

# ChemComm

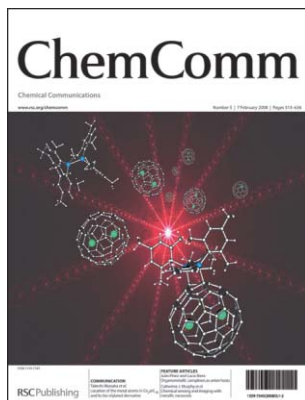
Chemical Communications

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## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (5) 513-636 (2008)



### Cover

See Takeshi Akasaka *et al.*, pp. 558–560.  
A new dimetallofullerene,  $Ce_2@C_{78}$ , and its bis-silylated derivative were successfully prepared and fully characterized. Image reproduced by permission of Michio Yamada, Takatsugu Wakahara, Takahiro Tsuchiya, Yutaka Maeda, Masahiro Kako, Takeshi Akasaka, Kenji Yoza, Ernst Horn, Naomi Mizorogi and Shigeru Nagase from *Chem. Commun.*, 2008, 558.



### Inside cover

See Jean-Marc Gaudin and Pascal Millet, pp. 588–590.  
A one step efficient synthesis of a perfumery ingredient that has a nice grapefruit and floral olfactory notes. (Author of the image: Migdonia Cuervo Diminger.)  
Image reproduced by permission of Jean-Marc Gaudin and Pascal Millet from *Chem. Commun.*, 2008, 588.

## CHEMICAL BIOLOGY

B9

Drawing together research highlights and news from all RSC publications, *Chemical Biology* provides a 'snapshot' of the latest developments in chemical biology, showcasing newsworthy articles and significant scientific advances.

# Chemical Biology

February 2008/Volume 3/Issue 2

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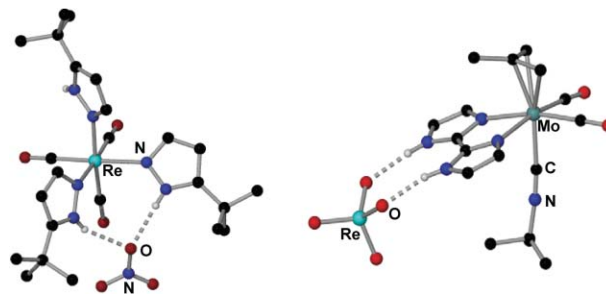
## FEATURE ARTICLES

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### Organometallic complexes as anion hosts

Julio Pérez\* and Lucía Riera

Salts of the inert, non-coordinating, lipophilic anion tetrakis(3,5-bis(trifluoromethyl)phenyl)borate and organometallic complexes with ligands containing hydrogen bond donor groups such as pyrazoles or biimidazole have been used for the first time as anion hosts.



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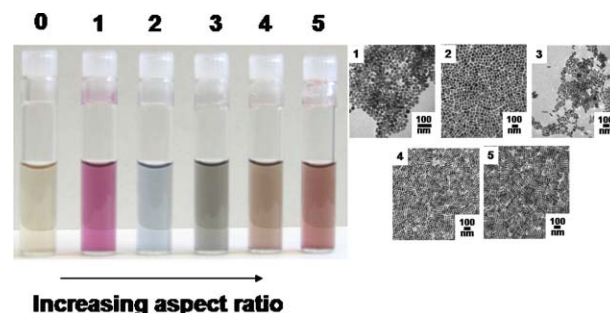
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### Chemical sensing and imaging with metallic nanorods

Catherine J. Murphy,\* Anand M. Gole, Simona E. Hunyadi, John W. Stone, Patrick N. Sisco, Alaaldin Alkilany, Brian E. Kinard and Patrick Hankins

The synthesis of gold nanorods has been well-described in the literature. The optical properties of gold nanorods are receiving great attention as novel chemical sensing and biological imaging agents.



