

## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (13) 1653-1792 (2005)



### Cover

See Ron Devon, Jordan Rose Figura, Daryl Douthat, Jerry Kudenov and Jerzy Maselko, page 1678. Self-construction of complex form in a  $\text{Fe}^{2+}$  -  $\text{Fe}(\text{CN})_6^{3-}$  system. The crystal tubes deliver building material to the top of the 'tree' and build-up continues. Image produced by permission of Ron Devon *et al.* from *Chem. Commun.*, 2005, 1678.



### Inside cover

See Christopher Reed, page 1669. Graphic representing carborane acid structure. Image reproduced by permission of Christopher Reed from *Chem. Commun.*, 2005, 1669.

## CHEMICAL SCIENCE

C25

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

## Chemical Science

April 2005/Volume 2/Issue 4

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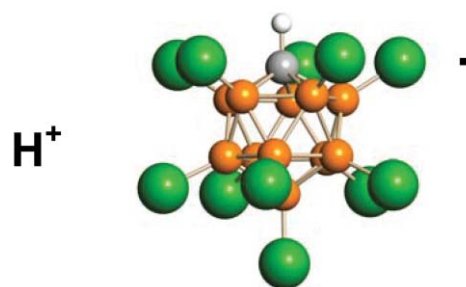
## FEATURE ARTICLE

1669

### Carborane acids. New "strong yet gentle" acids for organic and inorganic chemistry

Christopher A. Reed

Carborane acids eclipse traditional acids in both strength *and* gentleness, making possible the isolation of reactive cations such as carbenium ( $\text{R}_3\text{C}^+$ ), silylium ( $\text{R}_3\text{Si}^+$ ), benzenium ( $\text{C}_6\text{H}_7^+$ ), vinyl, fulleranium and hydronium ( $\text{H}_3\text{O}^+$ ,  $\text{H}_5\text{O}_2^+$ ,  $\text{H}_9\text{O}_4^+$ ) ions.



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1678

**Complex morphology in a simple chemical system**Ron Devon, Jordan RoseFigura, Daryl Douthat,  
Jerry Kudenov and Jerzy Maselko\*

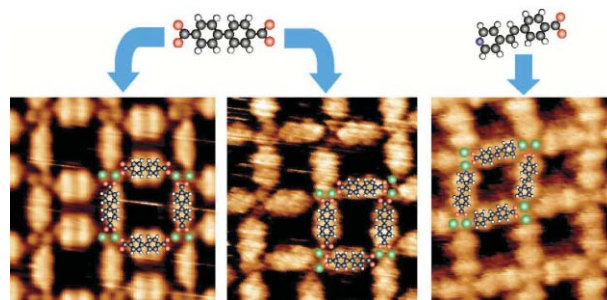
We are presenting a self-construction of unusually complex chemical forms that grow in a two-component  $\text{Fe}(\text{CN})_6^{3+}$ - $\text{Fe}^{2+}$  inorganic system. The growth mechanism is composed of many steps, organized in space and time. This structure, in its complexity, is reminiscent of the biological mangrove forest. Two distinguished forms have been found: the tree and the sponge.



1681

**Manipulating 2D metal-organic networks via ligand control**Nian Lin,\* Sebastian Stepanow, Franck Vidal,  
Johannes V. Barth and Klaus Kern

Scanning tunneling microscopy observations reveal in real-space at an atomic resolution how the choice of organic linker molecules can be used to control the topology of 2D metal-organic networks.

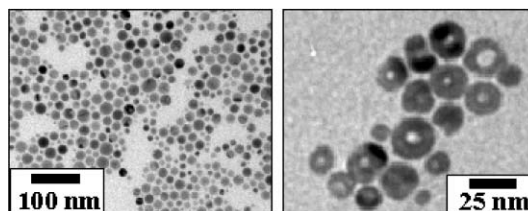


1684

**Hollow gold and platinum nanoparticles by a transmetallation reaction in an organic solution**

P.R. Selvakannan and Murali Sastry\*

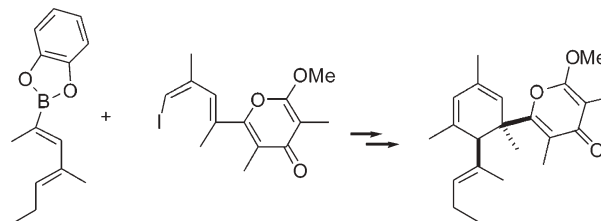
Transmetallation reaction between hydrophobized silver nanoparticles with hydrophobized chloraurate and chloroplatinate ions in chloroform results in the formation of hollow gold and platinum shell nanoparticles respectively.



