# **Applied Organometallic Chemistry**

(Appl. Organometal. Chem.)

# **CONTENTS**

**Volume 19 Number 1** *Papers published online January 2005* 

## Section: Bioorganometallic Chemistry

The medicinal properties of organometallic compounds are under intensive evaluation, especially in the treatment of cancer, as they exhibit certain properties that appear to be superior to those of other types of drug. We identify these properties and show where current

Design of organometallic pharmaceuticals.

Functional ligand with known biological activity.

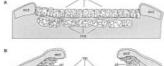
$$L_s = \begin{bmatrix} L_b \\ \text{Motal centre - can bind to DN} \\ \text{and/or proteins.} \end{bmatrix}$$
 Sacrificial ligand. 
$$L_s = \begin{bmatrix} L_c \\ \text{Functional ligand with conducive chemical properties.} \end{bmatrix}$$

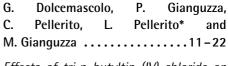
Metal centre - can bind to DNA Development of organometallic (organoand/or proteins. transition metal) pharmaceuticals

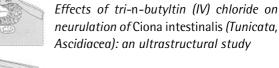
organometallic drugs are finding most interest, and describe some recent results from our own research.

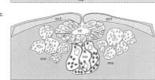
#### Section: Speciation Analysis and Environment

This paper reports the cytotoxic effects of tri-n-butyltin (IV) chloride, TBTCI, on the neurulation process of the ascidian *Ciona intestinalis*. Exposure of the embryos at early neurula stage in  $10^{-5}$  and  $10^{-7}$  M TBT (IV) chloride solutions for 1-2 h provoked the irreversible arrest of their development.









Reduction of total lipids (TL) content and significant variations of triglyceride (TG) and phospholipid (PL) fractions were observed as a consequence of exposure of *Ciona intestinalis* ovaries to tributyltin chloride (TBTCL) solutions.

	Control in SSW	Control in SSW containing 0.07% DMSO
% TL	8.4	8.0
σ	0.8	0.5
ANOVA	a	a
% TG	80.3	77.3
σ	4.1	2.8
ANOVA	a	a
% PL	18.0	20.0
σ	2.3	1.8
ANOVA	a	a

Lipid and fatty acid variations in Ciona intestinalis ovary after tri-n-butyltin(IV)-chloride exposure

Continued overleaf

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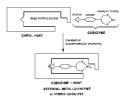


#### Section: Materials, Nanoscience and Catalysis

Palladium-catalysed cyclocarbonylation of isopulegol, dihydromyrcenol, and geraniol is achieved with good to high yields in the presence of the active species [Pd(H)(SnCl<sub>3</sub>)(L<sub>2</sub>)].

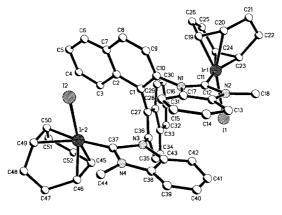
An efficient catalytic system for cyclocarbonylation of terpenes into lactones

This review is focused on the use of semisynthetic enzymes to produce efficient enantioselective hybrid catalysts for a given reaction. Also included are our recent research results and implications in developing the biotin—avidin technology to localize the biotinylated organometallic catalyst precursor within a well-defined protein environment.



Design of artificial metalloenzymes

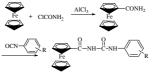
An axially chiral Ir(I) – NHC complex was successfully synthesized from the reaction of axially chiral binaphthyl dibenzimidazolium salt 5 with [Ir(COD)CI]<sub>2</sub> in THF in the presence of base KO<sup>t</sup>Bu under reflux. Its unique crystal structure is unambiguously disclosed by X-ray diffraction.



M. Shi\* and W.-L. Duan ......40-44

Synthesis of an axially chiral Ir-NHC complex derived from BINAM

We replaced the benzoyl moiety by ferrocenoyl in benzoylphenylurea, and synthesized a series of new benzoylphenylureas containing a ferrocenyl moiety by the reaction of carbamylferrocene with phenylisocyanate in good yields. The crystal structure and bioactivities of the new compounds was determined.



L. Chen, Q. Wang\*, R. Huang, C. Mao, J. Shang and H. Song .......45-48

Synthesis of ferrocenoylphenylureas and the crystal structure of FcCONHCONHC $_6H_5$ 

New phenylene-silylene-ethylene poly mers have been successfully synthesized using platinum-divinylsiloxane or rhodium and iridium silox-

P. Pawluc, B. Marciniec\*, I. Kownacki and H. Maciejewski ..............49 – 54

ide complex-catalysed polyhydrosilylation of divinylsubstituted carbosilanes with dihydrocarbosilanes or intermolecular hydrosilylation of new hydrovinylcarbosilane.

