

# NJC

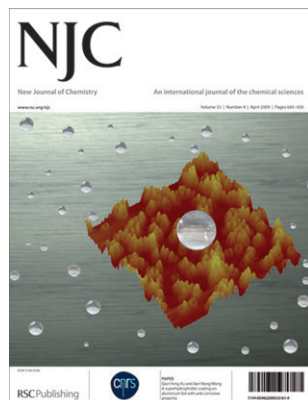
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## IN THIS ISSUE

ISSN 1144-0546 CODEN NJCHES 33(4) 685-920 (2009)



### Cover

See Qian Feng Xu and Jian Nong Wang, pp. 734-738.

Superhydrophobic (SH) coatings with high chemical stabilities and good anti-corrosive properties have been fabricated on Al foil. The SH properties can be maintained for water droplets with a wide range of pH values (from 2 to 12).

Image reproduced by permission of Qian Feng Xu and Jian Nong Wang, *New J. Chem.*, 2009, **33**, 734.



### Inside cover

See Robert Vianello and Zvonimir B. Maksić, pp. 739-748.

Polyenes dressed by cyano groups provide strong superacids, which upon deprotonation give stable, weakly coordinated anions. Image reproduced by permission of Robert Vianello and Zvonimir B. Maksić from *New J. Chem.*, 2009, **33**, 739.

## CHEMICAL SCIENCE

### C25

Drawing together research highlights and news from all RSC publications, *Chemical Science* provides a 'snapshot' of the latest developments across the chemical sciences, showcasing newsworthy articles and significant scientific advances.

## Chemical Science

April 2009/Volume 6/Issue 4

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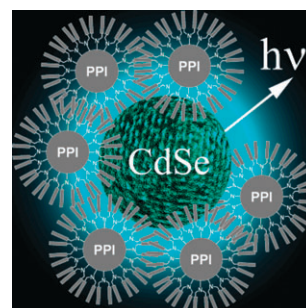
## LETTER

### 703

#### Water-soluble CdSe nanoparticles stabilised by dense-shell glycodendrimers

Amir Fahmi,\* Torsten Pietsch, Dietmar Appelhans,\* Nabil Gindy and Brigitte Voit

Uniform CdSe nanoparticles stabilized with glycodendrimers were synthesized in aqueous solution at room temperature. The optical and structural properties of the nanoparticles were studied by means of UV-vis and fluorescence spectroscopy, as well as TEM.



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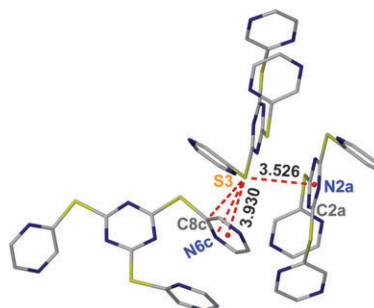
## LETTERS

707

**Intermolecular S... $\pi$  interactions in crystalline sulfanyl-triazine derivatives**

Chong-Qing Wan, Jie Han and Thomas C. W. Mak\*

Three types of intermolecular S... $\pi$  interaction geometry are identified in the crystal structures of four sulfanyl-triazine derivatives bearing pendant heterocyclic rings, in which the triazinyl, pyrazinyl, pyrimidinyl and pyridyl rings are found to exhibit different affinities for the divalent S atom.

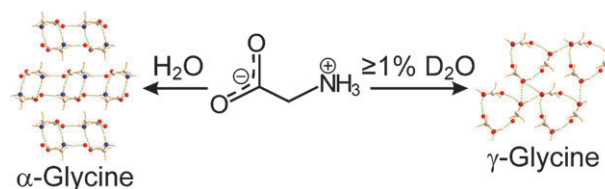


713

**The effect of deuteration on polymorphic outcome in the crystallization of glycine from aqueous solution**

Colan E. Hughes and Kenneth D. M. Harris\*

Formation of the  $\gamma$  polymorph of glycine in crystallization from aqueous solution is promoted by deuteration of the system (even as low as 1% deuteration) or by the use of extended crystallization times.

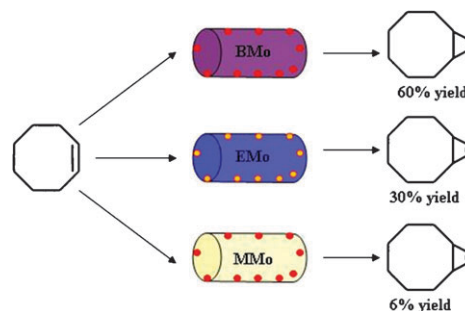


717

**The enhanced catalytic activity and stability of oxodiperoxomolybdenum-modified mesoporous organosilicas in selective epoxidation reactions**

Sankaranarayana pillai Shylesh, Mingjun Jia, Andreas Seifert, Sridhar Adappa, S. Ernst and Werner R. Thiel\*

Molybdenum complexes anchored over phenylene-bridged mesoporous organosilicas show a 10 times higher catalytic activity and high stability in liquid phase epoxidations with aqueous oxidants than the corresponding MCM-41 catalysts.

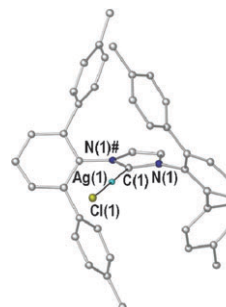


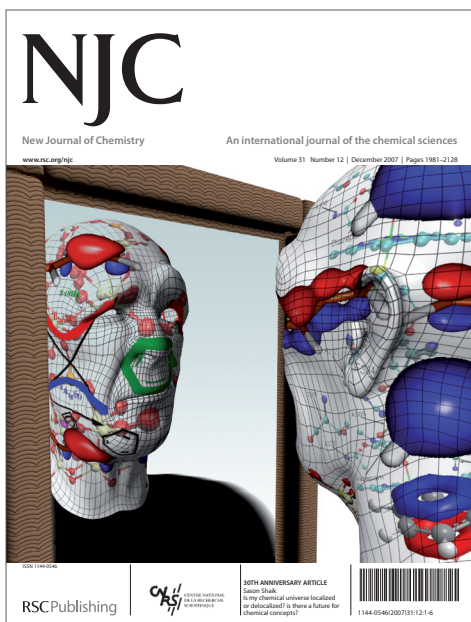
720

**Preparation of a *super* bulky silver *N*-heterocyclic carbene complex**

Sean G. Alexander, Marcus L. Cole\* and Jonathan C. Morris

The first metal complex containing a *super* bulky 1,3-bis(2,6-terphenyl)imidazol-2-ylidene *N*-heterocyclic carbene (NHC) has been prepared and fully characterised. This NHC has a cone angle that rivals those of bulky tertiary phosphines, thereby greatly extending the steric range of NHCs.





# Drawing disciplines together



## Introducing Professor Michael Scott

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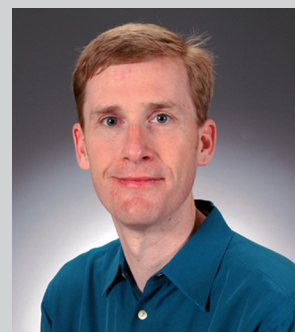
Michael is an associate professor in the Department of Chemistry and the director for the Center for Catalysis at the University of Florida in Gainesville, Florida. He is a fellow of both the Alfred P. Sloan Foundation and the Royal Society of Chemistry. His research interests focus on the design of ligands and metal complexes for the selective recognition and sequestration of cations and anions and for biomimetic catalysis.

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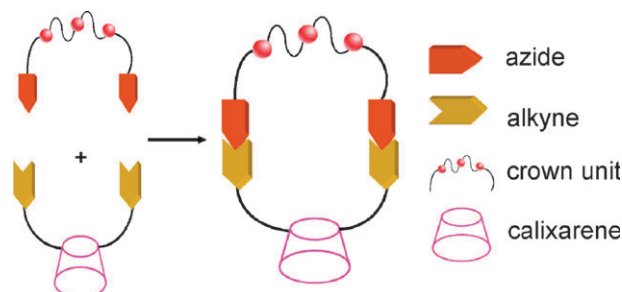
## LETTERS

725

**Synthesis of calix[4]crowns containing soft and hard ion binding sites *via* click chemistry**

Junyan Zhan, Demei Tian and Haibing Li\*

A series of triazole-modified calix[4]crowns containing hard and soft ion binding sites were synthesized in good yields *via* click chemistry. The hard and soft ion binding sites may selectively complex alkali metal or transition metal cations.

