Applied Organometallic Chemistry

(Appl. Organometal. Chem.)

CONTENTS

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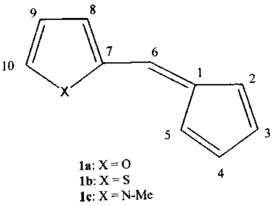
Section: Bioorganometallic Chemistry

Starting from 2-furylfulvene (**1a**), 2-thiophenylfulvene (**1b**), and 1-methyl-2-pyrrolylfulvene (**1c**), [1,2-di(cyclopentadienyl)-1,2-di-(2-furyl)ethanediyl] titanium dichloride (**2a**), [1,2-di(cyclopentadienyl)-1,2-di-(2-thiophenyl)ethanediyl] titanium dichloride (**2b**), and [1,2-di(cyclopentadienyl)-1,2-bis-(1-methyl-2-pyrrolyl)ethanediyl] titanium dichloride (**2c**) were synthesized.

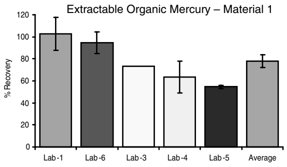


F.-J. K. Rehmann, L. P. Cuffe, O. Mendoza, D. K. Rai, N. Sweeney, K. Strohfeldt, W. M. Gallagher and M. Tacke* 293–300

Heteroaryl substituted ansa-*titanocene anti-cancer drugs derived from fulvenes and titanium dichloride*

Section: Speciation Analysis and Environment

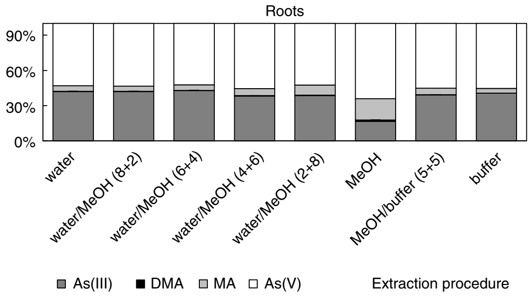
An inter-laboratory validation study of the EPA draft method 3200 was conducted by the United States Environmental Protection Agency (US EPA) on two specifically prepared soil matrices. The study has been performed successfully by a limited number of participating laboratories. Evaluation of the data demonstrates that the extraction method is more highly efficient for extracting the highly toxic methylmercury than inorganic mercury. An important finding was that the proposed method does not induce transformation of methylmercury to inorganic mercury.



G. M. Mizanur Rahman, H. M. 'Skip' Kingston*, J. C. Kern, S. W. Hartwell, R. F. Anderson and S.-Y. Yang.... 301–307

Inter-Laboratory Validation of EPA Method 3200 for Mercury Speciation Analysis Using Prepared Soil Reference Materials

Eight extraction agents (water, methanol–water mixtures in various ratios, methanol, a 20 mmol l^{-1} ammonium phosphate buffer, and a methanol–phosphate buffer) were tested for the extraction of arsenic compounds from fruits, stems + leaves, and roots of pepper plants. Quantitative extraction of arsenic compounds from different plant tissues requires the individual optimization of the extractant.



J. Száková*, P. Tlustoš, W. Goessler, D. Pavlíková and J. Balík 308–314

*Comparison of mild extraction procedures for determination of arsenic compounds in different parts of pepper plants (*Capsicum annum*, L.)*

