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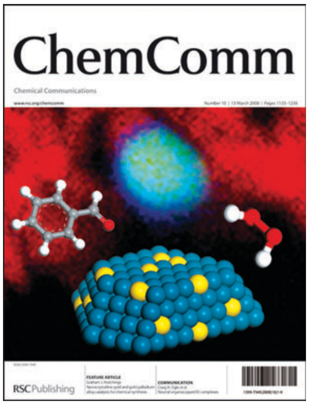
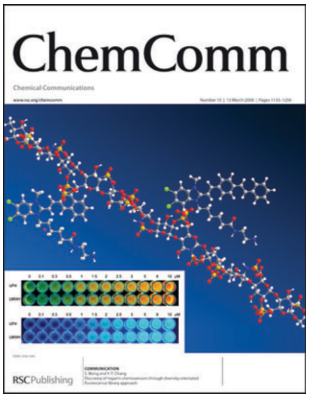
IN THIS ISSUE

ISSN 1359–7345 CODEN CHCOFS (10) 1133–1256 (2008)



Cover
See Graham J. Hutchings, pp. 1148–1164.
STEM-XEDS image of a carbon-supported gold palladium nanoparticle that is very effective for alcohol oxidation and direct H_2O_2 sythesis.
Image reproduced by permission of Graham J. Hutchings from *Chem. Commun.*, 2008, 1148.



Inside cover
See S. Wang and Y.-T. Chang, pp. 1173–1175.
Two novel heparin sensors, Heparin Orange and Heparin Blue were developed by DOFLA, showed remarkable properties and have the potential to monitor heparin levels in clinical plasma samples for point-of-care detection.
Image reproduced by permission of Shenliang Wang and Young-Tae Chang from *Chem. Commun.,* 2008, 1173.

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T17

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Chemical Technology
March 2008/Volume 5/Issue 3
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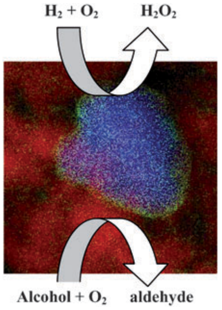
FEATURE ARTICLES

1148

Nanocrystalline gold and gold palladium alloy catalysts for chemical synthesis

Graham J. Hutchings

Gold in nanocrystalline form, either alone or alloyed with palladium displays remarkable activity as a heterogeneous catalyst for a broad range of redox reactions.



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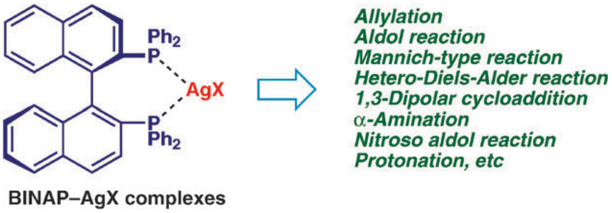
FEATURE ARTICLES

1165

Recent advances in chiral phosphine–silver(I) complex-catalyzed asymmetric reactions

Akira Yanagisawa* and Takayoshi Arai

In this feature article, recent examples of chiral phosphine–silver(I) complex-catalyzed asymmetric reactions such as allylation, aldol reaction, Mannich-type reaction, hetero-Diels–Alder reaction, 1,3-dipolar cycloaddition and nitroso aldol reaction are presented.



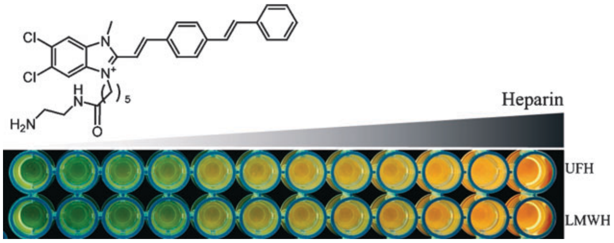
COMMUNICATIONS

1173

Discovery of heparin chemosensors through diversity oriented fluorescence library approach

Shenliang Wang and Young-Tae Chang*

Two novel heparin sensors, Heparin Orange and Heparin Blue, were developed by a diversity oriented fluorescence library approach (DOFLA) from a benzimidazolium library.

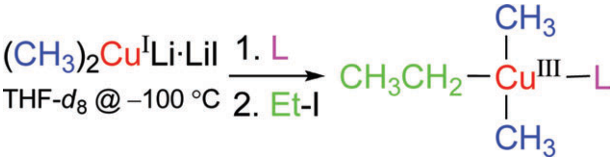


1176

Neutral organocopper(III) complexes

Erika R. Bartholomew, Steven H. Bertz,* Stephen Cope, Donna C. Dorton, Michael Murphy and Craig A. Ogle*

By using rapid injection NMR techniques, neutral organocopper(III) complexes have been prepared for a variety of ligands, L.



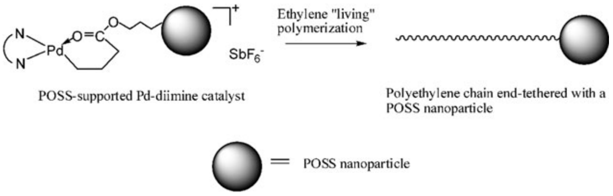
$$(CH_3)_2Cu^{I}Li\cdot LiI \xrightarrow[\text{THF-}d_8\text{ @ }-100\,^{\circ}C \quad 2.\ Et\text{-}I]{1.\ L} CH_3CH_2-\overset{CH_3}{\underset{CH_3}{Cu^{III}}}-L$$

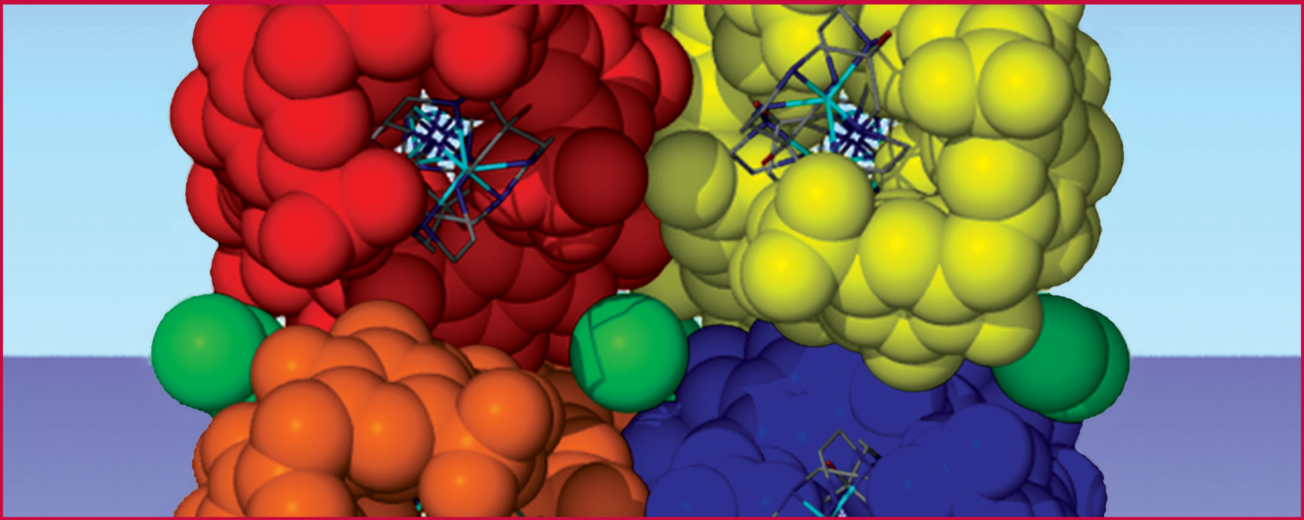
1178

Homogeneous polyhedral oligomeric silsesquioxane (POSS)-supported Pd–diimine complex and synthesis of polyethylenes end-tethered with a POSS nanoparticle *via* ethylene "living" polymerization

Yanwu Zhang and Zhibin Ye*

A homogeneous POSS-supported Pd–diimine complex was synthesized and applied for the unique synthesis of narrow-distributed polyethylene chains containing an end-tethered POSS nanoparticle by ethylene "living" polymerization.





