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X-RAY BIBLIOGRAPHY

H. CLEARY STYNES

York University, Downsview, Ontario, M3J 1P3 (Canada)

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Tri-μ-carbonyl-bis-(1,2-diphenyl-3,4-di-*t*-butylcyclobutadiene)di-iron ($I\bar{4}$) $Z = 4$, $R = 6.1\%$ for 1484 non-zero reflections. The dimeric molecule is located on a crystallographic two-fold axis passing through the centre of the Fe—Fe bond and a CO group. The most remarkable feature is the extremely short Fe—Fe bond, 2.177(3) Å, a triple bond. The two iron atoms are bridged by three CO groups, Fe—CO(avg) = 1.974 Å, the cyclobutadiene ring is essentially square planar.
S. Murahashi, T. Mizoguchi, T. Hosokawa, I. Moritani, Y. Kai, M. Kohara, N. Yasuoka and N. Kasai, J. Chem. Soc. Chem. Commun., (1974) 563.

Chloro-[4-(chloro-*t*-butyl)methylene-2,5-di-*t*-butyl-1-3-η-cyclopent-2-enyl]-triphenylphosphinepalladium, $[Pd\{Cl(Me_3CC_2H)_3\}Cl(PPh_3)]$, $MeCO_2H$ ($P2_1/c$) $Z = 4$, $R = 6.7\%$ for 3056 independent reflections. The organic ligand consists of a ((chloro-*t*-butylmethylene)cyclopent-2-enyl) unit which is approximately planar and the three endocyclic carbon atoms are somewhat asymmetrically bonded to the metal. The acetic acid of crystallization is hydrogen bonded to the Cl bound to Pd. The complex exists in two η^3-allylic forms, an exocyclic and an endocyclic one which interconvert.
P.M. Bailey, B.E. Mann, A. Segnitz, K.L. Kaiser and P.M. Maitlis, J. Chem. Soc. Chem. Commun., (1974) 567.

Closo-1,1-$(Me_2PhP)_2$-2,4-Me_2-1,2,4-$PtC_2B_9H_9$, A and *nido*-10,10-$(Et_3P)_2$-2,8-Me_2-10,2,8-$PtC_2B_7H_7$, B
A. Triclinic, $R = 4.2\%$ for 4330 reflections, B. ($P2_1/a$) $R = 10.7\%$ for 2810 significant reflections. In A the metal coordinates three boron and two carbon atoms to complete a *closo*-2,4-dicarbaicosahedron. Pt is ~1.75 Å above the C_2B_3 face. In B, Pt coordinates boron atoms 5, 6, 7 and 9 to produce a *nido*-10-atom polyhedron whose geometry best approximates to a bicapped [B(9) and C(2)] square antiprism.
M. Green, J.L. Spencer, F.G.A. Stone and A.J. Welch, J. Chem. Soc. Chem. Commun., (1974) 571.

Dithiocarbamato derivatives of allyl molybdenum nitrosyls $[(\eta^5\text{-}C_5H_5)Mo(NO)(CO)\text{-}\{\eta^2\text{-}H_2C{:}CHCH_2SC({:}S)NMe_2\}]$, A and $[(\eta^5\text{-}C_5H_5)Mo(NO)(H_2CCH{:}CH_2)(\eta^2\text{-}S_2CNMe_2)]$, B

A. ($P2_1/c$) $Z = 4$, $R = 10\%$ for 1702 independent reflections, B. ($P2_1/c$) $Z = 4$, $R = 5.6\%$ for 772 independent reflections. In A, MoNO and MoCO are linear, Mo—N = 1.79, Mo—C = 1.97 Å, the dithiocarbamate ligand is bonded to the allylic group rather than to the metal atom. There is restricted rotation about the S—C and C—N bonds. In B, Mo—N—O (178°) is linear S_2CNMe_2 is planar as is the pentahapto cp ring.

N.A. Bailey, W.G. Kita, J.A. McCleverty, A.J. Murray, B.E. Mann and N.W.J. Walker, J. Chem. Soc. Chem. Commun., (1974) 592.

UBr_4, a new MX_4 structure type

($C2/m$) $Z = 4$, $R = 15\%$ for 274 background-corrected pattern intensities. UBr_4 is a new MX_4 structure type, the configuration around the uranium atoms being the pentagonal bipyramid [U Br $Br_{6/2}$]

J.C. Taylor and P.W. Wilson, J. Chem. Soc. Chem. Commun., (1974) 598.

An unusual copper(I) polynuclear species: tetrakis (copper trifluoroacetate)-dibenzene, $Cu_4(F_3CCO_2)_4 \cdot 2C_6H_6$

($P2_1/n$) $Z = 4$, $R = 10.6\%$ for 2342 unique reflections. The structure may be described as a planar parallelogram of four copper atoms, shortest Cu—Cu = 3.07(1) Å. These Cu atoms are held together by bridging trifluoroacetate groups (TFA). Cu—O = 1.83—2.2 Å, O—Cu—O = 162—175°.

P.F. Rodesiler and E.L. Amma, J. Chem. Soc. Chem. Commun., (1974) 599.

$[NBu_4]^+[\mu\text{-}Fe(CO)_4\text{—}B_7H_{12}]^-$

($P2_1/c$) $Z = 4$, $R = 11\%$ for 2283 reflections. The ion is a B_6 pentagonal pyramid with a BH_3 group and an $Fe(CO)_4$ group inserted into bridging positions between and beneath basal protons. B—Fe(avg.) = 2.20(2) Å.

