## ChemComm

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#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (8) 765-880 (2007)



**Cover** See M. Shao *et al.*, page 793. Karman vortex street, a new way to nanomaterial arrays. Image reproduced by permission of Mingwang Shao, Hui Hu, Min Li, Huizhao Ban, Mengya Wang and Juan Jiang from *Chem. Commun.*, 2007, 793.

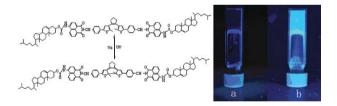
#### FEATURE ARTICLE

#### 781

#### Photochromic bisthienylethene as multi-function switches

#### He Tian\* and Sheng Wang

Bisthienylethenes (BTEs) are one of the most promising families of photochromic compounds for use in optoelectronic devices such as ultrahigh-density optical data storage, molecular switches, logic gates, sensors, *etc.* This article describes recent development of multi-switchable photochromic BTE materials and also highlights our recent contributions in this field.



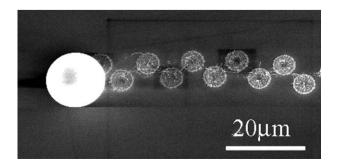
#### COMMUNICATION

#### 793

Karman vortex street assisted patterning in the growth of silicon nanowires

Mingwang Shao,\* Hui Hu, Min Li, Huizhao Ban, Mengya Wang\* and Juan Jiang

A Karman vortex street was employed to pattern catalysts and grow nanomaterial arrays, which were made of a disk-like superstructure built of silicon nanowires; there also existed nanowires connected with the disks.



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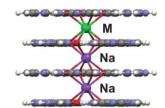
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#### 795

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#### Direct <sup>23</sup>Na NMR observation of mixed cations residing inside a G-quadruplex channel

#### Ramsey Ida, Irene C. M. Kwan and Gang Wu\*

The first direct <sup>23</sup>Na NMR observation is reported for mixed cations (Na<sup>+</sup>/K<sup>+</sup>, Na<sup>+</sup>/Rb<sup>+</sup>, Na<sup>+</sup>/Sr<sup>2+</sup>) residing inside a G-quadruplex channel.

#### 798

#### A tetradecanuclear copper dimeric macrocyclic complex with a body-centred heptanuclear core-structure and magnetism

Santokh S. Tandon,\* Scott D. Bunge and Laurence K. Thompson\*

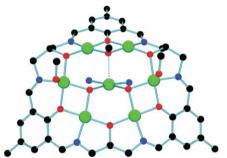
2,6-diformyl-4-methylphenol and 1,3-diamino-2hydroxypropane template condense with Cu(NO<sub>3</sub>)<sub>2</sub> and azide to produce a 3 : 3 macrocycle containing seven spin-coupled copper(II) ions, where the seventh metal completes a bodycentred heptanuclear lattice.

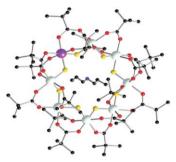
#### 801

#### Al, Ga and In heterometallic wheels and their by-products

E. Carolina Sañudo, Christopher A. Muryn, Madeleine A. Helliwell, Grigore A. Timco, Wolfgang Wernsdorfer and Richard E. P. Winpenny\*

Cyclic octanuclear complexes, each containing seven group 13 metals and one d-block metal are reported and preliminary physical characterisation of the compounds discussed.



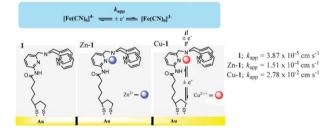


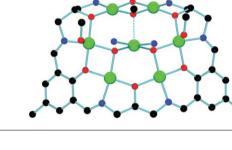
#### 804

#### Metal-mediated transport of electrons across molecular films

V. Ganesh, Maria Pilar Calatayud Sanz and Juan C. Mareque-Rivas\*

Electron transfer (ET) to a redox probe in solution across the self-assembled monolayers (SAMs) of a tris-(2-pyridylmethyl)amine-based ligand on gold electrodes is greatly enhanced by Cu-binding.







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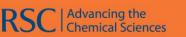


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#### 807

#### Ambient generation of fatty acid methyl ester ions from bacterial whole cells by direct analysis in real time (DART) mass spectrometry

Carrie Y. Pierce, John R. Barr, Robert B. Cody, Robert F. Massung, Adrian R. Woolfitt, Hercules Moura, Herbert A. Thompson and Facundo M. Fernandez\*

**Vacuum is not the limit**: Direct analysis in real time (DART), a new ionization method for ambient mass spectrometry, allows to directly probe microbial samples in the open air, bypassing lengthy sample preparation steps, and enhancing throughput by several orders of magnitude.

