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#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (47) 4961-5100 (2007)



Cover See Joseph M. Mabry et al., pp. 4992-4994. Molecular and surface visualization of fluorinated polyhedral oligomeric silsesquioxanes (F-POSS). F-POSS can be corner-capped with trifluoropropyl (left), methyl (right), or other organic groups for tunable hydrophobicity. Image reproduced by permission of Scott T. Iacono, Ashwani Vij, Wade Grabow, Dennis W. Smith, Jr. and Joseph M. Mabry from Chem. Commun., 2007, 4992.

#### CHEMICAL SCIENCE

C89

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**Chemical Science** 

December 2007/Volume 4/Issue 12 www.rsc.org/chemicalscience

#### FEATURE ARTICLE

#### 4978

### Carboranyl thymidine analogues for neutron capture therapy

Werner Tjarks,\* Rohit Tiwari, Youngjoo Byun, Sureshbabu Narayanasamy and Rolf F. Barth

The chemical, structural, and biological properties of 3CTAs (3-carboranyl thymidine analogues), a novel class of neutron capture therapy (NCT) agents, are discussed.



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Chemical Communications (print: ISSN 1359-7345; electronic: ISSN 1364-548X) is published 48 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF. All orders, with cheques made payable to the Royal Society of Chemistry, should be sent to RSC Distribution Services, c/o Portland Customer Services, Commerce Way, Colchester, Essex, UK CO2 8HP. Tel +44 (0)1206 226050; E-mail sale@rscdistribution.org

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#### 4992



#### silsesquioxane nanostructures Scott T. Iacono, Ashwani Vij, Wade Grabow,

Dennis W. Smith, Jr. and Joseph M. Mabry\*

New fluorinated polyhedral oligomeric silsesquioxane (F-POSS) structures possessing a high degree of hydrophobicity have been prepared by employing a cornercapping methodology.

Facile synthesis of hydrophobic fluoroalkyl functionalized

#### 4995

### Construction of a small-molecule-integrated semisynthetic split intein for *in vivo* protein ligation

Tomomi Ando, Shinya Tsukiji, Tsutomu Tanaka and Teruyuki Nagamune\*

Design and construction of a small-molecule-integrated semisynthetic split intein that can be used for protein ligation and protein semisynthesis on the cell surface and potentially inside cells is described.



#### 4998

#### Low valent phosphorus in the molecular anions $[P_5Se_{12}]^{5-1}$ and $\beta$ - $[P_6Se_{12}]^{4-1}$ : phase change behavior and near infrared second harmonic generation

In Chung, Joon I. Jang, Matthew A. Gave, David P. Weliky and Mercouri G. Kanatzidis\*

The Cs<sup>+</sup> salts of novel molecular anions  $[P_5Se_{12}]^{5-}$  and  $[P_6Se_{12}]^{4-}$  are described.  $[P_5Se_{12}]^{5-}$  has a  $P^{3+}$  center with octahedral coordination and its non-centrosymmetric salt exhibits SHG response. Both compounds are crystal–glass phase change materials.

#### 5001

### Highly crystalline anisotropic superstructures *via* magnetic field induced nanoparticle assembly

Jong-Il Park, Young-wook Jun, Jin-sil Choi and Jinwoo Cheon\*

A magnetic field is successfully utilized to induce the fabrication of size controllable one-dimensional (1-D) supercrystals which are composed of a highly crystalline assembly of fcc-packed cobalt nanoparticles; the anisotropy associated supercrystal magnetism is enhanced with four times higher coercivity than that of randomly aggregated nanoparticles.





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#### Monodisperse water-soluble magnetite nanoparticles prepared by polyol process for high-performance magnetic resonance imaging

Jiaqi Wan, Wei Cai,\* Xiangxi Meng and Enzhong Liu

Monodisperse water-soluble magnetite nanoparticles which have great potential as high-performance MRI contrast agents for cell or molecular imaging have been prepared *via* high temperature decomposition of an iron precursor in liquid polyols.