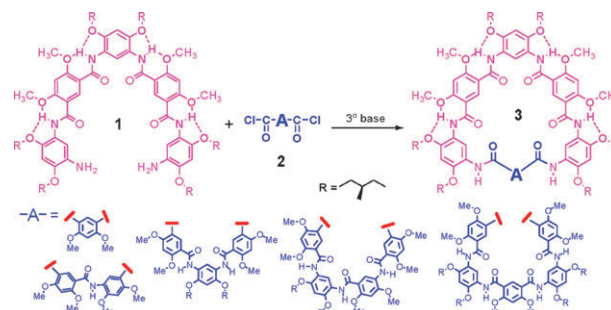


729

**Aromatic oligoamide macrocycles from the bimolecular coupling of folded oligomeric precursors**

Liuqing Yang, Lijian Zhong, Kazuhiro Yamato, Xiaoheng Zhang, Wen Feng, Pengchi Deng, Lihua Yuan,\* Xiao Cheng Zeng\* and Bing Gong\*

Aromatic oligoamide macrocycles consisting of six to ten *meta*-linked residues were prepared based on biomolecular coupling/cyclization, suggesting that H-bond-enforced folding of the oligomeric precursors may also promote the formation of macrocycles with large ring strains.



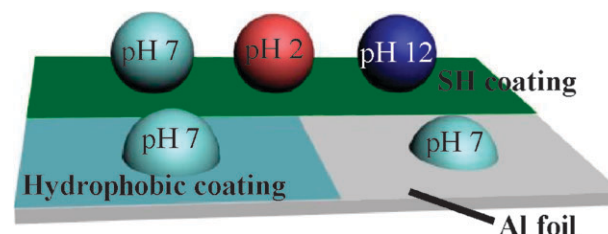
## PAPERS

734

**A superhydrophobic coating on aluminium foil with an anti-corrosive property**

Qian Feng Xu and Jian Nong Wang\*

Superhydrophobic (SH) coatings with a good chemical stability and an anti-corrosive property have been fabricated on an Al foil, and the SH property can be maintained for water droplets with a wide range of pH values (from 2–12).

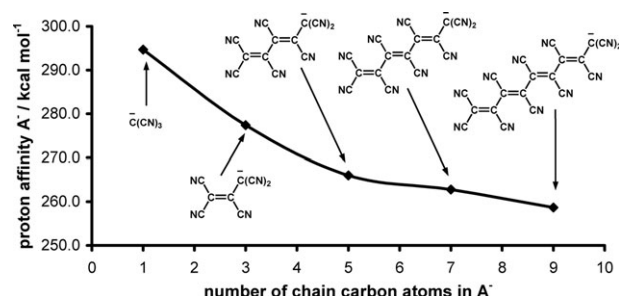


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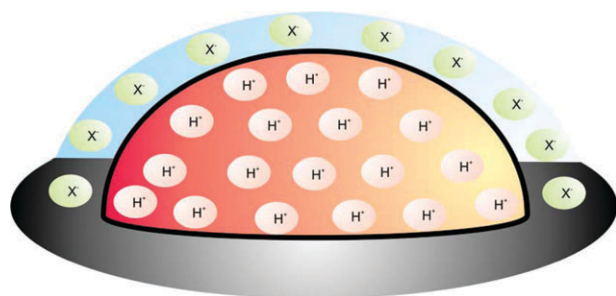
**The engineering of powerful non-ionic superacids *in silico*—a DFT-B3LYP study of open chain polycyanopolyenes**

Robert Vianello\* and Zvonimir B. Maksić

Polycyano-substituted polyenes, some of which have already been synthesized, exhibit superacidity in the gas phase that comes close to the hyperacidity threshold of 245 kcal mol<sup>-1</sup>.



749

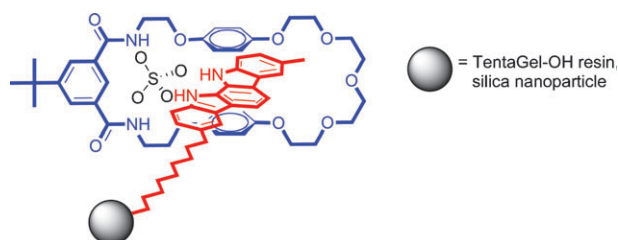


### Electrogenerated chemiluminescence at droplet-modified electrodes: towards biphasic $pK_a$ measurement *via* proton-coupled electron transfer at liquid|liquid interfaces

Carlos Lledo-Fernández, Imren Hatay, Michael J. Ball, Gillian M. Greenway and Jay Wadhawan\*

The degree of protonation at an aqueous|oil interface can be determined *via* the electrochemically-mediated generation of photons.

760

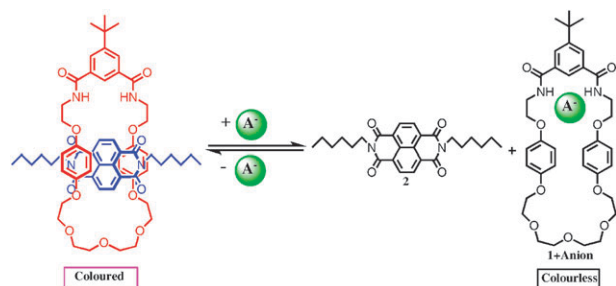


### Anion templated assembly of an indolocarbazole containing pseudorotaxane on beads and silica nanoparticles

Liyun Zhao, Kathleen M. Mullen, Michał J. Chmielewski, Asha Brown, Nick Bampos, Paul D. Beer\* and Jason J. Davis\*

The surface covalent attachment of fluorescent axles of indolocarbazole to polystyrene beads and silica nanoparticles enables anion templation to be exploited in the formation of pseudorotaxane assemblies.

769

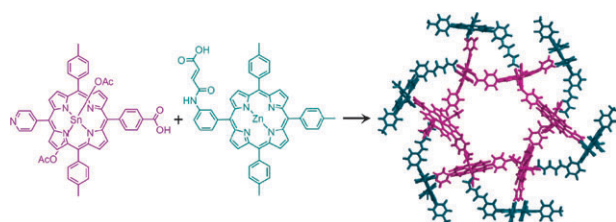


### Anion induced displacement studies in naphthalene diimide containing interpenetrated and interlocked structures

Kathleen M. Mullen, Jason J. Davis and Paul D. Beer\*

A pseudorotaxane assembly is shown to be capable of colorimetrically sensing anions *via* displacement of an electron deficient naphthalene diimide thread upon anion binding at the isophthalamide recognition site of the macrocycle.

777



### Cyclic oligomers based on complementary Zn(II) and Sn(IV)-porphyrins

Gerald A. Metselaar, Pablo Ballester and Javier de Mendoza\*

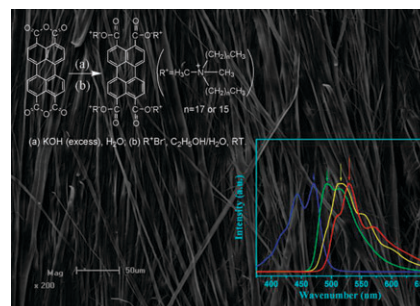
The aggregation of a 1 : 1 mixture of complementary Sn(IV)- and Zn(II)-porphyrins into exclusively cyclic decameric and dodecameric assemblies has been investigated by  $^1\text{H}$  NMR, GPC, DOSY and MALDI-TOF spectrometry.

784

### Ionic self-assembled derivatives of perylenetetracarboxylic dianhydride: facile synthesis, morphology and structures

Guihua Fu, Muli Wang, Yongliang Wang, Nan Xia, Xinjun Zhang, Miao Yang, Ping Zheng, Wei Wang\* and Christian Burger\*

A facile ion self-assembly method was used to synthesize perylene derivatives in water in which the perylene cores arrange into lamellar structures with different long periods, which control their fluorescence behavior.