810

#### Drastic effects of the second coordination sphere on neutral vs. anionic guest binding to a biomimetic Cu(II) center embedded in a calix[6]aza-cryptand

Guillaume Izzet, Xianshun Zeng, Huriye Akdas, Jérôme Marrot and Olivia Reinaud\*

A slight conformation change in the calix[6]arene core capped by a poly-aza tripodal ligand induces a dipolar inversion of the second coordination sphere that controls anion binding to the Cu(II) funnel complexes.

813

## Templated crystal nucleation: mixed crystals of very different copper(II) N, N', N''-trimethyltriazacyclononane complexes

Jonathan W. Steed,\* Andrés E. Goeta, Janusz Lipkowski, Dariusz Swierczynski, Vicky Panteleon and Sheetal Handa

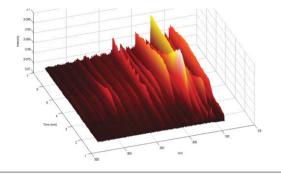
Crystals of a green mononuclear copper(II) N,N',N''-trimethyltriazacyclononane complex template the formation of a daughter phase of an olive-yellow ionic binuclear complex, which is in equilibrium with the monomer.

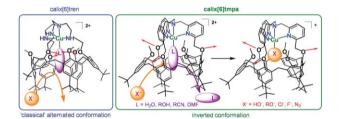
#### 816

## Poly{guanidinium} salts: application in the preparation of a coordinatively saturated aluminium cation

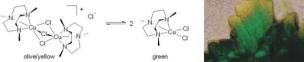
Pedro J. Aragón Sáez, Sarah H. Oakley, Martyn P. Coles\* and Peter B. Hitchcock

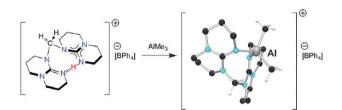
Bis(guanidine)  $H_2C{hpp}_2$  was converted to the mono- and dicationic guanidinium salts; reaction of the former with AlMe<sub>3</sub> directly generated a cationic main group compound supported by a chelating ligand set.











## flavours & fragrances 2007

24<sup>th</sup>-26<sup>th</sup> September

## Imperial College

Following the successful format of the previous meetings, the programme of the 2007 RSC-SCI 'flavours & fragrances' conference, organised by Philip Kraft and Karl A. D. Swift, will include these confirmed speakers: Angela Bassoli (University Milano), Constanze Brocke (Symrise), Robin A. Clery (Quest), Johannes le Coutre (Nestlé), Elisabet Duñach (University Nice), Céline Ellena (Charabot), Mark Erman (Millennium), Charles Fehr (Firmenich), Felix Flachsmann (Givaudan), Helmut Hügel (RMIT Melbourne), Thomas Hummel (University Dresden), Daniel Joulain (Robertet), Philip Kraft (Givaudan), Dietmar Krautwurst (Dt. Inst. Ernährungsf.), Christophe Laudamiel (IFF), Hiroyuki Matsuda (Takasago), Frank Ott (Symrise), Johannes Panten (Symrise), Caroline Plessis (Mane), Peter Schieberle (TU Munich), Geza Schoen (Escentric Molecules), Charles S. Sell (Quest), Roger Snowden (Firmenich), Reinhold Tacke (University Würzburg) and Chris Winkel (Quest).

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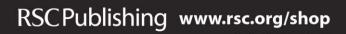
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#### 819

#### Controlled diastereoselectivity at the alkene-geometry through selective encapsulation: *E-Z* photoisomerization of oxazolidinone-functionalized enecarbamates within hydrophobic *nano*-cavities

Hideaki Saito, J. Sivaguru, Steffen Jockusch, Joanne Dyer, Yoshihisa Inoue, Waldemar Adam and Nicholas J. Turro\*

The selective encapsulation of the encarbamates and the following photoisomerization process are both diastereoselectively controlled by  $\gamma$ -CD.



