

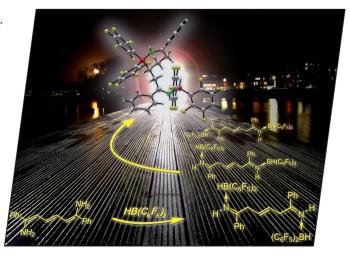


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# **COVER PICTURE**

The cover picture shows a sketch of the reaction of a rare example of a stable conjugated primary enamine with Piers' borane that initially leads to the formation of the borane-stabilized imine tautomers, which subsequently rearrange to the hydroboration product whose favoured imine—borane coordinated structure was characterized by X-ray diffraction. Details are discussed in the article by G. Erker et al. on p. 849ff.



## SHORT COMMUNICATION

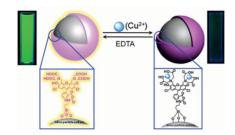
#### Cu Chemosensors

S. Seo, H. Y. Lee, M. Park, J. M. Lim, D. Kang,\* J. Yoon,\* J. H. Jung\* ...... 843-847



Fluorescein-Functionalized Silica Nanoparticles as a Selective Fluorogenic Chemosensor for Cu<sup>2+</sup> in Living Cells

**Keywords:** Sensors / Copper / Fluoresence / Nanoparticles / Silica



The optical binding ability of fluoresceinfunctionalized silica nanoparticles to heavy metal ions was investigated in aqueous solution. These nanoparticles act as a new type of synthetic fluorogenic chemosensor for imaging Cu2+ ions in living cells.

# **FULL PAPERS**

## **Hydroboration**

I. Peuser, R. Fröhlich, G. Kehr, G. Erker\* ...... 849-853

Reaction of a Stable Conjugated Primary Enamine with Piers' Borane

Keywords: Lewis acids / Tautomerization / Hydroboration / Boron / Enamine

Ph 
$$\rightarrow$$
 Ph  $\rightarrow$  P

The stable conjugated primary enamine Ph-C(NH<sub>2</sub>)=CH-CH=CH-CH=C-(NH<sub>2</sub>)-Ph (3) reacts with [HB(C<sub>6</sub>F<sub>5</sub>)<sub>2</sub>] by tautomerization and internal hydroboration to yield the heterocyclic product 6.

## **Polarized Phosphaalkenes**

V. B. Gudimetla, L. Ma,

M. P. Washington, J. L. Payton,

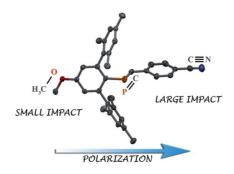
M. C. Simpson,

J. D. Protasiewicz\* ...... 854-865



meta-Terphenyl Phosphaalkenes Bearing Electron-Donating and -Accepting Groups

Keywords: Phosphorus / Phosphaalkenes / Solid-state structures / Electrochemistry

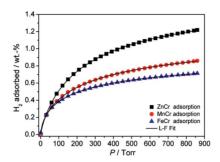


Substituted meta-terphenyl phosphaalkenes have been synthesized to allow systematic studies of the impact of remote substituents on the phosphaalkene unit. Introduction of remote groups on the less hindered phenyl ring generated more significant effects on the physical properties of the materials than did substituents on the hindered meta-terphenyl rings.



### **Hvdrogen Storage Materials**

Three porous Prussian blue analogues  $KM[Cr(CN)_6]$  (M = Zn, Mn, Fe) have been synthesized and characterized structurally. The thermal stabilities and permanent porosities were investigated by thermal gravimetric analysis, variable-temperature powder XRD experiments and gas sorption measurements. Hydrogen sorption studies demonstrated higher H<sub>2</sub> uptake capacities for the porous materials at 77 K.



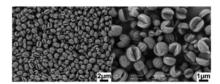
A.-H. Yuan,\* C.-X. Chu, H. Zhou, P. Yuan, K.-K. Liu, L. Li, O.-F. Zhang, X. Chen, Y.-Z. Li ...... 866-871

Syntheses, Crystal Structures and Gas Sorption Properties of Prussian Blue Analogues Constructed from [Cr(CN)<sub>6</sub>]<sup>3</sup> **Building Blocks** 

Keywords: Microporous materials / Prussian blue analogues / Structure elucidation / Adsorption

## **γ-AlOOH Superstructures**

Three-quarter-sphere-like γ-AlOOH was synthesized on the basis of the layered hydroxide structure. The decomposition of urea and acidity played important roles in the morphology of the γ-AlOOH products, which have potential application in removal of hexavalent chromium pollutants from toxic solutions and reduction of the amount of tobacco-specific nitrosamines (TSNAs) in cigarette smoke.



