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Interdisciplinary detection science

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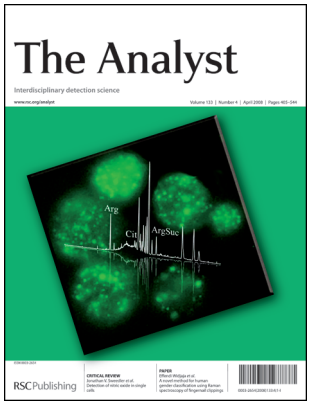

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The cover shows sensing nitric oxide production in individual PC12 cells with fluorescence microscopy and single cell capillary electrophoresis.

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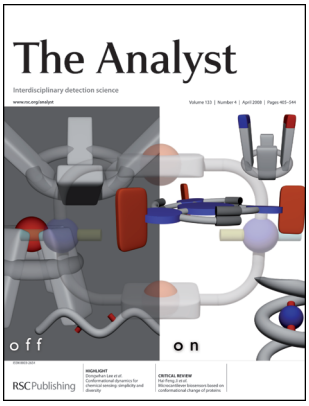

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A schematic rendition showing analyte-induced structural transition of selected shape-adaptive chemical sensors. Molecular symmetry simplifies the energy landscape of conformational switching, which serves as the key signal transduction mechanism shared by these dynamic constructs.

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CHEMICAL TECHNOLOGY

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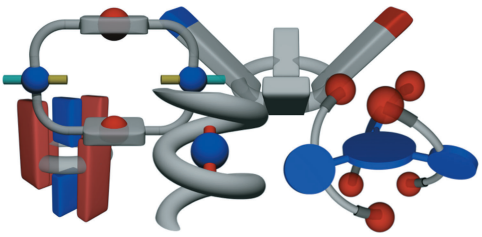

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COMMUNICATIONS

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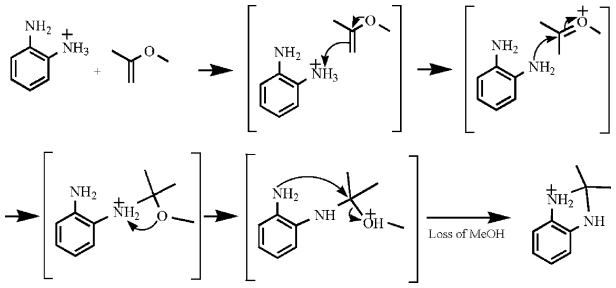
Regioselective ion–molecule reactions for the mass spectrometric differentiation of protonated isomeric aromatic diamines

Mingkun Fu, Penggao Duan, Sen Li, Steven C. Habicht, David S. Pinkston, Nelson R. Vinueza and Hilkka I. Kenttämaa*

Regioselective ion–molecule reactions allow the differentiation of protonated *ortho*-, *meta*- and *para*-isomers of phenylene- and toluenediamines in FT-ICR.



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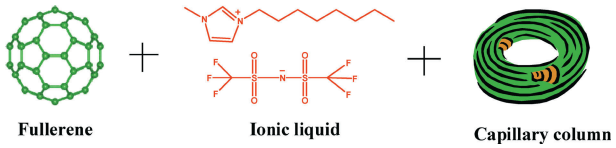
Fullerene-impregnated ionic liquid stationary phases for gas chromatography

Chieu D. Tran* and Santhosh Challa

Novel GC stationary phases based on amino and hydroxy derivatives of C_{60} were successfully prepared using ionic liquids, and were able to separate polar as well as non-polar analytes.



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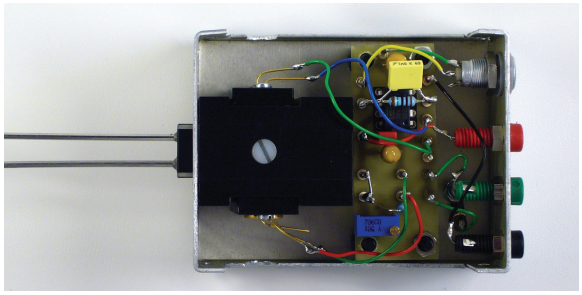
UV-absorbance detector for HPLC based on a light-emitting diode

Stefan Schmid, Mirek Macka and Peter C. Hauser*

Detection in the deep-UV at 255 nm has become possible using solid state optoelectronic components.



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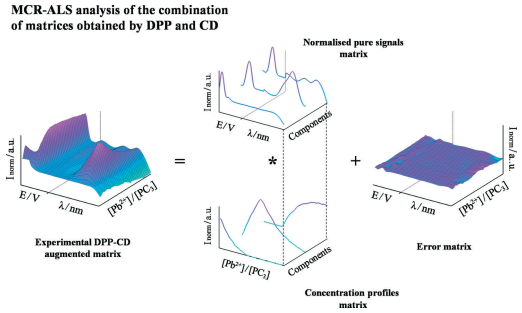
Combined use of the potential shift correction and the simultaneous treatment of spectroscopic and electrochemical data by multivariate curve resolution: analysis of a Pb(II)–phytochelatin system

Arístides Alberich, José Manuel Díaz-Cruz,* Cristina Ariño and Miquel Esteban

A combination of differential pulse polarographic and circular dichroism measurements with (potential shift corrected) multivariate curve resolution by alternating least squares for data treatment is applied to the study of the binding of Pb(II) by the phytochelatin $(\gamma\text{-Glu-Cys})_3\text{-Gly}$.