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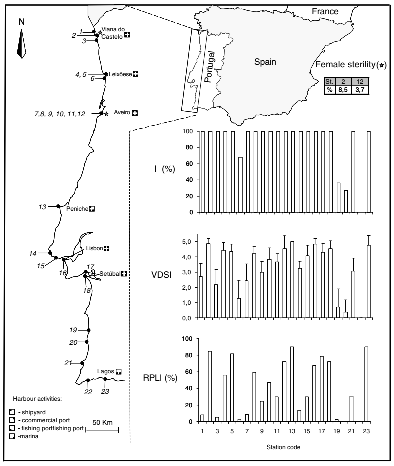
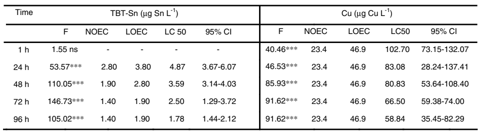
A survey of *Nassarius reticulatus* imposex levels and organotin b.b. was conducted along the Portuguese coast between May and August 2003 in order to assess the status of TBT pollution in the year that the IMO ban took place and to evaluate the temporal trend of organotin pollution over the last 3 years. The higher levels of imposex and TBT contamination occurred inside or close to harbours which were identified as "hotspots" of pollution along the Portuguese coast. No global trend in TBT pollution was observed over the 3 year period.



A. Sousa*, S. Mendo and C. Barroso 315–323

Imposex and organotin contamination in Nassarius reticulatus *(L.) along the Portuguese coast*

The acute toxicity of tributyltin TBT and copper to recently hatched veligers of the gastropped *Nassarius reticulatus* up to 96 h was assessed. Both TBT and copper had a highly significant effect on larvae survival ($p < 0.001$). TBT is far more toxic to *N. reticulatus* larvae than copper; however owing to the higher copper environmental concentrations the risk factors of the two biocides can approach each other. This stresses the need to find adequate substitutes for organotin biocides in antifouling paints in the future.



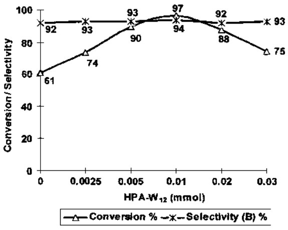
Time	TBT-Sn (µg Sn L^{-1})					Cu (µg Cu L^{-1})				
	F	NOEC	LOEC	LC 50	95% CI	F	NOEC	LOEC	LC50	95% CI
1 h	1.55 ns	-	-	-	-	40.46***	23.4	46.9	102.70	73.15-132.07
24 h	53.57***	2.80	3.80	4.87	3.67-6.07	46.53***	23.4	46.9	83.08	28.24-137.41
48 h	110.05***	1.90	2.80	3.59	3.14-4.03	85.93***	23.4	46.9	80.83	53.64-108.40
72 h	146.73***	1.40	1.90	2.50	1.29-3.72	91.62***	23.4	46.9	66.50	59.38-74.00
96 h	105.02***	1.40	1.90	1.78	1.44-2.12	91.62***	23.4	46.9	58.84	35.45-82.29

A. Sousa*, L. Génio, S. Mendo and C. Barrosoi 324–328

Comparison of the acute toxicity of tributyltin and copper to veliger larvae of Nassarius reticulatus *(L.)*

Section: Materials, Nanoscience and Catalysis

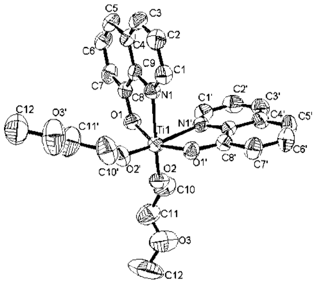
Active homogeneous catalytic systems based on $Rh_6(CO)_{16}$-heteropolyacids for the regioselective hydroformylation of styrene and 1-octene and their derivatives have been developed. The effects of the amount and the type of the heteropolyacid have been studied and showed a significant improvement of the conversion of styrene and the selectivity towards branched aldehydes.



B. El Ali*, J. Tijani, M. Fettouhi, A. Al-Arfaj and M. El-Faer 329–338

Heteropolyacids enhanced the catalytic activity of $Rh_6(CO)_{16}$ in the hydroformylation of alkenes

The reaction of $Ti(OCH_2CH_2OR)_4$ ($R{=}CH_3$ and C_2H_5) with 8-hydroxyquinoline yields $Ti(C_9H_6NO)_2(OCH_2CH_2OR)_2$. The molecular structure of $Ti(C_9H_6NO)_2(OCH_2CH_2OCH_3)_2$ has been determined by single-crystal x-ray structure analysis. The quinolinate derivatives of titanium alkoxides have very low susceptibility to hydrolysis.



