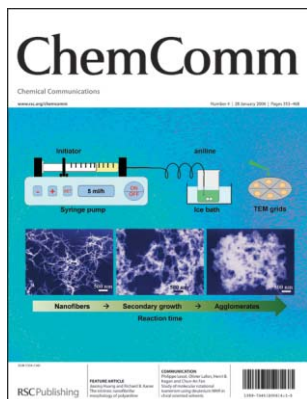


## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (4) 353-468 (2006)



### Cover

See Jiaying Huang and Richard B. Kaner page 367. A schematic illustration of the experimental set-up used to explore the morphological change of polyaniline during chemical polymerization and TEM images of the morphological evolution of polyaniline from nanofibers to agglomerates as the reaction proceeds. Image reproduced by permission of Jiaying Huang and Richard B. Kaner from *Chem. Commun.*, 2006, 367.

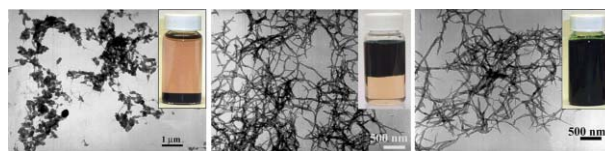
## FEATURE ARTICLE

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### The intrinsic nanofibrillar morphology of polyaniline

Jiaying Huang and Richard B. Kaner\*

Nanofibrillar morphology is found to be intrinsic to polyaniline chemically synthesized in water. In conventional synthesis (left), the product that readily precipitates out of the solution is a mixture of irregularly-shaped, micron-sized particulates and nanofibers. In interfacial polymerization (middle) and rapidly-mixed reactions (right), the products are pure nanofibers that disperse very well in water.



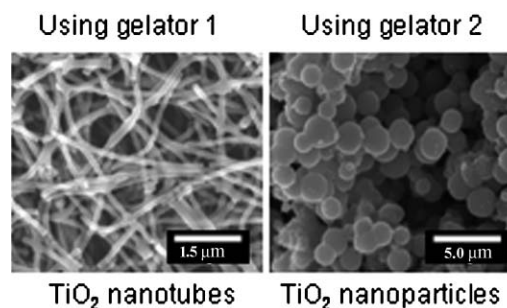
## COMMUNICATIONS

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### Fabrication of TiO<sub>2</sub> using L-lysine-based organogelators as organic templates: control of the nanostructures

Masahiro Suzuki,\* Yasushi Nakajima, Teruaki Sato, Hirofusa Shirai and Kenji Hanabusa

The control of the nanostructures of TiO<sub>2</sub> prepared through sol-gel polymerization using uncharged and negatively charged gelators as organic templates is described.



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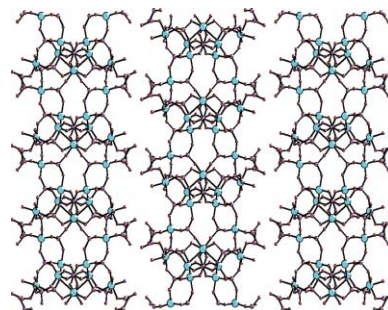
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### The ionothermal synthesis of SIZ-6—a layered aluminophosphate

Emily R. Parnham, Paul S. Wheatley and Russell E. Morris\*

Ionothermal synthesis—the use of an ionic liquid as both solvent and structure-directing agent in materials synthesis—has been used to prepare SIZ-6, a layered aluminophosphate. 1-Methyl-3-ethylimidazolium bromide is the solvent and the cation is incorporated intact into the structure.