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Professor Jonathan Sessler

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Happy to receive papers on important developments in supramolecular chemistry, Professor Sessler can be contacted via **chemcommun@cm.utexas.edu**

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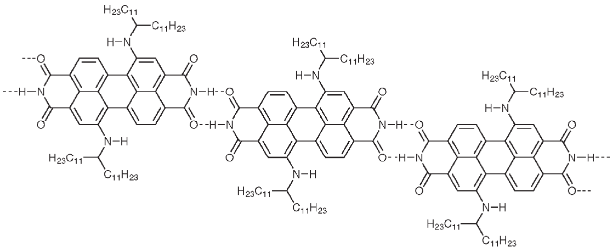
1181

Perylene bisimide J-aggregates with absorption maxima in the NIR

Hao Wang, Theo E. Kaiser, Shinobu Uemura and Frank Würthner*

NIR absorbing J-aggregates with biomimetic features of chlorophyll dye assemblies are obtained upon hydrogen-bond directed self-assembly of a green 1,7-diamino-substituted perylene bisimide dye.

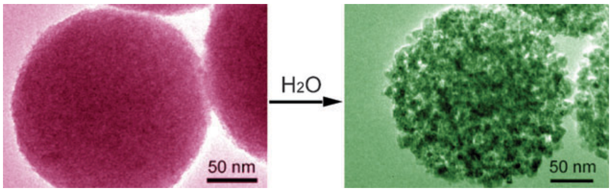


1184

Facile synthesis of nanoporous anatase spheres and their environmental applications

Liang-Shu Zhong, Jin-Song Hu, Li-Jun Wan* and Wei-Guo Song*

Highly nanoporous crystalline TiO_2 (anatase) spheres with an excellent ability in environmental applications have been successfully prepared *via in situ* hydrolysis of titanium glycolate precursor spheres.

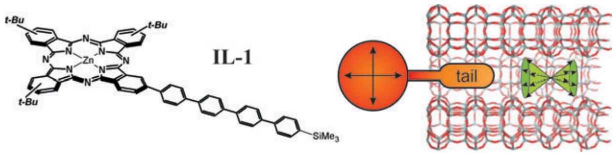


1187

Novel phthalocyanine-based stopcock for zeolite L

Le-Quyenh Dieu, André Devaux, Ismael López-Duarte, M. Victoria Martínez-Díaz, Dominik Brühwiler,* Gion Calzaferri* and Tomás Torres*

We report the first phthalocyanine-based stopcock for selective adsorption to the channel entrances of zeolite L and realisation of a new electronic dipole moment coupling situation.

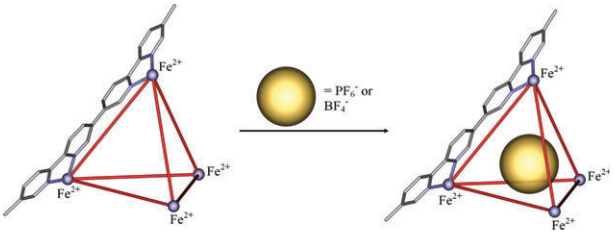


1190

A new Fe^{II} quaterpyridyl M_4L_6 tetrahedron exhibiting selective anion binding

Christopher R. K. Glasson, George V. Meehan,* Jack K. Clegg, Leonard F. Lindoy,* Peter Turner, Martin B. Duriska and Rick Willis

A rigid linear bis-bidentate quaterpyridine undergoes metal directed self-assembly with Fe(II) salts yielding M_4L_6 host–guest complexes; selective anion binding for PF_6^- over BF_4^- is observed.



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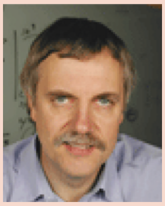

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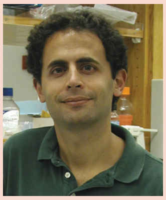

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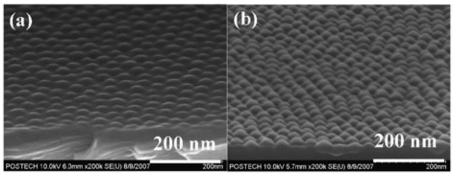
Two-dimensional arrays of luminescent metal-selenide nanoparticle

Savarimuthu Philip Anthony and Jin Kon Kim*

The direct synthesis of CdSe nanoparticles inside the core of PS-P4VP micellar structures has been utilized for the easy fabrication of 2-D CdSe nanoparticle arrays with variable sizes on a solid substrate.

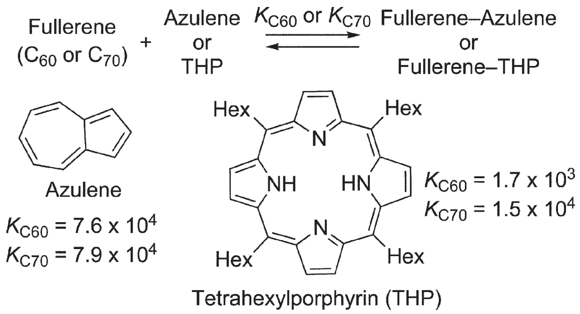


1196

Unexpectedly large binding constants of azulenes with fullerenes

A. F. M. Mustafizur Rahman, Sumanta Bhattacharya, Xiaobin Peng, Takahide Kimura and Naoki Komatsu*

Although azulene is a small hydrocarbon consisting of only 10 carbons, it shows unexpectedly large binding constants to C_{60} and C_{70}, which are larger than those of monoporphyrins and alternately conjugated aromatics.

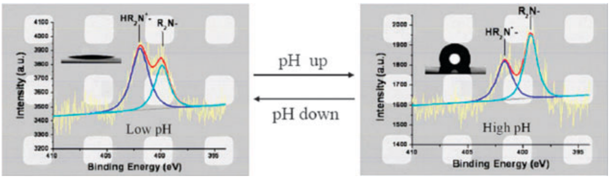


1199

Wettability switching between high hydrophilicity at low pH and high hydrophobicity at high pH on surface based on pH-responsive polymer

Qiaolan Zhang, Fan Xia, Taolei Sun, Wenlong Song, Tianyi Zhao, Mancang Liu and Lei Jiang*

The surface is highly hydrophilic at low pH and highly hydrophobic at high pH and effectively supplements the research on the wettability of solid surfaces based on pH-responsive polymers.

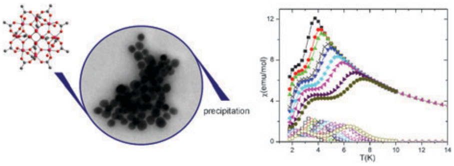


1202

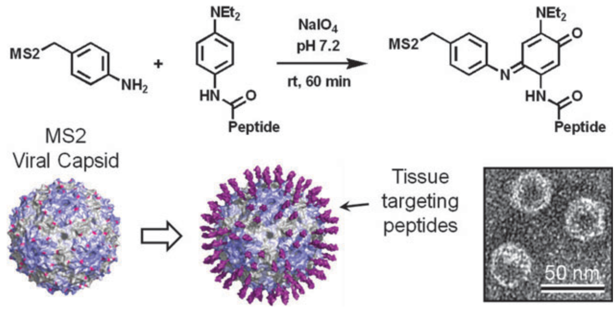
Single-molecule magnet behaviour in metal–organic nanospheres generated by simple precipitation of $Mn_{12}O_{12}$ clusters

Inhar Imaz, Fernando Luis, Chiara Carbonera, Daniel Ruiz-Molina and Daniel Maspoch*

Metal–organic sub-50 nm spherical particles that exhibit single-molecule magnet behaviour have been fabricated by direct precipitation of $Mn_{12}O_{12}$ clusters in a mixture of acetonitrile and toluene.