#### 5007

### Syntheses and structures of mononuclear lutetium imido complexes with very short Lu–N bonds

Tarun K. Panda, Sören Randoll, Cristian G. Hrib, Peter G. Jones, Thomas Bannenberg and Matthias Tamm\*

The imidazolin-2-iminato lutetium complexes 1 and 2 with terminal imido ligands feature the shortest Lu–N bonds on record, raising the question of lanthanide–nitrogen multiple bonding.



Fe Concentration (mM)

1/64 1/32 1/1

#### 5010

## Effective anion sensing based on the ability of copper to affect electron transport across self-assembled monolayers

V. Ganesh, Maria Pilar Calatayud Sanz and Juan C. Mareque-Rivas\*

The ability of copper ions to affect the charge-transfer resistance of self-assembled monolayers (SAMs) of a tris-(2-pyridylmethyl)amine-based ligand on to gold electrodes is used to create a novel, sensitive and selective electrochemical cyanide sensor.

#### 5013

### A hamburger-shaped helical stacking of disk-shaped ligands mediated by silver(II) ions

Ho Yong Lee, Jaejoon Park, Myoung Soo Lah and Jong-In Hong\*

We describe a hamburger-shaped helical structure of chiral and achiral  $C_3$ -symmetric disk-shaped ligands mediated by silver ions.







[Project Prospect] is an exciting application of ontologies that will help researchers. Midori Harris European Bioinformatics Institute

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#### Preferential separation of fullerene[84] from fullerene mixtures by encapsulation

Elisa Huerta, Enrique Cequier and Javier de Mendoza\*

The encapsulation of fullerenes with a cyclotriveratrylene derivative, capable to self-assemble into a dimer by means of three strong quadruple hydrogen bonding scaffolds allows an easy enrichment of  $C_{84}$  directly from fullerene mixtures.

#### 5019

#### **Electrochemical detection of kinase-catalyzed** thiophosphorylation using gold nanoparticles

Kagan Kerman and Heinz-Bernhard Kraatz\*

An electrochemical biosensor for kinase-catalyzed reactions is coupled with the thiophosphorylation of the substrate peptide using adenosine 5'-[ $\gamma$ -thio] triphosphate (ATP-S) as the co-substrate.



#### 5022

### A photocatalytic water splitting device for separate hydrogen and oxygen evolution

Elena Selli,\* Gian Luca Chiarello, Eliana Quartarone, Piercarlo Mustarelli, Ilenia Rossetti and Lucio Forni

Separate hydrogen and oxygen production from photocatalytic water splitting was achieved in a two-compartment Plexiglas cell, on a thin  $TiO_2$  layer deposited by magnetron sputtering on a Ti electrode.

#### 5025

### A diastereoselective carbocyclisation of allene-hydrazones through the intramolecular allylic transfer reaction

Sang-Hoon Kim, Seung-Ju Oh, Yurim Kim and Chan-Mo Yu\*

A highly diastereoselective synthesis of cyclic hydrazines was achieved from the carbocyclisation of allene-hydrazones by the Pd-catalyzed distannylation of an allene moiety, followed by the transmetallation of allylic stannane intermediates with TiCl<sub>4</sub>.



Reagents and conditions: i. Me<sub>3</sub>SnSnMe<sub>3</sub>, (π-allyl)<sub>2</sub>Pd<sub>2</sub>Cl<sub>2</sub> (3 mol%), -40 °C, 3 h, CH<sub>2</sub>Cl<sub>2</sub> ii. TiCl<sub>4</sub>, -78 °C, 2 h





5031

5034

2 BnNH<sub>2</sub>

HO

Q



1 mol% [Cp\*IrCl<sub>2</sub>]<sub>2</sub>

15 h, then

15 h

Bn

90%

OH.

OH

A multifunctional nanoassembly of mesogen-bearing amphiphiles and porphyrins for the simultaneous photodelivery of nitric oxide and singlet oxygen

Elisa B. Caruso, Enzo Cicciarella and Salvatore Sortino\*

The first example is shown of a molecular nanoassembly able to supply simultaneously, in the same region of space and under the exclusive control of visible light, nitric oxide and singlet oxygen, two species playing a key role in the therapy of cancer.

