

# ChemComm

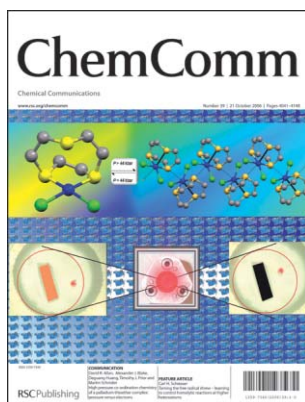
Chemical Communications

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## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (39) 4041–4148 (2006)



### Cover

See Alexander J. Blake *et al.*, page 4081.

The image represents structural and optical aspects of the pressure-induced transformation of a mononuclear, square planar Pd(II) complex into a chain polymer containing the metal in a distorted octahedral environment.

Image reproduced by permission of David R. Allan, Alexander J. Blake, Deguang Huang, Timothy J. Prior and Martin Schröder, from *Chem. Commun.*, 2006, 4081.

## CHEMICAL SCIENCE

C73

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

October 2006/Volume 3/Issue 10

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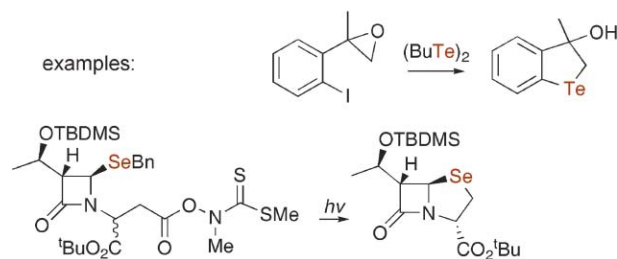
## FEATURE ARTICLE

4055

### Taming the free radical shrew – learning to control homolytic reactions at higher heteroatoms

Carl H. Schiesser

Understanding the mechanistic details of free radical homolytic substitution chemistry has led to the preparation of higher heterocycles of biological significance.



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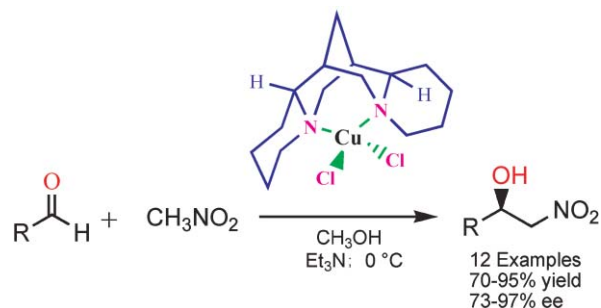
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4066

### Enantioselective nitroaldol (Henry) reaction using copper(II) complexes of (–)-sparteine

H. Maheswaran,\* K. Leon Prasanth, G. Gopi Krishna, K. Ravikumar, B. Sridhar and M. Lakshmi Kantam

The dichloro[(–)-sparteine-*N,N'*]copper(II) complex provides Henry adducts with high enantioselectivities (73–97% ee) in reaction between nitromethane and various aldehydes, particularly when the reaction is carried out in the presence of Et<sub>3</sub>N base in methanol.

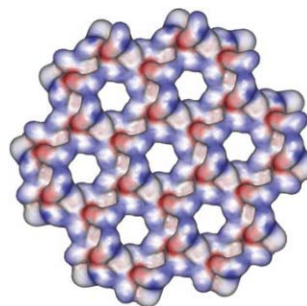


4069

### Porous 3-D honeycomb architecture by self-assembly of helical H-bonded molecular tapes

Arnaud-Pierre Schaffner, Gersande Lena, Solveig Roussel, Anne Wawrezinieck, André Aubry, Jean-Paul Briand, Claude Didierjean\* and Gilles Guichard\*

Dipeptide-derived 1,3,5-triazepan-2,6-diones form H-bonded helical molecular tapes with *P*-chirality in the solid state. In the case of compound **1a** derived from a L-phenylalanine–sarcosine sequence, chiral columnar tapes self-assemble through extensive aromatic–aromatic interactions to give hollow tubular structures.

