

# ChemComm

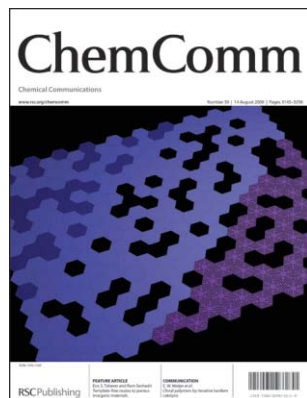
Chemical Communications

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## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (30) 3145-3256 (2006)



### Cover

See Eric S. Toberer and Ram Seshadri, page 3159.

An artist's view of the evolution of multiple levels of porosity arising through successive stages of leaching in a composite inorganic material. Image reproduced by permission of Eric S. Toberer and Ram Seshadri from *Chem. Commun.*, 2006, 3159.

## CHEMICAL TECHNOLOGY

T29

Chemical Technology highlights the latest applications and technological aspects of research across the chemical sciences.

## Chemical Technology

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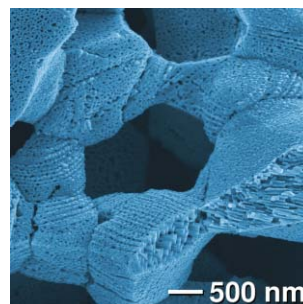
## FEATURE ARTICLE

3159

### Template-free routes to porous inorganic materials

Eric S. Toberer and Ram Seshadri

We describe a number of routes based on solid–solid and solid–gas reactivity that produce functional inorganic materials possessing hierarchies of pore sizes, without requiring the use of preformed templates.



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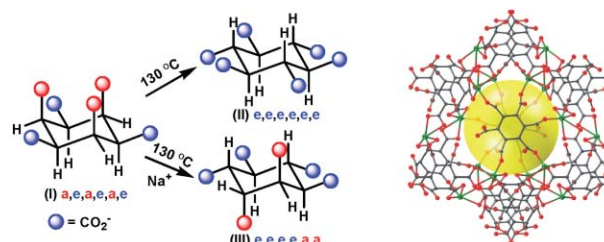
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3166

### Two new 3D metal–organic frameworks of nanoscale cages constructed by Cd(II) and conformationally-flexible cyclohexanehexacarboxylate

Jing Wang, Yue-Hua Zhang and Ming-Liang Tong\*

Two novel 3D MOFs with nanoscale cages were obtained from the hydrothermal reactions of  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  with *a,e,a,e,a,e*-1,2,3,4,5,6-cyclohexanehexacarboxylic acid ( $\text{H}_6\text{L}^1$ ), during which the  $\text{L}^1$  ligand transforms its conformation to the *e,e,e,e,e,e* form and to a mixture of the *e,e,e,e,e,e* and unprecedented *e,e,e,a,a* forms tuned by the auxiliary Na(I) as the template.

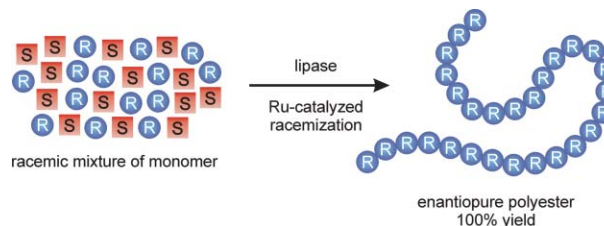


3169

### Chiral polymers by iterative tandem catalysis

Jeroen van Buijtenen, Bart A. C. van As, Jan Meuldijk, Anja R. A. Palmans, Jef A. J. M. Vekemans, L. A. Hulshof and E. W. Meijer\*

Racemic  $\omega$ -substituted caprolactones can be completely converted into chiral polyesters of remarkable MW and high ee by combining lipase-catalyzed ring-opening polymerization with Ru-catalyzed racemization.

