

NJC

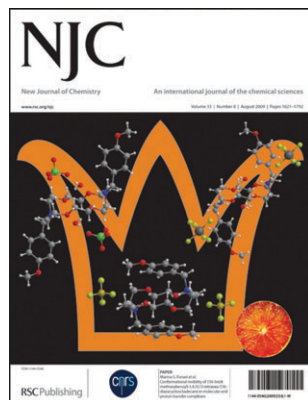
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IN THIS ISSUE

ISSN 1144-0546 CODEN NJCHES 33(8) 1621–1792 (2009)



Cover

See Marina S. Fonari *et al.*, pp. 1646–1656. In acidic medium the *N,N'*-bis(4-methoxybenzyl)-*N,N'*-diaz-18-crown-6 is subjected to essential conformational rearrangements estimated by X-ray and DFT facilities. Permission to reproduce orange slice from <http://www.flickr.com/photos/fdecomite/3375595540/> with license: <http://creativecommons.org/licenses/by/2.0/deed.en> Permission to reproduce king chess symbol from Chess Mark. Freeware. True Type Font by Armando H. Marroquin for diagrams and figurine notation. Modern simple design. Frame with co-ordinates possible. Source: <http://www.enpassant.dk/chess/fonteng.htm>



Inside cover

See Lihua Zhu, Heqing Tang *et al.*, pp. 1673–1679. Molecular imprinted polymer-coated titania photocatalysts bring about a breakthrough to highly efficient and selective photocatalytic removal of toxic organic pollutants. Image reproduced by permission of Xiantao Shen, Lihua Zhu, Hongwei Yu, Heqing Tang, Shushen Liu and Weiying Li from *New J. Chem.*, 2009, **33**, 1673.

HIGHLIGHTS IN CHEMICAL SCIENCE

C57

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Highlights in Chemical Science

August 2009/Volume 6/Issue 8

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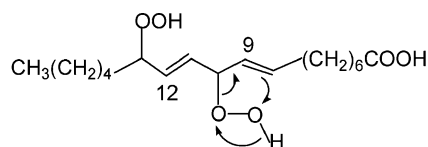
OPINION

1635

A non-radical mechanism for the rearrangement of linoleic acid dihydroperoxides

Arnold Nola Onyango* and Naomichi Baba

A non-radical rearrangement of the 10,13-dihydroperoxide of linoleic acid to the 8,13-dihydroperoxide in the absence of metal ions is proposed.



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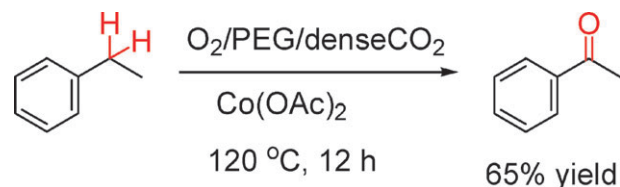
LETTERS

1637

Polyethylene glycol radical-initiated benzylic C–H bond oxygenation in compressed carbon dioxide

Jin-Quan Wang and Liang-Nian He*

The PEG radical originating from the thermal/oxidative degradation of PEG in dense CO₂ was successfully applied to the oxygenation of benzylic hydrocarbons under organic solvent-free conditions. In addition, in our study, dense CO₂ could improve the oxygenation reaction.

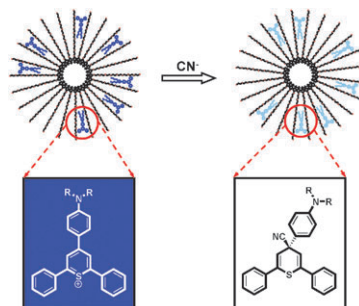


1641

Surfactant-assisted chromogenic sensing of cyanide in water

Tatiana Ábalos, Santiago Royo, Ramón Martínez-Mañez,* Felix Sancenón,* Juan Soto, Ana M. Costero, Salvador Gil and Margarita Parra

Cyanide sensing in pure water has been achieved by using a hydrophobic chromogenic reagent containing a thiopyrylium heterocycle embedded in micellar containers of Triton X-100.



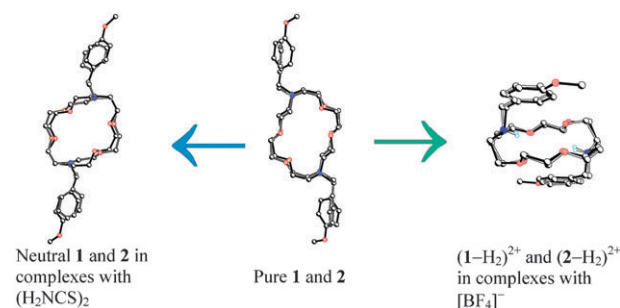
PAPERS

1646

Conformational mobility of 7,16-bis(4-methoxybenzyl)-1,4,10,13-tetraoxa-7,16-diazacyclooctadecane in molecular and proton-transfer complexes: X-ray and DFT studies

Marina S. Fonari,* Eduard V. Ganin, Yurii M. Chumakov, Mark M. Botoshansky, Kinga Suwinska, Stepan S. Basok and Yurii A. Simonov

The conformational mobility of the title compound in molecular and proton-transfer complexes was studied and compared with 7,16-dibenzyl-7,16-diaza-18-crown-6.

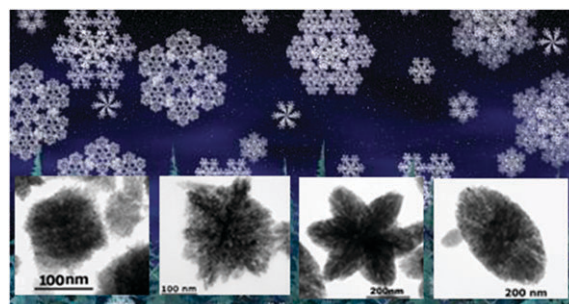


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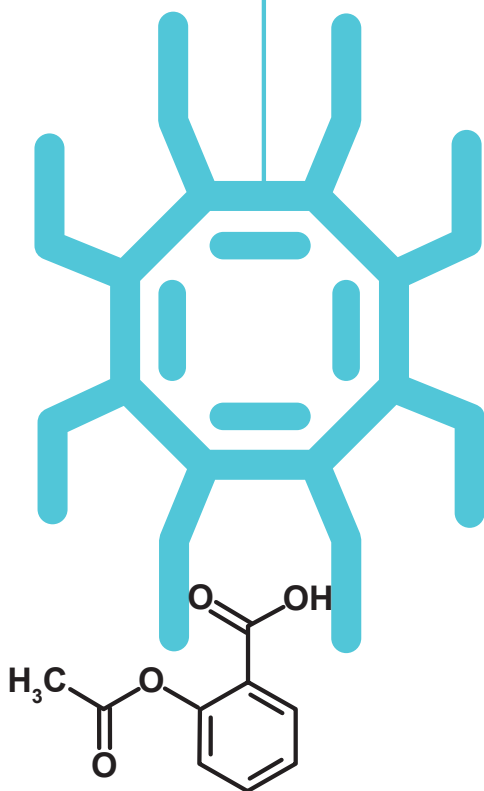
Synthesis of mesoporous LaPO₄ nanostructures with controllable morphologies

Zhanli Chai, Li Gao, Cheng Wang,* Hongjie Zhang, Rongkun Zheng, Paul A. Webley and Huanting Wang*

Mesoporous LaPO₄ and Eu³⁺ or Ce³⁺/Tb³⁺ doped LaPO₄ nanostructures were prepared by a facile solution–precipitation process. The morphologies of these nanostructures were readily tuned by adjusting the composition of the synthesis solution and adding different surfactants.



