

## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (7) 813-956 (2005)

### In this issue...

#### Tracking cancer cells

The uptake and distribution of zinc bis(thiosemicarbazone) complexes in living cancer cells can be studied by fluorescence microscopy. See Dilworth *et al.*, pp. 845-847.



Chemical biology articles published in this journal also appear in the *Chemical Biology Virtual Journal*: [www.rsc.org/chembiol](http://www.rsc.org/chembiol)



#### Cover

See Richard G. Compton, Craig E. Banks, Trevor J. Davies and Gregory G. Wildgoose, pp. 829-841. This feature article addresses the reactivity of carbon surfaces, with particular emphasis on the importance of edge-plane sites for both electron transfer and surface modification.

## CHEMICAL TECHNOLOGY

T5

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## Chemical Technology

February 2005/Volume 2/Issue 2

[www.rsc.org/chemicaltechnology](http://www.rsc.org/chemicaltechnology)

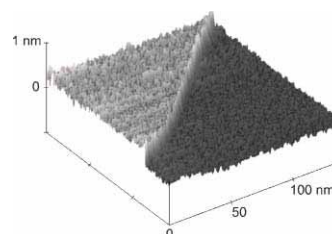
## FEATURE ARTICLE

829

### Electrocatalysis at graphite and carbon nanotube modified electrodes: edge-plane sites and tube ends are the reactive sites

Craig E. Banks, Trevor J. Davies, Gregory G. Wildgoose and Richard G. Compton\*

This feature article addresses the reactivity of carbon surfaces, with particular emphasis on the importance of edge-plane sites for both electron transfer and surface modification. Novel carbon nanotube and graphitic materials are contrasted and a critical overview of the recent literature is presented.



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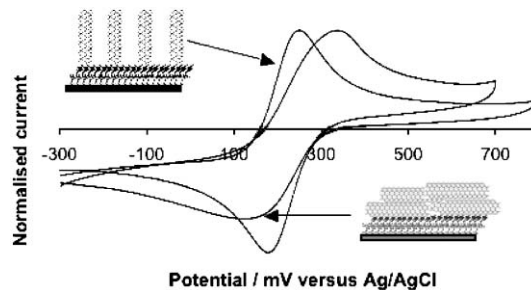
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**Demonstration of the importance of oxygenated species at the ends of carbon nanotubes for their favourable electrochemical properties**

Alison Chou, Till Böcking, Nagindar K. Singh and J. Justin Gooding\*

Definitive evidence is presented for the favourable electrochemical properties of carbon nanotube modified electrodes arising from the ends of SWNTs due to oxygenated carbon species in general, and carboxylic acid moieties in particular, produced during acid purification.

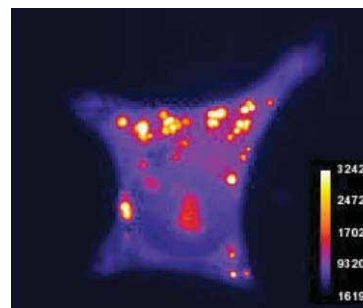


845

**Fluorescence studies of the intra-cellular distribution of zinc bis(thiosemicarbazone) complexes in human cancer cells**

Andrew R. Cowley, Jason Davis, Jonathan R. Dilworth,\* Paul S. Donnelly, Rachel Dobson, Adrian Nightingale, Josephine M. Peach, Ben Shore, David Kerr and Len Seymour

The intrinsic fluorescence of zinc complexes of bis(thiosemicarbazone) ligands is used to track for the first time their distribution in a range of living human cancer cells; this is shown to be dependent on both cell type and ligand substituents.

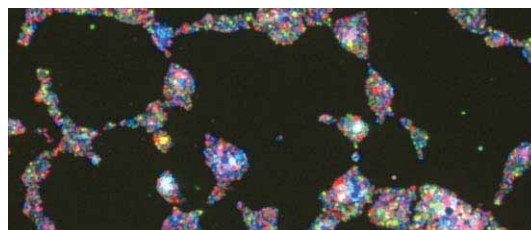


848

**Porous functionalised silica particles: a potential platform for biomolecular screening**

Angus P. R. Johnston, Bronwyn J. Battersby, Gwendolyn A. Lawrie and Matt Trau\*

Using a novel emulsion synthesis technique, porous, functionalized silica particles possessing controlled morphology have been developed. Fluorescent dyes can be covalently attached to the particles, which act like an optical barcode.