O. Hollander, W.R. Clayton and S.G. Shore, J. Chem. Soc. Chem. Commun., (1974) 604.

Planar iridium(II) complexes; *trans*-$[Ir(Bu^t_2PC_6H_4O)_2]$, A and *trans*-$[Ir\{Bu^t_2PC_6H_3(OMe)O\}_2]$, B

A. ($P2_1/c$) $Z = 2$, $R = 6\%$ based on 1851 reflections, B. ($P\bar{1}$) $Z = 1$, $R = 6.7\%$ based on 3039 independent reflections. A has exact C_i symmetry and is rigorously planar, $\mu_{eff} = 1.76$ B.M. Ir—P = 2.303, Ir—O = 1.99 Å. B has C_i symmetry, $\mu_{eff} = 1.78$ B.M. Ir—P = 2.306, Ir—O = 2.00 Å.

R. Mason, K.M. Thomas, H.D. Empsall, S.R. Fletcher, P.N. Heys, E.M. Hyde, C.E. Jones and B.L. Shaw, J. Chem. Soc. Chem. Commun., (1974) 612.

Dimerization and condensation of pentane-2,4-dione by $MOCl_4$

[M = Mo or W], Structure of $[C_{10}H_{13}O_2]^+[C_5H_7Cl_2WO_4]^-$ ($P2_1/n$) $Z = 4$, $R = 4.5\%$ for 2268 independent reflections. In the anion the tungsten is in a six coordinate distorted octahedral environment. Bond lengths are normal.

M.G.B. Drew, G.W.A. Fowles, D.A. Rice and K.J. Shanton, J. Chem. Soc. Chem. Commun., (1974) 614.

Models for the methylmercury—protein interaction: a comparison of the molecular structures of methyl-4-cysteinatomercury(II) and methyl-DL-methioninemercury(II), $MeHgSCH_2CH(NH_3^+)CO_2^-$, H_2O, A and $MeHgMeS(CH_2)_2CH(NH_2)CO_2^-$, B

A. ($P2_12_12_1$) $Z = 4$, $R = 6.5\%$ based on 980 reflections. B. ($P2_1/c$) $Z = 4$, $R = 9.9\%$ based on 1027 reflections. In A the amino-acid coordinates in the zwitterionic form $-SCH_2CH(NH_3^+)CO_2^-$ to $MeHg^+$ via a deportonated sulphydryl group such that an almost linear C—Hg—S unit is formed. Hg—C = 2.09(4), Hg—S = 2.352(12) Å. In B the methionine is coordinated via an amino group, Hg—N = 2.06(4) Å.

Y.S. Wong, N.J. Taylor, P.C. Chieh and A.J. Carty, J. Chem. Soc. Chem. Commun., (1974) 625.

Planar Ti_2N_2 rings with five-coordinate titanium(IV), structure of *catena*-di-μ-chloro-bis-μ-(trimethylsilylamino)-di-[chlorotitanium(IV)], $[Me_3SiNTiCl_2]_n$

($P2_1/c$) $Z = 8$, $R = 10\%$ for 1298 observed reflections. The structure contains planar four-membered $(Ti{-}N)_2$ rings with planar geometry at N which are linked by Cl— bridges, giving five coordinate Ti atoms in approximately trigonal bipyramidal geometry. Ti—N (avg) = 1.89, Ti—Cl (avg. terminal) = 2.21, Ti—Cl (bridging) = 2.43 — 2.51 Å.

N.W. Alcock, M. Pierce-Butler and G.R. Willey, J. Chem. Soc. Chem. Commun., (1974) 627.

Reaction products of Hofmann's base with corresponding acid; tetrakis (trifluoroacetoxymercuri)methane, $C(HgOCOCF_3)_4$, A, and tetrakis (acetoxymercuri)methane, $C(HgOCOCH_3)_4$, B

A. ($P4_2/n$) $Z = 2$, $R = 2.8\%$ based on 1100 reflections, B. ($P2_1/c$) $Z = 4$, $R = 11.4\%$ based on 1830 reflections. In A the carbon atom is bonded to four Hg atoms, in an almost regular tetrahedron, Hg—C—Hg = 112.36(2)°. Each Hg is linked to one carboxyl oxygen, Hg—O = 2.065(10) Å. The structure of B is similar to A with C—Hg = 2.03—2.07, Hg—O = 2.02—2.18 Å.

D. Grdenic, B. Kamenar, B. Korpar-Čolig, M. Sikirica and G. Jovanovski, J. Chem. Soc. Chem. Commun., (1974) 646.

Trisdimethyltin(IV) bis(orthophosphate)octahydrate $(Me_2Sn)_3(PO_4)_2.8H_2O$

(*Pnma*) $Z = 4$, $R = 8.1\%$ for 1586 unique reflections. The structure of $(Me_2Sn)_3(PO_4)_2.8H_2O$ shows one tin atom in a regular octahedral environment with *trans*-methyl groups, and the other tin atoms in a highly distorted (∠C—Sn—C = 147°, 150°) tetrahedral configuration due to weak coordination to two water molecules; all the H_2O molecules are involved in hydrogen bonding to each other and to phosphate oxygen atoms.

J.P. Ashmore, T. Chivers, K.A. Kerr and J.H.G. Van Roode, J. Chem. Soc. Chem. Commun., (1974) 653.

