Coordination Chemistry Reviews, 14 (1974) 107—114

X-RAY BIBLIOGRAPHY

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(Received February 28th, 1974)

$NaMnCl_3$

($R\bar{3}$) $Z = 6$, $R = 0.0329$ from 694 non-equivalent reflections. The structure can be regarded as trigonally distorted hexagonal close packed array of chloride ions. Mn—Na 3.646(2), Mn—Mn 3.815(2) and Mn—Cl 2.520, 2.585 (1) Å.

C.J.J. van Loon and G.C. Verschoor, *Acta Crystallogr., Sect. B*, 29 (1973) 1224.

Synthetic schmitterite, $UTeO_5$

($Pca2_1$) $Z = 4$, $R = 0.058$ from 641 reflections. The structure is built up of infinite chains $[UO_5]_n^{4n-}$ of pentagonal bipyramids sharing edges and running parallel to the c axis. The tellurium (+IV) atoms, coordinated on one side to four oxygen atoms, maintain the rigidity of the network.

G. Meunier and J. Galy, *Acta Crystallogr., Sect. B*, 29 (1973) 1251.

Diiodobis(triphenylphosphine)palladium(II), $[(C_6H_5)_3P]_2PdI_2.2CH_2Cl_2$

($P2_1/c$) $Z = 2$, $R_w = 0.081$ from 2716 observed reflections. The palladium atom is on an inversion centre of an octahedron formed by the atoms of iodine, phosphorus and the hydrogen on a β carbon of a phenyl ring. Bond lengths Pd—I 2.587, Pd—P 2.331 Å and angle I—Pd—P 87.1° suggest a nearly quadratic configuration.

T. Debaerdemaeker, A. Kutsglu, G. Schmid and L. Weber, *Acta Crystallogr., Sect. B*, (1973) 1283.

Bis(dimethylformamido)bis—(1,3-diphenyl-1,3-propanedionato)magnesium, $(DMF)_2(DPP)_2Mg$, $(MgC_{36}H_{36}N_2O_6)$.

($C2/c$) $Z = 4$, $R = 0.067$ for 1817 independent reflections. Each magnesium ion is octahedrally coordinated to the oxygen atoms of two DMF and two diketone molecules, with the DMF molecules cis to each other. Mg—O distances are 2.055(2) and 2.057(3) (DPP ligand) and 2.095(3)Å to DMF. O—Mg—O angles 86.3—95.5 and 174.0—175.4°.

F.J. Hollander, D.H. Templeton and A. Zalkin, *Acta Crystallogr., Sect. B*, 29 (1973) 1289.

Bis-(1,3-diphenyl-1,3-propanedionato)calcium hemiethanolate, $(Ca[(C_6H_5CO)_2CH]_2(C_2H_5OH)_{\frac{1}{2}})$

($P\bar{1}$) $Z = 4$, $R = 0.040$ for 4503 reflections. The complex consists of a centrosymmetric cluster containing four calcium atoms, two with six oxygen neighbors each and two with seven. Ca—O distances average 2.37 Å.

F.J. Hollander, D.H. Templeton and A. Zalkin, *Acta Crystallogr., Sect. B*, (1973) 1295.

Bis-(1,3)-diphenyl-1,3-propanedionato)strontium hemiacetonate, $Sr_2C_{63}H_{50}O_9$

($P\bar{1}$) $Z = 2$, $R = 0.088$ for 1561 reflections. The topology of the complex is similar to that of the corresponding $Ca(DPP)_2$ hemiethanolate. Distances form Sr—O (bridging oxygen) average 2.55 Å, Sr—O (unshared oxygen) average 2.44 and Sr—O (acetone) 2.60 Å.

F.J. Hollander, D.H. Templeton and A. Zalkin, *Acta Crystallogr., Sect. B*, 29 (1973) 1303.

