

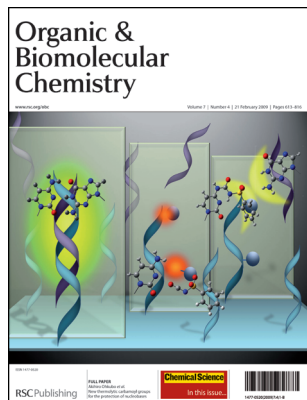
# Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry  
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## IN THIS ISSUE

ISSN 1477-0520 CODEN OBCRAK 7(4) 613–816 (2009)



### Cover

See Akihiro Ohkubo *et al.*,  
pp. 687–694.

ON–OFF switching of a G–C base pair by modification of an oligonucleotide using the psc group on a glass plate.

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## CHEMICAL SCIENCE

### C9

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

## Chemical Science

February 2009/Volume 6/Issue 2

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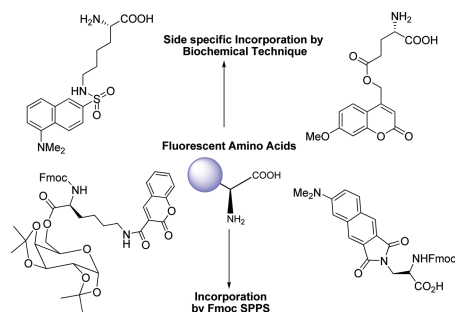
## PERSPECTIVE

### 627

#### Fluorescent amino acids: advances in protein-extrinsic fluorophores

Alan Roy Katritzky\* and Tamari Narindoshvili

This perspective discusses advances in fluorescent probes for proteins focusing on small extrinsic organic fluorophores highlighting fluorescent amino acids.



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# Organic & Biomolecular Chemistry

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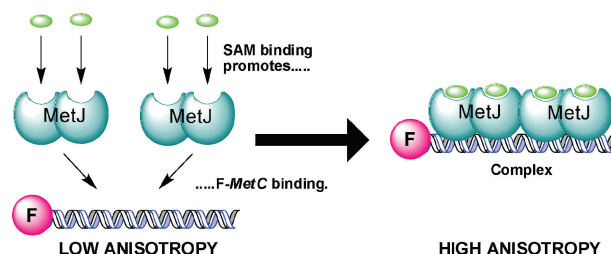
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**Identification of stable *S*-adenosylmethionine (SAM) analogues derivatised with bioorthogonal tags: effect of ligands on the affinity of the *E. coli* methionine repressor, MetJ, for its operator DNA**

Catherine Joce, Jamie Caryl, Peter G. Stockley, Stuart Warriner and Adam Nelson\*

The efficient synthesis of a range of stable SAM mimetics, and their ability to promote the binding of the *E. coli* methionine repressor (MetJ) to its operator DNA, is described. Active analogues functionalised with bioorthogonal tags were identified.

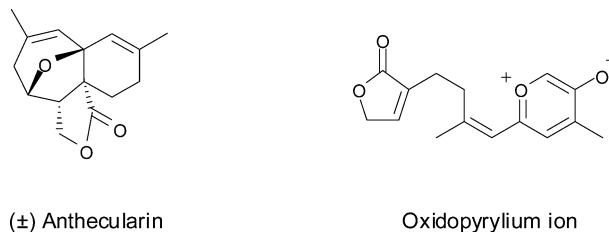


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**A concise total synthesis of ( $\pm$ )-antheclarin**

Yi Li, Christopher C. Nawrat, Gerald Pattenden\* and Johan M. Winne

Antheclarin is synthesized using a [5+2] (1,3-dipolar) intramolecular cycloaddition reaction from an oxidopyrylium ion intermediate.

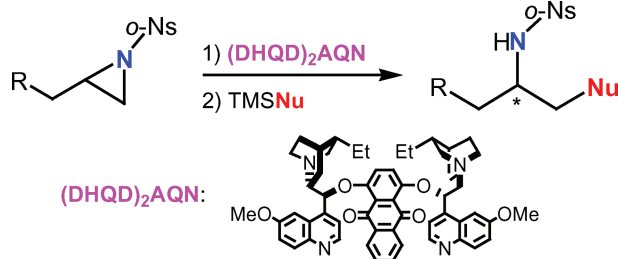


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**Asymmetric recognition and sequential ring opening of 2-substituted-*N*-nosylaziridines with (DHQD)<sub>2</sub>AQN and TMSNu**

Satoshi Minakata,\* Yuta Murakami, Masamitsu Satake, Ikumasa Hidaka, Yuriko Okada and Mitsuo Komatsu

A new method for asymmetric ring opening of terminal aziridines using a chiral amine, (DHQD)<sub>2</sub>AQN, is described; the reaction is based on the asymmetric recognition of aziridines using (DHQD)<sub>2</sub>AQN and on sequential ring opening using TMSNu.

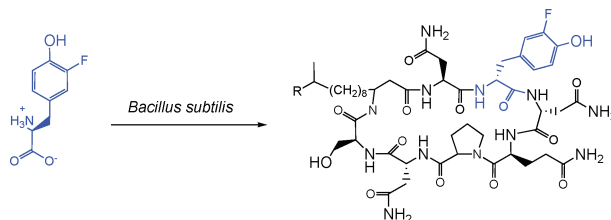


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**Precursor-directed biosynthesis of fluorinated iturin A in *Bacillus* spp.**

Stephen Moran, Dilip K. Rai, Benjamin R. Clark and Cormac D. Murphy\*

*Bacillus* sp. strain CS93 can incorporate 3-fluoro-L-tyrosine into iturin A in place of tyrosine, which might result in a more potent anti-fungal agent.

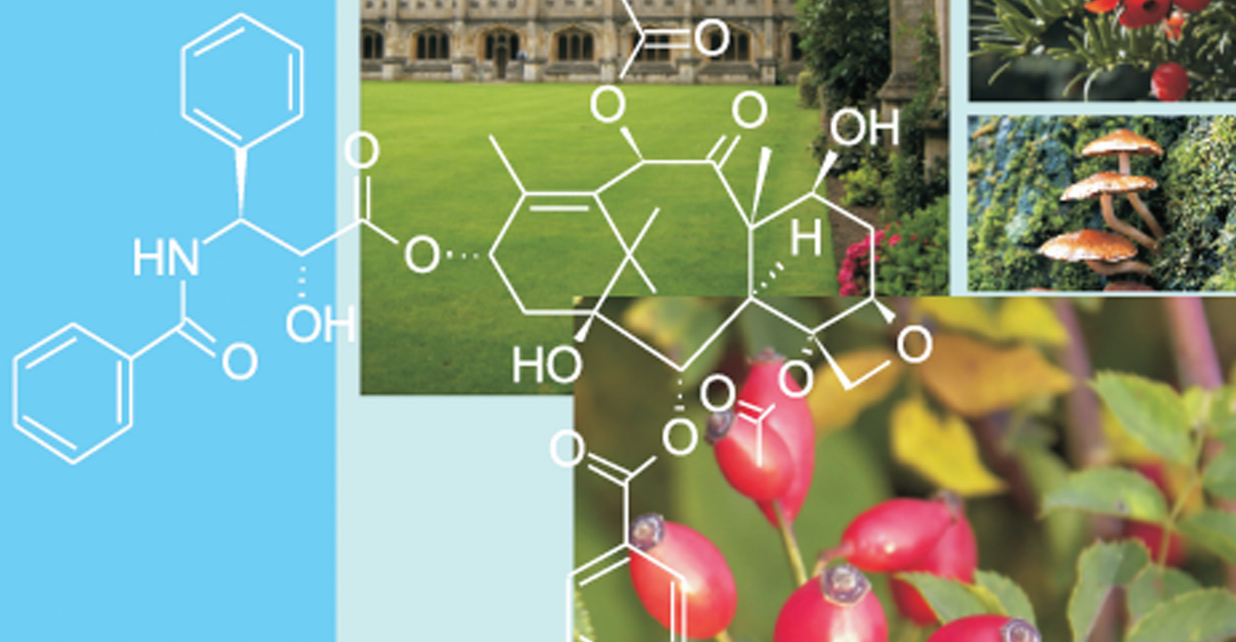
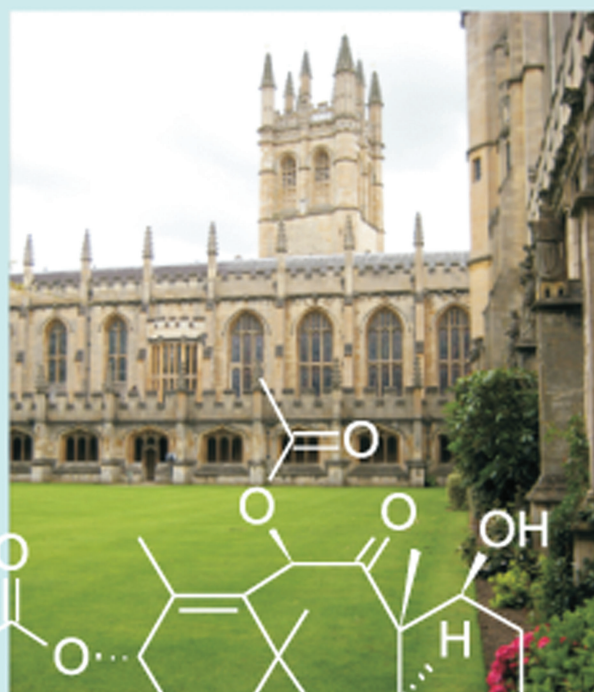


