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IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (22) 2197-2304 (2007)



Cover

See T. S. Andy Hor *et al.*, page 2225. This photo of an Au₆ triangle with Jurong Island (the "Chemical Island") in the background symbolizes Singapore's push in strategic chemistry research and its translation to economic output. Image reproduced by permission of Peili Teo, L. L. Koh and T. S. Andy Hor from *Chem. Commun.*, 2007, 2225.

CHEMICAL TECHNOLOGY

T41

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

Chemical Technology

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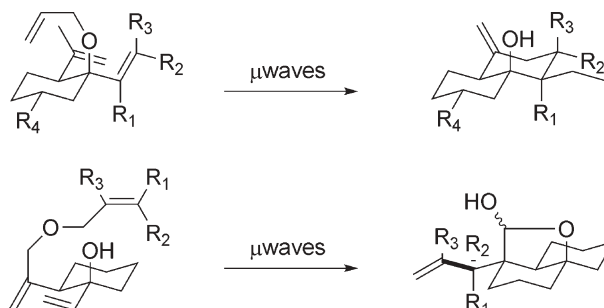
FEATURE ARTICLE

2211

Cascading pericyclic reactions: building complex carbon frameworks for natural product synthesis

Steve Arns and Louis Barriault*

Tandem reactions have emerged as powerful strategies to synthesize complex structures. The article describes recent advancement by our group in the development of novel tandem pericyclic reactions aimed at constructing diterpene frameworks.



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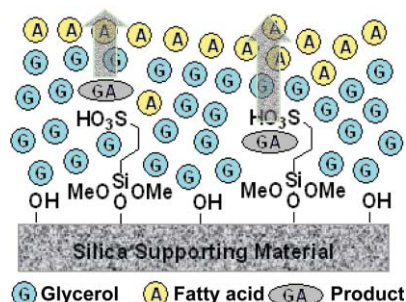
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Significant enhancement on selectivity in silica supported sulfonic acids catalyzed reactions

Ayman Karam, Yanlong Gu, François Jérôme,*
Jean-Paul Douliez and Joël Barrault

Consideration of the natural surface hydrophilicity of mesoporous silica supported sulfonic groups allows to perform more selective catalytic processes than homogeneous or usual solid acid catalysts for the direct transformation of polyfunctional derivatives.

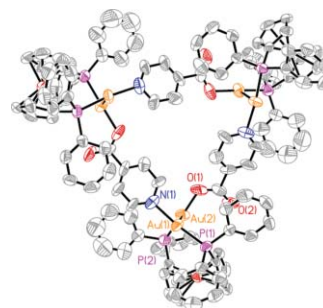


2225

Assembly of gold rings and chains with pyridyl carboxylate as directional spacer

Peili Teo, L. L. Koh and T. S. Andy Hor*

Different gold assemblies are realized through the use of pyridyl carboxylates with different spatial and directional properties. One of such examples is a hexagold triangular ring formed from cascading spacers and Au...Au aurophilic interaction.

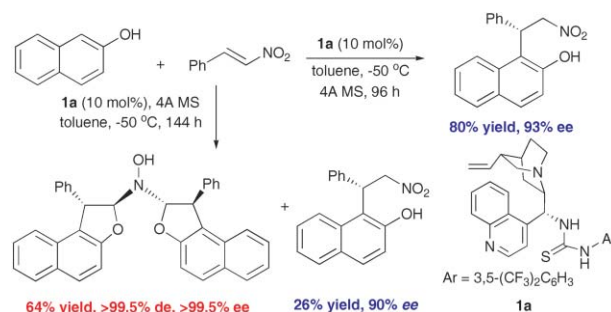


2228

Organocatalytic asymmetric Friedel–Crafts alkylation/cascade reactions of naphthols and nitroolefins

Tian-Yu Liu, Hai-Lei Cui, Qian Chai, Jun Long, Bang-Jing Li, Yong Wu, Li-Sheng Ding and Ying-Chun Chen*

The enantioselective Michael-type Friedel–Crafts alkylations of naphthols and nitroolefins catalysed by a bifunctional thiourea–tertiary amine were investigated; on extending the reaction time dimerisation of the F–C product could occur to give enantiopure 1,2-dihydronaphtho[2,1-*b*]furanyl-2-hydroxylamine derivatives.



2231

Computational structure–activity relationships in H₂ storage: how placement of N atoms affects release temperatures in organic liquid storage materials

Eric Clot, Odile Eisenstein* and Robert H. Crabtree*

For hydrogen storage in organic liquids, DFT calculations show that the ring type and N substituent positions strongly affect the dehydrogenation thermodynamics.



Design of materials by DFT calculations

Tissue Engineering in Microsystems

Lab on a Chip has gathered together a series of articles highlighting the very best research on cell and tissue engineering in microsystems.

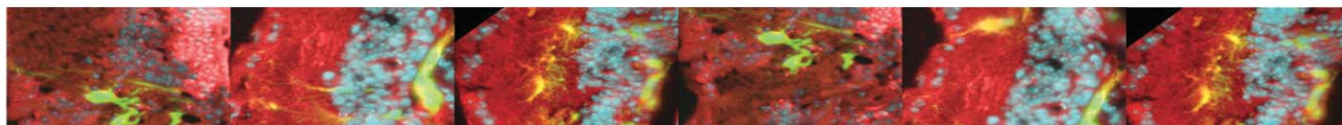
Guest editors Sangeeta Bhatia (MIT) and Christopher Chen (University of Pennsylvania) have commissioned articles from leading researchers to contribute to this *Lab on a Chip* issue, dedicated to state-of-the-art research on tissue engineering in microsystems.

The issue includes a critical review of cell micropatterning techniques; a tutorial review of perfusion culture of mammalian cells; and several high quality full papers on topics covering cell culture, patterning of biomaterials, stem cell differentiation, biocompatible implants, 3D tissue culture, embryoid bodies, cell cytotoxicity analysis and cell-cell communication.



“Tissue engineering is probably the most promising area of biology and biotechnology, this is an excellent issue featuring the best authors at the leading-edge of on-chip tissue engineering, — congratulations to Chris and Sangeeta”

Andreas Manz, ISAS, Dortmund



PAPERS INCLUDE:

A chip-based platform for the *in vitro* generation of tissues in three-dimensional organization

Eric Gottwald, Stefan Giselbrecht, Caroline Augspurger, Brigitte Lahni, Nina Dambrowsky, Roman Truckenmüller, Volker Piotter, Thomas Gietzelt, Oliver Wendt, Wilhelm Pfleging, Alex Welle, Alexandra Rolletschek, Anna M. Wobus and Karl-Friedrich Weibezahn, *Lab Chip* 2007, **7** (6)

Understanding microchannel culture: parameters involved in soluble factor signaling

Hongmei Yu, Caroline M. Alexander and David J. Beebe, *Lab Chip* 2007, **7** (6)

Efficient formation of uniform-sized embryoid bodies using a compartmentalized microchannel device

Yu-suke Torisawa, Bor-han Chueh, Dongeun Huh, Poornapriya Ramamurthy, Therese M. Roth, Kate F. Barald and Shuichi Takayama, *Lab Chip* 2007, **7** (6)

Micro-bioreactor array for controllable differentiation of human embryonic stem cells