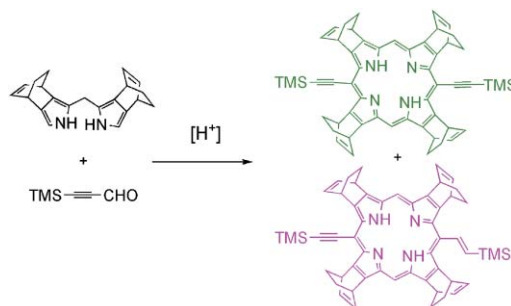


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### Novel one-pot synthesis of 5-alkenyl-15-alkynylporphyrins and their derivatisation to a butadiyne-linked benzoporphyrin dimer

Hiroko Yamada,\* Kayo Kushibe, Tetsuo Okujima, Hidemitsu Uno and Noboru Ono\*

5-Alkenyl-15-alkynylporphyrins have been obtained unexpectedly during a synthesis of 5,15-dialkynylporphyrin by [2 + 2] acid-catalyzed condensation of dipyrromethane and TMS propynal.

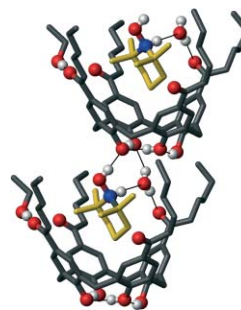


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### Supramolecular stabilization of hydroxylamine TEMPOH by complexation with an amphiphilic calixarene

Gennady S. Ananchenko,\* Michaela Pojarova, Konstantin A. Udachin, Donald M. Leek, Antony W. Coleman and John A. Ripmeester

Nitroxyl radical TEMPO abstracts a hydrogen atom from OH group of para-hexanoyl calix[4]arene, and the hydroxylamine TEMPOH formed yields a stable inclusion complex with another molecule of the calixarene.

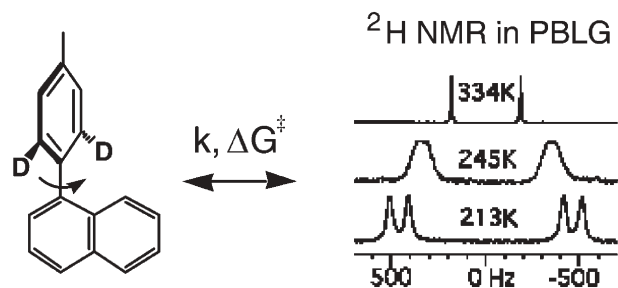


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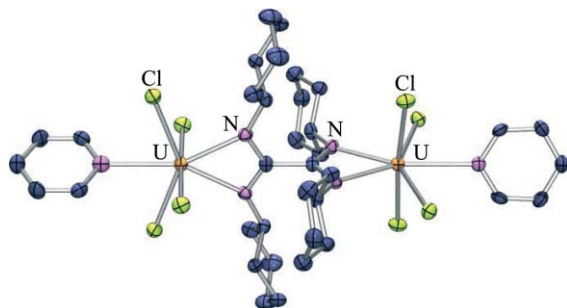
### Study of molecular rotational isomerism using deuterium NMR in chiral oriented solvents

Philippe Lesot,\* Olivier Lafon, Henri B. Kagan and Chun-An Fan

Deuterium NMR in polypeptide chiral liquid crystals is used for the first time to investigate the internal rotational isomerism phenomenon, illustrated in the case of 1-(2',6'-dideutero-4'-methylphenyl)naphthalene.



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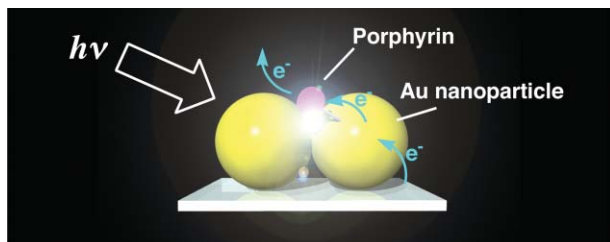


### Reductive coupling of carbodiimides with metallic lithium: a new route to metal oxalamidino complexes

Claude Villiers,\* Pierre Thuéry and Michel Ephritikhine\*

The syntheses of the first oxalamidinate complexes of an alkali metal and of a 5f-element show that the carbodiimides can be reductively coupled with lithium metal and with a low-valent uranium(III) compound.