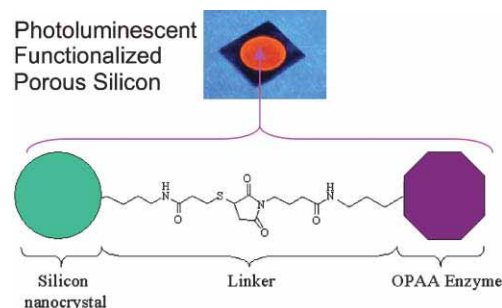


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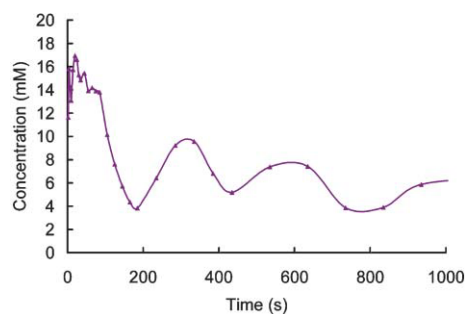
**Hydrolysis of acetylcholinesterase inhibitors – organophosphorus acid anhydrolase enzyme immobilization on photoluminescent porous silicon platforms**

Sonia E. Létant, Staci R. Kane, Bradley R. Hart, Masood Z. Hadi, Tu-Chen Cheng, Vipin K. Rastogi and John G. Reynolds\*

We report on the immobilization and consequent enzymatic behavior of an OPAA enzyme on luminescent porous silicon devices, and on the utilization of this new platform to hydrolyze *p*-nitrophenyl-soman.



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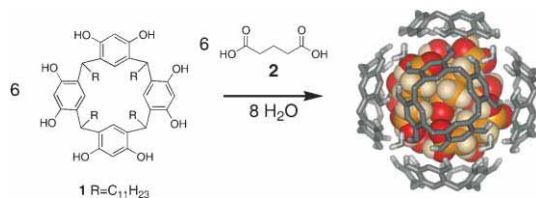


### Opening the 'black box': oscillations in organocuprate conjugate addition reactions

Michael D. Murphy, Craig A. Ogle\* and Steven H. Bertz\*

Complex oscillations are observed in the reactions of butyl Gilman reagents with 2-cyclohexenone, a typical substrate.

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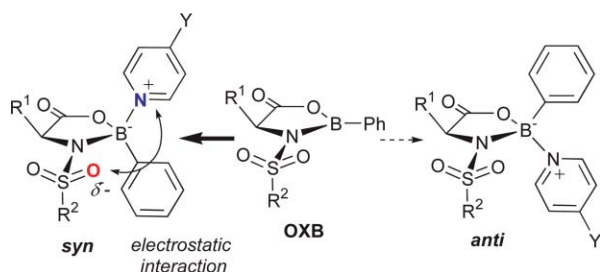


### Resorcinarene assemblies as synthetic receptors

Liam C. Palmer, Alexander Shivanyuk, Masamichi Yamanaka and Julius Rebek, Jr.

The host-guest complexes of resorcinarenes are re-examined in solution through modern spectroscopic methods. The assemblies are determined to be hexameric capsules and the guest exchange rates are measured by EXSY NMR spectroscopy. The neutral and cationic guest exchange rates correspond to barriers of approximately  $20 \text{ kcal mol}^{-1}$ .

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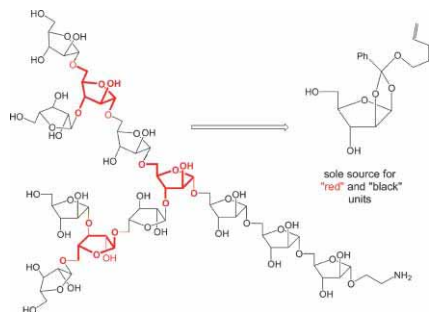


### High facial selectivity observed in amine coordination to chiral oxazaborolidinones

Toshiro Harada,\* Yuuki Yamamoto and Takahiro Kusukawa

High top-face selectivity can be rationalized by an electrostatic stabilizing interaction between the negatively charged sulfonyl oxygen atom and the positively charged pyridyl moiety.