#### Regioselective [2 + 2]-photocycloaddition reactions of chiral tetronates—influence of temperature, pressure, and reaction medium

Martin Fleck, Cheng Yang, Takehiko Wada, Yoshihisa Inoue\* and Thorsten Bach\*

Efficient double bond differentiation in a 1,3-divinyl-2cyclopentyl tetronate was observed if its intramolecular photocycloaddition was conducted in the presence of a  $\gamma$ -cyclodextrin.

#### 825

## Cross-coupling of alkyl halides with Grignard reagents using nickel and palladium complexes bearing $\eta^3$ -allyl ligand as catalysts

Jun Terao,\* Yoshitaka Naitoh, Hitoshi Kuniyasu and Nobuaki Kambe\*

The cross-coupling of Grignard reagents with alkyl bromides and tosylates has been achieved by the use of  $\eta^3$ -allylnickel and  $\eta^3$ -allylpalladium complexes as catalysts.

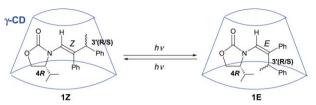


The first topologically controlled synthesis of doubly bridged  $\beta$ -cyclodextrin dimers

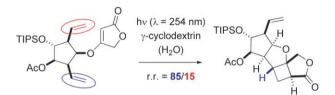
De-Qi Yuan,\* Kazutaka Koga, Isao Kouno, Toshihiro Fujioka, Makoto Fukudome and Kahee Fujita\*

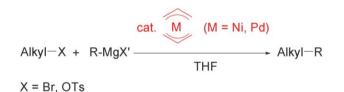
The occlusive dimer was selectively formed while the aversive dimer was not detected.

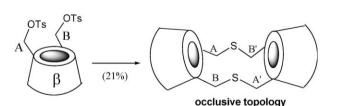












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**1987**: the Nobel prize in chemistry is awarded to Charles Pedersen, Jean-Marie Lehn and Donald Cram in recognition of their pioneering work in Supramolecular Chemistry.

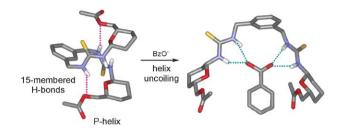
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#### 831



#### Promoting helicity in carbohydrate-containing foldamers through long-range hydrogen bonds

David Rodríguez-Lucena, Juan M. Benito, Carmen Ortiz Mellet\* and José M. García Fernández\*

A  $C_2$ -symmetric bis(thiourea) system allowing the propensity of carbohydrates to induce helical secondary structures through long-range hydrogen bonds to be probed has been designed. The existence of definite folding patterns strongly influences the recognition capabilities towards benzoate anion.

#### 834

## New insights into the initial steps of the formation of SBA-15 materials: an *in situ* small angle neutron scattering investigation

Marianne Impéror-Clerc,\* Isabelle Grillo, Andrei Y. Khodakov, Dominique Durand and Vladimir L. Zholobenko

Time-resolved *in situ* SANS investigations have provided direct experimental evidence for the three initial steps in the formation of the SBA-15 mesoporous material.

#### 837

## Synthesis and crystal structure of $Na_{1-x}Ge_{3+z}$ : a novel zeolite-like framework phase in the Na–Ge system

Matt Beekman, James A. Kaduk, Qing Huang, Winnie Wong-Ng, Zhi Yang, Dongli Wang and George S. Nolas\*

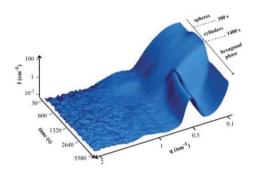
A novel crystalline binary phase is reported in the Na–Ge system, with an entirely new, zeolite-like crystal structure solved and refined by the combination of synchrotron X-ray and neutron powder diffraction techniques.

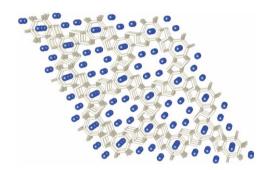
#### 840

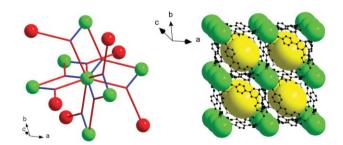
## Twelve-connected porous metal–organic frameworks with high $H_2$ adsorption