Cesium trichloromanganate, $CsMnCl_3$

($R\bar{3}m$) $Z = 9$, $R_w = 0.094$ from 481 observed data. There are three crystallographically unique Mn—Cl distances, 2.514(3), 2.545(9) and 2.557(14) Å in the facial-bridged $[MnCl_6]^{4-}$ octahedral trimers.

T. Li, G.D. Stucky and G.L. McPherson, *Acta Crystallogr., Sect. B*, 29 (1973) 1330.

CaV_4O_9

($P4/n$) $R = 0.038$. The structure contains VO_5 square pyramids sharing edges and forming sheets of $[V_4O_9]_n^{2n-}$.

J. Bouloux and J. Galy, *Acta Crystallogr. Sect. B*, 29 (1973) 1335.

$Cu_5V_2O_{10}$

($P2_1/c$) $Z = 4$, $R_w = 0.032$ from 1629 reflections. The structure consists of a network made up of a double chains of $Cu—O_6$ octahedra running parallel to b and chains formed from $Cu—O_6$ octahedra and $Cu—O_5$ trigonal bipyramids running parallel to c. These chains are linked to each other by edge sharing of the octahedra and trigonal bipyramids and corner sharing of the $V(2)O_4$ tetrahedra. Discussion of metal—oxygen distances.

R.D. Shannon and C. Calvo, *Acta Crystallogr., Sect. B*, 29 (1973) 1338.

[(Pyridine-2,6-dicarboxylato) (pyridine-2,6-dicarboxylic acid)] copper (II) hydrate

(Pc) $Z = 4$, $R = 0.10$ from 1983 observed reflections. The copper atom is octahedrally surrounded, being bonded to the nitrogen and two oxygen

atoms of each tridentate ligand. The two ligands coordinate in a different manner, one acting as anionic dipicolinate DP^{2-} and the other as neutral dipicolinic acid H_2DP.

C. Sarchet and H. Loiseleur, *Acta Crystallogr., Sect. B*, 29 (1973) 1345.

Lithium hexafluorotitanate dihydrate and lithium hexafluorostannate dihydrate, $Li_2TiF_6.2H_2O$ and $Li_2SnF_6.2H_2O$

Both ($C2/m$) $Z = 2$, $R = 0.10$ for 463 intensities (Ti) and $R = 0.078$ for 336 intensities (Sn). Both structures contain Li and M in distorted octahedral structures. (Mean Ti—F 1.94, Li—F 2.18 and Li—O 2.05 Å in the first structure and mean Sn—F 1.97, Li—F 2.08 and Li—O 2.06 Å in the second structure.)

E.A. Marseglia and I.D. Brown, *Acta Crystallogr., Sect. B*, 29 (1973) 1352.

$LaFe(CN)_6.5H_2O$

($P6_3/m$) $Z = 2$, $R = 0.030$ from 557 unique reflections. Cyanide bridges link octahedral FeC_6 groups to nine-coordinated $LaN_6(H_2O)_3$ groups. Two uncoordinated water molecules occupy holes in the structure. Bond lengths are Fe—C 1.931, La—N 2.613, La—O 2.585, C≡N 1.55 Å.

W.E. Bailey, R.J. Williams and W.O. Milligan, *Acta Crystallogr., Sect. B*, 29 (1973) 1365.

Potassium chloroplatinate, K_2PtCl_6

($Fm3m$) $Z = 4$, $R_w = 0.0702$ from 75 reflections. Distances are Pt—Cl 2.323(1), Cl—Cl 3.285(2) (in the same octahedron) 3.610(2) (in adjacent octahedra) and K—Cl 3.449(1) Å. Single crystal data compared to the powder method.

R.J. Williams, D.R. Dillin and W.O. Milligan, *Acta Crystallogr., Sect. B*, 29 (1973) 1369.

