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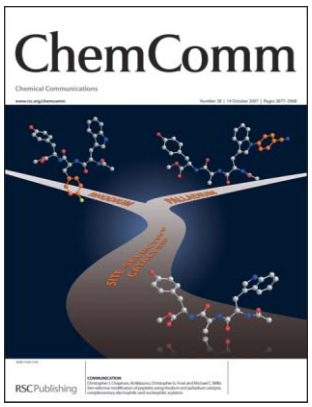
Chiral D- and L-penicillamine capped CdS quantum dots have been prepared. These quantum dots demonstrate circular dichroism (CD) with almost identical mirror images of their CD spectra.
Image reproduced by permission of Mícheál P. Moloney, Yurii K. Gun'ko and John M. Kelly from *Chem. Commun.*, 2007, 3900.



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The site-selective arylation of individual amino acid residues on a peptide is achieved using different transition-metal catalysed reactions.
Image reproduced by permission of Christopher J. Chapman, Ai Matsuno, Christopher G. Frost and Michael C. Willis from *Chem. Commun.*, 2007, 3903.

CHEMICAL TECHNOLOGY

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

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October 2007/Volume 4/Issue 10
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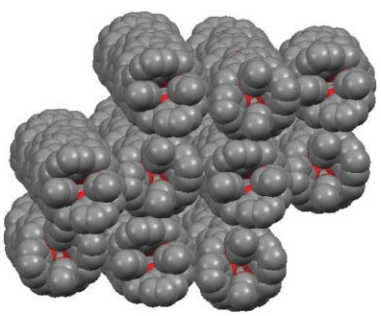
FEATURE ARTICLE



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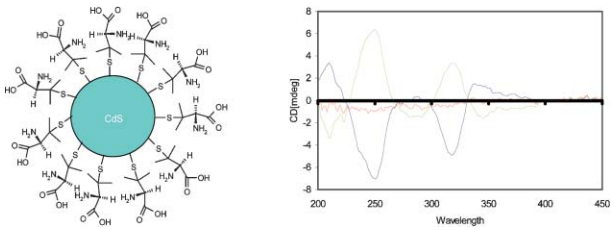
3900

Chiral highly luminescent CdS quantum dots

Mícheál P. Moloney, Yurii K. Gun'ko* and John M. Kelly

Strongly white emitting chiral D- and L- penicillamine capped CdS QDs have been prepared. Using CD spectroscopy we have shown that they are optically active with almost identical mirror images of one another in the range of 200–390 nm CD scans.

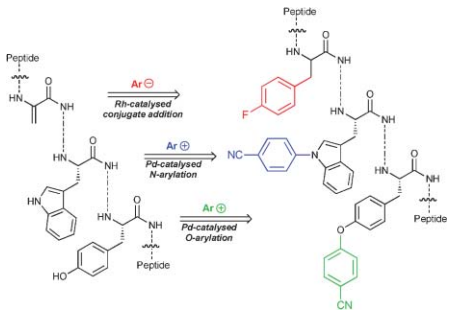


3903

Site-selective modification of peptides using rhodium and palladium catalysis: complementary electrophilic and nucleophilic arylation

Christopher J. Chapman, Ai Matsuno, Christopher G. Frost* and Michael C. Willis*

A unique site-selective modification of a peptide containing dehydroalanine, tyrosine and tryptophan residues using rhodium catalysed conjugate additions or palladium catalysed aryl-amination and -etherification reactions is described. The selectivity is controlled by choice of metal and ligand.

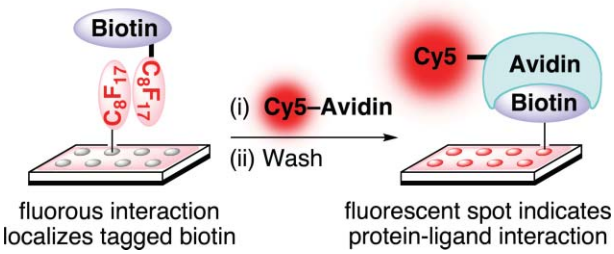


3906

Fluorous tagged small molecule microarrays

Rebecca L. Nicholson, Mark L. Ladlow and David R. Spring

The non-covalent fluorous interaction between fluorous tagged small molecules and a fluoroalkyl modified glass surface was shown to facilitate the detection of protein–ligand binding interactions in the fabrication and screening of small molecule microarrays.

