

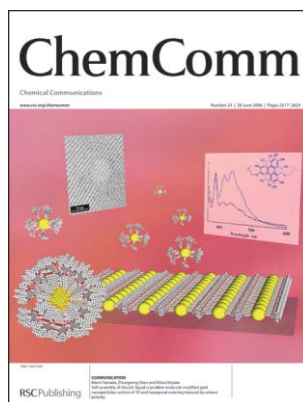
## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (24) 2517–2624 (2006)



### Cover

See Ian R. Baxendale, Jon Deeley, Charlotte M. Griffiths-Jones, Steven V. Ley, Steen Saaby and Geoffrey K. Tranmer, page 2566. The cover depicts the molecule oxamaritidine together with the specially commissioned work called Flowing I-V by the Cambridge artist Charlotte Cornish. Image reproduced by permission of Steven V. Ley *et al.* from *Chem. Commun.*, 2006, 2566.



### Inside cover

See Mami Yamada, Zhongrong Shen and Mikio Miyake, page 2569. 1D arrangement of discotic crystalline molecule-modified Au nanoparticles. Image reproduced by permission of Mami Yamada *et al.* from *Chem. Commun.*, 2006, 2569.

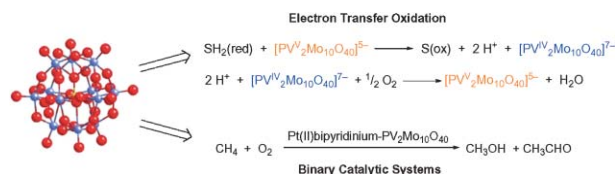
## FEATURE ARTICLE

2529

### Molecular oxygen and oxidation catalysis by phosphovanadomolybdates

Ronny Neumann\* and Alexander M. Khenkin

Synthetic and mechanistic aspects of aerobic catalytic oxidation mediated by phosphovanadomolybdates is described. Electron-transfer oxidation can lead to either oxygenation or oxydehydrogenation. Newer binary catalysts such as polyoxometalate–organometallic complexes can be applied for methane oxidation, while metal nanoparticles stabilized by polyoxometalates are useful of alkene epoxidation.



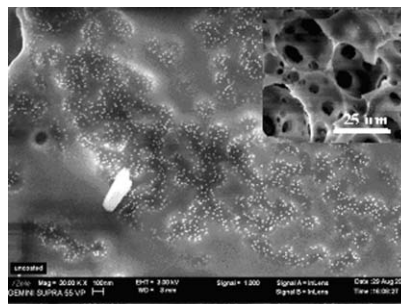
## COMMUNICATIONS

2539

### Synthesis of hierarchically porous inorganic–metal site-isolated nanocomposites

Haifei Zhang,\* Irshad Hussain, Mathias Brust and Andrew I. Cooper

Hierarchically porous inorganic nanocomposites have been synthesized combining interconnected macropores and mesopores with a high loading of site-isolated gold nanoparticles.



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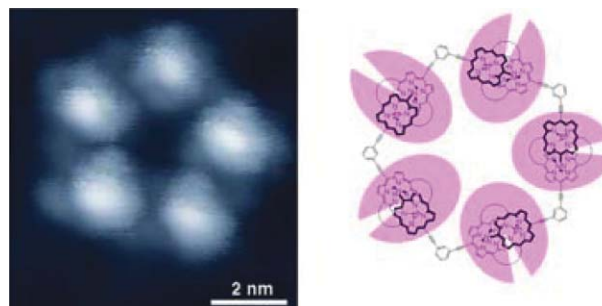
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### Single molecular observation of penta- and hexagonal assembly of bisporphyrin on a gold surface

Akiharu Satake, Hiroyuki Tanaka, Fatin Hajjaj, Tomoji Kawai\* and Yoshiaki Kobuke\*

Artificial light-harvesting antennae of coordination-organized macrocyclic porphyrin pentamer and hexamer are visualized by high resolution scanning tunneling microscopy (HRSTM) on a gold surface. Structural detail is assigned by comparison with those disassociated macrorings obtained with submolecular resolution.

