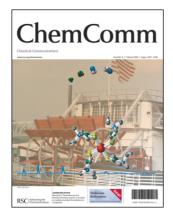
### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (9) 1097-1228 (2005)



#### Cover

Full steam ahead with a hexametallic molecular paddlewheel. See p. 1122. Image reproduced by permission of Stephen P. Jeffery, Jonghyuk Lee and Marcetta Y. Darensbourg



### Inside cover

Additives in bioinspired silicification regulate three key aspects - catalysis, aggregation and scaffolding. See p. 1113. Artwork kindly prepared by Jay Yocis, Photographic Services, University of Cincinnati. Image reproduced by permission of Siddharth V. Patwardhan, Stephen J. Clarson and Carole C. Perry

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C17

Drawing together the research highlights and news from all RSC publications, Chemical Science provides a 'snapshot' of the latest developments across the chemical sciences showcasing newsworthy articles, as well as the most significant scientific advances.

## Chemical Science

March 2005/Volume 2/Issue 3

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### **FEATURE ARTICLE**

1113

### On the role(s) of additives in bioinspired silicification

Siddharth V. Patwardhan,\* Stephen J. Clarson and Carole C. Perry

The results obtained from bioinspired silicification investigations are hypothesised to arise from specific modes of action of the organic additives—additives act as catalysts, aggregation promoting agents or structure-directing agents or typically, exhibit a combination of these behaviours.



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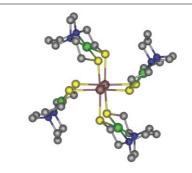
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### 1122

### Metallodithiolato ligands as bridges in multiply bonded dimolybdenum complexes

Stephen P. Jeffery, Jonghyuk Lee and Marcetta Y. Darensbourg\*

For the first time, a NiN<sub>2</sub>S<sub>2</sub> metallothiolate ligand is used as a bidentate bridging ligand for a multiply bonded dimetal unit.



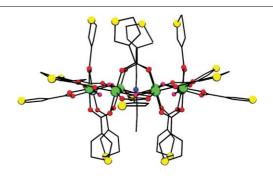
### 1125

### A family of heterometallic wheels containing potentially fourteen hundred siblings

Rebecca H. Laye, Finn K. Larsen, Jacob Overgaard, Christopher A. Muryn, Eric J. L. McInnes,\* Eva Rentschler,\* Veronica Sanchez, Simon J. Teat, Hans U. Güdel, Oliver Waldmann, Grigore A. Timco\* and Richard E. P. Winpenny\*

An extensive series of octanuclear heterometallic wheels has been made of the general formula  $[NR_2H_2][M_7M'F_8(O_2CR')_{16}]$ , where  $M=Cr^{III}$ ,  $Fe^{III}$  or  $V^{III}$ ;  $M'=Ni^{II}$ ,  $Zn^{II}$ ,  $Cd^{II}$ ,  $Co^{II}$ ,  $Fe^{II}$ ; R=a linear alkyl; R'=various. This is the largest family of isostructural polymetallic complexes known since the oxo-centered triangles were first reported.

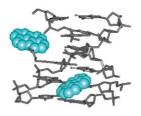
1128



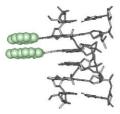
### Fluorometric sensing of the salt-induced B-Z DNA transition by combination of two pyrene-labeled nucleobases

Akimitsu Okamoto,\* Yuji Ochi and Isao Saito\*

We have developed a new fluorescent DNA probe containing two pyrene-labeled nucleobases,  $^{Pet}G$  and  $^{Py}C$ , and the fluorescence color was altered by the salt-induced B-Z DNA transition.



