#### IN THIS ISSUE

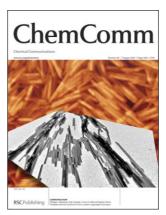
ISSN 1359-7345 CODEN CHCOFS (29) 3621-3744 (2005)



#### Cover

See Fumio Toda *et al.*, page 3646.

Packing diagram of the inclusion complex of axial chlorocyclohexane and 9,9'-bianthryl host viewed along the pivot bond of the host. Image reproduced by permission of Shinya Hirano, Shinji Toyota, Masako Kato and Fumio Toda from *Chem. Commun.*, 2005, 3646.



Inside cover
See Stephen Mann et al.,
page 3688.
Silica-coated nanotapes of
self-assembled porphyrin
J-aggregates are
synthesized by
supramolecular templating.
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J. Meadows, Erik Dujardin,
Simon R. Hall and Stephen
Mann from Chem.
Commun., 2005, 3688.

#### **CHEMICAL SCIENCE**

C57

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**

August 2005/Volume 2/Issue 8

www.rsc.org/chemicalscience

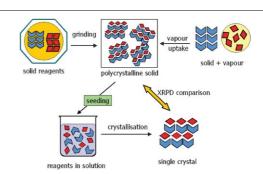
#### **FEATURE ARTICLE**

3635

### Making crystals from crystals: a green route to crystal engineering and polymorphism

Dario Braga\* and Fabrizia Grepioni\*

Supramolecular reactions between crystalline materials or reactions between a crystalline material and a vapour can be used to generate new crystalline substances. These solvent-free processes can be exploited to prepare hydrogen-bonded co-crystals and coordination networks by non-solution solvent-free methods.



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Isolation of axial conformers of chloro- and bromocyclohexane in a pure state as inclusion complexes with 9,9'-bianthryl, and the discovery of a novel 1,3 diaxial Cl···H weak interaction

Shinya Hirano, Shinji Toyota, Masako Kato\* and Fumio Toda\*

Axial conformers of chloro- and bromocyclohexane were isolated in a pure state as 1:1 inclusion complexes with 9,9'-bianthryl. A 1,3 diaxial Cl···H weak interaction was discovered in the axial chlorocyclohexane.



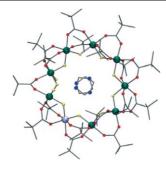


3649

#### Influencing the nuclearity and constitution of heterometallic rings via templates

Grigore A. Timco,\* Andrei S. Batsanov, Finn K. Larsen, Christopher A. Muryn, Jacob Overgaard, Simon J. Teat and Richard E. P. Winpenny\*

Octa-, nona- and deca-nuclear rings can be made by choice of amines or N-heterocycles as templates.



3652

#### Identification of broad specificity P450<sub>CAM</sub> variants by primary screening against indole as substrate

Ayhan Çelik, Robert E. Speight and Nicholas J. Turner\*

Primary screening of cytochrome P450<sub>CAM</sub> libraries, using oxidation of indole to indigo as a colorimetric detection method, leads to the identification of variants with activity towards diphenylmethane.

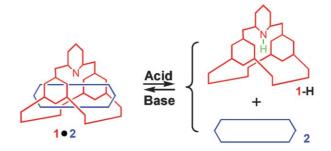


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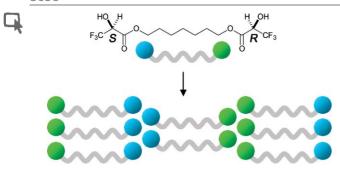
#### pH-Controlled assembly and disassembly of a cryptand/paraquat [2]pseudorotaxane

Feihe Huang, Karen A. Switek and Harry W. Gibson\*

It was demonstrated that pseudorotaxane formation between a pyridine-containing cryptand and paraquat can be reversibly switched off (and back on) by adding acid (and then base).



3658

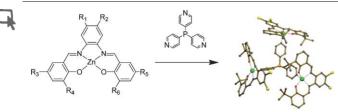


A binary hydrogen bonding motif based on homochiral recognition: crystal structures and hydrogen bonding networks of meso-(R,S)-bis(trifluorolactate)s

Satoshi Takahashi, Toshimasa Katagiri\* and Kenji Uneyama

A series of *meso*-compounds incorporating both enantiomers of a trifluorolactate constructed two-dimensional supramolecular sheets *via* homochiral hydrogen bonding chains as a binary hydrogen bonding motif.

