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IN THIS ISSUE

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Cover

See Martin Schröder *et al.*, pp. 6108–6110.
Hydrogen adsorption is enhanced within a porous anionic framework *via* cation exchange.
Image reproduced by permission of Sihai Yang, Xiang Lin, Alexander J. Blake, K. Mark Thomas, Peter Hubberstey, Neil R. Champness and Martin Schröder from *Chem. Commun.*, 2008, 6108.



Inside cover

See Paul F. Kelly *et al.*, pp. 6111–6113.
Imaging of inkjet traces or fingerprints (on a range of media, such as the glass vial shown) can be brought about by polymerisation of disulfur dinitride.
Image reproduced by permission of Paul F. Kelly, Roberto S. P. King and Roger J. Mortimer from *Chem. Commun.*, 2008, 6111.

CHEMICAL TECHNOLOGY

T89

Drawing together research highlights and news from all RSC publications, *Chemical Technology* provides a 'snapshot' of the latest applications and technological aspects of research across the chemical sciences, showcasing newsworthy articles and significant scientific advances.

Chemical Technology

December 2008/Volume 5/Issue 12

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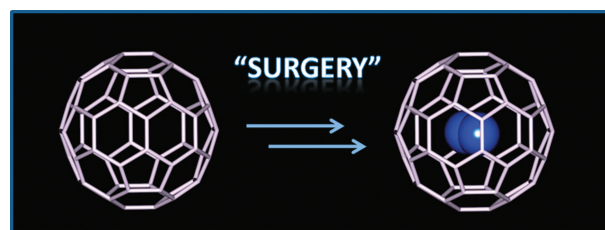
FEATURE ARTICLES

6083

Surgery of fullerenes

Michihisa Murata, Yasujiro Murata and Koichi Komatsu*

Recent attempts at the synthesis of endohedral fullerenes by organic reactions, so-called "molecular surgery" methods, are surveyed. An effective route to "suture" an opening was established to realize a new endohedral fullerene, $H_2@C_{60}$. Further development of this operation as well as the properties and reactions of $H_2@C_{60}$ are summarized. Also the application of the encapsulated H_2 molecule as an NMR probe for the study of aromaticity of ionic fullerenes is described.



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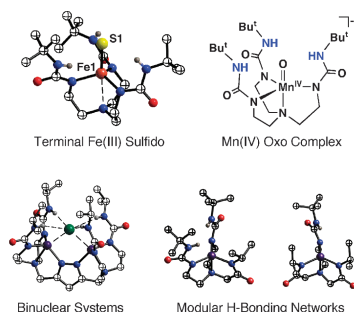
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6095

The effects of hydrogen bonds on metal-mediated O₂ activation and related processes

Ryan L. Shook and A. S. Borovik*

Using lessons obtained from the active sites of metalloproteins, the chemical and physical properties for a series of metal complexes with intramolecular hydrogen bonding networks are described.



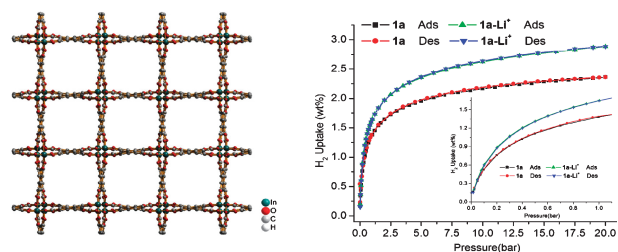
COMMUNICATIONS

6108

Enhancement of H₂ adsorption in Li⁺-exchanged co-ordination framework materials

Sihai Yang, Xiang Lin, Alexander J. Blake, K. Mark Thomas, Peter Hubberstey, Neil R. Champness* and Martin Schröder*

H₂ adsorption in an anionic co-ordination framework (Me₂NH₂)[In(L)] is enhanced by exchange of Me₂NH₂⁺ cations with Li⁺; the increase in H₂ capacity is related to an increase in the accessible pore volume on cation exchange.

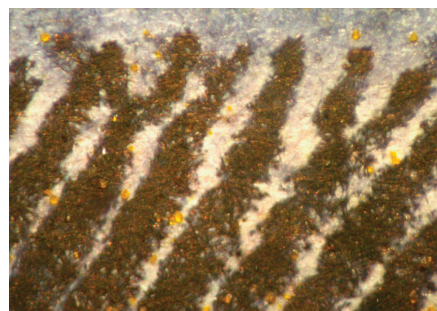


6111

Fingerprint and inkjet-trace imaging using disulfur dinitride

Paul F. Kelly,* Roberto S. P. King and Roger J. Mortimer

Exposure of fingerprints to S₂N₂ vapour results in the formation of (SN)_x on the prints, thereby imaging the latter. This can be achieved with prints on an unprecedented range of media; in addition, the same technique also enhances and exposes minute traces of inkjet ink.

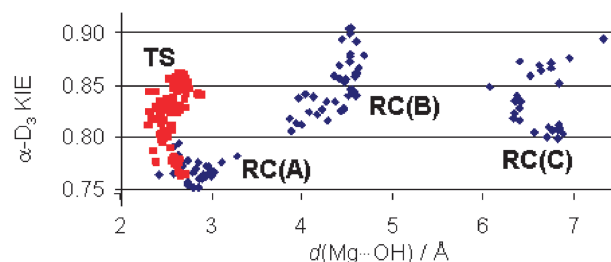


6114

QM/MM simulations for methyl transfer in solution and catalysed by COMT: ensemble-averaging of kinetic isotope effects

Natalia Kanaan, J. Javier Ruiz Pernía and Ian H. Williams*

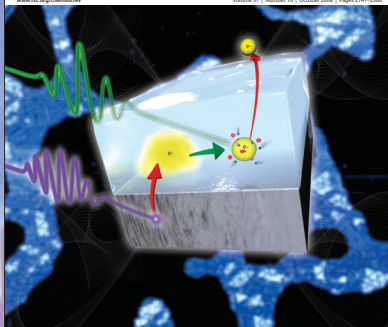
Sampling of structures from molecular dynamics reveals families of reactant-state conformers and yields KIEs for reactions in enzyme active sites and in solution, averaged over thermal fluctuations of the environment, that allows comparison of computed with experimental values.



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THEMATIC ISSUE: CHEMISTRY AT SURFACES
Guest editors: Hans-Peter Steinrück, Jörg Libuda and Sir David A. King



Dedicated to Professor Gerhard Ertl,
recipient of the 2007 Nobel Prize in Chemistry

Chemistry at Surfaces themed issue

The issue highlights state-of-the-art research from the scientific field pioneered by Professor Gerhard Ertl. Selected and representative examples of modern topics are presented by a number of colleagues who contributed to the emergence of surface science together with Professor Ertl, as well as by younger colleagues who significantly contributed to the present reputation of the field.

Driven by rapid progress in experimental and theoretical methodology, surface science turned into a well-established scientific area, nowadays laying the foundations for a molecular-level understanding of the chemistry behind 21st century key technologies in fields such as heterogeneous catalysis, corrosion, fuel cell research, and the semiconductor industry.

Reviews include:

Molecular surface chemistry by metal single crystals and nanoparticles from vacuum to high pressure

Gabor A. Somorjai and Jeong Y. Park

The nature of the active site in heterogeneous metal catalysis

Jens K. Nørskov, Thomas Bligaard, Britt Hvolbæk, Frank Abild-Pedersen, Ib Chorkendorff and Claus H. Christensen

A surface science approach to ultrafast electron transfer and solvation dynamics at interfaces

Julia Stähler, Uwe Bovensiepen, Michael Meyer and Martin Wolf

Oxide ultra-thin films on metals: new materials for the design of supported metal catalysts

Hans-Joachim Freund and Gianfranco Pacchioni

Nanoscale surface chemistry over faceted substrates: structure, reactivity and nanotemplates

Theodore E. Madey, Wenhua Chen, Hao Wang, Payam Kaghazchi and Timo Jacob

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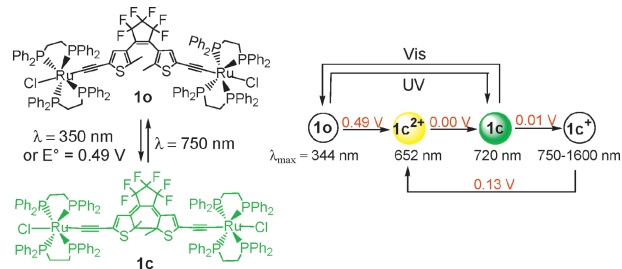
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6117

A multifunctional organometallic switch with carbon-rich ruthenium and diarylethene units

Yifei Liu, Corinne Lagrost,* Karine Costuas,*
Nouredine Tchour, Hubert Le Bozec and
Stéphane Rigaut*

The perturbation of a dithienylethene system by a ruthenium carbon-rich system, and *vice versa*, was used to reach a unique and sophisticated light- and electro-triggered multifunctional switch featuring multicolor electrochromism, electrocyclization at remarkably low voltage, and photo/electro tuning of electronic communication.

