#### IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (33) 4129-4236 (2005)



### Cover

See Seiji Shinkai *et al.*, page 4149.

A novel coordination gelator emits a beautiful red phosphorescence avoiding oxygen quenching in the gel phase. Image reproduced by

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RSC Publishing

#### Inside cover

See Jeroen J. L. M. Cornelissen, Floris P. J. T. Rutjes *et al.*, page 4172. Biohybrid amphiphiles. Image reproduced by permission of A. J. (Ton) Dirks, Sander S. van Berkel, Nikos S. Hatzakis, Joost A. Opsteen, Floris L. van Delft, Jeroen J. L. M. Cornelissen, Alan E. Rowan, Jan C. M. van Hest, Floris P. J. T. Rutjes and Roeland Nolte, *Chem. Commun.*, 2005, 4172.

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C65

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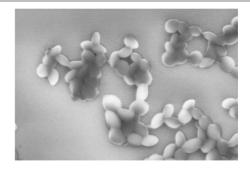
#### FEATURE ARTICLE

#### 4143

### Effects of nanoconfinement on the morphology and reactivity of organic materials

#### Wilhelm T. S. Huck\*

With advances in nanotechnology, organic matter can be confined to lengthscales where it should be possible to influence their physical and chemical properties. In this feature article a number of recent examples of the effects of nanoconfinement on polymers and small molecules are discussed.



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#### 4149

#### Organogel of an 8-quinolinol platinum(II) chelate derivative and its efficient phosphorescence emission effected by inhibition of dioxygen quenching

Michihiro Shirakawa, Norifumi Fujita, Takahiro Tani, Kenji Kaneko and Seiji Shinkai\*

The phosphorescent organogel formed by a newly synthesized 8-quinolinol platinum(II) chelate derivative possesses an attractive ability to inhibit dioxgen quenching of excited triplet states of a chelate moiety.

#### 4152

#### Layer-by-layer assembly of charged poly(phenylacetylene)s with induced macromolecular helicity

Katsuhiro Maeda, Yasuaki Matsushita, Muneyoshi Ezaka and Eiji Yashima\*

The macromolecular helicity of charged poly(phenylacetylene)s induced by small chiral guests in water can be retained by the alternative deposition of achiral polyelectrolytes with opposite charges, resulting in optically active multilayer thin films with a macromolecular helicity memory.

#### 4155

#### Novel HEXOL-type cyclometallated iridium(III) complexes: stereoselective synthesis and structure elucidation

Liangru Yang, Alex von Zelewsky\* and Helen Stoeckli-Evans

Two diastereoisomers of tetranuclear cyclometallated iridium complexes, either having an inner core of HEXOL-type [Ir(IrCl<sub>2</sub>)<sub>3</sub>]<sup>6+</sup> unit and a surface of six chiral, didentate, cyclometallated ligands, are stereoselectively synthesized from an enantiopure pinenopyridine derivative.

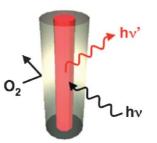
#### 4158

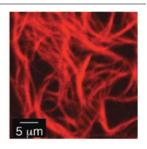
## Allosteric pitch length tuning of a dinuclear double helicate

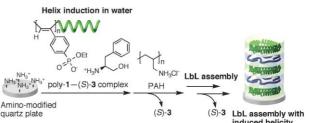
Christian J. Baylies, John C. Jeffery, Tom A. Miller, Ryan Moon, Craig R. Rice\* and T. Riis-Johannessen

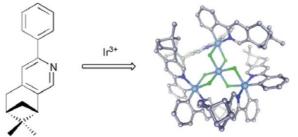
Self assembly of the ditopic ligand  $L^1$  with  $Cu^{2+}$  gives the dinuclear double helicate  $[Cu_2(L^1)_2]^{4+}$ , which can further coordinate s-block cations. This coordination alters the helicate pitch to a variety of different lengths depending upon the size and charge of the guest cation.

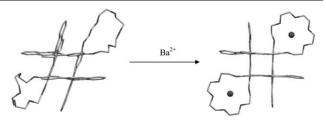






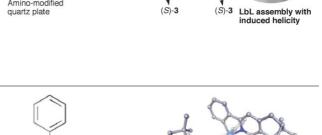






 $[Cu_2(L^1)_2]^{4+}$ 

 $[Cu_2(L^1)_2Ba_2]^{8+}$ 



#### 4161

4164



R = primary or secondary alkyl halide

Simple iron-amine catalysts for the cross-coupling of aryl Grignards with alkyl halides bearing β-hydrogens

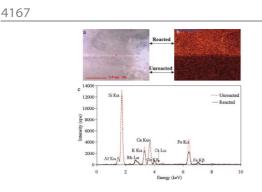
Robin B. Bedford,\* Duncan W. Bruce, Robert M. Frost and Michael Hird

Simple mixtures of iron(III) chloride and amines prove to be highly active catalysts in the coupling of aryl Grignard reagents with primary and secondary alkyl halides.

