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IN THIS ISSUE

ISSN 1359–7345 CODEN CHCOFS (27) 3073–3204 (2008)

Cover

See An-Xin Wu, Lyle Isaacs *et al.*, pp. 3133–3135. The picture shows the X-ray structure of a glycoluril derived tetrameric molecular bowl and its structural and functional macroscopic relative. Image reproduced by permission of Neng-Fang She, Xiang-Gao Meng, Meng Gao, An-Xin Wu and Lyle Isaacs from *Chem. Commun.*, 2008, 3133.

Inside cover

See Clarence Charnay *et al.*, pp. 3118–3120. Flower-like profusion of copper-functionalised mesoporous silica nanospheres with perfect pore ordering and high intra-pore metal dispersion. Image reproduced by permission of Gaëlle Derrien, Clarence Charnay, Jerzy Zajac, Deborah J. Jones and Jacques Roziére from *Chem. Commun.*, 2008, 3118.

CHEMICAL SCIENCE

C49

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

July 2008/Volume 5/Issue 7

www.rsc.org/chemicalscience

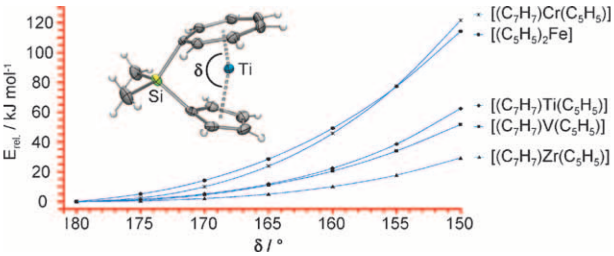
FEATURE ARTICLE

3089

Synthesis and reactivity of functionalized cycloheptatrienyl–cyclopentadienyl sandwich complexes

Matthias Tamm

This *feature article* provides an overview of the synthesis and reactivity of functionalized cycloheptatrienyl–cyclopentadienyl transition metal sandwich complexes of the type [(η^7-C_7H_7)-M(η^5-C_5H_5)] (M = group 4, 5 or 6 metal), which can be used as building blocks for the preparation of metallopolymers and polymetallic complexes.



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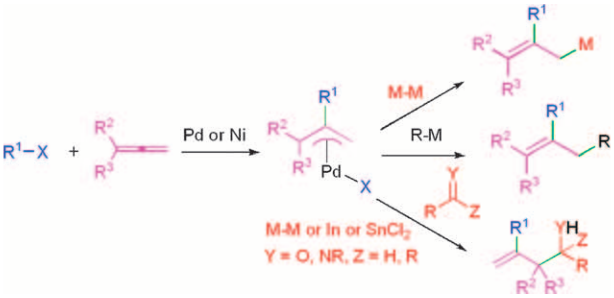
FEATURE ARTICLE

3101

Transition metal-catalyzed three-component coupling of allenes and the related allylation reactions

Masilamani Jeganmohan and Chien-Hong Cheng*

The feature article surveys various transition metal-catalyzed three-component coupling reactions of allenes to give various substituted allyl and vinyl metal reagents and complex organic molecules. The metal reagents or π-allyl–metal intermediates obtained were utilized for the allylation of aldehydes, ketones and imines, producing homoallylic alcohols and amines in a highly regio- and stereoselective manner.



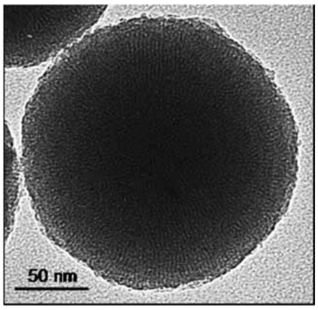
COMMUNICATIONS

3118

Copper-containing monodisperse mesoporous silica nanospheres by a smart one-step approach

Gaëlle Derrien, Clarence Charnay,* Jerzy Zajac,
Deborah J. Jones and Jacques Rozière

Copper-containing mesoporous silica spheres of size in the colloidal range with perfect conservation of pore-ordering, shape and monodispersity and high intra-pore metal dispersion were prepared *via* a new one-step synthesis and functionalisation route.

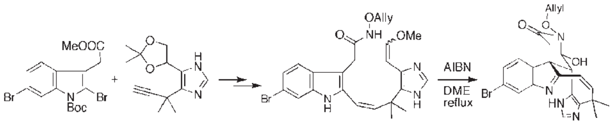


3121

Synthetic studies and biosynthetic speculation on marine alkaloid chartelline

Shigeo Kajii, Toshio Nishikawa* and Minoru Isobe

An important intermediate for chartelline C was synthesized through coupling of dibromoindole acetic acid with alkynylimidazole followed by further functionalization. Attempted cyclization gave an unexpected pentacyclic product, which inspired us to a new biosynthetic hypothesis for chartelline alkaloids.

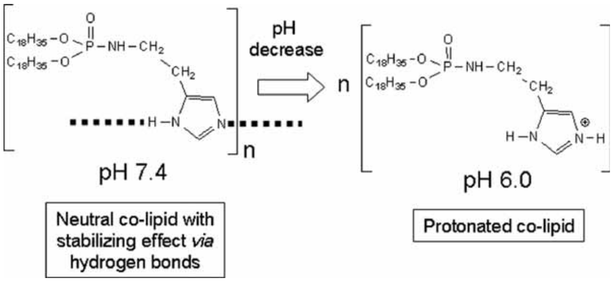


3124

Novel neutral imidazole-lipophosphoramides for transfection assays

Mathieu Mével, Cécile Neveu, Cristine Gonçalves,
Jean-Jacques Yaouanc,* Chantal Pichon,
Paul-Alain Jaffrès and Patrick Midoux*

Two neutral imidazole-lipophosphoramidates have been evaluated as helper lipids in formulations for transfection assays. Depending on their combination, high transfection efficiencies were observed.



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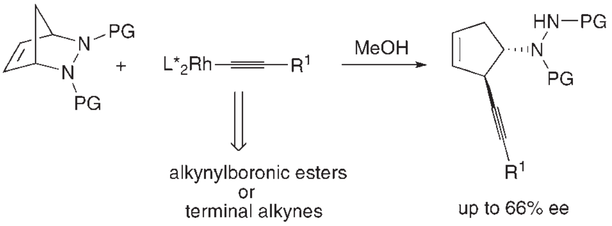
3127

Rhodium-catalyzed enantioselective desymmetrization of bicyclic hydrazines with alkynylboronic esters

Stefano Crotti, Ferruccio Bertolini, Franco Macchia and Mauro Pineschi*

A simple and synthetically useful rhodium-catalyzed alkynylation of bicyclic hydrazines, which constitutes the first successful asymmetric alkynylation of a symmetrical strained alkene, is reported.

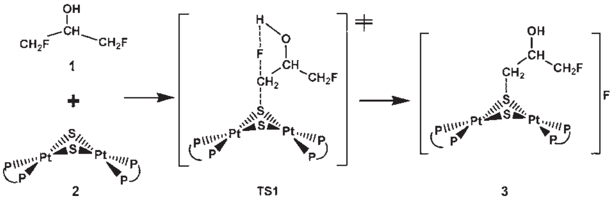


3130

C_{sp^3}–F bond activation by nucleophilic attack of the {Pt_2S_2} core assisted by non-covalent interactions

Ainara Nova, Rubén Mas-Ballesté, Gregori Ujaque, Pilar González-Duarte* and Agustí Lledós*

The high nucleophilicity of the sulfur atoms in [$Pt_2(dppp)_2(\mu\text{-}S)_2$] allows activation of one C–F bond of 1,3-difluoro-2-propanol under rather mild conditions. Experimental and theoretical data indicate that the O–H···F hydrogen bonding established from the alcohol group of the organic substrate is essential for assisting the departure of the fluoride anion.

