in vivo. The magnesium atom in ethyl chlorophyllide is displaced 0.4 Å from the plane of the chlorine ring in the same direction as the methyl ester substituent. A water molecule occupies the fifth coordination site.

C.E. Strouse, *Proc. Nat. Acad. Sci.* U.S.A., 71 (1974) 325.

Neptunium tellurides

The structures of the compounds have been identified: $NpTe_3$, $NpTe_{2-x}$, η- and γ-Np_2Te_3. Valence states have been assigned to Np.

D. Damien, *J. Inorg. Nucl. Chem.*, 36 (1974) 307.

Aqueous $[Cr(H_2O)_6]Cl_3$

A $[Cr(H_2O)_6]Cl_3$ aqueous solution, 0.25 M at $T = 20°C$, is investigated by X-ray diffraction. Cr^{3+}—H_2O, 1.90 Å, a distance coincident with the Cr^{3+}—H_2O distance found in crystalline $[Cr(H_2O)_6]Cl_3$. It can reasonably be stated that the $[Cr(H_2O)_6]^{3+}$ ions pass relatively undisturbed into solution where they constitute quite stable units.

A. Cristini, G. Licheri, G. Piccaluga and G. Pinna, *Chem. Phys. Lett.*, 24 (1974) 289.

K_2MnF_4

($I4/mmm$). X-ray and neutron diffraction investigations were carried out on single crystals of K_2MnF_4. Large crystals up to 100 mm^3 were used. The layer-type crystal structure of K_2MnF_4 was found to have significant anisotropy in its mosaic spread.

K. Bittermann and G. Heger, *J. Cryst. Growth*, 21 (1974) 82.

Double lithium orthogermanates — structures of Li_2MgGeO_4

($Pnma$) $R = 9.0\%$ for 160 visually observed reflections. Li, Mg and Ge are tetrahedrally coordinated to oxygen atoms, Ge—O 1.70—1.78, Li—O 1.92—2.01 Å.

B. Monnaye, C. Garrault, G. Perez and R. Bouaziz, *C.R. Acad. Sci.*, 278 (1974) 251.

$Na_2Mg_2Ti_6O_{14}F_2$
(*Pnma*) $R = 10\%$ for 150 visually observed reflection. Magnesium and titanium are octahedrally coordinated.
M. Mayer and G. Perez, *C. R. Acad. Sci.*, 278 (1974) 343.

$Rb_2Cr_3O_{10}$
(*Pbca*) $Z = 8$, $R = 4.2\%$ for 1802 reflections. The structure contains $Cr_3O_{10}^{2-}$ ions composed of three tetrahedra joined by shared corners. Cr—O(bridge), 1.705—1.829; Cr—O(non-bridging), 1.588—1.615 Å. Cr—O—Cr, 136.0, 140.0°. The coordination around the two crystallographically independent rubidium atoms is quite irregular with ten and eleven oxygen atoms in the range 2.91 to 3.39 Å.
P. Löfgren, *Chem. Scripta*, 5 (1974) 91.

α,α-Trehalose—calcium bromide monohydrate, $C_{12}H_{22}O_{11}.CaBr_2.H_2O$
($C222_1$) $Z = 4$, $R = 3\%$ for 925 independent reflections. Each calcium is coordinated to hydroxyl groups from four symmetry-related D-glucose moieties, thereby crosslinking the trehalose molecules. Each calcium has seven oxygens coordinated to it in a pentagonal-bipyramidal arrangement, Ca—O 2.323—2.474 Å.
W.J. Cook and C.E. Bugg, *Carbohydrate Res.*, 31 (1974) 265.

M_2SnCl_6 salts
The effects of univalent cations, M = K^+, NH_4^+, Rb^+, Cs^+ and $(CH_3)_4N^+$, on divalent anions $M'Cl_6^{2-}$, where M' = Sn, Pt, Re, Te and Pb, in $M_2M'Cl_6$ salts having space group ($Fm3m$) have been analyzed. Single-crystal X-ray data on the $SnCl_6^{2-}$ salts have shown Sn—Cl bond lengths to be 2.411(2) (K^+); 2.421(1) (NH_4^+); 2.423(4) (Rb^+); 2.423(5) (Cs^+), and 2.402(3) ($(CH_3)_4N^+$).
T.B. Brill, R.C. Gearhart and W.A. Welsh, *J. Magn. Resonance*, 13 (1974) 27.