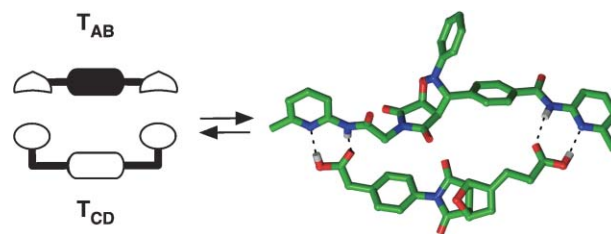


4072

### Reciprocal template effects in a simple synthetic system

Eleftherios Kassianidis and Douglas Philp\*

Two mutually-complementary templates are capable of catalysing the formation of each other, thus creating a framework for their reciprocal replication.

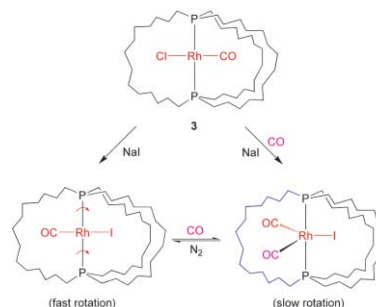


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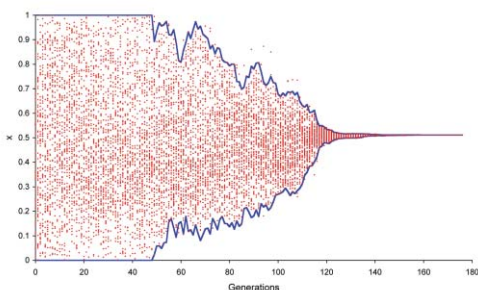
### Gyroscope-like molecules consisting of three-spoke rotators that enclose “switchable” neutral dipolar rhodium rotators; reversible cycling between faster and slower rotating Rh(CO)I and Rh(CO)<sub>2</sub>I species

Leyong Wang, Takanori Shima, Frank Hampel and J. A. Gladysz\*

Cage-like *trans*-Rh(CO)(Cl)(P((CH<sub>2</sub>)<sub>14</sub>)<sub>3</sub>P) (**3**) is prepared by a metathesis/hydrogenation sequence and converted to analogous Rh(CO)(I), Rh(CO)<sub>2</sub>(I), Rh(CO)(NCS), and Rh(CO)(Cl)(Br)(CCl<sub>3</sub>) species, modulating the rates of RhL<sub>m</sub> rotation.



4078

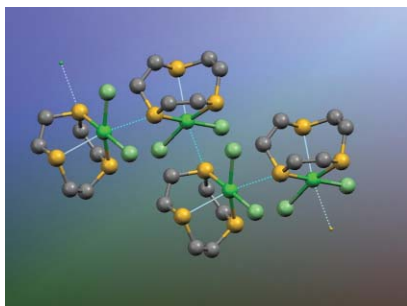


### Combined optimization using cultural and differential evolution: application to crystal structure solution from powder diffraction data

Samantha Y. Chong and Maryjane Tremayne\*

The principles of social and biological evolution have been combined in a highly efficient Cultural Differential Evolution hybrid global optimization technique and applied to crystal structure determination from powder diffraction data.

4081

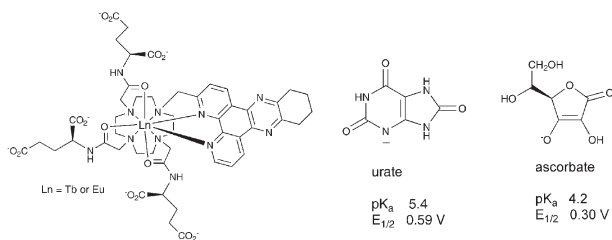


### High pressure co-ordination chemistry of a palladium thioether complex: pressure *versus* electrons

David R. Allan,\* Alexander J. Blake,\* Deguang Huang, Timothy J. Prior and Martin Schröder\*

The mononuclear Pd(II) complex *cis*-[PdCl<sub>2</sub>([9]aneS<sub>3</sub>)] ([9]aneS<sub>3</sub> = 1,4,7-trithiacyclononane) converts at 44 kbar pressure into an intensely coloured chain polymer which exhibits distorted octahedral co-ordination at the metal centre and an unprecedented [1233] conformation for the thioether crown.

