Coordination Chemistry Reviews
Elsevier Publishing Company, Amsterdam
Printed in The Netherlands

Editorial note

We begin in this issue an X-ray bibliography of structures of interest to inorganic chemists. Each issue of Coordination Chemistry Reviews will contain a continuing, up-to-date bibliography as the structures appear in the literature. Each entry contains the name of the compound, the reference and a brief description of the structure, together with pertinent crystallographic details such as the space group and the number of reflections.

It is hoped that this service will alert readers to new structures very soon after they appear in the literature. With the availability of advanced equipment which will complete a structure in a relatively short period, it is very difficult to keep up with the mass of structural information. We hope that this service will enable our readers to keep abreast of the developments in this area. At the end of each volume the bibliography will be indexed according to central element and ligand.

X-RAY BIBLIOGRAPHY

Compiled by M. KEETON

Department of Chemistry, McMaster University, Hamilton, Ontario (Canada)

Au_6Hg_5
Diffractometer data on regular hexagonal prism with pyramids on both ends. 122 independent reflexions. A number of models were tried. The best converged to $R = 7.6\%$. Space group $P6_3/mcm$. Structure is 10- and 12-coordination polyhedron around Hg.
T. Lindahl, *Acta Chem. Scand.*, 24 (1970) 946.

1,4-Diselenocyanotobenzene
$C_6H_4(SeCN)_2$, $R = 11.4\%$, $P2_1/c$. The C–Se–C bond angle is 94.4° and S ··· N contacts are of 3.08 and 3.27 Å making an approximate square plane around each selenium atom.
W.S. McDonald and L.P. Pettit, *J. Chem. Soc., (A)*, (1970) 2044.

Tetraphenylphosphenium π-cyclopentadienylbis-(1,2-dicyanoethylene-1,2-dithiolato)-molybdenum, $[Ph_4P^+][(\pi\text{-}C_5H_5)Mo\{S_2C_2(CN)_2\}_2^-]$
($P\bar{1}$), $R = 8.6$ for 3657 independent reflexions taken on a Berger diffractometer. The anion has approximate C_s symmetry with the molybdenum atom approximately seven-coordinate to two bidentate dithiolates and a tridentate π cyclopentadienyl ligand; Mo–S distances average 2.407 Å.
M.R. Churchill and J. Cooke, *J. Chem. Soc., (A)*, (1970) 2046.

Potassium pentafluorotellurate, $KTeF_5$
($Pbcm$) $R = 2.18\%$ for 646 observed reflexions. The structure contains isolated TeF_5^- ions arranged in approximate square pyramidal fashion but with strict C_s symmetry. Te–F distances are apical 1.864, basal 1.953, and 1.952 Å, respectively.
S.H. Martin, R.R. Ryan and L.B. Aspray, *Inorg. Chem.*, 9 (1970) 2100.

Erbium oxalate trihydrate, $Er(C_2O_4)(HC_2O_4) \cdot 3H_2O$

(P_4/n) $R = 7.2\%$ for 1641 independent reflexions. Eight oxygen atoms coordinate to a central erbium with distances ranging from 2.362 to 2.418 Å, and form a distorted square antiprism. A water molecule occupies a ninth position 2.441 Å above the larger face of the antiprism and oxalate anions are distributed randomly in the crystal. Hydrogen bonding occurs between the water molecule and the oxygen of the acid groups.

H. Steinfink and G.D. Brunton, *Inorg. Chem.*, 9 (1970) 2112.

$UO_2[(C_2H_5)_2NCS]_2(C_6H_5)_3XO$, where X is As or P

The crystals of the two compounds are isomorphous and monoclinic $R = 9.4$ and 9.3 respectively for 2802 and 2801 observed reflexions. The uranium is in a seven-coordinate pentagonal bipyramidal environment, the sulphur atoms are in the same plane. Space group (both compounds) $P2_1/n$.

R. Graziani, B. Zarli, A. Cassol, G. Bombieri, E. Forsellini and E. Tondello, *Inorg. Chem.*, 9 (1970) 2116.

μ-Chloro-μ-amido-octaamminedicobalt tetrachloride tetrahydrate,

$[(NH_3)_4Co \cdot Cl \cdot NH_2 \cdot Co(NH_3)_4] \cdot Cl_4 \cdot 4H_2O$

$R = 4.0\%$ for 1018 observed reflexions. The cobalt atoms are in octahedral environments sharing an edge with Co–Co 3.2 Å. The bridging nitrogen and chlorine atoms are disordered with the cation lying in the centre of symmetry. The structure is the same as for the hydroxide bridge compound previously reported.