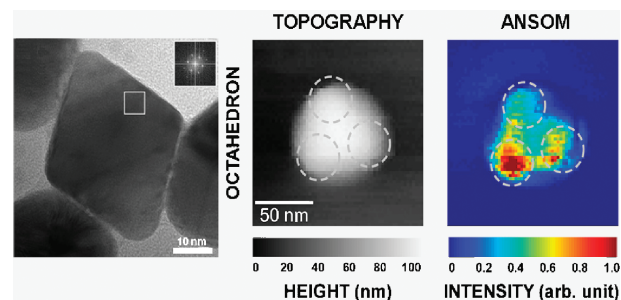


6120

Controlled synthesis and characterization of the enhanced local field of octahedral Au nanocrystals

Jinhwa Heo, Deok-Soo Kim, Zee Hwan Kim,*
Young Wook Lee, Dongheun Kim, Minjung Kim,
Kihyun Kwon, Hyung Ju Park, Wan Soo Yun* and
Sang Woo Han*

Octahedral Au nanocrystals with localized surface plasmon-assisted enhancing optical properties can be prepared in aqueous solution *via* the forced reduction of Au ions by ascorbic acid through the addition of NaOH.

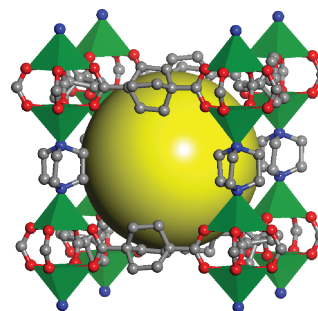


6123

Unique gas and hydrocarbon adsorption in a highly porous metal-organic framework made of extended aliphatic ligands

Kunhao Li, JeongYong Lee, David H. Olson,
Thomas J. Emge, Wenhua Bi, Matthew J. Eibling and
Jing Li*

High and unique gas and hydrocarbon adsorption in a highly stable guest-free microporous metal-organic framework constructed on rigid extended aliphatic ligands, H₂bodc and ted, is reported in this work.

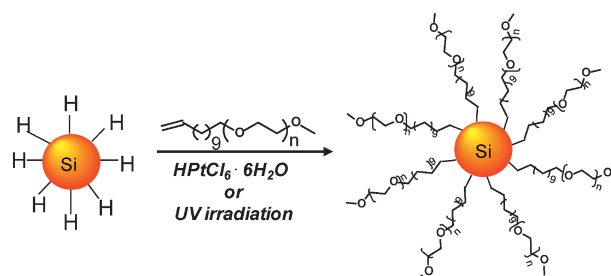


6126

PEGylated silicon nanoparticles: synthesis and characterization

P. K. Sudeep, Zachariah Page and Todd Emrick*

Photostable, amphiphilic PEGylated silicon nanoparticles prepared by hydrosilylation reactions with alkene-terminated PEG show solution fluorescence in water and organic solvents.



New journals from RSC Publishing in 2009!



Metallomics Integrated biometal science

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A unique, highly interdisciplinary journal focused on quantitative multi-scale biology using enabling technologies and tools to exploit the convergence of biology with physics, chemistry, engineering, imaging and informatics. Monthly from 2009.

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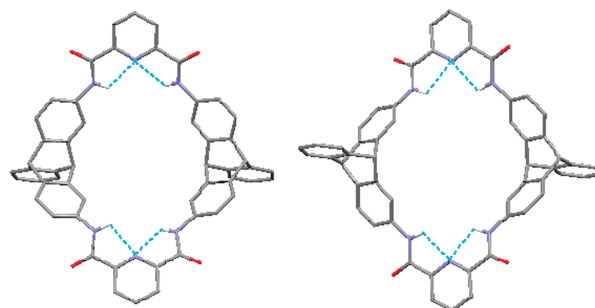
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6128

Triptycene-based tetralactam macrocycles: synthesis, structure and complexation with squaraine

Min Xue and Chuan-Feng Chen*

Two novel triptycene-based tetralactam macrocycles as a pair of diastereomers were synthesized, and showed highly efficient complexation with squaraine, which could subsequently protect the squaraine dye from polar solvents.

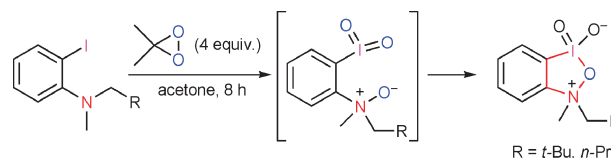


6131

Preparation and X-ray crystal structure of 2-iodyl-*N,N*-dialkylaniline oxides: first entry into the heterocyclic system of benziiodoxazole

Viktor V. Zhdankin,* Victor N. Nemykin,* Rashad R. Karimov and Zeinul-Gabiden Kazhkenov

Oxidation of 2-iodo-*N,N*-dialkylanilines with dimethyldioxirane affords 2-iodyl-*N,N*-dialkylaniline oxides, structural features of which are in agreement with the new heterocyclic system of benziiodoxazole.

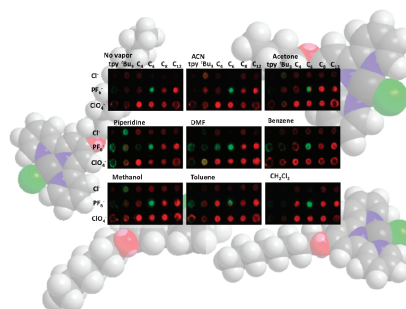


6134

Microarray pattern recognition based on Pt^{II} terpyridyl chloride complexes: vapochromic and vapoluminescent response

Maria L. Muro, Charles A. Daws and Felix N. Castellano*

Cross-reactive solid-state Pt^{II} terpyridyl chloride vapochromic sensor elements incorporated into microarrays generate distinctive colorimetric and luminometric responses upon exposure to a variety of volatile organic compounds.

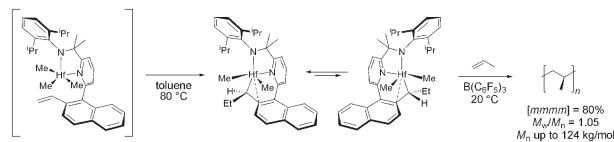


6137

Synthesis of a new olefin polymerization catalyst supported by an sp³-C donor *via* insertion of a ligand-appended alkene into the Hf–C bond of a neutral pyridylamidohafnium trimethyl complex

Gregory J. Domski, Joseph B. Edson, Ivan Keresztes, Emil B. Lobkovsky and Geoffrey W. Coates*

Unexpected reactivity of a ligand-appended alkene led to the discovery of a new living and isoselective olefin polymerization catalyst.



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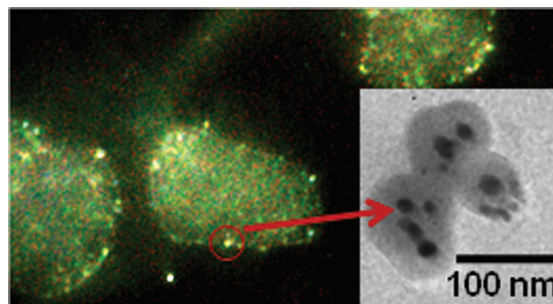
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6140

One-pot synthesis of silica-coated magnetic plasmonic tracer nanoparticles

Anand Gole,* Nalini Agarwal, Pratik Nagaria, Michael D. Wyatt and Catherine J. Murphy*

A synthetic route for multi-functional tracer nanoparticles with magnetic, elastic light scattering, Raman scattering and fluorescence properties for multimodal environmental and bio-tracking applications has been developed.

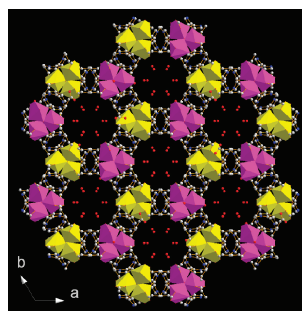


6143

Unprecedented helix-based microporous metal–organic frameworks constructed from a single ligand

Rui-Li Sang and Li Xu*

Unprecedented helix-based MMOFs with a cylindrical channel constructed from a single linker are built from alternately arranged left- and right-handed helical M-bim tubes interconnected by the carboxyl arms of the ligand.