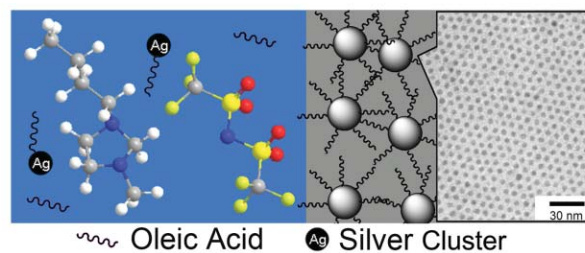


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### Oleic acid as the capping agent in the synthesis of noble metal nanoparticles in imidazolium-based ionic liquids

Yong Wang and Hong Yang\*

The synthesis and automatic separation of silver nanoparticles in imidazolium-based ionic liquids using oleic acid as the capping agent (the drawing does not reflect the real scale and configuration).

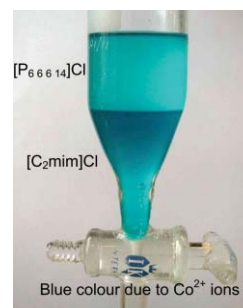


2548

### Mutually immiscible ionic liquids

Alberto Arce, Martyn J. Earle,\* Suhas P. Katdare, Héctor Rodríguez and Kenneth R. Seddon

We report ionic liquids that are mutually immiscible, some of which are also immiscible with solvents as diverse as water and alkanes; an archetypal biphasic system is trihexyltetradecylphosphonium chloride with 1-alkyl-3-methylimidazolium chloride (where the alkyl group is shorter than hexyl).

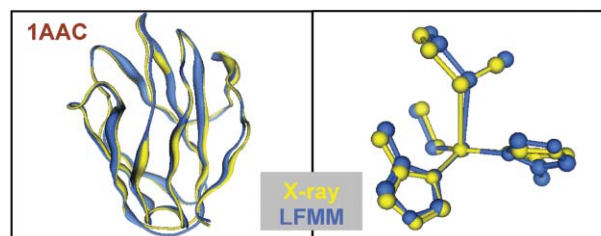


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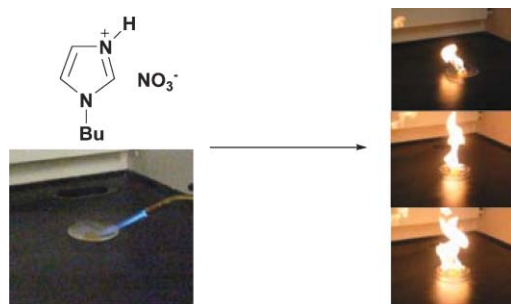
### A test of ligand field molecular mechanics as an efficient alternative to QM/MM for modelling metalloproteins: the structures of oxidised type I copper centres

Robert James Deeth

Ligand Field Molecular Mechanics based on homoleptic model systems delivers accurate, unbiased geometries of complete mononuclear blue copper proteins about four orders of magnitude faster than comparable QM/MM calculations.



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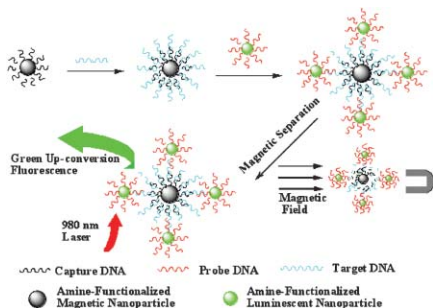


### Combustible ionic liquids by design: is laboratory safety another ionic liquid myth?

Marcin Smiglak, W. Mathew Reichert, John D. Holbrey, John S. Wilkes,\* Luyi Sun, Joseph S. Thrasher, Kostyantyn Kirichenko, Shailendra Singh, Alan R. Katritzky and Robin D. Rogers\*

Ionic liquids, as a class, cannot be considered as safe when working with heat or ignition sources; exothermic reactions can occur releasing highly flammable products.