3661

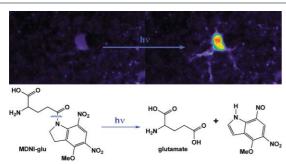


Encapsulated transition metal catalysts comprising peripheral Zn(II)salen building blocks: template-controlled reactivity and selectivity in hydroformylation catalysis

Arjan W. Kleij, Martin Lutz, Anthony L. Spek, Piet W. N. M. van Leeuwen and Joost N. H. Reek\*

Template-assisted catalyst encapsulation using zinc(II)—salphen building blocks: small changes in the ligand-template have a large effect on the structure of the assembly and consequently on the activity and selectivity of the hydroformylation catalysts formed.

3664

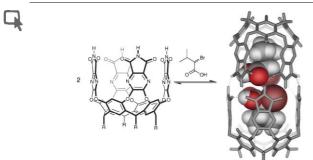


Synthesis of a caged glutamate for efficient one- and two-photon photorelease on living cells

Olesya D. Fedoryak, Jai-Yoon Sul, Philip G. Haydon and Graham C. R. Ellis-Davies\*

Synthesis and uncaging of MDNI-glu, a biologically inert, photosensitive derivative of the major excitatory neurotransmitters (L-glutamate) is described. MDNI-glu makes greater than 10-fold better use of incident light than all previous caged glutamates.

3667



Diastereoselection of chiral acids in a cylindrical capsule

Liam C. Palmer, Yi-Lei Zhao, K. N. Houk and Julius Rebek, Jr.\*

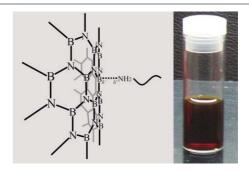
The cylindrical dimeric capsule binds two chiral carboxylic acids with modest selectivity for homochiral or heterochiral pairs. The selectivity is observed by NMR and further investigated by Monte Carlo conformational searching.

#### 3670

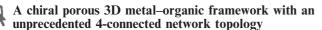
#### Solubilization of boron nitride nanotubes

Su-Yuan Xie, Wei Wang, K. A. Shiral Fernando, Xin Wang, Yi Lin and Ya-Ping Sun\*

A successful attempt in the functionalization and solubilization of boron nitride nanotubes is reported, and a functionalization mechanism based on interactions of amino functional groups with nanotube surface borons is proposed.

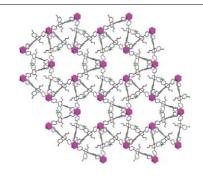


#### 3673

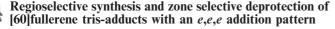


Chuan-De Wu and Wenbin Lin\*

A novel homochiral 3D metal—organic framework with an unprecedented 4-connected network topology was constructed based on the *cis*-geometry of the octahedral Cd nodes and was shown to possess permanent porosity.

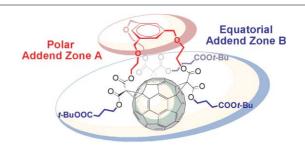


#### 3676

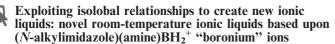


Florian Beuerle, Nikos Chronakis and Andreas Hirsch\*

We present an elegant route for the regioselective synthesis of e,e,e [60] fullerene tris-adducts utilizing  $D_{3h}$ -symmetrical tripodal tethers. The selective deprotection of the topologically distinct polar and equatorial addend zones provides a facile access to novel functionalized fullerene tris-adducts.

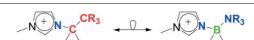


#### 3679

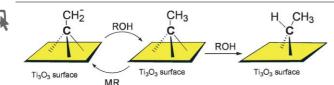


Phillip A. Fox, Scott T. Griffin, W. Matthew Reichert, E. Alan Salter, Ashley B. Smith, Morgan D. Tickell, Benjamin F. Wicker, Eugene A. Cioffi, James H. Davis, Jr.,\* Robin D. Rogers\* and Andrzej Wierzbicki

Readily prepared imidazole-based boronium ions form stable, hydrophobic, room-temperature ionic liquids (RTIL) with unique electronic and spectroscopic characteristics.