#### Entangled palladium nanoparticles in resin plugs

Romain Najman, Jin Ku Cho, Andrew F. Coffey, John W. Davies and Mark Bradley\*

Palladium nanoparticles were entrapped within resin plugs and used in a range of ligand-free cross-coupling reactions, easily recovered and multiply reused.

#### Iridium catalysed synthesis of piperazines from diols

Lars Ulrik Nordstrøm and Robert Madsen\*

Piperazines are prepared by cyclocondensation of diols with either a primary amine or a 1,2-diamine in aqueous media in the presence of a catalytic amount of [Cp\*IrCl<sub>2</sub>]<sub>2</sub>.



#### Tetravalent cerium carbene complexes

Ian J. Casely, Stephen T. Liddle, Alexander J. Blake, Claire Wilson and Polly L. Arnold\*

The tetravalent organometallic cerium complex [CeL<sub>4</sub>] is readily accessible from the oxidation of the trivalent [CeL<sub>4</sub>] is The [CeL<sub>4</sub>] and [UL<sub>4</sub>] analogue show significantly different structures, highlighting the differences between 4f and 5f metals.

#### 5040

#### The hydrothermal synthesis of tetragonal tungsten bronze-based catalysts for the selective oxidation of hydrocarbons

Pablo Botella, Benjamín Solsona, Ester García-González, José M. González-Calbet and José M. López Nieto\*

Mixed metal oxides with tetragonal tungsten bronze (TTB) structure, showing high activity and selectivity for the gas phase partial oxidation of olefins, have been prepared by hydrothermal synthesis from Keggin-type heteropolyacids.

#### 5043

### Strong inhibition of cholera toxin binding by galactose dendrimers

Hilbert M. Branderhorst, Rob M. J. Liskamp, Gerben M. Visser and Roland J. Pieters\*

Glycodendrimers derived from cheap galactose inhibit cholera toxin binding with such potency that they can compete with the natural complex pentasaccharide ligand.



CTB  $H_{D}$   $H_{D}$ 

#### 5046

#### Homoleptic Cu-phosphorus and Cu-ethene complexes

Gustavo Santiso-Quiñones,\* Andreas Reisinger, John Slattery and Ingo Krossing\*

Stable salts of the first homoleptic Cu–phosphorus and Cu–ethene complexes, isolated by the aid of the weakly coordinating anion (WCA)  $[Al(OC(CF_3)_3)_4]^-$ , were obtained by the reaction of a three-fold excess of CuI and Ag(WCA) in the presence of the ligand.



#### 5049

#### Bismesitylmagnesium: a thermally stable and non-nucleophilic carbon-centred base reagent for the efficient preparation of silyl enol ethers

William J. Kerr,\* Allan J. B. Watson and Douglas Hayes

Bismesitylmagnesium has been established as a practical non-nucleophilic carbon-centred base reagent for access to silyl enol ethers from ketone substrates at readily utilisable temperatures without the requirement for any amine species.





# 5058 R = 0 R = 1-naphthyl R = 1-naphthyl

4a 94% yield, 96% ee

#### Unique formation of two high-nuclearity metallamacrocycles from a mononuclear complex [Zn(dmpzdtc)<sub>2</sub>] (dmpzdtc = 3,5-dimethylpyrazole-1dithiocarboxylate) *via* CS<sub>2</sub> elimination

Hong-Xi Li, Hua-Zhou Wu, Wen-Hua Zhang, Zhi-Gang Ren, Yong Zhang and Jian-Ping Lang\*

Complete  $CS_2$  elimination from a mononuclear complex  $[Zn(dmpzdtc)_2]$  in solvents saturated with water led to the formation of two high-nuclearity complexes  $[Zn_4(\mu-dmpz)_6(\mu-OH)_2]_2$  and  $[Zn_4(\mu-dmpz)_6(\mu-OH)_2]_4$ .