4084

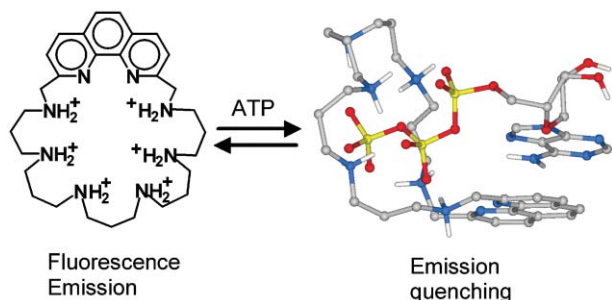


### A ratiometric and non-enzymatic luminescence assay for uric acid: differential quenching of lanthanide excited states by anti-oxidants

Robert A. Poole, Filip Kielar, Siobhan L. Richardson, Philip A. Stenson and David Parker\*

The urate anion differentially quenches the excited states of Tb and Eu complexes of a common ligand by up to a factor of 20, and exhibits >50 : 1 selectivity over ascorbate quenching, allowing its usage in the analysis of uric acid in urine.

4087



### ATP Recognition and sensing with a phenanthroline-containing polyammonium receptor

Carla Bazzicalupi, Silvia Biagini, Andrea Bencini,\* Enrico Faggi, Claudia Giorgi, Irene Matera and Barbara Valtancoli

A polyammonium macrocyclic receptor is able to recognise and sense ATP among triphosphate nucleotides, thanks to ATP-induced quenching of its fluorescence emission.

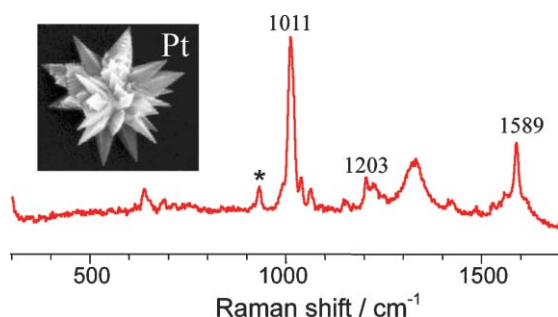


4090

### Electrochemical preparation of platinum nanothorn assemblies with high surface enhanced Raman scattering activity

Na Tian, Zhi-You Zhou, Shi-Gang Sun,\* Li Cui, Bin Ren and Zhong-Qun Tian

Platinum nanothorn assemblies with sharp tips and edges were prepared, which exhibit high SERS activity and yield an enhancement factor as high as 2000 for adsorbed pyridine.

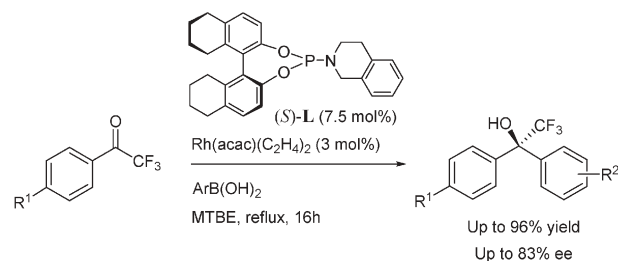


4093

### Enantioselective rhodium-catalyzed addition of arylboronic acids to trifluoromethyl ketones

Sébastien L. X. Martina, Richard B. C. Jagt, Johannes G. de Vries,\* Ben L. Feringa\* and Adriaan J. Minnaard\*

The catalytic asymmetric 1,2-addition of a series of arylboronic acids to 2,2,2-trifluoroacetophenones is described with high isolated yields (up to 96%) and good enantioselectivities (up to 83% ee) using a rhodium(I)/phosphoramidite catalyst.

