# ChemComm

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**Cover** Aromatic amides achieve conformational preference rather like windmills facing into the wind (pp. 127–135).

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# EATURE ARTICLE

# Atropisomers and near-atropisomers: achieving stereoselectivity by exploiting the conformational preferences of aromatic amides

Jonathan Clayden

By exploiting simple conformational preferences in amides, high levels of stereoselectivity may be achieved. This strategy has been applied to the synthesis of chiral ligands and auxiliaries, and enables ultra-remote stereocontrol.

## OMMUNICATIONS

### **Polymeric silver(I) coordination tubes**

Alexandra L. Pickering, Georg Seeber, De-Liang Long and Leroy Cronin\*

Isolated polymeric Ag(I) coordination tubes,  $\{[Ag(trans-tach)]CF_3SO_3\}_{\infty}$  and  $\{[Ag(cis-tach)](CH_3OH)CF_3SO_3\}_{\infty}$  are self-assembled from the rigid triamino ligands *cis,cis*-1,3,5-triaminocyclohexane (*cis*-tach) and *cis,trans*-1,3,5-triaminocyclohexane (*trans*-tach), forming two topologically equivalent 1-D frameworks.



Ag(I)

# Host-guest interactions template: the synthesis of a [3]catenane

Amy L. Hubbard, Gregory J. E. Davidson, Roopa H. Patel, James A. Wisner and Stephen J. Loeb\*

Synthesis of [3]catenanes containing 1,2-bis(4,4'-bipyridinium)ethane binding sites and various 24-crown-8 ethers shows that dibenzo-24-crown ether is ideally suited as an external template for the one-step synthesis of the [3]catenane that contains this crown. Solution studies demonstrate the reversible binding of the crown guest and its X-ray structure shows a unique three-layer adduct,  $\{[3]catenane \subset (DB24C8 \subset (2H_2O))\}$ .

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NMR and computational studies of the chemical reduction of [2.2]paracyclophane: formation of dianionic *p*-xylenyl oligomers

Ilya D. Gridnev\* and Fabio Pichierri

Reaction of [2.2]paracyclophane with K/Na alloy in THF gives a *p*-xylylenyl dianion together with its dimer and trimer which are relatively stable at low temperatures; at ambient temperatures further polymerization takes place.

## Inherent helicity in an extended tris-bipyridyl molecular cage

David F. Perkins, Leonard F. Lindoy,\* George V. Meehan\* and Peter Turner

A new molecular cage incorporating three bipyridyl units has been synthesised by a conventional multi-step procedure as well as, much more efficiently, by a Ni(II) template procedure.

# Synthesis of new thermotropic liquid crystalline polyurethanes containing biphenyl mesogens using a novel AB-type self-polycondensation

T. Ranganathan, C. Ramesh and Anil Kumar\*

A series of thermotropic main chain liquid crystalline polyurethanes containing biphenyl mesogens and flexible methylene spacers were synthesized using the novel AB-type selfpolycondensation approach for the first time.

# Structure and magnetism of a new pyrazolate bridged iron(II) spin crossover complex displaying a single HS–HS to LS–LS transition

Ben A. Leita, Boujemaa Moubaraki, Keith S. Murray,\* Jonathan P. Smith and John D. Cashion

The dinuclear iron(II) complex [(pypzH)(NCSe)Fe( $\mu$ -pypz)<sub>2</sub>Fe(NCSe)(pypzH)]· 2H<sub>2</sub>O displays a single, sharp spin crossover transition between the [HS–HS] and [LS–LS] states and is structurally characterised above and below the  $T_{1/2} = 225$ K value.

