### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (21) 2637-2760 (2005)



#### Cover

See Qing-Zheng Yang, Olivier Siri and Pierre Braunstein, page 2660. Structure of the zinc(II) complex formed with a new functional zwitterionic benzoguinonemonoimine containing a pendant amino group, superimposed on the H-bonded network generated when OH pendant groups are introduced by transamination reaction. Image kindly produced by Prof. R. Welter (ULP Strasbourg) by permission of Pierre Braunstein et al. from Chem. Commun., 2005, 2660.



#### Inside cover

See Jianguo Huang and Toyoki Kunitake, page 2680. TEM image of latex particles in tubing superimposed onto a SEM image of particles in membrane channels. Image reproduced by permission of Jianguo Huang and Toyoki Kunitake from *Chem. Commun.*, 2005, 2680.

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C41

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**Chemical Science** 

June2005/Volume 2/Issue 6 www.rsc.org/chemicalscience

### FEATURE ARTICLE

#### 2651

### Carbon dioxide and supramolecular chemistry

Dmitry M. Rudkevich\* and Heng Xu

Principles of supramolecular chemisty and molecular recognition are applied to the century-old chemistry between  $CO_2$  and amines in designing novel sensing systems and nanoscale, self-assembling polymeric materials and molecular networks.



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### 2660



### First transamination reactions for the one-pot synthesis of substituted zwitterionic quinones

### Qing-Zheng Yang, Olivier Siri and Pierre Braunstein\*

Reversible transamination reactions carried out in water are used for the first time in quinonoid chemistry to access a range of functional,  $6\pi + 6\pi$  electron molecules with enhanced H-bonding and metal-coordination properties.

### 2663

### Synthesis, characterization, and photoluminescence of isostructural Mn, Co, and Zn MOFs having a diamondoid structure with large tetrahedral cages and high thermal stability

Daofeng Sun, Shengqian Ma, Yanxiong Ke, Tracy M. Petersen and Hong-Cai Zhou\*

Three novel isostructural metal–organic frameworks having a diamondoid structure with large tetrahedral cages have been prepared and characterized.

### 2666

### Optical properties of donor-acceptor phenyleneethynylene systems containing the 6-methylpyran-2-one group as an acceptor

Jonathan C. Collings, Alexander C. Parsons, Laurent Porrès, Andrew Beeby, Andrei S. Batsanov, Judith A. K. Howard, Donocadh P. Lydon, Paul J. Low, Ian J. S. Fairlamb\* and Todd B. Marder\*

Push-pull systems **2** and **3** containing the 6-methylpyran-2-one group exhibit pronounced solvatochromism in fluorescence suggesting a highly polar excited state;  $\Phi_{\rm f}$  for **3** is >0.9.

### 2669

### Template-free, polymerase-free DNA polymerization

### Ye Tian and Chengde Mao\*

A combination of a DNA ligase and a restriction endonuclease provides a DNA polymerase activity, which might suggest a novel strategy for polymer synthesis.











DNA and amino-acid In aqueous



DNA-coated microcrystals

### Design in solid state chemistry based on phase homologies. $Sb_4Te_3$ and $Sb_8Te_9$ as new members of the series $(Sb_2Te_3)_m \cdot (Sb_2)_n$

Pierre F. P. Poudeu and Mercouri G. Kanatzidis\*

The  $(Sb_2Te_3)_{m}$   $(Sb_2)_n$  family represents an example of a natural superlattice. The flexibility achievable through variation of the semi-conducting part  $(Sb_2Te_3)$  with the semi-metallic part  $(Sb_2)$  can help control the electronic and thermal transport properties in this class of compounds.

### **DNA-coated microcrystals**

Michaela Kreiner, Geeta Fuglevand, Barry D. Moore\* and Marie-Claire Parker\*

Water-soluble micron-sized particles coated with a layer of bioactive DNA can be prepared by a generic self-assembly process.