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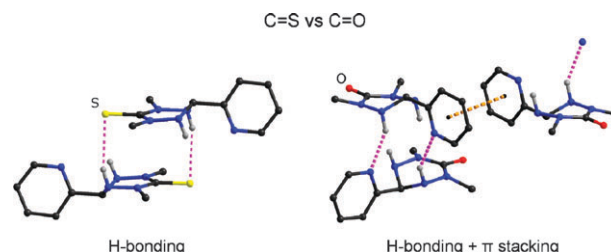
PAPERS

1663

Conformation and crystal packing sensitivity to substitution of thio- and oxo-tetrazane derivatives

Olivier Oms,* Lucie Norel, Lise-Marie Chamoreau, Hélène Rousselière and Cyrille Train*

A comparative crystallographic study of thioxotetrazanes and their oxo-analogues shows noticeably different solid-state organisation related to the variable involvement of C=S and C=O functions as hydrogen-bond acceptors.

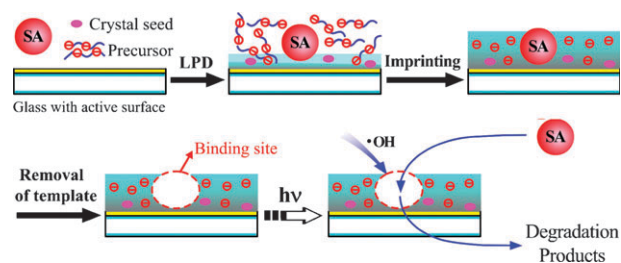


1673

Selective photocatalysis on molecular imprinted TiO_2 thin films prepared *via* an improved liquid phase deposition method

Xiantao Shen, Lihua Zhu,* Hongwei Yu, Heqing Tang,* Shushen Liu and Weiying Li

Molecular imprinted TiO_2 thin films were prepared by using an improved liquid phase deposition method in conjunction with the molecular imprinting technique. The films exhibited special molecular recognition ability, leading to selective adsorption and photodegradation of the target pollutant.

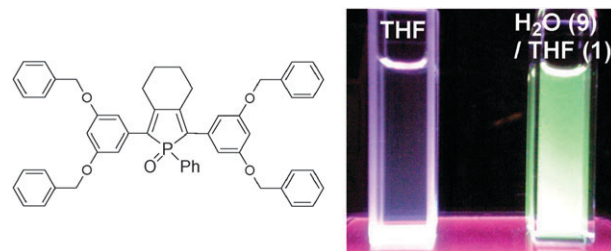


1680

Aggregation-induced emission of dendritic phosphole oxides

Kentarō Shiraishi, Taigo Kashiwabara, Takanobu Sanji* and Masato Tanaka*

Dendritic phosphole oxides display intense emission in the aggregate and solid states, but no emission in solution, because the intramolecular rotational motions of the peripheral dendritic groups linked to the phosphole oxide core are restricted.

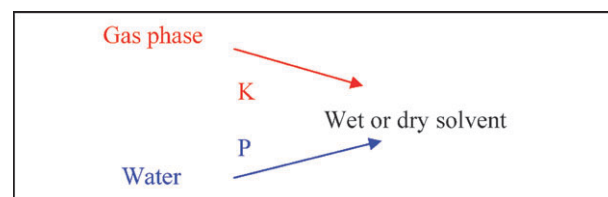


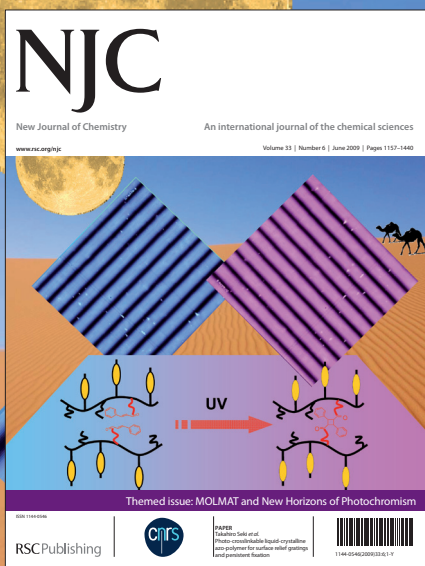
1685

Partition of compounds from water and from air into the wet and dry monohalobenzenes

Michael H. Abraham,* William E. Acree, Jr., Albert J. Leo and David Hoekman

K and P are the same for partition into the wet or dry monohalobenzenes. The main factors that increase K are solute dipolarity/polarisability and solute size. Both K and P are very similar for partition of a given solute into the four halobenzenes.





Themed Issue: MOLMAT and New Horizons of Photochromism

This themed issue comprises articles from two key meetings. Firstly, the 3rd International Symposium on Molecular Materials (MOLMAT) covering topical research within chemical synthesis to solid-state physics, nanotechnology and the theory of molecular materials based on inorganic and organometallic chemistry. This section of the issue is guest edited by Azzedine Bousseksou, Andreas Hauser and Jean-Paul Malrieu.

Secondly, the New Horizons of Photochromism meeting. Guest edited by Kenji Matsuda and Keitaro Nakatani, the 21 articles, (including 6 Letters and a Perspective) are largely written by attendants of the seminar, but also by other researchers involved in this topic.

MOLMAT articles include:

Perspective:

Cyano-bridged coordination polymer nanoparticles

Joulia Larionova, Yannick Guari, Claudio Sangregorio and Christian Guérin

Papers:

Molecular engineering to improve the charge carrier balance in single-layer silole-based OLEDs

Laurent Aubouy, Nolwenn Huby, Lionel Hirsch, Arie van der Lee and Philippe Gerbier

Chemical disorder and spin crossover in a mixed ethanol–2-propanol solvate of Fe^{II} tris(2-picolyamine) dichloride

Dmitry Chernyshov, Brita Vangdal, Karl Wilhelm Törnroos and Hans-Beat Bürgi

Photochromism articles include:

Letter:

The photochromic and self-assembling properties of diarylethenes having chiral amphiphilic chains at the reactive carbon atoms

Takashi Hirose, Masahiro Irie and Kenji Matsuda

Papers:

Photoswitching in diarylethene nanoparticles, a trade-off between bulk solid and solution: towards balanced photochromic and fluorescent properties

Jonathan Piard, Rémi Métivier, Marion Giraud, Anne Léaustic, Pei Yu and Keitaro Nakatani

Photo-crosslinkable liquid-crystalline azo-polymer for surface relief gratings and persistent fixation

Wenhan Li, Shusaku Nagano and Takahiro Seki

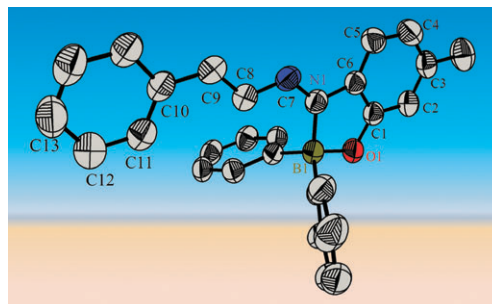
PAPERS

1693

Synthesis and non-linear optical characterization of novel borinate derivatives of cinnamaldehyde

Mario Rodríguez, José Luis Maldonado,*
Gabriel Ramos-Ortiz, Jean François Lamère,
Pascal G. Lacroix,* Norberto Farfán, Ma. Eugenia Ochoa,
Rosa Santillan, Marco Antonio Meneses-Nava,
Oracio Barbosa-García and Keitaro Nakatani

Molecular view of 6-methyl-((*E*)-3-phenylallylidene)-2,3-dihydrobenzo[*d*][1,3,2]oxazaborole (**2d**).