Oxotrichlorotriphenylphosphidesulphidemolybdenum(V) $[MoOCl_3(SPPh_3)]$

($P2_12_12_1$) $Z = 4$, $R = 4.5\%$ based on 3122 reflections. The title compound exists as discrete five coordinate molecules which have a distorted square pyramidal geometry. Mo—O = 1.647(3), Mo—Cl = 2.342(1), 2.305(1), 2.325(1), Mo—S = 2.460(1) Å.

P.M. Boorman, C.D. Garner, F.E. Mabbs and T.J. King, J. Chem. Soc. Chem. Commun., (1974) 663.

Products from the hydrolysis of *cis*-$PdCl_2(Ph_2PC{\equiv}CCF_3)_2$, structure of a 1-diphenylphosphine-3,3,3-trifluoropropen-2-olato complex, $PdCl(PPh_2OEt)[Ph_2PCHC(CF_3)O]CH_2Cl_2$

($Pbca$) $Z = 8$, $R = 8.5\%$ based on 2020 and observed reflections. The Pd is coordinated by a chloride, a diphenylethoxyphosphine, and a deprotonated 1-diphenylphosphine-3,3,3-trifluoropropen-2-ol (Dptpo) bidentate ligand. Pd^{II}—O = 2.102(13), Pd—P = 2.226(5), 2.242(5) Å.

S. Jacobson, N.J. Taylor and A.J. Carty, J. Chem. Soc. Chem. Commun., (1974) 668.

Trimethylsilyl and trimethylgermyl group migration from ruthenium to cycloheptatriene; $[Ru(SiMe_3)(CO)_2C_7H_7(C_6F_5)(SiMe_3)]$

(Cc) $Z = 4$, $R = 7.0\%$. The structure establishes an *exo*-configuration for the migrant $SiMe_3$.

J.A.K. Howard, S.A.R. Knox, V. Riera, B.A. Sosinsky, F.G.A. Stone and P. Woodward, J. Chem. Soc. Chem. Commun., (1974) 673.

cis-Chlorodicarbonyl(tricyclohexylphosphine oxide)rhodium(I), *cis*-$[RhCl(CO)_2(Cy_3PO)]$

($P2_1/c$, C^5_{2h}, No. 14), $Z = 4$, $R = 6.5\%$ for 1905 independent reflections. The overall coordination geometry about the rhodium atom is closely square planar, with the two carbonyl ligands *cis*, Rh—Cl = 2.322(4), Rh—C = 1.72(1), 1.75(1), Rh—O = 2.049(7) Å.

G. Bandoli, D.A. Clemente, G. Deganello, G. Carturan, P. Uguagliati and U. Belluco, J. Organometal. Chem., 71 (1974) 125.

A rhodium acetyl complex, $[Me_3PhN^+]_2[Rh_2I_6(MeCO)_2(CO)_2]^{2-}$

($P2_1/c$) $Z = 2$, $R = 10.0\%$ for 2867 reflections. The coordination about the rhodium is approximately octahedral and the two octahedra are joined by a double halogen bridge across the symmetry centre. Rh—I (bridge) = 2.679(2), and 3.001(2), Rh—C (acetyl) = 2.062(23) Å.

G.W. Adamson, J.J. Daly and D. Forster, J. Organometal. Chem., 71 (1974) C17.

trans-$PtI(CMe{=}NC_6H_4Cl)(PEt_3)_2$

($P2_1/c$) $Z = 4$, $R = 3.8\%$ from 1791 independent reflections. The structure consists of discrete molecular units, with each platinum having four coordinate square planar geometry. Pt—P = 2.314(3), 2.321(3), Pt—I = 2.712(1), Pt—C = 2.027(11) Å.

K.P. Wagner, P.M. Treichel and J.C. Calabrese, J. Organometal. Chem., 71 (1974) 299.

Trimethylplatinum azide $(CH_3)_3PtN_3$

($P\bar{3}c1$) $Z = 4$, $R = 9.9\%$ for 1030 observed reflections. The cubane structure consists of $[(CH_3)_3PtN_3]_4$ molecules in which each α-N atom of the azido group links three Pt atoms.

M. Atam and U. Müller, J. Organometal. Chem., 71 (1974) 435.

A novel transition metal substituted tin hydride, $H_2Sn_2[Mn(CO)_5]_4$

($C2/c$) $Z = 4$, $R = 2{\cdot}0\%$ using 2968 independent reflections. The compound has C_2 symmetry in the crystal. The tin atoms are in a strongly distorted tetrahedral environment, Mn(2)—Sn(1)—Mn(2) = 119.8°, Mn(1)—Sn(1)—Sn(1)′ = 121.9°, Mn(2)—Sn(1)—Sn(1)′ = 107.7°. Sn—Mn = 2.67, 2.73, Sn—Sn = 2.89 Å.

K.D. Bos, E.J. Bulten, J.G. Noltes and A.L. Spek, J. Organometal. Chem., 71 (1974) C52.

Triphenylborane, $(C_6H_5)_3B$

($Pbcn$) $Z = 4$, $R = 5.5\%$ for 749 reflections. In contrast to triphenylgallium and triphenylindium no intermolecular interactions are found for $(C_6H_5)_3B$. The phenyl groups are tilted by about 30° with respect to the boron valence plane. B—C = 1.577 Å.