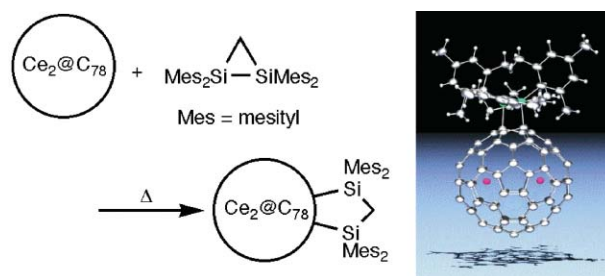
## COMMUNICATIONS

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### Location of the metal atoms in $Ce_2@C_{78}$ and its bis-silylated derivative

Michio Yamada, Takatsugu Wakahara, Takahiro Tsuchiya, Yutaka Maeda, Masahiro Kako, Takeshi Akasaka,\* Kenji Yoza, Ernst Horn, Naomi Mizorogi and Shigeru Nagase

Dimetallofullerene  $Ce_2@C_{78}$  and its bis-silylated derivative (1) were successfully prepared and characterized. The encapsulated Ce atoms are localized on the  $C_3$  axis of the  $C_{78}$  cage in  $Ce_2@C_{78}$  and more tightly localized in 1.

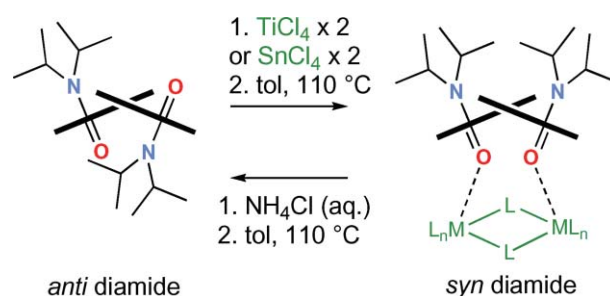


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### Conformational switching between diastereoisomeric atropisomers of arenedicarboxamides induced by complexation with Lewis acids

Jonathan Clayden,\* Lluís Vallverdú, James Clayton and Madeleine Helliwell

Tertiary diamides of xanthene-1,8-dicarboxylic acid and biphenyl-2,2'-dicarboxylic acid exhibit a thermodynamic preference for *anti* stereochemistry which is inverted in the presence of Ti- or Sn-based Lewis acids, allowing switching between *syn* and *anti* diastereoisomeric atropisomers.

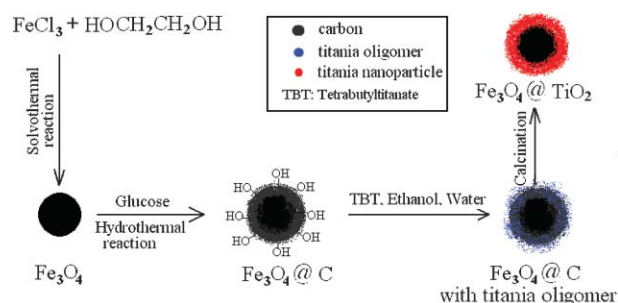


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### Novel approach for the synthesis of $Fe_3O_4@TiO_2$ core-shell microspheres and their application to the highly specific capture of phosphopeptides for MALDI-TOF MS analysis

Yan Li, Jinsong Wu, Dawei Qi, Xiuqing Xu, Chunhui Deng,\* Pengyuan Yang and Xiangmin Zhang\*

A novel approach is proposed to synthesize  $Fe_3O_4@TiO_2$  microspheres with a well-defined core-shell structure, and the synthesized microspheres were successfully applied for the simple and fast enrichment of phosphopeptides.



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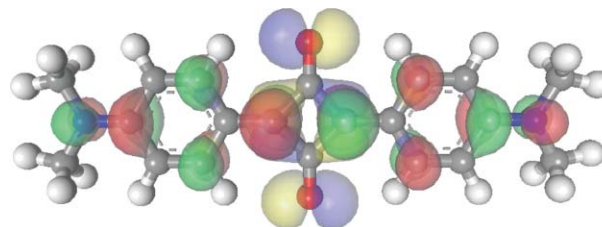


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### Squaraines as unique reporters for SERRS multiplexing

Robert J. Stokes, Andrew Ingram, Jane Gallagher, David R. Armstrong, W. Ewen Smith and Duncan Graham\*

Squaraine dyes provide strong, unique surface enhanced resonance Raman scattering that can be identified within any mixture of current reporters using longer, biologically compatible wavelengths of excitation.

