

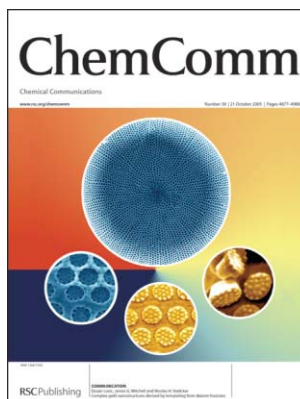
## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (39) 4877-4988 (2005)



### Cover

See David M. Hodgson and Deepshikha Angrish, page 4902. Unsaturated diazoesters undergo Grubbs(II)-catalysed dimerisation at the diazo functionality then ring-closing alkene metathesis, giving dienyl dilactones in a one-flask process. Image reproduced by permission of Dr. Karl Harrison (University of Oxford) from *Chem. Commun.*, 2005, 4902.



### Inside cover

See Dusan Losic, James G. Mitchell and Nicolas H. Voelcker, page 4905. Diatom frustules can be used as templates for the fabrication of complex gold nanostructures. Image reproduced by permission of Dusan Losic, James G. Mitchell and Nicolas H. Voelcker, from *Chem. Commun.*, 2005, 4905.

## CHEMICAL TECHNOLOGY

T37

Chemical Technology highlights the latest applications and technological aspects of research across the chemical sciences.

## Chemical Technology

October 2005/Volume 2/Issue 10

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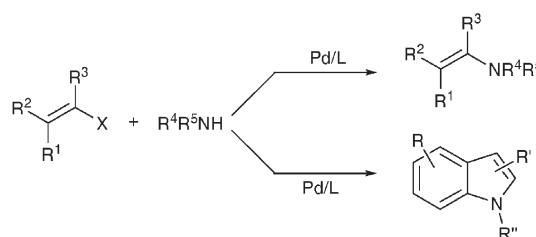
## FEATURE ARTICLE

4891

### Palladium catalyzed alkenyl amination: from enamines to heterocyclic synthesis

José Barluenga\* and Carlos Valdés

The most recent advances in the Pd catalyzed amination of alkenyl halides directed towards the synthesis of imines and enamines are reviewed. The application of this cross-coupling reaction to the synthesis of heterocycles appears to be a potentially powerful methodology. The few examples already available are also discussed.



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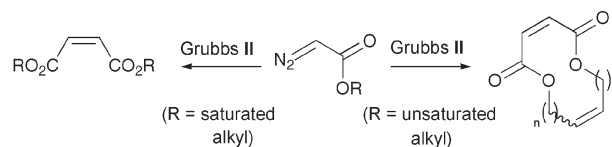
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4902

### Maleates from diazoacetates and dilactones from head-to-head dimerisation of alkenyl diazoacetates using Grubbs' 2nd-generation ruthenium carbene catalyst

David M. Hodgson\* and Deepshikha Angrish

Grubbs' 2nd-generation ruthenium carbene catalyst homocouples diazoacetates to maleates and also catalyses head-to-head dimerisation of alkenyl diazoacetates giving dienyl dilactones.

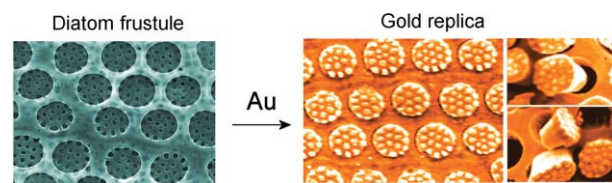


4905

### Complex gold nanostructures derived by templating from diatom frustules

Dusan Losic,\* James G. Mitchell and Nicolas H. Voelcker

Diatom frustules have been used for the first time as templates for the fabrication of gold nanostructures; high-precision replicas featuring complex three-dimensional gold nanostructures from the nano- to the microscale were achieved.

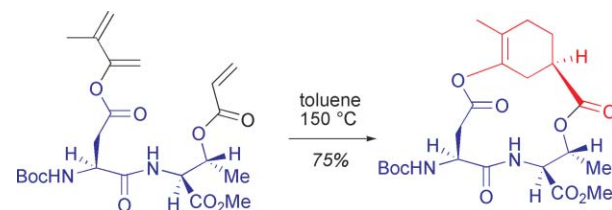


4908

### Regio- and stereocontrolled Diels–Alder reaction tethered by Asp–Thr dipeptide

Takayuki Doi,\* Yuko Miura, Susumu Kawauchi and Takashi Takahashi\*

The peptide-tethered Diels–Alder reaction proceeded in a regio- and stereoselective manner.