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### The antituberculosis, antitumor, multibranched dodecafuranoarabinan of *Mycobacterium* species has been assembled from a single n-pentenyl furanoside source

Jun Lu and Bert Fraser-Reid\*

An n-pentenyl furanosyl-1,2-orthoester can function as a donor or be rearranged leading to an n-pentenyl furanoside acceptor which is glycosylated by its progenitor, regioselectively or doubly, thereby enabling rapid fabrication of a multibranched dodecasaccharide, known to possess a wide variety of biological interactions.

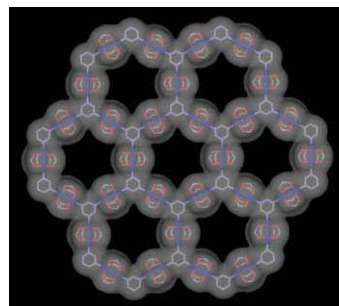


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**Rational synthesis of a two-dimensional honeycomb structure based on a paramagnetic paddlewheel diruthenium complex**

Shuhei Furukawa, Masaaki Ohba and Susumu Kitagawa\*

A novel 2-D coordination polymer with honeycomb (6,3) net topology is constructed from a paramagnetic paddlewheel diruthenium tetrabenzoate,  $[\text{Ru}^{\text{II}}_2(\text{O}_2\text{CPh})_4]$ , as a linear linker motif and triazine as a three-connected node.

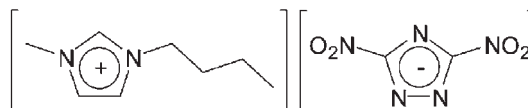


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**1-Butyl-3-methylimidazolium 3,5-dinitro-1,2,4-triazolate: a novel ionic liquid containing a rigid, planar energetic anion**

Alan R. Katritzky,\* Shailendra Singh, Kostyantyn Kirichenko, John D. Holbrey,\* Marcin Smiglak, W. Matthew Reichert and Robin D. Rogers\*

The novel crystalline salt, 1-butyl-3-methylimidazolium 3,5-dinitro-1,2,4-triazolate, has been synthesized and characterized. The salt melts at 35 °C and demonstrates how ionic liquids can be readily prepared with functionalized heterocyclic energetic anions.

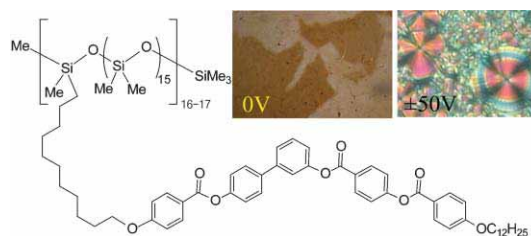


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**The first example of a liquid crystalline side-chain polymer with bent-core mesogenic units: ferroelectric switching and spontaneous achiral symmetry breaking in an achiral polymer**

Christina Keith, R. Amaranatha Reddy and Carsten Tschierske\*

The first bent-core side-chain polymeric liquid crystal, a dimethylsiloxane diluted polysiloxane, was synthesised and shows an optically isotropic ferroelectric switching polar smectic C phase consisting of a conglomerate of homogeneously chiral domains with opposite handedness.

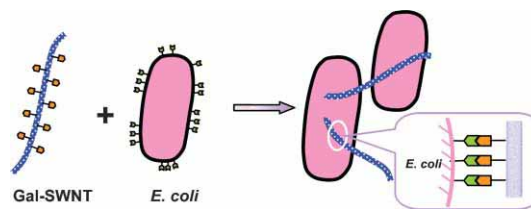


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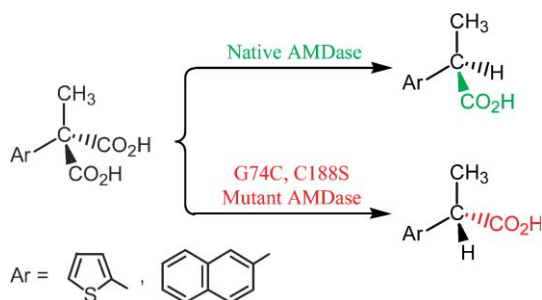
**Single-walled carbon nanotubes displaying multivalent ligands for capturing pathogens**

Lingrong Gu, Tara Elkin, Xiuping Jiang, Huaping Li, Yi Lin, Liangwei Qu, Tzuen-Rong J. Tzeng, Ronald Joseph and Ya-Ping Sun\*

A single-walled carbon nanotube was exploited for its semi-flexible pseudo-one-dimensional nanostructure as a unique scaffold to display multivalent carbohydrate ligands, with a specific demonstration showing that galactosylated carbon nanotubes were effective in the capturing of pathogenic *Escherichia coli* in solution.