Synthesis of phenylene – silylene – ethylene polymers via transition metal complex catalyzed hydrosilylation polymerization

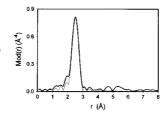
A convenient method for Suzuki cross-coupling reactions is presented

which employs a catalyst formed in situ from  $Pd(OAc)_2$  and air-stable 1,3-bis(alkyl)imidazolinium salts.

I. Özdemir\*, S. Demir, S. Yaşar and B. Çetinkaya ......55-58

Palladium-catalysed Suzuki reaction of aryl chlorides in aqueous media using 1,3-dialkylimidazolidin-2-ylidene ligands

A thorough characterization of cadmium bis(O-alkylxanthates), Cd(O-RXan)<sub>2</sub>, where O-RXan is  $CH_3CH_2OCS_2$  (O-EtXan) or  $(CH_3)_2CHOCS_2$  (O-iPrXan), was undertaken by means of a multi-technique approach, with particular attention to their thermal behavior and fragmentation pattern. The results indicate the suitability of cadmium bis(O-



alkylxanthates) as single-source precursors for the CVD of CdS thin films.

D. Barreca\*, A. Gasparotto, C. Maragno, R. Seraglia, E. Tondello, A. Venzo, V. Krishnan and H. Bertagnolli

Cadmium O-alkylxanthates as CVD precursors of CdS: a chemical characterization

Two new series of various substituted half-sandwich titanium complexes  $PhCH_2$ - $CpTiCl_2(OR)$  (R = Et (1),  $^iPr$  (2),  $^tBu$  (3),

cyclohexyl (4), benzyl (5)) and PhCH<sub>2</sub>CpTi(OR)<sub>3</sub> (R = Et (6), <sup>i</sup>Pr (7), <sup>t</sup>Bu (8), cyclohexyl (9), benzyl (10)) were prepared. All complexes were tested as catalyst precursors for the syndiotactic polymerization of styrene. The effect of variation in temperature, MAO/catalyst molar ratio and polymerization time was studied. The s-PS sample obtained exhibits low molecular weight ( $M_{\rm w} = 2.78 \times 10^4$ ) and narrow molecular weight distribution ( $M_{\rm w}/M_{\rm n} = 1.50$ ).

H. Zhang, Q. Chen, Y. Qian and J. Huang\* ......68 – 75

Synthesis of monoalkoxy and trialkoxy substituted half-sandwich titanium complexes  $PhCH_2CpTiCl_{3-n}$  (OR)<sub>n</sub> (n = 1 or 3) as catalysts for syndiotactic styrene polymerization

These complexes ( $n = \sim 6$  or  $\sim 33$ ; R = Me or Ph), as free radical initiators, afforded methyl methacrylate polymerization in chlorinated solvents.

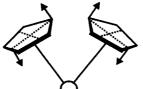
Molybdenum tetracarbonyl complexes with linear chain polyether-containing Schiff base ligands and their reactivity in the polymerization of methyl methacrylate

A thermoregulated phase-transfer (TRPT) Rh(I) complex catalyst A prepared from Rh(acac)(CO)<sub>2</sub> and a thermoregulated ligand  $CH_3(OCH_2CH_2)_mPPh_2$  ( $M_w=918$ ) was applied to the biphasic hydroformylation of 1-octene, and a high activity with an aldehyde yield of 97.5% was demonstrated.

F. Wen, H. Bönnemann\*, J. Jiang, D. Lu, Y. Wang and Z. Jin ......81-89

Evidence of colloidal rhodium formation during the biphasic hydroformylation of 1-octene with thermoregulated phasetransfer phosphine rhodium(I) catalysts

Reactions of antitumour active bent metallocenes with bioligands can be accompanied by full  $\eta^5$ -bonded cyclopentadienyl ring elimination or the bent metallocene fragment M(Cp) $_2$  remains unaffected. It has not been easy to decide unambiguously whether the reaction products contain M(Cp) $_2$  or M(Cp) fragments. We have found that Raman spectroscopy is



M. Pavlišta\*, R. Bína, Z. Černošek, M. Erben, J. Vinklárek and I. Pavlík ......90-93

Raman spectroscopic evidence for bent metallocene fragments  $[M(Cp)_2]^{2+}$ 

a very simple and sensitive tool for detection of the bent metallocene fragment  $M(Cp)_2$  in both solid state and solution.

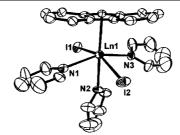
Complex [(COD)Pt(CH<sub>3</sub>)<sub>2</sub>] is available from Pt(acac)<sub>2</sub> in a one-pot procedure with 92% yield.