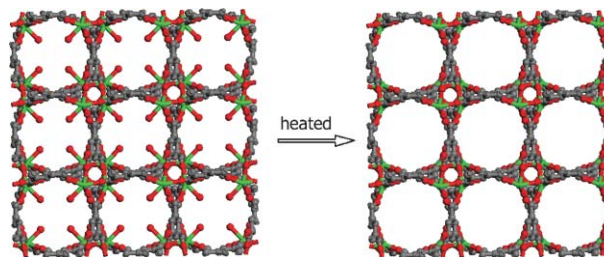


3172

### A lanthanide metal–organic framework with high thermal stability and available Lewis-acid metal sites

Xiaodan Guo, Guangshan Zhu,\* Zhongyue Li, Fuxing Sun, Zhenghong Yang and Shilun Qiu\*

A multifunctional lanthanide metal–organic framework,  $\text{Dy}(\text{BTC})(\text{H}_2\text{O}) \cdot \text{DMF}$ , exhibits excellent thermal stability, sufficient to remove the terminal coordinated molecules after calcination at  $300\text{ }^\circ\text{C}$ . The calcined sample shows a high surface area, high hydrogen and carbon dioxide storage capability, and available Lewis-acid metal sites.

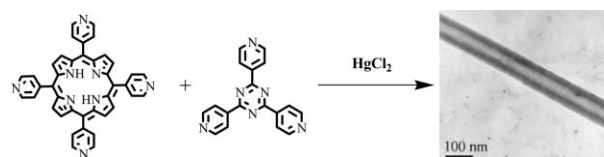


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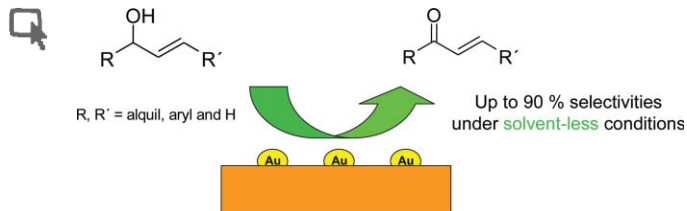
### Metal-mediated coordination polymer nanotubes of 5,10,15,20-tetrapyrridylporphine and tris(4-pyridyl)-1,3,5-triazine at the water–chloroform interface

Bing Liu, Dong-Jin Qian,\* Meng Chen, Tatsuki Wakayama, Chikashi Nakamura and Jun Miyake

Coordination polymer nanotubes have been prepared by using the  $\text{Hg}^{2+}$ -mediated co-assembly of two ligands, tetrapyrridylporphine (TPyP) and tris(4-pyridyl)-1,3,5-triazine (TPyTa), at the water–chloroform interface.



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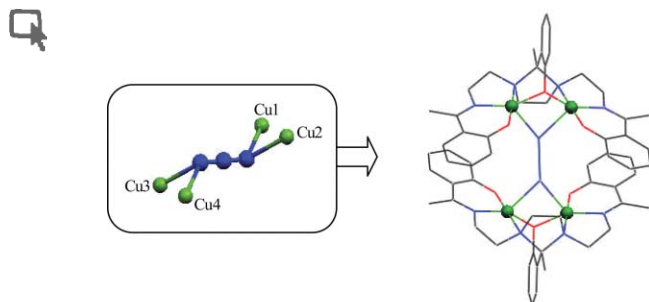


### Unique gold chemoselectivity for the aerobic oxidation of allylic alcohols

Alberto Abad, Carles Almela, Avelino Corma\* and Hermenegildo García\*

In contrast to palladium in which polymerization and isomerization occur, gold catalysts are extraordinarily selective for the solvent-less aerobic oxidation of allylic alcohols.

3181

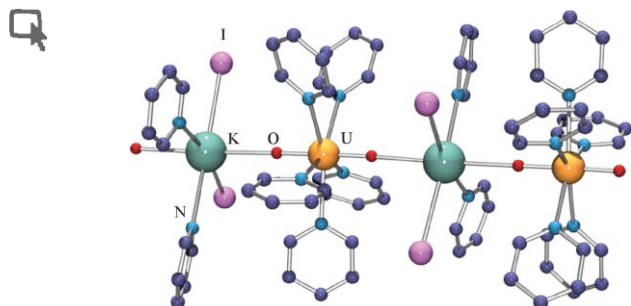


### Tetranuclear Cu(II) complex supported by a central $\mu_4$ -1,1,3,3 azide bridge

Prasant Kumar Nanda, Guillem Aromí\* and Debashis Ray\*

A novel  $\text{Cu}_4$  cluster has been assembled by connecting two dinuclear  $\text{Cu}^{\text{II}}$  moieties of a flexible amine-imine-phenol ligand *via* single  $\mu_4$ -1,1,3,3 azide bridging in a staggered conformation and characterized for its potential to mediate magnetic coupling.