### 2677 Coprecipitate into water-miscible non-solvent nanoparticles and salt in aqueous nanoparticle-coated microcrystals

### Nanoparticle-coated microcrystals

Muthu Murugesan, Douglas Cunningham, José-Luis Martinez-Albertos, Ranko M. Vrcelj and Barry D. Moore\*

Water-soluble micron-sized particles coated with a layer of metal nanoparticles can be prepared by a generic self-assembly process.

2680



### Latex particle-encapsulated titania/polymer composite nanotubings: free-standing, one-dimensional package of colloidal particles

Jianguo Huang and Toyoki Kunitake\*

Latex nanoparticles were packed inside nanometer-thick titania/polymer composite nanotubules templated by porous alumina membrane, to give free-standing particle-in-tubing "micro-beanpods" after removal of the template membrane.

### 2683



Lizhi Zhang,\* Jimmy C. Yu,\* Zhi Zheng and Cheuk Wan Leung

Hierarchical porous iron oxide ( $Fe_2O_3$  and  $Fe_3O_4$ ) films were selectively grown on iron substrates through hydrothermal reactions between an iron substrate and iodine powder in water or ethanol, respectively.

### 2686

# Roles of terminal groups of oligomer electrolytes in determining photovoltaic performances of dye-sensitized solar cells

Moon-Sung Kang, Young Jin Kim, Jongok Won and Yong Soo Kang\*

The terminal groups of oligomers significantly affect the photovoltaic characteristics of dye-sensitized solar cells (DSSC) by causing the changes in ionic conductivity, flatband potential and  $I_3^-$  concentration.

### 2689

### A concise and diastereoselective total synthesis of *cis* and *trans*-pterocarpans

Leticia Jiménez-González, Míriam Álvarez-Corral, Manuel Muñoz-Dorado and Ignacio Rodríguez-García\*

A new strategy for the diastereoselective and convergent synthesis of pterocarpans which is able to control the relative stereochemistry of the molecule through allylation of aromatic aldehydes with cyclic allylsiloxanes is described.

#### 2692

### Synthesis and reactivity of O-acyl selenophosphates

Janusz Rachon,\* Grzegorz Cholewinski and Dariusz Witt

The synthesis of several new *O*-acyl selenophosphates and their rearrangement to *Se*-acyl derivatives are investigated. Experimental evidence of the radical-driven mechanism of these rearrangements is presented.









der in



2698

2701



FAST

hv.

The synthesis of a sterically hindered samarium(II) bis(amidinate) and conversion to its homoleptic trivalent congener

Marcus L. Cole and Peter C. Junk\*

The first samarium(II) bis(amidinate) has been prepared by salt elimination, redox transmetallation/ligand exchange and transamination syntheses. Aspects of the novel chemistry of this species, including the formation of a sterically hindered Sm(III) homoleptic tris(amidinate), are presented.

### Development of novel polymer-type dehydrocondensing reagents comprised of chlorotriazines

Munetaka Kunishima,\* Kazuyoshi Yamamoto, Yasunobu Watanabe, Kazuhito Hioki and Shohei Tani

A novel polymer-type dehydrocondensing reagents has been synthesized by alternating copolymerization between chlorotriazine derivatives and a diol or a triamine, exploiting the chemical property of cyanuric chloride that readily binds to alcohol or amines.

### Illumination of the 9-mesityl-10-methylacridinium ion does not give a long-lived photoredox state

Andrew C. Benniston, Anthony Harriman,\* Peiyi Li, James P. Rostron and Jan W. Verhoeven

The stable radical observed upon photolysis of the 9-mesityl-10-methylacridinium cation should not be taken as evidence for unusually slow charge recombination within the photoredox state.

2704

q



### Microwave-assisted synthesis and *in-situ* self-assembly of coaxial Ag/C nanocables

Jimmy C. Yu,\* Xianluo Hu, Quan Li and Lizhi Zhang

We have demonstrated a new one-pot solution-phase route, namely microwave-assisted hydrothermal reduction/ carbonization (MAHRC), for the rapid synthesis of coaxial Ag/C nanocables that can self-assemble in an end-to-end fashion into interconnected chains.