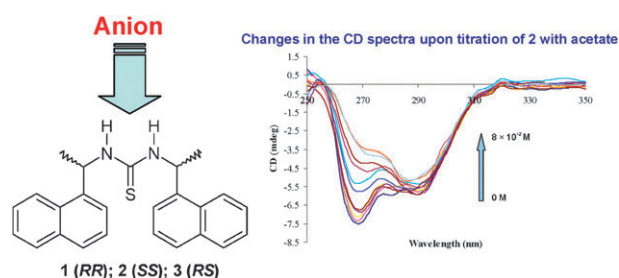


793

### Crystallographic, $^1\text{H}$ NMR and CD studies of sterically strained thiourea anion receptors possessing two stereogenic centres

Haslin Dato Paduka Ali, Susan J. Quinn, Thomas McCabe, Paul E. Kruger\* and Thorfinnur Gunnlaugsson\*

The synthesis of three charge neutral chiral receptors, **1** and **2** and the *meso* compound **3**, is described. Their ability to recognise anions using  $^1\text{H}$  NMR, UV-Vis, fluorescence and CD spectroscopies is described.

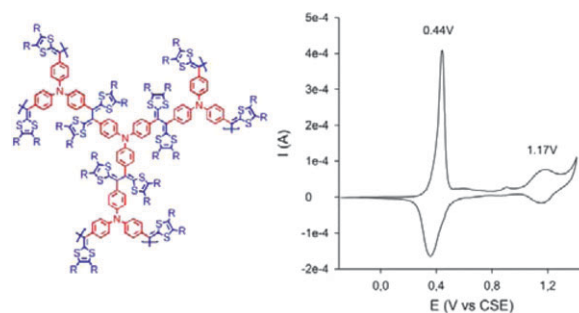


801

### Electropolymerization of triphenylamine–dithiafulvene hybrid extended pi-conjugated systems

Nicolas Cocherel, Philippe Leriche,\* Emilie Ripaud, Nuria Gallego-Planas, Pierre Frère and Jean Roncali

Extended hybrid conjugated systems based on a triphenylamine core with 1, 2 and 3 peripheral dithiafulvenyl units are presented. Their electrooxidation leads to polymers. The structure and properties of polymeric species are presented.

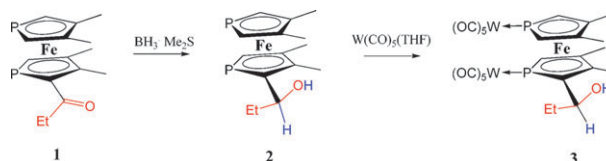


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### Unusual diastereoselective reduction of 2-propionyl-3,3',4,4'-tetramethyl-1,1'-diphosphaferrocene to the corresponding alcohol by $\text{BH}_3 \cdot \text{Me}_2\text{S}$ . X-Ray diffraction and DFT study

Bartosz Mucha, Arkadiusz Kłys, Janusz Zakrzewski,\* Anna Makal and Krzysztof Woźniak

2-Propionyl-3,3',4,4'-tetramethyl-1,1'-diphosphaferrocene **1** is stereoselectively reduced to **2** with  $\text{BH}_3 \cdot \text{Me}_2\text{S}$ . The configuration (*S,S*)/(*R,R*) of **2** has been determined from X-ray diffraction study of **3**.





## Faced with questions?

Can I search by structure to find articles?

Are there any related articles on this topic?

What groups and relationships are there for this compound?

Is there any Patent information?

Can I download files of these structures?

What's the definition of that term?

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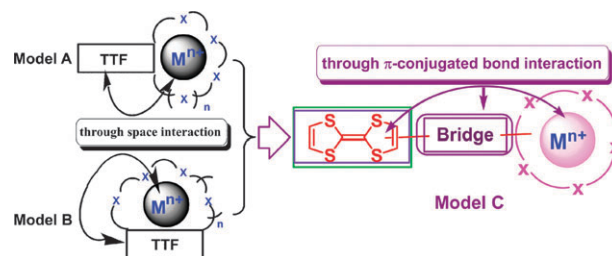
## PAPERS

813

### Tetrathiafulvalene derivatives bearing a crown ether with intramolecular charge transfer properties: synthesis and cation binding studies

Yao-Peng Zhao, Xiao-Jun Wang, Jing-Jing Wang, Gang Si, Yan Liu, Chen-Ho Tung and Li-Zhu Wu\*

In contrast to models **A** and **B**, the TTF unit in model **C** can communicate with metal cations through a triple bond, leading to remarkable changes in their UV-vis absorption,  $^1\text{H}$  NMR spectra and electrochemical properties.

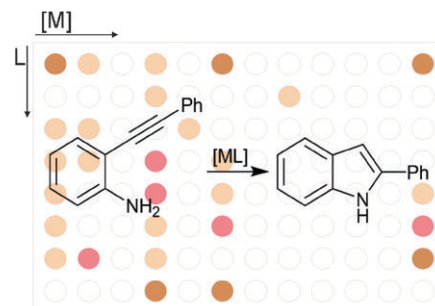


818

### Application of UV-Vis spectroscopy to high throughput screening of hydroamination catalysts

Danielle F. Kennedy, Barbara A. Messerle\* and Sarah L. Rumble

A detailed investigation of complexes identified as potential for hydroamination through high throughput screening was performed, one highly active catalyst system  $[\text{Rh}(\text{CO})\text{Cl}(\text{mesBIAN})]\text{-AgBF}_4$  has been developed and the formation of the unusual product *N*-(2-methylvinyl)-2-phenylindole was observed.

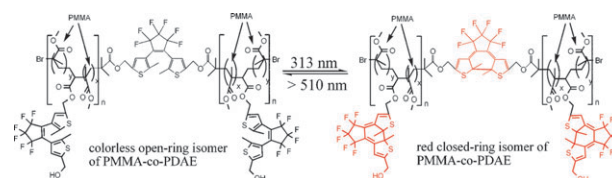


825

### Synthesis and properties of novel photochromic poly(methyl methacrylate-co-diarylethene)s

Liang Shen,\* Cha Ma, Shouzhi Pu, Chuanjie Cheng, Jingkun Xu,\* Long Li and Changqing Fu

Novel poly(methyl methacrylate)s substituted with photochromic diarylethenes as the main backbone and/or pendant functional groups were prepared by atom transfer radical polymerization (ATRP). The resulting copolymers exhibit good photochromic and fluorescence properties, easy film-forming and outstanding thermal performance.

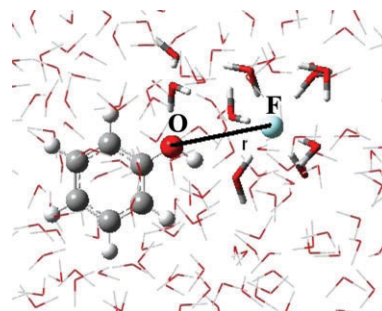


831

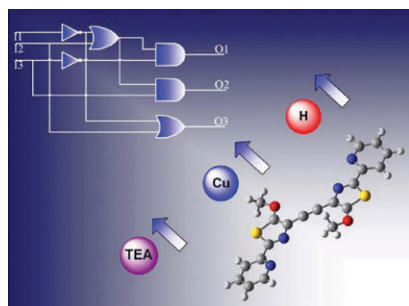
### An explicit solvent quantum chemistry study on the water environment influence on the interactions of fluoride with phenol

Piotr Cysewski,\* Beata Szeffler, Halina Szatyłowicz and Tadeusz Marek Krygowski

Aromaticity and H-bonding in fluorine-phenol systems are significantly affected by spatial fluctuations of water molecules in the first hydration layer.



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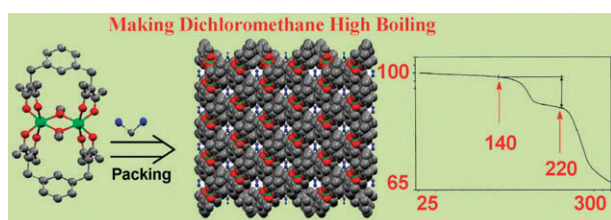


### Logic circuits constructed with an ion-sensitive fluorescent molecule 1,2-di[5-methoxy-2-(2-pyridyl)thiazoyl]ethyne

Chun-Hu Xu, Wei Sun, Yao-Rong Zheng, Chen-Jie Fang, Can Zhou, Jing-Yi Jin and Chun-Hua Yan\*

Based on the combinatorial and sequential effect of different recognition processes on a fluorescent switch DMPTE, 2-input logic gates and 3-digit keypad lock are constructed at molecular level.

