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

X-RAY BIBLIOGRAPHY

M. KEETON

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

(Received May 11th, 1971)

Di-μ-methylthio-(bis-π-cyclopentadienyltitanium)tetracarbonyl-molybdenum
(*Pbca*) $R = 5.2\%$ for 1535 significant reflections. The complex contains a four-membered ring system –Ti–S–Mo–S. Ti–Mo is 3.32 Å; Ti–S is 2.46 Å, Mo–S is 2 56 Å; and Mo–S–Ti is 83°.
G.R. Davies and B.T. Kilbourn, *J. Chem. Soc. (A)*, (1971) 87

Triethylplatinum(IV)chloride, tetra-μ_3-chloro tetrakis triethylplatinum(IV), Et_3PtCl_4
($P2_1/n$) $R = 8.2\%$ for 1300 reflections. In the tetrameric unit, the methyl groups are rigidly held by steric repulsions The molecule is found to have approximate $23(T)$ symmetry compared with $43m(Td)$ for Me_3PtCl_4
R.N. Hargreaves and M.R. Truter, *J. Chem. Soc. (A)*, (1971) 90

μ-formato-diethylene-triaminine copper(II) formate, $Cu(dien)(HCO_2)_2$
(*Pnam*), $R = 5.7\%$ for 893 reflections. Discrete $Cu(dien)(HCO_2)^+$ ions run parallel to the *a* axis with copper linked through formate groups in an anti-syn bridge arrangement. The coordination of each copper is essentially square pyramidal. The correct stereochemistry of the copper was previously predicted from an analysis of the electronic spectrum.
G. Davey and F.S. Stevens, *J. Chem. Soc. (A)*, (1971) 103.

Di-isothiocyanato-*N,N*-bis-(2-diethylaminoethyl)-2-diphenylarsinoethyl-amine-*N,N,N*-nickel(II)
($P2_1/c$), $R = 7.3\%$ for 2171 reflections. The nickel atom is surrounded by five nitrogen atoms in approximate square-pyramidal geometry. The arsenic atom does not coordinate. $Ni–N_{(NCS)}$ is 1.95 Å. The central Ni–N is 2.08 and the other two Ni–N, 2 20 Å.
M. Di Vaira, *J. Chem. Soc. (A)*, (1971) 148.

Diaquobis-(ethoxyacetato)copper(II)
($P1$), $R = 11.0\%$ for 999 reflections. The copper ions are in elongated tetragonally distorted environments with the two aquo-ligands perpendicular to the coplanar *trans* chelate rings.
C.K. Prout, J.R. Caruthers and F.J. Rossotti, *J. Chem. Soc. (A)*, (1971) 554.

Diaquobis(methoxyacetato)nickel(II)

($P2_1/n$), R = 11 9% for 589 independent, visually estimated reflections. The complex is isostructural with the copper analogue above Crystal data for the unidentate phenoxyacetato nickel(II) complex are also given.

C K. Prout, Clare Walker and F J.C. Rossotti, *J. Chem Soc (A)*, (1971) 556.

Ni^{II}, Cu^{II} and Zn^{II} diethyldiselenocarbamates

All compounds crystallised in the space group $P2_1/n$, $R_{(Ni)}$ = 7 6% for 928 reflections, $R_{(Cu)}$ = 11.5% for 2123 reflections; and $R_{(Zn)}$ = 7.5% for 1177 reflections Isomorphism with corresponding diethyldithiocarbamates enabled a comparison of M—Se and M—S, and of C—Se and C—S bonds to be made. The nickel coordination is approximately square planar; the copper, tetragonal pyramidal, and the zinc intermediate between tetragonal pyramidal and trigonal bipyramidal.

M. Bonamico and G Dessy, *J. Chem. Soc (A)*, 2 (1971) 264.

Ammonium pentachloroantimonate(III), $(NH_4)_2\ SbCl_5$

($C2/m$), R = 8 1% for 782 reflections The structure consists of ammonium ions and almost square pyramidal $SbCl_5{}^{2-}$ ions

M Webster and S Keats, *J. Chem. Soc. (A)*, 2 (1971) 264.

Bis(triphenylphosphine)tetrakis(dimethylglyoximato)dirhodium, $Rh_2(C_4H_7N_2O_2)_4(C_6H_5)_3P_2 \cdot H_2O \cdot C_3H_7OH$

($P2_1/c$), R = 8 5% for 3307 statistically significant reflections The dinuclear molecule consists of two equivalent halves, $(C_6H_5)_3PRh(C_4H_7N_2O_2)_2$ linked by a Rh—Rh bond of 2 936(2) Å. The P—Rh—Rh—P chain is almost linear with the two $Rh(C_4H_7N_2O_2)_2$ groups in an eclipsed rotational relationship, and perpendicular to the chain axis

K G. Caulton and F A. Cotton, *J Amer Chem. Soc*, 93 (1971) 1914

Bis(nitrotriamminepalladium(II)tetraamminepalladium(II)tetranitrate, $[Pd(NO_2)(NH_3)_3]_2 Pd(NH_3)_4\ (NO_3)_4$

($I\bar{4}2m$), R = 3.7% for 786 unique reflections. Palladium is square-planar coordinated in both ions and the packing of these precludes any metal—metal interactions Raman and infrared data are correlated with the structure.