### 2707

### Mechanistic studies on the asymmetric alkylation of amino ester enolates using a copper(II)salen catalyst

Donatella Banti, Yuri N. Belokon', Wen-Lan (Steffy) Fu, Elisabetta Groaz and Michael North\*

In competition reactions with amino ester enolates, substituted aryl halides react in preference to benzyl bromide. Hammett data indicate that electron transfer from the enolate to the benzylic bromide occurs in the transition state of the reaction.



### Structures of a CO adlayer on a Pt(100) electrode in HClO<sub>4</sub> solution studied by *in situ* STM

Mitsuru Wakisaka, Takaharu Ohkanda, Toshiki Yoneyama, Hiroyuki Uchida and Masahiro Watanabe\*

We for the first time have obtained an *in situ* STM atomic image of a CO adlayer,  $c(6 \times 2)$ -10CO, on a Pt(100)-(1 × 1) electrode in a 0.1 M HClO<sub>4</sub> solution, which can transform to  $c(4 \times 2)$ -6CO at E > 0.3 V vs. RHE.

### 2713

### High molar mass polymers by cationic polymerisation in emulsion and miniemulsion

Séverine Cauvin, François Ganachaud,\* Michel Moreau and Patrick Hémery

The *in situ* preparation of bulky, organosoluble super-acids allows fast cationic polymerisation of *p*-methoxystyrene in an emulsion and the production of high molar mass polymers.

### 2716

### Tin-catalyzed conversion of trioses to alkyl lactates in alcohol solution

Yukiko Hayashi and Yoshiyuki Sasaki\*

A high-yielding preparation of alkyl lactates is achieved by the reaction of trioses, dihydroxyacetone (DHA) and glyceraldehyde (GLA) with tin catalysts in alcohols.











### Synthesis of mesoporous aluminosilicate with zeolitic characteristics using vapor phase transport

Yiwen Zhang, Tatsuya Okubo and Masaru Ogura

A mesoporous material with zeolitic characteristics is synthesized using a vapor phase transport method, along with stabilization of the mesostructure during crystallization of the amorphous walls by carbon filling.

2727



### An intelligence ink for photocatalytic films

Andrew Mills,\* Jishun Wang, Soo-Keun Lee and Morten Simonsen

Major processes associated with a photocatalyst ink for the rapid assessment of photocatalyst film activity.



### One-step synthesis of phenol by $\mathbf{O}^-$ and $\mathbf{OH}^-$ emission material

Ting Dong, Jiang Li, Fan Huang, Lian Wang, Jing Tu, Yoshifumi Torimoto, Masayoshi Sadakata and Quanxin Li\*

A novel approach to the direct synthesis of phenol was obtained with high benzene conversion (30%) and phenol selectivity ( $\sim$ 90%) by using a microporous material  $[Ca_{24}Al_{28}O_{64}]^{4+}\cdot 4O^-$  (C12A7-O<sup>-</sup>) as catalyst with oxygen and water.

### Observations on the reactivity of pentafluorophenyl sulfonate esters

Stephen Caddick,\* Jonathan D. Wilden and Duncan B. Judd

Mechanistic and synthetic studies on PFP-sulfonates demonstrate that alkyl PFP-sulfonates undergo displacement with amines consistent with a sulfene intermediate. Recent work demonstrates compatibility of PFP-sulfonates with aqueous reaction conditions. Further opportunities for two-directional synthesis using bis-PFP derivatives are also presented.

### 2729



## Enantioselective catalytic diamination of alkenes with a bisimidoosmium oxidant

### Kilian Muñiz\* and Martin Nieger

The differentiation of the enantiotopic face of an olefin can be accomplished by application of an oxazolidinone as directing group for a Ti-TADDOL-catalyst. Thus, enantioselective diamination with a bisimido osmium reagent has been achieved for the first time.