$C_{18}H_{36}N_2O_6.CaBr_2.3H_2O$

($P2_12_12_1$) $Z = 4$, $R = 0.050$ for 1718 reflections. The Ca^{2+} ion is bonded to the six oxygen and two nitrogen atoms of a macrobicyclic ligand and one water molecule which form an approximate tricapped trigonal prism. Ca—Br distances are greater than 5 Å.

B. Metz, D. Moras and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 1377.

$C_{18}H_{36}N_2O_6.Ba(SCN)_2.H_2O$

($Pbca$) $Z = 16$, $R = 0.080$ for 5835 intensities. The barium atom located in the cavity of the ligand, bonded to the eight hetero-atoms, the water molecule and through the nitrogen atom to one thiocyanate anion.

B. Metz, D. Moras and R. Weiss, *Acta Crystallogr., Sect. B*, (1973) 1382.

$C_{20}H_{40}N_2O_7.Ba(SCN)_2.2H_2O$

($P2_1/c$) $Z = 4$, $R = 0.034$ from 2923 data. The barium ion is undecacoordinated (within the molecular cavity of the ligand) to the nine heteroatoms of the ligand and two water molecules. Ba—O distances range from 2.79—3.09 Å and Ba—N from 3.08—3.18 Å. The thiocyanate ions are at least 5.15 Å from the Ba^{2+} ion.

B. Metz, D. Moras and R. Weiss, *Acta Crystallogr. Sect. B*, 29 (1973) 1388.

Magnesium diethyl phosphate, $Mg[PO_2(OC_2H_5)_2]_2$

($C2/c$) $Z = 4$, $R = 0.061$ for 732 observed reflections.

Magnesium ions are coordinated in a nearly regular tetrahedral arrangement to four oxygen atoms with a mean O—Mg—O angle of 109.14° and a mean Mg—O distance of 1.091 Å.

F.S. Ezra and R.L. Collin, *Acta Crystallogr., Sect. B*, 29 (1973) 1398.

Positions of protons around Mn^{2+} in $La_2(Mg, Mn)_3(NO_3)_{12}.24H_2O$

The coordinates of protons in the complex $[Mn(H_2O)_6]^{2+}$ in $La_2Mg_3(NO_3)_{12}.24H_2O$ doped with a small amount of Mn^{2+} have been measured by means of electron nuclear double resonance (ENDOR) at T = 20K.

R. DeBeer, W. DeBoer, C.A. Van 't Hof and D. VanOrmont, *Acta Crystallogr., Sect. B*, 29 (1973) 1473.

$BaSnS_2$

($P2_1/c$) $R = 0.0574$ for 1049 intensities. The structure is a distortion of the NaCl structure and can be considered as a composite of the BaS and SnS structures.

J.E. Iglesias and H. Steinfink, *Acta Crystallogr., Sect. B*, 29 (1973) 1480.

Bis-(2,4-dithiobiureto)nickel(II) diperchlorate-ethanol, $Ni(HDTB)_2(ClO_4)_2.EtOH$

($P2_1/c$) $Z = 4$, $R = 0.075$ from 1363 reflections. The nickel atom is planar-coordinated to four sulphur atoms, with an average bond distance of Ni—S, 2.162 Å.

A. Pignedoli, G. Peyronel and L. Antolini, *Acta Crystallogr., Sect. B*, 29 (1973) 1490.

$C_2F_4Fe(CO)_4$

Structure determined by gas-phase electron diffraction. Distorted octahedral complex of iron. Fe—C (C_2F_2) 1.989(10), Fe—$C(O)_{eq}$ 1.846(10), Fe—$C(O)_{ax}$ 1.823(10) Å and the Fe—C—F angle is 11[illegible] 3°.

B. Beagley, D.G. Schmidling and D.W.J. Cruickshank, *Acta Crystallogr., Sect. B*, 29 (1973) 1499.

(—)-2,3-ferroceno-5-*exo*-methylcyclohex-2-en-1-one
($P2_12_12_1$) $Z = 4$, $R = 0.063$ from 615 non-zero reflections. Iron-carbon distances range from 2.041 to 2,064 Å.
C. Lecomte, Y. Dusausoy and J. Protas, *Acta Crystallogr., Sect. B*, 29 (1973) 1504.