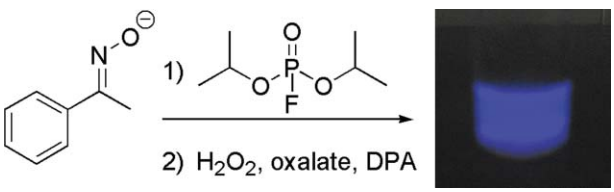


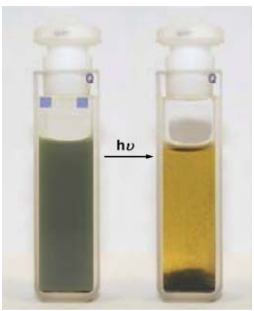
3909

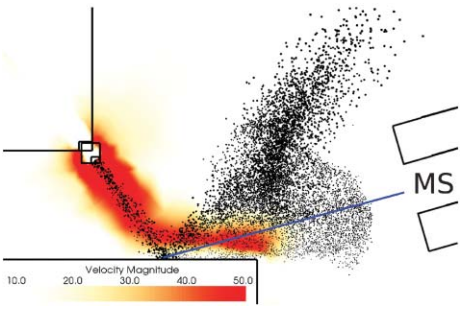
Novel chemiluminescent detection of chemical warfare simulant

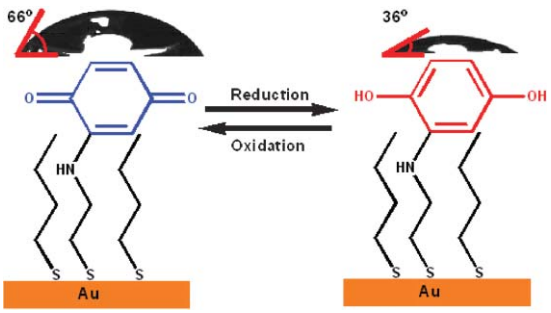
Himali S. Hewage, Karl J. Wallace and Eric V. Anslyn*

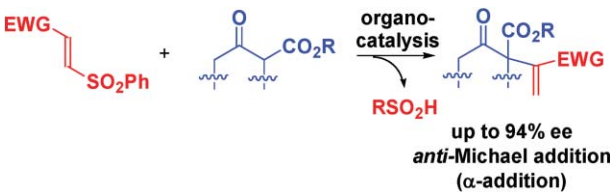
The authors describe a chemiluminescent methodology for the detection of the sarin/soman nerve gas analog DFP, which involves the creation of a super nucleophile that reacts with one of the reagents required in the chemiluminescence mechanism. However, this same super nucleophile also intercepts the sarin/soman analog, thereby rendering its ability to silence the glow mute; and hence in the presence of the nerve agent a glow occurs.