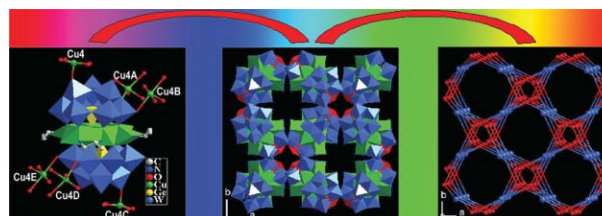


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### Combination between lacunary polyoxometalates and high-nuclear transition metal clusters under hydrothermal conditions: first (3,6)-connected framework constructed from sandwich-type polyoxometalate building blocks containing a novel {Cu<sub>8</sub>} cluster

Jun-Wei Zhao, Jie Zhang, Shou-Tian Zheng and Guo-Yu Yang\*

A novel 3-D {Cu<sub>8</sub>} sandwiched POM has been made.

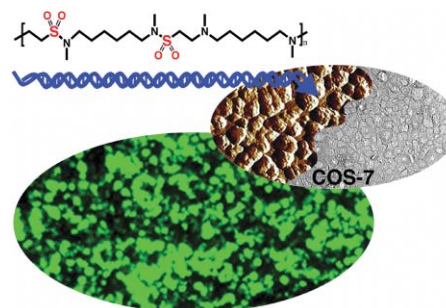


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### Poly( $\beta$ -aminosulfonamides) as gene delivery vectors: synthesis and *in vitro* screening

Lin Gan, Jennifer L. Olson, Clifton W. Ragsdale\* and Luping Yu\*

A series of poly( $\beta$ -aminosulfonamides) was synthesized and demonstrated to be efficient *in vitro* transfection reagents.

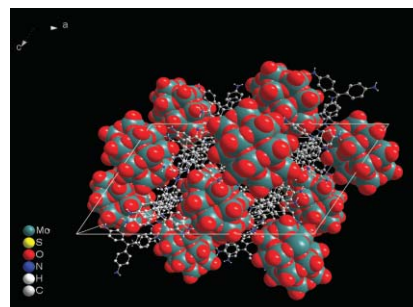


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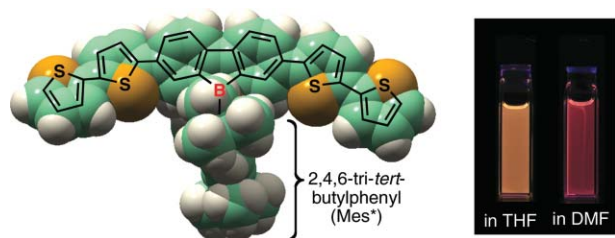
### Facile assembly of hybrid materials containing polyoxometalate cluster anions and organic dye cations: crystal structures and initial spectral characterization

Jingli Xie,\* Brendan F. Abrahams and Anthony G. Wedd\*

Salts of the pararosaniline dye cation and four polyoxometalate cluster anions have been isolated under both ambient and hydrothermal conditions; structural and initial spectroscopic data are consistent with significant perturbation of ion electronic states induced by charge-assisted N-H...O hydrogen bonds.



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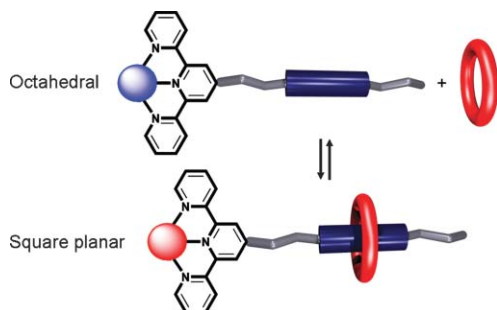


### Kinetically stabilized dibenzoborole as an electron-accepting building unit

Atsushi Wakamiya, Kotaro Mishima, Kanako Ekawa and Shigehiro Yamaguchi\*

Dibenzoborole derivatives kinetically well-stabilized by a bulky Mes\* group show orange-red emissions and stable electrochemical redox properties.