# Functional Molecules from Natural Sources



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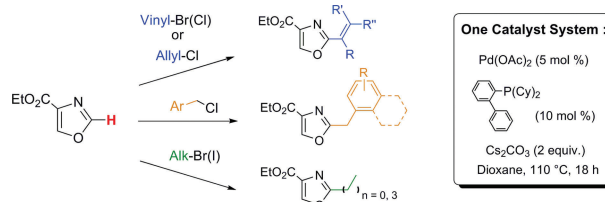


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### Direct palladium-catalyzed alkenylation, benzylation and alkylation of ethyl oxazole-4-carboxylate with alkenyl-, benzyl- and alkyl halides

Cécile Verrier, Christophe Hoarau\* and Francis Marsais

A unique Pd(OAc)<sub>2</sub>/JohnPhos ligand-based catalyst system was designed for direct palladium-catalyzed alkenylation, benzylation and alkylation of ethyl oxazole-4-carboxylate with vinyl-, benzyl- and alkyl halides. The first examples of direct C–H bond alkylation of a heteroaromatic with alkyl halides are reported.



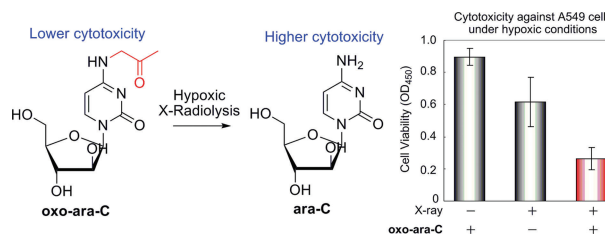
PAPERS

651

### Radiolytic activation of a cytarabine prodrug possessing a 2-oxoalkyl group: one-electron reduction and cytotoxicity characteristics

Nao Hirata, Yusuke Fujisawa, Kazuhito Tanabe,\* Hiroshi Harada, Masahiro Hiraoka and Sei-ichi Nishimoto\*

An anti-tumour agent of cytarabine (**ara-C**) was conjugated with a 2-oxopropyl group at the N(4) position to obtain a radiation-activated prodrug (**oxo-ara-C**) that released the toxic parent agent **ara-C** in hypoxic tumour cells *via* radiolytic one-electron reduction.

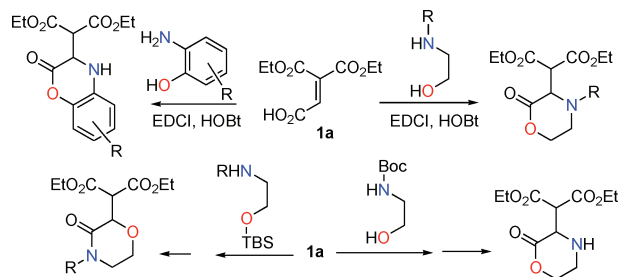


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### Efficient synthesis of heterocyclic compounds using ethenetricarboxylic acid diesters

Shoko Yamazaki,\* Yuko Iwata and Yugo Fukushima

The reaction of ethenetricarboxylic acid diester with 2-aminoalcohols in the presence of EDCI and HOBt in one pot gave *N,O*-containing heterocyclic compounds regioselectively. The stepwise method involving protected amine or alcohol nucleophiles afforded the regioisomeric 1,4-oxazine derivatives.

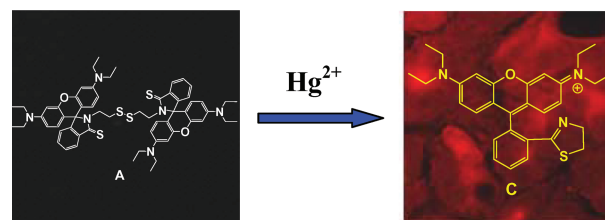


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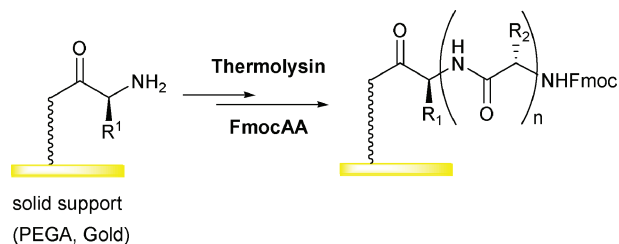
### Dithiolane linked thiorhodamine dimer for Hg<sup>2+</sup> recognition in living cells

Weimin Liu, Liwei Xu, Hongyan Zhang, Juanjuan You, Xiaoling Zhang,\* Ruilong Sheng, Huaping Li,\* Shikang Wu and Pengfei Wang\*

Thiorhodamine-based chemodosimeter **A** with a disulfide linker exhibits real-time responses, high sensitivity and unique selectivity for Hg<sup>2+</sup> over other cations, which are mechanistically ascribed to Hg<sup>2+</sup> induced desulfurization. The *in vitro* sensitivity to Hg<sup>2+</sup> was demonstrated in an HK-2 cell line using confocal microscopy.



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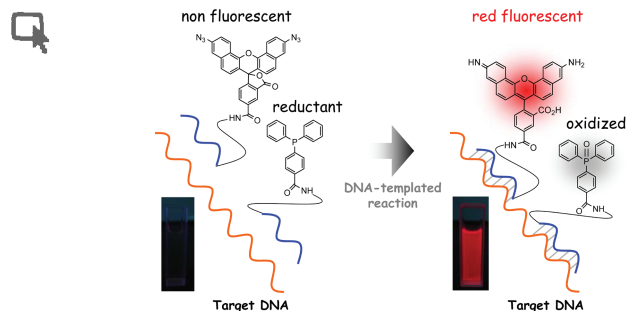


### Enzymatic synthesis of peptides on a solid support

Rose Haddoub, Martin Dauner, Fiona A. Stefanowicz, Valeria Barattini, Nicolas Laurent and Sabine L. Flitsch\*

We report the scope of a protease-catalysed peptide synthesis on PEGA resin using protected and glycosylated amino acids and show that such a method can also be applied on non-porous surfaces, in particular on gold.