Pt(acac)<sub>2</sub> + COD + 2 Al(CH<sub>3</sub>)<sub>3</sub> 
$$\rightarrow$$
 Pt $^{\text{CH}_3}$  + 2 (acac)Al(CH<sub>3</sub>)<sub>2</sub>

F. Wen and H. Bönnemann\* .....94-97

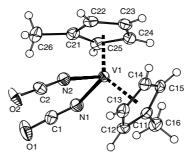
A facile one-pot synthesis of [(COD)- $Pt(CH_3)_2$ ]

The structures of the title compounds are mononuclear with each lanthanide bound by a single  $\eta^5$ -fluorenyl ligand, two trans-disposed iodides and three meridionally oriented pyridine molecules.



Crystallographic report:  $(\eta^5$ -Fluorenyl)-tris-pyridine-di-iodo-lanthanum(III) and -neodymium(III)

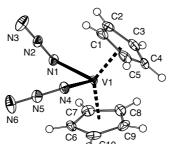
The crystal structure of the first cyclopentadienyl vanadium(IV) pseudohalide complex,  $(\eta^5-C_5H_4CH_3)_2V(NCO)_2$ , was determined. The molecule has a typical bent metallocene structure in which two  $\eta^5$ -bonded methyl-cyclopentadienyl rings and two nitrogen atoms of cyanato ligands occupy the pseudotetrahedral coordination sites around the vanadium(IV) center.



J. Honzíček, M. Erben, I. Císařová and J. Vinklárek\* ...... 100–101

Crystallographic report:  $Bis(\eta^5-methyl-cyclopentadienyl)-bis(cyanato)-vanadium (IV)$ 

The crystal structure of the cyclopentadienyl vanadocene complex  $(\eta^5-C_5H_5)_2V(N_3)_2$  was determined. The molecule has a typical bent metallocene structure in which two  $\eta^5$ -bonded cyclopentadienyl rings and two nitrogen atoms of azide ligands occupy the pseudotetrahedral coordination sites around the vanadium(IV) center.



Crystallographic report: The crystal structure of  $bis(\eta^5$ -cyclopentadienyl)-diazidovanadium(IV)

#### Section: Main Group Metal Compounds

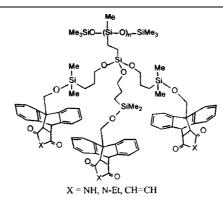
It was established that BBr<sub>3</sub> can bring about phenyl-bromine ligand exchange on germanium without breaking the coexisting benzyl-germanium bond(s). This reaction will terminate when one phenyl group is exchanged, even if two or more phenyl groups are bonded to germanium.

$$Ge + CH_2 - CH$$

Y. Takeuchi\*, K. Suzuki, T. Yagi, Y. Yoshida and Y. Suzuki .... 104-107

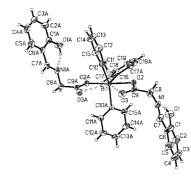
A novel phenyl-bromine ligand exchange reaction on germanium by boron tribromide

Anthracene-grafted polysiloxane was prepared by the dehydrocoupling between linear siloxane polymers and anthracenecarbinol. The cylindrical type of dendritic polysiloxane was prepared up to the second generation by the hydrosilation between polysiloxane and vinyltrichlorosilane, and the continual addition of allyl alcohol. The Diels-Alder reaction of anthracene moieties on polysiloxane and maleimide derivatives, maleic acid anhydride, and 1,4-quinone derivatives was carried out under mild conditions.



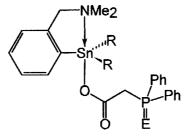
Diels-Alder reaction of anthracene on grafted polysiloxane and cylindrical carbosilane dendrimer

Four novel triarylbismuth(V) di(N-salicylidene)amino carboxylates were synthesized. Their structures were confirmed by  $^1$ H NMR, IR, elemental analysis and X-ray diffraction. The *in vitro* antitumor activities of all compounds against three human cancer cells at 10  $\mu$ M are reported.



Synthesis, characterization and in vitro cytotoxicity screening of some triarylbismuth(V) di(N-salicylidene)amino carboxylates and the crystal structure of  $(2-HOC_6H_4CH=NCH_2CO_2)_2Bi(C_6H_5)_3$ 

Monomeric organotin(IV) carboxylates  $R_2$ - $L^{NC}SnOC(0)CH_2P(E)Ph_2$ , where  $L^{NC}$  is a C,N-chelating 2-(dimethylamino)phenyl group, and R/E = Ph/void (1a), Ph/O (1b), Ph/S (1c), Me/void (2a), Me/O (2b) and Me/S (1c), were synthesized and characterized by multinuclear NMR, IR and MS spectra. The solid-state structures of 1b, 1c, 2b and 2c were determined by single-crystal X-ray diffraction.