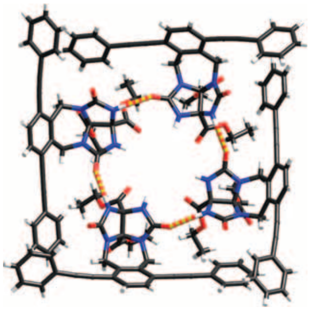


3133

Tetrameric molecular bowl assembled from glycoluril building blocks

Neng-Fang She, Xiang-Gao Meng, Meng Gao, An-Xin Wu* and Lyle Isaacs*

Glycoluril derivative **1**—whose bulky Ph–C≡C– substituents prevent formation of H-bonded tapes—undergoes solvent dependent assembly in the crystal; a tetrameric molecular bowl is formed by $R^4_4(24)$ H-bonding interactions from CH_2Cl_2 whereas DMF results in H-bond dimerization followed by oligomerization *via* C–H···π interactions.

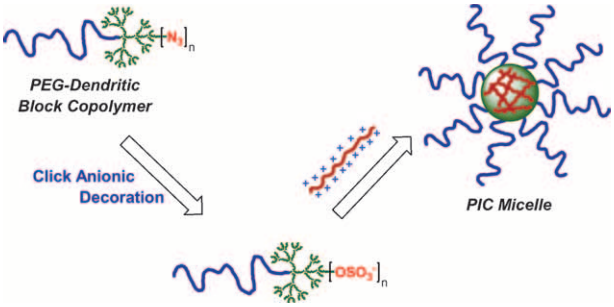


3136

Synthesis and supramolecular assembly of clicked anionic dendritic polymers into polyion complex micelles

Ana Sousa-Herves, Eduardo Fernandez-Megia* and Ricardo Riguera*

Remarkably stable polyion complex micelles with narrow size distribution result from the supramolecular assembly of clicked anionic PEG-dendritic block copolymers with oppositely charged polymers.



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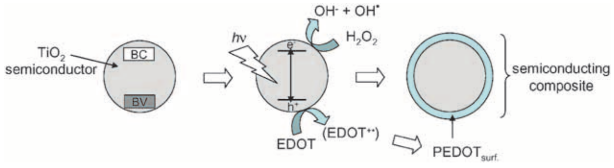
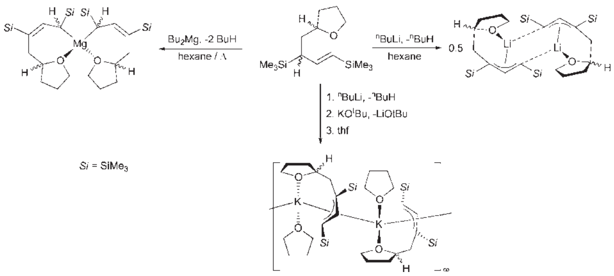


Elaboration of nanohybrid materials by photopolymerisation of 3,4-ethylenedioxythiophene on TiO_2

Fabrice Goubard,* Pierre-Henri Aubert, Kada Boukerma, Emmanuel Pauthe and Claude Chevrot

A method for the elaboration of a heterojunction composed on *n*-type inorganic semiconducting nanoparticles, TiO_2, and a *p*-type organic semiconducting polymer poly(3,4-ethylene dioxythiophene) by UV illumination is described.

3142

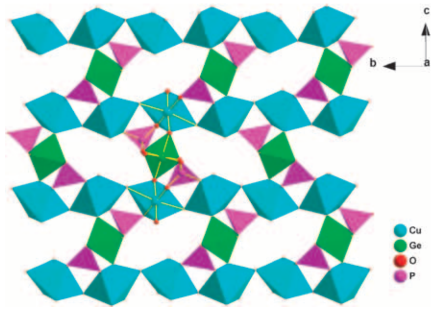


s-Block metal complexes of a bulky, donor-functionalized allyl ligand

Sophia A. Solomon, Christopher A. Muryn and Richard A. Layfield*

A study of the solid-state and solution-phase structures of a homologous series of donor-functionalized allyl s block metal complexes is reported.

3145

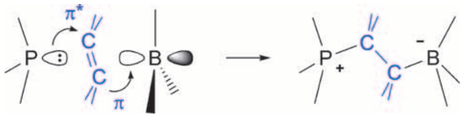


Synthesis of an open-framework copper–germanium phosphate $[Cu(H_2O)_2(OH)]_2Ge(PO_4)_2$

Yan Liu, Xiao-Li Yang, Jun Zhang, Yi-Zhi Li, You Song, Hong-Bin Du* and Xiao-Zeng You

A novel open-framework material $[Cu(H_2O)_2(OH)]_2Ge(PO_4)_2$, which was synthesized by a hydrothermal method, is built of GeO_6, CuO_6 octahedra and PO_4 tetrahedra, and possesses a network of interconnecting six- and eight-membered ring channels.

3148



Concerted attack of frustrated Lewis acid–base pairs on olefinic double bonds: a theoretical study

András Stirling, Andrea Hamza, Tibor András Rokob and Imre Pápai*

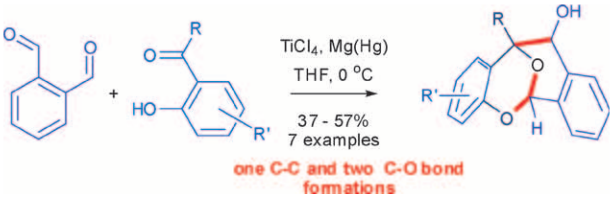
A computational approach reveals cooperative action of the preorganized acidic and basic centers of the frustrated $P(t\text{-}Bu)_3/B(C_6F_5)_3$ Lewis pair on olefinic bonds as the key to the observed regioselective addition reaction.

3151

An expeditious one-step entry to the tetracyclic core of integrastatins

C. V. Ramana,* Challa Nageswara Reddy and Rajesh G. Gonnade

The potential of the pinacol coupling reaction is exemplified by a one-step construction of structurally complex molecules representing the central [6/6/6/6/]-heterocycle of integrastatins.

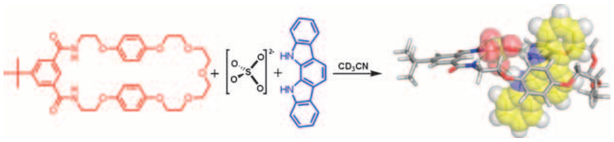


3154

Sulfate anion templation of a neutral pseudorotaxane assembly using an indolocarbazole threading component

Michał J. Chmielewski, Liyun Zhao, Asha Brown, David Curiel, Mark R. Sambrook, Amber L. Thompson, Sergio M. Santos, Vitor Felix, Jason J. Davis* and Paul D. Beer*

The first example of anion templated pseudorotaxane formation between two neutral components in solution and in surface assembled monolayers is described.

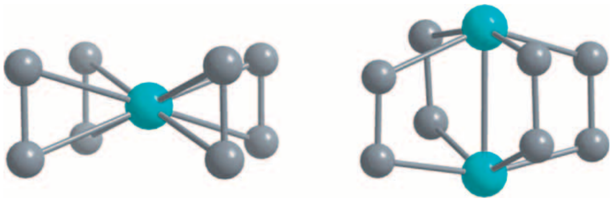


3157

Polycarbide nickel clusters containing interstitial $Ni(\eta^2\text{-}C_2)_4$ and $Ni_2(\mu\text{-}\eta^2\text{-}C_2)_4$ acetylide moieties: mimicking the supersaturated Ni–C solutions preceding the catalytic growth of CNTs with the structures of $[HNi_{25}(C_2)_4(CO)_{32}]^{3-}$ and $[Ni_{22}(C_2)_4(CO)_{28}Cl]^{3-}$

Cristina Femoni, Maria Carmela Iapalucci, Giuliano Longoni and Stefano Zacchini*

C–C Bond formation by forcing carbide atoms of molecular Ni polycarbide clusters to approach by decreasing the Ni/C ratio close or below 3.

