

X-RAY BIBLIOGRAPHY

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(Received June 8th, 1973)

Chromium (III) tris(diethyldithiophosphate), $\text{Cr}[\text{S}_2\text{P}(\text{OC}_2\text{H}_5)_2]_3$

($C2/c$) $Z = 4$, $R = 8.1\%$ for 1368 reflections. The chromium atoms are surrounded by six sulphur atoms in a slightly flattened octahedron, the angles at chromium in the Cr-S-P-S ring being only 82.3° . Cr-S 2.421(3)-2.430(3), S-P(average) 1.99 Å. No indication of different S-P bond distances is found.

H. Vincents F. Schousboe-Jenson and R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1375.

Tin (II) bromide hydrate, $2\text{SnBr}_2 \cdot \text{H}_2\text{O}$

($P2_1/m$) $Z = 6$, $R = 15.7\%$. Three different types of tin coordination occur in the crystal, namely Sn-8Br, Sn-7Br + 1H₂O and Sn-7Br. The coordination figure is essentially the same for all three types and consists of a trigonal prism of bromine atoms with one or two additional atoms (Br, H₂O) outside prism faces. Sn-Br 2.75-3.70 and Sn-O 2.28 Å.

J. Andersson, *Acta Chem. Scand.*, 26 (1972) 1730.

Di- μ -bromo- μ -1, 2-cyclo-hexylenetetra-bromoditellurium, $\text{Te}_2\text{Br}_6\text{C}_6\text{H}_{10}$

($Pnma$) $Z = 4$, $R = 5.9\%$ for 574 reflections. The tellurium atoms are surrounded by 4 bromine atoms in a plane and by one carbon atom at the apex of the pyramid. The two tellurium atoms are linked by two bromine atoms and the cyclohexane group. Te-Br 2.880(6) for bridging and 2.542(6) for terminal bonds.

H.C. Hazell, *Acta Chem. Scand.*, 26 (1972) 1510.

Cis (A) and *trans* (B) nickel (II) dithiosemicarbazide dinitrate, $\text{Ni}(\text{CH}_5\text{N}_3\text{S})(\text{NO}_3)_2$

($P2_1/a$) $Z = 4$, $R = 7.3\%$ for 818 reflections (A); ($P1$) $Z = 1$, $R = 5.6\%$ for 1061 reflections (B). The red compound is *cis* and the red-green dichroic compound is *trans*. The Ni-S bonds in (A) are shorter than in (B).

R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1365.

Pentathiazyl tetrachloroaluminate, $\text{S}_5\text{N}_5\text{AlCl}_4$

($Pnma$) $Z = 8$, $R = 8.7\%$ for 1523 reflections. The compound consists of discrete S_5N_5 heart-shaped, almost planar cations and AlCl_4^- anions. S-N 1.465-1.590 Å.

Al-Cl(mean) 2.117(7) Å.

A.C. Hazell and R.G. Hazell, *Acta Chem. Scand.*, 26 (1972) 1987.

Mercury (II) chromate hemihydrate, $\text{HgCrO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$

($C2/c$) $Z = 8$, $R = 10.1\%$ for 1702 independent reflections. Every mercury atom is bonded to two oxygen atoms, each belonging to a chromate tetrahedra at the distances 2.05(2) and 2.06(2) Å. O—Hg—O is 176.2(9)°. The chromate tetrahedron is slightly distorted with Cr—O distances elongated where the oxygen is bonded to mercury.

K. Aurivillius, *Acta Chem. Scand.*, 26 (1972) 2113.

NaHoO₄(GeO₄)₂O₂OH

($Pnma$) $Z = 4$, $R = 8.5\%$ for 934 independent reflections. The holmium atoms are seven-coordinated with oxygen atoms. The GeO₄ tetrahedra are fairly regular and are linked to the holmium and sodium coordination polyhedra.

A.N. Christensen, *Acta Chem. Scand.*, 26 (1972) 1955.

A tin (II) bromide hydrate, $6\text{SnBr}_2 \cdot \text{SH}_2\text{O}$

($P2_1/m$) $Z = 2$. All tin atoms are situated within a trigonal prism of bromine atoms with additional bromines or waters capping prism faces so that the tin coordination is sevenfold or eightfold. Sn—Br 2.78–3.79; Sn—O 2.18–2.33 Å.

J. Andersson, *Acta Chem. Scand.*, 26 (1972) 2543.

Cobalt (II) (–) –malate trihydrate, $\text{Co}(\text{OOC}-\text{CH}_2-\text{CHOH}-\text{COO}) \cdot 3\text{H}_2\text{O}$

($P2_1$) $Z = 2$, $R = 2.8\%$ for 996 independent reflections. Cobalt is octahedrally coordinated to six oxygens and the octahedra are bridged by O—C—O atoms forming an infinite spiral of malate groups. Co—O 2.07–2.09 Å and Co—O 2.14(3) Å.

L. Kryger and S.E. Rasmussen, *Acta Chem. Scand.*, 26 (1972) 2349.

Cadmium (II) hexacyano-chromate (III), $\text{Cd}_3[\text{Cr}(\text{CN})_6]_2 \cdot x\text{H}_2\text{O}$

($F432$), ($F\bar{4}3m$), or ($Fm\bar{3}m$) $Z = \frac{4}{3}$, $R = 3.5\%$ for 90 independent reflections. The structure is closely related to the other Prussian Blue analogues.

H.V. Güdel, *Acta Chem. Scand.*, 26 (1972) 2169.

Orthorhombic (A) and monoclinic (B) forms of di-μ-bromo-bis{diethylenethioureate-tellurium (II)} dibromide, $\text{Te}(\text{etu})_2\text{Br}_2$

($Fddd$) $Z = 4$, $R = 4.3\%$ (A) and ($C2/c$) $Z = 4$, $R = 3.2\%$ (B). In both cases, two bromine atoms are coordinated to tellurium in a distorted square planar *cis* arrangement. The bromine atoms are situated on twofold axes and bridge two tellurium atoms, resulting in a dinuclear cation.

P. Herland, M. Lunderland and K. Marøy, *Acta Chem. Scand.*, 26 (1972) 2567.

 Cu_3P

($P6_3cm$) $Z = 6$, $R = 4.8\%$ for 333 reflections. The structure can be described as a complex array of interconnected PCu_6 triangular prisms with one additional copper atom outside each of the rectangular and trigonal faces of the prisms.