847

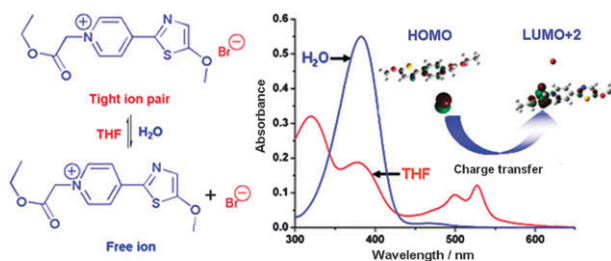


### Methoxy-bridged diiron(III) complex of *m*-xylylenebis(acetylacetonate) showing remarkable thermal stability for encapsulated dichloromethane

Supriya Dutta,\* Papu Biswas, Sujit K. Dutta and Kamalaksha Nag\*

The methoxy-bridged diiron(III) inclusion compound  $[\text{Fe}_2(m\text{-xba})_2(\mu\text{-OCH}_3)_2] \cdot \text{CH}_2\text{Cl}_2$  (**1**) derived from the title ligand shows remarkable thermal stability towards detraping of the solvent molecule, which takes place between 140 and 220 °C.

853

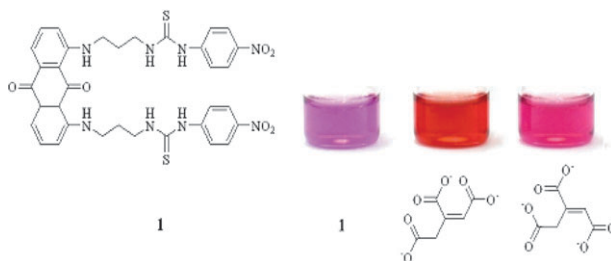


### Solvent-sensitive charge-transfer absorption behaviours and dual-emissive fluorescent properties of a thiazole-conjugated pyridinium complex

Zhan-Xian Li, Chun-Hu Xu, Wei Sun, Yan-Chun Bai, Chao Zhang, Chen-Jie Fang and Chun-Hua Yan\*

The solvent-sensitive charge transfer absorption in the visible region is tuned by the distance between the bromide anion and thiazolyl-pyridinium in our thiazole-conjugated pyridinium complex.

860



### Colorimetric anion chemosensors based on anthraquinone: naked-eye detection of isomeric dicarboxylate and tricarboxylate anions

Yi-Shan Lin, Guan-Min Tu, Chi-Yung Lin, Yen-Tzu Chang and Yao-Pin Yen\*

The new colorimetric sensor **1** showed distinct color changes when treated with *cis*-aconitate and *trans*-aconitate anions. Thus, it can act as an optical chemosensor for selective discrimination between *cis*-aconitate and *trans*-aconitate anions.

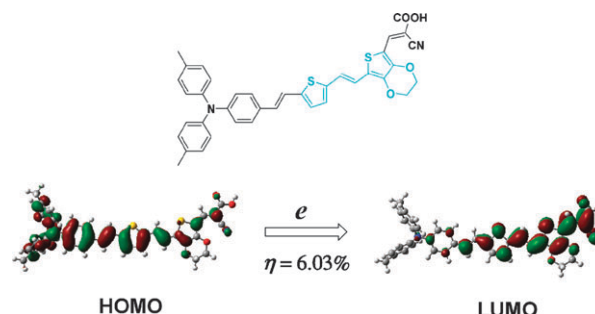
## PAPERS

868

**Molecular design of triarylamine-based organic dyes for efficient dye-sensitized solar cells**

Gang Li, Ke-Jian Jiang,\* Peng Bao, Ying-Feng Li, Shao-Lu Li and Lian-Ming Yang\*

Three donor-( $\pi$ -spacer)-acceptor dyes, coded as **DS-3**, **DS-5** and **DS-6**, were synthesized and applied in nanocrystalline TiO<sub>2</sub> based solar cells, among which the dye **DS-5** yielded a highest solar energy-to-electricity conversion efficiencies of 6.03% under AM 1.5 sunlight, reaching 77% with respect to that of an N3-based device fabricated and measured under similar conditions.

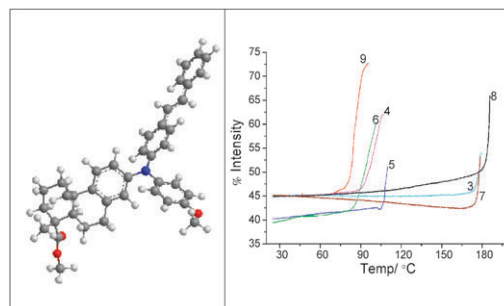


877

**Synthesis, spectroscopy, photophysics and thermal behaviour of stilbene-based triarylamines with dehydroabietic acid methyl ester moieties**

Bárbara Gigante,\* M. Alexandra Esteves, N. Pires, Matthew L. Davies, Peter Douglas, Sofia M. Fonseca, Hugh D. Burrows,\* Ricardo A. E. Castro, João Pina and João Seixas de Melo

Bulky dehydroabietic acid groups increase solubility and hinder crystallisation in novel triarylamines for molecular electronics applications.

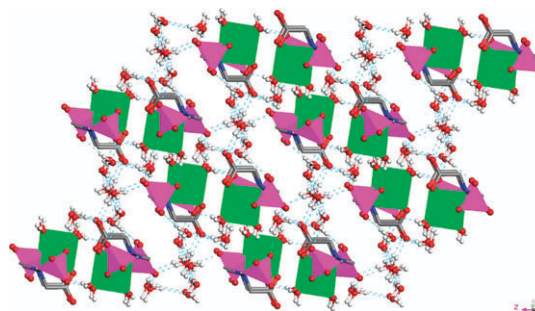


886

**Solvothermal syntheses, characterizations and properties of three transition metal (Ni(II), Co(II)) imino-carboxylate-diphosphonates**

Kuirong Ma, Jianing Xu, Lirong Zhang, Jing Shi, Daojun Zhang, Yulan Zhu, Yong Fan\* and Tianyong Song\*

Three compounds have been isolated from solvothermal reactions of a transition metal(II) (Ni(II), Co(II)) with *N,N*-bis(phosphonomethyl)aminoacetic acid, namely [NH<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>NH<sub>3</sub>](NH<sub>4</sub>)<sub>4</sub>[Ni(L)(H<sub>2</sub>O)]<sub>2</sub>, (H<sub>3</sub>O)<sub>3</sub>[Ni(L)(H<sub>2</sub>O)<sub>2</sub>] · 4H<sub>2</sub>O and (H<sub>3</sub>O)[Co(H<sub>2</sub>L)(H<sub>2</sub>O)<sub>2</sub>] · 2H<sub>2</sub>O.

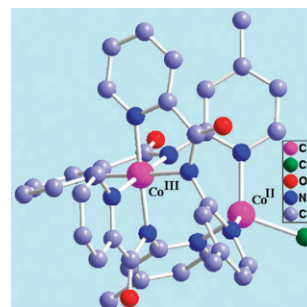


893

**Six-coordinate Co<sup>III</sup> and four-coordinate M<sup>II</sup> (M = Co, Zn) mixed-valence dimers supported by a deprotonated pyridine amide ligand: magnetism of a Co<sup>III</sup>Co<sup>II</sup> complex and C–H...O/Cl/Br interactions**

Wilson Jacob, Haritosh Mishra, Sharmila Pandey, Francesc Lloret and Rabindranath Mukherjee\*

Binuclear Co<sup>III</sup>Co<sup>II</sup> and Co<sup>III</sup>Zn<sup>II</sup> complexes have been synthesized using a pyridine amide ligand. Non-covalent interactions lead to the formation of supramolecular architectures.





# 13th International Symposium on Novel Aromatic Compounds July 19 – 24, 2009 Luxembourg

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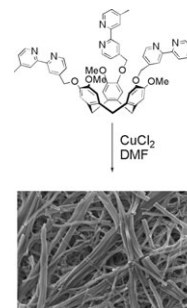
## PAPERS

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### Metallo-gels and organo-gels with tripodal cyclotrimeratrylene-type and 1,3,5-substituted benzene-type ligands

Aleema Westcott, Christopher J. Sumby,  
Richard D. Walshaw and Michael J. Hardie\*

Tripodal ligands based on both cyclotrimeratrylene-type and 1,3,5-substituted benzene units have been shown to form organo-gels or metallo-gels with Cu(II) and/or Ag(I).