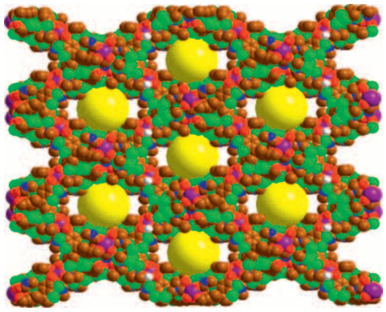


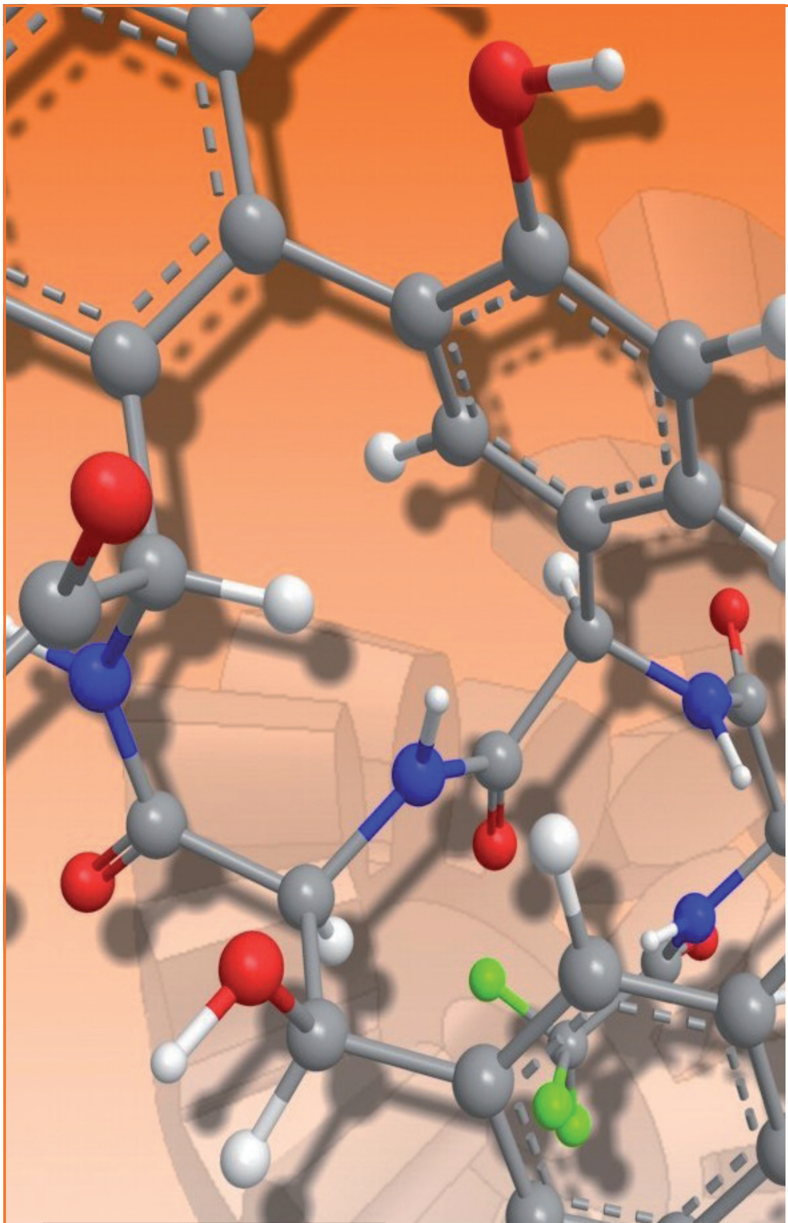
3160

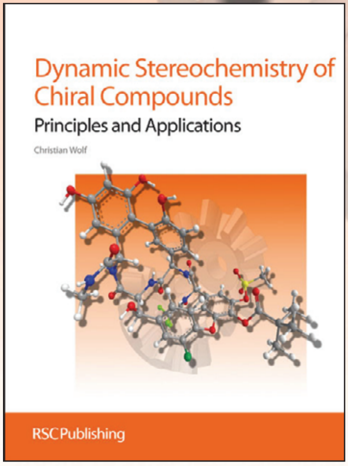
A three-dimensional lanthanide-organic radical open-framework

Nans Roques, Daniel Maspoch, Inhar Imaz, Angela Datcu, Jean-Pascal Sutter,* Concepció Rovira and Jaume Veciana*

A 3-D open-framework coordination polymer which associates a lattice complex *T* topology with large channels and ferromagnetic metal-radical interactions, has been prepared using Tb(III) ions with 3-connecting PTMTC radicals.







Dynamic Stereochemistry of Chiral Compounds
Principles and Applications
Christian Wolf
RSC Publishing

Dynamic Stereochemistry of Chiral Compounds

This book provides an overview of fundamental concepts of asymmetric synthesis highlighting the significance of stereochemical and stereodynamic reaction control. Topics include kinetic resolution (KR), dynamic kinetic resolution (DKR), dynamic kinetic asymmetric transformation (DYKAT), and dynamic thermodynamic resolution (DTR). In-depth discussions of asymmetric synthesis with chiral organolithium compounds, atropisomeric biaryl synthesis, self-regeneration of stereogenicity (SRS), chiral amplification with chiral relays and other commonly used strategies are also provided. Particular emphasis is given to selective introduction, interconversion and translocation of central, axial, planar, and helical chirality.

A systematic coverage of stereochemical principles and stereodynamic properties of chiral compounds guides the reader through the book and establishes a conceptual linkage to asymmetric synthesis, interconversion of stereoisomers, molecular devices that resemble the structure and stereomutations of propellers, bevel gears, switches and motors, and topologically chiral assemblies such as catenanes and rotaxanes. Racemization and diastereomerization reactions of numerous chiral compounds are discussed as well as the principles, scope and compatibility of commonly used analytical techniques.

- More than 550 figures, schemes and tables illustrating mechanisms of numerous asymmetric reactions and stereomutations of chiral compounds
- Technical drawings illustrating the conceptual linkage between macroscopic devices such as turnstiles, ratchets, brakes, bevel gears, propellers or knots and molecular analogs
- More than 3000 references to encourage further reading and facilitate additional literature research
- A comprehensive glossary with stereochemical definitions and terms which facilitate understanding and reinforce learning

This book will be of particular interest to advanced undergraduates, graduates and professionals working and researching in the fields of synthetic organic chemistry and stereochemistry.

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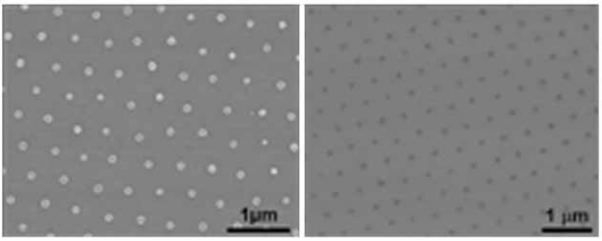
3163

Templated fabrication of sub-100 nm periodic nanostructures

Chih-Hung Sun, Wei-Lun Min and Peng Jiang*

Periodic polymer nanoposts and metal nanohole arrays with tunable size have been fabricated by templating from spin-coated two-dimensional non close-packed colloidal crystal–polymer nanocomposites.