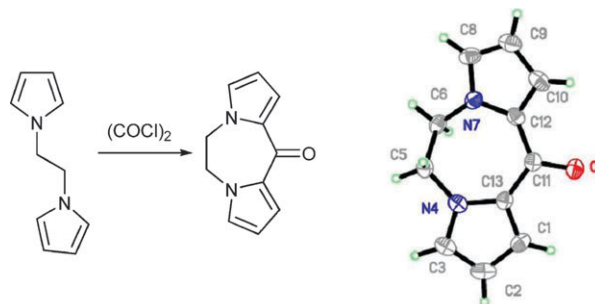


1703

Synthesis and properties of 5,6-dihydrodipyrrolo[1,2-*d*;2',1'-*g*][1,4]diazepin-11-one

Karen A. Johnston and Hamish McNab*

Treatment of 1,2-di(pyrrol-1-yl)ethane with oxalyl chloride gives 5,6-dihydrodipyrrolo[1,2-*d*;2',1'-*g*][1,4]diazepin-11-one which is shown to adopt a half-chair configuration by X-ray crystallography; the diazepinone is unreactive to electrophiles, but can be deoxygenated with lithium aluminium hydride.

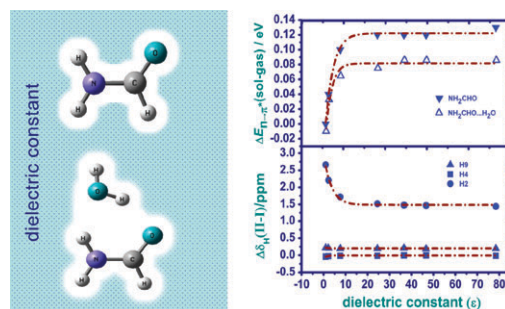


1709

Solvent effects on isolated formamide and its monohydrated complex: observations from PCM study

Anqun Chen, Xuemei Pu,* Shuhua He, Yanzhi Guo,
Zhining Wen, Menglong Li, Ning-Bew Wong and
Anmin Tian

A polarizable continuum model (PCM) is used to study solvent effects on geometries, vibrational frequencies, binding energies, ^1H chemical shifts and $n \rightarrow \pi^*$ transition energies of formamide in isolated and monohydrated forms.



1720

Optical properties of different polymer thin films containing *in situ* synthesized Ag and Au nanoparticles

Rafael Abargues,* Kamal Abderrafi, E. Pedrueza,
Rachid Gradess, J. Marqués-Hueso, Jose Luis Valdés and
Juan Martínez-Pastor

Ag and Au nanoparticles have been *in situ* synthesized inside several polymer thin films. The polymers used were able to reduce Ag(I) and Au(III) to the corresponding nanoparticles during the baking process in a one-step procedure.



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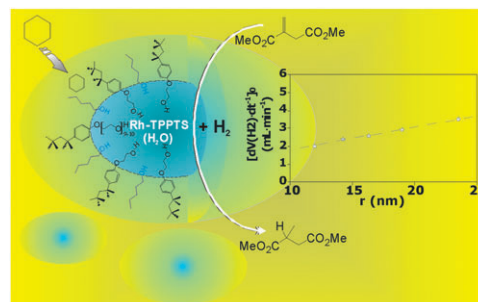
PAPERS

1726

Catalytic hydrogenation of dimethyl itaconate in non-ionic microemulsions: influence of the size of micelle

Juan Milano-Brusco, Sylvain Prévost, Dersy Lugo, Michael Gradzielski and Reinhard Schomäcker*

The initial hydrogenation rate of DMI is proportional to the Rh–TPPTS concentrated Igepal CA-520 micelle radius, which was measured with DLS, and also proportional to catalyst concentrated Triton X-100 micelle radius, determined from SANS spectra using Guinier approximations.

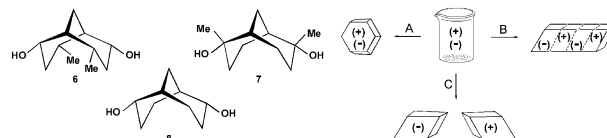


1736

Crystallisation of C_2 -symmetric *endo,endo*-bicyclo[3.3.1]nonane-2-6-diols: supramolecular synthons and concomitant degrees of enantiomer separation

Vi T. Nguyen, Isa Y. H. Chan, Roger Bishop,* Donald C. Craig and Marcia L. Scudder

Diol **6** crystallises as racemic crystals (path A), **7** as layers of opposite handedness (B), while **8** undergoes self-resolution (path C). The latter process represents a formal route to resolved bicyclo[3.3.1]nonanes of synthetic value.

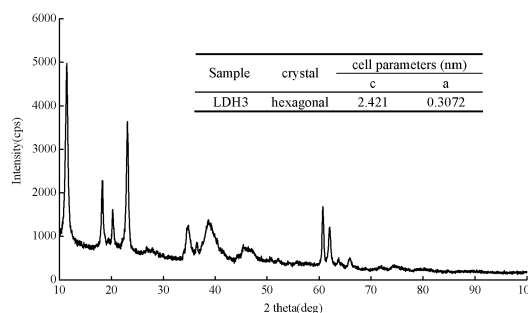


1742

Synthesis of stable W/O microemulsions of a hybrid grafted copolymer using a new inorganic cationic backbone within a unique “polyhedral cell”

Zheng Qi* and Peishi Qi

A graft copolymerization on a novel inorganic cationic backbone MgAl-LDH3 processing within a “polyhedral cell”.

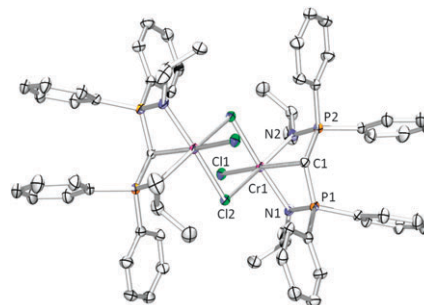


1748

Chromium (III)-bis(iminophosphoranyl)methanido complexes: synthesis, X-ray crystal structures and catalytic ethylene oligomerization

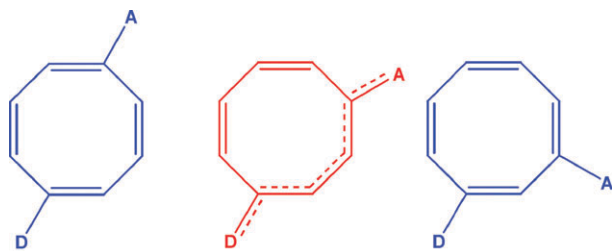
Christian Klemps, Antoine Buchard, Romaric Houdard, Audrey Auffrant, Nicolas Mézailles, Xavier Frédéric Le Goff, Louis Ricard, Lucien Saussine, Lionel Magna and Pascal Le Floch*

New Cr(III) complexes are active catalysts in ethylene oligomerization and polymerization.