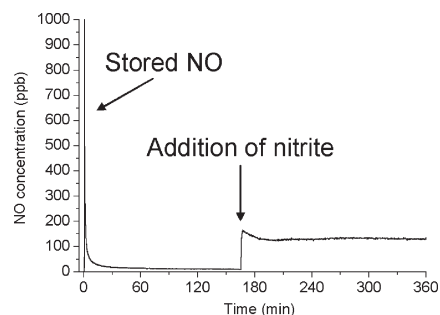


6146

Simultaneous and cooperative gas storage and gas production using bifunctional zeolites

Astrid-Karla Boes, Paul S. Wheatley, Bo Xiao, Ian L. Megson and Russell E. Morris

Zeolites can release stored nitric oxide on contact with water. On addition of nitrite ion solution they also chemically produce the gas, the two mechanisms occurring simultaneously and cooperatively.

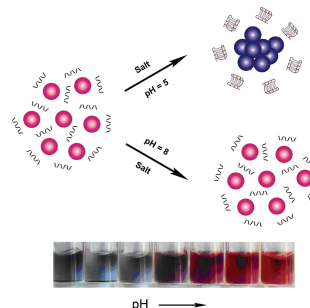


6149

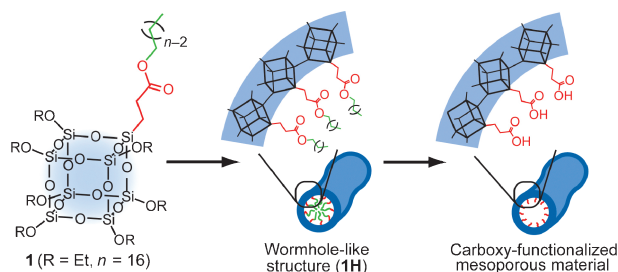
A simple and sensitive colorimetric pH meter based on DNA conformational switch and gold nanoparticle aggregation

Cuie Chen, Guangtao Song, Jinsong Ren* and Xiaogang Qu

A simple, rapid and sensitive pH meter based on the conformational switch of i-motif DNA and non-crosslinking AuNP aggregation has been developed with an average accuracy of ± 0.04 pH unit.



6152

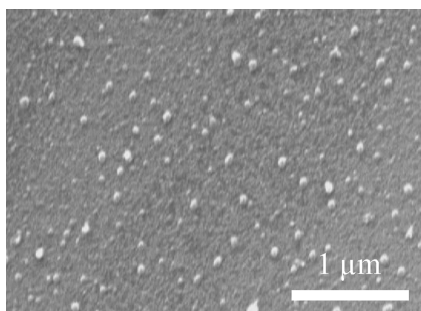


A hybrid mesoporous material with uniform distribution of carboxy groups assembled from a cubic siloxane-based precursor

Ryota Goto, Atsushi Shimojima,* Hideki Kuge and Kazuyuki Kuroda*

A novel hybrid mesoporous material functionalized with carboxy groups was synthesized by a technique that represents a promising approach to designing hybrid structures at both molecular and mesoscopic scales.

6155

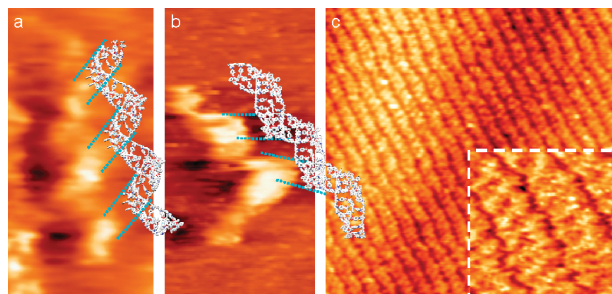


Highly efficient and stable palladium nanocatalysts supported on an ionic liquid-modified xerogel

Afsaneh Safavi,* Norouz Maleki, Nasser Iranpoor, Habib Firouzabadi, Ali Reza Banazadeh, Roya Azadi and Fatemeh Sedaghati

Incorporation of a phosphorylated ionic liquid as both a complexing and reducing agent for Pd(II) in a xerogel results in the formation of highly dispersed, uniformly sized Pd nanocatalysts.

6158

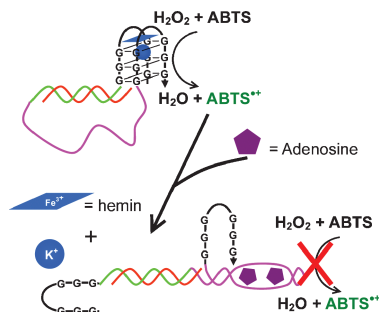


One-handed helical double stranded polybisisnorbornenes

Hui-Chun Yang, Sheng-Long Lee, Chun-hsien Chen,* Nai-Ti Lin, Hsiao-Ching Yang, Bih-Yaw Jin and Tien-Yau Luh*

Helical double stranded polymers incorporated with a covalently bound chiral ferrocene linker are synthesised and characterized by CD spectra and STM images and molecular dynamics simulations.

6161



Rational design of an optical adenosine sensor by conjugating a DNA aptamer with split DNAzyme halves

Na Lu, Congying Shao and Zhaoxiang Deng*

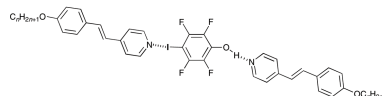
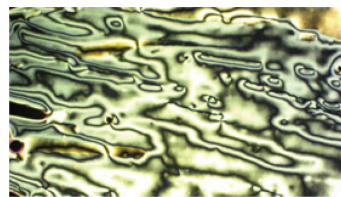
An anti-adenosine aptamer has been equipped with two split halves of a hemin-binding DNAzyme to achieve a highly selective and sensitive allosteric sensor for colorimetric adenosine detection.

6164

Trimeric liquid crystals assembled using both hydrogen and halogen bonding

Carsten Präsang, H. Loc Nguyen, Peter N. Horton, Adrian C. Whitwood and Duncan W. Bruce*

New liquid crystals are reported that are formed from non-mesomorphic components assembled using both halogen and hydrogen bonding.

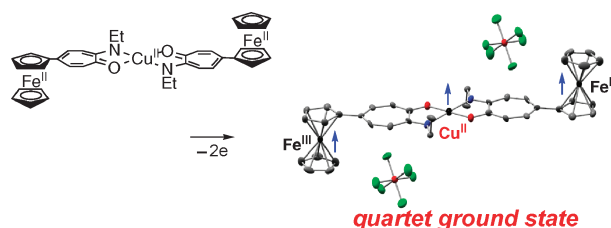


6167

Long-distance ferromagnetic coupling through spin polarization in a linear heterotrimeric iron(III)–copper(II)–iron(III) complex derived from 5-ferrocenyl-2-aminotropone

Yoshihiro Miyake, Sayaka Watanabe, Satoshi Aono, Tohru Nishinaga,* Akira Miyazaki, Toshiaki Enoki, Hitoshi Miyasaka, Hiroyuki Otani and Masahiko Iyoda*

A complex composed of two ferrocenium-ion moieties and copper ligated by 2-aminotropones showed strong intramolecular ferromagnetic coupling in the solid state owing to spin polarization.

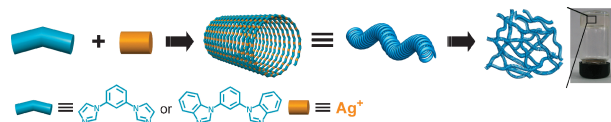


6170

Helical nonracemic tubular coordination polymer gelators from simple achiral molecules

Shiyong Zhang, Shengyong Yang, Jingbo Lan, Shuaijun Yang and Jingsong You*

A novel class of helical nonracemic tubular coordination polymers, which can immobilize a wide range of solvents at very low concentrations, have been synthesized for the first time in the absence of chiral influences and appended groups.

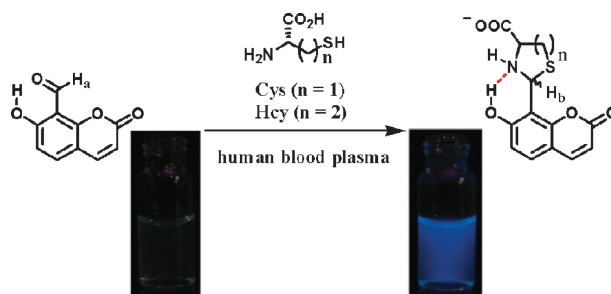


6173

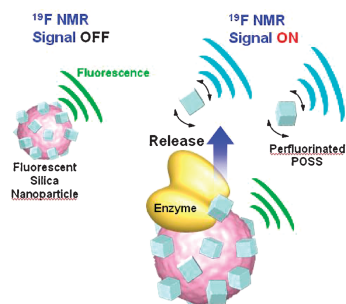
Fluorescence turn-on probe for homocysteine and cysteine in water

Kyung-Sik Lee, Tae-Ki Kim, Jong Ho Lee, Hae-Jo Kim* and Jong-In Hong*

A simple fluorescent probe based on an aldehyde-functionalized coumarin has been developed for selective detection of an aminothiols compound. The probe exhibits highly selective fluorescence turn-on signals for homocysteine and cysteine over other amino acids in water at biological pH.