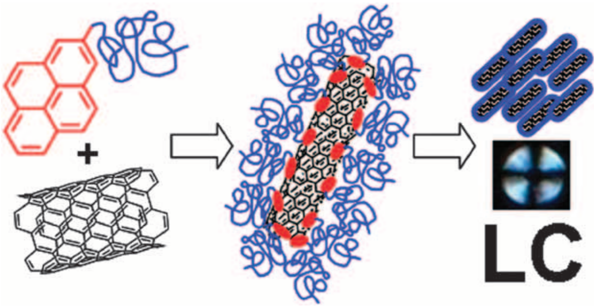


3166

Solubilisation of multi walled carbon nanotubes by α-pyrene functionalised PMMA and their liquid crystalline self-organisation

Stefan Meuer,* Lydia Braun and Rudolf Zentel

Multi walled carbon nanotubes were solubised and disentangled by pyrene functionalized PMMAs. Due to the steric stabilisation, the nanotubes self-organised as a liquid crystalline phase in PMMA and PEG 400 matrices.

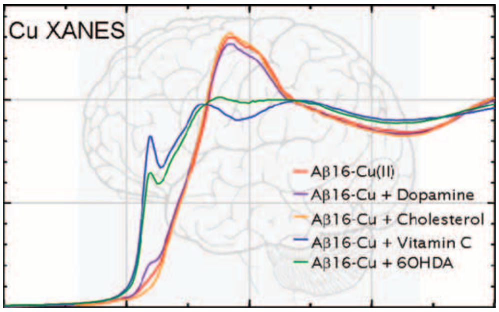


3169

Substrate mediated reduction of copper-amyloid-β complex in Alzheimer's disease

Victor A. Streltsov* and Joseph N. Varghese

X-Ray absorption spectroscopy shows the reduction of $A\beta(1\text{–}16)\text{-Cu}^{2+}$ complex by ascorbate and 6-hydroxydopamine, however dopamine and cholesterol are incapable of reducing of this complex.

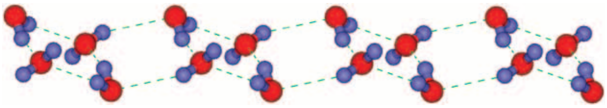


3172

Isolation of an [SNS]Pd(II) pincer with a water ladder and its Suzuki coupling activity in water

Shi-Qiang Bai and T. S. Andy Hor*

A Suzuki-active Pd(II) pincer complex with a hybrid [SNS] ligand-set crystallizes at 223 K with a ladder-like polymer of tetrameric hydrate. Extensive H-bonding interconnects the water cluster, uncoordinated Cl^- and ligand hydrogen in the lattice.




Green Chemistry
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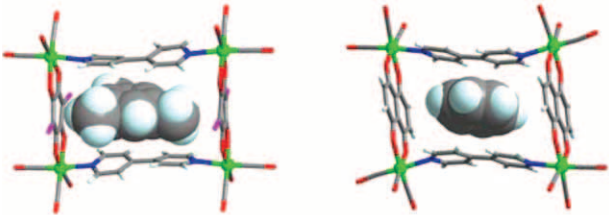
3175

Rhenium-based molecular rectangular boxes with large inner cavity and high shape selectivity towards benzene molecule

Rong-Tang Liao, Woei-Chyuan Yang, P. Thanasekaran, Chen-Chuan Tsai, M. Sathiyendiran, Yen-Hsiang Liu, T. Rajendran, Hsiu-Mei Lin, Tien-Wen Tseng and Kuang-Lieh Lu*

The self-assembly of rhenium-based rectangular boxes with a large inner cavity can be achieved *via* a simple one-step synthetic route. These molecules selectively recognize planar aromatic molecules, benzene in particular.

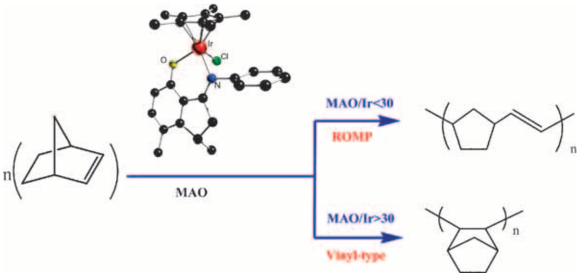


3178

Vinyl and ring-opening metathesis polymerization of norbornene with novel half-sandwich iridium(III) complexes bearing hydroxyindanimine ligands

Xia Meng, Guang-Rong Tang and Guo-Xin Jin*

Half-sandwich iridium complexes were synthesized and employed for norbornene polymerization. Pure ROMP polymer and vinyl-type polymer were obtained depending on the ratio of MAO to Ir.

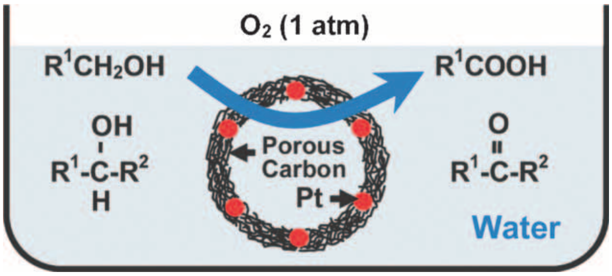


3181

An efficient and reusable carbon-supported platinum catalyst for aerobic oxidation of alcohols in water

Yun Hau Ng, Shigeru Ikeda,* Takashi Harada, Yoshihiro Morita and Michio Matsumura

Platinum nanoparticles embedded in a hollow porous carbon shell prepared by a photocatalytic reaction acted as a reusable catalyst for the aerobic oxidation of alcohols under atmospheric pressure of oxygen in water.

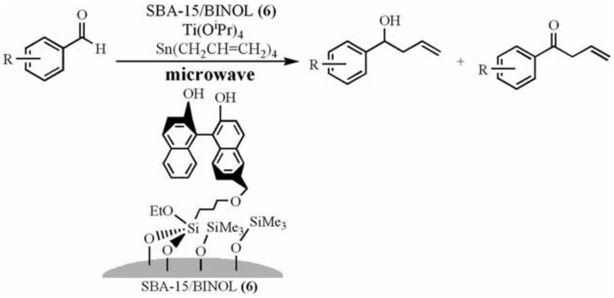


3184

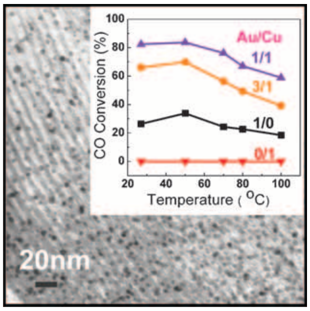
Microwave-assisted catalytic allylation of aldehydes promoted by a mesoporous silica-supported BINOL ligand in solid media

Guohua Liu,* Yan Gao, Xiaoquan Lu, Mouming Liu, Fang Zhang and Hexing Li*

An efficient and operationally simple method for catalytic allylation has been developed and its application in the microwave-assisted catalytic allylation of aldehydes in solid media was investigated.



3187

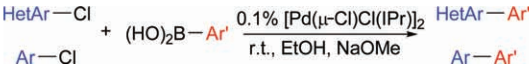


Au–Cu Alloy nanoparticles confined in SBA-15 as a highly efficient catalyst for CO oxidation

Xiaoyan Liu, Aiqin Wang, Xiaodong Wang, Chung-Yuan Mou and Tao Zhang*

Au–Cu Alloy nanoparticles with sizes of ~3 nm were prepared in the confined space of SBA-15 and showed much better performance than monometallic particles in catalyzing CO oxidation even in the rich presence of H_2.