B-DNA in 0.1 M NaCl Pale blue fluorescence



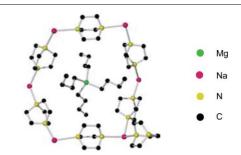
Z-DNA in 4.5 M NaCl Bluish-green fluorescence

### 1131

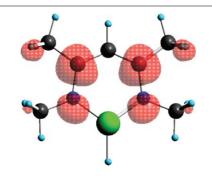
### Stoichiometrically-controlled reactivity and supramolecular storage of butylmagnesiate anions

Prokopis C. Andrikopoulos, David R. Armstrong, Eva Hevia,\* Alan R. Kennedy, Robert E. Mulvey\* and Charles T. O'Hara

Unlike its stoichiometric compatriot Na<sub>2</sub>MgBu<sub>4</sub>, DABCOactivated NaMgBu<sub>3</sub> surprizingly fails to metallate toluene, though both 'ates yield the same coproduct, which has a remarkable polycationic network structure with interstices filled by discrete  $(MgBu_4)^2$  dianions.



### 1134

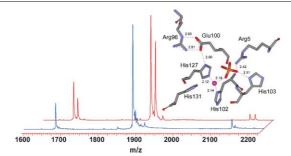


## A computational study of the reactions of a β-diketiminatoaluminium(I) complex with the hydrogen atom and the electron

Iain McKenzie, Paul W. Percival and Jason A. C. Clyburne\*

The  $\beta$ -diketiminatoaluminium(I) complex reacts with the hydrogen atom to produce a radical. The spin density in the resulting radical resides entirely on the  $\beta$ -diketiminato ligand.

### 1137

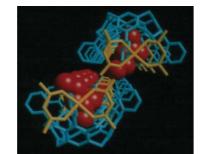


### Metal induced selectivity in phosphate ion binding in E9 DNase

Ewald T. J. van den Bremer, Anthony H. Keeble, Colin Kleanthous and Albert J. R. Heck\*

Insights into fine-tuning mechanisms of endonuclease colicins are investigated by native ESI mass spectrometry. Both  $Ni^{2+}$  and  $Zn^{2+}$  bind to these colicins but only  $Zn^{2+}$  induces subsequent specific phosphate ligation.

### 1140

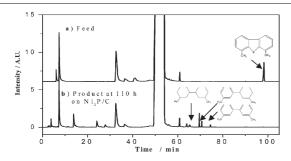


## One-dimensional void-space arrays constructed from a coordination polymer with bowl-like frameworks of cavitands

Makoto Tadokoro,\* Shin Mizugaki, Masatoshi Kozaki and Keiji Okada

A one-dimensional coordination polymer of [Mn<sup>II</sup>(hfac)<sub>2</sub>] bridged by new bowl-like ligands of cavitands was prepared and the crystal structure was determined by X-ray crystal analysis.

### 1143



### A new type of nonsulfide hydrotreating catalyst: nickel phosphide on carbon

Yuying Shu and S. Ted Oyama\*

The carbon-supported nickel phosphide (Ni<sub>2</sub>P/C) is a new promising hydrotreating catalyst and exhibits superior activity, selectivity, and stability for sulfur removal from the refractory compound 4,6-dimethyldibenzothiophene (4,6-DMDBT).

### 1146

## Silica-supported lanthanide silylamides for methyl methacrylate polymerisation: controlled grafting induces controlled reactivity

Régis M. Gauvin\* and André Mortreux

Controlled grafting of lanthanide silylamides onto dehydroxylated silica affords isotactic MMA polymerisation catalysts. Combined analyses allow us to assess the correlation between surface species structure and polymerisation activity and selectivity.

### 1149

### Gi Gi

### Group transfer polymerisation in hydrophobic ionic liquids

Ranganathan Vijayaraghavan and Douglas R. MacFarlane\*

Polymethylmethacrylate is prepared in high yield by group transfer polymerisation in a hydrophobic ionic liquid. The ionic liquid provides an ideal low water content environment for this type of polymerisation of acrylates.