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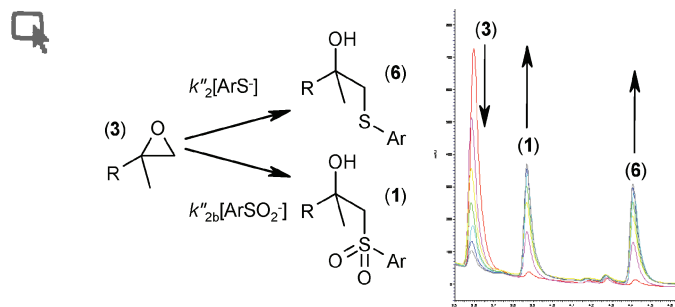


### Reduction-triggered red fluorescent probes for dual-color detection of oligonucleotide sequences

Kazuhiro Furukawa, Hiroshi Abe,\* Jin Wang, Miwako Uda, Hiroyuki Koshino, Satoshi Tsuneda and Yoshihiro Ito\*

A new red fluorogenic compound was synthesized from naphthorhodamine for a reduction-triggered fluorescence probe to sense oligonucleotides. The probes were applied to dual color detection of a single nucleotide difference.

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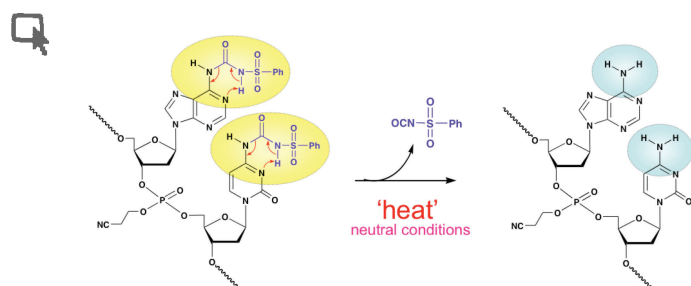


### Mechanistic studies on the synthesis of bicalutamide

Nabil Asaad\* and Shaun Fillery

A rigorous investigation into the kinetics and mechanism of the synthetically important formation of thioethers and sulfones from a parent halohydrin *via* the intermediate epoxide.

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### New thermolytic carbamoyl groups for the protection of nucleobases

Akihiro Ohkubo, Rintaro Kasuya, Kenichi Miyata, Hirosuke Tsunoda, Kohji Seio and Mitsuo Sekine\*

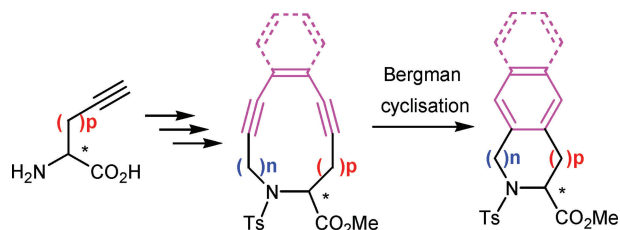
*N*-arylsulfonyl carbamoyl and *N*-(phenylsulfonyl)carbamoyl (psc) groups can be effectively introduced onto the amino groups of deoxycytidine and deoxyadenosine derivatives and can be removed thermolytically.

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### Synthesis and aromatisation of cyclic enediyne-containing amino acids

Jasper Kaiser, Bart C. J. van Esseveldt, Margot J. A. Segers, Floris L. van Delft, Jan M. M. Smits, Sam Butterworth and Floris P. J. T. Rutjes\*

Cyclic enediyne-containing amino acids of different ring sizes have been prepared starting from propargylglycine and homopropargylglycine.

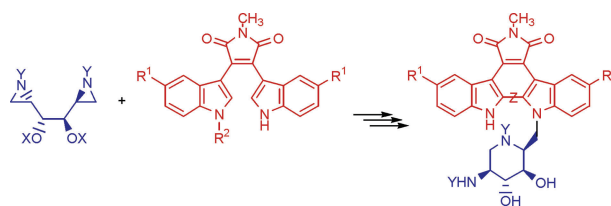


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### Synthesis of new aza-analogs of staurosporine, K-252a and rebeccamycin by nucleophilic opening of $C_2$ -symmetric bis-aziridines

Sandrine Delarue-Cochin and Isabelle McCort-Tranchepain\*

Nucleophilic opening of  $C_2$  symmetric  $N$ -activated bis-aziridines by bis-indolylmaleimides allows access to aminosugar staurosporine, K-252a and rebeccamycin analogs.

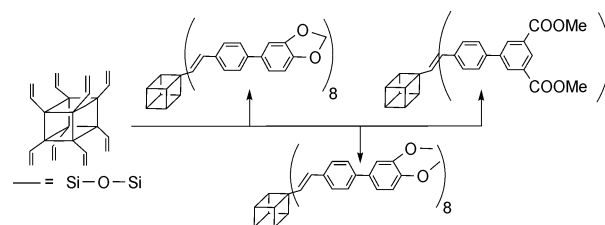


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### Synthesis and characterization of photoluminescent vinylbiphenyl decorated polyhedral oligomeric silsesquioxanes

Nicolas R. Vautravers, Pascal André,\* A. M. Z. Slawin and David J. Cole-Hamilton\*

Octavinylsilsesquioxane has been functionalized via Grubbs metathesis with fluorescent vinylbiphenyl-modified chromophores leading to new light-emitting organic-inorganic nanomaterials.

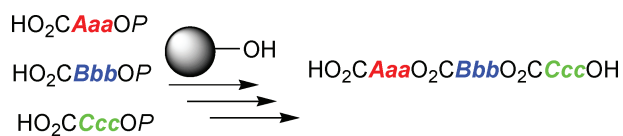


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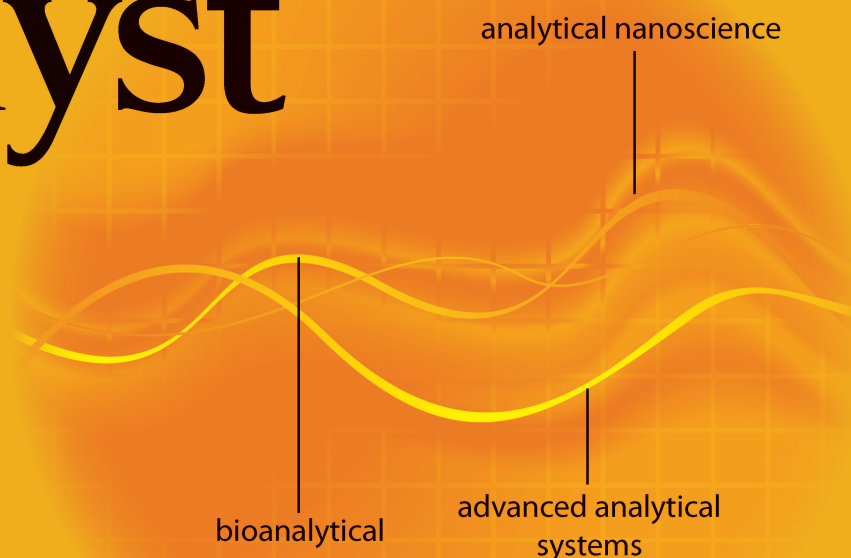
### Solid-phase synthesis of a library of linear oligoester ion-channels

Thomas Murray Fyles\* and Horace Luong

Semi-automated synthesis gives oligoester ion-channel candidates together with minor addition and deletion impurities.