3682

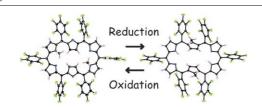


## Hydrocarbon species $\mu_3\text{-CCH}_2^-,\,\mu_3\text{-CCH}_3$ and $\mu\text{-CHCH}_3$ supported on $Ti_3O_3$

Octavio González-del Moral, Avelino Martín, Miguel Mena,\* María del Carmen Morales-Varela and Cristina Santamaría

The results here reported show the use of the molecular oxide model [{TiCp\*( $\mu$ -O)}<sub>3</sub>( $\mu$ <sub>3</sub>-CMe)] in the study of the transformation of the  $\mu$ <sub>3</sub>-ethylidyne unit into the hydrocarbon fragments  $\mu$ <sub>3</sub>-CCH<sub>2</sub><sup>-</sup> and  $\mu$ -CHCH<sub>3</sub>.

3685



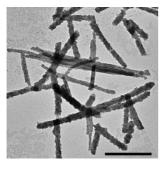
# Reversible caterpillar-motion like isomerization in a N,N'-dimethyl hexaphyrin(1.1.1.1.1) induced by two-electron oxidation or reduction

Masaaki Suzuki and Atsuhiro Osuka\*

A caterpillar-motion like rotational isomerization of N, N'-dimethyl hexaphyrins can be induced via two-electron reduction or oxidation.

3688





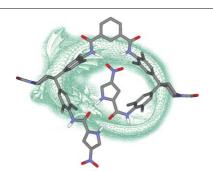
### Template-directed synthesis of silica-coated J-aggregate nanotapes

Philippa J. Meadows, Erik Dujardin, Simon R. Hall and Stephen Mann\*

Hybrid nanofilaments, consisting of an optically responsive core of stacked porphyrin anions encased within an ultrathin shell of amorphous silica, have been prepared by using self-assembled J-aggregate supramolecular nanotapes as templates for controlled inorganic deposition.

3691





#### Tailbiter: a new amide foldamer

Christopher A. Hunter,\* Andrea Spitaleri and Salvador Tomas\*

<sup>1</sup>H NMR chemical shifts were used to determine the three-dimensional structure of a new polyamide foldamer in solution.

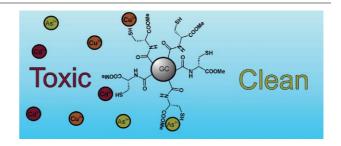
#### 3694

Cysteine methyl ester modified glassy carbon spheres for removal of toxic heavy metals from aqueous media

Gregory G. Wildgoose, Henry C. Leventis, Andrew O. Simm, John H. Jones and

Richard G. Compton\*

An inexpensive, highly effective method for removing toxic metal ions, such as cadmium(II), copper(II) and arsenic(III), from aqueous media involving the use of cysteine methyl ester modified glassy carbon spheres is presented with potential applications in environmental cleanup or third world drinking water filtration.

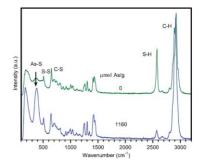


#### 3697

#### Trapping of arsenite by mercaptopropyl-functionalized mesostructured silica with a wormhole framework

Emily McKimmy, Joel Dulebohn, Jainisha Shah and Thomas J. Pinnavaia\*

Thiol-functionalized mesostructured silica, prepared by direct assembly methods, is an effective trapping agent for the removal of arsenite from water.

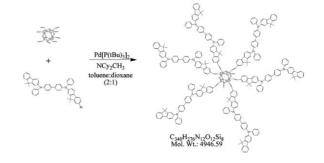


#### 3700

Heck coupling of haloaromatics with octavinylsilsesquioxane: solution processable nanocomposites for application in electroluminescent devices

Alan Sellinger,\* Ryo Tamaki, Richard M. Laine, Kazunori Ueno, Hiroshi Tanabe, Evan Williams and Ghassan E. Jabbour

A new hole transport functionalised, solution-processable nanocomposite has been prepared via the Heck coupling of octavinylsilsesquioxane with a selected bromoaromatic.



#### 3703

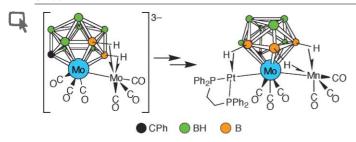


Enantioselective total synthesis of bioactive natural product (+)-Sch 642305: a structurally novel inhibitor of bacterial DNA primase and HIV-1 Tat transactivation

Goverdhan Mehta\* and Harish M. Shinde

The total synthesis of the bioactive natural product (+)-Sch 642305, a potent inhibitor of bacterial DNA primase and HIV-1 Tat transactivation, has been achieved with an overall yield of 12%. Starting with a readily available chiral building block, the synthesis highlights the use of an RCM protocol for the efficient construction of the key decalactone moiety.