#### Copper(I) mediated oligomerisation of a phosphaalkyne

Ulf Vogel, John F. Nixon and Manfred Scheer\*

The oligomerisation of *tert*-butylphosphaalkyne mediated by CuI yields an unprecedented  $C_4P_5$ -cage embedded in a CuI matrix, which can be removed with aqueous NaCN to yield the free cage compound along with rearrangement products.

#### Bicyclic guanidine-catalyzed enantioselective phospha-Michael reaction: synthesis of chiral $\beta$ -aminophosphine oxides and $\beta$ -aminophosphines

Xiao Fu, Zhiyong Jiang and Choon-Hong Tan\*

A chiral bicyclic guanidine has been found to catalyze the phospha-Michael reactions of diaryl phosphine oxide to nitroalkenes with high enantioselectivities, offering a direct methodology to prepare chiral  $\beta$ -aminophosphine oxides and  $\beta$ -aminophosphines.



Wavelength (nm)

### Regioregular poly(3-hexyl)selenophene: a low band gap organic hole transporting polymer

Martin Heeney,\* Weimin Zhang, David J. Crouch, Michael L. Chabinyc, Sergey Gordeyev, Rick Hamilton, Simon J. Higgins, Iain McCulloch, Peter J. Skabara, David Sparrowe and Steve Tierney

The first synthesis of regioregular poly(3-hexylselenophene) (P3HS) is reported, and its properties are compared to those of regioregular poly(3-hexylthiophene). P3HS is a promising photovoltaic donor material, exhibiting high crystallinity, high charge carrier mobility and an optical band gap of 1.6 eV.

#### 5064



### Asymmetric synthesis of (+)-*cis*-nemorensic acid from a chiral Diels–Alder adduct of 2,5-dimethylfuran

Jae Yi Sim, Geum-Sook Hwang, Kyung Hwa Kim, Eun Mi Ko and Do Hyun Ryu\*

(+)-*cis*-Nemorensic acid was synthesized from a chiral Diels– Alder adduct prepared by a catalytic enantioselective Diels– Alder reaction with 2,5-dimethylfuran and 2,2,2-trifluoroethyl acrylate.

#### 5066

## Ionic strength mediated hydrophobic force switching of CF<sub>3</sub>-terminated ethylene glycol self-assembled monolayers (SAMs) on gold

Nelly Bonnet, David O'Hagan\* and Georg Hähner\*

Novel oligo (ethylene glycol)  $CF_3$ -terminated switching self-assembled monolayers allow the force experienced by a hydrophobic object to be controlled *via* the ionic strength of the environment.



### Ligand-free Pd/C-catalyzed Suzuki–Miyaura coupling reaction for the synthesis of heterobiaryl derivatives

Yoshiaki Kitamura, Satoko Sako, Takahiro Udzu, Azusa Tsutsui, Tomohiro Maegawa, Yasunari Monguchi and Hironao Sajiki\*

A mild and efficient protocol for the ligand-free and heterogeneous Pd/C-catalyzed hetero Suzuki–Miyaura coupling reaction is described. This method is compatible with a wide range of heteroaryl substrates.

#### 5072

#### Rapid intramolecular heterolytic dihydrogen activation by a four-membered heterocyclic phosphane–borane adduct

Patrick Spies, Gerhard Erker,\* Gerald Kehr, Klaus Bergander, Roland Fröhlich, Stefan Grimme and Douglas W. Stephan

Dihydrogen adds rapidly to a four-membered internal phosphane-borane adduct to yield the ethylene-bridged phosphonium-hydridoborate zwitterion, which stoichiometrically reduces benzaldehyde.