M. Mirzaee and M. M. Amini* 339–342

Synthesis, spectroscopic and X-ray single-crystal structure study of bis(2-methoxyethanolato)-bis(8-quinolinato)titanium(IV)

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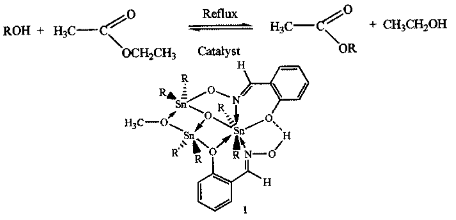

The reactivity and catalytic potential of the tin salicylaldoximate cluster $[(Me_2Sn)_2(Me_2SnO)(OCH_3)(HONZO)(ONZO)]$ (**1**), with HONZOH = *o*-HON=CH−C_6H_4OH, on the acylation reaction of various alcohols with ethyl acetate is reported. The catalyst is active toward primary and unhindered secondary alcohols, but inefficient toward tertiary and secondary bulky alcohols and phenols. A possible mechanism for the transesterification reaction catalyzed by **1**, accounting for the influence of steric factors, is proposed.

C. Camacho-Camacho, M. Biesemans*, I. Verbruggen and R. Willem **343–346**

Trinuclear tin salicylaldoximate cluster-catalyzed selective acylation of alcohols

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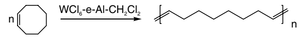
A typical low-strained monomer, cyclooctene, was polymerized via ring opening metathesis polymerization with electrochemically produced active species. A high cis polyoctenamer was obtained by this catalyst system with high yields in a short periods.



S. Çetinkaya, S. Karabulut, B. Düz and Y. İmamoğlu* **347–351**

Synthesis and characterization of polyoctenamer with $WCl_6-e^--Al-CH_2Cl_2$ catalyst system via ring opening metathesis polymerization

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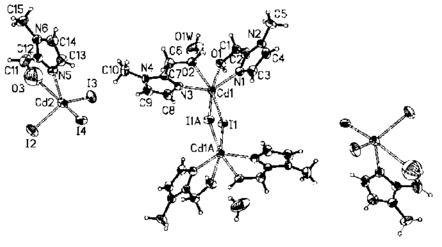
Section: Main Group Metal Compounds

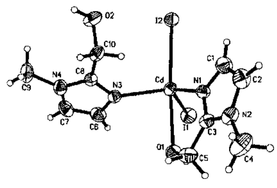
There are two types of Cd in the title compound, the six-coordinated Cd atom in the cation is in a distorted octahedral geometry while the four-coordinated Cd in the anion shows a distorted tetrahedral geometry.



Y.-Y. Yang, L. Szeto, G.-F. Ouyang, Z.-Q. Huang, W.-T. Wong* and S. Weng Ng **352–353**

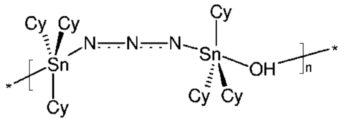
*μ_2-Diiodotetrakis(2-hydroxymethyl-1-methyl-1-imidazole-*N^3*)dicadmium(II) bis [triiodo(2-hydroxymetyl-1-methyl-1-imidazole-*N^3*)cadmate(II)] dihydrate*

The Cd atom in $Cd(Hmmi)_2I_2$ is five-coordinate with a trigonal bipyramidal geometry in which the apical sites are occupied by I and O atoms.