3190

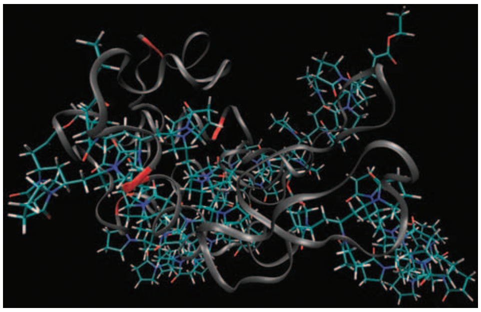


Room-temperature activation of aryl chlorides in Suzuki–Miyaura coupling using a [Pd(μ-Cl)Cl(NHC)]$_2$ complex (NHC = *N*-heterocyclic carbene)

Olivier Diebolt, Pierre Braunstein, Steven P. Nolan and Catherine S. J. Cazin*

A straightforwardly synthesised complex, [Pd(μ-Cl)Cl(NHC)]$_2$ (NHC = bis(2,6-diisopropylphenyl)imidazol-2-ylidene, IPr), has been employed to mediate Suzuki–Miyaura reactions involving aryl chlorides at very low catalyst loadings and at room temperature.

3193

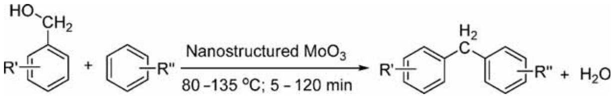


Polymer–protein conjugates from ω-aldehyde endfunctional poly(*N*-vinylpyrrolidone) synthesised *via* xanthate-mediated living radical polymerisation

Gwenaelle Pound,* Jean M. McKenzie,* Ronald F. M. Lange* and Bert Klumperman*

Aldehyde ω-endfunctional poly(*N*-vinylpyrrolidone) was synthesised *via* quantitative conversion of a xanthate endfunctional precursor obtained *via* RAFT-mediated polymerisation.

3196



Nanostructured molybdenum oxides and their catalytic performance in the alkylation of arenes

Feng Wang* and Wataru Ueda*

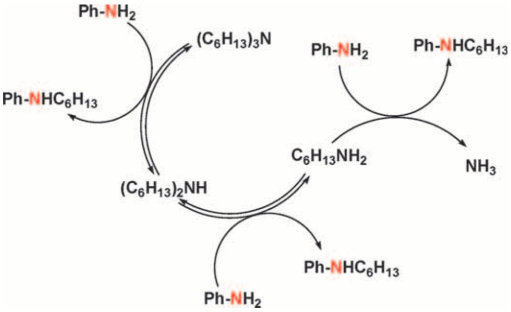
Nanostructured MoO_3 shows excellent catalytic performance in the benzylation of a broad range of arenes with various substituted benzyl alcohols as alkylating agents. In most reactions, the conversion of the alkylating agent was >99%, with >99% selectivity for the mono-benzylated product.

3199

N-Dealkylation of aliphatic amines and selective synthesis of monoalkylated aryl amines

Dirk Hollmann, Sebastian Bähn, Annegret Tillack and Matthias Beller*

Highly selective alkyl transfer processes of mono-, di- and trialkyl amines in the presence of the Shvo catalyst have been realized. In addition, a general method for *N*-alkylation of aniline with di- and trialkyl amines is presented.



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Chemical Science

Single DNA strands manoeuvred using miniaturised hooks and bobbins

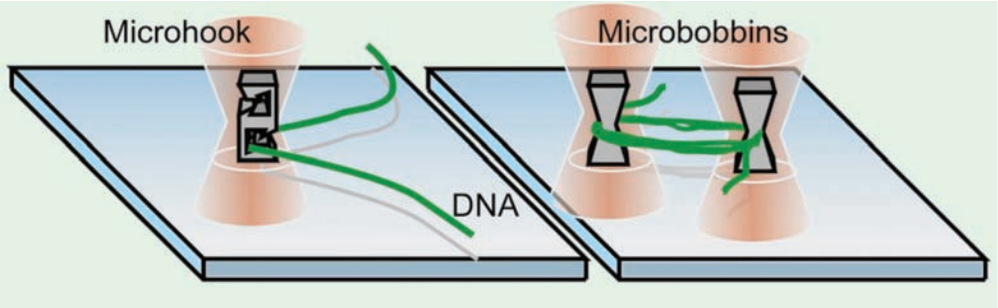
Miniaturised sewing machines

Single threads of DNA can now be manipulated using miniaturised hooks and bobbins, thanks to Japanese scientists.

Taking their inspiration from sewing, Kyohei Terao from Kyoto University and colleagues designed these laser-directed microdevices to pick up and manoeuvre giant individual molecules of DNA.

Single molecule analysis of DNA is limited by the difficulty of stretching out and handling these long molecules – eukaryotic DNA can range from millimetres to centimetres. A giant DNA molecule is very fragile, explains Terao, so to catch it and manipulate it without breaking it is a challenge.

Thinking of a strand of DNA as a piece of sewing thread, Terao developed microhooks to pick up the DNA, just like we would use our fingers to pick up thread. When thread is very long it becomes tiresome to manipulate it just with our fingers and instead we wind it around bobbins to make it compact. This is what inspired us to use microbobbins, says Terao.



Optical tweezers – where tightly focused laser beams trap and hold tiny objects – are used to catch and move these microdevices. The z-shaped microhook is directed by the tweezers to pick up a single strand of DNA, and barbs in the openings of the hook prevent the caught DNA unhooking. In the case of bobbins, two focused laser beams are used to revolve one bobbin around the other. The revolving motion winds the DNA molecule between the two bobbins.

This DNA manipulation technique should prove useful in applications such as fluorescence in situ hybridisation (FISH), says Terao. FISH is used to locate a gene on a DNA molecule using fluorescent gene markers, providing useful information for the diagnosis of genetic diseases and analysis of chromosome function. However, DNA has randomly-coiled conformations, which can hamper observations. But when a DNA molecule is manipulated and straightened by microhooks and bobbins, the gene location can be determined easily with high-spatial resolution, says Terao.

Sarah Corcoran

Handling DNA threads without breakage is challenging

Reference

K Terao, M Washizu and H Oana, *Lab Chip*, 2008, DOI: 10.1039/b803753a

In this issue

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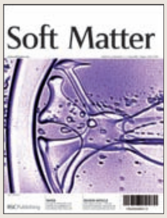


A snapshot of the latest developments from across the chemical sciences

Research highlights

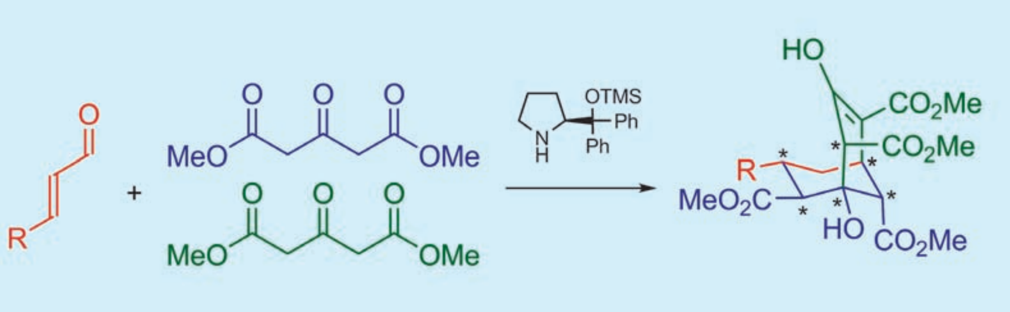
Organocatalysis is taken to a new level

Absolute control of stereochemistry

A one-step synthesis using an organocatalyst to control six chiral centres in a single molecule has been reported by Danish chemists.

It has always been a challenge to get the stereoisomer you want and reactions that control the stereochemistry have become valuable tools for organic chemists. Key to this is organocatalysis – the use of organic molecules as catalysts. These have all the benefits of traditional catalysts, without using expensive (and often toxic) metals.