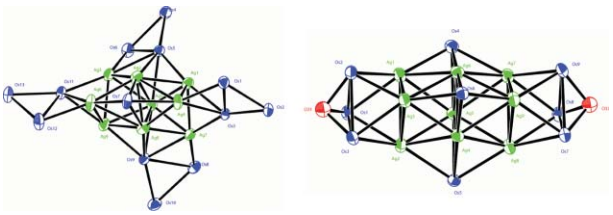


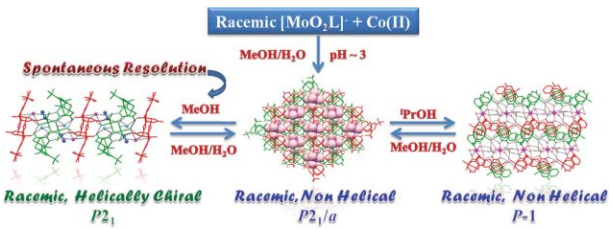


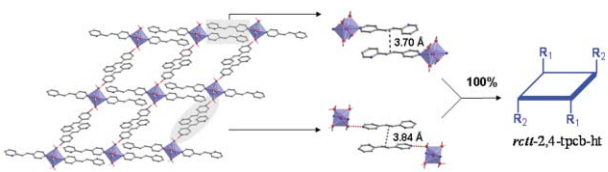


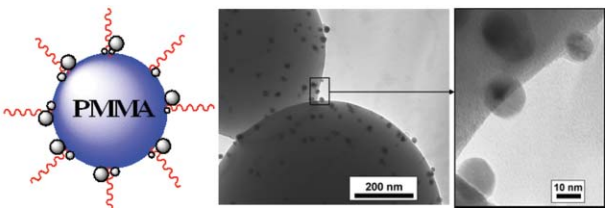


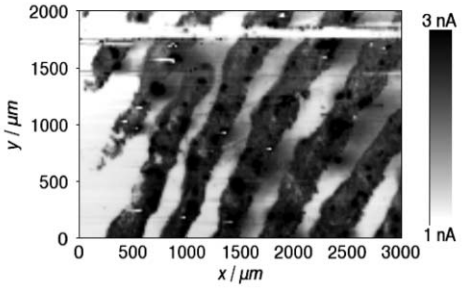


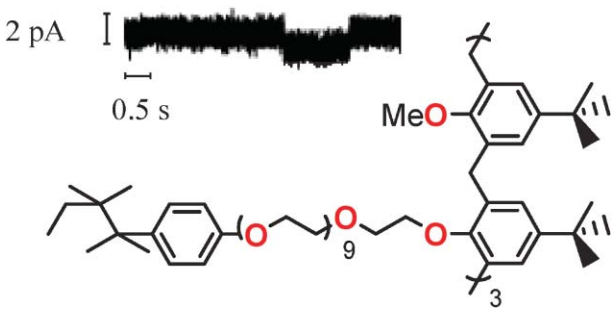


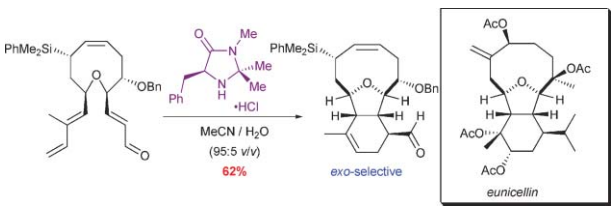


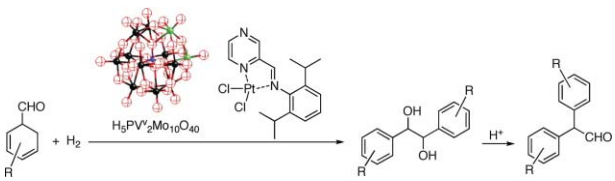




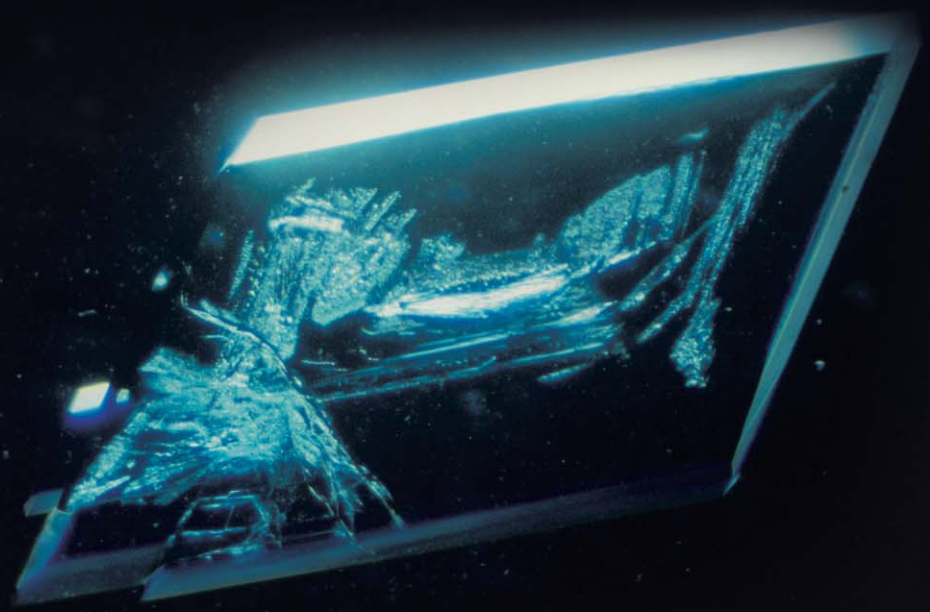


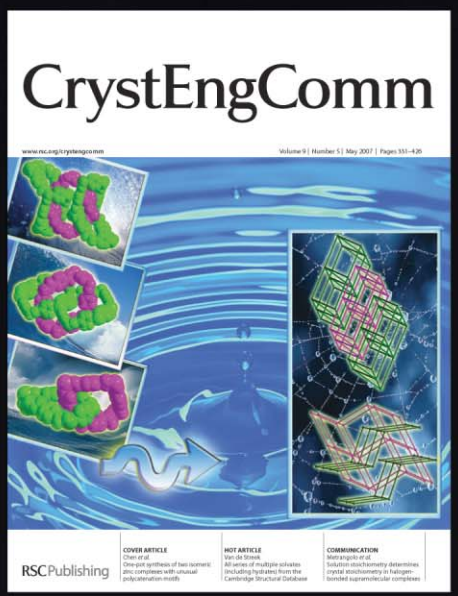






Crystallising all the best research





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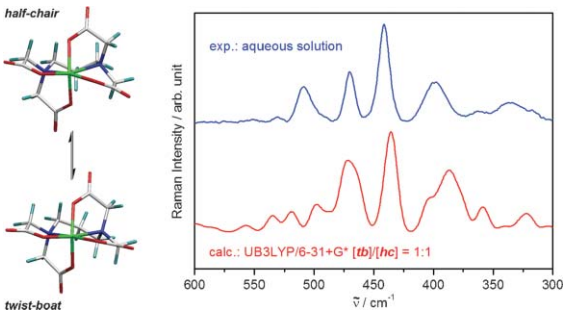

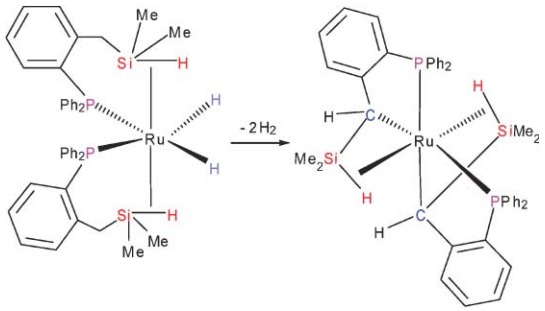

ADDITION AND CORRECTION

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Chemical Technology

Simply measuring particle size gives a good estimate of risk

Sizing up the danger of volcanic ash

Analysing the grain size of volcanic ash particles might provide a quick and easy way to calculate their potential threat to human health, according to a British scientist.