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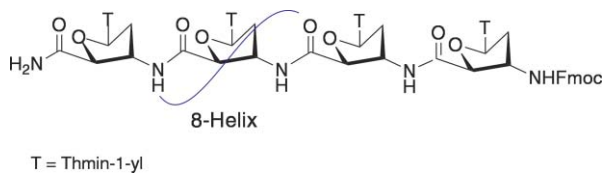


### Controlling the ON/OFF threading of a terpyridine containing [2]pseudorotaxane ligand *via* changes in coordination geometry

Sapna Sharma, Gregory J. E. Davidson and Stephen J. Loeb\*

The stability of a [2]pseudorotaxane can be controlled by the inclusion of a chelating terpyridine unit. A square planar geometry provides increased  $\pi$ -stacking which enhances the interaction between axle and wheel, while an octahedral geometry produces steric hindrance which dramatically reduces the association.

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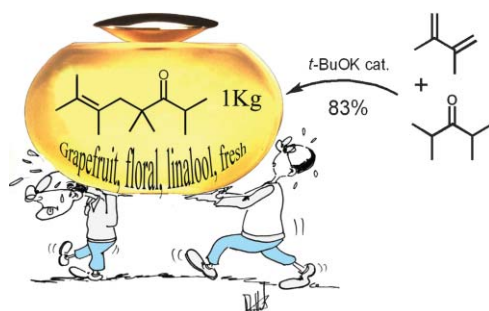


### Peptides derived from nucleoside $\beta$ -amino acids form an unusual 8-helix

Richard Threlfall, Andrew Davies, Nicola M. Howarth, Julie Fisher\* and Richard Cosstick\*

Peptides of varying length (dimers to octamers) were prepared from nucleoside  $\beta$ -amino acids and conformational studies, based on NOE observations, show that the  $\beta$ -peptides form an unusual 8-helix.

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### Transition metal-free addition of ketones or nitriles to 1,3-dienes

Jean-Marc Gaudin\* and Pascal Millet

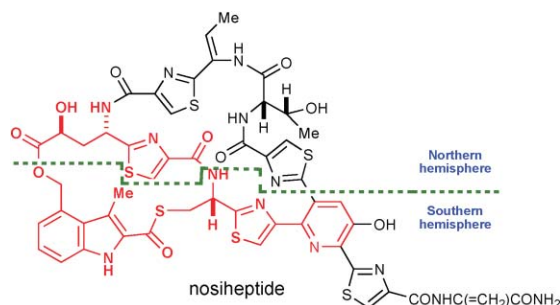
The simpler the better (or going green): The first examples of the catalytic addition of 1,3-dienes to simple ketones or nitriles are described. These reactions can be effected on a kilogram scale, representing the shortest access featuring a perfect atom economy to molecules of interest in the perfume industry.

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### Construction of macrocyclic thiopeptides: synthesis of a nosiheptide 'southern hemisphere' model system

Marc C. Kimber and Christopher J. Moody\*

A 20-membered macrocyclic thiopeptide has been synthesized as a model for the southern hemisphere of nosiheptide.

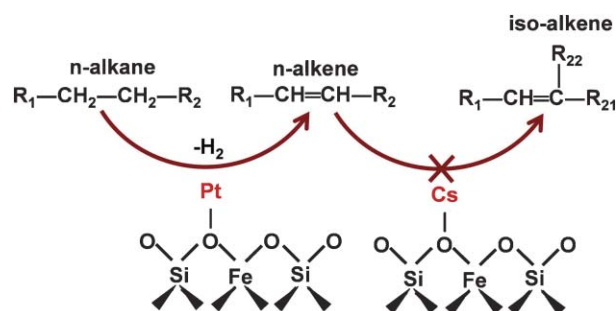


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### Pt/[Fe]ZSM-5 modified by Na and Cs cations: an active and selective catalyst for dehydrogenation of *n*-alkanes to *n*-alkenes

Xuebing Li and Enrique Iglesia\*

Pt clusters within [Fe]ZSM-5 channels provide active and stable sites for the selective catalytic dehydrogenation of *n*-alkanes to *n*-alkenes. Cs and Na cations titrate acid sites and inhibit skeletal isomerization and cracking side reactions.