6176

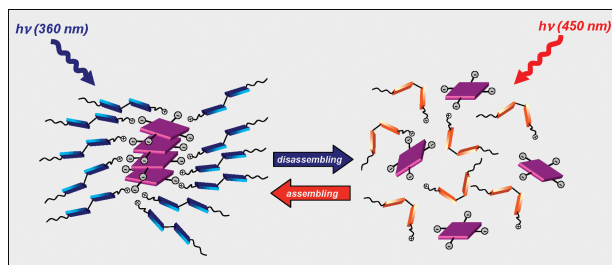


Multi-modal ^{19}F NMR probe using perfluorinated cubic silsesquioxane-coated silica nanoparticles for monitoring enzymatic activity

Kazuo Tanaka, Narufumi Kitamura, Kensuke Naka and Yoshiki Chujo*

Water-soluble perfluorinated cubic silsesquioxane was synthesised and immobilised onto the silica nanoparticles for suppressing the signals. ^{19}F NMR signals of the probes were recovered by releasing from nanoparticles.

6179

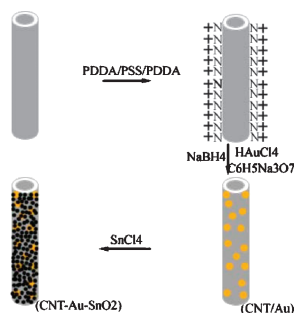


Controlling molecular assembling by photons: reversible light-powered monomer-aggregate interconversion of porphyrins

Fiorella L. Callari and Salvatore Sortino*

We report the first example of reversible monomer-aggregate interconversion of porphyrins in water exclusively controlled by light inputs of different energy absorbed by a photoswitching unit non-covalently linked to the porphyrin center.

6182

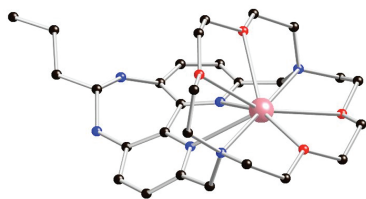


Homogeneous coating of Au and SnO_2 nanocrystals on carbon nanotubes *via* layer-by-layer assembly: a new ternary hybrid for a room-temperature CO gas sensor

Ning Du, Hui Zhang, Xiangyang Ma and Deren Yang*

CNT/Au/ SnO_2 nanotubes have been synthesized through homogeneous coating of Au and SnO_2 nanocrystals on carbon nanotubes (CNTs) *via* layer-by-layer assembly.

6185



A wavelength and lifetime responsive cryptate-containing fluorescent probe for zinc ions in water

Cara E. Felton, Lindsay P. Harding, Jennifer E. Jones, Benson M. Kariuki, Simon J. A. Pope* and Craig R. Rice*

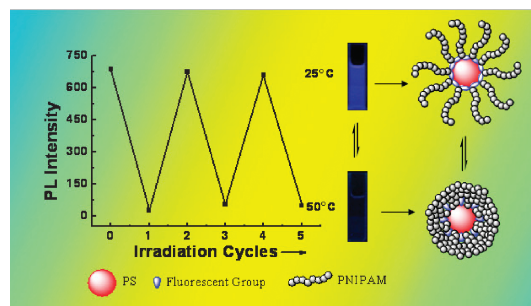
A functionalized bipyridine-based cryptate binds metallic cations in water. Visible light excitation results in metal-dependent luminescence properties allowing detection of zinc in ionic mixtures.

6188

Copolymer logical switches adjusted through core-shell micelles: from temperature response to fluorescence response

Qiang Yan, Jinying Yuan,* Weizhong Yuan, Mi Zhou, Yingwu Yin and Caiyuan Pan*

A type of copolymer logical switch can carry out temperature- and temperature-driven fluorescent transition adjusted through the architectural change of self-assembled micelles.

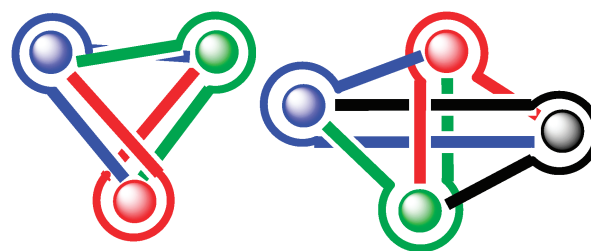


6191

Direct synthesis and structural characterisation of tri- and tetra-nuclear silver metallaknotanes by self-assembly approach

Julien Bourlier, Abdelaziz Jouaiti, Nathalie Kyritsakas-Gruber, Lionel Allouche, Jean-Marc Planeix* and Mir Wais Hosseini*

The combination of ligands bearing quinolinyl and oligoethyleneoxy units acting as primary and secondary sites, respectively, leads spontaneously to the formation of tri- and tetra-nuclear silver knots.

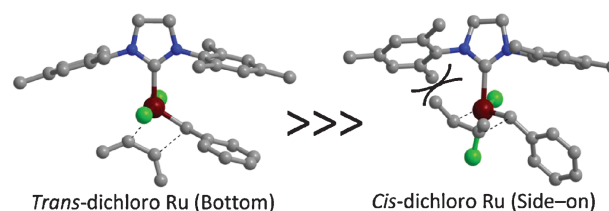


6194

Relevance of *cis*- and *trans*-dichloride Ru intermediates in Grubbs-II olefin metathesis catalysis ($H_2IMesCl_2Ru=CHR$)

Diego Benitez, Ekaterina Tkatchouk and William A. Goddard III*

Using density functional theory, we show that the $(H_2IMes)(Cl)_2Ru$ olefin metathesis mechanism is bottom-bound, with the chlorides remaining *trans* throughout the reaction.

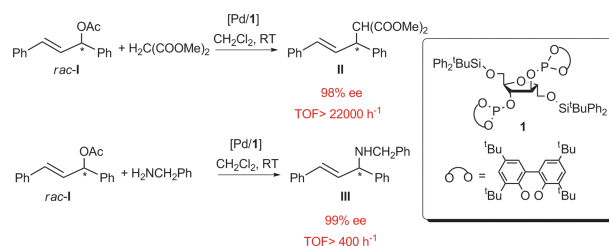


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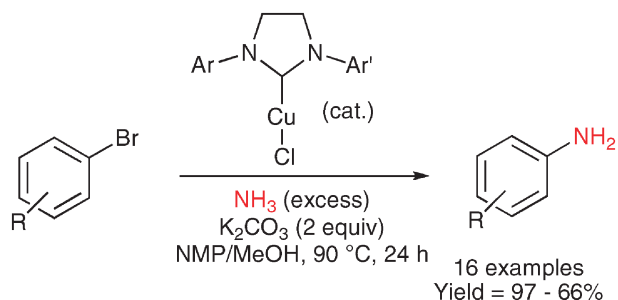
An outstanding palladium system containing a C_2 -symmetrical phosphite ligand for enantioselective allylic substitution processes

Angelica Balanta Castillo, Isabelle Favier, Emmanuelle Teuma, Sergio Castellón, Cyril Godard,* Ali Aghmiz, Carmen Claver* and Montserrat Gómez*

Pd systems bearing C_2 -symmetric chiral diphosphite ligands derived from carbohydrates are very active catalysts in asymmetric allylic substitution reactions, reaching TOFs $> 22\,000\ h^{-1}$ in allylic alkylation and $400\ h^{-1}$ in allylic amination.



6200

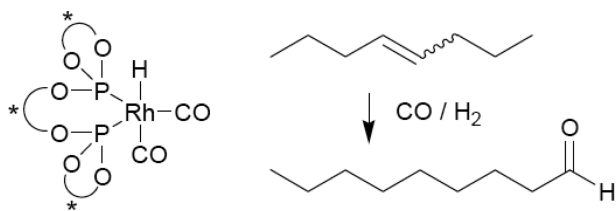


Cross-coupling of aryl/heteroaryl bromides with ammonia using a copper-carbene catalyst

Rukundo Ntaganda, Bhartesh Dhudshia, Charles L. B. Macdonald and Avinash N. Thadani*

A variety of aryl and heteroaryl bromides were cross-coupled with ammonia in good to high yields in the presence of a copper-NHC catalyst.

6203

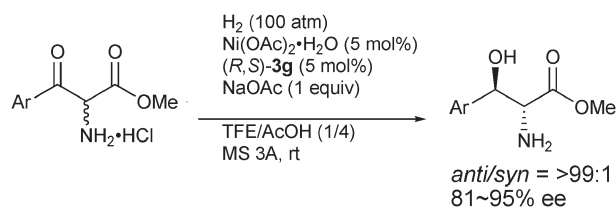


Diastereoisomeric bisphosphite ligands in the hydroformylation of octenes: rhodium catalysis and HP-NMR investigations

Detlef Selent,* Wolfgang Baumann, Klaus-Diether Wiese and Armin Börner*

Although a comparison of diastereoisomeric rhodium catalysts has revealed similar regioselectivities for 1-octene hydroformylation and a preferred bisequatorial coordination of the bisphosphite ligand, differences in the hydride complex formation pathway and individual performances with internal olefins used as substrates were observed.