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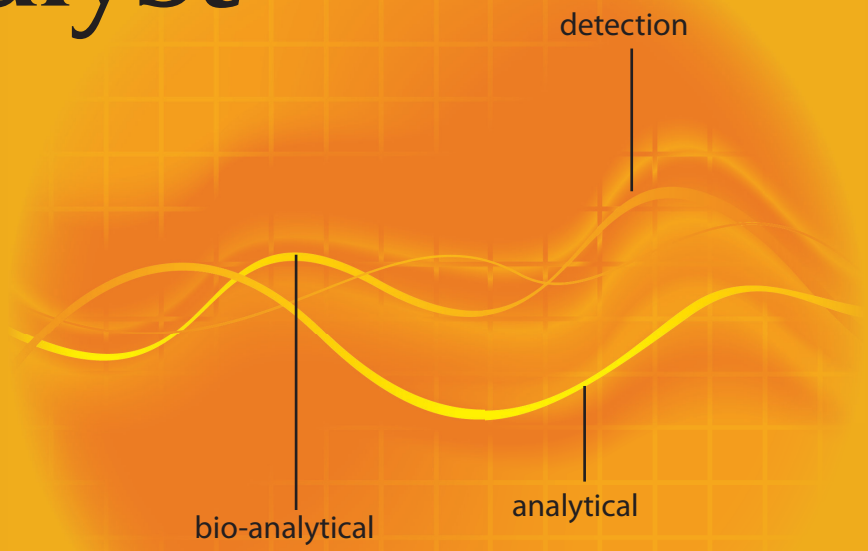

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Analyst, 2008, **133**, 17 - 24, **DOI**: 10.1039/b710236b

Photoelectrochemical ruler: measurement at the micron scale
Nicole Fietkau, Javier del Campo, Roser Mas, Francesc Xavier Muñoz and Richard G. Compton
Analyst, 2007, **132**, 983 - 985, **DOI**: 10.1039/b711828g

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Electrophoretic method for assessment of substrate promiscuity of a heterogeneous biocatalyst using an area imaging ultraviolet detector
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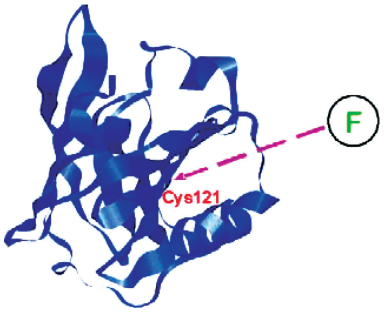

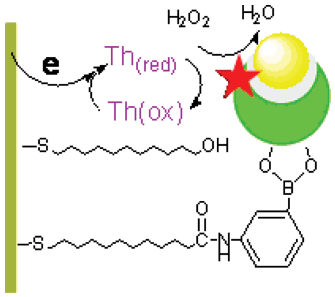

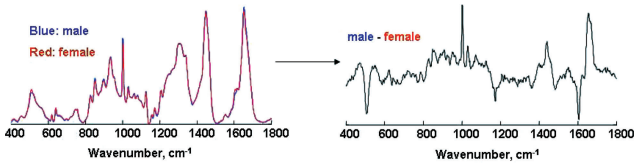

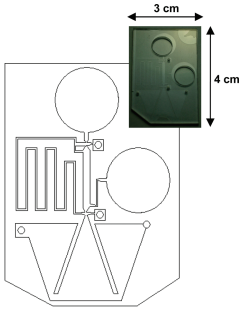

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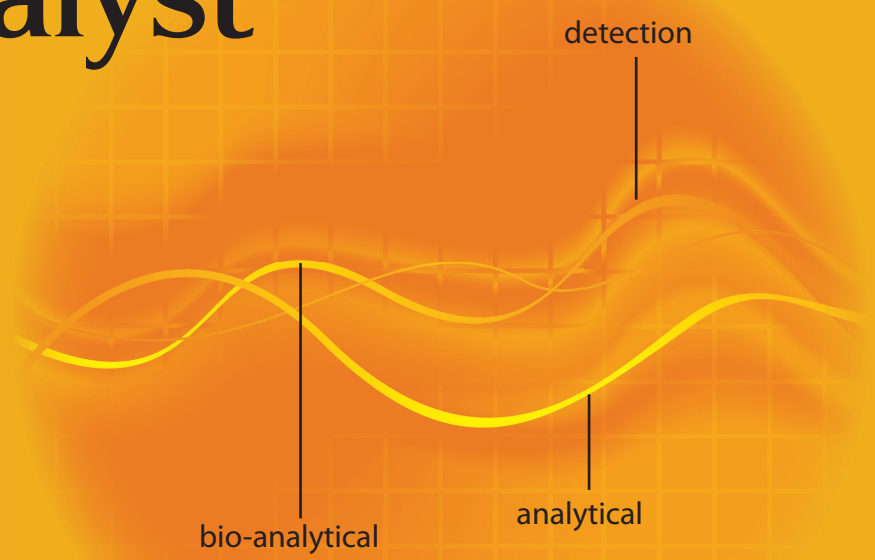

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Andrew Mills and David Hazafy
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Nanofluidics and the role of nanocapillary array membranes in mass-limited chemical analysis
Enid N. Gatimu, Jonathan V. Sweedler and Paul W. Bohn,
Analyst, 2006, **131**, 705 - 709, **DOI**: 10.1039/b600158k