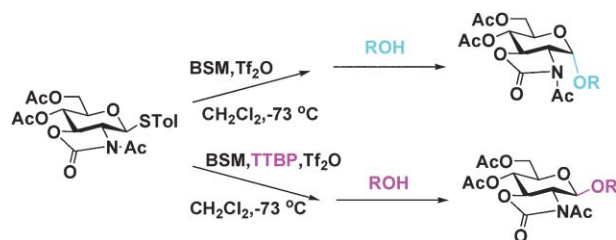


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### Pre-activation protocol leading to highly stereoselectivity-controllable glycosylations of oxazolidinone protected glucosamines

Yiqun Geng, Li-He Zhang and Xin-Shan Ye\*

Based on pre-activation protocol, a new efficient strategy for both  $\alpha$ - and  $\beta$ -stereoselective glycosylations of glucosamine donors was developed. The 4,6-di-*O*-acetyl-*N*-acetyl-oxazolidinone protected donor afforded either  $\beta$ - or  $\alpha$ -stereoselectivity simply by means of the addition of hindered base TTBP or the absence of base, leading to controllable glycosylation reactions.

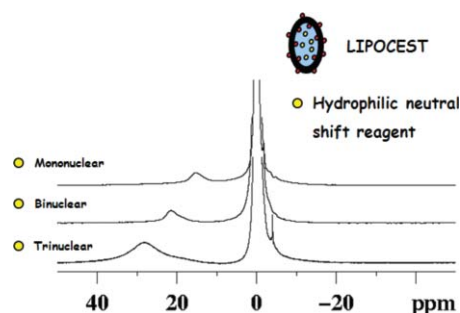


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### Highly shifted LIPOCEST agents based on the encapsulation of neutral polynuclear paramagnetic shift reagents

Enzo Terreno, Alessandro Barge, Lorena Beltrami, Giancarlo Cravotto, Daniela Delli Castelli, Franco Fedeli, Bhagavathsingh Jebasingh and Silvio Aime\*

Improved LIPOCEST MRI contrast agents with highly shifted intraliposomal water protons were prepared by entrapping neutral polynuclear Tm(III)-based paramagnetic shift reagents in phospholipidic vesicles.



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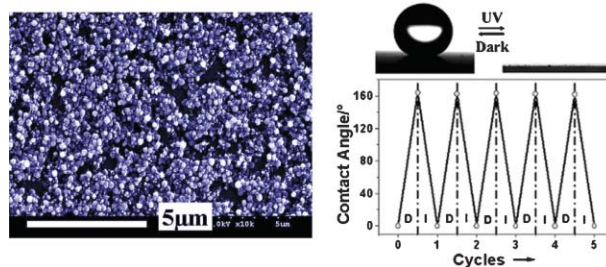


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### Reversible switching on superhydrophobic TiO<sub>2</sub> nano-strawberry films fabricated at low temperature

Wentao Sun, Shuyun Zhou, Ping Chen\* and Lianmao Peng

Superhydrophobic TiO<sub>2</sub> nano-strawberry rutile films, on which superhydrophobicity and superhydrophilicity can be reversibly switched by alternation of ultraviolet irradiation and dark storage, were fabricated on a large scale *via* a seeded growth method.