# Analyst



## Bioanalytical Science

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#### Ultrasensitive assays for proteins

Hongquan Zhang, Qiang Zhao, Xing-Fang Li and X. Chris Le  
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#### Electrochemical strategies for the label-free detection of amino acids, peptides and proteins

Grégoire Herzog and Damien W. M. Arrigan  
*Analyst*, 2007, **132**, 615 - 632, DOI: 10.1039/b701472d

#### Microwave-accelerated metal-enhanced fluorescence: application to detection of genomic and exosporium anthrax DNA in <30 seconds

Kadir Aslan, Yongxia Zhang, Stephen Hibbs, Les Baillie, Michael J. R. Previte and Chris D. Geddes  
*Analyst*, 2007, **132**, 1130 - 1138, DOI: 10.1039/b707876e

#### Surface immobilisation and properties of smooth muscle cells monitored by on-line acoustic wave detector

Xiaomeng Wang, Jonathan S. Ellis, Chung-Dann Kan, Ren-Ke Li and Michael Thompson  
*Analyst*, 2008, **133**, 85 - 92, DOI: 10.1039/b714210b

#### Protein-nanoparticle labelling probed by surface enhanced resonance Raman spectroscopy

Phil Douglas, Karen M. McCarney, Duncan Graham and W. Ewen Smith  
*Analyst*, 2007, **132**, 865 - 867, DOI: 10.1039/b707660f

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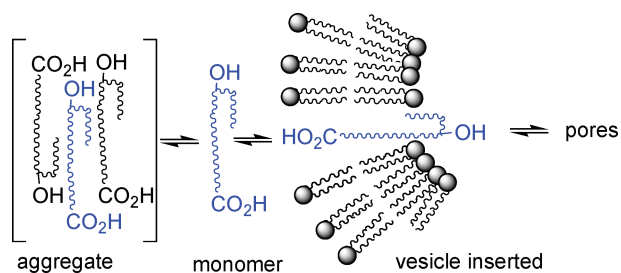


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### Structure–activity relationships in linear oligoester ion-channels

Thomas Murray Fyles\* and Horace Luong

Structure–activity studies reveal the influence of non-polar aggregates on the kinetics of ion channel activity.

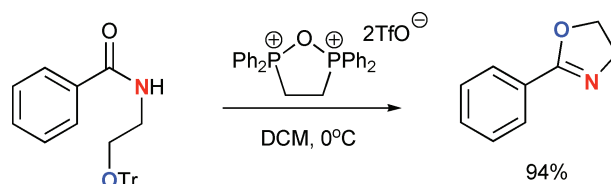


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### The use of phosphonium anhydrides for the synthesis of 2-oxazolines, 2-thiazolines and 2-dihydrooxazine under mild conditions

Maria J. Petersson, Ian D. Jenkins and Wendy A. Loughlin\*

A cyclic phosphonium anhydride can be used to synthesise oxazolines, thiazolines, a dihydro-1,3-oxazine and a tetrahydro-1,3-oxazepine from *O*-trityl and *S*-trityl protected amides, under very mild conditions.

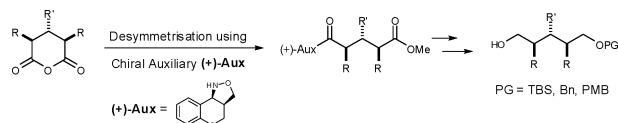


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### Highly diastereoselective desymmetrisation of cyclic meso-anhydrides and derivatisation for use in natural product synthesis

A. C. Evans, D. A. Longbottom, M. Matsuoka, J. E. Davies, R. Turner, V. Franckevičius and S. V. Ley\*

A new and efficient desymmetrisation of succinic and glutaric cyclic meso-anhydrides is described, providing excellent yields and diastereoselectivities. General synthetic utility of the method is established by its application towards a key fragment in the total synthesis of the immunosuppressant antitumour natural product, rapamycin.

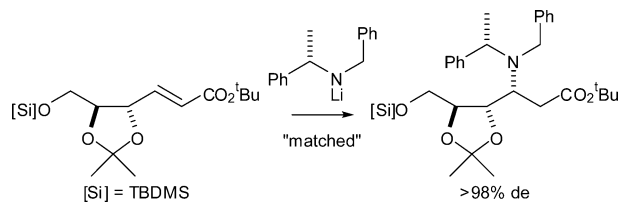


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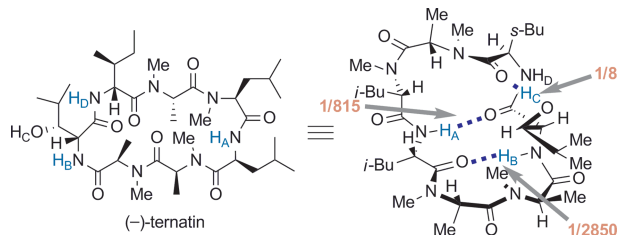
### Doubly diastereoselective conjugate addition of homochiral lithium amides to homochiral $\alpha,\beta$ -unsaturated esters containing *cis*- and *trans*-dioxolane units

Stephen G. Davies,\* Matthew J. Durbin, Euan C. Goddard, Peter M. Kelly, Wataru Kurosawa, James A. Lee, Rebecca L. Nicholson, Paul D. Price, Paul M. Roberts, Angela J. Russell, Philip M. Scott and Andrew D. Smith

The doubly diastereoselective conjugate addition of homochiral lithium amides to homochiral  $\alpha,\beta$ -unsaturated esters has been investigated.



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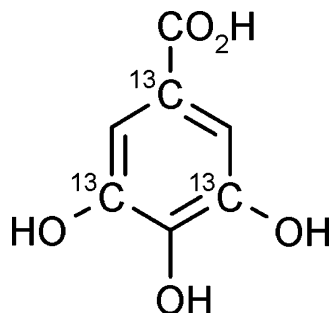


### Importance of the backbone conformation of (-)-ternatin in its fat-accumulation inhibitory activity against 3T3-L1 adipocytes

Kenichiro Shimokawa, Ryoka Miwa, Kaoru Yamada and Daisuke Uemura\*

Key relationships between the intramolecular H-bond-derived backbone conformation and the bioactivity of the novel fat-accumulation inhibitor (-)-ternatin were examined.

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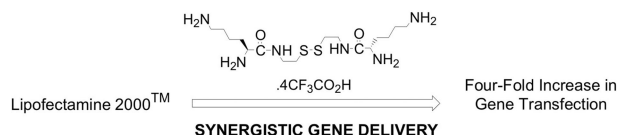


### First synthesis of [1,3,5-<sup>13</sup>C<sub>3</sub>]gallic acid

Laura J. Marshall, Karl M. Cable and Nigel P. Botting\*

The first synthesis of [1,3,5-<sup>13</sup>C<sub>3</sub>]gallic acid from non-aromatic precursors, namely [1,3-<sup>13</sup>C<sub>2</sub>]acetone, triethyl orthoformate and diethyl [2-<sup>13</sup>C]malonate, via the intermediate [3,5-<sup>13</sup>C<sub>2</sub>]4*H*-pyran-4-one, is presented.

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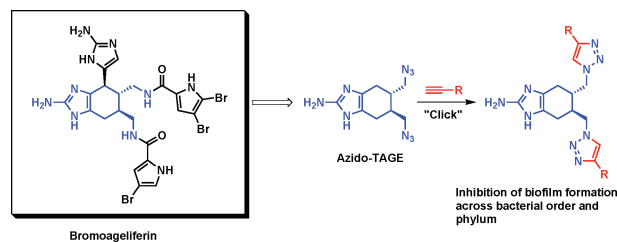


### Synergistic effects on gene delivery – co-formulation of small disulfide-linked dendritic polycations with Lipofectamine 2000™

John G. Hardy, Christine S. Love, Nathan P. Gabrielson, Daniel W. Pack and David K. Smith\*

Mixing low-generation, potentially biodegradable, cysteine-linked dendritic polycations with Lipofectamine 2000™ enhances the gene transfection achieved by the cationic lipid ability four-fold – a clear synergistic effect on gene delivery.

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### Inhibition of *Acinetobacter baumannii*, *Staphylococcus aureus* and *Pseudomonas aeruginosa* biofilm formation with a class of TAGE-triazole conjugates

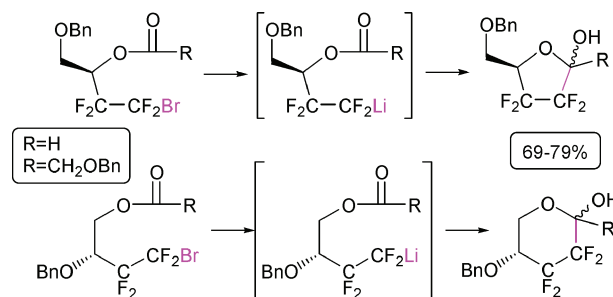
Robert W. Huigens III, Steven A. Rogers, Andrew T. Steinhauer and Christian Melander\*

A focused TAGE-triazole library has been constructed and assayed for the ability to inhibit biofilm formation. From these studies, one of the most potent inhibitors of *S. aureus* biofilms reported has been identified.