6206

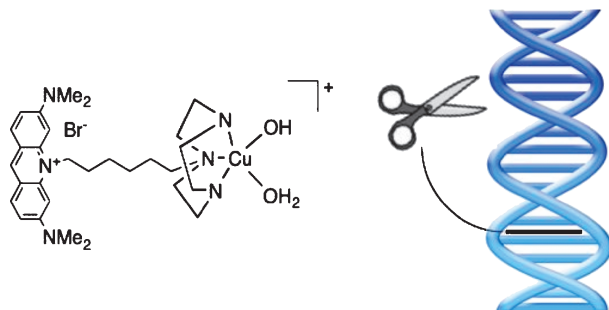


Catalytic asymmetric hydrogenation of α -amino- β -keto ester hydrochlorides using homogeneous chiral nickel-bisphosphine complexes through DKR

Yasumasa Hamada,* Yu Koseki, Takefumi Fujii, Tsukuru Maeda, Takuya Hibino and Kazuishi Makino

First asymmetric hydrogenation using homogeneous chiral nickel complexes through dynamic kinetic resolution has been developed for the conversion of α -amino- β -keto ester hydrochlorides to β -hydroxy- α -amino acid esters.

6209

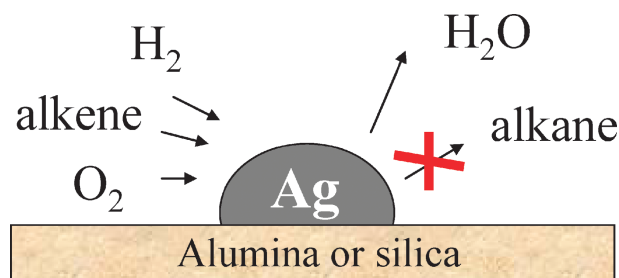


Synthesis and DNA cleavage activity of a bifunctional intercalator-linked copper(II) macrocycle

Ta-Sheng Andrew Tseng and Judith N. Burstyn*

An intercalator-linked macrocycle 3,6-bis(dimethylamino)-10-(6-[1,4,7-triazacyclononane]hexyl) acridinium bromide was synthesized, and the DNA cleavage activity of its copper(II) complex compared to that of other metal complexes and to intercalator-free (Cu[9]aneN₃)²⁺.

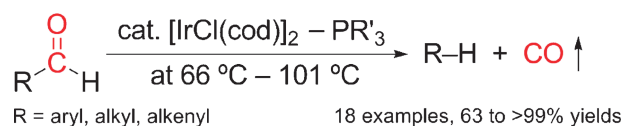
6212

Selective catalytic reduction of O₂ with excess H₂ in the presence of C₂H₄ or C₃H₆Burapat Inceesungvorn, Frederic C. Meunier,*
Chris Hardacre, Robbie Burch and Ken GriffinThe selective reduction of O₂ with excess H₂ in the presence of alkenes was achieved successfully for the first time.

6215

The iridium-catalyzed decarbonylation of aldehydes under mild conditions

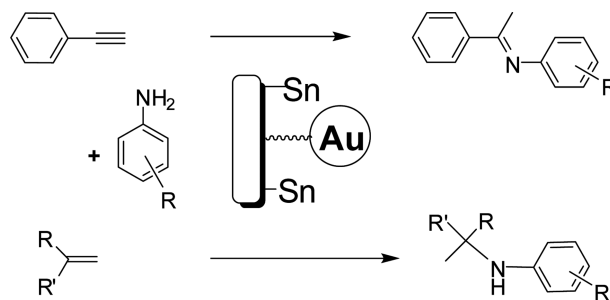
Tomohiro Iwai, Tetsuaki Fujihara and Yasushi Tsuji*

The iridium-catalyzed decarbonylation of aldehydes using commercially available [IrCl(cod)]₂ and an easily accessible monodentate phosphine such as PPh₃ or P(*n*-Bu)₃ has been developed. The reaction proceeds smoothly at considerably lower temperatures (at 66 °C–101 °C) than previous systems (typically >160 °C).

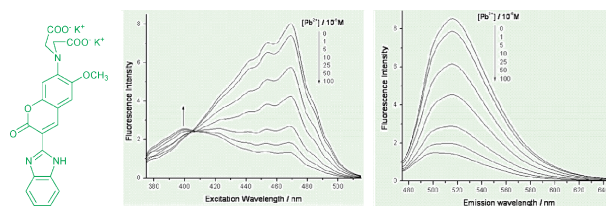
6218

Synthesis of bifunctional Au–Sn organic–inorganic catalysts for acid-free hydroamination reactionsAvelino Corma,* Camino González-Arellano,
Marta Iglesias,* M. Teresa Navarro and Félix Sánchez*

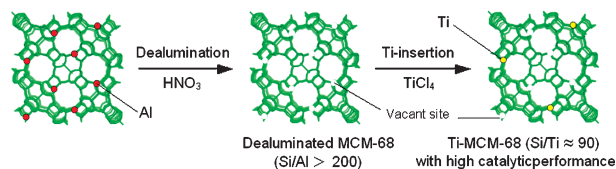
Gold(III) complexes heterogenized on the surface of a Sn-containing MCM-41 are efficient recyclable catalysts for hydroamination reactions, without requiring any acid promoters.



6221

Coumarin-based ratiometric fluorescent indicators with high specificity for lead ionsEmmanuel Roussakis, Spiros A. Pergantis and
Haralambos E. Katerinopoulos*The synthesis and the spectral profile studies of a series of potential intracellular fluorescent ion probes with well defined spectral changes in aqueous Pb²⁺ solutions is described in this report. The compounds exhibit a high selectivity for Pb²⁺ in the presence of other competitive ions.

6224

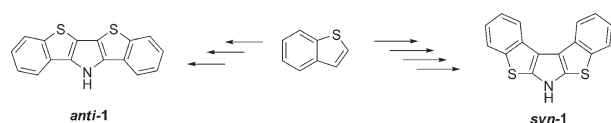


Synthesis and catalytic performance of Ti-MCM-68 for effective oxidation reactions

Yoshihiro Kubota,* Yoshihito Koyama, Taku Yamada, Satoshi Inagaki and Takashi Tatsumi

Microporous titanosilicate Ti-MCM-68 (Ti-MSE) was successfully synthesized by post-synthetic isomorphous substitution. Ti-MSE-type material proved to be a high-performance catalyst for the epoxidation of olefins and the oxidation of phenol using H₂O₂ as an oxidant.

6227

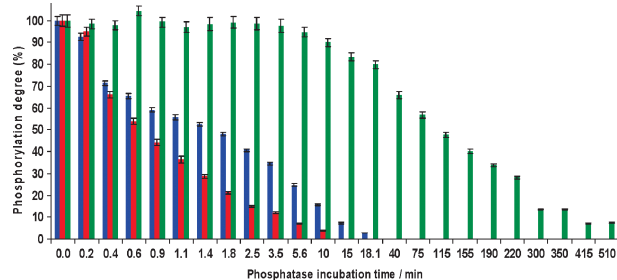


Synthesis and properties of the *anti* and *syn* isomers of dibenzothieno[*b,d*]pyrrole

Ting Qi, Yunlong Guo, Yunqi Liu,* Hongxia Xi, Hengjun Zhang, Xike Gao, Ying Liu, Kun Lu, Chunyan Du, Giu Yu and Daoben Zhu

The *anti* isomer exhibits better organic field-effect transistor properties compared with the *syn* isomer and its *N*-hexyl substitution.

6230

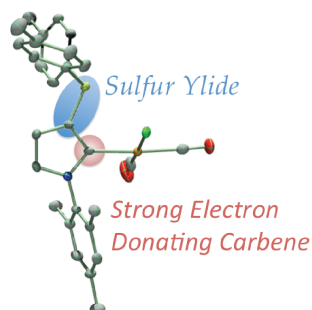


Element mass spectrometry as a tool for high-resolution temporal dynamics investigations of peptide phosphorylation

Ana Pereira Navaza, Jorge Ruiz Encinar and Alfredo Sanz-Medel*

Capillary HPLC coupled to element mass spectrometry has been successfully applied for the first time to high-resolution temporal dynamics studies of peptide phosphorylation.