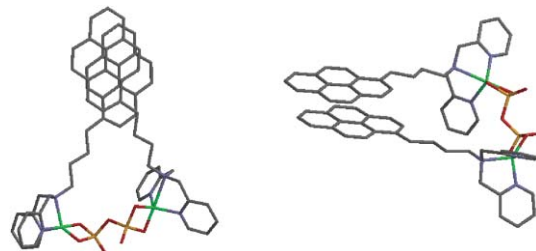
1687

**Biomimetic synthesis of ( $\pm$ )-9,10-deoxytridachione**John E. Moses, Robert M. Adlington, Raphaël Rodriguez,  
Serena J. Eade and Jack E. Baldwin\*

A tandem Suzuki-coupling/electrocyclisation reaction sequence was employed for the biomimetic synthesis of ( $\pm$ )-9,10-deoxytridachione.



1690

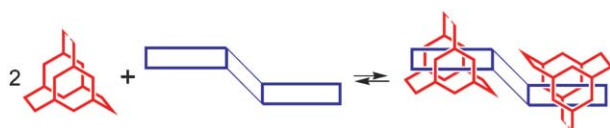


### A fluorescent pyrophosphate sensor *via* excimer formation in water

Hong Kwan Cho, Dong Hoon Lee and Jong-In Hong\*

A new fluorescent sensor based on a pyrene/ $Zn^{II}$ -dpa (dpa = bis(2-pyridylmethyl)amine) conjugate displays excimer emission selective for pyrophosphate over other anions.

1693

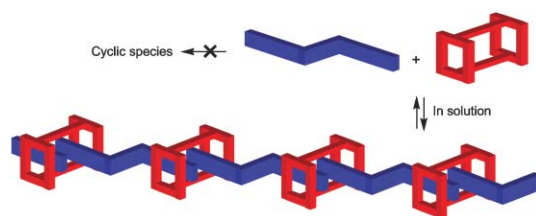


### Remarkably improved complexation of a bisparaquat by formation of a pseudocryptand-based [3]pseudorotaxane

Feihe Huang, Ilia A. Guzei, Jason W. Jones and Harry W. Gibson\*

Significant improvement of complexation of a bisparaquat guest was achieved by the formation of a pseudocryptand by chelation of a bisphenolic host with a trifluoroacetate anion and a water molecule. Thus a pseudocryptand-based [3]pseudorotaxane was prepared by self-assembly of seven components as shown by X-ray crystallography.

1696

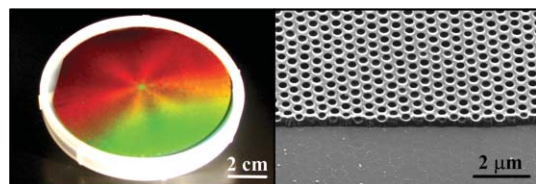


### A supramolecular poly[3]pseudorotaxane by self-assembly of a homoditopic cylindrical bis(crown ether) host and a bisparaquat derivative

Feihe Huang and Harry W. Gibson\*

A supramolecular poly[3]pseudorotaxane was prepared by self-assembly of a homoditopic cylindrical bis(crown ether) host and a bisparaquat derivative in solution by host-guest complexation. This was confirmed by proton NMR spectroscopy, mass spectrometry, and viscosity studies.

1699



### Wafer-scale fabrication of periodic polymer attolitre microvial arrays

Peng Jiang\*

Wafer-size periodic polymer attolitre microvial arrays of varying depth have been fabricated by templating from spin-coated 2D non-close-packed colloidal crystal-polymer nanocomposites.

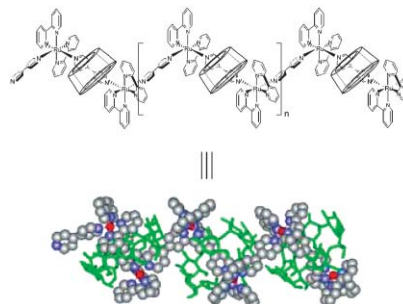


1702

**The construction of a supramolecular polymeric rotaxane from bipyridine-ruthenium and cyclodextrin**

Yu Liu,\* Shi-Hui Song, Yong Chen, Yan-Li Zhao and Ying-Wei Yang

A luminescent supramolecular link is constructed by a very simple method using bipyridine-ruthenium and cyclodextrin, which displays not only a quasi-linear structure, but also a satisfactory fluorescence emission in both solution and the solid state.