### Lower rim mono-functionalization of resorcinarenes

Frank Hauke, Andrew J. Myles and Julius Rebek, Jr.\*

A versatile, scaleable, one step synthesis of a lower rim mono-functionalized resorcinarene is described.

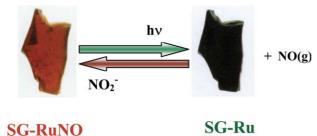


### A high throughput screening method for the selection of zeolites for binding cations

Edel M. Minogue,\* Tammy P. Taylor, Anthony K. Burrell, George J. Havrilla, Benjamin P. Warner and Michael T. Janicke

An effective high throughput screening technique using Micro X-Ray Fluorescence is described for the rapid analysis of zeolites as competitive binding agents for selective cationic sequestration.

4169



### Photochemical release of nitric oxide from a regenerable, sol-gel encapsulated Ru–salen–nitrosyl complex

Jeane Bordini, Peter C. Ford and Elia Tfouni\*

Light activation leads to release of NO from a silicate sol-gel material **SG-RuNO** prepared from the ruthenium complex,  $[Ru(salen)(OH_2)(NO)]^+$  (salen = N,N'-bis-(salicylidene)ethylenediaminato). After photochemical NO photolabilization, **SG-RuNO** can be regenerated from the spent material *via* the subsequent reaction with aqueous nitrite.

#### 4172

## Preparation of biohybrid amphiphiles *via* the copper catalysed Huisgen [3 + 2] dipolar cycloaddition reaction

A. J. (Ton) Dirks, Sander S. van Berkel, Nikos S. Hatzakis, Joost A. Opsteen, Floris L. van Delft, Jeroen J. L. M. Cornelissen,\* Alan E. Rowan, Jan C. M. van Hest, Floris P. J. T. Rutjes\* and Roeland J. M. Nolte

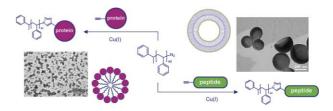
Biohybrid amphiphiles were prepared from azide functionalised PS and an alkyne functionalised peptide or protein *via* a Cu(I) catalysed Huisgen [3 + 2] dipolar cycloaddition reaction.

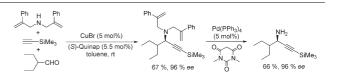
#### 4175

# 2-Phenallyl as a versatile protecting group for the asymmetric one-pot three-component synthesis of propargylamines

Nina Gommermann and Paul Knochel\*

2-Phenallyl was found to be a versatile protecting group of primary amines for the asymmetric three-component synthesis of propargylamines yielding up to 96% *ee.* Removal of 2-phenallyl by a Pd(0)-catalyzed allylic substitution using 1,3-dimethylbarbituric acid leads to primary propargylamines.





#### 4178

### New synthesis method for nickel phosphide hydrotreating catalysts

Shaofeng Yang and Roel Prins\*

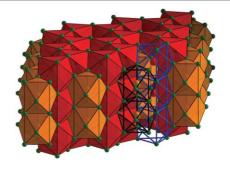
Structure of  $Ni_2P$ , a material that can now be made in very small particles on a support and used for the removal of sulfur and nitrogen from fuels. This material helps to keep the air clean.

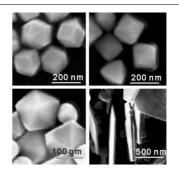
#### 4181

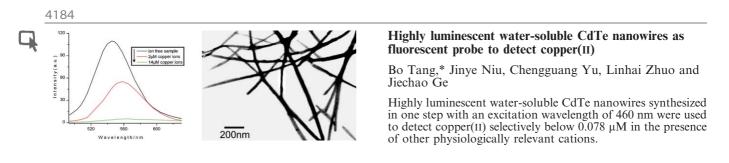
### Shape controlled growth of gold nanoparticles by a solution synthesis

Ying Chen, Xin Gu, Cha-Geng Nie, Zhi-Yuan Jiang, Zhao-Xiong Xie\* and Chang-Jian Lin\*

The shape of gold nanoparticles has been successfully tuned by introducing a small amount of salt into a *N*,*N*-dimethylformamide solution containing poly(vinyl pyrrolidone), and changing the temperature or the concentration of the gold precursor.

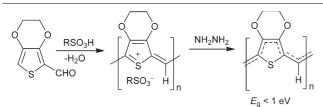






#### 4187

4190



#### A new simple synthesis of poly(thiophene-methine)s

Md. Badruz Zaman and Dmitrii F. Perepichka\*

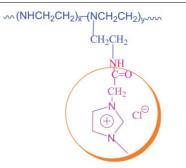
A new acid-promoted polycondensation reaction was applied to transform 3,4-(ethylenedioxy)thiophene-2-carbaldehyde into conjugated heavily *p*-doped poly(thiophene-methine). Dedoping this material with hydrazine leads to a neutral polymer with a band-gap below 1 eV.