Although the primary hazards of a volcanic eruption, such as pyroclastic flows, are of major concern after a volcanic eruption, secondary hazards, such as the short term and long term effects of the dust and ash ejected, are also a major problem. Volcanic ash may cause acute respiratory diseases and has the potential to cause chronic diseases, such as lung cancer. But assessing this risk can be difficult, as conventional medical studies may take months, years or even decades to conclude whether a dust is toxic or not.

What is certain, however, is that the ash cannot be harmful if the particles are too large to enter the lung. Therefore, Claire Horwell at the Institute of Hazard and Risk Research at Durham University developed a method that allowed her to estimate the amount of fine particles in volcanic ash without needing state of the art instruments.

PHOTODISC

Ash and dust can cause chronic and acute respiratory diseases

By characterising the grain size distribution of volcanic ash after eruptions, Horwell developed an equation for estimating the quantity of health-pertinent fractions when state-of-the-art techniques are unavailable. By focussing on techniques that allow cheap and quick assessment of the health hazards posed by volcanic emissions, Horwell hopes to provide hazard managers with a new tool to rapidly assess how bad an eruption is for the health.

'At the onset of future eruptions, local scientists can simply sieve the ash and immediately calculate the percentage of ash that is fine enough to enter the lung. This means that a preliminary assessment of the potential health hazard can be carried out in a matter of minutes rather than waiting for laboratory results. Hazard managers can then rapidly decide whether to distribute dust masks to a population or even to evacuate an area until ash fall has ceased,' explained Horwell.

Further research is needed to establish what actual health risk volcanic ash might pose. The biggest challenge, however, is bringing together scientists from across several disciplines (such as medics, mineralogists or toxicologists) to definitively determine the health risks of volcanic ash. In the meantime, Horwell's technique may help hazard managers make more informed decisions.

Edward Morgan

Reference
C Horwell, *J. Environ. Monit.*, 2007, DOI: 10.1039/b710583p

In this issue

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

Application highlights

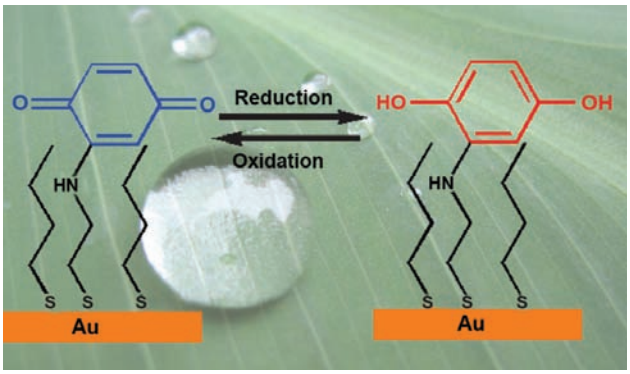
Smart surface changes hydrophobicity at the flick of a switch

Wet, not wet, wet

Bored of bath-cleaning? Wearied by window-washing? Thanks to a group of scientists in Israel these mundane chores could soon be consigned to the past.

Itamar Willner and colleagues at the Hebrew University of Jerusalem have made a quinine-coated 'smart surface' whose wettability can be switched using either an electrical or a chemical trigger. Such surfaces are inspired by self-cleaning systems in nature. The lotus leaf, for example, has a hydrophobic surface that allows water droplets to roll off the leaf, removing dirt from its surface.

Willner and his team coated a gold surface with hydrophobic benzoquinone, which is reduced to hydrophilic hydroquinone using either an applied voltage or a chemical reducing agent. Hydroquinone has two hydroxyl groups that interact strongly with the water, causing the surface to become 'wetter' when reduced.



Benzoquinone (blue) repels water, but the reduced hydroquinone (red) does not

'The hydroquinone/benzoquinone surface evolved from a much more complex system that, after a lot of effort, did not work at all. We were still surprised with how robust the [simple] system [was] and how dramatic the changes were that we observed,' said Willner.

The team's smart surface is easily prepared and the small size of quinone means that a lot of molecules can be packed into the surface, causing large macroscopic changes in the surface wettability.

Aside from self-cleaning applications, these clever surfaces may be used in microfluidic devices that could provide new analytical procedures for clinical diagnostics. For example, if the inner part of a capillary was coated with a conductive film functionalized with Willner's quinine monolayer it could be used to suck minute volumes of fluid from cells or organs.

'A smart idea to create a smart surface,' said Jilie Kong, a microfluidics expert at Fudan University, Shanghai. 'The reversible change of hydrophobicity/hydrophilicity is promising in the design of novel microfluidic chips or biosensors,' said Kong.

Ruth Doherty

Reference
A Wieckowska, A B Braunschweig and I Willner, *Chem. Commun.*, 2007, DOI: 10.1039/b710540a

Ultramicroelectrode probes coated by CVD to give uniform layer

Diamonds are for electron microscopy

A team of UK scientists have developed a way of controlling the chemical vapour deposition of diamond to uniformly coat ultramicroelectrodes. These electrodes often have a diameter of less than 25 microns, ideally suited for scanning electrochemical microscopy (SECM).

SECM is a type of microscopy that images surfaces using a physical probe. By mechanically moving the probe across a surface, an accurate image of the surface can be mapped. The electrodes that are used as probes are known as ultramicroelectrodes (UMEs).