3184

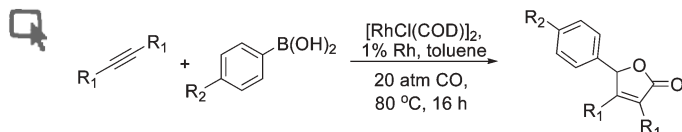


### Easy access to stable pentavalent uranyl complexes

Jean-Claude Berthet,\* Gérald Siffredi, Pierre Thuéry and Michel Ephritikhine

Reactions of  $\text{UO}_2\text{I}_2(\text{THF})_3$  or  $\text{UO}_2(\text{O}_3\text{SCF}_3)_2$  with  $\text{KC}_5\text{R}_5$  ( $\text{R} = \text{H}, \text{Me}$ ),  $\text{TiC}_5\text{H}_5$  or  $\text{K}(\text{Hg})$  in anhydrous organic solvents constitute facile and reproducible routes to stable uranyl(V) compounds.

3187



### Rhodium-catalyzed carbonylative arylation of alkynes with arylboronic acids: an efficient and straightforward method in the synthesis of 5-aryl-2(5H)-furanones

Özge Aksın, Nurcan Dege, Levent Artok,\* Hayati Türkmen and Bekir Çetinkaya

5-Aryl-2(5H)-furanones can be synthesized by the Rh-catalyzed reactions of arylboronic acids with internal alkynes under a CO atmosphere.

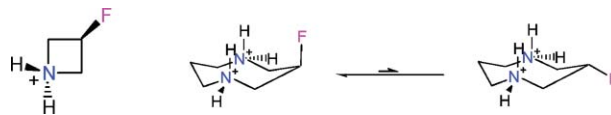


3190

### The intramolecular $\beta$ -fluorine $\cdots$ ammonium interaction in 4- and 8-membered rings

Natalie E. J. Gooseman, David O'Hagan,\*  
Alexandra M. Z. Slawin, Andrew M. Teale,  
David J. Tozer\* and Robert J. Young

The conformations of the 4-membered 3-fluoroazetidinium and the 8-membered 3-fluorodiazia ring systems have been explored by X-ray crystallography and DFT calculations and they are found to be significantly influenced by intramolecular C–F $\cdots$ N<sup>+</sup> interactions.

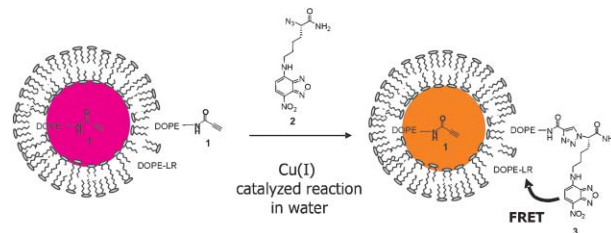


3193

### The chemical modification of liposome surfaces *via* a copper-mediated [3 + 2] azide–alkyne cycloaddition monitored by a colorimetric assay

Silvia Cavalli, Alicia R. Tipton, Mark Overhand and Alexander Kros\*

The chemical modification of liposome surfaces *via* a copper-mediated [3 + 2] azide–alkyne cycloaddition monitored by a colorimetric assay is reported.

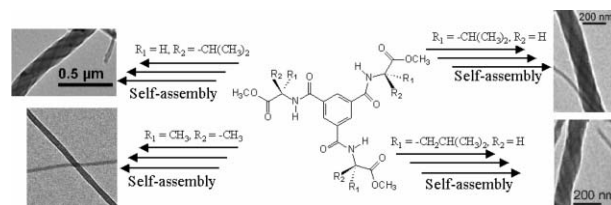


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### Formation of triple helical nanofibers using self-assembling chiral benzene-1,3,5-tricarboxamides and reversal of the nanostructure's handedness using mirror image building blocks

Partha Pratim Bose, Michael G. B. Drew, Apurba K. Das and Arindam Banerjee\*

Triple helical nanofibers with an overall handedness have been formed from self-assembling chiral benzene-1,3,5-tricarboxamides whereas, the achiral compound upon self-association gives rise to straight nanofibers without any twist.