Elisa Figallo, Christopher Cannizzaro, Sharon Gerecht, Jason A. Burdick, Robert Langer, Nicola Elvassore and Gordana Vunjak-Novakovic, *Lab Chip* 2007, **7** (6)

Survival, migration and differentiation of retinal progenitor cells transplanted on micro-machined poly(methylmethacrylate) scaffolds to the subretinal space

Sarah Tao, Conan Young, Stephen Redenti, Yiqin Zhang, Henry Klassen, Tejal Desai, Michael J. Young, *Lab Chip* 2007, **7** (6)



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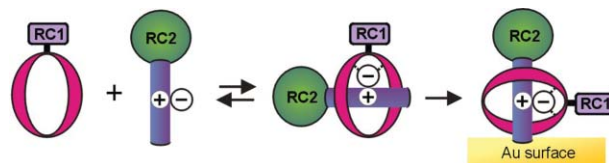
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2234

Anion templated surface assembly of a redox-active sensory rotaxane

Simon R. Bayly, Thomas M. Gray, Michał J. Chmielewski, Jason J. Davis* and Paul D. Beer*

Anion templation is used for the first time to assemble novel redox-active bis-ferrocene functionalised rotaxane SAMs on to gold electrode surfaces, which by virtue of the rotaxane SAM's interlocked cavity are capable of selectively sensing chloride anions using electrochemical methods.

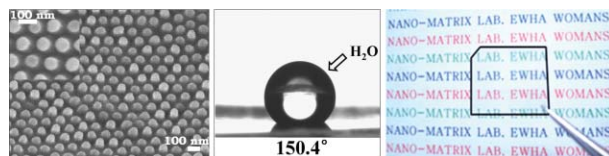


2237

A simple fabrication route to a highly transparent super-hydrophobic surface with a poly(dimethylsiloxane) coated flexible mold

Mihee Kim, Kyunghoon Kim, Nae Yoon Lee, Kyusoon Shin* and Youn Sang Kim*

A highly transparent super-hydrophobic surface is prepared using a nanoimprint lithographic technique with a PDMS-coated flexible mold.

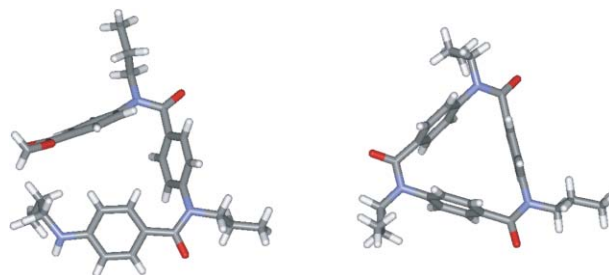


2240

Macrocyclic scaffolds derived from *p*-aminobenzoic acid

Fred Campbell, Jeffrey Plante, Christopher Carruthers, Michael J. Hardie, Timothy J. Prior and Andrew J. Wilson*

Synthetic routes to macrocyclic scaffolds with defined sequences of peripheral functional groups are rare. In this paper this is achieved for C₃ macrocyclic scaffolds derived from *p*-aminobenzoic acid monomers.

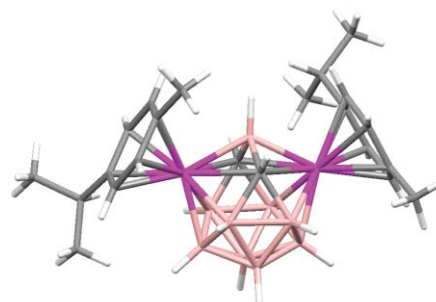


2243

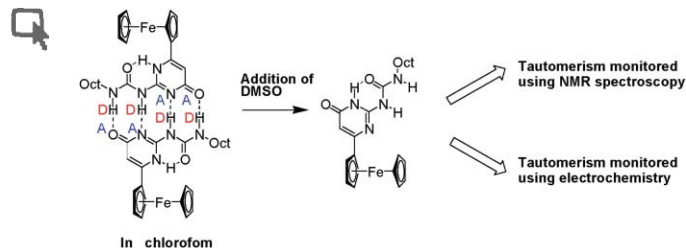
Symmetric and asymmetric 13-vertex bimetallacarboranes by polyhedral expansion

Maria Elena Lopez, Michael J. Edie, David Ellis, Anke Horneber, Stuart A. Macgregor, Georgina M. Rosair and Alan J. Welch*

Symmetric 4,5,2,3-*M*₂C₂B₉ 13-vertex bimetallacarboranes of cobalt and ruthenium and their asymmetric 4,5,1,6-*M*₂C₂B₉ isomers are described.



2246

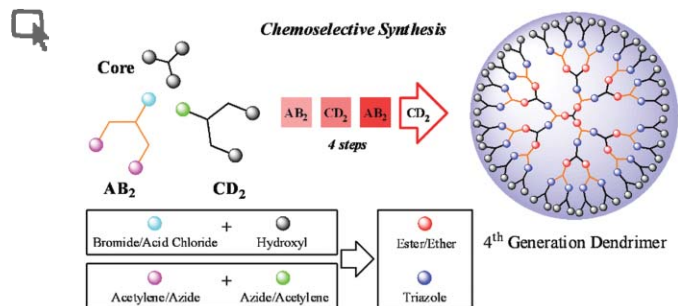


Probing the solvent-induced tautomerism of a redox-active ureidopyrimidinone

Anne-Marie Alexander, Marc Bria, Gunther Brunklaus, Stuart Caldwell, Graeme Cooke,* James F. Garety, Shanika G. Hewage, Yann Hocquel, Niall McDonald, Gouher Rabani, Georgina Rosair, Brian O. Smith, Hans Wolfgang Spiess, Vincent M. Rotello and Patrice Woisel

A ferrocene-functionalised ureidopyrimidinone has been synthesised that can signal solvent-induced tautomerism.

2249

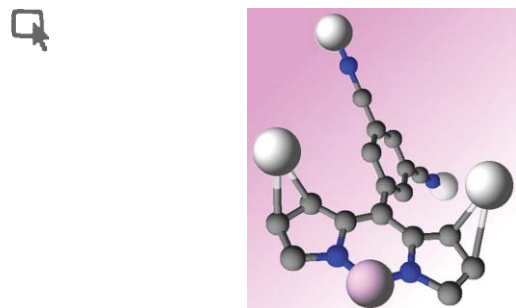


A chemoselective approach for the accelerated synthesis of well-defined dendritic architectures

Per Antoni, Daniel Nyström, Craig J. Hawker, Anders Hult and Michael Malkoch*

A chemoselective accelerated approach that goes beyond traditional strategies for dendrimers synthesis utilizing two highly versatile reactions in sequence which avoids numerous activation or deprotection steps.

2252

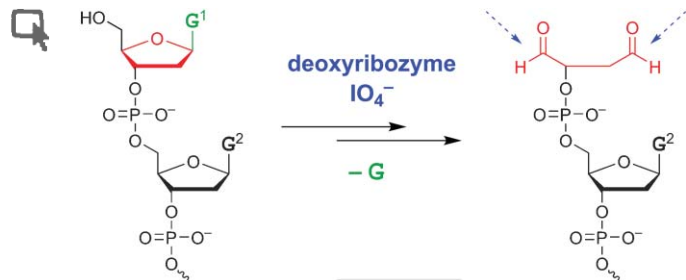


Beyond classical coordination: silver- π interactions in metal dipyrin complexes

Domingo Salazar-Mendoza, Stéphane A. Baudron* and Mir Wais Hosseini*

Homoleptic and heteroleptic Zn and Cu complexes of dipyrin type ligands bearing mono- and di-cyanophenyl groups when combined with silver cations lead to the formation of Ag(I)-C=C double bond interactions unprecedented in the crystalline phase for pyrrolic units.