F.P. Boer, V B. Carter and J.W. Turley, *Inorg Chem.*, 10 (1970) 651

Dichlorobis(1-methyltetrazole)zinc(II), $Zn(C_2N_4H_4)_2Cl_2$

($P2_1/c$), R= 9.7% for 1278 independent reflections The zinc is in a distorted tetrahedral coordination sphere and is almost planar with the tetrazole rings. The rings are coordinated from the 4 position

N C. Baenziger and R.J. Schultz, *Inorg. Chem.*, 10 (1971) 661.

(+)-4-Methylpyridinetrimethylaminebromohydroboron hexafluorophosphate, $(CH_3\cdot C_5H_4N)(CH_3)_3N\cdot Br\cdot H\,B^+(PF_6)^-$

($P2_1$), R = 8.9% for 2481 independent, non-zero reflections. The boron atom is surrounded by an almost regular tetrahedral array of ligands.

G. Allegra, E. Benedetti, C. Pedone and S.L. Holt, *Inorg. Chem.*, 10 (1971) 667.

Tetramethyl-1,3-diisothiocyanatodistannoane, $((CH_3)_2SnNCS)_2O_2$

($C2/m$), R = 8.0% for 546 visually estimated independent reflections. The dimeric molecule has $2/m$ symmetry with a central planar four-membered $Sn^{II}_2O_2$ ring.

Y.M. Chow, *Inorg. Chem.*, 10 (1971) 673.

Vanadium(V) oxide trinitrate–acetonitrile complex, $VO(NO_3)_3\cdot CH_3CN$

($Pbca$), R = 9.5% for 548 reflections observed. An equatorial pentagon of five oxygen atoms is formed by coordination of two bidentate nitrate groups and one unidentate group. The vanadyl oxygen and nitrogen of the acetonitrile group form the apices. Infrared and Raman data are correlated with the structure.

F.W.B. Einstein, E. Enwall, D.M. Morris and D. Sutton, *Inorg. Chem.*, 10 (1971) 678.

A Nickel(II) iodide complex of a macrocyclic tetradentate imine ether, Ni(1,8-diaza-3,4,10,11-dibenzo-5,12-dioxo-1,3,8,10-cyclotetra-decatetraene)I_2

($P2_1/c$) R = 4.7% for 943 statistically significant reflections. The two nitrogen and two oxygen atoms of the macrocyclic ligand define a plane with respect to which the two iodine atoms occupy the two apical octahedral positions at 2.880(3) Å from the Ni. Ni–N and Ni–O are 1.92(1) and 2.07(1) Å, respectively.

D.L. Johnston and W. DeW. Horrocks, Jr., *Inorg. Chem.*, 10 (1971) 687.

Ba_2MnSe_3 and Ba_2MnS_3

($Pnma$) R = 5.8% for 992 reflections in the selenide and R = 5.9% for 233 reflections in the sulphide. The structures consist of $Mn{-}X_4$ tetrahedra (X = S, Se) sharing corners to produce infinite chains parallel to the *b* axis. The magnetism of both compounds is discussed.

I.E. Grey and H. Steinfink, *Inorg. Chem.*, 10 (1971) 691.

Pyridinium tetracosabromoantimon(III)triantimon(V)ate $(C_5H_5NH)_6Sb^{III}Sb^{V}_3Br_{24}$

($Cmcm$) R = 7.4% for 3244 independent reflections. Three crystallographically independent pyridinium cations, one $Sb^{III}Br_6^{3-}$ and two $Sb^{V}Br_6^{-}$ anions form an asymmetric unit. All three anions are almost of octahedral symmetry.

S.L. Lawton, R.A. Jacobson and R.S. Frye, *Inorg. Chem.*, 10 (1971) 701.

Quinuclidinium dodecabromoantimon(III)antimon(V)ate-2-dibromine, $(C_7H_{13}NH)_4Sb^{III}Sb^{V}Br_{12}\cdot 2Br_2$

($Fm3m$)(Oh)^5, R = 6.8% for 198 independent reflections. No ordering of the Sb^{III} and Sb^{V} oxidation states was found. The average anions and threfold disordered bromine molecules form an NaCl-type arrangement.

S.L. Lawton and R.A. Jacobson, *Inorg. Chem.*, 10 (1971) 701.