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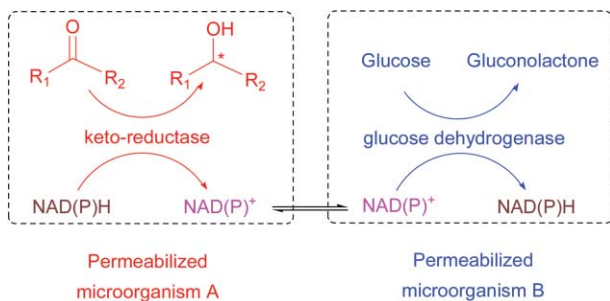


### Photocurrent enhancement in a porphyrin-gold nanoparticle nanostructure assisted by localized plasmon excitation

Tsuyoshi Akiyama,\* Masato Nakada, Nao Terasaki and Sunao Yamada\*

The porphyrin-gold nanoparticle assembly exhibits distinct photocurrent enhancement assisted by localized plasmon.

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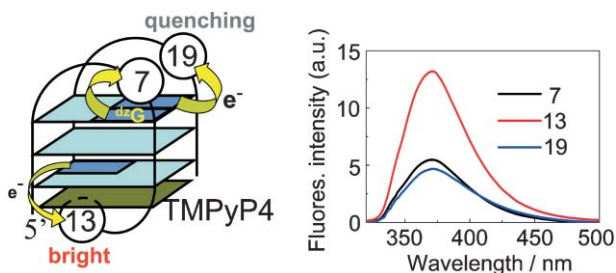


### Coupling of permeabilized microorganisms for efficient enantioselective reduction of ketone with cofactor recycling

Jie Zhang, Bernard Witholt and Zhi Li\*

A novel, simple and efficient cofactor recycling method for enantioselective bioreduction has been developed *via* coupled permeabilized cells that are easily prepared and repeatedly used.

401



### Detection of the G-quadruplex-TMPyP4 complex by 2-aminopurine modified human telomeric DNA

Takumi Kimura, Kiyohiko Kawai, Mamoru Fujitsuka and Tetsuro Majima\*

2-Aminopurine (Ap) modified human telomere sequences were used to monitor the specific complex formation of the G-quadruplex and 5,10,15,20-tetrakis(*N*-methyl-4-pyridyl)-porphyrin (TMPyP4).

403

**Syntheses and structures of new diaryl lead(II) compounds**  
**PbR<sub>2</sub> (1, R = 2,4,6-triphenylphenyl; 2, R = 2,6-bis(1'-naphthyl)phenyl)**

Xiao-Juan Yang, Yuzhong Wang, Pingrong Wei,  
 Brandon Quillian and Gregory H. Robinson\*

Reaction of RLi with lead(II) bromide affords the diaryl lead(II) compounds PbR<sub>2</sub> (R<sup>1</sup> = 2,4,6-triphenylphenyl, **1**; R<sup>2</sup> = 2,6-bis(1'-naphthyl)phenyl, **2**), which have carbene-like structures with bent two-coordinate Pb(II) centers.

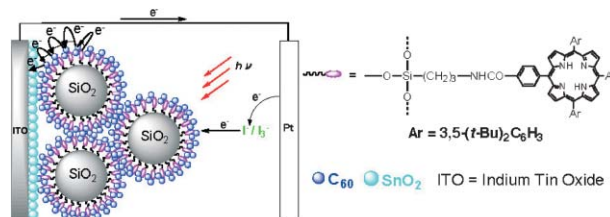


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**Efficient photocurrent generation by SnO<sub>2</sub> electrode modified electrophoretically with composite clusters of porphyrin-modified silica microparticle and fullerene**

Hiroshi Imahori,\* Keigo Mitamura, Tomokazu Umeyama,  
 Kohei Hosomizu, Yoshihiro Matano, Kaname Yoshida  
 and Seiji Isoda

A silica microparticle has been successfully employed as a nanoscaffold to self-organize porphyrin and C<sub>60</sub> molecules on a nanostructured SnO<sub>2</sub> electrode which exhibits efficient photocurrent generation.