### 1152



### 1,4-Cyclohexadienes as mechanistic probes for the Jacobsen epoxidation: evidence for radical pathways

Ulrike Engelhardt and Torsten Linker\*

Two completely different reaction pathways are followed during the oxidation of 1,4-cyclohexadienes 1. *m*-Chloroperoxybenzoic acid (MCPBA) affords exclusively epoxides 2, whereas cyclohexadienones 3 are formed in the presence of the Jacobsen catalyst, which gives evidence for radical intermediates.

### 1155

Synthesis of deuterium labelled L- and D-glutamate semialdehydes and their evaluation as substrates for carboxymethylproline synthase (CarB)—implications for carbapenem biosynthesis

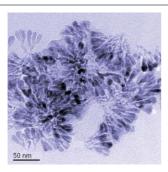
John L. Sorensen, Mark C. Sleeman and Christopher J. Schofield\*

Syntheses of deuterium labelled L- and D-glutamate semialdehydes is reported. Incubation studies reveal that carboxymethylproline synthase (CarB) condenses L-, but not D-glutamate semialdehyde in the committed step of carbapenem biosynthesis.

$$\begin{array}{c} \text{MeO} \\ \text{N} \\ \text{Me} \\ \text{BocNH} \\ t\text{-BuO}_2\text{C} \\ \end{array} \begin{array}{c} \text{1. Cp}_2\text{ZrDCI} \\ \text{2. H}^* \\ \text{HO}_2\text{C} \\ \text{CoA} \\ \text{only} \\ \end{array} \begin{array}{c} \text{CarB} \\ \text{Halonyl- CoASH} \\ \text{HO}_2\text{C} \\ \text$$

1158





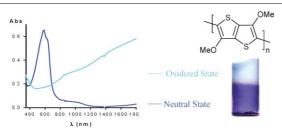
## Morphology-controlled large-scale synthesis of ZnO nanocrystals from bulk ZnO

Xinhua Zhong\* and Wolfgang Knoll\*

Gram-scale, teardrop-like, monodisperse ZnO nanocrystals with a well-resolved absorption onset and a strong sharp UV emission were prepared.

1161



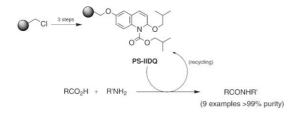


# Poly(3,6-dimethoxy-thieno[3,2-b]thiophene): a possible alternative to poly(3,4-ethylenedioxythiophene) (PEDOT)

Mathieu Turbiez, Pierre Frère,\* Philippe Leriche, Nicolas Mercier and Jean Roncali

Electropolymerisation of the 3,6-dimethoxy-thieno[3,2-b]-thiophene leads to a conjugated polymer with low redox potential, band gap, optical transparency in the doped state and stability similar to those of PEDOT.

1164

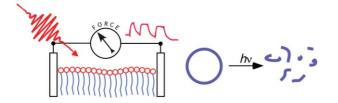


### PS-IIDQ: an efficient polymer-supported amide coupling reagent

Eric Valeur and Mark Bradley\*

Polystyrene-IIDQ is a polymer-supported coupling reagent that couples carboxylic acids to amines in good yields and high purity without the requirement of a pre-activation step.

1167



### Photoinduced morphism of gemini surfactant aggregates

Delphine Faure, Julien Gravier, Thomas Labrot, Bernard Desbat, Reiko Oda\* and Dario M. Bassani\*

The incorporation of a sturdy azobenzene chromophore in a gemini cationic surfactant leads to photoactive amphiphilic assemblies.

### 1170

### Low-melting sugar-urea-salt mixtures as solvents for Diels-Alder reactions

Giovanni Imperato, Ernst Eibler, Julia Niedermaier and Burkhard König\*

Using abundant bulk chemicals, such as sugar, urea and ammonium chloride, low-melting stable mixtures have been obtained, which are suitable as green solvents for organic reactions.