2557

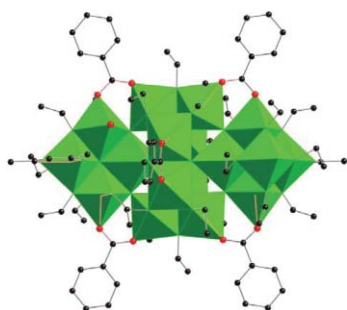


### Green upconversion nanocrystals for DNA detection

Leyu Wang and Yadong Li\*

By combining magnetic-field-assisted bioseparation and concentration technology with magnetite nanoparticles, novel green upconversion (UC) fluorescence nanocrystals ( $\text{NaYF}_4:\text{Yb}^{3+}/\text{Er}^{3+}$ ) have been applied to the sensitive detection of DNA.

2560

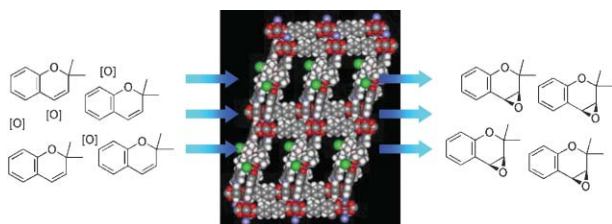


### A high-nuclearity, beyond “fully reduced” polyoxo(alkoxo)vanadium(III/IV) cage

Ian S. Tidmarsh, Rebecca H. Laye, Paul R. Brearley, Maheswaran Shanmugam, E. Carolina Sañudo, Lorenzo Sorace, Andrea Caneschi and Eric J. L. McInnes\*

A highly reduced polyoxo(alkoxo)vanadium(III/IV) cage, containing a  $\text{V(III)}_{16}\text{V(IV)}_2$  core, has been prepared by a simple solvothermal route.

2563



### A metal–organic framework material that functions as an enantioselective catalyst for olefin epoxidation

So-Hye Cho, Baoqing Ma, SonBinh T. Nguyen,\* Joseph T. Hupp\* and Thomas E. Albrecht-Schmitt

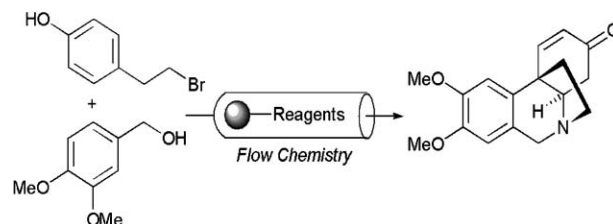
A new crystalline microporous metal–organic framework compound featuring chiral (salen)Mn struts is long-lived and highly effective as a substrate-size-selective asymmetric catalyst for olefin epoxidation.

2566

### A flow process for the multi-step synthesis of the alkaloid natural product oxomaritidine: a new paradigm for molecular assembly

Ian R. Baxendale, Jon Deeley, Charlotte M. Griffiths-Jones, Steven V. Ley,\* Steen Saaby and Geoffrey K. Tranmer

A flow process for the multi-step synthesis of the natural product ( $\pm$ )-oxomaritidine is described, mediated through the use of microfluidic pumping systems that progress material through various packed columns containing immobilized reagents.

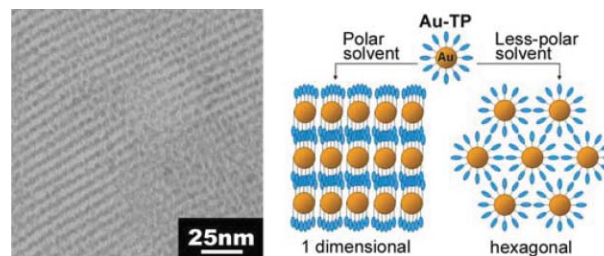


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### Self-assembly of discotic liquid crystalline molecule-modified gold nanoparticles: control of 1D and hexagonal ordering induced by solvent polarity

Mami Yamada,\* Zhongrong Shen and Mikio Miyake\*

Gold nanoparticles fully coated with discotic liquid crystalline molecules of hexaalkoxy-substituted triphenylene (Au-TP) have been synthesised; the self-assembled structure of the prepared Au-TPs could be controlled (hexagonal or 1D nanochain) just by altering the ratio of methanol to toluene in the solvent.