#### The first ternary tin(II) nitride: NaSnN

Nicholas S. P. Watney, Zoltán A. Gál, Matthew D. S. Webster and Simon J. Clarke\*

NaSnN features the layered Zintl ion [SnN]<sup>-</sup> which is isoelectronic and isostructural with the layers in elemental arsenic, and is the first example of a ternary nitride containing Sn–N bonds.

#### 4193



Na

### Immobilization of ionic liquid with polyelectrolyte as carrier

Yanfei Shen, Yuanjian Zhang, Qixian Zhang, Li Niu,\* Tianyan You\* and Ari Ivaska

An all-purpose approach to immobilize ionic liquids onto solid supports is proposed by chemical grafting on a polyelectrolyte carrier. As potential applications, the catalytic activity and electrochemically induced change in the contact angle of the surface are discussed.

#### 4196

#### Palladium(0) alkyne complexes as active species: a DFT investigation

Mårten Ahlquist, Giancarlo Fabrizi, Sandro Cacchi and Per-Ola Norrby\*

Alkynes have been found to be excellent ligands for Pd(0); the stability of a range of alkyne-Pd(0) complexes, and their reactivity in oxidative addition, have been investigated by DFT methods.

#### 4199

#### Entropy-controlled supramolecular photochirogenesis: enantiodifferentiating Z-E photoisomerization of cyclooctene included and sensitized by permethylated 6-O-benzoyl-β-cyclodextrin

Gaku Fukuhara, Tadashi Mori, Takehiko Wada and Yoshihisa Inoue\*

The use of the title compound as a flexible chiral sensitizing host for the enantiodifferentiating photoisomerization of (Z)-cyclooctene enables us to dynamically control the supramolecular photochirogenesis.

#### 4202

#### A facile route to fabrication of inorganic-small organic molecule cable-like nanocomposite arrays

Xiujuan Zhang, Weigang Ju, Mingming Gu, Xiangmin Meng, Wensheng Shi, Xiaohong Zhang\* and Shuittong Lee\*

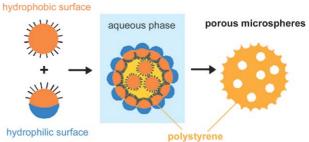
A novel and facile method is reported for the preparation of silver iodide-small organic molecule cable-like nanocomposite arrays in aqueous solution at room temperature.

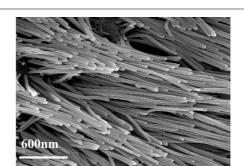
#### 4205

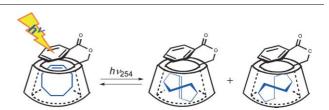
Porous polystyrene microspheres having dimpled surface structures prepared within micellar assemblies of amphiphilic silica particles in water

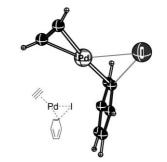
Yoshiko K. Takahara, Shigeru Ikeda,\* Koji Tachi, Takao Sakata, Toshiaki Hasegawa, Hirotaro Mori, Michio Matsumura and Bunsho Ohtani

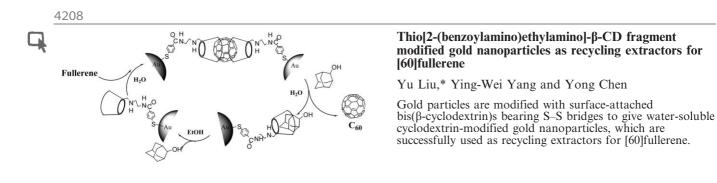
Production of porous polystyrene microspheres having dimpled surface structures was demonstrated using amphiphilic and hydrophobic silica particles as structure-directing agents.

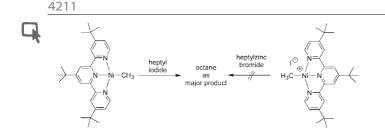












## Analysis of key steps in the catalytic cross-coupling of alkyl electrophiles under Negishi-like conditions

Gavin D. Jones, Chris McFarland, Thomas J. Anderson and David A. Vicic\*

A Ni(I)–alkyl complex and its related Ni(II)–alkyl halide complex have been prepared and used as mechanistic probes of key steps in alkyl cross-coupling reactions.

A simple synthesis of mesoporous carbons with tunable mesopores using a colloidal template-mediated vapor deposition polymerization

Jyongsik Jang,\* Byungkwon Lim and Moonjung Choi

Mesoporous carbons with highly uniform and tunable mesopores were easily fabricated by one-step vapor deposition polymerization (VDP) using colloidal silica particles as templates and polyacrylonitrile (PAN) as a carbon precursor.