### 1173

### Highly active and selective palladium catalyst for hydroesterification of styrene and vinyl acetate promoted by polymeric sulfonic acids

Hirohito Ooka,\* Tsutomu Inoue, Shinichi Itsuno and Masato Tanaka\*

1,2-Bis(di-tert-butylphosphinomethyl)benzene–palladium species used in conjunction with polymeric sulfonic acids of limited SO<sub>3</sub>H loadings generate high performance catalyst systems in hydroesterification of styrene and vinyl acetate affording branched structured esters.

### 1176

### Methoxycarbonylation of vinyl acetate catalysed by palladium complexes of bis(ditertiarybutylphosphinomethyl)benzene and related ligands

Adam J. Rucklidge, George E. Morris and David J. Cole-Hamilton\*

Palladium complexes of bis(ditertiarybutylphosphinomethyl)benzene are active catalysts for the methoxycarbonylation of vinylacetate to give acetyl protected lactate esters with 78% branched selectivity and minimal side product formation.

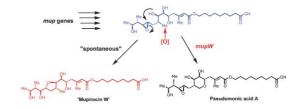
### 1179



### Mupirocin W, a novel pseudomonic acid produced by targeted mutation of the mupirocin biosynthetic gene cluster

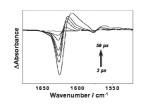
Sian M. Cooper, Russell J. Cox, John Crosby, Matthew P. Crump, Joanne Hothersall, Wanpen Laosripaiboon, Thomas J. Simpson\* and Christopher M. Thomas

Mutation of the mupW gene in the mupirocin biosynthetic gene cluster in Pseudomonas fluorescens results in efficient production of a novel pseudomonic acid metabolite, mupirocin W, which lacks the characteristic tetrahydropyran ring, and reveals the role of the mupW gene in pseudomonic acid biosynthesis.



1182

4



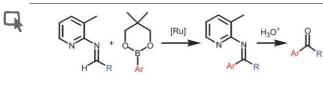
$$\left(\begin{array}{c} N^{M_{2}} \\ N^{-1} \\$$

Monitoring the effect of ultrafast deactivation of the electronic excited states of DNA bases and polynucleotides following 267 nm laser excitation using picosecond time-resolved infrared spectroscopy

Marina K. Kuimova, Joanne Dyer, Michael W. George,\* David C. Grills, John M. Kelly,\* Pavel Matousek, Anthony W. Parker,\* Xue Zhong Sun, Michael Towrie and Aine M. Whelan

A picosecond infrared spectroscopic study of DNA bases has demonstrated that electronic excitation creates vibrationally excited ground state molecules within the first picosecond.

1185

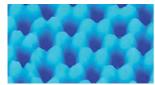


Ruthenium-catalyzed coupling of aldimines with arylboronates: new synthetic method for aromatic ketones

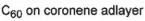
Young Jun Park, Eun-Ae Jo and Chul-Ho Jun\*

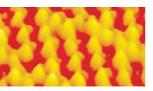
Using the chelation strategy, the reaction of aryl or alkyl aldimines bearing the 3-picolin-2-yl group with various arylboronates in the presence of ruthenium catalyst furnished the corresponding ketimines in high yields for a short reaction time. The resulting ketimines were readily converted to ketones by hydrolysis.

1188



R = aryl or alkyl





C<sub>60</sub> on perylene adlayer

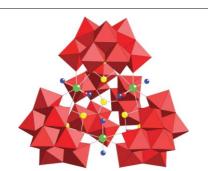
Effect of underlying coronene and perylene adlayers for [60]fullerene molecular assembly

Soichiro Yoshimoto, Eishi Tsutsumi, Oomi Fujii, Ryuji Narita and Kingo Itaya\*

Supramolecularly assembled layers of  $C_{60}$  on coronene- and perylene-modified Au(111) surfaces were investigated and the structure of the  $C_{60}$  adlayer was found to be strongly influenced by the underlying organic layers.