R. Barro, R.E. Marsh and W.P. Schaefer, *Inorg. Chem.*, 9 (1970) 2131.

Tetrafluorocyclobutenebis(dimethylarsine)triiron decacarbonyl,

$(CH_3)_2As\overline{C{=}C(As(CH_3)_2CF_2C}F_2 \cdot Fe_3(CO)_{10}$

$(P2_1/c)$ $R = 9\%$ for 2524 observed reflections. The complex is structurally a derivative of $Fe_3(CO)_{12}$ with one CO on each of the two equivalent iron atoms replaced by an arsenic atom of the diarsine ligand. The Fe–Fe distances closely match those in the structure of the parent compound.

P.J. Roberts, B.R. Penfold and J. Trotter, *Inorg. Chem.*, 9 (1970) 2137.

Bis[2,2′-iminobis(acetamidoxime)] nickel(II) chloride dihydrate and bis[2,2′-iminobis-(acetamidoxime)] copper(II) chloride

The new ligand (IBO) is potentially tridentate. For the nickel compound $(P2_1/a)$ $R = 3.2\%$ for 2023 observed reflections. The cation $Ni(IBO)_2^{2+}$ is a centro symmetric, slightly distorted octahedron with bonding to central amine nitrogen and to two oxime nitrogens. The copper complex, $(P2_1/a)$ $R = 4.0\%$ for 3500 observed reflections contain $Cu(IBO)_2^{2+}$ cations with the copper in a distorted square pyramidal penta-coordination. One ligand is tridentate, the other is bidentate with the oxime nitrogen occupying the apical position and the other oxime nitrogen free. The Cu–N (oxime) distances are 1.951, 1.960 and 1.976 Å. The Cu–N amine distances are 2.014 Å (equatorial) and 2.357 Å (apical).

D.L. Cullen and E.C. Lingafelter, *Inorg. Chem.*, 9 (1970) 1865.

Tetrakis(2-aminoethanethiol)trinickel(II) chloride, $[Ni\{Ni(NH_2CH_2CH_2S)_2\}_2]Cl_2$
($P\bar{1}$) $R = 4.9\%$ for 1213 independent reflections. Two $Ni(NH_2CH_2CH_2S)_2$ entities are chelated in *cis* fashion to the third nickel atom via sulphur in a square planar array. The Ni–Ni distances are 2.733 Å in the symmetrical array of linear nickel atoms.
Chin Asuar Wei and L.F. Dahl, *Inorg. Chem.*, 9 (1970) 1878.

Bis[hydratris(1-pyrazolyl)borato]cobalt(II), $[HB(C_3N_2H_3)_3]Co^{II}$
($P2_1/a$) $R_F = 7.28\%$ for 2340 independent reflections. The cobalt ion is in a trigonally distorted octahedral environment, coordination is to six nitrogen atoms with the molecule possessing approximate D_{3d} symmetry.
M.R. Churchill and C.E. Maw, Jr., *Inorg. Chem.*, 9 (1970) 1597.

μ-Amido-μ-nitro-bis[tetraamine cobalt(III)] tetrachloride tetrahydrate,
$[(NH_3)_4Co(NH_2)(NO_2)Co(NH_3)_4]Cl_4 \cdot 4H_2O$
($P2_1/m$) $R = 4.8\%$ for 1102 independent reflections. The cation contains the planar group $N_2Co \cdot N \cdot NO_2 \cdot CoN_2$. The unsymmetrical arrangement of the NO_2 bridge between Co atoms is confirmed as predicted from earlier IR studies. The structure is bound together by a network of hydrogen bonds among the water molecules, the cations and anions.
U. Thewalt and R.E. Marsh, *Inorg. Chem.*, 9 (1970) 1604.

μ_4-Oxo-hexa-μ-chloro-tetrakis[(2-methylpyridine)copper(II)] hydrate,
$Cu_4OCl_6(2\text{-mepy})_4 \cdot xH_2O$
($P2_1/c$) $R = 8.2\%$ for 1824 reflections (Weissenberg). In the structure of the tetrameric unit the copper atoms are arranged in tetrahedral array around a central oxygen atom. Each copper is in an environment intermediate between a trigonal bipyramid and a tetragonal pyramid.
N.S. Gill and M. Sterns, *Inorg. Chem.*, 9 (1970) 1619.

$Ir[C_3(C_6H_5)_3]Cl(CO)[P(CH_3)_3]_2[BF_4] \cdot CH_2Cl_2$
The complex is formed as intense purple crystals when triphenylcyclopropenium tetrafluoroborate is added to a methanolic solution of *trans*-$IrCl(CO)[P(CH_3)_3]_2$ ($P2_12_12_1$) $R = 6.0\%$ for 3257 reflections. The cyclopropenium ring is opened and occupies two coordination sites of the iridium(III) ion which is in a distorted octahedral environment. The authors claim the complex to be the first example of the oxidative addition of a metal ion across a carbon–carbon bond.
R.M. Tuffle and D.L. Weaver, *J. Amer. Chem. Soc.*, 92 (1970) 5524.