1205

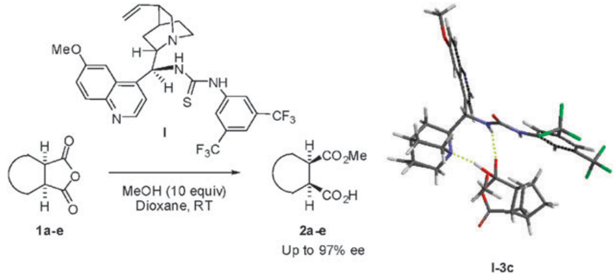


Oxidative coupling of peptides to a virus capsid containing unnatural amino acids

Zachary M. Carrico, Dante W. Romanini, Ryan A. Mehl and Matthew B. Francis*

Cell type-specific targeting peptides have been coupled site-selectively to a virus capsid containing aminophenylalanine residues.

1208

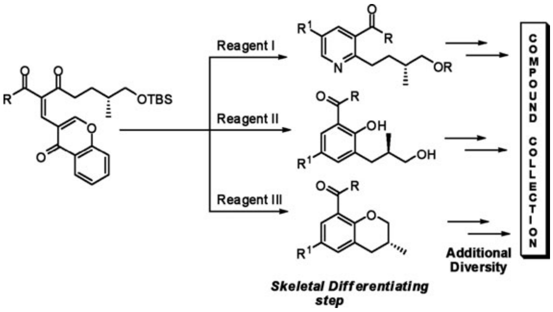


Bifunctional organocatalyst for methanolytic desymmetrization of cyclic anhydrides: increasing enantioselectivity by catalyst dilution

Ho Sik Rho, Sang Ho Oh, Ji Woong Lee, Jin Yong Lee, Jik Chin* and Choong Eui Song*

Highly enantioselective methanolysis was achieved with bifunctional organocatalyst **I**. Unusual concentration and temperature effects on the enantioselectivity were explained in terms of a mechanism involving monomer–oligomer equilibration of the catalyst.

1211

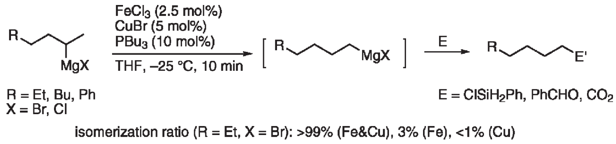


Reagent-controlled domino synthesis of skeletally-diverse compound collections

Herbert Waldmann,* Marc Kühn, Wei Liu and Kamal Kumar*

An efficient reagent-controlled methodology for generating highly substituted diverse scaffolds from a common substrate has been developed.

1214



Fe–Cu cooperative catalysis in the isomerization of alkyl Grignard reagents

Eiji Shirakawa,* Daiji Ikeda, Shigeru Yamaguchi and Tamio Hayashi*

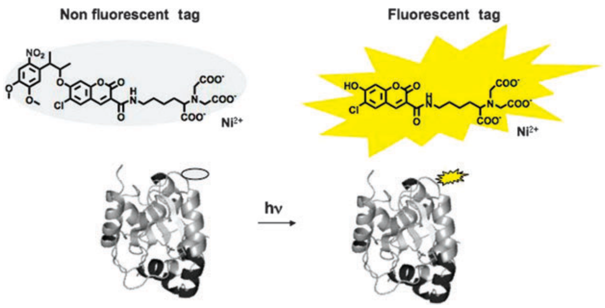
Cooperative catalysis by iron and copper was found to be highly effective in the isomerization of alkyl Grignard reagents to more stable ones, scoring high isomerization ratios (>99%).

1217

Synthesis and photochemical properties of a light-activated fluorophore to label His-tagged proteins

Clélia Orange, Alexandre Specht,* David Puliti, Elias Sakr, Toshiaki Furuta, Barbara Winsor and Maurice Goeldner

We have synthesized an efficient light-activated fluorophore carrying Ni^{2+}–nitriloacetic acid recognition motifs for His-tagged proteins and tested their cell permeation.

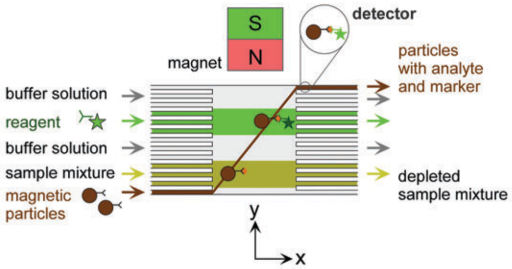


1220

Rapid on-chip multi-step (bio)chemical procedures in continuous flow – manoeuvring particles through co-laminar reagent streams

Sally A. Peyman, Alexander Iles and Nicole Pamme*

We describe a novel and extremely versatile microfluidic platform in which tedious multi-step biochemical processes can be performed in continuous flow within a fraction of the time required for conventional methods.

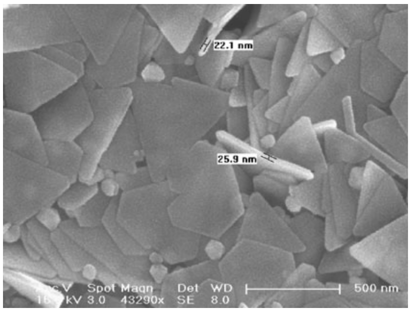


1223

Room-temperature controllable fabrication of silver nanoplates reduced by aniline

Jinling Song, Ying Chu,* Yang Liu, Lili Li and Wendong Sun

Aniline as a suitable reductant can obtain Ag nanoplates from $AgNO_3$ solution at room temperature. The size of Ag nanoplates can be controlled by changing the reaction time and the ratio of $AgNO_3$ and aniline.

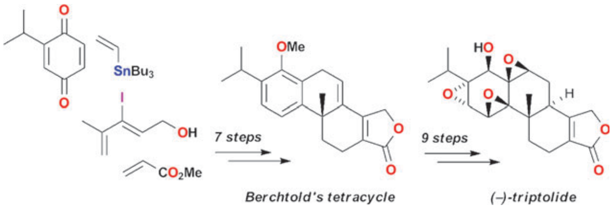


1226

Formal total synthesis of triptolide

Natalie A. Miller, Anthony C. Willis and Michael S. Sherburn*

A new approach to the medicinally-important natural product triptolide is significantly shorter than previous syntheses, highly convergent and avoids the use of protecting groups; key features include two Diels–Alder reactions and a new deoxygenative aromatisation process.



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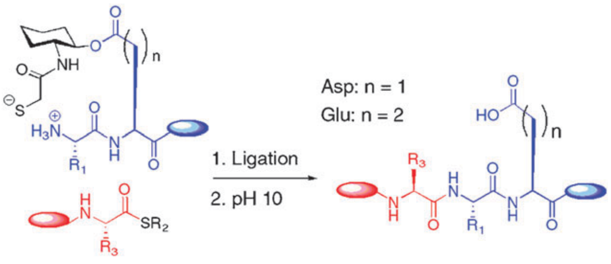
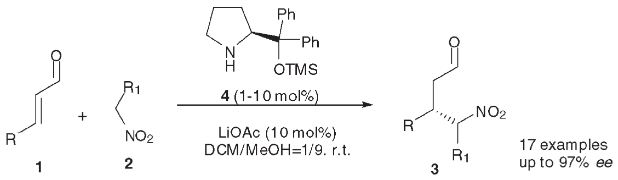


Peptide ligation *via* side-chain auxiliary

Marina-Yamit Lutsky, Natalia Nepomniaschiy and
Ashraf Brik*

A new peptide ligation strategy based on a side-chain auxiliary was developed. The auxiliary is fairly simple and can be removed, without product isolation, under basic conditions.