F. Zettler, H.D. Hansen and H. Hess, J. Organometal. Chem., 72 (1974) 157.

Dichlorobis(phenylamine)(bicyclo[2:2:1]-hepta-2,5-diene)ruthenium, $RuCl_2(C_7H_8)(C_6H_5NH_2)$

($C_{2v}^{12} - Cmc2_1$) $Z = 4$, $R = 2.5\%$ for 2642 observed reflections. The coordination geometry about the Ru atom is distorted octahedral with the norbornadiene moiety bound through the two double bonds, *trans*-chloro and *cis*-aniline ligands. Ru—ct (where ct is the centre of the olefinic bond) = 2.066(4), Ru—N = 2.213(3), Ru—Cl = 2.415(2), 2.407(1) Å.

J. Manoli, A.P. Gaughan Jr. and J.A. Ibers, J. Organometal. Chem., 72 (1974) 247.

Dichloro(difluoromethyl)carbonylbis(triphenylphosphine)iridium(III), $IrCl_2(CHF_2)(CO)(PPh_3)_2$

($P2_1/c$) $Z = 4$, $R = 5.9\%$ based on 1945 observed reflections. The structure consists of a slightly distorted octahedron *trans*-phosphine ligands. The difluoromethyl group is *trans* to Cl, Ir—C = 2.19(3) Å.

A.J. Schultz, J.V. McArdle, G.P. Khare and R. Eisenberg, J. Organometal. Chem., 72 (1974) 415.

Acetylacetonatoacetylacetonyltriphenylphosphine palladium(II) benzene solvate, $[Pd(acac)_2(PPh_3)0.5C_6H_6]$

($C2/c$) $Z = 8$, $R = 7.8\%$ for 2806 independent reflections. The coordination geometry around Pd is square-planar. One of the acetyl-acetone ligands is bonded to the Pd via its oxygen atoms, Pd—O = 2.048(10) and 2.062(10), and the other by its γ-carbon atom, Pd—C = 2.114(13) Å.
M. Horike, Y. Kai, N. Yasuoka and N. Kasai, J. Organometal. Chem., 72 (1974) 441.

trans-[Methyl(2-oxacyclopentylidene)bis(dimethylphenylphosphine)platinum(II)] hexafluorophosphate, *trans*-$[CH_3(CH_2COCH_2CH_2)Pt\{P(CH_3)_2C_6H_5\}_2]$-$(PF_6)$
($Pcca(D^8_{2h})$) $Z = 8$, $R = 4.4\%$ for 1763 reflections. The coordination geometry about Pt is square-planar. The cyclic carbene ligand is almost planar and is coordinated approximately perpendicular to the Pt square plane. Pt—P (mean) = 2.305(5), Pt—C(sp^3) = 2.080(18) and Pt—C(sp^2) = 2.00(2) Å.
R.F. Stepaniak and N.C. Payne, J. Organometal. Chem., 72 (1974) 453.

(Isopropylideneamino)dimethylaluminium dimer, $[(CH_3)_2AlNC(CH_3)_2]_2$
($P\bar{1}$) $Z = 1$, $R = 5.8\%$ for 1230 independent reflections. Mechanism of formation from thermal decomposition of $K[Al_2(CH_3)_6SCN]$ at 120° is discussed. Al—N = 1.926(2) Å.
S.K. Seal and J.L. Atwood, J. Organometal. Chem., 73 (1974) 27.

Racemic form of $(h^5\text{-}3\text{-}MeC_5H_3C(Me_2)C_6H_5)(h^5\text{-}C_5H_5)Ti(2,6\text{-}Me_2\text{-}C_6H_3O)Cl$
($P2_1/n$), $R = 6.2\%$ for 1939 reflections. The relative configuration of the two chiral moieties has been established, and this compound can be used as a reference for studies on dynamic stereochemistry around the titanium atom.
C. Lecomte, Y. Dusansoy, J. Protas, J. Tirouflet and A. Dormond, J. Organometal. Chem., 73 (1974) 67.

Bis[(η^5-cyclopentadienyldicarbonyliron)dimethylgermyl] oxide, $[(\eta^5\text{-}C_5H_5)(CO)_2FeGe(CH_3)_2]_2O$
($P2_1/n$) $Z = 4$, $R = 5.1\%$ based on 1780 reflections. The compound consists of two $(\eta^5\text{-}C_5H_5)(CO)_2FeGe(CH_3)_2$ moieties joined by a bridging oxygen atom. Two rotational isomers are present in the unit cell in a disordered fashion. Fe—Ge = 2.372, Ge—O = 1.785 Å, Ge—O—Ge′ = 134°.
R.D. Adams, F.A. Cotton and B.A. Frenz, J. Organometal. Chem., 73 (1974) 93.

Structures of π-arene complexes of the enneacarbonyltetracobalt cluster with *ortho*- and *meta*-xylene (a mixed crystal) and with benzene, $C_6H_4(CH_3)_2Co_4(CO)_9$, A, and $C_6H_6Co_4(CO)_9$, B
A. ($P2_1/c$) $Z = 4$, $R = 5.6\%$ for 1883 independent reflections. B. ($R\bar{3}$, No. 148) $Z = 2$, $R = 3.3\%$ for 491 reflections. Both structures consist of a tetrahedral cobalt cluster, one cobalt atom is π-bonded to the arene moiety,

while the other three are each bonded to two terminal and two bridging carbonyl groups. Base to apex Co—Co = 2.481(2) avg., basal Co—Co = 2.455(2) Å.

P.H. Bird and A.R. Fraser, J. Organometal. Chem., 73 (1974) 103.