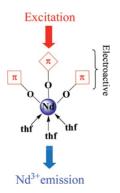
Y. Zhu, H. W. Hou,\* G. L. Tang, Q. Y. Hu\* ...... 872-878

Synthesis of Three-Ouarter-Sphere-Like γ-AlOOH Superstructures with High Adsorptive Capacity

**Keywords:** γ-AlOOH / Hydrothermal synthesis / Layered compounds / Chromium / **Nitrosamines** 

## Two novel mononuclear neodymium methoxides containing thienyl substituents have been synthesized and structurally characterized. The physico-chemical properties of a series of rare earth thienyl-substituted methoxides are presented. The luminescence spectra of the neodymium alkoxides reveal an energy transfer from the

ligand to the metal centre.



## **Luminescent Lanthanide Alkoxides**

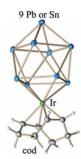
M. Veith,\* C. Belot, V. Huch, H. L. Cui, L. Guvard, M. Knorr, C. Wickleder ...... 879-889

Synthesis, Crystal Structure and Physico-Chemical Studies of Neodymium and Erbium Methoxides Containing Thienyl Substituents

**Keywords:** Lanthanides / Electrochemistry / Luminescence / Thienyl methoxides

## **Bimetallic Clusters**

The ions  $[Sn_9Ir(cod)]^{3-}$  and  $[Pb_9Ir(cod)]^{3-}$ (cod = 1,5-cyclooctadiene) were synthesized and the reactions of the labile Ir complexes with Sn<sub>9</sub><sup>4-</sup> and Pb<sub>9</sub><sup>4-</sup> studied. Both clusters possess a C<sub>4v</sub>-type closo-deltahedral structure with 22 electrons and are diamagnetic. The clusters described are the first Ir-Sn and Ir-Pb bimetallic clusters and are examples of homologous Sn-Pb transition-metal derivatives.



D. O. Downing, P. Zavalij, B. W. Eichhorn\* ...... 890-894

The closo- $[Sn_9Ir(cod)]^{3-}$  and  $[Pb_9Ir(cod)]^{3-}$ Zintl Ions: Isostructural IrI Derivatives of the *nido*- $E_9^{4-}$  Anions (E = Sn, Pb)

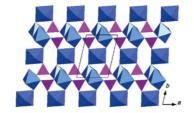
Keywords: Zintl anions / Cluster compounds / Structure elucidation / Charge transfer / Solid-state structures

# **CONTENTS**

### **Magnetic Metal Phosphonates**

Pillared Layered Metal Phosphonates Showing Field-Induced Magnetic Transitions

**Keywords:** Copper / Cobalt / Phosphonates / Layered compounds / Metamagnetism / Spin flop



Metal phosphonates  $M_3(pna)_2(H_2O)_2$  {1:  $M = Cu^{II}$ , 2:  $M = Co^{II}$ ;  $pnaH_3 = 6$ -phosphononicotinic acid} with pillared layered structures are reported. Compound 1 features a spin flop transition, whereas 2 shows metamagnetic behavior.

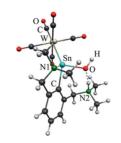
## **Organotin Cations**

R. Jambor,\* B. Kašná, S. G. Koller, C. Strohmann,\* M. Schürmann,

K. Jurkschat\* ...... 902-908

[{2,6-(Me<sub>2</sub>NCH<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>(H<sub>2</sub>O)Sn}W(CO)<sub>5</sub>]<sup>+</sup>-CB<sub>11</sub>H<sub>12</sub><sup>-</sup>: Aqua Complex of a Transition-Metal-Bound Organotin(II) Cation versus an Ammonium-Type Structure

**Keywords:** Tin / Tungsten / Cations / Structure elucidation / X-ray diffraction / Density functional calculations



Intramolecular N→Sn coordination stabilizes the transition-metal-bound tin(II) cation. However, this coordination can easily be broken by a water molecule to give the title compound. This implies that the coordination of organic substrates should also be possible and that this compound has the potential for catalysis.