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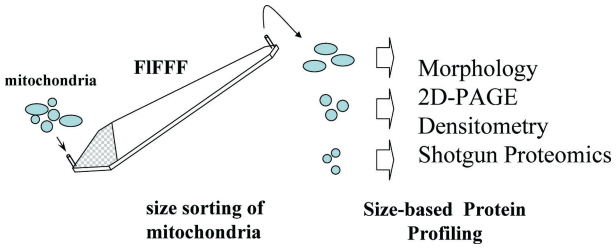

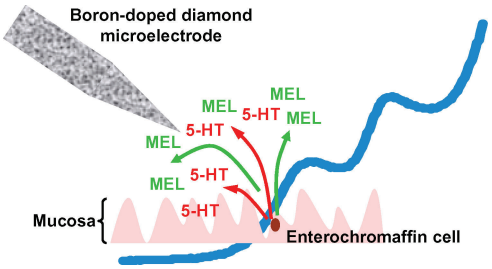

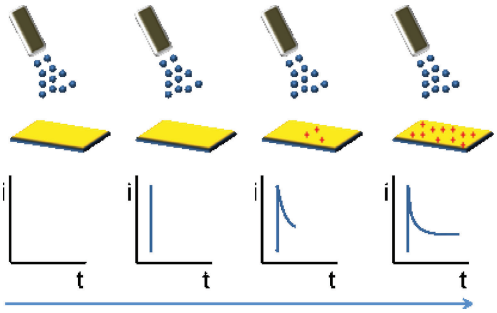

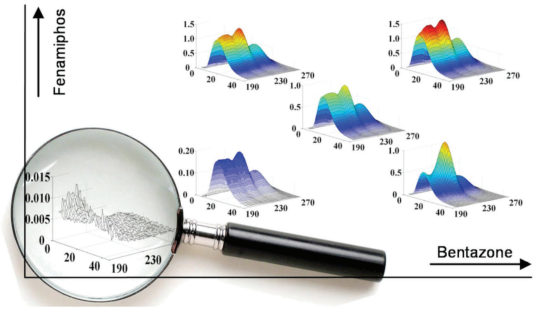

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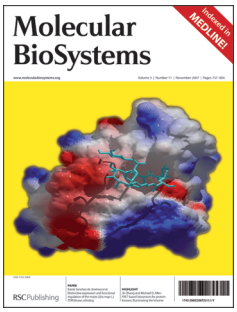

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Chemical Technology

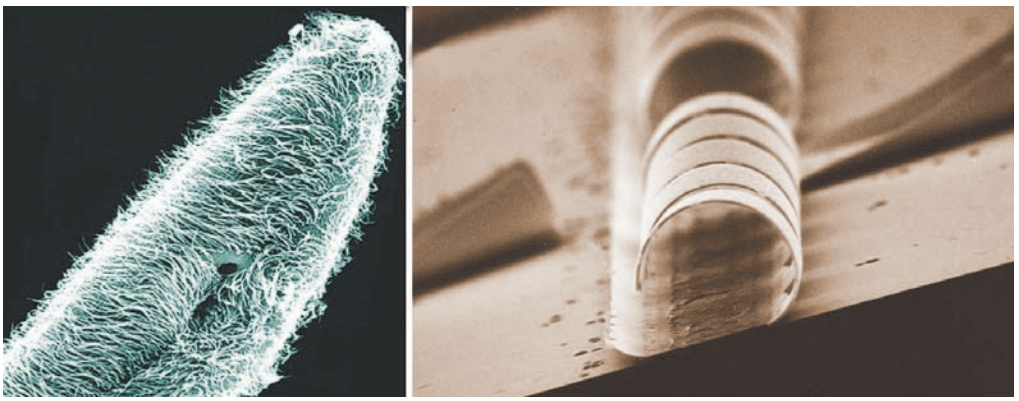
Layered polyimide and chromium curl and uncurl by applying a voltage

Miniature mixing inspired by nature

Dutch scientists have copied nature to develop a faster and more efficient method for mixing small volumes of liquid. They were inspired by micro-organisms that use tiny oscillating hairs (called cilia) to move through liquid, and made artificial cilia to use in microfluidics.

Microfluidics is the study of fluid flow in structures with dimensions smaller than a millimetre, with devices as small as a credit card. Key applications for these devices include biochips, especially the immediate point-of-care diagnosis of diseases. The problem with such a small device is that good mixing is almost impossible. Usually, mixing is done by etching grooves into the fluid channels or applying an electrical pulse or sound wave. However, there is no real control over the mixing in these cases, according to Jaap den Toonder of the Philips Research Laboratory in Eindhoven.

Toonder and colleagues have therefore made artificial cilia from a double layer of thin polymer, called polyimide, and a thin conductive layer of chromium. The artificial cilia looks a lot like a curled-up leaf.



Artificial cilia (right) mimic real cilia such as those on a Paramecium micro-organism (left)

These tiny mixers are activated by applying a voltage. When applied, the rolled-up cilia uncurl. When the voltage is turned off, the cilia roll back up by elastic recovery. In this way, the mixing can be turned on or off by demand and in any location in the device, an advantage over other mixing methods.

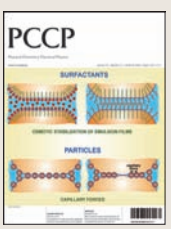
Andreas Manz, head of the Institute for Analytical Sciences in Dortmund, Germany, commented that the use of the artificial cilia for mixing was intriguing, as 'in biological systems cilia are used for transport'.