Preparation and structures of [2-(dimethylamino)phenyl]diorganotin(IV) acetates substituted with organophosphorus groups in the  $\alpha$ -position of the acetate ligand

Radiotracer batch ion-exchange experiments were employed to investigate the uptake of <sup>90</sup>Sr and <sup>137</sup>Cs isotopes by various cation-exchanged forms of a 30% cross-linked macroporous 1-vinyl-2-pyrrolidone-divinylbenzene cation-exchange resin. The results showed that the Li<sup>+</sup> and H<sup>+</sup> forms of the resin were more effective in removing <sup>90</sup>Sr and <sup>137</sup>Cs, both in carrier-free and with carrier concentrations.

M. Zamin\*, T. Shaheen and S. A. R. Zaidi ...... 125–128

lon exchange of <sup>90</sup>Sr and <sup>137</sup>Cs into 1vinyl-2-pyrrolidone – divinylbenzene cation-exchange resin

Arsoxanes  $(RAsO)_n$  were prepared from the reactions of RLi and bis(dimethylamino)chloroarsine; the X-ray crystal structure of tetrameric m-trifluromethylphenylarsine oxide is described.

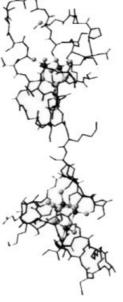
A convenient way of making arsoxanes  $(RAsO)_n$ , X-ray crystal structure of  $(m-F_3CC_6H_4AsO)_4$ 

Six new organotin(IV) complexes with *N*-maleoylglycine are reported; these complexes were duly characterized by multinuclear magnetic resonance, FT IR and <sup>119m</sup>Sn Mössbauer spectroscopies with MS and %CHN analysis.

In vivo toxicological effects and spectral studies of organotin(IV) Carboxylates of N-maleoylglycine

Spectroscopic analysis revealed octahedral geometry for diorganotin(IV) derivatives. All the organotin(IV) complexes were screened for *in vivo* antitumour activity against human tumoural cells; two compounds (3 and 4) showed antitumour activity *in vivo*, whereas all the compounds (1–6) displayed toxicity *in vitro*.

Metallothioneins, which show high affinity for metals such as zinc, copper and mercury, were purified from tissues of rat after oral intake of HgCl<sub>2</sub>, and then characterized using hyphenated method developed. Significant mercurybinding metallothioneins co-existing with copper and maybe zinc were found in kidney

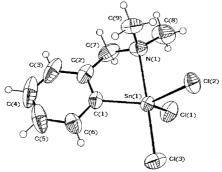


Investigation of mercury metallothionein complexes in tissues of rat after oral intake of HgCl<sub>2</sub>

Different organotin phenoxides react at room temperature with diethyl acetylene-dicarboxylate in diethyl ether, in the presence of lithium perchlorate to give a mixture of corresponding phenyl vinyl ethers and ring ethenylated phenols.

Studies on the catalysis of the reaction of organotin phenoxides with diethyl acetylenedicarboxylate

[2-( $Me_2NCH_2$ ) $C_6H_4$ ]SnCl $_3$  (1) and its DMSO adduct [2-( $Me_2NCH_2$ ) $C_6H_4$ ] SnCl $_3\cdot$ DMSO (1a) have been prepared. The NMR data ( $^1H$ ,  $^{13}C$ ,  $^{119}Sn$ ) in solution indicate 5- and 6-coordinated metal atoms in 1 and 1a, respectively. The molecular structure in solid state was established by single-crystal X-ray diffraction. The N atom of the pendant  $CH_2NMe_2$  arm is strongly coordinated to the tin atom, thus resulting in dis-



Synthesis, solution behaviour and X-ray structures of  $[2-(Me_2NCH_2)C_6H_4]SnCl_3$  and  $[2-(Me_2NCH_2)C_6H_4]SnCl_3 \cdot DMSO$ 

torted trigonal bipyramidal (C, N)SnCl<sub>3</sub> and octahedral (C, N)SnCl<sub>3</sub>O cores for 1 and 1a, respectively. For both compounds hydrogen bonding generates supramolecular associations in the crystal.