O. Olofsson, *Acta Chem. Scand.*, 26 (1972) 2777.

Dioxomolybdenum (VI) diethyldithiocarbamate, $\text{MoO}_2[(\text{C}_2\text{H}_5)_2\text{NCS}_2]_2$
 ($P2_1/c$) $Z = 4$, $R = 12.9\%$ for 551 independent reflections. The coordination around molybdenum is a distorted octahedron with four sulphur and two oxygen atoms, Mo—S 2.44(1), 2.63(1); Mo—O 1.63(3) Å. The NCS_2 group is planar.
 A. Kopwille, *Acta Chem. Scand.*, 26 (1972) 2941.

Nb_2WO_8
 ($Pbcm$) $Z = 8$, $R = 6.5\%$. The oxygen network is constructed of building units each consisting of one pentagonal MO_7 bipyramid linked by equatorial edge-sharing to five MO_6 -octahedra. Such units are further connected by corner sharing.
 M. Lundberg, *Acta Chem. Scand.*, 26 (1972) 2932.

Thallium (I) diisopropyldithiocarbamate, $\text{Tl}^+\text{S}_2\text{CN}(\text{C}_3\text{H}_7)_2$
 ($P2_1/n$) $Z = 4$, $R = 11.2\%$ for 732 independent reflections. The dimers are packed in layers. The thallium-sulphur coordination is fivefold with sulphurs located approximately at the corners of a tetragonal pyramid.
 P. Jennische, A. Olin and R. Hesse, *Acta Chem. Scand.*, 26 (1972) 2799.

$\beta\text{-}[\text{Pb}_6\text{O}(\text{OH})_6](\text{ClO}_4)_4 \cdot \text{H}_2\text{O}$
 ($Pbca$) $Z = 8$, $R = 7.1\%$ for 2713 reflections. Discrete $[\text{Pb}_6\text{O}(\text{OH})_6]^{4+}$ groups are present in the structure. The lead atoms occupy the corners of three distorted tetrahedra connected by common faces. The central tetrahedron contains an oxygen atom at its centre. The outer tetrahedra have one hydroxide oxygen outside each of their six unshared faces.
 A. Olin and R. Söderquist, *Acta Chem. Scand.*, 26 (1972) 3505.

A tin (II) bromide hydrate, $3\text{SnBr}_2 \cdot \text{H}_2\text{O}$
 ($P2_1$) or ($P2_1/m$) $Z = 4$. The tin atom sits in a trigonal prism of bromine atoms with bromine and water, capping prism faces. Sn—Br 2.77–3.68 Å, Sn—O 2.35 Å.
 J. Anderssen, *Acta Chem. Scand.*, 26 (1972) 3813.

Tris (diethyldithiocarbamate) phenyltellurium (IV), $[\text{Te}(\text{Et}_2\text{NCS}_2)_3\text{C}_6\text{H}_5]$
 ($P2_1/c$) $Z = 4$, $R = 6.2\%$ for 4183 reflections. The central tellurium atom is bonded to all six sulphur atoms in the molecule and to a carbon in the phenyl group in a distorted pentagonal bipyramidal configuration. The axial Te—C length is 2.124 (11) Å, Te—S(axial) is 3.228(4). The Te—C bond is nearly normal to the equatorial TeS_5 plane but the angle between the two axial bonds is $144.6(2)^\circ$. The five equatorial and nearly coplanar Te—S bonds are 2.606–2.816(3) Å.
 S. Esperas and S. Husebye, *Acta Chem. Scand.*, 26 (1972) 3293.

$\text{Na}_2[\text{Fe}(\text{CN})_5\text{NH}_3]2\text{H}_2\text{O}$
 ($Pnmm$) $Z = 4$, $R = 8.6\%$. The $[\text{Fe}(\text{CN})_5\text{NH}_3]^{2-}$ has approximate C_{4v} symmetry and Fe—C—N bonds are almost linear. The cyanide group *trans* to the ammonia molecule appears to be more firmly bonded than the rest of the ligands.
 A. Tullberg and N. Vannerberg, *Acta Chem. Scand.*, 26 (1972) 3382.

An imidazolato-bridged polynuclear copper (II) imidazole chloride complex,

$\text{Cu}(\text{C}_3\text{H}_3\text{N}_2)(\text{C}_3\text{H}_4\text{N}_2)\text{Cl}$

($P2_1/mn$) $Z = 2$, $R = 5.0\%$ for 1101 independent reflections. The imidazolato ring is lying in a mirror plane with both nitrogens bonded to equivalent copper atoms in the same plane, forming a bridge between copper atoms. The two imidazole rings are on either side of the mirror plane coordinated to copper through nitrogen atoms. The copper-chlorine bond is in the mirror plane. $\text{Cu}-\text{Cl}$ 2.559(2), $\text{Cu}-\text{N}(\text{C}_3\text{H}_3\text{N}_2^-)$ 1.986(5), $\text{Cu}-\text{N}(\text{C}_3\text{H}_4\text{N}_2)$ 2.058(4) Å.

B.K.S. Lundberg, *Acta Chem. Scand.*, 26 (1972) 3903.

Tetrakisimidazole Cu^{II} sulphate, $\text{Cu}(\text{C}_3\text{H}_4\text{N}_2)_4\text{SO}_4$

($C2/c$) $Z = 4$, $R = 5.0\%$ for 2026 independent reflections. The coordination around copper is a distorted octahedron and the ligand atoms are four imidazole nitrogens and two sulphate oxygens. $\text{Cu}(\text{C}_3\text{H}_4\text{N}_2)_4^{2+}$ are linked together by SO_4^{2-} ions. $\text{Cu}-\text{N}$ 2.000 and 2.021; $\text{Cu}-\text{O}$ 2.574 Å.

G. Fransson and B.K.S. Lundberg, *Acta Chem. Scand.*, 26 (1972) 3969.

A redetermination of $\text{K}_2\text{TeO}_3(\text{OH})$

($P2_1/a$) $Z = 4$, $R = 4.4\%$ for 3706 reflections. The structure contains $\text{Te}-\text{O}_6$ octahedra which share edges, forming infinite chains. Apart from the electrostatic forces due to the potassium ions, the $[\text{TeO}_3(\text{OH})]_n^{n-1}$ chains are held together by strong hydrogen bonds with distances of 2.480 Å.