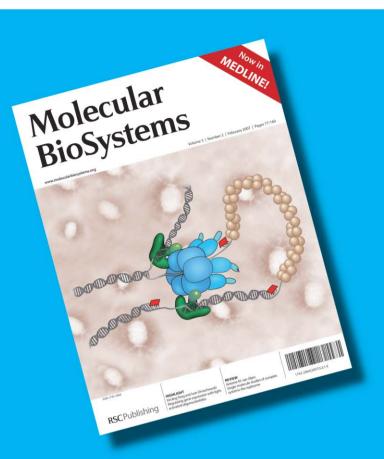
Junhua Jia, Xiang Lin, Claire Wilson, Alexander J. Blake, Neil R. Champness,\* Peter Hubberstey,\* Gavin Walker, Edmund J. Cussen and Martin Schröder\*

The twelve-connected metal–organic frameworks  $\{[Ni_3(OH)(L)_3]\cdot n(solv)\}_{\infty} 1$  and  $\{[Fe_3(O)(L)_3]\cdot n(solv)\}_{\infty} 2$   $[LH_2 = pyridine-3,5-bis(phenyl-4-carboxylic acid)]$  can be desolvated to form porous materials that show adsorption of H<sub>2</sub> up to 4.15 wt% at 77 K.









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Veronica Åberg, Erik Fällman, Ove Axner, Bernt Eric Uhlin, Scott J. Hultgrend and Fredrik Almqvist

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#### 843

#### D-Glucosamine-based supramolecular hydrogels to improve wound healing

Zhimou Yang, Gaolin Liang, Manlung Ma, A. Sunny Abbah, W. William Lu and Bing Xu\*

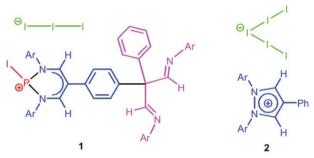
A simple supramolecular hydrogel based on D-glucosamine, a naturally occurring aminosaccharide, promotes wound healing.



#### β-Diiminato ligand (L) transformations in reactions of KL with PI<sub>3</sub> and $I_2 [L = {N(C_6H_3Pr_2^2-2,6)C(H)}_2CPh]$

Peter B. Hitchcock, Michael F. Lappert,\* Gang Li and Andrey V. Protchenko

Potassium  $\beta$ -dialdiminate K[{N(C<sub>6</sub>H<sub>3</sub>Pr<sup>i</sup><sub>2</sub>-2,6)C(H)}<sub>2</sub>CPh] with PI<sub>3</sub> or I<sub>2</sub> produced intermolecular *C*, *C*- or intramolecular N, N-coupled salts 1 or 2, respectively.



OH

Gel II

Gel II-treated wound site



#### Organocatalytic asymmetric 5-hydroxyisoxazolidine synthesis: A highly enantioselective route to β-amino acids

Ismail Ibrahem, Ramon Rios, Jan Vesely, Gui-Ling Zhao and Armando Córdova\*

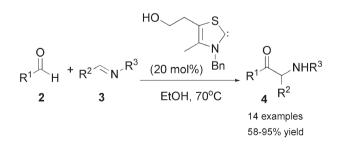
The highly chemo- and enantioselective organocatalytic tandem reaction between N-protected hydroxyl amines and  $\alpha,\beta$ -unsaturated aldehydes is presented. The reaction provides direct access to 5-hydroxyisoxazolidines, 5-isoxazolidinones and  $\beta$ -amino acids, which are furnished in high yields and with 90-99% ee.

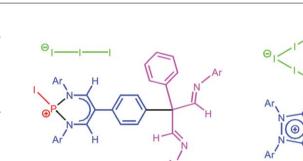
#### 852

#### Thiazolium-derived N-heterocyclic carbene-catalyzed cross-coupling of aldehydes with unactivated imines

Gong-Qiang Li, Li-Xin Dai and Shu-Li You\*

Direct cross-coupling of aromatic aldehvdes or benzoins with unactivated imines catalyzed by an N-heterocyclic carbene (NHC) has been realized, affording  $\alpha$ -amino ketones smoothly in moderate to excellent yields.