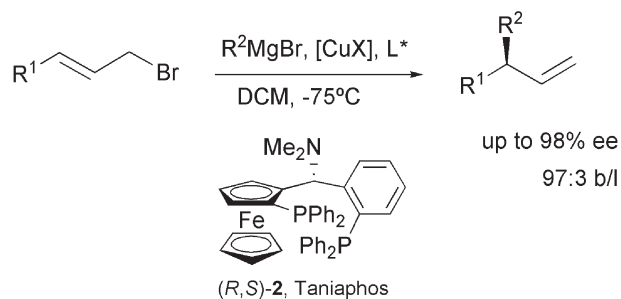


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**Highly enantioselective Cu-catalysed allylic substitutions with Grignard reagents**

Fernando López, Anthoni W. van Zijl,  
 Adriaan J. Minnaard and Ben L. Feringa\*

With use of the Taniaphos ligand, enantioselectivities up to 98% and excellent regioselectivities can be achieved in allylic alkylation reactions with a range of substrates and Grignard reagents.

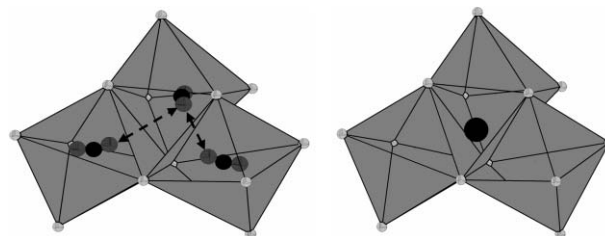


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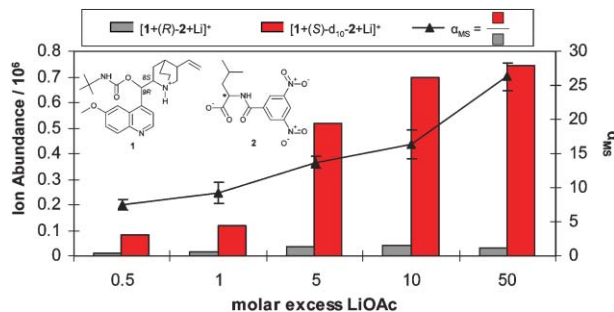
**The structure of lithium garnets: cation disorder and clustering in a new family of fast Li<sup>+</sup> conductors**

Edmund J. Cussen

The structure of the fast lithium-ion conducting garnets Li<sub>5</sub>La<sub>3</sub>M<sub>2</sub>O<sub>12</sub> (M = Ta, Nb) reveals Li<sup>+</sup> on both tetrahedral and octahedral sites and that Li<sup>+</sup> mobility occurs *via* a clustering mechanism.



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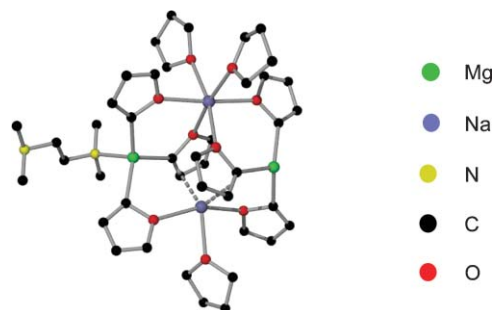


### Chiral recognition mass spectrometry: remarkable effects observed from the relative ion abundances of ternary diastereomeric complexes using electrospray ionization

Kevin A. Schug,\* Norbert M. Maier and Wolfgang Lindner\*

Analysis of cinchona-alkaloid selector–chiral acid selectand systems by competitive binding ESI-MS in the presence of alkali metal ion salts significantly increases the measured enantioselectivity ( $\alpha_{MS}$ ) based on ternary diastereomeric complex ion abundances.