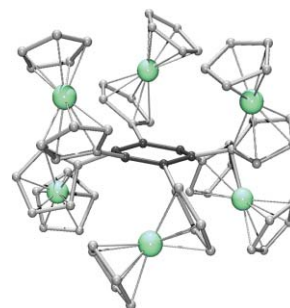


2572

### Hexaferrocenylbenzene

Yong Yu, Andrew D. Bond, Philip W. Leonard, Ulrich J. Lorenz, Tatiana V. Timofeeva, K. Peter C. Vollhardt,\* Glenn D. Whitener and Andrey A. Yakovenko

Hexaferrocenylbenzene has been synthesized by six-fold Negishi type ferrocenylation of hexabromo- or hexaiodobenzene.

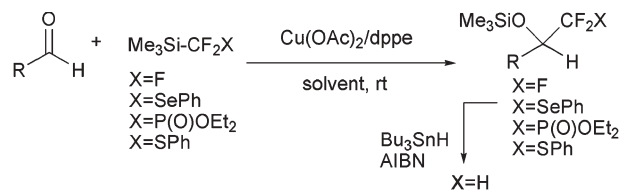


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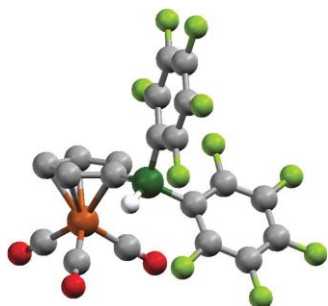
### Lewis acid-catalyzed tri- and difluoromethylation reactions of aldehydes

Satoshi Mizuta, Norio Shibata,\* Shinichi Ogawa, Hiroyuki Fujimoto, Shuichi Nakamura and Takeshi Toru\*

The first Lewis acid-catalyzed tri- and difluoromethylation reactions of aldehydes with  $\text{Me}_3\text{SiCF}_2\text{X}$  under  $\text{Cu}(\text{OAc})_2/\text{dppe}$  conditions are described.



2578

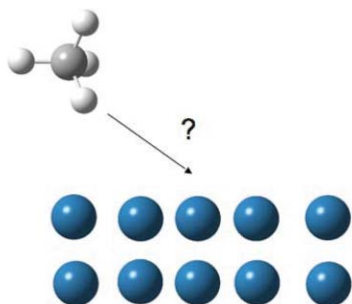


### Migratory insertion of $[B(C_6F_5)_2]$ into C–H bonds: CO promoted transfer of the boryl fragment

Simon Aldridge,\* Deborah L. Kays (née Coombs), Amal Al-Fawaz, Kevin M. Jones, Peter N. Horton, Michael B. Hursthouse, Ross W. Harrington and William Clegg

The reaction of  $(\eta^5-C_5H_5)Fe(CO)_2B(C_6F_5)_2$  with CO proceeds *via* migratory insertion of the  $[B(C_6F_5)_2]$  fragment into a C–H bond of the Cp ligand, thereby revealing a new mode of attack at C–H bonds by a transition metal boryl complex.

2581

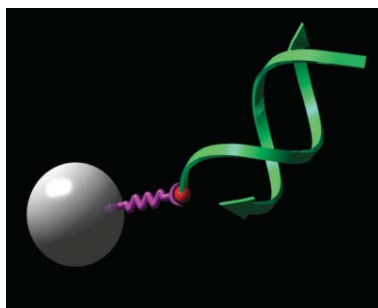


### Reactions of platinum clusters $Pt_n^{\pm}$ , $n = 1-21$ , with $CH_4$ : to react or not to react

Christian Adlhart and Einar Uggerud\*

Bimodal (on–off) behaviour is observed in reactions between platinum clusters with methane.