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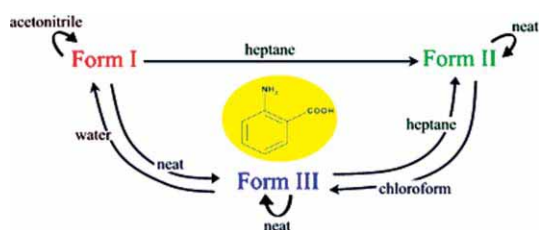


### Inversion of enantioselectivity of asymmetric biocatalytic decarboxylation by site-directed mutagenesis based on the reaction mechanism

Yoichiro Ijima, Kaori Matoishi, Yosuke Terao, Nobuhide Doi, Hiroshi Yanagawa and Hiromichi Ohta\*

Arylmalonate decarboxylase is an unique enzyme which catalyses the enantioselective decarboxylation of  $\alpha$ -aryl- $\alpha$ -methylmalonate to give optically pure arylpropionate. The enantioselectivity of this enzyme was dramatically changed by the introduction of two mutations (G74C/C188S) based on the reaction mechanism.

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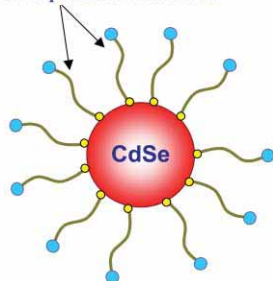
### Selective polymorph transformation *via* solvent-drop grinding

Andrew V. Trask, Ning Shan,\* W. D. Samuel Motherwell, William Jones,\* Shaohua Feng, Reginald B. H. Tan and Keith J. Carpenter

A method of inducing specific polymorph transformations is exemplified with two single-component systems, whereby a given crystal form undergoes conversion when subjected to solid state grinding in the presence of a minor quantity of a certain solvent.

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### 2-Mercaptoethane sulfonate

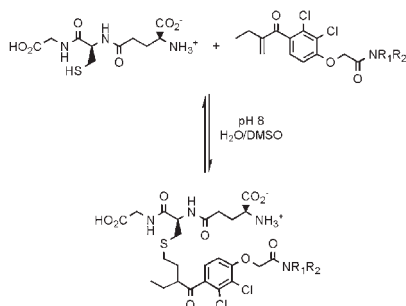


### Photoactivated luminescent CdSe quantum dots as sensitive cyanide probes in aqueous solutions

Wei Jun Jin, María T. Fernández-Argüelles, José M. Costa-Fernández, Rosario Pereiro and Alfredo Sanz-Medel\*

The first application of quantum dots as highly sensitive and selective luminescent probes for anions determination in aqueous solutions is described. The synthesized nanoparticles proved to be one of the most sensitive detection systems available for monitoring the highly toxic cyanide anion.

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### Reversible Michael addition of thiols as a new tool for dynamic combinatorial chemistry

Baolu Shi and Michael F. Greaney\*

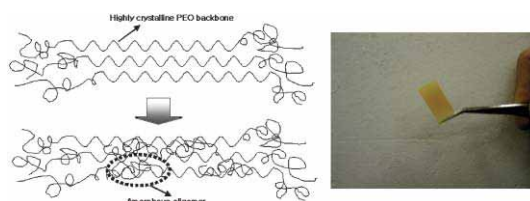
Glutathione reacts with six ethacrynic acid derivatives to form a twelve membered dynamic combinatorial library of Michael adducts and enone starting materials. The library reaches equilibrium in three hours, is freely reversible and can be switched on or off by a simple pH change.

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### Dye-sensitized solar cells based on composite solid polymer electrolytes

Moon-Sung Kang, Jong Hak Kim, Young Jin Kim, Jongok Won, Nam-Gyu Park and Yong Soo Kang\*

Dye-sensitized solar cells employing composite polymer electrolytes show a remarkable energy conversion efficiency of  $\sim 3.84\%$  at  $100 \text{ mW cm}^{-2}$ . The increased efficiency is primarily due to the addition of small-sized oligomeric poly(propylene glycol) into high molecular weight PEO with electrolytes of KI and  $\text{I}_2$  for both enhancing the ionic conductivity and more importantly enlarging the interfacial contact area between dyes and polymer electrolytes.