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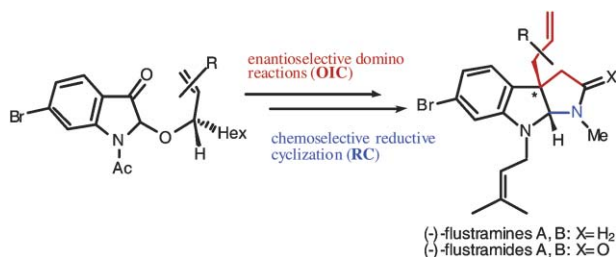


### Building an extended inverse crown motif *via* alkali-metal-mediated $\alpha$ -magnesiumation of furan

David V. Graham, Eva Hevia, Alan R. Kennedy, Robert E. Mulvey,\* Charles T. O'Hara and Christine Talmard

Selectively  $\alpha$ -metallating furan *via* sodium-mediated magnesiumation leads ultimately to the construction of a new inverse crown structure, the 'guest' furyl anions of which are triply stabilised through a combination of Mg–C  $\sigma$ , Na–O  $\sigma$  and Na $\cdots$ C–C  $\pi$  interactions.

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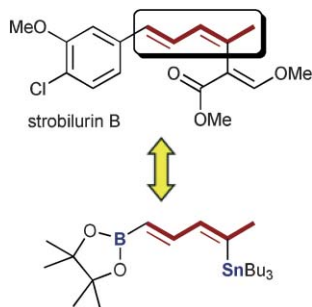


### Enantioselective total synthesis of (–)-flustramines A, B and (–)-flustramides A, B *via* domino olefination/isomerization/Claisen rearrangement sequence

Tomomi Kawasaki,\* Masashi Shinada, Daigo Kamimura, Mayu Ohzono and Atsuyo Ogawa

Enantioselective total synthesis of four title marine alkaloids has been accomplished *via* the domino olefination/isomerization/Claisen rearrangement (OIC) and the chemoselective reduction–cyclization (RC) as key steps.

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### Total synthesis of strobilurin B using a hetero-*bis*-metallated pentadiene linchpin

Robert S. Coleman\* and Xiaoling Lu

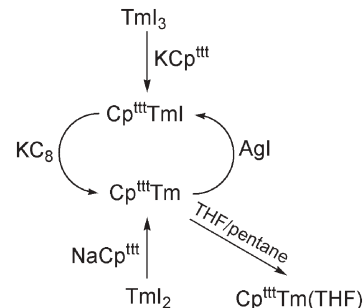
Hetero-*bis*-metallated 1,3-pentadiene is employed as the linchpin in fragment coupling in a six-step total synthesis of the antifungal agent strobilurin B. Sequential Suzuki–Miyaura and Stille coupling interpolate this boron/tin diene between the aromatic ring and  $\beta$ -methoxyacrylate.

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### Synthesis of a new stable, neutral organothulium(II) complex by reduction of a thulium(III) precursor

Florian Jaroschik, François Nief\* and Louis Ricard

The stable  $\text{Tm}^{\text{II}}$  complex  $(\text{Cp}^{\text{tnt}})_2\text{Tm}$  [ $\text{Cp}^{\text{tnt}} = 1,2,4\text{-tris}(t\text{-Bu})\text{C}_5\text{H}_2$ ] can be obtained either by reaction of  $\text{TmI}_2(\text{THF})_3$  with  $\text{NaCp}^{\text{tnt}}$  or by reduction of  $(\text{Cp}^{\text{tnt}})_2\text{TmI}$  with  $\text{KC}_8$  in toluene, a route which may prove useful for other “non-classical” low-valent organolanthanides.