Y.-Y. Yang, Z.-Q. Huang, X.-M. Chen, W.-T. Wong* and S. W. Ng .. **354–355**

*Diiodobis(2-hydroxymethyl-1-methyl-1-imidazole-*N^3*) cadmium(II)*



The structures of $(Cy_3Sn)_2N_3(OH)$ (**1**) and $(Me_3Sn)_2N_3(OH)$ (**2**) are both polymeric with infinite chains of molecules linked by regularly alternating μ_2 bridging azide and hydroxide groups. In **2**, azide bridges in α,α mode with one terminal nitrogen atom forming both bridging bonds, while the other acts as acceptor for O−HN···hydrogen bonds, thus creating two-dimensional connectivity. In **1**, azide bridges in α,γ mode, where both terminal nitrogen atoms form bridging bonds and no hydrogen bonds are present.

R. Alan Howie*, J. L. Wardell and S. M. S. V. Wardell **356–359**

Bis[tris(cyclohexyl)tin] azide hydroxide, $(Cy_3Sn)_2N_3(OH)$: X-ray structure determination and comparison with analogous compounds

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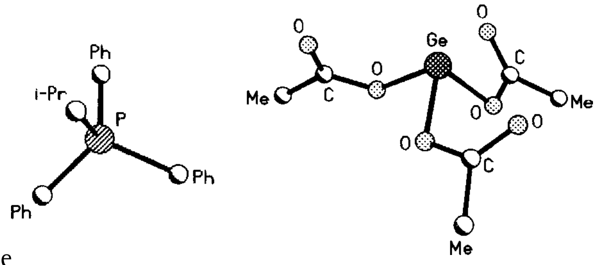
An X-ray diffraction study reveals an unusual structure of the new germanium(II) ate complex $[Ph_3P^iPr][Ge(OAc)_3]$ containing a discrete $[Ge(OAc)_3]^{(-)}$ anion containing monodentate acetate ligands with a trigonal pyramidal germanium centre.

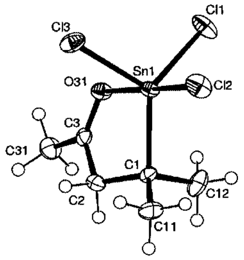


V. N. Khrustalev*, M. Yu. Antipin, N. N. Zemlyansky, I. V. Borisova, Yu. A. Ustynyuk, V. V. Lunin and K. Izod 360–362

The germanium(II) ate complex $[Ph_3P^iPr][Ge(OCOMe)_3]$: the first structurally characterized compound containing a discrete $[E^{14(II)}O_3]^{(-)}$ ($E^{14(II)}$ = Si, Ge, Sn or Pb) anion

---

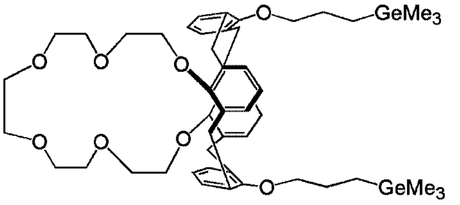
Ab initio calculations have been carried out on the structures of $X_3SnCR_2CH_2COMe$ (X = halide; R = H (**3**) or R = Me (**2**)) and compared with those determined by X-ray crystallography for **2** (X = Cl, I).



B. F. Milne, R. P. Pereira, A. M. Rocco, J. M. S. Skakle, A. J. Travis, J. L. Wardell* and S. M. S. V. Wardell 363–371

Experimental and ab initio *structural study of the ketotin compounds, $X_3SnCR_2CH_2COMe$: crystal structures of $X_3SnCMe_2CH_2COMe$ (X = Cl and I)*

With the expectation of preparing a new type of heteroditopic host where a germanium-containing side chain acts as an anion host, a calixcrown, **1a**, modified with germanium-containing side chains was prepared. For reference, the carbon analog **1b** was also prepared. Both **1a** and **1b** showed much the same cation transport ability, indicating that the designed structure of **1a** is not appropriate to establishing the role of germanium in cation transportation.