## Enantioselective synthesis of tetrafluorinated ribose and fructose

Bruno Linclau,\* A. James Boydell, Roxana S. Timofte, Kylie J. Brown, Victoria Vinader and Alexander C. Weymouth-Wilson

The synthesis of tetrafluorinated aldoses and ketoses, both in the furanose and pyranose forms, is described using a tetrafluoroethylidene lithium mediated cyclisation reaction, in 4 steps overall from a commercially available alkene.



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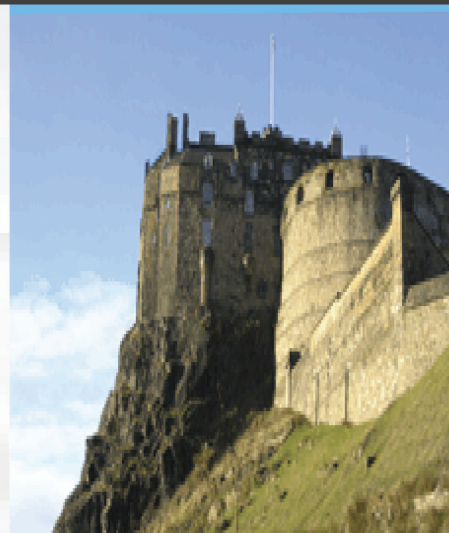
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# Chemical Science

## Biology could provide us with cheap and biodegradable gas-storage materials A natural solution to man-made problems

Nature could provide us with the tools to help us power and clean-up after the cars of the future, according to Italian scientists. The team has shown that amino acid-based molecules can be used to store methane, hydrogen and carbon dioxide efficiently.

Piero Sozzani and colleagues from the University of Milan Bicocca have used nanoporous crystalline dipeptides to absorb and store these three gases. Their simple dipeptide systems are made of natural amino acids – valine and either alanine or isoleucine.

Carbon dioxide levels in the atmosphere are a concern as this greenhouse gas is believed to be a major contributor to global warming. One approach for reducing levels is to capture the gas at major sources of emission (such as fossil fuel power plants) for storage underground. Another route is replacing petrol and diesel in cars with cleaner fuels such as methane and hydrogen. Finding new materials to store these gases safely and economically is a major



**The new Honda FCX Clarity is powered by hydrogen**

field of research. Most studies explore artificial materials, explains Sozzani. He adds that he hopes his team's results will stimulate further research into using biomaterials for gas storage.

Haoshen Zhou, an expert on gas storage materials from the National Institute of Advanced Industrial Science and Technology, Tsukuba, Japan, says 'this work is the first evidence for the applicability of

ultramicroporous biomolecule crystals in gas separation and storage.' He points out that although the hydrogen storage capacity is not particularly high, 'the biocompatible and hydrophobic properties together with the regular pore system and small pore sizes certainly make them a promising group of materials for further research.'

'The rich molecular biodiversity around us could provide an infinite number of other readily available and cheap porous systems,' says Sozzani. 'These materials are intrinsically biodegradable and biocompatible,' he points out.

In addition to just storing gas, the scientists found that the crystalline dipeptides are selective for carbon dioxide over methane. They suggest that the systems could also be used to purify methane by removing carbon dioxide impurities.

*Freya Mearns*

### Reference

A Comotti *et al*, *Chem. Commun.*, 2009, 284 (DOI: 10.1039/b820200a)

## In this issue

### A raincoat that keeps us cool

Scientists uncover a superhydrophobic coating for air conditioning units

### Monitoring melamine in milk

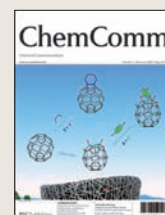
Mass spectrometry outwits dairy fraudsters

### Polymers move smartly

This month's Instant insight introduces polymers that respond to chemical and biological stimuli through movement

### Reaction hero

Fast cars, skydiving, new catalytic concepts...Joanne Thomson asks: are there no barriers to Scott Denmark's adventures?



A snapshot of the latest developments from across the chemical sciences

# Research highlights

Scientists uncover a superhydrophobic coating for air conditioning units

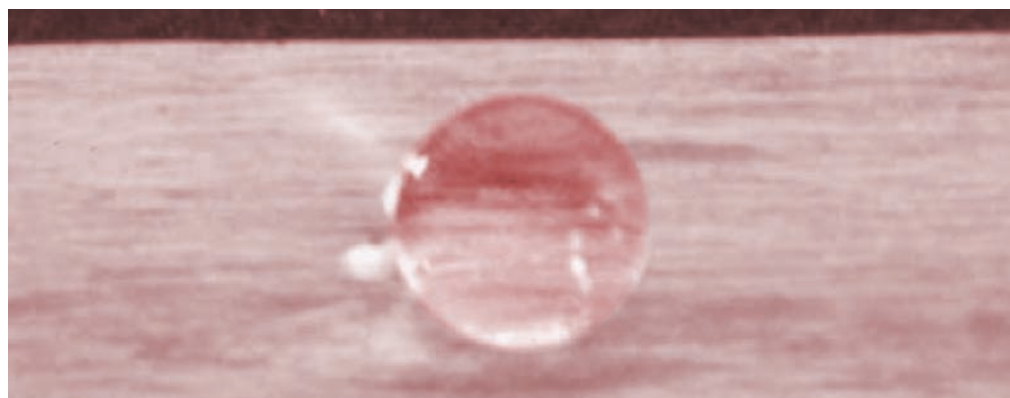
## A raincoat that keeps us cool

A new water repellent coating for aluminium foil could prolong the lifetime of air conditioning units, say researchers in China.

Jian Nong Wang from Tongji University, Shanghai, and Qian Feng Xu from Shanghai Jiao Tong University, have developed a superhydrophobic (very difficult to wet) silica coating that could be applied to the aluminium foil used in air conditioning units – protecting it from corrosion.

Wang and Xu prepare their coating by dipping the foil in a mixture of silica colloid particles and a polystyrene template. The polystyrene is then removed leaving a silica network with a controlled surface roughness.

Currently, superhydrophobic coatings on aluminium are prepared as elaborate nano- or micro-structures, by various methods such as chemical etching. However, chemical etching can harm the aluminium



and decrease the anti-corrosive property.

'Beyond wettability, Wang and Xu have examined other attributes that a practical coating would likely need, such as resistance to aging in air and attack by acidic solutions,' says Chuck Extrand, a specialist in hydrophobic coatings from Entegris Inc, Minnesota, US.

'If the superhydrophobic

**Silica colloid particles coat the aluminium foil**

#### Reference

J N Wang and Q F Xu, *New J. Chem.*, 2009, DOI:10.1039/b817130k

technology is successfully used in the air-conditioning unit, the lifetime of the unit will be greatly prolonged,' says Wang. This technology 'has a bright future in many other industrial fields, such as buildings, electronic devices, and surface protection of metals or alloys used under severe conditions,' he adds.

*Michael Brown*

A new technetium complex has been made for accurate heart imaging

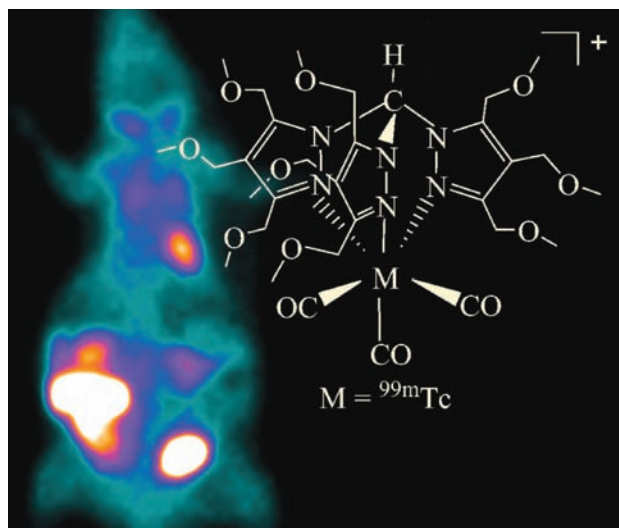
## Complex matters of the heart

A complex for improved imaging of the heart has been developed, which could help detect symptomless heart problems.