1232

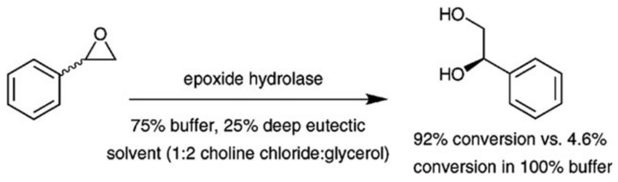


An efficient enantioselective method for asymmetric Michael addition of nitroalkanes to α,β-unsaturated aldehydes

Yongcan Wang, Pengfei Li, Xinmiao Liang,*
Tony Y. Zhang and Jinxing Ye*

The addition of nitroalkanes to α,β-unsaturated aldehydes under the catalysis of (*S*)-2-(diphenyl(trimethylsilyloxy)methyl) pyrrolidine and lithium acetate as additive afforded γ-nitroaldehydes in good yield and up to 97% ee.

1235

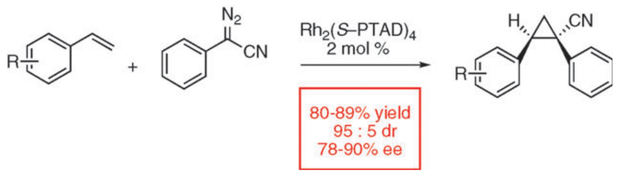


Hydrolase-catalyzed biotransformations in deep eutectic solvents

Johnathan T. Gorke, Friedrich Srienc* and
Romas J. Kazlauskas*

Hydrolases show good catalytic activity in deep eutectic solvents, despite the presence of urea, which can denature enzymes, or alcohols, which can interfere with hydrolase-catalyzed reactions.

1238



Stereoselective construction of nitrile-substituted cyclopropanes

Justin R. Denton, Kevin Cheng and Huw M. L. Davies*

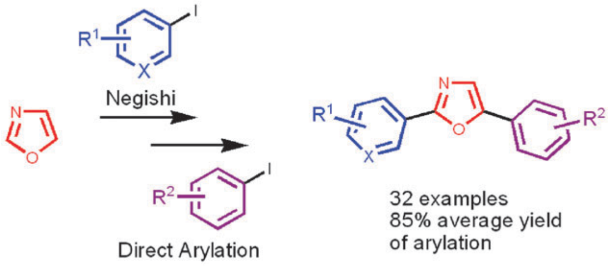
Nitrile-substituted cyclopropanes are readily synthesized in a stereocontrolled fashion from the intermolecular cyclopropanation between 2-diazo-2-phenylacetonitrile and electron-rich olefins, catalyzed by the chiral dirhodium complex, $Rh_2(S\text{-PTAD})_4$.

1241

Direct arylations on water: synthesis of 2,5-disubstituted oxazoles balsoxin and texaline

Stephan A. Ohnmacht, Patrizia Mamone, Andrew J. Culshaw and Michael F. Greaney*

An efficient two-step palladium catalysed synthesis of 2,5-disubstituted oxazoles is reported.

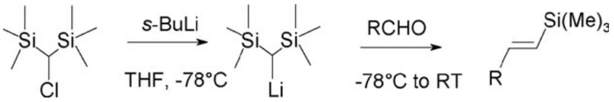


1244

Rapid and efficient entry to vinyl silanes from aldehydes employing a novel metalation–Peterson sequence

James McNulty* and Priyabrata Das

Bis(trimethylsilyl)chloromethane undergoes a rapid and selective metalation with *s*-BuLi, yielding the nucleophilic bis(trimethylsilyl)methyl anion and providing a straightforward general entry to vinyl silanes from aromatic, aliphatic and vinyl aldehydes.

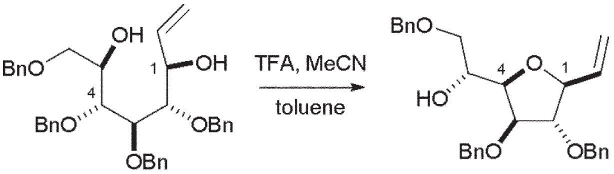


1246

Stereospecific debenzylative cycloetherification of carbohydrate-derived allylic alcohols, ethers and esters to form vinyl *C*-furanosides

Riccardo Cribiù and Ian Cumpstey*

Benzyl ether protected polyhydroxylated alkene compounds containing allylic alcohol, ether or ester functionality undergo a stereospecific cyclisation reaction upon treatment with TFA–acetonitrile–toluene to give tetrahydrofurans.



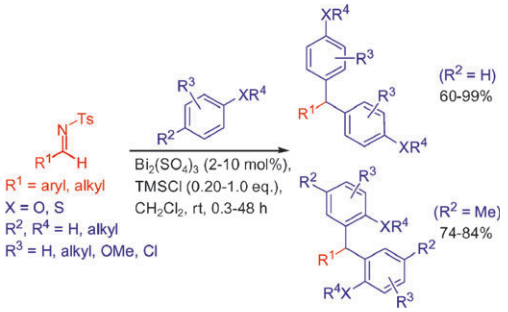
stereospecific: retention at C-4, inversion at C-1

1249

Catalytic selective bis-arylation of imines with anisole, phenol, thioanisole and analogues

Cong-Rong Liu, Man-Bo Li, Cui-Feng Yang and Shi-Kai Tian*

A highly efficient double Friedel–Crafts reaction of *N*-tosyl imines with anisole, phenol, thioanisole and analogues has been developed, for the first time, to produce symmetric diarylmethanes and triarylmethanes.



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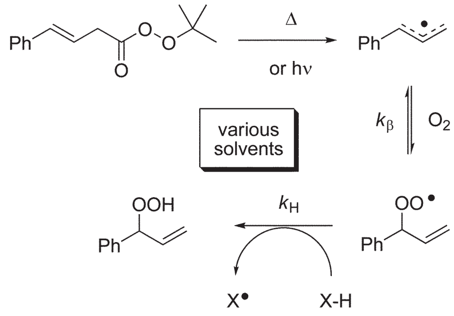

AUTHOR INDEX

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March 2008 / Volume 5 / Issue 3 / ISSN 1744-1560 / CTHEC2 / www.rsc.org/chemicaltechnology

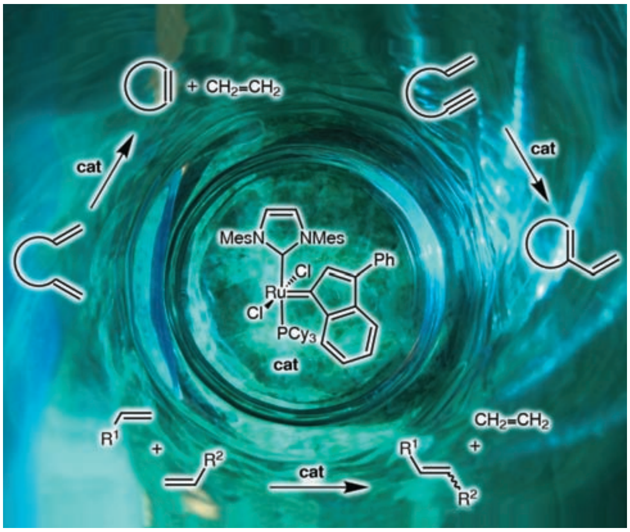
Chemical Technology

Sonication emulsifies organic phase, allowing high turnover

Water-based metathesis

Polish chemists have demonstrated the unprecedented catalytic efficiency of olefin metathesis reactions in a water solution.

Karol Grela and his colleagues from the Polish Academy of Sciences, Warsaw University and Warsaw University of Technology studied the performance of commercially available olefin metathesis catalysts in water. Grela explained: 'Water is an ideal "green" replacement for conventional organic solvents because of its low cost and lack of organic vapours.'