T. Sakurai and Y. Takeuchi* .. 372–376

Synthesis of a calix[4]crown modified with germanium-containing side chains

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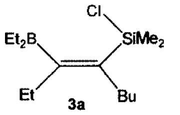
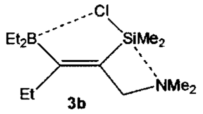
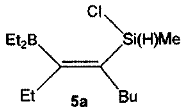
Alkyn-1-yl-chloro(methyl)silanes (**1**, **2**) react with triethylborane by 1,1-ethylboration to give mainly alkenes with (*E*)-configuration (**3**, **5**), in which the boryl and silyl groups occupy cis positions. NMR data indicate intramolecular N–Si coordination and the formation of weak Si–Cl–B bridges in **3b** and **5b**.



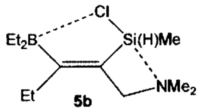
3a



3b



5a



5b

B. Wrackmeyer*, K. Shahid and S. Ali 377–382

1,1-Ethylboration of alkyn-1-yl-chloro-(methyl)silanes: alkenes with chloro(methyl)silyl and diethylboryl groups in cis positions

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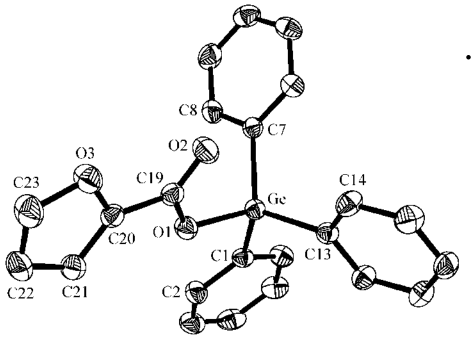
The germanium atom in the title compound is in a distorted tetrahedral geometry defined by an oxygen donor, derived from an effectively monodentate carboxylate ligand, and three *ipso*-carbon atoms from the phenyl substituents.



H.-D. Yin* and C.-H. Wang 383–383

Crystallographic report: Crystal and molecular structure of $Ph_3GeO_2CC_4H_3O$

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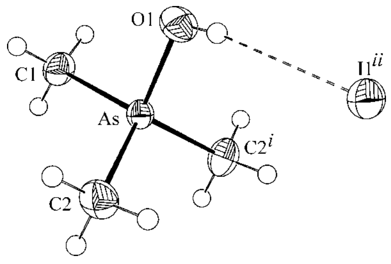
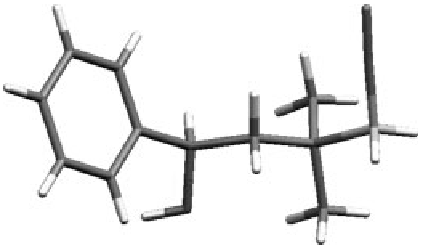
Hydroxytrimethylarsonium iodide, $[Me_3AsOH]I$, was obtained from the reaction of Me_2AsI and MeI in strong basic aqueous solution. The arsenic atom, lying on a mirror plane, is surrounded by one OH and three Me groups, forming a tetrahedral centre.



B. O. Patrick, H. Sun, M. W. Fricke and W. R. Cullen* 384–385

Crystallographic report: Hydroxytrimethylarsonium iodide, [Me$_3$AsOH]I

The structure of the new amine cyanoborane; dimethyl-2-phenylethan-2-olamine cyanoborane, reveals that the geometry around the boron atom is tetrahedral and the B–C≡N moiety has bent geometry.



K. Takrouri, E. Shalom, I. Goldberg, J. Katzhendler and M. Srebnik* 386–387

Crystallographic report: Dimethyl-2-phenylethan-2-olamine cyanoborane

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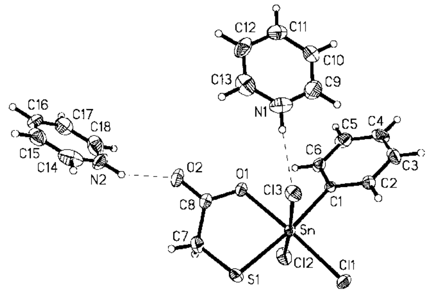
The tin atom in the anion of $[C_5H_5NH]_2$ $[PhSn(SCH_2COO)Cl_3]$ is six-coordinated within an octahedral geometry defined by a CCl_3OS donor set; the cations and anion interact via hydrogen bonds.