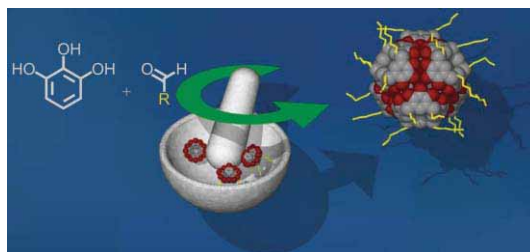


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### Solvent-free, direct synthesis of supramolecular nano-capsules

Jochen Antesberger, Gareth W. V. Cave, Matthew C. Ferrarelli, Michael W. Heaven, Colin L. Raston and Jerry L. Atwood\*

The single step direct synthesis of pyrogallol[4]arene *via* a solvent free protocol yields the pure product as a self-assembled nano-capsule, comprised of six macrocyclic building blocks.

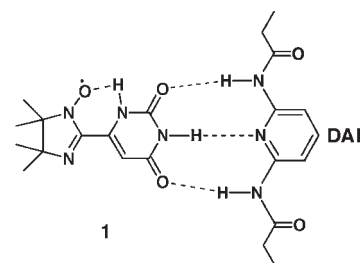


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### Molecular recognition in a uradinyl-functionalized stable radical

Patrick Taylor, Paul M. Lahti,\* Joseph B. Carroll and Vincent M. Rotello

2-(6-Uradinyl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazole-1-oxyl (**1**) binds to hydrogen-bonding complement 2,6-di(propylamido)pyridine (DAP) in chloroform with association constant  $K_a = 220 \text{ M}^{-1}$  at  $33 \text{ }^\circ\text{C}$ ; ESI-MS shows not only **1**:DAP complementary dyad formation, but also **1**:(DAP)<sub>2</sub> formation at higher relative concentrations of DAP.

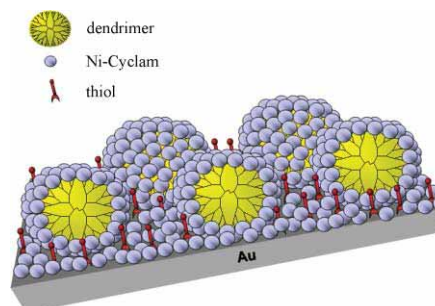


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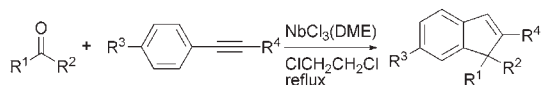
### Ni(II)1,4,8,11-tetraazacyclotetradecane electrocatalytic films prepared on top of surface anchored PAMAM dendrimer layers. A new type of electrocatalytic material for the electrochemical oxidation of methanol

Miguel A. González-Fuentes, J. Manríquez, S. Gutiérrez-Granados, A. Alatorre-Ordaz and Luis A. Godínez\*

The presence of interfacially anchored PAMAM dendrimers on a Au modified electrode, enhances the electrocatalytic activity of Ni-cyclam towards the electrochemical electrooxidation of methanol.



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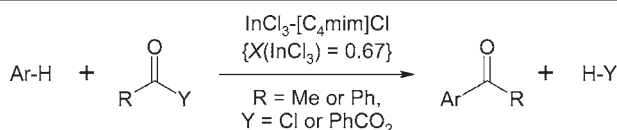


**Low-valent Nb(III)-mediated synthesis of 1,1,2-trisubstituted-1*H*-indenes from aliphatic ketones and aryl-substituted alkynes**

Yasushi Obora, Masahiro Kimura, Makoto Tokunaga and Yasushi Tsuji\*

A variety of 1,1,2-trisubstituted-1*H*-indenes are synthesized by the reaction of aliphatic ketones, aryl-substituted alkynes and NbCl<sub>3</sub>(DME) in 1,2-dichloroethane under reflux conditions.