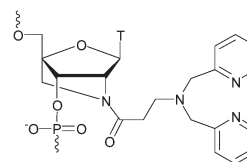


1705

**Optimized DNA targeting using *N,N*-bis(2-pyridylmethyl)- $\beta$ -alaninyl 2'-amino-LNA**

B. Ravindra Babu, Patrick J. Hrdlicka, Christine J. McKenzie and Jesper Wengel\*

A strategy for optimized high-affinity DNA targeting using bipyridyl-functionalized 2'-amino-LNA in the presence of divalent metal ions is introduced.

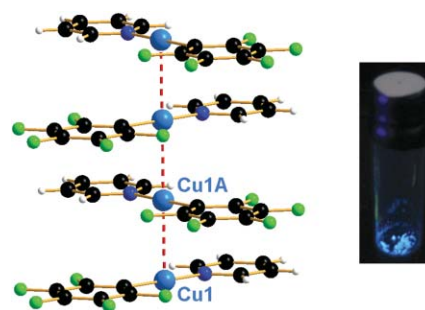


1708

**Cuprophilic and  $\pi$ -stacking interactions in the formation of supramolecular stacks from dicoordinate organocopper complexes**

Anand Sundararaman, Lev N. Zakharov, Arnold L. Rheingold and Frieder Jäkle\*

The unsupported organocopper pyridine complex  $C_6F_5Cu(py)$  forms one-dimensional chains of copper atoms with  $Cu \cdots Cu$  distances of 2.8924(3) Å and shows blue luminescence in the solid state.

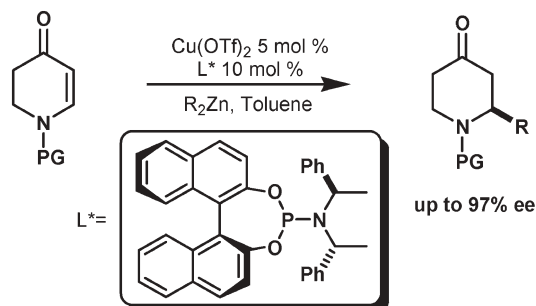


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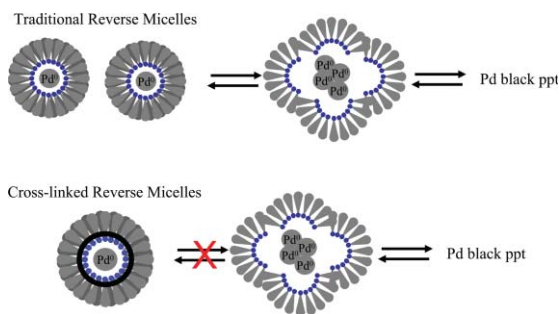
**Catalytic enantioselective conjugate addition of dialkylzinc reagents to *N*-substituted-2,3-dehydro-4-piperidones**

Radovan Šebesta, Maria Gabriella Pizzuti, Arnold J. Boersma, Adriaan J. Minnaard\* and Ben L. Feringa\*

Highly enantioselective, copper/phosphoramidite-catalyzed conjugate addition of dialkylzinc reagents to *N*-substituted-2,3-dehydro-4-piperidones is described. Enantiomerically enriched alkyl piperidones were obtained with up to 97% ee.



1714

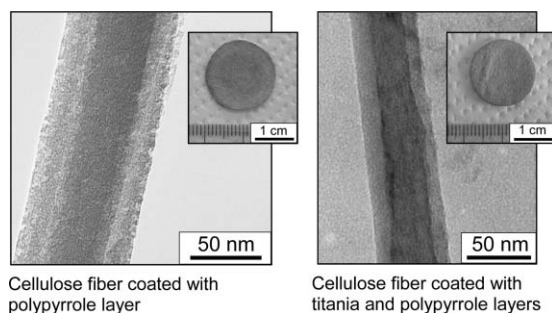


### A cross-linked reverse micelle-encapsulated palladium catalyst

Kristin E. Price and D. Tyler McQuade\*

Cross-linked reverse micelle-palladium catalysts are effective and stable cross-coupling catalysts; cross-linking is crucial for stability.