PAPERS

1753

D – π -electron donor, A – π -electron acceptor**Substituent effects in mono- and disubstituted 1,3,5,7-cyclooctatetraene derivatives in natural and planar conformations**

Marcin Palusiak* and Tadeusz M. Krygowski

In COT the presence of the substituents leads to a partial delocalization in the antiaromatic $4n$ π -electron system, while in benzene, being a $4n + 2$ π -electron system, exactly the opposite effect can be observed.

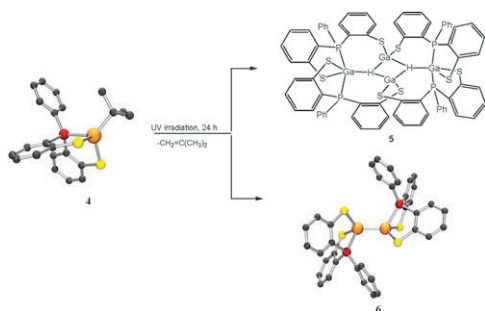
1760

**Reactions of $\text{H}_2\text{Os}_3(\text{CO})_{10}$ with triallylboranes: formation of novel triosmium boron-containing olefin clusters**

O. A. Kizas,* S. Yu. Erdyakov, D. Yu. Antonov, I. A. Godovikov, E. V. Vorontsov, F. M. Dolgushin, M. G. Ezernitskaya and I. G. Barakovskaya

$\text{H}_2\text{Os}_3(\text{CO})_{10}$ reacts with triallylboranes to give novel B-containing olefine clusters *via* a complex reaction pathway involving cleavage of the B-allyl bond. The mechanism of the reaction is discussed.

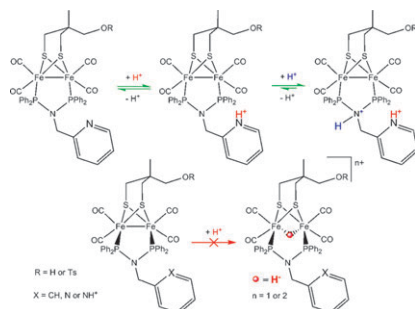
1771

**Stabilisation of an inorganic digallane by the phosphinobisthiolato P,S,S pincer ligand $\text{PPh}(2\text{-SC}_6\text{H}_4)_2$**

Ana-Maria Vălean, Santiago Gómez-Ruiz, Peter Lönnecke, Ioan Silaghi-Dumitrescu, Luminita Silaghi-Dumitrescu* and Evamarie Hey-Hawkins*

The pincer ligand $\text{PPh}(2\text{-HSC}_6\text{H}_4)_2$ reacts with GaCl_3 or GaR_3 to yield $[\text{Ga}\{\text{PPh}(2\text{-SC}_6\text{H}_4)_2\text{-}\kappa^3\text{S,S',P}\}\{\text{PPh}(2\text{-SC}_6\text{H}_4)_2\text{-}\kappa^2\text{S,S'}\}]^-$ or $\text{GaR}\{\text{PPh}(2\text{-SC}_6\text{H}_4)_2\text{-}\kappa^3\text{S,S',P}\}$ [$\text{R} = \text{Me}, \text{'Bu}$ (4)]. 4 decomposes in daylight or under UV irradiation to give the digallanes 5 and 6 among other compounds.

1780

**Synthesis and characterisation of three diiron tetracarbonyl complexes related to the diiron centre of [FeFe]-hydrogenase and their protonating, electrochemical investigations**

Yanwei Wang, Zhimei Li, Xianghua Zeng, Xiufeng Wang, Caixia Zhan, Yinqiu Liu, Xirui Zeng, Qiuyan Luo and Xiaoming Liu*

The rigidities and shielding effect from the PNP phosphine ligands prevent these diiron tetracarbonyl complexes from forming bridging hydrides upon protonation.

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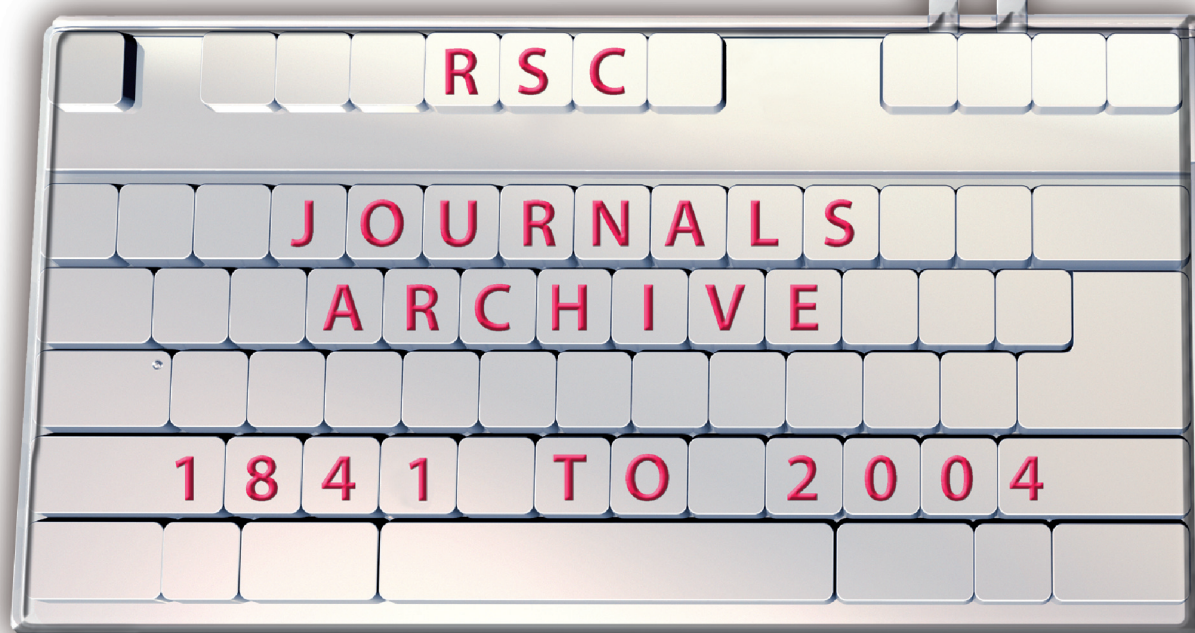
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Highlights in Chemical Science

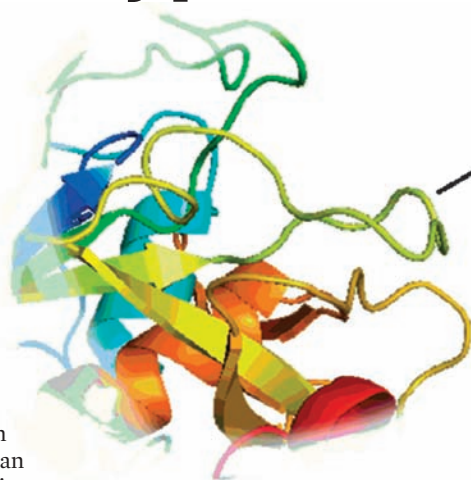
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A Nobel Prize-winning C=C bond forming reaction is applied to proteins Tag and modify proteins

New protein structures can be created using a process called tag and modify, suggest UK scientists.¹ They hope the process will lead to developments such as new drugs and biochemical probes.