Diamond is an attractive material for electrodes because of its electrochemical characteristics and chemical inertness. However, up until now diamond UMEs could not be made using conventional chemical vapour deposition techniques. The team led by John Foord at the University of Oxford successfully modified the deposition process to produce diamond-coated ultramicroelectrodes. By combining efficient nucleation methods and an electrochemical bias during growth, the electrode can be coated with a uniform, polycrystalline diamond layer.

The team have been working in the area of diamond electrochemistry for a number of years and aim to produce diamond electrodes for probing biological media. Foord explained the challenge: 'Conventional microelctrodes fail to function in biological media because of adsorption of biological media, which foul the electrode,' he said. Diamond can be chemically functionalised to make it more stable than other electrodes under these conditions, he said, making it an ideal target material.

Diamond's chemical properties make it ideal for use as electrodes

Frank Marken from the University of Bath is an expert in novel electrode design and believes this work could have a wide impact. 'The availability of sharp, conducting diamond tools could be of wider significance... tools to "dissect" and image individual biological cells and their content could be envisaged as future developments,' he said.

However, Foord believes that more work is needed before the full potential of these ultramicroelectodes is realised. A thin insulating coating for the body of the electrode is needed, which can be removed from the electrode tip without damaging it. The group are currently exploring the use of polymer coatings for this purpose.

May Copsey

Reference
J Hu, J S Foord and K B Holt, *Phys. Chem. Chem. Phys.*, 2007, DOI: 10.1039/b710241k

Electrothermal vaporisation and ICPMS combine to detect radionuclides

Finding fission by-products

Researchers in Canada have developed a method to rapidly measure ultra-trace amounts of strontium 90 (^{90}Sr) in environmental samples.

^{90}Sr is a by-product of the fission of uranium and plutonium in nuclear reactors and nuclear weapons, with a half-life of 29 years. It was widely dispersed into the environment during nuclear weapons testing in the 1950s and '60s. It is chemically similar to calcium so it accumulates in bone and blood-forming tissue. Exposure to ^{90}Sr may be linked to certain cancers.

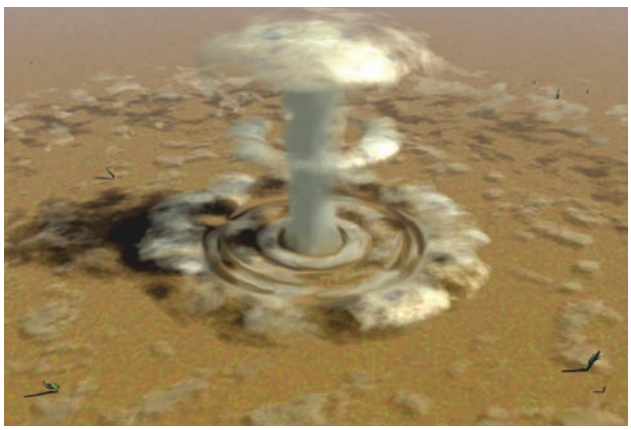
Patricia Grinberg and coworkers at the Institute for National Measurements Standards in Ottawa used ICPMS combined with electrothermal vaporisation and dynamic reaction cell technology to measure ^{90}Sr concentrations as low as 3.5 picograms per gram. The method was tested by measuring natural Sr in marine sediments, river water and biological material and the recovery of ^{90}Sr spikes added to the samples.

'The determination of ^{90}Sr is non-trivial both as a consequence of its extremely low concentration levels and because it suffers from interferences, frequently necessitating laborious separation procedures, which can cause contamination and increase the analysis time,' said Grinberg.

Grinberg's method takes advantage of the ability of electrothermal vaporisation systems to use thermal programming to selectively remove interferences and simplify sample preparation procedures.

The work is part of a project financed by the Chemical Radiological and Nuclear Research Technology Initiative (CRTI) in Canada on the development of new technologies for the rapid detection and identification of radionuclides.

'Radionuclides have traditionally been measured using radiometric techniques which are not ideally suited to rapid and/or accurate determination,' said Grinberg. 'Rapid determination is crucial in the event of a nuclear or radiological accident so as to assess and minimize adverse health, economic and environmental effects,' she continued. *Joanna Stevens*



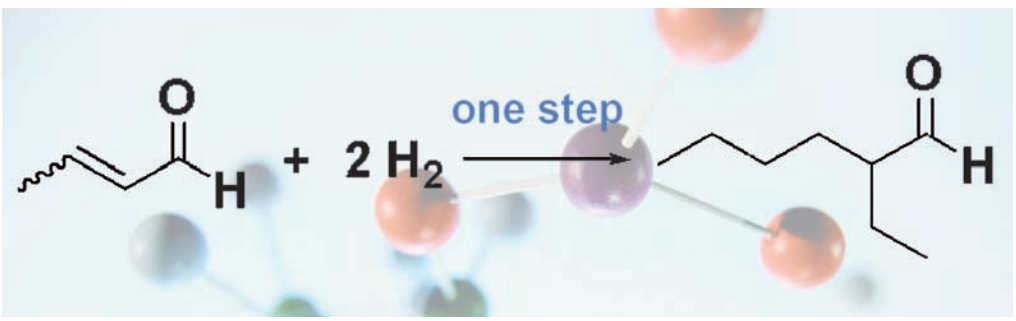
ISTOCKPHOTOS

Strontium 90 was dispersed into the atmosphere during nuclear testing

Reference
P Grinberg, S Willie and R E Sturgeon, *J. Anal. At. Spectrom.*, 2007, DOI: 10.1039/b708018b

Carbon dioxide solvent and palladium catalyst improve synthesis

Organic synthesis goes supercritical



Swiss chemists have developed a greener and more efficient way of making an industrially important aldehyde.

