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X-RAY BIBLIOGRAPHY

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The structures of the following compounds appeared in:

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(i) The potassium salt of the binuclear Mo^{III} anion, di-μ-hydroxo-μ-acetato-μ-ethylenediaminetetraacetato-bis[molybdenum(III)], $K[Mo_2(OH)_2(C_{12}H_{15}O_{10}N_2)]$

(ii) The rubidium salt of *N*-(purin-6-ylcarbamoyl)-L-threonine tetrahydrate, a hypermodified base in the anticodon loop of some tRNA's, $C_{10}H_{11}N_6O_4Rb4H_2O$

(iii) $(+)_{589}$-*cis*-β-Carbonato-(3*S*,8*S*-dimethyltriethylenetetramine)cobalt(III) perchlorate

(iv) Indium monoselenide, (InSe)

(v) Te_4O_9

(iv) The stereochemistry of some organic derivatives of group Vb elements, IX. Three severely disordered molecules: structures of the perchlorate, chloride and azide derivatives, $[(CH_3)_3SbX]_2O$, derivatives of μ-oxo-trimethylantimony(V), $[(CH_3)_3SbX]_2O$

(vii) Caesium pentamolybdate, $Cs_2Mo_5O_{16}$ and caesium heptamolybdate, $Cs_2Mo_7O_{22}$

(viii) $Yb_{0.5}Eu_{0.5}Fe_2O_4$

(ix) An independent investigation of the crystal structure of $Na_2Cu(CO_3)_2$-$(H_2O)_3$ with diffractometer data

(x) Calcium *cis*(N)-*trans*(O_6)-bis-(L-aspartato)cobaltate(III)-water(2/15), 2{Ca-*cis*(N)-*trans*(O_6)-[Co(L-asp)$_2$]$_2$}.15H_2O

(xi) A five-coordinate copper(II) macrocyclic complex: chloro-(2,7,12-trimethyl-3,7,11,17-tetraazabicyclo[11,3,1]heptadeca-1(17),2,11,13,15-pentaene)copper(II) nitrate dihydrate, $Cu(C_{16}H_{24}N_4)(Cl)(NO_3).2H_2O$

(xii) The dimeric 6-mercaptopurine copper(1) chloride complex, $[C_5H_5N_4S.CuCl_2.H_2O]_2$

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(iii) An unusual sulphur incorporation: formation and crystal structure of $[\overline{Mn(CO)_3\{\mu\text{-}SC(SMe)}NMe\}]_2$

J. Chem. Soc. Chem. Commun., No. 11, 1975

(i) Unusual bonding patterns in halogen-substituted η^5-cyclopentadienyl metal derivatives; structure of η^5-pentachlorocyclopentadienylcyclooctadienerhodium

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(iv) $[Pt(C_2F_4)(C_2H_4)_2]$ and tris(bicyclo[2.2.1]heptene)platinum

(v) Reactions of hexafluoacetone, hexafluoropropene and tetrafluoroethylene with bis(cycloocta-1,5-diene)platinum; structure of $[Pt_2\{(CF_3)_2CO\}(1,5\text{-}C_8H_{12})_2]$

Cesium azidotrimethyl aluminate, $Cs[Al(CH_3)_3N_3]$

(*Pbcm*) $Z = 4$, $R = 4.6\%$ from 556 independent reflections. The anion lies on a crystallographic mirror plane; Al—N = 1.97(1) Å. Within the azide ion the two nitrogen—nitrogen lengths differ: 1.13(2) and 1.21(2) Å.

J.L. Atwood and W.R. Newberry, III, *J. Organometal. Chem.*, 87 (1975) 1.

The Chemistry and the stereochemistry of poly(*N*-alkyliminoalkanes), III. Structure of the adduct $(HAlN\text{—}i\text{-}Pr)_6AlH_3$

($P\bar{1}$) $Z = 2$, $R = 4.9\%$ from 4358 reflections. The cage-type molecular structure consists of two six-membered rings, $(AlN)_3$ joined together by four adjacent transverse Al—N bonds; the loss of two of these bonds allows the complexation of one alane molecule, thus Al is five-coordinate (trigonal bipyramidal geometry), with two Al—N and two Al—H—Al bridge bonds. Al—N = 1.873—1.959 Å. Al—H(avg) = 1.50(1) Å for the four coordinate Al.