Ben Davis at the University of Oxford and his team tagged a protein with an amino acid to make a structure that can be transformed into new proteins in a Nobel Prize-winning reaction called olefin cross metathesis. This carbon-carbon double bond forming process is an important transformation reaction in organic chemistry with a broad spectrum of use, explains Davis, but until their work last year no one knew how to apply it to proteins.²

The team investigated different allylation pathways to incorporate a modified form of the cysteine amino acid, called S-allyl cysteine, into proteins. Davis explains that having complementary ways to incorporate the tag will widen the scope for



cross metathesis in protein modification.

Robert Grubbs from the California Institute of Technology, Pasadena, US, who shared the 2005 Nobel Prize in chemistry, in particular for his contribution to work on the metathesis reaction, says that Davis' method is excellent. 'It demonstrates a new technique for protein modification with a wide

Amino acids can be incorporated into proteins to make new structures

array of functionality,' he adds.

There are many possibilities for using cross metathesis to form new protein structures, says Davis.

For example, ring-closing cross metathesis could be used to stabilise large structures like enzymes, he suggests. 'Ultimately, we believe we can use a panel of tags and reactions to modify to re-programme protein structure and function at will, simply using chemistry,' he says. David Crich, an expert in carbohydrate synthesis at The National Centre for Scientific Research, Gif-sur-Yvette, France, comments: 'This combination of direct allylation and subsequent metathesis is sure to find widespread application.'

Katherine Davies

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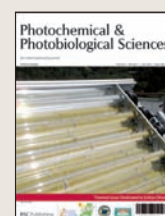
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A microelectrode can remotely control the movement of a nanomotor

Tiny pushes from a distance

Nanomotor movement can now be controlled remotely, say US researchers.

Joseph Wang at the University of California San Diego and colleagues have, for the first time, demonstrated electrochemical control of a fuel-driven nanomotor. Wang and his team pioneered a new generation of fuel-driven catalytic nanomotors and now they can also control their speed and starting and stopping motion.

Nanomotors are tiny molecular devices capable of converting energy into movement. Wang developed a nanomotor where the on/off mechanism is controlled by a gold electrode placed near the motor applying different potentials. The potential in the microelectrode triggers electrolytic reactions of the fuel constituents. Wang explains that 'a positive or negative potential in the electrode leads to generation or consumption of oxygen, respectively, which leads to stoppage or acceleration of motion,



respectively.' By controlling the exact negative potential, the oxygen level is controlled and hence the motor speed.

Henry Hess, an expert on engineering applications of nanoscale motors at the University of Florida, US, highlights that 'this principle might ultimately lead to networks of electrodes which guide the speed and direction of catalytic nanomotors like radio beacons guide airplanes along

Nanomotor speed and starting and stopping motions can be controlled remotely

Reference

P Calvo-Marzal *et al*, *Chem. Commun.*, 2009, DOI: 10.1039/b909227g

their path.'

Wang is excited by the possibilities that have opened up by this work. The on-demand triggering of motion and the control of speed is 'a first step towards the challenging design of functional nanomachines,' he says. The team's future work will include trying to achieve faster on/off activation and more complex movement patterns.
Roxane Owen

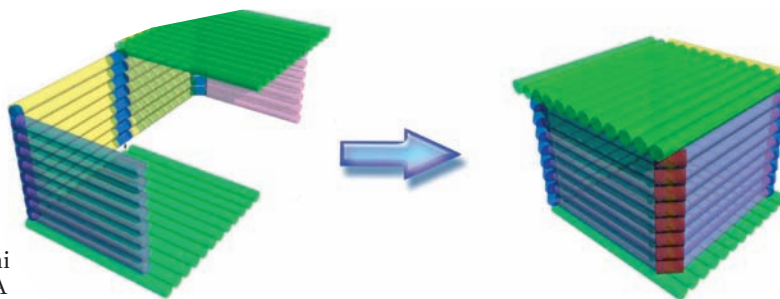
Scientists have made a potential drug delivery vessel from DNA

DNA-in-the-box

DNA boxes to encapsulate nanomaterials have been constructed by Japanese researchers.¹

Assembling DNA molecules into organised and programmable structures is known as DNA nanotechnology. One important area is DNA origami in which single-stranded DNA is folded to construct arbitrary structures. These can then be used as platforms for nanostructures such as protein arrays, gold nanoparticles and messenger RNA.

Until now, researchers have focused on 2D origami, but Makoto Komiyama and Akinori Kuzuya at the University of Tokyo have shifted their focus to 3D structures. The pair folded DNA strands into an open-box shape and identified it using atomic force microscopy. 'For guest encapsulation, the box is



designed to fold into an open form first, and can be closed afterwards by adding appropriate DNA strands,' explains Komiyama. 'The outside and inside of the box can be distinguished throughout the process so it may be possible to place a guest molecule in the face of the open form before closing it.'

The boxes are similar in size to virus capsids, which are currently gaining interest as nanocontainers, says Komiyama, but the faces of

The open-box DNA could encapsulate a guest molecule before being closed up into a box

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the DNA boxes are easier to chemically modify, enabling selective capture of various guest molecules.

Komiyama admits that this will be challenging. 'DNA is a polyanionic material, so the inner cavity of 3D origami is surrounded by thousands of anions,' he says. 'We will have to check if useful guests like proteins will enter such a small space and stay there.'

Kurt Gothelf, an expert on DNA nanotechnology at the University of Aarhus, Denmark, comments: 'DNA origami is going 3D and this box is a beautiful example. Like Hao Yans' DNA tetrahedron² and our DNA box,³ the internal cavity of this structure may potentially be used for drug delivery.'

Keith Farrington

A solution to keeping the atmosphere clean could lie at the heart of the rainforest

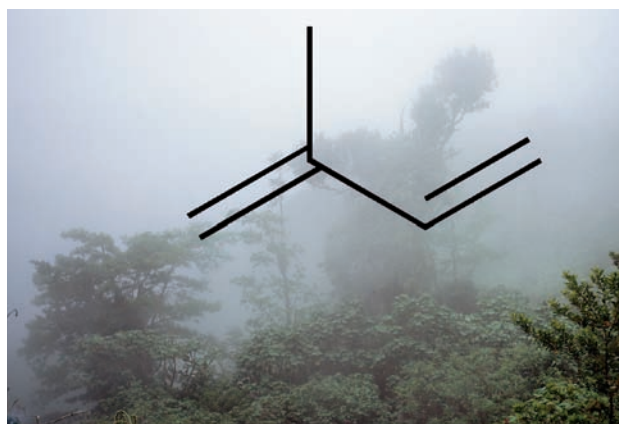
Tropic wonder

Tropical rainforest emissions of the biogenic compound isoprene increase the self-cleansing power of the atmosphere, say scientists in Belgium.

Using theoretical models, Jozef Peeters and colleagues from the Catholic University of Leuven proposed chemical mechanisms showing how the amount of hydroxyl radical – the detergent of the atmosphere – could be enhanced by isoprene oxidation.

The hydroxyl radical is a naturally occurring compound that scrubs many manmade pollutants from our atmosphere. Popular opinion has long been that in unpolluted air, like that of a rainforest, the large amounts of isoprene emitted from plants would deplete the hydroxyl radical. So in regions with high isoprene levels you might expect to find low hydroxyl concentrations.