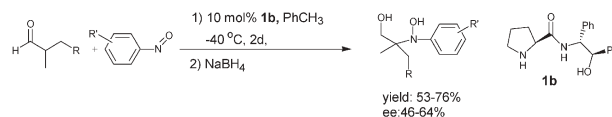


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### L-Prolinamide-catalyzed direct nitroso aldol reactions of $\alpha$ -branched aldehydes: a distinct regioselectivity from that with L-proline

Hai-Ming Guo, Li Cheng, Lin-Feng Cun, Liu-Zhu Gong,\* Ai-Qiao Mi and Yao-Zhong Jiang\*

The first direct enantioselective *N*-nitroso aldol reactions of aldehyde with nitrosobenzene catalyzed by an L-prolinamide derivative proceed smoothly furnishing the  $\alpha$ -hydroxyamino carbonyl compounds, the otherwise disfavored products, in good yields with up to 64% ee.

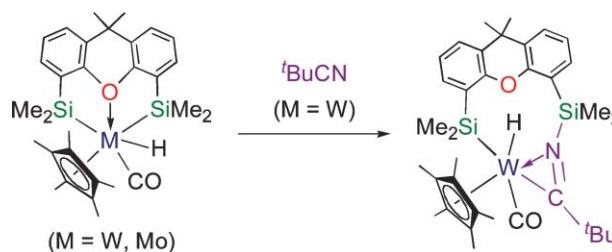


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### Group 6 metal complexes with a hemilabile tridentate xantsil ligand and facile insertion of $t\text{BuCN}$ into a W–Si bond [xantsil = (9,9-dimethylxanthene-4,5-diyl)-bis(dimethylsilyl)]

Rockshana Begum, Takashi Komuro and Hiromi Tobita\*

Novel group 6 metal complexes having a xanthene-based bis(silyl) ligand (xantsil) were synthesised and characterised, and the hemilability of the xantsil ligand in the tungsten complex was proved by nitrile-insertion into a W–Si bond giving an  $\eta^2$ -iminoacyl complex.

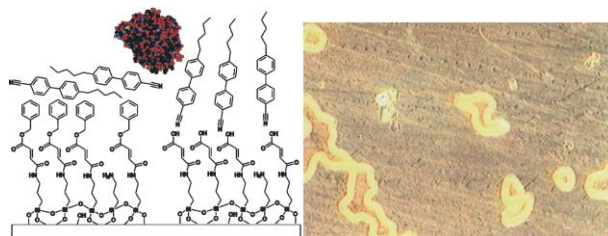


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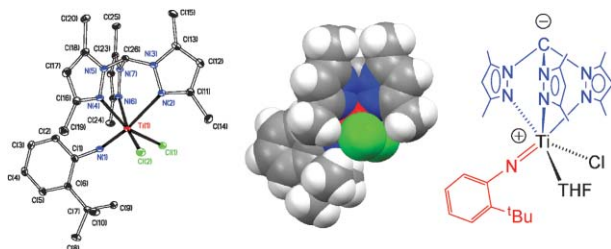
### LCD-based detection of enzymatic action

Johan Hoogboom,\* Kelly Velonia, Theo Rasing, Alan E. Rowan and Roeland J. M. Nolte

By incorporating an ester-containing substrate in a self-assembled alignment layer for liquid crystal cells, the presence of lipase B from *Candida antarctica* is detected by its enzymatic action on the alignment layer, without the need for fluorescent labelling or enzyme assays.



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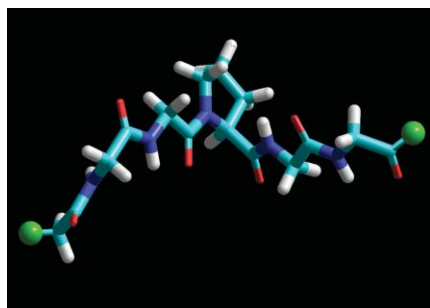


**A remarkable inversion of structure–activity dependence on imido N-substituents with varying co-ligand topology and the synthesis of a new borate-free zwitterionic polymerisation catalyst**

Helen R. Bigmore, Stuart R. Dubberley, Mirko Kranenburg, Sally C. Lawrence, Andrew J. Sealey, Jonathan D. Selby, Martin A. Zuideveld, Andrew R. Cowley and Philip Mountford\*

Highly active tris(pyrazolyl)methane-supported ethylene polymerisation catalysts are reported.