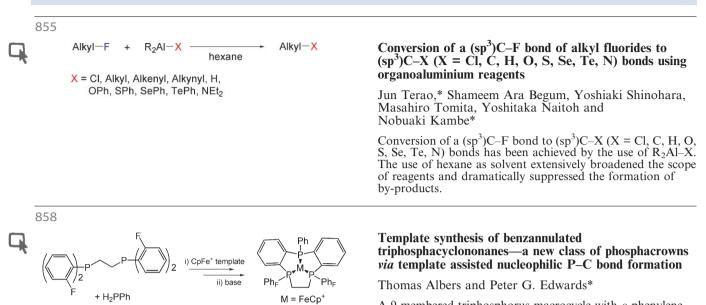


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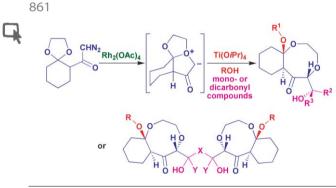
Untreated wound site



(20 mol%



A 9-membered triphosphorus macrocycle with o-phenylene backbone functions has been stereoselectively prepared on a CpFe<sup>+</sup> template by nucleophilic attack of a coordinated phosphide on a coordinated o-fluorophenylbiphosphine.



Multicomponent reactions involving tricyclooxonium ylide intermediate: diastereoselective synthesis of mono- and bisalkoxyoctahydro-1,4-benzodioxocin-6(5*H*)-one frameworks

Sengodagounder Muthusamy,\* Janagiraman Krishnamurthi and Eringathodi Suresh

A highly diastereoselective tandem ring-enlargement and aldol condensation process with alcohols, carbonyl compounds in the presence of titanium(IV) isopropoxide is described to afford benzodioxocinone ring systems.

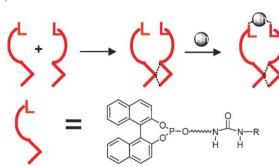
### UREAphos: supramolecular bidentate ligands for asymmetric hydrogenation

Albertus J. Sandee, Alida M. van der Burg and Joost N. H. Reek\*

Urea-based supramolecular bidentate phosphite ligands are applied in the rhodium catalysed asymmetric hydrogenation of various alkenes, providing moderate to high enantioselectivities.



q



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## Highly stereoselective chlorination of β-substituted cyclic alcohols using PPh<sub>3</sub>–NCS: factors that control the stereoselectivity

E. A. Jaseer, Ajay B. Naidu, Sreehari S. Kumar, R. Koteshwar Rao, Krishna G. Thakur and G. Sekar\*

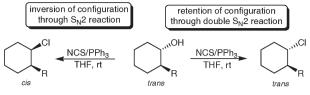
A variety of *trans*- $\beta$ -substituted cyclic alcohols were stereoselectively chlorinated to either the corresponding *cis*-chloride (inversion of configuration) or *trans*-chloride (retention of configuration) with good to excellent yields.

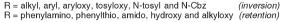


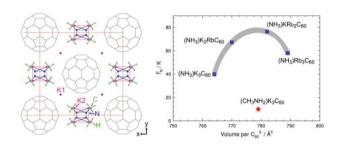
## Direct observation of magnetic ordering in the $(CH_3NH_2)K_3C_{60}$ fulleride

Yasuhiro Takabayashi, Alexey Yu. Ganin, Matthew J. Rosseinsky\* and Kosmas Prassides\*

The methylaminated potassium fulleride  $(CH_3NH_2)K_3C_{60}$  exhibits long range antiferromagnetic order with an unusually low Néel temperature  $T_N$ .







873

#### Mn<sub>2</sub> bis(pentalene): a mixed-spin bimetallic with two extremes of bonding within the same molecule

Gabor Balazs, F. Geoffrey N. Cloke,\* Andrew Harrison, Peter B. Hitchcock, Jennifer Green\* and Owen T. Summerscales

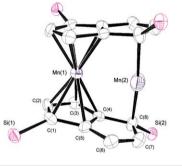
Structural, magnetic and theoretical studies show that the bimetallic pentalene complex,  $Mn_2(C_8H_4^{1,4-Si'Pr_3})_2$ , contains both high and low spin Mn(II) in two very different sites.

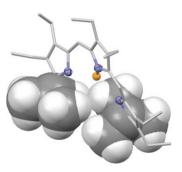
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#### **Free-base tripyrrins**

Martin Bröring,\* Serguei Prikhodovski and Esther Cónsul Tejero

Sensitive tripyrrins have been prepared as the free-base ligands by the cyanide promoted demetallation of nickel(II) chelates and by steric shielding of the reactive  $\alpha$ -position, and have been characterized structurally and spectroscopically.





#### 878

Hao Li, Jian Wang, Timiyin E-Nunu, Liansuo Zu, Wei Jiang, Shaohua Wei and Wei Wang

One-pot approach to chiral chromenes *via* enantioselective organocatalytic domino oxa-Michael-aldol reaction

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