Karl Anker Jørgensen and colleagues from the University of Aarhus, Denmark, have now controlled the formation of no fewer than six stereocentres in a single reaction, using the power of simple organocatalysts. This, says Jorgensen, equates to the selective synthesis of just 1 out of 64 possible stereoisomers, and 'takes organocatalysis to a new level'. They studied the condensation of three molecules, an unsaturated aldehyde and two tricarbonyl compounds, to give a fused ring system.



Using achiral starting materials and a chiral organocatalyst – an enantiomerically-pure silylated hydroxyamine – just one product was formed, with its absolute stereochemistry controlled at all six centres. Jørgensen says that by testing the limits of our catalysts in this way, 'we learn about catalytic systems and can use this knowledge in the design of new and improved catalysts'. He says the ultimate aim is to mimic nature, which can often create several stereocentres simultaneously.

'This fascinating research', says Ben Feringa from the University of Groningen, The Netherlands, 'again shows the power of organocatalysis, and might well set the stage for new ways to approach total synthesis in the future'.

David Barden

Only 1 out of 64 possible stereoisomers is made

Reference

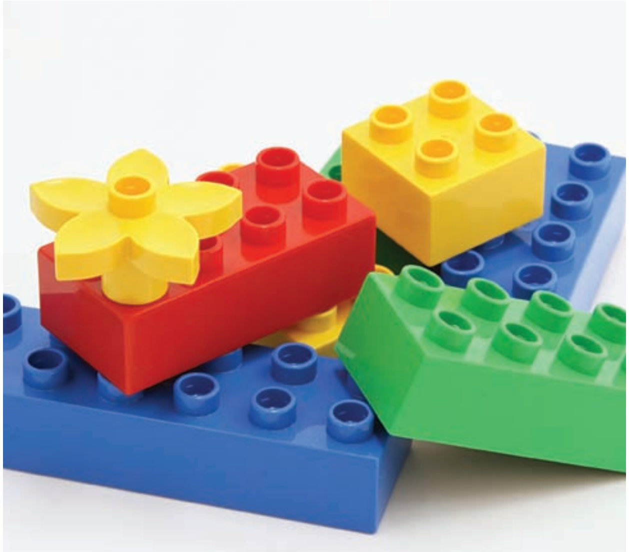
S Bertelsen, R L Johansen and K A Jørgensen, *Chem. Commun.*, 2008, 3016 (DOI: 10.1039/b806418k)

New route for assembling macromolecules has environmental credentials

Molecule Lego turns green

An international team of chemists have developed an environmentally-friendly coupling reaction for connecting together the molecular equivalent of Lego building blocks into large structures.

Janet Scott at Monash University, Clayton, Australia, and colleagues devised a widely applicable schema to prepare open-ended series of macromolecules, while remaining true to the environmentally aware philosophies of green chemistry. She designed various types of building blocks with different structural and chemical characteristics, which can be rationally connected together into specific sequences using aldol-type Claisen–Schmidt condensation reactions.


EGAL / DREAMSTIME

Scott explains that 'male' building blocks contain activated nucleophilic methylene groups, while 'female' blocks have non-enolisable aldehyde groups. Some blocks have just one of these active functional groups and are called terminal, while the connecting blocks that contain two are coined unions. There are also rigid and flexible blocks, allowing the properties of the molecular architectures built from them to be tailored closely to their intended use.

Outlining the benefits and green credentials of her strategy, Scott reports that couplings proceed 'catalytically and in high atom economy with good chemo, regio and stereoselectivity', adding that the reactions' major by-product is water.

Looking towards the future, Scott says she sees many possible applications for macromolecules engineered in this way, including as polymer and peptide linkers, chelating agents, molecular wires and switches.

David Parker

Coupling reaction connects together molecular building blocks

Reference

M A Giarrusso *et al*, *Green Chem.*, 2008, DOI: 10.1039/b802755b

Rough surfaces blamed for aging chocolate's grey coating

Chocolate's sweet secrets

Studying microstructural changes in chocolate could help confectioners stop that seductive shiny surface from turning an unappetising dull grey in poorly stored bars and boxes.

Chemists in Sweden and Canada have used environmental scanning electron microscopy to examine how filled and plain chocolates develop fat bloom – the unappealing dull grey coating that can develop on the surface of hoarded Easter eggs, boxes of pralines and other chocolate treats. Dérick Rousseau at Ryerson University, Toronto, Canada, says that understanding chocolate microstructure could reveal ways to control fat bloom.

Fat bloom forms on chocolate when tiny temperature fluctuations as small as ± 2°C cause cocoa butter crystals to melt and then recrystallise, forming large needle-like structures that scatter light giving a dull appearance. 'Temperature fluctuations are the death knell of chocolate – and once it leaves the factory floor, there's no way for companies to control it,' says Rousseau.

The surface of chocolate close up looks like a mountain range

To get an insight into the way the bloom formed, Rousseau, along with Paul Smith at the Institute for Surface Chemistry in Stockholm, Sweden, studied chocolate structure as it aged. 'We found that the chocolate surface was very heterogeneous – and that bloom crystals only grew from specific points on the surface,' says Rousseau. So controlling chocolate making to minimise surface imperfections could be a good way to control bloom, he adds.

The team also looked at filled chocolates, and found that liquid-state fat from the filling migrates through the chocolate, accelerating bloom formation and ultimately making the chocolate very soft.

'This is certainly a problem that haunts the whole chocolate industry,' says Nigel Sanders, senior research scientist at Cadbury in Toronto, Canada, who adds that Rousseau's study confirms 'quite a few of the mishmash of ideas others have had regarding bloom formation'.

'As an industry, we haven't got to the bottom of what tools we have to stop bloom formation from happening,' adds Sanders. 'Companies as large as Cadbury do their own research – but that never gets published, so it's nice to see an academic study that helps the whole industry, and isn't just for the big boys.'

James Mitchell Crow

Reference

D Rousseau and P Smith, *Soft Matter*, 2008, DOI: 10.1039/b718066g

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Easy separation method aids study of arthritis

A joint effort

A new way of isolating calcium phosphate crystals will greatly benefit research into the diagnosis and treatment of arthritis, say scientists from Ireland.

Patients with conditions such as arthritis and pseudogout often have calcium phosphate crystals in the fluid of their joints. Isolating and detecting these crystals is no easy matter, making it difficult to understand the role they play in the diseases.

Now Gillian McMahon from Dublin City University and colleagues have solved the problem, using superparamagnetic beads that have bisphosphonate groups attached. When the beads are mixed into a sample of joint fluid, the bisphosphonate groups selectively bind to the calcium phosphate. The beads, and the bound crystals, are then easily removed using a magnet. The researchers say their method is less time consuming, with higher sensitivity, than previous techniques.

Calcium phosphate bound to magnetic beads are easily removed from joint fluid

'We are starting now to examine the crystals present in a bank of patient samples, with a view to diagnosing the type of arthritis the patient has,' says McMahon. 'In particular osteoarthritis is of interest, as it is not known whether the crystals are a cause or an effect in this disease.' To answer this question, the group will carry out a range of studies including monitoring samples from the same patients over an extended period of time.

'This is an original, intriguing approach that deserves continued study,' enthuses Ralph Schumacher, an expert in osteoarthritis and related diseases from the University of Pennsylvania, Philadelphia, US. He adds that further development of the method is necessary to make it capable of quantitating the isolated crystals. 'If there are eventual treatments to decrease crystals you need to be able to quantitate change,' he explains.

McMahon agrees that quantitation is important for studying the progression of crystal deposition over time, and diagnosing the severity of disease. The group are working on combining their isolation method with techniques such as spectrophotometry, which will allow quantitative analysis.

Danièle Gibney

Reference

A Hernandez-Santana *et al*, *Chem. Commun.*, 2008, 2686 (DOI: 10.1039/b801750f)

s-Block catalysts offer a greener route to moisturising lactates

Livening up lactates

Lactides, derived from corn, can be converted into alkyl lactates

A fast, environmentally-friendly route to alkyl lactates – compounds widely used by the cosmetic and food industries – has been derived by Thai scientists.