O. Lindqvist, *Acta Chem. Scand.*, 26 (1972) 4107.

(Glycyl-L-histidylglycinato)-copper (II) sodium perchlorate hydrate

($P2_12_12_1$) $Z = 4$, $R = 7.5\%$ for 1449 independent reflections. Each tripeptide bonds one copper atom at its amino, peptide and imidazole nitrogen atoms, and a second copper atom at the carbonyl atom. $\text{Cu}-\text{N} = 2.01, 1.93$ and 1.93 ; $\text{Cu}-\text{O} = 2.03$ Å. The second carbonyl oxygen and a water interact weakly with the copper atom at 2.76 and 2.74 Å.

R. Österberg, B. Sjöberg and R. Söderqvist, *Acta Chem. Scand.*, 26 (1972) 4185.

Diimidazole cobalt (II) dichloride, $\text{Co}(\text{C}_3\text{H}_4\text{N}_2)_2\text{Cl}_2$

($P2_1/c$) $Z = 4$, $R = 12.4\%$ for 1204 independent reflections. The coordination about cobalt is tetrahedral. $\text{Co}-\text{N}$ 1.989 and 1.997; $\text{Co}-\text{Cl}$ 2.264 and 2.237 Å.

C. Antti and B.K.S. Lundberg, *Acta Chem. Scand.*, 26 (1972) 3995.

$\text{M}-\text{SbOF}$

($Pbca$) $Z = 16$, $R = 5.3\%$ for 709 reflections. Three oxygen and one fluorine atom are bonded to Sb^{3+} , all to one side, and such polyhedra form layers parallel to the ab plane by corner and edge sharing.

A. Aström, *Acta Chem. Scand.*, 26 (1972) 3849.

Diimidazole-copper (II) dichloride, $\text{Cu}(\text{C}_3\text{H}_4\text{N}_2)_2\text{Cl}_2$

($Pbn2_1$) $Z = 4$, $R = 6.9\%$ for 729 independent reflections. The copper atom coordinates five ligand atoms in a tetragonal pyramidal configuration. The four closest atoms, lying approximately at the corners of a square plane, are two nitrogens $\text{Cu}-\text{N}$ 1.97 and 1.99, and two chlorine atoms $\text{Cu}-\text{Cl}$ 2.32 and 2.37 Å. An apical chlorine is 2.75 Å from the copper.

B.K.S. Lundberg, *Acta Chem. Scand.*, 26 (1972) 3977.

 $\text{Na}_2[\text{Hg}(\text{SO}_3)_2]\text{H}_2\text{O}$

($P2_1/n$) $Z = 4$, $R = 6.4\%$ for 1355 independent reflections. $\text{Hg}-\text{S}$ 2.402(6) and 2.411(6) Å; $\text{S}-\text{Hg}-\text{S}$ 172.8(2)°. The structure consists of discrete $\text{Hg}(\text{SO}_3)_2^{2-}$ groups held together by sodium ions. Two independent distorted NaO_6 octahedra exist; the oxygens are from five different sulphite groups and one water.

B. Nyberg and I. Cynkier, *Acta Chem. Scand.*, 26 (1972) 4176.

Gold (I) dipropylthiocarbamate dimer, $(\text{C}_3\text{H}_7)_2\text{NCS}_2\text{Au}$

($P4_22$) $Z = 4$, $R = 10.5\%$ for 274 independent reflections. The structure consists of isolated $[(\text{C}_3\text{H}_7)_2\text{NCS}_2\text{Au}]_2$ dimers. $\text{Au}-\text{Au}$ is 2.76 Å. Each gold is linked to two sulphur atoms in two different ligands by linear coordination. The molecules are packed so as to form linear gold chains with alternating distances 2.76 and 3.40 Å.

R. Hesse and P. Jennische, *Acta Chem. Scand.*, 26 (1972) 3855.

 $\mu\text{-Chloro-}\mu\text{-hydrido-bis[chloro-(pentamethylcyclopentadienyl)rhodium(III)]}$, $[\pi\text{C}_5(\text{CH}_3)_5\text{RhCl}]_2\text{HCl}$

($Pbcn$) $Z = 4$, $R = 5.78\%$ for 1526 independent reflections. The molecule has precise C_2 symmetry with planar $\text{Rh}(\text{H})(\text{Cl})$ Rh bridge. $\text{Rh} \cdots \text{Rh}$ 2.906(1) $\text{Rh}-\text{Cl}(\text{bridging})$, 2.437(2); $\text{Rh}-\text{Cl}(\text{terminal})$ 2.393(2); $\text{Rh}-\text{H}(\text{bridging})$ 1.85(5) Å.

M.R. Churchill and S.W. Ni, *J. Amer. Chem. Soc.*, 95 (1973) 2150.

Triindenylsamarium, $\text{Sm}(\text{C}_9\text{H}_7)_3$

($Pbca$) $Z = 8$, $R = 7.0\%$ for 1791 reflections. In the monomeric molecule the samarium atom is symmetrically bonded to the five-membered portion of the indenyl system with an average $\text{Sm}-\text{C}$ bond distance of 2.75 Å. There is no evidence for preferential bonding of the samarium atom to the electron-rich C-1 position on the indenyl groups.

J.L. Atwood, J.H. Burns and P.G. Laubereau, *J. Amer. Chem. Soc.*, 95 (1973) 1830.

Tricyclopentadienylscandium, $\text{Sc}(\text{C}_5\text{H}_5)_3$

($Pbc2_1$) $Z = 4$, $R = 4.1\%$ for 1013 observed reflections. The structure consists of $\text{Sc}(\text{C}_5\text{H}_5)_2$ units bridged together by the remaining cyclopentadienyl groups. Two rings are coordinated to scandium in a pentahapto fashion and two rings are associated through essentially only one carbon atom.

J.L. Atwood and K.D. Smith, *J. Amer. Chem. Soc.*, 95 (1973) 1488.