### 2732

### Highly regio- and enantioselective thermal [2 + 2] cycloaddition of coumarin in a crystalline inclusion complex under high vacuum

Yongqiang Wen, Yanlin Song,\* Dongbo Zhao, Kuiling Ding,\* Jiang Bian, Xue Zhang, Jingxia Wang, Yang Liu, Lei Jiang and Daoben Zhu

Highly regio- and stereoselective thermal [2 + 2] cycloaddition was achieved by coumarin or thiocoumarin in their inclusion crystalline complexes. Hydrogen bond and high vacuum were found to be indispensable factors for the reaction.

### 2735

## Synthesis of mesoporous titania networks consisting of anatase nanowires by templating of bacterial cellulose membranes

Dayong Zhang and Limin Qi\*

Mesoporous titania networks consisting of interconnected anatase nanowires were synthesized by using unique bacterial cellulose (BC) membranes as natural biotemplates.





### 

### 2738

Synthesis of novel palladium OCN-pincer complexes: unprecedented sequential  $C(sp^3)$ -H activation and aerobic oxidation in the reaction of *N*,*N*-dialkyl-3-[(*N*,*N*-dimethylamino)methyl]-2-iodoanilines with Pd<sub>2</sub>(dba)<sub>3</sub>

Daniel Solé,\* Lluís Vallverdú, Xavier Solans and Mercé Font-Bardia

Palladium OCN-pincer complexes are obtained by reaction of 2-haloanilines with  $Pd_2(dba)_3$  and  $O_2$  by means of an unprecedented process.

### 2741

2744

0.01

dV/dD / cm<sup>3</sup>/(g\*Å)

4 6 8



e3+/H\_C

18 20 22

REACTOR



CO2 /H2O

### In situ <sup>13</sup>C DEPT-MRI as a tool to spatially resolve chemical conversion and selectivity of a heterogeneous catalytic reaction occurring in a fixed-bed reactor

Belinda S. Akpa, Michael D. Mantle, Andrew J. Sederman and Lynn F. Gladden\*

Natural abundance <sup>13</sup>C DEPT-MRI is used to map chemical conversion and selectivity in a fixed-bed reactor.

### Room temperature detemplation of zeolites through H<sub>2</sub>O<sub>2</sub>-mediated oxidation

Ignacio Melián-Cabrera,\* Freek Kapteijn and Jacob A. Moulijn

Detemplation of zeolite beta has been successfully achieved at low temperature by controlled oxidation of the template using  $H_2O_2$  and catalyzed by traces of Fe<sup>3+</sup>, and the pristine structure of the material is preserved.

### 2747

2750



pore diameter (D) / Å

10 12 14 16

### Tetraalkynyl calix[4]arenes with advanced NLO properties

Gunther Hennrich,\* M. Teresa Murillo, Pilar Prados, Kai Song, Inge Asselberghs, Koen Clays,\* André Persoons, Jordi Benet-Buchholz and Javier de Mendoza

Hyper-Raleigh scattering is used to establish the second-order hyperpolarizability values  $\beta$  as well as to address conformational aspects, in particular the dihedral angle  $\theta$ , of two tetraalkynyl-calix[4]arenes in solution.

#### Energetic salts of azotetrazolate, iminobis(5-tetrazolate) and 5, 5'-bis(tetrazolate)

Chengfeng Ye, Ji-Chang Xiao, Brendan Twamley and Jean'ne M. Shreeve\*

1-Methyl-4-aminotriazolium azotetrazolate has a layered structure and exhibits a heat of formation of +4360 kJ kg<sup>-1</sup>.





### 2753

### RMA-3: synthesis and structure of a novel Rbaluminosilicate zeolite

Takuji Ikeda and Keiji Itabashi\*

A novel aluminosilicate zeolite RMA-3 incorporating rubidium ion was hydrothermally synthesized. The framework composed of two sub-building units have two-dimensional straight channels with different sizes of 8-membered rings.



### 2756

## Generation of iodobenzoborirene, a boraaromatic cyclopropabenzene derivative

Holger F. Bettinger\*

Iodobenzoborirene is obtained photochemically ( $\lambda = 308$  nm) from diiodophenylborane by elimination of HI in solid argon at 10 K.



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