3706

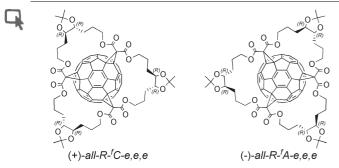


The bis-molybdenum(0) trianion  $[1,3,6-\{Mo(CO)_3\}-3,6-(\mu-H)_2-1,1,1-(CO)_3-2-Ph-closo-1,2-MoCB_9H_7]^3$  and its use as a scaffold in the assembly of heteropolymetallic complexes

Peng Lei, Thomas D. McGrath and F. Gordon A. Stone\*

The title dimolybdenacarborane trianion undergoes successive addition of manganese and platinum fragments to give a neutral trimetallic species.

3709

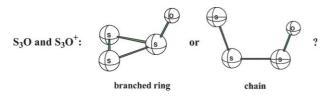


Regio- and stereoselective synthesis of enantiomerically pure [60] fullerene tris-adducts with an inherently chiral e,e,e addition pattern

Nikos Chronakis and Andreas Hirsch\*

The regio- and stereoselective synthesis of the enantiomerically pure [60]fullerene tris-adducts (+)-all- $R^{-f}C$ -e,e,e and (–)-all- $R^{-f}A$ -e,e,e was achieved via the Bingel cyclopropanation of  $C_{60}$  with a chiral  $D_3$ -symmetrical cyclo-tris(malonate) tether. The  $^fC$  and  $^fA$  enantiomers were separated and purified by simple column chromatography on SiO<sub>2</sub>.

3712

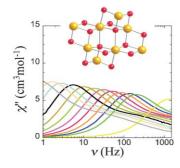


Structures of the trisulfur oxides S<sub>3</sub>O and S<sub>3</sub>O<sup>\*+</sup>: branched rings, not open chains

Ming Wah Wong\* and Ralf Steudel

Higher-level *ab initio* calculations showed that the global energy minimum for both  $S_3O$  and  $S_3O^{*+}$  is a branched, three-membered ring, not an open chain form.

3715



A novel octanuclear Mn(III) aggregate as a single-molecule magnet

Meenal D. Godbole, Olivier Roubeau,\* Rodolphe Clérac, Huub Kooijman, Anthony L. Spek and Elisabeth Bouwman\*

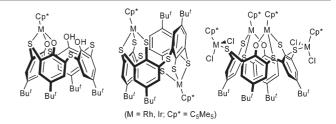
The dc and ac magnetic properties of the novel complex [Mn<sub>8</sub>O<sub>2</sub>(OH)<sub>2</sub>(OMe)<sub>12</sub>(OAc)<sub>2</sub>(Mesalim)<sub>4</sub>] evidence its high spin ground state and SMM behaviour. The complex is a new addition to the relatively small number of single molecule magnets.

#### 3718

#### Syntheses and structures of mono-, di- and tetranuclear rhodium or iridium complexes of thiacalix[4]arene derivatives

Kenji Hirata, Toshiaki Suzuki, Ai Noya, Izuru Takei and Masanobu Hidai\*

The reactions of  $[Cp*MCl_2]_2$  (M = Rh, Ir) with thiacalix[4]arene and tetramercaptothiacalix[4]arene gave [(Cp\*M) $\{\eta^3$ -TC4A(OH)<sub>2</sub>(O)<sub>2</sub> $\}$ ] and [(Cp\*M)<sub>2</sub> $\{\eta^3$ -TC4A(S)<sub>4</sub> $\}$ ] respectively, while the analogous reactions with dimercaptothiacalix[4]arene produced the tetranuclear complexes  $[(Cp*M)_2(Cp*MCl_2)_2\{\eta^3:\eta^3:\eta^1:\eta^1-\eta^2\}]$  $TC4A(O)_2(S)_2$ ].