6233



A novel ylide-stabilized carbene; formation and electron donating ability of an amino(sulfur-ylide)carbene

Junji Kobayashi, Shin-ya Nakafuji, Atsushi Yatabe and Takayuki Kawashima*

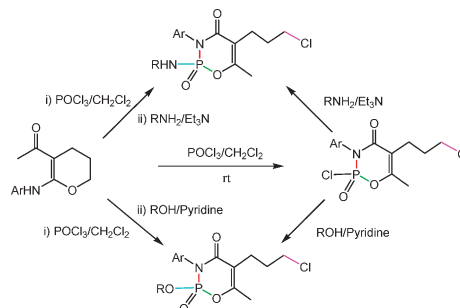
An amino(sulfur-ylide)carbene (ASYC), which is stabilized by electron-donation from the carbanion of a sulfur ylide, was synthesized. IR carbonyl stretching frequencies of the Rh complex of ASYC showed the strong electron-donating ability of ASYC.

6236

Efficient and divergent synthesis of cyclophosphamide analogues from 2-arylamino-3-acetyl-5,6-dihydro-4H-pyrans

Dexuan Xiang, Peng Huang, Kewei Wang, Guangyuan Zhou, Yongjiu Liang and Dewen Dong*

A facile and efficient one-pot synthesis of substituted cyclophosphamidic chlorides and their analogues has been developed from readily available enaminones, 2-arylamino-3-acetyl-5,6-dihydro-4H-pyrans.

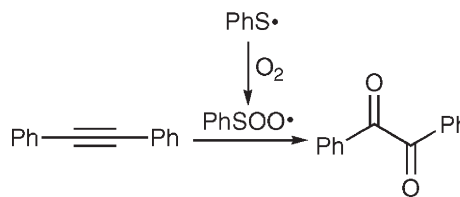


6239

Activation of molecular oxygen by S-radicals: experimental and computational studies on a novel oxidation of alkynes to α -diketones

Kristine J. Tan and Uta Wille*

Mechanistic studies provide strong indication that intermediately formed thiylperoxyl radicals, PhSOO^\bullet , are directly involved in a newly discovered oxidation of bis-aromatic alkynes to α -diketones using thiyl radicals in the presence of molecular oxygen.

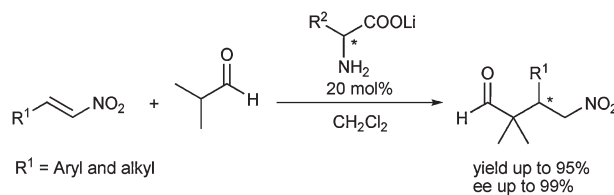


6242

Primary amino acid lithium salt as a catalyst for asymmetric Michael addition of isobutyraldehyde with β -nitroalkenes

Atsushi Sato, Masanori Yoshida* and Shoji Hara


Phenylalanine lithium salt was found to be an effective catalyst for asymmetric Michael addition of isobutyraldehyde with beta-nitroalkenes to give quaternary carbon-containing nitroalkanes.



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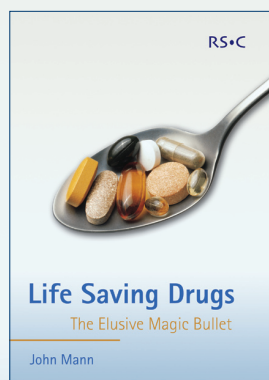
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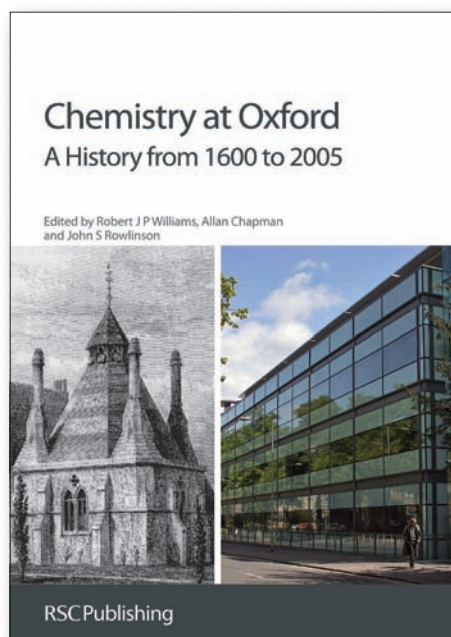
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Chemical Technology

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Danish scientists have developed a precise method to test for chromium isotopes in rocks and meteorites. The method will help to reveal more details about the evolution of the Solar System, they claim.

Supernovae explosions in the Solar System generate chromium-50, chromium-52 and chromium-53 isotopes. The amounts of these isotopes vary and are recorded in materials formed as a result of these explosions, such as meteorites, asteroids and planets.

Using thermal ionisation mass spectrometry, Anne Trinquier and colleagues from the Geological Museum at the University of Copenhagen purified and measured chromium isotopes in meteorites at concentrations as low as 10 parts per million, a level of accuracy that has never before been achieved. Their method is simple and quick, which minimises cost and contamination. Also, it uses only a small amount of meteorite, a bonus when the starting material is so hard to replace.

The results enable Trinquier to distinguish between planetary



Explosive results: isotopes released from supernovae can be measured in meteorites

bodies that were formed from different mixes of components and hence different explosions, something of great interest to scientists studying cosmochemistry.

'Precise chromium isotope ratio measurements are of great importance in any research related to the formation and early evolution of our Solar System,' comments Thorsten Kleine, an expert in Solar System chronology from the Institute of Isotope Geochemistry and Mineral Resources, ETH Zürich, Switzerland.

Trinquier says the next step is to improve the reproducibility of the results, with the hope that the measurements 'might reveal additional differences between planetesimals [small solar system bodies] and planets and help constrain further our understanding of planetary formation processes and timing'.

Rebecca Brodie

Reference

A Trinquier, J -L Birck and C L Allègre, *J. Anal. At. Spectrom.*, 2008, DOI:10.1039/b809755k

NASA

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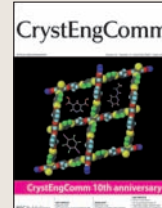
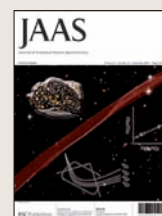
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The latest applications and technological aspects of research across the chemical sciences

Application highlights

Solid–solid reactions generate new types of metal-organic frameworks

Back to the grind-stone

UK scientists have studied a greener way to make microporous materials that could lead to new types of metal-organic frameworks.

Stuart James and Anne Pichon, at Queen's University Belfast, investigated a wide range of solvent-free mechanochemical reactions. Mechanochemistry is the initiation of chemical reactions by grinding two or more solids together using, for example, a mechanical ball-mill. Since mechanochemistry avoids the use of solvents, it could be a greener alternative to solution reactions.

James and Pichon surveyed 60 metal complexation reactions between 12 different metal salts and five bridging ligands using a ball-mill to grind the solid components together. They found that many of



the mixtures were highly reactive and gave crystalline products within a few minutes. The study revealed some interesting trends, including an inverse correlation between ligand melting point and reactivity, which could prompt further investigation. They also

Grinding solids in a ball-mill can generate crystalline products within minutes

Reference

A Pichon and S L James, *CrystEngComm*, 2008, DOI: 10.1039/b810857a

obtained new types of structures than those formed using solvents.

'Mechanochemistry is actually a very old method,' explains James. 'However, most chemists are not tempted to try it because it is counter-intuitive that two solids can react with each other. It's comparatively recent that the impetus for avoiding solvents has become strong enough to start making chemists think again.'

Graham Bowmaker, an expert in mechanochemical synthesis from the University of Auckland, New Zealand, says that James' study 'provides clues about the possible mechanisms of mechanochemical reactions, which in turn will greatly enhance further exploitation of the method'.

Ruth Doherty

Fundamental Toxicology

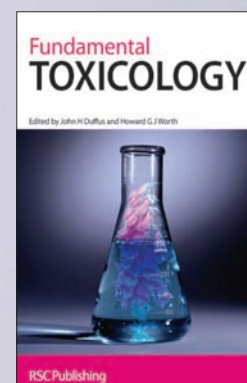
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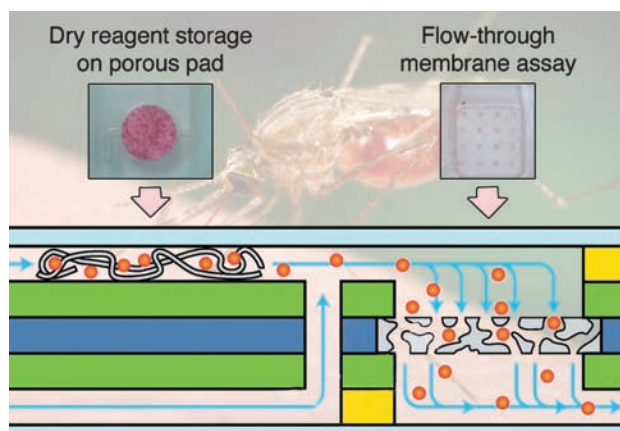
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Sugar-dried labelling agents detect malaria on a card

Sweet solution for on-card reagent storage

US scientists have developed a method for storing dry reagents on low-cost disposable cards. They claim the cards could be used for point-of-care diagnostics in the developing world, where high temperatures and a lack of refrigeration make it difficult to preserve reagent functions.