1717

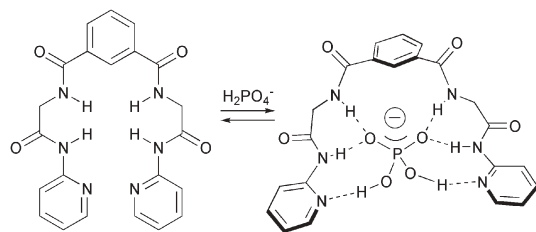


### Nanocoating of natural cellulose fibers with conjugated polymer: hierarchical polypyrrole composite materials

Jianguo Huang, Izumi Ichinose\* and Toyoki Kunitake

Astonishingly uniform polypyrrole nanocoating on morphologically complex cellulosic substances was achieved without disrupting the hierarchical network structures of individual cellulose fibers by means of polymerization-induced adsorption.

1720

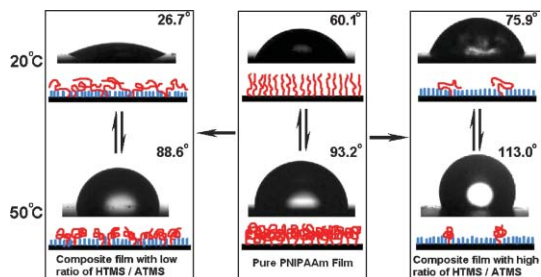


### Selective recognition of dihydrogen phosphate by receptors bearing pyridyl moieties as hydrogen bond acceptors

Shin-ichi Kondo,\* Yuichi Hiraoka, Namiko Kurumatani and Yumihiko Yano

Dihydrogen phosphate anion is selectively recognized by amide-based receptors bearing pyridyl moieties as hydrogen bond acceptors in 0.5% DMSO-acetonitrile.

1723



### Control over the responsive wettability of poly(*N*-isopropylacrylamide) film in a large extent by introducing an irresponsive molecule

Taolei Sun,\* Wenlong Song and Lei Jiang\*

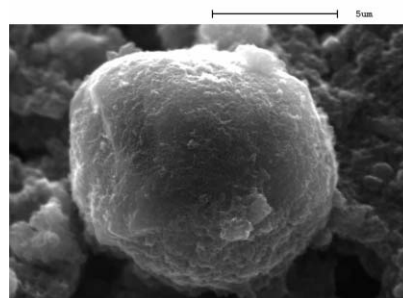
An irresponsive but very hydrophobic molecule—fluoroalkylsilane—can be used to cooperate with PNIPAAm to obtain tunable and enhanced responsive wettability, and thus largely extend the application scope of PNIPAAm film.

1726

### Assembling carbon nanotubosomes using an emulsion-inversion technique

Marc in het Panhuis\* and Vesselin N. Paunov\*

Novel micro-capsules (carbon nanotubosomes) have been fabricated by cross-linking shells of amine-functionalised multi-walled carbon nanotubes (MWNTs) produced by their adsorption on water-in-oil emulsion drops followed by an emulsion-inversion.

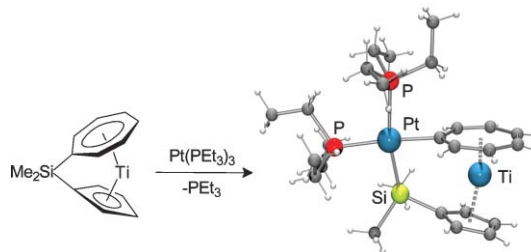


1729

### Regioselective Si–C bond activation in silicon-bridged *ansa*-cycloheptatrienyl-cyclopentadienyl complexes

Matthias Tamm,\* Andreas Kunst and Eberhardt Herdtweck

Treatment of silicon-bridged *ansa*-cycloheptatrienyl-cyclopentadienyl Ti and V complexes with  $[\text{Pt}(\text{PEt}_3)_3]$  results in oxidative addition and regioselective insertion of a  $\text{Pt}(\text{PEt}_3)_2$  moiety. The resulting Pt–Si-bridged complexes are promising catalysts for the ring-opening polymerization (ROP) of the original highly strained sandwich molecules.

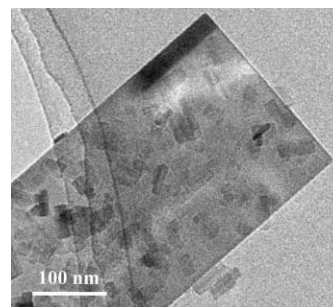


1732

### Hydrothermal synthesis of crystalline rectangular titanoniobate particles

Bo Li, Yukiya Hakuta and Hiromichi Hayashi\*

$\text{KTiNbO}_5$  powders possessing rectangular particle shapes and large surface areas are successfully synthesized by a novel hydrothermal method under subcritical and supercritical water conditions.