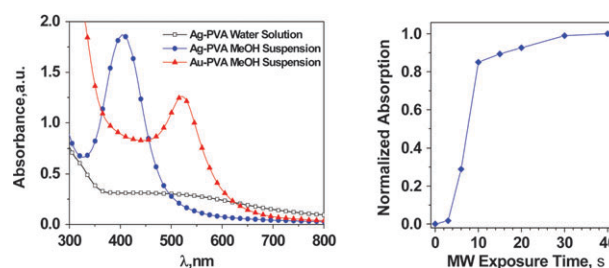


913

### Scalable heterogeneous synthesis of metallic nanoparticles and aggregates with polyvinyl alcohol

Rafael Abargues,\* Rachid Gradess, Josep Canet-Ferrer,  
Kamal Abderrafi, José Luís. Valdés and  
Juan Martínez-Pastor

A potentially scalable route to synthesize colloidal silver and gold nanoparticles based on the extremely fast microwave-assisted heterogeneous reduction of metal salts.



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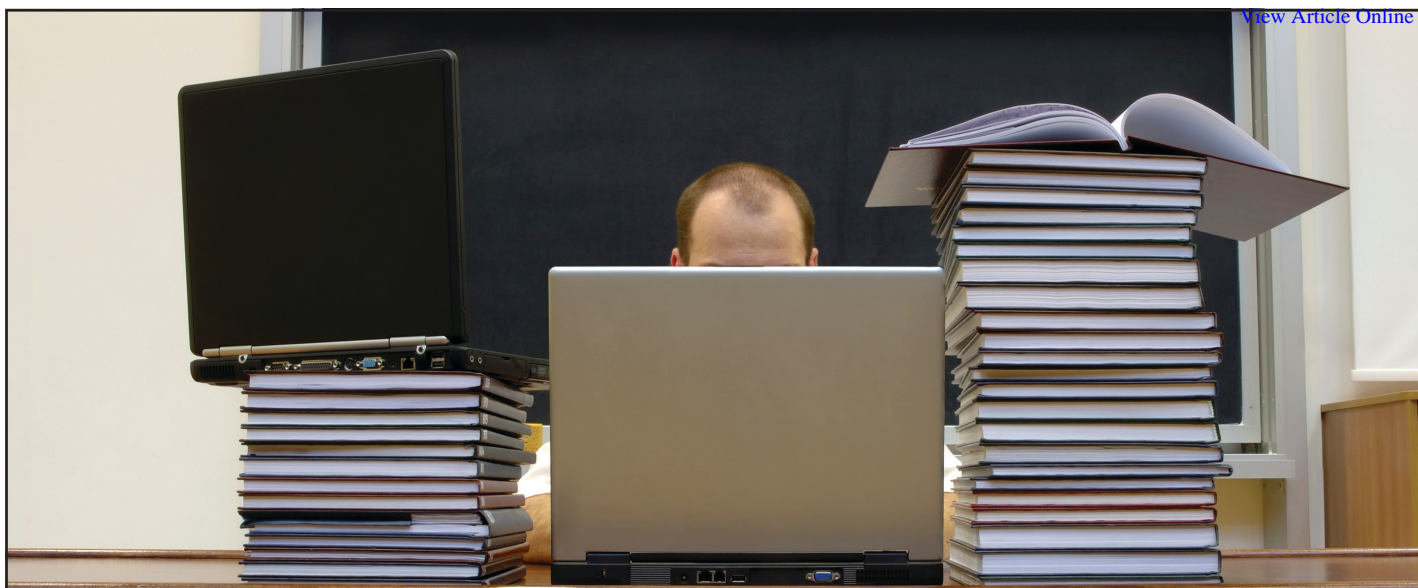
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# Chemical Science

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An artificial vascular system has been made using candy floss as a template

## Making capillaries with candy floss

Candy floss (also known as cotton candy) has been used by US scientists to create a web of microscopic tubes to mimic the capillary network that carries blood to human tissue.

Leon Bellan at Cornell University's Nanobiotechnology Center, Ithaca, and colleagues, mimicked the capillary network structure by sticking two sugar rods to a candy floss ball. They poured a molten polymer over the candy floss, left it to solidify, then dissolved the sugar, leaving a complex network of channels connecting two larger inlet and outlet channels. They then injected fluorescently labelled blood into the system and followed its progress using a video fluorescence microscope. They found that the blood flowed through as it would in a real system.

Bellan's method addresses a limitation in tissue engineering: how to make an artificial vascular system for the new tissue. Since blood can only diffuse a few hundred micrometres from a capillary, organs need these networks to deliver oxygen and nutrients to every



cell. His technique is cheaper and less time consuming than existing methods for making the networks, such as layer-by-layer 2D structure stacking or 3D printing, where templates for growing cells are built up.

Candy floss is an ideal template as it is cheap, non-toxic, water soluble

**Molten polymer was poured over candy floss to create a capillary network model**

**Reference**  
L M Bellan *et al*, *Soft Matter*, 2009, DOI: 10.1039/b819905a

and sticky. The stickiness allows junctions between the sugar rods and the candy floss to form easily. The only equipment required is a candy floss machine, which can be purchased for as little as \$40 (approximately £30), says Bellan.

'Finding inspiration from something in everyday life is very clever,' says Jeff Borenstein, director of the Biomedical Engineering Center at Draper Laboratory, Cambridge, US. 'It reminds me of how the pioneering tissue engineer, Jay Vacanti, was inspired to create 3D scaffolds for tissue engineering by observing the structure of seaweed while on a Cape Cod beach.'

Bellan says that potential applications for his method, aside from helping to grow organs in the laboratory, could include making self-healing polymers that can fracture and heal, over and over again in the same place. 'The simplicity and low cost of this new fabrication technique should render many applications of 3D microfluidic networks commercially viable,' he says. *James Hodge*

## In this issue

### Fighting MRSA with ionic liquids

Ionic liquids could be used to tackle hospital acquired infections

### Hydrogen storage steps up a gear

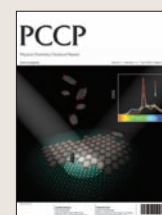
Improved storage takes us closer to hydrogen fuelled cars

### The growth of nanotoxicology

This month's Instant insight looks at analytical techniques used to assess nanotechnology's effects on health

### Creating a new world

Mukund Chorghade talks about his fascination with natural products and their role in India's future



A snapshot of the latest developments from across the chemical sciences

Aquatic organisms' feeding behaviour can be affected by ionic liquids

# Ionic liquids put zebra mussels off their food

Non-lethal doses of ionic liquids can have a significant effect on aquatic ecosystems, claim US scientists.

Ionic liquids are green alternatives to the volatile organic solvents that are released into the environment as a result of agriculture and manufacturing. But their solubility in water means that they can contaminate aquatic environments. Knowledge of their toxicity in these environments is limited, but even less is known about their non-lethal effects on aquatic organisms.

Now, David Costello and colleagues from the University of Notre Dame have studied how ionic liquids affect aquatic organisms' feeding rates as well as their survival.

The team looked at zebra mussels, which feed by filtration and can tolerate high doses of ionic liquids. They fed the mussels algae and exposed them to six different ionic liquids. They found that while changing the heterocyclic base of the



ionic liquid's cation had no effect, increasing the length of its alkyl chain increased toxicity and decreased the mussels' feeding rate. 'A reduction in algal consumption could allow

**Ionic liquids decreased the zebra mussel's feeding rate**

increases in algal populations that are resistant to ionic liquids,' says Costello.

'The work is a valuable contribution to the knowledge base that the scientific community is generating on potential harmful effects that will have to be considered if ionic liquids are to be used on a large scale,' says Johannes Ranke, an expert in the environmental risk assessment of ionic liquids, from the University of Bremen, Germany.

Costello says he hopes that within 5 to ten years, he will find more environmentally friendly solvents and that green solvents will be used more in general. He is currently investigating the nutrient cycle (the transfer of nutrients from one part of an ecosystem to another) in invasive species and contaminants and studying how contaminants affect nitrogen and phosphorus movement in the environment.