# The regiospecific Fischer indole reaction in choline chloride $2ZnCl_2$ with product isolation by direct sublimation from the ionic liquid

Raul Calderon Morales, Vasuki Tambyrajah, Paul R. Jenkins,\* David L. Davies and Andrew P. Abbott

The Fischer indole synthesis occurs in high yield with one equivalent of the ionic liquid choline chloride  $2ZnCl_2$ ; exclusive formation of 2,3-disubstituted indoles is observed in the reaction of alkyl methyl ketones, and the products readily sublime directly from the ionic liquid.

i v



v



(R =  $C_6H_{13}$ , OBu, (CH<sub>2</sub>)<sub>2</sub>Ph, (CH<sub>2</sub>)<sub>3</sub>OTBDMS, (CH<sub>2</sub>)<sub>3</sub>OC(O)CH<sub>3</sub>, N-Phthalimide)

63-88%



# Selective host–guest interaction of single-walled carbon nanotubes with functionalised fullerenes

under microwave irradiation.

David A. Britz,\* Andrei N. Khlobystov,\* Jiawei Wang, Adam S. O'Neil, Martyn Poliakoff, Arzhang Ardavan and G. Andrew D. Briggs

Ester functionalised fullerenes were inserted into nanotubes using  $scCO_2$  without degrading functional groups. Carboxylate functionalised fullerenes aggregated to form a supramolecular complex on the surface of the nanotube, which sterically hindered encapsulation.

# $\begin{array}{c} & & \\$

# Preparation of tripyrrane analogues from resorcinol and 2-methylresorcinol for applications in the synthesis of new benziporphyrin systems

Kae Miyake and Timothy D. Lash\*

Resorcinol and methylresorcinol reacted with 2 equiv. of an acetoxymethylpyrrole in the presence of acid catalysts to form 4,6-dihydroxy-1,3-bis(2-pyrrolylmethyl)benzenes that structurally resemble the tripyrranes; following deprotection of the terminal ester moieties, these underwent acid catalyzed condensations with dialdehydes to afford a series of novel benziporphyrins.





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EDC, H2O / DMF, pH 4.75, 62%

# H<sub>2</sub>L + Fe<sup>3\*</sup> + Fe<sub>4</sub>L<sub>6</sub> + Intermolecular Interactions

# Synthesis and Bergman cyclization of a $\beta$ -extended porphyrenediyne

John D. Spence,\* Eric D. Cline, Domingo M. LLagostera and Patrick S. O'Toole

Condensation of a porphyrin-2,3-dione with a 1,2-

diaminoarenediyne affords a  $\beta$ -extended porphyrinic-enediyne. Upon thermal Bergman cyclization the quinoxaline spacer positioned between the macrocycle and the enediyne prevents tandem radical cyclization to a picenoporphyrin.

# A fluorescent analogue of UDP-*N*-acetylglucosamine: application for FRET assay of peptidoglycan translocase II (MurG)

Jian-Jun Li and Timothy D. H. Bugg\*

A direct continuous fluorescence assay for translocase II MurG based on fluorescence resonance energy transfer (FRET) has been developed using a 6-substituted fluorescent analogue of UDP-*N*-acetylglucosamine.

# A concise stereocontrolled formal total synthesis of (±)-podophyllotoxin using sulfoxide chemistry

Mike Casey\* and Claire M. Keaveney

A highly stereoselective *one-pot* construction of a tetralin is the key step in a concise new route to the important anti-cancer drug precursor  $(\pm)$ podophyllotoxin.

# A three dimensional porous metal–organic framework $[Fe_4L_6 \cdot (DMF)_3 \cdot (H_2O)_{10}]$ constructed from neutral discrete $Fe_4L_6$ pyramids $[H_2L = 1,3$ -benzodihydroxamix acid]

Yan Bai, Dong Guo, Chun-ying Duan,\* Dong-bin Dang, Ke-liang Pang and Qing-jin Meng\*

A 3-D porous zeolite-like metal–organic framework assembled from a well-defined tetrahedral Fe<sub>4</sub>L<sub>6</sub> cavity by the cooperativity of hydrogen bonds and  $\pi$ - $\pi$  stacking showing the ability to survive guest removal.