Cesium *trans*-tetrachloroaquocarbonylruthenate(II), $Cs_2[Ru(CO)(H_2O)Cl_4]$
($Amam$) $R = 5.0\%$ for 712 reflections. The ruthenium atom is octahedrally coordinated with four chloride ligands in an elongated plane and the water and carbon monoxide above and below the plane in a *trans* arrangement.
Ru–C, 1.804; Ru–Cl, 2.395; C–O, 1.091.
J.A. Stanko and Surang Chaipayungpundhu, *J. Amer. Chem. Soc.*, 92 (1970) 5580.

Copper(II) formate monourea, $(HCO_2)_2Cu \cdot (NH_2)_2CO$

($P\bar{1}$) R = 8.9% for 616 independent reflections. The structure is made up of $[Cu(O_2CH)_2OC(NH_2)_2]_2$ units with four bidentate formate anions forming *syn–syn* bridges between isolated pairs of copper atoms separated by 2.657 Å. This structure is closely related to several other carboxylate adducts but no apparent structural tendencies can be correlated with the magnetic data which exist. An *O*-bonded model versus an *N*-bonded urea seems to be the more likely structure.

P.B.W. Yawney and R.J. Doedens, *Inorg. Chem.*, 9 (1970) 1626.

The acetylacetoxime adduct of ytterbium acetylacetonate, $Yb(C_5H_7O_2)_3(CH_3COCH{=}C(NH_2)CH_3)$

R = 7.3% for 1301 independent reflections ($P2_1/c$). Each ytterbium is bonded to seven oxygens, six from the acac ions and one from the acetylacetoximine group. The shortest Yb–N distance (intramolecular) is 4.27 Å. The Yb–O bonds are longer when the oxygen is involved in hydrogen bonding to an NH_2 group.

M.R. Richardson, P.W.R. Corfield, D.E. Sands and R.E. Sievers, *Inorg. Chem.*, 9 (1970) 1632.

Dipyridinium oxytetrachloroselenate(IV), $C_{10}H_8N_2H_2{}^{2+}SeOCl_4{}^{2-}$

($C2/c$) 2731 observed reflections (Weissenberg) with R = 9.8%. The structure consists of a chainlike arrangement of Cl–N–N–Cl hydrogen-bond linkages with square pyramidal $SeOCl_4{}^-$ units joined to this chain by a Se–Cl interaction. Each chlorine is hydrogen bonded to two bipyridyl molecules. The dipyridine exists as dipyridinium ions as evidenced by the large C–N–C angles.

Bi Cheng Wang and A.W. Cordes, *Inorg. Chem.*, 9 (1970) 1643.

Sorbic acid iron tricarbonyl, $(C_6H_8O_2)Fe(CO_3)$

($P\bar{1}$) R = 4.7 for 2086 unique reflections. All hydrogen atoms were located and the carbon skeleton of the butadiene fragment are coplanar. The unit cell contains two crystallographically unique dimers held together by hydrogen bonds between the hydrogen, from a carbonyl group, of one sorbic acid ligand, to the carbonyl oxygen of the second. The coordination within a monomer does not appear to be significantly affected by packing forces and all angles at carbon atoms of the butadiene group are essentially 120°.

R. Eiss, *Inorg. Chem.*, 9 (1970) 1650.

Trimeric phosphonitrilic isothiocyanate, $N_3P_3(NCS)_6$

($P\bar{1}$) R = 12% for 1246 independent reflections (Weissenberg). The nitrogen atoms of two isothiocyanate groups are bonded to each phosphorus atom in the six-membered ring. One atom of the ring, a nitrogen, is 0.15 Å out of the plane of the ring. The average P–N bond distance is 1.58 Å. The average P–N exocyclic bond distance is 1.63 Å.

J.B. Faught, T. Moeller and I.C. Paul, *Inorg. Chem.*, 9 (1970) 1656.

Oxalatobis(ethylenediamine)chromium(III)bis oxalatoethylene diamine chromate(III) dihydrate, $[\mathrm{Cren_2C_2O_4}][\mathrm{Cren(C_2O_4)_2}] \cdot 2\mathrm{H_2O}$

($P2/c$) $R = 9.23\%$ for 2021 independent reflections. Discrete $[\mathrm{Cr(en)_2ox}]^+$ and $[\mathrm{Cr(en)(ox)_2}]^-$ ions are linked by hydrogen bonding to neighboring ions and to water molecules. The confirmation of the ethylene diamines is $\Delta\lambda\lambda$ and $\Delta\delta,\Delta\lambda$ in the anion and cation, respectively.