2255



Site-selective depurination by a periodate-dependent deoxyribozyme

Claudia Höbartner, P. I. Pradeepkumar and Scott K. Silverman*

Ribozyms and deoxyribozyms that use small-molecule compounds as cofactors are interesting from both fundamental and applied viewpoints. A deoxyribozyme has been identified that site-selectively depurinates its 5'-terminal guanosine nucleotide using periodate (IO_4^-) as an obligatory cofactor.

2258

***b*-Bilene to *a,c*-biladiene transformation during syntheses of isoporphyrins and porphyrins**

Celinah Mwakwari, Frank R. Fronczek and Kevin M. Smith*

Zinc(II) isoporphyrins are formed in good yields from reactions of *b*-bilenes with α -ketoesters by way of *a,c*-biladiene complexes; the resulting zinc(II) isoporphyrins can be smoothly transformed into *meso*-substituted porphyrins by one-pot saponification and decarboxylation.

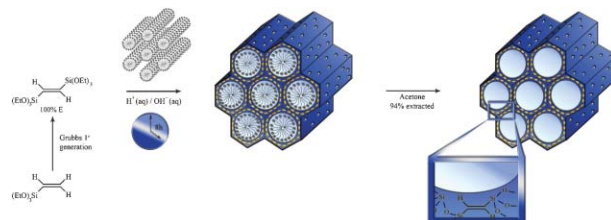


2261

Ultra-fast hydrothermal synthesis of diastereoselective pure ethylene-bridged periodic mesoporous organosilicas

Carl Vercaemst,* Matthias Ide, Bart Allaert, Nele Ledoux, Francis Verpoort and Pascal Van Der Voort*

An ultra-fast synthesis procedure of diastereoselective pure ethylene-bridged PMO's with exceptional properties, combined with a more efficient, mild and fast extraction method for polymeric templates is presented.

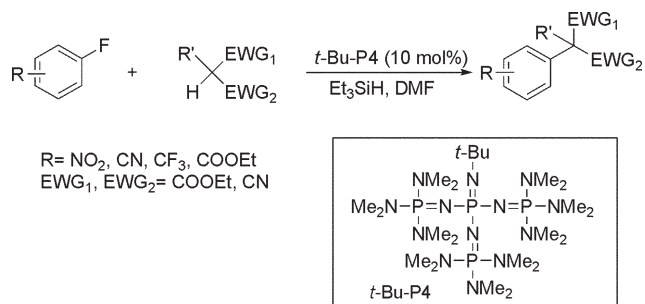


2264

Nucleophilic aromatic substitution using Et₃SiH/cat. *t*-Bu-P4 as a system for nucleophile activation

Masahiro Ueno, Misato Yonemoto, Masahiro Hashimoto, Andrew E. H. Wheatley, Hiroshi Naka and Yoshinori Kondo*

Excellent deprotonative arylations of nucleophiles were achieved using Et₃SiH and catalytic *t*-Bu-P4 as a novel activation system.

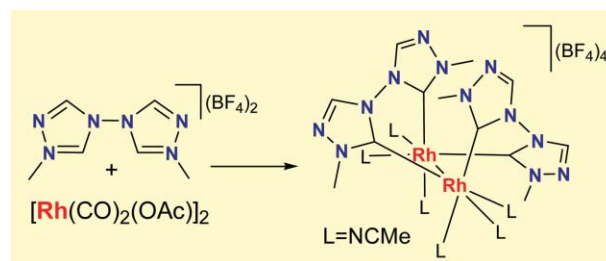


2267

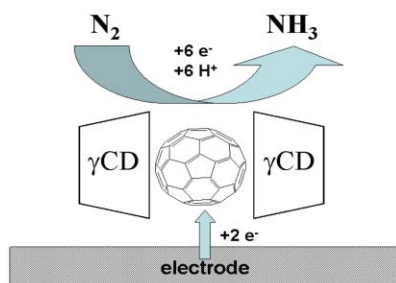
A planar chelating bitriazole N-heterocyclic carbene ligand and its rhodium(III) and dirhodium(II) complexes

Macarena Poyatos, William McNamara, Chris Incarvito, Eduardo Peris* and Robert H. Crabtree*

A new planar dipyrindyl-like bitriazole-2-ylidene ligand (bitz ligand) is described. Its very high chelating tendency contrasts to other bis-NHCs with alkyl linkers. The bitz ligand also coordinates under milder conditions than those for conventional NHCs, no base being required. Mono- and bis-chelate Rh(III) complexes as well as an unprecedented dirhodium(II) species are reported.



2270

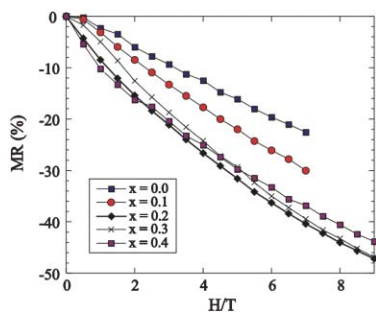


Electrochemical conversion of dinitrogen to ammonia mediated by a complex of fullerene C₆₀ and γ -cyclodextrin

Lubomír Pospíšil,* Jana Bulíčková, Magdaléna Hromadová, Miroslav Gál, Svatopluk Civiš, Jaroslav Cihelka and Ján Tarábek

Electrochemical conversion of N₂ to NH₃ at ambient pressure and 60 °C is reported. Nitrogen fixation process is mediated by the complex [C₆₀-(γ CD)₂] reduced on a mercury pool electrode.

2273

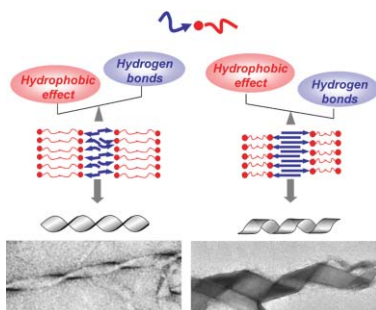


Enhancement of large magnetoresistances in ruthenocuprates by Ta substitution

A. C. McLaughlin,* L. Begg, A. J. McCue and J. P. Attfield

An unexpected enhancement of the large negative magnetoresistance (MR) observed in RuSr₂Nd_{0.95}Y_{0.15}Ce_{0.9}Cu₂O_{10- δ} up to -47% at 4 K and 9 T is evidenced upon dilution of the Ru magnetic order by substitution of Ta for Ru; this enhancement of -MR scales with the cell volume.