G.-Y. Zhong, H.-B. Song, L.-J. Sun and Q.-L. Xie* 388–389

Crystallographic report: Dipyridium mercaptoacetotrichlorophenylstannate

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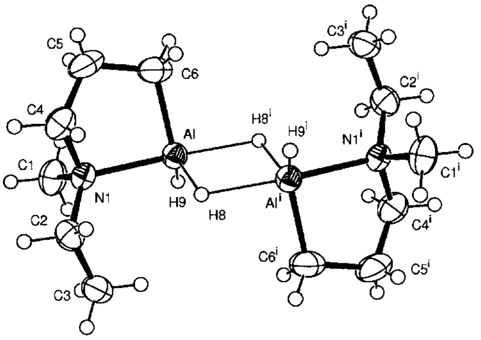
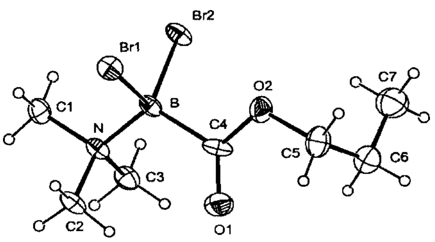
Dimeric and centrosymmetric $[MeEtN(CH_2)_3AlH_2]_2$ comprises aluminum centers, coordinated in a distorted trigonal bipyramidal fashion by three hydrogen atoms, one nitrogen atom and one carbon atom. The aluminum atoms are bridged by hydrogen atoms, creating a planar, four-membered Al_2H_2 ring.



E. Hecht* 390–390

Crystallographic report: [N,N-Methylethylaminopropylalane]$_2$

The conformation around the boron atom is nearly tetrahedral, with Br–B–Br bond angles of 109.14(19)° and slightly wider N–B–C bond angles up to 113.6(3)°.



E. Shalom, K. Takrouri, I. Goldberg, J. Katzhendler and M. Srebnik* 391–392

Crystallographic report: Trimethylamine-dibromopropoxycarbonylborane

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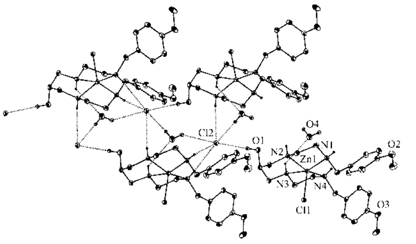
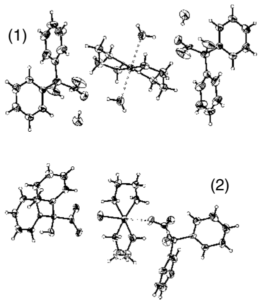
The zinc(II) center in the molecule of [$(C_{23}H_{36}N_4O_3)ZnCl$]ClH_2O is coordinated by four nitrogen atoms of HL (1,3-bis[2-[2-[(4-methoxybenzyl) amino]ethylamino]]-2-propanol) and one chloro anion. The coordination moieties are connected by hydrogen bonds to form a one-dimensional structure.



X. Liu, Y. Xie and Q. Liu* 393–393

Crystallographic report: Chloro[1,3-bis[2-[2-[4-methoxybenzyl] amino]ethylamino]]-2-propanol]zinc(II) chloride hydrate, [$(C_{23}H_{36}N_4O_3)ZnCl$]ClH_2O

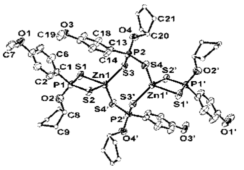
Compounds [$Cu(en)_2(OH_2)_2$]$(HB)_2\cdot 2H_2O$ (**1**) and [$Cu(HB)(en)_2(OH_2)$](HB) (**2**) (HB = benzilate; en = ethylenediamine) present the Cu(II) atom coordinated by different N_4O_2 donor sets, with 4 + 2 and 4 + 1 + 1 coordination geometries, respectively. In compound **1** the Cu(II) atom is located on a centre of inversion.