G. Perego, M. Cesari, G. Del Piero, A. Balducci and E. Cernia, *J. Organometal. Chem.*, 87 (1975) 33.

The chemistry and the stereochemistry of poly(*N*-alkyliminoalkanes), IV. Structure of $[H(HAlN\text{—}i\text{-}Pr)_5AlH_2] \cdot LiH/Et_2O$

($Pna2_1$) $Z = 4$, $R = 5.9\%$ from 2487 independent reflections. The structure is a pseudo-hexameric cage consisting of a five-membered fragment, Al—N—Al—N—Al, crosslinked to a six-membered cyclohexane type ring, $(AlN)_3$. Al—N(avg) = 1.919(4) Å.

M. Cesari, G. Parego, G. Del Piero, M. Corbellini and I. Immirzi, *J. Organometal. Chem.*, 87 (1975) 43.

The chemistry and the stereochemistry of poly(*N*-alkyliminoalkanes), V. Structure of the pentamer$[(HAlN{-}i\text{-}Pr)_2(H_2AlNH{-}i\text{-}Pr)_3]$

($C2/c$) $Z = 4$, $R = 5.6\%$ from 1826 reflections. The molecule is made up of a cyclohexane-type ring, $[(HAlN{-}i\text{-}Pr)_2(H_2AlNH{-}i\text{-}Pr)]$, in skew boat conformation, on each side of which is bonded an $-H_2AlNH{-}i\text{-}Pr-$ bridging unit between a nitrogen atom and an aluminum atom of the ring. Al—N = 1.901—1.985 Å.

G. Perego, G. Del Piero, M. Cesari, A. Zazzetta and G. Dozzi, *J. Organometal. Chem.*, 87 (1975) 53.

Nitratotris(triphenylstannyl)tin(IV)

($P2_1/n$) $Z = 4$, $R = 9.8\%$ from 8404 independent reflections. The structure of the new organotin nitrate compound of formula $[Sn^{IV}(NO_3)\{(C_6H_5)_3Sn^{IV}\}_3]$ is reported. It contains a tetranuclear system of tin atoms.

G. Pelizzi, *J. Organometal. Chem.*, 87 (1975) C1.

Triphenylsilicon isothiocyanate, Ph_3SiNCS

($P2_1/c$) $Z = 4$, $R = 5.5\%$ from 2841 reflections. The structure consists of isolated molecules. Some mean lengths and angles are: Si—N = 1.735(5), Si—C = 1.851(4), N—C = 1.156(4) Å, C—Si—N = 106.3(8)°.

G.M. Sheldrick and R. Taylor, *J. Organometal. Chem.*, 87 (1975) 145.

$Fe_2(CO)_6[P(C_6F_5)_2][(C_6F_5)_2PC_4(C_6H_5)_2]$: A new type of organoiron complex containing a 3-electron donor phosphorus heterocycle

($P2_1/c$) $Z = 4$, $R = 8.7\%$ from 4296 reflections. A new five-membered phosphorus heterocycle coordinated to two iron atoms in the binuclear complex $Fe_2(CO)_6[(C_6H_5)_2PC_4(Ph)_2]P(C_6F_5)_2$ is formed in the reaction of $Fe_2(CO)_9$ with $(C_6F_5)_2PC{\equiv}CPh$. The heterocycle, a derivative of 1-bis(pentafluorophenyl)phosphonia-2,5-diphenyl-2,4-cyclopentadiene, functions as a symmetrical *dihapto* bridging 3*e* ligand.

N.J. Taylor, H.N. Paik, P.C. Chieh and A.J. Carty, *J. Organometal. Chem.*, 87 (1975) C31.

fac, A, and *mer*, B, -*trans*-Bromotricarbonylbis(dimethoxyphenylphosphine) manganese(I)

A. ($P2_12_12_1$) $Z = 4$, $R = 4.1\%$ from 1498 observed reflections. B. ($P2_1/n$) $Z = 8$, $R = 11.6\%$ from 3191 observed reflections. Both structures exhibit slightly distorted octahedral geometry about the manganese with one bromine, two phosphorus and three carbon atoms occupying the coordinate positions. A significant structural *trans*-effect of the carbonyl groups paralleling the known strong carbonyl kinetic *trans*-effect was not observed.

G.J. Kruger, R.O. Heckroodt, R.H. Reimann and E. Singleton, *J. Organometal. Chem.*, 87 (1975) 323.