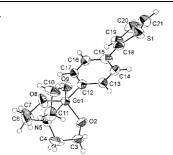
A one-pot synthesis of various unsymmetrical 2-bromo-5-pyridylselenium compounds has been carried out under non-cryogenic conditions by selective mono bromine-magnesium exchange of 2,5-dibromopyridine using isopropy-

K. K. Bhasin\*, V. Arora, S. K. Sharma and P. Venugopalan ...... 161–166

A novel and facile one-pot synthesis of pyridylselenium compounds through selective bromine-magnesium exchange with isopropylmagnesium halide

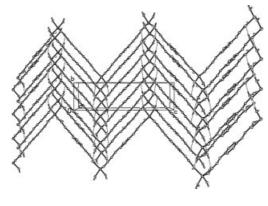
Imagnesium chloride. This exchange, upon insertion of elemental selenium, followed by treatment with alkyl halide, gives the title compounds in good yield. The molecular structure of 2-bromo-5-selenopyridyltribromomethane has been examined: the compound crystallizes in the monoclinic space group  $P2_1/n$ ; the X-ray structure shows that intermolecular  $Br\cdots Br$ ,  $N\cdots Se$  and  $Se\cdots Br$  interactions control its crystal packing.

The X-ray crystal structure of 1-[4-(2-thienyl)phenyl]germatrane reveals that there are two molecules connected by a center of pseudoinversion; the germanium atom is penta-coordinated and adopts trigonal bipyramidal geometry. The (2-thienyl)phenyl group and the nitrogen atom each occupy an apical position with transannular  $N\rightarrow$ Ge bond of 2.247(4) Å and 2.219(4) Å.



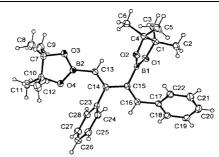
Crystallographic report: 1-[4-(2-Thienyl)-phenyl]germatrane

The zinc(II) atom in the crystal structure of the title coordination polymer, [Zn(p-BDOA)·  $2H_2O]_n$  (p-BDOA<sup>2-</sup> = benzene-1,4-dioxyacetate), exists in a trigonal distorted prismatic geometry. Adjacent zinc(II) ions linked are by the p-BDOA<sup>2-</sup> ligands to furnish a one-dimensional zigzag chain. A three-dimensional network structure is stabilized by extended hydrogen bonds.



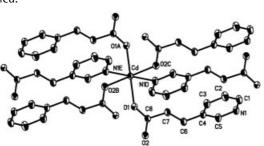
Crystallographic report: Diaqua(benzene-1,4-dioxyacetate)zinc(II): a one-dimensional zigzag chain

The compound comprises a 1*Z*,3*E*-butadiene moiety substituted by two pinacol boronate functional groups.



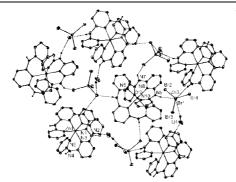
Crystallographic report: 2,4-Diphenyl-1,3-bis(4,4,5,5-tetramethyl[1,3,2]dioxaboro-lan-2-yl)-buta-1Z,3E-diene

In  $[bis(3-pyridylacrylato)cadmium(II)]_n$ , the local coordination geometry around the cadmium center is based on an octahedron. The carboxylate acts as a tridentate ligand by bridging two cadmium atoms and binds a third cadmium atom via the pyridyl group, with the result that a two-dimensional layered network is formed.



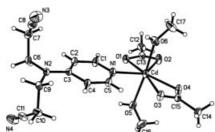
Crystallographic report:  $[Bis(3-pyridyla-crylato)cadmium(II)]_n$ 

The zinc(II) atom in the complex cation of [Zn(tpda)<sub>2</sub>][ZnBr<sub>4</sub>]·H2O is octahedrally coordinated, whereas the zinc(II) atom in the anion is tetrahedrally coordinated. The cations and the anions are connected by hydrogen bonds, affording a two-dimensional network.



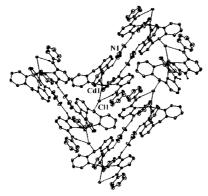
Crystallographic report: Bis(tripyridyldia-mine)zinc(II) tetrabromozincate(II) hydrate

In the molecule of  $Cd(CH_3CO_2)_2(C_{11}H_{12}N_4)(CH_3OH)_2$ , the cadmium atom is coordinated by the pyridine-nitrogen atom of the 4-[N, N-bis(2-cyanoethyl)amino]pyridine ligand, two oxygen atoms of two methanol molecules and four oxygen atoms of two acetate groups in a distorted pentagonal bipyramidal geometry.



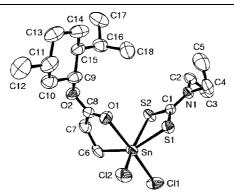
Crystallographic report: Bis(acetato-O,O') -{4-[N,N-bis(2-cyanoethyl)amino]pyridine} bis(methanol)cadmium(II), [Cd(II)( $C_{11}$  H<sub>12</sub>N<sub>4</sub>) (CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub>(CH<sub>3</sub>OH)<sub>2</sub>]

The cadmium(II) atom in [(tpda)<sub>2</sub>CdCl] NO<sub>3</sub>, is octahedrally coordinated to one chlorine and five pyridyl-nitrogen atoms. The coordination cations and nitrate anions are connected by multiple hydrogen bonds, affording a two-dimensional structure.