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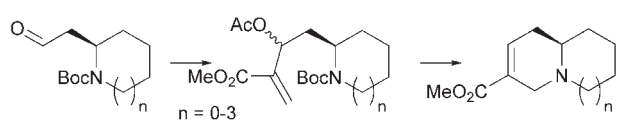


**Chloroindate(III) ionic liquids: recyclable media for Friedel–Crafts acylation reactions**

Martyn J. Earle, Ullastiina Hakala, Christopher Hardacre, Johanna Karkkainen, Barry J. McAuley, David W. Rooney, Kenneth R. Seddon, Jillian M. Thompson and Kristina Wähälä

Chloroindate(III) ionic liquids are versatile reaction media for Friedel–Crafts acylation reactions; the system is catalytic and totally recyclable.

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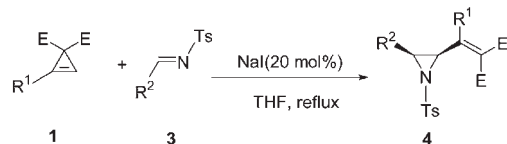


**A general method for making bicyclic compounds with nitrogen at a bridgehead—application to the halichlorine spiro subunit**

Derrick L. J. Clive,\* Maolin Yu and Zhiyong Li

*N*-Protected  $\beta$ -amino aldehydes having the nitrogen in a ring are easily converted into Morita–Baylis–Hillman adducts; *O*-acetylation and *N*-deprotection result in spontaneous cyclization to bicyclic structures having nitrogen at a bridgehead.

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**NaI-catalyzed highly regioselective ring-opening [1 + 2] cycloaddition reaction of cyclopropenes with imines: highly stereoselective synthesis of *cis*-vinylic aziridines**

Shengming Ma,\* Junliang Zhang, Lianghua Lu, Xin Jin, Yangjun Cai and Hairong Hou

*cis*-Vinylic aziridines were prepared highly stereoselectively via a NaI-catalyzed regioselective ring-opening [1 + 2] cycloaddition reaction of cyclopropenes with imines.



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**Regioselective silylphosphination of methyl vinyl ketone with complexes containing cyclic and linear iron–silicon–phosphorus reacting sites**

Masaaki Okazaki,\* Kyeong A Jung and Hiromi Tobita\*

Methyl vinyl ketone was incorporated into iron–silicon–phosphorus reacting sites in three different fashions. Structures of the products were fully characterized by X-ray diffraction studies.

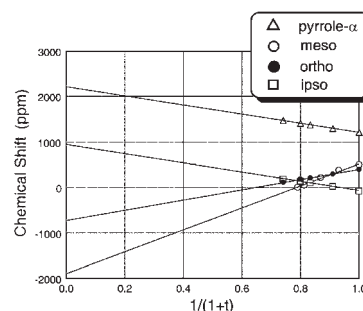


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**Determination of the  $^{13}\text{C}$  NMR chemical shifts in an  $a_{2u}$  type iron(III) porphyrin cation radical**

Akito Hoshino and Mikio Nakamura\*

The chemical shifts of all the carbons in an  $a_{2u}$  type iron(III) porphyrin radical cation have been determined by the titration method.

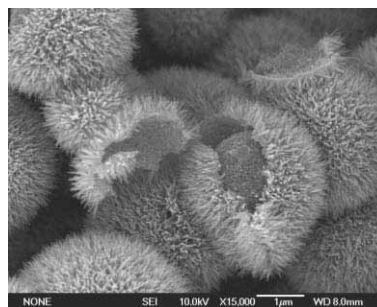


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**One-step solution-based catalytic route to fabricate novel  $\alpha$ - $\text{MnO}_2$  hierarchical structures on a large scale**

Zhengquan Li, Yue Ding, Yujie Xiong, Qing Yang and Yi Xie\*

A novel  $\alpha$ - $\text{MnO}_2$  hierarchical structure, a core-shell structure with spherically aligned nanorods, was conveniently synthesized by a room-temperature solution-based catalytic route.

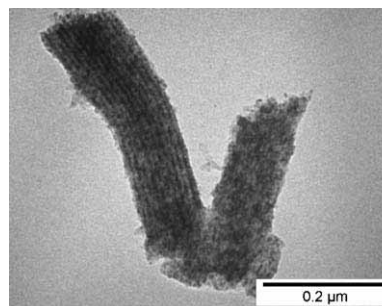


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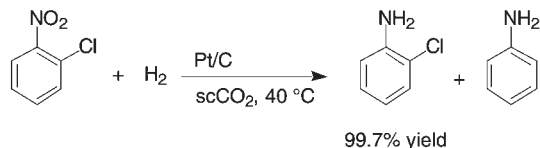
**The use of tin-decorated mesoporous carbon as an anode material for rechargeable lithium batteries**

I. Grigoriants, L. Sominski, Hongliang Li, Ilan Ifargan, D. Aurbach and A. Gedanken\*

We report on the preparation of mesoporous carbon particles, decorated with metallic tin nanoparticles. These composites were proved to be a promising active mass for anodes in rechargeable Li batteries.