Dicyclopentadienylvanadium, $(C_5H_5)_2V$ and dicyclopentadienylchromium, $(C_5H_5)_2Cr$, determined by gas phase electron diffraction

The best agreement between calculated and experimental curves is obtained for models with eclipsed C_5H_5 rings, D_{5h} symmetry, but models with staggered rings (D_{5d} symmetry) cannot be definitely ruled out. Cr—C = 2.169(4), C—C = 1.431(2) in $(C_5H_5)_2Cr$, V—C = 2.280(5), C—C = 1.434(3) Å in $(C_5H_5)_2V$. The C—H bonds in $(C_5H_5)_2Cr$ are bent 2.9(1.1)° out of the plane of the carbon atoms towards the metal atom.

E. Gard, A. Haarland, D.P. Novak and R. Seip, *J. Organometal. Chem.*, 88 (1975) 181.

Trifluorosilylmanganese pentacarbonyl, an electron diffraction determination in the gas phase

The principle parameters in $SiF_3Mn(CO)_5$ are: r_a(C—O) = 113.1(3), r(Mn—C)$_{av}$ = 186.0(6), r(Mn—Si) = 236.0(7), r(Si—F) = 158.3(4) pm. <(F—Si—F) = 112.5(4).

D.W.H. Rankin and A. Robertson, *J. Organometal. Chem.*, 88 (1975) 191.

Bis(1,3-dimethylindenyl)iron(III) hexafluorophosphate

($P\bar{1}$) $Z = 2$, $R = 4.38\%$ from 2136 independent reflections. The structure consists of discrete cationic and anionic units. The metal is coordinated to the five-membered rings of each indenyl ligand. It has roughly a staggered configuration with six-membered rings assuming positions differing by 93.5° rotation of one ring. Fe—centroid = 1.716 Å.

P.M. Treichel, J.W. Johnson and J.C. Calabrese, *J. Organometal. Chem.*, 88 (1975) 215.

Acetylcyclopentadienyl-manganese and -rhenium tricarbonyls, $CH_3COC_5H_4$-$Re(CO)_3$, A, $CH_3COC_5H_4Mn(CO)_3$, B.

A. ($P2_1/a$) $Z = 4$, $R = 8.9\%$ from 1480 non-zero independent reflections.
B. ($P2_1/c$) $Z = 4$, $R = 12.0\%$ from 627 non-zero independent reflections.
The orientation of the CO groups with respect to the cyclopentadienyl ring differs in A and B. A is less symmetrical.

T.L. Khotsyanova, S.I. Kuznetsov, E.V. Bryukhova and Yu.V. Makarov, *J. Organometal. Chem.*, 88 (1975) 351.

The new cluster dianion μ-iodotetra-μ_3-carbonyldi-μ-carbonyldecacarbonyl-polyhedro-heptarhodate(2—) in its tetraethylammonium salt, $[Rh_7(CO)_{16}I]^{2-}$

($P\bar{1}$) $Z = 2$, $R = 5.6\%$ from 2532 reflections. The metal atom cluster consists of a monocapped octahedron. Ten of the CO ligands are coordinated terminally, two edge-bridging and four face-bridging; the iodine atom is in an edge-bridging position.

V.G. Albano, G. Ciani, S. Martinengo, P. Chini and G. Giordano, *J. Organometal. Chem.*, 88 (1975) 381.

Tetrakis(dimethylphenylphosphine)molybdenum(0): $Mo(PMe_2Ph)_3$-(η-$PhMe_3$)

($P\bar{1}$) $Z = 2$, $R = 6.8\%$ from 1203 independent reflections. The complex is shown to be an eighteen electron complex, mean Mo—P and Mo—C = 2.44 and 2.88 Å respectively.

R. Mason, K.M. Thomas and G.A. Heath, *J. Organometal. Chem.*, 90 (1975) 195.

μ-(1,2,6-η:3-5-η-Bicyclo[6.10]nona-1,3,5-triene)hexacarbonyldiiron (Fe—Fe), $(C_9H_{10})Fe_2(CO)_6$

($P\bar{1}$) $Z = 2$, $R = 3.4\%$ from 2651 reflections. Contrary to previous claims, the structure of the molecule is of the asymmetric type already established for $(C_8H_{10})Fe_2(CO)_6$ and $(C_{10}H_{12})Fe_2(CO)_6$. The most identical bond parameters amongst the three structures are noted. ^1H NMR down to —127° is also discussed.