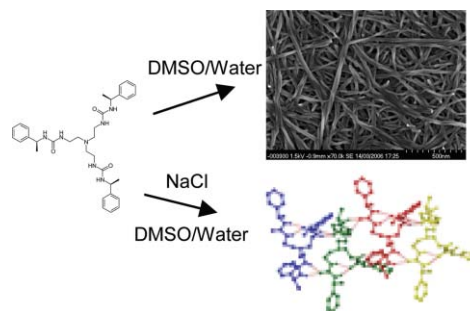


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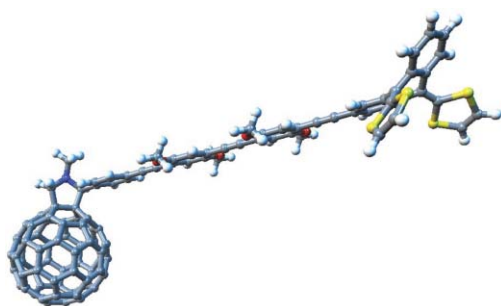
### Anion binding inhibition of the formation of a helical organogel

Claire E. Stanley, Nigel Clarke, Kirsty M. Anderson,  
Judith A. Elder, Joseph T. Lenthall and  
Jonathan W. Steed\*

A tris(urea) forms a gel in DMSO–water but is induced to crystallise upon addition of chloride.



3202

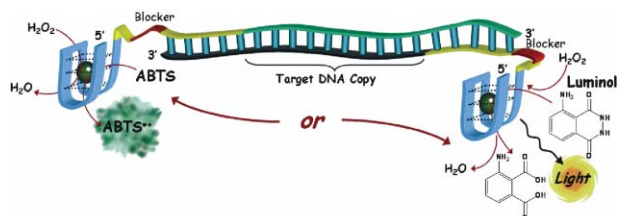


### Tuning electron transfer through *p*-phenyleneethynylene molecular wires

Carmen Atienza, Nazario Martín,\* Mateusz Wielopolski, Naomi Haworth, Timothy Clark and Dirk M. Guldi

The experimental damping factor values ( $\beta$ ) determined for oligo-*p*-phenyleneethynylenes (*o*PPE) reveal that these oligomers exhibit an efficient molecular wire behavior; these values are compared with those obtained for the analogous oligo-*p*-phenylenevinylens (*o*PPV).

3205

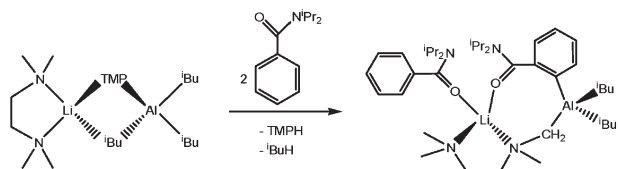


### Ultrasensitive detection of DNA by the PCR-Induced generation of DNAzymes: The DNAzyme primer approach

Zoya Cheglakov, Yossi Weizmann, Moritz K. Beissenhirtz and Itamar Willner\*

PCR-Induced generation of a DNAzyme allows ultrasensitive colorimetric or chemiluminescent detection of DNA.

3208

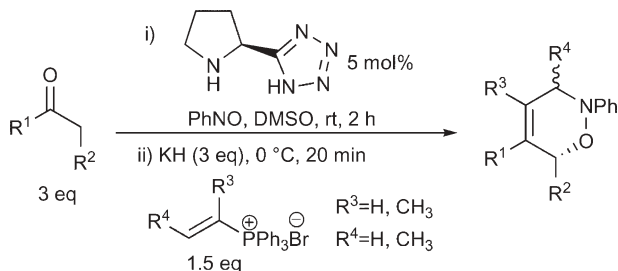


### New reactivity and structural insights of alkali-metal-mediated alumination in directed *ortho*-alumination of a tertiary aromatic amide

Joaquín García-Álvarez, David V. Graham, Alan R. Kennedy, Robert E. Mulvey\* and Susan Weatherstone

A TMEDA-stabilised lithium alkyl(TMP)aluminate reacts with *N,N*-diisopropylbenzamide to generate an unusual heterobimetallic-heterotriangular complex in which TMEDA is deprotonated.