924

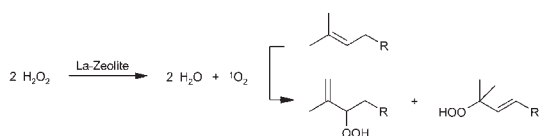


### The role of carbon dioxide in chemoselective hydrogenation of halonitroaromatics over supported noble metal catalysts in supercritical carbon dioxide

Shinichiro Ichikawa, Mizuki Tada, Yasuhiro Iwasawa and Takao Ikariya\*

Chemoselective hydrogenation of halogenated nitrobenzenes over Pt/C catalysts proceeds effectively in supercritical carbon dioxide (scCO<sub>2</sub>) to produce halogenated anilines with an excellent selectivity in which the rate of the hydrogenation is markedly enhanced and the dehalogenation reaction is significantly suppressed.

927

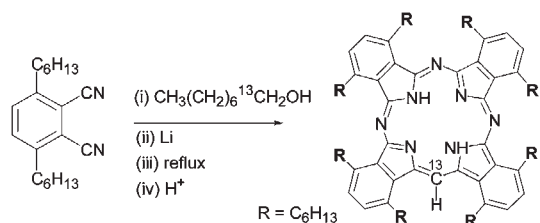


### Lanthanum-exchanged zeolites as active and selective catalysts for the generation of singlet oxygen from hydrogen peroxide

Joos Wahlen, Dirk De Vos, Sigrid De Hertogh, Véronique Nardello, Jean-Marie Aubry, Paul Alsters and Pierre Jacobs\*

Lanthanum(III)-exchanged zeolites Beta and USY are active and selective catalysts for the generation of singlet oxygen from H<sub>2</sub>O<sub>2</sub> showing superior activity and oxidant efficiency compared to unsupported La-catalysts, *e.g.* La(OH)<sub>3</sub>.

930

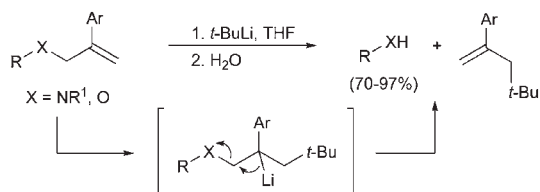


### A remarkable side-product from the synthesis of an octaalkylphthalocyanine: formation of a tetrabenzotriazaporphyrin

Andrew N. Cammidge,\* Michael J. Cook,\* David L. Hughes, Fabien Nekelson and Muhibur Rahman

Subtle changes to the experimental protocol of the lithium alkyloxide induced conversion of 3,6-dihexylphthalonitrile into 1,4,8,11,15,18,22,25-octaalkylphthalocyanine can suppress or enhance the unexpected formation of the correspondingly substituted tetrabenzotriazaporphyrin side-product.

933



### 2-Aryllallyl as a new protecting group for amines, amides and alcohols

José Barluenga,\* Francisco J. Fañanás, Roberto Sanz, César Marcos and José M. Ignacio

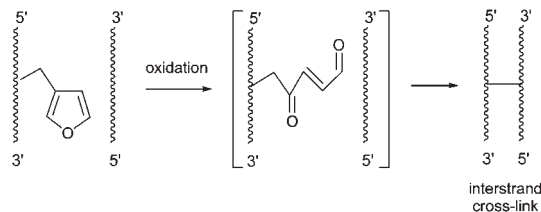
A new protecting allyl-type group, 2-aryllallyl, for amines, amides and alcohols is reported. Selective deprotection with *t*-BuLi probably involves an intermolecular carbolithiation.

936

**Fine-tuning furan toxicity: fast and quantitative DNA interchain cross-link formation upon selective oxidation of a furan containing oligonucleotide**

Sami Halila, Trinidad Velasco, Pierre De Clercq and Annemieke Madder\*

*In situ* oxidation of a duplex formed between a furan modified oligonucleotide and its complement transforms the furan moiety into a reactive enal that triggers fast and clean interchain cross-link formation.