J. Takats, *J. Organometal. Chem.*, 90 (1975) 211.

Dichlorodimethyltin(IV)1/1 salicylaldehyde adduct; structure and Mössbauer parameters, $Sn^{IV}Cl_2(CH_3)_2$.salH

($P2_12_12_1$) $Z = 4$, $R = 4.7\%$ from 981 independent reflections. The tin atom is five-coordinate with a very distorted trigonal bipyramidal geometry. Cl^- is axial, Sn—Cl(axial) = 2.403(6), and the oxygen of the monodentate, salH, forms the second axial bond, Sn—O = 2.680(13), Sn—C = 2.098, 2.107 Å.

D. Cunningham, I. Douek, M.J. Fraser, M. McPartlin and J.D. Matthews, *J. Organometal. Chem.*, 90 (1975) C23.

μ-Diiminobis(pentacarbonylchromium), $N_2H_2[Cr(CO)_5]_2$.2THF at —40°C

($P\bar{1}$) $Z = 2$, $R = 6.8\%$ from 1310 reflections. Two pentacarbonylchromium moieties are bridged by a diimino ligand, and each proton of the diimino ligand forms a hydrogen bridge with a THF oxygen atom. The diimino ligand has *trans* configuration because of crystallographic symmetry, $N—N_{(mean)} = 125$ pm, $Cr—N_{(mean)} = 208$ pm.

G. Huttner, W. Gartzke and K. Allinger, *J. Organometal. Chem.*, 91 (1975) 47.

Cyclic polysilanes, VIII. Structure of 1,2,3,4-tetra-*tert*-butyltetramethylcyclotetrasilane, $[Si(CH_3)(t\text{-}C_4H_9)]_4$

($P4_2/n$) $Z = 2$, $R = 3.5\%$ from 745 independent reflections. Each tetrameric molecule was found to be arranged about a $\bar{4}$ axis. The four membered ring of silicon atoms is non-planar with an unusually large dihedral angle of 36.8°. Mean bond lengths are Si—Si = 2.377(1), Si—C(CH_3) = 1.893(4), Si—C(t-C_4H_9) = 1.918(3) Å.

C.J. Hurt, J.C. Calabrese and R. West, *J. Organometal. Chem.*, 91 (1975) 273.

Hexamethylcyclotristannathiane, $[(CH_3)_2SnS]_3$

($P4_1$) $Z = 4$, $R = 5.1\%$ from 1660 reflections. The six-membered ring $(SnS)_3$ has a twisted boat-conformation. The tin atoms are tetrahedrally surrounded by two carbon atoms (Sn—C = 2.16 Å) and two sulfur atoms (Sn—S = 2.41 Å). The existence of a second, unstable modification of $[(CH_3)_2SnS]_3$ was established.

B. Menzebach and P. Bleckmann, *J. Organometal. Chem.*, 91 (1975) 291.

The flexible geometry of the $[HW_2(CO)_{10}]^-$ anion, $[(Ph_3P)_2N]^+[HW_2(CO)_{10}]^-$

($P\bar{1}$) $Z = 2$, $R = 5.0\%$ from 3282 non-zero reflections. The $[HW_2(CO)_{10}]^-$ anion can exist in both linear and bent forms: in $[Et_4N]^+[HW_2(CO)_{10}]^-$ the anion adopts a linear D_{4h} structure with eclipsed equatorial carbonyl groups, but in $[(Ph_3P)_2N]^+[HW_2(CO)_{10}]^-$, the anion has a bent backbone and staggered equatorial carbonyl groups.

R.D. Wilson, S.A. Graham and R. Bau, *J. Organometal. Chem.*, 91 (1975) C49.

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

($C2/c$) $Z = 4$, $R = 20\%$ from 2968 independent reflections. (No absorption correction was applied.) The crystals contain discrete $H[Mn(CO)_5]_2Sn$—$Sn[Mn(CO)_5]_2H$ molecules. The tin atoms are in a strongly distorted tetrahedral environment, <Mn—Sn—Mn = 119.8°, <Mn—Sn—Sn = 197.7, 121.9°.

K.D. Bos, E.J. Bulten, J.G. Noltes and A.L. Spek, *J. Organometal. Chem.*, 92 (1975) 33.