2-Ethylhexanal is a key compound in the manufacture of perfumes and paints. Until now, its large-scale synthesis was dogged by the need for several consecutive reaction steps, poor yields or the need for large excesses of certain reagents.

Alfons Baiker and his colleagues at the Swiss Federal Institute of Technology (ETH) in Zürich have now developed a method of making it in a highly selective way and in a single step from crotonaldehyde (but-2-enal) using only a small excess of hydrogen.

Supercritical carbon dioxide (where it is in a fluid state that is mid-way in character between a gas and a liquid) at 60°C and 16 MPa pressure is used as the reaction medium for the starting materials, and the reaction takes place by passing it over a palladium catalyst at a constant flow rate.

According to Baiker, 'the catalyst exhibits high activity, selectivity and a long lifetime', adding that the only by-product, butyraldehyde, is also industrially useful and easily separated by distillation.

'The ... method ... may provide a promising alternative for the industrial production of [these] two important aldehydes,' he said.

Martyn Poliakoff, a fellow of clean technology at the University of Nottingham and chair of Green Chemistry's editorial board, considers Baiker's work an 'interesting development'. He added that he saw 'considerable potential for [the development of] sustainable chemical processes'. *David Parker*

One step ahead: previous routes have several steps or give poor yields

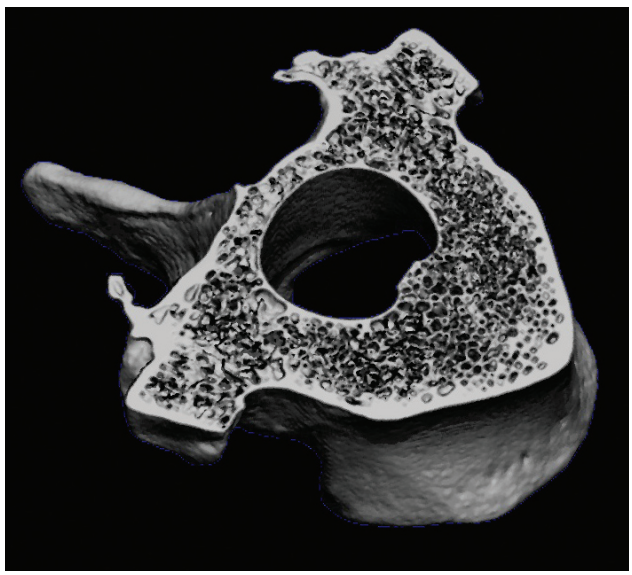
Reference
T Seki, J-D Grunwaldt and A Baiker, *Chem. Commun.*, 2007, DOI: 10.1039/b710129e

Reference materials allow more localized investigation of bone density

The phantom of the bone scanners

Research by Swiss scientists could open the way to better diagnosis and treatment for osteoporosis sufferers.

Susanne Schweizer and colleagues at ETH Zurich in Switzerland have synthesised reference materials – or 'phantoms' – that allow micro-computed tomography (μ-CT) to be used for measuring mineral content of individual bone struts. Scans currently used to diagnose osteoporosis measure the mineral density of bones over a relatively large area. Knowledge of the mineral density of patient's bones on a more local scale should aid the diagnosis of osteoporosis at an earlier stage, and help determine the most appropriate treatment, explained Schweizer.



Micro-computed tomography can already image bone in 3D

μ-CT is a well established research technique for studying bones in three dimensions. Previous attempts to use it in accurate measurements of bone density were hindered by a lack of good phantoms needed to calibrate the machine for this purpose, explained Schweizer. She has now overcome the hurdles, making phantoms that properly mimic the way bones reflect X-rays from the scanner.

Schweizer said that these phantoms are already being used in μ-CT studies of live rodents, with clinical applications being the end goal.