1191





Polyoxoanions functionalized by diorganotin groups: the tetrameric, chiral tungstoarsenate(III),  $[\{Sn(CH_3)_2(H_2O)\}_2\{Sn(CH_3)_2\}As_3(\alpha-AsW_9O_{33})_4]^{21}$ 

Firasat Hussain and Ulrich Kortz\*

The tetrameric, hybrid organic–inorganic tungstoarsenate(III)  $[\{Sn(CH_3)_2(H_2O)\}_2\{Sn(CH_3)_2\}As_3(\alpha-AsW_9O_{33})_4]^{21-}$  is composed of four  $(B-\alpha-AsW_9O_{33})$  fragments that are linked by three dimethyltin groups and three As(III) atoms resulting in an unprecedented, chiral polyoxoanion assembly with  $C_1$  symmetry.

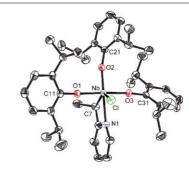
### 1194



Regioselective insertion of 2-vinylpyridine in niobium and tantalum aryloxide complexes: an alternate route to pyridylalkyl metallacycles

Rex A. Corbin,\* Brandon E. Dusick, Khamphee Phomphrai, Phillip E. Fanwick and Ian P. Rothwell

A series of niobium and tantalum aryloxide compounds containing  $\eta^2$  pyridylalkyl ligation have been synthesized *via* a regioselective insertion of 2-vinylpyridine into a metal–hydride bond. The latter originates from <sup>n</sup>BuSnH.



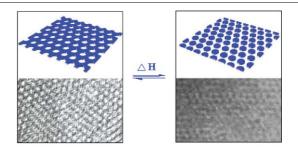
### 1197



### Structural inversion in 3-D hexagonal organization of coil-rod-coil molecule

Long Yi Jin, Jinyoung Bae, Jong-Hyun Ahn and Myongsoo Lee\*

We report an unusual example of supramolecular structural inversion in coil-rod-coil molecules, from organized coil perforations in a rod layer to organized discrete rod-bundles in a coil matrix, while maintaining a 3-D hexagonal superlattice.

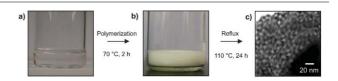


### 1200

### Fabrication of mesoporous polymer using soft template method

Jyongsik Jang\* and Joonwon Bae

Nanoporous polymer materials (see image c) with tunable pore sizes were fabricated from micelle/polymer precursors (see image b) which were prepared by the micelle template method in reverse microemulsion systems (see image a) and the pore size could be tuned by varying the type and concentration of surfactant.



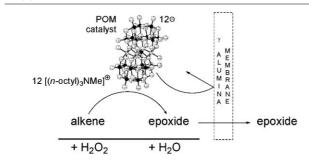
### 1203

### Enhanced reactivity in OH/NH/ $\pi$ polyfunctional systems through coupled proton/electron transfer in the excited state: the photocyclisation of 2-allyl-3-aminophenol

Edgar A. Leo, Rosa Tormos and Miguel A. Miranda\*

The photocyclisation rate of 2-allyl-3-(or 5-)aminophenols (1, 4 and 8) is dramatically enhanced, when compared with reference compounds, as a consequence of a coupled proton/electron transfer process.

1206

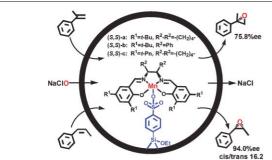


Highly efficient recycling of a "sandwich" type polyoxometalate oxidation catalyst using solvent resistant nanofiltration

Peter T. Witte, Sankhanilay Roy Chowdhury, Johan E. ten Elshof,\* Dorit Sloboda-Rozner, Ronny Neumann and Paul L. Alsters\*

A "sandwich" type polyoxometalate catalyst ([MeN(n-C<sub>8</sub>H<sub>17</sub>)<sub>3</sub>]<sub>12</sub>[WZn<sub>3</sub>(ZnW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]) was very efficiently recycled by nanofiltration with almost quantitative retention, using an  $\alpha$ -alumina supported mesoporous  $\gamma$ -alumina membrane.