When questioned about the future, den Toonder said the next steps were to find alternative ways to activate the artificial cilia, such as magnetically-driven systems. The team's final goal is to develop artificial cilia 'into a versatile method for medical diagnostic lab-on-a-chip devices'.

Rebecca Brodie

Reference
J den Toonder *et al*, *Lab Chip*, 2008, DOI: 10.1039/b717681c

In this issue



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

Application highlights

Amides in minutes using continuous flow microreactor

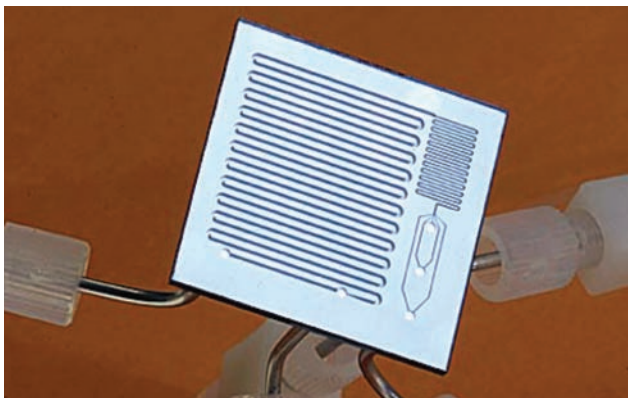
Big impact for small reactors

Microreactors can be a powerful tool for product synthesis in the pharmaceutical industry, say Swiss and Swedish scientists. Their application of microreactor technology to an amide bond formation reaction illustrates this.

Peter Seeberger from the Swiss Federal Institute of Technology, Zurich, and colleagues showed that their method can cope with a range of substrates, and applied it to the synthesis of two pharmaceutically active compounds. They claim that the microreactor-based technique is faster and safer than traditional methods, and that scale-up to bulk synthesis level is not a problem.

'One can think of a microreactor as a small pipe,' explained Seeberger. 'Chemicals are pumped through the pipe and additional inlets can be used to add reagents.' Microreactors allow fast mixing, quick and even heating, and the generation of high pressures. Together this makes for short reaction times; Seeberger's amide bond formation was completed within two minutes, compared to four to sixteen hours using conventional methods. In addition, microreactor systems are safer because the small reaction volumes mean that any hazardous intermediates are formed only in small amounts.

PETER SEEBERGER



Safe, efficient and versatile: microreactors could become the method of choice for industry

Reference
T Gustafsson *et al*, *Chem. Commun.*, 2008, 1100 (DOI: 10.1039/b719603b)

Steve Haswell, an expert in microreactor chemistry from the University of Hull, UK, said that microreactor technology has great potential for industry. Possible applications are reaction optimisation, library building, and safe and efficient product production, he said. The type of work performed by Seeberger 'should make medicinal chemists sit up and look seriously at the contribution microreactor technology has to offer,' said Haswell.

To illustrate the technique's versatility, Seeberger's group is working on applying microreactors for fluorinations and in radical and photochemistry. The group also has close collaborations with industry to look at real-life problems that microreactors could be used to solve. 'Industry is getting into this technology big-time,' said Seeberger.

Danièle Gibney

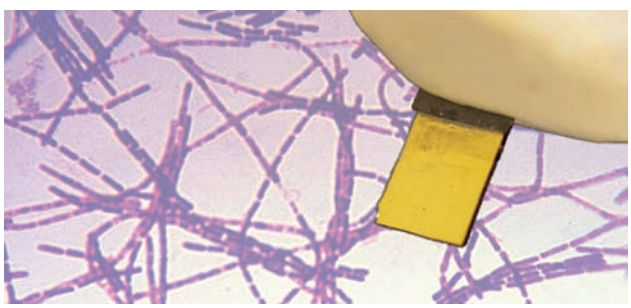
Piezoelectric cantilever picks out anthrax in real time

Detecting a microbe among millions

A sensor that can discriminate between closely related bacteria has been developed by materials scientists in the US.

Wan Shih and colleagues at Drexel University in Philadelphia immobilised a rabbit antibody to *Bacillus anthracis* (anthrax) on a piezoelectromicrocantilever sensor (PEMS). They then exposed the sensor to suspensions of anthrax spores and some of its close relatives. Because the chemical structures in the spore coats of these bacteria are similar, they all bind to the anthrax antibody to some extent. However, the binding interactions of the 'close relatives' are weaker than that of anthrax itself.

PEMS uses antibodies, which have high affinity and specificity for different microbes, attached to tiny cantilevers. 'These can be thought of as tiny diving boards made from piezoelectric material that can detect small particles, like deadly *Bacillus anthracis* spores, by vibrating,' explained Wei-Heng Shih, a member of the US team. The cantilevers' combination of specific antibody and sensitivity enables the sensor to accurately detect any micro-organism (depending on the antibody used) in real time, with no added chemicals, radioactive tags or external manipulation. 'The sensor can detect its preferred microbe in the presence of a million of its closely related cousins. It can detect a needle in a haystack!' said Wei-Heng Shih.



The PEMS cantilever vibrates when anthrax spores bind to an attached antibody

Reference
J-P McGovern *et al*, *Analyst*, 2008, DOI:10.1039/b715948j

To test if the PEMS could preferentially detect anthrax spores, the team flowed suspensions of the different bacteria across the sensor. The team observed that, at first, increasing the flow speed of the bacterial suspensions across the sensor led to increased binding of all four species because the sensor surface was exposed to more spores. But at higher flow rates, preferential anthrax–antibody binding was increased and binding to close relatives was suppressed.