Paul Yager and colleagues from the University of Washington, Seattle, and Boston College, Chestnut Hill, demonstrated that their storage system works in an automated on-card microfluidic test for malaria. The card's main components are a porous membrane patterned with malaria antibodies and a fibrous pad containing gold-antibody conjugates in sugar. The sugar stabilises the dry conjugates and preserves their function as labelling agents. When Yager pumped samples containing malaria antigens through the card, the conjugates bound to the antigens, causing red spots to form on the



card. They used a scanner to capture images of the cards and calculate the changes in spot intensity.

'The proposed on-card dry reagent storage method is a good solution for reagent storage issues,' says Christopher Ko, an expert in microfluidics and molecular diagnostics at the Samsung Advanced Institute of Technology,

Malaria antigens cause red spots to form on the card

Reference
D Y Stevens *et al*, *Lab Chip*, 2008, DOI: 10.1039/b811158h

Suwon, South Korea. 'Of course, in order to be useful in the developing world, much more, in addition to dry reagent, is needed, such as eliminating the costly microfluidic pumps and scanners used in this study,' he adds.

Yager's team are working to develop a diagnostic system called the DxBox, which consists of a portable reader and disposable test cards. They aim to incorporate both immunoassays and nucleic acid assays on to the cards to allow them to test blood samples for multiple diseases at once.

'Our target markets will be under-resourced communities in areas such as rural India, Brazil and sub-Saharan Africa,' says Dean Stevens, a scientist in Yager's team. 'Our goal is to make the portable reader and each card fit within a reasonable diagnostic budget – a dollar per card would be an ideal, if challenging, goal.'

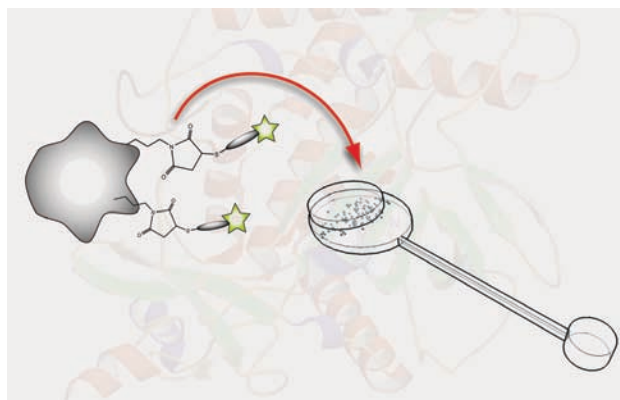
Freya Mearns

Bead-based sensor detects deadly poison

Microfluidics joins fight against bioweapon

US scientists have developed a new sensor capable of detecting trace levels of a lethal neurotoxin. The sensor is quicker and more sensitive than the mouse bioassay currently used for detecting the toxin and, unlike the bioassay, does not require animal sacrifice.

David Beebe and colleagues at the University of Wisconsin–Madison designed a portable microfluidic device that can detect botulinum toxin A (BoNT/A). Although known for its use in Botox cosmetic treatments, BoNT/A is one of the most poisonous naturally occurring substances – eating around 70 micrograms can kill the average person. It causes the muscle paralysis illness botulism, which poses a serious bioterrorism threat. Many people can be affected by a single contaminated food source so a quick and effective detection



method is required.

Beebe's sensor contains toxin-specific beads, which react with a BoNT/A solution to release fluorescently labelled fragments. The solution then flows down a microfluidic channel to a detection port, where evaporation of the solution concentrates

The beads react with the toxin and release fluorescent fragments

the fluorescent fragments and amplifies the signal.

Hugh Fan, an expert in microfluidics from the University of Florida, Gainesville, US, appreciates the clever design of the sensor. 'It exploits unique features of enzymatic cleavage on the bead surface and an evaporation-induced flow in a microchannel,' he says. 'The technique can also be extended to other biological assays,' Fan adds, and Megan Frisk from the Beebe group agrees.

'I'm hoping that we can simplify our current microfluidic systems to meet the needs of developing countries, particularly in the area of rapid and reliable tests for tuberculosis and HIV,' she says.

Roxane Owen

Reference
M L Frisk *et al*, *Lab. Chip*, 2008, DOI: 10.1039/b811075a

Volatile functional groups assist thin film synthesis

New spin on electronics production

Chemists have taken a significant step closer to the goal of cheap, flexible and printable organic electronic displays, an idea they claim could revolutionise the electronics industry.

A Japanese team, led by Tetsuo Okujima and Noboru Ono at Ehime University, Matsuyama, synthesised thin films of phthalocyanine (Pc) and the related compound, naphthalocyanine (Nc), without using costly ultra-high vacuum techniques.

Pc and Nc are insoluble so Ono and Okujima added functional groups to the molecules to improve solubility. They then dissolved the molecules in an organic solvent and spun the solution rapidly on a glass plate, evaporating the solvent and forming a thin film of the molecules



on the glass. When they heated the films, a retro Diels-Alder reaction released the volatile solubility-imparting groups. The final films were totally insoluble and acted as semiconductors.

The group then made an organic field-effect transistor (OFET)

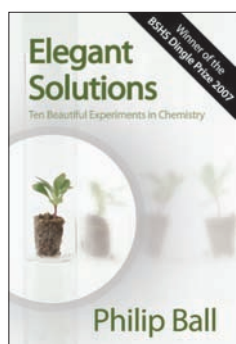
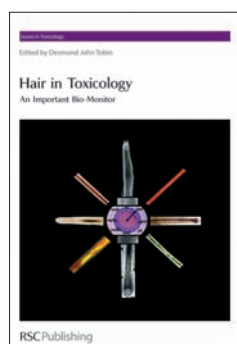
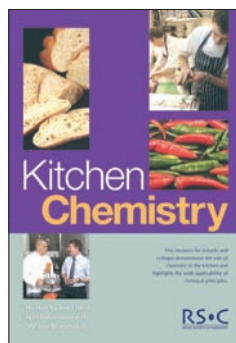
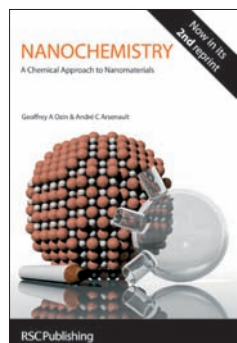
Heating the spin-coated film converts it to an insoluble semiconductor

Reference
A Hirao et al, *Chem. Commun.*, 2008, DOI: 10.1039/b811674a

using the films. OFETs are essential components of flexible organic electronic displays, which are starting to appear on the market but are hampered by high-cost production.

Okujima says that his work could result in organic electronic devices becoming cheaper and easier to make. 'This is the first example of solution-processed parent Pc- or Nc-based OFETs which are fabricated easily, at low cost and over a large area,' he explains.

'This work represents a step forward to realising organic semiconductor-based, low-cost, printable and flexible electronics,' says Jerzy Kanicki, an expert in organic electronics at the University of Michigan, Ann Arbor, US. *James Hodge*



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Taking the lab to the field

Gillian Greenway talks to Freya Mearns about taking analytical chemistry out of the lab and into the real world



Gillian Greenway

Professor Gillian Greenway is Head of Environmental Monitoring at the Hull Environment Research Institute and the new president of the Analytical Division of the Royal Society of Chemistry. Her research interests include chemical miniaturisation and environmental analysis.

Who inspired you to become a scientist?

I was really inspired by family. My father and uncles were engineers and my aunt was a pharmacist. I lived near the sea and when we went to the beach to play, my uncle, who was a civil engineer, had us using scientific principles to design dams.

What projects are you working on?

My main project is trying to take measurements out of the laboratory and into the environment or to crime scenes. I'm working with engineers and physicists to try to make truly portable systems. Although there have been a lot of lab-on-a-chip proof-of-concepts in the lab, there are still very few instruments that actually work out in the field in a reliable way – trying to convert the lab-based systems into something that works is challenging. The sort of concepts that I'm working on are: trying to make the systems robust, immobilising reagents, including redundancy, using engineering approaches to fault testing, using feedback to find out when things aren't working and determining how to overcome problems. It is also important not just to repeat the way the chemistry is carried out in the lab but to find different ways that will work better in the environment.

You work across a broad range of subjects. How do you strike a balance between specialising in one area and knowing enough about the other areas?

I think you do have to specialise in one area, so the key thing is learning to communicate with people from other disciplines. That can be surprisingly difficult. It takes patience because each discipline seems to have its own language; often the same word has different meanings. So it's important to overcome these barriers and be able to tell biologists and engineers, for example, what you need and to understand what they are telling you.