A dicarbenoid complex of gold(III), $[\{p\text{-MeC}_6H_4NH)_2C\}_2AuI_2]ClO_4 \cdot Et_2O$ ($P2_1/c$) $Z = 4$, $R = 5.4\%$ based on 3521 independent reflections. The discrete gold(III) cations contain Au^{III} in a square planar environment, with the chemically equivalent ligands *trans* to each other. The *trans* influence of the carbenoid ligands is discussed. Au—C = 2.07(2), 2.09(2), Au—I = 2.601(2), 2.606(2) Å.

L. Manojlovic-Muir, J. Organometal. Chem., 73 (1974) C45.

An optically active diphosphine—nickel complex, $NiCl_2[(-)\text{-Diop}]$ ($P2_12_12_1$) $Z = 4$, $R = 7.8\%$ for 1156 reflections. The molecular structure of dichloro[(—)-2,3-*O*-isopropylidene-2,3-dihydroxy-1,4-bisdiphenylphosphino)butane]nickel(II) shows the coordination geometry around the nickel to be that of a strongly distorted tetrahedron. Ni—Cl = 2.20(1), Ni—P = 2.30(1) Å, Cl—Ni—Cl = 130°.

V. Gramlich and Ch. Salomon, J. Organometal. Chem., 73 (1974) C61.

Bis(6,6-dimethylpentafulvene)pentacarbonyldiiron, A, and 6,6-diphenylpentafulvenepentacarbonyldiiron, B

A. ($P2_1/c$) $Z = 4$, $R = 2.7\%$ based on 1773 reflections, B. ($P2_1/c$) $Z = 4$, $R = 4.0\%$ based on 2040 reflections. These structures provide the first detailed information about the bonding in 6,6-disubstituted pentafulvenecarbonyliron complexes.

U. Behrens and E. Weiss, J. Organometal. Chem., 73 (1974) C64.

6,6-Diphenylpentafulvenepentacarbonyldiruthenium, triclinic modification. ($P\bar{1}$) $Z = 2$, $R = 2.8\%$ for 3165 reflections. Detailed structural data are given. Ru—Ru = 2.845 Å.

U. Behrens and E. Weiss, J. Organometal. Chem., 73 (1974) C67.

(Dibenzylideneacetone)(pentamethylcyclopentadienyl)rhodium(I), $Rh(C_5Me_5)(dba)$

(C_{2h}^5—$P2_1/a$) $Z = 4$, $R = 3.5\%$ based on 2100 reflections. The compound is monomeric. The Rh atom is bonded to the C_5Me_5 ring on one side and to the dba molecule through the olefinic double bonds on the other side. The dba molecule is in the *S-cis*, *S-cis* conformation.

J.A. Ibers, J. Organometal. Chem., 73 (1974) 389.

A trinuclear π-cyclopentadienylniobium complex containing the formate, hydroxylic and oxygen bridges, $[C_5H_5NbOOCH_3]_3(OH)_2(O)_2$

Monoclinic, $Z = 4$, $R = 11.5\%$. The niobium atoms are located at the vertices of an almost regular triangle with Nb—Nb = 3.14 Å. The location of the Nb atoms indicates that they bind only by the formate, hydroxylic and oxygen bridges instead of by direct Nb—Nb bonds.

V.T. Kalinnikov, A.A. Pasyaskii, G.M. Larin, V.M. Novotortsev, T. Struchkov, A.I. Gusev and N.I. Kirillova, J. Organometal. Chem., 74 (1974) 91.

Bis(tri-*p*-tolyphosphine)(*trans*-stilbene)nickel(0) hemitetrahydrofuranate, $Ni[P(C_6H_4CH_3)_3]_2[(C_6H_5)HC = CH(C_6H_5)]. 0.5(C_4H_8O)$

(D_{2h}^{10}—*Pccn*) $Z = 8$, $R = 9.2\%$ based on 2417 reflections. The Ni atom has trigonal coordination if the olefin is regarded as a monodentate ligand. The two olefinic carbon atoms are equidistant from the Ni atom. Ni—P (avg.) = 2.181(4), Ni—C (avg.) = 2.019(13) Å.

S.D. Ittel and J.A. Ibers, J. Organometal. Chem., 74 (1974) 121.

$CaNaHSiO_4$

(*C*222,) $Z = 4$, $R = 8.4\%$. The crystal structure of $CaNaHSiO_4$ has been interpreted by means of the rhomb method.

V.I. Lyatin, E.A. Kuz'min, V.V. Ilyukhin and N.V. Belov, Sov. Phys. Crystallogr., 19 (1974) 60.

Silver vanadate, $Ag_{4-x}V_4O_{12}$, $x = 1.05$.

The structure of the title compound has been solved using the Shenk method.

Yu.N. Drozdov, E.A. Kuz'min and N.V. Belov, Sov. Phys. Crystallogr., 19 (1974) 65.

Bis(pyridinealdehydethiosemicarbazonate)cobalt(III) chloride, $[Co(C_7H_7N_4S)_2]Cl.3H_2O$

(*I*112/*a*) $Z = 8$, $R = 14\%$ for 990 non-zero reflections. The ligands ($C_7H_7N_4S$ = PAT) were found to be planar. The coordination geometry around Co^{III} is distorted octahedral, comprising the sulphur and nitrogen atoms of the hydrazine residues and pyridine from two PAT ligands. Co—S = 2.22(2), 2.28(2) Å. Co—N = 2.01, 1.87, 1.90 Å.

T.N. Tarkhova, K.N. Akatova and N.V. Belov, Sov. Phys. Crystallogr., 19 (1974) 70.