Khamphee Phomphrai and his team, from Mahidol University, Bangkok, use lithium, potassium and calcium catalysts to make a range of alkyl lactates from lactides in a matter of minutes. The lactides are derived from lactic acid which is made from the fermentation of carbohydrates such as corn – a renewable feedstock.

Lactates are widely used in the cosmetic industry to make moisturisers and skin rejuvenating creams and can be found in food as flavourings and preservatives. Lactates are also used as effective solvents or cleaning agents, replacing more harmful solvents.

Sjoerd Harder, from the Universität Duisburg-Essen in Germany, an expert in s-block metal catalysis, says that Phomphrai's research is 'especially noteworthy not only because it is based on a renewable feedstock but also on account of the environmentally-friendly nature of the metals used as catalysts'.

Currently alkyl lactates are made by reacting lactic acid with excess alcohol at temperatures of 80°C or higher for up to eight hours. Phomphrai's metal catalysed reaction produces pure alkyl lactates in five minutes at significantly lower temperatures, and even works at room temperature in some cases.

Another problem with the current method is the formation of polymer by-products which need to be removed, requiring even more time and energy. Depending on the metal catalyst used, Phomphrai's reaction can be tailored to produce alkyl lactyllactate or alkyl lactate, with the problem of side-products eradicated.

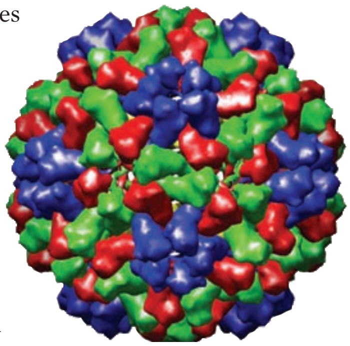
'It remains to be seen, whether alkyl lactacte or lactyllactate production from lactides is economically feasible,' says Harder, but the product selectivity and its easily extendable range might make this method convenient.

'We are now working on using our process for the synthesis of other commercially valuable compounds having the lactic acid functionality,' says Phomphrai.
Ruth Doherty

Reference
K Phomphrai *et al*, *Dalton Trans.*, 2008, 3048 (DOI: 10.1039/b800308d)

Bio-inspired route to titania nanoparticles unveiled

Viruses incubate nanoparticles



Nanoparticle size controlled by interior shape of the virus

US scientists have demonstrated how viruses can be used to make titania nanoparticles of fixed shapes and sizes.

Trevor Douglas and coworkers from Montana State University, Bozeman, found that when the pH is reduced inside a viral capsid containing soluble Ti(IV) salts, insoluble titania (TiO_2) nanoparticles are formed. Importantly the nanoparticle shape and size are controlled by the interior shape of the virus being used – the cowpea chlorotic mottle virus.

Titania nanoparticles with narrow size distributions give reliable homogeneous properties that are useful for a variety of applications including photocatalysts, sensors, semiconductors and pigments.

The titania nanoparticles can be used directly as a photocatalyst whilst still contained within the virus capsid. This is because the cage is transparent to light and simply acts as a template, explains Douglas. In addition, the capsid's protein nature means its surface can be chemically or genetically modified to enable binding to other surfaces if required. The team also expects that in the future different sized protein cages could be used to make particles of different sizes.

Douglas has previously used this route to make tungsten- and vanadium-based minerals inside the same viral capsid. Douglas explains the importance of his method: 'Since we are utilising bio-inspired techniques, we are operating in aqueous media under very mild conditions. This green approach toward synthesis could potentially reduce the use of harsh solvents and high temperatures that are often required in traditional synthesis.'

Rajesh Naik of the Air Force Research Laboratory, Dayton, US, whose research interests include the application of biological methods to making nanoparticles, was interested by the demonstration of the photocatalytic activity: 'Improvement in the materials properties could result in the use of these materials in photovoltaic applications where one might be able to exploit the outer protein coat for functionalisation with dye molecules.' *Christina Hodkinson*

Reference
M T Klem, M Young and T Douglas, *J. Mater. Chem.*, 2008, DOI: 10.1039/b805778h

Interview

Green by name, green by nature

Mike Green talks to Sarah Dixon about the role of green chemistry in industry

Mike Green

Mike Green is the manager of the homogenous and organic synthesis group of the Sasol Technology Research and Development division in Sasolburg, South Africa. He is an editorial board member of *Green Chemistry* and an RSC Council member.

What led you into the field of green chemistry?

It evolved from working in industry. I carried out a lot of work where there were key targets we wanted to achieve to improve yields and selectivities and minimise waste. By definition, those are green objectives. I also worked for BP for nineteen years and I was around in the 1980s when the Venture Research Unit was opened by Don Braben. They were looking at topics that had no immediate commercial application but were areas of science that BP wanted to fund. I remember two of the proposals that came in at the time – one from Ken Seddon on ionic liquids and one from Martyn Poliakoff on supercritical fluids. I got to know them both when they were starting off in the green chemistry area and I got involved in their areas of research. These topics still remain key areas of interest.

Where do you see the field developing in the next few years?

I think it's a question of having the resources to do the research that's needed. There is always the incentive to make processes more effective and efficient and when you couple that with legislation, particularly environmental legislation, I think things naturally come together. It's simply a question of finding the internal resources to put sufficient money into research and development to get to the right solutions.

What are the difficulties faced by industry?

There is a lot of very good work going on in industry, but it's a question of whether the public are willing to pay the additional costs of green chemicals. At the moment, they aren't prepared to pay the extra money and so industry finds itself in a very difficult situation. It has the opportunity to develop green chemicals and find new applications but the customer and market place mean that they struggle to get them off the ground. For example, there have been a number of biodegradable polymers developed over the last few years but getting them into the market place is difficult.

What part of your job do you enjoy the most?

The nice thing about working in industry is that you interact with a lot of different groups, for example customers, technologists, universities and intellectual property people. It is that interaction with people and the way that the technology moves forward as a result of those communications and collaborations that make the job interesting. It's like a big family of people, both within and outside the organisation.

You work a lot with academic groups based in universities. How important do you find this collaboration between academia and industry?

There is so much that needs to be done that companies can't afford to do it all by themselves, either because they don't have the money or simply because they don't have the variety of skills that the new technologies require. Collaboration with academia is a natural consequence. It is important, though, to establish collaborations in which both parties have exactly the same objective because then success is much easier to achieve.

What is the most difficult problem you have had to overcome?

The most difficult thing you face as an industrial researcher is to realise that the research may not meet the required objective. It's the fact that you can only take the chemistry so far and then you have to make a decision whether it's going to be commercially viable or not. At that stage, you have to take a hard decision – if it's not going to meet the company's objective, you have to stop the work. I think a lot of industrial chemists find that difficult but it's something we accept as part of the job. Only 1 in 20 reactions that we look at will ever reach commercialisation. If mentally you can come to terms with that then you can have a very successful career in industry.

If you weren't a scientist, what would you be?