$[(\text{Me}_3\text{Si})_2\text{Fe}(\text{CO})_4]_2$, reformulated as $[(\text{Me}_3\text{SiOC})_4\text{Fe}_2(\text{CO})_6]$

(*P1*) $Z = 2$, $R = 8.5\%$ for 1486 reflections. The molecule contains a substituted butadiene fragment bonded to one $\text{Fe}(\text{CO})_3$ group with two σ bonds to form an iron-containing heterocyclic diene. This five-membered ring is π -bonded to a second $\text{Fe}(\text{CO})_3$ group and an iron-iron bond (2.50 Å) completes the linkage between two non-equivalent $\text{Fe}(\text{CO})_3$ groups.

M.J. Bennett, W.A.G. Graham, R.A. Smith and R.P. Stewart, Jr., *J. Amer. Chem. Soc.*, 95 (1973) 1684.

Tris (*N,N*-di-*n*-butyldithiocarbamato) nickel (IV) bromide, $\text{NiC}_{27}\text{H}_{54}\text{N}_3\text{S}_6\text{Br}$

(*P31C*) $Z = 2$, $R = 3.8\%$ for 736 reflections. The crystal consists of bromine atoms arranged in a trigonal antiprismatic manner about columns composed of discrete, approximately D_3 symmetry, units of $\text{Ni}(\text{n-Bu}_2\text{Dt})_3^+$. The NiS_6 core is twisted by 45.4° from a trigonal prismatic arrangement. Ni-S 2.261, S-C 1.708, C-N 1.318 Å. The nickel atoms along the columns are separated by 5.384 Å.

J.P. Fackler Jr., A. Ardeef and R.G. Fischer, Jr., *J. Amer. Chem. Soc.*, 95 (1973) 774.

$\text{SbCl}_5 \cdot \text{CH}_3 \cdot 4\text{-C}_6\text{H}_4\text{COCl}$ (A) and $[\text{SbCl}_6]^- [\text{CH}_3 \cdot 4\text{-C}_6\text{H}_4\text{CO}]^+$ (B)

(*Pbcm*) $Z = 4$, $R = 3.9\%$ for 1063 reflections (A) and

(*P2₁/C*) $Z = 4$, $R = 4.2\%$ for 2586 reflections (B). Complex (A) is composed of an SbCl_5 group coordinated with the oxygen atom of the *p*-toluoyl chloride group, Sb-O 2.253 Å. Distortions of the SbCl_5O octahedron have been interpreted as arising from intramolecular steric effects. The ionic salt (B) consists of discrete hexachloroantimonate anions and methyl-4-phenyloxocarbenium cations. The closest interionic distances are found between the chlorine atoms of the SbCl_6^- anions and carbonyl carbon of the cations.

B. Chevrier, J. LeCarpentier and R. Weiss, *J. Amer. Chem. Soc.*, 94 (1972) 5718.

$[(\text{CH}_3)_2\text{PC}_6\text{H}_5]_2\text{PtB}_3\text{H}_7$

(*C2/c*) $Z = 8$, $R = 5.4\%$ for 2950 reflections. The crystal consists of discrete molecules with two phosphine ligands and a π -bonded B_3H_7^- ligand coordinated to an essentially square planar platinum atom. The dihedral angle between the B_3 plane and the platinum phosphorus plane is $116.8(24)^\circ$. Pt-P 2.301(4) and 2.311(4) Å. The Pt-B distances are 2.38(4) to a boron atom in the PPtP plane and 2.18(4) and 2.13(3) Å to the boron atoms whose midpoint is in this plane. B-B 1.86(5) and 1.92(4) Å.

L.J. Guggenberger, A.R. Kane and E.L. Muetterties, *J. Amer. Chem. Soc.*, 94 (1972) 5665.

Dicaesium- μ -oxo-decafluorodiantimonate, $\text{Cs}_2 [\text{Sb}_2\text{F}_{10}\text{O}]$

(*P2/c*) $Z = 2$, $R = 8.8\%$ for 2232 reflections. The anion is of C_2 symmetry and consists of two octahedra coupled together by an Sb-O-Sb bridge. Sb-O 1.884(10) Å.

W. Haase, *Chem. Ber.*, 106 (1972) 41.

K[Pt((C₂H₅)₂C(OH)C≡CC(OH)(C₂H₅)₂)Cl₃]

(*P*2₁/*c*) *Z* = 4, *R* = 6.9% for 1662 independent reflections. The coordination around platinum is planar and the triple bond of the acetylenic ligand is approximately perpendicular to the coordination plane. C≡C is 1.18(3) Å and the C≡C—C angles (≈ 160°) show a deformation of the ligand in a direction away from the platinum atom with the two hydroxyl groups in *cis* configuration, above and below the plane of the PtCl₃ group.

A.L. Beauchamp, F.D. Rochon and T. Theophanides, *Can. J. Chem.*, 51 (1973) 126.

Hexaaquocobalt (II) — hexafluorosilicate (IV), [Co(H₂O)₆] [SiF₆]

(*R*3̄) *Z* = 3, *R* = 4.3% for 151 observed reflections. The structure consists of discrete Co(H₂O)₆ and [SiF₆]²⁻ octahedra joined systematically by O ··· H ··· F hydrogen bonds into chains parallel to the 3̄ axis.

H. Lynton and P.-Y. Siew, *Can. J. Chem.*, 51 (1973) 227.

LiVO₃, lithium metavanadate

(*C*2/*c*) *Z* = 8, *R* = 2.5% for 851 reflections. Bands of Li—O edge-shared octahedra run parallel to *C* and are linked by chains of VO₄ tetrahedra which also run parallel to *C*. Li—O are 2.153(5) and 2.284(3) and the difference is related to the degree of distortion of individual distances from the mean. V—O 1.821, 1.795, 1.662 and 1.628 Å.

R.D. Shannon and C. Calvo, *Can. J. Chem.*, 51 (1973) 265.

Di-μ-hydroxobis [di(1, 10-phenanthroline) chromium (III) chloride hexahydrate,

[Cr(phen)₂OH]₂Cl₄ · 6H₂O

(*P*1̄) *Z* = 2, *R* = 7.7% for 3392 independent reflections. The complex consists of pairs of chromium atoms which are linked by two hydroxo bridges, with 1, 10-phenanthroline groups coordinated to each metal. The coordination geometry around each chromium is roughly octahedral. The average Cr—N is 2.052(6) and Cr—O is 1.927(6) Å; Cr—Cr is 3.008(3) and Cr—O—Cr is 102.7(4)°.