'This result means that flow can be used to enhance the specificity of antibody-based detection,' said Wan Shih. 'With the right antibodies or receptors our sensor can detect air or food-borne pathogens and blood or urinary biomarkers for human diseases like cancer or AIDS...and it will make detection possible in minutes instead of days,' she added.

Janet Crombie

X-ray fluorescence reveals metals diffusing from paint into glass

Watching paint die

A new analytical technique from German scientists may help restore decaying glass paintings.

Birgit Kanngiesser led researchers from Berlin's Federal Institute for Materials Research and Testing, and the Technical University of Berlin, in studying reverse glass paintings – made by painting onto the back of a glass sheet. Unlike stained glass, these paintings are not fired and the contact between glass and paint is fragile.

The team used a technique called 3D micro x-ray fluorescence (3D micro XRF) to identify the elements in the corroded areas of the glass of a 19th century painting and compare them to healthy areas, without further damaging the artwork.

As with any XRF technique, micro XRF involves firing an x-ray beam at the sample to eject electrons. Higher-orbital electrons fall down to fill the gaps left by removed electrons, and in doing so emit photons of radiation characteristic to a particular element. In micro XRF, though, the beam can be focused onto a very small area. By using a powerful x-ray source and applying their recently-discovered calibration improvements to the technique, the researchers were able to study the composition of layers buried under the surface.

Reverse glass paintings: beautiful but fragile

Kangiesser's team identified the paints used and found that lead and mercury had diffused into the glass. The information should help conservators find the best way to protect and restore reverse glass artworks .

Kanngiesser hopes the non-destructive technique will extend to other fields, such as environmental science or archaeometry. 'Unlike other elemental imaging methods, there is no principal size restriction of the objects investigated,' she explains. 'And for life sciences the possibility of obtaining elemental distribution in virtual cross sections without having to cut the sample might open up new ways of in vivo measurements.' *Clare Boothby*

Reference
B Kanngiesser *et al, J. Anal. At. Spectrom.*, 2008, DOI: 10.1039/b717286a

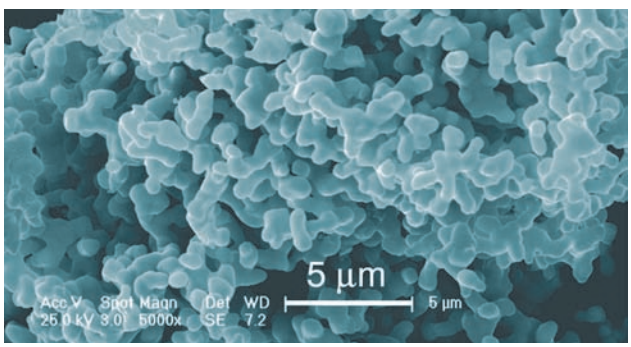
An environmentally friendly alternative to pyrometallurgical processes

Metals without the meltdown

Scientists have found a practical, cheap and environmentally friendly way of producing the industrially important metal niobium.

Niobium is used in a variety of applications, from superalloys to optics. Total world production is nearly 23 000 tonnes per year. But like many industrially important metals, it is extracted from metal ores and minerals using high temperatures – a relatively costly process. 'Alternative processes are essential to provide environmentally acceptable and energy efficient routes to metal extraction,' explains Frank Walsh, an expert on industrial electrochemistry from the University of Southampton, UK. 'In particular, minimising the number of steps in a production route and achieving direct transformation of minerals to metals is a critical subject area.'

George Zheng Chen from the University of Nottingham, UK, and colleagues at Wuhan University, China, have now cut the energy consumption of niobium extraction by a third, by improving on the already green FFC Cambridge process, named for its inventors Fray, Farthing and Chen, and originating in the late 1990s. In the FFC process, which is carbon-emission free, metals and alloys are extracted directly from their solid oxides using electrolysis in molten calcium chloride.



Niobium oxide pellets after electrolysis in calcium chloride

Chen and co-workers found that electrolysing niobium oxide in a three-electrode cell using a variable voltage uses significantly less energy than the FFC's constant voltage two-electrode system. However, the set-up is more complicated and so impractical in large scale operations. To get around this problem the scientists mimicked variable voltage with computer-aided control (CAC) of the two-electrode cell. Modifying the FFC process in this way results in a 'more than 37 per cent saving in energy consumption,' the team says. Chen says the energy-saving modification shouldn't require any changes to the FFC cells.

'CAC is demonstrated in principle for niobium extraction, but it is perfectly suitable for other reactive metals such as tantalum, titanium, zirconium and many rare earths,' says Chen. Also, adds Walsh, 'the system described provides an attractive and direct route to the formation of speciality alloys.'