### **Switch of Coordination Mode**

Zinc(II), Copper(II), and Nickel(II) Complexes of Bis(tripodal) Diamide Ligands — Reversible Switching of the Amide Coordination Mode upon Deprotonation

**Keywords:** Tripodal ligands / Dimetallic complexes / N,O ligands / Amides / Coordination modes

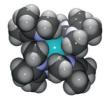
Dinuclear complexes of type 1 ( $M = Zn^{II}$ ,  $Cu^{II}$ ,  $Ni^{II}$ ) rearrange upon deprotonation of the amide N atoms of the dinucleating ligand to give complexes 2. In the rearrangement the coordination chemistry of one metal atom remains unchanged while

the second one becomes coordinated by the two amidate nitrogen atoms generated by deprotonation and the aliphatic amine donors. The deprotonation/rearrangement reaction is reversibel.

## **Hydridoruthenium Complexes**

Neutral and Cationic Hydridoruthenium Tetrakiscarbene Complexes

**Keywords:** Ruthenium / Hydrides / Carbene ligands

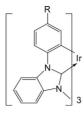


Carbene-supported, neutral or cationic hydridoruthenium complexes can be prepared selectively by using LiAlH<sub>4</sub> or LiBHEt<sub>3</sub> as the hydride source. Neutral *trans*-[RuH<sub>2</sub>(IMe)<sub>4</sub>] (2) has two hydrido ligands in a *trans* arrangement, while cationic [RuH(IMe)<sub>4</sub>]<sup>+</sup> (3) features a vacant coordination pocket, which renders this complex very attractive for the binding and activation of small molecules.



**Blue Phosphorescent Ir Complexes** 

Facial and meridional isomers of tris(4-R-phenyl)benzimidazolinato  $Ir^{III}$  complexes (R = H, CF<sub>3</sub>, CN, OCH<sub>3</sub>) were prepared. Some of them showed efficient pure blue emission in thf. Their quantum yields, lifetimes, and oxidation potential energies were measured. In addition, X-ray single crystallography and TD-DFT calculations were applied to investigate the structures and energies of complexes.



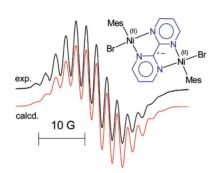
1a, 1b: R = H 2a, 2b: R = CF<sub>3</sub> 3a, 3b: R = CN 4a, 4b: R = OCH<sub>3</sub> [a : fac isomer] [b : mer isomer] K. Tsuchiya, S. Yagai, A. Kitamura, T. Karatsu,\* K. Endo, J. Mizukami, S. Akiyama, M. Yabe ................ 926-933

Synthesis and Photophysical Properties of Substituted Tris(phenylbenzimidazolinato) Ir<sup>III</sup> Carbene Complexes as a Blue Phosphorescent Material

**Keywords:** Phosphorescence / Carbene ligands / OLEDs / Iridium / Density functional calculations

#### **Electron Transfer**

The mono- and binuclear organometallic  $Ni^{II}$  complexes  $[(\mu\text{-bpym})\{Ni(Mes)Br\}_n]$  can be easily reduced to give the corresponding radical complexes. EPR spectroscopy reveals the almost exclusively ligand-centred unpaired electron and hampered metal-to-ligand overlap for the binuclear complex.

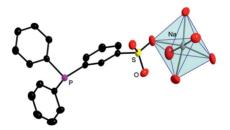


A. Klein,\* A.-K. Schmieder, N. Hurkes, C. Hamacher, A. O. Schüren, M. P. Feth,\* H. Bertagnolli .............................. 934–941

Mono- and Binuclear Arylnickel Complexes of the  $\alpha$ -Diimine Bridging Ligand 2,2'-Bipyrimidine (bpym)

**Keywords:** Nickel / Electrochemistry / EPR spectroscopy / N ligands / Radical complexes / Spectroelectrochemistry

A simple and high-yield synthesis of the monosulfonated triphenylphosphane (TPPMS $^{\rm Na}$ ) sodium salt is described. The solid-state structure of TPPMS $^{\rm Na}$ .2.5H $_2{\rm O}$  was determined.



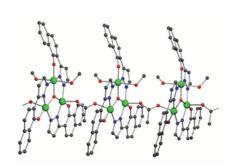
#### Monosulfonated Triphenylphosphane

High-Yield Synthesis and Crystal Structure Determination of Sodium Triphenylphosphane Monosulfonate (TPPMS $^{\mathrm{Na}}$ )

**Keywords:** Phosphane ligands / Surfactants / Sulfur / Structure elucidation

## **Molecular Magnetism**

An oxide-centered trinuclear  $Mn^{III}$  compound,  $[Mn_3(\mu_3\text{-O})(naphpz)_3(O_2CMe)$ - $(MeOH)_2]_m$ , in which the trinuclear units are bridged by acetate ligands, is presented. There are strong  $\pi-\pi$  interactions between the 1-D chains. Overall antiferromagnetic interactions exist between the  $Mn^{III}$  ions of the trinuclear unit and also between the trinuclear units within the 1-D chain and between the chains.