Ben Merison

## Reference

D M Costello, L M Brown and G A Lamberti, *Green Chem.*, 2009, DOI: 10.1039/b822347e

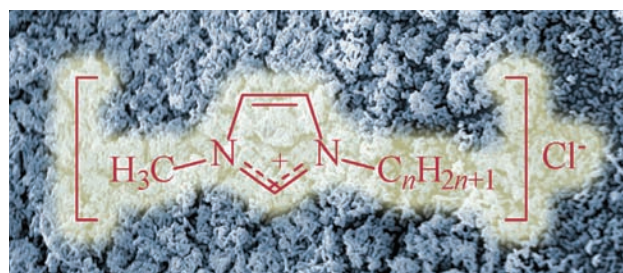
Ionic liquids could be used to tackle hospital acquired infections

# Fighting MRSA with ionic liquids

Hospital cleaners may one day use ionic liquids to clean wards. Brendan Gilmore and co-workers at the Queen's University, Belfast, UK, have shown that the compounds are effective antibacterial agents that can be used to break down microbial biofilms, a cause of hospital acquired infections such as MRSA.

Ionic liquids are low temperature molten salts formed from cations and anions. While ionic liquids must be tested for environmental toxicity before they can be used as safer alternatives to industrial solvents, Gilmore is using their toxicity for the benefit of human health. 'Altering the cation and anion pairing allows you to tune the toxicity,' says Gilmore.

Gilmore tested the effects of 1-alkyl-3-methylimidazolium chloride ionic liquids on the



bacterial biofilms of several pathogens including methicillin-resistant *Staphylococcus aureus* and *Escherichia coli*. The team found that antibiofilm potency increased with the length of the alkyl chain. Biofilms are bacterial communities that enclose themselves in a protective polymer. They are more resistant to antibiotics or other sterilisation methods than their free-swimming counterparts.

**1-Alkyl-3-methylimidazolium chloride ionic liquids were effective against *Staphylococcus aureus* and *E. coli* bacterial biofilms**

## Reference

L Carson *et al*, *Green Chem.*, 2009, DOI: 10.1039/b821842k

'Resistance to antimicrobials is an increasing global threat to public health,' says Jan Michiels, an expert in biofilms at the Catholic University of Leuven, Belgium. Ionic liquids could be applied to a surface already hosting a biofilm to help sterilise it, but Gilmore says he hopes that ionic liquids will be used to coat surfaces to prevent biofilms forming.

According to Gilmore, the advantages might not be limited to the health sector. Microbial biofilms can foul pipes in industrial machinery, and marine antifouling – a coating painted on to the hull of ships – could be another potential application.

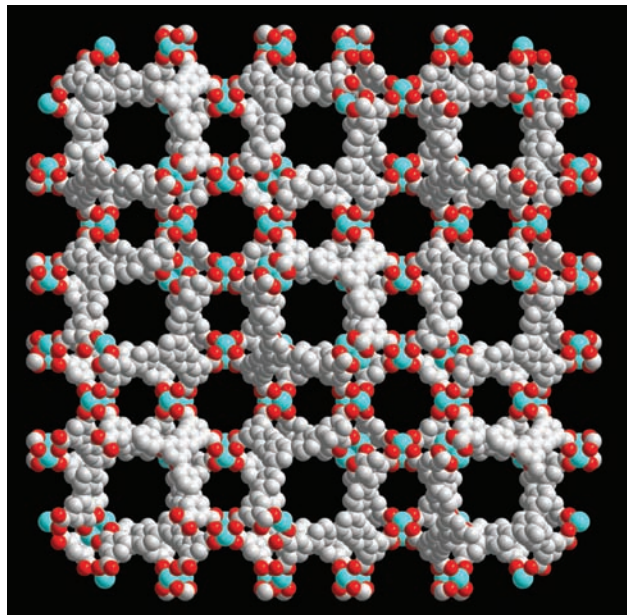
Gilmore's team is currently working on novel ionic liquids with improved antimicrobial and antibiofilm activities.

Russell Johnson



# Improved storage takes us closer to hydrogen fuelled cars

## Hydrogen storage steps up a gear

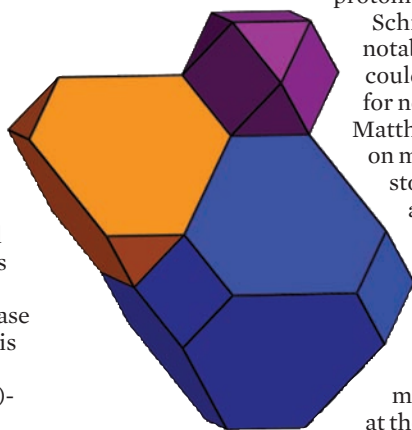


Martin Schröder at the University of Nottingham, UK, and colleagues, have made a porous solid for hydrogen storage with significantly increased hydrogen capacity. Ping Wang and colleagues at the Chinese Academy of Sciences, Shenyang, have discovered that hydrogen release from ammonia borane, a material with high hydrogen storage capability, can be accelerated by mechanical milling with magnesium hydride.

'Hydrogen represents an important potential energy source with zero carbon emissions at the point of use,' explains Schröder. The main barrier to its use as a vehicle fuel is the enormous storage volumes needed when it is carried in its molecular form, so how to increase capacity in any storage material is a key issue.

Schröder's solid is a copper(II)-based metal-organic polymer made up of three polyhedral cages that fit together to provide a hollow framework. The polymer can take up 10 wt% hydrogen at 77 bar and 77 Kelvin. 'This uptake is amongst the highest to date for this class of porous material and is a major contribution to

**The cage arrangement promotes hydrogen adsorption at high and low pressures maximising the obtainable storage capacity. An example of how the cages fit together is shown below**



### References

- 1 Y Yan *et al*, *Chem. Commun.*, 2009, 1025 (DOI: 10.1039/b900013e)
- 2 X Kang *et al*, *Phys. Chem. Chem. Phys.*, 2009, DOI: 10.1039/b820401b

the 2010 target of 6.5 wt% for a whole storage system set by the US Department of Energy,' says Schröder.

Another key requirement for hydrogen storage systems is fast hydrogen charge and discharge rates to meet consumer expectations for refuelling. Wang worked with ammonia borane, which has exceptional hydrogen storage capacity but a slow release rate. His milling technique speeds up hydrogen release. More than 8 wt% hydrogen can be released within four hours at 100°C, the lowest temperature obtained in any hydride system tested so far, says Wang. Low temperatures are important for controlling hydrogen release and spent fuel regeneration.

'Promoting hydrogen release by mechanically milling solid ammonia borane is not new,' explains Wang, 'but our studies show a completely different chemical activation mechanism that doesn't take place via alkali metal amidoboranes.' According to Wang, hydrogen is released through a destabilising solid phase reaction between the hydridic  $H^{-}$  in magnesium hydride and the protonic  $H^{+}$  in ammonia borane.

Schröder's structure is notable and its properties could help guide the search for new systems, says Matthew Rosseinsky, an expert on materials for energy storage and generation at the University of Liverpool, UK.

Schröder says that the next challenge is to increase the strength of hydrogen binding within his material to enable storage at the higher, more ambient temperatures needed for automobile-based applications. For Wang, understanding how magnesium hydride destabilises ammonia borane is key to designing systems with better capacity and kinetic performance. Janet Crombie

## News in brief

### This month in Chemical Technology

#### The medical power of attraction

Magnetic microbeads that cleanse blood of toxic pathogens could save thousands of lives

#### Nanowire forests repel liquids

An oil-repelling surface could prevent leaks from pipelines, say Chinese researchers

#### Crystal clear method for identifying powders

NMR and theory join forces to characterise drugs

#### Bone repair breakthrough

Thomas Webster and colleagues explain why today's bone implants are so much more than your grandparent's hip replacement

#### Molecular logic

A P de Silva discusses sensors, supramolecular chemistry and how Sri Lankan percussion can play a part in Irish music

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#### Biofilms under control

Microbe communities shun an artificial sweetener for a means to study biofilm growth

#### Stainless skin cancer diagnosis

An infrared imaging technique that can distinguish different types of skin cancer has been developed by scientists in France

#### Food for thought

How much does our diet have an effect on human memory and learning? Jeremy Spencer considers the case of the flavonoids

#### Inspired by nature

Ann Valentine talks about bioinorganic chemistry, titanium and avoiding rust

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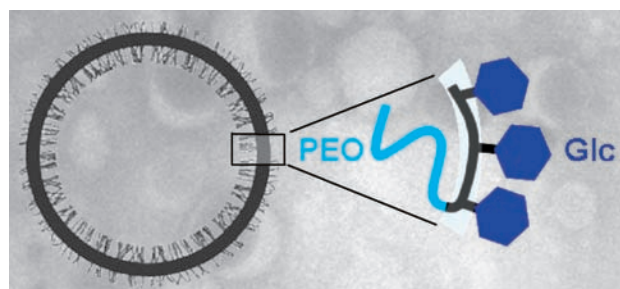
## Glycopolymers may be better living cell mimics and drug delivery vessels

# Polymer's coats multi-task in drug delivery

Polymer spheres with a sugar coating on the outside and plastic coating on the inside have been made by European scientists. This gives them dual functionality to target and deliver drugs.