Nina Athey-Pollard

Reference
S Schweizer *et al*, *Analyst*, 2007, DOI: 10.1039/b703220j

Keeping catalysts apart enables them to perform at their best

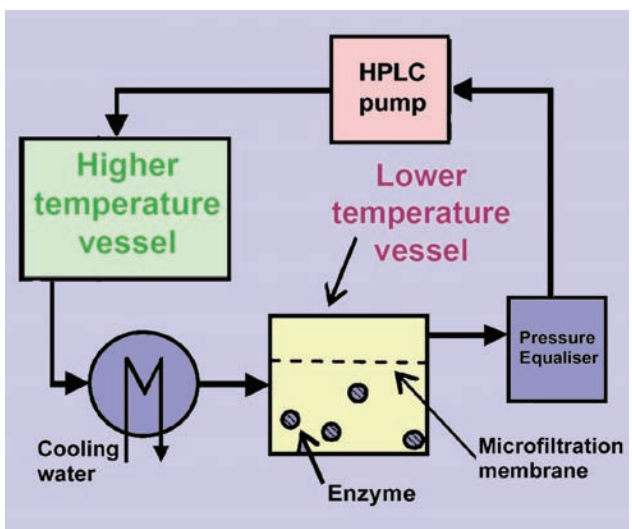
Membranes do the trick

Researchers in the UK and New Zealand have shown that using a membrane could help catalysts operating in the same system work more efficiently.

The team, led by Paul Taylor at the University of Warwick and Andrew Livingston at Imperial College London, used a membrane to keep catalysts in environments where they work best.

Taylor explained that in a process where two or more catalytic steps are combined in one operation, called a tandem catalytic process, the catalysts normally have to compromise on their performance. This is because the same operating conditions are imposed on both catalysts. 'We use technological tricks to avoid the compromise,' he said, 'and allow the catalysts to operate under their respective optimum conditions, while in terms of the process they are in the same synthetic operation.'

The team used the membrane in a tandem catalytic process called dynamic kinetic resolution, a process used to make enantiomerically enriched products. Jonathan Williams, professor of organic chemistry at the University of Bath, explained that, although there are many opportunities for using catalysts in tandem catalytic processes, there are practical problems associated with their use because of the different conditions they require. 'These researchers have provided an elegant solution to this problem by using a membrane to retain an enzyme catalyst in a lower temperature vessel whilst metal-catalysed racemisation occurs in a higher temperature vessel, leading to an effective dynamic kinetic resolution process,' he said.



The enzyme stays in the cold while the metal catalyst feels the heat

The partnership involved collaboration between chemists interested in tandem catalysis and chemical engineers interested in membrane technology. Taylor explained that the collaboration resulted from effective networking with colleagues in industry interested in membrane separation.

Katherine Davies

Reference
C Roengpithya *et al*, *Chem. Commun.*, 2007, DOI: 10.1039/b709035h

Instant insight

Science and art in harmony

Marc Aucouturier, Centre for Research and Restoration of the Museums of France, and Evelyne Darque-Ceretti, Paris School of Mines, illustrate the benefit of a multidisciplinary approach to preserving our cultural heritage

The understanding and preservation of cultural heritage has been a major challenge for all civilisations. The most modern tools of materials science are rising to the challenge. Knowing the composition and rebuilding the history of an artefact needs sophisticated laboratory instruments and a long enquiry involving intensive collaboration between materials scientists and art historians. Data on the physical constitution of an artefact, its authenticity, its history, the circumstances of its discovery, its treatment after being abandoned and/or stored, are useful not only to increase our knowledge of civilisation and art history, but also to inform conservation policy.

The study of cultural heritage artefacts and their preservation and restoration begins with – and is often limited to – a comprehensive characterisation of their surface by non-destructive methods. The application of surface science to cultural heritage materials has undergone a dramatic development in the past few decades, thanks to the impressive improvement of analysis and investigation equipment.

One example of objects recently submitted to in depth investigation are beautiful ancient ceramics with a metallic lustre decoration. This technique was born in the 9th century in factories created by the Arabs during their conquests in Mesopotamia, Egypt and Persia. With the passing of the centuries, Arab potters spread their know-how all over the Islamic world. It reached Spain and was finally transmitted to the workshops of the Italian Renaissance at the end of the 15th century, giving rise to what is known as majolica ceramics.

All that glitters is not gold: the iridescent sheen on this pottery is the work of ancient nanoscientists.

Lustred ceramics attracted the attention of conservators and scientists on account of the structure of the thin surface film that is responsible for their specific aspect. They exhibit an iridescent shine that sometimes imitates a gold, silver or copper deposit in specular reflection and appears from deep red to bright yellow by diffused light observation. In order to understand this very sophisticated technique and trace its propagation through the ages, a series of investigation were conducted in several materials science laboratories. They showed that the surface film is made of vitreous matter in which nanoparticles of metallic silver and/or copper are embedded. In other words, ancient Islamic potters invented nanotechnology eleven centuries before our solid state physicists.

Another example of an ancient decoration technique that is still not fully understood is 'black bronze'. It is the result of intentional surface treatments by ancient craftsmen in order to change the surface aspect and colour of bronze artefacts. It was discovered on bronze artefacts from Egypt (2nd millennium BC), the Roman Empire (1st century AD), China (4th century AD) and Japan (14th century AD). It consists of a chemical treatment applied to specific copper alloys containing always gold (1 to 8 wt%) and/or silver (1 to 4 wt%) in order to give them a black or velvet colour. A comprehensive study has been conducted recently on a large body of Egyptian and Roman Empire pieces from the Louvre museum in France. Most of the black patina appears to be pure cuprite, Cu_2O, containing small amounts of gold and/or silver. The natural colour of cuprite is red, and the research on the role of the precious metals on its black coloration is still under investigation.