Crystallographic report: Chlorobis(tripyridyldiamine)cadmium(II) nitrate

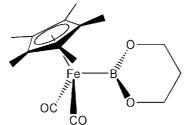
The tin atom in the title compound adopts a distorted octahedral geometry within a CCl<sub>2</sub>OS<sub>2</sub>



L. Tian, Z. Shang\*, Q. Yu and L. Zhang ...... 179–180

Crystallographic report L-(-)-Dichloro( $\beta$ -menthoxycarbonylethyl)tin N,N-diethyl-dithiocarbamate

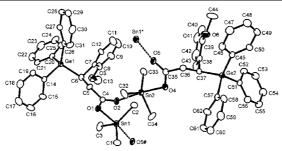
The structure of the pentamethylcyclopentadienyliron boryl complex ( $\eta^5$ -C<sub>5</sub>Me<sub>5</sub>) Fe(CO)<sub>2</sub>B(tmg) (tmg = - OCH<sub>2</sub>CH<sub>2</sub>C-) reveals an Fe-B distance (2.024(4) Å) significantly in excess of that reported for related catecholboryl complexes.



A. Rossin, S. Aldridge\* and L.-l. Ooi ... 181 – 182

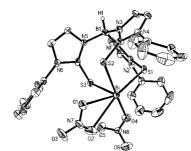
Crystallographic report:  $(\eta^5-C_5Me_5)$ - $Fe(CO)_2(BOCH_2CH_2CH_2O)$ : an organoiron complex containing the (trimethylene-glycolato)boryl ligand

The crystal structure of bimetallic compound [3-triphenylgermyl)-3-o-me-thoxyphenylpropionato] trimethyltin(IV) is reported. The structure is polymeric owing to the presence of bidentate bridging carboxylate ligands.



Crystallographic report: Polymeric [3-(triphenylgermyl)-3-o-methoxyphenyl-propionato]trimethyltin(IV)

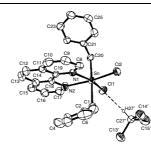
 $Bi[HB(tim^{Ph})_3](NO_3)_2$  features a distorted pentagonal pyramidal geometry defined by a sulfur-rich tripodal ligand and three oxygen atoms, derived from mono- and bi-dentate nitrate ligands.



M. Shu\*, J. Cui and J. Sun ...... 184–185

Crystallographic report: Hydro[tris(3-phenyl-2-thioimidazol-1-yl)]boratobis-muth(III) dinitrate

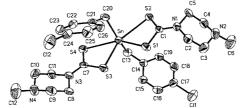
The monomeric title compound features a distorted octahedral tin (IV) centre within a C<sub>2</sub>Cl<sub>2</sub>N<sub>2</sub> donor set with two *cis* Cl atoms and two *trans* benzyl groups.



B. S. Krishnamoorthy, S. Chandrasekar, P. Arunkumar and K. Panchanatheswaran\*......186

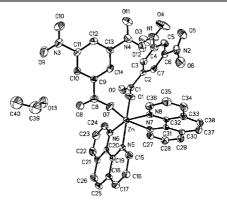
Crystallographic report: Dibenzyl(dichloro)(1,10-phenanthroline)tin (IV) chloroform solvate

The tin atom in  $(4-CI-C_6H_4CH_2)_2Sn$   $[S_2CN(CH_2CH_2)_2NCH_3]_2$  is in a  $C_2S_4$  skew-trapezoidal bipyramidal geometry with the two carbon atoms being disposed over the weaker Sn-S bonds.



H. D. Yin\* and S. C. Xue .......... 187

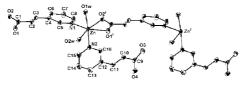
Crystallographic report: Di(p-chlorobenzyl)tin bis(N-methyl piperazinyldithiocarbamate) Cis-[Zn(3,5-dinitrobenzoato)<sub>2</sub>(1,10-phenanthroline)<sub>2</sub>]·CH<sub>3</sub>CH<sub>2</sub>OH features unidentate and cis-disposed 3,5-dinitrobenzoate ligands and chelating 1,10-phenanthroline ligands so that a distorted octahedral N<sub>4</sub>O<sub>2</sub> coordination geometry results.