4911

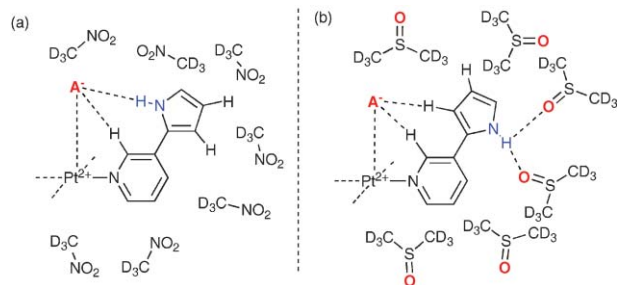
### Zeolite micropattern for biological applications

Wenqing Sun, Koon Fung Lam, Ling Wai Wong and King Lun Yeung\*

A facile method was established using composition-gradient pattern on zeolite surface to guide the deposition and formation of chemical and biomolecular patterns with features as small as five microns. The figure shows BSA-FITC deposited on 70, 10 and 5 microns wide aluminium-rich, square-wave micropattern of ZSM-5 against a Sil-1 background.



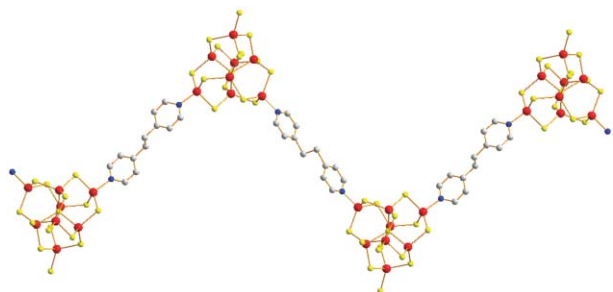
4913



### NH vs. CH hydrogen bond formation in metal–organic anion receptors containing pyrrolypyridine ligands

Ismael El Drubi Vega, Philip A. Gale,\* Mark E. Light and Stephen J. Loeb

4916

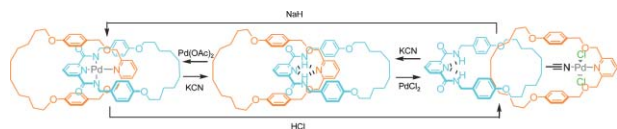


### One-dimensional coordination polymers containing penta-supertetrahedral sulfide clusters linked by dipyriddy ligands

Jingli Xie, Xianhui Bu, Nanfeng Zheng and Pingyun Feng\*

Three new 1-D coordination polymers  $[\text{Zn}_8\text{S}(\text{SC}_6\text{H}_5)_{14} \cdot \text{C}_{12}\text{H}_{10}\text{N}_2](1)$ ,  $[\text{Zn}_7\text{CoS}(\text{SC}_6\text{H}_5)_{14} \cdot \text{C}_{13}\text{H}_{14}\text{N}_2](2)$  and  $[\text{Zn}_8\text{S}(\text{SC}_6\text{H}_5)_{14} \cdot \text{C}_{13}\text{H}_{14}\text{N}_2](3)$  have penta-supertetrahedral clusters linked by linear dipyriddy ligands; the two complexes show optical transitions with band gaps of  $\sim 3.44$  eV (**1**) and  $\sim 3.54$  eV (**2**).

4919

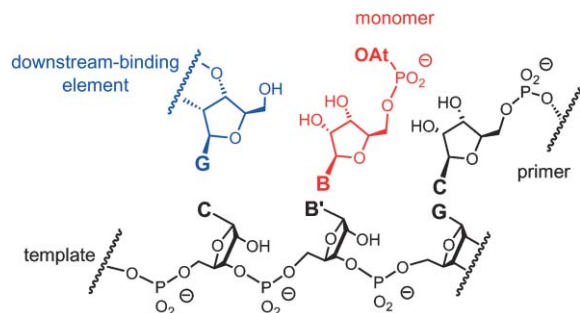


### Half-rotation in a [2]catenane *via* interconvertible Pd(II) coordination modes

David A. Leigh,\* Paul J. Lusby, Alexandra M. Z. Slawin and D. Barney Walker

Reaction of a [2]catenane with different Pd(II) salts results in different macrocycle orientations and dynamics.

