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Contents

Papers

Neil A. Law, Wolfgang Dietzsch, Norman V. Duffy

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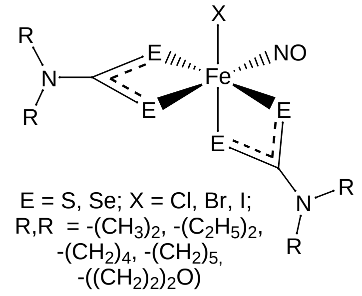
A multinuclear (^{1}H, ^{13}C, ^{15}N) NMR study of *cis*-halonitrosylbis(dithiocarbamato)iron(II) complexes: effect of replacement of S by Se

^{1}H and $\{^1H\}^{13}C$ NMR spectra were measured for several *cis*-$Fe(E_2CNR_2)_2X$ complexes (R = organic substituent; E = S,Se; X = Cl^-, I^-, Br^-), and the natural abundance ^{15}N NMR spectrum of *cis*-$Fe(S_2CN(C_2H_5)_2)_2I$. The data indicate structural rigidity for *cis*-$Fe(E_2CNR_2)_2X$ in solution and are compared to spectra from structurally similar *cis*-$Fe(S_2CNR_2)_2(CO)_2$ complexes. The α-^{13}C NMR signals ($E_2CN(CH_2\text{-}R)_2$) depend on the proximate chalcogen and the orientation of the other first-coordination sphere donor atoms.



Mohammad Akbar Ali, A.H. Mirza, C.W. Voo, Ai Ling Tan, P.V. Bernhardt

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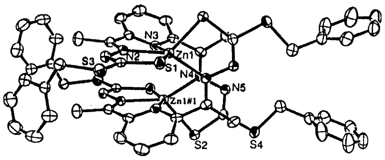
The preparation of zinc(II) and cadmium(II) complexes of the pentadentate N_3S_2 ligand formed from 2,6-diacetylpyridine and *S*-benzyldithiocarbazate (H_2SNNNS) and the X-ray crystal structure of the novel dimeric $[Zn_2(SNNNS)_2]$ complex

The complexes, [M(SNNNS)] (M=Zn^{2+}, Cd^{2+}; $SNNNS^{2-}$ = doubly deprotonated form of the 2,6-diacetyl-pyridine Schiff base of S-benzyldithiocarbazate) have been prepared and characterized. An X-ray structure analysis shows that the zinc(II) complex is a dimer in which the pyridine nitrogen, the azomethine nitrogen and the thiolate sulfur atoms of one ligand coordinate to one of the zinc(II) ions whereas the azomethine nitrogen and thiolate sulfur atoms from another ligand complete pentacoordination around the zinc(II) ion.



Herbert B. Silber, Victor Maraschin, Scott Sibley, Claudia Richter, Nadira Arif, Larry Contreras, Peter Djurovich, Toby Ratansiripong, Jon Stoddard

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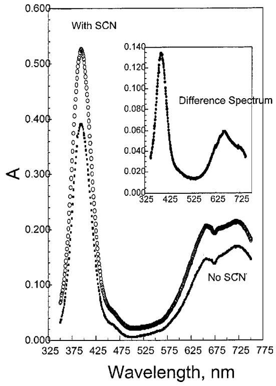
Spectrophotometric investigations of the complexation between Ni(II) and thiocyanate in aqueous methanol

The addition of NaSCN to $Ni(ClO_4)_2$ solutions changes the extinction coefficients for the complex compared to the free Ni(II) ion, indicating the absence of any geometry change in aqueous methanol. Equilibrium constants as a function of solvent and temperature were obtained.



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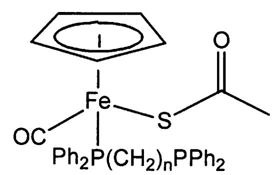
Mohammad El-khateeb, Ibrahim Jibril, Hisham Barakat, Gerd Rheinwald, Heinrich Lang

Polyhedron 22 (2003) 3445

Controlled synthesis of mono-substituted diphosphine iron thiocarboxylate complexes $CpFe(CO)(Ph_2P(CH_2)_nPPh_2)SCOR$ [$n = 1$ (dppm), $n = 2$ (dppe)]. X-ray crystal structure of $CpFe(CO)(dppm\text{-}S)SCO\text{-}3,5\text{-}(NO_2)_2C_6H_3$

Controlled photolytic CO-substitution reaction of the organoiron thiocarboxylate complexes $CpFe(CO)_2SCOR$ ($R = CH_3$, 2-NO_2 C_6H_4, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$) with diphosphines ($Ph_2P(CH_2)_nPPh_2$) [$n = 1$ (dppm), $n = 2$ (dppe)] at 0 °C using 1:1 (metal:ligand) molar ratio afforded exclusively the monosubstituted complexes $CpFe(CO)(Ph_2P(CH_2)_nPPh_2)SCOR$. The complex CpFe(CO) ($Ph_2$ $PCH_2P = S(Ph)_2$) SCO-3,5-$(NO_2)_2C_6H_3$ has been prepared and its X-ray structure was determined.