Bis(dimethyl-O-thiolophenyl-arsine)platinum(II), $PtAs_2S_2C_{16}H_2$

($P2_1/c$) $R = 2.9\%$ for 1555 reflections The molecule is a *trans* isomer in which the thiol functions as a bidentate ligand. The molecule is planar and the Pt–As bond distance is 2.354(1) Å indicative of $d\pi$–$d\pi$ bonding.

J.P. Beale and N.C. Stephenson, *Acta Crystallogr. Ser. B*, 27 (1971) 73.

Dichlorodiaquobis(dicyandiamide)copper(II), $Cu(OH_2)_2(C_2N_4H_4)Cl_2$

($P\bar{1}$) $R = 8.3\%$. The copper is surrounded by a planar arrangement of two water molecules (Cu–O = 2.00 Å) and two nitrile nitrogens (Cu–N = 1.92 Å) with two chlorine atoms in the *trans* positions (Cu–Cl = 2.87 Å).

A. Chiesi, L. Coghi, A. Mangia, M. Nardelli and G. Pelizzi, *Acta Crystallogr. Ser. B*, 27 (1971) 192.

$Cl\text{-}Mn(CO_4)P(C_6H_5)_3$

($P\bar{1}$) $R = 8.3\%$ for 1467 reflections. The manganese is in octahedral coordination, Mn–Cl 2.36 Å, Mn–P 2.40 Å. The *cis* ligand arrangement deduced from the IR spectrum is confirmed.

H. Vahremkamp, *Chem. Ber*, 104 (1971) 449.

trans-Bis(diethylselenide)palladium(II)dichloride, $PdCl_2(Et_2Se)_2$

($P\bar{1}$) $R = 7.96\%$ for 1046 reflections The configuration around the selenium is pyramidal. The palladium atom is in square-planar coordination

P.E. Shakke and S.E. Rasmussen, *Acta Chem. Scand.*, 24 (1970) 2634.

Potassium pentacyanonitrosylvanadate(1), $K_3V(CN)_5NO\ 2H_2O$

($Pccn$) $R = 7\ 8\%$ for 481 unique reflections. The structure contains distorted $V(CN)_5NO^{3-}$ octahedra, V–N (1.66 Å), V–C (2.17 Å).

S. Jugner and N. Vannerberg, *Acta Chem. Scand*, 24 (1970) 1988.

Potassium oxodiperoxooxalatomolybdate(VI), $K_2MoO(O_2)_2(C_2O_4)$

($P2_1/n$) $R = 11.2\%$ for 1173 reflections Molybdenum is surrounded by a pentagonal bipyramidal ligand arrangement. Two peroxo groups and one of the oxygens of the oxalate groups are in the plane while one double-bonded oxygen and the other oxygen of the oxalato group form the apices.

R. Stomberg, *Acta Chem. Scand.*, 24 (1970) 2024.

(Tri-)ammonium fluoride oxoperoxotetrafluoromolybdate(VI), $(NH_4)_3F[MoO(O_2)F_4]$

($Pbca$) $R = 12.9\%$ for 1079 observed reflections. Each molybdenum is surrounded by three oxygen atoms and four fluorine atoms in a pentagonal bipyramidal arrangement, the equatorial plane containing the two peroxidic oxygen atoms and three fluorines A double-bonded oxygen and a fluorine occupy the apical positions.

I. Larking and R. Stomberg, *Acta Chem. Scand.*, 24 (1970) 2043.

Bisethylene-1,2-bis(methylphenylphosphine)palladium(II)chloride,
$Pd(C_6H_5CH_3PCH_2CH_2PCH_3C_6H_5)_2Cl_2 \cdot C_2H_5OH \cdot$

The space group is in square-planar coordination Pd—P is 2.33 Å and P—C (mean) is 1.81. The complex may be described in terms of a MESO-anti-*trans* form.

P. Groth, *Acta Chem. Scand.*, 24 (1970) 2785.

Lithium hydrogen phosphite, LiH_2PO_3

($P2_{1nb}$) R = 6.3% for 285 independent reflections. One hydrogen is bonded to phosphorus, the other is in a hydroxyl group. The lithium atom is tetrahedrally surrounded by oxygen atoms at 1.91, 1.97 and 1.97, 2.00 Å.

E. Philippot and O. Lindgrist, *Acta Chem. Scand.*, 24 (1970) 2803.

Cis-α-nickel(II)triethylenetetramine isothiocyanate, α-Ni(trien)$(NCS)_2$

($C2/c$) R = 4.3% for 1553 reflections. The complex is approximately octahedral and has a 2-fold axis coincident with the crystallographic 2-fold axis. The (trien) is bonded in an almost strain-free arrangement and the *cis* thiocyanates are bonded via nitrogen.

A. Clausen and A.C. Hazell, *Acta Chem. Scand.*, 24 (1970) 2811.