4922



### Accelerating chemical replication steps of RNA involving activated ribonucleotides and downstream-binding elements

Stephanie R. Vogel, Christopher Deck and Clemens Richert\*

Non-enzymatic, sequence-selective replication steps of RNA proceed more than one order of magnitude faster when azabenzotriazolides, rather than 2-methylimidazolides, are used as monomers at pH 8.9 in the presence of downstream-binding elements.

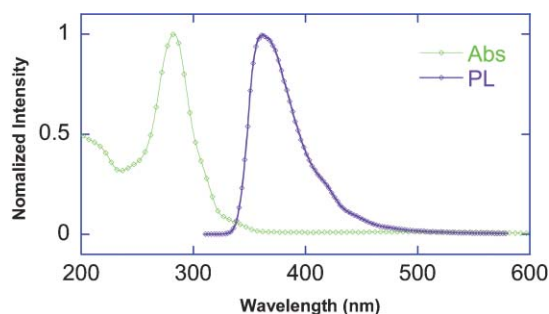


4925

**Ultraviolet-emitting conjugated polymer poly(9,9'-alkyl-3,6-silafluorene) with a wide band gap of 4.0 eV**

Yueqi Mo, Renyu Tian, Wei Shi and Yong Cao\*

An ultraviolet-emitting conjugated polymer, poly(9,9'-alkyl-3,6-silafluorene) with a wide band gap of 4.0 eV and PL emission peaked at 355 nm, has been synthesized and characterized.

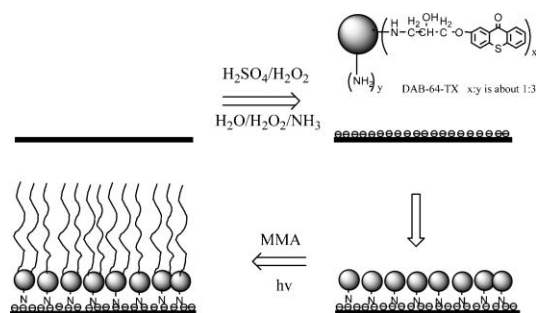


4927

**Photoinitiated synthesis of polymer brush from dendritic photoinitiator electrostatic self-assembly**

Xuesong Jiang and Jie Yin\*

We report on a simple and effective method to prepare polymer brush by electrostatic self-assembly of dendritic macrophotoinitiator and photoinitiated polymerization.

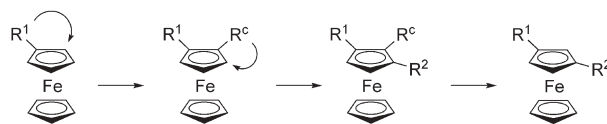


4929

**Stereoselective synthesis of chiral, non-racemic 1,2,3-tri- and 1,3-disubstituted ferrocene derivatives**

Marianne Steurer, Karin Tiedl, Yaping Wang and Walter Weissensteiner\*

Chiral, non-racemic 1,3-disubstituted ferrocene derivatives are accessible in 3 steps from monosubstituted ferrocenes.

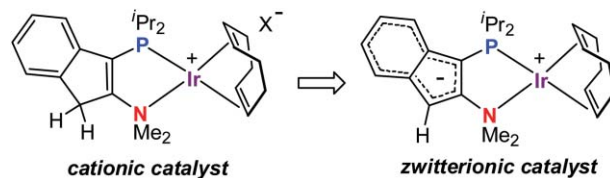


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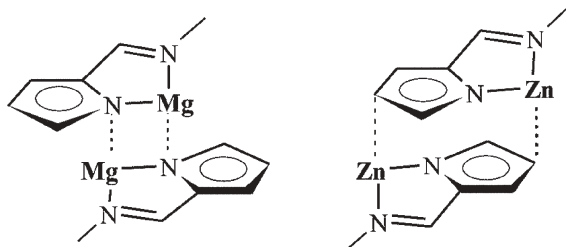
**New bidentate cationic and zwitterionic relatives of Crabtree's hydrogenation catalyst**

Judy Cipot, Robert McDonald and Mark Stradiotto\*

A new class of bidentate cationic and zwitterionic iridium complexes have been developed that are capable of mediating the hydrogenation of alkenes under mild conditions ( $\sim 1$  atm  $H_2$ ,  $22^\circ C$ ).