J.T. Veal, W.E. Hatfield and D.J. Hodgson, *Acta Crystallogr., Sect. B*, 29 (1973) 12.

Lithium succinate, (CH₂ · COOLi)₂

(*R*3̄) *Z* = 9, *R* = 3.9% for 1049 independent reflections. The succinate ion has a centre of symmetry between the central C atoms. C—C distances are 1.525 and 1.518 Å. Four oxygen atoms form a slightly distorted tetrahedron around the Li ion with Li—O 1.943 to 1.96 Å.

H. Klapper and H. Kiippers, *Acta Crystallogr., Sect. B*, 29 (1973) 21.

Di-μ-(*N,N*-diethylnicotinamide-*O,N*-)diisothiocyanatomanganese (II),

Mn(C₁₀H₁₄N₂O)₂(NCS)₂

(*P*1̄) *Z* = 1, *R* = 4.2% for 3385 independent reflections. Two positions in the octahedron around manganese are occupied by amidic O atoms, two by pyridine N atoms and two by isothiocyanato N atoms. Pairs of *N,N*-diethylnicotinamide molecules bridge two metal atoms.

F. Bigoli, A. Braibanti, M.A. Pellinghelli and A. Tiripicchio, *Acta Crystallogr., Sect. B*, 29 (1973) 39.

CaHAsO₄ · 3H₂O

(*Pbca*) $Z = 8$, $R = 3.4\%$ for 1321 reflections. Isolated Ca octahedra are sandwiched between As tetrahedra. The crystal is isostructural with MgHPO₄ · 3H₂O (newberyite). M. Catti and G. Ferraris, *Acta Crystallogr., Sect. B*, 29 (1973) 90.

Carbonatotetrakis (pyridine) cobalt (III) perchlorate monohydrate,

(CoC₂₁H₂₀N₄O₇Cl · H₂O)

(*P2₁/c*) $Z = 4$, $R = 4.9\%$ for 3302 reflections. The cobalt atom is octahedrally surrounded by four nitrogen atoms of the pyridine molecules and two oxygen atoms of the bidentate carbonate group. The octahedron is distorted due to strain in the four-membered chelate ring and steric hindrance between the pyridine molecules. $\angle \text{O-Co-O}$ is 69.3°.

K. Kaas and A.M. Sorenson, *Acta Crystallogr., Sect. B*, 29 (1973) 113.

A hydrated calcium bromide salt of lactobionic acid, C₁₂H₂₁O₁₂CaBr · 4H₂O

(*P2₁2₁2₁*) $Z = 4$, $R = 5.8\%$. The calcium ion binds to three water molecules and to the gluconate moieties of two lactobionate ions. One gluconate residue is coordinated to the calcium ion through a carbonyl oxygen and through two hydroxyl oxygens located at the α and β positions adjacent to the carbonyl group. The second gluconate residue is coordinated through the oxygen atoms of terminal hydroxyl groups. The eight oxygen atoms in the calcium coordination shell assume a distorted square-antiprism arrangement.

W.J. Cook and C.E. Bugg, *Acta Crystallogr., Sect. B*, 29 (1973) 215.

[(C₂H₅)₃NH]₂CuCl₄

(*P2₁/c*) $Z = 4$, $R = 6.4\%$ for 1883 reflections. The structure of the CuCl₄²⁻ ion is that of a squashed tetrahedron with Cl-Cu-Cl angles 135° and 98°. There is a N-H ··· Cl hydrogen bond between the tetrachlorocuprate anion and the organic cation.

J. Lamotte-Brasseur, L. Dupont and O. Dideberg, *Acta Crystallogr., Sect. B*, 29 (1973) 241.

Bis(thiourea) lead (II) formate monohydrate, Pb(HCOO)₂ · [SC(NH₂)₂]₂ · H₂O

(*P $\bar{1}$*) $Z = 2$, $R = 15.2\%$ for 1686 reflections. The first coordination sphere of lead has three sulphur atoms and five oxygen atoms in a distorted dodecahedral arrangement. The sulphur atoms of the thiourea molecules are bicoordinate while bridging formate ions each have one monocoordinate and one bicoordinate oxygen. The remaining water molecule, thiourea molecule and formate ion of the formula unit are monocoordinated to lead. Two parallel S ··· Pb ··· S chains extend along [001] with each pair of centrosymmetrically related lead ions bridged by two similarly related formate ions.

I. Goldberg and F.H. Herbstein, *Acta Crystallogr., Sect. B*, 29 (1973) 246.

MV₃O₇ (M = Ca, Sr, Cd) structure of CaV₃O₇

(*Pnam*) The layer structure is built up from subunits of three square pyramids VO₅, sharing edges. The stereochemistry of vanadium (IV) is discussed.

J.-C. Bouloux and J. Galy, *Acta Crystallogr., Sect. B*, 29 (1973) 269.

Bissalicylidene-1,5-diamino-3-azapentane dioxouranium (VI)

(*Pnn̄a*) $Z = 4$, $R = 6.9\%$ for 716 unique reflections. The ligand is quinquedentate and gives rise to an approximately pentagonal bipyramidal coordination. The uranyl group which defines the axis of the bipyramid is linear and is perpendicular to the least squares plane through the five equatorial atoms which form a slightly puckered pentagon.

M.N. Akhtar and A.J. Smith, *Acta Crystallogr., Sect. B*, 29 (1973) 275.

Bis(isocyanurato) diamminocopper (II), $[\text{Cu}(\text{C}_3\text{N}_3\text{O}_3\text{H}_2)_2(\text{NH}_3)_2]$

(*P̄1*) $Z = 2$, $R = 10\%$ for 837 reflections. The complex consists of ammine groups and isocyanuric acid rings, bonded through nitrogen to copper atoms, forming square planar arrangements cross linked by hydrogen bonds. The ammonia groups lie approximately 13.5° away from the normals to the rings.

P.G. Slade, M. Raupack and E.W. Radoslovich, *Acta Crystallogr., Sect. B*, 29 (1973) 279.