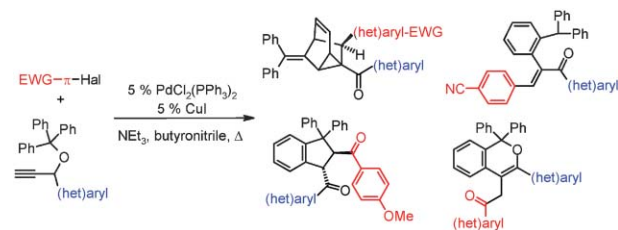


4096

### Coupling–isomerization–Claisen sequences – mechanistic dichotomies in hetero domino reactions

Daniel M. D'Souza, Frank Rominger and Thomas J. J. Müller\*

**Structural diversity by mechanistic dichotomy:** A new coupling–isomerization–Claisen domino reaction starting from electron poor halides and propargyl trityl ethers dichotomizes in the concluding steps of the sequence and gives rise to the formation of tricyclo[3.2.1.0<sup>2,7</sup>]oct-3-enes, enones, 1*H*-isochromenes, or indans as a consequence of minimal substituent effects.

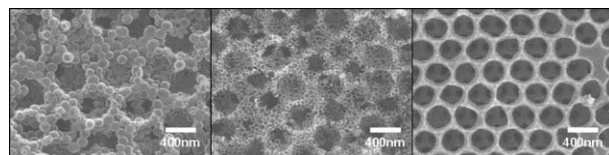


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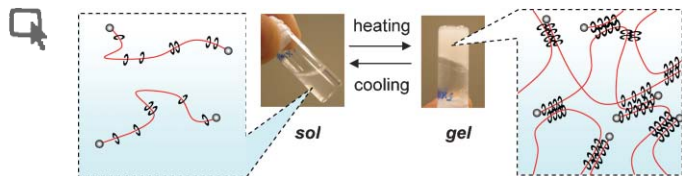
### Three-dimensionally ordered macroporous carbons having walls composed of hollow mesosized spheres

Sang-Wook Woo, Kaoru Dokko, Keiji Sasajima, Takashi Takei and Kiyoshi Kanamura\*

Three-dimensionally ordered macroporous carbons were prepared from bimodal polymer–silica colloidal crystals; the resulting carbons had interconnected macropores, and the walls of the macropores were composed of hollow mesosized spheres.



4102

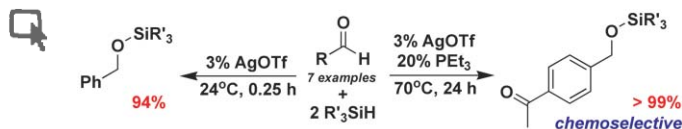


### Thermoreversible sol-gel transition of an aqueous solution of polyrotaxane composed of highly methylated $\alpha$ -cyclodextrin and polyethylene glycol

Masatoshi Kidowaki,\* Changming Zhao, Toshiyuki Kataoka and Kohzo Ito

Thermoreversible sol-gel transition is induced by localisation and delocalisation of methylated  $\alpha$ -CDs in polyrotaxane due to their hydrophobic interaction in water through LCST.

4104

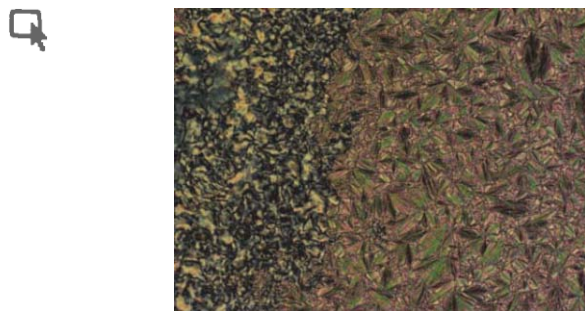


### Silver-catalyzed hydrosilylation of aldehydes

Bradley M. Wile and Mark Stradiotto\*

The first systematic application of silver species as catalysts for the hydrosilylation of unsaturated organic substrates is reported.