Isabel Santos and co-workers at the Institute of Nuclear Technology, Sacavém, Portugal, have made a technetium based complex, using a simple one step reaction, that can be used to image the heart more accurately than the state-of-the-art contrast agent.

The current  $^{99m}\text{Tc}$ -sestamibi complex, used for imaging, is slow to clear the bloodstream and liver which makes it difficult to distinguish the heart from other non-targeted organs and surrounding tissues.

Santos explains: 'due to the high incidence of cardiovascular diseases there is a need for good performing radiopharmaceuticals for rapid and accurate detection' before a heart attack occurs.



Santos's  $^{99m}\text{Tc}$  tricarbonyl complex, with ether functionalisation, clears the blood and liver three times faster than

**This complex may improve early heart disease detection**

the state-of-the-art complex in the clinical trials performed on rats, which allows for a much better contrast image between the heart and the liver, allowing for faster and more accurate diagnoses of coronary artery disease.

Francesco Tisato, an expert in the use of technetium in medical and biological chemistry at the Institute of Inorganic Chemistry and Surfaces, Padova, Italy, says that the significant and persistent heart uptake with favourable clearance of the liver and lungs may help to improve heart imaging.

Santos goes on to say that the next stage of development is to test the complex in clinic trials to confirm its performance in humans.

*Paul Cooper*

#### Reference

L Maria *et al*, *Dalton Trans.*, 2009, 603 (DOI: 10.1039/b817451b)

Mass spectrometry outwits dairy fraudsters

# Monitoring melamine in milk



Two leading groups of mass spectrometrists have applied their expertise to improve melamine detection in milk.

They were responding to the demand for a simple, fast and cheap melamine detection technique after the industrial chemical was found to be present in Chinese milk in September 2008. Tainted milk powders were blamed for the deaths of four babies, and for illnesses affecting tens of thousands of infants.

Melamine, commonly used as a fire retardant and a plastic resin, was added to milk during processing to artificially boost its apparent protein content as assayed by total nitrogen content analysis.

The two new techniques share the advantages of being highly specific, accurate, simple and quick. Both use ambient ionisation – the samples are ionised in their native environment. This means they have potential to be developed into a portable detection kit for use in product quality control. The two groups' techniques differ in the details of the sample ionisation.

Renato Zenobi, ETH Zurich, Switzerland, and colleagues used ultrasound to turn the sample into a fine spray (nebulise) melamine-spiked liquid milk samples; the spray was then ionised by extractive electrospray ionisation mass spectrometry (EESI) and analysed using tandem mass spectrometry.<sup>1</sup> The method is said to take 30 seconds per sample allowing a high sample throughput. The lower limit of detection of melamine is in the range of a few nanograms of melamine per gram of milk.

**Tainted milk powders have been blamed for the deaths of four infants**

Zenobi comments on his technique saying 'ultrasonic nebulisation for EESI sample delivery is extremely simple and extremely rapid, while maintaining a reasonable sensitivity.'

Graham Cooks, Purdue University, West Lafayette, US, and his co-workers used a low temperature plasma probe to ionise their samples and, using the same type of mass spectrometry, achieved similar speeds and limit of detection.<sup>2</sup> The detection limits seen by both groups are well below the minimum level at which melamine becomes toxic to humans.

Cooks says that the existing technique for melamine determination is comparatively complex, 'the newspapers carried extensive discussions on the melamine tampering scandal and reported on the accepted triple quadrupole liquid chromatography–mass spectrometry methodology for its detection. We took it as a challenge to use simpler instrumentation and develop a faster method based on ambient ionisation.'

David Muddiman, professor of mass spectrometry at North Carolina State University, Raleigh, US, describes the techniques as 'marvellous examples of how innovative, direct analysis ionisation methods, when coupled with mass spectrometry have the ability to address contemporary problems facing the world. They have removed all the major obstacles allowing for mass spectrometry not only to compete, but to take the lead in these types of analyses.'

*James Hodge*

## References

- 1 L Zhu *et al*, *Chem. Commun.*, 2009, 559 (DOI: 10.1039/b818541g)
- 2 G Huang, Z Ouyang and R G Cooks, *Chem. Commun.*, 2009, 556 (DOI: 10.1039/b818059h)

## News in brief

### This month in *Chemical Technology*

#### Microthrusters are go!

Electricity boosts space travel on the microscale

#### Uranium exposed

US scientists have developed a way to tell if war veterans have been in contact with depleted uranium

#### Sun shines on a solution for hydrogen production

UK scientists have attached an enzyme and a light-harvesting dye to titanium dioxide particles to make a hydrogen-producing system powered by sunlight

#### Scratching at the surface of biosensors

Justin Gooding discusses how surface chemistry lets porous silicon biosensors fulfil their potential

#### Mixing it up

In this month's interview, Steven Soper talks about interdisciplinary science and a little bit of luck

See [www.rsc.org/chemicaltechnology](http://www.rsc.org/chemicaltechnology) for full versions of these articles

### This month in *Chemical Biology*

#### Spotting the flu virus

Identifying the flu virus in patients could become quicker and easier if a test developed by US chemists becomes commercially available

#### Detecting cancer on the move

Scientists are looking to the elements to detect cancer cells before they spread

#### Chemical connections

Building a protein can be likened to a jigsaw puzzle. Stephen Kent puts the pieces together for us

#### Shining a light on the proteome

In this month's interview, Ben Cravatt talks about the function of the proteome and his success in cloning the cDNA of a hotly pursued enzyme

See [www.rsc.org/chembiology](http://www.rsc.org/chembiology) for full versions of these articles

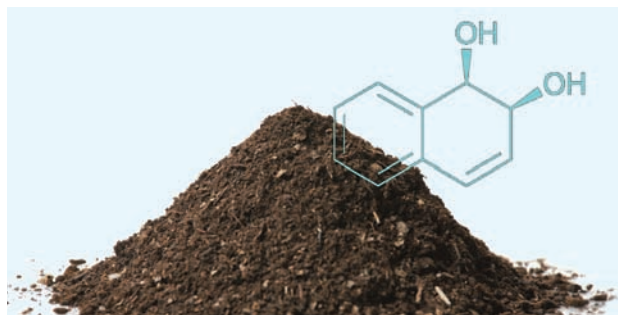
## Bacteria retired after new complex found for degrading aromatic compounds

# Iron complex mimics soil bacteria

Scientists in the US report the first synthetic compound to catalyse a key step in the degradation of double bonds in aromatic rings.

The search for synthetic catalysts which mimic the action of enzymes is something which can enhance areas such as drug discovery, synthetic chemistry and environmental issues. In recent years scientists have explored potential synthetic catalysts which mimic the way in which soil bacteria degrade aromatic compounds.

The natural method for the degradation of aromatic compounds starts with the *cis*-dihydroxylation of an aromatic double bond by non-heme iron enzymes. The best known of these enzymes is naphthalene 1,2-dioxygenase (NDO), which catalyses the conversion of naphthalene to *cis*-(1*R*,2*S*)-1,2-dihydro-1,2-



naphthalenediol. Although synthetic catalysts able to *cis*-hydroxylate olefin double bonds are known, scientists were yet to discover a synthetic catalyst which could carry out the same reaction on aromatic double bonds. Lawrence Que Jr and colleagues from the University of Minnesota, US, have now made a synthetic non-heme iron complex

### *cis*-(1*R*,2*S*)-1,2-Dihydro-1,2-naphthalenediol

#### Reference

Y Feng *et al.* *Chem. Commun.*, 2009, 50 (DOI:10.1039/b817222f)

able to catalyse this reaction.