I would have loved to be a cricketer. For many years, I was a member at Old Trafford. I used to sneak out of the lab on sunny days when I was doing my PhD in Manchester to go and watch the cricket. Whether I would have been any good or not is another matter! But one can always dream.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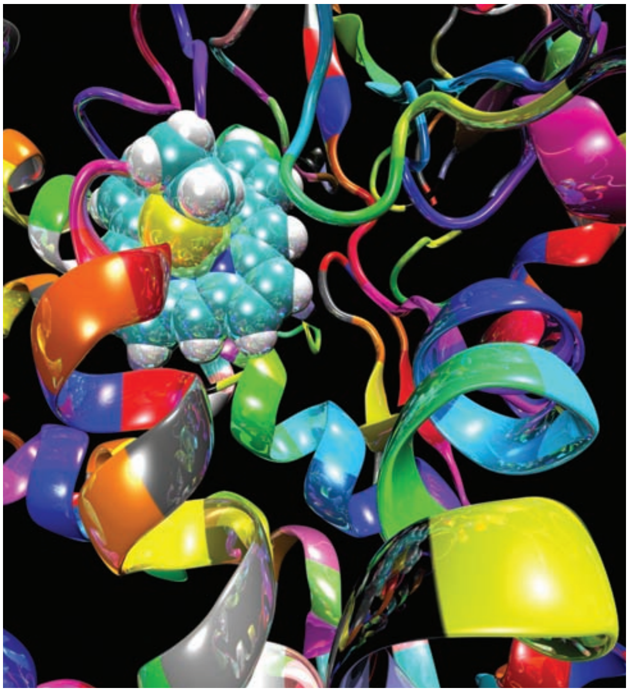
Instant insight

Cooperating catalysts

Mark Davis at California Institute of Technology, Pasadena, US, explains why catalysts need to learn to cooperate in order to compete with nature



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An enzyme's catalytic activity largely occurs within a relatively small region known as the active site. Here the side chains of multiple amino acids interact both with each other and with reacting molecules, with the joint effects of the different interactions having a large influence on the enzyme's catalytic activity and selectivity. The way these interactions work together is characteristic of enzymes, and is known as cooperative catalysis. An ability to mimic this behaviour using synthetic catalyst materials is highly desirable.

Numerous research groups around the world are attempting to harness the power of cooperative catalysis, to make improved solid catalysts that can combine high catalytic activity and selectivity with the easy separation and recycling offered by solid catalysts. These catalysts typically consist of small organic molecules attached to the surface of a solid support such as silica gel, which fixes the location of the catalytic species much in the same way amino acids are spatially localised within enzymes. A short tether (usually consisting of a few carbon atoms) gives local flexibility and motion that can be important for catalysis to occur.

While enzymes have a considerable head start (thanks to million of years of evolution), researchers are beginning to be able to make highly active catalysts in which multiple different functional groups play an important role. Materials functionalised with both acid and thiol groups, for instance, are excellent catalysts for the synthesis of bisphenol A, a key building block to polycarbonate plastics. These solid catalysts exhibit high activity and selectivity without the stench for which thiol-containing molecules are notorious. They can also replace the use of mineral acids, leading to greener processes.

Synthetic catalysts containing carboxylate, amine and imidazole groups can mimic protease enzymes, which use these three functionalities in the catalytic triad. In these catalysts, all three functionalities are necessary for good catalytic results.

Other catalysts containing both acidic and basic groups benefit from the immobilisation of the two otherwise mutually-destructive agents. In solution acids and bases rapidly neutralise, but when both are attached to a solid surface neutralisation can be avoided, and cooperativity between the two types of molecules leads to catalytic activity greater than that achievable through acid or base catalysis alone. These catalysts are shown to be synthetic mimics of aldolase enzymes, which catalyse carbon-carbon bond forming reactions.

Researchers are attempting to develop synthetic catalysts that mimic enzymes activity and selectivity

Rapid advances have also been made in the engineering of polyfunctional surfaces with particular spatial arrangements. The two cooperating functional groups have been arranged in discrete pairs, resulting in synthetic active sites not unlike those in enzymes, and the distance between the two groups can be tuned for a reaction of interest. The next steps will likely involve arranging three (or more) different groups in a single active site, and further refinements of the synthetic methods necessary to position them accurately. The ultimate goal is to engineer an active site on a solid support where the distance between each functional group is optimised for the desired catalysed reaction.

While natural enzymes can exhibit very high activity and selectivity in catalysing chemical reactions, synthetic heterogeneous catalysts can offer other advantages such as solvent versatility, thermal stability, high volumetric productivity and recyclability. By designing future catalysts to utilise cooperativity among multiple functional groups the utility and scope of heterogeneous catalysts will likely be greatly increased.

Read Eric Margelefsky, Ryan Zeidan and Mark Davis' Tutorial Review on 'Cooperative catalysis by silica-supported organic functional groups' in issue 6, 2008 of Chemical Society Reviews

Reference

E L Margelefsky *et al*, *Chem. Soc. Rev.*, 2008, **37**, 1118 (DOI: 10.1039/b710334b)

Essential elements

Expanding the chemical sciences

RSC Publishing is set to increase its journal portfolio from 2009 following announcement of the launch of two new RSC journals. A press release on June 12th confirmed that two interdisciplinary titles, *Integrative Biology* and *Metallomics*, will both publish their first issue in January 2009.

Integrative Biology will provide a unique venue for research that leads to a greater understanding of biological processes and mechanisms. A highly interdisciplinary journal, it will focus on quantitative multi-scale biology using enabling technologies and tools to exploit the convergence of biology with physics, chemistry, engineering, imaging and informatics. The editorial board chair for this prestigious new journal will be Mina Bissell from Lawrence Berkeley National Laboratory, US.

Metallomics will cover the research fields related to metals in biological systems and is expected to be the core publication for the emerging metallomics community. Metallomics is receiving great attention as a new frontier of trace elements in biology and is expected to develop as an interdisciplinary science complementary to genomics and proteomics. Joseph Caruso of the University of Cincinnati/Agilent Technologies Metallomics Center of the Americas and a leading player in this emerging field, will chair the editorial board of this timely new journal.

RSC Publishing boasts an accomplished record in launching new products. With *Integrative Biology* and *Metallomics* set to follow in the footsteps of success stories like *Soft Matter* and *Molecular BioSystems*, RSC Publishing again reinforces its position as a world-class scientific publisher.

From launch, the latest issues of both *Integrative Biology* and *Metallomics* will be made freely available to all readers via the website. Free institutional access to all issues of each journal published in 2009 and 2010 will be available following a simple registration process.

Watch www.rsc.org/journals for all the latest news

Taking to the global stage

Scientists wanting to access the latest research linking all aspects of the chemical sciences relating to energy conversion and storage, alternative fuel technologies and environmental science have a new resource at their disposal.

The first Advance Articles for *Energy & Environmental Science*, the latest journal from RSC Publishing, are now available. The publication of the journal's first issue is anticipated shortly, and will be freely available online.

Enthusiastically welcomed by the community, the first issue will demonstrate the range of subjects included in the scope: from new insights into photovoltaics to the synthesis of important new hydrogen storage materials; from exciting new developments in hydrogen production from biomass to global climate change; from artificial photosynthesis to fuel cells; and from environmental catalysis to nanostructured materials for energy applications – they are all included.

Read the first articles on the website: www.rsc.org/ees

Molecules of Murder

This fascinating new book by John Emsley, due for publication in August 2008, is about infamous murderers and famous victims! It includes the stories of people such as Harold Shipman, Alexander Litvinenko, Adelaide Bartlett and Georgi Markov and takes the reader on a journey of discovery into the world of poisons. Few books on poisons analyse these crimes from the viewpoint of the poison itself, and doing so throws a new light on how the murders or attempted murders were carried out and ultimately how the perpetrators were uncovered and brought to justice. *Molecules of Murder* looks at how forensic chemists have developed cunning ways to detect minute traces of dangerous substances, and why some of these poisons are now being researched as possible life-savers!

John Emsley is a great science communicator. His entertaining books have contributed to the advancement of a positive awareness of science. In 2004 John was elected as an honorary member of The Society of Chemical Industry (SCI) in recognition of a lifetime of achievement and contributions to chemistry. He has written numerous popular science books and articles.

For more information on this exciting book and other forthcoming RSC Publishing titles please visit www.rsc.org/books

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