Tricesium octabromodimolybdate, $Cs_3Mo_2Br_8$
($P\bar{6}2c$ or $P6_3/mmc$) $Z = 2$, The structure consists of Cs^+ cations and $Mo_2Br_8^{3-}$ anions (M_2X_9 confacial bioctahedron with crystallographically disordered vacancies in the bridging positions). Mo—Mo = 2.439(7); Mo—Br (terminal) = 2.554(3); Mo—Br (bridge) = 2.672(5) Å.
F.A. Cotton, B.A. Frenz and Z.C. Mester, *Acta Crystallogr., Sect. B*, 29 (1973) 1515.

Cesium chromium tribromide, $CsCrBr_3$
($P6_3mc$) $Z = 2$, $R_w = 0.054$ from 164 reflections. Isostructural with $CsCrCl_3$, and no evidence of a static Jahn—Teller distortion. Cr—Br, 2.574(12) and 2,780(13) Å.
T. Li and G.D. Stucky, *Acta Crystallogr., Sect. B*, 29 (1973) 1529.

NdSBr
($P2_1/b$) $Z = 4$, $R = 0.062$ from 344 reflections. The structure consists of bromine atoms alternating with layers of Nd_4S tetrahedra.
N. Savigny, C. Adolphe, A. Zalkin and D.H. Templeton, *Acta Crystallogr., Sect. B*, 29 (1973) 1532.

Potassium heptabromodialuminate, KAP_2Br_7
($P2_1/c$) $Z = 4$, $R_w = 0.18$ from 2053 reflections. The Al_2Br_7 anion consists of two $AlBr_4$ tetrahedra sharing one corner with a bent Al—Br—Al bridge (109.3°). The potassium ion is surrounded by nine bromine ions (3.3—4.0 Å).
E. Rytter, B.E.D. Rytter, H.A. Øye and J. Krogh-Moe, *Acta Crystallogr., Sect. B*, 29 (1973) 1541.

Di-π-allyl(dihydropentalelenylene)dinickel, $(C_8H_6)Ni_2(C_3H_5)_2$
($P2_1/a$) $Z = 2$, $R = 0.121$ for 1151 non-zero reflections. Distances of nickel to the ring carbons range from 2.03 to 2.27 Å. The orientation of the π-allyl groups is similar to those found in other π-allyl complexes.
Y. Kitano, M. Kashiwagi and Y. Kinoshita, *Bull. Chem. Soc. Jap.*, 46 (1973) 723.

Redetermination of tin(II) chloride dihydrate, $SnCl_2.2H_2O$
($P2_1/c$) $Z = 4$, $R = 0.04$ from 1695 reflections. The tin atom forms a pyra-

midal complex, Sn—O, 2.33 and Sn—Cl, 2.50 and 2.56 Å.
H. Kiriyama, K. Kitahama, O. Nakamura and R. Kiriyama, *Bull. Chem. Soc. Jap.*, 46 (1973) 1389.

Potassium salt of chloranil
($P2_1 2_1 2_1$) $Z = 4$, $R = 0.13$ for 146 reflections. Chloranil anions are stacked with equal intervals at 3.47 Å.
M. Konno, H. Kobayashi, F. Marumo and Y. Saito, *Bull. Chem. Soc. Jap.*, 46 (1973) 1987.