Václav Haber, Ivana Císařová, Jan Fabry

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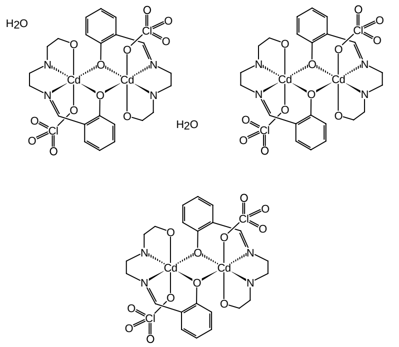
X-ray structure determination of bis{(-[*N*-(2-[2-hydroxyethylamino]ethyl)salicylaldiminato]-perchlorate cadmium(II))}monohydrate ([(Cd sadol $ClO_4)_2] \cdot H_2O$) and two modifications of bis{(-[*N*-(2-[2-hydroxyethylamino]ethyl)salicylaldiminato]-perchlorate cadmium (II))} ([(Cd sadol $ClO_4)_2$])

Preparations and structures of three related Cd-complexes are reported. Two of these structures differ by content of water that can be reversibly released and absorbed depending on outer conditions. The water-containing compound is the first case of a Cd-perchlorate complex not coordinated by water though it is present in the structure. The water-free structures are polymorphs.

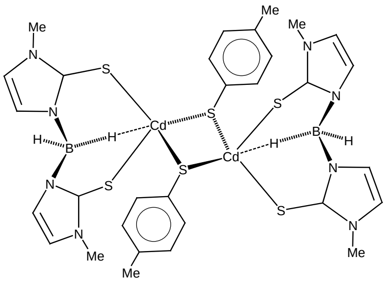


Lauren A. Philson, Dania M. Alyounes, Lev N. Zakharov, Arnold L. Rheingold, Daniel Rabinovich

Polyhedron 22 (2003) 3461

Bis(mercaptoimidazolyl)borates and the control of nuclearity in cadmium thiolate complexes

The bis(mercaptoimidazolyl)borate cadmium (II) thiolate complexes $(Bm^{Me})CdSR$ ($R = Ph$, *p*-Tol, C_6F_5) have been cleanly prepared by metathesis of $(Bm^{Me})CdBr$ and the corresponding thallium(I) thiolates Tl(SR). These new compounds have been characterized by a combination of analytical and spectroscopic techniques and, in the case of the thiocresolate derivative, by X-ray crystallography.



Lin Rong-Guang, Tao Zhu, Xue Sai-Feng, Zhu Qian-Jiang, W. Gregory Jackson, Wei Zhan-Bing, Long La-Sheng

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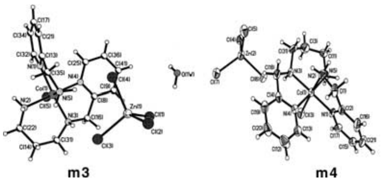
C–H$\cdots\pi$ interactions in the $[Co(N\text{-}(2\text{-pyridylmethyl)-1,3-diaminopropane)(2-aminomethylpyridine)Cl}]^{2+}$ system: syntheses, 2D NMR spectroscopy, X-ray structures and energy minimisations

Two of the 10 possible asymmetric $[Co(ptma)(ampy)X]^{2+}$ ($X = Cl^-$, OH^-) isomers have been synthesized and their structures deduced from 2D ^1H NMR studies and confirmed by single crystal X-ray diffraction work. The ab initio calculations support the idea that the two most stable isomers shown are each characterized by a significant C–H$\cdots\pi$-aromatic interaction.

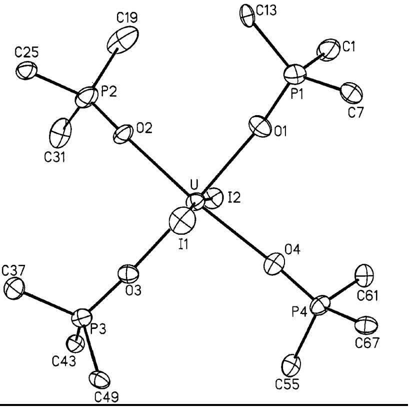


Jean-Claude Berthet, Martine Nierlich, Michel Ephritikhine

Polyhedron 22 (2003) 3475

A comparison of analogous 4f- and 5f-element compounds: syntheses and crystal structures of triphenylphosphine oxide complexes of lanthanide(III) and uranium(III) triflates and iodides $[MX_2\ (OPPh_3)_4][X]$ (X = OTf and M = Ce or U; X = I and M = Nd, Ce, La, U)

Crystal structures of the title compounds show that the differences in the M–O distances reflect the variation in the ionic radii of the metals whereas the U–I distances are shorter than the lanthanide–iodide bond lengths.