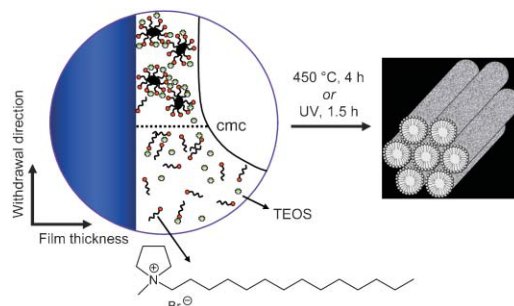


939

***N*-Alkyl-*N*-methylpyrrolidinium salts as templates for hexagonally meso-ordered silicate thin films**

Andrew M. Dattelbaum, Sheila N. Baker and Gary A. Baker\*

A series of *N*-alkyl-*N*-methylpyrrolidinium bromide salts (C14, C16, or C18) have been explored as templates for the synthesis of ordered, mesoporous silica films. "Soft" annealing based on deep-UV calcination is also reported.

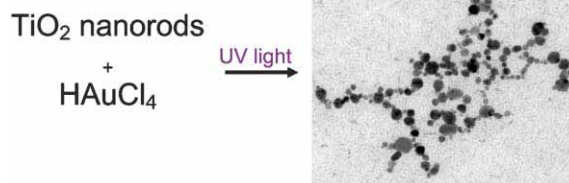


942

**Low-dimensional chainlike assemblies of TiO<sub>2</sub> nanorod-stabilized Au nanoparticles**

P. D. Cozzoli,\* E. Fanizza, M. L. Curri, D. Laub and A. Agostiano

Chainlike Au nanoparticle assemblies are formed upon UV-irradiating H<sub>AuCl</sub><sub>4</sub> in the presence of TiO<sub>2</sub> nanorods as stabilizers in apolar media.

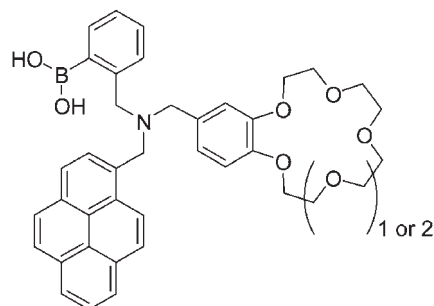


945

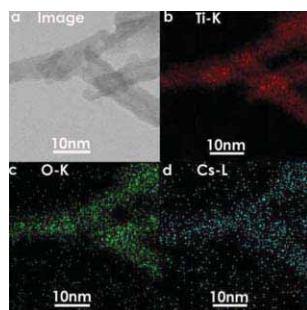
**A ditopic fluorescent sensor for potassium fluoride**

Suvi J. M. Koskela, Thomas M. Fyles and Tony D. James\*

Ditopic fluorescent sensors for potassium fluoride have been prepared using a boronic acid receptor (fluoride) and crown ether receptors (potassium)



948

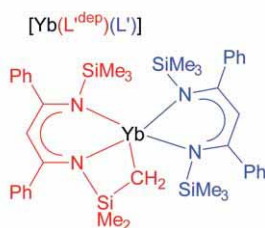
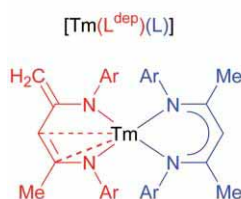
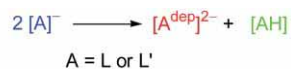


### Alkali metal cation intercalation properties of titanate nanotubes

Renzhi Ma,\* Takayoshi Sasaki and Yoshio Bando

Hydrothermally synthesized titanate nanotubes show high alkali metal cation intercalation reactivity evidenced by high resolution microscopic examination, elemental mapping and profiling techniques as well as wet chemical analysis.

951



### New reactions of $\beta$ -diketiminatolanthanoid complexes: sterically induced self-deprotonation of $\beta$ -diketiminato ligands

Peter B. Hitchcock, Michael F. Lappert\* and Andrey V. Protchenko

Attempted synthesis of sterically demanding bis- or tris- $\beta$ -diketiminato complexes of lanthanoids resulted in ligand deprotonation and the formation of complexes containing both a “normal” and a deprotonated ligand; one of these on protonation gave the first cationic  $\beta$ -diketiminato–Ln complex.




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*Jay S. Siegel, Switzerland*

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