Recent measurements in the Amazonian rainforest have shown the amount of hydroxyl radical



Understanding the chemistry of isoprene is a leading challenge for atmospheric chemists

Reference

J Peeters, T L Nguyen and L Vereecken, *Phys. Chem. Chem. Phys.*, 2009, **11**, 5935 (DOI: 10.1039/b908511d)

present greatly exceeds that predicted using current atmospheric chemical models. So why is this? Peeters says it is because rather than removing the hydroxyl radical species, isoprene oxidation actually recycles it.

Isoprene reacts with hydroxyl radicals to form OH–isoprene adducts which rapidly add oxygen

yielding several hydroxy-peroxy radicals. The mechanism put forward by Peeters and the team is based on the fact that the major hydroxy-peroxy radicals can eliminate oxygen so fast that the various isomers interconvert. A hydrogen shift on one form of the hydroxy-peroxy radical regenerates the HO radical.

‘Regarding specifically the atmospheric oxidation of isoprene, the experimentalists face the considerable but urgent challenge of confirming – or disproving – the new chemistry we have proposed,’ says Peeters.

‘Understanding the chemistry of isoprene and other plant emitted organics is currently the leading challenge for atmospheric chemistry,’ says Mat Evans, an expert in atmospheric composition modelling at the University of Leeds, UK. He adds: ‘This work invokes new tools to aid our understanding and provides a unique insight.’

Sarah Corcoran

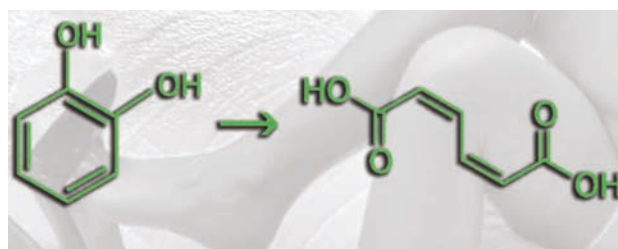
An immobilised enzyme could change the face of nylon production

Greener nylon with nanosponges

A biocatalyst with the potential to green up industrial nylon production has been created by scientists in Italy and the UK.

The nylon monomer adipic acid not only has a precursor derived from carcinogenic benzene, but its formation also requires an oxidation step using nitric acid. This process increases atmospheric nitrous oxide levels, contributing to global warming and ozone depletion.

Now, Gianfranco Gilardi from the University of Turin and colleagues have produced an alternative precursor, *cis-cis* muconic acid, from catechol, which is greener and cheaper than benzene. The team used their biocatalyst to cleave catechol, giving the muconic acid, which can be simply hydrogenated to give adipic acid. Gilardi explains: ‘The availability of a biocatalytic system able to produce [muconic acid] has an important environmental



The enzyme was used to produce the nylon precursor *cis-cis* muconic acid from catechol

Reference

G Di Nardo *et al*, *Dalton Trans.*, 2009, DOI: 10.1039/b903105g

impact for the full process [of nylon production].’

Gilardi made the biocatalyst by immobilising an enzyme called catechol 1,2-dioxygenase onto nanosponges, which strongly increased the enzyme’s stability, a result beyond the team’s expectations. Not only does the biocatalyst have increased thermostability and pH tolerance, but it is also more stable to storage than the free enzyme.

The nanosponges consist of cyclodextrins (cyclic cone-shaped

oligosaccharides) linked by carbonate groups. ‘Cyclodextrins make an interesting immobilisation medium,’ says Dean Brady, an expert in biocatalysis and applied enzyme and process biotechnologies from The Council of Scientific and Industrial Research, Biosciences, Modderfontein, South Africa. ‘Not only are they enantioselective so may be selective in their binding, allowing for specific orientation of the protein, they can also re-fold denatured protein, which would be an advantage for extended biocatalyst performance.’

Gilardi’s team engineered a small scale bioreactor containing their catalyst and they were able to efficiently convert catechol into muconic acid for 70 days. ‘The idea now,’ says Gilardi, ‘is to develop a continuous flow system for the development of an industrial bioreactor.’ *Frances Galvin*

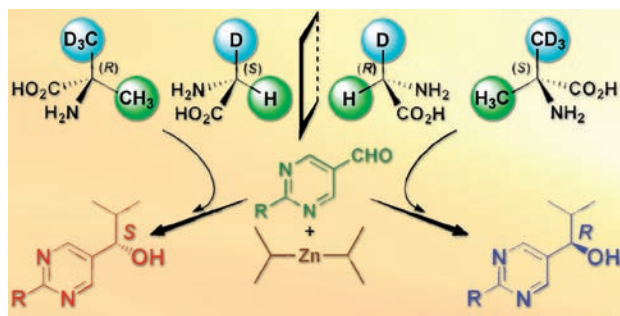
High deuterium levels in meteorites could have been responsible for life on Earth

The achiral key to life

In living organisms, chiral compounds such as amino acids and sugars exist as single enantiomers. What is the origin of this preference? Japanese scientists believe that achiral molecules from space could provide the answer.

One theory for the presence of single enantiomers in nature is that slight excesses of one enantiomer in the prebiotic world could have been increased further by autocatalysis, a reaction where the product promotes its own formation. These initial excesses may be extraterrestrial in origin, having been observed for a variety of chiral molecules found in meteorites.

Now, Kenso Soai and co-workers from the Tokyo University of Science claim that a high deuterium content in meteoritic environments could mean that



achiral compounds are just as important. 'Even the most simple achiral amino acid, glycine, becomes chiral when one of the methylene group's hydrogen atoms is substituted by deuterium,' explains Soai.

Detecting enantiomers formed by isotope substitution is difficult though, says Soai. Despite this, the team found that deuterium-

The achiral molecules in meteorites may have been made chiral by deuterium

Reference
T Kawasaki *et al*, *Chem. Commun.*, 2009, 4396 (DOI: 10.1039/b908754k)

enriched amino acids glycine and α -methylalanine catalysed reactions between aldehydes and organozinc compounds to give enantioenriched alcohols in high excesses. The alcohols' chirality depended on that of the amino acid. Soai says that his team is the first to show that isotopically chiral amino acids can promote such a highly specific reaction.

'Substantive interest lies in analysis of meteorites with a small enantiomeric excess in amino acid content,' says John Brown, an expert in asymmetric catalysis at the University of Oxford, UK. 'Soai's suggestion is that this chemistry could be used to discover whether the known deuterium excess in meteorites is correlated with asymmetry in otherwise achiral amino acids.'

Bailey Fallon

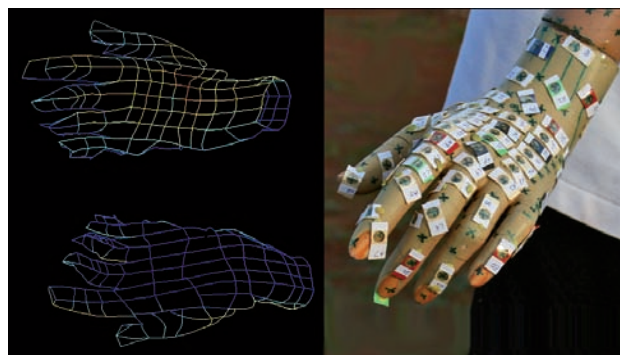
Skin cancer's location on the body depends on the body's distance from the equator

Where the sun does most damage

A link between the location of skin cancer on the body and the sun's position in the sky has been found by Australian scientists.

Australia has the highest incidence rate of non-melanoma skin cancer (NMSC) in the world. The country is at a low latitude (a short distance from the equator), which means that it receives higher UV exposure levels throughout the day than countries at higher latitudes.