Helmut Schlaad from the Max Planck Institute of Colloids and Interfaces, Potsdam, and colleagues from Germany and Switzerland made the spheres by dissolving glycosylated polybutadiene–poly(ethylene oxide) block copolymers in water. When dissolved, the copolymers spontaneously formed hollow colloids called vesicles with a glucose coating on the outside and a poly(ethylene oxide) coating on the inside.

The polymer vesicles could be used as living cell mimics or drug delivery vessels. Thanks to their adjustable properties – stability,



fluidity and dynamics – they could be better models for biomedical research than vesicles made from the phospholipids found in cells. Usually, the coatings on both sides of a vesicle's membrane are the same. As the outside and inside of Schlaad's vesicles are different, it may be possible to assign different tasks to each side. 'It would be very interesting to have vesicles with an asymmetric membrane for many

**The plastic and sugar coatings give the vesicles dual functionality to target and deliver drugs**

#### Reference

H Schlaad *et al*, *Chem. Commun.*, 2009, 1478 (DOI: 10.1039/b820887e)

applications, especially in life sciences,' says Schlaad.

For example, they could be used to target drugs and biomolecules to injured or cancerous tissues, says René Roy, an expert in carbohydrate chemistry and glycobiology at the University of Québec, Montréal, Canada. 'Schlaad's compounds have great potential in emerging glycobiology research. I see them having superb opportunities in carbohydrate-based vaccine technologies,' he adds.

Schlaad says that in the future, he hopes to generate smart vesicles with pH- or temperature-responsive membranes. 'External stimuli shall be used to induce either a morphological change or vesicle collapse to trigger cargo molecule or drug release,' he says. *Elizabeth Davies*

## Tea leaves produce cancer-fighting gold nanoparticles

# Time to put the kettle on?

Gold nanoparticles made using chemicals found in tea leaves could be used to combat cancer, say US scientists.

Kattesh Katti, Raghuraman Kannan and colleagues at the University of Missouri, Columbia, used phytochemicals (bioactive compounds) from Darjeeling tea to reduce gold salts to gold nanoparticles. The phytochemicals also stabilised the nanoparticles and covered them in a robust and non-toxic coating. Since only natural chemicals are used in this reaction, no toxic waste products are produced, making it a 100 per cent green process, says Katti.

Tea has been known for its health benefits for centuries and compounds found in tea have been used as dietary supplements and natural pharmaceuticals. The compounds scavenge disease-causing free radicals in the body. They are powerful reducing agents too, but research into these reactions is still in its infancy. Discovering that



phytochemicals in tea can initiate gold nanoparticle formation under non-toxic conditions is of paramount importance for medical and technological applications, says Katti.

**Bioactive compounds in Darjeeling tea produced nanoparticles with anticancer properties**

#### Reference

S K Nune *et al*, *J. Mater. Chem.*, 2009, DOI: 10.1039/b822015h

Typical reactions for forming gold nanoparticles use toxic chemicals, making them unsuitable as medicines. Also, thiols are used to stabilise and prevent merging of the nanoparticles, but this means that the particles can't bind to drug moieties that target disease sites. Katti's method gets around this problem, as the coating formed by the phytochemicals stops the nanoparticles merging but still allows them to bond with the drug moieties.

Katti's team tested their nanoparticles against prostate and breast cancer cells. They found that the particles had excellent affinity for the cancer cells' receptors, which means that they could be used in anticancer drugs.

'Green nanotechnology is an emerging area interfacing nanotechnology and natural sciences,' says Katti. 'Our process is feasible on larger scales and thus allows the discovery of more medical and technological applications of gold nanoparticles.' *Philippa Ross*



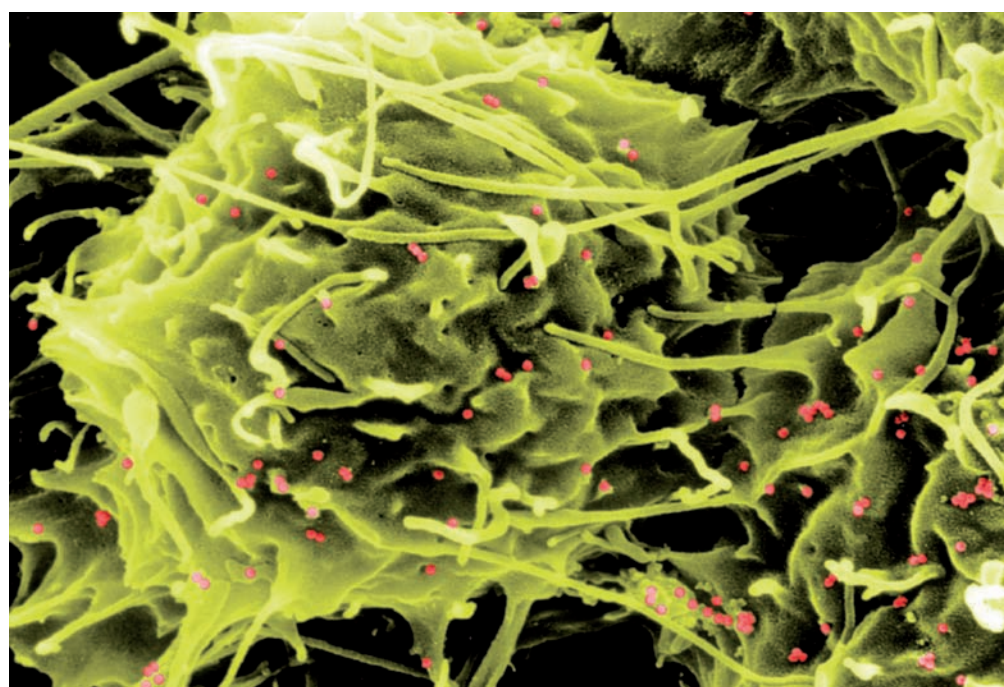
# The growth of nanotoxicology

Christy Haynes and colleagues from the University of Minnesota, US, look at analytical techniques used to assess nanotechnology's effects on health

The use of engineered nanomaterials in consumer products is expanding – a current report by the Woodrow Wilson International Center for Scholars and the Pew Charitable Trusts identifies more than 800 commercial nanomaterial-containing products, accounting for \$147 billion (approximately £104 billion) yearly. The materials show promise in disease treatment or solar power generation. Yet, despite the fact that so many are in commercial use, very little is known about their effects on health. As scientists around the world try to fill this information void, nanotoxicology research has grown rapidly and a wide variety of analytical techniques are used to assess biodistribution (tracking where the compounds travel in the body), cellular uptake and both in vivo and in vitro toxicity.

Nanotoxicity experiments are typically conducted on mice or rats and focus on LD<sub>50</sub> (exposure amount resulting in 50 per cent population death), changes to tissues or organs, or changes in blood cell populations and serum. These experiments give valuable information but are often time consuming, expensive and provide relatively little mechanistic information about underlying toxicity causes. There is also an ethical imperative to reduce the large animal numbers used in these studies.

In vitro assessment may be a better alternative. It can provide inexpensive and rapid nanomaterial interaction analysis on the cellular level. Material uptake and location can be assessed using electron microscopy, fluorescent confocal microscopy or



elemental analysis. On their own, these techniques have limitations so are best used in concert to get a good representation. In vitro assessment often relies on using bulk tissue samples from immortalised cell lines and toxicity biomarker probe molecules. In vivo toxicity is difficult to predict from the results – some nanomaterial classes (such as carbon nanotubes) interact with probe molecules directly, providing misleading results.