Que used a complex which had previously been successful in the *cis*-dihydroxylation of olefins,  $[\text{Fe}^{\text{II}}(\text{TPA})(\text{NCMe})_2](\text{OTf})_2$  [where TPA = tris(2-pyridylmethylamine), Tf = triflate]. Using  $\text{H}_2\text{O}_2$  as the oxidant, Que identified four products, the major of which was the *cis*-diol, identical to that produced in the enzyme-catalysed reaction. They also carried out mechanistic studies and found that the process is assisted by water.

Que now aims to further develop the potential for biomimetic catalysis of oxidations that were previously only carried out by enzymes. 'The fact that the catalyst is based on iron, an economical and environmentally friendly metal,' says Que, 'makes it even more relevant for today's societal concerns.' *Richard Kelly*



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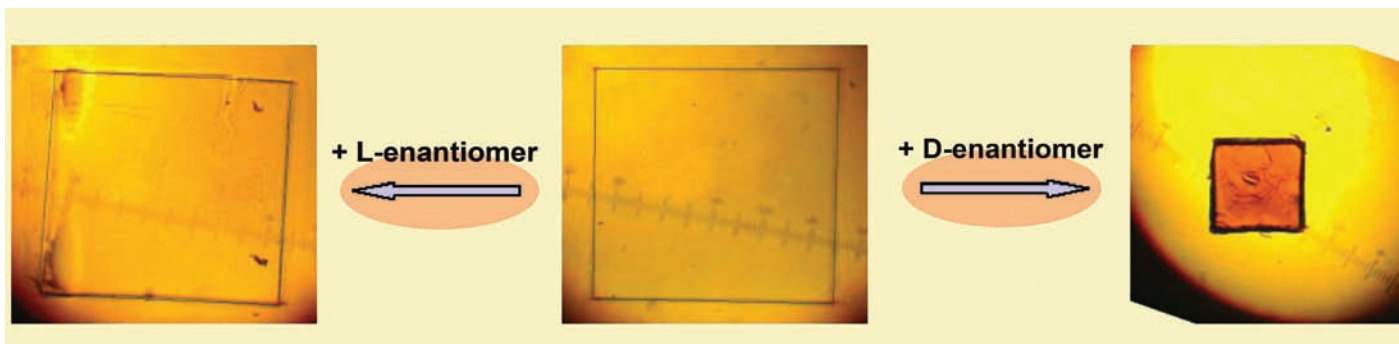
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# Polymers move smartly

Hans-Jörg Schneider and Kazuaki Kato, University of Saarlandes, Germany, introduce polymers that respond to chemical and biological stimuli through movement



Chemomechanical polymers are a new type of smart polymer with specific recognition sites that can respond selectively to chemical and biological stimuli through large movements. They have the unique feature of combining a sensor and an actuator (a mechanical device for moving or controlling a mechanism or system) within one single unit, without the need of external devices such as a transducer or a power supply.

When exposed to chemical or biological stimuli – such as nucleotides, amino acids or peptides – in the local environment these polymers produce large and reversible expansions and contractions. They can also be downsized to thin films or microparticles, with enhanced velocity and sensitivity of response.

Until recently, chemomechanical polymers were only known for responding to rather unspecific changes in pH, salts and solvents. In the last few years the Schneider group has applied known principles of supramolecular chemistry to make hydrogels with suitable recognition sites, leading to materials that respond selectively to external organic stimuli by non-covalent interactions.

A large variety of flexible hydrogels have now been made containing these supramolecular binding sites that can selectively identify specific organic molecule signals being transmitted from the surrounding aqueous environment.

A particular highlight of these polymers is an ability to distinguish between optical isomers. Recently the interaction of chitosan gels and tartaric acid derivatives was for the first time shown to directly translate chiral recognition into large shrinkages of the hydrogel particle. It was shown that exposing a chitosan hydrogel particle to D-dibenzoyltartaric acid caused it to shrink by 94 per cent, but when the same hydrogel was exposed to the L-enantiomer only 20 per cent shrinkage was seen.

The motions of these smart hydrogels are also strongly dependent on the pH of the surrounding environment. And scientists have now taken advantage of this to make hydrogels that can act as simple logic gates, where the motion depends on both the presence of a trigger (such as a nucleotide) and the correct pH.

A related logic gate effect is also seen in polymers with both ester and amine functional groups. A

**Enantioselective contraction of a chitosan hydrogel particle can be induced by L- or D-dibenzoyltartaric acid**

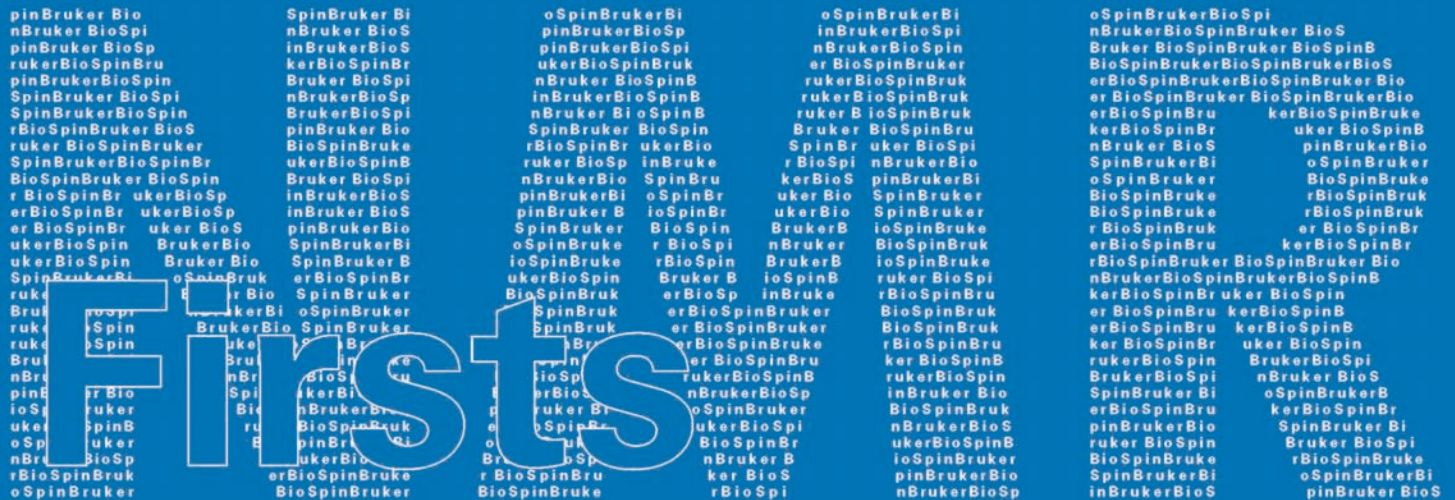
rather spectacular example is a polymethyl(methyl)acrylate-based gel containing ethylenediamine-type binding sites – where the motion induced by amino acids and peptides is only triggered when copper or zinc ions are also present.

It is hoped that these smart materials will find multiple future uses including in systems for controlled drug delivery, in the uptake of toxic compounds, in controlling flow in medical devices and even in microfluidic machineries. Also of interest is putting these smart hydrogels into tubes or onto flexible sheets to make artificial muscles that can translate the energy produced by non-covalent binding of a trigger molecule into mechanical motion.

The future for this research is bright, and it is hoped that the implementation of more sophisticated recognition elements into chemomechanical polymers and a better understanding of the underlying mechanism will lead to even smarter materials.

*Read Hans-Jörg Schneider and Kazuaki Kato's Highlight article 'Molecular recognition in chemomechanical polymers' in issue 5, 2009 of Journal of Materials Chemistry.*

**Reference**  
H-J Schneider and K Kato, *J. Mater. Chem.*, 2009, **19**, 569 (DOI: 10.1039/b814979h)



It's all in the name...