H. D. Yin\*, Q. B. Wang ............ 188

Crystallographic report: Cis- $[Zn(3,5-dini-trobenzoato)_2(1,10-phenanthroline)_2]$ CH<sub>3</sub>CH<sub>2</sub>OH

The Zn center in [bis(3-pyridylacrylato)diaquazinc(II)] $_n$  is in a slightly distorted octahedral geometry within a cis-N $_2$ O $_4$  donor set. Each tridentate 3-pyridylacrylate ligand

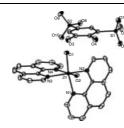


O. Wu, X.-F. Huang, T. Zhou, Y.-Z. Tang and R.-G. Xiong\*............... 189–190

Crystallographic report: Polymeric [diaqua bis(3-pyridylacrylato)zinc(II)]

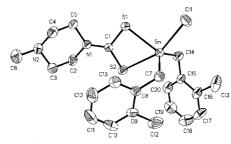
links two Zn centers, resulting in the formation of a linear chain.

The zinc atom has a distorted octahedral geometry defined by two 1,10-phenanthroline and two *cis* water molecules. A three-dimensional network structure arises owing to extensive hydrogen bonds involving all the components of  $[Zn(phen)_2(H_2O)_2][C_6H_2(OH)_2(SO_3)_2]\cdot 3H_2O$ .



Crystallographic report: Diaquabis(1,10-phenanthroline)zinc(II) 4,5-dihydroxy-1,3-benzenedisulfonate trihydrate

The tin atom in  $(2-Cl-C_6H_4CH_2)_2Sn(Cl)S_2CN(CH_2CH_2)_2NCH_3$  is in a trigonal bipyramid geometry defined by a  $C_2ClS_2$  donor set with the chlorine and weakly bound sulfur atoms in axial positions.



H.-D. Yin\* and S.-C. Xue ...... 193

Crystallographic report: Chlorodi(ochlorobenzyl)tin N-methylpiperazinyldithiocarbamate

The tin atom in  $\{(2-Cl-C_6H_4CH_2)_2Sn[S_2CN(CH_2CH_2)_2NCH_3]_2\}_2$  is in a skew-trapezoidal bipyramidal geometry defined by a  $C_2S_4$  set with C-Sn-C 150.61(19)°. Centrosymmetric pairs associated via weak  $Sn \cdots S$  to form a dimer.

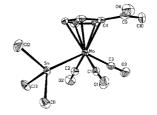
H.-D. Yin\* and S.-C. Xue ...... 194

Crystallographic report: A weakly bridged dimer: di(o-chlorobenzyl)tin(IV) bis(N-methylpiperazinyldithiocarbamate)

The centrosymmetric  $\{[(HOOCCH_2PPh_2)]_2(CH_2)_4\}^{2+}$  cation adopts an extended conformation in which the phosphorus center adopts a tetrahedral geometry.  $O-H\cdots O$  and  $C-H\cdots O$  hydrogen bonding interactions expand this structure to form a two-dimensional layered architecture.

Crystallographic report: 1,4-Bis(carboxy-methyldiphenylphosphonio)butane dinitrate  $(CH_2)_4[(HOOCCH_2)Ph_2P^{(+)}]_2$ .  $2(NO_3^{(-)})$ 

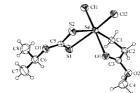
The tin atom in  $\text{Cl}_3\text{SnMo}(\text{CO})_3\text{C}_5\text{H}_4\text{COCH}_3$  is in a distorted tetrahedral geometry with the molybdenum atom adopting a 3:4 piano stool geometry.



X.-M. Zhao, J.-T. Wang and L.-F. Tang\* ..... 197

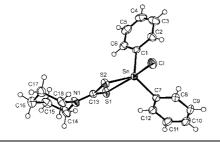
Crystallographic report: Tricarbonylacetyl-cyclopentadienylmolybdenum-tin(IV) tri-chloride,  $Cl_3SnMo(CO)_3C_5H_4COCH_3$ 

The tin atom in  $CH_3OCOCH_2CH_2SnCl_2[S_2COCH(CH_3)_2]$  adopts a distorted octahedral geometry via the bidentate xanthate ligand and intramolecular carbonyl coordination.



Crystallographic report: Dichloro( $\beta$ -methoxycarbonylethyl)tin(IV) isopropylxanthate

A trigonal bipyramidal  $C_2CIS_2$  coordination geometry for tin is found in  $Ph_2Sn(S_2CN(CH_2)_5)CI$ .



S. Ali\*, S. U. Ahmad, Sadiq-ur-Rehman, S. Shahzadi, M. Parvez and M. Mazhar.....200

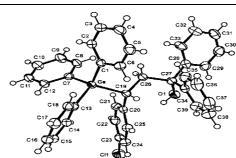
Crystallographic report: Chlorodiphenyltin (IV) piperidine-1-carbodithioate

 $Me_2Sn(S_2CN(CH_2)_5)Cl$  contains five-coordinated tin with a bidentate dithiocarbamate ligand spanning equatorial and axial positions in a distorted trigonal bipyramidal geometry.