To investigate how this affects skin cancer incidence, Nathan Downs and Alfio Parisi at the University of Southern Queensland, Toowoomba, spent four years measuring UV exposure levels on a life-sized mannequin in Australia. They took measurements at 1453 body sites, improving upon previous data. When the sun was high in the sky, all parts of the mannequin received the highest irradiation levels. This is because the sunlight takes a shorter path through the atmosphere. But then the pair found



that when the sun was lower in the sky, larger areas of exposed skin, such as the leg, received more low-level UV exposure.

Downs and Parisi then compared the results with the distribution of pre-cancerous patches of skin called solar keratoses (SKs) and the two most common NMSC types, squamous cell carcinoma (SCC) and basal cell carcinoma (BCC). They found that areas with greater UV exposure, such as the nose, tended to have more BCC tumours.

UV exposure was measured at 1453 points on the body, improving on previous data

Reference
N Downs and A Parisi, *Photochem. Photobiol. Sci.*, 2009, DOI: 10.1039/b901741k

SKs were found to have a stronger relationship with UV exposure, being more frequent on the cheeks, top of the ears and nose. 'A more detailed collection of SKs, SCC and BCC incidence data would be of great benefit, particularly if this data could be mapped onto the body wireframe models at each of the 1453 body sites,' says Downs.

Downs plans to investigate the effect of playing sport on UV exposure. 'At the moment I am looking at measuring UV exposures to golfers, swimmers and snorkellers with human and mannequin subjects,' he adds.

Marko Weber, an expert in laser and optical radiation safety at Seibersdorf Laboratories, Austria, says: 'Any study that tries to improve our knowledge of skin cancer and tries to reveal the causes for its development is really important, because the incidence rate will increase in the future and could become a burden for our health systems.'

Sylvia Pegg

The organic chemist's toolbox

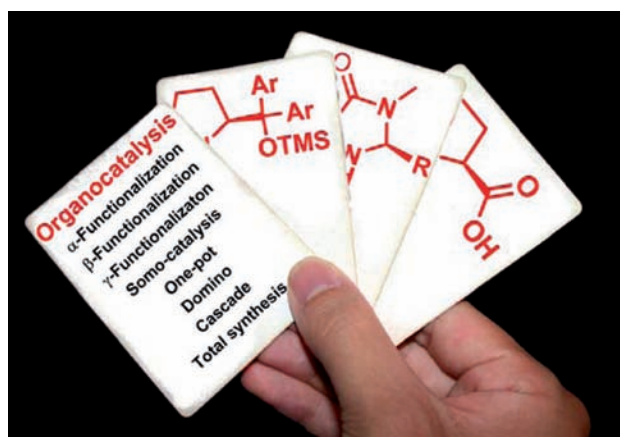
Karl Anker Jørgensen and Søren Bertelsen from Aarhus University, Denmark, talk about impressive achievements in organocatalysis

Asymmetric synthesis has been the focus of a number of organic chemists for many years. Now, constructing stereogenic carbon atoms is a fundamental part of organic chemistry, not least in life science, where strict regulations regarding stereogenic pharmaceuticals, for example, make stereoselective synthesis highly interesting. As such, methods for constructing and/or manipulating stereocentres are central tools in the organic chemist's toolbox.

Traditionally, stereocentres have been obtained and transformed from natural sources, but asymmetric catalysis has allowed us to construct non-racemic stereocentres *de novo* using substoichiometric catalyst amounts. Clearly, designing and understanding the catalysts' mechanisms are highly important.

Enzymes and metals have played central roles in asymmetric catalysis and are still considered the best for a number of transformations. Recently, though, organocatalysis has entered the scene, promising easy procedures to synthesise valuable optically active intermediates. Organocatalysis complements established procedures involving metal or enzyme catalysts, but it allows a greater substrate scope than most enzymes and avoids the use of metals, which can be difficult to remove from products.

A privileged organocatalyst family is the secondary amines. Stereoselective carbonyl compound transformations by secondary amine catalysts have been known for several decades, but the interest in their use exploded less than a decade ago when they efficiently catalysed aldol and [4+2]-cycloaddition reactions. In particular, the naturally occurring amino acid proline has proven to be a highly privileged



Holding all the cards – the versatility of organocatalysis

structure. It is able to catalyse reactions with amazing results considering the simplicity of its structure. The usual substrates for many of these reactions are carbonyl compounds, such as aldehydes or ketones. They are easy to transform, making versatile products for further reactions, and the secondary amines have shown great potential as carbonyl compound activators.

Organocatalysts have impressed us with their ability to promote stereoselective incorporation of non-metallic elements into an α -C–H bond relative to the carbonyl compound. Several studies have focused on elucidating the underlying mechanism, but what about other synthetic challenges?

A close interplay between experimental and theoretical chemists has led them to develop new catalysts designed to address specific synthetic problems. Whereas proline, and more recently, other chiral secondary amines, have shown unsurpassed generality in the α -functionalisation of carbonyl compounds, organocatalysts are also being developed for stereoselective β -functionalisation. Most non-metallic (and non-halogenic) compounds can now, routinely, be

incorporated into the β -position with high stereoselectivities and good yields. Understanding the active mechanisms has been crucial in developing these transformations.

Recently, chemists have developed two new activation methods to allow for unprecedented transformations. The first was γ -functionalisation – on inspecting the mechanism, the possibility of an umpolung (a reversal of polarity when a functional group is modified) was realised. Consequently, it is now possible to perform stereoselective C–H to C–N or C–C bond transformations in the remote γ -position relative to the carbonyl. The other novel activation method employs radicals in mechanisms related to the α -functionalisations. Using these new methodologies, the previously elusive organocatalytic stereoselective α -alkylation of aldehydes has now been achieved.

Organocatalysts' complementary activation methods, then, open new vistas for one-pot, cascade or tandem reactions leading to highly complex products using simple procedures. Their versatility allows for intricate reaction protocols that lead to multiple activations of specifically designed molecules. One eminent example is the facile total synthesis of Oseltamivir, an antiviral drug to treat influenza, using a one-pot procedure. Various other applications of amino catalysts further underline the importance of this relatively new research field. Organocatalysis, we believe, will remain a highly competitive field, providing us with unexpected and fascinating new discoveries.

Read more in Søren Bertelsen and Karl Anker Jørgensen's tutorial review 'Organocatalysis – after the gold rush' in issue 8, 2009 of Chemical Society Reviews.

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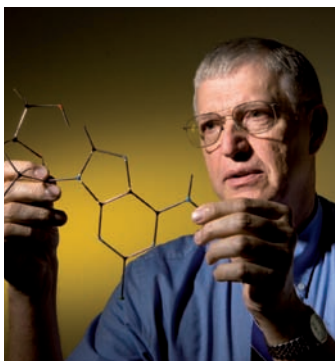
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Understanding fundamentals

Morris Robins talks to Kathleen Too about turning crystalline sugars black, discovering Epivir and Cladribine and his new career in farming



Morris J Robins

Professor Morris J Robins is a former professor at Brigham Young University, Utah, US. His main achievements have been in the areas of bioorganic, medicinal and organic chemistry and cancer research. He has pioneered several nucleosides as commercial drug candidates, for example Didanosine, Epivir and Cladribine.

Who or what inspired you to become a chemist?

I was inspired by a freshman chemistry professor, Lloyd Malm, at the University of Utah, US. He was very dynamic, fascinated by chemistry and he took an interest in me. That's why I really decided to pursue chemistry.