3211



### A highly selective, organocatalytic route to chiral 1,2-oxazines from ketones

Sirirat Kumarn, David M. Shaw and Steven V. Ley\*

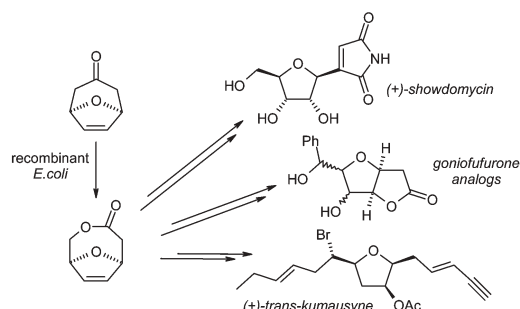
A sequential, organocatalysed asymmetric reaction to access chiral 1,2-oxazines from achiral ketone starting materials is reported, which proceeds in excellent enantioselectivity with moderate to good yields.

3214

### Accessing tetrahydrofuran-based natural products by microbial Baeyer–Villiger biooxidation

Marko D. Mihovilovic,\* Dario A. Bianchi and Florian Rudroff

A heterobicyclic lactone obtained by stereoselective Baeyer–Villiger biooxidation with recombinant whole-cells expressing cyclopentanone monooxygenase from *Comamonas* sp. NCIMB 9872 was used for formal total syntheses of various natural products containing a tetrahydrofuran structural motif.

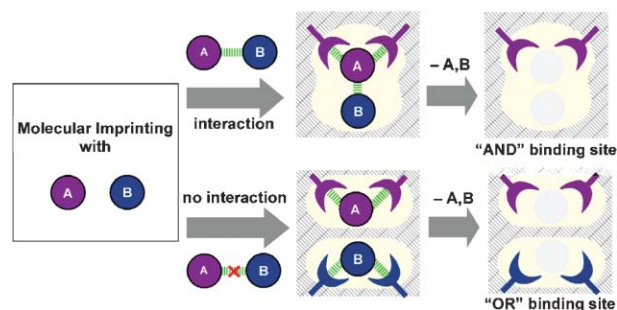


3217

### Molecularly-imprinted polymeric logic gates selective for predetermined chemical input species

Jun Matsui,\* Takuji Sodeyama, Katsuyuki Tamaki and Naoki Sugimoto\*

Polymeric logic gates (AND, OR) were composed by molecular imprinting with simultaneous use of two kinds of template species, where intermolecular interactions between the template species govern the resultant binding behaviour (AND, OR) of the imprinted polymers.

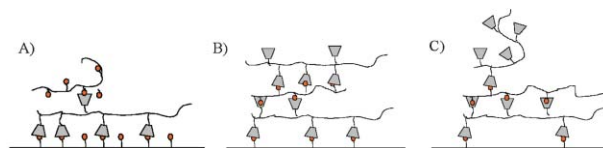


3220

### Multilayer films based on host–guest interactions between biocompatible polymers

Angéline Van der Heyden,\* Marie Wilczewski, Pierre Labbé and Rachel Auzély\*

Multilayer films are formed using host–guest interaction between two derivatized chitosans, one, with  $\beta$ -cyclodextrin cavities and the other with adamantyl moieties.

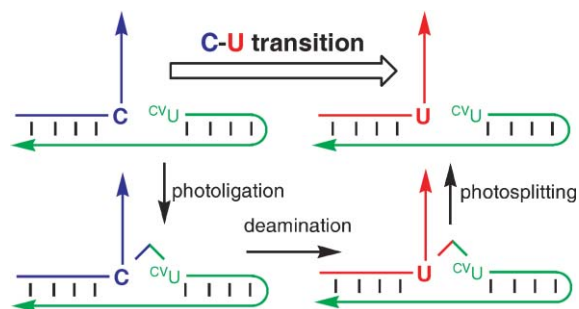


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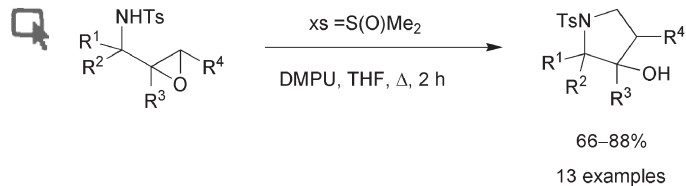
### Site-specific transition of cytosine to uracil via reversible DNA photoligation

Kenzo Fujimoto,\* Shigeo Matsuda, Yoshinaga Yoshimura, Takashi Matsumura, Masayuki Hayashi and Isao Saito

We report that deamination coupled with 5-carboxyvinyldeoxyuridine-mediated photobranching causes the heat-induced transition of cytosine to uracil with high efficiency without any side reaction.