2583

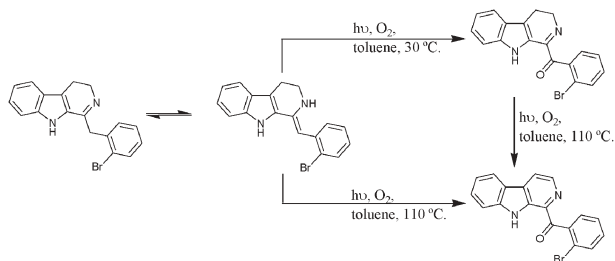


### Separation of stereoisomers of dinuclear metal complexes by binding affinity chromatography using non-duplex DNA

Jayden A. Smith and F. Richard Keene\*

Affinity chromatography – using non-duplex DNA as the affinity ligand – has been used as a highly efficient means of separating stereoisomers of dinuclear polypyridyl ruthenium(II) complexes.

2586



### Regioselective photo-oxidation of 1-benzyl-4,9-dihydro-3H- $\beta$ -carbolines

Marcos D. García, A. James Wilson, Daniel P. G. Emmerson and Paul R. Jenkins\*

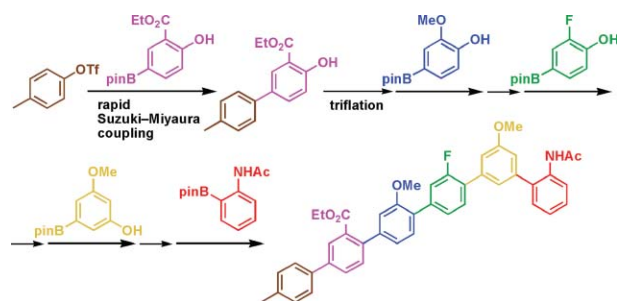
The regioselective self-sensitized photo-oxidation of 1-benzyl-4,9-dihydro-3H- $\beta$ -carbolines by means of light and molecular oxygen provides a new “green” methodology for the synthesis of 1-benzoyl-3,4-dihydro- $\beta$ -carbolines and 1-benzoyl- $\beta$ -carbolines.

2589

### Synthetic method for multifunctionalized oligoarenes using pinacol esters of hydroxyphenylboronic acids

Shunpei Ishikawa and Kei Manabe\*

A synthetic method for multifunctionalized oligoarenes using rapid Suzuki–Miyaura coupling of pinacol esters of hydroxyphenylboronic acids and subsequent triflation of the hydroxy group was developed.

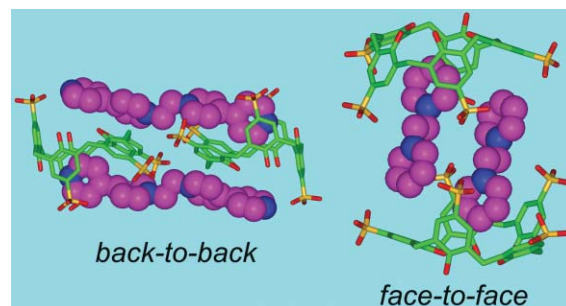


2592

### Unique conformation and packing structure of *p*-sulfonatocalix[5]arene induced by 1,2-bis(pyridinium)ethane compounds

Dong-Sheng Guo, Heng-Yi Zhang, Chun-Ju Li and Yu Liu\*

In the presence of 1,2-bis(4,4'-dipyridinium)ethane, *p*-sulfonatocalix[5]arene (C5AS) forms back-to-back dimers; upon complexation with 1,2-bis(pyridinium)ethane, C5AS crystallizes in face-to-face dimers.