The FFC Cambridge Process is being commercialised by a spin-out company – Metalysis, which has formed strategic partnerships with industry, including BHP Billiton and Rolls-Royce. *Freya Mearns*

Reference
T Wu *et al, Phys. Chem. Chem. Phys.*, 2008, **10**, 1809 (DOI: 10.1039/b719369f)

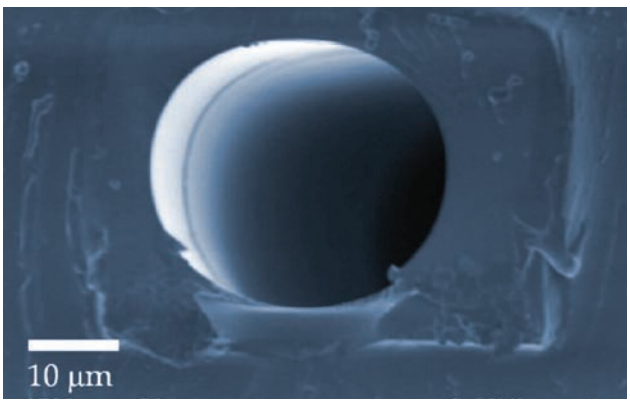
Coating improves chemical resistance of channels in microfluidic devices

Glass-coated microchannels

Scientists in the US have developed a simple method of coating the channels of microfluidic devices to make them more resistant to chemicals. David Weitz and colleagues from Harvard University, Cambridge, used a sol–gel method to create a glass coating on polydimethylsiloxane (PDMS) microchannels.

PDMS, a type of silicone rubber, is easy to make into microfluidic devices using soft lithography, where the material is 'stamped' with a channel pattern. This makes it ideal for large-scale use. However, PDMS is permeable to liquids and gases, which can affect reactions occurring in the channels. Additionally, organic solvents make the PDMS channels swell, degrading device performance. Glass is far more chemically robust but is much more difficult to make into microfluidic devices.

The glass coating developed by Weitz's group is easily deposited on PDMS channels and acts as a barrier, providing resistance to chemicals and solvents. Weitz said that this coating method would make device production easier as it 'combines the chemical robustness of glass with the ease of fabrication of PDMS.'



The best of both worlds: glass-coatings for PDMS channels mean robustness and easy device fabrication

To form the coating, Weitz's group used a sol–gel mixture that begins as a fluid and hardens into a glass. They filled the channels with the mixture, initiated a gelation reaction and then used air to flush out most of the material, leaving the glass coating.

The scientists discovered that the coated channels were resistant to the fluorescent chemical Rhodamine B. After an hour of exposure to the organic solvent toluene the channels changed very little. By contrast, uncoated channels swelled upon exposure to toluene.

Stephen Haswell, who develops microfluidic devices at the University of Hull, UK, said that although there would be issues with performing reactions at high temperatures, the work represented a step towards merging the advantages of PDMS and glass. 'Lack of chemical resistance is a big problem, and it will be something of a breakthrough to extend the fabrication benefits of PDMS to give more glass-type robustness,' he said.

Weitz's group is working on refining the technique so that the thickness of the coating can be more finely controlled.

Fay Riordan

Reference

A R Abate *et al*, *Lab Chip*, 2008, DOI: 10.1039/b800001h

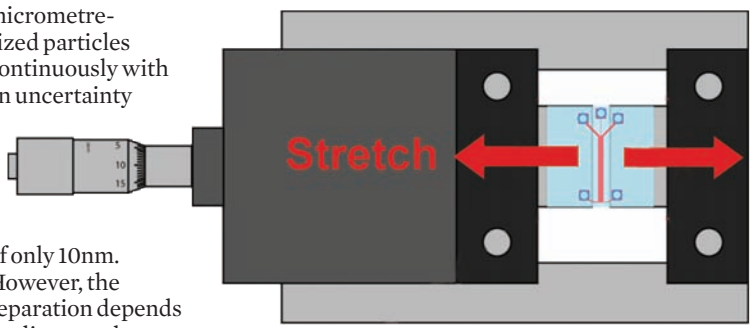
Tuneable system picks out particles of the required size

To sort, simply stretch

Researchers in Sweden have come up with a straightforward method for sorting particles in microfluidics – simply stretching the microchannel.

Sorting particles by size, anything from cells to plastic beads, is very useful in both analytical and preparative chemistry, but it is not always easy to achieve. In particular, a tuneable system, so that particles of different sizes can be sorted by the same device, is a major challenge. Jason Beech and Jonas Tegenfeldt of Lund University have struck upon a solution that makes use of the fact that the material used to make many microfluidic devices is very elastic.

Several methods have been proposed to sort particles in microfluidic devices, and one of the most promising is 'deterministic lateral displacement'. Originally reported by Huang and co-workers,[1] the technique is able to separate micrometre-sized particles continuously with an uncertainty of only 10nm. However, the separation depends on distances between obstacles in a narrow channel and so the channel has to be designed to separate only the particle of desired size. Beech and Tegenfeld realised that simply stretching the microchip changes the separation parameters. This gives a tuneable sorting device.



Stretching a microchip changes its separation parameters giving a tuneable sorting system

'The concept of macroscopic stretching to change the dimensions of fluidics devices on the nanometre to micrometre scale is something that has not been exploited fully in the microfluidics community and could, with a little imagination, lead to promising applications,' explained Beech 'Having had the idea that stretching the devices would tune them, we began to realise the wealth of interesting applications that this could lead to.'