S. Ali\*, S. U. Ahmad, S. Shahzadi, Sadiqur-Rehman, M. Parvez and M. Mazhar .....201

Crystallographic report: Chlorodimethyltin (IV) piperidine-1-carbodithioate

The germanium atom in  $[(C_6H_5)_3$  GeCH(4-ClC<sub>6</sub>H<sub>4</sub>)CH<sub>2</sub>C(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>OH] is in a distorted tetrahedral geometry. Steric hindrance precludes O-H···O intra- or inter-molecular bonding.



M. K. Khosa, M. Parvez, M. Mazhar\*, S. Ali and Sadiq-ur-Rehman 202

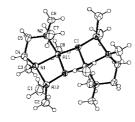
Crystallographic report: 1,1-Diphenyl-3-(triphenylgermyl)-3-(4-chlorophenyl) propanol

The molecular structure of  $[Zn(O_2CC_6 H_4NO_2-m)_2(pyridine)_2]$  exhibits a distorted  $N_2O_2$  tetrahedral geometry; the molecule has two fold symmetry.

H.-D. Yin\* and Q.-B. Wang ...... 203

Crystallographic report: Bis(m-nitro-benzoato)bis(pyridine)zinc(II)

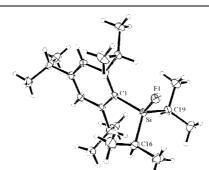
Dimeric and centrosymmetric [MeAlO·Me<sub>2</sub>AlNMe(CH<sub>2</sub>)<sub>2</sub> NMe<sub>2</sub>]<sub>2</sub> comprises two different kinds of aluminum center. One is tetrahedrally coordinated by two methyl groups, the nitrogen atom of one ligand molecule and one bridging oxygen atom, and the other is coordinated by one methyl group, two bridging oxygen atoms and two nitrogen atoms, derived from the amide ligand molecule in a distorted trigonal bipyramidal fashion.



E. Hecht\* ...... 204-205

Crystallographic report: [Methyl-aluminum- $\mu$ -oxo-dimethylaluminum-tri-methylethylenediamide] $_2$ 

Owing to steric congestion in  $i\text{-Pr}_2(2,4,6\text{-}i\text{-Pr}_3C_6H_2)$ SiF, the geometry at the Si atom deviates slightly from ideal tetrahedral geometry with an increased C-Si-C angle of 119.02(9)° and elongated Si-C and Si-F bond distances.



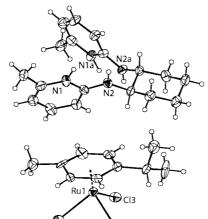
R. Pietschnig\* and K. Merz... 206-207

Crystallographic report: Fluoro-bis-iso-propyl-(2,4,6-tris-iso-propylphenyl)silane, i- $Pr_2(2,4,6$ -i- $Pr_3C_6H_2)$ SiF

The Zn center in  $[ZnCl_2(L-S-S-L)]$ , where L-S-S-L = bis(1-methylimidazole-2)disulfide, adopts a tetrahedral configuration defined by two Cl atoms and two N atoms from L-S-S-L, which was obtained by *in situ* oxidation of 1-methylimidazole-2-thione.

Crystallographic report: Dichloro[bis(1-methylimidazole-2)disulfide] zinc(II)

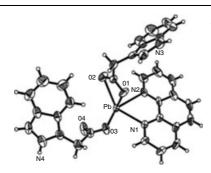
The title compound has been obtained in high yield by treating  $[RuCl_2(p\text{-cymene})]_2$  with an excess of hydrochloric acid in the presence of one equivalent of N,N'-bis-(6-methylpyrid-2-yl)-(1R,2R)-1,2-diaminocyclohexane.



J. A. Cabeza\*, I. da Silva, I. del Río and S. García-Granda ...... 209-210

Crystallographic report: [N,N'-Bis-(6-methylpyrid-2-ylium)(1R,2R)-1,2-diam-inocyclohexane] bis-[(p-cymene)-trichlororuthenate(II)]

The lead atom in Pb(phen)(IA) $_2$  is in a heavily distorted square pyramidal geometry surrounded by an N $_2$ O $_3$  donor set with Pb-O distances ranging from 2.354(5) to 2.726(5) Å.



**⊘**CI2

Z.-F. Chen\*, L. Huang, R.-X. Hu, S.-M. Shi, H. Liang\*\* and Y. Li ....... 211–212

Crystallographic report: Bis(indole-3-acetato)(1,10-phenanthroline)lead(II)

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