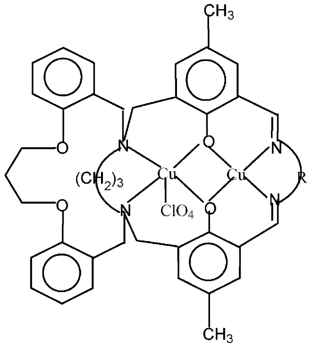


P. Akilan, M. Thirumavalavan, M. Kandaswamy

Polyhedron 22 (2003) 3483

Synthesis of mono and binuclear copper(II) complexes using new macrobicyclic tricompartmental unsymmetrical ligands: magnetic, electrochemical and catalytic studies

A series of lateral macrobicyclic compartmental ligands and their mono and binuclear copper(II) complexes were synthesized. Spectral, magnetic, electrochemical and catalytic studies of the complexes were carried out. Magnetic studies indicate that anti-ferromagnetic coupling is taking place in binuclear copper(II) complexes. Cyclic voltammetric studies of the complexes show quasireversible one-electron reduction waves in cathodic region. The catalytic activities of the complexes towards catechol oxidation increase with macrocyclic ring size. The more distortion in the geometry around the metal centres due to increase in ring size is evidenced by spectral, electrochemical and catalytic studies.

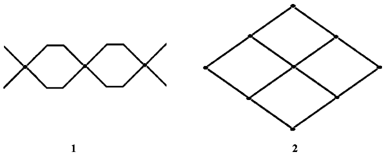


Chun-Hua Ge, Xiang-Dong Zhang, Peng Zhang, Wei Guan, Fang Guo, Qi-Tao Liu

Polyhedron 22 (2003) 3493

Construction of coordination polymers by different conformational flexible pyridyl-type ligands and copper (II) ions

Two coordination polymers $\{[Cu(L)_2(H_2O)]\cdot(ClO_4)_2\cdot(H_2O)_6\}$ (**1**), $\{[Cu(L)_2(ClO_4)]\cdot(ClO_4)\}$ (**2**) (L = 1,2-bis(4-pyridinecarboxamido)ethane have been synthesized and structurally determined; the one- and two-dimensional coordination motifs are constructed by different conformations of L.

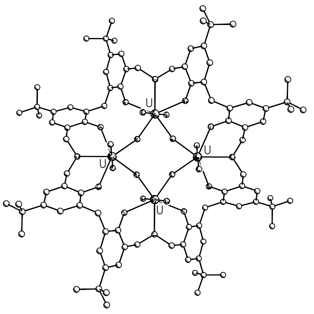


Pierre Thuéry, Bernardo Masci

Polyhedron 22 (2003) 3499

Versatility of large homooxacalixarenes as ligands in polymetallic complexes: the case of UO_2^{2+} and *p-tert*-butyloctahomotetraoxacalix[8]arene

Depending upon the experimental conditions used, different polynuclear uranyl ion complexes of *p-tert*-butyloctahomotetraoxacalix[8]arene can be obtained. Tetranuclear clusters bridged by oxygenated ligands can be complexed either by eight phenoxide groups or by eight phenol and four ether ones, whereas the use of pyridine prevents the formation of clusters and results in a dinuclear complex in which the two uranyl moieties are isolated from each other.

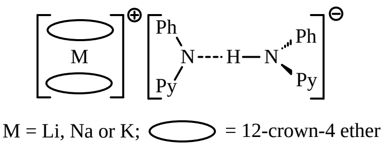


Stephen T. Liddle, William Clegg

Polyhedron 22 (2003) 3507

A homologous series of crown-ether-complexed alkali metal amides as discrete ion-pair species: synthesis and structures of $[M(12\text{-crown-}4)_2][PyNPh \cdot PyN(H)Ph]$ (M = Li, Na and K)

Attempts to produce [(12-crown-4)M(L)], where L^- is the 2-phenylamidopyridine ligand and M = Li, Na, K, have produced instead the salts $[(12\text{-crown-}4)_2M]^+[LH{\cdot}L]^-$, containing discrete crown-encapsulated alkali metal cations and amide anions linked to parent amine molecules by hydrogen bonds, irrespective of the stoichiometry of reagents used in the syntheses.