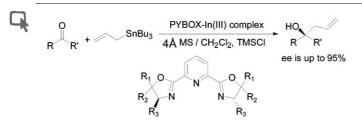
### Enantioselective allylation of ketones catalyzed by chiral In(III)-PYBOX complexes

Jun Lu, Mei-Ling Hong, Shun-Jun Ji,\* Yong-Chua Teo and Teck-Peng Loh\*

In the presence of 20 mol% of chiral catalytic complex prepared from In(OTf)3 and chiral PYBOX, allyltributylstannane reacted with achiral ketones to afford the corresponding homoallylic alcohols in moderate to high enantioselectivities (54–95% ee), which constitutes the first example of enantioselective allylation of ketones catalyzed by the chiral In(III)-PYBOX complex.

4217

4214



100 nm

#### 4219

#### The synthesis of enantiomerically pure 4-substituted [2.2]paracyclophane derivatives by sulfoxide-metal exchange

Peter B. Hitchcock, Gareth J. Rowlands\* and Rakesh Parmar

The use of a chiral sulfoxide moiety allows the simple preparation of a range of enantiomerically pure 4-substituted [2.2]paracyclophane derivatives from a common precursor.

#### 4222

#### The elusive phosphorescence of pyrromethene– $BF_2$ dyes revealed in new multicomponent species containing Ru(II)-terpyridine subunits

Maurilio Galletta, Sebastiano Campagna,\* Manuel Quesada, Gilles Ulrich and Raymond Ziessel\*

Two new multichromophoric systems containing pyrromethene– $BF_2$  dyes and Ru(II) polypyridine subunits are reported. In these species, phosphorescence of the organic chromophores has been obtained for the first time.

#### 4225

## MCuB<sub>7</sub>O<sub>12</sub>·nH<sub>2</sub>O (M = Na, K ): A new copper borate with 14-ring channels

Tao Yang, Guobao Li, Liping You, Jing Ju, Fuhui Liao and Jianhua Lin $\ensuremath{^*}$ 

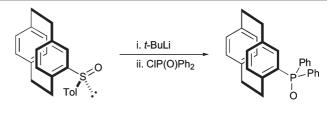
A new series of compounds  $MCuB_7O_{12} \cdot nH_2O$  (M = Na, K) were synthesized in boric acid flux under mild conditions (493 K). The structure consists of octahedral (CuO<sub>6</sub>), tetrahedral (BO<sub>4</sub>) and triangular (BO<sub>3</sub>) units with a 14-member ring borate framework and exchangeable cations.

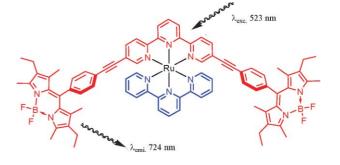
#### 4228

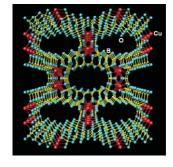
#### Solvolytic routes to new nonabismuth hydroxy- and alkoxy-oxo complexes: synthesis, characterization and solid-state structures of novel nonabismuth polyoxo cations $Bi_9(\mu_3-O)_8(\mu_3-OR)_6^{5+}$ (R = H, Et)

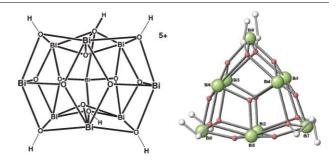
John H. Thurston, Dale C. Swenson and Louis Messerle\*

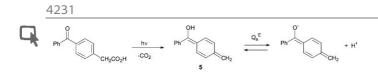
Base hydrolysis of BiO(ClO<sub>4</sub>) yields  $ClO_4^-$  salts of the novel nonabismuth polyoxo cation  $Bi_9(\mu_3-O)_8(\mu_3-OH_6^{5+}$  (1); ethanolysis converts 1 to the ethoxide  $Bi_9(\mu_3-O)_8(\mu_3-OEt)_6^{5+}$  (2).











Ketonization of the remarkably strongly acidic elongated enol generated by flash photolytic decarboxylation of *p*-benzoylphenylacetic acid in aqueous solution

Yvonne Chiang, A. Jerry Kresge,\* Ikenna Onyido, John P. Richard, Peter Wan and Musheng Xu

Photodecarboxylation of *p*-benzoylphenylacetic acid in aqueous solution produces the elongated enol **5**, whose strength as an oxygen acid ( $pQ_a^E = 7.67$ ) makes it more acidic than simple enol analogs by several orders of magnitude.

#### ADDITIONS AND CORRECTIONS

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	~	-	

Roy A. Periana, Oleg Mironov, Douglas J. Taube and Scott Gamble

High yield conversion of methane to methyl bisulfate catalyzed by iodine cations

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