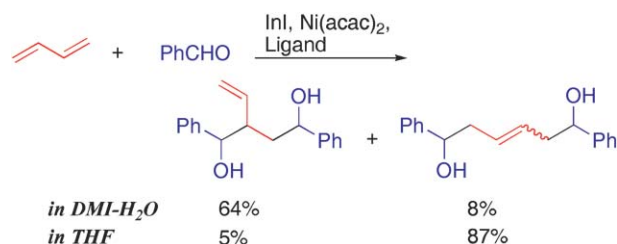


2595

### Nickel-catalyzed indium(I)-mediated double addition of aldehydes to 1,3-dienes

Tsunehisa Hirashita,\* Shinya Kambe, Hiromitsu Tsuji and Shuki Araki

In the presence of InI, Ni(acac)<sub>2</sub> and PPh<sub>3</sub>, several 1,3-dienes were reacted with two molecules of aldehyde to give the corresponding 1,4- and 1,6-diols.

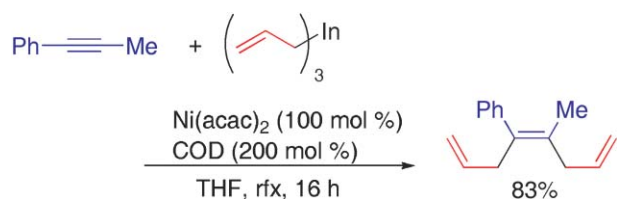


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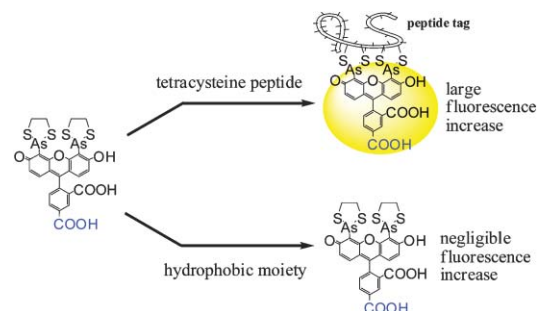
### Nickel-catalysed bis-allylation of internal alkynes with triallylindium

Tsunehisa Hirashita,\* Kazuhiko Akutagawa, Toshiya Kamei and Shuki Araki

The Ni-catalysed reaction of triallylindium with internal alkynes underwent bis-allylation to afford octa-1,4,7-trienes in high yield.



2601

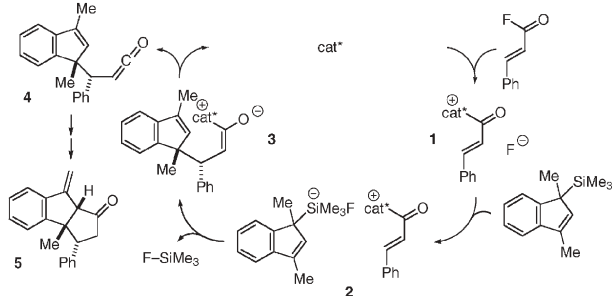


### CrAsH: a biarsenical multi-use affinity probe with low non-specific fluorescence

Haishi Cao, Baowei Chen, Thomas C. Squier and M. Uljana Mayer\*

CrAsH is a tetracysteine-binding probe which is insensitive to hydrophobic moieties, resulting in improved fluorescence properties.

2604

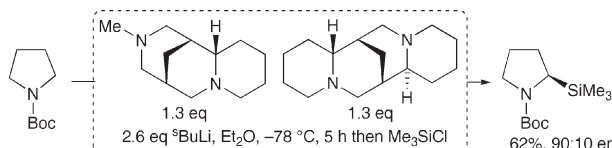


### Asymmetric [3 + 2] annulations catalyzed by a planar-chiral derivative of DMAP

Erhard Bappert, Peter Müller and Gregory C. Fu\*

A planar-chiral DMAP derivative catalyzes an intriguing [3 + 2] annulation reaction of silylated indenones to produce diquinanes that bear three contiguous stereocenters (one quaternary and two tertiary).