Rubidium ozonide, RbO_3 at —16°C

($P2_1/c$) $Z = 4$, $R = 15\%$. The structure consists of intersecting layers of ozonide groups with rows of rubidium atoms between them. The angle between the O—O bonds in the triangular O_3^- ion is 101°, O—O = 1.40, 1.45 (1.42 - mean) Å.

V.A. Sarin, V.Ya. Dundarev and M.S. Dubrolyubova, Sov. Phys. Crystallogr., 19 (1974) 74.

Structures of $Ce_6Ni_6Si_3$ and related compounds

The structure of $Ce_6Ni_6Si_3$ is of a new type and the following isostructural compounds have been found: $La_6Ni_2Si_3$, $Pr_6Ni_2Si_3$, $Nd_6Ni_2Si_3$.

O.I. Bodak, E.I. Gladyshevskii and O.I. Kharchenko, Sov. Phys. Crystallogr., 19 (1974) 80.

Potassium europium molybdate, $KEu(MoO_4)_2$

($P\bar{1}$) $Z = 4$, $R = 11\%$ for 1500 reflections. In the structure two independent Mo atoms have tetrahedral coordination (Mo—O mean = 1.78, 1.79 Å), and the K and Eu atoms lie within eight vertex figures. The polyhedra are linked into a three dimensional structure via common edges of the K and Eu eight-vertex figures and vertices of the Mo tetrahedra. Eu—O (avg.) = 2.43, K—O (avg.) = 2.78 Å.

R.F. Klevtsova, L.P. Kozeeva and P.V. Klevtsov, Sov. Phys. Crystallogr., 19 (1974) 89.

Vanadium garnet, $Na_{0.9}Ca_{2.05}Co_2V_3O_{12}$

Metal oxygen distances, V—O = 1.72, Co—O = 2.09, Na, Ca—O = 2.43 Å. Comparison of the structure with that of other garnets shows the vanadates to have the most regular MO_4 tetrahedra.

E.L. Dukhovskaya and B.V. Mill, Sov. Phys. Crystallogr., 19 (1974) 84.

$Li_2Cr_2O_7.2H_2O$, a neutron and X-ray diffraction study

The complete structure is $Li_2Cr_2O_6(OH)_2.H_2O$.

I.D. Datt and R.P. Ozerov, Sov. Phys. Crystallogr., 19 (1974) 110.

Adduct of Cu^{II} salicylate with dioxane, $Cu_2(HOC_6H_4COO)_4C_4H_8O_2.2H_2O$

($P2_1/c$) $Z = 2$, $R = 18.6\%$. The structure consists of $Cu_2(HOC_6H_4COO)_4.2H_2O$ dimers, Cu—Cu = 2.64 Å. Each copper has octahedral coordination, made up of four oxygen atoms of the carboxyl groups (Cu—O = 1.98—2.03 Å), the neighbouring copper atom and H_2O.

A.V. Ablov, G.A. Kiosse, G.I. Dimitrova, T.I. Malinovskii and G.A. Popovich, Sov. Phys. Crystallogr., 19 (1974) 168.

The tetrahydrate of europium nitrato-bis-isonicotinate, $[Eu(\text{iso-Nicot})_2(H_2O)_4]NO_3$

($C2/c$) $Z = 4$, $R = 11.9\%$ for 1988 independent reflections. The structure contains infinite chains of $\{[Eu(\text{iso-Nicot})_2(H_2O)_4]^+\}$. The bidentate-bridge carboxyl groups provide a bond between the neighbouring Eu atoms forming eight membered centrosymmetrical metal cycles. $Eu—O_{coo}$ = 2.34—2.41 Å, $Eu—O_{(H_2O)}$ = 2.51—2.54 Å.

L.A. Aslanov, I.D. Kiekbaev, I.K. Abdul'minev and M.A. Porai-Koshits, Sov. Phys. Crystallogr., 19 (1974) 170.

The double oxide, $UMo_{10}O_{32}$

($Cccm$) $Z = 8$, $R = 13.6\%$ for 448 independent reflections. It is possible to

distinguish infinite layers of Mo_2O_7 in the structure. The coordination polyhedra of U and Mo between the layers are $(UO_2)O_6$ hexagonal bipyramids and MoO_6 octahedra.
V.N. Serezhkin, L.M. Kovba and V.K. Trunov, Sov. Phys. Crystallogr., 19 (1974) 231.

Lithium aluminoborate, $Li_6[Al_2(BO_3)_4]$
($P\bar{1}$) $Z = 1$, $R = 10.6\%$ for 150 non-zero reflections. The structure contains infinite chains of the aluminoborate radical $[Al_2(BO_3)_4]_{\infty}^{6-}$ and band of lithium tetrahedra.
G.K. Abdullaev and Kh.S. Mamedov, Sov. Phys. Crystallogr., 19 (1974) 165.

Dithiocyanato-bis(thiosemicarbazide)nickel(II), $Ni(NCS)_2(CH_5N_3S)_2$
($P2_1/c$) $Z = 2$, $R = 13.8\%$ for 590 observed reflections. The nickel atom is coordinated octahedrally by four nitrogen and two sulphur atoms. The thiosemicarbazide molecule is bonded to the nickel atom as a chelate through the nitrogen atom of the hydrazine group (2.13(2) Å) and the sulphur atom (2.45(1) Å). Ni—N(NCS) = 2.05(2) Å.
M. Dunaj-Jurčo, J. Garaj and A. Sirota, Collect. Czech. Chem. Commun., 39 (1974) 236.