2275

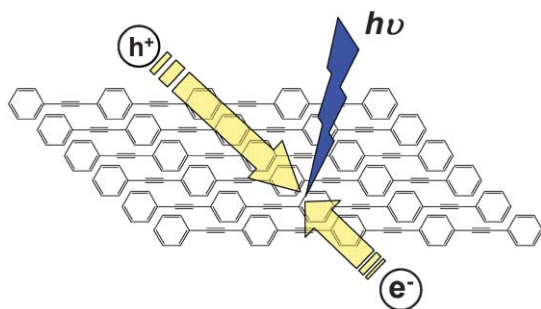


Control of nano-micrometric twist and helical ribbon formation with gemini-oligoalanine *via* interpeptidic β -sheet structure formation

Aurélie Brizard, Roni Kiagus Ahmad and Reiko Oda*

Twist or helical: confinement of alanine peptides at membrane surfaces can induce their secondary structure formation. Such organised peptides can reciprocally transfer their chirality to membranes with non-chiral amphiphiles and form a variety of chiral structures.

2278



Electrical properties of 1,4-bis(4-(phenylethynyl)-phenylethynyl)benzene and its application for organic light emitting diodes

Larysa Fenenko, Guang Shao, Akihiro Orita, Masayuki Yahiro, Junzo Otera, Sergei Svechnikov and Chihaya Adachi*

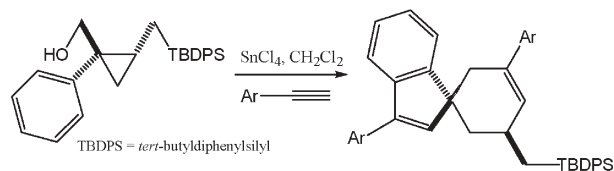
A phenylene ethynylene derivative, 1,4-bis(4-(phenylethynyl)-phenylethynyl)benzene (BPPB), provides very high blue photoluminescence efficiency both in solution ($\Phi_{\text{PL}} = 95 \pm 3\%$) and thin films ($\Phi_{\text{PL}} = 71 \pm 3\%$), leading to efficient blue luminescence.

2281

Lewis acid-catalyzed formation of indene derivatives via tandem reactions of arylacetylenes with the cations generated from 2-silylmethyl cyclopropyl carbinols

Veejendra K. Yadav,* Naganaboina Vijaya Kumar and Masood Parvez

2-Silylmethyl cyclopropyl carbinols undergo tandem intermolecular cation–arylacetylene cyclization to generate indene derivatives.

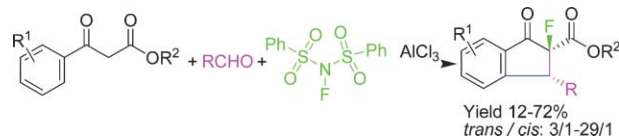


2284

Stereoselective construction of fluorinated indanone derivatives via a triple cascade Lewis acid-catalyzed reaction

Han-Feng Cui, Ke-Yan Dong, Guang-Wu Zhang, Lian Wang and Jun-An Ma*

A triple cascade Knoevenagel condensation/Nazarov cyclization/electrophilic fluorination process was developed. This Lewis acid-catalyzed sequence proceeds to afford a series of fluorinated 1-indanone derivatives in moderate to good yields with high stereoselectivities.

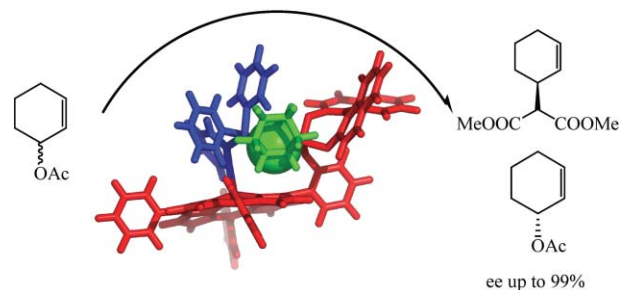


2287

SUPRAphos-based palladium catalysts for the kinetic resolution of racemic cyclohexenyl acetate

Xiao-Bin Jiang, Piet W. N. M. van Leeuwen and Joost N. H. Reek*

High-throughput screening of the SUPRAphos library revealed a palladium catalyst based on supramolecular ligands that gave fast and highly efficient kinetic resolution of cyclohexenyl acetate.

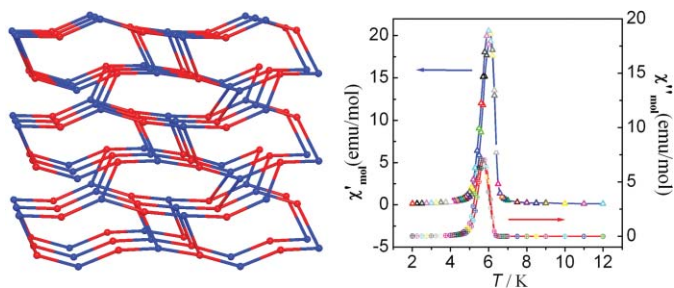


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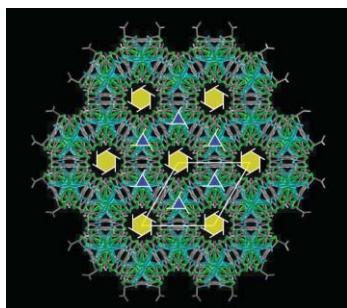
Magnetic canting or not? Two isomorphous 3D Co^{II} and Ni^{II} coordination polymers with the rare non-interpenetrated (10,3)-d topological network, showing spin-canted antiferromagnetism only in the Co^{II} system

Jian-Rong Li, Qun Yu, Ying Tao, Xian-He Bu,* Joan Ribas and Stuart R. Batten

Two isomorphous complexes, $[M_2(L)_2(H_2O)_2]_n$ ($M = Co^{II}$, Ni^{II} ; $L = 2,1,3$ -thiadiazole-4,5-dicarboxylate), with non-interpenetrated (10,3)-d (**utp**) network and showing spin-canted antiferromagnetism for the Co^{II} system, but antiferromagnetic coupling for the Ni^{II} are reported.



2293

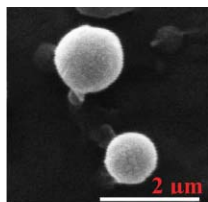


Unprecedented interweaving of single-helical and unequal double-helical chains into chiral metal-organic open frameworks with multiwalled tubular structures

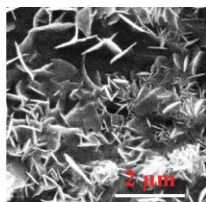
Su-Na Wang, Hang Xing, Yi-Zhi Li, Junfeng Bai,*
Manfred Scheer,* Yi Pan* and Xiao-Zeng You

Two novel chiral metal-organic open frameworks with unprecedented multiwalled tubular channels and interweaving of single-helical and unequal double-helical units are reported.

2296



Peptide Vesicles



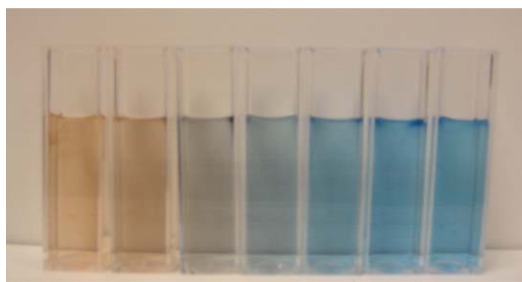
Disrupted Vesicles

Self-assembly and potassium ion triggered disruption of peptide-based soft structures

Surajit Ghosh, Sukhmani Kaur Singh and Sandeep Verma*

The authors describe the formation of self-assembled structures by PWWP tetrapeptide, guest entrapment studies and its disruption in the presence of potassium ions.