$(\eta^5\text{-}C_5H_5)_2Co$

($P2_1/c$) $Z = 2$, $R = 7.6\%$ from 765 independent reflections. Both cp rings are parallel to each other and in a staggered conformation. C—C(avg) = 1.41(1), Co—C = 2.096(8) Å.

W. Bünder, and E. Weiss, *J. Organometal. Chem.*, 92 (1975) 65.

Conformations of fused cycloalkanes in organometallic complexes, IV. Structure of tricyclo[6.4.0.0^{2,7}]dodeca-3,5-dienetricarbonyliron

($P2_1$) $Z = 2$, $R = 5.5\%$ from 1733 reflections. The $Fe(CO)_3$ moiety is bound to the conjugated diene portion of the cyclohexadiene ring. The feature of greatest interest is the mutual influence of the conformations of the two fused cycloalkane rings, whose intrinsically preferred conformations are mutually incompatible.

F.A. Cotton, V.W. Day and K.I. Hardcastle, *J. Organometal. Chem.*, 92 (1975) 369.

Structural studies on iron carbonyl derivatives, II. Structure of a complex prepared by the reaction of $Fe_3(CO)_{12}$ and 2-mercaptopyridine, $C_5H_4N(CO)_6$-Fe_2—μ_4-S—$Fe_2(CO)_6SC_5H_4N$

($P2_1/n$) $Z = 4$, $R = 4.1\%$ from 2089 independent reflections. The molecule contains four $Fe(CO)_3$ groups with two iron—iron bonds bridged in a different way, and a sulphur atom tetrahedrally coordinated to the four iron atoms.
G. LeBorgne and D. Grandjean, *J. Organometal. Chem.*, 92 (1975) 381.

Organo—metal complexes, XI. Structure of bis(dipivaloylmethyl)mercury, $[Hg(DPM)_2]_2$
($P\bar{1}$) $Z = 2$, $R = 6.0\%$ from 2581 reflections. In the dimer, bis(dipivaloylmethyl)mercury, there are strong Hg—C (2.13 Å) and weak Hg—O (2.70 Å) bonds.
K. Dietrich, H. Masso and R. Allmann, *J. Organometal. Chem.*, 93 (1975) 15.

Possible mode of action of anti-tumour platinum drugs: X-ray evidence for *cis* binding by platinum of two inosine 5′-monophosphate molecules via the N(7) positions, $Na_2[Pt(NH_3)_2(5'\text{-IMP})_2].nH_2O$, $n \simeq 16$
($C222_1$) $Z = 4$, $R = 7.8\%$ from 2404 independent reflections. Structural evidence shows that platinum binds to the two nucleotide moieties via the N(7) positions on the bases. The implications of this finding are discussed in terms of possible modes of anti-tumour action of Pt compounds.
D.M.L. Goodgame, I. Jeeves, F.L. Phillips and A.C. Skapski, *Biochim. Biophys. Acta*, 378 (1975) 153.

Refinement of the structure of carp muscle calcium-binding parvalbumin
P.C. Moews and R.H. Kretsinger, *J. Mol. Biol.*, 91 (1975) 201.

Hemerythrin, $[HrFe_2^{III}(H_2O)_8]$
($P4$). Two molecules of the octamer per unit cell. Further work in progress.
J.S. Loehr, K.N. Meyerhoff, L.C. Sieker and L.H. Jensen, *J. Mol. Biol.*, 91 (1975) 521.

Pyroelectric α-$Cu(IO_3)_2$. Crystal structure of the transition metal iodates, III.
($P2_1$) $Z = 2$, $R = 3.5\%$ from 4272 reflections. The iodate ions form a nearly-regular hexagonal closest-packed array, with Cu^{2+} in the resulting octahedral interstices. I—O varies from 1.82 Å to 2.95 Å. The CuO_6 octahedron is tetragonally distorted. Cu—O(eq) = 1.97, Cu—O(ax) = 2.39 Å.
R. Liminga, S.C. Abrahams and J.L. Bernstein, *J. Chem. Phys.*, 62 (1975) 4388.

Potassium dithiooxalate, $K_2S_2C_2O_2$
($P2_12_12_1$) $Z = 4$, $R = 2.2\%$ from 941 reflections. The $S_2C_2O_2^{2-}$ ion has C_2-symmetry. The torsional angle is 75.5°, starting from the *trans* rotamer. C—C = 1.516(4), C—O = 1.227(4), 1.239(4), C—S = 1.712(3), 1.697(3) Å.
R. Mattes, W. Meschede and W. Stork, *Chem. Ber.*, 108 (1975) 1.