4935

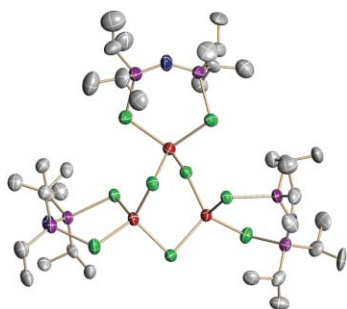


### Divergent coordination mode of magnesium and zinc alkyls supported by the bifunctional pyrrolylaldiminato ligand

Janusz Lewiński,\* Maciej Dranka, Izabela Kraszewska, Witold Sliwiński and Iwona Justyniak

The first magnesium and zinc alkyls derived from *N*-(iso-propyl)-pyrrolylaldimine have been synthesised and structurally characterised.

4938

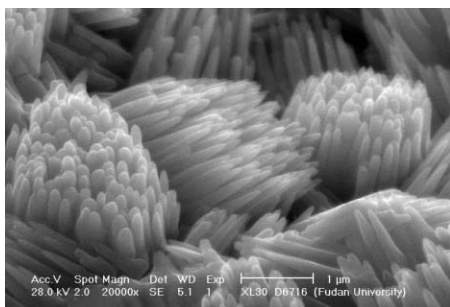


### Formation of Ga<sub>2</sub>Te<sub>2</sub> and M<sub>3</sub>Te<sub>3</sub> (M = Ga, In) rings from reactions of sodium ditelluroimidodiphosphate with Group 13 halides

May C. Copley and Tristram Chivers\*

Metathetical reactions of Na[N(Pr<sub>2</sub>PTE)<sub>2</sub>] with Group 13 metal halides produce the telluride complexes {Ga(μ-Te)[N(Pr<sub>2</sub>PTE)<sub>2</sub>]<sub>2</sub> and {M(μ-Te)[N(Pr<sub>2</sub>PTE)<sub>2</sub>]<sub>3</sub>, M = In and Ga, which contain central Ga<sub>2</sub>Te<sub>2</sub> and M<sub>3</sub>Te<sub>3</sub> rings, respectively.

4941

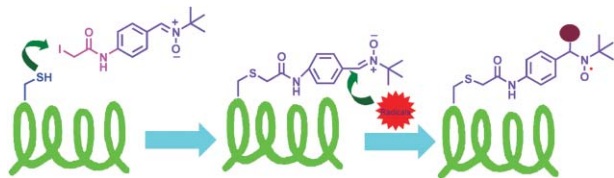


### Single crystal tin nano-rod arrays electrodeposited by a soft template

Qin Zhou,\* Xiaoying Liu, Yaomin Zhao, Nengqin Jia, Ling Liu, Manming Yan and Zhiyu Jiang\*

Metal tin nano-rod arrays were fabricated *via* electrodeposition by using the amphiphilic triblock copolymer P123 as a soft template. The rods show typical diameters of 120–140 nm with each rod in a single crystalline structure.

4943



### A novel spin trap for targeting sulphhydryl-containing polypeptides

Yang Ping Liu, Yi Qiong Ji, Yu Guang Song, Ke Jian Liu,\* Bin Liu, Qiu Tian and Yang Liu\*

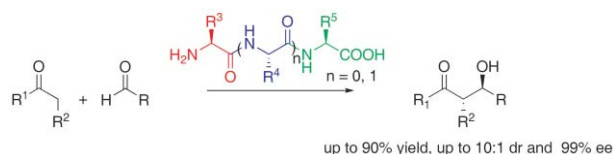
A novel spin trap containing an iodoacetamide group has been synthesized and then used to target polypeptides, *i.e.* glutathione and bovine serum albumin, by which the resulting covalently bonded bioconjugates exhibit great potential for the application of spin trapping of transient radicals in biological systems.

4946

**Small peptides as modular catalysts for the direct asymmetric aldol reaction: ancient peptides with aldolase enzyme activity**

Weibiao Zou, Ismail Ibrahim, Pawel Dziedzic, Henrik Sundén and Armando Córdoba\*

Simple modular peptides and their analogues with a primary amino group as the catalytic residue mediate the direct asymmetric intermolecular aldol reaction with high stereoselectivity and furnish the corresponding aldol products in high yield with up to 99% ee.