Many other examples could be given, such as the fruitful application of Raman spectroscopy to the identification of pigments and the restoration of painted artefacts, the use of analytical spectroscopy to organise a preservation policy for the Swedish warship Vasa, the study of environmental degradation of medieval stained glass windows by secondary ion mass spectroscopy or the identification of degradation mechanisms of old paper manuscripts by spectrophotometry and atomic force microscopy.

Read the full Tutorial Review 'The surface of cultural heritage artefacts: physico-chemical investigations for their knowledge and their conservation' in issue 10 of Chemical Society Reviews.

Reference
M Aucouturier and E Darque-Ceretti, *Chem. Soc. Rev.*, 2007, **36**, 1605, DOI: 10.1039/b605304c

Interview

Chemical conservation

David Saunders explains to Joanne Thomson how chemistry can be used to preserve ancient artefacts

David Saunders

David Saunders is Keeper of the Department of Conservation, Documentation and Science at the British Museum, London. His research focuses on the scientific examination of artefacts, principally using non-destructive imaging and spectroscopic techniques.

What inspired you to develop a career in the analytical sciences?
I was inspired to specialise in chemistry by a teacher at school whose enthusiasm for the subject caught my imagination. I have also had a longstanding interest in museums since my father took me to them as a child.

When I was coming to the end of my postgraduate studies, I was very interested in the way that science is applied to the arts and archaeology. When I was doing post doctoral work, I saw a job opportunity at the National Gallery in London and that was really how I started in the field.

History and not chemistry springs to mind when most people think of museums. How big a role does chemistry play at the British Museum?
Chemistry is involved in two aspects of the museum's activities. Firstly, it is used in the preservation and restoration of the collections. Secondly, through the chemical analysis of the objects, we bring another aspect into their interpretation that augments the history side. We can shed light on how objects were made, what they're made of and how cultures have changed, developed and traded. Increasingly, we're finding the public are engaged by this type of information.

What techniques do you use to examine artefacts?
We begin by using simple microscopy to magnify an object. We then use scanning electron microscopy (SEM) to look at objects at much higher magnification. With this we have the potential to conduct energy dispersive X-ray analysis (SEM-EDX), which is an absolute workhorse for the department. This can give us some elemental information.

We also use other X-ray techniques like X-ray fluorescence and diffraction and X-radiography. X-Radiography is very important – we use it routinely to look inside objects that we can't otherwise look in.

We use various spectroscopic techniques, including Raman spectroscopy. This has been the big new thing for museums in the last 10–15 years, particularly Raman microscopy, where the Raman is directed through the optics of a microscope. The fingerprinting of various materials by Raman has become of sufficient significance that the organisation that was putting together the infrared library of museum-related materials has extended it into Raman.

What is the most rewarding aspect of your work?
It's adding to the sum total of something that's known about an object. Preservation is, of course, enormously important but it is the intellectual enquiry that I find most rewarding. I enjoy using the documentary sources together with the knowledge of specialist art historians and archaeologists to piece together what the science is telling us about the object with what we know about the object from other sources.

Do you have a message for young scientists?
Don't get too narrow. Read around; look at what is happening in other fields. You can't be an expert in everything but you can have a broader knowledge and that's when you start to see the interconnections. Bringing in the best of other fields is a terribly important facet of how scientists can think within an organisation.

Which scientist do you most admire and why?
Michael Faraday, because he was heavily involved in the development of science in museums and galleries in Britain. Everyone knows Faraday for his very straight scientific work but he got involved in everything. He was involved in the commission that looked at relocating museums out of London in the mid-19th century because of fears about pollution damage to the collections. Although he was engaged in very serious study in a single area, it didn't stop him retaining that breadth.

If you weren't a scientist, what would you be?
I have always regretted giving up history at such an early stage. I would quite like to be a historian. If I couldn't give up the science completely, perhaps I'd end up doing history of science. Maybe I would go the whole way and become an art historian or an archaeologist, although the idea of being knee deep in mud in an archaeological site doesn't necessarily appeal!

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Essential elements

And the winner is...

Left to right - Richard Kidd, Richard Gedye and Bernard Donovan

Months of hard work were rewarded recently as *RSC Project Prospect* was named as winner of the 2007 ALPSP/Charlesworth Award for Publishing Innovation.

In making the award, which recognises a significantly innovative approach to any aspect of scholarly publication, the judges described *RSC Project Prospect* as 'the clear winner ... journals incorporate standard metadata within the full text of articles and combine this with an elegant and intuitive on-screen manifestation of the advantages of including this metadata. As a result, sophisticated and effective searching of the literature is greatly improved and the value gained from reading each article is significantly enhanced. It is delightfully simple to use and benefits to authors and readers are immediately obvious.'

Receiving the award at the ALPSP Annual Dinner in London on September 13th, project manager Richard Kidd declared: 'RSC Publishing is proud to win the 2007 award, which is great recognition for the work our publishing staff and academic partners have put into the development and evolution of *Project Prospect*.'

This is the first time that RSC Publishing has received the award for publishing innovation, and staff are understandably delighted.

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