Copper(II) dicyanato-2,4-lutidine, $[Cu(NCO)_2(2,4\text{-lutidine})]$
($P\bar{1}$) $Z = 2$, $R = 16.5\%$ for 1087 independent non-zero reflections. The structure consists of polynuclear chains. The approximately square-planar coordination around the Cu^{II} formed by three nitrogen atoms from OCN groups, and one nitrogen from 2,4-lutidine, is completed by one nitrogen atom from an OCN group to form distorted tetragonal pyramidal coordination, (or a hybrid of a tetragonal pyramid with a trigonal bipyramid). Cyanate groups function as bridges between two copper atoms. Cu—Cu = 3.22—3.44 Å, $Cu—N_1$ = 1.98—2.06, $Cu—N_2$ = 2.46 Å.
F. Valach, M. Dunaj-Jurčo, J. Garaj and M. Hvastijova, Collect. Czech. Chem. Commun., 39 (1974) 380.

Tetramethyloctasila-dodecascaphan, $Si_8C_{17}H_{36}$
($F\bar{4}3c$-T_d^5) $Z = 4$, $R = 5.8\%$ for 247 reflections. In the polycyclic skeleton Si—C = 1.885(2), 1.893(5) and 1.888(6), whereas $Si—CH_3$ = 1.874(8) Å.
H.G.V. Schnering, G. Sawitski, K. Peters and K.F. Tebbe, Z. Anorg. Allg. Chem., 404 (1974) 38.

On chalkogenolates. LXV, Studies on thioformic acids, structure of potassium dithioformate, $K[HCS_2]$
($I4/mcm$) $Z = 8$, $R = 9.3\%$ for 266 reflections. The structure contains sheets of thioformate ions at $Z = 0$ and 0.5 with sheets of K ions half way between the parallel XY-plane. The coordination number of K is 8, that of S is 4.
R. Engler, G. Kiel and G. Gattow, Z. Anorg. Allg. Chem., 404 (1974) 71.

Bis(cis-1-mercapto-2-*p*-bromobenzoyl-ethylene)nickel(II)
($C2/c$) $Z = 8$, $R = 10.8\%$ for 4600 reflections. The complex is square planar $Ni[S_2O_2]$, Ni—S = 2.14, Ni—O = 1.88 Å. The sulphur atoms are arranged in *cis*-positions. The quasiaromatic character of the chelate system is discussed.
L. Kutschabsky, Z. Anorg. Allg. Chem., 404 (1974) 239.

Iodo-bis-[2-(diphenylphosphinomethyl)pyridine]nickel(II) iodide, $C_{36}H_{32}I_2N_2NiP_2$
($P2_1/n$) $Z = 4$, $R = 6.1\%$ for 4321 observed reflections. The coordination around Ni is approximately square pyramidal with two nitrogen and two phosphorus atoms in the basal plane and iodine at the apex, Ni—I = 3.047(3) Å. The N and P atoms have a *cis* configuration. Ni—N (mean) = 1.963(13), Ni—P = 2.166(5) Å.
W. Haase, Z. Anorg. Allg. Chem., 404 (1974) 273.

A new crystal structure of the general formula $Me^{2+}M_2^{3+}O_4$; $SrCr_2O_4$
($Pmmn$-D_{2h}^{13}). $SrCr_2O_4$ shows a hitherto unknown crystal type. Cr^{3+} is octahedrally and Sr^{2+} trigonal prismatically surrounded by oxygen.
H. Pausch and Hk. Müller-Buschbaum, Z. Anorg. Allg. Chem., 405 (1974) 1.

α-$CaCr_2O_4$
($Pmmn$-D_{2h}^{13}). α-$CaCr_2O_4$ is isotypic with $SrCr_2O_4$.
H. Pausch and Hk. Müller-Buschbaum, Z. Anorg. Allg. Chem., 405 (1974) 113.

The tetrachloride azides of niobium and tantalum, structure of tantalum tetrachloride azide, $TaCl_4N_3$
($P2_1/c$) $Z = 8$, $R = 8.2\%$ for 1263 reflections. $TaCl_4N_3$ is dimeric in solution and in the solid state. The monomers are linked via the αN atoms of the azide groups to form the centrosymmetric dimers. Two slightly different forms of the dimers are found in the crystal structure. They differ in the angle between the azide group and the $(Ta—\alpha N)_2$ ring. Vibrational spectra are reported.
J. Strähle, Z. Anorg. Allg. Chem., 405 (1974) 139.

Cyclooctadecasulphur, S_{18}, A and cycloicosasulphur, S_{20}, B
A. ($P2_12_12_1$) $R = 3.7\%$ for 1872 observed reflections; B. (PbCN)lc(CN) $R = 8.9\%$ for 1135 observed reflections. In S_{18} S—S = 2.059(3) (avg.), in S_{20} S—S = 2.047(5) (avg.) Å. Preparations and structures are discussed.
H. Schmidt, E. Wilhelm, I. Debaerdemaeker, E. Hellner and A. Kutoglu, Z. Anorg. Allg. Chem., 405 (1974) 153.

2-Chloro-1,3,6-trithia-2-stibaoctane, ($C_4H_8ClS_3Sb$)
($P2_12_12_1$) $Z = 8$, $R = 3.8\%$ for 2231 observed reflections. The eight-membered ring has a deformed boat-conformation with 1,5-intraannular

Sb—S-interaction. The coordination around Sb is approximately trigonal bipyramidal with axial distances Sb—Cl = 2.51, Sb—S = 2.86 Å, and equatorial Sb—S = 2.44 and 2.45 Å.
M. Dräger and R. Engler, Z. Anorg. Allg. Chem., 405 (1974) 183.