2299



Sugar sensing based on induced pH changes

Youngmi Kim, Scott A. Hilderbrand, Ralph Weissleder and Ching-Hsuan Tung*


A novel sensing assembly consisting of a pH sensitive NIR dye and a boronic acid derivative suggests a new approach for the detection of sugar based on pH changes induced in the sensory assembly.

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Chemical Technology

Washing with eutectic solvents cleans up biodiesel and produces glycerol

Mild green ionic liquids

Biodiesel production could become more environmentally friendly thanks to researchers in the UK who are using non-toxic ionic liquids to remove unwanted by-products.

The main by-product in the production of biodiesel from vegetable oils is glycerol. This syrupy alcohol must be removed from the fuel as it can damage engines. Andrew Abbott and colleagues from the University of Leicester have developed a simple new approach to this sticky problem: they use green ionic liquids called deep eutectic solvents to just wash the glycerol out of the biodiesel.

Deep eutectic solvents are two or more substances mixed in a ratio that has a melting point much lower than any of the constituents. These are generally made from an organic halide salt that is complexed with something that will form a hydrogen bond. Abbott's team used quaternary ammonium salts complexed with glycerol as the washing liquid. This green washing



liquid has many advantages including its low cost and toxicity; in fact, one salt used is choline chloride – vitamin B4.

This procedure not only has potential as a greener method of cleaning up biodiesel, but also as a method of producing pure glycerol. Alexei Lapkin of the University of Bath commented that the work 'is a very interesting development for downstream glycerol treatment and utilisation'.

Making a green process even greener

Abbott hopes that 'further research will reveal a better method for recycling the salt and recovering the glycerol'. He added that the team want 'to collaborate with a biodiesel producer to test this technology on a practical scale'.

Wendy Crocker

Reference

A P Abbott *et al*, *Green Chem.*, 2007 (DOI: 10.1039/b702833d)

In this issue

Hot-wiring enzymes for fuel cells

Linking laccase to electrodes delivers electrons straight to the active site

The clean art of conservation

Supercritical carbon dioxide used to restore ancient cloth

Interview: Health and natural hazards

José A Centeno talks to Kathryn Lees about how Mother Nature affects our health

Instant insight: Back in black

Hydrothermal carbonisation of biomass: an efficient process to treat the CO₂ problem?



The latest applications and technological aspects of research across the chemical sciences

Application highlights

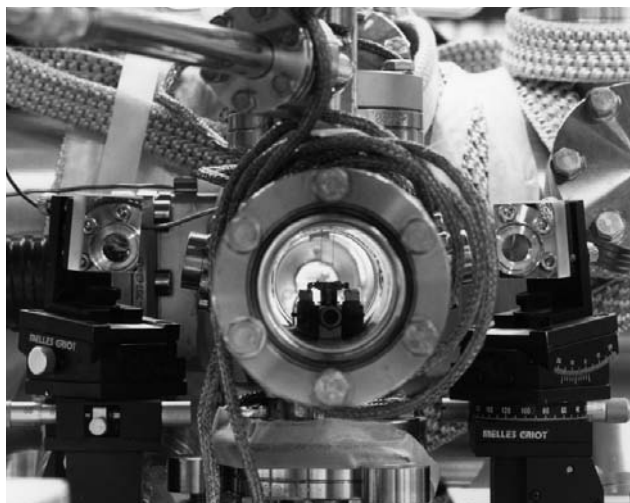
Polycrystal catalysts studied by IR at realistic pressures

Bridging the industry–laboratory gap

The challenge of relating results from laboratory tests of single crystal catalysts to their more complex industrial applications is being met head on by researchers at Shell Global Solutions.

Forming long chain hydrocarbons from carbon monoxide and hydrogen by the Fischer–Tropsch process is of huge interest. Unfortunately most studies into new catalysts do not use catalysts or conditions that reflect their use in industry. However, Heiko Oosterbeek at Shell Global Solutions in the Netherlands has studied the surface science of Fischer–Tropsch catalysts under more realistic conditions to try and bridge the gap between lab and industrial conditions.

Traditionally, catalyst characterisation is done under conditions very different to those found in industry, for example, ultra high vacuum (UHV) characterisation of catalysts to be



Lab tests are often under unrealistic conditions

Reference

H Oosterbeek, *Phys. Chem. Chem. Phys.*, 2007, DOI: 10.1039/b703003g

used at high pressures. Oosterbeek prepared both single and polycrystals of cobalt that resembled real Co/SiO₂ catalysts. He then used infra-red techniques to characterise them, and tested the catalysts at both UHV and high pressure.

‘The work aims to show the synergy between robust industrial experience and subtle theoretical insight into the structural aspects of heterogeneous catalysis,’ said Oosterbeek.

These methods could lead to valuable new discoveries. Carlos Otero Arean from Universidad de las Islas, Spain, said ‘Oosterbeek’s work precisely addresses the gap between model studies and working catalysts, as applied to Fischer–Tropsch synthesis. He successfully shows how a combination of carefully designed experimental work and state-of-the-art techniques holds the potential to yield highly relevant new insights.’

Oosterbeek said the next challenge is to apply the techniques at pressures above atmospheric pressure and further close the material, pressure and instrument gap between the lab and industry. *Edward Morgan*

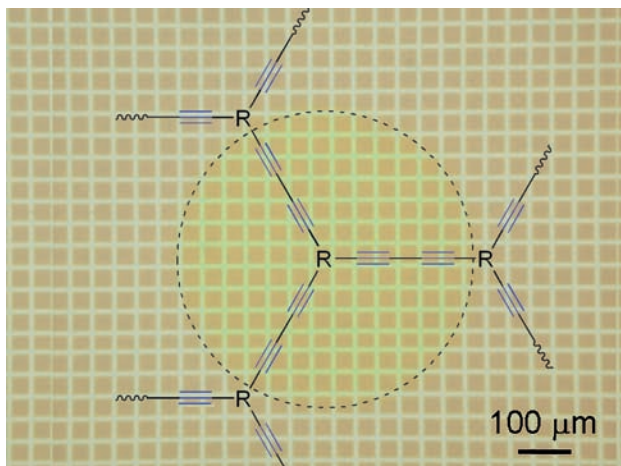
Organometallic cluster enhances polymer’s optical properties

Tunable photonics with high refractive index

Scientists in Hong Kong have created a polymer with a refractive index that can be tuned as high as 1.81.

The refractive index (RI) is a measure of how much the speed of light is reduced in a medium. Conventional organic glasses such as PMMA have a particularly low RI and also within a narrow range. Therefore although they are cheap to produce, flexible and easy to coat onto large surfaces, their optical properties are limited.

Ben Zhong Tang’s group from the Hong Kong University of Science and Technology have attached organometallic nanoclusters to the triple bond network of a hyperbranched polydiyne. This had the effect of increasing the refractive index to 1.81, vastly improving its optical performance.



In addition, they discovered that under UV irradiation the organometallic moiety broke away from the polymer complex. This caused the RI to drop by as much as 0.05 in the 1500–1600 nm region,

The polymer is a hyperbranched polydiyne

producing a visible photochromic response.