μ-Peroxo-bis[nitrobis(ethylenediamine)-cobalt(III)] dinitrate tetrahydrate
$[NO_2(en)_2Co—O_2—Co(en)_2NO_2](NO_3)_2.4H_2O$
($P2_1/n$) $Z = 2$, $R = 0.106$ from 1994 non-zero reflections. The coordinating ligands form nearly regular octahedra about the cobalt atoms, with Co—N(en), Co—N(NO_2) and Co—O distances of 1.95, 1.94 and 1.89 Å respectively. Both NO_2^- groups are *trans* to the O—O bridge, the Co—O—O—Co is trans planar with O—O, 1.53 Å and Co—O—O, 110°.
T. Shibahara, S. Koda and M. Mori, *Bull. Chem. Soc. Jap.*, 46 (1973) 2070.

$CuSeO_3.2H_2O$
(D_4^2-$P2_1 2_1 2_1$) $Z = 4$, $R = 0.045$ for 1347 independent reflections. The copper atom is in a square pyramid of oxygen atoms, with the copper 0.137 Å above the basal plane. Average distance of three basal Cu—O (SeO_3) bonds is 1.952 Å, with Cu—O (apical) distance of 2.323 Å. A selenite anion forms a slightly distorted trigonal pyramid with an average Se—O distance of 1.70 Å.
T. Asai and R. Kiriyama, *Bull. Chem. Soc. Jap.*, 46 (1973) 2395.

$(-)_{589}$-Acetylacetonatobis(trimethylenediamine)cobalt(III) arsenic(V) (+)-tartrate monohydrate, $(-)_{589}$-[Co acac tn_2][As-(+)-tart]$_2.H_2O$
($P2_1$) $Z = 2$, $R = 0.104$ for 1707 reflections. The absolute configuration of the cation can be denoted as Δ. The two Co—tn chelate rings assume the chair form, and the Co-acac ring is almost planar.
K. Matsumoto, H. Kawaguchi, H. Kuroya and S. Kawaguchi, *Bull. Chem. Soc. Jap.*, 46 (1973) 2424.

β-Cyanoethyl(D(−)-*erythro*1,2-diphenyl-2-hydroxyethylamine)bis(dimethylglyoximato)cobalt, $C_{25}H_{33}N_6O_5—Co$
($P2_1$) $Z = 4$, $R = 0.14$ for 2127 reflections. The Co(DMG)$_2$ groups are planar except for the methyl groups. The average Co—N distance and N—Co—N angle are 1.89 Å and 82° respectively. The axial Co—N distance is 2.08 Å and the Co—C distance is 2.04 Å.

Y. Ohashi, Y. Sasada, Y. Tashiro, Y. Ohgo, S. Takeuchi and J. Yoshimura, *Bull. Chem. Soc. Jap.*, 46 (1973) 2589.

Laevorotatory cobalt-doped, α-$Zn(H_2O)_6SeO_4$
($P4_12_12$ or $P4_32_12$) $Z = 4$, $R = 0.092$ from 436 reflections. Isomorphous with α-$Ni(D_2O)_6SO_4$. Average Zn—O, 2.12, Se—O 1.66 Å.
K.D. Gailey, H.F. Giles, Jr. and R.A. Palmer, *Chem.Phys.Lett.*, 19 (1973) 561.

Adenine-glycylglycine-copper(II)
($P\bar{1}$) $Z = 2$, $R = 0.13$ for 799 reflections. Square-pyramid structure formed from amino and amide nitrogen and carboxyl oxygen of the dipeptide, N of adenine and one water molecule. Cu—N (adenine) 2.04, Cu—N (amino) 2.03, Cu—N(amide) 1.91, Cu—O (carboxylate) 2.03 and Cu—O (H_2O) 2.28 Å. The chelating atoms of glycylglycine and adenine form the base.
K. Tomita, T. Izuno and T. Fujiwara, *Biochem. Biophys. Res. Commun.* 54 (1973) 96.

Nickel iodate dihydrate, $Ni(IO_3)_2.2H_2O$
(*Pbca*) $Z = 4$, $R = 0.047$ from 8883 reflections. Structure consists of a network of corner sharing IO_6 octahedra with Ni ions occupying octahedral interstices. Ni—O, 2.042—2.062 Å and O—Ni—O, 88.3—89.9°.
S.C. Abrahams, J.L. Bernstein, J.B.A.A. Elemans and G.C. Verschoor, *J. Chem. Phys.* 59 (1973) 2007.