Which research are you most proud of in your career?

I had a research programme that was sort of focussed on two areas. One was the development of potential drugs, both antiviral and anticancer, and the other was the discovery of new reactions. So from a point of view of satisfaction, the development of a drug that would help people was a really major thing. But personally I also found it exciting to develop a new reaction so that you could do things that hadn't been performed before.

When I was a professor at the University of Alberta, in Edmonton, Canada, I met David Lorne John Tyrrell (he goes by Lorne Tyrrell). He was the head of the department of microbiology and infectious diseases in Alberta. We met for lunch and talked about the mode of replication of hepatitis B that had just been published. Since a reverse transcriptase activity was involved in the replication of the hepatitis B virus and we'd developed active compounds against AIDS, we talked about the possibility of working together on the hepatitis B virus. We discovered the first inhibition of hepatitis B by dideoxy nucleoside analogues, and then we partnered with Glaxo to develop Epivir (also known as Lamivudine), which could be applied to combat hepatitis B. A couple of months ago, my friend and colleague at the University of Alberta phoned me and said that now Epivir is the number one selling drug in China. There, hepatitis B is endemic and so it is quite pleasing that it is the number one drug. In 1998, Lorne Tyrrell and I were jointly awarded the Prix Galien Canada Research Award for this work.

Do you remember your first independent discovery?

When I was in graduate school, I actually developed the first synthesis of a compound, 2-chloro-2'-deoxyadenosine, which has the generic drug name of Cladribine. I got the idea from a group seminar in which we were talking about a fusion reaction developed in Japan where a purine and a sugar could be melted together to evolve acetic acid and form a glycosyl linkage. And so I thought, 'Ah, we could do that and make deoxynucleosides!' I took the deoxy sugar acetate from deoxyadenosine

and fused it with different purines, but the deoxy sugar was much less stable and decomposed readily and I had multiple failures. I could turn beautiful white crystalline purines and colourless clear deoxy triacetate sugars into black mixtures very well. Eventually I got it right!

Dennis Carson at Scripps (La Jolla, US) discovered Cladribine's anticancer activity against B and T cell type malignancies, and so this drug is the current drug of choice for hairy-cell leukaemia. Hairy-cell leukaemia is a minor form of the disease in terms of the total number of people that have it, but the drug is curative or provides long-term remission for essentially 90 per cent of patients, which is quite amazing! Over the years I've seen this drug developed as an anticancer agent.

What is your approach to success?

Well, in my opinion, academic chemists should be involved with the discovery of new creative chemistry. I always focussed on the discovery of new reagents and new reactions. We were very fortunate in finding compounds with biological activity and we always had this application in the back of our minds. So my approach involved new creative discovery of new reactions, and then application to compounds that were previously difficult to access, rather than taking a compound and making multiple derivatives. Now that doesn't mean that I pass judgement on other academic chemists who use different approaches, but that is my approach.

What is your message to young people?

I would advise young people to find out what they are really interested in, what they can be patient about pursuing, and then become immersed in it. They should understand it and go beyond where most people are willing to go. I was able to make discoveries by understanding the fundamentals and going a step beyond on a more qualitative rather than quantitative basis.

How do you plan to embrace retirement?

Enthusiastically! For me it's just a change of emphasis. I'm not retiring in any sense of the word but the time has come where it's right for me to not be teaching classes of 300 and to stop running a big group. So I bought a farm. I'm still interested in creative things and I get to figure out things like biofuels and green chemistry. I am as involved as ever and as busy now as I was before.

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Nearly all the RSC journals increased in impact factor, immediacy index and article influence, with 3 journals noticeably standing out. *Chem Soc Rev* increased by 33 per cent to an incredible 17.42, confirming its position as a first class international chemistry journal.

Lab on a Chip celebrates a 28 per cent rise taking its impact factor to 6.48, placing it within the top ten journals in

the multidisciplinary chemistry category. *PCCP* also fared well, with an impressive 20 per cent increase to its highest ever impact factor of 4.06. Its new immediacy index (0.81) also remains the highest value for any journal publishing general primary research in the fields of physical chemistry and chemical physics.

In addition, RSC journals feature in the top 10 rankings (by impact factor and immediacy index) in 6 of the 7 core chemistry categories* as listed on ISI, and, of the top 100 chemistry journals,



ranked by impact factor, 15 are from RSC Publishing.

James Milne, editorial director for RSC Publishing comments: 'RSC journals have attracted a significant increase in submissions, with nearly 60 per cent more material published (over the past 5 years).' He continues: 'To provide more articles and also higher quality articles is a clear reflection of the dedicated support the journals receive from authors, editors and referees throughout the world; for this contribution, I would like to sincerely thank all the scientists involved.'

Visit www.rsc.org/publishing to read more about the journals' impact factor results.

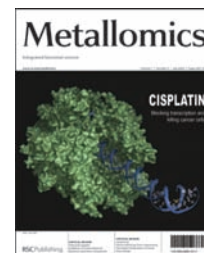
* The 7 chemistry journal subject-categories as listed by ISI: Chemistry, Analytical; Chemistry, Applied; Chemistry, Inorganic & Nuclear; Chemistry, Medicinal; Chemistry, Multidisciplinary; Chemistry, Organic; Chemistry, Physical.

Metallomics

Since its launch in January this year, new journal *Metallomics: Integrated Biometal Science* has attracted articles from some of the leading names in the field. This timely new journal is expected to be the core publication for the emerging metallomics community as they strive to fully understand the role of metals in biological, environmental and clinical systems.

Metallomics celebrated its 2009 launch at the Second International Symposium on Metallomics (ISM'09), held in Cincinnati, Ohio, US, from 7–10 June. The journal was proud to sponsor this high profile meeting, which attracted many leading researchers, and saw sessions covering human metallomics, microbial metallomics, metallomics technology, phytometallomics and environmental metallomics. *Metallomics* is the recommended avenue of publication for ISM'09: watch out for a themed issue early in 2010 featuring work presented at the conference.

The current issue of *Metallomics* is free to all readers online throughout 2009 and 2010, and free institutional online access to all 2009 and 2010 content is available following a simple registration process.



Find out more at www.rsc.org/metallomics

e-Platform

In 2010 RSC Publishing will launch a powerful new content delivery platform that supports multiple content types. The new website will deliver world-class RSC-hosted journal, book and database content in a single platform. The new platform will span more than 165 years of premium content, including 20 000 book chapters; 300 000 journal articles; and 450 000 database records.

RSC has worked with experts in software engineering to develop the platform functionality and has consulted a leading web design agency

on building the user interface. Designed around our readers' preferences (identified from a detailed user-interview process), our user-friendly platform will offer faster browsing, intelligent searching, consistent user experience irrespective of content type sought, and simpler more intuitive navigation.

Graham McCann, publisher, is spearheading the project and his enthusiasm for the platform makes it clear something exciting is happening. 'The next stage is beta-testing; we can't wait to show some of our users

the innovative platform we're working on,' he says. 'Our aim is to combine rich functionality and powerful searching with some additional features that will deliver an exceptional online experience. True to RSC reputation as an innovator in chemical science publishing, we're going to deliver something unique and different.'

For the rest of 2009, the platform will undergo extensive user testing. To be among the first to hear the latest news about the new Platform follow ChemPub on Twitter (www.twitter.com/ChemPub).

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