‘Such an unusually high RI change makes our polymer an interesting material for waveguide, memory and holographic storage applications,’ said Tang.

In the future, the group hope to induce the change in the RI of this polymer in a reversible manner. They believe that this could lead to write and read as well as erase and rewrite optical memories. ‘Such photonic materials could dramatically enhance the data processing speed and give optical holographic data storage applications with much higher accessible capacity,’ said Tang. *Jenna Wilson*

Reference

M Häußler *et al*, *Chem. Commun.*, 2007, DOI: 10.1039/b702915b

Supercritical carbon dioxide used to restore ancient cloth

The clean art of conservation

Scientists have developed the first environmentally friendly approach to cleaning fragile historic textiles that have seen better days.

Ana Aguiar-Ricardo from the New University of Lisbon, Portugal, and colleagues, used liquid and supercritical carbon dioxide (CO₂) as dry cleaning solvents to restore the garments of the Virgin and Child, an eighteenth century sculpture from Necessidades Palace in Lisbon.

The cleaning of antique textiles is often an unavoidable step in their conservation, but many traditional mechanical or wet methods are considered too harsh to use on much deteriorated textiles. In this situation the conservator may decide the risk is too great or use dry cleaning with perchloroethylene and other toxic organic solvents that remove the dirt and stabilise the textile.

There is an urgent need to preserve our history and cultural heritage. However, to do this art and science must work together so that technological developments do not harm the environment or our quality of life, said Aguiar-Ricardo.



Time has not been kind to the clothes on the Virgin and Child

Using CO₂ is perfect as it is considered non-flammable, relatively non-toxic and relatively inert: 'It allows the conservation and restoration of the textile without being harmful to the operator and the environment,' said Aguiar-Ricardo. Above a critical temperature and pressure CO₂ will become supercritical and have gas-like viscosities and liquid-like densities. Small changes in temperature or pressure cause dramatic changes in its density, viscosity and dielectric properties making it an unusually tunable, versatile and selective solvent.

'What a beautiful application of supercritical carbon dioxide,' said Martyn Poliakov, an expert on clean technology at the University of Nottingham, UK. 'Scaling this up to work on real objects will provide wonderful opportunities for interdisciplinary research between chemists, engineers and conservators,' he said.

Sarah Corcoran

Reference

M Sousa et al, *Green Chem.*, 2007, DOI: 10.1039/b617543k

Linking laccase to electrodes delivers electrons straight to the active site

Hot-wiring enzymes for fuel cells

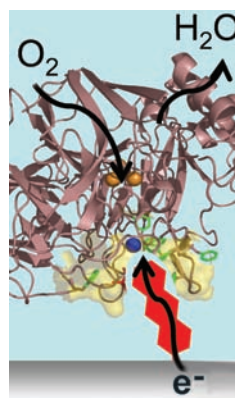
Stable enzyme-modified graphite electrodes could be used in fuel cells, according to researchers in the UK.

Laccases are copper-containing enzymes that use electrons to reduce oxygen to water efficiently, making them of interest as electrocatalysts at the cathodes of hydrogen-oxygen fuel cells. In this type of fuel cell, hydrogen is oxidised to hydrogen ions and electrons, which flow to the cathode (supplying power) where they reduce oxygen.

Fraser Armstrong and colleagues at the University of Oxford chemically attached anthracene to graphite electrodes to form a

stable attachment between the electrode and laccase. Armstrong described anthracene as 'a long hydrophobic molecule, similar to laccase's own substrate molecules, which can penetrate the active site pocket and approach closely to one of the copper atoms' where the oxygen reduction takes place. The anthracene not only binds the laccase to the electrode, but, as it can conduct electrons, it also delivers them deep into the enzyme close to the site for oxygen reduction.

The anthracene 'plug' modification of the electrode is essential to provide the high and long-term oxygen-reduction activity. Without it, the electrons



Reference
C F Blanford, R S Heath and F A Armstrong, *Chem. Commun.*, 2007, 1710

are not delivered efficiently into the enzyme and the binding of the laccase to the electrode is not stable, both of which leads to reduced activity. The ability to use graphite is also an advantage; it is relatively cheap compared to precious metals, such as gold, which have been previously used as electrodes for laccases.

To complete the fuel cell, Armstrong said that in future research they would like to 'identify rational attachment strategies for other enzymes, particularly hydrogenases, which can be incorporated with laccases to produce small fuel cells'.

Nicola Convine

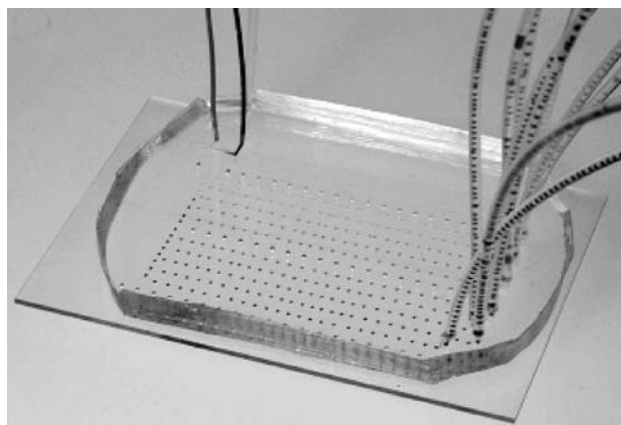
A range of conditions can be quantitatively assessed in a few hours

On-chip solubility screening

Scientists in France have devised a new way to rapidly and cheaply screen how soluble compounds are.

Chemical and biological systems often rely on a large number of parameters. Complete investigations of these parameters need time and significant amounts of product. Currently these investigations use robotic fluidic workstations, but these are expensive, and require labour and large product volumes. Alternatively, high throughput microfluidic techniques are used as they allow rapid screening, but fabrication and multiplexing are difficult.

Philippe Laval and colleagues working for a joint Rhodia–CNRS research centre have produced a chip for screening phase and solubility conditions. A series of droplets with differing compositions is produced by



mixing silicone oil and aqueous phases at different rates. Ten channels containing different droplet compositions can be created in less than 20 minutes.

After the device is full it is rapidly cooled to induce crystallisation. Once the solutions have

A heat gradient is applied to a 2D array of concentrations

Reference

P Laval *et al*, *Lab Chip*, 2007, DOI: 10.1039/b700799j

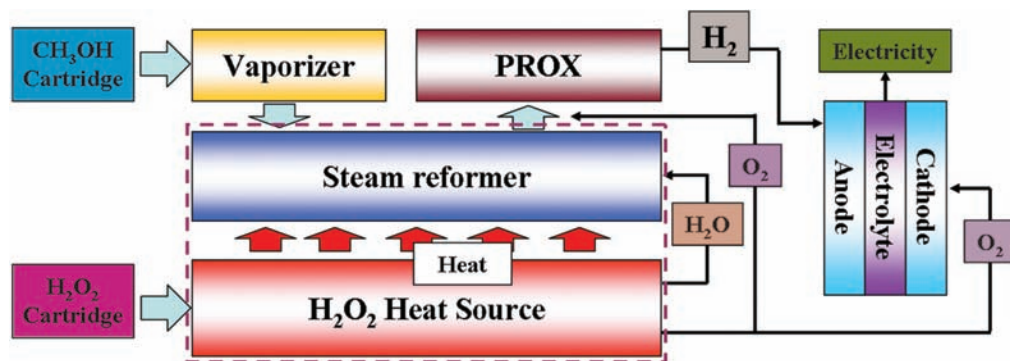
crystallised, a temperature gradient is applied to produce a two-dimensional array of droplets of different concentrations at different temperatures. In droplets where the temperature is higher than the solubility temperature, the crystals dissolve. In other droplets the crystals are partly solubilised, but still exist in equilibrium, producing a direct and quantitative solubility assessment.