3226

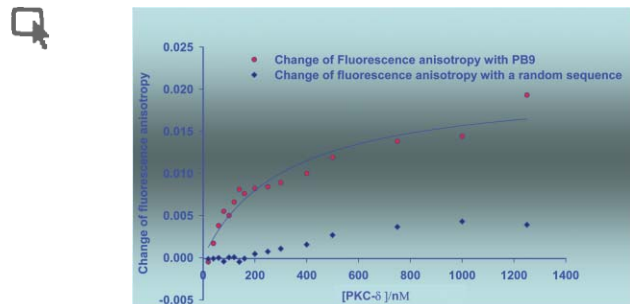


### 3-Hydroxypyrrolidines from epoxysulfonamides and dimethylsulfoxonium methylide

David M. Hodgson,\* Matthew J. Fleming, Zhaoqing Xu, Changxue Lin and Steven J. Stanway

*N*-Tosyl-protected 3-hydroxypyrrolidines are prepared by reaction of dimethylsulfoxonium methylide with readily available epoxysulfonamides.

3229

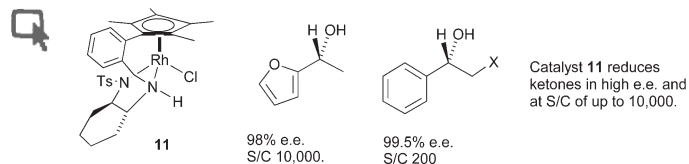


### Selection of DNA ligands for protein kinase C- $\delta$

Prabodhika Mallikaratchy, Robert V. Stahelin, Zehui Cao, Wonhwa Cho and Weihong Tan\*

CE-SELEX was employed to identify DNA ligands that specifically bind to serine-threonine kinase :PKC $\delta$ , *in vitro*

3232

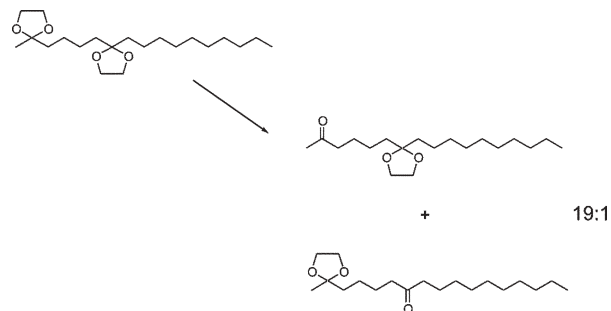


### An outstanding catalyst for asymmetric transfer hydrogenation in aqueous solution and formic acid/triethylamine

Daljit S. Matharu, David J. Morris, Guy J. Clarkson and Martin Wills\*

A Rh/tetramethylcyclopentadienyl complex containing a tethered functionality has been demonstrated to give excellent results in the asymmetric transfer hydrogenation of ketones in both aqueous and formic acid/triethylamine media.

3235



### Addressing the regioselectivity problem in organic synthesis

Fredric M. Menger\* and Hao Lu

A screening process uncovered a heterogeneous catalytic system that hydrolyzes one of two nearly identical ketals in several diketals with a high selectivity.

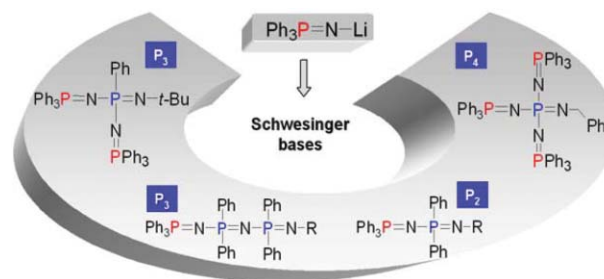


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### A new and convenient method for the synthesis of strong non-ionic bases

Marc Taillefer,\* Nicolas Rahier, Aurélien Hameau and Jean-Noël Volle\*

Various strong non-ionic phosphazene bases were obtained by a new, efficient and very simple method involving the lithium phosphonium azayliide  $\text{Ph}_3\text{P}=\text{NLi}$  as a precursor.