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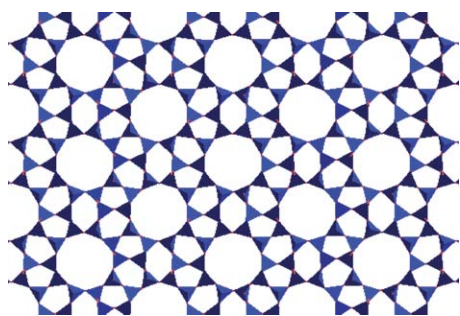


**Glycine position permutations and peptide length alterations change the aggregation state and efficacy of ion-conducting, pore-forming amphiphiles**

Riccardo Ferdani, Robert Pajewski, Jolanta Pajewska, Paul H. Schlesinger and George W. Gokel\*

Small variations in the peptide chain dramatically alter chloride ion conduction properties.

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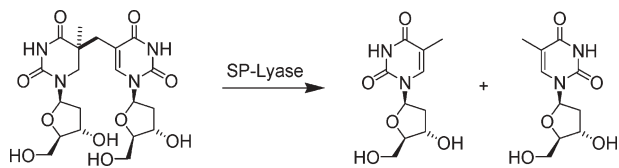


**Incidence and properties of nanoscale defects in silicalite**

Naseem A. Ramsahye and Ben Slater\*

Crystal intergrowths are predicted to form more readily than other extended defects in a model zeolite membrane and cause a reduction in molecular flux.

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**The spore photoproduct lyase repairs the 5*S*- and not the 5*R*-configured spore photoproduct DNA lesion**

Marcus G. Friedel, Olivier Berteau, J. Carsten Pieck, Mohamed Atta, Sandrine Ollagnier-de-Choudens, Marc Fontecave\* and Thomas Carell\*

The spore photoproduct lyase was challenged with synthetically pure substrates and recognized a synthetic 5*S*-configured spore lesion without the central phosphodiester bond. The 5*R*-configured lesion is not a substrate.

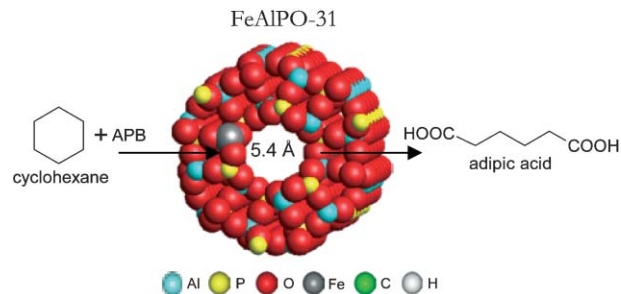


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### Highly efficient one-step conversion of cyclohexane to adipic acid using single-site heterogeneous catalysts

Robert Raja,\* John Meurig Thomas, Mingcan Xu, Kenneth D. M. Harris, Michael Greenhill-Hooper and Kieran Quill

Acetylperoxyborate (APB), when dissolved in aqueous solution, is shown to be an effective oxidant for the catalytic oxidation of cyclohexane to adipic acid and for the epoxidation of olefins in high yields and high selectivities in the presence of single-site heterogeneous catalysts.

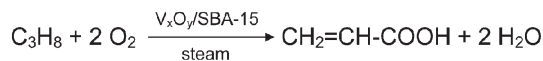


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### Importance of nanostructured vanadia for selective oxidation of propane to acrylic acid

Christian Hess,\* Ming Hoong Looi, Sharifa Bee Abd Hamid and Robert Schlögl

Highly dispersed nanostructured vanadia supported on mesoporous silica SBA-15, prepared by controlled grafting/ion-exchange, exhibits high selectivities in propane partial oxidation to acrylic acid demonstrating its unique potential for mixed metal oxide catalyst development.