Potassium "cryptate", $(\text{C}_{18}\text{H}_{36}\text{N}_2\text{O}_6\text{K})^+\text{I}^-$

(*C2/c*) $Z = 4$, $R = 4.6\%$ for 1249 observed reflections. The complex cation has D_3 symmetry. Potassium is located at the centre of the molecular cavity of the bicyclic ligand and is surrounded by all the eight heteroatoms. Metal anion distances are all greater than 6.5 Å.

D. Moras, B. Metz and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 383.

Rubidium and caesium "cryptates"

The crystals of these compounds are isomorphous; (*P21/C*) $Z = 4$, $R = 4.6\%$ for 1802 and $R = 4.1\%$ for 3797 independent reflections respectively. The structures are similar to that found for potassium cryptate and indicate the flexibility of the organic bicyclic ligand which adapts its molecular cavity to the ionic radius of the metal.

D. Moras, B. Metz and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 388.

Sodium cryptate, $\text{C}_{18}\text{H}_{36}\text{N}_2\text{O}_6 \cdot \text{NaI}$

(*P31c*) $Z = 2$, $R = 7.0\%$ for 795 reflections. The crystals are twinned and contain two molecular species in the trigonal cell. Ionic packing of complex cations and I^- anions is discussed.

D. Moras and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 396.

Lithium cryptate, $\text{C}_{14}\text{H}_{28}\text{N}_2\text{O}_4 \cdot \text{LiI}$

(*P4_2, 2_1*) $Z = 4$, $R = 4.6\%$ for 1251 reflections. The Li^+ cation is enclosed within the molecular cage. The mean bond distances are equal to 2.28 for $\text{Li}-\text{N}$ and 2.13 Å for $\text{Li}-\text{O}$.

D. Moras and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 400.

Caesium nonafluorodiuranate (IV), CsU_2F_9

(*C2/c*) $Z = 4$, $R = 4.8\%$ for 1075 reflections. Uranium is coordinated to eight fluorines in polyhedra which share edges to form $(\text{U}_4\text{F}_{16})$ sheets parallel to (100). The full uranium coordination approximates a tricapped trigonal prism, the ninth corner of the poly-

hedron being a half-filled eightfold set of fluorines so that the effective coordination number is 8.5. U—F are 2.28–2.42 with average 2.33 Å.

A. Rosanzweig, R.R. Ryan and D.T. Cromer, *Acta Crystallogr., Sect. B*, 29 (1973) 460.

(3*a*, 4–8, 9*a*–*n*)-(4, 5, 7-trimethyl-4*H*-cyclohepta[*b*]thiophene)-tricarbonylchromium (A) and (3*a*, 4–8, 8*a*–*n*)-(5, 7, 8-trimethyl-8*H*-cyclohepta[*b*]thiophene)-tricarbonylchromium (B)

($P2_1/c$) $Z = 8$, $R = 5.9\%$ for 975 independent reflections (A) and ($P2_1/c$) $Z = 4$, $R = 6.7\%$ for 836 independent reflections (B). In both cases the tricarbonyl chromium group is fixed on the cycloheptatriene ring. The conformation of the ring is different from that in the free ligand. The chromium–ring distances allow explanation of the results of the condensation reaction of hexacarbonyl chromium with thiophenotropilidene derivatives.

Y. Dusauroy, J. Protas and R. Guillard, *Acta Crystallogr., Sect. B*, 29 (1973) 477.

The $F = 122^\circ\text{C}$ form of 2,1'-trimethylene-1-[α -phenyl- α -hydroxypropyl]ferrocene, ($\text{FeC}_{22}\text{H}_{24}\text{O}$)

($P2_1/n$) $Z = 4$, $R = 7.1\%$ for 1212 independent reflections. The relative configuration of the compound with fusion point 122°C is (*R*)-2,1'-trimethylene-1-[(*S*)- α -phenyl- α -hydroxypropyl]ferrocene or (*S*)-2,1'-trimethylene-1-[(*R*)- α -phenyl- α -hydroxypropyl]ferrocene. The two five-membered rings are planar, the dihedral angle between the cyclopentadienyl rings being 10° . The mean bond distances are Fe–C 2.056, C–C 1.453 Å (in the cyclopentadienyl rings).

C. Lecomte, V. Disauroy, J. Protas, C. Moise and J. Tirouflet, *Acta Crystallogr., Sect. B*, 29 (1973) 488.

Thallous fluoride carbonate

($P2_1/m$) $Z = 2$, $R = 7.2\%$ for 611 reflections. In the ionic structure the three independent Tl^+ ions have almost identical environments, each with seven neighbours in capped octahedral geometry. There are three long Tl–O bonds (3.09 and 3.18 Å twice) to oxygens in the face of the octahedron and Tl–F 2.751 (twice) and Tl–O 2.85 in the opposite face. Tl–O(capping) is Tl–O 2.61 Å.

N.W. Alcock, *Acta Crystallogr., Sect. B*, 29 (1973) 498.

Copper (II) di- γ -aminobutyrate [$\text{Cu}(\text{NH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{COO})_2$], (Cu GABA-1) (A) and its dihydrate [$\text{Cu}(\text{NH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{COO})_2 \cdot 2\text{H}_2\text{O}$], (Cu GABA-2) (B)

Both are ($P2_1/c$) $Z = 2$; $R = 7.8\%$ and 7.7% for 1018 and 1275 reflections for (A) and (B) respectively. The structure of (A) consists of infinite one-dimensional chains in which two GABA ligands connect two copper (II) atoms by *trans* double bridging. In (B) there are infinite two-dimensional networks and the water molecule is not directly coordinated to copper but links neighbouring networks. The coordination about copper is quite similar in the two complexes, distorted octahedral.

A. Takenaka, E. Oshima, S. Yamada and T. Watanabe, *Acta Crystallogr., Sect. B*, 29 (1973) 503.

1, 7, 10, 16-Tetraoxa-4, 13-diazacyclooctadecane-dichlorocopper (II),



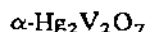
($P2_1/c$) $Z = 4$, $R = 4.4\%$ for 1569 observed reflections. The copper atom is situated in the cavity of the $\text{C}_{12}\text{H}_{26}\text{N}_2\text{O}_4$ ring, and its coordination polyhedron is a distorted octahedron with two chlorine, two nitrogen and two oxygen donor atoms. The conformation of the $\text{C}_{12}\text{H}_{26}\text{N}_2\text{O}_4$ ring has C_2 symmetry with the twofold axis almost coincident with the $\text{Cl}(1)-\text{Cu}-\text{Cl}(2)$ axis.