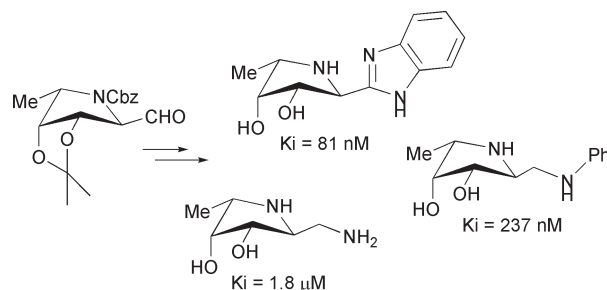


4949

**Stereoselective synthesis of (2*S*,3*S*,4*R*,5*S*)-5-methylpyrrolidine-3,4-diol derivatives that are highly selective  $\alpha$ -L-fucosidase inhibitors**

Antonio J. Moreno-Vargas, Ana T. Carmona, Federico Mora, Pierre Vogel and Inmaculada Robina\*

A novel and efficient route to new leads of highly selective and competitive inhibitors of  $\alpha$ -L-fucosidases starting from D-mannose diacetonide has been developed.

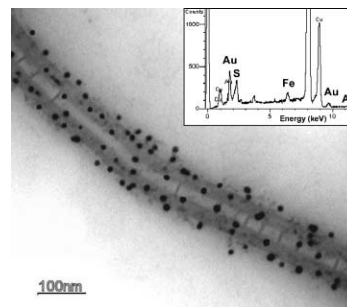


4952

**Programmed assembly of multi-layered protein/nanoparticle-carbon nanotube conjugates**

Mei Li, Erik Dujardin and Stephen Mann\*

Programmed attachment of ferritin and Au nanoparticles onto carbon nanotubes is achieved by stepwise assembly using biotin-streptavidin recognition and oligonucleotide duplexation. These interactions are exploited for reversible assembly/disassembly of Au nanoparticles.

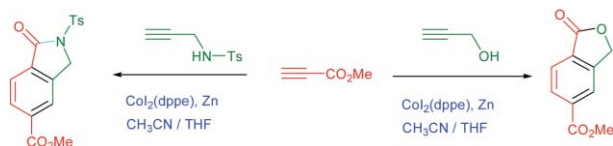


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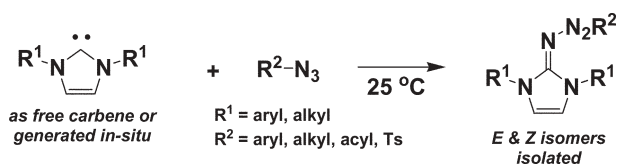
**One-pot synthesis of benzolactones and lactams via a cobalt-catalyzed regioselective [2 + 2 + 2] cocyclotrimerization of alkynyl alcohols and amines with propiolates**

Hong-Tai Chang, Masilamani Jegannmohan and Chien-Hong Cheng\*

An efficient method for the synthesis of benzolactones and benzolactams *via* a cobalt-catalyzed [2 + 2 + 2] cocyclotrimerization of alkynyl alcohols and alkynyl amines with propiolates is described.



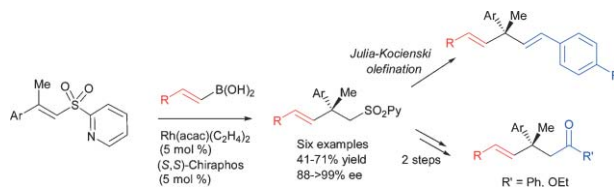
4958

**Triazene formation *via* reaction of imidazol-2-ylidenes with azides**

Dimitri M. Khramov and Christopher W. Bielawski\*

Treatment of N-heterocyclic carbenes (as their free carbenes or generated *in situ*) with alkyl, aryl, acyl or tosyl azides afforded the respective substituted triazenes in excellent yields.