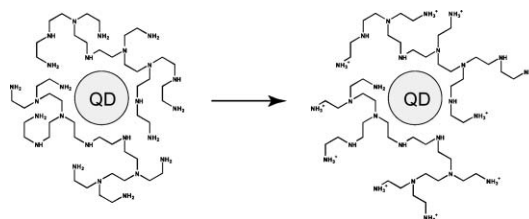


1735

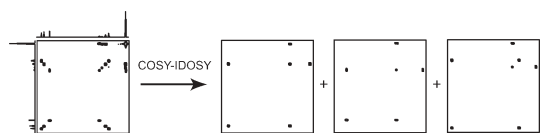
### Phase-transfer of CdSe@ZnS quantum dots using amphiphilic hyperbranched polyethylenimine

Thomas Nann

The amphiphilic character of hyperbranched PEI and its ability to act as ligand for quantum dots enable it to be used as a QD transfer agent from non-polar solvents to water.



1737

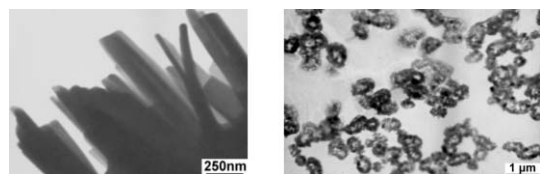


### Improving pulse sequences for 3D DOSY: COSY-IDOSY

Mathias Nilsson, Ana M. Gil, Ivonne Delgadillo and Gareth A. Morris\*

COSY-IDOSY separates a COSY spectrum into the spectra of individual components of different size.

1740

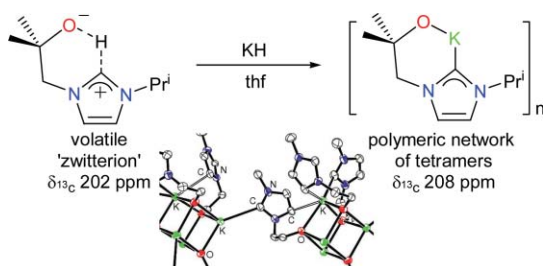


### Controlled synthesis of nanorods/nanorings of a novel Co–Cu complex in microemulsion at room temperature

Genban Sun, Minhua Cao, Yonghui Wang, Changwen Hu,\* Ling Ren and Kunlin Huang

Novel Co–Cu complex nanorods with diameters of 100–200 nm and nanorings with a ring-diameter of 80 nm were synthesized *via* a microemulsion method at room temperature. Using this method, addition of  $\text{Co}(\text{NH}_3)_6^{3+}$  to aqueous solutions of Cu(II) in excessive carbonate results in the formation of a new highly charged anion,  $[\text{Cu}_4(\text{OH})(\text{CO}_3)_8]^{9-}$ .

1743

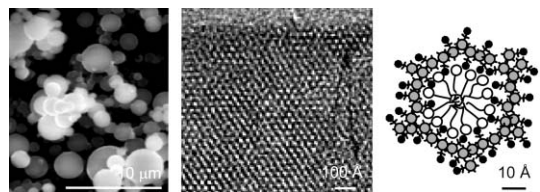


### Thermally stable potassium N-heterocyclic carbene complexes with alkoxide ligands, and a polymeric crystal structure with distorted, bridging carbenes

Polly L. Arnold,\* Mark Rodden and Claire Wilson

Alkoxide-functionalised N-heterocyclic carbenes (NHCs) stabilise the potassium-imidazole-2-ylidene fragment against 1,2 alkyl rearrangement reactions; this allows the first structural characterisation of a potassium NHC complex, which reveals distorted and unusual bonding interactions between the imidazole ring and the potassium cation.

1746



### New routes to mesoporous silica-based spheres with functionalised surfaces

Bruno Alonso,\* Christian Clinard, Dominique Durand, Emmanuel Véron and Dominique Massiot

Mesoporous hybrid silica-based spheres with functionalised siloxane surfaces and abundant highly ordered domains have been obtained using for the first time a simple synthetic route based on spray-drying processes.