An Oxide-Centered Trinuclear Manganese(III) Compound with a Bulky Phenol-Pyrazolate Ligand

**Keywords:** Manganese / Cluster compounds / Magnetic properties / Bridging ligands / Phenol-pyrazole ligand



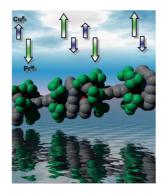
## **CONTENTS**

## 3d-4f Magnetic Chains

F. Pointillart,\* K. Bernot ...... 952-964

Determination of the Nature of Exchange Interactions in the 3d-4f Magnetic Chain  $\{[Cu(salen)Pr(hfac)_3]_2(L)\}_n$  (L = 4,4'-Bipyridine, Pyrazine)

> Keywords: Copper / Nickel / Praseodymium / Crystal field / Magnetic properties



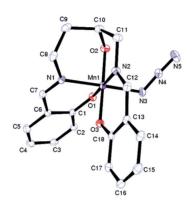
The nature of three magnetic interactions in a Cu<sup>II</sup>-Pr<sup>III</sup> magnetic chain {[{Cu(salen)}- ${Pr(hfac_3)_2(pyz)}](H_2O)_3$  has been determined by using a comparative method. The coordination of a nitrogenated ligand to the PrIII ion changes its crystal field and affords a significant antiferromagnetic coupling between the Cu<sup>II</sup> and Pr<sup>III</sup> atoms.

#### **Reactive Oxygen Scavengers**

V. Daier, D. Moreno, C. Duhayon, J.-P. Tuchagues, S. Signorella\* .... 965-974

Synthesis, Characterization and Combined Superoxide Dismutase and Catalase Activities of Manganese Complexes of 1,4-Bis(salicylidenamino)butan-2-ol

Keywords: Manganese / Superoxide dismutase / Catalase / Biomimetic catalyst / Kinetics / Enzyme catalysis



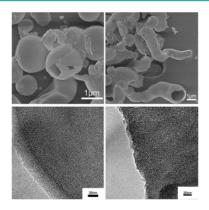
The asymmetric Schiff base ligand 1,4bis(salicylidenamino)butan-2-ol stabilizes Mn<sup>IV</sup> under conditions where related symmetric Schiff bases yield Mn<sup>III</sup> complexes. The ligand arrangement around the metal center and the size of the chelate rings adjust the potential of the Mn<sup>III</sup>/Mn<sup>IV</sup> couple to approximately 400 mV. The accessibility of the Mn<sup>IV</sup> state enables the complex to act as ROS scavenger.

## **Microporous Silica Materials**

M. W. Zhao, Y. N. Gao, L. Q. Zheng,\* W. P. Kang, X. T. Bai, B. Dong .... 975-982

Microporous Silica Hollow Microspheres and Hollow Worm-Like Materials: A Simple Method for Their Synthesis and Their Application in Controlled Release

Keywords: Microporous materials / Nanostructures / Template synthesis / Evaporation method / Ionic liquids

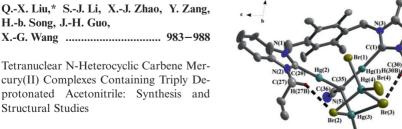


Hollow silica microspheres and hollow worm-like materials with microporous structures were synthesized by utilizing the combination of evaporation and an emulsion template. The morphologies of the product can be modulated by adjusting the temperature or stirring time. Preliminary tests show that the materials may have potential in controlled release.

#### **N-Heterocyclic Carbenes**

H.-b. Song, J.-H. Guo,

X.-G. Wang ...... 983-988



Two funnel-like tetranuclear NHC-mercury(II) complexes [durene(CH2bimynPr)2-Hg<sub>2</sub>(CCN)Hg<sub>2</sub>Br<sub>5</sub>] (2a) and [durene(CH<sub>2</sub> $bimynBu_2Hg_2(CCN)Hg_2Br_5$  (2b) containing triply deprotonated acetonitrile were synthesized and characterized. In the crystal packings of 2a and 2b, the doublestranded 1D supramolecular chains are formed through intermolecular weak interactions.

protonated Acetonitrile: Synthesis and Structural Studies

Keywords: Carbenes / Mercury / Structure elucidation / Nitrogen heterocycles

If not otherwise indicated in the article, papers in issue 5 were published online on February 2, 2010

<sup>\*</sup> Author to whom correspondence should be addressed. Supporting information on the WWW (see article for access details).