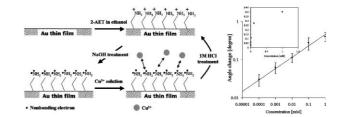
3721



#### Fabrication of reusable sensor for detection of Cu<sup>2+</sup> in an aqueous solution using a self-assembled monolayer with surface plasmon resonance spectroscopy

Taewook Kang, Surin Hong, Jungwoo Moon, Seogil Oh and Jongheop Yi\*

The proposed procedure for recycling the sensor surface consists of (1) the self-assembly of 2-aminoethanethiol hydrochloride, (2) the neutralization of zwitterion-like species,  $-NH_3^+Cl^-$  to  $-NH_2$ , (3) the detection of  $Cu^{2+}$ , and finally (4) regeneration of the sensor surface.

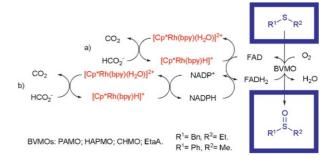


#### 3724

#### [Cp\*Rh(bpy)(H<sub>2</sub>O)]<sup>2+</sup> as a coenzyme substitute in enzymatic oxidations catalyzed by Baeyer-Villiger monooxygenases

Gonzalo de Gonzalo, Gianluca Ottolina,\* Giacomo Carrea and Marco W. Fraaije

The organometallic complex [Cp\*Rh(bpy)H]+ can be used as a coenzyme substitute in Baeyer-Villiger monooxygenase catalyzed oxidations. The presence of NADPH enhances enzyme enantioselectivity by properly shaping the active site of these enzymes.



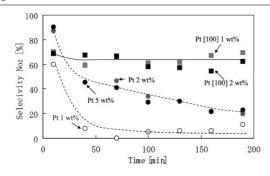
### 3727

#### Probing the mechanism of Prins cyclisations and application to the synthesis of 4-hydroxytetrahydropyrans

Conor S. Barry, Nick Bushby, John R. Harding, Rachael A. Hughes, Gregory D. Parker, Richard Roe and Christine L. Willis\*

Reaction of homoallylic MEM ethers with acid gives intermediates which have been trapped with carbon-based nucleophiles. A further insight into the factors affecting such reactions enables the design of efficient syntheses of 4-hydroxy-2,6-disubstituted tetrahydropyrans.

3730



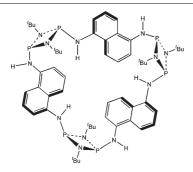
### Nitrite reduction on morphologically controlled Pt nanoparticles

Akane Miyazaki,\* Toru Asakawa, Yoshio Nakano and Ioan Balint

On the surface of morphologically controlled Pt nanoparticles, statistically rich in [100] crystalline facet, nitrite ions were reduced by  $H_2$  to  $N_2$  with high and stable selectivity.

3733





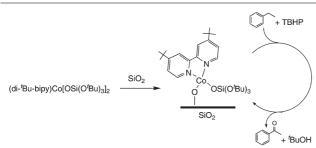
Synthesis and structure of the calixarene-like phosph(III)azane macrocycle  $[\{P(\mu-N^tBu)\}_2\{1,5-(NH)_2C_{10}H_6\}]_3$ 

Fay Dodds, Felipe García, Richard A. Kowenicki, Mary McPartlin, Alexander Steiner and Dominic S. Wright\*

The trimeric macrocycle  $[\{P(\mu-N^tBu)\}_2\{1,5-(NH)_2C_{10}H_6\}]_3$  (1) is the largest macrocycle of this type to be reported, possessing a cone-shaped (calixarene-like) arrangement with an 8.8 Å diameter cavity.

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Liquid-phase oxidation of alkylaromatics by a H-atom transfer mechanism with a new heterogeneous CoSBA-15 catalyst

Richard L. Brutchey, Ian J. Drake, Alexis T. Bell and T. Don Tilley\*

A new pseudo-tetrahedral Co(II) complex has been grafted onto the surface of SBA-15 and successfully utilized for the catalytic oxidation of alkylaromatic substrates with *tert*-butyl hydroperoxide *via* an H-atom transfer mechanism.

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Supramolecular self-assembly on a solid support: metal-directed complementarity

Edwin C. Constable,\* Catherine E. Housecroft, John N. Lambert\* and Dan A. Malarek

Resin bound bis(2,2'-bipyridine)s template the formation of the complementary bis(2,2':6',2"-terpyridine)s in the presence of copper(II).

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Supramolecular self-assembly on a solid support: metal-directed complementarity

Edwin C. Constable, Catherine E. Housecroft, John N. Lambert and Dan A. Malarek

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