4107

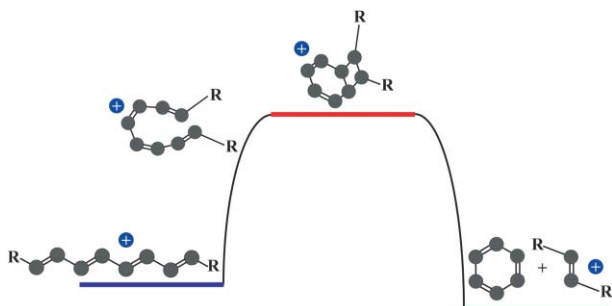


### Interfacial layer interactions: their effects on synclinc and anticlinic smectic mesophase behaviour in liquid crystals

Stephen J. Cowling\* and John W. Goodby

Through the use of bulky cyclic terminal groups, anticlinic smectic C phases have been observed in 2-methylbutyl materials based on the MHPOBC motif.

4110



### Mechanism for the elimination of aromatic molecules from polyenes in tandem mass spectrometry

Thais Guaratini, Norberto P. Lopes, Ernani Pinto, Pio Colepicolo and Paul J. Gates\*

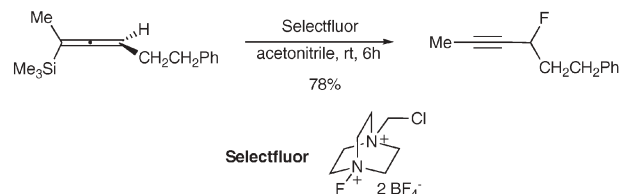
A mechanism is reported for the loss of aromatic molecules from polyene containing compounds in mass spectrometry. The requirements for the mechanism are four conjugated double bonds with a low *trans-cis* isomerisation energy.

4113

### Synthesis of propargylic fluorides from allenylsilanes

Laurence Carroll, M<sup>a</sup> Carmen Pacheco, Ludivine Garcia and Véronique Gouverneur\*

In the presence of the electrophilic fluorinating reagent Selectfluor, allenylsilanes are converted at room temperature and in good yields into the corresponding propargylic fluorides.

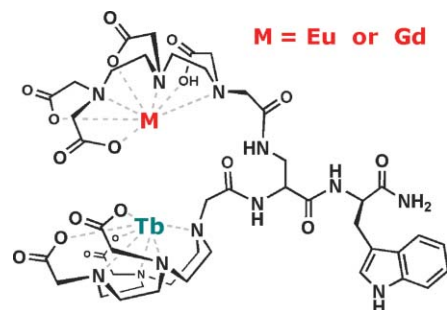


4116

### Synthesis of luminescent heterometallic bis-lanthanide complexes *via* selective, sequential metallation

Matthew S. Tremblay and Dalibor Sames\*

We report the synthesis of a branched tetrapeptide scaffold containing both DOTA and DTPA chelates and its sequential, differential metallation with two different lanthanide ions. The [Tb:DOTA][Eu:DTPA] bis-complex has interesting photophysical properties, most notably a solvent polarity-dependent ratiometric luminescence.

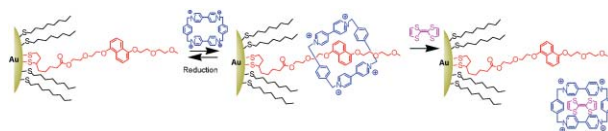


4119

### The tuneable complexation of gold nanoparticles

Graeme Cooke,\* James F. Garety, Shanika Gunatilaka Hewage, Gouher Rabani, Vincent M. Rotello and Patrice Woisel

Schematic diagram showing possible complexation process.