Scientists have developed new nanomaterial distribution and toxicity methods to tackle these challenges, but further methodological developments are needed. These include toxicity analysis techniques to discriminate individual cellular function within

**Toxicology studies reveal nanoparticles' uptake in the body, for example, the iron oxide particles (red) on the surface of white blood cells shown here**

mixed culture environments, label-free dynamic nanoparticle uptake analysis, nanoparticle surface characterisation within complex biological environments and point-of-source nanoparticle exposure analysis for workers.

Also, the worldwide nanotechnology community would benefit greatly from a set of standard toxicity screening protocols for engineered nanomaterials. This would allow scientists to develop safe nanotechnologies and would lessen public fear regarding exposure to nanomaterials, ultimately helping to unlock the full potential of these exciting materials.

Read more in 'Analytical methods to assess nanoparticle toxicity' in issue 3, 2009 of *Analyst*.

#### Reference

B J Marquis *et al*, *Analyst*, 2009, 425 (DOI: 10.1039/b818082b)

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# Creating a new world

*Mukund Chorghade speaks to Elinor Richards about his fascination with natural products and their role in India's future*



**Mukund Chorghade**

**Mukund Chorghade is President of Chorghade Enterprises and Chief Scientific Officer at THINQ (Technology, Health, Innovation, Novelty and Quality) Pharma, where he provides consultations to pharmaceutical companies on collaborations with academic, government and industrial laboratories. He is also a member of the IUPAC Chemistry and Human Health division.**

## **What inspired you to become a chemist?**

My father bought me a book called *Chemistry Creates a New World* by Bernard Jaffe when I was a teenager. It opened my eyes to all the wonderful things that chemistry can do. I read it from cover to cover in a day and I was spellbound by a chapter on new pharmaceuticals. I decided that this was what I was going to study, much to the dismay of my father, who wanted me to be a physicist.

## **When did your interest in natural products begin?**

When I started studying chemistry, a lot of the organic chemistry research was focused on natural products. In India, many professors were working on the isolation of natural products from traditional sources and I was fascinated by the rich variety of structures. We have come full circle because there is now an increased emphasis on the new ideas of reverse pharmacology. This concept brings natural products we have used for centuries back into mainstream sciences and proves the therapeutic efficacy due to their structures.

## **What projects are you currently involved in?**

At THINQ (Technology, Health, Innovation, Novelty and Quality) Pharma, we define new scalable process routes to new chemical entities. Someone could approach us with a medicinal chemistry route and ask us to make it more efficient, or to find different routes. Our goal is to make the drug better, faster and cheaper. We are also involved in contract medicinal chemistry where we synthesise compounds and analogues; we aim to do the drug discovery work ourselves using collaborations we have established with academics.

## **What was your proudest moment?**

In my industrial career, I was involved in the discovery of new processes, in particular a route to an antiepileptic drug called Tiagabine, which is now sold as Gabitril. My grandmother had suffered from epilepsy so it gives me a lot of pleasure to see a prescription filled using these particular antiepileptics.

## **What is your involvement with IUPAC?**

As a member of IUPAC's Chemistry and Human Health division, I have carried out some successful projects. These include compiling new glossaries of terms used in process chemistry and

pharmaceutics and producing a report on the use of natural products in traditional medicines in India and China.

## **What is the situation for the pharma industry in India?**

Drug discovery as a science is in its infancy, but is a rapidly growing area. Historically, the World Trade Organisation approved deals to allow poorer nations to import generic medicines manufactured in India and China, overriding international patents. Recent changes in the patent laws resulted in increased impetus for Indian pharmaceutical companies to invent new drugs. The government in India has been extraordinarily supportive of such ventures. As yet, there is no Indian drug on the market but I'm very optimistic.

## **Do academia and industry collaborate successfully in India?**

In India, there can be a gulf between the academic and industrial worlds. Some very good work from industry using state of the art techniques doesn't see the light of day because of patent and confidentiality issues. Another problem is the lack of industrial scientists delivering lectures in symposia. Industry and academia need to be encouraged to collaborate more in order to obtain research funding.

## **What is funding like in India?**

Now there are increased motivators for doing research in India, the whole scientific infrastructure has increased in size, sophistication and financing. The Indian government has announced many new initiatives where they will fund projects. If you have a good idea and if you can carry out the pioneering research, the government will fund it. Pharma companies in the West have been increasingly looking to India as a font of innovation.

## **How do you see the future of chemistry developing?**

I'm a tremendous supporter of chemistry, and not just because I am a chemist. I feel that chemistry is still the central science. Sometimes there are new trends and some might say that the computer can solve all your problems, or that biology can solve all the problems. That is not the case. Chemistry, biology and all these other disciplines need to work synergistically with each other.



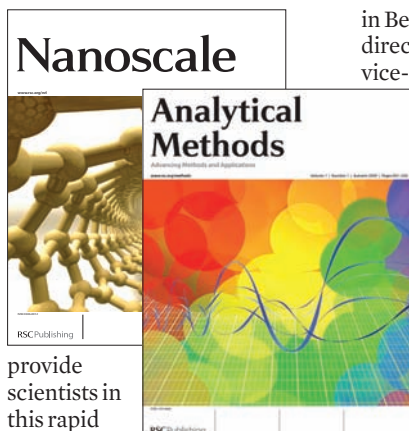
## Announcing two new journals

The prestigious RSC Publishing journal portfolio is set for further expansion with the launch of two new monthly titles in autumn 2009.

*Analytical Methods* will highlight new and improved methods for the practical application of analytical science. The journal will complement the existing RSC journal portfolio of analytical science publications, and with its focus on fundamental and applied modern analytical science, will appeal to both academic and industrial scientists.

*Analytical Methods* was announced at Pittcon in Chicago, IL, US, on 8 March. Delegates had the opportunity to be the first to find out about this exciting new journal.

*Nanoscale* will publish experimental and theoretical work across the breadth of nanoscience and nanotechnology. Highly interdisciplinary, the journal will



provide scientists in this rapid growth field with a new platform characterised by the quality and innovation for which RSC Publishing products are renowned.

*Nanoscale* will be published in collaboration with leading nanoscience research centre, the National Center for Nanoscience and Technology (NCNST)

in Beijing, China. Chunli Bai, director of NCNST and executive vice-president of the Chinese

Academy of Sciences, will be editor-in-chief of a new Asia-Pacific editorial office for *Nanoscale*. Markus Niederberger of ETH Zurich, Switzerland, and Francesco Stellacci from Massachusetts Institute of Technology, US, will head two further regional offices in Europe and North America.

From launch, the latest issue of *Analytical Methods* and *Nanoscale* will be freely available to all readers via the website. Free institutional online access to all 2009 and 2010 content will be available following a simple registration process.

Visit [www.rsc.org/methods](http://www.rsc.org/methods) and [www.rsc.org/nanoscale](http://www.rsc.org/nanoscale) to find out more.

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## Organic & Biomolecular Chemistry's 150th issue!

Issue 6, 2009 is the 150th issue of *OBC*. Since the first issue was published in January 2003, *OBC* has achieved tremendous success. With an impact factor of 3.167, can any other young journal boast such highly cited papers, published quickly after independent peer review?

Jeffrey Bode, University of Pennsylvania, US, comments, '*OBC* encourages and appreciates the development and application of innovative organic chemistry to a wide variety of contemporary problems. It is our choice for the

publication of new methods and concepts that reach beyond the traditional subdivisions of organic chemistry.'

Take a look at some of the high impact papers from leading scientists published in this 150th issue of *OBC*: a perspective on the design and synthesis of phosphole-based systems for novel organic materials by Yoshihiro Matano and Hiroshi Imahori; an emerging area article on metal-catalysed halogen exchange reactions of aryl halides by Tom Sheppard;

a communication on highly enantioselective asymmetric autocatalysis using chiral ruthenium complex-ion-exchanged synthetic hectorite as a chiral initiator by Kenso Soai and colleagues; and a full paper on ruthenium-based metallacrown complexes for the selective detection of lithium ions in water and in serum by fluorescence spectroscopy by Kay Severin *et al.* Don't miss these and the other articles in this celebratory issue. For more details visit [www.rsc.org/obc](http://www.rsc.org/obc)

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