2607

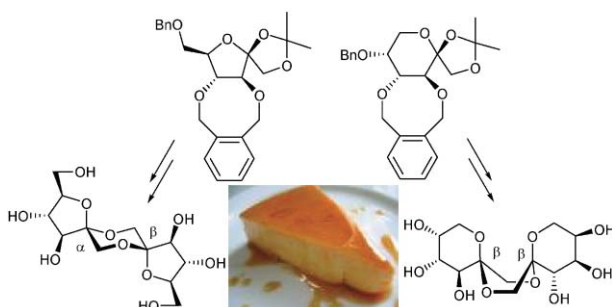


### Reactivity series for *s*-BuLi/diamine-mediated lithiation of *N*-Boc pyrrolidine: applications in catalysis and lithiation of *N*-Boc piperidine

Matthew J. McGrath, Julia L. Bilke and Peter O'Brien\*

Competition experiments between a range of ligands and (–)-sparteine were used to determine a reactivity series for *N*-Boc pyrrolidine lithiation using *s*-BuLi/diamines which has been applied in the selection of ligand pairs for ligand exchange catalytic asymmetric lithiation of *N*-Boc pyrrolidine and a more efficient asymmetric lithiation of *N*-Boc piperidine.

2610



### The *o*-xylylene protecting group as an element of conformational control of remote stereochemistry in the synthesis of spiroketals

Patricia Balbuena, Enrique M. Rubio, Carmen Ortiz Mellet\* and José M. García Fernández\*

The use of the *o*-xylylene group to favour *trans*-diequatorial dispositions in 1,2-diol systems has been exploited to control the stereochemical outcome of spirocyclisation reactions. The scope of the methodology is illustrated by the stereoselective synthesis of two bis-spirodisaccharides present in caramel.



2613

### Magnetic water-soluble cyano-bridged metal coordination nano-polymers

Yannick Guari,\* Joulia Larionova,\* Karine Molvinger, Benjamin Folch and Christian Guérin

A two-step approach towards highly stable aqueous solutions of cyano-bridged coordination polymer nanoparticles was developed using chitosan, a biocompatible polymer, as template and stabilizing agent.

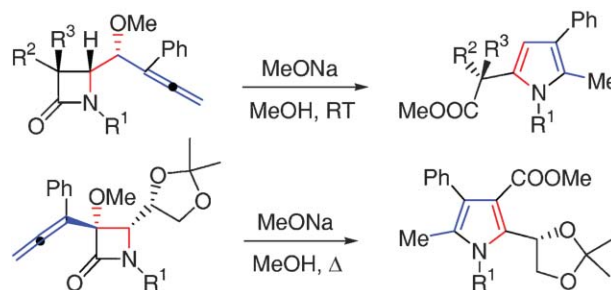


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### Domino metal-free allene-β-lactam-based access to functionalized pyrroles

Benito Alcaide,\* Pedro Almendros\* and María C. Redondo

A novel transition metal-free domino reaction sequence in allene-β-lactams, leading to the biologically relevant pyrrole frame has been developed using a sodium methoxide-methanol system.

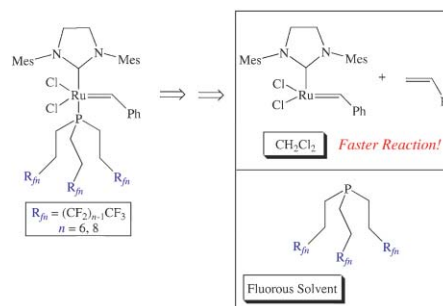


2619

### Fluorous phase-transfer activation of catalysts: application of a new rate-enhancement strategy to alkene metathesis

Rosenildo Corrêa da Costa and J. A. Gladysz\*

Analogs of Grubbs' second generation catalyst with fluorous phosphines are prepared and shown to give enhanced rates of metathesis under fluorous/organic biphasic conditions, presumably due to phase-transfer scavenging of the phosphine generated in the initial reversible step.




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