M. Herceg and R. Weiss, *Acta Crystallogr., Sect. B*, 29 (1973) 542.



($R\bar{3}$) $Z = 2$, $R = 4.5\%$ for 1092 observed reflections. Interatomic Pu-Pu are 3.44–3.97, Pu-Pd 2.89–3.30, Pd-Pd 2.87–3.69 Å. The structure consists of Pd atoms in special position sets 3(*a*) and 3(*b*); and Pu and Pd atoms in the general position set 18(*f*) of space group No. 148, ($R\bar{3}$). This is believed to be a new structure type.

D.T. Cromer, A.C. Larson and R.B. Roof, Jr., *Acta Crystallogr., Sect. B*, 29 (1973) 564.



($Pnma$) $Z = 4$, $R = 8.8\%$ for 642 reflections. The structure contains $[\text{VO}_3]$ infinite chains parallel to *b*. The mercury atoms link two chains by essentially ionic bonds, forming $[\text{Hg}(\text{VO}_3)_2]$ infinite units between which neutral $[\text{HgO}]$ infinite chains are inserted.

M. Quarton, J. Angenault and A. Rimsky, *Acta Crystallogr., Sect. B*, 29 (1973) 567.

Refinement of chalcopyrite, CuFeS_2

($I\bar{4}2d$) $Z = 4$, $R = 3.1\%$ for 694 reflections. Cu-S 2.302, Fe-S 2.257 are significantly closer than those previously reported.

S.R. Hall and J.M. Stewart, *Acta Crystallogr., Sect. B*, 29 (1973) 579.

Calcium chloride tetrahydrate

($P\bar{1}$) $Z = 2$, $R = 8.5\%$ for 1104 independent reflections. The two chloride ions and four water molecules form a trigonal prismatic coordination polyhedron about the calcium ion.

U. Thewalt and C.E. Bugg, *Acta Crystallogr., Sect. B*, 29 (1973) 615.

Silver sodium sulphate dihydrate, $\text{AgNaSO}_3 \cdot 2\text{H}_2\text{O}$

($P\bar{1}$) $Z = 2$, $R = 6.7\%$ for 727 observed reflections. The silver atom is nearly linearly coordinated by the sulphur atom and one of the sulphite oxygen atoms, resulting in a chain-like structure for AgSO_3^- . Ag-S 2.474(4), Ag-O 2.194(9) Å. Sodium is octahedrally coordinated by three sulphite oxygen atoms and three oxygens from water molecules. Na-O(average) is 2.418 Å.

L. Niinisto and L.O. Larson, *Acta Crystallogr., Sect. B*, 29 (1973) 623.

Indium oxyfluoride, InOF

(*Fddd*) $Z = 16$, $R = 5.9\%$ for 822 reflections. The indium atom has a distorted octahedral coordination with an ordered arrangement of oxygen and fluorine atoms. The In-(O, F) octahedra are joined together by edges and corners to form a loose three-dimensional network.

M. Vlasse, J.-C. Massies and B.L. Chamberland, *Acta Crystallogr., Sect. B*, 29 (1973) 627.

MgHAsO₄ · 7H₂O, roesslerite

(*C2/c*) $Z = 8$, $R = 3.0\%$ for 2157 non-zero reflections. The two crystallographically independent magnesium atoms are coordinated by six water molecules. The seventh water molecule is linked by hydrogen bonds only. No oxygen atoms are shared by coordination polyhedra and the crystal structure consists of layers parallel to [010] or [001].

G. Ferraris and M. Franchini-Angela, *Acta Crystallogr., Sect. B*, 29 (1973) 286.

Bis(pyrazine-2-carboxamide) copper (II) perchlorate, Cu(pyaH)₂(ClO₄)₂

(*P2₁/a*) $Z = 2$, $R = 8\%$ for 876 observed reflections. The complex is centrosymmetric with two ligand molecules chelating to the central copper atom in *trans* positions through the amide oxygen atoms and the *ortho* nitrogen atoms of the rings. This part has a square planar structure and the *meta* nitrogen atoms of the pyrazine ring of two neighbouring complex ions coordinate weakly to the central copper atom from above and below the coordination plane, thus completing octahedral coordination.

M. Sekizaki, *Acta Crystallogr., Sect. B*, 29 (1973) 327.

Samarium sulphide, SmS

(*R $\bar{3}m$*) $Z = 2$, $R = 7.9\%$ for 421 reflections. The structure is made up of sheets of sulphide (SmS)_n parallel to [001] consisting of almost regular SmS₄ tetrahedra. These sheets alternate with double layers of iodine atoms.

N. Savigny, P. Laruelle and J. Flahaut, *Acta Crystallogr., Sect. B*, 29 (1973) 345.

Er₃GaS₆

(*Cmc2₁*) $Z = 2$, $R = 7.9\%$ for 506 reflections. The erbium atoms have sevenfold coordination.

S. Jaulmes and P. Laruelle, *Acta Crystallogr., Sect. B*, 29 (1973) 352.

Bis(2, 4-dithiobiureto) nickel (II) glycol

(*P2₁/a*) $Z = 2$, $R = 6.0\%$ for 2011 reflections. The structure consists of centrosymmetric Ni(DTB)₂ molecules. The carbon and oxygen atoms of the glycol molecule are coplanar. The Ni(DTB)₂ unit has bond distances and angles very similar to those previously determined for the pure complex.

A. Pignedoli, G. Peyronel and L. Antolini, *Gazz. Chim. Ital.*, 102 (1972) 679.

Bis(hydrogen-pyridine-2, 6-dicarboxylato) copper (II) trihydrate,**Cu(C₇H₄NO₄)₂ · 3H₂O**

(*Pc*) $Z = 4$, $R = 7.7\%$ for 2315 independent reflections. There are two non-equivalent

complex molecules in the asymmetric unit. In both, the coordination is bipyramidally distorted octahedral as a consequence of the Jahn-Teller effect. Each copper is coordinated to two ligand molecules. One of them shows both carboxylic groups deprotonated while the other maintains its hydrogen atoms.

M.B. Cingi, A.C. Villa, C. Guastini and M. Nardelli, *Gazz. Chim. Ital.*, 102 (1972) 1026.