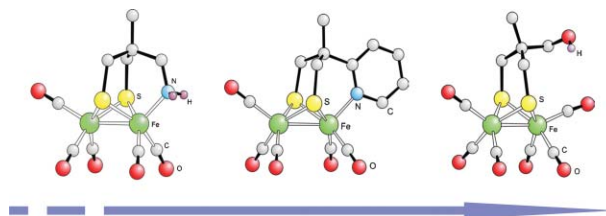


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### Controlling carbon monoxide binding at di-iron units related to the iron-only hydrogenase sub-site

Fenfen Xu, Cédric Tard, Xiufeng Wang, Saad K. Ibrahim, David L. Hughes, Wei Zhong, Xirui Zeng, Qiuyan Luo, Xiaoming Liu\* and Christopher J. Pickett\*

Hemi-labile bonds, protonation and thermodynamics of CO binding

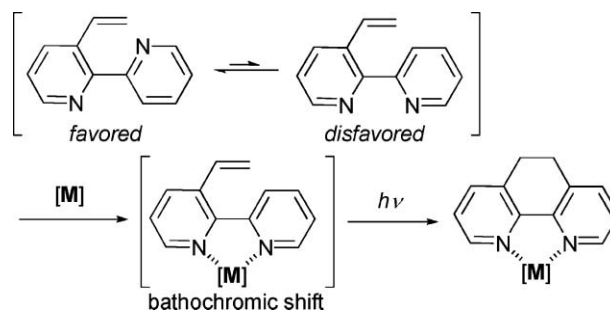


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### Chelation-assisted electrocyclic reactions of 3-alkenyl-2,2'-bipyridines: an efficient method for the synthesis of 5,6-dihydro-1,10-phenanthroline and 1,10-phenanthroline derivatives

Akihiko Takahashi, Yuko Hirose, Hiroyuki Kusama\* and Nobuharu Iwasawa\*

An efficient method for the synthesis of substituted 5,6-dihydro-1,10-phenanthrolines and 1,10-phenanthrolines has been developed using chelation-assisted photochemical electrocyclic reactions of 3-alkenyl-2,2'-bipyridines.

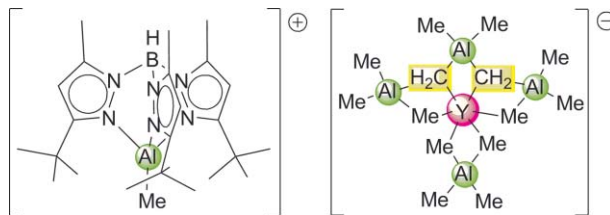


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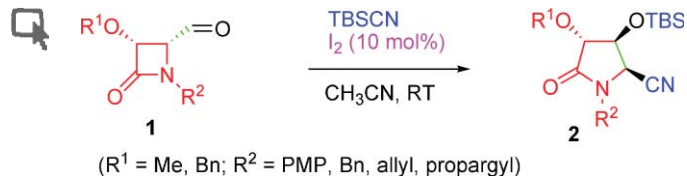
### Ln(III) methyl and methylene complexes stabilized by a bulky hydrotris(pyrazolyl)borate ligand

Melanie Zimmermann, Josef Takats, Gong Kiel, Karl W. Törnroos and Reiner Anwander\*

The *tetramethylaluminate route* gives access to unprecedented trispyrazolylborate-supported Ln–Al heterobimetallic moieties such as Ln(CH<sub>3</sub>)[(μ-CH<sub>3</sub>)AlMe<sub>3</sub>] and “double-Tebbe-like” Ln[(μ-CH<sub>2</sub>)<sub>2</sub>AlMe<sub>2</sub>], involving C–H bond activation reactions.