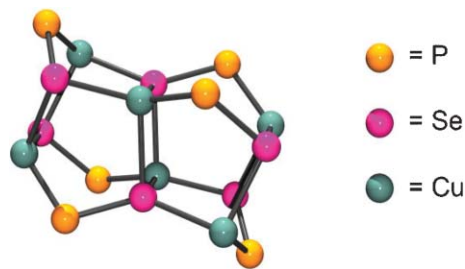


3240

### Discrete copper(I) clusters with $\text{Cu}_6\text{P}_6\text{Se}_6$ and $\text{Cu}_6\text{P}_4\text{Se}_6$ cores

Robert P. Davies,\* M. Giovanna Martinelli and Andrew J. P. White

Selenophosphinites react with copper(I) metal salts to give a new class of copper chalcogenolate cluster complex containing novel multi-metallic  $\text{Cu}_6\text{P}_6\text{Se}_6$  or  $\text{Cu}_6\text{P}_4\text{Se}_6$  cluster cores.

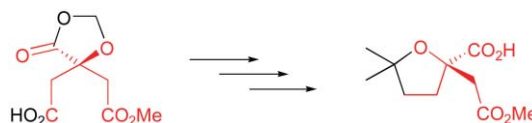


3243

### Synthesis of the ester side chains of some potentially antileukemic harringtonia alkaloids from chiral citrates

Rachael A. Ancliff, Andrew T. Russell\* and Adam J. Sanderson

Rosenmund reduction of enantiomerically enriched citrates afforded aldehydes that underwent chemoselective vinyl-Grignard addition and cyclisation to set-up a palladium mediated hydrogenolysis/acid mediated cyclisation to afford the side chains of *e.g.* anhydroharringtonine.

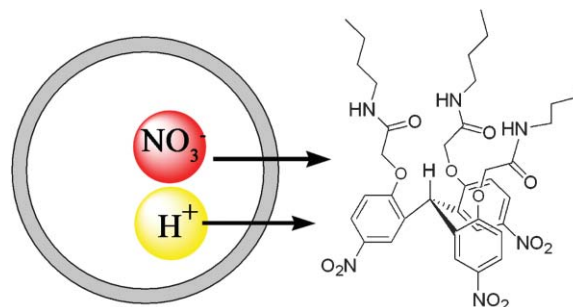


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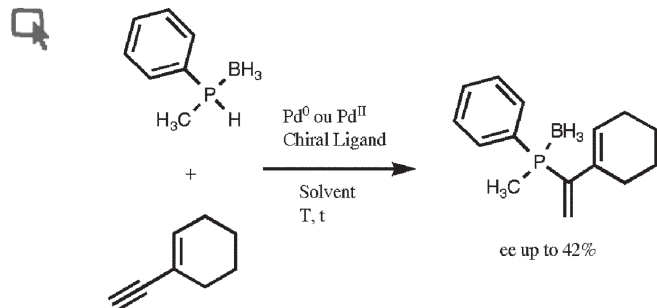
### A transmembrane anion transporter selective for nitrate over chloride

Paul V. Santacroce, Oluyomi A. Okunola, Peter Y. Zavalij and Jeffery T. Davis\*

The  $\text{C}_3$ -symmetric triamide **1** selectively transports  $\text{NO}_3^-$  anions across lipid vesicles. This  $\text{H}^+-\text{NO}_3^-$  co-transporter alters the pH inside of liposomes experiencing a  $\text{NO}_3^-/\text{Cl}^-$  gradient.



3249

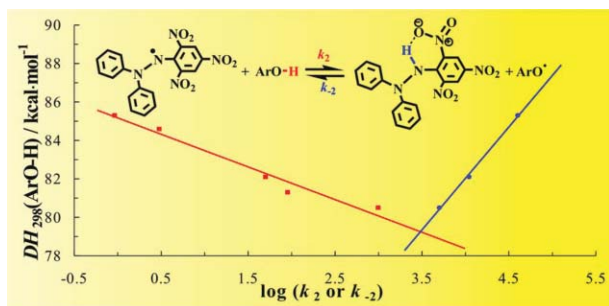


### Pallado-catalysed hydrophosphination of alkynes: access to enantio-enriched P-stereogenic vinyl phosphine-boranes

Benoît Join, David Mimeau, Olivier Delacroix and Annie-Claude Gaumont\*

The synthesis of non-racemic P-stereogenic phosphine-boranes is carried out by hydrophosphination of alkynes in the presence of a chiral catalyst.