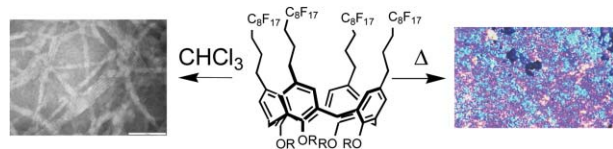
4961

**Enantioselective construction of stereogenic quaternary centres *via* Rh-catalyzed asymmetric addition of alkenylboronic acids to  $\alpha,\beta$ -unsaturated pyridylsulfones**

Pablo Mauleón and Juan C. Carretero\*

The Rh-catalyzed enantioselective addition of alkenylboronic acids to  $\beta,\beta$ -disubstituted  $\alpha,\beta$ -unsaturated 2-pyridylsulfones allows the highly enantioselective construction of quaternary stereogenic centres. The chemical versatility of the resulting sulfones enlarges the synthetic interest of this asymmetric conjugate addition procedure.

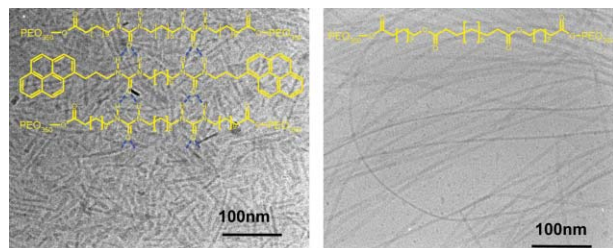
4964

**Solution self-assembly and solid state properties of fluorinated amphiphilic calix[4]arenes**

Oana M. Martin, Lian Yu and Sandro Mecozzi\*

We describe the properties of a new class of water-soluble semifluorinated amphiphilic calix[4]arenes. Fluorous-phase-directed self-assembly drives the formation of an array of different structures in solution whereas liquid crystals are formed in the solid state.

4967

**Morphological control and molecular recognition by bis-urea hydrogen bonding in micelles of amphiphilic tri-block copolymers**

Natalia Chebotareva, Paul H. H. Bomans, Peter M. Frederik, Nico A. J. M. Sommerdijk and Rint P. Sijbesma\*

Introduction of bis-urea groups into amphiphilic tri-block copolymers modifies the morphology of micelles from worm- to rod-like. The bis-urea hydrogen bonding motif allows molecular recognition of small molecules with complementary hydrogen bonding units.

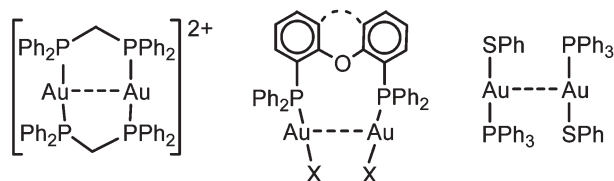


4970

### First EXAFS studies on aurophilic interactions in solution

Héctor de la Riva, Aranzazu Pintado-Alba, Mark Nieuwenhuyzen, Christopher Hardacre\* and M. Cristina Lagunas\*

EXAFS has been used to directly show the existence of Au...Au interactions in dissolved Au(I) complexes for the first time, thus allowing a better understanding of the optical properties of these materials. Three types of complexes with 'fully-supported', 'semi-supported' or 'unsupported' aurophilic interactions have been studied.

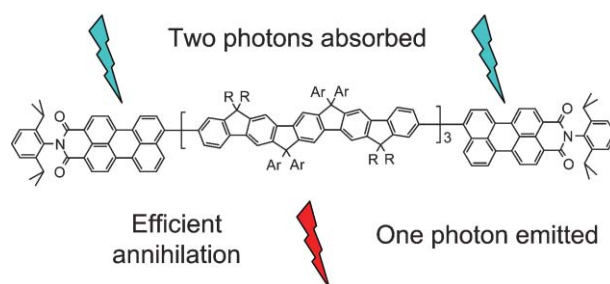


4973

### Charge transfer enhanced annihilation leading to deterministic single photon emission in rigid perylene end-capped polyphenylenes

Toby D. M. Bell, Josemon Jacob, Maria Angeles-Izquierdo, Eduard Fron, Fabian Nolde, Johan Hofkens, Klaus Müllen\* and Frans C. De Schryver\*

Annihilation between charge transfer states in the molecule shown makes it an efficient single photon source at room temperature in PMMA even though the perylene imide chromophores are  $\sim 7$  nm apart.