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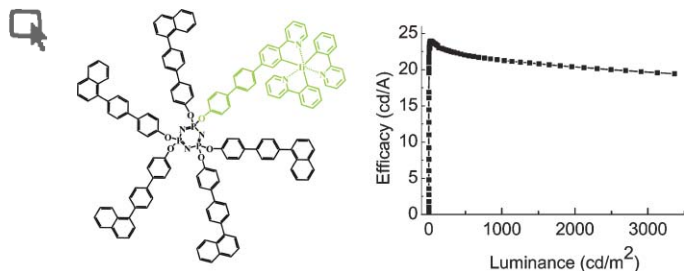


**I<sub>2</sub>-Catalyzed enantioselective ring expansion of β-lactams to γ-lactams through a novel C3–C4 bond cleavage. Direct entry to protected 3,4-dihydropyrrolidin-2-one derivatives**

Benito Alcaide,\* Pedro Almendros, Gema Cabrero and M. Pilar Ruiz

I<sub>2</sub> (10 mol%) efficiently catalyzes the ring expansion of 4-oxoazetidine-2-carbaldehydes **1** in the presence of *tert*-butyldimethyl cyanide to afford protected 5-cyano-3,4-dihydroxy-pyrrolidine-2-ones **2** through a novel C3–C4 bond cleavage of the β-lactam nucleus.

618

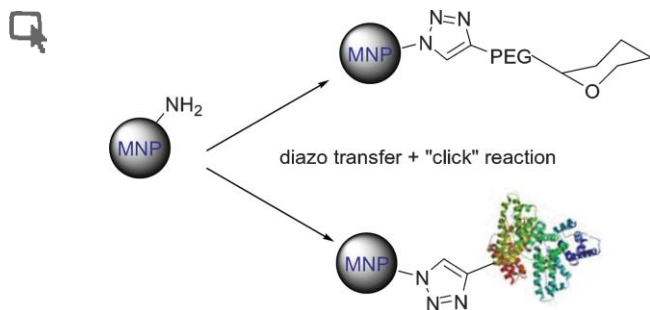


**Solution processable phosphorescent dendrimers based on cyclic phosphazenes for use in organic light emitting diodes (OLEDs)**

Henk J. Bolink,\* Sonsoles Garcia Santamaria, Sundarraj Sudhakar, Changgua Zhen and Alan Sellinger\*

A novel solution processable phosphorescent dendrimer based on a cyclic phosphazene (CP) core has been prepared and used as emissive layers in simple OLED architectures, providing efficiencies of 24.0 cd A<sup>-1</sup> and 16.7 lm W<sup>-1</sup>.

621

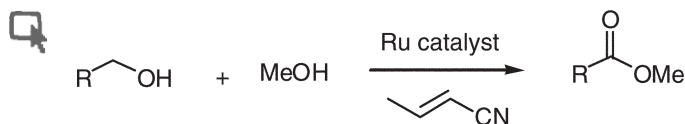


**One-step bioengineering of magnetic nanoparticles *via* a surface diazo transfer/azide–alkyne click reaction sequence**

Laura Polito, Diego Monti, Enrico Caneva, Eleonora Delnevo, Giovanni Russo and Davide Prospero\*

We have developed an efficient conversion of amino iron oxides to carbohydrate and protein derived nanoparticles with highly conserved bioactivity through a combination of diazo transfer and azide–alkyne click technology.

624



**Oxidation of primary alcohols to methyl esters by hydrogen transfer**

Nathan A. Owston, Alexandra J. Parker and Jonathan M. J. Williams\*

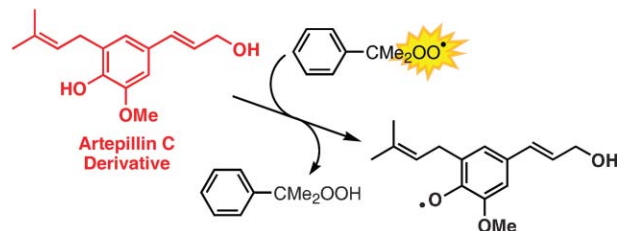
A ruthenium catalyst has been used to oxidise primary alcohols by hydrogen transfer to crotonitrile, selectively leading to methyl esters.

626

### Enhanced radical-scavenging activity of naturally-oriented artemillin C derivatives

Sushma Manda, Ikuo Nakanishi,\* Kei Ohkubo, Yoshihiro Uto, Tomonori Kawashima, Hitoshi Hori, Kiyoshi Fukuhara, Haruhiro Okuda, Toshihiko Ozawa, Nobuo Ikota, Shunichi Fukuzumi\* and Kazunori Anzai\*

More than two-fold augmentation in the radical-scavenging activity of artemillin C could be achieved *via* altering the O–H bond dissociation enthalpy of artemillin C by means of structural modifications.