‘The microfluidic device has been developed to improve research productivity’, said Laval, adding ‘This low cost system, compared to robotic platforms, enables us to carry out high throughput screening of concentration vs. temperature diagrams with very little amount of products’. He continued that an additional advantage is that ‘tens of experimental conditions can be tested in just a few hours’.

Vikki Chapman

Hydrogen peroxide is decomposed to provide heat for fuel cells

The drive to reform methanol



Researchers in Korea have increased the efficiency of polymer electrolyte membrane (PEM) fuel cells by about 10%.

Sejin Kwon and colleagues at the Korea Advanced Institute of Science and Technology, Daejeon, used the decomposition of hydrogen peroxide to provide the heat needed to drive the production of hydrogen

from methanol.

PEM fuel cells produce electrical energy from hydrogen and oxygen. But since hydrogen has such a low energy density when stored as a gas, it is often stored indirectly as a liquid fuel, such as methanol. Hydrogen is produced from the methanol as required in a process called ‘steam reforming’, but as

Crucially, the water and oxygen also produced is recycled

this process is endothermic a heat source is also needed.

Kwon made a compact methanol reformer that uses the decomposition of hydrogen peroxide as the heat source. One of the key features of the reformer is that the products of the reaction, water vapour and oxygen, are then recycled. The water vapour is used for the steam reforming and the oxygen to oxidise any carbon monoxide produced during methanol reforming and also to supplement the oxygen from the air in the fuel cell itself. Furthermore, this oxygen enrichment boosts the efficiency of the fuel cell.

The researchers envisage the use of such a system as substitutes for batteries in mobile applications.

Madelaine Chapman

Reference

S Kwon, J S Hwang and T Kim, *Lab Chip*, 2007, DOI: 10.1039/b700040e

Back in black

Markus Antonietti, Arne Thomas and Maria Titirici discuss the hydrothermal carbonization of biomass – is it a solution to the CO₂ problem?

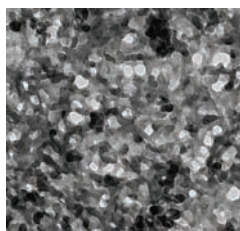
The global atmospheric concentration of carbon dioxide (CO₂) has increased markedly as a result of human activities since the industrial revolution. The inevitable impact on the world climate, recently announced by the IPCC (Intergovernmental Panel on Climate Change),¹ has prompted intensive discussions from politics, industry and the scientific community about how to treat the CO₂ problem.

Energy storage and saving and a more responsible usage of the remaining energy resources is unavoidable. Replacing parts of the energy production system by biomass schemes (bioethanol, -diesel or -gas) or the artificial sequestration of CO₂ (in the ocean or into underground geological formations) are also under discussion. All these methods can lower future CO₂ emission, but cannot compensate for past and current emissions of CO₂ from fossil fuels. However, the IPCC report claims that anthropogenic warming and sea level rise will continue for centuries due to the timescales associated with climate processes and feedback, even if greenhouse gas concentrations were to be stabilized immediately. It would therefore be highly desirable to not only slow down further CO₂ emissions, but also to invert the current development by sequestering the atmospheric CO₂ released by many years of industrialization.

The biggest carbon converter, with the highest efficiency to bind CO₂ from the atmosphere, is biomass. Interestingly, the removal of just 8.5% of freshly produced biomass from active ecosystems would compensate for all the CO₂ liberated from oil. Coal formation from biomass is one of the natural sinks that has been active in the past on the longest scale. Natural coalification of biomass takes place on a timescale of some hundred



Biomass can be converted to a form of carbon much like coal



A variety of nanostructures and porous architectures can be made

References

- <http://www.ipcc.ch/>
- M-M Titirici, A Thomas and M Antonietti, *New J. Chem.*, 2007, DOI: 10.1039/b616045j

(peat) to hundred million (black coal) years. Due to its slowness, it is usually not considered in renewable energy exploitation schemes or as an active sink in CO₂ cycles. Nevertheless, it is obvious that carbon fixation into coal is a lasting effort, as brown or black coal are practically not biodegradable. Thus, turning coal formation into an active element of carbon sequestration schemes would be very meaningful, but requires the acceleration of the underlying coalification processes.

Hydrothermal carbonization (HTC) can be such a process:² HTC describes heating carbohydrate sources in water, e.g. biomass dispersions, in a closed reaction vessel for 4–24 h to temperatures around 200°C. Upon dehydration of the carbohydrates, carbon with a chemical composition similar to brown coal is observed. Thus, HTC is an extremely simple, cheap and easily scalable process. Furthermore, it was shown that HTC of carbohydrates can yield interesting carbon micro- and nanostructures, such as carbon microspheres. Using suitable additives, carbon nanocables and fibres, porous carbon architectures and metal-carbon composite materials can be generated.

In CO₂ conversion schemes, HTC

has a number of other practical advantages: once activated, HTC is a spontaneous, exothermic process, it liberates up to a third of the combustion energy stored in the carbohydrate throughout dehydration. Furthermore, HTC inherently requires wet starting products or biomass, as effective dehydration only occurs in the presence of water, plus the final carbon can be filtered easily from the reaction solution. This way, complicated drying schemes and costly isolation procedures can be avoided. In addition, most of the original carbon stays bound to the final structure. Carbon structures produced by this route—either for deposit or materials use—are therefore the most CO₂-efficient.

Thus, the carbonization of biomass from fast growing plants can be an efficient process for removing atmospheric CO₂. For a negative atmospheric CO₂ balance, the generated carbonaceous materials have to be deposited on a large scale, and potential carbon landfills may lay the foundations for chemical starting materials of the next century. Another quite attractive application with immediate impact is the use of such carbons as water- and ion-binding components to improve soil quality. This is a chemical process that is also found in nature, and 'carbonaceous soil' is the largest active carbon sink on earth. Instead of clearing the rainforest for questionable palm oil production, such a 'carbon-reinforced rainforest' would produce at least 10 times the energy, but stored in carbon, while also being CO₂-benign for the climate and supporting biodiversity at the same time.

In that sense, HTC can be seen as much more than just a technique for making carbon-rich substances.

Read the full Opinion article in issue 6 of New Journal of Chemistry.

Training for Industry 2007

Each year the RSC organises a programme of events dedicated to the advancement of the chemical sciences. With over 30 conferences, one-day symposia and training courses, we offer a range of development and networking opportunities at the cutting edge of science for members and non-members across academia and industry. The remaining training courses organised for 2007 are listed below.