Initial work found that ultrasonication of water-insoluble reactants floating on water formed an emulsion, in which smooth catalytic metathesis took place in up to quantitative yields after addition of water-insoluble, commercially available catalysts. Next, Grela's team extended their experiments in water to more challenging cross-metathesis reactions with electron-deficient substrates. Grela said 'This transformation is extremely rare in aqueous conditions, usually leading to poor conversions and low selectivities.'

Grela's team revealed that cross-metathesis of electron-poor substrates was also possible in water, proceeding with very good yields and selectivities. 'To our knowledge, such reactions represent the first successful example of the high-yielding aqueous cross-metathesis between an alkene and an electron-deficient partner,' he said. 'We speculate that under such conditions the sensitive ruthenium intermediates are "protected" inside the water-insoluble organic droplets, thus allowing higher turnovers.'

Aqueous metathesis is high-yielding and has environmental advantages

Jason Eames, a synthetic organic chemist at the University of Hull, UK, was impressed. 'Aqueous olefin metathesis has significant environmental and process advantages,' he said. 'Much attention has been focused previously on the use of water-soluble metathesis reagents, but Grela has now demonstrated that water-insoluble metathesis reagents can be used successfully through simple sonication.' Further studies leading to improve the ultrasound conditions for metathesis applications are currently underway in Grela's laboratory.

Michael Spencelayh

Reference

Ł Gułajski *et al*, *Green Chem.*, DOI: 10.1039/b719493e

In this issue

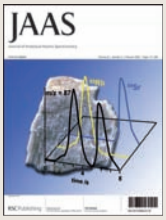
Improving enzymes by error
More efficient and selective version of cytochrome P450 created

Energy saving sieves
Nanosieves that survive extreme conditions could replace distillation

Instant insight: Sensing NO in single cells
Jonathan Sweedler and colleagues describe fluorescent and electrochemical methods for detecting this crucial biomolecule

Interview: A material world
Eugenio Coronado talks to Gavin Armstrong about the molecular design of magnetic materials and current challenges in the field


ChemComm


JAAS


Journal of Materials Chemistry


Lab on a Chip

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

Application highlights

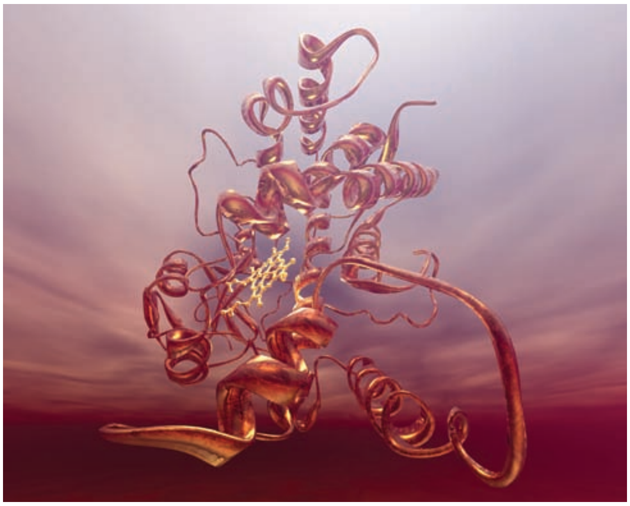
More efficient and selective version of cytochrome P450 created

Improving enzymes by error

Mutant enzymes could make for greener chemical synthesis, say UK scientists.

Luet-Lok Wong's group from the University of Oxford have grown variant forms of a cytochrome P450 monooxygenase (P450) enzyme that can oxidise hydrocarbons under ambient conditions with greatly improved efficiency and selectivity over the natural enzyme.

'Biocatalysis is an increasingly important branch of chemical synthesis which avoids the use of high temperatures and pressures and is typically pollution-free,' explained Wong. P450 enzymes are metabolic enzymes found in all types of life. They typically catalyse the insertion of an oxygen atom into a C–H bond. 'Potential applications for the new variants being explored include the production of fine chemicals, flavours and fragrances, the degradation of environmental contaminants such as dioxins, and the preparation of drug metabolites from pharmaceuticals,' said Wong.



ANNA TANCZOS / WWW.TANCZOS.CO.UK

'Error-prone' PCR was used to create an artificial cytochrome P450

Working with the soil bacterium *Bacillus megaterium*, Wong's team used a variation of the PCR (polymerase chain reaction) technique to copy the DNA from which the enzyme is made. Using an 'error-prone' PCR introduced changes in the DNA to create several new artificial P450 enzymes. A simple screening test – can this variant turn indole into indigo? – identified promising variants. The team went on to pick out variants which efficiently oxidised naphthalene, propylbenzene and pentane.

'It is intriguing that preselection with one small substrate, indole, allowed isolation of efficient catalysts for several different reactions. This seems to contradict the accepted principle that "you get what you screen for",' said Elizabeth Gillam, an expert on P450 enzymes from the University of Queensland, Brisbane, Australia.

Wong accepts that there is a way to go before commercial biosynthesis is practical. 'Scaling up P450 reactions is the greatest single challenge. Control over the regio- and stereo-selectivity of oxidation also needs to be considerably enhanced before chemicals can be manipulated to order,' he said.

Clare Boothby

Reference

C J C Whitehouse *et al*, *Chem. Commun.*, 2008, 966 (DOI: 10.1039/b718124h)

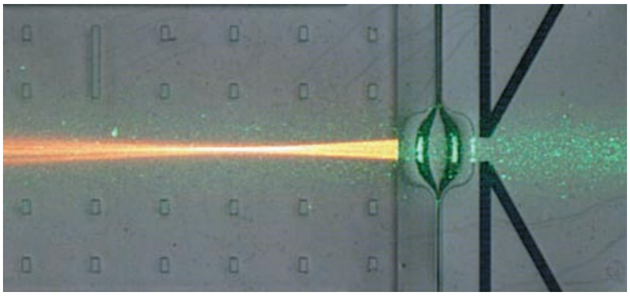
Smooth fluid interface means no polishing is required

Liquid lenses

Liquid lenses that can fit in a microchip are now possible, thanks to researchers in the US.

Sindy Tang and Claudiu Stan in George Whitesides' group at the University of Harvard, Cambridge, US, explained why they chose liquids: 'Conventional solid optical components are too bulky to be placed directly on a chip.'

The role of a lens is to focus light. They have achieved this with streams of liquid flowing in a microfluidic channel. 'We can vary the shape, and therefore the focus, of the lens by changing the flow of the liquids inside the microchannels,' said Tang. 'Liquids have some unique properties that are useful for optics. The interface between the two liquids in microfluidic systems is intrinsically smooth. Fluidic optical compounds, therefore, do not require the polishing that solid optical parts need.'



The focus can be altered simply by changing the flow of liquids

Tang explained their motivation: 'An example of an application is a microfluidic flow cytometer, where localized excitation of fluorescent labels in cells or beads is essential in counting them accurately.'

Albert van den Berg, a microfluidics expert at the University of Twente, the Netherlands, said the work is 'an original and creative way to make an optical lens', and that it had the advantage of relatively simple construction and being able to be integrated into a lab-on-a-chip system. He cautioned that it may not have the optical precision of other, already commercialized, technologies: 'Electrowetting-based lenses are much faster and have better optical quality.'

Tang said they are building a 'toolbox' of optical components. 'We have developed waveguides and splitters. We are now working on prisms and sensors based on similar principles,' she said. However, Tang noted there are still limitations to what you can fit onto a chip. 'We have made a microfluidic dye laser as an on-chip light source, but it still requires an external laser. It is even harder to integrate a detector directly on a chip.'