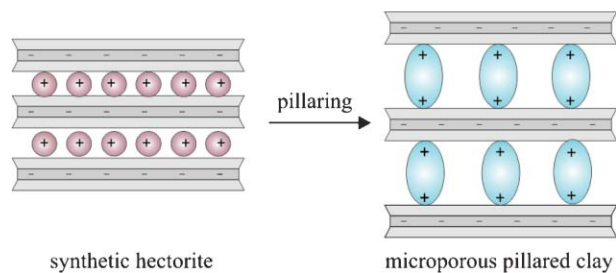


629

### Realisation of truly microporous pillared clays

Melanie Stöcker, Wolfgang Seidl, Lena Seyfarth, Jürgen Senker and Josef Breu\*

With clays synthesized *via* the melt, the concept of pillaring, which is appealing to the present day for manifold reasons, may be revitalized and a new class of truly microporous materials is (re-)established.

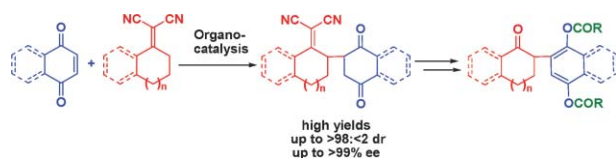


632

### Organocatalytic asymmetric vinylogous addition to quinones – formation of optically active $\alpha$ -aryl ketones

José Alemán, Christian Borch Jacobsen, Kim Frisch, Jacob Overgaard and Karl Anker Jørgensen\*

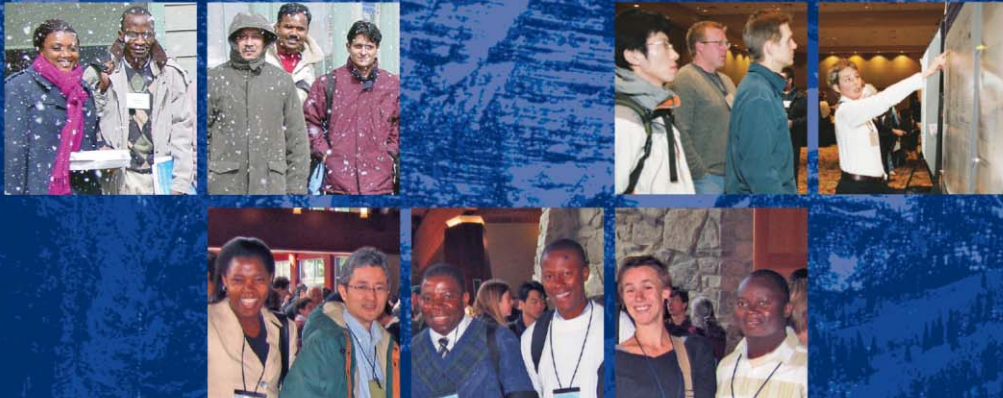
The first organocatalytic allylic additions of alkylidene derivatives to quinones by using Cinchona alkaloid catalysts have been presented (up to 99% ee). The products can be derivatized to  $\alpha$ -arylated alkylidenes and ketones.





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- Molecular Basis for Biological Membrane Organization
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- Plant Hormones and Signaling
- Plant Innate Immunity
- NF- $\kappa$ B
- Cell Migration in Invasion and Inflammation
- Wnt/ $\beta$ -Catenin Signaling in Development and Disease
- Cancer Genomics and Epigenomics

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- NK and NKT Cell Biology
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- HIV Pathogenesis
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- Nuclear Receptors: Steroid Sisters
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- Islet and Beta-Cell Biology
- Islet and Beta Cell Development and Transplantation
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- Molecular Evolution as a Driving Force in Infectious Diseases
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### MAY 2008

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