# A simple copper salt catalysed the coupling of imidazole with arylboronic acids in protic solvent

Jing-Bo Lan, Li Chen, Xiao-Qi Yu,\* Jing-Song You and Ru-Gang Xie\*



In the presence of a catalytic amount of a simple copper salt, the coupling of imidazole with arylboronic acids was performed in methanol to give corresponding *N*-arylimidazoles in almost quantitative yields.







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Exhaust Oxidising Aged catalysts Pd/CZ & Pd/Al CO conversion / % 75 50 25 75 Oxidising 50 8 573 473 573 Temperature 473 573 Temperature

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# 11-Aminoundecanoic acid: a versatile unit for the generation of low molecular weight gelators for water and organic solvents

Anthony D'Aléo, Jean-Luc Pozzo, Frédéric Fages,\* Marc Schmutz, Gudrun Mieden-Gundert, Fritz Vögtle,\* Vesna Caplar and Mladen Zinic\*

11-Aminoundecanoic acid represents a powerful building-block for the systematic, easy synthesis of efficient gelators, including chiral ones. Unprecedentedly, some racemates are found to be stronger gelators than the corresponding pure enantiomers.

# Synthesis and structural characterization of the first unsymmetrical diarylpalladium complex trans-Pd(C<sub>6</sub>F<sub>5</sub>)(2,4,6-C<sub>6</sub>F<sub>3</sub>H<sub>2</sub>)(PEt<sub>3</sub>)<sub>2</sub>, derived from transmetallation between 2,4,6-trifluorophenylboronic acid and $trans-Pd(C_6F_5)I(PEt_3)_2$

Yasushi Nishihara, Hiroyuki Onodera and Kohtaro Osakada\*

The first example of an X-ray analysis for an unsymmetrical diarylpalladium(II) complex that contains fluorine atoms in the ortho positions, is accessible via transmetallation of an arylboronic acid in the presence of Ag<sub>2</sub>O.

# Intramolecular charge separation in a hydrogen bonded tyrosineruthenium(II)-naphthalene diimide triad

Olof Johansson, Henriette Wolpher, Magnus Borgström, Leif Hammarström,\* Jonas Bergquist, Licheng Sun and Björn Åkermark\*

Long-lived charge-separated states in the ns to µs range were observed upon laser flash excitation of a donor-chromophore-acceptor triad based on tris(bipyridine) ruthenium(II) as photo-sensitizer, naphthalene diimide as acceptor, and a hydrogen bonded phenol as donor.

# Reactivation of aged model Pd/Ce<sub>0.68</sub>Zr<sub>0.32</sub>O<sub>2</sub> three-way catalyst by high temperature oxidising treatment

N. Hickey, P. Fornasiero, R. Di Monte, J. Kašpar,\* J. R. González-Velasco,\* M. A. Gutiérrez-Ortiz, M. P. González-Marcos, J. M. Gatica and S. Bernal\*

Pd/Ce<sub>0.68</sub>Zr<sub>0.32</sub>O<sub>2</sub> (PdCZ) catalyst is remarkably reactivated when subjected to a high temperature oxidising treatment whereas this effect is only marginal for a conventional Pd/Al<sub>2</sub>O<sub>3</sub> (PdAl), indicating the importance of Pd–Ce<sub>0.68</sub>Zr<sub>0.32</sub>O<sub>2</sub> interactions in obtaining highly active catalysts.

# C<sub>n</sub> microspheres as surrogate membranes in glycosidase-catalysed hydrolysis of glycolipids

José A. R. Martins, David H. G. Crout\* and Peter Critchley

Glycosidase catalysed hydrolysis of glycolipids non-covalently attached to  $C_n$ microspheres proceeds to completion for appropriate glycolipid-microsphere combinations.



C<sub>n</sub> Microsphere surface



iх

# Purification by HPLC and the UV/Vis absorption spectra of the nitrogen-containing *incar*-fullerenes *i*NC<sub>60</sub>, and *i*NC<sub>70</sub>









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# Mito Kanai, Kyriakos Porfyrakis, G. Andrew. D. Briggs and T. John S. Dennis\*

The nitrogen-containing *incar*-fullerenes  $iNC_{60}$  and  $iNC_{70}$ , have been purified and characterised by UV-Vis absorption spectroscopy.