Colin Batchelor

Reference

S K Y Tang, C A Stan and G M Whitesides, *Lab Chip*, 2008, **8**, 395 (DOI:10.1039/b717037h)

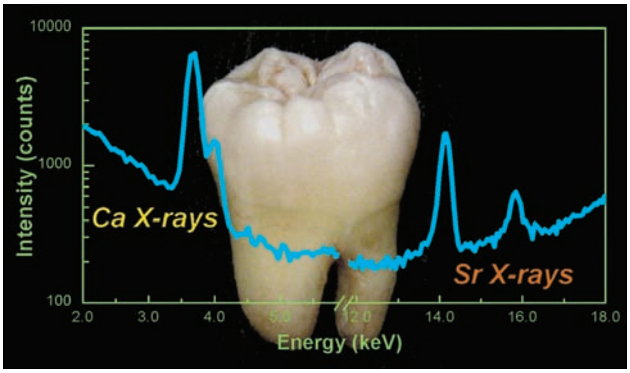
Element linked to rickets and bone cancer detected in the body

Measuring strontium with a smile

Canadian scientists have reported a non-invasive method to measure strontium levels in human teeth.

Eric Da Silva and co-workers at Ryerson University, Toronto, used a ^{125}I excitation source with EDXRF (energy dispersive X-ray fluorescence spectroscopy) to measure characteristic strontium X-rays, which can allow the amount of strontium within the tooth to be calculated.

Measuring strontium levels in the skeleton is desirable as strontium has been linked to bone disorders such as rickets in children and to increased cases of bone cancer in locations affected by nuclear fallout (due to exposure to radioactive ^{90}Sr). Strontium compounds have also been shown to be therapeutic at low levels in the treatment of the bone disorder osteoporosis.



It's easier to examine teeth than bones

Da Silva said 'The subject would just have to smile in front of the excitation source for approximately 20 minutes and absolute strontium concentrations can potentially be extracted,' but he also warned 'for this to be used on humans, a proper study of the dosimetry would be required.'

Previously, measuring strontium was only possible on bones, which had to be removed from the body as the soft tissue covering them significantly weakened the X-ray signals. Teeth are also part of the skeleton, but are easily accessible and not covered by tissue.

The team also carried out a study of dental strontium concentrations in Toronto, an urban area of high ethnic diversity, and the values had a much larger range than those of isolated populations. This may be due to factors such as differences in diet or general bone health between ethnic groups.

Da Silva hopes 'this simple and non-destructive technique can be easily extended to the measurement of other elements in calcified tissues'. *Ian Gray*

Reference

E Da Silva, A Pejović-Milić and D V Heyd, *J. Anal. At. Spectrom.*, 2008, DOI:10.1039/b717029g

Aqueous route to form cyclohexanone in high yields

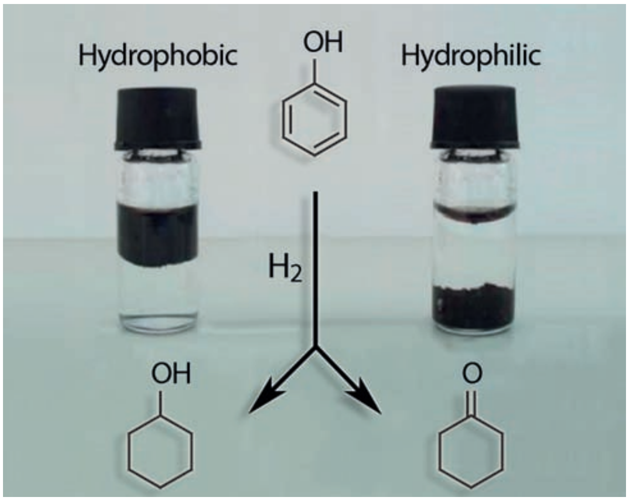
Going for green

An easy and environmentally friendly route to a supported palladium catalyst for highly selective partial hydrogenations has been achieved by European researchers.

Frédéric Goettmann, Maria-Magdalena Titirici and colleagues from the Max Planck Institute of Colloids and Interfaces in Potsdam, Germany, and the Institute of Separative Chemistry in Marcoule, France, made the new catalyst, based on palladium particles supported on hydrophilic carbon, in an aqueous one-pot reaction by a process called hydrothermal carbonisation.

The catalyst exhibits high selectivity towards batch partial hydrogenation of phenolic compounds. The team tested it to form cyclohexanone, one of the main intermediates in the preparation of compounds used to manufacture nylon-6, nylon-6,6 and polyamide resins.

Cyclohexanone is normally prepared from phenol with a supported palladium catalyst in the gas phase, but this is not an easy task in batch reactors, and functional phenolic compounds are difficult to hydrogenate selectively. Only a few studies report successful reactions in a liquid phase, as the selectivity of hydrogenation is poor, leading to difficult separation steps.



The palladium catalyst is fixed to a hydrophilic carbon support

But with Goettmann's new catalyst, water can be used as a solvent, and in one of their reactions, cyclohexanone was obtained in 95% yield with a conversion rate close to 99%.

Goettmann plans to compare the new catalyst with palladium on charcoal catalysts used not only in other hydrogenation reactions, but also in C–C bond forming reactions such as Heck and Sonogashira couplings, functionalisation of allylic derivatives and dehydrogenation reactions. 'Our catalyst opens up the possibility to test all these reactions directly in water and, possibly, to observe alternative selectivities,' he said.

The catalyst, however, is slower than palladium on charcoal, and Goettmann wonders if this would be too much of a drawback for potential applications.
Elinor Richards

Reference

P Makowski *et al*, *Chem. Commun.*, 2008, 999 (DOI: 10.1039/b717928f)

Nanosieves that survive extreme conditions could replace distillation

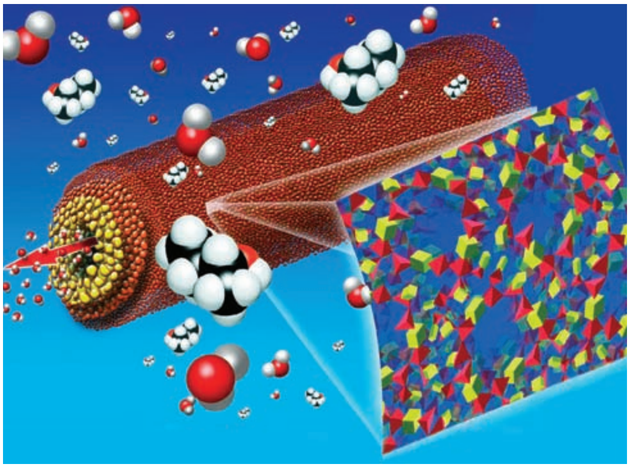
Energy saving sieves

Dutch scientists have made molecular sieves that are stable at high temperatures for over a year. The stability of these nanosieves means that they could replace conventional separation techniques in the petrochemical industry.

Much research has been carried out to develop stable membranes for industrial separation. However, until now the silica-based membranes could not be used in industrial applications because they would not operate both at high temperatures and in the presence of water. This is because the Si–O–Si bonds in the silica are open to attack by water.

Hessel Castricum from the University of Twente and co-workers have replaced some of the Si–O–Si bonds with organic linkers such as $-Si-CH_2-CH_2-Si-$ that do not hydrolyse. The materials produced act as effective sieves even after 18 months at very high operating temperatures.

At the moment, industrial separations involve techniques such as distillation that require large amounts of energy and can have efficiencies as low as 10%. Instead of distilling to obtain a pure product, a nanosieve could be used to filter small molecules, such as water or hydrogen, leaving behind the larger ones. Biofuels, such as bioalcohols, could be dried using this technology. According to Castricum, the vast energy needs of current distillation plants are proving to be a major stumbling block in implementing biofuel technologies.