2,2-Dichloro-1,3,6,2-trithiastannaocane, $C_4H_8Cl_2S_3Sn$
(*Pbca*) $Z = 8$, $R = 2.4\%$. The eight-membered ring has the boat—chair conformation with 1,5-transannular Sn···S-interaction. The coordination of Sn is trigonal-bipyramidal; equatorial distances: Sn—S = 2.386, 2.388, Sn—Cl = 2.348, Axial, Sn—S = 2.760, Sn—Cl = 2.392 Å.
M. Dräger and R. Engler, *Chem. Ber.*, 108 (1975) 17.

The Cobalt-cluster $H_4(C_5H_5)_4Co_4$
(*C2/c*) $Z = 8$, $R = 5.9\%$. The compound contains a slightly distorted tetrahedron of cobalt atoms, each one coordinated to a planar π-bonded cyclopentadienyl ring. All four faces of the Co_4 cluster are bridged by H atoms. Co—Co(mean) = 246.7 pm.
G. Huttner and H. Lorenz, *Chem. Ber.*, 108 (1975) 983.

N-Nitrosodimethylamine, $(CH_3)_2NNO$ at —130°C
(*Pnma*) $Z = 4$, $R = 8.8\%$ from 370 reflections. Bond lengths in the exactly planar molecule are: N—N = 1.320(6), N—O = 1.260(6), C—N = 1.461(7), 1.465(7) Å. The bonding is significantly closer to the polar structure $(CH_3)_2N^+{=}N{-}O^-$ compared to the free molecule in the gas phase, this being stabilized by intermolecular π-interactions perpendicular to the planes of the molecules.
B. Krebs and J. Mandt, *Chem. Ber.*, 108 (1975) 1130.

Compounds with a covalent metal—metal bond, III. Structure of octacarbonylbis[μ-(pentacarbonylrhenium)indium(III)] dirhenium, $Re_2(CO)_8\{\mu\text{-}InRe(CO)_5\}_2$
(*P*2$_1$/*n*) $Z = 2$, $R = 4.8\%$ from 3301 reflections. The molecule contains a planar Re_2In_2 ring, Re—Re = 3.232(1) Å, <Re—In—Re = 71.07(3)°, consistent with the existence of a Re—Re bond. The Re atoms of $Re(CO)_5$ have a *trans*-configuration with respect to Re_2In_2 ring. Mean distances: In—Re = 2.766(1), Re—C = 1.98(2) Å.
H. Preut and H.-J. Haupt, *Chem. Ber.*, 108 (1975) 1447.

2,2-Dichloro-1,3,6,2-trithiagermocane, $C_4H_8Cl_2GeS_3$
(*Pbca*) $Z = 8$, $R = 5.2\%$ from 1771 reflections. The eight-membered ring has the boat-chair conformation with 1,5-transannular Ge····S interaction. Ge coordination is trigonal-bipyramidal, Ge—S = 2.191(3), Ge—Cl = 2.149(3), Ge—S(ax) = 3.005(3), Ge—Cl(ax) = 2.208(3) Å.
M. Dräger, *Chem. Ber.*, 108 (1975) 1723.

Bis(triphenylphosphin)iminium-γ-tetracarbonylpropyl iron, $[\{(C_6H_5)_3P\}_2N]$-$[Fe(CO)_4C_3H_7]$
(*C2/c*) $Z = 8$, $R = 7\%$. Within the trigonal bipyramidal coordination polyhedron of the $[Fe(CO)_4C_3H_7]$ anion, the propyl group occupies an axial position. $Fe{-}C_{propyl} = 220(2)$ pm, mean$Fe{-}C_{CO} = 175(2)$ pm.
G. Huttner and W. Gartzke, *Chem. Ber.*, 108 (1975) 1373.

Pentacarbonyl[ethoxy(phenylethinyl)carbene]chromium(0), $(CO)_5\{C_2H_5O(C_6H_5C{\equiv}C)C\}Cr$

($P2_1/c$) $Z = 4$, $R = 8.8\%$ from 795 non-zero reflections. $Cr—C_{carbene} =$ 200(2) pm. All atoms of the carbene ligand including those of the phenyl ring are coplanar within the limits of error.