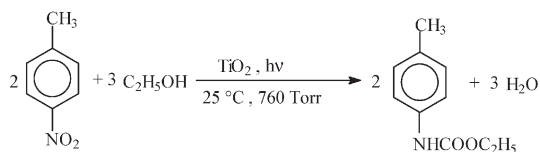


1749

**Photocatalytic formation of a carbamate through ethanol-assisted carbonylation of *p*-nitrotoluene**

Andrea Maldotti,\* Rossano Amadelli, Luca Samiolo, Alessandra Molinari, Andrea Penoni, Stefano Tollari and Sergio Cenini

The nitroarene *p*-nitrotoluene is converted with a selectivity higher than 85% to the corresponding carbamate at room temperature and atmospheric pressure, using photoexcited particles of TiO<sub>2</sub> as catalyst and EtOH as carbonylating species.

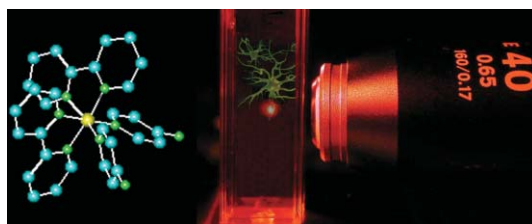


1752

**Two-photon uncaging of neurochemicals using inorganic metal complexes**

Volodymyr Nikolenko, Rafael Yuste, Leonardo Zayat, Luis M. Baraldo and Roberto Etchenique\*

Neuroactive compounds can be photoreleased by means of two photon excitation using a new kind of caged compounds based on transition metal chemistry.

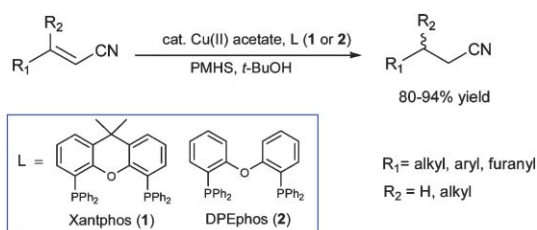


1755

**Highly efficient conjugate reduction of  $\alpha,\beta$ -unsaturated nitriles catalyzed by copper/xanthene-type bisphosphine complexes**

Daesung Kim, Bu-Mahn Park and Jaesook Yun\*

An efficient conjugate reduction of  $\alpha,\beta$ -unsaturated nitriles using copper/xanthene-type bisphosphine catalysts was carried out.

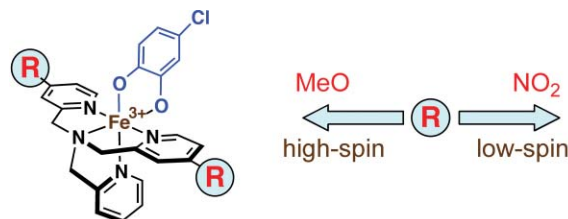


1758

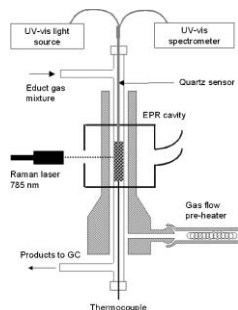
**Tuning of spin crossover equilibrium in catecholatoiron(III) complexes by supporting ligands**

Yutaka Hitomi,\* Masakazu Higuchi, Hisataka Minami, Tsunehiro Tanaka and Takuzo Funabiki\*

Introduction of electron-withdrawing groups on co-ligands effectively raises the spin crossover temperature of catecholatoiron(III) complexes and induces significant amount of the low spin species even in solution at around room temperature.



1761

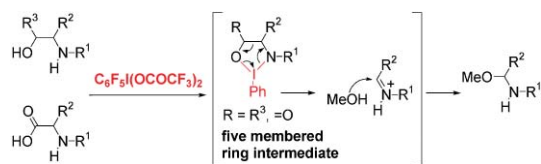


### Killing three birds with one stone—simultaneous operando EPR/UV-vis/Raman spectroscopy for monitoring catalytic reactions

Angelika Brückner

The first triple coupling of operando techniques allows simultaneous recording of EPR, UV-vis and Raman spectra of a working catalyst along with its catalytic performance.