Organic linkers between silicon atoms prevent water attacking the sieves

Henk Verweij, an expert in ceramic engineering from Ohio State University, Columbus, US, said that the membranes 'could be used for dehydration of industrial alcohol-water mixtures at temperatures up to 150°C'.

Castricum said that his nanosieves have removed the 'serious limitations for industry to become involved in membrane technology and it should be seen as a highly energy-efficient alternative for existing processes.'

'The next challenges are to confirm our findings in an industrial-scale pilot test and to develop materials that withstand even more extreme conditions, such as strong bases and very high temperatures,' he said. *Ruth Doherty*

Reference

H L Castricum *et al, Chem. Commun.*, 2008, 1103 (DOI: 10.1039/b718082a)

Polymers and nanocrystals combine to give off white light

Nanocomposite solid-state lighting

Nanocrystal-based devices produce a higher quality white light than current light sources and present an opportunity for higher efficiencies, say US scientists.

Solid-state lighting is the generation of light through the recombination of positive and negative carriers in a semiconducting material. The technology is synonymous with light-emitting diodes (LEDs) and is likely to replace the inefficient lighting currently used in our businesses and homes. According to the US Department of Energy this could reduce the amount of energy needed for lighting in 2025 by as much as a third and lower carbon emission by 42 million tons per year in the US alone.

However, the quality of the white light used in current LEDs is not adequate for everyday use. Sandra Rosenthal and colleagues at Vanderbilt University, Nashville, approached this problem by developing a thermoplastic fluorocarbon polymer to encapsulate white-light-emitting nanocrystals. 'This is the first major step towards making a viable phosphor LED,' said Michael Schreuder, one of the researchers in Rosenthal's group.

This encapsulation is essential to extend the longevity of these nanophosphors while keeping the high-quality white light, allowing their use in commercial applications. The findings are very significant due the difficulty of finding an encapsulant that not only provides the protection necessary for a lighting device, but also does not reduce the quality of the light emitted from the nanocrystals. Nanocrystals have previously been encapsulated in other polymers, but they typically quench the nanocrystal luminescence or cause the nanocrystals to aggregate. Further, polymers often emit light of their own, which will modify the colour of light emitted.

A fluorescent polymer encapsulates CdSe nanoparticles

Currently, the efficiency of this material is too low for a viable product and is not on par with commercially available LEDs. 'However, there is potential not only to maintain the high-quality white-light, but to surpass the efficiency of present-day devices,' says Schreuder.

In the future, Rosenthal hopes to increase the fluorescence efficiency of the nanocrystals while improving nanocrystal loading in the encapsulant by enhancing the nanocrystal–encapsulant interactions. One particular challenge is to maintain the high-quality white light produced while improving the fluorescence efficiency. *Kathleen Too*

Reference

M A Schreuder *et al, J. Mater. Chem.*, 2008, 970 (DOI: 10.1039/b716803a)

Sensing NO in single cells

Xiaoying Ye, Stanislav Rubakhin and Jonathan Sweedler of the University of Illinois, Urbana, US, describe fluorescent separation and electrochemical methods for detecting a crucial, but tiny, biomolecule

First described in the 1980s, the cell-signalling molecule nitric oxide (NO) has garnered tremendous interest in the scientific community. Investigations have revealed a wide variety of biological functions for NO, which regulates the immune, cardiovascular and central nervous systems. Pharmaceutical treatments, such as Viagra for impotence and BiDil for heart disease, quickly followed these discoveries.

NO, in contrast to most physiologically-active biomolecules, is surprisingly small. Interestingly, most cells are affected by NO, but only a few actually produce it. Nonetheless, the endogenous generation of this simple molecule is complex. Organisms have evolved an elaborate biochemical system that employs several variants of nitric oxide synthase and six cofactors – Ca^{2+}, calmodulin, NADPH, FAD, FMN, and tetrahydrobiopterin. Furthermore, other enzymes, such as argininosuccinate synthase and lyase, are required to complete the NO cycle. This sophisticated system exerts precise control over NO production: the right amount at the right time and in the correct location is pivotal for NO function. Reliable measurements of NO production and the levels of NO-related metabolites are key to understanding NO function in normal and diseased states, and important in both research and clinical settings.

Tissue homogenates are often used in functional assays. Unfortunately, results represent an average from many cells and can therefore be misleading, especially for inherently heterogeneous systems such as the brain. The low abundance of cells that release NO and their scattered distribution make single cell analysis important. Why? Because bulk assays of whole tissues or pooled samples may not accurately reflect the presence of NO and particularly, NO cycle metabolite levels. Furthermore, NO is generated at nanomolar to low micromolar levels and subject to relatively fast deactivation. These factors combine to present significant analytical challenges.

There are a variety of methods for single cell NO measurement. One option, fluorescence-based detection, combines high sensitivity with high spatial resolution. Since NO does not luminesce significantly under typical experimental conditions, a variety of fluorescence indicators have been introduced that interact with NO in a fast, sensitive and selective manner. Advances in nanofabrication and NO-binding compounds, including fluorescent dyes, now allow the creation of biologically inert nanostructures that can be protected from inactivation and targeted to specific cells. Alternatively, proteins that interact with NO can be genetically encoded in cells to become a new class of NO indicator. One example, Piccell, exhibits exceptional sensitivity and real-time monitoring of changes in NO levels in vivo.

ALAMY IMAGES

Understanding the cell-signalling molecule nitric oxide helped scientists to develop drugs such as Viagra

Although fluorescence indicators offer impressive performance, absolute quantitation and the potential effects of the probes themselves on NO levels must also be considered. Electrochemical detection, especially useful for extracellular monitoring, provides another strategy for single cell NO measurement. NO is a highly reactive molecule and can be rapidly oxidised under in vivo conditions. NO-selective electrodes provide fast, quantitative measurement of small fluctuations in NO concentration; they offer significant advantages when monitoring local concentrations of NO.

Another concern during NO measurement is that the complex and dynamic cellular environment can interfere with the measurement process, possibly leading to false-positive or -negative responses. Capillary-scale separations of the intracellular or extracellular environment provide chemically-rich information on NO cofactors and catabolites. Thus, by coupling capillary electrophoresis separations with laser induced fluorescence electrochemical detection, NO measurements from single identified neurons have been accomplished.

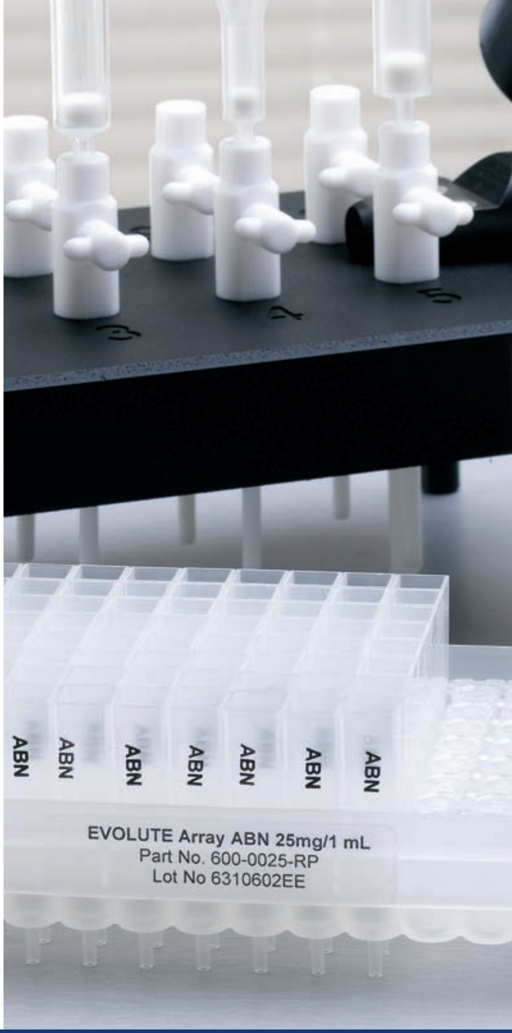
The spectrum of tools available for probing NO at the single cell level has contributed significantly toward revealing the secrets of this surprisingly enigmatic, cell–cell signaling molecule.