What's the trickiest problem you've had to overcome in your research and how did you get around it?

I've got dyspraxia, which is a coordination-specific

learning problem. That means that, practically, I can be a bit of a disaster in the lab. The fact that I became a chemist must show my determination. I need lots of practise with practical skills. Fortunately, I chose a degree with industrial placements and the day-to-day experience in the laboratories gave me the confidence I needed. After that, the number of breakages, spills and floods decreased.

What's the role of the Analytical Division?

The role of the Analytical Division is to promote analytical chemistry and science. It's about promoting research in the area and communicating with the public so that people understand the importance of good chemical measurements in their everyday lives. It's also about working with industry, encouraging collaboration between industry and academia, and going into schools and encouraging children to be interested in science. The Analytical Division's there to support the members too, to organise meetings and to help people network. It's been very useful in my career to network through the Royal Society of Chemistry.

What would you like to achieve as president?

Obviously, what's really important is to keep promoting analytical science and to get people to understand its importance. Within the Analytical Division, I'd like to get people more involved by getting them enthused and willing to participate and to promote their ideas. We're very lucky in the Analytical Division because we have the Analytical Chemistry Trust Fund (ACTF) – if you have good ideas, you can go to the ACTF to get funds to promote them. We have a lot of initiatives at the moment including the development of a new studentship scheme and the encouragement of science in developing countries.

Finally, if you weren't a scientist, what would you be?

I think I would be a teacher. It is a really good feeling when you are able to inspire people although it is hard work.

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Photochemistry goes micro

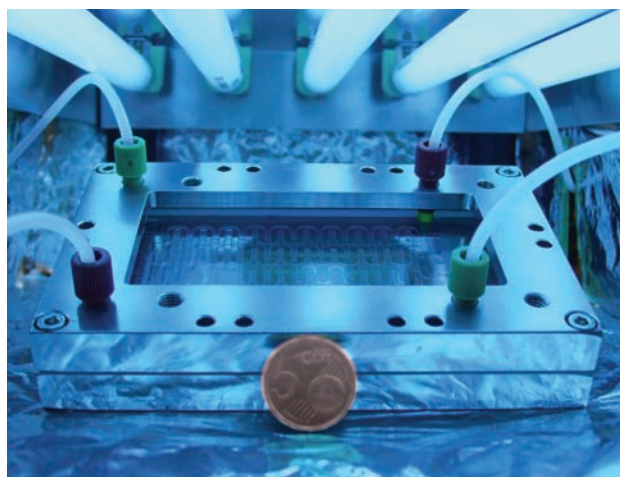
Michael Oelgemöller and Emma Coyle of Dublin City University, Ireland, discuss how microreactors may change synthetic organic photochemistry

At the International Congress of Applied Chemistry in New York in 1912,¹ Giacomo Ciamician, the father of organic photochemistry, presented his spectacular vision of 'The Photochemistry of the Future': 'On the arid lands there will spring up industrial colonies without smoke and without smokestacks; forests of glass tubes will extend over the plains, and glass buildings will rise everywhere; inside of these will take place the photochemical processes that hitherto have been the guarded secret of the plants, but that will have been mastered by human industry which will know how to make them even more abundant fruit than nature, for nature is not in a hurry and mankind is.'

Generally speaking, light can be used to efficiently and selectively induce chemical changes. It can be easily tuned and controlled, literally with a flick of a switch. Photochemistry also allows scientists to construct exotic, high-energy molecules with relative ease. Due to the specialised equipment and reaction conditions, however, chemical transformations with light have been widely ignored by the chemical industry. More than 90 years later, Ciamician's vision is yet to be realised.

Microreactors, otherwise known as microchannelled or microstructured reactors, have recently become widespread in research. Originally developed for analytical applications as the famous 'lab on a chip', these devices have also found promising uses in synthetic organic chemistry.

Since many commercially available microreactors are glass-based and transparent, they can be easily adopted for photochemical applications. Miniature light sources, such as light-emitting diodes, can be used and offer real



A glass microreactor (Dwell device, mikroglas) under a UV exposure panel (Luzchem). A five Euro-coin is used to illustrate the size of the reactor

advantages over conventional light sources – they are small, energy efficient, come in a range of wavelengths and produce very little heat, thus reducing the need to cool the reaction. Because the reaction channels in a microreactor are shallow, light can penetrate even concentrated solutions. Solution flow rate controls the exposure to light and can be easily varied to rapidly optimise photochemical reactions. Additionally, the reactions can be monitored on-line, for example by analysing the effluent using a UV spectrometer. Microreactors can also be used in parallel to scale-up reactions, a process known as numbering up.

There are a number of different microreactor types available today. As their name suggests, serpentine channel reactors have long, snaking reaction channels, which range from several centimetres to more than a metre in length. The dwell-reactor produced by mikroglas, for example, is the size of an external floppy drive but its reaction channel is 1.15 metres long. The reactor consists

of a reaction channel and a second, cooling channel through which water flows. Another design is the falling film reactor, which generates a thin falling film of solution like a waterfall that passes by the light source. This device is especially advantageous for gas–liquid reactions, such as photooxidations or photohalogenations. Many researchers, however, continue to custom build their own reactors based on their needs and applications.

The photochemical transformations studied to date in microreactors include homogeneous reactions, such as photocyanation and photodecarboxylation; heterogeneous reactions between liquid and gaseous reagents, such as photooxygenations; and photocatalytic processes using semiconductors. In many cases, the selectivities and yields are better than those from large scale experiments, clearly demonstrating the feasibility and superiority of microphotochemistry.

Ciamician's vision may thus be realised in the form of a microchip, rather than the glass buildings he envisaged. By scaling down photochemical reactions using microreactors, photochemical reactions can be conveniently carried out in research laboratories, for example for finding and developing leads for drug discovery. In addition, numbering up, rather than scaling up, may enable photochemical products to be produced industrially.

Read more in 'Micro-photochemistry: photochemistry in microstructured reactors. The new photochemistry of the future?' in issue 11 of Photochemical & Photobiological Sciences²

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- 1 G Ciamician, *Science*, 1912, **36**, 385
- 2 E E Coyle and M Oelgemöller, *Photochem. Photobiol. Sci.*, 2008, **7**, 1313 (DOI: 10.1039/b808778d)

Board member wins Nobel Prize

The Nobel Prize in Chemistry 2008 has been awarded to Roger Tsien (below right), University of California, San Diego, US, a member of the editorial board for the upcoming RSC journal *Integrative Biology* (to be launched in January 2009), and colleagues for their work in the development of the gene marker green fluorescent protein (GFP).

Harp Minhas, editor of *Integrative Biology*, says: 'Congratulations to Professor Tsien, from all of us at the RSC. We are all immensely pleased that 2008 Nobel Prize winner Roger Tsien is an editorial board member for *Integrative Biology*; his work typifies the quality of material we are seeking in the development of biology through new tools and technologies.'

Derivatives of GFP are used in experiments to observe



cell dynamics and behaviour – their fluorescent glow allows scientists to visualise processes inside cells.

Furthermore, as it is non-toxic to cells it can be used in live



cell (in vitro) studies meaning that real time analysis of cells is possible.

GFP is a protein first extracted from the jellyfish *Aequorea victoria* in the 1960s by Osamu Shimomura, who was jointly awarded this year's prize with Tsien and Martin Chalfie.

Variants of GFP can fluoresce in different colours, allowing several different proteins in a cell to be studied simultaneously.

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Announcing *Lab on a Chip* prize winners

Lab on a Chip, the miniaturisation journal for chemistry, biology and bioengineering, has yet again shown extensive community support by sponsoring some of the most prestigious prizes in the miniaturisation field. At this year's μ -TAS meeting in San Diego the journal, together with Corning Inc., awarded the 'Pioneers in Miniaturisation Prize' to Patrick Doyle, professor at the department of chemical engineering at MIT, US. Jean-Louis Viovy from the Institute Curie comments on Doyle's

work: '[Patrick] developed the "stop-flow lithography" technological platform, which I consider a major breakthrough in microfluidics.'

Lab on a Chip also awarded the 'Widmer Young Researcher Poster Award' to Maged Fouad for best poster and presentation. Among 589 candidates, this poster titled 'Nanotechnology meets plant biotechnology: carbon nanotubes deliver DNA and incorporate into the plant cell structure' caught the judges' eyes.

A new award named 'Art in

Science' recognised the aesthetic value in scientific illustrations.

'The winner, Yu Wen Huang (Texas A & M University) clearly understood the principles of this award and produced an image that was reminiscent of a tall city building seen in an early morning fog. The picture is an optical effect generated by concentrated double-stranded DNA in the vicinity of a 50 micrometre wide electrode inside a microchannel,' comments Harp Minhas, editor of *Lab on a Chip*, who proudly presented all awards to the winners.

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