1764

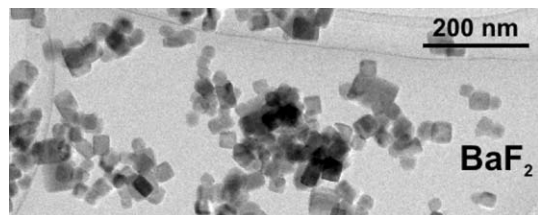


### The novel and efficient direct synthesis of *N,O*-acetal compounds using a hypervalent iodine(III) reagent: an improved synthetic method for a key intermediate of discorhabdins

Yu Harayama, Masako Yoshida, Daigo Kamimura and Yasuyuki Kita\*

The use of hypervalent iodine(III) reagents allowed us to develop the novel and efficient direct synthesis of *N,O*-acetal compounds *via* the oxidative fragmentation reaction of  $\alpha$ -amino acids or  $\alpha$ -amino alcohols.

1767

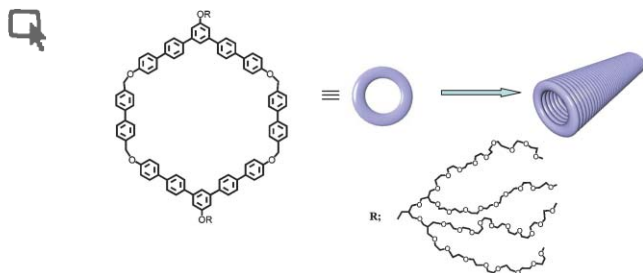


### Flame synthesis of calcium-, strontium-, barium fluoride nanoparticles and sodium chloride

Robert N. Grass and Wendelin J. Stark\*

Non-oxidic salts such as NaCl, CaF<sub>2</sub>, SrF<sub>2</sub> and BaF<sub>2</sub> were synthesised using a flame spray method. Optional doping of such fluoride nanoparticles with rare earth elements suggests possible applications in optics.

1770



### Tubular assembly of amphiphilic rigid macrocycle with flexible dendrons

Ja-Hyoung Ryu, Nam-Keun Oh and Myongsoo Lee\*

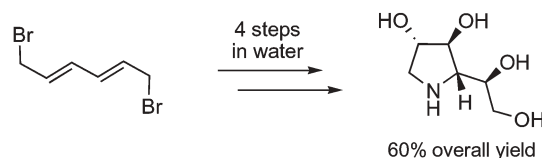
An amphiphilic rigid macrocycle was shown to self-assemble into tubular aggregates that can solubilize SWNTs in aqueous solution through significant  $\pi$ - $\pi$  interactions.

1773

### Efficient asymmetric synthesis of an azasugar in water

Ulf M. Lindström,\* Rui Ding and Olle Hidestål

An extremely efficient asymmetric synthesis of a pyrrolidine azasugar was completed in only four steps and 60% overall yield from a simple, achiral bis-electrophile. All steps were performed in water, no protecting groups were employed, and the need for chromatographic purification was minimal. Thus, we have demonstrated that water can be a superior solvent for green and efficient multistep syntheses of attractive targets.

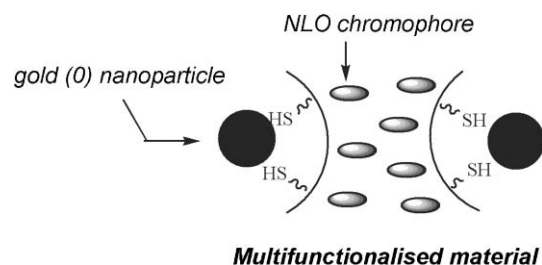


1775

### One-step template-directed synthesis of multifunctionalised nanoporous silica: on the way to interactive nanomaterials

Eric Besson, Ahmad Mehdi, Victor Matsura, Yannick Guari, Catherine Reyé and Robert J. P. Corriu\*

A one-step synthesis of multifunctionalised nanoporous silica with NLO chromophore in the framework and mercaptopropyl groups in the channel pores was achieved thanks to the direct liquid crystal templating approach. We have shown that the mercaptopropyl groups were able to stabilize gold(0) nanoparticles.

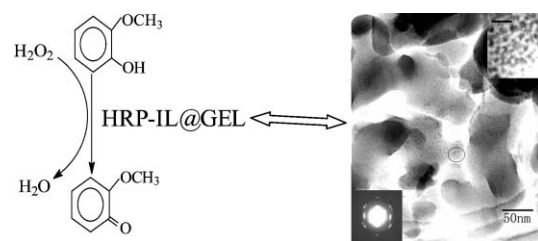


1778

### Highly active horseradish peroxidase immobilized in 1-butyl-3-methylimidazolium tetrafluoroborate room-temperature ionic liquid based sol-gel host materials

Yang Liu, Meijia Wang, Jun Li, Zhiying Li, Ping He, Hongtao Liu and Jinghong Li\*

A novel enzyme host matrix was prepared by using 1-butyl-3-methylimidazolium tetrafluoroborate room-temperature ionic liquid (RTIL) as the template for the matrix and the stabilizer of the enzyme, resulting in high activity of the enzyme.