3252



### Kinetic and thermodynamic parameters for the equilibrium reactions of phenols with the dpph<sup>•</sup> radical

Mario C. Foti\* and Carmelo Daquino

The kinetics and energetics of the reversible reaction of phenols with the dpph<sup>•</sup> radical have been studied. Steric shielding of the divalent N by the *o*-NO<sub>2</sub> in dpph<sup>•</sup> seems to be the main cause of the entropic barriers of this reaction which determine unusually low *A*-factors.

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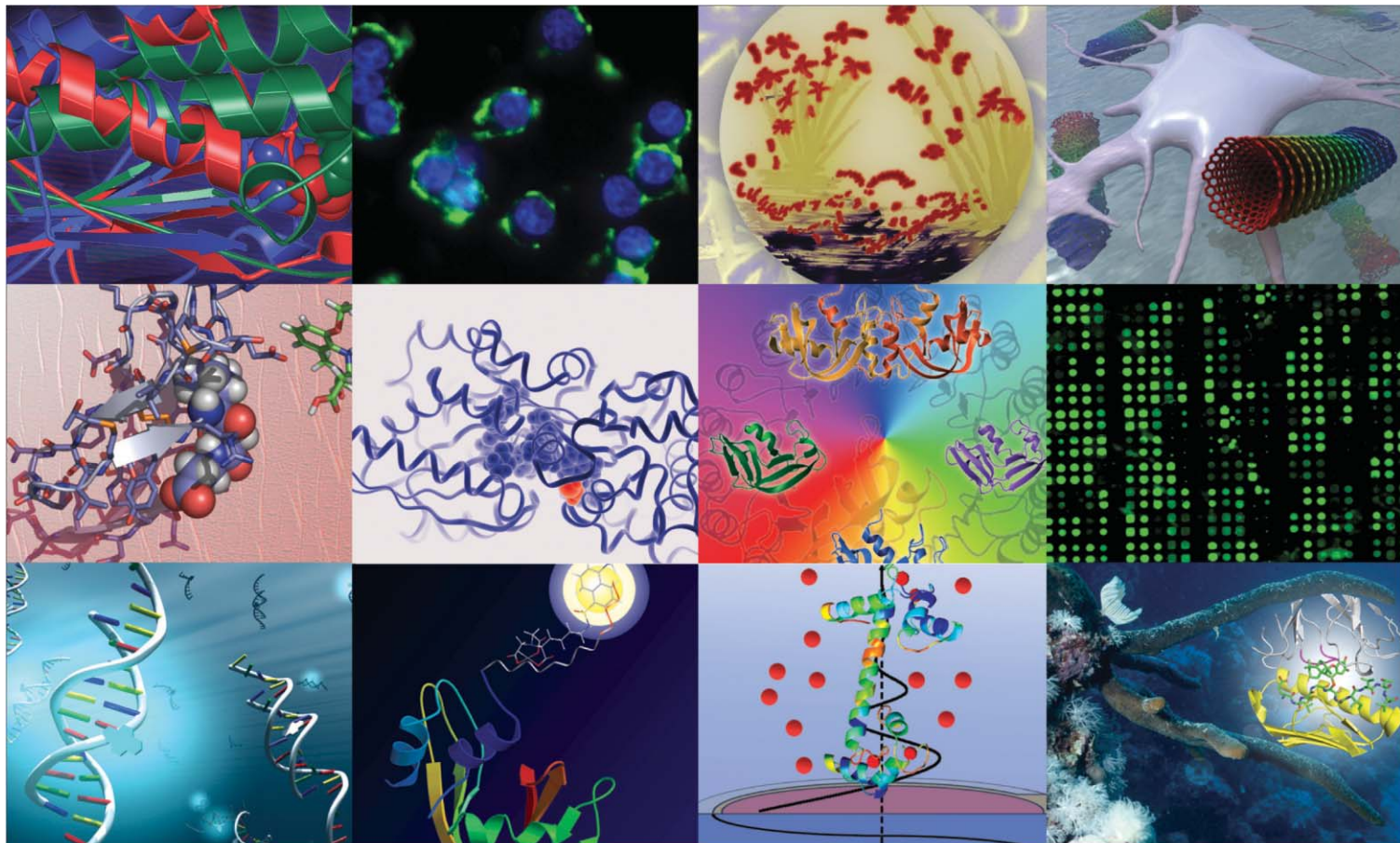
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