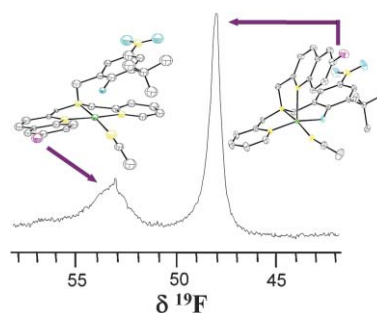


4122

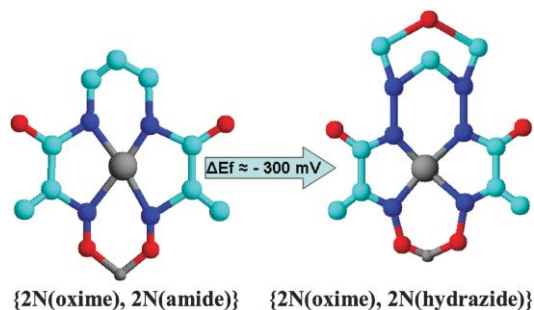
### Galactose Oxidase models: <sup>19</sup>F NMR as a powerful tool to study the solution chemistry of tripodal ligands in the presence of copper(II)

Fabien Michel, Sylvain Hamman, Fabrice Thomas,\* Christian Philouze, Isabelle Gautier-Luneau and Jean-Louis Pierre

Copper(II) complexes of <sup>19</sup>F labelled tripodal ligands have been synthesized: the protonation state of the phenol moiety and its position are shown to be correlated to the linewidth of the <sup>19</sup>F NMR signal.



4125

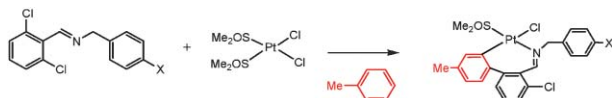


### Efficient stabilization of copper(III) in tetraaza pseudo-macrocyclic oxime-and-hydrazide ligands with adjustable cavity size

Igor O. Fritsky,\* Henryk Kozłowski,\* Olga M. Kandalak, Matti Haukka, Jolanta Świątek-Kozłowska, Elżbieta Gumienna-Kontecka and Franc Meyer

Substitution of the amide donors in open-chain {2N(oxime), 2N(amide)} ligands by hydrazide donors gives new pseudo-macrocyclic copper complexes that show a significant decrease of the Cu<sup>3+/2+</sup> redox potentials.

4128

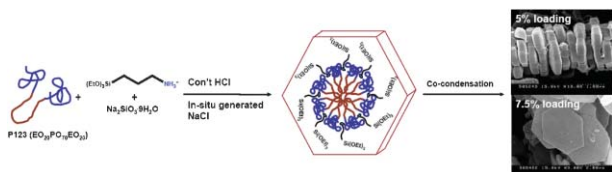


### Unprecedented intermolecular C–H bond activation of a solvent toluene molecule leading to a seven-membered platinumacycle

Alejandro Capapé, Margarita Crespo,\* Jaume Granell,\* Albert Vizcarro, Javier Zafrilla, Mercè Font-Bardía and Xavier Solans

A novel platinum-mediated process involving intermolecular activation of a C<sub>aryl</sub>–H bond of toluene, intramolecular activation of an imine C<sub>aryl</sub>–Cl bond and formation of a C–C bond is reported.

4131

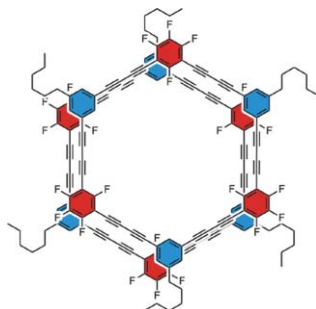


### Amino-functionalized SBA-15 type mesoporous silica having nanostructured hexagonal platelet morphology

Sujandi, Sang-Eon Park,\* Dae-Soo Han, Sang-Cheol Han, Myung-Jong Jin and Tetsu Ohsuna

Amino-functionalized SBA-15 having an unusually wide and thin hexagonal platelet morphology with submicrometer length (100–300 nm) pore channels running parallel to the thickness of the hexagonal plates has been synthesized directly using microwave irradiation.