R. Carballo*, B. Covelo, E. García-Martínez, and E. M. Vázquez-López 394–395

Crystallographic report: Benzilate of diaquabis(ethylenediamine)copper(II) dihidrate and Benzilate of aquabenzilatobis(ethylenediamine)copper(II)

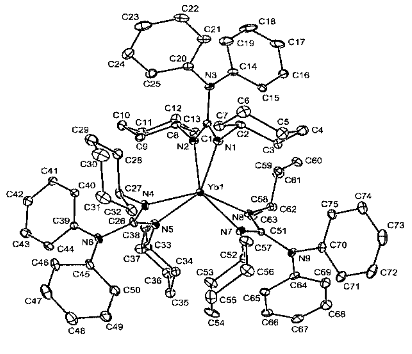
The centrosymmetric complex is a binuclear complex with an eight-membered central ring that consist of two (PS_2) moieties from ligand molecules plus two Zn(II) cations.



M. Karakus*, H. Yilmaz, E. Bulak and P. Lönnecke 396–397

Crystallographic report: Bis{μ-[O-cyclopentyl(4-methoxyphenyl)dithiophosphonato]1κ:S,2κ:S-[O-cyclopentyl(4-methoxyphenyl)dithiophosphonato]1κ^2 S,S'}dizinc(II)

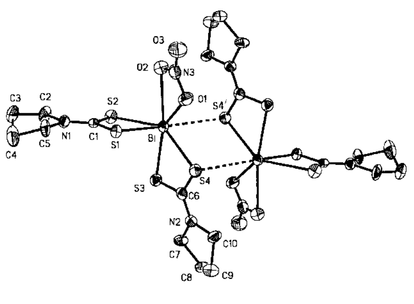
The title complex, [$Ph_2NC(NCy)_2$]$_3Yb\cdot 2$ $PhCH_3$ is a monomer with a six-coordinate ytterbium center ligated by six nitrogen atoms of three chelating bidentate guanidinate ligands. The coordination geometry around the lanthanide ion is best described as a distorted trigonal prism.



L. Zhou, Y. Yao, Y. Zhang, H. Sheng, M. Xue and Q. Shen* 398–399

Crystallographic report: Crystal structure of a homoleptic lanthanide guanidinate complex: [$Ph_2NC(NCy)_2$]$_3Yb\cdot 2PhCH_3$

The centrosymmetric structure of [$Bi(NO_3)(S_2CNC_4H_8)_2$]$_2$ features chelating dithiocarbamate and nitrate ligands, as well as intermolecular Bi–S interactions, so that a distorted pentagonal bipyramidal S_5O_2 coordination geometry results.



H. D. Yin* and C. H. Wang ... 400–400

Crystallographic report: Bis[(nitro)bis(dithiotetrahydropyrrolocarbamato)bismuth(III)]

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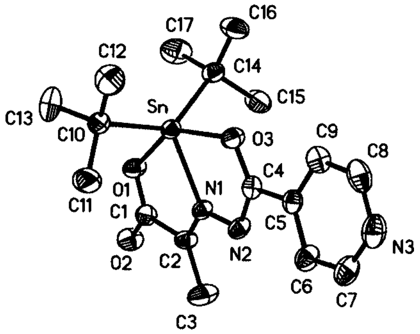

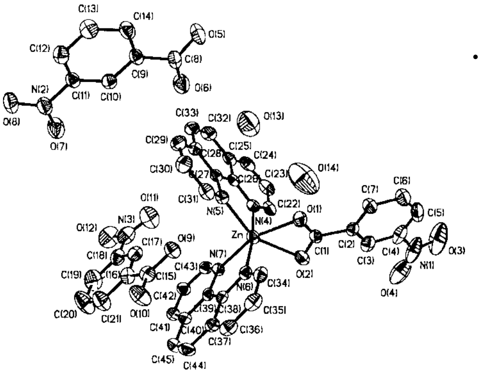

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