# Unprecedented metal template effect on the coupling of dithiafulvene moieties

Dominique Lorcy,\* Michel Guerro, Pascal Pellon and Roger Carlier

The first approach to an original vinylphosphine, the dithiafulvenyldiphenylphosphine, and its chelating ability towards the  $Mo(CO)_4$  fragment are reported. Upon oxidation, the  $Mo(CO)_4$  complex incorporating two such ligands leads to a novel metallacycle substituted by a redox active vinylogous tetrathiafulvalene.

# Nuclear inelastic scattering spectroscopy of iron-sulfur cubane compounds

Vasily S. Oganesyan, J. Elaine Barclay, Sinead M. Hardy, David J. Evans, Christopher J. Pickett and Upali A. Jayasooriya\*

The potential of NIS spectroscopy to study the iron–sulfur clusters in metalloproteins is illustrated using model compounds. The origin of the intense low energy transfer bands is discussed.

# An efficient chemical fixation of nitric oxide: convenient and practical synthesis of 1,2,3-oxadiazole 3-oxides

Takumichi Sugihara,\* Kimiko Kuwahara, Akihito Wakabayashi, Hiroko Takao, Hiroshi Imagawa and Mugio Nishizawa

Nitric oxide reacts efficiently with alkynyllithium at low temperature producing 1,2,3-oxadiazole 3-oxides in good yields.

# The first ligand-modulated oxidative Heck vinylation. Efficient catalysis with molecular oxygen as palladium(0) oxidant

Murugaiah M. S. Andappan, Peter Nilsson and Mats Larhed\*

The discovery of ligand-promoted Pd(II)-catalysed vinylation of arylboronic acids, low catalyst loading, oxidatively stable ligand, and dioxygen as clean oxidant of Pd(0) are the highlights.

х



Chem. Commun., 2004

хi



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### Polymers of intrinsic microporosity (PIMs): robust, solutionprocessable, organic nanoporous materials

Peter M. Budd, Bader S. Ghanem, Saad Makhseed, Neil B. McKeown,\* Kadhum J. Msayib and Carin E. Tattershall

Microporous materials can be derived directly from soluble polymers whose randomly contorted shapes prevent an efficient packing of the macromolecules in the solid state.

# Dichloro(dodeca-2,6,10-triene-1,12-diyl)ruthenium(IV): a highly efficient catalyst for the isomerization of allylic alcohols into carbonyl compounds in organic and aqueous media

Victorio Cadierno,\* Sergio E. García-Garrido and José Gimeno\*

The bis(allyl)-ruthenium(IV) complex  $[Ru(\eta^3;\eta^2:\eta^3-C_{12}H_{18})Cl_2]$  ( $C_{12}H_{18}$  = dodeca-2,6,10-triene-1,12-diyl) has been found to be an efficient catalyst for the isomerization of allylic alcohols into carbonyl compounds both in THF and water as solvent.

# Polyferrocenes: metallopolymers with tunable and high refractive indices

Chantal Paquet, Paul W. Cyr, Eugenia Kumacheva\* and Ian Manners\*

The refractive index, molar refraction and Abbé number of polyferrocene derivatives are reported and the values indicate that these materials are very promising for a range of photonics applications.

# The intramolecular Baylis–Hillman reaction: easy preparation of versatile substrates, facile reactions, and synthetic applications

Jung Eun Yeo, Xiuling Yang, Hee Jin Kim and Sangho Koo\*

Easy preparation of  $\omega$ -formyl- $\alpha$ , $\beta$ -unsaturated carbonyl compounds and their facile intramolecular Baylis–Hillman reactions can be applied to the efficient syntheses of useful cyclic frameworks for polycyclic natural products.