#### Liquid crystals carrying stereodefined vicinal difluoroand trifluoro- alkyl motifs

Marcello Nicoletti, Matthias Bremer, Peer Kirsch\* and David O'Hagan\*

Two novel materials are prepared carrying two or three vicinal fluorines and both display negative dielectric anisotropy ( $\Delta \varepsilon$ ). The conformation of the fluorinated alkyl chains is examined in relation to the magnitude of  $\Delta \varepsilon$ .



rapid umbrella motion  $(k \approx 10^4 \, \text{s}^{-1} \, \text{at} \, 373 \, \text{K})$ 



#### Umbrella motion in aziridines: use of simple chemical inputs to reversibly control the rate of pyramidal inversion

Mark W. Davies, Adam J. Clarke, Guy J. Clarkson, Michael Shipman\* and James H. R. Tucker\*

The molecular motion associated with atomic inversion at an aziridine nitrogen can be repeatedly switched "off" and "on" using simple chemical inputs.



#### Ansa-tris(allyl) complexes of alkali metals: tripodal analogues of cyclopentadienyl and ansa-metallocene ligands

Richard A. Layfield,\* Felipe García, Julien Hannauer and Simon M. Humphrey

Alkali metal complexes of ansa-tris(allyl) ligands are reported. The isoelectronic relationship between these podand-like ligands and the cyclopentadienyl and ansa-metallocene ligands is discussed.

5084

5081



#### Macropolyhedral boron-containing cluster chemistry. Novel intercluster linkages from the reaction of [Pt(cod)Cl<sub>2</sub>] and [PtMe<sub>2</sub>(PMe<sub>2</sub>Ph)<sub>2</sub>] with $6,6'-(B_{10}H_{13})_2O$

Jonathan Bould and John D. Kennedy\*

Neutral  $[(PMe_2Ph)_2PtB_{10}H_{10}-O,H-B_{10}H_{11}Pt(PMe_2Ph)]$  and the  $[(B_{10}H_{13}OB_{10}H_{11})Pt-\{(B_{10}H_{10}OB_{10}H_{12})]^2$  dianion exhibit unusual inter-cluster connectivity modes that involve B–O–B linkages.

#### The unprecedented ring transformation from thiazolinespiro-thiophene to thieno[2,3-*b*]pyrazine involved in the reaction of 2-thiocarbamoyl thiazolium salts with dimethyl acetylenedicarboxylate

Yang Guang Ma and Ying Cheng\*

Reaction of 2-thiocarbamoyl thiazolium salts with dimethyl acetylenedicarboxylate proceeded *via* a tandem [3 + 2] cycloaddition and a unprecedented ring transformation to produce functionalized thieno[2,3-*b*]pyrazine derivatives in good to excellent yields.

#### 5090

### Removal of amorphous carbon for the efficient sidewall functionalisation of single-walled carbon nanotubes

Lidong Shao, Gerard Tobias,\* Christoph G. Salzmann, Belén Ballesteros, Sung You Hong, Alison Crossley, Benjamin G. Davis and Malcolm L. H. Green

The sidewall functionalisation of carbon nanotubes using the standard nitric acid treatment can be greatly enhanced by first removing the amorphous carbon present in the sample.

#### 5093

#### New water-soluble polyanionic dendrimers and binding to acetylcholine in water by means of contact ion-pairing interactions

Cátia Ornelas, Elodie Boisselier, Victor Martinez, Isabelle Pianet, Jaime Ruiz Aranzaes and Didier Astruc\*

A new dendrimer containing 81 benzoate termini binds acetylcholine in water by contact ion-pairing and encapsulation as indicated by <sup>1</sup>H NMR spectroscopy, including DOSY experiments which show that its diameter  $(11 \pm 1 \text{ nm})$  does not increase upon acetylcholine binding.

#### 5096

#### Synthesis of 5'-amino-5'-deoxyguanosine-5'-N-phosphoramidate and its enzymatic incorporation at the 5'-termini of RNA molecules

David Williamson, Martin J. Cann and David R. W. Hodgson\*

5'-Amino-5'-deoxyguanosine-5'-*N*-phosphoramidate (GNHP) was synthesised in four steps from guanosine. GNHP was found to initiate T7 RNAP-promoted transcriptions to afford 5'-GNHP-RNA which hydrolyzes readily to yield 5'-H<sub>2</sub>N-RNA that can be conjugated to activated esters.











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