J.W. Lethbridge, L.S. Dent Glasser and H.F.W. Taylor, *J. Chem. Soc., (A)*, (1970) 1862.

Bis-μ-diphenylphosphido-bis(dicarbonylnickel), $[(\mathrm{OC})_2\mathrm{Ni(PPh_2)_2Ni(CO)_2}]$

($P\bar{1}$) $R = 4.5\%$ for 2068 independent reflections. The two nickel atoms are linked by a planar di-μ-phosphido-bridge (Ni–P 2.19 Å) and a strong Ni–Ni (2.51 Å) bond. The Ni–P–Ni bond is reduced to 70° but all other distances and angles in this nickel(I) complex are similar to those in tetrahedral Ni(0) complexes.

J.A.J. Jarvis, R.H.B. Mais, P.B. Owston and D.T. Thompson, *J. Chem. Soc., (A)*, (1970) 1867.

trans-Dichloro(π-di-tert-butylacetylene)-*p*-toluidineplatinum(II), $[\mathrm{PtCl_2(t\text{-}BuC{\equiv}CBu\text{-}t)(MeC_6H_4NH_2)}]$

($P2_1/m$) $R = 3.8\%$ for 1380 independent reflections. The coordination of the platinum is square planar and the acetylene ligand is coordinated in a π-type bond with the C–C axis tilted 85% out of the coordination plane. Pt–C distances average 2.16 Å.

G.R. Davies, W. Hewertson, R.H.B. Mais, P.A. Owston and C.G. Patel, *J. Chem. Soc. (A)*, (1970) 1873.

Acetato-C-rac-(5,7,7,12,14,14,hexamethyl-1,4,8,11-tetra-azacyclotetradecane)nickel(II) perchlorate

($P2_1/c$) $R = 12.6\%$ for 2753 (Weissenberg) independent reflections. The cyclic tetraamine is folded with four secondary amine groups coordinated to adjacent octahedral sites of the nickel(II) ion. The acetate ion is symmetrically chelated to the remaining two sites. The tetramine is in the C-rac-modification, both pairs of asymmetric nitrogens also have the racemic arrangement but are of opposite helicity.

P.O. Whimp, M.F. Bailey and N.F. Curtis, *J. Chem. Soc. (A)*, (1970) 1956.

$(\pi\text{-C}_5\mathrm{H}_5)\mathrm{Ni}(\pi\text{-C}_3\mathrm{Ph}_3)$

The compound crystallises in deep red-orange prisms in ($Pna2_1$). $R = 6.2\%$. The nickel is complexed in sandwich fashion and resembles the cobalt analog reported previously (*J. Amer. Chem. Soc.*, 89 (1967) 5505).

M.D. Rausch, R.M. Tuggle, D.L. Weaver, *J. Amer. Chem. Soc.*, 92 (1970) 4981.

$[\mathrm{B_9C_2H_{11}\cdot Co\cdot B_8C_2H_{10}\cdot Co\cdot B_8C_2H_{10}\cdot Co\cdot B_9C_2H_{11}}^{3-}]\ [(\mathrm{C_2H_5})_4\mathrm{N}^+]$

This is a fused polyhedral structure; each of three Co(III)d^6 ions are sandwich bonded to two π bidentate $\mathrm{B_8C_2H_{10}}^{4-}$ and two π monodentate $\mathrm{B_9C_2H_{11}}^{2-}$ ligands. Deep red crystals from acetone–water; ($P\bar{1}$) $R = 14.1\%$ for 3994 independent non-zero reflections.

M.R. Churchill, A.H. Reis, Jr., J.N. Francis and M.F. Hawthorne, *J. Amer. Chem. Soc.*, 92 (1970) 4993.

Barium nickel sulphide

(*P*4/*nmm*) *R* = 3.9% for 184 unique reflections. A nickel atom is coordinated to five sulphur atoms at the apices of a slightly distorted square pyramid. Each of these units shares its equitorial edges with an equivalent inverted unit. Barium atoms are located between layers of nickel sulphide units.

I.E. Grey and H. Steinfink, *J. Amer. Chem. Soc.*, 92 (1970) 5093.

$[C_6H_4(OH)(CHNC_3H_6C_3H_6OH)]_2Fe_2Cl_2$

(*P*2$_1$/*c*) *R* = 12% for 418 unique reflections. The coordination of the iron is square pyramidal with three oxygens and nitrogens forming a planar base. The two iron atoms and bridging oxygens are planar.

J.A. Bertrand, J.L. Breece, A.R. Kalyanaraman, G.J. Long and W.A. Baker, Jr., *J. Amer. Chem. Soc.*, 92 (1970) 5233.