# Synthesis and ring opening reactions of a 2-silabicyclo[2.1.0]pentane

Gerhard Maas,\* Birgit Daucher, Alexandra Maier and Voker Gettwert

The first 2-silabicyclo[2.1.0]pentane was synthesized and found to undergo different ring opening reactions, including formation of an allyl(alkoxysilyl)ketene.









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CO<sub>2</sub>Me





# $\begin{array}{c} \text{OH} \\ \text{R} \\ \text{R} \\ \text{R} \\ \text{CH}_2 \\ \text{OH} \end{array} \xrightarrow[V^{n+}]{} \text{NHPI, } [V^{n+}], \text{RX or amine} \\ \text{MeCN, 50-75^{\circ}C, 1atm. } O_2, 1-22h \\ \text{RX = LiCl, } Bu_4 \\ \text{NCl; amine = py, 4-Me-py} \\ \text{RCOOH} \\ \text{[V^{n+}] = VO(acac)_2, VO(OPr)_3, } [Bu_4 \\ \text{N}] \\ \text{VO}_3 \end{array}$

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Synthesis and characterization of organic–inorganic hybrid mesoporous silica materials with new templates

Byunghwan Lee, Huimin Luo, C. Y. Yuan, J. S. Lin and Sheng Dai\*

A new surfactant system has been successfully explored for a self-assembly synthesis of periodic mesoporous organosilica (PMO) materials.

# Photochemical oxidative addition of B–H bonds at ruthenium and rhodium

Philip L. Callaghan, Rodrigo Fernández-Pacheco, Naser Jasim, Sébastien Lachaize, Todd B. Marder, Robin N. Perutz,\* Eleonora Rivalta and Sylviane Sabo-Etienne

B–H oxidative addition occurs on photolysis of metal phosphine dihydrides in the presence of HB(pin); the rate of the elementary step of B–H oxidative addition lies between those for the corresponding oxidative addition reactions of Et<sub>3</sub>SiH and dihydrogen.

# New efficient aerobic oxidation of some alcohols with dioxygen catalysed by *N*-hydroxyphtalimide with vanadium co-catalysts

Paweł J. Figiel, Jarosław M. Sobczak\* and Józef J. Ziółkowski

New efficient vanadium co-catalysts have been developed for the oxidation of some alcohols with  $O_2$  catalysed by *N*-

hydroxyphthalimide (NHPI). Various alcohols (primary and secondary) were selectively oxidized by  $O_2$  under mild conditions with high conversions (from 72 to 96%) and selectivities (66–100%) after 1–22 hours.



# The different role of Cu<sup>++</sup> and Zn<sup>++</sup> ions in affecting the interaction of prion peptide PrP106-126 with model membranes

Domenico Grasso,\* Danilo Milardi, Carmelo La Rosa and Enrico Rizzarelli

Differential scanning calorimetric (DSC) experiments have shown that the ability of PrP106-126 to perturb 1,3-dipalmitoyl-*sn*-glycero-3-phosphocholine (DPPC) model membranes is differently affected by  $Cu^{++}$  and  $Zn^{++}$  ions.



# On-chip electrochemical measurement of $\beta\mbox{-galactosidase}$ expression using a microbial chip

Takatoshi Kaya, Kuniaki Nagamine, Nobuto Matsui, Tomoyuki Yasukawa, Hitoshi Shiku and Tomokazu Matsue\*

 $\beta$ -Galactosidase expression in a small number of *Escherichia coli* cells embedded in collagen gel has been measured in real time with an electrochemical sensor chip using *p*-aminophenyl  $\beta$ -D-galactopyranoside as a substrate.



# Indium(I) trifluoromethanesulfonate and other soluble salts for univalent indium chemistry

Charles L. B. Macdonald,\* Andrea M. Corrente, Christopher G. Andrews, Alexis Taylor and Bobby D. Ellis

We report the synthesis, structure and preliminary reactivity studies of a series of unusually soluble indium(I) salts that are improved alternatives to indium(I) halide reagents.

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