Essential Skills Management Courses	Date and Location	
Fundamentals of Project Management	11–12 September	London
Networking	26 September	London
Presentation Skills	2 October	London
Thinking Strategically	11–12 October	London
Team Working and Co-operation	1–2 November	London
Change Management	19–20 November	London
Time Management and Personal Effectiveness	29 November	London
4 Chemists Courses		
Chemical Biology 4 Chemists	18–20 June	Leeds
Chemistry 4 Chemical Engineers	4–6 September	Leeds
Concepts of Chemical Engineering 4 Chemists	10–12 September	London
Molecular Modelling 4 Chemists	17–19 September	Cardiff
Formulation 4 Chemists	27 September	Bristol

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Health and natural hazards

José A Centeno talks to Kathryn Lees about how Mother Nature affects our health



José A Centeno

José A Centeno is a physical chemist and senior research scientist at the US Armed Forces Institute of Pathology in Washington, D.C., and a member of the editorial board for the *Journal of Environmental Monitoring*. His research in the area of medical geology brings together toxicology, geosciences, environmental medicine and human health.

What is medical geology?

Medical geology is the scientific relationship between natural geological factors and their impact on the development of health problems in humans and animals. The natural environment could be the geochemical composition of rocks or contaminants in water or soil. Medical geology aims to understand how natural geological events, such as dust storms, earthquakes and volcanic eruptions, affect disease and other population health issues.

But medical geology is not a new area: it's been with us for centuries. What is new are the worldwide efforts to improve interactions and research opportunities between environmental scientists, toxicologists, epidemiologists, biologists, earth scientists, public health authorities, pathologists and clinicians to better understand how the natural environment impacts our health.

Recently, the International Medical Geology Association (www.medicalgeology.org) has been launched. This association is a unique opportunity for medical scientists, geoscientists, epidemiologists and toxicologists to share the latest goings-on in medical geology.

What problem poses the greatest challenge for medical geologists?

Earth scientists and medical professionals have long been interested in studies such as water quality, arsenic, mercury and radon. Today, an important research area involves understanding more about how we can map geochemical information and correlate this with environmental epidemiology and disease registry data. If you can overlap local, regional or global geochemical data with disease registry data you could potentially identify what the likelihood is of a particular population developing particular kinds of diseases.

We have disease registry data going back to the 1900s from which we have been able to study, retrospectively, diseases such as the flu pandemic – if you can overlap this health information with geochemical data, it could be a fascinating area.

Is there an undiscovered problem that medical geology could be used to solve in the future?

One of the most challenging problems will be understanding and characterising the environmental and natural factors that contribute to emerging and re-emerging infectious diseases. Infectious diseases are a major cause of human suffering and mortality. Soil-borne and dust-borne human pathogens are potential contributors to the rapid transportation of many infectious agents.

Many regional and local measurements have been taken but, in my opinion, the problem is global. An important step in elucidating the role of natural dust as a contributor of disease is to facilitate cooperation among medical professionals, geologists, climatologists and microbiologists, which will assist in characterising the properties of geogenic dusts, their dispersal and the toxicological pathways of infectious agents they may transport.

So, how much of a threat do dust storms pose?

Research shows that there are ecological and health problems associated with geogenic dust. For example, in the south west of the US, the dust has a peculiar fungus in it which is characteristic of the soil in the region. Geochemical disturbances which cause soil disruption, like earthquakes and human activities such as deforestation and construction, release the fungus. In humans and animals, it can cause respiratory problems, which if undetected, can spread to other parts of the body and cause organ damage.

Recent research by leading organisations has shown that cadmium, mercury, arsenic, lead and chromium could be mobilised in dust. As medical geologists, we need to understand the chemistry and the health implications of these elements.

Can science make a difference to these problems or do solutions depend on government cooperation?

It is evident that governments have to be involved in both the short and long term. They have to be involved in every aspect of medical geology and environmental medicine, and develop programmes to improve integration of these areas.

As a way to initiate integration between earth sciences and public health, the United Nations and the International Union of Geological Sciences have recently launched a global activity – International Year of Planet Earth (IYPE). This aims to demonstrate the societal benefits of geosciences and the need for developing international collaborations. IYPE have selected medical geology as one of the ten topics to focus on in geosciences and public health. As a result, there will be emphasis on outreach programmes and on achieving high quality research.

What message do you have for young scientists?

To look for what is not only academically interesting but for what benefits society as well. Scientists should try and work in areas which benefit the public. That's what makes me passionate about what I do.

Essential elements

Pioneering RSC work on RSS feeds

First came enhanced HTML in electronic journal articles in the first phase of Project Prospect – now further developments means RSC Publishing has become the first ever publisher to enhance RSS feeds with structured subject and compound information.

Journal RSS feeds from RSC Publishing were the first from a scientific publisher to include the graphical abstract. This latest innovation means that RSS feeds from RSC journals will also display ontology terms and compound details, including the 2D image. Users will be able to see at a glance whether the paper is relevant to their research. In addition, hidden coding within the RSS feed allows the metadata to be read by machine – another step towards the ‘semantic web’.

This is the latest development

in Project Prospect, a unique service developed by RSC Publishing with academic partners. Phase one, launched in February 2007, provided



hyperlinked compound information within the HTML, links to terms from the IUPAC Gold Book, plus links from ontology terms to definitions and related papers. Enthusiastically

received by authors and readers, feedback since launch has also resulted in improvements to the Toolbox from which these enhancements are available.

Compound structures are now shown with a single click, navigation within the Toolbox has been enhanced – and the Toolbox itself can be made transparent, avoiding any interference in visibility when viewing the article.

To see for yourself, look out for the Project Prospect icon in RSC journal contents lists that identifies enhanced articles. So far, 500 articles have been published that feature these enhancements.

Find out more at: www.projectprospect.org

Books on chemistry in industry

The RSC is continually aiming to provide both scientists in the lab and those working in industry with reliable and high-quality reference material. Three recently published titles focus on industry-related topics and make excellent reading:

Concepts of Chemical Engineering 4 Chemists (S Simons) – An essential handbook and resource guide for scientists who find themselves working in a chemical engineering-type environment.

Metal-Catalysis in Industrial Organic Processes (G P Chiusoli, P Maitlis) – Covers the major areas of the field, discussing the logic of using catalysis in industrial processes as well as the mechanisms involved.

Article breaks new ground

Research from across four institutions, in two countries, and involving eight researchers published in *Molecular BioSystems* has become the first RSC Open Science paper. Authors from the UK and US collaborated on the work, which involved screening chemical compounds and monitoring changes in tissue during early skeletal development in zebrafish.

The study is an excellent example of research carried out at the chemistry–biology

interface, which is a prime focus of the *Molecular BioSystems* journal.

Zebrafish embryos can be monitored relatively easily outside of the uterus and are transparent, so changes can be clearly observed. They serve as models in the study of Menkes disease in humans, a developmental disease associated with copper metabolism.

In the study, mercapto-pyridine-*N*-oxide (MCP) was found to affect the development

of the notochord, an important tissue involved in early skeletal development. Results suggested that MCP targets the copper-dependent enzyme lysine oxidase.

RSC Open Science gives authors the option of paying a fee in exchange for making their accepted communication, research paper or review article openly available to all via the web.

For more information on RSC Open Science visit: www.rsc.org/openscience



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Freeze-Drying of Pharmaceuticals and Biopharmaceuticals (F Franks) – Describes the technology with particular emphasis on the properties of the material to be processed. For more information see: www.rsc.org/books

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