Read more in Jonathan Sweedler et al*'s critical review 'Detection of nitric oxide in single cells' in issue 4 of* The Analyst.

Reference
X Ye *et al*, *Analyst*, 2008, DOI: 10.1039/b716174c

Further reading

A R Butler and R Nicholson, *Life, death and nitric oxide*, RSC Publishing (2003) (ISBN 9780854046867)



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Interview

A material world

Eugenio Coronado talks to Gavin Armstrong about the molecular design of magnetic materials and current challenges in the field

Eugenio Coronado

Eugenio Coronado is professor of inorganic chemistry at the University of Valencia, Spain, and is director of the university's institute of molecular science. His interests lie in the area of molecular magnetism, in particular the design and study of multifunctional magnetic materials and molecular nanomagnets.

What attracted you to chemistry?

I have always been interested in science but chemistry allowed me to use a lot of creativity; chemists should create compounds that they or other scientists study. Chemistry also contains a lot of physical principles. This combination of chemical creativity with solid-state physics allows me to construct interesting new systems.

What types of magnetic systems are you working on?

There are several kinds of materials that I find interesting. Magnetic solids based on molecules are one type but they cannot be processed easily. In most cases, you cannot dissolve them as they are coordination polymers. Currently, we are trying to make these crystalline solids easier to work with. If you can solubilise them, this will allow you to use nanotechnology, for example soft lithography, to print the materials onto a surface and thus generate organised magnetic nanostructures. This 'top-down' approach allows you to fabricate nanosystems from crystals.

The other possibility is to start from molecules and functionalise them to make them suitable for creating a nanostructure. This is the 'bottom-up' approach. Organising these molecules in one, two or three dimensions is a challenge because it cannot be fully controlled. They can self-assemble spontaneously to give rise to a certain organisation. However, there is a relationship between the functionalisation of the molecules and the structure of the final materials you obtain, so if you design your system well, you can get the system you want.

You can also impose organisation on a molecular system, playing, for example, with the nature of the surface on which the molecules are deposited. The supramolecular interactions of the molecules that dictate the self-assembly are now not the only important ones - you also have the interactions with the surface. We are just starting out with these systems. They are not very well known and that attracted me to work in this area.

You are interested in multifunctional materials. Could you explain a little about these types of materials?

Developing multifunctional materials is a big challenge in materials science and to obtain them by design is not easy. We are trying to design materials that show ferromagnetism and superconductivity and investigate the new physics behind this type of combination, which very few systems display.

We are also developing switchable magnetic materials. Materials in which, for example, you apply pressure, light or a change in temperature and the material changes its magnetic behaviour. This unique possibility, provided by the molecular chemistry, is very promising in view of the potential applications of these materials in molecular electronics or spintronics.

What is the biggest challenge facing molecular magnetic chemistry?

The biggest challenge is developing systems that are useful at high temperature. Currently, our materials' properties are good but only at low temperatures, from 4 to 20 Kelvin.

A challenge that I expect to be solved within the next few years is measuring the magnetic properties of one single molecule. We can measure the magnetism of nanoparticles but these are 1000 metal atoms in size. For molecular nanomagnets, we are talking about 10 atoms.

What is the secret to being a successful scientist?

A scientist must look around the edges of what they are doing to avoid losing many important discoveries along the way. It is important to focus your activity in the boundary between different fields. The evolution of other fields around you may inspire your research and, with your expertise, you can contribute to the development of these fields.

What would you be if you weren't a scientist?

An artist. I debated whether to become an artist or a scientist but if you are not an excellent painter and a genius of creativity, it is very difficult to succeed to a good level. Being a scientist is more of an intellectual activity but artistic creativity is also essential and certainly contributes to scientific success.

Essential elements

ChemComm makes an impact

The first in a series of *ChemComm* International Symposia was held, with great success, in China in December. The meeting, on Polymers and Polymer Science, featured a mix of speakers from the UK, the Netherlands, the US and China and was held in three different venues: The Institute of Chemistry of the Chinese Academy of Sciences, Beijing; Fudan University, Shanghai; and Sun Yat-Sen University, Guangzhou.

ChemComm editor Sarah Thomas explained the aim of the symposium: 'The purpose of this event was to bring together scientists in a stimulating and friendly environment that will foster collaborations between the researchers and the universities involved. This was successfully achieved with the first symposium, which was met with an overwhelmingly positive response from all who took part.'

ChemComm editorial board member, and speaker at the symposium, David Haddleton was extremely impressed by the event and what it represents for advancing the chemical sciences. 'The RSC is right to focus efforts on China,' he said, 'my impression is that the Chinese are on the whole very receptive of the UK and the RSC.'

ChemComm is the flagship journal of the RSC, publishing some of the most significant work in the chemical sciences. A long and successful history has seen the journal adapt and evolve to meet the changing publishing environment. Today the journal is the fastest at publishing general chemistry communications.

Building on the success of the first symposium, two more *ChemComm* international symposia are planned over the next 18 months, both likely to be held in Asia.

Watch out for details of these events and other exciting ChemComm developments at www.rsc.org/chemcomm

Journal free for all

RSC Publishing is using a new approach to provide access to *Energy & Environmental Science*, the new journal scheduled for launch in summer 2008.

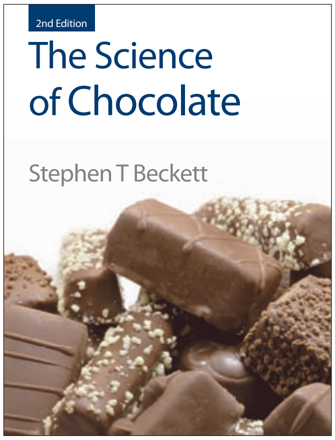
All online content will be available for free during 2008 and 2009. Access, which will be managed by institution and IP address, will be provided following a simple registration process. In addition, the current issue of the journal will be freely available to everyone online, without the need for any registration.

The new approach is in response to feedback from scientists, librarians and other information specialists. The free access will help both scientists and librarians/information specialists to fully evaluate the new journal, before they consider taking out a license or a subscription in 2010.

'We're delighted to launch this new journal in response to feedback from the community,' commented John Haynes, RSC Publishing's editorial director. 'Authors will also benefit from publishing in the journal because the access model is designed to maximise ease of use and global visibility.'

To register for free access at your institution, please complete the online form at www.rsc.org/ees_registration

Hot off the press



First published in 2000 *The Science of Chocolate* has become an international best seller! Widely used in industry and academia throughout the world, *The Science of Chocolate* has even been translated into Spanish and Japanese.

February 2008 sees the arrival of the highly anticipated 2nd edition. Readers will be pleased to hear the 2nd edition has been fully revised and updated: describing the complete chocolate making process from the growing of beans to the sale in the shops. New chapters cover topics such as nutrition, including why chocolate is good for you; how to stop it melting in hot countries; and possible methods of putting bubbles inside a chocolate bar.

A perfect book for chocolate enthusiasts and scientists alike! *For more information visit www.rsc.org/books*

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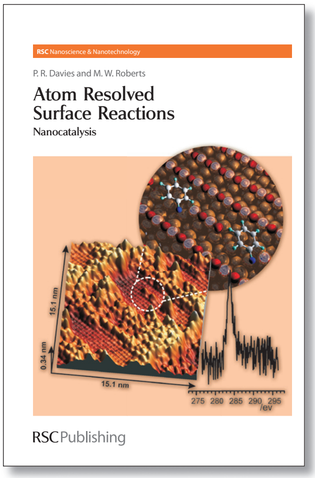
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