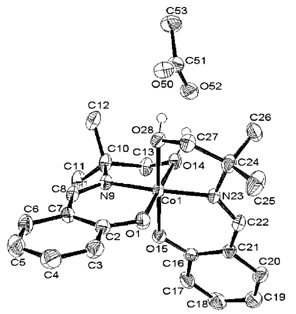


Mishtu Dey, Chebrolu P. Rao,
Pauli K. Saarenketo, Kari Rissanen,
Erkki Kolehmainen, Philippe Guionneau

Polyhedron 22 (2003) 3515

Mn(IV) and Co(III)-complexes of –OH-rich ligands possessing O_2N, O_3N and O_4N cores: syntheses, characterization and crystal structures

Mn(IV) and Co(III) complexes of tridentate –OH-rich ligands possessing O_2N, O_3N and O_4N donor set were synthesized, characterized and their structures were established by single crystal X-ray diffraction, where the binding core is O_4N_2. In all the structurally characterized complexes, the coordination geometry about the metal ion was found to be distorted octahedral.

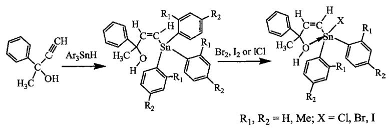


Dong-Sheng Zhu, Ze-Min Mei,
Chun-Sheng Lü, Wei Gao, Yue-Tao Zhang,
Ying Mu, Zong-Mu Wang

Polyhedron 22 (2003) 3523

Synthesis, characterization and crystal structures of (*Z*)-1-(triarylstannyl)-3-phenyl-1-buten-3-ols and their arylhalostannyl derivatives

(*Z*)-1-(Tri-*o*-tolylstannyl)-3-phenyl-1-buten-3-ol (**1**) and (*Z*)-1-(tri-*p*-tolylstannyl)-3-phenyl-1-buten-3-ol (**2**) were synthesized by the additive reaction of 3-phenyl-1-butyn-3-ol with tri-*o*-tolyltin and tri-*p*-tolyltin hydride. One of the aryl groups in compound **1** and **2** was substituted by Cl, Br, I to yield derivatives of the type $PhC(CH_3)(OH)CHCHSn(aryl)_2X$ [aryl = *o*-tolyl, X = Cl (**3**); aryl = *p*-tolyl, X = Cl (**4**); aryl = *o*-tolyl, X = Br (**5**); aryl = *p*-tolyl, X = Br (**6**); aryl = *o*-tolyl, X = I (**7**); aryl = *p*-tolyl, X = I (**8**)].

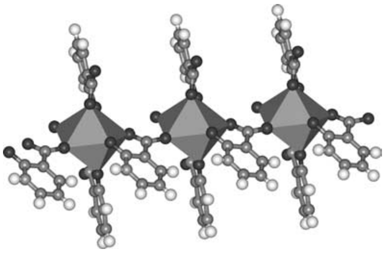


Paula C.R. Soares-Santos,
Helena I.S. Nogueira, João Rocha,
Vitor Félix, Michael G.B. Drew,
Rute A. Sá Ferreira, Luis D. Carlos,
Tito Trindade

Polyhedron 22 (2003) 3529

Lanthanide complexes of 2-hydroxynicotinic acid: synthesis, luminescence properties and the crystal structures of $[\mathrm{Ln(HnicO)_2(\mu\text{-}HnicO)(H_2O)}]\cdot n\mathrm{H_2O}$ (Ln = Tb, Eu)

The 2-hydroxynicotinate ligand coordinates through *O,O*-chelation to lanthanide(III) ions (Eu, Gd, Tb, Er,Tm). Photoluminescence measurements were performed for the Eu(III) and Tb(III) complexes and the energy transfer mechanisms between the lanthanide ions and the ligand are discussed.



Zbynek Janousek, Josef Holub,
Drahomır Hnyk,
Michael G.S. Londesborough,
Richard K. Shoemaker

Polyhedron 22 (2003) 3541

Synthesis of the first 11-vertex *arachno*-dicarbathiaborane anion, $[1,6,7\text{-}\mathrm{C_2SB_8H_{11}}]^-$. Theoretical refinement of its structure

The addition of elemental sulphur to the 10-vertex *nido* dicarborane $[6,9\text{-}\mathrm{C_2B_8H_{10}}]^{2-}$(**2**) engenders the new 11-vertex species $[1,6,7\text{-}\mathrm{C_2SB_8H_{11}}]^-$ (**2**), which has been structurally characterised using the ab initio/GIAO/NMR method and by deductions made from observations on its reaction chemistry.