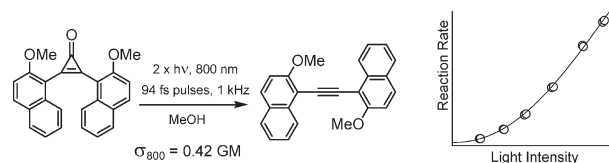


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### Two-photon induced photodecarbonylation reaction of cyclopropenones

Nurtay K. Urdabayev, Andrei Poloukhine and Vladimir V. Popik\*

Irradiation of cyclopropenones with 800 nm pulses of ultrafast laser results in a photodecarbonylation reaction *via* nonresonant two-photon absorption of light.

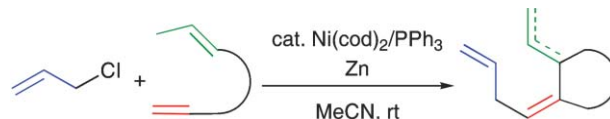


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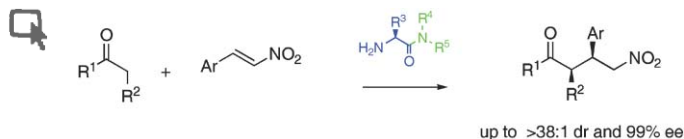
### Nickel-catalyzed coupling of allyl chlorides and enynes

Shin-ichi Ikeda,\* Kaori Suzuki and Kazunori Odashima

The Ni-catalyzed coupling of allyl chlorides and alkene-tethered alkynes has been developed; the cyclization of enynes was triggered by addition of  $\pi$ -allylnickel species to the alkyne part, followed by the incorporation of the alkene part.



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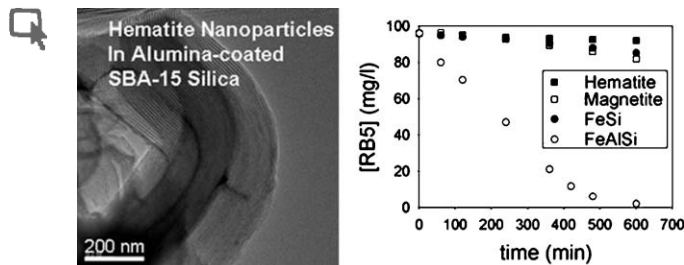


### Simple highly modular acyclic amine-catalyzed direct enantioselective addition of ketones to nitro-olefins

Yongmei Xu and Armando Córdova\*

We have demonstrated the unprecedented use of simple highly modular primary amino acid amides as catalysts for the direct asymmetric addition of ketones to nitro-olefins. The non-toxic primary amino acid derivatives can catalyze the reaction with high regio-, distereo- and enantioselectivity to furnish the corresponding  $\gamma$ -nitro-ketones in high yield with up to >38 : 1 dr and 99% ee.

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### Highly active heterogeneous Fenton catalyst using iron oxide nanoparticles immobilized in alumina coated mesoporous silica

Hacgyu Lim, Jinwoo Lee, Sunmi Jin, Jaeyun Kim, Jeyong Yoon\* and Taeghwan Hyeon\*

A highly active heterogeneous Fenton catalyst was fabricated by impregnating iron oxide nanoparticles in alumina coated mesoporous SBA-15 silica.

## ADDITION AND CORRECTION

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Nicolai Dieltiens, Diederica D. Claeys, Bart Allaert, Francis Verpoort Christian V. Stevens

### Synthesis of 1,3-dioxo-hexahydropyrido[1,2-c][1,3]-diazepine carboxylates, a new bicyclic skeleton formed by ring expansion–RCM methodology

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