4134



### Shape-persistent macrocycle with a self-complementary recognition pattern based on diacetylene-linked alternating hexylbenzene and perfluorobenzene rings

Lijin Shu and Marcel Mayor\*

The macrocycle, consisting of alternating electron rich and electron poor sub-units, displays a considerably enlarged dimerization tendency in solution, which is investigated by <sup>1</sup>H NMR titration and vapour pressure osmometry.

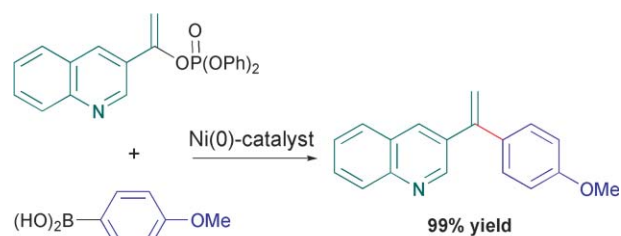


4137

**Direct synthesis of 1,1-diarylalkenes from alkenyl phosphates *via* nickel(0)-catalysed Suzuki–Miyaura coupling**

Anders L. Hansen, Jean-Philippe Ebran,  
Thomas M. Gøgsig and Troels Skrydstrup\*

Suzuki–Miyaura coupling of 1-aryalkenyl phosphates with aryl boronic acids/esters with a Ni(0)-catalyst provides a convenient route to 1,1-diarylalkenes in good to excellent yields.

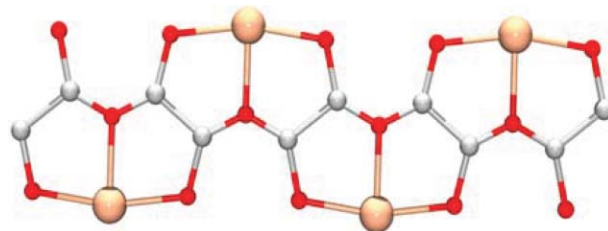


4140

**[Zn<sub>n</sub>(polyox)(pmtz)<sub>n</sub>]: the first polyoxalate-containing coordination polymer from an unforeseen chemical rearrangement of 5-pyrimidyl-tetrazole under hydrothermal conditions**

A. Rodríguez-Diéguez and E. Colacio\*

Reaction of ZnCl<sub>2</sub> with Napmtz (pmtz<sup>-</sup> = 5-(pyrimidyl)tetrazolate anion) under hydrothermal conditions yields the title compound, which represents the first structural report on a polyoxalate-containing complex.

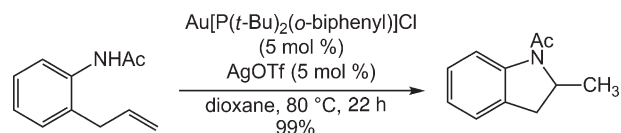


4143

**Gold(I)-catalyzed intramolecular hydroamination of unactivated alkenes with carboxamides**

Christopher F. Bender and Ross A. Widenhoefer\*

*N*-Alkenyl carboxamides undergo gold-catalyzed intramolecular *exo*-hydroamination to form nitrogen heterocycles in excellent yield.



## ADDITIONS AND CORRECTIONS

4145

**A modular approach to the synthesis of 2,3,4-trisubstituted tetrahydrofurans**

Christopher G. Nasveschuk, Nathan T. Jui and Tomislav Rovis

**The tuneable complexation of gold nanoparticles**

Graeme Cooke, James F. Garety,  
Shanika Gunatilaka Hewage, Gouher Rabani,  
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**Porous 3-D honeycomb architecture by self-assembly of helical H-bonded molecular tapes**