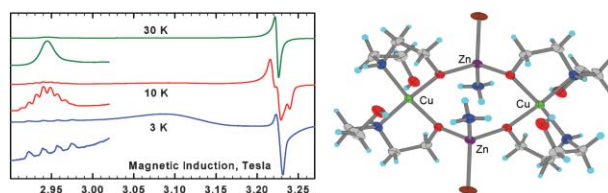


4976

### A Cu–Zn–Cu–Zn heterometallomacrocyclic shows significant antiferromagnetic coupling between paramagnetic centres mediated by diamagnetic metal

Elena A. Buvaylo, Vladimir N. Kokozay, Olga Yu. Vassilyeva, Brian W. Skelton, Julia Jezierska, Louis C. Brunel and Andrew Ozarowski\*

Antiferromagnetic exchange mediated by two –O–Zn–O– bridges, with  $J = 35.0 \text{ cm}^{-1}$ , was observed between two copper centers separated by  $5.7062(9) \text{ \AA}$  in the diethanolamine ( $\text{H}_2\text{L}$ ) complex  $[\text{Cu}_2\text{Zn}_2(\text{NH}_3)_2\text{Br}_2(\text{HL})_4]\text{Br}_2 \cdot \text{CH}_3\text{OH}$ .

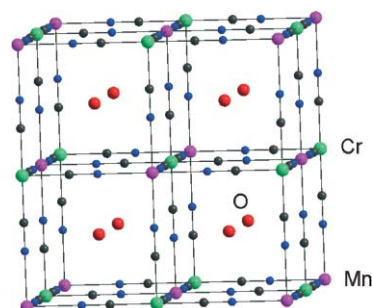


4979

### Structure and magnetic properties of a new ferrimagnet containing a paramagnetic $[\text{Cr}(\text{CN})_5(\text{NO})]^{3-}$ building block

Hui-Bo Zhou, Wei Zhang, Kazuyoshi Yoshimura, Yan Ouyang, Dai-Zheng Liao,\* Zong-Hui Jiang, Shi-Ping Yan and Peng Cheng

A new molecular magnet containing a paramagnetic  $[\text{Cr}(\text{CN})_5(\text{NO})]^{3-}$  building block has been synthesized and characterized. The observed magnetic behavior displays the nature of a ferrimagnet.





### A novel and facile Zn-mediated intramolecular five-membered cyclization of $\beta$ -tetraarylporphyrin radicals from $\beta$ -bromotetraarylporphyrins

Dong-Mei Shen, Chao Liu and Qing-Yun Chen\*

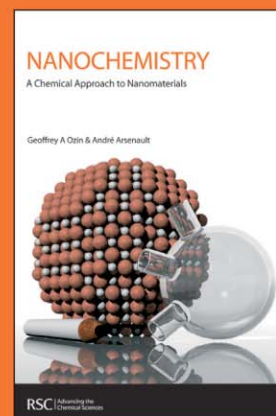
A novel and facile method for the Zn-mediated intramolecular cyclization of  $\beta$ -porphyrin radicals has been developed for the convenient and effective construction of newly fused five-membered porphyrin systems from readily available  $\beta$ -bromotetraarylporphyrins.

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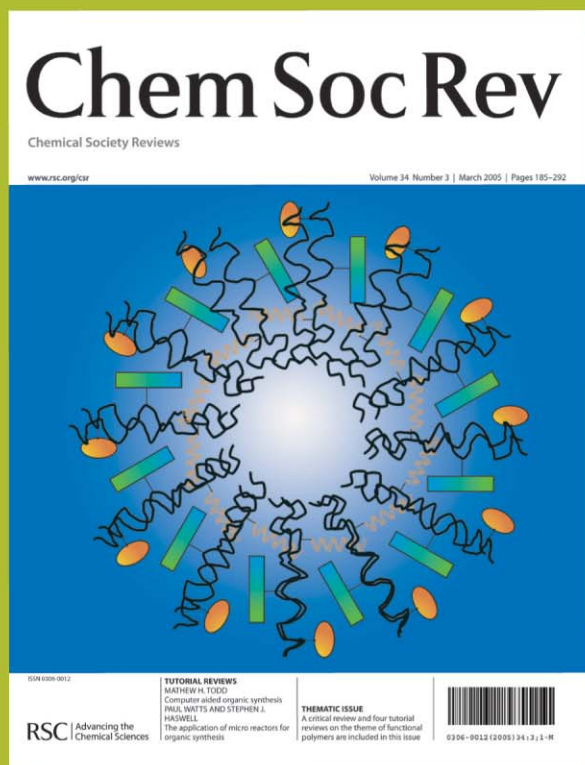


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