Bis(*N,O*-isonicotinato) monoquoalead (II), $[\text{Pb}(\text{C}_6\text{H}_4\text{NO}_2)_2(\text{OH}_2)]$

(*Cc*) $Z = 4$, $R = 5.0\%$ for 1770 independent reflections. The coordination around lead involves five oxygen atoms from water molecules 2.47(2)–2.82(2) Å; two oxygen atoms from waters, 2.90(2), 2.96(2); and one nitrogen atom from a pyridine at 2.62 Å. The coordination polyhedra are linked in zigzag chains along [001] with one carboxylic oxygen and one water oxygen atom in a bridging position between adjacent metal atoms.

M.B. Cingi, A.G. Manfredotti, C. Guastini and M. Nardelli, *Gazz. Chim. Ital.*, 102 (1972) 1034.

cis-Dichloro(2,2'-oxydi-3-butene) platinum (II), $\text{PtCl}_2((\text{CH}_2=\text{CH}-\text{CH}(\text{CH}_3))_2\text{O})$

(*PT*) $Z = 2$, $R = 5.2\%$ for 2441 independent reflections. In the monomeric complex, both double bonds of the diolefin ether are coordinated to the platinum atom as in Zeise's salt and the square planar coordination is completed by two chlorine atoms. The $(=\text{CH}_2)$ groups of both double bonds are found on the same side of the coordination plane.

J. Hubert, A.L. Beauchamp and T. Theophanides, *Can. J. Chem.*, 51 (1973) 604.

Tris(acetylacetonato) (1, 10-phenanthroline) europium (III), $\text{Eu}(\text{C}_6\text{H}_7\text{O}_2)_3(\text{C}_{12}\text{H}_8\text{N}_2)$

(*P2₁/c*) $Z = 4$, $R = 6.5\%$ for 2855 reflections. The molecules are monomeric and the europium ion is coordinated to eight ligand atoms which form a distorted square antiprism. Six Eu–O lengths average 2.397 Å and two Eu–N average 2.643 Å. The distortion enables all characteristic electronic transitions to become allowed.

W.H. Watson, R.J. Williams and N.R. Stemple, *J. Inorg. Nucl. Chem.*, 34 (1972) 501.

AmI_2

(*P4/nmm*). The compound is isostructural with EuI_2 . Magnetic data are discussed.

R.D. Bayborz, L.B. Asprey, C.E. Strouse and E. Fukushima, *J. Inorg. Nucl. Chem.*, 34 (1972) 3427.

AmCl_2 and AmBr_2

R.D. Bayborz, *J. Inorg. Nucl. Chem.*, 35 (1973) 483.

Californium trichloride

Two crystalline modifications were studied, the hexagonal UCl_3 type, and the orthorhombic PuBr_3 type. In the hexagonal form the Cf atom is nine-coordinated by six Cl atoms at 2.815(3) Å and three at 2.924(4) Å while in the orthorhombic form, the coordination is eightfold with two Cf–Cl distances of 2.690(7) Å, four of 2.806(4) Å and of 2.940(6) Å. An ionic radius of 0.932(3) Å was derived for the six-coordinated Cf^{3+} ion.

J.H. Burns, J.R. Peterson and R.D. Bayborz, *J. Inorg. Nucl. Chem.*, 35 (1973) 1171.

Tetraethylammonium tris(*o*-ethylxanthato) cadmiumate (II)

($P2_1/c$) $Z = 4$, $R = 7.5\%$ for 2612 independent reflections. The anion is five-coordinate with two bidentate and one monodentate xanthato groups. The environment of the cadmium is tetragonal pyramidal. C—S are 1.63(2)–1.67(2) in the bidentate xanthato groups and 1.66 in the monodentate xanthate group for the sulphur atom not bonded to cadmium and 1.72(2) for the bonded sulphur atom.

B.F. Hoskins and B.P. Kelly, *Inorg. Nucl. Chem. Lett.*, 8 (1972) 875.

$\text{Cd}(\text{NH}_3)_2\text{Hg}(\text{CN})_4 \cdot 2\text{C}_6\text{H}_6$. A new-type clathrate compound

($P\bar{1}$) $Z = 2$, $R = 6.3\%$ for 3501 reflections. A mercury atom is tetrahedrally coordinated to the carbon atoms of four cyanide groups (Hg—C 2.19–2.21 Å) whereas the cadmium atom is octahedrally surrounded by six nitrogen atoms. Two of the latter are from ammonias attached to the cadmium, *trans* to each other, Cd—N 2.31, 2.37 Å, and four are cyanides, Cd—N 2.34–2.35 Å.

R. Kuroda, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 13.

$[(\text{CH}_3)_4\text{N}]_2[\text{Tc}^{\text{IV}}(\text{NCS})_6]$

J. Hauck and K. Schwochau, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 303.

[Bis(*N*-ethylenedimethylaminesalicylaldiminato) dioxouranium (VI), $\text{C}_{22}\text{H}_{30}\text{N}_4\text{O}_4\text{U}$

($P\bar{1}$) $Z = 2$, $R = 5.0\%$ for 2706 reflections. Each uranium is in a pentagonal bipyramidal environment with two oxygens and three nitrogen atoms forming the base of the pyramid and uranyl oxygens occupying the apical positions. (U—N(CH₃)₂) is 2.69 Å.

G. Bandoli, D.A. Clemente, F. Benetollo, M. Vidali, P.A. Vigato and U. Casellato, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 433.

μ -1,2-Dimethylthioethane bis[tungsten (VI) sulphide tetrachloride], $\text{C}_4\text{H}_{10}\text{Cl}_8\text{S}_4\text{W}_2$

($P2_1/n$) $Z = 4$, $R = 8.0\%$ for 1277 independent reflections. The addition of a ninth atom (*trans* to W=S) has little effect on the square pyramidal configuration of the WCl_4 groups, which are equivalent to those found in WCl_4 . The WCl_4 entities are not symmetrical with respect to the ligand plane, but are rotated such that the C—S—W—Cl dihedral angles are $\sim 45^\circ$ (or 135°), the molecule being centrosymmetric.

D. Britnell, M.G.B. Drew, G.W.A. Fowles and D.A. Rice, *Inorg. Nucl. Chem. Lett.*, 9 (1973) 501.