### First for NMR...

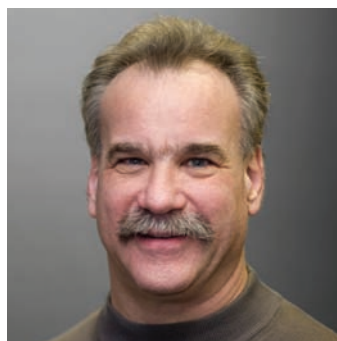
- 1971 World's first commercial FT NMR spectrometer
- 1972 First to deliver high field NMR
- 1983 Automation - first reliable NMR tube samplechanger
- 1994 AVANCE - first digital NMR spectrometer
- 1996 First actively-shielded magnets for HR applications
- 1996 CryoProbe - world's first commercial, cryogenically cooled, high sensitivity probe
- 2000 2nd generation digital hardware
- 2003 UltraShield PLUS magnets at 500 and 600 MHz with 5-gauss line within magnet footprint!
- 2006 700 US+ and 800 US+ compact magnets with enhanced shielding for reduced dimensions
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# Reaction hero

*Fast cars, skydiving, new catalytic concepts...Joanne Thomson asks: are there no barriers to Scott Denmark's adventures?*



**Scott Denmark**

**Scott Denmark is professor of chemistry at the University of Illinois at Urbana, US. His research interests are in structural, synthetic and mechanistic organic chemistry. Scott is a member of the Chemical Communications advisory editorial board.**

**Why did you decide to become a chemist?**

I am one of the children of the Sputnik generation. Chemistry sets were very popular and my parents bought me a Gilbert set when I was eight. I was fascinated by it. I would sit in my basement for hours on end doing experiments. My laboratory grew from that little set to a full blown laboratory when I was in high school. It was at a time when anyone could buy chemicals from chemical stores. I remember carrying bottles of nitric and sulfuric acid on the bus back from a nearby town. It wasn't until I went to university that I learned that you could actually have a career as a scientist in academia.

**Your research is primarily concerned with the invention of new synthetic reactions. What are you working on at the moment?**

It has been a continuous inspiration to find new ways of using the periodic table's versatility to create new kinds of chemical reactivity. I have come up with a fundamental new concept of how one does catalysis. There is a vast world of interesting chemistry that is characteristic of the main group elements that I think has been overlooked in terms of catalysis. I have created a new paradigm for catalysis that is different from transition metal, Lewis acid, enzymatic or organocatalysis. It is going to be uniquely applicable to the interesting chemistry in the main group. We are busily demonstrating our proof of principle for that concept.

**What's hot in organic chemistry?**

Naturally what I am doing! If I didn't think it was hot, then I should not be doing it. Other than that, I find the work of one of my colleagues, Jeff Moore, really fascinating. He is involved in an area called mechanochemistry, which is the use of mechanical energy to induce or control chemical reactions. It is fundamental physical organic chemistry that has tremendous applications in the real world. It could be used to induce materials to repair themselves under stress, which I think is just brilliant.

**What scientific discovery would you like to have been responsible for?**

Pasteur's work on chirality. I would love to have been the person to have made the conceptual connection between molecular dissymmetry and

optical rotation. It was one of the most beautiful experiments in chemistry.

**What are the major barriers to scientific research at present?**

Money, money, money. The overall funding environment in the US is deteriorating. Like never before in my 28 year career, I am feeling real pressure that I am not going to be able to maintain the level of funding that I am used to. I have always enjoyed a lot of industrial support in addition to federal support and that has almost evaporated. Companies are under huge financial pressures and extramural funding is the first to be cut.

In my early days, if asked about what limits me, I would say just my own imagination. But now I have more ideas than I have the money for.

**Do you have any advice for young chemists?**

Be cognisant that you need to create your own identity. In that talent pool of young people, there is no shortage of intelligence, creativity, drive, ambition and willingness to work hard. What is missing from the equation is people asking themselves: 'What problem do I want to solve?' Separate yourself sufficiently from your mentors and think carefully because you're going to invest so much of your own lifetime and energy into your work. You want to make sure that it has impact and will make a real difference and change the way people think about chemistry. You have to really transform the science.

**You are a fan of fast cars and regularly take part in road races. What is it that attracts you to this sport?**

I am very competitive and techy. The combination of engineering, technology and adrenaline is an amazing drug. When I am not racing my car, I am riding one of several very fast motorcycles. I've also jumped out of aeroplanes. I get a real adrenaline rush from speed, danger and challenge. The car has all these features to it but it also is a highly refined skill – it takes a lot of training, discipline and a competitive nature.

**What would you be if you weren't a chemist?**

Naturally, a Formula One race car driver! But, if I could start my career again, I would study neuroscience. I would love to decipher the molecular basis of memory and cognition.

## Journal celebrations

The new year brings a host of celebrations for RSC journals. *Soft Matter* and *Molecular BioSystems* mark their fifth year of publication in 2009 and look back over a catalogue of successes. Since their launch in 2004, both journals have gone from strength to strength, establishing themselves as leading publications in their field. At 4.12,\* the latest impact factor for *Molecular BioSystems* is a sure indication of the significance of the work in this exciting interdisciplinary journal, publishing cutting-edge research at the interface between the -omic sciences and systems biology. *Soft Matter* – as the number one journal in the field for both impact and immediacy – is first choice for fundamental soft matter research.



For *Soft Matter*, 2009 marks a double celebration as – thanks to a continued increase in submissions – the journal moves from publishing 12 to 24 issues a year. What better measure of the journal's success? In fact, 2008 saw journal submissions and acceptances across the whole of RSC Publishing increase by 33% and 29%, respectively. Joining *Soft Matter* in reflecting this achievement, the frequency of two other journals is set to

double in 2009. Leading journal in miniaturisation science, *Lab on a Chip*, also moves to 24 issues: an indication of the significant increase in submissions over the years. Hardly surprising: with an impact factor of 5.1\* *Lab on a Chip* guarantees high visibility and quality research. Review journal *Natural Product Reports* (NPR), with an impact factor of 7.67\*, doubles to 12 issues, meaning you can now get hold of the most topical reviews in key areas even faster, including bioorganic chemistry, chemical biology, natural product synthesis, chemical ecology and carbohydrates.

Make sure you keep up to date with all the latest issues of RSC journals by signing up for our free e-alert service.

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\*2007 Thomson (ISI) Journal Citation Reports

## Virtual collaboration

Articles from selected RSC Journals are now included in the Virtual Journals in Science and Technology series, sponsored by the American Institute of Physics and the American Physical Society. Each of the virtual journals presents an online collection of relevant papers from a broad range of 'source' journals in the physical sciences selected by expert editors.



'We're very pleased that the RSC is contributing to the Virtual Journals, and we're certain that the addition of their publications will make it easier for specialists in the fields covered by the series to stay current with the top-flight research published by the Society,' says Mark Cassar, AIP publisher, Journals and Technical Publications.

Browse the virtual journals at [www.virtualjournals.org](http://www.virtualjournals.org)

## 23 years of devotion

When Jim Harnley joined the *Journal of Analytical Atomic Spectrometry* (JAAS) as the North American Editor in 1985, little did he expect to become the longest serving member of the JAAS staff and editorial board.

After 23 years of service with JAAS, Jim is now retiring from his position but will maintain his association with the journal as a member of the advisory board.

He reminisces on his early days: 'My position was established in an attempt to shorten the manuscript review time. At that time, prior to e-mail, correspondence between



the US and the UK took a week (unless you forgot to put air mail on the envelope, in which case delivery sometimes took two months). Submission and review in the US could shorten the process by up to two weeks.'

'Jim has been involved with

JAAS since the launch of the journal and has contributed significantly to its continuing success. As editor for the Americas, he has been very successful at raising and maintaining the profile of the journal in this region and we would like to thank him for all his hard work,' says Niamh O'Connor, JAAS editor. In 2009 JAAS marks its 24th year of publishing innovative research on the fundamental theory, practice and analytical application of spectrometric techniques to elemental research.

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