Beech hopes the idea will spread. 'It will be interesting to see if others put the concept of stretching fluidics devices to use,' he said, 'but the challenges are those of engineering and materials science, namely making more stretchable, more robust and more homogenous devices.' *Edward Morgan*

References

J P Beech and J O Tegenfeldt, *Lab Chip*, 2008, DOI: 10.039/b719449h

1 L R Huang *et al*, *Science*, 2004, **304**, 987

Instant insight

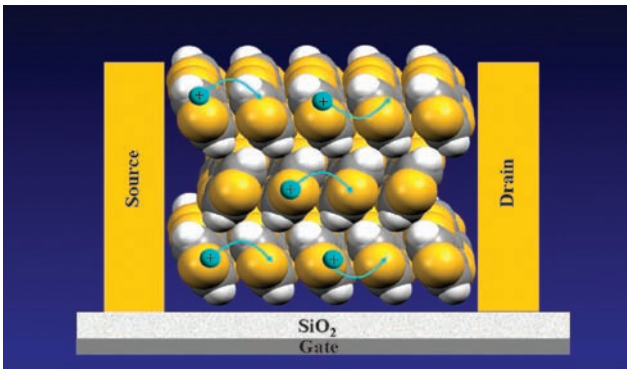
Organic field-effect transistors

Marta Mas-Torrent and Concepció Rovira of the Institute of Science of Materials (CSIC) in Barcelona, Spain, look at how small molecules can be used as processable semiconductors

Our daily life involves the continuous use of field-effect transistors: they are the main logic units, functioning as either switches or amplifiers, controlling current flow in electronic circuits. A field-effect transistor is a three-terminal device in which current flows through a semiconductor from the 'source' terminal to the 'drain'. This flow is controlled at the third 'gate' terminal by a voltage that creates an electric field through the insulator (dielectric) on which the semiconductor is deposited. Since the invention of the first transistor in 1947 by John Bardeen, William Shockley and Walter Brattain, the vast majority of electronic devices have been based on inorganic semiconductors and, in particular, on silicon.

Over the past few years, however, organic field-effect transistors (OFETs) have attracted a great deal of interest due to their unique processing characteristics. Organic materials offer the benefit that they can be printed over large areas on plastic, flexible substrates at low temperature by solution-based techniques, which would result in a dramatic reduction of manufacturing costs. Though the first OFETs did not transport charge as well as inorganic materials, the best ones nowadays are achieving charge carrier mobilities of the same order as amorphous silicon. Organic-based electronics will not replace high density and high speed silicon circuits, but might play an important role in applications such as identification tags, electronic bar codes or active matrix elements for displays.

OFETs have been mainly based on two types of semiconductors that feature π–π interactions: conjugated polymers and small conjugated molecules. Polymers are deposited from solution, allowing for low cost electronics. However, the higher molecular disorder in polymers limits their charge transport, typically resulting in lower mobilities compared to devices based on small molecules. On the other hand, devices prepared with small conjugated molecules have to be prepared more expensively by evaporating organic materials, due to their low solubility in common organic solvents. Therefore, to promote the development and use of organic semiconductors, there is a clear need to find materials that can be processed in solution and that simultaneously achieve a high OFET mobility.



Soluble organic molecules offer new perspectives as transistors in low-cost electronics

One way of imparting solubility to organic semiconductors is to prepare a precursor compound that can be converted into the parent semiconductor by heat or irradiation. An alternative strategy is to structurally modify organic semiconductors to impart solubility and, if possible, also achieve higher stability and increase π–π interactions. Semiconductors such as acenes and oligothiophenes (classed as p-type, since the charge carriers are mainly holes) have been processed by techniques such as spin coating and drop or zone casting, and very high performances have been achieved.

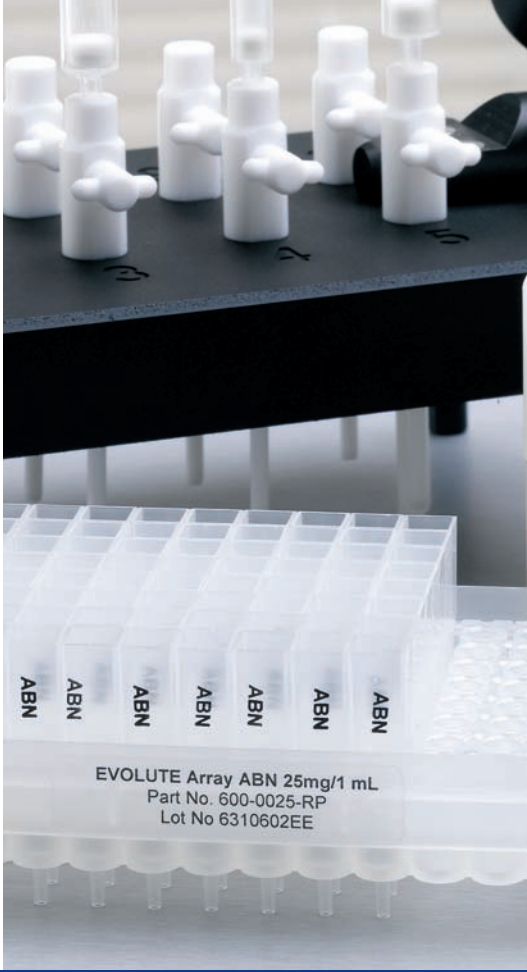
Currently, there is a growing interest in developing n-type semiconductors (where charge carriers are electrons) and ambipolar devices (which conduct both electrons and holes) in order to fabricate complementary circuits. The development in these devices is still far from the performance achieved with p-type materials, because transport in n-channel conductors is easily degraded by air and finding suitable metals for contacts is difficult.

Circuits based on organic transistors are being extensively investigated for a range of applications. One of the first emerging devices realised with OFETs is flexible electronic paper (see *Chemistry World*, April 2007, p15), where organic transistors switch pixels formed by charged pigments. OFETs also offer great promise for applications in chemical and biological sensing: organic semiconductors can interact with different analytes and it is possible to transduce the chemical information to electronic information, creating an 'electronic nose'. And the electrical switching of an OFET has been combined with an organic semiconductor's ability to generate light, to create organic light-emitting transistors (OLETs).