$Ba_2Tl_2O_5$
($Pcmn\text{-}D_{2h}^{16}$) $R = 8\%$. $Ba_2Tl_2O_5$ is isotypic with $Ca_2Fe_2O_5$.
R.V. Schenck and Hk. Müller-Buschbaum, Z. Anorg. Allg. Chem., 405 (1974) 197.

The quaternary fluoride, $BaLiCrF_6$
($P2_1/c$) $Z = 4$, $R = 5.7\%$ for 1561 observed reflections. The structure contains icosahedra of BaF_{12} (Ba—F = 2.89 Å) within a framework built up of isolated LiF_4 tetrahedra (Li—F = 1.86 Å) and CrF_6-octahedra (Cr—F = 1.90 Å) mutually linked by corners. All polyhedra are distorted.
D. Babel, Z. Anorg. Allg. Chem., 406 (1974) 23.

Compounds of the type $Cu_3MS_xSe_{4-x}$ (M = Nb, Ta), structure of Cu_3TaSSe_3
The preparation, X-ray data, vibrational and electronic spectra of Cu_3NbS_3Se, $Cu_3NbS_2Se_2$, Cu_3NbSSe_3, Cu_3TaS_3Se, $Cu_3TaS_2Se_2$ and Cu_3TaSSe_3 are reported. The powder patterns of all compounds could be indexed on the basis of a simple cubic lattice. The compounds crystallize in pseudo-sulvanite type with the S and Se atoms in random distribution.
A. Müller and W. Sievert, Z. Anorg. Allg. Chem., 406 (1974) 80.

Tricyclic transition metal complexes with ring systems containing silicon: structures of $Ni(C_6H_{17}N_5S_4Si_2)$, A, and $Co(C_4H_{12}N_4S_4OSi_2)$, B
A. ($Pbcn$) $Z = 4$, $R = 6.0\%$ for 914 observed reflections; B. ($P2_1/c$) $Z = 4$, $R = 11.4\%$ for 1164 observed reflections. The two almost planar MN_2S_2 chelate rings per molecule intersect with an angle of 16.2° in A and 27.0° in B. The six-membered rings containing Si are puckered in such a way that both molecules exhibit (approximate) C_2 symmetry.
U. Thewalt and M. Schlingmann, Z. Anorg. Allg. Chem., 406 (1974) 319.

$S_3N_5PF_2$
($R3m\text{-}C_{3v}^{5}$) $Z = 9$, $R = 4.3\%$ for 499 independent reflections. The structure is built of discrete $S_3N_5PF_2$ molecules which can be looked upon as S_4N_4 derivatives: two opposite sulfur atoms are N-bridged and a third is substituted for a PF_2 group. The environment of phosphorus is tetrahedral, S—N = 1.69, S···N = 1.61, S=N = 1.55, P—N = 1.56, P—F = 1.51 Å.
J. Weiss, I. Ruppert and R. Appel, Z. Anorg. Allg. Chem., 406 (1974) 329.

Ba_2CrO_4
($Pn2_1a\text{-}C_{2v}^{9}$). A discussion is given concerning the correlation between Ba_2CrO_4 and the hitherto known crystal structures of Ba_2CoO_4 and Ba_2TiO_4.

Hj. Mattausch and Hk. Müller-Buschbaum, Z. Anorg. Allg. Chem., 407 (1974) 129.

Thiotellurites, $BaTeS_3.2H_2O$, A, and $(NH_4)TeS_3$, B

A. ($Pbcm$-D_{2h}^{11}) $Z = 4$, $R = 11.5\%$ for 424 observed reflections; B. ($Pnma$-D_{2h}^{16}) $Z = 4$, $R = 10\%$ for 2251 observed reflections. The structures show that the anion of trithiotelluric acid TeS_3^{2-} is a distorted trigonal pyramid. Te—S = 2.34—2.36 Å.

H. Gerl, B. Eisenmann, P. Roth and H. Schafer, Z. Anorg. Allg. Chem., 407 (1974) 135.

Two modifications of dichloro-2-2′-bipyridylplatinum(II), green and yellow $[PtCl_2dipy]$

The difference between both forms consists only in the packing of the planar complex units within the crystal. The red coloured $[PtCl_2dipy]$ contains a columnar structure in which the nearest distance between planar complex units stacked above each other is 3.40 Å. The yellow modification does not form a columnar structure. It contains isolated molecules, (shortest Pt...Pt = 4.5 Å).

M. Textor and H.R. Oswald, Z. Anorg. Allg. Chem., 407 (1974) 244.

$(C_6H_5)_2SnSAT$ (SAT = tridentate dianion from 2-(*o*-hydroxyphenyl)benzothiazoline)

($P2_1/n$-C_{2h}^5) $Z = 4$, $R = 3.9\%$ for 3150 independent reflections. The coordination polyhedron around Sn has essentially trigonal bipyramidal structure, which is distorted, N and two Sn—phenyl groups occupy the equatorial positions. Sn—S = 2.496(1), Sn—O = 2.093(2), Sn—N = 2.217(3), Sn—C(phenyl) = 2.120(3), 2.126(3) Å.

H. Preut, H.J. Haupt, F. Huber, R. Cefalu and R. Barbieri, Z. Anorg. Allg. Chem., 407 (1974) 257.