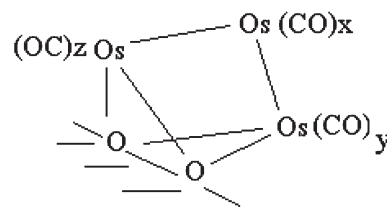


1781

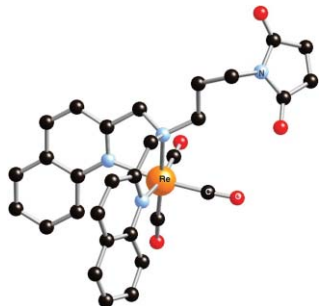
### Unexpectedly superior enantioselectivity for *trans*-stilbene *cis*-dihydroxylation over anchored triosmium carbonyl species in confined Al-MCM-41 channels

Valérie Caps, Ioannis Paraskevas and Shik Chi Tsang\*

Anchored triosmium carbonyl precursor in confined Al-MCM-41 channels.



1784

**Site directed maleimide bifunctional chelators for the  $M(CO)_3^+$  core ( $M = {}^{99m}Tc, Re$ )**

Sangeeta Ray Banerjee, John W. Babich and Jon Zubieta\*

The  $Re(CO)_3^+$  complexes and glutathione conjugates of a series of bifunctional chelates containing a tridentate donor set for complexation of the  $M(CO)_3^+$  core and a maleimide group for site-specific coupling to peptides and proteins containing free thiol groups have been prepared and structurally characterized. Ligands suitable for both fluorescence and radiotargeting studies of proteins and peptides have been designed.

## ADDITIONS AND CORRECTIONS

1787

**Sub- $T_c$  electron transfer at the HTSC/polymer interface**

Nicolas Le-Poul, Stephen J. Green and J. Paul Attfield

**A novel isocyanide based three component reaction**

Oliver T. Kern and William B. Motherwell

**STM-based molecular detection of “catch-and-release” of protons for bipyridine bound to phenylene-ethynylene thiol**

Emiko Koyama, Takao Ishida, Hideo Tokuhisa, Abdelhak Belaïssaoui, Yoshinobu Nagawa and Masatoshi Kanosato

**Novel chemoselective tosylation of the alcoholic hydroxyl group of *syn*- $\alpha,\beta$ -disubstituted  $\beta$ -hydroxy carboxylic acids**

Yikang Wu and Ya-Ping Sun

**Bisindoles containing a 2,1,3-benzothiadiazole unit: novel non-doping red organic light-emitting diodes with excellent color purity**

Qiang Fang, Bing Xu, Biao Jiang, Haitao Fu, Xiaoyao Chen and Amin Cao




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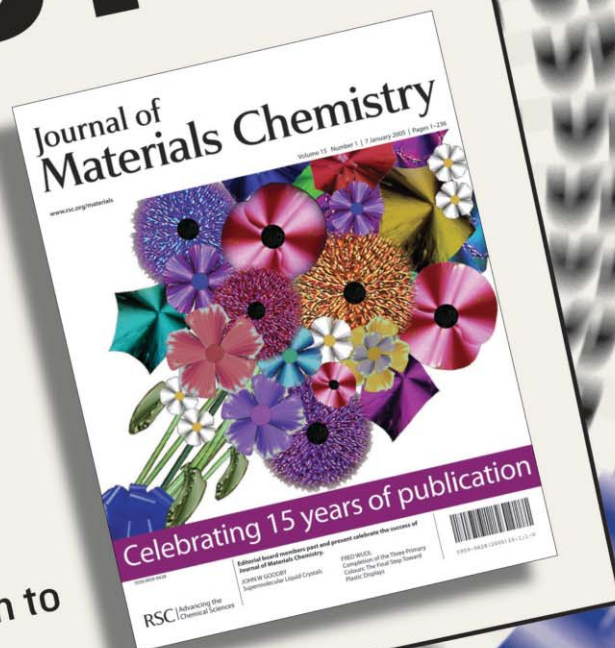
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