OFETs promise to be important in applications ranging from sophisticated medical diagnostics to 'smart' clothes that can display changing images. New markets will undoubtedly appear in areas where electronics meets with information technology, biomedicine or optics.

Read Concepció Rovira and Marta Mas-Torrent's critical review in issue 4, 2008, of Chemical Society Reviews.

Reference
M Mas-Torrent and C Rovira, *Chem. Soc. Rev.*, 2008, **37**, 827 (DOI: 10.1039/b614393h)



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Interview

Flying high with nanomedicine

Jinwoo Cheon tells Stephen Davey how nanoparticles can be used in medical diagnostics

Jinwoo Cheon

Jinwoo Cheon is professor of chemistry at Yonsei University, Korea, and head of the nanomaterials division of the Nano-Medical National Core Research Center of Korea. His research areas include the fabrication and shape control of inorganic nanocrystals, nanoscale biomagnetics, and the applications of nanocrystals for biomedical sciences. Jinwoo is on the editorial board of *Journal of Materials Chemistry*.

What inspired you to become a scientist?
I have always loved the arts, and art is what I saw in my first chemistry classes. The representation of molecules and the systematic formation or cleavage of bonds to give different molecules with different properties fascinated me. I found it easy to comprehend what was happening in this molecular world and I viewed it as if playing a game of Lego.

Can you tell me a little about your background?
My training is in the area of synthetic inorganic chemistry. I studied organometallic and materials chemistry under the guidance of some wonderful mentors including Gregory Girolami at the University of Illinois, John Arnold at Berkeley, and Jeffrey Zink at UCLA. The areas of molecular precursor chemistry for materials synthesis and their transformations from a mechanistic point of view are of particular interest. I have extended these concepts to nanoparticle synthesis in which the size, shape, and composition are of significant importance to their material properties. I hope to make major contributions towards solving today's health issues, such as cancer, and energy storage concerns by using nanoscale materials.

One of the applications of your research into magnetic nanomaterials is in medical diagnostics. Can you explain how this works?
Nanoparticles are important in biomedical applications due to their small size and wonderful properties. Magnetic nanoparticles are especially useful for biological separation, diagnostics and treatment of diseases. By tuning their magnetism by varying their size or composition, we have been able to fabricate extremely sensitive magnetic resonance imaging (MRI) probes. They selectively target biological species with strong enhancement of detection sensitivity for the diagnosis of smaller cancers. We are also developing biocompatible nanoparticles which are free of potential toxicity without losing their materials properties.

What's the trickiest problem that you've faced, and how did you solve it?
Interdisciplinary research topics such as nanomedicine require collaboration between different research fields. This is not always easy as it is difficult to get people to buy into concepts that they are not familiar with.

I often have to be persistent and an advocate for our nanomaterials. However, synergistic results between research groups are the most important outcome. As scientists, researchers and educators, we should try to understand each other's language and to overcome the boundaries between different disciplines.

As chemists, we are in a good position to be translators of these different languages since chemistry plays a pivotal role in interfacial problem solving and bridges interdisciplinary subjects.

What is the secret to running a successful research group?
Highly motivated and enthusiastic students are the most important assets. A research group's goals cannot be realised without open communication and ownership. Members must share the group's vision but also be willing to take a chance with new ideas and bring their own insight to the table. There has to be an atmosphere of trust and mutual respect in order to nurture the growth of all involved.

What is the most rewarding aspect of your work?
Working with next generation chemists who will be the leaders in our global community through our research efforts is the most rewarding part. Hopefully, in the future, new nanomaterials that are developed as a result of our work can contribute towards enhancing the quality of human life in terms of medical sciences or energy-related technologies.

Do you have a message for young scientists?
Try to develop your own interpretation of science. In your pursuit of becoming a scientist, be persistent and enjoy the ride and you will be rewarded in the future, regardless of where the road takes you.

Which scientist, current or historic, do you most admire and why?
Marie Curie. She was not only a great chemist but she was successful despite being in adverse surroundings, which included gender and racial discrimination and extremely poor research environments.

If you weren't a scientist, what would you be?
A pilot, if wearing glasses did not matter!

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Hot off the press

The μTAS 2008 conference in San Diego, US, is featuring a new award sponsored by *Lab on a Chip* titled 'Under the Looking Glass: Art from the World of Small Science.' Applications are encouraged from any person attending the conference and the winner will be selected by a panel of senior scientists in the field of μTAS.

Applications must show a photograph, micrograph or other accurate representation of a system that would be of interest to the μTAS community. They must also contain a brief caption that describes the illustration's content and its scientific merit. The winner will be selected on the basis of aesthetic appeal, artistic allure and scientific merit. In addition to having the image featured on the cover of *Lab on a Chip*, the winner will also receive a financial award at the conference.

For more information email loc@rsc.org

Chemical Technology (ISSN: 1744-1560) is published monthly by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge UK CB4 0WF. It is distributed free with *Chemical Communications*, *Journal of Materials Chemistry*, *The Analyst*, *Lab on a Chip*, *Journal of Atomic Absorption Spectrometry*, *Green Chemistry*, *CrystEngComm*, *Physical Chemistry Chemical Physics* and *Analytical Abstracts*.
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