1209

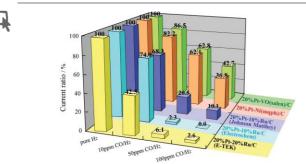


Enantioselective epoxidation of unfunctionalised olefins catalyzed by Mn(salen) complexes immobilized in porous materials *via* phenyl sulfonic group

Huidong Zhang, Song Xiang and Can Li\*

New heterogeneous chiral Mn(salen) catalysts axially immobilized on mesoporous materials *via* phenyl sulfonic groups result in remarkably higher ee values (75.8 *vs.* 55.0% and 94.0 *vs.* 54.8%) and ratio of *cisltrans* (16.2 *vs.* 0.38) for asymmetric epoxidation of unfunctionalised olefins than their analogous homogeneous counterparts.

1212



New CO tolerant electro-catalysts exceeding Pt-Ru for the anode of fuel cells

Hiroshi Yano, Chisato Ono, Hidenobu Shiroishi and Tatsuhiro Okada\*

Novel types of CO tolerant electro-catalysts have been developed from Pt and organic metal complexes that are far superior to Pt–Ru as anode catalysts in reformate gas fuel cells.

1215

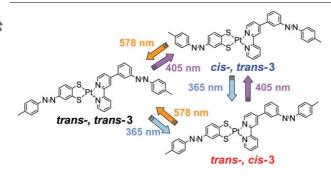


Photo-controllable tristability of a dithiolato-bipyridine-Pt(II) complex molecule containing two azobenzene moieties

Ryota Sakamoto, Masaki Murata, Shoko Kume, Hidekazu Sampei, Manabu Sugimoto\* and Hiroshi Nishihara\*

A new platinum complex with both an azo-bound dithiolato ligand and an azobenzene-bound bipyridine ligand exhibits tristability reversibly controllable using different energy lights.

### 1218

### Resonance stabilized bis-thiadiazinyl radicals

Leanne Beer, Robert C. Haddon, Mikhail E. Itkis, Alicea A. Leitch, Richard T. Oakley,\* Robert W. Reed, John F. Richardson and Donald G. VanderVeer

The resonance stabilized *bis*-thiadiazinyl framework holds potential as a stable and versatile building block for the design of radical-based conductors and magnetic materials.

### 1221

## First successful application of diphosphite ligands in the asymmetric hydroformylation of dihydrofurans

Montserrat Diéguez,\* Oscar Pamies and Carmen Claver\*

Good enantioselectivities and excellent regioselectivities are achieved in the Rh-catalyzed asymmetric hydroformylation of 2,5- and 2,3-dihydrofuran using diphosphite ligands; whereby the backbone of the ligand is crucial to suppressing isomerization and obtaining high ee's.

#### ee up to 75% regio up to 100%

### **ADDITIONS AND CORRECTIONS**

### 1224

An ultrasensitive nucleic acid biosensor based on the catalytic oxidation of guanine by a novel redox threading intercalator

Natalia C. Tansil, Fang Xie, Hong Xie and Zhiqiang Gao

A new approach to construct full-length glycosylphosphatidylinositols of parasitic protozoa and [4-deoxy-Man-III]-GPI analogues

Asif Ali, D. Channe Gowda and Ram A. Vishwakarma

Methoxycarbonylation of vinyl acetate catalysed by palladium complexes of bis(ditertiarybutylphosphinomethyl)benzene and related ligands Adam J. Rucklidge, George E. Morris and David J. Cole-Hamilton

Rh(II) catalysed intramolecular C-H insertion of diazo substrates in water: a simple and efficient approach to catalyst reuse Nuno R. Candeias, Pedro M. P. Gois Carlos and A. M. Afonso

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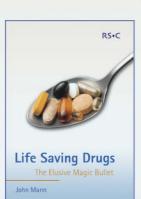
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