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Solveig Roussel, Anne Wawrezynieck, André Aubry,  
Jean-Paul Briand, Claude Didierjean and  
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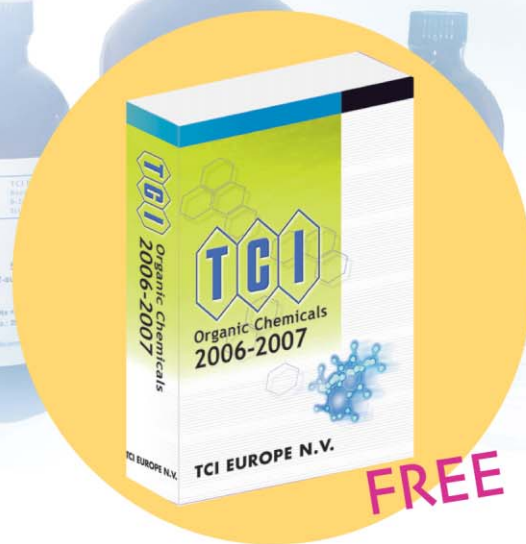
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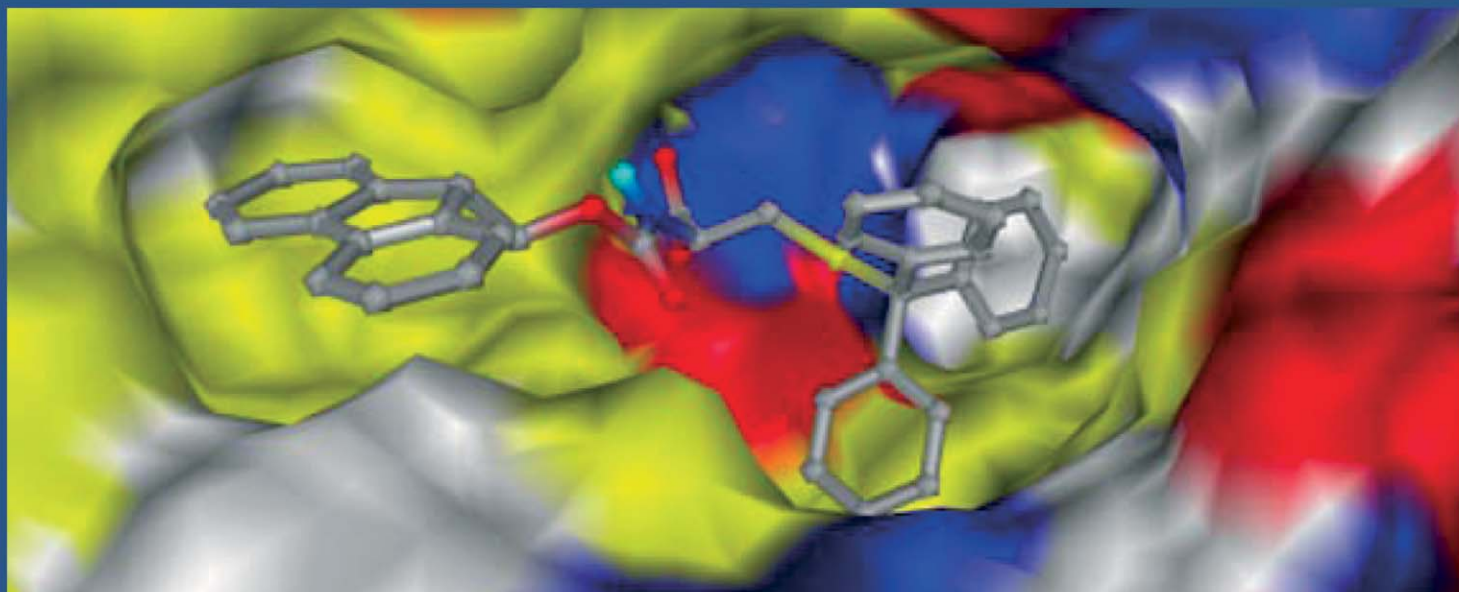
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