G. Huttner and H. Lorenz, *Chem. Ber.*, 108 (1975) 1864.

Structure and absolute configuration of an optically active square pyramidal molybdenum complex, $(+)_{579}$-$[C_5H_5Mo—(CO)_2NN']^+PF_6^-$ where NN' = Schiff base derived from 2-pyridine carbaldehyde and S-(—)-α-phenylethylamine

($P2_12_12_1$) $Z = 4$, $R = 7.3\%$ from 2359 reflections. In the approximately square pyramidal cation, the center of the π-bonded cp ring occupies the apex of the pyramid. The Mo atom is about 0.5 Å above the nearly coplanar C1, C2, N1, N2 atoms. Mo—cp = 2.265—2.369 Å.

S.J. LaPlaca, I. Bernal, H. Brunner and W.A. Herrmann, *Angew. Chem. Int. Ed. Engl.*, 87 (1975) 379.

Bis(1,3-butadiene)monocarbonylmanganese, $(C_4H_6)_2MnCO$

($P\bar{4}2_1m$) $Z = 2$, $R = 7.7\%$. The butadiene ligands are strictly planar (crystallographic C_{2v} symmetry imposed). Their "opening" points in the direction of the terminal CO group, their terminal carbon atoms form the base of an almost square pyramid whose apex is occupied by the carbonyl group. $Mn—C_{(terminal)} =$ 215(1) pm, Mn—CO = 184(1) pm, $Mn—C_{(central)} =$ 206(1) pm.

G. Huttner, D. Neugebauer and A. Razavi, *Angew. Chem. Int. Ed. Engl.*, 87 (1975) 353.

Thallium germanium sulphide, Tl_2GeS_3

($P\bar{1}$) $Z = 4$, Powder data are given.

T.J. Isaacs, *J. Appl. Crystallogr.*, 8 (1975) 391.

α-Barium 12-tungstosilicate, α-$Ba_2SiW_{12}O_{40}·16H_2O$

($C2/c$) $Z = 4$, $R = 13\%$ from 2952 relfections. The crystal consists of the Keggin ion, $SiW_{12}O_{40}^{4-}$, having a SiO_4 tetrahedron in the center, bound by Ba^{2+} and water molecules to form a three dimensional framework.

A. Kobayashi and Y. Sasaki, *Bull. Chem. Soc. Jap.*, 48 (1975) 885.

Polyvanadotungstates, I. α-$(CN_3H_6)_4V_2W_4O_{19}$

($P2_1$) $Z = 2$, $R = 8.6\%$ from 2362 reflections. The $V_2W_4O_{19}^{4-}$ anion has the hexaniobate-type structure consisting of 6-edge-shared MO_6 octahedra. Each M position is randomly occupied by V and W atoms with a 2:4 probability.

K. Nishikawa, A. Kobayashi and Y. Sasaki, *Bull. Chem. Soc. Jap.*, 48 (1975) 889.

Sodium molybdate dihydrate, $Na_2MoO_4.2H_2O$

(*Pbca*) $Z = 8$, $R = 3.7\%$ from 1497 reflections. The crystals are composed of alternate layers of MoO_4^{2-} tetrahedra and water molecules, interlinked by sodium cations and hydrogen bonds.

K. Matsumoto, A. Kobayashi and Y. Sasaki, *Bull. Chem. Soc. Jap.*, 48 (1975) 1009.

$(\pi\text{-}C_5H_5)Fe_3(CO_7)C_2C_6H_5$ obtained from $(\pi\text{-}C_5H_5)Fe(CO)_2C{\equiv}CC_6H_5$ with $Fe_2(CO)_9$

($P\bar{1}$) $Z = 2$, $R = 3.5\%$ from 2488 reflections. The structure consists of an isosceles triangle of the three iron atoms, each two of which bonds to three terminal carbonyl groups and one bonds to the cp ring. The phenethynyl group is above the iron plane and is σ-bonded to one iron atom with three carbonyls and π-bonded symmetrically to the other two iron atoms. The characteristic shortening in the Fe—C σ-bond and in the C≡C bond was observed. $Fe\text{—}C_\sigma = 1.829(6)$, $C\text{—}C = 1.299(9)$ Å.

K. Yasufuku, K. Aoki and H. Yamazaki, *Bull. Chem. Soc. Jap.*, 48 (1975) 1616.