Tris (2,2-dimethylhydrazino)borane, $B[NHN(CH_3)_2]_3$
($P6_3/m$) $Z = 2$, $R = 0.127$ from 162 reflections. The molecule is planar with respect to the BN_6 partial structure. B—N, 1.420(14) Å.
H. Nöth, R. Ullman and H. Vahrenkamp, *Chem. Ber.*, 106 (1973) 1165.

Tetrakis(π-cyclopentadienyl)-di-μ-silyleno-dititanium, $[(C_5H_5)_2TiSiH_2]_2$
($P4_2/mnm$) $Z = 2$, $R = 0.09$ from 243 reflections. The molecule consists of two $(C_5H_5)_2Ti$ units linked by two SiH_2 bridges. The bond angle Ti—Si—Ti is 102.8°
G. Hencken and E. Weiss, *Chem. Ber.*, 106 (1973) 1747.

Pentacarbonyl (2,4,6-triphenylphosphorine)chromium(0)
($P2_1/c$) $Z = 4$, $R = 0.079$ from 1500 reflections. The pentacarbonyl chromium moiety is coordinated to the phosphorus atom of the phosphorine ring such that the Cr—P bond is inclined by an angle of 8° out of the ring plane. Cr—P, 2.37 Å
H. Vahrenkamp and H. Noth, *Chem. Ber.*, 106 (1973) 2227.

Diiodo[2-(β-methylaminoethyl)pyridine]nickel(II) (A) and dibromo[2-(β-methylaminoethyl)pyridine]nickel(II) (B)
($P2_1/c$) $Z = 4$, $R = 0.096$ for A (1480 reflections) and 0.069 for B (1952 reflections). Coordination around the nickel atoms is described as a distorted tetrahedron. Ni—N = 2.01(2), Ni—I = 2.564(4) Å and Ni—Br = 2.324(3) Å, average distances.
W, Haase, *Chem. Ber.*, 106 (1973) 2468.

Tetracarbonyl-μ-(dimethylarsenido)-(tetracarbonyl(ferrio))manganese, $FeMn(CO)_8As(CH_3)_2$
($Pn2_1a$) $Z = 4$, $R = 0.044$ from 746 reflections. Three of the iron and manganese atoms are indistinguishable. The structure framework is a triangle of iron, manganese and arsenic. The two metal—arsenic bonds are 2.353 Å (ave) and the metal—metal bond is 2.85 Å.
H. Vahrenkamp, *Chem. Ber.*, 106 (1973) 2570.

Halogeno(2-diethylaminoethanolato)copper(II)complexes (Halogen: chlorine or bromine)
($P4_12_12$ or $P4_32_12$) $Z = 16$ and $P2_1/n$), $R = 0.057$ for 1333 reflections, and 0.075 for 1449 reflections, respectively. The tetrameric clusters of the molecules have the cubane type structure with Cu—Cu separations of 2.937(2) Å (mean value), 3.426(2) Å and 3.459(2) Å for the chloro derivative. The bromo derivative is centrosymmetric dimeric with Cu—Cu separation of 3.033(5) Å.
W. Haase, *Chem. Ber.*, 106 (1973) 3132.

3,3-Bis(methoxycarbonyl)-4-phenyl-1-pyrazoline-*N*-(tetracarbonyliron), $C_{17}H_{14}N_2O_8Fe$
($P2_1/c$) $Z = 4$, $R = 0.063$ from 2560 reflections. The 1-pyrazole derivative is linked through a σ-bond to the apical position of a $Fe(CO)_4$ residue. Fe—C, 1.773 to 1.822 Å, Fe—N, 1979 Å.
C. Kruger, *Chem. Ber.*, 106 (1973) 3230.